

ENCLOSURE (1)8

SUMMARY

Iso-butane and propylene were alkylated using the concentrated sulphuric acid process and 16.5 wt % yield of crude alkylate to charged propylene was obtained. The octane value of the alkylate was 85 to 90, and 100 octane aviation gasoline could be made by blending 60% of alkylate with 40% of 92 octane base gasoline and adding 0.1% of tetra ethyl lead.

The optimum conditions of the alkylation process were determined to be as follows:

H ₂ SO ₄ concentration	97 to 99%
Ratio of charging stock and H ₂ SO ₄ ..	1 : 1 (vol)
Reaction temperature	25 to 30°C
Reaction pressure	5 kg/cm ²
Contact time	60 min
Stirring rate	300 RPM
Catalyst	0.2% chromic acid

I. INTRODUCTION

Since Elkington (Betaafische Petroleum Maatschappij Engineer) discovered, in 1937, the process of condensation of paraffinic and olefinic hydrocarbons in presence of concentrated sulphuric acid, the production of high octane gasoline by the method of "alkylation" has been studied by many (for example, Erch, Dunstan, Fidler, Dim, Tait, Gard, Blaunt, Karpi, Caesar, François, McAllister, Anderson, Ball, and Ross).

In this investigation the author has studied the alkylation reaction, especially the following aspects of the reaction of iso-butane with propylene: determination of best conditions of alkylation; reaction promoters; and properties of the alkylate.

The author established that alkylation was not direct condensation only, but was also accompanied by the eight reactions (Hydrogenation, Polymerization, Depolymerization, Rearrangement, Isomerization, Carbon to carbon cleavage, Dehydrogenation and addition of radicals).

The vital point of the alkylation reaction, the emulsification of catalyst and hydrocarbons, was studied mainly with regard to the formation of emulsion by two mechanical methods: stirring and injection.

In laboratory tests these two methods were almost equal, but in pilot plant tests stirring gave better results.

A pilot plant with total hydrocarbons plus sulphuric acid charging capacity of 10 to 20 lit/hr. was constructed and operated during 1941-1942. All reaction conditions compared well with the results of laboratory tests.

Although the author observed the alkylation plants of Nederlandsche Kolonial Petroleum Maatschappij and Betaafische Petroleum Maatschappij at Palembang in 1942 and admired both the alkylation feed gas fractionation equipment and the simple stirring type reactor, yet the design could not be directly utilized in Japan owing to the very limited supply of alkylation feed gases, especially iso-butane. In this latter connection the author has also made studies of methods for producing alkylation feed gases.

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II. DETAILED DESCRIPTION

A. Laboratory Test

Details of the laboratory apparatus used in alkylation studies are given by Figures 1(B)8 and 2(B)8.

Iso-butane and propylene were mixed in the gas holder at given ratio, compressed in a one hp - compressor and liquefied by cooling with solid CO₂ - alcohol solution in the measuring cylinder I. Autoclaves I and II were filled with concentrated H₂SO₄ from measuring cylinder II and the motor driver stirrers started. Liquefied hydrocarbons were introduced into the autoclaves in the required amounts. The temperature of the reaction was regulated by circulation of cooling water. When the reaction was over, unreacted gases were released through the gas meter to the gas holder. The volume and concentration of spent sulphuric acid were measured. The volume and specific gravity of the alkylate were also determined and it was then distilled into three fractions, the first up to 30°C, second 30 to 170°C, and the third over 170°C.

1. Effect of reaction time

A reaction time of at least 60 minutes is required. (See Table I(B)8 and Figure 3(B)8.

Conditions for these tests were:

H ₂ SO ₄ Volume	150cc
Conc.	98%
Reaction temp.	25°C
RPM	300
Liquid charge C ₃ H ₆ and i-C ₄ H ₁₀	400cc

2. Effect of reaction temperature and concentration of sulphuric acid.

Alkylation cannot proceed with an acid concentration below 85% (H₂SO₄.H₂O), and a concentration of 96 to 100% is best. The relation between concentration of H₂SO₄ and reaction temperature is very interesting; refer to Table II(B)8. See also Figures 4(B)8 to 6(B)8.

Conditions for these tests were:

Volume of H ₂ SO ₄	150cc
Feed	400cc
RPM	300

In conclusion, the best conditions are given below: (other conditions same as Table I(B)8.)

<u>Reaction temperature °C</u> <u>under best condition</u>	<u>Conc. of</u> <u>H₂SO₄</u>	<u>Max.</u> <u>yield</u>
10°	99%	150%
25°	99%	165%
30°	98%	163%
40°	97%	164%

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3. Effect of amount of H₂SO₄

The volume ratio of liquid raw material of H₂SO₄ must be less than 3/1, as Table III(B)8 and Figure 7(B)8.

Conditions for these tests were:

Conc. of H₂SO₄ 98%
 Feed 400cc
 RPM 300
 Temp. 30°C

4. Effect of pressure

A reaction pressure higher than 5 kg/cm² had no effect; refer to Table IV(B)8 and Figure 8(B)8.

Conditions for these tests were:

H₂SO₄
 Volume 150cc
 Conc. 98%
 Reaction temp. 30°C
 Ratio 1.c₄/c₃ = 3/1
 RPM 300
 Reaction time 60 min
 H₂ pressure used

5. Effect of stirring

The effect of stirring is very important. In the autoclave 60 to 120 RPM was too little (in such a case polymerization or esterification of propylene only occurs), and 300 RPM was found to be the optimum rate. See Table V(B)8 and Figure 9(B)8.

Conditions for these tests were:

Ratio 1.c₄/c₃ = 3/1
 Volume of H₂SO₄ 150cc
 HC feed 400°C (liquid)
 Reaction time 60 min

6. Effect of feed gas ratio

The iso-butane to propylene ratio should be over three. If the ratio is less than three, polymerization of propylene occurs. (See Table VI(B)8 and Figure 10(B)8.

Conditions for these tests were:

H₂SO₄
 Volume 150cc
 Conc. 98%
 HC feed 400cc
 RPM 300
 Reaction time 60 min

7. Effect of emulsifiers

The results of adding emulsifiers were not good, refer to Table II(B)8.

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Conditions for these tests were:

H ₂ SO ₄	
Volume	150cc
Conc.	98%
Ratio	1.0 ₄ /c ₃ 3/1
Reaction Time	60 min
Temp.	30°C
RPM	135
HC feed	400cc

8. Effect of electrolytes

Nitric acid and ferric sulphate were more effective, but not as attractive as the use of oxydizing agents; refer to Table VIII(B)8.

Conditions for these tests were:

H ₂ SO ₄	
Volume	150cc
Conc.	98%
HC feed	400cc
Temp.	30°C
RPM	300
Gas ratio	3 : 1

9. Effect of oxydizing agents

Oxydizing agents were very good promoters.

The concentration of spent sulphuric acid was higher and the yield of aviation gasoline also was higher; refer to Table IX(B)8.

Conditions for these tests were:

Volume of H ₂ SO ₄	150cc
Feed	400cc
Reaction time	60 min
Gas ratio	3 : 1
Reagents	0.2% (wt)

10. Properties of alkylate

The 30° to 170°C fraction of alkylates was good aviation fuel, having the following properties.

