WHITE PAPER

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CALCIUM SULPHONATE GREASES

Performance and application overview

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CALCIUM SULFONATE GREASES

by Dr Gareth Fish

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Calcium sulfonates are the fastest growing grease thickener type, finding new and diverse applications such as steel manufacturing, off-highway equipment, nuclear power plants and food machinery. The general process for making gelled calcium sulfonate grease has remained largely unchanged since these types of greases were first developed in the 1960s.(1)

The first step in the manufacturing process is to add an overbased calcium sulfonate with a total base number (TBN) of 300 to 500 mg KOH / g eq. to a grease kettle. The highly overbased sulfonate is mixed with base oil and treated with acid to destabilize the micellular structure. Then promoters and water are added, and the material is heated to its gelation temperature. The gelation temperature depends on the promoters used, but is typically 60 to 90 °C. Provided that the water and promoters are retained in the reaction mixture, the amorphous calcium carbonate is converted to crystalline calcite within 1 to 5 hours, followed by removal of the water and promoters from the mixture. The penetration is then adjusted to the correct NLGI grade. The gelation temperature needs to be controlled to below 95 °C because above this temperature, the conversion reaction favours the formation of vaterite, an undesirable crystalline form of calcium carbonate, resulting in poor grease properties.

As seen in Table 1, the typical anti-wear and extreme pressure (EP) properties of sulfonate greases without additives are good. Base greases give very low four-ball wear scars, typically < 0.35 mm, Timken OK loads of at least 60 pounds, and four-ball weld loads of 400 kg and above. The calcium sulfonate greases also have good rust resistance, as measured by ASTM D1743, and give low oil bleed.

Prior to 1985, calcium sulfonate greases based on natural sulfonates were a niche product being manufactured and sold only in small quantities. The greases had several desirable characteristics: they had high dropping points (above 300 °C) and low oil separation, as well as very good anti-wear and EP properties without the use of additives. The negative aspects were the high thickener content, manufacturing complexity and associated cost compared to other greases. Historically, satisfactory water washout as measured by ISO 11009 contrasted with poor results in water spray off testing as measured by ASTM D4049, which is used as the measure of performance in steel mills. Calcium sulfonate complexes were subsequently developed, in which an additional saponification step to form calcium soap is carried out. The addition of components such as added pre-formed calcite, boric acid to form calcium borate and phosphoric acid to form calcium phosphate were also reported. It is well-known that anhydrous calcium 12-hydroxystearate soaps have superior water resistance to other simple soaps and their presence aided the water resistance properties of the grease as well as reducing the thickener content of the sulfonate. One negative aspect of the soap is that the excellent thermal stability of the sulfonate gel is reduced. A further issue is that

calcium sulfonate complex greases can have issues with pumpability and can age-harden.

In 2008, Lubrizol introduced new calcium sulfonate grease technology[2] that addressed many of the historical shortcomings of these greases and greatly simplified the manufacturing process. The gelling acid and the promoter were incorporated into the 400 TBN sulfonate and only base oil and

TABLE 1 – TYPICAL PROPERTIES OF A GELLED 400TBN SULFONATE GREASE

Property	Test Method	Typical Values
Base oil viscosity at 40 °C (mm^2/s)	ISO 3104	120
Dropping point (°C)	ASTM D2265	>316
4-Ball wear scar diameter (mm)	ASTM D2266	<0.35
Timken OK load (pounds)	ASTM D2509	≥60
4-Ball EP weld point (kg)	ASTM D2596	400
Water corrosion (rating)	ASTM D1743	Pass
Oil bleed (%)	ASTM D1742	<0.5
Water washout at 80 °C (%)	ISO 11009	<2.0
Water spray off (%)	ASTM D4049	15

