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EFFECT OF PROTECTION OF AMINO GROUP ON DIRECTION OF REACTION OF 2-AMINOINDOLE WITH α,β-UNSATURATED KETONES

R. S. Sagitullin, T. Yagodzin'ski,
A. N. Kost, and V. V. Men'shikov

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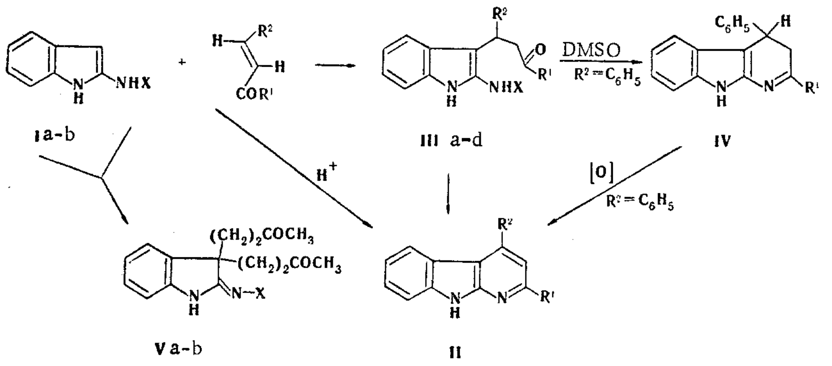
The electron-acceptor properties of arylsulfonyl and carbobenzoxy groups decrease the probability of primary electrophilic attack at the nitrogen atom. They also determine primary attack at the 3-C atom of the indole ring. Michael addition products, 3,4-dihydro-α-carbolines, and α-carbolines themselves are obtained, depending on the reaction conditions. The reaction with α,β-unsaturated ketones proceeds under milder conditions in the case of 2-benzyloxycarbonylaminoindole.

The 2-aminoindole molecule has three interrelated nucleophilic centers, and two different pairs of these centers (both nitrogen atoms or the nitrogen atom of the amino group and the 3 position of indole) consequently react with 1,3-difunctional compounds, depending on the conditions, to give pyrimidoindole or α-carboline structures [1]. However, if the amino group is protected with a tosyl or carbobenzoxy grouping, the analogous cyclization with 1,3-dioxo compounds proceeds with detachment of the protective group, and only α-carbolines are formed [2, 3]. Intermediate addition products and substances whose synthesis proceeds with involvement of the pyrrole imino group cannot be detected.

We have found that the corresponding α-carbolines (II) are also formed in the condensation of 2-carbobenzoxyaminoindole (Ia) with α,β-unsaturated ketones (in ethanol containing hydrobromic acid), although the process takes place in a considerably more complex manner in some cases. If benzalacetophenone or benzalacetone participates in the condensation, the reaction requires prolonged heating, and the carbolines are obtained in low yields; i.e., both the electrophilicity of the β-carbon atom of the double bond of the unsaturated ketone and the steric hindrance at this atom, which has a greater effect on attack at the 3-C atom, have a substantial effect on the process.



I, III, V a $X = COOCH_2C_6H_5$; b $X = SO_2C_6H_4CH_3$-*p*

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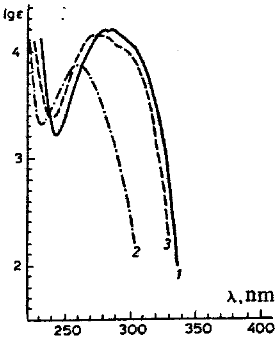


