Synthesis of Indolo[1,2-*f*]phenanthridines from Palladium-Catalyzed Reactions of Arynes

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General All the reactions were carried out under the nitrogen atmosphere in oven-dried flasks. CH₃CN was distilled from CaH₂ using benzophenone as the indicator. Toluene and THF were distilled from Na using benzophenone as the indicator. O-(trimethylsilyl)phenyl triflate and 4,5-dimethyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate were prepared according to the method of reference 1. 1-(2-bromophenyl)indole and its derivatives were prepared according to the method of reference 2. The other materials were purchased from common commercial sources and used without additional purification. ¹H NMR spectra were recorded at 400 MHz using TMS as internal standard. ¹³C NMR spectra were recorded at 100 MHz using TMS as internal standard. Mass spectroscopy datas of the product were collected on an HRMS-ESI instrument.

Representative procedure for the preparation of 1-(2-bromophenyl)indole: Under N₂, indole (1.17 g, 10 mmol), CuI (95 mg, 0.5 mmol), ethane-1,2-diamine (30 mg, 0.5 mmol) , K₃PO₄ (4.24 g, 20 mmol) were added into a 50 mL oven-dried flask. A solution of 1-bromo-2-iodobenzene (2.83 g, 10 mmol) in dioxane (30 mL) was added , the reaction mixture was allowed to reflux at 110°C for 24 hours. After the reaction completed and cooled to room temperature, the reaction mixture was filtered through a pad of cellite and the solvent was removed under reduced pressure. The residue was further purified by column chromatography on a silica gel column and the product was obtained as yellow oil.

Representative procedure for the reaction of 1-(2-bromophenyl)indole with benzyne:

1-(2-bromophenyl)indole (136 mg, 0.5 mmol), CsF (228 mg, 1.5 mmol), Pd₂(dba)₃·CHCl₃ (13 mg, 0.0125 mmol), dppp (10 mg, 0.025 mmol) were placed in an oven-dried flask. CH₃CN (2 mL) and toluene (2 mL) were added under N₂. *o*-(Trimethylsilyl)phenyl triflate (149 mg, 0.5 mmol) in CH₃CN (1 mL) and toluene (1 mL) was added dropwise. After the completion of the addition, the reaction mixture was allowed to react at 110 °C for 24 h. Then the reaction mixture was cooled to room temperature and filtered through a pad of cellite. The filtrate was distilled under reduced pressure, and the product was obtained by flash chromatography on a silica gel column.

Characterization data of the starting materials

(1) 1-(2-bromophenyl)-5-methoxy-1H-indole [New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.74 (d, $J = 8.4$ Hz, 1 H), 7.41 (m, 2 H), 7.29 (m, 1 H), 7.20 (d, $J = 3.2$ Hz, 1 H), 7.14 (d, $J = 2.4$ Hz, 1 H), 6.98 (d, $J = 8.8$ Hz, 1 H), 6.84 (d, $J = 8.8$ Hz, 1 H), 6.60 (d, $J = 3.6$ Hz, 1 H), 3.86 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.5, 138.6, 133.9, 132.0, 129.6, 129.3, 129.2, 128.8, 128.2, 121.8, 112.4, 111.3, 102.8, 102.6, 55.8. HRMS (ESI) Calcd for $\text{C}_{15}\text{H}_{12}\text{BrNO}$: $[\text{M}+\text{H}]^+$ 302.0175; Found, 302.0185.

(2) 1-(2-bromophenyl)-6-methoxy-1H-indole [New compound]

¹H NMR (400 MHz, CDCl₃, TMS) δ 7.85 (d, *J* = 7.6 Hz, 1 H), 7.66 (d, *J* = 8.8 Hz, 2 H), 7.50 (d, *J* = 4.0 Hz, 1 H), 7.37 (m, 1 H), 7.21 (d, *J* = 3.2 Hz, 1 H), 6.96 (d, *J* = 9.2 Hz, 1 H), 6.73 (d, *J* = 3.2 Hz, 1 H), 6.69 (d, *J* = 2 Hz, 1 H), 3.85 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 156.8, 138.6, 137.6, 134.0, 129.8, 129.6, 129.5, 128.5, 127.7, 122.6, 122.0, 121.6, 110.3, 103.1, 94.3, 55.7. HRMS (ESI) Calcd for C₁₅H₁₂BrNO: [M+H]⁺ 302.0175; Found, 302.0185.

