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MISCELLANEOUS

JAPANESE FUELS AND LUBRICANTS - ARTICLE 8-NAVAL RESEARCH ON THE CANTS

U.S. NAVAL TECHNICAL MISSION TO JAPAN CARE OF FLEET POST OFFICE SAN FRANCISCO, CALIFORNIA

12 February 1946

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From:

Chief, Naval Technical Mission to Japan.

To:

Chief of Naval Operations.

Subject:

Target Report - Japanese Fuels and Lubricants, Article 8 -

Naval Research on Lubricants.

Reference: (a)"Intelligence Targets Japan" (DNI) of 4 Sept. 1945.

- Subject report, covering lubricant research by Japanese Navy outlined by Targets X-09, X-10, and X-38(N) of Fascicle X-1 of reference (a), is submitted herewith.
 - The investigation of the target and the target report were accomplished by Comdr. G. L. Neely, USNR, Lt. Comdr. C. S. Goddin, USNR, Lieut. W. H. Millet, USNR, with the assistance of Ens. E. R. Dalbey, as interpreter and translator.

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JAPANESE FUELS AND LUBRICANTS - ARTICLE 8 NAVAL RESEARCH ON LUBRICANTS

"INTELLIGENCE TARGETS JAPAN" (DNI) OF 4 SEPT. 1945
FASCICLE X-1, TARGETS X-09, X-10, AND X-38(N)

FEBRUARY 1946

U.S. NAVAL TECHNICAL MISSION TO JAPAN

SUMMARY

MISCELLANEOUS TARGETS

JAPANESE FUELS AND LUBRICANTS-ARTICLE 8
NAVAL RESEARCH ON LUBRICANTS

Japanese naval research pertaining to lubricating oils has been investigated. The scarcity of Japanese crude oils having characteristics suitable for the manufacture of aircraft engine lubricants necessitated the use of imported crude oils for this purpose. As this supply was depleted, it became necessary to consider non-pertoleum sources. The synthesis of lubricating oils from shale oil, paraffin wax, fatty oils, Fischer-Tropsoh liquid, rubber, and other sources was studied at the First Naval Fuel Depot and other research laboratories. In applying these lubricants to practice, the use of oridation inhibitors, oiliness agents, and other additives was frequently necessitated, in view of service performance limitations. Hence, additive studies were included as an essential part of the Navy's research program.

Perhaps the most important phase of lubricant research included in this report is the fundamental study of the relationship between structure and lubricant characteristics made by Dr. I. KAGKHIRA and co-workers. This research indicated that structures in which saturated cyclohaxane rings are joined in the "para"-position either by single bonds or by paraffinic chains, are the most suitable for lubricating oils. These theories were reflected in the synthetic lubricant research conducted at the First Naval Fuel Depot.

It is believed that this report presents a broad picture of the lubricant requirements of the Japanese Navy during the war and includes detailed information on many special products peculiar to naval use.

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REFERENCES

Location of Target:

First Naval Fuel Depot, OFUNA, Kanagawa Prefecture.
Third Naval Fuel Depot, TOKUYAMA.
First Naval Technical Depot, YOKOSUKA.
Army Fuel Research Institute, FUCHU.
Miike Synthetic Oil Company, CMUTA, Kyushu.
TEIKOKU NENRYO K. K. (Imperial Fuel Co.), Ube Plant, UBE.
NIHON HATSUDOKIYU K. K. (Japanese Motor Oil Co.), UBE.
Kyoto Imperial University, KYOTO.

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I. KAGEHIRA, Ph. D., Chemical Engineering Captain, Japanese Navy, Head of Section of Inspection and Planning, First Haval Fuel Depot, (a chemist of cutstanding ability specializing in synthetic lubricants). N. MATSUO, Naval Engineer, Head of Lubricants Section, First Naval Fuel Depot.

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T. FUJINOTO, Chemical Engineering Lieutenant, Japanese Navy, Head of Lubricant Testing Section, First Haval Fuel Depot.

Research Assistants of the First Naval Fuel Depot whose names are included in individual Japanese reports.

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I. MOTOYOSHI, Engineering Captain, Japanese Mavy, Chief Engineer of
Third Maval Fuel Depot, TOKUTAMA.

R. TAKKI, Chief Engineer of Mike Synthetic Oil Company, CMUTA.

Kyushu.
C. KITA, Professor of Chemistry (Retired), Kyoto Imperial University
(an outstanding research chemist specializing in catalysts for the
Pischer-Tropsch synthesis).
A. IBUKI, Professor or Bio-Chemistry, Kyoto Imperial University,
Kyoto.

S. MOMURA, Head of the Momura Office, TOKIO, (one of the leading
petroleum executives of Japan).

Referenced Japanese Reports:

"Technical Notes on Research Nork" prepared by the Army Fuel Research Institute, FUCHU. 2 September 1944.

INTRODUCTION

At no time during the war did Japan's supply of lubricating oil become as critical as did the supply of fuel of all types. In spite of this fact, however, a substantial portion of the hydrocarbon research activity of the Japanese Navy was devoted to the synthesis of lubricants from substitute sources.

This report summarizes the findings of the Petroleum Section of the U.S. Naval Technical Mission to Japan relative to lubricant research conducted by the Japanese Ravy at the First Naval Depot, OFUNA. It includes studies on petroleum refining, the synthesis of lubricants, and data relative to lubricating oil additives and greases. The information from OFUNA has been supplemented by that obtained from other laboratories, commercial plants, and naval installations.

