Ortho Selective Direct Cross-Coupling Reaction of 2-Aryloxazolines and 2-Arylimidazolines with Aryl and Alkenyl Halides Catalyzed by Ruthenium Complexes

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General Experimental Methods and Materials

All reactions were performed in Schlenk tubes under a N₂ atmosphere. Anhydrous NMP was purchased and used as received. K₂CO₃ was dried at 250 °C under reduced pressure and stored under N₂. Aryloxazolines **1b-1i** were prepared from corresponding benzoic acid chlorides and ethanolamine, as described in the literature.¹ *N*-Acylphenylimidazolines **7b-7d** were prepared by acylation of 2-phenyl-2-imidazoline (**7a**) with the corresponding acid anhydrides, as described in the literature.² *N*-Tosyl-2-phenyl-2-imidazoline (**7e**) was prepared in a similar procedure using tosyl chloride.

Spectroscopic and Analytical Data

3aa. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.77 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.49 (td, *J* = 7.8, 1.4 Hz, 1H), 7.41-7.29 (m, 7H), 4.10 (t, *J* = 9.5 Hz, 2H), 3.91 (t, *J* = 9.5 Hz, 2H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 166.0, 141.9, 141.3, 130.5, 130.3, 130.1, 128.3, 128.0, 127.6, 127.12, 127.06, 67.7, 55.0. IR (neat): 2925, 1654, 697 cm⁻¹. HRMS (ESI). Calcd for C₁₅H₁₄NO (M+H⁺): 224.1070. Found: 224.1065.

4aa. Mp 140.6-141.2 °C; ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.52 (t, *J* = 7.7 Hz, 1H), 7.45 (dt, *J* = 7.3, 1.8 Hz, 4H), 7.39 (d, *J* = 7.7 Hz, 2H), 7.38 (td, *J* = 7.3, 1.8 Hz, 4H), 7.32 (tt, *J* = 7.3, 1.8 Hz, 2H), 3.88 (t, *J* = 9.4 Hz, 2H), 3.58 (t, *J* = 9.4 Hz, 2H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 163.9, 142.4, 140.9, 129.5, 128.8, 128.6, 128.0, 127.6, 127.2, 67.3, 55.1. IR (KBr): 2893, 1655, 1040, 758 cm⁻¹. Anal. Calcd for C₂₁H₁₇NO: C, 84.25; H, 5.72; N, 4.68. Found: C, 84.03; H, 5.85; N, 4.63.

3ba. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.3 (d, *J* = 7.8 Hz, 1H), 7.49-7.25 (m, 8H), 3.61 (s, 2H), 1.18 (s, 6H); ¹³C-NMR (CDCl₃, 100 MHz): δ (ppm) 164.5, 141.8, 141.2, 130.2, 130.1, 129.8, 128.7, 128.4, 127.8, 127.0, 83.9, 67.2, 27.1. IR (neat): 2972, 1647, 1043, 698 cm⁻¹. Anal. Calcd for C₁₇H₁₇NO: C, 81.24; H, 6.82; N, 5.57. Found: C, 81.09; H, 7.03; N, 5.49.

4ba. Mp 90.0-90.5 °C. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.51-7.23 (m, 13H), 3.30 (s, 2H), 0.96 (s, 6H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 162.1, 142.1, 140.7, 129.1, 128.9, 128.6, 128.2, 127.8, 127.2, 83.4, 67.3, 27.0. IR (KBr): 2977, 1651, 1265, 701 cm⁻¹. Anal. Calcd for C₂₃H₂₁NO: C, 84.37; H, 6.46; N, 4.28. Found: C, 84.47; H, 6.74; N, 4.24.

3ca.³ ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.72 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.47 (td, *J* = 7.6, 1.4 Hz, 1H), 7.44-7.25 (m, 7H), 3.78 (s, 2H), 1.28 (s, 6H); ¹³C-NMR (CDCl₃, 125 MHz): 163.7, 141.7, 141.2,

130.3, 130.1, 130.0, 128.4, 128.1, 127.9, 127.1, 127.0, 119.6, 79.4, 67.5, 28.0. IR (neat): 3058, 2965, 1655, 1311, 1038 cm^{-1} .

