ENCLOSURE (B) #7

COAL HYDROGENATION IN SEMI-COMMERCIAL PILOT PLANT

by

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SUMMARY

Three to three and one-half tons per day of OYAMA coal and NAEBUCHI coal were treated in the semi-commercial hydrogenation pilot plant, and the results were as shown in Table I(B)17.

I. INTRODUCTION

In previous experiments, the hydrogenation of brown coal had been studied in an autoclave and in a small continous pilot plant with a charge capacity of 5 kg of paste per hour.

To obtain data for design and operation of industrial plants, the present experiments in a semi-commercial scale unit were undertaken at TOKUYAMA during the period from August 1938 to October 1939.

II. DETAILED DESCRIPTION

A process flow sheet of the semi-commercial coal hydrogenation unit used in these experiments is given in Plate I(B)17. Photographs of this unit are also enclosed.

Coal was crushed to 20-60 mesh, washed with 3% $ZnCl_2$ solution, and ground to 150 mesh in a tube mill. The powder was then made into a paste, in the tube mill, by mixing with heavy recycle oil (in the ratio 2:3) from the bottom of the high-temperature separator tower.

The paste was heated to 130°C in a heat-exchanger, to 250°C with hydrogen in the pre-heater, and then brought to the final reaction temperature by internal electric heaters in the reaction tower.

The reaction mixture was agitated with recirculated hydrogen. The reaction products were separated in the high-temperature separator into light oil and gas from top, and heavy oil from the bottom. The latter was separated from solid substances by centrifuge, and was recycled to the paste mill.

Coal and paste used in these experiments had the analysis shown in Table II(B)17. The reaction product from the top of the high-temperature separator was composed of gas, tar, and water, and the bottom was a mixture of tar and solid matter, the latter consisting of unchanged coal and ash. Experimental results are summarized in Table III(B)17.

III. CONCLUSIONS

OYAMA coal, from the South Manchuria Railway Co., and NAEBUCHI coal, from the SAKHALIN Artificial Oil Co., were hydrogenated at 410°C under 200 kg/cm^2 of hydrogen, in the presence of a $ZnCl_2$ catalyst.

Five days of continuous smooth operation were made on OYAMA coal, and one day on NAEBUCHI, and fairly good yields of oil were obtained, as follows:

	OYAMA (wt.%)	NAEBUCHI (wt.%)
Liquid yield from coal	77.9	77.0
Hydrogen consumed for coal	7.97	7.4
Yield of light oil (below 180°C)	3.0	4.0
Yield of middle oil (180-280°C)	40.0	40.0
Yield of heavy oil (above 280°C)	53.0	52.0

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Difficulties were encountered during operation in uniform mixing of paste with hydrogen in the reaction tower, and centrifugal separation of the solid residue from the liquid product of the bottom tar.

Table I(B)17
RESULTS OF SEMI-COMMERCIAL HYDROGENATION
OF
OYAMA AND NAEBUCHI

	OYAMA	NAEBUCHI
Liquefied (wt.%)	77.9	77.0
H_2 consumed / pure coal(wt.%)	7.97	7.4
Tar yield (kg/day)	244	226

Table II(B)17
ANALYSIS OF COAL AND PASTE

Coal		"A" OYAMA	"B" NAEBUCHI
Composition (wt. %)	Water	5.5	6.1
	Ash	7.0	11.5
	Fixed Carbon	46.4	38.1
	Volatile Matter	41.1	44.3
Calorific Value (cal)		7200	6814
Elementary analysis (wt %)	C	73.7	77.19
	H	5.7	6.24
	O (by diff.)	12.0	14.85
	S	0.5	0.37
	N	1.2	1.38
	H/C ratio	0.92	0.98
Paste Composition (wt %)	Carbon	39.2	42.7
	Ash	2.1	2.5
	Oil	51.9	52.3
	Water	6.8	2.5
Oil Used In Paste (wt %) (Distillation Test)	Below 280°C	16.0	
	280 - 320	18.5	
	Above 320	63.9	
	Water	0.7	
Fresh Hydrogen (vol. %)	CO_2	0.0	0.1
	CO	0.4	1.4
	H_2	93.7	91.8
	C_nH_{2n}	0.4	0.7
	C_nH_{2n+2}	1.0	1.0
	n	1.0	1.0

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Table III(B)17
EXPERIMENTAL RESULTS

