

NLGI SPOKESMAN

Serving the Grease Industry Since 1933 - VOL. 86, NO. 1, MARCH/APRIL 2022

In this issue:...

- 14 The Impact of Viscosity of Naphthenic Oils and Extreme-Pressure Additives on Lithium-Based Lubricating Greases
- 28 An Innovative Preformed Thickener for the Preparation of Structurally Stable and Tribologically Effective Polyurea Greases
- 37 Education Committee Update
- 39 The Lithium Crisis for the Grease Industry
- 47 Electric Compatability of Grease with Elastomeric Seals Retrospective
- 58 Sustainability Survey
- 61 High-Performance Multiuse (HPM) Grease Column





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NLGI SPOKESMAN

Serving the Grease Industry Since 1933 - VOL. 86, NO. 1, MARCH/APRIL 2022

- 4** **President's Podium**
Jim Hunt, NLGI President
- 6** **Industry Calendar of Events**
Welcome New NLGI Members | Advertiser's Index
- 8** **NLGI Annual Meeting**
- 14** **The Impact of Viscosity of Naphthenic Oils and Extreme-Pressure Additives on Lithium-Based Lubricating Greases**
Mehdi Fathi-Najafi, Ameneh Schneider and Jinxia Li
- 28** **An Innovative Preformed Thickener for the Preparation of Structurally Stable and Tribologically Effective Polyurea Greases**
Liwen Wei, Carl F. Kernizan, and Noura Smaili
Novitas Chem Solutions, LLC
- 37** **Education Committee Update**
- 39** **The Lithium Crisis for the Grease Industry**
- 47** **Electric Compatability of Grease with Elastomeric Seals Retrospective**
- 58** **Sustainability Survey**
- 61** **High-Performance Multiuse (HPM) Grease Column**
- 63** **In Memoriam - Dr. Huafeng "Bill" Shen**
- 64** **In Memoriam - Arnold C. Witte, Jr.**



ON THE COVER
Happy Spring!

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PRESIDENT'S PODIUM

Dear NLGI Family,

As always, I hope that you and your family are healthy and happy. We are all moving full speed ahead into 2022 and it is proving to be an interesting year indeed. The bright spot is that it appears we are quickly departing from the dreaded COVID phase that has crippled our world. Although there are a few countries that may still be impacted, we should be long on our way to getting our lives back to normal. Let's all take a few moments to remember and honor our loved ones that may have survived this pandemic. We also need to feel blessed for those of us that managed to survive this deadly pandemic.

Jim Hunt
NLGI President
2020 – 2022



As life gets better, new challenges are occurring. We are now experiencing one of the worst inflation periods in some of our lifetimes. The increased cost on normal goods and lack of supply is hitting every family. No one is immune from the fall out of these issues. We remain very optimistic that 2022 would move us into a more positive direction and we must all keep the faith, that will be the case. We should all take a few moments to provide our blessings and prayers for the families that have been devastated in Ukraine. We are all one big global family that truly should be there for each other in times of need.

NLGI has some very excited news to share with our valued members. The 89th Annual Meeting is off and running. The 89th Annual Meeting will be held at the Westin Harbour Castle in beautiful downtown Toronto, Canada June 12-15, 2022. Early bird registration launched at the end of February and was a big success. In fact, we had a record number of early attendees register! Please plan to join us this year for what we expect this to be one of our best annual meetings ever. There will be numerous opportunities to network with your friends and business associates. The annual meeting will also offer dynamic presentations focusing on a variety of industry topics. We will continue to provide stellar educational classes instructed by some of the most knowledgeable professionals in the grease industry. If you have not signed up for the meeting, we highly recommend that you do so soon. Once you register, you will receive a confirmation e-mail with information on how to secure your hotel room. For more information on the agenda, technical presentations, optional events and more, please visit the [NLGI website](#). Looking forward to seeing you all there and getting the band back together.

As part of our ongoing commitment to provide updates on the High Performance Multi-use Grease (HPM) certification program, we are honored and pleased to inform you that there are currently six greases that have received the HPM grease certification. Of the six certifications, four include enhanced performance tag and one product that is dual certified with HPM and GC-LB. The greases that have achieved HPM certification are from some of the global industry leaders including, BP Lubricants, Shell, Exxon Mobil, Valvoline and Molykote. These global grease leaders have recognized the value that the HPM certification provides to their organization and customers. NLGI appreciates their commitment to achieve this milestone and we want to congratulate all of you. There are additional greases in the process of being tested to obtain the HPM certification. Licensed HPM brands undergo a rigorous qualification testing to meet or exceed the HPM grease specification requirements. NLGI and CQA anticipate a considerable increase in HPM grease certifications in immediate future and for many years to come. If you have are interested in submitting your grease for HPM or need assistance, please feel free to contact the NLGI office and/or Mike Kunselman at Center for Quality Assurance. For more information including specification details, pricing and more, please visit the [NLGI website](#).

Once again, as this year begins to fly by, NLGI wants to remind you to consider becoming a NLGI committee volunteer. We welcome everyone. For a list of committees and how to sign up, please visit the [NLGI website](#).

In closing, we want to wish all of you and your families the very best life has to offer. Hope to see all of you at the NLGI AM in June.

Jim Hunt
NLGI President



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Industry Calendar of Events 2022

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

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



Warm Welcome to our New NLGI Member

Barentz North America, LLC *Supplier* USA

Advertiser's Index

ProSys Servo Filling Systems, page **35**

STLE, page **7**

Valvoline, page **13**

Vanderbilt Chemicals, LLC, **Inside Front Cover**

Zschimmer & Schwarz Inc., page **5**

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76th STLE Annual Meeting & Exhibition May 15-19, 2022



Walt Disney World Swan & Dolphin Resort
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- 12 Lubrication-specific Education Courses
- 80-exhibitor Trade Show
- Commercial Marketing Forum
- Business Networking
- International Audience

Register now!

Log on to www.stle.org to register and make your room reservation at the Walt Disney World Swan & Dolphin Resort.

Early Birds! Register by April 12 and save \$115 on your meeting fee.

2022 TECHNICAL TRACKS

- 2D Materials + Superlubricity - Materials Tribology and Nanotribology Joint Session
- Biotribology
- Biotribology at the Nanoscale - Biotribology and Nanotribology Joint Session
- Commercial Marketing Forum (*purchased time slots*)
- Condition Monitoring
- Contact Mechanics
- Electric Vehicles, Engine, Drivetrain
- Environmentally Friendly Fluids
- Fluid Film Bearings
- Gears
- Grease
- Lubrication Fundamentals
- Materials Tribology
- Metalworking Fluids
- Nanotribology
- Nonferrous Metals
- Power Generation
- Rolling Element Bearings
- Seals
- Surface Engineering
- Synthetic Lubricants and Hydraulics
- Tribochemistry - Materials Tribology and Nanotribology Joint Session
- Tribology of Biomaterials - Biotribology and Materials Tribology Joint Session
- Tribotesting
- Wear
- Wind Turbine Tribology



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NLGI 89TH ANNUAL MEETING Finding The Green In Grease

JUNE 12-15, 2022

Westin Harbour Castle | Toronto, ON Canada

[Agenda](#)

[Price List](#)

[Hotel / General Info](#)

[COVID-19 Info /
FAQs](#)

[Exhibitors &
Sponsors](#)

[Industry Speaker](#)

[Educational
Courses](#)

[Optional Events](#)

[Register HERE](#)

[Attendee List](#)

[2022 Award
Information](#)

[Technical Sessions](#)

Registration now open!
[REGISTER HERE](#)

Cancellations received in NLGI's office through May 6, 2022, will received a 100% refund.
Beginning May 7, 2022, cancellation will only receive a 50% refund.





NLGI 89TH ANNUAL MEETING

Finding The Green In Grease

JUNE 12-15, 2022
Westin Harbour Castle
Toronto, ON Canada

SUNDAY - June 12, 2022

6:30am - 1:30pm	Golf Tournament	Offsite - Lionshead Golf Course
8am - 5pm	Basic Education Course Day 1	Marine
8am - 5pm	Advanced Education Course Day 1	Queens Quay
12pm - 2 pm	Exhibit setup	Metropolitan Ballroom
2pm - 5pm	Registration/Exhibit Open	Metropolitan Ballroom
2:30pm - 4:30pm	BOD Meeting	Dockside 3
4:45pm - 5:45pm	New Member/First Timer Reception	<i>By Invitation Only</i>
5:45pm - 6:45pm	Welcome Reception	5th Floor Roof Top Terrace

MONDAY - June 13, 2022

7am - 5pm	Registration Open	Metropolitan Ballroom
7am - 8am	Networking Breakfast (dedicated exhibitor time)	Metropolitan Ballroom
8:15am - 8:45am	Opening General Session (annual business meeting)	Frontenac Ballroom
8:45am - 9:55am	Industry Speaker	Frontenac Ballroom
10:00am - 5:00pm	Basic Education Course Day 2	Marine
10:00am - 5:00pm	Advanced Education Course Day 2	Queens Quay
10am - 10:30am	Break	Metropolitan Ballroom
10:30am - 11:55am	Tech Sessions	Frontenac Ballroom
11:55am - 12:55pm	Networking Lunch (dedicated exhibitor time)	Metropolitan Ballroom
1pm - 1:45pm	Awards Ceremony	Frontenac Ballroom
1:45pm - 2pm	Break	Metropolitan Ballroom
2pm - 3:30pm	Tech Sessions	Frontenac Ballroom
3pm - 3:30pm	PM Break	Metropolitan Ballroom
3:30pm - 4:45pm	Bio-Based Working Group Meeting	Frontenac Ballroom
5pm - 6pm	Exhibitor Happy Hour (dedicated exhibitor time)	Metropolitan Ballroom

TUESDAY - June 14, 2022

6:15am - 8am	Fun Run	Main Lobby
7am - 5pm	Registration Open	Metropolitan Ballroom
7am - 8:30am	Networking Breakfast (dedicated exhibitor time)	Metropolitan Ballroom
8:15am - 12:10pm	Tech Sessions	Frontenac Ballroom
10am - 12:30pm	Spouse / Guest Activity	TBD
12:10pm - 1:15pm	Networking Lunch (dedicated exhibitor time)	Metropolitan Ballroom
1:15pm - 2:30pm	Grease Particle Working Group Meeting	Frontenac Ballroom
2pm - 4pm	CLGS Exam	Dockside 9
2:30pm - 2:45pm	Break	Metropolitan Ballroom
2:45pm - 5pm	Tech Sessions	Frontenac Ballroom
3:15pm - 3:30pm	Break	Metropolitan Ballroom

WEDNESDAY - June 15, 2022

7am - 1pm	Registration Open	Metropolitan Ballroom
7am - 8:30am	Networking Breakfast (dedicated exhibitor time)	Metropolitan Ballroom
7am - 8:30am	BOD Meeting	Dockside 3
8:30am - 9:45am	Sustainability Town Hall Meeting	Frontenac Ballroom
9:45am - 10:00am	Break	Metropolitan Ballroom
10am - 12:10pm	Tech Sessions	Frontenac Ballroom
12:10pm - 1:15pm	Networking Lunch (dedicated exhibitor time)	Metropolitan Ballroom
1:30pm - 3:00pm	Exhibit Teardown	Metropolitan Ballroom
1:30pm - 3:45pm	Tech Sessions	Frontenac Ballroom
3:45pm - 4:00pm	Break	Metropolitan Ballroom
4pm - 5:15pm	Food Grade Working Group Meeting	Frontenac Ballroom
6pm - 9pm	Closing Party	TBD

COVID-19 FAQs

*Information provided as of March 21, 2022



NLGI 89TH ANNUAL MEETING
Finding The Green In Grease

- 1. You must use ArriveCAN to provide the mandatory travel information before and after your entry into Canada.**
 - This can be downloaded via Apple Store or Google Play. The link below gives you specific step by step instructions of what is required for you to prepare and upload to the app prior to your travel to Canada. <https://www.canada.ca/en/public-health/services/diseases/coronavirus-disease-covid-19/arrivecan.html>
 - Attendees should plan to bring their passport and proof of vaccination.

- 2. What COVID-19 requirements are there to enter Canada?**

As of March 2022, those entering Canada must:

 1. be fully vaccinated, meaning:
 - have received at least 2 doses of a vaccine accepted for travel, a mix of 2 accepted vaccines
 - or at least 1 dose of the Janssen/Johnson & Johnson vaccine
 - have received your second dose at least 15 calendar days before you enter Canada
 2. Prior to April 1st. must provide proof of an accepted type of
 - rapid antigen test taken the day prior to the schedule flight (home tests not accepted) OR
 - molecular test result (PCR, NAT, NAAT, RT-LAMP) within 72 hours of the initial scheduled departure time of the flight to Canada.
 - Please click [here](#) to search requirements for entry into Canada, as each country may be different.
 - Click [here](#) for COVID-19 travel / testing requirements.

- 3. What if I have a connecting flight?**
 - the test must be taken within 72 hours of the scheduled departure time of your final flight that lands in Canada
 - you may need to schedule the test in your transit city

- 4. What if my flight is cancelled?**

If your flight is cancelled by the airline, there are no extensions to the 72-hour limit for your pre-entry test. If your new scheduled flight is not within the 72-hour period, you'll need to be retested.

- 5. What if my flight is delayed?**

If your flight is delayed by the airline, your negative COVID-19 molecular test can be used for up to an additional 24 hours (to a maximum of 96 hours) from the scheduled departure time. If the delay causes your test to be more than 96 hours old, you'll need to be retested.

- 6. What do I do if my test results are negative/positive?**
 - If negative: provide proof of a negative result taken within 72 hours of your scheduled flight or land entry
 - If positive: you no longer have symptoms and provide proof of a previous positive test result taken at least 10 calendar days and no more than 180 calendar days before entering Canada. Counting starts the day following the day of testing.

- 7. Will I be subject to random testing?**

Upon your entry to Canada by air or at a land border crossing, the border services officer may notify you that you have been randomly selected for a mandatory arrival test. Fully vaccinated travelers are not exempt from mandatory randomized arrival testing. If selected for random testing, you no longer have to quarantine as you await your results.

8. How does COVID testing work in order for me to travel home?

Please check your local country's government website as each country's requirements may be different. Click [HERE](#) for information on returning to the United States.

9. How do I obtain a Visa to attend the meeting?

Please click [here](#) to search requirements for entry into Canada, as each country may be different. Please contact NLGI HQ (nlgi@nlgi.org) if a visa letter is needed.

10. What safety protocols will NLGI have in place during the meeting?

NLGI will require each attendee to complete a COVID screening form (and potentially an updated waiver if regulations have changed since registration launched in February 2022). Anyone experiencing symptoms will not be allowed to attend the meeting until their symptoms subside. Additionally, NLGI will have hand sanitizing stations throughout the meeting space, provide masks for attendees, offer colored sticker options for badges to identify attendee's comfort level with contact, designate separate entrance and exit doors and provide social distancing as much as possible.

11. What are current policies at Toronto airports?

For all passengers and employees, regardless of vaccination status: To help reduce the spread of COVID-19, all passengers and airport employees must wear a mask at all times. This includes the pre- and post-security screening areas of the terminals, parking facilities, inter-terminal train, sidewalks/curbs outside the terminals and other outdoor public areas. Please note when sitting at a dining establishment while not eating or drinking, you must still wear your mask at all times.

12. What local mandates or regulations are in place for the Annual Meeting?

Patrons no longer have to show proof of vaccination to enter hotel / restaurants / bars. However, in Toronto, masks are required to be worn in indoor public spaces. A mask must cover the nose, mouth and chin without gapping. Face shields and neck gaiters are not alternatives to a mask.

All annual meeting participants will be asked to complete a COVID-19 health screening waiver before picking up their badge.

13. What is the current COVID-19 situation in Toronto? (cases, hospitalization, etc.)

You can view the City of Toronto COVID-19 Dashboard here:

<https://www.toronto.ca/home/covid-19/covid-19-pandemic-data/covid-19-monitoring-dashboard-data/>

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The Impact of Viscosity of Naphthenic Oils and Extreme-Pressure Additives on Lithium-Based Lubricating Greases

Mehdi Fathi-Najafi¹, Ameneh Schneider² and Jinxia Li¹

¹Nynas AB, SE-149 82 Nynäshamn/Sweden

²Optimol Instruments Prüftechnik GmbH, Munich/Germany

Abstract:

The behavior of lubricating grease in different tribological contacts has been discussed intensively for many decades. It is also known that there are a vast number of material related parameters that may have significant impact on the tribological behavior of grease, such as thickener type and amount, viscosity and type of the oil, type, and concentration of additives. The above parameters are directly related to the cost of the formulation, however there are additional parameters that are affecting the operational cost and conditions such as the contact pressure and the operating temperature.

Hence, ideally it is preferred to have fewer types of test equipment that can be used for the development and optimization of new grease formulations.

The aim of this study is to investigate, step by step, how the formulation of lithium and lithium complex greases may be optimized using tribological tests.

The thickeners chosen were lithium and lithium complex since more than 75 percent of global grease production is based on these two thickener systems. Furthermore, three straight cuts of mineral base oils with various viscosities; (150, 375 and 600 mm²/s) and two additive packages: I) an additive package that consists of anti-oxidant, anti-wear and extreme pressure components and II) additive package I + an extra dosage of the same extreme pressure additive used in package I. All the greases were produced in a laboratory pilot plant at atmospheric pressure.

Besides the characterisation of the greases according to the state of the art, the tribological properties of all formulated greases were studied by using the new generation tribometer, SRV@5. In the first stage, the tribological tests were run with respect to various ASTM methods such as D5706 B, D5707, D 2266 and D2596.

The outcome suggests that by running different tribological tests under conditions very close to relevant application fields, the formulation of the grease can be optimised further resulting in a more cost-efficient solution.

1. Introduction

Lubricating greases can be used in a wide range of applications and conditions. Various lubricating grease types, depending on the type and amount of thickener, additives and viscosities of the base oils provide the main parameters for complexity.

Complexity in the tribology of greases has been discussed throughout the last decades in which the effect of thickeners on the tribological performances has been the core of number of publications e.g., [1], [2]. The film formation of greases in rolling/sliding can be influenced by a number of the parameters such as temperature and roughness of material. However, in a tribological context, it is much more complicated than that.

The aim of this work was to evaluate the impact of the characteristics of the base oils, thickener type (conventional lithium and lithium complex) and additive concentration tribologically by using various SRV® methods as well as four-ball methods.

2. Experimental work

The experimental work that was carried out in this study can be divided into the following parts: characterization of the selected base oils, manufacturing, and characterization of the lubricating greases as well as tribological evaluation of the lubricating greases.

2.1 The base oils

Three hydrotreated naphthenic base oils (BO 150, BO 400, and BO 600) were chosen for this study.

Table 1 Typical characteristics of the naphthenic oils

Characteristics	Unit	Method / ASTM	BO 150	BO 400	BO 600
Density @ 15°C	kg/dm ³	D4052	0.919	0.923	0.932
Viscosity @ 40°C	mm ² /s	D445	150	375	600
Viscosity @ 100°C	mm ² /s	D445	10.5	20.0	21.5
Viscosity Index		D 2270	15	45	-12
Flash Point, PM	°C	D93	222	246	250
Pour Point	°C	D97	-24	-15	-12
Aniline Point (AP)	°C	D611	86	98	89
Copper Corrosion	rating	D130	1	1	1
Sulfur Content	wt. %	D2622	0.130	0.120	0.300
Color	rating	D1500	<2.5	<2.5	<2.5
Total Acid Number	mgKOH/g	D974	<0.01	<0.01	<0.01

Some comments regarding the selected base oils shown in Table 1;

- a) Typically, for the choice of base oils in multipurpose greases, a very important factor is viscosity, with higher viscosity being used for heavy loaded and low speed applications. Hence, theoretically speaking, BO 600 followed by BO 400 should be most suitable products for this kind of application compared to BO 150.
- b) These three naphthenic oils were all wax free and subsequently the viscosity is the dominating parameter that controls to the pour point.

2.2 The greases

In total, six greases (A, B, C, D, E and F) were produced in a pilot plant at atmospheric pressure. Grease A, B and C are conventional lithium (Li), and Grease D, E and F are lithium complex greases (Li X).

The acids used were 12-hydroxystearate acid (12-HSA) and azelaic acid. In addition, two additive packages were used.

