Reaction of 3-(2-nitrophenyl)-1-arylprop-2-en-1-ones with triethylphosphite in microwave revisited: One-pot synthesis of 2-arylindoles and 2-arylquinolines

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EXPERIMENTAL

General

All the commercially available reagents were purchased from Aldrich and were used without further purification. The reactions were performed with a Discover™ single mode cavity MW synthesizer (CEM Corporation). Melting points (°C) were measured in open glass capillaries using Perfit melting point apparatus and are uncorrected. The progress of the reaction was monitored by thin layer chromatography (TLC) using silica gel pre-coated aluminium sheets (60 F254, Merck). Visualization of spots was effected by exposure to ultraviolet light (UV) at 254 nm, iodine vapours, 2% 2,4-dinitrophenylhydrazine in methanol containing few drops of H₂SO₄ and draggendroff reagent. Column chromatography was performed on silica gel (60-120 mesh). IR spectra (v, cm⁻¹) were recorded on Perkin-Elmer FTIR spectrophotometer using KBr discs. ¹H NMR and ¹³C NMR spectra in CDCl₃ as solvents were recorded on Burker AC-400 spectrometer operating at 400 MHz for ¹H and 100 MHz for ¹³C, with tetramethylsilane (TMS) as internal standard. Electron impact mass spectra (EIMS) were recorded on Micro Mass VG-7070 H mass spectrometer at 70 ev. Elemental analysis were performed on Leco CHNS-932 analyzer.

General procedure

A solution of 3-(2-nitrophenyl)-1-arylprop-2-en-1-ones (1 mmol) in P(OEt)₃ (6 mmol) in a capped vial was exposed to MW irradiation at 110 °C (80 W). After the completion of the reaction (TLC), the reaction mixture was cooled to room temperature, transferred to a 10 mL round-bottomed flask and concentrated under vacuum at 80 °C in a rotary evaporator. The residue so obtained upon column chromatography yield pure products **2(a-i; 57-62% yield)** and **3(a-i; 27-30% yield)**.

Spectral data of the synthesized compounds:

(1*H*-indol-2-yl)(phenyl)methanone (2a)^[1]: Yellow solid, yield: 62%, m.pt. 145-147 °C (lit. 150-151 °C), ¹H NMR (400 MHz, CDCl₃): δ 9.65 (br s, 1H, NH), 8.04 (d, *J* = 7.8 Hz, 2H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.66 (t, *J* = 7.0 Hz, 1H), 7.60 – 7.52 (m, 3H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.20 (t, *J* = 7.4 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 187.3, 138.0, 137.6, 134.3, 132.4, 129.2, 128.5, 127.7, 126.5, 123.2, 121.0, 112.9, 112.2. IR (KBr, ν_{max} cm⁻¹): 3315, 1618, 1577, 1512, 1328, 1261, 1132, 734. MS(ESI) (*m/z*) : 222. Anal. calcd. for C₁₅H₁₁NO : C, 81.43; H, 5.01; N, 6.33%. Found: C, 81.52; H, 5.02; N, 6.31%.

(1*H*-indol-2-yl)(*p*-tolyl)methanone (2b)^[1]: Light brown solid, yield: 61%, m.pt. 182-184 °C (lit. 189-190 °C), ¹H NMR (400 MHz, CDCl₃): δ 9.78 (br s, 1H, NH), 7.97 (d, *J* = 7.9 Hz, 2H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.40 (dd, *J* = 14.2, 7.4 Hz, 3H), 7.19 (d, *J* = 10.8 Hz, 2H), 2.50 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 187.0, 143.1, 137.5, 135.3, 134.5, 129.4, 129.2, 127.7, 126.3, 123.1, 120.9, 112.5, 112.3, 21.7. IR (KBr, ν_{max} cm⁻¹): 3307, 1616, 1510, 1261, 1170, 1014, 825, 754. MS(ESI) (*m/z*) : 236. Anal. calcd. for C₁₆H₁₃NO : C, 81.68; H, 5.57; N, 5.95. Found: C, 81.54; H, 5.56; N, 5.97.

