

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

TMJ

MT

X-38(N)-10

15 February 1946

RESTRICTED

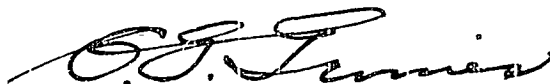
From: Chief, Naval Technical Mission to Japan.
To : Chief of Naval Operations.

Subject: Target Report - Japanese Fuels and Lubricants, Article 10 -
Miscellaneous Oil Technology and Refining Installations.

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

1. Subject report, covering miscellaneous oil technology and oil refining installations in Japan 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, Lt. Comdr. C. S. Goddin, USNR, Lieut. W. H. Millet, USNR, and Ens. E. R. Dalbey, USNR, interpreter and translator.



C. G. GRIMES
Captain, USN

30705

**JAPANESE FUELS AND LUBRICANTS - ARTICLE 10
MISCELLANEOUS OIL TECHNOLOGY
AND REFINING INSTALLATIONS**

**"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 10 OIL TECHNOLOGY AND REFINING INSTALLATIONS

This report includes certain miscellaneous data pertaining to petroleum refining installations and technology obtained by the Petroleum Section of the U. S. Naval Technical Mission to Japan.

The refining industry in Japan was based largely on imports of foreign crudes principally from the United States, and the refining techniques utilized were chiefly drawn from American practice.

An expensive industry was developed for the manufacture of refining equipment and the newer refineries were essentially 100% Japanese-made. The quality and appearance of such equipment as pressure vessels, tanks, pumps, and heat exchangers compared favorably with that produced in America.

The Japanese refining technology differed from American mainly with regard to processes utilized for the production of aviation gasoline. The manufacture of iso-octane by alkylation and polymerization was very limited due to inadequate supplies of C₄ hydrocarbons. Emphasis was put on the hydrocracking of kerosene and gas oils, with strenuous efforts being made to develop a fixed-bed catalytic cracking process. Several plants were put into operation by late 1943, using Japanese acid clay as a catalyst. The fluid-catalyst technique was unknown to Japan.

With regard to manufacture of oil from shale, a plant for refining crude wax shipped from the shale works at FUSHUN, was visited at TOKUYAMA, but no evidence of the existence of any shale mining or retorting operations in Japan was discovered.

TABLE OF CONTENTS

Summary	Page 1
List of Enclosures	Page 3
References	Page 4
Introduction	Page 5
The Report	Page 7

LIST OF ENCLOSURES

- (A) List of Documents in Japanese Pertaining to Research on Miscellaneous Oil Technology at the First Naval Fuel Depot, OFUNA, forwarded through ATIS to the Washington Document Center Page 11
- (B) Detailed Reports of Research on Miscellaneous Oil Technology at the First Naval Fuel Depot, OFUNA
 - (B)1 "Studies on Acid Clay" by Chem. Eng. Comdr. H. FUJIMOTO Page 15
 - (B)2 "Entrainment and Plate Efficiency or Bubble-Cap. Rectifying Columns" by Chem. Eng. Lieut. T. YOKOYAMA ... Page 21
- (C) "Development of Catalytic Cracking in Japan" by N. NAKAHARA, President of Toa Nenryo K. K. Page 33
- (D) "The Petroleum Industry in Japan" by J. AOKI and G. NARA of the Shun Nomura Office Page 43
- (E) "Nippon Oil Co., Kudamatsu Plant" - Report by NavTechJap Page 59
- (F) "The Third Naval Fuel Depot, Tokuyama Refinery" Report by NavTechJap Page 67
- (G) "The Nippon Seiro K. K., Tokuyama Plant" - Report by NavTechJap Page 81

REFERENCES

Location of Target:

First Naval Fuel Depot, OFUNA
Third Naval Fuel Depot, TOKUYAMA
Nippon Seiro K. K., TOKUYAMA
Nippon Oil Co., KUDAMATSU

Japanese Personnel Interviewed:

H. FUJIMOTO: Chemical Engineering Commander, Japanese Navy, head of the cracking, dry distillation and pine root oil sections at the First Naval Fuel Depot, OFUNA.

T. YOKOYAMA: Chemical Engineering Lieutenant, Japanese Navy, at the First Naval Fuel Depot, OFUNA.

N. NAKAHARA: President of the Toa Nenryo K. K. (closely associated with development of catalytic cracking in Japan. Visited United States in 1939 in attempt to purchase Houdry Patents).

S. NOMURA: President of the Tatsumi Engineering Company and a leading importer of crude oil into Japan.

NISHINO: Vice-Superintendent of the Nippon Oil Company Refinery at KUDAMATSU.

I. WATANABE: Rear Admiral, Japanese Navy, Director of the Third Naval Fuel Depot, TOKUYAMA.

M. TAMURA: General Manager of the Nippon Seiro Plant at TOKUYAMA.

In addition to the key personnel listed above, a number of technical assistants were interviewed and are listed in the Enclosures contained in this report.

INTRODUCTION

In the course of gathering information on fuel and lubricant developments in Japan, several petroleum refining installations were inspected and certain miscellaneous data pertaining to oil refining technology were secured by the Petroleum Section of the U. S. Naval Technical Mission to Japan. No attempt was made to completely survey conventional refining installations or to obtain statistical information on imports, production, inventories etc., since such aspects were being thoroughly studied by other agencies, especially the U. S. Strategic Bombing Survey and the U. S. Army Intelligence Branches.

In view of the foregoing, the following comments refer only to refining features or refinery data thought to be of special interest and not discussed in the other fuel and lubricant reports prepared by NavTechJap.

Two crude oil refineries, the Nippon Oil Co. refinery at KUDAMATSU and the Japanese Navy refinery at TOKUYAMA, were inspected and are described in Enclosures (E) and (F). An inspection was also made of the Nippon Seiro Co. at TOKUYAMA, which manufactured paraffin wax from crude shale wax shipped from the shale retorting installations at FUSHUN (Enclosure (G)). Of interest are two papers prepared at the request of the Petroleum Section; one reviewing the development of catalytic cracking in Japan by N. NAKAHARA, President of the Toa Nenryo Co., and the other surveying the development of the civilian petroleum industry in Japan, prepared by the Shun Nomura Office, (Enclosures (C) and (D)). Certain miscellaneous reports and a list of documents obtained from the First Naval Fuel Depot, OFUNA, are also presented, Enclosures (A) and (B).



THE REPORT

Prior to the war, Japan's domestic consumption of petroleum amounted to about 20,000,000 barrels per year. Domestic production of crude oil was only about 2,500,000 barrels per year maximum, (including Formosa), and synthetic oil manufacture, including shale oil from Manchuria, amounted to about 850,000 barrels per year. Consequently, about 84% of the nation's peacetime requirement had to be met by importation of foreign crudes, especially from America. The petroleum refining industry in Japan was built largely on the basis of processing imported crudes, and the technology utilized was chiefly based on American distillation, cracking, and treating processes. The total refining capacity in the four main islands of Japan has been estimated at 35-40,000,000 barrels per year.

In anticipation of the war, Japan purchased crude and refined products in excess of requirements, and it has been reported that more than 60,000,000 barrels were in storage prior to Pearl Harbor. Tremendous storage installations were constructed for this purpose. For example, the bulk storage capacity of the Third Naval Fuel Depot at TOKUYAMA was some 9,000,000 barrels; the Sasebo Navy Yard, 4,000,000 barrels; and the Kure Navy Yard, 1,000,000 barrels, all mostly underground.

After the outbreak of the war, imports practically stopped until oil from BORNEO and SUMATRA became available in 1942. By the end of 1943, the submarine and air blockade caused imports of these East Indies oils to drop rapidly. By the beginning of 1945, most refineries in Japan were shut down due to lack of crude. This is illustrated by the following data obtained from the 1500kl/day Navy Refinery at TOKUYAMA, and the 800kl/day Nippon Oil Co. Refinery at KUDAMATSU.

CRUDE RUN - KILOLITERS PER YEAR

YEAR	TOKUYAMA		KUDAMATSU		
	EAST INDIES	CALIF.	EAST INDIES	CALIF.	MEXICO & MISC.
1942	0	550,000	16,300	22,200	28,300
1943	290,000	260,000	34,500	130	600
1944	240,000	226,000	69,600	0	0
1945	12,000	0	18,100	0	0

Further data on various aspects of the civilian petroleum industry in Japan are included in Enclosure (D), prepared by the staff of the Shun Nomura Office in TOKYO.

Both the Japanese Navy and Army operated refineries in the Japanese Empire. The Navy had three refineries, a 17,000 bbl/day plant at YOKKAICHI (The Second Naval Fuel Depot), a 9500 bbl/day unit at TOKUYAMA (The Third Naval Fuel Depot), and a small unit at TAKAO Formosa (The Sixth Naval Fuel Depot). The Army initiated an active fuel and research program only shortly before the war and constructed several refineries, the largest of which was begun in 1940 at IWAKUNI.

Both the Yokkaichi and Tokuyama refineries were very similar with respect to the processes and types of equipment utilized. The refinery at TOKUYAMA was the older of the two, the first pipe-still in Japan having been installed there

in 1920. This refinery was visited and is described in Enclosure (F). This refinery was staffed by Naval petroleum specialists and was one of the most modern and best equipped refineries in Japan, comparing favorably to the best American practice. It was equipped to carry on crude distillation, thermal cracking, iso-octane manufacture by hydrogenation of butene polymers, hydrogenation of cracked gasoline, hydrocracking of kerosene, synthesis of methanol, solvent refining of lube oils, and the manufacture of greases and tetra-ethyl lead. Near the close of the war preparations were being made for the processing of crude pine root oil for the manufacture of aviation gasoline.

One of the impressive features about the newer refining and synthetic oil plants visited in Japan was the fact that the actual equipment, including furnaces, towers, tanks, piping, pumps, heat exchangers, instruments, etc., was almost completely manufactured in Japan, and further, this equipment appeared to be of high quality.

Japanese refineries differed from modern American plants primarily in the methods employed for manufacturing aviation gasoline. The most successful processes from the standpoint of the amount of aviation gasoline actually produced appear to have been high pressure hydrogenation of cracked gasoline and hydrocracking of kerosene or gas oil. Strenuous efforts were made to develop a fixed-bed catalytic cracking process after negotiations to purchase rights to the Houdry and U.O.P. catalytic cracking processes failed due to the Moral Embargo in 1939. An interesting review of the development of the catalytic cracking process in Japan, prepared by Mr. N. NAKAHARA, President of Toa Nenryo Co., is given in Enclosure (C).

The fluid catalyst technique was not applied in Japan, and no indication of research studies or application of this principal to any process was evidenced at any of the several petroleum activities visited by the Petroleum Section of NavTechJap. Catalytic cracking as developed in Japan was a simple process utilizing activated Japanese acid clays as catalyst and adiabatic type reactors (with the exception of the Toa Nenryo Co., which used steam under pressure as a heat-removing medium). The Japanese Navy installed two 2000 bbl/day catalytic cracking units at the Yokkaichi Refinery, one of which was completed and produced 10,000kl. of aviation gasoline in 1944. Supplies of gas and oil charging stock were exhausted and the second unit completed in 1945 was never operated. These units had automatic valves and operated on a cycle of 10 minutes cracking and 20 minutes regeneration.

Japanese acid clay is worthy of special note because of the wide variety of its application in oil refining. It was used as a catalyst for catalytic cracking (Enclosure (B)1), catalytic reforming (article 4 of this series, "The Pine Root Oil Program in Japan", Index No. X-38(N)-4 Enclosures (B)-3 and (B)-4), catalytic dehydration of alcohols (article 2 of this series, "Aviation Gasoline Research by the Japanese Navy", Index No. X-38(N)-2, Enclosure (B)-10), and in the treating of gasolines and lube oils (Enclosure (D)).

The Japanese Navy employed an acid clay from the Takeda Acid Clay Co., produced near KOMATSU City, Ishikawa District, for the catalytic cracking unit at YOKKAICHI. This clay was said to be at least as effective as U.O.P. Synthetic. A typical analysis of this clay, after treatment by boiling three hours with 2N. sulphuric acid, follows:

Ignition Loss.....	8.5%
Al ₂ O ₃	24.5
Si O ₂	60.1
Fe ₂ O ₃	3.6
Ca O.....	0.7
Mg O.....	2.8

Total 100.2%

The more active Japanese clays were said to be equivalent to American "Filtrol" type.

A visit was made to the Nippon Seiro K. K., Tokuyama plant, which had been reported as processing shale oil, and it was found that this plant was established in 1929 by the South Manchurian Railway Co. to produce several grades of refined paraffin wax from crude pressed wax obtained from the shale oil installations at FUSHUN, Manchuria, (Enclosure (G)). Imports of the crude pressed wax had been cut off in the middle of 1945, and no information was available to the plant personnel of the status of Fushun operations. A copy of a Japanese book entitled "Shale Oil", written by Mr. Koki ISHIBASHI of the Research Department of the Fushun Coal Manufacturing Co. of the South Manchurian Railway Co. and published in 1940, was obtained and forwarded through ATIS to the Washington Document Center (refer to ATIS Number 4602, Document Number ND26-0031). This book contains some description of the shale mining, retorting and refining operations at FUSHUN. It was stated by executives of the Nippon Seiro K. K. that no shale mining or retorting industry, other than that at FUSHUN, existed in the Japanese Empire.

Enclosure (A) lists the titles of Japanese reports pertaining to miscellaneous investigations on petroleum and other subjects at the First Naval Fuel Depot, OFUNA, which were forwarded through ATIS to the Washington Document Center.



RESTRICTED

ENCLOSURE (A)

ENCLOSURE (A)

LIST OF DOCUMENTS
IN JAPANESE PERTAINING TO RESEARCH
ON MISCELLANEOUS OIL TECHNOLOGY
AT THE FIRST NAVAL FUEL DEPOT, OFUNA

ENCLOSURE (A)

LIST OF DOCUMENTS IN JAPANESE PERTAINING TO
RESEARCH ON MISCELLANEOUS OIL TECHNOLOGY
AT THE FIRST NAVAL FUEL DEPOT, OFUNA.

(Forwarded through ATIS to the Washington Document Center
Refer to ATIS No. 4585 and NavTechJap Document No. listed below)

<u>NavTechJap No.</u>	<u>Title</u>	<u>Author</u>	<u>Year</u>
ND26-0014.1	On Polymerization.	S. KOMATSU	Dec. 1926.
14.2	On the Quantitative Testing Method of Carbon.	Y. FURUYA	Apr. 1927.
14.3	Experiments on the Prevention of Corrosion of Oil Tank and Pipe in Oil Tanker.	M. AKITA Y. FURUYA	Aug. 1927.
14.4	Manufacture of Petrolene from Petroleum Pitch.	R. EEPFU K. MIYATA	Dec. 1927.
14.5	On the Quantitative Testing Method of Sulphur in Lighter Oil.	Y. FURUYA	Nov. 1927.
14.6	Petroleum Resources in Japan.	Z. TOKUDA	Dec. 1927.
14.7	Preservation and Transportation of Petroleum and Its Products.	S. NOMIYA	Dec. 1927.
14.8	To the Fuel Investigator.	S. KOMATSU	Feb. 1928.
14.9	Petroleum Refining.	H. KOYAMA	Aug. 1928.
14.10	Petroleum Industries in Japan.	R. FUKUDA	Nov. 1928.
14.11	Melting Point Testing Method of Tar Pitches.	T. OGAWA S. SUMIMOTO F. NEMOTO	July, 1929.
14.12	Thermodynamics.	S. HORIBA	Aug. 1929.
14.13	Studies on the Tar Acid.	R. NAKAI	Dec. 1929.
14.14	Thermal Cracking of Tar Acid.	R. NAKAI	Aug. 1930.
14.15	Organic Chemistry.	R. NOTSU	Mar. 1931.
14.16	On the Progress and Its Future of Soda Industry.	T. YUASE	Apr. 1931.
14.17	Studies on the Petrolene.	T. YOKOTA	July, 1931.
14.18	Studies on Tar Pitch. Part II.	S. SUMIMOTO F. NEMOTO	Sept. 1931.
14.19	Studies on Tar Pitch. Part III.	S. SUMIMOTO F. NEMOTO	Oct 1931.

ENCLOSURE (A)

<u>Nav. Tech. Rep. No.</u>	<u>Title</u>	<u>Author</u>	<u>Year</u>
ND26-0014.20	Chemistry of Organic Higher Molecular Weight Compound.	R. NOTSU	Mar. 1932.
14.21	Studies on the Analytical Method of Gas. Part I.	S. YAMAGUCHI S. KITAMURA	Jan. 1933.
14.22	Studies on the Analytical Method of Gas. Part II.	S. YAMAGUCHI S. KITAMURA	Apr. 1933.
14.23	Qualitative and Quantitative Analysis of Sulphur in Petroleum.	M. AKITA	Feb. 1934.
14.24	Analysis of Japanese Crude Oil.	I. WATANABE	July, 1934.
14.25	Combustion of Carbon Monoxide	K. ISOGAI S. KANEDA	Oct. 1934.
14.26	Cracking of Hexahydrocresol.	H. FUJIMOTO	Sept. 1935.
14.27	Properties of Ecuador Crude Oil.	M. AKITA I. KAGEHIRA H. SHINOYAMA H. KONISHI S. SAKAMOTO T. WADA	Oct. 1935.
14.28	National Defense and Fuel.	H. YAMANAKA	Dec. 1935.
14.29	Manure Industry in Germany.	K. ANDO	Apr. 1936.
14.30	Petroleum Conditions in Italy.	T. NAMIKAWA T. TAKEI	May, 1936.
14.31	Problem on the Persian Oil Fields.	T. NAMIKAWA T. TAKEI	July, 1936.
14.32	Oil Industries in Rumania and Iraq.	T. NAMIKAWA T. TAKEI	July, 1936.
14.33	Petroleum Resources in India and Venezuela.	T. NAMIKAWA T. TAKEI	Sept. 1936.
14.34	Fish Oils and Hardening Oil Industry.	T. NAMIKAWA	Oct. 1937.
14.35	Chemical Engineering and Chemical Industry.	S. UCHIDA	Feb. 1938.
14.36	On the Catalytic Action of Metallic Oxide in the Cracking of Petroleum Kerosene.	A. YAMAOKA	Dec. 1941.
14.37	On the Experiment of the Mechanism of Extraction. Part I.	S. SANKA	Jan. 1943.

ENCLOSURE (A)

<u>NavTechJap No.</u>	<u>Title</u>	<u>Author</u>	<u>Year</u>
ND26-0014.38	On Japanese Acid Clay.	K. MITSUI T. IIZIMA	Mar. 1943.
14.39	On the Investigation of an Alloy which Resists to 5% Phosphoric Acid Vapour. (1-2)	T. SATO	May, 1943.
14.40	Anti-Corrosive Properties of a Low-Chromium-Alloy for the Action of a Mixed Vapor of H ₂ S and Steam.	T. SATO	Dec. 1943.
14.41	Density of Gas Oils at Low Temperature.	N. ISOGAI	Dec. 1943.
14.42	Rubber Resources in the South Asia-District.	I. KAGEHIRA T. ISHIWATA S. INABA	May, 1944.
14.43	Fatty Oil Resources in the South Asia-District.	I. KAGEHIRA T. DAN	July, 1944.
14.44	Attitude for Researchers.	S. KOMATSU	Dec. 1944.

RESTRICTED

ENCLOSURE (B)

ENCLOSURE (B) 1

STUDIES ON THE ACID CLAY

by

CHEMICAL ENGINEERING COMMANDER
H. FUJIMOTO

Research Period: 1940-1942

Prepared for and Reviewed with Authors
by U. S. Naval Technical Mission to Japan

December 1945

ENCLOSURE (B)₁LIST OF TABLES
AND ILLUSTRATIONS

Figure 1(B)1	Apparatus for Testing Acid Clay Catalysts.....	Page 19
Table I(B)1	Composition, Source, and Catalytic Effect of Typical Clays.....	Page 19
Table II(B)1	Effect of Acid Treatment on Typical Clays on Composition and Activity.....	Page 20

ENCLOSURE (B)1

SUMMARY

It was the object of this research to find 1) the most reactive clay catalyst in our territory for use in catalytic cracking and 2) the most desirable conditions for activation. Typical clays produced in Japan were tested for effectiveness in catalytic cracking, and acid clay gave the most satisfactory results. The activating conditions of several acid clays selected from various sources were studied, using sulphuric acid as activating agent. An acid clay produced near KOMATSU City, ISHIKAWA Prefecture gave the best result. This catalyst was used in the commercial plant at YOKKAICHI, Second Naval Fuel Depot.

