Supplementary Material

A Visible Light Photoredox Catalysed Radical Pictet-Spengler Reaction

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1. General Information

All reagents were purchased from commercial suppliers and used without further purification. Proton NMR (\(^1\)H NMR) and carbon NMR (\(^{13}\)C NMR) were recorded on Bruker 400 Ultrashield (400 MHz) and Ascend 400 (400 MHz). Chemical shifts (\(\delta\)) are quoted in parts per million (ppm). Multiplicities are abbreviated as s, singlet; d, doublet; dd, doublet of doublets; t, triplet; q, quarter; m, multiplet; b, broad. Coupling constants (\(J\)) are reported in Hertz (Hz). Analytical thin layer chromatography (TLC) was carried out using aluminium-backed Merck Kieselgel KG60 F254 silica plates and visualised by exposure to UV light (254 nm). Flash chromatography was performed on Grace Davidson Davisil LC60A 40-63 micron silica gel. Yields refer to isolated yields, unless otherwise stated. After purification product purity was determined to be >95% by \(^1\)H NMR spectroscopy.

Mass spectrometric analyses were performed on a Thermo Scientific Q Exactive mass spectrometer fitted with a HESI-II ion source. Positive ion electrospray mass spectra were recorded in an appropriate mass range set for 140,000 mass resolution. Positive ion EI mass spectra were run on a Thermo Scientific DFS mass spectrometer using an ionisation energy of 70 eV. Accurate mass measurements were obtained with a resolution of 5000-10000 using PFK (perfluorokerosene) as the reference compound.

Photocatalytic reactions were performed in a custom made LED photoreactor as shown in Figure X. The photoreactor comprised of two plates of blue LEDs (7 × 1 W @ 447 nm, www.luzeonstar.com) with PC cooling fans and heat sinks for temperature control.
Figure S1. Custom-built LED photoreactor; a) blue LED assembly; b) PC cooling fans; c) heat sinks; d) intensity control; e) 12 V power supply.
2. Experimental Procedures

Synthesis of Phenylethylamines

\[
\begin{align*}
\text{BH}_3\text{-THF} & \quad \text{THF, reflux} \\
\text{CN} & \quad \text{NH}_2 \\
28: X = I & \quad 30: X = I \\
29: X = F & \quad 31: X = F
\end{align*}
\]

General Procedure

A solution of BH$_3$ in THF (1 M, 50.2 ml, 2.4 equiv.) was added dropwise, at 0 °C, to a solution of phenylacetonitrile (5.00 g, 1.0 equiv.) in anhydrous THF (25 ml). The reaction mixture was heated at 65 °C under inert atmosphere overnight. After cooling to r.t., the reaction mixture was diluted with methanol and concentrated under reduced pressure. The residue was dissolved in 6 M aqueous HCl solution and heated at reflux for 1 h. After cooling to r.t., the reaction was washed with diethyl ether (3×) and the combined aqueous layers were basified to pH 12 with 6 M aqueous NaOH solution. The mixture was extracted with CH$_2$Cl$_2$ (3×), washed with brine, and dried (MgSO$_4$). The solvent was removed under reduced pressure to give the desired product, which was used without further purification.

![2-Iodophenylethanalmine](image)

2-Iodophenylethanalmine (30): obtained as an orange oil (4.14 g, 81% yield)

$^1$H NMR (CDCl$_3$, 400 MHz): δ = 7.87 (dd, $J = 7.9$, 1.2 Hz, 1H), 7.33-7.29 (m, 2H), 7.26-7.24 (m, 2H), 6.95 (td, $J = 15.1$, 1.9, 1H), 3.00-2.97 (m, 2H), 2.93-2.89 (m, 2H), 1.29 (br s, 2H) $^{13}$C NMR (CDCl$_3$, 100 MHz): δ = 142.5, 139.7, 130.0, 128.4, 128.2, 100.9, 44.8, 42.5
Data match that reported in the literature.\textsuperscript{1}

\begin{center}
\includegraphics[width=0.2\textwidth]{image}
\end{center}

\textbf{2-Fluorophenylethanolamine (31):} obtained as a pale yellow oil (2.83 g, 92% yield)

\textbf{\textsuperscript{1}H NMR (CDCl\textsubscript{3}, 400 MHz):} \( \delta = 7.21\text{-}7.16 (m, 2H), 7.08\text{-}6.99 (m, 2H), 2.95 (t, J = 7.1 \text{ Hz}, 2H), 2.78 (t, J = 6.9 \text{ Hz}, 2H), 1.13 (br s, 2H) \)

\textbf{\textsuperscript{13}C NMR (CDCl\textsubscript{3}, 100 MHz):} \( \delta = 162.6 \text{ (d, } J_{C,F} = 244.7 \text{ Hz}), 131.2 \text{ (d, } J_{C,F} = 5.1 \text{ Hz}), 128.0 \text{ (d, } J_{C,F} = 7.9 \text{ Hz}), 126.9 \text{ (d, } J_{C,F} = 15.9 \text{ Hz}), 124.1 \text{ (d, } J_{C,F} = 3.4 \text{ Hz}), 115.5 \text{ (d, } J_{C,F} = 22.3 \text{ Hz}), 42.5, 33.7 \)

HRMS (ESI) \( m/z \) calc. for C\textsubscript{8}H\textsubscript{11}NF [M+H]\textsuperscript{+} 140.0870, found 140.0870

\textbf{Synthesis of 2-Iodophenylethanol Intermediates}

\textbf{2-Iodo-5-methoxyphenylethanol\textsuperscript{2}}

\begin{center}
\begin{tikzpicture}
\node[below=0.5cm] at (current bounding box.south) {32};
\node[below=0.5cm] at (current bounding box.south) {33};
\node[below=0.5cm] at (current bounding box.south) {32};
\end{tikzpicture}
\end{center}

