Supporting Information

SO$_2$F$_2$ mediated dehydrative cross-coupling of alcohols with electron-deficient olefins in DMSO using Pd-catalyst: one-pot transformation of alcohols to 1,3-dienes

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1. General considerations

All reactions were carried out in dried glassware. All reactions were carried out under Ar atmosphere. Unless otherwise stated, NMR spectra were recorded in CDCl$_3$ on a 500 MHz (for $^1$H), 126 MHz (for $^{13}$C) spectrometer. All chemical shifts were reported in ppm relative to TMS ($^1$H NMR, 0 ppm) as internal standards. All the yields mentioned were isolated. The coupling constants were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Melting points were measured and uncorrected. Unless otherwise noted, reagents and solvents used in this work were purchased from commercial sources and used as received.
2. Screening the optimized reaction conditions

Table 1. Screening the catalyst loading

<table>
<thead>
<tr>
<th>entry</th>
<th>Pd(OAc)$_2$ (mol%)</th>
<th>3a yield (%)$^b$</th>
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<tr>
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<td>0.1</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>0.5</td>
<td>70</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>77</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>74</td>
</tr>
</tbody>
</table>

$^a$Reaction condition: a mixture of 2-([1,1-biphenyl]-4-yl)ethanol (1a, 1.0 mmol, 1.0 eq.), K$_2$CO$_3$ (1.5 mmol, 1.5 eq.), DMSO (2.0 mL) was stirred at room temperature, charged with a SO$_2$F$_2$ balloon for 2 h. Then methylacrylate (2a, 2.0 mmol, 2.0 eq.), Pd(OAc)$_2$ and NaHCO$_3$ (2.0 mmol, 2.0 eq.) were added into the mixture to react for an additional 24 h under Ar atmosphere (an Ar balloon) at 50 °C. $^b$Isolated yields.

Table 2. Screening the reaction temperature

<table>
<thead>
<tr>
<th>entry</th>
<th>T (°C)</th>
<th>3a yield (%)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
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<tr>
<td>2</td>
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<tr>
<td>4</td>
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<td>54</td>
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</table>

$^a$Reaction condition: a mixture of 2-([1,1-biphenyl]-4-yl)ethanol (1a, 1.0 mmol, 1.0 eq.), K$_2$CO$_3$ (1.5 mmol, 1.5 eq.), DMSO (2.0 mL) was stirred at room temperature, charged with a SO$_2$F$_2$ balloon for 2 h. Then methylacrylate (2a, 2.0 mmol, 2.0 eq.), Pd(OAc)$_2$(1 mol%) and NaHCO$_3$ (2.0 mmol, 2.0 eq.) were added into the mixture to react for an additional 24 h under Ar atmosphere (an Ar balloon) at the corresponding temperature. $^b$Isolated yields.
Table 3. Screening the reaction time$^a$

\[
\begin{array}{ccc}
\text{entry} & \text{time (h)} & 3a \text{ yield (%)$^b$} \\
1 & 24 & 77 \\
2 & 10 & 91 \\
3 & 2 & 99 \\
4 & 1 & 81 \\
5 & 0.5 & 70 \\
\end{array}
\]

$^a$Reaction condition: a mixture of 2-([1,1-biphenyl]-4-yl)ethanol (1a, 1.0 mmol, 1.0 eq.), K$_2$CO$_3$ (1.5 mmol, 1.5 eq.), DMSO (2.0 mL) was stirred at room temperature, charged with a SO$_2$F$_2$ balloon for 2 h. Then methylacrylate (2a, 2.0 mmol, 2.0 eq.), Pd(OAc)$_2$(1 mol%) and NaHCO$_3$ (2.0 mmol, 2.0 eq.) were added to the mixture to react for an additional 0.5-24 h under Ar atmosphere (an Ar balloon) at 50 ℃. $^b$Isolated yields.

Table 4. Screening the baseloading$^a$

\[
\begin{array}{ccc}
\text{entry} & \text{NaHCO$_3$ (eq.)} & 3a \text{ yield (%)$^b$} \\
1 & 1 & 83 \\
2 & 1.5 & 90 \\
3 & 2 & 99 \\
\end{array}
\]

$^a$Reaction condition: a mixture of 2-([1,1-biphenyl]-4-yl)ethanol (1a, 1.0 mmol, 1.0 eq.), K$_2$CO$_3$ (1.5 mmol, 1.5 eq.), DMSO (2.0 mL) was stirred at room temperature, charged with a SO$_2$F$_2$ balloon for 2 h. Then methylacrylate (2a, 2.0 mmol, 2.0 eq.), Pd(OAc)$_2$(1 mol%) and NaHCO$_3$ were added to the mixture to react for an additional 2 h under Ar atmosphere (an Ar balloon) at 50 ℃. $^b$Isolated yields.
3. General procedure

