Novel convenient *one-pot* method for the synthesis of indoloquinolines*

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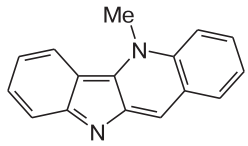
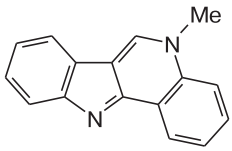
A novel *one-pot* method based on the sequence of the Fisher reaction between nitroacetophenone and phenylhydrazines, the reduction with metallic tin directly in polyphosphoric acid, and acylation has been developed for the synthesis of indoloquinolines. The obtained indolo[3,2-*c*]quinolines are precursors of the analogs of isocryptolepine alkaloid.

Key words: Fischer indole synthesis, isocryptolepine, reduction, *one-pot* synthesis, indole, *Cryptolepis Sanguinolenta*.

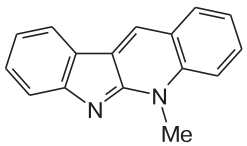
The indole cycle system is an important moiety of many drugs and several thousands of alkaloids exhibiting various types of biological activity. It is known that indole derivatives are more than a quarter of all the known alkaloids.[1] More than a dozen of alkaloids were extracted from roots of the South African plant *Cryptolepis Sanguinolenta*, and many of which demonstrated antimalarial and antitumor activities.[2—6] Alkaloids of this plant are mainly present by tetracyclic systems containing an indole core, *e.g.*, indolo[3,2-*b*]quinoline, indolo[3,2-*c*]quinoline, indolo[2,3-*b*]-quinoline, and indolo[3,2-*b*][1]benzazepine, whereas isocryptolepine, neocryptolepine, and cryptolepine can be distinguished among them. Their analogs, *e.g.*, 5-aminopyrido[3′,2′:4,5]thieno[3,2-*c*]isoquinolines reported previously,[7] are not of less interest from the point of view of the biological activity.



Cryptolepine



Isocryptolepine



Neocryptolepine

Usually, the synthesis of indolo[3,2-*c*]quinolines includes a final assembly of one of the cores, either quinoline[8—13] or indole[14—22] one, while the second core was already present in the substrate. Many of these methods involve expensive starting compounds and include many steps with the isolation of intermediate compounds. The assembly of both cores was also reported,[23—28] including our recent work,[28] wherein a *one-pot* synthesis of indolo[3,2-*c*]quinolines was revealed as a sequence of the Fisher reaction between phenylhydrazine **1** and 2-aminoacetophenone **2**, the acylation of resulting 2-(2-aminophenyl)-indoles **3**, and the cyclization into target product **4**. Obtained indoloquinolines **4** either already contain a methyl group at the position 5 or can be easily converted into the corresponding isocryptolepine derivatives by the treatment with dimethyl sulfate or methyl iodide (Scheme 1).

Despite the high product yields in this reaction, the major drawback of that method is the utilization of 2-aminoacetophenones as the starting compounds. We believed that the replacement of 2-aminoacetophenones **2** by their precursors, 2-nitroacetophenones **5**, is an interesting idea. To implement this approach, it was necessary to add a step of the reduction in polyphosphoric acid, but there were no reported examples of such transformations in the literature. We have previously investigated a number of interesting conversions proceeding in polyphosphoric acid,[29—31] including the opportunity to reduce aliphatic nitro compounds into amides of carboxylic acid using phosphorus trichloride.[32] We assumed that this transformation could be carried out similarly to the reduction by metals dissolving in the presence of acids, such as HCl, H_3PO_4, and AcOH. For example, some works[25,26] reported on the reduction of 2-(2-nitrophenyl)indoles by Fe—HCl system in ethanol.

