

RESTRICTION STATEMENT

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MISCELLANEOUS TARGETS

JAPANESE FUELS AND LUBRICANTS - ARTICLE 2

NAVAL RESEARCH ON AVIATION GASOLINE

U.S. NAVAL TECHNICAL MISSION TO JAPAN

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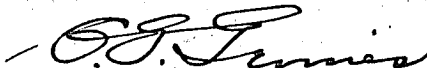
To : Chief of Naval Operations.

Subject: Target Report - Japanese Fuels and Lubricants, Article
2 - Naval Research on Aviation Gasoline.

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

1. Subject report, dealing with aviation gasoline research by the Japanese Navy outlined by Targets X-09, X-10 and X-38(N) of Fascicle X-1 of reference (a), is submitted herewith.

2. The investigation of the target and the target report were accomplished by Comdr. G.L. Neely, USNR, assisted by Lt. Comdr. C.S. Goddin, USNR, Lieut. W.H. Millett, USNR, and Ens. E.R. Dalbey, USNR, interpreter and translator.



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Captain, USN

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JAPANESE FUELS AND LUBRICANTS - ARTICLE 2
NAVAL RESEARCH ON AVIATION GASOLINE

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

FEBRUARY 1946

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MISCELLANEOUS TARGETS

JAPANESE FUELS AND LUBRICANTS - ARTICLE 2
NAVAL RESEARCH ON AVIATION GASOLINE

The Japanese fleet air arm utilized a combat aviation gasoline of 91 or 92 CFR-M octane number except during the last months of the war when concessions in quality became necessary to maintain supply. Aviation fuel of 100 octane number was made only in experimental quantities.

Considerable research attention was concentrated by the First Naval Fuel Depot, OFUNA, on developing a wide variety of processes, including alkylation, isomerization polymerization, hydrogenation, catalytic reforming and catalytic cracking, for manufacturing iso-octane and other high octane blending stocks. Commercial scale installations were made of all these processes, with the exception of isomerization and catalytic reforming. Particular ingenuity was demonstrated in the development of a large commercial plant for synthesis of iso-octane from acetylene derived from calcium carbide. The research and development work of the Japanese Navy played a leading part in the growth of the growth of the aviation industry in Japan.

Complete facilities were installed at the First Naval Fuel Depot, OFUNA, for the service testing of aviation fuels and lubricants, including full-scale aircraft engine test stands and an elaborate high-altitude low-temperature cold room. Numerous special fuel blends, dopes and anti-detonants were tested, and fundamental combustion studies were made.

Although in the last year of the war frantic efforts were made to utilize various substitute aviation fuel sources, especially alcohol and pine root oil, none of these materials, and no dopes or anti-detonants other than tetra-ethyl fluid were actually used in combat aviation fuels; however, ethyl alcohol was used for training purposes.

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REFERENCES

Location of Target:

First Naval Fuel Depot, OFUNA, Kanagawa Prefecture.
 First Naval Technical Depot, YOKOSUKA.
 Third Naval Fuel Depot, TOKUYAMA.
 Army Fuel Research Institute, FUCHU.
 Kyushu Imperial University, FUKUOKA, Kyushu.

Japanese Personnel Interviewed:

- K. NAKATA, Engineering Commander, IJN, Head of Aviation Gasoline Testing Section of the First Naval Fuel Depot, Specialist in Combustion Research.
 - H. FUJIMOTO, Ph.D., Engineering Commander, IJN, Head of Cracking and Dry Distillation Sections of the First Naval Fuel Depot.
 - Y. YAMAMOTO, Engineering Lieutenant-Commander, IJN, Head of Hydrogenation and Gasoline Blending Section of the First Naval Fuel Depot.
 - H. HOSHIMIYA, Engineering Lieutenant-Commander, IJN, Research Chemist of the First Naval Fuel Depot, specializing in aviation fuels.
 - K. SONE, Engineering Lieutenant, IJN, Research Chemist of the First Naval Fuel Depot, specializing in hydrocracking.
 - T. KONDO, Engineering Captain, IJN, Head of Aircraft Engine Test Section of the First Naval Technical Depot.
 - I. WATANABE, Engineering Rear-Admiral, IJN, Superintendent of the Third Naval Fuel Depot.
 - I. MOTOYOSHI, Engineering Captain, IJN, Chief Engineer of the Third Naval Fuel Depot.
 - KIROSHIMA, Ph.D., Professor of Chemistry, Kyushu Imperial University, Specialist in the Chemistry of Rubber.
 - N. NAKAHARA, President of Toa Nenryo K.K. (East Asia Fuel Co.), closely associated with the development of catalytic cracking in Japan.
 - S. NOMURA, The Nomura Office, TOKYO, one of the foremost petroleum executives in Japan.
- Research Assistants of the First Naval Fuel Depot, whose names are included in individual Japanese reports submitted as Enclosure (B).

Referenced Japanese Reports:

- "Technical Notes on Research Work", Army Fuel Research Institute, FUCHU.
- "Development of Catalytic Cracking in Japan" by N. NAKAHARA. Submitted as Enclosure C of NavTechJap Report, "Japanese Fuels and Lubricants, Article 10 - Miscellaneous Oil Technology and Refining Installations", Index No. X-38(N)-10.

INTRODUCTION

One of the most pressing problems confronting the Japanese throughout the war was that of maintaining an adequate supply of aviation gasoline in the face of insufficient resources and equipment.

This report summarizes the findings of the Petroleum Section of the U. S. Naval Technical Mission to Japan relative to aviation gasoline research conducted by the Japanese Navy at the First Naval Fuel Depot, OFUNA, supplemented by pertinent information obtained from other laboratories, commercial plants and naval installations. There is included in the report a discussion of the methods used in manufacturing aviation gasoline base stocks from non-petroleum sources, the development of aviation gasoline blends and dopes, and the service testing of products in aircraft engines. Changes in aviation gasoline specifications necessitated by the decreasing supply of raw material are described. There is also included a discussion of the fundamental engine combustion research conducted by the Japanese Navy.