3da. Mp 72.5-74.0 °C; ^1H -NMR (CDCl_3 , 250 MHz): δ (ppm) 7.43-7.28 (m, 6H), 7.22-7.18 (m, 2H), 4.09 (t, $J = 9.3$ Hz, 2H), 3.83 (t, $J = 9.3$ Hz, 2H), 2.41 (s, 3H); ^{13}C -NMR (CDCl_3 , 62.5 MHz): δ (ppm) 164.4, 142.0, 141.2, 137.5, 129.4, 129.0, 128.4, 128.2, 128.0, 127.2, 127.1, 67.1, 55.1, 19.8. IR (KBr): 2924, 1664, 1043, 764 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{15}\text{NO}$: C, 80.98; H, 6.37; N, 5.90. Found: C, 80.89; H, 6.51; N, 5.81.

3ea. ^1H -NMR (CDCl_3 , 500 MHz): δ (ppm) 7.68 (d, $J = 8.3$ Hz, 1H), 7.41-7.33 (m, 5H), 7.25-7.19 (m, 2H), 4.11-4.07 (m, 2H), 3.92-3.87 (m, 2H), 2.41 (s, 3H); ^{13}C -NMR (CDCl_3 , 125 MHz): δ (ppm) 166.1, 141.7, 141.5, 140.6, 131.1, 130.1, 129.0, 128.3, 128.1, 127.9, 127.8, 127.0, 124.7, 67.6, 54.9, 21.4. IR (neat): 2925, 1653, 1073, 1040, 699 cm^{-1} . HRMS (ESI). Calcd for $\text{C}_{16}\text{H}_{16}\text{NO}$ ($\text{M}+\text{H}^+$): 238.1226. Found: 238.1226.

4ea. Mp 162.6-163.1 °C. ^1H -NMR (CDCl_3 , 400 MHz): δ (ppm) 7.45-7.41 (m, 4H), 7.38-7.32 (m, 4H), 7.32-7.27 (m, 2H), 7.20-7.19 (m, 2H), 3.84 (2H, t, $J = 9.4$ Hz), 3.55 (2H, t, $J = 9.4$ Hz), 2.42 (3H, s); ^{13}C -NMR (CDCl_3 , 100 MHz): δ (ppm) 164.0, 142.2, 141.0, 139.4, 129.5, 128.4, 127.8, 127.0, 124.6, 67.1, 55.0, 21.3. IR (KBr): 2892, 1656, 1039, 765, 701 cm^{-1} . Anal. Calcd for $\text{C}_{22}\text{H}_{19}\text{NO}$: C, 84.31; H, 6.11; N, 4.47. Found: C, 84.44; H, 6.24; N, 4.44.

3fa. ^1H -NMR (CDCl_3 , 250 MHz): δ (ppm) 7.59 (s, 1H), 7.35-7.24 (m, 7H), 4.09 (t, $J = 9.0$ Hz, 2H), 3.89 (t, $J = 9.0$ Hz, 2H), 2.39 (s, 3H); ^{13}C -NMR (CDCl_3 , 62.5 MHz): δ (ppm) 166.2, 141.2, 138.9, 136.8, 131.2, 130.6, 130.2, 128.2, 127.9, 127.2, 126.9, 67.7, 54.9, 20.8. IR (neat): 2925, 1651, 702 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{15}\text{NO}$: C, 80.98; H, 6.37; N, 5.90. Found: C, 80.76; H, 6.45; N, 5.79.

4fa. ^1H -NMR (CDCl_3 , 250 MHz): δ (ppm) 7.47-7.21 (m, 12H), 3.77 (t, $J = 9.4$ Hz, 2H), 3.43 (t, $J = 9.4$ Hz, 2H), 2.16 (s, 3H); ^{13}C -NMR (CDCl_3 , 125 MHz): δ (ppm) 164.0, 140.9, 139.5, 139.3, 135.3, 131.2, 129.3, 128.60, 128.57, 128.55, 128.50, 128.48, 128.0, 127.9, 127.6, 127.0, 126.9, 67.0, 54.9, 20.5. IR (KBr): 2925, 1655, 1261, 1094, 701 cm^{-1} . HRMS (ESI). Calcd for $\text{C}_{22}\text{H}_{20}\text{NO}$ ($\text{M}+\text{H}^+$): 314.1539. Found: 314.1541.