Fig. 1

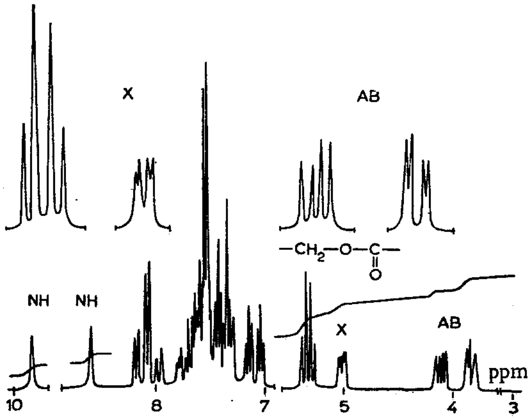


Fig. 2

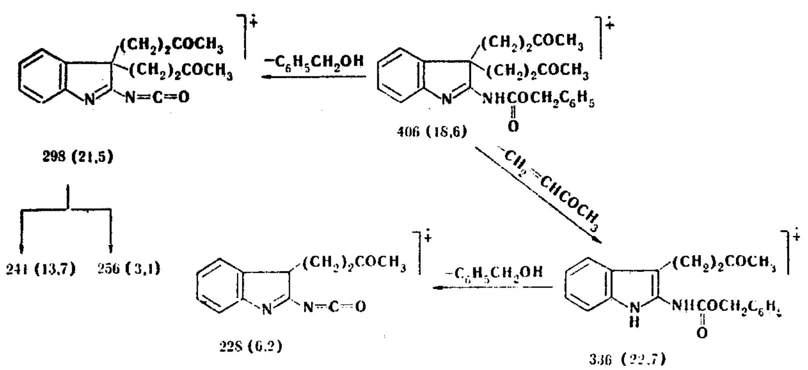
Fig. 1. UV spectra: 1) 3,3-bis(3-oxobutyl)-2-carbobenzoxyiminoindoline (Va) (in chloroform); 2) 1,3,3-trimethyl-2-iminoindoline hydrochloride (in water); 3) 3,3-bis(3-oxobutyl)-2-tosyliminoindoline (Vb) (in chloroform).

Fig. 2. PMR spectrum (in $CDCl_3$) of 3-(1,3-diphenyl-3-oxopropyl-2-carbobenzoxyaminoindole (IIId) with a Bruker-360 spectrometer.

However, in the case of methyl vinyl ketone, according to the mass-spectral data on the results of elementary analysis, the resulting Va contains residues of two methyl vinyl ketone molecules. Its IR spectrum contains an absorption band at 3260 cm^{-1}, which is characteristic for NH vibrations, and broad absorption bands of carbonyl groups (1670 and 1710 cm^{-1}). With respect to the character of the curve and the position of the absorption maxima, the UV spectrum of Va (Fig. 1) is similar to the spectrum of 1,3,3-trimethyl-2-iminoindoline [4]. The PMR spectrum (in $CDCl_3$) does not contain the signal of a proton attached to the 3-C atom but does contain a broad signal of an NH group at 10.86 ppm. The chemical shifts and the multiplicity of the signals of the methylene protons are characteristic for the proposed Va structure. The character of the mass-spectral fragmentation is also in agreement with this structure.

It is known [5] that oxindole also gives bis(ketoethylation) products at the 3-C atom in the Michael reaction.

However, these experiments did not give an unambiguous answer to the question of the trend of the process, since hydrolysis to the corresponding 2-aminoindole salt, which is known [6] to react with α,β-unsaturated ketones to give α-carbolines (after autoxidation), could take place initially in the presence of a strong acid. We therefore set up experiments with 2-tosylaminoindole (Ib), which is more resistant to hydrolysis; in this case ketones of the III type could be obtained, and Vb, similar to Va, could be obtained in the case of methyl vinyl ketone. The same substance (Vb) was obtained in the reaction of 1-diethylamino-3-butanone with amide Ib in the presence of sodium ethoxide.



In the case of amide Ia, by using substantially milder conditions (in acetic acid without heating) we also isolated products of the addition of one molecule of benzalacetophenone and benzalacetone; however, Va was isolated from the reaction of Ia with methyl vinyl ketone under these conditions.

