Supporting Information

© Copyright Wiley-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2013

Synthesis of Conjugated Dienes via a Biomimetic Aerobic Oxidative Coupling of Two C\textsubscript{vinyl}–H Bonds

Nicolas Gigant and Jan-E. Bäckvall*[a]

chem_201301771_sm_miscellaneous_information.pdf
Supporting Information

Synthesis of Conjugated Dienes via a Biomimetic Aerobic Oxidative Coupling of two C_vinyl-H Bonds

Nicolas Gigant and Jan-Erling Bäckvall*

Department of Organic Chemistry, Arrhenius Laboratory
Stockholm University, 106 91 Stockholm - Sweden
jeb@organ.su.se

Table of Contents

1. General Information .............................................................................................................................. 2
2. Experimental Procedures ...................................................................................................................... 3
   2.1. Starting alkenes ............................................................................................................................ 3
   2.2. General procedure for the synthesis of conjugated dienes ............................................................ 3
   2.3. Optimization of the biomimetic coupling .................................................................................. 4
   2.4. Products characterization .............................................................................................................. 5
3. NMR Spectra ...................................................................................................................................... 16
1. General Information

THF and toluene were dried with a solvent purifier prior use. CH₂Cl₂ was distillated over CaH₂. Reactions were monitored by thin-layer chromatography (TLC) analysis using silica gel (60 F254) plates. Compounds were visualized by UV irradiation and/or spraying with a solution of potassium permanganate, followed by charring at 150 °C. Flash column chromatography was performed on silica gel 60 (230-400 mesh, 0.040-0.063 mm). ¹H and ¹³C NMR spectra were recorded on a spectrometer at 400 MHz (¹³C, 100 MHz). Chemical shifts are given in parts per million from tetramethylsilane (TMS) as internal standard. The following abbreviations are used for the proton spectra multiplicities: s: singulet, d: doublet, t: triplet, q: quartet, qt: quintuplet, m: multiplet, br.: broad, dd: double doublet, dt: double triplet. Coupling constants (J) are reported in Hertz (Hz). HRMS were recorded using ESI-TOF techniques. All reagents were obtained from commercial suppliers unless otherwise stated.
2. Experimental Procedures

2.1. Starting alkenes

Alkenes 1c, 1d, 1e, 1g, 1h, 1i, 1j, 1k, 1m, 1n were synthesized according to the literature procedures.

2.2. General procedure for the synthesis of conjugated dienes

Method A

A Schlenk tube, charged with Pd(OAc)₂ (5 mol%), p-benzoquinone (20 mol%), iron phthalocyanine (2.5 mol%), was degassed three times under reduced pressure before introducing oxygen gas with a balloon. Then, substrate 1 (2 equiv.), olefin 2 (1 equiv.) and AcOH (0.5 ml/mmol) were added and the resulting mixture was stirred in an oil bath at 90 °C for 24 hours. The reaction mixture was cooled to room temperature, followed by the addition of saturated aqueous NaHCO₃ and AcOEt. The organic layer was separated and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over MgSO₄, filtered and concentrated. Products were purified by flash chromatography with hexane/ethyl acetate to yield the desired conjugated dienes.

Method B

A Schlenk tube, charged with Pd(OAc)₂ (2.5 mol%), p-benzoquinone (5 mol%), iron phthalocyanine (1 mol%) and substrate 1 (1 equiv.), was degassed three times under reduced pressure before introducing oxygen gas with a balloon. Then, olefin 2 (2 equiv.) and AcOH/DMA (1/1, 1.2–1.5 mL/mmol, or 1.35 g/mmol for PivOH) were added and the resulting mixture was stirred in an oil bath at 70 °C for 24 hours. The reaction mixture was cooled to room temperature, followed by the addition of saturated aqueous NaHCO₃ and AcOEt. The organic layer was separated and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over MgSO₄, filtered and concentrated. Products were purified by flash chromatography with hexane/ethyl acetate to yield the desired conjugated dienes.

---

Method C

A Schlenk tube, charged with Pd(OAc)$_2$ (5 mol%), $p$-benzoquinone (10 mol%), iron phthalocyanine (2.5 mol%) and substrate 1 (1 equiv.), was degassed three times under reduced pressure before introducing oxygen gas with a balloon. Then, olefin 2 (2 equiv.) and AcOH/DMA (1/1, 1.35 mL/mmol) were added and the resulting mixture was stirred in an oil bath at 70 °C for 24 hours. The reaction mixture was cooled to room temperature, followed by the addition of saturated aqueous NaHCO$_3$ and AcOEt. The organic layer was separated and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over MgSO$_4$, filtered and concentrated. Products were purified by flash chromatography with hexane/ethyl acetate to yield the desired conjugated dienes.

2.3. Optimization of the biomimetic coupling

Optimization of the biomimetic aerobic oxidative coupling between $\alpha$-methylstyrene 1a and $n$-butyl acrylate 2a

<table>
<thead>
<tr>
<th>Entry</th>
<th>1a (equiv.)</th>
<th>2a (equiv)</th>
<th>Cat. [Pd] (mol%)</th>
<th>Oxidant (mol%)</th>
<th>Fe(Pc) (mol%)</th>
<th>Solvent</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>56</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>PivOH</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>EtCOOH</td>
<td>60 (messy)</td>
</tr>
<tr>
<td>4</td>
<td>5</td>
<td>1</td>
<td>Pd(TFA)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>23</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
<td>1</td>
<td>Pd(OAc)$_2$ (2.5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>17</td>
</tr>
<tr>
<td>6</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (6)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>53</td>
</tr>
<tr>
<td>7</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>56</td>
</tr>
<tr>
<td>8</td>
<td>1</td>
<td>3</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>27</td>
</tr>
<tr>
<td>9</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>2,6-DMBQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>35</td>
</tr>
<tr>
<td>10</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>Chloranil (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>28</td>
</tr>
<tr>
<td>11</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (10)</td>
<td>2.5</td>
<td>AcOH</td>
<td>32</td>
</tr>
<tr>
<td>12</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>5</td>
<td>AcOH</td>
<td>49</td>
</tr>
<tr>
<td>13</td>
<td>2</td>
<td>1</td>
<td>Pd(OAc)$_2$ (5)</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH/DMA</td>
<td>19</td>
</tr>
<tr>
<td>14</td>
<td>2</td>
<td>1</td>
<td>-</td>
<td>BQ (20)</td>
<td>2.5</td>
<td>AcOH</td>
<td>-</td>
</tr>
</tbody>
</table>

