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Boric acid-accelerated, one-pot three-component synthesis of imidazo[1,2-*a*] pyridine derivatives

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Abstract: An efficient, simple, and rapid synthesis of imidazo[1,2-*a*]pyridine derivatives has been described by the three-component reaction of aromatic aldehydes, 2-aminopyridines derivatives and cyclohexylisocyanide using boric acid as a catalyst under solvent-freeconditions. This novel protocol has the advantages of short reaction times, convenient operation, low cost, and environmental benign method.

Keywords: Imidazopyridines, Boric acid, 2-Aminopyridine, Isocyanide, Three-component reactions

Introduction

Heterocyclic compounds always attribute remarkable attention in pharmaceutical industry due to their wide therapeutic values. Nitrogen-containing heterocyclic compounds are widespread in nature, and their applications to pharmaceuticals, agrochemicals, and functional materials are becoming more and more important [1-3].

Derivatives containing the imidazo[1,2-*a*] pyridine ring system have been shown to possess a broad range of useful pharmacological activities, including antibacterial, antifungal, anthelmintic, antiviral, antiprotozoal, anti-inflammatory, anticonvulsant, anxiolytic (Alpidem), hypnotic (Zolpidem), gastrointestinal, antiulcer (Zolmidine), and immunomodulatory (Kifunensine) activities (Fig. 1) [4-8].

Recently, It has been reported that some of the imidazo[1,2-*a*]pyridine derivatives are potential candidate for fluorescent probes, which can be used for fluorescence imaging in clinical diagnostics and biomedical research [9-10].

Due to their applications in a varietyof fields, several synthetic routes have been developed for the synthesis of imidazo[1,2-*a*]pyridines [11-12]. Particularly, the last decadehas witnessed a remarkable advancement in the synthesisof

imidazo[1,2-*a*]pyridines by employing various interestingapproaches [13-14].Basically, the common method for the synthesis of imidazo[1,2-*a*]pyridinesis based on a multi-component reaction (MCRs) of an aldehyde, an isocyanide and a 2-aminoazine using different catalysts such as $HClO_4$[15],HOAc [16],scandium triflate ($Sc(OTf)_3$) [17-18],pTSA [19], $ZnBr_2$ [20],glyoxalic acid [21], ionic liquid [22],cellulose sulfuric acid [23],$ZnCl_2$ [24], NH_4Cl [25],N-fluoropyridinium salts [26],K_2CO_3 [27],bromodimethylsulfonium bromide (BDMS) [9],nano-sized $LaMnO_3$ [28] andFe_3O_4-SiO_2nanoparticles [29].However, some of the reported methods require prolonged reaction time, use of expensive catalyst, low yields of products, and toxic solvents.Thus developing versatile approaches towards the synthesis ofimidazo[1,2-*a*]pyridine derivatives is of importance.

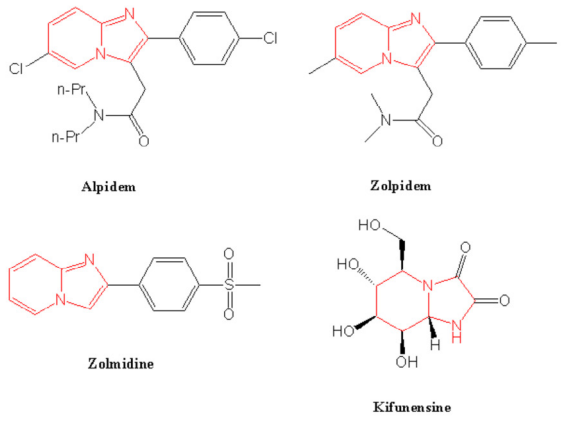


Figure 1. Some of the important imidazo[1,2-*a*] pyridines

Herein, we like to report an efficient method to synthesize imidazo[1,2-*a*]pyridine derivatives by one-step cyclocondensation of aromatic aldehyde, 2-aminopyridine derivatives and cyclohexylisocyanidein the presence of boric acid under solvent-free conditions (Scheme 1).

Experimental

All reagents were commercially available and used without further purification. Melting points were measured by an electrothermal type 9100 melting point apparatus. The infrared (IR) spectra were recorded on a Bruker Tensor 27. NMR spectra were determined on a Bruker AC 250 MHz spectrometer as $CDCl_3$ solutions.

