## CONDENSED HETEROCYCLIC SYSTEMS WITH A QUATERNARY NITROGEN ATOM.

## III.\* SYNTHESIS OF BENZIMIDAZO[3,2-b]ISOQUINOLINIUM PERCHLORATES

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Isochromylium cations containing easily detached groupings (NHCOR,  $NH_2$ ,  $OCH_3$ ) in the 3 position react readily with o-phenylenediamine. The reaction is accompanied by opening of the benzopyrylium ring and condensation of the resulting compound at one or two amino groups of o-phenylenediamine. Benzimidazo-[3,2-b]isoquinolinium salts were synthesized alternatively by acylation of 2-(3,4-dimethoxybenzyl)benzimidazole. The tautomerism of the synthesized N-(2aminoaryl)isoquinolinium perchlorates was investigated.

It has been shown [2] that N-diazoarylpyridinium diperchlorates are cyclized via the Pschorr method to give quinolizinium cations. The substantial noncoplanarity of the pyridinium and N-phenyl rings [3, 4] in the starting compounds is not an obstacle to the formation of a planar quinolizinium system. This has been explained [5] by interaction of the N-phenyl and  $\alpha$ -phenyl groupings, apparently as a result of twisting vibrations. In this connection, we investigated the structure and properties of the amino group in 3-hydroxy-N-(2-aminoaryl)isoquinolinium salts in which the vibrations of the N-aryl grouping about the C-N bond should be stabilized by an intramolecular hydrogen bond. A convenient method for the synthesis of N-(2-aminoaryl)isoquinolinium salts, which we presented in [1], consists in reaction of isochromylium salts I with o-phenylenediamine. However, we found that benzimidazo[3,2-b]isoquinolinium perchlorates are formed along with the desired compounds.

The direction of the reaction depends to a considerable degree on the solvents used and the temperature. Thus N-(2-aminoaryl)isoquinolinium perchlorates (V) are formed in acetone in the cold. Benzimidazo[3,2-b]isoquinolinium perchlorates (VI) were obtained by heating 2-benzopyrylium salts and o-phenylenediamine in ethanol. Ketones III were isolated when 80% aqueous acetone was used as a solvent. The use of other solvents [dimethylformamide (DMF), tetrahydrofuran (THF), and chloroform] leads to a reduction in the yields of salts V or to resinification.



\*See [1] for communication II.

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		1		Empirical	Fou %	nd,		Ca %	lc.,		IR spects cm <sup>-1</sup>	:a,	d, %
Doun	R	X	mp, C	formula	с	н	N	с	Н	N	<sup>ν</sup> C = 0	v <sub>N H</sub>	<b>Yiel</b>
IIIa IIIb IIIc IIId IIIe IIIf IIIg	CH3 CH3 C2H5 C2H5 C3H7 C3H7 C3H7 C3H7	NHCOCH <sub>3</sub> NH <sub>2</sub> NHCOC <sub>2</sub> H <sub>5</sub> NH <sub>2</sub> NHCOC <sub>3</sub> H <sub>7</sub> NH <sub>2</sub> OCH <sub>3</sub>	$\begin{array}{c} 164 - 165\\ 234 - 235\\ 106 - 107\\ 203 - 204\\ 229 - 230\\ 147 - 148\\ 170 - 171\end{array}$	$\begin{array}{c} C_{14}H_{17}NO_5\\ C_{12}H_{15}NO_5\\ C_{16}H_{21}NO_5\\ C_{13}H_{17}NO_4\\ C_{18}H_{25}NO_5\\ C_{18}H_{18}NO_4\\ C_{18}H_{18}O_5 \end{array}$	59,8 60,4 62,1 62,3 64,3 62,7 <b>68,</b> 3	6,5 6,0 6,9 7,6 7,3 6,1	5,4 6,2 4,1 5,9 4,0 5,5 —	60,2 60,8 62,5 62,1 64,5 63,3 68,7	6,1 6,3 6,8 6,8 7,5 7,2 5,7	5,0 5,9 4,5 5,5 4,1 5,2 —	1735, 1715 1725, 1660 1720, 1670 1725, 1675 1725, 1645 1725, 1675 1725, 1675 1730, 1715	3205 3225 3205 3225 3225 3380 3230	90 91 84 50 71 94 66

TABLE 1. Ketones III Formed as a Result of Opening of the Isochromylium Rings

Addition product II, which is recyclized to N-arylisoquinolinium perchlorate IV, as in the reaction of 2-benzopyrylium salts with primary aromatic amines [6], is probably formed in an intermediate step of the reaction. The 3-substituted N-arylisoquinolinium salts (IV) obtained in this way undergo hydrolysis in aqueous acetone in the cold to give 3-hydroxy-Narylisoquinolinium perchlorates V, whereas they are cyclized to benzimidazo[3,2-b]isoquinolinium perchlorates VI on heating in ethanol.

