Food Grade High Temperature Grease

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Introduction

High viscosity synthetic esters have been used in high temperature industrial lubricants for many years. They are effective up to 288°C (550°F) and show little evaporation or oxidative degradation compared to other common lubricant basestocks. Inolex Chemical Company recently developed a synthetic ester that has earned the prestigious NSF HX1 approval for use in indirect food contact applications. The wide industrial acceptance of synthetic esters suggests that food processing plants will benefit from the long lasting, clean lubrication they provide. This paper describes high temperature food grade greases made from this unique base stock.

Background

The food industry represents one of the most demanding challenges for a lubricant. The lubricant must not only perform the familiar functions of reducing friction, wear, and corrosion, but it must conform to rigorous standards to ensure food safety is never compromised. Food safety is the first responsibility of any company that manufactures edible products. Consumers have every right to expect food plants will comply with government regulations and industry best practices at every stage of the process.

There are many associations and government bodies around the world that are involved in the safety of food products because of the obvious public health considerations and associated repercussions of contamination. In the United States, the regulatory environment begins with the US Food and Drug Administration (FDA). Interpreting the Federal regulations can be a monumental task, so NSF International provides guidance that helps food processors understand which products are approved for use in food plants and how they are best applied.

Food grade lubricants are made from base fluids and additives that are safe for food manufacturing. Regulations also require food grade lubricants to be tasteless, odorless and colorless. Until 1998, USDA Food Safety and Inspection Service, published "the white book", a list of proprietary substances and non-food compounds that were food grade or USDA H1 approved. The USDA cancelled all its activity to review and approve lubricants in September 1998. After this cancellation, NSF International adopted the procedures.

NSF International, a not-for-profit, non-governmental organization helps the FDA classify and manage approvals for lubricants in the food industry. NSF International provides a conformity assessment for the FDA and then categorizes the lubricant appropriately. NSF adopted these procedures to certify lubricants as H1 and H2. NSF defines H1 lubricants as "lubricants for incidental food contact special approval criteria based on FDA List 21 CFR Part 178.3570".

NSF H1 food grade lubricants are appropriate in situations where potential risk scenarios exist such that small amounts of lubricant that could potentially come in contact with edible products. NSF H1 lubricants offer the food industry a sense of security and safety from incidental contamination in food from more toxic, non-food grade lubricants.

At this writing, there were over 3000 NSF listed lubricants approved for incidental food contact (H1). Surprisingly, none of these have been entirely adequate for extended service in high temperature environments. Temperatures above 400°F (204°C) can be found in many food processing operations such as roasting, baking, braising, sterilizing, drying, broiling, and frying. Therefore, it seems the market would demand a food grade grease product with superior high temperature performance. In this paper, we describe the potential of greases made with new HX-1 rated synthetic ester basestocks.

Often the temperature performance of a grease is defined by its dropping point. That is, the point where the thickener loses its ability to maintain the grease structure. The base oil is usually only an afterthought because most oils are stable at ambient or moderately elevated temperatures. However, many oils cannot withstand temperatures above 204°C (400°F) and a true premium oil is required for extended performance at 260°C (500°F) and above. Therefore, the thickener and oil are both critical in this temperature regime.

The primary mechanisms for oil degradation at high temperatures are oxidative and/or thermal breakdown, and polymerization. Breakdown, in which scission of the lubricant molecule occurs, leads to the formation of lower molecular weight volatile compounds. Evaporation of these compounds can cause changes in viscosity, oil loss, and the production of excessive smoke. This can lead to poorer lubrication, higher cost, reduced cleanliness of the plant, poorer product quality, and higher exposure to potentially toxic organic compounds. Polymerization will lead to formation of insoluble gums and varnishes that can build up in the work environment or even contaminate food products. Cleaning these deposits requires additional maintenance and generates chemical waste materials for disposal. Further, production is lost as machinery is taken out of service for cleaning.

Industrial lubricants employ different base oils depending on the severity of the application. Lower temperature lubricants generally use hydrocarbons or natural esters. Synthetic esters, particularly those based on neopolyol chemistry, provide significantly better oxidative and thermal stability and have long been the preferred choice in high temperature industrial applications. Synthetic esters have not historically been approved for food contact lubricants, but in 2008, NSF International granted HX-1 approval to four synthetic esters from Inolex Chemical Company. These products can be used to formulate NSF H1 greases with outstanding temperature stability.

