A Dual Photoredox-Nickel Strategy for Remote Functionalization via Iminyl Radicals: Radical Ring-Opening–Arylation, –Vinylolation and –Alkylation Cascades

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1 Table of Contents

2 General Experimental Details ........................................................................................................ 2
3 Starting Material Preparation......................................................................................................... 3
4 Ring-Opening–Arylation Cascades ............................................................................................... 7
5 Ring-Opening–Vinylolation Cascades .......................................................................................... 16
6 Ring-Opening–Alkylation Cascades ............................................................................................. 21
7 Flow Procedures .......................................................................................................................... 25
8 Safety Tests ................................................................................................................................. 27
9 NMR Spectra ............................................................................................................................... 29
10 References .................................................................................................................................... 63
2 General Experimental Details

All required fine chemicals were used directly without purification unless stated otherwise. All air and moisture sensitive reactions were carried out under nitrogen atmosphere using standard Schlenk manifold technique. THF was distilled from sodium/benzophenone, CH$_2$Cl$_2$ and was distilled from CaH$_2$, CH$_3$CN was distilled from activated 4Å molecular sieves, EtN(i-Pr)$_2$ was distilled over KOH. $^1$H and $^{13}$C Nuclear Magnetic Resonance (NMR) spectra were acquired at various field strengths as indicated and were referenced to CHCl$_3$ (7.26 and 77.0 ppm for $^1$H and $^{13}$C respectively). $^1$H NMR coupling constants are reported in Hertz and refer to apparent multiplicities and not true coupling constants. Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, qi = quintet, sx = sextet, sp = septet, m = multiplet, dd = doublet of doublets, etc.), proton assignment (determined by 2D NMR experiments: COSY, HSQC and HMBC) where possible. High-resolution mass spectra were obtained using a JEOL JMS-700 spectrometer or a Fissions VG Trio 2000 quadrupole mass spectrometer. Spectra were obtained using electron impact ionization (EI) and chemical ionization (CI) techniques, or positive electrospray (ES). Infra-red spectra were recorded using a JASCO FT/IR 410 spectrometer or using an ATI Mattson Genesis Seris FTIR spectrometer as evaporated films or liquid films. Analytical TLC: aluminum backed plates pre-coated (0.25 mm) with Merck Silica Gel 60 F254. Compounds were visualized by exposure to UV-light or by dipping the plates in permanganate (KMnO$_4$) stain followed by heating. Flash column chromatography was performed using Merck Silica Gel 60 (40–63 µm). All mixed solvent eluents are reported as v/v solutions. UV/Vis spectra were obtained using an Agilent 6453 spectrometer and 1 mm High Precision Cell made of quartz from Hellma Analytics. The LEDs used are Kessil H150-blue. All the reactions were conducted in CEM 10 mL glass microwave tubes.
3 Starting Material Preparation

General Procedure for Oxime Preparation – GP1

A solution of the ketone (1.0 equiv.) in MeOH (0.2 M) was treated with 1-carboxy-1-methylethoxyammonium chloride (1.5 equiv.), anhydrous NaOAc (3.0 equiv.) and heated under reflux until the reaction was judged complete by TLC analysis (1 to 6 h). The mixture was cooled to room temperature and diluted with K₂CO₃(aq) and Et₂O. The layers were separated and the organic layer washed with K₂CO₃(aq) (x 2). The combined aqueous extractions were then acidified with HCl (30% in H₂O) and extracted with CH₂Cl₂ (x 3). The combined organic layers were dried (MgSO₄), filtered and evaporated.

2-((Cyclobutylideneamino)oxy)-2-methylpropanoic Acid (1)

Following GP1, cyclobutanone (1.0 g, 14.3 mmol) gave 1 (1.757 g, 72%) as an oil. ¹H NMR (400 MHz, CDCl₃) δ 2.95 (4H, dd, J = 12.5, 7.8 Hz), 2.03 (2H, p, J = 8.1 Hz), 1.50 (6H, s); ¹³C NMR (101 MHz, CDCl₃) δ 178.2, 162.2, 81.2, 32.2, 31.7, 24.6, 14.9. Data in accordance with the literature.¹

2-(((3-(Benzyloxy)cyclobutylidene)amino)oxy)-2-methylpropanoic Acid (S1)

Following GP1, 3-(benzyloxy)cyclobutan-1-one (200 mg, 1.14 mmol) gave S1 (252 mg, 80%) as an oil. ¹H NMR (500 MHz, CDCl₃) δ 7.39–7.28 (5H, m), 4.48 (2H, s), 4.26–4.20 (1H, m), 3.29–3.21 (1H, m), 3.20–3.13 (1H, m), 3.01–2.85 (2H, m), 1.50 (3H, s), 1.49 (3H, s); ¹³C NMR (126 MHz, CDCl₃) δ 178.7, 149.8, 130.2, 128.7, 127.8, 127.3, 81.7, 73.8, 73.2, 71.2, 37.7, 35.0, 24.1; HRMS (ASAP) Found MH⁺ 278.1385, C₁₅H₂₀NO₄ requires 278.1387.
2-((7-(tert-Butoxycarbonyl)-7-azaspiro[3.5]nonan-2-ylidene)amino)oxy)-2-methylpropanoic acid (S2)

Following GP1, tert-Butyl 2-oxo-7-azaspiro[3.5]nonane-7-carboxylate (0.5 g, 2.1 mmol) gave S2 (654 mg, 92%) as an oil. FT-IR ν_{max} (film)/cm\(^{-1}\) 3015, 1682, 1435, 1355, 1215, 1173; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 10.97 (1H, br s), 3.41–3.27 (4H, m), 2.67 (2H, s), 2.63 (2H, s), 1.58 (4H, t, \(J = 5.6\) Hz), 1.48 (6H, s), 1.43 (9H, s); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 179.1, 156.4, 155.0, 80.8, 79.8, 41.9, 41.2, 36.5, 33.4, 28.5, 24.2; HRMS (ESI) Found MH\(^-\) 339.1925, C\(_{17}\)H\(_{28}\)N\(_2\)O\(_5\) requires 339.1920.

2-(((2,2-Dimethylcyclopentylidene)amino)oxy)-2-methylpropanoic Acid (S3)

Following GP1, 2,2-dimethylcyclopentanone (0.22 mL, 1.79 mmol) gave S3 (381 mg, quant.) as an oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 2.54 (2H, t, \(J = 7.5\) Hz), 1.82–1.74 (2H, m), 1.64 (2H, t, \(J = 6.9\) Hz), 1.49 (6H, s), 1.16 (6H, s); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 176.6, 175.1, 81.1, 42.8, 40.7, 27.7, 26.4, 24.3, 20.8. Data in accordance with the literature.\(^1\)

2-((Cyclohexylideneamino)oxy)-2-methylpropanoic Acid (S4)

Following GP1, 2-phenylcyclohexan-1-one (500 mg, 2.87 mmol) gave S4 (552 mg, 70%) as an oil. \(^1\)H NMR (500 MHz, CDCl\(_3\), E/Z isomers) \(\delta\) 7.34–7.14 (5H, m), 4.74 (0.4H, br s), 3.56 (0.6H, dd, \(J = 8.4, 6.7\) Hz), 2.82–2.71 (0.6H, m), 2.39–2.22 (1.4H, m), 2.16–1.88 (2H, m), 1.84–1.66 (2H, m), 1.61–1.41 (2H, m), 1.32 (1.2H, s), 1.3 (1.8H, s); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 180.7, 180.6, 162.6, 162.3, 141.3, 140.1, 128.6, 128.5, 128.3, 127.4, 126.5, 126.1,
82.3, 82.3, 47.2, 37.7, 33.5, 26.7, 25.9, 25.5, 25.4, 25.1, 24.9, 24.5, 23.8. Data in accordance with the literature.\(^1\)

**2-(((1-(tert-Butoxycarbonyl)azetidin-3-ylidene)amino)oxy)-2-methylpropanoic acid (S5)**

Following GP1, tert-butyl 3-oxoazetidine-1-carboxylate (0.5 g, 2.3 mmol) gave S5 (450 mg, 86%) as an oil. FT-IR \(\nu_{\text{max}} \text{(film)/cm}^{-1}\) 2995, 1690, 1532, 1171, 974, 755; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 11.20 (1H, br s), 4.63 (4H, dd, \(J = \) 8.2, 2.9 Hz), 1.51 (6H, s), 1.45 (9H, s); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 179.0, 156.4, 149.0, 81.6, 80.9, 58.38, 28.4, 24.0; HRMS (ESI) Found MH\(^+\) 271.1298, C\(_{12}\)H\(_{20}\)N\(_2\)O\(_5\) requires 271.1294.

