## REACTIONS OF CYCLIC AMMONIUM CATIONS VII.\* QUINOLINATION OF 1-ALKYL-2,3-DIHYDROINDOLES AND 1-ALKYL-1,2,3,4-TETRAHYDROQUINOLINES

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A method for the introduction of a quinoline residue into an aromatic ring by means of 1-acylquinolinium salts was developed. The effect of the anion was examined. A number of quinolylindoles and quinolyltetrahydroquinolines were synthesized by this method. It is shown that electron-acceptor or electron-donor substituents in the benzene ring of the quinolinium salt have practically no effect on the course of the process.

A method for the introduction of a quinoline residue into an activated benzene ring by means of 1acylquinolinium salts, obtained by the reaction of quinoline with acyl halides, was described in [1]. In developing this method we found that different (electron-donor or electron-acceptor) substituents in the benzene ring of the quinolinium salt have virtually no effect on the reaction with dimethylaniline. In all cases, the corresponding 1-acyl-2-(p-dimethylaminophenyl)-1,2-dihydroquinolines (I) are formed in good yields (see Table 1). This is particularly valid for the 8-methylquinolinium salt, where the  $CH_3$  group can have a substantial effect on the coplanarity of the acyl group and, consequently, can disrupt the entire conjugation system.

 $R \bigoplus_{\substack{N' \\ R' - C = 0}} \chi \Theta + \bigotimes_{\substack{N' \\ R' - C = 0}} R \bigoplus_{\substack{N' \\ R'$ 

As for substitution in the pyridine portion of the molecule, our preliminary observations [1] regarding the possibility of introducing quinaldinium or lepidinium salts into this reaction were not confirmed. The presence of CH<sub>3</sub> groups in both the  $\alpha$  and  $\gamma$  positions turns out to be sufficient to block the reaction.

It is interesting to note that the nature of the anions of the N-acylquinolinium salts has a substantial effect on the yields of I. Thus, in the reaction of quinoline and benzoyl fluoride, benzoyl chloride, benzoyl bromide, and benzoyl iodide with dimethylaniline under standard conditions (100°, 8 h), the yields of 1-benzoyl-2-(p-dimethylaminophenyl)-1,2-dihydroquinoline increased regularly with increasing nucleophilicity of the acylquinolinium salt anion (24, 45, 90, and 95%, respectively). The observed regularity should apparently be associated with the increase in the reactivity of the benzoyl halides in this series in connection with the fact that the halogen-carbon bond strength decreases appreciably in this direction [2]. Such regularity in the change in the reactivity in the acyl halide series during the acylation of amines was also observed by Litvinenko and co-workers [3], who associated the observed phenomenon with the change in the halogen-carbon bond strength. In addition, the results of our experiments with the various benzoyl halides make it possible to assume the participation in the reactions of a charge-transfer complex, formed due to transfer of a valence electron of the anion to the lower vacant molecular orbital of the quinolinium cation,

\*See [1] for Communication VI.

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			9	Y			ш	ound, 9			Calc., 9		rield,
M	<u>ک</u>	Mp, -C	2	uu uu	8	Luputcat iormula	υ	Н	z	υ	н	z	%
6-CH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>	167—168 <b>a</b>	0,45	250	4,36	C25H24N2O	81,62 81,71	6,85 6,84	7,86 7,87	81,49	6,57	7,60	34
6-Br	C <sub>6</sub> H <sub>5</sub>	122—123 b	0,35	1	1	$C_{24}H_{21}BrN_2O$	66,84 66,70	5,06 4,92	1	66,52	4,88	1	37
6-NO <sub>2</sub>	C <sub>6</sub> H <sub>5</sub>	180—181 <b>a</b>	0,63	260 340	4,37 3,94	C <sub>24</sub> H <sub>21</sub> N <sub>3</sub> O <sub>3</sub>	72,29 72,41	5,64 5,43	10,86 10,63	72,17	5,30	10,52	50
6-(CH <sub>3</sub> ) <sub>2</sub> Ņ	C <sub>6</sub> H <sub>5</sub>	169—170 b	0,17	1	I	$C_{26}H_{27}N_{3}O$	78,69 78,45	6,71 6,63	10,85 10,72	78,56	6,85	10,57	35
8-CH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>	175—176c (acetone)	0,50	250	4,44	$C_{25}H_{22}N_2O$	81,45 81,68	6,52 6,33	7,80 7,92	81,49	6,57	7,60	38
Н	2-Fury1	155—156 b	0,43	257	4,50	$C_{22}H_{20}N_2O_2$	76,43 76,56	5,93 5,92	8,43 8,20	76,72	5,85	8,13	29
Н	CH <sub>3</sub>	122—1 <b>23 d</b>	0,52	260	4,44	C <sub>19</sub> H <sub>20</sub> N <sub>2</sub> O	77,80 77,69	6,81 6,70	9,28 9,42	78,05	6,90	9,58	41
Н	N(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> .	195—196 b	0,64	I	1	$C_{30}H_{27}N_{3}O$	81,17 81,04	6,32 6,18	9,49 9,68	80,87	6,11	9,43	15
7-CH <sub>3</sub>	C <sub>6</sub> H <sub>6</sub>	137—138 c	0,32	240	4,42	C25H24N2O	81,35 81,22	6,48 6,34	7,78 7,52	81,49	6,57	7,60	42
											<b>-</b>		
a) From dime	ethylformamide	. b) From €	thano	l. c)	Fron	n acetone. d) Fro	E E	ethanc					

