

ENCLOSURE (B) 3

STUDIES ON THE PREPARATION  
OF AEROENGINE OILS FROM SHALE OIL

by

CHEM. ENG. COMDR. DR. I. KAGHIRA

CHEM. ENG. LT. COMDR. H. IIMURE

CHEM. ENG. SUB. LIEUT. M. MARDA

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SUMMARY

Studies were conducted on the synthesis of a superior aero-engine oil from a kerosene or gas oil fraction of shale oil. Investigations were made especially on the pre-refining method of oil, also on the optimum conditions of polymerization of pre-refined oil and the method of recovering  $AlCl_3$  from its sludge. Results obtained were as follows:

1. A kerosene or gas oil fraction contains about 20-30% of basic, acidic and highly unsaturated compounds and it is necessary to remove these compounds before the polymerization to synthesize an aero-engine oil. It was found that the best results were obtained by pre-refining of the raw material with HCl gas and NaOH solution and polymerization of the pre-refined oil. By this method, about 10% of a superior aero-engine oil from a kerosene or gas oil fraction of shale oil was obtained.

The optimum conditions for each procedure are given in Figure 1(B)3.

2. Basic, acidic and highly unsaturated compounds were almost completely removed by treating with NaOH solution and concentrated sulphuric acid. It was difficult to prepare an excellent aero-engine oil by this pre-refining method, but it is suitable for the synthesis of a mobile oil. Actually, these studies were utilized in the preparation of aero-torpedo-engine oil. The optimum conditions of each procedure are shown in Figure 2(B)3.

3. The recovery of aluminium chloride from its sludge was accomplished by distilling off oil from the sludge at  $390^{\circ}C$ , coking the distillation residue at  $500-600^{\circ}C$  for 1-3 hours and, finally, the chlorination of the cokes at  $800^{\circ}C$ . The theoretical yield of aluminium chloride recovered was about 80% and its activity was almost the same as that of  $AlCl_3$  obtained from the market.

I. INTRODUCTIONA. History

Shale oil was one of the important raw materials for preparing aero-engine oils in Japan, but it contains about 20-30% of acidic, basic and highly unsaturated compounds. These compounds must be removed before its polymerization to prepare aero-engine oils from shale oil. The authors studied especially the preliminary purification of higher fractions of shale oil with HCl gas and caustic soda solution or caustic soda solution and sulphuric acid, and also the optimum conditions of polymerization of pre-refined oil and the recovery of  $AlCl_3$  from its sludge.

B. Research Personnel

Chem. Eng. Lt. Comdr. N. IZUMI

Chem. Eng. Lieut. N. KANDA

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## II. DETAILED DESCRIPTION

### A. Raw Materials

The kerosene and gas oil fraction of shale oil, used as the raw materials for this research project, were obtained from FUSHUN in South Manchuria, and were prepared as described in Figure 3(B)3. Their properties are shown in Table I(B)3.

### B. Procedures and Results

Studies were carried especially on the pre-refining method of acidic, basic and highly unsaturated compounds contained in the raw materials, and also on the optimum conditions of polymerization of pre-refined oil and the recovering of  $AlCl_3$  from its sludge. The procedures and the results are described as follows:

#### 1. Pre-refining Raw Materials

a. Hydrochloric Acid Caustic Soda Method. Hydrochloric acid reacts with highly unsaturated and basic compounds and changes them to oil-insoluble matter, except in the case of monoolefines or aromatics. Caustic soda reacts with acidic compounds, forming a soap. Utilizing these behaviors, hydrochloric acid gas and caustic soda were used as the pre-refining agents. Results obtained were as follows:

(1) Hydrochloric Acid Gas Treatment. A gas oil fraction of shale oil was treated with various amounts of hydrochloric acid gas by introducing it into the oil, at  $34-450C$  and then separating the oil part and the sludge. The hydrochloric acid absorbed in the oil was determined by the increase in weight of oil. The results obtained are given in Table II(B)3.

From these results it was observed that the greater part of acidic and basic compounds and all asphaltic compounds were removed by 2% of HCl gas absorbed.

When the gas oil fraction was treated with the separated sludge before the HCl gas treatment, the removal of basic compound was completely accomplished by 3% of HCl gas as shown in Table III(B)3.

(2) Alkali-Treatment. All the basic and asphaltic compounds and most of the acidic compounds in the gas oil fraction could be removed as sludge by pre-treatment with hydrochloric acid sludge and 3% of hydrochloric acid gas, but 1 - 2% of the acidic compounds still remained.

