3-Acylindoles via Palladium-Catalyzed Regioselective Arylation of Electron-Rich Olefins with Indoles

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1. General Information

Flash chromatography was performed with freshly distilled solvents. \(^1\)H NMR (400 MHz) and \(^{13}\)C NMR (100 MHz) spectra were recorded using CDCl\(_3\) as solvent. Chemical shifts (\(\delta\)) are reported in ppm, using TMS as an internal standard. Data are presented as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). The amount of water was determined with an 831 KF Coulometer (Metrohm, Switzerland).

Solvents were purified by using the following Method: DMF and DMSO were dried over CaH\(_2\) for 24 h and distilled under reduced pressure. Toluene and dioxane were dried over sodium for 4 h, and distilled under N\(_2\) atmosphere. Cu(OAc)\(_2\) was refluxed in acetic anhydride for 5 h before use.

2. The effect of water

2.1 The determination of water amount

The actual amount of water in the reaction was determined in a blank experiment via the following procedure. \(N\)-Methyl indole (0.5 mmol) and DMSO : DMF (1:1, 8.0 mL) were added into an oven-dried 25 mL pressure tube (with a teflon cap). The tube was evacuated and refilled with N\(_2\), which was repeated 3 times. After addition of 1 mmol of \(n\)-Butyl vinyl ether under pressure of N\(_2\) through syringe, the sealed tube was placed in a 70 °C oil bath and stirred 12 h. After completion, the reaction mixture was cooled down to room temperature. The amount of water in the solution was determined with an 831 KF Coulometer and 12.6 mg water (0.7 mmol) was found. The amount of added water in each reaction is shown in the text.

2.2 The effect of water on the reaction

\(N\)-Methyl indole (0.5 mmol), Pd(OTFA)\(_2\) (0.05 mmol), anhydrous Cu(OAc)\(_2\) (1.5 mmol) and dried DMSO : DMF (1:1, 8.0 mL) and water (see Table S1) were added into an oven-dried 25 mL pressure tube (with a teflon cap). The tube was evacuated and refilled with N\(_2\), which was repeated 3 times. After addition of \(n\)-butyl vinyl ether under a positive pressure of N\(_2\) through syringe, the sealed tube was placed in a 70 °C oil bath and stirred 12 h. After completion, the reaction mixture was cooled down to room temperature and quenched by water and extracted with ethyl acetate. The combined organic layer was washed with brine, and dried over MgSO\(_4\). After the evaporation, the residue was purified with silica gel chromatography with petroleum ether/ethyl acetate as eluent to afford the products.
Table S1. The effect of water

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*Reactions conditions: N-methyl indole (0.5 mmol), olefin (1 mmol), Pd(OTFA)$_2$ (10 mol%), oxidant (1.5 mmol), H$_2$O (0-6 equivalent), solvent (8 mL), 70 °C, 12h.*

3. Procedures for synthesis of 3-acylindoles

3.1 General method for the synthesis of 3-acylindole

N-Methyl indole (0.5 mmol), Pd(OTFA)$_2$ (0.05 mmol), anhydrous Cu(OAc)$_2$ (1.5 mmol), DMSO:DMF (1:1, 8.0 mL) and water (see Analytic data for products below) were added into an oven-dried 10 mL pressure tube (with a teflon cap). The tube was evacuated and refilled with N$_2$, which was repeated 3 times. After addition of 1 mmol of n-butyl vinyl ether under pressure of N$_2$ through syringe, the sealed tube was placed in a 70 °C oil bath and stirred 12 h. After completion, the reaction mixture was cooled down to room temperature, quenched by water and extracted with ethyl acetate (25 x 3 mL). The combined organic layer was washed with brine (45 x 3 mL), and dried over MgSO$_4$. After the evaporation, the residue was purified with silica gel chromatography with petroleum ether/ethyl acetate as eluent to afford the products.

