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ON THE COVER
Happy New Year!

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Welcomes the Awards Committee

Patrick Walsh
NLGI Awards Committee Chair
President
Texas Refinery Corp

Recognition
noun rec-o-gni-tion | re-kig-ˈni-shən, -kəg-
Definition
1 : the action of recognizing : the state of being recognized: such as
   a : acknowledgment especially : formal acknowledgment of the political existence of a government or nation
   b : knowledge or feeling that someone or something present has been encountered before
2 : special notice or attention

Above is the definition of the word Recognition. This year the word RECOGNITION can take on added meaning, for you can play a key role in having an NLGI colleague recognized at the NLGI Annual Meeting in Las Vegas, Nevada.

Each year, at the NLGI Annual Meeting, distinguished individuals are recognized for their efforts in advancing knowledge and understanding in the grease industry. The NLGI strives to recognize and honor those who have furthered themselves, their company and this industry. Most likely you know of an individual who is deserving of special recognition. Make this the year that you spring into action by nominating someone for one of the following NLGI Awards:

- **NLGI Award for Achievement** – The Institute’s highest award honors the achievement of those who have made exceptional contributions to the growth and development of the Institute.
- **NLGI Fellows Award** – Acknowledges valuable work within the Institute, in the technical development of greases, grease tests, or the promotion of grease usage.
- **John A. Bellanti Sr. Memorial Award** – Acknowledges meritorious service on the NLGI Board, or on Technical Committee projects or to the industry.
- **NLGI Honorary Membership** – Entitles lifetime honorary membership to those who, over a period of years, have served the Institute in some outstanding capacity and are not now with a member company.
- **Award for Educational Excellence** – For outstanding instruction as exemplified by subject knowledge and presentation skills in NLGI educational courses.
- **NLGI Author Award (Development)** – For the best paper presented at our Annual Meeting that focuses on formulation, development, and manufacture of finished greases.
- **NLGI Author Award (Application)** – For the best paper presented at our Annual Meeting that focuses on testing, selection, application or use of greases.
- **Clarence E. Earle Memorial Award** – For an outstanding contribution to the technical literature relating to lubricating greases during the year.

If you have thought about nominating someone in the past, then now is the time to act! The next NLGI Annual Meeting will be held June 8-11, 2019 at the fabulous JW Marriott Resort & Spa in Las Vegas, Nevada. Imagine the pride you will feel should your nominee be recognized, among their peers, with an NLGI Award. Thank you in advance for your participation. We hope to welcome you to the JW Marriott Resort & Spa in June!

The 2019 deadline for nomination is March 1, 2019. NLGI will also accept nominations for 2020. Click here for awards nomination form. Click here.

Patrick Walsh, Chair
Chuck Coe, Joe Kaperick, Anoop Kumar, David Turner
RESEARCH GRANT INFORMATION

WELCOME, RESEARCHERS!

Founded in 1933, the non-profit National Lubricating Grease Institute (www.nlgi.org) has long supported the global lubricating grease industry with a variety of programs: annual meetings, technical publications, reference books, standards and manuals, education courses, professional and product certifications, and more. With that history in mind, the Institute has established a grants program to fund original scientific research into any aspect of lubricating grease technology. The Institute is committed to an open and fair process for soliciting, evaluating and awarding the yearly grants, as outlined below. We invite and encourage your participation.

Size of Grant Available: $1,000 to $50,000 per year, in part or whole, and potentially extendable to a second or third year, upon application and with NLGI Board of Directors approval.

Types of Research to be considered include fundamental grease chemistry; thickener systems; improved test methods; advances in processing technologies; and sustainability of grease production, materials or use. Pre-competitive research of interest to the wider grease community will be favored, and Researchers and NLGI Members are invited to bring desired research topics to the attention of the Research Grant Review Committee.

Researcher Qualifications:
• Any University or Non-profit Research Institute worldwide may apply.
• The research should be post-graduate level (master’s or doctoral) work.
• Applicants should download and complete the Research Grant Form and submit it to NLGI Headquarters, along with the lead researcher’s qualifications and references, a summary of the research work proposed and how it meets NLGI’s Main Criteria for research grants, and who will conduct it. In five to 10 pages, this summary should detail how the researcher plans to complete the proposed work. It should include a brief, clear description and timeline of required tasks; the budget for researcher support, equipment and consumables; and the research tools and in-kind contributions that the applicant may have available to successfully support his or her work. Also list any previous publications that support the proposed project and its feasibility.
• Only one application per lead researcher or group will be considered each year.
• NLGI being a non-profit technical institute, not more than 10 percent of the overall grant may be used by the selected institution for indirect, overhead and/or administrative costs.

NLGI Expectations:
• NLGI will appoint a liaison as principal contact to work with the researcher and facilitate communication.
• Regular, half-yearly progress reports (one-page) should be submitted to NLGI Headquarters.
• A Final Research Report is due not more than six (6) months after completion of the contracted research. Final Reports are to be made available to the membership on NLGI’s website.
• If a Master’s or PhD. thesis is written, an electronic copy of the final thesis should be provided to NLGI Headquarters and made available to the NLGI Membership.
• Any publications that result from this grant should name NLGI and its support in the publication’s acknowledgement section.
• It is expected that the research findings will be presented at an NLGI Annual Meeting. At its discretion, NLGI’s Board may consider granting a separate travel stipend to assist the researcher to attend the NLGI Annual Meeting to present his or her findings.
• Intellectual property developed under the grant is to be freely available to the public at no cost in order to expand the understanding of lubricating grease technology.

QUESTIONS? Contact NLGI at 816-524-2500 or nlgi@nlgi.org

ANNUAL SCHEDULE FOR APPLICATIONS AND AWARDS:
Applications will be accepted on-line from December 4, 2018 to March 15, 2019.

The Research Grant Review Committee will make its recommendation to the NLGI Board of Directors by May 31, 2019.

Recipient of the Grant will be notified by June 15 and pre-milestone first set of funds to be issued by August 2019 for research work to begin in the Fall of 2019.

