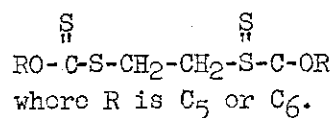
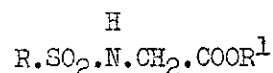


(7) Mesulfol 2.

The particular xanthate (dissolved in ketones, alcohol or water) is reacted with ethylene dichloride at 70°C. The product is washed with water and distilled.

(8) K.S.E.

where R₁ = C₁₅ radicle

R is derived from 180-250°C alcohols
from isobutanol synthesis.

0.46 g. of a C₁₅ hydrocarbon fraction, treated with SO₂ and Cl₂ to give a sulphonyl chloride is reacted with 2.2 g. mesamidoacetic acid and 0.69 g. of the alcohol fraction at 150°C in the presence of 0.002 g. β-naphthalene sulphonic acid as catalyst and propyl ether or ester distillate as azeotrope-forming agent to effect removal of the water produced during esterification. The reaction mixture is neutralised with carbonate solution, washed with 5% NaCl solution or distilled at 120°C (base temperature) at 10 mm. Hg., diluted with 50% middle oil and washed with a mixture of 2 parts methanol and 1 part water. The washed crude ester is distilled at 2-5 mm Hg until the still bottom temperature reaches 140°C and the liquid remaining in the still is refined with Fullers' Earth.

Other compounds not yet used in fuels or lubricating oils, which were being studied, included

- (1) Ester 1321 - a mixture of adipic esters of C₉ alcohols.
- (2) Ester 1473 - the reaction product of C₉ branched-chain alcohols with one mole of ethylene oxide and adipic acid.
- (3) Mellit L-85 - a reaction product of trimethylol ethane with carboxylic acid and 160-180°C alcohols.
- (4) Paladinol - a plasticiser made from phthalic acid and n-butanol or 140 - 180°C alcohols.

II. Special Lubricants.

- (a) Waffenol Blau 44, a rust preventive gun oil containing Mesulfol 2 in an amount corresponding to 3% sulphur in the final product. The other components are ester E-455 and a light distillate from synthetic lubricating oil known as VT-120. No production figures were available.

Composition:	VT-120	45%
	E.455	45%
	Mesulfol 2	10%
Properties:	Viscosity	1.9°E at 20°C.
	Flash Point	125°C.

- (b) Torpedo Oil T-1 was developed as a replacement for neatsfoot oil. It was made only at Leuna and was tested at Gdynia. Total production for 1944 was 100 Tons, and the last shipment from Leuna was 40 Tons of finished product.

Composition:	SS.903	35%
	E.515	63%
	KSE	2%

Properties:	Viscosity	11.5-12.5°E at 20°C
	Pour Point	below - 50°C

- (c) V-weapon Oil SS-1631 was developed at Leuna after about nine months of research. A monthly production of 100-200 tons was planned, but total production amounted to only 100 Tons. The last shipment of this material had been to Göttingen.

Composition:	SS.903	25%
	E.515	72%
	KSE	3%

Properties:	Viscosity	2°E at 50°C
	Pour Point	below -45°C
	Flash Point	145°C

- (d) Low Pour-point oils, K.10 and K.19. were developed for use in Russia and had been reported as satisfactory in service.

		<u>K.10</u>	<u>K.19</u>
Composition:	SS.903	50%	75%
	E.515	50%	25%
Properties:	Viscosity	1.65°E at 99°C	2.0°E at 99°C
	Pour Point	below -50°C	below -45°C
	Viscosity Index	135	130

- (e) "Y" railroad axle oil was developed for use in extremely cold weather after the experiences of the first winter of the Russian campaign. Tests have shown that an additional 100% loading can be put on railroad cars using this lubricant and all equipment failures due to lubricants are eliminated. A test has been run using the oil as an automotive lubricant in one German army corps in Russia and all the Leuna vehicles use it. These tests showed no ring-sticking or corrosion difficulties.

Composition:	"R" Oil	80%
	E.504	20%
	Dye	0.1%

"R" Oil is the oil obtained on neutralising the $AlCl_3$ catalyst complex in synthetic lubricating oil manufacture.