Detailed reports of the lubricant research projects investigated at the First Maval Fuel Depot have been prepared in English by the technical personnel of the Depot and are included in this report as Enclosure (B)1 to (B)37, inclusive. A summary of these reports has been prepared in English by Chemical Engineering Captain I. KAUEHIRA and is submitted herewith as Enclosure (A).

Bince all of the research files of the First Haval Fuel Depot were burned in Angust, 1945 by order of the Director of the Depot, it was necessary to summon the Japanese personnel and reconstruct the information from laboratory notebooks, laboratory apparatus, and pilot-plant equipment. The preparation of these reports and drawings continued for a period of nearly three months, during which time each Japanese author was frequently interrogated and was assisted in the organization and translation of his report. This material, which is submitted as Enclosures (A) and (B), constitutes an integral part of this report and, although it may include minor errors in construction introduced in the translation by the Japanese authors, it does serve as an accurate indication of the quality and extent of the lubricant research conducted by the Japanese Havy.

THE REPORT

Part I REFINING OF LUBRICATING OILS

The principal Japanese crude oils are those obtained from the AKITA and NIIGATA districts. These crudes are of a highly naphthenic nature and, in general, are not suitable sources of aviation engine lubricants. Oils refined from Niizu crudes (NIIGATA) are characterized by exceptionally low pour points and negative viscosity indices, whereas most of the Akita oils have high wax contents and the viscosity-indices of the refined oils are less than 50. One exception is Omonogawa crude from AKITA which gives the best lubricating oil yield of any Japanese crude oil, a yield 2% or less (based on total crude) of 85-90 viscosity-index lubricating oil.

Oha orude produced in SAKHALIN was of a more paraffinic nature than the Japanese crudes discussed above, and from this crude a limited amount of aviation lubricating oil of from 90 to 95 V.I. was produced by solvent extraction with treating yields from 0.3 to 5% of total crude, depending upon the degree of treatment and type of treating process utilized (Knolosure (B)4).

The small amount of domestic crudes available, coupled with the low yield of refined products, rendered the production of aircraft cils from these sources quite inadequate, and, in order to meet this situation, Japan imported huge reserves of crude cil and finished products from the United States before the war. These crude cils constituted the charging stocks for the Japanese refineries producing the high viscosity-index cils needed as aircraft engine lubricants.

Two of Japan's largest oil refineries, the Second and Third Naval Fuel Depots, located at YOKKATCHI and TOKUYAMA, respectively, were owned and operated by the Japanese Navy. These two plants had a combined refining capacity of approximately 27,000 barrels of crude oil per day which represented one quarter of Japan's total refining capacity. All research activity pertaining to the processes utilized at these refineries was concentrated at the First Naval Fuel Depot, OFUNA. At first, the Ofuna research was directed towards solvent refining techniques for both Japanese and imported crude oils. However, as the stock of imported crudes diminished, the research emphasis was shifted to the synthesis of lubricants from various non-petroleum sources.

A comprehensive picture of solvent refining-studies at the First Maval Fuel Depot is presented in Enclosures (A) and (B)4 to (B)5, inclusive. The first process daveloped consisted of a combined deasphalting-dewaxing operation using propene, followed by Duo-sol solvent extraction. This so-called "Mavy" process was utilized at both naval refineries but considerable difficulty. With the propens dewaxing operation was encountered in practice. The process was later modified to incorporate the use of Barisol dewaxing after solvent treations.

There were three pilot plants at OFUMA used in connection with these studies. One of these pilot plants, built in TOMUYAMA in 19)7 and transferred to OFUMA in 19)9, was designed to study the combined propane desaphalting-dewaxing process. In conjunction with this unit there was a single-stage phenol-creacl extraction pilot plant at OFUMA. Each of these units had a capacity of 10 liters of charge per hour. In 1944 an acetone-bensol dewaxing pilot plant, having a capacity of 20 liters of charge per hour, was built.

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Another refining process which was studied was one in which amyl alcohol was used for the deasphalting-dewaxing operation and furfural was used for solvent extraction. This method was particularly attractive to the Japanese in view of amyl alcohol as a by-product of alcohol fermentation and the suitability of furfural for naphthenic oils. However, it was never put into commercial operation.

A report on the inspection of the Third Naval Fuel Depot at TOKUYAMA is submitted as Enclosure (F) of Article 10 of this series. This refinery had two propane deasphalting, Duo-sol extraction plants, each with a capacity of 160 kiloliters of charge per day. The capacity of the Barisol dewaxing unit was 30 kiloliters of product per day.

During the war the aircraft engine lubricants refined in this equipment were obtained 1 om topped Rhodessa, Osage, and Avalu crudes. The lubricating oil production in 1944 was 4120 kiloliters of aircraft engine oil, 4550 kiloliters of oylinder oil, and 1950 kiloliters of bearing oil.

While the lubricating oil treating data presented in detail as enclosures in this report disclose no new techniques, this material is included to show the nature of the Japanese stocks and to serve as a background for evaluating their overall lubricant position.

An interesting report entitled, "The Petroleum Industry in Japan" by I. ACKI and G. NARA has been written through the co-operation of S. NOMURA of the Nomura Office, TOKYO, for the Petroleum Section of the U. S. Naval Technical Mission to Japan. This report is submitted as Enclosure (D) of Article 10 of this series. The report discusses the history and recent developments in the Japanese petroleum industry. The characteristics of the various Japanese crude cils, Japanese crude oil production, and a discussion of the major refining equipment in Japan is included.

