Supporting Information

Copper-catalyzed 1,1-arylalkylation of terminal alkynes with diazo esters and organoboronic acids

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1. General Information

All deuterated solvents were purchased from Cambridge Isotope Laboratories. $^1$H NMR and $^{13}$C NMR spectra were recorded at 25 ºC on a Brüker Advance 400 spectrometer ($^1$H: 400 MHz and $^{13}$C:100 MHz). $^1$H NMR chemical shifts were determined relative to internal (CH$_3$)$_4$Si (TMS) at $\delta$ 0.00 ppm or to the signal of the residual protonated solvent: CDCl$_3$ at $\delta$ 7.26 ppm. $^{13}$C NMR chemical shifts were determined relative to the signal of the solvent: CDCl$_3$ at $\delta$ 77.00 ppm. Data for $^1$H, $^{13}$C NMR were recorded as follows: chemical shift ($\delta$, ppm), multiplicity ($s$ = singlet, $d$ = doublet, $t$ = triplet, $m$ = multiplet, $q$ = quartet, $dd$ = doublet of doublets), coupling constants (Hz) and integration. Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. High-resolution mass data were recorded on a Waters LCT PremierxeTM (USA). Single-crystal X-ray crystallography was carried out on a Bruker Smart Apex II diffractometer system.

Materials and Methods:

Unless otherwise stated, starting materials were purchased from Aldrich or Energy-Chemical Limited and used as supplied without further purification. Solvents were used directly without further purification. The following starting materials were prepared according to the procedures described previously in the literature: $2b$,[1] $2c$,[2] $2d$,[2] $4a'$,[3] and $4a''$.[4] The 1,1-arylalkylation of alkynes were performed under a nitrogen atmosphere in flame dried flasks. All reactions were monitored by thin layer chromatography (TLC) with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 200-300 mesh silica gel at increased pressure.
2. General Procedure for 1,1-Arylalkylation of Terminal Alkynes

4a as an example

\[
\begin{align*}
\text{Cul (10 mol\%)} & \quad \text{Phen (10 mol\%)} \\
\text{K}_3\text{PO}_4 (2.0 equiv) & \quad 1,4\text{-dioxane, 100 °C}
\end{align*}
\]

To a solution of the ethyl 2-diazoacetate 2a (33 μL, 0.3 mmol) in 1,4-dioxane (1.0 mL) was added the ethynylbenzene 1a (41 μL, 0.36 mmol), p-tolylboronic acid 3a (124.9 mg, 0.9 mmol), Phen (5.4 mg, 0.03 mmol), CuI (5.7 mg, 0.03 mmol), and K$_3$PO$_4$ (130.0 mg, 0.6 mmol) under a N$_2$ atmosphere in a Schlenck tube. The reaction mixture was stirred at 100 °C for 1.5 h. After the reaction finished, the reaction mixture was cooled to room temperature and quenched by water. The mixture was extracted with EtOAc (3.0 mL×3), the combined organic phases were dried over anhydrous Na$_2$SO$_4$ and the solvent was evaporated under vacuum. The residue was purified by column chromatography to give the corresponding products 4a (71.0 mg, 85%).

3. Synthetic Applications

3.1 Hydrolysis of Product 4a

Compound 4a (56.0 mg, 0.2 mmol) was taken in a Schlenck tube in EtOH (5 mL) with NaOH (0.4 g, 10 mmol). Schlenck tube was tightly closed with teflon cap and heated at 110 °C for 6 h. After completion of the reaction, the mixture was cooled to room temperature. Then 2N HCl was added to the aqueous layer until pH = 2. Next, the aqueous layer was extracted with EtOAc (2 x 20 mL). The organic layer was collected and dried over MgSO$_4$. After concentration in vacuum, the residue was purified by column chromatography to give the corresponding products 6 (45.0 mg, 89%).
3.2 Synthesis of 7

7a as an example

Following the general procedure described previously in the literature,[5] (E)-ethyl 4-phenyl-3-(p-tolyl)but-3-enoate 4a (84 mg, 0.3 mmol) was dissolved in conc. H\textsubscript{2}SO\textsubscript{4} (300 mg) and stirred at room temperature for 2 h. The reaction mixture was carefully poured over cold water (2 mL). Next, the aqueous layer was extracted with EtOAc (3 x 5 mL), and the product 7a (52 mg, 74\%) was isolated by flash chromatography.

3.3 Synthesis of 9

Following the abovementioned general procedure in S2, the reaction with 8 (70 mg, 0.36 mmol), 2a (33 \(\mu\)L, 0.3 mmol), 3a (124.9 mg, 0.9 mmol), Phen (5.4 mg, 0.03 mmol), Cul (5.7 mg, 0.03 mmol), and K\textsubscript{3}PO\textsubscript{4} (130.0 mg, 0.6 mmol) under a N\textsubscript{2} atmosphere for 1.5 h at 100 °C afforded 9 as colorless oil (85 mg, 76\% yield).
4. Analytical Data of Compounds 4-7 and 9

(E)-ethyl 4-phenyl-3-(p-tolyl)but-3-enoate 4a

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.18 (t, $J$ = 7.2 Hz, 3H), 2.38 (s, 3H), 3.71 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 7.02 (s, 1H), 7.19 (d, $J$ = 7.6 Hz, 2H), 7.28-7.43 (m, 7H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 21.1, 36.6, 60.7, 126.0, 127.0, 128.4, 128.7, 129.1, 130.4, 134.5, 137.3, 137.5, 138.7, 171.6. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{21}$O$_2$, [M+H]$^+$ m/z 281.1542, Found 281.1537.