Main Criteria on which proposals will be judged:
• The research must be related to lubricating greases.
• The research should contribute to the scientific understanding of grease in a unique and novel way.
• The research should be of impactful benefit to industry.
Abstract
Color contributes to the first impression upon viewing a grease product or sample. Although grease color does not provide specific technical benefits, it is an important indicator of grease quality from the perspective of many customers. Off-colored products typically lead to customer complaints and loss of confidence in the manufacturer/supplier. Currently, grease color is evaluated in a subjective manner by a simple visual appearance test. Due to the subjective nature of human vision and various light sources, grease color is perceived differently by each observer. This subjectivity leads to inconsistent approval or rejection of products during manufacturing. This study evaluated the Hunter ColorFlex instrument as a means to quantitatively measure grease color and replace subjective visual appearance tests for quality control of product color. This instrument measures color based on 3 parameters: light vs dark (L), red vs green (a), and blue vs yellow (b). These color data points are utilized to set up quality control standard parameters that allow for simple pass/fail testing of grease color. Implementation of quantitative grease color testing removes subjectivity, increases efficiency in matching product color, and eliminates product color concerns.

Prior to discussing how to test a product’s color, we first need to establish a basis of how color is perceived. Color theory has developed substantially over the years. Dating back to Pythagoras, Aristotle, and Plato, color has been a topic of discussion and research for a long time. In the 18th century, Moses Harris proposed the Color Wheel as an attempt to demonstrate how all colors are composed of the “primary” colors: red, yellow, and blue (RYB). RYB theory proposed that different mixtures of these 3 primary colors result in all other possible color variations. This became the basis for color vision theories, which were an attempt to explain the fundamental principle of sight. The RYB color vision theory was refined further to include “complementary” colors or contrasting blends of primary colors towards the middle of the 18th century (Figure 1).
In the early 19th century, explanations of how human eyesight functioned began to reshape color theory. The trichromatic theory, or Young-Helmholtz theory of color vision, stated that there are 3 color receptors in the human eye that are responsible for producing color and therefore eyesight. This idea was proposed initially by Thomas Young in 1802 and stated that the eye contained photoreceptor cells. These photoreceptor cells were sensitive to different wavelengths of light in the visible spectrum (400-700 nm). Hermann von Helmholtz expanded this idea in the mid-1800’s to state that photoreceptor cells are sensitive to different wavelengths of light: short-wavelength (blue), medium-wavelength (green), and long-wavelength (red). The brain interprets color based upon the strength of each wavelength that contacts the photoreceptor cells. Through experimentation, Helmholtz showed that humans can match colors when adjusting 3 wavelengths of light (red, green, blue), but they cannot match colors with only 2 wavelengths of light.

Researchers did not discover the 3 photoreceptors responsible for vision until more than 70 years after the Young-Helmholtz theory was proposed. These receptors are more commonly referred to as short-wavelength cones, middle-wavelength cones, and long-wavelength cones.

Alongside the trichromatic theory, the opponent-process theory was proposed by Ewald Hering in 1878. The opponent process theory states that vision is produced by 3 different receptor complexes in the eye: red-green, blue-yellow, and black-white complexes. Hering proposed this theory as he noted that there are colors the human eye does not perceive. Stated differently, the eye cannot see a bluish-yellow color as only blue would be detected and not yellow. The opponent-process theory functions through positive or negative neurological responses. Blue creates a positive response, whereas yellow produces a negative response on opponent neurons. These responses are produced by the various wavelengths of light. Although each theory appears to contradict the other, both theories are utilized to explain human vision. The trichromatic theory is the response to color at the receptor level in the eye, whereas, the opponent-process theory is the neurological response to color.

Hunter Color theory was developed by Richard Hunter in 1938. This theory provided the basis for the Hunter L a b color space proposed in 1948, which was later refined to become the standardized CIE L*a*b* color space in 1976. Hunter color theory was developed based upon the principles of the opponent-process theory. The Hunter L a b and CIE L*a*b* color spaces are 3-axis graphs with the red-green complex on the x-axis, blue-yellow complex on the z-axis, and black-white complex on the y-axis (Figure 2).

![Figure 2 Image courtesy of HunterLab](image)

The L*a*b* color space utilizes L* for the lightness/darkness value, a* for the red/green value, and b* for the blue/yellow value. An L* value of 100 is perfectly white, whereas an L* value of 0 is perfectly black. Positive a* values refer to red, and negative a* values refer to green. Positive b* values are in the yellow region, and negative b* values are in the blue region of the color space.

This color space allows for perceived color to be converted to numerical values. Measuring color with a colorimeter relies on 3 requirements: a white light source (standard illuminant), an object to be measured, and a human observer (standard observer). Quantifying color in a consistent and accurate manner requires all 3 to be standardized.
The International Commission on Illumination (CIE) introduced the A, B, C, D, E, and F standard illuminants in 1931. The purpose of these standard illuminants is to provide a means of representing different light sources using a table of numbers of energy vs wavelength. A, B, and C represent average incandescent light, direct sunlight, and average daylight, respectively. Illuminant D represents a different phase of daylight, E is an equal energy illuminant, and F represents different fluorescent light sources (Figure 3). CIE advises that all colorimetric calculations requiring representative daylight utilize Illuminant D65, which is a standard light source developed by CIE. The standard illuminants allow for the white light source to be standardized across colorimetric calculations.

Quantifying an object to be measured is a relatively simple task, as humans use a diffraction grating within spectrophotometers to separate light into its individual wavelengths. Objects will modify light by absorbing or reflecting various wavelengths of light. Humans perceive the reflected light as the color of the object. A colorimeter operates in the same manner and uses reflected light to produce a reflectance spectrum. The reflectance spectrum can then be utilized in colorimetric calculations. Figure 4 shows an example of a reflectance spectrum of leaves during different seasons of the year. During summer, the leaves reflect more light in the 500-600 nm wavelength range, making them appear green. In the fall season, leaves begin to reflect light in the 600-700 nm range and reflect almost zero wavelengths below 550 nm, making them appear red. The reflectance spectrum makes it possible to quantitatively measure the color of an object based on calculations using the wavelengths of reflected light.
The final requirement for colorimetric calculations was the standard human observer. Some experimentation was necessary to determine how to accurately standardize human observers. In 1931, CIE developed the so-called 2° standard observer. When developing the 2° standard observer, CIE conducted experiments to quantify the ability of the human eye to perceive color. A participant was asked to look at a white screen through a hole allowing for a 2° field of view (Figure 5).

