

## Quaternary Ammonium Salts Derived from Jojoba Oil

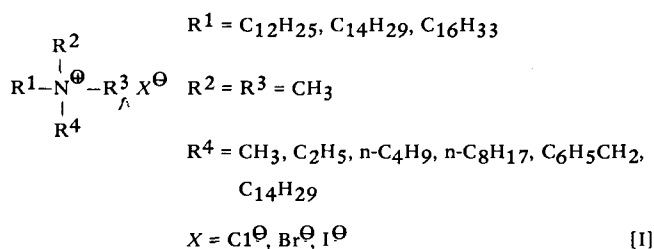
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### ABSTRACT

Quaternary ammonium salts derived from jojoba oil have been prepared either from jojobyl halides and tertiary amines or from dimethyljojobyl- and dimethylhomojojobylamines and alkyl halides. These salts have special potential as germicides, surfactants, emulsifiers and phase transfer reagents, for example. The jojobyl iodide and jojobyl mesylate react more readily with amines than the chloride, which is consistent with the expected relative leaving group reactivity of the different halides. The nucleophilicity of the tertiary amine, containing one long chain derived from jojoba oil, is similar to that of other tertiary amines containing only short alkyl chains. The salts are thermally stable up to 100-110 C.

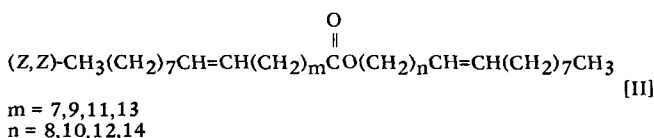
### INTRODUCTION

Tetraalkyl and aryl ammonium salts such as I, are known to be physiologically active and chemically reactive and hence are widely used in the chemical industry, in organic synthesis, in medicine and in product processing (1-6).



At least one R group of the salts is a long chain alkyl group. Most of these salts in present use possess saturated C<sub>12</sub>-C<sub>16</sub> chains; it is therefore of interest to prepare and test compounds containing both unsaturated and longer chains. Monounsaturated long chain alkyl groups ranging from C<sub>18</sub> to C<sub>24</sub> atoms, but composed mainly of C<sub>20</sub> and C<sub>22</sub> atoms, can be obtained from jojoba oil — a liquid wax which is extracted from the seeds of the desert bush *Simmondsia chinensis* (Link) Schneider. Jojoba oil is a mixture of esters having the general structure II and composition as given in Table I. The position of the double bond is mainly at the ω-C<sub>9</sub> on both ends, but there are several positional isomers in which the double bonds are located at other carbons along the chain (7).

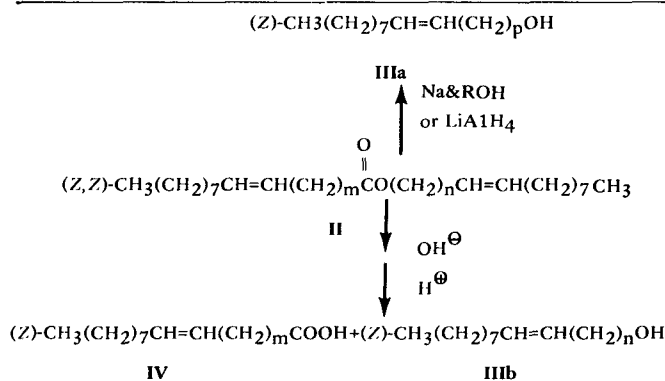
Several factors have contributed to the increasing interest and rapid development in jojoba oil production (8-14).



