Attachment IV

Leuna - Werke March 29, 1943
Confidential

Report

Production of Aviation Engine Oils By Copolymerization

In addition to the previously used processes for producing aviation engine lubricating oils,

1. entirely from mineral oil by appropriate processing of petroleum oil; or
2. by mixing a diluent, obtained from petroleum by appropriate treatment, with a highly viscous synthetic oil such as SS 906 in the ratio 1:1, we also made use of
3. copolymerization of crude SS oil with a pretreated mineral oil fraction, which produces a finished oil directly.

This process, which was developed by Dr. Zorn as far back as 1930 with crude polymerizates from cracked paraffin, is described in the following together with an outline of its qualitative and quantitative advantages.

Mineral Raw Material

The mineral oil fraction to be used for copolymerization should have a flash-point in excess of 225° and be dewaxed. Only small quantities of extract, say 6% of Ostmark petroleum, need be removed by selective solvent treatment, but in the production of the aforementioned oils and blended oils an extraction of at least 25% or more is necessary. For the purposes of copolymerization a larger portion of extractable products may remain in the mineral oil and are then partly converted to valuable lubricating oils during the course of the reaction, and partly separated with the $AlCl_3$. Consequently, copolymerization results in an important saving of valuable mineral oil.

Process:

As in the manufacture of SS oil, ethylene is polymerized, introduced into the preheated mineral oil and blended with it in a ratio of 1.5 parts of mineral oil to one part of pure SS oil. After completion of the reaction the bulk of the sludge is withdrawn after a settling period of about 2 hours, while the remainder is separated in a centrifuge. The aluminum chloride is removed from the sludge by washing with water in the presence of a solvent and a black, asphaltic, soft mass is obtained, the so-called sludge oil. R-Oil suitable for the manufacture of journal oil is not obtained under these conditions. The possible use of this sludge oil in the rubber and lacquer industries is still being investigated. The sludge-free, acid crude oil is neutralized by mixing it with hydrated lime, which is removed in filter presses while the neutral crude oil is topped and the distillation residue subjected to a final treatment with bleaching clay. The process is illustrated in flow sheets 1 and 2. Scheme 2, in particular, shows that the copolymerization component obtained in the treatment of 100,000 Jato* petroleum oil is just sufficient to process the SS oil obtained in the SS oil plant Heydebreck I to copolymerized oil.

p.2 The experiments on which this scheme is based were carried out on an experimental scale with 200 kg,and with 4 tons on a larger scale.

The process may be adjusted to the variable viscosities of the SS 900 oil and of the mineral oil by selecting different ratios. In scheme II the ratio of synthetic oil to mineral oil is 1:1.5; in scheme III the ratio is 1:1. In the latter case the amount of finished aviation engine oils obtainable from 100,000 Jato petroleum oil increases to 60,970 tons.

Oil Analysis:

The mineral oil fraction used in the aforementioned experiments was prepared from crude oil, vacuum fractions 1, 2, 3 of an Ostmark petroleum. Fractions

*Jato = metric tons per year.

2 and 3 were de-resined with propane, and the 1-3 mixture then dewaxed with ethylene chloride. As indicated by the coke test, very little asphalt is removed. Extraction with 6% of phenol improves tests and treatment.

	Density	E_{50}	E_{99}	V.I.	Fl.pt.	Solid.pt.	Coke test
Min. Oil-Copolym. Component	0.915	7.93	1.83	55	220°	- 18	-
SS 970 r (Red Ring)	0.874	18.50	3.11	106	230	- 30	0.20
Copolymerizate	0.878	18.45	3.08	106	230	- 28	0.18

Consequently the oil obtained by copolymerization is analytically equivalent to the Red Ring oil (SS 970 r) used at present.

Operation in Motors:

A series of test runs was made in the BMW one-cylinder engine in the Oppau Engine Testing Laboratory with the following results.

Running time with Red Ring D (Reference oil)		7 hours
	SS 970 r	12 hours
	Copolymerizate MP r	15 "

In the processing of 100,000 tons of Ostmark crude oil the following quantities of finished aviation engine oils are obtained depending on whether this crude oil is used as a diluent or as a copolymerization component:

	A	B	
Mixing ratio Min. oil : SS 906	Physical Mixture tons	Copolymerization tons	Additional Production copolymerizates tons
50 : 50	26 000	60 790	34 970
60 : 40	21 700	49 760	28 060

The following quantities of raw materials are required for the production figures given in this table.