TABLE 1. 1-Acyl-2(p-dimethylaminophenyl)-1,2-dihydroquinolines

,2-dihydroquinolines
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Yield	<i>6</i>	57	53	-76	60	58	57	30	42
alo	Z.	7,64	7,36	7,36	7,10	6,86	6,14	7,56	6,86
Calc.,	H	6,05	6,36	6,36	6,64	6,91	6,18	5,99	6,91
I	υ	81,94	82,07	82,07	82,20	82,32	84,18	77,81	82,32
	z	7,37 7,45	7,52 7,47	7,52	7,03 7,18	6,68 6,56	<b>6,20</b> 5,97	7,61 7,83	7,07
‰puno,	Н	6,09 6,11	6,60 6,41	6,57 6,57	6,86 6,78	6,70 7,00	$6,42 \\ 6,24$	6,07 6,17	6,87 6,94
Щ	υ	81,69 81,77	81,86 81,91	82,13 82,17	81.92 81,80	81,97 82,05	83,81 83,98	78,05 77,97	82,63 82,52
Empirical	formula	C <sub>25</sub> H <sub>22</sub> N <sub>2</sub> O	C26H24N2O	C26H24N2O	C27H26N2O	$C_{28}H_{28}N_2O$	$C_{32}H_{28}N_2O$	$C_{24}H_{22}N_{2}O_{2}$	C <sub>26</sub> H <sub>26</sub> N <sub>2</sub> O
	8 8	4,24	4,38	4,31	4,32	4,18	1	4,38	4,31
Y	шu	265	260	270	275	270	I	265	270
Q	f v	0,36	0,40	0,55	0,59	0,50	0,43	0,35	0,78
	Mp, °c	115-1168	1251268	150—151 b.c	110—112 b.d	<del>69</del> 70 a	110-111e	121—122 <b>a</b>	107—108 f
	u –			67	67	61	21	64	2
	<u>ي</u>	Н	Н	н	Н	Н	Н	Н	CH3
	à	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>	CH3	C <sub>2</sub> H5	C <sub>3</sub> H <sub>7</sub>	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>
	۲ ۲	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H5	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	2-Furyl	C <sub>6</sub> H <sub>5</sub>

a) From ethanol. b) From acetone. c) Previously [7] erroneously published as 52-53°. d) Previously [7] erroneously published as 58-59°. e) From Petroleum ether. f) From dimethylformamide.

