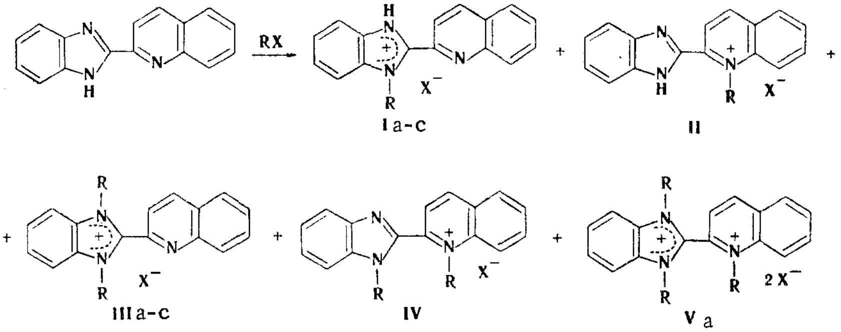
QUATERNARY SALTS OF QUINOLYBENZIMIDAZOLES

I. V. Romanenko, A. K. Sheinkman,
S. N. Baranov, V. N. Poltavets,
and N. A. Klyuev

UDC 541.62:547.785.1.2'832

The nitrogen atoms of the benzimidazole ring are alkylated initially in the quaternization of 2-(2-quinolyl)benzimidazole, and the nitrogen atom of the quinoline ring is alkylated only after this. Pyridyl- and quinolylbenzimidazoles and their quaternary salts were synthesized in order to study geometrical isomerism in the dihetaryl series. It was concluded that geometrical isomerism cannot occur in the case of the protonated forms or any other quaternary salts of dihetaryls, since as a result of the mutual electron-acceptor effect of the heteroaromatic cations the carbon–carbon bond between them is converted to a single σ bond, despite the fact that it is a bond between two sp^2 carbon atoms.

The first atom to undergo quaternization in the alkylation of 2-(4-pyridyl)benzimidazole is the heteroatom of the pyridine ring to give benzimidazolylpyridinium salts [1], which display high physiological activity [2]. On the other hand, 2-(2-pyridyl)benzimidazole undergoes quaternization in the benzimidazole ring [1]. It seemed of interest to determine the direction of quaternization of 2-quinolylbenzimidazole, which is a dihetaryl system with "pyridine" nitrogen atoms with similar steric accessibility and basicities in different rings. The formation of five different compounds (I-V) is theoretically possible in this reaction:



I a R=H, X=I; b R=CH_3, X=I; c R=CH_3, X=CH_3SO_4; III, V a R=CH_3, X=I; III b R=CH_3, X=CH_3SO_4; c R=C_2H_5, X=I

The direction of the reaction depends both on steric factors and on the electron-density distribution in the molecule, which, in turn, is a function of the conformational processes in the molecule and the associated different degrees of conjugation of the hetaryls in it.

Recently two of us [3] in the case of quinolylindoles demonstrated for the first time the possibility of the existence of the previously predicted [4] hindered s-cis and s-trans conformers in the dihetaryl series. It was assumed that this sort of isomerism is observed in all cases in which the rings in the conjugated molecules of diaryls or dihetaryls contain substituents in the meta positions that do not create steric hindrance or heteroatoms in the ortho or meta positions relative to the interannular bond; this is explained by the hindrance to rotation of the rings about this bond, which has a considerable degree of double bond character [4]. Other examples that confirm the existence of hindered rotation about the formal σ bond between the two sp^2-hybridized carbon atoms in pyridylazoles [2] and other dihetaryls and their protonated forms [5] have appeared in the literature. However, we assumed that impeded rotation of the rings cannot occur in the case of the protonated forms or any

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Institute of Physical Organic Chemistry and Coal Chemistry, Academy of Sciences of the Ukrainian SSR, Donetsk 340048. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 12, pp. 1662-1669, December, 1980. Original article submitted October 17, 1979; revision submitted May 19, 1980.