The major chemical interest in the oil has been in determining the exact chemical composition and in studying chemical transformations such as reduction of the ester function to alcohols by Na/ROH, hydrogenation, isomerization, epoxidation, sulfurization, sulfochlorination, polymerization, saponification and transesterification (14). In most of these reactions, the oil is used in the form of its 36-44 carbon chain unit or transformed to another ester. The Na/ROH reduction of the wax results in mixed long chain

monounsaturated alcohols; the hydrolysis of the ester yields the two components of the wax (10) as shown in Scheme 1. These mixtures can be used as a new source for

### SCHEME 1



### Percentage of Products from Hydrolysis and Reduction (15)

	C <sub>18</sub>	C <sub>20</sub>	C <sub>22</sub>	C <sub>24</sub>	Total
Hydrolysis					
(m) =	(7) 10-11	(9) 70-71	(11) 13-15	(13) 2-3	95-
(n) =	(8) 1-2	(10) 45-46	(12) 42-43	(14) 7-8	95-
Reduction					
(p) =	(8) 5-6	(10) 57-58	(12) 27-29	(14) 5-6	94-

long chain monounsaturated alcohols, acids and their derivatives, in 3 different components. The minimal percentage of the 4 main components of ester II is 94-95% of the mixture and thus is homogenous in chemical and physical properties. The few percentages left include other hydrocarbons, esters, free acids and alcohols, and sterols (each less than 1%) (10).

In this study, we investigated different syntheses of jojobyl (jojobyl or jobj- stands for the mixture of the long chains derived from ester II) halides for their reactivity toward tertiary amines in the preparation of quaternary ammonium salts. We also investigated a procedure for adding one more carbon atom to the jojobyl chain to yield a homojojobyl chain (homojojobyl is the next homolog aliphatic chain in the series. Thus, if p in IIIa is mainly 10 and 12 for jojobyl, then homojojobyl is p + 1 as in XII [Scheme 3] and derivatives). Preparation of jojobylamine and homojojobylamine as precursors to the quaternary ammonium salts was also investigated. These reactions supply information on the nucleophilicity of the amine moiety in long chain compounds.

### EXPERIMENTAL PROCEDURES

#### General

Crude jojoba oil was purified by mixing it with bleaching earth at 60 C for 30 min and then filtering under vacuum.

TABLE I  
Main Components of Jojoba Oil<sup>a</sup>

Wax esters	%	Alcohol portion		Acid portion	
		No. carbon atoms:no. double bonds	%	No. carbon atoms:no. double bonds	%
C <sub>36</sub>	1.61	18:1	1.1	18:1	11.2
C <sub>38</sub>	6.23	20:1	43.8	20:1	71.3
C <sub>40</sub>	30.56	22:1	44.9	22:1	13.6
C <sub>42</sub>	49.50	24:1	8.9	24:1	1.3
C <sub>44</sub>	8.12				

<sup>a</sup>An average of several samples from different regions (10).

The crude product after each chemical transformation was used in the next step without further purification. Next, we poured the reaction mixture into H<sub>2</sub>O, extracted with petroleum ether (60-80), washed with saturated NaCl solution and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Infrared (IR) and nuclear magnetic resonance (NMR) spectra provided monitors for the chemical change occurring in each reaction. Purity was determined by NMR (16). All NMR spectra gave the following: terminal CH<sub>3</sub> as triplet at  $\delta$  0.92-0.94; an intense signal at 1.2-1.4 for all aliphatic hydrogens; a signal at 1.98-2.05 for allylic hydrogens; and a quintet at 5.20 (J=5 cps) for the olefinic hydrogens. Other signals are summarized in Table II. Integration curves were consistent for the assignment of the different hydrogens. The NMR spectra were determined on a Varian XL-100 in CCl<sub>4</sub> or CDCl<sub>3</sub> solution. The IR spectra were determined with a Perkin Elmer Model 377 spectrometer. The samples were taken undiluted or in CHCl<sub>3</sub> solution.

Analytical thin layer chromatography (TLC) plates were prepared with Silica Gel KGS-254. Microanalyses were performed in the Microanalytical Laboratory of the Applied Research Institute, Ben-Gurion University (BGU), Israel.

