Supporting Information

Heteroaromatic Imidazo[1,2-*a*]pyridines Synthesis from C-H/N-H Oxidative Cross-Coupling/Cyclization

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General Information

Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (boiling point is between 60-90 °C). Gradient flash chromatography was conducted eluting with a continuous gradient from petroleum to the indicated solvent, and they are listed as volume/volume ratios. NMR spectra were recorded on a Varian Mercury spectrometer at 300 MHz (¹H NMR), 75 MHz (¹³C NMR) or on a Bruker spectrometer at 400 MHz (¹H NMR), 100 MHz (¹³C NMR). Tetramethylsilane was used as an internal standard. All ¹H NMR spectra were reported in delta (δ) units, parts per million (ppm) downfield from the internal standard. Coupling constants are reported in Hertz (Hz). High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT instrument, accurate masses are reported for the molecular ion ([M]⁺). Selective ratios were recorded with a Varian GC 2000 gas chromatography instrument with a FID detector. GC-Ms spectra were recorded on a Varian GC-Ms 3900-2100T.

General Procedures for Preparation of

Imidazo[1,2-*a*]pyridines:



2-Phenylimidazo[1,2-*a*]pyridine (3aa).¹

A mixture of 2-aminopyridine **1a** (1.0 mmol), phenylacetylene **2a** (0.5 mmol), and Ag₂CO₃ (1.0 mmol) in dioxane (6 mL) was stirred in N₂ at 110 °C for 10 h. After completion of the reaction, as indicated by TLC and GC-Ms, the solid was filtered out and washed with dichloromethane. After removal of the solvent of the filtrate, the pure product was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 2:1) to afford **3aa** in 71% yield. $R_f = 0.4$ (petroleum ether/ethyl acetate 2:1). The spectroscopic data of all the products are presented below. All the known compounds gave satisfactory spectroscopic values and are analogue to spectroscopic data reported in the literature. ¹H NMR (300 MHz, CDCl₃): δ 7.97-7.92 (m, 3H), 7.73 (s, 1H), 7.59 (d, J = 9.0 Hz, 1H), 7.41 (t, J = 7.4 Hz, 2H), 7.32 (d, J = 6.9 Hz, 1H), 7.09 (t, J = 7.8 Hz, 1H), 6.66 (t, J = 6.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 146.0, 145.9, 134.0, 128.9, 128.2, 126.3, 125.8, 124.9, 117.7, 112.6, 108.4.



6-Methyl-2-phenylimidazo[1,2-*a*]pyridine (3ba).¹

Isolated yield: 73%; ¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 8.0 Hz, 2H), 7.85 (s, 1H), 7.74 (s, 1H), 7.52 (d, *J* = 9.2 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.99 (d, *J* = 9.2 Hz, 1H), 2.54 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 145.7, 145.0, 134.2, 128.9, 128.1, 128.0, 126.1, 123.6, 122.2, 117.0, 108.1, 18.4.



5-Methyl-2-phenylimidazo[1,2-*a*]pyridine (3ca).

Isolated yield: 68%; ¹H NMR (300 MHz, CDCl₃): δ 7.97 (d, *J* = 7.2 Hz, 2H), 7.65 (s, 1H), 7.51 (d, *J* = 9.0 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.08 (t, *J* = 8.0 Hz, 1H), 6.52 (d, *J* = 6.6 Hz, 1H), 2.50 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 146.3, 145.8, 134.5, 134.1, 128.8, 128.0, 126.2, 125.0, 114.9, 111.6, 105.4, 18.8. HRMS (APCI) calcd for C₁₄H₁₂N₂ [M]⁺: 208.1000; found 208.1003.



7-Methyl-2-phenylimidazo[1,2-*a*]pyridine (3da).²

Isolated yield: 76%; ¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 7.6 Hz, 3H), 7.73 (s, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.37 (s, 1H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.56 (d, *J* = 6.4 Hz, 1H), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 146.4, 145.7, 135.8, 134.2, 128.9, 128.0, 126.2, 125.0, 116.1, 115.2, 107.7, 21.6.



6-Chloro-2-phenylimidazo[1,2-*a*]pyridine (3ea).¹

Isolated yield: 63%; ¹H NMR (300 MHz, CDCl₃): δ 8.14 (s, 1H), 7.93 (d, *J* = 7.5 Hz, 2H), 7.81 (s, 1H), 7.57 (d, *J* = 9.6 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.2 Hz, 1H), 7.13 (d, *J* = 9.6 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 147.1, 144.3, 133.6, 129.1, 128.6, 126.3, 123.6, 120.8, 118.1, 108.8.