I.C. 7370

Properties: Pour Point -45 to -50°C.
Viscosity Index. 118-122

- (f) Aviation hydraulic Oil was developed to pass tentative specification Do 2000, put into effect in late 1943. The oil has excellent corrosion-resistant properties and low rubber-swelling properties (less than 2% increase in volume after 24 hours immersion). A fluorescent dye was added to the oil to meet the Luftwaffe colour specification.

Composition: V-120 84%
E.3022 or E.3023 12%
KSE 4%
Dye .006%

Properties: Viscosity 1.75°E at 20°C
650°E at -60°C

Pour Point -70°C
Flash Point 120°C.

Conclusions and recommendation.

The use of esters in order to obtain low pour-point lubricants merits further close study. The excellent extreme-pressure properties of Mesulfol 2 and the outstanding properties of KSE as a corrosion preventive should also be investigated further.

XII. SYNTHETIC LUBRICATING OIL - SCHKOPAU.

Introduction:

Construction of Buna Werke, A.G. Schkopau, the first and largest synthetic rubber plant in Germany, was started in 1937 and completed in 1939. It is owned and operated by I.G. Farbenindustrie. The lubricating oil plant at Schkopau was built by I.G. as a semi-works plant to utilise the excess acetylene from the large synthetic rubber plant, Buna Werke A.G. In the latter plant, acetylene, manufactured from calcium carbide and water, is converted to butadiene by the aldol process, and then polymerized with styrene made from ethyl-benzene for the preparation of Buna S. The normal Buna S production is 6,000 T/month when operating at capacity. In the Buna S plant, part of the acetylene is hydrogenated to ethylene, and the ethylene reacted with purchased benzene to make ethyl benzene. Excess acetylene can be converted to ethylene in the same manner and diverted to the manufacture of synthetic lubricating oil, where the ethylene is subsequently polymerized with aluminium chloride, neutralized, reduced to the desired viscosity and contacted with fine clay for the manufacture of SS-903 and SS-906, synthetic lubricating oils. Normal average production is 500 T/month, or slightly more than 100 B/D (42 U.S. Gal.)

Description of the Process:

Excess acetylene from the rubber plant is hydrogenated to ethylene using 0.01% palladium oxide catalyst supported on silica gel. Hydrogen is obtained by pipe line from Leuna. The reaction takes place at 200-250°C and atmospheric pressure. Each oven contains approximately 4 cubic metres of catalyst arranged in three beds with direct water spray between stages to control the reaction temperature. At the end of 6-8 months, the catalyst is reactivated with air at 500-600°C. The yield of ethylene was reported to be 72% of theoretical. After water washing, the gas is fractionated in the Linde plant to yield 99% pure ethylene.

From the Linde plant, the ethylene passes to the polymerization step. The six reaction vessels, for polymerizing the ethylene, are made of stainless steel (V₁A), 12 m. high and 800 mm. or 1200 mm. i.d., pressure tested at 200 atmospheres, nominal working capacity, 6-8 cu. metres. The reactors are water and steam jacketed with bolted, flange heads, with provisions for admitting the aluminium chloride and the light recycle material through the top flange. A small stirrer, with stainless steel blades, is driven through a liquid seal stuffing box in the bottom flange. A 5 HP electric motor with reduction gears drives the stirrer at 120 RPM. Ethylene is also admitted through a suitable connection in the bottom flange.

The reactors are operated batchwise. 1.5 cu.m. of the "Vorlauf-öl" (low boiling hydrocarbon recycle stock) is charged to the reactor along with a quantity of aluminium chloride equivalent to approximately 7% by weight of the finished polymer. Ethylene is then bubbled through the aluminium chloride-hydrocarbon mixture at 60-100 ats pressure and with the temperature maintained at 120-140°C. The initial reaction is exothermic and cooling water must be circulated through the jacket to absorb the excess heat evolved. As the reaction proceeds, heat must be supplied to maintain the temperature at

120-140°C. The reaction is completed in 8-10 hours. The sour oil (polymer-aluminium chloride sludge mixture), now at atmospheric pressure, is transferred to small intermediate tanks, equipped with stirrers to keep the sludge in suspension, 10-12% by volume of methanol is added, and the resultant mixture centrifuged.

