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SYNTHESIS OF 2,10,11-TRISUBSTITUTED INDOLO[3,2-*b*]QUINOLINES

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A new method has been developed for the synthesis of derivatives of indolo[3,2-b]quinolines-11 based on N-oxidization of 2-nitro-10-substituted indolo[3,2-b]quinolines with subsequent conversion of the mixtures obtained into 2-nitro-11-substituted indolo[3,2-b]quinolinones-11. A series of 2-nitro-11-substituted indolo[3,2-b]quinolines was prepared.

Keywords: N-acetylindoxyl, indolo[3,2-*b*]quinoline, the Wilsmeier complex, N-oxidation, the Zincke–König reaction.

Recently we have established a new synthesis for derivatives of indolo[3,2-*b*]quinolines, based on the reaction of N-acetylindoxyl (**1**) with aromatic amines with subsequent closure of the quinoline ring during the Wilsmeier reaction [1]. To obtain 11-functionalised derivatives of these systems ketone **1** was reacted with anthranilic acid derivatives and cyclization of the 3-*ortho*-carboxy(ethoxycarbonyl)phenylaminoindoles was brought about *via* either the ethoxycarbonyl or carboxyl group. In these cases indolo[3,2-*b*]quinolinones-11 were obtained but the yields of the oxo derivatives was not large and these methods are suitable for preparative work [1]. Since derivatives of these heterocyclic systems have high antitumour activity [2, 3], it seemed essential to develop a synthesis for this type of compound. In this report we have studied the possibility of obtaining 11-oxo- and other 11-substituted indolo[3,2-*b*]quinolines based on N-oxidation of the pyridine nitrogen atom and transformation of the N-oxide obtained into the corresponding pyridinone derivatives. In the first stage of the study 10-acetyl derivatives **2a** were chosen as starting materials, which were oxidized by hydrogen peroxide in acetic acid. It appeared that even during the oxidation the formed N-oxides **3a** underwent rearrangement and, to judge from its ^{1}H NMR spectrum, a second component of the mixture obtained was the indoloquinolone **4a**. Refluxing the mixture in acetic anhydride gave a mixture of the expected quinolone **4a** and the deacetylated quinolone **4b** (apparently formed during working up of the reaction mixture) in a 2:1 ratio.

To avoid the complex separation of the reaction products the mixture was subjected to alkaline hydrolysis as a result of which 2-nitroindolo[3,2-*b*]quinolinone-11 [**4b**] was obtained in 40% yield (based on the initial **2a**). Some problems associated with the preparation of compound **4b** are connected with the partial 10-N-deacetylation during the reaction (N-acylindoles are well known to have relatively low stability). As a result of this the N-unsubstituted indoloquinoline **2b** was isolated after N-oxidation and rearrangement. N-Oxidation with hydrogen peroxide in acetic acid gave a mixture of the N-oxide **3b** and the quinolone **4b**, which on short heating with acetic anhydride gave the indoloquinolone (**4b**) in 85% yield based on **2b** – prolonged refluxing gave partial acetylation. So a suitable preparative route has been discovered for the synthesis of the valuable 11-oxo derivatives of inolo[3,2-*b*]quinolines. 10-Substituted 11-oxoindoloquinolines have been

