

ENCLOSURE (B) 4

DESIGN STUDIES ON THE SIMPLIFIED
TREATING PROCESS FOR PINE ROOT OIL

by

NAV. CHEM. ENG. LT. COMDR. S. SANKA

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SUMMARY

~~Design studies were made of a process to make aviation gasoline from pine root oil.~~ This process has three steps. The 1st step is distilling and coking of pine root oil. The next is the catalytic reforming of the gasoline fraction. The 3rd step is redistillation of the reformed oil.

Batch type operation is employed for each step. Photographs and detailed blue-prints of each unit are attached.

I. INTRODUCTIONA. History

Carbonization retorts were already in operation in our country, but were widely scattered. In consequence, large central pine root oil refineries were not practical due to transportation difficulties, and design work was started on apparatus to refine the oil on the spot.

In April 1945, orders were given to study this project. Since reliable data was not available, we constructed a pilot plant based on common sense principles and chiefly studied how to operate this apparatus.

B. Key Research Personnel Working on Project

Nav. Eng.	T. SHIBAZAKI
Nav. Chem. Eng. Lt. Comdr.	S. SANKA
Nav. Chem. Eng. Lt.	T. YOKOYAMA
Nav. Chem. Eng. Lt.	T. UEDA
Nav. Chem. Eng. Lt.	R. SUSUKI
Nav. Chem. Eng. Lt.	Y. MOTOYAMA
Nav. Sub-Eng.	M. KUBOTA

II. DETAILED DESCRIPTIONA. Distilling and Coking Apparatus

1. Test Procedure. A diagram of the distilling and coking apparatus is shown in Plate I(B)4. In this apparatus crude pine root oil is fractionally distilled into light and heavy cuts. The volume of the batch still is 1.2 kl and the volume of pine root oil charged is 0.75 kl. The relation between yield of oil, temperature of retort, and time is given in Figure 2(B)4. It is necessary to control firing carefully in oil temperature range of 90°C to 110°C to avoid sudden foaming of oil due to occluded water. After water is distilled over, the firing rate is increased to approximately 20kg/hr of wood. When distillation of the oil is completed, the retort is allowed to cool for 2-3 hours until temperature of retort becomes less than 120°C.

The coke is removed from the bottom of the retort every 4 runs. When the crude pine root oil contains more than 7% of water, the required time per cycle becomes very long. Accordingly, when the crude oil has more than 7% of water, it is settled about 3.5 hours at 60-70°C. By this treatment, the water content is reduced to less than 4%.

The first cut is taken at 185°C still temperature and is used as feed for the catalytic reforming unit. It was planned to use the

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distillate above 185°C as feed for catalytic cracking.

2. Experimental Results. Experimental results are given in Table I(B)4.

B. Simplified Catalytic Reforming Apparatus

1. Test Procedure. A diagram of this apparatus is shown in Plate II(B)4. 0.5 liters of the gasoline fraction (185°C end point) obtained from the "distilling and coking apparatus" is charged into a batch still, (1.2 kl Volume) and distilled by direct wood firing.

The oil vapors are passed through the reaction chambers, filled with activated Japanese clay catalyst. The reaction conditions are about 450°C and atmospheric pressure.

After many experiments the operating curve of Figure 3(B)4 was obtained. This apparatus has two firing grates, one on the still pot side and the other on the reaction chamber side, as shown in Figure 3(B)4. The reaction chambers are fired first, until the catalyst temperature reaches about 200°C, then firing of the still pot is started. After about 2 hours the oil vapors pass downward through the two parallel reaction chambers in which temperature has increased to 300°C. The catalyst size is 6mm(diameter) X 6mm(length) and is random packed, 25kg in each chamber. When catalytic action begins, the temperature T₂ suddenly rises to 550-600°C. By controlling rate of firing and valve openings the temperature is regulated to 450°C.

