Supporting Information

Efficient Metal-Catalysed Direct Benzylation and Allylic Alkylation of 2,4-Pentanediones

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General: Unless otherwise noted, all materials were obtained from commercial suppliers and were used without further purification. Solvents for extraction and chromatography were technical grade and distilled prior to use. For thin-layer chromatography (TLC), silica gel coated aluminium plates (Merck, silica gel 60 F₂₅₄) were used and chromatograms were visualised by irradiation with UV light at 254 nm. Column chromatography was performed using Merck silica gel 60 (particle size 0.040-0.063 mm). Solvents mixtures are understood as volume/volume.

¹H-NMR and ¹³C-NMR were recorded on a Bruker AM 250 spectrometer in CDCl₃. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated (bs (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet)); coupling constants (*J*) are in Hertz (Hz). MALDI-TOF mass spectra were obtained on a VG-TofSpec spectrometer. ESI mass spectra were obtained on a VG-Platform II (Fisons instruments). Infrared (IR) spectra were recorded on a Bruker Tensor 27 FT-IR spectrometer and are reported in terms of frequency of absorption (cm⁻¹).

Preperation of 3-(1-phenylethyl)pentane-2,4-dione - General Procedure for the alkylation of 1-phenylethanol and benzylalcohol: In a typical experiment Bi(OTf)₃(H₂O)₄ (0.01 mmol; 0.5 mol%) and 2,4 pentanedione (3 mmol) were dissolved in nitromethane (4 mL) and heated up to 100°C. 1-Phenylethanol (1 mmol) was dissolved in nitromethane (1mL), slowly added for 2.5 h and the remaining solution stirred at 100°C for 30 minutes. The mixture was evaporated under vacuuo and purified by column chromatography on silica gel (gradient hexane / ethylacetate 20:1) to afford 185 mg (93 %) of a colorless oil which solidifies upon standing.

3-benzylpentane-2,4-dione¹ (Table 2 – Entry 1): 58% as a pale yellow oil; ¹H-NMR (250 MHz, CDCl₃): δ = 7.40-7.10 (m, 10H, keto+enol), 4.02 (t, *J* = 7.5 Hz, 1H, keto), 3.67 (s, 2H, enol), 3.16 (d, *J* = 7.5 Hz, 2H, keto), 2.13 (s, 6H), 2.08 (s, 6H); ¹³C-NMR (63 MHz, CDCl₃): δ = 203.6, 191.9, 139.7, 138.0, 128.8, 128.7, 128.6, 127.4, 126.8, 126.3, 108.3, 70.0, 34.3, 32.9, 29.8, 23.3.

3-(1-phenylethyl)pentane-2,4-dione² (Table 2 – Entry 2): 91% as a colorless oil which solidifies upon standing; ¹H-NMR (250 MHz, CDCl₃): δ = 7.18 (m, 5H), 3.97 (d, *J* = 11.3 Hz, 1H), 3.52 (m, 1H), 2.20 (s, 3H), 1.76 (s, 3H), 1.14 (d, *J* = 6.9 Hz, 3H); ¹³C-NMR (63 MHz, CDCl₃): δ = 203.5, 203.4, 143.0, 128.8, 127.3, 127.0, 76.7, 40.4, 29.8, 29.7, 20.9.

2-benzyl-1-phenylbutane-1,3-dione (Table 2 – Entry 3): 72% as a pale yellow oil which solidifies upon standing; ¹H-NMR (250 MHz, CDCl₃): 7.95-7.75 (m, 2H), 7.56-7.44 (m, 1H), 7.45-7.32 (m, 2H), 7.19-7.05 (m, 5H), 4.72 (t, *J* = 7.2, Hz, 1H), 3.25 (dd, *J* = 7.2, 4.0 Hz, 2H), 2.06 (s, 3H); ¹³C-NMR (63 MHz, CDCl₃): δ = 203.3, 195.8, 138.4, 136.5, 133.8, 128.9, 128.8, 128.7, 128.7, 126.7, 64.8, 34.8, 28.7; IR (neat): 3447, 1709, 1678, 1450, 1362, 1259, 757, 694, 405 cm⁻¹; MS (ESI): *m/z* (%) = 253.0 (100) ([M+H]⁺); EA: calcd. for C₁₇H₁₆O₂: C 80.93, H 6.39; found: C 81.11, H 6.55.