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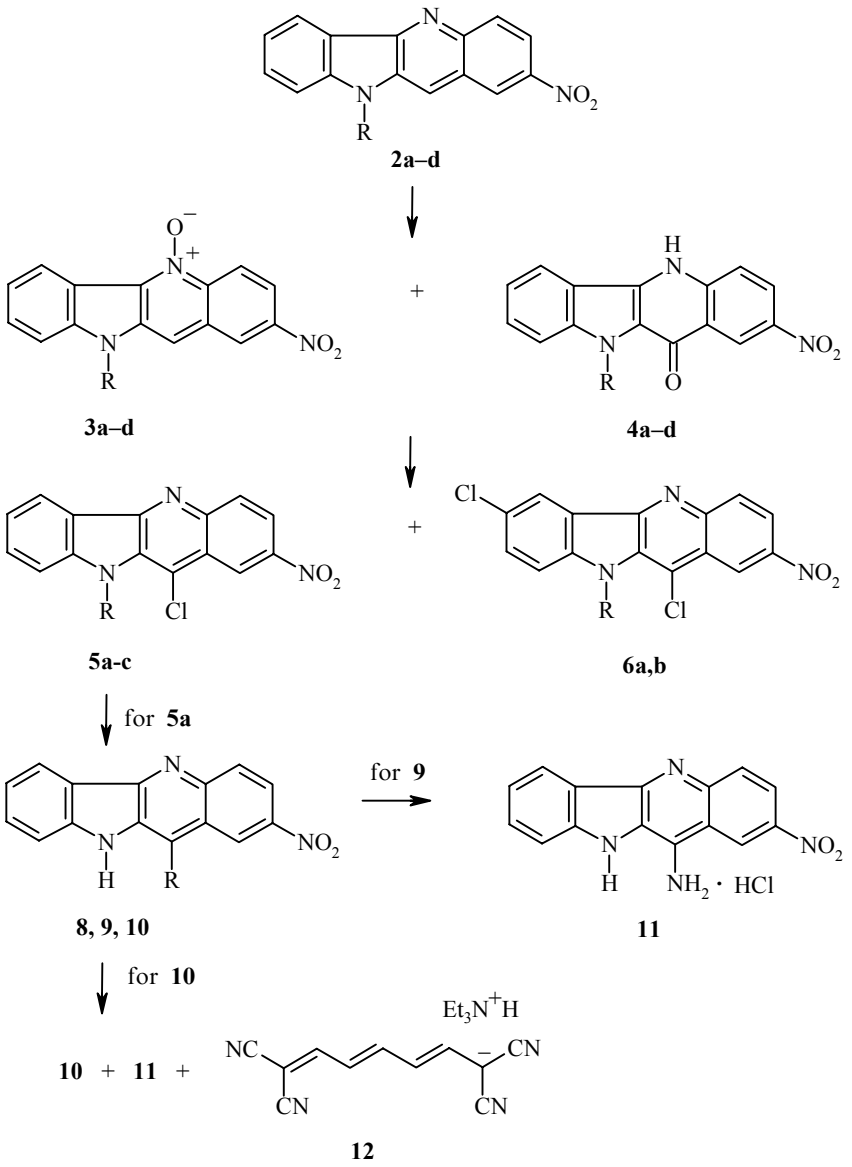
synthesized analogously, starting from the previously prepared 10-methyl-(**2c**) and 10-benzyl-(**2d**) derivatives [4]. The yields of the tetracycles **4c,d** was 61 and 41% respectively (in two steps, calculated on **2c,d** respectively). The ^{1}H NMR spectra are given in Table 1.



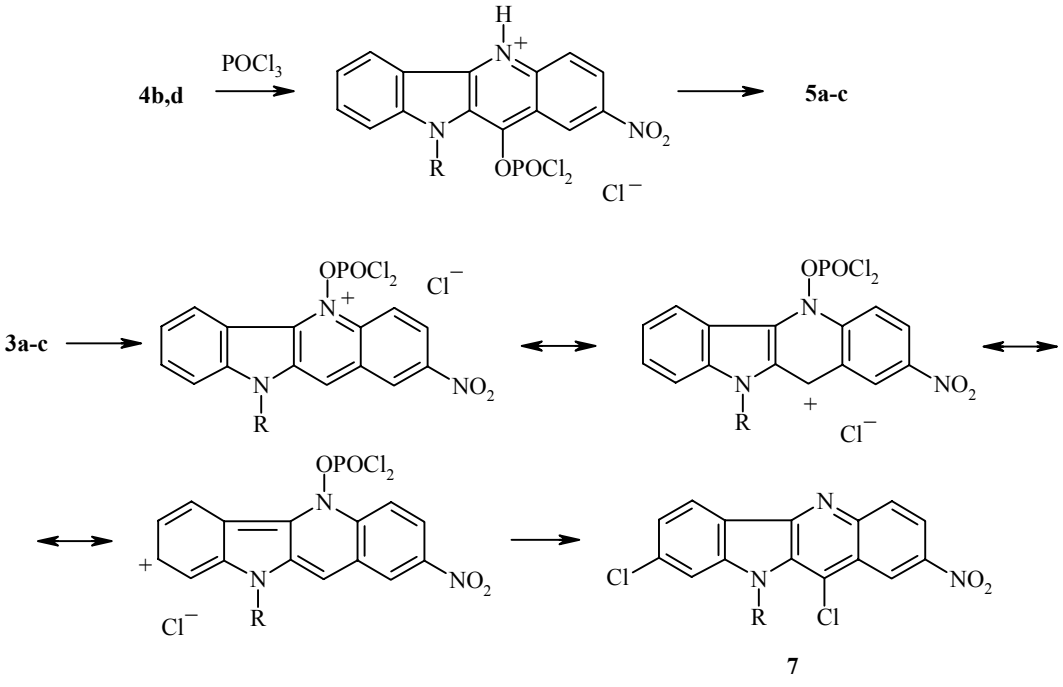
2-4 a R = Ac, **b** R = H, **c** R = Me, **d** R = CH_2Ph; **5**, **6**: **a** R = H, **b** R = Me, **c** R = CH_2Ph;
8 R = morpholino; **9** R = piperidino; **10** R = pyridinium, Cl^-

