Attachment III

Copolymerization of SS Oil (Synthetic Ethylene Polymer) with Mineral Oil

(I. G. Leuna - February 1, 1943)

Confidential

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In the overall evaluation of the copolymerization process, the ratio of crude ethylene polymer to mineral oil taken was given primary consideration with reference to the quantitative side of the reaction. Investigation of the quality of the products and their usefulness as aircraft oils is still in progress. The mineral oil component taken was a distillate from a mine in Hauskirchen (Lubricating Oil Distillates 1-3), dewaxed and deasphalted with propane but not treated with sulfuric acid or refined by extraction, which had the following properties:

d (density at 20°C) = 0.915 Flash Point = 220°C

V 20 (viscosity at 20°C) = 53.3°E Pour Point = -18°

V 38 = 15.46°E Acid No. = 0.28

V 50 = 7.93°E Saponification No. = 1.23

V 99 = 1.826°E Conradson Carbon = 1.09%

VI = 54.5

The reaction was conducted in the following manner very similarly to the earlier experiments of Dr. Zorn and Dr. Haag at Oppau. Ethylene was polymerized in an autoclave as usual. After polymerization was completed, the total crude product, while still at a temperature of 110°C, was stirred together with mineral oil which had been preheated to 90-150°, and the mixture was stirred at the temperature of the reaction for three hours. For determining properties on purified ethylene polymer, it was necessary to withdraw a small test sample before mixing and to process it. Account was taken of this in the yield calculation.

Pertinent data are presented in the following table. Definitions and derivations of the values given in the various columns are explained below:

Charge in kilograms		Yield	kg	Charge to Copolymerization			Yields	
1. Vorlauf Oil (Low boiling Hydrocarbon Recycle Stock)	2. $AlCl_3$	3. SS-Polymer	4. SS-Test Sample	5. Crude Ethylene Polymer	6. Mineral Oil	7. Total	8. Crude Oily Product	9. Sludge
8	1.4	35.4	1.1	34.3	21.0	55.3	49.3	6.0
8	1.4	34.7	1.1	33.6	21.0	54.6	49.2	5.4
8	1.4	33.5	0.9	32.6	20.0	52.6	47.2	5.4
8	1.4	35.8	0.8	35.0	20.0	55.0	49.0	6.0
32	5.6	139.4	3.9	135.5	82.0	217.5	194.7	22.8

The figures in Columns 1, 2, 4, 6, 7, 8, and 9 were determined gravimetrically. The values in the other columns are calculated as follows:

Column 5 = total yield 7 - mineral oil charge 6.
Column 3 = Column 5 + test sample 4.
The total quantity of ethylene processed is calculated as 101.8 kg. (Column 3 - (1 + 2)).