Reactions were performed at 90 °C for 24 h. Isolated yields.
Optimization of the biomimetic aerobic oxidative coupling between substrate 1b and methyl acrylate 2b

![Coupling Reaction Diagram]

<table>
<thead>
<tr>
<th>Entry</th>
<th>Pd(OAc)$_2$ (mol%)</th>
<th>BQ (mol%)</th>
<th>Fe(Pc) (mol%)</th>
<th>Solvent</th>
<th>T (°C)</th>
<th>Time (h)</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>20</td>
<td>2.5</td>
<td>AcOH</td>
<td>70</td>
<td>60</td>
<td>51</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>20</td>
<td>2.5</td>
<td>AcOH</td>
<td>90</td>
<td>60</td>
<td>trace</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>20</td>
<td>2.5</td>
<td>AcOH</td>
<td>70</td>
<td>24</td>
<td>78</td>
</tr>
<tr>
<td>4</td>
<td>5</td>
<td>10</td>
<td>2.5</td>
<td>AcOH</td>
<td>70</td>
<td>24</td>
<td>76</td>
</tr>
<tr>
<td>5</td>
<td>2.5</td>
<td>10</td>
<td>2.5</td>
<td>AcOH</td>
<td>70</td>
<td>24</td>
<td>49</td>
</tr>
<tr>
<td>6</td>
<td>2.5</td>
<td>10</td>
<td>2.5</td>
<td>AcOH/DMA</td>
<td>70</td>
<td>24</td>
<td>79</td>
</tr>
<tr>
<td>7</td>
<td>2.5</td>
<td>5</td>
<td>1</td>
<td>AcOH/DMA</td>
<td>70</td>
<td>24</td>
<td>76</td>
</tr>
<tr>
<td>8</td>
<td>1</td>
<td>5</td>
<td>2.5</td>
<td>AcOH/DMA</td>
<td>70</td>
<td>24</td>
<td>26</td>
</tr>
<tr>
<td>9</td>
<td>2.5</td>
<td>2.5</td>
<td>1</td>
<td>AcOH/DMA</td>
<td>70</td>
<td>24</td>
<td>24</td>
</tr>
</tbody>
</table>

*Isolated yields.*

### 2.4. Products characterization

**(2E)-Butyl 5-phenylhexa-2,4-dienoate (3a)**

Prepared following the general procedure (Method A) using **1a** (260 µL, 2.00 mmol), **n-butyl acrylate 2a** (143 µL, 1.00 mmol), Pd(OAc)$_2$ (11.2 mg, 0.05 mmol), p-benzoquinone (21.6 mg, 0.20 mmol), Fe(Pc) (11.4 mg, 0.02 mmol) in AcOH (0.50 mL). Flash chromatography (hexane/ethyl acetate: 97/3 to 95/5) afforded **3a** ($E:Z = 86:16$) (137 mg, 56%) as a colourless oil.

$R_f$ 0.87 (hexane/ethyl acetate: 9/1); $^1$H NMR (400 MHz, CDCl$_3$) δ 0.99 (t, 3H, $J = 7.4$ Hz), 1.45 (qt, 2H, $J = 7.6$ Hz), 1.71 (qt, 2H, $J = 6.7$ Hz), 2.32 (d, 3H, $J = 1.1$ Hz), 4.22 (t, 2H, $J = 6.7$ Hz), 6.02 (d, 1H, $J = 15.0$ Hz), 6.60 (dt, 1H, $J_1 = 11.7$ Hz, $J_2 = 1.1$ Hz), 7.33-7.40 (m, 3H), 7.51 (m, 2H), 7.78 (dd, 1H, $J_1 = 15.1$ Hz, $J_2 = 11.7$ Hz); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 13.9, 16.7, 19.3, 30.9, 64.3, 121.5, 124.8, 126.1, 128.4, 128.6, 140.7, 142.1, 145.4, 167.6; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{16}$H$_{20}$NO$_2$ 267.1356, found 267.1344.
(2R,3S,4R)-2-(Acetoxymethyl)-5-((E)-3-methoxy-3-oxoprop-1-en-1-yl)-3,4-dihydro-2H-pyran-3,4-diyl diacetate (3b)

Prepared following the general procedure (Method B) using 1b (2.00 g, 7.35 mmol), methyl acrylate 2b (1.33 mL, 14.7 mmol), Pd(OAc)$_2$ (41.2 mg, 0.18 mmol), p-benzoquinone (39.7 mg, 0.37 mmol), Fe(Pc) (41.8 mg, 0.07 mmol) in AcOH/DMA (1/1, 10 mL). Flash chromatography (hexane/ethyl acetate: 7/3 to 6/4) afforded 3b (2.12 g, 81%) as a yellow oil.$^{11}$

R$_f$ 0.34 (hexane/ethyl acetate: 7/3) ; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.05 (s, 3H), 2.05 (s, 3H), 2.06 (s, 3H), 3.69 (s, 3H), 4.16 (dd, 1H, $J_1 = 12.4$ Hz, $J_2 = 3.9$ Hz), 4.41 (dd, 1H, $J_1 = 11.6$ Hz, $J_2 = 7.5$ Hz), 4.46 (m, 1H), 5.13 (t, 1H, $J = 3.5$ Hz), 5.55 (dd, 1H, $J_1 = 3.4$ Hz, $J_2 = 1.3$ Hz), 5.59 (dd, 1H, $J_1 = 15.8$ Hz, $J_2 = 0.3$ Hz), 6.96 (s, 1H), 7.17 (d, 1H, $J = 15.9$ Hz) ; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 20.9, 21.0, 21.1, 51.8, 61.3, 63.1, 66.7, 74.4, 109.7, 114.1, 141.3, 152.6, 167.7, 169.6, 170.1, 170.6 ; HRMS (ESI) m/z $[M+Na]^+$ calcd for C$_{16}$H$_{20}$NaO$_9$ 379.1000, found 379.0981.

