REACTION OF INDOLES WITH ALDEHYDES. SYNTHESIS OF DIHYDROPYRROLO[3,4-b]INDOLES

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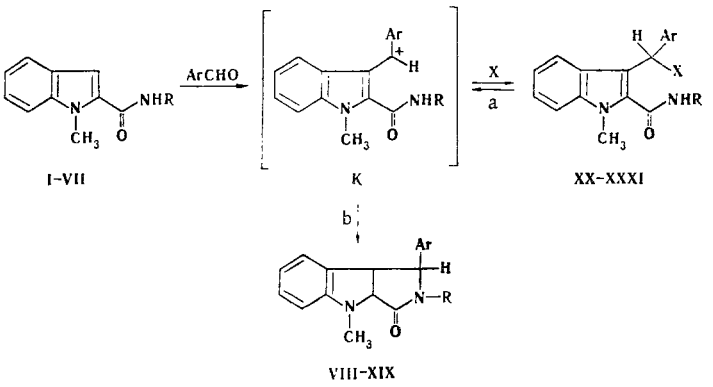
Aromatic aldehydes react with amides of 1-methylindole-2-carboxylic acid under acid catalysis conditions to give 1-aryl-4-methyldihydropyrrolo[3,4-b]indol-3-ones. The intermediate 1-methyl-2-CONHR-3(α-X-benzyl)indoles, which are subsequently converted to the indicated cyclic compounds, were isolated. o-Acetyl derivatives were obtained by the action of acetic anhydride on derivatives of unsubstituted amides. Dihydropyrrolo[3,4-b]indol-3-ones were reduced by $LiAlH_4$ to the corresponding dihydropyrrolo[3,4-b]indoles. A mechanism for the formation of dihydropyrrolo[3,4-b]indoles is proposed.

The synthesis of a heterocyclic system containing condensed indole and pyrrole rings was first accomplished by the Fischer reaction [1, 2]. We have previously obtained 2-benzylideneaminopyrrolo[3,4-b]indoles by construction of a pyrrole ring attached to indole by the action of aromatic aldehydes on indole-2-carboxylic acid hydrazide [3]. A mechanism for the reaction of indoles with aldehydes was later proposed in [4]; this mechanism indicates the possibility of the synthesis of three-ring indole-containing structures on the basis of amides and anilides and alkylindole-2-carboxylic acid. We synthesized a number of amides and anilides of 1-methylindole-2-carboxylic acid (Table 1), which were subjected to reaction with aromatic aldehydes having electron-donor and electron-acceptor substituents under acid catalysis conditions at 0°C. Aldehydes containing electron-donor substituents formed three-ring structures VIII-XI both with unsubstituted amide I and substituted amides II and IV. Aldehydes containing electron-acceptor substituents and unsubstituted benzaldehyde formed stable intermediate 1-methyl-2-CONHR-3-(α-X-benzyl)indoles (XX-XXXI).

When 1-methyl-2-carbanilido-3-(α-chlorobenzyl)indoles XXV-XXXI were heated, they underwent cyclization to 1,2-diphenyl-4-methyldihydropyrrolo[3,4-b]indol-3-ones (XIII-XIX). The existence of a strong electron-acceptor substituent ($p\text{-}NO_2$) in the phenyl ring leads to the production of "chloride" XXVI, which undergoes cyclization only in alcoholic alkali.

It is known that electrophilic attack of indole by aldehydes in the 3 position leads to the formation of (3-indolyl)phenylcarbinol, which is converted to (3-indolyl)phenylmethyl cation C in acidic media via the following scheme:



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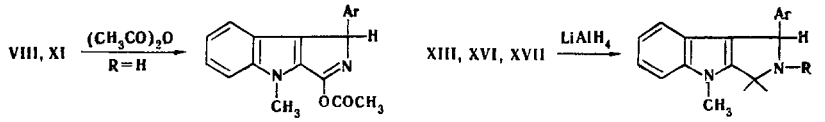


