Supporting Information
for DOI: 10.1055/s-0036-1589119
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1. General Methods

Reagents and solvents were purchased from Acros Organics, Alfa Aesar GmbH & Co. KG, Fisher Scientific, FluoroChem, Merck KGAA and Sigma Aldrich GmbH. Styrene was distilled prior to use by a Büchi GKR-50 Kugelrohr apparatus. All other reagents obtained from commercial sources were used as received unless mentioned otherwise.

\(^1\)H, \(^{13}\)C and \(^{19}\)F NMR spectra were recorded at 298 K on a Bruker DPX 300, Bruker DPX 400, a Bruker DPX 500 or a Bruker DPX 600. All resonances are reported relative to TMS. Spectra were calibrated relative to solvents’ residual proton and carbon chemical shifts: CHCl\(_3\) (\(\delta = 7.26\) ppm for \(^1\)H NMR and \(\delta = 77.16\) for \(^{13}\)C NMR). Coupling constants (\(J\)) are reported in Hertz (Hz). The multiplicity of the signals are given as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad), dsept (doublet of septets) and td (triplet of doublets).

Thin layer chromatography (TLC) was performed to monitor the reactions using precoated aluminium sheets of Merck silica gel 60 F254 (0.20 m), and detection of compounds was done under UV light (254 nm) or dipping into a solution of KMnO\(_4\) (1.5 g in 200 mL H\(_2\)O, 5 g NaHCO\(_3\)) or Ekkert’s reagent (50 mL glacial acetic acid, 1 mL conc. sulfuric acid, 0.5 mL \(p\)-anisaldehyde).

Flash column chromatography was performed using Merck silica gel 60 (40-63 μm) to purify products applying nitrogen pressure of about 0.2 bar or on a Biotage Isolera Four using Biotage cartridges SNAP Ultra 10 g. The solvents were used as laboratory grade.

GC-MS (EI, 70eV) was recorded on a combined set up of an Agilent 6890N chromatograph equipped with a DB-35ms column (30 m x 0.250 μm, film thickness 0.15 μm) using H\(_2\) (~1 bar) as carrier gas and a Waters GCT premier TOF mass spectrometer.

HRMS were measured by the EPSRC Mass Spectrometry Facility in Swansea University on a Waters Xevo G2-S and on a Thermo Scientific LTQ Orbitrap XL or at Cardiff University on a Waters LCT Premier XE. Ions were generated by Atmospheric Pressure Ionisation (APCI), Electrospray Ionisation (ESI), Nanospray Ionisation (NSI), Electron Ionisation (EI) or Atmospheric Sample Analysis Probe (ASAP).

Melting points (m.p.) were determined with a Gallenkamp variable heater and are not corrected.

Infrared spectra (IR) were recorded on a Shimadzu FTIR Affinity-1S spectrometer.
2. General procedure (GP) for the iodoacetoxylation

A suspension of ground iodine (127 mg, 0.500 mmol) and ground Oxone (307 mg, 1.00 mmol, 2.0 equiv.) in acetic anhydride (1.5 mL, 32 mmol, 64 equiv.) and glacial acetic acid (3.0 mL, 52 mmol, 104 equiv.) was stirred for 21 h at 50 °C in the absence of light. Afterwards, the alkene or alkyne (0.8 mmol, 1.6 equiv.) was added to the reaction mixture and stirred for further 2 h at room temperature. Next, the reaction mixture was quenched with sat. aq. solution of NaHCO₃ (40 mL) and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with sat. aq. solution of Na₂S₂O₃ (5 mL) and brine (20 mL), dried over MgSO₄ and concentrated in vacuo. The crude mixture was purified by flash column chromatography.

3. Optimisation

Table S1: Optimisation of the reaction conditions for the iodoacetoxylation of 1a.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ac₂O [mL]</th>
<th>AcOH [mL]</th>
<th>Equiv. of Oxone</th>
<th>Reaction time t₁</th>
<th>Reaction time t₂</th>
<th>Yield of 2a (%)&lt;sup&gt;[a]&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.0</td>
<td>0.2</td>
<td>1.0</td>
<td>4 days</td>
<td>50 min</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>5.0</td>
<td>0.2</td>
<td>2.0</td>
<td>4 days</td>
<td>50 min</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>5.0</td>
<td>0.2</td>
<td>3.0</td>
<td>4 days</td>
<td>50 min</td>
<td>77</td>
</tr>
<tr>
<td>4</td>
<td>5.0</td>
<td>0.2</td>
<td>3.5</td>
<td>4 days</td>
<td>50 min</td>
<td>64</td>
</tr>
<tr>
<td>5</td>
<td>5.0</td>
<td>0.2</td>
<td>4.0</td>
<td>4 days</td>
<td>50 min</td>
<td>58</td>
</tr>
<tr>
<td>6</td>
<td>5.0</td>
<td>0.2</td>
<td>3.0</td>
<td>3 days</td>
<td>50 min</td>
<td>76 (77)</td>
</tr>
<tr>
<td>7</td>
<td>5.0</td>
<td>0.2</td>
<td>3.0</td>
<td>2 days</td>
<td>50 min</td>
<td>57 (65)</td>
</tr>
<tr>
<td>8</td>
<td>5.0</td>
<td>0.2</td>
<td>3.0</td>
<td>1 day</td>
<td>50 min</td>
<td>50 (48)</td>
</tr>
<tr>
<td>9</td>
<td>0.0</td>
<td>3.0</td>
<td>3.0</td>
<td>4 days</td>
<td>50 min</td>
<td>0&lt;sup&gt;[b]&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>1.5</td>
<td>3.0</td>
<td>3.0</td>
<td>21 hrs</td>
<td>50 min</td>
<td>95\textsuperscript{[b]}</td>
</tr>
<tr>
<td>---</td>
<td>-----</td>
<td>-----</td>
<td>------</td>
<td>--------</td>
<td>--------</td>
<td>----------------</td>
</tr>
<tr>
<td>10</td>
<td>1.5</td>
<td>3.0</td>
<td>3.0</td>
<td>21 hrs</td>
<td>50 min</td>
<td>89\textsuperscript{[b]}</td>
</tr>
<tr>
<td>11</td>
<td>1.5</td>
<td>3.0</td>
<td>3.0</td>
<td>7 hrs</td>
<td>50 min</td>
<td>(70)</td>
</tr>
<tr>
<td>12</td>
<td>1.5</td>
<td>3.0</td>
<td>3.0</td>
<td>21 hrs</td>
<td>10 min</td>
<td>(76)</td>
</tr>
<tr>
<td>13</td>
<td>1.5</td>
<td>3.0</td>
<td>3.0</td>
<td>21 hrs</td>
<td>120 min</td>
<td>(71)</td>
</tr>
<tr>
<td>14</td>
<td>1.5</td>
<td>3.0</td>
<td>3.5</td>
<td>21 hrs</td>
<td>120 min</td>
<td>(75)</td>
</tr>
<tr>
<td>15</td>
<td>1.5</td>
<td>3.0</td>
<td>2.5</td>
<td>21 hrs</td>
<td>120 min</td>
<td>(84)</td>
</tr>
<tr>
<td>16</td>
<td>1.5</td>
<td>3.0</td>
<td>2.0</td>
<td>21 hrs</td>
<td>120 min</td>
<td>(52)</td>
</tr>
<tr>
<td>17</td>
<td>1.5</td>
<td>3.0</td>
<td>1.5</td>
<td>21 hrs</td>
<td>120 min</td>
<td>(41)</td>
</tr>
<tr>
<td>18</td>
<td>1.5</td>
<td>3.0</td>
<td>1.0</td>
<td>21 hrs</td>
<td>120 min</td>
<td>(41)</td>
</tr>
</tbody>
</table>