6-Bromo-2-phenylimidazo[1,2-*a*]pyridine (3fa).³

Isolated yield: 67%; ¹H NMR (300 MHz, CDCl₃): δ 8.22 (s, 1H), 7.92 (d, J = 7.8 Hz,

2H), 7.78 (s, 1H), 7.51 (d, *J* = 9.6 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.21 (d, *J* = 9.6 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 146.8, 144.3, 133.4, 129.1, 128.6, 128.3, 126.3, 125.8, 118.3, 108.5, 107.2.



6-Iodo-2-phenylimidazo[1,2-*a*]pyridine (3ga).⁴

Isolated yield: 70%; ¹H NMR (300 MHz, CDCl₃): δ 8.36 (s, 1H), 7.94-7.91 (m, 2H), 7.78 (s, 1H), 7.44-7.41 (m, 3H), 7.34-7.31 (m, 2H); ¹³C NMR (75 MHz, CDCl₃): δ 146.5, 144.5, 133.4, 132.9, 130.7, 129.1, 128.6, 126.4, 118.8, 108.1.



2-Phenylimidazo[2,1-*a*]isoquinoline (3ha).⁵

Isolated yield: 38%; ¹H NMR (400 MHz, CDCl₃): δ 8.74 (d, *J* = 8.0 Hz, 1H), 8.01 (d, *J* = 6.8 Hz, 2H), 7.90 (d, *J* = 7.2 Hz, 1H), 7.83 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 7,33 (t, *J* = 7.4 Hz, 1H), 7.04 (d, *J* = 7.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 144.3, 143.6, 134.3, 129.8, 129.0, 128.4, 128.4, 127.9, 127.2, 126.1, 124.1, 123.8, 123.2, 113.4, 110.1.



2-Phenylimidazo[1,2-*a*]quinoline (3ia).

Isolated yield: 43%; ¹H NMR (300 MHz, CDCl₃): δ 8.28 (s, 1H), 8.00 (d, J = 7.5 Hz, 2H), 7.90 (d, J = 8.1 Hz, 1H), 7.77 (d, J = 7.8 Hz, 1H), 7.64-7.56 (m, 2H), 7.50-7.41 (m, 4H), 7.35-7.31 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 145.2, 144.4, 134.0, 132.8, 129.5, 129.1, 128.1, 126.6, 126.1, 125.0, 123.6, 117.4, 115.4, 107.0. HRMS

(APCI) calcd for $C_{17}H_{12}N_2$ [M]⁺: 244.1000; found 244.0999.



6-(1*H*-Imidazol-1-yl)-2-phenylimidazo[1,2-*a*]pyridine (3ja).

Isolated yield: 40%; ¹H NMR (400 MHz, CDCl₃): δ 8.27 (s, 1H), 7.97-7.94 (m, 3H), 7.82 (s, 1H), 7.74 (d, J = 9.2 Hz, 1H), 7.48-7.44 (m, 2H), 7.39-7.35 (m, 1H), 7.27-7.24 (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 147.8, 144.6, 136.5, 133.3, 131.1, 129.1, 128.8, 126.4, 125.9, 121.4, 119.4, 119.4, 118.6, 109.5. HRMS (APCI) calcd for C₁₆H₁₂N₄ [M]⁺: 260.1062; found 260.1064.



2-*p*-Tolylimidazo[1,2-*a*]pyridine (3ab).⁶

Isolated yield: 77%; ¹H NMR (300 MHz, CDCl₃): δ 7.99 (d, *J* = 6.6 Hz, 1H), 7.83 (d, *J* = 7.8 Hz, 2H), 7.73 (s, 1H), 7.59 (d, *J* = 9.0 Hz, 1H), 7.22 (d, *J* = 7.8 Hz, 2H), 7.10 (t, *J* = 8.0 Hz, 1H), 6.68 (t, *J* = 6.6 Hz, 1H), 2.37 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 145.9, 145.7, 137.9, 131.0, 129.6, 126.0, 125.7, 124.7, 117.4, 112.4, 108.0, 21.5.