3.2 Alkylation of indole (1a) with olefin (2a) in the presence of water

N-Methyl indole (0.5 mmol), Pd(OTFA)$_2$ (0.05 mmol), anhydrous Cu(OAc)$_2$ (1.5
mmol), H₂O (90 mg, 10 eq) and DMSO:DMF(1:1, 8.0 mL) were added into an oven-dried 10 mL pressure tube (with a teflon cap). The tube was evacuated and refilled with N₂, which was repeated 3 times. After addition of 1 mmol of n-butyl vinyl ether under pressure of N₂ through syringe, the sealed tube was placed in a 70 °C oil bath and stirred 12 h. After completion, the reaction mixture was cooled down to room temperature, quenched by water and extracted with ethyl acetate (25 x 3 mL). The combined organic layer was washed with brine (45 x 3 mL) and dried over MgSO₄. After the evaporation, the residue was purified with silica gel chromatography with petroleum ether/ethyl acetate as eluent to afford alkylation product (3a) with 80% yield and 3,3-biindole (4a) with 15% yield.¹

4. Analytic data for products

3,3’-(Ethane-1,1-diyl)bis(1-methyl-1H-indole) (3a)¹

![3,3’-(Ethane-1,1-diyl)bis(1-methyl-1H-indole) (3a)]

Colorless oil. Rₜ = 0.3 (petroleum ether/ethyl acetate 15:1); ¹HNMR (400 MHz, CDCl₃) δ 7.59-7.57 (d, 2H, J = 7.9 Hz), 7.29-7.27 (d, 2H, J = 8.2 Hz), 7.21-7.17 (t, 2H, J = 7.3 Hz), 7.05-7.01 (t, 2H, J = 7.2 Hz), 6.78 (s, 2H), 4.69-4.64 (m, 1H), 3.70 (s, 6H), 1.79-1.78 (d, 3H, J = 6.0 Hz); ¹³CNMR (100 MHz, CDCl₃) δ 137.4, 127.4, 126.0, 121.4, 120.5, 119.9, 118.5, 109.1, 32.6, 28.1, 22.3. IR (KBr): 3051, 2361, 1648, 1474, 1370, 1235, 1082, 738 cm⁻¹.

1-(1-Methyl-1H-indol-3-yl) ethanone (5a)²

![1-(1-Methyl-1H-indol-3-yl) ethanone (5a)]

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 106-107 °C; Rₜ = 0.3 (petroleum ether/ethyl acetate 3:1); ¹HNMR (400 MHz, CDCl₃) δ 8.37-8.34 (m, 1H), 7.66 (s, 1H), 7.31-7.29 (m, 3H), 3.81 (s, 3H), 2.5 (s, 3H); ¹³CNMR (100 MHz, CDCl₃) δ 192.9, 137.4, 135.9, 126.2, 123.2, 122.4, 122.1, 116.7, 109.8, 33.4, 27.5. IR (KBr): 3421, 2362, 1648, 1561, 1411, 1260, 1076, 792 cm⁻¹; HRMS (ESI): calc. for (M + Na⁺) 196.0738; found: 196.0730.
1-(1,6-Dimethyl-1H-indol-3-yl) ethanone (5b)

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 95-97 °C; Rf = 0.3 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) $\delta$ 8.23-8.21 (m, 1H), 7.62 (s, 1H), 7.14-7.12 (m, 2H), 3.79 (s, 3H), 2.5 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$) $\delta$ 192.8, 137.9, 135.2, 133.3, 124.2, 124.0, 122.2, 117.0, 109.5, 33.4, 27.5, 21.8. IR (KBr): 3056, 1636, 1524, 1468, 1369, 1226, 1102, 929 cm$^{-1}$; HRMS (ESI): calc. for (M + Na$^+$) 210.0889; found: 210.0879.

1-(1,7-Dimethyl-1H-indol-3-yl)ethanone (5c)

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 170-171 °C; Rf = 0.3 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) $\delta$ 8.25-8.24 (d, 1H, $J = 7.6$ Hz), 7.53 (s, 1H), 7.15-7.11 (t, 1H, $J = 7.6$ Hz), 6.98-6.97 (d, 1H, $J = 7.6$ Hz), 4.00 (s, 1H), 2.72 (s, 1H), 2.47 (s, 1H); $^{13}$CNMR (100 MHz, CDCl$_3$) $\delta$ 192.7, 137.4, 136.2, 127.4, 126.1, 122.7, 121.4, 120.6, 116.4, 37.6, 27.5, 19.5; IR (KBr):3053, 1622, 1533, 1454, 1311, 1245, 1105, 950 cm$^{-1}$; HRMS (ESI): calc. for (M + Na$^+$) 210.0889; found: 210.0881.

