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ONE-POT SYNTHESIS OF 6*H*-INDOLO[2,3-*b*]QUINOLINES FROM 2-NITROBENZALDEHYDE AND INDOLE DERIVATIVES VIA DOMINO REACTION

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Abstract – An efficient and simple method for the synthesis of 6*H*-indolo[2,3-*b*]-quinolones was described. The construction of this fused heterocycles system was achieved by alkylation–reduction–cyclization domino reaction of 2-nitro-benzaldehyde with indoles using $SnCl_2$ as a promoter in [bmim]Br medium.

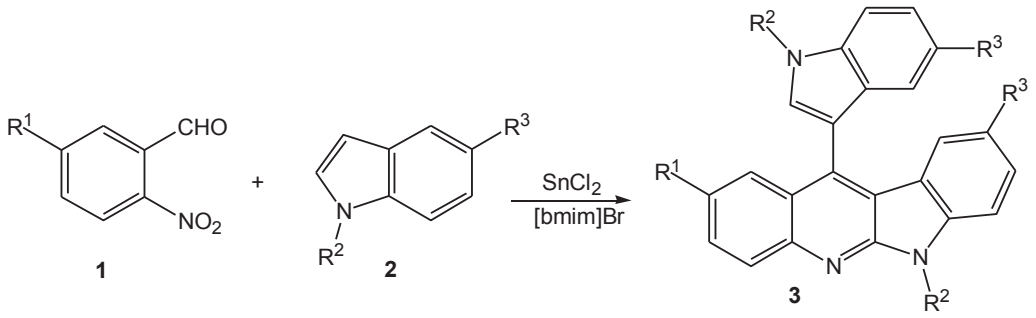
The indole-fused heterocyclic system is frequently found in natural products isolated from plants used in traditional medicine. Due to the broad spectrum of biological and pharmacological activities associated with compounds incorporating this structural skeleton, it has been recognized as an important privileged scaffold in drug discovery.[1] Particularly, indoloquinolines have been well established to be useful as antibacterial, antifungal, antimalarial, anticancer, antiplatelet, antihypertensive agents as well as exhibiting several other activities.[2] It is believed that they act as DNA intercalating agents[3] and topoisomerase II inhibitors.[4] Among them, 6*H*-indolo[2,3-*b*]quinoline is the precursor to cryptotakieine (neocryptolepine) alkaloid,[5] and displays a strong antiplasmodial activity[6] in addition to antimicrobial and cytotoxic activity.[7] So development of a new and efficient methodology for the synthesis of biologically potent indolo[2,3-*b*]quinolines has drawn great attention of synthetic as well as medicinal chemists.[8]

$SnCl_2 \cdot 2H_2O$ is an effective reagent that has been used for various reductive cyclization processes due to its easy availability, environmental safety, low cost and water-tolerant catalyst.[9] In 2008, Kundu and co-workers reported the synthesis of 6*H*-indolo[2,3-*b*]quinoline derivatives from 2-substituted nitroarenes under refluxing conditions in methanol by using $SnCl_2 \cdot 2H_2O$.[8d] However, in this method, the preparation of starting materials involved multiple steps.

Most of the known procedures are associated with several shortcomings with respective to the formation

of side products, use of toxic solvents, tedious procedures of work-up and use of expensive catalysts. Hence, the development of a simple, convenient and environmentally benign method for the synthesis of 6*H*-indolo[2,3-*b*]quinoline derivatives is still a welcoming topic in organic synthesis.

On the other hand, domino reactions have emerged as an effective tool for the assembly of complex cyclic structures by the combination of two or more distinct reactions into a one-pot transformation.[10] Recently, we have developed several domino reactions toward the fused heterocycles system.[11] In continuation of this project, we now report the 6*H*-indolo[2,3-*b*]quinolines **3** preparation *via* domino reaction from 2-nitrobenzaldehyde **1** with indoles **2**, as a stable and inexpensive starting material, providing indole-fused quinolines by $SnCl_2 \cdot 2H_2O$ as a promoter in [bmim]Br medium (Scheme 1).



