

# **A Study on the Alkylation of Coker and Straight-Run Naphtha Fractions for the Production of Middle Distillate Fuels Using Conc. Sulphuric Acid Catalyst. Part 1.**

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## **Abstract**

The alkylation of both coker gasoline and straight-run (S.R.) naphtha fractions, using Sulphuric acid catalyst is investigated. The production of alkylates in the range of kerosene and gas oil (middle distillate fuels) are the main objective of the study.

The alkylation reaction was carried out at the following experimental conditions; reaction temperature 0 °C, space velocity 0.35 and acid / feed ratio 3:1. The effects of varying total alkylating agents to olefins were investigated. The study shows that using an excess of alkylating agents in the reaction zone promotes the primary alkylation reaction with olefins on the expense of side reactions.

**Key words:** alkylation, straight-run naphtha, Sulphuric acid, middle distillate, gasoline, kerosene, gas oil.

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## Introduction

Alkylation reaction between isoparaffins and olefins in the presence of Sulphuric acid produces the alkylate which has excellent properties suitable for blending in improving the quality of the fuels as for example, the middle distillates.

Concentrated sulphuric acid catalyst, in the United States, sulphuric acid was used as catalyst for production of about 53% of the alkylate or gasoline produced by alkylation (1), the remainder use HF. In the 1960s, ~ 75% of the alkylates produce used H<sub>2</sub>SO<sub>4</sub> catalyst. From the 1960s to around 1986, the relative importance of alkylations using HF increased as compared with those using H<sub>2</sub>SO<sub>4</sub>. UOP and Philips Petroleum Co. developed HF processes that were well received. In 1986 tests, liquid HF was released to the atmosphere (2). Under Select conditions, HF can form aerosol clouds which contain lethal levels of HF and travel at ground level for several miles down ward. In 1987, the accidental release of gaseous HF in Texas City resulted in emergency treatment for several hundred people.

In the A.R.E., there is a surplus of gasoline which amounts to about one million ton. On the other hand, there is a shortage of about half a million ton of middle distillate fuels such as kerosene and gas oil. A problem which appears to be unique in the world.

In this study the alkylation of both coker gasoline (main source of olefins) and S.R. naphtha fractions is carried out to form a highly branched-chain paraffinic product.

The coker distillates produced from the thermal cracking of topped crude oil, are fractionated to yield the coker gasoline which is enrich in olefins. The S.R. naphtha produced from primary distillation of crude oil (the naphtha fraction used in this work) contain an appreciable

quantities of alkylating agents such as cycloparaffins, isoparaffins and aromatics.

Intensive work has been done before dealing with the problem of production of middle distillate fuels from lighter products (3-7) which is the main objective of this work.

## **Experimental**

### **Feed stocks:**

- 1- Coker gasoline: A sample of Land Balayim coker product being hydrogenated was kindly supplied by the Suez Petroleum Processing Company. Coker gasoline was fractionated to obtain a fraction of boiling range 100-160 °C. This fraction represented 67.4 by volume of the coker product.
- 2- Heavy naphtha: A sample of heavy naphtha fraction boiling range 77-165 °C was kindly supplied by Cairo Refining Company.

### **Properties and carbon type analysis of feed stocks:**

- Refractive index was measured by an abbe refractometer.
- Specific gravity (8).
- ASTM distillation (9).
- Average molecular weight (10) was carried out by standard methods.
- Total saturates olefins and aromatics contents were carried out by florescence indicator adsorption method (FIA) (11).
- The total naphthenes content were determined by refractivity intercept-density method (12).

The saturates of heavy naphtha and coker fraction were obtained after treating each fraction by conc. Sulphuric acid (98%) to remove olefins and aromatics.

Normal paraffins determination by using gas liquid chromatographic analysis (a Perkin Elmer Sigma 3B chromatograph) equipped flame ionization detector (FID), 6 ft. packed OV- 101 column.

#### **Alkylation reaction:**

Alkylation of S.R. naphtha with coker fraction catalyzed by conc. Sulphuric acid was carried out in a 500 ml three necked round bottom flask. The flask was fitted with a mechanical stirrer, graduated burette and a condenser fitted with anhydrous calcium chloride gage. The flask was immersed in thermostate adjusted at 0 °C temperature. The reaction flask was charged with the calculated amount of Sulphuric acid and calculated amount of S.R naphtha, stirred for 10 minutes at 0 °C. The required amount of coker fraction was then transferred into the flask by the burette according the appreciated space velocity. Five minutes after the content of the flask was transferred to a separating funnel and allowed to settle (13).