Package I consisted of anti-oxidant (1 wt.%), anti-wear (0.50 wt.%) and extreme pressure (1.5 wt.%) components and

Package II consisted of Package I + an extra dosage (2.0 wt. %) of the same extreme pressure agent.

Notable that the additives that are used in this study are ashless, high sulfur content, and low-level of active sulfur.

Table 2 Some of the characteristics of the unadditivated greases

Properties	Grease A	Grease B	Grease C	Grease D	Grease E	Grease F
Base Oil (naphthenic)	BO 150	BO 400	BO 600	BO 150	BO 400	BO 600
Thickener Type	Li	Li	Li	Li X	Li X	Li X
Thickener Content (wt. %)	7.45	5.02	4.57	10.55	9.70	8.55
Dropping Point (°C)	197.0	200.3	197.5	> 280	>280	> 280
Pen. after 60 str. (mm ⁻¹)	258	274	276	265	281	269
Diff after 10 ⁵ strokes (mm ⁻¹)	+42	+26	+30	+40	+41	+50
Cu-Corrosion (rating)	1b	1b	1b	1b	1b	1b
Flow Pressure@-20°C (mbar)	320	370	645	345	320	720

A review of Table 2 suggests:

- Low thickener content for all greases, however, BO 600 results in the lowest thickener content followed by BO 400. This can be attributed to a combination of better solvency and the higher kinematic viscosity of BO 600.
- Dropping point was measured according to IP396. Dropping points for the lithium and lithium complex greases fulfil e.g., the NLGI grease guide.
- The shear stability of the greases after 100,000 strokes was measured according to ASTM D217 which exhibited relatively consistent results for both lithium and lithium complex greases despite the low thickener contents that were used. , it is notable that the repeatability of the worked penetration test is seven units.
- Pumpability of the lubricating greases can be simulated by different methods e.g., measurement of the flow pressure according to DIN 51805. Parameters such as consistency of the grease, polymer content, kinematic viscosity of the oil, pour point as well as the degree of the wax content in the base oil are the main parameters that can affect the mobility of the greases. In this study, we eliminated parameters such as wax content and polymers so the main parameters that can affect the low temperature mobility of the greases are the thickener content, the viscosity and the pour point of the oils. Hence, good low temperature mobility for all greases at -20 (°C) was noted. Nevertheless, the higher pressure needed for the BO 600 based greases should be attributed to the higher pour point.

2.3 Tribological study

The tribological study is based on four different methods; two oscillatory tests by using SRV instrument; ASTM D5707 (friction coefficient and wear test) and ASTM D7506B (step load test), and two rotational tests by using four-ball machine; wear scar test (ASTM D2266) and weld load test (ASTM D2596). The standard methods and the evaluation of lubricating greases on the SRV oscillation tribometer is described in literature. [1]

2.3.1 SRV Apparatus & Measurements

Modern tribometric test systems like SRV® (Schwing Reib Verschleiß) provide both suitable tools and test methods to get a deeper understanding of tribological and lubricity behavior of the greases. In a previous study, some of the tribological aspects of unadditivated lithium and unadditivated lithium complex greases based on two of the oils that are used in this study (BO 400 and BO 600) were determined by using SRV®. [2] However, the frame of this work has been extended further by focusing on the additivated greases as well.

The focus of the investigation in this study was on the following issues:

- 1) What will be the response of the additive package in various types of greases?
- 2) Can lower soap content (the greases contain different soap content but same consistency) create better opportunity for the additives to perform (since soap typically competes with additives on the available positions on the metal surfaces)?
- 3) Is the additive package going to perform equally on various grease formulations?

The following standards were achieved by SRV® for the investigation of different tribological performances of greases for decades:

- 1) Friction and wear performance - ISO 19291 / ASTM D5707
- 2) Load-carrying capacity (EP load) - ISO 19291 / ASTM D5706B

Wear values were estimated according to ASTM D5705 “Standard Practice for Determining the Wear Volume on Standard Test Pieces Used by High-Frequency, Linear-Oscillation (SRV®) Test Machine”. Figure 1 shows the oscillation test chamber.



Figure 1 SRV® oscillation chamber

Test specimen for SRV®:

Lower specimen:

Disk 24 x 7.9 (mm), both surfaces multi-directional lapped, according to DIN 51834, surface roughness 0.50-0.65 ($\mu\text{m Rz}$), material: 100Cr6, hardened

Upper specimen:

Steel ball 10 (mm), polished, DIN 51834, material: 100Cr6

The greases will be applied onto the surface of the disc, using a special grease caliper, so the quantity of the grease is fixed for all tests.

2.3.2 Test matrix for the tribological investigation

The first two standard methods (determination of friction and wear properties of lubricating greases) have been run for all tests. In Table 3 and 4 the most important test parameters of the standard tests are summarised, and in Table 5 the complete test matrix for all SRV® tests can be found.

Table 3 Test parameters SRV® According to ASTM D5707

Parameters	Determination of friction and wear properties of lubricating grease
Temperature (°C)	80
Stroke (mm)	1.0
Frequency (Hz)	50
Load during running-in (N)	50
Running-in Time (s)	30
Normal Force (N)	200
Test Time (min)	120

Table 4 Test parameters SRV® According to ASTM D5706B

Parameters	Determination of extreme pressure properties of lubricating grease
Temperature (°C)	80 & 120
Stroke (mm)	1.5
Frequency (Hz)	50
Load during running-in (N)	50, 100
Running-in Time (min)	0.5 to 15
Normal Force	Increasing stepwise by 100 (N)
Test Time (min)	Max 55

Table 5 Test matrix for the SRV® tests

Greases	ASTM D5707 @ 80 °C	ASTM D5706B @ 80 °C	ASTM D5706B @ 120 °C
Grease A*, B*, C*, D*, E*, F*	X	X	X
Grease A, B, C, D, E, F,	X	X	X
Grease C**, F**	X	X	X

In Table 5, * represents greases with additive package I, and ** represents greases with additive package II.

3. Results of friction and wear measurements

The following part of the study was conducted using the SRV rig according to ASTM D5707 under the following test parameters:

- Temperature: 80 (°C)
- Load: 200 (N)
- Stroke: 1 (mm)
- Frequency: 50 (Hz)
- Time: 2 (hrs.)

With 200 (N) normal load the calculated Hertzian pressures are:

Mean contact pressure $P_m = 1.84$ (Gpa) and Maximum contact pressure $P_{max} = 2.76$ (Gpa)

Figure 2 shows exemplary a COF (coefficient of friction) progression for Grease F.

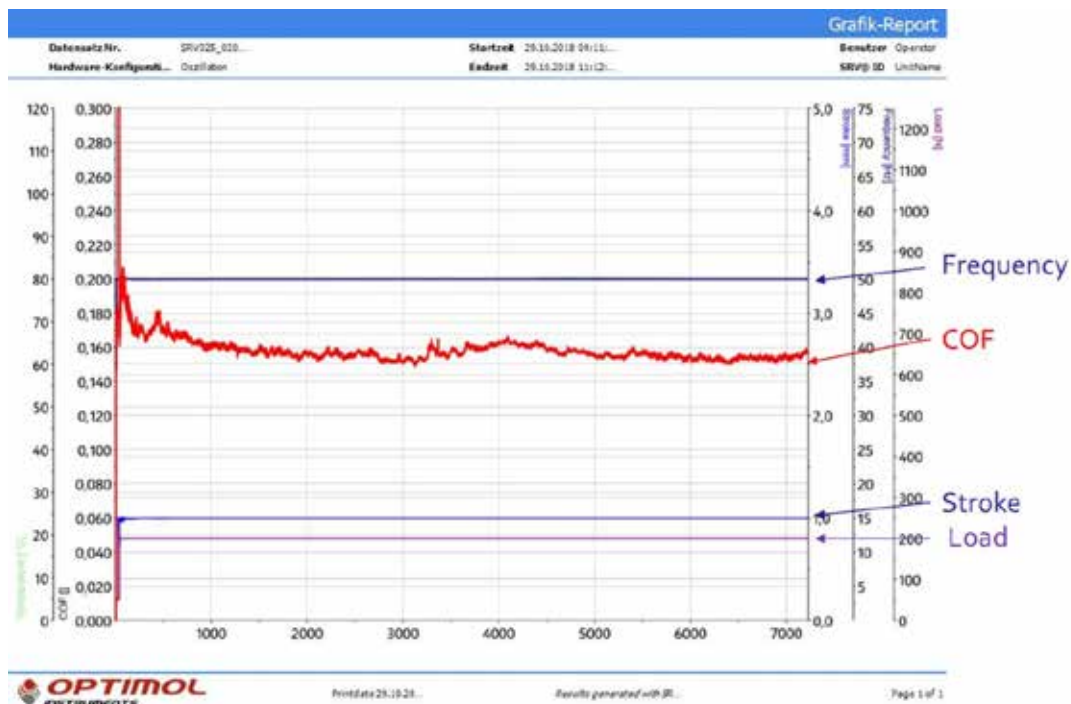


Figure 2 demonstrates the behavior of the friction coefficient of Grease F

Figure 3 illustrates the wear scar on the disk and ball at the end of the test for Grease F made by a microscope. The measured wear values according to ASTM D7755 can be conducted by tip stylus profilometer or laser scanning microscope.

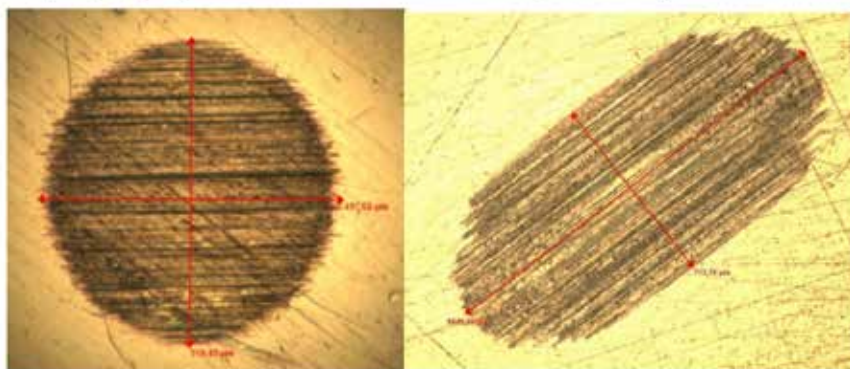


Figure 3 Wear scar on the ball (left), wear scar on the disk (right)

After measuring the diameter of wear scar on the ball as well as the width and length of the wear scar on the disk (Figure 3), the planimetric perpendicular surface area (Figure 4) and using the formula written in standard ASTM D7755, the wear volume can be calculated.

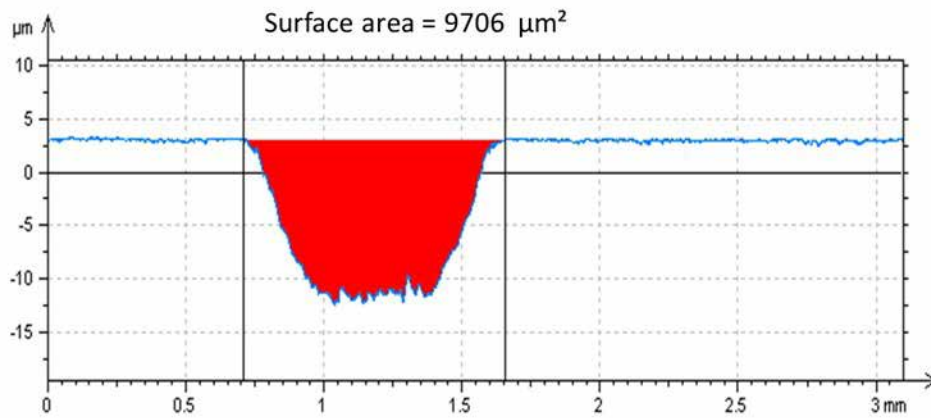


Figure 4 Profilogram displayed on a screen and taken perpendicularly in the center of the wear track on an SRV® test disk

It should be noted that the higher values of COF do not always result in an increased wear volume value. This is more pronounced in case of greases where many phenomena such as structure or depletion of thickener molecules, characteristic of the base oil, etc. contribute to the complexity of the tribo-system.

To interpret the measured data for the six greases, the following aspects were considered:

- A) Impact of viscosity at the applied temperature
- B) Impact of the thickener system
- C) Impact of the concentration of the additives (only for BO 600 based greases; Grease C and F)

3.1 SRV tests on the greases based on BO 150

Figure 5 shows the COF values of four different formulated greases based on BO 150 (Grease A and Grease D).

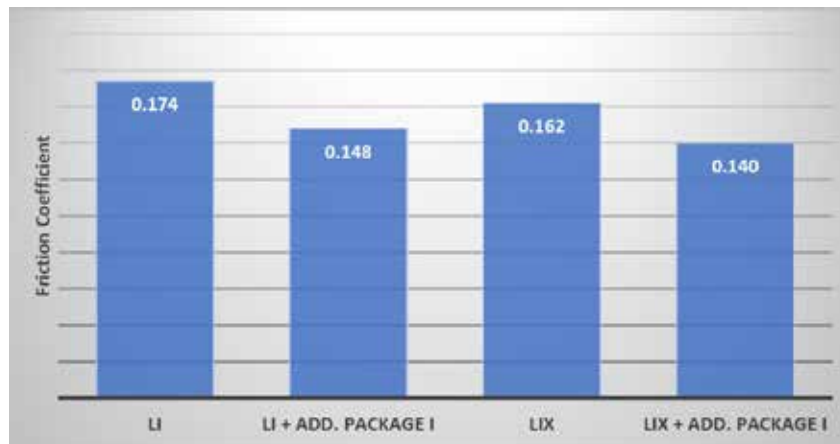


Figure 5 Friction of coefficient for the greases based on BO 150

Figure 5 suggests that **a)** the friction coefficient was lower for the additivated greases when comparing different thickener systems (lithium vs lithium complex), and **b)** the lithium complex grease ran at a lower COF than the lithium grease. The CoFs indicate that boundary lubrication exists and that tribochemistry is more important than rheology.

Furthermore, when considering the wear volume on the disk and the ball and finally the total wear, the differences between the formulations were significant, Figure 6.

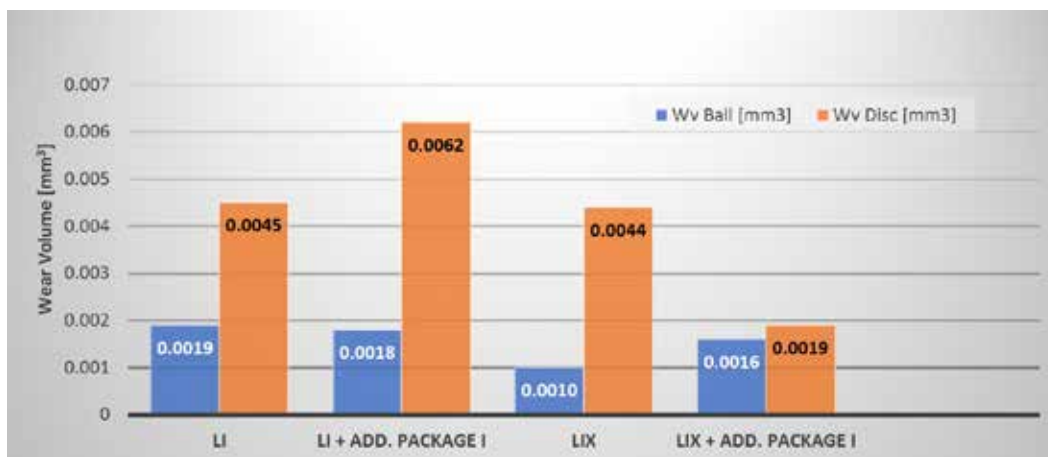


Figure 6 Wear volumes of different formulated greases based on BO 150

Figure 6 suggests that in the case of additivated lithium Grease, the wear scar volume diameter remained unchanged, but the wear volume on disc showed a significant increase which cannot be explained. Hence, a separated investigation is required in order to find a possible reason.

3.2 SRV tests on the greases based on BO 400

Figure 7 presents the COF results for four different formulated greases based on BO 400 (Grease B and Grease E).

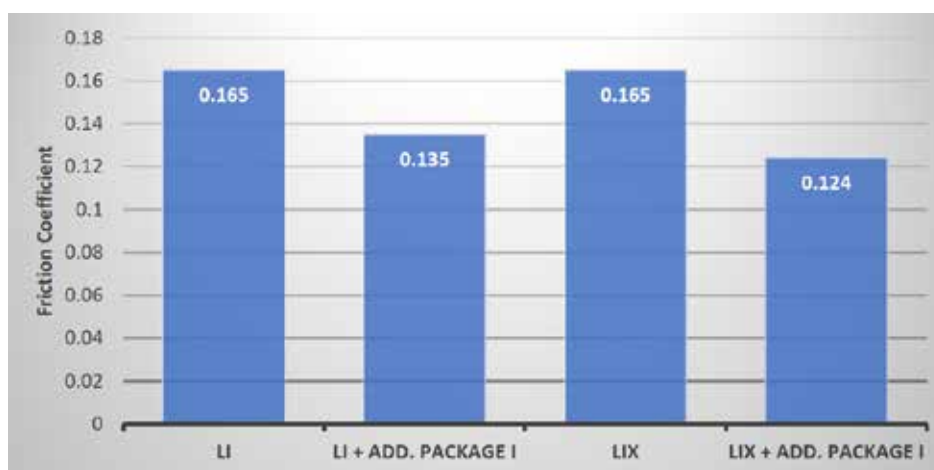


Figure 7 Friction of coefficient for the greases based on BO 400

Figure 7 illustrates the positive effect of the additive on COF for both thickener systems. Comparing the wear volumes, the results were not the same as for BO 150. Regarding lithium complex as thickener the additive reduced the wear volume especially on disk and, consequently, the total wear volume was lower. In this case the additive was, most probably, able to build-up a supporting layer on the disk.

Figure 8 shows significantly higher wear volume on disk for the lithium thickener, even though this test was repeated twice. Hence, it is speculated that the differences in polarity between the additive molecules and this thickener may, at least to some extent, cause some antagonistic effects.

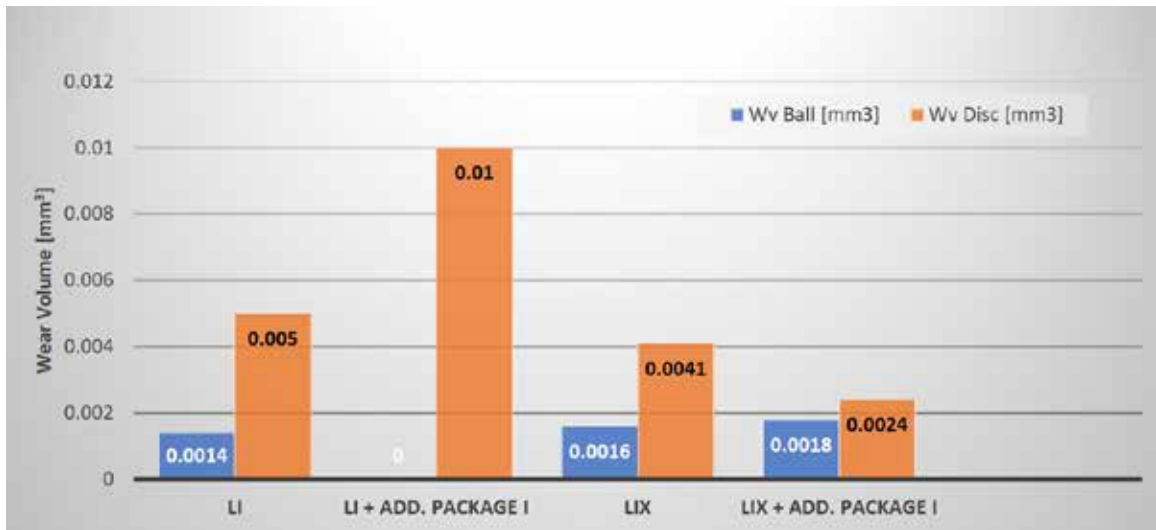


Figure 8 Wear volumes of different formulated greases based on BO 400

3.3 Tribological tests on the greases based on BO 600

In Figure 9, the friction coefficients for the six greases based on BO 600 are presented.

