XIIA. SYNTHETIC LUBRICATING OIL MANUFACTURE AT STETTIN-POLITZ

Source 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	90-100

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 preheater 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	

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 125 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 1103 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:

	SS 1103	SS 1106	
$AlCl_3$ 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 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:

	Lubricating Oil SS-1103	Lubricating Oil SS-1106	Diesel Oil	Steam Cylinder Oil
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.			72-75	
FBP °C			302 min.	
Sulphur %			0	

Yields:

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

	Type of Operation	
Weight per cent	SS-1103	SS-1106
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