ENCLOSURE (B) 3

RESEARCH ON THE PRODUCTION OF ISOBUTANE

by

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LIST OF TABLES AND ILLUSTRATIONS

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SUMMARY

Fischer condensate oil was cracked in a pilot plant at 220°C, under atmospheric pressure, using 30 wt.% of anhydrous $AlCl_3$ as the catalyst. Yields of 28.6 wt.% of isobutane and 30-40 wt.% of gasoline of 95 octane (with 0.15% lead) were obtained.

This process is considered a promising source of aviation gasoline.

I. INTRODUCTION

A. HISTORY OF PROJECT

The production of isobutane from wax had been studied of OFUNA from April 1937 to September 1944.

The first work was done on laboratory glass scale and included an investigation of i-butane production by isomerization of various types of charge stocks with $AlCl_3$. It was found that addition of HCl increased yield. The yield also increased proportionately to the amount of $AlCl_3$ used, up to a certain maximum.

Typical data are given below for optimum conditions.

TABLE I(B)3

	$AlCl_3$ used % by wt.	HCl used % by wt.	Yield of isobutane % by wt.	Yield of gasoline % by wt.
Crude shale wax	20	5	27.5-33	32-33
Pure Shale wax	15	5	34-42	27-36
Wax from East Indies oil	10	5	43-44	30-32

On the basis of these early studies the following conclusions were drawn.

1. The wax from the East Indies oil was the best raw material for production of isobutane.

2. The reaction temperature range was 200-300°C and when the amount of catalyst was increased, the reaction temperature was lowered.

3. About 8-10 hours were required for the completion of the reaction.

4. The largest part of the products from wax was isobutane, and the formation of higher hydrocarbons was small.

5. Hydrocarbons containing less than 3 carbon atoms were not produced, and all hydrocarbons were isomeric paraffines with one, or sometimes two, methyl branches.

In April, 1943, a pilot plant was installed to at OFUNA to investigate production of isobutane. A run was made on crude shale wax but was unsatisfactory due to plugging difficulties caused by the high melting point.

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Subsequent pilot plant work, was then concentrated on Fischer-Tropsch condensate oil, which contained straight chain paraffines of lower melting point.

Work on this project was stopped in September 1944 due to limitations in the supply of waxy oils.

B. KEY RESEARCH PERSONNEL WORKING ON PROJECT

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II. DETAILED DESCRIPTION

A flow sheet of the pilot plant for isobutane production is given by Fig. 1(B)3. Properties of the Fischer condensate oil are given below.

The feed oil and the catalyst ($AlCl_3$) were stirred to a homogenous state in the mixer and pumped to the reaction chamber.

The necessary amount of HCl gas was then added to the reaction chamber. Pressure of the reaction chamber was atmospheric. The reaction mixture was circulated by the hot oil pump through the pipe still, continuously.

Isobutane and light hydrocarbons were removed overhead, condensed and caustic washed.

Periodically the sludge was drawn off from the bottom of the reaction chamber, and batches of fresh oil and $AlCl_3$ were added from the mix-tank.

TABLE II(B)3

		Run No.		
		1	2	3
Isobutane Pilot Plant Results	$AlCl_3$, wt. % of oil charge	30	20	20
	HCl, wt. % of catalyst	0	0	6.5
	Temperature of mixer, °C	120-135	150-170	150-170
	Temperature of reaction chamber °C	180-210	170-220	170-220
	Duration of reaction, hrs.	4-12	8-12	8-12
Average Yield, wt.% Oil Charge	Isobutane	(28.6)22%	16%	3.5%
	Isopentane	(14.2)12%		
	Gasoline	(30.0)32%	21%	46%
	Propane	4%		

Note: Figures in parentheses indicate maximum yield observed.

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TABLE III(B)3

Properties of Gasoline

		Run No. 1	Average of 2 and 3
d_4^{15}		0.696	0.677
ASTM Distillation	I.B.I.	22.5°C	21.0°C
	10%	44.0°C	30.0°C
	50%	151.0°C	71.0°C
	90%	213.0°C	205.0°C
	97%	233.5°C	220.0°C
	D.P.	237.0°C	223.0°C
Octane No.	clear	71.3	75.5
	0.1% lead	90.3	94.3
	0.15% lead	95.1	97.3
Yield of av. gush below	130°C,(vol%)	40	40

Because of a shortage of HCl, the experiments using HCl were not completed, but its beneficial effect in increasing yield of i-C_4 is apparent.