0009-3122/80/1612-1261$07.50

TABLE 1. Quaternary Salts of Quinolylbenzimidazoles and Dibenzimidazolyls

Compound	mp,* °C	UV spectrum λmax, nm (log ε)	Found, %				Empirical formula	Calc., %				Yield, %
			C	H	I	N		C	H	I	N	
Ia	254—256	206 (4.44), 245 (4,44), 285 (4,07)	51,2	3,4	34,1	11,2	$C_{16}H_{12}IN_3$	51,5	3,2	34,0	11,3	60
Ib	170—171		52,4	3,7	32,5	10,7	$C_{17}H_{14}IN_3$	52,7	3,7	32,8	10,8	70
Ic	143—144	206 (4,31), 242 (4,28), 333 (4,06)	58,4	4,8	—	11,2	$C_{18}H_{17}N_3O_4S$	58,2	4,6	—	11,3	72
IIIa	282—284	206 (4,39), 246 (4,39), 282 (4,02)	53,7	4,2	31,7	10,4	$C_{18}H_{16}IN_3$	53,9	4,0	31,6	10,5	52
IIIb	200—201	205 (4,26), 246 (4,29), 283 (3,92)	59,2	5,0	—	10,7	$C_{19}H_{19}N_3O_4S$	59,2	4,9	—	10,9	47
IIIc	220—223	205 (4,62), 233 (4,46), 285 (3,95)	56,2	4,8	29,8	9,7	$C_{20}H_{20}IN_3$	55,9	4,7	29,6	9,8	65
Va	243—245		42,2	3,6	46,6	7,5	$C_{19}H_{19}I_2N_3$	42,0	3,5	46,7	7,7	30
XIIIa	156—158	205 (4,61), 236 (4,43), 320 (4,23)	56,6	4,0	—	11,7	$C_{17}H_{14}ClN_3O_4$	56,8	3,9	—	11,7	61
XIIIb	303—305	205 (4,92), 323 (4,25)	53,7	4,2	31,6	10,4	$C_{18}H_{16}IN_3$	53,9	4,0	31,6	10,5	56
XIIIc	>340	206 (4,42), 236 (4,36), 320 (4,19)	59,0	4,8	—	10,8	$C_{19}H_{19}N_3O_4S$	59,2	4,9	—	10,9	58
XIIId	318—320	206 (4,25), 240 (4,21), 323 (4,07)	61,9	5,1	—	11,4	$C_{19}H_{18}BrN_3$	61,9	4,9	—	11,4	30
XIIIe	292—293		54,6	4,2	30,5	10,2	$C_{19}H_{18}IN_3$	54,9	4,4	30,6	10,1	59
XIVa	>340		50,4	3,4	—	8,0	$C_{22}H_{17}Cl_2N_3O_8$	50,6	3,3	—	8,1	35
XIVb	295—296		59,8	4,1	27,3	9,0	$C_{23}H_{18}IN_3$	59,6	3,9	27,4	9,1	28
XV	>340	217 (4,64), 272 (4,57), 339 (4,25)	53,1	3,5	32,8	10,9	$C_{34}H_{26}I_2N_6$	52,9	3,4	32,9	10,9	46
XVI	250—252	223 (4,79), 305 (4,39)	50,4	4,3	31,6	14,1	$C_{17}H_{17}IN_4$	50,5	4,2	31,4	13,9	30
XVII	317—318	270 (4,20), 277 (4,25)	39,4	3,6	46,2	10,2	$C_{18}H_{20}I_2N_4$	39,6	3,7	46,5	10,3	35
XVIIIb	224		52,1	4,8	—	13,2	$C_{14}H_{15}N_3O_4S$	52,3	4,7	—	13,1	65

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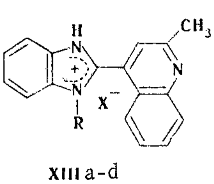
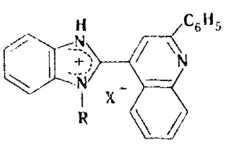
*The compounds were recrystallized: IIIa, c, XIIIa, b, and XVII from methanol, XIVa, b and XVIIIc from ethanol, Ia-c, IIIb, V, and XIIIe from isopropyl alcohol, and XIIIc, d, XV, and XVI from DMF.

