Carbonylative Coupling of Allylic Acetates with Arylboronic Acids

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1. General information

NMR spectra were recorded on a 400 MHz spectrometer with TMS as the internal standard. All coupling constants (J values) were reported in Hertz (Hz). Data are presented as follows: chemical shift in ppm and multiplicity as \( s = \) singlet, \( d = \) doublet, \( t = \) triplet, \( q = \) quartet and \( m = \) multiplet. Thin layer chromatography (TLC) was performed on glass backed silica gel plates. Column chromatography was performed on silica gel 200-300 mesh. Flash chromatography was performed with freshly distilled solvents. HRMS (ESI) were performed on a Fourier Transform Ion Cyclotron Resonance Mass Spectrometer.

2. Optimization of reaction conditions

2.1 The purification of aryl boronic acids

Aryl boronic acids were purified by the following method: 10 mmol of arylboronic acid was dissolved in 15 mL of 1 M NaOH solution and stirred for 20 min. 1 M HCl solution was added dropwise and a white precipitate formed instantly when pH value was adjusted to 7. The white solid was filtered and recrystallised from \( \text{H}_2\text{O/CH}_3\text{CN} \). The solid was filtered, dried under reduced pressure and used directly.

2.2 Typical reaction procedure for the optimization of reaction conditions

The reaction was carried out in an autoclave containing a 10 mL Teflon reaction tube. Pd source (0.02 mmol), ligand (0.05 mmol) and a magnetic stir bar were placed in the tube, which was then capped with a stopper. Then, aryl boronic (0.5 mmol), allyl acetate (1mmol), solvent (3 mL) were added to the tube. The tube was placed in the autoclave. Once sealed, the autoclave was purged several times with CO at room temperature and heated in an oil bath at 120 °C for 12 hours. The autoclave was then cooled to room temperature and vented to discharge the excess CO. Water (10 mL) was added, and the product was extracted with EA (3×3 mL). The organic layers were washed with brine, dried over \( \text{Na}_2\text{SO}_4 \), and evaporated. The crude product was purified by column chromatography on silica gel using a mixture of ethyl acetate and petroleum ether as eluent to give the desired product. Structures of ligands screened in
this paper are shown in Scheme S1.

**Table S1 Screening of ligands and Pd sources**

![Scheme S1](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>cat</th>
<th>ligand</th>
<th>Base</th>
<th>solvent</th>
<th>Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pd(PPh₃)₄</td>
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<td>KOH</td>
<td>PhCH₃</td>
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</tr>
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<td>2</td>
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<tr>
<td>3</td>
<td>No</td>
<td>0</td>
<td>KOH</td>
<td>PhCH₃</td>
<td>0</td>
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<tr>
<td>4</td>
<td>Pd(OAc)₂</td>
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<td>KOH</td>
<td>PhCH₃</td>
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</tr>
<tr>
<td>5</td>
<td>Pd(dba)₃</td>
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<td>KOH</td>
<td>PhCH₃</td>
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<td>Pd(PPh₃)₄</td>
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<td>KOH</td>
<td>PhCH₃</td>
<td>10%</td>
</tr>
<tr>
<td>7</td>
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<td>PPh₃ (8%)</td>
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<td>PhCH₃</td>
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<tr>
<td>8</td>
<td>Pd(OAc)₂</td>
<td>PPh₃ (8%)</td>
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<td>PhCH₃</td>
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<td>11</td>
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<tr>
<td>13</td>
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<tr>
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<td>A</td>
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<td>16</td>
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<td>B</td>
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<td>PhCH₃</td>
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<td>C</td>
<td>KOH</td>
<td>PhCH₃</td>
<td>16%</td>
</tr>
<tr>
<td>18</td>
<td>Pd(OAc)₂</td>
<td>PCy₃</td>
<td>KOH</td>
<td>PhCH₃</td>
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**Scheme S1 Structures of ligands.**