\textsuperscript{[a]} Yield determined via \textsuperscript{1}H-NMR using 1,2,4,5-tetrachlorobenzene as internal standard; isolated yield in parentheses. \textsuperscript{[b]} Yield determined via GC-MS analysis.

4. Characterisation of products 2a-8e

2-Iodo-1-phenylethyl acetate (2a):

Styrene (114 \textmu L, 1.00 mmol, 1.6 equiv.) was reacted according to GP to give 2a as colourless oil in 84\% yield (244 mg, 0.840 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

\textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}): \( \delta = 7.37-7.33 \) (m, 5H, 5xCH\textsubscript{arom}), 5.88 (dd, \( J = 7.5, 5.8 \) Hz, 1H, CHOAc), 3.52-3.48 (m, 2H, CH\textsubscript{2}I), 2.14 (s, 3H, CH\textsubscript{3}) ppm. \textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{3}): \( \delta = 170.0, 138.5, 128.9, 128.8, 126.5, 75.3, 21.2, 8.0 \) ppm. The spectroscopic data are in agreement with the literature.\textsuperscript{1}

1-([1,1’-Biphenyl]-4-yl)-2-idoethyl acetate (2b)

4-Vinylbiphenyl (144 mg, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2b as colourless oil in 79\% yield (231 mg, 0.630 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).
$^1$H NMR (300 MHz, CDCl$_3$): $\delta = 7.62$-$7.58$ (m, 4H, 4xC$_{arom}$), 7.49-$7.34$ (m, 5H, 5xC$_{arom}$), 5.94 (dd, $J = 5.8$, 5.7 Hz, 1H, CHOAc), 3.57-$3.47$ (m, 2H, CH$_2$I), 2.17 (s, 3H, CH$_3$) ppm.

$^{13}$C NMR (75 MHz, CDCl$_3$): $\delta = 170.0$, 141.8, 140.6, 137.5, 129.0, 127.7, 127.6, 127.2, 127.0, 75.1, 21.2, 7.8 ppm. The spectroscopic data are in agreement with the literature.$^1$

2-Iodo-1-(naphthalen-2-yl)ethyl acetate (2c)

2-Vinyl naphthalene (123 mg, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2c as colourless oil in 74% yield (201 mg, 0.590 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta = 7.87$-$7.83$ (m, 4H, 4xC$_{arom}$), 7.54-$7.48$ (m, 2H, 2xC$_{arom}$), 7.44 (dd, $J = 8.5$, 1.7 Hz, 1H, CH$_{arom}$), 6.05 (dd, $J = 7.5$, 5.2 Hz, 1H, CHOAc), 3.62-$3.51$ (m, 2H, C$_{H_2}$I), 2.17 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta = 169.8$, 135.8, 133.4, 133.1, 128.7, 128.2, 127.8, 126.6 (2C), 126.1, 123.7, 75.4, 21.2, 7.8 ppm. The spectroscopic data are in agreement with the literature.$^1$

1-(3-Chlorophenyl)-2-iodoethyl acetate (2d)

3-Chlorostyrene (102 $\mu$L, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2d as yellow oil in 98% yield (255 mg, 0.790 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 7.34$-$7.29$ (m, 3H, 3xC$_{arom}$), 7.23 (dt, $J = 6.4$, 1.8 Hz, 1H, CH$_{arom}$), 5.83-$5.81$ (m, 1H, CHOAc), 3.47-$3.41$ (m, 2H, CH$_2$I), 2.15 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta = 169.8$, 140.5, 134.8, 130.1, 129.1, 126.6, 124.9, 74.5, 21.1, 7.3 ppm. IR (neat): $\nu = 2359$, 2344, 2112, 1744, 1597, 1574, 1476, 1431, 1371, 1302, 1231, 1207, 1180, 1096, 1080, 1061, 1051, 1020, 964, 926, 893, 804, 787, 721, 696, 646, 608 cm$^{-1}$. HRMS (EI): $m/z = 323.9414$ calcd. for C$_{10}$H$_{10}$ClIO$_2$+$[M]^+$, found: 323.9418.
1-(2-Chlorophenyl)-2-iodoethyl acetate (2e)  

\[
\begin{align*}
\text{OAc} & \\
\text{Cl} & \\
\end{align*}
\]

2-Chlorostyrene (102 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2e as yellow oil in 83% yield (0.660 mmol, 215 mg) after flash column chromatography (n-hexane:EtOAc = 18:1).  

\[\delta = 7.43-7.41 \text{ (m, 1H, } \text{CH}_{\text{arom}}) , \text{ 7.38-7.36 (m, 1H, } \text{CH}_{\text{arom}}), \text{ 7.31-7.27 (m, 2H, 2xCH}_{\text{arom}}), \text{ 6.18 (dd, } J = 7.9, \text{ 4.0 Hz, 1H, } \text{CHOAc}), \text{ 3.60 (dd, } J = 10.8, \text{ 4.0 Hz, 1H, } \text{CH}_{2}\text{I}), \text{ 3.42 (dd, } J = 10.8, \text{ 7.9 Hz, 1H, } \text{CH}_{2}\text{I}), \text{ 2.18 (s, 3H, } \text{CH}_{3}\text{)} \text{ ppm.} \]  

\[\delta = 169.6, \text{ 136.5, 132.3, 130.0, 129.8, 127.2, 71.6, 21.1, 6.7 ppm.} \]  