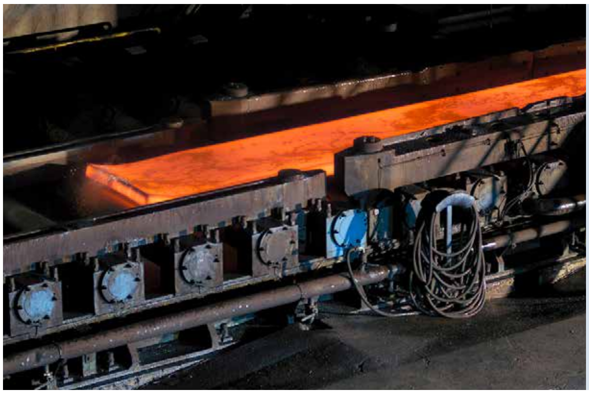
water were needed to gel it. This negated the flash issue with isopropanol and other alcohols used in gelation, and opened up the potential for sulfonate grease technology to a wider number of grease makers.

One of the interesting performance aspects of sulfonate greases is that base oil viscosity, rather than consistency class, is seen to have a bigger influence on the properties of the grease than it does with soap-thickened greases. One performance difference is that for soap-thickened greases, consistency class seems to affect spray off, while with calcium sulfonate greases, the base oil viscosity is more important. Looking at yield and thickener content, soap-thickened greases behave in an opposite manner than do sulfonates. Lithium soap greases give better yields and soap structures when saponified in lower viscosity naphthenic oils but for sulfonates, the same oil would cause the grease to be very soft with a much lower yield. Higher viscosity paraffinic oils give sulfonate greases better yields than do lower viscosity paraffinic oils.

Steel Mill Greases

Historically, clay and aluminium complex greases were used in steel mill applications. In the past, both of these types of grease have had issues, and steel mill operators required improved grease. Clay greases have good higher temperature properties but are incompatible with other thickeners and many grease additives systems. Aluminium complex greases have similar issues with additives, but have excellent water and high temperature resistance. It is not easy to formulate either of these two thickener types to have good EP and anti-wear properties. For steel mill applications, calcium sulfonate greases are superior because they have good high temperature stability, good water resistance and inherent EP and anti-wear properties. Lithium complex greases fortified with polymer have also been used in steel mill applications, but they can be problematic. These types of greases need anti-wear and EP additives to increase their load carrying capacity, many of which are not stable in the presence of significant amounts of water. A polymer-fortified grease may stay in place in the presence of the water, but may lose performance through additive hydrolysis.

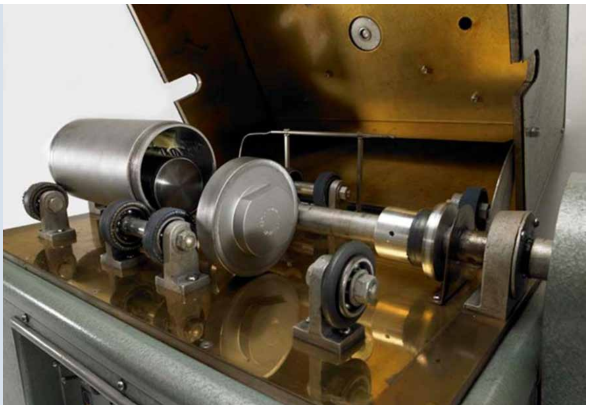
Looking at requirements for steel mill greases[3], a typical base oil viscosity is ISO VG 460, but with slightly tighter limits (± 30) than required by the ISO standard. The viscosity index is typically > 90, and the pour point is typically –12 °C or lower, indicating that a largely paraffinic mineral oil is required. Other key properties reflected by standard specifications are: high dropping point, shear stability, water washout and spray off, corrosion prevention (some specifications also require salt fog or humidity cabinet tests), and oxidation resistance. Tribological properties are defined as low wear, typically as measured by the ASTM D2266 four-ball test, and good load carrying capacity, as defined by either or both of the Timken OK and 4-ball EP tester.