I. INTRODUCTION

This research was carried out from December, 1940 to August, 1942 in cooperation with Takeda Acid Clay Co. Ltd. at MIZUSAWA, YAMAGATA Prefecture, by Lt. Eng. M. MURAKAMI and Assist. Eng. M. MIYASAKI and Eng. J. ISHIKAWA, the latter being a representative of the company.

II. DETAILED DESCRIPTIONA. Apparatus

The activity test apparatus for catalytic cracking catalysts is shown in Figure 1(B)1.

B. Procedure

A definite kerosene fraction of Midway crude oil was selected for use in the catalyst activity tests.

150cc of the oil was passed into the reaction tube containing 100cc of catalyst, at a constant rate for 1.5 hours (space velocity 1.0). Reaction conditions were held constantly at a temperature of 450°C, and atmospheric pressure for all catalyst samples. Reaction products were distilled using an Engler distillation flask, and fractions boiling up to 150°C were collected and measured. The catalyst activity of our experiments were compared by the term "decomposition rate", which is expressed as the volume % of the fraction boiling up to 150°C to the initial charge. Analyses of the composition and activity of many kinds of natural clays produced in Japan were made.

C. Results

Results of typical tests are shown in Table I(B)1. These results show that acid clays give higher activity than the others and that there is no linear relation between activity and composition.

Several samples of acid clays were activated by boiling for 3 hours with sulphuric acid, having concentrations varying from 1 N to 7.5N, so that changes of activity and chemical components could be observed. The experimental results were as tabulated in Table II(B)1.

III. CONCLUSIONS

From these results it was concluded as follows:

ENCLOSURE (B)1

The activity of acid clay is not dependent on its chemical components, but on its origin.

It seems, in general, that the clays which have high content of MgO show high activity.

The molecular ratio $\text{SiO}_2/\text{Al}_2\text{O}_3$ of the activated acid clays showing maximum activity appears generally to be approximately 6.

Best normality of H_2SO_4 for activating acid clays for use as catalysts lies between 1 to 4.5 N as compared to that of activating acid clays for use as adsorbent in decolouring petroleum or fatty oils, which varies from 3 to 6 N H_2SO_4 according to the origin of the clay.

The acid clay produced near KOMATSU City was activated by Takeda Acid Clay Co., Ltd., and has been used in the commercial catalytic cracking plant at the Second Naval Fuel Depot.

It is also suitable for use in the catalytic reforming of pine root oil. Experimental results for this application are given in another report.

ENCLOSURE (B)₁

Table I(B)₁
COMPOSITION, SOURCE, AND CATALYTIC EFFECT OF TYPICAL CLAYS

Name of Clay	Source	Ign. loss	SiO ₂	Component							Activity Test	
				Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	CaO	MgO	SO ₃	P ₂ O ₅	Decomposition rate (%)	
Diatomaceous Earth	FUDOSAN, Ureshino Saga Prefecture	9.5	72.8	11.7	3.7	0.9	0.7	trace	0.6	trace	4.4	
Bentnite	SHIRIKUNIYUNANAI, Hokkaido	5.0	74.8	13.9	2.5	trace	2.3	2.2	0.2	0.1	6.5	
Acid clay	KOMATSU City, Ishikawa Prefecture	11.0	61.8	21.7	2.1		0.7	4.3			16.7	
Acid clay	NAKAJO, Niigata Prefecture	7.1	74.0	14.0	2.0	0.0	1.8	2.8	0.0	0.0	10.0	
Acid clay	MIZUSAWA, Yamagata Prefecture	11.1	73.2	17.1	0.1	trace	0.1	1.8	0.1	trace	11.9	
Kaoline	HOKUTO, Taihoku City, Formosa	14.9	42.6	39.1	4.2	0.6	0.7	0.2	2.5	0.0	1.4	

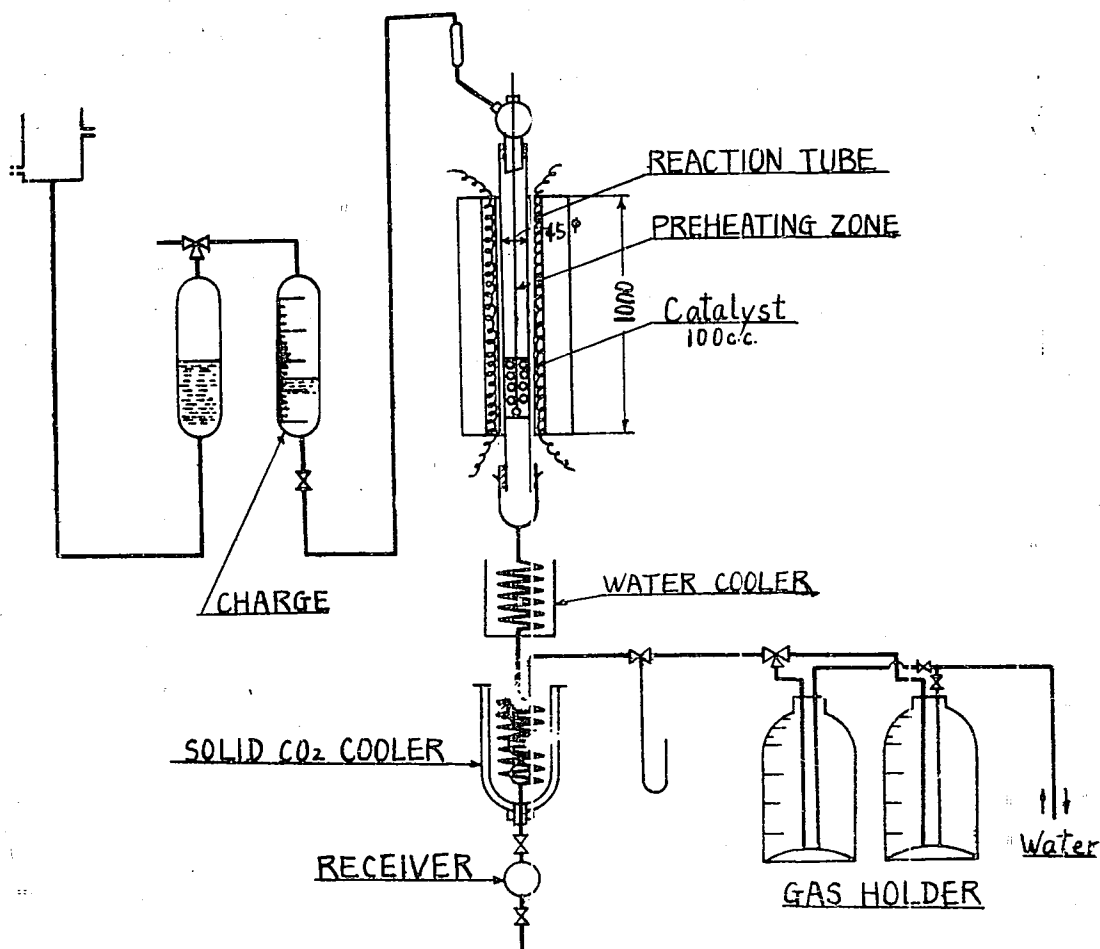


Figure 1(B)₁
APPARATUS FOR TESTING ACID CLAY CATALYSTS

ENCLOSURE (B)₁Table II(B)₁

EFFECT OF ACID TREATMENT ON TYPICAL CLAYS ON COMPOSITION AND ACTIVITY

	Conc of H ₂ SO ₄ for activation	Component						Activity test	
		ign loss	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	MgO	Decomposition rate (%)	
Acid Clay from IZU	non treated	12.3	29.0	50.7	5.1	0.3	2.9	9.6	
	1 N	12.0	28.8	52.2	4.9	0.3	2.6	12.0	
	2 N	11.4	25.7	55.4	4.6	0.1	2.5	14.3	
	3 N	11.1	22.6	60.6	4.1	0.2	1.3	17.4	
	4.5 N	9.0	15.8	70.7	3.7	0.1	1.0	18.2	
	6 N	8.6	14.0	74.2	1.3	0.1	0.8	13.5	
	7.5 N	7.4	8.8	81.2	1.3	0.1	0.5		
Acid Clay from MIZUSAWA	non treated	8.4	24.5	59.1	4.0	0.7	2.7	8.8	
	1 N	8.5	24.5	60.1	3.6	0.7	2.8	11.6	
	2 N	8.1	20.7	66.1	2.9	0.8	2.5	15.5	
	3 N	7.2	17.2	68.3	3.1	0.8	2.5	14.7	
	4.5 N	6.4	11.3	75.7	2.3	0.6	1.7	9.6	
	6 N	5.2	7.4	81.3	1.5	0.7	1.1		
Acid Clay from KOMATSU	non treated	11.0	21.7	61.8	2.1	0.7	4.3	16.7	
	1 N	7.0	17.9	70.3	1.4	0.6	3.9	17.7	
	2 N	6.9	16.2	70.9	1.2	0.6	3.5	20.3	
	3 N	6.5	13.9	76.0	1.1	0.5	2.3	13.4	
	4.5 N	5.4	10.1	82.4	0.7	0.5	1.7	17.0	
	6 N	4.2	3.3	91.0	0.7	0.5	0.7	12.4	
	7.5 N	5.2	0.8	92.7	0.4	0.4	0.3		
Acid Clay from SONCHOYAMA	non treated	10.3	21.2	58.7	4.1	1.4	4.8	16.1	
	1 N	8.8	20.3	61.2	3.1	0.5	4.6	17.8	
	2 N	8.1	20.3	64.6	2.5	0.4	4.0	22.0	
	3 N	7.5	16.7	70.0	2.2	0.3	3.0	15.3	
	4.5 N	6.9	14.2	74.2	1.8	0.4	2.5	13.4	
	6 N	6.8	7.1	82.1	1.3	0.3	1.1	7.4	
	7.5 N	6.8	4.1	86.3	0.7	0.2	0.4		
Acid Clay from NAKAJO	non treated	9.4	20.1	64.1	2.4	0.6	3.5	17.5	
	1 N	7.8	19.2	66.3	2.2	0.4	3.4	21.2	
	2 N	7.2	18.7	66.9	2.0	0.4	3.3	19.3	
	3 N	6.9	13.7	72.1	1.9	0.4	2.9	16.4	
	4.5 N	5.3	7.4	82.4	1.3	0.3	2.9	9.7	
	6 N	3.8	7.0	87.1	0.3	0.3	1.4	8.2	
	7.5 N	3.7	2.1	92.4	0.3	0.3	1.1		
Fentnite from YAMAGATA	non treated	5.1	20.2	67.4	3.6	1.7	2.4	14.6	
	1 N	6.1	20.0	69.3	2.6	0.7	1.4	14.0	
	2 N	6.1	19.9	70.1	2.3	0.6	1.2	14.6	
	3 N	6.4	19.3	71.1	2.1	0.6	1.1	14.0	
	4.5 N	6.2	16.6	73.4	2.0	0.6	1.0	13.1	
	6 N	5.9	13.3	76.5	2.0	0.5	0.9		
	7.5 N	4.7	10.7	79.2	1.6	0.5	0.8		
Acid Clay from MATSUNE	non treated	5.7	13.1	75.8	1.3	1.6	3.5	12.3	
	1 N	5.7	12.6	76.7	1.3	0.4	2.6	16.5	
	2 N	5.0	11.6	78.7	1.2	0.4	2.3	14.2	
	3 N	5.1	10.9	80.0	1.0	0.4	2.1	14.3	
	4.5 N	4.3	9.6	83.2	1.0	0.3	1.6	12.7	
	6 N	4.2	5.2	88.9	0.5	0.3	0.8	10.6	
	7.5 N	3.3	2.3	92.5	0.1	0.2	0.4		

ENCLOSURE (B) 2

ENTRAINMENT AND PLATE EFFICIENCY
OF BUBBLE-CAP RECTIFYING COLUMNS

by

CHEMICAL ENGINEERING LIEUTENANT
T. YOKOYAMA

Research Period: January 1944-April 1945

Prepared for and Reviewed with Authors
by U. S. Naval Technical Mission to Japan

December 1945

ENCLOSURE (B)2

LIST OF TABLES
AND ILLUSTRATIONS

Table I(H)2	Properties of Distillation Substances	Page 27
Figure 1(H)2	Chart for Determining Constant	Page 28
Figure 2(H)2	Diagram of Distillation Test Apparatus	Page 29
Figure 3(H)2	Entrainment vs Vapor Mass Velocity	Page 30
Figure 4(H)2	Vapor Velocity vs Pressure Drop	Page 31
Figure 5(H)2	Distillation Apparatus for Process Engineering	Page 31

ENCLOSURE (B)2

I. INTRODUCTION

A. History of Project

There have been many investigations of the plate efficiency of bubble-cap fractionating towers, but the information on allowable vapor velocity is still incomplete. Even today, large scale commercial towers are operating with relatively low vapor velocities. After review of the information on this subject, it was concluded that the main factors controlling the vapor velocity are entrainment and pressure drop across the bubble plate.

In recent years, the importance of entrainment as a factor in determining the allowable vapor velocity has come to be more fully recognized. Sherwood and Jenny reported experimental data for the water-air system, and pointed out that the entrainment for this system is substantially negligible until vapor velocity of 1.2 meters per second is reached. This conclusion cannot be directly applied to a petroleum system since, the physical and chemical properties of water, steam, and air are quite different from those of petroleum. Particularly, the specific gravity of water is greater than that of petroleum, and, on the other hand, the specific gravity of oil vapor is greater than that of air or steam. In consequence, the suspending velocities of oil particles are higher than for water particles. Experimental data are needed on entrainment during actual distillation. The work presented here represents an effort to clear these points with a view to raising the capacity of bubble towers.

B. Key Research Personnel Working on the Project

Chem. Eng. Lieut. T. YOKOYAMA

Chem. Eng. Sub. Lieut. H. KINGO

Chem. Eng. Sub. Lieut. I. KANISE

II. DETAILED DESCRIPTION

A. Description of Apparatus

The apparatus used in this work is shown in Figure 1(B)2. This shows the apparatus used for the fundamental experiments, and for these experiments two columns were used. One is a small laboratory column with three single-cap plates. In designing this column, much care was taken to provide more severe conditions for entrainment than ordinarily exist in actual columns.

On the basis of data on the dimensions of commercial towers in this country and information on structural factors affecting entrainment reported by previous investigators, a plate spacing of 25 centimeters (minimum size for petroleum fractionator), and a slot area per plate section of about 4% were adopted for the small laboratory column. Liquid submergence (distance between top of slot and liquid level), is variable up to a maximum of 2.5 centimeters. Another semi-commercial column was devised to simulate an actual column as closely as possible. This could be attached to the same still-put and condenser. Sufficient heating and condensing surface were provided to permit running at high vapor velocities. Most previous investigators adopted only two plates, and calculated the entrainment from the analysis of liquid on the upper plate, and liquid drops which were carried away from the upper plate by vapor into the condenser were neglected. In our apparatus, however, all entrainment from the middle plate was caught by the upper plate, and entrainment data

ENCLOSURE (B)2

were corrected for the same. Only data with good material balances were utilized in correlations. In order to withdraw a definite volume of sample quickly, a special calibrated sampling pipette was provided on each plate. A sight glass, hand hole, and level gauge were also provided on each plate. Due to the nature of this investigation, downflow pipes were not installed.

The columns, still hot, and vapor lines were insulated with asbestos covering.

B. Test Procedures

In order to find the characteristic properties of the apparatus with regard to previous investigations, entrainment and pressure drop were determined for the water-steam system. At first, to preheat the column, water was added to the still-pot from the feed tank, and brought to boiling by heating with steam.

The vapor flowed up the column, condensed on the wall, and accumulated on each plate. When the temperature of the top of column reached 100°C, the heating steam valve was closed, and the condensed water on each plate was withdrawn quickly through the three-way-cock of the level gauge. Next, a definite volume (1000cc) of 0.1 N NaOH solution was introduced on the lower plate and same volumes of distilled water were introduced on the middle and upper plates. These liquids also had been preheated to their boiling points (a successful test demanded much care in preparation). Then the still was carefully heated until the vapor velocity reached the desired value. When a steady state was obtained, liquid samples were withdrawn from each plate simultaneously through sampling pipettes, and after maintaining the steady state for a definite time, samples were again withdrawn as before. The content of NaOH on each plate was determined by titration with standard sulphuric acid, and a NaOH material balance was calculated.

The data were utilized only when the reduction of NaOH on the lower plate agreed well with the increase on the middle and upper plates. Entrainment was calculated by the same method as described by Holbrook and Baker or Sherwood and Jenny. Vapor velocity through the column was calculated from the flowmeter reading and the volume of condensate.

The next determinations were made on a narrow cut aviation gasoline-gasoline vapor system, and finally on the toluol-toluol vapor system.

Inspections on these substances are given in Table I(B)2. In the table, it is seen that the properties of the water-air system and hot water-steam system are similar and the gasoline-gasoline vapor system and toluol-toluol vapor system are also similar. For the hydrocarbon systems, analyses were made as follows. A definite volume of dyed solution was fed on the lower plate and the same volume of distilled water was fed on the middle and upper plates. To the liquid on the lower plate was added a definite volume of non-volatile dye, and the concentration of dye on the middle and upper plate caused by entrainment during the process was estimated from the color determined with a colorimeter.

To investigate other variables effecting plate efficiency, and to determine over all plate efficiencies, a larger continuous rectification pilot plant column was constructed. Experimental work was done with this column on the methanol-water system and benzol-toluol system, but since the apparatus lacked an automatic controller, steady state could not be reached and satisfactory material balances were not obtained. Consequently, these data were discarded and it was planned to modify the

ENCLOSURE (B)2

apparatus. This could not be realized, however, because of circumstances caused by the war.

C. Experimental Results

1. Entrainment. The experimental data are shown graphically in Figure 2(B)2 and are compared with data reported by previous investigators. It is seen that the data for the hot water-steam system correlate well with the water-air data of Sherwood and Jenny obtained on a single-cap laboratory column with column diameter of 25 centimeters and variable plate spacing ranging from 31 to 61.5 centimeters. If Sherwood and Jenny's data are extrapolated to a plate spacing of 25 centimeters, they coincide with the author's. This is to be expected, since the internal structures of both columns are similar, as well as the physical and chemical properties for both systems. By this comparison, the characteristics of the test apparatus, and confidence in the experimental procedure, were established.

Investigation of the hydrocarbon systems were made on the same apparatus. At first, a narrow cut fraction of aviation gasoline, 110°C-130°C was used. However, the investigation of this complex mixture was unsuccessful because the moles of vaporization and condensation at each plate were not equal, resulting in flooding. Therefore, it was decided to investigate the toluol-toluol vapor system, which has properties similar to the aviation gasoline fraction, as shown in Table I(B)2. As a result of this investigation, entrainment for the toluol-toluol vapor system was found to be unexpectedly low as compared with the water-steam system.

2. Pressure Drop. The pressure drop data are shown in Figure 3(B)2 in which the pressure drop across one plate in millimeters of mercury is plotted against vapor velocity through the column. On the basis of Figure 3(B)2 it is calculated that a vapor velocity of 1.0 meter per second can be safely used in a commercial petroleum column (atmospheric pressure) without exceeding the flooding point.

3. Discussion of Results. With regard to the fact that the entrainment ratio for the toluol-toluol vapor system is unexpectedly low, the authors explain this as follows:

If the data are plotted against linear velocity, the curves for both systems fall more closely together than when plotted against mass velocity. However even when plotted against linear velocity, entrainment for the hydrocarbon system is far lower than the water-steam system. In reviewing the factors affecting entrainment, especially surface tension and densities of liquid and vapor (refer to Table I(B)2) the effect of surface tension appears to be more controlling than density difference between liquid and vapor, and the smaller the surface tension, the smaller the entrainment ratio. When it is considered that mercury, which has a high surface tension, is easily scattered into fine particles, whereas ships at sea often pump oil (with low surface tension) to calm the waves, it can be appreciated that the phenomenon of entrainment is much affected by the tendency to form liquid particles. Most fractionating columns have been designed by the equation of Souder and Brown, but in this equation the effect of surface tension is not considered as a direct variable.

ENCLOSURE (B)2

Souder-Brown's equation for allowable vapor velocity

$$W = C \sqrt{D_2(d_1 - d_2)}$$

where W = mass velocity (Kg/m² hr)
 d_1 = density of liquid (Kg/m³)
 d_2 = density of vapor (Kg/m³)
 C = constant affected by surface tension and plate spacing determined from Figure 1(B)2.

III. CONCLUSION

It is shown by this investigation that, for a hydrocarbon system, entrainment is less than for the water-air system. Sherwood and Jenny pointed out, as the result of their experiments, that efficiency is not substantially reduced by entrainment until an entrainment ratio 0.1 is reached, and that this ratio would not be reached at ordinary vapor velocities in an actual column. As can be seen from Figure 2(B)2, for the small laboratory column an entrainment ratio 0.1 is reached at a vapor velocity of about 1.0 meter per second. In the 40 centimeters semi-commercial column the entrainment ratio is about 0.01 for the same vapor velocity.