The percentage of heteroatoms was determined only in those products which were relatively pure or which were obtained from reactions that were essentially quantitative. The average molecular weight (MW) of jojoba oil, based on the composition of the various components, was calculated to be 590. The MW of the jojobyl residue, obtained from the reduction of ester II, was calculated to be 299.

#### Reaction Conditions

Several of the ammonium salts were prepared in a closed stainless steel reactor (lined with a Teflon sleeve) and heated in a hot oil bath at the temperature specified in

Tables III and IV. The autogenous pressure (A.P.) inside the reactor was estimated to be 2-3 atm (17). All other reactions were run at atmospheric pressure with or without a solvent.

All the reactions were followed by NMR using the -CH<sub>2</sub>-Y group signal to identify the components of the mixture and to calculate its exact composition, i.e.,  $\pm$  2% of the absolute amount (16) (see Table II). This procedure was fast, easy, reliable, reproducible and sufficiently sensitive for practical and even semiquantitative work. In general, the reaction was continued until complete transformation occurred, as determined by NMR.

#### Solvents

Petroleum ether (60-80) was dried over CaCl<sub>2</sub> and distilled. Ether was dried over CaCl<sub>2</sub>, then over Na and distilled. Pyridine was kept over KOH prior to its use. Acetone was dried over CaCl<sub>2</sub>. Tetrahydrofuran (THF) was kept over KOH, passed through basic alumina and dried over CaH<sub>2</sub>. Benzene was dried over Na. Dimethyl sulfoxide (DMSO) and dimethyl formamide (DMF) were used without drying.

Known procedures were used to prepare the following compounds and references are given in each case. Some adjustments were made to fit the procedures to the jojoba derivative.

*Jojobyl chloride* (V): prepared from IIIa, SOCl<sub>2</sub> and pyridine (18), 87% yield,  $n_D^{27}$  1.4610. Calcd for Cl, 11.2%; found, 11.3%.

*Jojobyl mesylate* (VI) (16): calcd for S, 8.46%; found, 8.27%.

*Jojobyl iodide* (VII) (16): calcd for I, 31.05%; found 30.1%.

*Jojobyl phthalimide* (VIII): prepared from VII and potassium phthalimide (19), 86% yield.

TABLE II

Chemical Shifts of  $\alpha$ -Methylene ( $t$ ; J = 6 cps) in Different Long Chain Derivatives  
(Z)-CH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>CH = CH(CH<sub>2</sub>)<sub>x</sub>CH<sub>2</sub>Y

Y	OMes				OH	Cl	Br	I	NH <sub>2</sub>			CN		N(CH <sub>3</sub> ) <sub>2</sub>
$\delta^a$	4.10	3.95	3.61	3.40-3.65 <sup>b</sup>	3.45	3.38	3.26	3.08	2.61	2.26	2.24	2.24	2.20	2.13 <sup>c</sup>

<sup>a</sup> $\delta$ , ppm downfield from TMS as internal standard in CC1<sub>4</sub> solution.

<sup>b</sup>Multiplet from the quadropole effect of nitrogen.

<sup>c</sup>Partially covered by the singlet of the two methyl groups.

*Jojobylamine (IX)*: prepared by hydrolysis of VIII with hydrazine hydrate (20), 74% yield,  $n_D^{27}$  1.4601. Calcd for N, 4.48%; found 4.4%.

*Dimethyljojobylamine (X)*: prepared from IX and HCOOH and formaldehyde (21), 95% yield,  $n_D^{23}$  1.4598. Calcd for N, 4.11%; found, 3.81%.

*Homojojobyl nitrile (XI)*: prepared according to Shani et al. (22).

*Homojojobylamine (XII)*: prepared by reduction of XI with  $LiAlH_4$  to yield 80 g of XII (93%),  $n_D^{26.5}$  1.4601. No amide absorption in the IR spectrum was observed.