Taking these typical steel mill grease requirements into consideration, the sulfonate needed to meet them can be sketched out. Most typical sulfonates come in low-to-medium viscosity fluids. Adding bright stock to them will only increase the viscosity to around 150 mm²/s. Adding high viscosity polyalphaolefin (PAO) fluids such as PAO 100 will only increase viscosity to 300 mm²/s, but does improve spray off and washout characteristics. Rather than adding high molecular weight polymers (>50,000 Daltons), the optimum solution is to add a polyisobutylene (PIB) to the base oil. By blending a suitable mid-viscosity paraffinic mineral oil with lower molecular weight (2000 to 3500 Daltons) PIB, the correct viscosity can be achieved, without negatively impacting the pour point. After adding an anti-oxidant, a passivated polysulfide and a corrosion inhibitor to the gelled sulfonate grease, the results shown in Table 2 were obtained.

TABLE 2 – PROPERTIES OF STEEL MILL GREASE FORMULATION

Property	Test method	Requirement / Typical	Fully formulated grease
Base oil viscosity at 40 °C (mm²/s)	ISO 3104	ISO VG 460 (430–490)	444
Base oil viscosity index (VI)	ISO 2909	> 95	136
Unworked & worked penetration	ISO 2137	265–295	268 & 268
Shear stability	ISO 2137	Δ, W100k No significant change	+7
Timken OK load (pounds)	ASTM D2509	≥ 60	65
4-Ball EP (kg)	ASTM D2596	Weld point ≥ 500	620
4-Ball wear scar diameter (mm)	ASTM D2266	≤0.45	0.34
Water corrosion (rating)	ASTM D1743	pass	pass
Water washout at 80 °C (%)	ISO 11009	2.75 max	1.4
Water spray off (%)	ASTM D4049	<25	<10
Oxidation stability, ΔP (kPa)	ASTM D942	100 hours ≤ 35.0	32.4



One steel mill operator requested a calcium sulfonate grease with less than 5% spray off as measured by ASTM D4049. Two solutions were considered. One incorporated a functionalized polymer into the grease. The polymer was added by dissolving it into a portion of the added base fluid and then incorporating it into the gelling phase of the grease. The acid functionality reacted with the free lime of the sulfonate to form the calcium salt. The spray off was determined to be <4% in two duplicate tests. The other solution was to dissolve a styrene isoprene polymer (SIP) into the added base oil for the calcium sulfonate grease. When net treated at 1%wt polymer, the spray off was 7.2% for two repeat runs, and when net treated at 2%wt, it gave 2.8%, clearly meeting the target.

In some countries, compatibility with water is important for steel mill greases. Testing the miscibility and roll stability with water is used to investigate this. The test is to run a roll stability test similar to that defined by ASTM D1831. The 60 stroke worked 1/2 scale penetration of the grease is determined as per ISO 2137 at 25 °C. The quantity of test grease is then applied onto the inner walls of the cylinder. The 5 kg weight is added followed by the desired weight of water, and then the closure cap is fitted and tightened.

TABLE 3 – ROLL STABILITY OF STEEL MILL GREASE

Condition	Amount used (g)		Rolling conditions		½ Scale penetrations		Δ FSE
	Grease	Water	Temperature (°C)	Time (hours)	Initial	Final	
dry	50	0	RT	2	138	139	+2
wet	50	10	RT	2	137	121	–32
wet	63	12.6	RT	2	137	127	–20
dry	50	0	80	100	140	145	+10
wet	50	10	80	100	139	147	+32

IMPROVING THE ALREADY GOOD

Ever since the 1980´s, lubricating greases based on calcium sulphonate complex thickeners have been available on the market. This technology, developed primarily in the USA and Canada, was claimed to provide very high load carrying capacity, exceptional mechanical stability, and excellent resistance to water and corrosion and in combination with outstanding performance in high temperature applications. Unfortunately, these greases have also shown some less desirable properties and were generally unsuitable for low and ambient temperatures. As long as the conditions were hot and wet, these greases performed well especially in selected applications such as steel mills. But they were not really suitable as multi-purpose products. In addition, calcium sulphonate complex greases were protected by patents, both when it came to the manufacturing processes and to the formulations themselves.