**General procedure for the synthesis of 1,3-diene derivatives.**

To an oven-dried reaction flask (25 mL) with stirring bar charged with homobenzylic alcohol(1, 1.0 mmol, 1.0 eq.), K$_2$CO$_3$ (1.5 mmol, 1.5 eq.), the tube was then sealed with a septum, DMSO (2.0 mL) was added through syringe, and SO$_2$F$_2$ gas (sulfuryl fluoride) was introduced by needle from a balloon filled with the gas (degassed with SO$_2$F$_2$ for 10-30 seconds). The reaction mixture was vigorously stirred at room temperature for 2 h. When the alcohol was consumed (Monitoring by TLC), olefin (2, 2.0 mmol, 2.0 eq.), Pd(OAc)$_2$ (1 mol%), NaHCO$_3$ (2.0 mmol, 2.0 eq.) were added into the mixture (degassed with Argon for 10-30 seconds). And then the mixture was allowed to react for an additional 2 h under Argon atmosphere (balloon) at 50 °C. Subsequently, the mixture was diluted with water (50 mL) and extracted with EtOAc (3×10 mL). The combined organic layer was washed with brine (25 mL), dried over anhydrous Na$_2$SO$_4$, and concentrated to dryness. The crude residue of compounds 3 (ratio of E/Z and E/E about 1:1) was dissolved in MeCN (2.0 mL), I$_2$ (1.0 mmol) was added and the mixture was allowed to stir at room temperature for 3 h. Subsequently, the mixture was diluted with saturated Na$_2$S$_2$O$_3$ and water (50 mL) and extracted with EtOAc (3×10 mL). The combined organic layer was washed with brine (25 mL), dried over anhydrous Na$_2$SO$_4$, and concentrated to dryness. The residue was purified by silica gel chromatography through gradient elution with EtOAc / Petroleum ether to afford pure products (4) as 1-E-3-E-conjugated-dienes.
4. Product Characterization

(2E,4E)-methyl 5-([1,1'-biphenyl]-4-yl)penta-2,4-dienoate (4a). White solid (261 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1a and methyl acrylate 2a, isolated yield 99%). M.p. 148-150 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl₃) δ 7.62-7.60 (m, 4H), 7.54 (d, $J$ = 8.5 Hz, 2H), 7.51-7.44 (m, 3H), 7.37 (t, $J$ = 7.5 Hz, 1H), 6.93 (d, $J$ = 8.5 Hz, 2H). $^1$H NMR (500 MHz, CDCl₃) δ 6.02 (d, $J$ = 15 Hz, 1H), 6.02 (d, $J$ = 15 Hz, 1H), 3.79 (s, 3H).

(2E,4E)-methyl 5-([1,1'-biphenyl]-4-yl)penta-2,4-dienoate (4b). Pale yellow liquid (81 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1b and methyl acrylate 2a, isolated yield 40%). $^1$H NMR (500 MHz, CDCl₃) δ 7.56-7.54 (m, 1H), 7.50 (dd, $J$ = 15.5 Hz, $J$ = 11.5 Hz, 1H), 7.22-7.15 (m, 4H), 6.80 (dd, $J$ = 15 Hz, $J$ = 11 Hz, 1H), 6.00 (d, $J$ = 15.5 Hz, 1H), 3.78 (s, 3H), 2.39 (s, 3H). $^1$C NMR (126 MHz, CDCl₃) δ 167.6, 145.3, 138.3, 136.7, 135.0, 130.8, 129.0, 127.3, 126.4, 125.7, 120.8, 51.6, 19.8.

(2E,4E)-methyl 5-([1,1'-biphenyl]-4-yl)penta-2,4-dienoate (4b). Pale yellow liquid (137 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1c and methyl acrylate 2a, isolated yield 68%). $^1$H NMR (500 MHz, CDCl₃) δ 7.47-7.42 (m, 1H), 7.28-7.23 (m, 3H), 7.13 (d, $J$ = 6.5 Hz, 1H), 6.87-6.86 (m, 2H), 5.99 (d, $J$ = 15.5 Hz, 1H), 3.77 (s, 3H), 2.36 (s, 3H). $^1$C NMR (126 MHz, CDCl₃) δ 167.7, 145.1, 140.9, 138.6, 136.1, 130.1, 128.8, 128.0, 126.2, 124.6, 120.7, 51.7, 21.5.

(2E,4E)-methyl 5-(p-tolyl)penta-2,4-dienoate (4d). White solid (162 mg from 2-(p-tolyl)ethanol 1d and methyl acrylate 2a, isolated yield 80%). M.p. 37-38 °C. The NMR data is identical to that reported in literature. $^2$H NMR (500 MHz, CDCl₃) δ 7.45 (dd, $J$ = 15 Hz, $J$ = 15 Hz, 1H),
10 Hz, 1H), 7.36 (d, J = 8 Hz, 2H), 7.16 (d, J = 8 Hz, 2H), 6.87 (d, J = 15.5 Hz, 1H), 6.82 (dd, J = 15.5 Hz, J = 10 Hz, 1H), 5.97 (d, J = 15 Hz, 1H), 3.77 (s, 3H), 2.36 (s, 3H).