	A		B	
Min. Oil : SS 906	Diluent	SS 906	Copolymerization component	SS 906
50 : 50	13 000	13 000	32 780	32 780
60 : 40	13 000	8 700	32 780	21 900

Consequently, in the copolymerization process finished aviation engine oils are obtained which both in quality to quantity are superior to products obtainable by the older method. Furthermore, the two above tables show that the copolymerization process always gives a much higher yield of finished aviation engine oils, regardless of the mixing ratio.

In the following we shall see how the lubricating oil plan of the I. G. is altered by the introduction of copolymerization. This plan had heretofore included the following products:

	SS 906	R-Journal Oil	
Leuna I	10 000	700	4 000
Leuna II	7 500	520	
Schkopau	10 000	700	2 500
Heydebreck I	22 000	1 540	
Heydebreck II	10 000	700	
Moosbierbaum	4 000	280	
Oppau	2 000	140	
Auschwitz	-	-	4 000
	65 500	4 580	10 500

The modification of this plan through the adoption of the copolymerization process was governed by the necessity of using the smallest possible quantities of iron. Therefore, the introduction of the MP process* at Leuna was abandoned because the present installations would have to be considerably enlarged. Furthermore, since ester oils are to be produced in Leuna and Schkopau, it seemed preferable to use the Leuna SS oil plant in combination with the two ester plants for the manufacture of special aviation engine oils.

At Heydebreck II the introduction of the MP process was also temporarily

* MP process = Copolymerization process.

abandoned because of the ester oil plant at Auschwitz. This plant is also to be used principally for the manufacture of special engine oils.

The excess of SS oils relative to the ester oils is then to be blended with a mineral diluent (e.g. from Pressburg) for finished aviation engine oils.

The following plants using the copolymerization process thus remain.

1.) Schkopau
2.) Heydebreck I
3.) Moosbierbaum
4.) Oppau.

No decision has so far been made with regard to Oppau. It is possible that the synthesis might be integrated with a mineral oil refinery located there. Tentative construction plans have been worked out for 1, 2 and 3, based on copolymerization in the ratio: Synth. oil;min. oil = 40:60.

The following production figures apply to these three plants.

	Components required SS 906	Min. oil component	Finished MP Aviation Engine Oil	Sludge oil	Journal oil deficiency
Schkopau	10 000	15 000	22 750	2 250	- 700
Heydebreck I	22 000	33 000	50 050	4 750	- 1540
Moosbierbaum	4 000	6 000	8 000	900	- 280
	36 000	54 000	80 800	8 100	- 2520

According to Flow sheet 1 the following quantities are obtainable:

100 parts SS 906 ⟶ 227.5 parts copolymerizate, for which are required 150 parts mineral component = 458 parts crude oil. Similarly, 200 parts copolymerizate require 138 parts mineral oil component = 423 parts crude oil.

If 100 parts SS 906 are blended as before with the diluent in the ratio 1:1, 200 parts blended oil are obtained. For this purpose 100 parts mineral oil component are required. Basing our estimate on a Duosol process yield of 13,000 tons diluent from 100,000 tons of crude oil, 777 parts of crude oil will be required for 200 parts of blending oil.

According to the original production plan, 65,500 tons of SS 906 are to be produced.

65,500 tons SS 906 = 131,000 tons blended oil

65,500 Duosol-dilution oil = 508,935 tons of crude oil.

If this quantity of SS 906 were to be processed to copolymerizate there would be obtained 655 x 227.5 = 149,000 tons of MP oil, equal to 18,000 tons of finished aviation engine oil in excess of the given synthetic oil capacity. 149,000 x 0.69 = 102,810 tons of mineral oil component = 315,880 tons of crude oil would thus be necessary.