**\((E)-2-(((2-(Hex-5-yn-1-yl)cyclobutylidene)amino)oxy)-2-methylpropanoic Acid (44)\)**

This compound was prepared using the following procedure:

2-Cyclobutylidene-1,1-dimethylhydrazine (S6)

A solution of cyclobutanone (3.2 mL, 42 mmol, 1.0 equiv.) in benzene (75 mL) was treated with \(N,N\)-dimethylhydrazine (2.7 mL, 64 mmol, 1.5 equiv.) and TFA (5 drops). The mixture was heated under reflux in a Dean-Stark apparatus for 16 h. The mixture was cooled to r.t. and diluted with Et\(_2\)O (50 mL) and H\(_2\)O (30 mL). The aqueous layer was extracted with Et\(_2\)O (2 × 30 mL). The combined organic phases were washed NaCl(aq) (20 mL), dried (MgSO\(_4\)), filtered and evaporated. Distillation under reduced pressure (76 °C, 10 mbar) gave S6 (3.8 g, 84%) as a liquid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 2.95 (2H, \(t, J = 8.0\)Hz), 2.89 (2H, \(t, J = 8.0\) Hz), 2.55 (6H, s), 1.94 (2H, qt, \(J = 8.0\) Hz, 4.0
Hz); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 160.4, 46.8, 35.7, 35.2, 14.4. Data in accordance with the literature.$^2$

2-(Hex-5-yn-1-yl)cyclobutan-1-one (S7)
S6 (1.5 g, 13.3 mmol, 1 equiv.) was dissolved in dry THF (30 mL), cooled to −5 °C and treated, dropwise, with n-BuLi (8.0 mL, 1.5 equiv., 2.5M in hexane). The mixture was stirred for 1 h at −5 °C and 6-iodo-1-hexyne (2.6 mL, 20.0 mmol, 1.5 equiv.) was added. After 1 h, the mixture was warmed to r.t. and stirred for 6 h. The mixture was diluted with HCl(aq) (15 mL, 10%) and stirred overnight. The aqueous layer was extracted with EtOAc (5 × 30 mL). The combined organic layers were washed with NaHCO$_3$(aq) (30 mL), Na$_2$S$_2$O$_3$(aq) (30 mL) and NaCl(aq) (30 mL). The combined organic layers were dried (MgSO$_4$), filtered, and evaporated. The crude product was used in the next step without further purification.

(E)-2-(((2-(Hex-5-yn-1-yl)cyclobutylidene)amino)oxy)-2-methylpropanoic Acid (44)
Following GP1, S7 (0.4 g, 2.6 mmol) gave 44 (448 mg, 67%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 3095, 2241, 1710, 1131, 836, 744; $^1$H NMR (400 MHz, CDCl$_3$) δ 10.47 (1H, br s), 2.65–2.38 (7H, m), 2.01 (1H, t, $J = 2.6$ Hz), 1.92–1.82 (2H, m), 1.66–1.42 (8H, m), 1.20–1.10 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 177.9, 172.0, 82.3, 80.9, 69.3, 38.6, 34.1, 33.3, 28.1, 24.5, 24.3, 22.6, 17.1, 14.2; HRMS (ESI) Found MNa$^+$ 274.1418, C$_{14}$H$_{21}$O$_3$NNa requires 274.1419.
4 Ring-Opening–Arylation Cascades

General Procedure for Ring-Opening–Arylation Cascades – GP2

To an oven dried microwave vial, was added the oxime (1.5 equiv.), [Ir(dtbbpy)(ppy)$_2$(PF$_6$)] (5 mol%), and, if solid, the aryl bromide (1.0 equiv.). The microwave vial was sealed and evacuated and back-filled with nitrogen (3 cycles). A separate microwave vial was charged with NiCl$_2$•glyme (10 mol%) and ligand (10 mol%). This vial was evacuated and back-filled with nitrogen (3 cycles) then the solvent (0.1M) was added and the mixture stirred for 10-15 mins. The Ni mixture was then added to the other microwave vial along with TMG (2 eq.), and this resulting solution was degassed with nitrogen. If liquid, the aryl bromide (1.0 equiv) was added, the lid sealed with parafilm and the vial irradiated with a blue lamp and cooled with a fan for 16h. The reaction was diluted with brine (5 mL), and extracted with EtOAc (3 x 15 mL). The layers were separated and the combined organic layers were dried (MgSO$_4$), filtered and evaporated. The residue was purified by column chromatography on silica gel.

4-(4-Acetylphenyl)butanenitrile (3)

Following GP2 running the reaction in EtOAc, 1-(4-bromophenyl)ethan-1-one (20 mg, 0.1 mmol) gave 3 (11 mg, 59%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.92 (2H, d, $J = 8.3$ Hz), 7.30 (2H, d, $J = 8.2$ Hz), 2.85 (2H, t, $J = 7.5$ Hz), 2.59 (3H, s), 2.36 (2H, t, $J = 7.0$ Hz), 2.06–1.98 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 197.6, 145.3, 135.5, 128.7, 128.6, 119.1, 34.2, 26.5, 26.4, 16.3. Data in accordance with the literature.$^3$

4-(4-Benzoylphenyl)butanenitrile (4)

Following GP2 running the reaction in EtOAc, (4-bromophenyl)(phenyl)methanone (26 mg, 0.1 mmol) gave 4 (18 mg, 73%) as a solid. FT-IR $\nu_{max}$ (film)/cm$^{-1}$ 2921, 2851, 1655, 1606, 1279; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.82–7.75 (4H, m), 7.60 (1H, t, $J = 7.4$ Hz), 7.49 (2H, t, $J = 7.5$ Hz), 7.31 (2H, d, $J = 7.9$ Hz), 2.88 (2H, t, $J = 7.5$ Hz), 2.37 (2H, t, $J = 7.0$ Hz), 2.09–
1.99 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) δ 196.3, 144.7, 137.6, 136.0, 132.4, 130.6, 130.0, 128.4, 128.3, 119.2, 34.4, 26.6, 16.5; HRMS (ESI) Found MNa$^+$ 272.1042, C$_{17}$H$_{15}$ONa requires 272.1046.

**Methyl 4-(3-cyanopropyl)benzoate (5)**

Following GP2 running the reaction in DME, methyl 4-bromobenzoate (21 mg, 0.1 mmol) gave 5 (12 mg, 58%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.01–7.97 (2H, m), 7.28–7.24 (2H, m), 3.91 (3H, s), 2.84 (2H, t, $J$ = 7.2 Hz), 2.06–1.96 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) 166.8, 145.0, 129.9, 128.4, 119.2, 52.0, 34.3, 26.5, 26.5. Data in accordance with the literature.$^4$

**4-(3-Cyanopropyl)benzonitrile (6)**

Following GP2 running the reaction in DME, 4-bromobenzonitrile (18 mg, 0.1 mmol) gave 6 (13 mg, 76%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.61 (2H, d, $J$ = 8.1 Hz), 7.31 (2H, d, $J$ = 7.9 Hz), 2.85 (2H, d, $J$ = 7.7 Hz), 2.36 (2H, t, $J$ = 7.0 Hz), 2.06–1.93 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 145.3, 132.5, 129.2, 118.9, 118.7, 110.5, 34.4, 26.3, 16.5. Data in accordance with the literature.$^4$

**4-(4-Formylphenyl)butanenitrile (7)**

Following GP2 running the reaction in DME, 4-bromobenzaldehyde (19 mg, 0.1 mmol) gave 7 (12 mg, 67%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 9.96 (1H, s), 7.84 (2H, dd, $J$ = 8.2, 2.4 Hz), 7.37 (2H, d, $J$ = 7.9 Hz), 2.88 (2H, t, $J$ = 7.5 Hz), 2.36 (2H, t, $J$ = 7.0 Hz), 2.07–1.98 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 191.8, 147.0, 134.9, 130.1, 129.1, 119.1, 34.4, 26.4, 16.4. Data in accordance with the literature.$^5$
4-(4-(Methylsulfonyl)phenyl)butanenitrile (8)

Following GP2 running the reaction in EtOAc, 1-bromo-4-(methylsulfonyl)benzene (23 mg, 0.1 mmol) gave 2 (13 mg, 57%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2957, 2924, 2849, 1305, 1148; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.90 (2H, d, $J$ = 8.4 Hz), 7.41 (2H, d, $J$ = 8.3 Hz), 3.05 (3H, s), 2.90 (2H, t, $J$ = 7.5 Hz), 2.37 (2H, t, $J$ = 7.0 Hz), 2.08–1.97 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 146.3, 139.0, 129.4, 127.9, 118.9, 44.6, 34.3, 26.5, 16.6; HRMS (ESI) Found MNa$^+$ 246.0557, C$_{11}$H$_{13}$O$_2$NSNa requires 246.0559.