A positive effect of the additive package I on COF values was obtained for both thickener systems (lithium and lithium complex) in line with the previously reported data related to greases based on BO 150 and BO 400. However, the new finding was that the increased concentration of the extreme-pressure additive (Additive package II) had no further effect on greases with either thickener system which led us to the conclusion that more additive does not necessarily result in better performance, at least in this type of tribological contact.

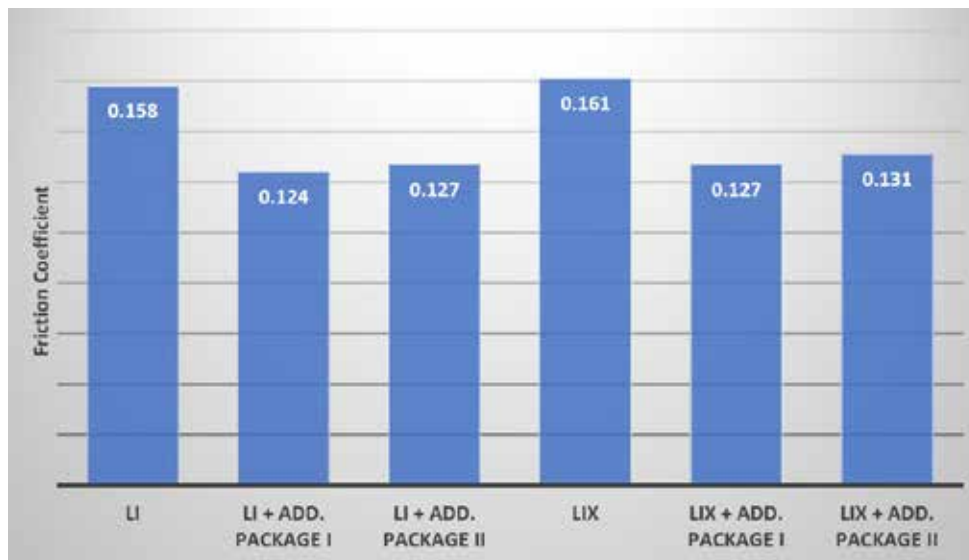


Figure 9 Friction of coefficient for the greases based on BO 600

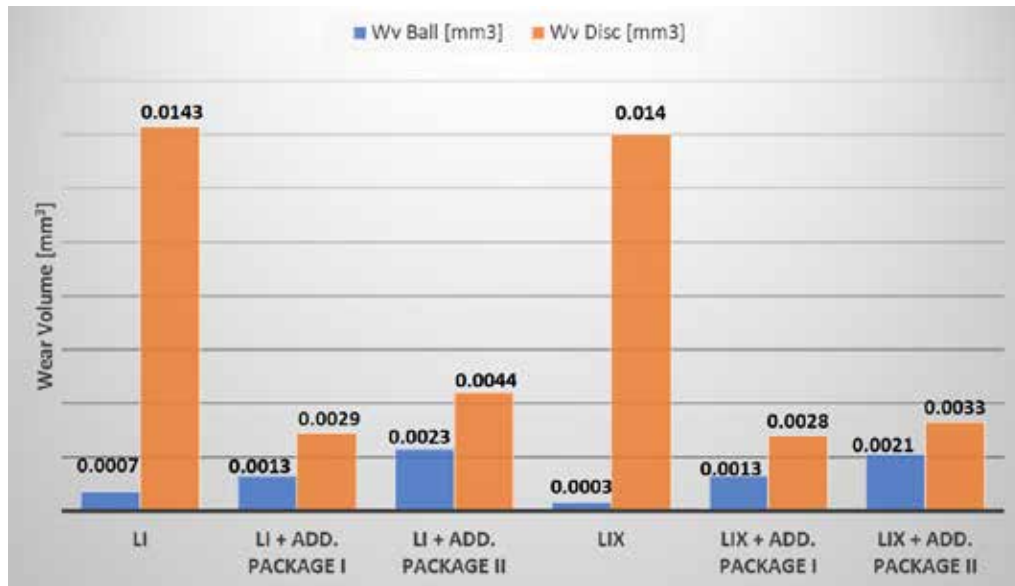


Figure 10 Wear volumes of different greases based on BO 600

In Figure 10, it is interesting that for both thickener systems the extra dosage of extreme-pressure additive (Additive package II) had obviously a negative impact on this tribological system. One possible explanation could be that active sulfur content in the extreme pressure additive interferes with the anti-wear additive when the dosage is increased, or that the activation energies of the anti-wear and EP additives are different.

Comparison of the tribological behavior of the two thickener systems recorded by SRV

Within the frame of our SRV measurements, it was found that the wear volumes for additivated greases did not follow expectations. For example, Figure 11 which shows the wear values of three non-additivated greases with different base oil viscosity and the corresponding ones with lithium and lithium complex thickener confirmed that the greases based on highest base oil viscosity (BO 600) contributed to highest total wear volume while the wear on the ball remained the lowest.

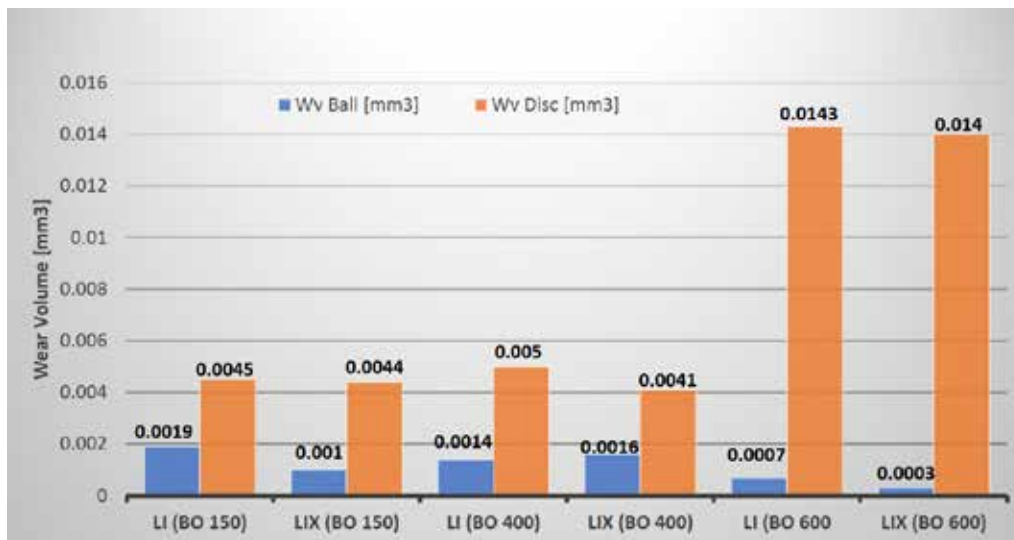


Figure 11 The Wear Volumes for non-additivated greases.

In order to get a better understanding of the achieved results, the tests were run with the base oils (without additive), Figure 12 compares the COF values of the three base oils.

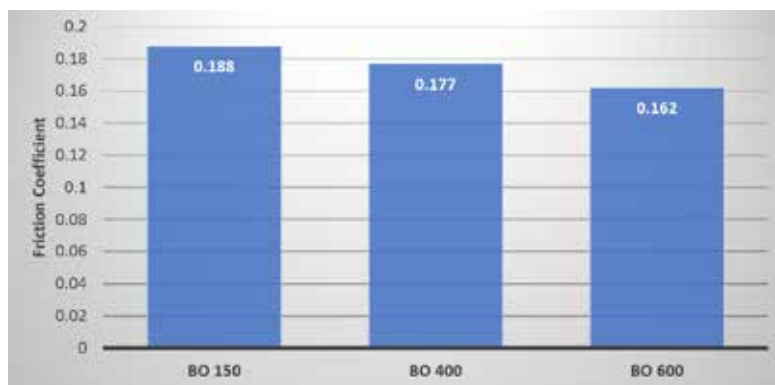


Figure 12 Friction of coefficient of the three base oils

Figure 12 suggests that there might be a relationship between the viscosity of the oil and the friction of coefficient, the higher viscosity the lower COF. This trend can also be found on the wear scar diameter on the ball at least for the most viscous oil (BO 600), Figure 13. However, in the same figure it is obvious that the wear volume on the disk moves in the opposite direction which is more pronounced for the BO 600.

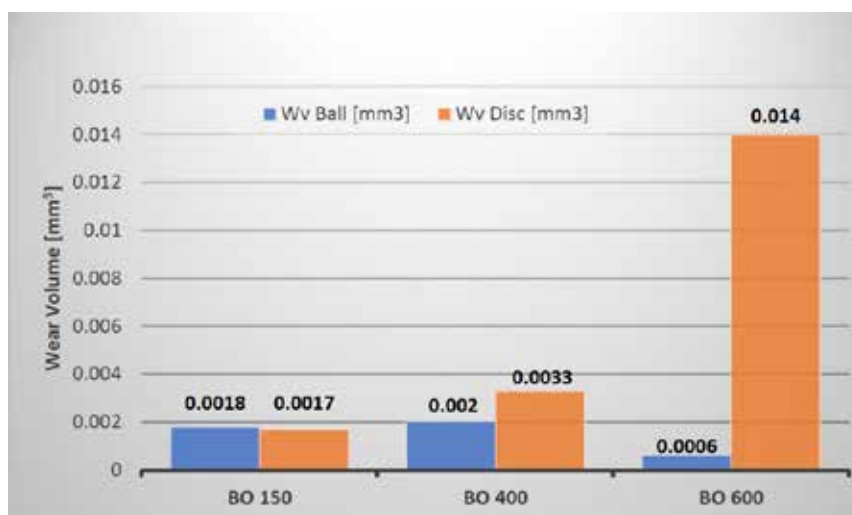


Figure 13 The Wear volumes for the three base oils

3.4 SRV test according to standard ASTM D5706 B

The load carrying capacities of additivated greases using the SRV® standard method ASTM D5706 B were determined at 80 (°C) as described in the procedure. The results of these tests do not show any significant differences. By increasing the test temperature to 120 °C, we can differentiate between the greases and these results are shown and discussed here. It is assumed that at higher temperatures, the extreme pressure additive adheres more easily to the metal surface and is subsequently activated resulting in the formation of a supporting film on the contact point as expected.

The test parameters were as follows:

- Temperature: 120 (°C)
- Stroke: 1.5 (mm)
- Frequency: 50 (Hz)
- Time: max. 55 (min)
- Load: 50 (N) / 30 (s) ; 100 (N) / 15 (min)
- Load step: 100 (N) every 2 (min)

Figure 14 demonstrates exemplarily the progression of the values during this test for one of the greases. This grease achieved a maximal load of 1200 (N).

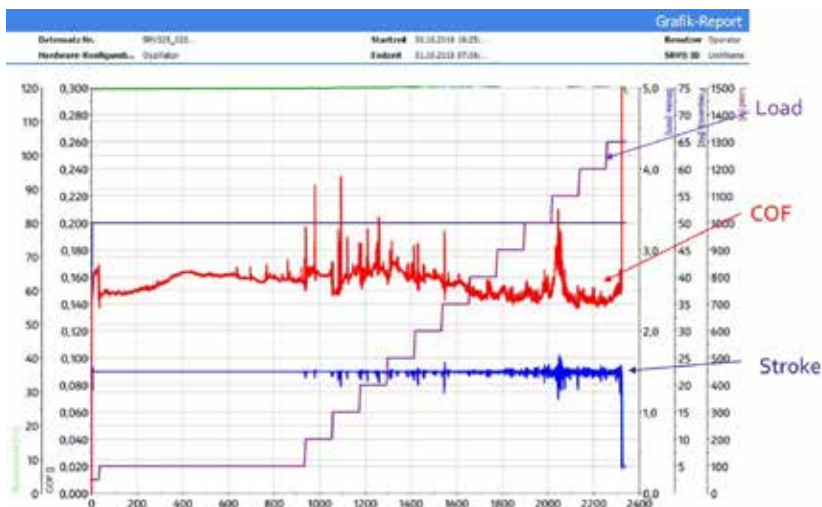


Figure 14 Standard test ASTM D7506B for Grease E + additive package I

The cutoff criteria (the moment that seizure occurred) is defined in this standard ASTM D5706 B; COF > 0.2 for 20 (s).

Table 6 Results of load carrying capacity tests measured by SRV®5 at 120 (°C)

Remarks	Max load carrying capacity (N)
Grease A + Additive Package I	1400
Grease B + Additive Package I	1200
Grease C + Additive Package I	1200
Grease D + Additive Package I	1200
Grease E + Additive Package I	1000
Grease F + Additive Package I	1000
Grease C + Additive Package II	1300
Grease F + Additive Package II	1000

Based on the results obtained from this test, there was a tendency that the greases with lower viscosity and subsequently higher thickener content, showed a better response to additive package I. Greases C and F, with the higher concentration of the extreme pressure agent (additive package II), did not show any increase in the load carrying capacity as expected.

3. 5 Four-ball tests according to ASTM D 2266 and ASTM D2596

Beside linear oscillating movement of ball on disk with SRV®, four-ball tests according to ASTM D2266 (wear scar test) and ASTM D2596 (weld load test) were conducted on all greases. It is inviable to highlight the major differences between the four ball tests and SRV tests; for example, in the case of four-ball tests we have point contact and rotational condition while in the case of the SRV tests, used in this study, ball on plain surface and oscillatory movement.

The obtained results are summarised in Table 7.

Table 7 Results of load carrying capacity and wear tests measured by four-ball machine.

Sample No.	Specifications	Wear scar (mm) ASTM D2266	Weld load (kg f) ASTM D2596
1	Grease A	1.58	160
2	Grease A + Additive Package I	0.53	620
3	Grease B + Additive Package I	0.63	620
4	Grease C + Additive Package I	0.69	620
5	Grease C + Additive Package II	0.52	800
6	Grease D	1.51	200
7	Grease D + Additive Package I	0.85	800
8	Grease E + Additive Package I	0.65	800
9	Grease F + Additive Package I	0.54	800
10	Grease F + Additive Package II	0.56	>800

Table 7 revealed a number of interesting results such as:

- a) **Comparing two unadditivated lithium greases** (Sample # 1 & 6); an increase of the weld load by 25 percent probably has to be attributed to the viscosity of the base oil (BO 600). The difference in wear scar diameters for the two greases was within the repeatability of the test. Furthermore, the significantly lower thickener content in Grease D had no negative impact on the wear scar or on the weld load.
- b) **The additive package I** performs differently in lithium and lithium complex greases;
 - i. In lithium complex greases, significantly higher weld load was obtained.
 - ii. In the case of wear scar diameter, the additive package performed worst in lithium complex grease based on BO 150 (Sample # 7) and best in lithium grease based on the same base oil (Sample # 2), an increase by almost 38 percent.
- c) **The impact of additive package II** in lithium and lithium complex greases based on BO 600 (Sample # 5 & 10); in the case of lithium greases (sample # 4 & 5), significantly higher load and lower wear scar diameter were measured which was in line with the expectations, however this was not found in the case of lithium complex greases (sample # 9 and 10). Regardless of the reason behind this, it can be concluded that a higher concentration of the extreme pressure additive, used in package II, contributes to a more expensive formulation. This finding verifies the earlier observation that was found by SRV measurements, Table 5.

4 Summary

Within the frame of this study, three lithium and three lithium complex greases were produced based on various naphthenic base oils. The use of naphthenic base oils kept the thickener content low, however, a direct correlation between the viscosity of these naphthenic base oils and the thickener content was found: the higher the viscosity, the lower the thickener content.

The tribological investigations proved to be crucial when optimising the grease formulation for specific tribological contacts. However, the most interesting differences between the two different thickener systems were observed in additivated greases;

- a) the additivated lithium greases based on BO 150 and BO 400 showed higher wear values than the non-additivated greases. While same package reduced significantly the wear values of disk when used in lithium complex greases, Figure 6 and 8.
- b) the “Additive package II” (which contains an extra dosage of extreme pressure additive) performed differently in different test instruments;
 - **SRV tests**; an increase of the wear values regardless the thickener type for the greases based on BO 600 was observed, Figure 10.
 - **Four-ball tests**; positive impacts (both on the wear scar diameter and load carry capacity) were observed for the lithium grease. In the case of the lithium complex grease (Sample # 10) this positive effect was only monitored on the load carrying capacity, Table 7.
- c) **The thickener content** of a grease seems to affect the tribological performance which, in turn, is dominated by the characteristics of the base oil, e.g., its polarity, degree of solvency and viscosity.
- d) **The conditions in SRV and four-ball rigs** are different which can explain the different outputs. This also emphasises the necessity of selection of relevant test methods that are supposed to simulate the actual conditions of applications in the fields, otherwise the obtained data may lead to the wrong conclusions.

In complex systems, such as lithium and lithium complex greases, the components involved may sometimes interfere with one other, hence, in order to formulate a high-performance grease, the use of tribological tests, as demonstrated in this study, could be of valuable assistance.

Acknowledgment

The authors would like to thank Dr. George Dodos at Eldon’s for the four-ball measurements.

Reference:

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An Innovative Preformed Thickener for the Preparation of Structurally Stable and Tribologically Effective Polyurea Greases

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Abstract

This paper describes the making of fully formulated polyurea (PU) greases based on a novel preformed thickener and a unique set of nonmetallic, non-sulfur additives in a variety of base oils. Batches were made via a two-step process (thickening and oil dilution) or a patent pending master batch approach, and the greases were shown to exhibit outstanding structural stability and load carrying, anti-wear and friction reduction performance through extensive tribological and rheological testing under extreme conditions without resorting to heavy metal- and traditional sulfur-based additives. This combination of preformed PU thickener and novel additives offers grease manufacturer easy and convenient means to prepare high performance polyurea greases for applications that traditionally use lithium complex or overbased calcium sulfonate grease.

Introduction

Polyurea (PU) greases are nonmetallic greases prepared with polyurea thickeners. They are known for their high temperature stability, low noise characteristics, good mechanical properties, oxidation resistance and high shear stability [1]. These attributes make PU greases the first choice of OEMs and end users for premium grease applications such as sealed-for-life bearings, constant velocity joints, ball bearings, and electric motors.

PU greases, however, are underutilized in heavy duty applications under extreme loads and in harsh environments because overbased calcium sulfonate (OBSCS) greases are often preferred due to their inherent load-carrying properties [2]. For example, in the steel industry, although OBSCS greases become rheologically unstable and lose their effectiveness after extended use at high temperatures, they are favored over PU greases. In an early attempt, we improved the rheology of an OBSCS grease by blending it with a PU grease [Wei et al. [3]]. Furthermore, for more widespread use of PU grease, it is necessary to overcome the difficulties and challenges in its so-called in situ manufacturing that requires the use of toxic isocyanates, such as MDI (diphenylmethane diisocyanate) and TDI (toluenediisocyanate), and hazardous amines as raw materials. Handling and reacting these raw materials is difficult for many grease manufacturers, which has hampered the development and customization of PU greases. In this paper, we present the successful development of a polyurea grease that was made via a preformed PU thickener. This preformed thickener is in powder form (Figure 1) and provides an easy and flexible method to formulate PU greases in a variety of base oils with the use of effective additives to enhance tribology performance [4].



re 1 Preformed PU thickener powder

In this study, additives were evaluated under extreme tribological (ET) conditions such as high load, extreme temperature or other severe environmental or operating conditions [5]. In the real world, ET represents situations where lubricated devices or systems operate under conditions that are often outside their design specifications. In this study, our aim was to improve grease tribology by using additives that had minimum impact on rheological properties and enhanced the value of the PU greases.

Experimental

This paper is divided into two parts. First, we describe the preparation of polyurea base grease via a two-stage process. In the first stage, preformed PU thickener powder was dispersed and gelled under heat in base oil to make a PU master batch of base grease (typically NLGI grade 4-5 or stiffer). In the second stage, diluent oil was added, and the master batch was milled to adjust the consistency of the base grease to the desired level. Additives were added to the base grease in the second step to make a fully formulated grease. These two stages were carried out via either a batch process or a continuous process. The batch process was performed in a KitchenAid mixer in the laboratory, and the continuous process was done via a proprietary extrusion process. Post-milling via a 3-roll mill or a homogenizer was essential to develop the structural stability of the PU greases.

In the second part of this paper, we describe tribological testing performed with an MFT-5000 tribometer (Rtec-instruments, San Jose, CA) with 4-ball, pin-on-disc, and ball-on-disc modules and a Pin & V Block Test Machine (Compass Instruments Inc., Sugar Grove, IL). A Brookfield viscometer and a Haake™ Mars™ 60 rheometer (Thermo Fisher Scientific Inc., Waltham, MA) were used for rheological testing.