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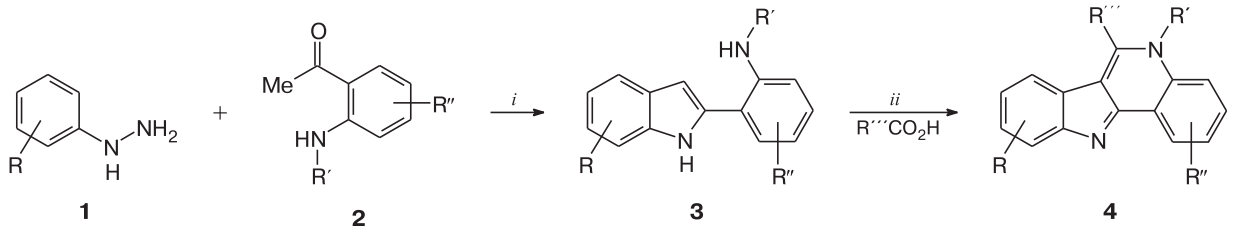
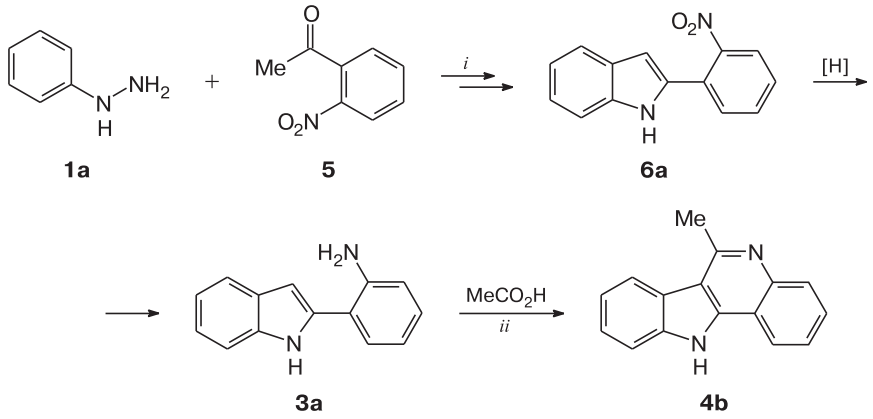
Scheme 1



Reagents and conditions: *i*. 1) 120 °C, 2) polyphosphoric acid (PPA), 100 °C; *ii*. PPA.

Scheme 2



Reagents and conditions: *i*. PPA, 60 °C; *ii*. PPA.

In order to verify this hypothesis, we prepared 2-(2-nitrophenyl)indole **6a** *via* the Fisher reaction in PPA medium at 60 °C for 2 h (Scheme 2). Zinc dust (1 equiv.) was added in portions every 1 h to the resulting reaction mixture until the nitro compound disappeared in the reaction mixture. Acetic acid was then added, the mixture was heated for another 30 min, and the product was isolated. The reaction yield was about 5% (Table 1). We explain this result by the high instability of 2-(2-nitrophenyl)indoles **6** under the reaction conditions, which are rapidly polymerized at the temperatures above 80 °C. In addition, the zinc dust is insoluble in polyphosphoric acid. To accelerate the reduction process, zinc was replaced by tin, which should produce Sn^{2+} salts soluble in PPA. At this end, Sn^0 worked slightly better than Sn^{2+}. Yields of the obtained products are given in Table 1.

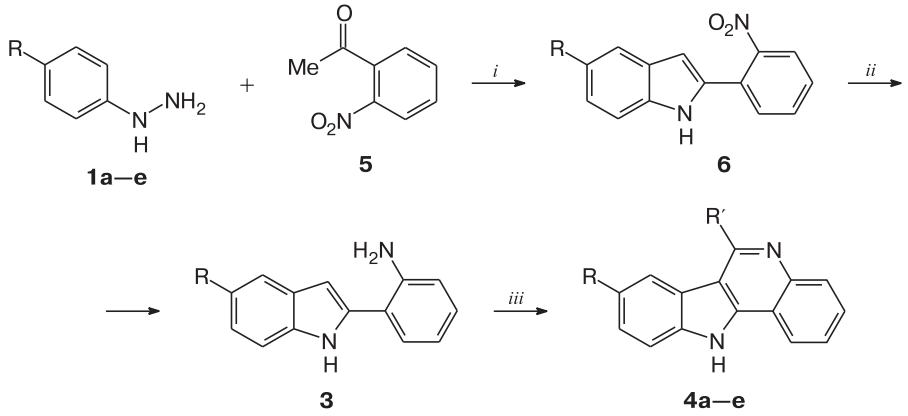
We have earlier demonstrated[28] that corresponding 11*H*-indolo[3,2-*c*]quinolines **4** can be obtained in the yields of 75—86%, while the step of acylation of 2-(2-aminophenyl)indole **3** proceeded in a quantitative yield. As one can see from the optimization table, the main reason of the decreased yield is the thermal decomposition of 2-(2-nitrophenyl)indole **6** during the reaction. The faster reduction resulted in the higher yield. We suggested that a nature of the acylating agent should not significantly affect the reaction yield. To verify this hypothesis, we have varied the acylating reagents and also the substituents at the position 8 of indoloquinoline (Scheme 3). The results are given in Table 2.