Part II SYNTHESIS OF LUBRICATING OILS

A. FUNDAMENTAL CONSIDERATIONS

All research activity of the First Naval Fuel Depot pertaining to synthetic lubricants fell under the jurisdiction of Dr. I. KACHHIRA; who held a specialist's commission as Captain in the Japanese Mayy. For the past sixteen years Dr. KACHHIRA has been studying the effect of molecular structure on the proporties of lubricating oils. He has been particularly interested in the synthesis of pure compounds of naphthenic or mixed naphthenic-paraffinic type prepared by the hydrogenation of aromatic compounds and has studied the relationship of the ring structure of these products to their viscosity, stability, and related properties.

Dr. KACKHIRA has concluded that the most desirable structure for lubricants of this type is that of a saturated compound in which cyclohexans rings are join-ed by single bonds in the para position. Applying his theories, several promising lubricants have been synthesized in the laboratory by completely hydrogenating such compounds as the condensation product of one sale of diphenylmethans with two moles of cetene or one mole of dibenxyl benzene with one mole of cetene. The lubricants thus prepared are characterized by high or the various studies which have been made are given in Englosures (A). (B)1 and (B)2. In this connection, it is of interest that the theories of this research are reflected in the overall synthetic lubricant progress of the Japanese Bavy.

B. SOURCES OF SYNTHETIC LUBRICANTS

As Japan's accumulated reserves of imported crude oil stocks diminished, it

became increasingly imperative that additional sources of lubricant supplies be developed. The Japanese Navy pioneered this development, and a discussion of the principal sources investigated is presented herewith.

- 1. Shale Oil. A large supply of the kerosane and gas oil fractions obtained from shale was available from the South Manchurian Railway plant at FUSHUN, Manchuria. The synthesis of lubricating oils by first refining these fractions and then polymerizing in the presence of aluminum chloride was studied at the First Naval Fuel Depot. Satisfactory airoraft lubricants were obtained in 10% yield but the process was applied only to the manufacture of aerial torpedo lubricants (Enclosures (B)) and (B)19).
 - 2. Paraffin Wax. Since 1942, high viscosity index aircraft lubricants, obtained by the polymerization of cracked paraffin wax from Sanga Sanga and Pendopo crude oils using aluminum chloride catalysts, had been commercially produced by the Fuel Depot at BALKPAPAN, South Bornec. The product was inferior-in-respect to oxidation stability and a study of modifications of the process was undertaken at the First Naval Fuel Depot in 1940. It was found that an improved product of greatly increased stability could be obtained by condensing the cracked wax distillate with aromatic compounds, sulfur, or combinations thereof. Stable aircraft oils were also prepared by polymerizing the cracked wax distillates with different topped crude oils. The utilization in this process of the wax obtained from Barison dewaxing plants was investigated but the results were not promising. Laboratory data and a discussion of the project are presented in Enclosure (B)10 and (B)11.

Commercially, this process for manufacturing aircraft lubricating cils was in operation at the Balikpapan Fuel Depot and at the Second Naval Fuel Depot, YOKKAICHI. The Nippon Oil Company also manufactured small quantities of a similar product at its Yokohama refinery. A paraffin wax, melting at 50°C, was cracked in a reconstructed Cross vapor-phase unit of 30 kiloliters per day capacity. The reaction conditions and the properties of the product are given below:

Reaction Conditions

Cracking Temperature	550-570 ⁰ C.
Polymerization Temperature	60 - 80°C.
Catalwat	AlCla(3-4%)
Dechlorination Catalyst Deahlorination Temperature	Clay, (4-5%)

Properties of Product

Reaction	Houtral
Wentrelization Humber	0.41
Flash Point	260°C.
Pour Point. Viscosity, SUB, at 21000	120
Viscosity This.	108
Viscosity Index	0.6%
Anh	0.012%
British Air Ministry Oxi	1 KA
Carbon Residue Rati	01.61
Neutralization Numb	

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3. Fatty Oils. Polymerization in the presence of aluminum chloride of the olefines obtained by the dry distillation of sodium scaps of coccanut oil and related fatty cils was investigated at the First Naval Fuel Depot. The method could only be applied to non-drying or hydrogenated cils since unsaturated fatty cils yielded low viscosity-index products, (Enclosure B-12, 13 and 14). A commercial unit applying this process was under construction at the Sixth Naval Fuel Depot, FORMCSA, but it was destroyed by bombardment prior to completion.