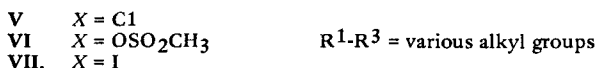
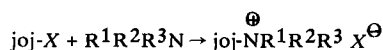
*Dimethylhomojojobylamine (XIII)* (tertiary amine): prepared by the same procedure as used for dimethyljojobylamine (X), with the same yield and purity.

*Trialkyljojobyl ammonium salts (XIV)*: a solution of jojobyl halide (V, VII) or mesylate (VI) and the appropriate trialkylamine was treated as summarized in Table III and Scheme 2.

*Trialkyljojobyl and homojojobyl ammonium salts (XV)*: a solution of X or XIII and the appropriate halide was treated as summarized in Table VI and Scheme 4.

## RESULTS AND DISCUSSION

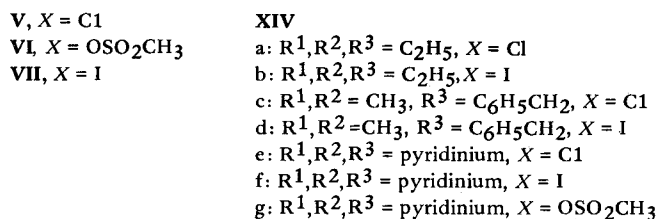
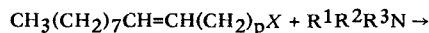
Two possible synthetic approaches to quaternary ammonium salts derived from jojoba oil are obvious. The first of these is the reaction of a jojobyl halide (V, VII) or mesylate (VI), with a tertiary amine:



The jojobyl halides have been prepared from the alcoholic mixtures derived from the wax in different compositions. Jojobyl chloride (V) was prepared from alcohol IIIa and  $SOCl_2$ . The intermediate dijojobyl sulfite could be isolated when the molar ratio of alcohol to  $SOCl_2$  was 2:1. On further reaction of this intermediate with  $SOCl_2$ , the chloride was obtained. The mesylate (VI) was best prepared using  $(C_2H_5)_3N$  as a base. (When pyridine was used as a base, jojobyl pyridinium mesylate contaminated the product [16]). The iodide (VII) was readily prepared from the mesylate with NaI in acetone (see Scheme 3).

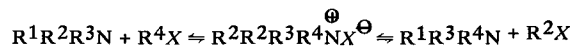
Four amines have been reacted with jojobyl chloride, iodide and mesylate:  $(C_2H_5)_3N$  (TEA),  $joj-N(CH_3)_2$  (DMJA),  $C_6H_5CH_2N(CH_3)_2$  (DMBA) and  $C_5H_5N$ .

SCHEME 2

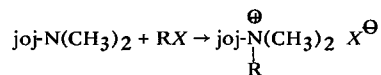


Pyridine, is expected to be the weakest nucleophile, whereas the other three should be stronger nucleophiles

with the same level of reactivity. Triethylamine might show somewhat lower reactivity because of steric hindrance. The results of reactions of jojobyl chloride (V) and jojobyl iodide (VII) with the 4 amines are illustrated in Figure 1, where we compare the yields of the quaternary ammonium salt vs time. All reactions were conducted below decomposition temp (see later) we can thus eliminate the possible equilibrium of *trans*-alkylation, such as

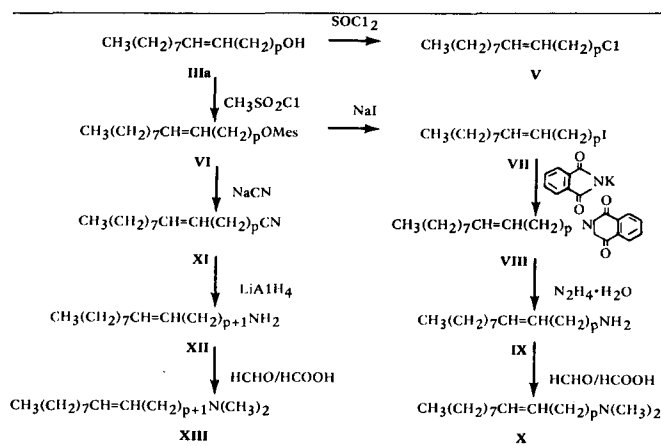


The second approach to the preparation of the desired quaternary salts was the reaction of dimethyljojobylamine (DMJA) and dimethylhomojojobylamine (DMHJA) with alkyl halides:



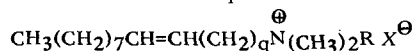
Jojobylamine (IX) could be obtained via the phthalimide VIII. Further methylation of IX with HCHO/HCOOH produced DMJA (X). DMHJA (XIII),

SCHEME 3

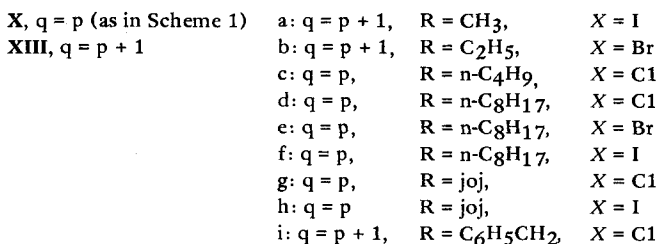


could be prepared from the mesylate VI via the nitrile XI, which then reduced to homojojobylamine (XII) and was methylated with HCHO/HCOOH to yield XIII (See Scheme 3).

SCHEME 4



XV



Nine halides and a mesylate were reacted, resulting in 4 chlorides ( $C_4H_9Cl$ ,  $C_8H_{17}Cl$ ,  $joj-Cl$  and  $C_6H_5CH_2Cl$ ), 2 bromides ( $C_2H_5Br$  and  $C_8H_{17}Br$ ) and 3 iodides ( $CH_3I$ ,

TABLE III  
Preparation of Trialkyljojobyl Ammonium Salts (XIV) from Jojobyl Halide and Trialkylamine

Joj-X reacted (g, mol)	Trialkylamine reacted (g, mol)	Reaction conditions				Yield of salt (%)	Liquid temp (C)	Microanalysis	
		Solvent (ml)	Temp (C)	Pressure	Time (hr)			Calc. for (%)	Found (%)
V (3.17, 0.01)	(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub> N (1.01, 0.01)	MeOH(30)	95 - 105	A.P. <sup>a</sup>	72	42(XIVa)	150 - 5	N = 2.74	n = 2.62
VII (2.03, 0.005)	(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub> N (0.5, 0.005)	EtOH(10)	75	reg. <sup>b</sup>	36	quant (XIVb)		i = 24.9	i = 26.4
V (3.17, 0.01)	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> N(CH <sub>3</sub> ) <sub>2</sub> (1.35, 0.01)	MeOH(10)	100 - 110	A.P.	72	48(XIVc)			
VIII (4.05, 0.01)	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> N(CH <sub>3</sub> ) <sub>2</sub> (1.35, 0.01)	EtOH(5)	75	reg.	6	94(XIVd)			
V (3.17, 0.01)	C <sub>5</sub> H <sub>5</sub> N (0.8, 0.01)	MeOH(25)	95 - 110	A.P.	12	30(XIVe)			
VII (4.05, 0.01)	C <sub>5</sub> H <sub>5</sub> N (2.0, 0.025)	Pyridine(2)	95 - 100	reg.	1.5	quant(XIVf)	200 - 5	N = 2.87	N = 2.9
VII (2.03, 0.005)	C <sub>5</sub> H <sub>5</sub> N (0.4, 0.005)	EtOH(5)	75	reg.	48	quant(XIVf)		I = 26.02	I = 26.0
VI (7.6, 0.02)	C <sub>5</sub> H <sub>5</sub> N (1.6, 0.02)	EtOH(30)	75	reg.	1	quant(XIVg)	170 - 5	N = 3.06	N = 2.75
								S = 7.00	S = 6.6

<sup>a</sup>Reactions conducted in stainless steel reactor under A.P. (2-3 atm).