(E)-Methyl 3-(2-(acetoxymethyl)-3,4-dihydro-2H-pyranyl-5-yl)acrylate (3c)

Prepared following the general procedure (Method B) using 1c (50 mg, 0.32 mmol), methyl acrylate 2b (58 µL, 0.64 mmol), Pd(OAc)$_2$ (1.8 mg, 0.008 mmol), p-benzoquinone (1.7 mg, 0.016 mmol), Fe(Pc) (1.8 mg, 0.003 mmol) in AcOH/DMA (1/1, 0.40 mL). Flash chromatography (hexane/ethyl acetate: 7/3 to 6/4) afforded 3c (51 mg, 66%) as a yellow oil.

R$_f$ 0.53 (hexane/ethyl acetate: 7/3) ; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.72 (m, 1H), 1.97 (m, 1H), 2.08 (s, 3H), 2.21 (m, 2H), 3.73 (s, 3H), 4.12-4.26 (m, 3H), 5.64 (d, 1H, $J = 15.5$ Hz), 6.87 (s, 1H), 7.25 (d, 1H, $J = 15.5$ Hz) ; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 19.1, 20.9, 23.1, 51.5, 65.7, 74.4, 111.9, 125.9, 144.6, 151.8, 168.1, 170.9 ; HRMS (ESI) m/z $[M+Na]^+$ calcd for C$_{12}$H$_{16}$NaO$_5$ 263.0890, found 263.0896.

(2E)-$\text{tert}$-Butyl 5-(naphthalen-2-yl)hexa-2,4-dienoate (3d)

Prepared following the general procedure (Method A) using 1d (168 mg, 1.00 mmol), t-buty1 acrylate 2c (73 µL, 0.50 mmol), Pd(OAc)₂ (5.6 mg, 0.025 mmol), p-benzoquinone (10.8 mg, 0.10 mmol), Fe(Pc) (7.1 mg, 0.013 mmol) in AcOH (0.25 mL). Flash chromatography (hexane/ethyl acetate: 99/1 to 97/3) afforded 3d (E:Z = 97:03) (71 mg, 52%) as a colourless oil.

Rf 0.59 (hexane/ethyl acetate: 97/3); ¹H NMR (400 MHz, CDCl₃) δ 1.54 (s, 9H), 2.40 (d, 3H, J = 1.1 Hz), 5.98 (d, 1H, J₁ = 15.0 Hz), 6.72 (dt, 1H, J₁ = 11.7 Hz, J₂ = 1.1 Hz), 7.48 (m, 2H), 7.64 (dd, 1H, J₂ = 8.7 Hz, J₃ = 1.9 Hz), 7.74 (dd, 1H, J₁ = 15.1 Hz, J₂ = 11.7 Hz), 7.80-7.87 (m, 3H), 7.91 (d, 1H, J = 1.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 16.6, 28.3, 80.4, 123.6, 123.9, 125.3, 125.4, 126.4, 126.5, 127.7, 128.1, 128.5, 133.2, 133.4, 139.3, 139.8, 144.5, 167.0; HRMS (ESI) m/z [M+Na]⁺ calcd for C₂₀H₂₂NaO₂ 317.1512, found 317.1493.

(E)-Methyl 3-(3-methyl-1H-inden-2-yl)acrylate (3e)

Prepared following the general procedure (Method A) using 1e (132 mg, 1.00 mmol), methyl acrylate 2b (45 µL, 0.50 mmol), Pd(OAc)₂ (5.6 mg, 0.025 mmol), p-benzoquinone (10.8 mg, 0.10 mmol), Fe(Pc) (7.1 mg, 0.013 mmol) in AcOH (0.25 mL). Flash chromatography (hexane/ethyl acetate: 97/3 to 95/5) afforded 3e (72 mg, 67%) as a yellow oil.

Rf 0.34 (hexane/ethyl acetate: 97/3); ¹H NMR (400 MHz, CDCl₃) δ 2.29 (t, 3H, J = 1.8 Hz), 3.53 (d, 2H, J = 1.4 Hz), 3.80 (s, 3H), 6.05 (d, 1H, J = 15.6 Hz), 7.28 (dd, 1H, J₁ = 7.1 Hz, J₂ = 4.4 Hz), 7.32 (m, 1H), 7.38 (m, 1H), 7.44 (m, 1H), 7.86 (d, 1H, J = 15.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 11.0, 37.2, 51.7, 116.5, 120.2, 123.8, 126.8, 127.1, 136.9, 138.0, 142.9, 145.9, 146.1, 168.1; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₄H₁₄NaO₂ 237.0886, found 237.0886.

(E)-Methyl 4-cyclohexylidenebut-2-enoate (3f)

Prepared following the general procedure (Method A) using 1e (120 µL, 1.00 mmol), methyl acrylate 2b (45 µL, 0.50 mmol), Pd(OAc)₂ (5.6 mg, 0.025 mmol), p-benzoquinone (10.8 mg, 0.10 mmol), Fe(Pc) (7.1 mg, 0.013 mmol) in AcOH (0.25 mL). Flash chromatography (hexane/ethyl acetate: 98/2) afforded 3f (7 mg, 8%) as a colourless film.