		"A"	"B"
Conditions	Length of Run (hr.)	120	28
	Average Reaction Temp.	415°C	410°C
	Average Pressure (kg/cm^2)	200	200
	Fresh H_2 (m^3/hr)	185	162
	Circulated H_2 (m^3/hr)	1000	1000
	Paste (kg/hr)	282	265
	Total Paste (kg)	33,840	7,461
	Temp. of High-Temp. Separator		
	Top	365°C	336°C
	Bottom	375°C	341°C
Yields of Products	Product from Top (kg)	15,825	
	Tar	15,825	2,960
	Water	3,884	490
	Product from Bottom (kg)		
	Tar	16,753	3,800
	Water	67	23
	Solid Residue	1,908	380
Gas Analysis (vol.%)	CO_2	0.5	0.4
	CO	1.3	2.6
	H_2	81.0	79.1
	C_nH_{2n}	1.2	1.0
	C_nH_{2n+2}	10.2	10.8
	n	1.2	1.6
Properties of Light Oil	Density d_4^{30}	0.937	0.924
	Distillation Tests		
	I.B.P.	83°C	78°C
	below 180°C (wt %)	6.6	9.6
	180 - 280	50.0	48.3
	280 - 320	19.9	18.6
	above 320	24.5	21.9
	Water	1.0	1.3
Properties of Heavy Oil	Water	0.4	0.6
	Solid	9.0	10.0
	Distillation Tests		
	below 220°C (wt %)	0.7	2.0
	220 - 280	5.2	6.4
	280 - 320	10.1	13.6
	above 320	74.3	67.1

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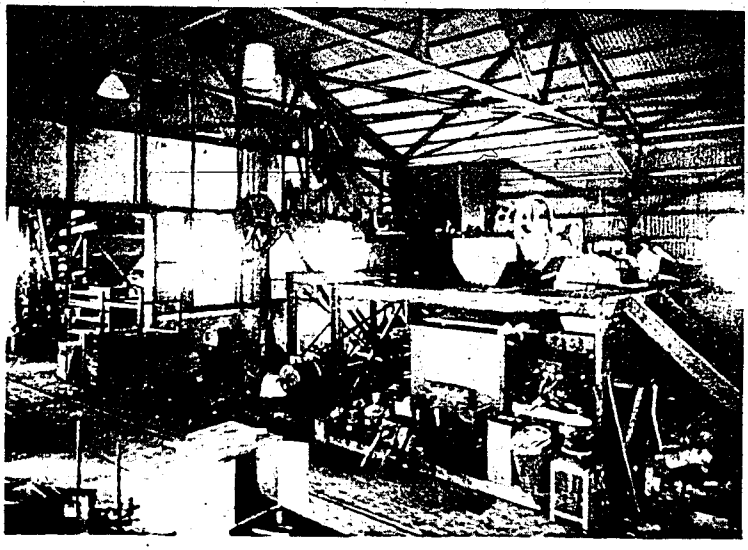