A solution of 3-methoxyphenylacetic acid (3.00 g, 18.05 mmol, 1.00 equiv.) and N-iodosuccinimide (4.26 g, 18.96 mmol, 1.05 equiv.) in MeCN (24 ml) was stirred at room temperature overnight, whilst being protected from light. The reaction mixture was extracted with Et\textsubscript{2}O (3\times), washed with saturated aqueous NaS\textsubscript{2}O\textsubscript{3} solution and brine, and dried (MgSO\textsubscript{4}). The solvent was removed under reduced pressure to give 2-iodo-5-methoxyphenylacetic acid as an off-white solid, which was used without further purification.
A solution of BH$_3$ in THF (1 M, 2.00 equiv.) was added dropwise, at 0 °C, to a solution of 5-methoxy-2-iodophenylacetic acid (1.00 equiv.) in anhydrous THF. The mixture was heated at reflux under inert atmosphere overnight. The reaction was allowed to cool to room temperature, diluted with water and extracted with Et$_2$O (3×). The combined organic extracts were washed with brine and dried over anhydrous MgSO$_4$. The solvent was removed under reduced pressure and the residue was purified by flash chromatography on silica, eluting with 15-20% EtOAc/n-pentane to give 5-methoxy-2-iodophenyl ethanol.

2-(2-Iodo-5-methoxyphenyl)ethanol (33): obtained as a pale yellow oil (1.78 g, 95%)

$^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ = 7.69 (d, $J$ = 8.7 Hz, 1H), 6.84 (d, $J$ = 3.0 Hz, 1H), 6.55 (dd, $J$ = 8.7, 3.0 Hz, 1H), 3.68 (t, $J$ = 6.3 Hz, 2H), 3.78 (s, 3H), 2.99 (t, $J$ = 6.7 Hz, 2H)

$^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ = 160.1, 142.2, 140.2, 116.6, 114.4, 89.3, 62.4, 55.5, 43.9

2-Iodophenylethanol Intermediates with Electron Poor Substituents

General Procedure

A solution of phenylacetic acid (1.00 g, 1.00 equiv.), Pd(OAc)$_2$ (0.10 equiv.), PhI(OAc)$_2$ (0.75 equiv.), and I$_2$ (0.75 equiv.) in anhydrous DMF was stirred at 60 °C for 24 h. After cooling to room temperature, the reaction mixture was filtered through a plug of Celite with Et$_2$O. The filtrate was washed with saturated aqueous Na$_2$S$_2$O$_3$ solution, extracted with Et$_2$O (3×) and the combined organic extracts were washed with
brine and dried over MgSO₄. The solvent was removed under reduced pressure to give 2-iodophenylacetic acid, which was used without further purification.

A solution of BH₃ in THF (1 M, 2.00 equiv.) was added dropwise, at 0 °C, to a solution of 2-iodophenylacetic acid (1.00 equiv.) in anhydrous THF. The mixture was heated at reflux under inert atmosphere overnight. The reaction was allowed to cool to room temperature, diluted with water and extracted with Et₂O (3×). The combined organic extracts were washed with brine and dried over anhydrous MgSO₄. The solvent was removed under reduced pressure and the residue was purified by flash chromatography, eluting with 15-20% EtOAc/n-pentane to give the desired product.

**Characterisation Data**

![Structure of 2-(5-Bromo-2-iodophenyl)ethanol](image)

**2-(5-Bromo-2-iodophenyl)ethanol (38):** obtained as a white solid (1.15 g, 48% yield).

**¹H NMR (CDCl₃, 400 MHz):** δ = 7.69 (d, J = 8.4 Hz, 1H), 7.42 (d, J = 2.4 Hz, 1H), 7.08 (dd, J = 8.4, 2.4 Hz, 1H), 3.88 (q, J = 6.3 Hz, 2H), 3.00 (t, J = 6.6 Hz, 2H)

**¹³C NMR (CDCl₃, 100 MHz):** δ = 143.5, 140.9, 133.2, 131.6, 122.7, 98.8, 62.1, 43.6

Data match that reported in the literature.¹

![Structure of 2-(5-Chloro-2-iodophenyl)ethanol](image)

**2-(5-Chloro-2-iodophenyl)ethanol (39):** obtained as a white solid (0.66 g, 32% yield).

**¹H NMR (CDCl₃, 400 MHz):** δ = 7.74 (d, J = 8.4 Hz, 1H), 7.25 (d, J = 1.0 Hz, 1H) overlapping with CDCl₃, 6.93 (dd, J = 8.6, 2.5 Hz, 1H), 3.87 (m, 2H), 2.99 (t, J = 6.6
Hz, 2H). \[^{13}\text{C}\] NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta = 143.2, 140.6, 134.7, 130.4, 128.6, 97.9, 62.1, 43.6\). HRMS (EI) \(m/z\) calc. for C\textsubscript{8}H\textsubscript{8}ClIO 281.9308, found 281.9301

2-(4-Fluoro-2-iodophenyl)ethanol (40): obtained as a colourless solid (182.5 mg, 22 %). \(^1\text{H}\) NMR (400 MHz, CDCl\textsubscript{3}): \(\delta = 7.55 (\text{dd, } J = 2.6, 8.1 \text{ Hz, 1H}), 7.22 (\text{dd, } J = 6.0, 8.5 \text{ Hz, 1H}), 7.02 (\text{dt, } J = 2.6, 8.3 \text{ Hz, 1H}), 3.83 (\text{t, } J = 6.7 \text{ Hz, 2H}), 2.98 (\text{t, } J = 6.7 \text{ Hz, 2H})\) \[^{13}\text{C}\] NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta = 160.8 (\text{d, } J_{C:F} = 250.0 \text{ Hz}), 137.1 (\text{d, } J_{C:F} = 3.3 \text{ Hz}), 130.8 (\text{d, } J_{C:F} = 8.0 \text{ Hz}), 126.5 (\text{d, } J_{C:F} = 23.4 \text{ Hz}), 115.4 (\text{d, } J_{C:F} = 20.5 \text{ Hz}), 99.7 (\text{d, } J_{C:F} = 8.1 \text{ Hz}), 62.3 (\text{s}), 42.8 (\text{s})\).