4-(4-(Trifluoromethyl)phenyl)butanenitrile (9)

Following GP2 running the reaction in DME, 1-bromo-4-chlorobenzene (22 mg, 0.1 mmol) gave 9 (13 mg, 59%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.56 (2H, d, $J$ = 8.0 Hz), 7.30 (2H, d, $J$ = 8.0 Hz), 2.85 (2H, t, $J$ = 7.6 Hz), 2.35 (2H, t, $J$ = 7.2 Hz), 2.04–1.96 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 143.8, 129.5, 128.8, 125.7 (q, $J$ = 3.7 Hz), 124.1 (q, $J$ = 271.0 Hz), 119.1, 34.2, 26.6, 16.5; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ −62.4. Data in accordance with the literature.$^4$

4-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)butanenitrile (10)

Following GP2 running the reaction in EtOAc, 2-(4-bromophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (56 mg, 0.2 mmol) gave 10 (11 mg, 38%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.76 (2H, d, $J$ = 8.0 Hz), 7.20 (2H, d, $J$ = 8.0 Hz), 2.79 (2H, t, $J$ = 7.4 Hz), 2.31 (2H, t, $J$ = 7.1 Hz), 2.07–1.92 (2H, m), 1.34 (12H, s); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 143.0, 135.2, 132.5, 127.9, 119.4, 83.8, 34.6, 26.8, 24.9, 16.4; HRMS (APCI) Found MH$^+$ 272.1809, C$_{16}$H$_{23}$O$_2$NB requires 272.1816.
4-(4-Chlorophenyl)butanenitrile (11)

Following GP2 running the reaction in DME, 1-bromo-4-(trifluoromethyl)benzene (19 mg, 0.1 mmol) gave 11 (5 mg, 25%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.30–7.25 (2H, m), 7.15–7.10 (2H, m), 2.76 (2H, t, $J = 7.2$ Hz), 2.32 (2H, t, $J = 7.2$ Hz), 2.20–1.90 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 138.0, 132.3, 129.7, 128.7, 119.3, 33.6, 26.7, 16.3. Data in accordance with the literature.$^4$

4-(3,5-Bis(trifluoromethyl)phenyl)butanenitrile (13)

Following GP2 running the reaction in EtOAc, 1-bromo-3,5-bis(trifluoromethyl)benzene (29 mg, 0.1 mmol) gave 13 (17 mg, 62%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2928, 1381, 1278, 1173, 1131; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.77 (1H, s), 7.66 (2H, s), 3.00–2.90 (2H, m), 2.41 (2H, t, $J = 6.9$ Hz), 2.10–2.00 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 142.2, 132.0 (q, $J = 33.2$ Hz), 128.5, 123.2 (q, $J = 272.6$ Hz), 120.8 (p, $J = 3.9$ Hz), 118.7, 34.2, 26.6, 16.7; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –62.9; HRMS (ESI) Found M$^{-}$ 280.0567, C$_{12}$H$_8$NF$_6$ requires 280.0566.

4-(1-Oxo-1,3-dihydroisobenzofuran-5-yl)butanenitrile (14)

Following GP2 running the reaction in EtOAc, 5-bromoisobenzofuran-1(3H)-one (21 mg, 0.1 mmol) gave 14 (11 mg, 56%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2916, 2851, 1759, 1044; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.88 (1H, d, $J = 7.8$ Hz), 7.37 (1H, d, $J = 7.9$ Hz), 7.33 (1H, s), 5.31 (2H, s), 2.94 (2H, t, $J = 7.4$ Hz), 2.38 (2H, t, $J = 6.9$ Hz), 2.09–2.00 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 170.7, 147.4, 146.9, 129.6, 126.1, 124.4, 122.0, 118.9, 69.4, 34.7, 26.7, 16.6; HRMS (ESI) Found M$^+$Na$^+$ 224.0681, C$_{12}$H$_{11}$O$_2$NNa requires 224.0682.
4-(2-Methylpyridin-4-yl)butanenitrile (16)

In this case, the reaction was run using GP2 but using (Ir[dF(CF$_3$)ppy]$_2$(dtbpy))(PF$_6$) as the photocatalyst, NiCl$_2$•glyme as the nickel catalyst, 4,4’-dimethyl-2,2’-dipyridyl as the ligand in DMF, 4-bromo-2-methylpyridine (17 mg, 0.1 mmol) gave 16 (8 mg, 47%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2925, 2852, 1606, 1214; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.42 (1H, d, $J$ = 5.2 Hz), 7.00 (1H, s), 6.93 (1H, d, $J$ = 5.4 Hz), 2.75 (2H, t, $J$ = 7.6 Hz), 2.54 (3H, s), 2.35 (2H, t, $J$ = 7.0 Hz), 2.04–1.96 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 158.8, 149.4, 148.8, 123.3, 120.8, 119.0, 33.6, 25.9, 24.4, 16.5; HRMS (ESI) Found MNa$^+$ 183.0892, C$_{10}$H$_{12}$N$_2$Na requires 183.0893.

4-(2-Fluoropyridin-4-yl)butanenitrile (17)

In this case, the reaction was run using GP2 but using (Ir[dF(CF$_3$)ppy]$_2$(dtbpy))(PF$_6$) as the photocatalyst, NiCl$_2$•glyme as the nickel catalyst, 4,4’-dimethyl-2,2’-dipyridyl as the ligand in DMF, 4-bromo-2-fluoropyridine (18 mg, 0.1 mmol) gave 17 (11 mg, 62%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2921, 2851, 1613, 1413; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.16 (1H, d, $J$ = 5.1 Hz), 7.08–6.98 (1H, m), 6.86–6.71 (1H, m), 2.90–2.78 (2H, m), 2.39 (2H, t, $J$ = 7.0 Hz), 2.07–1.97 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 164.2 (d, $J$ = 239.0 Hz), 154.6 (d, $J$ = 7.5 Hz), 147.9 (d, $J$ = 15.3 Hz), 121.5 (d, $J$ = 4.0 Hz), 118.7, 109.2 (d, $J$ = 36.8 Hz), 33.5 (d, $J$ = 3.1 Hz), 25.7, 16.6; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –68.0; HRMS (ESI) Found MH$^+$ 165.0820, C$_9$H$_{10}$N$_2$F requires 165.0823.

4-(5-(Trifluoromethyl)pyridin-2-yl)butanenitrile (18)

In this case, the reaction was run using GP2 but using (Ir[dF(CF$_3$)ppy]$_2$(dtbpy))(PF$_6$) as the photocatalyst, NiCl$_2$•glyme as the nickel catalyst, 4,4’-dimethyl-2,2’-dipyridyl as the ligand in DMF, 2-Bromo-5-(trifluoromethyl)pyridine (22 mg, 0.1 mmol) gave 18 (12 mg, 56%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.80–8.65 (1H, m), 7.80 (1H, dd, $J$ = 8.1 Hz), 7.30 (1H, d, $J$ = 8.1 Hz), 3.04 (2H, t, $J$ = 7.4 Hz), 2.44 (2H, t, $J$ = 7.0 Hz), 2.24–2.09 (2H, m); $^{13}$C NMR
(101 MHz, CDCl₃) δ 161.2 (q, J = 1.4 Hz), 147.3 (q, J = 4.0 Hz), 133.7 (q, J = 3.5 Hz), 124.5 (q, J = 33.1 Hz), 123.6 (q, J = 272.3 Hz), 122.9, 119.2, 36.2, 24.4, 16.6. Data in accordance with the literature.⁶

4-(Quinolin-6-yl)butanenitrile (19)

Following GP2 running the reaction in EtOAc but using K₂CO₃ as the base, 6-bromoquinoline (21 mg, 0.1 mmol) gave 19 (10 mg, 51%) as an oil. FT-IR νmax (film)/cm⁻¹ 2931, 2855, 1610, 1217; ¹H NMR (400 MHz, CDCl₃) δ 8.89 (1H, dd, J = 4.3, 1.7 Hz), 8.12 (1H, dt, J = 8.4, 1.2 Hz), 8.07 (1H, d, J = 8.7 Hz), 7.63 (1H, s), 7.56 (1H, dd, J = 8.6, 2.0 Hz), 7.41 (1H, dd, J = 8.3, 4.2 Hz), 2.99 (2H, t, J = 7.4 Hz), 2.37 (2H, t, J = 7.0 Hz), 2.15–2.05 (2H, m); ¹³C NMR (126 MHz, CDCl₃) δ 150.2, 147.3, 138.0, 135.6, 130.4, 129.9, 128.3, 126.7, 121.4, 119.3, 34.3, 26.7, 16.5; HRMS (ESI) Found MNa⁺ 219.0890, C₁₃H₁₂N₂Na requires 219.0893.