Under the jurisdiction of the Army, the Japanese Vegetable Oil Company, (Nippon Yushi K. K.) constructed a hydrogenation plant at AMACASAKI, for the synthesis of 7000 kiloliters per year of aircraft oils from soya bean oil. The hydrogenating units of this plant were destroyed by an explosion which occurred during the initial test run and these units never rebuilt. Dr. Y. NACAI, of Tokyo Imperial University, had studied this process for many years. Extensive pilot plant and laboratory tests had been conducted by the Army at the Army Fuel Research Institute, FUCHU, prior to the construction of the plant at AMACASAKI. The original process consisted of the polymerization of soya bean oil, hydrogracking of the polymerized oil in the presence of molybdenum trisulfide catalyst, hydrogenation of the resultant oil in the presence of nickel catalyst, and vacuum topping to yield a lubricating oil of the desired viscosity. The research at FUCHU was directed towards the simplication of the process and the following conditions were established as those which were to be utilized at the AMACASAKI plant:

Polymerization

(Soya bean oil thermally polymerized)

Manus amatuma		 330-340°C.
Tembergrone	• • • • • • • •	
Pressure		 ····750 ==
Time		 24 hours
Yield		
IIUIUA CAAAA		

Hydrogracking (Liquid phase hydrogracking using a flowing catalyst)

Temperature	320-360°C.
Pressure	150 atmospheres
Oil Velocity	1/4 Vol/hr/vol. of
	reaction tube.
Hydrogen Velocity	2000 vol/hr/vol. of
-	manation tube
Catalyst	Cu:M1:Acid Clay(1:1:1)
	Gu:Ni:Aoid Clay(1:1:1) 3-5% as metallio
	omide

Vacuum Distillation (In presence of 20% clay.)

No accurate data of the physical and chemical properties and service characteristics of this lubricant were available. A 16,435 kilometer non-stop test flight in MANCHURIA using the lubricant manufactured in the pilot plant has been reported.

Other processes utilizing fatty soids for the synthesis of lubricating oils were studied by commercial companies but none were applied to large scale production.

4. Fischer Liquid. A promising source of rew material for the synthesis of lubricating oils is the product of the Fischer-Tropach coal liquefaction

process. This liquid is of a highly paraffinic nature and should yield a stable and high viscosity-index lubricating oil. No research in regard to this problem was undertaken by the Navy, but other laboratories have made initial investigations of methods by which this liquid might be utilized for the production of lubricating oils.

The Milke Synthetic Oil Company, OMUTA, was the most productive Fischer-Tropsch plant in Japan, and in 1941 they were ordered by the Army to concentrate on the construction of a lubricating oil polymerization plant which was to utilize the product of a U.O.P. Dubbs catalytic crecking plant under construction at the time. When the war ended, neither of these units had been completed.

The process which was to be put into operation at OMUTA was first, to prepare a highly unsaturated gasoline by the vapor phase cracking of the heavier oil fractions from the Fischer-Tropsch synthesis. This cracked distillate, having an end point of 250°C. was then to be charged to a polymerizer with 3 anhydrous aluminum chloride and maintained with agitation at 60°C. for 8 hours, or 80°C. for 7 hours. The product was to be settled, dechlorinated, purified with active clay (3 hours at 150°C, 5% by weight of clay), filtered, and topped. A light lubricating oil and an aircraft lubricating oil were to be obtained from the product. Small scale pilot plant tests indicated that these oils would have the following chemical and physical properties:

	Aircraft Lubricating oil	Light Lubricating oil.
Specific Gravity, 15/4°C. Flash Point, °C. Pour Point, °C. Neutralization Number Viscosity (5.U.S.) at 100°F. at 210°F. Viscosity Index Carbon Residue, \$ Ash, \$	0.85-0.87 240 20 0.05 1550 125 107 0.08 0.003	0.82-0.83 145 25 0.06 104 34 54-

According to Dr. KITA of Kyushu Imperial University, Fischer liquid prepared by means of an iron catalyst should be easily adapted to the synthesis of lubricating oils, since this product is higher in unsaturates and can be more readily polymerized than the product obtained using cobait catalysts. More complete information pertaining to Dr. KITA's work on Fischer-Tropsch catalysts and the Mike Synthetic Oil Company is presented in Article 7 of this series, Enclosures (D) and (G), respectively.

- 5. Rubber. Rubber was one of the few types of hydrocarbons readily available to Japan during the first years of the war, hence it is only natural that it should have been considered as a source of fuels and lubricants. During 1943 and 1944 the First Haval Fuel Depot studied various methods by which aircraft engine lubricants might be obtained from rubber. The methods studied were:
 - a. Polymerization of the isoprene fraction of creeked rubber distil-
 - b. Copolymerisation of cracked rubber distillate with 2.54 volumes of the cracked distillate of paraffin wax.
 - e. Eydrocracking of a rubber-paraffinic hydrocarbon paste in a contimuous hydrocracking pilot plant,

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Both the copolymerization and hydrocracking methods yielded products of high viscosity index and satisfactory oxidation stability but neither process was applied to large scale units. Details of the research are described in Enclosure (B)15.

It is of interest that several companies prepared automotive lubricants from rubber on a commercial scale. Since the product could not be used as prepared because of poor oxidation stability, it was blended with mineral oil before use. Five plants utilized a process consisting of the following steps:

- a. Shredded rubber dissolved in kerosene or comparable solvent (210°C. for 30 hours).
 - b. Rubber polymerized using acid clay catalyst (222°C. for 8 hours).
 - c. Filtration.
 - d. Topping and distillation of light oil.
 - e. Blending.

The combined monthly capacity of these plants was 3550 kiloliter of product. The name, location and capacity of each factory is tabulated below:

Name of Plant	Location	Capacity (kl/mo)
Nippon Sekiyu K. K.	ARITA	830
Nippon Sekiyu K. K.	NIIGATA	500
Showa Sekiyu K. K.	NIIGATA	370
Toa Nenryo K. K.	SHIMIZU	1500
Telkoku Henryo Kogyo K. K.	UEE	350

6. Other Sources. Two other possible sources of synthetic lubricant were investigated at the first Raval Fuel Depot during the last few months of the war. These two sources were brown coal tar and pine root oil. It was planned to study the products obtained by polymerizing selected fractions of the dry distillation product of each of these materials in the presence of sluminum chleride. In the short time that was spent on these studies, no significant data had been obtained.