The overall picture is as follows:

65,500 tons of SS 906 → 131,000 tons Blended Oil ～ 508,935 tons Crude Oil

65,500 tons of SS 906 → 149,000 tons MP Oil ～ 315,880 tons Crude Oil

The I. G. Lubricating Oil Program will thus be as follows:

	MP -Oil	Mipoko*	SS 906	Diluent	Sludge oil	Journal oil
Leuna I	-	-	10,000	10,000	-	700
Leuna II	-	-	7,500	7,500	-	520
Schkopau	22,750	15,000	-	-	2,250	-
Heydebreck I	50,050	33,000	-	-	4,950	-
Heydebreck II	-	-	10,000	10,000	-	700
Moosbierbaum	9,100	6,000	-	-	900	-
Oppau	-	-	2,000	2,000	-	140
	81,900	54,000	29,500	29,500	8,100	2,060

To summarize, the following two production plans are contemplated.

* Mipoko = copolymer component.

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	Plan I	Plan II
MP - Oil	-	81,900
Mipoko	-	54,000
Crude Oil	-	173,000
SS 906	65,000	27,000
Diluent	65,000	27,000
Crude oil	508,935	209,800
Sludge oil	-	8,100
Journal oil	4,580	4,560
Finished aviation oil, total	131,000	135,900
Crude oil requirements, total	508,935	382,800

The 54,000 Mipoko (copolymer component) provided for in the above plan II will be produced in a new refinery to be built at Moosbierbaum with a capacity of 35,000 Jato (tons per year) from 100,000 tons crude oil, and in the Apollo at Pressburg (capacity 21,000 Jato).