Sp. gr. 20°/40	0.6821
Oct. No.	100 (0.1% lead)
Vap. press	0.56 kg/cm ²
Freezing pt.	-50° max
Fract. distillation	
Initial pt.	48°C
10%	63.5°C
50%	92°C
90%	124°C
97%	170°C

11. Corrosion test of materials of construction

The results are given below:

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13% Cr-steel slightly corroded
 18-8 stainless no change
 Ni-Cr-7 steel no change
 85 kg Ni-Cr-steel poor
 35 kg Ni-Cr-steel very bad
 Mild steel very bad
 Abesta 832S stainless steel no change

13% Cr-steel was selected out of the above seven steels as material for alkylation plants construction.

B. Pilot Plant

A flow sheet of the reaction section of the continuous pilot plant is given in Plate I(B)8.

Pure iso-butane and propylene, or catalyst cracking plus hydrocracking gas, were used as raw materials.

The feed stock was compressed, liquefied by brine cooling, and stored in a cooled tank. The liquefied gas and concentrated sulphuric acid were sent to the reaction chamber by plunger type pumps. After the reaction the emulsion was settled and separated into product and pent sulphuric acid. The product was washed by caustic soda and water.

Since 1941 five runs were made, but all data were burnt this year, and only the following points can be recalled:

1. The plunger pump for concentrated sulphuric acid was not satisfactory, because the asbestos packing and plungers (even stellite welding) were corroded by the acid.
2. The injection type reactor was also not satisfactory, because it was soon plugged by dirt and impurities in the feed.
3. The stirrer type reactor was very good and all pilot plant results using this type agreed with laboratory data (165 to 170% yield and 85 to 90 octane rating), but total olefine content of the feed stock should be less than 20% to maintain sufficiently high acid concentration.
4. The separation of hydrocarbons and sulphuric acid from the emulsion was very difficult, requiring two hours settling time minimum.

TABLE I(B)8

EFFECT OF REACTION TIME

Test No.	Conditions of Reaction			Properties of Products							
	Reaction Time (min.)	Press. (Kg/cm ²)	C ₃ content (vol. %)	Spent gas (lit.)	Spent H ₂ SO ₄		Total oil Product	30~170° (cc)	Yield % to C ₃	Blending O.N.	170°~ (cc)
					Vol. (cc)	Conc. (%)					
285	20	5.7	24.7	55.5	153	90.06	122	50	78.4		16
156	60	4.9	27.1	40.9	143	94.18	165	101.1	143.9	88.8	11.5
162	120	4.2	25.1	34.6	155	94.91	165	98.3	149.9	86.5	9.7
163	180	4.9	25.5	36.3	150	94.62	170	102	155.0		9.8

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TABLE II(B)1
EFFECT OF REACTION TEMPERATURE AND CONCENTRATION OF SULFURIC ACID

Test No.	Conc. of H ₂ SO ₄ (%)	Reaction Temp. (°C)	Max. Temp. (°C)	Reaction Press. (kg/cm ²)		Spent gas	US gal. S ₂ in spent gas	Spent H ₂ SO ₄ (cc)	Conc. of spent H ₂ SO ₄ (%)	Total oil Product (cc)	Sp. Gr. (15°/15°)	High to Cr No.	Heavy Alkylate Amount (cc)	Blending Oil No.
				Initial	Final									
25	102	-30	-25.0	0	0	251.6	2.75	151	81.4	0				
21	102	-10	-4.6	2.0	3.5	54.1	2.0	200	82.6	36	12.5	100	33.0	
17	102	5	1.7	4.5	3.8	76.7	4.05	200	92.2	46	19.0	100	28.0	
9	102	10	14.3	6.2	5.8	78.0	4.02	182	78.2	78	22	14.0	0.6993	8.0
8	101	10	13.3	10.5	9.0	78.0	3.16	225		45	9.5	19.0	0.6972	29.0
133	101	30	32.6	4.5	4.0	50.0	1.34	180		45	35.6	15.1	0.6950	71.4
7	100	10	12.8	10.0	8.7	70.7	3.10	200		47	3.5	25	0.6956	37.5
118	100	20	23.0	3.1	2.5	131.3	0.1	168	93.14	135	44.4	78.6	0.6958	130.8
152	100	25	26.0	3.2	3.0	26.7		172	93.82	111	41.0	92.0		0.4
153	100	30	32.0	5.0	3.7	50.0		153	93.41	127	48.0	63.0	0.6955	90.6
119	100	40	42.6	5.0	3.8	27.0	1.28	150	93.69	97	30.5	15.5	0.6959	68.1
116	99	110	12.3	3.2	2.7		1.2	170	94.56	158	52.5	96.0	0.6978	153.3
300	99	20	20.5	5.3	4.4	53.8	10.9	157	94.09	143	49	80	0.6979	125.3
154	99	25	26.0	4.0	3.3	58.6		110	94.76	205	69.5	118.1	0.6933	168.2
155	99	30	30.5	3.5	3.3	27.1		150	94.06	200	98.5	32.5	0.7006	137.0
167	99	35	35.8	5.2	4.7	47.0	0.65	150	92.8	180	46.0	91.0	3.7003	113.5
151	99	40	40.2	5.0	4.9	14.8	0.83	164		120	39.3	68.7	0.6956	100.5
3	98	10	12.8	3.8	3.2	101.3	0.55	163	89.9	92	22.0	50.5	0.6968	68.1
156	98	25	25.2	4.9	3.5	165.9	0.85	145	94.18	165	42.4	101.1	0.6936	143.9
194	98	30	31.5	4.8	3.7	44.7	0.6	163	93.99	178	67.5	105	0.7017	165.4
169	98	35	36.5	5.0	4.2	45.9	0.3	113	93.23	140	45.5	105	0.7036	135.8
170	98	40	41.0	5.8	5.2	46.6	1.28	145	92.50	140	56.0	70.0	0.7011	110.5
132	97	20	21.0	2.8	2.1	51.2	0.77	145	93.50	137	57.0	74.0	0.6971	109.8
123	97	25	26.6	3.8	2.9	48.2	1.3	170		160	55.5	95.0	0.6976	141.3
153	97	30	30.0	4.9	3.6	27.1		158	94.77	175	61.0	95.0	0.6972	125.3
292	97	35	36.0	5.5	5.0	43.5	2.7	169	91.83	162	54	84.0	0.7024	131.5
172	97	40	40.0	4.9	3.9	37.0	1.08	158	91.66	168	48.5	107.5	0.7029	167.9
4	96	10	11.0	9.0	9.0	76.4	0.74	237	55.1	690	9.0	32.0	0.6969	49.4
160	96	25	25.3	4.8	3.9	42.6	0.84	170	91.93	140	35.3	88.2	0.6990	115.0
161	96	30	30.6	5.8	3.5	35.0	0.77	150	90.63	150	48.0	90.0	0.6987	138.1
173	96	35	35.2	4.8	4.2	24.99	1.04	172	89.56	168	61.0	94.0	0.7005	148.0
174	96	40	43.4	6.0	5.0	40.8	1.02	152	87.97	158	50.0	100.5	0.7032	157.4
190	95	35	35.0	5.5	5.1	50.0	0.75	170	89.89	110	43.0	93.7	0.7009	95.4
181	95	40	40.3	6.0	5.0	44.0	1.33	170	90.92	130	36.5	84.5	0.7018	127.6
192	94	35	34.5	5.0	4.0	48.7	1.11	159	83.34	138	50.5	57.5	0.7016	91.1
193	94	40	42.2	5.1	4.2	42.2	0.4	165	89.74	135	54.7	71.3	0.7016	112.6