![Figure 5 Image courtesy of HunterLab](image1)

A black partition was placed in the middle of the screen and ½ of the screen was illuminated by a test light. The test light included a filter so that the screen would show a certain color. The participants adjusted 3 primary colored lights, red, green, and blue, to match the test light color. The 2° field of view was chosen as it was understood, although incorrectly, that the response of a human eye to color was contained within a 2° arc inside the fovea. The fovea (part of the retina) has the highest concentration of photoreceptor cones in the human eye.

Once it became known that receptor cones extended further than a 2° arc, the standard observer experimentation was repeated with a 10° field of view in 1964. Color perception with a 10° field of view showed higher intensity across all three primary colors, red, green, and blue (Figure 6). In the figure below, the dotted lines show the 10° standard observer color perception in comparison to the 2° standard observer depicted by the solid lines. CIE recommends the 10° standard observer for colorimetric calculations as it closely correlates to average human visual perception.

ColorFlex colorimeters from Hunter Associates Laboratory, Inc. (HunterLab), Reston, VA, USA utilize all three of the above standards, the white light source, object to be measured, and the standard observer, as a means of measuring color and calculating numerical values. The numerical calculation produces CIE tristimulus color values of x, y, and z (Figure 7).

![Figure 6 Image courtesy of HunterLab](image2)
The tristimulus values are calculated by multiplying the standard illuminant by the reflectance spectrum, which produces the average visual stimulus. The visual stimulus is then multiplied by each of the red (x), green (y), and blue (z) standard observers producing the CIE tristimulus x, y, and z color values. The tristimulus values can be converted to either Hunter L a b or CIE L*a*b* values with simple mathematics. The reasoning behind moving from x, y, z color values to L*a*b* is due to the simplicity of understanding the color scale and to better represent color differences in terms of saturation and hue. The CIE tristimulus values do not capture saturation or brightness. Utilizing L*a*b* allows for the calculation to provide values that simulate human vision on a scale that is easily understood. Figure 8 compares the tristimulus scale to the L a b scale.
Grease Color Measurement

The grease industry is currently without any quantitative method for determining the color of a grease. The oil industry relies on ASTM D1500 Standard Test Method for ASTM Color of Petroleum Products. D1500 provides an ASTM Standard color scale with a rating scale of 0.5-8 in 0.5 increments. The observer compares the sample versus the standard in a two-field or thee-field viewing instrument. This method works well for oil samples that range from colorless to dark brown; however, it does not address the multitude of available grease colors. ASTM D1500 also requires that the sample be translucent, so that light can illuminate the sample for the observer. This causes a concern for greases as some products are not translucent due to the thickener system and/or solid additives such as graphite or molybdenum disulfide.

For grease, the only method available is a qualitative appearance test which requires storing physical color standards of each product. The *NLGI Lubricating Grease Guide Sixth Edition* states “that a lubricating grease in an opaque container can be observed under reflected light from a position approximately perpendicular to the surface. Colors of lubricating greases are best described in terms of the predominant hue such as amber, brown, green, red with a qualifying adjective describing intensity in terms of light, medium, or dark.” But this qualitative color test is too subjective for lot to lot comparisons. Variations in raw materials, QC technicians, lighting, age of color standards, and grease containers all play a role in how the color of a grease is observed visually. Humans also perceive color differently based upon age, gender, stress, fatigue, and mood. These variations can cause the color of a grease to drift away from the true standard color of the product. Removing the subjectivity in color testing is the goal behind ColorFlex testing and equipment.

The ColorFlex produces color values using either Hunter L a b or CIE L*a*b* units. The instrument allows for different standard illuminants and either the 2° or 10° standard observer. The recommendation for testing greases with a ColorFlex is to use standard Illuminant D65 and the 10° standard observer. Figure 9 shows the ColorFlex screen as seen in the Chemtool QC lab setup for D65/10° and reporting values in L*a*b*. The instrument does allow for either the illuminant or the observer to change; however, these test conditions are optimal for the closest match to human color perception.

The ColorFlex instrument operates by emitting a flash of white light onto the sample’s surface. The sample reflects light back to the spectrophotometer, which generates the reflectance spectrum. The recommended ColorFlex setup is either a 45°/0° or 0°/45° directional geometry. Directional 45°/0° geometry is illuminated at a 45° angle to the sample and the spectrophotometer is at 0° or perpendicular to the sample surface. Directional 0°/45° geometry is the exact opposite. Illumination occurs at 0° or perpendicular to the sample surface and the spectrophotometer is at a 45° angle to the sample surface. Figure 10 displays the directional geometries.
The directional geometry is critical to proper color measurements as this geometry avoids the specular reflection from the sample surface. Specular reflection is more commonly referred to as gloss. When shining a light onto any surface, specular reflection causes a glossy sheen that distorts color for the observer. Most observers tilt the surface or the light source to eliminate this specular reflection and observe the true surface color. The 45°/0° and 0°/45° directional geometries are purposefully designed so that the specular reflection is not included in the colorimetric measurements.

The ColorFlex test can be described as a QuEChERS method or quick, easy, cheap, effective, rugged, and safe. ColorFlex testing requires a sample size of 25-30 grams of grease when using disposable Petri dishes as sample containers (Figure 11). The test is non-destructive, and test time is approximately 2-3 minutes including sample preparation.

**Calibration**

Any piece of lab equipment is useless without proper calibration. The ColorFlex instrument calibration is quite simple. The instrument is supplied with 2 NIST traceable standard tiles: white and black. These white and black tiles have specific L*a*b* values and are used as references when the instrument is recalibrated. Calibration is a straightforward process that takes approximately 1-2 minutes. Instrument calibration frequency can be done on a time schedule or after a set number of samples are analyzed. Between calibrations, the instrument is also supplied with a green tile that can be used to check that it is within specification.