In a large scale commercial column with plate spacing of 50 centimeters or over, allowable vapor velocity of 1.2 meters per second, at least, can be safely used from the standpoint of entrainment and pressure drop across the plate.

Bibliography

- | | |
|---|--|
| Sherwood and Jenny: | Ind. Eng. Chem. <u>27</u> 265 (1935) |
| E. M. Baker: | Ind. Eng. Chem. <u>31</u> 717 (1939) |
| Holbrook and Baker: | Ind. Eng. Chem. <u>26</u> 1063 (1934) |
| Souder and Brown: | Ind. Eng. Chem. <u>26</u> 98 (1934) |
| C. C. Peavy and E.M. Baker: | Trans. Amer. Inst. Chem. Eng. <u>33</u> 315 (1937) |
| Pyott, Jackson: | Ind. Eng. Chem. <u>26</u> 1068 |
| Ashraf. Cubbage, and Huntington: | Ind. Eng. Chem. <u>26</u> 1068 (1934) |
| Kirshbaum: "Distillier und Rektifizierteknik" | (1940) |

ENCLOSURE (B)2

Table I(E)2
 PROPERTIES OF DISTILLATION SUBSTANCES

Substance	Molecular weight (g.m.)	Specific gravity		Surface tension (dyne/cm)	B.P. (°C)
		liquid	vapor		
92 Octane Av. Gas 110-130°C	ca.100	ca.0.733	0.0027 (100°C)	19.0	125 (50% pt.)
Toluol	92	0.796 (111°C)	0.0029 (111°C)	19.9 (111°C)	111
Water	18	0.985 (100°C)	0.0059 (100°C)	61.5 (100°C)	100
Air	29		0.0013 (room temp.)		
System	Difference of Density between liquid & vapor	Surface Tension (dyne/cm)	Viscosity(c.p.)		
			liquid	vapor	
Water-air	0.999	72.1 (25°C)	0.0185 (25°C)	0.895 (25°C)	
Water-steam	0.957	61.5 (100°C)	0.013 (100°C)	0.284 (100°C)	
92 Octane aviation gasoline	0.730	19.0 (ca.100°C)	0.011-0.020 (100°C)	0.20-0.29 (100°C)	
Toluol- Toluol vapor	0.793	19.9 (110°C)	0.025 (110°C)	0.25 (110°C)	

ENCLOSURE (E)2

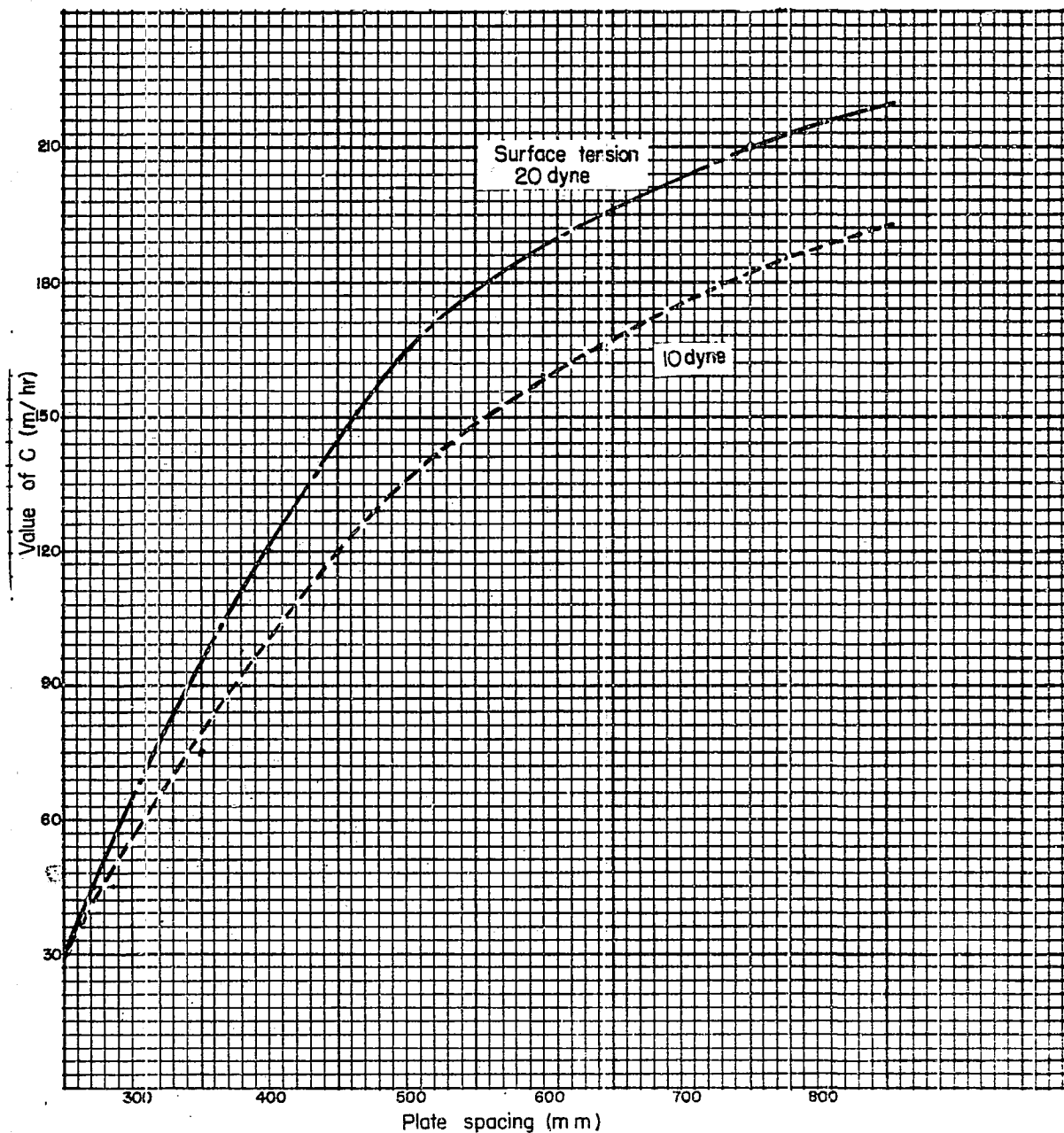


Figure 1 (E)2
CHART FOR DETERMINING CONSTANT

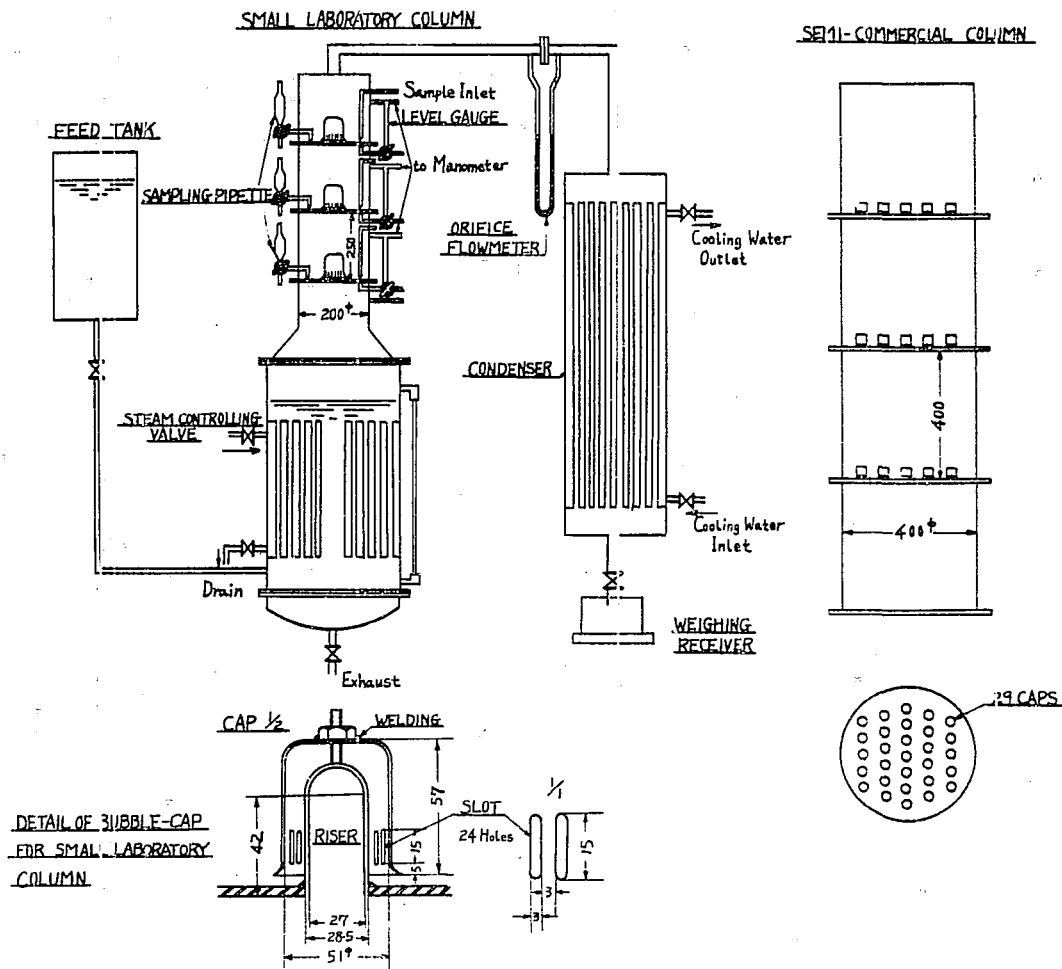


Figure 2 (B)2
DIAGRAM OF DISTILLATION TEST APPARATUS

ENCLOSURE (B)2

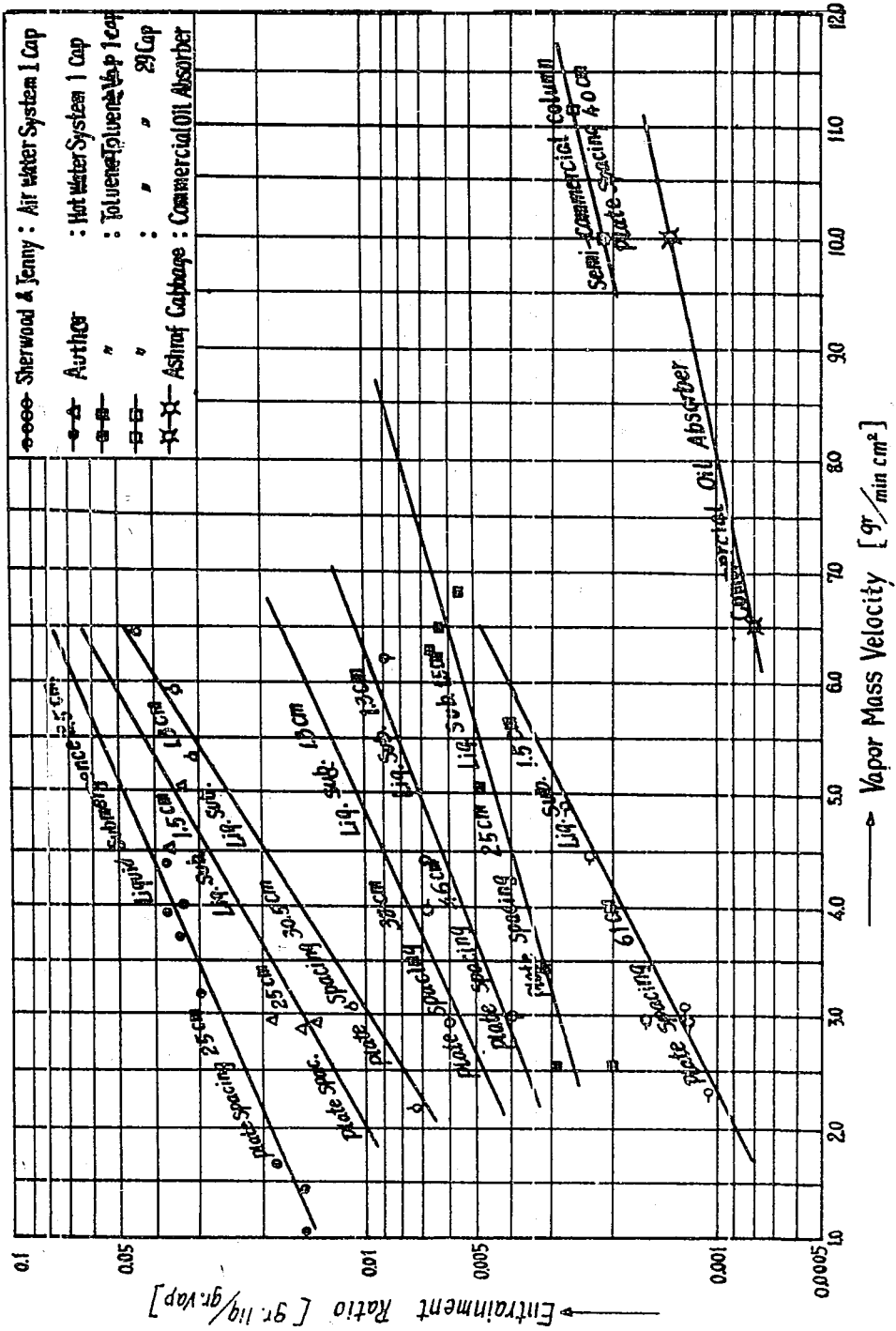


Figure 3 (B)2
ENTRAINMENT vs VAPOR MASS VELOCITY

ENCLOSURE (B)2



Figure 4 (B)2
VAPOR VELOCITY vs PRESS. DROP

ENCLOSURE (B)2

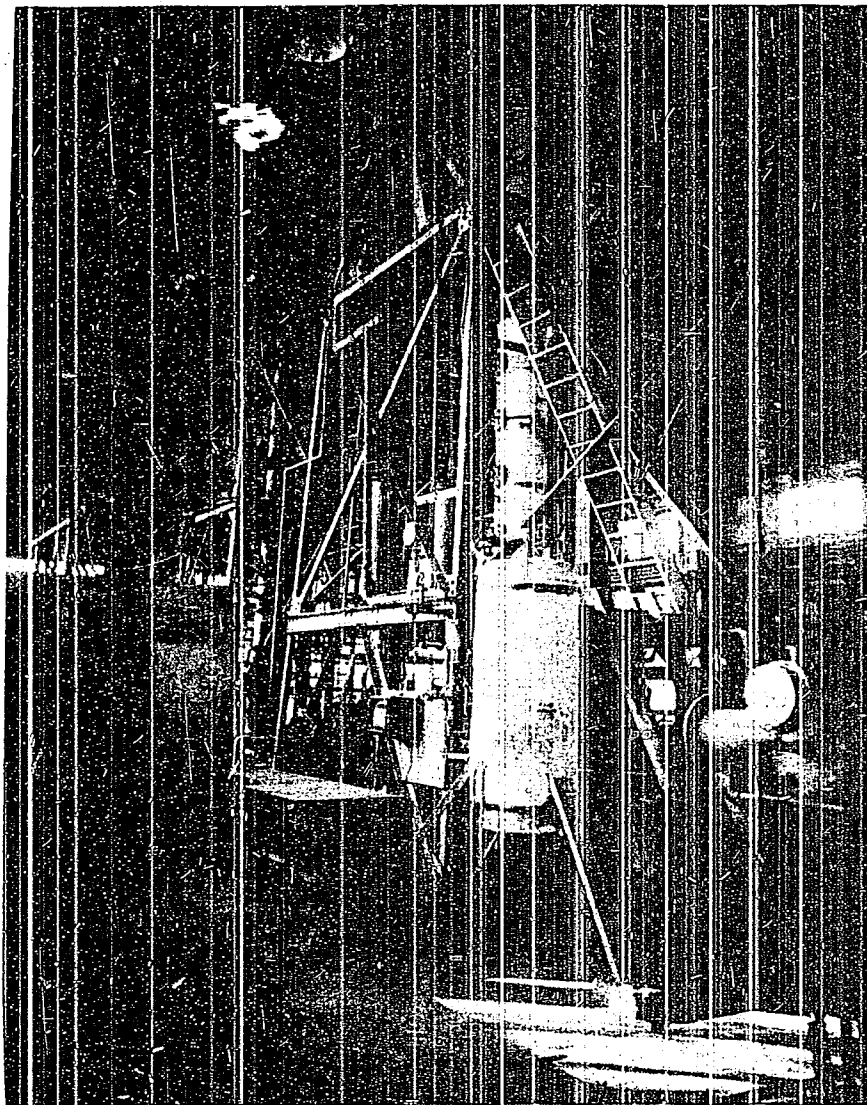


Figure 5 (B)2
DISTILLATION APPARATUS FOR PROCESS ENGINEERING

RESTRICTED

ENCLOSURE (C)

ENCLOSURE (C)

DEVELOPMENT OF CATALYTIC CRACKING
IN JAPAN

BY
N. NAKAHARA
PRESIDENT OF TOA NENRYO K.K.

ENCLOSURE (G)

TABLE OF CONTENTS

The Report	Page 35
Appendix I Catalytic Cracking Tests of the Nippon Oil Co.	Page 39
Appendix II List of Papers Obtained Dealing With Catalytic Cracking Research in Japan.....	Page 41

LIST OF TABLES
AND ILLUSTRATIONS

Figure 1(C) Rough Sketch of Inner Construction of Toa Nenryo Type Reaction Tower	Page 37
Figure 2(C) Flow Chart of Cooling System, Toa Nenryo Type Reaction Chamber	Page 38

ENCLOSURE (C)

The study of catalytic cracking of kerosene and gas oil was first started by Ryonosuke KOBAYASHI and the late Haru KAJIMOTO under the direction of Prof. TANAKA of the Imperial Japanese University of TOKYO in 1934. In 1937, Ryonosuke KOBAYASHI, with the assistance of Yuzo KOGA (Ogura Oil Co.) and Tohei TAKAYAMA, (Nippon Soda Co.), continued research work but failed to make much headway as demand for aviation gasoline was not great at that time. In 1938, stimulated by report of the success of the Houdry Process in U.S.A., KOBAYASHI and WATANABE succeeded in proving that 92 octane gasoline (0.1% lead added) was obtainable by this method. From this time on study of the process was very much encouraged, and KOBAYASHI was sent to America to study the process. Upon his return, more was learned about the progress of the Houdry Process and also the fact that U. O. P. was making headway in this work. The possibility of buying the license was discussed by the Army and Navy, and study of the process was more vigorously continued by Kobayashi Research Laboratory, Ogura Oil, Nippon Soda, the Army and Navy.

In 1939, with the opening of the Army Fuel Institute, Engineers of the Ogura Oil Co., (KOGA, ISHIMARU, ISHIKAWA), designed the first catalytic cracking pilot plant under the direction of Lieut. Col. AKASHI. This plant still exists in the Fuchu Army Fuel Institute. In the fall of 1939 Toa Fuel Co. sent NAKAHARA and KOGA to the U. S. A. to purchase the Houdry patents. Lieut. Col. YANAGI of the Army and Lieut. Comdr. FUJIMOTO of the Navy also went to the U. S. A., with HORIS of the Nippon Gasoline Co., to negotiate for purchase of U. O. P. patents. In the meantime, Toa Fuel Co. (TSUKUDA et al.), confirmed that 92 octane gasoline could be obtained by catalytic cracking. All negotiations for purchase of the patents failed since the Moral Embargo was declared in the U. S. A. about that time. The necessity for completing the process in Japan became imperative and design of commercial scale units began. As a result, the Army started construction of two units in MARIFU, the Navy two units in YOKKAICHI and one in Formosa, and Toa Fuel Co. one unit in NAKAYAMA. Of these, only one unit each was completed by the Army and Navy before termination of the war.

In 1941, orders were given to the oil companies by the War Dept. and Dept. of Commerce regarding the conversion of thermal cracking units. Nippon Oil converted their cracking units at KANSAI and YOKOHAMA to catalytic cracking, and these were in operation before the termination of war. (The Kansai unit was first to be converted and started operating in 1943). Showa Oil converted their Schwartz units to catalytic cracking and started operations before August, 1945. Nippon Soda continued studies at its subsidiary, Koa Oil, but the work was mostly centered on reforming and did not progress very far. One of the two catalytic cracking units in the Army Refinery started operation in the summer of 1943, and one of Navy's Yokkaichi plants also started about the same time. All the Army and Navy units had automatic circulation of gas, oil, air and steam, but the Nippon Oil unit had to be switched by hand. None of these units was equipped with the molten salt, or other method, of absorbing reaction heat.