General procedure for the synthesis of imidazo[1,2-*a*]pyridines

A mixture of aromatic aldehydes **1** (2 mmol), 2-aminopyridine derivatives **2** (2 mmol), cyclohexylisocyanide**3** (2 mmol) and boric acid (10 mol%) was stirred at 80 ˚C for 5 min. Upon completion, monitored by TLC (n-hexane/ ethyl acetate: 2/1), the reaction mixture was allowed to cool to room temperature. The crude products were purified by recrystallization from chloroform/petroleum ether.

Characterization data of some representative compounds

*2-(4-bromophenyl)-N-cyclohexyl-H-imidazo[1,2-a]pyridin-3-amine (**4d**)*

IR (KBr) (ν_{max} /cm^{-1}): 3230, 2945, 2855, 1640; ^{1}HNMR (250MHz, $CDCl_3$): 1.61-1.90 (m, $5CH_2$ and CH, of cyclohexyl), 2.93 (bs, 1H, NH), 6.87 (t, 1H, *J*=6.75 Hz, ArH), 7.19-7.23 (m, 1H, ArH), 7.53-7.64 (m, 3H, ArH), 7.97 (d, 2H, *J*=8.25 Hz, ArH), 8.14 (d, 1H, *J*=6.50 Hz, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 23.4, 24.1, 33.6, 51.4, 115.1, 122.2, 126.5, 126.8, 127.6, 129.5, 130.0, 130.4, 135.4,138.7, 144.6 ppm. Anal.CalcdFor $C_{19}H_{20}BrN_3$: C, 61.63; H, 5.44; N, 11.35; Found: C, 61.55; H, 5.47; N, 11.29.

*N-cyclohexyl-2-(4-nitrophenyl) H-imidazo[1,2-a]pyridin-3-amine (**4g**)*

IR (KBr) (ν_{max} /cm^{-1}): 3225, 2923, 2850, 1631; ^{1}HNMR (250MHz, $CDCl_3$): 1.17-1.86 (m, $5CH_2$ and CH, of cyclohexyl), 2.98 (bs, 1H, NH), 6.84

(t, 1H, *J*=6.75 Hz, ArH), 7.19 (t, 1H, *J*=7.5 Hz, ArH), 7.56 (d, 1H, *J*=9 Hz, ArH), 8.07 (d, 1H, *J*=6.75 Hz, ArH), 8.27-8.32 (m, 4H, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 23.5, 28.2, 33.8, 51.3, 115.1, 120.6, 121.9, 122.2, 126.6, 128.1, 130.5, 135.5, 139.0, 144.7, 148.1 ppm.Anal. CalcdFor $C_{19}H_{20}N_4O_2$: C, 67.84; H, 5.99; N, 16.66; Found: C, 67.80; H, 5.97; N, 16.59.

*N-cyclohexyl-2-(2-methoxyphenyl) H-imidazo[1,2-a]pyridin-3-amine (**4l**)*

IR (KBr) (ν_{max} /cm^{-1}): 3348, 2927, 2853, 1735; ^{1}HNMR (250MHz, $CDCl_3$): 1.05-1.71 (m, 5CH_2 and CH, of cyclohexyl), 2.64 (bs, 1H, NH), 3.87 (s, 3H, OCH_3), 6.75 (t, 1H, *J*=6.75 Hz, ArH), 6.98-7.13 (m, 3H, ArH), 7.30-7.34 (m, 1H, ArH), 7.53 (d, 1H, *J*=9 Hz, ArH), 7.84 (d, 1H, *J*=7 Hz, ArH) 8.08 (d,1H, *J*=6.75 Hz, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 23.0, 28.3, 33.7, 51.3, 56.0, 114.5, 115.2, 119.4, 121.4, 122.1, 126.5, 128.1, 128.3, 129.6, 130.0, 135.4, 144.3, 157.7 ppm.Anal.CalcdFor $C_{20}H_{23}N_3O$: C, 74.74; H, 7.21; N, 13.07; Found: C, 74.65; H, .7.17; N, 13.15.