Scheme 1. Synthesis of 6*H*-indolo[2,3-*b*]quinoline derivatives

In our initial study, various reaction conditions including solvents and temperatures were tested in the synthesis of 11-(1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline **3a** from indole **1a** and 2-nitro- benzaldehyde **2a** using $SnCl_2 \cdot 2H_2O$ as a promoter. Different solvents, such as EtOH, MeCN, AcOH, and ionic liquids ([bmim]Br, [bmim]BF_4) were examined, respectively. The results are summarized in Table 1. The best result was obtained when the reaction was carried out in [bmim]Br at 80 °C in 76% yield.

When the same reactions were performed at room temperature for 2 h, only a very trace amount of product **3a** was formed, the corresponding bis(indolyl)methane was isolated in 85%. In the experimental procedure, indole and aldehydes underwent smooth transformation (3-6 h) to the corresponding bis-(indolyl)methanes in ionic liquid in good yields.[12] However, in the absence of ionic liquid, the reaction did not yield any product even after a long reaction period (10-15 h).

In addition, heating the solution of **1a** and **2a** in an acetic acid and concentrated hydrochloric acid (v/v,1:2) at reflux for 12 h using commonly used zinc dust as the reductant led to an unexpected conjugated tetracyclic product **3a** in 52% yield. Iron powder was less active for this transformation (20%).

Table 1. Optimization of reaction conditions on the synthesis of **3a***

Entry	Catalyst / (eq.)	Solvent	Temp (°C)	Time (h)	Yield (%)
1	$SnCl_2·2H_2O$ (1.0)	EtOH	80	24	43
2	$SnCl_2·2H_2O$ (1.0)	MeCN	80	24	36
3	$SnCl_2·2H_2O$ (1.0)	AcOH	120	18	61
4	$SnCl_2·2H_2O$ (1.0)	[bmim]Br	80	4	76
5	$SnCl_2·2H_2O$ (1.0)	[bmim]Br	100	4	75
6	$SnCl_2·2H_2O$ (1.0)	[bmim]Br	120	2	63
7	$SnCl_2·2H_2O$ (1.0)	[bmim]BF_4	80	5	70
8	$SnCl_2·2H_2O$ (0.8)	[bmim]Br	80	8	72
9	$SnCl_2·2H_2O$ (1.2)	[bmim]Br	80	4	73
10	Zn-HCl (1.5)	HOAc	80	12	52
11	Zn-HCl (1.5)	[bmim]Br	80	10	55
12	Fe-HCl (1.5)	HOAc	80	10	20

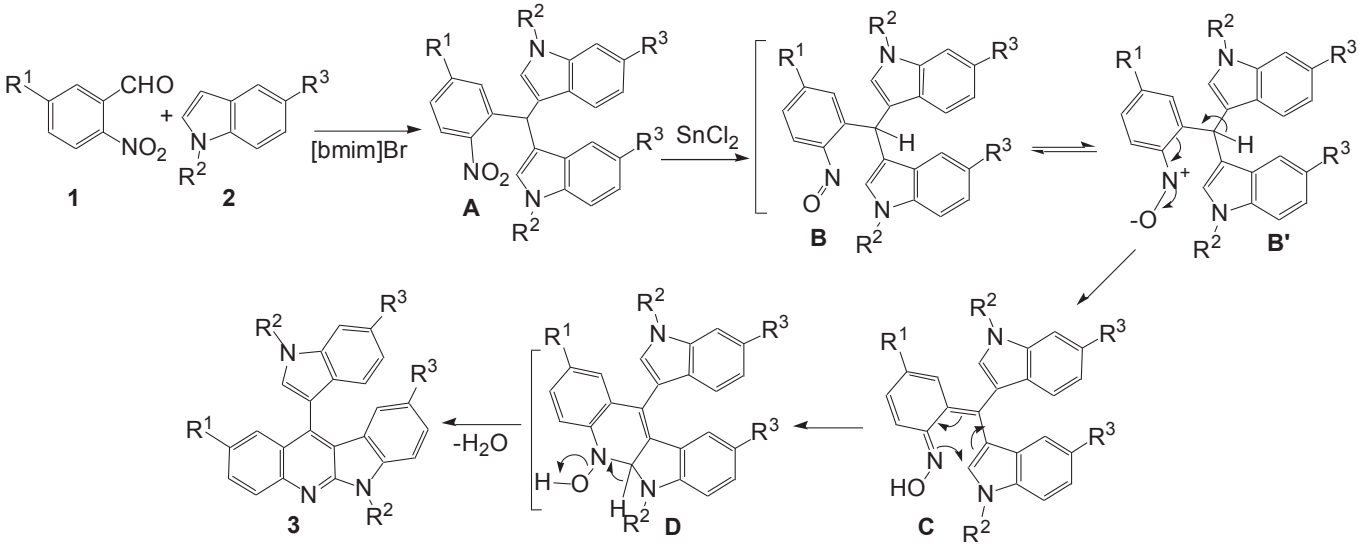
*Reaction conditions: indole (**1**, 2.0 mmol), 2-nitrobenzaldehyde (**2a**, 1.0 mmol), solvent (5 mL).