**Sample Preparation**

After calibrating the instrument, the sample must be prepared for measurement. Preparing a sample for measurement requires that the sample be:

- Prepared in the same, repeatable manner
- Representative of the entire product
- Placed into a clear and colorless container, free from scratches or surface obtrusions
- Between 76-78°F (25°C +/-0.5°C)
- Free from entrapped air

Figure 10 Image courtesy of HunterLab
Sample Containers

Figure 11

Disposable Petri dishes are suitable sample containers due to being clear and colorless. A round, flat Petri dish measures 60 mm x 15 mm and is composed of polyethylene, which allows for optical clarity. Petri dishes with other diameters can be used so long as the sample viewing port on the instrument is fully covered by the sample.

Depth of the sample container should be a minimum of 10 mm. The test requires that light be reflected from the sample and back to the detector. A sample container that is thinner than 10 mm allows light to pass through the sample, which produces inconsistent measurements. Petri dishes deeper than 10 mm allow for enough sample to reduce the light escaping through the grease. Exceptional care should be taken when storing Petri dishes as their surfaces can become scratched. Once its surface has become scratched, a Petri dish can no longer be used as a sample container because the scratches will distort light, thus invalidating the test results.

The simplest means of filling a Petri dish with grease is to use a small, flat bladed spatula. Similar to filling an ASTM D217 worker cup, care must be taken when
filling the Petri dish. The operator must avoid air pockets in the grease as they will alter test results. A sample should be properly deaerated prior to testing.

**Measuring Color**
The sample in a Petri dish is centered over the sample viewing port so that the test can begin. The sample viewing port must be completely covered by the Petri dish in order to obtain valid test results. After running the test, a report (Figure 12) is generated. Figure 12 contains multiple elements that can be evaluated after completing a test. The ColorFlex report contains the L*a*b* color space in the top right, data table with L*a*b* values in the top center, the sample name on the far left, and the reflectance spectrum at the bottom.

As previously stated, physical grease color standards are typically required to be maintained for qualitative color tests. In Figure 12, a red rectangle is placed over the L*a*b* color space. The rectangle represents the minimum/maximum a*b* values that a sample can be within and still meet the intended color specifications. Again, the a* value represents the red/green axis, and the b* values represents blue/yellow axis. The L* value parameter is represented by the black/white bar to the right of the color space. Minimum/maximum L* values are shown as red lines on the black/white bar. The physical color standard can be replaced by the L*a*b* specifications, ensuring the batch meets the intended color parameters every time. Individual sample test runs are depicted by a small red or green dot.

![Figure 12 Image courtesy of HunterLab](image)

**Effect of Air on Grease Color**
Figure 13 compares a sample with minimal air (left) and a sample that was purposefully aerated (right) to show the effects of entrapped air on color. The entrapped air sample has a significantly higher L* value showing that air causes the sample to become much lighter in color. Along with a higher L* value, the a* and b* values also increase, making the aerated sample appear in the yellow region of the color space while the sample without air is in the blue color space. Likewise, the air pockets increased the a* value and moved the sample color towards the red region of the color space.
Effect of Temperature

When testing grease color, samples should all be held at the same temperature. The color of a grease typically becomes darker as it cools. This appears to be a function of the grease’s density. Cooler grease has a slightly higher density than warmer grease. As the density of the grease increases, its color becomes more concentrated, and it is darker. Following ASTM D217, it is recommended to test all grease samples for color at 76-78°F (25°C +/- 0.5°C). This allows for simplicity in testing because as a grease is cooled prior to performing ASTM D217, a small amount of cooled grease can also be utilized for color testing. Figure 14 shows the difference in L*a*b* values of a grease at 77°F (right) and at 180°F (left). In this study, 180°F (82°C) was chosen as an example test temperature simply to showcase the effect temperature has on grease color.
Quality Control
The standard for this product has the following QC specification ranges: L* 0-18.26, a* 4.25-28.59, and b* -4.64 – 4.86. For a sample to pass the ColorFlex test, every value must be within these specified ranges. If a value falls outside of a range, the sample fails the test and does not match the intended standard color.

Figure 15 displays two red greases that appear to the eye to match in color. When tested on the ColorFlex instrument, the sample on the left (Sample 1) failed the L* value by being too light. The small dot placed in the ColorFlex color space turns red upon measuring a result outside the standard specifications.

Figure 15 displays a scenario that has a high likelihood of occurring in grease production. A batch of grease is produced and is measured to be too light in color. With this information, the manufacturer can adjust the color of the batch prior to packaging. In the above case with Sample 1, the manufacturer would add an extra, small portion of red dye. The extra red dye treat would bring the batch further down into the red region of the color space. Extra dye would also darken the batch, bringing the L* value within the standard specifications. After the addition of the extra dye, the batch would be sampled and tested again with the ColorFlex. If the batch passes, it would be approved. The above scenario works well when a grease is too light in color. If a product is too dark due to processing or raw material issues, another approach may be necessary.

Conclusions
This study delivers reasons for the grease industry to adopt the ColorFlex as a standard method for testing grease color. ColorFlex instruments are simple to set up, run, and calibrate, relatively maintenance free, and low cost. This system is currently in use by Quality Control at Chemtool, Inc.

A ColorFlex has been used to adjust the color of in-process production batches to meet the required color specifications at Chemtool. Utilization of this equipment has reduced customer color concerns and cases where product was returned simply based on color issues.
When a color concern arises, it can be handled using objective L*a*b* data rather than subjective visual data provided by the customer. The L*a*b* report can be shared with a customer, outlining the product meeting the standard color specifications. Objective measurements with the Colorflex system remove the ambiguity a customer may have when viewing products.

When an entire portfolio of products has objective L*a*b* specifications, customer color concerns should become non-existent. Eliminating concerns based upon color would allow the grease manufacturer to focus on other processes without having to use resources to resolve quality concerns based on product color.

Acknowledgements
The author gratefully acknowledges Saurabh Lawate and Jeff St. Aubin for their help and support of this study.