4-(Benzo[d]thiazol-2-yl)butanenitrile (20)

Following GP2 running the reaction in EtOAc, 2-bromobenzo[d]thiazole (21 mg, 0.1 mmol) gave 20 (15 mg, 75%) as an oil. ¹H NMR (400 MHz, CDCl₃) 7.96 (1H d, J = 8.1 Hz), 7.81 (1H, t, J = 8.2 Hz), 7.50–7.44 (1H, m), 7.40–7.35 (1H, m), 3.27 (2H, t, J = 7.1 Hz), 2.56 (2H, t, J = 7.1 Hz), 2.33–2.24 (2H, m); ¹³C NMR (101 MHz, CDCl₃) δ 168.5, 152.9, 134.7, 125.9, 124.8, 122.4, 121.3, 118.8, 32.1, 24.4, 16.2. Data in accordance with the literature.⁷

4-(1H-Benzо[d]imidazol-2-yl)butanenitrile (21)

In this case, the reaction was run using GP2 but using (Ir[ΔF(CF₃)ppy]₂(dtbbpy))(PF₆) as the photocatalyst, NiCl₂•glyme as the nickel catalyst, 4,4’-dimethyl-2,2’-dipyridyl as the ligand in DMF, 2-bromo-1H-benzo[d]imidazole (20 gm, 0.1 mmol) gave 21 (10 mg, 55%) as an oil. FT-IR νmax (film)/cm⁻¹ 2923, 2854, 1456, 1214; ¹H NMR (500 MHz, CDCl₃) δ 9.03 (1H, br s), 7.71 (1H, br s), 7.42 (1H, br. s), 3.09 (2H, t, J = 7.1 Hz), 2.56 (2H, t, J = 6.9 Hz), 2.34–
2.25 (2H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 151.8, 119.2, 118.7, 27.5, 23.3, 16.6; HRMS (ESI) Found MNa$^+$ 208.0845, C$_{11}$H$_{11}$N$_3$Na required 208.0845.

4-(1,3,7-Trimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-8-yl)butanenitrile (22)

Following modified GP2 running the reaction in DME but using NiCl$_2$·dtbbpy S7, 8-bromo-1,3,7-trimethyl-3,7-dihydro-1H-purine-2,6-dione (27 mg, 0.1 mmol) gave 22 (12 mg, 46%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 3.94 (3H, s), 3.55 (3H, s), 3.40 (3H, s), 2.89 (2H, t, $J$ = 7.1 Hz), 2.59 (2H, t, $J$ = 6.9 Hz), 2.22 (2H, p, $J$ = 7.0 Hz); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 155.3, 151.6, 151.5, 147.7, 118.9, 107.5, 31.6, 29.7, 27.9, 24.8, 22.5, 16.8; HRMS (ESI) Found MNa$^+$ 284.1113, C$_{12}$H$_{15}$O$_2$N$_5$Na requires 284.1118. Data in accordance with literature.$^7$

4-(4-Acetylphenyl)-3-(benzyloxy)butanenitrile (23)

Following GP2 running the reaction in EtOAc, S1 (56 mg, 0.2 mmol) gave 23 (12 mg, 20%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.91 (2H, d, $J$ = 8.2 Hz), 7.36–7.29 (5H, m), 7.26–7.22 (2H, m), 4.59 (1H, d, $J$ = 11.7 Hz), 4.51 (1H, d, $J$ = 11.7 Hz), 3.91 (1H, p, $J$ = 5.9 Hz), 3.99 (1H, d, $J$ = 13.9, 6.8 Hz), 2.98 (1H, dd, $J$ = 13.8, 5.9 Hz), 2.61 (3H, s), 2.53 (1H, dd, $J$ = 16.8, 5.4 Hz), 2.47 (1H, dd, $J$ = 16.8, 5.5 Hz); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 197.7, 142.2, 137.0, 136.0, 129.8, 128.7, 128.6, 128.1, 127.8, 117.2, 75.0, 72.2, 40.3, 26.6, 22.7; HRMS (ESI) Found MNa$^+$ 316.1304, C$_{19}$H$_{19}$O$_2$N$_5$Na requires 316.1308.

tert-Butyl 4-(4-acetylbenzyl)-4-(cyanomethyl)piperidine-1-carboxylate (24)

Following GP2 running the reaction in EtOAc, 4′-bromoacetophenone (20 mg, 0.1 mmol) gave 24 (22 mg, 62%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2941, 2836, 2232, 1780, 1730, 1621, 1587, 1325, 974; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.91 (2H, d, $J$ = 7.8 Hz), 7.29 (2H, d, $J$ = 7.9
Hz), 3.64 (2H, dt, J = 12.1, 5.0 Hz), 3.29 (2H, ddd, J = 13.6, 8.6, 3.9 Hz), 2.87 (2H, s), 2.59 (3H, s), 2.23 (2H, s), 1.61 (2H, td, J = 8.5, 2.9 Hz), 1.52 (2H, ddd, J = 13.8, 6.4, 4.0 Hz), 1.45 (9H, s); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 197.7, 154.8, 141.6, 136.1, 130.7, 128.6, 117.7, 80.1, 43.3, 39.5, 35.8, 34.2, 28.5, 26.7, 25.0.; HRMS (ESI) Found MH\(^+\) 357.2183, C\(_{21}\)H\(_{29}\)N\(_2\)O\(_3\) requires 357.2178.

6,6-Diphenylhexanenitrile (26)

Following GP2 running the reaction in EtOAc, bromobenzene (16 mg, 0.1 mmol) gave 26 (11 mg, 41%) as an oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.32–7.18 (8H, m), 7.12–7.09 (2H, m), 3.88 (1H, t, J = 7.8 Hz), 2.26–2.09 (2H, m), 2.03–1.95 (2H, m), 1.82–1.76 (2H, m), 1.39–1.34 (2H, m); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 143.6, 128.5, 128.4, 126.9, 126.2, 119.2, 51.1, 35.1, 27.8, 25.3, 16.6. Data in accordance with the literature.\(^8\)

6-(4-Acetylphenyl)-6-phenylhexanenitrile (27)

Following GP2 running the reaction in EtOAc, 4'-bromoacetophenone (20 mg, 0.1 mmol) gave 27 (17 mg, 56%) as an oil. FT-IR \(\nu_{\text{max}}\) (film)/cm\(^{-1}\) 2928, 2232, 1740, 1131, 836; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.85 (2H, d, J = 8.6 Hz), 7.38–7.28 (4H, m), 7.18–7.15 (3H, m), 3.93 (1H, t, J = 7.8 Hz), 2.61 (3H, s), 2.42–2.28 (2H, m), 2.09–2.01 (2H, m), 1.89–1.82 (2H, m), 1.51–1.38 (2H, m); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 197.2, 149.0, 143.1, 132.0, 129.9, 128.6, 128.5, 127.0, 119.2, 51.1, 35.2, 27.9, 26.7, 25.5, 17.2; HRMS (ESI) Found MNa\(^+\) 314.1517, C\(_{20}\)H\(_{21}\)NONa requires 314.1521.