The organic layer was separated, neutralized with 5% NaOH solution followed by distilled water and finally dried over anhydrous calcium chloride.

The alkylation product was fractionated into two cuts:

- Cut I which have a boiling range below 165 °C.
- Cut II which have a boiling range above 165 °C.

Cut II of some experiments was fractionated into two fractions cut IIa and cut IIb.

The specifications of every cut were investigated involved: specific gravity (8), refractive index, ASTM distillation (9), flash point (14), aniline point (15), pour point (16), smoke point (17) kinematic

viscosity (18), sulphur content (19), carbon residue (20), ash content (21), cetane index (22) and diesel index (23).

## Results and Discussion

In the presence of concentrated sulphuric acid, olefins with the exception of ethylene undergo alkylation reaction with hydrocarbons containing tertiary carbon atom, as well as with aromatics (24-26). The product of alkylation is fully saturated. It was until 1938 that a reaction using sulphuric acid catalyst appeared in the literature (27).

The alkylation reaction produces different types of products, primary one and others are produced via side reactions (28). The side reaction products are produced via, polymerization, polyalkylation, destructive alkylation and hydrogen-transfer reactions. The first two types of these side reactions produce heavier reaction products than the primary one that produced through the alkylation reaction. Meanwhile the products are rich in isomeric species. Light products are produced via the destructive alkylation process.

The alkylation reaction using concentrated sulphuric acid catalyst, may fit well the use of olefins in coker gasoline (before being hydrogenated) to alkylate a heavy naphtha fraction to produce middle distillate fuels. The boiling range of locally produced kerosene is 150-300°C (about C<sub>9</sub>-C<sub>17</sub>). The solar-diesel fuels have a boiling range of 300-400°C (about C<sub>17</sub>-C<sub>25</sub>). The standard specifications of these petroleum products are reported (29). The coker gasoline fraction used in the investigation has a boiling range of (100-160°C), while the heavy naphtha fraction is (77 - 165°C ).

The alkylation of heavy naphtha fraction of a boiling range 77-165°C (C<sub>6</sub>-C<sub>9</sub>) with coker gasoline fraction of boiling range 100-160°C

(C<sub>7</sub>-C<sub>9</sub>) was investigated using concentrated sulphuric acid catalyst under experimental conditions which are not necessarily optimum. The effect of total alkylating agents (Aromatics, Isoparaffins and Cycloparaffins, except cyclohexane) to olefins mole ratios is discussed throughout this work.

### **Catalytic Alkylation of Coker Gasoline with Straight Run Naphtha. Properties of Gasoline Fraction.**

Data given in Table 1 show that the concentration of the coker fraction of a boiling range 100-160°C is about 67.4% by vol. of the full-range gasoline. The refractive index,  $n_D^{20}$ , specific gravity, 20/4, average molecular weight, total sulphur content, wt.% and ASTM distillation data are also given. On the other hand, the specifications of heavy naphtha fraction of a boiling range 77-165°C are also given in Table 1.

Tables 2,3 represent the gas chromatographic analysis for n-paraffins of the heavy naphtha and coker fractions.

Table 4 shows the hydrocarbon group analysis for the gasoline fractions. The data reveal that the coker gasoline fraction is rich with olefins (47.3% by vol.), and it has total alkylating agents (aromatics, cycloparaffins except cyclohexane and isoparaffins), 37.0% by vol. On the other hand, the heavy naphtha fraction is rich in the total alkylating agents, 57.7% by vol.

The average molecular weight of coker gasoline fraction and heavy naphtha are 118 and 112 respectively which indicates that both fractions have average carbon number of C<sub>8</sub>. Consequently, the primary alkylation products have average carbon number of C<sub>16</sub>. On the other hand, the other alkylates produced via side reactions may be heavier or lighter than C<sub>16</sub>.

Data in Table 3 reveal also that, both coker gasoline and heavy naphtha fractions have non-alkylating agents which are normal paraffins

and cyclohexane in concentrations 15.7% and 42.3% by wt. respectively. These non-alkylating agents have no tertiary carbon atoms, which form the most stable carbonium ions that are involved in the mechanism of alkylation reaction.