Under these optimized reaction conditions, a series of 6*H*-indolo[2,3-*b*]quinoline derivatives **3** were synthesized. As shown in Table 2, the reaction was successful for 2-nitrobenzaldehyde **1** and indoles **2** incorporating R groups carrying either electron-donating or electron-withdrawing substituents reacted efficiently giving good yields (70-83%).

Table 2. Synthesis of 6*H*-indolo[2,3-*b*]quinolines **3**

Entry	R^1 (**1**)	R^2/ R^3 (**2**)	Time (h)	Product (**3**)	Yield (%)
1	**1a** H	**2a** H/H	4	**3a**	76
2	**1b** Cl	**2a** H/H	5	**3b**	70
3	**1a** H	**2b** Me/H	4	**3c**	80
4	**1b** Cl	**2b** Me/H	5	**3d**	83
5	**1a** H	**2c** H/Br	7	**3e**	76
6	**1b** Cl	**2d** H/OMe	5	**3f**	78
7	**1a** H	**2e** H/CN	6	**3g**	75
8	**1b** Cl	**2f** H/Br	7	**3h**	74

The proposed mechanism of the process is summarized in Scheme 2. Firstly, 2-nitrobenzaldehyde **1** reacts with indole **2** to generate bis(indolyl)methane **A** *via* alkylation of indole in the presence of $SnCl_2$ as cartalyst.[12,13] Meanwhile, intermediate **B** could be obtained *via* a reduction of the nitro gruop by $SnCl_2$. Subsequent prototropy to **C** initiates a 6 π electrocyclization to **D** from which the products **3** result following dehydration (Scheme 2).



Scheme 2. Proposed mechanism for the synthesis of indolo[2,3-*b*]quinolines **3**

In summary, we have demonstrated the efficient method for the synthesis of 6*H*-indolo[2,3-*b*]quinolines from 2-nitrobenzaldehyde and indoles through alkylation–reduction–cyclization domono reaction. This approach offers an effective route for the construction of indolo[2,3-*b*]quinoline frameworks in a one-step process from commercially available starting materials.

EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. The NMR spectra were recorded with a Bruker Avance 400 spectrometer (400 MHz for ^{1}H and 100 MHz for ^{13}C) using TMS an internal reference. IR spectra were measured on Shimadzu FTIR-8300 spectrophotometer. C, H and N analyses were performed by a HP-MOD 1106 microanalyzer.

Typical Procedure for the Preparation of 11-(1*H*-Indol-3-yl)-6*H*-indolo[2,3-*b*]quinolines. To a solution of 2-nitrobenzaldehydes (**1**, 1.0 mmol) and indoles (**2**, 2.0 mmol) in [bmim]Br (5.0 mL) $SnCl_2 \cdot 2H_2O$ (1.0 mmol) was added, and the reaction was heated at 80 °C. After completion monitored by TLC, the reaction mixture was allowed to cool to room temperature, and then water (20 mL) was added to the mixture. EtOAc (50 mL) was added to the mixture. The organic layer was washed with brine (50 mL), dried over anhydrous Na_2SO_4 and evaporated to dryness. The residue was recrystallized from isopropanol to afford the corresponding products **3a-h**.