(1*H*-indol-2-yl)(4-methoxyphenyl)methanone (2c)^[1]: White solid, yield: 62%, m.pt. 189-190 °C (lit. 190-191 °C), ¹H NMR (400 MHz, CDCl₃): δ 9.58 (br s, 1H, NH), 8.07 (d, *J* = 8.5

Hz, 2H), 7.75 (d, $J = 8.1$ Hz, 1H), 7.52 (d, $J = 8.3$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 1H), 7.23 – 7.16 (m, 2H), 7.06 (d, $J = 8.5$ Hz, 2H), 3.94 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 185.9, 163.2, 137.3, 134.5, 131.5, 130.6, 127.7, 126.2, 123.0, 120.9, 113.8, 112.1, 111.9, 55.5. IR (KBr, ν_{max} cm^{-1}): 3296, 1610, 1510, 1309, 1257, 1168, 1026, 833, 758. MS(ESI) (m/z) : 252. Anal. calcd. for $\text{C}_{16}\text{H}_{13}\text{NO}_2$: C, 76.48; H, 5.21; N, 5.57%. Found: C, 76.32; H, 5.20; N, 5.55%.

(1*H*-indol-2-yl)(3-methoxyphenyl)methanone (2d)^[1]: Brown solid, yield: 58%, m.pt. 125-126 °C (lit. 128-129 °C), ^1H NMR (400 MHz, CDCl_3): δ 9.93 (br s, 1H, NH), 7.76 (d, $J = 8.0$ Hz, 1H), 7.66 (d, $J = 7.6$ Hz, 1H), 7.58 – 7.54 (m, 2H), 7.48 (t, $J = 7.9$ Hz, 1H), 7.41 (t, $J = 7.5$ Hz, 1H), 7.25 – 7.18 (m, 3H), 3.93 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 187.1, 159.6, 139.3, 137.8, 134.3, 129.5, 127.7, 126.5, 123.2, 121.9, 121.0, 118.6, 113.9, 113.0, 112.3, 55.5. IR (KBr, ν_{max} cm^{-1}): 3298, 1614, 1508, 1312, 1265, 1159, 1020, 839, 752. MS(ESI) (m/z) : 252. Anal. calcd. for $\text{C}_{16}\text{H}_{13}\text{NO}_2$: C, 76.48; H, 5.21; N, 5.57%. Found: C, 76.32; H, 5.22; N, 5.56% .

(4-Chlorophenyl)(1*H*-indol-2-yl)methanone (2e)^[1]: Light brown solid, yield: 58%, m.pt. 194-196 °C (lit. 196-198 °C), ^1H NMR (400 MHz, CDCl_3): δ 9.40 (br s, 1H, NH), 7.97 (d, $J = 8.5$ Hz, 2H), 7.75 (d, $J = 8.1$ Hz, 1H), 7.53 (dd, $J = 12.9, 8.5$ Hz, 3H), 7.44 – 7.40 (m, 1H), 7.23 – 7.17 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 185.8, 138.8, 137.6, 136.2, 134.0, 130.6, 128.8, 127.6, 126.8, 123.3, 121.2, 112.8, 112.2. IR (KBr, ν_{max} cm^{-1}): 3307, 1624, 1504, 1342, 1259, 1089, 1014, 831, 754. MS(ESI) (m/z) : 256, 258. Anal. calcd. for $\text{C}_{15}\text{H}_{10}\text{NOCl}$: C, 70.46; H, 3.94; N, 5.48%. Found: C, 70.59; H, 3.93; N, 5.50%.

(4-Bromophenyl)(1*H*-indol-2-yl)methanone (2f): Light yellow solid, yield: 60%, m.pt. 184-186 °C, ^1H NMR (400 MHz, CDCl_3): δ 9.37 (br s, 1H, NH), 7.90 (d, $J = 8.4$ Hz, 2H), 7.73 (dd, $J = 16.8, 8.3$ Hz, 3H), 7.51 (d, $J = 8.3$ Hz, 1H), 7.42 (t, $J = 7.6$ Hz, 1H), 7.20 (dd, J

= 16.0, 8.4 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 185.9, 137.6, 136.7, 133.9, 131.8, 130.72, 127.7, 127.3, 126.8, 123.3, 121.2, 112.8, 112.1. IR (KBr, ν_{max} cm^{-1}): 3309, 1622, 1512, 1327, 1263, 1134, 1010, 829, 752. MS(ESI) (m/z) : 300, 302. Anal. calcd. for $\text{C}_{15}\text{H}_{10}\text{NOBr}$: C, 60.02; H, 3.36; N, 4.67%. Found: C, 60.13; H, 3.35; N, 4.66%.