PART III LABORATORY STUDIES ON FUNDAMENTAL LUBRICANT PROPERTIES

(A. Oiliness

A considerable amount of research was carried on during the war at the First Baval Fuel Depot in regard to ciliness agents for lubricating cils, particularly for diesel engine lubricants and aircraft engine cils both petroleum and synthetic types.

A number of ciliness test machines were constructed for this purpose and it was concluded that Stanton's pendulum-type tester was the most suitable for routine work. Also a special machine, closely resembling the Almen Machine, was installed at the First Havel Fuel Depot in 1944, to carry out tests in regard to master rod bearing failures in Japanese aircraft engines. While no practical results were obtained due to poor design features, a description of this apparatus is included in this report in view of the use of magneto-

striction to measure torque instead of the hydraulic means used in the Almen Machine. (Enclosure (B)30.)

This oiliness research work indicates that the Japanese investigator recognized the value of such test variables as bearing metal combinations, rubbing speed and test temperatures on oiliness determinations, but the applicability of such variables was not considered in selecting oiliness agents for practical engine tests.

The most interesting ciliness research carried out at the First Naval Fuel Depot was a systematic study to find the relation between the chemical structure and the ciliness properties of lubricants based on static friction determinations in a modified Deeley Machine using steel on steel surfaces. It was observed that in the case of hydrocarbons, cyclic compounds were better than chain commounds with the same number of carbon atoms, while compounds in which six-membered rings are companed with single bonds were better than those in which the rings were combined in condensed form (Enclosure (B)27). Fundamental studies on the ciliness characteristics of stearic acid, benzene, and their derivitives indicated that the NH2 and COCH groups were the most effective, whereas other groups containing oxygen and nitrogen atoms were comparatively less effective. (Enclosure (B)28)

Another interesting oiliness study related to the use of soya been phosphatides. Research work based on kinetic friction determinations for steel on steel at low rubbing speeds and at room temperature indicated that the soya been phosphatides were effective us oiliness agents and that the higher the acid value the better the oiliness characteristics. (Enclosure (B)29)

B. Anti-Oxidents

1. First Naval Fuel Depot, OFUNA. Research work of the First Naval Fuel Depot in regard to anti-oxidants fell in two categories, namely anti-oxidants for lubricants from petroleum sources and anti-oxidants for lubricants made synthetically. In these laboratory studies the means for evaluating oxidation stability was the use of the British Air Ministry Oxidation Test, wherein increase in viscosity and Conradson Carbon after air-blowing in the absence of metal catalysts is determined. Some of these tests were paralleled by oxygen absorption tests conducted in the Warburg Bio-chemical Oxidation Apparatus described below. A fundamental weakness of the laboratory oxidation data obtained is the lack of adequate study of the formation of organic soids and supporting engine test results.

The lubricants prepared synthetically and discussed in detail in Part II of this report were usually deficient in oxidation stability and the inhibitors suitable for natural oils were not, as a rule, suitable for the synthetic oils. For example, for aircraft engine oil produced from petrolsum, trioresyl phosphite was the best anti-oxidant investigated, whereas for synthetic aircraft engine oil prepared from paraffin wax or Fischer-Tropsoh condensate, copper scaps were the most effective (Enclosure (B)23)

One of the most interesting laboratory findings was the improved exidation stability of siroraft engine, compressor, and turbine oils when utilizing soya bean phosphatides (Enclosure (B)29).

2. Kroto Imperial University, KIUTO, During the period 1944-45, Professor A. ISUAI of the Kyoto Imperial University conducted research relative to the oxidation of pure straight chain and cyclic compounds. In this work he used methyl cleate and tetralin as being typical, respectively, of unsaturated straight chain compounds and ring compounds.

Oxidation was studied by the use of the Warburg Bio-chemical Oxidation Apparatus in which the oxygen absorption of 0.5 grams of oil is indicated

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by change in pressure. The test is conducted at 50°C: for a period of 120 minutes and the millimoles of 02 absorbed are measured. Western Land Beleviter (Sales)

Name of Inhibitor	Absorption in cu.mm. of 02 by 0.5 gram methyl cleate with 1% (weight) of Inhibitor
b-naphthylamine	5.0
a-naphthylamine	11.0
a-naphthol	12.0
b-nephthol	17.5
Phenol	19.5
Aniline	41.0
No inhibitor	52.5
Cyclohexanol	03.0
	Tetralin
(Phenol	5.0
Aniline	13.5 18.8
No inhibitor	

These data are of a preliminary nature and no conclusions have been drawn by Dr. IBUKI.