When the Apollo phenol extraction plant is ready for operation in the spring of 1944 it will be possible to produce 21,000 tons of Mipoko directly. In Moosbierbaum a treating plant for about 100,000 tons of crude oil is to be built from equipment taken from French refineries. In projecting this plant a strict program has been followed for using the smallest possible amounts of iron, but the construction and installation of the individual production units is such that extension and expansion is possible with minimum quantities of iron.

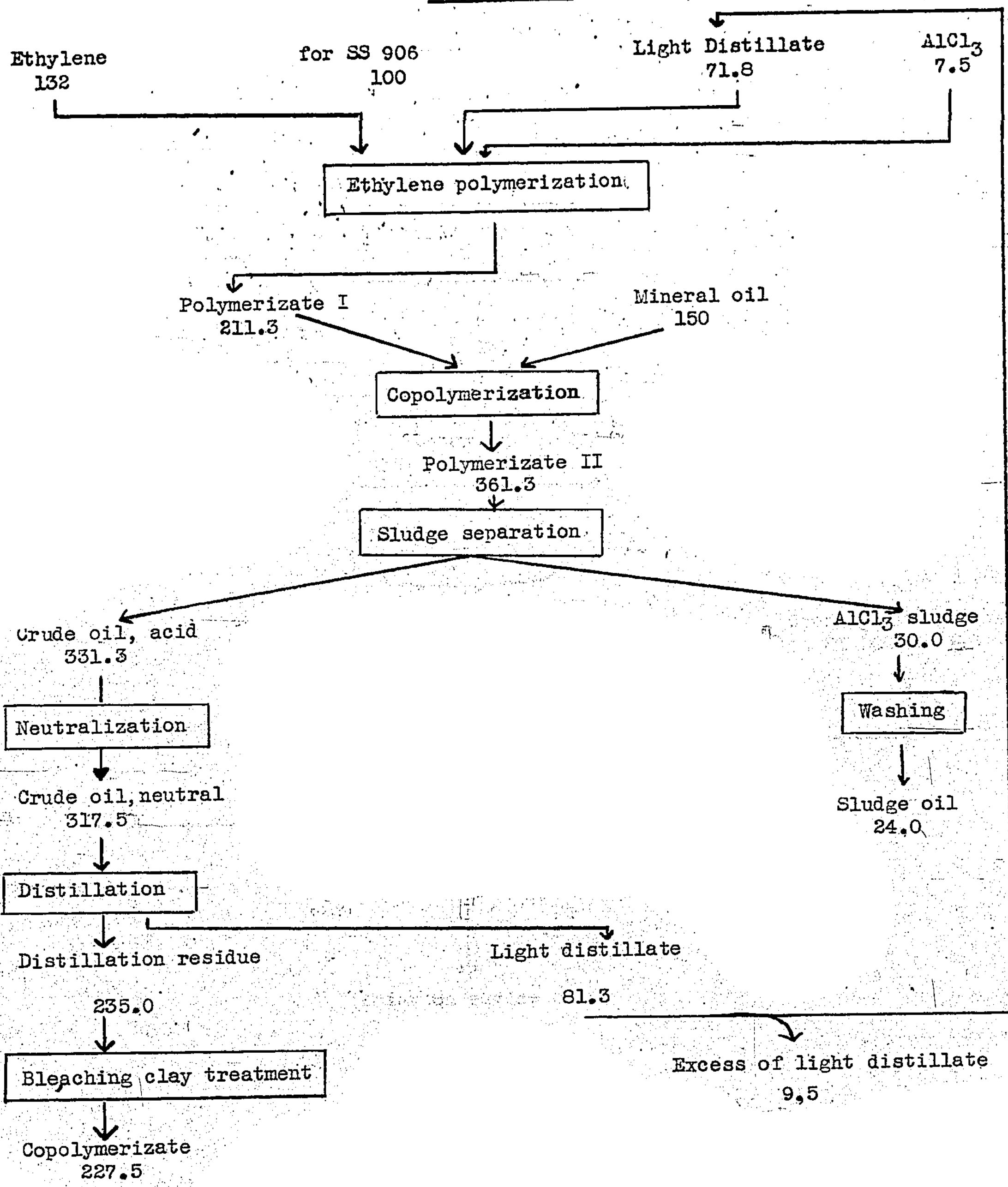
At first it was planned to produce only 33,000 tons of the copolymerization component. For this purpose lubricating oil fractions, 1, 2 and 3, were to be used. Processing of the lubricating oil fraction 4 to steam cylinder oil must be at present omitted in order to save iron. This fraction can be sent either to the Apollo for further treatment or remain in the distillation residue. The latter is to be sent to the hydrogenation plant at Pölitz to be converted into gasoline and Diesel oil.

