NLGI SPOKESMAN

Serving the Grease Industry Since 1933 - VOL. 85, NO. 1, MARCH/APRIL 2021

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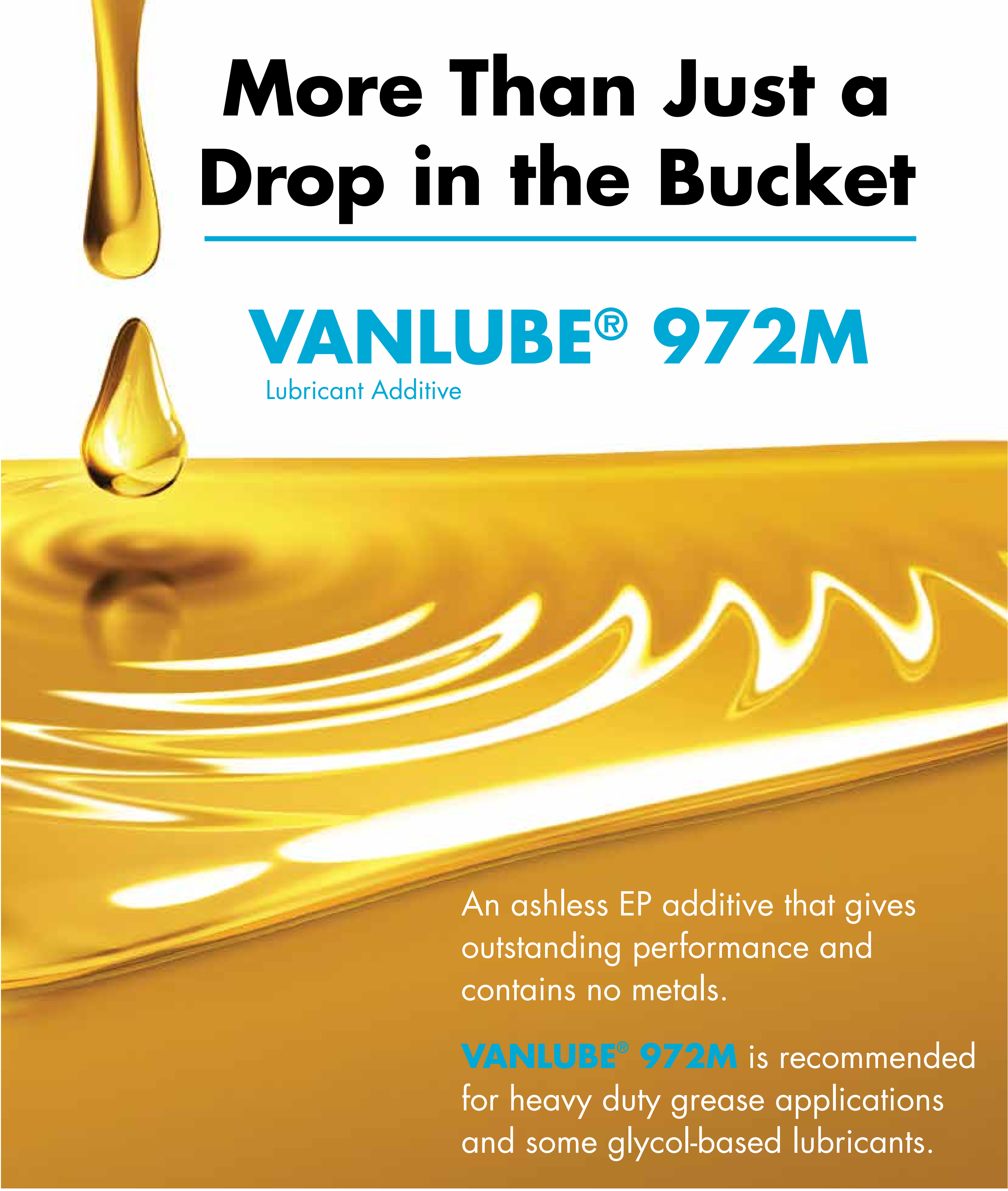
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Happy Spring!

ON THE COVER

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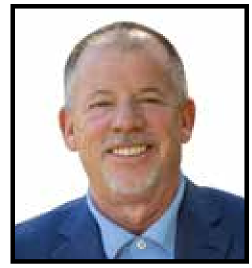
PRESIDENT'S PODIUM



Jim Hunt
NLGI President
2020 – 2022

Dear NLGI Family,

We sincerely hope that you and your families are safe and healthy.
As more vaccines continue to be distributed, we all hope to resume our normal lives in the very near future.
Let's keep the faith…

The NLGI sincerely wants to continue to keep our six strategic priorities front and center for all our valued members. I will report the current status of each strategic priority throughout this year's Spokesman issues. As you may recall, the Jan/Feb issue featured an update on membership growth, engagement and global outreach. In this issue, I'd like to focus on "Enhancing opportunities for networking."

The timing to focus on the networking opportunities could never be better with the recent announcement of the **NLGI Annual Meeting** planned at the **Loews Ventana Canyon, Tucson, AZ, September 27-30, 2021**. The theme of this year's annual meeting is **"The Future of High-Performance Lubricating Greases."** The NLGI Annual Meeting has always been the best opportunity for our members to network. We sincerely hope we all goes well and we can resume our in-person meeting this September. I feel confident that we will never take this amazing opportunity to network for granted again.

The details of this years meeting will be announced in the coming weeks. We appreciate your patience as we address the logistical details. Creating a safe, worry-free meeting experience is our top priority. We look forward to seeing you all in September!

To ensure we provide the best NLGI networking opportunities, all our members also have access to the real-time membership list to better connect with other NLGI members. You must be logged into the members' only section of the website to access this information. This is truly another value-added benefit for the NLGI members.

Additionally, consider expanding your network through other NLGI offerings including:

- Monthly HPM webinar series
- NLGI LinkedIn group
- NLGI committees

*Contact NLGI HQ for more information
As a friendly reminder, we are still actively collecting NLGI membership dues. During this challenging time, membership retention and growth remain essential to the long-term health and sustainability of the NLGI. If you have not yet renewed your membership, please do so at your earliest convenience.

Once again, the NLGI is tremendously grateful for your continued support, loyalty and commitment.

Stay safe and healthy,
Jim Hunt
President, 2020 – 2022

Industry Calendar of Events 2021

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

23rd NLGI India Chapter Lubricating Grease Conference	**Apr 16 – Apr 17**	Virtual	India Chapter Meeting Info
1st Virtual ELGI-STLE Tribology Exchange Workshop	**Apr 19 – 20, 2021**	Virtual	www.elgi.org
2nd ELGI Virtual Working Group Meetings	**Apr 21 - 23, 2021**	Virtual	www.elgi.org
ELGI Virtual Grease Symposium	**Apr 26 – 27, 2021**	Virtual	www.elgi.org
STLE Virtual Annual Meeting & Exhibition	**May 17 – 20, 2021**	Virtual	STLE Annual Meeting
2021 ILMA Re-Engage	**Jun 2 – 4, 2021**	Orlando, FL, USA	ILMA Re-Engage
NLGI 2021 Annual Meeting	**Sept 27 – 30, 2021**	Tucson, AZ, USA	NLGI Annual Meeting
ILMA Annual Meeting	**October 9-12, 2021**	Phoenix, AZ	ILMA Annual Meeting
NORIA Machinery Lubrication Conference& Exhibition	**October 19-21, 2021**	Louisville, KY	NORIA Machinery Lubrication Conference & Exhibition

NLGI Serving the grease industry since 1933

Warm Welcome to our New NLGI Members

North American Lubricants Company	*Marketing – Low*	**USA**
Bardahl Manufacturing Corporation	*Manufacturer*	**USA**
Lubdeco SA de CV	*Manufacturer*	**Mexico**

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elgi
2021 Spring
Virtual Events

NLGI 2021
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THE FUTURE OF
HIGH-PERFORMANCE
LUBRICATING GREASES

September 27-30 | Tucson, AZ USA

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✓ STEP 2: **Reserve your hotel** – Hotel link / information included in registration confirmation email

✓ STEP 3: **Book your flight to sunny Tucson, AZ.** *See you in September!*

Agenda

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Industry Speaker

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Attendee List

For more information visit:
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A Fresh Look at Lithium Complex Greases Part 1: How Did We Get Here?

J. Andrew Waynick
NCH Corporation

Abstract
In 1942, five U.S. Patents were issued with Clarence E. Earle as named inventor. These five patents defined simple lithium soap greases for subsequent decades. However, simple lithium soap greases were limited in their high temperature utility owing, at least in part, to their dropping points – typically about 200 C. In August, 1959, only months after the Earle patents expired, a lithium complex grease patent issued that featured for the first time what would become the most common compositional approach. Stearic acid was used as the long chain fatty acid. An alkyl diester of sebacic acid was also used. Reaction with aqueous lithium hydroxide accomplished the thickener formation. Reported dropping points were between 248 C and 276 C or higher when mineral oil was used as the base oil.

Since that 1959 patent, many modifications and advances in lithium complex grease formulation and manufacturing have been documented. All such development work can be placed into one or more of only three categories: formulation change, process change, or manufacturing equipment change. Interestingly, no organized and critical review of all the decades of development of lithium complex greases can be found in the published literature. Due to the very large amount of such development work, an exhaustive review is beyond the scope of this paper. However, a review that focuses on some of the most important of those developments can be provided. This paper provides such a review. By doing so, explanations of certain physical and chemical behavior never fully documented are brought into full focus. This, in turn, allows those involved in the formulation and manufacture of lithium complex greases to gain an improved perspective on potential future paths to improve the cost-effectiveness of these greases. Given the recent sharp rise in lithium prices, such an improved perspective is more relevant now than ever before.

Beginnings

As early as the late 1930's, it had been reported that lithium soaps were not effective grease thickeners.[1] This was proven incorrect when five U.S. patents with Clarence E. Earle as the named inventor issued in 1942.[2-6] Those patents used lithium stearate as the thickener and provided the defining foundation for every lithium-based lubricating grease that would be subsequently developed.

Within less than one year of the issuance of the Earle patents, Harold M. Fraser filed for a U.S. patent using lithium 12-hydroxystearate as the thickener. That patent issued in 1946.[7] Fraser claimed that such greases had several improvements over lithium stearate-thickened greases, including superior batch to batch consistency, improved initial structure, and improved shear stability. These improvements were optimized by mixing the batch as it cooled from its nearly melted state at about 218 C. This was in contrast to lithium stearate greases where such mixing resulted in less initial structural uniformity and inferior structural stability. However, by the time Fraser's patent issued, additional developments in lithium-based lubricating greases were well underway. A good discussion of those developments has already been provided elsewhere.[8] A few representative examples are discussed here.

A common theme for improving simple lithium soap greases was the incorporation and neutralization of additional acids besides the longer chain fatty acids that served as the thickener acids. One example of this was provided in another U.S. patent by Fraser that was issued in 1948.[9] In this patent, a lithium soap grease made using a mixture of longer chain fatty acids (hydrogenated fish fatty acids) was modified by minor amounts of acetic and naphthenic acids that were co-neutralized. A minor improvement in dropping point (217 C) and improvements in oil bleed and shear stability were obtained.

Similar improvements were documented when other supplementary acids were used:

1. Acids produced by the oxidation of high molecular weight microcrystalline wax.[10]
2. Castor oil that had been phosphated prior to saponification (or the corresponding phosphated fatty acids).[11]
3. C3 to C18 fatty acids that had a hydroxyl group covalently bonded to a carbon less than nine carbons from the carboxylate carbon (preferably on the beta, gamma, or delta carbons).[12,13]

Another approach that was used to modify simple lithium soap greases involved oxidizing either the base oil [14] or the fatty oil used for saponification [15]. In both of these cases, the additional acids formed by the oxidation process were also neutralized to form the corresponding lithium salts.

The above examples are only a representative few of a much larger body of similar work. Thus, by the mid-1950's, many such examples of the use of supplementary thickener or thickener modifying acids had been documented.

By that same time, the investigation of the effect of manufacturing process conditions on grease properties was also well underway. This investigation was aided by the availability and use of a relatively new tool: electron microscopy. Grease thickener fiber structure began to be examined within a few years of the Earle and Fraser patents using electron microscopy. This tool allowed the evaluation of the effects of various grease manufacturing process variables on lithium soap grease thickener fiber structure. This in turn allowed correlations to be observed between the thickener fiber structure and finished grease properties.[16]

One important manufacturing process variable was the top processing temperature. In an important 1952 study by Brown and co-workers [17], the authors showed that if the lithium soap grease was heated to about 204 C before cooling, well-formed thickener fibers resulted. If the top temperature was lower (160 C – 199 C), the thickener structure was either granular or otherwise characterized by less well-formed fibers.

Cooling rate from the top processing temperature was also determined to be important. More rapid cooling resulted in shorter final thickener fibers. Slower cooling from top temperature resulted in longer thickener fibers. Obviously, giving the thickener fiber formation process more time encouraged a slower thickener crystallization process resulting in longer fibers being formed. Once the cooling had progressed past a critical temperature threshold value (about 185 C), thickener fibers would have been fully formed. The effect of cooling rate beyond this point would be expected to be minimal.

Two examples of lithium soap-thickened grease development that took advantage of these manufacturing process insights were:

1. Use of a mixture of slow cooled lithium soap grease (longer fiber) and rapid cooled lithium soap grease (shorter fiber) to improve the structural stability of the final grease.[18]
2. Upon cooling from top temperature, holding the grease at 94 C – 120 C for up to four hours to allow the thickener to achieve an improved structural equilibrium prior to final cooling and milling.[19,20]

These two developments were published in 1952 and 1953, respectively.

The above brief summary highlights the significant body of development work that focused on the use of differing and/or complementary thickener acids and the advantageous use of heating and cooling rates. In view of this, the development of higher dropping point co-crystallized lithium complex greases beginning in 1959 was probably inevitable. The next section summarizes key examples of this development.

The First Higher Dropping Point Lithium Complex Grease

The first lithium complex grease using a longer chain monocarboxylic acid and a shorter chain dicarboxylic acid as the two thickener acids was documented in 1959 with the issuance of a U.S. patent by Pattendon and co-workers [21]. Key features of this technology included the following:

1. Di-esters of sebacic acid (C10) or adipic acid (C6) were used. They were the source of the shorter chain dicarboxylic acid.
2. The longer chain monocarboxylic acid was stearic acid.
3. The stearic acid and diester of the dicarboxylic acid were added to base oil and then reacted with LiOH(aq); then heated to about 204 C; then cooled and finished.
4. The stearic/sebacic ratio was 2.1 (wt/wt).
5. The dropping point ranged from 248 C to >260 C.

