Supporting information

3-Carboxylate Substituted Isoindolinones in K₂CO₃ Catalyzed Michael Reactions.

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MATERIALS AND METHODS

All reactions were performed using commercially available compounds without further purification. Isoindolinones were prepared according to reported procedures. Column chromatographic purification of products was carried out using silica gel 60 (70–230 mesh, Merck). The NMR spectra were recorded on Bruker DRX 400, 300, 250 spectrometers (400 MHz, 300 MHz, 250 MHz, ¹H; 100 MHz, 75 MHz, 62,5 MHz ¹³C). Spectra were referenced to residual CHCl₃ (7.26 ppm, ¹H, 77.23 ppm, ¹³C). Coupling constants *J* are reported in Hz. Yields are given for isolated products showing one spot on a TLC plate and no impurities detectable in the NMR spectrum. Mass spectral analyses were carried out using an electrospray spectrometer, Waters 4 micro quadrupole. Elemental analyses were performed with FLASHEA 1112 series-Thermo Scientific for CHNS-O apparatus.

The isoindolinones **1a**, **1b** and **1c** were synthesized according to reported procedures;¹ only **1b** has never been described and the spectroscopic data are given.

1a . Ethyl 2-(phenylmethyl)phthalimidine-3-carboxylate^{1a-c}

Yield 87%; solid, mp 75°-76°C. Anal. Calcd. for C₁₈H₁₇NO₃: C, 73.20; H, 5.80; N, 4.74. Found: C, 73.30; H, 5.75; N, 4.65.

1b. Ethyl 2-butyl-3-oxoisoindoline-1-carboxylate

Yield 85%; oil. ¹H-NMR (400 MHz, CDCl₃) δ: 7.99-7.95 (m, 1H, Ar), 7.74-7.45 (m, 3H), 5.31 (s, 1H, CH), 4.47-4.24 (m, 2H, CH₂), 4.23-4.15 (m, 1H, CH), 3.43-3.32 (m, 1H, CHH), 1.79-1.69 (m, 2H, CH₂), 1.54-1.35 (m, 5H, CH₂-CH₂-CHH), 1.07 (t, 3H, *J* = 7.2 Hz, CH₃). ¹³C-NMR (100 MHz, CDCl₃) δ: 168.7, 168.5, 139.5, 132.2, 131.9, 129.3, 124.0, 122.7, 62.3, 62.2, 41.3, 30.2, 20.3, 14.3, 13.9. MS (ESI): *m/z* = 262 (M + H⁺), Anal. calcd for C₁₅H₁₉NO₃ C, 68.94; H, 7.33; N, 5.36; found C, 68.83; H, 7.46; N, 5.30.

1c. Ethyl 3-oxoisoindoline-1-carboxylate²

Yield 90%; solid, mp 90-91°C. Anal. Calcd. for C₁₁H₁₁NO₃: C, 64.38; H, 5.40; N, 6.63. Found: C, 64.30; H, 5.49; N, 6.55

General procedure for K₂CO₃ catalyzed Michael reaction of isoindolinones **1**.

The Michael acceptor (1.5 eq.) was added at r.t. to a solution of isoindolinones **1** (0.10 mmol) and K₂CO₃ (0.5 eq, 0.05 mmol) in CH₃CN (1 mL). The reaction was stirred until the disappearance of **1** detected by TLC, then the solvent was evaporated and the mixture purified directly on chromatographic column in 8/2 hexane/ethyl acetate, affording the adducts **2** (or **3**) in the range of 75-99% yields

