ENCLOSURE (B) 7

THE DESIGN AND OPERATION OF HYDROGEN PEROXIDE CONCENTRATION PLANTS AT THE FIRST NAVAL FUEL DEPOT

by

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LIST OF TABLES AND ILLUSTRATIONS

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SUMMARY

Hydrogen peroxide concentration plants were designed according to the following conditions:

Feed stock .... 30 wt. % H_2O_2
Product .... 82 wt. % H_2O_2

Product Output

Type 1 plant* ..... 100 tons per month design capacity.

Type 2 plant ...... 250 tons per month design capacity.

* Two units of this type were installed at the First Naval Fuel Depot.

It was planned to install this type at the Second Naval Fuel Depot and at other points, but construction was not started.

A pilot plant (TypeI) was erected at OFUNA. The product of these plants was to be used for SHUSUI, a rocket airplane. Pure 82% concentration hydrogen peroxide of sufficient quality for SHUSUI was obtained continuously.

I. INTRODUCTION

A. History of Project

The design and erection of H_2O_2 concentration plants was ordered by the Naval Supplies Bureau (Gunjukyoku) in August 1944.

A flask type concentration plant was first installed at OFUNA. A drawing of one unit of this plant is shown by Figure 3(B)7. This plant started to operate in October 1944 and was shut down in December 1944. It was equipped with 1000 5 - liter flasks and had a total capacity of 30 tons per month of 82% H_2O_2.

The first continuous plant started its operation in November 1944 and produced about 30 tons of 82% H_2O_2 per month. The design capacity of the plant was 100 tons per month, but the mist separators had not been installed and actual output was only 30 tons/month. The plant was constructed by the Hitachi Co. Ltd. (IBARAGI Prefecture). 30% H_2O_2 was shipped in from the Edogawa Co. at YAMAKITA and the Sumitomo Co. at OSAKA.

The second continuous plant, also Type I, was completed in August 1945, but it did not begin functioning.

B. Key Research Personnel Working on Project

Nav. Chem. Eng. T. SHIBAZAKI
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Nav. Chem. Eng. S. SHINODA
Chem. Eng. Lt. Comdr. Y. YAMAMOTO

II. DETAILED DESCRIPTION

A. The Plant

A process flow chart for Type I plant is given by Plate I(B)7. Type II plant is similar in design.

Each plant has four preliminary rectifying columns and one column for the final rectification. The columns and condensers are all inter-changeable. The preliminary rectifying step serves chiefly to remove impurities included in the feed stock to prevent possible violent decomposition of concentrated H_2O_2 in the final rectification.

A mist separator is essential to remove impurities carried from the retort with the H_2O_2 vapor, and to increase the purity of the intermediate product. If a sufficiently pure intermediate product is not obtained, the H_2O_2 in the final rectifying retort can not be concentrated to above 80%, and the yield is decreased due to decomposition.

B. Construction Materials

Construction materials used in the continous plants were as follows:

1. Retorts, mist separators, rectifying columns, Raschig-rings, vacuum receivers and piping used over about 30°C .... Porcelain.

2. Condenser tubes .... Aluminium.

3. Heating coil in retort .... 18-8 Cr-Ni Steel (The surface of the coil is thoroughly polished).

4. Intermediate product and product coolers, storage tanks, and tubes used under about 30°C .... Tin.

5. Gaskets for porcelain pipes .... Low sulphur rubber rings covered with tin plate.

6. For high temperature and large diameter parts, such as the retort covers, mist separators, and rectifying columns, smooth ground joints were adopted successfully.

C. Material and Heat Balances

The material and heat balances were calculated on the basis of the following assumptions:

Operating period per month .... 75%, or about 23 days.
Loss of H_2O_2 by decomposition and leakage .... zero.
Loss of heat from the surface of retorts, columns, etc. .... zero.

1. Material Balances

a. Overall Plant. Basis: 500 kg/hr charge of 30% H_2O_2.