(2E,4E)-methyl 5-(2,5-dimethylphenyl)penta-2,4-dienoate (4e). Pale red liquid (120 mg from 2-(2,5-dimethylphenyl)ethanol 1e and methyl acrylate 2a, isolated yield 55%). 1H NMR (500 MHz, CDCl 3 ) δ 7.49 (ddd, J = 15 Hz, J = 11 Hz, J = 0.5 Hz, 1H), 7.36 (s, 1H), 7.14 (d, J = 15 Hz, 1H), 7.08-7.02 (m, 2H), 6.80 (dd, J = 15.5 Hz, J = 11 Hz, 1H), 6.00 (d, J = 15.5 Hz, 1H), 3.78 (s, 3H), 2.35 (s, 3H), 2.33 (s, 3H). 13C NMR (126 MHz, CDCl 3 ) δ 167.7, 145.4, 138.5, 135.8, 134.7, 133.7, 130.7, 129.9, 127.0, 126.3, 120.6, 51.7, 21.1, 19.3.

(2E,4E)-methyl 5-(4-(tert-butyl)phenyl)penta-2,4-dienoate (4f). Pale yellow solid (151 mg from 2-(4-(tert-butyl)phenyl)ethanol 1f and methyl acrylate 2a, isolated yield 62%). M.p. 88-90°C. 1H NMR (500 MHz, CDCl 3 ) δ 7.45 (dd, J = 15.5 Hz, J = 10.5 Hz, 1H), 7.43-7.37 (m, 4H), 6.90 (d, J = 15.5 Hz, 1H), 6.84 (dd, J = 15.5 Hz, J = 10 Hz, 1H), 5.98 (d, J = 15 Hz, 1H), 3.77 (s, 3H), 1.33 (s, 9H). 13C NMR (126 MHz, CDCl 3 ) δ 167.7, 152.7, 145.3, 140.7, 133.4, 127.2, 125.9, 125.6, 120.4, 51.6, 34.9, 31.3.

(2E,4E)-methyl 5-(3-(trifluoromethoxy)phenyl)penta-2,4-dienoate (4g). Yellow liquid (163 mg from 2-(3-(trifluoromethoxy)phenyl)ethanol 1g and methyl acrylate 2a, isolated yield 60%). 1H NMR (500 MHz, CDCl 3 ) δ 7.43 (ddd, J = 15 Hz, J = 7 Hz, J = 3 Hz, 1H), 7.38-7.37 (m, 2H), 7.30 (s, 1H), 7.16-7.14 (m, 1H), 6.88-6.86 (m, 2H), 6.04 (d, J = 15.5 Hz, 1H), 3.78 (s, 3H). 13C NMR (126 MHz, CDCl 3 ) δ 167.4, 149.9, 144.1, 138.7, 138.3, 130.3, 128.0, 125.7, 122.3, 121.6, 121.3, 119.4, 51.8.
(2E,4E)-methyl 5-(4-(trifluoromethoxy)phenyl)penta-2,4-dienoate (4h). White solid (175 mg from 2-(4-(trifluoromethoxy)phenyl)ethanol 1h and methyl acrylate 2a, isolated yield 64%). M.p. 74-76 °C. 1H NMR (500 MHz, CDCl₃) δ 7.47 (d, J = 8.5 Hz, 2H), 7.43 (dd, J = 15.5 Hz, J = 9.5 Hz, 1H), 7.19 (d, J = 8 Hz, 2H), 6.89-6.80 (m, 2H), 6.02 (d, J = 15 Hz, 1H), 3.77 (s, 3H). 13C NMR (126 MHz, CDCl₃) δ 167.4, 149.6, 149.6, 144.4, 138.7, 134.8, 128.6, 127.2, 121.7, 121.3, 51.7.

(2E,4E)-methyl 5-(4-methoxyphenyl)penta-2,4-dienoate (4i). Pale yellow solid (157 mg from 2-(4-methoxyphenyl)ethanol 1i and methyl acrylate 2a, isolated yield 72%). M.p. 116-118 °C. The NMR data is identical to that reported in literature. 1H NMR (500 MHz, CDCl₃) δ 7.46-7.39 (m, 3H), 6.89-6.83 (m, 3H), 6.74 (dd, J = 15.5 Hz, J = 10.5 Hz, 1H), 5.94 (d, J = 15 Hz, 1H), 3.82 (s, 3H), 3.76 (s, 3H).

(2E,4E)-methyl 5-(3,5-dimethoxyphenyl)penta-2,4-dienoate (4j). Pale yellow solid (149 mg from 2-(3,5-dimethoxyphenyl)ethanol 1j and methyl acrylate 2a, isolated yield 60%). M.p. 96-98 °C. 1H NMR (500 MHz, CDCl₃) δ 7.43 (dd, J = 15 Hz, J = 6.5 Hz, J = 3.5 Hz, 1H), 6.84-6.83 (m, 2H), 6.61 (d, J = 2 Hz, 2H), 6.43 (t, J = 2 Hz, 1H), 6.00 (d, J = 15.5 Hz, 1H), 3.81 (s, 6H), 3.77 (s, 3H). 13C NMR (126 MHz, CDCl₃) δ 167.6, 161.2, 144.7, 140.6, 138.1, 126.8, 121.2, 105.4, 101.6, 55.5, 51.7.