IR (neat): \( \nu = 1748, 1472, 1441, 1416, 1371, 1227, 1207, 1180, 1065, 1051, 1034, 1018, 964, 924, 756, 729, 708, 602 \text{ cm}^{-1}. \]  

HRMS (NSI): \( \text{m/z} = 197.0364 \text{ calcd. for } \text{C}_{10}\text{H}_{10}\text{ClO}_{2}^+ [M-I]^+, \text{ found: 197.0364.} \)

1-(2-Chlorophenyl)-2-iodoethan-1-ol (2e’)  

\[
\begin{align*}
\text{OH} & \\
\text{Cl} & \\
\end{align*}
\]

2e’ was obtained as yellow oil in 4% yield (10 mg, 0.030 mmol) after flash column chromatography (n-hexane:EtOAc = 18:1).  

\[\delta = 7.62-7.60 \text{ (m, 1H, } \text{CH}_{\text{arom}}), \text{ 7.35-7.24 (m, 3H, 3xCH}_{\text{arom}}), \text{ 5.20-5.16 (m, 1H, } \text{CHOH}), \text{ 3.66-3.62 (m, 1H, } \text{CH}_{2}\text{I}), \text{ 3.33-3.27 (m, 1H, } \text{CH}_{2}\text{I}), \text{ 2.55 (br, 1H, } \text{OH}) \text{ ppm.} \]  

\[\delta = 138.6, \text{ 131.9, 129.7, 129.5, 127.4, 127.1, 70.7, 13.8 ppm.} \]  

IR (neat): \( \nu = 3447, 1989, 1472, 1443, 1412, 1263, 1179, 1063, 1049, 1034, 970, 754, 735, 704 \text{ cm}^{-1}. \)  

HRMS (EI): \( \text{m/z} = 281.9308 \text{ calcd. for } \text{C}_{8}\text{H}_{8}\text{ClO}^+ [M]^+, \text{ found: 281.9307.} \)
1-(2,6-Dichlorophenyl)-2-iodoethyl acetate (2f)

![Chemical Structure Image]

2,6-Dichlorostyrene (109 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2f as pale yellow solid in 79% yield (228 mg, 0.640 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

M.p.: 55-59 °C. $^1$H NMR (300 MHz, CDCl$_3$): $\delta =$ 7.33-7.30 (m, 2H, 2xC$_{arom}$), 7.18 (dd, $J =$ 8.8, 7.2 Hz, 1H, CH$_{arom}$), 6.55 (dd, $J =$ 9.4, 6.0 Hz, 1H, CHOAc), 3.81 (dd, $J =$ 10.4, 9.4 Hz, 1H, CH$_2$I), 3.64 (dd, $J =$ 10.4, 6.0 Hz, 1H, CH$_2$I), 2.13 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta =$ 167.9, 133.2, 130.9, 128.2, 127.6, 70.6, 18.8, 0.0 ppm. IR (neat): $\nu =$ 2355, 1742, 1580, 1562, 1437, 1373, 1229, 1211, 1179, 1140, 1121, 1090, 1069, 1018, 966, 924, 878, 497, 779, 772, 735, 673, 652, 602 cm$^{-1}$. HRMS (EI): $m/z =$ 230.9980 calcd. for C$_{10}$H$_9$O$_2$Cl$_2$+ [M-I]$^+$, found: 230.9980.

2-Iodo-1-(pentafluorophenyl)ethyl acetate (2g)

![Chemical Structure Image]

2,3,4,5,6-Pentafluorostyrene (110 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2g as colourless oil in 70% yield (212 mg, 0.560 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta =$ 6.16 (t, $J =$7.8 Hz, 1H, CHOAc), 3.64 (dd, $J =$ 10.0, 7.2 Hz, 1H, CH$_2$I), 3.55 (dd, $J =$ 10.0, 8.6 Hz, 1H, CH$_2$I), 2.11 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta =$ 169.6, 145.3 (dsept, $^1$J$_{C,F}$ = 251.8 Hz, $^3$J$_{C,F}$ = 4.0 Hz), 141.8 (dsept, $^1$J$_{C,F}$ = 256.2 Hz, $^3$J$_{C,F}$ = 6.0 Hz), 137.7 (dsept, $^1$J$_{C,F}$ = 250.7 Hz, $^3$J$_{C,F}$ = 6.0 Hz), 112.0 (td, $^2$J$_{C,F}$ = 15.1 Hz, $^3$J$_{C,F}$ = 4.0 Hz), 67.2, 20.7, 2.0 ppm. $^{19}$F NMR (471 MHz, CDCl$_3$): $\delta =$ -140.9 (m, 2F), -152.2--152.3 (m, 1F), -161.0--161.1 (m, 2F) ppm. IR (neat): $\nu =$ 2361, 2344, 2097, 1749, 1653, 1522, 1504, 1427, 1373, 1306, 1221, 1152, 1132, 1065, 1022, 997, 932, 907, 837, 743, 685, 667, 625 cm$^{-1}$. HRMS (EI): $m/z =$ 379.9339 calcd. for C$_{10}$H$_6$F$_5$O$_2$+ [M]$^+$, found: 379.9339.
2-Iodo-1-(3-nitrophenyl)ethyl acetate (2h)

3-Nitrostyrene (102 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2h as yellow oil in 70% yield (187 mg, 0.790 mmol) after flash column chromatography (n-hexane:EtOAc = 10:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 8.24$-$8.20$ (m, 2H, $2\times CH_{arom}$), 7.57 (t, $J = 7.9$ Hz, 1H, $CH_{arom}$), 7.70-$7.68$ (m, 1H, $CH_{arom}$), 5.91 (t, $J = 6.4$ Hz, 1H, CHOAc), 3.52-$3.46$ (m, 2H, $CH_2$I), 2.17 (s, 3H, $CH_3$I) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta = 169.7$, 148.5, 140.6, 133.0, 129.9, 123.8, 121.6, 74.0, 21.1, 6.9 ppm. The spectroscopic data are in agreement with the literature.  