Detailed reports of the aviation gasoline research projects investigated at the First Naval Fuel Depot have been prepared in English by the technical personnel of the Depot and are included in this report as Enclosures (B)1 to (B)38, inclusive. A summary of thirty-four of these reports has been prepared in English by Chemical Engineering Lt. Commander H. HOSHIMIYA of the Japanese Navy and is submitted herewith as Enclosure (A).

Since all of the research files of the First Naval Fuel Depot were burned in August 1945, by order of the Superintendent of the Depot, it was necessary to recall the Japanese personnel to reassemble this information from laboratory notebooks and available records. The preparation of reports and drawings continued for a period of nearly three months, during which time each Japanese author was frequently interrogated and assisted in the organization and translation of his reports by the Petroleum Section of the U. S. Naval Technical Mission to Japan. The 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 authors, it does serve as an accurate indication of the quality and extent of the aviation gasoline research conducted by the Japanese Navy.

THE REPORT

Part I MANUFACTURING METHODS

A. MANUFACTURE OF AVIATION BASE STOCKS FROM PETROLEUM SOURCES

1. Distillation of Crudes

The standard military combat aviation gasolines up to 1944 were 87-92 CFR-M octane fuels containing a maximum of 0.1 volume per cent of tetra-ethyl lead (see Table 4 for a list of Navy combat aviation gasoline specifications). These fuels were made from light naphtha cuts from selected crudes, or by blends of straight run naphthas with iso-octane and hydrocracked naphthas.

Prior to the war substantial quantities of aviation gasoline, aviation blending stocks and selected crudes containing aviation grade naphtha were imported from foreign sources, especially the United States. It has been estimated that some 7,000,000 barrels of aviation gasoline and the equivalent of 14,000,000 barrels of aviation naphtha contained in crude oils, were imported during the period 1937-1941. Kettleman Hills was the crude imported in greatest volume. After Pearl Harbor, aviation base crudes were obtained from Borneo and North and South Sumatra, including such fields as SERIA, SANGA SANGA, MLIRI, ABABU, TALANG AKAR, and DJAMBI.

Japan's domestic production of crudes containing high octane aviation naphtha was very limited. One such crude is known to be Omonogawa, from the AKITA field, which contains some 17% of naphtha of 76 octane number (92 with 0.1% T.E.L.).

As the supplies of high quality crudes dwindles in the latter stages of the war, Japan was forced to shift emphasis from the preparation of aviation gasoline by simple distillation of crudes, to more complicated processes for converting gases, gas oils, and low octane naphthas into suitable high octane blending stocks.

2. Catalytic Cracking and Reforming

Although catalytic cracking studies were initiated by the Tokyo Imperial University in 1934, not much attention was given to the process until after announcement of the Houdry Process in America. Attempts to purchase licensing rights to the Houdry and Universal Oil Products Co. catalytic cracking processes in 1939 failed due to the Moral Embargo, and strenuous efforts were made thereafter to develop a satisfactory process in Japan.

A catalytic cracking pilot plant of 25 barrels per-day charge capacity was completed by the Navy at OFUNA in September, 1942. This unit was based, at least in part, on the Universal Oil Products Company's design, and utilized a Japanese activated acid clay catalyst which was found to be superior to the U.O.P. synthetic catalyst as manufactured by the Nippon Oil Company. Adiabatic reactors were employed and the catalyst was regenerated by circulating flue gas with oxygen content controlled to 2-6%. Cracking tests on East Indies crudes were continued until January 1945, when tests were started on pine root oil fractions (NavTechJap Report, "Japanese Fuels and Lubricants, Article 4 - Pine Root Oil Program," Index No. X-38(N)-4; Enclosure (B)5). On the basis of pilot plant data, the Navy started construction of four 2,000 barrel per day catalytic cracking units, two at YOKKAICHI and two in FORMOSA. Of the Units at YOKKAICHI, one unit was completed and produced 10,000 kiloliters of aviation gasoline in 1944; the second unit, although completed in 1945, was never operated,

due to lack of gas oil charging stock. The two units at FORMOSA were not completed by the end of the war. The Japanese Army constructed a 3,000 barrel per day unit and also unsuccessfully attempted modification of a thermal cracking unit at its MARIFU refinery. The Toa Nenryo Oil Company also began, but did not complete, construction of a 3,000 barrel per day unit at ~~WAIYAMA~~. In addition to these larger units, the Nippon Oil Company and Showa Oil Company were reported to have converted a total of three thermal cracking units to catalytic cracking. These units were small, about 150 barrels per day charge capacity, and it was reported that operations were not highly successful.

In connection with the pine root oil program, it was planned, in 1945, to erect some 21 small catalytic cracking units each having a charging capacity of 10kl to 30kl per day of pine root oil fraction, yielding about 35 per cent of 90-92 octane, (0.15 vol. % T.E.L.) aviation gasoline. None of these units were completed, however.

No evidence of research studies or industrial application of the fluid catalyst cracking process was encountered.

Some catalytic reforming investigations were made by the Toa Oil Company, but no industrial applications were reported. The Japanese Navy at CFUNA made extensive studies in 1945 of small simplified catalytic reforming units to process pine root gasoline of 185°C. end point, yielding 50-60% of 92-94 octane (0.15% of T.E.L.) aviation gasoline (NavTechJap Report, Index No. X-38(N)-4, Enclosures (B)5, (B)6). Japanese acid clay was used as the catalyst in these units.

In summary, the Japanese catalytic cracking and reforming processes were late in their commercial application, and no significant amount of aviation gasoline was produced by these methods.

3. Hydrocracking

The Japanese Navy initiated autoclave tests in 1935 on the high pressure hydrocracking of mineral oils, in conjunction with the coal liquefaction program. In 1940, continuous pilot plant studies were undertaken with emphasis on producing 92 octane (0.1 vol. % T.E.L.) aviation gasoline from high and low temperature tar and from East Indian crudes. In 1945, the hydrocracking of substitute oils from pine roots, soya beans and rubber were investigated. Typical pilot plant results are given in Table I.