(4-Fluorophenyl)(1*H*-indol-2-yl)methanone (2g) ^[1]: White solid, yield: 60%, m.pt. 183-185 °C (lit. 185-186 °C), ^1H NMR (400 MHz, CDCl_3): δ 9.81 (br s, 1H, NH), 8.08 (dd, J = 8.6, 5.5 Hz, 2H), 7.76 (d, J = 8.1 Hz, 1H), 7.54 (d, J = 8.3 Hz, 1H), 7.42 (t, J = 7.6 Hz, 1H), 7.28 (d, J = 2.9 Hz, 1H), 7.22 (dd, J = 13.9, 5.9 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 185.8, 166.6, 164.1, 137.7, 134.1, 131.7, 127.6, 126.6, 123.2, 121.1, 115.8, 115.6, 112.8, 112.3. IR (KBr, ν_{max} cm^{-1}): 3311, 1627, 1508, 1327, 1247, 1143, 1010, 833, 756. MS(ESI) (m/z) : 240. Anal. calcd. for $\text{C}_{15}\text{H}_{10}\text{NOF}$: C, 75.30; H, 4.21; N, 5.85%. Found: C, 75.42; H, 4.19; N, 5.87%.

(1*H*-indol-2-yl)(3,4-dimethoxyphenyl)methanone (2h) ^[2]: White solid, yield: 57%, m.pt. 129-131°C, (lit. 126-127 °C), ^1H NMR (400 MHz, CDCl_3): δ 10.08 (br s, 1H, NH), 7.70-7.67 (t, J = 8 Hz, 1H), 7.53-7.46 (m, 3 H), 7.13-7.10 (d, J = 12 Hz, 2H), 6.82-6.80 (d, J = 8 Hz, 2H), 3.92 (s, 3H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 185.8, 152.8, 148.8, 137.5, 134.4, 130.3, 127.6, 126.0, 124.2, 123.9, 122.9, 120.8, 119.9, 111.8, 109.8, 56.0, 55.9. IR (KBr, ν_{max} cm^{-1}): 3299, 1612, 1509, 1305, 1254, 1173, 1002, 839, 790, 761. MS(ESI) (m/z) : 282. Anal. calcd. for $\text{C}_{17}\text{H}_{15}\text{NO}_3$: C, 72.58; H, 5.37; N, 4.98%. Found: C, 72.67; H, 5.38; N, 4.97%.

(Furan-2-yl)(1*H*-indol-2-yl)methanone (2i) ^[1]: Brown solid, yield: 59%, m.pt. 134-135 °C (lit. 130-132 °C), ^1H NMR (400 MHz, CDCl_3): δ 9.50 (br s, 1H, NH), 7.77 (dd, J = 16.4, 6.7 Hz, 3H), 7.54 – 7.48 (m, 2H), 7.40 (t, J = 7.6 Hz, 1H), 7.20 (t, J = 7.2 Hz, 1H), 6.67 (dd, J = 3.4, 1.6 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 172.6, 152.7, 146.5, 137.2, 133.4, 128.0,

126.5, 123.3, 121.0, 118.6, 112.5, 112.1, 111.5. IR (KBr, $\nu_{\max}$ cm^{-1}): 3301, 1610, 1507, 1344, 1275, 1129, 1010, 837, 743. MS(ESI) (m/z) : 212. Anal. calcd. for $\text{C}_{13}\text{H}_9\text{NO}_2$: C, 73.92; H, 4.29; N, 6.63%. Found: C, 73.80; H, 4.27; N, 6.62%.

2-Phenylquinoline (3a)^[3]: Oil, yield: 28%, ^1H NMR (400 MHz, CDCl_3): δ 8.44 – 8.08 (m, 4H), 7.89 (dd, $J = 18.8, 8.3$ Hz, 2H), 7.77 (t, $J = 7.6$ Hz, 1H), 7.59 – 7.51 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3): δ 157.4, 148.2, 139.7, 136.8, 129.7, 129.3, 128.8, 128.3, 127.6, 127.4, 127.2, 126.3, 119.0. IR (KBr, $\nu_{\max}$ cm^{-1}): 3053, 2958, 1710, 1596, 1551, 1446, 1282, 1074, 828, 765. MS(ESI) (m/z) : 206. Anal. calcd. for $\text{C}_{15}\text{H}_{11}\text{N}$: C, 87.77; H, 5.40; N, 6.82%. Found: C, 87.65; H, 5.38; N, 6.81.

2-p-Tolylquinoline (3b)^[3]: Light brown solid, yield: 28%, m.pt. 82-84 °C (lit. 80-82 °C), ^1H NMR (400 MHz, CDCl_3): δ 8.21 (d, $J = 8.5$ Hz, 2H), 8.11 (d, $J = 8.1$ Hz, 2H), 7.86 (dd, $J = 17.5, 8.3$ Hz, 2H), 7.75 (t, $J = 7.7$ Hz, 1H), 7.54 (t, $J = 7.5$ Hz, 1H), 7.37 (d, $J = 8.0$ Hz, 2H), 2.47 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 157.3, 148.3, 139.4, 136.8, 136.7, 129.6, 129.6, 127.4, 127.1, 126.1, 118.9, 21.3. IR (KBr, $\nu_{\max}$ cm^{-1}): 3054, 2934, 1702, 1595, 1498, 1431, 1286, 1039, 814, 787. MS(ESI) (m/z) : 220. Anal. calcd. for $\text{C}_{16}\text{H}_{13}\text{N}$: C, 87.64; H, 5.98; N, 6.39%. Found: C, 87.75; H, 5.97; N, 6.41%.

