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Ionic liquid promoted one-pot synthesis of 3-aminoimidazo[1,2-*a*]pyridines

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Abstract—3-Aminoimidazo[1,2-*a*]pyridines have been synthesized in good to excellent yields in the presence of the ionic liquid 1-butyl-3-methylimidazolium bromide [bmim]Br, the reaction workup is simple and the ionic liquid can be easily separated from the product and reused.

Imidazo[1,2-*a*]pyridines, an important class of pharmaceutical compounds exhibit a wide spectrum of biological activities.[1] As a result, numerous two- and three-component approaches for their synthesis have been reported.

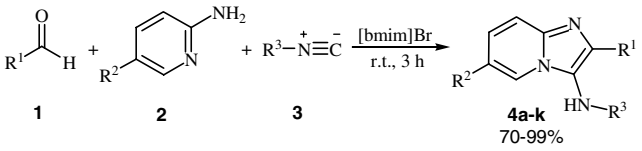
The classical synthesis of imidazo[1,2-*a*]pyridines involves the condensation of α-haloketones with 2-aminopyridines.[2] Being only a two-component condensation, this reaction is less suitable for the generation of a large compound library.

Recently, several isocyanide-based multi-component reactions (MCRs) have been reported for the synthesis of these compounds by the condensation of an aldehyde, an isocyanide and a 2-aminoazine in the presence of a strong protic acid[3–5] (AcOH, $HClO_4$) or Lewis acid[5] ($Sc(OTf)_3$). However, these reactions require long times for completion and isolation and recovery procedures are complicated. For example, in the case of $Sc(OTf)_3$, the reaction mixture was agitated for 72 h at ambient temperature, then it was allowed slowly to adsorb onto Dowex 50WX 2–200 strongly acidic cation exchange resin. The resin was washed with MeOH, CH_2Cl_2 and MeOH and finally the product was eluted using 2 M NH_3 in MeOH and the solvent evaporated.

The synthesis of imidazo[1,2-*a*]pyridines has also been reported under microwave irradiation in the presence of a solid acid, montmorillonite K_{10},[6] and $Sc(OTf)_3$,[7] however, special instrumentation is required.

During recent years, ionic liquids have attracted interest as environmentally benign reagents due to their favorable properties and a variety of catalytic reactions have been successful using ionic liquids.[8] The solvophobic properties of ionic liquids are able to generate an internal pressure and promote the association of the reactants in a solvent cavity during the activation process and accelerate a reaction. This property of ionic liquids is very efficient for MCRs in which the entropy of reaction is decreased in the transition state.

During the course of our studies on the development of new routes for the synthesis of organic compounds using ionic liquids[9] and our interest in isocyanide-based MCRs,[10] we developed the synthesis of 3-aminoimidazo[1,2-*a*]pyridines via the three-component condensation of an aldehyde **1**, 2-amino-5-methylpyridine or 2-amino-5-bromopyridine **2** and isocyanide **3** in 1-butyl-3-methylimidazolium bromide ([bmim]Br) at room temperature (Scheme 1).



Scheme 1.

Keywords: Imidazo[1,2-*a*]pyridine; Ionic liquid; Isocyanide; Multi-component reactions; [bmim]Br.
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Table 1. Synthesis of 3-aminoimidazo[1,2-*a*]pyridines in [bmim]Br

Entry	R^1	R^2	R^3	Product	Yield (%)
1	Ph	Br	Cyclohexyl	**4a**	98 (95, 92, 90, 85)[a]
2	Ph	Me	Cyclohexyl	**4b**	98
3	4-$CH_3C_6H_4$	Me	Cyclohexyl	**4c**	99
4	4-ClC_6H_4	Me	Cyclohexyl	**4d**	92
5	3-$O_2NC_6H_4$	Me	Cyclohexyl	**4e**	99
6	4-Pyridyl	Me	Cyclohexyl	**4f**	97
7	Ph	Br	*tert*-Butyl	**4g**	99
8	Ph	Me	*tert*-Butyl	**4h**	90
9	4-$CH_3C_6H_4$	Me	*tert*-Butyl	**4i**	86
10	Ph	Me	2,6-$(Me)_2C_6H_3$	**4j**	72
11	4-$CH_3C_6H_4$	Me	2,6-$(Me)_2C_6H_3$	**4k**	70

[a] The same ionic liquid was used for each of the five runs.