Objective

The purpose of this paper is to assess the performance of high temperature food grade grease by focusing on the oil/thickener system. We made laboratory samples of food grade synthetic ester greases using four different thickeners as shown below. All of these formulations included food grade antioxidants, corrosion inhibitors, and antiwear additives. The high temperature degradation of base oils generally occurs by thermal oxidation, so antioxidants play a significant role in promoting high temperature stability. It should be noted that these high temperature greases are not designed to provide EP performance. All ingredients are listed with the FDA/NSF, so the formulations should be suitable for achieving an H1 rating from NSF International.

To provide a useful comparison, we also purchased several commercially available food grade greases. These used a variety of base oils and thickeners and were rated for temperatures between

NSF HX-1 esters	Density @25°C	Viscosity cSt		Viscosity Index	Flash Point °C*	Fire Point °C*	Pour Point °C
		40°C	100°C				
Ses-68	0.99	70	10	125	315	345	-50
Ses-100	0.99	100	12	120	310	350	-40
Ses-220	0.97	220	19	95	315	350	-25
Ses-350	0.97	350	24	85	305	335	-20

**containing appropriate antioxidant additives*

300°F and 700°F as shown in the table below. Of course, these will also contain food grade anti-oxidants and other additives.

Experimental

This test is not meant to simulate any specific working environment, but provides a simple comparison of how temperature affects both the thickener and base oil. A two gram sample of grease is weighed into a 50 mm aluminum pan and then topped with a 70 mm cover.

Up to nine specimens are set on a cookie sheet and placed in an oven for 20 hours at the specified temperature. The specimens are removed and allowed to cool for one hour. The pans and covers are weighed again to determine weight loss of the grease and weight increase in the cover pan attributed to soot and varnish deposited from oil vapors. They are also photographed to document the appearance of the grease and pans.

Testing started at 400°F (204°C). If the grease survived, a fresh sample was used for testing at a higher temperature.

Table 1
Experimental greases made from synthetic ester basestocks

ID	NLGI #	Base oil	Thickener
SesAlx13	2	Ses-350	Al Complex
SesPtfe14	2	Ses-350	PTFE
SesSil15	0	Ses-350	Silica
SesClay16	2	Ses-68	Clay

Table 2
Commercial grease samples

ID	NLGI #	Oil	Thickener	Claim Max temp
MoAlx1	2	Petroleum	Al complex	500°F
MoAlx2	1	Petroleum	Al complex	500°F
MoAlx3	2	Petroleum	Al complex	375°F
MoAl4	2	Petroleum	Aluminum	300°F
ShcPtfe5	2	PAO	PTFE	400°F
ShcSil6	2	PAO	Silica	700°F
ShcSil7	2	PAO	Silica/PTFE	650°F
VoAlx8	2	Veg Oil	Al complex	500°F
ShcPtfe9	2	PAO	PTFE	600°F
ShcCas21	2	PAO	Ca Sulfonate	360°F
MoCas22	2	Petroleum	Ca Sulfonate	360°F
MoAlx23	2	Pao/Wmo	Al complex	N/a
VoAlx24	2	Veg Oil	Al complex	N/a
MoCas25	2	Petroleum	Ca Sulfonate	N/a
ShcCal28	2	PAO	Ca complex	N/a
MoCas30	2	Petroleum	Ca Sulfonate	300°F
VoSil31	2	Veg Oil	Silica	N/a

Results

All of the aluminum complex greases lost their structure at 400°F. The PTFE, calcium, clay and silica greases held shape, although some showed bleeding or skinning. All of the vegetable oil based greases solidified, presumably from oxidative polymerization. Beyond that, the thickener was the main cause of failure after 20 hours at 400°F. With the exception of ShcPtfe5 and VoSil31, the samples showed little base oil evaporation or vapor deposits. Some trends are starting to emerge, but the only base oils that could not be used at 400°F were vegetable oils.

Starting at 450°F, we begin to see the predominant effect of the base oil. Some samples show significant evaporation loss and vapor deposits. Many of the samples also showed a hard skin on the surface. This can be caused by oxidative polymerization of the base oil or by evaporation of the base oil leaving a surface layer of thickener.