2a. Ethyl 2-benzyl-3-oxo-1-(3-oxobutyl)isoindoline-1-carboxylate

Reacting **1a** (0.1 mmol, 30 mg), methyl vinyl ketone (1.5 eq, 11 mg, 13 μ L) and K₂CO₃ (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 33 mg, 90%. ¹H-NMR (400 MHz, CDCl₃) δ : 7.91 (d, J =8 Hz, 1H, Ar), 7.55-7.46 (m, 2H, Ar), 7.4 (m, 3H, Ar), 7.27 (q, J =7.6Hz, 3H, Ar), 5.1 (d, J =15.2Hz, 1H, *N*-CHH), 4.3 (d, J =15.2 Hz, 1H, *N*-CHH), 4.0 (q, J =7.2 Hz, 2H, CH₂), 2.74-2.67 (m, 1H, CHH), 2.39-2.35 (m, 1H, CHH), 1.61 (s, 3H, CH₃), 1.48-1.41 (m, 2H, CH₂), 1.09 (t, J =7.0 Hz, 3H, CH₃). ¹³C-NMR (100 MHz) δ : 206.5, 169.8, 169.1, 142.9, 137.2, 132.2, 131.5, 129.2, 129.0, 128.4, 127.6, 123.9, 121.4, 71.6, 62.1, 44.7, 35.9, 29.4, 26.2, 13.7. MS (ESI): m/z = 366 (M + H⁺), Anal. calcd for C₂₂H₂₃NO₄ C, 72.31; H, 6.34; N, 3.83; found C, 72.20; H, 6.45; N, 3.70;

2b. Ethyl 2-butyl-3-oxo-1-(3-oxobutyl)isoindoline-1-carboxylate

Reacting **1b** (0.1 mmol, 26 mg), methyl vinyl ketone (1.5 eq, 11 mg, 13 μ L) and K₂CO₃ (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 30 mg, 89%. ¹H-NMR (400 MHz, CDCl₃) δ : 7.84 (d, J =7.4Hz, 1H, Ar), 7.56-7.47 (m, 2H, Ar), 7.40 (d, J =7.1Hz, 1H, Ar), 4.18-4.09 (m, 2H, CH₂), 3.44-3.36 (m, 1H, CHH), 3.34-3.31 (m, 1H, CHH), 2.86-2.78 (m, 1H, CHH), 2.54-2.47 (m, 1H, CHH), 2.03-1.94 (m, 1H, CHH), 1.75 (s, 3H, CH₃), 1.73-1.68 (m, 2H, CH₂), 1.61-1.55 (m, 1H, CHH), 1.39 (q, J =7.5Hz, 2H, CH₂), 1.16 (t, J =7.1 Hz, 3H, CH₃), 0.94 (t, J =7.3 Hz, 3H, CH₃). ¹³C-NMR (100 MHz) δ : 206.6, 170.2, 169.1, 142.8, 132.1, 132.0, 129.2, 123.6, 121.6, 71.3, 62.1, 41.5, 36.4, 30.3, 29.9, 25.9, 20.5, 13.8, 13.6. MS (ESI): m/z = 332 (M + H⁺), Anal. calcd for C₁₉H₂₅NO₄ C, 68.86; H, 7.60; N, 4.23; C, 68.94; H, 7.75; N, 4.15.

2c. Ethyl 3-oxo-1-(3-oxobutyl)isoindoline-1-carboxylate

Reacting **1c** (0.1 mmol, 20 mg), methyl vinyl ketone (1.5 eq, 11 mg, 13 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 23 mg, 85%. 1H -NMR (400 MHz, $CDCl_3$) δ : 7.82 (d, J = 7.28, 1H, Ar), 7.63-7.51 (m, 3H, Ar), 6.68 (brs, 1H, NH), 4.22 (q, J = 6.6 Hz, 2H, CH_2), 2.56-2.25 (m, 2H, CH_2), 2.19-2.12 (m, 2H, CH_2), 2.03 (s, 3H, CH_3), 1.26 (t, J = 7.1 Hz, 3H, CH_3). ^{13}C -NMR (100 MHz) δ : 206.8, 170.3, 169.8, 144.1, 132.6, 131.0, 129.3, 123.8, 123.3, 67.0, 62.4, 37.1, 30.9, 29.9, 13.9. MS (ESI): m/z = 276 ($M + H^+$), Anal. calcd for $C_{15}H_{17}NO_4$ C, 65.44; H, 6.22; N, 5.09; C, 65.54; H, 6.34; N, 5.15.