1-phenyl-2-(1-phenylethyl)butane-1,3-dione³ (Table 2 – Entry 4): 93% as a white solid; ¹H-NMR (250 MHz, CDCl₃): δ = 8.10-7.86 (m, 2H), 7.57-7.47 (m, 1H), 7.47-7.36 (m, 2H), 7.29-7.08 (m, 5H), 4.83 (d, *J* = 11.0 Hz, 1H), 3.80 (qd, *J* = 11.0, 6.8 Hz, 1H), 1.82 (s, 3H), 1.14 (d, *J* = 6.8 Hz, 3H); ¹³C-NMR (63 MHz, CDCl₃): δ = 203.2, 195.2, 143.2, 137.3, 133.9, 128.9, 128.9, 127.5, 127.0, 70.9, 41.0, 28.0, 21.6.

2-benzyl-1,3-diphenylpropane-1,3-dione⁵ (Table 2 – Entry 5): 84% as a pale yellow solid; ¹H-NMR (250 MHz, CDCl₃) δ = 7.89-7.71 (m, 4H), 7.51-7.40 (m, 2H), 7.36-7.27 (m, 4H), 7.19-7.03 (m, 5H), 5.44 (t, *J* = 6.7 Hz, 1H), 3.37 (d, *J* = 6.7 Hz, 2H); ¹³C-NMR (63 MHz, CDCl₃) δ = 195.4, 139.1, 136.1, 133.5, 129.0, 128.9, 128.6, 126.7, 59.1, 35.3.

1,3-diphenyl-2-(1-phenylethyl)propane-1,3-dione² (Table 2 – Entry 6): 96% as a white solid; ¹H-NMR (300 MHz, CDCl₃): δ 8.03-7.88 (m, 2H), 7.72-7.61 (m, 2H), 7.54-7.42 (m, 1H), 7.41-7.29 (m, 3H), 7.24-7.15 (m, 4H), 7.13-7.05 (m, 2H), 7.04-6.95 (m, 1H), 5.53 (d, *J* = 10.1 Hz, 1H), 4.00 (qd, *J* = 10.1, 7.0 Hz, 1H), 1.27 (d, *J* = 7.0 Hz, 3H); ¹³C-NMR (63 MHz, CDCl₃): δ = 195.0, 194.6, 143.9, 137.2, 136.9, 133.6, 133.0, 128.9, 128.8, 128.5, 128.5, 128.4, 127.8, 126.6, 65.0, 41.2, 20.3.

3-(1-(4-bromophenyl)ethyl)pentane-2,4-dione (Table 3 – Entry 2): 84% as a white solid; ¹H-NMR (250 MHz, CDCl₃): δ = 7.39-7.30 (m, 2H), 7.03-6.96 (m, 2H), 3.92 (d, *J* = 11.3 Hz, 1H), 3.58-3.42 (m, 1H), 2.19 (s, 3H), 1.80 (s, 3H), 1.11 (d, *J* = 6.9 Hz, 3H); ¹³C-NMR (63 MHz, CDCl₃): δ = 203.0, 202.9, 142.2, 131.9, 129.1, 120.8, 76.6, 39.7, 29.8, 29.6, 20.7; mp: 87°C; IR (KBr): 2965, 1722, 1697, 1491, 1363, 1284, 1176, 1146, 822 cm⁻¹; MS (ESI): *m/z* (%) = 304.8 (100) ([M+Na]⁺), 306.9 (98) ([M+Na]⁺); EA: calcd. for C₁₃H₁₅BrO₂: C 55.14, H 5.34; found: C 55.14, H 5.40.

