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Novel multicomponent synthesis of 2,9-dihydro-9-methyl-2-oxo-4-aryl-1*H*-pyrido[2,3-*b*]indole-3-carbonitrile compounds

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Abstract. Novel multicomponent approach for the synthesis of 2,9-dihydro-2-oxo-4-aryl-1*H*-pyrido[2,3-*b*]indole-3-carbonitrile derivatives via one-pot cyclocondensation reaction of substituted (triethoxymethyl)arene, 1-methyl-1*H*-indol-2-ol and cyanoacetamide in the presence of silica supported ionic liquid [pmim]$HSO_{4\,SiO2}$ (silica-supported 1-methyl-3-(triethoxysilylpropyl)imidazolium hydrogensulphate) has been reported.

Keywords. Pyrido[2,3-*b*]indole; multicomponent; silica supported ionic liquid.

1. Introduction

Following the discovery of α-carboline structure and its cytotoxic activity toward L1210 leukemia cells,[1] in the recent years, there has been interest in the synthesis of functionalized pyrido[2,3-*b*]indoles. Synthetic α-carboline compounds have also been found to exhibit antiviral and antitumour activities.[2–4] Moreover, pyrido[2,3-*b*]indoles have been known as the biological control agent for receptor research on bio-enzyme inhibitors, such as the inhibition of HLE (human leukocyte elastase).[5–8]

Synthetic approaches for this class of compounds mostly involve construction of either pyridine ring from 2-amino-3-substituted indole derivatives[9,10] or synthesis of pyrrole ring via cross coupling between an appropriately substituted pyridine and aniline derivatives.[11,12] In addition, synthesis of pyrido[2,3-*b*]indole derivatives via condensation of 3-substituted indole-2(3*H*)-one derivatives and enamines followed by thermal cyclization with ammonium acetate has been reported.[13,14] Other approaches involve intramolecular Diels–Alder reaction of 2(1*H*)-pyrazinones and conjugated carbodiimides.[15,16]

In the recent years, ionic liquids have become a powerful alternative to conventional molecular organic solvents due to their particular properties, such as undetectable vapour pressure and the ability to dissolve many organic and inorganic substances.[17] Among these, ionic liquids possessing HSO_4 as a counteranion find a broad application in organic synthesis, acting as both solvents and catalysts. Recently, immobilization processes involving acidic ionic liquids on solids supports have been designed.[18–20] The heterogenization of catalysts and reagents can offer important advantages in handling, separation and reuse procedures. Based on economic criteria, it is desirable to minimize the amount of ionic liquid utilized in a potential process. Immobilized acidic ionic liquids have been used as novel solid catalysts for a wide spectrum of reactions.[21,22]

In continuation of our ongoing research in developing new multicomponent synthetic approach,[23–27] we considered it to be of interest to devise a facile, efficient, versatile and one-pot synthetic method for construction of pyrido[2,3-*b*]indoles utilizing easily accessible 1-methyl-1*H*-indol-2-ol, substituted (triethoxymethyl)arene, and cyanoacetamide in the presence of immobilized [pmim]$HSO_{4\,SiO2}$ being a silica-supported ionic liquid with acidic counteranion of HSO_4^- (figures 1 and 2). Here, we report the details of this study.

2. Experimental

2.1 *General*

All Chemicals and accessible ionic liquids were purchased from Merck, Fluka and Aldrich Chemical

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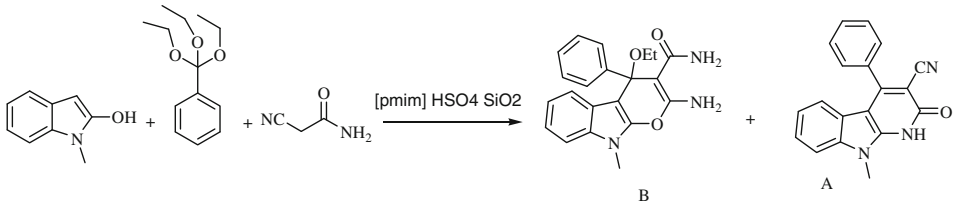


	time (h)	B:A ratio
MeOH, 70 °C	3.0	10:0
	5.0	10:0
	8.0	10:0
DMF, 110 °C,	1.5	8:2
	2.0	6:4
	4.0	1:9
	5.0	1:9

Figure 1. One-pot synthesis of pyrido[2,3-*b*]indoles.