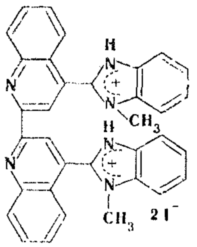
other quaternary salts of dihetaryls if there is no steric hindrance, since as a consequence of the mutual electron-acceptor effect of the heteroaromatic cations, the carbon—carbon bond between them is converted to a single σ bond. To study the conformational effects we synthesized known [6-8] and previously undescribed compounds, viz., 2-(2-pyridyl)benzimidazole (VI), 2-(3-pyridyl)benzimidazole (VII), 2-(4-pyridyl)benzimidazole (VIII), 1-methyl-2-(2-quinolyl)benzimidazole (IX), 1,1'-dimethyl-2,2'-dibenzimidazolyl (X), 2,2'-dibenzimidazolyl (XI), and 2-(2-quinolyl)benzimidazole (XII), as well as their mono- and bisquaternary salts (Table 1):



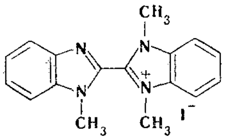
XIII a-d



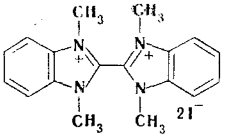
XIV a,b



XV



XVI



XVII

XIII, XIV a R=H, X=ClO_4; b R=CH_3, X=I; XIII c R=CH_3, X=CH_3SO_4; d R=C_2H_5, X=Br; e R=C_2H_5, X=I

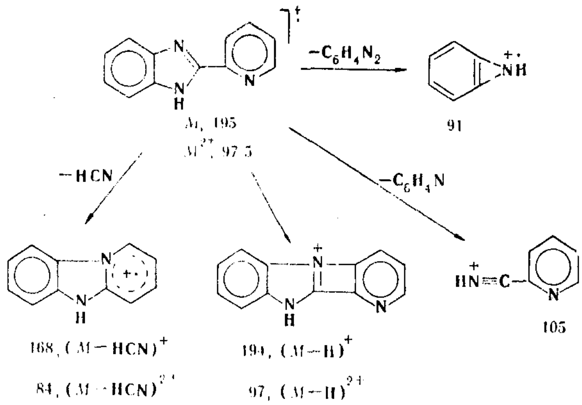
All salts I-V and XIII-XVII were obtained by direct alkylation of the corresponding bases with methyl iodide or dimethyl sulfate. It was found that of the five possible compounds, only three, viz., I, III, and V, are formed in the quaternization of 2-(2-quinolyl)benzimidazole, i.e., the nitrogen atoms of the benzimidazole ring are alkylated initially, and the nitrogen atom of the quinoline ring is alkylated only after this. The structure of Ib was confirmed by alternative synthesis. A compound identical to salt Ib was obtained as a result of the addition of hydriodic acid to a genuine sample of 1-methyl-2-(2-quinolyl)benzimidazole. The IR spectra of Ia-c contain absorption bands of stretching vibrations of NH groups at 3400-3500 cm^{-1}. Their UV spectra are similar: λ_{max}(log ε): 206 (4.44), 245 (4.44), and 285 nm (4.07). The PMR spectra of I and III each contain one signal of methyl protons of a benzimidazole ring (with intensities of 3H and 6H, respectively) at 4.16-4.2 ppm, while the spectrum of salt V contains two signals of protons of methyl groups of benzimidazole [4.2 ppm (6H)] and quinoline [4.59 ppm (3H)] rings; this is in complete agreement with the described PMR spectra of pyridylbenzimidazole methiodides [1]. The mass spectra of salts I, III, and V also confirm their molecular weights and structures: the spectrum of I contains, in addition to a molecular ion, peaks of HI ions and starting base IX, the fragmentation of which is similar to the fragmentation of the isomeric pyridyl- and quinolylbenzimidazoles (Table 2), while peaks of ions due to cleavage of the carbon—carbon interannular bond and splitting out of methyl groups together with a gegenion in the form of CH_3I and $(CH_3)_2SO_4$, which lead to ions with the structure of the corresponding base, appear in the mass spectra of salts III and V.