XXXII, XXXIII XXXIV-XXXVI

Carbonium ions C that have electron-acceptor substituents in the phenyl ring are extremely electrophilic and rapidly "capture" any nucleophile – chloride ion or an alcohol molecule – to give, respectively, chloro (XXV-XXXI) or alkoxy (XX-XXIV) derivatives in 1-2 min. In this case the reaction is kinetically controlled. When halides XXV-XXXI are heated in ethylene glycol to 150°, carbonium ion C is formed due to equilibrium *a* and a shift to favor the formation of three-ring structures due to the irreversibility of cyclization step b. The appearance of a characteristic color (λ_{max} 460 nm) when the chloro derivatives are heated and the disappearance of the color upon complete conversion to dihydropyrrolo[3,4-b]indoles constitute evidence for the formation of cation C.

Aldehydes that have electron-donor substituents form stable carbonium ion C, which is slowly (10-12 h) converted to three-ring compounds VIII-XII (thermodynamic control). The decrease in the nucleophilicity of the amide nitrogen atom* by conversion to an anilide leads to subsequent slowing down of the cyclization and predominance of an intermolecular reaction to give chloro derivatives XXV-XXXI.

In contrast to 1-methylindole-2-carboxylic acid, where the formation of products of condensation of 2 moles of indole with 1 mole of aldehyde was frequently observed, the amides of this acid are less inclined to undergo condensation. We noted instances of the formation of bis(1-methyl-2-carboxyisopropylamido-3-indolyl)-phenylmethanes (XXXVII, XXXVIII); this is evidently associated with steric hindrance during attack on the carbonium center by the amide nitrogen atom. Bis(1-methyl-2-anilidocarbonyl)phenylmethane (XXXIX) was isolated as an impurity in the preparation of cyclic derivative XIX.

The IR spectra of dihydropyrrolo[3,4-b]indol-3-ones (Table 2) differ from the spectra of the starting amides (Table 1) with respect to higher frequencies of the carbonyl absorption ($\Delta\nu_{CO}$ 20-40 cm^{-1}); this is due to the formation of the five-membered ring of a lactam structure. The UV spectra change only slightly on passing from amide I and anilides IV-VII to cyclic compounds VIII-XII and XIII-XIX, respectively, and a bathochromic shift of 8-10 nm is observed for all of the structures. The derivatives of anilides XIII-XIX have a maximum at 308-313 nm, and, from a comparison with λ_{max} 300 nm for derivatives of amide VIII-XII, it can be concluded that the phenyl group in the 2 position of the dihydropyrrole ring participates in conjugation with the 2-indoyl system, while the phenyl group in the 1 position is isolated and does not affect the form of the electronic spectra.

The PMR spectra of 1-phenyl-2-R^2-dihydropyrrolo[3,4-b]indol-3-ones (Table 2) prove the presence of a methyl group in the indole ring ($N_{(4)}-CH_3$ 3.9 ppm), of a methyl group in the phenyl ring (3.67 ppm), and of a lone benzyl proton in the 1 position (5.8 ppm).

1-Phenyl-4-methyldihydropyrrolo[3,4-b]indol-3-ones (VIII, XI) form 3-acetoxy derivatives XXXII and XXXIII in satisfactory yields (O-acylation) on treatment with refluxing acetic anhydride; the IR spectra of the products contain a band at 1720 cm^{-1} (stretching vibrations of an ester group), and the PMR spectrum of XXXII shows the appearance of a singlet of a methyl group at strong field (2.56 ppm).

The carbonyl group of the dihydropyrrole fragment of VIII-XIX cannot be reduced by the usual method, and only the use of dioxane as the solvent and an increase in the temperature to 100° make it possible to obtain dihydropyrrolo[3,4-b]indoles (XXXIV-XXXVI). 2-Phenyl-substituted XXXVI and XXXV were isolated in the form of bases, and only the single derivative XXXIV gave an analytically pure hydrochloride, whereas the other bases formed colored uncrystallizable oils when they were treated with ether or alcohol solutions of hydrogen chloride.