2-(2-Iodo-5-methylphenyl)ethanol (41): obtained as a white solid (199.9 mg, 68 %). \(^1\text{H}\) NMR (400 MHz, CDCl\textsubscript{3}): \(\delta = 7.69 (\text{d, } J = 8.0 \text{ Hz, 1H}), 7.08 (\text{d, } J = 2.2 \text{ Hz, 1H}), 6.75 (\text{dd, } J = 2.1, 7.9 \text{ Hz, 1H}), 3.84 (\text{q, } J = 6.5 \text{ Hz, 2H}), 2.97 (\text{t, } J = 6.8 \text{ Hz, 2H}), 2.29 (\text{s, } 3\text{H})\) \[^{13}\text{C}\] NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta = 141.0, 139.6, 138.6, 131.6, 129.7, 96.9, 62.4, 43.8, 21.0\)

Synthesis of 2-Iodophenethyl 4-Methylbenzenesulfonate Intermediates\(^5\)

![Synthesis diagram](image)

General Procedure
p-Toluenesulfonyl chloride (1.6 equiv.) was added to a solution of 2-iodophenyl ethanol (1.0 equiv.) and triethylamine (3.7 equiv.) in anhydrous CH₂Cl₂ at 0 °C. Ice bath was removed, and the reaction mixture was stirred at room temperature overnight. It was diluted with CH₂Cl₂, washed with 2 M aqueous HCl solution, followed by saturated aqueous NaHCO₃ solution and brine, and dried (MgSO₄). The solvent was removed under reduced pressure and the residue was purified by flash chromatography, on silica, eluting with 5-10% EtOAc/n-pentane to give the desired 2-iodophenethyl 4-methylbenzenesulfonate.

Characterisation Data

![Chemical Structure of 2-Iodo-5-methoxyphenethyl 4-methylbenzenesulfonate](attachment:image1)

2-Iodo-5-methoxyphenethyl 4-methylbenzenesulfonate (42): obtained as a colourless oil (1.23 g, 49% yield) ⁱH NMR (CDCl₃, 400 MHz): δ = 7.71-7.67 (m, 2H), 7.59 (d, J = 8.7 Hz, 1H), 7.28-7.26 (m, 2H) overlapping with CDCl₃, 6.73 (d, J = 3.0 Hz, 1H), 6.53 (dd, J = 8.7, 3.0 Hz, 1H), 4.23-4.19 (m, 2H), 3.75 (s, 3H), 3.04 (t, J = 7.0 Hz, 2H), 2.43 (s, 3H) ¹³C NMR (CDCl₃, 100 MHz): δ =160.1, 144.8, 140.1, 139.9, 133.0, 130.0, 127.9, 116.6, 115.1, 88.7, 69.0, 55.5, 40.2, 21.8

HRMS (ESI) m/z calc. for C₁₆H₁₄O₄S [M+H]+ 432.9965, found 432.9959

![Chemical Structure of 5-Bromo-2-iodophenethyl 4-methylbenzenesulfonate](attachment:image2)

5-Bromo-2-iodophenethyl 4-methylbenzenesulfonate (43): obtained as a white solid (1.35 g, 87% yield) ⁱH NMR (CDCl₃, 400 MHz): δ = 7.70 (d, J = 8.3 Hz, 2H), 7.58 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.3 Hz, 2H), 7.25 (d, J = 2.4 Hz, 1H), 7.04 (dd, J = 8.4,
2.4 Hz, 1H), 4.24 (t, \( J = 6.7 \) Hz, 2H), 3.04 (t, \( J = 6.7 \) Hz, 2H), 2.43 (s, 3H) \(^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz)}: \delta = 144.9, 141.2, 140.9, 133.4, 132.8, 131.9, 129.9, 127.9, 122.7, 98.3, 68.5, 39.8, 21.8

HRMS (EI) \( m/z \) calc. for C\(_{15}\)H\(_{14}\)O\(_3\)BrIS 479.886, found 479.8895

5-Chloro-2-iodophenethyl 4-methylbenzenesulfonate (44): obtained as a pale yellow solid (0.77g, 76% yield) \(^1\text{H NMR (CDCl}_3, 400 \text{ MHz)}: \delta = 7.70-7.68 \text{ (m, 2H)}, 7.64 \text{ (d,} \( J = 8.4 \text{ Hz, 1H)}), 7.29 \text{ (m, 2H)}, 7.11 \text{ (d,} \( J = 2.5 \text{ Hz, 1H)}), 6.91 \text{ (dd,} \( J = 8.4, 2.5 \text{ Hz, 1H)}), 4.24 \text{ (t,} \( J = 6.7 \text{ Hz, 2H)}), 3.05 \text{ (t,} \( J = 6.7 \text{ Hz, 2H)}), 2.43 \text{ (s, 3H)} \(^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz):} \delta = 144.9, 140.9, 140.6, 134.8, 132.8, 130.6, 130.0, 129.0, 127.9, 97.3, 68.5, 39.9, 21.8

HRMS (EI) \( m/z \) calc. for C\(_{15}\)H\(_{14}\)O\(_3\)ClIS 435.9397, found 435.9388

4-Fluoro-2-iodophenethyl 4-methylbenzenesulfonate (45): solid (402.4 mg, 92%).

\(^1\text{H NMR (400 MHz, CDCl}_3\):} \delta = 7.62 - 7.71 \text{ (m, 2H)}, 7.40 - 7.46 \text{ (m, 1H)}, 7.23 - 7.30 \text{ (m, 2H)}, 7.13 \text{ (dd,} \( J = 5.9, 8.4 \text{ Hz, 1H)}), 6.96 \text{ (dt,} \( J = 2.6, 8.3 \text{ Hz, 1H)}), 4.19 \text{ (t,} \( J = 6.8 \text{ Hz, 2H)}), 3.04 \text{ (t,} \( J = 6.7 \text{ Hz, 2H)}), 2.43 \text{ (s, 3H)} \(^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz):} \delta = 161.01 \text{ (d,} \( J_{C,F} = 251.0 \text{ Hz)}), 144.87 \text{ (s)}, 134.88 \text{ (d,} \( J_{C,F} = 3.3 \text{ Hz)}), 132.72 \text{ (s)}, 131.26 \text{ (d,} \( J_{C,F} = 8.0 \text{ Hz)}), 129.88 \text{ (s)}, 127.89 \text{ (s)}, 126.40 \text{ (d,} \( J_{C,F} = 23.6 \text{ Hz)}), 115.52 \text{ (d,} \( J_{C,F} = 21.0 \text{ Hz)}), 99.23 \text{ (d,} \( J_{C,F} = 7.8 \text{ Hz)}), 68.89 \text{ (s)}, 62.27 \text{ (s)}, 42.73 \text{ (s)}, 39.03 \text{ (s)}, 21.72 \text{ (s).}
2-Iodo-5-methylphenethyl 4-methylbenzenesulfonate (46): solid (459.2 mg, 90%).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.65 - 7.72$ (m, 2H), 7.58 (d, $J = 8.0$ Hz, 1H), 7.23 – 7.31 (m, 2H), 6.97 (d, $J = 2.2$ Hz, 1H), 6.66 - 6.78 (m, 1H), 4.20 (t, 2H), 3.02 (t, 2H), 2.39 – 2.45 (br s, 3H), 2.24 (br s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 144.9, 139.5, 138.7, 131.7, 130.0, 128.0, 96.3, 69.3, 40.0, 21.9, 21.1$.