- 3. Article 10 of this series, Enclosure (D), presents data relative to the oxidation characteristics of a blend of synthetic lubricant prepared from soya bean oil with 5% of naphthenic base oil and 0.1% of an organic salt (formula unknown). According to laboratory tests, this oil possessed superior oxidation stability characteristics when compared to the laboratory tests when compared to the laboratory tests when compared to the laboratory tests. typical direraft engine lubricants manufactured in America. No comparative engine data were available.
- 4. Pour Point Depressants. The synthesis of "Paraflow" was studied and a most effective product was obtained by condensing naphthalene and chlorimost effective product was obtained by condensing maphthalene and efforinated wax in the presence of aluminum chloride. A significant finding was
 that it was necessary to control the reaction by the addition of small
 quantities of water. The need for water was accidentally discovered and
 was not mentioned in the U.S. patent on "Paraflow". This pour point depressant differed somewhat in physical properties from "Paraflow", but it
 was effective in depressing the pour points of oils and was manufactured
 on a commercial scale throughout Japan. (Enclosure (B)35).

Part IV ENGINE LUBRICARIS

A. Aircraft Engine Oils

1. Mineral Cils. In Japan there was a sufficient supply of imported aircraft engine oil until about 1943, consisting principally of Texaco Cil Craft engine oil until about 1943, consisting principally of Texaco Cil Mo. 120. However, it then became necessary for the Japanese Mayy to use oil designated as "K-120-K" which was prepared in Japan from Phillips Osage Cil designated in the Poo-Gol plant at Tokhrama. Full-scale aircraft engine tests (conducted at the First Technical Depot, Toxosuka) indicated that "K-120-K" was not equal in service performance to Texaco Cil Mo. 120 that "K-20-K" was not equal in service performance to Texaco Cil Mo. 120 and, eccordingly, full-scale tests were made at the Mitsubishi, Makajima and Aichi Companies. The cil was compounded with 25 by weight of both trioresyl phosphate and trioresyl phosphate (this compounding was abosen in view of isboretory research oxidation and stability tests discussed in Minelosure (8)23) Results of the engine tests were not conclusive, but inminerate (8)23) Results of the engine tests were not conclusive, but inminerated sufficient improvement on master rod bearing and piston ring condition to justify its practical use in combat planes faring July and engine 1945. There is no information in regard to the practical results

obtained with compounded "K-120-K" oil used during the last two months of the war. (Enclosures (B)25 and (B)26)

As discussed in Part III, Section B, laboratory data are presented in Enclosure (B)29 relative to the improved oxidation stability of aircraft engine oils by the use of soya been phosphatides as additive agents. A small amount of laboratory data are also presented on the simultaneous use of a phosphatide and trioresyl phosphate in aircraft engine oils of both the natural and synthetic types. No engine test data relative to the use of these compounds are available.

- 2. Synthetic Oils. For synthetic aero engine oils prepared from paraffin war or Fischer-Tropsch Oil, as discussed in Part II, B, of this report, it was found that the anti-oxidants most suitable for petroleum oils were not suitable, but that dopper soaps were markedly effective. Again there is no supporting engine data for the synthetic oil so compounded. (Enclosure (B)23.)
- Wiscosity Index Improvers. Japanese naval aircraft engine oil "K-120-K" was produced from Mid-Continent crude by solvent extraction at the Third Naval Fuel Depot to meet the minimum viscosity index requirement of 90. In order to improve the viscosity index, iso-butylene polymers were prepared following the method of the Standard Oil Company of New Jersey and were blended with aircraft engine oil No. 80 purchased from the Texas Oil Company. This raised the viscosity from 80 S.U.S. to 121 S.U.S. at 2100 and the viscosity index from 94 to 101. In single cylinder aircraft engine tests it was indicated that wear with the blended oil was somewhat greater than that obtained with natural petroleum oils, and it was concluded that it was necessary to improve the heat stability of the polymer engine oil blend before it could be satisfactorily used in aircraft engines. (Enclosure (B)33.)
- 4. Reclaining. In view of the increasing shortage of aircraft engine lubricants in the latter part of the war and the difficulty of transporting used lubricants from the airfields to the refineries for re-refining, a portable unit, which could be fabricated at the airfields from materials on hend, was developed for reclaining used lubricating oil. The unit consisted essentially of a tank with a perforated plate in the bottom covered with a sheet of paper. The oil was placed in this tank and the cashes of wood, grass, or roots were added, using 15% of askes by weight. The oil was heated to 130°C and drawn through the paper into smother tank using a vacuum system. Analyses of the used and reclaimed oil in one case showed reduction in the ash content from 2 to .01% and in acid rate of from 7 to 20 litters per hour by this apparatus.

showed reduction in the sah content from .2 to .01% and in soid value from .26 to .08. Reclaimed oil could be produced at the rate of from 7 to 20 liters per hour by this apparatus.

B. Marine and Aero-Tropedo Engine Lubricants

During the period of 1938 to 1943 research was carried out at the First Haval Puel Depot to develop lubricants particularly suited for marine and derotorpedo angines. Significent results included the following:

- l. Marine Torpedo Engines. For marine torpedo engines the lubrication requirements were judged to be similar to those of aircraft engines, and aircraft engines oil prepared from the crude by solvent extraction (Part I, this report), and having a viscosity-index of 99 and a pour point of -1100, gave excellent results.
- 2. Aero-Torpedo Engines. Aero-torpedo engines operate at a much lower temperature than those of marine torpedos and consequently, require special

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lubricants. A synthetic oil prepared from crude shale oil by the polymerization method (Part II, Section B-1) was found to be satisfactory in practical service tests. This product had a viscosity-index of 92 and a pour point of -32.