To remove the residual acidic compounds, a gas oil fraction treated with 3% of HCl gas was washed at various temperatures with 20% NaOH solution. Results obtained are shown in Table IV(B)3.

These results showed that the higher the temperature and the amount of NaOH solution, the lower the content of acidic compounds in the treated oil. Next, the effect of

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the amount of 20% NaOH solution on the content of acidic compounds of treated gas oil was examined, and the following results were obtained (See Table V(B)3).

From the results in Table IV(B)3 and V(B)3, it appears that the acidic compounds in a gas oil could be almost completely removed by treating with 50% or 20% NaOH solution at 80°C. Properties of the refined kerosene and gas oil obtained by the above mentioned method are shown in Table VI (B)3.

b. Caustic Soda - Sulphuric Acid Method. By treating with concentrated caustic soda solution at comparatively high temperature, the acidic compounds are converted to soap, and the unstable nitrogen compounds and highly unsaturated compounds polymerize to higher molecules. If the product obtained by the alkali treatment is washed with hot water to remove the remaining free alkali and treated with sulphuric acid, the unstable nitrogen and highly unsaturated compounds should be almost completely removed. Working on this assumption, the authors studied the optimum conditions of alkali and sulphuric acid treatment.

A gas oil fraction of shale oil was used as the raw materials and the degree of the removal of acidic, basic and highly unsaturated compounds was judged by the ease of separation of sludge and the yield and specific gravity of the refined product. The results obtained were as follows:

(1) Necessary Amount of Caustic Soda Solution. A gas oil was washed with various amounts of 1.0% NaOH solution at 80°C for 20 minutes and allowed to stand for 20 minutes. The separated oil was washed with an equal volume of hot water and filtered with 3% of CaCl<sub>2</sub>.

Results obtained are given in the Table VII (B)3.

From these results it was observed that 15% - 20% of 10% NaOH solution was required in the treatment of a gas oil.

(2) Effect of Temperature on the Alkali Treatment. A gas oil was treated with 15% by weight of 10% NaOH solution at various temperatures. Results are given in the Table VIII (B)3 and it was observed that the suitable treating temperature was about 60°C.

(3) Necessary Amount of Concentrated H<sub>2</sub>SO<sub>4</sub>. A gas oil treated with 15% by weight of 10% NaOH solution and washed with an equal volume of hot water three times, was used as a sample. The sample was treated with various amounts of 98% H<sub>2</sub>SO<sub>4</sub> at temperatures below 45°C for 10 minutes with vigorous agitation and allowed to stand for 30 minutes. The separated oil was filtered with 5% of acid clay and distilled in vacuum to cut a gas oil fraction (boiling from first drop to 300°C, the calculated temperature at 760 mmHg).

Results obtained are given in the Table IX (B)3 and it was observed that the necessary amount of H<sub>2</sub>SO<sub>4</sub> to be used was 6%.

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2. Polymerization of Pre-refined Oila. Polymerization of the pre-refined oil obtained by treatment with HCl gas and NaOH.

Studies were carried out on the effect of temperature and time in the polymerization process on the properties of the final product. The raw material used was prepared by treating a gas oil fraction with hydrochloric acid sludge, 2% of HCl gas and 50% of 20% NaOH solution and distilling up to 300°C. Procedures taken for this study were as follows:

The pre-refined gas oil was subjected to polymerization in the presence of  $AlCl_3$  and after standing for 24 hours, the sludge was separated. The polymerized product was treated with 5% of acid clay, 10% NaOH solution and hot water, and distilled in vacuum. Three cuts were obtained: the diesel oil fraction (up to 350°C at 760mm Hg), the light lubricating oil fraction (350 - 450°C at 760mm Hg) and the aero-engine oil fraction (450°C over at 760mm Hg). Results obtained are as follows:

(1) The effects of the polymerization temperature on the yield of an aero-engine oil fraction are given in the Table I(B)3. It was observed that the polymerization proceeded incompletely at lower temperatures and that cracking partially occurred at higher temperature. The optimum temperature for polymerization was 80 - 100°C from the view point of the yield of aero-engine oil.

(2) The effect of reaction time on the polymerization is shown in Table II(B)3 and it was observed that the longer the reaction time, the higher the yield but the lower the viscosity index of aero-engine oil fraction. From the view point of viscosity index of product, the suitable reaction time should be 10 hours.