The fact of the reduction of the carbonyl group was confirmed by the disappearance of absorption at 1600-1700 cm^{-1} in the IR spectrum and a hypsochromic shift of the maximum in the UV spectrum from 309 to 275 nm; this is characteristic for transition from indoles containing a carbonyl group in the 2 position to indoles with substituents that do not participate in conjugation with the indole system (Fig. 1).

1-Phenyldihydropyrrolo[3,4-b]indoles XXXIV-XXXVI are less stable with respect to strong acids than the 1-unsubstituted derivatives [1] as a consequence of the case of cleavage of the $C_{(1)}-N$ bond, which leads to the

*1-Methylindole-2-carboxamide is sufficiently basic and forms crystalline salts in ether or acetic acid containing hydrogen halide.

TABLE 1. 1-Methylindole-2-carboxylic Acid Amides

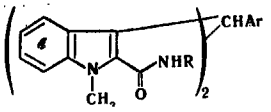
Compound	R	mp, °C	Empirical formula	N, % found	N, % calc.	UV spectrum, λmax, (log ε), nm	IR spectrum, cm⁻¹ ν_{CO}	IR spectrum, cm⁻¹ ν_{NH}	Yield, %
I	H	166	$C_{10}H_{10}N_2O$	16,0	16,1	293 (4,20)	1662	3190 3390	90
II	CH_3	105	$C_{11}H_{12}N_2O$	14,7	14,9		1645	3330	80
III	$CH(CH_3)_2$	160—162	$C_{13}H_{16}N_2O$	12,9	12,9		1638	3265	90
IV	C_6H_5	166	$C_{16}H_{14}N_2O$	11,2	11,2	304 (4,41)	1660	3335	90
V	p-$CH_3C_6H_4$	183	$C_{17}H_{16}N_2O$	10,5	10,6		1643	3270	90
VI	p-$CH_3OC_6H_4$	154—155	$C_{17}H_{16}N_2O_2$	10,1	10,0		1645	3243 3265	90
VII*	p-ClC_6H_4	217—218	$C_{16}H_{13}ClN_2O$	9,9	9,8		1650	3310	90

*Found: Cl 12.6%. Calculated: Cl 12.5%.