Heating of the indoloquinolinones-11 **4b,d** in phosphorus oxychloride in the presence of triethylamine hydrochloride the corresponding 11-chloro derivatives **5a,c** were readily obtained in high yield. However, an unexpected difficulty appeared when the mixture of N-oxides and quinolones, formed in the oxidation stage of the initial tetracycles **2**. On heating the mixtures **3a** and **4a**, **3b** and **4b**, **3c** and **4c** in $POCl_3$ the dichloro derivatives **6a,b** were formed along with the expected 11-chloro derivatives **5a,b**.

Thus if the reactions of indoloquinolones with $POCl_3$ occurs clearly in the usual direction to give the corresponding 11-chloro derivatives, then the presence of the N-oxide (and H_2O_2) in the reaction mixture leads to a similar process the introduction of a second chlorine atom into the benzene ring of the indole unit.

According to the scheme presented below, if the second chlorine atom is introduced into the molecule by a nucleophilic substitution mechanism, the expected product is the 8,11-dichloro derivative **7**. However, during the reaction a compound is formed in which, to judge from its ^{1}H NMR spectra, the second chlorine atom goes not to position 8 of the ring, but to position 7, i.e., a compounds of type **6** were formed.

The structures of the dichloro derivatives were determined from ^{1}H NMR spectroscopic data. A characteristic of the ^{1}H NMR spectrum of the 11-chlorotetracycle **5a** (in DMSO-d_6 solution) is the weak-field position of the 6-H doublet (~8.26 ppm) which overlaps the multiplets of the 3- and 4-H protons to form an AA^1 system at ~8.26 ppm. The doublet of proton 9-H is observed at higher field (7.56 ppm). The signals at 7.32 (7-H) and 7.69 ppm (8-H) appear as triplets. Comparison of the chemical shifts of the 3-, 4-, 6-, 7-, 8-, and 9-H protons of the monochloro derivative **5a** with those of derivatives without a substituent at position 11*, shows a comparatively good coincidence of the chemical shifts of the corresponding protons. From a theoretical examination of the two possible positions of incorporation of the second chlorine atom in the unsubstituted benzene ring at position 7 (**6a**) or 8 (**7**) it can be predicted that in the case of dichloride **6a** a doublet for 6-H should be observed at weak field in the ^{1}H NMR spectrum at ~ 8.30 ppm*^{2}, interacting with proton 8-H with a *meta* constant; in its turn proton 8-H forms a quartet by interaction with protons 6- and 9-H at ~7.70 ppm, and a doublet for 9-H should be observed at ~7.60 ppm. A different situation should be observed in the ^{1}H NMR spectrum of the dichloride **7**. The 6-H doublet should be virtually unchanged in form and position in comparison with the monochloride **5a** at ~8.30 ppm, whereas there should be a quartet (coupling with protons 6- and 9-H) in place of the triplet at 7.32 ppm (7-H) in approximately the same field; the doublet for proton 9-H (coupled with proton 7-H) should be observed at ~ 7.60 ppm.



The ^{1}H NMR spectrum of the mixture of the monochloro **5a** and dichloro derivatives in a 70 to 30% ratio showed that, along with signals for compound **5a**, the positions of which practically coincided with those of the isolated monochloro product described above, clearly identified signals of the dichloride at 8.06 (d,

* δ 8.33 (2H, d, 3-, 4-H), 8.40 (1H, d, 6-H), 7.35 (1H, t, 7-H), 7.68 (1H, t, 8-H), 7.63 ppm (1H, d, 9-H) [5].