Therefore, we have developed a convenient method for the synthesis of 11*H*-indolo[3,2-*c*]quinolines, which excludes the utilization of *o*-aminoacetophenones that are inaccessible and unstable under the reaction conditions. The obtained compounds can be converted into the

Table 1. Optimization of conditions for the synthesis of compound **4b**

Concentration of P_2O_5 in PPA (%)	Reducing agent (equiv.)	*T*/°C	Yield of **4b** (%)
76	Sn (2)	60	0
80	Sn (2)	60	0
86	Sn (2)	60	25
86	Sn (2)	70	15
86	Sn (2)	80	0
86	$SnCl_2 \cdot 2\,H_2O$ (2)	60	20
86	Zn (3)	60	5

Scheme 3



Reagents and conditions: *i*. PPA, 60 °C; *ii*. PPA, Sn, 60 °C; *iii*. **A**, PPA, 90—120 °C.

Table 2. Structures and yields of compounds **4a**—**e**

Reagents		Product	R	R′	Yield
1	**A**			(%)	
a	1,3,5-Triazine	**4a**	H	H	25
b	AcOH	**4b**	H	Me	25
c	PhCOOH	**4c**	H	Ph	24
d	1,3,5-Triazine	**4d**	Me	H	22
e	PhCOOH	**4e**	Pr^i	Ph	26

isocryptolepine derivatives by their treatment with methyl iodide according to the known procedure.[33]

Experimental

^{1}H NMR spectra were recorded on a Bruker AVANCE III HD instrument (the operating frequency of 400.40 MHz) in DMSO-d_6 or $CDCl_3$ using the residual solvent signals as the internal standard. Mass spectra of the compounds were obtained on a Bruker Maxis impact spectrometer using the direct injection system, the ionization method was ESI, and the calibrant was HCO_2Na—HCO_2H.

IR spectra were recorded on a Shimadzu IRAffinity-1S FTIR spectrometer equipped with an ATR device (ATR means attenuated total reflection).

The purity of compounds was controlled by TLC on Silufol UV-254 plates, the eluent was acetone—hexane mixture (from 1 : 5 to 1 : 1), and the plates were visualized by UV irradiation. Melting points were measured on a Stuart SMP30 instrument.

Polyphosphoric acids were obtained either according to the standard procedure[34] or by dissolving P_2O_5 in H_3PO_4 (85%).[35]

Synthesis of 11*H*-indolo[3,2-*c*]quinolines (general procedure). A mixture of 2-nitroacetophenone **5** (82 mg, 0.5 mmol), corresponding phenylhydrazine **1** (0.5 mmol), and polyphosphoric acid (1.0 g, the P_2O_5 content of 86%) was stirred at room temperature for 5 min. The process was strongly exothermic. The mixture was then stirred at 60 °C until the complete disappearance of the starting reagents (~1.5 h, TLC control). Metallic Sn (1 mmol) was added, the mixture was stored for another 2 h, and the acylating agent (1.2 mmol) was added. The reaction mixture was then heated to 130 °C (1,3,5-triazine was used as the synthetic equivalent of HCOOH at 100 °C) and stored until the intermediate product disappeared completely (about 2 h). Next, The reaction mixture was cooled to room temperature, diluted with water (40 mL), and neutralized with 20% aqueous ammonia (~7 mL) until basic pH. The resulting mixture was extracted with ethyl acetate (4×15 mL), evaporated, and purified by column chromatography using an acetone—hexane mixture (from 1 : 5 to 1 : 1) as the eluent.

11*H*-Indolo[3,2-*c*]quinoline (4a). Colorless crystals, the yield of 27 mg (25%), m.p. 340—341 °C (*cf.* Ref. 28: m.p. 340—341 °C). IR, ν/cm^{-1}: 3047, 2775, 1571, 1519, 1462, 1373, 1341, 1242, 1158, 933, 771, 740. ^{1}H NMR (DMSO-d_6), δ: 7.34 (t, 1 H, H(8), J = 7.3 Hz); 7.55—7.45 (m, 1 H, H(9)); 7.78—7.65 (m, 3 H, H(2), H(3), H(10)); 8.14 (d, 1 H, H(7), J = 7.9 Hz); 8.32 (d, 1 H, H(1), J = 7.8 Hz); 8.53 (dd, 1 H, H(4), J = 8.0 Hz, J = 1.1 Hz); 9.60 (s, 1 H, H(6)); 12.73 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 111.8 (C(10)), 114.3 (C(6a)), 117.1 (C(6b)), 120.1 (C(9)), 120.6 (C(7)), 121.9 (C11b), 122.1 (C(8)), 125.5 (C(2)), 125.7 (C(3)), 128.0 (C(1)), 129.5 (C(4)), 138.8 (C(10a)), 139.8 (C(11a)), 144.8 (C(6)), 145.4 (C(C4a)). MS (ESI—TOF): found: m/z 219.0917 $[M + H]^+$; $C_{15}H_{11}N_2$; calculated: M = 219.0917. R_f = 0.19 (ethyl acetate), R_f = 0.5 (hexane—acetone, 1 : 1).