TABLE 3. 2-Subst	ituted Qui	nolines												
					ц	ound, <sup>g</sup>	9	Ĭ	Calc.	10		Mono	picrate	
ļ			-	Empirical							rield,		'n	8
a X	à	Mp, "c	<i>f</i> .	formula	υ	Н	N	c	н	z	<i>d</i> <sub>0</sub>	.Mp. °c	punoj	calc.
p-(CH₃)₂NC₀H₄	6-HC <sub>3</sub>	185—186b	0,62	C <sub>18</sub> H <sub>18</sub> N <sub>2</sub>	81,66	7,02	10,51	82,41	6,92	10,68	46	206-207	13,91	14,25
<i>p</i> -(CH <sub>3</sub> ) <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	6-Br	1 <u>981</u> 95 b	0,80	C <sub>17</sub> H <sub>15</sub> BrN <sub>2</sub>	21'D/	/,I4 	8,83	62,40	4,62	8,56	25	215-216	12,90	12,59
<i>p</i> -(CH <sub>3</sub> ) <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	6-i-C <sub>3</sub> H <sub>7</sub>	125-126c	0,33	C20H22N2	I	1	9,48	1	1	9,56	12	161-061	13,82	13,48
p-(CH <sub>3</sub> ) <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	7-CH <sub>3</sub>	<b>p</b> 161061	0,70	C <sub>18</sub> H <sub>18</sub> N <sub>2</sub>		1	10,77	1	1	10,68	10	210-212	14,48	14,25
<i>p</i> -(CH <sub>3</sub> ) <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	8-CH <sub>3</sub>	156-156d	0,65	C <sub>18</sub> H <sub>18</sub> N <sub>2</sub>	82,46	7,12	11,14	82,41	6,92	10,68	ŝ	212-213	14,90	14,25
1-CH <sub>3</sub> . THQ	Н	113—114c	0,70	C <sub>19</sub> H <sub>18</sub> N <sub>2</sub>	82,88	6,71 6,71	10,53	83,18	6,61	10,21	46	204-205	14,01	13,91
I-C <sub>2</sub> H <sub>5</sub> - <b>T</b> HQ	H	73—74 c	0,73	C20H20N2	83,42	7,24	9,58	83,30	6,99	9,71	35	209-210	13,61	13,53
1-C2H5-2-CH3- THQ	н	70—71 c	0,63	$C_{21}H_{22}N_2$	77'00	1	9,28	1	1	9,26	9	227-228	13,06	13,18
1-n-C <sub>3</sub> H <sub>7</sub> - THQ	Н	e I	0,73	C21H22N2	1	1	0,12 12 12 12	ŀ	1	9,26	16	192-193	13,48	13,18
1-n-C4H9- THQ	H	l I	0,74	C22H24N2	83,74	7,51	700°	83,50	7,64	8,55	32	218-220	12,58	12,84
1-C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> -THQ	Н	7880c	0,75	$C_{25}H_{22}N_{2}$	85,95 85,95	6,37	8,01 8,01 8,02	85,68	6,33	2,99	18	228-230	11,80	12,06
I-CH <sub>3</sub> . DHI	Н	<b>80</b>	0,50	C <sub>18</sub> H <sub>16</sub> N <sub>2</sub>	In'n	۲ <u>۰</u>	2 2 2			ł	15	147148	14,58	14,31
1-C2H5- DHI	Н	ьо Г	0,58	C <sub>19</sub> H <sub>18</sub> N <sub>2</sub>		1	1		1	1	10	186—187	14,12	13.91
•.		-	-	•	-									

a) THQ is 1,2,3,4-tetrahydro-6-quinolyl; DHI is 2,3-dihydro-5-indolyl. b) From benzene. c) From ethanol. d) From propanol. e) Bp 230-235° (20 mm). f) Bp 275-280° (20 mm). g) Oil.

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as occurs in pyridinium [4] and alkylquinolinium [5] salts, rather than the participation of an N-acylquinolinium cation:



The formation of a complex of this type is facilitated with increasing anion nucleophilicity, and the regularity we observed can be explained by this.

This method of quinolination of activated aromatic rings was suitable for the introduction of a quinoline residue into the benzene ring of 1-alkyl-2,3-dihydroindoles and 1-alkyl-1,2,3,4-tetrahydroquinolines, as we have briefly reported in [6, 7]:



The benzene ring in indoline and tetrahydroquinoline molecules is activated by two electron-donor substituents – the amino group and the alkyl residue. Several isomeric products of quinolination can therefore be formed under conditions of electrophilic attack. However, when the reaction is carried out under mild conditions (50–60° under nitrogen) only one isomer, to which we assigned structure II on the basis of the analogy of the UV and IR spectra with the spectra of the corresponding quinolinated dialkylanilines (I), was always obtained.

For a more rigorous proof of the structure of the compounds obtained, 2-(1-methyl-1,2,3,4-tetrahydro-6-quinolyl)quinoline-4-carboxylic acid was obtained by reaction of aniline, 1-methyl-6-formyl-1,2,3,4-tetrahydroquinoline, and pyruvic acid; decarboxylation of the product acid led to 1-methyl-6-(2quinolyl)-1,2,3,4-tetrahydroquinoline, which was identical to a sample obtained by hydrolysis of the product of reaction of the 1-benzoylquinolinium salt with 1-methyl-1,2,3,4-tetrahydroquinoline:



Thus, as in the case of quinolination of dialkylanilines, ring-coupling at the  $\alpha$  position of the quinoline ring and in the para position with respect to the amino group linked to the benzene ring occurs during reaction of N-acylquinolinium salts with 1-alkyl-2,3-dihydroindoles and 1-alkyl-1,2,3,4-tetrahydroquinolines (Table 2).