(2E,4E)-methyl 5-(3,4,5-trimethoxyphenyl)penta-2,4-dienoate (4k). Yellow liquid (139 mg from 2-(3,4,5-trimethoxyphenyl)ethanol 1k and methyl acrylate 2a, isolated yield 50%). 1H NMR (500 MHz, CDCl₃) δ 7.43 (dd, J = 15.5 Hz, J = 10 Hz, 1H), 6.84-6.78 (m, 2H), 6.68 (s, 2H), 5.99 (d, J = 15.5 Hz, 1H), 3.89 (s, 6H), 3.87 (s, 3H), 3.77 (s, 3H). 13C NMR (126 MHz, CDCl₃) δ 167.7, 153.6, 144.8, 140.6, 139.4, 131.8, 125.8, 120.7, 104.6, 61.1, 56.3, 51.7.
(2E,4E)-methyl 5-(2-(benzylxyloxy)phenyl)penta-2,4-dienoate (4l). Yellow liquid (182 mg from 2-(2-(benzylxyloxy)phenyl)ethanol 1l and methyl acrylate 2a, isolated yield 62%). $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.49 (dd, $J = 7.5$ Hz, $J = 1$ Hz, 1H), 7.41-7.36 (m, 5H), 7.33-7.29 (m, 2H), 7.22-7.20 (m, 1H), 6.94-6.90 (m, 3H), 5.89 (d, $J = 15.5$ Hz, 1H), 5.08 (s, 2H), 3.72 (s, 3H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 167.8, 156.8, 146.0, 136.8, 135.8, 130.3, 128.8, 128.2, 127.6, 127.5, 127.0, 125.5, 121.2, 120.2, 112.7, 70.5, 51.6.

(2E,4E)-methyl 5-(3-(benzylxyloxy)phenyl)penta-2,4-dienoate (4m). White solid (186 mg from 2-(3-(benzylxyloxy)phenyl)ethanol 1m and methyl acrylate 2a, isolated yield 63%). M.p. 89-91 $^\circ$C. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.44-7.37 (m, 5H), 7.34-7.32 (m, 1H), 7.26-7.25 (m, 1H), 7.07-7.06 (m, 2H), 6.94-6.92 (m, 1H), 6.85-6.84 (m, 2H), 5.99 (d, $J = 15$ Hz, 1H), 5.08 (s, 2H), 3.77 (s, 3H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 167.6, 159.3, 144.8, 140.5, 137.6, 136.9, 130.0, 128.8, 128.2, 127.6, 126.7, 121.1, 120.3, 115.8, 113.5, 70.2, 51.7.

(2E,4E)-methyl 5-(4-(methylthio)phenyl)penta-2,4-dienoate (4n). White solid (180 mg from 2-(4-(methylthio)phenyl)ethanol 1n and methyl acrylate 2a, isolated yield 77%). M.p. 101-103 $^\circ$C. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.43 (ddd, $J = 15$ Hz, $J = 8$ Hz, $J = 2$ Hz, 1H), 7.37 (d, $J = 8.5$ Hz, 2H), 7.21 (d, $J = 8.5$ Hz, 2H), 6.84-6.82 (m, 2H), 5.97 (d, $J = 15$ Hz, 1H), 3.77 (s, 3H), 2.49 (s, 3H). $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 167.6, 145.0, 140.3, 140.1, 132.9, 127.7, 126.4, 125.6, 120.5, 51.7, 15.5.
(2E,4E)-methyl 5-phenylpenta-2,4-dienoate (4o). White solid (133 mg from 2-phenylethanol 1o and methyl acrylate 2a, isolated yield 71%). M.p. 60-62 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.48-7.43 (m, 3H), 7.36 (t, $J = 7$ Hz, 2H), 7.30 (t, $J = 7$ Hz, 1H), 6.92-6.84 (m, 2H), 6.00 (d, $J = 15$ Hz, 1H), 3.77 (s, 3H).

![4p](image)

(2E,4E)-methyl 5-(4-fluorophenyl)penta-2,4-dienoate (4p). White solid (140 mg from 2-(4-fluorophenyl)ethanol 1p and methyl acrylate 2a, isolated yield 68%). M.p. 90-92 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.45-7.40 (m, 3H), 7.04 (t, $J = 9$ Hz, 2H), 6.85 (d, $J = 15.5$ Hz, 1H), 6.78 (dd, $J = 15.5$ Hz, $J = 10.5$ Hz, 1H), 5.98 (d, $J = 15$ Hz, 1H), 3.77 (s, 3H).

![4q](image)

(2E,4E)-methyl 5-(3-chlorophenyl)penta-2,4-dienoate (4q). Red solid (115 mg from 2-(3-chlorophenyl)ethanol 1q and methyl acrylate 2a, isolated yield 52%). M.p. 49-51 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.45-7.41 (m, 1H), 7.38 (dt, $J = 10.5$ Hz, $J = 4$ Hz, 2H), 7.32 (dt, $J = 9$ Hz, $J = 2$ Hz, 2H), 6.84-6.83 (m, 2H), 6.00 (d, $J = 15.5$ Hz, 1H), 3.77 (s, 3H).