*^{2} The increments for a chlorine atom introduced into a benzene ring are for *o*-protons Δδ = 0.03, for *m*-protons Δδ = -0.02, and for *p*-protons Δδ = -0.09 ppm [6].

TABLE 1. ^{1}H NMR Spectra of Compounds **3-6** and **8-11**

Com-pound	Chemical shifts, δ, ppm (DMSO-d_6)						
	1-H	3-H	4-H	6-, 7-, 8-, 9-H	10-R	11-R	N–H
3a	9.14	8.49	8.80	8.38 d, 7.60 t, 7.83 t, 9.07 d	2.99	9.06 s	—
4a	9.07	8.48	7.89	8.27 d, 7.52 t, 7.70 t, 8.28 d	2.79	—	—
3b	9.27	8.31	8.83	8.84 d, 7.35 t, 7.70 t, 7.60 d	12.08 sh. s	8.30 s	—
4b*	9.13	8.40	7.84	8.16 d, 7.24 t, 7.52 (m, 8-, 9-H)	12.02	—	13.09
3c	9.16	8.33	8.84	8.87 d, 7.40 t, 7.78 t, 7.74 d	3.96 s	8.41 s	—
4c	9.11	8.47	8.06	8.34 d, 7.26 t, 7.60 t, 7.68 d	4.29	—	—
3d	9.18	8.35	8.85	8.90 d, 7.40 t, 7.75 (m, 8-, 9-H)	5.74 c (CH_2); 7.15-7.35 (10-CH_2Ph)	8.52 s	—
4d*[2]	9.16 br. s	8.51 br. s	8.12 br. s	8.40 br. s, 7.30 br. t, 7.57 br. t, 7.75 br. s	6.15 br. s	—	12.93 sh. s
4a	9.07	8.49	7.90	8.28 d, 7.52 t, 7.61 t, 8.20 d	2.80	—	—
4b	9.15	8.42	7.86	8.18 d, 7.26 t, 7.52 (m, 8-, 9-H)	12.00	—	13.10
5a	8.88	8.26 (2H, m)		8.25 d, 7.32 t, 7.69 t, 7.56 d	11.01 sh. s	—	—
6a	8.74	8.21	8.16	8.06 d, 7.61 q, 7,50 d	12.16 sh. s	—	—
5b*[3]	9.11	8.39 (2H, m)		8.38 d, 7.41 t, 7.80 t, 7.77 d	4.24 s	—	—
6b	9.32	8.41	8.33	8.44 d, 7.65 q, 7.37 d	4.25 s	—	—
5c	9.13	8.44 (2H, m)		8.46 d, 7.45 t, 7.77 (m, 8-, 9-H)	6.10 (2H, s, CH_2); 7.12-7.30(4H, m, 10-CH_2Ph)		
8	9.27	8.33	8.30	8.36 d, 7.53 t, 7.69 (m, 8-, 9-H)	11.28 (s, 10-H); 3.56 (m, $N(CH_2)_2$); 3.99 (m, $O(CH_2)_2$)		
9	9.17	8.28	8.23	8.31 d, 7.29 t, 7.62-7.68 (m, 8-, 9-H)	11.00 (s, 10-H); 1.73-1.94 (m, $(CH_2)_3$); 3.53 (m, $N(CH_2)_2$)		
10	8.33	8.51	8.65	8.53 d, 7.49 t, 7.82 t, 7.65 d	12.33 br. s, 8.62 (2H, m); 9.10 (1H, t); 9.56 (2H, d)		
11	9.59	8.55	8.26	8.47 d, 7.34 t, 7.68 (m, 8-, 9-H)	12.60 sh. s	9.55 (NH_2^+)	—

* The signal at 12.86 ppm belongs to the H_2O_2 which forms a complex with two molecules of **4b**. In the ^{1}H NMR spectrum of a mixture of **3b**:**4b** diluted five-fold with solvent this signal is observed to move to high field at 12.24 ppm.

*[2] The multiplicity of signals are masked because of overlap with other signals.

*[3] Solvent $CDCl_3$.