6-Methyl-2-*p*-tolylimidazo[1,2-*a*]pyridine (3bb).⁶

Isolated yield: 50%; ¹H NMR (300 MHz, CDCl₃): δ 7.82-7.78 (m, 3H), 7.65 (s, 1H), 7.49 (d, J = 9.3 Hz, 1H), 7.22 (d, J = 7.8 Hz, 2H), 6.95 (d, J = 9.0 Hz, 1H), 2.37 (s, 3H), 2.25 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 145.8, 144.9, 137.8, 131.3, 129.6, 127.8, 126.0, 123.5, 122.0, 116.8, 107.7, 21.5, 18.3.



2-(4-Methoxyphenyl)imidazo[1,2-*a*]pyridine (3ac).¹

Isolated yield: 55%; ¹H NMR (300 MHz, CDCl₃): δ 8.06 (d, *J* = 6.6 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.74 (s, 1H), 7.60 (d, *J* = 9.0 Hz, 1H), 7.13 (t, *J* = 8.0 Hz, 1H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.73 (t, *J* = 6.6 Hz, 1H), 3.84 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 159.8, 145.8, 127.5, 126.7, 125.7, 124.7, 117.5, 114.3, 112.5, 107.5, 55.5.



2-(4-Chlorophenyl)imidazo[1,2-*a*]pyridine (3ad).¹

Isolated yield: 48%; ¹H NMR (300 MHz, CDCl₃): δ 8.09 (d, J = 6.6 Hz, 1H), 7.88 (d, J = 8.7 Hz, 2H), 7.82 (s, 1H), 7.62 (d, J = 9.0 Hz, 1H), 7.40 (d, J = 8.4 Hz, 2H), 7.18 (t, J = 7.5 Hz, 1H), 6.78 (t, J = 6.5 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 146.0, 145.0, 133.9, 132.6, 129.2, 127.5, 125.9, 125.2, 117.8, 112.9, 108.5.



2-(4-Bromophenyl)imidazo[1,2-*a*]pyridine (3ae).⁶

Isolated yield: 34%; ¹H NMR (300 MHz, CDCl₃): δ 8.09 (d, J = 6.6 Hz, 1H), 7.83-7.80 (m, 3H), 7.61 (d, J = 9.3 Hz, 1H), 7.55 (d, J = 8.4 Hz, 2H), 7.18 (t, J = 8.0 Hz, 1H), 6.78 (t, J = 6.6 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 146.0, 145.0, 133.0, 132.1, 127.8, 125.9, 125.2, 122.1, 117.8, 112.9, 108.5.



2-(2-Methoxyphenyl)imidazo[1,2-*a*]pyridine (3af).⁷

Isolated yield: 57%; ¹H NMR (300 MHz, CDCl₃): δ 8.42 (d, J = 7.5 Hz, 1H), 8.16 (s, 1H), 8.06 (d, J = 6.6 Hz, 1H), 7.60 (d, J = 9.0 Hz, 1H), 7.29 (t, J = 7.5 Hz, 1H), 7.10

(t, J = 7.5 Hz, 2H), 6.97 (d, J = 8.1 Hz, 1H), 6.68 (t, J = 6.6 Hz, 1H), 3.95 (s, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 156.9, 144.5, 141.3, 128.9, 128.8, 125.8, 124.6, 122.5, 121.1, 117.3, 112.7, 112.1, 111.0, 55.5.



Ethyl 3-(imidazo[1,2-a]pyridin-2-yl)benzoate (3ag).

Isolated yield: 71%; ¹H NMR (300 MHz, CDCl₃): δ 8.56 (s, 1H), 8.17 (d, J = 7.8 Hz, 1H), 8.08 (d, J = 6.9 Hz, 1H), 8.00 (d, J = 7.8 Hz, 1H), 7.88 (s, 1H), 7.62 (d, J = 9.0 Hz, 1H), 7.49 (t, J = 7.8 Hz, 1H), 7.15 (t, J = 7.7 Hz, 1H), 6.74 (t, J = 6.6 Hz, 1H), 4.41 (q, J = 7.1 Hz, 2H), 1.41 (t, J = 7.2 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 166.7, 145.8, 144.9, 134.3, 131.1, 130.5, 129.1, 129.0, 127.1, 125.9, 125.1, 117.6, 112.8, 108.8, 61.2, 14.6. HRMS (APCI) calcd for C₁₆H₁₄N₂O₂ [M]⁺: 266.1055; found 266.1057.