The difference between the Toa Fuel Co. and Army and Navy units was that in the former the absorption of reaction heat at the time of regeneration of catalyst was effected by means of water vapor. As to the number of the reaction towers, the Army and Navy plants were equipped with three units per one apparatus, or a multiple thereof; i.e. reaction time was equal to regeneration time, while Toa Nenryo had four units or a multiple; i.e., the regeneration time was twice the reaction time. The interior construction of all the reaction towers was similar; i.e., a cylindrical reaction tower equipped with a certain number of perforated longitudinal pipes, some of which were used as oil vapor inlet pipes and others as oil vapor outlet pipes. The oil vapor inlet pipe was a single pipe for the Army and Navy, and a double pipe for Toa Nenryo. (Refer to Figure 1(C)).

ENCLOSURE (C)

The results of Nippon Oil's test run are shown in Appendix I. The Army obtained a somewhat higher aviation gasoline yield, but the octane value was between 90-91 (0.184 Ethyl Fluid). This was caused mainly by the difference between the automatic and hand worked method of switching circulation, and also due to the fact that in the reaction tower, the holes of the oil vapor inlet nozzle were arranged in uniform pitch in the Nippon Oil design, while in Army design, the holes were arranged in non-uniform pitch with a smaller number towards the base of the reaction tower, thereby causing more uniform distribution of oil vapor in the reaction tower.

The Army installed a pilot plant at FUCHU for research work, and the following points were mainly studied: (1) design of the oil vapor inlet nozzle, (2) catalyst studies: a mixture of active acid clays obtained from the acid clays produced at NISHIKANBARA District, Niigata Prefecture, and in the neighborhood of ITOIGAWA, to which was added a small quantity of chromium oxide, showed good results. "Kanuma" earth, however, was better. In particular, activated "Kanuma" earth had a higher activity after regeneration than when fresh, and its activity was maintained for a longer time. (3) As to the reaction tower, it was clearly shown that over-heating at the time of regeneration could be prevented in a single cylindrical tower having no internal structure, instead of using a tower with cooling tubes, if five times or more of the theoretical quantity of air, required was fed in at the time of regeneration of the catalyst.

Catalytic cracking research studies were also made by the Nippon Oil and the Mitsubishi Oil Companies, but no significant results were reported. One example of Mitsubishi's research work is given in a report presented to the Army, (Refer to Appendix II). The Toa Nenryo Co. studied various catalytic cracking catalysts, the cracking of kerosenes and gas oils produced in the Southern Islands, and also reaction tower design. The data thereon are given in reports listed in Appendix I. The Navy carried on research work independently, details of which were not disclosed.

ENCLOSURE (C)

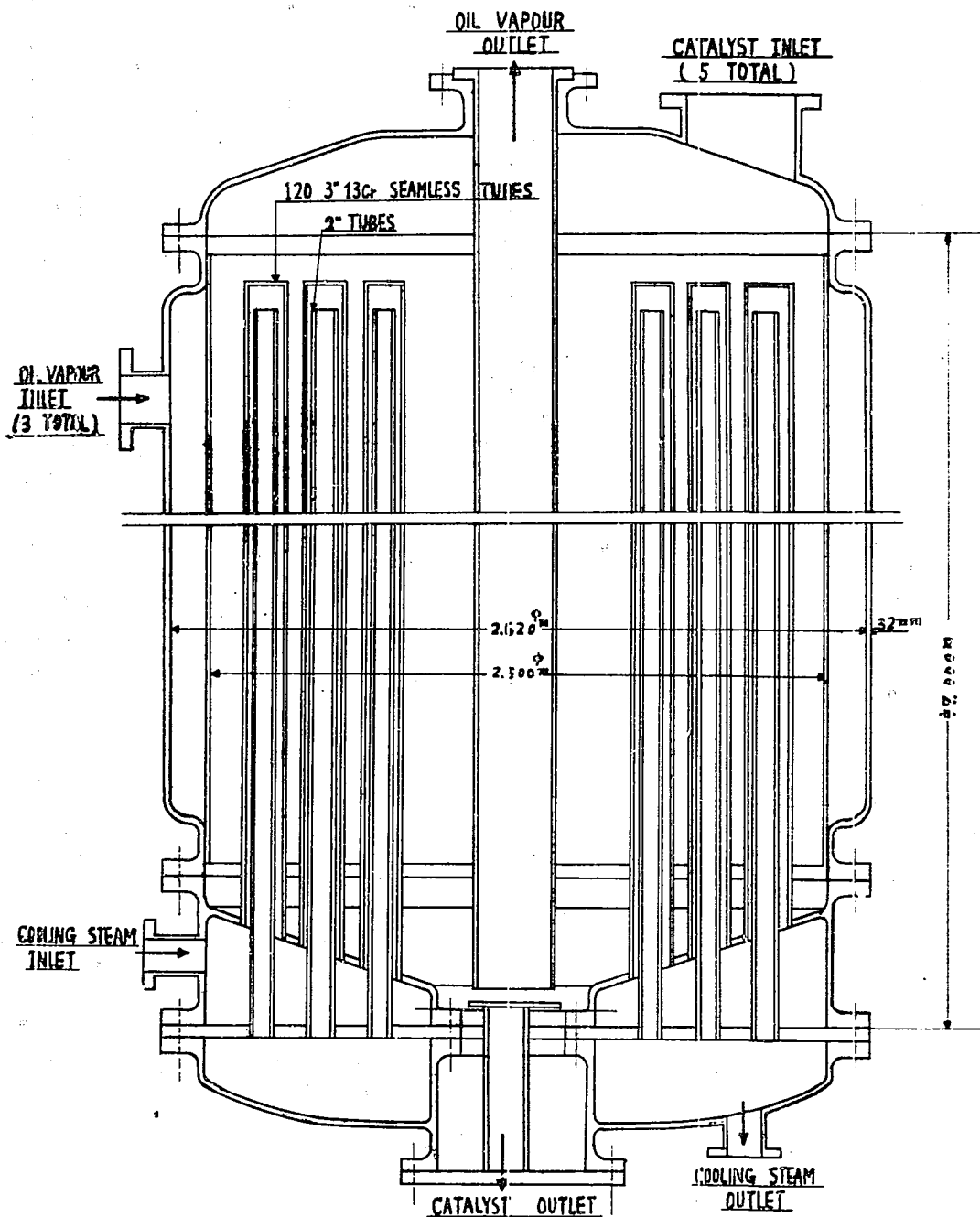


Figure 1 (C)

ROUGH SKETCH OF INNER CONSTRUCTION OF TOA NENRYO TYPE REACTION TOWER

ENCLOSURE (C)

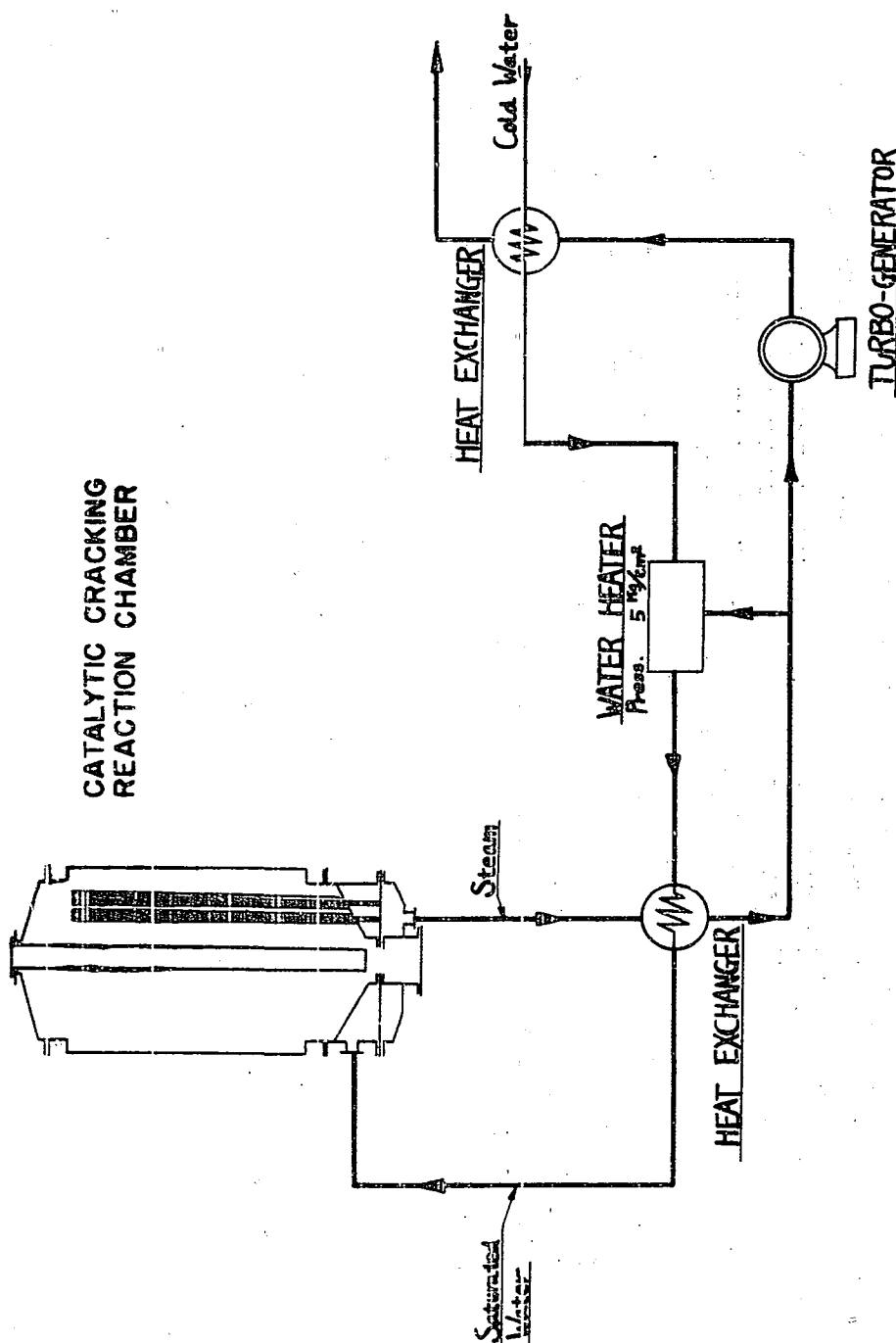


Figure 2 (C)
 FLOW CHART OF COOLING SYSTEM
 TYPE NENRYO TYPE REACTION CHAMBER

ENCLOSURE (C)

APPENDIX I

CATALYTIC CRACKING TESTS OF THE NIPPON OIL CO., LTD.
28 Nov. 1945

I. The catalytic cracking of kerosene from Sumatra crude was studied on a laboratory scale at the Tsurumi and the Kansai Refineries of the Nippon Oil Co. and at the Tsurumi Refinery on an industrial scale. The Sumatra kerosene had the following inspection:

Specific gravity.....	0.828
Distillation	
IBP.....	156°C
10%.....	212°C
97%.....	270°C
Sulphur.....	0.03%
Unsats.....	2%
Aromatics.....	12%
Naphthenes.....	0%
Paraffins.....	86%

II. Tests of Japanese Active Acid Clays

The following reaction conditions were used in the tests of the various clays:

Vol. of Catalyst.....	3.6 lit
Oil preheat Temp.....	460-470°C
Cracking Temp.....	400-450°C
Oil flow.....	60 cc/min
Space velocity.....	1.0

Test No.

Clay	<u>1</u> "A" from Takeda Clay Co.	<u>2</u> "B" from Takeda Clay Co.	<u>3</u> "D" from Nippon Clay Co.
<u>Clay Analysis</u>			
S. O ₂ wt%	67.1	69.8	68.0
Al ₂ O ₃	19.3	17.3	14.1
<u>Total Cracked Oil</u>			
Yield, %	83.2	95.0	87.5
S. G.	.821	.822	.823
<u>Aviation Dist. (below 150°C)</u>			
Yield, %	18.0	13.3	17.3
S. G.	.755	.761	.755
<u>Octane No.</u>			
Clear	77.4	76.2	76.4
0.1% lead	89.2	88.6	88.4

ENCLOSURE (C)

III. Effect of Reaction Conditions

	Test No.						
	1	2	3	4	5	6	7
Takeda Clay	C	C	C	C	A+B	A+B	A+B
Vol. of Clay, (lit)	19	19	19	19	19	19	19
Run length, (hr)	-	-	-	-	-	-	-
Oil Rate, (cc/min)	300	250	250	250	240	240	240
Preheat Temp., (°C)	500	450	440	480	480	450	440
React. Temp. (°C)	450	460	460	450	450	450	450
Space Velocity	.95	.79	.79	.79	.79	.79	.79
<u>Cracked Oil</u>							
Yield (%)	97.2	99.0	99.1	99.0	94.0	97.5	99.0
S. G.	.821	.822	.824	.826	.820	.824	.818
IBP (°C)	74	62	67	66	64	52	51
F.B.P. (°C)	287	289	280	285	285	287	283
<u>Aviation Dist.</u>							
Yield on Cracked Oil (%)	10.4	10.6	10.3	10.9	12.5	9.7	14.6
S. G.	.766	.768	.760	.763	.755	.759	.745
IBP (°C)	42	44	40	58	46	48	41
50% pt (°C)	123	118	113	122	117	124	117
95% pt (°C)	154	148	147	153	151	153	147
<u>Octane No.</u>							
Clear	76	73	75	76	78	77	77
0.1% lead	81	88	89	88	90	90	90

IV. Treatment for Improving Octane Number

1. Acid Treatment. By treating the aviation gasoline with 2% of 80% H₂SO₄ and 2% of 98% H₂SO₄ aviation gasoline of 91-91.5 (with 0.1% lead) was obtained in yield of 80-82%.

2. Catalytic Treatment. By passing 60 cc/min of cracked oil boiling below 200°C over 3.6 L. of Takeda B or C clay catalyst at a reaction temperature of 330-350°C, an aviation fuel was produced with an octane number of 92.1-92.4 (with 0.1% lead).

ENCLOSURE (C)

APPENDIX II

LIST OF PAPERS OBTAINED DEALING WITH
CATALYTIC CRACKING RESEARCH IN JAPAN

(ATIS No. 4647)

<u>NavTechRep No.</u>	<u>Title (& Co.)</u>	<u>Date</u>
ND26-0032.1	Result of Catalytic Cracking on Pilot Plant Scale. (Mitsubishi Oil Co.)	Oct., 1943.
0032.2	Catalytic Cracking of Gas Oil Fraction of Palembang Crude. (Toa Fuel Research Lab.)	Apr., 1943.
0032.3	Report on the Test Run of Catalytic Cracking (Toa Fuel Ind. Co.)	July, 1943.
0032.4	Catalytic Cracking of Gas Oil from Kettleman Hill Crude (Toa Fuel Research Lab.)	May, 1942.
0032.5	Catalytic Cracking of Gas Oil from Mangunjaja Crude. (Toa Fuel Research Lab.)	Apr., 1943.
0032.6	Catalytic Cracking of Gas Oil from Djambi Crude. (Toa Fuel Research Lab.)	June, 1942.
0032.7	Catalytic Cracking of Gas Oil from Synthetic Oil by Fischer-Tropsch. (Toa Fuel Research Lab.)	June, 1942.
0032.8	Report on the Result of Test Run of the NNK Unit. (Nippon Oil Co.)	Nov., 1943.
0032.9	Studies on the Catalytic Cracking Reaction Tower. (Toa Fuel Research Lab.)	

RESTRICTED

ENCLOSURE (D)

ENCLOSURE (D)

THE PETROLEUM OIL INDUSTRY IN JAPAN

by

I. ACKI

G. NARA

of the SHUN NOMURA Office

January 1946

ENCLOSURE (D)

TABLE OF CONTENTS

I.	Introduction	Page 45
II.	History of the Petroleum Oil Industry in Japan	Page 45
III.	Production of Oil	
	A. Origin of Oil in Japan	Page 49
	B. Distribution of Oil Fields in Japan	Page 50
	C. Amount of Japanese Crude Oil Production	Page 53
IV.	Supply-Demand and Relation of Oil in Japan	
	A. Consumption of Oil	Page 54
	B. Oils Imported to Japan	Page 54
V.	Oil Refining Processes	
	A. Crude Stills	Page 55
	B. Cracking Units	Page 55
	C. Vacuum Distillation Units	Page 55
	D. Lubricating Oil Refining	Page 56

LIST OF TABLES
AND ILLUSTRATIONS

Table I(D)	Nagai Oil Comparison Table	Page 57
Figure 1(D)	Flow Sheet for Refinery Operating on Crude Oil Produced in Liliku, Sumatra	Page 58

ENCLOSURE (D)

I. INTRODUCTION

Extraordinary development of modern mechanical industry in Japan has required much petroleum. Moreover, the development in internal combustion engines necessitated not only increased production of oil, but also improvement in its properties, thereby resulting in the present development of the petroleum industry.

In order to meet the above conditions, fractions of naturally produced hydrocarbons, suitable for use in modern machinery, were collected and at the same time unsuitable fractions thereof were decomposed, polymerised or re-refined by solvent to make use of these fractions completely. At the same time research work on coal liquefaction and conversion of animal and vegetable oils into petroleum hydrocarbons was carried on.

As to motor fuel, topping of natural crude oil, decomposition of topped bottom oil, thermal catalytic cracking of naphtha with active acid clay and hydrocracking of naphtha have been effected. A very small quantity of motor fuel has been produced by means of coal liquefaction. The coal liquefaction plants were converted to hydrocracking of petroleum naphtha at the request of the Army and Navy during the war.

As to lubricant oils, low class lubricating oil has been treated by a process in which the distillate from vacuum distillation is treated with sulphuric acid and washed with alkali and warm water. Middle class lubricating oil has been produced by the dewaxing of paraffinic crude oil or by blending high class lubricating oil with low class lubricating oil. High class lubricants have been produced only by the Duo-sol or Furfural solvent refining processes. In case of de-waxing, the Barisol and Acetone-Benzol processes have been adopted. For final refining, the contact re-run process has been adopted.

The conversion of animal and vegetable oils into high class lubricants was planned; the Soap Manufacturing Co. intended to construct a commercial plant, but testing of the product was not completed. Conversion of rubber into lubricating oil, etc. has been accomplished commercially, but good results were not obtained.

II. HISTORY OF THE PETROLEUM OIL INDUSTRY IN JAPAN

It is mentioned in the Nippon Shoki (an ancient Japanese biography), that burning soil and burning water were presented to the Emperor TENJI by the people of ECHIGO in Niigata Prefecture in 668. In 1613, Mr. Nihei MAGARA constructed an oil well at KARAMEGI, Nakakanbara District, Niigata Prefecture. This crude oil was supplied to a simple distillation plant, and light oil was produced and sold. Later, Mr. Yasunojo HIRANO co-operated with Mr. Cynchroton, an American, and constructed an oil well by hand at KUROKAWA, Niigata Prefecture. Even to the present day, this hand sunk oil well has been called a "foreigner's oil well". In 1871 Mr. Shuzo ISHIZAKA founded a modern oil company with a capitalization of ¥30,000. At the same time a rotary drilling system was imported from America. In 1888, the Nippon Oil Co., with capitalization of ¥150,000 was founded by Mr. Hisahiro NAITO. The company imported a cable type drilling machine and installed it at Ogose Shore, NIIGATA. In 1892 the Nagaoka Iron Pipe Company was founded and in 1893 this company laid iron pipe to feed oil from HIGASHIYAMA to oil companies located in the city of NAGAOKA. In 1900 the Nishiyama oil field was developed and production of oil reached 1739 barrels per day.

In 1900 the International Oil Co., with a capitalization of ¥10,000,000 was founded, and the company constructed a large scale oil refinery near NACETSU, Niigata Prefecture, for operation on purchased Nishiyama crude oil. In 1911

ENCLOSURE (D)

the company failed to exploit the Hokkaido oil fields and all of the company's business and property were assigned to the Nippon Oil Company. In 1910 the Nippon Oil Co. obtained 1577 barrels per day of crude oil at NIIZU KARAMEGI, Niigata Prefecture. In 1904 the Nanboku Oil Company was founded and the company installed an oil refinery plant of large scale at HODOGARA, Kanagawa Prefecture to process crude oil imported from foreign countries. Later on, this company was amalgamated with the Takarada Oil Company. In 1914 the Nippon Oil Co. bought a rotary drilling machine and obtained 11,345 barrels per day of crude oil at the Kurokawa oil field, Akita Prefecture.

In 1918, as the result of the first European war, the import of steel drill pipe was stopped, thereby hindering oil well drilling. The Nippon Kokan Co., Ltd., therefore, was founded for the purpose of manufacturing steel pipe.