### **Alkylation of Heavy Naphtha with Coker Gasoline Using Sulphuric Acid Catalyst**

Sulphuric acid catalyst is one of the most important alkylation catalysts which has been early used (24). It has a good advantage above other catalysts that the product of alkylation obtained is mainly free from impurities such as sulphur compounds as well as nitrogen compounds specially when this catalyst is used for alkylation of petroleum hydrocarbons which contains such compounds as impurities.

Many variables affect the alkylation process using sulphuric acid catalyst (25), as reaction temperature, acid concentration, mixing degree of reactants, feed addition rate, acid to feed ratio as well as the mole ratio of olefins/alkylating agents.

The reaction temperature was one of the most important parameter which highly affects the reaction products (24,25,27). As the reaction temperature becomes higher than 0 °C, another competitive undesirable reaction product can be formed through the sulphonation and oxidation for olefins and aromatics. At the same time, decreasing the reaction temperature below 0 °C, increases the viscosity of the acid leading to poor mixing of reactants with the acid which highly affects the yield of the alkylation reaction (28, 31).

Acid concentration is one of the most important parameter that affects the alkylation reaction. The concentration of sulphuric acid used in this reaction was about 96%. Below this acid concentration the activity of the catalyst decreases, and the other side reaction arises such as the

polymerization of olefins. On the other hand, higher acid concentration accelerate the undesirable side reactions as oxidation and sulphonation.

Efficient mixing of the reactants with the acid catalyst has been used to provide good phase contact between the acid and hydrocarbon feed. This good contact between the catalyst and the hydrocarbons accelerates the alkylation reaction due to the ease transfer of the acid protons to the reactants to form the carbonium ions.

Space velocity of 0.35 volume of feed per volume of sulphuric acid catalyst per hour (vol./vol./hr) was used. Shorter space velocities were of little effect on the yield of total alkylate, probably because of the high reactivity of the alkylation reaction (32).

Acid to hydrocarbon ratio 3/1 weight ratio was used, the amount of acid in the acid-hydrocarbon reaction mixture had an important effect on the alkylate yield and quality. Reduction of this amount led to more unsaturation and lower yields of the alkylate products. On the other hand, high ratios of acid were undesirable because of the consumption represent a major operating cost in an alkylation unit of this type (33).

#### **Effect of Varying Total Alkylating Agents (Cycloparaffins, Isoparaffins and Aromatics) to Olefins Weight Ratio.**

The study of this effect was carried out using total alkylating agents to olefins wt. ratios in the range of 4/1 to 1/1. The experiments were carried out at 0°C reaction temperature, a feed space velocity of 0.35 vol./hr./vol. and an acid to feed wt. ratio of 3/1. These experimental conditions were previously investigated (33, 34).

Data obtained in Table 4 represents the study of the effect of varying the total alkylating agents to olefins wt. ratio on the alkylation products obtained. The investigation was carried out between 4/1 to 1/1 wt ratios. These used ratios were obtained by mixing up the calculated



amounts of both coker and the heavy naphtha fractions. The coker fraction alone has total alkylating agents to olefins wt. ratios of 0.6/1.

The total alkylates obtained throughout the study decreased from 85.7% to 80.0% by wt. as the total alkylating / olefins wt. ratio decreased from 4/1 to 1/1, respectively, as shown in Table 4 and Fig. 1. This means that as the total alkylating agents increases with respect to olefins in the alkylating feed; The increase in the total alkylate due to the decrease in olefins sulphonation which accompanies the increase in the probability of olefin alkylation. Consequently, the loss in reaction products throughout sulphonation products will be small as the ratio increases. At the same time the primary alkylation reaction products increased on the expense of the other side reaction product that will be mixed in the acid catalyst layer as the sulphonation products, catalyst complexes and esters formation rather than in the total alkylate layer.

The alkylation reaction gave a mixture of primary alkylation products through the reaction of the alkylating agents with the olefins of the feed. The feed mixture introduced into the reaction had an average number of 8 carbon atoms. Consequently, the primary alkylation products had an average carbon number of  $C_{16}$ .

The by products that obtained with the primary alkylation products and produced throughout many side reactions hydrogen transfer, destructive alkylation, disproportionation, polyalkylation and polymerization affected greatly the boiling range of the reaction products. This attributed to some reactions produced heavy reaction products such as polymerization and polyalkylation reactions and others lighter than the primary alkylation products such as disproportionation reactions.