**Table S2 Screening of base and additive**

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<th>Entry</th>
<th>cat</th>
<th>ligand</th>
<th>Base</th>
<th>solvent</th>
<th>additive</th>
<th>Yield</th>
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<tbody>
<tr>
<td>1</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>KF·2H$_2$O</td>
<td>PhCH$_3$</td>
<td></td>
<td>23%</td>
</tr>
<tr>
<td>2</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>NaHCO$_3$</td>
<td>PhCH$_3$</td>
<td></td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>NEt$_3$</td>
<td>PhCH$_3$</td>
<td></td>
<td>trace</td>
</tr>
<tr>
<td>4</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>HCOONa·2H$_2$O</td>
<td>PhCH$_3$</td>
<td></td>
<td>trace</td>
</tr>
<tr>
<td>5</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>NaOH</td>
<td>PhCH$_3$</td>
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<td>35%</td>
</tr>
<tr>
<td>6</td>
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<td>PCy$_3$</td>
<td>NaOH</td>
<td>PhCH$_3$</td>
<td>H$_2$O (2eq)</td>
<td>70%</td>
</tr>
<tr>
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<td>PCy$_3$</td>
<td>K$_3$PO$_4$</td>
<td>PhCH$_3$</td>
<td></td>
<td>30%</td>
</tr>
<tr>
<td>8</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>K$_3$PO$_4$·3H$_2$O</td>
<td>PhCH$_3$</td>
<td></td>
<td>60%</td>
</tr>
<tr>
<td>9</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>K$_3$PO$_4$·3H$_2$O</td>
<td>PhCH$_3$</td>
<td>H$_2$O (2eq)</td>
<td>64%</td>
</tr>
<tr>
<td>10</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>K$_3$PO$_4$·3H$_2$O</td>
<td>PhCH$_3$</td>
<td>H$_2$O (4eq)</td>
<td>72%</td>
</tr>
<tr>
<td>11</td>
<td>Pd(OAc)$_2$</td>
<td>PCy$_3$</td>
<td>K$_3$PO$_4$·3H$_2$O</td>
<td>PhCH$_3$ / dioxane</td>
<td>H$_2$O (2eq)</td>
<td>81%</td>
</tr>
</tbody>
</table>
In the case of aryl boroxine (0.17 mmol), a dramatically decreased yield (29%) of 3a was observed. Therefore, water was added to reduce aryl boroxiane produced. The yield of 3a increased to 70% (Scheme S2, eq 4), when p-tolylboronic acid (0.5 mmol) was used as substrate and 2 equiv of water as additive. General reaction conditions for Scheme S2: Allylic acetoxy (1 mol, 2 eq), Pd(OAc)$_2$ (4% mol), PCy$_3$ (10% mol), NaOH (0.5 mmol), toluene (3 mL), under 5 bar of CO. The reaction was stirred at 120 °C for 12 h. The yield of 3a was detected by NMR.

3. Typical procedure for allylic carbonylation of aryl boronic acids with allyl acetate

The reaction was carried out in an autoclave containing a 10 mL Teflon reaction tube. Pd(OAc)$_2$ (4% mol), PCy$_3$ (10% mol), and a magnetic stir bar were placed in the tube, which was then capped with a stopper. Then, aryl boronic acid (0.5 mmol), allyl acetate (1.0 mmol), solvent (toluene/dioxane = 1 : 1, 3 mL), K$_3$PO$_4$·3H$_2$O (0.5 mmol)
and H₂O (18mg, 2 eq) were added to the tube. The tube was placed in the autoclave. Once sealed, the autoclave was purged several times and then pressurized to 5 atm with CO at room temperature and heated in an oil bath at 120 °C for 12 hours (for substrates 1a, 1b, 1c, 1d, 1e, 1f, 1i, 1k, 1l, 1o, 1p) or 24 hours (for substrates 1g, 1h, 1j, 1m, 1n). The autoclave was then cooled to room temperature and vented to discharge the excess CO. Water (10 mL) was added, and the product was extracted with EA (3×3 mL). The organic layers were washed with brine, dried over Na₂SO₄, and evaporated. The crude product was purified by column chromatography on silica gel using a mixture of ethyl acetate and petroleum ether as eluent to give α, β-unsaturated aryl ketones.

4. Typical procedure for carbonylation of allyl acetates with p-tolylboronic acid

**Method A:** The reaction was carried out in an autoclave containing a 10 mL Teflon reaction tube. Pd(OAc)₂ (4% mol), PCy₃ (10% mol), and a magnetic stir bar were placed in the tube, which was then capped with a stopper. Then, aryl boronic acid (0.5 mmol), allyl acetate (1.0 mmol), solvent (toluene, 3 mL) and K₃PO₄ (0.5 mmol) were added to the tube. The tube was placed in the autoclave. Once sealed, the autoclave was purged several times and then pressurized to 5 atm with CO at room temperature and heated in an oil bath at 120 °C for 24 hours. The autoclave was then cooled to room temperature and vented to discharge the excess CO. Water (10 mL) was added, and the product was extracted with EA (3×3 mL). The organic layers were washed with brine, dried over Na₂SO₄, and evaporated. The crude product was purified by column chromatography on silica gel using a mixture of ethyl acetate and petroleum ether as eluent to give the desired products 4b-e.