$^4J_{6,8}$ = 2.2), 7.61 (q, $^4J_{6,8}$ = 2.2, $^3J_{8,9}$ = 8.8) and 7.50 ppm (d, $^3J_{8,9}$ = 8.8 Hz), the positions and multiplicities of which clearly agree only with the possible form **6a**, i.e., the second chlorine atom is unambiguously at position 7 of the tetracycle. An analogous picture was observed for spectra of mixtures of **5b** and **6b**.

These data indicate unambiguously that the products of the second chlorination are the 7,11-dichloro derivatives. From this it can be concluded that the second chlorination is not a nucleophilic substitution. The presence in the reaction medium of oxidants (N-oxide and H_2O_2) and chloride anions suggests that chlorine radical are formed under the reaction conditions and that the second chlorination occurs *via* radical mechanism. It seems that a special study is required to confirm this postulate.

The feasibility and the evident generality of the route to functionalise indoloquinolines to 11-chloro derivatives permits the use of these compounds for the synthesis of other 11-substituted derivatives of these heterocyclic systems. The chlorine atom in compound **5a** is sufficiently reactive that on refluxing it with morpholine and piperidine the 11-morpholino and 11-piperidino derivatives (**8** and **9**) were isolated in 70 and 80% yields respectively. Moreover, heating the chloro derivative **5a** with pyridine gave the corresponding salt **10** in excellent yield (81%).

When the latter compound was heated for a short time with piperidine, 11-amino-2-nitroindolo[3,2-*b*]-quinoline (**11**) was obtained in close to quantitative yield. The reaction apparently occurs by a Zinke–König type of reaction *via* addition of the amine at the α-position of the pyridine unit with subsequent elimination of a derivative of glutaconic aldehyde by a scheme which we recently described in a study of the reaction of 1-(2-pyridyl)pyridinium salts with amines [7]. The amino derivatives **11** are also formed by the reaction of the pyridinium salt **10** with sodium hydroxide, sodium methoxide, or triethylamine in methanol. However in these cases large amounts of polymerization products are formed (^{1}H NMR and mass spectroscopy). The reaction of salt **10** with malononitrile in pyridine occurs differently from the corresponding reaction with 1-(2-pyridyl)pyridinium salts [7]. Aminotriene systems of type **12** are characteristically formed in the latter reaction with malononitrile, whereas with compound **10** a mixture of the starting pyridinium salt **10**, the 11-amino derivative **11**, and the triethylammonium salt of 1,1,7,7-tetracyanoheptatrienide (**12**) is formed in a 10:18:25 ratio according to ^{1}H NMR spectroscopic data. Signals are observed in the ^{1}H NMR spectrum for protons corresponding to compounds **10**, **11,** and also signals for protons corresponding to the salt **12**.

EXPERIMENTAL

IR spectra were recorded as nujol mulls with a Perkin-Elmer 457 spectrometer, mass spectra with a Finnigan-MAT SSQ-710 with indirect injection of the sample into the ion source, with ionizing electron energy of 70 eV, and temperature of the ionisation chamber 150°C. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity+400 (400 MHz) with TMS as internal standard. Monitoring of the course of a reaction and of the purity of a substance was carried out by TLC on Silufol UV-254 strips with chloroform–methanol 1:10 as solvent. Materials were revealed by UV light.

N-Oxide of 10-Acetyl-2-nitroindolo[3,2-*b*]quinoline (3a) and 10-acetyl-2-nitro-5H-indolo[3,2-*b*]-quinolin-11-one (4a). Hydrogen peroxide (1.5 ml, 30%) was added to a suspension of tetracycle **2a** (0.5 g, 1.6 mmol) in glacial acetic acid (20 ml) and the mixture was stirred for 10 h at 90°C, additional hydrogen peroxide (1 ml) being added twice. The reaction mixture was cooled to 20°C, the precipitate was filtered off and washed with 1:1 methanol–water to give a mixture of **3a** and **4a** (0.3 g, 57%); mp >300°C. M^+ 321.

N-Oxide of 2-Nitro-10H-indolo[3,2-*b*]quinoline (3b) and 2-Nitro-5,10H-indolo[3,2-*b*]quinolin-11-one (4b). Hydrogen peroxide (15 ml, 30%) was added to a suspension of tetracycle **2b** (5 g, 19 mmol) in glacial acetic acid (150 ml) at 30°C and the mixture was stirred for 5 h at 75-80°C. The reaction mixture was cooled to 20°C, the precipitate was filtered off and washed with 1:1 methanol–water to give a mixture of **3b** and **4b** (4.83 g, 91%); mp >320°C. $M^{+\cdot}$ 279.