Input

30% H_2O_2 .... 4 columns x 125 = 500 kg/hr

Output

83% of H_2O_2 .... 181 kg/hr
Distilled water .... 4 columns x 68.2 46.2 = 319 kg/hr
500 kg/hr

b. Preliminary Rectifying Column

Input

30 % of H_2O_2 .... 4 columns x 125 = 500 kg/hr

Output

66% of H_2O_2 .... 4 columns x 56.8 = 227.2 kg/hr
Distilled water .... 4 columns x 68.2 = 272.8 kg/hr
500.0 kg/hr

c. Final Rectifying Column

Input

66% of H_2O_2 .... 227.2 kg/hr

Output

83% H_2O_2 .... 181 kg/hr
Distilled water .... 46.2 kg/hr
227.2 kg/hr

2. Heat Balances

a. Overall Plant. (See Tables I(B)7 and II(B)7.

b. Preliminary Rectifying Columns. (See Tables III(B)7 and IV(B)7).

c. Final Rectifying Column. (See Tables V(B)7 and VI(B)7).

D. Equilibrium Data

Pressure-boiling point relations of H_2O_2 and vapor-liquid equilibrium relations of H_2O and H_2O_2 are given by Figure 2(B)7 (based on International Critical Tables) and Figure 1(B)7 (Canadian Journal of Research, 1940).

E. Product Specification and Inspections

Tentative specifications of product: (see Table VII(B)7) Stability: Decomposition must ve less than 10% when heated to 96°C for 24 hr (amount of stabilizer, 8-oxyquinoline 0.3 gm/lit, sodium pyrophosphate ($Na_4P_2O_7.10H_2O$) 0.1 gm/lit). Ignition residue: When heated to 800°C, the residue must be less than 70 mg/lit. Acidity: The H_2SO_4 content must be less than 100 mg/lit.

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Table I(B)7
INPUT

Material	kg/hr	Temp. (°C)	Kcal/hr
30% H_2O_2	4 x 125 = 500	0	
Steam to retorts Preliminary rect.	4 x 127 = 508	Only latent heat calculated	4 x 66.95 x 10^3 -207 - 3 x 10
Final rect. (1kg/cm²)	117	Only latent heat calculated	61.9 x 10^3 329.7 x 10^3

Table II(B)7
OUTPUT*

Material		Weight (kg/hr)	Temp. (°C)	Kcal/hr
83% H_2O_2		181	20	2.36x10^3
Distilled water	Preliminary rect. column	4x68.2=272.8	29	4x1.98x10^3 = 7.92x10^3
	Final rect. column	46.2	29	1.34x10^3
Cooling water	Partial condenser	(4x2.59+3.28)x10^3 = 13.64x10^3	16-25	(4x23.3+29.5)x10^3 =122.7x10^3
	Total condenser	(4x4.39+2.98)x10^3 = 20.54x10^3	16-25	(4x39.5+26.8)x10^3 =184.8x10^3
	Intermediate product cooler	4x0.675x10^3=2.70x10^3	16-18	4x1.35x10 = 5.4x10^3
	Product cooler	2.60x10^3	16-18	5.18x10^3 329.70x10^3

*Note: Total Steam Consumption: 508-117= 625kg/hr.

Total Cooling Water Consumption: 13.64+20.54+2.60=39.48 ton/hr

ENCLOSURE (B)7

Table III(B)7
INPUT

Material	Kg/hr.	Temp. (°C)	Kcal/hr.
30% H_2O_2	4x125=500	0	0
Steam to retorts (1kg/cm²)	4x127=508	Only latent heat calculated	$4x66.95x10^3=267.8x10^3$ $267.8x10^3$

Table IV(B)7
OUTPUT

Material	Kg/hr	Temp. (°C)	Kcal/hr
66% H_2O_2	4x56.8=227.2	20	$4x0.82x10^3=3.28x10^3$
Distilled Water	4x68.2=272.8	29	$4x1.98x10^3=7.92x10^3$
Cooling Water			
Partial Condenser	$4x2.59x10^3=10.36x10^3$	16-25	$4x23.3x10^3=93.2x10^3$
Total Condenser	$4x4.39x10^3=17.56x10^3$	16-25	$4x39.5.10^3=158.0x10^3$
Intermediate Product cooler	$4x0.675x10^3=2.7x10^3$	16-18	$4x1.35x10^3=5.4x10^3$ $267.80x10^3$