![4r](image)

(2E,4E)-methyl 5-(4-chlorophenyl)penta-2,4-dienoate (4r). White solid (156 mg from 2-(4-chlorophenyl)ethanol 1r and methyl acrylate 2a, isolated yield 70%). M.p. 122-124 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.45-7.41 (m, 1H), 7.38 (dt, $J = 10.5$ Hz, $J = 4$ Hz, 2H), 7.32 (dt, $J = 9$ Hz, $J = 2$ Hz, 2H), 6.84-6.83 (m, 2H), 6.00 (d, $J = 15.5$ Hz, 1H), 3.77 (s, 3H).

![4s](image)
(2E,4E)-methyl 5-(3,4-dichlorophenyl)penta-2,4-dienoate (4s). Pale yellow solid (136 mg from 2-(3,4-dichlorophenyl)ethanol 1s and methyl acrylate 2a, isolated yield 53%). M.p. 114-115 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.53 (d, J = 2 Hz, 1H), 7.43-7.38 (m, 2H), 7.28 (d, J = 2 Hz, 1H), 6.84 (dd, J = 15.5 Hz, J = 10.5 Hz, 1H), 6.77 (d, J = 16 Hz, 1H), 6.03 (d, J = 15.5 Hz, 1H), 3.77 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 167.3, 144.0, 137.7, 136.2, 133.2, 132.9, 130.9, 128.8, 128.0, 126.3, 122.4, 51.8.

(2E,4E)-methyl 5-(2-bromophenyl)penta-2,4-dienoate (4t). Red solid (155 mg from 2-(2-bromophenyl)ethanol 1t and methyl acrylate 2a, isolated yield 58%). M.p. 60-62 °C. The NMR data is identical to that reported in literature. ¹H NMR (500 MHz, CDCl₃) δ 7.46-7.44 (m, 2H), 7.36-7.33 (m, 2H), 7.31-7.29 (m, 1H), 6.88-6.83 (m, 2H), 5.99 (d, J = 15 Hz, 1H), 3.76 (s, 3H).

(2E,4E)-methyl 5-(3-bromophenyl)penta-2,4-dienoate (4u). Red liquid (160 mg from 2-(3-bromophenyl)ethanol 1u and methyl acrylate 2a, isolated yield 60%). The NMR data is identical to that reported in literature. ¹H NMR (500 MHz, CDCl₃) δ 7.47-7.45 (m, 2H), 7.41 (ddd, J = 15 Hz, J = 8.5 Hz, J = 1 Hz, 1H), 7.31-7.29 (m, 1H), 6.89-6.81 (m, 2H), 6.00 (d, J = 15.5 Hz, 1H), 3.77 (s, 3H).

(2E,4E)-methyl 5-(4-bromophenyl)penta-2,4-dienoate (4v). White solid (165 mg from 2-(4-bromophenyl)ethanol 1v and methyl acrylate 2a, isolated yield 62%). M.p. 136-138 °C. The NMR data is identical to that reported in literature. ¹H NMR (500 MHz, CDCl₃) δ 7.47 (d, J = 8.5 Hz, 2H), 7.41 (ddd, J = 15 Hz, J = 8.5 Hz, J = 1 Hz, 1H), 7.31 (d, J = 8.5 Hz, 2H), 6.87-6.79 (m, 2H), 6.00 (d, J = 15.5 Hz, 1H), 3.77 (s, 3H).
(2E,4E)-methyl 5-(4-iodophenyl)penta-2,4-dienoate (4w). White solid (172 mg from 2-(4-iodophenyl)ethanol 1w and methyl acrylate 2a, isolated yield 55%). M.p. 66-68 °C. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.47-7.45 (m, 2H), 7.35 (t, J = 7 Hz, 2H), 7.30 (t, J = 7 Hz, 1H), 6.92-6.84 (m, 2H), 6.00 (d, J = 15 Hz, 1H), 3.77 (s, 3H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 167.6, 144.9, 140.7, 128.9, 127.3, 126.3, 120.9, 51.6.

(2E,4E)-methyl 5-(4-nitrophenyl)penta-2,4-dienoate (4x). Yellow solid (116 mg from 2-(4-nitrophenyl)ethanol 1x and methyl acrylate 2a, isolated yield 50%). M.p. 160-162 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl$_3$) δ 8.21 (d, J = 8.5 Hz, 2H), 7.59 (d, J = 8.5 Hz, 2H), 7.45 (ddd, J = 15.5 Hz, J = 9 Hz, J = 1.5 Hz, 1H), 6.93 (d, J = 15.5 Hz, 1H), 6.06 (d, J = 15 Hz, 1H), 3.79 (s, 3H).