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Test No.	Conditions							Properties of Products												
	Amount of H ₂ SO ₄ (cc)	Amount of Raw Material (cc)	Reaction Temp. (°C)	Reaction Pressure (Kg/cm ²)			Content of C ₃ (ml.-%)	Spent gas (lit)	C ₃ vol. % in spent gas	Amount of Spent H ₂ SO ₄ (cc)	Conc. of Spent H ₂ SO ₄ (%)	Total alk products (cc)	30°~170° fraction (cc)	Amount (cc)	Sp. Gr. (15°C)	Yield to C ₃ (%)	Dr No.	Amount (cc)	Dr No.	Blending O.N.
152	400	400	33.0	3.3	3.7	2.1	25.1	20.3	2.8	308	97.69	203	92.7	100.1	0.6692	122.5	1.1	10.2	10.9	83.8
154	200	400	30.8	4.5	5.0	3.8	21.1	43.2	0.99	200	94.8	160	49.0	97.0	0.6677	147.5	0.0	14	1.5	91.8
156	100	400	31.5	4.8	8.2	3.7	24.7	41.7	0.60	163	93.99	178	67.5	105	0.7017	16.8		5.7		86.6
46	100	400	31.0	5.8	5.8	4.7	25.4	36.6	0.80	118	96.67	134	58	65	0.7035	120.7		11	3.3	77.8
45	75	400	33.8	6.2	6.2	5.0	25.0	72.4	1.6	160		10	a	5		9.7				
44	50	400	32.6	7.5	7.5	6.1	25.0	63.5	1.4	151		1	0	1		1.9				
109	100	100	30.8	3.1	4.1	3.0	21.2	21.3	7.56	130	96.66	30	10	11.0	0.7176	23.7	20.7	3.0		
104	100	200	15.0	3.7	4.1	2.4	25.2	29.2	1.4	113	97.28	63	26.8	24.2	0.7041	37.6	1.3	11.0	27.1	
105	100	200	30.3	3.0	4.3	3.0	25.2	37.8	0.81	100	96.11	106	51.4	48.8	0.7040	70.4	6.2	11.3	34.9	
106	100	600	30.0	3.8	4.4	3.4	25.4	104.4	1.8	175	69.56	30	10	25.0	0.7141	23.7	66.6			

Test No.	Pressure (Kg/cm ²)			Properties of Products								
	Initial	Max	Final	Spent gas		Spent H ₂ SO ₄		Total oil (cc)	Light Alkylate (30°~170°)			Heavy Alkylate (cc)
				Amount (lit)	C ₃ vol. %	Amount (cc)	Conc. (%)		Amount (cc)	Yield to C ₃ (%)	Blending O.N.	
521	4.5	10.1	10.1	46.4	0.3	155	92.85	160	81	123.9	84.3	11.0
522	4.7	15.1	15.1	57.0	0.59	150	96.81	173	93	141.3	82.8	13.0
523	6.0	20.0	20.0	76.6	0.55	150	93.92	160	83	126.8	85.3	19.0
524	6.0	30.0	30.0	89.0	0.85	153	92.81	170	87	132.9	87.3	11.2
194	4.8	3.7	3.7	41.7	0.60	163	93.99	178	105	165.9	86.6	5.7

Test No.	Conditions of Reaction				Properties of Products							
	Conc. of H ₂ SO ₄ (%)	Temp. (°C)	Press. (Kg/cm ²)	Stirrer (RPM)	Spent gas (lit)	Spent H ₂ SO ₄ (cc)	Conc. of H ₂ SO ₄ (%)	Total oil product (cc)	30°~170° (cc)	Yield % to C ₃ H ₆	Octane value	170°~ heavy oil (cc)
107	98	30	4.5	60	70.8	188		40	10	15.1		17
295	98	30	5.7	120	58.7	180		72	22	34.5		25
157	98	30	4.6	300	36.8	164	94.31	180	111	158.8	88.3	11.4
294	99	25	5.8	60	72.7	180	92.92	30	7	10.9		17
293	99	25	5.1	120	57.0	170	90.44	75	24	37.7		26
154	99	25	4.0	300	58.6	140	94.76	205	118.5	168.2	88.8	17

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Table VI(B)8
EFFECT OF FEED GAS RATIO

Test No.	Conditions of Reaction				Properties of Products							
	Feed gas ratio $\frac{1}{C_4/C_3}$	Max. temp. (°C)	Max. Press. (Kg/cm ²)	C ₃ content (mol. %)	Spent gas (lit)	Spent H ₂ SO ₄ (cc)	Conc. of spent H ₂ SO ₄ (%)	Total oil products (cc)	30°~170° (cc)	Yield % to C ₃	Blending O.N.	170°~ (cc)
26	1/1	34.5	7.3	30.0	30.0	215	60.6	60	24.5	18.4		10.0
28	2/1	32.0	5.0	33.3	43.6	160		230	160.	185.2	79.8	17.5
194	3/1	31.5	4.8	24.7	41.7	160	93.99	178	105	165.9	86.6	9.0
120	1/1	31.4	3.9	16.9	45.6	153	97.22	157	89	202.8	89.3	13.0
32	8/1	33.4	6.0	11.1	39.5	150		155	76.5	271.5		9.2

Table VII(B)8
EFFECT OF EMULSIFIER

Test No.	Conditions of Reaction				Properties of Products			
	Reagents added	Amount of Reagent (gm)	Temp. max. (°C)	Press. range (Kg/cm ²)	C ₃ in spent gas (cc)	Sp. gr. (15°/4°)	Total oil product (cc)	Yield % to C ₃ H ₆
241	Benzenesulphonic Acid	1	34.5	17~16	0.76	0.6811	37.0	75.4
235	Toluenesulphonic Acid	1	36.3	14~12	1.87	0.7001	46.0	96.4
239	Naphtholsulphonic Acid	1	39.5	13~12	1.80	0.7035	33.4	70.4
247	Naphthylaminesulphonic Acid	1	34.9	16	3.20	0.6839	52.2	106.9
250	Stearic Acid	0.2		17~14	0.76	0.6918	41.4	87.5
250'		0.1	32.0	17~14	1.13	0.7001	60.3	126.0
205	None	0	31.5	17~16	0.37	0.6898	61.2	126.2

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Table VIII(B)8
EFFECT OF ELECTROLYTES

Test No.	Conditions			Properties of Products							
	Reagents added	Amount (gm.)	Press. max.	Spent H ₂ SO ₄		Spent gas (lit)	Oil product (cc)	30°~170° (cc)	Yield (%)	O.N. Blend	170°~ (cc)
				Amount (cc)	Conc. (%)						
116	None	0	4.1	150	94.77		125	83	126.9	84.8	17.0
125	SeO ₂	0.59	4.5	149	89.93	69.5	141	72	111.1	85.3	8.7
126	HNO ₃	2	4.5	160	93.11	53.4	137	86	136.2	84.8	11.0
127	HCl	2	4.1	145	94.88	55.2	132	83.5	131.3	84.8	9.0
129	ZnSO ₄	0.4	4.0	153	89.40	51.6	118	86	124.5	85.3	7.0
130	Al ₂ (SO ₄) ₃	0.4	4.5	155	94.92	58.8	121	75.6	118.1		7.5
131	Fe ₂ (SO ₄) ₃	0.4	5.0	150	94.17	49.8	142	87	138.1	87.8	8.5
128	H ₃ PO ₄	5.0	5.9	209		72.2	10		15		