6-(3,5-Bis(trifluoromethyl)phenyl)-6-phenylhexanenitrile (28)

Following GP2 running the reaction in EtOAc, 1-Bromo-3,5-bis(trifluoromethyl)benzene (30 mg, 0.1 mmol) gave 28 (24 mg, 58%) as an oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.87 (2H, s), Ph NC Ph NC O Me Ph NC CF\(_3\) CF\(_3\)
7.85 (1H, s), 7.31–7.27 (2H, m), 7.22–7.17 (3H, m), 4.00 (1H, t, $J = 7.8$ Hz), 2.40 (2H, t, $J = 6.7$ Hz), 2.42–1.39 (2H, m), 1.75–1.61 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 146.7, 140.3, 132.3 (q, $J = 33.5$ Hz), 129.3, 129.2, 128.3, 125.4, 122.4 (t, $J = 16$ Hz), 121.7, 119.6, 51.2, 33.0, 31.9, 27.4, 24.8, 21.6, 17.2; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ –62.9. Data in accordance with the literature.
5 Ring-Opening–Vinylolation Cascades

General Procedure for Ring-Opening–Vinylolation Cascades – GP3

To an oven dried microwave vial, was added the oxime (1.0 equiv.), 4CzIPN (5 mol%), NiCl$_2$•dtbbpy (10 mol%), Cs$_2$CO$_3$ (0.5 equiv.) and, if solid, alkyne (1.3 equiv.). The microwave vial was sealed and evacuated and back-filled with nitrogen (3 cycles). Acetonitrile (0.1M) and water (20 equiv.) were added and the mixture degassed for 10 mins. Next, if liquid, the alkyne (1.3 equiv.) was added, the lid sealed with parafilm and the vial irradiated and cooled with a fan for 60 h. The reaction was diluted with brine (5 mL), and extracted with EtOAc (3 x 15 mL). The layers were separated and the combined organic layers were dried (MgSO$_4$), filtered and evaporated. The residue was purified via silica gel chromatography.

5-Methylenedecanenitrile (30)

Following GP3, hept-1-yne (12 mg, 0.13 mmol) gave 30 (12 mg, 73%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2930, 2851; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.80 (1H, s), 4.75 (1H, s), 2.34 (2H, t, $J$ = 7.2 Hz), 2.16 (2H, t, $J$ = 7.4 Hz), 2.03–1.95 (2H, m), 1.80 (2H, p, $J$ = 7.2 Hz), 1.48–1.20 (10H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 147.5, 119.8, 110.6, 35.9, 34.8, 31.9, 27.8, 23.5, 22.8, 16.7, 14.3; HRMS (ASAP) Found MH$^+$ 165.1512, C$_{11}$H$_{20}$N requires 165.1517.

5-Methylenenonanenitrile (31)

Following GP3, hex-1-yne (11 mg, 0.13 mmol) gave 31 (10 mg, 64%) as an oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 4.80 (1H, s), 4.75 (1H, s), 2.34 (2H, t, $J$ = 7.2 Hz), 2.17 (2H, t, $J$ = 7.5 Hz), 2.00 (2H, t, $J$ = 7.6 Hz), 1.80 (2H, p, $J$ = 7.3 Hz), 1.37–1.28 (4H, m), 0.91 (3H, t, $J$ = 7.2 Hz); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 147.2, 119.4, 110.3, 35.4, 34.6, 29.9, 23.4, 22.3, 16.4, 13.8. Data in accordance with literature.

9
5-Cyclohexylhex-5-enenitrile (32)

Following GP3, ethynylcyclohexane (14 mg, 0.13 mmol) gave 32 (9 mg, 49%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2925, 2853; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 4.81 (1H, s), 4.71 (1H, s), 2.34 (2H, t, $J = 7.0$ Hz), 2.18 (2H, t, $J = 7.6$ Hz), 1.84–1.72 (10H, m), 1.31–1.23 (4H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 152.7, 119.7, 108.4, 44.0, 33.6, 32.4, 26.7, 26.3, 23.8, 16.6; HRMS (ASAP) Found M$^+$ 178.1588, C$_{12}$H$_{20}$N requires 178.1596.

5-cyclopropylhex-5-enenitrile (33)

Following GP3, ethynylcyclopropane (12 $\mu$L, 0.13 mmol) gave 33 (33%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.68 (s, 1H), 4.67 (s, 1H), 2.36 (2H, t, $J = 7.2$ Hz), 2.22 (2H, t, $J = 7.4$ Hz), 1.92–1.83 (2H, m), 1.40–1.32 (1H, m), 0.68–0.64 (2H, m), 0.46–0.41 (2H, m).

4-Methyleneoctanedinitrile (34)

Following GP3, pent-4-ynenitrile (10 mg, 0.13 mmol) gave 34 (5 mg, 35%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2928, 2859; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.98 (1H, s), 4.95 (1H, s), 2.54–2.49 (2H, m), 2.42–2.36 (4H, m), 2.22 (2H, t, $J = 7.6$ Hz), 1.89–1.79 (2H, m); HRMS (ASAP) Found M$^+$ 149.1072, C$_9$H$_{13}$N$_2$ requires 149.1073.

tert-Butyl (5-cyano-2-methylenepentyl)carbamate (35)

Following GP3, tert-butyl prop-2-yn-1-ylcarbamate (20 mg, 0.13 mmol) gave 35 (10.5 mg, 45%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 3347, 2927, 1702, 1509, 1367, 1214, 1167; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.99 (1H, s), 4.89 (1H, s), 4.64 (1H, br s, NH), 3.73–3.61 (2H, m), 2.36 (2H, t, $J = 7.1$ Hz), 2.20 (2H, t, $J = 7.6$ Hz), 1.84 (2H, p, $J = 7.2$ Hz), 1.45 (9H, s); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 155.9, 144.3, 119.4, 111.6, 79.6, 44.8, 32.4, 28.4, 23.2, 16.6; HRMS (ESI) Found MNa$^+$ 247.1412, C$_{12}$H$_{20}$O$_2$N$_2$Na requires 247.1417.
8-Chloro-5-methyleneoctanenitrile (36)

Following GP3, 5-chloropent-1-yn (13 mg, 0.13 mmol) gave 36 (6 mg, 35%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2923, 2852, 1456, 1215, 756; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.86 (1H, s), 4.84 (1H, s), 3.55 (2H, t, $J$ = 6.5 Hz), 2.35 (2H, t, $J$ = 7.1 Hz), 2.18 (4H, m), 1.96–1.87 (2H, m), 1.82 (2H, p, $J$ = 7.2 Hz); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 145.5, 111.6, 44.4, 34.6, 32.6, 31.0, 23.3, 16.6; HRMS (ASAP) Found MH$^+$ 172.0883, C$_9$H$_{15}$NCl requires 172.0888.

8-(1,3-Dioxoisindolin-2-yl)-5-methyleneoctanenitrile (37)

Following GP3, 2-(pent-4-yn-1-yl)isoindoline-1,3-dione (28 mg, 0.13 mmol) gave 37 (16 mg, 57%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2921, 2851, 1710, 1467, 1396, 1215; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.85 (2H, dd, $J$ = 5.4, 3.1 Hz), 7.72 (2H, dd, $J$ = 5.5, 3.0 Hz), 4.86 (1H, s), 4.81 (1H, s), 3.69 (2H, t, $J$ = 7.3 Hz), 2.34 (2H, t, $J$ = 7.2 Hz), 2.18 (2H, t, $J$ = 7.5 Hz), 2.11–2.05 (2H, m), 1.89–1.73 (4H, m); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 168.4, 145.6, 134.0, 132.1, 123.2, 119.6, 111.2, 37.6, 34.6, 32.9, 26.4, 23.3, 16.5; HRMS (ASAP) Found MH$^+$ 283.1432, C$_{17}$H$_{19}$N$_2$O$_2$ requires 283.1441.

tert-Butyl (cyanomethyl)(2-methyleneheptyl)carbamate (38)

Following GP3, 1-heptyne (13 mg, 0.13 mmol) gave 38 (12.0 mg, 41%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2855, 2347, 2198, 1624, 1587, 1345, 954, 648; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 4.96 (1H, s), 4.88 (1H, s), 4.13 (1H, br s), 4.00 (1H, br s), 3.99 (2H, s), 1.95 (2H, t, $J$ = 7.6 Hz), 1.53–1.43 (11H, m) 1.33–1.25 (4H, m), 0.89 (3H, td, $J$ = 7.0, 2.2 Hz); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 154.8, 143.9, 119.7, 116.0, 113.2, 81.8, 51.8, 33.3, 31.6, 28.3, 28.3, 27.4, 22.6, 14.2; HRMS (ESI) Found MNa$^+$ 289.1887, C$_{15}$H$_{26}$O$_2$N$_2$Na requires 289.1892.
**tert-Butyl (cyanomethyl)(5-(1,3-dioxoisoindolin-2-yl)-2-methylenepentyl)carbamate (39)**