**Method B:** The reaction was carried out in an autoclave containing a 10 mL Teflon reaction tube. Pd(OAc)₂ (4% mol), PCy₃ (10% mol), and a magnetic stir bar were placed in the tube, which was then capped with a stopper. Then, aryl boronic acid (0.5 mmol), allyl acetate (1.0 mmol), solvent (toluene / dioxane = 1:1, 3 mL), K₃PO₄·3H₂O (0.5 mol) and H₂O (18 mg, 2 eq) were added to the tube. The tube was then placed in the autoclave. Once sealed, the autoclave was purged several times and
then pressurized to 5 atm with CO at room temperature and heated in an oil bath at 120 °C for 24 hours (for substrates 2f, 2g, 2h, 2l, 2m, 2n) or 12 hours (for substrates 2i, 2j, 2k). The autoclave was then cooled to room temperature and vented to discharge the excess CO. Water (10 mL) was added, and the product was extracted with EA (3×3 mL). The organic layers were washed with brine, dried over Na₂SO₄, and evaporated. The crude product was purified by column chromatography on silica gel using a mixture of ethyl acetate and petroleum ether as eluent to give the desired products.

5. Procedures for study of reaction mechanism

An autoclave containing a 10 mL Teflon reaction tube was charged with a magnetic stir bar. p-Tolylboronic acid (68 mg, 0.5 mmol, 1eq), 2f (114 mg, 2eq), Pd(OAc)₂ (4 mg, 4% mol), PCy₃ (14 mg, 10% mol), K₃PO₄·3H₂O (133 mg, 1 eq), toluene / dioxane = (1 : 1) (3 mL) and water (18 mg, 2 eq.) were added to the tube with a syringe. The tube was placed in the autoclave. Once sealed, the autoclave was pressurized with CO (5 bar) and heated in an oil bath at 120 °C for 1 h or 24 h. The yields of 4f and 4f-a were determined by NMR.

6. Data for products

3a. (E)-1-(p-tolyl)but-2-en-1-one:

Yield: 81% (12 h, 65 mg); pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.83 (d, J = 8.1 Hz, 2 H), 7.24 (d, J = 2.8 Hz, 2 H), 7.09 – 7.00 (m, 1H), 6.9 (d, J = 15.3 Hz, 1 H), 2.40 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 190.2, 144.4, 143.3, 135.3, 129.2, 128.6, 127.5, 21.6, 18.5; IR (KBr): 3082, 1671, 1621, 1443, 966, 799, 742 cm⁻¹; HRMS (ESI) Calcd for C₁₁H₁₂O [M] + Na⁺ = 183.0782, Found = 183.0775.

3b. (E)-1-phenylbut-2-en-1-one:
Yield: 71% (52 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.92 (d, $J$ = 7.8 Hz, 2 H), 7.54 (t, $J$ = 6.6 Hz, 1 H), 7.46 (t, $J$ = 7.5 Hz, 2 H), 7.11–7.01 (m, 1 H), 6.91 (d, $J$ = 15.3 Hz, 1 H), 2.00 (d, $J$ = 6.8 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.8 145.0, 137.9, 132.6, 128.5, 127.6, 18.6; IR (KBr): 3059, 1671, 1624, 1443, 966, 691 cm$^{-1}$; HRMS (ESI) Calcd for C$_{10}$H$_{10}$O [M] + Na$^+$ = 169.0629, Found = 169.0666.

3c. (E)-1-(4-ethylphenyl)but-2-en-1-one:

Yield: 73% (63.5 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.87 (d, $J$ = 8.3 Hz, 2 H), 7.28 (d, $J$ = 8.3 Hz, 2 H), 7.11–7.02 (m, 1 H), 6.91 (d, $J$ = 15.3 Hz, $J$ = 1.5 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.3, 149.6, 144.4, 135.6, 128.7, 128.0, 127.5, 28.9, 18.5, 15.1. IR (KBr): 3031, 1671, 1607, 1413, 969, 810 cm$^{-1}$; HRMS (ESI) Calcd for C$_{12}$H$_{14}$O [M] + Na$^+$ = 197.0942, Found = 197.0940.

3d. (E)-1-(4-propylphenyl)but-2-en-1-one:

Yield: 71% (66 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.86 (d, $J$ = 8.2 Hz, 2 H), 7.26 (d, $J$ = 8.1 Hz, 2 H), 7.11–7.02 (m, 1 H), 6.91 (dd, $J$ = 15.3 Hz, $J$ = 1.4 Hz, 1 H), 2.64 (t, $J$ = 7.5 Hz, 2 H), 1.98 (dd, $J$ = 6.8 Hz, $J$ = 1.3Hz, 3H), 1.66 (m, 2 H), 0.94 (t, $J$ = 7.3 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.3, 148.0, 144.4, 135.6, 128.7, 128.6, 127.5 38.0, 24.2, 18.5, 13.7. IR (KBr): 3036, 1668, 1618, 1440, 1299, 969 cm$^{-1}$; HRMS (ESI) Calcd for C$_{13}$H$_{16}$O [M] + Na$^+$ = 211.1094, Found = 211.1087.