6-Methyl-11*H*-indolo[3,2-*c*]quinoline (4b). Colorless crystals, the yield of 29 mg (25%), m.p. 208—210 °C (*cf.* Ref. 28: m.p. 208—210 °C. IR, ν/cm^{-1}: 3054, 2932, 2853, 1599, 1555, 1452, 1359, 1114. ^{1}H NMR (DMSO-d_6), δ: 3.10 (s, 3 H, C(6)Me); 7.37 (ddd, 1 H, H(8), J = 8.0 Hz, J = 7.0 Hz, J = 0.6 Hz); 7.52 (ddd, 1.H, H(9), J = 8.1 Hz, J = 7.0 Hz, J = 0.8 Hz); 7.65 (ddd, 1 H, H(2), J = 7.9 Hz, J = 6.8 Hz, J = 0.9 Hz); 7.76—7.70 (m, 2 H, H(3), H(10)); 8.06 (d, 1 H, H(7), J = 8.1 Hz); 8.22 (d, 1 H, H(1), J = 8.0 Hz); 8.52 (dd, 1 H, H(4), J = 8.0 Hz, J = 0.9 Hz); 12.90 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 22.5 (Me), 112.0 (C(10)), 113.0(C(9)), 116.0 (C(7)), 121.0 (C(6a)), 121.6 (C(8)), 122.1 (C(2)), 122.3 (C(3)), 125.3 (C(6b)), 125.4 (C(1)), 127.3 (C(4)), 128.6 (C(11b)), 138.9 (C(10a)), 140.3 (C(11a)), 143.3 (C(4a)), 154.0 (C(6)). MS (ESI—TOF): found: m/z 233.1074 $[M + H]^+$;

$C_{16}H_{13}N_2$; calculated: M = 233.1073. R_f = 0.5 (ethyl acetate), R_f = 0.72 (hexane—acetone, 1 : 1).

6-Phenyl-11*H*-indolo[3,2-*c*]quinoline (4c). Colorless crystals, the yield of 35 mg (24%), m.p. 248—250 °C (*cf.* Ref. 28: m.p. 248—250 °C). IR, ν/cm^{-1}: 3172, 3054, 2917, 2843, 1560, 1530, 1501, 1452, 1359, 1320, 1241, 1222. ^{1}H NMR (DMSO-d_6), δ: 7.14 (ddd, 1 H, H(8), *J* = 8.0 Hz, *J* = 7.0 Hz, *J* = 0.9 Hz), 7.50—7.33 (m, 6 H, H(9), Ph); 7.62—7.57 (m, 2 H, H(2), H(3)); 7.97—7.90 (m, 2 H, H(10), H(7)); 8.24 (dd, 1 H, H(1) , *J* = 8.2 Hz, *J* = 0.8 Hz); 8.33 (d, 1 H, H(4), *J* = 8.3 Hz); 12.24 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 111.8 (C(10)), 113.4 (C(6a)), 116.8 (C(6b)), 120.8 (C(9)), 121.8 (C(7)), 121.9 (C(8)), 122.8 (C(11b)), 125.6 (C(2)), 125.6 (C(3)), 128.6 (2 C, $C(3)_{6\text{-Ph}}$, $C(5)_{6\text{-Ph}}$), 128.7 (C(1)), 128.9 (C(4)), 129.0 (2 C, $C(2)_{6\text{-Ph}}$, $C(6)_{6\text{-Ph}}$), 129.1 ($C(4)_{6\text{-Ph}}$), 139.4 ($C(1)_{6\text{-Ph}}$), 140.3 (C(10a)), 141.8 (C(11a)), 145.3 (C(4a)), 156.9 (C(6)). MS (ESI—TOF): found: *m*/*z* 295.1237 $[M + H]^+$; $C_{21}H_{15}N_2$; calculated: M = 295.1230. R_f = 0.30 (hexane—ethyl acetate, 1 : 1), R_f = 0.75 (hexane—acetone, 1 : 1).