Synthesis of 2-Iodophenylethanamines$^4, 6$

![Chemical structure](image)

General Procedure

Sodium azide (2.0 equiv.) and sodium iodide (0.5 equiv.) were added to a solution of 2-iodophenylethylsulfonate (1.0 equiv.) in DMF at room temperature, under inert atmosphere. The mixture was heated at 80 °C overnight. The reaction was allowed to cool to room temperature and quenched with water. The mixture was extracted with Et$_2$O (3×), washed with brine and dried over MgSO$_4$. The in-situ azide was used in the next step without further purification.

A solution of azide (1.0 equiv.) and PPh$_3$ (1.5 equiv.) in Et$_2$O was stirred at 0 °C for 3 h. Water was added to the reaction mixture. The reaction was allowed to warm to room temperature and stirred overnight. It was then acidified with 1 M aqueous HCl solution and washed with Et$_2$O (3×). The aqueous layers were basified with 1 M aqueous NaOH.
solution, extracted with Et₂O (3×), washed with brine and dried over MgSO₄. The solvent was evaporated under reduced pressure to give 5-substituted-2-iodophenylethylamines.

**Characterisation Data**

![Structure](image)

**2-(2-Iodo-5-methoxyphenyl)ethanamine (47):** obtained as a yellow oil (1.09 g, 69% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 7.69 (d, J = 8.7 Hz, 1H), 6.80 (d, J = 3.0 Hz, 1H), 6.53 (dd, J = 8.8, 3.0 Hz, 1H), 3.77 (s, 3H), 2.97-2.93 (m, 2H), 2.85-2.82 (m, 2H), 1.29 (br s, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 160.1, 143.6, 140.1, 116.2, 114.1, 89.4, 55.5, 45.0, 42.5

HRMS (ESI) m/z calc. for C₉H₁₃ONI [M+H]^+ 278.0036, found 278.0034

![Structure](image)

**2-(5-Bromo-2-iodophenyl)ethanamine (48):** obtained as a white solid (0.57 g, 68% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 7.67 (d, J = 8.4 Hz, 1H), 7.36 (d, J = 2.4 Hz, 1H), 7.05 (dd, J = 8.4, 2.4 Hz, 1H), 2.97-2.93 (m, 2H), 2.85-2.82 (m, 2H), 1.16 (br s, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 144.7, 140.9, 132.8, 131.3, 122.7, 98.8, 44.6, 42.2

HRMS (ESI) m/z calc. for C₈H₁₀NBrI [M+H]^+ 325.9036, found 325.9030
2-(5-Chloro-2-iodophenyl)ethanamine (49): obtained as a white solid (0.28 g, 65% yield) $^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 7.74$ (d, $J = 8.4$ Hz, 1H), 7.21 (d, $J = 2.5$ Hz, 1H), 6.92 (dd, $J = 8.4$, 2.6 Hz, 1H), 2.97-2.93 (m, 2H), 2.85-2.82 (m, 2H), 1.26 (br s, 2H) $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 144.4$, 140.6, 134.6, 129.9, 128.4, 97.9, 44.7, 42.3

2-(4-Fluoro-2-iodophenyl)ethanamine (50): colourless oil (80.6 mg, 56 %) $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.52$ (dd, $J = 2.6$, 8.1 Hz, 1H), 7.14 (dd, $J = 6.0$, 8.5 Hz, 1H), 6.98 (dt, $J = 2.6$, 8.3 Hz, 1H), 2.77-2.94 (m, 4H). $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 160.6$ (d, $J_{C-F} = 249.7$ Hz), 138.3 (d, $J_{C-F} = 3.2$ Hz), 130.3 (d, $J_{C-F} = 7.9$ Hz), 126.3 (d, $J_{C-F} = 23.3$ Hz), 115.3 (d, $J_{C-F} = 20.6$ Hz), 99.7 (d, $J_{C-F} = 7.9$ Hz), 43.7 (s), 42.4 (s).

2-(2-Iodo-5-methylphenyl)ethanamine (51): colourless oil (141.2 mg, 74 %) $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.67$ (d, $J = 8.0$ Hz, 1 H), 7.03 (d, $J = 2.0$ Hz, 1 H), 6.73 (dd, $J = 1.9$, 7.9 Hz, 1 H), 2.98 - 2.89 (m, 2 H), 2.87 - 2.78 (m, 2 H), 2.28 (s, 3 H). $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 142.3$, 139.7, 138.7, 131.1, 129.5, 97.0, 44.5, 42.6, 21.2.
**General Procedure for the Preparation of Aldimines**

A solution of 2-iodophenylethamine (1.0 equiv.) and benzaldehyde (1.05 equiv.) in CH₂Cl₂ was stirred at room temperature overnight. The reaction was filtered through a plug of Celite with CH₂Cl₂. The solvent was then removed under reduced pressure and the residue was dried under vacuum to remove traces of benzaldehyde. The aldimines were used without further purification.