Details of the above experiments are given in Enclosure (B)16 (Parts 1-6), (B)18, and (B)19 of this report, also NavTechJap Report, Index No. X-38(N)-4, Enclosures (B)7, (B)8.

A number of commercial scale hydrocracking plants were installed in the Japanese Empire. A tentative list of such plants, based mainly on information secured from the Teikoku Nenryo Company in TOKYO, is given in Table II. All of these plants were based primarily on the Navy process using NiO-MoO₃ catalyst and operating at 200 atmospheres and 400-450°C., with a space velocity of 0.5-1.0.

A related process successfully operated for production of aviation gasolines was the hydrogenation of cracked gasoline, accomplished at 100 atmospheres, and 330-350°C., using a NiO-acid clay (50:50), or a NiO-MoO₃-MgO (14:43:43) catalyst. A plant of this type was first installed at TOKUYAMA about 1937 (NavTechJap Report, Index No. X-38(N)-10, Enclosure F).

Although firm production figures were not obtained, it is fairly evident that hydrocracking of oil and hydrogenation of gasoline were the most successful processes in Japan for producing high octane aviation stocks (exclusive of simple distillation of crudes), insofar as actual volume of

production during the war was concerned. Catalytic cracking was considered to be a more economical process, but was late in getting started.

Table I
TYPICAL PILOT PLANT HYDROCRACKING TEST RESULTS

Stock	Catalyst	Reaction Temperature (°C.)	Reaction Pressure (Atms.)	Yield of AvGas (%)	CFR-M Octane With 0.1% T.E.L.
High Temperature Tar	MoS ₃	470	200	25	92
Low Temperature Tar	MoS ₃	430	200	50	89
Sumatra Kerosene	NiO-MoO ₃	420	200	70	87
Omonogawa Gas Oil	NiO-MoO ₃	430	200	73	89
Oha Gas Oil	NiO-MoO ₃	410	200	70	92
Soya Bean Oil	MoS ₃	450	200	40	85*
Pine Root Oil	NiO-MoO ₃	430	200	50	95*
Oil From Rubber	NiO-MoO ₃	460	200	42	92*

*Includes 0.15 vol. % of T.E.L.

Table II
OIL HYDROGENATION AND HYDROCRACKING PLANTS IN JAPAN

Operated By	Location	No. of Units	Process	AvGas Capacity (kl/year)
JAPANESE NAVY				
Second Naval Fuel Depot	YOKKAICHI, Honshu	2	Kerosene hydrocracking	2x20,000
		2	Cracked gasoline hydrogenation	2x20,000
Third Naval Fuel Depot	TOKUYAMA, Honshu	1	Kerosene hydrocracking	10,000
		1	Cracked gasoline hydrogenation	10,000
JAPANESE ARMY				
Iwakuni Refinery	IWAKUNI, Honshu	1	Kerosene hydrocracking	20,000
Shiheigai Refinery	SHIHEIGAI (Ssu-p'ing-kai) Manchuria	1	Kerosene or gas oil hydrocracking	unfinished
CIVILIAN				
Toa Henryo Co. Wakayama Works	WAKAYAMA, Honshu	1	Cracked gasoline hydrogenation	-
Toho Kagaku Kogyo Co.	NAGOYA, Honshu	1	Kerosene or gas oil hydrocracking	10,000
Teikoku Henryo Kogyo Ube Works	UBE, Honshu	1	Low temperature tar hydrocracking	10,000
Nippon Yuka Kogyo Co. Kawasaki Works	KAWASAKI, Honshu	1	Low temperature tar hydrocracking	10,000
Chosen Jinzo Sekiyu Co. Agochi Works	AGOCHE, Korea	1	Low temperature tar or kerosene hydrocracking	-
Manshu Jinzo Sekiyu Co. Fushun Works	FUSHUN, Manchuria	1	Low temperature tar or kerosene hydrocracking	5,000

4. Isomerization

The Japanese Navy conducted research experiments at OFUNA on the production of iso-paraffins, starting about 1937. At this time a program was initiated for the preparation of iso-butane from n-butane, for use in ~~alkylation (Enclosure (B)6)~~. A pilot plant of 10 liters per hour butane charge capacity was operated at OFUNA from 1942-1944. This unit was based at least in part, on observations of a Dutch plant in PALEMBANG, and utilized $AlCl_3$ catalyst supported on acid clay with operating conditions of $110^\circ C$. and 15-20 atmospheres. A research study also was made of the production of iso-butane by cracking Fischer-Tropsch condensate oil in the presence of $AlCl_3$ (Enclosure (B)3).

The synthesis of high octane isomeric hexanes from pinacone and pinacolone, and by isomerization of n-hexane were studied (Enclosures (B)4 and (B)5). In this connection the manufacture of acetone from acetylene was investigated as a source of pinacone and pinacolone (Enclosure (B)12). Synthesis of iso-dodecane as a high octane safety fuel was also studied (Enclosure (B)4, Part 2).

No evidence of the industrial application of isomerization or synthesis of any high octane isomeric compounds, other than iso-octane, was obtained.

B. MANUFACTURE OF ISO-OCTANE

1. Synthesis From C_4 Hydrocarbons

The synthesis of iso-octane in Japan was accomplished by two processes, alkylation with iso-butane, and polymerization of butenes plus hydrogenation of the dimer. Research on polymerization of butylenes was carried on at OFUNA during the period 1937-1945 (Enclosures (B)2 and (B)10). As the result of this work a catalyst composed of $BaSO_4$, acid clay and H_3PO_4 in the ratio of 80:5:15 was developed and used at TOKUYAMA and other plant installations in JAPAN for polymerization of butylenes at $150-170^\circ C$. and 40 atmospheres (NavTechJap Report, Index No. X-38(N)-10, Enclosure F). It was claimed that this type of catalyst had better activity and selectivity than standard U.O.P. catalyst.