Results

Preparation and structural and mechanical properties of PU base greases

In the first part of this study, NLGI grade 2 PU base greases were prepared successfully from preformed PU powder thickeners in a variety of base oils. Table 1 provides the thickener yields to achieve grade 2 PU greases. These yields were very similar for different base oil viscosities and processing equipment. In general, yields (%) were higher in naphthenic and aromatic than polar/non-hydrocarbon base oils.

Table 1 Grease yields

Base Oil	Thickener Yield
Alkylated Naphthalene	8-10%
Naphthenic	7-10%
Paraffinic	8-10%
Polyol Ester (POE)	10-12%
Polyalkylene Glycol (PAG)	10-14%
Di-capped PAG	10-12%

The PU powder thickeners used in this study were prepared by reacting MDI with amines through a proprietary process, and the thickeners were in the form of fine powders (see Figure 1). Finished greases were made as described in the experimental section via a two-step process. The conversion was easy, but care must be taken to maximize the thickening/ gelling (first step) and to fully disperse the fiber/platelet microstructure in the oil addition step and via milling (second step). In Figure 2, comparison of FTIR spectra shows good agreement between base greases made via the in situ method and the two-step method with preformed PU powder, and the preformed PU powder.

Figure 2 FTIR spectra (from top to bottom) for: A) a commercial PU grease made in situ; finished PU greases made in B) alkylated naphthalene (AN), C) polyolester (POE), D) a 50/50 blend of polyalphaolefin (PAO) and AN, and E) di-capped polyalkylene glycol (PAG); and F) the preformed PUGT (PU grease thickener) powder.

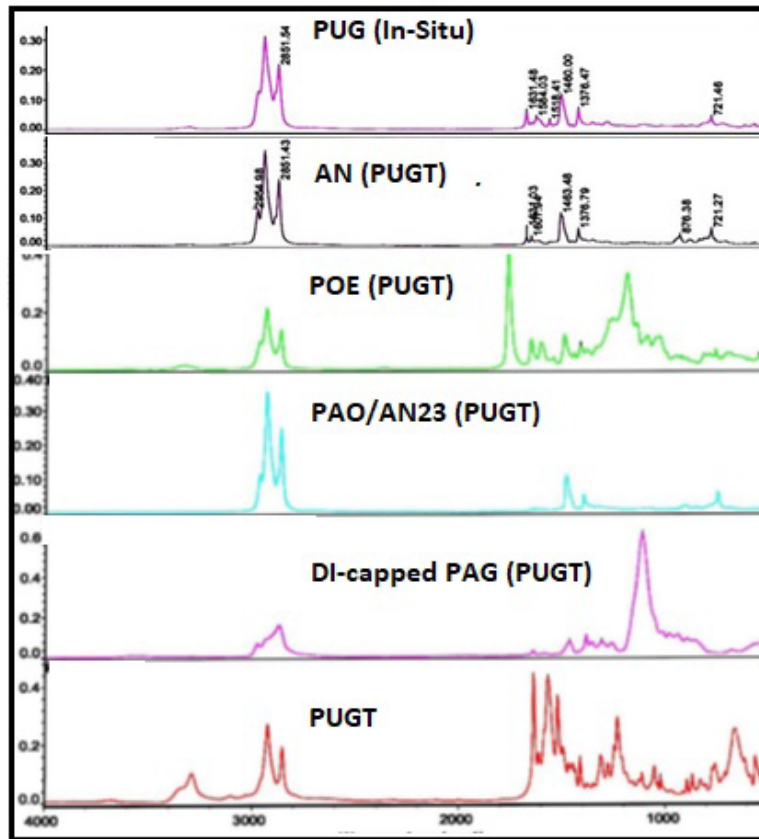
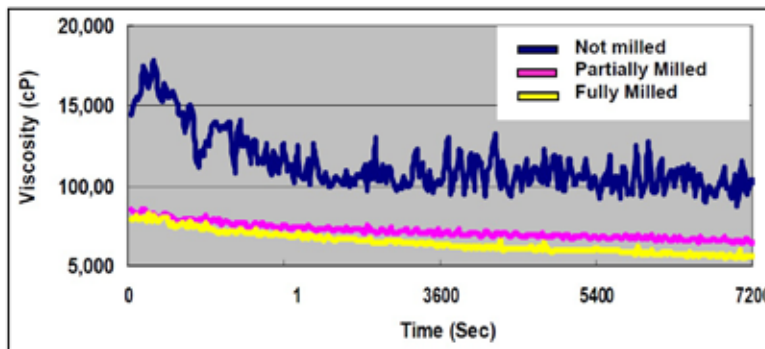


Figure 3 Milling effects - PU grease made from PU powder in 500N base oil



Effect of Milling

A 3-roll mill or a homogenizer was used for milling under conditions typically used in soap-based grease manufacturing. Figure 3 shows the effect of milling, which reduced and smoothed the viscosity curve. The smooth yellow curve represents the fully milled polyurea grease.

Structural stability and dropping points

Table 2 shows data for representative NLGI grade 2 base greases prepared in AN23 (alkylated naphthalene, 193 cSt at 40°C, 19.8 cSt at 100°C) and milled via a three-roll mill. These greases retained their consistency after they were worked for 60 (P60) and 10,000 (P10,000) strokes and had low oil separation and high dropping points. The results for P10,000 are particularly important because many end users prefer greases that maintain their consistency and thickener structure in long-term applications, and some greases soften when they are worked for 10,000 strokes.

Table 2 PU finish greases (in AN23 base oil)

<u>Powder ID</u>	<u>Yield %</u>	<u>P0</u>	<u>P60</u>	<u>P10,000</u>	<u>Oil Separation %</u>	<u>Dropping Point, C</u>
G-175	9%	263	265	269	0%	280
G-179	8%	262	265	308	0.9%	280
G-157	7.5%	277	279	308	1.1%	275

Rheological testing of PU base and fully formulated grease – thermal stability

PU greases are known for their robust temperature performance. To benchmark/compare with commercial grease, we carried out rheology testing in which thin films of grease were heated between two parallel plates at 150°C for 10 min in a rheometer.

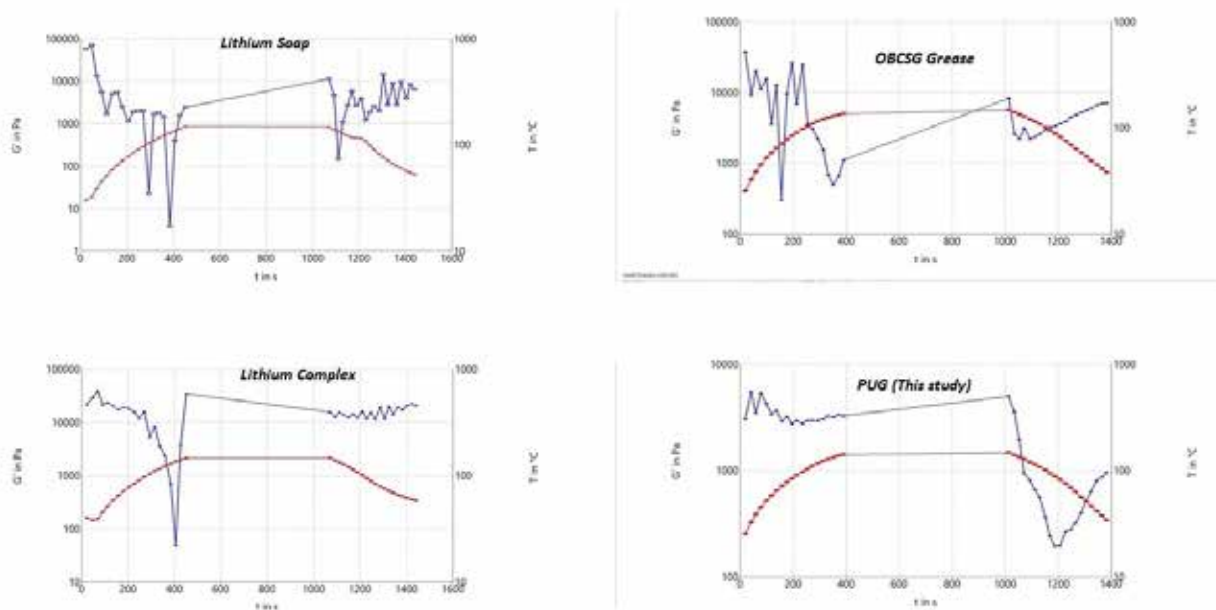
Figure 4 Photographs of PU base grease prepared from preformed PU powder in AN23 base oil before (left) and after (center, bottom) thermal aging in a rheometer at 150°C for 10 min and three commercial greases prepared in situ (PU, OBCS, and lithium complex) after the same thermal aging.



Figure 4 shows photos of PU greases after heat aging. There is a striking difference between commercial PU grease (black) and other commercial greases made in situ (brown and red) and grease made from preformed PU powder in this study (whitish and tan). The integrity of the grease made via preformed PU powder remained intact, but the PU, OBCS, and lithium complex greases (made in situ) separated into pieces.

Figures 5 shows thermal aging profiles (G' and temperature vs. time) for the four greases shown in Figure 4. The PU grease prepared in this study had the most relatively stable G' , but the three commercial greases all gave significant and multiple drops in G' as the temperature ramped up to 150°C, a sure sign of thermal instability.

Figure 5 G' and temperature vs. time



Tribological testing under extreme conditions

Polyurea grease is known for its longevity that is three to five times better than that of lithium-based grease [5]. It can be modified with many additives in the trade, and on many occasions, sulfur-based and heavy metal-based additives are used. In this study, we focused on additives with non-sulfur chemistry and explored a variety of extreme tribological conditions into maximum/boundary regions as shown in Figure 6.

Extreme tribological conditions usually mean tests performed at the maximum load or speed of a tribometer. But real-life conditions can be more demanding. In this study, we used multiple tribological tests with point (4-ball, pin-on-disk (POD), ball-on-disk (BOD)) and line (Pin & Vee) contacts to evaluate greases, as summarized in Table 3.

Figure 6 Extreme Tribology

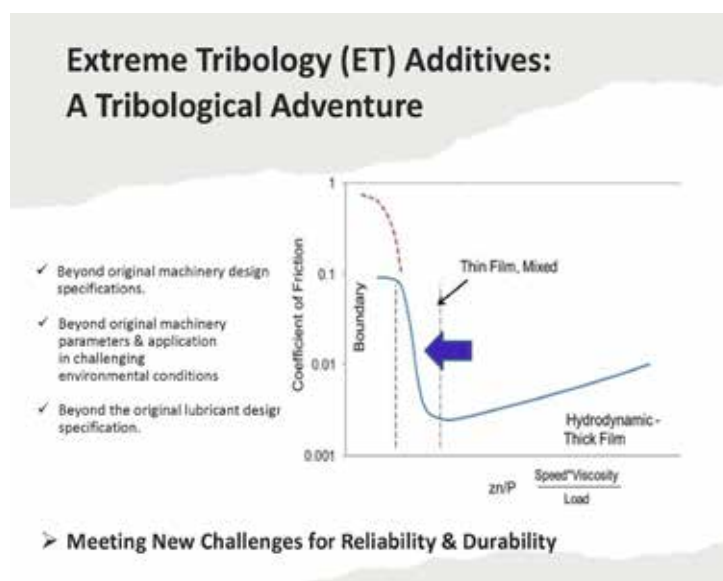


Table 3 Tribology test results for a grease prepared with preformed PU thickener, AN23, and various additive packages. Tests were conducted via 4-ball, Pin & Vee, ball-on-disc (BOD) and pin-on-disc (POD) methods.

Grease sample	Total Dosage	Add package	Solid booster	Sulfur EP	DTC/DMTD	FM	4-ball Weld	Falex P&V	Ball on Disc wear	Pin on Disc wear	4-Ball AW
PUG-AN23	7%	A	X		X	X	800+	1750+	Nil	Best	0.65
PUG-AN23	5%	B		X	X		315+	1750+	High	Worst	0.56
PUG-AN23	7%	C	X	X	X	X	800+	4500+	High	Better	0.55
PUG-AN23	7%	D			X	X	800+	4500+	N/A	N/A	0.6

Additive package B is commercially available, contains sulfur EP and DTC/DMTD (dithiocarbamate/2,5-dimercapto-1,3,4-thiadiazole) additives, and is used in greases that meet GC-LB requirements. In this study, the POD and BOD results were poor. In Figure 7, the wear scar is obvious.

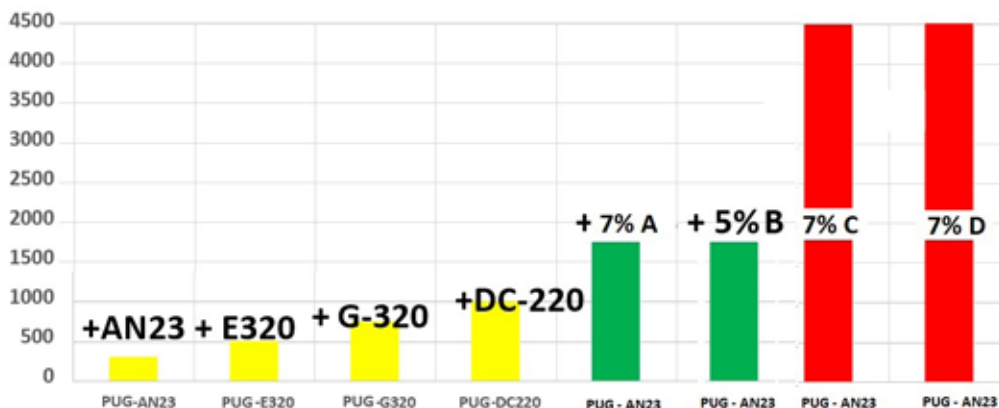
For additive package C, the addition of a solid booster (2%) increased the 4-ball weld load from 315+ to 800+kg and the Pin & Vee EP from 1750+ to 4500+ lb., and there was a slight improvement in POD wear.

In additive package A, we replaced the sulfur EP additive with the solid booster (used in additive package C) and a frictional modifier (FM). This gave the best overall tribological test results in this study.

Additive package D is targeted for use in greases to meet the new HPMG (High Performance Multiuse Grease) and higher specifications currently under development. We replaced the solid booster with another friction modifier and the DTC/DMTD booster. The Pin & Vee and 4-ball weld results were comparable to those for additive package C. No POD and BOD data were available when this paper was written, but they are expected to show minimum wear.

As shown in Figure 7, PU base greases formulated in AN23, E320 (ISO 32 polyolester), G-320 (ISO 320 PAG), and DC-220 (ISO 220 di-capped PAG) gave Pin & Vee weld values up to 1000 lb. The maximum load was achieved with additive packages C and D in PU grease formulated in AN23 base oil.

Figure 7 Pin & Vee weld loads



Each tribological bench test has its unique use that often is tied to historic grease/lubricant requirements with its specific contact geometry, speed, and load and mode of operation. For instance, the Pin & Vee test provides a weld load range up to 4,500 lb. that seems to easily differentiate between base grease and formulated grease (Figure 7). 4-ball weld requirements are included in GC-LB and HPMG specifications, but 4-ball wear testing does not seem to be overly effective compared to BOD and POD testing (Figure 8). BOD and POD instruments offer a wide range of test conditions such as rotational speed and loading in stages (Figure 9). These methods can be used to model different conditions corresponding to the Stribeck curve (Figure 6) from hydrodynamic to elastohydrodynamic, to boundary conditions. The wear scars are readily visible (Figure 10). In this study, the wear tracks were not analyzed quantitatively, but they were rated qualitatively (Table 3).

An additive package with an overall superior set of bench test results (additive packages A and D) as shown in Table 3 would with no doubt help to provide a well-defined and well-rounded option to replace the present sulfur- or heavy metal-based additive chemistries.

Figure 8 POD wear tracks for model greases formulated with additive packages A (best) > C (moderate) > B (worst)

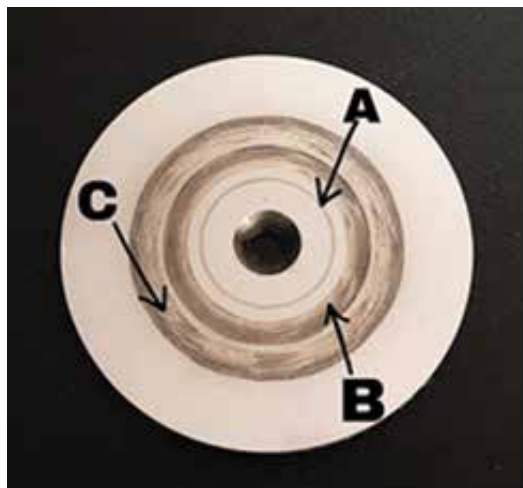
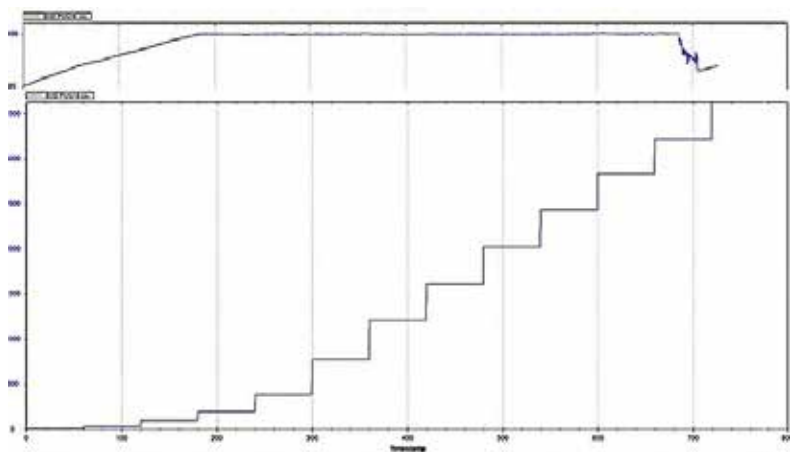


Figure 9 BOD and POD Test Conditions: varying speed and loads from 50 to 800 N in stages



Conclusions

A novel preformed PU thickener powder was used to make laboratory-scale batches of PU greases. The process was easy, and it can conceivably be scaled up for greater efficiency.

PU greases were made in a variety of base oils with yields from 8 to 12%. The polyurea base greases showed good structural stability and dropping points greater than 270°C. In a thermal aging test at 150°C in a rheometer, a layer of base grease prepared with PU preformed thickener in alkylated naphthalene base oil remained intact, while commercial PU, OBCS, and lithium complex greases (all made in situ) separated into pieces.

The use of non-sulfur based additives under extreme tribological (ET) conditions delivered superior load carrying performance and wear protection and showed potential to replace the present sulfur-based chemistries (offensive odors) and heavy metal-based chemistries (environment hazards). The combination of preformed PU thickener powders with ET additives will help the grease industry extend the use of polyurea greases beyond current sealed-for-life applications by OEMs to broader applications such as those of lithium and other greases.

Acknowledgements

This paper was based on a presentation given at the 2020 NLGI Annual General Meeting, which was virtual. The tribological testing was carried out by scientists and engineers at the Institute for Innovative Research (IFIR), a

wholly owned subsidiary of Novitas Chem Solutions. Mary Moon, a consultant to Novitas Chem Solutions, revised this manuscript.

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NLGI RESEARCH GRANT REPORTS



Strategies for Optimizing Greases to Mitigate Fretting Wear in Rolling Bearings

2020 – The University of Akron

Grease Lubrication of New Materials for Bearing in EV Motors

2019 - University of California – Merced

Strategies for Optimizing Greases to Mitigate Fretting Wear

2018 - The University of Akron

Determination of Grease Life in Bearings via Entropy

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

COURSES – Focuses on perfecting the basic & advanced lubricating grease courses provided during the NLGI annual meeting.

- **Basic Grease Lubricating Course** Chair: Gareth Fish; Co-Chair: Anuj Mistry
- **Advanced Grease Lubricating Course** Chair: Martin Keenan; Co-Chair: Chuck Coe
 - ✓ Both courses will be available during the 89th annual meeting in Toronto, Canada. For more information, please visit [HERE](#).
- **Hands-On Training Course** Chair: David Turner; Co-Chair: Constantin Madius
 - ✓ A RFP to host the training has been distributed to NLGI member companies. More details to come.