In 1921 the Nippon Oil and the Takarada Oil united to form the Nippon Oil Co., Ltd. with capitalization of ¥80,000,000. In 1925 agreement on oil field rights in North SAKHALIN was reached in the Russo-Japanese treaty. In 1926 the Kita Karafuto Oil Co., with a capitalization of ¥10,000,000, was founded. In 1930 Borneo Oil Co., with combined capital of Japan and Holland, was founded to start test drilling of oil fields in Borneo. The Nippon Oil Co. installed a natural gasoline absorption plant and a carbon black works in 1931 at KINSUI, Formosa. In 1934 the Innai oil field, AKITA Prefecture, and the Kokuni and Omonogawa oil fields were exploited. In 1936 the Yabase oil field, AKITA Prefecture, was exploited, and the field proved to be a very good one.

The names and locations of oil refining companies in Japan as of 1 November 1939, are as follows:

Hayama Oil Co.	TOKYO
Nippon Oil Co.	TOKYO
Niizu Oil Co.	NIIGATA
Toyo Oil Co.	OSAKA
Toho Oil Co.	YOKOHAMA
Toyo Shoko Oil Co.	TOKYO
Ogura Oil Co.	TOKYO
Yamabun Oil Co.	OSAKA
Yamagishi Oil Co.	TOKYO
Maruzen Oil Co.	KOBE
Edogawa Oil Co.	TOKYO
Asahi Oil Co.	TOKYO
Aikoku Oil Co.	TOKYO
Aoki Oil Co.	EHIME
Kyoei Oil Co. (dissolved)	TOKYO
Marusu Oil refinery plant	TOBATA
Mitsubishi Oil Co.	TOKYO
Mikado Oil Co.	HYOGO

The names and locations of oil refining companies still existing as of 1 May 1944, are as follows:

Nippon Oil Co.	(Nippon Oil, Aikoku Oil and Ogura Oil are united).
Maruzen Oil Co.	(Toyo Oil, Toho Oil and Kyushu Oil refinery plant are united).
Koa Oil Co.	

ENCLOSURE (D)

Mitsubishi Oil Co.

Showa Oil Co. (Asahi Oil, Hayama Oil and Niizu Oil are united).

Taikyo Oil Co. (Yamagishi Oil and Ogura in NIIGATA Prefecture are united).

Nippon Kogyo Co.

Aoki Oil Co.

More recent oil refining plant developments are listed as follows:

<u>Company</u>	<u>Location</u>	<u>Type of Plant</u>	<u>Year Established</u>
Nippon Oil	AKITA	Continuous distillation	1915
Nippon Oil	TSURUMI	Tubbs Cracking	1924
Nippon Oil	NIIGATA	Cross Cracking	1926
Ogura Oil	TOKYO	Jenkins Cracking	1926
		Schultz Vacuum Distillation	1926
Nippon Oil	NIIGATA	Schultz Vacuum Distillation	1927
Nippon Oil	KUDAMATSU	Cross Cracking	1927
Ogura Oil	YOKOHAMA	Cross Cracking	1927
Mitsubishi Oil	MITSUBISHI	Cross Cracking	1927
Nippon Oil	KUDAMATSU	Foster Pipe Still	1930
Nippon Oil	KUDAMATSU	Smith-Leslie Pipe Still	1930
Mitsubishi Oil	MITSUBISHI	Kellogg Pipe Still	1931
Ogura Oil	YOKOHAMA	Kellogg Pipe Still	1931
Nippon Oil	TSURUMI	N. N. C. Pipe Still (Nippon Oil Patent)	1932
Yamabun Oil		Madger Vacuum Distillation	1935
Hayama Oil	KAWASAKI	Schwartz Cracking	1935
Nippon Oil	TSURUMI	N. N. C. Cracking (Nippon Oil Patent)	1935
Aikoku Oil		N. N. C. Cracking	1937
Aikoku Oil		N. N. C. Pipe Still	1937

ENCLOSURE (D)

<u>Company</u>	<u>Location</u>	<u>Type of Plant</u>	<u>Year Established</u>
Osaka Mineral Oil		Badger Vacuum Distillation	1937
Toyo Oil	TSURUMI	Badger Vacuum Distillation	1937
Ogura Oil		Barisol Dewaxing Process	1937
Toyo Shoko		Barisol Dewaxing Process	1938
Niitsu Oil		Schwartz Cracking	1938
Asahi Oil		Badger Vacuum Distillation	1938
Hayama Oil	KAWASAKI	Badger Vacuum Distillation	1939
Toho Oil		Badger Vacuum Distillation	1939
Toa Nenryo		Topping Pipe Still	1939
Hayama Oil	KAWASAKI	Duo-sol Process	1939
		Contact Re-run Process	1939
Maruzen Oil		Barisol Dewaxing Process	1939
		Contact Re-run Process	1939
		Duo-sol Process	1939

Note: The above companies were established before 1944.

A table showing oil refining plants provided with comparatively modern equipment follows:

<u>Location of Plant</u>	<u>Name of Co.</u>	<u>Feature of Plant</u>
KASHIWAZAKI, Niigata Pref.	Nippon Oil Co.	Gasoline, Paraffin Pipe Still.
NIIGATA Pref.	Nippon Oil Co.	Schultz Vacuum Distil. Paraffin, Gross Cracking Distillation, Cold-settling, Dewaxing.
TSUCHIZAKI, Niigata, Pref.	Nippon Oil Co.	Cold-settling Dewaxing, N.N.C. Pipe Still.
TSURUMI, Kanagawa Pref.	Nippon Oil Co.	Dubbs Cracking, N.N.C. Pipe Still N.N.C. Cracking.

ENCLOSURE (D)

<u>Location of Plant</u>	<u>Name of Co.</u>	<u>Feature of Plant</u>
KUDAMATSU, Yamaguchi Pref.	Nippon Oil Co.	Cross Cracking, Foster Pipe Still, Smith-Leslie Pipe Still.
TOKYO	Ogura Oil Co.	Jenkins Cracking, Schultz. Vacuum Distillation.
YOKOHAMA	Ogura Oil Co.	Schultz Vacuum Dist. Kellogg Pipe Still, Cross Cracking.
KAWASAKI	Mitsubishi	Kellogg Pipe Still, Cross Cracking.
KAWASAKI	Hayama Oil Co.	Heckmann Vacuum Distillation, Schwartz Cracking Topping Distillation, Vacuum Distillation, Badger Vacuum Distillation, Barisol, Duo-Sol, Contact Re-run.
KANAGAWA Pref.	Toho	Heckmann Vacuum Distillation, Badger Vacuum Distillation.
KANAGAWA Pref.	Aikoku	N.N.C. Pipe Still, N.N.C., Cracking.
TOKYO	Edogawa Refinery	Heckmann Vacuum Distillation.
TOKYO	Asahi Oil Co.	Heckmann Vacuum Distillation, Badger Pipe Still.
NIIGATA	Niizu	Heckmann Vacuum Distillation. Schwartz Cracking
OSAKA	Maruzen	Barisol, Duo-sol. Contact-Rerun, Heckmann Vacuum Distillation, Cracking. Contact-Rerun, Heckmann Vacuum Distillation, Cracking.
OSAKA	Toyo	Heckmann Vacuum Distillation Badger Pipe Still.
KANAGAWA Pref.	Toyo Shoko	Badger Pipe Still.
OSAKA	Yamabun	Badger Pipe Still.

III. PRODUCTION OF OIL.A. Origin of Oil in Japan

Petroleum oil consists of a mixture of various hydrocarbons, a very small quantity of compounds of oxygen, sulphur and nitrogen, and other

ENCLOSURE (D)

inorganic substances. Miscellaneous theories on the formation of oil have been discussed among scholars. These are mainly divided into the inorganic origin theory, as represented by carbide origin theory, and the organic origin theory which is further divided into the vegetable origin theory, the animal origin theory, and the sapropolite origin theory. Japanese scholars have adopted the animal origin theory and sapropolite origin theory to explain the formation of oil in Japan, which theory assumes that marine animals are buried and that decomposition, polymerisation, etc., are effected by means of pressure and heat during long periods to form the present petroleum oil. This presumption has also been tested experimentally. In 1888 Mr. Engler obtained oil by the dry distillation of fish. Dr. K. KOBAYASHI has proved that the above decomposition of marine animals could easily be carried out at atmospheric pressure in the presence of acid clay. Dr. Y. TANAKA ascertained the presence of aliphatic acids such as palmitic, stearic, etc., in Japanese crude oils. All oil strata in Japan belong to the tertiary aqueous rock.

Japanese crude oil contains mainly naphthene base hydrocarbons and is called naphthene base oil.

B. Distribution of Oil Fields in Japan

The oil fields in Japan are distributed from Sagara oil field in SHIZUOKA Prefecture through NAGANO, NIIGATA and AKITA to HOKKAIDO, extending across the Southern and Northern districts like the backbone of an animal. These oil fields are classified into (1) oil fields in NIIGATA Prefecture, (2) oil fields in AKITA Prefecture and (3) oil fields in HOKKAIDO. Properties of crudes from these districts are given in the following table.

Properties of Japanese Crude Oils

Name of Crude Oil	Color	Pitch (%)	Paraffin (%)	Degrees (°Be)	Flash pt (°C)	Remarks
KUBIKI (NIIGATA)	Dark green	4.0	0.847	37.2	10.5	Contains about 30% of gasoline, light oil and kerosene respectively.
OGOSE (NIIGATA)	Green Red Opaque	1.0	2.11	35.0	55.0	Contains almost no gasoline, 50% of light oil, and about 40% of kerosene. Flash pt. is high.
KOSHIMA (NIIGATA)	Beautiful red, transparent	1.0	less than 0.5	41.7	less than 0	Contains 45% of gasoline, 15% of light oil, and about 25% of kerosene. Excellent quality.

ENCLOSURE (D)

Name of Crude Oil	Color	Pitch (%)	Paraffin (%)	Degrees ($^{\circ}$ Be)	Flash pt($^{\circ}$ C)	Remarks
NISHIYAMA Light (NIIGATA)	Dark green Opaque	3.0	0.158	27.3	60.0	Contains little gasoline and about 35% of heavy oil. Used as raw material for lubricant oil.
NISHIYAMA Heavy (NIIGATA)	Dark green Opaque	2.5-3.5	0.864	30.1	26.0	Contains 30% of gasoline 25% of light oil and about 30% of kerosene, is the best crude oil in Japan. The bottom oil is used as raw material for paraffin and lubricant oil.
NISHIYAMA IRIWADA Deep Stratum (NIIGATA)	Conc. green by reflected ray yellow brown by passed ray		1.23	42.2	less than 20 below zero	Contains about 43% of gasoline, 18% of light oil, about 24% of kerosene and about 1% of paraffin. Excellent paraffin base crude oil.
NISEIYAMA TAKEMACHI (NIIGATA)	Black green transparent		3.319	40.0	Below zero	Has high content of gasoline and a small quantity of pitch. The pour pt. of light oil component is elevated owing to high paraffin content.
HIGASHIYAMA (NIIGATA)	Black brown Opaque	6.0	0.691	27.6	26.5	Contains 15-20% of gasoline and 45-50% of kerosene. The bottom oil is raw material for paraffin and also suitable for heavy cylinder oil.
OHCMO (NIIGATA)	Black brown Opaque	6.0	0.337	27.5	37.0	Contains 10-15% of gasoline and light oil can also be manufactured.

ENCLOSURE (D)

Name of Crude Oil	Color	Pitch (%)	Paraffin (%)	Degrees ($^{\circ}\text{Be}$)	Flash pt ($^{\circ}\text{C}$)	Remarks
NANOKAICHI (NIIGATA)	Conc. green black Opaque	10.5	0.117	13.8	103.0	Excellent machine oil can be manufactured from the heavy component. The pour pt. of crude oil is less than -20°C .
NIIZU (NIIGATA)	Black brown Opaque	12.5	0.395	13.2	114.0	The pour pt. is less than 20°C . No gasoline and light oil components. Raw material for kerosene, transformer oil and lubricant oil.
NIIZU KUMAZAWA (NIIGATA)	Black	10.0	0.156	13.10	102	The pour pt. is less than -20°C . Suited for manufacture of lubricant oil.
KATTE (AKITA)	Black Opaque	5.8	1.867	32.7	25	Contains gasoline, kerosene and light oil. Suitable as raw material for cylinder oil.
KINSHOZAN (AKITA)	Black Opaque	13.5	0.277	13.7	63	Suitable as raw material for light oil, kerosene, lubricant oil and asphalt.
MICHIKAWA (AKITA)	Black Opaque	36.1	Trace	13.9	121	Is raw material for kerosene, heavy oil and lubricant oil. Asphalt can be obtained.
KUROKAWA (AKITA)	Black Opaque	24.0	0.319	16.1	88	Lubricant oil, light oil and asphalt can be obtained.
TOYOKAWA (AKITA)	Black Opaque	30.7	0.32	14.7	-	Represents Japanese asphalt base crude oil. Suitable for manufacture of lubricating oil and light oil. 37% of asphalt can be obtained.

ENCLOSURE (D)

Name of Crude Oil	Color	Pitch (%)	Paraffin (%)	Degrees (°Be)	Flash pt(°C)	Remarks
KATSURANE (AKITA)	Blue Black	9.0	0.983	34.0	less than 15	A mixed base crude oil.
ASAHIGAWA (AKITA)	Black Opaque	20.0	0.542	21.2	30	Suitable for manufacture of gasoline, kerosene, light oil and lubricating oil. Raw material for asphalt.
DOYOMIGAWA (AKITA)	Black Opaque	15.0	0.502	20.7	51	Contains 20% of gasoline, 20% of light oil and 28% of kerosene. Suitable for manufacture of lubricating oil and asphalt.
HANEKAWA (AKITA)	Blue Black Opaque	5.6	2.108	32.0	23	Contains much paraffin, about 22% of light oil and about 20% of kerosene. Raw material for lubricating oil.
ISHIKARI (HOKKAIDO)	Blue Black Opaque		1.313	43.1	less than zero	Contains about 45% of gasoline and comparatively rich in paraffin.
SAGARA (SHIZUOKA)	Blue Green Transparent		1.192	42.0	-9	High content of gasoline and light oil.

C. Amount of Japanese Crude Oil Production

The crude oil production (barrels) in Japan (including FORMOSA) from 1930 to 1936 is as follows:

1930	2,048,564 barrels
1931	1,966,007
1932	1,627,253
1933	1,455,186
1934	1,820,479
1935	2,249,198
1936	2,539,311

ENCLOSURE (D)

The crude oil production in various districts in Japan is listed as follows:

<u>Prefecture</u>	<u>Barrels</u>
NIIGATA	797,176
AKITA	529,105
HOKKAIDO	92,018
SHIZUOKA	163
NAGANO	151
YAMAGATA	119
Total	1,418,732

The crude oil produced in various oil fields in Japan from January to June, 1935 is shown as follows:

<u>Oil Field</u>	<u>Production (bbls)</u>	
	<u>Jan.-June</u>	<u>Per Day</u>
NISHIYAMA, Niigata	146,255	794
NIIZU, Niigata	117,900	647
NAKANO OGUNI, Akita	141,859	794
TOYOKAWA, Akita	36,980	476
ASAHIKAWA, Akita	65,878	363
HIGASHIYAMA, Niigata	37,453	204
OHOMO, Niigata	27,811	159
YURI, Akita	22,359	125
ATSUMA, Hokkaido	26,215	147
ISHIKARI, Hokkaido	15,628	85
NIPPON OGUMI, Akita	11,877	65
INNAI, Akita	177,607	976
ASAHI INNAI, Akita	44,174	250

IV. SUPPLY-DEMAND AND RELATION OF OIL IN JAPANA. Consumption of Oil

About 1926 Japan's demand for petroleum was 20% fulfilled by Japanese crude oils and 80% by imported oils. These imported oils included both refined and crude oils. As the production of Japanese crude oils did not increase in proportion to the demand, the ratio to the imported oils became smaller. By 1936 only about 10% of the demand was met by Japanese crude oils. Thus, in 1936, the demand of oil for civilian use in Japan was 3,119,000 tons per annum, while the production of Japanese crude oil was about 320,000 tons and imports of refined oil amounted to 1,811,000 tons, and of imported crude oil, 1,048,000 tons per annum.

B. Oils Imported to Japan

1. Crude oil for manufacture of gasoline. In 1923 the Nippon Oil Co. imported crude oil for the first time from California through the Mitsui Bussan Co. The oil was Long Beach crude of 24° Be supplied by the General Oil Co. Thereafter, 28° Be and 30° Be crude oils were imported from the Associated Oil Co. and Union Oil Co., respectively. Of gasoline that was manufactured, 25% came from these oils. In 1927 the Nippon Oil Co. imported 25° Be Elwood crude oil produced by the Rio Grande Oil Co. and containing more than 30% of gasoline. In 1931 Kettleman crude, which contained more sulphur than Elwood crude, and had a 40% yield of gasoline, was purchased at a comparatively cheap price. The Asano Bussan Co. also imported

ENCLOSURE (D)

Kettleman crude from the Standard Oil Co. in California. Thus, the crude oil imported into Japan was mainly Kettleman. Quantities were also imported from Ecuador for manufacture of gasoline and light oil. Poza Rica crude oil was imported from Mexico. The above crudes were mainly used for manufacture of automotive gasoline.

Midway Sunset, Olinda Brea, San Joaquin Valley and Wilmington crudes were imported for the purpose of manufacturing aviation gasoline. Gulf Coastal crude was also imported for the same purpose.

2. Crude for manufacture of lubricating oil. Coalinga, Foso Creek, Miri, Round Mountain, and other crude oils were imported for the purpose of manufacturing lubricating oils.

V. OIL REFINING PROCESSESA. Crude Stills

Most of the oil refinery plants of large scale have adopted pipe-stills for continuously distilling crude oil. In 1930 the Nippon Oil Co. constructed a Foster Pipe-still at the Kudamatsu Oil refinery plant for distilling crude oil. A Smith-Leslie Pipe-Still was also constructed there. A pipe-still patented by the Nippon Oil Co. was installed at the Tsurumi Oil Refinery Plant of the same company in 1932 and at the Aikoku Oil Co. in 1937.

B. Cracking Units

In 1924 the Nippon Oil Co. constructed a Dubbs cracking system at the Tsurumi Oil Refinery. Gross units were installed at the Niigata Oil Refinery of the Nippon Oil Co. in 1926, at the Kudamatsu Oil Refinery of the Ogura Oil Co. in 1931, and at the Kawasaki Oil Refinery of the Mitsubishi Oil Co. in 1931. In 1926 a Jenkins unit was constructed at the Tokyo Oil Refinery of the Ogura Oil Co. In 1933 a Gyro unit was constructed at the Navy Tokuyama Refinery for research purposes. Schwartz units were constructed at the Kawasaki Oil Refinery plant of the Hayama Oil Co. in 1935 and at the Niizu Oil Co. in 1938. The Nippon Oil Co. constructed a N.N.C. system in April 1935. The Aikoku Oil Co. also adopted the N.N.C. system.

C. Vacuum Distillation Units

Vacuum distillation plants have been adopted in this country as follows:

Schwartz (Continuous)	Nippon Oil Co.	in 1927
	Ogura Oil Co.	in 1926
Heckmann (Continuous)	Maruzen Oil Co.	in 1929
(Batch)	Edogawa Oil Refinery Plant	in 1929
	Hayama Oil Co.	in 1931
M. H. (Batch)	Niizu Oil Co.	in 1931
(Improvement of Heckmann)		
Kellogg (Continuous)	Mitsubishi Oil Co.	in 1932
	Ogura Oil Co.	in 1937
Badger (Continuous)	Osaka Oil Refinery Plant	in 1937
	Toyo Oil Co.	in 1937
	Toyo Shoko Oil Co.	in 1938
Cubro (Continuous)	Hayama Oil Co.	in 1937

ENCLOSURE (D)

D. Lubricating Oil Refining

The most common process of refining lubricating oils consists of washing the distillate with concentrated sulphuric acid, caustic soda solution, and warm water, and heating in a large iron tank to clarify the oil. In recent years the Sharples Centrifuge has been used in large plants for sulphuric acid washing. Acid oil obtained from distillates of high viscosity tends to emulsify and water can not easily be separated therefrom after washing with caustic soda solution. Accordingly, after the concentrated sulphuric acid washing, active acid clay is used instead of caustic soda washing to effect deoxidation and decoloration at the same time. The active acid clay is one which is obtained by activating acid clay with hydrochloric acid or sulphuric acid and has a decolorizing power of 2.5 to 3 times that of acid clay. If the crude oil contains paraffin, it must be completely dewaxed.