Rf 0.84 (hexane/ethyl acetate: 97/3); ¹H NMR (400 MHz, CDCl₃) δ 1.60 (m, 6H), 2.20 (m, 2H), 2.40 (m, 2H), 3.74 (s, 3H), 5.79 (d, 1H, J = 15.1 Hz), 5.93 (dd, 1H, J₁ = 11.7 Hz, J₂ = 0.9 Hz), 7.63 (dd, 1H, J₁ = 15.1 Hz, J₂ = 11.7 Hz); ¹³C NMR (100 MHz, CDCl₃) No satisfying analysis was obtained due to the degradation of the product.
(E)-Methyl 3-(1-benzyl-6-oxo-1,4,5,6-tetrahydropyridin-3-yl)acrylate (3g)

Prepared following the general procedure (Method B) using 1g (50 mg, 0.27 mmol), methyl acrylate 2b (48 µL, 0.53 mmol), Pd(OAc)₂ (1.5 mg, 0.007 mmol), p-benzoquinone (1.4 mg, 0.014 mmol), Fe(Pc) (1.5 mg, 0.003 mmol) in AcOH/DMA (1/1, 0.40 mL). Flash chromatography (hexane/ethyl acetate: 7/3 to 6/4) afforded 3g (48 mg, 66%) as a white solid.

Rf 0.48 (hexane/ethyl acetate: 7/3) ; ¹H NMR (400 MHz, CDCl₃) δ 2.50 (t, 2H, J = 8.3 Hz), 2.69 (t, 2H, J = 8.0 Hz), 3.73 (s, 3H), 4.73 (s, 2H), 5.69 (dd, 1H, J₁ = 15.5 Hz, J₂ = 0.4 Hz), 6.47 (s, 1H), 7.22-7.35 (m, 6H) ; ¹³C NMR (100 MHz, CDCl₃) δ 20.2, 30.6, 49.6, 51.6, 114.2, 116.3, 127.9, 128.1, 129.0, 136.2, 136.5, 143.1, 167.8, 169.3 ; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₆H₁₇NNaO₃ 294.1101, found 294.1094.

(E)-Methyl 3-(1-acetamido-3,4-dihydronaphthalen-2-yl)acrylate (3h)

Prepared following the general procedure (Method B) using 1h (50 mg, 0.27 mmol), methyl acrylate 2b (49 µL, 0.54 mmol), Pd(OAc)₂ (1.5 mg, 0.007 mmol), p-benzoquinone (1.5 mg, 0.013 mmol), Fe(Pc) (1.5 mg, 0.003 mmol) in AcOH/DMA (1/1, 0.40 mL). Flash chromatography (hexane/ethyl acetate: 3/7 to 2/8) afforded 3h (46 mg, 63%) as a brown solid.

Rf 0.67 (hexane/ethyl acetate: 2/8) ; ¹H NMR (400 MHz, DMSO) δ 2.12 (s, 3H), 2.52 (t, 2H, J = 8.5 Hz), 2.80 (t, 2H, J = 8.3 Hz), 3.70 (s, 3H), 6.12 (d, 1H, J = 15.8 Hz), 7.48 (t, 1H, J = 7.8 Hz), 7.54 (m, 1H), 7.65 (d, 1H, J = 15.8 Hz), 7.92 (m, 1H), 8.08 (m, 1H), 9.56 (s, 1H) ; ¹³C NMR (100 MHz, DMSO) δ 22.7, 22.7, 26.6, 51.4, 117.3, 122.7, 124.4, 126.4, 127.4, 128.1, 128.6, 136.5, 137.0, 141.0, 167.1, 169.0 ; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₆H₁₇NNaO₃ 294.1101, found 294.1102.

(E)-Methyl 3-(1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)acrylate (3i)

Prepared following the general procedure (Method B) using 1i (50 mg, 0.36 mmol), methyl acrylate 2b (71 µL, 0.67 mmol), Pd(OAc)₂ (2.0 mg, 0.009 mmol), p-benzoquinone (1.9 mg, 0.018 mmol), Fe(Pc) (2.0 mg, 0.003 mmol) in AcOH/DMA (1/1, 0.40 mL). Flash chromatography (hexane/ethyl acetate: 7/3 to 6/4) afforded 3i (48 mg, 66%) as a white solid.

Rf 0.48 (hexane/ethyl acetate: 7/3) ; ¹H NMR (400 MHz, CDCl₃) δ 2.50 (t, 2H, J = 8.3 Hz), 2.69 (t, 2H, J = 8.0 Hz), 3.73 (s, 3H), 4.73 (s, 2H), 5.69 (dd, 1H, J₁ = 15.5 Hz, J₂ = 0.4 Hz), 6.47 (s, 1H), 7.22-7.35 (m, 6H) ; ¹³C NMR (100 MHz, CDCl₃) δ 20.2, 30.6, 49.6, 51.6, 114.2, 116.3, 127.9, 128.1, 129.0, 136.2, 136.5, 143.1, 167.8, 169.3 ; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₆H₁₇NNaO₃ 294.1101, found 294.1094.
mg, 0.004 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 2/8 to 1/9) afforded 3i (59 mg, 74%) as a white solid.12

Rf 0.55 (hexane/ethyl acetate: 1/9) ; 1H NMR (400 MHz, CDCl3) δ 3.36 (s, 3H), 3.46 (s, 3H), 3.74 (s, 3H), 6.96 (d, 1H, J = 15.7 Hz), 7.26 (d, 1H, J = 15.8 Hz), 7.43 (s, 1H) ; 13C NMR (100 MHz, CDCl3) δ 28.2, 37.6, 51.7, 108.9, 118.7, 136.7, 145.0, 150.8, 161.4, 168.1 ; HRMS (ESI) m/z [M+Na]+ calcd for C10H12N2NaO4 247.0689, found 247.0678.