2-Iodo-1-(3-nitrophenyl)ethan-1-ol (2h’)

2h’ was obtained as pale yellow solid in 26% yield (38 mg, 0.13 mmol) after flash column chromatography (n-hexane:EtOAc = 10:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 8.27$ (m, 1H, $CH_{arom}$), 8.20-$8.18$ (m, 1H, $CH_{arom}$), 7.74-$7.72$ (m, 1H, $CH_{arom}$), 7.56 (t, $J = 7.9$ Hz, 1H, $CH_{arom}$), 4.94-$4.91$ (m, 1H, CHOH), 3.55 (dd, $J = 10.5$, 3.8 Hz, 1H, $CH_2$I), 3.41 (dd, $J = 10.5$, 8.2 Hz, 1H, $CH_2$I), 2.70 (br, 1H, O$H$) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta = 148.5$, 143.3, 132.1, 129.8, 123.4, 121.1, 72.8, 14.7 ppm. The spectroscopic data are in agreement with the literature.

2-Iodo-1-(2-α-tolyl)ethyl acetate (2i)

2-Methylstyrene (103 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2i as colourless oil in 79% yield (193 mg, 0.630 mmol) after flash column chromatography (n-hexane:EtOAc = 33:1).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta = 7.36$-$7.33$ (m, 1H, $CH_{arom}$), 7.24-$7.17$ (m, 3H, $3\times CH_{arom}$), 6.09 (dd, $J = 7.9$, 5.5 Hz, 1H, CHOAc), 3.47-$3.37$ (m, 2H, $CH_2$I), 2.42 (s, 3H, $CH_3$I), 2.13 (s,
3H, CH₃) ppm. ¹³C NMR (75 MHz, CDCl₃): δ = 169.9, 137.1, 135.5, 130.8, 128.7, 126.6, 125.2, 72.3, 21.2, 19.4, 6.8 ppm. The spectroscopic data are in agreement with the literature.³

2-Iodo-1,1-diphenylethyl acetate (2j)

1,1-Diphenylethylene (141 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2j as pale yellow solid in 91% yield (268 mg, 0.730 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

M.p.: 81-84 °C. ¹H NMR (500 MHz, CDCl₃): δ = 7.40-7.38 (m, 4H, 4xC₆H₄), 7.36-7.33 (m, 4H, 4xC₆H₄), 7.30-7.27 (m, 2H, 2xC₆H₄), 4.64 (s, 2H, CH₂I), 2.25 (s, 3H, CH₃) ppm. ¹³C NMR (75 MHz, CDCl₃): δ = 169.1, 142.6, 128.4, 127.7, 126.2, 83.5, 22.3, 16.5 ppm. IR (neat): ν = 3090, 3059, 3032, 1744, 1493, 1449, 1414, 1366, 1229, 1207, 1053, 1013, 984, 939, 777, 750, 727, 696, 608 cm⁻¹. HRMS (EI): m/z = 306.9984 calcd. for C₁₄H₁₂I [M-OAc]⁺, found: 306.9982.

1-Iodo-2-phenylpropan-2-yl acetate (2k)

α-Methylstyrene (104 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2k as yellow oil in 71% yield (172 mg, 0.57 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

¹H NMR (400 MHz, CDCl₃): δ = 7.39-7.27 (m, 5H, 5xC₆H₄), 3.86-3.78 (m, 2H, CH₂I), 2.14 (s, 3H, OCOCH₃), 1.96 (s, 3H, CH₃) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 169.6, 142.0, 128.6, 127.9, 124.7, 80.9, 26.3, 22.2, 17.5 ppm. The spectroscopic data are in agreement with the literature.¹

2-Iodo-1-phenylethan-1-one (2l) and 2-bromo-1-phenylethan-1-one (2m)

α-Bromostyrene (104 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give an inseparable mixture of 2l and 2m (1.25:1, determined via ¹H-NMR) as pale yellow oil in 70% combined yield (126 mg) after flash column chromatography (n-hexane:EtOAc = 10:1).
$^1$H NMR (500 MHz, CDCl$_3$): $\delta =$ 8.00-7.98 (m, 4H, 4xC$_{\text{arom}}$), 7.63-7.58 (m, 2H, 2xC$_{\text{arom}}$), 7.52-7.46 (m, 4H, 4xC$_{\text{arom}}$), 4.46 (s, 2H, CH$_2$Br), 4.36 (s, 2H, CH$_2$I) ppm. $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta =$ 192.9, 191.4, 134.1, 133.9, 133.6, 129.2, 129.1, 129.0, 129.0, 31.1, 1.8 ppm. The spectroscopic data are in agreement with the literature.$^4$

**Ethyl 3-(acetyloxy)-2-iodo-3-phenylpropanoate (2n)**

![Ethyl 3-(acetyloxy)-2-iodo-3-phenylpropanoate (2n)](image)

Ethyl cinnamate (134 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2n as pale yellow oil in 64% yield (185 mg, 0.510 mmol) after flash column chromatography (n-hexane:EtOAc = 10:1).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta =$ 7.42-7.34 (m, 5H, 5xC$_{\text{arom}}$), 6.15 (d, $J =$ 10.9 Hz, CHOAc), 4.61 (d, $J =$ 10.9 Hz, 1H, CHI), 4.32-4.21 (m, 2H, CH$_2$), 1.99 (s, 3H, CH$_3$), 1.30 (t, $J =$ 7.1 Hz, 3H, CH$_2$CH$_3$) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta =$ 169.4, 168.8, 136.7, 129.3, 128.6, 128.2, 76.9, 62.2, 23.2, 20.8, 14.0 ppm. The spectroscopic data are in agreement with the literature.$^5$

**2-Cyano-2-iodo-1-phenylethyl acetate (2o)**

![2-Cyano-2-iodo-1-phenylethyl acetate (2o)](image)

Cinnamonicitrile (101 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP for 4 days to give 2o as pale yellow solid in 34% yield (85 mg, 0.27 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

M.p.: 68-71 °C. $^1$H NMR (500 MHz, CDCl$_3$): $\delta =$ 7.44-7.40 (m, 5H, 5xC$_{\text{arom}}$), 5.96 (d, $J =$ 5.2 Hz, 1H, CHOAc), 4.53 (d, $J =$ 5.2 Hz, 1H, CHI), 2.22 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta =$ 169.2, 135.3, 130.0, 129.1, 127.0, 116.4, 75.4, 20.9, 0.5 ppm. IR (neat): $\nu =$ 2963, 2236, 2183, 1749, 1495, 1456, 1373, 1219, 1063, 1026, 764, 702, 608 cm$^{-1}$. HRMS (ASAP): $m/z =$ 333.0100 calcd. For C$_{11}$H$_{14}$IN$_2$O$_2$NH$_4^+$ [M+NH$_4^+$], found: 333.0109.