### Fractional Distillation of Total Alkylate

Fractional distillation of the total alkylates produced two cuts: I and II. The boiling range of these Cuts are, (initial boiling point to 165°C) and (> 165°C to final boiling point), respectively.

Cut I contains hydrocarbons of paraffinic nature of branched and unbranched chains in the range of C<sub>6</sub> to C<sub>9</sub>. Hydrocarbon type analysis of cut I showed the absence of any olefins or aromatics Table 4.

Cut II consists of primary alkylation in addition to secondary reaction products. These products are characterized with highly branched nature. The secondary reaction products are lighter than the primary alkylation products which have an average carbon number of C<sub>16</sub>.

Data in Table 4, and represented in Fig.2, show the effect of total alkylating agents to olefins wt. ratio on the yield of cut I and cut II wt% of charge. The yield of cut I decreased as the ratio of total alkylating agents to olefins decreased, while contrary, cut II showed high yield of 20.7% wt. of charge is obtained at 1/1 ratio, while for cut I, 77.1% wt. of charge at 4/1 ratio.

The effect of the total alkylating agents to olefins wt ratio on the yields of cut II related to theoretical yield of C<sub>16</sub>, are given in Table 4 and illustrated in Fig. 3, data revealed that on increasing the ratio of total alkylating agents to olefins from 1/1 to 4/1, the ratio of cut II to theoretical yield increased from 0.29 to 0.35, respectively.

Based on the above discussed data, it is clear that the effect of the total alkylating agents to olefins wt ratios in accelerating the side reaction in case of lower ratios. It is therefore apparent that an excess of alkylation agents limits the competitive polymerization reaction of the highly reactive C<sub>8</sub> olefins that occurring simultaneously with the alkylation reaction.

## **Self Alkylation of Coker Fraction Using Sulphuric Acid Catalyst**

Data given in Table 5 represent the alkylation of the coker fraction only without using the heavy naphtha fraction in the so called self alkylation. Coker fraction had total alkylating agents to olefins wt. ratio 0.6/1. Concentrated sulphuric acid is was used as alkylating catalyst as well as the same experimental condition used as in the previous alkylation experiments.

Cut II represents 21.6% by wt. of charge. Whereas cut I is 54.5% by wt. of charge as shown in Table 5. By comparing these data with that obtained in Exp. No 4, Table 4, a slight improvement in the yield of cut II that obtained throughout self alkylation of coker fraction was found. This may due to the relative higher olefin content in the self alkylation feed (coker fraction alone) as compared with that feed in Exp. No 4, which may undergo polymerization reaction rather than sulphonation. The polymerization increased the content of cut II slightly.

## **Fractional Distillation of Cut II**

The distillation of cut II of experiment No.4, Table 4 was carried out which produced two cuts cut II-a; has b.r. 170-280°C and cut II-b, b.r 282-389°C. The specifications of each cut had been done. Data obtained are shown in Table 6.

Data of the fractional Distillation in Table 6 show that cut II-a represent 5.8%wt of charge. The distillation curve given in Fig.6, shows that cut II-a in the range of C<sub>10</sub>-C<sub>15</sub>. The other specifications for cut II-a as shown in Table 6 are found to be within the required local standard specifications of kerosene.

Data in Table 6 concerning Cut II-b revealed that it is 14.9% wt. of charge. On the other hand, the distillation curve given in Fig.7, indicate that Cut II-b lies in the range of C<sub>14</sub>-C<sub>21</sub>. The specifications of cut II-b as

given in Table 6 reveal that this product can easily meet the required local standard specifications of diesel oil. It may be noted that the pour point of cut II-b is below  $-48\text{ }^{\circ}\text{C}$  which may reflect the isoparaffinic nature of its components.

### Conclusions

The results obtained in this investigation indicate the following conclusions:

- 1- Catalytic alkylation of a coker gasoline fraction (b.r.  $100\text{-}165\text{ }^{\circ}\text{C}$ ) with a heavy naphtha fraction (b.r.  $77\text{-}165\text{ }^{\circ}\text{C}$ ) provide a possible route for the production of hydrocarbons boiling in the range of middle distillate fuels.
- 2- The alkylates obtained which boil above  $165\text{ }^{\circ}\text{C}$  to end point (cut II) are characterized by low olefin contents indicating that primary alkylation is the predominant reaction. Consequently, the side reactions, especially polymerization are minimized.
- 3- In general the properties of cut IIa as well as the specifications of cut IIb are within the required local specifications of kerosene and gas oil , respectively.