<sup>b</sup>Reactions conducted under regular conditions, e.g., atmospheric pressure.

TABLE IV

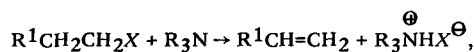
Preparation of Trialkyljojobyl and Trialkylhomojojobyl Ammonium Salts (XV) from Dimethyljojobyl and Dimethylhomojojobylamine and Alkyl Halide

Trialkylamine reacted (g, mol)	Alkyl halide reacted (g, mol)	Reaction conditions				Yield of salt (%)	Liquid. temp	Microanalysis	
		Solvent (ml)	Temp (C)	Pressure	Time (hr)			Calc. for (%)	Found (%)
XIII (3.75, 0.011)	CH <sub>3</sub> I(2.2, 0.011)	--	30	reg. <sup>a</sup>	1	quant (XVa)	225 - 30	N = 2.90	N = 2.85
XIII (6.84, 0.02)	C <sub>2</sub> H <sub>5</sub> Br(2.7, 0.025)	EtOH(10)	0 - 5	reg.	2	quant (XVb)	165 - 70	I = 26.32	I = 24.1
X (1.7, 0.005)	n-C <sub>4</sub> H <sub>9</sub> Cl(0.55, 0.006)	MeOH(20)	100 - 5	A.P. <sup>b</sup>	72	65 (XVc)		N = 3.11	N = 2.87
X (3.45, 0.01)	n-C <sub>8</sub> H <sub>17</sub> Cl(1.5, 0.01)	MeOH(35)	100 - 5	A.P.	72	70 (XVd)		Br = 17.8	Br = 16.7
X (0.34, 0.001)	n-C <sub>8</sub> H <sub>17</sub> Br(0.19, 0.001)	EtOH(5)	75	reg.	48	quant(XVe)			
X (0.34, 0.001)	n-C <sub>8</sub> H <sub>17</sub> I(0.24, 0.001)	EtOH(5)	75	reg.	6	98 (XVf)			
X (0.85, 0.0025)	joj-Cl(0.79, 0.0025)	MeOH(10)	100 - 5	A.P.	72	45 (XVg)			
X (0.85, 0.0025)	joj-I(1.02, 0.0025)	EtOH(5)	75	reg.	24	98 (XVh)	45 - 50	N = 1.90	N = 1.87
X (2.67, 0.008)	joj-I(3.2, 0.008)	MeOH(25)	100 - 5	A.P.	24	quant (XVh)		I = 17.28	I = 16.8
XIII (6.84, 0.02)	C <sub>2</sub> H <sub>5</sub> CH <sub>2</sub> Cl(2.53, 0.02)	--	78 - 80	reg.	immed.	97 (XVi)	145 - 159	N = 3.00	N = 2.98
								C1 = 7.61	C1 = 7.3

<sup>a</sup>Reactions conducted under regular conditions, e.g., atmospheric pressure.

<sup>b</sup>Reactions conducted in stainless steel reactor at A.P. (2-3 atm).

$C_8H_{17}I$  and  $joj-I$ ). Chlorides are expected to be less reactive than bromides, which in turn are less reactive than iodides, toward tertiary amines in the  $S_N2$  reaction; this characteristic is based on (a) bond energies of  $C-X$ , which are 81, 68 and 51 Kcal/mol for  $X = Cl, Br$  and  $I$ , respectively, and (b) acid strength of  $HX$  ( $HCl < HBr < HI$ ). The results of the reactions of all halides with DMJA, as a representative tertiary amine, are illustrated in Figure 2. Again, all reactions were conducted below decomposition temp. Since the halides are all primary we can ignore elimination, another competitive reaction, e.g.,



which is typical to tertiary halides. Thus, we can evaluate the nucleophilicity of the amines toward the halides and the reactivity of the halides as leaving groups, based on the chemical yields. The reaction conditions in the kinetic measurements are similar: either reflux in ethanol at 75 C or in methanol under Autogenous pressure (A.P.).