The structure of the synthesized benzimidazo[3,2-b]isoquinolinium perchlorates (VI) was confirmed by alternative synthesis. The reaction may also be realized by direct heating of 2-(3,4-dimethoxybenzyl)benzimidazole (VII) with perchloric acid in the appropriate anhydride (R = Alk). In this case the acyl group attacks the veratrole ring rather than the benzimidazole ring in the 1 position, as sometimes is observed [7]. This was proved by means of the IR spectra and also by alkylation of VIII (R = CH<sub>3</sub>) at the NH group.



We studied the structure and properties of N-(2-aminoaryl)isoquinolinium perchlorates V, during which the most expedient task was to study the tautomerism, which determines the presence of an intramolecular hydrogen bond.

At present the tautomerism of isoquinolines has been studied quite completely [8, 9]. It has been established that, depending on the solvent and the character of the substituent in the 1 position, isoquinolones IX exist in lactam or lactim forms. However, in subsequent papers [10-13] the existence of isoquinolones in the form of 3-oxo-4H derivative X is assumed. This was explained by the absence in the IR spectra of IX of the absorption band of a hydroxyl group [6, 11]. The synthesis of 4,4-dimethyl-1-phenyl-2-isoquinolone as a model compound has also been realized [14], but the electronic spectra of the latter have not been studied. The most likely interpretation of the IR spectra was presented by McCorkindale and McCulloch [9], who observed a change in the intensity of a group of bands in the IR spectra of isoquinolones IX (R<sup>1</sup> = H) on passing from the lactam form in THF to the lactim form in ethanol. The IR spectra of solid isoquinolones IX provide evidence for the existence of strong intermolecular hydrogen bonds. The signals of only aromatic protons have been identified in the PMR spectra of isoquinolines IX [9].



The synthesized N-aminoarylisoquinolinium perchlorates (V) should not exist in the lactam form, and the possible equilibrium was therefore studied. The signals of methylene protons characteristic for Va are absent in the PMR spectra of V. Solutions of V are violet (like phenols) in the presence of ferric chloride. Compounds V do not display the properties of o-quinoid tautomers on reaction with N-phenylmaleinimide by the method in [8]. The IR spectra

-p			Empirical	Fo	ounc	1, %	,	Ca	ılc.,	%		λ <sub>mar</sub> , nm	d, 0%
Con	R	mp, °C	formula	с	н	CI	N	с	н	C1	N	(lg ε)	Yiel
Va	CH3	305—306	$C_{18}H_{19}CIN_2O_7$	53.0	4,9	8,3	6,9	52,6	4,6	8,6	6,8	255 (4,73) 320 (3,81) 420 (3,63)	71
Vb Vc	$\begin{array}{c} C_2H_5\\ C_3H_7 \end{array}$	230—231 162—163	C <sub>19</sub> H <sub>21</sub> ClN <sub>2</sub> O <sub>7</sub> C <sub>20</sub> H <sub>23</sub> ClN <sub>2</sub> O <sub>7</sub>	54, <b>1</b> 55,0	5,2 5,6	8,0 8,4	6,8 6,9	53,7 54,7	4,9 5,2	8,3 8,1	6,6 6,3	$260 (4,73)^*$ 255 (4,65) 405 (3.57)	75 72
Vd	C <sub>6</sub> H <sub>5</sub>	235236	C <sub>23</sub> H <sub>21</sub> ClN <sub>2</sub> O <sub>7</sub>	58,0	4,7	7,3	6,0	58,4	4,4	7,5	5,0	$ \begin{vmatrix} 280 & (4,87) \\ 310 & (4,72) \\ 410 & (3,53) \end{vmatrix} $	71

TABLE 2. 3-Hydroxy-6,7-dimethoxy-N-(2-aminophenyl)isoquinolinium Perchlorates (V)

\*Longwave structureless absorption at 410 nm.