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CLGS Chair: John Sander

- ✓ A standard that certifies that an individual possesses a defined level of expertise in the field of lubricating grease. There are currently 43 CLGS holders.

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

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Lubricating Grease



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

Contact nlgi@nlgi.org for more information on how to submit.

The Lithium Crisis for the Grease Industry

Chuck Coe
Grease Technology Solutions

Contents

- Lithium end uses and global supply/demand outlook
- Lithium in grease
 - How much is used
 - Trends in lithium grease production versus alternative thickeners
 - Comparison of lithium grease properties to other thickeners
- Alternatives to reduce lithium usage in lithium and lithium complex greases
 - Replace LiOH monohydrate + water with dehydrated LiOH dispersion
 - NCH patent – Andy Waynick NLGI papers
 - Borated esters as “dropping point enhancers”
- Substituting other thickeners – challenges
- Regulatory issues
- The future

Lithium End Uses

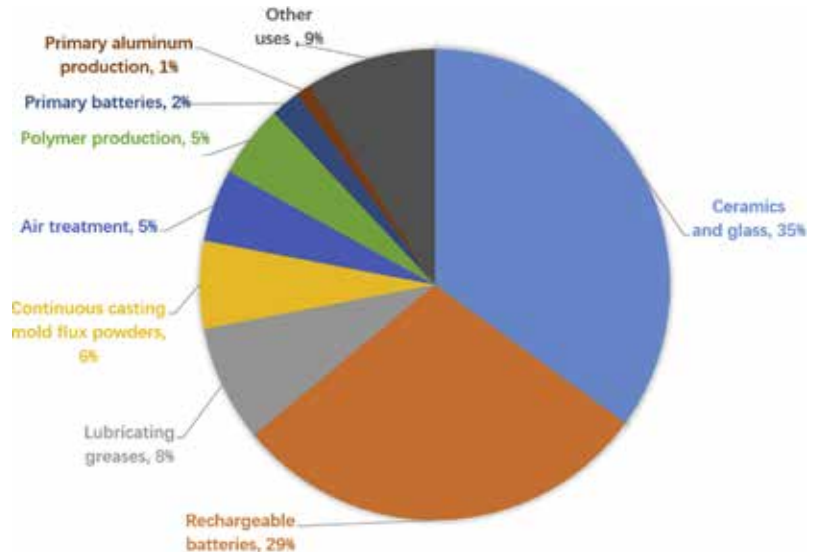


Figure 1 Source Ding Weng, et al., Progress in Natural Science: Materials International, <https://doi.org/10.1016/j.pnsc.2020.01.017>

Usage in greases is declining as a percent of the total due in part to a decline in lithium and lithium complex grease production, and also as a result of growth in the rechargeable battery demand. The grease industry is challenged by the recent increases in price and decreases in availability of lithium hydroxide, driven by the growing demand for lithium for batteries for mobile electronics and especially, *electrified vehicles*



Many countries are mandating and supporting with tax incentives minimum EV market share.

US EPA has just approved increased auto maker MPG requirements, expected to drive EV sales to 17% of new vehicles.

Where is the lithium?

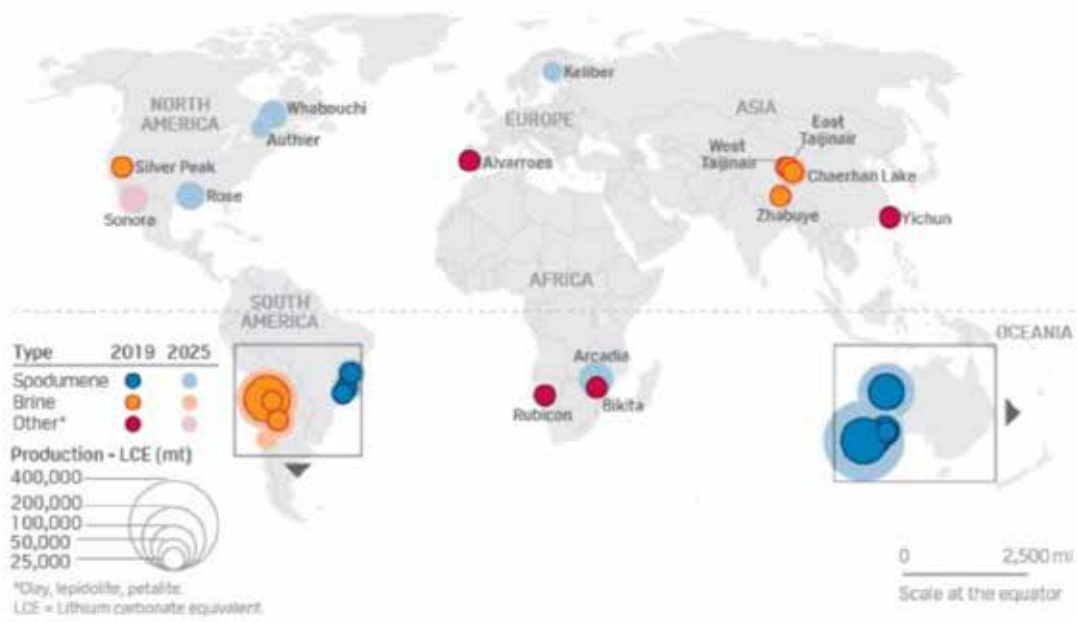


Figure 2 Source: S&P Global Market Intelligence, S&P Global Platts

US is hard rock and brine; Argentina, Chile, and Bolivia are brine; Australia is hard rock; Brazil is hard rock; Portugal is hard rock; Zimbabwe is hard rock; China is hard rock and brine.

Supply/Demand Outlook

- Ongoing pandemic related restrictions on deployment of people, resources are impacting the ability to bring on new sources
- ongoing supply chain challenges restricting movement of equipment, product, significantly increasing labor, logistics, raw material costs
- long timelines and high capital slowing the development of new resources
- pandemic-related restrictions on expansion projects' schedules (DELAYS) as well as capex, opex and investment economics

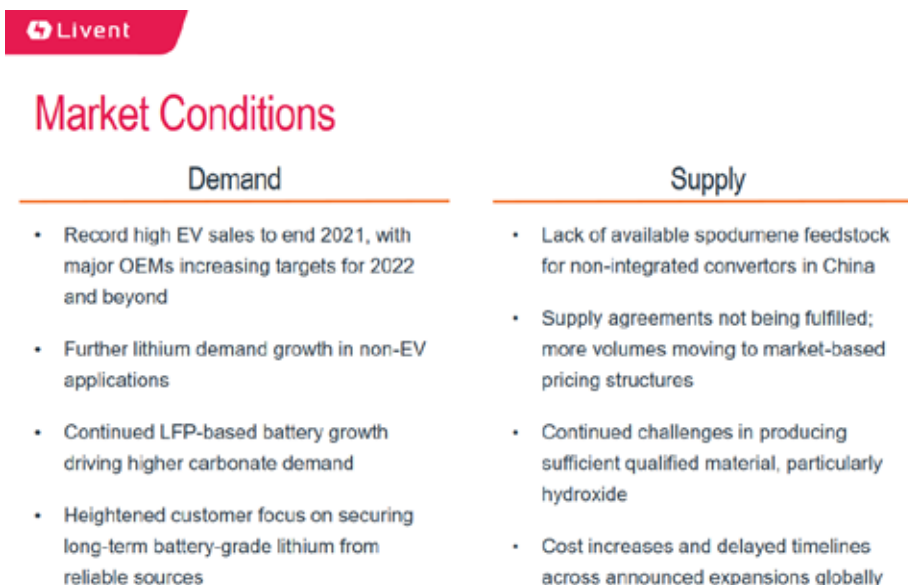


Figure 3 Source: Livent market update December 2021

Lithium in Grease

Lithium plus lithium complex account for over 70% of global production:

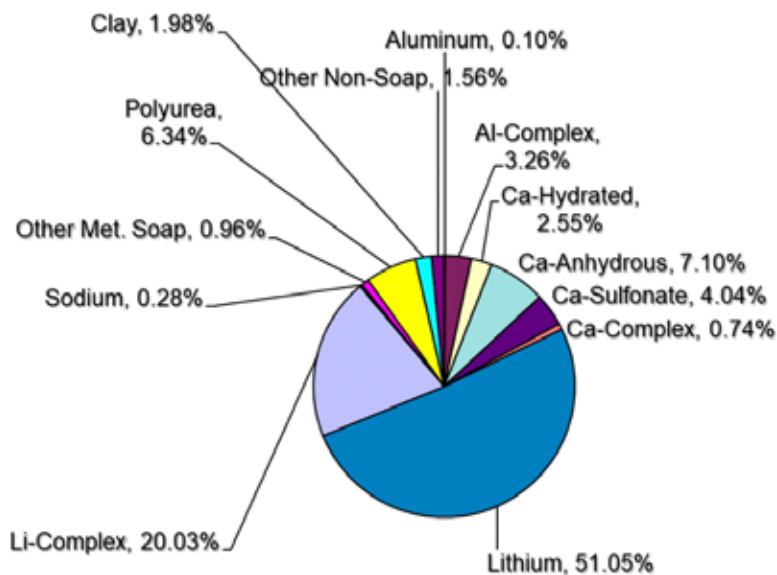


Figure 4 Source 2020 NLGI Global Grease Production Survey

Estimated NA LiOH consumption		Lbs	MT	Kg	30 \$/Kg	
Li soap		1,857,380	842	842,489	\$25,274,675	25 million dollars
Li complex		4,557,830	2,067	2,067,386	\$62,021,583	62 million dollars
					\$87,296,258	87 M\$

Based on 0.12% LiOH₂O in Li and 0.20% LiOH₂O in LiX

Estimated global LiOH consumption		Lbs	MT	Kg	30 \$/Kg	
Li soap		15,019,770	6,813	6,812,818	\$204,384,528	200 million dollars
Li complex		9,823,617	4,456	4,455,894	\$133,676,828	130 million dollars
					\$338,061,356	330 million \$

Table 2 Source 2020 NLGI Global Grease Production Survey

Trends in lithium grease production versus alternative thickeners

Simple lithium grease production has been declining for years, probably largely driven by a shift to lithium complex and other thickeners for higher performance, is now also being driven by increasing lithium hydroxide monohydrate prices.

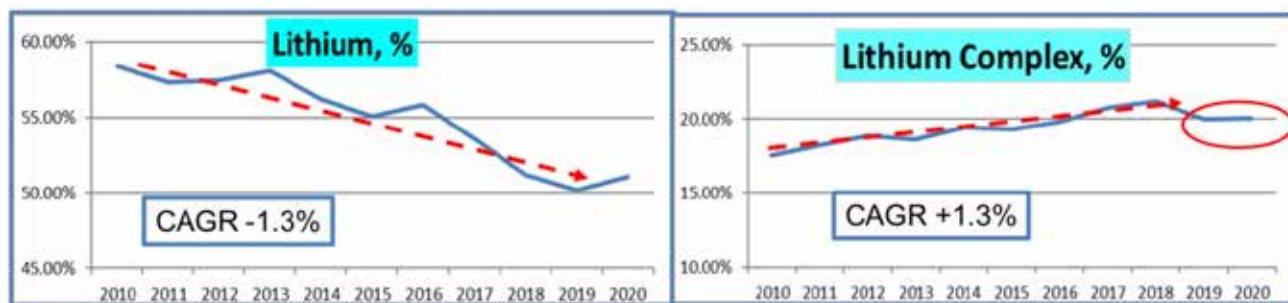


Figure 5 Source 2020 NLGI Global Grease Production Survey

At the same time, polyurea and calcium sulfonate grease production are both increasing, while aluminum complex grease production is not showing the same consistent growth. These three thickeners can be viable replacements for lithium complex thickeners, depending on the application.

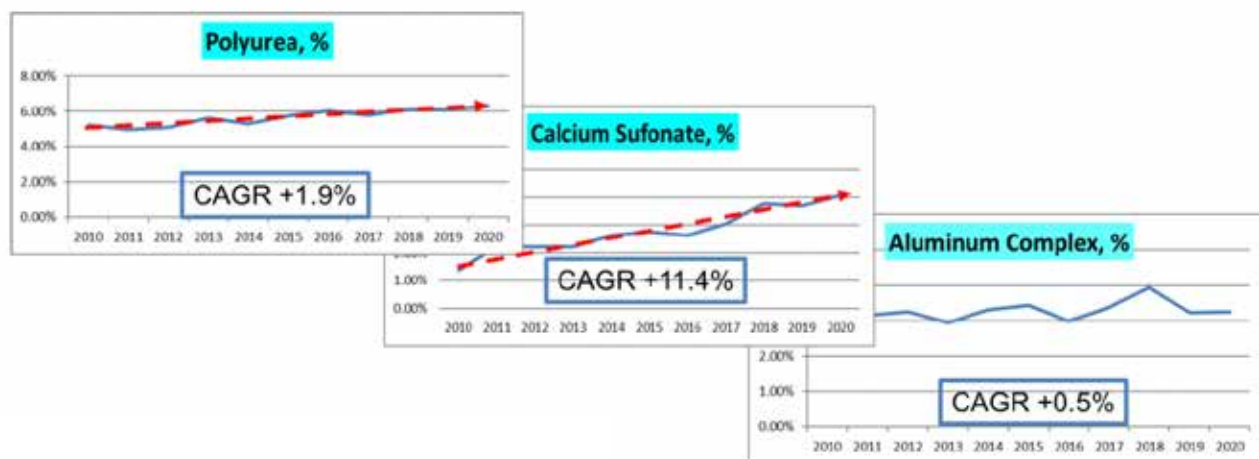


Figure 6 Source NLGI Lubricating Grease Production Surveys 2010-2020

Comparison of lithium grease properties to other thickeners

Lithium complex thickeners provide high dropping points, good mechanical stability, water stability, storage stability and pumpability. They do usually require additives to enhance wear resistance, load carrying, water and corrosion resistance and oxidation stability. They can be fine-tuned to a certain extent by choice of complexing agent and varying the ratio of complexing agent to lithium 12-hydroxy stearate thickener.

Aluminum complex thickeners also provide high dropping points, good mechanical stability, good pumpability, and excellent water resistance. They also generally require additives to improve corrosion resistance, oxidation stability, wear resistance and load carrying ability. They can be fine-tuned by varying the molar ratio of Benzoic Acid to Fatty Acid (BA:FA), and/or Total Acids to Aluminum (TA:Al). This is to manipulate thickening efficiency (yield), dropping point, and mechanical stability.

Calcium sulfonate complex thickeners also provide high dropping points, good mechanical stability, inherent water resistance, rust protection, and load carrying performance. They require only minimal additives, especially antioxidants. Their formulation and manufacture must be carefully controlled to provide the best pumpability possible, given the typically high thickener contents.

Polyurea thickeners provide high dropping points, fairly good mechanical stability (diureas), and inherent oxidation stability. Multiple variations of components and ratios provide the ability to customize performance to specific applications. The main challenges are a tendency towards poor compatibility with other thickener types as well as safety concerns with raw material handling.

All of these thickener types are suitable for a wide range of applications, as shown in Table 3.

Lithium Complex	Aluminum Complex	Calcium Sulfonate Complex	Polyurea
Automotive - GC/LB	Automotive	Automotive	Under-the-hood applications
General Industrial - HPM	Steel Mills	Construction & Mining	Constant Velocity Joints
Construction & Mining	Construction & Mining	Open Gears	Industrial
Steel Mills	Open Gears	Steel Mills	Steel Mills – Continuous Casting
Paper Mills	Slow, Heavily Loaded Bearings	Paper Mills	Electric Motor Bearings
Electric Motor Bearings	Thread Compounds	Marine	Fill-for-life applications
<i>new development</i> – HX1	Food grade - HX1	Food Grade - HX1	Food Grade - HX1
	Anti-Seize Compounds	Power Generation	

Table 3 Source NLGI Advanced Grease Course 2021

So how do we reduce lithium usage in greases?

The options fall into one of two categories:

1. Reduce LiOH usage in Li Complex greases
- Or
2. Switch to a different thickener type

First let's look at three options for reducing LiOH usage in Li Complex greases:

1. Replace LiOH monohydrate + water with dehydrated LiOH dispersion [1]
 - Higher raw material cost at typical 2:1 acid ratio (12 HSA : diacid)
 - Faster reaction
 - Allows reduction in diacid content (to 5:1 acid ratio), resulting in lower net formulation cost
 - Reduces amount of lithium equivalents needed

2. Add overbased (400 TBN) magnesium sulfonate at beginning of saponification and complexing reaction [2]
 - Facilitates more intimate co-crystallization of lithium complex thickener salts
 - Overbased calcium sulfonate (400 TBN) can also be used
 - Acid ratios can be decreased from about 2:1 to 3-5.8:1 (12 HSA : azelaic acid), greatly reducing lithium equivalents needed
 - Grease properties are equivalent to typical Li azelate complex grease

3. Replace dicarboxylic acid complexing agent with a borated ester [1]
 - Reduces lithium equivalent requirement by a factor of nearly 3x
 - Simplifies manufacturing, with borated ester addition occurring towards the end of the batch cycle, when typical performance additives are incorporated
 - Provides equivalent performance to dicarboxylate acid complexed grease, including dropping point, high temperature rheology, wear and life testing
 - Due to reduced thickener content (no lithium-dicarboxylate salt), improved low temperature pumpability is realized

Now let's look at switching to a different thickener type

1. From the end user's perspective there are several considerations
 - Cost
 - Compatibility
 - Performance

2. From the manufacturer's perspective there are also several considerations
 - Raw material handling
 - Equipment requirements
 - Processing changes

From the end user's perspective:

Cost

- The three primary alternative high-performance thickeners to replace Li complex are all more costly
- Compatibility
- When switching greases end users must be extremely careful about compatibility
- Polyurea and Aluminum complex are frequently incompatible with Lithium complex thickeners
- Performance

- The specific application must be considered in order to define needed water resistance, load-carrying, pumpability, mechanical stability, long life, etc. in order to choose the best thickener type.

From the manufacturer's perspective:

Raw material handling

- Polyurea raw materials are hazardous and require special handling and storage

Equipment requirements

- Polyurea, Aluminum complex and Calcium sulfonate complex typically require kettles, not contactors or continuous units
- Filters are frequently needed for polyurea greases

Processing changes

- Alternative thickeners require careful sequencing and cleaning of equipment
- Operators must be educated about new procedures and raw material handling
- Managing volatile, hazardous by products both handling and storage may be required

Regulatory issues

Proposed harmonization and classification of lithium hydroxide

In 2019, ANSES (French Agency for Food, Environmental and Occupational Health & Safety) submitted a proposal to the European Chemicals Agency (ECHA) for harmonized classification of lithium carbonate, lithium chloride and lithium hydroxide under the CLP Regulation (Classification, Labelling and Packaging of products).

ATIEL (Technical Association of the European Lubricants Industry), UEIL (Union of the European Lubricants Industry) and ELGI (European Lubricating Grease Institute) jointly opposed the proposal.

If the proposal is accepted (*which is very likely*), the lithium salts in question will have to be labelled as follows: "May damage fertility or the unborn child: Category 1A (H360FD)"

The proposed classification for LiOH is to be classified as Toxic to Reproduction for effects on fertility and development in category 1A H360FD. This effectively means that any concentration of this substance in lithium salts (soaps) greater than 0.1% (in the U.S.) would lead to the classification of the whole product. Since the grease industry does not generally sell the salts (soaps), one must assume that the same limits would apply to the greases which incorporate the soaps. During the manufacture of lithium soap greases, the alkalinity (free LiOH) in the soap concentrate is typically 0.02 to 0.05%, and of course, that number gets smaller (due to dilution) as additional oil and additives are added to make the finished grease. Therefore, this classification should not have a negative impact on lithium grease manufacturers or marketers.

However, the labeling requirement should impact the LiOH-H₂O suppliers who will need to label as noted. It might impact safety rules for LiOH-H₂O handling for manufacturers, but not labelling of the grease.

Note that 0.3% is the general concentration limit for classification in EU.

The future: a shortage is possible through 2030

According to Fitch Solutions:

- Well established lithium producing countries will record further growth
- New lithium-producing countries will emerge, amid rising interest, government support and increasing capital dedicated towards lithium projects
- Technological advancements in extraction make progress, posing upside risks to supply
- Actual supply could rise faster than expected, as a host of new players are developing new extraction techniques, namely geothermal brines and sedimentary (clay) deposits, which could upend primary supply of lithium
- Lithium reserves are ample and keep on growing, suggesting plenty of potential to boost supply in the long term

According to Green Authority's article "10 alternatives to lithium-ion batteries: Which new tech will power the future?":

- Emerging technologies are expected to begin to replace lithium-ion batteries – magnesium, sodium, and others.