(E)-ethyl 3,4-di-p-tolylbut-3-enoate 4b

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.19 (t, $J$ = 7.2 Hz, 3H), 2.37 (s, 3H), 2.38 (s, 3H), 3.71 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 6.99 (s, 1H), 7.18 (d, $J$ = 5.2 Hz, 2H), 7.20 (d, $J$ = 5.2 Hz, 2H), 7.29 (d, $J$ = 8.0 Hz, 2H), 7.41 (d, $J$ = 8.0 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 21.1, 21.2, 36.6, 60.7, 126.0, 128.6, 129.1, 130.4, 133.8, 134.6, 136.8, 137.2, 138.9, 171.7. HRMS (ESI-TOF). Calcd for C$_{20}$H$_{23}$O$_2$, [M+H]$^+$ m/z 295.1698, Found 295.1688.
(E)-ethyl 4-(4-ethylphenyl)-3-(p-tolyl)but-3-enoate 4c

Colorless oil. \( ^1 \text{H NMR} (400 \text{ MHz, } \text{CDCl}_3): \delta = 1.18 (t, J = 7.2 \text{ Hz, } 3 \text{H}), 1.26 (t, J = 7.6 \text{ Hz, } 3 \text{H}), 2.37 (s, 3 \text{H}), 2.67 (q, J = 7.6 \text{ Hz, } 2 \text{H}), 3.71 (s, 2 \text{H}), 4.11 (q, J = 7.2 \text{ Hz, } 2 \text{H}), 6.98 (s, 1 \text{H}), 7.17 (d, J = 8.0 \text{ Hz, } 2 \text{H}), 7.21 (d, J = 8.0 \text{ Hz, } 2 \text{H}), 7.31 (d, J = 8.0 \text{ Hz, } 2 \text{H}), 7.40 (d, J = 8.0 \text{ Hz, } 2 \text{H}); ^{13} \text{C NMR} (100 \text{ MHz, } \text{CDCl}_3): \delta = 14.1, 15.5, 21.1, 28.6, 36.7, 60.7, 126.0, 127.9, 128.7, 129.1, 130.4, 133.9, 134.9, 137.2, 139.0, 143.2, 171.7. \) HRMS (ESI-TOF). Caled for \( \text{C}_{21}\text{H}_{24}\text{NaO}_2, [\text{M}+\text{Na}]^+ \text{ m/z 331.1674, Found 331.1660.} \)

(E)-ethyl 4-(4-propylphenyl)-3-(p-tolyl)but-3-enoate 4d

Colorless oil. \( ^1 \text{H NMR} (400 \text{ MHz, } \text{CDCl}_3): \delta = 0.98 (t, J = 7.2 \text{ Hz, } 3 \text{H}), 1.19 (t, J = 7.2 \text{ Hz, } 3 \text{H}), 1.65-1.70 (m, 2 \text{H}), 2.37 (s, 3 \text{H}), 2.62 (t, J = 7.6 \text{ Hz, } 2 \text{H}), 3.73 (s, 2 \text{H}), 4.12 (q, J = 7.2 \text{ Hz, } 2 \text{H}), 6.99 (s, 1 \text{H}), 7.18 (d, J = 6.4 \text{ Hz, } 2 \text{H}), 7.20 (d, J = 6.4 \text{ Hz, } 2 \text{H}), 7.31 (d, J = 8.0 \text{ Hz, } 2 \text{H}), 7.41 (d, J = 8.0 \text{ Hz, } 2 \text{H}); ^{13} \text{C NMR} (100 \text{ MHz, } \text{CDCl}_3): \delta = 13.8, 14.1, 21.1, 24.5, 36.6, 37.8, 60.7, 126.0, 128.5, 128.6, 129.1, 130.4, 133.8, 134.9, 137.2, 138.9, 141.6, 171.7. \) HRMS (ESI-TOF). Caled for \( \text{C}_{22}\text{H}_{27}\text{O}_2, [\text{M}+\text{H}]^+ \text{ m/z 323.2011, Found 323.1998.} \)
(E)-ethyl 4-(4-butylphenyl)-3-(p-tolyl)but-3-enoate 4e

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 0.95 (t, $J$ = 7.2 Hz, 3H), 1.18 (t, $J$ = 7.2 Hz, 3H), 1.34-1.43 (m, 2H), 1.58-1.66 (m, 2H), 2.37 (s, 3H), 2.63 (t, $J$ = 7.6 Hz, 2H), 3.72 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 6.99 (s, 1H), 7.17-7.20 (m, 4H), 3.72 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 6.99 (s, 1H), 7.17-7.20 (m, 4H), 7.30 (d, $J$ = 8.0 Hz, 2H), 7.40 (d, $J$ = 8.4 Hz, 2H);

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.0, 14.1, 16.1, 21.1, 22.4, 33.6, 35.4, 36.7, 60.7, 126.0, 128.4, 128.7, 129.1, 130.4, 133.8, 134.8, 137.2, 138.9, 141.9, 171.7. HRMS (ESI-TOF). Caled for C$_{23}$H$_{29}$O$_2$, [M+H]$^+$ m/z 337.2168, Found 337.2168.