The principal mass-spectrometric criteria of the existence of conjugation of the heterorings and double-bond character of the bond between them in diaryls are the stability of the molecule with respect to electron impact W_M (high W_M values comparable to the W_M values for the heterocycles that make up the molecule of the diheteroaromatic system) and the absence in the spectrum of ion peaks of the corresponding heterocyclic fragments [3]. It is apparent from Table 2 that the stabilities of the molecules are quite high and comparable to the W_M values of quinoline, benzimidazole, and pyridine [9]; their W_M values decrease somewhat in the order of substituted pyridines in the direction $4 > 2 > 3$, which corresponds to the literature data for various dipyridyls [10] and diquinolyls [4]. The spectra do not contain peaks of ions of benzimidazole, pyridine, and quinoline, the formation of which is due to cleavage of the bond between the hetaryl rings in the molecules; this constitutes evidence for the double-bond character of the bond between them. The most intensive processes involved in fragmentation of the molecular ion (M^+) are associated with cyclization of the rings with elimination of neutral acetylene or HCN particles (detachment of HCN is realized only from

TABLE 2. Analytical Peaks of the Ions of Isomeric Pyridyl- and Quinolylbenzimidazoles and Dibenzimidazolyls (the intensities are given in percent of the maximum ion peak)

Ions	Compounds						
	VI	VII	VIII	IX	X	XI	XII
W_M	25,5	19,5	34,9	28,0	21,5	35,1	39,4
M^+	100,0	100,0	100,0	100,0	82,0	100,0	100,0
$(M-H)^+$	20,7	41,8	15,7	8,1	100,0	12,2	8,3
$(M-C_2H_2)^+$	14,3	34,9	—	—	—	3,6	—
$(M-HCN)^+$	25,9	8,5	5,2	—	1,6	2,4	2,6
$[(M-H)-HCN]^+$	13,5	8,7	9,6	—	6,6	1,0	2,0
M^{2+}	1,1	12,8	14,9	4,6	6,0	3,9	3,6
$(M-H)^{2+}$	1,2	1,0	2,3	3,2	1,0	3,1	—
$(M-C_2H_2)^{2+}$	1,7	—	—	—	—	1,2	—
$(M-HCN)^{2+}$	7,8	3,8	—	—	3,6	—	—
$[(M-H)-HCN]^{2+}$	2,0	1,7	1,3	—	2,0	5,1	—
m/e 105	9,9	—	1,2	—	1,6	—	2,2
m/e 91	12,4	6,4	1,4	—	3,2	7,2	3,5
m/e 90	27,0	9,6	3,0	1,3	4,7	8,6	6,9

the pyridine part of the molecule, since the 2 position in the benzimidazole ring is blocked) and the formation of double charged ions and fragment ions that have polynuclear aromatic structures. The fragmentation of isomeric VI-XII is described by the following scheme (in the case of VI, Table 2):



A similar fragmentation pathway is also retained in the case of the dissociative ionization of quinolylbenzimidazole IX (Table 2). The presence of a methyl substituent in the benzimidazole part of the molecule leads to expansion of the imidazole ring to a pyridine ring [see the $M^+/(M-H)^+$ peak ratio (Table 2)].

Thus the mass spectra of pyridyl- and quinolylbenzimidazoles VI-IX and XII, as well as those of dibenzimidazolyls X and XI, confirmed the existence of conjugation of the heterorings in their molecules and the double-bond character of the bond between them, which is not cleaved under the influence of electron impact, whereas this bond is not cleaved in quaternary salts III and V. In this case bases VI-XII, like quinolylindoles [3], should exist in the hindered cis or trans form, the preferred realization of which is determined by steric and electronic factors, as well as the possibility of the formation of an intramolecular hydrogen bond [11]. To verify this assumption we measured the dipole moments of VI-X and XII (Table 3). As a consequence of the low solubilities of the benzimidazole derivatives in ordinary nonpolar solvents, the measurements of the dielectric permeabilities were made in dioxane solutions at 30±1° by the method in [12]. The dipole moments were calculated from the formula $\mu = 0.233\sqrt{\rho_\infty - \rho_e}$.