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Table IX(B)8
EFFECT OF OXIDIZING AGENTS

Test No.	Conditions				Properties of Products						
	Reagents	Conc. of H ₂ SO ₄ (%)	Temp. (°C)	Press. (Kg/cm ²)	Spent H ₂ SO ₄ (cc)	Conc. of H ₂ SO ₄ (%)	Spent gas (lit)	Oil product (cc)	30°~170° (cc)	Yield	Octane Value
567	none	97	35	5.5	150	93.51	38	157	89	135.9	84.0
568	K ₂ Cr ₂ O ₇	97	35	5.1	160	94.34	39	182	99	151.1	87.0
573	(NH ₄) ₂ S ₂ O ₈	97	35	5.0	160	94.14	38	165	88	135.2	87.8
554	none	98	30	5.4	158	93.49	46	140	98	159.0	87.8
555	K ₂ Cr ₂ O ₇	98	30	5.0	154	93.04	44	160	91	140.7	
572	(NH ₄) ₂ S ₂ O ₈	98	30	4.9	150	95.13	38	180	108	164.9	89.0

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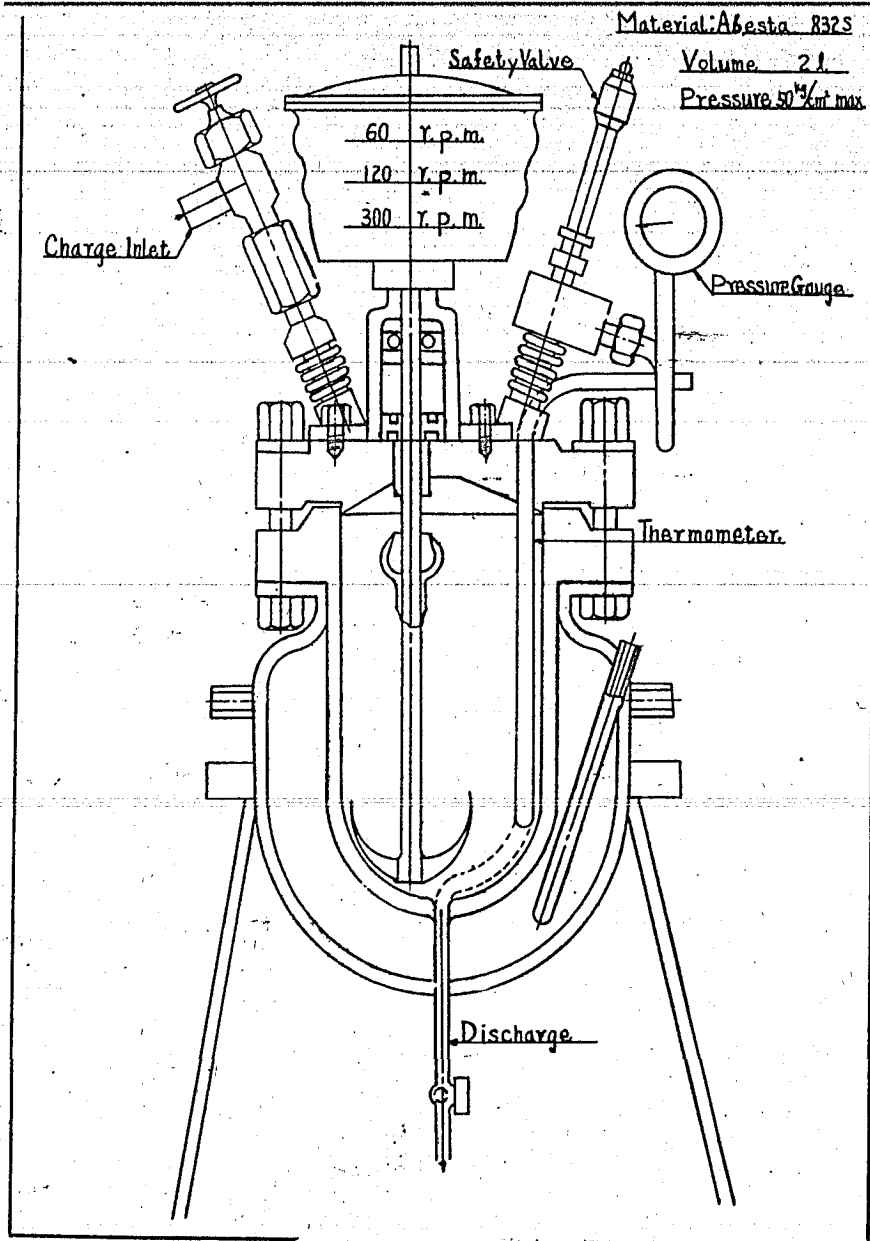


Figure 1(P)8
AUTOCLAVE FOR AIRYLATION

ENCLOSURE (B) B

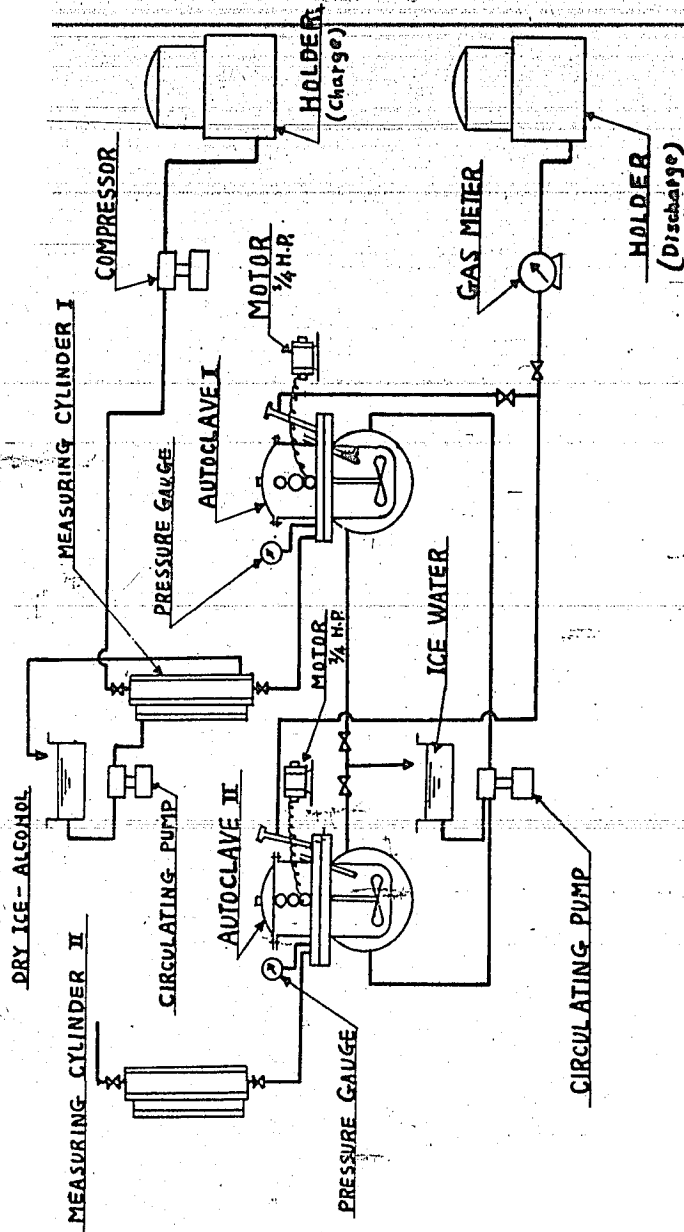


Figure 2 (B) B
L. J. GILBERT, JR., PHYSICIST, P. O. BOX 41, BOSTON, MASS.