TABLE 2. 1-Aryl-2-R-4-methyl-1,2-dihydropyrrolo[3,4-b]indol-3-ones

Compound	Ar	R	mp, °C	Empirical formula	Found, % C	Found, % H	Found, % Cl	Found, % N	Calculated, % C	Calculated, % H	Calculated, % Cl	Calculated, % N	ν_{CO}, cm⁻¹	PMR spectrum, ppm	Yield, %
VIII	p-$CH_3OC_6H_4$	H	221—222	$C_{18}H_{16}N_2O_2$	74,4	5,5	—	9,5	74,0	5,5	—	9,6	1700	8,22—7,50 (m,8H, aromatic protons) 5,84 (s, 1H) 3,97 (s, 3H, $N_{(4)}$—CH_3) 3,67 (s, 3H, OCH_3)	72
IX	3-CH_3O, 4-OHC_6H_3	H	264—265	$C_{18}H_{16}N_2O_3$	70,3	5,2	—	9,0	70,1	5,2	—	9,1	1695		75
X	p-HOC_6H_4	H	286—287	$C_{17}H_{14}N_2O_2$	73,3	5,0	—	10,1	73,4	5,0	—	10,1	1685		70
XI	3,4-(—OCH_2O)—C_6H_3	H	227—229	$C_{18}H_{14}N_2O_3$	70,7	4,6	—	9,1	70,6	4,6	—	9,2	1708		75
XII	p-$CH_3OC_6H_4$	CH_3	149—150	$C_{19}H_{18}N_2O_2$	74,7	6,0	—	9,1	74,5	5,9	—	9,2	1692		72
XIII	C_6H_5	C_6H_5	198—200	$C_{23}H_{18}N_2O$	81,5	5,3	—	8,2	81,6	5,3	—	8,3	1680	7,32—7,17 (m,14H, aromatic protons) 5,93 (s, 1H) 3,95 (s, 3H, $N_{(4)}$—CH_3)	93
XIV	o-ClC_6H_4	C_6H_5	188—190	$C_{23}H_{17}ClN_2O$	74,0	4,6	9,6	7,5	74,1	4,6	9,5	7,5	1690		92
XV	p-$CH_3OC_6H_4$	C_6H_5	165—167	$C_{24}H_{20}N_2O_2$	78,5	5,4	—	7,5	78,3	5,4	—	7,6	1700	7,25—7,16 (m,13H, aromatic protons) 5,8 (s, 1H) 3,87 (s, 3H, $N_{(4)}$—CH_3) 3,63 (s, 3H, OCH_3)	79
XVI	o-ClC_6H_4	p-$CH_3C_6H_4$	170	$C_{24}H_{19}ClN_2O$	74,4	4,9	9,3	7,3	74,5	4,9	9,2	7,2	1610		79
XVII	C_6H_5	p-$CH_3C_6H_4$	182—184	$C_{24}H_{20}N_2O$	81,6	5,4	—	8,0	81,8	5,7	—	7,9	1610		85
XVIII	C_6H_5	p-$CH_3OC_6H_4$	144—146	$C_{24}H_{20}N_2O_2$	78,3	5,3	—	7,6	78,3	5,4	—	7,6	1680	7,25—7,15 (m,13H, aromatic protons) 5,8 (s, 1H) 3,9 (s, 3H, $N_{(4)}$—CH_3) 3,67 (s, 3H, CH_3O)	80
XIX	C_6H_5	p-ClC_6H_4	190—192	$C_{23}H_{17}ClN_2O$	74,0	4,6	9,5	7,5	74,1	4,6	9,5	7,5	1705		60

TABLE 3. Bis(1-methyl-2-carboxamido-3-indolyl)phenylmethanes



Compound	Ar	R	mp, °C	Empirical formula
XXXVII	C_6H_5	$CH(CH_3)_2$	218—220	$C_{33}H_{36}N_4O_2$
XXXVIII	p-$CH_3OC_6H_4$	$CH(CH_3)_2$	272	$C_{34}H_{38}N_4O_3$
XXXIX	C_6H_5	p-ClC_6H_4	282	$C_{39}H_{30}Cl_2N_4O_2$

TABLE 3 (cont.)

Compound	Found, %				Calculated, %				ν_{CO}, cm^{-1}	Yield, %
	C	H	Cl	N	C	H	Cl	N		
XXXVII	75,9	7,0	—	10,5	76,2	6,9	—	10,8	1630	80
XXXVIII	74,0	7,0	—	10,1	74,2	6,9	—	10,2	1635	80
XXXIX	71,3	4,6	10,9	8,4	71,2	4,6	10,8	8,5	1655	30