1-(6-Fluoro-1-methyl-1H-indol-3-yl)ethanone (5d)$^{[3]}$

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 90-92 °C; Rf = 0.32 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) $\delta$ 8.31-8.28 (m, 1H), 7.64 (s, 1H), 7.05-6.95 (m, 2H), 3.76 (s, 3H), 2.5 (s, 3H).
2.47 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$) δ 192.7, 160.4 (d, $J = 239$ Hz), 137.6, 135.9, 123.7, 122.6, 117.1, 110.5 (d, $J = 23.6$ Hz), 96.2 (d, $J = 26.2$ Hz), 33.5, 27.3.
IR (KBr): 3047, 1639, 1521, 1436, 1372, 1233, 1083, 945 cm$^{-1}$; HRMS (ESI): calc. for 214.0638 (M + Na$^+$); found: 214.0631.

1-(6-Chloro-1-methyl-1H-indol-3-yl)ethanone (5e)$^{[3]}$

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 133-135 °C; R$_f$ = 0.32 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) δ 8.26-8.24 (d, 1H, $J = 8.4$Hz), 7.59 (s, 1H), 7.26-7.21 (m, 2H), 3.74 (s, 3H), 2.46 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$) δ 192.6, 137.9, 129.4, 124.7, 123.5, 123.1, 116.9, 109.7, 33.5, 27.4; IR (KBr): 2955, 1667, 1610, 1481, 1306, 1097, 934 cm$^{-1}$; HRMS (ESI): calc. for 230.0343 (M + Na$^+$); found: 230.0344.

1-(1-Benzyl-1H-indol-3-yl)ethanone (5f)$^{[4]}$

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 97-100 °C; R$_f$ = 0.3 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) δ 8.17 (s, 1H), 7.65 (s, 1H), 7.25-7.13 (m, 2H), 7.19-7.13 (m, 2H), 3.81 (s, 3H), 2.50 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$) δ 192.8, 135.8, 132.1 126.5, 124.8, 122.2, 116.5, 109.2, 33.4, 27.5, 21.5; IR (KBr): 3445, 1650, 1596, 1261, 1151, 1006, 873, 828 cm$^{-1}$; HRMS (ESI): calc. for 210.0889 (M + Na$^+$); found: 210.0884.

1-(5-Methoxy-1-methyl-1H-indol-3-yl)ethanone (5g)$^{[4]}$

Electronic Supplementary Material (ESI) for RSC Advances
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According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 146-148 °C ; Rf = 0.25  (petroleum ether/ethyl acetate 3:1) ; $^1$HNMR (400 MHz, CDCl$_3$) δ 7.90 (s, 1H), 7.64 (s, 1H), 7.23-7.20 (m, 1H), 3.81 (s, 3H), 2.49 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$), 192.8 156.5, 135.7, 132.5, 127.1, 116.7, 113.8, 110.4, 103.9, 55.8, 33.6, 27.3; IR (KBr): 2943, 1655, 1456, 1369, 1264, 1107, 950 cm$^{-1}$; HRMS (ESI): calc. for 226.0838 (M + Na$^+$); found: 226.0834.

1-(5-(Benzyloxy)-1-methyl-1H-indol-3-yl)ethanone (5h)

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 106-107 °C; Rf = 0.25 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) δ 8.01-7.99 (m, 1H), 7.58-7.57 (m, 1H), 7.50-7.48 (m, 2H), 7.40-7.36 (m, 2H), 7.33-29 (m, 1H), 7.20-7.17 (m, 1H), 7.03-6.99 (m, 1H), 5.15 (s, 2H), 3.74 (s, 3H), 2.46 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$) δ 192.7, 155.6, 137.4, 135.8, 132.8, 132.1, 128.5, 127.7, 127.0, 116.6, 116.2, 114.4, 110.4, 105.4, 70.7, 33.6, 27.4, One carbon is not visible due to overlapping peaks; IR (KBr): 2938, 1655, 1461, 1369, 1257, 1107, 955, 791 cm$^{-1}$; HRMS (ESI): calc. for (M + Na$^+$) 302.1151; found: 302.1154.