According to my assessment based on considerable reading of the available literature, there are so many unknowns in the supply side that forecasting is highly challenging to say the least. Additionally, shortages of LiOH in the near term may reduce the EV demand below what is currently promised by the auto industry. And there is the elephant in the room: EV charging infrastructure is slow in coming and consumers continue to be hesitant to purchase EVs due to range and charging concerns. So, the supply shortfall may somewhat resolve itself over the next decade or so.

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1 NLGI paper 2018 G.Fish: Lubricating Grease Thickeners: How to Navigate you Way through the Lithium Crisis

2 NLGI paper 2020 A.Waynick: A Fresh Look at Lithium Complex Greases Part 2: One Possible Path Forward

RETROSPECTIVE

Electric Compatability of Grease with Elastomeric Seals

For many years, seal materials have been part of moving mechanical equipment. They are used to close open spaces between moving components on equipment thereby stopping the lubricant from running out while at the same time preventing harmful contaminants from entering into the machine and causing damage. In the role of sealing the lubricant into the machine, the lubricant will come in contact with the seal material. This means that the lubricant must be compatible with the seal material. Popular seal construction materials include fibers; such as paper, leather or felt, metals, or elastomers such as; rubber, nitrile, polyacrylate, silicone, fluoro-elastomer, propylene, PTFE, and combinations of these. The chemistry of the seal must be compatible with that of the lubricant. If not, there will be negative consequences for seal service lifespan and ultimately machine reliability.

The importance of seal compatibility has been known for many years. In fact, ASTM D4289 Standard Test Method for Elastomer compatibility of Lubricating Greases and Fluids has been a standardized test method that has been used by grease formulators for many years. Today it is part of the NLGI's new High Performance Multi-use (HPM) grease standard along with its predecessor ASTM D4950 Standard Classification and Specification for Automotive Service Greases. As with many things that seem like common knowledge today, this retrospective takes us back to an article published in this magazine when seal compatibility was a mere concept rather than a standard. NLGI always strives to provide a platform for the introduction of new concepts into the grease industry. We hope you enjoy this historical retrospective article on seal compatibility.

Evaluating Compatibility Of Greases With Elastomeric Seals

By: T. M. Verdura, General Motors Corp.

Presented at the NLGI 45th Annual Meeting in San Francisco, California, October 1977

Abstract

The elastomeric seals on grease-lubricated automotive components must be compatible with the greases applied so that the seals will satisfactorily perform their functions of retaining grease and excluding contaminants. Tests suitable for evaluating compatibility should correlate with service experience, and should be simple and easy to conduct.

A test commonly used, which correlates with service, involves the measurement of volume and hardness changes of a seal specimen caused by immersion in a grease. To develop a test which would not be affected by seal variability, other methods were investigated. These included determination of the aniline point and aromatics concentration (by infrared analysis) of the fluid component extracted from the grease. Results correlate well with those of the immersion test.

Introduction

The satisfactory performance of automotive suspension and linkage joints depends on the ability of the joint seals to retain grease and exclude contaminants. Since some greases can adversely affect rubber seals, grease-elastomer compatibility is an important factor in seal performance. Incompatible combinations will cause undesirable dimensional changes in seals such as illustrated in Figure 1.

Excessive swelling can defeat the purpose of a seal. The lip of an excessively swollen ball joint seal could pull away from the stud as the joint is rocked through extreme angles. Any opening between the seal and stud may permit the intrusion of water and other contaminants. Although greases can be formulated to tolerate some contamination, the grease formulation task can be simplified if the dimensions and integrity of the seal can be preserved.

Concomitant with excessive swell is a marked deterioration in the physical properties of the elastomer which may adversely affect seal performance; such properties include tensile strength, elongation, and hardness.¹ Flex life, ozone resistance, and low-temperature performance may also be affected. High-swelling lubricants can cause serious losses in the tear resistance of Neoprene², which may be the most serious consequence of using an incompatible grease in ball joints. Weakened ball joint and linkage seals are susceptible to damage since they are mounted near the wheels and/or close to the ground, where they can be struck by road debris.

Because some greases can adversely affect seal performance, auto makers generally include an elastomer compatibility requirement in their specifications for chassis greases. Compatibility is commonly evaluated by measuring the volume and hardness changes which occur when a portion of a rubber seal is immersed in a grease for a specified time at a given temperature. Details of such immersion tests are often sketchy since there is no industry-accepted test for evaluating the compatibility of greases with seals. The tests used in

specifications are adaptations of procedures used either to evaluate elastomers rather than lubricants or to evaluate fluid lubricants rather than greases.

The lack of an industry-accepted method for evaluating greases for compatibility with seals can cause problems for both the producers and users of both seals and greases. However, a solution to this problem is anticipated. ASTM Technical Division B (Automotive Lubricants) is developing a technical description of greases intended for automotive use. In the course of the ASTM work, it became evident that an adequate description would require an expression of the compatibility of greases with the elastomers used for automotive seals. Since a standard test is not available, Technical Division G (Lubricating Grease) was requested to develop a test that would satisfy the needs of Technical Division B.

To satisfy automotive engineers' natural preference for tests which utilize automotive components, the author believes that ASTM will develop an immersion test utilizing either actual seals or seal material in slab form. However, there are other methods of evaluating compatibility which may be worth investigating. Aniline point has long been used to describe the rubber swell properties of mineral oil.¹ The present study compares the aniline points of the hexane-soluble components of greases with the volume changes which occur when seal specimens are immersed in the greases. In addition, the results of a new, infrared analysis method are also compared to volume change results.

Materials

Seals — Three Neoprene ball joint seals were used in the immersion tests. Table 1 lists their ASTM D2000 — SAE J200 specifications. All three seals have the same, rather broad fluid resistance requirements. The designation E14 indicates (among other things) that the volume change shall be within -10 and +15 percent after immersion in ASTM No. 1 Oil (under specified conditions), and E34 indicates that the volume change shall not exceed +120 percent after immersion in ASTM No. 3 Oil.

Table 1 — Specifications for Test Seals
ASTM D 2000 — SAE J200

Seal	Specification
S1, S3	2BC620A14B14E14E34F17Z1Z2
S2	2BC525A14B14C12E14E34F17

Greases — The compatibility results reported in this paper were obtained over a long time period. They can be separated into two categories, early tests (conducted 10 to 15 years ago) and recent tests. The greases used in the early tests are not tabulated since little is known about their properties. However, the greases used in the recent tests are listed in Table 2 along with their thickener identity and NLGI consistency grade as well as typical viscometric data for their base

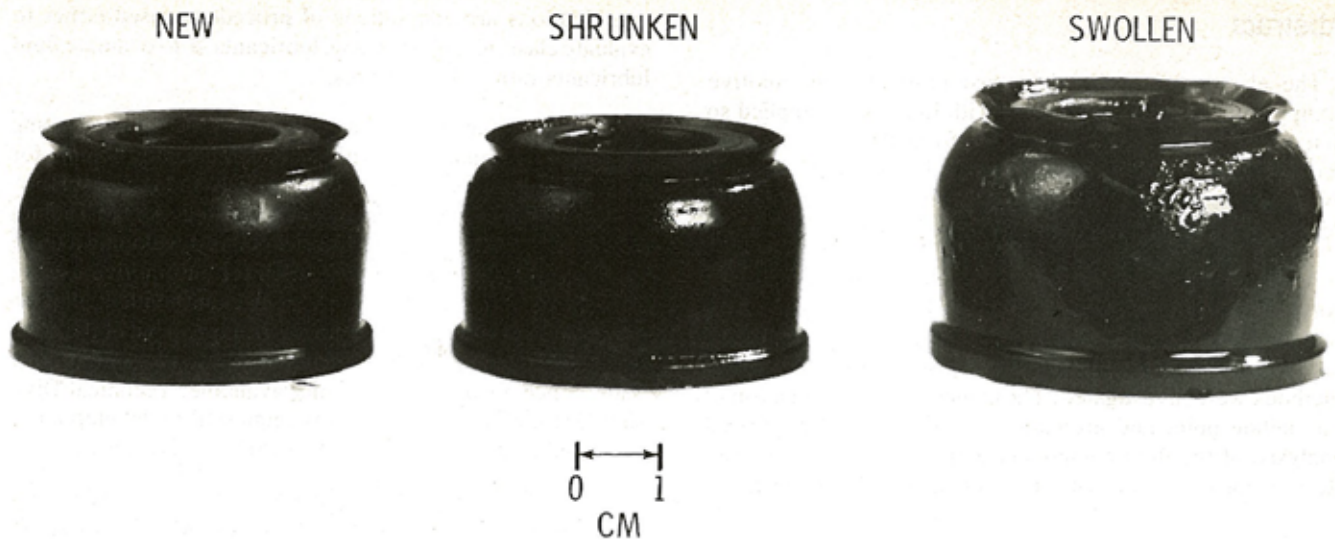


Figure 1 — Comparison of New Seal with Slightly Shrunken and Severely Swollen Seals.

oils. Also included in the table are some remarks about the usage of these greases. With the exception of grease J (NLGI Reference System A), all of the greases were formulated for use in automotive applications. Greases B, E, G, and H have been used at one time or another for the initial-fill of ball joints and/or steering linkage pivots. Greases D and F are initial-fill wheel bearing greases. Greases B and C are service station chassis greases; K and L are qualified automotive and artillery greases (MIL-G-10924B). Greases A and I were submitted as candidates for initial fill of ball joints, but were not approved.

Methods

Immersion Test — In the commonly used method of determining the effect of a lubricant on an elastomeric seal, the seal is immersed in a lubricant for a given time at a given temperature, and the change in seal properties measured. Typical examples of this approach are ASTM Method D 471

and Federal Test Method Standard No. 791b, Method 3603.4. Although these methods appear to be simple tests, there are some difficulties in practice, some of which will be discussed later.

The immersion test used in this investigation was a modification of Method 3603.4. Details of the modified procedure are given in Appendix A. Basically, the test consists of determining the volume of a section (approximately 25 x 50 mm) of test seal by means of water displacement, immersing it in the test grease for 70 hours at 100°C, then determining the change in volume. Hardness measurements are also made before and after immersion using a Shore Durometer A (ASTM D 2240). Only seal specimens of consistent initial specific gravity are used. If the seals have molded-in, plastic or metal reinforcements rings, they are cut out so that only elastomeric material is used in the test.

Extraction Technique — The effect of lubricants on seal compatibility depends primarily on the type of base oil used (seals are less affected by paraffinic oils than by naphthenic or

Table 2 — Composition and Properties of Test Greases

Grease	Thickener	NLGI Grade	Base Oil		Usage
			Vis., cSt at 37.8°C	V.I.	
A	Clay	2	165	91	Initial-fill candidate (not approved)
B	Ca complex	1	222	65	Initial-fill, service
C	Li hydroxystearate	2	194	90	Service
D	Li complex	2	130	96	Initial-fill
E	Li hydroxystearate	2	108	90	Initial-fill
F	Al complex	2	106	90	Initial-fill
G	Li hydroxystearate	2	109	64	Initial-fill
H	Ba complex	2	37	144	Initial-fill
I	Li hydroxystearate	2	65	75	Initial-fill candidate (not approved)
J	Li hydroxystearate	1	69	48	NLGI Reference System A
K	Ca hydroxystearate	2	12	57	MIL-G-10924B
L	Ca hydroxystearate	2	15	51	MIL-G-10924B

aromatic oils). However, some of the additives used to improve certain properties of the lubricant may affect seal compatibility.³ It would appear, therefore, that if the aniline point or IR analysis is to correctly reflect the contribution of the grease to seal swell, then the analysis of the oil-additive mixture may be more informative than that of the base oil alone.

The two-phase nature of grease precludes the direct measurement of the aniline point or quantitative infrared (IR) analysis of the fluid component. The logical solution to this problem is to analyze the base before the grease is made. However, base oils are seldom available to grease users. Therefore, it is necessary that the solid and liquid components of the grease be separated and the fluid portion subsequently analyzed.

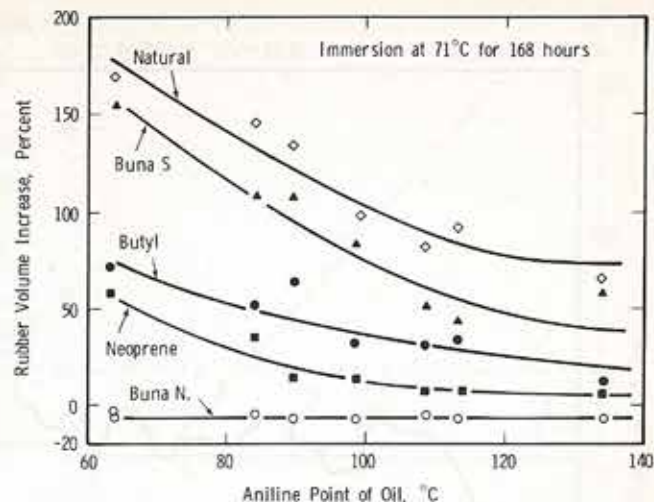
The separation procedure consists of extracting 25 g of the test grease with n-hexane in a Soxhlet extractor for two hours. This will yield enough sample (about 11 mL) for both an IR analysis and an aniline point determination. The solvent containing the oil and additives is cooled to room temperature, and filtered through a 5 μ Millipore filter to remove any insolubles which may have been carried over during the extraction. The sample is recovered by distilling off the bulk of the solvent, and removing the residual hexane with a thin-film, vacuum evaporator.

Aniline Point Test — It is well known that the cyclic (aromatic and naphthenic) constituents of mineral oils swell rubbers to a greater extent than do the paraffinic constituents. It follows that knowledge of the chemical nature of a mineral oil would permit an evaluation of the effect of the oil on rubber. Mineral oils can be chemically characterized by means of their aniline points.⁴

The aniline point of a mineral oil is the lowest temperature at which equal volumes of oil and aniline are mutually soluble. Aniline, a cyclic amine, has a structure more closely allied to ring compounds than to chain-like paraffinic compounds. Consequently, at the same temperature, aniline is more soluble in oils having a higher concentration of cyclic compounds. To achieve complete solution of aniline in paraffinic oils, a higher temperature is required. Since most mineral oils are a mixture of cyclic and paraffinic compounds, the solution temperature (aniline point) is an indicator of the amount of cyclic materials present. The lower the aniline point, the higher the concentration of cyclic compounds; and conversely, the higher the aniline point, the lower the concentration of cyclic materials.

The relationship between the aniline point of mineral oils and the swelling of several types of elastomers is illustrated in Figure 2.³ The elastomers vary substantially in their response to attack by oils. More important, there is a definite relationship between aniline point and the amount of swell with a given rubber. This relationship should permit the prediction of the elastomer-swell properties of an oil if the aniline point is known.

In order to determine the correlation between aniline point and the seal volume change obtained in the aforementioned immersion test, the greases were extracted as described previously and the aniline points determined with the thin-film,



Source: Anon., "Significance of Specific Lubricant Tests," *Lubrication* 45, 139 (1959).

Figure 2 — Rubber Swelling in Lubricating Oil. Relationship Between Volume Increase and Aniline Point.

aniline point apparatus described in ASTM D 611. Basically, the aniline point determination consists of placing 10 mL portions each of aniline and oil into a sample vessel fitted with a stirrer and thermometer. The mixture is heated until complete solution occurs. Then the solution is cooled at a specified rate until the two phases reappear as indicated by a sudden clouding; the temperature is noted. This temperature is defined as the aniline point.

Infrared Analysis — As mentioned previously, elastomers are less affected by paraffinic oils than by naphthenic or aromatic oils, and aniline points are considered a measure of the concentration of naphthenic and aromatic compounds in mineral oils. Infrared spectral analysis can be used to measure the concentration of aromatic and paraffinic (but not naphthenic) compounds directly. However, the correlation of such measurements with elastomer swell has not been demonstrated. If correlatable, IR analysis would offer two distinct advantages over aniline point determinations: 1) Considerably less sample is needed, two drops for IR versus ten mL for aniline point, and 2) IR analysis is faster and provides a permanent record (as well as additional compositional information). Sometimes, small pools of oil are found on the surfaces of greases in containers. In such instances, the oil could be analyzed by IR without using the extraction procedure, saving even more time.

The infrared spectra of the extracted fluids were recorded with a Perkin-Elmer, Model 337, grating spectrophotometer using 0.105 mm KBr cells.

Brandes states that the absorptions at 1610 and 813 cm^{-1} are related to the aromatic compounds in mineral oils, and the peak at 722 cm^{-1} is due to long-chain paraffins.⁵ The absorbances of these peaks were calculated from the measurements of peak intensities (as illustrated in Figure 3) according to the following equation:

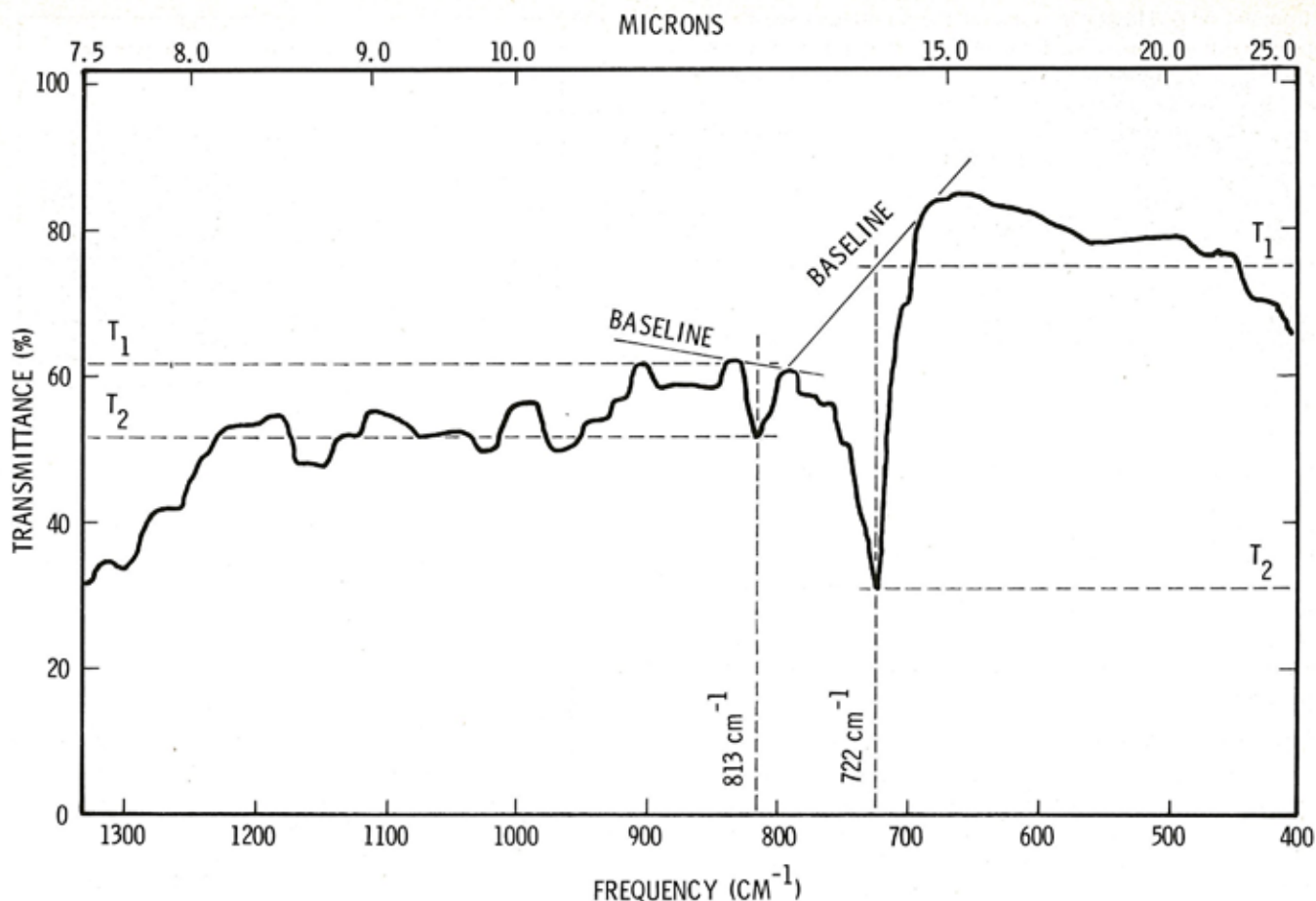


Figure 3 — Example of Measurements of Intensities of 813 cm^{-1} Aromatic Absorption and 722 cm^{-1} Paraffinic Absorption.

$$A = (1/h) [\log(T_1/T_2)]$$

where:

A = Absorbance, cm^{-1}

h = Cell thickness in centimetres

T_1 = Transmittance of the base line, percent

T_2 = Transmittance of the absorption, percent

(1)

Table 3 — Effect of Seal Specific Gravity on Volume Change

Grease	Seal S4 Immersion for 70 Hours at 100°C	
	Δ Volume, %	
	SG = 1.270	SG = 1.340
M	8.2	4.4
N	36.9	3.9
		15.4
O	62.0	14.3
		33.7
		34.0

Results And Discussion

Early Immersion Tests — About 15 years ago, a colleague of the author's learned that variations in seals of the same purported composition and properties significantly affected volume change.⁶ In the course of evaluating the compatibility of several greases with a Neoprene steering knuckle seal, it was observed that the repeatability of the volume change determinations was poor unless care was exercised to use only seals having the same initial specific gravity. Table 3 shows the effect of specific gravity on the volume change results. (The seal and greases used in this early study have not been included in Tables 1 and 2 because information about their properties is not available.) It can be seen that seals having higher specific gravity do not swell as much as those of lower specific gravity. The table also shows that the repeatability of the volume change determinations was good when seals of like specific gravity were used.