Table V(B)7
INPUT

Material	Kg/hr	Temp. (°C)	Kcal/hr
66% H_2O_2	181	20	$3.28x10^3$
Steam to retort (1kg/cm²)	46.2	29	$61.9\ x10^3$ $65.18x10^3$

ENCLOSURE (B)7

Table VI(B)7
OUTPUT

Material	Kg/hr	Temp. (°C)	Kcal/hr
83% H_2O_2	181	20	2.36×10^3
Distilled water	46.2	29	1.34×10^3
Cooling water			
Partial condenser	3.28×10^3	16-25	29.5×10^3
Total condenser	2.98×10^3	16-25	26.8×10^3
Product cooler	2.60×10^3	16-18	5.18×10^3
			65.18×10^5

Table VII(B)7
TYPICAL PRODUCT AND FEED STOCK, INSPECTIONS

	Feed stock	Product
Conc. of H_2O_2	32%(wt)	82% (wt)
Stability	5% (Amount of decomp. at 80°C, 6 hr)	7%
Ignition residue	(Spec.:Less than 300 mg/l)	45 mg/l
Acidity	50 mg/l	90 mg/l

ENCLOSURE (B)7

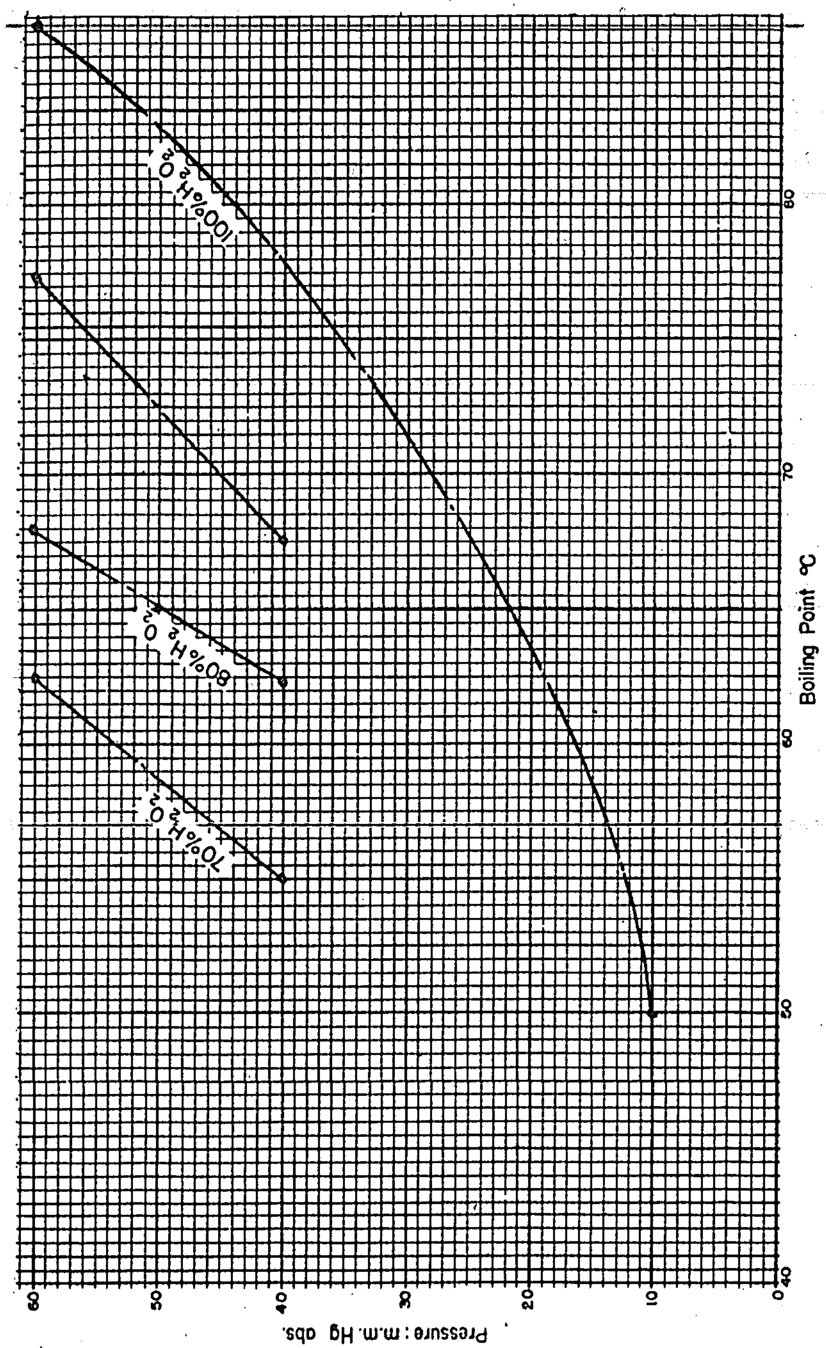


Figure 1 (B)7

PRESSURE-BOILING POINT RELATIONS AT CONSTANT CONCENTRATIONS OF H_2O_2

ENCLOSURE (B)7

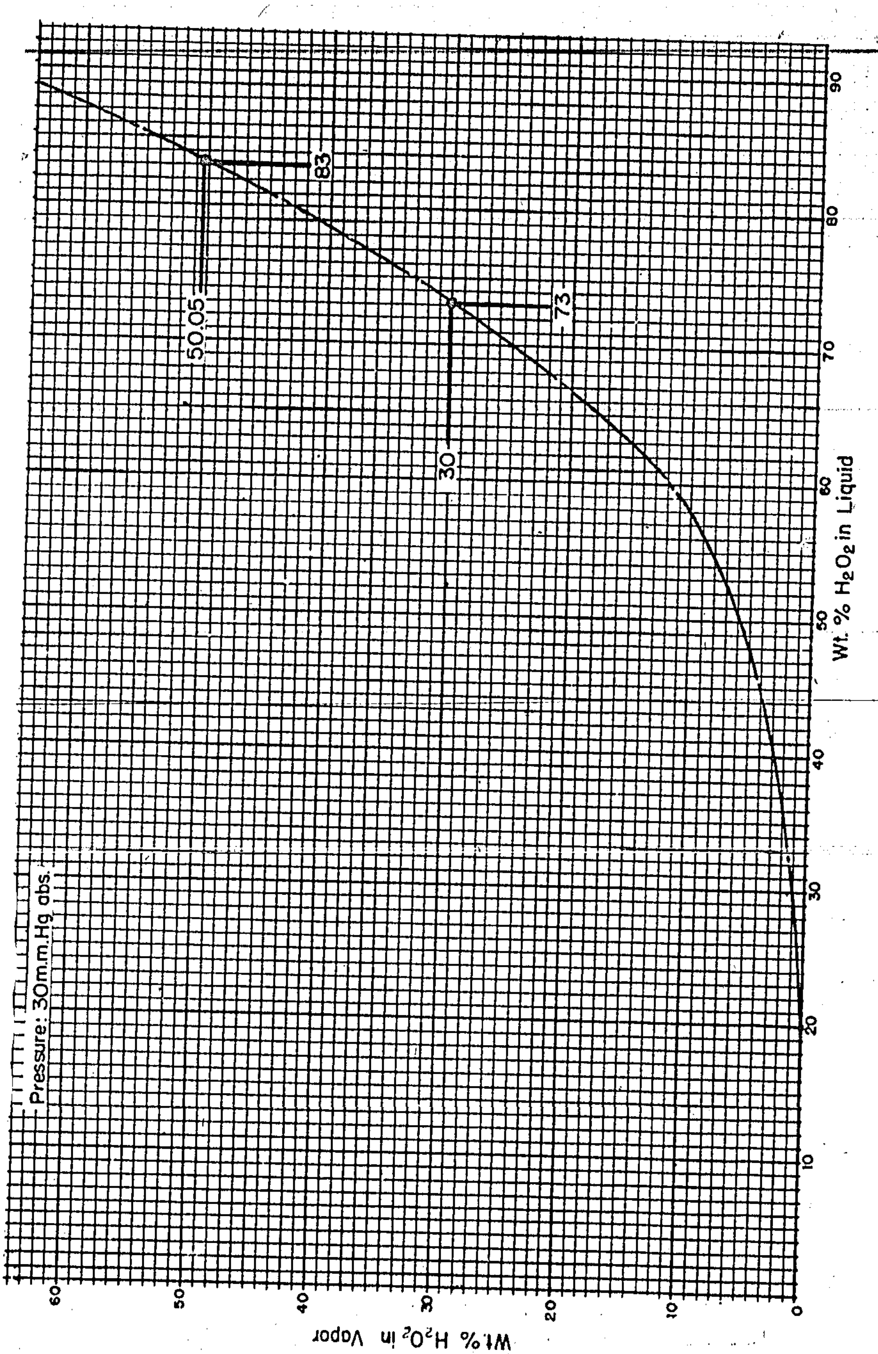
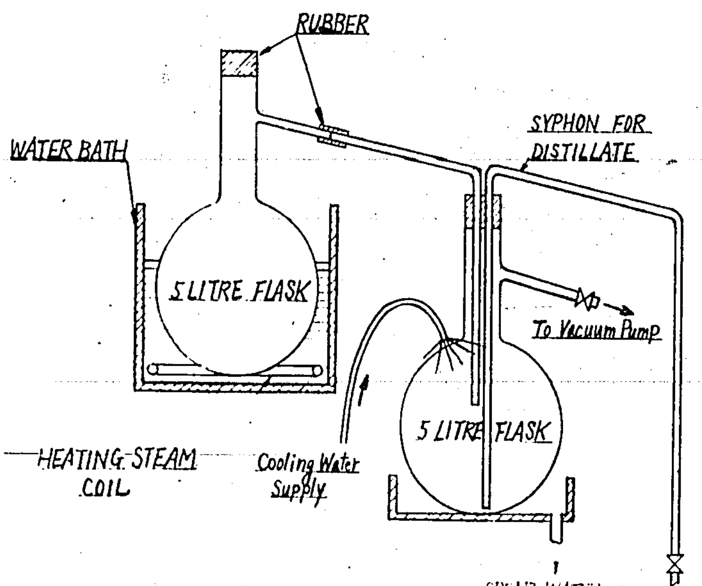