3-(1-(3-bromophenyl)ethyl)pentane-2,4-dione (Table 3 – Entry 3): 65% as a colorless oil; ¹H-NMR (250 MHz, CDCl₃) δ = 7.41-7.29 (m, 2H), 7.21-7.06 (m, 2H), 4.00 (d, *J* = 11.3 Hz, 1H), 3.56 (qd, *J* = 11.3, 6.9 Hz, 1H), 2.25 (s, 3H), 1.88 (s, 3H), 1.19 (d, *J* = 6.9 Hz, 3H); ¹³C-NMR (63 MHz, CDCl₃) δ = 202.9, 202.8, 145.5, 130.4, 130.4, 130.2, 126.0, 122.8, 76.4, 39.9, 29.9, 29.7, 20.7; IR (neat): 2968, 2933, 1726, 1699, 1427, 1358, 1201, 1157, 785, 698 cm⁻¹; MS (ESI): *m/z* (%) = 299.9 (100) ([M+NH₄]⁺), 301.9 (98) ([M+NH₄]⁺), EA: calcd. for C₁₃H₁₅BrO₂: C 55.14, H 5.34; found: C 55.26, H 5.41.

3-(1-(naphthalen-2-yl)ethyl)pentane-2,4-dione (Table 3 – Entry 4): 87% as a white solid; ¹H-NMR (250 MHz, CDCl₃) δ = 7.87-7.76 (m, 3H), 7.69-7.63 (m, 1H), 7.55-7.42 (m, 2H), 7.40-7.34 (m, 1H), 4.20 (d, *J* = 11.3 Hz, 1H), 3.81 (qd, *J* = 11.3, 6.8 Hz, 1H), 2.34 (s, 3H), 1.87 (s, 3H), 1.33 (d, *J* = 6.8 Hz, 3H); ¹³C-NMR (63 MHz, CDCl₃) δ = 203.4, 140.5, 133.5, 132.5, 128.7, 127.8, 127.7, 126.2, 126.0, 125.8, 125.3, 76.6, 40.5, 29.8, 29.8, 20.9; mp:

93°C; IR (KBr): 2962, 1691, 1359, 1193, 1153, 904, 864 cm^{-1} ; MS (ESI): m/z (%) = 272.0 (100) ($[\text{M}+\text{NH}_4]^+$); EA: calcd. for $\text{C}_{17}\text{H}_{18}\text{O}_2$: C 80.28, H 7.13; found: C 80.39, H 7.22.

3-(1-*o*-tolylethyl)pentane-2,4-dione (Table 3 – Entry 5): 85% as a colorless oil; ^1H -NMR (250 MHz, CDCl_3) δ = 7.16-6.89 (m, 4H), 4.09 (d, J = 11.4 Hz, 1H), 3.89-3.73 (m, 1H), 2.32 (s, 3H), 2.21 (s, 3H), 1.74 (s, 3H), 1.05 (d, J = 6.8 Hz, 3H); ^{13}C -NMR (63 MHz, CDCl_3) δ = 203.5, 203.3, 141.4, 135.4, 130.9, 126.6, 126.5, 125.5, 76.0, 35.0, 29.9, 29.4, 20.6, 19.6; IR (neat): 2969, 1697, 1356, 1184, 1159, 762, 729 cm^{-1} ; MS (ESI): m/z (%) = 240.9 (100) ($[\text{M}+\text{Na}]^+$), 236.1 (67) ($[\text{M}+\text{NH}_4]^+$); EA: calcd. for $\text{C}_{14}\text{H}_{18}\text{O}_2$: C 77.03, H 8.31; found: C 77.14, H 8.38.