Companies. All yields refer to isolated products. The products were characterized by their spectral data. IR spectra were recorded on a Shimadzu-IR 470 spectrophotometer. ^{1}H NMR spectra were recorded on a Bruker 100-MHz spectrometer in chloroform as the solvent and TMS as internal standard. Elemental analysis (C, H, N %) was carried out by Perkin-Elmer 2400 series-II elemental analyzer. [pmim]$HSO_{4\,SiO2}$ (extent of labelling 0.25 mol/g loading) was prepared according to the literature.[28]

2.2 *General procedure for synthesis of compounds **IV(a–k)** and **III***

A mixture of substituted (triethoxymethyl)arene (1 mmol), 3-hydroxyindole and cyanoacetamide (1.1 mmol), 1-methyl-1*H*-indol-2-ol (1 mmol) and ionic liquid catalyst (0.15 mmol) in appropriate solvent (DMF or methanol) (6 mL) was stirred for requisite time. The reaction was monitored by TLC, and after completion of the reaction, the catalyst was simply recovered by filtration and washed by dichloromethane. The solvent was removed from filtrate by distillation and crude product was purified by column chromatography on silica gel.

2.2a *2-Amino-4-ethoxy-4,9-dihydro-9-methyl-4-phenylpyrano[2,3-b]indole-3-carboxamide (**III**)*: IR (KBr, ν_{max}, cm^{-1}): 3260, 3144, 2979, 2835, 1660, 1578, 1462, 1220 cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6): δ = 3.55 (3H, s, indole), 10.85 (1H, s, -NHCO-), 7.65 (1H, d, J = 7.2 Hz, ArH), 7.36 (1H, d, J = 7.2 Hz,

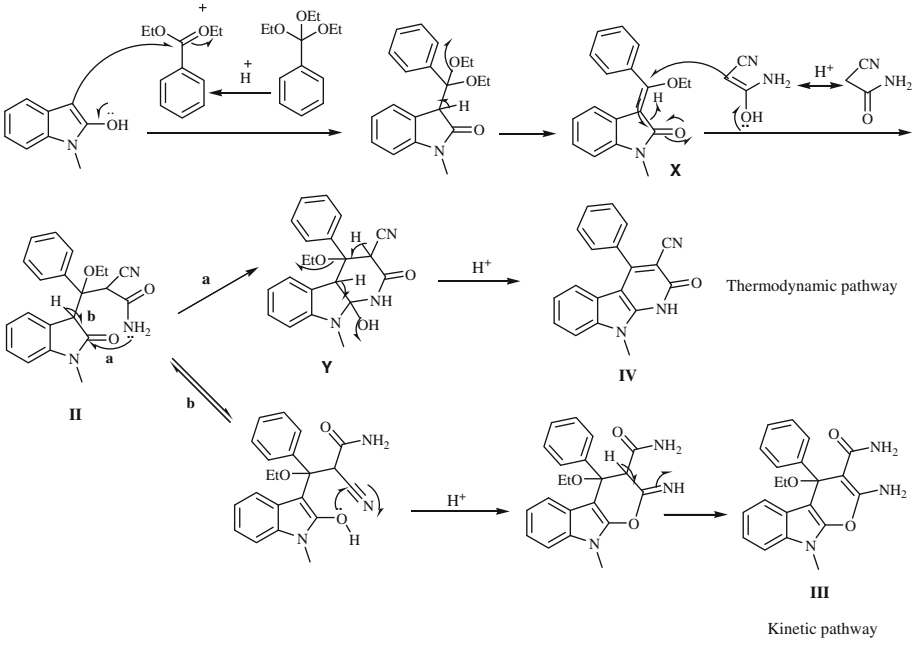


Figure 2. Proposed mechanism for one-pot three-component synthesis of pyrido[2,3-*b*]indole derivatives catalysed by [pmim] $HSO_{4\,SiO2}$.