Following **GP3**, N-(4-Pentynyl)phthalimide (28 mg, 0.13 mmol) gave 39 (24.0 mg, 48%) as an oil. FT-IR ν\textsubscript{max} (film)/cm\textsuperscript{–1} 3085, 2997, 2245, 2202, 1786, 1760, 1630, 1335, 1145; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.84 (2H, dd, \textit{J} = 5.5, 3.0 Hz), 7.71 (2H, dd, \textit{J} = 5.4, 3.0 Hz), 5.03 (1H, s), 4.95 (1H, s), 4.14–4.02 (2H, m), 3.92 (2H, s), 3.69 (2H, t, \textit{J} = 7.1 Hz), 2.05–2.02 (2H, m) 1.88–1.84 (2H, m), 1.55–1.44 (9H, m); \textsuperscript{13}C NMR (126 MHz, CDCl\textsubscript{3}) δ 168.5, 156.3, 149.8, 134.1, 132.2, 123.4, 115.9, 113.7, 82.1, 51.8, 37.6, 30.4, 28.3, 26.3, 24.1; HRMS (ESI) Found M\textsuperscript{Na}\textsuperscript{+} 406.1740, C\textsubscript{21}H\textsubscript{25}O\textsubscript{4}N\textsubscript{3}Na requires 406.1743.

**tert-Butyl 4-(cyanomethyl)-4-(2-methyleneheptyl)piperidine-1-carboxylate (40)**

Following **GPX**, 1-heptyne (13 mg, 0.13 mmol) gave 40 (26 mg, 58%) as an oil. FT-IR ν\textsubscript{max} (film)/cm\textsuperscript{–1} 2963, 2845, 2213, 2178, 1780, 1582, 1344, 1165, 974; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 5.02 (1H, s), 4.87 (1H, s), 3.56–3.30 (4H, m), 2.42 (2H, s), 2.24 (2H, s), 2.07–2.01 (2H, m) 1.54–1.53 (4H, m), 1.48 (9H, s), 1.36–1.27 (6H, m), 0.91 (3H, t, \textit{J} = 6.8 Hz); \textsuperscript{13}C NMR (126 MHz, CDCl\textsubscript{3}) δ 154.9, 145.0, 118.0, 115.6, 80.0, 42.8, 41.0, 38.4, 35.4, 31.6, 28.5, 25.7, 24.0, 22.7, 17.4, 14.2; HRMS (ESI) Found M\textsuperscript{Na}\textsuperscript{+} 357.2511, C\textsubscript{20}H\textsubscript{34}O\textsubscript{2}N\textsubscript{2}Na requires 357.2518.

**3-(2-Methylene cyclohexyl)propanenitrile (45)**

Following **GP3**, 44 (200 mg, 0.8 mmol) gave 27 (55 mg, 46%) as an oil. FT-IR ν\textsubscript{max} (film)/cm\textsuperscript{–1} 3009, 2198, 1750, 1620, 1235, 985, 755; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 4.75 (1H, s), 4.61 (1H, s), 2.41–2.29 (2H, m), 2.27–2.15 (2H, m), 2.10–1.94 (2H, m), 1.81–1.70 (1H, m), 1.69–1.48 (5H, m), 1.40–1.32 (1H, m); \textsuperscript{13}C NMR (101 MHz, CDCl\textsubscript{3}) δ 150.2, 120.0, 107.3, 42.0, 33.9, 33.3, 28.4, 27.6, 23.5, 15.2; HRMS (ESI) Found M\textsuperscript{H}\textsuperscript{+} 150.1277, C\textsubscript{10}H\textsubscript{16}N requires 150.1283.
3-(1-Oxaspiro[2.5]octan-4-yl)propanenitrile (S8)

A solution of 45 (50 mg, 0.33 mmol, 1.0 equiv.) in CH₂Cl₂ (2 mL) at room temperature was treated with m-CPBA (70 mg, 0.4 mmol) and NaHCO₃ (34 mg, 0.4 mmol). The mixture was stirred for 2 h, and then Na₂SO₃ (5%) aqueous solution was added. The organic layer was dried (MgSO₄), filtered and evaporated. The crude was purified by column chromatography on silica gel, eluting with hexane–EtOAc (90:10), to give S8 (46 mg, 92%) as an oil as a mixture of diastereomers. FT-IR νₓ_max (film)/cm⁻¹ 2978, 2745, 2260, 1680, 1356, 1245, 1132, 835; ¹H NMR (400 MHz, CDCl₃) δ 2.67–2.64 (1H, m), 2.50 (1H, dd, J = 6.1, 4.5 Hz), 2.42–2.28 (2H, m), 1.93–1.87 (1H, m), 1.86–1.21 (10H, m) ; ¹³C NMR (101 MHz, CDCl₃) δ 119.9M, 119.7m, 61.0M, 60.7m, 53.2M, 50.8m, 40.2M, 39.4m, 33.4M, 31.4m, 30.4M, 29.4m, 25.6M, 25.1m, 25.1M, 25.0m, 23.6M, 21.9m, 15.6M, 15.3m; HRMS (ESI) Found MNa⁺ 188.1046, C₁₀H₁₅ONa requires 188.1051.

7a-(Hydroxymethyl)octahydro-1H-inden-1-one (46)

A mixture of Cp₂TiCl₂ (150 mg, 2.50 mmol) and Zn (110 mg, 1.6 mmol) in deoxygenated THF (4 mL) was stirred at r.t. until the solution turned green. In a separate flask, S8 (40 mg, 0.24 mmol) was dissolved in deoxygenated THF (5 mL). The green Ti(III) solution was slowly added via syringe to S8. After 30 min, an excess of NaH₂PO₃(aq) was added, and the mixture was stirred for 20 min. The aqueous layer was extracted with Et₂O (x 3). The combined organic layers were washed with NaHCO₃(aq) and NaCl(aq), dried (MgSO₄), filtered and evaporated. Purification by column chromatography on silica gel, eluting with hexane–EtOAc (90:10), gave 46 (35 mg, 88%) as an oil. FT-IR νₓ_max (film)/cm⁻¹ 2978, 2745, 2260, 1680, 1356, 1245, 1132, 835; ¹H NMR (400 MHz, CDCl₃) 3.66 (1H, d, J = 11.2 Hz), 3.56 (1H, d, J = 11.2 Hz), 2.63 (1H, br s), 2.41–2.33 (1H, m), 2.20–1.87 (2H, m), 1.88–1.78 (2H, m), 1.60–1.27 (8H, m); ¹³C NMR (101 MHz, CDCl₃) δ 224.0, 63.6, 52.6, 36.6, 36.5, 25.2, 24.1, 22.8, 21.5, 21.1; HRMS (ESI) Found MNa⁺ 191.1043, C₁₀H₁₆O₂Na requires 191.1048.
6 Ring-Opening–Alkylation Cascades

General Procedure for Ring-Opening–Alkylation Cascades – GP4

To an oven dried microwave vial, was added the oxime (1.5 equiv.), \text{Ir}[dF(CF_3)ppy]_2(dtbbpy)(PF_6) (5 mol%), NiCl_2•dtbbpy complex (10 mol%) and Cs_2CO_3 (2 equiv.). The microwave vial was sealed and evacuated and back-filled with nitrogen (3 cycles). The solvent (0.1M) was added and the mixture degassed for 10 mins. The alkyl bromide (1.0 equiv.) was added, the lid sealed with parafilm and the vial irradiated and cooled with a fan for 2h. The reaction was diluted with brine (5 mL), and extracted with EtOAc (3 x 15 mL). The layers were separated and the combined organic layers were dried (MgSO_4), filtered and evaporated. The residue was purified via silica gel chromatography.

7-Phenylheptanenitrile (48)

Following GP4, (3-bromopropyl)benzene (20 mg, 0.1 mmol) gave 48 (12 mg, 66%) as an oil. 