(E)-ethyl 4-(4-(tert-butyl)phenyl)-3-(p-tolyl)but-3-enoate 4f

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.18 (t, $J$ = 7.2 Hz, 3H), 1.35 (s, 9H), 2.37 (s, 3H), 3.73 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 6.99 (s, 1H), 7.18 (d, $J$ = 8.0 Hz, 2H), 7.34 (d, $J$ = 8.4 Hz, 2H), 7.39-7.42 (m, 4H);

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 21.1, 31.3, 34.6, 36.7, 60.7, 125.3, 126.0, 128.5, 129.1, 130.3, 133.9, 134.6, 137.2, 138.9, 150.0, 171.7. HRMS (ESI-TOF). Caled for C$_{23}$H$_{29}$O$_2$, [M+H]$^+$ m/z 337.2168, Found 337.2170.
(E)-ethyl 4-(4-pentylphenyl)-3-(p-tolyl)but-3-enolate 4g

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 0.91$ (t, $J = 6.4$ Hz, 3H), 1.18 (t, $J = 7.2$ Hz, 3H), 1.28-1.36 (m, 4H), 1.59-1.66 (m, 2H), 2.37 (s, 3H), 2.62 (t, $J = 7.6$ Hz, 2H), 3.72 (s, 2H), 4.12 (t, $J = 7.2$ Hz, 2H), 6.99 (s, 1H), 7.15-7.20 (m, 4H), 7.30 (d, $J = 7.6$ Hz, 2H), 7.40 (d, $J = 7.6$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.0$, 14.1, 21.1, 22.5, 31.1, 31.5, 35.7, 36.7, 60.7, 126.0, 128.4, 128.7, 129.1, 130.4, 133.8, 134.8, 137.2, 139.0, 141.9, 171.7. HRMS (ESI-TOF). Calcd for C$_{24}$H$_{30}$NaO$_2$, [M+Na]$^+$ m/z 373.2143, Found 373.2135.

(E)-ethyl 4-(4-fluorophenyl)-3-(p-tolyl)but-3-enolate 4h

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.18$ (t, $J = 7.2$ Hz, 3H), 2.37 (s, 3H), 3.65 (s, 2H), 4.11 (q, $J = 7.2$ Hz, 2H), 6.95 (s, 1H), 7.06 (t, $J = 8.8$ Hz, 2H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.34-7.40 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.1, 36.5, 60.8, 115.3 (d, $J = 21.0$ Hz), 126.0, 129.2, 129.3, 130.4 (d, $J = 8.0$ Hz), 133.6 (d, $J = 4.0$ Hz), 134.6, 137.5, 138.5, 161.9 (d, $J = 245.0$ Hz), 171.5. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$FO$_2$, [M+H]$^+$ m/z 299.1447, Found 299.1438.
(E)-ethyl 4-(4-chlorophenyl)-3-(p-tolyl)but-3-enolate 4i

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.18$ (t, $J = 7.2$ Hz, 3H), 2.37 (s, 3H), 3.65 (s, 2H), 4.11 (q, $J = 7.2$ Hz, 2H), 6.94 (s, 1H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.31-7.36 (m, 4H), 7.39 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.1, 36.6, 60.9, 126.1, 128.6, 129.1, 129.2, 130.1, 132.9, 135.2, 136.0, 137.6, 138.4, 171.4. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$ClO$_2$, [M+H]$^+$ m/z 315.1152, Found 315.1148.

(4)-ethyl 4-(4-bromophenyl)-3-(p-tolyl)but-3-enolate 4j

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.16$ (t, $J = 7.2$ Hz, 3H), 2.35 (s, 3H), 3.63 (s, 2H), 4.10 (q, $J = 7.2$ Hz, 2H), 6.90 (s, 1H), 7.17 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.37 (d, $J = 8.0$ Hz, 2H), 7.48 (d, $J = 8.4$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.1, 36.5, 60.8, 121.0, 126.0, 129.1, 129.2, 130.4, 131.5, 135.2, 136.4, 137.6, 138.3, 171.3. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$BrO$_2$, [M+H]$^+$ m/z 359.0647, 361.0626, Found 359.0638, 361.0614.
(E)-ethyl 4-(3-fluorophenyl)-3-(p-toly1)but-3-enoate 4k

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.38 (s, 3H), 3.69 (s, 2H), 4.13 (q, $J = 7.2$ Hz, 2H), 6.96 (s, 1H), 6.98-7.01 (m, 1H), 7.11-7.16 (m, 2H), 7.19 (d, $J = 8.0$ Hz, 2H), 7.31-7.37 (m, 1H), 7.40 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.0$, 21.1, 36.6, 60.9, 113.9 (d, $J = 21.0$ Hz), 115.5 (d, $J = 22.0$ Hz), 124.5 (d, $J = 3.0$ Hz), 126.1, 129.1 (d, $J = 2.0$ Hz), 129.2, 129.8 (d, $J = 8.0$ Hz), 135.6, 137.7, 138.3, 139.7 (d, $J = 7.0$ Hz), 162.7 (d, $J = 244.0$ Hz), 171.3. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$FO$_2$, [M+H]$^+$ $m/z$ 299.1447, Found 299.1451.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.38 (s, 3H), 3.69 (s, 2H), 4.13 (q, $J = 7.2$ Hz, 2H), 6.96 (s, 1H), 6.98-7.01 (m, 1H), 7.11-7.16 (m, 2H), 7.19 (d, $J = 8.0$ Hz, 2H), 7.31-7.37 (m, 1H), 7.40 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.0$, 21.1, 36.6, 60.9, 113.9 (d, $J = 21.0$ Hz), 115.5 (d, $J = 22.0$ Hz), 124.5 (d, $J = 3.0$ Hz), 126.1, 129.1 (d, $J = 2.0$ Hz), 129.2, 129.8 (d, $J = 8.0$ Hz), 135.6, 137.7, 138.3, 139.7 (d, $J = 7.0$ Hz), 162.7 (d, $J = 244.0$ Hz), 171.3. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$FO$_2$, [M+H]$^+$ $m/z$ 299.1447, Found 299.1451.