As indicated in Table 1, the reaction of aldehydes with 2-amino-5-methylpyridine or 2-amino-5-bromopyridine and isocyanides afforded 3-aminoimidazo[1,2-*a*]pyridines in [bmim]Br as a promoter in very high yields. The structures of the products **4a**–**k** were deduced from their IR, mass, ^{1}H NMR and ^{13}C NMR spectra.

To illustrate the need for [bmim]Br, the reaction of *p*-methylbenzaldehyde, 2-amino-5-methylpyridine and cyclohexyl isocyanide was studied in the absence of [bmim]Br. The yield of product was only 25% at room temperature after 12 h. Obviously, [bmim]Br is an important component of the reaction.

One of the advantages of ionic liquids is their ability to function as a recyclable reaction medium. We were able to separate [bmim]Br from the reaction medium easily by washing with water and evaporating the solvent under vacuum, and reuse it for subsequent reactions.

In conclusion, we have introduced an efficient and environmentally friendly approach for the synthesis of 3-aminoimidazo[1,2-*a*]pyridines via condensation of an aldehyde, 2-amino-5-methylpyridine or 2-amino-5-bromopyridine and an isocyanide using [bmim]Br in good to excellent yields at room temperature. To the best our knowledge, this is the first report on the synthesis of 3-aminoimidazo[1,2-*a*]pyridines in an ionic liquid and these new reaction conditions open an important alternative to the use of volatile organic solvents.

All the products (except **4h**) are new compounds, which were identified by IR, ^{1}H NMR and ^{13}C NMR spectral data and mass spectroscopy.

Typical procedure: preparation of 6-bromo-*N*-cyclohexyl-2-phenylimidazo[1,2-*a*]pyridin-3-amine (**4a**)—To a solution of 2-amino-5-bromopyridine (0.17 g, 1 mmol), benzaldehyde (0.13 g, 1.2 mmol) and cyclohexyl isocyanide (0.12 g, 1.1 mmol) were added [bmim]Br[11] (0.3 g, 1.4 mmol). The resulting mixture was stirred for 3 h at room temperature. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 2:1), the reaction mixture was washed with water (2 × 10 ml) and the solid residue was crystallized from ethyl acetate to give **4a** as colorless crystals (0.36 g, 98%): mp 207–208 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3250 (NH), 2920, 1602. MS, *m*/*z* (%): 372 (MH^+, ^{81}Br, 28), 370 (MH^+, ^{79}Br, 30), 288 (68), 286 (65), 261 (100), 259 (95), 158 (70), 156 (78), 76 (30). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H (ppm) 1.17–1.98 (10H, m, $5CH_2$ of cyclohexyl), 2.95 (1H, m, CH–N of cyclohexyl), 3.72 (1H, bs, NH), 7.28–8.32 (8H, m, H-Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C (ppm) 24.76, 25.63, 34.08 (carbons of cyclohexyl), 56.84 (CH–N of cyclohexyl), 106.87, 117.71, 123.02, 125.23, 127.03, 127.77, 128.62, 128.64, 133.36, 136.89, 139.53 (C-Ar).