of solid V contain absorption bands of the  $v_{\rm S}$  and  $v_{\rm aS}$  stretching vibrations of an amino group at 3485 and 3555 cm<sup>-1</sup>, respectively, and a group of intense bands at 1630 cm<sup>-1</sup>. The low solubility of perchlorates V in organic solvents does not make it possible to study their IR spectra in solution and establish the presence of an intramolecular hydrogen bond. The existence of the latter probably can be confirmed by an indirect method. It is known that this sort of bond is capable of inhibiting many chemical reactions [15]. Thus, compounds V, in contrast to isoquinolones IX, do not form O-acetyl derivatives and are not alkylated on fusion with methyl p-toluenesulfonate; this is characteristic for a chelated phenolic hydroxyl group. On the other hand, the amino group in salts V is inert with respect to acylating and diazotizing reagents and also does not undergo reaction with aldehydes. Despite the drawing together of the reaction centers due to an intramolecular hydrogen bond, we were unable to bring about the intramolecular condensation of V by heating in ethanol, glacial acetic acid, or acetic anhydride. This is apparently explained both by the inert character of the phenolic hydroxyl group and by the probable decrease in the basicity of the amino group because of compression of the molecule.



All of the material set forth above makes it possible to assume that perchlorates V exist in hydroxyl form Vb, which is stabilized by an intramolecular hydrogen bond.

## **EXPERIMENTAL**

The IR spectra of mineral oil suspensions of the compounds were recorded with UR-20 and Specord 71IR spectrometers. The UV spectra of methanol solutions  $(10^{-5} \text{ M})$  were recorded with an SF-4a spectrophotometer. The PMR spectra of trifluoroacetic acid solutions were recorded with an RYa-2305 spectrometer at room temperature (with hexamethyl disiloxane as the internal standard).

<u>1-Alky1-3-acetylamino-6,7-dimethoxy-2-benzopyrylium Perchlorates (Ic, f).</u> These salts were obtained by the method in [12] as yellow-green substances, which were crystallized from glacial acetic acid (see Table 5).

<u>1-Alkyl-3-amino-6,7-dimethoxy-2-benzopyrylium Chlorides (Ia,d,g) (Table 5)</u>. These compounds were obtained by heating the acylamino derivative in concentrated hydrochloric acid for 1 h. The products were colorless prisms (from concentrated HCl), as were the perchlorates (from ethanol). The bands at 1648 and 1620 cm<sup>-1</sup>, which characterize the isochromylium structure, are retained in the IR spectra as an inflection on the intense band at 1669 cm<sup>-1</sup> from the scissors vibrations of the NH<sub>2</sub> group. The  $v_s$  and  $v_{as}$  stretching bands of the amino group are found at 3390 and 3550 cm<sup>-1</sup>, respectively. TABLE 3. Benzimidazo[3,2-b]isoquinolinium Perchlorates (VI)

F			Empirical	Foui	, pr	₫0		Ca	lc.,	\$			1%'1
ounod woj	2	mp. c	formula	υ	Ξ	Ū	z	υ	H.	ū	z	(3 gi) IIIII (3 gi)	γιεια
VIa	CH <sub>3</sub>	316-317	C <sub>18</sub> H <sub>17</sub> CIN <sub>2</sub> O <sub>6</sub>	55.0	4,5	8,7	7.0	55,0	4,3	9,0	7,1	250 (4,87) 315	67
Vib	$C_2H_5$	313-314	C <sub>19</sub> H <sub>19</sub> ClN <sub>2</sub> O <sub>6</sub>	55,6	5,0	8,8	7,3	56,1	4,7	8,7	6,8	(4,10) 303 (3,/8) 280 (4,30) 290	26
VIC	$C_3H_7$	307-308	C <sub>20</sub> H <sub>21</sub> CIN <sub>2</sub> O <sub>6</sub>	56,5	5,2	8,3	6,8	57,0	5,0	8,4	6,7	278 (4,88) 340 278 (4,88) 340	50
ΝId	C <sub>6</sub> H <sub>5</sub>	223-224	C <sub>23</sub> H <sub>19</sub> CIN <sub>2</sub> O <sub>6</sub>	61,1	4,3	7,6	6,4	60,7	4,2	7,8	6,1	(3,40) 300 (3,80) 280 (4,77); 310 (4.58)	30
		_	_	-	-	-	-	-	-		-	(00(1)	