2d. Ethyl 2-benzyl-3-oxo-1-(3-oxopentyl)isoindoline-1-carboxylate

Reacting **1a** (0.1 mmol, 30 mg), ethyl vinyl ketone (1.5 eq, 13 mg, 15 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 36 mg, 95%. 1H -NMR (400 MHz, $CDCl_3$) δ : 7.89 (d, J = 1.5 Hz, 1H, Ar), 7.87-7.43 (m, 3H, Ar), 7.38-7.35 (m, 1H, Ar), 7.27-7.20 (m, 4H, Ar), 5.06 (d, J = 15.3 Hz, 1H, $N-CHH$), 4.31 (d, J = 15.3 Hz, 1H, $N-CHH$), 3.97-3.92 (m, 2H, CH_2), 2.72-2.67 (m, 1H, CHH), 2.44-2.33 (m, 1H, CHH), 1.80-1.76 (m, 2H, CH_2), 1.45-1.36 (m, 2H, CH_2), 1.08-1.02 (m, 3H, CH_3), 0.72 (t, J = 7.3 Hz, 3H, CH_3). ^{13}C -NMR (100 MHz) δ : 209.6, 170.2, 169.5, 143.7, 137.9, 132.5, 131.9, 129.5, 129.4, 128.7, 127.8, 124.2, 121.9, 72.0, 62.4, 45.1, 35.7, 35.0, 26.5, 14.0, 8.2. MS (ESI): m/z = 380 ($M + H^+$), Anal. calcd for $C_{23}H_{25}NO_4$ C, 72.80; H, 6.64; N, 3.69; found C, 72.95; H, 6.60; N, 3.59.

2e. Ethyl 2-benzyl-3-oxo-1-(3-oxopropyl)isoindoline-1-carboxylate

Reacting **1a** (0.1 mmol, 30 mg), acrolein (1.5 eq, 8 mg, 10 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 32 mg, 90%. 1H -NMR (300 MHz, $CDCl_3$) δ : 9.10 (s, 1H, CHO), 7.89-7.86 (m, 1H, Ar), 7.88-7.41 (m, 6H, Ar), 7.37-7.20 (m, 2H, Ar), 5.07 (d, J = 15.2 Hz, 1H, $N-CHH$), 4.31 (d, J = 15.2 Hz, 1H, $N-CHH$), 4.01-3.88 (m, 2H, CH_2), 2.75-2.65 (m, 1H, CHH), 2.44-2.34 (m, 1H, CHH), 1.48 (t, J = 7.7 Hz, 2H, CH_2), 1.05 (t, J = 7.1 Hz, 3H, CH_3). ^{13}C -NMR (75 MHz) δ : 200.0, 170.1,

169.4, 143.0, 137.4, 132.6, 131.9, 129.7, 129.3, 128.8, 128.0, 124.4, 121.6, 71.7, 62.5, 45.1, 37.2, 24.8, 14.0. MS (ESI): $m/z = 352$ ($M + H^+$), Anal. calcd for $C_{21}H_{21}NO_4$ C, 71.78; H, 6.02; N, 3.99, found C, 71.90; H, 6.12; N, 3.87

2f. Ethyl 2-butyl-1-(3-methoxy-3-oxopropyl)-3-oxoisindoline-1-carboxylate

Reacting **1b** (0.1 mmol, 26 mg), methyl acrylate (1.5 eq, 13 mg, 14 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 26 mg, 75%. 1H -NMR (250 MHz, $CDCl_3$) δ : 7.82 (d, $J=6.5$ Hz, 1H, Ar), 7.54-7.40 (m, 3H, Ar), 4.19-4.08 (m, 2H, Ar), 3.55 (s, 3H, CH_3), 3.47-3.32 (m, 2H, CH_2), 2.94-2.84 (m, 1H, CHH), 2.62-2.51 (m, 1H, CHH), 1.95-1.83 (m, 1H, CHH), 1.73-1.62 (m, 3H, CH_2-CHH), 1.4 (q, $J=7.3$ Hz, 2H, CH_2), 1.15 (t, $J=7.1$ Hz, 3H, CH_3). 0.92 (t, $J=7.1$ Hz, 3H, CH_3). ^{13}C -NMR (60 MHz) δ : 172.6, 170.2, 169.1, 142.5, 132.1, 131.9, 129.2, 123.7, 121.4, 71.3, 62.2, 51.6, 41.5, 30.3, 27.5, 27.4, 20.5, 13.7, 13.6. MS (ESI): $m/z = 348$ ($M + H^+$), Anal. calcd for $C_{19}H_{25}NO_5$ C, 65.69; H, 7.25; N, 4.03; found C, 65.80; H, 7.40; N, 4.09.