$^1$H NMR (400 MHz, CDCl_3) $\delta$ 7.32–7.26 (2H, m), 7.22–7.14 (3H, m), 2.62 (2H, t, $J = 7.5$ Hz), 2.32 (2H, t, $J = 7.0$ Hz), 1.72–1.58 (4H, m), 1.52–1.33 (4H, m); $^{13}$C NMR (126 MHz, CDCl_3) $\delta$ 142.5, 128.5, 128.3, 125.6, 119.9, 35.7, 31.0, 28.4, 28.2, 25.2, 17.0; HRMS (ESI) Found MNa$^+$ 210.1247, C_{13}H_{17}NNa requires 210.1253. Data in accordance with the literature.$^{10}$

Ethyl 7-cyanoheptanoate (49)

Following GP4, ethyl 4-bromobutanoate (20 mg, 0.1 mmol) gave 49 (10 mg, 55%) as an oil.

$^1$H NMR (400 MHz, CDCl_3) 4.12 (2H, q, $J = 7.1$ Hz), 2.35–2.28 (4H, m), 1.70–1.60 (4H, m), 1.51–1.44 (2H, m), 1.42–1.32 (2H, m), 1.26 (3H, t, $J = 7.1$ Hz); $^{13}$C NMR (126 MHz, CDCl_3) 173.5, 119.7, 60.3, 34.1, 28.3, 28.2, 25.2, 24.6, 17.1, 14.2; HRMS (ESI) Found MNa$^+$ 206.1146, C_{10}H_{17}NO_2Na requires 206.1152. Data in accordance with the literature.$^{11}$
Decanedinitrile (50)

Following GP4, 6-bromohexanenitrile (18 mg, 0.1 mmol) gave 50 (11.0 mg, 64%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 2.55 (4H, td, $J = 6.9, 1.2$ Hz), 2.40 (4H, t, $J = 6.9$ Hz), 1.88–1.43 (8H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 119.4, 42.9, 27.0, 24.9, 21.1, 17.2. Data in accordance with the literature.\textsuperscript{12}

6-Chlorohexanenitrile (51)

Following GP4, 1-bromo-2-chloroethane (28 mg, 0.2 mmol) gave 51 (16.0 mg, 60%) as an oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 3.56 (2H, t, $J = 6.5$ Hz), 2.38 (2H, t, $J = 6.9$ Hz), 1.88–1.77 (2H, m), 1.75–1.67 (2H, m), 1.66–1.59 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 119.4, 43.4, 31.6, 26.0, 24.7, 17.1. Data in accordance with the literature.\textsuperscript{13}

9-(1,3-Dioxoisoindolin-2-yl)nonanenitrile (52)

Following GP4, 2-(5-bromopentyl)isoindoline-1,3-dione (30 mg, 0.1 mmol) gave 52 (15.0 mg, 52%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 3019, 2253, 1690, 1650, 1375, 1038, 917, 735; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.77 (2H, dd, $J = 5.4, 3.1$ Hz), 7.65 (2H, dd, $J = 5.5, 3.1$ Hz), 3.63 (2H, t, $J = 7.1$ Hz), 2.28 (2H, t, $J = 7.1$ Hz), 1.69–1.62 (2H, m), 1.47–1.34 (10H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 168.4, 134.1, 132.1, 123.3, 119.6, 41.0, 37.4, 27.8, 27.0, 25.9, 25.0, 17.1; HRMS (ESI) Found MNa$^+$ 307.1419, C$_{17}$H$_{20}$O$_2$N$_2$Na requires 307.1422.

8-(1,3-Dioxolan-2-yl)octanenitrile (53)

Following GP4, 2-(4-Bromobutyl)-1,3-dioxolane (21 mg, 0.1 mmol) gave 53 (10.0 mg, 46%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2975, 2836, 1611, 1583, 1552, 1099, 921, 884, 763, 614; $^1$H NMR (400 MHz, CDCl$_3$) δ 4.73 (1H, t, $J = 4.5$ Hz), 3.85–3.82 (2H, m), 3.74–3.70 (2H, m), 2.40 (2H, t, $J = 7.0$ Hz), 2.28–2.21 (4H, m), 1.68–1.53 (6H, m), 1.49–1.43 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 119.6, 103.9, 64.9, 42.8, 32.8, 25.3, 24.8, 23.0, 21.0, 17.1; HRMS (ESI) Found MNa$^+$ 220.1315, C$_{11}$H$_{19}$O$_2$NNa requires 220.1313.
6,9-Diphenylnonanenitrile (56)

Following GP4, (3-bromopropyl)benzene (20 mg, 0.1 mmol) gave 56 (19.0 mg, 65%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 2997, 2226, 1698, 1337, 1284, 774; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36–7.31 (4H, m), 7.26–7.20 (6H, m), 2.73–2.67 (5H, m), 2.43–2.35 (4H, m), 2.05–1.97 (2H, m), 1.86–1.68 (4H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 141.4, 141.3, 128.6, 128.6, 128.5, 128.4, 126.2, 126.1, 119.7, 40.3, 35.1, 35.0, 33.4, 30.3, 26.3, 24.9, 17.1; HRMS (ESI) Found MH$^+$ 292.2069, C$_{21}$H$_{26}$N requires 292.2065.

Ethyl 9-cyano-5-phenylnonanoate (57)

Following GP4, ethyl 4-bromobutanoate (20 mg, 0.1 mmol) gave 57 (19.0 mg, 68%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 3251, 2854, 2264, 1766, 1622, 1389, 1145, 965, 741; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.38–7.26 (3H, m), 7.18–7.15 (2H, m), 4.17 (2H, q, $J$ = 7.1 Hz), 2.58–2.44 (5H, m), 2.32–2.16 (4H, m), 2.08–1.82 (6H, m), 1.29 (3H, t, $J$ = 7.1 Hz); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 171.1, 141.2, 128.5, 128.4, 126.3, 126.1, 119.7, 60.4, 41.0, 35.0, 30.3, 25.0, 24.9, 21.1, 17.1, 14.2; HRMS (ESI) Found MNa$^+$ 310.1778, C$_{18}$H$_{25}$O$_2$NNa requires 310.1783.

Methyl (S)-2-((tert-Butoxycarbonyl)amino)-6-cyanohexanoate (59)

Following GP4, L-Br-serine 58 (28 mg, 0.1 mmol) gave 59 (10.0 mg, 36%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 3257, 2998, 2278, 2146, 1785, 1702, 1521, 1470, 1323, 1149, 941, 825; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.04 (1H, d, $J$ = 8.5 Hz), 4.31–4.26 (1H, m), 3.73 (3H, s), 2.67 (2H, t, $J$ = 6.8 Hz), 1.83–1.77 (2H, m), 1.67–1.59 (2H, m), 1.43 (9H, s), 1.44–1.42 (2H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 173.5, 155.5, 79.8, 53.6, 51.2, 32.6, 31.0, 30.5, 28.4, 23.5, 18.5; HRMS (ESI) Found MH$^+$ 271.1658, C$_{13}$H$_{23}$N$_2$O$_4$ requires 271.1652.
Methyl (S)-7-Amino-2-((tert-butoxycarbonyl)amino)heptanoate (60)

A solution of CoCl$_2$•6H$_2$O (5 mg, 0.02 mmol) and 59 (27 mg, 0.1 mmol) in THF (6 mL) and H$_2$O (3 mL) was stirred vigorously and cooled intermittently with an ice-water bath while NaBH$_4$ (53 mg, 1.4 mmol) was added in portions over 8 min. The reaction was exothermic, producing black precipitate and ample quantities of hydrogen. After a total time of 2h, aqueous NH$_4$OH solution (2 ml) was added and the mixture was then filtered through Celite and washed with methanol. The combined supernatants were concentrated at reduced pressure, then the aqueous residue was extracted with (4 x 10 ml) of DCM. The combined DCM layers were dried (MgSO$_4$) and concentrated to afford 60 (16.5 mg, 59%) as an oil. FT-IR $\nu_{\text{max}}$ (film)/cm$^{-1}$ 3526, 3214, 2989, 2265, 1764, 1780, 1724, 1556, 865, 705; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.10 (1H, d, $J = 8.5$ Hz), 4.28–4.22 (1H, m), 3.69 (3H, s), 3.14 (2H, t, $J = 7.1$ Hz), 1.96 (2H, t, $J = 1.4$ Hz), 1.80–1.75 (2H, m), 1.64–1.56 (2H, m), 1.40 (9H, s), 1.42–1.34 (4H, m); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 173.5, 155.5, 80.0, 53.5, 52.4, 42.0, 33.3, 32.7, 31.1, 28.4, 22.7; HRMS (ESI) Found MNa$^+$ 297.1793, C$_{13}$H$_{26}$O$_4$N$_2$Na requires 297.1790.
7 Flow Procedures

Flow equipment set up
The flow process was performed using a standard setup on the commercially available VapourTech E series 3-pump EasyMedchem fitted with a photochemical 450 nm LED reactor [Part number 50-1319, 10 mL internal volume] and a heating mode at 40°C. A single pump fitted with a blue pump tube [Part number 50-1301] was used to transfer the homogeneous batch solution through the photochemical reactor. The output of the flow reactor was a homogenous solution of a darker yellow/orange compared to the input solution which was yellow.