Perhaps the most important observation reported in this work was the fact that if sebacic or adipic acid was used instead of the esters, a low yield grainy product was obtained. The dropping point of such products was only about 182 C. Therefore, the esters of the shorter chain dicarboxylic acid gave much higher dropping points than the corresponding free acids. The reason for this was almost certainly due to a coupling effect caused by the esters and the transient alcohols formed by their hydrolysis. As the thickener reactions proceeded, the resulting alcohols and the yet unreacted esters could, by virtue of their high polarity, facilitate a more intimate association of the two lithium thickener salts as they formed. This would improve the co-crystallization of the lithium stearate and the di-lithium sebacate/adipate. This in turn would be expected to better impart the much higher melting point of the shorter chain di-lithium salt to the overall thickener fiber structure. Table 1 provides literature melting points [22-24] for the most common co-crystallized lithium complex thickener salts.

TABLE 1:
LITHIUM COMPLEX THICKENER SALTS

Thickener salt	Melting Point, C
Lithium stearate	220
Lithium 12-hydroxystearate	200
Di-lithium sebecate	>400
Di-Lithium azelate	>400
Di-lithium adipate	>400

The first significant use of the term "lithium complex grease" was applied to these co-crystallized greases. However, true Werner coordination complexes are not formed in such greases. The term "lithium complex" has always been and remains a marketing term.[25]

About 21 years later, it was reported that lithium complex grease development began in late 1959. According to the authors of this 4-page 1980 paper [26], the first attempts used formic and acetic acid as complexing thickener acids without success. Eventually azelaic and sebacic acids were used, but problems were still encountered. However, when the diesters of these acids were used, the desired high dropping point greases were obtained. No record of these details could be found in the literature prior to 1980 except for the above described 1959 Pattendon patent. However, that patent was filed with the U.S. Patent and Trademark Office on August 23, 1956, and the work documented in that patent application had obviously been done even earlier. Thus, the statement that lithium complex grease development began in late 1959 cannot be correct. Nonetheless, this short 1980 paper proves that the beneficial effect of esters on lithium complex thickener co-crystallization was recognized two decades after it was first documented.

Further Developments In The Co-Crystallization Process

After the 1959 Pattendon patent issued, significant further development was reported that focused on different thickener reactants and processing conditions. Four representative examples of this are provided in this section.

In 1960, another U.S. patent by Pattendon and co-workers issued.[27] This work used somewhat different key features:

1. A mixture of a longer chain fatty acid, shorter chain dicarboxylic acid, and polyhydric alcohol was heated to 148 C – 177 C for one to five hours to form complex acidic polyester mixture.
2. Typically, stearic acid was the longer chain fatty acid.
3. Adipic, sebacic, or azelaic acid were the shorter chain dicarboxylic acids.
4. Ethylene glycol was the polyhydric alcohol.
5. The reaction mixture was cooled, added to base oil, and reacted with LiOH(aq).
6. The mixture was heated again to as high as 204 C to fully form and dehydrate the grease.
7. Alcoholic bi-products would be the ethylene glycol that was regenerated when the polyester was hydrolyzed and reacted with lithium hydroxide.
8. The stearic/azelaic ratio was 1.5 (wt/wt).
9. The dropping point was ~ 260 C.

It should be noted that this process used two distinct heating/cooling cycles. The first was when the complex polyester mixture was made. The second was when this mixture was reacted with aqueous lithium hydroxide to form the finished grease. The complex polyester material was formed due to the use of di-functional acids and alcohols. The longer chain fatty acid (a mono-carboxylic acid) would serve to terminate the polymer chain growth. Only part of the longer chain fatty acid would be needed to cap the polyester polymer. The remainder would result in the overall mixture being acidic. When the acidic polyester mixture was reacted with aqueous lithium hydroxide, the ester linkages would be hydrolyzed, and the thickener acid salts would form. The same effect of unreacted ester moieties and transient alcohols on co-crystallization would explain the resulting high dropping point values. However, this technology used typically more di-carboxylic acid than the 1959 technology, as seen in the ratio of the longer chain to shorter chain acids.

Another aspect of this technology involved the ethylene glycol that was used. Ethylene glycol has a boiling point of 198 C. If the other components of the lithium complex grease did not inhibit the volatilization of the ethylene glycol, it should have been released at a top temperature of 204 F. Obviously, the release of such materials would not be considered acceptable by today's standards. However, based on the information provided in the 1960 patent, it was not clear if the glycol would be absent in the final grease or still present at some measurable level.

Two U.S. patents by the Gilani and co-workers issued in 1972 and 1974. These patents provided higher dropping point co-crystallized lithium complex greases without requiring the use of esters or glycols [28,29]. Both patents claimed that residual glycol in lithium complex greases were deleterious to oxidation stability and water resistance. Additionally, both patents used 12-hydroxystearate (12-HSA) and azelaic acid as the two thickener acids.

The first of these two Gilani patents [28] provided the following key features:

1. 12-HSA and dicarboxylic acid (azelaic) were dissolved in base oil at 82 C – 93 C.
2. LiOH(aq) was slowly added and reacted.
3. During the reaction, the mixture was heated to 204 C – 221 C to complete the reaction and dehydrate the initial product.
4. The initial product was rapidly cooled to about 104 C.
5. Then it was heated again to about 177 C – 190 C.
6. Then it was cooled as rapidly as possible to at least 116 C, and the grease was finished.
7. The 12-HSA/azelaic ratio ranged from 1.6 to 2.95 (wt/wt).
8. Dropping point was as high as 282 C.

The most important point of this process was the two heating/cooling cycles. Both thickener acids (neither being in ester form) were reacted simultaneously with the aqueous lithium hydroxide. However, the reaction mixture did not experience just one heating and cooling cycle like greases made by the 1959 Pattendon patent. Instead, two heating/cooling cycles were used. When the work of this patent was done, it was well known that heating to higher temperatures promoted better co-crystallization. And if the mixture was heated to higher temperatures more than once, this would further enhance the desired co-crystallization process. Apparently, using not one but two heating/cooling cycles provided sufficient co-crystallization despite not using the ester form of the di-carboxylic thickener acid.

The second Gilani patent [29] was very similar to the first one. However, each thickener acid was separately reacted with a stoichiometric equivalent portion of aqueous lithium hydroxide. The 12-HSA was reacted first. Then the mixture was heated to 149 C and then cooled. Then, the azelaic acid was reacted with its stoichiometric equivalent portion of aqueous lithium hydroxide and the mixture was once again heated. This time it was heated to about 198 C before being cooled and finished. Dropping points were reported as high as 329 C. The 12-HSA/azelaic acid ratio ranged from 2.0 to 3.2 (wt/wt). Note that both Gilani patents required two separate heating/cooling cycles.

Finally, another U.S. patent by Carley and co-workers issued in 1984 that provided a process for making co-crystallized lithium complex greases that required only one heating and cooling cycle.[30]

This was accomplished by utilizing the following process steps:

1. Adding both thickener acids (12-HSA and azelaic acid) in an initial portion of base oil.
2. Neutralizing the mixture by the very slow and controlled addition of aqueous lithium hydroxide at a temperature below 100 C.
3. Holding the mixture at that temperature until the reaction was complete.
4. Heating the mixture to between 199 C and 204 C.
5. Rapidly quenching to 190 C by base oil addition.
6. Cooling and finishing as typical for a lithium complex grease.

Dropping points were greater than 260 C. The 12-HSA/azelaic acid ratio was 2.6 (wt/wt) or lower.

Although the inventors provided no explanation as to why their process provided high dropping points without use of esters or two heating/cooling cycles, they did state that the very slow addition rate of the aqueous lithium hydroxide was the critical feature. Apparently, the very slow addition rate of the base allowed the two thickener acids to react in such a way that relative rate of consumption of both acids was about the same. This would be expected since the reactivity of the carboxylic acid group on 12-HSA would be about the same as the two carboxylic acid groups on azelaic acid. Such a reaction profile would allow a more gradual and uniform co-crystallization of the two thickener salts that would be further enhanced during the one heating/cooling cycle. Apparently, this was sufficient to provide the high dropping points.

The basic features of these four patents are summarized and compared in Table 2.

TABLE 2: COMPARISON OF CO-CRYSTALLIZED LITHIUM COMPLEX GREASE PATENTS

U.S. Patent Number	2,940,930	3,681,242	3,791,973	4,435,299
Date of issuance	6/14/1960	8/1/1972	2/12/1974	3/6/1984
Used ester form of dicarboxylic acid	No	No	No	No
Used initially formed reactive polyester polymer	Yes	No	No	No
Thickener formation reactions done in concert or separately	In concert	In concert	Separately	In concert
Number of heating/cooling cycles	2	2	2	1
Top processing temperature, C	204	204-221	198	199-204
Dropping Point, C	~260	282	329	260
12-HSA/azelaic (sebacic) ratio, wt/wt	1.5	1.6-2.95	2.0-3.2	2.6

Use of Anhydrous Lithium Hydroxide Dispersion

In all the previous examples of preparing lithium complex greases, the first step was to add one or both thickener acids to a base oil and heat the mixture until the acids were melted and dissolved. Then an aqueous solution of lithium hydroxide was added to form the thickener salts. Since this aqueous solution was insoluble and immiscible in the hydrocarbon base oil, the reaction of acid and base was a two-phase reaction limited to the water/oil interface.

An alternative approach was disclosed in 2005 that involved preparing and using a stable dispersion of anhydrous lithium hydroxide in base oil.[31] In that initial work, a polyisobutenyl succinic acid was used as the dispersant. Several additional papers [32-35] and U.S. Patents [36,37] have been published since this initial work that provide further information on the use of such anhydrous lithium hydroxide dispersions when making lithium-based greases. Key features of these papers included the following:

1. The anhydrous lithium hydroxide dispersion was applicable to simple lithium and lithium complex greases.
2. Reaction with acids proceeded rapidly allowing an earlier heating to top temperature.
3. Heating to top temperature took less time since no water from LiOH was added (no latent heat of vaporization required).
4. Only one heating/cooling cycle was required when making lithium complex greases.
5. There was a reduction in process kettle residence time and associated energy/manpower costs.
6. Yields were at least as good as traditional methods that use LiOH(aq).
7. Dropping points were somewhat higher than when LiOH(aq) was used.
8. Simple lithium or lithium complex greases could be made in hydrolytically unstable ester base oils, thus avoiding the use of pre-formed lithium soaps.

The last point is especially noteworthy. When making lithium-based greases in vegetable oils and other hydrolytically unstable synthetic base oils, the thickener reaction competes with the hydrolysis of the base oil. The result is usually a very poor grease structure or no grease structure at all. Since the lithium hydroxide dispersion contains no water, the potential for unwanted hydrolysis of the ester base oil is limited to the very small amount of water formed during the acid-base thickener reaction. By running that reaction close to the boiling point of water, essentially no base oil hydrolysis occurred.

Lithium Soap Greases Using Boric Acid

The lithium complex greases made with the previously described technologies had high dropping points due to the efficient co-crystallization of the two thickener salts. This efficient co-crystallization resulted in thickener fibers that were significantly impacted by the much higher melting point of the lithium di-carboxylate salt. However, another method to significantly increase the dropping point of simple lithium soap greases was developed that involved the use of boron-containing compounds. This section deals with the use of boric acid. The next section deals with organo-boron compounds.

Under all but the most severe conditions, boric acid is not a Bronsted acid, but is instead a mono-basic Lewis acid [38]. In view of this, the formula for boric acid may best be written as B(OH)3 instead of the more common H3BO3. The Lewis acidity of boric acid is due to the boron atom having only six valence electrons and a single low-energy orbital that contains no electrons. Thus, the boron atom in boric acid is electrophilic and will readily accept a pair of electrons from an electron-rich donor. The most common such donors will be compounds that contain nitrogen, oxygen, or sulfur atoms with such available electron pairs.

This Lewis acidity of boric acid was applied to lithium soap-thickened greases in a 1973 U.S. patent by Gary L. Harting.[39] The same technology was discussed a few years later in a published paper.[40] The key features of this technology were as follows:

1. The thickener components were a long chain hydroxy-carboxylic acid (12-HSA), B(OH)3, and LiOH(aq).
2. The LiOH(aq) and B(OH)3 were added to 12-HSA in base oil at about 82 C.
3. After the thickener formation reactions were completed, the product was mixed and heated to 199 C.
4. Additional base oil was added; then the grease was cooled and finished.
5. An optional second hydroxy hydrocarbyl acid could be used (salicylic acid).
6. Dropping points ranged from 251 C to >260 C.

The most likely reason for the high dropping points of these greases was the reaction of boric acid with an electron-rich moiety in the lithium 12-hydroxystearate salt. Only two such moieties are possible: (1) one of the two equivalent oxygen atoms in the carboxylate anion; (2) the oxygen on the 12-hydroxy group. The second choice is the most likely. The reason for this is that if the boric acid reacted with one of the carboxylate oxygens, the resonance stability of that anion group would be lost. No such loss of stability would occur if the reaction was with the 12-hydroxy group. The reaction of boric acid with the 12-hydroxy group would result in possible -O-B-O- bridges between two 12-hydroxystearate units. Such crosslinking would be expected to increase the stability and effective melting point of the thickener. Addition of the second hydroxyl hydrocarbyl acid would provide an added diversity to the overall crosslinked structure.

Another similar use of boric acid as a means to increase simple lithium soap grease dropping points was provided in a 1983 U.S. patent by James F. Stadler.[41] Key features of this technology were as follows:

1. The thickener components were a long chain hydroxy-carboxylic acid (12-HSA), B(OH)3, LiOH(aq).
2. A poly-hydric alcohol was required when LiOH(aq) was reacted with B(OH)3.
3. The poly-hydric alcohol was preferably glycerol or cis-di-hydroxybenzene.
4. The LiOH(aq) and B(OH)3 were added to 12-HSA in base oil at about 82 C in the presence of the alcohol.
5. After the thickener formation reactions were completed, the product was mixed and heated to 199 C.
6. Additional base oil added; grease cooled and finished
7. Dropping points ranged from 261 C to >315 C; without the poly-hydric alcohol dropping points ranged from 218 C to 228 C.

Lithium Soap Greases Using Organo-Boron Compounds

Only a few years after the establishment of boric acid as a reagent to increase simple lithium soap grease dropping point, an extention of this approach began to be developed and disclosed. This approach involved first reacting boric acid with a compound from one of many organic chemical families. Then, the resulting organo-borated compound was added to a simple lithium soap-thickened grease to elevate the dropping point. The various organic chemical families involved all had at least one electron-rich reactive funtional group that could react with boric acid. This technology was disclosed by the same group of inventors in a series of at least 15 U.S. Patents that issued between 1986 and 1997.[42-56]

Key features of this technology were as follows:

1. The compounds that were borated included epoxides, alcohols, catechols, Mannich bases, alkoxylate alcohols, oxazolone, alkyoxylated amides, and amines.
2. The borated compounds were added to the lithium 12-hydroxystearate base grease at typical temperatures for grease additives.
3. Optional organic S/P compounds were sometimes used to provide further increase in dropping point.
4. The S/P compounds included zinc dithiophosphates and/or mixtures of organic phosphates, phosphites, and sulfurized olefins/sulfurized triacylglycerides.
5. Dropping points were as high as 327 C.