Since this investigation, some seal specifications have included a minimum specific gravity requirement. Also, our common laboratory practice is to use only seal specimens having a specific gravity within 0.010 of the mean and to report the initial specific gravity as well as the volume change results. Another laboratory technique which the author has found beneficial is to dip the seal specimens in wetting agent prior to weighing in water. This precludes the adherence of air bubbles, thereby improving the repeatability of the water weighings.

With the adoption of these practices, the immersion test was standardized in our laboratory. Subsequently, a somewhat more extensive study of the repeatability of volume change determinations was conducted. Table 4 shows that

when seals of consistent specific gravity were used, repeatability was good; with one exception (seal S3, grease P), the relative standard deviation (RSD) was less than 5 percent.

Table 4 — Repeatability of Immersion Tests

Seal	Grease	Δ Volume, %		ΔV, %		σ, %	Relative Std. Dev., %
S2	G	33.6	32.7	30.5	32.3	1.59	4.9
S2	H	34.0	35.9	35.0	35.0	0.95	2.7
S2	K	87.4	86.7	82.4	85.5	2.71	3.2
S2	S	61.2	60.5	59.9	60.5	0.65	1.1
S3	P	21.5	18.3	16.2	18.7	2.67	14
S3	Q	26.1	25.5	25.1	25.6	0.50	2.0
S3	R	34.0	33.8	33.7	33.8	0.15	0.5

Concurrent with the development of the immersion test, greases were evaluated in seals on actual vehicles. The volumes of whole seals (S3) were determined; the seals were installed on the ball joints of vehicles and filled with the test greases. After nine months of general transportation service, the seals were removed, cleaned, and their volumes again determined. Figure 4 shows that the volume changes determined in the immersion tests correlated (index of determination = 0.99) with those determined in vehicle tests, indicating that laboratory immersion testing is a valid method of evaluating the effect of greases on seals.

Recent Immersion Tests — The evaluation of the compatibility of lubricants and seals is an ongoing program in General Motors. Immersion tests are now routine, having undergone development many years ago. When new seal designs are introduced or elastomer specifications changed, it is general practice to run immersion tests with initial-fill greases. Such was the case with seal S1. When this currently used seal was adopted, immersion tests were run using initial-fill greases; other greases which had given a wide range of volume change results (with other seals) were also tested. There was a twofold purpose for these tests: 1) To evaluate the compatibility of this seal with approved initial-fill greases, and 2) to obtain data for use in demonstrating the utility of

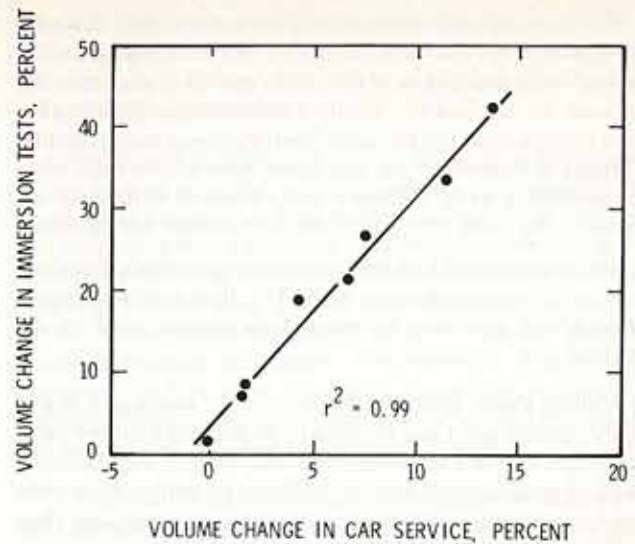


Figure 4 — Correlation of Volume Changes of Seal S3 Determined in Immersion Tests and in Car Service.

aniline point determinations and infrared analysis.

Table 5 shows the results of the immersion tests with the previously described greases. Also shown in Table 5 are some volume change results obtained with the formerly used seals, S2 and S3. Compatibility requirements vary somewhat among the several chassis grease specifications. The hardness change limitation is the same for all specifications, 0 to -10 points. However, the maximum allowable volume change ranges from 25 to 35 percent, depending on the particular specification.

All initial-fill greases gave satisfactory results with seal S1, but with the other seals, grease H gave borderline results. With the miscellaneous greases, only A and C were satisfactory. The last four greases gave unacceptable amounts of swelling and softening. The military specification greases (K and L) were particularly bad in this respect, swelling the seals as much as 76 percent and softening them as much as 24 points.

Table 5 — Compatibility of Lubricating Greases with Neoprene Ball Joint Seals

Grease	Seal S1 (SG = 1.395)		Seal S2 (SG = 1.370)		Seal S3 (SG = 1.340)	
	Δ Volume, %	Δ Hardness, Pts.	Δ Volume, %	Δ Hardness, Pts.	Δ Volume, %	Δ Hardness, Pts.
Initial-Fill Greases						
B	-6.2	0	—	—	—	—
D	-1.5	-3	—	—	—	—
E	0.5	0	—	—	—	—
F	4.6	0	—	—	—	—
G	8.2	-3	32.3	-9	18.6	-7
H	12.8	-10	35.0	-9	26.5	-10
Miscellaneous Greases						
A	-10.4	+2	9.1	-4	1.0	-3
C	-3.0	+1	17.6	-7	8.3	-3
I	28.0	-11	—	—	—	—
J	39.0	-13	—	—	—	—
K	60.0	-20	85.5	-21	58.3	-19
L	76.0	-24	108.1	-30	79.0	-32

The varied results obtained with the several seals illustrate the limitation of the immersion test. For example, grease G caused volume changes of 8.2, 32.3, and 18.6, respectively, for seals S1, S2, and S3. Yet the elastomer specifications for each of these seals has the same fluid resistance requirements. Variable performance was also noted in the earlier tests when the specific gravity influence was observed with seals obtained at the same time but of different production batches.

It is apparent that if an immersion test to evaluate greases is selected for standardization by ASTM, then a reliable supply of standard, precisely formulated elastomers must be developed.

Aniline Point Determinations — As mentioned previously, aniline point can be used to evaluate the rubber swell properties of fluid lubricants. To determine if the elastomer swell of greases could also be predicted by aniline points, the fluid components of the greases were extracted and their aniline points measured.

Preliminary tests were run to determine the time required to extract a representative sample of the fluid components. Initially, overnight extractions were used since the virtually 100 percent fluid recovery was believed desirable. Further experimentation showed that the extraction time could be reduced to two hours with no sacrifice in the quality of the sample (although the yield was less). Shorter extraction times were found to be impractical, since some greases will not yield a sufficient sample in less than two hours. Table 6 compares the aniline points of fluids extracted in two hours with those extracted overnight. It is obvious that there is no significant difference between the values. Consequently, the shorter extraction time was considered as giving a representative sample and was used in all the compatibility evaluations.

Table 6 — Effect of Extraction Time on Aniline Points of Grease Extracts

Grease	Aniline Point (t), °C	
	16-Hour Extraction	2-Hour Extraction
C	108.0	107.0
K	67.8	67.8
S	84.6	84.5

The repeatability of the extraction-aniline point method was checked with two greases, with results shown in Table 7. Each of the values represents a separate two-hour extraction and subsequent aniline point determination. It can be seen that the repeatability is quite good; the relative standard deviation (RSD) was less than 0.5 percent. Because of the excellent repeatability, it was considered unnecessary to run more than a single extraction-aniline point determination.

Table 7 — Repeatability of Aniline Point Determinations of Grease Extracts

Grease	Aniline Point (t), °C				t °C	σ, °C	Relative Std. Dev., %
	1	2	3	4			
H	92.6	92.4	92.4	92.0	92.4	0.25	0.3
K	67.8	67.8	67.3		67.6	0.29	0.4

The fluids were extracted from fresh samples of the greases used in immersion tests. Table 8 lists the aniline points of the extracts as well as their IR absorbances (to be discussed subsequently). In Figure 5 the aniline points are compared with the volume changes obtained in the immersion tests with seal S1. A least squares polynomial regression analysis indicated that these data fit the following second order equation with an index of determination of 0.98:

$$\Delta V = 340 - 5.75t + 0.0238t^2 \quad (2)$$

where:

ΔV = Volume change of seal, percent

t = Aniline point of the hexane extract, °C

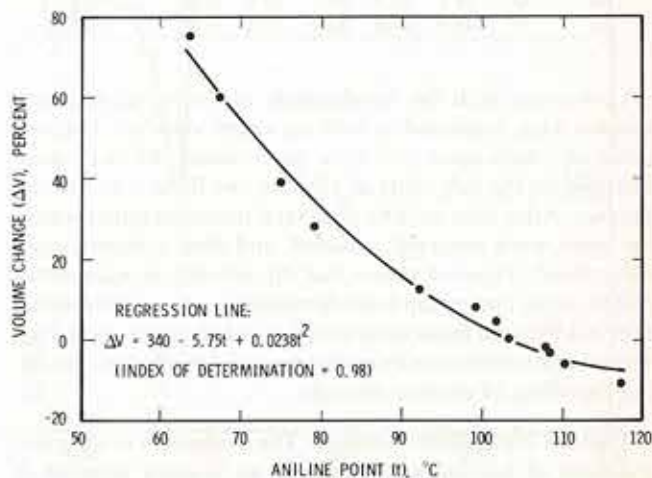


Figure 5 — Relationship Between Volume Change (ΔV) of SEAL S1 and Aniline Point (t) of Hexane-Solubles.

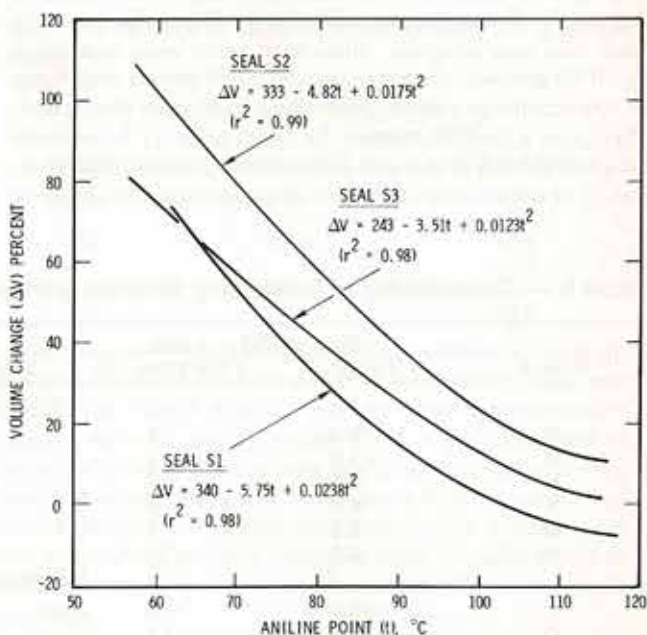


Figure 6 — Relationship between volume change (ΔV) of three seals and aniline point (t) of hexane-solubles.

Similar techniques were used to determine the correlation with seals S2 and S3. Figure 6 compares the correlation curves for the three seals. Although, the fluid resistance requirements of the seals are identical, each seal gave a different volume change-aniline point curve.

Table 8 — Test Results with Neoprene Ball Joint Seal S1

Grease	Aniline Point (I), °C	IR Absorbance (A) at 813 cm ⁻¹	Volume Change (ΔV), %
A	117.2	4.06	-10.4
B	110.1	7.81	-6.2
C	108.5	9.64	-3.0
D	108.0	7.86	-1.5
E	103.3	8.23	0.5
F	101.8	5.91	4.6
G	99.2	4.62	8.2
H	92.2	11.1	12.8
I	79.0	19.9	28.0
J	75.0	21.2	39.0
K	67.2	23.3	60.0
L	63.6	27.2	76.0

These results indicate that the correlation between volume change and aniline point must be established for each seal. Although this step requires considerable test time, once the correlation is established, the swelling characteristics of any other grease with that seal can be rapidly and accurately predicted. Furthermore, the comparison of greases by aniline points is not subject to the uncertain availability of identically formulated seals, as is the case with immersion tests.

Infrared Analyses — As mentioned previously, the composition of the fluid portion (including soluble additives) of a grease determines its effect on elastomers. Infrared spectral analysis was used to determine the composition of the hexane extracts of the test greases. The intensities of the aromatic absorptions at 1610 and 813 cm⁻¹ and the paraffinic absorption at 722 cm⁻¹ were measured, and the absorbances calculated according to equation (1). Attempts were made to correlate the absorbances to volume change.

The relationship between volume change and the absorbances of either the 1610 or 722 cm⁻¹ peaks was too poor to consider using these peaks to predict seal swell. On the other hand, the absorbance of the 813 cm⁻¹ peak correlates fairly well with seal swell. This relationship is shown in Figure 7. A least squares polynomial regression analysis indicated the data fit the following second order equation with an index of determination of 0.95:

$$\Delta V = 3.31 - 1.72A + 0.164A^2 \quad (3)$$

where:

ΔV = Volume change of the seal, percent

A = Absorbance at 813 cm⁻¹ (from eq. 1)

Similar to the aniline point method, once the correlation

between volume change and absorbance has been established with a given seal, the swelling characteristics of any other grease can be rapidly predicted from an IR analysis of the fluid component of the grease.

Assessment of Methods — One purpose of this investigation was to examine several methods of evaluating the compatibility of greases with elastomers, with a view toward the development of a standard test. While the immersion test used in this investigation is suitable for evaluating the compatibility of lubricating greases with elastomers, it has one serious shortcoming. Just as ASTM D 471 requires standard fluids to evaluate elastomers, the immersion test (if standardized) will require standard elastomers to evaluate greases. Precise elastomer formulations will have to be developed as well as a reliable source for them.

The standard-elastomer supply problem can be obviated by the use of aniline point determinations. However, the relationship between volume change and aniline point must be determined for each seal elastomer. Also, if aniline points are to be used in grease specifications, it should be determined if the aniline points of the base oil will correlate as well as the aniline points of the extracted fluids. If the base oil correlation is satisfactory, the grease formulator need know only the aniline points of his base oils, to develop greases of predictable seal compatibility characteristics. An extraction-aniline point determination of each batch of grease would be unnecessary.

Other comparisons between the two methods can also be made. The test duration for the immersion test is three days, but only about three hours for the extraction-aniline point procedure. The time requirement, in man-hours, is about the same for both methods if a small number of samples is evaluated (about one man-hour per sample for four samples). Considering only the time requirements, the immersion test is better suited to routine evaluations since it requires fewer man-hours per sample as the number of sample increases. On the other hand, when rapid evaluations are needed, the extraction aniline point method is more suitable.

Finally, with regard to the extraction-IR analysis procedure, the author believes that it could be utilized as a quick indicator of compatibility. However, it may be more difficult to standardize an extraction-quantitative infrared analysis technique than to standardize either of the other two procedures. Figure 7 suggests that the data scatter at the lower absorbances may be somewhat greater than desired. Also, there are some indications that the repeatability of the absorbance measurements is poorer than that of the aniline point determination.

Acknowledgment

The author thanks Mr. G. A. Peters (now retired from General Motors) for his able assistance and guidance in developing the immersion test techniques and also for the many immersion tests he performed.

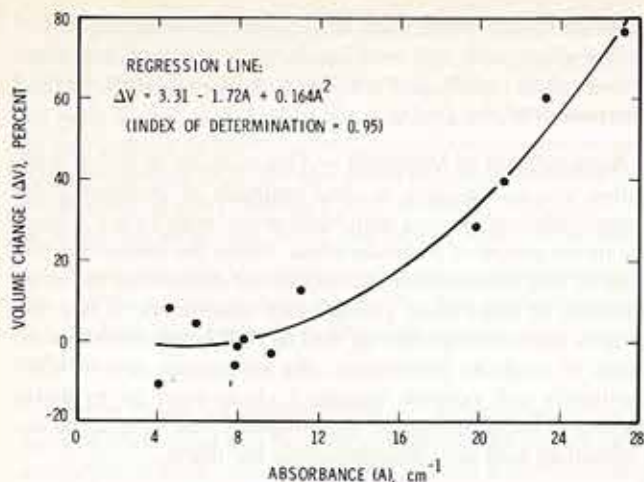


Figure 7 — Relationship Between Volume Change (ΔV) of Seal S1 and Absorbance (A) of 813 cm^{-1} Aromatic Peak of Hexane-Solubles.

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Appendix A

Test For Compatibility Of Lubricating Grease With Elastomers By Immersion GM 9098-P

1. Scope

- 1.1 This method determines the compatibility of lubricating grease with elastomer specimens cut from standard sheets or actual seals. A procedure is described for determining the changes in volume and hardness which occur when elastomer specimens are immersed in a test grease normally for 70 ± 0.5 hours at $100 \pm 2^\circ\text{C}$.

2. Significance

- 2.1 Because of the wide variations in service conditions, correlation between this test and particular applications should be determined on an individual basis. (In one instance, this test correlated with a vehicle test, $r^2 = 0.99$.) However, this test method yields comparative results by which service quality of greases may be judged and is especially useful for specification purposes.

3. Apparatus

- 3.1 Heat resistant glass beaker, approximately 50 mm in diameter by 70 mm deep (100 mL capacity) having volumetric markings, with cover (watch glass).
- 3.2 Analytical balance having a minimum sensitivity of 1 mg, with a hanger wire suitable for weighing specimens in both air and water.
- 3.3 Laboratory oven capable of maintaining $100 \pm 2^\circ\text{C}$.

4. Materials

- 4.1 Elastomer specimens cut from a standard sheet or actual seal having approximate dimensions $50 \times 25 \times 2$ mm, with punched hole suitable for suspending specimen in water. Specimens cut from seals must have molded-in reinforcements (if present) removed before measurements.
- 4.2 Commercial hexane (ASTM D 1836).
- 4.3 Methanol or ethanol, anhydrous.
- 4.4 Wetting agent, dioctyl sodium sulfosuccinate, 1% aqueous, made by suitable dilution of Aerosol OT (commercially available in 10% and 25% concentrations).
- 4.5 Distilled or deionized water, boiled.
- 4.6 Cheesecloth.
- 4.7 Filter paper.

5. Procedure

- 5.1 Cut elastomer specimens to the dimensions given in 4.1. If seals with molded-in reinforcements are used as specimens, remove the reinforcements with a scalpel or other suitable tool so that only elastomeric material is used in the test. Punch or cut (cork borer) a 2 - 4 mm diameter hole near the center of one of the short edges. If desired, notches or corners may be cut for identification.
- 5.2 Determine the initial hardness of the specimen according to ASTM D 1415 or D 2240, as required by the seal or elastomer specification.
- 5.3 Rub the cut specimen with cheesecloth slightly moistened with hexane to remove any mold-release compound which may be present on the surface. Remove any adhering cheesecloth fibers.