TABLE 1. Intensities of the Principal Ions (in percent of J_{max})

Compound	R*	X	Fragments				
			$M^{+\cdot}$	F_1	F_2	F_3	F_2/F_1
IIIa	CH_3	Ts	36,8	27,7	34,8	23,7	1,3
IIIb	C_6H_5	Ts	14,2	19,0	20,9	11,1	1,1
IIIc	CH_3	$COOCH_2C_6H_5$	69,5	24,4	31,7	8,6	1,3
IIId	C_6H_5	$COOCH_2C_6H_5$	77,2	72	55,3	42	0,8

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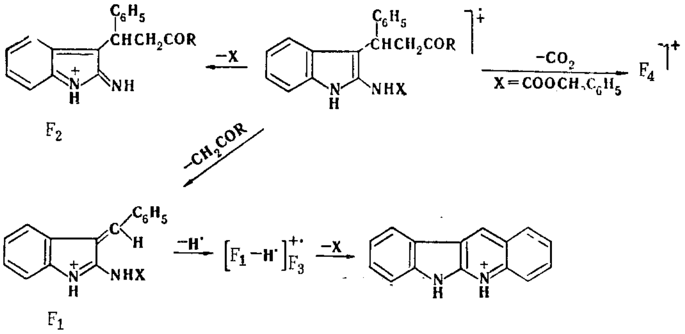
* $R^2 = C_6H_5$.

The UV spectra of IIIa-d in chloroform contain characteristic absorption bands at 280-285 and 292-296 nm. The IR spectra contain bands at 1690-1720 (C=O) and 3280-3300 and 3340-3380 cm^{-1} (NH); this is in agreement with the proposed structure. The character of the resonance of the aromatic protons in the PMR spectra was in agreement with the proposed structure. An unresolved multiplet was observed in the aliphatic region; however, we obtained the characteristic pattern of an ABX system (Fig. 2) with a Bruker spectrometer (360 MHz).

The molecular-ion peaks in the mass spectra of III have rather high intensities (16-70%). The mass spectra reflect competition in the initial fragmentation of the molecular ion via two pathways: cleavage of the β bond and detachment of a CH_2COR group to give ion peak F_1, as well as detachment of the protective group to give ion F_2. The probability of the detachment of one or the other group can be determined from the ratio of the intensities of the ion: $K = (M-X)^+/(M-CH_2COR)$. When $R = C_6H_5$, K = 1, and when $R = CH_3$, K = 1.3; i.e., fragmentation to give F_2 ions predominates for III ($R = CH_3$). The fact that the subsequent fragmentation of the resulting ions (F_3) is similar to the fragmentation of arylidene derivatives [8] additionally confirms structures IIIa-d (Table 1).



The definitive proof of structure III was conversion of IIIc,d to known α-carbolines. This heterocyclization proceeds well in dimethyl sulfoxide (DMSO), which has good dissolving, oxidizing, and dehydrating abilities. We simplified the latter process, in analogy with the described synthesis of pyrimidines [8], by the use of molecular sieves; this made it possible to raise the yields of the carbolines. One can carry out the condensation of an unsaturated ketone with amide Ia immediately in DMSO solution containing molecular sieves; the corresponding carbolines II are formed in good yields [for example, 2-methyl-4-phenyl-α-carboline (IIa, $R^1 = CH_3$, $R^2 = C_6H_5$) is formed from benzalacetone]. The yield depends on the temperature and the reaction time. The best result was obtained at 140-145°C after 7-8 h. When methyl vinyl ketone and ethylideneacetone were used, the synthesis was inefficient because of pronounced resinification. In the case of the reaction of amide Ia with benzalacetophenone (in DMSO in a stream of dry argon) we were able to isolate intermediate dihydrocarboline VI as a yellow crystalline substance that is quite stable in the solid state, does not fluoresce in UV light, and is identical to the compound described in [9]. Tosyl derivative Ib gives a difficult-to-separate mixture in the case of condensation with unsaturated ketones in DMSO.