Some studies were also made on catalysts and optimum conditions for hydrogenation of the butylene polymer (Enclosure (B)10, pages 41-50). The hydrogenation step, as performed in the TOKUYAMA plant, utilized a nickel oxide catalyst at $200^\circ C$. and five atmospheres pressure. Research studies on the alkylation of iso-butylene with propylene and n-butylene in the presence of concentrated H_2SO_4 were made at OFUNA during the period 1939-1942 (Enclosure (B)8). A pilot plant based on this process was also constructed.

A shortage of C_4 hydrocarbons caused by the small crude throughputs and limited cracking capacity of Japanese refineries, prevented wide scale application of iso-octane synthesis plants. Alternative sources were sought and successful processes were developed for the synthesis of butylene from butanol and acetylene. Available data indicate that only one alkylation plant was built in JAPAN, -- a 2,000kl per year unit installed at YOKKAICHI in 1942. Installations of butylene polymerization-hydrogenation plants were more numerous. The largest plant was installed by the Korean Nitrogen Fertilizer Company at KONAN, Korea, with a rated capacity of 30,000kl per year of iso-octane, operating on butylenes from acetylene. Actual output was substantially less than this, probably around 10,000kl per year. The next largest plant, located at YOKKAICHI, was designed to produce 10,000kl per year, operating on butylenes from butanol. Actual output of this plant was reported as negligible, however, due to shortages in the supply of butanol obtained from Formosa. A large installation, for producing iso-octane from acetylene (derived from natural gas by the arc

process) and from butanol was also under construction at the sixth Naval Fuel Depot at SHINCHIKU in ~~1944~~.

The remainder of the plants in Japan were much smaller, in the range of 200kl to 2,000kl per year, all of which operated on butylenes in refinery ~~cracked gases. These smaller plants reported to be in existence included~~ one unit each at TOKUYAMA, MARIFU, TSURUMI and KAWASAKI, as well as an additional small unit at YOKKAICHI.

There is evidence that the actual total production of iso-octane in the Empire did not exceed 12,000kl per year. In any event, the amount produced was insufficient to permit production of 100 octane gasoline in any more than experimental quantities. The large bulk of the iso-octane, both imported and of Japanese manufacture, was used in the blending of 91 and 92 octane aviation fuels.

2. Production of Butylenes

Several alternate sources for the production of butylenes to be used as the raw material for iso-octane manufacture were investigated, and some of these sources were applied commercially. The dehydrogenation of n-butane using an aluminum oxide-chromic acid catalyst was studied briefly at the First Naval Fuel Depot but was discontinued because of the scarcity of cracked gas (Enclosure (B)1).

Before the war, elaborate plans were formulated for producing butylenes from n-butanol. Fermentation plants for the production of butanol from sugar molasses and sweet potatoes were erected throughout Japan. These plants were intended to provide sufficient butanol for the production of 100,000kl per year of iso-octane, but by 1944 they had all been converted to the production of ethyl alcohol because of the serious shortage of aviation gasoline and the higher yields obtained in the ethyl alcohol fermentation process. Details of this alcohol program and the related research projects studied by the Japanese Navy are described in the NavTechJap Report, "Japanese Fuels and Lubricants, Article 3 - Naval Research On Alcohol Fuel", Index No. X-38(N)-3.

It is of particular interest that the largest iso-octane plant in the Japanese Empire utilized butylenes synthesized from acetylene. Construction of this plant was begun in 1937 by the Chosen Chisso Hiryo K.K. (Korean Nitrogen Fertilizer Company) at KONAN, Korea, and had a design capacity of 30,000kl per year of iso-octane. The acetylene was obtained from calcium carbide, a method particularly attractive to the Japanese in view of abundant supplies of coal and hydro-electric power. Research pertaining to this process was continued by the Navy, in cooperation with the Korean Nitrogen Fertilizer Company throughout the war.

Construction of another iso-octane plant utilizing acetylene was started at the Second Naval Fuel Depot, YOKKAICHI, but was never fully completed. The acetylene was to be prepared by the cracking of refinery gas using an electric arc process, details of which are given in Enclosures (B)14 and (B)15.

The synthesis of iso-butene from acetylene proceeded through the following steps:

- (a) The preparation of acetaldehyde from acetylene using a dilute sulfuric acid solution of mercuric sulfate as catalyst.
- (b) The preparation of crotonaldehyde from acetaldehyde using the aldol condensation method and subsequent distillation.

(c) The preparation of butanol from crotonaldehyde using a mixed NiO-CuO catalyst supported on diatomaceous earth (Ni:Cu=1:3).

(d) The dehydration of n-butanol to n-butene using an activated Japanese acid clay catalyst.

(e) Isomerization of n-butene to iso-butene at 350°-400°C. using a phosphoric acid catalyst supported on charcoal.

The research conducted by the Japanese Navy relative to each of the above steps is described in detail in Enclosure (B)10. One of the major drawbacks encountered in the commercial application of the process was attributed to the loss of mercury catalyst in converting acetylene to acetaldehyde, especially in view of the scarcity of mercury in Japan. To circumvent this difficulty, other methods for effecting the conversion were studied (Enclosure (B)13). Of particular interest in this connection are the pilot plant studies relative to the methylation of acetylene and subsequent hydrolysis of the vinyl methyl ether thus formed.

The preparation of n-butene from acetylene by catalytic polymerization to vinyl acetylene followed by partial hydrogenation was also studied but was not considered suitable for commercial application (Enclosure (B)11).

C. THE MANUFACTURE OF AVIATION BASE STOCKS FROM NON-PETROLEUM SOURCES

The acute shortage of aviation gasoline base stock during the latter part of 1944 necessitated the consideration of many non-petroleum sources for this type of fuel. Although the study of most of these substitutes never advanced beyond the laboratory or pilot plant scale, yet there were two sources for which large scale production programs were initiated. These two programs involved the use of ethyl alcohol and pine root oil previously discussed in this report and described in detail in the two NavTechJap Reports, "Japanese Fuels and Lubricants, Article 3 - Naval Research on Alcohol Fuel", Index No. X-38(N)-3, "Japanese Fuels and Lubricants, Article 4 - Pine Root Oil Program", Index No. X-38(N)-4.