3-(1-phenylallyl)pentane-2,4-dione⁴ (Table 3 – Entry 6): 54% as a pale yellow oil–Keto / Enol (1.2 : 2); ^1H -NMR (250 MHz, CDCl_3) δ = 7.45-7.17 (m, 9H, keto+enol), 6.57-6.03 (m, 3H, keto + enol), 3.83 (t, J = 7.3 Hz, 1H, keto), 3.19 (dd, J = 5.2, 1.5 Hz, 2H, enol), 2.78 (dt, J = 7.2, 1.4 Hz, 1H, keto), 2.24 (s, 3H, keto), 2.19 (s, 6H, enol); ^{13}C -NMR (63 MHz, CDCl_3) δ = 203.6, 191.6, 137.1, 136.8, 132.8, 130.1, 128.9, 128.6, 128.6, 127.7, 127.5, 127.3, 126.2, 126.1, 125.5, 68.4, 31.6, 30.5, 29.4, 23.1.

3-(1-(3,4,5-trimethoxyphenyl)ethyl)pentane-2,4-dione (Table 3 – Entry 7): 76% as a white solid; ^1H -NMR (250 MHz, CDCl_3) δ = 6.32 (s, 2H), 3.94 (d, J = 11.3 Hz, 1H), 3.77 (s, 6H), 3.74 (s, 3H), 3.47 (qd, J = 11.1, 6.8 Hz, 1H), 2.20 (s, 3H), 1.82 (s, 3H), 1.13 (d, J = 6.9 Hz, 3H); ^{13}C -NMR (63 MHz, CDCl_3) δ = 203.5, 203.2, 153.3, 138.8, 136.8, 104.2, 76.7, 60.8, 56.1, 40.7, 29.9, 29.6, 20.9; mp: 148-149°C; IR (KBr): 2962, 1724, 1590, 1456, 1421, 1363, 1251, 1132 cm^{-1} ; MS (ESI): m/z (%) = 312.1 (36) ($[\text{M}+\text{NH}_4]^+$), 317.1 (29) ($[\text{M}+\text{Na}]^+$); EA: calcd. for $\text{C}_{14}\text{H}_{18}\text{O}_2$: C 65.29, H 7.53; found: C 65.41, H 7.61

3-(1-(biphenyl-4-yl)ethyl)pentane-2,4-dione (Table 3 – Entry 8): 90% as a white solid; ^1H -NMR (250 MHz, CDCl_3) δ = 7.53-7.41 (m, 4H), 7.41-7.30 (m, 2H), 7.30-7.13 (m, 3H), 4.00 (d, J = 11.3 Hz, 1H), 3.68-3.45 (m, 1H), 2.21 (s, 3H), 1.81 (s, 3H), 1.17 (d, J = 6.8 Hz,

3H); ^{13}C -NMR (63 MHz, CDCl_3) δ = 203.5, 203.4, 142.1, 140.6, 139.9, 128.8, 127.7, 127.5, 127.3, 127.0, 76.8, 40.1, 29.9, 29.8, 20.9; mp: 104-105°C; IR (neat): 2970, 1724, 1693, 1362, 1147, 764 cm^{-1} ; MS (ESI): m/z (%) = 298.0 (35) ($[\text{M}+\text{NH}_4]^+$), 303.0 (32) ($[\text{M}+\text{Na}]^+$); EA: calcd. for $\text{C}_{16}\text{H}_{20}\text{O}_4$: C 81.40, H 7.19; found: C 81.58, H 7.24.

2-(4-chlorobenzyl)-1,3-diphenylpropane-1,3-dione⁶ (Table 4 – Entry 2): 84% as a pale yellow solid; ^1H -NMR (250 MHz, CDCl_3) δ = 7.88-7.75 (m, 4H), 7.54-7.42 (m, 2H), 7.42-7.27 (m, 4H), 7.16-7.06 (m, 4H), 5.39 (t, J = 6.8 Hz, 1H), 3.34 (d, J = 6.8 Hz, 2H); ^{13}C -NMR (63 MHz, CDCl_3) δ = 195.2, 137.5, 135.9, 133.7, 132.5, 130.4, 128.9, 128.7, 128.6, 59.2, 34.6.