ArH), 7.30–7.15 (8H, m, ArH), 7.05 (2H, d, J = 7.4 Hz, ArH), 3.73 (q, J = 12.5 Hz, O-CH_2-), 1.35 (t, J = 12.5 Hz, -CH_3). Anal. Calcd for $C_{21}H_{21}N_3O_3$: C, 69.41; H, 5.82; N, 11.56%. Found: C, 69.22; H, 5.79; N, 11.55%.

2.2b *2,9-Dihydro-4-(4-methoxyphenyl)-9-methyl-2-oxo-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–a)***: IR (KBr, ν_{max}, cm^{-1}): 3255, 3110, 2955, 2830, 2235, 1645, 1555, 1440, 1133 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.85 (1H, s, -NHCO-), 7.60–7.35 (2H, m, ArH), 7.28 (2H, d, J = 7.1 Hz, ArH), 7.12 (2H, d, J = 7.4 Hz, ArH), 6.95 (2H, d, J = 7.1 Hz, ArH), 3.80 (s, OCH_3), 3.65 (3H, s, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 165.8, 155.1, 150.4, 135.8, 128.2, 126.3, 124.6, 124.4, 124.2, 122.1, 121.4, 119.6, 115.3, 111.9, 112.6, 99.8, 55.8, 35.2. Anal. Calcd for $C_{20}H_{15}N_3O_2$: C, 72.94; H, 4.59; N, 12.76%. Found: C, 72.56; H, 4.55; N, 12.73%.

2.2c *2,9-Dihydro-9-methyl-2-oxo-4-phenyl-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–b)***: IR (KBr, ν_{max}, cm^{-1}): 3250, 3120, 2965, 2833, 2230, 1642, 1551, 1431 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 11.02 (1H, s, -NHCO-), 7.55 (1H, d, J = 6.3 Hz, ArH), 7.43 (1H, d, J = 6.3 Hz, ArH), 7.33–7.15 (7H, m, ArH), 3.62 (3H, s, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 169.3, 155.2, 138.5, 129.2, 125.5, 124.2, 123.7, 122.47 120.4, 118.5, 118.0, 117.5, 116.1, 114.6, 110.8, 96.2, 34.5. Anal. Calcd for $C_{19}H_{13}N_3O$: C, 76.24; H, 4.38; N, 14.04%. Found: C, 75.90; H, 4.34; N, 14.42%.

2.2d *2,9-Dihydro-9-methyl-2-oxo-4-p-tolyl-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–c)***: IR (KBr, ν_{max}, cm^{-1}): 3254, 3119, 2976, 2835, 2218, 1647, 1551, 1422 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.80 (1H, s, -NHCO-), 7.46 (1H, d, J = 7.1 Hz, ArH), 7.57 (1H, d, J = 7.1 Hz, ArH), 7.30–7.12 (6H, m, ArH), 3.52 (s, -CH_3, indole), 2.12 (s, -CH_3). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 161.9, 156.1, 139.6, 135.5, 129.3, 128.7, 127.1, 125.9, 125.3, 124.0, 121.8, 120.1, 117.8, 115.1, 112.9, 95.2, 34.6, 24.9. Anal. Calcd for $C_{20}H_{15}N_3O$: C, 76.66; H, 4.82; N, 13.41%. Found: C, 76.49; H, 4.78; N, 13.38%.

2.2e *4-(4-Bromophenyl)-2,5-dihydro-9-methy-2-oxo-1H-pyrido[3,2-b]indole-3-carbonitrile* ***(IV–d)***: IR (KBr, ν_{max}, cm^{-1}): 3246, 3125, 2983, 2826, 2217, 1656, 1509, 1452 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.90 (1H, s, -NHCO-), 7.57 (1H, d, J = 6.2 Hz, ArH), 7.40–7.30 (3H, m, ArH), 7.15 (2H, d, J = 6.4 Hz, ArH), 7.80 (2H, d, J = 6.4 Hz, ArH), 3.55 (s, -CH_3, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 168.5, 160.7, 139.2, 132.3, 129.5, 128.3, 127.9, 124.8, 124.1, 122.6, 121.8, 121.6, 117.3, 116.5, 111.3, 96.6, 32.7. Anal. Calcd for $C_{19}H_{12}BrN_3O$: C, 60.34; H, 3.20; N, 11.11%. Found: C, 60.21; H, 3.14; N, 11.05%.

