

Attachment III

Ammonia Works Merseburg

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Ms 127

(Confidential)

Copolymerization of SS Oil with Mineral Oil

The conditions prevailing in the copolymerization of crude ethylene polymerizates with mineral-oil were to be checked, chiefly with regard to the quantitative side of the reaction. The quality of the resulting oils and their usefulness as aviation engine oils is an object of an investigation still in progress. The mineral oil component available was a mineral distillate (lubricating oil distillates 1-3) which had been dewaxed and deasphalted with propane but not pretreated with sulfuric acid, nor refined by extraction, originating from a Hauskirchen well and having the following properties:

d 20	=	0.915	Flash point	220°
V 20	=	53.3 °E	Solid. point	18°
38	=	15.46 °E	Acid number	= 0.28
50	=	7.93 °E	Sap. number	= 1.23
99	=	1.826 °E	Conradson coke	
V.I.	=	54.5	test	1.09 %

The reaction was carried out in a very similar manner to older experiments of Dr. Zorn and Dr. Haag in Oppau, as follows: ethylene was polymerized in an autoclave in the customary manner. After polymerization was complete the crude product, still at a temperature of 110°, was introduced, with agitation, into mineral oil heated to 90-150° and the mixture was kept 3 hrs., with agitation, at the reaction temperature. In order to test the properties of the pure ethylene polymerizate before mixing a small sample had to be taken and treated separately (which had to be taken into account in calculating the yield). A few batches are shown in the following tabulation; the significance and origin of the figures given in the different columns is explained in the following table.

Charge in kg	Yield	kg	Charge for copolymerization	Yield				
1. Light Oil	2. AlCl <sub>3</sub>	3. SS-Polymerizate	4. SS-Sple for anal.	5. Crude Ethylene polym.	6. Min. Oil	7. Total	8. Crude Oil	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 1,2,4,6,7,8,9 were determined gravimetrically.

The figures in other columns were calculated as follows:

Column 5 from total yield 7 - mineral oil charge 6.

Column 3 from column 5 + analysis sample 4.

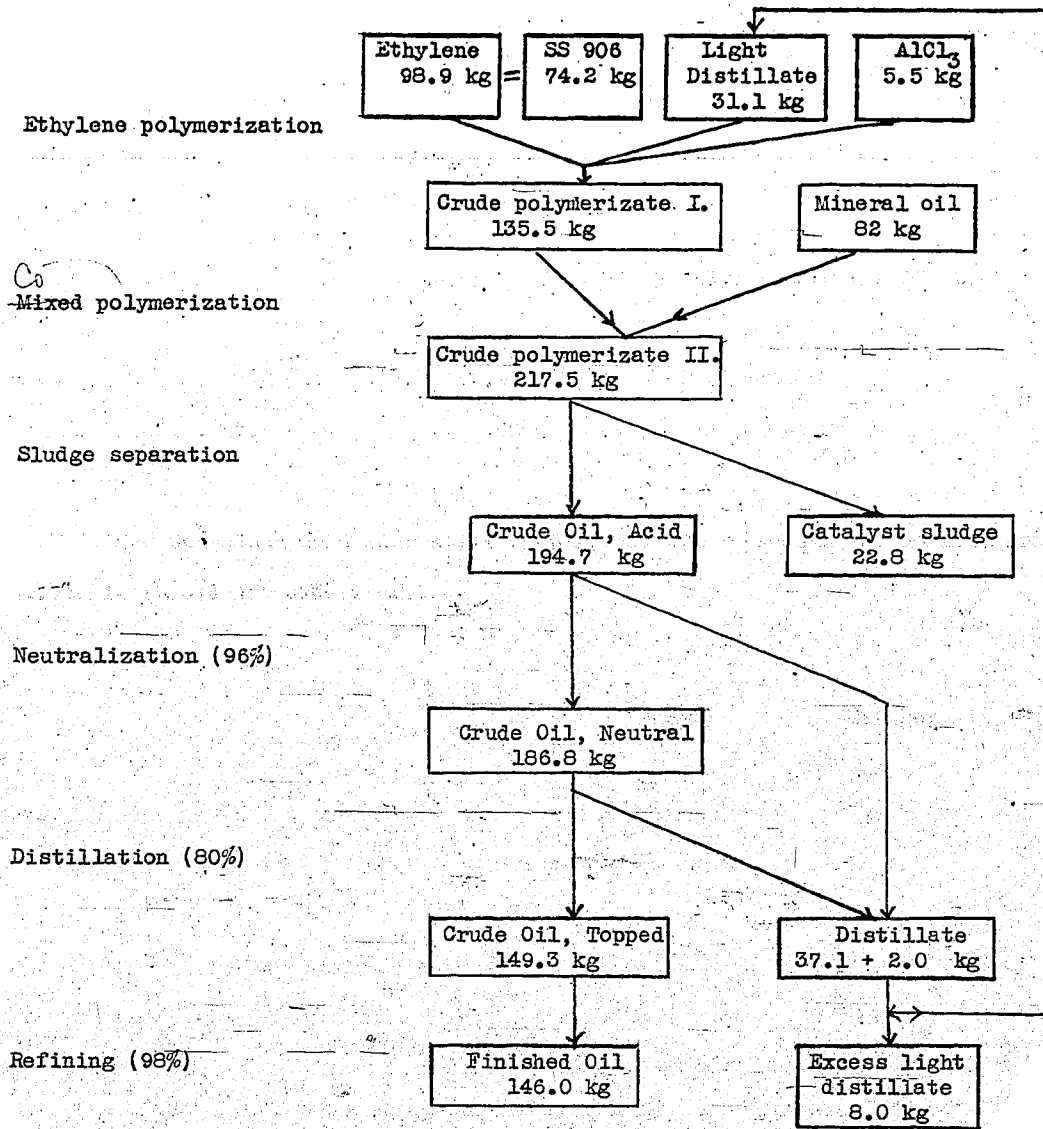
The total amount of ethylene treated is calculated at 101.8 kg from column 3-(1 + 2).