3ga. ^1H -NMR (CDCl_3 , 500 MHz): δ (ppm) 8.06 (d, $J = 1.7$ Hz, 1H), 7.74 (dd, $J = 1.7, 8.0$ Hz, 1H), 7.51 (d, $J = 8.0$ Hz, 1H), 7.41-7.36 (m, 5H), 4.15 (t, $J = 9.2$ Hz, 2H), 3.94 (t, $J = 9.2$ Hz, 2H); ^{13}C -NMR (CDCl_3 , 125 MHz): δ (ppm) 164.8, 145.3, 139.9, 130.9, 129.5 (q, $J_{\text{C-F}} = 33$ Hz), 128.3, 128.2, 127.9, 127.3 (q, $J_{\text{C-F}} = 3.8$ Hz), 127.1 (q, $J_{\text{C-F}} = 3.5$ Hz), 123.8 (q, $J_{\text{C-F}} = 271$ Hz), 68.0, 55.1. IR (KBr): 2973, 1653, 1313, 1129 cm^{-1} . Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{F}_3\text{NO}$: C, 65.98; H, 4.15; N, 4.81. Found: C, 65.97; H, 4.54; N, 4.66.

3ha. Mp 117.4-118.2 °C. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 8.08 (d, *J* = 8.0 Hz, 1H), 7.93 (d, *J* = 7.5 Hz, 1H), 7.85 (d, *J* = 8.8 Hz, 1H), 7.60-7.30 (m, 8H), 4.25-4.16 (m, 2H), 4.03-3.94 (m, 2H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 163.9, 141.2, 139.8, 132.3, 131.5, 129.9, 128.6, 128.0, 127.9, 127.5, 127.3, 127.1, 126.1, 125.4, 124.9, 67.3, 55.4. IR (KBr): 2878, 1663, 1001, 767, 704 cm⁻¹. Anal. Calcd for C₁₉H₁₅NO: C, 83.49; H, 5.53; N, 5.12. Found: C, 83.36; H, 5.89; N, 4.98.

4ha. Mp 134.0-135.4 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 8.02 (d, *J* = 8.4 Hz, 1H), 7.90 (d, *J* = 8.1 Hz, 1H), 7.60-7.25 (m, 13H), 3.49-2.87 (m, 4H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 163.1, 142.4, 141.8, 141.6, 140.0, 133.6, 131.6, 130.5, 129.1, 128.6, 128.3, 127.7, 127.1, 127.0, 126.3, 125.4, 124.9, 119.6, 66.2, 54.4. IR (KBr): 3052, 1666, 761, 705 cm⁻¹. Anal. Calcd for C₂₅H₁₉NO: C, 85.93; H, 5.48; N, 4.01. Found: C, 85.62; H, 5.65; N, 3.85.

3ia. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 8.33 (s, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.86 (d, *J* = 8.1 Hz, 1H), 7.84 (s, 1H), 7.58-7.51 (m, 2H), 7.45-7.47 (m, 2H), 7.44-7.40 (m, 2H), 7.38-7.34 (m, 1H), 4.15 (t, *J* = 9.0 Hz, 2H), 3.96 (t, *J* = 9.0 Hz, 2H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 166.1, 141.4, 138.5, 134.1, 131.7, 130.6, 129.3, 128.9, 128.4, 128.3, 128.0, 127.8, 127.6, 127.1, 126.6, 67.9, 55.1. IR (neat): 3055, 1651, 1072, 701 cm⁻¹. Anal. Calcd for C₁₉H₁₅NO: C, 83.49; H, 5.53; N, 5.12. Found: C, 83.23; H, 5.73; N, 4.99.

4ia. Mp 57.5-59.0 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.90 (d, *J* = 8.2 Hz, 1H), 7.89 (s, 1H), 7.61 (d, *J* = 8.5 Hz, 1H), 7.56-7.32 (m, 12H), 3.78 (t, *J* = 9.4 Hz, 2H), 3.49 (t, *J* = 9.4 Hz, 2H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 163.9, 140.9, 140.7, 138.4, 138.2, 133.7, 131.2, 130.2, 128.8, 128.3, 128.1, 128.0, 127.7, 127.4, 127.2, 127.1, 126.9, 126.6, 126.4, 67.2, 54.9; IR (KBr): 3056, 1655, 1254, 1085, 702 cm⁻¹. HRMS (ESI). Calcd for C₂₂H₂₀NO (M+H⁺): 350.1539. Found: 350.1540.