2.2f *4-(2-Bromophenyl)-2,5-dihydro-9-methy-2-oxo-1H-pyrido[3,2-b]indole-3-carbonitrile* ***(IV–e)***: IR (KBr, ν_{max}, cm^{-1}): 3255, 3134, 2991, 2806, 2228, 1650, 1544, 1487 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.57 (1H, s, -NHCO-), 7.60 (1H, d, J = 7.0 Hz, ArH), 7.45–7.37 (2H, m, ArH), 7.22–7.14 (5H, m, ArH), 3.63 (s, -CH_3, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 163.3, 155.2, 139.7, 135.5, 132.1, 129.8, 125.9, 125.2, 124.6, 123.3, 121.9, 121.0, 118.8, 118.6, 118.1, 115.7, 94.1, 31.6. Anal. Calcd for $C_{19}H_{12}BrN_3O$: C, 60.34; H, 3.20; N, 11.11%. Found: C, 60.27; H, 3.13 N, 11.07%.

2.2g *4-(4-Chlorophenyl)-2,9-dihydro-9-methyl-2-oxo-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–f)***: IR (KBr, ν_{max}, cm^{-1}): 3263, 3115, 2980, 2802, 2212, 1652, 1535, 1470 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 11.02 (1H, s, -NHCO-), 7.55 (1H, d, J = 7.5 Hz, ArH), 7.46 (1H, d, J = 7.7 Hz, ArH), 7.25–7.18 (4H, m, ArH), 7.04 (2H, d, J = 7.7 Hz, ArH), 3.55 (s, -CH_3, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 166.4, 161.1, 138.9, 135.3, 129.7, 127.4, 124.93, 124.7, 123.6, 122.1, 121.5, 120.5, 118.8, 116.7, 111.1, 93.6, 33.1. Anal. Calcd for $C_{19}H_{12}ClN_3O$: C, 68.37; H, 3.62; N, 12.59%. Found: C, 67.91; H, 3.53; N, 12.51%.

2.2h *4-(2-Chlorophenyl)-2,9-dihydro-9-methyl-2-oxo-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–g)***: IR (KBr, ν_{max}, cm^{-1}): 3250, 3142, 2997, 2812, 2211, 1667, 1553, 1465 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.92 (1H, s, -NHCO-), 7.50 (1H, d, J = 6.5 Hz, ArH), 7.43 (1H, d, J = 6.5 Hz, ArH), 7.30–7.24 (2H, m, ArH), 7.20–7.05 (4H, m, ArH), 3.60 (s, -CH_3, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 169.5, 158.4, 139.7, 134.4, 131.1, 128.9, 125.5, 124.2, 122.9, 122.1, 120.8, 120.1, 118.9, 118.7, 116.9, 116.3, 98.5, 34.5. Anal. Calcd for $C_{19}H_{12}ClN_3O$: C, 68.37; H, 3.62; N, 12.59%. Found: C, 67.84; H, 3.55; N, 12.49%.

2.2i *2,9-Dihydro-9-methyl-4-(4-nitrophenyl)-2-oxo-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–i)***: IR (KBr, ν_{max}, cm^{-1}): 3255, 3175, 2995, 2810, 2217, 1637, 1552, 1516, 1481, 1345 cm^{-1}. ^{1}H-NMR (400 MHz,

DMSO-d_6): δ = 10.85 (1H, s, -NHCO-), 8.09 (2H, d, J = 7.2 Hz, ArH), 7.65–7.50 (3H, m, ArH), 7.40 (1H, d, J = 7.5 Hz, ArH), 7.10 (2H, d, J = 7.7 Hz, ArH), 3.62 (s, -CH_3, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 167.7, 159.2, 154.1, 148.3, 144.4, 129.9, 127.4, 125.9, 125.2, 123.3, 122.1, 121.7, 120.4, 119.5, 115.6, 114.1, 95.5, 34.6. Anal. Calcd for $C_{19}H_{12}N_4O_3$: C, 66.28; H, 3.51; N, 16.27%. Found: C, 66.01; H, 3.42; N, 16.16%.