N-Oxide of 10-Methyl-2-nitroindolo[3,2-*b*]quinoline (3c) and 10-Methyl-2-nitro-5H-indolo[3,2-*b*]-quinolin-11-one (4c) was obtained from tetracycle **2c** [5] (1 g, 3.6 mmol) under the conditions for the synthesis of a mixture of compounds **3b** and **4b**. Reaction time 1h 30 min. Obtained 0.99 g (93%) of a mixture of compounds **3c** and **4c**; mp 290-295°C. $M^{+\cdot}$ 293.

N-Oxide of 10-Benzyl-2-nitroindolo[3,2-*b*]quinoline (3d) and 10-Benzyl-2-nitro-5H-indolo[3,2-*b*]-quinolin-11-one (4d) was obtained from tetracycle **2d** [5] (0.7 g, 2 mmol) under the conditions for the synthesis of a mixture of compounds **3b** and **4b**. Reaction time 3h . Obtained 0.59 g (81%) of a mixture of compounds **3d** and **4d**; mp 265°C. $M^{+\cdot}$ 369.

10-Acetyl-2-nitro-5H-indolo[3,2-*b*]quinolin-11-one (4a) and 2-Nitro-5,10H-indolo[3,2-*b*]quinolin-11-one (4b). A suspension of a mixture of compounds **3a** and **4a** (0.45 g) was refluxed in acetic anhydride for 9 h 30 min. The precipitate which formed in the hot solution was filtered off after cooling to 20°C and washed with methanol to give a mixture of compounds **4a** and **4b** (0.35 g) 60:30 according to the ^{1}H NMR spectrum. $M^{+\cdot}$ 321, $M^{+\cdot}$ 279.

2-Nitro-5,10H-indolo[3,2-*b*]quinolin-11-one (4b). A. The filtrate from the synthesis of a mixture of compounds **4a** and **4b** was poured into water (100 ml), and neutralised with Na_2CO_3 to pH 7. The filtrate was filtered off and washed with water to give 0.11 g of compound **4b**; mp >320°C (DMF). IR spectrum, ν, cm^{-1}: 3380, 1630, 1590, 1575. $M^{+\cdot}$ 279. Found, %: C 64.24; H 3.38; N 15.06. $C_{15}H_9N_3O_3$. Calculated, %: C 64.52; H 3.23; N 15.05.

B. Sodium hydroxide solution (1.5 ml, 1N) was added to a suspension of compounds **4a** and **4b** (0.3 g) in dioxan (5 ml) and the reaction mass was refluxed for 3 h. The precipitate from the boiling solution was filtered off after cooling to 20°C, washed with water to give compound **4b** (0.24 g).

C. A suspension of a mixture of compounds **3b** and **4b** (2 g, 7.2 mmol), obtained from tetracycle **2b**, was heated while stirring in acetic anhydride (15 ml) for 15 min. The reaction mixture was cooled to 20°C, added to ethyl acetate (30 ml), the precipitate was filtered off and washed with ethyl acetate to give compound **4b** (1.86 g, 93%). A mixed melting point with samples made by mehods A or B gave no depression.

10-Methyl-2-nitro-5H-indolo[3,2-*b*]quinolin-11-one (4c) was obtained from a mixture of compounds **3c** and **4c** (0.27 g, 0.7 mmol), obtained from tetracycle **2c**, under the synthesis conditions for tetracycle **4b**, method B, reaction time 30 min, to give compound **4c** (0.13 g, 65%); mp >300°C (1:1 MeOH–DMF). $M^{+\cdot}$ 293. Found, %: N 14.34. $C_{16}H_{11}N_3O_3$. Calculated, %: N 14.32.

10-Benzyl-2-nitro-5H-indolo[3,2-*b*]quinolin-11-one (4d) was obtained from a mixture of compounds **3d** and **4d** (0.2 g, 0.5 mmol), obtained from tetracycle **2d**, under the synthesis conditions for tetracycle **4b**, method C, reaction time 1 h, to give compound **4d** (0.1 g, 50%); mp >330°C (1:1 MeOH–DMF). $M^{+\cdot}$369. Found, %: N 11.47. $C_{22}H_{15}N_3O_3$. Calculated, %: N 11.38.