In recent years, refining plants for the manufacture of high class lubricating oils by means of solvents have been installed because of the development of aircraft. The Showa Oil Co. and Maruzen Oil Co. adopted refining processes in which the furfural process, Duo-sol process, Barisol dewaxing process and contact re-run process are combined to form one plant. In 1942 the Showa Oil Co. and Maruzen Oil Co. commenced the manufacture of aviation lubricating oil. A Japanese crude oil highly suited for refining of high class lubricants by means of solvent refining is Omonogawa crude oil from AKITA Prefecture. The viscosity index of this crude oil is about 85-90.

Two processes for solvent refining lubricating oils are used in Japan. One is Duo-sol refining, Barisol dewaxing, and contact re-run refining.

The Navy used a propane de-asphalting process, propane dewaxing, and active acid clay refining, but good results were not obtained.

The Nippon Oil Co. used a process in which the furfural extraction and propane de-asphalting were combined. In April, 1945, the Army constructed the Marifu Oil Refinery of 2,000 bbl/day in YAMAGUCHI Prefecture. This plant, later destroyed by bombardment, planned to refine Liliku crude oil produced in Sumatra by means of the Duo-sol process. A flow chart for the plant is given by Figure 1(D).

The conversion of animal and vegetable fatty acid into high class lubricating oils has been earnestly studied in Japan. Plants based on this conversion were constructed and pilot plant products were produced. However, at the termination of the war the refined oil was not produced on commercial scale. The Nippon Tokushu Oil Co. adopted a process in which condensation of benzol and paraffin was utilized. The Nippon Fatty Co. adopted a process in which fatty acid, after hydro-cracking, is condensed in the presence of aluminium chloride. The Miyoshi Chemical Engineering Co. adopted a process in which fatty acid, after saponification with alkali, is condensed in the presence of aluminium chloride. The Miyoshi Chemical Engineering Co. adopted a process in which fatty acid, previously heat treated, was decomposed and polymerized in the presence of active acid clay, and the product thus obtained was hydrogenated to yield refined oil. The best result was obtained when oxidation inhibitor was added to this oil. Dr. Yuzaburo NAGAI, a professor at Tokyo Imperial University, studied the last mentioned process for many years. A 7,000 KL/year plant based on this process was completed at AMAGASAKI, HYOGO and was under test operation at the termination of the war. The product obtained from the pilot plant was used for the 16,435 km. non-stop flight in MANCHUKUO under the auspices of the Asahi Newspaper. In this flight lu-

ENCLOSURE (D)

bricant consumption was 4-5 gm hp/hour at maxium horse power, and 0.9 gm hp/hour at the normal cruising output. This showed that the oil from the above pilot plant was excellent in every respect. Test results of this lubricant are as follows (wherein the oil was obtained from Nagai's synthetic oil to which was added blending oil obtained from naphthenic base petroleum oil and organic metallic salt inhibitor):

Table I(D)
NAGAI OIL COMPARISON TABLE

Oil Tested		Viscosity			Carbon After Oxidation (%)	Increase of Hydrogen	
		Centistokes		Index			Ratio at 100°F
		210°F	100°F				
Phillips*		26.03	460.1	82.2	1.54	2.602	1.952
Gulf Frilde*		25.84	463.6	80.4	1.60	2.190	1.722
City Service*		26.06	454.8	83.6	1.58	2.490	1.634
Nagai's Oil**	BO 5/I 0	24.88	317.9	106.6	1.41	0.820	0.634
	BO 5/I 0.01	24.82	317.5	106.6	1.23	0.532	0.234
	BO 5/I 0.05	24.88	317.9	106.6	1.16	0.470	0.224
	BO 0/I 0.10	24.82	317.4	106.6	1.16	0.472	0.216
	BO 10/I 0	24.79	325.6	104.6	1.28	0.890	0.712
	BO 10/I 0.01	24.76	324.1	104.9	1.24	0.598	0.400
	BO 10/I 0.05	24.76	323.7	105.0	1.17	0.524	0.334
	BO 10/I 0.10	24.73	323.7	104.9	1.16	0.536	0.346
	BO 15/I 0	24.73	330.6	103.3	1.35	0.926	0.724
	BO 15/I 0.01	24.73	330.2	103.4	1.25	0.706	0.504
	BO 15/I 0.05	24.79	330.1	103.7	1.19	0.600	0.399
	BO 15/I 0.10	24.82	330.4	103.7	1.18	0.588	0.406

* (made in America)

** BO - Blending Oil

I - Inhibitor

For example, BO 5/I 0.01 indicates that 5% blending oil and 0.01% inhibitor were added.

ENCLOSURE (D)

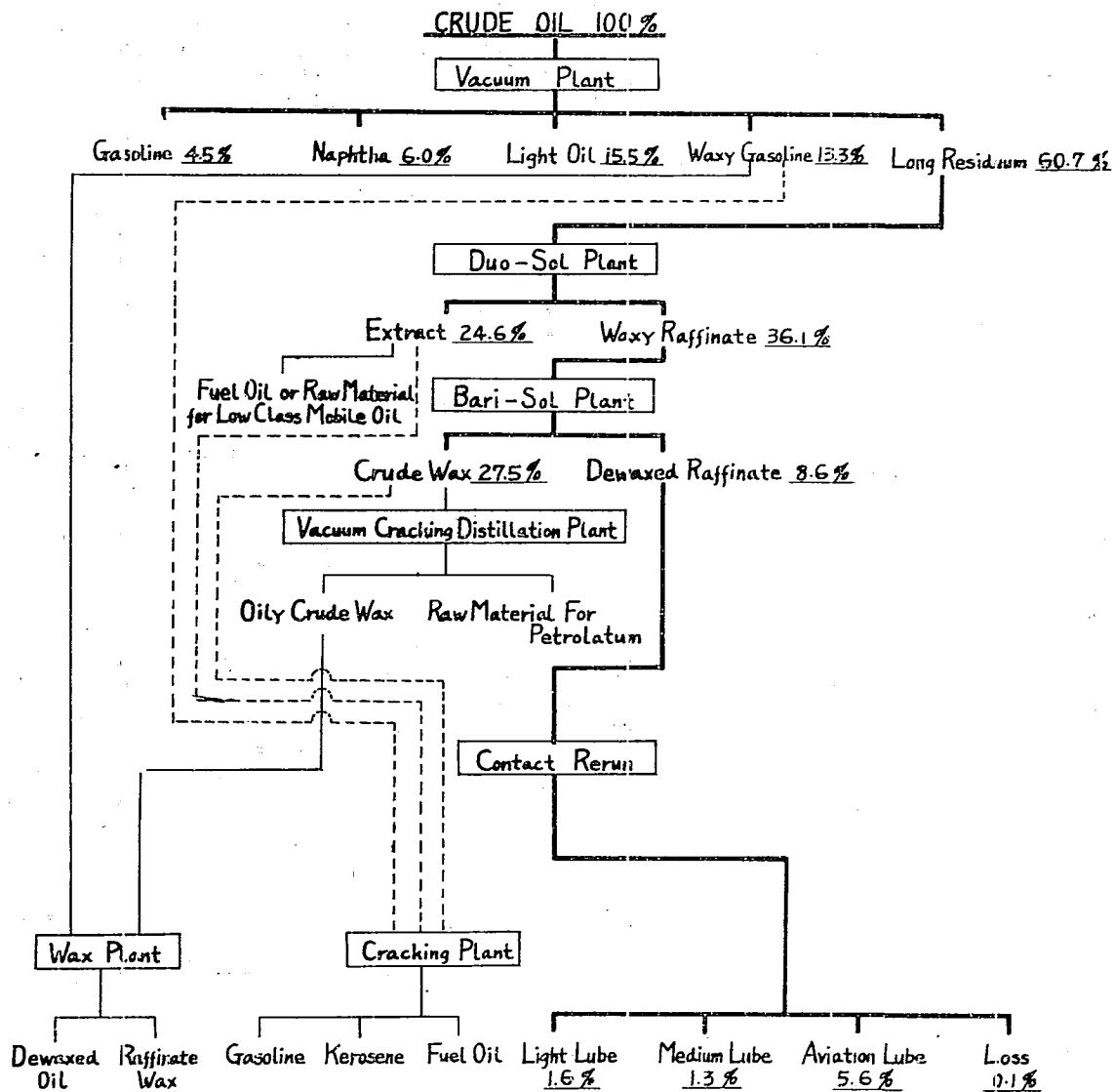


Figure 1 (D)

FLOW SHEET FOR REFINERY OPERATING ON CRUDE OIL
 PRODUCED IN LILIKU SUMATRA
 PLANT CAPACITY: CRUDE OIL 2000 KL/DAY

RESTRICTED

ENCLOSURE (E)

ENCLOSURE (E)

R E P O R T O N

N I P P O N O I L C O M P A N Y

K U D A M A T S U R E F I N E R Y

ENCLOSURE (E)

TABLE OF CONTENTS

I. Introduction	Page 61
II. History	Page 61
III. Description of Refinery	Page 61

LIST OF TABLES
AND ILLUSTRATIONS

Table I(E)	Crude Oil Distilled at Kudamatsu Refinery, 1942 ...	Page 63
Table II(E)	Crude Oil Distilled at Kudamatsu Refinery, 1943 ...	Page 64
Table III(E)	Crude Oil Distilled at Kudamatsu Refinery, 1944 ...	Page 65
Table IV(E)	Crude Oil Distilled at Kudamatsu Refinery, January- June, 1945	Page 65
Figure 1(E)	General Flow Sheet	Page 66

ENCLOSURE (E)

I. INTRODUCTION

This report records and summarizes technical data on the Nippon Oil Co. (Nippon Sekiyu K.K.) refinery at KUDAMATSU, obtained by the Petroleum Section of Nav Tech Jap on 31 October 1945. The following Japanese personnel were interviewed and assisted in supplying the information presented herewith:

Mr. NISHINO - Vice-Superintendent of Refinery

Mr. MIYAHARA - Business Manager

This plant is a conventional crude oil refining installation with capacity of about 23,000 kl per month based on Kettleman Hill's crude. Facilities are available for thermal cracking and solvent refining of lube oil cuts.

II. HISTORY

This plant is owned and operated by Nippon Oil Co. which it was stated, also operated refineries at AMAGASAKI (near KOBE), YOKOHAMA, TSURUMI, AKITA, NIIGATA, KASHIWAZAKI, HOKKAIDO (near SAPPORO), and in Formosa. All of these installations are crude oil refineries.

The crude unit in the Kudamatsu refinery was built in 1929, and the refinery was laid out to process crudes from the United States. During the war, however, other sources of crude were utilized, especially Sumatra. The Government maintained control of the crude stocks and dictated the amount and type of crudes which could be processed. Table I(E) is interesting in this regard, showing the amounts and different types of crude stocks processed during the war.

The refinery suffered severe damage in three bombing raids, on 29 June, 15 and 22 July, 1945, and was completely shut down as a result.

The total number of employees before the end of the war was 385.

III. DESCRIPTION OF REFINERY

An overall flow sheet for the Kudamatsu Refinery is given by Figure 1(E). Crude oil was charged to Foster-type crude unit built in 1939, with capacity of 23,000 kl/month based on charging Kettleman Hill's crude. Crude gasoline was taken overhead, and kerosene and gas oil taken off as side cuts. The straight run gasoline was treated with Doctor solution, and the kerosene and light oil were washed with dilute caustic and H_2SO_4 . During the war, however, a shortage of both caustic and acid resulted in elimination of the treating steps for these products.

Reduced crude from the atmospheric crude tower was flashed in a vacuum tower operating at about 70mm Hg abs. and waxy oil side cut taken off. This oil could be dewaxed by chilling to $-12^{\circ}C$ and pressing. The capacity of the wax pressing system is 100 kl/day of oil charge or maximum wax production of 250 tons per month. The de-waxed oil was distilled and the light overhead sent to diesel, and the bottoms, after treating, to machine or engine oil.

A portion of the waxy oil side cut from the vacuum tower was also sent to the plant for making high quality lube oils constructed by Lummus in 1941. This plant consists of a 50 kl/day methyl-ethyl ketone de-waxing unit, a clay treating unit, and a 15 kl/day vacuum distillation unit. In the vacuum distillation unit the several viscosity cuts required for marine and automotive engine oils were prepared.

ENCLOSURE (R)

Under normal conditions, crude heavy or light gas oils were charged to the two Cross units. These units, each of 200 kl/day charge capacity, were constructed by the M. W. Kellogg Co. in 1929 and 1930. During the war, however, the Japanese Government dictated the amount and type of stock, (viz. kerosene, light gas oil or heavy gas oil), which could be cracked. The Cross units produced bunker oil, and cracked gasoline which, before the war, was acid-treated and re-run in a 120 kl/day Smith-Leslie unit.

During the latter stages of the war, pine root oil was being batch distilled and the products blended with natural oils. Some blends of natural oils with vegetable oils, especially palm and soya bean oils, also were used for the manufacture of lube oils.

ENCLOSURE (E)

Table I(E)
CRUDE OIL DISTILLED AT KUDALATSU REFINERY, 1942

Month	California (Olinda Brea)	Sumatra	California (San Joaquin)	Arabia	Mexico (Pozarilka)	California (Santa Fe)	Balikpapan (Sanga Sanga)	Mexico Iran	Total
Jan.	4,180,500	2,893,800							7,074,300
Feb.	206,002	4,998,242	1,871,411						7,045,655
Mar.	4,832,450	3,932,556		1,840,053	2,326,466				11,931,525
Apr.	4,384,099	900,072	1,179,360		2,782,756	466,169			9,712,456
May	5,066,590				3,244,708				8,311,298
June								8,484,394	8,484,394
July									
Aug.							4,606,800		4,606,800
Sept.							22,759	7,017,441	7,040,200
Oct.					3,112,200				3,112,200
Nov.									
Dec.									
Total	18,669,641	11,724,670	3,050,771	1,840,053	11,466,130	466,169	4,629,559	15,501,885	67,348,828

Table II(E)
CRUDE OIL DISTILLED AT KUNIAMATSU REFINERY, 1942

Month	Sumatra	California (SanJoaquin)	Mexico (Pozarika)	Palembang (Ababu,Pentocho)	N.Borneo (Seria)	Total
Jan.		131,500			1,236,400	1,367,900
Feb.	4,058,960				1,889,580	5,948,540
Mar.	3,130,528			2,718,207	6,606	5,855,341
Apr.				8,451,296	1,562,050	10,013,346
May					997,714	997,714
June			589,546	8,135,154		8,724,700
July	3,409,717			4,047,483		7,457,200
Aug.					6,000,000	6,000,000
Sept.	1,425,600				5,046,100	6,471,700
Oct.	11,221,500				2,210,100	13,431,600
Nov.	6,381,583					6,381,583
Dec.	3,688,880				8,917,530	12,606,410
Total	33,316,768	131,500	589,546	23,352,140	27,866,080	85,256,034

Table III(E)
CRUDE OIL DISTILLED AT KUDAMATSU REFINERY, 1944

Month	Sumatra	N. Borneo (Seria)	Total
Jan.	7,405,990	7,298,160	14,704,150
Feb.	12,525,540		12,525,540
Mar.		10,021,300	10,021,300
Apr.		5,540,340	5,540,340
May		4,507,330	4,507,330
June		6,939,410	6,939,410
July		696,660	696,660
Aug.		3,599,990	3,599,990
Sept.		9,468,560	9,468,560
Oct.		1,652,910	1,652,910
Nov.			
Dec.			
Total	19,931,530	49,724,660	69,656,190

Table IV(E)
CRUDE OIL DISTILLED AT KUDAMATSU REFINERY
January-June, 1945

Month	Soerabaja Palembang (Ababu)	N. Borneo (Seria)	Total
Jan.			
Feb.			
Mar.	5,350,060		5,350,060
Apr.	6,255,214	2,000,000	8,255,214
May	1,298,486	1,968,431	3,266,917
June	1,292,075		1,292,075
Total	14,195,825	3,968,431	18,164,256

ENCLOSURE (E)

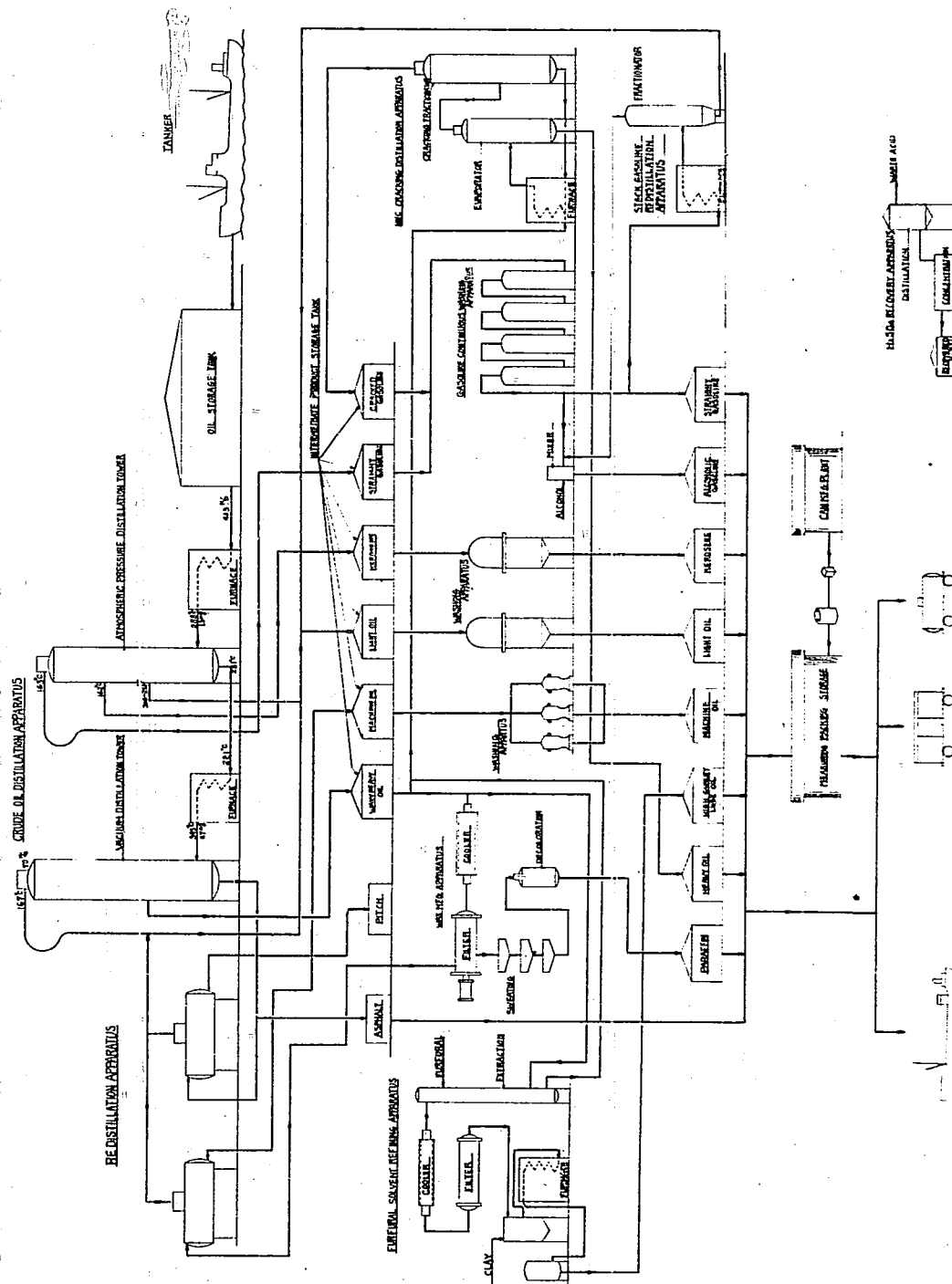


Figure 1(E)
GENERAL FLOW SHEET

RESTRICTED

ENCLOSURE (F)

ENCLOSURE (F)

REPORT ON
THE THIRD NAVAL FUEL DEPOT
TOKUYAMA REFINERY

ENCLOSURE (F)

TABLE OF CONTENTS

I.	Introduction	Page 69
II.	History and Organization	Page 69
III.	Description of Refinery	
A.	General	Page 69
B.	Crude Distillation Units	Page 70
C.	Cracking Units	Page 70
D.	Iso-Octane Plant	Page 70
E.	Cracked Gasoline Hydrogenation Plant	Page 71
F.	Kerosene Hydrocracking Plant	Page 72
G.	Hydrogen Manufacture	Page 72
H.	Lubricating Oils and Greases	Page 73
I.	Synthetic Methanol	Page 73
J.	Ethyl Fluid Plant	Page 73
K.	Pine Root Oil	Page 74
IV.	Refinery Throughput	Page 74
V.	Quality of Products	
A.	Aviation Gasoline	Page 74
B.	Motor Gasoline	Page 74
C.	Kerosene	Page 75
D.	Gas Oil	Page 75
E.	Heavy Oil	Page 75
F.	Fuel Oil	Page 75
G.	Lubricating Oils	Page 75
H.	Greases	Page 75

LIST OF TABLES
AND ILLUSTRATIONS

Table	I(F)	Source of Raw Materials, Third Naval Fuel Depot, Tokuyama	Page 76
Table	II(F)	Consumption of Raw Materials, Third Naval Fuel Depot, Tokuyama	Page 77
Table	III(F)	Actual Production of Finished Products, Third Naval Fuel Depot, Tokuyama	Page 78
Figure	1(F)	Third Naval Fuel Depot Map of Oil Tank Storage Areas	Page 79
Figure	2(F)	Flow Sheet of Lubricating Manufacture, Third Naval Fuel Depot, Tokuyama	Page 80
Plate	I(F)	Refinery Layout Map, Third Naval Fuel Depot, Tokuyama	

ENCLOSURE (F)

I. INTRODUCTION

The Third Naval Fuel Depot, at TOKUYAMA, was inspected by the Petroleum Section of the U. S. Naval Technical Mission to Japan during the period 30 October - 2 November 1945. This report records and summarizes the technical information obtained during this visit. The following Japanese personnel connected with the Depot assisted in supplying the information presented herewith.