**2-Iodo-1,2,3,4-tetrahydronaphthalen-1-yl acetate (2p)**

![2-Iodo-1,2,3,4-tetrahydronaphthalen-1-yl acetate (2p)](image)
1,2-Dihyronaphthalene (104 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2p as orange oil in 72% yield (181 mg, 0.570 mmol) after flash column chromatography (n-hexane:EtOAc = 13:1).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 7.31-7.16$ (m, 4H, 4xC$_{\text{arom}}$), 6.21 (d, $J = 4.2$ Hz, 1H, CHOAc), 4.62-4.59 (m, 1H, CHI), 3.06-2.98 (m, 1H, Ar-CH$_2$), 2.94-2.87 (m, 1H, Ar-CH$_2$), 2.31-2.14 (m, 2H, CH$_2$), 2.10 (s, 3H, CH$_3$) ppm. The spectroscopic data are in agreement with the literature.$^1$

2-Iodo-2,3-dihydro-1H-inden-1-yl acetate (2q)

![2-Iodo-2,3-dihydro-1H-inden-1-yl acetate](image)

Indene (93 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2q as brown solid in 53% yield (128 mg, 0.430 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta = 7.43-7.40$ (m, 1H, C$_{\text{arom}}$), 7.37-7.32 (m, 1H, C$_{\text{arom}}$), 7.30-7.28 (m, 2H, 2xC$_{\text{arom}}$), 6.39 (d, $J = 3.7$ Hz, 1H, CHOAc), 4.51-4.45 (m, 1H, CHI), 3.75 (dd, $J = 17.1$, 6.8 Hz, 1H, CH$_2$), 3.33 (dd, $J = 17.1$, 4.5 Hz, 1H, CH$_2$), 2.10 (s, 3H, CH$_3$) ppm. The spectroscopic data are in agreement with the literature.$^1$

2-Iodo-1-phenylcyclohexyl acetate (2r)

![2-Iodo-1-phenylcyclohexyl acetate](image)

1-Phenyl-1-cyclohexene (127 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2r as pale yellow solid in 57% yield (158 mg, 0.460 mmol) after flash column chromatography (n-hexane:EtOAc = 10:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 7.41-7.28$ (m, 5H, 5xC$_{\text{arom}}$), 4.73-4.72 (m, 1H, CHI), 2.97-2.94 (m, 1H, CH$_2$), 2.66 (td, $J = 13.8$, 3.9 Hz, 1H, CH$_2$), 2.41-2.34 (m, 1H, CH$_2$), 2.11-2.08 (m, 1H, CH$_2$), 2.04 (s, 3H, CH$_3$), 1.87-1.76 (m, 2H, CH$_2$), 1.69-1.66 (m, 1H, CH$_2$), 1.59-1.50 (m, 1H, CH$_2$) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 168.7$, 144.2, 128.2, 128.0, 125.2, 82.9, 42.8, 32.6, 25.8, 21.9, 21.2 ppm. IR (neat): $\nu = 3059$, 3030, 2936, 2862, 2365, 2345, 2326, 1744, 1560, 1541, 1508, 1491, 1474, 1447, 1437, 1364, 1300, 1263, 1227, 1165, 1115, 1076, 1042, 981, 962, 847, 767, 716, 697, 607.
1146, 1080, 1055, 1011, 957, 920, 868, 837, 793, 758, 729, 696, 654, 625 cm⁻¹. Mass spectrum and melting point could not be provided due to rapid decomposition.

2-Iodo-1-(pyridin-2-yl)-ethyl acetate (2s)

2-Vinylpyridine (86 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2s as orange oil in 88% yield (205 mg, 0.710 mmol) after flash column chromatography (n-hexane:EtOAc = 2:1).

¹H NMR (300 MHz, CDCl₃): δ = 8.61-8.59 (m, 1H, CH_arom), 7.71 (td, J = 7.7, 1.8 Hz, 1H, CH_arom), 7.37-7.54 (m, 1H, CH_arom), 7.28-7.34 (m, 1H, CH_arom), 5.89 (dd, J = 6.5, 5.3 Hz, 1H, CHOAc), 3.75-3.70 (m, 1H, C_H₂I), 3.63 (m, 1H, C_H₂I), 2.18 (s, 3H, C_H₃) ppm. ¹³C NMR (75 MHz, CDCl₃): δ = 170.0, 157.0, 149.7, 136.9, 123.6, 121.9, 75.2, 21.1, 7.0 ppm. The spectroscopic data are in agreement with the literature.²

2-Iodo-3-oxo-1,3-diphenylpropyl acetate (2t)

Chalcone (167 mg, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 2t as orange oil in 70% yield (220 mg, 0.560 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

¹H NMR (400 MHz, CDCl₃): δ = 8.08-8.05 (m, 2H, 2xCH_arom), 7.65-7.61 (m, 1H, CH_arom), 7.54-7.50 (m, 4H, 4xCH_arom), 7.44-7.35 (m, 3H, 3xCH_arom), 6.45 (d, J = 10.6 Hz, 1H, CHOAc), 5.63 (d, J = 10.6 Hz, 1H, CHI), 1.90 (s, 3H, CH₃) ppm. ¹³C NMR (100 MHz, CDCl₃): δ = 192.8, 168.8, 137.6, 134.2, 134.0, 129.2, 129.0, 128.8, 128.5, 128.3, 76.1, 27.1, 20.8 ppm. IR (neat) ν = 3063, 3032, 2361, 1740, 1678, 1593, 1578, 1493, 1447, 1369, 1339, 1269, 1215, 1161, 1119, 1072, 1015, 984, 957, 922, 899, 876, 802, 779, 756, 737, 718, 698, 687, 652, 617 cm⁻¹. HRMS (EI): m/z = 267.1021 calcd. for C₁₇H₁₅O₃ [M-I]⁺, found: 267.1012.

