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 (V4A), 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-81" (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-81" (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 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:-

Description :	SS-903 Oil	SS-906 Oil
Inspection :		
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.
Vorlauf-Öl	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 tons
Fuel Gas (2000 kg.Cal/M³)	250 M³
Electricity	150 KWH
Water	150 M³

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 pfg. per kg. from natural gas (methane) by the electric arc process used at Hüls, 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.