(3) The amount of catalyst ( $AlCl_3$ ) used should be the most important factor in the polymerization. Hence, the raw material was subjected to polymerization in the presence of various amounts of  $AlCl_3$  at 80°C for 10 hours and the properties of the products and its aero-engine oil fraction was examined in detail. Results obtained were tabulated in Table III(B)3. From these results, it was observed that a superior aero-engine oil was prepared by polymerization in the presence of 10% or more of  $AlCl_3$ .

b. Polymerization of Pre-refined Oil Prepared by Treatment With Caustic Soda Solution and Sulphuric Acid. Raw material used for the polymerization was prepared from the gas oil fraction by treating with 20% by weight of 10% NaOH solution and 6% of 98%  $H_2SO_4$  and distilling to 300°C. The raw material was polymerized by the same procedure as described in the section (b) - (1). Results obtained are given below.

Yield for Product of Polymerization (wt%) .....	10.3
Properties	
Density (15/4) .....	0.9041
Viscosity in S.U.S. at 210°F .....	111.9
Viscosity Index .....	77

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Four Point .....	-19.5
Conradson's Carbon (%) .....	0.49

Comparing with the results shown in Tables XII(B)3, it was concluded that the hydrochloric acid gas - caustic soda method was a better pre-refining process than the caustic soda-sulphuric acid method from the view point of preparing an aero-engine oil in high yield and with superior properties.

C. Recovery of AlCl<sub>3</sub> from its Sludge

Aluminium chloride sludge separated with a yield of about 20% by weight in the polymerization stage. It consisted of 5% aluminium, 17.5% chlorine and 72.6% oil. Its mean molecular weight was 296, and the composition of sludge could be indicated as 2.88 parts AlCl<sub>3</sub> and 1.51 parts oil. From an economical point of view, the recovery of AlCl<sub>3</sub> from the sludge is an important problem. The authors, therefore, adopted a method, consisting of the distillation of the oil, coking the distillation residue and chlorinating the cokes. The optimum conditions of each procedure were studied and also the activity of recovered AlCl<sub>3</sub> as the polymerization catalyst. Results obtained were as follows:

1. Distillation of Aluminium Chloride Sludge. An aluminium chloride sludge was distilled in a three-necked flask at 375°C - 390°C. Results obtained are tabulated in Table XIII(B)3.
2. Coking of Residue. The residue obtained by the distillation of aluminium chloride sludge was subjected to coking in a porcelain tube at 500°C - 800°C by electrical heating. Results obtained are shown in Table XIV(B)3 and it was observed that coking the residue prepared by the distillation of aluminium chloride sludge should be carried out at 500°C - 600°C for 1 - 3 hours, from the point of view of higher yield of cokes and larger content of chlorine in the cokes.
3. Chlorination of Cokes. Cokes were subjected to chlorination in a porcelain tube electrically heated at 600 - 800°C by treating with chlorine gas. The results obtained are shown in Table XV(B)3. It was observed that the higher the chlorination temperature the larger the yield of recovered AlCl<sub>3</sub>.
4. Activity of Recovered Aluminium Chloride as the Polymerization Catalyst. The activity of recovered aluminium chloride was determined by comparing the iodine values of the polymerized products of pre-refined gas oil fractions when recovered AlCl<sub>3</sub> and when commercial grade AlCl<sub>3</sub> was used.

The results obtained are shown below. From these results, it may be concluded that the activity of recovered AlCl<sub>3</sub> is a little less than that of the commercial material but that it may be compensated by using a little more catalyst.

Yield of Pre-refined Oil for Gas Oil (wt%) .....	71.0
Properties of Pre-refined Oil	
Density (15/4) .....	0.8251
Iodine Value .....	57.0

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AlCl<sub>3</sub> Used for Polymerization Iodine Value of Polymerized Products

5% of commercial catalyst	28.0
5% of recovered catalyst	42.7
7% of recovered catalyst	25.0

III. CONCLUSIONS

The kerosene or gas oil fraction in shale oil contains about 20 - 30% of basic, acidic and highly unsaturated compounds and these must be removed before polymerizing for the preparation of an aero-engine oil. Hence, studies were conducted pertaining to the pre-refining method and it was found that a method consisting of treating with hydrochloric acid gas and concentrated caustic soda solution was the best as the pre-refining method for the preparation of an aero-engine oil and a method consisting of treating with caustic soda solution and concentrated sulphuric acid was suitable for the preparation of a mobile oil or a generally used lubricating oil. Actually, these studies were utilized in the preparation of the aero-torpedo engine oil from shale gas oil.