2g. Ethyl 2-butyl-1-(2-cyanoethyl)-3-oxoisindoline-1-carboxylate

Reacting **1b** (0.1 mmol, 26 mg), acrylonitrile (1.5 eq, 8 mg, 10 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 31 mg, 97%. 1H -NMR (250 MHz, $CDCl_3$) δ : 7.86-7.83 (m, 1H, Ar), 7.84-7.50 (m, 2H, Ar), 7.44-7.41 (m, 1H, Ar), 4.20-4.05 (m, 2H, CH_2), 3.51-3.42 (m, 1H, CHH), 3.37-3.27 (m, 1H, CHH), 2.98-2.86 (m, 1H, CHH), 2.65-2.53 (m, 1H, CHH), 1.99-1.86 (m, 1H, CHH), 1.73-1.65 (m, 2H, CH_2), 1.41 (q, $J=7.2$ Hz, 2H, CH_2), 1.27-1.25 (m, 1H, CHH), 1.15 (t, $J=7.1$ Hz, 3H, CH_3), 0.92 (t, $J=7.1$ Hz, 3H, CH_3). ^{13}C -NMR (60 MHz) δ : 169.6, 168.9, 141.4, 132.5, 132.2, 129.9, 124.2, 121.2, 118.1, 70.7, 62.6, 60.3, 41.6, 30.5, 28.5, 20.5, 13.7, 11.0. MS (ESI): $m/z = 315$ ($M + H^+$), Anal. calcd for $C_{18}H_{22}N_2O_3$ C, 68.77; H, 7.05; N, 8.91; C, 68.90; H, 7.14; N, 8.82.

2h. Ethyl 3-oxo-1-(4-oxopentan-2-yl)isindoline-1-carboxylate

Reacting **1c** (0.1 mmol, 20 mg), 3-penten-2-one (1.5 eq, 13 mg, 14 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a mixture of diastereomers, 28 mg, 95%. 1H -NMR (400 MHz,

CDCl₃) δ : 7.81 (d, J =7.5 Hz, 1H, Ar), 7.69 (d, J =7.7 Hz, 1H, Ar), 7.60 (t, J =7.5 Hz, 1H, Ar), 7.5 (t, J =7.5 Hz, 1H, Ar), 7.18 (brs, 1H, NH), 7.07 (brs, 1H, NH, minor diastereomer), 4.22-4.16 (m, 2H, CH₂), 3.19-3.14 (m, 1H, CH), 2.50 (dd, J =16.8 Hz, 3.6 Hz, 1H, CH), 2.41 (dd, J =16.8 Hz, 9.2 Hz, 1H, minor diastereomer), 2.17 (s, 3H, CH₃), 1.87 (s, 1H, minor diastereomer), 1.26-1.19 (m, 3H, CH₃), 1.08 (d, J =9.2 Hz, 2H, CH₂), 0.59 (t, J =6.7 Hz, 3H, CH₃). ¹³C-NMR (100 MHz, CDCl₃) δ : 206.6, 170.9, 170.8, 144.4, 132.7, 131.6, 129.5, 123.8, 123.7, 71.5, 62.6, 46.3, 35.7, 30.9, 14.2, 13.6. MS (ESI): m/z = 290 (M + H⁺), Anal. calcd for C₁₆H₁₉NO₄: C, 64.42; H, 6.62; N, 4.84; found: : 64.55; H, 6.52; N, 4.71.