3e. (E)-1-(4-(tert-butyl)phenyl)but-2-en-1-one:

Yield: 85% (86 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm) 7.88 (d, $J$ = 8.2 Hz, 2 H), 7.48 (d, $J$ = 8.2 Hz, 2 H), 7.11-7.02 (m, 1 H), 6.91 (d, $J$ = 15.3 Hz, 1
H), 1.99 (d, J = 6.8 Hz, 3H), 1.34 (s, 9 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm):
190.3, 156.3, 144.5, 135.3, 128.5, 127.5, 125.5, 35.1, 31.1, 18.6; IR (KBr): 3042, 1671, 1621, 1440, 966, 810 cm$^{-1}$; HRMS (ESI) Calcd for C$_{14}$H$_{18}$O [M]+Na$^+$ = 225.1251, Found = 225.1240.

3f. (E)-1-[(1, 1’-biphenyl)-4-yl]but-2-en-1-one:

![Chemical Structure]

Yield: 60% (67 mg); white solid, Mp (91-92 °C). $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 8.01 (d, J = 8.4 Hz, 2 H), 7.68 (d, J = 8.4 Hz, 2 H), 7.62 (d, J = 7.2 Hz, 2 H), 7.48-7.37 (m, 3 H), 7.15 – 7.06 (m, 1H), 6.95 (dd, J = 15.2 Hz, J = 1.5 Hz, 1 H), 2.01 (dd, J = 6.8 Hz, J = 1.5 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.2, 145.3, 144.9, 140.0, 136.6, 129.1, 128.9, 127.5, 127.3, 127.2, 18.6; IR (KBr): 3056, 1665, 1618, 1440, 919, 760 cm$^{-1}$; HRMS (ESI) Calcd for C$_{16}$H$_{14}$O [M]+ Na$^+$ = 245.0937, Found = 245.0931.

3g. (E)-1-(4-methoxyphenyl)but-2-en-1-one:

![Chemical Structure]

Yield: 62% (54 mg); colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.93 (d, J = 8.8 Hz, 2 H), 7.09 – 7.00 (m, 1 H), 6.94-6.89 (m, 3 H), 3.85 (s, 3 H), 1.97 (dd, J = 6.7 Hz, J = 1.2 Hz, 3 H); $^{13}$C NMR(100 MHz, CDCl$_3$) δ (ppm): 188.9, 163.3, 143.9, 130.8, 127.2, 113.7, 55.4, 18.5; IR (KBr): 3067, 1665, 1612, 1443, 1027, 1071, 813 cm$^{-1}$; HRMS (ESI) Calcd for C$_{11}$H$_{12}$O$_2$ [M]+ Na = 199.0730, Found = 199.0725.

3h. (E)-1-(4-chlorophenyl)but-2-en-1-one:

![Chemical Structure]
Yield: 30% (27 mg); colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.87 (d, $J = 8.6$ Hz, 2 H), 7.44 (d, $J = 8.6$ Hz, 2H), 7.13 – 7.04 (m, 1H), 6.87 (dd, $J = 15.3$ Hz, $J = 1.6$ Hz, 1 H), 2.01 (dd, $J = 6.9$ Hz, $J = 1.6$ Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 189.4, 145.6, 139.0, 136.2, 129.9, 128.8, 127.1, 18.6; IR (KBr): 3061, 1668, 1618, 1260, 1091, 805 cm$^{-1}$; HRMS (ESI) Calcd for C$_{10}$H$_9$ClO $[M] + Na^+$ = 203.0234, Found = 203.0218.

3i. (E)-1-(m-tolyl)but-2-en-1-one:

Yield: 88% (70 mg); colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.74–7.70 (m, 2 H), 7.37–7.32 (m, 2 H), 7.11–7.02 (m, 1 H), 6.90 (dd, $J = 15.3$ Hz, $J = 1.5$ Hz, 1 H), 2.41 (s, 3 H), 2.00 (dd, $J = 6.8$ Hz, $J = 1.5$ Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.9, 144.7, 138.3, 137.9, 133.3, 129.0, 128.3, 127.7, 125.7, 21.3, 18.5; IR (KBr): 2959, 1651, 1615, 1257, 1099, 802 cm$^{-1}$; HRMS (ESI) Calcd for C$_{11}$H$_{12}$O $[M] + Na^+$ = 183.0780, Found = 183.0776.

3j. (E)-1-(3-methoxyphenyl)but-2-en-1-one:

Yield: 78% (69 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm): 7.50 – 7.46 (m, 2 H), 7.36 (t, $J = 8.0$ Hz, 2 H), 7.12–7.03 (m, 2 H), 7.12–7.03 (m, 2H), 6.89 (dd, $J = 15.3$ Hz, $J = 1.5$ Hz, 1 H), 3.86 (s, 3 H), 2.00 (dd, $J = 6.8$ Hz, $J = 1.5$ Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.5, 159.8, 145.1, 139.3, 129.5, 127.6, 121.7, 119.2, 112.8, 55.4, 18.6; IR (KBr): 3003, 1671, 1624, 1460, 1035, 780 cm$^{-1}$; HRMS (ESI) Calcd for C$_{11}$H$_{12}$O$_2$ $[M] + Na^+$ = 199.0730, Found = 199.0721.