N-Cyclohexyl-6-methyl-2-phenylimidazo[1,2-*a*]pyridin-3-amine (**4b**)—Colorless crystal (0.30 g, 98%): mp 203–206 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3195 (NH), 2925, 1613. MS, *m*/*z* (%): 306 (M^++1, 71), 305 (M^+, 73), 222 (87), 195 (100), 92 (87), 65 (62), 55 (45), 41 (31). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 1.06–1.74 (10H, m, $5CH_2$ of cyclohexyl), 2.40 (3H, s, CH_3), 2.84 (1H, m, CH–N of cyclohexyl), 5.16 (1H, bs, NH), 7.36–7.64 (5H, m, H-Ar), 8.10 (2H, d, $^3J_{HH} = 7.6$ Hz, H-Ar), 8.37 (1H, s, H-Ar). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 17.71 (CH_3), 24.40, 25.21, 33.18 (carbons of cyclohexyl), 56.11 (CH–N of cyclohexyl), 112.95, 115.64, 122.11, 122.01, 126.13, 126.74, 128.37, 128.74, 132.35, 136.82, 145.88 (C-Ar).

N-Cyclohexyl-6-methyl-2-phenylimidazo[1,2-*a*]pyridin-3-amine (**4c**)—Colorless crystal (0.31 g, 99%): mp 216–218 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3225 (NH), 2920, 1661, 1613. MS, *m*/*z* (%): 320 (M^++1, 62), 319 (M^+, 71), 236 (87), 209 (100), 92 (83), 65 (54), 55 (42), 41 (29). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 1.05–1.69 (10H, m, $5CH_2$ of cyclohexyl), 2.35 (3H, s, CH_3), 2.42 (3H, s, CH_3), 2.83 (1H, m, CH–N of cyclohexyl), 5.20 (1H, bs, NH), 7.34–8.49 (7H, m, H-Ar). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 16.78 (CH_3), 18.17 (CH_3), 24.87, 25.69, 33.61 (carbons of cyclohexyl), 56.51 (CH–N of cyclohexyl), 112.55, 122.82, 126.06, 126.34, 127.20, 129.96, 134.17, 134.49, 136.39, 146.29, 153.06 (C-Ar).

2-(4-Chlorophenyl)-*N*-cyclohexyl-6-methylimidazo[1,2-*a*]-pyridin-3-amine (**4d**)—Colorless crystal (0.31 g, 92%): mp 210–212 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3250 (NH), 2920, 1651, 1604. MS, *m*/*z* (%): 340 (M^++1, 21), 339 (M^+, 50), 256 (87), 209 (67), 92 (100), 65 (48), 55 (27), 41 (19). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H (ppm)

1.16–1.81 (10H, m, $5CH_2$ of cyclohexyl), 2.36 (3H, s, CH_3), 2.92 (1H, m, CH–N of cyclohexyl), 3.27 (1H, bs, NH), 7.02 (1H, d, $^3J_{HH} = 9.1$ Hz, H-Ar), 7.37 (2H, d, $^3J_{HH} = 8.6$ Hz, H-Ar), 7.46 (1H, d, $^3J_{HH} = 9.2$ Hz, H-Ar), 7.85 (1H, s, H-Ar), 8.02 (2H, d, $^3J_{HH} = 8.6$ Hz, H-Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C (ppm) 18.49 (CH_3), 24.80, 25.68, 34.13 (carbons of cyclohexyl), 56.72 (CH–N of cyclohexyl), 116.20, 120.44, 121.97, 124.72, 128.13, 128.60, 132.17, 133.05, 134.43, 140.12, 147.28 (C-Ar).