At 500°F, the thickeners are still holding the grease in shape, and the base oil performance is becoming even more pronounced. All of the samples lost at least 10% of their original weight, and most left a heavy stain on the cover pan as the evaporating oil left solid deposits on the nearby surfaces.

Table 3
Test conditions: 400°F (204°C), 20 hours

ID	Thickener Stability	Skinning	Weight Loss	Vapor Deposit (mg)
MoAlx1	Liquid	None	14%	0.7
MoAlx2	Liquid	None	14%	0.4
MoAlx3	Liquid	None	11%	1.2
MoAl4	Liquid	None	6%	0.8
VoAlx8	Liquid, polymerized	Yes	11%	0.7
MoAlx23	Liquid	None	9%	0.4
VoAlx24	Liquid, polymerized	Yes	7%	0
SesAlx13	Liquid	None	8%	-0.1
ShcPtfe5	No drop	Thin skin	44%	1.2
ShcPtfe9	No drop, some bleed	None	4%	0.4
SesPtfe14	No drop	None	0%	0.1
ShcCas21	No drop	None	2%	0
MoCas22	No drop	None	2%	0.3
MoCas25	No drop	None	2%	0.4
MoCas30	Liquid	None	13%	0
ShcCal28	No drop	Thin skin	6%	1.3
ShcSil6	No drop	None	8%	1
ShcSil7	No drop	None	8%	1.1
VoSil31	Liquid, polymerized	Yes	32%	0
SesSil15	Sagged	None	1%	0.6
SesClay16	No drop	None	5%	0.1

Under these severe conditions, the cleanliness of the synthetic ester base oils becomes apparent. The two samples with the least vapor deposits were both made with synthetic ester oils. Interestingly, SesClay16 used a low viscosity (ISO 68) base oil and showed significant evaporation, but it evaporated cleanly without leaving varnish on the cover pan. SesPtfe14 was made with an ISO 350 base oil. The higher molecular weight oil gave much less evaporation as well as low deposits.

The best way to compare deposits is by looking at the photographs at the end of the report. Some samples left such heavy vapor deposits that the varnish stained the outside of the neighboring pans. For example, most of the weight gain recorded for SesPtfe14 was on the outside of the pan and did not become the synthetic ester base oil.

At 550°F, the calcium sulfonate grease samples begin to sag. All of the greases except SesPtfe14 developed a thick skin, and most showed heavy to extreme vapor deposits. The two synthetic ester samples are still very clean relative to the others. SesPtfe14 did harden somewhat, but it had no solid skin or other residue that would prevent efforts to flush a bearing with fresh grease.

Table 4
Test conditions: 450°F (232°C), 20 hours

ID	Thickener Stability	Skinning	Weight Loss	Vapor Deposit (mg)
ShcPtfe5	No drop, shrunk	Thick skin	55%	2.4
MoCas30	Liquid	None	23%	1.9
ShcSil7	No drop	Thick skin	14%	1.8
ShcCal28	No drop	Thick skin	11%	1.3
MoCas25	Sagged	None	5%	1.5
ShcPtfe9	No drop, heavy bleed	Thin skin	8%	0.5
MoCas22	No drop, some bleed	Thin skin	5%	0.5
ShcCas21	No drop	None	5%	0.3
SesPtfe14	No drop	None	2%	0.1

Table 5
Test conditions: 500°F (260°C), 20 hours

ID	Thickener Stability	Skinning	Weight Loss	Vapor Deposit (mg)
ShcSil6	No drop, heavy deposit	Solid	35%	27.2
ShcSil7	No drop, heavy deposit	Thick skin	23%	17.7
ShcCal28	No drop, heavy bleed	Thick skin	15%	9.3
MoCas25	No drop, some bleed	Thick skin	12%	7.1
MoCas22	No drop, some bleed	Thick skin	11%	6.9
ShcPtfe9	No drop, heavy bleed	Solid	16%	6.5
ShcCas21	No drop, some bleed	Thick skin	11%	4.0
SesPtfe14	No drop, some bleed	No skin	10%	3.9
SesClay16	No drop, shrunk	Solid	65%	1.6

Discussion

This experiment was designed to separate the performance of the grease thickener from the base oil. The base oil causes weight loss and vapor deposits in the grease sample at high temperature. The thickener determines the dropping point.