3k. (E)-1-(3,5-dimethylphenyl)but-2-en-1-one:
Yield: 86% (75 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.35 (s, 2 H), 7.17 (s, 1 H), 7.09-7.00 (m, 1 H), 6.90 (dd, $J = 15.3$ Hz, $J = 1.5$ Hz, 1 H), 2.36 (s, 6 H), 1.98 (dd, $J = 6.8$ Hz, $J = 1.6$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 191.1, 144.5, 138.1, 138.0, 134.2, 127.8, 129.4, 126.3, 21.2, 18.5; IR (KBr): 3042, 1674, 1601, 1307, 1188, 1043 cm$^{-1}$; HRMS (ESI) Calcd for C$_{12}$H$_{14}$O [M] + Na$^+$ = 197.09, Found = 197.0931.

3l. (E)-1-(o-tolyl)but-2-en-1-one:

Yield: 63% (50 mg); colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.37-7.30 (m, 2 H), 7.26-7.20 (m, 2 H), 6.77- 6.68 (m, 1 H), 6.50 (d, $J = 15.7$ Hz, 1 H), 2.38(s, 3 H), 1.94 (d, $J = 6.8$ Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 196.9, 146.7, 139.0, 136.6, 132.4, 131.1, 130.1, 127.9, 125.2, 20.0, 18.5; IR (KBr): 3021, 1651, 1621, 1451, 1032, 763 cm$^{-1}$. HRMS (ESI) Calcd for C$_{11}$H$_{12}$O [M] + Na$^+$ = 183.0780, Found = 183.0776.

3m. (E)-1-(2-fluorophenyl)but-2-en-1-one:

Yield: 60% (49 mg); pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm): 7.69 (td, $J = 7.5$Hz, $J = 1.8$ Hz, 1H), 7.51-7.45 (m, 1 H), 7.22 (td, $J = 7.6$ Hz, $J = 1.0$ Hz 1 H), 7.14 – 7.09 (m, 1 H), 7.04-6.95 (m, 1 H), 6.77-6.71 (m, 1 H), 1.98 (dd, $J = 6.9$ Hz, $J = 1.6$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm): 188.6 (d, $J_{C,F} = 2.2$ Hz), 159.9 (d, $J_{C,F} = 251$ Hz), 144.7, 132.5 (d, $J_{C,F} = 8.6$ Hz), 130.1 (d, $J_{C,F} = 5.4$ Hz), 129.7 (d, $J_{C,F} = 2.8$ Hz), 126.0 (d, $J_{C,F} = 13.8$ Hz), 123.3 (d, $J_{C,F} = 3.5$ Hz), 115.4 (d, $J_{C,F} = 22.8$ Hz), 115.4 (d, $J_{C,F} = 22.8$ Hz),

3n. (E)-1-(2-chlorophenyl)but-2-en-1-one:

Yield: 68% (61 mg); colorless oil. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.42-7.29 (m, 4 H), 6.76-6.67 (m, 1 H), 6.48 (dd, J = 15.7 Hz, J = 1.6 Hz, 1 H), 1.96 (dd, J = 6.8 Hz, J = 1.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 194.2, 148.0, 139.0, 132.0, 131.1, 130.1, 129.0, 126.6, 18.6; IR (KBr): 3059, 1660, 1618, 1435, 1038, 763 cm⁻¹; HRMS (ESI) Calcd for C₁₀H₉ClO [M] + Na⁺ = 203.0234, Found = 203.0231.

3o. (E)-1-(thiophen-3-yl)but-2-en-1-one:

Yield: 66% (50 mg); pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.75 (d, J = 3.4 Hz, 1 H), 7.63 (d, J = 4.8 Hz, 1 H), 7.17-7.08 (m, 2 H), 6.82 (d, J = 15.2 Hz, 1 H), 1.98 (d, J = 6.9 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 182.2, 145.1, 144.2, 133.6, 131.8, 128.1, 126.9, 18.4. IR (KBr): 3097, 1665, 1618, 1435, 1038, 763 cm⁻¹. HRMS (ESI) Calcd for C₈H₈OS [M] + Na⁺ = 175.0184, Found = 175.0182.