Six basket-type centrifuges (baskets rotating at 900 RPM on a horizontal axis and driven by 35 HP electric motors) built by Escher Wyss, Ravensburg, are used. The supernatant oil is pumped to a neutralizing vessel, equipped with stirrers, and enough lime is added to neutralize any free HCl. Normally, 2% by weight of lime is needed, but this varies with the amount of HCl evolved during the polymerizing step. The neutralized oil is pumped to plate-and-frame filter presses to remove the calcium chloride. Six presses have been installed, only three are normally used. The neutralized oil is then reduced to the desired viscosity, using vacuum and steam at a maximum temperature of 250°C. The methanol vapours from the reducing still, along with the methanol vapours from the centrifuges and the intermediate handling tanks, pass to the methanol recovery system where 97.0 - 98.0% of the methanol is recovered by rectification. The "Vorlauf-öl" (light hydrocarbon recycle oil) is returned to the reaction vessel for re-use.

The bottoms from the vacuum reducing still are pumped to the clay contact units and contacted with 5% by wt. of fine clay or silica gel. Two contactors are in use, each with a capacity of 10 cubic metres, equipped with stirrers operating at 50 RPM., clay treatment is carried out at 100-120°C for 30 minutes. The oil-clay mixture is pumped to a plate-and-frame filter press where the fine clay is removed. The filtered oil is next pumped to a plate-and-frame filter press where the fine clay is removed. The filtered oil is next pumped to a de Laval centrifuge to remove any traces of fine clay leaking through the filter press. The filter cake is blown with nitrogen to recover the oil soakage.

The finished lubricating oil, usually 8-10° Engler at 100°C., is blended with oil of lower viscosity for 5.8 - 6.2° Engler, and shipped to various blending stations where it is blended with an equal volume of natural mineral oil for use in aircraft engines. Synthetic lubricating oil from Schkopau was used only in the production of aviation lubricating oil because of its excellent non-ring sticking properties. Typical tests of Schkopau synthetic lubricating oil are as follows:-

<u>Description:</u>		SS-903 Oil	SS-906 Oil
<u>Inspection:</u>			
Specific Gravity		0.80-0.83	0.805-0.835
Viscosity, Engler° 100°C		2.9 - 3.0	5.8 - 6.0
Viscosity Index		105 - 115	105 - 115
Flash: °C		205	225
Pour: °C		-35	-30
Carbon Residue: %		0.02	0.03
Neutralization No.		nil	nil
Ash %		nil	nil
Chlorides: %		nil	nil

Typical quantities of raw materials and utilities needed per ton of synthetic lubricating oil, are as follows:

Acetylene	1700 kg.
Methanol	2.7 kg.
Vorlauföl	1.2 "
Aluminium Chloride	70.0 "
Lime	30.0 "
Fuller's Earth	10.0 "
High Pressure Steam	1.7 tons
Low Pressure Steam	6.0 "
Fuel Gas (2000 kg. Cal/M ³)	250 M ³
Electricity	150 KWH
Water	150 M ³

Miscellaneous:

The German technicians interrogated maintain that little corrosion difficulties have been encountered; stainless steel is used in the reaction vessels, while intermediate sour oil tanks have synthetic rubber liners faced with brick. Some corrosion was noticed in the vapour condensers of the vacuum reducing still but this was eliminated by adding caustic soda to the reflux to the vapour fractionator.

Aluminium chloride is obtained from the I.G. Farbenindustrie A.G., Ludwigshafen-am-Rhein, in special metal drums. The drums are dumped into the reactor by hand using a funnel that fits the end of the drum. A gate valve controls the rate of admittance, when charging the reactor. The effect of impurities in the aluminium chloride was not known.

The rubber plant employs 15,000 men when operating at normal capacity. The lubricating oil plant normally employs 65 men. Operation is intermittent, depending upon the supply of acetylene from the rubber plant. The plant has not been damaged to any extent and operations can be resumed as soon as a supply of ethylene can be obtained.