3db. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.34 (d, *J* = 8.7 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 1H), 7.17 (m, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 4.14 (t, *J* = 9.3 Hz, 2H), 3.86 (t, *J* = 9.3 Hz, 2H), 3.80 (s, 3H), 2.39 (s, 3H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 164.5, 158.7, 141.5, 137.3, 133.6, 129.4, 129.3, 128.5, 128.0, 127.1, 113.3, 67.0, 55.1, 55.0, 19.7. IR (neat): 2957, 1666, 1515, 1041 cm⁻¹. HRMS (ESI). Calcd for C₁₇H₁₈NO₂ (M+H⁺): 268.1332. Found: 268.1332.

3dc. Mp 64-65 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.32 (t, *J* = 7.6 Hz, 1H), 7.27 (ddd, *J* = 8.2, 7.4, 1.7 Hz, 1H), 7.20 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.20-7.17 (m, 2H), 6.94 (td, *J* = 7.4, 1.0 Hz, 1H), 7.91 (dd, *J* = 8.2, 1.0 Hz, 1H), 4.04 (t, *J* = 9.4 Hz, 2H), 3.77 (t, *J* = 9.4 Hz, 2H), 3.73 (s, 3H), 2.42 (s, 3H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 164.5, 156.4, 138.6, 137.1, 130.7, 130.1, 129.0, 128.9, 128.8, 128.5, 128.1, 120.1, 110.6, 66.9, 55.5, 55.0, 20.1. IR (KBr): 3016, 1656, 1467, 936, 763 cm⁻¹. Anal. Calcd for C₁₇H₁₇NO₂: C, 76.38; H, 6.41; N, 5.24. Found: C, 76.00; H, 6.58; N, 5.09.

3dd. Mp 87.6-88.4 °C. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.96 (d, *J* = 8.3 Hz, 2H), 7.50 (d, *J* = 8.3 Hz, 2H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 7.5 Hz, 1H), 7.20 (d, *J* = 7.5 Hz, 1H), 4.14 (t, *J* = 9.5 Hz, 2H), 3.85 (t, *J* = 9.5 Hz, 2H), 2.60 (s, 3H), 2.35 (s, 3H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 197.6, 163.9, 145.9, 140.6, 137.6, 135.6, 129.5, 129.4, 128.4, 128.0, 127.9, 126.8, 67.1, 55.0, 26.4, 19.6. IR (KBr): 2981, 1662, 1038, 935, 768 cm⁻¹. Anal. Calcd for C₁₈H₁₇NO₂: C, 77.40; H, 6.13; N, 5.01. Found: C, 77.06; H, 6.09; N, 4.76.

3de. Mp 109.4-109.9 °C. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.36-7.00 (m, 7H), 4.10-3.85 (m, 2H), 3.77-3.67 (m, 2H), 2.41 (s, 3H), 2.13 (s, 3H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 174.9, 164.1, 141.8, 140.4, 137.2, 136.1, 129.5, 129.3, 129.0, 128.8, 127.2, 127.0, 124.7, 67.0, 55.0, 20.2, 19.8. IR (KBr): 2954, 1663, 1038, 768, 729 cm⁻¹. Anal. Calcd for C₁₇H₁₇NO: C, 81.24; H, 6.83; N, 5.51. Found: C, 81.11; H, 6.93; N, 5.42.

3dg. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.86 (d, *J* = 8.2 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.48-7.36 (m, 5H), 7.31 (d, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 3.82-3.77 (m, 1H), 3.73-3.67 (m, 1H), 3.59-3.54 (m, 2H), 2.47 (s, 3H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 164.0, 140.4, 138.7, 137.4, 133.3, 131.9, 129.6, 129.2, 129.0, 128.3, 127.8, 127.5, 126.7, 126.3, 125.7, 125.5, 124.8, 66.8, 54.8, 20.0. IR (neat): 3057, 1661, 1045, 780, 701 cm⁻¹. HRMS (ESI). Calcd for C₂₀H₁₈NO (M+H⁺): 288.1383. Found: 288.1382.