Thus the electron-acceptor properties of the arylsulfonyl or carbobenzoxy group markedly decrease the probability of primary electrophilic attack at the nitrogen atom; this also predetermines the primary attack at the site of highest electron density – the 3-C atom. Cyclization and aromatization occur under more severe conditions. Primary attack at the 3-C atom under similar conditions was observed in the reaction of substituted 2-aminoindoles with other aldehydes or ketones [8, 10]. The ready cyclization of ketones of the III type with participation of the amide nitrogen atom and detachment of the protective group has been observed in a number of analogous cyclizations (for example, see [11]).

In the case of amide Ia the reaction with both β-diketones and with α,β-unsaturated ketones proceeds un-

TABLE 2. Compounds IIIa-d

Com-pound	R^{1}*	X	mp, °C (from ethanol)	Found, %			Empirical formula	Calculated, %			IR spectrum (mineral oil), cm^{-1}		PMR ($CDCl_3$), ppm		UV spectrum ($CHCl_3$)		Yield, %
				C	H	N		C	H	N	NH	C=O	$CH=CH_2$	NH	λ_{max}, nm	lg ε	
IIIa	CH_3	Ts	157—159	69,2	5,7	—	$C_{25}H_{24}N_2O_3S$	69,5	5,6	—	3270—3320	1715	3,1—6,1	9,1 10,0	280 294	4,12 3,98	51
IIIb	C_6H_5	Ts	205—207	72,9	5,3	5,4	$C_{30}H_{26}N_2O_2S$	72,9	5,3	5,7	3265—3390	1695	3,34—4,8	9,6 10,2	280 293	4,12 4,04	66
IIIc	CH_3	COOBz	129—132	75,8	5,9	6,8	$C_{26}H_{24}N_2O_3$	75,7	5,9	6,8	3300—3380	1720, 1690 1640	3,24—4,8	8,48 9,86	283 295	4,11 4,09	82
IIId	C_6H_5	COOBz	67—70	78,7	5,5	6,1	$C_{31}H_{26}N_2O_3$	78,5	5,5	5,8	—	1720 1680 1640	3,78—5,08	8,6 9,84	283 295	4,18 4,18	90

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* $R^2 = C_6H_5$.

TABLE 3. α-Carbolines*

Compound	R^1	R^2	mp, °C	Yield, %, by method A	B	C
IIa	CH_3	C_6H_5	230—231	2,0	62	67
IIb	C_6H_5	CH_3	190—192	0,5	—	—
IIc	C_6H_5	C_6H_5	222—224	14,0	75	70

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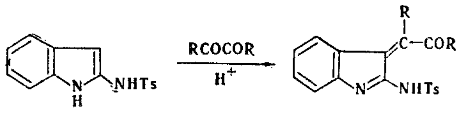
* The yields from indole Ia are given.

der milder conditions than in the case of tosyl derivative Ib; this is directly associated with the difference in the electron-acceptor character of the substituent attached to the amino group. The cyclization step also proceeds more readily in the case of Ia. However, in the case of arylsulfonamido derivative Ib cleavage of the N–S bond apparently occurs only in the aromatization step. This is also indicated by the fact that the principal products in the condensation of α-diketones with 2-arenesulfonamidoindoles of the Ib type were alkylidene (arylidene) derivatives VI.



VI a,b

VI a $R=CH_3$; b $R=C_6H_5$

EXPERIMENTAL

The IR spectra of mineral oil suspensions and chloroform solutions of the compounds were recorded with a UR-10 spectrometer. The UV spectra of chloroform solutions of the compounds were recorded with a Cary-15 spectrophotometer. The PMR spectra of deuterochloroform solutions of the compounds were recorded with Varian T-60 and XL-100 spectrometers with hexamethyldisiloxane as the internal standard. The mass spectra were recorded with an MKh-1303 spectrometer with a system for direct introduction of the samples into the ion source (at 75 eV and 200°C).