**Characterisation Data**

*N*-Phenethyl-1-phenylmethanimine (25): obtained as a yellow solid (0.68 g, 79% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 8.17 (s, 1H), 7.71-7.69 (m, 2H), 7.42-7.40 (m, 3H), 7.30-7.27 (m, 2H), 7.25-7.18 (m, 3H), 3.89 (td, J = 7.4, 1.2 Hz, 2H), 3.04 (t, J = 7.5 Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 161.6, 140.1, 136.3, 130.7, 129.2, 128.7, 128.5, 128.2, 126.2, 63.3, 37.6.

Data match that reported in the literature.⁷
**N-(2-Fluorophenethyl)-1-phenylmethanimine (26):** obtained as a brown oil (0.36 g, 87% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 8.19 (s, 1H), 7.71-7.69 (m, 2H), 7.42-7.39 (m, 3H), 7.25-7.16 (m, 2H), 7.07-7.00 (m, 2H), 3.88 (td, J = 7.4, 1.2 Hz, 2H), 3.08 (t, J = 7.4 Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 162.6 (d, J_C-F = 244.9 Hz), 161.7, 136.3, 131.6 (d, J_C-F = 4.8 Hz), 130.7, 128.7, 128.2, 128.1 (d, J_C-F = 8.0 Hz), 126.9 (d, J_C-F = 15.9 Hz), 124.0 (d, J_C-F = 3.3 Hz), 115.4 (d, J_C-F = 22.2 Hz), 61.6, 30.9

HRMS (ESI) m/z calc. for C₁₅H₁₅NF [M+H]⁺ 228.1183, found 228.1179

![Chemical Structure](image)

**N-(2-Iodophenethyl)-1-phenylmethanimine (5):** obtained as a yellow oil (0.62 g, 91% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 8.20 (s, 1H), 7.84 (d, J = 7.8 Hz, 1H), 7.73-7.70 (m, 2H), 7.43-7.39 (m, 3H), 7.25-7.24 (m, 2H), 6.91-6.87 (m, 1H), 3.87 (td, J = 7.6, 1.2 Hz, 2H), 3.16 (t, J = 7.5 Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 161.9, 142.6, 139.6, 136.3, 130.8, 130.6, 128.7, 128.4, 128.2, 100.9, 61.2, 42.2

HRMS (ESI) m/z calc. for C₁₅H₁₃NI [M+H]⁺ 336.0244, found 336.0239

![Chemical Structure](image)

**N-(2-Iodophenethyl)-1-(4-methoxyphenyl)methanimine (52):** obtained as an orange solid (0.27 g, 74% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 8.11 (s, 1H), 7.83 (d, J = 7.6 Hz, 1H), 7.69 (d, J = 8.7 Hz, 2H), 7.25-7.24 (m, 2H) overlapping with CDCl₃, 6.94-6.87 (d, J = 8.8 Hz, 2H) overlapping with 6.90 (m, 1H), 3.85 (s, 3H) overlapping with
3.84-3.80 (m, 2H), 3.15 (t, J = 7.4 Hz, 2H) \(^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz)}: \delta = 161.8, 161.3, 142.6, 139.6, 130.6, 129.9, 129.1, 128.4, 128.2, 114.1, 100.9, 61.2, 55.5, 42.3

HRMS (ESI) m/z calc. for C\(_{16}\)H\(_{17}\)ONI [M+H]\(^{+}\) 366.0349, found 366.0346

![](image)

1-(4-(tert-Butyl)phenyl)-N-(2-iodophenethyl)methanimine (53): 4-tert-butyl benzaldehyde (2.05 equiv.), obtained as a pale yellow solid (0.43 g, 90% yield) \(^{1}\text{H NMR (CDCl}_3, 400 \text{ MHz)}: \delta = 8.18 (s, 1H), 7.84 (d, J = 7.8 Hz, 1H), 7.66-7.63 (m, 2H), 7.44-7.41 (m, 2H), 7.25-7.24 (m, 2H) overlapping with CDCl\(_3\), 6.90-6.87 (m, 1H), 3.84 (td, J = 7.5, 1.2 Hz, 2H), 3.14 (t, J = 7.5 Hz, 2H), 1.33 (s, 9H) \(^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz)}: \delta = 161.7, 154.1, 142.6, 139.6, 133.6, 130.6, 128.3, 128.1, 128.0, 125.7, 100.9, 61.5, 42.3, 35.0, 31.4

HRMS (ESI) m/z calc. for C\(_{19}\)H\(_{23}\)NI [M+H]\(^{+}\) 392.0870, found 432.9966

![](image)

\(N\)-(2-Iodophenethyl)-1-(p-tolyl)methanimine (54): obtained as an off white solid (0.30 g, 82% yield) \(^{1}\text{H NMR (CDCl}_3, 400 \text{ MHz)}: \delta = 8.16 (s, 1H), 7.83 (d, J = 7.7 Hz, 1H), 7.61 (d, J = 8.1 Hz, 2H), 7.25-7.20 (m, 4H), 6.91-6.86 (m, 1H), 3.84 (td, J = 7.1, 1.2 Hz, 2H), 3.15 (t, J = 7.4 Hz, 2H), 2.38 (s, 3H) \(^{13}\text{C NMR (CDCl}_3, 100 \text{ MHz)}: \delta = 161.8, 142.7, 141.0, 139.6, 133.7, 130.6, 129.4, 128.3, 128.2, 128.2, 100.9, 61.4, 42.3, 21.6

S16
1-(4-chlorophenyl)-N-(2-iodophenethyl)methanimine (55): 4-chlorobenzaldehyde (1.15 equiv.), obtained as a dark yellow solid (0.32 g, 76% yield) $^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 8.15$ (s, 1H), 7.84 (d, $J = 7.8$ Hz, 1H), 7.66-7.63 (m, 2H), 7.39-7.37 (m, 2H), 7.25-7.22 (m, 2H) overlapping with CDCl$_3$, 6.91-6.87 (m, 1H), 3.86 (td, $J = 7.4$, 1.2 Hz, 2H), 3.15 (t, $J = 7.4$ Hz, 2H) $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 160.5$, 142.5, 139.6, 136.7, 134.7, 130.6, 129.4, 129.0, 128.4, 128.2, 120.9, 61.4, 42.1