The main features are:

- The most reactive nucleophile is DMBA, whereas DMJA is more reactive than TEA, as expected with steric hindrance. Pyridine is the least reactive. This trend continues in 3 sets of reactions: with jobobyl iodide (yields with 4 amines after 12 hr: DMBA 95%, DMJA 90%, TEA 73% and pyridine 72%); with jobobyl chloride under regular conditions after 48 hr (DMBA 29%, DMJA 14%, TEA and pyridine no reaction); with jobobyl chloride under A.P. after 72 hr (DMBA = 48%, DMJA = 45%, TEA = 42% and pyridine = 30%).
- The reactivity of jobobyl iodide is much higher than that of the chloride. In fact, for the less nucleophilic amines (TEA and pyridine), forceful conditions are needed for jobobyl chloride; the reactions were therefore conducted in a closed stainless steel reactor under A.P.
- The expected order of reactivity  $RI > RBr > RCl$  is maintained in general, when R is roughly the same in nature and length of chain.
- The chain length effect is pronounced such that bromide of shorter chain is more reactive than iodide of longer chain ( $C_2H_5Br = 100\%$  after 2 hr and  $C_8H_{17}I = 98\%$  after 6 hr with DMJA). This is limited to a certain length (jobobyl iodide = 90% and  $C_8H_{17}Br = 63\%$ ) after 6 hr with DMJA.
- The length of chain has its effect in the same family, i.e., chlorides or iodides ( $C_4H_9Cl = 65\%$ ,  $C_8H_{17}Cl = 70\%$  and jobobyl chloride = 45% after 72 hr under A.P. with DMJA;  $CH_3I = 100\%$  after 1 hr, whereas  $C_8H_{17}I = 98\%$  and jobobyl iodide = 90% after 6 hr with DMJA).
- The mesylate (VI) seems to be as reactive as the iodide (VII) as observed in its reaction with pyridine, the nucleophilic amine least studied.

Based on these results one can assume that long chain alkyl halides and tertiary amines with one long chain behave, in principle, like alkyl derivatives with short chains, although the reactions proceed somewhat more slowly.

The physical properties of the quaternary ammonium salts reflect that these are mixtures of several compounds. Thus, all are semisolids which do not melt but gradually liquify over a wide range of temperatures. The long chain nature of at least one alkyl group (jobobyl or homojobobyl) causes the salts to be more hydrophobic and lipophilic; thus, they are partially soluble in apolar solvents such as petrol ether, ether and benzene, but are completely soluble in  $CHCl_3$  and form stable emulsions in water.

The effect of temperature is summarized in Table V. Above 100-110 C, decomposition occurs so all reactions

