Asian Journal of Chemistry; Vol. 35, No. 5 (2023), 1225-1230

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2023.27602

An Efficient, One-Pot Synthesis of 1,3-Disubstituted imidazo[1,5-*a*]pyridines, $Fe(OTf)_3$ Mediated C-C Bond Formation

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Received: 3 February 2023; *Accepted*: 16 April 2023; *Published online*: 28 April 2023; AJC-21232

A vital approach of $Fe(OTf)_3$ and MS3Å (molecular sieves 3Å) mediated one pot synthesis of 1,3-disubstituted imidazo[1,5-*a*]pyridines from dithioester, 2-methylaminopyridine and alcohol. This methodology involves the intramolecular cyclization and C–C bond formation under mild condition and operates in a single step yielding the products in good to excellent yields. This protocol was compatible to construct various 1,3-disubstituted imidazo[1,5-*a*] pyridines derivatives.

Keywords: C-C bond formation, Benzhydrol, 1,3-Disubstituted imidazo[1,5-*a*]pyridine, $Fe(OTf)_3$, T3P.

INTRODUCTION

The term "azaheterocyclic compounds" is used to describe a class of heterocycles that includes 1,3-disubstituted imidazo-[1,5-*a*]pyridines [1,2]. They are important building blocks for the synthesis of important bioconjugates due to their wide spectrum of therapeutic importance and their potential as bioactive agents. The design and construction of new azaheterocyclic ring systems are the need in the drug discovery to achieve specific drug–receptor interactions [3-6]. These imidazo[1,5-*a*]-pyridines are promising building blocks of many therapeutic agents such as tumors [7], thrombosis [8] and Alzheimer's disease [9].

These substituted imidazo[1,5-*a*]pyridines are not only play a crucial role in pharmaceuticals but also play an important role in the field of material sciences [10]. Imidazo[1,5-*a*]pyridines used as cell imaging reagents and fluorescence probes due to their strong fluorescence nature [11-14]. These imidazo[1,5-*a*]-pyridines derivatives were also shown applications in dye sensitized solar cells (DSSCs) [15] and organic light-emitting diodes (OLEDs) [16]. Due to their wide applications in the field of both biological and material science, many synthetic approaches have been reported, some of them are conventional methods like cyclo-dehydrogenation of amides to form imidazo[1,5-*a*]-pyridines derivatives using strong acidic and corrosive reagents like thionyl chloride and $POCl_3$ [17,18].

In continuation of our work on the synthesis of bioactive heterocyclic compounds [19], applications of propanephosphonic acid anhydride (T3P) [20,21] and application of dithioesters [22-25] in organic synthesis, herein, a protocol for the synthesis of 1,3-disubstituted imidazo[1,5-*a*]pyridines from dithioesters using $Fe(OTf)_3$ is reported. Recently, our research group synthesized imidazopyridines (**3**) from dithioester (**2**) and picolamine (**1**) using 2 equiv. of DMSO and 1 equiv. of T3P in THF about 2-4 h of reaction time [26].