(E)-Methyl 3-(1-methyl-6-oxo-1,6-dihydropyridin-3-yl)acrylate (3j)

Prepared following the general procedure (Method B) using 1j (50 mg, 0.46 mmol), methyl acrylate 2b (79 µL, 0.92 mmol), Pd(OAc)2 (2.6 mg, 0.011 mmol), p-benzoquinone (2.5 mg, 0.023 mmol), Fe(Pc) (2.6 mg, 0.023 mmol) in PivOH (0.60 g) for 36 h at 120 °C. Flash chromatography (ethyl acetate) afforded 3j (47 mg, 53%) as a yellow solid.13

Rf 0.40 (ethyl acetate) ; 1H NMR (400 MHz, CDCl3) δ 3.56 (s, 3H), 3.78 (s, 3H), 6.13 (d, 1H, J = 15.8 Hz), 6.60 (d, 1H, J = 9.5 Hz), 7.40 (dd, 1H, J1 = 15.8 Hz, J2 = 0.4 Hz), 7.45 (d, 1H, J = 2.5 Hz), 7.57 (dd, 1H, J1 = 9.5 Hz, J2 = 2.6 Hz) ; 13C NMR (100 MHz, CDCl3) δ 38.0, 51.8, 114.5, 115.2, 121.4, 136.2, 139.8, 140.9, 162.7, 167.4 ; HRMS (ESI) m/z [M+H]+ calcd for C10H12NO3 194.0812, found 194.0821.

(E)-Methyl 3-(1-benzyl-6-(4-methoxyphenyl)-4-oxo-1,4,5,6-tetrahydropyridin-3-yl)acrylate (3k)

Prepared following the general procedure (Method B) using 1k (50 mg, 0.17 mmol), methyl acrylate 2b (31 µL, 0.34 mmol), Pd(OAc)2 (1.0 mg, 0.004 mmol), p-benzoquinone (0.9 mg, 0.009 mmol), Fe(Pc) (1.0 mg, 0.002 mmol) in AcOH/DMA (1/1, 0.20 mL). Flash chromatography (hexane/ethyl acetate: 6/4 to 5/5) afforded 3k (17 mg, 16%) as a yellow solid.8

Rf 0.47 (hexane/ethyl acetate: 5/5) ; 1H NMR (400 MHz, CDCl3) δ 2.71 (dd, 1H, J1 = 16.4 Hz, J2 = 6.4 Hz), 2.93 (dd, 1H, J1 = 16.4 Hz, J2 = 7.2 Hz), 3.73 (s, 3H), 3.81 (s, 3H), 4.26 (d, 1H, J = 14.5 Hz), 4.44 (d, 1H, J = 14.5 Hz), 4.52 (t, 1H, J = 6.8 Hz), 6.70 (d, 1H, J = 15.6 Hz), 6.87 (d, 2H, J = 8.8 Hz), 7.11 (d, 2H, J = 8.7 Hz), 7.13 (m, 2H), 7.21 (d, 1H, J = 15.6 Hz), 7.38 (m, 3H), 7.56 (s, 1H) ; 13C NMR (100 MHz, CDCl3) δ 44.2, 51.3, 55.5, 58.2, 60.1, 106.4, 111.5, 114.8, 128.0, 128.3, 128.9, 129.4, 129.7, 134.2,


S9
140.2, 157.0, 160.0, 169.7, 188.0; HRMS (ESI) m/z [M+Na]⁺ calcd for C₂₅H₂₃NNaO₄ 400.1519, found 400.1530.

(E)-Methyl 3-(4-oxo-4H-chromen-3-yl)acrylate (3l)

Prepared following the general procedure (Method B) using 11 (50 mg, 0.36 mmol), methyl acrylate 2b (65 µL, 0.71 mmol), Pd(OAc)₂ (2.0 mg, 0.009 mmol), p-benzoquinone (1.9 mg, 0.018 mmol), Fe(Pc) (2.0 mg, 0.004 mmol) in PivOH (0.50 g) for 36 h at 120 °C. Flash chromatography (hexane/ethyl acetate: 6/4 to 5/5) afforded 3l (49 mg, 60%) as a white solid.¹⁴

Rf 0.23 (hexane/ethyl acetate: 8/2); H NMR (400 MHz, CDCl₃) δ 3.79 (s, 3H), 7.30 (d, 1H, J = 15.8 Hz), 7.39-7.49 (m, 3H), 6.69 (m, 1H), 8.11 (s, 1H), 8.26 (ddd, 2H, J₁ = 8.0 Hz, J₂ = 1.7 Hz, J₃ = 0.4 Hz); C NMR (100 MHz, CDCl₃) δ 51.8, 118.2, 119.4, 121.9, 124.3, 126.0, 126.5, 134.2, 135.8, 155.7, 157.6, 168.0, 176.0; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₃H₁₀NaO₄ 253.0471, found 253.0476.

(E)-Methyl 4-(1,3-dithiolan-2-ylidene)-5-oxohex-2-enoate (3m)

Prepared following the general procedure (Method C) using 1m (60 mg, 0.37 mmol), methyl acrylate 2b (68 µL, 0.75 mmol), Pd(OAc)₂ (4.2 mg, 0.019 mmol), p-benzoquinone (4.0 mg, 0.037 mmol), Fe(Pc) (5.3 mg, 0.009 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 8/2 to 7/3) afforded 3m (57 mg, 63%) as a yellow solid.

Rf 0.18 (hexane/ethyl acetate: 8/2); H NMR (400 MHz, CDCl₃) δ 2.35 (s, 3H), 3.38 (m, 4H), 3.78 (s, 3H), 6.12 (d, 1H, J = 16.0 Hz), 7.75 (d, 1H, J = 16.0 Hz); C NMR (100 MHz, CDCl₃) δ 28.6, 36.2, 39.0, 51.8, 120.5, 122.0, 141.2, 167.5, 169.5, 193.4; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₀H₁₂NaO₃S₂ 267.0120, found 267.0131.