(E)-ethyl 4-(3-chlorophenyl)-3-(p-toly1)but-3-enoate 4l

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.36 (s, 3H), 3.65 (s, 2H), 4.11 (q, $J = 7.2$ Hz, 2H), 6.92 (s, 1H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.24-7.32 (m, 3H), 7.37-7.39 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.1, 36.7, 60.9, 126.1, 126.9, 127.1, 128.8, 128.9, 129.2, 129.6, 134.3, 135.8, 137.7, 138.3, 139.4, 171.2. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$ClO$_2$, [M+H]$^+$ $m/z$ 315.1152, Found 315.1141.
(E)-ethyl 4-(2-fluorophenyl)-3-(p-tolyl)but-3-enoate 4m

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.16 (t, $J$ = 7.2 Hz, 3H), 2.37 (s, 3H), 3.65 (s, 2H), 4.09 (q, $J$ = 7.2 Hz, 2H), 6.95 (s, 1H), 7.06-7.15 (m, 2H), 7.18 (d, $J$ = 8.0 Hz, 2H), 7.26-7.30 (m, 1H), 7.41 (d, $J$ = 8.0 Hz, 2H), 7.44-7.47 (m, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.0, 21.1, 36.8, 60.8, 115.4 (d, $J$ = 22.0 Hz), 123.1 (d, $J$ = 4.0 Hz), 123.9 (d, $J$ = 4.0 Hz), 125.2 (d, $J$ = 15.0 Hz), 126.1, 128.3, 129.0 (d, $J$ = 8.0 Hz), 129.1, 130.3 (d, $J$ = 3.0 Hz), 136.5, 137.9 (d, $J$ = 65.0 Hz), 160.5 (d, $J$ = 246.0 Hz), 171.3. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$FO$_2$, [M+H]$^+$ m/z 299.1447, Found 299.1438.

(E)-ethyl 4-(2-chlorophenyl)-3-(p-tolyl)but-3-enoate 4n

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.16 (t, $J$ = 7.2 Hz, 3H), 2.37 (s, 3H), 3.60 (s, 2H), 4.09 (q, $J$ = 7.2 Hz, 2H), 7.02 (s, 1H), 7.19 (d, $J$ = 8.0 Hz, 2H), 7.22-7.33 (m, 2H), 7.41-7.44 (m, 3H), 7.51 (dd, $J_1$ = 1.6 Hz, $J_2$ = 6.8 Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.0, 21.1, 36.7, 60.8, 126.2, 126.6, 127.6, 128.6, 129.2, 129.4, 130.3, 134.3, 135.7, 135.9, 137.7, 138.1, 171.4. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{20}$ClO$_2$, [M+H]$^+$ m/z 315.1152, Found 315.1149.
(E)-ethyl 5-((tert-butoxycarbonyl)amino)-3-(p-tolyl)pent-3-enoate 4o

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.22$ (t, $J = 7.2$ Hz, 3H), 1.45 (s, 9H), 2.33 (s, 3H), 3.53 (s, 3H), 3.92 (t, $J = 5.6$ Hz, 2H), 4.12 (q, $J = 7.2$ Hz, 2H), 4.86 (s, 1H), 5.97 (t, $J = 6.8$ Hz, 1H), 7.12 (d, $J = 8.0$ Hz, 2H), 7.26 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.0, 28.4, 36.0, 38.9, 61.0, 79.3, 125.8, 127.2, 129.1, 135.3, 137.3, 138.4, 155.8, 171.1. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{28}$NO$_4$, [M+H]$^+$ m/z 334.2018, Found 334.2032.

(E)-ethyl 5-methoxy-3-(p-tolyl)pent-3-enoate 4p

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.33 (s, 3H), 3.39 (s, 3H), 3.52 (s, 2H), 4.10 (q, $J = 7.2$ Hz, 2H), 4.16 (d, $J = 6.4$ Hz, 2H), 6.07 (t, $J = 6.4$ Hz, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.0, 36.4, 58.2, 60.8, 69.4, 125.9, 127.5, 129.0, 135.4, 137.2, 138.5, 170.9. HRMS (ESI-TOF). Calcd for C$_{15}$H$_{20}$NaO$_3$, [M+Na]$^+$ m/z 271.1310, Found 271.1303.
(E)-ethyl 5-(oxiran-2-ylmethoxy)-3-(p-tolyl)pent-3-enoate 4q

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.33 (s, 3H), 2.63 (dd, $J_1 = 2.8$ Hz, $J_2 = 4.8$ Hz, 1H), 2.81 (t, $J = 3.6$ Hz, 1H), 3.17-3.19 (m, 1H), 3.44 (dd, $J_1 = 6.0$ Hz, $J_2 = 11.2$ Hz, 1H), 3.53 (s, 2H), 3.79 (dd, $J_1 = 2.8$ Hz, $J_2 = 11.2$ Hz, 1H), 4.10 (q, $J = 7.2$ Hz, 2H), 4.27-4.30 (m, 2H), 6.08 (t, $J = 6.4$ Hz, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.0, 36.4, 44.2, 50.8, 60.8, 68.1, 70.9, 125.9, 127.1, 129.0, 135.7, 137.3, 138.4, 170.8. HRMS (ESI-TOF). Calcd for C$_{17}$H$_{22}$NaO$_4$, [M+Na]$^+$ m/z 313.1416, Found 313.1418.