EXPERIMENTAL

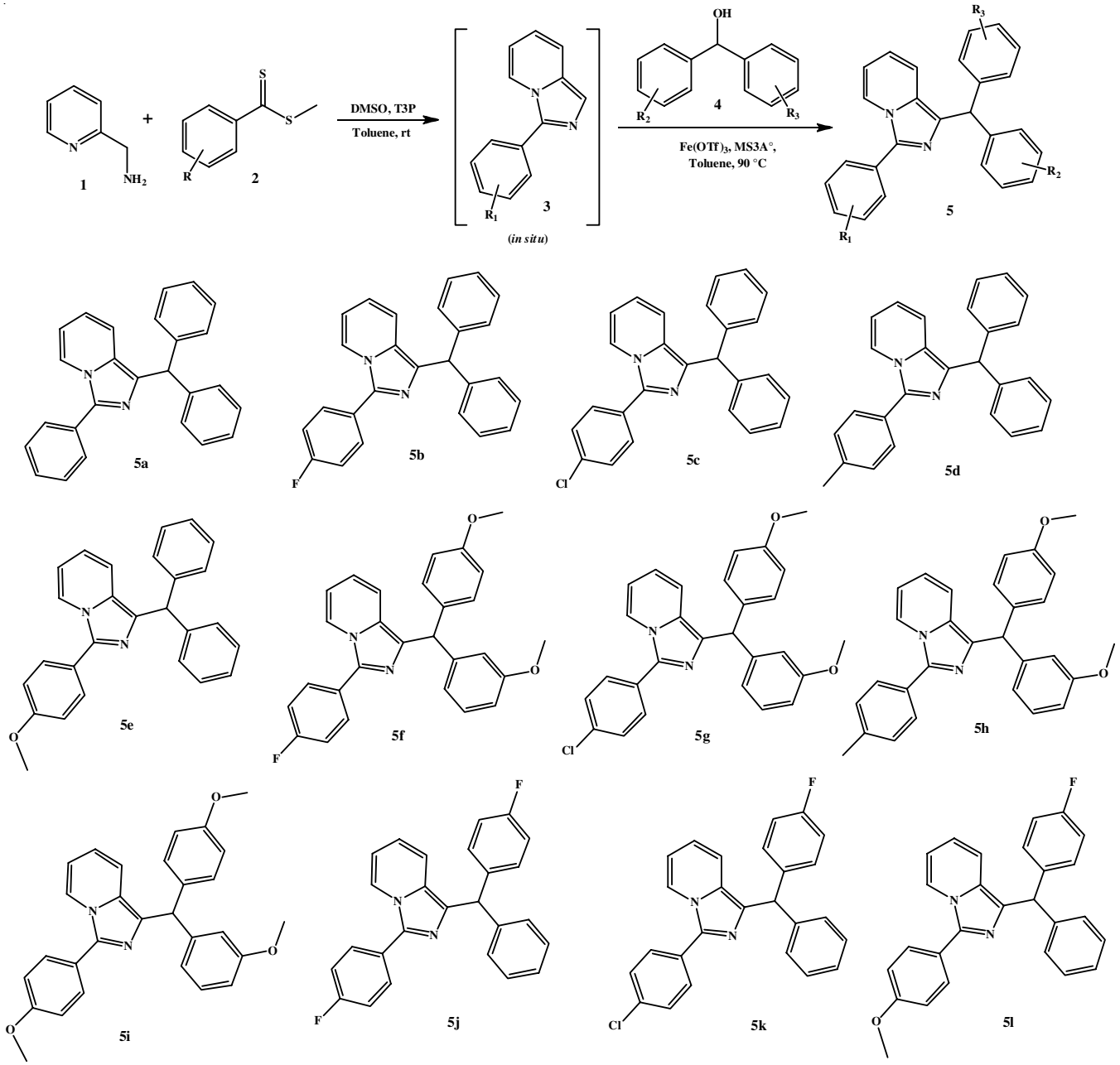
Thin-layer chromatography was performed using Merck silica gel $60F_{254}$ aluminium plates and was visualized under UV light. Analytical grade mobile phases such as chloroform, methanol, hexane, and ethyl acetate with different ratios were employed for TLC. Chemical characterization for newly synthesised compounds was done using ^1H and ^{13}C NMR spectra

were recorded on an Agilent WM (400 MHz), using $CDCl_3$ or DMSO-d_6 solution and tetramethylsilane (TMS) was used as an internal standard. Chemical shifts were recorded in ppm relative to TMS. Mass and purity were recorded on an LC-MSD-Trap-XCT (Agilent Technologies Inc). Fourier transforms spectrophotometer was used to analyses the functional groups and bonding. All the reagents and chemicals procured from Sigma Aldrich, India.

Synthesis: To a solution of dithioester (1 eq) in toluene (8 vol) and picoline amine (1.1 eq) was added; the resulting mixture was stirred for 45 min. The dithioester was no longer detectable when it was monitored by TLC. To the above mixture T3P (1.0 equiv, 50% solution in EtOAc) and DMSO (2.0 eq) were added. To the insitu generated imidazopyridine substituted alcohol (1 eq), $Fe(OTf)_3$ (200 mol%) and MS3Å (1.1 eq) were added and stirred at 90 °C. After the reactant disappeared (monitored by TLC), the reaction mixture was cooled to room temperature and quenched with water. The aqueous layer was extracted with ethyl acetate and then organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The solvent was removed under reduced pressure; the residue was purified by column chromatography using ethyl acetate:pet. ether (1:9) mixture to get pure compound (**Scheme-I**).