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**Table 1: Properties of Gasoline Fractions**

<b>Test</b>	<b>Heavy naphtha</b>	<b>Coker fraction</b>
	b.r. 77-165 °C	b.r. 100-160 °C
Vol% of full range gasoline	100	67.4
Refractive index, $n_D^{20}$	1.4141	1.4226
Specific gravity, 20/4	0.7263	0.7514
Average molecular weight	112	118
Total sulphur content, wt.%	0.025	0.36
ASTM Distillation, °C		
I.b.p.	77	42
10%	88	94
20%	92	107
30%	102	119
40%	112	123
50%	120	130
60%	128	136
70%	135	141
80%	145	146
90%	151	153
F.b.p.	165	155

**Table 2: Gas Chromatographic Analysis for n-Paraffins of Heavy Naphtha**

<b>n-Paraffin:</b>	<b>wt%</b>
n-Pentane	1.2
n-Hexane	3.2
n-Heptane	13.4
n-Octane	12.3
n-Nonane	11.0
n-Decane	1.2
<b>Total n-Paraffins</b>	<b>42.3</b>

**Table 3: Hydrocarbon Group Analysis for Gasoline Fractions**

<b>Test</b>	<b>Heavy naphtha</b>	<b>Coker fraction</b>
	<b>b.r. 77-165 °C</b>	<b>b.r. 100-160 °C</b>
<b>Hydrocarbon-type(FIA)</b>		
<b>Analysis, vol. %</b>		
Saturates	88.6	40.5
Olefins	0.0	47.3
Aromatics	11.4	12.2
<b>Total</b>	<b>100</b>	<b>100</b>
<b>Saturates group analysis,</b>		
<b>vol. % Branched naphthenes</b>		
Cyclohexane	1.7	Trace
n-Paraffins	40.6	15.7
Isoparaffins	16.2	13.8
<b>Total</b>	<b>88.6</b>	<b>40.5</b>



**Table 4: Effect of Varying Total Alkylating Agents to Olefins Wt. Ratio on the Alkylation of Heavy Naphtha with Coker Gasoline Fraction Using Sulphuric Acid as Catalyst**

Exp. No.	1	2	3	4
(Coker Fraction + Heavy Naphtha), gm.	73.3	73.5	73.7	74.4
Alkylation feed : Olefins, gm.	8.9	11.6	16.1	26.1
Alkylating agents, gm. (Aromatics, Isoparaffins, Cycloparaffins)	36.0	35.9	35.4	26.1
Alkylating agents/Olefin, Wt. Ratio	4/1	3/1	2/1	1/1
<b>Total Alkylate:</b>				
Specific gravity, 20/4	0.7213	0.7260	0.7330	0.7454
wt., gm	62.8	61.7	60.1	59.6
wt.% of charge	85.7	84.0	81.5	80.0
<b>Cut I, b.r. &lt; 165 °C:</b>				
Boiling range, °C	73-165	79-165	88-165	79-165
Specific gravity, 20/4	0.7094	0.7159	0.7175	0.7238
wt., gm	56.5	54.3	50.0	44.5
wt.% of charge	77.1	74.0	67.8	59.8
Olefins or Aromatics, vol. %	0.0	0.0	0.0	0.0
<b>Cut II, b.r. &gt;165 °C:</b>				
Specific gravity, 20/4	0.8499	0.8469	0.8363	0.8321
wt., gm	6.2	7.8	10.2	15.4
wt.% of charge	8.5	10.6	13.8	20.7
Olefin content wt.% of charge	0.9	0.5	0.3	2.3
Theoretical yield, gm.	17.8	23.2	32.2	52.2
Ratio of Cut II to theoretical yield	0.35	0.34	0.32	0.29

[Reaction temperature = 0 °C ; Coker gasoline space velocity = 0.35 vol./hr./vol. ;Catalyst to feed wt. Ratio = 3/1]

