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THE MANUFACTURE AND APPLICATION OF LUBRICANTS IN GERMANY

1. Introduction.

In allied nations there have been many technical developments during recent years in the manufacture and application of lubricants, both because of new demands and because this field has been a subject of extensive chemical research. Because of its highly industrialized and mechanized economy, it was to be anticipated that Germany also would have given great attention during recent years to the development and manufacture of lubricants.

Whereas in America and in most other nations petroleum is the source of essentially all liquid fuels and lubricants, in Germany the bulk of liquid fuels and some lubricants were known to have come from synthetic processes. It was expected that new developments would have occurred at least in the field of synthetic lubricants.

To obtain a comprehensive view of German developments in this field, technical personnel of all of the producers of synthetic lubricants were found and interrogated and most of the plants producing these synthetic lubricants were visited.

To augment and substantiate the information gained from interrogation and observation, many documents, reports and descriptive papers were accumulated, and samples of some of the newer synthetic materials were obtained.

In the following sections are discussed the supply and composition of German lubricating oils, the manufacturing methods used for mineral oils, and the development and production of synthetic oils and their components. New additive developments are described and discussed.

It should be pointed out that in reporting herein the information gained from many sources and under many different circumstances, the opinions of those contacted were obviously reflected in the information given. Further, in some cases it has not been possible to obtain or present documents with test data to confirm opinions and contentions.

1. Introduction (Cont'd.)

Substantiating data will be desirable in many instances using analyses and test procedures that are in standard use in America.

2. Commercial Supply and Composition of German Lubricating Oils.

In Table I is shown an approximate breakdown of the German supply of lubricating oils from petroleum and the various synthetic processes. These figures were obtained from documents and from interrogation of a government oil planning board official (Dr. Butefisch). The volume figures apply to the period of about 1942. Subsequent to 1942, efforts were made to increase by four-fold the production of aviation oils, and for this program many plants were still being constructed in 1944, when the Allied bombing raids began to be concentrated on the oil industry. Hence, in 1943 the supply of oils may have exceeded somewhat the figures in Table I.

Despite the development of several types of synthetic lubricating oils, it will be seen that the bulk of the commercial volume still came from petroleum. The crude petroleum used for the production shown in Table I was from Germany, Austria, Hungry and Rumania.

With the exception of the small volume produced by Rheinpreussen, the synthetic lubricating oils went almost entirely to aircraft oil. The aircraft oil supplied to the Luftwaffe was normally a mixture of equal parts of synthetic and highly refined mineral oil. The specifications for aircraft oil and some approximate analytical data on the synthetic and mineral oil components and the blend thereof are shown in Table II. This mixed oil was normally used without additives or inhibitors.

For engines other than aircraft, in general, refined mineral oils only have been used in Germany. An exception was the use by the German Navy of the Rheinpreussen synthetic lubricating oil in diesel engines. Gasoline and diesel engines were normally supplied with the same grades of oils with no effort being made to supply a special oil for diesels. It was the general opinion of both oil and engine people in Germany that the well refined mineral oils were adequate for today's diesel engine requirements. Again, no special additives

TABLE I

SUPPLY OF GERMAN LUBRICATING OILS
(all figures are in barrels per day)

SUPPLIER	METHODS OF MANUFACTURE	VOLUME OF SUPPLY	DISPOSITION AIRCRAFT OILS	DISPOSITION OTHER
Various mineral refiners	Conventional refining methods	15750	750	15000
Brabag Zeitz	TTH Hydrogenation of tars	600	-	600(1)
Pölitz	Olefin Polymerization	250	250	-
Rhenania Ossag	Olefin Polymerization	200	200	-
Ruhrchemie	Olefin Polymerization	200	-	200(2)
Rheinpreussen	Aromatic-Parrafin condensation	50	-	50(3)
IG-Leuna	Ethylene Polymerization	300	300	-
IG-Leuna	Ester Synthesis	100	100	-
	Totals	17,450	1,600	15,850

(1) Relatively low quality, used for industrial and automobile lubricants.
(2) Used by Army for trucks, tanks, etc.
(3) Consumed entirely by German Navy for Diesel engines.

2. Commercial Supply and Composition of German Lubricating Oils. (Cont'd.)

or inhibitors were included.