Electronic polarization ρ_e was assumed to be equal to the refraction and was calculated from the sum of the refractions of the bonds and groups [13]. The atomic polarization was not taken into account. The polarization at infinite dilution (ρ_∞) was calculated by the method in [14]. The dielectric permeability of dioxane was determined with respect to benzene [15] and was found to be 2.200. The magnitudes and directions of the group dipole moments were taken from [16-18]. We assumed that when the hydrogen atom attached to the pyr-

TABLE 3. Data from the Determination of the Dipole Moments in VI-X and XII

Compound	Concn. range ($c \cdot 10^4$ mole fraction)	α_{av}	β_{av}	ρ_e	ρ_∞	μ, D		φ*
						found	calc.	
VI	6,42—10,23	6,459	0,966	57,20	239,13	3,00		133°
VII	2,92—6,94	7,687	1,497	57,20	265,70	3,22		127°
VIII	4,64—5,05	10,879	0,554	57,20	391,89	4,08	4,30 (trans)	—
IX	1,35—4,31	6,117	−9,895	76,17	513,33	4,66		87°
X	2,36—4,02	4,480	3,709	77,18	123,71	1,52		159°
XII	3,04—7,39	7,520	0,642	71,32	295,32	3,34	4,09 (trans)	124°

*The angle of twisting of the rings relative to the orientation of the two hetaryls with a cis configuration of the "pyrrole" and "pyridine" nitrogen atoms of the different rings.

TABLE 4. Resonance Energies of the cis, trans, and Transition Forms of 2,2-Diquinolyl and 2-(2-Quinolyl)benzimidazole

Compound	E, kcal/mole
2,2-Diquinolyl (cis form)	2081,32
2,2-Diquinolyl (trans form)	2080,67
2,2-Diquinolyl (transition form)	2068,47
2-(2-Quinolyl)benzimidazole (cis form)	2016,19
2-(2-Quinolyl)benzimidazole (trans form)	2015,95
2-(2-Quinolyl)benzimidazole (transition form)	1997,34

role nitrogen atom of benzimidazole is replaced by a CH_3 group, the direction of the moment does not change, since the methyl group displays electron-donor properties, and only its magnitude changes. It is apparent from Table 3 that all of the investigated compounds except for VIII exist in solution in dioxane as nonplanar conformers that approach the s-trans forms. The absence of an effect of an intramolecular hydrogen bond in VI, VII, and XII on the stabilization of the cis forms is evidently explained by the formation of an intermolecular hydrogen bond with the dioxane molecules. The fact that the angle of twisting relative to the plane of the cis form is much larger for these compounds than for IX is also explained by the same reason. The solvate of larger volume than the CH_3 group that is formed in these cases probably leads to great pushing apart in IX.

Using the self-consistent-field (SCF) method with allowance for the coulombic repulsion of the electrons we calculated the π-electron distribution and bond orders in VIII and XII and, in addition, the resonance energies of the cis and trans conformations of 2-(2-quinolyl)benzimidazole XII and, for comparison, 2,2'-diquinolyl, as well as the energies of the transition state between these forms, in which the rings are mutually perpendicular. The parameters of the nitrogen atom and the method of calculation were assumed to be the same as in [19]. It is apparent from Table 4 that the differences in the resonance energies of the cis and trans forms (0.24 and 0.65 kcal/mole) are insignificant; this is in agreement with the literature data for other dihetaryls [5] and does not exceed the limits of the error in the computational method itself, whereas the barrier to transition from one form to the other proved to be extremely significant (18.6 and 12.2 kcal/mole, respectively). The barriers to rotation of the hetaryls in pyridylbenzimidazoles found experimentally by means of the PMR spectra do not exceed 4-5 kcal/mole [2]. The distribution of the π-electron charges in the quinolylbenzimidazole molecule (see Fig. 1) confirms our conclusion regarding the initial direction of alkylation during the formation of its quaternary salts in the imidazole ring, since the electron density on the "pyridine" nitrogen atom of the imidazole ring is higher than that on the pyridine nitrogen atom.