The product was purified by recrystallization and column chromatography using ethyl acetate:pet. ether (1:9) mixture to get pure compound.

1-Benzhydryl-3-phenylimidazo[1,5-*a*]pyridine (5a): Brown gummy solid, yield: 380 mg, 89%; m.p.: 143-145 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.19 (d, J = 8 Hz, 1H),



Scheme-I: For the formation of compound **5a-l**

7.49-7.33 (m, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.28 (s, 8H), 7.22 (s, 3H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.48-6.46 (m, 2H), 5.96 (s, 1H). ^{13}C NMR (101 MHz, $CdCl_3$) δ ppm: 143.45, 137.29, 134.57, 130.67, 130.39, 130.18, 129.36, 129.00, 128.66, 128.40, 128.37, 126.45, 121.48, 120.21, 118.72, 118.14, 112.96, 111.36, 77.49, 77.17, 76.85, 50.77.HRMS (ESI); *m/z* calcd. for $C_{26}H_{20}N_2$ $[M + H]^+$: 360.1626, found: 360.1619

1-Benzhydryl-3-(4-fluorophenyl)imidazo[1,5-*a*]-pyridine (5b): Pale yellow solid, yield: 320 mg, 82%; m.p.: 154-156 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.09 (d, *J* = 8 Hz, 1H), 7.74 (d, *J* = 4 Hz, 2H), 7.32 (d, *J* = 4 Hz, 7H), 7.30 (d, *J* = 1.6 Hz, 4H), 7.22 (d, *J* = 8 Hz, 1H), 6.51-6.42 (m, 2H), 5.95 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 164.12, 161.64, 143.35, 136.33, 134.55, 130.34, 130.26, 130.16, 130.07, 129.34, 128.57, 128.44, 126.52, 121.21, 118.76, 118.24, 116.24, 116.02, 114.29, 113.21, 77.53, 77.21, 76.90, 50.72. HRMS (ESI): *m/z* calcd. for $C_{26}H_{19}FN_2$ $[M+H]^+$: 379.1532, found 379.1531.

1-Benzhydryl-3-(4-chlorophenyl)imidazo[1,5-*a*]pyridine (5c): Brown gummy solid, yield: 290 mg, 77%; m.p.: 191-193 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.14 (t, *J* = 4.0 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.47 (s, 2H), 7.32 (m, 8H), 7.30 (s, 2H), 6.93 (d, *J* = 1.2 Hz, 1H), 6.53-6.49 (m, 2H), 5.92 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 143.26, 134.83, 134.28, 129.37, 129.20, 129.13, 128.90, 128.74, 128.32, 126.39, 121.14, 118.67, 118.16, 113.15, 77.36, 77.04, 76.72, 50.62. HRMS (ESI): *m/z* calcd. for $C_{26}H_{19}ClN_2$ $[M+H]^+$: 395.1237, found 395.0759.

1-Benzhydryl-3-(*p*-tolyl)imidazo[1,5-*a*]pyridine (5d): Brown gummy solid, yield: 400 mg, 78%; m.p.: 161-162 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.16-8.14 (m, 1H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 10H), 7.30 (s, 2H), 6.87 (d, *J* = 8.0 Hz, 1H), 6.48-6.44 (m, 2H), 5.94 (s, 1H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 143.42, 138.49, 134.27, 129.56, 129.25, 128.27, 128.15, 127.47, 126.31, 121.44, 118.57, 117.79, 112.62, 77.37, 77.05, 76.73, 50.70, 21.41. HRMS (ESI): *m/z* calcd. for $C_{27}H_{22}N_2$ $[M+H]^+$: 375.1783, found 375.1793.