3dh. Mp 100.0-100.8 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.77 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 1H), 7.38-7.30 (m, 5H), 7.07 (d, *J* = 7.7 Hz, 1H), 3.76-3.69 (m, 1H), 3.60-3.56 (m, 1H), 3.53-3.47 (m, 2H), 2.46 (s, 3H), 2.21 (s, 3H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 164.0, 139.7, 137.5, 136.4, 133.8, 132.9, 131.5, 129.9, 129.5, 129.1, 128.2, 127.8, 127.3, 127.2, 126.4, 125.5, 124.5, 66.7, 54.7, 20.7, 19.9. IR (KBr): 2925, 1655, 1046, 937 cm⁻¹. Anal. Calcd for C₂₁H₁₉NO: C, 83.69; H, 6.35; N, 4.65. Found: C, 83.32; H, 6.58; N, 4.62.

6da (*E:Z* = 1:0.1). ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.35 (d, *J* = 7.7 Hz, 1H), 7.26 (t, *J* = 7.7 Hz, 0.1H), 7.22 (t, *J* = 7.7 Hz, 1H), 7.16 (d, *J* = 7.7 Hz, 0.1H), 7.09 (d, *J* = 7.7 Hz, 0.1H), 7.05 (d, *J* = 7.7 Hz, 1H), 6.49-6.44 (m, 1.1H), 6.20 (dq, *J* = 15.6, 6.6 Hz, 1H), 5.80 (dq, *J* = 11.6, 7.1 Hz, 0.1H), 4.41 (t, *J* = 9.5 Hz, 2H), 4.36 (t, *J* = 9.5 Hz, 0.2H), 4.10 (t, *J* = 9.5 Hz, 2H), 4.06 (t, *J* = 9.5 Hz, 0.2H), 2.34 (s, 0.3H), 2.31 (s, 3H), 1.86 (dd, *J* = 6.6, 1.7 Hz, 3H), 1.78 (dd, *J* = 7.1, 1.9 Hz, 0.3H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm); 164.3, 137.2, 137.0, 136.7, 129.3, 128.8, 128.21, 128.19, 128.13, 127.9, 127.8, 127.7, 127.1, 126.6, 122.5, 67.19, 67.16, 55.2, 55.1, 19.62, 19.58, 18.8, 14.5. IR (neat): 2932, 1663, 1045 cm⁻¹. HRMS (ESI). Calcd for C₁₃H₁₆NO (M+H⁺): 202.1226. Found: 202.1225.

6db (*E:Z* = 1:0.03). ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.53 (d, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 7.4 Hz, 2H), 7.36-7.20 (m, 5.21H), 7.16-7.11 (m, 1.03H), 7.04(d, *J* = 16.1 Hz, 1H), 6.67 (d, *J* = 12.2 Hz, 0.03H), 6.59 (d, *J* = 12.2 Hz, 0.03H), 4.44 (t, *J* = 9.6 Hz, 2H), 4.38-4.33 (m, 0.06H), 4.14 (t, *J* = 9.6 Hz, 2H), 4.09-4.04 (m, 0.06H), 2.37 (s, 0.09H), 2.36 (s, 3H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm): 164.4, 164.3, 137.44, 137.42, 137.39, 137.29, 136.9, 130.8, 129.6, 128.8, 129.1, 128.8, 128.7, 128.6, 128.5, 128.1, 128.0, 127.7, 127.0, 126.8, 126.7, 126.3, 122.8, 67.3, 67.2, 55.33, 55.26, 19.8, 19.7. IR (neat): 2961, 1660, 1045 cm⁻¹. Anal. Calcd for C₁₈H₁₇NO: C, 82.10; H, 6.31; N, 5.32. Found: C, 81.88; H, 6.51; N, 5.23.