Figure 1(B)17
COAL WASHER

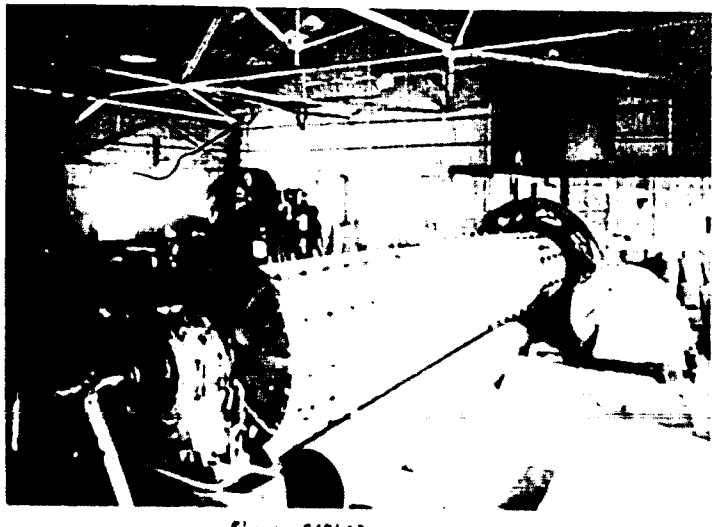

Figure 2(B)17
TUBE MILL FOR PREPARING PASTE

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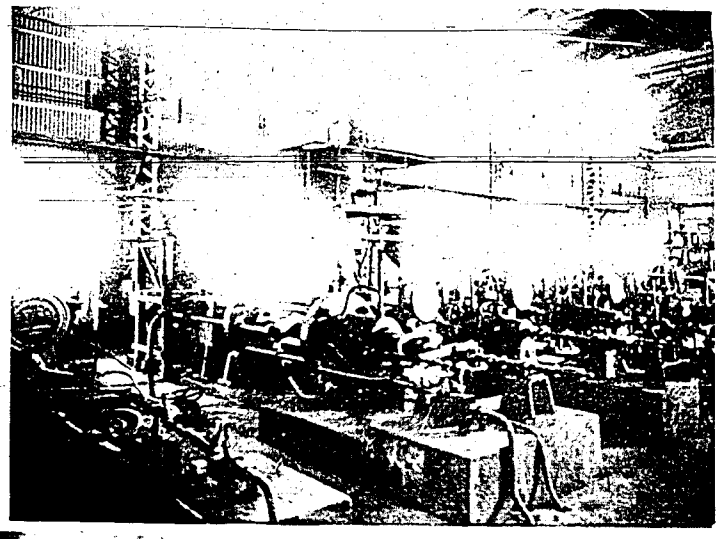

Figure 3(C)17
CONTROLLING PLATFORM
INCLUDING HIGH-PRESSURE
CHARGING PUMP

Figure 4(b)17
REACTION CHAMBER OF
COAL HYDROGENATION

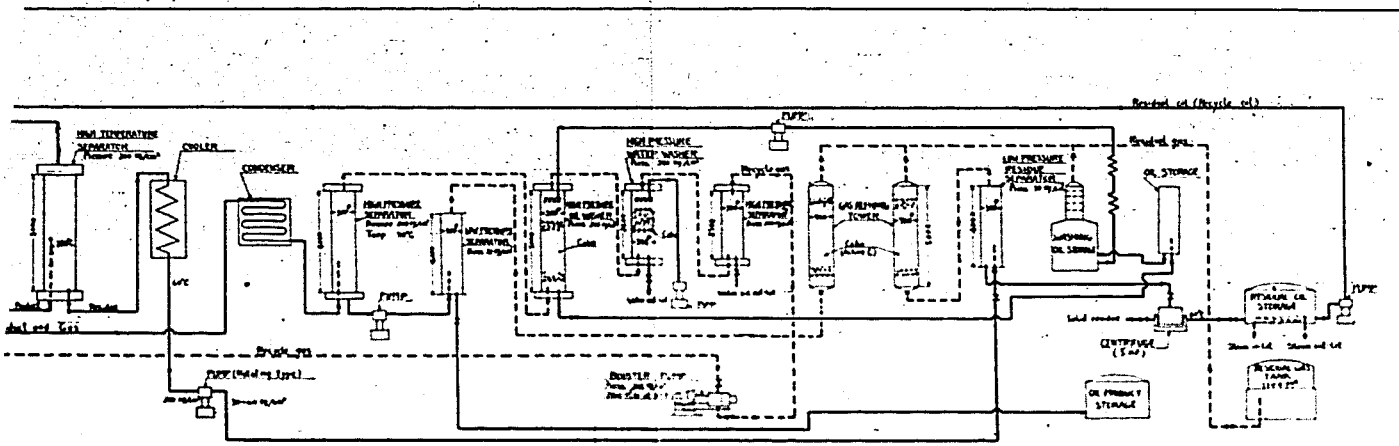

FIG. 1

FLOW CHART OF SEMI-COMMERCIAL COAL HYDROGENATION PILOT PLANT

The Naval Fuel Depot

Prepared for U.S. Naval Technical Mission to Japan

Date	Drawn by
15 Dec 1945	A. Stone

PLATE I(B)17
FLOW CHART OF SEMI COMMERCIAL COAL
HYDRO GENATION PILOT PLANT
(THE 1st NAVAL FUEL DEPOT)