The inventors claimed that overborating the organic compound produced the greatest increase in dropping point. This likely indicated that extended –O-B-O- linkages were being formed in or between molecules of the organic compound. When the borated compound was added to the simple lithium soap grease, further reaction would be expected to occur to link the borated organic compound to the 12-hydroxystearate groups. The borated organic material could also provide a means to provide linkages between 12-hydroxystearate groups in a manner similar to when boric acid was used. What was not clear was whether or not such cross linkages occurred during the actual grease making process, or during the actual running of the dropping point test. Either way, the measured dropping point was significantly increased by the use of this technology. Table 3 provides a summary of these patents.

TABLE 3: U.S. PATENTS COVERING ORGANO-BORON COMPOUNDS TO INCREASE SIMPLE LITHIUM SOAP GREASE DROPPING POINTS								
U.S. Patent Number	4,582,617	4,600,517	4,655,948	4,743,386	4,780,227	4,781,850	4,828,732	4,828,734
Date of issuance	4/15/1986	7/15/1986	4/7/1987	5/10/1988	10/25/1988	11/1/1988	5/9/1989	5/9/1989
Organic compound family to be borated	hydrocarbyl epoxides	hydrocarbyl alcohols	catechols	phenolic or thio amine Mannich base	alkoxylated alcohol	catechols	hydrocarbyl diol	oxazolines

TABLE 3 (CONTINUED): U.S. PATENTS COVERING ORGANO-BORON COMPOUNDS TO INCREASE SIMPLE LITHIUM SOAP GREASE DROPPING POINTS							
U.S. Patent Number	4,961,868	5,068,045	5,084,194	5,211,860	5,211,863	5,242,610	5,595,961
Date of issuance	10/9/1990	11/26/1991	1/28/1992	5/18/1993	5/18/1993	9/7/1993	1/21/1997
Organic compound family to be borated	organic S/P, S/N, or S/P/N compound	alkoxylated amide	hydrocarbyl amines	n-hydrocarbyl propylenediamine	organic S/P, S/N, or S/P/N compound	organic S/P, S/N, or S/P/N compound	hydrocarbyl esters

Within about two years of the issuance of the first of these organo-boron patents, a U.S. Patent issued by Hans D. Grasshoff and co-workers.[57] This patent combined the technologies of using shorter chain dicarboxylic acids and boric acid to increase the dropping point of simple lithium soap greases. The inventors apparently overcame an obviousness objection during patent application prosecution by specifically limiting the shorter chain dicarboxylic acid to a branched chain with 5 to 14 carbons total and 4 to 10 carbons in the non-branched portion of the entire hydrocarbyl group. Specifically, 3-tert-butyl adipic acid was preferred. The use of this branched shorter chain thickener acid provided superior FE9 bearing life at both 150 C and 200 C when compared to greases that used the more established non-branched dicarboxylic azelaic and sebacic acids. Some additional improvement in dropping point was also documented.

By 1995, the combined use of the more established sebacic and azelaic acids with boric acid was also being used to increase the dropping point of lithium-based lubricating greases.[58]

As these organo-boron patents began to expire, various borated oil-soluble additives including esters and amines became available specifically for the purpose of increasing the dropping point of lithium soap-thickened greases. Corresponding to this commercialization, papers were presented and published describing the ability of such additives to increase lithium-based grease dropping point and modify the temperature-dependent rheological properties.[59-63] Some of these borated additives were also shown to provide additional benefits such as improved oxidation resistance and corrosion protection. Additionally, certain interactions were noted in some of the studies. For instance, if a borated additive was used in a lithium soap grease made with hydrogenated castor oil (HCO), the dropping point was not elevated to the same extent as when the grease was made with 12-HSA. This appeared due to a competing reaction of the borated compound with the glycerol that derived from the HCO. Apparently, this reaction prevented the desired cross linking of 12-hydroxystearate moieties.

Other Methods To Increase Dropping Point
As lithium complex greases became more established, additional methods to increase the dropping point of simple lithium soap-thickened greases were developed. In 1983, a U.S. patent by Michiharu Naka and co-workers issued that taught a lithium co-crystallized grease using stearic acid and a di-ester of sebacic acid.[64] The base oil was usually a hydrolytically stable polyol ester. The resulting initial lithium complex grease was further reacted with an organo-phosphate or phosphite ester. Dropping points ranged from 238 C to more than 260 C and bearing life at 135 C was extended.

Phosphate chemistry was also used in a 1989 U.S. patent by Takehiro Koizumi and co-workers.[65] A lithium 12-hydroxystearate grease was modified by reacting either phosphoric acid or a phosphorous acid ester with lithium hydroxide. A lithium borate was also included in the final reacted grease. Dropping points as high as 261 C were obtained.

A 1985 U.S. patent by Rebecca C. Pehler and co-workers taught a simple lithium 12-hydroxystearate grease modified by a multi-component additive system as a means to elevate the dropping point and increase high temperature utility in CV-joints.[66] The additive system was a mixture of zinc di-alkyl di-thiophosphate, zinc di-amyl di-thiocarbamate, sulfurized sperm oil substitute, and the reaction product of coconut oil and diethanolamine. Dropping points were as high as 276 C.

A 1990 U.S. patent by George P. Newsoroff provided a method to increase the dropping point of lithium 12-hydroxystearate greases by co-reacting aqueous lithium hydroxide with 12-HSA and di-alkyl esters of terephthalic acid.[67] The di-esters were exclusively taught as being required for higher dropping points. This is consistent with what has been discussed earlier in this paper regarding the coupling effects of the esters of thickener acids. Dropping points were reported as high as 295 C.

Finally, a 1993 U.S. patent by Patricia R. Todd taught the use of various combinations of organo-sulfur and organo-phosphorus compounds and/or their amine salts as a means to elevate the dropping point of simple lithium soap-thickened greases by at least 30 C.[68]

Manufacturing Equipment
The previous sections of this paper have dealt with the advances in chemistry and manufacturing processes involved in making higher dropping point lithium-based greases. Concurrent with the development of these advances, improvements in the grease manufacturing equipment were also developed.

The oldest equipment used to make lubricating greases has been kettles. The earliest such kettles were open to the atmosphere [69] and were heated by open fire. Such heating was eventually replaced by electricity or by jackets that utilized steam or hot oil. Closed kettles that can be pressurized during the reaction and heating portions of the process provided an improvement to open kettles. This improvement was due to the closed reaction of the thickener components. As already mentioned, the reaction to form the thickener salts proceeds at the base oil/water interface. Like all chemical reactions, this reaction proceeds more rapidly at higher temperatures. However, in an open kettle, the reaction temperature must be kept sufficiently below 100 C so as to prevent loss of the water. In a closed and pressurized kettle, the temperature can be higher without such loss of water. This allows the thickener reaction to proceed at significantly higher temperatures than when run in open kettles.

Another method of making lithium-based greases involves the use of a Contactor™ reactor. Contactor™ reactors have been so used since at least 1929.[70] A Contactor™ reactor is basically a closed pressurized grease kettle with a high-speed impeller at the bottom of the central tube section that forces the reaction mixture back up through a surrounding annular space. The recycled mixture is then directed to the top of the center section.[71] In this way the thickener reactants are recycled at high space velocity and reacted under heat and pressure. The final reacted and dehydrated grease is pumped out to a finishing kettle where additives are added and the final consistency is adjusted.

One example that took advantage of the sealed, pressurized, and high shearing conditions of the Contactor™ reactor was a 1995 U.S. Patent by Phillip W. Brewster.[72] According to the process in this patent, a co-crystallized lithium complex grease using 12-HSA and a typical shorter chain dicarboxylic acid was dissolved in an initial portion of base oil. The mixture was heated in the Contactor™ reactor under pressure to a temperature above the boiling point of water. Then lithium hydroxide was added, and the reaction was taken to completion. The remaining process steps were similar to what has been previously described.

The advantages of using a Contactor™ reactor include shortened manufacturing time, lower heating costs, less requirements on the milling/homogenizing equipment, improved yield, and smoother consistency.

In 1969, a major advance in grease manufacturing equipment was first disclosed by John H. Green and co-workers: continuous manufacturing.[73-74] This new technology involved both new equipment and the associated process. This technology was first developed primarily for simple lithium soap greases, but was eventually modified by Arnold C. Witte and co-workers to allow the manufacture of lithium complex greases.[75,76]. Several papers have also been presented and published that describe this continuous grease manufacturing process.[77,78]

The original continuous grease manufacturing process generally involved three sections of equipment: the reaction section, the dehydration section, and the finishing section. The engineering of these sections allows liquid raw materials to be continually metered into the reaction section, and finished product of the desired consistency to be pumped out of the finishing section. Over subsequent decades, improvements to the original continuous manufacturing process and other equipment designs were developed.[79,80] In one relatively recent U.S. patent, Gian L. Fagan provided a continuous process specific to high dropping point lithium-based greases that used a borated additive but did not use a shorter chain di-carboxylic acid.[81] Advantages of continuous manufacturing processes include higher manufacturing throughput, elimination of batch-to-batch inconsistency, and improved yield.

Conclusions

The following conclusions are supported by the information reviewed in this paper:

1. Today's lithium complex grease technology is the result of gradual improvements in three areas:
   a. Chemistry.
   b. Processing.
   c. Equipment.

2. These three areas did not improve separately; instead, most improvements involved a combination of two or all three.

3. Three broad categories of lithium complex greases are:
   a. Lithium 12-hydroxystearate/di-lithium azelate (sebacate).
   b. Lithium 12-hydroxystearate enhanced with boron-containing and/or other additives.
   c. A combination of the first two categories that reduces but does not eliminate the dicarboxylic acid.

4. Typically, in all reaction schemes, a source of LiOH is added to a source of the thickener acid(s) (separately or together) in a portion of the base oil.

5. An appropriate combination of mixing, heating, cooling, and usual finishing methods result in the final grease.

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NLGI Interviews Michael M. Khonsari, PhD

Dow Chemical Endowed Chair and Professor of Mechanical Engineering
Department of Mechanical and Industrial Engineering
Louisiana State University
Baton Rouge, Louisiana

By Mary Moon and Raj Shah

Dr. Michael Khonsari's love of solving problems led him to three academic degrees in mechanical engineering and a productive career as an educator, researcher, author, and director of CeRoM, the Center for Rotating Machinery, at Louisiana State University (Baton Rouge). NLGI awarded him a grant to research failure of mechanical components and greases, and then recognized the results of his studies with the Clarence Earle Award. In this interview with NLGI, Dr. Khonsari explains his research as well as his patent for mechanical seals, the bearing whirl controversy, and his newest studies using contact angle measurements to evaluate water resistance and consistency of very small grease samples. To learn more, read on! (*Photos courtesy of Michael N. Khonsari except where noted*)

Education

NLGI: Please tell us a little bit about your education and your interest in engineering.

MK: Two areas of science attracted me: medical sciences and engineering. My love of solving problems (and discomfort at the sight of blood) tilted my career to engineering. As would any Texan, it was my dream to study at the main campus of the University of Texas at Austin, the flagship of the UT System. There were over 40,000 students and excellent engineering programs at UT Austin.

Today, the University continues to be a top-ranked public institution with an excellent reputation for research and many distinguished faculty members. In 2019, John Goodenough, a professor in the Cockrell School of Engineering, received the Nobel Prize for Chemistry for his part in developing the lithium-ion battery, an essential power source for most portable electronic devices and a means to store energy produced by solar panels and wind turbines. This was the ninth Nobel Prize awarded to UT faculty including Ilya Prigogine (Chemistry, 1977) and Steven Weinberg (Physics, 1979). I am proud that I was a Longhorn and received my BS, MS, and PhD degrees from UT Austin.

NLGI: Why did you choose mechanical engineering?

MK: Mechanical engineering is, in a sense, the mother of the engineering field. It is a particularly flexible and uniquely stable profession because mechanical engineers can work on electronics, chemicals, civil infrastructure, and even aerospace applications.

NLGI: Why did you get your doctorate?

MK: As an undergraduate, I knew that the BS would not be my terminal degree. During my senior year in college, I enrolled in graduate school without missing a beat. I wrote a thesis, received my MS, and continued directly to pursue a

PhD. I was super interested in what I was learning and had the energy and the drive to continue my education.

One never knows what will happen from one day to the next.... My modus operandi has always been: If you can do it now, don't wait for tomorrow. It is never too late to pursue an advanced degree. Follow your interests today, don't wait for tomorrow.

If you can do it now, don't wait for tomorrow.

NLGI: What are some of the things you learned along the way?

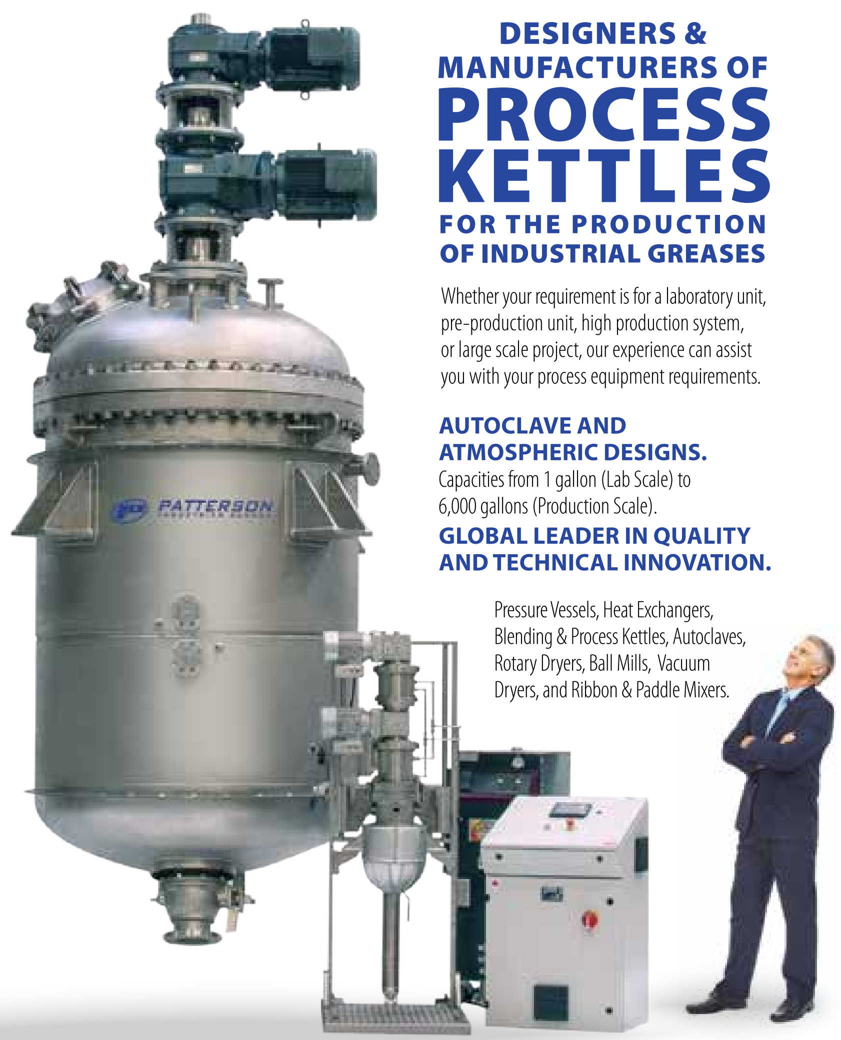
MK: "Learn more and figure out what excites me professionally for the rest of my life" has been my mission and continues to be my vision. My masters' thesis was on thermal energy storage. Then, I became interested in the tribology of mechanical systems before it became a common subject in the mid to late 80s. So, I was lucky to learn two distinctly different areas of the mechanical engineering field. This helped me appreciate and make connections between thermal and mechanical systems.