*N-cyclohexyl-7-methyl-2-phenyl-H-imidazo[1,2-a]pyridin-3-amine (**4q**)*

IR (KBr) (ν_{max} /cm^{-1}): 3320, 2918, 2845, 1720; ^{1}HNMR (250MHz, $CDCl_3$): 1.15-2.26 (m, 5CH_2 and CH, of cyclohexyl), 2.39 (s, 3H, CH_3), 2.94 (bs, 1H, NH), 6.64 (d, 1H, *J*=7 Hz, ArH), 7.26-7.61 (m, 5H, ArH), 8.02 (d, 2H, *J*=7.25 Hz, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 23.4, 24.6, 28.3, 33.8, 51.7, 117.4, 122.1, 124.1, 124.4, 127.7, 128.6, 129.5, 133.3, 135.4, 144.2, 151.5 ppm.Anal.CalcdFor $C_{20}H_{23}N_3$: C, 78.65; H, 7.59; N, 13.76; Found: C, 78.60; H, 7.49; N, 13.83.

*N-cyclohexyl-2-(4-methoxyphenyl)-7-methyl-H-imidazo[1,2-a]pyridin-3-amine (**4z**)*

IR (KBr) (ν_{max} /cm^{-1}): 3260, 2927, 2853, 1642; ^{1}HNMR (250MHz, $CDCl_3$): 1.18-1.81 (m, 5CH_2 and CH, of cyclohexyl), 2.38 (s, 3H, CH_3), 2.96 (bs, 1H, NH), 3.85 (s, 3H, CH_3), 6.60 (d, 1H, *J*=7.25 Hz, ArH), 6.97 (d, 2H, *J*=8.75 Hz, ArH), 7.25-7.27 (m, 1H, ArH), 7.94-7.98 (m, 3H, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 23.5, 24.5, 28.2, 33.8, 51.3, 55.7, 114.1, 114.9, 117.4, 122.2, 124.1, 124.4, 125.7, 128.1, 135.5, 151.3, 160.5 ppm.Anal.CalcdFor $C_{21}H_{25}N_3O$: C, 75.19; H, 7.51; N, 12.53; Found: C, 75.10; H, 7.43; N, 12.62.

*N-cyclohexyl-7-methyl-2-(pyridin-2-yl) H-imidazo[1,2-a]pyridin-3-amine (**4ac**)*

IR (KBr) (ν_{max} /cm^{-1}): 3286, 3055, 2927, 2852, 1644; ^{1}HNMR (250MHz, $CDCl_3$): 1.18-1.89 (m, 5CH_2 and CH, of cyclohexyl), 2.35 (s, 3H, CH_3), 3.03 (bs, 1H, NH), 6.15 (s, 1H, ArH), 6.56 (d, 1H, *J*=7 Hz, ArH), 7.05-7.10 (m, 1H, ArH), 7.70 (t, 1H, *J*=7.75 Hz, ArH), 7.85 (d,1H, *J*=7 Hz, ArH), 8.12 (d, 1H, *J*=8 Hz, ArH), 8.50-8.52 (m, 1H, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 24.5, 28.1, 33.6, 49.1, 51.7, 109.2, 116.2, 117.2, 123.2, 124.1, 124.4, 135.2, 136.3, 139.8, 144.4, 155.3 ppm.Anal.CalcdFor $C_{19}H_{22}N_4$: C, 74.48; H, 7.24; N, 18.29; Found: C, 74.35; H, 7.30; N, 18.20.

*N-cyclohexyl-2-(furan-2-yl)-7-methyl-H-imidazo[1,2-a]pyridin-3-amine (**4ad**)*

IR (KBr) (ν_{max} /cm^{-1}): 3317, 2927, 2854,1645; ^{1}HNMR (250MHz, $CDCl_3$): 1.10-2.90 (m, 5CH_2 and CH, of cyclohexyl), 2.36 (s, 3H, CH_3), 2.94 (bs, 1H, NH), 6.32-7.93 (m, 6H, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 23.5, 24.5, 28.2, 33.4, 50.6, 105.2, 107.5, 117.2, 122.2, 124.1, 124.4, 142.7, 144.1, 144.9, 151.5, 157.3 ppm. Anal.CalcdFor $C_{18}H_{21}N_3O$: C, 73.19; H, 7.17; N, 14.23; Found: C, 73.10; H, 7.25; N, 14.17.