- 5.4 Determine the initial specific gravity and volume by water displacement. Determine the weight, M_1 , of the test specimen and the hanger wire (which is used for the water weighings) in air, to the nearest 1 mg. Using the hanger wire, dip the specimen successively in wetting agent and distilled water, draining momentarily after each dipping. Suspend the specimen from the balance hook; determine the weight, M_2 , of the specimen in distilled water which has been boiled 5 minutes and cooled to 20-25°C. (Although a correction for the buoyancy of the hanger wire would be required for precise determinations of volume and specific gravity, it is not applied in this method since its effect is insignificant.)
- 5.5 Blot the specimen dry with filter paper.
- 5.6 Calculate the initial specific gravity, SG, according to 6.1. Use only specimens that are within 0.010 of the specified specific gravity. If no specific gravity is specified, use only specimens that are within 0.010 of the mean specific gravity.
- 5.7 Distribute approximately 10 mL of the test grease in the bottom of the beaker. Using fingers, apply a thick coating of test grease on the elastomer specimen. Place specimen in the beaker and with a suitable spatula, pack test grease around the specimen, eliminating air pockets. Fill beaker to 80 mL mark with test grease. Cover with watch glass.
- 5.8 Store each sample in oven at $100 \pm 2^\circ\text{C}$ for 70 ± 0.5 hours, unless otherwise specified.
- 5.9 Check each sample daily to determine if specimens are in complete contact with test greases. Use a spatula to close any voids which may develop.
- 5.10 At the end of the test period, remove the specimens from the test greases using a forceps. Cool to room temperature.
- 5.11 Wipe off bulk of adhering grease with cheesecloth. Wipe off residual grease with cheesecloth liberally moistened with hexane. Dip specimens in methanol or ethanol and blot dry with filter paper.
- 5.12 Determine the weight of each specimen in air, M_3 , and that of each specimen in water, M_4 , as described in 5.4.
- 5.13 Calculate the volume change, ΔV , to the nearest 0.1 percent, of each specimen according to 6.2.
- 5.14 Measure the final hardness as in 5.2 and calculate change.
6. Calculations
- 6.1 Calculate the specific gravity as follows:
- $$SG = \frac{M_1}{M_1 - M_2}$$
- where: SG = Specific gravity
 M_1 = Initial weight of specimen in air, g
 M_2 = Initial weight of specimen in water, g
- 6.2 Calculate the change in volume as follows:
- $$\Delta V = \frac{(M_3 - M_4) - (M_1 - M_2)}{M_1 - M_2} \times 100$$
- where: ΔV = Volume change, percent
 M_1 = Initial weight of specimen in air, g
 M_2 = Initial weight of specimen in water, g
 M_3 = Final weight of specimen in air, g
 M_4 = Final weight of specimen in water, g
7. Report
- Report the following for each test run according to this method:
- 7.1 Test temperature and duration.
- 7.2 Date and grease sample identity.
- 7.3 Specimen identity and specific gravity.
- 7.4 Initial hardness and hardness change (points).
- 7.5 Volume change (percent).
8. Precision
- 8.1 Precision has not yet been determined.

About the Author



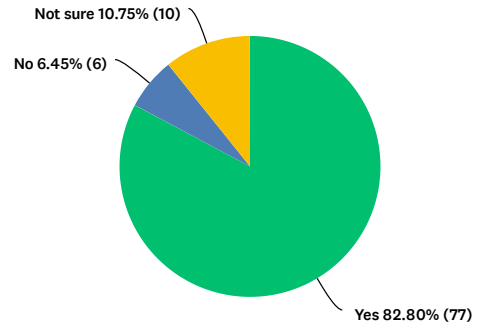
T. M. VERDURA

received a BS degree from the University of Detroit in 1961. He began work at General Motors Research Laboratories in 1955 investigating the properties and performance of lubricants. He is presently an associate senior research chemist in the Fuels and Lubricants Department where for the past 15 years he has been studying the performance of lubricating greases in automotive applications. He is an active member of ASTM, his company's Technical Representative to NLGI, and a previous contributor to the Spokesman.

SUSTAINABILITY SURVEY

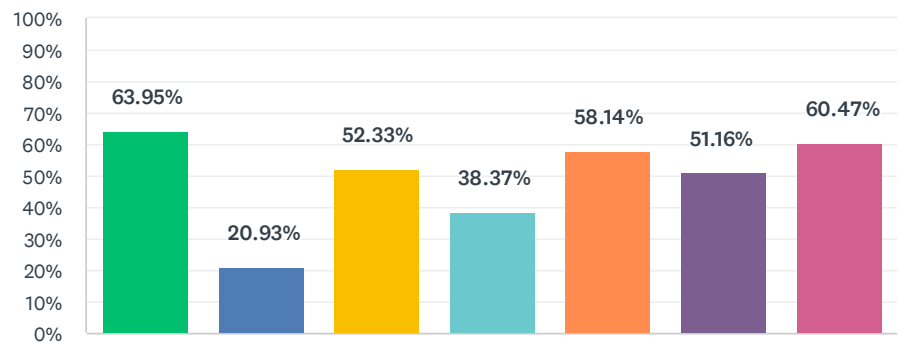
As you may have heard, sustainability is the theme of this year's NLGI Annual Meeting in Toronto, Canada, June 12-15, 2022. As NLGI explores sustainability further, we asked for your input on how our organization can assist you/your company in sustainability efforts. We received a number of responses and thank you in advance for your feedback. The data supports that NLGI should provide more resources to help members with their sustainability efforts. Below is a synopsis of the data received.

Should NLGI be providing more resources to help members with their sustainability efforts?

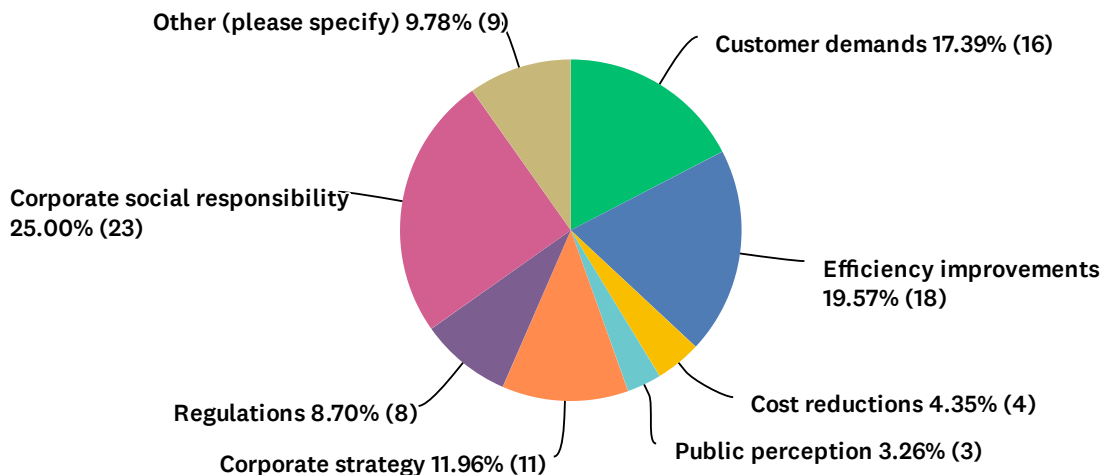


What areas should NLGI put more focus on?

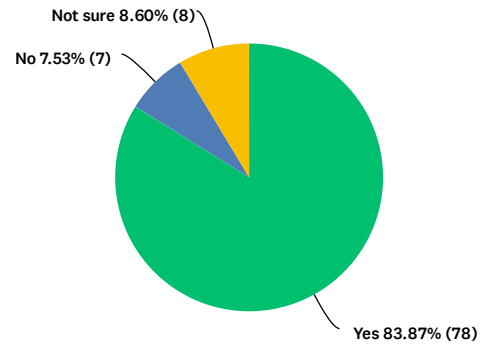
- Identifying/measuring footprint, lifecycle analysis
- Lobbying
- Energy efficiency/savings
- Offsetting carbon emissions
- Environmental/Health/Safety impact
- Forum for discussion (working group)
- Regular updates from experts in the field focused on sustainability - webinars, publications



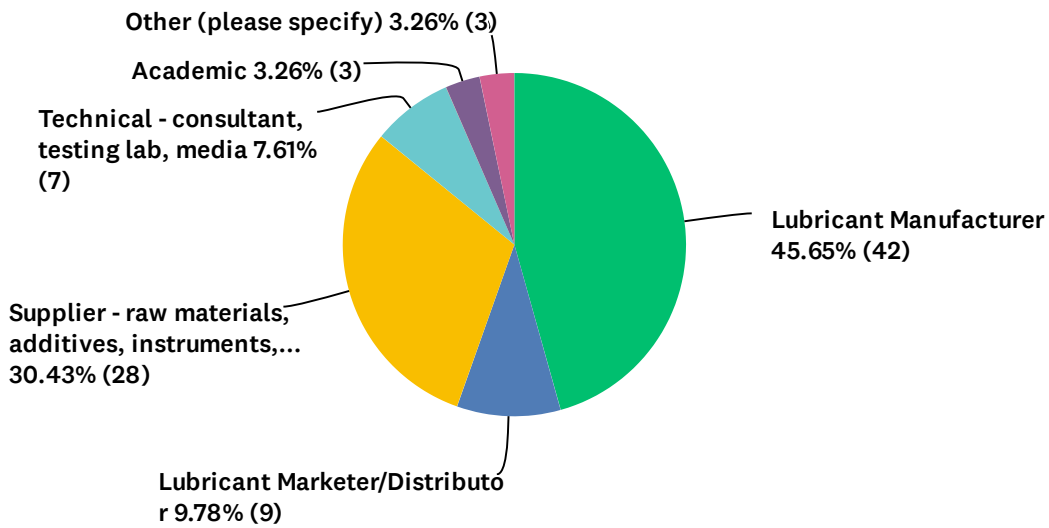
What is the most important aspect of sustainability for your company?



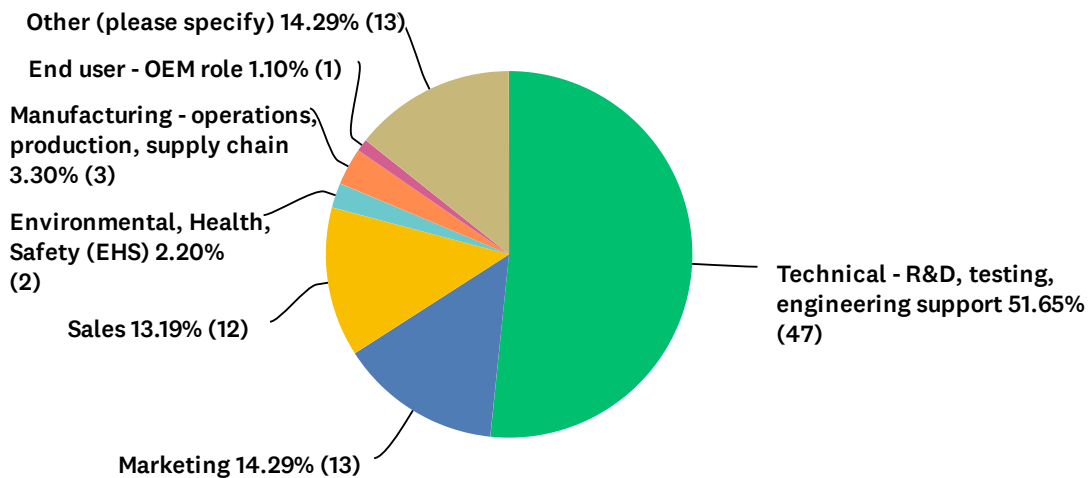
United Nations definition of sustainability states “meeting the needs of the present without compromising the ability of future generations to meet their own needs.? Do you agree?



What category does your company fall under?



What is your role within your company (ex: R&D, sales, etc.)?



How does your company define sustainability?

“Providing products to our customers that exceeds expectations while reducing environmental impacts of its operations, activities and services.”

“Responsible consumption and sourcing”

“Sustainability means environmental stewardship and protecting our planet, keeping people safe and healthy, and ensuring that businesses prosper.”

How do you define sustainability?

“The ability to consistently produce, manufacture, consume, and recycle while limiting the environmental and social impact as well as amount of waste produced”

“Sustainability is the ability to exist, develop, and progress while protecting natural resources.”

“Policy and practice designed to limit use of finite resources, while ensuring the health and growth of global communities today and into the future.”



**Thank you to all those who participated.
Your feedback was greatly appreciated.**

**NLGI will keep you apprised on
future developments related to sustainability.**



High-Performance Multiuse (HPM) Grease Column



[How Do I Apply for HPM?](#)



FIVE STEP CERTIFICATION PROCESS for Grease Manufacturers and Marketers

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

[How Do I Test my Products for HPM?](#)

Test Labs USA

Test Labs Non-USA

See below for additional information related to HPM including list of approved products, pricing and more!

Specification
Details & Testing

GC-LB Comparison

Webinars

Historical
Information

Certification &
Application Process

Renewal Audit
Process

Test Labs

HPM Approved
Products

Pricing

High-Performance Multiuse Grease

Easy to consolidate your inventory

NLGI



GREASE GUIDE (7TH EDITION)

CHAPTERS:

- A Historical Overview
- Characteristics of Lubricating Greases
- Testing of Lubricating Greases
- Formulation and Process Development ★ *New Chapter* ★
- Manufacturing Lubricating Greases
- Food Machinery Lubricants ★ *New Chapter* ★
- Handling and Dispensing Grease
- Grease Selection
- Troubleshooting Guide
- NLGI Product Certifications ★ *New Chapter* ★
- Environmentally Acceptable Lubricants (EALs) ★ *New Chapter* ★
- Grease HS&E Requirements

COMING IN 2022!



Contact nlgi@nlgi.org with any questions.



In Memoriam

NLGI Tribute

to Dr. Huafeng “Bill” Shen

Huafeng “Bill” Shen, 56, passed away peacefully in his home on Sunday, February 13, 2022 after a long and courageous battle with cancer. He was surrounded with loving family and friends by his side at the time of death.

Huafeng was born on February 25, 1965 in China, where he attended college at the young age of 15. Graduating with a degree in chemistry, he then went on to obtain a Master’s at Lanzhou University before moving to Beijing for work. There, he met and married his wife of 30 years, Yanxin Guan. Two years later, he relocated to the United States, where he obtained a PhD from the University of North Texas, after which he settled in New Jersey to work at Calumet as a research chemist, eventually leading an R&D department.

As a person, Huafeng was highly intelligent and well respected by his peers in the field. He was also a pillar of Monmouth Chinese Christian Church, a spiritual individual, with an unwavering faith in God, even in times of trouble. He was guided by a strong sense of morality, always willing to do what was right, and never seeking praise. He was honest and humble, charitable and kind, a true leader, offering gentle guidance to all who were in need.

Huafeng is survived by his wife, two sons, three siblings and many other loving relatives.

“Bill was a close friend for over 20 years. He was a wonderful, compassionate man that was idolized by many. Bill was one of the kindest souls I’ve ever met and he will be deeply missed.”

– Jim Hunt, Tiarco Chemical,
NLGI President

“Bill impressed his colleagues as knowledgeable, perceptive, hard-working, and very appreciative of his US citizenship. He was proud to work at Bel-Ray and dedicated to doing a good job.”

– Mary Moon, Presque Isle Innovations LLC



“Bill was always a pleasure to talk to, and always made me feel like I’d known him my whole life.”

– Chad Chichester,
MOLYKOTE® Specialty Lubricants

“We admired and respected his expertise, diligence, kindness and courage. He tackled problems with a smile, often saying “I take care of it” your loss is an emptiness that will last forever, we miss you Dr Shen, an extraordinary intellect and moral compass, we will never forget you. Our thoughts and prayers will always be with your family, Mary, Jerry, Michael and the rest of the Shen family.”

– Muibat Gbadamosi,
Calumet Branded Products

“The sadness we feel is only buffered by the wonderful gift of a legacy Bill shares with us in his passing. His humble intellect and expertise were overshadowed by his genuine kindness and courage. Bill so effortlessly stood for what you must work tirelessly for to build a culture of high standards, mutual respect and selflessness.

We take comfort in seeing all that Bill stood for reflected in Jerry and Michael, while our thoughts and prayers will forever be with Mary and the rest of his wonderful family. May Bill rest in peace – we miss him dearly.”

– Chris Creedon,
Calumet Branded Products

In Memoriam

NLGI Tribute to Arnold C. Witte, Jr.



Mr. Arnold C. Witte, Jr., 83, of Houston, Texas, passed away on Sunday, March 6, 2022, following a brief illness.

Arnold was born on July 20, 1938, in Bellville, Texas, to Leona and A.C. "Big Foot" Witte. He graduated from Henrietta M. King High School in Kingsville, Texas in 1956, then earned a Bachelor of Science degree in Chemical Engineering from Texas Tech University in 1961 and a Master of Science in Engineering from Lamar University in 1971. He spent his career working at Texaco's Port Arthur Research Laboratories, where he was a recognized expert in the field of lubrication and grease product and process development. He was instrumental in the laboratory development and subsequent commercialization of the Texaco Continuous Grease Manufacturing Process, regarded in the industry as an economical method of manufacturing large volumes of greases with reduced manpower and utility costs. He was also pivotal in the design and start-up of Texaco grease plants in the US, Brazil, Europe, and Africa, and developed innovative formulations of railroad lubricants.

Arnold's work for Texaco earned R&D's Outstanding Contributor Award on three occasions, and in 1995 he was named a Texaco Honorary Fellow by its Chairman/CEO and Vice Chairman in recognition of his achievements in technology and his exceptional contributions to the company and the industry. He authored numerous papers and presentations, received thirteen patents in the area of grease processing and product formulation, and was a contributing author of the Encyclopedia of Chemical Processing and Design. He was also a member of the National Lubricating Grease Institute (NLGI), where he was awarded three Clarence E. Earle awards, the Fellows Award, the Meritorious Service Trophy, and the Award for Achievement, recognizing his exceptional contributions to the growth and development of NLGI as an author, instructor, and long-standing member of the Technical Committee. Arnold was an outstanding mentor to many and continued to advance grease manufacturing technology and education globally after his retirement from Texaco in 1996.

Arnold met his future wife, Billie Jean (Guidry), while both were working at Texaco and they married in 1963. They enjoyed 57 devoted years together building a family, gardening, and traveling across the US, Europe, and Australia, and to one of his favorite places – British Columbia. He led an active life raising his two daughters and spending time with his grandson. Arnold was the rock of his family – we will always remember his strong mind, his fierce will, his adventurous spirit, and his kind and loving heart.

Arnold was preceded in death by Billie Jean in 2021, and is survived by daughters Ann Christine Witte and Catherine Witte, grandson Jonathan Tantillo, sons-in-law Jason Tantillo and Michael Shepherd, and brother David Witte.

I had the privilege to work for and with Arnold Witte for several years at the Texaco Research Laboratory. He was an ideal team leader and he fostered a great team environment - no politics, no micro-managing. He was an excellent mentor and coach and an absolute joy to work with. The years I worked with Arnold were some of the most satisfying of my career. I will miss him.

- Ricky Stamps, Retired

Arnold was both a mentor and friend. His great humility, unassuming manner and positive leadership style were wonderful traits to both observe and emulate. Many of his contributions to the grease industry are still practiced today. He will be greatly missed.

- Ed Fliss, Retired

Arnold Witte, ACW or "Ace" to many of his friends and co-workers, was my first boss upon entering the lubricants industry in 1981. He was not only my boss, he was my mentor in the grease industry, and, more than anything, he was my friend. Arnold taught me so much about grease formulation and manufacture. He, along with W. B. "Bill" Green invented the Texaco continuous grease manufacturing process, which I had the good fortune to become associated with. I learned about international travel from Arnold, including how not to pack too heavily, and how to navigate European train and subway schedules. We had great times together, with me absorbing it all. Arnold was an institution at NLGI for many years, teaching in the education courses and participating in the technical working groups. He was well-known and friends with just about everyone in his generation at NLGI. He received many awards from NLGI, including the Fellows Award in 1992 and the Award for Achievement in 2000. My best memories of Arnold Witte are of long days working on grease plant projects, along with fun times in interesting locations. More than anything, I already miss my friend.

- David Turner, CITGO Petroleum Corp.