HRMS (ESI) m/z calc. for C$_{16}$H$_{17}$NI [M+H]$^+$ 350.0400, found 350.0394

$\text{H} \text{N} \text{R} \text{M}$

$\text{H} \text{N} \text{R} \text{M}$

$\text{H} \text{N} \text{R} \text{M}$

$\text{H} \text{N} \text{R} \text{M}$

$\text{H} \text{N} \text{R} \text{M}$

1-(4-chlorophenyl)-N-(2-iodophenethyl)methanimine (56): 4-(trifluoromethoxy)benzaldehyde (2.05 equiv.), obtained as a yellow solid (0.38 g, 86% yield) $^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 8.18$ (s, 1H), 7.84 (d, $J = 8.3$ Hz, 1H), 7.76-7.73 (m, 2H), 7.25-7.24 (m, 4H) overlapping with CDCl$_3$, 6.92-6.88 (m, 1H), 3.87 (td, $J = 7.4$, 1.1 Hz, 2H), 3.15 (t, $J = 7.4$ Hz, 2H) $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 160.1$, 150.9, 142.4, 139.7, 134.8, 130.6, 129.7, 128.4 (d, $J_{C-F} = 12.6$ Hz), 121.8 (q, $J_{C-F} = 258.2$ Hz), 121.0, 100.9, 61.4, 42.1.

HRMS (ESI) m/z calc. for C$_{16}$H$_{14}$ONF$_3$I [M+H]$^+$ 420.0067, found 420.0069
**N-(2-Iodophenethyl)-1-(4-(trifluoromethyl)phenyl)methanimine (57):** 4-(trifluoromethyl)-benzaldehyde (2.05 equiv.), obtained as a pale yellow oil (0.40 g, 94% yield)

**1H NMR (CDCl$_3$, 400 MHz):** δ = 8.22 (s, 1H), 7.84-7.81 (m, 3H), 7.67 (d, J = 8.2 Hz, 2H), 7.25-7.24 (m, 2H), 6.92-6.88 (m, 1H), 3.90 (td, J = 7.3, 1.2 Hz, 2H), 3.17 (t, J = 7.4 Hz, 2H)

**13C NMR (CDCl$_3$, 100 MHz):** δ = 160.4, 142.3, 139.7, 139.4, 132.4 (q, J$_{C-F}$ = 32.6 Hz), 130.6, 128.4, 128.3 (q, J$_{C-F}$ = 8.8 Hz), 125.7 (q, J$_{C-F}$ = 3.8 Hz), 125.4 (q, J$_{C-F}$ = 272.4 Hz), 100.9, 61.4, 42.0

HRMS (ESI) m/z calc. for C$_{16}$H$_{14}$NF$_3$I [M+H]$^+$ 404.0118, found 404.0119

**N-(2-Iodo-5-methoxyphenethyl)-1-phenylmethanimine (58):** obtained as a yellow solid (0.14 g, 70% yield)

**1H NMR (CDCl$_3$, 400 MHz):** δ = 8.20 (s, 1H), 7.73-7.70 (m, 2H), 7.69 (d, J = 8.7 Hz, 1H), 7.43-7.39 (m, 3H), 6.83 (d, J = 3.0 Hz, 1H), 6.53 (dd, J = 8.7, 3.0 Hz, 1H), 3.86 (td, J = 7.4, 1.2 Hz, 2H), 3.71 (s, 3H), 3.12 (t, J = 7.4 Hz, 2H)

**13C NMR (CDCl$_3$, 100 MHz):** δ = 161.9, 159.9, 143.6, 139.9, 136.3, 130.8, 128.7, 128.2, 116.3, 114.5, 89.3, 61.4, 55.4, 42.3

HRMS (ESI) m/z calc. for C$_{16}$H$_{17}$ONI [M+H]$^+$ 366.0349, found 366.0348
**N-(5-Bromo-2-iodophenethyl)-1-phenylmethanimine (59):** obtained as a yellow solid (0.16 g; 82% yield) ¹H NMR (CDCl₃, 400 MHz): \( \delta = 8.23 \) (s, 1H), 7.73-7.71 (m-2H), 7.67 (d, \( J = 3.2 \) Hz, 1H), 7.43-7.41 (m, 4H), 7.05 (dd, \( J = 8.4, 2.4 \) Hz, 1H), 3.85 (td, \( J = 7.2, 1.2 \) Hz, 2H), 3.11 (t, \( J = 7.4 \) Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): \( \delta = 162.1, 144.8, 140.8, 136.2, 133.4, 131.3, 130.9, 128.7, 128.3, 122.6, 98.8, 61.1, 42.1 \)

HRMS (ESI) \( m/z \) calc. for C₁₅H₁₄NBrI [M+H]⁺ 413.9349, found 413.9348

**N-(5-Bromo-2-iodophenethyl)-1-(4-methoxyphenyl)methanimine (60):** obtained as a yellow solid (0.16 g; 80% yield) ¹H NMR (CDCl₃, 400 MHz): \( \delta = 8.16 \) (s, 1H), 7.67-7.65 (m, 3H), 7.41 (d, \( J = 2.4 \) Hz, 1H), 7.04 (dd, \( J = 8.5, 2.4 \) Hz, 1H), 6.94-6.92 (m, 2H), 3.85 (s, 3H), 3.80 (td, \( J = 7.4, 1.0 \) Hz, 2H), 3.10 (t, \( J = 7.4 \) Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): \( \delta = 161.8, 161.4, 145.0, 140.8, 133.3, 131.2, 129.8, 129.1, 122.5, 114.1, 98.8, 61.0, 55.5, 42.2 \)

HRMS (ESI) \( m/z \) calc. for C₁₆H₁₆ONBrI [M+H]⁺ 443.9454, found 443.9456
**N-(5-Bromo-2-iodophenethyl)-1-(4-chlorophenyl)methanimine (61):** obtained as an off white solid (0.18 g, 86% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 8.17 (s, 1H), 7.67-7.64 (m, 3H), 7.40-7.38 (m, 3H), 7.05 (dd, J = 8.4, 2.4 Hz, 1H), 3.84 (td, J = 7.3, 1.3 Hz, 2H), 3.11 (t, J = 7.3 Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 160.7, 144.7, 140.8, 136.8, 134.6, 133.4, 131.3, 129.5, 129.0, 122.6, 98.8, 61.0, 41.9