Hardening or skinning may be a chemical change in the oil or thickener or may be a result of oil evaporation that concentrates the thickener left behind. Previous experience shows that long exposures to temperatures above 400°F can cause some oils to polymerize and harden independent of the thickener, so this must be considered when working in this temperature range.

In the Appendix of this report are several photographs that show the appearance of the grease and cover after testing. The smaller pan on the left held the grease, and the larger pan on the right covered the grease during the experiment. Each photograph also shows a grease smear on the upper left. Thickened or skinned greases do not spread evenly and often look like a blob in the photograph.

Conclusions

Many NSF International H1 food grade greases give outstanding performance at temperatures up to 400°F (204°C), but most of these cannot withstand continuous operation (20+ hours) above 400°F. This failure is attributed to evaporation or thermal degradation of the base oil.

Newly NSF registered polyol ester base oils can be thickened with PTFE to make outstanding grease that is capable of continuous performance at 500°F (266°C) and intermittent use at 550°F (288°C).

Please note that the Inolex Chemical Company has pending patents on NSF H1 and HX-1 registered synthetic esters.

Table 6
Test conditions: 550°F (288°C), 4 hours

ID	Thickener Stability	Skinning	Weight Loss	Vapor Deposit (mg)
ShcSil6	No drop, heavy deposits	Thick skin	29%	53.2
ShcSil7	No drop, heavy deposits	Thick skin	5%	29.9
MoCas22	Sagged, heavy deposits	Thick skin	9%	17.0
ShcCas21	Sagged, heavy deposits	Thick skin	9%	16.7
MoCas25	Sagged, heavy deposits	Thick skin	10%	15.5
ShcCal28	No drop, heavy deposits	Thick skin	7%	13.6
SesClay16	No drop, some bleed	Solid	22%	8.3
ShcPtfe9	Heavy bleed	Thick skin	11%	7.5
SesPtfe14.	No drop, some bleed	No skin	7%	2.2

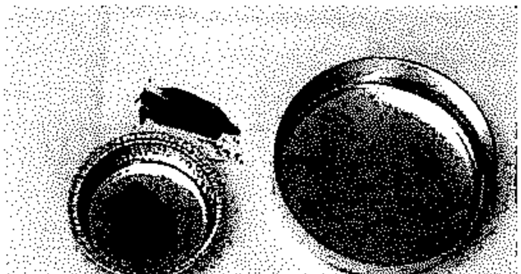
About the Authors

Tyler Housel received his B.S. in Chemistry, 1983 from RPI, and his M.S. in Materials Science, 1985 from Caltech. He has been with Inolex for 19 years in lubricant and polymer applications and is currently the Director of Industrial Applications. Tyler was previously with PDA Engineering working on light and heat reactive polymers for military applications.

No biographical information was available for Sarah Plimpton Murphy as of press time.

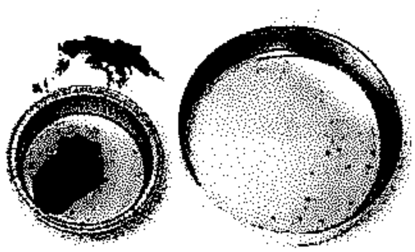
See the following four pages for Appendices 1-4 . . .