11-(1*H*-Indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3a):[8h] Yellow crystals. mp 260-262 °C; IR (KBr): *v* 3325, 3052 1602, 1590 cm^{-1}; ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.21 (dd, *J* = 6.8, 7.2 Hz, 1H), 7.42 (d, *J* = 8.8 Hz, 1H), 7.61-7.69 (m, 2H), 7.74 (dd, *J* = 8.0, 8.2 Hz, 1H), 7.77-7.82 (m, 4H), 8.04-8.05 (m, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 8.42 (d, *J* = 8.4 Hz, 1H), 8.54 (d, *J* = 7.6 Hz, 1H), 11.1 (s, 1H), 12.0 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 107.8, 112.3, 112.5, 119.7, 119.9, 120.0, 120.5, 121.7, 121.8, 122.2, 124.9, 125.9, 126.1, 126.2, 127.1, 127.3, 129.6, 129.8, 132.0, 137.0, 144.4, 144.6, 145.6. *Anal*. Calcd for $C_{23}H_{15}N_3$: C 82.86, H 4.54, N 12.60. Found: C 82.91, H 4.57, N 12.64.

2-Chloro-11-(1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3b):[8h] Yellow crystals. mp > 300 °C; IR (KBr): *v* 3341, 3024, 1607, 1595 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.04-7.05 (m, 1H), 7.17-7.26 (m, 3H), 7.56-7.65 (m, 4H), 7.93-7.96 (m, 2H), 8.27 (d, *J* = 8.4 Hz, 1H), 8.38 (d, *J* = 7.6 Hz, 1H), 11.1 (s, 1H), 11.9 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 107.2, 112.4, 112.7, 119.7, 119.8, 120.0, 120.3, 121.7, 121.9, 122.3, 124.4, 126.6, 126.9, 127.1, 127.4, 129.5, 130.2, 131.7, 132.5, 137.0, 142.7, 144.8, 146.2. *Anal*. Calcd for $C_{23}H_{14}ClN_3$: C 75.10, H 3.84, N 11.42. Found: C 75.17, H 3.86, N 11.46.

6-Methyl-11-(1-methy-1*H*-lindol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3c):[8a] Yellow crystals. mp 261-263 °C; IR (KBr): *v* 3031, 1613, 1597 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.44 (s, 3H), 4.16 (s, 3H), 7.43 (dd, *J* = 7.6, 8.0 Hz, 1H), 7.49 (dd, *J* = 7.4, 8.0 Hz, 1H), 7.57 (dd, *J* = 7.6, 8.0 Hz, 1H), 7.69-7.72 (m, 3H), 7.83-7.88 (m, 3H), 7.89-7.92 (m, 2H), 8.42 (d, *J* = 7.6 Hz, 1H), 8.59 (d, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 31.4, 33.3, 106.9, 110.0, 110.8, 119.5, 119.8, 120.2, 120.3, 121.3, 121.6, 122.3, 125.4, 125.6, 126.5, 128.0, 129.4, 129.6, 130.3, 131.0, 132.6, 136.7, 143.8, 145.7, 145.9. *Anal*. Calcd for $C_{25}H_{19}N_3$: C 83.08, H 5.30, N 11.63. Found: C 83.16, H 5.37, N 11.68.

2-Chloro-6-methyl-11-(1-methyl-1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3d): Yellow crystals. mp > 300 °C; IR (KBr): *v* 3061, 1606, 1591 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.23 (s, 3H), 3.98 (s, 3H), 6.99-7.05 (m, 2H), 7.26 (d, *J* = 7.2 Hz, 1H), 7.32 (d, *J* = 7.2 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.58-7.66 (m, 4H), 7,74 (s, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 8.38 (d, *J* = 6.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 31.3, 33.3, 106.2, 110.2, 110.9, 119.4, 120.5, 120.5, 121.1, 121.8, 122.5, 123.9, 125.5, 125.7, 126.9, 128.8, 129.4, 130.0, 130.6, 131.2, 131.5, 136.7, 142.1, 146.1, 146.2. *Anal*. Calcd for $C_{25}H_{18}ClN_3$: C 75.85, H 4.58, N 10.61. Found: C 75.92, H 4.61, N 10.64.