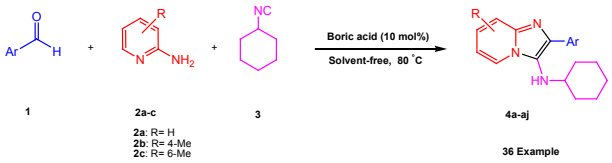
*N-cyclohexyl-5-methyl-2-(4-nitrophenyl) H-imidazo[1,2-a]pyridin-3-amine (**4ag**)*

IR (KBr) (ν_{max} /cm^{-1}): 3386, 3055, 2926, 2852, 1652; ^{1}HNMR (250MHz, $CDCl_3$): 1.14-1.64 (m, 5CH_2 and CH, of cyclohexyl), 2.71 (s, 3H, CH_3), 3.03 (bs, 1H, NH), 6.77 (d, 1H, *J*=6.8 Hz, ArH), 7.40 (t, 1H, *J*=7.2 Hz, ArH), 7.70 (d, 1H, *J*=8.8 Hz, ArH), 8.02 (d, 2H, *J*=8.8 Hz, ArH), 8.42 (d,

2H, *J*=8.8 Hz, ArH) ppm; ^{13}CNMR (62.5MHz, $CDCl_3$): 15.2, 17.4, 22.1, 22.8, 35.3, 42.3, 48.4, 120.1, 121.8, 122.1, 128.6, 134.9, 136.4, 139.5, 144.1, 145.1 ppm.Anal.CalcdFor $C_{20}H_{22}N_4O_2$: C, 68.55; H, 6.33; N, 15.99; Found: C, 68.66; H, 6.41; N, 16.07.

Results and Discussion

Continuing our ongoing studies on the application of boric acid as a catalyst in the organic synthesis [30-34], herein, imidazo[1,2-*a*]pyridine derivatives **4a–aj** were prepared from the three-component reaction of aromatic aldehydes **1**, 2-aminopyridine derivatives **2a-c** and cyclohexylisocyanide**3**in the presence of boric acid at 80°C undersolvent-freeconditions.In order to standardize the reaction, 4-chlorobenzaldehyde (2 mmol), 2-aminopyridine (2 mmol), and cyclohexylisocyanide (2 mmol) were explored first to give 2-(4-chlorophenyl)-N-cyclohexylH-imidazo[1,2-*a*]pyridin-3-amine **4b**. The mixture was heated in an oil-bath at 80 °C. Interestingly compound **4b** has been obtained after 5 min using 10 mol% of boric acid (Table 1, Entry 2).



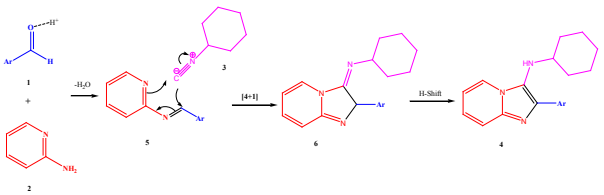
Scheme 1. Synthesis of imidazo[1,2-a]pyridine derivatives

In order to confirm the generality of the reaction, we applied this strategy to various structurally diverse aldehydes. As shown in Table 1, these reactions occur fast, within a few minutes (1-5 min), and give excellent yields (85–95%) of products **4a–aj** with high purity. The results are summarized in Table 1. However to further explore the potential of this protocol; we investigated one-pot reactions involving substituted 2-aminopyridine (**2b**, **2c**). The reactions proceeded smoothly and desired products **4q-4aj**were obtained in 5 min with good yields.

All aromatic aldehydes containing electron-withdrawing groups (such as nitro group, halide) or electron-donating groups (such as hydroxyl group, alkoxy group) were employed and reacted well to give the corresponding product **4** in good to excellent yields under these reaction conditions, so we conclude that no obvious effect of electron and nature of substituents on the aromatic ring were observed.

It is to be noted that heteroaromatic aldehyde such as 2-pyridine carbaldehyde and furfural (Table 1, entries **4m**, **4p**, **4ac** and **4ad**) were converted into the corresponding products with excellent yields. The structures of **4a–aj** were determined on the basis of their elemental analyses, ^{1}H and ^{13}C NMR data, and IR spectral data.