2-(4-Methoxyphenyl)quinoline (3c)^[3]: Light brown solid, yield: 30%, m.pt. 121-123 °C (lit.122-124 °C), ^1H NMR (400 MHz, CDCl_3): δ 8.18 (t, $J = 9.3$ Hz, 4H), 7.89 – 7.79 (m, 2H), 7.74 (t, $J = 7.7$ Hz, 1H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.08 (d, $J = 8.5$ Hz, 2H), 3.91 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 160.8, 156.9, 148.2, 136.6, 132.2, 129.6, 129.5, 128.9, 127.4, 126.9, 125.9, 118.6, 114.2, 55.4. IR (KBr, $\nu_{\max}$ cm^{-1}): 3058, 2956, 1707, 1602, 1502, 1462, 1251, 1028, 821, 754. MS(ESI) (m/z) : 236. Anal. calcd. for $\text{C}_{16}\text{H}_{13}\text{NO}$: C, 81.68; H, 5.57; N, 5.95%. Found: C, 81.52; H, 5.55; N, 5.94%.

2-(3-Methoxyphenyl)quinoline (3d) ^[3]: Oil, yield: 28%, ¹H NMR (400 MHz, CDCl₃): δ 8.23 (dd, *J* = 15.2, 8.6 Hz, 2H), 7.88 (dd, *J* = 14.0, 8.4 Hz, 2H), 7.81 – 7.71 (m, 3H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.9 Hz, 1H), 7.05 (dd, *J* = 8.2, 2.4 Hz, 1H), 3.97 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 160.1, 157.1, 148.2, 141.1, 136.8, 129.8, 129.7, 129.7, 127.3, 126.3, 120.0, 119.1, 115.4, 112.7, 55.4. IR (KBr, ν_{max} cm⁻¹): 3059, 2932, 1712, 1599, 1557, 1467, 1287, 1041, 830, 781. MS(ESI) (*m/z*) : 236. Anal. calcd. for C₁₆H₁₃NO : C, 81.68; H, 5.57; N, 5.95%. Found: C, 81.52; H, 5.59 N, 5.94%.

2-(4-Chlorophenyl)quinoline (3e) ^[3]: Brown solid, yield: 29%, m.pt. 110-112 °C (lit. 112-113 °C), ¹H NMR (400 MHz, CDCl₃): δ 8.26 (d, *J* = 8.6 Hz, 1H), 8.16 (dd, *J* = 13.0, 8.6 Hz, 3H), 7.87 (dd, *J* = 8.2, 3.9 Hz, 2H), 7.77 (t, *J* = 7.1 Hz, 1H), 7.59 – 7.51 (m, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 156.0, 148.2, 138.0, 137.0, 135.5, 130.9, 129.7, 129.0, 128.8, 127.5, 127.2, 126.5, 118.6. IR (KBr, ν_{max} cm⁻¹): 3061, 2923, 1710, 1593, 1559, 1428, 1285, 1094, 815, 753. MS(ESI) (*m/z*) : 240, 242. Anal. calcd. for C₁₅H₁₀NCl : C, 75.16; H, 4.21; N, 5.84%. Found: C, 75.24; H, 4.20; N, 5.82%.

2-(4-Bromophenyl)quinoline (3f) ^[3]: Light brown solid, yield: 27%, m.pt. 120-122 °C (lit. 121-123 °C), ¹H NMR (400 MHz, CDCl₃): δ 8.25 (d, *J* = 8.6 Hz, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 8.08 (d, *J* = 8.2 Hz, 2H), 7.86 (dd, *J* = 8.4, 2.8 Hz, 2H), 7.77 (t, *J* = 7.7 Hz, 1H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.57 (t, *J* = 7.5 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 156.0, 148.2, 138.5, 137.0, 131.9, 129.8, 129.1, 127.5, 127.2, 126.5, 123.9, 118.5. IR (KBr, ν_{max} cm⁻¹): 3058, 2923, 1712, 1596, 1551, 1426, 1221, 1072, 818, 767. MS(ESI) (*m/z*) : 284, 286. Anal. calcd. for C₁₅H₁₀NBr: C, 63.40; H, 3.55; N, 4.93%. Found: C, 63.53; H, 3.56; N, 4.91%.