For still lower temperature conditions of operation, a synthetic cil prepared by the polymerization of a fraction of cracked war distillate (Part II, Section B-2 of this report), compounded with 11% aluminum cleate, gave satisfactory results. Its viscosity-index was 112 and its pour point, -46°C. The aluminum cleate was added to prevent the cil from being washed out by sea water in the aero-torpedo engine at the end of its run on trial shots. This product was tested and approved by the Naval Aeronautical Arsenal, YOKOSUKA, in 1943 for use under all conditions of temperature.

C. TURBINE OILS

Turbine oils were, in general, prepared from Japanese crudes. These turbine oils had high pour points unless dewaxed and the capacity of the dewaxing plants in Japan was insufficient for treating turbine oils, since the plants were chiefly used for the production of aircraft engine lubricants. For this reason, pour point depressants were manufactured as discussed in Part III, C, of this report, and added to turbine oils, depressing their pour points from approximately 15°C to below 0°C. This method was applied on a commercial scale.

No other additives such as oxidation inhibitors or rust-proof compounds were used with turbine oils and no information in regard to service performance characteristics of turbine oil was obtained.

D. DIESEL ENGINE OILS

- 1. Straight Mineral Oils. Prior to the war, turbine oils had been mainly used for marine diesel engine lubrication in the Japanese Navy, but this type of oil, except that prepared from Oha crude, was not found satisfactory from the viewpoint of cylinder wear. When aircraft engine oil No. 60 (produced by the Texas Oil Company) was used as a diesel engine lubricant, cylinder wear was decreased to about one-fifth that obtained with the turbine oil, but the carbon and lacquer deposits on the pistons were excessive. Lubricating oil prepared from Oha crude by vacuum distillation, phenol extraction, dewaxing and soid clay treating gave good results in engine tests and did not require the use of an additive, but a treating yield of only 0.3% was obtained. (Enclosure (B)20.)
- 2. Compounded Oils (Laboratory Tests). In 1942 and 1943 a series of laboratory tests were conducted to determine additives suitable for use in submarine diesel engine lubricating oils. These compounds were studied from the standpoint of ciliness, oil stability, and dispersion or peptizing of carbon deposits. It was found that calcium phenyl stearate was the most effective from the standpoint of detergency and dispersion of carbon deposits and that lacithin (soya bean phosphatide) and trioresyl phosphate were the most effective as ciliness agents and anti-ordents. The detergency tests were made by washing a piece of flannel stained with soot, with gasoline containing 2% of each additive, whereas the laboratory dispersion test consisted in massuring by means of a photometer, the translucency of white oil to which had been added oil sludge and one of the several additives being investigated.

As a result of this laboratory work, it was concluded that calcium phenyl stearate was suitable as an additive for high viscosity aero-type engine cils intended for use as diesel engine lubricants, whereas lecithin and tricresyl phosphate were beneficial as additives for low viscosity-index cils when used in diesel engines. (Enclosure (B)29.)

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3. Compounded Oils (Engine Tests). No actual engine data were obtained on diesel engine lubricating oils either straight or compounded. It was reported, however, that practical tests in diesel engines of turbine oils

compounded with lecithin (the active constituent of soya bean phosphatide) showed this additive was not sufficiently effective to render this oil stock satisfactory as a diesel engine lubricant. It was further reported that diesel engine tests showed that the use of calcium phenyl stearate did not sufficiently reduce the carbon deposition characteristics of aircraft engine oil No. 80.

Part V SPECIAL OILS

A. "PRECISE" OILS

The term "precise oil" is applied by the Japanese Navy to such types of lubricants as instrument oils and hydraulic oils. Specifications for five different grades of these oils were established by the Japanese Navy in 1943. All but one of them consisted of highly refined and selected fractions of Niizu crude oil obtained from the NIIGATA district in northwestern HONSHU.

The refined product was highly naphthenic and, although it had an exceptionally low pour point, difficulties were encountered in practice which were attributed to the low viscosity index (-100) and low aniline point (60-65°C.) of these cils. It was reported that, in service, rubber softening and swelling of packings was frequently encountered. Several explosions had occurred because of the bursting of rubber hose and all rubber fittings had to be renewed frequently. During the war the First Naval Fuel Depot was actively engaged in developing synthetic lubricants which would surmount the difficulties encountered with mineral cil. A comprehensive picture of this development is presented in Enclosures (A), (B)21, and (B)22.

Four precise oils synthesized from non-conventional sources were developed by the Kayy at the First Naval Fuel Depot. The method by which each was prepared may be briefly stated as follows:

1. Number 5 Precise Oil

- a. 80% of a fraction of polymerized dodecene boiling from 180°C . to 300°C . at a pressure of 5 mm of mercury.
- b. 19.8% of a selected fraction of refined Mizu crude oil.
- o. 0.2% of rapeseed oil.

2. Special Precise Oil

- a. 33.5% of the polymerized product of thermally cracked paraffin war distillate.
- b. 66.5% of a selected fraction of refined Milzu crude oil.
- 3. Special Match Oil. The fraction boiling from 120°C, to 300°C, of the polymerisation product of a mixture of 20 parts of tolucl with 100 parts of a thermally cracked wax distillate.
 - 4. Precise Oil from Shark Liver Oil. Squalene, obtained from shark liver oil distillate, has hydrogenated in the presence of a mickel catalyst.

Details of the methods of preparation and the properties of the products are given in Enclosure (B)21. The Number 5 precise oil has been used since 194)

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as an instrument oil for serial torpedoes and the special watch oil was utilized in time bomb mechanisms. The other two oils did not progress beyond the experimental stage, but were to be utilized as hydraulic oils.