**Table 5: Self Alkylation of Coker Gasoline Fraction Using Sulphuric Acid as Catalyst**

<b>Total Alkylate:</b>	
Specific gravity, 20/4	0.7541
wt., gm	57.3
wt.% of charge	76.3
<b>Cut I, b.r. &lt;165 °C:</b>	
Boiling range, °C	100-165
Specific gravity, 20/4	0.7220
wt., gm	40.9
wt.% of charge	54.5
Olefins or Aromatics, vol. %	0.0
<b>Cut II, b.r. &gt; 165 °C:</b>	
Specific gravity, 20/4	0.8454
wt., gm	16.2
wt.% of charge	21.6
Olefin content, wt. % of charge	3.9
Theoretical yield, gm.	49.6
Ratio of Cut II to theoretical yield	0.32

Reaction temperature = 0 °C ; Coker gasoline space velocity = 0.35 vol./hr./vol.  
;Catalyst to feed wt. Ratio = 3/1; (Coker fraction + Heavy naphtha) gm. =75.1  
Alkylation feed, olefin, gm. =35.6 ; Alkylating agents, gm. = 24.8; Alkylating agents  
to olefins wt ratio = 0.6/1

**Table 6: Alkylation of Heavy Naphtha of b.r. 77-165 °C with Coker Fraction of b.r. 100-160 °C Using Sulphuric Acid Catalyst, Specifications of Products of Fractionation of Cut II Obtained from Exp, No. 4**

Product	Cut IIa	Cut IIb
Boiling range °C	170-280	282-389
Specific gravity, 20/4	0.7993	0.8840
Refractive index, $n_D^{20}$	1.4420	1.4872
wt% of charge	5.8	14.9
ASTM Distillation, °C		
I.b.p.	170	282
10%	173	290
20%	178	300
30%	193	315
40%	221	322
50%	235	335
60%	242	340
70%	249	359
80%	259	370
90%	275	384
F.b.p	280	
Flash point by Pensky Martens (closed tester) °C	-	38
Aniline point, °C	68	55
Pour point, °C	-	- 48
Smoke point, Mm	31	-
Kinematic viscosity at 20 °C (sec)	3.0	20.9
Sulphur content, wt.%	-	0.25
Colour	0	1.0
Diesel index	-	36.9
Carbon residue, wt %	-	0.026
Ash content, wt.%	-	nil
Cetane index	-	56

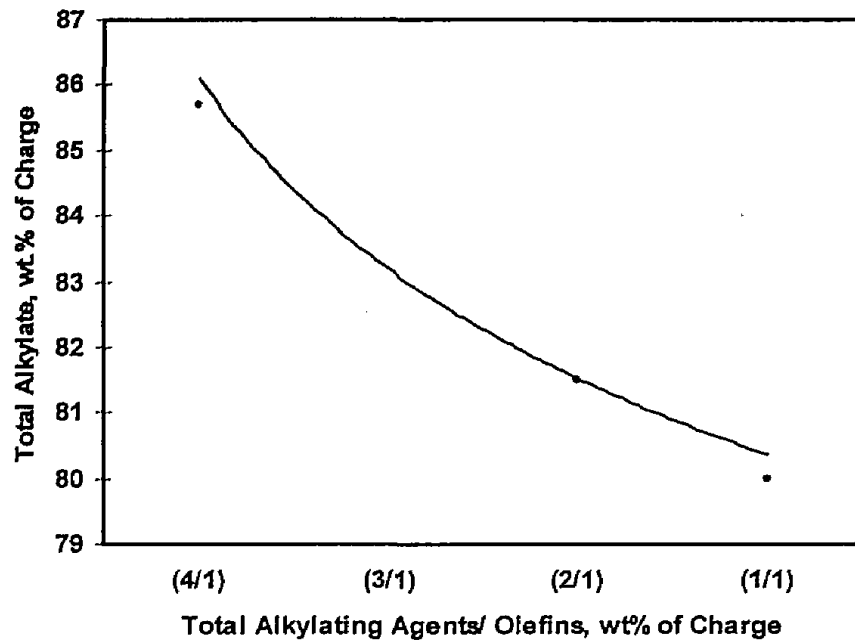


Fig. 1: effect of total alkylating agents / olefins ratio on the yield of total alkylate