2i. Ethyl 3-oxo-1-(3-oxo-1,3-diphenylpropyl)isoindoline-1-carboxylate

Reacting **1c** (0.1 mmol, 20 mg), chalcone (1.5 eq, 31 mg, 29 μ L) and K₂CO₃ (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a mixture of diastereomers, 38 mg, 92%. ¹H-NMR (300 MHz, CDCl₃) δ : 8.39 (brs, 1H, NH) 7.97 (d, J =7.4 Hz, 2H, Ar) 7.79 (d, J =7.7 Hz, 1H, Ar), 7.58-7.49 (m, 3H, Ar) 7.43-7.31 (m, 4H, Ar) 7.13 (d, J =7.3 Hz, 2H, Ar) 6.98 (d, 2H, J = 6.7 Hz, Ar), 6.60 (brs, 1H, minor diast.), 4.68-4.65 (m, 1H, minor diast.), 4.57 (dd, 1H, J_2 =3.9 Hz, J_1 =10 Hz, CH), 4.23 (q, 2H, J =7.2 Hz, CH₂), 4.01 (dd, J_2 =10 Hz J_1 =17 Hz, 1H, CH), 3.36 (dd, 1H, J_2 =3.8 Hz, J_1 =17 Hz, CH), 2.55-2.50 (m, 1H, minor diast.) 1.26 (t, J =7.2 Hz, 3H, CH₃) 1.08 (t, J =7.2 Hz, minor diast., 3H, CH₃). ¹³C-NMR (60 MHz) (CDCl₃) δ : 196.8, 170.9, 170.2, 143.9, 136.6, 136.5, 133.1, 131.8, 131.3, 128.9, 128.4, 128.3, 128.1, 127.6, 127.0, 124.1, 123.3, 71.5, 62.6, 47.2, 40.0, 13.89. MS (ESI): m/z = 414 (M + H⁺), Anal. calcd for C₂₆H₂₃NO₄: C, 75.53; H, 5.61; N, 3.39; found: C, 75.40; H, 5.75; N, 3.25.

3a. Ethyl 2,3,5,9b-tetrahydro-3-hydroxy-5-oxo-1H-pyrrolo[2,1-a]isoindole-9b-carboxylate

Reacting **1c** (0.1 mmol, 20 mg), acrolein (1.5 eq, 8 mg, 10 μ L) and K₂CO₃ (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a wax-like solid, 24 mg, 90%. ¹H-NMR (300 MHz, CDCl₃) δ : 7.77 (d, J =7.1 Hz, 1H, Ar), 7.61-7.45 (m, 3H, Ar), 5.71 (q, J =6.3 Hz, 1H, CH), 4.26-4.21 (m, 2H, CH₂), 3.63 (d, 1H, J = 6.1 Hz, OH), 2.80-2.70 (m, 1H, CHH), 2.68-2.63 (m, 1H, CHH), 2.24-2.18 (m, 1H, CHH), 1.72-1.67 (m, 1H, CHH), 1.25 (t, J =6.9 Hz, 3H, CH₃). ¹³C-NMR (100 MHz) δ : 171.4, 171.0, 145.1, 133.2, 131.9, 129.7, 124.9, 123.3, 80.2, 75.5, 62.7, 37.9, 34.2, 14.2. MS (ESI): m/z = 262 (M + H⁺), Anal. calcd for C₁₄H₁₅NO₄: C, 64.36; H, 5.79; N, 5.36; found: : C, 64.51; H, 5.65; N, 5.43.