ENCLOSURE (B)8

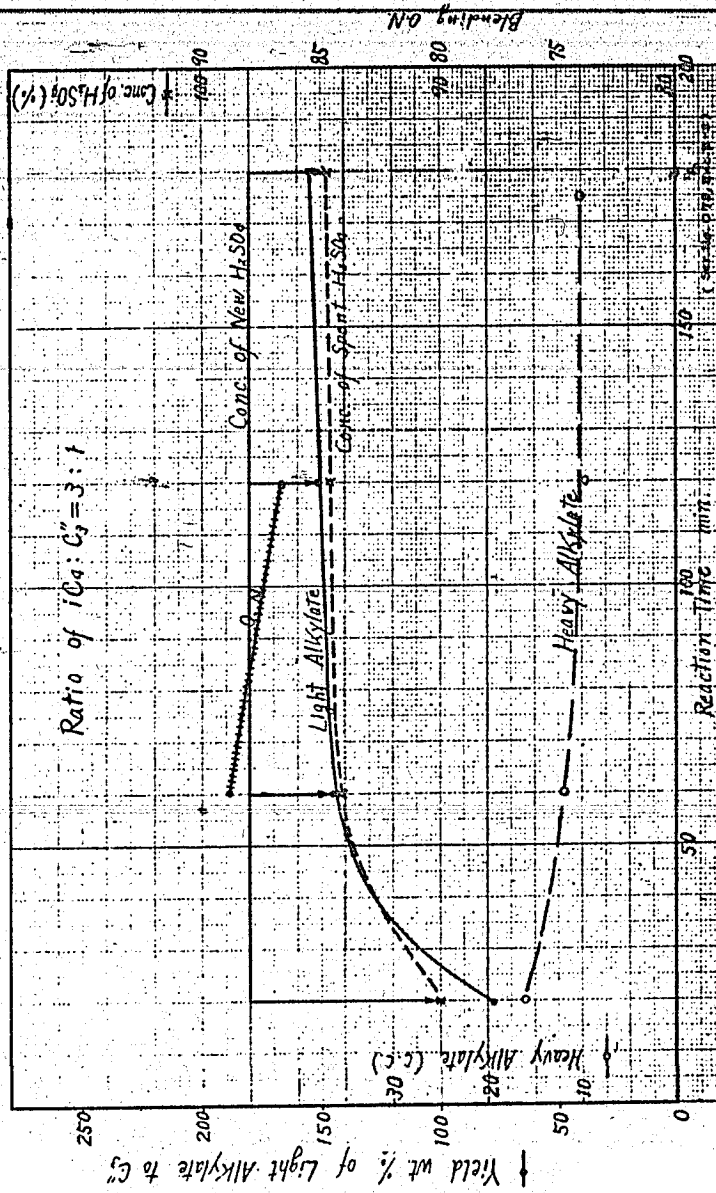


Figure 3(F)8
EFFECT OF REACTION TIME

ENCLOSURE (B)8

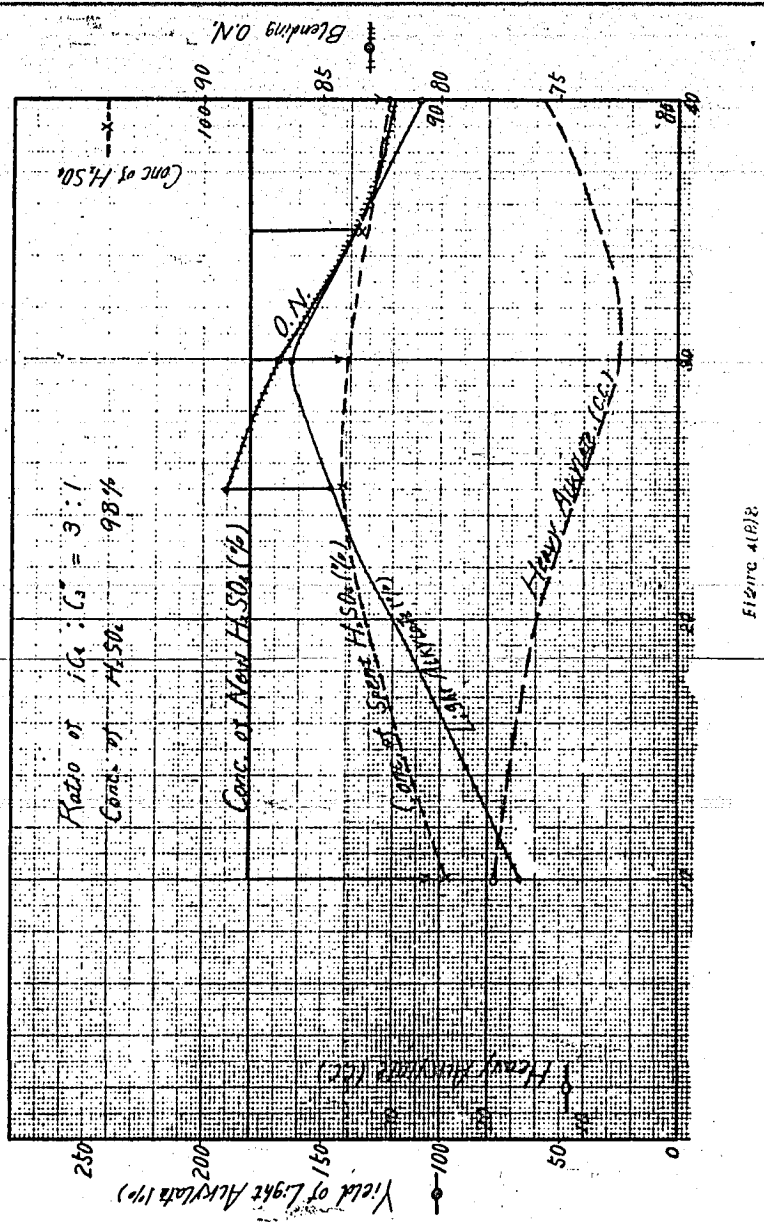


Figure 4(B)8
EFFECT OF REACTION TEMPERATURE (%)