Mixture of (*E*)-6db, (*Z*)-6db, and 6dc (1:0.04:0.21). ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.72-7.69 (m, 0.21H), 7.54-7.50 (m, 2.21H), 7.46 (d, *J* = 7.4 Hz, 2H), 7.36-7.20 (m, 6.54H), 7.17-7.12 (m, 1.04H), 7.04 (d, *J* = 16.1 Hz, 1H), 6.67 (d, *J* = 12.2 Hz, 0.04H), 6.59 (d, *J* = 12.2 Hz, 0.04H), 5.57 (d, *J* = 1.2 Hz, 0.21H), 5.32 (d, *J* = 1.2 Hz, 0.21H), 4.44 (t, *J* = 9.6 Hz, 2H), 4.38-4.33 (m, 0.08H), 4.14 (t, *J* = 9.6 Hz, 2H), 4.09-4.04 (m, 2H), 3.85 (t, *J* = 9.4 Hz, 0.42H), 3.65 (t, *J* = 9.4 Hz, 0.42H), 2.37 (s, 0.12H), 2.36 (s, 3H), 2.35 (s, 0.63H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 164.2, 163.9, 149.5, 141.0, 137.4, 137.33, 137.26, 136.8, 130.7, 129.51, 129.48, 129.33, 129.30, 129.1, 129.0, 128.5, 128.0, 127.9, 127.8, 127.7, 127.6, 127.4, 127.31, 127.29, 127.0, 126.6, 126.2, 122.8, 115.4, 67.3, 66.7, 55.3, 54.8, 19.8, 19.7. IR (neat): 2961, 1660, 1046 cm⁻¹. Anal. Calcd for C₁₈H₁₇NO: C, 82.10; H, 6.31; N, 5.32. Found: C, 82.01; H, 6.58; N, 5.23.

8ba. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.54-7.33 (m, 9H), 4.08-3.82 (m, 2H), 3.81-3.40 (m, 2H), 1.55 (s, 3H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 166.8, 140.4, 132.7, 131.9, 131.8, 131.71, 131.70, 130.9, 130.7, 130.2, 129.7, 128.9, 128.5, 128.3, 128.21, 128.20, 127.4, 52.5, 47.3, 23.2. IR (neat): 3244, 1638, 1557, 742, 699 cm⁻¹. HRMS (ESI). Calcd for C₁₇H₁₇N₂O (M+H⁺): 265.1335. Found: 265.1336.

9ba. Mp 121.9-127.8 °C; ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.53-7.25 (m, 13H), 3.61 (t, *J* = 8.8 Hz, 2H), 3.52-3.28 (m, 2H), 1.63 (s, 3H); ¹³C-NMR (DMSO-*d*₆, 125 MHz, 330 K): δ (ppm) 165.8, 140.4, 128.5, 127.9, 127.2, 40.6, 28.4. IR (KBr): 3057, 1629, 1384, 765, 700 cm⁻¹. Anal. Calcd for C₂₃H₂₀N₂O: C, 81.15; H, 5.92; N, 8.23. Found: C, 80.84; H, 5.95; N, 8.01.

8ca. Mp 110-118 °C. ¹H-NMR (CDCl₃, 250 MHz): δ (ppm) 7.61 (d, *J* = 7.0 Hz, 1H), 7.47-7.26 (m, 8H), 3.95 (t, *J* = 8.2 Hz, 2H), 3.83-3.65 (m, 2H), 0.81 (s, 9H); ¹³C-NMR (CDCl₃, 62.5 MHz): δ (ppm) 175.2, 163.1, 141.1, 139.3, 132.4, 129.4, 129.3, 128.5, 128.4, 127.9, 127.2, 127.1, 55.2, 47.8, 39.3, 26.7. IR (KBr): 3052, 1655, 1316, 736 cm⁻¹. HRMS (ESI). Calcd for C₂₀H₂₃N₂O (M+H⁺): 307.1805. Found: 307.1804.

9ca. Mp 163.4-163.8 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.46-7.42 (m, 1H), 7.38-7.30 (m, 10H), 7.28-7.23 (m, 2H), 3.59-3.52 (m, 4H), 0.88 (s, 9H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 174.4, 160.7, 141.7, 140.0, 131.9, 128.8, 128.7, 128.4, 127.8, 126.8, 55.4, 47.4, 39.4, 26.7. IR (KBr): 2926, 1655, 700 cm⁻¹. Anal. Calcd for C₂₆H₂₆N₂O: C, 81.64; H, 6.85; N, 7.32. Found: C, 81.24; H, 6.88; N, 7.18.