The scheme of these procedures is given in Figures 1(B)3 and 2(B)3.

Table I(B)3  
 PROPERTIES OF THE KEROSENE AND GAS OIL FRACTIONS  
 OF SHALE OIL

		Kerosene Fraction	Gas Oil Fraction
Properties	Boiling Point (°C)	180 - 250	200 - 300
	Density (15/4)	0.8217	0.8607
	Flash Point (°C)	60.5	73.0
	Pour Point (°C)	-28.5	-13.0
	Iodine Value	90.6	63.1
Composition of Fraction	Acidic Compound (%)	3.4	8.8
	Basic Compound (%)	2.8	4.6
	Neutral Oil (%)	93.8	86.6
Composition of Neutral Oil	Unsaturation (%)	27.6	
	Aromatics (%)	13.8	
	Naphthenes (%)	0.0	
	Paraffines (%)	58.6	



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Table II(B)3  
ANALYSIS OF HYDROGEN CHLORIDE TREATED PRODUCT  
OF GAS OIL FRACTION

Composition of Oil Separated (wt %)	HCl Gas Absorbed in the Oil (wt %)							
	0	0.5	0.75	1.0	1.5	2.0	3.0	5.4
Acidic Compound	7.32	3.77	3.17	5.64	1.10	1.32	1.78	0.52
Basic Compound	6.53	5.83	4.37	5.79	1.66	0.51	0.78	0.60
Asphaltic Compound	14.85	3.48	3.27	1.53	0.10	0	0	0
Neutral Oil	71.30	86.92	89.19	87.04	97.14	98.17	98.44	98.80

Table III(B)3  
ANALYSIS OF PRODUCT OF GAS OIL FRACTION TREATED WITH  
HYDROCHLORIC ACID SLUDGE BEFORE HYDROCHLORIC ACID TREATMENT

	HCl Gas Absorbed in the Oil (wt %)				
	1	2	3	4	5
Oil Separated (wt %)	82.9	85.3	82.9	85.8	85.9
Composition of Oil Separated (wt %)					
Acidic Compound	1.62	1.22	0.59	0.26	
Basic Compound	0.36	0.15	0	0	
Neutral Oil	98.12	98.63	99.41	99.74	

Table IV(B),  
EFFECT OF TREATMENT WITH 20% NaOH SOLUTION ON THE CONTENT  
OF ACIDIC COMPOUNDS IN GAS OIL AT VARIOUS TEMPERATURES

	Amount of 20% NaOH Solution (%)			
	5	10	15	
Content of Acidic Compounds in the Treated Oil	at 30°C	1.56	0.70	0.84
	at 50°C	1.92	0.76	0.80
	at 80°C	1.44	0.64	0.50

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Table V(B)<sub>3</sub>  
EFFECT OF 20% NaOH SOLUTION ON THE CONTENT OF ACIDIC  
COMPOUND IN GAS OIL FRACTION\*

	Amount of 20% NaOH Solution		
	30	50	100
Yield of Treated Oil (wt % for HCl Treated Oil)	86.7	84.5	83.3
Content of Acidic Compounds in the Treated Oil	0.32	0.32	0.12
Content of Basic Compounds in the Treated Oil	0.36	0.28	0.12

\*Temperature of treating. 80°C

Table VI(B)<sub>3</sub>  
PROPERTIES OF REFINED KEROSENE AND GAS OIL FRACTION AFTER  
TREATMENT WITH 3% HCl GAS AND 50% OF 20% NaOH SOLUTION AT 80°C

	Kerosene		Gas Oil	
	Raw Material	Refined Oil	Raw Material	Refined Oil
Density (15/4)	0.8217	0.8136	0.8607	0.8308
Pour Point (°C)	-28.5	-28.0	-13.0	-11.0
Conradson's Carbon (wt %)	0.19	0.07	0.83	0.12
Content of Acidic Compounds (wt %)	2.8	0.48	8.8	0.4
Content of Basic Compounds	3.4	0.40	4.8	0.4

Table VII(B)<sub>3</sub>  
EFFECT OF AMOUNT OF NaOH SOLUTION IN TREATING  
SHALE GAS OIL

	Amount of 10% NaOH Solution used (wt %)				
	5	10	15	20	25
Refined Oil Yield (wt %)	95.0	92.5	91.0	92.0	91.5
Density (15/4)	0.8490	0.8471	0.8459	0.8453	0.8457
Separation of Oil	*	easy	easy	easy	easy

\*A small amount of tarry matter remained in the oil.