1-(7-Methoxy-1-methyl-1H-indol-3-yl)ethanone (5i)

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 139-140 °C; Rf = 0.25 (petroleum ether/ethyl acetate 3:1); $^1$HNMR (400 MHz, CDCl$_3$) δ 7.95-7.93 (d, 1H, J = 8 Hz), 7.52(s, 1H), 7.17-7.13 (m, 1H), 6.96-6.77 (d, 1H, J = 8Hz), 4.06 (s, 3H), 3.91 (s, 3H), 2.48 (s, 3H); $^{13}$CNMR (100 MHz, CDCl$_3$) δ 192.8, 147.7, 136.5, 128.6, 127.1, 123.2, 116.8, 115.0, 104.1, 55.4, 37.5, 27.6; IR (KBr): 2934, 1653, 1451, 1365, 1259, 1097, 948, 781 cm$^{-1}$; HRMS (ESI): calc. for 226.0838 (M + Na$^+$); found: 226.0831.
1-(1-Benzyl-1H-indol-3-yl)ethanone (5j)[3]

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 109-111 °C ; Rf = 0.35 (petroleum ether/ethyl acetate 3:1); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.40-8.38 (m, 1H), 7.71 (s, 1H), 7.34-7.21 (m, 6H), 7.14-7.12 (m, 2H), 5.3 (s, 2H), 2.48 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 193.0, 137.1, 135.9, 135.0, 129.1, 128.2, 127.1, 127.0, 126.5, 123.5, 122.7, 117.5, 110.2, 50.7, 27.7, Two quaternary carbons are not visible due to overlapping peaks.; IR (KBr): 3086, 2369, 2337, 1634, 1517, 1334, 1137, 1079 cm\(^{-1}\); HRMS (ESI): calc. for (M + Na\(^+\)) 272.1045; found: 272.1044

1-(1-Benzyl-5-methoxy-1H-indol-3-yl)ethanone (5k)[5]

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 121-122 °C; Rf = 0.28 (petroleum ether/ethyl acetate 3:1); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.92 (m, 1H), 7.91 (m, 1H), 7.69-7.28 (m, 3H), 7.15-7.12 (m, 3H), 6.89-6.87 (m, 1H), 5.29 (s, 2H), 3.88 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)), \(\delta\) 192.9, 156.5, 135.8, 135.0, 132.0, 129.0, 128.2, 127.3, 126.9, 117.2, 114.0, 110.9, 103.9, 55.8, 50.9, 27.4, Two quaternary carbons are not visible due to overlapping peaks; IR (KBr): 2978, 1653, 1526, 1456, 1369, 1266, 1105, 959 cm\(^{-1}\); HRMS (ESI): calc. for 302.1151 (M + Na\(^+\)); found: 302.1148.

1-(1-Benzyl-6-fluoro-1H-indol-3-yl)ethanone (5l)[3]

According to the general method, water (27 mg) was added to the reaction system.
Yellow solid. mp = 133-134 °C; Rf = 0.32 (petroleum ether/ethyl acetate 3:1);
\(^1\)HNMR(400MHz, CDCl\(_3\)) \(\delta\) 8.36-8.32 (m, 1H), 7.72 (s, 1H), 7.35-7.34 (m, 3H), 7.16-7.14 (m, 2H), 7.06-7.01 (m, 1H), 6.96-6.94 (m, 3H), 5.29 (s, 2H), 2.49 (s, 3H); \(^{13}\)CNMR (100MHz, CDCl\(_3\)) \(\delta\) 191.8, 159.2 (\(J_{C-F} = 239.4\) Hz), 136.1, 134.2, 128.1, 127.4, 125.9, 122.8 (\(J_{C-F} = 9.6\) Hz), 121.8, 116.5, 110.2 (\(J_{C-F} = 23.6\) Hz), 95.1 (\(J_{C-F} = 26.2\) Hz), 49.8, 27.4; IR (KBr): 2943, 1655, 1451, 1369, 1257, 1102, 952, 793 cm\(^{-1}\); HRMS (ESI): calc. for 290.0951 (M + Na\(^+\)); found: 290.0948.