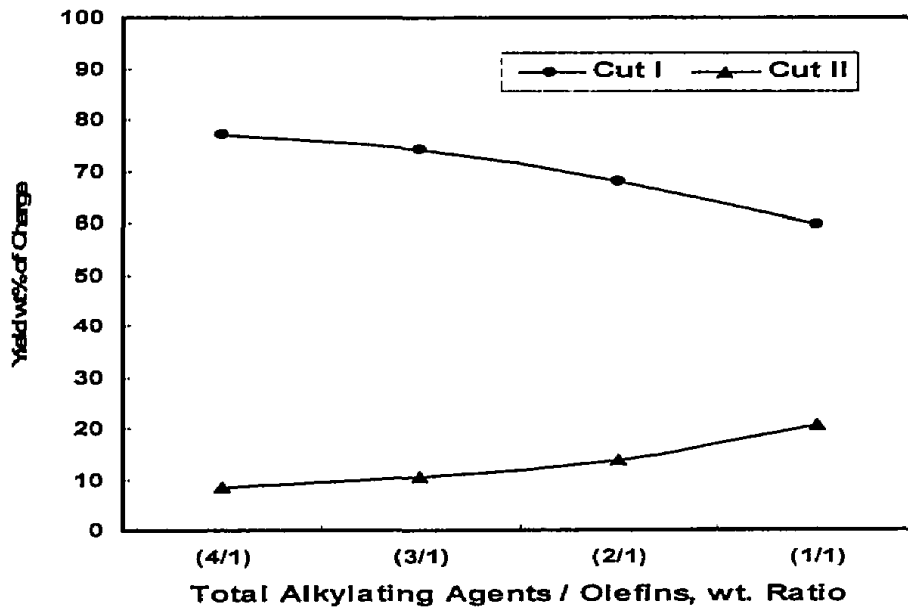


Fig. 2: Effect of total alkylating agents / olefins wt. ratios on the yield of cut I and cut II

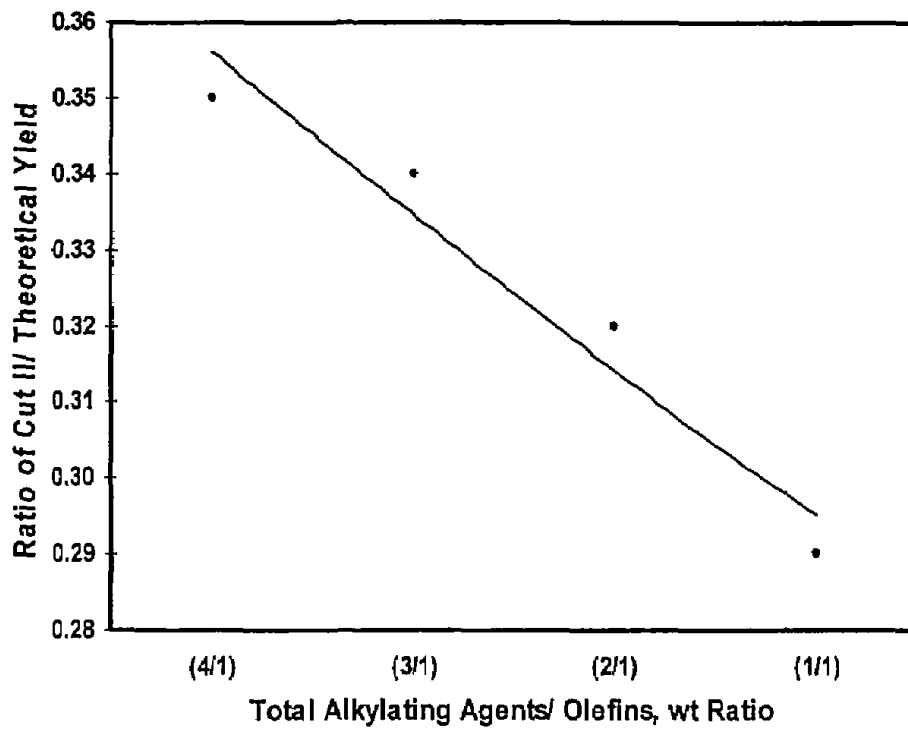


Fig. 3: Effect of total alkylating agents / olefins wt. ratios on the ratio of cut II / theoretical yield cut II