(3) 1-(2-bromophenyl)-5-methyl-1H-indole [New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.84 (d, $J = 8$ Hz, 1 H), 7.58 (s, 1 H), 7.50 (s, 1 H), 7.49 (s, 1 H), 7.37 (m, 1 H), 7.28 (d, $J = 3.2$ Hz, 1 H), 7.11 (m, 2 H), 6.71 (d, $J = 2.8$ Hz, 1 H), 2.57 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.7, 135.2, 133.9, 129.7, 129.6, 129.4, 128.8, 128.7, 128.3, 123.9, 121.9, 120.7, 110.3, 102.7, 21.5. HRMS (ESI) Calcd for $\text{C}_{15}\text{H}_{12}\text{BrN}$: $[\text{M}+\text{H}]^+$ 286.0226; Found, 286.0240.

(4) 1-(2-bromophenyl)-4-methyl-1H-indole [New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.86 (d, J = 8 Hz, 1 H), 7.52 (m, 2 H), 7.39 (m, 1 H), 7.34 (d, J = 3.2 Hz, 1 H), 7.23 (t, J = 7.6 Hz, 1 H), 7.09 (q, 2 H), 6.84 (d, J = 3.2 Hz, 1 H), 2.74 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.7, 136.6, 134.0, 130.5, 129.8, 129.5, 128.3, 128.2, 128.1, 122.5, 120.6, 108.3, 101.7, 18.8. HRMS (ESI) Calcd for $\text{C}_{15}\text{H}_{12}\text{BrN}$: $[\text{M}+\text{H}]^+$ 286.0226; Found, 286.0240.

(5) 1-(2-bromophenyl)-1H-indole [New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.85 (m, 2 H), 7.52 (m, 2 H), 7.40 (m, 1 H), 7.36 (d, $J = 3.6$ Hz, 1 H), 7.33 (m, 2 H), 7.25 (d, $J = 8.4$ Hz, 1 H), 6.84 (d, $J = 3.6$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.6, 136.8, 134.0, 129.8, 129.6, 128.8, 128.5, 128.4, 122.4, 122.0, 121.1, 120.4, 110.7, 103.3. HRMS (ESI) Calcd for $\text{C}_{14}\text{H}_{10}\text{BrN}$: $[\text{M}+\text{H}]^+$ 272.0069; Found, 272.0080.

(6) methyl 1-(2-bromophenyl)-1H-indole-4-carboxylate [New compound]

¹H NMR (400 MHz, CDCl₃, TMS) δ 7.96 (d, *J* = 6.8 Hz, 1 H), 7.73 (d, *J* = 8.4 Hz, 1 H), 7.55 (t, *J* = 7.8 Hz, 2 H), 7.50 (d, *J* = 3.2 Hz, 1 H), 7.47 (d, *J* = 3.2 Hz, 1 H), 7.41 (t, *J* = 7.4 Hz, 1 H), 7.34 (d, *J* = 2.8 Hz, 1 H), 7.26 (t, *J* = 8.0 Hz, 1 H), 4.02 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 167.8, 140.2, 137.5, 133.9, 130.8, 129.9, 129.8, 128.5, 128.0, 123.8, 121.8, 121.6, 115.6, 115.5, 104.3, 51.8. HRMS (ESI) Calcd for C₁₆H₁₂BrNO₂: [M+H]⁺ 330.0124; Found, 330.0126.

¹H NMR (400 MHz, CDCl₃, TMS) δ 7.80 (d, *J* = 8.0 Hz, 1 H), 7.52 (m, 2 H), 7.37 (d, *J* = 8.0 Hz, 1 H), 7.33 (d, *J* = 3.2 Hz, 1 H), 7.30 (d, *J* = 3.2 Hz, 1 H), 7.24 (t, *J* = 7.8 Hz, 1 H), 7.12 (d, *J* = 8.4 Hz, 1 H), 7.07 (d, *J* = 8.4 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃) δ 140.3, 134.0, 131.0, 132.8, 130.6, 129.6, 129.2, 128.6, 128.1, 126.5, 125.2, 121.9, 120.5, 111.5, 103.8. HRMS (ESI) Calcd for C₁₅H₉BrN₂: [M+Na]⁺ 318.9841; Found, 318.9850.