Two (2) grades of motor oil were supplied to the Wehrmacht, one for winter and one for summer use. The winter grade had a viscosity of approximately 52 S.S.U. at 210 degrees fahrenheit and a viscosity index of about 100, while the corresponding values for the summer grade were 62 and 95. The great difficulty encountered in Russia in the winter of 1941-42 with cold starting of trucks and tanks when using these winter motor oils was an impetus to the synthetic ester oil development which is discussed later.

It will be noted in Table II that the synthetic component is much more viscous than the mineral oil component. Since the viscosity of the synthetic oils can be rather easily controlled, these oils were made viscous so that mineral oil bright stocks (high viscosity fractions) could be released for industrial purposes.

The synthetic oils have been applied in small volume to many specialty uses which are discussed in connection with the individual syntheses.

Because of the low temperatures encountered in Germany's war campaign, lowering of the pour point of lubricating oils was desired for almost every application. Special problems concerned the supply of a low temperature lubricant for torpedoes, a low temperature oil for weapons, a water-soluble oil for use with torpedoes, and a journal oil for the German railroad. These problems all were solved through the supply of special synthetic products.

The lubricating oils used in normal automobile service and for general industrial equipment apparently were little different from those used in America.

3. Lubricating Oil from Petroleum.

It was seen in Table I that raffinates from mineral oil were still the preponderant source of lubricants in Germany. These oils came from a variety of crudes and via many different refining processes.

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TABLE II

PROPERTIES OF GERMAN AIRCRAFT OIL

	Air Ministry Specifications for Aircraft Oil	Typical Synthetic Oil from Olefin Polymerization	Typical Refined Mineral Oil Fraction	Typical Mixture of Equal Parts of Synthetic & Mineral Oils
Density at 68°F.	0.895 max	0.84	0.89	0.875
Viscosity, SSu at 212°F.	95 min	215	57	105
Viscosity, SSu at 122°F.	580-660	1450	250	590
Viscosity Index	98 min	120	98	110
Pour Point, °F	-4 max	-7	± 0	-13
Flash Point, °F	437 min	604	437	464
Fire Point, °F.	437 min	670	500	534
Conradson Carbon % wt.	0.25 max	0.38	0.16	0.22
Ash Content, % wt.	0.01 max	0.001	0.00	0.001
Neutralization Number, Mg. KOH/g.	0.06 max.	0.03	0.03	0.03
Saponification Number, Mg. KOH/g.	0.2 max.	0.11	0.11	0.08

3. Lubricating Oil from Petroleum, (Cont'd)

The crude oils processed for lubricating oil manufacture included Hannover (Germany), Zistersdorf (Austria), Rumanian Pakura (distillation bottoms from Rumanian crude), Hungarian and Galizian.

Dewaxing and solvent extraction were employed by all of the major producers. Propane and ethylene dichloride dewaxing were employed. Solvent extraction methods included furfural, SO_2- benzol, phenol and propane (Duo-sol), and phenol as a single solvent.

Aside from the medium and low VI oils made for industrial uses, the bulk of the lubricating oil processed from petroleum was refined to a VI in the region of 80 to 100. Because of the good native quality of the lubricating oil fraction in the crudes being processed, the production of high VI products did not entail large losses to selective solvent extracts. For example, in the refining of Zistersdorf crude, the final yield of deasphalted, dewaxed, solvent extracted, acid treated, clay filtered lubricating oil was 50 percent by weight of the gas oil free residue from the crude topping units.

Many of the German crude oil refineries were built by American construction firms and most of the lubricating oil refining processes were operated under license from American or Allied companies.

The following documents transmitted to the Bureau of Ships, relates to this subject:

I. Brochaus Plan
(Material balance and flow diagram of a refining system manufacturing lubricating oil from an Austrian crude oil).

4. Mixed Polymerization Process applied to Lubricating Oils from Petroleum.

A new synthetic process involving mineral oil lubricating fractions is that of "mischpolymerisation" or mixed-polymerization. It was developed primarily by I. G. and was described by them in reports of 1942 and 1943.

4. Mixed Polymerization Process applied to Lubricating Oils from Petroleum.

A deasphalted and dewaxed but not solvent extracted lubricating oil distillate is reacted with a synthetic polymer of ethylene or other olefins, such as those obtained from the thermal cracking of waxes. A mixture of roughly equal parts of the two components is allowed to react in the presence of $AlCl_3$. The product is an oil of superior quality. The yield exceeds substantially that which could be obtained by mixing the olefin polymer and the mineral oil fraction after such solvent extraction that the mixture would have the same VI as the product of mixed polymerization.