Figure 2 (B)7
VAPOR-LIQUID EQUILIBRIUM RELATIONS AT CONSTANT TOTAL PRESSURE FOR MIXTURES OF H_2O AND H_2O_2

ENCLOSURE (B)7



Work Condition

Charge	3 Lit.	32% H_2O_2
Product	3/4 Lit.	82% H_2O_2
Distillate	2 1/4 Lit.	10% H_2O_2
Vacuum		80 mm Hg
Temperture		85 °C Max.

Figure 3 (B)7
FLASK PLANT UNIT

ENCLOSURE (B)7

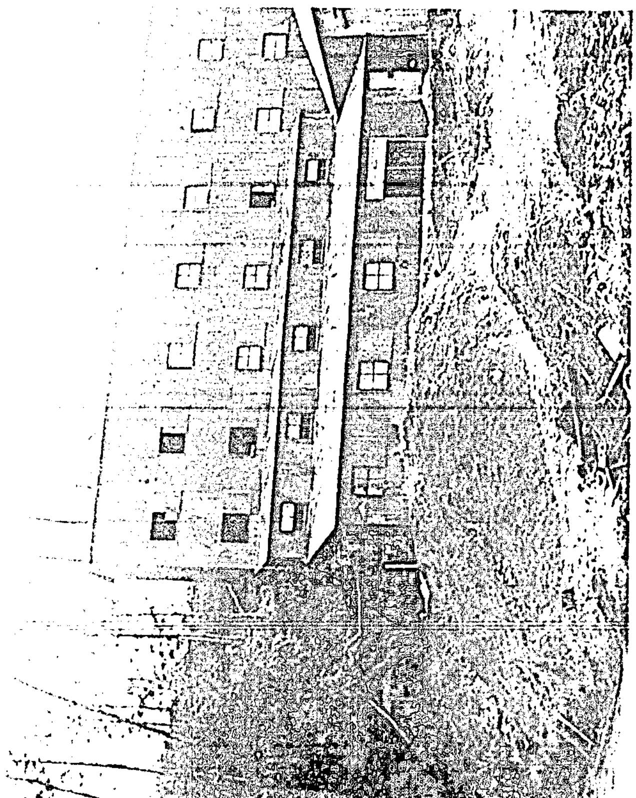


Figure 4 (B)7
HYDROGEN PEROXIDE CONCENTRATION APPARATUS
1. GENERAL VIEW

ENCLOSURE (B)7

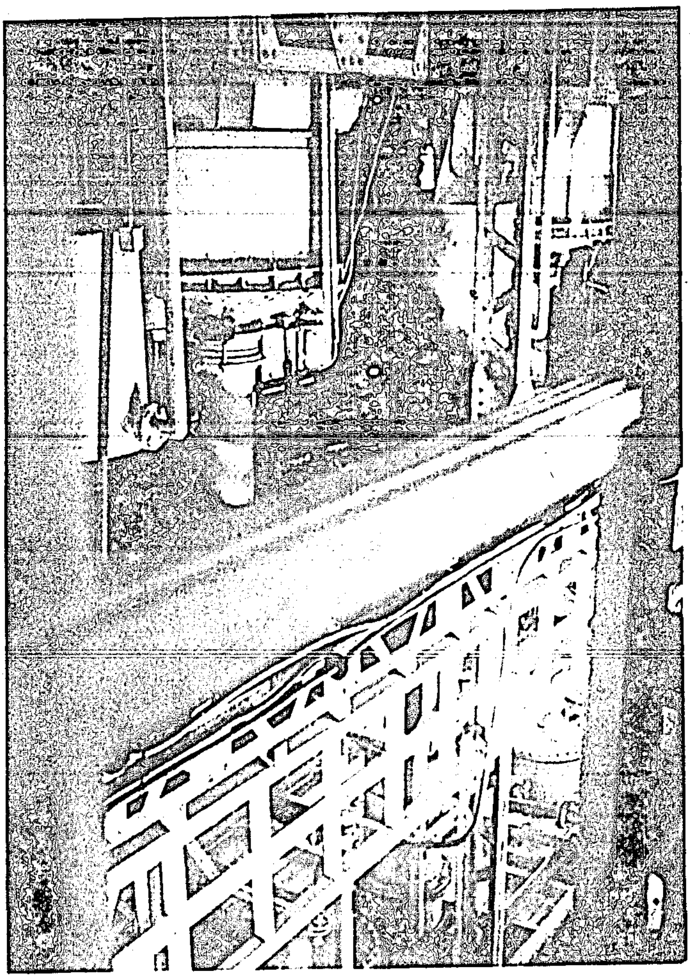


Figure 5 (B)7
HYDROGEN PEROXIDE CONCENTRATION APPARATUS
2. DETAIL OF THE CONCENTRATION TOWER