Characterization data of the products

(1) 11-methoxyindolo[1,2-f]phenanthridine [**T 2-1**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.48 (d, J = 8.4 Hz, 1 H), 8.34 (d, J = 8 Hz, 1 H), 8.26 (m, 2 H), 8.13 (m, 1 H), 7.59 (t, J = 7.6 Hz, 1 H), 7.52 (m, 2 H), 7.36 (t, J = 7.6 Hz, 1 H), 7.27 (s, 1 H), 7.20 (s, 1 H), 7.04 (d, J = 9.2 Hz, 1 H), 3.96 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.9, 137.0, 136.6, 135.9, 135.4, 131.4, 128.9, 127.9, 124.5, 124.4, 123.6, 123.5, 122.8, 122.4, 121.8, 115.6, 114.9, 111.2, 101.9, 94.8, 55.5. HRMS (ESI) Calcd for $\text{C}_{21}\text{H}_{15}\text{NO}$: $[\text{M}+\text{H}]^+$ 298.1222; Found, 298.1222. Yellow powder, mp 155-156°C.

¹H NMR (400 MHz, CDCl₃, TMS) δ 8.48 (d, *J* = 9.6 Hz, 1 H), 8.33 (d, *J* = 8 Hz, 1 H), 8.22 (m, 1 H), 8.10 (m, 1 H), 7.90 (d, *J* = 2.0 Hz, 1 H), 7.74 (d, *J* = 8.4 Hz, 1 H), 7.59 (t, *J* = 7.8 Hz, 1 H), 7.49 (m, 2 H), 7.37 (t, *J* = 7.8 Hz, 1 H), 7.27 (s, 1 H), 7.06 (d, *J* = 4.2 Hz, 1 H), 4.00 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 156.1, 135.9, 134.5, 134.3, 128.5, 128.1, 127.3, 126.45, 126.37, 124.6, 123.9, 123.6, 122.9, 122.3, 122.2, 121.3, 115.9, 110.8, 99.0, 95.9, 56.0. HRMS (ESI) Calcd for C₂₁H₁₅NO: [M+H]⁺ 298.1222; Found, 298.1222. Yellow powder, mp 184-185°C.

(3) 11-methylindolo[1,2-f]phenanthridine [**T 2-3**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.52 (d, J = 8.4 Hz, 1 H), 8.33 (d, J = 8 Hz, 1 H), 8.25 (m, 2 H), 8.13 (m, 1 H), 7.58 (m, 2 H), 7.49 (t, J = 4.6 Hz, 2 H), 7.35 (t, J = 7.6 Hz, 1 H), 7.21 (m, 2 H), 2.54 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.0, 135.2, 132.2, 131.2, 130.6, 128.7, 128.1, 127.7, 126.8, 126.2, 124.1, 123.9, 123.6, 122.8, 122.4, 121.9, 120.6, 116.1, 113.9, 95.7, 21.4. HRMS (ESI) Calcd for $\text{C}_{21}\text{H}_{15}\text{N}$: $[\text{M}+\text{H}]^+$ 282.1277; Found, 282.1273. Yellow powder, mp 178-180°C.

(4) 10-methylindolo[1,2-f]phenanthridine [**T 2-4**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.57 (d, J = 8.4 Hz, 1 H), 8.35 (d, J = 8 Hz, 1 H), 8.26 (m, 2 H), 8.20 (m, 1 H), 7.60 (t, J = 7.4 Hz, 1 H), 7.51 (t, J = 3.8 Hz, 2 H), 7.37 (t, J = 7.6 Hz, 1 H), 7.31 (t, J = 7.8 Hz, 1 H), 7.26 (s, 1 H), 7.17 (d, J = 6.8 Hz, 1 H), 2.72 (s, 3 H);
 ^{13}C NMR (100 MHz, CDCl_3) δ 136.0, 134.6, 133.6, 130.2, 130.0, 128.7, 128.1, 127.6, 126.7, 126.2, 124.0, 123.9, 123.0, 122.4, 122.1, 121.9, 119.5, 116.3, 111.8, 94.6, 19.0. HRMS (ESI) Calcd for $\text{C}_{21}\text{H}_{15}\text{N}$: $[\text{M}+\text{H}]^+$ 282.1277; Found, 282.1273. Yellow powder, mp 192-193°C.

(5) indolo[1,2-f]phenanthridine [**T 2-5**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.56 (d, J = 8.4 Hz, 1 H), 8.39 (d, J = 8.4 Hz, 1 H), 8.34 (d, J = 8 Hz, 1 H), 8.24 (m, 1 H), 8.15 (m, 1 H), 7.84 (d, J = 7.6 Hz, 1 H), 7.59 (t, J = 7.6 Hz, 1 H), 7.50 (m, 2 H), 7.36 (m, 3 H), 7.28 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.0, 135.2, 133.9, 130.3, 128.7, 128.2, 127.8, 126.8, 126.1, 124.1, 124.0, 123.0, 122.4, 122.1, 122.0, 121.8, 121.0, 116.3, 114.2, 96.2. HRMS (ESI) Calcd for $\text{C}_{20}\text{H}_{13}\text{N}$: $[\text{M}+\text{H}]^+$ 268.1121; Found, 268.1113. Yellow powder, mp 140-142°C.