3p. (E)-1-(naphthalen-1-yl)but-2-en-1-one:

Yield: 80% (78 mg); pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.25 (d, J = 7.8 Hz, 1 H), 7.95 (d, J = 8.2 Hz, 1 H), 7.88 (d, J = 7.4 Hz, 1 H), 7.65 (d, J = 7.0 Hz, 1 H), 7.57-7.47 (m, 3 H), 6.90-6.81 (m, 1 H), 6.68 (d, J = 15.6 Hz, 1 H), 1.97 (d, J =...
6.8 Hz, 3 H), 7.03 (dd, J = 1.4 Hz, J = 5.2 Hz, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 196.1, 146.9, 136.9, 133.8, 132.8, 131.3, 130.5, 128.3, 127.3, 126.9, 126.4, 125.6, 124.4, 18.6; IR (KBr): 3056, 1668, 1621, 1296, 1185, 816 cm$^{-1}$; HRMS (ESI) Calcd for C$_{14}$H$_{12}$O [M] + Na$^+$ = 219.0782, Found = 219.0772.

4b. (E)-4-phenyl-1-(p-tolyl)but-2-en-1-one

Yield: 55% (65 mg); pale yellow oil. $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.90 (d, J = 8.2 Hz, 2 H), 7.37 (d, J = 7.3 Hz, 2 H), 7.31-7.19 (m, 5 H), 6.56-6.43 (m, 2 H), 3.87 (d, J = 6.1 Hz, 3 H), 2.4 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 197.6, 144.0, 137.1, 134.2, 133.4, 129.4, 128.5, 128.4, 127.4, 126.3, 122.8, 42.7, 21.7; IR (KBr): 3028, 1674, 1604, 1257, 1177, 694 cm$^{-1}$; HRMS (ESI) Calcd for C$_{17}$H$_{16}$O [M] + Na$^+$ = 259.1099, Found = 259.1096.

4c. (E)-4-(m-tolyl)-1-(p-tolyl)but-2-en-1-one

Yield: 56% (70 mg); pale yellow oil; $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.91 (d, J = 8.1 Hz, 2 H), 7.28 (d, J = 8.0 Hz, 2 H), 7.21-7.18 (m, 3 H), 7.04 (d, J = 5.7 Hz, 1 H), 6.54 – 6.43 (m, 2 H), 3.88 (d, J = 5.9 Hz, 2 H), 2.42 (s, 3 H), 2.34 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 197.7, 144.0, 141.3, 138.0, 137.0, 134.2, 133.5, 129.4, 128.5, 128.4, 128.2, 126.9, 123.5, 122.6, 42.7, 21.7, 21.4; IR (KBr): 3028, 1674, 1607, 1282, 1179, 752 cm$^{-1}$; HRMS (ESI) Calcd for C$_{18}$H$_{18}$O [M] + H$^+$ = 251.1430, Found = 251.1424.

4d. (E)-4-(2-ethylphenyl)-1-(p-tolyl)but-2-en-1-one
Yield: 45% (60 mg); pale yellow oil. $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.92 (d, $J$ = 8.0 Hz, 2 H), 7.46 (d, $J$ = 7.4 Hz, 2 H), 7.28 (d, $J$ = 8.0 Hz, 2 H), 7.18 (m, 3 H), 6.80 (d, $J$ = 15.7 Hz, 1 H), 6.38-6.30 (m, 1 H), 3.91 (d, $J$ = 6.8 Hz, 2 H), 2.68 (q, $J$ = 7.6 Hz, 2 H), 2.42 (s, 3 H), 1.18 (t, $J$ = 7.6 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$), δ = 197.7, 144.0, 141.3, 135.6, 134.2, 131.2, 129.3, 128.6, 128.5, 127.5, 126.1, 126.0, 124.3, 43.0, 26.3, 21.6, 15.3; IR (KBr): 3028, 1674, 1607, 1260, 1178, 784 cm$^{-1}$; HRMS (ESI) Calcd for C$_{19}$H$_{20}$O [M] + Na$^+$ = 287.1406, Found =287.1404.

4e. (E)-4-(2-methoxyphenyl)-1-(p-tolyl)but-2-en-1-one

Yield: 65% (86 mg); pale yellow oil. $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.90 (d, $J$ = 8.1 Hz, 2 H), 7.45 (dd, $J$ = 7.6 Hz, $J$ = 1.4 Hz, 2 H), 7.27 (d, $J$ = 8.3 Hz, 2 H), 7.22-7.18 (m, 1 H), 6.92-6.84 (m, 3 H), 6.50-6.43 (m, 1 H), 3.90 (dd, $J$ = 6.9 Hz, $J$ = 1.4 Hz, 2 H), 3.84 (s, 3 H), 2.42(s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$), δ (ppm): 197.8, 156.5, 143.9, 134.3, 129.3, 128.4, 128.2, 126.8, 126.2, 123.4, 120.7, 110.8, 55.5, 43.2, 21.6; IR (KBr): 2956, 1671, 1599, 1488, 1243, 1024 cm$^{-1}$; C$_{18}$H$_{18}$O$_2$ [M] + Na$^+$ = 289.1200, Found = 289.1202.