(2E,4E)-methyl 5-(4-(trifluoromethyl)phenyl)penta-2,4-dienoate (4y). Pale yellow solid (159 mg from 2-(4-(trifluoromethyl)phenyl)ethanol 1y and methyl acrylate 2a, isolated yield 62%). M.p. 108-110 °C. The NMR data is identical to that reported in literature. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.60 (d, J = 8.5 Hz, 2H), 7.55 (d, J = 8.5 Hz, 2H), 7.45 (ddd, J = 15.5 Hz, J = 9 Hz, J = 1.5 Hz, 1H), 6.97-6.89 (m, 2H), 6.04 (d, J = 15 Hz, 1H), 3.78 (s, 3H).

(2E,4E)-methyl 5-(naphthalen-2-yl)penta-2,4-dienoate (4z). White solid (175 mg from 2-(naphthalen-2-yl)ethanol 1z and methyl acrylate 2a, isolated yield 74%). M.p. 100-101 °C. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.83-7.80 (m, 4H), 7.65 (dd, J = 8.5 Hz, J = 1.5 Hz, 1H), 7.54-7.48 (m, 3H), 7.07 (d, J = 15.5 Hz, 1H), 6.99 (dd, J = 15.5 Hz, J = 10.5 Hz, 1H), 6.04 (d, J = 15 Hz, 1H), 3.79 (s, 3H). $^{13}$C NMR (126 MHz, CDCl$_3$) δ 167.6, 145.0, 140.8, 133.8, 133.7, 133.6, 128.7, 128.4, 128.3, 127.9, 126.8, 126.7, 126.6, 123.5, 121.0, 51.7.
(2E,4E)-methyl 5-(naphthalen-1-yl)penta-2,4-dienoate (4aa). Yellow solid (156 mg from 2-(naphthalen-1-yl)ethanol 1aa and methyl acrylate 2a, isolated yield 65%). M.p. 48-49 °C. ^1H NMR (500 MHz, CDCl$_3$) $\delta$ 8.13 (d, $J = 8.5$ Hz, 1H), 7.87 (d, $J = 7.5$ Hz, 1H), 7.84 (d, $J = 8.5$ Hz, 1H), 7.74-7.70 (m, 2H), 7.61 (dd, $J = 15.5$ Hz, $J = 11$ Hz, 1H), 7.57-7.52 (m, 2H), 7.48 (t, $J = 8$ Hz, 1H), 6.96 (dd, $J = 15.5$ Hz, $J = 11$ Hz, 1H), 6.06 (d, $J = 15$ Hz, 1H), 3.81 (s, 3H). ^13C NMR (126 MHz, CDCl$_3$) $\delta$ 167.6, 145.1, 137.4, 133.8, 133.3, 131.2, 129.6, 128.9, 126.7, 126.2, 125.6, 124.3, 123.3, 121.2, 51.8.

(2E,4E)-methyl 5-(benzo[b]thiophen-3-yl)penta-2,4-dienoate (4ab). Red liquid (235 mg from 2-(benzo[b]thiophen-3-yl)ethanol 1ab and methyl acrylate 2a, isolated yield 96%). ^1H NMR (500 MHz, CDCl$_3$) $\delta$ 7.94 (d, $J = 8$ Hz, 1H), 7.88 (d, $J = 8$ Hz, 1H), 7.64 (s, 1H), 7.52 (dd, $J = 15.5$ Hz, $J = 11.5$ Hz, 1H), 7.47-7.44 (m, 1H), 7.42-7.38 (m, 1H), 7.19 (d, $J = 15$ Hz, 1H), 6.96 (dd, $J = 15.5$ Hz, $J = 11$ Hz, 1H), 6.03 (d, $J = 15$ Hz, 1H), 3.79 (s, 3H). ^13C NMR (126 MHz, CDCl$_3$) $\delta$ 167.7, 145.1, 140.6, 137.4, 133.2, 132.2, 127.4, 125.1, 124.9, 124.9, 123.2, 121.9, 120.9, 51.7.

(2E,4E)-methyl 5-(thiophen-2-yl)penta-2,4-dienoate (4ac). Red solid (134 mg from 2-(thiophen-2-yl)ethanol 1ac and methyl acrylate 2a, isolated yield 69%). M.p. 82-84°C. ^1H NMR (500 MHz, CDCl$_3$) $\delta$ 7.39 (d, $J = 15.5$ Hz, $J = 11$ Hz, 1H), 7.29 (d, $J = 5.5$ Hz, 1H), 7.11 (d, $J = 3.5$ Hz, 1H), 7.03-7.00 (m, 2H), 6.67 (dd, $J = 15$ Hz, $J = 11$ Hz, 1H), 5.96 (d, $J = 15$ Hz, 1H), 3.76 (s, 3H). ^13C NMR (126 MHz, CDCl$_3$) $\delta$ 167.6, 144.5, 141.5, 133.1, 128.6, 128.1, 126.9, 125.8, 120.5, 51.7.
(2E,4E)-methyl 5-(1-tosyl-1H-indol-3-yl)penta-2,4-dienoate (4ad). Red solid (190 mg from 2-(1-tosyl-1H-indol-3-yl)ethanol 1ad and methyl acrylate 2a, isolated yield 50%). M.p. 124-126 °C.1H NMR (500 MHz, CDCl3) δ 8.00 (d, J = 8.5 Hz, 1H), 7.79-7.75 (m, 4H), 7.45 (dd, J = 15.5 Hz, J = 10 Hz, 1H), 7.38-7.35 (m, 1H), 7.32-7.29 (m, 1H), 7.23 (d, J = 8 Hz, 2H), 6.98 (d, J = 15.5 Hz, 1H), 6.93 (dd, J = 16 Hz, J = 10 Hz, 1H), 5.99 (d, J = 15 Hz, 1H), 3.78 (s, 3H), 2.34 (s, 3H).13C NMR (126 MHz, CDCl3) δ 167.6, 145.5, 145.2, 135.7, 135.0, 131.3, 130.2, 128.5, 127.1, 127.1, 126.0, 125.5, 124.0, 120.5, 120.4, 119.8, 114.0, 51.7, 21.7.