Appendix 1

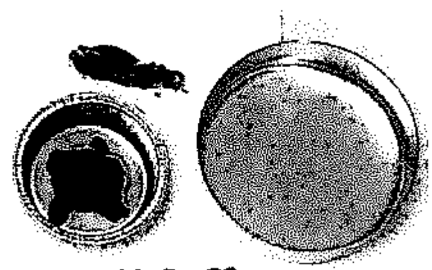


ShcSil7
400°F (204°C), 20 hours

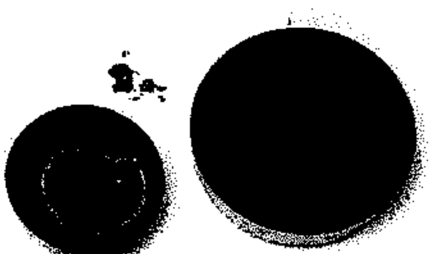
MoCas22
400°F (204°C), 20 hours



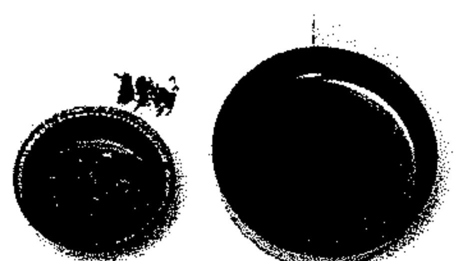
ShcSil7
450°F (232°C), 20 hours



MoCas22
450°F (232°C), 20 hours



ShcSil7
500°F (260°C), 20 hours

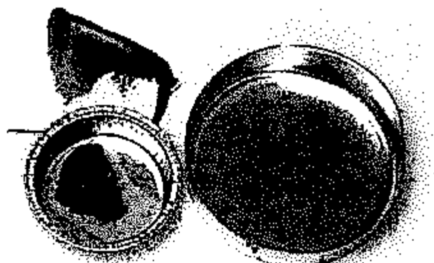


MoCas22
500°F (260°C), 20 hours

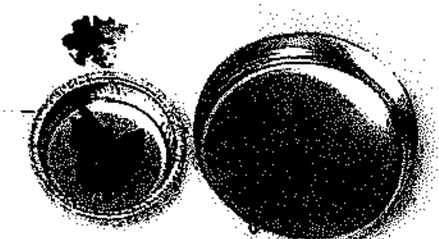
ShcSil7
550°F (288°C), 4 hours

MoCas22
550°F (288°C), 4 hours

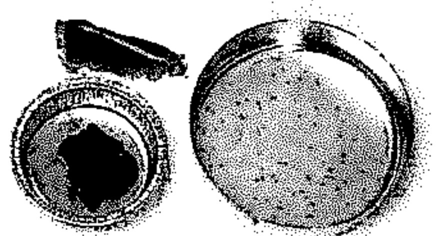
Appendix 2



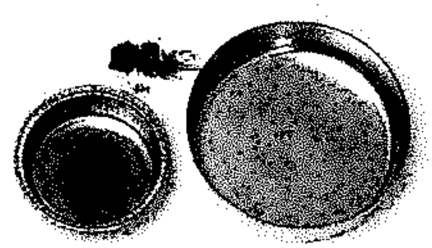
SHCCAS21
400°F (204°C), 20 HOURS



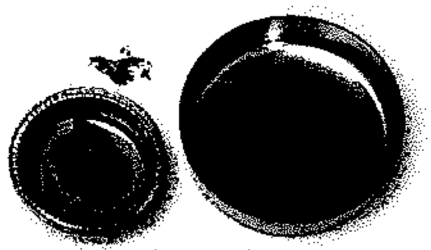
SHCCAL28
400°F (204°C), 20 HOURS



SHCCAS21
450°F (232°C), 20 HOURS

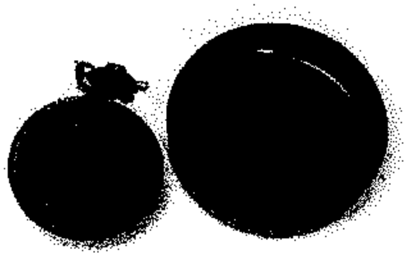


SHCCAL28
450°F (232°C), 20 HOURS



SHCCAS21
500°F (260°C), 20 HOURS

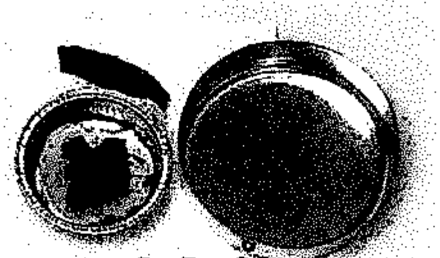
SHCCAL28
500°F (260°C), 20 HOURS



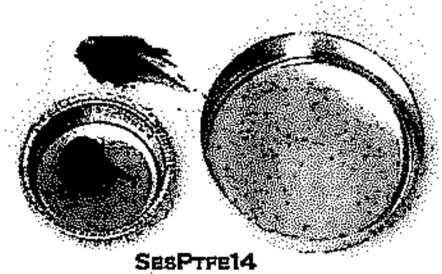
SHCCAS21