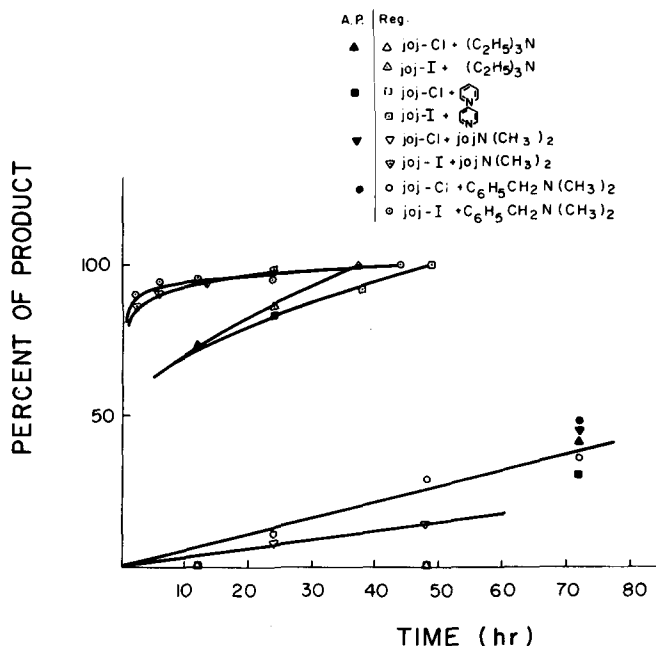


FIG. 1. Reactions of jobobyl chloride and jobobyl iodide with tertiary amines.

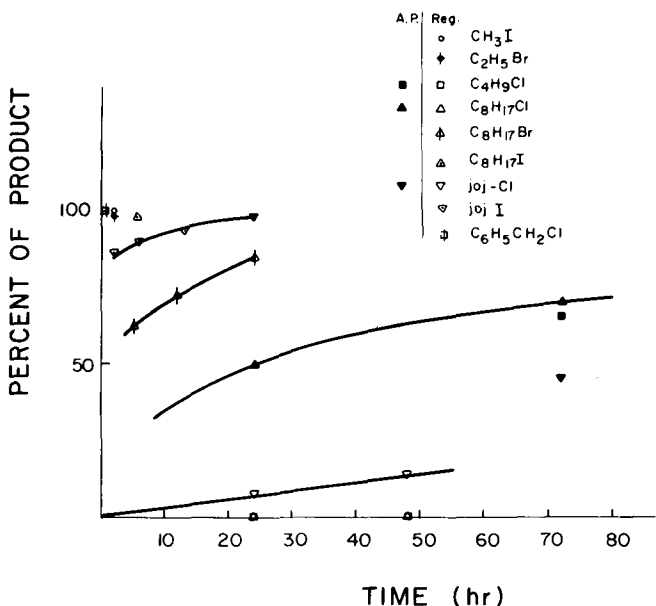


FIG. 2. Reactions of alkyl halides with dimethyljobobylamine.

TABLE V

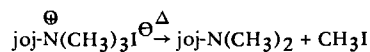
Thermal Decomposition of  $joj-N(CH_3)_3I^\oplus$  in Aromatic Solvents

Time (hr)	Percentage of decomposition <sup>a</sup>		
	benzene (80 C)	toluene (115 C)	xylene (140 C)
1	0	5	5
2	0	5	5
4	0	5	10
6	0	10	16
12	0	14	16

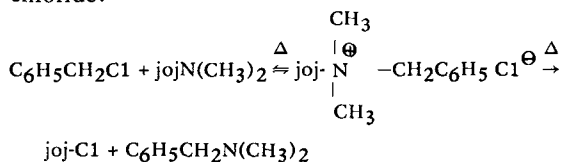
<sup>a</sup>Percentage of decomposition was determined by NMR of a sample, measuring the amount of salt left in solution.

were conducted below this temperature range.

The thermal instability of the quaternary ammonium salts is illustrated by decomposition to alkyl halide and trialkylamine. When trimethyljojobyl ammonium iodide is heated, no jojobyl iodide was isolated except for DMJA. For this reason, the volatility of  $\text{CH}_3\text{I}$  might shift the decomposition process or the easier  $\text{S}_{\text{N}}2$  displacement:



When DMBA is incorporated into the salt it is probably the better leaving group upon heating (isolated as  $\text{HCl}$  salt), because of its relative stability compared to benzyl chloride:



It could be that an early and fast decomposition to DMJA and benzyl chloride results in an equilibrium, which is then shifted toward the more stable decomposition product,

since breaking the  $\text{N}^{\oplus}-\text{CH}_2\text{C}_6\text{H}_5$  bond is the easier reaction (resonance stabilization).

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