The following plants will be constructed in Moosbierbaum:

1. A combined Foster-Wheeler topping and vacuum distillation plant, complete from the French Gravenschon refinery. Capacity 100,000 Jato.
2. A phenol extraction plant from the Port Jérome refinery. In this plant a new extraction tower is to be erected.
3. A dichlorethylene dewaxing plant from the Port Jérome refinery.

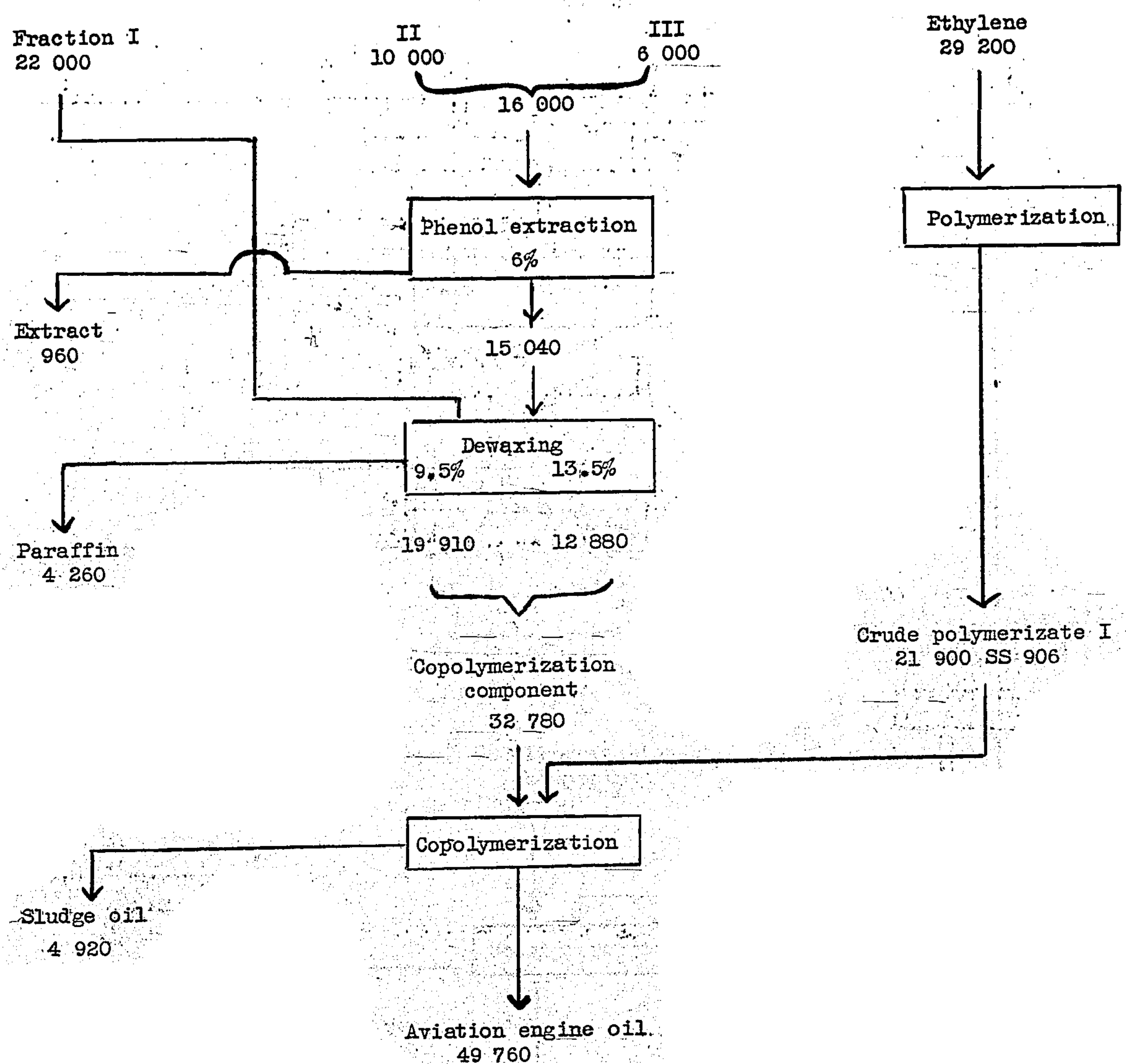
The gasoline-kerosene and gas oil fractions are to be treated in equipment of the HF plant. The resulting crude paraffin is to be first converted into pure paraffin in the Apollo unit. Tank storage, pipe connections, pipelines, pumps, etc. are to be taken as much as possible from the French refinery of Port Jérome.