The vaporization of oil is continued for 8 hours at the rate of about 60 lit/hr. If the firing rate on the evaporator side is too high, the oil flow is too great and the temperature of catalyst cannot be maintained at 450°C. The fire man must have experience to acquire the proper technique for maintaining a uniform reaction temperature. The final still pot temperature is about 230°C. The valves connecting catalyst and oil chamber are then closed. About 8 liters of water are added to each reactor, forming steam which blows down the lines. Catalyst temperature is about 300°C and air, at atmospheric temperature and pressure of 500mm Hg, is blown through the catalyst. The air blow is about 500m³/hr for both retorts. If the blower capacity is less than this, not only is the steel shell of the reaction chamber often burnt out by the reaction heat, but the catalyst also loses activity. The air flow is continued for about 35 minutes and catalyst temperature increases to 650°C maximum. Eight liters of water are added again to decrease the temperature. This cycle is repeated 4 times (refer to Figure 3(B)4). After this, the temperature of catalyst increases gradually due to the natural flow of air. The still pot is allowed to cool, and when the temperature is 100°C fresh oil is charged. The fuel consumption on each side is about 12-15 kg/hr and the total fuel consumption in one cycle is about 300 kg of wood.

2. Experimental results are given in Table II(B)4.

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C. Re-Distilling Apparatus

1. Test Procedure. A diagram of this process is given by Figure 1(B)4. This apparatus is used to refine the reformed oil from the catalytic reforming apparatus. ~~The batch still itself is the same as in the simplified catalytic reforming unit.~~ The distillation tower is packed with clay Raschig rings, 3- X 30mm in size.

This operation is very simple and is not reported. Oil is distilled at the rate of 75 lit/hr and the fuel consumption is 15 kg/hr.

2. Experimental Results. Experimental Results are given in Table III(B)4.

Table I(B)4
OPERATION AND PRODUCTS OF PINE ROOT STILL

	Fraction	Engler Distillation	Distillate from Pilot Retort
Yield of Product	Below 185°C	20% (Vol)	20% (Vol)
	185-300	28	25
	Above 300	{42 (as residue)	30
	Cokes		5
	Water		5
Gas and Loss	7	15	
Operating Time			9.5 hr/run
Capacity of Still			0.75 kl/run
Fuel Consumption			180 kg(wood)/run
Operators Required			2 men
Discharge of Cokes			one discharge/4 runs

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Table II(B)4
GASOLINE FROM CATALYTIC REFORMING

		Charge Oil	Reformata
Specific Gravity		0.86	0.85
Dist Temp. (°C)	1st Drop	110	70
	10%	140	135
	50%	170	165
	97%	190	200
	Dry Point	250	280
Octane Value	0% T.E.L.	70	85
	0.15% T.E.L.	75	90

Yield of Reformed Oil

Reformed Oil 80% (vol)
 Gas and Loss 20%
 Space Velocity of Oil 1 vol oil/vol cat/hr
 Reaction Temperature 450°C

Operating Time

Catalytic Reforming 8 hr
 Reactivation of Catalyst 3 hr
 Charge and Others 5 hr
 Total 16 hr

Table III(B)4
REDISTILLATION OF REFORMED OIL

	Before Treatment	After Treatment
1st Drop	70°C	70°C
10%	135	130
50%	160	160
97%	200	190
Dry Point	280	250
Color	deep green	light brown

Operating Time 10 hr/run
 New Charge 0.75 kl/batch
 Yield of Oil
 Refined Oil 80% (vol)
 Residue 20%
 Fuel Consumption 150 kg/run