An application worked out by I.G. involved the treatment of a lubricating oil distillate from a German crude oil. To prepare 1.0 volumes of oil meeting aircraft oil specifications by solvent extraction, 1.5 parts of lubricating oil distillate (deasphalted and dewaxed) were needed. This would require the discard of 0.5 parts as extract. However, by mixed polymerization of the 1.5 volume of distillate with 1.5 volumes of ethylene polymer, an approximately 3.0 volume yield of oil could be obtained equal in quality (both by laboratory and full scale engine tests) to either the ethylene polymer itself or the solvent extracted mineral oil fraction. Thus, the yield of aircraft oil could be increased from 2.5 to 3.0 volumes; i.e., 0.5 volumes of low grade extract could be made into aircraft oil.

To accomplish the mixed polymerization reaction, ethylene is first polymerized with $AlCl_3$ (described in detail in a later section on manufacture of Ethylene Polymers) The polymer product - $AlCl_3$ mixture, containing about 5 percent weight $AlCl_3$ is mixed in an autoclave with an equal weight of mineral oil. A temperature of 210 to 250 degrees fahrenheit is maintained and the mixture is agitated for 3 to 4 hours. The reaction is explained as being primarily between aromatics, (and any olefins present) in the mineral oil and the olefin polymers of ethylene. The reaction product is then freed of $AlCl_3$ and neutralized. Fractionation may or may not be necessary, depending upon whether or not any low boiling components entered the ethylene polymerization system.

The mixed polymerization process is claimed to yield oils which are actually superior in engine ring-sticking performance to a mixture of the ethylene polymer and a heavily extracted mineral oil

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TABLE III

MIXED POLYMERIZATION TREATMENT OF

A LUBRICATING OIL DISTILLATE FROM PETROLEUM

	Deasphalted and Dewaxed Petroleum Distillate	Ethylene Polymer	Mixed Polymerization Product of equal parts of Ethylene Polymer and Petroleum Distillate
Input, parts by wieght	52.5	47.5(1)	-
Outturn, parts by weight	-	-	93.5(2)
Density at 68°F.	0.915	0.855	0.870
Viscosity, S.S.u. at 100°F.	540	2950	1200
Viscosity, S.S. u. at 210°F.	58	205	108
Viscosity Index	54	109	108
Flash Point, °F.	428	428	428
Pour Point, °F.	±0	-22	-22
Conradson Carbon, % wt.	1.09	0.08(est)	0.15

(1) The input is expressed as the amount of ethylene polymer of this quality that would have been produced by the ethylene charged. To produce 47.5 weights of this polymer oil, 63.3 weights of ethylene is required.

(2) In addition 5.1 weights of low molecular weight material is produced.

4. Mixed Polymerization Process applied to Lubricating Oils from Petroleum, (Cont'd.)

raffinate. (See section on "Ethylene Polymers as Lubricating Oils" for description of ring-sticking test.)

Some brief yield and analytical data on the mixed polymerization treatment of a fraction of Hauskirchen crude oil are given in Table III.

In 1943 the German government had ordered I. G. to proceed with plants at Leuna, Heydebrek, and Moosbierbaum to produce a total of about 1500 barrels per day of aircraft lubricating oil by the mixed-polymerization process. The olefin raw material was to be ethylene. However, because of subsequent war developments, none of these plants was completed.

The following documents, transmitted to the Bureau of Ships, relate to this process:

II Uber das Prinzip der Schmierol - Mischpolymerisation. ((I.G. Leuna-Aktennotiz of January 12, 1942).
III Mischpolymerisation non SS-Ol mit Mineralol (I.G. Leuna February 1, 1943).
IV Herstellung von Flugmotorenol durch Mischpolymerisation. (I. G. Leuna-Bericht of March 29, 1943).

5. The TTH Process for Lubricating Oil Manufacture

The TTH process will be mentioned here only because it was a source of some of Germany's lubricating oil (see Table I). The process is being fully described in a report of the U. S. Navy Technical Mission in Europe entitled "The Production of Synthetic Fuels by the Hydrogenation of Solid and Liquid Carbonaceous Materials."

The TTH process (Tief-Temperature Hydrierung) is a mild high pressure hydrogenation of braun coal tar. Instead of yielding gasoline as the main end product, as is the case with most coal and coal tar high pressure hydrogenation operations, this process is a milder hydrogenation and yields substantial volumes of diesel fuel, lubricating oil, and wax. The only commercial installation in Germany was the Brabag plant in Zeitz. The lubricating oil produced there had a