Conclusions:

While the production of lubricating oil from acetylene is of interest, the cost of the finished product is high. Assuming that acetylene can be made for 25-30 pf. per kg. from natural gas (methane) by the electric arc process used at Huls, the large capital expenditure for equipment and the number of men required for operation, make the cost of the synthetic lubricating oil too high to compete with lubricating oils from selected American crude oils, unless an examination of the samples obtained, indicates that these oils have some valuable properties for special uses.

XIIA. SYNTHETIC LUBRICATING OIL MANUFACTURE AT STETTIN-POLITZSource of Information:

Information on this plant was secured in the course of an interview with Dr. Zorn at Leuna on 15th May 1945.

Summary:

A mixture of Fischer-Tropsch wax (maximum melting point 176°F), and a special wax from brown coal tar distillation (made by a combination cracking/hydrogenation process at Ludwigshafen) is cracked under closely controlled conditions to give 95-97% olefines. These are polymerised at low pressure in the presence of aluminium chloride. The oil is separated, neutralised and vacuum distilled with steam to remove light ends.

Ultimate yields from the paraffin wax charged are:-

	Weight %
Gas	27-30
Lubricating Oil ...	49-52
Diesel Oil (light ends)	6-7
Steam cylinder oil (from decomposition of catalyst complex).	8-11
Total accounted for	<u>90-100</u>

Production capacity of this plant was rated as 1000-1200 metric tons/month (242-290 B/D). In the final 6 months' operation, production was about one-half this figure.

Description of Process:Wax Cracking:

The wax is preheated in a pipe still at low pressure to 842-896°F and completely vaporised. It then flows upward in a parallel tube bank heated by circulating flue gas. The outlet temperature is controlled at 968-977°F at atmospheric pressure. This cracking tube bank is made of 18-8 stainless steel (V2A) to avoid coking and dehydrogenation. Residence time in the pre-heater is 1-2 minutes, and in the cracking coil about 0.1 second. No steam is used in the furnaces.

The effluent is quenched in a steam heat exchanger to 500°F. All condensate at this temperature is recycled to the furnace. The vapours (about one-third of total) are cooled indirectly with 68°F water and the gas is vented from this condensation.

Gas composition is:

	%
Hydrogen	1-2 (analysis used for plant control).
Ethylene	30
Propylene	15
n-Butylenes	15
Paraffins	40

I.C. 7370

Ultimate yield of gas on fresh feed is about 30 weight per cent, with a minimum of 27-28 weight per cent. This gas was not used for polymerisation.

The liquid product is 95-97% unsaturated. Determination of the iodine number is used as one control in the cracking step. This liquid is used directly in the polymerisation step.

Polymerisation:

Polymerisation is carried out at atmospheric pressure in reactors of 12 barrel capacity, water-jacketed, and equipped with straight blade stirrers revolving at 100 RPM. The aluminium chloride used in polymerisation is first suspended in light oil from the process. A part of the olefine feed is added the kettle is heated to 158-176°F to start the polymerisation and then controlled at the desired polymerisation temperature by the rate of addition of cold feed. The total olefine feed is added in about 22 hours and the reaction is completed by heating the batch to 212-230°F and stirring at that temperature for two hours to further polymerise lower polymers formed in the early part of the reaction. Two grades of lubricating oil have been manufactured in this equipment:

SS 1105	nominally 3° Engler Viscosity at 100°C
SS 1106	" 6 " " " "

Their respective characteristics are discussed later. Operating conditions for the reactors for these products are as follows:

	<u>SS 1103</u>	<u>SS 1106</u>
Al Cl ₃ catalyst consumption (% of finished oil) ...	7-8	14-16
Reaction temperature °F ...	158	(Low as possible. (Winter - 104 (Summer - 122

Refining of Polymerised Oil.

The total product from the polymerisation reactors is separated into oil and aluminium chloride addition compounds by ordinary centrifuges. No methanol is needed for this separation. Centrifuging is done at 176°-212°F without any additional heat being supplied. The aluminium chloride addition product so separated is in a liquid form.