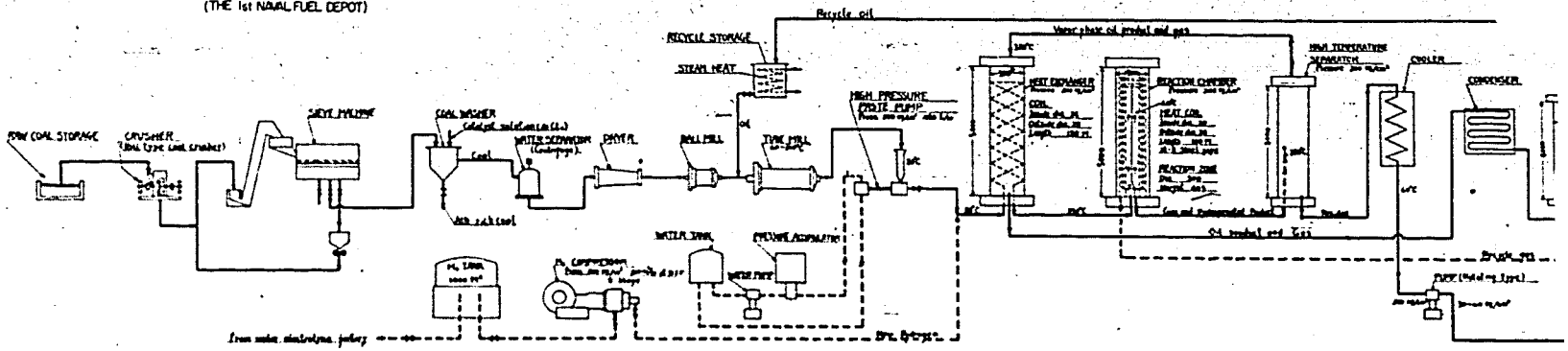

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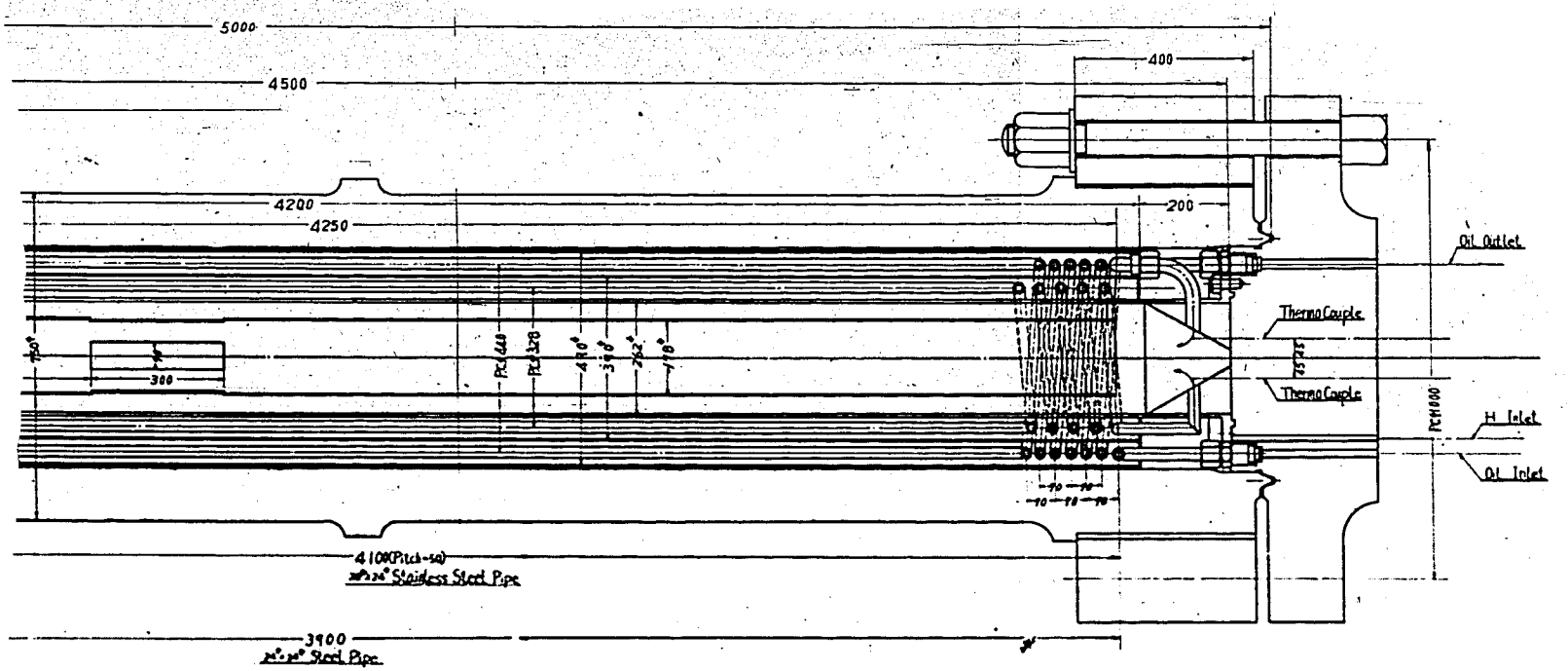
PLATE II(B)17

CROSS SECTION OF REACTION CHAMBER OF 07 TYPE EXPERIMENTAL APPARATUS

(TOKUYAMA NAVAL FUEL DEPOT)



TOKUYAMA NAVAL FUEL DEPOT

CROSS SECTION OF REACTION CHAMBER OF 07 TYPE EXPERIMENTAL APPARATUS

DEC 19TH 1940

RESTRICTED

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PLATE II(B)17

CROSS SECTION OF REACTION CHAMBER OF O7 TYPE EXPERIMENTAL APPARATUS

(TOKUYAMA NAVAL FUEL DEPOT)