TABLE 4. 1-Methyl-2-carboxamido-3-(α-X-benzyl)indoles

Compound	Ar	R	X	mp, °C	Empirical formula	Found, %		Calculated, %		PMR spectrum, ppm	Yield, %
						Cl	N	Cl	N		
XX	C_6H_5	H	OC_2H_5	215	$C_{19}H_{20}N_2O_2$	—	8,9	—	9,1	7,22—7,15 (m, 9H, aromatic protons) 6,1 (s, 1H) 3,88 (s, 3H, N—CH_3) 3,4 (q, 2H, CH_2 from C_2H_5) 1,17 (t, 3H, CH_2 from C_2H_5)	68
XXI	p-$NO_2C_6H_4$	H	OCH_3	180—181	$C_{18}H_{17}N_3O_4$	—	12,3	—	12,4	8,1—7,33 (m, 8H, aromatic protons) 6,07 (s, 1H) 3,9 (s, 3H, N—CH_3) 3,44 (s, 3H, OCH_3)	70
XXII	p-$NO_2C_6H_4$	H	OC_2H_5	203—204	$C_{19}H_{19}N_3O_4$	—	11,9	—	11,9	8,66—7,33 (m, 8H, aromatic protons) 6,13 (s, 1H) 4,33 (s, 3H, N—CH_3) 4,01 (q, 2H, CH_3 from C_2H_5) 1,33 (t, 3H, CH_2 from C_2H_5)	70
XXIII	C_6H_5	CH_3	OCH_3	126—127	$C_{19}H_{20}N_2O_2$	—	13,5	—	13,6	7,25—7,1 (m, 9H, aromatic protons) 6,27 (s, 1H) 3,42 (s, 3H, N—CH_3) 2,30 (s, 3H, $NHCH_3$) 2,24 (s, 3H, OCH_3)	75
XXIV	C_6H_5	CH_3	OC_2H_5	142—143	$C_{20}H_{22}N_2O_2$	—	13,4	—	13,4	7,05—6,83 (m, 9H, aromatic protons) 5,56 (s, 1H) 3,57 (s, 3H, N—CH_3) 2,7 (s, 3H, $NHCH_3$) 4,07 (q, 2H, CH_2 from C_2H_5) 0,97 (t, 3H, CH_3 from C_2H_5)	75
XXV	C_6H_5	C_6H_5	Cl	149—151	$C_{23}H_{19}ClN_2O$	9,4	7,5	9,5	7,5		77
XXVI	p-$NO_2C_6H_4$	C_6H_5	Cl	145—146	$C_{23}H_{18}ClN_3O_3$	8,3	10,1	8,5	10,0		85
XXVII	o-ClC_6H_4	C_6H_5	Cl	163—165	$C_{23}H_{18}Cl_2N_2O$	17,4	6,7	17,4	6,8		80
XXVIII	C_6H_5	p-$CH_3C_6H_4$	Cl	154	$C_{24}H_{21}ClN_2O$	9,2	7,1	9,1	7,2		77
XXIX	C_6H_5	p-$CH_3OC_6H_4$	Cl	148—150	$C_{24}H_{21}ClN_2O_2$	8,7	7,0	8,8	6,9	7,2—7,1 (m, 13H, aromatic protons) 5,9 (s, 1H) 3,9 (s, 3H, $N_{(3)}$—CH_3) 3,6 (s, 3H, OCH_3)	70
XXX	C_6H_5	p-ClC_6H_4	Cl	145—146	$C_{23}H_{18}Cl_2N_2O$	17,5	6,9	17,4	6,8		70
XXXI	o-ClC_6H_4	p-$CH_3C_6H_4$	Cl	148	$C_{24}H_{20}Cl_2N_2O$	16,8	6,5	16,8	6,6		

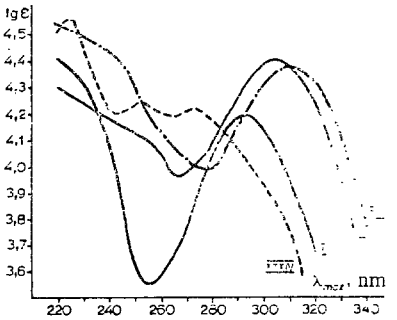
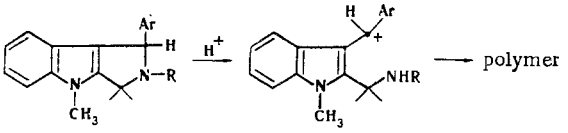


Fig. 1. UV spectra: 1-methyl-2-carboxamidoindole (I), 1-methyl-2-carboxanilidoindole (IV), 1,2-diphenyl-4-methyl-1,-2-dihydropyrrolo[3,4-b]indol-3-one (XIII), and 1,2-diphenyl-4-methyldihydropyrrolo[3,4-b]-indole (XXXIV) in ethanol.