Ethyl 3-(6-methylimidazo[1,2-*a*]pyridin-2-yl)benzoate (3bg).

Isolated yield: 55%; ¹H NMR (300 MHz, CDCl₃): δ 8.53 (s, 1H), 8.11 (d, *J* = 7.2 Hz, 1H), 7.97 (d, *J* = 7.2 Hz, 1H), 7.75-7.71 (m, 2H), 7.48-7.43 (m, 2H), 6.94 (d, *J* = 9.0 Hz, 1H), 4.40 (q, *J* = 6.8 Hz, 2H), 2.20 (s, 3H), 1.40 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 166.6, 144.8, 144.4, 134.4, 130.9, 130.2, 128.8, 128.1, 126.8, 123.4, 122.2, 116.7, 108.4, 61.1, 18.0, 14.4. HRMS (APCI) calcd for C₁₇H₁₆N₂O₂ [M]⁺: 280.1212; found 280.1214.



Ethyl 3-(6-chloroimidazo[1,2-*a*]pyridin-2-yl)benzoate (3eg).

Isolated yield: 46%; ¹H NMR (300 MHz, CDCl₃): δ 8.50 (s, 1H), 8.12-8.08 (m, 2H),

8.00 (d, J = 7.5 Hz, 1H), 7.81 (s, 1H), 7.54-7.45 (m, 2H), 7.09 (d, J = 9.3 Hz, 1H), 4.41 (q, J = 6.9 Hz, 2H), 1.42 (t, J = 7.1 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 166.6, 145.8, 144.1, 133.7, 131.1, 130.4, 129.3, 129.0, 127.0, 126.4, 123.6, 120.8, 117.9, 109.1, 61.3, 14.5. HRMS (APCI) calcd for C₁₆H₁₃ClN₂O₂ [M]⁺: 300.0666; found 300.0670.



2-(Benzo[d][1,3]dioxol-5-yl)imidazo[1,2-a]pyridine (3ah).

Isolated yield: 32%; ¹H NMR (400 MHz, CDCl₃): δ 8.08 (d, J = 6.8 Hz, 1H), 7.73 (s, 1H), 7.60 (d, J = 9.2 Hz, 1H), 7.46 (d, J = 8.0 Hz, 1H), 7.44 (s, 1H), 7.15 (t, J = 8.0 Hz, 1H), 6.88 (d, J = 8.0 Hz, 1H), 6.75 (t, J = 6.8 Hz, 1H), 5.99 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 148.3, 147.8, 145.9, 145.8, 128.4, 125.7, 124.8, 120.0, 117.6, 112.6, 108.9, 107.7, 106.9, 101.4. HRMS (APCI) calcd for C₁₄H₁₀N₂O₂ [M]⁺: 238.0742; found 238.0743.



2-(Thiophen-2-yl)imidazo[1,2-a]pyridine (3ai).⁸

Isolated yield: 14%; ¹H NMR (400 MHz, CDCl₃): δ 8.08 (d, J = 6.4 Hz, 1H), 7.78 (s, 1H), 7.61 (d, J = 9.2 Hz, 1H), 7.48-7.47 (m, 1H), 7.32-7.30 (m, 1H), 7.19-7.15 (m, 1H), 7.11-7.09 (m, 1H), 6.78 (t, J = 6.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 145.7, 141.1, 137.8, 128.0, 125.7, 125.4, 125.2, 124.0, 117.7, 112.9, 107.7.



Imidazo[1,2-*a*]pyridin-2-ylmethyl pivalate (3aj).

Isolated yield: 25%; ¹H NMR (300 MHz, CDCl₃): δ 8.09 (d, J = 6.9 Hz, 1H), 7.70-7.66 (m, 1H), 7.58 (s, 1H), 7.20-7.15 (m, 1H), 6.80-6.76 (m, 1H), 5.29 (s, 2H),

1.23 (s, 9H); ¹³C NMR (75 MHz, CDCl₃): δ 178.7, 147.5, 138.6, 126.0, 125.0, 117.9, 112.7, 111.3, 61.1, 39.0, 27.4. HRMS (APCI) calcd for C₁₃H₁₆N₂O₂ [M]⁺: 232.1212; found 232.1217.