8da. Mp 126.2-126.8 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.67 (dd, *J* = 6.6, 2.4 Hz, 1H), 7.42-7.36 (m, 5H), 7.34-7.28 (m, 3H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.15 (t, *J* = 7.4 Hz, 2H), 6.89 (d, *J* = 7.4 Hz, 2H), 3.95 (t, *J* = 8.3 Hz, 2H), 3.64 (t, *J* = 8.3 Hz, 2H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 167.5, 161.1, 140.6, 140.0, 134.4, 131.1, 130.9, 130.0, 129.5, 129.4, 128.6, 128.2, 127.9, 127.6, 127.4, 127.3, 54.2, 50.0. IR (KBr): 3058, 1680, 1365, 702 cm⁻¹. Anal. Calcd for C₂₂H₁₈N₂O: C, 80.96; H, 5.56; N, 8.58. Found: C, 80.92; H, 5.93; N, 8.30.

9da. Mp 162.8-163.4 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.50-7.40 (m, 5H), 7.40-7.29 (m, 9H), 7.18 (t, *J* = 7.5 Hz, 2H), 6.83 (d, *J* = 7.5 Hz, 2H), 3.53 (t, *J* = 8.4 Hz, 2H), 3.33 (t, *J* = 8.4 Hz, 2H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 166.4, 158.7, 141.3, 140.6, 134.8, 130.8, 130.0, 128.7, 127.8, 127.7, 127.1, 126.7, 54.5, 49.3. IR (KBr): 2923, 1663, 1637, 1364, 763, 701 cm⁻¹. Anal. Calcd for C₂₈H₂₂N₂O: C, 83.56; H, 5.51; N, 6.96. Found: C, 83.54; H, 5.65; N, 6.92.

8bc. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.51 (d, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 1H) 7.43-7.36 (m, 2H), 7.31-7.27 (m, 1H), 7.23 (d, *J* = 7.4 Hz, 1H), 6.91 (t, *J* = 7.4 Hz, 1H), 6.92-6.90 (m, 1H), 4.00-3.69 (m, 3H), 3.69 (s, 3H), 3.69-3.36 (m, 1H), 1.71 (s, 3H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 155.7, 137.5, 131.9, 131.8, 131.7, 131.6, 131.2, 131.0, 129.4, 129.1, 128.4, 128.3, 128.2, 127.0, 120.0, 110.4, 55.8, 52.5, 47.2, 23.6. IR (neat): 2960, 1672, 1277 cm⁻¹. HRMS (ESI). Calcd for C₁₈H₁₉N₂O₂ (M+H⁺): 295.1441. Found: 295.1440.

8ce. Mp 82.4-83.8 °C. ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) 7.60-7.57 (m, 1H), 7.43-7.37 (m, 2H), 7.23-7.13 (m, 5H), 3.95-3.81 (m, 2H), 3.73-3.58 (m, 2H), 2.20 (s, 3H), 0.90 (s, 9H); ¹³C-NMR (CDCl₃, 125 MHz): δ (ppm) 175.1, 163.2, 140.1, 138.4, 136.1, 133.7, 130.3, 129.9, 129.7, 128.5, 128.3, 127.3, 127.2, 125.0, 55.0, 47.9, 39.2, 26.9, 20.3. IR (KBr): 2971, 1656, 1341, 769 cm⁻¹. Anal. Calcd for C₂₁H₂₄N₂O: C, 78.71; H, 7.55; N, 8.74. Found: C, 78.40; H, 7.53; N, 8.60.

10.⁴ Mp 187.6-188.4 °C (lit.⁴ 184 °C). ¹H-NMR (CDCl₃, 400 MHz): δ (ppm) 7.52 (t, *J* = 7.5 Hz, 1H), 7.43-7.35 (m, 12H); ¹³C-NMR (CDCl₃, 100 MHz): δ (ppm) 173.5, 140.3, 140.2, 129.6, 129.0, 128.4, 128.3, 127.6, 119.6. IR (KBr): 2923, 1698, 1299 cm⁻¹.

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Selected ^1H and ^{13}C NMR

3a

3ea

3dg

6da