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Table VIII(B)3  
EFFECT OF TEMPERATURE ON ALKALI TREATMENT

	Treating Temp. (°C)				
	20	35	45	60	80
Refined Oil Yield (wt %)	91.0	91.5	90.5	92.5	91.5
Density (15/4)	0.8447	0.8452	0.8450	0.8468	0.8459
Separation of Oil	**	*	*	easy	easy

\*Soft tarry matter separated.  
\*\*Hard tarry matter separated.

(See page 72 for Table IX(B)3.)

Table I(B)3  
EFFECT OF TEMPERATURE ON POLYMERIZATION\*

Reaction Temp. °C	Product of Polymerization		Aero-Engine Oil Fraction	
	Mean M.W.	Iodine Value	Yield (wt %)	Mean M.W.
50	275	42.9	12.4	731
60	281	33.5	20.2	720
100	282	22.7	18.2	623
130	258	23.5	9.6	640

Note: In each case, reaction time, eight hours, and 5% AlCl<sub>3</sub> (wt) were used.

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Table II(B)3  
EFFECT OF AMOUNT OF 96% H<sub>2</sub>SO<sub>4</sub> IN TREATING SHALE GAS OIL

96% H <sub>2</sub> SO <sub>4</sub> Used (wt %)	Treating Temperature (°C)	Separation of Sludge and Oil	Property of Sludge	Yield of Product for Raw Material (wt %)	Filtered Product			Fract. Boiling from Lab. Depp to 300°C of Filtr. Prod.		
					Yield for Raw Material (%)	Density (15/4°C)	Iodine Value	Density (15/4)	Iodine Value	Yield of Filtered Product (wt %)
2	24	easy	hard	96.0	0.8380	61.7	0.8353	61.2	85.0	
4	27.5	easy	hard	87.7	0.8290	63.1	0.8297	62.1	85.2	
6	27.0	easy	soft	82.0	0.8280	62.0	0.8251	61.1	84.7	
8	21.0	easy	soft	88.7	0.8252	61.8	0.8231	64.1	85.1	
10	19.0	easy	soft	89.4	0.8250	60.1	0.8254	61.7	85.3	

Table II(B)3  
EFFECT OF REACTION TIME ON POLYMERIZATION\*

Reaction Time (hr)	Percent of Polymerization	Fraction of Product											
		Diesel Oil Fraction (30-450°C)					Aero-Engine Oil Fraction (450°C-)						
20	97.3	21.2	57.4	22.6	0.8692	13.4	76	-45	14.6	0.9032	126.6	81	-25
20	84.1	11.7	21.6	0.8685	13.1	64	-49	17.7	0.9053	127.4	79	-27	
													Yield (wt %)
20	84.1	14.9	40.0	14.3	0.8677	12.0	55	-50	18.8	0.9051	118.1	73	-27

\*20 each case, reaction temperature, 40°C, and 55 AlCl<sub>3</sub> (wt) were used.

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Table XIII(B)3  
EFFECT OF AMOUNT OF  $AlCl_3$  ON POLYMERIZATION

Conditions of Polymerization	AlCl <sub>3</sub> used (wt %)		12	10	7	5
	Reaction Temp(°C)		80	80	80	80
	Reaction Time(hr)		10	10	10	10
Product of Polymerization	Yield (wt %)		85.0	87.5	90.0	87.0
	Iodine Value		10.5	11.2	14.3	20.8
Aero-Engine	Viscosity in S.U.S.at 250°F		129.5	100.5	94.5	103.4
	Viscosity Index		105	104	96	84
Oil	British	Viscosity Ratio	1.58	1.60	1.81	2.00
		Conradson's Carbon (%)	1.29	2.02	2.36	3.66
	Air Ministry Oxidation Test	Acid Value	0.31	0.35	0.35	0.36
		Saponif. Value	14.09	15.73	18.32	17.29
Fraction	Yield for Product (wt %)		23.0	23.5	20.1	14.2