550°F (288°C), 4 HOURS

SHCCAL28
550°F (288°C), 4 HOURS

Appendix 3

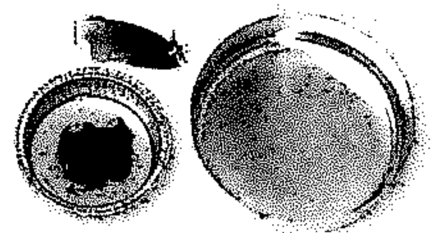


SHCPTFE9
400°F (204°C), 20 HOURS

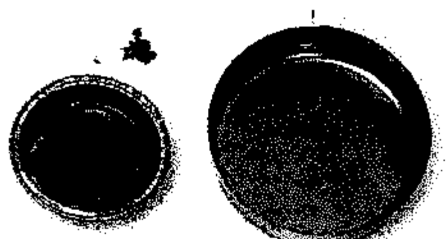


SESPTFE14
400°F (204°C), 20 HOURS

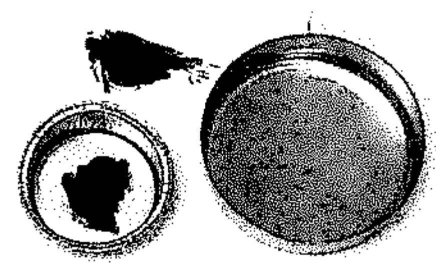
SHCPTFE9
450°F (232°C), 20 HOURS



SESPTFE14
450°F (232°C), 20 HOURS

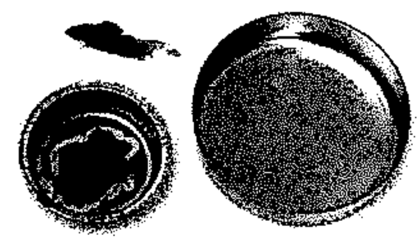


SHCPTFE9
500°F (260°C), 20 HOURS



SESPTFE14
500°F (260°C), 20 HOURS

SHCPTFE9
550°F (288°C), 4 HOURS



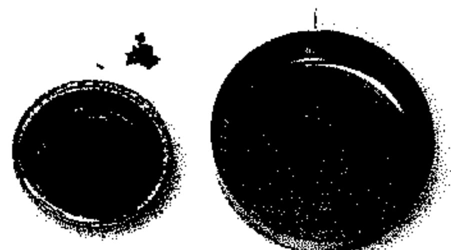
SESPTFE14
550°F (288°C), 4 HOURS

Appendix 4

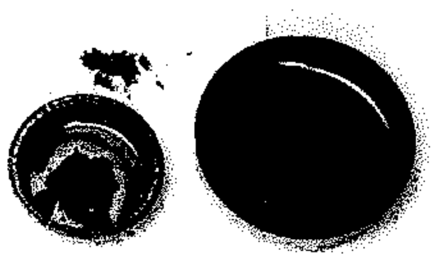
ShcSil6
550°F (288°C), 4 hours

ShcSil6
500°F (260°C), 20 hours

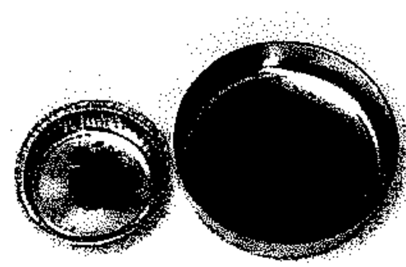
ShcPtfe9
550°F (288°C), 4 hours



ShcPtfe9
500°F (260°C), 20 hours

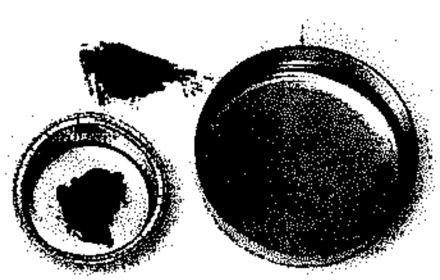


SesClay16
550°F (288°C), 4 hours



SesClay16
500°F (260°C), 20 hours

SesPtfe14
550°F (288°C), 4 hours



SesPtfe14
500°F (260°C), 20 hours