Investigations of the application of additional non-petroleum sources to the preparation of substitute aviation fuels are described below:

1. Soya Bean Oil

The preparation of gasoline from soya bean oil by cracking in the presence of activated Japanese acid clay had been investigated in Japanese research laboratories, but the quality of the fuel thus formed was unsatisfactory for aircraft use. In 1944 the First Naval Fuel Depot initiated autoclave studies on the preparation of aviation gasoline by the thermal cracking of soya bean oil. A 20 per cent yield of gasoline having an octane number of 89 (0.15% lead by volume) was obtained. Complete data are presented in Enclosure (B)17.

In 1945 laboratory and pilot plant studies of the catalytic hydrocracking of soya bean oil were undertaken. Gasoline having an octane number of 85 (0.15% lead by volume) was prepared in 40 per cent yield using a molybdenum trisulfide catalyst at 200 atmospheres and 420-450°C. Subsequent autoclave tests indicated that titanium dioxide was a more effective catalyst, but no pilot plant data were obtained. Details of the catalytic hydrocracking of soya bean oil are given in Enclosure (B)18.

2. Rubber

In 1942 Japan's supply of natural rubber was more than adequate for her needs, and, in consequence, this raw material was considered as a source of hydrocarbons for aviation gasoline. Although gasoline was never prepared from rubber commercially, many laboratories investigated the problem

during the war period.

In pilot plant studies at the First Naval Fuel Depot, rubber was decomposed in a pipe still, cracked, and fractionated in a continuous unit (Enclosure (B)19). Hydrocracking of the product yielded a gasoline with an octane number of 73 (unleaded), or 92.4 (0.15% lead by volume). This product consisted mainly of naphthenes (41.2%) and paraffines (46%) and appeared to be suitable as an aviation gasoline, although service testing of the fuel was never undertaken in view of the scarcity of rubber by 1944.

Similar pilot plant studies were conducted at the Army Fuel Research Institute, FUCHU. The procedures used in each of two processes studied are described as follows:

- (a) Crude rubber was depolymerized and liquefied at 280°C., under steam pressure. The oil product was hydrocracked in the liquid phase using a slurry catalyst, and the middle oil thus obtained was hydrogenated in the vapor phase to aviation gasoline. The gasoline thus prepared had an octane number of 91 with 0.15 per cent lead by volume.
- (b) The rubber was depolymerized as in method (a), thermally cracked at 420-450°C., and the middle oil thus obtained was hydrogenated in the vapor phase using a molybdenum catalyst. The product had an octane number of 95 with 0.15 per cent lead by volume.

Professor KIROSHIMA of Kyushu Imperial University, who has specialized in the chemistry of rubber for many years, also studied the possibility of obtaining a high octane aviation fuel from rubber. A summary of the several different methods investigated in his laboratory and the characteristics of the resulting gasolines is presented in Table III. Detailed reports (in Japanese) of these studies were obtained and are listed in Enclosure (C).

Table III
AVIATION GASOLINE FROM RUBBER

(Data obtained from Kyushu Imperial University)

Method	Yield (Wt. %)	Analysis			Octane Number	
		Olefines	Aromatics	Naphthenes and Paraffine	Clear	Leaded 0.15% by vol.
Dry Distillation	36	42	30	28	75	77
Dry Distillation followed by catalytic cracking with acid clay	31	12	27	61	80	85
Dry Distillation in presence of AlCl ₃	25	14	24	62	75	88
Dry Distillation in presence of AlCl ₃ followed by catalytic cracking with acid clay	45	3	86	11	90	94
Hydrogenation using NiO catalyst at 450°C. and 100 atmospheres	48	2.4	37.3	59.3	81	96

3. Other Sources

During 1945, the Army Fuel Research Institute tested several other materials as possible sources of aviation gasoline. Although these were only laboratory studies, it is of interest that such substances as camphor, camphor oil, birch bark, pine needles, and orange peel were considered.

Part II
SERVICE TESTS

A. ENGINE TEST FACILITIES AT THE FIRST NAVAL FUEL DEPOT, OFUNA

1. Single Cylinder and Full-Scale Aircraft Engine Tests

Engine test facilities were installed at the First Naval Fuel Depot in 1942 in order to examine the service performance characteristics of aviation fuels and lubricants. Up until this time, the service testing had been carried out at the First Naval Technical Depot, YOKOSUKA, and the new installations at OFUNA were added to provide a more convenient means for the study of aviation fuels and lubricants in connection with research on manufacture. The test equipment comprised one laboratory equipped with several single-cylinder aircraft engines, and two separate test installations for full scale multi-cylinder engine work. The engine tests were supported by two other laboratories, one equipped to carry on analytical tests of lubricants, and the other for the chemical testing of fuel, and C.F.R. Motor Method octane number determinations. There was no evidence in the Japanese Navy's research of any routine testing in C.F.R. type engines of anti-knock properties of fuels under rich-mixture conditions.

The single-cylinder and multi-cylinder full scale test installations are described in Enclosure (B)33, and the test operating methods are discussed. These full scale stands were well laid out, soundproofed, and seemed to be comparable to American standards. Details of the single-cylinder and multi-cylinder engine installations are shown in Figures 1 to 11. Special attention is called to Figure 4, which shows the design features of the "KINSEI", No. 4 type, single-cylinder, variable-compression ratio, counter-balanced aircraft test engine manufacture by the Ishikawajima Aircraft Company, Ltd.

In detonation tests in aircraft engines, the maximum allowable boost and corresponding horsepower were determined over the useful range of air-fuel ratio, using cylinder head temperature and exhaust flame color to indicate limits due to detonation.