HRMS (ESI) m/z calc. for C₁₅H₁₃NBrClI [M+H]^⁺ 447.8959, found 447.8962

**N-(5-Chloro-2-iodophenethyl)-1-phenylmethanimine (62):** obtained as an off-white solid (0.16 g, 81% yield) ¹H NMR (CDCl₃, 400 MHz): δ = 8.24 (s, 1H), 7.74-7.71 (m, 3H), 7.43-7.40 (m, 3H), 7.27 (d, J = 2.6 Hz, 1H) overlapping with CDCl₃, 6.92 (dd, J = 8.5, 2.5 Hz, 1H), 3.85 (td, J = 7.4, 1.2 Hz, 2H), 3.12 (t, J = 7.4 Hz, 2H) ¹³C NMR (CDCl₃, 100 MHz): δ = 162.1, 144.5, 140.5, 136.1, 134.5, 130.9, 130.5, 128.7, 128.4, 128.3, 97.9, 61.1, 42.1

HRMS (ESI) m/z calc. for C₁₅H₁₄NCII [M+H]^⁺ 369.9854, found 369.9851
**N-(5-Chloro-2-iodophenethyl)-1-(4-methoxyphenyl)methanimine (63):** obtained as a pale yellow solid (0.17 g, 86% yield)

**\(^1\)H NMR (CDCl\(_3\), 400 MHz):** \(\delta = 8.16\) (s, 1H), 7.73 (d, \(J = 8.4\) Hz, 1H), 7.67-7.65 (m, 2H), 7.26 (m, 1H) overlapping with CDCl\(_3\), 6.94-6.88 (m, 3H), 3.85 (s, 3H), 3.81 (td, \(J = 7.4\), 1.2 Hz, 2H), 3.10 (t, \(J = 7.4\) Hz, 2H)

**\(^{13}\)C NMR (CDCl\(_3\), 100 MHz):** \(\delta = 161.8, 161.3, 144.6, 140.5, 134.5, 130.4, 129.8, 129.1, 128.3, 114.1, 97.9, 61.0, 55.5, 42.3\)

HRMS (ESI) m/z calc. for C\(_{16}\)H\(_{16}\)ONClI [M+H]\(^+\) 399.9960, found 399.9958

**N-(5-Chloro-2-iodophenethyl)-1-(4-chlorophenyl)methanimine (64):** obtained as a white solid (0.12 g, 87% yield)

**\(^1\)H NMR (CDCl\(_3\), 400 MHz):** \(\delta = 8.18\) (s, 1H), 7.74 (d, \(J = 8.4\) Hz, 1H), 7.66-7.64 (m, 2H), 7.40-7.38 (m, 2H), 7.25 (d, \(J = 2.5\) Hz, 1H), 6.92 (dd, \(J = 8.4\), 2.6 Hz, 1H), 3.85 (td, \(J = 7.3\), 1.2 Hz, 2H), 3.11 (t, \(J = 7.3\) Hz, 2H)

**\(^{13}\)C NMR (CDCl\(_3\), 100 MHz):** \(\delta = 160.7, 144.4, 140.5, 136.8, 134.6, 134.5, 130.5, 129.5, 129.0, 128.4, 97.9, 61.0, 42.0\)

HRMS (ESI) m/z calc. for C\(_{15}\)H\(_{13}\)NCl\(_2\)I [M+H]\(^+\) 403.9464, found 403.9464
General Procedure for the Preparation of Ketimines

Benzophenone imine (558 mg, 3.1 mmol) was added to a solution of 2-(2-iodophenyl)ethylamine (760 mg, 3.1 mmol) in dichloromethane (5.5 mL). 3A molecular sieves (760 mg) were added to the solution, and it was stirred at room temperature overnight. The mixture was filtered through a pad of Celite with dichloromethane and concentrated under reduced pressure. The crude reaction mixture was purified under reduced pressure using bulb-to-bulb distillation under reduced pressure.

Characterisation Data

N-(Diphenylmethylene)-2-(2-iodophenyl)ethanamine (65) : brown oil (1.6 g, 63 %).

1H NMR (400 MHz, CDCl3): δ = 7.69 - 7.80 (m, 1H), 7.53 - 7.65 (m, 2H), 7.27 - 7.53 (m, 6H), 7.17 - 7.24 (m, 2H), 6.93 - 7.01 (m, 2H), 6.81 - 6.90 (m, 1H), 3.66 (t, J = 7.24 Hz, 2H), 3.13 (t, J = 7.24 Hz, 2H).

13C NMR (CDCl3, 100 MHz): δ = 168.9, 142.8, 139.8, 139.4, 136.7, 130.6, 129.9, 128.5, 128.4, 128.4, 128.3, 128.1, 128.1, 127.9, 127.7, 100.9, 53.6, 42.1.
HRMS (ESI) m/z calc. for C$_{21}$H$_{18}$IN [M+H]$^+$ 412.0517, found 412.0544.

![Structural formula of N-(Diphenylmethylene)-2-(4-fluoro-2-iodophenyl)ethanamine](image1)

**N-(Diphenylmethylene)-2-(4-fluoro-2-iodophenyl)ethanamine (66):** (71.9 mg, 72%)

$^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ = 7.53 - 7.74 (m, 2H), 7.30 - 7.52 (m, 9H), 7.17 (dd, $J$ = 5.97, 8.51 Hz, 1H), 6.92 - 7.01 (m, 1H), 3.64 (t, $J$ = 7.14 Hz, 2H), 3.10 (t, $J$ = 7.04 Hz, 2H).

$^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ = 178.44 (s), 169.02 (s), 160.54 (d, $J_{C-F}$ = 249.5 Hz), 139.74 (s), 138.82 (d, $J_{C-F}$ = 3.3 Hz), 136.69 (s), 130.96 (d, $J_{C-F}$ = 7.8 Hz), 130.08 (s), 128.84 (s), 128.57 (s), 128.45 (s), 128.41 (s), 128.15 (s), 127.76 (s), 127.34 (s), 127.25 (s), 126.02 (d, $J_{C-F}$ = 23.5 Hz), 115.10 (d, $J_{C-F}$ = 20.6 Hz), 99.73 (d, $J_{C-F}$ = 7.9 Hz), 53.59 (s), 41.15 (s).