Rear-Adm.	I. WATANABE	Head of Depot.
Captain	E. MOTOYOSHI	Director of Gen. Dept.
Captain	S. MATSUNAGA	Director of Refining Dept.

II. HISTORY AND ORGANIZATION

A Naval coal briquetting plant was established at TOKUYAMA in 1905, shortly after the close of the Russo-Japanese War, to supply this fuel to the fleet. Starting about 1920, Naval vessels were gradually equipped with boilers burning heavy fuel oil. In 1921 the name of the plant at TOKUYAMA was changed to the Naval Fuel Depot, and oil refining and research departments were established. In 1940-41, all research work was transferred to OFUNA and in April, 1941, the Depot at TOKUYAMA was designated as the Third Naval Fuel Depot.

The Third Naval Fuel Depot at TOKUYAMA was gradually expanded into one of the most modern and best equipped crude oil refineries in Japan. The plant was completely owned by the Navy and operated under the supervision of commissioned Naval petroleum specialists. The first crude oil pipe-still in Japan, a Trumble unit, was installed at TOKUYAMA in 1920. The plant was designed to operate on imported crude oil and tremendous storage facilities were installed. It was stated that prior to the war approximately 1,250,000 tons of crude oil, mostly from California, were in storage at TOKUYAMA. Although this stock was supplemented by imports of East Indies crude, the storage apparently was exhausted by the end of 1944.

The refinery suffered a major explosives raid on 10 May 1945, and was put completely out of action.

Total peak employment at the refinery was about 3,000.

Maximum total crude capacity was 1,500 kl/day, equivalent to 9,500 bbls/day.

III. DESCRIPTION OF REFINERYA. General

The Tokuyama refinery was a complete, modern crude refinery equipped to carry on crude distillation, thermal cracking, iso-octane manufacture by butene polymerization and hydrogenation, hydrocracking, and the manufacture of lube oils, greases, synthetic methanol, and tetra-ethyl lead. A map of the refinery area showing location of the several units is given by Plate I(F). The refinery had excellent protected harbor facilities for handling incoming and outgoing tanker shipments. Extensive storage facilities were provided within the refinery area and in the nearby hills (refer to Figure 1(F)). Total storage capacity prior to removal of some of the tanks during the war was 1,400,000 metric tons.

ENCLOSURE (F)

Details on the various units are given below.

B. Crude Distillation Units

Three continuous crude stills were installed. The first was a Trumble unit purchased from the U. S. in 1920 with capacity of 350 kl/day; the second, a U. S. Foster type unit with capacity of 350 kl/day; and the third, a two-tower unit designed by the Navy with capacity of 800 kl/day. Total capacity was 1,500 kl/day based on California crude. Typical yields were as follows:

Aviation Gasoline.....	20%
Motor Spirit.....	3%
Kerosene.....	5%
Light Oil.....	8%
Heavy Oil.....	63%
Loss.....	1%
Total.....	100%

A close fractionation plant consisting of two 26 plate columns, each with a charge capacity of 200 kl/day, was installed for preparation of aviation and special naphtha cuts. A pipe-still unit of 200 kl/day capacity equipped with a 4 plate flash tower and a 19-plate column was also installed for re-running miscellaneous stocks.

A vacuum distillation pipe-still, operating at 60mm Hg., and with capacity of 80 kl/day, was built for preparation of lube oil stocks.

All of the above equipment, as well as the cracking unit described below, was constructed by the FUJINAGATA Shipyard.

C. Cracking Units

The first cracking unit was installed about 1933 and was essentially a Cross unit, except that the design was modified by the Navy to eliminate the high pressure reaction chamber by increasing volume of the furnace tubes. Charge capacity of this unit was 250 kl/day.

The second unit was a U. S. design conventional Cross Unit, utilizing a reaction chamber (40 kg/cm² working press.) constructed in Japan, with capacity of 250 kl/day, and the third unit was a Gyro unit of 160 kl/day capacity. Typical yields on these units, presumably from gas oil, were as follows:

Stabilized Gasoline.....	40%
Fuel Residue.....	45%
Gas and Loss.....	15%
Total.....	100%

Cracked gasoline was sent to a vapor-phase clay treating plant equipped with re-run tower. A portion was also sent to the hydrogenation plant.

D. Iso-octane Plant

Iso-octane was manufactured by the polymerization of butylenes and hydrogenation of the polymer. Liquid cracked stabilizer overhead of the following composition was charged to the feed preparation system:

ENCLOSURE (F)

C ₁	0%
C ₂	2%
C ₃	20%
C ₃	35%
C ₄	15%
C ₄	25%
C ₅	3%
Total....	100%

Ethane and lighter were separated in the first column, operating at 20 kg/cm² and reduced temperature, and sent to the tetra-ethyl lead plant. Liquid propane and propylene were separated in the next tower and sent to a small hydrogenation unit where propylene was converted to propane at 200°C and 20 kg/cm² over a catalyst composed of 50% NiO and 50% acid clay. Maximum output was 5 kl/day of 98% propane for use in lube oil refining.

The butane-butylene bottoms from the propane separator was charged to the polymerization reactors operating at 170°C and 4.0 kg/cm² and utilizing Navy type phosphoric acid catalyst consisting of 15% H₃PO₄, 5% Japanese acid clay, and 80% BaSO₄. The reactors were of the tubular type, cooled with superheated water on outside of tubes.

The butylene polymer was hydrogenated at 200°C and 5 kg/cm² over a Nickel catalyst. Reactors were of the tabular type cooled with superheated water on outside of tubes.

The plant was designed by the Navy and manufactured by KOBE SEIKOSHO. Output of hydrogenated product was about 6 kl/day.

E. Cracked Gasoline Hydrogenation Plant

A hydrogenation plant was installed in 1934 for the processing of 80 kl/day of cracked gasoline. Hydrogenation was accomplished over one of the following catalysts:

<u>Catalyst A</u>		<u>Catalyst B</u>	
NiO	50%	NiO	14%
Acid Clay	50%	MoO ₃	43%
		MgO	43%

Reaction conditions were 330-350°C, 100 kg/cm², circulating 5.5 m³ of H₂ per kl of oil. Two reactors were employed containing a total of 2,000 kg of catalyst. A comparison of feed and product oils follows:

ENCLOSURE (F)

		Cracked Gasoline	Hydrogenated Oil
Vapor Press. (kg/cm ²)		0.5	0.6
Distillation	IBP (°C)	40	38
	10%	62	63
	50%	104	105
	90%	148	149
	97%	168	170
Octane No.	Clear 0.1% Lead	72	87

F. Kerosene Hydrocracking Plant

A hydrocracking unit was installed for the manufacture of aviation gasoline from straight run gas oils boiling from 200-300°C. Reaction conditions were 420-470°C, 200 kg/cm² using a catalyst with composition of 14% NiO, 43% MoO₃, and 43% acid clay. A yield of 35-40% of aviation gasoline with 87-91 octane number (0.1% tetra-ethyl lead) was obtained. Oil charge capacity of the unit was 2.5 kl/hr.

The hydrocracking unit was designed by the Navy, based on extensive research work at TOKUYAMA and OFUNA, and constructed by the Kobe Seiko Co.

G. Hydrogen Manufacture

Hydrogen for the several plants, (1-octene hydrogenation, kerosene hydrocracking, cracked gasoline hydrogenation and methanol manufacture), was made from two sources; by cracking of waste gas (methane) from No. 3 Gyro Unit, and from water gas.

The methane cracker was designed and constructed by the Mitsubishi Kakoki Co. to charge 1,500 m³/hr cracking over an iron oxide catalyst (an ore imported from England and containing about 180% iron), in the presence of steam. A checker brick regenerative heating system was utilized for heating the gas to the required temperature. Operation of this unit was found to be very difficult due to heavy carbon deposits and variations both in the amount and quality of the charge gas.

The main source of hydrogen was water gas generated from TAGAWA semi-anthracite.

Two 750 m³/hr. generators were designed and constructed by the Mitsubishi Kakoki Co. and four 500 m³/hr. generators designed by the Navy and constructed by the Ishii Iron Factory. Difficulties with ash removal were encountered with both types.

Mixed gas from the generators was sent to the water gas conversion plant where CO was converted to CO₂ with steam by passing over the Nippon Kasei Co. patented iron oxide catalyst at 470°C. Two units, one of 2,400 m³/hr., and the other of 1,200 m³/hr. charge capacity, were provided. Typical gas composition before and after conversion were as follows:

ENCLOSURE (F)

	<u>Charge</u>	<u>Converted Gas</u>
CO ₂	8%	29%
CO	30%	2%
H ₂	54%	62%
CH ₄ CN ₂	7%	6%
C _n H _{2n}	1%	1%
Total	100%	100%

In the case of the cracked gasoline hydrogenation plant, "converted" gas was purified by removing CO₂ in a medium pressure tower operating at 16 kg/cm² and a high pressure tower at 110 kg/cm². CO remaining in the gas was removed by washing with cupric formate solution under pressure of 110 kg/cm².

In the case of the kerosene hydro-cracking unit, a different system was employed: the CO₂ was removed by washing first with water at 30 kg/cm²; then with 8% caustic solution. The gas was then liquified, using ammonia and nitrogen refrigerating cycles, and relatively pure (98.5%) hydrogen prepared by fractionation at -210°C.

H. Lubricating Oils and Greases

Three lubricating oil plants were installed, a Barisol de-waxing plant of 30 kl/day charge capacity, and two 160 kl/day Duo-Sol extraction plants equipped with propane-de-asphalting equipment. A simplified flow chart for lubricating oil manufacture at TOKUYAMA is given by Figure 2(F).

As part of the dispersal program, one of the Duo-sol plants was being dismantled, prior to end of the war, for shipment to FUKAGAWA.

A small grease plant of 2 ton/day capacity was also installed.

I. Synthetic Methanol

A plant, designed by the Navy to produce 2 kl/day of methanol from water gas, was installed. The composition of the water gas was adjusted to H₂:CO ratio of 2:1 by passing over an Fe₂O₃ catalyst at 200 kg/cm² and 400°C. The gas was cooled, desulfurized, CO₂ removed by water scrubbing, and sent to the reactor containing CuO : U₃O₈ of 92 : 5 mol ratio catalyst and operating at 250°C and 150 kg/cm². Recirculation of reactor exit gases was employed to increase yields.

J. Ethyl Fluid Plant

A plant for producing 150 kl/year of ethyl fluid was installed. Ethyl chloride was made by reacting ethanol with HCl at 140°C and atm. press. in the presence of a 60% FeCl₃ solution. The ethyl chloride was reacted with sodium-lead amalgam (10% by wt. of sodium) in a one cubic meter closed stirring-type autoclave under conditions of 50-70°C, 5-7 atm. press., and 12 hours reaction time. The crude product was filtered to give 45% of theoretical yield of 97% purity tetra-ethyl lead. Ethylene bromide from the Toyo Soda Co., and stabilizer were added to give a

ENCLOSURE (F)

product of the following composition:

Tetra-ethyl lead.....	63-64%	
Ethylene Bromide.....	35-34%	
Kerosene.....	1-1.2%	
Dye.....	0.14%	(Sudane Blue G)
Stabilizer.....	0.1%	(Hydrazo-benzene)

Some difficulties were encountered due to solidification of lead dust and in prevention of lead poisoning.

K. Pine Root Oil

Towards the end of the war preparations were made for the refining of pine root oil, especially for the preparation of aviation gasoline therefrom. A batch type distillation plant of 50 kl/day crude pine root oil charge capacity was constructed. The refining plan was to prepare cuts from the crude oil boiling up to 85°C. from 185-250°C, and heavy oil. The light naphtha was to be either hydrogenated or reformed over Japanese acid clay in "Simplified" catalytic reforming units. The middle cut was to be either hydrocracked in the existing units, or catalytically cracked in a simplified type unit. One hydrocracking urn was made and an aviation gasoline with clear octane of 71 and 93 octane with 0.15% T. E. L. was produced.

IV. REFINERY THROUGHPUT

Table I(F) summarizes the source of raw materials consumed at TOKUYAMA. Table II(F) summarizes the amounts of these materials used during the war years. It will be noted that the production of finished products slumped off badly during 1945, due primarily to shortage of crude.

V. QUALITY OF PRODUCTS

Products were made to meet Navy specifications, and were blended as follows:

A. Aviation Gasoline

Grade No. 2 was made of 50% straight run gasoline from California Crude and 50% of iso-octane, plus 0.15% T.E.L.

Grade No. 1 was made of 80% of straight run from California Crude and 20% of iso-octane, plus 0.15% T.E.L.

Grade 91 was 76 octane straight run California naphtha plus 0.15 T.E.L.

Grade 87 was 70 octane straight run naphtha plus 0.15% T.E.L.

Grade 85 was 65-70 octane straight run naphtha plus 0.15 T.E.L.

Grade 70 was thermally cracked gasoline with no T.E.L.

B. Motor Gasoline

Grade No. 1 was either straight run or cracked gasoline with no T.E.L. (octane about 50).

Grade No. 2 was a blend of No. 1 with 1% by vol. of ethanol.

ENCLOSURE (F)

Grade No. 3 was similar to No. 1 except wider cut.

C. Kerosene

Kerosenes were straight run cuts, acid-treated, and caustic-washed.

D. Gas Oil

Untreated straight run cuts with boiling range of 240-300°C were prepared for use in diesel engines.

E. Heavy Oil

Grade No. 1 is Tarakan crude used for slow speed diesels.

Grade No. 2 is made from 40% of Tarakan crude and 60% of FUSHUN Shale oil. Cetane number is greater than 50.

F. Fuel Oil

Fuel oils were either straight run or cracked resid, or blends of same. No preheater fouling difficulties were reported.

G. Lubricating Oils

The scheme of manufacture of the various lubricating oils is shown by Figure 2(F). Topped crudes were imported from the U. S. No additives were added to aviation lubricating oil. Only one grade of aviation lubricating oil (120) was made, and in winter time it was necessary to heat the oil prior to aircraft takeoffs. Lubricating oil was changed every 40 hours, and was reclaimed by civilian companies. If the reclaimed oil was up to specifications, it would be used again in aircraft engines.

Diesel engine lubricating oil was made from U. S. crude and no additives were used. Turbine oil was made from Sakhalin crude, and contained paraffin as a pour point depressant.

H. Greases

Only three grades (#1, 2, 3) of cup grease were manufactured, employing calcium soaps from whale oil fatty acids. Other greases were obtained from the NIPPON Oil Co.

ENCLOSURE (F)

Table I(F)
SOURCE OF RAW MATERIALS THIRD NAVAL FUEL DEPOT, TOKUYAMA

California Crude	American (Via Kure Military Supply, Tokuyama Branch)
East Indies Crude	Shipped from the South (Producing district unknown)
Aviation 91 Gasoline	Shipped from the South (Producing district unknown)
Aviation 87 Gasoline	Shipped from the South (Producing district unknown)
Motor Gasoline	Domestic (Nippon Oil Company, Ltd.)
Shale Heavy Oil	Manchurian (South Manchurian Railway Company, Ltd.)
Iso-Octane	Korean (Nippon Nitrogen Company)
Alcohol	Domestic (Toa Fuel Co. - Via Hiroshima Local Fuel Station)
Ethyl Fluid	Domestic (Nippon Soda Co. Ltd., Hodogaya Chemical Co. Ltd.)
Raw Materials for Aircraft Lubricating Oil	American (Via Kure Military Supply, Tokuyama Branch)
Raw Materials for Lubricating Oil	American (Via Kure Military Supply, Tokuyama Branch)
Sakhalin Heavy Oil	Sakhalin (Northern Sakhalin Fuel Co. Ltd.)
Fatty Acid	Domestic (Nippon Grease Co. Ltd.)
Paraffin	Domestic (Asahi Electric & Chemical Co. Ltd.)
Tagawa Coal	Domestic (Mitsui Bussan Co. Ltd. - Via Nippon Coal Co. Ltd.)
Cokes	Domestic (Mitsubishi Kasei Co. Ltd. - Via Nippon Coal Co. Ltd.)
Raw Methanol	Korean (Nippon Nitrogen Co. Ltd.)
Ingot Lead	Domestic (Nippon Metal Control Distribution Co. Ltd.)
Ethylene Bromide	Domestic (Toyo Soda Co. Ltd.)
Bromine	Domestic (Toyo Soda Co. Ltd.)
Ethyl Chloride	Domestic (Naniwa Gosei Co. Ltd.)

ENCLOSURE (F)

Table II(F)
 CONSUMPTION OF RAW MATERIALS THIRD NAVAL FUEL DEPOT, TOKUYAMA
 (Kiloliters*)

Name	1941	1942	1943	1944	1945
California Crude	492,000	550,000	260,000	226,000	0
East Indies Crude	0	0	290,000	240,000	12,000
Aviation 91 Gasoline	0	0	24,000	36,000	11,000
Aviation 87 Gasoline	0	0	36,000	24,000	0
Motor Gasoline Materials	0	0	47,000	32,000	1,000
Shale Heavy Oil	70,000	70,000	60,000	45,000	0
Iso-Octane		30,000	64,000	50,000	3,000
Alcohol		425	4,610	7,000	0
Ethyl Fluid	180	220	320	300	0
Lubricating Oils for Aircraft	Unknown	Unknown	Unknown	Unknown	0
Raw Materials for Lubricating Oils	Unknown	Unknown	Unknown	Unknown	200
Sakhalin Heavy Oil	Unknown	Unknown	Unknown	Unknown	1,000
Fatty Acid	0	0	0	Unknown	0
Paraffin	0	0	0	Unknown	0
Tagawa Coal	Unknown	Unknown	Unknown	Unknown	0
Cokes	Unknown	Unknown	Unknown	Unknown	0
Raw Methanol	0	0	0	0	1,000
Ingot Lead		220	320	410	0
Ethyl Fluid	180	220	320	300	0
Ethylene Bromide	0	0	0	55	0
Bromine	0	43	85	0	0
Ethyl Chloride	0	20	50	0	0

* Except for Ingot Lead in tons

ENCLOSURE (F)

Table III(F)
 ACTUAL PRODUCTION OF FINISHED PRODUCTS THIRD NAVAL FUEL DEPOT, TOKUYAMA
 (Kiloliters*)

Item	1941	1942	1943	1944	1945
Aviation Gasoline No. 2	0	0	0	0	700
Aviation Gasoline No. 1	0	0	0	0	100
Aviation Gasoline No. 92	100,000	120,000	100,000	0	0
Aviation Gasoline No. 91			100,000	130,000	6,024
Aviation Gasoline No. 87	50,000	80,000	90,000	120,000	7,760
Aviation Gasoline No. 85	20,000	22,000	24,000	40,000	0
Aviation Gasoline No. 70	8,000	5,000	6,000	10,000	41
Gasoline No. 1	5,000	6,000	5,000	4,000	1,200
Gasoline No. 3			7,000	6,000	12
Gasoline No. 2	12,000	14,000	12,000	10,000	12
Kerosene No. 1	2,000	3,000	2,000	2,000	1,052
Kerosene No. 2	11,000	12,000	12,000	11,000	25
Light Oil	4,000	5,000	4,000	3,000	2,484
Heavy Oil	348,000	38,000	42,000	32,000	5,000
No. 120 Aviation Lubricating Oil	Unknown	Unknown	Unknown	4,120	862
Cylinder Oil	0	0	Unknown	4,550	60
Bearing Oil	0	0	Unknown	1,950	40
Grease	0	0	0	500	0
Spindle Oil	0	0	0	Unknown	0
Ethyl Fluid	0	90	130	180	0
Refrigerating Oil	0	0	0	0	50
Ether	0	0	0	130	0
No. 1 Methanol	Unknown	Unknown	Unknown	Unknown	75
Car Lubricating Oil	0	0	0	0	3