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VOLUME 82, NUMBER 6
Environmentally Acceptable Grease That is Water Resistant and Provides Extreme Pressure Protection for Use in Marine and Industrial Applications

Doug Adams
RSC Bio Solutions LLC
Indian Trail, NC USA

Abstract
In 2013, the US Environmental Protection Agency (EPA) enacted Vessel General Permit (VGP) regulations for large vessels operating in US territorial waters. VGP mandates the use of Environmentally Acceptable Lubricants (EALs), motivating the lubricant industry to develop compliant products. A VGP compliant lubricant must be biodegrade by >60% over a 28-day period, not bioaccumulate and be non-toxic to several specified aquatic organisms. This paper presents test results for a new EAL grease formulated with lithium complex thickener and PAO and related base fluids. Test results show that this new EAL grease performs at a level required for marine and many industrial applications.

Base Fluids
Due to the fact that grease is primarily composed of base fluid, the industry had to initially focus their development programs on improving the base fluids for EALs. The base fluid imparts several critical characteristics of the base grease: it can provide lubrication, act to solubilize the additives, affect low temperature characteristics and either inhibit or promote thickener development. It can also impact the lubricant’s ability to remain in the application.

The thickener and additives used in grease are selected to ensure the finished grease meets the operating temperatures and performance requirements encountered in the desired application.

The EAL greases initially available to the market utilized plant-derived base fluids. Greases made from these triglycerides (TG) can have exceptional antiwear characteristics and can be improved with additives to meet many performance parameters. Some TG base fluids developed for this industry are made from soybean and rapeseed oils. The concerns about the triglyceride-based greases are primarily related to their oxidative stability over time or when they are exposed to heat. In addition, TG fluids are prone to hydrolysis.

Ester-based lubricants remain popular and were state of the art technology several years ago when a significant number of additives were developed to improve their performance characteristics. Greases made with synthetic esters can perform in a manner similar to many mineral oil-based greases, but only if they are not exposed to water.

Synthetic esters possess good frictional characteristics and can also have high viscosity indices, however they are also prone to oxidation and hydrolysis in the presence of heat and water.

During the hydrolysis process, the oil viscosity begins to fall. This decrease in viscosity poses a very real threat to equipment life. As the viscosity drops, the fluid’s load-carrying capacity diminishes, resulting in the equipment operating in a boundary lubrication regime and more pronounced wear. The esters also break down in the presence of water and heat to their constituent acids and alcohols, which also increases the acidity level in the application. This increase in acidity can lead to corrosion.

Polyalkylene glycols (PAG) are synthetic base fluids; some are fire resistant. Typically, they are incompatible with most other base fluids. In the presence of water, PAGs can react and generate acids, which can also lead to corrosion in many applications.
Polyalphaolefins (PAO) and related fluids (PR) possess excellent hydrolytic stability, long life and are most like the traditional mineral oils (MO) currently in use.

It is generally accepted that lubricants made with 100% Group I and Group II MOs provide the best cost/performance profile. However, these mineral oils do not satisfy VGP criteria for EALs.

The fluids previously described have been known to be used together in order to optimize the performance of the lubricant while maintaining the requirements of an EAL.

**EAL Grease**

The lubricant industry has focused in the past on how to provide the best product at the lowest price, which involved using additives containing heavy metals like lead and antimony to provide exceptional extreme pressure protection at relatively low treat rates.

The first generation of environmentally friendly greases was made with TG base fluids that were difficult to manufacture, especially if the grease had to be heated significantly during the manufacturing process. If a thickener structure formed and the grease was thinned to the appropriate penetration, the grease age hardened out of the targeted penetration grade. In addition, there was a limited number of performance additives that could safely be used in formulations to ensure that the grease was biodegradable, not toxic and not bioaccumulative.

Since that time, the grease industry has been improving the performance of the greases used in environmentally sensitive areas due to the introduction of new base fluids and additives, as well as the publication of information that describes the levels that base fluids and additives can be used in lubricant formulations.
Performance Test Data
Table 1 compares the strengths and shortcomings of a standard lithium thickened grease made with MO versus the new lithium complex grease that utilizes PAO and PR.

<table>
<thead>
<tr>
<th>Tests</th>
<th>Lithium Grease made with MO</th>
<th>Lithium Complex Grease made with PR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Penetration, worked 60, mm/10</td>
<td>263</td>
<td>275</td>
</tr>
<tr>
<td>ASTM D 217</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dropping Point, °C</td>
<td>187</td>
<td>280</td>
</tr>
<tr>
<td>ASTM D 2265</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Low Temperature performance,</td>
<td>7.76</td>
<td>1.87</td>
</tr>
<tr>
<td>torque at -40°C, Nm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ASTM D 4693</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weld Point, kgf</td>
<td>250</td>
<td>400</td>
</tr>
<tr>
<td>ASTM D 2596</td>
<td></td>
<td></td>
</tr>
<tr>
<td>EP Performance: LWI, kgf</td>
<td>39</td>
<td>65</td>
</tr>
<tr>
<td>ASTM D 2596</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-Ball Wear, scar diameter, mm</td>
<td>0.47</td>
<td>0.44</td>
</tr>
<tr>
<td>ASTM D 2266</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Timken EP, OK Load, Lbs.</td>
<td>55</td>
<td>55</td>
</tr>
<tr>
<td>ASTM D 2509</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water Spray off, % loss</td>
<td>42.5</td>
<td>4.0</td>
</tr>
<tr>
<td>ASTM D 4049</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water Resistance at 80°C, % loss</td>
<td>3.5</td>
<td>4.0</td>
</tr>
<tr>
<td>ASTM D 1264</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oil Separation, % loss</td>
<td>2.4</td>
<td>0.9</td>
</tr>
<tr>
<td>ASTM D 1742</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rust Protection, Rating</td>
<td>Pass</td>
<td>Pass</td>
</tr>
<tr>
<td>ASTM D 1743</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Copper Corrosion,</td>
<td>1b</td>
<td>1b</td>
</tr>
<tr>
<td>ASTM D 4048</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Salt Fog test 2S result after</td>
<td>2S result after</td>
<td>10 out of 10 after</td>
</tr>
<tr>
<td>B 117</td>
<td>264 hours.</td>
<td>1,000 hours</td>
</tr>
<tr>
<td>Humidity Cabinet</td>
<td>Test in Progress</td>
<td>10 out of 10 after</td>
</tr>
<tr>
<td>ASTM D 1748</td>
<td></td>
<td>1,500 hours</td>
</tr>
</tbody>
</table>

Table 1. Comparison of lithium greases

The MO lithium grease, which is advertised with an NLGI LB badge, and the environmentally acceptable lithium complex grease made with PR base fluid both possess good extreme pressure characteristics. Both greases achieved a 55 lb. Timken pass, and both greases have four ball weld results that are 250 kgf or greater.