The ethylene contained in the copolymerization charge is calculated proportionally from the total ethylene  $\times \frac{\text{column 5}}{\text{column 3}}$  at 98.9 kg, the overhead distillate corresponding to  $32 \times \frac{135.3}{139.4} = 31.1$  kg, AlCl<sub>3</sub> to  $5.6 \times \frac{135.5}{139.4} = 5.45$  kg.

The crude reaction product was run off hot for further treatment and left to settle for 12 hrs., allowing the catalyst sludge to separate as a heavy asphaltic mass which could be readily and completely separated. The supernatant acid crude oil was mixed with 0.5% methanol, neutralized with 2% slaked lime and forced through a filter press. The yield was uniformly 96% of neutral crude oil from several charges, both on a laboratory scale and in 100 kg batches. After subsequent vacuum distillation the residue amounted on the average to 80% of the charge. The refining loss does not exceed 2%.

From practical experience in the SS oil plant at Leuna the yield of SS 906 from ethylene can be set at 75%. Consequently from 98.9 kg of ethylene, 74.2 kg of SS oil could be produced.

The following sheet is based on experimental work completed



The end product is composed of 74.2 kg of SS oil = 50.8 %  
and 71.8 kg of Min. oil = 49.2%,

the two components being used practically in the ratio 1:1.

When ethylene is used for copolymerization it should be noted that instead of a yield of R oil, to the extent of 7% of the finished SS oil, an asphalt is obtained which can be subjected to distillation but no longer gives any lubricants.

The yield figures are being checked in large scale experiments in Me 126.

The engine tests of the oil are still in progress. The following tabulation shows the constants of the copolymerizate in comparison with those of the pure SS oil, on the basis of analytical data.

	SS 906	(Charge N 865,866)	Copolymerizate (MP-10-12)
V 38	86.7	85.0	34.85
99	5.69	5.68	3.11
V.I.	109.5	109.3	108
Fl. pt.	224°	206°	221°
Solid. pt.	- 34°	- 30°	- 30°
Density	0.855		0.870

The Conradson coke test still varies, but after process refinements are complete it should not exceed 0.15.

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