Derivatives I and II were converted to the corresponding 2-substituted quinolines (Table 3) by alkaline or acidic hydrolysis. Moreover, in contrast to the hydrolysis of I, in which two compounds are formed [1], hydrolysis of II in all cases led only to aromatic structure III. Traces of the second compound were not detected:



Thus we were able to show that quinolination is a method that is quite broad in its synthetic possibilities. Our results on quinolination by this route of thiophene and pyrrole derivatives will be the subject of one of our future communications.

## EXPERIMENTAL

The purity of the coke-industry byproduct quinoline used in this investigation of the synthesized quinolines, 1-alkyl-2,3-dihydroindoles, and 1-alkyl-1,2,3,4-tetrahydroquinolines was verified by gas-liquid chromatography (UKh-1 chromatograph, 140°, column length 6 m, diameter 4 mm, stationary phase 0.4% ethylene oxide-tetrahydrofuran copolymer on sodium chloride, helium gas carrier). Thin-layer chromatography on aluminum oxide (activity II) in all cases was carried out with elution with a benzene-hexane-chloroform mixture (6:1:30) with development with iodine vapors in UV light. The UV spectra in ethanol were obtained with an SF-4 spectrophotometer; the IR spectra of the compounds were obtained with a UR-10 spectrophotometer (in KBr pellets or in 5-10% chloroform solutions).

1-Acyl-2-(p-dimethylaminophenyl)-1,2-dihydroquinolines (I). These were obtained as previously described in [1] (Table 1).

The 6-methyl-, 6-nitro-, 6-dimethylamino-, 6-bromo-, 7-methyl-, and 8-methylquinolines used in the reactions were obtained via the Skraup synthesis [8]; after distillation in vacuo or recrystallization (in the case of 6-nitro- and 6-dimethylaminoquinoline), the purity of the preparations was no less than 98-99% according to gas-liquid chromatography.

<u>Furoyl Chloride.</u> This was obtained by oxidation of furfural to furan-2-carboxylic acid with subsequent refluxing of this acid with thionyl chloride. The product had bp 89-90° (32 mm) [9].

Benzoyl Fluoride. This was obtained from benzoic anhydride and potassium fluoride [10] and had bp 150-155° and 35-38° (5 mm).

Benzoyl Bromide. This was obtained from benzoic acid and phosphorous tribromide [11] and had bp 216-218° and 75-78° (5 mm).

Benzoyl Iodide. This was obtained via the method in [12] and had bp 116-118° (8 mm) and 95-97° (5 mm).

<u>Reaction of Quinoline with Dimethylaniline and Benzoyl Halides.</u> A reaction mixture consisting of 0.08 mole of anhydrous quinoline, 0.04 mole of freshly distilled (over zinc dust) dimethylaniline, and 0.04 mole of freshly distilled benzoyl halide was heated at  $100 \pm 5^{\circ}$  under nitrogen for 8 h. The reaction mass was then steam distilled, and the residual amorphous product was extracted, dried, and recrystallized from ethanol. The yields of 1-benzoyl-2-(p-dimethylaminophenyl)-1,2-dihydroquinolines, respectively, were: 3.4 g (24%) in the reaction with benzoyl fluoride, 6.4 g (45%) with benzoyl chloride, 12.8 g (90%) with benzoyl bromide, and 13.5 g (95%) with benzoyl iodide. The product had mp 180-181°. Mixtures of samples obtained in experiments with the various benzoyl halides did not give a melting-point depression with an authentic sample.

<u>1-Alkyl-1,2,3,4-tetrahydroquinolines.</u> These were obtained as described in [13]. The purity of the samples used was no less than 98% according to gas-liquid chromatography.

<u>1-Alkyl-2,3-dihydroindoles.</u> These were obtained by alkylation of indoline [14]. The percentage of pure substance in the samples ranged from 97 to 98%.