1-Iodo-3-phenylpropan-2-yl acetate (4a)
Allylbenzene (106 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 4a as colourless oil in 35% yield (85 mg, 0.28 mmol) after flash column chromatography (n-hexane:EtOAc = 50:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 7.32$-$7.29$ (m, 2H, 2xCH$_{arom}$), 7.27-$7.23$ (m, 3H, 3xCH$_{arom}$), 4.88 (tt, $J = 6.6$, 5.0 Hz, 1H, CHOAc), 3.34 (dd, $J = 10.7$, 5.0 Hz, 1H, CH$_2$I), 3.20 (dd, $J = 10.7$, 5.0 Hz, 1H, CH$_2$I), 3.02-$2.93$ (m, 2H, Ar-$CH_2$), 2.07 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 170.2$, 136.3, 129.6, 128.8, 127.1, 73.0, 40.1, 21.2, 7.8 ppm. The spectroscopic data are in agreement with the literature.\(^1\)

2-Iodo-3-phenylpropyl acetate (5a)

![Image of 2-Iodo-3-phenylpropyl acetate](image)

5a was obtained as colourless oil in 32% yield (77 mg, 0.25 mmol) after flash column chromatography (n-hexane:EtOAc = 50:1).

$^1$H NMR (300 MHz, CDCl$_3$): $\delta = 7.36$-$7.27$ (m, 3H, 3xC$H_{arom}$), 7.21-$7.18$ (m, 2H, 2xC$H_{arom}$), 4.44-$4.23$ (m, 3H, $C_2H$ and $C_2H$OAc), 3.31-$3.15$ (m, 2H, Ar-$CH_2$), 2.10 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta = 170.4$, 138.7, 129.1, 128.7, 127.2, 68.6, 43.4, 29.7, 21.0 ppm. The spectroscopic data are in agreement with the literature.\(^1\)

1-Iodo-3-(4-methoxyphenyl)propan-2-yl acetate (4b)

![Image of 1-Iodo-3-(4-methoxyphenyl)propan-2-yl acetate](image)

4-Allylanisole (123 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 4b as colourless oil in 40% yield (106 mg, 0.320 mmol) after flash column chromatography (n-hexane:EtOAc = 50:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 7.17$-$7.14$ (m, 2H, 2xCH$_{arom}$), 6.86-$6.83$ (m, 2H, 2xCH$_{arom}$), 4.85-$4.80$ (m, 1H, CHOAc), 3.79 (s, 3H, OCH$_3$), 3.32 (dd, $J = 10.7$, 4.8 Hz, 1H, CH$_2$I), 3.18 (dd, $J = 10.7$, 5.1 Hz, 1H, CH$_2$I), 2.96-$2.86$ (m, 2H, Ar-$CH_2$), 2.07 (s, 3H, CH$_3$) ppm. $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 170.3$, 158.7, 130.6, 128.2, 114.2, 73.2, 55.4, 39.2, 21.2, 7.9 ppm. IR (neat): $\nu =$ 2999, 2932, 2833, 1736, 1611, 1584, 1512, 1464, 1441, 1416, 1371, 1302, 1234, 1179, 1107, 1032, 1022, 829, 820, 756, 600 cm$^{-1}$. HRMS (NSI): $m/z = 352.0404$ calcd. for C$_{12}$H$_{13}$IO$_3$NH$_4^+$ [M+NH$_4^+$], found: 352.0406.
2-Iodo-3-(4-methoxyphenyl)propyl acetate (5b)

![Chemical Structure](attachment:structure.png)

5b was obtained as colourless oil in 27% yield (71 mg, 0.21 mmol) after flash column chromatography (n-hexane:EtOAc = 50:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 7.12$-$7.09$ (m, 2H, 2xC$_{arom}$), 6.87-6.84 (m, 2H, 2xC$_{arom}$), 4.37-4.30 (m, 2H, CH$_3$I and CH$_2$OAc), 4.24 (dd, $J = 11.2$, 5.7 Hz, 1H, CH$_2$OAc), 3.80 (s, 3H, OC$_3$H$_7$), 3.20 (dd, $J = 14.5$, 6.4 Hz, 1H, Ar-C$_{CH}$), 2.11 (s, 3H, C$_3$H$_3$) ppm.

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 170.5$, 158.8, 130.8, 130.1, 114.1, 68.4, 55.4, 42.5, 30.6, 21.0 ppm. IR (neat): $\nu = 2953$, 2835, 1740, 1611, 1584, 1512, 1456, 1441, 1381, 1364, 1302, 1246, 1177, 1034, 970, 891, 833, 812, 754 cm$^{-1}$. HRMS (NSI): m/z = 352.0404 calcd. for C$_{12}$H$_{15}$IO$_3$NH$_4^+$ [M+NH$_4^+$], found: 352.0406.

1-Iodoctane-2-yl acetate (4c) and 2-iodohexyl acetate (5c)

1-Hexene (99 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give an inseparable mixture of 4c and 5c (2:1, determined via $^1$H-NMR) as yellow oil in 42% combined yield (91 mg, 0.34 mmol) after flash column chromatography (n-hexane:EtOAc = 10:1).

$^1$H NMR (500 MHz, CDCl$_3$): $\delta = 4.72$-$4.67$ (m, 1H, CHOAc), 4.35 (dd, $J = 11.6$, 6.4 Hz, 1H, CH$_2$OAc), 4.26-4.22 (m, 1H, CH$_2$OAc), 4.19-4.14 (m, 1H, CH$_3$I), 3.35 (dd, $J = 10.6$, 4.9 Hz, 1H, CH$_3$I), 3.25 (dd, $J = 10.6$, 5.4 Hz, 1H, CH$_2$I), 2.09 (s, 3H, OCOCH$_3$), 2.09 (s, 3H, OCOCH$_3$), 1.80-1.75 (m, 2H, CH$_3$CH$_2$CH$_2$CH$_2$), 1.68-1.64 (m, 2H, CH$_3$CH$_2$CH$_2$CH$_2$), 1.36-1.25 (m, 8H, 2xC$_{CH}$), 0.93-0.89 (m, 6H, 2xC$_{CH}$) ppm. $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta = 170.5$, 170.5, 72.5, 69.4, 36.3, 33.9, 31.5, 31.2, 27.3, 22.5, 22.0, 21.2, 21.0, 14.0, 14.0, 8.5 ppm. The spectroscopic data of 4c are in agreement with the literature.$^6$

1-Iodo-3-(iodomethyl)butane-2,3-diy diacetate (4d)

![Chemical Structure](attachment:structure.png)