2-(4-methylbenzyl)-1,3-diphenylpropane-1,3-dione⁵ (Table 4 – Entry 3): 65% as a pale yellow solid; ^1H -NMR (250 MHz, CDCl_3) δ = 7.80-7.67 (m, 4H), 7.46-7.31 (m, 2H), 7.30-7.17 (m, 4H), 7.04-6.94 (m, 2H), 6.92-6.81 (m, 2H), 5.33 (t, J = 6.6 Hz, 1H), 3.25 (d, J = 6.6 Hz, 2H), 2.11 (s, 3H); ^{13}C -NMR (63 MHz, CDCl_3) δ = 195.5, 136.2, 136.1, 136.0, 133.5, 129.3, 128.9, 128.8, 128.7, 59.3, 34.8, 21.0.

2-(3,4-dimethoxybenzyl)-1,3-diphenylpropane-1,3-dione (Table 4 – Entry 4): 87% as a pale green oil; ^1H -NMR (250 MHz, CDCl_3) δ = 7.89-7.74 (m, 4H), 7.52-7.41 (m, 2H), 7.39-7.27 (m, 4H), 6.74-6.61 (m, 3H), 5.40 (t, J = 6.7 Hz, 1H), 3.75 (s, 3H), 3.69 (s, 3H), 3.33 (d, J = 6.7 Hz, 2H); ^{13}C -NMR (63 MHz, CDCl_3) δ = 195.6, 148.9, 147.8, 136.1, 133.5, 131.6, 128.8, 128.6, 120.9, 112.6, 111.4, 59.5, 55.9, 55.8, 35.1; IR (neat): 3445, 1696, 1660, 1516, 1448, 1262, 1028, 692 cm^{-1} ; MS (ESI): m/z (%) = 375.2 (54) ($[\text{M}+\text{H}]^+$), 392.0 (65) ($[\text{M}+\text{NH}_4]^+$), 397.2 (22) ($[\text{M}+\text{Na}]^+$); EA: calcd. for $\text{C}_{24}\text{H}_{22}\text{O}_4$: C 76.99, H 5.92; found: C 76.80, H 6.09.

2-(2-methoxybenzyl)-1,3-diphenylpropane-1,3-dione (Table 4 – Entry 5): 85% as a pale yellow solid which solidifies upon standing; ^1H -NMR (250 MHz, CDCl_3) δ = 7.86-7.71 (m, 4H), 7.42-7.31 (m, 2H), 7.29-7.18 (m, 4H), 7.15-7.10 (m, 1H), 7.06-6.92 (m, 1H), 6.70-

6.60 (m, 2H), 5.54 (t, $J = 6.8$ Hz, 1H), 3.71 (s, 3H), 3.26 (d, $J = 6.7$ Hz, 2H); ^{13}C -NMR (63 MHz, CDCl_3) $\delta = 195.9, 157.4, 136.2, 133.3, 132.1, 128.7, 128.6, 128.0, 127.0, 120.7, 110.1, 56.2, 55.2, 31.0$; IR (neat): 3448, 1698, 1665, 1595, 1494, 1447, 1343, 1275, 1246, 1195, 1114, 1029, 761, 686, 404 cm^{-1} ; MS (ESI): m/z (%) = 344.9 (100) ($[\text{M}+\text{H}]^+$); EA: calcd. for $\text{C}_{14}\text{H}_{18}\text{O}_3$: C 80.21, H 5.85; found: C 80.04, H 5.93.

2-cinnamyl-1-phenylbutane-1,3-dione⁷ (Table 5 – Entry 3): 73% as a pale yellow oil; ^1H -NMR (250 MHz, CDCl_3) $\delta = 8.14\text{--}7.90$ (m, 2H), 7.69–7.47 (m, 3H), 7.38–7.17 (m, 5H), 6.55–6.44 (m, 1H), 6.25–6.06 (m, 1H), 4.64 (t, $J = 7.1$ Hz, 1H), 2.99–2.85 (m, 2H), 2.21 (s, 3H); ^{13}C -NMR (63 MHz, CDCl_3) $\delta = 203.5, 195.8, 136.9, 136.4, 133.9, 132.7, 129.0, 128.8, 128.5, 127.5, 126.2, 125.9, 63.2, 32.4, 28.3$.