(6) methyl indolo[1,2-f]phenanthridine-10-carboxylate [**T 2-6**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.59 (d, J = 8.4 Hz, 1 H), 8.51 (d, J = 8.4 Hz, 1 H), 8.37 (d, J = 8 Hz, 1 H), 8.27 (q, 2 H), 8.12 (d, J = 8 Hz, 1 H), 8.02 (s, 1 H), 7.60 (t, J = 7.6 Hz, 1 H), 7.55 (m, 2 H), 7.40 (t, J = 8.2 Hz, 2 H), 4.06 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.6, 136.9, 135.0, 134.5, 129.8, 128.5, 128.1, 128.0, 126.8, 125.4, 124.8, 124.4, 123.8, 123.1, 122.0, 121.9, 120.6, 120.5, 118.3, 116.1, 97.0, 51.6. HRMS (ESI) Calcd for $\text{C}_{22}\text{H}_{15}\text{NO}_2$: $[\text{M}+\text{Na}]^+$ 348.0995; Found, 348.0994. Yellow powder, mp 149-151°C.

(7) indolo[1,2-f]phenanthridine-11-carbonitrile [**T 2-7**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.32 (m, 3 H), 8.20 (d, $J = 7.2$ Hz, 1 H), 8.04 (s, 2 H), 7.54 (m, 4 H), 7.40 (t, $J = 7.8$ Hz, 1 H), 7.16 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.1, 134.89, 134.86, 129.8, 128.9, 128.7, 128.5, 126.9, 125.9, 125.1, 124.3, 124.2, 124.1, 122.4, 122.3, 121.7, 120.1, 116.4, 114.6, 104.7, 96.2, 21.4. HRMS (ESI) Calcd for $\text{C}_{21}\text{H}_{12}\text{N}_2$: $[\text{M}+\text{H}]^+$ 293.1073; Found, 293.1069. Yellow powder, mp 228-230°C.

(8) 11-methoxy-6,7-dimethylindolo[1,2-f]phenanthridine [**T 2-8**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.48 (d, J = 8.4 Hz, 1 H), 8.33 (d, J = 8 Hz, 1 H), 8.30 (d, J = 18 Hz, 1 H), 7.99 (s, 1 H), 7.88 (s, 1 H), 7.56 (t, J = 8 Hz, 1 H), 7.34 (t, J = 7.4 Hz, 1 H), 7.27 (s, 1 H), 7.25 (s, 1 H), 7.01 (d, J = 8.8 Hz, 1 H), 3.95 (s, 3 H), 2.44 (s, 3 H), 2.43 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.9, 137.0, 136.6, 135.9, 135.4, 131.4, 128.9, 127.9, 124.5, 124.4, 123.6, 123.5, 122.8, 122.4, 121.8, 115.6, 114.9, 111.2, 101.9, 94.8, 55.5, 20.2, 19.8. HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{19}\text{NO}$: $[\text{M}+\text{H}]^+$ 326.1539; Found, 326.1532. Yellow powder, mp 143-145°C.

(9) 12-methoxy-6,7-dimethylindolo[1,2-f]phenanthridine [**T 2-9**, New compound]

¹H NMR (400 MHz, CDCl₃, TMS) δ 8.36 (d, *J* = 8.4 Hz, 1 H), 8.17 (d, *J* = 8 Hz, 1 H), 7.84 (s, 1 H), 7.81 (s, 1 H), 7.70 (s, 1 H), 7.69 (d, *J* = 8.8 Hz, 1 H), 7.48 (t, *J* = 7.8 Hz, 1 H), 7.45 (d, *J* = 4.4 Hz, 1 H), 7.27 (t, *J* = 7.4 Hz, 1 H), 7.05 (t, *J* = 9.2 Hz, 1 H), 7.03 (s, 1 H), 4.00 (s, 3 H), 2.34 (s, 3 H), 2.33 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 155.8, 137.1, 135.5, 133.9, 129.6, 129.3, 127.7, 124.8, 124.24, 124.18, 124.1, 123.5, 122.8, 122.7, 122.3, 121.1, 115.7, 110.4, 99.2, 94.1, 56.1, 20.1, 19.8. HRMS (ESI) Calcd for C₂₃H₁₉NO: [M+H]⁺ 326.1539; Found, 326.1532. Yellow powder, mp 89-91°C.