2.2j *4-(4-Fluorophenyl)-2,9-dihydro-9-methyl-2-oxo-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–j)***: IR (KBr, ν_{max}, cm^{-1}): 3240, 3155, 2985, 2795, 2236, 1656, 1540, 1477 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 11.12 (1H, s, -NHCO-), 7.60 (1H, d, J = 6.9 Hz, ArH), 7.52 (1H, d, J = 7.1 Hz, ArH), 7.44 (2H, d, J = 6.9 Hz, ArH), 7.20–6.95 (4H, m, ArH), 3.54 (s, -CH_3, indole). ^{13}C NMR (250 MHz, DMSO-d_6): δ = 168.67 164.6, 140.1, 135.6, 129.5, 127.2, 124.9, 122.7, 122.1, 121.8, 1205, 120.2, 119.4, 117.4, 114.2, 96.7, 32.8. Anal. Calcd for $C_{19}H_{12}FN_3O$: C, 71.92; H, 3.81; N, 13.24%. Found: C, 71.38; H, 3.73; N, 13.18%.

2.2k *2,9-Dihydro-9-methyl-4-(naphthalen-1-yl)-2-oxo-1H-pyrido[2,3-b]indole-3-carbonitrile* ***(IV–k)***: IR (KBr, ν_{max}, cm^{-1}): 3265, 3140, 2990, 2866, 2234, 1651, 1568, 14545 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.80 (1H, s, -NHCO-), 7.65–7.50 (4H, m, ArH), 7.40–7.25 (5H, m, ArH), 7.05 (2H, d, J = 7.4 Hz, ArH), 3.45 (s, -CH_3, indole). ^{13}C-NMR (250 MHz, DMSO-d_6): δ = 169.6, 159.5, 141.4, 133.1, 128.2, 128.0, 126.3, 125.1, 124.6, 124.4, 124.6, 124.2, 123.7, 122.1, 121.4, 119.6, 115.3, 111.9, 112.6, 99.8, 32.9. Anal. Calcd for $C_{23}H_{15}N_3O$: C, 79.07; H, 4.33; N, 12.03%. Found: C, 78.91; H, 4.26; N, 11.95%.

3. Results and discussion

In order to study the feasibility of the synthesis of pyrido[2,3-*b*]indoles, a 15% mol of the immobilized

Table 1. Results of one-pot three component synthesis of pyrido[2,3-*b*]indoles.[a]

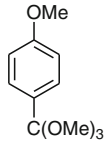
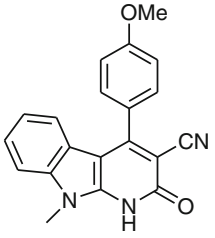
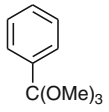
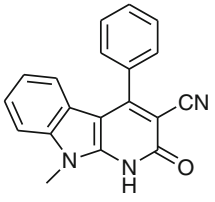
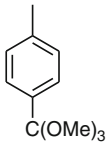
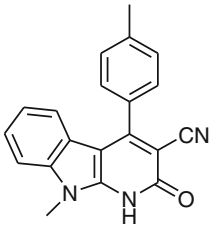
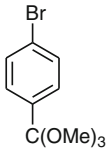
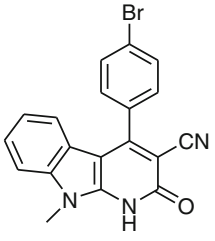
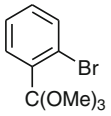
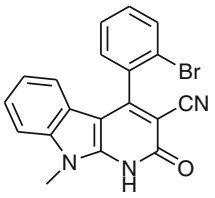
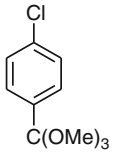
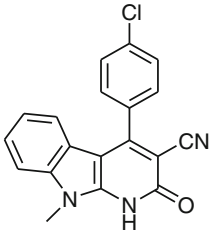
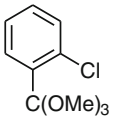
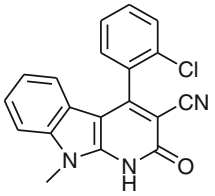
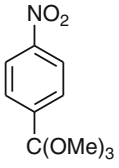
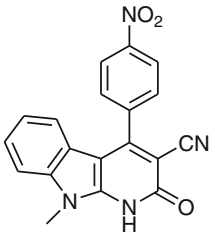
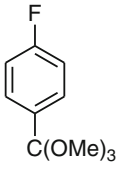
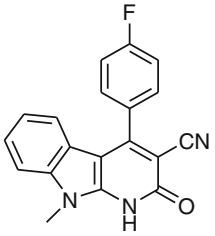
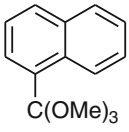
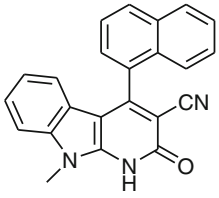
Entry	(triethoxymethyl)arene	Product[b]	Time (h)	Yield (%)[c]
1			2	73
2			3	65
3			3	65
4			4	61