General Flow Procedure – GP5
To a 1 neck 100 ml flask open to the air, was added 1 (1.5 equiv), [Ir(dtbbpy)(ppy)](PF₆) (5.0 mol%) and the bromoarene (1.0 equiv.). The flask was sealed with a septum and evacuated and backfilled with N₂ (x 3). A separate flask was charged with NiCl₂•6H₂O (10 mol%) and dtbbpy (10 mol%) and a magnetic stirrer bar. The flask was sealed with a septum and evacuated and backfilled with N₂ (x 3). Anhydrous DME and DMF (5:2, 0.05M) were added and the solvent stirred and degassed by bubbling with N₂ for 10 min. The green solution was then added to the first flask, followed by TMG (1.7 equiv.) and the septum sealed with parafilm. Prior to the pumping of the reagent the reactor was fully liquid filled with DME:DMF (5:2) from the solvent reservoir. The reaction solution was pumped at 0.35 mL/min (input flowrate on control interface) resulting in a theoretical residence time of 28.5 min within the photochemical reactor. Once the entire contents of the flask had been pumped from the flask DME:DMF mixture (5:2) was pumped from the solvent reservoir until it was deemed all the reaction solution had been collected. The resulting solution was diluted with water (20 mL) then extracted with ethyl acetate (3 x 100 mL). The combined organic layers were dried (MgSO₄), filtered and evaporated. The residue was purified by FCC with 5% → 20% EtOAc:hexane to give desired product.

4-(4-Acetylphenyl)butanenitrile (3)
Following GP5, 4’bromoacetophenone (796 mg, 4 mmol, 1 equiv) gave 3 (417 mg, 2.24 mmol, 56%) at a flow rate of 0.35mL/min and reactor temperature of 40 °C.

4-(3-Cyanopropyl)benzonitrile (6)
Following GP5, 4-bromobenzonitrile (364 mg, 2 mmol, 1 equiv) gave 6 (195 mg, 1.14 mmol, 57%) at a flow rate of 0.35mL/min and reactor temperature of 40°C.
The temperature was monitored during the flow run as well as NMR samples of aliquots of the reaction solution. The temperature within the photochemical reactor was set at 40 °C with minimal deviation from this observed during the course of the run.

![Graph showing % product (NMR) during the run.](image)

**Figure S1.** Graph showing % product (NMR) during the run.

To aliquots of the reaction solution was added TNCB as a standard in EtOAc. The resulting solution was diluted with water and EtOAc. The aqueous layer was washed with EtOAc (3x5 mL) and the combined organics were dried over MgSO₄, filtered and the solvent removed in vacuo. The ¹H NMR data shows a gradual decrease in yield as the reaction progresses.

A reaction was ran after the pre-mixed reaction solution had been stored overnight and there was no significant drop in yield, suggesting the mixture does not decompose over time.
8 Safety Tests

(Aminooxy)-2-methylproanoic Acid Hydrochloride

This chemical was purchased from Fluorochem (Cat.No.447946, Lot:FCBO16818).
The heat of decomposition for this sample (Figure S2) is above the 800 J/g threshold value
which would indicate potential explosive properties. Thus, indicating that the material may
have explosive properties. Subsequent high-rate Carius tube test results indicated that the
material does not have explosive properties. Although not explosive this energetic material
could be hazardous depending on usage.

![Figure S2. DSC result for (Aminooxy)-2-methylproanoic acid hydrochloride](image)

2-[(Cyclobutylideneamino)oxy]-2-methylproanoic acid (1)

The heat of decomposition for this sample (Figure S3) is above the 800 J/g threshold value
which would indicate potential explosive properties. Thus, indicating that the material may
have explosive properties. Subsequent high-rate Carius tube test results indicated that the
material does not have explosive properties. Although not explosive this energetic material could be hazardous depending on usage.

**Figure S3.** DSC result for 2-[(cyclobutylideneamino)oxy]-2-methylpropanoic acid 1.
NMR Spectra

S2 $^1$H NMR (400 MHz, CDCl$_3$)

S2 $^{13}$C NMR (101 MHz, CDCl$_3$)
S5 $^{1}$H NMR (400 MHz, CDCl$_3$)

S5 $^{13}$C NMR (101 MHz, CDCl$_3$)
S8 $^1$H NMR (400 MHz, CDCl$_3$)

S8 $^{13}$C NMR (101 MHz, CDCl$_3$)
44 $^1$H NMR (400 MHz, CDCl$_3$)

44 $^{13}$C NMR (101 MHz, CDCl$_3$)
4 $^1$H NMR (400 MHz, CDCl$_3$)

4 $^{13}$C NMR (126 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

$^1$H NMR (101 MHz, CDCl$_3$)
10 $^1$H NMR (400 MHz, CDCl$_3$)

$^1$H NMR spectra

10 $^{13}$C NMR (126MHz, CDCl$_3$)

$^{13}$C NMR spectra
$^{13}$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (126 MHz, CDCl$_3$)
$^{19}$F NMR (471 MHz, CDCl$_3$)
$^{14}$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (126 MHz, CDCl$_3$)
$^{16}\text{H NMR (400 MHz, CDCl}_3\text{)}$

$^{13}\text{C NMR (126 MHz, CDCl}_3\text{)}$
$^{17}$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (126 MHz, CDCl$_3$)
$^{17}$F NMR (471 MHz, CDCl$_3$)
21 $^1$H NMR (500 MHz, CDCl$_3$)

21 $^{13}$C NMR (126 MHz, CDCl$_3$)
23 $^1$H NMR (400 MHz, CDCl$_3$)

23 $^{13}$C NMR (126 MHz, CDCl$_3$)
$^{24}$H NMR (400 MHz, CDCl$_3$)

$^{24}$C NMR (101 MHz, CDCl$_3$)
27 $^1$H NMR (400MHz, CDCl$_3$)

27 $^{13}$C NMR (101 MHz, CDCl$_3$)
30 $^1$H NMR (400 MHz, CDCl$_3$)

30 $^{13}$C NMR (126 MHz, CDCl$_3$)
$^{32}$H NMR (500 MHz, CDCl$_3$)

$^{32}$C NMR (126 MHz, CDCl$_3$)
35 \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3})

35 \textsuperscript{13}C NMR (126 MHz, CDCl\textsubscript{3})
36 $^1$H NMR (400 MHz, CDCl$_3$)

36 $^{13}$C NMR (126 MHz, CDCl$_3$)
$^{37}$H NMR (400 MHz, CDCl$_3$)

$^{37}$C NMR (126 MHz, CDCl$_3$)
$^{38}\text{H NMR (400 MHz, CDCl}_3\text{)}$

$^{38}\text{C NMR (126 MHz, CDCl}_3\text{)}$
39 $^1$H NMR (400 MHz, CDCl$_3$)

39 $^{13}$C NMR (126 MHz, CDCl$_3$)
$^{1}H$ NMR (400 MHz, CDCl$_3$)

$^{13}C$ NMR (101 MHz, CDCl$_3$)
45 $^1$H NMR (400 MHz, CDCl$_3$)

45 $^{13}$C NMR (101 MHz, CDCl$_3$)
46 $^1$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
$^{1}H$ NMR (400 MHz, CDCl$_3$)

$^{13}C$ NMR (101 MHz, CDCl$_3$)
53 $^1$H NMR (400 MHz, CDCl$_3$)

53 $^{13}$C NMR (101 MHz, CDCl$_3$)
56 ¹H NMR (400 MHz, CDCl₃)

56 ¹³C NMR (101 MHz, CDCl₃)
$^1$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
59 $^1$H NMR (400 MHz, CDCl$_3$)

59 $^{13}$C NMR (101 MHz, CDCl$_3$)
60 $^1$H NMR (400MHz, CDCl$_3$)

60 $^{13}$C NMR (101 MHz, CDCl$_3$)
10 References