2. Low-Temperature and Low-Pressure Experimental Laboratory

An elaborate high-altitude, low-temperature cold room installation was constructed to conduct research on fuels, lubricating oils and hydraulic oils under conditions up to 20,000 meters altitude and temperatures as low as -55°C. This unit was designed by the Eba Company, Ltd. The cost of the installation was estimated to be 2,300,000 yen at the time construction commenced in December 1943. In August 1945, although construction of the plant was well advanced, work was discontinued because of the destruction of the Eba Company by bombing, at which time the designer was killed, and many of the design details, models, and castings were destroyed.

The plant consisted of two parts. One part comprised the low-temperature and low-pressure rooms and refrigerating means, using Freon 12 and Freon 11 as cooling media. The other part consisted of a single-cylinder aircraft engine testing plant designed for low-temperature, low-pressure operation. The first section was 80 per cent completed, but the second section reached the design stage only, except for the engine foundations. The plant is described in Enclosure (B)34, which include detailed drawings of the design features, refrigeration and carbon dioxide fire extinguisher systems, and the single-cylinder test installation. These design data are included in Figures 1 to 5, inclusive, and were prepared for the Petroleum Section of the U.S. Naval Technical Mission to Japan. The layout is shown in this much detail in view of the interest in such research in the United States.

B. WARTIME MODIFICATION OF AVIATION GASOLINE SPECIFICATIONS

In 1937, the Japanese Navy's specifications for aviation grade gasoline included 92 minimum octane number requirement, a minimum of tetraethyl lead content of 0.1 per cent by volume, and maximum temperature of 105°C. and ~~150°C. at the 50 percent and 90 per cent distillation points, respectively.~~ These specifications were modified during the war in an attempt to increase the supply of aviation gasoline. The original specification and subsequent modifications are tabulated in Table IV. Significant changes are discussed below.

1. Distillation Range

With the increasing shortage of aviation gasoline, tests were initiated in the middle of 1943 to modify the specifications to permit heavier gasoline. Engine performance tests conducted at the First Naval Fuel Depot and the First Naval Technical Depot showed that the maximum distillation temperatures for the 50 per cent and 90 per cent points could be raised, but that in so doing, the octane number of the product was lowered from 0.2 to 0.8 for each per cent increase in the yield of gasoline from the crude oil due to the lower octane numbers of the higher boiling fractions.

The two alternate specifications investigated are shown as Modification No. 1 and Modification No. 2 in Table IV. Calculations based on the proposed changes indicated an increased yield of aviation gasoline from East Indies crude of 25 per cent by the first modification and 80 per cent by the second modification. This represented an increased production of 300,000 kiloliters and 1,000,000 kiloliters of aviation gasoline per year, respectively, on the basis of expected imports of 8,450,000 kiloliters per year of East Indies crude oil.

Additional service tests carried out at the First Naval Technical Depot showed that starting characteristics, when using Modification No. 1 gasoline, were unsatisfactory at atmospheric temperatures below 10°C., whereas with Modification No. 2, aviation gasoline starting problems were encountered at temperatures below 20°C. To avoid this difficulty, carburetted engines were supplied with five liters of standard aviation gasoline, from a special tank, for starting purposes. Furthermore, lubricating oil analyses after using these two fuels under flight test conditions showed increased dilution, particularly with Modification No. 2 gasoline.

With the above limitations, it was concluded that Modification No. 1 was satisfactory for combat planes even in winter and that No. 2 was satisfactory for use in summer. In view of these findings, the Japanese Navy's specifications for aviation gasoline were changed accordingly and were used successfully during the balance of the war (Enclosures (B)24 (Part I), and (B)25).

2. Octane Number

In 1942 it was found necessary to decrease the specified minimum octane number of aviation gasoline from 92 to 91 because of difficulties in manufacturing a sufficient quantity of the 92 octane fuel. In 1945, coincident with the decrease in volatility, the minimum octane number was lowered to 87 for the summer grade gasoline specified by Modification No. 2.

3. Tetraethyl Lead Content

Engine tests were carried out relative to increasing the tetraethyl lead content to compensate for the loss in anti-knock quality mentioned in the

preceding paragraphs. In 1944 the lead content of aviation gasoline was changed from 0.10 per cent to 0.15 per cent (by volume) to compensate for this. The ethylene dibromide equivalent was maintained at 1.0. Engine tests were also conducted in 1944 using aviation fuels containing 0.3 per cent by volume of tetraethyl lead and with ethylene dibromide equivalents of 1.0, 1.3, and 1.5. Significant findings included the following:

(a) It was found that in endurance tests in full-scale engines using gasolines of low volatility, as discussed under Modifications 1 and 2, the use of 0.3 tetraethyl lead with 1.5 ethylene dibromide equivalent was unsatisfactory, due to the burning of the plugs or fouling with lead deposits, causing the engine to misfire after 10 to 15 hours operating time.

(b) Endurance tests were also made with aviation gasoline of the boiling range specified prior to 1943, but containing 0.3 per cent by volume of tetraethyl lead, and it was found that the most suitable equivalent of ethylene dibromide was 1.3. This finding was of theoretical interest only since it became necessary to obtain increased gasoline production by changing the distillation range (Enclosure (B)24, Part 2, and (B)26).