The lubricating oil portion is treated with water at room temperature to decompose traces of aluminium chloride, then treated with lime and Fuller's earth, filtered and distilled under vacuum with steam. The light ends are partially recirculated for suspension of aluminium chloride in the polymerisation reactors and partially yielded as Diesel oil. This oil can also be hydrogenated and then treated with sulphur dioxide and chlorine in the Mersol synthesis.

The aluminium chloride addition product from the centrifuges is decomposed with water, treated with lime and Fuller's earth and used as steam

I.C. 7370

cylinder oil after stripping of low boiling hydrocarbons for flash point specifications. This steam cylinder oil is considered better than the corresponding product from ethylene, because it is obtained at a lower polymerisation temperature and is less unsaturated.

Tests on Products:

	<u>Lubricating Oil</u>		<u>Diesel Oil</u>	<u>Steam Cylinder Oil</u>
	<u>SS-1103</u>	<u>SS-1106</u>		
Viscosity °Engler at 100°C.	3.0	5.5-5.6		6.0
SSU at 210°F	105	200-205		220
Viscosity Index	115-124	108-112		115-116
Flash Point °F	428 min.	482-500		572-590
Pour Point °F	-22 max.	13 max.	32±2	-4 max.
Conradson C.Res.	0.2 max.	0.2 max.		0.4-0.5
Iodine No.				20
Cetane No.			72-73	
IBP			302 min.	
Sulphur %			0	

Yields:

The ultimate yields from the original paraffin are summarised as follows:

<u>Weight per cent</u>	<u>Type of Operation</u>	
	<u>SS-1103</u>	<u>SS-1106</u>
Gas from Cracking	27.0 - 30.0	27.0 - 30.0
Finished Lubricating Oil	49.0 - 52.5	49.0 - 52.5
Diesel Oil	7.0	5.6
Steam Cylinder Oil	8.4	11.2
Unaccounted for	8.6 - 2.1	7.2 - 0.7
Total:	100.0	100.0

XIII. AVIATION FUEL MANUFACTURE AND ENGINE TESTING.

Introduction:

The following information was obtained by interrogation of Dr. Ester, Mr. Scholz and Mr. Honicker.

The test engines originally installed at Leuna have now been evacuated to Wethau and Stossen. These locations were visited.

Octane Number Determination and Specification:

For day-to-day control of aviation gasoline component quality, Octane Number tests were carried out on C.F.R. Motor Method engines and on the I.G. Prufmotors; the latter being used almost exclusively as spare parts for the C.F.R. engines became impossible to obtain. In 1944 there were 170 of these engines in use in Germany.

The Octane Number specifications in force in early 1945 were as follows:

D.H.D. Gasoline	78	Octane No. clear
Hydro Gasoline	70	" " "
	87	" " with 0.09% vol. T.E.L.

I.G. Prufmotor

Swept volume	342 ccs.
Compression Ratio	Variable up to a max. of 14:1
R.p.m.	900 for Motor Method 600 for Research Method
Spark Advance	26° for Motor and Research Method
Jacket Temperature	100°C for Motor Method 100°C for Research Method
Mixture Temperature	165°C for Motor Method Room temperature for Research Method

This engine is used with a multi-bowl carburettor in the usual way and all Octane Number determinations are made at max. knock mixture strength. Bouncing pins and knock meters made by Siemens are employed. The normal method of carrying out Octane Number determinations is to adjust the mixture strength to max. knock; increase the compression ratio until a standard rating on the knock meter is obtained; measure this compression ratio by a conventional micrometer and determine Octane Number directly from a standard chart in which Octane Number is plotted against compression ratio. The Octane Number can also be directly read from a scale attached to a mechanism geared to the movable cylinder head.

This engine has also been used for supercharged tests and 5 to 10 supercharged engines were stated to be in use in various parts of Germany. This development followed initial tests in 1941 in Oppau, and Leuna first carried out supercharged tests of this nature at the end of 1941. In these tests pressure fuel tanks and a pressure carburettor are employed together with an