Table XIII(B)3  
DISTILLATION OF ALUMINIUM CHLORIDE SLUDGE

Exp. No.	Distillation Temp. (°C) (in sludge)	Yield of Residue (wt %)	Distilled Oil		Gas	
			Yield (wt %)	Density $\frac{15}{4}$	Yield (wt %)	Calorific Power Cal/m <sup>3</sup>
1	375	42.8	44.0	0.815	13.2	5856
2	390	42.0	44.0	0.815	14.0	9652
3	390	41.0	43.9	0.830	15.1	17150

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Table XIV(B)3  
EFFECT OF TIME AND TEMPERATURE OF COKING  $AlCl_3$  SLUDGE

Coking		Yield of Cokes (wt %)		Composition of Cokes			
Temp. (°C)	Time (hr)	For Residue	For Sludge	Al (wt %)	Cl (wt %)	C (wt %)	Experimental Formula
500	1	73.1	30.7	17.10	17.52	65.39	$AlCl_{0.78}$
600	1	75.0	36.4	12.39	14.45	73.16	$AlCl_{0.89}$
600	3	74.1	32.3	12.59	14.51	79.90	$AlCl_{0.96}$
700	1	66.2	28.8	10.43	9.86	79.71	$AlCl_{0.72}$
700	3	63.0	27.4	8.94	9.34	81.72	$AlCl_{0.80}$
800	1	60.9	26.5	9.89	9.85	80.26	$AlCl_{0.76}$
800	3	62.3	27.1	9.69	9.41	80.92	$AlCl_{0.74}$

Table XV(B)3  
CHLORINATION OF COKE\*

Coking of Residue		Chlorination of Cokes		Yield of $AlCl_3$ Recovered for Sludge (wt %)
Temperature (°C)	Time (hr)	Temperature (°C)		
500	1	600		58.0
500	1	700		79.4
500	1	800		83.0
500	3	600		71.8
500	3	700		79.4
500	3	800		90.3
600	3	600		76.6
600	3	700		70.6
600	3	800		77.3

\*In each chlorination, time was 3 hours, and the passing rate of chlorine was 5 lit/hr/20gms of coke.

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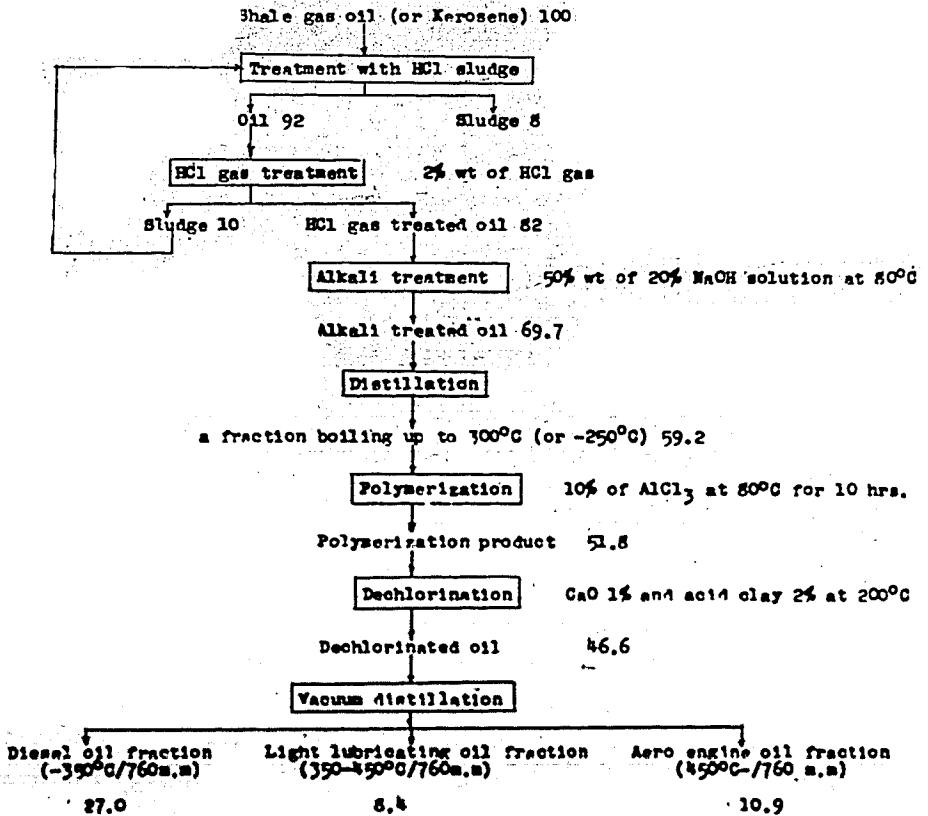