(E)-ethyl 5-(2-hydroxyethoxy)-3-(p-tolyl)pent-3-enoate 4r

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.27 (s, 1H), 2.34 (s, 3H), 3.53 (s, 2H), 3.62 (t, $J = 4.4$ Hz, 2H), 3.76 (t, $J = 4.4$ Hz, 2H), 4.10 (q, $J = 7.2$ Hz, 2H), 4.26 (d, $J = 6.4$ Hz, 2H), 6.09 (t, $J = 6.4$ Hz, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.1, 36.5, 60.9, 61.9, 68.0, 71.6, 125.9, 127.1, 129.1, 136.0, 137.4, 138.4, 171.0. HRMS (ESI-TOF). Calcd for C$_{16}$H$_{23}$O$_4$, [M+H]$^+$ m/z 279.1596, Found 279.1589.
(E)-ethyl 4-cyclopropyl-3-(p-tolyl)but-3-enoate 4s

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 0.48$-$0.51$ (m, 2H), 0.85-$0.87$ (m, 2H), 1.21 (t, $J = 7.2$ Hz, 3H), 1.60-$1.67$ (m, 1H), 2.32 (s, 3H), 3.62 (s, 2H), 4.13 (q, $J = 7.2$ Hz, 2H), 5.32 (d, $J = 9.6$ Hz, 1H), 7.10 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.4$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 7.5$, 11.3, 21.0, 36.4, 60.6, 125.4, 128.9, 130.7, 135.7, 136.2, 139.2, 171.7. HRMS (ESI-TOF). Calcd for C$_{16}$H$_{21}$O$_2$, [M+H]$^+$ m/z 245.1542, Found 245.1538.

(E)-ethyl 3-(p-tolyl)hept-3-enoate 4t

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 0.96$ (t, $J = 7.2$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H), 1.47-$1.52$ (m, 2H), 2.19 (q, $J = 7.2$ Hz, 2H), 2.32 (s, 3H), 3.49 (s, 2H), 4.09 (q, $J = 7.2$ Hz, 2H), 5.91 (t, $J = 7.2$ Hz, 1H), 7.11 (d, $J = 8.0$ Hz, 2H), 7.27 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 13.9$, 14.1, 21.0, 22.7, 31.0, 36.1, 60.6, 125.8, 128.9, 131.8, 132.3, 136.4, 139.6, 171.5. HRMS (ESI-TOF). Calcd for C$_{16}$H$_{23}$O$_2$, [M+H]$^+$ m/z 247.1698, Found 247.1690.

(E)-ethyl 3-(p-tolyl)oct-3-enoate 4u
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): δ = 0.92 (t, J = 7.2 Hz, 3H), 1.18 (t, J = 7.2 Hz, 3H), 1.35-1.47 (m, 4H), 2.21 (q, J = 7.2 Hz, 2H), 2.32 (s, 3H), 3.49 (s, 2H), 4.09 (q, J = 7.2 Hz, 2H), 5.91 (t, J = 7.2 Hz, 1H), 7.11 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ = 14.0, 14.1, 21.0, 22.4, 28.7, 31.6, 36.0, 60.6, 125.8, 128.9, 132.0, 132.0, 136.4, 139.5, 171.6. HRMS (ESI-TOF). Calcd for C$_{17}$H$_{25}$O$_2$, [M+H]$^+$ m/z 261.1855, Found 261.1846.

![Structure 1](images/structure1.png)

(E)-ethyl 3-(4-ethylphenyl)-4-phenylbut-3-enoate 5a

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): δ = 1.17 (t, J = 7.2 Hz, 3H), 3.72 (s, 2H), 4.12 (q, J = 7.2 Hz, 2H), 7.04 (s, 1H), 7.28-7.40 (m, 8H), 7.51-7.53 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ = 14.0, 36.7, 60.7, 126.3, 127.2, 127.5, 128.4, 128.7, 131.2, 134.7, 137.4, 141.7, 171.5. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{19}$O$_2$, [M+H]$^+$ m/z 267.1385, Found 267.1378.

![Structure 2](images/structure2.png)

(E)-ethyl 3-(4-ethylphenyl)-4-phenylbut-3-enoate 5b

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): δ = 1.17 (t, J = 7.2 Hz, 3H), 1.26 (t, J = 7.6 Hz, 3H), 2.67 (q, J = 7.6 Hz, 2H), 3.70 (s, 2H), 4.11 (q, J = 7.2 Hz, 2H), 7.02 (s, 1H), 7.20 (d, J = 8.4 Hz, 2H), 7.27-7.39 (m, 5H), 7.43 (d, J = 8.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ = 14.1, 15.5, 28.5, 36.7, 60.7, 126.1, 127.1, 127.9, 128.4, 128.7, 130.5, 134.5, 137.6, 139.0, 143.7, 171.6. HRMS (ESI-TOF). Calcd for C$_{20}$H$_{23}$O$_2$, [M+H]$^+$ m/z 295.1698, Found 295.1701.
\( (E) \)-ethyl 4-phenyl-3-(4-propylphenyl)but-3-enoate 5c

Colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta = 0.96 \) (t, \( J = 7.2 \) Hz, 3H), 1.16 (t, \( J = 7.2 \) Hz, 3H), 1.63-1.69 (m, 2H), 2.60 (t, \( J = 7.6 \) Hz, 2H), 3.70 (s, 2H), 4.11 (q, \( J = 7.2 \) Hz, 2H), 7.03 (s, 1H), 7.18 (d, \( J = 8.0 \) Hz, 2H), 7.27-7.39 (m, 5H), 7.42 (d, \( J = 8.4 \) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta = 13.8, 14.1, 24.5, 36.6, 37.7, 60.7, 126.0, 127.0, 128.4, 128.5, 128.7, 130.4, 134.5, 137.6, 139.0, 142.2, 171.6 \). HRMS (ESI-TOF). Calcd for C\(_{21}\)H\(_{25}\)O\(_2\), [M+H]\(^+\) \( m/z \) 309.1855, Found 309.1847.