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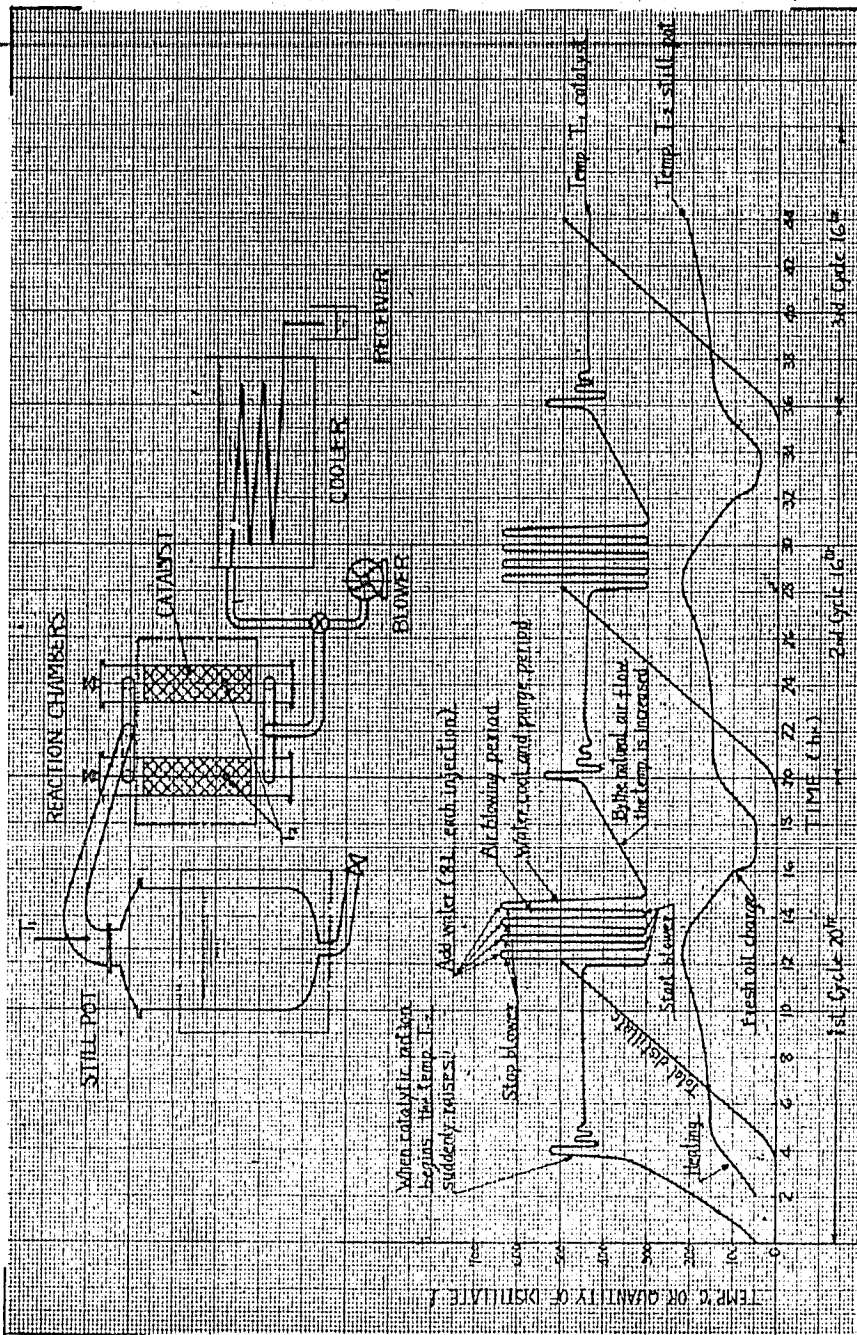