2-cinnamyl-1,3-diphenylpropane-1,3-dione⁸ (Table 5 – Entry 5): 62% as a pale yellow oil which solidifies upon standing; ^1H -NMR (250 MHz, CDCl_3) $\delta = 7.98\text{--}7.78$ (m, 4H), 7.54–7.42 (m, 2H), 7.41–7.30 (m, 4H), 7.23–7.05 (m, 5H), 6.38 (d, $J = 15.8$ Hz, 1H), 6.24–6.03 (m, 1H), 5.28 (t, $J = 6.7$ Hz, 1H), 2.94 (t, $J = 6.9$ Hz, 2H); ^{13}C -NMR (63 MHz, CDCl_3) $\delta = 195.6, 137.0, 136.0, 133.6, 132.5, 129.0, 128.6, 128.5, 127.4, 126.8, 126.2, 57.2, 33.0$.

1-(5,6,7-trimethoxy-1,3-dimethyl-1H-inden-2-yl)ethanone (2b): 70% as a pale yellow oil; ^1H -NMR (300 MHz, CDCl_3) $\delta = 6.67$ (s, 1H), 3.87 (s, 3H), 3.84 (s, 3H), 3.79 (s, 3H), 3.70–3.55 (m, 1H), 2.58 (d, $J = 2.1$ Hz, 3H), 2.36 (s, 3H), 1.26 (d, $J = 7.3$ Hz, 3H); ^{13}C -NMR (63 MHz, CDCl_3) $\delta = 196.5, 154.8, 150.6, 149.1, 146.9, 143.2, 141.5, 128.5, 102.5, 61.3, 60.9, 56.2, 45.0, 31.0, 17.6, 15.2$; IR (neat): 2931, 1629, 1599, 1463, 1340, 1230, 1101, 1031 cm^{-1} ; MS (ESI): m/z (%) = 277.1 (100) ($[\text{M}+\text{H}]^+$); EA: calcd. for $\text{C}_{16}\text{H}_{20}\text{O}_4$: C 69.54, H 7.30; found: C 69.48, H 7.50.

References

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BN 283 Diastereomer I - File: xf14bnru.7893

203.165
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127.034
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27.961
21.608

ppm (f1)

200

150

S17 100

50

0

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1.258

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4.008
3.998
3.975

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7.947
7.942
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7.675
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BN 284 - File: uf16bnru.986

ppm (f1)

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¹³C

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0

250
ppm (t1)

200

150

100
S23

50

0

BN 294 - File:xg05bnru.8372

BN 309 - File:xg13bnru.8620

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BN 300 - File: xg11bnru.8479

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128.684	
132.503	
133.515	
140.532	
203.433

BN 304 - File: xg11bnru.8529

S31

BN 252 - File: xe30bnru.7378

31.610
30.515
29.414
23.085

68.357

125.460
126.086
126.167
127.319
127.537
127.647
128.565
128.594
128.935
130.101
132.794
136.798
137.080

191.585
203.615

BN 308 - File: xg13bnru.8610

ppm (f1)

BN 305 - File: xg12bnru.8555

20.930
29.747
29.887

40.092

76.753

126.998
127.323
127.516
127.724
128.798
139.871
140.599
142.133

203.406
203.493

32.443
28.343

63.177

125.938
126.206
127.457
128.538
128.805
128.971
132.729
133.865
136.381
136.945

195.818
203.519

BN 261 - File: xe18bnru.7135

ppm (t1)

200

150

S45

100

50

0

BN 243 - File: xe17bnru.7095

BN 308 - Fr. 1 - File: ug12bnu.1152