α-Carbolines IIa-c. A) A 4-mmole sample of 2-carbobenzoxyaminoindole (Ia) and 5 mmole of freshly distilled or recrystallized α,β-unsaturated ketone were refluxed in 2 ml of absolute ethanol and 1 ml of 45% HBr for 30 min to 7 h, after which the mixture was cooled and treated with a dilute aqueous solution of sodium hydroxide. The solution was extracted with chloroform, and the extract was dried with magnesium sulfate. The solvent was removed by vacuum evaporation, and the residue was purified preparatively on plates with activity II (Brockmann scale) aluminum [in a benzene–ethyl acetate system (3 : 1)] and recrystallized from benzene. The yields and constants of α-carbolines IIa-c are given in Table 3.

B) A mixture of 2 mmole of Ia and 2.2 mmole of freshly recrystallized benzalacetone was dissolved in 25 ml of dry DMSO, 4 g of molecular sieves was added, and the mixture was heated in a stream of argon at 140-145°C for 8 h. The solvent was partially removed by evaporation in a stream of argon, and the residue was cooled and diluted with water. The resulting precipitate was removed by filtration and washed with water. It was then dissolved in ether, and the ether solution was dried with magnesium sulfate. The ether was removed by distillation to give 2-methyl-4-phenyl-α-carboline, with mp 230-231°C (mp 230-232°C [1]), in 62% yield.

C) As in method B, IIIc,d were heated in DMSO, and the mixtures were worked up to give the corresponding α-carbolines. The yields and constants are given in Table 3.

Reaction of 2-Carbobenzoxyaminoindole with Benzalacetophenone. A 0.53-g (2 mmole) sample of amine Ia and 0.46 g (2.2 mmole) of benzalacetophenone were dissolved in 25 ml of dry DMSO, 4 g of molecular sieves (4 Å) was added, and the solution was heated in a stream of argon at 140-145°C for 9 h. The solvent was then partially removed by evaporation in vacuo in a stream of argon, and the residue was cooled and diluted with water. The resulting precipitate was removed by filtration, washed with water, and dissolved in ether. The ether solution was dried with magnesium sulfate, and the ether was removed by distillation to give 0.48 g (75%) of a mixture of **2,4-diphenyl-α-carboline** (IIc) and **2,4-diphenyl-3,4-dihydro-α-carboline** (IV). Preparative separation on plates with aluminum oxide [with a benzene–ethyl acetate system (15 : 1)] gave 0.40 g of IIc, with mp 222-224°C (from benzene), and 0.03 g of yellow crystals of IV (trituration with petroleum ether gave a product with mp 189-191°C). The products were identical to genuine samples with respect to their UV and IR spectra and melting points [9].

3,3-Bis(3-oxobutyl)-2-carbobenzoxyiminoindoline (Va). A) A mixture of 0.53 g (2 mmole) of amine Ia, 0.42 g (6 mmole) of freshly prepared methyl vinyl ketone, and 20 ml of ethanol containing 1-2 ml of hydrobromic acid was refluxed for 10-15 min, after which the resulting solution was cooled, and the alcohol was partially removed by evaporation. The concentrated solution was diluted with water and neutralized with 5% NaOH solution, and the precipitate was separated, washed with water, and air dried to give 0.58 g (71%) of Va with mp 169-170°C (from alcohol). UV spectrum ($CHCl_3$): λ_{max} 280, 285, and inflection at 294 nm (log ε 4.20, 4.19, and 4.11). IR spectrum (mineral oil): 1710 and 1670 (C=O); 3240-3260 cm^{-1} (NH). Found: C 71.0; H 6.6; N 6.7%. $C_{24}H_{26}N_2O_4$. Calculated: C 70.9; H 6.5; N 6.9%.

B) A mixture of 0.53 g (2 mmole) of Ia, 0.42 g (6 mmole) of methyl vinyl ketone, and 25 ml of glacial acetic acid was allowed to stand at 20°C for 24 h, after which the resulting solution was diluted with water, and sodium bicarbonate was added until a precipitate formed. The precipitate was separated, washed several times with water, and air dried to give 0.76 g (93%) of Va with mp 169-170°C (from alcohol).