B. HYDRAULIC OILS

The Japanese Navy used two hydraulic oils in addition to the "precise oils" described above. One was a highly refined fraction of Niizu crude oil, and the other was a butanol-eastor oil mixture. No research on either of these oils was conducted at the First Naval Fuel Depot during the war.

C. ESTERIFIED OILS

Although the Japanese Navy conducted no research on this type of lubricant, it is of interest that the Japanese Army did use esterified oils as automotive lubricants. Enclosure (D) outlines the information obtained from the Japanese Motor Oil Company (Nihon Hatsudokiyu K. K.) pertaining to the manufacture of these cils. Of particular interest is the low temperature lubricating oil prepared by blending 87 volumes of refined butyl ricincleate with 13 volumes of polymerized soys bean oil. The lubricant was manufactured for the use of the Army in Manchurian operations, but production was stopped early in 1944 since there was no appreciable military activity in that area.

D. ANTI-CORROSIVE CYLINDER OIL

A cylinder oil for aircraft engines which served as a rust preventive was developed at the First Naval Fuel Depot in 1943. Details of the manufacture of this product are given in Enclosure (B)39. The composition (wt. \$) of the oil is as follows:

Refined rapeseed oil .	75
Aluminum stearate	10-14
Triethanol amine	10-8

E. ELECTRIC CABLE INSULATING OILS

In 1944 the preparation of polyisobutylene to be used as an insulating oil was studied at OFUNA. The reaction was carried out at a temperature of 150°C. and a polymer having an average molecular weight of 170,000 was obtained. The process was not applied commercially. The method of preparation follows that described in U. S. Patents and is outlined in Enclosure [B]34.

Part VI

Research work conducted at the First Naval Fuel Depot on lubricating greases, primarily for aircraft use, was undertaken in 1938 using natural mineral oil stocks. In 1943, due to the increasing shortage of oil stocks, the research on greases from petroleum sources was paralleled with studies of synthetic lubricating oils as substitutes. Generally speaking, natural lubricants from Japanese crude oil consisted of arcmatic hydrocarbons which have good scap solubility, characteristics compared to paraffinic hydrocarbons. Synthetic paraffinic-type oils were less desirable due to solubility characteristics, particularly the high viscosity grades. Then using synthetic lubricating oils, it was found that for calcium or soda scap greases, condensation products of paraffinic and arcmatic hydrocarbons were suitable.

Prior to 1938, special greases for aviation engines and auxiliary parts were imported chiefly from the United States and up until that time, practically no research had been carried out in Japan along these lines.

The procedure adopted in 1938 was first, to install modern greese manufacturing equipment, including the latest electrically heated experimental greese

kettles and milling machines of the three-roll type. The second step was to manufacture greases whose characteristics matched those of imported greases found to be satisfactory in service. After matching these greases in small equipment, experimental products were manufactured at the First Naval Fuel Depot in equipment of pilot plant size.

- 1. Magneto Grease. A grease for aircraft engine magnetoes was developed whose service performance matched that of Bosch Magneto Grease manufactured in the United States. The Japanese grease was composed of a soda soap of castor oil with an excess of soda and was prepared at a temperature of 230°C. Quick cooling was used and was followed by milling to provide a smooth texture. For manufacturing magneto grease from synthetic oils and castor oil soap, it was found necessary to specify the viscosity and maximum aniline point of the synthetic oils in view of solubility considerations.
 - 2. Rocker Arm Grease. Two experimental greases having satisfactory service performance characteristics were developed. Both of these greases contained a mixture of sodium cleate and sodium stearate scaps and a small amount of glycerine. There is no information in regard to the manufacture of this lubricant using synthetic cits.
- 3. Controllable Pitch Propeller Grease. A satisfactory product was made by using a mixture of aluminum stearate, aluminum cleate, and lead cleate scaps, plus a small amount of glycerine, compounded with approximately 90% of viscous lubricating oil stock. Friction tests in a machine resembling the Timken Lubricant Test Machine showed that the presence of lead cleate imparted friction reducing properties.
- 4. Liquid Grease for Framework of Aero-Torpedoes. A grease containing 7% aluminum stearate and 0.2% calcium stearate and 92.0% of cylinder stock gave excellent results for the prevention of corrosion of engine parts. The role of the calcium stearate in this product was to modify the plasticity of the aluminum stearate.
- 5. Special Grease for Preventing Corrosion of the Interior of Compressed Air Cylinders of Aero-Torpedes. Up until 1943, heavy cylinder oil was found unsatisfactory in adhesiveness and in anti-freezing characteristics. A product overcoming these deficiencies was prepared by heating 12% aluminum stearate and 0.36% calcium stearate with cylinder stock, followed by milling.
- 6. See Water-Proof Grease. This product was prepared as an anti-corresive, anti-wash compound to be used on machine guns mounted on sub-marines. A satisfactory composition consisted of 13% aluminum stearate and 87% heavy lubricating oil.
- 7. Special Greases. A minor amount of work was done on special greases such as alcohol-proof greases and greases that were resistant to concentrated nitric acid. (Enclosure (B)36, Part III.)

Part VII LUBRICANT SPECIFICATIONS JAPANESE HAVY

Official specifications of lubricants used by the Japanese Mavy during the war period are included as an Ecolosure to Article 1 of this series.