Already in the 1980´s, AXEL studied the possibility of offering such products to our customer base but, in the process of analysing samples of existing commercial products, we made an alarming discovery. The products exhibited excellent performance in many of the laboratory tests but failed miserably in OEM bearing tests. They did everything but lubricate. One scary example of this is the SKF

The grease is then rolled for the required duration at 20 °C to 35 °C, cooled back to 25 °C and then the worked 1/2 scale penetration is re-checked. The differences in penetration before and after rolling converted to full scale (FSE) are calculated and the results for wet and dry rolling under the same conditions are compared. The data for various amounts of water and running at different time and temperatures are in Table 3. Lithium complex greases run with water for similar durations and temperatures would suffer from much larger changes in consistency if no polymer was added. The data in Table 3 further reinforces that calcium sulfonate greases have significantly better water resistance properties compared to soap-thickened greases.

Off-highway Heavy Duty Calcium Sulfonate Greases

One of the most widely recognized uses of calcium sulfonate technology today is in off-highway equipment. Off-highway, heavy duty industrial machinery suffers under the most demanding conditions for lubrication. Heavy duty equipment manufacturers typically specify grease with a very high load carrying capacity and excellent water resistance.

The base oil viscosity of this type of grease is lower than the steel mill grease at 320 mm²/s for summer or all-season use and 220 mm²/s for cold

condition or winter use. It was decided to formulate a grease targeted at the ISO VG 320 using PIB as well as mineral oil and make it at the stiff end of the NLGI 2 range. As all of these types of off-highway products contain 5% molybdenum disulfide, it was decided to add 5% of a technical fine grade. Anti-oxidant and a rust inhibitor were added to the sulfonate along with some passivated polysulfide. The product was tested against typically specified characteristics and the results are listed in Table 4. As can be seen from the data, the grease easily meets the typical requirements.

Food Grade Sulfonate Greases

Regulations limit the type of grease chemistry, base oils, thickeners and additives that can be used in incidental food contact greases. The grease needs to

R2F test which is designed to assess the lubricating ability and high temperature performance of greases. There are/were two versions, the "A" test which involves running bearings without external heating, and the "B" test where the bearings are heated up to and maintained at predetermined temperatures (e.g. 140°C). The latter proved to be no problem at all for these "new" greases but, in the "A" test, they failed miserably. The grease performance is evaluated by measuring the wear on the rollers and cage. In this test, significant wear will only occur as a consequence of the inability of the grease to maintain a lubricant film in the rolling and sliding contact during the full test period. The test rig is run under the predetermined conditions for a period of 20 days before the bearings are dismounted and the wear is measured. In analysing the first generation of this type of product in the mid 1980´s, we tested a considerable number of commercially available greases in the R2F A test. With no exceptions, they all failed. And failed *badly*. The fact is that the bearings ran dry after only 2–3 days and the grease was completely destroyed leaving only a dry, black, carbonaceous residue. This is the worst result we had ever seen in these tests. We therefore decided to abandon the idea of promoting these products and instead we introduced a new and improved technology called Alassca. This has been described in previous White Papers. In the ensuing period, some advances were

use a food grade base oil and all of the components need to be registered. Standard sulfonates supplied in group I mineral oils cannot be food grade-registered and so overbased sulfonates in either technical white oil or PAO are necessary.

The next challenge is to employ a gelling process that is capable of being registered for food grade use. Taking a 400TBN overbased sulfonate in white oil, the calcium carbonate can be converted to calcite by adding acetic acid to destabilize the micellular structure, adding white mineral oil or PAO, a food grade-approved promoter and water. The amount of acetic acid added controls the rate of conversion. By adding sufficient acid to destabilize