We assumed that deprotonation to give anhydro bases, as in the case of 4-indolylpyridinium [20] and 2-indolylquinolinium [21] salts, would occur under the influence of alkali on

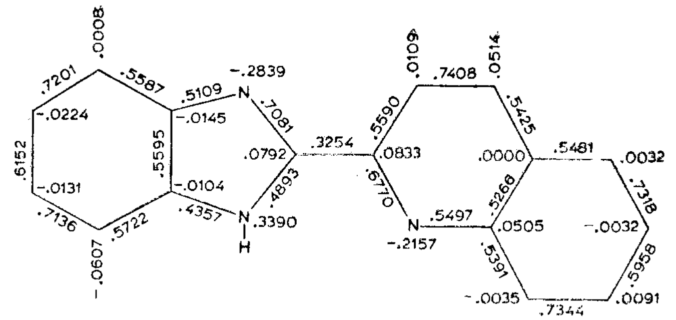
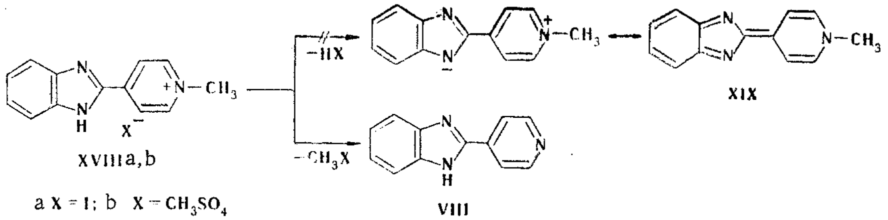


Fig. 1. Molecular diagram of 2-(2-quinolyl)-benzimidazole.

benzimidazolylpyridinium quaternary salts. However, it was found that salts XVIII unusually readily split out a methyl group to give base VIII rather than anhydro base XIX when they are treated with an alcohol solution of alkali:



EXPERIMENTAL

The mass spectra were obtained with a Varian Mat-311 spectrometer at an accelerating voltage of 70 eV and a cathode emission current of 300 μA (with direct introduction of the samples into the ion source at 70°C). The dipole moments were measured with an E 12-1 dielcometer with an oscillographic zero indicator. The IR spectra of saturated solutions of the compounds in chloroform were recorded with a UR-20 spectrometer. Chromatography was carried out on Al_2O_3 with development with UV light or iodine vapors. The PMR spectra of solutions in trifluoroacetic acid were recorded with a BS-467 spectrometer with tetramethylsilane as the internal standard. The UV spectra of $2 \cdot 10^{-5}$ solutions of the compounds in aqueous ethanol were recorded with a Specord spectrophotometer.

1,3-Dimethyl-2-(2-quinolyl)benzimidazolium Iodide (IIIa). A solution of 2.45 g (0.01 mole) of 2-(2-quinolyl)benzimidazole and 4.2 g (0.03 mole) of methyl iodide in 35 ml of DMF was refluxed for 8 h, after which it was cooled, and the resulting precipitate was recrystallized several times from methanol to give 2.09 g (52%) of a product with mp 282-284°C and R_f 0.81 (methanol). UV spectrum, λ_{max}(log ε) 206 (4.39), 246 (4.39), and 282 nm (4.02). Found: C 53.7; H 4.2; I 31.7; N 10.4%. $C_{18}H_{16}IN_3$. Calculated: C 53.9; H 4.0; I 31.6; N 10.5%. The methanol mother liquor was evaporated to give 1.6 g (30%) of 1,3-dimethyl-2-(1-methyl-2-quinolyl)benzimidazolium diiodide (V) with mp 240-245°C (isopropyl alcohol) and R_f 0.85 (methanol). Found: C 42.2; H 3.6; I 46.6; N 7.5%. $C_{19}H_{19}I_2N_3$. Calculated: C 42.0; H 3.5; I 46.7; N 7.7%.

Salts IIIb, c and XV-XVII (Table 1) were similarly obtained.