Flow Sheet 1

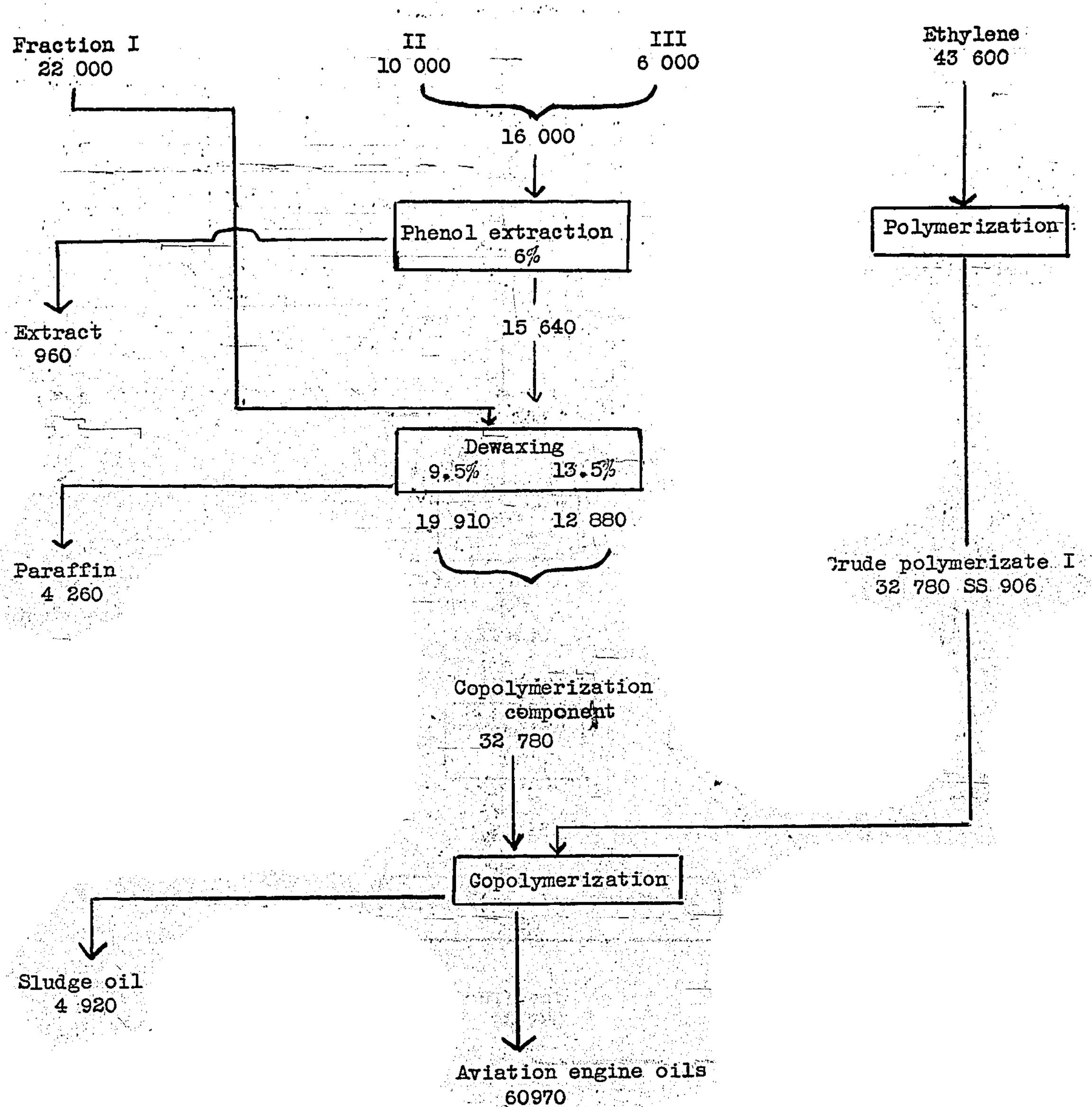


Ethylene
132
for SS 906
100
Light Distillate
71.8
$AlCl_3$
7.5
Ethylene polymerization
Polymerizate I
211.3
Mineral oil
150
Copolymerization
Polymerizate II
361.3
Sludge separation
Crude oil, acid
331.3
$AlCl_3$ sludge
30.0
Washing
Sludge oil
24.0
Neutralization
Crude oil, neutral
317.5
Distillation
Distillation residue
235.0
Light distillate
81.3
Excess of light distillate
9.5
Bleaching clay treatment
Copolymerizate
227.5