1-Benzhydryl-3-(4-methoxyphenyl)imidazo[1,5-*a*]-pyridine (5e): Brown gummy solid, yield: 450 mg, 88%; m.p.: 141-142 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.11-8.09 (m, 1H), 7.70-7.68 (m, 2H), 7.32 (d, *J* = 4.0 Hz, 8H), 7.29 (s, 2H), 7.27 (s, 2H), 7.03 (d, *J* = 8 Hz, 1H), 6.48-6.44 (m, 2H), 5.95 (s, 1H), 3.86 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 159.90, 143.31, 137.10, 129.74, 129.25, 128.29, 128.09, 126.34, 121.34, 118.61, 117.81, 114.36, 112.71, 77.37, 77.05, 76.73, 55.39, 50.61. HRMS (ESI): *m/z* calcd. for $C_{27}H_{22}N_2O$ $[M+H]^+$: 391.1732, found 390.9874.

3-(4-Fluorophenyl)-1-((3-methoxyphenyl)(4-methoxy-phenyl)methyl)imidazo[1,5-*a*]pyridine (5f): Brown gummy solid, yield: 420 mg, 93%; m.p.: 220-222 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.08 (d, *J* = 2.8 Hz, 1H), 7.75-7.72 (m, 2H), 7.26 (t, *J* = 8.0 Hz, 4H), 7.19 (t, *J* = 1.6 Hz, 2H), 6.92 (d, *J* = 9.2 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 4H), 6.49-6.45 (m, 2H), 5.82 (s, 1H), 3.87 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 163.91, 161.44, 158.05, 147.98, 136.14, 135.89, 135.16, 132.19, 130.13, 130.02, 128.26, 126.63, 122.17, 121.01, 118.70, 117.79, 116.02, 115.80, 113.66, 112.90, 77.36, 77.04, 76.72, 55.21, 49.02. HRMS (ESI): *m/z* calcd. for $C_{28}H_{23}FN_2O_2$ $[M+H]^+$: 438.1744 found 438.1739.

3-(4-Chlorophenyl)-1-((3-methoxyphenyl)(4-methoxy-phenyl)methyl)imidazo[1,5-*a*]pyridine (5g): Brown gummy solid, yield: 400 mg, 93%; m.p.: 228-230 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.12 (d, *J* = 8.0 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.26 (m, 3H), 6.97 (d, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 3H), 6.52 (t, *J* = 8.0 Hz, 2H), 5.82 (s, 1H).,3.77 (s, 6H), ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 162.83, 158.04, 135.88, 135.75, 135.43, 132.20, 130.01, 129.69, 129.33, 129.07, 121.07, 118.78, 118.03, 113.65, 113.45, 113.14, 77.34, 77.03, 76.71, 55.43, 55.21, 48.97. HRMS (ESI): *m/z* calcd. for $C_{28}H_{23}ClN_2O_2$ $[M+H]^+$: 454.1448, found 454.1439.

1-((3-Methoxyphenyl)(4-methoxyphenyl)methyl)-3-(*p*-tolyl)imidazo[1,5-*a*]pyridine (5h): Pale yellow solid, yield: 400 mg, 88%; m.p.: 150-152 ºC, ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 8.14 (d, *J* = 8 Hz, 1H), 7.78 (s, 1H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 2.4 Hz, 3H), 7.21 (s, 1H), 6.84 (d, *J* = 1.2 Hz, 4H), 6.47-6.43 (m, 2H), 5.83 (s, 1H), 3.78 (s, 6H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ ppm: 152.89, 133.34, 130.83, 129.73, 127.10, 125.67, 124.95, 124.40, 123.02, 122.96, 122.28, 116.26, 113.59, 112.55, 108.52, 108.35, 107.50, 50.34, 50.12, 43.93, 24.58. HRMS (ESI): *m/z* calcd. for $C_{29}H_{26}N_2O_2$ $[M+H]^+$: 434.1994, found 434.1984.