1-Methyl-2-(2-quinolyl)benzimidazole hydriodide (Ib) was similarly obtained by refluxing a solution of 2.45 g (0.01 mole) of 2-(2-quinolyl)benzimidazole and 1.4 g (0.01 mole) of methyl iodide in 25 ml of acetone for 18 h. Workup of the reaction mixture gave 2.6 g (70%) of a product with mp 170-171°C. UV spectrum, λ_{max}(log ε): 206 (4.44), 245 (4.44), and 285 nm (4.07). IR spectrum ($CHCl_3$): 3400-3500 cm^{-1} (NH). PMR spectrum, δ: 4.16 ppm (s, 3H, $N-CH_3$). Found: C 52.4; H 3.7; I 32.5; N 10.7%. $C_{17}H_{13}N_3 \cdot HI$. Calculated: C 52.7; H 3.7; I 32.8; N 10.8%

Salts Ia, c, XIIIa-e, XIVa, b, and XVIIIa, b (Table 1) were similarly obtained.

2-Methyl-4-(2-benzimidazolyl)quinoline. A mixture of 2.2 g (0.02 mole) of o-phenylenediamine and 1.8 g (0.01 mole) of 2-methylquinoline-4-carboxylic acid was heated for 6 h with 20 g of polyphosphoric acid (PPA) at 180-200°C. The precipitate was recrystallized several times from methanol to give 2.1 g (76%) of a product with mp 254-255°C and R_f 0.27 [benzene—hexane—chloroform (6:1:30:]. UV spectrum λ_{max}(log ε): 206.6 (4.31), 240 (4.28), and 323 nm (4.13). Found: C 73.4; H 5.3; N 15.1%. $C_{17}H_{13}N_3 \cdot H_2O$. Calculated: C 73.6; H 5.5; N 15.2%. The picrate had mp 233-235°C (isoamyl alcohol). Found: C 56.5; H 3.5; N 17.1%. $C_{17}H_{13}N_3 \cdot C_6H_3N_3O_7$. Calculated: C 56.6; H 3.3; N 17.2%.

A similar procedure was used to obtain 2-phenyl-4-(2-benzimidazolyl)quinoline [70% yield, mp 215-216°C, and R_f 0.28 (benzene—hexane—chloroform, 6:1:30). Found: C 77.7; H 5.2; N 12.3%. $C_{22}H_{15}N_3 \cdot H_2O$. Calculated: C 77.8; H 5.1; N 12.4%. The picrate had mp 249-250°C (isoamyl alcohol). Found: C 61.1; H 3.4; N 15.3%. $C_{22}H_{15}N_3 \cdot C_6H_3N_3O_7$. Calculated: C 61.1; H 3.3; N 15.3%], 2,2'di[4-(2-benzimidazolyl)quinolyl] [70% yield, mp>360°C (DMF). Found: N 16.3%. $C_{32}H_{20}N_6 \cdot 2H_2O$. Calculated: N 16.0%], and 1-methyl-2-(2-quinolyl)benzimidazole [90% yield, mp 132-134°C, and R_f 0.75 (benzene—hexane—chloroform, 6:1:30). Found: C 78.6; H 4.8; N 16.5%. $C_{17}H_{13}N_3$. Calculated: C 78.7; H 5.1; N 16.2%

Dimethylation of 4-(2-Benzimidazolyl)-1-methylpyridinium Iodide. A solution of 1.68 g (5 mmole) of 4-(2-benzimidazolyl)-1-methylpyridinium iodide in an aqueous alcohol solution of alkali (15g of KOH in 15 ml of water and 75 ml of ethanol) was allowed to stand for 5 h, after which it was poured into 200 ml of water, and the resulting precipitate was recrystallized from aqueous ethanol to give 0.47 g (45%) of 4-(2-benzimidazolyl)pyridine with mp 220-222°C [6] and R_f 0.87 (methanol). UV spectrum, λ_{max}(log ε): 207 (4.13) and 311 nm (4.12). Found: C 73.6; H 4.9; N 21.3%. $C_{13}H_{11}N_3$. Calculated: C 73.8; H 4.7; N 21.5%. No melting point depression was observed for a mixture of this product with a genuine sample, and the UV spectra of the two samples were similar.

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