NLGI: What did you like about your coursework?

MK: I am very much a self-taught learner, someone who takes the initiative. At UT Austin, I took a number of self-paced courses where I could study, advance from one chapter to the next, and take exams at my own pace. I particularly remember one of my favorite courses that was about Nuclear Reactors and involved fairly complex mathematical treatments.

NLGI: Did you study lubrication or tribology?

MK: Yes, primarily at the PhD level. I did my dissertation on how thermal effects influence



the performance of journal bearings. Dr. Joseph Beaman, my PhD advisor, was a major influence on my development. His superb intellect set a great example. He gave me a great gift by allowing me to think independently and work on my own.

A graduate-level course on lubrication and bearing design was my favorite course in the mechanical engineering curriculum. It was taught by the late Professor H. Grady Rylander, a leading expert and a very practical engineer. He later became a member of my PhD dissertation committee and evaluated my research before I graduated.

> ***Learn more and figure out what excites me professionally for the rest of my life***

Career in Education and Research

NLGI: What was your career path?

MK: I always liked teaching. As a graduate student, I enjoyed working as a teaching assistant for different courses. If you really like to find out whether you have a good understanding of a subject matter, try teaching it to someone else!

After finishing my PhD, I left warm Austin, Texas and headed to my first professional job at The Ohio State University in cold Columbus, Ohio. That winter, the temperature went down to -16 F. We even had snow in the month of May! Then, my career path took me to professorial positions at the University of Pittsburgh (Pennsylvania) and Southern Illinois University (Carbondale, Illinois). After a few years, I accepted a position as the first holder of the Dow Chemical Endowed Chair and Professor of Mechanical Engineering at Louisiana State University (Baton Rouse, Louisiana). I have held this Chair at LSU for 21 years.

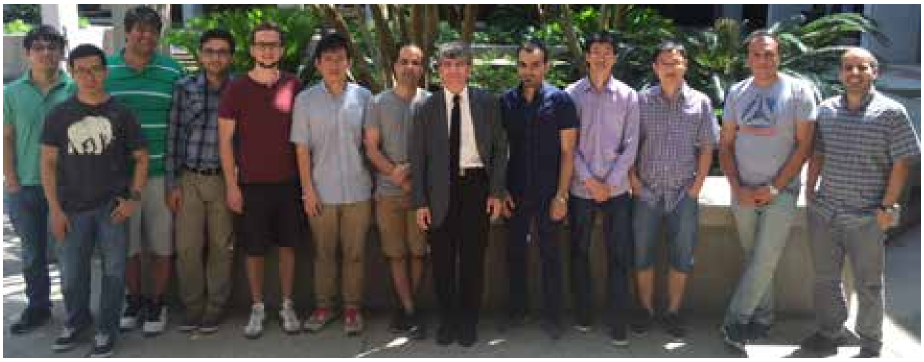
NLGI: Do you have any suggestions for developing a successful career?

MK: Lee Iacocca, former CEO of the Chrysler Corporation, said that in business, "You lead, follow, or get out of the way!" Well, this is also true when it comes to research. Following others does not work in the long run. So, you either lead or get out of the way. It is most rewarding to be in a leadership position in research. For me, it has been extremely satisfying and productive to train students to become a highly-skilled workforce and push the envelope of research to new heights. Translation of research to industrial applications is of utmost importance to ensure that the fruits of research are realized by those who need them and can put them to good use.

NLGI: What skills are important for effective educators?

MK: Clarity of presentation and organization. Educators are role models to their students. Giving clear presentations and generating excitement in the subject matter can spark the interest of many young talents. Who knows where the next Steve Job or Bill Gates will come from? They may be among your audience or even the readers of this interview.

NLGI: How would you describe your philosophies of education and research?



Michael with students in the courtyard at LSU

MK: I challenge my students to think independently, watch for surprising results, and be creative. We always report our research in archival, peer-reviewed journals, which requires responding to critical questions posed by the anonymous reviewers. I insist that my students report all of their data and the details of their work so that any reader can repeat the analysis or experiment and validate the results.

When my students are writing papers, I also remind them to always ask themselves, "SO WHAT?" I expect that each paper shows how our results can be put to use by readers, particularly those working in industry. Accordingly, in many of our papers, we provide examples of how to apply our results in practice.

NLGI: Which work habits are particularly helpful for researchers?

MK: Independent thinking and curiosity. If a supervising professor outlines every step of research for the students and does not give them a chance to think independently, their students will not grow. By allowing their creative juice to run, I learn more from my graduate students through their research.

Michael in the lab at CeRoM

NLGI: Please describe your research group and laboratory at LSU.

MK: I am the founding director of the LSU Center for Rotating

Machinery (CeRoM), which focuses on interdisciplinary problem solving and R&D. Much of CeRoM's work is done in close collaboration with industrial partners to provide cutting-edge technological innovations to complex problems in engineering systems. The Center serves as an efficient avenue for technology transfer and a forum for exchanging ideas with OEMs, end-users, and industrial researchers.

The Center's activities cover many aspects of tribology:

- lubrication, friction, and wear;
- dynamics and vibration analysis of machinery;
- materials selection, fatigue, and damage analysis;
- measurement, testing, and sensing;
- modeling and simulation;
- and education.

These activities directly support improvements in design, manufacturing, diagnostics, reliability, performance, and durability of bearings, seals, gears, turbines, compressors, generators, and other vital mechanical systems and components.

Tribology and fatigue testing facilities at CeRoM include an optical profilometer, a laser texturing device, and a host of specialized equipment for testing mechanical seals and heavily-loaded journal and roller bearings.

Wear, fretting, and fatigue are manifestations of the same physics – the natural tendency for disorder (entropy) to increase. Degradation of grease due to shearing also falls into this category.

NLGI: Please tell us about your current research interests and projects.

MK: We are concentrating on the science of materials degradation to understand how machinery component performance changes with age due to disorder. Wear, fretting, and fatigue are all examples of degradation in mechanical systems. All of these phenomena (as well as corrosion) are manifestations of the same physics – the natural tendency for disorder (entropy) to increase. In fact, degradation of grease due to shearing also falls into this category. We are developing new means to predict the behavior of machines and estimate their remaining useful life.

NLGI: What inspired you to write your book, *Applied Tribology: Lubrication and Bearing Design*?

MK: Over the past five decades, enormous advances have been made in the frontiers of tribology R&D. Fundamentals have been developed to the point that lubrication regimes are fairly well understood, computerized design codes are available, and performance analyses and prediction methodologies are well developed. Nevertheless, information about these advances is spread out in tribology journals. I saw the need to collect this information in a readily digestible form for students and practitioners of tribology. This inspired me to team up with Dr. E. R. Booser to write a well-balanced book that fulfills the needs of students and practicing engineers. To this end, our book contains numerous examples, sample problems, and illustrations that show how each concept can be applied in practice. Our book is now in its third edition; a number of universities have adopted it for their courses.

***Applied Tribology: Lubrication and Bearing Design*, 3rd Edition**

By Michael M. Khonsari and E. Richard Booser

Tribology in Practice Series, Wiley, 2017
Print ISBN:9780470057117
Online ISBN:9780470059456

NLGI: Do you have a favorite laboratory test?

MK: Our labs are well equipped with a variety of testing machines for lubrication and grease studies, e.g., viscometers, a temperature-controlled rheometer, a grease worker, a penetrometer, etc. We even have a grease kettle on loan from an industrial partner to test one of our new ideas! I'm particularly interested in our contact angle goniometer, which is an instrument for measuring the contact angle of a droplet on a substrate. To the best of my knowledge, this test has not been widely used for greases until now.

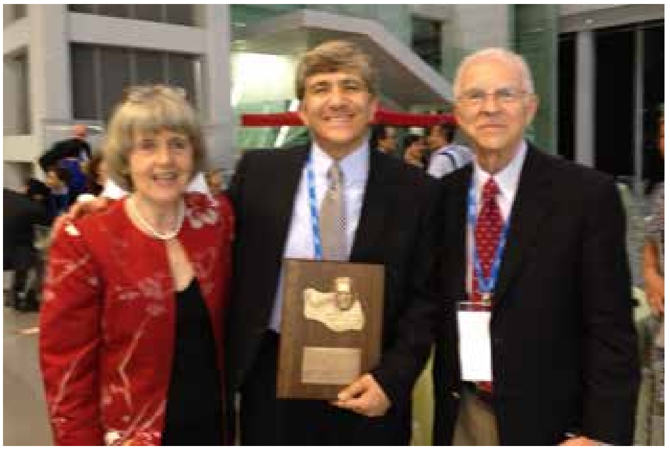
NLGI: Have you discovered something new?

MK: We discovered that we can examine the water resistance of grease by measuring the contact angle of a water droplet on a small grease sample. And, we can use this instrument to assess grease consistency using only a very small sample! This is ideal for testing used or in-service grease.

Imagine having a portable device that a user can take to the field to immediately measure the consistency of a grease sample from a bearing on-site! We have recently published journal articles on our discovery, applied for a patent, and plan to build a portable prototype. We hope that this new procedure will eventually become a standard test to help practicing engineers and maintenance crews maintain machinery.

NLGI: Please tell us about your favorite patent.

MK: Mechanical seals are used widely in chemical, petrochemical, process, and other industries. Yet, these seals are very susceptible to failure, and the results can be catastrophic. For this reason, seals are replaced on a regular basis—some of them after only a few months in service—to avoid failure. Careful examination showed that many mechanical seals failed due to interfacial frictional heat between the mating and primary seal rings. With this realization, we came up with a new seal design that substantially reduces interfacial temperature.



Michael with Prof. Richard F. Salant (Georgia Institute of Technology, Atlanta, Georgia) and Prof. Salant's wife, Barbel, after Michael received the 2013 Mayo D. Hersey Award at the World Tribology Congress (Torino, Italy). The ASME (American Society of Mechanical Engineers) presents the Mayo D. Hersey Award to individuals in recognition of distinguished and continued contributions over a substantial period of time to the advancement of the science and engineering of tribology.

Tests of new prototype seals in the CeRoM laboratories showed the efficacy of our patented design. One of our new seals has already undergone a field test in a pump at a chemical plant in Baton Rouge. It has been operating for over 10 years without failure! The chemical company painted their pump purple and gold in honor of LSU.

The take-home message is that by carefully examining the root cause of failure, one can come up with a solution—a new design, in this case—that can solve the problem.

NLGI: What has been your most challenging research problem?

MK: Dr. John Burt Newkirk (1920–2019, a former research scientist at General Electric Company) discovered bearing whirl, a peculiar form of journal bearing instability that gives rise to rapid and uncontrollable system failure. In the 1950s, he published a paper that suggested heating the oil supply to suppress this instability. Colleagues' extensive test results appeared to contradict what Newkirk reported. As to precisely how temperature affects bearing whirl remained a mystery.

I became very interested in bearing whirl after communicating with researchers at General Motors Company. In the early 2000s, I collaborated with former students to build a test rig and figure out the reason for this discrepancy. In 2005, our experimental and analytical results finally settled this controversy.

So, who was right? We discovered a very peculiar phenomenon that had not been reported before. There is a peculiar operating region in the bearing stability map that is very sensitive to temperature. You can stabilize the bearing by sufficiently heating or cooling the oil supply! So, both Newkirk and his colleagues were correct, and the 50-year mystery was finally solved! The "SO WHAT?" of this problem is that engineers can control bearing whirl by either heating or cooling the oil supply and avoid catastrophic failure. We published these results in journals. To communicate these findings to engineers working in industry, we also wrote a book with comprehensive details.

Influence of Inlet Oil Temperature on the Instability Threshold of Rotor-Bearing System, J.K. Wang and M.M. Khonsari, *Journal of Tribology*, 128(2), 2006.

Applied Tribology: *Lubrication and Bearing Design*
3rd Edition, Chapter 8

Thermohydrodynamic Instability in Fluid-Film Bearings

By J. K. Wang and M. M. Khonsari

John Wiley & Sons, West Sussex, UK, 2016
ISBN 978-0-470-05721-6

Grease Industry

NLGI: What are your thoughts about the global lubricating grease industry?

MK: Grease is an amazing material, and it is hard to imagine a better substitute lubricant for many applications. Nonetheless, I see many opportunities to improve standards based on the latest R&D. Unfortunately, not many universities specialize in this subject matter, and this trend needs to be reversed. It is great to see that NLGI has taken a major step by providing research grants to universities.

NLGI: Which developments show promise for affecting the grease industry?

MK: Automating grease production, optimizing grease performance, and taking advantage of new research to improve degradation behavior can have profound implications for the grease industry.

NLGI: Which technologies show promise for greases?

MK: Chemistries are needed to minimize oxidation and improve service life, develop "green greases", and better means are needed to predict remaining useful grease life.

NLGI: Which new engineering developments will influence greases and their use?

MK: I believe mechanical and chemical engineering will continue to be the major influencers.

NLGI

NLGI: Are you active in NLGI?

MK: When it comes to grease, the NLGI Annual Meeting is the place to go. I plan to attend upcoming meetings. My students are interested and plan to attend, too. I hope that some of our latest research work can be disseminated at NLGI Meetings and considered for standardization.

Dr. Khonsari received the Clarence Earle Memorial Award (Photo courtesy of NLGI)

NLGI: How have you benefitted by attending NLGI meetings?