Table 1. (continued).

Entry	(triethoxymethyl)arene	Product[b]	Time (h)	Yield (%)[c]
5			4	56
6			6	61
7			6	55
8			11	42
9			7	53
10			2	61

[a]Reaction conditions: 1.0 equiv. of 1-methyl-1*H*-indol-2-ol, 1.0 equiv. of substituted (triethoxymethyl)arene, 1.1 equiv. of cyanoacetamide, 15 mol% of [pmim] $HSO_{4\,SiO2}$, 6 mL of DMF as solvent and at 100°C. [b]The products were identified by NMR and IR spectrometer. [c]Isolated yields

catalyst of [pmim]$HSO_{4\,Sio2}$ along with a mixture of (triethoxymethyl)benzene, 1-methyl-1*H*-indol-2-ol and cyanoacetamide were chosen as the model reaction and examined for the synthesis of 2,9-dihydro-9-methyl-2-oxo-4-phenyl-1*H*-pyrido[2,3-*b*]indole-3-carbonitrile (**A**) (figure 1). The reaction was carried out in methanol under reflux condition. Surprisingly, pyrano[2,3-*b*]indole (**B**) was produced as the main product. Moreover, when the reaction mixture was refluxed in ethanol, **A:B** molar ratio was independent on reaction time and **B** was the single product. Next, we carried out the model reaction in different solvent having higher boiling points to

synthesis pyrido[2,3-*b*]indole and DMF was found to be the best solvent for this transformation. In other words, when the model reaction was carried out using DMF at reflux condition, the corresponding pyrido[2,3-*b*]indole was obtained as the major product and **A:B** molar ratio was dependent on reaction time.

Initially, **B** was major product, but the amount of **A** was enhanced as reaction time became longer. We believe that depending on the reaction temperature, the reaction might be controlled by either kinetic or thermodynamic factors. It sounds that conversion of intermediate (**II**) (figure 2) to **A** is more rapid than **B**.

We proposed a three-step mechanism for one-pot synthesis of pyrido[3,2-*b*]indole derivatives (figure 2). Cross coupling between 3-hydroxyindole and (triethoxymethyl)benzene results in α,β-unsaturated compound (**X**), possessing ethoxy substituent on its β position which can act as leaving group when needed. Nucleophilic attack of cyanoacetamide on β-position of intermediate (**X**) generates the key intermediate (**II**) which can proceed the reaction through either kinetic or thermodynamic pathway. Condensation reaction between N-amide atom and carbonyl group on 2-position of indole leads to pyrido[2,3-*b*]indole (thermodynamic pathway), while regeneration of hydroxyl group on 2-position of indole and its nucleophilic attack on nitrile moiety produces pyrano[2,3-*b*]indoles (kinetic pathway).

To generalize the scope, we extended the model reaction using different derivatives of substituted (triethoxymethyl)arene (table 1). It has revealed that the electronic nature of substituted groups on (triethoxymethyl)arene influenced the reaction times and chemical yields. Additionally, steric effects reduced chemical yields or increased reaction times to some extent (table 1, entries 4–7).

4. Conclusion

Novel one-pot cyclocondensation of substituted (triethoxymethyl)arene, 1-methyl-1*H*-indol-2-ol and cyanoacetamide to afford a series of 2,9-dihydro-2-oxo-4-aryl-1*H*-pyrido[2,3-*b*]indole-3-carbonitrile derivatives efficiently catalysed by silica-supported ionic liquid of [pmim]$HSO_{4\,SiO2}$.

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