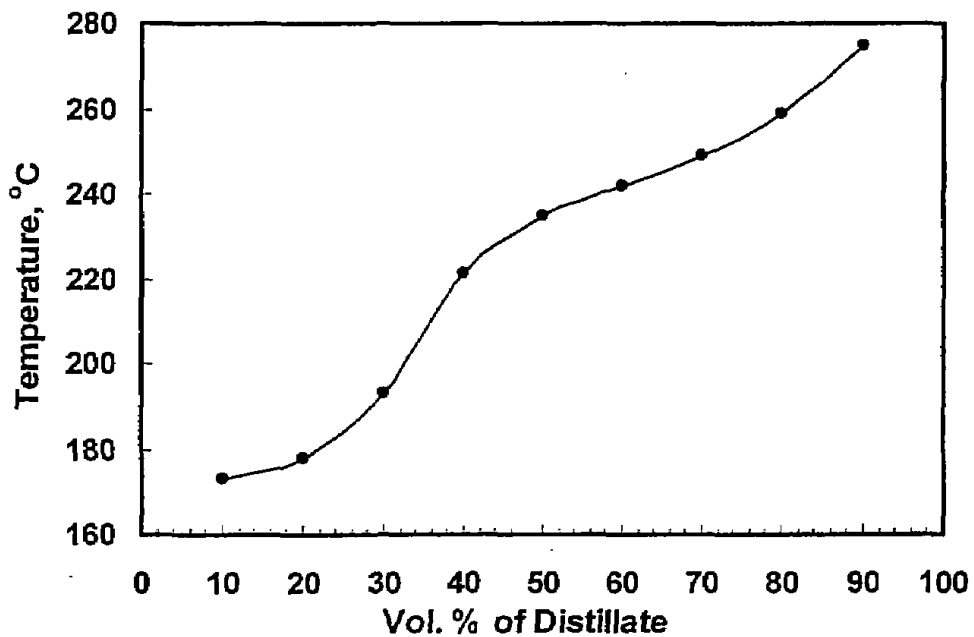


Fig. 4: Distillation curve of cut IIa (Exp. No. 4)

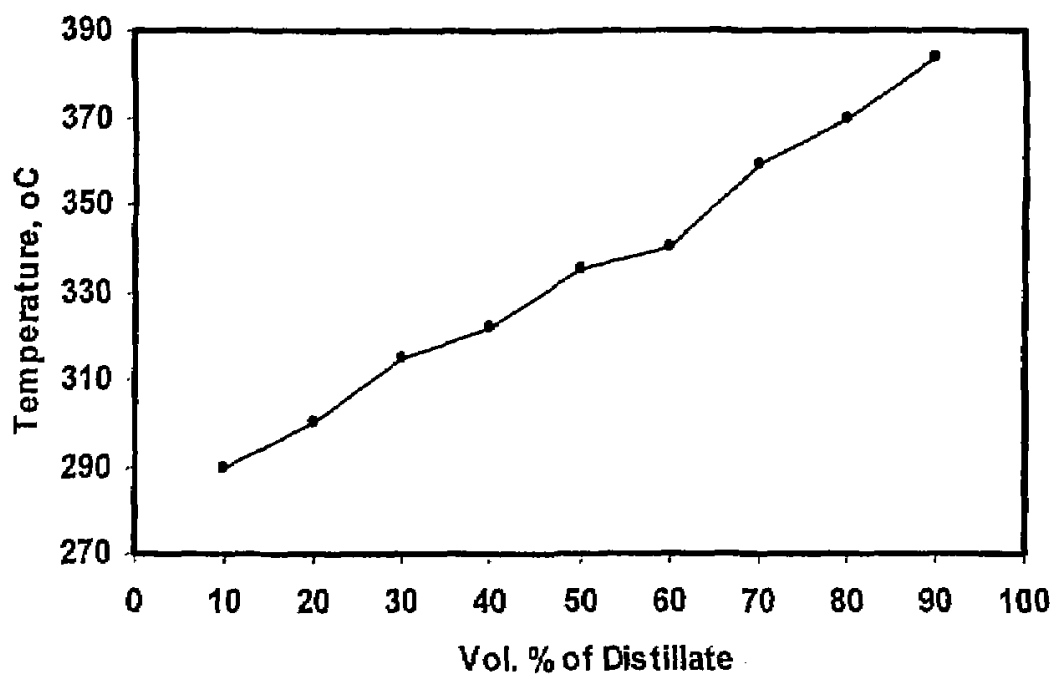


Fig. 5: Distillation curve of cut IIb (Exp. No. 4)