The PR lithium complex grease has a thickener content of ~15%, and its viscosity is >100 cSt @ 40°C. The MO lithium grease is believed to have a thickener content of ~8% and an oil viscosity of 180 cSt @ 40°C. This MO grease was chosen for comparison as it had the performance profile of greases that were commonly used in marine applications prior to the enactment of the 2013 VGP.

Both greases performed well in the four ball weld tests; the PR grease had a LWI result of 65 kgf, while the MO grease had a 39 kgf LWI. Both of the greases met the minimum steel and copper corrosion test requirements.
The low temperature torque test results at -40°C for both greases were below the maximum allowable limit of 15.5 N.m that is described in some specifications. However, the low temperature torque result for the PR grease was lower than the MO grease by 5.89 N.m.

The four ball wear results of the two greases were comparable at 0.44 mm (PR) and 0.47 mm (MO).

The water resistance performance of the greases when indirectly exposed to water were comparable; the oil separation results were also similar.

Significant differences were noted in the performance of the greases in the water spray-off test, which measured how well the greases adhered to the metal surface when directly sprayed with water. The PR grease had an exceptional water spray-off result of 4% while the MO grease had an average result of 42.5%.

Table 2. Additional results for lithium complex grease made with PR

<table>
<thead>
<tr>
<th>Test</th>
<th>Requirement</th>
<th>Lithium Complex Grease made with PR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weld point, kgf</td>
<td>350 minimum</td>
<td>400</td>
</tr>
<tr>
<td>ASTM D 2596</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LWI, kgf</td>
<td>50 minimum</td>
<td>65</td>
</tr>
<tr>
<td>ASTM D 2596</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Timken, OK load, lbs</td>
<td>45 Pass minimum</td>
<td>55 Pass</td>
</tr>
<tr>
<td>ASTM D 2509</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Salt Fog</td>
<td>1,000 hours Pass</td>
<td>1,000 hours Pass</td>
</tr>
<tr>
<td>B 117</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Humidity Cabinet</td>
<td>1,500 hours Pass</td>
<td>1,500 hours Pass</td>
</tr>
<tr>
<td>ASTM D 1748</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The salt fog and humidity cabinet results for the PR grease show that this grease can withstand severe corrosion conditions typically encountered in marine and industrial environments.

When making the decision to move from a mineral oil-based grease to an EAL, the specific application for which the grease is targeted should be taken into consideration.

The performance of the new EAL grease meets or exceeds the requirements advertised for an effective wire rope grease. The salt fog and humidity cabinet results were 10 out of 10 after the timed test, which represents the fact that there was no corrosion in the test area.

The new PR grease performs at a level required for many marine and industrial applications.

Conclusions

The 2013 VGP guidelines provide an option for vessel operators to not use an EAL if it is not technically feasible, i.e., EALs that do not perform at the same level as mineral oil-based lubricants. Technology now exists that provides stable and effective greases that meet the VGP requirements. This paper provides test results for a new EAL grease based on a readily biodegradable base oil that is currently being used successfully in marine and industrial applications.

Acknowledgements

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Dr. Larry Beaver – Vice President, Research & Development
George Cook – Senior Applications Product Manager
Anne Otto – Senior Applications Development Manager
Moving Forward...
Can Lubricating Grease Be Produced in a More Efficient Way?

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² STRATCO, Inc. – Scottsdale, Arizona (USA)

Abstract
Replacement of lithium based thickeners is, of course, on the agenda of most grease manufacturers, although it’s easier said than done. It has been shown that it is possible to minimize the consumption of the lithium hydroxide in these thickener types by using naphthenic oils and/or a combination of naphthenic and paraffinic oils in pressurized vessels without compromising the performance of the final grease product.

During the last two decades, much effort has been put into the adaptation of grease formulations to make them more environmentally “friendly”. Toxic components are gradually being phased out, although there is still much work left to do, especially on the additive side. However, this study investigates the impact of raw materials on another aspect of environmental friendliness, namely energy savings during grease manufacturing. Based on the outcomes of this study, these authors suggest that the grease industry should minimize soap content in order to reduce the energy consumption per unit weight of produced grease as well as minimize the impact of the price fluctuations of lithium hydroxide. Both the environment and commercial enterprises will enjoy benefits from lower soap content in lithium and lithium complex greases.

Key words: Naphthenic oil, Paraffinic oil, Solvency power, Lithium greases, Energy consumption, Pressurised reactor

Introduction
According to the NLGI Grease Production Survey (calendar year 2016), conventional lithium grease and lithium complex grease represented 55 and 20 percent, respectively, of the global grease production (see Figure 1)¹. In 2017², these percentages were close to those in 2016.

Figure 1. Global Grease Production (2016) by Thickener Type
Furthermore, the growth trend documented in the NLGI Grease Production Surveys from 2008 to 2016 (see Figure 2) shows that lithium thickener greases have maintained their dominance and continued to grow in global production. However, lithium cost has been dramatically impacted in recent years due to demands from competing industries.

![Figure 2. Global Lithium Grease Production (2008 to 2016)](image)

According to a recent report that was published in April 2018 in *Lubes’n’Greases Magazine*, the lithium price in 1998 was about 2,000 USD/tonne; however, in 2017 it was almost 14,000 USD/tonne. The increased production of battery driven vehicles seems to be the main reason behind this price increase. The grease industry consumes almost 7 percent of the manufactured lithium metal, while the EV industry consumes more than 50 percent of this metal. The continuing increase in production of battery driven vehicles will create a larger demand for this precious light metal resulting in higher costs for the grease manufacturers.