3-(4-Methoxyphenyl)-1-((3-methoxyphenyl)(4-methxy-phenyl)methyl)imidazo[1,5-*a*]pyridine (5i): Pale yellow solid, yield: (320 mg, 85%); m.p. = 240-242 °C, ^{1}H NMR (400 MHz, $CdCl_3$) δ 8.09 (d, *J* = 8.0 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 4H), 7.01-6.81 (m, 7H), 6.47-6.41 (m, 2H), 5.82 (s, 1H), ^{13}C NMR (101 MHz, $CdCl_3$) δ 159.76, 157.97, 137.05, 135.99, 134.23, 132.12, 130.04, 129.64, 127.89, 127.71, 122.91, 121.09, 118.65, 117.44, 114.27, 113.77, 113.60. 112.47 HRMS (ESI): *m/z* calcd. for $C_{27}H_{22}N_2O$ $[M+H]^+$: 450.1943, found 450.1938.

3-(4-Fluorophenyl)-1-((4-fluorophenyl)(phenyl)methyl)-imidazo[1,5-*a*]pyridine (5j): Brown gummy solid, yield: (375 mg, 92%); m.p. = 175-177 °C, ^{1}H NMR (400 MHz, $CdCl_3$) δ 8.10 (d, *J* = 4.0 Hz, 1H), 7.76-7.72 (m, 2H), 7.33-7.16 (m, *J* = 9.0 Hz), 7.00-6.90 (m, 3H), 6.55-6.48 (m, 2H), 5.89 (s, 1H), ^{13}C NMR (101 MHz, $CdCl_3$) δ 163.96, 162.38, 161.50, 160.27, 148.02, 143.18, 139.03, 136.35, 134.19, 130.65, 130.57, 130.07, 129.05, 128.36, 126.47, 121.10, 118.38, 118.12, 116.09, 115.88, 115.12, 114.91, 112.99 HRMS (ESI): *m/z* calcd. for $C_{26}H_{18}N_2F_2$ $[M+H]^+$: 396.1438, found 396.1427.

3-(4-Chlorophenyl)-1-((4-fluorophenyl)(phenyl)methyl)-imidazo[1,5-*a*]pyridine (5k): Pale yellow solid, yield: (330 mg, 84%); m.p. = 185-187 °C, ^{1}H NMR (400 MHz, $CdCl_3$) δ 8.13 (t, *J* = 4.0 Hz, 1H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 7H), 7.00 (t, *J* = 8.0 Hz, 2H), 6.55-6.50 (m, 2H), 5.88 (s, 1H), ^{13}C NMR (101 MHz, $CdCl_3$) δ 162.70, 160.27, 148.02, 143.10, 130.63, 130.55, 129.33, 129.12, 129.03, 128.75, 128.63, 128.36, 126.48, 122.17, 121.15, 118.43, 118.29, 117.53, 115.12, 114.91, 113.17 HRMS (ESI): *m/z* calcd. for $C_{26}H_{18}N_2ClF$ $[M+H]^+$: 412.1143, found 412.1242.

1-((4-Fluorophenyl)(phenyl)methyl)-3-(4-methoxy-phenyl)imidazo[1,5-*a*]pyridine (5l): Pale yellow solid, yield: (345 mg, 88%); m.p. = 200-202 °C, ^{1}H NMR (400 MHz, $CdCl_3$)

δ 8.10 (d, *J* = 4.0 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.33-7.28 (m, 7H), 7.03-7.00 (m, 4H), 6.98 (d, *J* = 1.2 Hz, 1H), 6.48-6.43 (m, 2H), 5.90 (s, 1H), ^{13}C NMR (101 MHz, $CdCl_3$) δ 162.68, 160.25, 159.86, 143.31, 139.16, 137.29, 133.79, 130.69, 130.61, 129.66, 129.10, 128.32, 128.04, 126.41, 122.75, 121.35, 118.31, 117.81, 114.86, 114.34, 112.59 HRMS (ESI): *m/z* calcd. for $C_{27}H_{22}N_2O$ $[M+H]^+$: 408.1638, found 408.1629.