\( (E) \)-ethyl 3-(4-isopropylphenyl)-4-phenylbut-3-enoate 5d

Colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta = 1.15 \) (t, \( J = 7.2 \) Hz, 3H), 1.26 (d, \( J = 7.2 \) Hz, 6H), 2.85-2.95 (m, 1H), 3.69 (s, 2H), 4.10 (q, \( J = 7.2 \) Hz, 2H), 7.02 (s, 1H), 7.22 (d, \( J = 8.4 \) Hz, 2H), 7.25-7.38 (m, 5H), 7.43 (d, \( J = 8.0 \) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta = 14.0, 23.9, 33.7, 36.6, 60.7, 126.1, 126.5, 127.0, 128.3, 128.7, 130.4, 134.4, 137.5, 139.0, 148.3, 171.6 \). HRMS (ESI-TOF). Calcd for C\(_{21}\)H\(_{25}\)O\(_2\), [M+H]\(^+\) \( m/z \) 309.1855, Found 309.1846.
(E)-ethyl 3-(4-(tert-butyl)phenyl)-4-phenylbut-3-enoate 5e
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.17$ (t, $J = 7.2$ Hz, 3H), 1.34 (s, 9H), 3.70 (s, 2H), 4.12 (q, $J = 7.2$ Hz, 2H), 7.04 (s, 1H), 7.27-7.40 (m, 7H), 7.45 (d, $J = 8.8$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 31.3, 34.5, 36.6, 60.7, 125.4, 125.8, 127.1, 128.4, 128.8, 130.5, 134.4, 137.6, 138.7, 150.6, 171.6. HRMS (ESI-TOF). Calcd for C$_{22}$H$_{27}$O$_2$, [M+H]$^+$ $m/z$ 323.2011, Found 323.1996.

(E)-ethyl 4-phenyl-3-(4-vinylphenyl)but-3-enoate 5f
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.18$ (t, $J = 7.2$ Hz, 3H), 3.72 (s, 2H), 4.12 (q, $J = 7.2$ Hz, 2H), 5.27 (d, $J = 10.8$ Hz, 1H), 5.78 (d, $J = 17.6$ Hz, 1H), 6.74 (dd, $J_1 = 10.8$ Hz, $J_2 = 17.6$ Hz, 1H), 7.07 (s, 1H), 7.29-7.32 (m, 1H), 7.37-7.43 (m, 6H), 7.48 (d, $J = 8.4$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 36.5, 60.8, 113.8, 126.3, 127.2, 128.4, 128.8, 131.0, 134.2, 136.4, 136.9, 137.4, 141.0, 171.5. HRMS (ESI-TOF). Calcd for C$_{20}$H$_{21}$O$_2$, [M+H]$^+$ $m/z$ 293.1542, Found 293.1530.
(E)-ethyl 3-(4-chlorophenyl)-4-phenylbut-3-enoate 5g
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.17 (t, $J$ = 7.2 Hz, 3H), 3.68 (s, 2H), 4.11 (q, $J$ = 7.2 Hz, 2H), 7.00 (s, 1H), 7.30-7.39 (m, 7H), 7.43 (d, $J$ = 8.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.6, 60.9, 127.4, 127.6, 128.5, 128.6, 128.7, 131.7, 133.4, 133.6, 137.1, 140.2, 171.3. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{18}$ClO$_2$, [M+H]$^+$ m/z 301.1095, Found 301.0992.

(E)-ethyl 3-(4-bromophenyl)-4-phenylbut-3-enoate 5h
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.18 (t, $J$ = 7.2 Hz, 3H), 3.68 (s, 2H), 4.11 (q, $J$ = 7.2 Hz, 2H), 7.01 (s, 1H), 7.29-7.41 (m, 7H), 7.50 (d, $J$ = 8.8 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.5, 60.9, 121.5, 127.4, 127.9, 128.4, 128.7, 131.5, 131.7, 133.6, 137.0, 140.6, 171.2. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{18}$BrO$_2$, [M+H]$^+$ m/z 345.0490, 347.0470, Found 345.0486, 347.0462.

(E)-ethyl 4-phenyl-3-(m-tolyl)but-3-enoate 5i
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.17 (t, $J$ = 7.2 Hz, 3H), 2.39 (s, 3H), 3.70 (s, 2H), 4.11 (q, $J$ = 7.2 Hz, 2H), 7.02 (s, 1H), 7.12 (d, $J$ = 7.2 Hz, 1H), 7.24-7.32 (m, 4H), 7.38 (d, $J$ = 4.0 Hz, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 21.5, 36.7, 60.7, 123.3, 127.0, 127.1, 128.3, 128.4, 128.7, 131.0, 134.8, 137.5, 138.0, 141.7, 171.6. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{21}$O$_2$, [M+H]$^+$ m/z 281.1542, Found 281.1540.

$$\text{(E)}$$-ethyl 3-(3-methoxyphenyl)-4-phenylbut-3-enolate 5j

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.17 (t, $J$ = 7.2 Hz, 3H), 3.69 (s, 2H), 3.84 (s, 3H), 4.11 (q, $J$ = 7.2 Hz, 2H), 6.84-6.87 (m, 1H), 7.04-7.10 (m, 3H), 7.26-7.31 (m, 2H), 7.38 (d, $J$ = 4.4 Hz, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.8, 55.3, 60.8, 112.1, 113.0, 118.7, 127.2, 128.4, 128.7, 129.4, 131.3, 134.6, 137.3, 143.3, 159.7, 171.5. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{21}$O$_3$, [M+H]$^+$ m/z 297.1491, Found 297.1490.