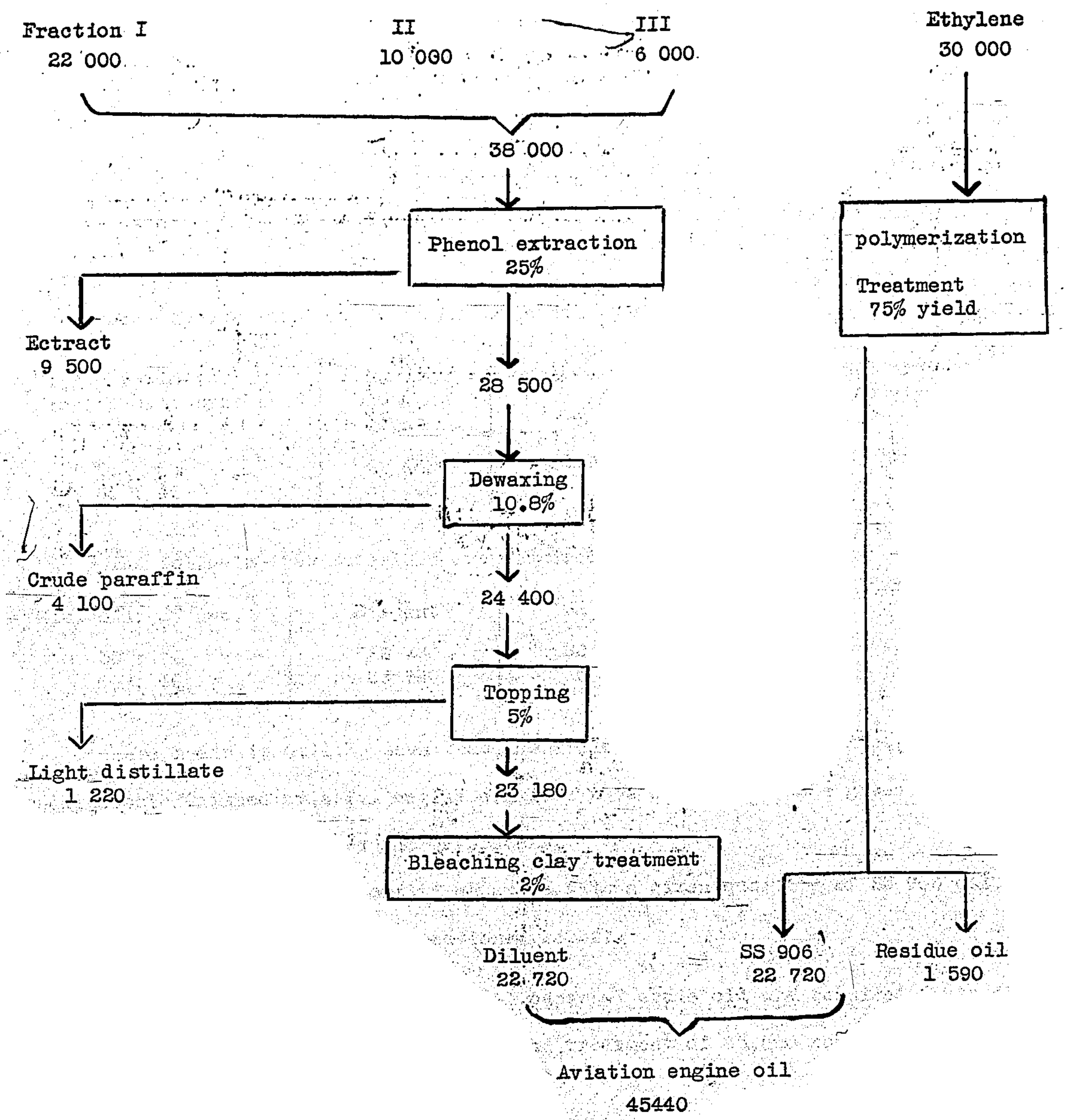
Flow sheet II



Flow sheet III



Flow sheet II a



Appendix to the report: "Manufacture of Aviation Engine Oil by Copolymerization"

dated 3/29/43

In this report the diluent required for physical blending has been assumed to be 13%, referred to the crude oil. This yield applies to the Duosol process.

Should it be possible to increase the yield of the diluent, as illustrated in the attached Flow Sheet IIa, the comparative figures for copolymerization follow:.

From 100,000 tons of Ostmark crude oil there is obtained:

Mixing ratio Mineral oil: synthetic	A Physical blending tons	B Copolymerization tons	Additional production through copolymerization tons	%
50 : 50	45 440	60 970	15 530	34
60 : 40	37 900	49 760	11 960	32

The following raw materials are required:

Min. oil: SS 906	A Diluent	A SS 906	B Mipoko	B SS 906
50 : 50	22 720	22 720	32 780	32 780
60 : 40	22 720	15 180	32 780	21 900

Here again it will be seen that, regardless of the mixing ratio, about 30% additional finished aviation engine oil is always obtained from a given amount of crude oil (100,000 tons) in the MP process.*

The picture is different if we start with a given quantity of SS 906 oil, as is shown on page 4 of the aforementioned report.

For 100 parts of diluent only 441 parts of crude oil are required instead of 777. In this instance the picture for the treatment of 65,500 tons of SS 906 is as follows:

* Copolymerization process.

	Plan I	Plan II
MP oil	-	81 900
Mipoko	-	54 000
Crude oil	-	173 000
SS 906	65 500	27 000
Diluent	65 500	27 000
Crude oil	288 855	119 698
Sludge oil	-	8 100
Journal oil	4 580	4 560
Finished aviation oil total	131 000	135 900
Crude oil consumption, total	288 855	292 698

Here the additional production of 4,900 tons of finished aviation engine oil no longer results in a simultaneous saving of crude oil, but the crude oil requirements in both cases are practically the same. However, the better quality and the economical advantage of the MP oils are important factors to be considered.

Since at the present time the diluent is not obtainable in this high yield and in the required amount, the development of the MP process is important.