ENCLOSURE (B)8

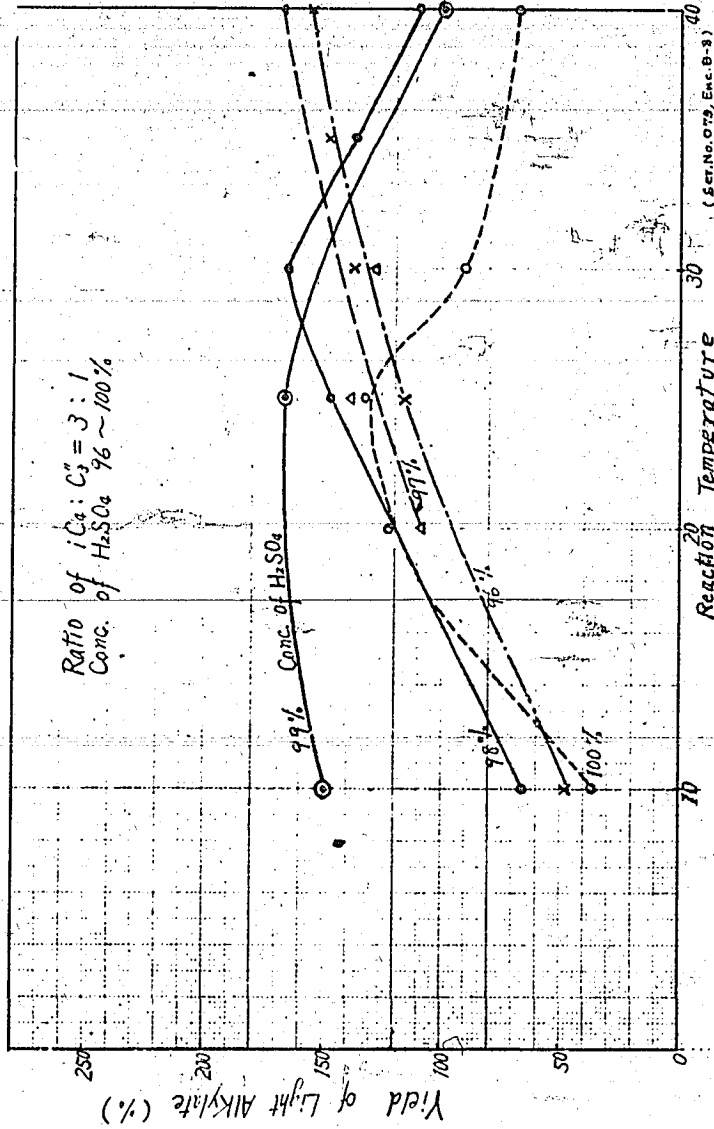


FIGURE 5(B)8
YIELD OF REACTION TEMPERATURE (C)