RESULTS AND DISCUSSION

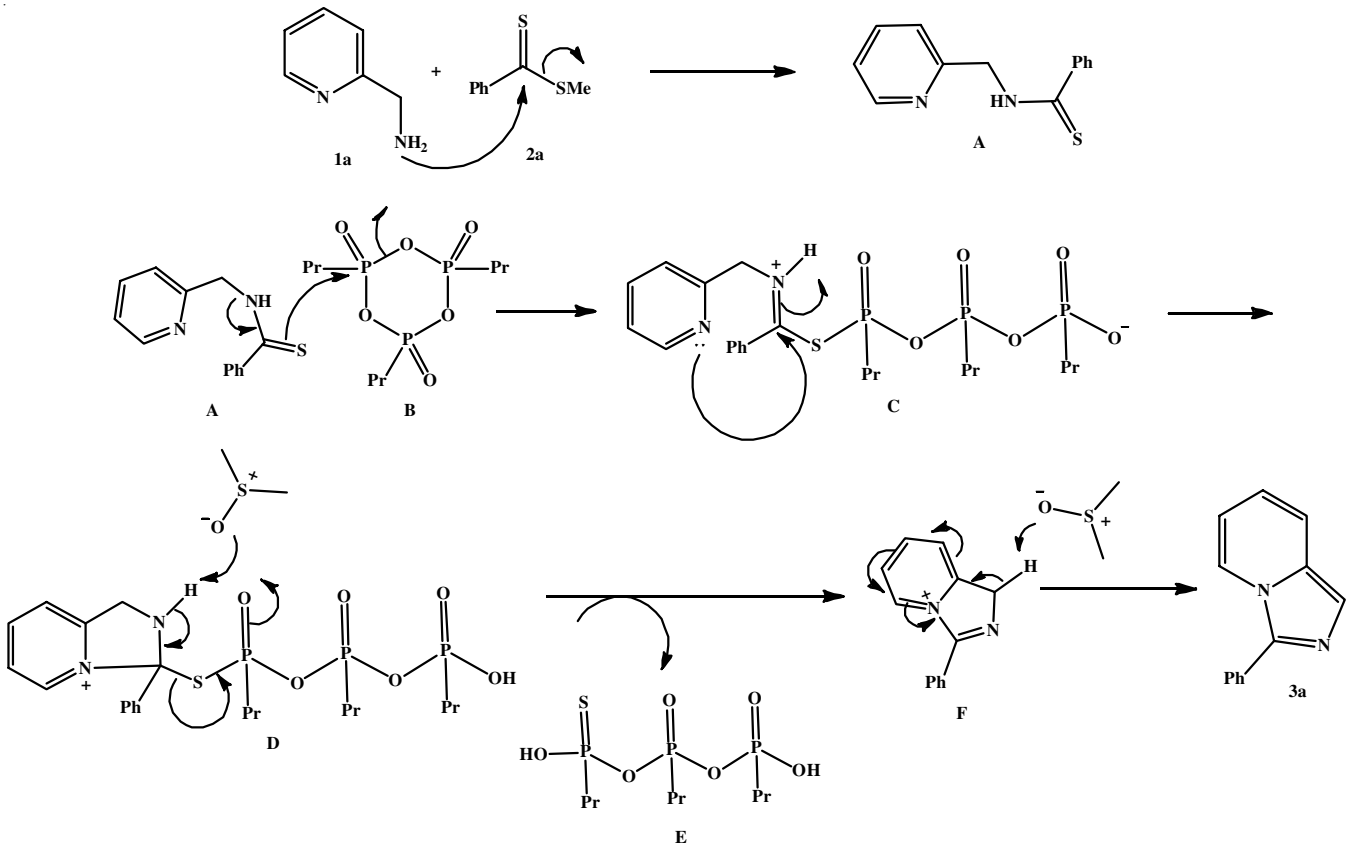
In compound **3**, the first position of imidazo moiety is still vacant, therefore, we intend to introduce a diphenylmethyl (**4**) moiety for stabilization of this reaction. Thus, 2-methylaminopyridine (**1**), dithioester (**2**) and benzhydrols (**4**) as pilot compounds were chosen. Preliminarily, we stabilized mol% of $Fe(OTf)_3$ and MS3Å (molecular sieves 3Å) by varying the mol% in following increments of 25, 50, 75, 100, 200 and 250 in toluene at 90 ºC about 24 h, (Table-1, entry 1-6). As increasing the mol% of $Fe(OTf)_3$ from 25-200 observed significant rise in yield, further increase in mol% causes reduction of yield. Then, we moved on to optimize the reaction temperature by conducting same experiment in different temperature (Table-1, entry 7-9). On increasing temperature the yield of reaction also increased, at lower temperature reduction in yields was observed and at reflux temperature the yield of the reaction is attained maximum. For the stabilization of solvent for this method, we carried out the reaction in different solvents like xylene, 1,4-dioxane, DMF, H_2O, THF and solvent free conditions