(3-indolyl)phenylmethyl cation [4] and, evidently, its subsequent polymerization due to the presence in the molecule of two functional groups – a carbonium center and an amino group.

The ability of 3-(α-methylaminobenzyl)indole to undergo cleavage of the $C_{(1)}-N$ bond was recently used for the synthesis of (3-indolyl)phenylacetonitrile [5].

EXPERIMENTAL

The UV spectra of $0.2-0.3 \cdot 10^{-4}$ M solutions of the compounds in ethanol were recorded with an SF-16 spectrophotometer. The IR spectra of mineral oil suspensions of the compounds were recorded with a UR-22 spectrometer. The PMR spectra of trifluoroacetic acid and nitrobenzene solutions of the compounds containing hexamethyldisiloxane as the internal standard were recorded with a spectrometer with an operating frequency of 60 MHz. The purity of the isolated products was verified by thin-layer chromatography (TLC) on Silufol with a luminophore coating in a cyclohexane–ethyl acetate system (3 : 1).

The amides and anilides of 1-methylindole-2-carboxylic acid were synthesized by the method in [6] by the action of ammonia, methylamine, isopropylamine, aniline, or substituted aniline on the acid chloride obtained by reaction of 1-methylindole-2-carboxylic acid with phosphorus pentachloride.

1-(p-Methoxyphenyl)-4-methyl-1,2-dihydropyrrolo[3,4-b]indol-3-one (VIII). A 10-ml sample of acetic acid saturated with hydrogen bromide was added with cooling (ice water) to a mixture of 1.74 g (0.01 mole) of 1-methyl-2-carboxamidoindole and 1.36 g (0.01 mole) of anisaldehyde. The components gradually dissolved. The solution was cooled for 30 min, after which it was allowed to stand at room temperature for 15 h. The reaction mass was washed three times with hexane-ether (1 : 1) and twice with hexane, after which it was triturated in ethanol. The resulting crystals were removed by filtration and purified by crystallization from ethanol to give 2.1 g (72%) of VIII.

Compounds IX-XII and XV were similarly synthesized: IX-XI were synthesized from the appropriate aldehyde and 1-methyl-2-carboxamidoindole, XII was synthesized from anisaldehyde and 1-methyl-2-carboxymethylamidoindole, and XV was synthesized from anisaldehyde and 1-methyl-2-carboxyanilidoindole.

1-Methyl-2-carboxanilido-3-(α-chlorobenzyl)indoles (XXV). A 20-ml sample of ether saturated with hydrogen chloride (19 g of HCl per 100 g of ether) was added with cooling (ice water) to a mixture of 2.5 g (0.01 mole) of 1-methyl-2-carboxanilidoindole and 2.23 g (0.021 mole) of benzaldehyde. The components dissolved. After 2-3 min, XXV precipitated from the solution, and was removed by filtration, washed with ether, and crystallized from benzene to give 2.88 g (77%) of XXV.

Compounds XXVI-XXXI were similarly synthesized from the appropriate aldehyde and anilide.

1,2-Diphenyl-4-methyl-1,2-dihydropyrrolo[3,4-b]indol-3-one (XIII). A 3.7-g (0.01 mole) sample of XXV was heated in 30 ml of ethylene glycol at 150° for 40 min. After 20 min, the pyrroloindolone began to precipitate from the solution. The cooled mixture was diluted with 20 ml of water, and the resulting precipitate was removed by filtration and crystallized from ethanol to give 3.1 g (92%) of XIII.

Compounds XIV and XVI-XIX were similarly synthesized from the appropriate 3-(α-chlorobenzyl) derivatives.

1-Methyl-2-carboxamido-3-(α-ethoxybenzyl)indole (XX). A 10-ml sample of acetic acid saturated with hydrogen bromide was added with cooling (ice water) to a mixture of 1.74 g (0.01 mole) of 1-methyl-2-carboxamidoindole and 1.06 g (0.01 mole) of benzaldehyde, and the solution was cooled for 30 min. Ether was then added, and the liberated oil was washed three times with hexane-ether (1 : 1). The resulting oily residue was dissolved in 20 ml of ethanol, and the solution was allowed to stand at room temperature for 30 min. The mixture was neutralized with 10% NaOH solution, and the resulting precipitate was removed by filtration, washed with water, and crystallized from ethanol to give 2 g (68%) of XX.