Figure 2(B)4
RELATION BETWEEN YIELD, TEMP. AND TIME FOR DISTILLATION OF CRUDE PINE ROOT OIL

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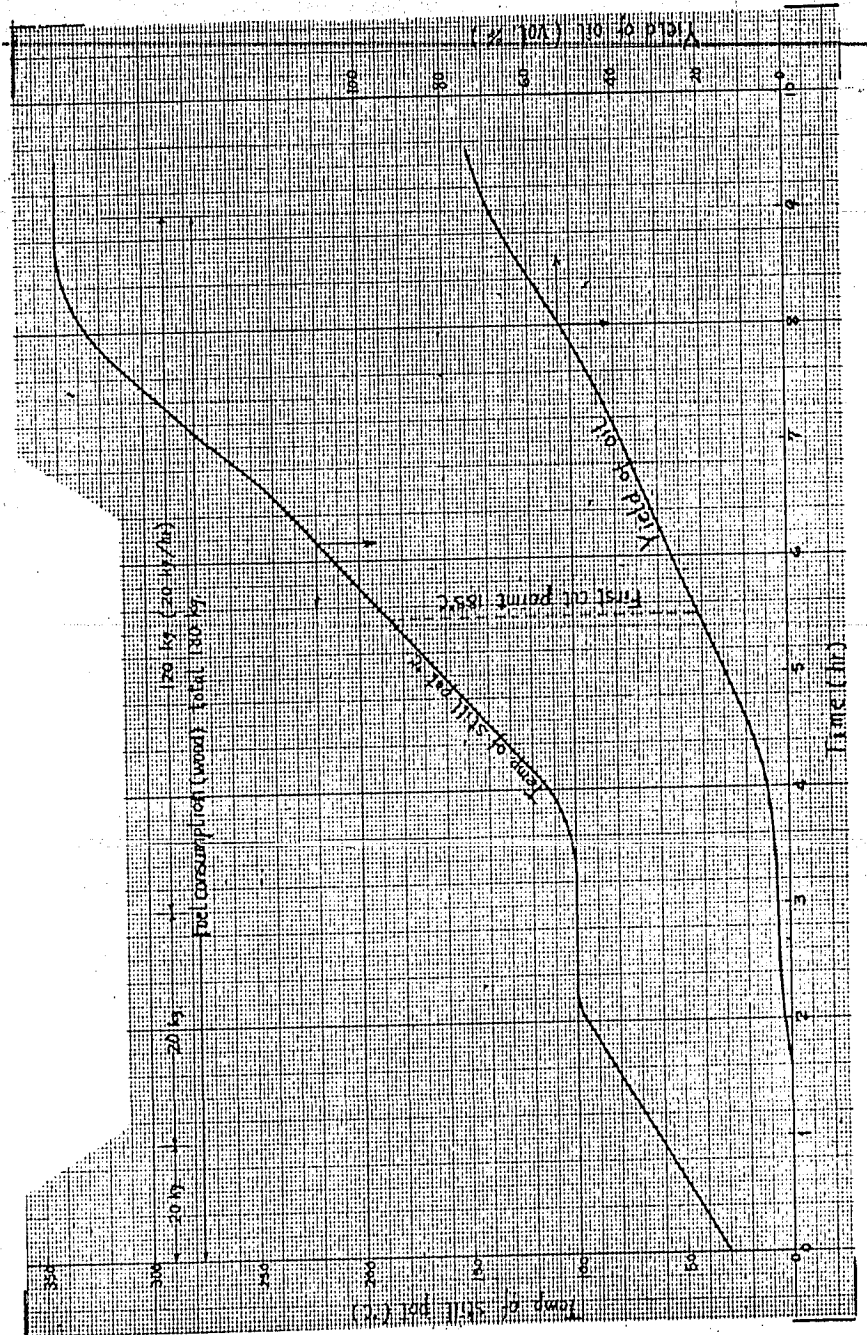


Figure 3(B)4
OPERATING CURVES ON CATALYTIC REFORMING UNIT

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Figure 4(B)4
SIMPLIFIED DISTILLATION
AND CARBONIZATION PLANT