(2E,4E)-ethyl 5-([1,1'-biphenyl]-4-yl)penta-2,4-dienoate (4ba). Pale yellow solid (214 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1a and ethyl acrylate 2b, isolated yield 77%). M.p. 106-108 °C. The NMR data is identical to that reported in literature.1H NMR (500 MHz, CDCl3) δ 7.62-7.60 (m, 4H), 7.54 (d, J = 8.5 Hz, 2H), 7.50-7.44 (m, 3H), 7.37 (t, J = 7.5 Hz, 1H), 6.96-6.89 (m, 2H), 6.01 (d, J = 15.5 Hz, 1H), 4.25 (q, J = 14.5 Hz, J = 7.5 Hz, 2H), 1.33 (t, J = 7.5 Hz, 3H).

(2E,4E)-phenyl 5-([1,1'-biphenyl]-4-yl)penta-2,4-dienoate (4ca). Pale yellow solid (277 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1a and phenyl acrylate 2c, isolated yield 85%). M.p. 124-126 °C.1H NMR (500 MHz, CDCl3) δ 7.64-7.62 (m, 4H), 7.58 (d, J = 8.5 Hz, 2H), 7.46 (t, J = 7.5 Hz, 3H), 7.42-7.38 (m, 3H), 7.24 (d, J = 7.5, 1H), 7.17-7.16 (m, 2H), 7.01 (d, J = 8.5 Hz, 2H), 6.20 (d, J = 15 Hz, 1H).13C NMR (126 MHz, CDCl3) δ 165.6, 151.0, 146.7, 142.2, 141.2, 140.4, 135.0, 129.5, 129.0, 128.0, 127.9, 127.7, 127.1, 126.2, 125.8, 121.8, 120.3.
(2E,4E)-benzyl 5-([1,1'-biphenyl]-4-yl)penta-2,4-dienoate (4da). White solid (296 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1a and benzyl acrylate 2d, isolated yield 87%). M.p. 124-125 °C. 1H NMR (500 MHz, CDCl₃) δ 7.62-7.60 (m, 4H), 7.55-7.50 (m, 3H), 7.47-7.35 (m, 8H), 6.97-6.89 (m, 2H), 6.07 (d, J = 15.5 Hz, 1H), 5.24 (s, 2H). 13C NMR (126 MHz, CDCl₃) δ 167.0, 145.3, 142.1, 140.4, 136.3, 135.1, 129.0, 128.7, 128.3, 127.8, 127.6, 127.1, 126.3, 121.0, 66.3.

(4E,6E)-7-([1,1'-biphenyl]-4-yl)hepta-4,6-dien-3-one (4ea). Pale yellow solid (231 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1a and pent-1-en-3-one 2e, isolated yield 88%). M.p. 112-114 °C. 1H NMR (500 MHz, CDCl₃) δ 7.62-7.60 (m, 4H), 7.54 (d, J = 8.5 Hz, 2H), 7.46 (t, J = 7.5 Hz, 2H), 7.39-7.34 (m, 2H), 6.98 (d, J = 15.5 Hz, 1H), 6.92 (dd, J = 15.5 Hz, J = 10 Hz, 1H), 6.31 (d, J = 15.5 Hz, 1H), 2.64 (q, J = 7.5 Hz, 2H), 1.16 (t, J = 7.5 Hz, 3H). 13C NMR (126 MHz, CDCl₃) δ 201.2, 142.4, 142.0, 140.8, 140.4, 135.2, 129.5, 129.0, 127.8, 127.6, 127.1, 126.9, 34.1, 8.4.

(2E,4E)-5-([1,1'-biphenyl]-4-yl)-N,N-dimethylpenta-2,4-dienamide (4fa). White solid (227 mg from 2-([1,1'-biphenyl]-4-yl)ethanol 1a and N,N-dimethylacrylamide 2f, isolated yield 82%). M.p. 146-148 °C. 1H NMR (500 MHz, CDCl₃) δ 7.61-7.58 (m, 4H), 7.52 (d, J = 8.5 Hz, 2H), 7.50-7.43 (m, 3H), 7.35 (t, J = 7 Hz, 1H), 6.96 (dd, J = 15.5 Hz, J = 10 Hz, 1H), 6.90 (d, J = 15.5 Hz, 1H), 6.48 (d, J = 14.5 Hz, 1H), 3.12 (s, 3H), 3.05 (s, 3H). 13C NMR (126 MHz, CDCl₃) δ 166.9, 142.6, 141.4, 140.5, 138.6, 135.6, 128.9, 127.7, 127.6, 127.5, 127.0, 120.8, 37.4, 36.0.
5. Reference