Table IV
WARTIME SPECIFICATIONS FOR JAPANESE NAVY COMBAT AVIATION GASOLINE

	Date Established	Distillation (°C max)					Sum of 10% 50% and 90% (min)	Reid Vapor Pressure (kg/cm ² min)	Sulfur (% max)	Unsaturation (% max)	Freezing Point (°C max)	Octane No. C.P.R. Motor Methods (min)	T.M.L. Volume (% max)
		I.B.P.	10%	50%	90%	97%							
Aviation 92	1937	60	80	105	150	170	260	0.6	0.1	1.0	-50	92	0.10
Aviation 91	1942	60	80	105	150	170	260	0.53	0.1	1.0	-50	91	0.10
Aviation 91 (Mod. 1)	1944	60	90	115	160	170	260	0.6	0.1	1.0	-50	91	0.15
Aviation 87 (Mod. 2)	1945	70	90	125	180	200	260	0.6	0.1	1.0	-50	87	0.15

4. Ethylene Dibromide Substitutes

With the curtailment of imported stocks of ethyl fluid in 1941, coupled with the decrease in production of bromine from brine during the same period, a substitute for ethylene dibromide became an urgent problem, and chlorine compounds were investigated. These studies disclosed that butylene chloride was the most suitable substitute for ethylene dibromide, but when this substance was used, the spark plugs had to be changed after every 25 hours of engine operation (Enclosure (B)27).

SPECIAL STOCKS AND BLENDS

1. Aromatics

Engine tests were carried out during the period 1942 to 1944 in connection with the use of aromatic hydrocarbons as aviation fuels. These fuels were produced at the First Naval Fuel Depot and were tested in single cylinder aircraft engines at the First Naval Fuel Depot and multi-cylinder engines at the First Naval Technical Depot. Benzol, toluene, Xylene, solvent naphtha, and "Benzex" (sulphur dioxide extract from East Indian topped gasoline) were investigated. Significant findings included:

(a) Single Cylinder Kinsei Engine

(1) When the concentration of benzol added to gasoline exceeded 20 per cent, detonation occurred in spite of high octane number

as indicated by the C.F.R. engines.

(2) Straight toluene had the best performance of the aromatics tested as aviation fuel and equalled or surpassed iso-octane.

~~(3) Xylene and higher aromatics gave good anti-knock properties, but these aromatics had poor volatility characteristics.~~

(b) Multi-Cylinder Engines (Homare 20, Kasai 20 and Atsuta 20)

(1) "Benzex" and solvent naphtha in 85 per cent concentrations both were found unsatisfactory because their volatilities were too low for use even with direct injection engines and the exhaust gases from these fuels contained excessive carbon which coated the spark plugs with heavy deposits after a few hours operation.

(2) The aromatics actually used in aviation gasoline consisted of 20 to 30 per cent of "Benzex" blended with natural gasoline made from EAST BORNEO crude (Enclosure (B)22 and (B)23). This fuel was used principally in the Netherlands East Indies war theater.

2. Oxy-Compounds

In the period 1937 to 1938, research was conducted on oxy-compounds, such as ketones and ethers, as blending stocks in lieu of iso-octane. These compounds were added in 20 per cent concentrations to TALAGIMAR gasoline, which had an octane number of 89.9 when containing 0.1 per cent of tetraethyl lead. While C.F.R. engine tests showed the ketones and ethers promising, aircraft engine tests indicated that these compounds were deficient in anti-knock characteristics (Enclosure (B)31).

3. Ethyl Alcohol

See NavTechJap Report "Japanese Fuel and lubricants, Article 3 - Naval Research on Alcohol Fuel", Index No. X-38(N)-3, Enclosures (B)17 to (B)24, inclusive.

4. Pine Root Oil Gasoline

See NavTechJap Report, "Japanese Fuels and Lubricants, Article 4 - Pine Root Oil Program", Index No. X-38(N)-4, Enclosures (B)10 and (B)11).

D. ANTI DETONANTS AND DOPES

1. Aromatic Amines

The Japanese aviation gasoline octane number (C.F.R. Motor Method) was established in 1942 at a 91 minimum, and gasoline produced from Sumatra crude could not reach this figure with the addition of only 0.15 per cent by volume of tetraethyl lead, the maximum amount permitted as discussed in Section B-2 above. Consequently, tests were carried out in regard to the use of aromatic amines in combination with gasoline produced from Sumatra crude and containing 0.15 per cent by volume of lead. It was found that of the aromatic amines tested, aniline was the most suitable and an octane number of 91 was obtained by the addition of 1 per cent to 3 per cent, although many problems were encountered, such as the formation of supercharger and piston head deposits, solvent action on the paint of the oil tight seams and of the cork in the carburettor float, and corrosive action on copper and iron. Steps were taken to overcome these objections so that this type of fuel could be used in combat planes (Enclosure (B)29), although the application of aromatic amines was not reduced to actual prac-

tice.

2. Selenides

Research was carried out from 1937 to 1939 on the use of selenium compounds as anti-detonants for producing gasolines of more than 100 octane number by the addition of such materials to Stanavo 100 octane fuel. These selenides were tested in a one-cylinder aircraft engine and the results showed that 0.8 per cent by volume of diethyl selenide increased the octane number to 112, as determined by the C.F.R. Motor-Method (by extrapolation), and increased the horse power in single cylinder aircraft engine tests by 15 per cent. It was further determined that diethyl selenide was the most desirable of the selenide compounds tested, that it was not poisonous and did not possess a disagreeable odor, if prepared in a pure state. This research work was not pursued further due to the small amount of selenium available in Japan (Enclosure (B)30).

3. Influence of Moisture on Tetraethyl Lead

Following the findings of J.R. McGregor, Standard Oil Company of California, tests were conducted from 1942 to 1943 in a C.F.R. engine to determine the effect of the humidity of the air on octane number determinations. These experiments gave the following results:

- (a) For unleaded gasoline, the influence of moisture was within one octane number.
- (b) With leaded gasolines, the influence of moisture on octane number was appreciable.

A correcting formula was established for leaded gasolines in lieu of using humidifiers attached to the C.F.R. engines in routine knock testing (Enclosure (B)32).

Part III ENGINE COMBUSTION RESEARCH

This section of the report deals with investigations of flame propagation and detonation in engine cylinders conducted by Engineering Commander K. NAKATA, IJN, over a period extending from 1933 to 1944. These studies are included herein because they relate primarily to aviation fuels.