TABLE-1
OPTIMIZATION OF THE REACTION CONDITION

Entry	Solvent	$Fe(OTf)_3$, MS3Å (% mol)	Temp. (°C)	Time (h)	Yield (%)
1	Toluene	25	90	24	16
2	Toluene	50	90	24	28
3	Toluene	75	90	24	32
4	Toluene	100	90	24	48
5	Toluene	200	90	24	76
6	Toluene	250	90	24	64
7	Toluene	200	Reflux	12	89
8	Toluene	200	100	24	68
9	Toluene	200	80	24	54
10	Xylene	200	Reflux	12	30
11	1,4-Dioxane	200	Reflux	12	46
12	DMF	200	Reflux	12	Trace
13	H_2O	200	Reflux	12	No result
14	THF	200	Reflux	12	20
15	No solvent	200	Reflux	12	–

(Table-1, entries 10-15). In presence of xylene, 1,4-dioxane, DMF and THF yields were poor compared to toluene. In case of H_2O and solvent free conditions no desired product was obtained. Hence, compound **5a** yield was more in toluene at reflux temperature about 12 h with 200 mol% of $Fe(OTf)_3$ and MS3Å.

With this optimization condition to explore the substrate scope for this synthetic methodology, initially different halo substitution like fluoro and chloro at *para* position of dithioesters yields products **5b** and **5c** with good yields (77-82%),



Scheme-II: Plausible reaction mechanism for the formation of **3a**

while the electron donating groups like methyl and methoxy at *para* position in dithioester yields products **5d** and **5e** with good to excellent yield (78-88%). However, when different substitution introduce on benzhydrols, primarily *para*-methoxy benzhydrol was investigated with different *para* substituted dithioesters which yields compounds **5f-5i** with excellent yields (85-93%). Finally, benzhydrol substituted with fluoro group and reacted with different dithioesters yields compounds **5j-5l** with excellent yields (84-92%).

Plausible mechanism for the formation of 3a: A probable mechanism for the cyclization reaction leads to formation of imidazo[1,5-*a*]pyridine **3a** is shown in **Scheme-II**. In this reaction, 2-methylaminopyridine **1a** reacts with dithioester **2a** to give N-2-pyridylmethyl thioamide **A**. This thioamide **A** reacts with **B** (T3P) to give an intermediate **C**, which undergoes intramolecular cyclization to give the cyclic compound **D** and then it leads to deprotonation of the cyclic product **D** by DMSO to gives **E**, which on further deprotonation to give **3a**.

Plausible mechanism for the formation of 5a: A probable mechanism for the cyclization reaction leads to formation of **5a** is shown in **Scheme-III**. The mechanism takes place when lone pair of electrons on the oxygen atom of **4a** (benzohydrol) attacks on **B** $Fe(OTf)_3$ to form an intermediate **G** and this activated intermediate **G** was attacked by imidazo[1,5-*a*]pyridine **3a** to form intermediate **I** by eliminating the by-product open hydrated $Fe(OTf)_3$ which is **H**. The **H** abstracts hydrogen from intermediate **I** and followed by re-aromatization to give final product **5a** with the expulsion of byproduct **J**.