4f. (E)-1-(p-tolyl)pent-2-en-1-one

Yield: 60% (52 mg); pale yellow oil; $^1$H NMR (400M Hz, CDCl$_3$): δ = 7.84 (d, $J$ = 8.2 Hz, 2 H), 7.26 (d, $J$ = 7.9 Hz, 2 H), 7.13 – 7.06 (m, 1H), 6.87 (td, $J$ = 15.4 Hz, $J$ = 1.6 Hz, 1 H), 2.42 (s, 3 H), 2.38-2.30 (m, 3 H), 1.14 (t, $J$ = 7.4 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$), δ = 190.6, 150.7, 143.3, 135.5, 129.2, 128.7, 124.9, 25.9, 21.6,
12.4. IR (KBr): 3113, 2920, 1733, 1503, 1389, 1255, 1192, 751 cm$^{-1}$. C$_{12}$H$_{14}$O [M] $+ \text{Na}^+$ = 197.0937, Found = 197.0932.

4g. (E)-4-cyclohexyl-1-(p-tolyl)but-2-en-1-one

Yield: 56% (68 mg); pale yellow oil. $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.84 (d, $J$ = 8.2 Hz, 2 H), 7.26 (d, $J$ = 7.9 Hz, 2 H), 7.08-7.00 (m, 1 H), 6.85 (td, $J$ = 15.3 Hz, $J$ = 1.2 Hz, 1 H), 2.41 (s, 3 H), 2.22-2.18 (m, 2 H), 1.77-1.63 (m, 5 H), 1.51 – 1.46 (m, 1 H), 1.29-1.13 (m, 3 H), 1.02-0.92 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.3, 148.4, 143.3, 135.5, 129.2, 128.7, 126.9, 40.8, 37.5, 33.2, 26.4, 26.2, 21.6; IR (KBr): 3028, 1668, 1610, 1446, 1263, 1016, 805 cm$^{-1}$; C$_{17}$H$_{22}$O [M] $+ \text{H}^+$ = 243.1743, Found = 243.1739.

4h. (E)-1-(p-tolyl)non-2-en-1-one

Yield: 43% (50 mg); pale yellow oil. $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.84 (d, $J$ = 8.2 Hz, 2 H), 7.26 (d, $J$ = 8.0 Hz, 2 H), 7.09-7.02 (m, 1 H), 6.87 (td, $J$ = 15.4 Hz, $J$ = 1.3 Hz), 2.41 (s, 3 H), 2.33-2.28 (m, 2 H), 1.55-1.48 (m, 2 H), 1.37-1.28 (m, 6 H), 0.89 (t, $J$ = 6.8 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.5, 149.6, 143.3, 135.5, 130.2, 129.2, 128.7, 125.8, 32.8, 31.6, 28.9, 28.2, 22.6, 21.6, 14.1; IR (KBr): 2970, 1696, 1649, 1538, 1451, 1041 cm$^{-1}$; C$_{16}$H$_{22}$O [M] $+ \text{Na}^+$ = 253.1559, Found = 253.1559.

4i. 3-methyl-1-(p-tolyl)but-2-en-1-one
Yield: 84% (73 mg); pale yellow oil. $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.83 (d, $J$ = 8.2 Hz, 2 H), 7.23 (d, $J$ = 8.0 Hz, 2 H), 6.72 (t, $J$ = 1.2 Hz, 1 H), 2.39 (s, 3 H), 2.19 (d, $J$ = 0.8 Hz, 3 H), 2.00 (d, $J$ = 0.9 Hz, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm): 190.3, 154.8, 141.9, 135.7, 128.1, 127.3, 120.2, 26.9, 20.5, 20.1; IR (KBr): 2961, 1660, 1610, 1254, 1013, 805 cm$^{-1}$; HRMS (ESI) Calcd for C$_{12}$H$_{14}$O [M] + Na$^+$ = 197.0937, Found = 197.0933.

4j. 4-methyl-1-(p-tolyl)pent-3-en-1-one

pale yellow oil; $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.87 (d, $J$ = 8.2 Hz, 2 H), 7.26 (d, $J$ = 7.9 Hz, 2 H), 5.45 - 5.41 (m, 1 H), 3.66 (d, $J$ = 6.9 Hz, 2 H), 2.41 (s, 3 H), 1.76 (d, $J$ = 1.1 Hz, 3 H), 1.69 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm) 198.3, 143.7, 135.3, 134.4, 129.2, 128.4, 116.5, 38.4, 25.8, 21.6, 18.2; IR (KBr): 2975, 1665, 1604, 1503, 1293, 1179, 816 cm$^{-1}$; HRMS (ESI) Calcd for C$_{13}$H$_{16}$O [M] + Na$^+$ = 211.1093, Found = 211.1107.