General Procedures for Preparation of Zolimidine:



2-(4-(Methylthio)phenyl)imidazo[1,2-a]pyridine (3ak).

A mixture of 2-aminopyridine **1a** (1.0 mmol), (4-ethynylphenyl)(methyl)sulfane **2k** (0.5 mmol), and Ag₂CO₃ (1.0 mmol) in dioxane (6 mL) was stirred in N₂ at 110 °C for 10 h. After completion of the reaction, as indicated by TLC, the solid was filtered out and washed with dichloromethane. After removal of the solvent of the filtrate, the pure product was obtained by flash column chromatography on silica gel to afford **3ak** in 60% yield. ¹H NMR (400 MHz, CDCl₃): δ 8.04 (d, *J* = 6.8 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.77 (s, 1H), 7.60 (d, *J* = 9.2 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.13 (t, *J* = 7.8 Hz, 1H), 6.72 (t, *J* = 6.4 Hz, 1H), 2.50 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 145.9, 145.6, 138.4, 130.9, 126.9, 126.6, 125.8, 124.9, 117.7, 112.7, 108.1, 16.0. HRMS (APCI) calcd for C₁₄H₁₂N₂S [M]⁺: 240.0721; found 240.0718.



2-(4-(Methylsulfonyl)phenyl)imidazo[1,2-*a*]pyridine (Zolimidine) (4ak).

To a solution of 2-(4-(methylthio)phenyl)imidazo[1,2-*a*]pyridine **3ak** (113 mg) in EtOAc (3 mL) was added *m*CPBA (240 mg) at 0 °C. After stirring for 10 h, the mixture was quenched with water, and extracted with ethyl acetate, and the extract was washed with brine, and dried over sodium sulfate. The pure product was obtained by flash column chromatography on silica gel to afford **4ak** in 68% yield. ¹H NMR (400 MHz, CDCl₃): δ 8.16-8.13 (m, 3H), 8.00-7.98 (m, 3H), 7.64 (d, *J* = 9.2 Hz, 1H),

7.23 (t, J = 7.8 Hz, 1H), 6.84 (t, J = 6.8 Hz, 1H), 3.10 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 146.2, 143.8, 139.5, 139.5, 128.1, 126.8, 126.1, 125.8, 118.1, 113.3, 109.9, 44.9. HRMS (APCI) calcd for C₁₄H₁₂N₂O₂S [M]⁺: 272.0619; found 272.0616.

Procedure for the Synthesis of Phenylacetylene Silver⁹:

To a solution of 1-trimethylsilyl-2-phenylacetylene (5 mmol) in 20 mL aqueous methanol (H₂O:MeOH = 1:3), was added silver nitrate (5 mmol) at room temperature. The starting materials rapidly disappeared and a white precipitate formed within 5-15 min. This solid was recovered by filtration and washed with cold methanol (stored at 0° C). Subsequent drying led to the phenylacetylene silver as a white powder.

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Spectrum:





















7 147.13 144.32 133.55 133.55 128.56









146.84 144.32 133.44 133.44 128.55 128.55 128.32 128.32 128.32 126.30 126.30 125.80 125.80 118.33 108.54









146.51 144.45 133.39 13

















145.21 144.44 144.44 134.02 134.02 134.02 134.02 134.02 134.02 132.84 132.84 126.64 126.64 126.64 126.64 126.64 126.64 122.65 122.65 122.65 122.65 122.65 122.65 122.55 125 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.55 125.5









147.80 144.58 133.34 133.34 133.34 133.34 133.34 133.34 133.34 133.34 133.34 133.34 123.37 12

























кј-4-051-В.с

Solvent: CDC13 Ambient temperature Mercury-300BB "mercury300" Relax. delay 1.000 sec Fulse 19.1 degrees Fulse 19.1 degrees Vidth 17698.1 Hz DECOUPLE 41.300.0819042 NHz Power 42 dB continuously on VAIT2-15 modulated DAIT2-15 modulated DE DOUBLE 41.300.04194 For the Drondented DF Size 27658 Total time 2 hr, 24 min, 14 sec









146.00 144.94 133.02 127.81 127.81 125.87 125.87 125.87 125.87 125.20 12

















































145.74 - 141.14 - 137.75 - 137.75 - 137.75 - 137.75 - 137.75 - 125.35 - 125.35 - 125.35 - 125.15 - 125