11-Chloro-2-nitro-10H-indolo[3,2-*b*]quinoline (5a). A. Triethylamine hydrochloride (0.4 g, 3 mmol) was added to a suspension of tetracycle **4b** (1 g, 3.6 mmol) in phosphorus oxychloride (10 ml) and the mixture was refluxed for 15 min and then cooled to 20°C, the precipitate was filtered off and washed with water to give compound **5a** (1.05 g, 98%); mp 310-314°C (2:5 propanol-2–DMF). $M^{+\cdot}$ 297. Found, %: C 60.48; H 2.64; Cl 11.75; N 14.07. $C_{15}H_8ClN_3O_2$. Calculated, %: C 60.50; H 2.69; Cl 11.93; N 14.12.

10-Benzyl-11-chloro-2-nitroindolo[3,2-*b*]quinoline (5c) was obtained from a mixture of N-oxide **3d** and quinolinone **4d** (0.27 g, 0.7 mmol), prepared from tetracycle **2d**, under the conditions for the synthesis of the chloro derivative **5a** in a yield of 0.2 g (71%); mp 263-265°C (acetonitrile). $M^{+\cdot}$ 387. Found, %: C 68.43; H 3.35; Cl 9.45; N 10.45. $C_{22}H_{14}ClN_3O_2$. Calculated, %: C 68.13; H 3.61; Cl 9.16; N 19.84.

11-Morpholino-2-nitro-10H-indolo[3,2-*b*]quinoline (8). A suspension of the chloro derivative **5a** (0.3 g, 1 mmol) was refluxed in morpholine (3 ml) for 1 h. The precipitate was filtered off and washed with water and methanol to give compound **8** (0.26 g, 74%); mp >360°C (DMF). $M^{+\cdot}$ 348. Found, %: C 64.99; H 4.58; N 15.63. $C_{19}H_{16}N_4O_3$. Calculated, %: C 65.52; H 4.60; N 16.08.

2-Nitro-10H-11-piperidinoindolo[3,2-*b*]quinoline (9). A suspension of the chloro derivative **5a** (0.3 g, 1 mmol) was refluxed in piperidine (3 ml) for 1 h. The reaction mixture was poured into water, the precipitate was filtered off and washed with water to give compound **9** (0.28 g, 80%); mp 295-299°C (propanol-2). $M^{+\cdot}$ 346. Found, %: C 69.36; H 5.50; N 15.92. $C_{20}H_{18}N_4O_2$. Calculated, %: C 69.33; H 5.24; N 16.18.

1-(2-Nitro-10H-indolo[3,2-*b*]quinolinyl-11)pyridinium Chloride (11). A suspension of compound **10** (0.2 g, 5 mmol) in piperidine (1 ml) was heated until the precipitate dissolved. The solution was then kept at 20°C for 3 h. Methanol (10 ml) and hydrochloric acid (2-3 ml) was then added to the solution. The precipitate was filtered off and washed with methanol to give compound **11**, (0.17 g, 99%); mp >340°C (DMF). $M^{+\cdot}$ 278. Found, %: C 54.29; H 3.87; Cl 10.84; N 16.72. $C_{15}H_{11}N_4O_2 \cdot HCl \cdot H_2O$. Calculated, %: C 54.145; H 3.91; Cl 10.68; N 16.84. Found, %: 5.49 H_2O. Calculated, %: 5.41 H_2O.

Reaction of Pyridinium Salt 10 with Malonodinitrile . Triethylamine (0.15 ml) was added to a mixture of compound **10** (0.4 g, 1.1 mmol), malononitrile (0.07 g, 1 mmol) in pyridine (10 ml). The precipitate dissolved slowly. The reaction mixture was maintained at 20°C for 48 h. The pyridine was evaporated to dryness. Toluene was added to the residue and the solvent was evaporated to dryness to give a mixture of compounds **10-12**. ^{1}H NMR spectrum (compound **12**) δ, ppm: 5.91 (2H, t, 3-, 5-H); 7.15 (1H, t, 4-H); 7.26 (2H, d, 1-, 6-H); 9.42 (1H, br. s, N^+H); 1.18 (9H, t, $(C\underline{H}_3CH_2)_3N^+$); 3.05 (6H, q, $(CH_3C\underline{H}_2)_3N^+$).

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