4f-a. (Z/E)-1-(p-tolyl)pent-3-en-1-one

pale yellow oil; $^1$H NMR (400M Hz, CDCl$_3$) δ (ppm): 7.89 (m, 2 H), 7.28 (m, 2 H), 5.75-5.61 (m, 2 H), 3.76-3.67 (m, 2 H), 2.43 (s, 3 H), 1.75-1.67 (m, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm) 198.4, 197.9, 143.8, 134.3, 134.2, 129.4, 129.3, 128.5, 128.4, 127.4, 123.6, 122.5, 42.4, 37.1, 21.7, 18.2, 13.2; IR (KBr): 3036, 1668, 1607, 1279, 1018, 810 cm$^{-1}$; HRMS (ESI) Calcd for C$_{12}$H$_{14}$O [M] + Na$^+$ = 197.0937, Found = 197.0932.

4k. (E)-7-oxo-7-(p-tolyl)hept-5-en-1-yl acetate
Yield: 52% (67 mg); pale yellow oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.85 (d, \(J = 8.1\) Hz, 2 H), 7.27 (d, \(J = 7.1\) Hz, 2 H), 7.07-7.00 (m, 1 H), 6.90 (d, \(J = 15.4\) Hz), 4.09 (t, \(J = 6.3\) Hz, 2 H), 2.42 (s, 3 H), 2.36 (q, \(J = 7.0\) Hz, 2 H), 2.06 (s, 3 H), 1.72-1.67 (m, 4 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm) 198.3, 143.7, 135.3, 134.4, 129.2, 128.4, 116.5, 38.4, 25.8, 21.6, 18.2; IR (KBr): 2989, 1732, 1662, 1615, 1235, 1038 cm\(^{-1}\); HRMS (ESI) Calcd for C\(_{16}\)H\(_{20}\)O [M] + Na\(^+\) = 283.1305, Found = 283.1296.

4l. (E)-4, 8-dimethyl-1-(p-tolyl) nona-3, 7-dien-1-one

Yield: 40% (51 mg); pale yellow oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 7.87 (d, \(J = 8.2\) Hz, 2 H), 7.26 (d, \(J = 7.9\) Hz, 2 H), 5.46-5.41 (m, 1 H), 5.08-5.05 (m, 1 H), 3.67 (d, \(J = 6.8\) Hz, 2 H), 2.41 (s, 3 H), 2.08 (m, 4 H), 1.69 (s, 3 H), 1.64 (s, 3 H), 1.58 (s, 3 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) (ppm): 198.3, 143.7, 138.8, 134.4, 131.6, 129.2, 128.5, 124.0, 116.4, 39.7, 38.4, 26.5, 25.6, 21.6, 17.7, 16.6; IR (KBr): 2981, 1676, 1610, 1285, 1105, 813 cm\(^{-1}\); HRMS (ESI) Calcd for C\(_{18}\)H\(_{24}\)O [M] + H\(^+\) = 257.1890, Found = 257.1893.

7. References

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8. Copies of the $^1$H NMR and $^{13}$C NMR spectra of products

3a. (E)-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3b. (E)-1-phenylbut-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3c. (E)-1-(4-ethylphenyl)but-2-en-1-one:

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

![NMR spectrum](image)

**$^{13}$C NMR (100 MHz, CDCl$_3$)**

![NMR spectrum](image)
3d. (E)-1-(4-propylphenyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3e. (E)-1-(4-(tert-butyl)phenyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3f. (E)-1-[(1,1'-biphenyl]-4-yl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3g. (E)-1-(4-methoxyphenyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3h. (E)-1-(4-chlorophenyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3i. (E)-1-(m-tolyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3j. (E)-1-(3-methoxyphenyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3k. (E)-1-(3,5-dimethylphenyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3l. (E)-1-(o-tolyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3m. (E)-1-(2-fluorophenyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3n. (E)-1-(2-chlorophenyl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3o. (E)-1-(thiophen-3-yl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
3p. (E)-1-(naphthalen-1-yl)but-2-en-1-one:

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4b. (E)-4-phenyl-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4c. (E)-4-(m-tolyl)-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4d. (E)-4-(2-ethylphenyl)-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4e. (E)-4-(2-methoxyphenyl)-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4f. (E)-1-(p-tolyl)pent-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4g. (E)-4-cyclohexyl-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4h. (E)-1-(p-tolyl)non-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4i. 3-methyl-1-(p-tolyl)but-2-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4j. 4-methyl-1-(p-tolyl)pent-3-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4f-a. (Z/E)-1-(p-tolyl)pent-3-en-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4k. (E)-7-oxo-7-(p-toly)hept-5-en-1-yl acetate

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

$^{13}$C NMR (100 MHz, CDCl$_3$)
4l. (E)-4, 8-dimethyl-1-(p-tolyl) nona-3, 7-dien-1-one

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

$^{13}$C NMR (100 MHz, CDCl$_3$)