(2E,2′E)-Dimethyl 3,3′-2,8-dioxaspiro[5.5]undeca-3,9-diene-4,10-diyl)diacrylate (3n)

Prepared following the general procedure (Method B) using 1n (50 mg, 0.33 mmol), methyl acrylate 2b (119 µL, 1.31 mmol), Pd(OAc)₂ (3.7 mg, 0.016 mmol), p-benzoquinone (3.6 mg, 0.033 mmol), Fe(Pc) (3.7 mg, 0.007 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 8/2 to 6/4) afforded 3n (17 mg, 16%) as a colourless oil.

Rᵣ 0.23 (hexane/ethyl acetate: 7/3) ; ¹H NMR (400 MHz, CDCl₃) δ 2.04 (m, 4H), 3.74 (s, 6H), 3.72 (d, 2H, J = 11.0 Hz), 3.90 (d, 2H, J = 11.0 Hz), 5.64 (dd, 2H, J₁ = 15.6 Hz, J₂ = 0.5 Hz), 6.89 (s, 2H), 7.30 (dd, 2H, J₁ = 15.6 Hz, J₂ = 0.5 Hz) ; ¹³C NMR (100 MHz, CDCl₃) δ 29.4, 29.5, 51.5, 70.0, 110.7, 112.3, 144.1, 151.4, 167.9 ; HRMS (ESI) m/z [M+Na]+ calcd for C₁₇H₂₀NaO₆ 343.1152, found 343.1137.

(2E)-Methyl 5-phenylhexa-2,4-dienoate (3p)

Prepared following the general procedure (Method A) using 1a (260 µL, 2.00 mmol), methyl acrylate 2b (91 µL, 1.00 mmol), Pd(OAc)₂ (11.2 mg, 0.05 mmol), p-benzoquinone (21.6 mg, 0.20 mmol), Fe(Pc) (11.4 mg, 0.02 mmol) in AcOH (0.50 mL). Flash chromatography (hexane/ethyl acetate: 97/3 to 95/5) afforded 3p (E:Z = 93:07) (110 mg, 54%) as a colourless oil.

Rᵣ 0.58 (hexane/ethyl acetate: 95/5) ; ¹H NMR (400 MHz, CDCl₃) δ 2.30 (d, 3H, J = 1.1 Hz), 3.78 (s, 3H), 5.99 (d, 1H, J = 15.1 Hz), 6.57 (dt, 1H, J₁ = 11.7 Hz, J₂ = 1.0 Hz), 7.31-7.39 (m, 4H), 7.49 (m, 1H), 7.76 (dd, 1H, J₁ = 15.1 Hz, J₂ = 11.7 Hz) ; ¹³C NMR (100 MHz, CDCl₃) δ 15.7, 50.6, 119.9, 123.8, 125.1, 127.5, 127.6, 140.0, 141.1, 144.7, 166.9 ; HRMS (ESI) m/z [M+Na]⁺ calcd for C₁₃H₁₄NaO₂ 225.0886, found 225.0889.

(2E)-Methyl 5-(4-chlorophenyl)hexa-2,4-dienoate (3q)

Prepared following the general procedure (Method A) using 1q (143 µL, 1.00 mmol), methyl acrylate 2b (45 µL, 0.50 mmol), Pd(OAc)₂ (5.6 mg, 0.025 mmol), p-benzoquinone (10.8 mg, 0.10 mmol), Fe(Pc) (7.1

mg, 0.013 mmol) in AcOH (0.25 mL). Flash chromatography (hexane/ethyl acetate: 97/3 to 95/5) afforded \(3q\) (E:Z = 95:05) (71 mg, 60%) as a colourless oil.

\[ R_f 0.61 \text{ (hexane/ethyl acetate: 95/5) ; } ^1H \text{ NMR (400 MHz, CDCl}_3\) \delta 2.26 (d, 3H, } J = 1.2 \text{ Hz), 3.77 (s, 3H), 5.99 (d, 1H, } J = 15.1 \text{ Hz), 6.54 (dt, 1H, } J_1 = 11.7 \text{ Hz, } J_2 = 1.2 \text{ Hz), 7.31 (d, 2H, } J = 8.7 \text{ Hz), 7.71 (dd, 1H, } J_1 = 15.1 \text{ Hz, } J_2 = 11.7 \text{ Hz) ; } ^{13}C \text{ NMR (100 MHz, CDCl}_3\) \delta 15.6, 50.7, 120.4, 124.1, 126.3, 127.7, 133.3, 139.4, 139.7, 143.2, 166.8 \text{ ; HRMS (ESI) m/z [M+Na]^+ caled for C}_{13}H_{13}ClNaO_2 259.0496, found 259.0534.} \]

\( (2E)-\text{tert-Butyl 5-phenylhexa-2,4-dienoate (3r)} \)

Prepared following the general procedure (Method A) using \(1a\) (260 µL, 2.00 mmol), \(\text{t-butyl acrylate} 2c\) (147 µL, 1.00 mmol), Pd(OAc)_2 (11.2 mg, 0.05 mmol), \(p\)-benzoquinone (21.6 mg, 0.20 mmol), Fe(Pc) (11.4 mg, 0.02 mmol) in AcOH (0.50 mL). Flash chromatography (hexane/ethyl acetate: 97/3 to 95/5) afforded \(3r\) (E:Z = 93:07) (101 mg, 41%) as a colourless oil.\(^{15}\)

\[ R_f 0.73 \text{ (hexane/ethyl acetate: 95/5) ; } ^1H \text{ NMR (400 MHz, CDCl}_3\) \delta 1.53 (s, 9H), 2.90 (d, 3H, } J = 1.3 \text{ Hz), 5.93 (d, 1H, } J = 15.1 \text{ Hz), 6.56 (d, 1H, } J = 11.1 \text{ Hz), 7.31-7.39 (m, 4H), 7.48 (m, 1H), 7.68 (dd, 1H, } J_1 = 15.0 \text{ Hz, } J_2 = 11.6 \text{ Hz) ; } ^{13}C \text{ NMR (100 MHz, CDCl}_3\) \delta 16.6, 28.3, 80.3, 123.4, 124.9, 126.1, 128.3, 128.6, 139.8, 142.2, 144.8, 166.9 \text{ ; HRMS (ESI) m/z [M+Na]^+ caled for C}_{16}H_{20}NaO_2 267.1356, found 267.1342.} \]