8-Methyl-11*H*-indolo[3,2-*c*]quinoline (4d). Colorless crystals, the yield of 26 mg (22%), m.p. 305—311 °C (*cf.* Ref. 28: m.p. 305—311 °C). IR, ν/cm^{-1}: 3042, 2773, 2366, 1570, 1363, 1239. ^{1}H NMR (DMSO-d_6), δ: 2.52 (s, 3 H, H(8)Me); 7.32 (dd, 1 H, H(9), *J* = 8.3 Hz, *J* = 1.3 Hz); 7.61 (d, 1 H, H(10), *J* = 8.3 Hz); 7.77—7.64 (m, 2 H, H(2), H(3)); 8.14—8.09 (m, 2 H, H(1), H(7)); 8.50 (dd, 1 H, H(4), *J* = 8.0 Hz, *J* = 1.1 Hz); 9.54 (s, 1 H, H(6)); 12.59 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 21.2 (C(8)Me), 111.5 (C(10)), 114.1 (C(6a)), 117.2 (C(6b)), 119.8 (C(9)), 122.0 (C(7)), 122.1 (C(11b)), 125.6 (C(2)), 126.9 (C(3)), 127.9 (C(1)), 129.4 (C(8)), 129.5 (C(4)), 137.0 (10a), 139.8 (11a), 144.7 (6), 145.4 (C(4a)). MS (ESI—TOF): found: *m*/*z* 233.1076 $[M + H]^+$; $C_{16}H_{13}N_2$; calculated: M = 233.1073. R_f = 0.24 (hexane—ethyl acetate, 4 : 1), R_f = 0.44 (hexane—acetone, 1 : 1).

8-Isopropyl-6-phenyl-11*H*-indolo[3,2-*c*]quinoline (4e). Colorless crystals, the yield of 44 mg (26%), m.p. 129.0—132.6 °C (*cf.* Ref. 27: m.p. 129—132 °C). IR, ν/cm^{-1}: 3056, 2920, 2366, 1615, 1590, 1557, 1516, 1490, 1443. ^{1}H NMR (DMSO-d_6), δ: 1.14 (d, 6 H, $H(8)_{Pr^i}$, *J* = 6.9 Hz); 2.87 (sept, 1 H, $H(8)_{Pr^i}$, *J* = 6.9 Hz); 7.34 (m, 2 H, H(7), H(9)); 7.66—7.60 (m, 4 H, $H(3)_{5\text{-Ph}}$, $H(4)_{5\text{-Ph}}$, $H(5)_{5\text{-Ph}}$, H(10)); 7.71—7.67 (m, 1 H, H(2)); 7.75 (t, 1 H, H(3), *J* = 7.2 Hz); 7.83 (dd, 2 H, $H_{6\text{-Ph}}(2)$, $H(6)_{6\text{-Ph}}$, *J* = 7.1 Hz, *J* = 1.8 Hz); 8.12 (d, 1 H, H(1), *J* = 8.2 Hz); 8.55 (d, 1 H, H(4), *J* = 8.0 Hz); 12.77 (s, 1 H, NH). ^{13}C NMR (DMSO-d_6), δ: 24.4 (2 C, C(8)Me), 33.4 (C(8)CH), 111.6 (C(10)), 112.0 (C(6a)), 116.4 (C(6b)), 118.0 (C(9)), 121.7 (C(11b)), 121.9 (C(7)), 124.6 (C(2)), 125.6 (C(3)), 128.3 (2 C, $C(3)_{6\text{-Ph}}$, $C(5)_{6\text{-Ph}}$), 128.4 (C(1)), 128.9 (C(4)), 129.0 (2 C, $C(2)_{6\text{-Ph}}$, $C(6)_{6\text{-Ph}}$), 129.4 ($C(4)_{6\text{-Ph}}$), 137.6 ($C(1)_{6\text{-Ph}}$), 140.2 (C(8)), 140.7 (C(10a)), 141.1 (C(11a)), 144.9 (C(4a)), 155.5 (C(6)). MS (ESI—TOF): found: *m*/*z* 337.1703 $[M + H]^+$; $C_{24}H_{21}N_2$; calculated: M = 337.1699. R_f = 0.71 (hexane—ethyl acetate, 1 : 1), R_f = 0.78 (hexane—acetone, 1 : 1).

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