MK: The 2019 NLGI Annual Meeting was a great opportunity to meet many grease experts and learn from their experiences. Dr. Piet Lugt was the keynote speaker. We have been communicating about working on some very fascinating problems associated with grease. These types of interactions could not have happened otherwise.

NLGI: In 2020, you received the Clarence Earle Memorial Award from NLGI for contributions to the science of grease. Please tell us about your contributions.

MK: This Award recognizes the results of research where we applied the principles of thermodynamics to analyze the mechanical degradation of grease. We asked ourselves, "What happens to a material as it ages or degrades?" The answer is that degradation is a consequence of disorder that develops as the material ages. We applied the principles of irreversible thermodynamics to figure out the rate of degradation in terms of entropy generation in the materials. We reported how to predict the remaining useful life of a mechanical system that is wearing out or degrading due to cyclic fatigue, or grease that is aging due to mechanical shearing.

We are grateful to NLGI for providing a research initiation grant to fund these studies. I am grateful to my students, K.P. Lijesh and Alan Gurt, who worked on this project. They presented their results at the 2019 NLGI Annual Meeting and published two papers in *The NLGI Spokesman*. We wrote an introductory book on the thermodynamics of fatigue to teach scientists and engineers how to apply this new development.

Introduction to Thermodynamics of Mechanical Fatigue

By M. M. Khonsari and M. Amiri

CRC Press, Taylor & Francis Group, Boca Raton, FL, 2013

ISBN 978-1-4665-1179-8

Perspectives

NLGI: Please tell us about your family.

MK: My wife, Karen, and I have three sons, Maxwell, Milton, and Mason. Maxwell earned his mechanical engineering degree from LSU and now works in Philadelphia; Milton is a student at LSU, and Mason is in high school.

Michael and his wife, Karen, on Lake Mary in Mississippi

NLGI: What do you like to do when you have free time?

MK: I am very interested in art, particularly oil paintings. My favorites are the classical works of old European masters—portraits that beautifully capture a moment with very distinct facial expressions of the subjects. So, I enjoy collecting and displaying these types of artwork. I also enjoy reading and listening to audiobooks.

NLGI: Where is your favorite place to travel?

MK: My favorite place to visit is Vienna, Austria. Ludwig Boltzmann, an Austrian scientist interested in entropy, is buried in a cemetery outside Vienna. The Boltzmann equation, which describes entropy, is carved on his tombstone.

NLGI: If NLGI members travel to Louisiana, do you recommend special places to visit?

MK: Visit the LSU campus. We have a live tiger on campus! Plan to visit Patrick Taylor Hall, which houses the engineering departments—the largest single engineering building in the country. And of course, visit the Center for Rotating Machinery.

NLGI: Would you like to recommend any of your favorite books or technical journals to NLGI members?

MK: I am the Editor-in-Chief of the *ASME Journal of Tribology*, which publishes the latest research works on tribology after rigorous review.

Surely You're Joking Mr. Feynman, my favorite book, describes the life and experiences of the Nobel Prize-winning physicist. He was truly a curious character.

NLGI: If you could have dinner with any three people, living or deceased, who would you choose, and what would be on the menu?

MK: Leonardo Da Vinci, Richard Feynman, and Nicola Tesla. We would have fish and vegetables!

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.

Michael with sons, Milton, Maxwell, and Mason, in the Blue Ridge Mountains, Atlanta, GA

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. Her experience in the lubricant and specialty chemicals industries includes R&D, project management, and applications of tribology and electrochemistry. She served as Section Chair of the Philadelphia Section of STLE.

Dr. Raj Shah is currently a Director at Koehler Instrument Company, Long Island, NY where he has lived for the last 25 years. An active NLGI member and he served on the NLGI board of directors from 2000 to 2017. A Ph.D in Chemical Engineering from Penn State University and a Fellow from the Chartered Management Institute, London, Dr. Shah is a recipient of the Bellanti Sr. memorial award from NLGI. He is an elected fellow by his peers at NLGI, IChemE, STLE, INSTMC, AIC, MKI, Energy Institute and the Royal Society of Chemistry. He has over 300 publications and is currently an Adjunct Professor at the Dept. of Material Science and Chemical engineering, State University of New York, Stony Brook. Currently active on the board of directors of STLE he volunteers on the advisory boards of several universities. More information on Raj can be found at https://www.nlgi.org/nlgi-veteran-member-raj-shah-presented-with-numerous-honors-in-2020/

High-Performance Multiuse (HPM) Grease Column

HISTORY AND GOALS

NLGI currently licenses lubricating grease through its Certification Mark program, which includes the well-known GC-LB Performance Classification. Today, NLGI has licensed over 300 products and growing daily.

In 2015, NLGI began efforts to upgrade the GC-LB specification due to advancements in materials, technologies and applications, as well as precision issues with several of the D4950 tests. In July 2019, the decision was made to develop a new set of specifications with higher performance and broader utility to the industry.

These new specifications evolved into what today is known as NLGI's High-Performance Multiuse (HPM) Grease Specification. An additional goal was to define greases that meet core HPM specification and subcategories with tests and limits for enhanced performance in the following areas:

- HPM Core Spec
- HPM Core with enhanced Water Resistance (HPM + WR)
- HPM Core with enhanced Salt Water Corrosion Resistance (HPM + CR)
- HPM Core with High Load Carrying Capacity (HPM + HL)
- HPM Core with enhanced Low Temp Performance (HPM + LT)

FREQUENTLY ASKED QUESTIONS

What is NLGI doing to promote the HPM certification to end users, grease manufacturers and marketers?

NLGI has launched a marketing campaign that includes exposure to over one million targeted, industry professionals! NLGI's marketing campaign includes print, digital and social media exposure in several publications and organizations including:

- Efficient Plant
- Plant Engineering
- Pumps & Systems
- Construction Business Owner
- Targeted Groups on LinkedIn
- NLGI India Chapter
- ELGI
- ILMA
- STLE
- ALIA
- F&L Asia
- Lubes'n'Greases

I'd like to help spread the word about HPM. Does NLGI have any guidelines around doing so?

Yes. Please click HERE for the marketing guidelines on promoting HPM. Please contact NLGI for a sample HPM logo for internal and external use.

How do I certify my product(s)?

NLGI has partnered with Center of Quality Assurance (CQA) to administer this program. A five-step process is required to register your products. Please visit HERE for information on how to download the application documents.

Are we required to submit a sample as part of the application process?
Yes. CQA will collect a sample and test against the HPM specification using a third-party lab.

Has CQA started accepting applications?
Yes. NLGI has started accepting applications from grease manufacturers and marketers for the HPM certification program and expects several companies to offer grease with the HPM trademark as the year progresses.

Where can I find a copy of the HPM specification?
You can find the HPM specification HERE.

What are the future plans for HPM?
NLGI will:

- Continue work on high-temperature and/or long-life categories (two to five years), and consider initiation of work on other categories as is deemed appropriate.
- Continue to refine testing and work on current test reproducibility issues in conjunction with ASTM.
- Adjust or add to specifications as needed to reflect testing capabilities and industry needs with the intention to treat the specifications as “living documents.”
- Continue to encourage active involvement from NLGI membership and industry partners in steering the development process into the future.

For more information, please contact NLGI HQ at 816-524-2500 or CQA at 989-496-2399

GC-LB Retrospective

ASTM D4950 / NLGI GC-LB Certification

With the introduction of NLGI's new series of High Performance Multiuse (HPM) grease specifications, NLGI wanted to highlight our original set of grease specifications for Automotive Service Greases and offer a reprint of a Spokesman article from 1990. In this article, Tom Verdura describes the development process and content of this well-known classification. The GC-LB certification remains extremely popular today with over 300 products currently registered against this classification. NLGI will maintain this certification for the foreseeable future, even with the addition of the new HPM certification.

■ HISTORY

It has long been recognized that the diversity of specifications for Automotive Service Greases, established by the Original Equipment Manufacturer (O.E.M), have made it difficult, if not impossible, for the marketer of lubricating greases to make available all the many specified products.

With the issuance of ASTM D4950 Standard Classification and Specification for Automotive Service Grease, it became not only possible, but convenient to offer the products needed to provide proper service of automotive equipment.

■ ASTM D4950 Includes Specifications For Two Category Groups:

Chassis lubricants (letter designation L) and wheel bearing lubricants (letter designation G). Performance Classifications within these Groups result in two letter designations for chassis greases (LA and LB), and three for wheel bearing greases (GA, GB, and GC). The automotive industry is in general agreement that the highest performance classification, to date, in each group (LB and GC) is suitable for service relubrication.

To facilitate communicating this information to users, NLGI has developed an identifying symbol, i.e., the NLGI Certification Mark. The O.E.M.s Owner's Manuals, most of which refer to this Mark, advise users to use those greases carrying this Mark on the label. It is, therefore, advantageous for marketers of automotive grease to incorporate the Mark into their product label.

Since only the highest performance classification is acceptable to O.E.M.'s, only products meeting LB and GC standards are authorized to use the Mark. Should any future changes occur in ASTM D4950, NLGI will similarly revise its highest performance classifications for the Mark.

■ MORE INFORMATION

- Click HERE for a list of current GC-LB products
- Interested in applying? Click HERE to learn more.
- Interested in purchasing the ASTM D4950 standard? Click HERE.

Classification and Specification for Automotive Service Greases — A New Industry Standard

By Thomas M. Verdura, General Motors

Presented at NLGI's *56th Annual Meeting, October* 1989, St. *Louis,* MO USA

Thomas M. Verdura received a B.S. (cum laude) in Chemistry from the University of Detroit in 1961. He joined General Motors Research Laboratories in 1955 as a Laboratory Technician investigating the properties of fluid lubricants. He was promoted to Research Chemist in 1961 and assigned research projects in automotive applications of lubricating greases. Mr. Verdura is presently a Staff Research Scientist and continues his studies of the performance of greases with added responsibilities for research in properties of automatic transmission fluids. He is chairman of ASTM DO2 Subcommittee G on Lubricating Greases and is an active member in a number of other technical organizations. A former instructor in the NLGI Education Course, Mr. Verdura has contributed several technical papers to the NLGI Spokesman. He has received the NLGI Clarence E. Earle Memorial Award in 1978; the NLGI Fellows Award in 1983; the NLGI Authors Award in 1983 and the NLGI Meritorious Service Award in 1988. He is a member of ANSI, ASTM and SAE.

Introduction: *How can the quality and suitability of automotive service greases be determined and specified?*

How can the auto maker inform his customers as to which greases are satisfactory for vehicle service?

How can the vehicle owner be reasonably confident that the grease used to service his vehicle meets the auto maker's recommendations?

These and similar questions have posed problems for auto makers, grease manufacturers, service station operators, and vehicle owners since the birth of the auto industry.

When the auto industry was young and service lubrication quite frequent, auto makers issued generic recommendations such as: use water-resistant cup grease for chassis or repack wheel bearings with short-fiber grease of medium-firm consistency. Somehow, such indefinite descriptions were adequate for the times; no doubt, because of the short lubrication intervals - as short as once a day or every 50 miles. [1][1]

By the early thirties, service intervals for chassis points had lengthened to as much as 1000 miles, and most auto companies recommended that wheel bearings be lubricated only at long intervals, 5000 to 10 000 miles. [2]

With these long intervals came a growing concern over the proper recommendations for service lubricants, but it was recognized as a complex problem. In 1933, Mougey reported that the SAE had undertaken the development of a grease classification system. [2] He noted:

> *The Society of Automotive Engineers has recognized the problems of chassis-lubricant classification for many years. It has appeared so difficult that until recently no active steps were taken to suggest any classification.* [2]

Evidently in 1933 as before, the SAE found that grease classification was still too difficult since no classification system ever materialized.

By the sixties, the auto makers concern over proper recommendations had intensified. Improvements in both grease and automotive technology permitted even longer extensions of lubrication-service intervals, 6000 to 30 000 miles. To ensure some reasonable probability of servicing with acceptable-quality greases,

1. *Numbers in brackets refer to References at end.*
2. *At that time, the term* chassis lubricant, *had a broader definition than today. It included greases for all chassis application: suspension, wheel bearings, universal joints, steering gears, water pumps, etc.*

the auto makers resorted to recommendations by specification number, by part number, or, in a few instances, by product brand name. Such recommended products were generally followed by "or equivalent." Service station operators could no longer service all vehicles from a stock of one or two greases; now, they needed a half-dozen or more. It is no wonder that auto owners and mechanics were confused. Clearly, this was not a good situation.

Voicing their problems and needs to SAE, the auto makers reiterated those early concerns regarding the lack of an industry-accepted classification of greases and requested the development of a means for describing grease-performance properties in technical language.

So 33 years after the first attempt, SAE again decided to try for a grease classification system. This time, however, they recognized that additional expertise was needed, and they decided to enlist the help of others. In 1966, SAE Fuels and Lubricants Technical Committee 4 requested help from ASTM Committee D-2 Section B-IV (now known as Section D02.BO.04) which has ASTM responsibility for standards related to automotive greases. [3] Although the initial request was directed just to Section B-IV, eventually, the expertise of many other groups was solicited, all of which contributed significantly to the development of ASTM D 4950, Standard Classification and Specification for Automotive Service Greases. Appendix 1 lists these groups, their chairmen, and a brief description of their responsibilities.

Significant Events: In the 23 years it took to develop the classification system and its corresponding specifications, too much has happened to give a full accounting in this paper; only the highlights can be described. (A review of the history, from 1966 to 1982, of the Service Grease Classification System was presented by Lane. [3]) A graphic representation of the significant events in the development of D 4950 is shown in Figure 1.

SAE F&L Technical Committee 4 initiated this sequence of events in 1966 when they requested ASTM D-2 Section B-IV to develop "technical language to define greases" - that is, B-IV should develop the means for describing grease quality in terms of performance in standard tests. At that time, Technical Committee 4 (including the auto company representatives) believed that such technical language would enable auto makers to develop the kinds of recommendations they needed to pass on to their customers. Because they believed that fulfillment of their request would be sufficient, TC 4 stated that they did not want specifications for automotive greases.

Later that same year, SAE TC4 sponsored an Open Forum on the Testing, Evaluation, and Designation of Automotive Lubricating Greases. This started the process of determining what was required to develop technical language. Many smaller meetings were conducted subsequently by ASTM B-IV and its subordinate groups in order to determine the grease-performance requirements, related grease properties, and the means (tests) for their determination. Early on, a B-IV Task Force decided on the important properties of both chassis and wheel bearing greases (Table 1). (Later, B0.04 would revise their estimation of importance of these grease properties, as can be seen in the final requirements listed in Tables 1 and 2 of Appendix 2.)

Eight years after the initial request, the determinations of ASTM D-2 B-IV and its subgroups were used to draft a Technical Language Information Report which was informally submitted to SAE Technical Committee 4 in 1974. Upon review of the draft, SAE determined that their initial request and the resultant ASTM Report were inadequate and what was actually needed was a classification system with corresponding specification, including test methods and limits, for service chassis and wheel bearing greases. Accordingly, a revised request was issued in 1975. Shortly thereafter, the NLGI was invited to participate also.