3,3-Bis (3-oxobutyl)-2-tosyliminoindoline (Vb). A) A mixture of 0.5 g (1.75 mmole) of 2-tosylaminoindole (Ib), 0.35 g (5 mmole) of freshly prepared methyl vinyl ketone, and 6 ml of absolute alcohol containing 1 ml of hydrobromic acid was refluxed for 1 h, after which 2-3 ml of the solvent was removed by distillation, and the resulting precipitate was separated and washed with 2 ml of absolute alcohol to give 0.65 g (87%) of Vb with mp 159-161°C (from alcohol). UV spectrum ($CHCl_3$): λ_{max} 278 and shoulder at 294 nm (log ε 4.12 and 3.99). IR spectrum (in mineral oil): 1720 (C=O) and 3290 cm^{-1} (N−H). PMR spectrum ($CDCl_3$): 1.86-2.33 (14H, m, aliphatic protons), 2.46 (3H, s, CH_3), 7.1-8.1 (8H, m, aromatic protons), and 10.53 ppm (1H, s, NH). Mass spectrum, m/e: 426 [M$^{+\bullet}$, 4%], 356 [M−($CH_2 = CH_2-COCH_3$), 56%], 313 [356-$COCH_3$, 4%], 201 [356-Ts, 100%], 183 (18%), 159 (18%), 158 (21%), 91 (20%). Found: C 64.5; H 6.2%. $C_{23}H_{26}N_2O_4S$. Calculated: C 64.8; H 6.1%.

B) A mixture of 0.5 g (1.75 mmole) of Ib, 0.7 g (5 mmole) of 1-diethylamino-3-butanone, 10 ml of absolute alcohol, and 5 ml of sodium ethoxide was stirred at 50-70°C for 3 h, after which 10 ml of water was added, and the mixture was acidified with 10 ml of 5% hydrochloric acid. The resulting precipitate was removed by filtration and washed with alcohol to give 0.49 g (65%) of Vb with mp 159-161°C (from alcohol).

3-(1,3-Diphenyl-3-oxopropyl)-2-tosylaminoindole (IIIb). A mixture of 1 g (3.5 mmole) of 2-tosylaminoindole (Ib), 5 mmole of freshly crystallized benzalacetophenone, 20 ml of absolute alcohol, and 2-3 ml of hydrobromic acid was refluxed for 5-6 h, after which it was cooled, and the resulting precipitate was separated and washed with alcohol to give 1.12 g (66%) of IIIb, the constants of which are presented in Table 2.

Reaction of 2-Tosylaminoindole with Benzalacetone. A mixture of 1 g (3.5 mmole) of 2-tosylaminoindole (Ib), 5 mmole of freshly crystallized benzalacetone, 20 ml of absolute alcohol, and 2-3 ml of hydrobromic acid was refluxed for 5 h, after which it was cooled, 5-7 ml of water was added, and the resulting precipitate was removed by filtration and washed with 5 ml of alcohol to give 0.77 g (51%) of 3-(1-phenyl-3-oxobutyl)-2-tosylaminoindole (IIIa) with mp 157-159°C (from alcohol), the constants of which are presented in Table 2. The filtrate was made alkaline to pH 8-9 with 0.2 N NaOH solution and extracted with chloroform. The extract was dried with magnesium sulfate, the solvent was removed by vacuum distillation, and the residue was separated on plates with aluminum oxide [with a benzene–ethyl acetate system (3 : 1) to give 0.16 g (18%) of 2-methyl-4-phenyl-α-carboline with mp 230-231°C (from benzene) (mp 230-232°C [1]).