6. NMR Spectra

4a, 1H NMR, 500 MHz, CDCl₃

4b, 1H NMR, 500 MHz, CDCl₃
4b, -13C NMR, 126 MHz, CDCl$_3$

4c, -1H NMR, 500 MHz, CDCl$_3$
**4c**, $\text{-}^{13}$C NMR, 126 MHz, CDCl$_3$

![NMR spectrum of 4c](image)

**4d**, $\text{-}^1$H NMR, 500 MHz, CDCl$_3$

![NMR spectrum of 4d](image)
4e, -1H NMR, 500 MHz, CDCl$_3$

4e, -13C NMR, 126 MHz, CDCl$_3$
**4f. -1H NMR, 500 MHz, CDCl₃**

![1H NMR spectrum of 4f](image1)

**4f. -13C NMR, 126 MHz, CDCl₃**

![13C NMR spectrum of 4f](image2)
4g, -1H NMR, 500 MHz, CDCl₃

4g, -13C NMR, 126 MHz, CDCl₃
4h, -1H NMR, 500 MHz, CDCl$_3$

4h, -13C NMR, 126 MHz, CDCl$_3$
**4i.** 1H NMR, 500 MHz, CDCl$_3$

**4j.** 1H NMR, 500 MHz, CDCl$_3$
4j, -13C NMR, 126 MHz, CDCl$_3$

4k, -1H NMR, 500 MHz, CDCl$_3$
4k, -13C NMR, 126 MHz, CDCl$_3$

![NMR spectrum of 4k](image1.png)

COOMe
MeO

4k

4l, -1H NMR, 500 MHz, CDCl$_3$

![NMR spectrum of 4l](image2.png)

COOMe
Ph
4l, $^{13}$C NMR, 126 MHz, CDCl$_3$

4m, $^1$H NMR, 500 MHz, CDCl$_3$
**4m**, $^{13}$C NMR, 126 MHz, CDCl$_3$

![NMR spectrum of 4m](image)

**4n**, $^1$H NMR, 500 MHz, CDCl$_3$

![NMR spectrum of 4n](image)
4n, -13C NMR, 126 MHz, CDCl₃

4o, 1H NMR, 500 MHz, CDCl₃
4p. -1H NMR, 500 MHz, CDCl$_3$

4q. -1H NMR, 500 MHz, CDCl$_3$
4r, -1H NMR, 500 MHz, CDCl$_3$

4s, -1H NMR, 500 MHz, CDCl$_3$
4s, -13C NMR, 126 MHz, CDCl$_3$

4t, -1H NMR, 500 MHz, CDCl$_3$
4u, $^1$H NMR, 500 MHz, CDCl$_3$

![NMR spectrum of 4u](image)

4v, $^1$H NMR, 500 MHz, CDCl$_3$

![NMR spectrum of 4v](image)
$4w$, $\text{-}1\text{H NMR, 500 MHz, CDCl}_3$

$4w$, $\text{-}13\text{C NMR, 126 MHz, CDCl}_3$
4x, $^1$H NMR, 500 MHz, CDCl$_3$

4y, $^1$H NMR, 500 MHz, CDCl$_3$
4z, -1H NMR, 500 MHz, CDCl<sub>3</sub>

![1H NMR spectrum of 4z](image)

4z, -13C NMR, 126 MHz, CDCl<sub>3</sub>

![13C NMR spectrum of 4z](image)
4aa, -1H NMR, 500 MHz, CDCl₃

4aa, -13C NMR, 126 MHz, CDCl₃
4ab, -1H NMR, 500 MHz, CDCl₃

4ab, -13C NMR, 126 MHz, CDCl₃
4ac, -1H NMR, 500 MHz, CDCl$_3$

4ac, -13C NMR, 126 MHz, CDCl$_3$
**4ad.** -1H NMR, 500 MHz, CDCl$_3$

![1H NMR spectrum of 4ad](image)

**4ad.** -13C NMR, 126 MHz, CDCl$_3$

![13C NMR spectrum of 4ad](image)
4ba, -1H NMR, 500 MHz, CDCl$_3$

\[ \text{Ph} \quad 4ba \]

4ca, -1H NMR, 500 MHz, CDCl$_3$

\[ \text{Ph} \quad 4ca \]
4ca, ^{13}C NMR, 126 MHz, CDCl$_3$

4da, ^1H NMR, 500 MHz, CDCl$_3$
4da, -13C NMR, 126 MHz, CDCl₃

![Chemical structure of 4da]

4ea, -1H NMR, 500 MHz, CDCl₃

![Chemical structure of 4ea]
4ea, -13C NMR, 126 MHz, CDCl₃

4fa, -1H NMR, 500 MHz, CDCl₃
$4fa$, -13C NMR, 126 MHz, CDCl$_3$