Replacement of lithium based thickeners is, of course, on the agenda of most grease manufacturers, although it’s easier said than done. The popularity of this thickener type has resulted in significant investments in certifications, formulations and procedural developments that are not easily set aside. However, it is possible to minimize the consumption of lithium hydroxide in these thickener types by using naphthenic oils and/or a combination of naphthenic and paraffinic oils in pressurized vessels without compromising the performance of the final grease product. Previous studies have demonstrated, based upon commercial experience, that manufacturing lithium base grease concentrate in a pressurized Contactor™ reactor can provide cost savings for both raw materials (thickener) and energy (see Table 1). A goal of this study is to determine if further cost savings can be obtained in both categories by using naphthenic base oils.

During the last two decades, many grease manufacturers have focused on developing grease formulations that are more environmentally friendly. This focus has been primarily aimed at the application of the products. Although toxic components are gradually being phased out, there is much work left to do, especially on the additive side. But, surprisingly, relatively little work has been done to study the impact of using more suitable raw materials on reducing energy consumption and use of non-renewable resources in the grease manufacturing process. These issues will be considered in this study.
The aim of this paper is to measure the soap concentration and the energy consumption of conventional lithium greases based on hydrotreated naphthenic oil versus paraffinic Group I oil and their blends. The key parameters, such as time, temperature and pressure, remained constant during the manufacturing of each grease to ensure a meaningful comparison.

Experimental work
This study can be divided into three parts: the selection and the characterisation of the base oils, the manufacturing process and some data related to the lubricating greases.

The chosen base oils are among the most common types of fluids that are used in the grease industry, especially when it comes to the preparation of the lithium based greases, namely paraffinic SN525 and naphthenic T 110.

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Unit</th>
<th>Method / ASTM</th>
<th>SN525</th>
<th>T 110</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density @ 15°C</td>
<td>kg/dm³</td>
<td>D 4052</td>
<td>0.874</td>
<td>0.915</td>
</tr>
<tr>
<td>Viscosity @ 40°C</td>
<td>mm²/s</td>
<td>D 445</td>
<td>98</td>
<td>109</td>
</tr>
<tr>
<td>Viscosity @ 100°C</td>
<td>mm²/s</td>
<td>D 445</td>
<td>10.9</td>
<td>8.8</td>
</tr>
<tr>
<td>Flash Point, COC</td>
<td>°C</td>
<td>D 92</td>
<td>274</td>
<td>228</td>
</tr>
<tr>
<td>Pour Point</td>
<td>°C</td>
<td>D 97</td>
<td>-15</td>
<td>-30</td>
</tr>
<tr>
<td>Aniline Point (AP)</td>
<td>°C</td>
<td>D 611</td>
<td>119</td>
<td>87</td>
</tr>
<tr>
<td>Copper Corrosion</td>
<td>rating</td>
<td>D 130</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Sulfur content</td>
<td>wt.%</td>
<td>D 2622</td>
<td>0.08</td>
<td>0.11</td>
</tr>
<tr>
<td>Color</td>
<td>rating</td>
<td>D 1500</td>
<td>0.4</td>
<td>&lt;1.0</td>
</tr>
<tr>
<td>Total Acid Number</td>
<td>mg KOH/g</td>
<td>D 974</td>
<td>0.01</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>

Table 2. Typical characteristics of the two base oils

The characteristics of the two base oils in Table 2 reveal two important differences that may be appreciated by grease manufacturers: the pour point and the aniline point. The pour point for paraffinic SN525 is -15°C, while the pour point is significantly lower (-30°C) for naphthenic T 110. This means that T 110 will have a greater ability than SN525 to enhance the low temperature mobility of grease. The lower the aniline point, the higher the solvency. Naphthenic T 110 with aniline point of 87°C is a stronger solvent than paraffinic SN525 (aniline point 119°C).
The manufacturing process that was used in this study included a 6-gallon (22 litre) STRATCO® Contactor™ pilot plant reactor and a 10-gallon (38 litre) conventional finishing kettle. An electric thermal oil heater and pump supplied heat transfer oil for heating the product up to a peak temperature of 392°F (200°C). Product heating was controlled according to typical commercial practices and was identical for the test batches in this study. The base grease was manually transferred to the finishing kettle to allow the product to be weighed for an accurate determination of soap content. From the finishing kettle, the product was recirculated through a Model G-5 colloid mill and back to the kettle. The equipment configuration is shown in Figure 3.

![Figure 3. The Pilot Plant](image)

The addition of a viewing port to the Contactor reactor lid (Figure 4), allows the grease flow to be observed during pressurized operation. Internal recirculation is shown in Figure 5. Monitoring and maintaining grease flow are important to avoid local overheating during the production process. Proper flow and mixing of the experimental greases required the use of the full base oil requirement of the formula during the reaction stage. Non-visual indicators of good flow included the Contactor motor amperage and product temperature rise during heating.

![Figure 4. Top View Port](image) ![Figure 5. Grease Flow Inside Reactor](image)
The greases produced were:
Grease A composed of 100% T 110 naphthenic base oil.
Grease B composed of 100% SN525 paraffinic base oil.
Grease C composed of 50% T 110 naphthenic base oil and 50% SN525 paraffinic base oil.

The procedures employed in the reaction phase (in the Contactor) were as follows:
1. Add initial base oil charge (approximately 22 lbs. = 10 kg).
2. Commence internal circulation at 50 percent speed (900 RPM).
3. Add 12-hydroxystearic acid and close lid.
4. Set product temperature setpoint at 170°F (77°C) and raise speed to 100 percent.
5. When setpoint is reached, open lid to confirm acid is melted.
6. After confirmation, add lithium hydroxide (LiOH) and seal lid.
7. Raise setpoint to 392°F (200°C).
8. As temperature rises, monitor product temperature, thermal oil temperature, motor amperage and pressure.
9. As temperature rises, manually vent to regulate pressure between 85 and 95 psig (6 and 6.5 bar).
10. After product reaches the target temperature, slowly vent the pressure to atmospheric pressure and open lid.
11. Add quench oil (approximately 8.8 lbs. = 4 kg).
12. At 350°F (177°C), open drain valve to manually transfer and repeat until minimal flow, then turn off Contactor motor.
13. Close drain valve and add flush oil (approximately 28.6 lbs. = 13 kg).
14. Start Contactor motor and run at 100% speed.
15. After 5 minutes, drain and transfer flush oil, and then shut down heater and Contactor.