\( (2R,3S,4R)-2-(\text{Acetoxymethyl})-5-((E)-3-oxoprop-1-en-1-yl)-3,4-dihydro-2H-pyran-3,4-diyl diacetate (3s) \)

Prepared following the general procedure (Method B) using \(1b\) (100 mg, 0.37 mmol), acrolein \(2d\) (49 µL, 0.73 mmol), Pd(OAc)_2 (2.1 mg, 0.009 mmol), \(p\)-benzoquinone (2.0 mg, 0.018 mmol), Fe(Pc) (2.1 mg, 0.004 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 6/4 to 5/5) afforded \(3s\) (83 mg, 69%) as a colourless oil.

\[ R_f 0.51 \text{ (hexane/ethyl acetate: 5/5) ; } ^1H \text{ NMR (400 MHz, CDCl}_3\) \delta 2.05 (s, 3H), 2.07 (s, 3H), 2.08 (s, 3H), 4.16 (dd, 1H, } J_1 = 11.2 \text{ Hz, } J_2 = 4.1 \text{ Hz), 4.44-4.55 (m, 2H), 5.17 (t, 1H, } J = 3.4 \text{ Hz), 5.56 (dd, 1H, } J_1 = 3.2 \text{ Hz, } J_2 = 1.4 \text{ Hz), 5.92 (dd, 1H, } J_1 = 15.6 \text{ Hz, } J_2 = 7.5 \text{ Hz), 6.98 (d, 1H, } J = 15.8 \text{ Hz), 7.09 (s, 1H), 9.45 (d, 1H, } J = 7.6 \text{ Hz) ; } ^{13}C \text{ NMR (100 MHz, CDCl}_3\) \delta 20.7, 20.7, 20.8, 61.0, 62.7, 66.2, 74.9, 109.9, 116.2, 125.3, 153.7, 168.4, 169.8, 170.5, 193.1 \text{ ; HRMS (ESI) m/z [M+Na]^+ caled for C}_{18}H_{18}NaO_8 394.0894, found 349.0889.} \]
(2R,3S,4R)-2-(Acetoxymethyl)-5-((E)-styril)-3,4-dihydro-2H-pyran-3,4-diyl diacetate (3t)

Prepared following the general procedure (Method C) using 1b (100 mg, 0.37 mmol), styrene 2e (85 µL, 0.73 mmol), Pd(OAc)₂ (4.2 mg, 0.018 mmol), p-benzoquinone (4.0 mg, 0.036 mmol), Fe(Pc) (5.2 mg, 0.009 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 8/2 to 7/3) afforded 3t (79 mg, 57%) as a colourless oil.¹¹

Rf 0.44 (hexane/ethyl acetate: 7/3) ; ¹H NMR (400 MHz, CDCl₃) δ 2.09 (s, 3H), 2.11 (s, 3H), 2.11 (s, 3H), 4.21 (m, 1H), 4.46 (m, 2H), 5.19 (t, 1H, J = 3.7 Hz), 5.76 (d, 1H, J = 3.4 Hz), 6.26 (d, 1H, J = 17.3 Hz), 6.56 (d, 1H, J = 16.4 Hz), 6.81 (s, 1H), 7.33 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 20.9, 21.0, 21.0, 61.5, 63.8, 67.3, 73.9, 110.9, 124.1, 125.1, 126.1, 127.3, 128.8, 137.5, 146.6, 169.7, 170.4, 170.6; HRMS (ESI) m/z [M+Na]+ calcd for C₂₀H₂₂NaO₇ 397.1258, found 397.1256.

(2R,3S,4R)-2-(Acetoxymethyl)-5-((E)-2-cyanovinyl)-3,4-dihydro-2H-pyran-3,4-diyl diacetate (3u)

Prepared following the general procedure (Method C) using 1b (100 mg, 0.37 mmol), acrylonitrile 2f (49 µL, 0.73 mmol), Pd(OAc)₂ (4.2 mg, 0.018 mmol), p-benzoquinone (4.0 mg, 0.036 mmol), Fe(Pc) (5.2 mg, 0.009 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 7/3 to 6/4) afforded 3u (59 mg, 50%) as a yellow oil.¹¹

Rf 0.21 (hexane/ethyl acetate: 7/3) ; ¹H NMR (400 MHz, CDCl₃) δ 2.09 (s, 3H), 2.09 (s, 3H), 2.10 (s, 3H), 4.17 (dd, 1H, J₁ = 11.9 Hz, J₂ = 4.2 Hz), 4.45 (dd, 1H, J₁ = 11.9 Hz, J₂ = 7.7 Hz), 4.51 (m, 1H), 5.09 (dd, 1H, J₁ = 16.4 Hz, J₂ = 0.6 Hz), 5.13 (t, 1H, J = 3.3 Hz), 5.56 (dd, 1H, J₁ = 3.2 Hz, J₂ = 1.4 Hz), 6.85 (d, 1H, J = 16.5 Hz), 6.97 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 20.8, 20.8, 20.9, 61.0, 61.8, 66.3, 74.9, 92.1, 109.6, 118.6, 146.5, 152.8, 169.4, 170.0, 170.4; HRMS (ESI) m/z [M+Na]+ calcd for C₁₅H₁₇NNaO₇ 346.0897, found 346.0881.