Figure 1(B)3  
 SCHEME FOR THE PREPARATION OF AN AEROENGINE OIL  
 FROM SHALE GAS OIL OR KEROSENE

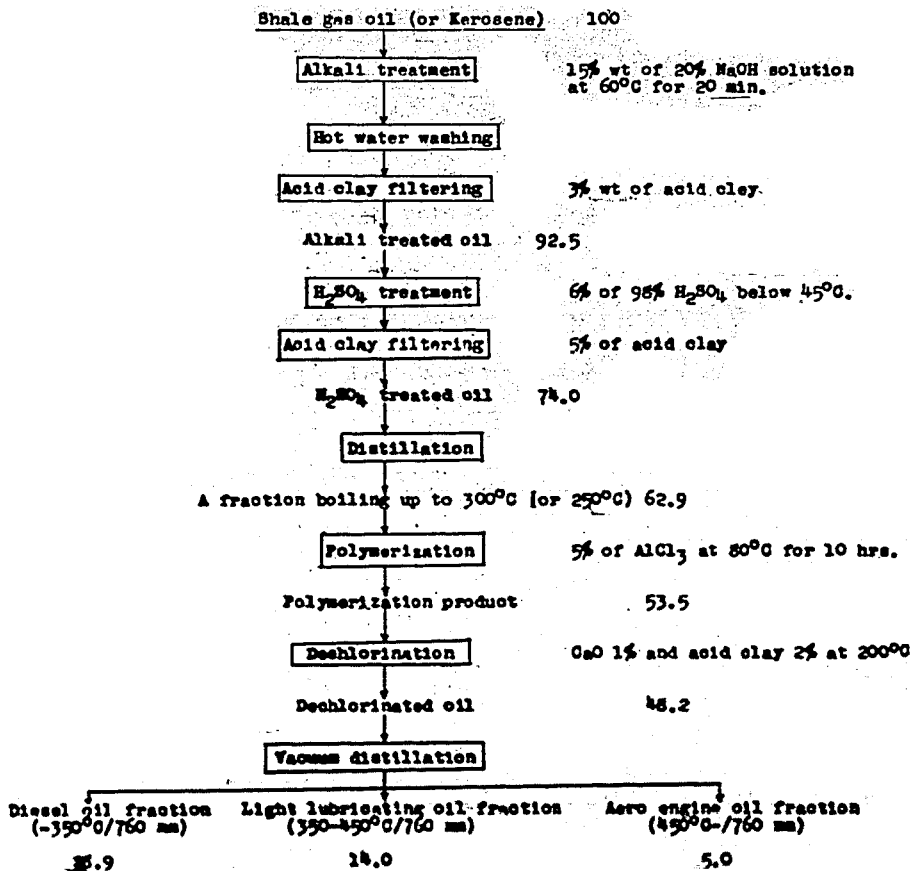
ENCLOSURE (B)<sub>2</sub>

Figure 2(B)<sub>2</sub>  
 SCHEM FOR THE PREPARATION OF A MOBILE OIL  
 FROM SHALE GAS OIL OR KEROSENE



ENCLOSURE (B)