(10) 6,7,11-trimethylindolo[1,2-f]phenanthridine [**T 2-10**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.51 (d, $J = 8.4$ Hz, 1 H), 8.30 (d, $J = 8$ Hz, 1 H), 8.27 (d, $J = 8.4$ Hz, 1 H), 7.97 (s, 1 H), 7.87 (s, 1 H), 7.62 (s, 1 H), 7.56 (t, $J = 7.8$ Hz, 1 H), 7.34 (t, $J = 7.8$ Hz, 1 H), 7.28 (s, 1 H), 7.20 (d, $J = 8.4$ Hz, 1 H), 2.57 (s, 3 H), 2.43 (s, 3 H), 2.42 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.1, 136.6, 135.7, 135.4, 132.1, 130.9, 130.7, 127.9, 124.6, 123.9, 123.5, 123.2, 122.9, 122.6, 121.9, 120.4, 120.1, 116.0, 113.8, 94.8, 21.3, 20.2, 19.8. HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{19}\text{N}$: $[\text{M}+\text{H}]^+$ 310.1590; Found, 310.1592. Yellow powder, mp 135-136°C.

(11) 6,7,10-trimethylindolo[1,2-f]phenanthridine[**T 2-11**, New compound]

^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.50 (d, J = 8.4 Hz, 1 H), 8.21 (d, J = 8.4 Hz, 1 H), 7.86 (s, 1 H), 7.81 (s, 1 H), 7.52 (t, J = 7.8 Hz, 1 H), 7.30 (t, J = 7.8 Hz, 1 H), 7.18 (s, 1 H), 7.16 (s, 1 H), 2.73 (s, 3 H), 2.36 (s, 3 H), 2.35 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.1, 136.6, 135.7, 134.9, 133.4, 130.1, 129.9, 127.9, 124.53, 124.49, 123.9, 123.5, 122.9, 122.7, 122.1, 121.7, 121.6, 116.1, 111.8, 93.3, 20.2, 19.8, 19.0. HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{19}\text{N}$: $[\text{M}+\text{H}]^+$ 310.1590; Found, 310.1592. Yellow powder, mp 123-124°C.

¹H NMR (400 MHz, CDCl₃, TMS) δ 8.51 (d, *J*= 8.4 Hz, 1 H), 8.38 (d, *J*= 6.8 Hz, 1 H), 8.24 (d, *J*= 8.0 Hz, 1 H), 7.90 (s, 1 H), 7.82 (s, 1 H), 7.54 (t, *J*= 8.0 Hz, 1 H), 7.39 (m, 2 H), 7.32 (t, *J*= 7.8 Hz, 1 H), 7.17 (m, 1 H), 2.39 (s, 3 H), 2.37 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 137.1, 136.7, 135.6, 135.4, 133.7, 130.4, 127.9, 124.62, 124.59, 123.8, 123.6, 122.9, 122.7, 122.1, 121.54, 121.52, 120.7, 116.1, 114.1, 95.1, 20.2, 19.8. HRMS (ESI) Calcd for C₂₂H₁₇N: [M+H]⁺ 296.1434; Found, 296.1425. Yellow powder, mp 103-105°C.

UV-Vis absorbance spectra and fluorescence spectra of the selected new compounds

UV-Vis absorbance spectra and Fluorescence spectra of T 2-1 (Left) and T 2-2 (Right)

UV-Vis absorbance spectra and Fluoresence spectra of T 2-3 (Left) and T 2-4 (Right)

UV-Vis absorbance spectra and Fluoresence spectra of T 2-5

UV-Vis absorbance spectra and fluorescence spectra of T 2-6 (Left) and T 2-7 (Right)

Table 1 Fluorescence quantum yield of the above new compounds ^a

entry	R ₁ =	quantum yield ^b
1	5-MeO (T2-1)	0.39
2	6-MeO (T2-2)	0.41
3	5-Me (T2-3)	0.36
4	4-Me (T2-4)	0.37
5	H (T2-5)	0.28
6	4-COOMe (T2-6)	0.28
7	5-CN (T2-7)	0.27

^a Exciting wavelength was 275 nm

^b Calculated in THF based on terphenyl as the standard ($\Phi=0.93$, 287nm)

References

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