(2R,3S,4R)-2-(Acetoxymethyl)-5-((E)-3-acetoxyprop-1-en-1-yl)-3,4-dihydro-2H-pyran-3,4-diyl diacetate (3v)
Prepared following the general procedure (Method B) using 1b (100 mg, 0.37 mmol), allyl acetate 2g (79 µL, 0.73 mmol), Pd(OAc)$_2$ (2.1 mg, 0.009 mmol), $p$-benzoquinone (2.0 mg, 0.018 mmol), Fe(Pc) (2.1 mg, 0.004 mmol) in AcOH/DMA (1/1, 0.50 mL). Flash chromatography (hexane/ethyl acetate: 7/3 to 6/4) afforded 3v (Conjugated:unconjugated = 86:14) (59 mg, 43%) as a colourless oil.

R$_f$ 0.31 (hexane/ethyl acetate: 6/4) ; $^1$H NMR (400 MHz, CDCl$_3$) δ 2.04 (s, 3H), 2.06 (s, 3H), 2.07 (s, 3H), 2.08 (s, 3H), 4.15 (dd, 1H, $J_1$ = 10.9 Hz, $J_2$ = 3.0 Hz), 4.36-4.45 (m, 2H), 4.49-4.59 (m, 2H), 5.12 (t, 1H, $J$ = 3.4 Hz), 5.42 (dt, 1H, $J_1$ = 15.8 Hz, $J_2$ = 6.4 Hz), 5.58 (m, 1H), 6.09 (d, 1H, $J$ = 15.9 Hz), 6.69 (s, 1H) ; $^{13}$C NMR (100 MHz, CDCl$_3$) δ 20.8, 20.9, 20.9, 21.1, 61.2, 63.7, 65.2, 67.0, 73.8, 109.5, 119.2, 129.9, 147.0, 169.6, 170.2, 170.5, 170.8 ; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{17}$H$_{22}$NaO$_9$ 393.1156, found 393.1171.

(E)-Diethyl (2-(1-methyl-6-oxo-1,6-dihydropyridin-3-yl)vinyl)phosphonate (3w)

Prepared following the general procedure (Method B) using 1j (50 mg, 0.46 mmol), diethyl vinylphosphonate 2h (141 µL, 0.92 mmol), Pd(OAc)$_2$ (2.6 mg, 0.011 mmol), $p$-benzoquinone (2.5 mg, 0.023 mmol), Fe(Pc) (2.6 mg, 0.023 mmol) in PivOH (0.60 g) for 36 h at 120 °C. Flash chromatography (MeOH/ethyl acetate: 5/95) afforded 3w (77 mg, 62%) as a yellow solid.

R$_f$ 0.57 (MeOH/ethyl acetate: 5/95) ; $^1$H NMR (400 MHz, CDCl$_3$) δ 1.31 (t, 6H, $J$ = 7.0 Hz), 3.51 (s, 3H), 4.08 (m, 4H), 5.88 (dd, 1H, $J_1$ = 17.3 Hz, $J_2$ = 16.3 Hz), 6.57 (d, 1H, $J$ = 9.5 Hz), 7.18 (dd, 1H, $J_1$ = 22.4 Hz, $J_2$ = 17.4 Hz), 7.41 (d, 1H, $J$ = 2.5 Hz), 7.54 (dd, 1H, $J_1$ = 9.6 Hz, $J_2$ = 2.6 Hz) ; $^{13}$C NMR (100 MHz, CDCl$_3$) δ 16.4 (d, $J$ = 6.4 Hz), 37.9, 61.8 (d, $J$ = 5.8 Hz), 110.5 (d, $J$ = 191 Hz), 115.1, 121.1, 135.9, 140.5, 143.5 (d, $J$ = 7.6 Hz), 162.6 ; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{12}$H$_{18}$NNaO$_4$P 294.0866, found 294.0831.

(E)-1-Methyl-5-(2-(phenylsulfonyl)vinyl)pyridin-2(1H)-one (3x)

Prepared following the general procedure (Method B) using 1j (50 mg, 0.46 mmol), phenyl vinyl sulfone 2i (154 mg, 0.92 mmol), Pd(OAc)$_2$ (2.6 mg, 0.011 mmol), $p$-benzoquinone (2.5 mg, 0.023 mmol), Fe(Pc) (2.6 mg, 0.023 mmol) in PivOH (0.60 g) for 36 h at 120 °C. Flash chromatography (MeOH/ethyl acetate: 0/10 to 2/98) afforded 3x (62 mg, 49%) as a yellow solid.
Rf 0.37 (ethyl acetate); ^1H NMR (400 MHz, CDCl₃) δ 3.55 (s, 3H), 6.55 (m, 2H), 7.42 (m, 2H), 7.51-7.61 (m, 3H), 7.89 (m, 2H); ^13C NMR (100 MHz, CDCl₃) δ 38.1, 112.4, 121.4, 124.3, 127.6, 129.5, 133.5, 136.1, 137.8, 140.9, 142.3, 162.4; HRMS (ESI) m/z [M+Na]^+ calcd for C₁₄H₁₃NNaO₃S 298.0508, found 298.0508.
$3c$, CDCl$_3$, 400 MHz
NGI-A-140tot 10 1 C:\Bruker\TOPSPIN nicolas

3d, CDCl₃, 400 MHz
$\text{3d, CDCl}_3, 100 \text{ MHz}$
NGI-A-081H 10 1 C:\Bruker\TOPSPIN nicolas

3f, CDCl₃, 400 MHz
NH$_2$

3h, DMSO, 100 MHz
3k, CDCl₃, 100 MHz
$3_p, \text{CDCl}_3, 100 \text{ MHz}$
$3^r$, CDCl$_3$, 100 MHz
$3v$, CDCl$_3$, 100 MHz