 $\frac{1-\text{Benzoyl}-2-(1'-\text{methyl}-1,2,3,4-\text{tetrahydro}-6-\text{quinolyl})-1,2-\text{dihydroquinoline (II)} (R = C_6H_5, R' = CH_3, R' = H, n = 2). A mixture of 32.3 g (0.25 mole) of anhydrous quinoline, 17.6 g (0.125 mole) of freshly distilled benzoyl chloride, and 18.4 g (0.125 mole) of 1-methyl-1,2,3,4-tetrahydroquinoline was heated at 50-60° under dry nitrogen for 8 h. The reaction mixture was then decomposed with ammonium hydroxide, steam distilled, and the residual solid product was separated, dried, and recrystallized from acetone to give 36 g (76%) of snow-white crystals of 1-benzoyl-(1-methyl-1,2,3,4-tetrahydro-6-quinolyl)-1,2-dihydro-quinoline.$ 

The other 1-benzoyl-2-(1-alkyl-1,2,3,4-tetrahydro-6-quinolyl)-1,2-dihydroquinolines and -1,2-dihydroindoles (II) were similarly obtained. The yields and characteristics are presented in Table 2.

<u>1-Methyl-6-(2-quinolyl)-1,2,3,4-tetrahydroquinoline</u>. A. Fused sodium hydroxide (288 g) was added to a solution of 76 g of 1-benzoyl-2-(1-methyl-1,2,3,4-tetrahydro-6-quinolyl)-1,2-dihydroquinoline in 1 liter of 70% ethanol, and the mixture was refluxed for 3 h. After this, 1 liter of water was added to the reaction mixture, the ethanol was removed by distillation, and the residual reaction mass was repeatedly extracted with benzene. The benzene extracts were dried, half of the solvent was removed, 400 ml of nitro-

benzene was added, and the mixture was refluxed for 2 h. After this, the solution was repeatedly extracted with 10% hydrochloric acid. The acid extracts were made alkaline to pH 9-10, the resulting precipitate was separated and repeatedly washed with distilled water, dried, and recrystallized from ethanol to give 25 g (46%) of lemon-yellow crystals of 1-methyl-6-(2-quinolyl)-1,2,3,4-tetrahydroquinoline with  $\lambda_{max}$  284, 325, 377 nm, log  $\varepsilon$  4.14, 4.17, 4.37. The picrate (bright-red crystals) had mp 204-205° (from acetone).

The hydrolysis of other compounds of the I and II type was similarly carried out. The yields and characteristics of the compounds obtained are presented in Table 3.

B. One drop of sulfuric acid was added to a solution of 3.5 g (0.02 mole) of 1-methyl-6-formyl-1,2,3,4-tetrahydroquinoline (see below) and 1.87 g (0.02 mole) of aniline in 25 ml of absolute ethanol, and the mixture was heated for 1 h at 100°. After this, a solution of 1.76 g (0.02 mole) of freshly distilled pyruvic acid in 10 ml of absolute ethanol was added to the reaction mixture with cooling and stirring. The dark-red reaction mixture was heated at 100° for 20 h, and the solvent was removed to give 2 g of 2-(1methyl-1,2,3,4-tetrahydro-6-quinolyl)quinoline-4-carboxylic acid with mp 120-122°. This acid, without additional purification, was heated slowly to 300° on a metal bath for 1 h. The solid melt which formed was distilled in vacuo to give a light-brown, crystalline substance with bp 230-240 (20 mm), mp 110-112°, and  $R_f$  0.70. The spot on the chromatogram was identical to the spot of 1-methyl-6-(2-quinolyl)-1,2,3,4tetrahydroquinoline with respect to the  $R_f$  value and gave bright-blue fluorescence in UV light. The picrate was red and had mp 206-207° (from ethanol); a sample mixed with the picrate of the preparation obtained by method A did not give a melting-point depression.

<u>1-Methyl-6-formyl-1,2,3,4-tetrahydroquinoline</u>. Phosphorus oxychloride [11.5 g (0.075 mole)] was added dropwise with adequate stirring and cooling to a solution of 11.1 g (0.075 mole) of 1-methyl-1,2,3,4-tetrahydroquinoline in 16.4 g (0.225 mole) of dry dimethylformamide at such a rate that the temperature did not rise above 20°. After this, stirring was continued for another 1 h at the same temperature, and the mixture was then heated at 100° for 3 h. The reaction mixture was poured over ice, and the resulting mixture was neutralized with 0.5-N alkali and allowed to stand overnight in the cold. The precipitated crystals of 1-methyl-6-formyl-1,2,3,4-tetrahydroquinoline were then filtered. Distillation in vacuo gave 8.4 g (67%) of light-yellow plates with mp 28-29 g and bp 216-218° (20 mm). According to [15], 1-methyl-6-formyl-1,2,3,4-tetrahydroquinoline with N-methylformanilide.

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