N-Cyclohexyl-6-methyl-2-3-nitrophenylimidazo[1,2-*a*]-pyridin-3-amine (**4e**)—Yellow crystal (0.34 g, 97%): mp 208–210 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3240 (NH), 2920, 1609, 1560. MS, *m*/*z* (%): 351 (M^++1, 13), 350 (M^+, 36), 267 (100), 240 (73), 220 (12), 194 (10), 92 (73), 65 (25), 55 (31), 41 (12). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H (ppm) 1.18–1.89 (10H, m, $5CH_2$ of cyclohexyl), 2.39 (3H, s, CH_3), 2.97 (1H, m, CH–N of cyclohexyl), 3.47 (1H, bs, NH), 7.11 (1H, d, $^3J_{HH} = 9.2$ Hz, H-Ar), 7.57 (1H, d, $^3J_{HH} = 9.2$ Hz, H-Ar), 7.61 (1H, dd, $^3J_{HH} = 8.0$ Hz, $^3J_{HH} = 8.0$ Hz, H-Ar), 7.95 (1H, s, H-Ar), 8.13 (1H, d, $^3J_{HH} = 8.0$ Hz, H-Ar), 8.56 (1H, d, $^3J_{HH} = 8.0$ Hz, H-Ar), 9.05 (1H, s, H-Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C (ppm) 18.45 (CH_3), 24.83, 25.60, 34.20 (carbons of cyclohexyl), 57.02 (CH–N of cyclohexyl), 116.59, 120.33, 121.28, 121.60, 122.15, 125.47, 128.40, 129.27, 132.54, 133.49, 135.80, 140.58, 148.35 (C-Ar).

N-Cyclohexyl-6-methyl-2-(pyridin-4-yl)imidazo[1,2-*a*]-pyridin-3-amine (**4f**)—Yellow crystal (0.30 g, 99%): mp 235–237 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3245 (NH), 2920, 1598. MS, *m*/*z* (%): 307 (M^++1, 27), 306 (M^+, 52), 223 (78), 196 (54), 92 (100), 65 (60), 55 (35), 41 (31). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H (ppm) 1.13–1.83 (10H, m, $5CH_2$ of cyclohexyl), 2.34 (3H, s, CH_3), 2.95 (1H, m, CH–N of cyclohexyl), 3.20 (1H, bs, NH), 7.01 (1H, d, $^3J_{HH} = 9.2$ Hz, H-Ar), 7.45 (1H, d, $^3J_{HH} = 9.2$ Hz, H-Ar), 7.82 (1H, s, H-Ar), 8.00 (2H, d, $^3J_{HH} = 5.8$ Hz, H-Ar), 8.62 (2H, d, $^3J_{HH} = 5.8$ Hz, H-Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C (ppm) 18.46 (CH_3), 25.83, 25.61, 34.22 (carbons of cyclohexyl), 57.02 (CH–N of cyclohexyl), 116.95, 120.29, 120.99, 122.09, 126.69, 128.36, 133.02, 140.91, 142.10, 149.60 (C-Ar).

N-*tert*-Butyl-6-bromo-2-phenylimidazo[1,2-*a*]pyridin-3-amine (**4g**)—Colorless crystal (0.34 g, 99%): mp 206–208 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3285 (NH), 2955, 1619. MS, *m*/*z* (%): 346 (MH^+, ^{81}Br, 40), 344 (MH^+, ^{79}Br, 42), 289 (85), 287 (100), 261 (90), 259 (90), 158 (75), 156 (73), 76 (59), 57 (25). ^{1}H NMR (300 MHz, $CDCl_3$): δ_H (ppm) 1.04 (9H, s, $C(CH_3)_3$), 3.37 (1H, bs, NH), 7.28–8.41 (8H, H-Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ_C (ppm) 30.28 ($C(CH_3)_3$), 55.59 ($C(CH_3)_3$), 106.67, 117.69, 123.81, 123.92, 127.87, 127.94, 128.13, 128.41, 134.10, 139.79, 139.98 (C-Ar).

N-*tert*-Butyl-6-methyl-2-phenylimidazo[1,2-*a*]pyridin-3-amine (**4h**)—Colorless crystal (0.25 g, 90%): mp 217–220 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3285 (NH), 2955, 1666, 1599. MS, *m*/*z* (%): 280 (M^++1, 85), 279 (M^+, 44), 222 (85), 195 (100), 108 (23), 92 (77), 65 (46), 57 (28), 41 (27). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 0.98 (9H, s, $C(CH_3)_3$), 2.33 (3H, s, CH_3), 4.69 (1H, bs, NH), 7.13 (2H, d, $^3J_{HH} = 9.2$ Hz, H-Ar), 7.25–7.53(4H, m, H-Ar), 7.72 (1H, s, H-Ar), 8.12 (1H, d, $^3J_{HH} = 7.5$ Hz, H-Ar). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 18.30 (CH_3), 30.44 ($C(CH_3)_3$), 55.26 ($C(CH_3)_3$), 111.17, 115.93, 121.15, 122.20, 128.16, 128.52, 135.00, 137.14, 139.96, 142.79, 155.33 (C-Ar).