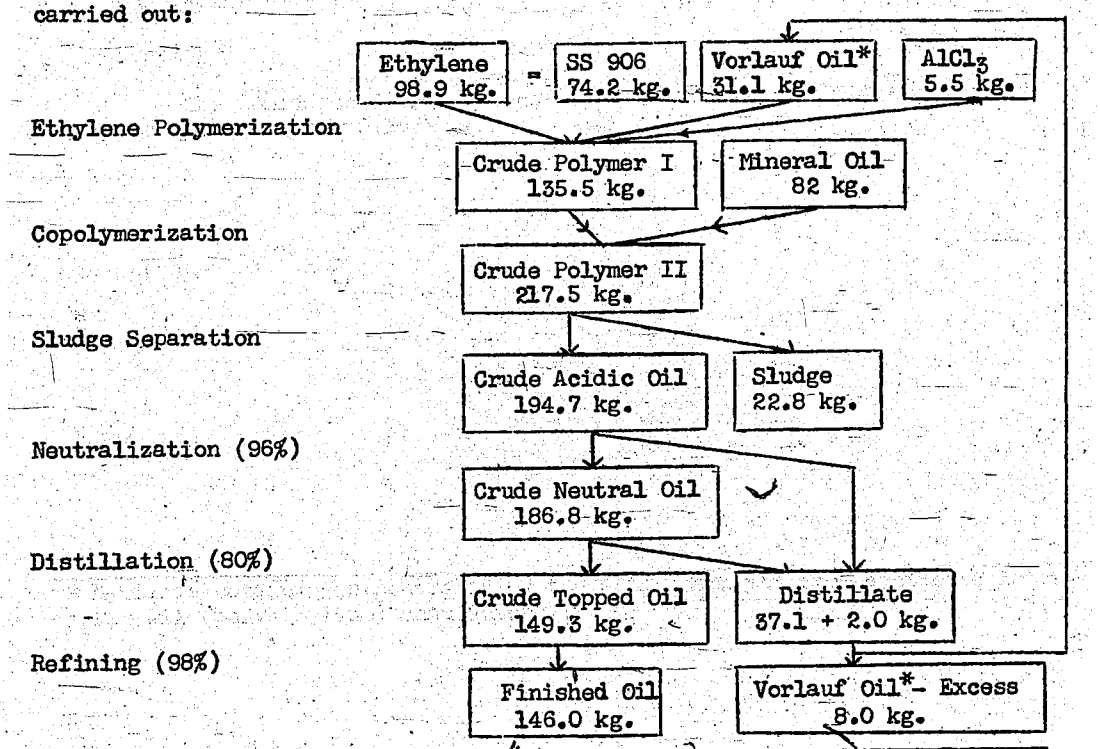
The ethylene contained in the charge to the copolymerization is calculated as 98.9 kg. (proportional to the total ethylene x $\frac{\text{Column 5}}{\text{Column 3}}$);

The "Vorlauf Oil" corresponded to 32 x $\frac{135.5}{139.4}$ = 31.1 kg.; aluminum chloride, to 5.6 x $\frac{135.5}{139.4}$ = 5.45 kg.

The crude reaction product is withdrawn while hot for further processing and allowed to stand for 12 hours, during which time the sludge precipitates out as a heavy asphaltic mass and can easily be completely separated. The supernatant acidic crude oily product is stirred with 0.5 per cent methanol, neutralized with 2 per cent slaked lime, and passed through a filter press. The yield of neutral crude oily product consistently amounted to 96 per cent in several laboratory scale experiments (100 kg. quantity). The bottoms obtained in the final vacuum distillation amounted to 80% of the charge (on the average, for all cases). The loss on refining was established as 2% at most.

According to experience at the Leuna SS-Oil Plant, a 75% yield of SS 906 could be obtained (on the basis of ethylene). For example, it was possible to obtain 74.2 kilograms of SS Oil from 98.9 kilograms of ethylene.

The following flow scheme was derived from the experimental work carried out:



*"Vorlauf Oil" is low-boiling hydrocarbon recycle stock.

The final product was obtained from 74.2 kg. of SS Oil = 50.8%
and 71.8 kg. of mineral oil = 49.2%,
i.e., the two components were in this case taken in a ratio of almost 1:1. It is to be considered that, when ethylene is employed for copolymerization, instead of R-Oil** being formed (to an extent of 7% on the basis of the finished SS-Oil), an asphaltic material is formed which must be disposed of by burning, for example, and does not yield any more lubricant.

The yield data were checked on a plant scale in Run No. 126.

Engine tests on the oils are still in progress. In the following table, properties of the copolymer are compared with those of the straight SS-Oil, as obtained on the small samples taken for analysis:

	SS 906 (Item No. 865,866)		Copolymer (MP 10-12)
Viscosity at 38°C (°E)	86.7	85.0	34.85
Viscosity at 99°C (°E)	5.69	5.68	3.11
V.I.	109.5	109.3	108
Flash Point (°C)	224°	206°	221°
Pour Point (°C)	-34°	-30°	-30°
Density	0.855		0.870

The Conradson Carbon values still fluctuate, but will eventually be found not to exceed 0.15.

** R-Oil is obtained on neutralizing the $AlCl_3$ catalyst from synthetic lubricating oil (polyethylene) manufacture.