$$\text{(E)}$$-ethyl 3-(3-formylphenyl)-4-phenylbut-3-enolate 5k

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.16 (t, $J$ = 7.2 Hz, 3H), 3.74 (s, 2H), 4.11 (q, $J$ = 7.2 Hz, 2H), 7.09 (s, 1H), 7.30-7.40 (m, 5H), 7.55 (t, $J$ = 7.6 Hz, 1H), 7.77 (d, $J$ = 8.0 Hz, 1H), 7.82 (d, $J$ = 7.2 Hz, 1H), 8.01 (s, 1H), 10.06 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.6, 60.9, 127.5, 127.6, 128.5, 128.7, 128.8, 129.2, 132.3, 132.7, 133.6, 136.6, 136.9, 142.8, 171.1, 192.2. HRMS (ESI-TOF). Calcd for C$_{19}$H$_{19}$O$_3$, [M+H]$^+$ m/z 295.1334, Found 295.1326.
(E)-ethyl 3-(3-fluorophenyl)-4-phenylbut-3-enoate 5l

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.17 (t, $J$ = 7.2 Hz, 3H), 3.68 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 6.97-7.02 (m, 1H), 7.04 (s, 1H), 7.19-7.22 (m, 1H), 7.28-7.39 (m, 7H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.6, 60.9, 113.3 (d, $J$ = 22.0 Hz), 114.4 (d, $J$ = 21.0 Hz), 121.9 (d, $J$ = 3.0 Hz), 127.5, 128.5, 128.8, 129.9 (d, $J$ = 8.0 Hz), 132.1, 133.7 (d, $J$ = 2.0 Hz), 137.0, 144.1 (d, $J$ = 7.0 Hz), 163.0 (d, $J$ = 244.0 Hz), 171.2. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{18}$F$_2$O, [M+H]$^+$ m/z 285.1291, Found 285.1295.

(E)-ethyl 3-(3-chlorophenyl)-4-phenylbut-3-enoate 5m

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.18 (t, $J$ = 7.2 Hz, 3H), 3.68 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 7.02 (s, 1H), 7.27-7.41 (m, 8H), 7.49 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.6, 60.9, 124.4, 126.5, 127.5, 127.6, 128.5, 128.7, 129.7, 132.3, 133.5, 134.4, 136.9, 143.6, 171.2. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{18}$ClO$_2$, [M+H]$^+$ m/z 301.0995, Found 301.0992.

(E)-ethyl 3-(3-bromophenyl)-4-phenylbut-3-enoate 5n

(E)-ethyl 3-(3-bromophenyl)-4-phenylbut-3-enoate 5n...
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.18$ (t, $J = 7.2$ Hz, 3H), 3.67 (s, 2H), 4.12 (q, $J = 7.2$ Hz, 2H), 7.01 (s, 1H), 7.22-7.32 (m, 2H), 7.36-7.44 (m, 6H), 7.65 (t, $J = 1.6$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 36.6, 60.9, 122.6, 124.9, 127.5, 128.5, 128.7, 129.5, 129.9, 130.5, 132.4, 133.5, 136.9, 144.0, 171.1. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{18}$BrO$_2$, [M+H]$^+$ m/z 345.0490, 347.0470, Found 345.0479, 347.0460.

(E)-ethyl 3-(3,5-dimethylphenyl)-4-phenylbut-3-enoate 5o

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.19$ (t, $J = 7.2$ Hz, 3H), 2.35 (s, 6H), 3.70 (s, 2H), 4.13 (q, $J = 7.2$ Hz, 2H), 6.96 (s, 1H), 7.02 (s, 1H), 7.13 (s, 2H), 7.28-7.31 (s, 1H), 7.39 (d, $J = 4.4$ Hz, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 21.4, 36.7, 60.7, 124.1, 127.0, 128.4, 128.7, 129.3, 130.8, 134.8, 137.6, 137.8, 141.7, 171.6. HRMS (ESI-TOF). Calcd for C$_{20}$H$_{23}$O$_2$, [M+H]$^+$ m/z 295.1698, Found 295.1692.

(E)-ethyl 3-(3,5-difluorophenyl)-4-phenylbut-3-enoate 5p

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.20$ (t, $J = 7.2$ Hz, 3H), 3.65 (s, 2H), 4.14 (q, $J = 7.2$ Hz, 2H), 6.73-6.77 (s, 1H), 7.01-7.06 (s, 3H), 7.30-7.42 (s, 5H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1$, 36.5, 61.0, 102.8 (t, $J = 26.0$ Hz), 109.2 (dd, $J_1 = 8.0$ Hz, $J_2 = 19.0$ Hz), 127.7, 128.5, 128.7, 132.8 (t, $J = 3.0$ Hz), 133.0, 136.6, 145.3 (t, $J = 10.0$ Hz), 163.1 (dd, $J_1 = 13.0$ Hz, $J_2 = 246.0$ Hz), 171.0. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{17}$F$_2$O$_2$, [M+H]$^+$ m/z 303.1197, Found 303.1191.
(E)-ethyl 3-(3,5-dichlorophenyl)-4-phenylbut-3-enoate 5q

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.20 (t, $J$ = 7.2 Hz, 3H), 3.64 (s, 2H), 4.14 (q, $J$ = 7.2 Hz, 2H), 7.02 (s, 1H), 7.29-7.41 (s, 8H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 36.5, 61.0, 124.9, 127.4, 127.7, 128.5, 128.7, 132.5, 133.3, 135.0, 136.5, 144.9, 170.9. HRMS (ESI-TOF). Calcd for C$_{18}$H$_{17}$Cl$_2$O$_2$, [M+H]$^+$ m/z 335.0606, Found 335.0601.