III. CONCLUSIONS

A. This process was considered promising as a source of aviation gasoline since 30% of isobutane and 30-40% of high octane gasoline could be produced from the Fischer-Tropsch condensate oil. By modification of plants to handle higher melting point stocks, it is considered that the comparatively abundant waxy stocks from shale wax and East Indies oil could be used as food stocks.

B. The amount of catalyst ($AlCl_3$) was decreased by the use of hydrogen chloride.

C. A reactor of the semi-continuous type is preferable for long time operation to allow for periodic removal of sludge.

D. It would be essential to recover the aluminum chloride from the cracked residue in a large scale plant. No investigations were made of the problems of such recovery.

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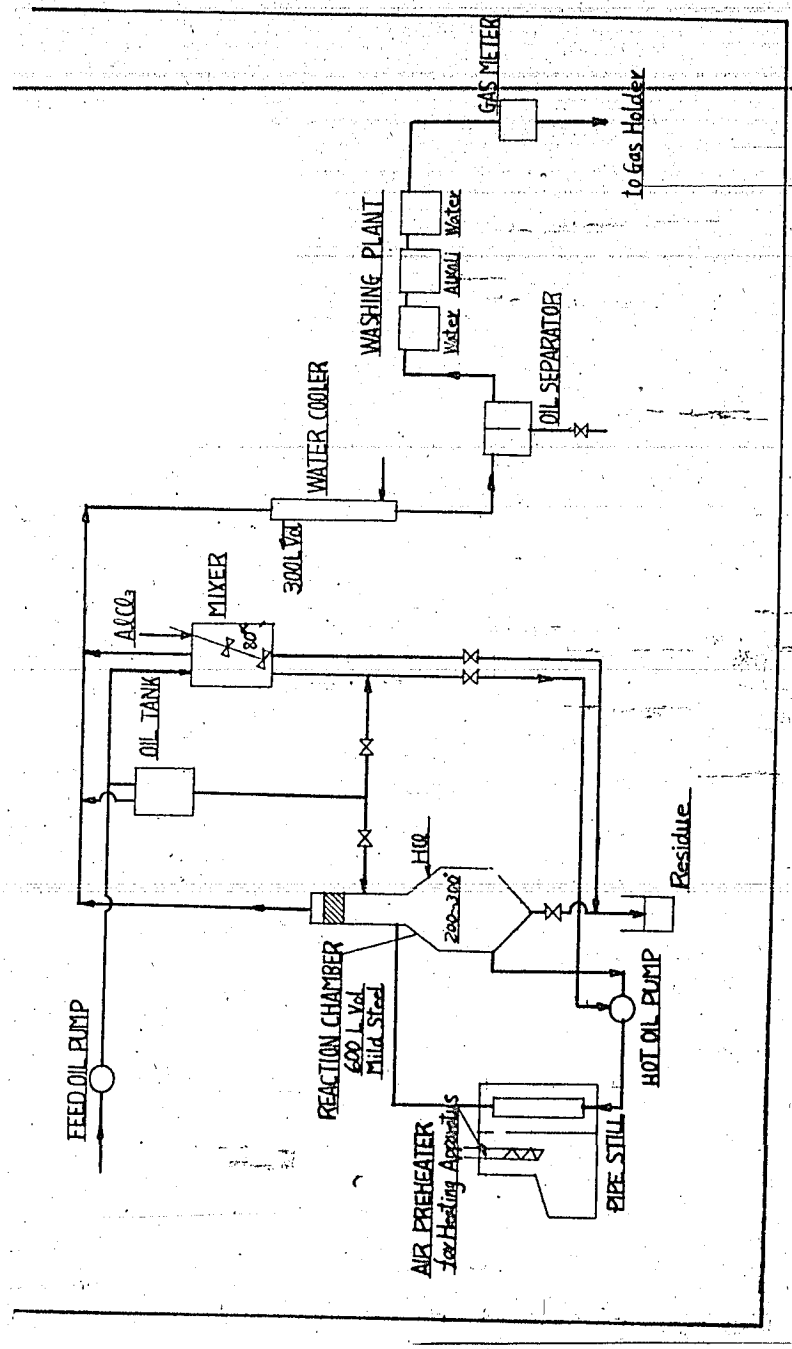
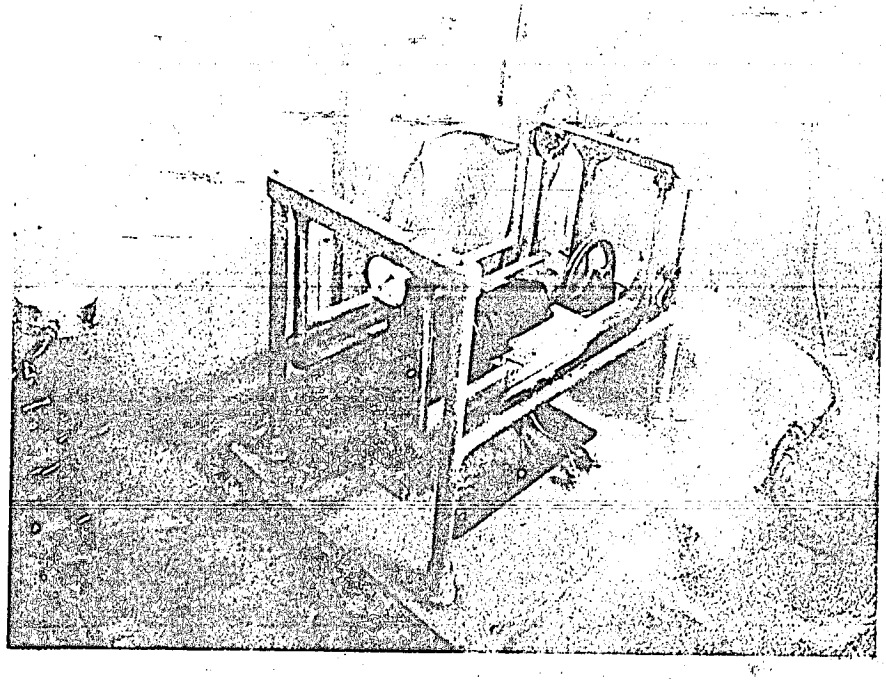