1,5-Hexadiene (47 µL, 0.40 mmol, 0.8 equiv.) was reacted according to GP to give a mixture of diasteromers of 4d in 12% yield (23 mg, 0.05 mmol) after flash column chromatography (n-hexane:EtOAc = 50:1).
H NMR (500 MHz, CDCl₃): δ = 4.73-4.69 (m, 2H, CHOAc), 3.34 (dd, J = 5.3, 3.1 Hz, 1H, CH₂I), 3.31 (dd, J = 5.3, 3.0 Hz, 1H, CH₂I), 3.26 (dd, J = 10.6, 5.3 Hz, 2H, CH₂I), 2.10 (s, 6H, 2xC₃H₃), 1.76-1.69 (m, 4H, 2xC₂H₂) ppm. ¹³C NMR (75 MHz, CDCl₃): δ = 170.3, 71.9, 71.6, 29.7, 29.7, 21.1, 21.1, 7.6, 7.4 ppm. IR (neat): 2957, 2924, 2853, 2367, 1724, 1420, 1371, 1227, 1188, 1018, 961, 922, 860, 808, 735, 702, 654 cm⁻¹. HRMS (ESI): m/z = calcd. for C₈H₁₃IO₂ [M-OAc]⁺ 394.9005, found: 394.9022.

Regioisomers 5d and 5d’ were obtained as an inseparable mixtures of diasteromers as yellow oil in 42% combined yield (49 mg, 0.11 mmol) after flash column chromatography (n-hexane:EtOAc = 5:1).

¹H NMR (500 MHz, CDCl₃): δ = 4.81-4.71 (m), 4.42-4.34 (m), 4.27-4.19 (m), 4.18-4.10 (m), 3.37-3.30 (m), 3.28-3.24 (m), 2.11 (s), 2.10 (s), 2.10 (s), 2.10 (s) ppm. ¹³C NMR (125 MHz, CDCl₃): δ = 170.4, 170.4, 170.4, 170.3, 72.0, 71.8, 71.7, 71.4, 69.1, 69.0, 69.0, 69.0, 69.0, 36.3, 35.6, 34.1, 33.9, 32.2, 32.0, 29.8, 29.7, 29.2, 29.1, 28.7, 28.5, 21.2, 21.2, 21.0, 20.9, 7.6, 7.5, 7.5, 7.3 ppm.

4-Hydroxy-3-iodo-2-methylbutan-2-yl acetate (5e)

2-Methyl-3-buten-2-ol (81 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give 5e as yellow oil in 40% yield (86 mg, 0.32 mmol) after flash column chromatography (n-hexane:EtOAc = 3:1).

¹H NMR (500 MHz, CDCl₃): δ = 4.45-4.39 (m, 2H, CH₂), 4.36-4.34 (m, 1H, CHI), 2.11 (s, 3H, OCOCH₃), 2.00 (br, 1H, OH), 1.46 (s, 3H, CH₃), 1.44 (s, 3H, CH₃) ppm. ¹³C NMR (125 MHz, CDCl₃): δ = 170.5, 71.7, 67.1, 46.8, 28.1, 27.8, 21.1 ppm. The spectroscopic data are in agreement with the literature.

2-Iodo-3,3-dimethoxypropyl acetate (5f)
3,3-Dimethoxy-1-propene (95 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give 5f (1:1 mixture of two rotamers) as pale yellow oil in 46% yield (105 mg, 0.360 mmol) after flash column chromatography (n-hexane:EtOAc = 2:1).

\[ \text{ Dimethoxy-1-propene (95 µL, 0.80 mmol, 1.6 equiv.) } \]

\[ \text{ was reacted according to GP to give 5f (1:1 mixture of two rotamers) as pale yellow oil in 46% yield (105 mg, 0.360 mmol) after flash column chromatography (n-hexane:EtOAc = 2:1).} \]

\[ \text{1H NMR (500 MHz, CDCl}_3\text{): } \delta = 4.41-4.37 \text{ (m, 2H, } \text{CH}_2\text{OAc), 4.35-4.31 \text{ (m, 2H, } \text{CH}_2\text{OAc), 4.28-4.24 (m, 4H, } \text{CH(OMe)}_2\text{ and CHI), 3.45-3.41 (m, 12H, } \text{CH}_3\text{).} \]

\[ \text{13C NMR (75 MHz, CDCl}_3\text{): } \delta = 170.5, 104.4, 66.0, 55.4 \text{ (br), 28.6, 21.0 (br) ppm.} \]

\[ \text{IR (neat): } \nu = 3532, 3468, 3420, 3001, 2930, 2361, 1744, 1705, 1420, 1360, 1221, 1092, 1036, 903, 781 \text{ cm}^{-1} \text{.} \]

\[ \text{HRMS (EI): } m/z = 287.9859 \text{ calcd. for } \text{C}_7\text{H}_{13}\text{IO}_4^+ [M]^+, \text{ found: 287.9858.} \]

3-Chloro-2-iodobutyl acetate (5g)

\[ \text{3-Chloro-1-butene (80 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give 5g as colourless oil in 38% yield (71 mg, 0.26 mmol) after flash column chromatography (n-hexane:EtOAc = 5:1).} \]

\[ \text{1H NMR (500 MHz, CDCl}_3\text{): } \delta = 4.51 \text{ (dd, } J = 11.0, 6.2 \text{ Hz, 1H, } \text{CH}_2\text{OAc), 4.47-4.44 (m, 1H, } \text{CH}_2\text{I), 4.36 (dd, } J = 11.0, 7.6 \text{ Hz, 1H, } \text{CH}_2\text{I), 3.86 (ddd, } J = 13.1, 6.6, 2.4 \text{ Hz, 1H, } \text{CHClI), 2.09 (s, 3H, } \text{OCOC}_3\text{H}_3\text{), 1.61 (d, } J = 6.6 \text{ Hz, 3H, } \text{CH}_3\text{).} \]

\[ \text{13C NMR (75 MHz, CDCl}_3\text{): } \delta = 170.2, 66.9, 57.1, 36.2, 25.2, 20.9 \text{ ppm. IR (neat): } \nu = 3319, 2968, 2928, 2882, 2367, 2342, 1742, 1458, 1379, 1368, 1341, 1300, 1236, 1225, 1161, 1128, 1107, 1067, 1030, 951, 816, 696, 667 \text{ cm}^{-1} \text{. HRMS (EI): } m/z = 275.9414 \text{ calcd. for } \text{C}_6\text{H}_{10}\text{ClIO}_2^+ [M]^+, \text{ found: 275.9417.} \]