TABLE 4 – OFF-HIGHWAY HEAVY MACHINERY GREASE

Property	Test method	Requirement / Typical	Fully formulated grease
Base oil viscosity at 40 °C (mm2/s)	ISO 3104	ISO VG 220 or 320	320
Unworked penetration	ISO 2137	265–295	269
Worked penetration	ISO 2137	265–295	261
Shear stability	ISO 2137	Δ, 100k No significant change	+7
Timken OK load (pounds)	ASTM D2509	≥ 60	65
4-Ball EP (kg)	ASTM D2596	Weld point ≥ 500 LWI > 80	800 124.6
4-Ball wear scar diameter (mm)	ASTM D2266	≤ 0.45	0.37
Water corrosion (rating)	ASTM D1743	pass	pass
Water washout at 80°C (%)	ISO 11009	< 2	1.1
Water spray off (%)	ASTM D4049	<10	1.3
Oxidation stability, ΔP (kPa)	ASTM D942	100 hours ≤ 35	29

made by suppliers of these calcium sulphonate complex greases and it was later claimed that there was a new and patented process which would overcome the before-mentioned deficiencies while retaining the other positive advantages of the technology. However, the SKF R2F A test still exposed the weakness of such products. Admittedly, the products now survived the full 20 day period but they still failed badly in the wear measurements. To pass the test, the wear on the rollers should not exceed 20 mg. In multiple tests, the wear levels fluctuated between 1600 and 3600 mg. So they were still very poor lubricants. Some reasons offered were that the greases were far too stable when it comes to oil separation (they could contain up to 35% thickener) and/or the different forms of calcium carbonate in the micelles were too hard, varying between 3–4 Mohs depending on the mineral source.

Since the market demand for these types of products continued to increase over the years (they do perform excellently in a number of specific applications), AXEL decided to revisit the possibility of developing and improving the technology further. Many different formulations and manufacturing procedures were investigated but these were almost always protected by patents. In 2005, we were approached by a major customer and asked to offer a very large quantity of a calcium sulphonate complex grease to cover their global needs in the marine sector. At the same time, we were asked to improve the water spray off properties since their current product (also sulphonate complex) was deficient in this test. This was very surprising since sulphonate complexes were claimed to have excellent resistance to water. Subsequent testing confirmed customer claims showing that these "marine" greases had water spray off values in excess of 90%.

the micellular structure of the sulfonate, conversion can be completed within one to two hours. However the resultant grease is soft, typically softer than an unworked penetration of 350. Based on published data, sulfonic acids can be used to thicken the grease, but the choice of which acid to use needs to be carefully considered. Some of the best acids for aiding gelation and stiffening are not approved for food grade use. The calcium salts of some acids are approved, and one of these acids needs to be chosen for use as the gelation aid. The yield of the food grade sulfonates is lower than that seen with the technology outlined by Denis and Sivik(2), and much more sulfonate is needed in comparison. One way of boosting the yield for sulfonates is to incorporate low viscosity grade PIB (Mw = 1000 to 2500 Daltons). Some PIBs are food grade-registered and, if approval is desired, it is necessary to select one of these. Good, general properties can be readily achieved. Some food grade additives are available, and these can be incorporated to improve properties such as oxidation.

A laboratory kettle batch of grease was made using an HX-1 registered 400TBN calcium sulfonate in white oil, food grade promoter and water, acetic and sulfonic acids, and using a blend of PAO 6 and food grade-registered PIB. As can be seen from the data in Table 5, the resulting food grade grease without additional additives has good properties.

TABLE 5 – PROPERTIES OF FOOD GRADE SULFONATE GREASES

Property	Test method	White oil food grade grease
Base oil viscosity at 40 °C (mm2/s)	ISO 3104	390
Unworked penetration	ISO 2137	315
Worked penetration	ISO 2137	305
Water washout at 80 °C (%)	ISO 11009	2.0
Oil separation (%)	ASTM D1742	0
Water corrosion (rating)	ASTM D1743	pass
Dropping point (°C)	ASTM D2265	>316
4-Ball wear scar (mm)	ASTM D2266	0.37
4-Ball EP weld point / LWI (kg)	ASTM D2596	500 / 93.4
Water spray off (%)	ASTM D4049	26.8

At that point in time, we had three possible options. The first option was to purchase a "base grease" from the patent holders and improve the product simply by including special additives. The second option was to purchase an intermediate "instant grease" which could be gelled by simply adding water and isopropyl alcohol. This second option demonstrated excellent water spray off properties which we believe came from the inclusion of functional polymers. However, neither of these two options fulfilled all the requirements of the customer and, in addition, both were far too expensive. So we were left with a third option, back to square one, producing our own calcium sulphonate technology from scratch.