The following year, NLGI formed a Task Force to develop the user language, and by the end of 1976, they had completed their task of developing the Category Designations and Performance Descriptions. In order to protect the classification system, NLGI registered the designations and descriptions with the U.S. Copyright Office. In 1979, the letter designations for the chassis grease categories were revised to avoid a conflict with a grease-service designation already being used by the Department of Agriculture. Since then, some minor changes have been made; the final version is shown in Section 5 of D 4950, Appendix 2.

While the NLGI Task Force worked on their assignment, word descriptions of the required grease performance (D 4950, Section 4) were being developed by ASTM B-IV. But, it was another eight years before the first draft of the classification and specifications was produced.

Why did it take so long? Simply, a word description of grease performance was just not adequate. In order to completely develop the technical language that would be acceptable to the industry, specifications would be necessary. This meant that test methods to describe grease performance would have to be selected and limits to describe acceptable grease quality would have to be determined.

The specifications could not be completed until the necessary test methods were developed and accepted. For various reasons, some of these, the low-temperature torque test for instance, took a long time (Figure 1). Additional time would be required for the evaluation of production greases so that quality limits could be determined.

Development of Performance Tests: In 1975, when B-IV got its revised assignment, it was determined that several grease performance tests would have to be developed, namely:

1. A test to determine brine sensitivity (noise), torque or frictional stability, and wear protection provided by greases used in ball joints and steering-linkage pivots, [4,5]
2. A test to determine the high-temperature durability or

Table 1

Adapted from Lane. [3]

Important Properties Related to the Performance of Automotive Greases
Preliminary Determination by ASTM B-IV
April 17, 1967

Chassis Greases	Wheel Bearing Greases
A. Functional Properties*	A. Functional Properties**
1. Noise Prevention in Ball Joints	1. Wear Prevention in Ball Bearings
2. Rust Prevention	2. Fretting Corrosion Prevention
3. Seal Compatibility	3. Low-Temperature Torque
4. Torque Stability in Ball Joints	4. Wear Prevention in Roller Bearings
5. Wear Prevention	5. Rust Prevention
	6. Seal Compatibility
B. Other Properties Related to Functional Properties	B. Other Properties Related to Functional Properties
1. Evaporation	1. Evaporation
2. Feedability (flow to pump suction)	2. High-Temperature Properties
3. High-Temperature Properties	3. Mechanical Stability (effect of shear on consistency)
4. Low-Temperature Properties	4. Mobility
5. Mechanical Stability (effect of shear on consistency)	5. Oil Separation
6. Oil Separation	6. Retention (in bearings, particularly on retainers)
7. Pumpability	7. Water Resistance (resistance to washout)
8. Water Resistance (resistance to washout)	8. Water Stability (effect of water on consistency)
9. Water Stability (effect of water on consistency)	
C. Other Properties Not Related to Functional Properties	C. Other Properties Not Related to Functional Properties
1. Compatibility	1. Compatibility
* Over an ambient temperature range of -20°F to 120°F.	** Over an ambient temperature range of -20°F to 300°F.

life of greases used in serviceable wheel bearings, [6,7]

3. A test to determine the tendencies of greases to leak out of wheel bearings operating at high temperature, [7]

4. A test to determine the low-temperature torque performance of wheel bearing greases, [8,9]

5. A test to determine the fretting-wear protection characteristics of chassis greases, [10] and

6. A test to determine the compatibility of chassis and wheel bearing greases with reference elastomers representative of those used in seals. [11,12]

ASTM Subcommittee G, without a specific request from B-IV, had already developed a Test for Brine Sensitivity, Torque Stability, and Wear with Ball Joint Grease; it was approved later that year as D 3428. Also, the development of the High-Temperature Wheel Bearing Grease Life Test (later, D 3527) was nearing completion.

In addition to these two tests, B-IV requested Subcommittee G to develop certain other grease performance tests. (At that time, Subcommittee B had never written a standard test method and believed that Subcommittee G was better able

Table 1

"L" Chassis Grease Categories

Category	Test	Property	Acceptance Limit
LA	D 217	Consistency, worked penetration, mm/10	220-340[A]
	D 566 or D 2265	Dropping Point, °C, min	80
	D 2266	Wear Protection, scar diameter, mm, max	0.9
	D 4289	Elastomer CR Compatibility:	
		volume change, %	0 to 30
		hardness change, Durometer-A points	0 to -10
LB	D 217	Consistency, worked penetration, mm/10	220-340[A]
	D 566 or D 2265	Dropping Point, °C, min	150
	D 2266	Wear Protection, scar diameter, mm, max	0.6
	D 4289	Elastomer CR Compatibility:	
		volume change, %	0 to 30
		hardness change, Durometer-A points	0 to -10
	D 1742	Oil Separation, mass %, max	10
	D 1743	Rust Protection, rating, max	Pass
	D 2596	EP Performance:	
		load wear index, kgf, min	30
		weld point, kgf, min	200
	D 4170	Fretting Protection, mass loss, mg, max	10[B]
	D 4693	Low Temperature Performance,	
		torque at -40°C, N•m, max	15.5

[A] Vehicle manufacturer's requirement may be more restrictive; grease containers should display NLGI Consistency Number as well as category designation.

[B] The fretting wear requirement is significant in passenger car and light-duty truck service, but it has not been shown to be significant in heavy-duty truck applications.

to develop test methods. Coincidentally, Subcommittee G, independent of any interests shared with B-IV, had determined that they would develop grease test methods and would rather not work on specifications.) The performance tests and the time spans required to develop them are depicted in Figure 1.

The long time spans needed to develop these methods were due to attempts to improve a problem common to all of them - precision. However time consuming, the efforts to improve the precision of the methods, by revisions in the test equipment and/or procedures, for the most part, were effective. A notable exception was the Ball Joint Test, D 3428.

It took six years to develop the ball joint test into the standard, D 3428. Shortly thereafter, the manufacturer of the test joint used in D 3428 ceased production of all ball joints. Several substitute ball joints were investigated, but the attempts to find a suitable replacement for the original joint were unsuccessful. A number of revisions in both test equipment and procedures were investigated, but repeatable results could not be obtained with any of these.

In 1985, B-IV was informed that it appeared unlikely that a suitable ball joint test would be available within the next few years. Consequently, it was decided to proceed with the classification and specification without a D 3428 requirement.

Table 2

"G" Wheel Bearing Grease Categories

Category	Test	Property	Acceptance Limit
GA	D 217	Consistency, worked penetration, mm/10	220-340[A]
	D 566 or D 2265	Dropping Point, °C, min	80
	D 4693	Low Temperature Performance, torque at -20°C, N•m, max	15.5
GB	D 217	Consistency, worked penetration, mm/10	220-340[A]
	D 566 or D 2265	Dropping Point, °C, min	175
	D 4693	Low Temperature Performance, torque at -40°C, N•m, max	15.5
	D 1264	Water Resistance at 80°C, %, max	15
	D 1742	Oil Separation, mas %, max	10
	D 1743	Rust Protection, rating, max	Pass
	D 2266	Wear Protection, scar diameter, mm, max	0.9
	D 3527	High Temperature Life, hours, min	40
	D 4289	Elastomer NBR-L Compatibility:	
		volume change, %	-5 to + 30
		hardness change, Durometer-A points	+2 to -15
	D 4290	Leakage Tendencies, g, max	24
GC	D 217	Consistency, worked penetration, mm/10	220-340A
	D 566 or D 2265	Dropping Point, °C, min	220
	D 4693	Low Temperature Performance, torque at -40°C, N•m, max	15.5
	D 1264	Water Resistance at 80°C, %, max	15
	D 1742	Oil Separation, mass %, max	6
	D 1743	Rust Protection, rating, max	Pass
	D 2266	Wear Protection, scar diameter, mm, max	0.9
	D 3527	High Temperature Life, hours, min	80
	D 4289	Elastomer NBR-L Compatibility:	
		volume change, %	-5 to + 30
		hardness change, Durometer-A points	+2 to -15
	D 4290	Leakage Tendencies, g, max	10
	D 2596	EP Performance:	
		load wear index, kgf, min	30
		weld point, kgf, min	200

[A] Vehicle manufacturer's requirement may be more restrictive; grease containers should display NLGI Consistency Number as well as category designation.

At the time, it was thought that the specification could be revised to include a ball joint test requirement when a suitable test was finally developed. Now, however, it appears improbable that such a test will ever be developed. All work on ball joint test development has ceased, and D 3428 was withdrawn as a standard in 1988.

Although one might consider that the deletion of the ball joint test lessens the value of the specification, the need for this test is not as quite as critical as it once was. With respect to brine sensitivity, improved seal designs have greatly reduced the chance of contamination. Use of these seals with today's grease formulations make ball joint noise a rarity. Regarding wear protection, this ball joint grease property is determined by D 2266. However, except for the defunct D 3428, no other means of evaluating torque stability has been investigated. At this point, doing without a torque stability test appears to be an acceptable risk.

Determination of Specification Requirements: Tables 1 and 2 of Appendix 2 list the tests and acceptability limits for the two chassis-grease and three wheel bearing grease categories. B0.04's determinations of the limits for tests that had been in existence for a number of years were agreed upon by concensus with little controversy. On the other hand, some of the limits for the newer, performance-type tests were not determined so easily.

Fortunately, the Subcommittee G Subsections responsible for the development of these methods included in their cooperative test programs many greases having a history of vehicle operation. Among them were greases identified as good, bad, and "minimum acceptable quality," vis-a-vis vehicle performance. These included service station greases, and current and former factory-fill greases. Including such greases gave producer and user alike a reference by which each could evaluate the performance of his grease-product line.[3]

The first significant controversy arose with the determination of the limits for D 3527, High Temperature Life. A particular grease (sample G-827) was conceded to represent minimum acceptable quality for Category GC. The cooperators determined the mean D 3527 life of this grease to be 112 hours, but the individual determinations ranged from 60 to 280 hours.

Based on the mean life, as determined by all the cooperators, the users initially proposed a specification limit of 100 hours. This limit was opposed by some suppliers. Their argument being that, if the specification limit were 100 hours, then the cooperators who obtained less than this value would not be able to obtain passing results with a grease defined as being acceptable. This was a telling argument, but what to do about it? The users did not want a lower limit because this would have the opposite effect of passing greases that were not acceptable. This also was a telling argument, but again what to do about it?

The root of the problem was the poor precision of the D

3. *One could ask: why would a supplier admit to providing a "bad" grease? First, all samples were coded, and generally, the only ones aware of the identification of a particular sample were the grease supplier and the sample coordinator. Second, "bad" meant only with respect to current technology. These greases were satisfactory for earlier times, but had been replaced with more advanced formulations as technology progressed and the performance requirements became more severe.*

Table X2

Guide to requirements for grease categories

TEST	DESCRIPTION	LA	LB	GA	GB	GC
D 217	Penetration	✔	✔	✔	✔	✔
D 566*	Dropping Point	✔	✔	✔	✔	✔
D 1264	Water Washout	—	—	—	✔	✔
D 1742	Oil Separation	—	✔	—	✔	✔
D 1743	Rust Protection	—	✔	—	✔	✔
D 2266	4–Ball Wear	✔	✔	—	✔	✔
D 2596	4–Ball EP	—	✔	—	—	✔
D 3527	High Temperature Life	—	—	—	✔	✔
D 4170	Fretting Wear	—	✔	—	—	—
D 4289	Elastomer Compatibility	✔	✔	—	✔	✔
D 4290	Leakage	—	—	—	✔	✔
D 4693	Low Temperature Torque	—	✔	✔	✔	✔

* D 2265 may be substituted

3527 test method. When the original D-3527 method was developed, the data were found to fit a Weibull distribution. However, when this method was revised (in 1984) in an attempt to improve precision, the cooperators previously had been directed to not analyze the cooperative results using Weibull statistics because of the problems associated with trying to understand Weibull statistics. Instead, they were directed to analyze the data using normal, Gaussian-distribution statistics - which they did, despite the fact that the data were not normally distributed.

In carrying out this directive, the ASTM RR D2-1007 statistical method was followed - including its rigid design-of-experiments - which specifies only duplicate testing. Furthermore, only duplicate tests are allowed for the determination of repeatability (precision determined by one operator). Exacerbating the problem, only single tests are to be used in comparing the results between laboratories (reproducibility.)

This really is not sufficient testing for engineering-type test methods that produce results which are inherently quite variable - such as D 3527. Although justified by the specified statistical analysis of the duplicate test results, the end result is a precision statement that emphasizes the poor precision of the method.

Weibull Analysis Issue: The implications of this decision to avoid Weibull analysis and follow RR D-1007 were not understood at the time. However, the group was stuck with trying to live with an imprecise method and its precision statement. Furthermore, they faced the problem of accounting for the imprecision, not only in the determination of the significance of the test results, but also in the determination of specification limits. The course of action to be taken on this latter problem proved to be a major source of disagreement until a solution was found.

The fundamental difference in the disagreement was due to different views as to how the poor precision of the test method is to be applied to the determination of specification limits.

The suppliers took a pragmatic approach in recognizing that many product buyers interpret specification limits as absolute limits of acceptability and ignore the precision statement in the test methods. However, this should not be the case, according to ASTM D 3244, Utilization of Test Data to Determine Conformance with Specifications. The main purpose of D 3244 is to indicate how test imprecision should be interpreted relative to specification values. It was developed to provide a means of settling disputes over product quality.

With this in mind, the users were concerned about the application of D 3244 to a requirement that was set using test data but included compensation for poor precision. For example, if we account for the poor precision by setting the GC-specification limit at 80 hours (instead of at 100 hours as suggested by the data), in the future, someone unaware of the previous compensation could account for the poor precision by citing and applying D 3244. Such an event, in effect, would make allowance for poor precision twice.