ENCLOSURE (B)8

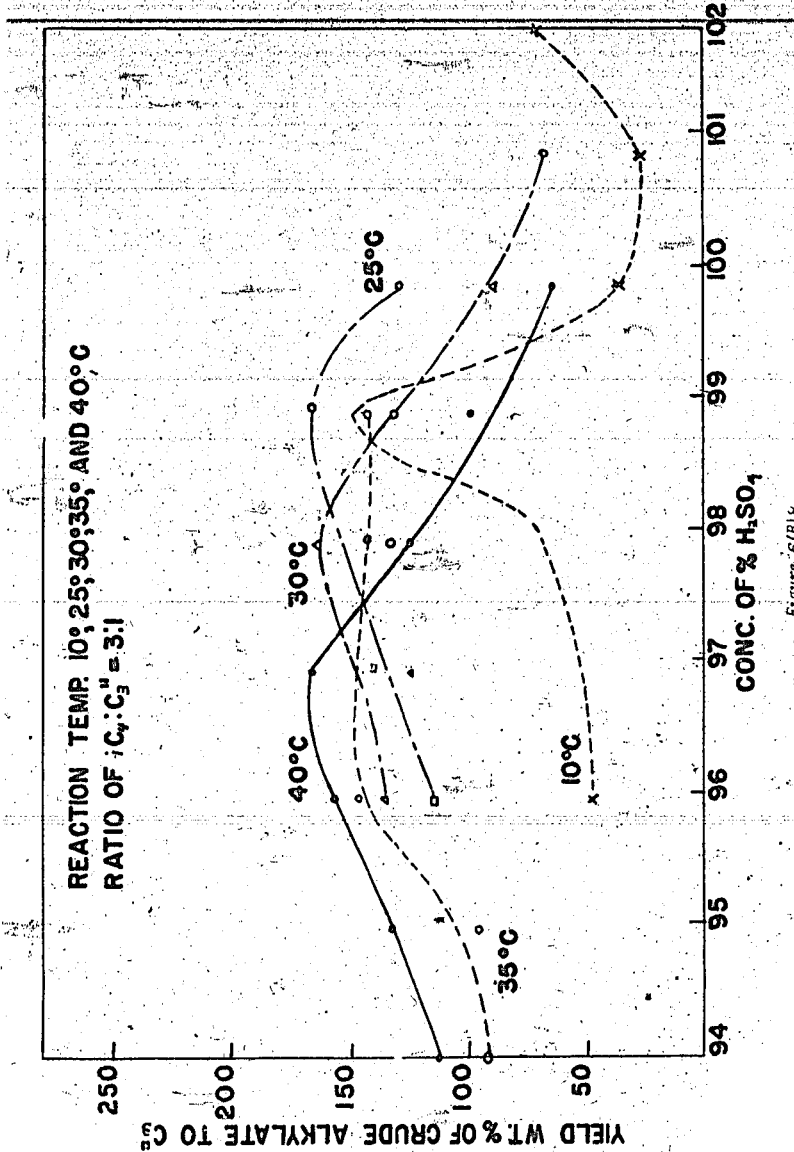


Figure 6(B)8
CONCENTRATION OF H₂SO₄ AND YIELD

ENCLOSURE (B) 8

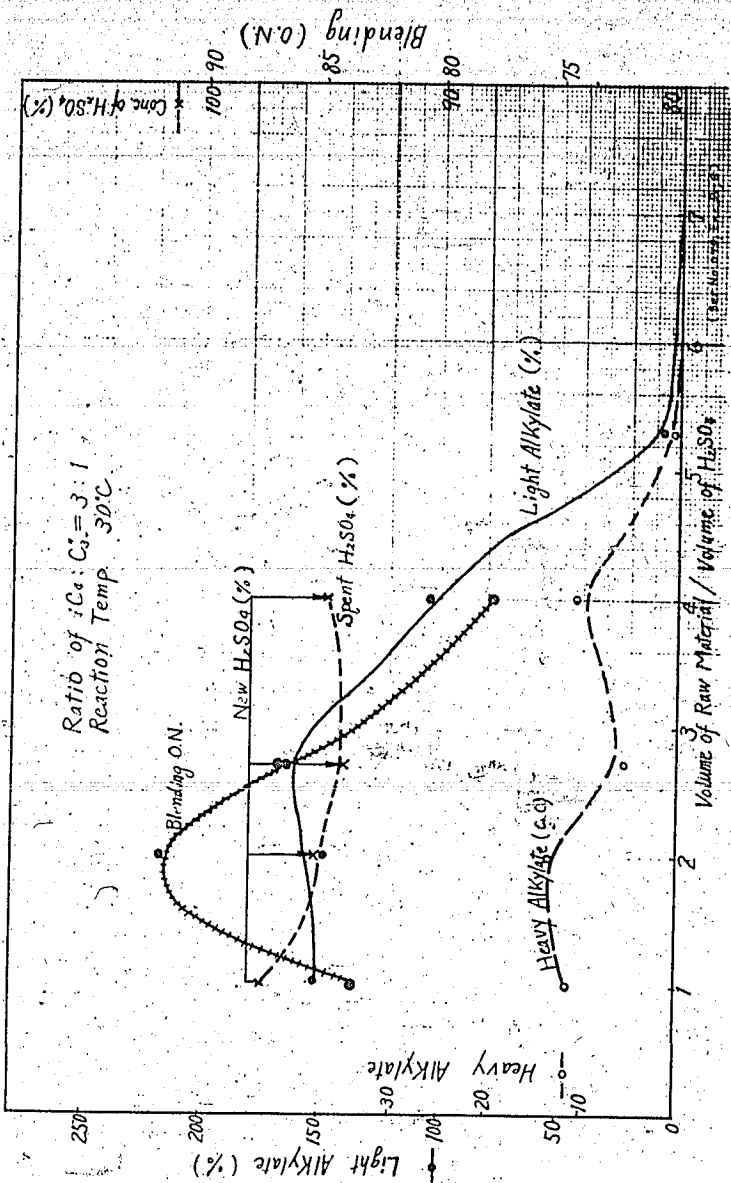


Figure 7(1) b
RELATIONSHIP BETWEEN VOLUME OF H_2SO_4

ENCLOSURE (B)8

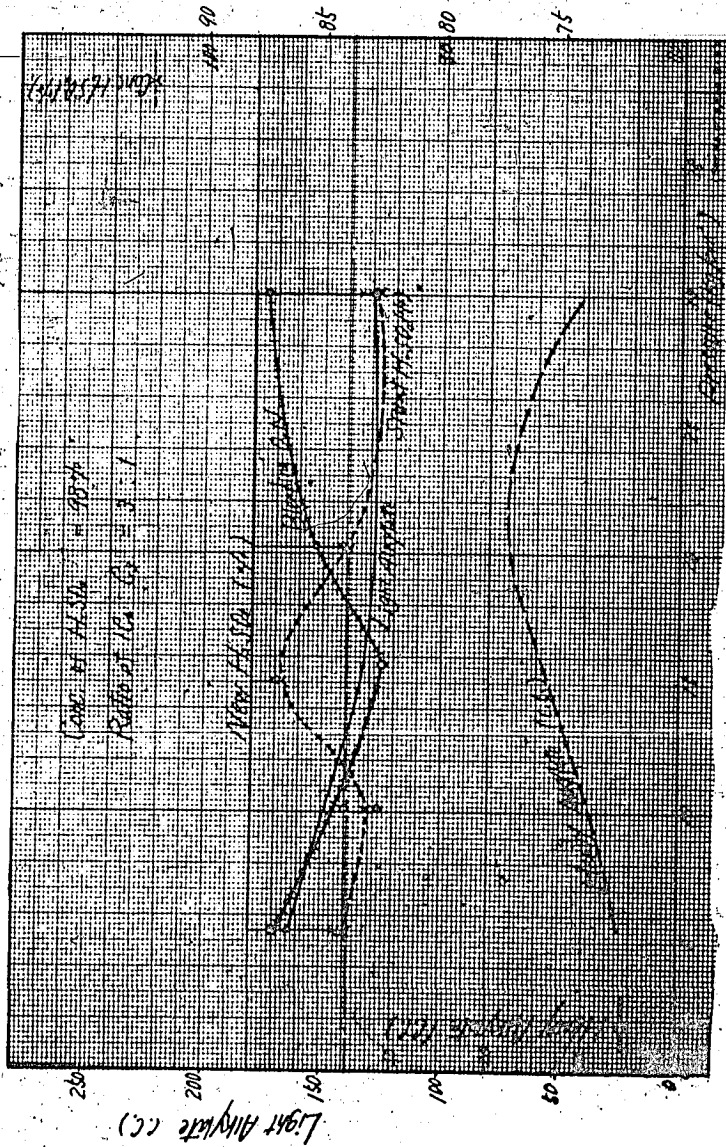


Figure 8(R)6
EFFECT OF PRESSURE

ENCLOSURE (B)8

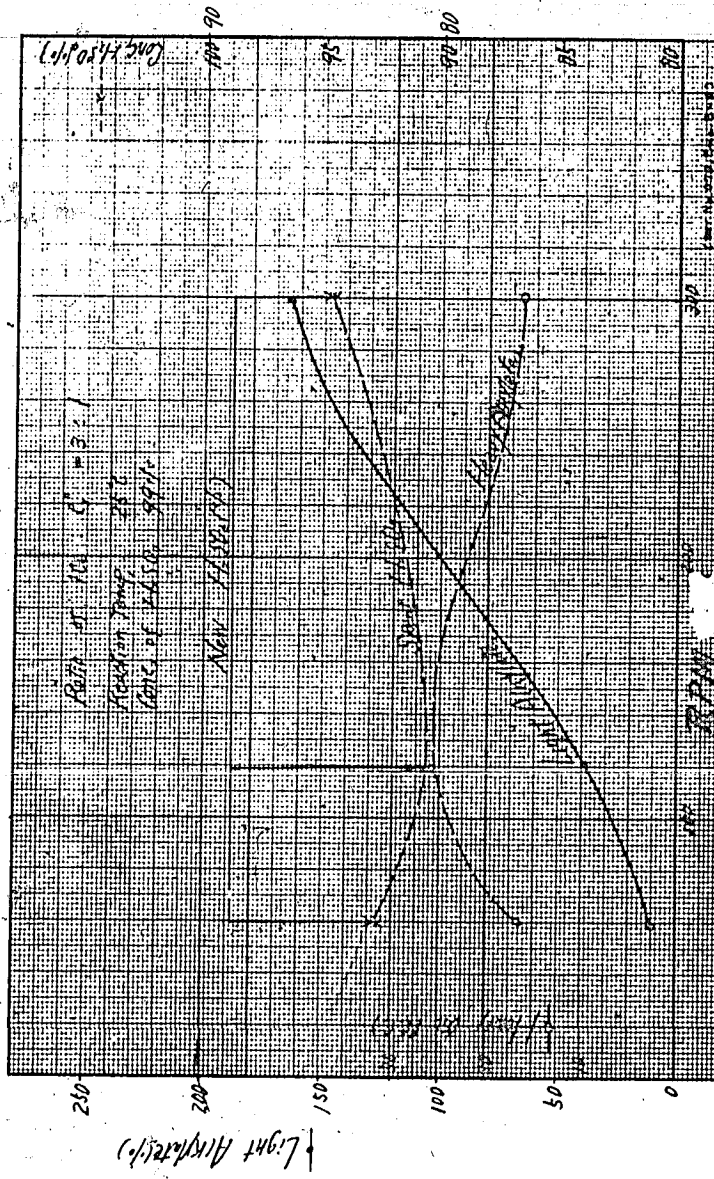


Figure 9(B)8
EFFECT OF STARTING

ENCLOSURE (B) 8

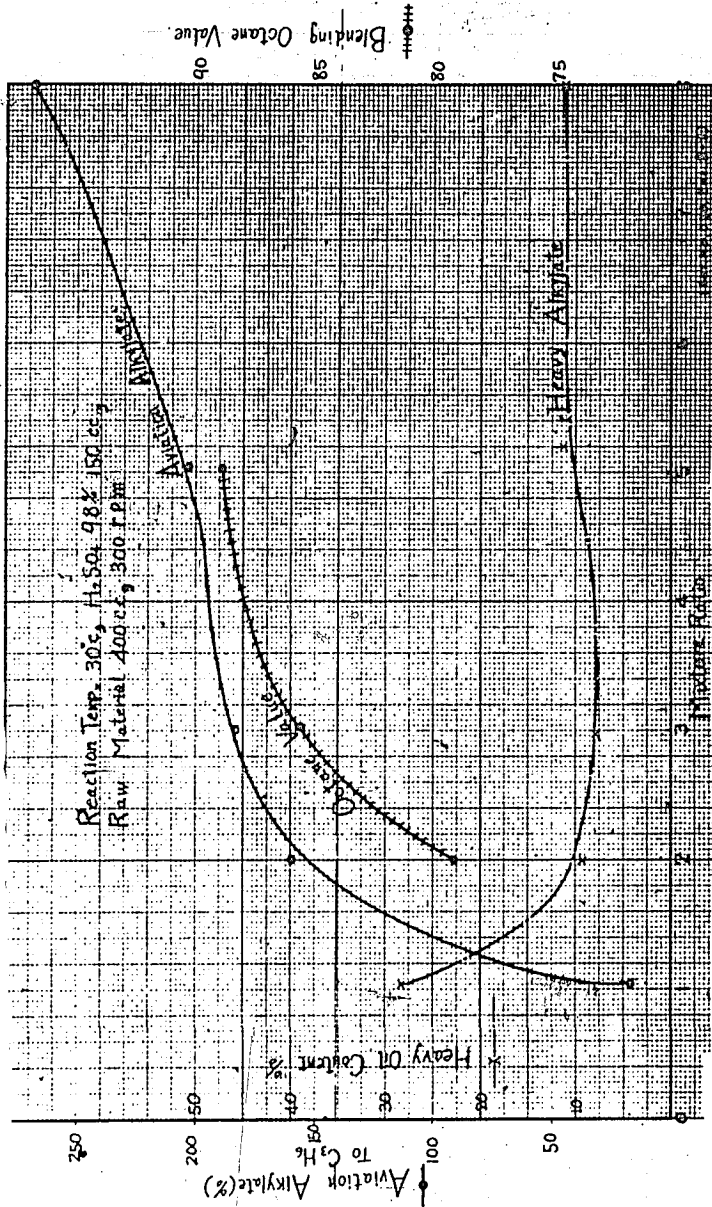