A mechanism for the reaction is outlined in Scheme 2.Initially aromatic aldehyde (activated by boric acid) reacts with amidine to form imine **5**by loss of H_2O, which reacts instantly with isocyanide in a [4+1] cycloaddition manner to give intermediate **6**. The final product imidazo[1,2-*a*]pyridines **4** was obtained from the intermediate **6** by 1,3-hydrogen shift, as suggested earlier [9].



Scheme 2. Proposed mechanism

Conclusions

In conclusion, we have developed a novel and one-pot reaction of aldehydes, cyclohexylisocyanide, and 2-aminopyridinederivatives in the

Table 1. Synthesis of imidazo[1,2-*a*]pyridine **4a–aj**using 10mol% of boric acid

Entry	R	Ar	Product	Yield (%)[b]	m.p (˚C)[15-29]
1	H	C_6H_5	**4a**	85	178-179
2	H	4-ClC_6H_4	**4b**	95	187-190
3	H	2-ClC_6H_4	**4c**	85	Gummy
4	H	4-BrC_6H_4	**4d**	90	144-145
5	H	4-FC_6H_4	**4e**	90	203-205
6	H	4-CNC_6H_4	**4f**	95	179-181
7	H	4-$NO_2C_6H_4$	**4g**	85	214-216
8	H	3-$NO_2C_6H_4$	**4h**	90	202-204
9	H	2,4-$Cl_2C_6H_3$	**4i**	85	Gummy
10	H	4-$CH_3C_6H_4$	**4j**	95	154-156
11	H	4-$OCH_3C_6H_4$	**4k**	95	144-145
12	H	2-$OCH_3C_6H_4$	**4l**	95	Gummy
13	H	2-Pyridyl-	**4m**	85	Gummy
14	H	1-naphthyl	**4n**	80	Gummy
15	H	2-naphthyl-	**4o**	95	148-150
16	H	2-furyl-	**4p**	80	Gummy
17	4-Me	C_6H_5	**4q**	95	157-159
18	4-Me	4-ClC_6H_4	**4r**	85	212-214
19	4-Me	4-BrC_6H_4	**4s**	80	200-202
20	4-Me	4-FC_6H_4	**4t**	87	143-145
21	4-Me	4-CNC_6H_4	**4u**	85	219-220
22	4-Me	4-$NO_2C_6H_4$	**4v**	87	234-235
23	4-Me	3-$NO_2C_6H_4$	**4w**	80	190-191
24	4-Me	4-$CH_3C_6H_4$	**4x**	85	168-170
25	4-Me	3-$CH_3C_6H_4$	**4y**	98	Gummy
26	4-Me	4-$OCH_3C_6H_4$	**4z**	87	148-149
27	4-Me	$(CH_3)_2NC_6H_4$	**4aa**	80	Gummy
28	4-Me	2-naphthyl-	**4ab**	80	181-183
29	4-Me	2-Pyridyl-	**4ac**	90	Gummy
30	4-Me	2-furyl-	**4ad**	90	Gummy
31	6-Me	C_6H_5	**4ae**	80	88-90
32	6-Me	4-ClC_6H_4	**4af**	85	Gummy
33	6-Me	4-$NO_2C_6H_4$	**4ag**	85	243-245
34	6-Me	3-$NO_2C_6H_4$	**4ah**	85	220-222
35	6-Me	4-$OCH_3C_6H_4$	**4ai**	90	95-96
36	6-Me	2-furyl-	**4aj**	87	113-115

Notes: [a]Reaction conditions: aromatic aldehyde (2 mmol), 2-aminopyridinederivatives (2 mmol),cyclohexylisocyanide (2 mmol), boric acid (10 mol%), 5 min at 80 ˚C
[b]Isolated yield

presence of boric acid for the preparation of imidazo[1,2-*a*]pyridines of potential synthetic and pharmacological interest. Readily available and inexpensive catalyst, excellent yields of the products, fast reaction times, mild reaction conditions as well as solvent-free conditions are the advantages of this method. Furthermore, product isolation does not require purification by column chromatography. In addition, the products also exhibit interesting fluorescence properties, which may be useful for fluorescent probe. Their fluorescence studies are in progress.

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