Although D 3244 appeared to be a cause for concern, it also appeared to provide a solution to the problem. This standard states that an acceptance limit is not the same as a

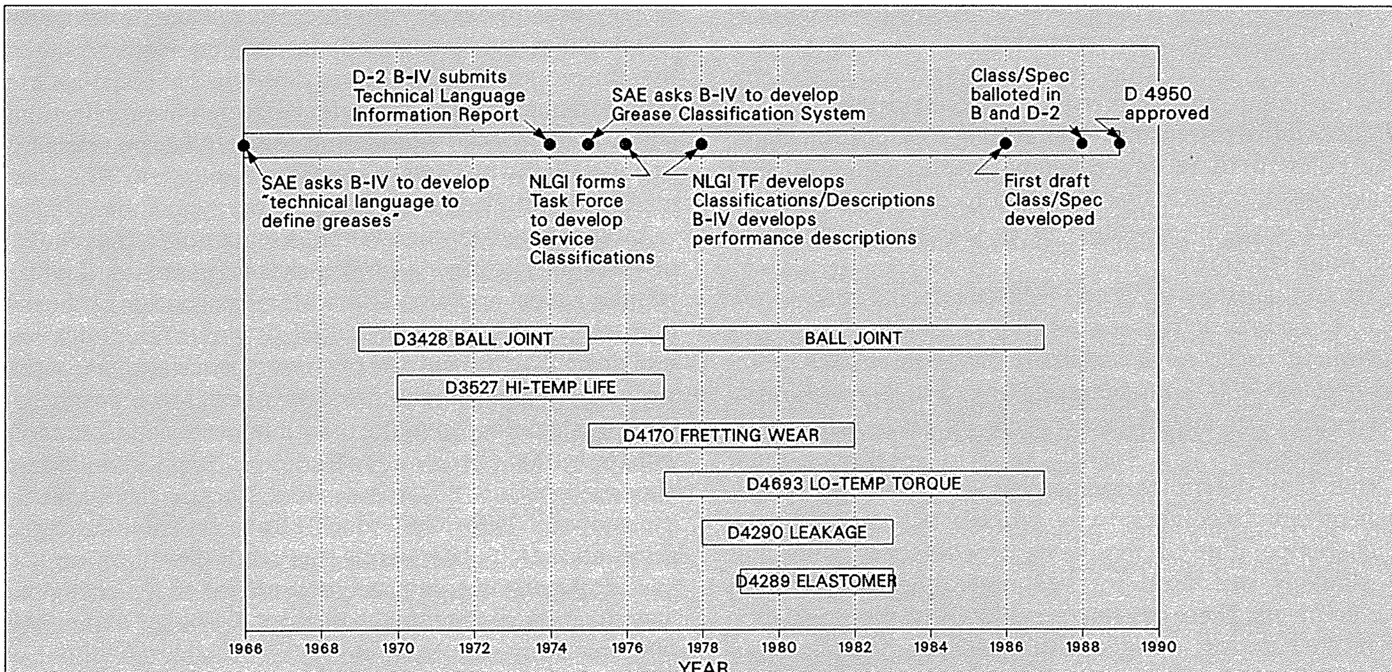


Figure 1. Development History of D 4950, Automotive Service Grease Classification and Specification

specification limit. It goes on to define the acceptance limit as "the numerical value that defines the point between acceptable and unacceptable quality, and takes into account the specification value, the test method precision, and the confidence level for defining minimum acceptable quality relative to the specification value."

What would be the result of this? To determine this, D 3244 was used to calculate the *minimum acceptable value* using an assumed specification value of 100 hours and the mean value for the minimum-acceptable-quality grease as determined in the cooperative tests. By applying D 3244 to the actual test data, the minimum acceptable value was determined to be 77 hours. (The calculation is shown in Appendix 3.) In addition, it was discovered that this determination would reject the same results (with the minimum-acceptable-quality grease) that would be rejected by a supplier-proposed 80-hours specification limit without the application of D 3244. However, if D 3244 were applied to a limit of 80 hours and D 3244 - in effect, twice accounting for the imprecision of the method - the minimum value for acceptance would be 57 hours, and none of the results with the minimum-quality grease would be rejected, not even the statistical outliers.

Other Grease Testing: In addition to the minimum quality GC grease, the cooperators also tested a sub-GC-quality grease. If the specification limit were 100 hours and the minimum acceptable value were 77 hours, only one laboratory (out of 12) would have passed this grease. On the other hand, if the specification limit were set at 80 hours and, in this instance, D 3244 erroneously applied to determine a minimum acceptable value of 57 hours, eight of the 12 laboratories would have determined that the sub-GC grease meets the D 3527-life requirement for GC-quality; whereas, in reality, it is an unacceptable product.

This lengthy description illustrates the potential problems that can be encountered in trying to account for the variability in cooperative test results when setting specification limits. The solution to B0.04's dilemma was to utilize D 3244 equations and the actual test data to determine *minimum acceptable values* as described in D 3244, and include the following statement:

The test requirements (acceptance limits) given in this specification are, as the case may be, minimum or maximum acceptable values for valid duplicate test results. No *additional corrections for test precision, such as described in* D 3244, *are to be applied inasmuch as the precision of the test methods was taken into account in the determination of the requirements.*

This limit-determination process was significant. It not only provided a solution to the problem of determining the High-Temperature Life requirement for the specification, but it similarly was used to determine other specification requirements. These details are provided with the expectation that other groups, in similar situations, may profit from our experience. Also, it may be the first documented description of using D 3244 and actual test data to determine a specification requirement.

Approval of the Standard: The result of all these developments was a document that classifies automotive service grease into two chassis grease categories and three wheel bearing grease categories. Specification requirements were developed for each category. The key requirements for certain performance tests were developed using actual test data and statistical methods.

Following the determination of specification requirements, the document was submitted as a proposed standard for the approval process. The first hurdle outside the section was the Subcommittee B, 1987 Fall Ballot. Five negative votes were received. After some minor editorial changes, three were eventually withdrawn and one changed to abstain. Portions of the fifth negative vote were sustained and the document was referred back to Section B0.04 for additional work. In order to satisfy the intentions of Subcommittee B (and the negative voter), a footnote was deleted from Tables 1 and 2, as Subcommittee B determined that it was an inappropriate qualifying statement.[4]

Some Negative Votes: The document was revised further to include a footnote regarding the fretting wear requirement (appendix 1, Table 1, Footnote A). Appropriately revised, the document was resubmitted for approval in the next Subcommittee B ballot, Spring 1988. It received three negative votes.

One negative voter wanted the category designations changed to correspond with the ISO grease-classification system (actually a possibility foreseen by Lane [3] before the ISO classification even existed). In response, it was explained that the proposed standard was developed in response to a specific request for a system oriented to definite applications - and application - oriented specifications are explicitly excluded in the ISO grease-classification system. The voter reluctantly withdrew his negative vote.

Another negative voter wanted a reduction in the number of categories to one each for chassis grease and wheel bearing grease and also wanted the designations changed from letter codes to a "user-friendly" style. In rebuttal, it was explained that the automotive descriptions encompassed more vehicles than just automobiles and light trucks and that the five categories constitute the minimum needed to comply with the ASTM Subcommittee B scope and the SAE definition of *automotive vehicles*. B0.04 agreed to explore the "user-friendly designations" with NLGI which has responsibility for the designations. (Subsequently, the NLGI Classification and Specifications Subcommittee determined that a change to "user-friendly designations" was not feasible.) The voter withdrew his negative.

4. *The intended purpose of the deleted footnote had been to preclude misapplication of the standard if the tables were construed as complete listings of the requirements and one failed to consider the generic requirements (Appendix 1, Sections 4 and 5). With reluctance, the following footnote was deleted:*
It is the responsibility of the producer of greases meeting these specification limits to also ensure that the greases are suitable for the intended application.

The third negative voter wanted the fretting wear limit increased and the elastomer compatibility requirement deleted. When the method by which the fretting wear limit was determined was fully explained, the voter withdrew that portion of his negative vote.

However, the negative vote, vis-a-vis the elastomer compatibility requirements, was not withdrawn and the voter presented a number of arguments to support his position. (Coincidentally with the ballot in Subcommittee B, Subcommittee G simultaneously was balloting a revision of the Elastomer Compatibility test method; this same voter cast a negative on it as well.) The voter's claims and the rebuttals are summarized in Appendix 4.

After hearing both the support and rebuttal arguments, Subcommittees B and G rejected the negative votes, finding them "not persuasive". The actions of both Subcommittees were supported by Committee D02. (Despite the fact that his negative vote was not sustained, Subcommittee G believed that the voter's concerns were significant, and they decided to pursue appropriate action. As a first step, Subcommittee G subsequently requested the help of ASTM Committee D11 to develop suitable reference elastomers.)

With these impediments successfully negotiated, the document was submitted for approval in the Committee D02, Fall 1988 ballot. It received three negative votes, all based on the previous negative regarding the elastomer compatibility requirement. All three voters withdrew their negatives when informed of both sides of the issue.

In response to comments received on the ballot, several minor editorial changes were made and the document was submitted for ASTM Society Ballot. It was assigned the designation D 4950 and successfully passed the April, 1989 ballot. It will be published in the next issue of *Annual Book of ASTM Standards*.

Summary and Author's Comments: Perceiving a need for means of describing satisfactory greases for chassis and wheel bearing service lubrication, in 1966, the SAE requested ASTM to develop "technical language to define grease." Several years later, this request was revised to: develop a standard classification system with concomitant specifications for chassis and wheel bearing service greases. ASTM Subcommittee B, Section 4 was given this responsibility, but they did not have the expertise to complete their task without help. The NLGI was called upon to develop the designations and performance descriptions of grease categories. ASTM Subcommittee G was given the responsibility for developing several grease performance tests, without which development of the standard could not proceed. Subcommittee B, Section 4 developed the description of the service requirements and the specifications, including the determination of limits. Some specification limits were determined by a process not heretofore described. D 4950, Standard Classification of and Specification for Automotive Service Grease, was approved in 1989.

It has been a long, arduous journey to get to where we are now, but despite the problems and frustrations, perseverance has finally paid off. If one were to ask whether it was really worth it, I would have to give it a qualified yes. Certainly, it is not nearly as beneficial to the industry now as it would have been had it been in place when first proposed some two decades ago. However, despite the growing usage of permanently-grease components, in the foreseeable future, some vehicles requiring maintenance regreasing will still be manufactured. Having a standard available for the recommendation of service greases should make better greases available for these vehicles as well as those earlier vehicles still needing periodic lubrication.

Acknowledgment: So many persons have contributed to the development of a standard that was 23 years in the making that it is not practical to list all of them. Without intention of slighting any contributor by omission, the author nonetheless must single out several key individuals. A special thanks is due to Mr. J.W. Lane for initiating the development of D 4950, for his gentle pressure to get the job done, and for his patience over those sometimes trying years. Thanks, too, to Mr. W.P. Scott and the NLGI Task Force for quickly completing their assignment to develop the user language. We appreciate the efforts of Mr. D.V. Culp in overcoming a certain inertia in B-IV, and those of Messrs. A.W. Gilbert and R.M. Olree for pressing on. Thanks especially to all the chairmen of the numerous groups in Subcommittees B and G who developed the document and the essential test methods. Finally, we express our greatest appreciation for the efforts of all those who, though not specifically mentioned, worked diligently to develop the performance test methods without which D 4950 literally would have been impossible.

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12. J.C. Root, "ASTM D 4289-83, Compatibility of Lubricating Greases with Elastomers," NLGI *Spokesman* 49, 190-97 (August, 1985).

Appendix 1

Organizations Participating in Development of D 4950

<u>SAE</u>
Fuels and Lubricants Technical Committee 4 - *Automotive Greases*
(Chairmen: J.W. Lane, L.W. Okon)
Initiated work on Grease Classification System; responsible for promotion of use of D 4950 within the automotive industry.

<u>NLGI</u>
Literature Subcommittee Task Force on *Classification*
(Chairman: W.P. Scott)
Responsible for development of the "user language" (designations for the grease categories and the corresponding Performance Classification descriptions).

<u>ASTM D-2</u>
B-IV (later, D02.B0.04) - *Automotive Greases*
(Chairmen: C.J. Herbenar, A.T. Polishuk, D.V. Culp, A.W. Gilbert, R.M. Olree)
Responsible for development of ASTM D 4950, Standard for Classification of and Specification for Automotive Service Greases.

B-IV Task Force - *Wheel Bearing and Chassis Grease Performance*
(Chairmen: J.L. Dreher, D.P. Pellerito, C.H. West)
Responsible for determination of which properties relate to performance in automotive chassis and wheel bearing applications, for the evaluation of proprietary grease tests, and for recommendations for the development related performance tests. (Later, these responsibilities were assumed by D02.BO.04)

B-IV-1 - *Classification and Information*
(Chairmen: M. Ehrlich, J.C. Root)
Responsible for development of the classification system and its technical language.

B-IV-2 - *Properties and Performance*
(Chairman: T.M. Verdura)
Responsible for drafting specifications and requirements.

B0.04.01 - *Properties and Performance Panel*
(Chairman: T.M. Verdura)
Merger of B-IV-1 and B-IV-2 with combined responsibilities.

G-II-9 - *Elastomer Compatibility Test*
(Chairman: J.C. Root)
Responsible for development of Test for Compatibility of Greases with Elastomers, D-4289.

G-III-3A (later, G0.05.02) - *Low-Temperature Torque Test*
(Chairmen: T.C. Wilson, J.F. Stadler)
Responsible for development of Low-temperature Wheel Bearing Grease Test, D-4693.

G-III-9 (later, G0.04.01) - *Ball Joint Tests*
(Chairmen: H. Raich, T.M. Verdura)
Responsible for Test for Brine Sensitivity, Torque Stability, and Wear with Ball Joint Greases, D-3428.

G-III-11 (later, G0.05.01) - *High-Temperature Leakage Tests*
(Chairmen: F.S. Sayles, J.A. Keller)
Responsible for development of Wheel Bearing Leakage Test, D-4290. Later, additionally responsible for maintenance of D 3527.

G-III-12 - *Fretting Wear Tests*
(Chairman: T.M. Verdura)
Responsible for development of Fretting Wear Test, D-4170.

G-IV-6 - *High-Temperature Life Test*
Low-Temperature Torque Test
(Chairman: D.J. Sargent)
Responsible for development of High-Temperature Wheel Bearing Grease Life Test, D-3528. (After development of standard, this responsibility transferred to G-III-11.) Responsible for selection of low-temperature torque test; after selection, responsibility for development of standard transferred to G-III-3A.

Appendix 2

The following is the final version of the proposed standard which was submitted for the April, 1989 ASTM Society Ballot and approved on mm/dd/yy. It is scheduled for publication in the 1990 edition of Annual Book of ASTM Standards, Volume 05.03.

ASTM Designation D 4950

Standard Classification and Specification for
AUTOMOTIVE SERVICE GREASES[1,2]

Introduction: This specification describes current categories of lubricating greases for automotive service-fill applications. A specific designation is assigned to each category. The system is open ended, that is, new designations are assigned for use with new categories as each new set of grease performance characteristics is defined. Grease categories are referenced by automotive manufacturers in making lubrication recommendations, and used by grease suppliers and users in identifying products for specific applications.

1. Scope

1.1 This specification covers lubricating greases suitable for the periodic relubrication of chassis systems and wheel bearings of passenger cars, trucks, and other vehicles.

1.2 This specification defines the requirements used to describe the properties and performance characteristics of chassis greases and wheel bearing greases for service-fill applications.