TABLE 4. 2-(2-Acyl-4,5-dimethoxybenzyl)benzimidazoles (VIII)

r		r
ʻp	Viel Viel	60 62 66 66
ctrum,	N N H	3465 3465 3215 3215 3460
IR spe cm <sup>-1</sup>	VC=0	1725 1745 1730 1720
	z	9,0 8,6 7,5
	Н	0,0,0 4,0,2,8
Ca] %	υ	69,6 70,3 70,9 74,1
	z	9,3 8,7 8,0 7,1
Ъd,	Н	5,7 6,7 6,1 5,7
Four %	U	69,2 70,5 70,7 74,3
Empirical	formula	C <sub>18</sub> H <sub>18</sub> N <sub>2</sub> O <sub>3</sub> C <sub>19</sub> H <sub>20</sub> N <sub>2</sub> O <sub>3</sub> C <sub>20</sub> H <sub>22</sub> N <sub>2</sub> O <sub>3</sub> C <sub>23</sub> H <sub>20</sub> N <sub>2</sub> O <sub>3</sub>
	ר קוו חוף	228—229 167—168 102—103 214—215
	ĸ	CH C3H C3H
Com-	punod	VIIIa VIIIb VIIIb VIIIc VIIIc

TABLE 5. 1-Alkv1-3-amino(acylamino)-6,7-dimethoxy-2-benzopyrylium Salts (1)

E CLARK	11610, %	Quantitative Quantitative 86 Quantitative Quantitative 79 Quantitative Quantitative
	z	ν4 ων 4 ω 4 4 ν 6 ο - 0 4 ο 0
96	G	13,8 9,1 11,0 13,1 13,1 10,6 12,4 10,2
Calc.,	H	ສຸດ, 4, 3, 5, 4, 5 2, 4, 7, 8, 9, 7, 4, 5 2, 4, 7, 8, 9, 7, 4, 5 2, 4, 7, 8, 8, 9, 7, 7, 7, 7, 7, 7, 7, 7, 7, 7, 7, 7, 7,
	υ	56,4 45,0 57,8 51,7 48,4 59,2 7,8 48,4 48,4
	z	0,4,0,0,4,0,0,4,0,0,4,0,0,4,0,0,4,0
.0	ū	13,6 11,2,6 10,3,9,2,8,9 10,3,3,9,2,8,9 10,4,2,8,9,5,5 10,4,2,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5,5
ound, 9	H	ထိုနှက်တွင်တွင် ဝက်ဖင်စုဝင်စု-
F	υ	56,7 45,2 45,4 57,9 46,4 46,9 46,4 48,6
Empirical	formula	C12H14NO3C1 C12H14NO3C1 C12H14NO3C1 C13H16NC103 C13H16NC103 C13H16NC103 C13H16NC103 C13H16NC103 C13H16NC103 C13H16NO3C1 C14H18NO3C1
*), um	, dan	238–239 254–255 188–189 139–140 139–140 145–146 199–200 199–200
Anton V-	T HOTH	000000000000000000000000000000000000000
;	×	NH2 NH2 NHCOC2H5 NH2 NH2 NH2 NH2 NH2 NH2
	¥	
Com-	punod	84 75 87 94

\*Salts Ia,d,g were crystallized from concentrated hydrochloric acid; Ib,e,h were crystallized from ethanol; and Ic,f were crystallized from glacial acetic acid.

<u>1-Phenyl-3,6,7-trimethoxy-2-benzopyrylium Perchlorate</u>. A mixture of 1.05 g (5 mmole) of methyl homoveratrate, 2.4 ml (10 mmole) of benzoyl chloride, 2.5 g (10 mmole) of silver perchlorate, and 10 ml of absolute nitromethane was heated on a water bath for 10 min, after which it was poured over ice, and the resulting oil was separated and triturated with ether. The resulting yellow precipitate was removed by filtration and crystallized from glacial acetic acid to give a product with mp 170° in 60% yield. The physical constants of the product are in agreement with the data in [6].

1-Alky1-3-hydroxy-6,7-dimethoxy-N-(2-aminopheny1) isoquinolinium Perchlorates (Va-c) (Table 2). A 2.37-g (8 mmole) sample of salt I, 0.81 g (8 mmole) of o-phenylenediamine, and 20 ml of acetone containing 2% water were mixed, during which heat evolution was observed. The mixture was then allowed to stand at room temperature for 10 h, after which the solvent was removed by distillation. The residual mass was triturated with ethanol, and the solid material was removed by filtration and purified by reprecipitation from DMF by the addition of water to give crystalline substances, the physical constants of which are presented in Table 2.

<u>l-Phenyl-3-hydroxy-6,7-dimethoxy-N-(2-aminophenyl)isoquinolinium Perchlorate (Vd)</u> (Table 2). The compound was obtained by the method described above in glacial acetic acid. The crystallization solvent was nitromethane.

Ketones IIIa-g (Table 1). These ketones were obtained by the method described above with a water content of more than 2 moles in the reaction mixture. The precipitate that formed after the reaction mixture had been stirred for 10 h was removed by filtation to give colorless prisms (from ethanol).