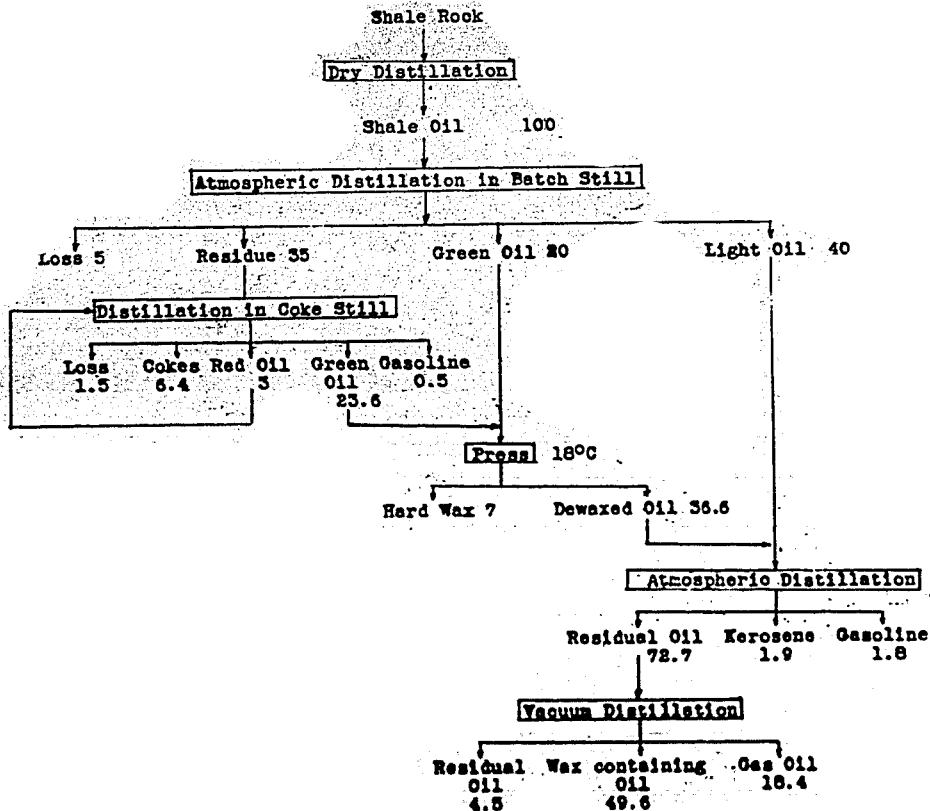


Figure 3(B)3

SCHEME OF PRODUCTION OF GAS OIL AND KEROSENE FROM SHALE

ENCLOSURE (B)3

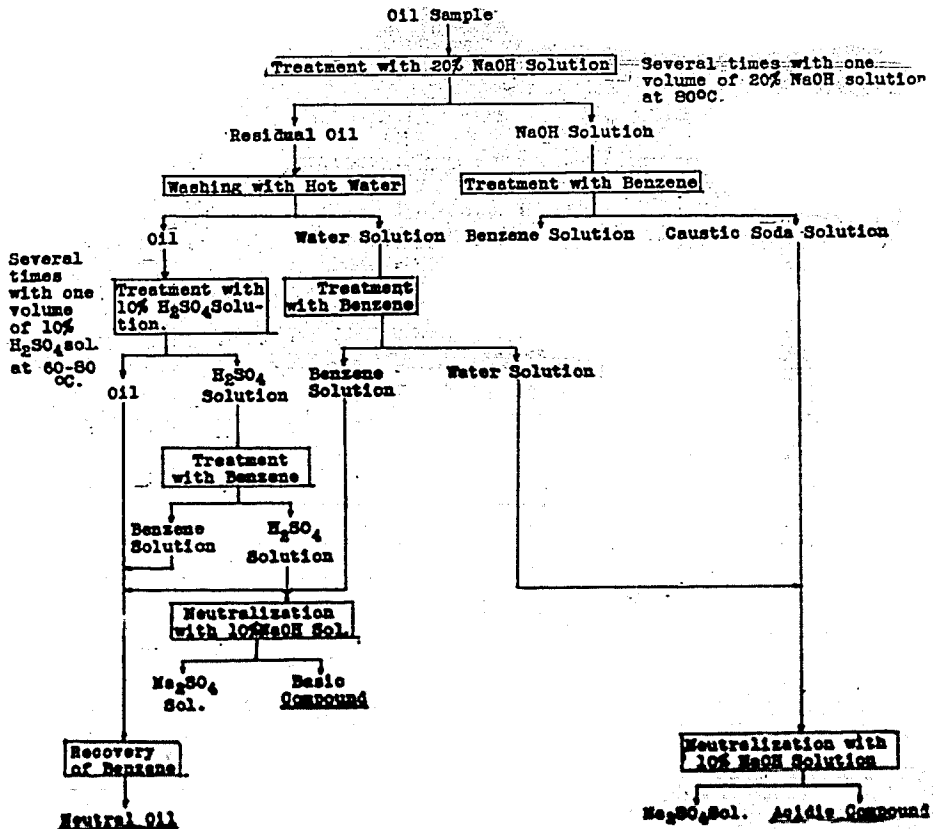


Figure 4(B)  
METHOD FOR DETERMINING ACIDIC AND BASIC COMPOUNDS IN SHALE OIL