3b. Ethyl 2,3,5,9b-tetrahydro-3-hydroxy-5-oxo-1-phenyl-1H-pyrrolo[2,1-a]isoindole-9b-carboxylate

Reacting **1c** (0.1 mmol, 20 mg), cinnamaldehyde (1.5 eq, 19 mg, 20 μ L) and K_2CO_3 (0.5 eq, 0.05 mmol, 7 mg), the title compound was obtained as a mixture of diastereomers, 34 mg, 99%. 1H -NMR 300 MHz ($CDCl_3$) δ : 7.85-7.80 (m, 1H, minor diast., Ar), 7.59-7.55 (m, 1H, Ar), 7.43-7.40 (m, minor diast., Ar), 7.29-7.22 (m, 3H, Ar), 7.10-6.91 (m, 3H, Ar), 6.79 (dd, 2H, $J_2=1.5$ Hz, $J_1=6.2$ Hz), 6.12 (q, 1H, $J=6.4$ Hz), 5.82 (t, $J=5.3$ Hz, minor diast., 1H, CH), 4.39-4.20 (m, 3H), 3.99-3.84 (m, minor diast.), 3.64 (d, 1H, $J=6.3$ Hz), 3.13 (q, $J=7.0$ Hz, minor diast.), 2.90 (ddd, 1H, $J_3=2.6$ Hz, $J_2=6.7$ Hz, $J_1=12$ Hz), 2.70 (ddd, 1H, $J_3=4.7$ Hz, $J_2=7.7$ Hz, $J_1=13$ Hz), 1.35 (t, $J=7.1$ Hz, 3H, CH_3) 0.93 (t, $J=7.1$ Hz minor diast., 3H, CH_3). ^{13}C -NMR 75MHz ($CDCl_3$) δ : 171.7, 170.0, 143.3, 141.9, 138.2, 134.7, 132.2, 132.0, 129.5, 129.0, 128.6, 128.2, 127.9, 127.7, 127.0, 125.4, 124.3, 124.0, 79.4, 79.0, 62.7, 62.3, 54.5, 50.0, 44.5, 43.2, 14.0, 13.2. MS (ESI): m/z = 338 ($M + H^+$), Anal. calcd for $C_{20}H_{19}NO_4$: C, 71.20; H, 5.68; N, 4.15; found: C, 71.32; H, 5.77; N, 4.02.

4. Ethyl 2,3,5,9b-tetrahydro-3-oxo-5-oxo-1-phenyl-1H-pyrrolo[2,1-a]isoindole-9b-carboxylate

A mixture of compound **3b** (1 eq., 20 mg, 0.06 mmol) and pyridinium chlorochromate (6 eq., 38 mg, 0.36 mmol) in dichloromethane (1.5 mL) was stirred for 24 h at room temperature. The solid was filtered off and the residue purified by chromatography column in 8/2 hexane/ethyl acetate, affording the product **4** as a white solid. The title compound was obtained as a mixture of diastereomers, 20 mg, 95%. 1H -NMR 300MHz ($CDCl_3$) δ : 7.93 (d, $J=8.7$ Hz, 1H, minor diast., Ar), 7.68 (d, $J=7.5$ Hz, 1H, Ar), 7.43-7.40 (m, minor diast., Ar), 7.39-7.32 (m, 4H, Ar), 7.04-6.96 (m, 4H, Ar), 4.39 (d, $J=7.8$ Hz, 1H), 4.33-4.20 (m, 2H), 4.01-3.49 (m, 1H, minor diast.), 3.40 (dd, 2H, $J_2=7.7$, $J_1=17$ Hz), 2.97 (d, 1H, $J=16$ Hz, minor diast.), 2.94 (d, 1H, $J=17$ Hz), 1.25 (t, $J=7$ Hz, 3H, CH_3) 0.97 (t, $J=7.2$ Hz minor diast., 3H, CH_3). ^{13}C -NMR 75MHz ($CDCl_3$) δ : 171.4, 170.6, 170.1, 167.2, 164.6, 163.7, 143.3, 141.5, 137.2, 134.1, 133.9, 133.6, 130.6, 130.5, 130.2, 129.7, 129.0, 128.9, 128.0, 126.9, 126.0, 125.8, 125.5, 124.6, 63.4, 62.2, 51.8, 46.7, 42.4, 40.3, 14.1, 13.8. MS (ESI): m/z = 336 ($M + H^+$), Anal. calcd for $C_{20}H_{17}NO_4$: C, 71.63; H, 5.11; N, 4.18; found: C, 71.52; H, 5.27; N, 4.02.

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