1.3 The test requirements (acceptance limits) given in this specification are, as the case may be, minimum or maximum acceptable values for valid duplicate test results. No additional corrections for test precision, such as described in D 3244, are to be applied inasmuch as the precision of the test methods was taken into account in the determination of the requirements.

1.4 The values stated in SI units are to be regarded as the standard; values in US-conventional units are for information only.

2. Referenced Documents

2.1 ASTM Standards:

D 217 Test Method for Cone Penetration of Lubricating Grease[3]

D 566 Test Method for Dropping Point of Lubricating Grease[3]

D 1264 Test Method for Water Washout Characteristics of Lubricating Greases[3]

D1742 Test Method for Oil Separation from Lubricating Grease During Storage[3]

D1743 Test Method for Corrosion Preventive Properties of Lubricating Greases[3]

D 2265 Test Method for Dropping Point of Lubricating Grease over Wide Temperature Range[4]

D 2266 Test Method for Wear Preventive Characteristics of Lubricating Grease (Four-Ball Method)[4]

D 2596 Method for Measurement of Extreme-Pressure Properties of Lubricating Grease (Four-Ball Method)[4]

D 3527 Test Method for Life Performance of Automotive Wheel Bearing Grease[4]

D 4170 Test Method for Fretting Wear Protection by Lubricating Grease[5]

D 4289 Method of Testing Compatibility of Lubricating Grease with Elastomers[5]

D 4290 Test Method for Determining the Leakage Tendencies of Automotive Wheel Bearing Grease Under Accelerated Conditions[5]

D 4693 Test Method for Low-Temperature Torque of Grease-Lubricated Wheel Bearings[5]

3. Terminology

3.1 Descriptions of Terms Specific to This Standard

3.1.1 **automotive service grease** - a lubricating grease suitable for the periodic relubrication of serviceable-type, chassis components or wheel bearings of passenger cars, trucks, and other vehicles and distinct from factory-fill greases (also known as initial-fill and OEM greases) initially installed by the original equipment manufacturer.

3.1.2 **chassis grease** - an automotive service grease used to lubricate ball joints, steering pivots, universal joints, and, other lubrication points designated in the vehicle owner's service guide.

3.1.3 **classification** - with respect to automotive service grease, the systematic arrangement into categories according to differing levels of performance.

3.1.4 **category** - with respect to automotive service grease, a designation, such as LB, GC, etc., for a given level of performance in standardized tests.

3.1.5 **"L" category group** - automotive service greases of such composition, properties, and performance characteristics as to be suitable for the service lubrication of those types of suspension, steering, and drive-line components that require periodic relubrication.

3.1.6 **"G" category group** - automotive service greases of such composition, properties, and performance characteristics as to be suitable for the service lubrication of those types of wheel bearings that require periodic relubrication.

3.1.7 **multipurpose grease** - an automotive service grease suitable for both chassis and wheel bearing lubrication.

Discussion: Commercial lubricating greases other than automotive service greases are often designated as multipurpose greases.

3.2 Abbreviations:

ASTM - American Society for Testing and Materials
NLGI - National Lubricating Grease Institute
SAE - Society of Automotive Engineers
max - maximum
min - minimum

4. Performance Classification[6]

4.1 Automotive service greases are classified into two general groups. Those designated with an "L" prefix (chassis greases) are intended for the service lubrication of ball joints, steering pivots, universal joints, and other chassis components as designated by the equipment manufacturer. Those designated with a "G" prefix are intended primarily for the service lubrication of wheel bearings. These groups are further subdivided into categories with intended service applications as follows:

4.1.1 LA - Service typical of chassis components and universal joints in passenger cars, trucks, and other vehicles under mild duty only. Mild duty will be encountered in vehicles operated with frequent relubrication in non-critical applications.

4.1.2 LB - Service typical of chassis components and universal joints in passenger cars, trucks, and other vehicles under mild to severe duty. Severe duty will be encountered in vehicles operated under conditions which may include prolonged relubrication intervals, or high loads, severe vibration, exposure to water or other contaminants, etc.

4.1.3 GA - Service typical of wheel bearings operating in passenger cars, trucks, and other vehicles under mild duty. Mild duty will be encountered in vehicles operated with frequent relubrication in noncritical applications.

4.1.4 GB - Service typical of wheel bearings operating in passenger cars, trucks, and other vehicles under mild to moderate duty. Moderate duty will be encountered in most vehicles operated under normal urban, highway, and off-highway service.

4.1.5 GC - Service typical of wheel bearings operating in passenger cars, trucks, and other vehicles under mild to severe duty. Severe duty will be encountered in certain vehicles operated under conditions resulting in high bearing temperatures. This includes vehicles operated under frequent stop-and-go service (buses, taxis, urban police cars, etc), or under severe braking service (trailer towing, heavy loading, mountain driving, etc.)

5. Performance Description [7]

5.1 The performance characteristics of the several categories of automotive service greases are described as follows:

5.1.1 LA - The grease shall satisfactorily lubricate chassis components and universal joints where frequent relubrication is practiced (at intervals of 3200 km or 2000 miles or less for passenger cars). During its service life, the grease should resist oxidation and consistency degradation and protect the chassis components and universal joints from corrosion and wear under lightly loaded conditions. NLGI No. 2 consistency greases are commonly recommended, but other grades may also be recommended. (NLGI Consistency Numbers are shown in Table X1 of the Appendix.)

5.1.2 LB - The grease shall satisfactorily lubricate chassis components and universal joints at temperatures as low as -40°C (-40°F) and at temperatures as high as 120°C (248°F) over prolonged relubrication intervals (more than 3200 km or 2000 miles for passenger cars). During its service life, the grease should resist oxidation and consistency degradation while protecting the chassis components and universal joints from corrosion and wear even when aqueous contamination and heavily loaded conditions occur. NLGI No. 2 consistency greases are commonly recommended, but other grades may also be recommended.

5.1.3 GA - The grease shall satisfactorily lubricate wheel bearings over a limited temperature range. Many products of this type are limited to bearing temperatures of -20°C to 70°C (-4°F to 158°F). No additional performance requirements are specified for these greases.

5.1.4 GB - The grease shall satisfactorily lubricate wheel bearings over a wide temperature range. The bearing temperatures may range down to -40°C (-40°F), with frequent excursions to 120°C (248°F) and occasional excursions to 160°C (320°F). During its service life, the grease shall resist oxidation, evaporation, and consistency degradation while protecting the bearings from corrosion and wear. NLGI No. 2 consistency greases are commonly recommended, but NLGI No. 1 or No. 3 grades may also be recommended.

5.1.5 GC - The grease shall satisfactorily lubricate wheel bearings over a wide temperature range. The bearing temperatures may range down to -40°C (-40°F), with frequent excursions to 160°C (320°F) and occasional excursions to 200°C (392°F). During its service life, the grease shall resist oxidation, evaporation, and consistency degradation while protecting the bearings from corrosion and wear. NLGI No. 2 consistency greases are commonly recommended, but NLGI No. 1 or No. 3 grades may also be recommended.

6. Performance Requirements [7]

6.1 The greases identified by these categories shall conform to the requirements listed in Tables 1 and 2. A guide to the requirements of all the grease categories is given in Table X2 of the Appendix.

6.2 The consistency requirements in Tables 1 and 2 cover NLGI Consistency Numbers 1 through 3 (see Table X1). However, because the equipment manufacturers recommendations may be more restrictive, it is recommended that grease containers display the consistency number as well as the grease category designation.

6.3 Some grease makers market products under the term "multipurpose grease," implying or stating that such products are suitable for both chassis and wheel bearing lubrication. To comply with this specification, greases intended and suitable for both chassis and wheel bearing lubrication may carry such designation but, in addition, shall carry both an "L" and "G" designation (LB-GC, for example) and conform to the appropriate requirements listed in Tables 1 and 2.

continued . . .

Appendix
(Nonmandatory Information)

X1. Supplementary Information on Properties

X1.1 The National Lubricating Grease Institute has classified greases according to their consistency as measured by the worked penetration (D 217) at 25°C. The classification is as follows:

X1.2 Table X2 is a guide to the requirements for the grease categories; it is meant to provide a quick comparison of the properties defined for each category. Refer to Tables 1 and 2 for the actual values of the requirements.

X2. Classification Maintenance

X2.1 The automotive service grease classification is designed to keep abreast of changing requirements by redefining existing, or adding new categories. To expeditiously accomplish such action, close coordination among the ASTM, NLGI, and SAE is required. Although it is neither possible nor desirable to develop rigid operating rules, the following is a summary of the recommended guidelines described in detail in SAE J1146.[8]

X2.1.1 Any individual, company, or society can request changes in, or additions to, the grease categories.

X2.1.2 SAE, with cooperation from ASTM and NLGI, considers whether the request is consistent with the overall classification objectives.

X2.1.3 SAE, with concurrence of ASTM and NLGI, either accepts or rejects the request.

X2.1.4 If the proposal is accepted by SAE, it is referred to ASTM for selection and standardization of test techniques and development of performance criteria, and it is referred to NLGI for development of user language.

X2.1.5 ASTM, NLGI, and SAE are kept informed of progress by liaison membership in the task groups developing the proposal. Each society completes its part of the development, documents it, and solicits comments from the other societies. When the societies are in agreement, each publishes the results of its program.[9]

X3. NLGI Symbol

X3.1 The NLGI is considering the development of a symbol that could be used on containers of greases that conform to the requirements of one or more categories listed in Tables 1 and 2. Additional information can be obtained from the National Lubricating Grease Institute, 4635 Wyandotte Street, Kansas City, MO 64112.

Footnotes

[1] This method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.B on Automotive Lubricants.

Current edition approved M/D/Y. Published M/D/Y.

[2] This specification was developed as a cooperative effort among the American Society for Testing and Materials (ASTM), the National Lubricating Grease Institute (NLGI), and the Society of Automotive Engineers (SAE).

[3] Annual Book of ASTM Standards, Vol 05.01.

[4] Annual Book of ASTM Standards, Vol 05.02.

[5] Annual Book of ASTM Standards, Vol 05.03.

[6] The letter designations for the grease categories and the corresponding Performance Classification descriptions in Section 4 were developed by an ad hoc panel of the NLGI Literature Subcommittee in cooperation with ASTM D02.B0.04.02, (Subsection on) Automotive Grease Specifications. Although these designations and descriptions of the categories have been adopted in toto in this standard, the NLGI Literature Subcommittee retains jurisdiction over them as published in, "Chassis and Wheel Bearing Service Classification System," available from the National Lubricating Grease Institute, 4635 Wyandotte Street, Kansas City, MO 64112. It is the intention of Subcommittee B to include in this standard future revisions to these descriptions providing they are deemed acceptable by ASTM.

[7] The Performance Descriptions and Performance Requirements for the grease categories, as described in Sections 5 and 6, were developed by ASTM D02.BO.04.02 in cooperation with the NLGI Literature Subcommittee. ASTM Subcommittee B retains jurisdiction over these descriptions (cf footnote 6).

[8] "SAE J1146 Automotive Fuel and Lubricant Performance and Service Classification Maintenance Procedure," SAE *Handbook*, Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

[9] In addition to each society maintaining and publishing its respective portion of the Automotive Service Grease Classification System (which includes this specification), the NLGI Letter Designations and Classification Description, and the essentials of D 4950, Standard Specification for Automotive Service Greases, is reprinted in "SAE J310 Automotive Lubricating Greases," (SAE *Handbook*, Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096) in order to receive widespread dissemination among the automotive industry.

Appendix 3

Determination of GC-Quality Acceptability Limit For D 4950 High-Temperature Life Requirement

Using ASTM D 3244, eq 2*

$$AL = S+[0.255 \times (2/N)^{0.5} \times R \times D]$$

Where:

AL = Acceptability limit

S = Specification limit

N= Number of laboratory results

R= Reproducibility of method (for D 3527, R=1.2xM, where M = average of two test results)

D= Deviation of AL from S for product acceptance at a given probability. For a non-critical specification, at a 95% probability, D = -1.645

Assuming:

Grease G-827 represents a minimum-GC-quality grease

A non-critical specification

A specification value (S) of 100 hours

A mean value (M) of 112 hours (round-robin mean with G-827

$$AL = 100 + [0.255 \times (2/12)^{0.5} \times 1.2 \times 112 \times (-1.645)]$$
$$AL = 100 - [0.255 \times 0.408 \times 1.2 \times 112 \times 1.645]$$
$$AL = 100 - 23$$
$$AL = 77$$

*D 3244 should be consulted for definitions of terms.

Appendix 4

Negative Votes on Elastomer Compatibility Test and Requirements

The arguments supporting the negative votes in Subcommittees B and G and the rebuttals are summarized as follows:

Argument: The Reference Elastomers are 40-year old formulations, not representative of current formulations. Test data were shown to support this position.

Rebuttal: This is immaterial, as it is their performance in the test, and not the age of the recipe, that is the proper criterion for suitability; test data were presented in support of this position. Furthermore, there are no other reference elastomers of these types.

Argument: Reference CR is not compatible with many EP greases; test data in support of this position were presented.

Rebuttal: "That is precisely why we need the test." The fact that the incompatible greases happened to be EP greases was coincidental. Four of five EP greases tested in the round robin passed.

Argument: Some of the elastomer additives are no longer available and are carcinogenic.

Rebuttal: It is true that some ingredients are no longer items of commerce; however, because of retained stocks of additives, the reference compound still can be made according to specification. The fact that one of the additives may be suspect carcinogen is immaterial to users of the elastomer compatibility test, as they do not come in contact with additives, and the reference elastomers themselves are not considered carcinogenic.

Argument: There is excessive batch variability. During their presentation showing performance differences between two batches, they admitted that one of the batches - the one they had made themselves - was not formulated precisely to specification.

Rebuttal: Even when made in compliance with the reference elastomer specification, batch variability is greater than desired, but that happens to be a characteristic of rubber technology just as grease-batch variability is characteristic of grease technology.

Argument: Subcommittee G is aware of these problems but is not acting on them.

Rebuttal: Not entirely true; attempts to find other reference materials and suppliers were unsuccessful.

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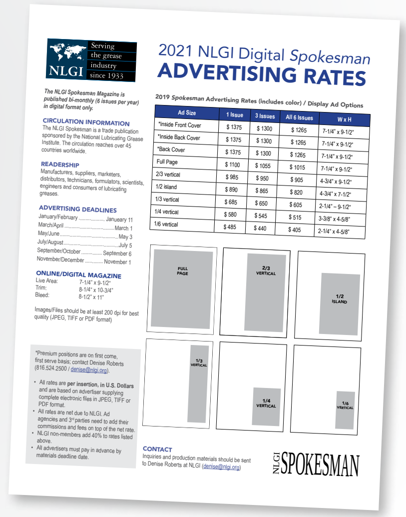
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