7-Alky1-9,10-dimethoxybenzimidazo[3,2-b]isoquinolinium Perchlorates (VI). A. An equimolecular mixture of benzopyrylium salt I and o-phenylenediamine was heated in ethanol for 2 h, after which isoquinolinium perchlorate VI was removed by filtration.

B. A 1-mmole sample of 70% perchloric acid was added gradually to a solution of 1 mmole of 2-(3,4-dimethoxybenzyl)benzimidazole in 16 mmole of acetic anhydride, after which the mixture was heated at 80° for 10 min, and the precipitated VI was removed by filtration. The crystallization solvent was nitromethane.

The benzimidazo[3,2-b]isoquinolinium perchlorates synthesized by the two methods had identical melting points and IR spectra and were obtained as yellow crystalline substances (from nitromethane) (Table 3).

<u>2-(3,4-Dimethoxybenzyl)benzimidazoles (VII)</u>. A mixture of 1.08 g (10 mmole) of ophenylenediamine, 1.97 g (10 mmole) of homoveratric acid, 1 ml of ethanol, 1.5 ml of concentrated HCl, and 1 ml of orthophosphoric acid was heated at 130° for 2 h, after which it was dissolved in 10% hydrochloric acid and refluxed with charcoal. The product was precipitated by the addition of ammonia and was obtained as colorless prisms, which were crystallized from aqueous ethanol to give 2 g (74%) of a product with mp 159-160°. IR spectrum: 3510 and 1620 cm<sup>-1</sup>. Found, %: C 71.3; H 6.4; N 10.4. C<sub>16</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>. Calculated, %: C 71.6; H 6.0; N 10.4.

2-(2-Acyl-4,5-dimethoxybenzyl)benzimidazoles (VIIIa-c). These compounds were obtained by heating VIIa-c in the appropriate anhydride in the presence of traces of 70% perchloric acid. The products were colorless (crystallization from ethanol). The physical constants of VIIIa-c are presented in Table 4.

<u>2-(2-Benzoyl-4,5-dimethoxybenzyl)benzimidazole (VIIId)</u>. A 0.25-ml (18 mmole) sample of benzoyl chloride and a catalytic amount of silver perchlorate were added to a solution of 0.5 g (18 mmole) of VII in 5 ml of dry nitromethane, and the resulting colorless precipitate was removed by filtration and crystallized from ethanol (Table 4).

<u>1-Methyl-2-(3,4-dimethoxy-6-acetylbenzyl)benzimidazole</u>. An excess amount of an ether solution of diazomethane was added to a cold solution of VIIIa in ethanol, and the mixture was allowed to stand at 0° for 2 h and at room temperature for 10 h. The resulting precipitate was removed by filtration and crystallized from ethanol to give a colorless product with mp 135° in 53% 53% yield. Found, %: C 69.9; H 6.1; N 8.3.  $C_{19}H_{20}N_2O_3$ . Calculated, %: C 70.4; H 6.2; N 8.6.

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STUDY OF THE KINETICS OF THE CHICHIBABIN REACTION

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The rate of hydrogen and ammonia evolution during the amination of N-heteroaromatic systems with sodium amide was studied. The ease of the Chichibabin reaction of heterocycles decreases in the following order: 1-methylbenzimidazole > isoquinoline > 1-methylperimidine > benzo[f]quinoline > pyridine >> acridine. The decrease in the rate of amination in the presence of freeradical inhibitors constitutes evidence for a free-radical step in the Chichibabin reaction. It is shown that the Chichibabin reaction has autocatalytic character and is accelerated in the presence of sodium salts of heterylamines. Opinions regarding the mechanism of this autocatalysis are expressed.

The mechanism of the amination of nitrogen heterocycles by means of sodium amide (the Chichibabin reaction) remains one of the least investigated nucleophilic substitution reactions in the aromatic and heteroaromatic series. This is due to a considerable extent to the difficulty in the study of the kinetics of an amination process that takes place under heterogeneous conditions and at high temperatures. However, the evolution of hydrogen and a small amount of ammonia is one of the characteristic features of the Chichibabin reaction [1]. In the present research we attempted to study the reaction rate from the amount of evolved hydrogen and also attempted to determine the ratio of hydrogen and ammonia during the amination of various heterocycles. Compounds — pyridine, isoquinoline, benzo[f]quinol-ine, 1-methylbenzimidazole, and 1-methylperimidine [1] — for which the Chichibabin reaction proceeds most smoothly and with the minimum amount of side products were selected as the subjects for this investigation. Several experiments were also carried out with acridine, the amination of which proceeds with complications [2]. It was assumed that the results of this

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