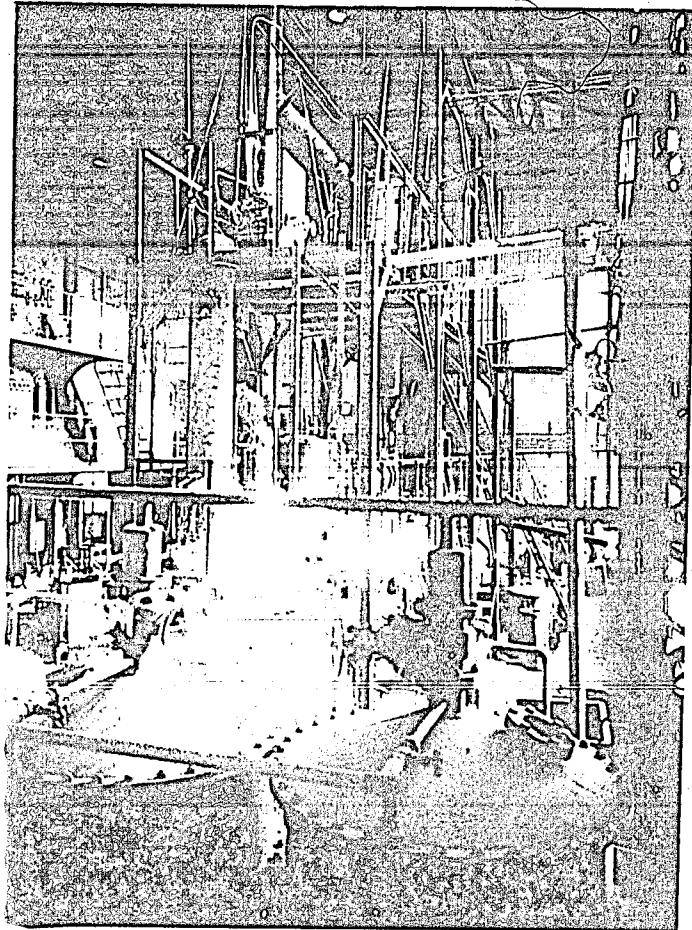


Figure 101E18

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ENCLOSURE (B)8



1966 - Figure 11108
Interior view of the project

ENCLOSURE (B) 8

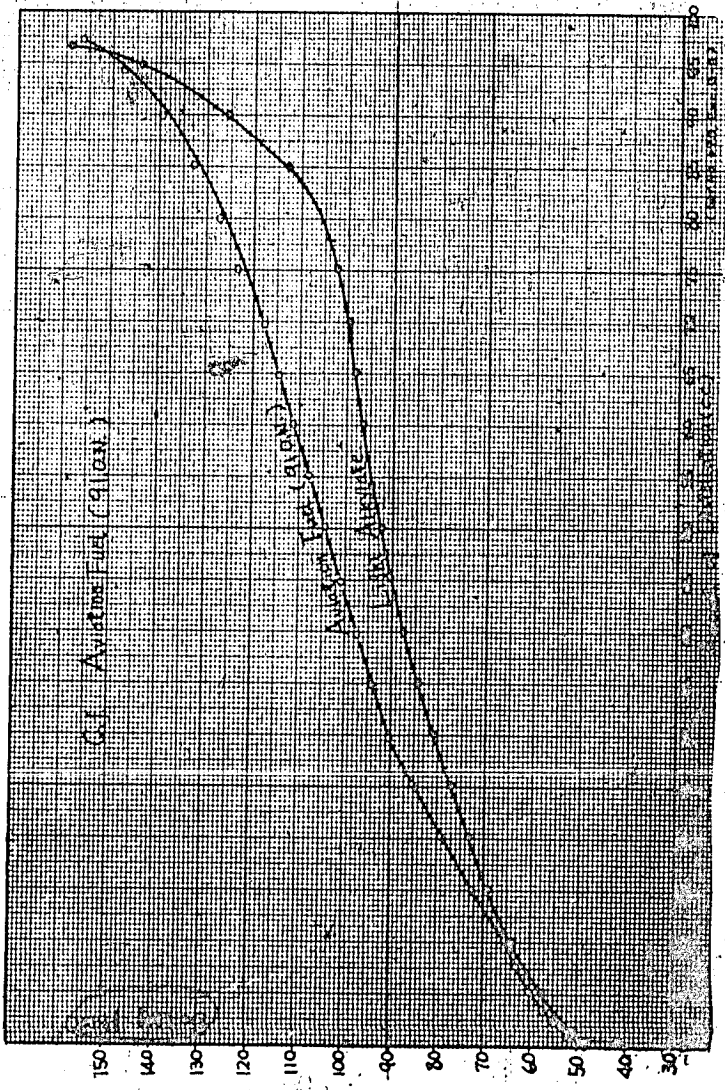


Figure 12(B) 8
DISTILLATION CURVE

RESTRICTED

X-38(N)-2

US NAVAL TECHNICAL MISSION TO JAPAN RESTRICTED

REACTION SECTION ALKYLATION PILOT PLANT

19 DEC 45

PLATE I (B) 8

X-38(N)-2