Compounds XXI-XXII were similarly obtained from 1-methyl-2-carboxamidoindole and p-nitrobenzaldehyde; XXIII-XXIV were obtained from 1-methyl-2-carboxyamidomethylaminoindole and benzaldehyde.

1-(p-Methoxyphenyl)-3-acetoxy-4-methyl-1,2-dihydropyrrolo[3,4-b]indole (XXXII). A 2.92-g (0.01 mole) sample of VIII was refluxed in 15 ml of acetic anhydride for 2 h, after which the mixture was cooled and treated with 20 ml of water. The crystals that formed in 10-15 min were removed by filtration and recrystallized from ethanol to give 1.95 g (58%) of XXXII with mp 182°. IR spectrum: 1692 (C=N), 1720 cm^{-1} (CO). Found: C 72.0; H 5.4; N 8.5%. $C_{20}H_{18}N_2O_3$. Calculated: C 71.8; H 5.4; N 8.4%.

1-(3,4-Methylenedioxyphenyl)-3-acetoxy-4-methyl-1,2-dihydropyrrolo[3,4-b]indole (XXXIII). This compound, with mp 175°, was obtained in 60% yield by the method used to synthesize XXXII. IR spectrum: 1695 (C=N), 1720 cm^{-1} (CO). Found: C 68.9; H 5.0; N 8.1%. $C_{20}H_{17}N_2O_4$. Calculated: C 68.8; H 4.9; N 8.0%.

1,2-Diphenyl-4-methyldihydropyrrolo[3,4-b]indole (XXXIV). A solution of 0.3 g (0.0065 mole) of XIII in 10 ml of absolute dioxane was added to a suspension of 0.07 g (0.02 mole) of $LiAlH_4$ in 15 ml of absolute ether, after which the mixture was stirred and the ether was removed by distillation as dioxane was simultaneously added dropwise. After the temperature of the vapors had reached 98-100°, the mixture was maintained at this temperature for 4 h. It was then cooled and treated with 1.5-2.0 ml of water until a lumpy precipitate formed. The dioxane layer was decanted and dried with Na_2SO_4, and ether satured with HCl was added carefully to it dropwise. The resulting precipitate of the hydrochloride of XXXIV was removed by filtration to give 0.13 g (46%) of a product with mp 232°. Found: Cl 9.8; N 7.1%. $C_{23}H_{20}N_2 \cdot HCl$. Calculated: Cl 9.9; N 7.8%. UV spectrum, λ_{max} (ε): 225 (38500), 252 (17800), 273 nm (17100).

The reduction of XXXV and XXXVI was carried out similarly, but after preparation of the dioxane solution the product was isolated in the form of the base by the addition of 50 ml of water. The liberated oil began to crystallize on trituration and was purified by crystallization from ethanol.

1-Phenyl-2-(p-tolyl)-4-methyldihydropyrrolo[3,4-b]indole (XXXV). This compound, with mp 168°, was obtained in 60% yield. Found: N 8.1%. $C_{24}H_{22}N_2$. Calculated: N 8.3%. UV spectrum, λ_{max} (ε): 225 (53200), 255 (38500), 272 nm (19400).

1-(o-Chlorophenyl)-2-(p-tolyl)-4-methyldihydropyrrolo[3,4-b]indole (XXXVI). This compound, with mp 112°, was obtained in 43% yield. Found: Cl 9.1; N 7.3%. $C_{24}H_{21}N_2Cl$. Calculated: Cl 9.6; N 7.5%. UV spectrum, λ_{max} (ε): 225 (50000), 255 (31000), 270 nm (23500).

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