1-(1-Benzyl-7-methyl-1H-indol-3-yl)ethanone (5m)

According to the general method, water (27 mg) was added to the reaction system. Yellow solid. mp = 133-135 °C; Rf = 0.32 (petroleum ether/ethyl acetate 3:1); \(^1\)HNMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.34-8.32 (d, 1H, \(J = 8\) Hz), 7.65 (s, 1H), 7.29-7.25 (m, 3H), 7.23 (m, 1H), 6.96-6.92 (m, 3H), 5.56 (s, 2H), 2.47 (s, 6H); \(^{13}\)CNMR (100 MHz, CDCl\(_3\)), \(\delta\) 192.9, 137.9, 137.1, 135.9, 129.1, 127.9, 127.5, 126.5, 126.6, 122.9, 121.3, 120.7, 117.2, 52.9, 27.6, 19.4. Two quaternary carbons are not visible due to overlapping peaks; IR (KBr): 2934, 1655, 1538, 1458, 1259, 1107, 952, 784 cm\(^{-1}\); HRMS(ESI): calc. for 286.1206 (M + Na\(^+\)); found: 286.1199.

1-(1-Benzyl-5-methyl-1H-indol-3-yl)ethanone (5n)

According to the general method, water (27 mg) and NaOAc (0.05 mmol) were added to the reaction system. Yellow solid. mp = 140-142 °C, Rf = 0.32 (petroleum ether/ethyl acetate 3:1); \(^1\)HNMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.69 (s, 1H), 7.33 (s, 1H), 7.32-7.28 (m, 3H), 7.17-7.05 (m, 4H), 5.29 (s, 2H), 2.49 (s, 3H), 2.46 (s, 3H); \(^{13}\)CNMR (100 MHz, CDCl\(_3\)) \(\delta\) 193.0, 135.9, 135.5, 135.0, 132.0, 129.0, 126.9, 126.7, 125.0, 122.4, 117.2, 109.8, 50.8, 21.6, 21.4. Two quaternary carbons are not visible due to overlapping peaks; IR (KBr): 2934, 1646, 1528, 1461, 1367, 1261, 1102, 952 cm\(^{-1}\);
HRMS (ESI): calc. for 286.1206 (M + Na⁺); found: 286.1202.

1-(1-Benzyl-5-nitro-1H-indol-3-yl)ethanone (5o)

According to the general method, water (27 mg) was added to the reaction system. Yellow solid, mp = 184-186 °C, Rf = 0.35 (petroleum ether/ethyl acetate 3:2); ¹H NMR (400 MHz, CDCl₃) δ 9.29 (s, 1H), 8.15-8.12 (m, 1H), 7.86 (s, 1H), 7.39-7.33 (m, 4H), 7.17-7.15 (d, 2H, J = 5.6 Hz), 5.41 (s, 2H), 2.53 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 192.2, 144.0, 139.7, 137.1, 134.7, 129.3, 128.7, 126.9, 125.8, 119.7, 119.1, 119.0, 110.3, 51.2, 27.7 Two quaternary carbons are not visible due to overlapping peaks; IR (KBr): 3112, 2360, 2344, 1632, 1528, 1369, 1236, 1095 cm⁻¹; HRMS (ESI): calc. for (M + Na⁺) 317.0896; found: 317.0897.

5. References


6. Copies of the $^1$H NMR, $^{13}$C NMR and HRMS spectra of products
## Display Report

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- **Method:** pol_low_1000_m
- **Sample Name:** ilyang
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**Bruker Compass DataAnalysis 4.0**

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Display Report

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