The research work was done, for the most part, at the First Naval Fuel Depot, YOKOSUKA, and is presented in four papers (Enclosures (B)35 to (B)38, inclusive). In reviewing these papers, it should be borne in mind that the Japanese files had been burned and that the reports were, therefore, prepared by Commander NAKATA from some personal notes and from memory.

It is believed significant that the Japanese Navy was sufficiently research-minded not only to engage in this field of research in peace time, but also to carry on fundamental combustion studies in quartz tubes during the height of the war.

The four reports on combustion research, are briefly discussed below:

A. PHOTOGRAPHIC INVESTIGATIONS OF FLAME PROPAGATION AND DETONATION IN ENGINE CYLINDERS (ENCLOSURE (B)35)

This paper deals with exploratory tests made from 1933 to 1935 on flame travel under detonating and non-detonating conditions. An air-cooled 1 1/2 hp "11"

single-cylinder test engine with water-cooled head was fitted with a narrow quartz window, through which flame propagation was studied photographically. A film drum was mounted above the engine upon which a standard size film was wound. Sodium bicarbonate was used to color the flame. In view of the low compression ratio of the engine, knocking was induced by the addition of 20 to 30 per cent of ethyl nitrate to the fuel. The findings included:

1. There was no difference in flame velocity for the knocking and non-knocking condition in that portion of the cycle between the spark position and the knocking position.
2. It was impossible to measure flame speed in the knocking region, as here the flame front was always vertical to the time axis.
3. Vertically striped bright flame patterns were observed in the detonation region. The frequency of these patterns was measured and found to decrease as the piston went down in the cylinder, presumably because of the adiabatic compression of burned gas due to detonation waves.

These early flame studies were primarily carried out to acquaint the investigator with some of the fundamentals of engine combustion and to provide a basis for further research in this field.

B. ENGINE DETONATION STUDIES BY PIEZO-ELECTRIC INDICATOR (ENCLOSURE (B)36)

In order to determine whether the bright-striped patterns discussed in the previous section were due to the adiabatic compression or rarefaction in the detonation waves, research was undertaken to measure the pressure inside the cylinder with an indicator of small inertia and high natural frequency. A water-cooled Piezo-electric Indicator had previously been used at Tokyo Imperial University, but its natural frequency was too low. The author developed an air-cooled Piezo-electric quartz pick-up unit having a natural frequency in the order of 50,000 cycles per second, and this design (Enclosure (B)36, Figure 3) gave satisfactory results. A pressure-time diagram was obtained, using an electro-magnetic oscillograph.

With this unit, experiments were carried out on a number of engines, including a C.F.R. engine equipped with a quartz window. On full-scale aircraft engines, vibration attributed to the amplifier tubes occurred in the pressure line when the engine detonated. Significant findings included:

1. When indicator cards were taken simultaneously with photographs of engine flame, the number of vibrations on the pressure line was nearly twice the number of striped patterns observed photographically in the detonating flame. In the case of normal combustion, the pressure curve on the indicator card was smooth, and no striped patterns were observed.
2. Under detonating conditions the frequency of vibrations was greater the smaller the bore of the engine used, and this frequency decreased as the piston progressed downward from the top dead-center position.

No other forms of indicators were built by Comdr. NAKATA, but the French "Detonindex" indicator was tried. This indicator was said to consist of a bouncing pin and induction circuit whereby the EMF produced caused a neon lamp to glow under detonating conditions.

C. FLAME PROPAGATION IN ENGINE CYLINDERS, STUDIES BY THE IONIZATION METHOD (ENCLOSURE (B)37)

During the period 1935 to 1940 flame propagation in aero-engine cylinders was studied by the ionization gap method. This method was utilized in view of the difficulty of installing a quartz window over the head of a full scale aircraft engine cylinder. Tests were made in the DVL variable compression engine equipped

with a water-cooled BMW cylinder. In these tests a number of ionization plugs were installed in the cylinder head and cylligrams were obtained, which were converted to rate of propagation by using the time scale recorded on the same film. The following conclusions are of interest:

1. ~~Flame velocity is higher with high-octane fuels than with low-octane fuels.~~
2. Flame velocity is greatly reduced under lean and rich mixture conditions.
3. Velocity of flame propagation is increased with increase in engine speed.
4. There is a slight increase in flame speed with increase in boost pressure.
5. Flame front is markedly distorted by turbulence.

Commander NAKATA concluded that the ionization gap method is well-suited for investigating flame propagation in cylinders of full-scale aircraft engines.

D. STUDIES OF SLOW OXIDATION OF HYDROCARBONS BY ABSORPTION SPECTRA (ENCLOSURE (B)38)

In 1938, after reviewing reports written by Rassweiler and Withrow of the General Motors Research Corporation, Comdr. NAKATA constructed an engine fitted with two quartz windows on opposite sides of the cylinder and a stroboscopic arrangement enabling flame study at any phase of the engine's revolution by observing emission or absorption spectra. However, in view of the difficulties encountered in the engine's operation, this procedure was abandoned and quartz tube slow oxidation research by absorption spectra was undertaken, using the method developed by Ubbelohde and Edgerton.

Tests were carried out with various typical hydrocarbons at different temperatures. Hydrocarbon-air mixtures were used in a mixture strength ratio of 13 to 1, under both flowing and stagnant conditions. The conclusions obtained in these tests include the following:

1. Flow Method

- (a) Iso-octane is stable with temperature, but low octane fuel, such as normal heptane, is reactive with oxygen at relatively low temperatures.
- (b) The oxygen absorption of cyclo-hexane begins at a temperature between that of heptane and that of iso-octane, and the form of absorption differs from both.

2. Stagnant Method

- (a) Octane numbers of hydrocarbons, except aromatics, can be determined by reaction temperatures measured in a quartz tube.
- (b) In the case of normal heptane the appearance of absorption bands at 2600 A⁰, called the "U" band, accompanies a depression of pressure reportedly due to the formation of peroxides.
- (c) The presence of tetraethyl lead in normal heptane does not suppress the formation of the substance causing the "U" band, but does have some effect on the spectrum at higher temperatures.