Figure 1(B)3
ISOBUTANE PILOT PLANT



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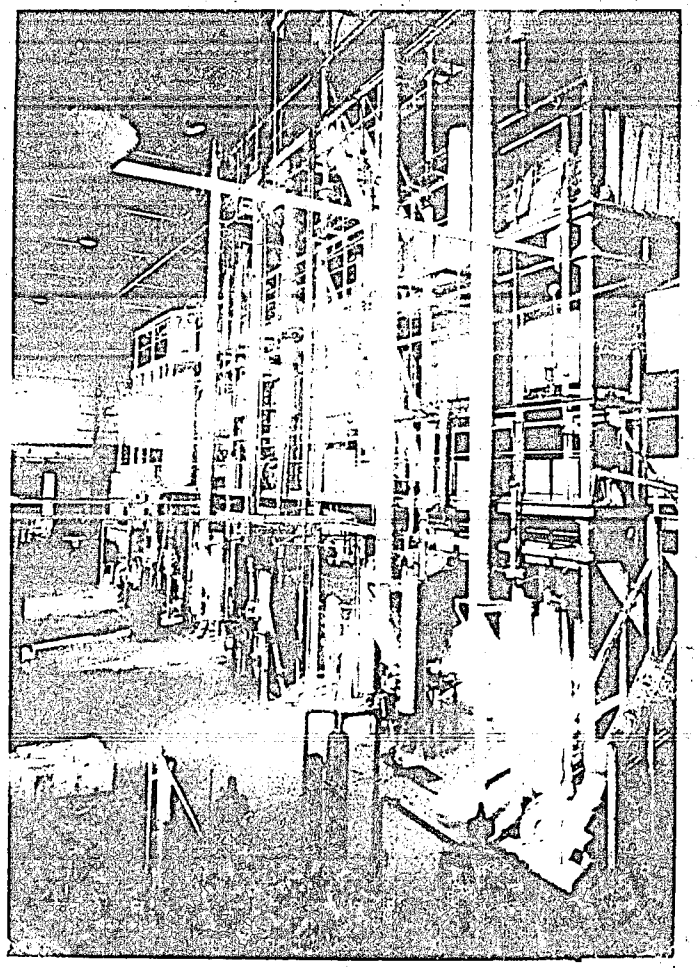

Figure 3(B)3.
ISOTOPIZATION PLANT OF X-FUTAN