(E)-2-Iodo-1-phenylvinylacetate (7a) and 2,2-diiodo-1-phenylethene-1-one (8a)

\[ \text{Phenylacetylene (88 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give an inseparable mixture of 7a and 8a (1:1, determined via } \text{1H-NMR) as yellow oil in 45% combined yield (96 mg, based on I}_2 \text{ as limiting reagent) after flash column chromatography (n-hexane:EtOAc = 9:1).} \]

\[ \text{1H NMR (600 MHz, CDCl}_3\text{): } \delta = 8.04-8.03 \text{ (m, 2H, } 2\times \text{CH}_\text{arom}, \text{), 7.63-7.59 (m, 3H, } 3\times \text{CH}_\text{arom}, \text{), 7.50-7.47 (m, 2H, } 2\times \text{CH}_\text{arom}, \text{), 7.41-7.37 (m, 3H, } 3\times \text{CH}_\text{arom}, \text{), 6.51 (s, 1H, } \text{CHI}_2\text{), 6.34 (s, 1H,} \text{, S16} \]
CHI), 2.15 (s, 3H, CH₃) ppm. ¹³C NMR (150 MHz, CDCl₃): δ = 188.4, 168.7, 151.3, 134.6, 134.3, 129.7, 129.6, 129.2, 129.0, 128.8, 128.3, 67.2, 21.0, —28.8 ppm. The spectroscopic data are in agreement with the literature.⁸,⁹

**(1E)-2-Iodo-1-phenylprop-1-en-1-yl acetate (7b)**

![Chemical structure](image)

1-Phenyl-1-propyne (100 µL, 0.800 mmol, 1.6 equiv.) was reacted according to GP to give 7b as yellow oil in 71% yield (172 mg, 0.570 mmol) after flash column chromatography (n-hexane:EtOAc = 10:1).

¹H NMR (300 MHz, CDCl₃): δ = 7.50-7.47 (m, 2H, 2xC₄H₅arom), 7.39-7.32 (m, 3H, 1-H, 2xC₄H₅arom), 2.51 (s, 3H, 9C₃H₃), 2.13 (s, 3H, OCOC₃H₃) ppm. ¹³C NMR (75 MHz, CDCl₃): δ = 168.3, 146.7, 137.3, 129.7, 129.1, 128.1, 87.2, 27.4, 20.7 ppm. The spectroscopic data are in agreement with the literature.⁸

**1,1-Diiodohexan-2-one (7c)**

![Chemical structure](image)

1-Hexyne (92 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give 7c as yellow oil in 73% yield (129 mg, 0.370 mmol, based on I₂ as limiting reagent) after flash column chromatography (n-hexane:EtOAc = 5:1).

¹H NMR (500 MHz, CDCl₃): δ = 5.49 (s, 1H, CHH₂), 3.03 (t, J = 7.3 Hz, 2H, COCH₂), 1.69-1.63 (m, 2H, CH₂CH₂CH₂), 1.41-1.33 (m, 2H, CH₂CH₂), 0.95 (t, J = 7.4 Hz, 3H, CH₃) ppm. ¹³C NMR (75 MHz, CDCl₃): δ = 198.3, 33.8, 27.4, 22.2, 13.9, —23.5 ppm. The spectroscopic data are in agreement with the literature.¹⁰

**(E)-3,4-diiodo-2-methylbut-3-en-2-ol (7d)**

![Chemical structure](image)

2-Methyl-3-butyln-2-ol (78 µL, 0.80 mmol, 1.6 equiv.) was reacted according to GP to give 7d as yellow oil in 39% yield (65 mg, 0.19 mmol) after flash column chromatography (n-hexane:EtOAc = 5:1).
\(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta = 7.20\) (s, 1H, CHI), 2.28 (br, 1H, OH), 1.62 (s, 6H, 2xCH\(_3\)) ppm. \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta = 114.3, 76.2, 72.2, 29.9\) ppm. The spectroscopic data are in agreement with the literature.\(^\text{11}\)

**2-Iodo-1-phenylpropan-1-one (7e)**

Propiophenone (106 \(\mu\)L, 0.800 mmol, 1.6 equiv.) was reacted according to GP with addition of boron trifluoride diethyl etherate (296 \(\mu\)L, 2.40 mmol, 4.8 equiv.) before addition of propiophenone to give 7e as yellow solid in 70% yield (145 mg, 0.560 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 8.03-8.00\) (m, 2H, 2xCH\(_{arom}\)), 7.60-7.56 (m, 1H, CH\(_{arom}\)), 7.50-7.46 (m, 2H, 2xCH\(_{arom}\)), 5.50 (q, \(J = 6.7\) Hz, 1H, CHICH\(_3\)), 2.08 (d, \(J = 6.7\) Hz, 3H, CH\(_3\)), ppm. \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta = 194.9, 133.7, 133.7, 128.9, 128.8, 22.1, 18.2\) ppm. The spectroscopic data are in agreement with the literature.\(^\text{11}\)

**1-Oxo-1-phenylpropan-2-yl acetate (8e)**

8e was obtained as colourless oil in 5% yield (7 mg, 0.05 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta = 7.96-7.93\) (m, 2H, 2xCH\(_{arom}\)), 7.61-7.57 (m, 1H, CH\(_{arom}\)), 7.50-7.46 (m, 2H, 2xCH\(_{arom}\)), 5.97 (q, \(J = 7.0\) Hz, 1H, CHOAc), 2.15 (s, 3H, OCOCH\(_3\)), 1.53 (d, \(J = 7.0\) Hz, 3H, CH\(_3\)), ppm. \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \(\delta = 197.0, 170.6, 134.5, 133.8, 128.9, 128.6, 71.6, 20.9, 17.3\) ppm. The spectroscopic data are in agreement with the literature.\(^\text{11}\)

**Experiment of 1d with tetradeuteroacetic acid**

3-Chlorostyrene (26 \(\mu\)L, 0.20 mmol, 1.6 equiv.) was reacted according to GP with ground I\(_2\) (32.0 mg, 0.125 mmol) and ground Oxone (77 mg, 0.25 mmol) in acetic anhydride (375 \(\mu\)L, 8.00 mmol) and tetradeuteroacetic acid (750 \(\mu\)L, 13.0 mmol) to give deuterated and non-deuterated 2d (D/H = 2:1) as colourless oil in 69% yield (47 mg, 0.15 mmol) after flash column chromatography (n-hexane:EtOAc = 20:1).
5. NMR spectra
6. References