We started by revisiting all of our previous work. The next step was to fine-tune the existing formulations and try to replace both the complexing agents and the activators to avoid patent infringement. And, to address the issue of water spray off improvement, lots of different polymer systems were tested. We even had to change the manufacturing process and consider investment needs to ensure dedicated equipment.

In many of the patented versions, boric acid is used as (one of) the complexing agent(s). In retrospect, this is actually seen to be one of the main factors behind the poor lubrication. Replacing boric acid has given us a much "better" product when it comes to achieving good results in the bearing tests. Admittedly, boric acid gives a somewhat better result in special high temperature tests, but its replacement instead provides excellent lubrication over a much wider temperature range. And, in addition, since boric acid is regarded as a "material of extremely high concern" by the ECHA, we were able to achieve a better HSE solution.

PAO-based Sulfonate Greases

The 400TBN sulfonate in PAO described above can also be gelled with non-food grade components to make a high performance industrial grade of calcium sulfonate grease. A batch of grease was made in a laboratory kettle using a blend of PAO 6 and PAO 100 as the base oil. The blend gave a final base oil viscosity grade ISO 68. An industrial grade of alkylbenzene sulfonic acid was used along with acetic acid. A promoter and water were also added, and the grease was gelled in 3 hours, followed by stripping at 145 °C and collecting the product.

A second grease was made in a resin flask, this time using PIB instead of the PAO as the viscosity fortifier. Also, by increasing the acetic acid quantity to a small excess over normal and keeping the amount of sulfonic acid and promoter constant, the gelling time was reduced from 3 hours to 45 minutes. The results of both these greases are reported in the Table 6 below. Both greases were made without additional additives.

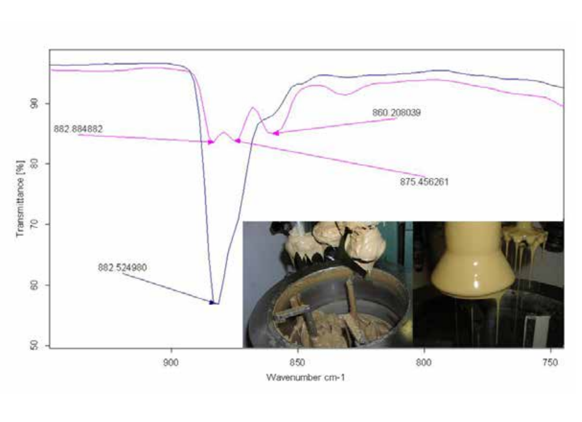
A further gelation was carried out by adding more PIB to the grease and the amount of water spray off was reduced to 33%. Based on other work, the oxidation stability and extreme pressure properties can be improved by the addition of further additives, as were used in the automotive and steel mill greases.

TABLE 6 – PROPERTIES OF PAO-BASED SULFONATE GREASES

Property	Test method	PAO based gelled sulfonate grease	
		Neat PAO	With PIB
Base oil viscosity at 40 °C (mm^2/s)	ISO 3104	68	220
Unworked penetration	ISO 2137	272	274
Worked penetration	ISO 2137	261	266
Water washout at 80 °C (%)	ISO 11009	0.5	1.5
Water corrosion (rating)	ASTM D1743	Pass	Pass
Dropping point (°C)	ASTM D2265	>316	>316
4-Ball wear scar diameter (mm)	ASTM D2266	0.35	0.37
4-Ball EP weld point / LWI (kg)	ASTM D2596	620 / 76.8	500 / 77.8
Water spray off (%)	ASTM D4049	53.6	51.2

Vital to the manufacturing process is the transformation of amorphous calcium carbonate and vaterite into the *calcite* form (wafer-like morphology). This is necessary to be able to gel the material into a grease-like substance. We were successful in finding a combination of activators which are both efficient in thickening power and safe in use (no need for investment in extra equipment to remove flammable gases like isopropyl alcohol or ethyl dioxitol).