N-*tert*-Butyl-6-methyl-2-*p*-tolylimidazo[1,2-*a*]pyridin-3-amine (**4i**)—Colorless crystal (0.25 g, 86%): mp 210–212 °C. IR (KBr) (ν_{max}/cm^{-1}): 3230 (NH), 2960, 1663, 1613. MS, *m*/*z* (%): 294 (M^++1, 23), 293 (M^+, 19), 236 (87), 209 (100), 92 (65), 65 (44), 57 (23), 41 (17). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 0.98 (9H, s, $C(CH_3)_3$), 2.33 (3H, s, CH_3), 2.37 (3H, s, CH_3), 4.70 (1H, bs, NH), 7.25 (2H, d, $^3J_{HH} = 7.5$ Hz, H-Ar), 7.36 (1H, d, $^3J_{HH} = 8.3$ Hz, H-Ar), 7.59 (1H, d, $^3J_{HH} = 8.3$ Hz, H-Ar), 7.97 (2H, d, $^3J_{HH} = 7.5$ Hz, H-Ar), 8.39 (1H, s, H-Ar). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 17.71 (CH_3), 20.86 (CH_3), 29.84 ($C(CH_3)_3$), 55.91 ($C(CH_3)_3$), 113.73, 122.32, 122.89, 123.93, 127.73, 128.83, 130.89, 130.95 137.48, 137.96, 146.65 (C-Ar).

6-Methyl-*N*-(2,6-dimethylphenyl)-2-phenylimidazo[1,2-*a*]pyridin-3-amine (**4j**)—Colorless crystal (0.23 g, 72%): mp 165–168 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3150 (NH), 2750, 1662, 1621. MS, *m*/*z* (%): 328 (M^++1, 19), 327 (M^+, 87), 220 (29), 208 (100), 195 (29), 130 (13), 108 (44), 92 (87), 80 (37), 77 (33), 65 (68). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 1.86 (6H, s, $2CH_3$), 2.26 (3H, s, CH_3), 5.25 (1H, bs, NH), 6.61–7.87 (11H, m, H-Ar). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 16.37 ($2CH_3$), 17.82 (CH_3), 112.42, 115.78, 120.04, 120.49, 121.15, 121.74, 125.30, 126.57, 127.06, 128.02, 129.31, 135.13, 140.86, 144.85, 153.20 (C-Ar).

6-Methyl-*N*-(2,6-dimethylphenyl)-2-*p*-tolylimidazo[1,2-*a*]pyridin-3-amine (**4k**)—Colorless crystal (0.24 g, 70%): mp 209–211 °C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3150 (NH), 2750, 1663, 1621. MS, *m*/*z* (%): 342 (M^++1, 17), 341 (M^+, 35), 267 (69), 240 (40), 222 (27), 108 (35), 92 (100), 80 (21), 65 (58), 55 (37), 41 (20). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H (ppm) 1.85 (6H, s, $2CH_3$), 2.13 (3H, s, CH_3), 2.25 (3H, s, CH_3), 4.28 (1H, bs, NH), 6.61–7.79 (10H, m, H-Ar). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 16.85 ($2CH_3$), 18.31 (CH_3), 18.84 (CH_3), 113.00, 115.93, 120.53, 121.06, 121.62, 121.84, 125.77, 127.00, 128.84, 129.20, 129.84, 135.40, 141.37, 145.43, 153.65 (C-Ar).

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