* Except for Heavy Oil and Grease in tons

ENCLOSURE (F)

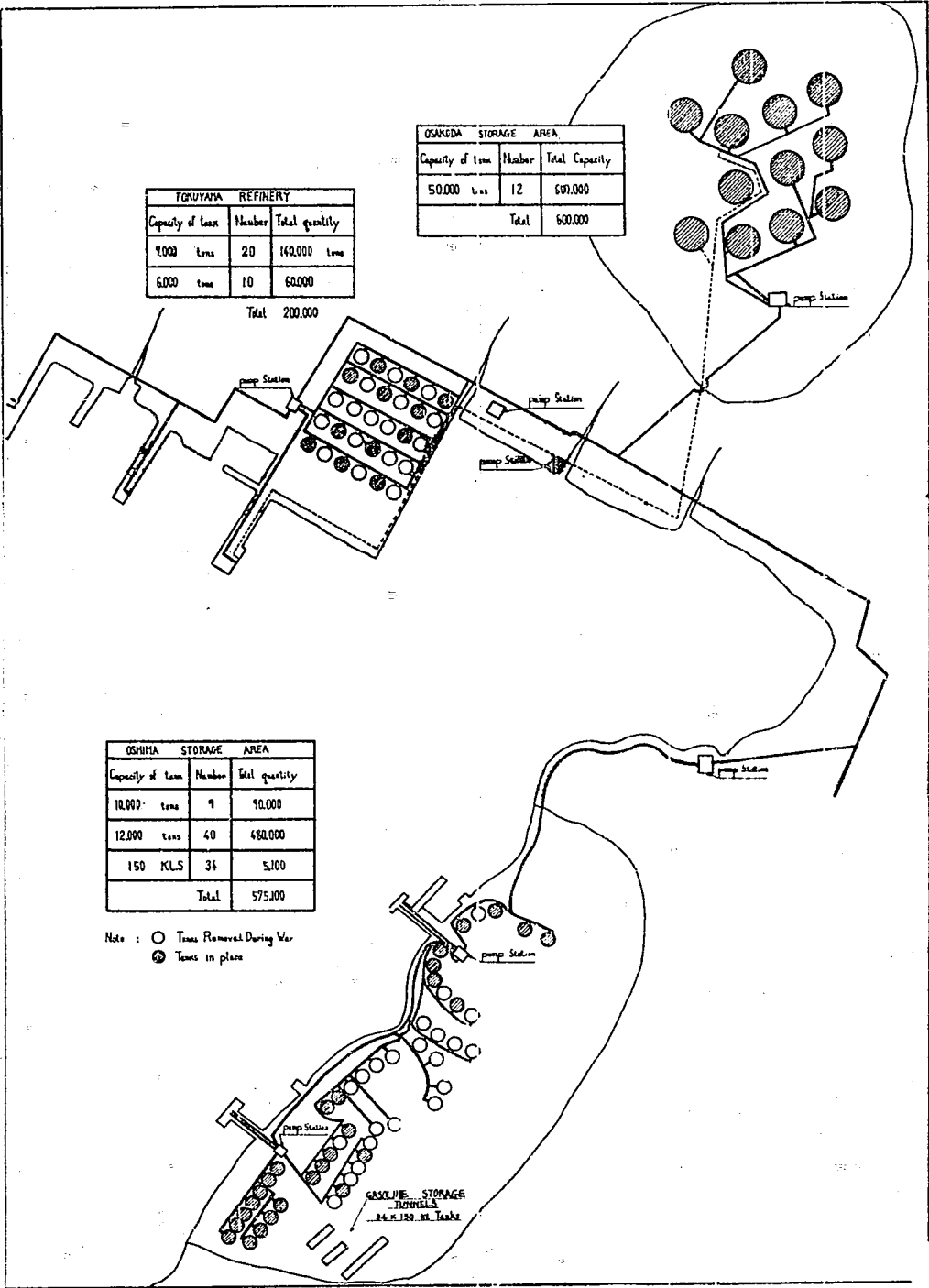


Figure 1 (F)
THIRD NAVAL FUEL DEPOT MAP
OF OIL TANK STORAGE AREAS

ENCLOSURE (F)

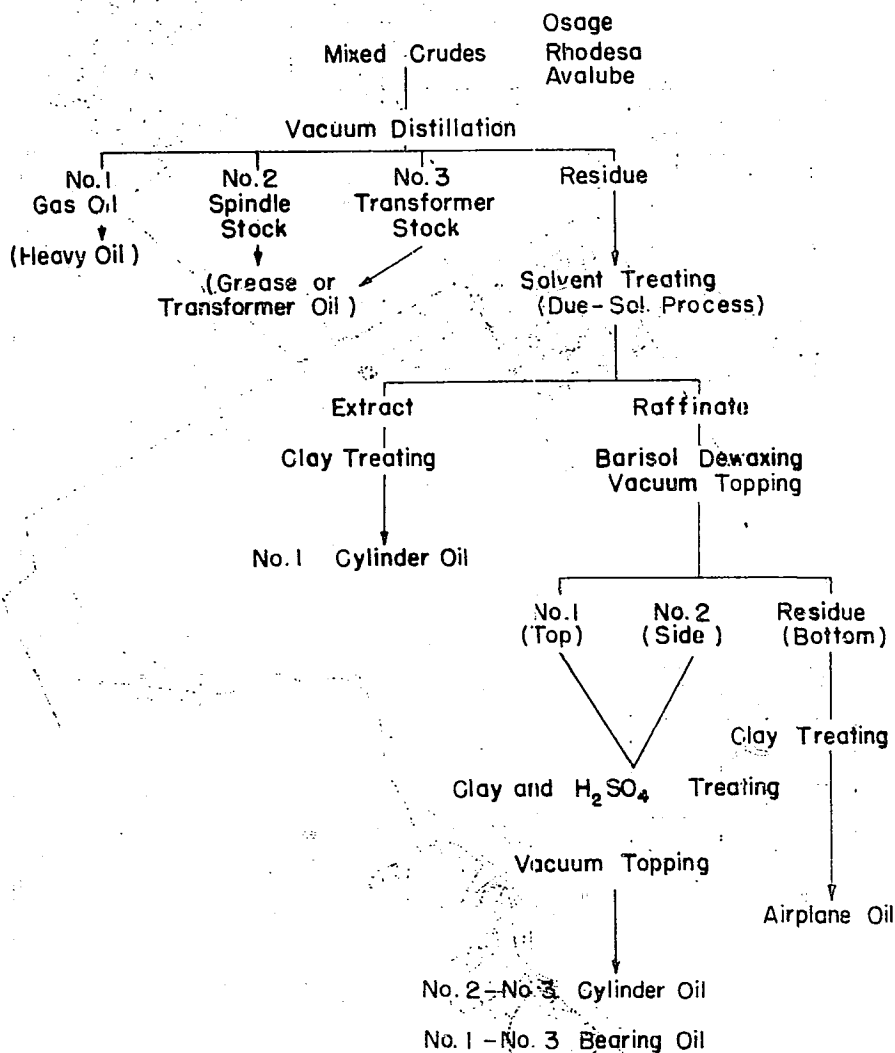


Figure 2 (F)
 FLOW SHEET OF LUBRICATING MANUFACTURE
 THIRD NAVAL FUEL DEPOT, TOKUYAMA

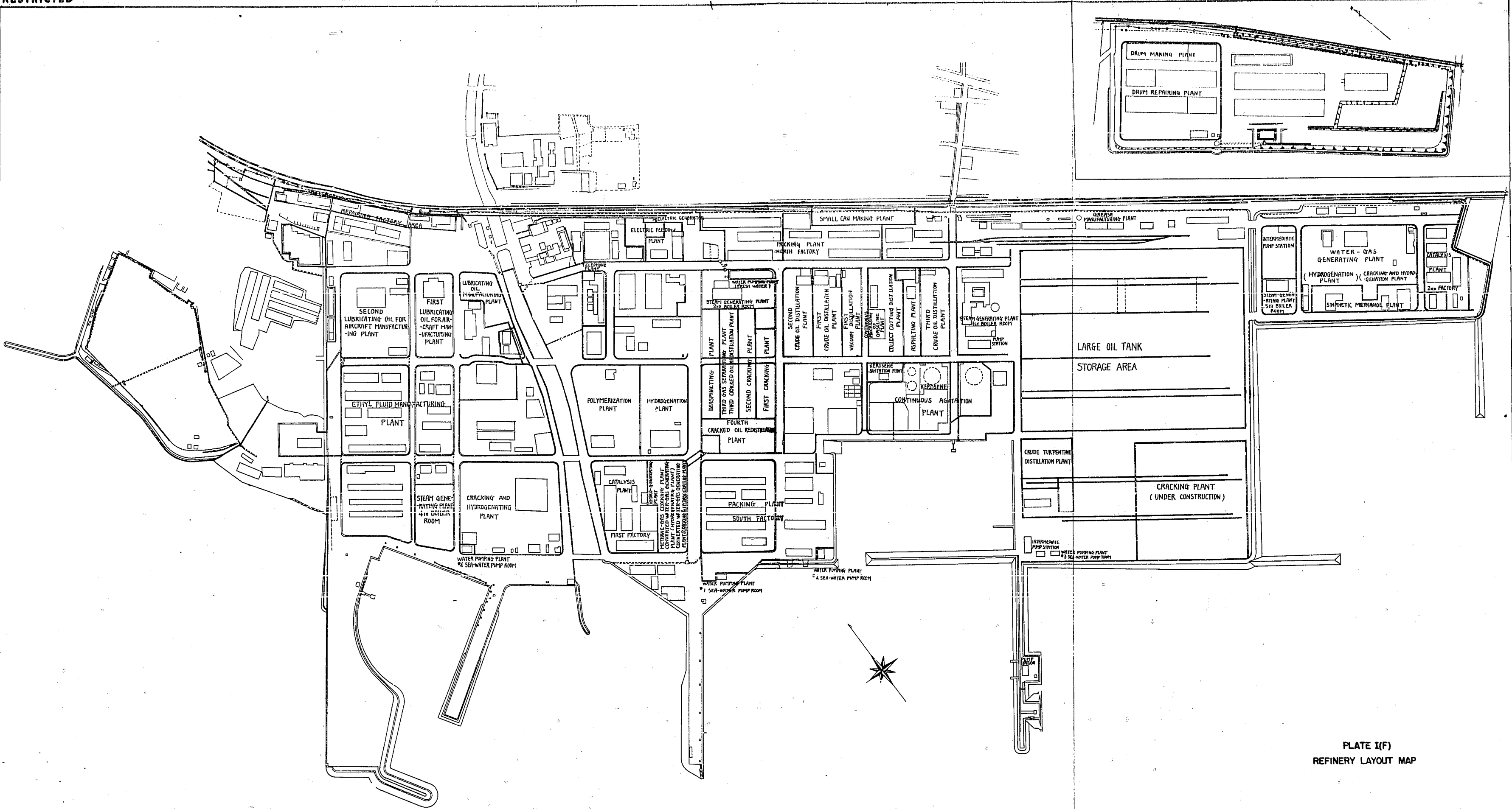


PLATE I(F)
REFINERY LAYOUT MAP

RESTRICTED

ENCLOSURE (G)

ENCLOSURE (G)

REPORT ON
THE NIPPON SEIRO K. K.
TOKUYAMA PLANT

ENCLOSURE (G)

TABLE OF CONTENTS

I. Introduction	Page 83
II. History and Organization	Page 83
III. Description of Process	Page 83
IV. Production and Quality	Page 84

LIST OF TABLES
AND ILLUSTRATIONS

Table I(G) Annual Production Totals at Nippon Wax, Ltd., Tokuyama Plant	Page 85
Table II(G) Typical Inspections on Products at Nippon Wax, Ltd., Tokuyama Plant	Page 85
Figure 1(G) Flow Sheet of Nippon Wax, Ltd., Tokuyama Plant	Page 86

ENCLOSURE (C)

I. INTRODUCTION

This report records and summarizes technical information of the Nippon Sairo K. K. (The Japan Paraffin Wax Refining Co., Ltd.) plant at TOKUYAMA, obtained by the Petroleum Section of NavTechJap on 30 October 1945. The following Japanese personnel were interviewed and assisted in supplying the information presented herewith:

Mr. M. TAMURA, General Manager.
Mr. H. KATAGIRI Head of General Affairs Dept.
Mr. Y. HAGA, Head of Manufacturing Dept.
Mr. S. KOBAYAKAWA, Head of Maintenance Dept.

The investigation of this plant was made primarily to establish its relation to the Japanese shale oil industry.

II. HISTORY AND ORGANIZATION

This plant was founded in 1929 to refine crude pressed wax imported from the Fushun Mining Corp. in Manchuria. Construction of the plant was completed in 1930. Total capitalization of the plant is 4,000,000 and all stock is held by the South Manchurian Railway Co. No other plants of this type are operated by S. M. R. in Japan.

The plant suffered one air raid, in which only two outlying storage tanks were hit and destroyed. Imports of oil made by shipping crude wax from FUSHUN to DAIREN by rail, and from DAIREN to TOKUYAMA by boat, were cut off about June, 1945, and at the time of the inspection, supplies of raw wax feed stock in storage were exhausted.

The total number of employees at the plant during the war was 240.

III. DESCRIPTION OF PROCESS

The feed stock at TOKUYAMA is crude pressed wax obtained from shale oil at FUSHUN by the following operations: The crude shale oil is distilled into a light gasoline cut(20%), a middle waxy cut(60%), and a residuum. The residuum is coked to yield an overhead distillate(about 60% on coke still charge)which is blended with the middle waxy cut. The blend is chilled and crude wax separated by filter-pressing. The yield of crude pressed wax is about 20% of Fushun crude shale oil.

A flow sheet of operations at TOKUYAMA is given by Figure 1(G). The crude pressed wax containing 35% oil is subjected to three successive chilling and dewaxing steps; warm dewaxing at 25°C, middle dewaxing at 15°C, and cold dewaxing at 0°C. The pressed wax from each step is acid treated with 1% by volume of 80° Be. sulphuric acid and sweated. The sweated waxes of different melting points are filtered over active clay prior to moulding and packaging. The hard pitch produced by distillation of the pressed oil from the middle dewaxing step, and the heavy pressed oil from the cold dewaxing step, are also marketed.

The chilling and pressing equipment is of conventional design, consisting of ammonia chilling tanks and 13 plate and frame filter presses, each with 70 plates one meter square. The sweating ovens were stated to have some novel features based on improvements of the original Czechoslovakian design. Each oven consisted of a tier of 10 horizontal rectangular troughs. The sides of the trough were about 6 inches high and the bottom was V-shaped so that the center of the trough was 12 inches deep. A wire screen (1/8 inch mesh) was placed at the 6 inch depth to support the wax. Twenty 1/2 inch pipes

ENCLOSURE (G)

at 4-5 inch spacing ran horizontally in the trough at the 3 inch level. The filling procedure was to fill the V-bottom with water, and then introduce melted wax in the top to fill the trough. After the wax solidified the water was drawn off, leaving the wax supported on the screen. Each trough holds one ton of wax. In the sweating operation, water at controlled temperature is circulated through the pipes, and sweated oil is withdrawn from the bottom of the V-troughs. The whole tier of troughs is placed in a closed room and heated air of controlled temperature is circulated by blowers. The length of the sweating operation is about 85 hours. Improvements made at this plant related mainly to the circulation and distribution of hot air to permit closely controlled temperature.

IV. PRODUCTION AND QUALITY

A summary of annual production totals is given in Table I(G). Typical inspections on each product are given in Table II(G). An estimated distribution of each product over the last three years follows:

Paraffin Wax

Army	5%
Navy	5%
Civil Government ..	90%

Heavy Oil

Navy	90%
Domestic Fuel.....	10%

Hard Pitch

Electrodes	50%
Domestic Fuel	50%

It was planned to divert a substantial proportion of the wax production for manufacture of lubricating oil at the second Naval Fuel Depot, YOKKAICHI.

ENCLOSURE (G)

Table I(G)
ANNUAL PRODUCTION TOTALS AT NIPPON WAX, LTD., TOKUYAMA PLANT*

Period	Crude Wax Charged	Products		
		Paraffin Wax	Heavy Oil	Pitch
Oct. 1940 Sept. 1941	21,979	12,585	6,202	238
Oct. 1941 Sept. 1942	20,630	11,387	6,750	238
Oct. 1942 Sept. 1943	21,616	11,505	7,051	204
Oct. 1943 Sept. 1944	14,752	7,375	6,086	234
Oct. 1944 Sept. 1945	8,380	4,017	3,655	213

* Metric Tons

Table II(G)
TYPICAL INSPECTIONS ON PRODUCTS AT NIPPON WAX, LTD., TOKUYAMA PLANT

Product	Specific Gravity	Melting Point(°C)	Flashing Point(°C)	Viscosity (Red W. No. 1)	Paraffin Content(%)
Paraffin Wax No. 1	(at 60°C) 0.784	60.0	225.0		
No. 2	0.782	55.0	210.0		
No. 3	0.780	50.0	198.0		
No. 44	0.778	45.0	188.0		
Heavy Oil	(at 15°C) 0.886	4.8	105.3	(at 30°C)	10.5
Hard Pitch		112.3			

ENCLOSURE (G)

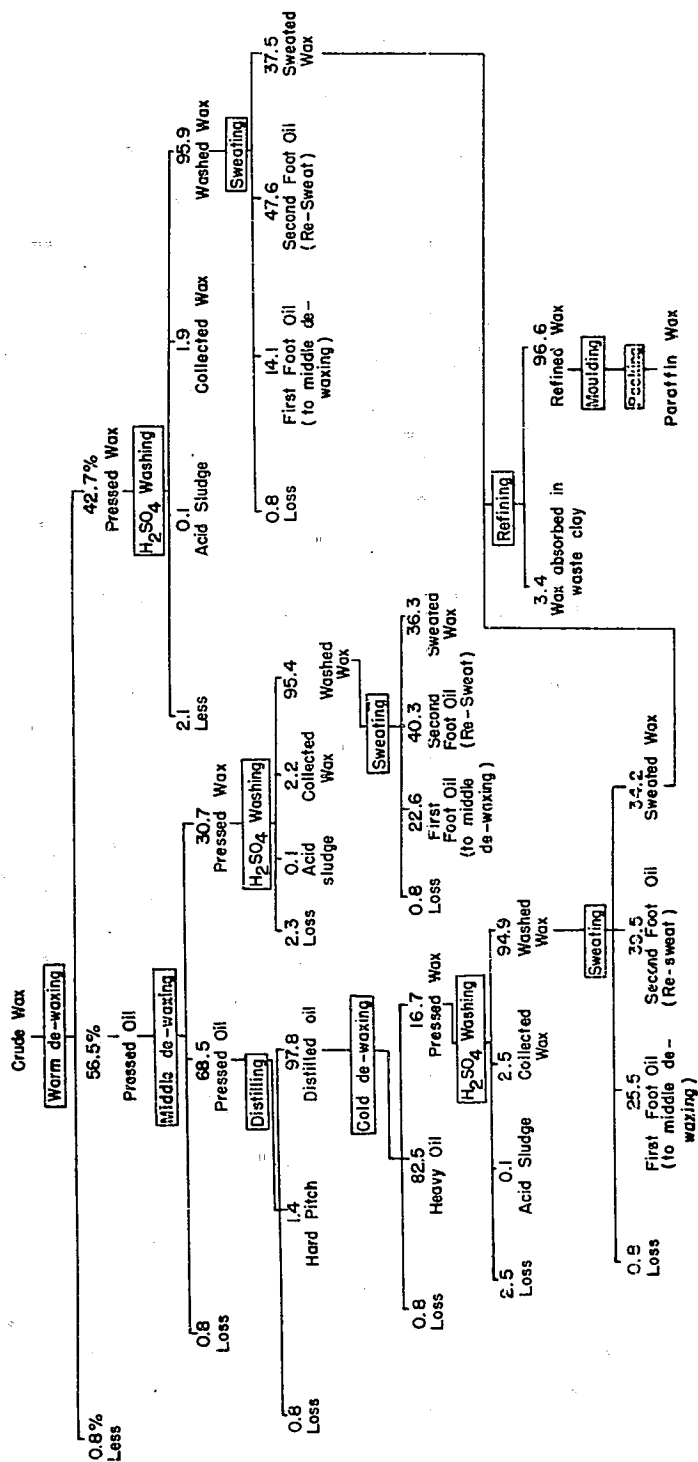


Figure 1(G)
FLOW SHEET OF NIPPON SEIRO K. K. TOKUYAMA PLANT