In order to improve the water spray off values, it is necessary to understand the mechanisms required to keep the grease on the surface of the test panel. The product has to be both adhesive (sticks to the metal surface) and cohesive (sticks to itself). Depending on the spray off level in the particular customer specification, it is often wise to use two different polymers, one for adhesion, the other for



Summary

Using recently developed calcium sulfonate grease technology makes it easy for the grease manufacturer to make and to formulate calcium sulfonate greases to meet a wide variety of applications. The extreme pressure (EP) properties can be enhanced to give up to 800 kg 4-ball weld points, without compromising on wear or copper corrosion by using passivated polysulfides. Molybdenum disulfide can also be incorporated to boost the EP properties. Antioxidants and anti-fretting wear additives can be incorporated to enhance the properties to meet most general purpose requirements and many common industrial and automotive specifications.

Good water resistance is a desirable prerequisite property in applications employing calcium sulfonate grease as the lubricant. In particular, steel mills, motor-operated valves and food processing are areas where there have been significant use of calcium sulfonate greases. The calcium sulfonate greases used for such applications are characterized by the desirable high temperature performance, high EP, good wear, good rust and corrosion, as well as good water resistance properties. From the perspective of water resistance, calcium sulfonate greases show good performance in either or both the washout and spray off standard test methods for grease. Washout levels of 1–3% were observed for good calcium sulfonate greases; while, typical spray off performance representing good performance were between <10% to <30% grease loss in ASTM D4049 tests. Ultimately, performance is verified in the actual application depending upon the demand of the given application.

References:

(1) McMillen, R.L., “Basic Metal-Containing Thickened Oil Compositions,” US Patent 3,242,079, USPTO March 22, 1966

(2) Denis, R.A and Sivik, M. R., “Calcium Sulfonate Grease-Making Processes,” NLGI Spokesman, Volume 73, number 5, pages 30-37 (2009)

(3) Rush, R.E., “Greases for Steel Mill Lubrication”, NLGI Spokesman, Volume 57, number 3, pages 240-243 (1993)

cohesion. This can now be tailored to meet different demands. The first prototype for marine applications had a water spray off value below 10% (quite an improvement from the original 95%) but the product proved to be “too tacky” and caused problems with fling-off in high speed applications on deck. This was then adjusted back to 25% and this product is now used on ships all over the World.

The manufacturing process was also changed dramatically by daring to challenge the status-quo and doing things differently. In so doing, we have been able to accomplish a number of different things including a significantly shorter production time. But, by far the biggest advantage was gained by being able to produce these types of greases using a much lower thickener content. In the original type, more than 30% thickener was needed to produce NLGI 2 grade products. This not only impaired the products´ ability to lubricate but also caused problems with pumpability (especially important in the industrial versions). By reducing the content of thickener to 15–20% (i.e. virtually half of the original amount) we have managed to produce a much better product.

So moving on from an already good technology, we have been able to overcome the previously mentioned deficiencies and are now able to offer excellent greases for multi-purpose applications in industrial, heavy automotive, mining and marine applications.

Good, better, best?

NEXT ISSUE

The next issue of the Lubrisense™ White Papers will focus on lubricating greases for the mining industry. There are many different and special applications in both underground and surface mining which require specially formulated products. These need to function under slow moving and heavily loaded conditions and the surrounding environment is often, to say the least, challenging. Conventional mining greases are based on heavy oils or even bitumen and more than often loaded with black solid particles such as graphite and molybdenum disulphide. There is however a new generation of mining greases which are based on more modern technology and which offer considerable cost savings for the end user. The only problem is that they do not meet the OEM specifications for the old products. They are so much better. If you want new products, you need new specifications. Articles will be written by industry specialists, covering both the old and the new technologies.

As usual, we encourage reader contribution, feedback and proposals concerning relevant topics for future issues of our White Papers.

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