Figure 5(B)4
SIMPLIFIED CATALYTIC REFORMING
OF PINE-ROOT OIL



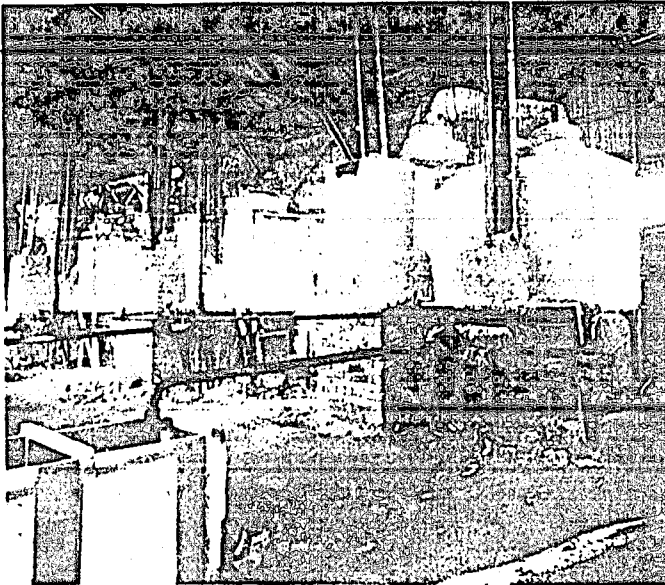


Figure 6(B)4
SIMPLIFIED CATALYTIC REFORMING
OF PINE-ROOT OIL

Figure (B)4
SIMPLIFIED CATALYTIC
REFORMING PLANT



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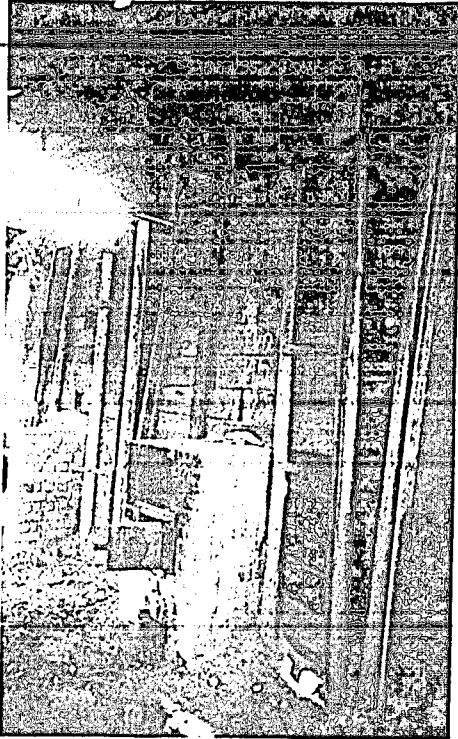


Figure 8(B)4
SIMPLIFIED REDISTILLATION PLANT

Figure 9(P)4
SIMPLIFIED REDISTILLATION PLANT

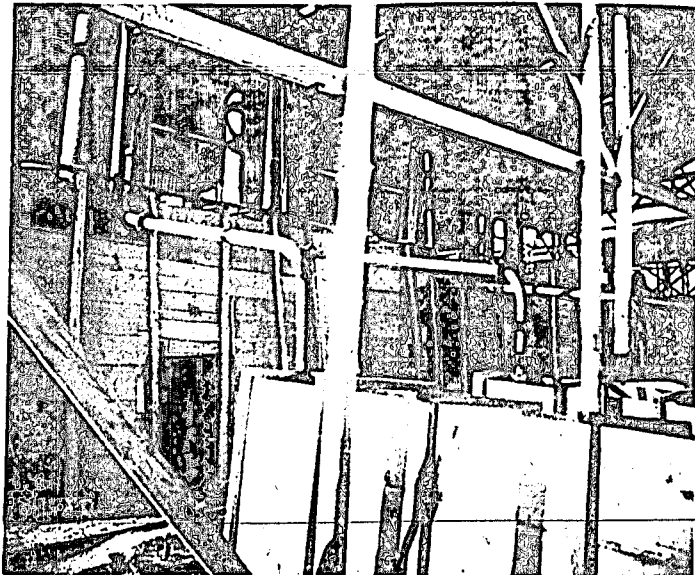


PLATE I (B) 4
 DISTILLING AND COKING OF PINE ROOT OIL
 DAILY OUTPUT 15 M.
 (1st NAVAL FUEL DEPOT)