Conclusion

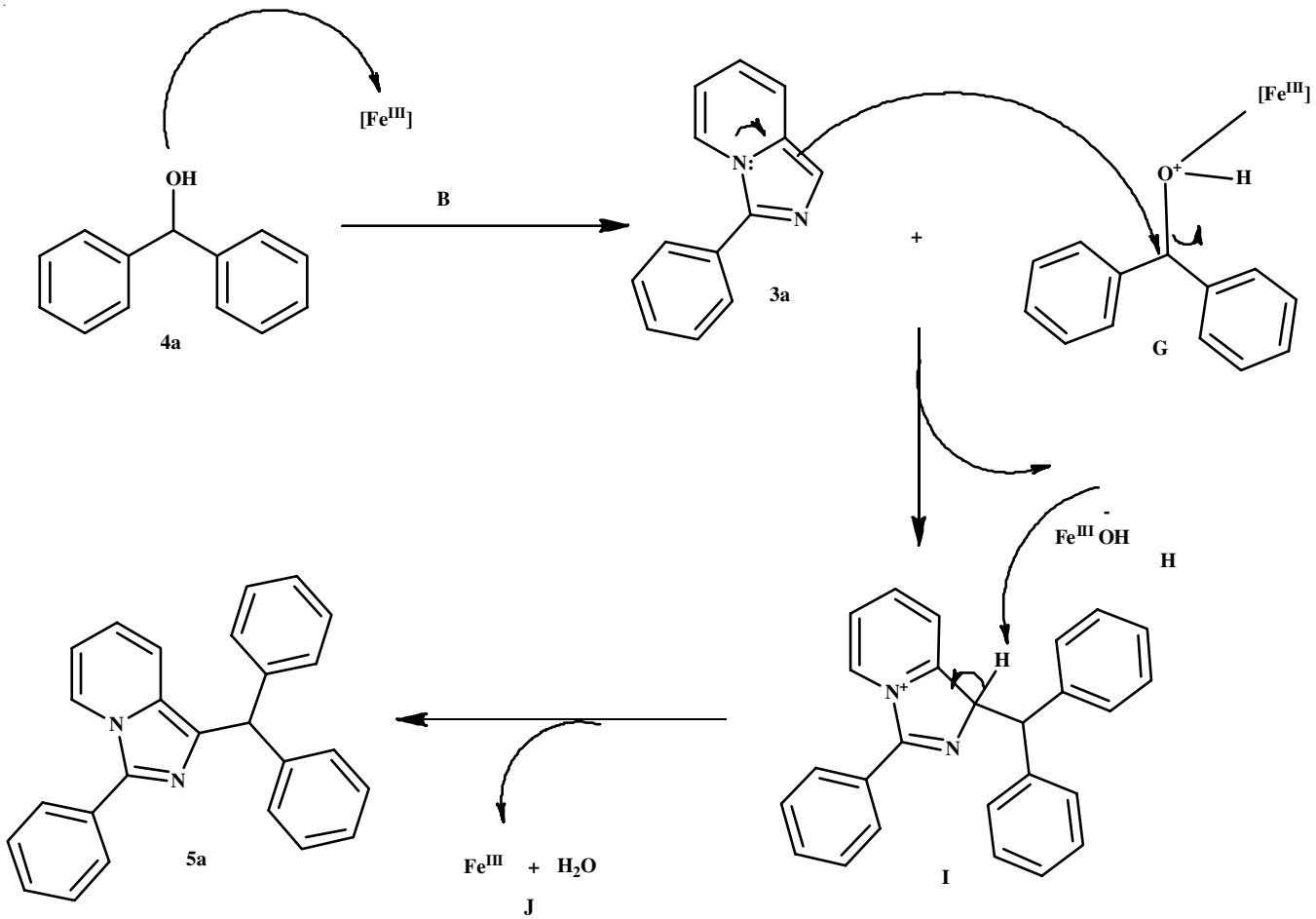
These substituted imidazo[1,5-*a*]pyridines play a crucial role in materials science as well as in the field of pharmaceuticals. The first position of imidazo moiety is still vacant therefore, diphenyl methyl groups are introduced in one pot synthesis. Then, introduce different substitution on benzhydrols, primarily *para*-methoxy benzhydrol was tested with different *para* substituted dithioesters which yields compounds **5f**, **5g**, **5h** and **5i** with excellent yields and all compounds are simple to deal with purify and scale up.

ACKNOWLEDGEMENTS

The authors thank the University of Mysore, Mysuru, India for spectral analysis. One of the authors, Kemparajegowda thank the Karnataka State Open University, Mysuru, India for providing lab facilities.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.



Scheme-III: Plausible reaction mechanism for the formation of **5a**

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