3-(1-Phenyl-3-oxobutyl)-2-carbobenzoxyaminoindole (IIIc) and 3-(1,3-Diphenyl-3-oxopropyl)-2-carbobenzoxyaminoindole (IIId). A mixture of 2 mmole of Ia, 2 mmole of freshly recrystallized benzalacetone (or benzalacetophenone), and 25 ml of glacial acetic acid was allowed to stand at 20°C for 2 days, after which the resulting solution was diluted with water and treated with sodium bicarbonate until a precipitate formed. The precipitate was separated, washed with water, and air dried. The yields and constants of IIIc,d are given in Table 2.

Reaction of 2-Carbobenzoxyaminoindole with Benzaldehyde. A mixture of 0.53 g (2 mmole) of indole Ia, 0.11 g (2.1 mmole) of freshly distilled benzaldehyde, and 20 ml of benzene was refluxed in the presence of a catalytic amount of acetic acid for 3 h, after which the benzene was removed by vacuum distillation, and the residue was separated on plates with aluminum oxide [in a benzene–ethyl acetate system (4 : 1)] to give 0.44 g (71%) of bis(2-carbobenzoxyaminoindolyl)phenylmethane with mp 161-162°C [benzene–heptane (1 : 1)]. IR spectrum (mineral oil): 1710 and 1640 cm^{-1} (C=O). UV spectrum ($CHCl_3$): λ_{max} 283, and 295 nm (log ε 4.40 and 4.39). Found: C 75.8; H 5.2; N 9.1%. $C_{39}H_{32}N_4O_2$. Calculated: C 75.5; H 5.2; N 9.0%.

3-(α-Acetylethylidene)-2-tosylaminoindolenine (VIa). A mixture of 0.5 g (1.7 mmole) of 2-tosylaminoindole (Ib), 0.2 g (2.1 mmole) of diacetyl, 10 ml of absolute alcohol, and 1-2 mole of hydrobromic acid was refluxed for 2 h, after which 5 ml of alcohol was removed by distillation, and the residual mixture was cooled. The resulting precipitate was separated and washed with 10 ml of 0.2 N NaOH solution and 50% alcohol to give 0.53 g (56%) of VIa with mp 160-162°C (from alcohol). UV spectrum ($CHCl_3$): λ_{max} 259, 165, 313, and 380 nm (log ε 4.42, 4.45, 3.67, and 3.51). IR spectrum (mineral oil): 1710 (C=O) and 3280 cm^{-1} (NH). PMR spectrum (CF_3COOH): 2.06 (3H, s), 2.33 (6H, s), and 7.23-8.1 ppm (8H, m, aromatic protons). Mass spectrum, m/e: 354 (M^+, 20%), 339 ($M-CH_3$, 5.6%), 311 ($M-COCH_3$, 14%), 199 (M–Ts, 37%), 157 (199-CH_2CO, 100%), 156 (199-$COCH_3$, 38%), 131 (16%). Found: C 64.3; H 5.3; N 7.7%. $C_{19}H_{18}N_2O_3S$. Calculated: C 64.5; H 5.1; N 7.9%.

3-(α-Benzoylbenzylidene)-2-tosylaminoindolenine (VIb). Similarly, heating 0.5 g (1.7 mmole) of 2-tosylaminoindole Ib and 0.42 g (2 mmole) of benzil for 5 h gave 0.11 g (13%) of VIb with mp 259-261°C (from alcohol). UV spectrum ($CHCl_3$): λ_{max} 267, 327, and 395 nm (log ε 4.43, 3.79, and 3.55). IR spectrum (mineral oil): 1680 (C=O) and 3280 cm^{-1} (NH). Mass spectrum, m/e: 478 ($M^{+\cdot}$, 4.7%), 373 (M–PhCO, 3.2%), 323 (M–Ts, 100%), 218 (323-PhCO, 4.1%), 166 (16%), 155 (34%), 105 (29%). Found: C 72.6; H 4.7%. $C_{29}H_{22}N_2O_3S$. Calculated: C 72.9; H 4.6%.

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