![Structural formula of N-(Diphenylmethylene)-2-(2-iodo-5-methylphenyl)ethanamine](image2)

**N-(Diphenylmethylene)-2-(2-iodo-5-methylphenyl)ethanamine (67):** (71.3 mg, 70%)

$^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ = 7.33 - 7.63 (m, 11H), 7.04 (d, $J$ = 2.15 Hz, 1H), 6.69 (dd, $J$ = 2.15, 8.02 Hz, 1H), 3.66 (t, $J$ = 7.24 Hz, 2H), 3.09 (t, $J$ = 7.24 Hz, 2H), 2.23 (s, 3H).

$^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ = 178.0, 168.4, 142.1, 139.4, 138.6, 137.5, 136.3, 131.1, 129.5, 128.5, 128.4, 128.0, 128.0, 127.8, 127.6, 127.4, 126.8, 96.4, 53.3, 41.6, 20.5.

![Structural formula of 2-(5-Bromo-2-iodophenyl)-N-(diphenylmethylene)ethanamine](image3)

**2-(5-Bromo-2-iodophenyl)-N-(diphenylmethylene)ethanamine (68):** (47.9 mg, 72%)

$^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ = 7.55 - 7.72 (m, 11H), 6.96 - 7.04 (m, 3H), 3.65
(t, J = 7.14 Hz, 2H), 3.09 (t, J = 7.14 Hz, 2H). $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 177.9$, 144.7, 139.3, 139.6, 132.7, 130.2, 129.6, 127.6, 121.2, 98.2, 52.1, 41.5. HRMS (ESI) m/z calc. for C$_{21}$H$_{17}$BrIN [M+H]$^+$ 489.9679, found 489.9651

N-(Diphenylmethylene)-2-(2-iodo-5-methoxyphenyl)ethanamine (69): (66 %) $^1$H NMR (CDCl$_3$, 400 MHz): $\delta = 7.58 - 7.64$ (m, 2H), 7.29 - 7.49 (m, 9H), 6.92 - 6.99 (m, 1H), 6.79 (d, J = 3.13 Hz, 1H), 6.48 (dd, J = 2.93, 8.61 Hz, 1H), 3.68 - 3.75 (m, 3H), 3.04 - 3.12 (m, 2H). $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta = 168.7$, 159.6, 143.7, 139.6, 139.5, 136.5, 129.8, 128.3, 127.9, 127.6, 116.2, 114.0, 89.3, 55.1, 53.4, 42.1 HRMS (ESI) m/z calc. for C$_{22}$H$_{20}$INO [M+H]$^+$ 442.0623, found 442.0662.

**Photoredox Cyclisation**

![Diagram of reaction](image)

**General Procedure for the Cyclisation of Aldimines**

A solution of the aldimine (67 mg, 0.2 mmol), fac-Ir(ppy)$_3$ (3.3 mg, 5.0 x 10$^{-3}$ mmol), DIPEA (0.8 mmol, 140 µL), in DMSO (2.00 mL) was freeze-pump-thaw degassed under inert atmosphere. The reaction was stirred at room temperature under irradiation with 10 W blue LED light for 16 h. Internal standard (sulfolene, 23.6 mg, 0.2 mmol) was added and analysed by $^1$H NMR spectroscopy.
General Procedure for the Cyclisation of Ketimines

A solution of ketimine (0.2 mmol), \( \text{fac-Ir(ppy)}_3 \) (3.3 mg, \( 5.0 \times 10^{-3} \) mmol), DIPEA (0.4 mmol, 70 μL), in DMSO (2.0 mL) was freeze-pump-thaw degassed under inert atmosphere. The reaction was stirred at room temperature under irradiation with 10 W blue LED light for 16 h. Internal standard (sulfolene, 23.6 mg, 0.2 mmol) was added and the reaction mixture was analysed directly by \(^1\text{H} \) NMR spectroscopy.

Representative \(^1\text{H} \) NMR spectrum:

Figure S2. \(^1\text{H} \) NMR spectra (DMSO-\( d_6 \)) of reaction mixture of THIQ (blue) with sulfolene (red) as internal standard highlighting the resonances used for integration
Photoredox Dimerisation

\[
\begin{align*}
5 \quad (X = I) \\
26 \quad (X = F)
\end{align*}
\]

General Procedure

A solution of imine (0.2 mmol), photocatalyst (2.0 \times 10^{-3} \text{ mmol}), base (0.24 mmol), in solvent (2.00 mL) was freeze-pump-thaw degassed under inert atmosphere. The reaction was stirred at room temperature under irradiation with 10 W blue LED light for 16 h. Internal standard (sulfolene, 23.6 mg, 0.2 mmol) was added and analysed by \textsuperscript{1}H NMR spectroscopy.

3. References

4. $^1$H NMR and $^{13}$C NMR spectra

![1H NMR spectrum](image1)

![13C NMR spectrum](image2)

**CDCl$_3$, 400 MHz**

![1H NMR spectrum](image3)

![13C NMR spectrum](image4)

**CDCl$_3$, 100 MHz**
CDCl$_3$, 400 MHz

CDCl$_3$, 100 MHz
$^{1}H$ NMR spectra of compound 49 in CDCl$_3$, 400 MHz and 100 MHz.
50

CDCl₃, 400 MHz

50

CDCl₃, 100 MHz
CDCl$_3$, 400 MHz

51

CDCl$_3$, 100 MHz

51
$^1$H NMR spectra of compound 26 in CDCl$_3$, 400 MHz and 100 MHz.

**CDCl$_3$, 400 MHz**

**CDCl$_3$, 100 MHz**

*S45*
CDCl₃, 400 MHz

CDCl₃, 100 MHz

60

OCH₃
**Compound 65**

**CDCl₃, 400 MHz**

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**CDCl₃, 100 MHz**

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S60