9-Bromo-11-(5-bromo-1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3e): Yellow crystals. mp > 300 °C; IR (KBr): *v* 3394, 3026, 1602, 1584 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (s, 1H), 7.30 (d, *J* = 8.4 Hz, 1H), 7.48-7.49 (m, 2H), 7.64-7.66 (m, 4H), 7.95 (s, 1H), 8.25 (d, *J* = 8.4 Hz, 1H), 8.47 (s, 1H), 11.1 (s, 1H), 12.0 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 107.2, 111.7, 112.7, 114.3, 114.6, 120.3, 121.8, 123.7, 123.9, 124.8, 125.5, 125.7, 126.3, 126.7, 128.8, 129.0, 129.7, 132.2, 132.4, 135.7, 143.2, 144.3, 144.5. *Anal.* Calcd for $C_{23}H_{13}Br_2N_3$: C 56.24, H 2.67, N 8.55. Found: C 56.32, H 2.69, N 8.59.

2-Chloro-9-methoxy-11-(5-methoxy-1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3f):[8h] Yellow crystals.

mp > 300 °C; IR (KBr): *v* 3343, 3058, 1615, 1595 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): *δ* 3.55 (s, 3H), 3.88 (s, 3H), 6.01 (s, 1H), 7.88 (d, *J* = 6.8 Hz, 1H), 7.22 (d, *J* = 6.4 Hz, 1H), 7.45-7.50 (m, 3H), 7.59 (d, *J* = 8.2 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.94 (s, 1H), 8.23 (d, *J* = 7.4 Hz, 1H) 10.8 (s, 1H), 11.7 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 55.6, 56.0, 101.2, 103.4, 107.0, 112.7, 113.4, 119.8, 119.9, 120.3, 121.8, 124.4, 126.5, 127.3, 127.8, 128.2, 129.2, 131.7, 132.0, 132.8, 139.5, 142.5, 146.0, 153.9, 154.3. *Anal*. Calcd for $C_{25}H_{18}ClN_3O$: C 70.18, H 4.24, N 9.82. Found: C 70.24, H 4.27, N 9.85.

11-(5-Cyano-1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline-9-carbonitrile (3g):[8a] Yellow crystals. mp > 300 °C; IR (KBr): *v* 3352, 3028, 2224, 2209, 1605, 1584 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): *δ* 7.34 (d, *J* = 8.4 Hz, 1H), 7.41-7.44 (m, 2H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.61-7.63 (m, 3H), 7.79 (s, 1H), 7.98 (s, 1H), 8.14 (d, *J* = 8.4 Hz, 1H), 8.39 (s, 1H), 11.2 (s, 1H), 12.1 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 101.6, 102.3, 108.2, 113.3, 113.9, 120.4, 120.5, 120.9, 122.1, 125.1, 125.4, 125.6, 126.1, 126.4, 126.7, 127.0, 127.2, 129.8, 130.1, 132.5, 132.9, 138.8, 144.4, 144.9, 146.3. *Anal.* Calcd for $C_{25}H_{13}N_5$: C 78.32, H 3.42, N 18.27. Found: C 78.39, H 3.46, N 18.29.

2-Chloro-11-(1*H*-indol-3-yl)-6*H*-indolo[2,3-*b*]quinoline (3h): Yellow crystals. mp > 300 °C; IR (KBr): *v* 3317, 3046, 1613, 1580 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): *δ* 7.53 (dd, *J* = 7.6, 8.4 Hz, 1H), 7.58-7.61 (m, 2H), 7.65 (s, 1H), 7.69 (dd, *J* = 7.6, 8.0 Hz, 1H), 7.77 (dd, *J* = 7.6, 8.4 Hz, 1H), 7.87-7.92 (m, 2H), 7.79 (s, 1H), 8.10 (s, 1H), 8.26 (d, *J* = 7.8 Hz, 1H), 8.39 (s, 1H), 11.49 (s, 1H), 12.39 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 106.6, 111.9, 112.9, 114.3, 114.7, 119.4, 121.8, 123.4, 124.0, 124.9, 126.8, 127.0, 128.9, 129.0, 130.0, 131.7, 132.4, 132.7, 135.7, 142.6, 143.2, 144.7, 170.7. *Anal.* Calcd for $C_{23}H_{12}Br_2ClN_3$: C 52.56, H 2.30, N 7.99. Found: C 52.63, H 2.33, N 8.04.

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