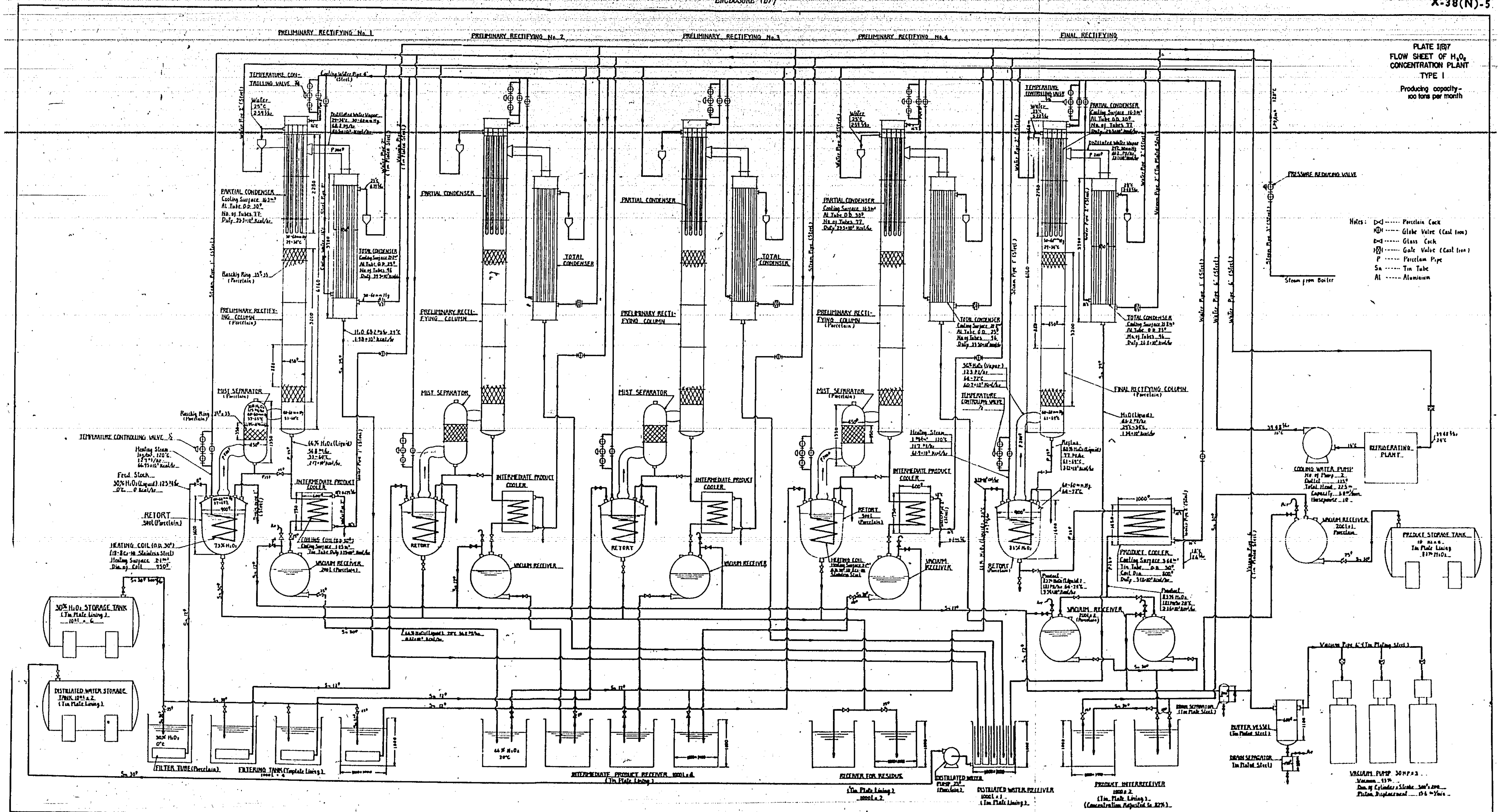


PLATE I(B)7
FLOW SHEET OF H_2O_2
CONCENTRATION PLANT
TYPE I
Producing capacity-
100 tons per month
PRELIMINARY RECTIFYING No. 1
PRELIMINARY RECTIFYING No. 2
PRELIMINARY RECTIFYING No. 3
PRELIMINARY RECTIFYING No. 4
FINAL RECTIFYING
PARTIAL CONDENSER
TOTAL CONDENSER
PRELIMINARY RECTIFYING COLUMN (Porcelain)
FINAL RECTIFYING COLUMN (Porcelain)
MIST SEPARATOR
INTERMEDIATE PRODUCT COOLER
RETORT
VACUUM RECEIVER
TEMPERATURE CONTROLLING VALVE
HEATING COIL
PRODUCT COOLER
REFRIGERATING PLANT
COOLING WATER PUMP
PRODUCT STORAGE TANK
PRESSURE REDUCING VALVE
Steam from Boiler
BUFFER VESSEL (Tin Plated Steel)
DRAIN SEPARATOR (Tin Plated Steel)
VACUUM PUMP
30% H_2O_2 STORAGE TANK (Tin Plate Lining)
DISTILLATED WATER STORAGE TANK (Tin Plate Lining)
FILTER TUBE (Porcelain)
FILTERING TANK (Tinplate Lining)
INTERMEDIATE PRODUCT RECEIVER (Tin Plate Lining)
RECEIVER FOR RESIDUE (Tin Plate Lining)
DISTILLATED WATER PUMP
DISTILLATED WATER RECEIVER (Tin Plate Lining)
PRODUCT INTERRECEIVER (Tin Plate Lining) (Concentration Adjusted to 82%)
Notes: Porcelain Cock; Globe Valve (Cast Iron); Glass Cock; Gate Valve (Cast Iron); P ---- Porcelain Pipe; Sn ---- Tin Tube; Al ---- Aluminium