2-(4-Fluorophenyl)quinoline (3g) ^[3]: Brown solid, yield: 27%, m.pt. 95-96 °C (lit. 91-93°C), ¹H NMR (400 MHz, CDCl₃): δ 8.25 (d, *J* = 8.6 Hz, 1H), 8.19 (dd, *J* = 8.8, 5.5 Hz, 3H), 7.86 (d, *J* = 8.5 Hz, 2H), 7.76 (t, *J* = 7.7 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.24 (t, *J* =

8.7 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 165.0, 162.5, 156.2, 148.2, 136.9, 135.8, 129.5, 127.5, 127.0, 126.3, 118.6, 115.9, 115.6. IR (KBr, ν_{max} cm^{-1}): 3049, 2936, 1707, 1589, 1579, 1434, 1235, 1083, 812, 757. MS(ESI) (m/z) : 224. Anal. calcd. for $\text{C}_{15}\text{H}_{10}\text{NF}$: C, 80.70; H, 4.51; N, 6.27%. Found: C, 80.83; H, 4.50; N, 6.25%.

2-(3,4-Dimethoxyphenyl)quinoline (3h): Yellow oil, yield: 29%, ^1H NMR (400 MHz, CDCl_3): δ 8.23-8.17 (m, 2H), 7.90-7.83 (m, 2H), 7.76-7.68 (m, 2H), 7.55-7.53 (m, 2H), 7.04-7.01 (d, J = 12 Hz, 1H), 4.08 (s, 3H), 3.97 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 161.0, 156.8, 147.9, 136.7, 129.6, 129.4, 127.4, 126.9, 126.0, 123.3, 120.2, 118.6, 111.0, 110.3. IR (KBr, ν_{max} cm^{-1}): 3052, 2942, 1702, 1599, 1512, 1457, 1245, 1035, 830, 775. MS(ESI) (m/z) : 266. Anal. calcd. for $\text{C}_{17}\text{H}_{15}\text{NO}_2$: C, 76.96; H, 5.70; N, 5.28%. Found: C, 77.09; H, 5.69; N, 5.27%.

2-(Furan-2-yl)quinoline (3i) ^[4]: Brown solid, yield: 28%, m.pt. 92-94 °C (lit. 88-90 °C), ^1H NMR (400 MHz, CDCl_3): δ 8.17 (t, J = 9.7 Hz, 2H), 7.82 (dd, J = 15.5, 8.4 Hz, 2H), 7.73 (t, J = 7.1 Hz, 1H), 7.66 (s, 1H), 7.52 (t, J = 7.1 Hz, 1H), 7.25 (d, J = 2.7 Hz, 1H), 6.62 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 153.6, 149.0, 148.0, 144.1, 136.7, 129.9, 129.3, 127.6, 127.1, 126.2, 117.5, 112.2, 110.2. IR (KBr, ν_{max} cm^{-1}): 3059, 2945, 1710, 1613, 1526, 1489, 1242, 1057, 820, 719. MS(ESI) (m/z) : 196. Anal. calcd. for $\text{C}_{13}\text{H}_9\text{NO}$: C, 79.98; H, 4.65; N, 7.17%. Found: C, 80.09; H, 4.63; N, 7.19%.

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(1*H*-indol-2-yl)(phenyl)methanone (2a)

(1*H*-indol-2-yl)(p-tolyl)methanone (2b)

(1*H*-indol-2-yl)(4-methoxyphenyl)methanone (2c)

(1*H*-indol-2-yl)(3-methoxyphenyl)methanone (2d)

(4-Chlorophenyl)(1*H*-indol-2-yl)methanone (2e)

(4-Bromophenyl)(1*H*-indol-2-yl)methanone (2f)

(4-Fluorophenyl)(1*H*-indol-2-yl)methanone (2g)

(1*H*-indol-2-yl)(3,4-dimethoxyphenyl)methanone (2h)

(Furan-2-yl)(1*H*-indol-2-yl)methanone (2i):

2-Phenylquinoline (3a)

2-p-Tolylquinoline (3b)

2-(4-Methoxyphenyl)quinoline (3c)

2-(3-Methoxyphenyl)quinoline (3d)

2-(4-Chlorophenyl)quinoline (3e)

2-(4-Bromophenyl)quinoline (3f)

2-(4-Fluorophenyl)quinoline (3g)

2-(3,4-Dimethoxyphenyl)quinoline (3h)

2-(Furan-2-yl)quinoline (3i)