To ensure that the target soap content was achieved, the mass of the base grease plus the mass of the flush oil and residual grease was compared to the mass of the raw materials initially used. The loss percentage was applied to the original soap and base oil ingredients to calculate the balance of oil to be added to the kettle to maintain the original target soap content. To optimize the 50/50 Grease C, only naphthenic oil was used in the reaction phase, including quench, and paraffinic oil was added later in the flush.

The procedures employed in the finishing phase (in the kettle) were as follows:
1. After the transferred base grease is fully measured, the balance of the base oil required to bring the soap content to the target amount is added to the kettle.
2. The kettle is then closed and the agitator is started.
3. Due to the length of pipe between the kettle pump and the kettle outlet, the kettle is pressurized to commence flow.
4. The kettle pump is turned on and the material recirculated back to the kettle.
5. When recirculation is verified, flow is diverted through the colloid mill and then the mill is turned on.
6. After 30 minutes, the product samples are pulled and the kettle, pump and mill are shut down.
7. The kilowatt-hours of both meters are recorded.
Results and Discussion
The thermal (hot) oil supply temperature (HOS) and the product temperature profiles for the three greases are shown in Figure 6. These profiles illustrate the similarity in heating and temperature data for the three products. The batch size for all three greases was the same, 30 kg.

![Figure 6. Time versus Temperature for Greases A, B and C](image_url)

It is well known that more efficient grease production can be obtained by employing a pressurized reactor in the “cooking stage” of the manufacturing process. However, to the knowledge of the authors, the effect of the base oil on the efficiency of the grease production has never been published. Hence, it was interesting to explore the impact of the solvency power of the base oil on the energy consumption. As mentioned above, three conventional lithium greases with different base oil solvency were produced with the same batch size (30 kg), and all other process parameters were kept constant. Typical properties of these greases, along with the measured energy consumption, are illustrated in Table 3.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Method</th>
<th>Grease A</th>
<th>Grease B</th>
<th>Grease C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of Base Oil</td>
<td>.</td>
<td>Naph</td>
<td>Paraf</td>
<td>Naph + Paraf</td>
</tr>
<tr>
<td>Base oil</td>
<td>.</td>
<td>T 110</td>
<td>SN525</td>
<td>T 110:SN525 (1:1)</td>
</tr>
<tr>
<td>NLGI grade</td>
<td>.</td>
<td>3</td>
<td>2.5</td>
<td>2</td>
</tr>
<tr>
<td>Penetration (60), mm⁻¹</td>
<td>ASTM D217</td>
<td>239</td>
<td>258</td>
<td>272</td>
</tr>
<tr>
<td>Dropping Point, °C</td>
<td>ASTM D2265</td>
<td>206</td>
<td>207</td>
<td>209</td>
</tr>
<tr>
<td>Thickener content, wt.%</td>
<td>-</td>
<td>7.92</td>
<td>9.02</td>
<td>7.82</td>
</tr>
<tr>
<td>Thickener content (dry), wt.%</td>
<td>-</td>
<td>7.06</td>
<td>8.04</td>
<td>6.79</td>
</tr>
<tr>
<td>Energy Consumption, kWh</td>
<td>-</td>
<td>15</td>
<td>17</td>
<td>15</td>
</tr>
</tbody>
</table>

Table 3. Typical characteristics of the greases as well as the energy consumption
A comparison between Greases A and B, based on the data shown in Table 3, shows that Grease B has higher thickener content (a 14% difference) than Grease A, although the penetration after 60 strokes indicates that Grease B is 0.5 NLGI grade thinner or softer than Grease A. While the improved yield in greases with T 110 oil was expected, the lower energy consumption (2 kWh) was a surprise. Grease C, which was “cooked” with T 110 base oil and cooled with SN 525, was almost identical to Grease A in terms of energy consumption and thickener content, but C was an NLGI grade thinner or softer than A.

The pilot plant used for this study was a miniature version of the commercial scale versions that have been used worldwide for decades. The Contactor reactor on the pilot scale has been shown to have a high correlation to performance on commercial scales6. If the energy saving of 2 kWh / 30 kg grease determined in this study translates to the commercial scale, then it is possible to estimate how much total energy cost can be saved. A middle-sized plant may produce 3,000 metric tons (3,000,000 kg) of conventional lithium grease on an annual basis. Electricity at a location like Kansas City, USA will cost 6.43 ¢/kWh. Based on these factors, a grease plant will experience almost 13,000 USD in annual energy savings:

\[
6.43\,\text{¢/kWh} \times 2 \, \text{kWh} \times 3,000,000 \, \text{kg} / 30 \, \text{kg} = 1,286,000\,\text{¢} = 12,860 \, \text{USD}
\]

This figure does not include the extra savings from better yields using T 110 instead of SN525.

This analysis may be used as a guideline for grease manufacturers to perform their own investigations and verifications. It is obvious that industrial electrical utility rates differ in specific states in the USA. For example, the cost of electricity in Los Angeles is almost twice the rate in Kansas City, and certainly the rate might be higher or lower in various countries, as well.

**Summary**

Grease manufacturers have recognised that the use of pressurised reactors in the preparation of greases significantly reduces the production time and the soap content as well. However, this study showed that the use of naphthenic oils, such as T 110, demonstrated value versus paraffinic SN525. T 110 based greases made in the Contactor reactor showed better optimisation than SN525 based greases due to the higher degree of base oil solvency and a surprising reduction in energy consumption during the manufacturing process.

The savings in soap content represents both a raw material cost savings to the manufacturer and a reduction of demand in lithium, a finite, critical and costly natural resource. Similarly, the reduced energy during production represents both a utility cost savings to the manufacturer and reduced demand on the electrical infrastructure. These benefits represent more environmentally friendly means of producing lubricating greases.

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NLGI Grease Production Survey presented at the 84th Annual Meeting, June 2017, Olympic Valley, CA (USA).
NLGI Grease Production Surveys presented at the NLGI Annual Meetings, calendar years 2008 through 2016.
Lubes’N’Greases Europe-Middle East-Africa, April 2018
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2008  Jaime Spagnoli, ExxonMobil
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