(E)-ethyl 3-(3,4-dimethylphenyl)-4-phenylbut-3-enoate 5r

Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 1.19 (t, $J$ = 7.2 Hz, 3H), 2.28 (s, 3H), 2.30 (s, 3H), 3.69 (s, 2H), 4.12 (q, $J$ = 7.2 Hz, 2H), 7.00 (s, 1H), 7.13 (d, $J$ = 8.0 Hz, 1H), 7.22-7.29 (s, 3H), 7.38 (d, $J$ = 4.8 Hz, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 14.1, 19.4, 19.9, 36.6, 60.7, 123.6, 127.0, 127.5, 128.4, 128.7, 129.7, 130.3, 134.6, 136.1, 136.5, 137.6, 139.3, 171.6. HRMS (ESI-TOF). Calcd for C$_{20}$H$_{23}$O$_2$, [M+H]$^+$ m/z 295.1698, Found 295.1692.
(E)-N,4-diphenyl-3-(p-tolyl)but-3-enamide 5s
White solid. mp: 178-179 ºC. 1H NMR (400 MHz, CDCl$_3$): \( \delta = 2.38 \) (s, 3H), 3.83 (s, 2H), 7.07 (t, \( J = 7.2 \) Hz, 1H), 7.17 (s, 1H), 7.23 (d, \( J = 8.4 \) Hz, 2H), 7.28-7.42 (m, 10H), 7.49 (d, \( J = 8.0 \) Hz, 2H); 13C NMR (100 MHz, CDCl$_3$): \( \delta = 21.1, 40.0, 119.8, 124.4, 126.0, 127.6, 128.7, 128.8, 128.9, 129.7, 131.7, 134.6, 136.8, 137.6, 138.2, 138.4, 168.5 \). HRMS (ESI-TOF). Calcd for C$_{23}$H$_{22}$NO, [M+H]$^+$ m/z 328.1701, Found 328.1697.

![Chemical Structure](image)

(E)-ethyl 2,4-diphenyl-3-(p-tolyl)but-3-enoate 5t
Colorless oil. 1H NMR (400 MHz, CDCl$_3$): \( \delta = 1.06 \) (t, \( J = 7.2 \) Hz, 3H), 2.31 (s, 3H), 4.03-4.09 (m, 2H), 5.35 (s, 1H), 7.01-7.06 (s, 3H), 7.20-7.36 (m, 12H); 13C NMR (100 MHz, CDCl$_3$): \( \delta = 13.9, 21.1, 52.5, 60.9, 126.9, 127.2, 128.0, 128.1, 128.4, 128.5, 128.7, 129.3, 132.0, 137.0, 137.1, 137.2, 138.1, 138.7, 172.2 \). HRMS (ESI-TOF). Calcd for C$_{25}$H$_{25}$O$_2$, [M+H]$^+$ m/z 357.1855, Found 357.1846.

![Chemical Structure](image)

(E)-4-phenyl-3-(p-tolyl)but-3-enoic acid 6
White solid. mp: 154-155 ºC. 1H NMR (400 MHz, CDCl$_3$): \( \delta = 2.39 \) (s, 3H), 3.75 (s, 2H), 7.06 (s, 1H), 7.20 (d, \( J = 8.0 \) Hz, 2H), 7.30-7.43 (m, 7H); 13C NMR (100 MHz, CDCl$_3$): \( \delta = 21.1, 36.1, 126.0, 127.2, 128.5, 128.7, 129.3, 131.0, 133.5, 137.3, 137.6, 138.3, 177.9 \). HRMS (ESI-TOF). Calcd for C$_{17}$H$_{17}$O$_2$, [M+H]$^+$ m/z 253.1229, Found 253.1225.
3-(p-tolyl)naphthalen-1-ol 7a
White solid. mp: 150-151 °C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 2.44$ (s, 3H), 5.54 (s, 1H), 7.07 (s, 1H), 7.29 (d, $J = 7.6$ Hz, 2H), 7.48-7.60 (m, 4H), 7.67 (s, 1H), 7.88 (d, $J = 7.6$ Hz, 1H), 7.21 (d, $J = 8.0$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 21.1, 108.3, 118.4, 121.4, 123.4, 125.2, 126.8, 127.1, 127.9, 129.5, 135.0, 137.2, 137.9, 138.8, 151.6$. HRMS (ESI-TOF). Caled for C$_{17}$H$_{15}$O, [M+H]$^+$ m/z 235.1123, Found 235.1115.

7-ethyl-3-(p-tolyl)naphthalen-1-ol 7b
White solid. mp: 132-134 °C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 1.32$ (t, $J = 7.6$ Hz, 3H), 2.39 (s, 3H), 2.81 (q, $J = 7.6$ Hz, 2H), 5.42 (s, 1H), 7.00 (s, 1H), 7.23 (d, $J = 7.6$ Hz, 2H), 7.35 (d, $J = 8.0$ Hz, 1H), 7.53 (d, $J = 8.0$ Hz, 2H), 7.58 (s, 1H), 7.75 (d, $J = 8.4$ Hz, 1H), 7.93 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 15.6, 21.1, 29.2, 108.4, 118.2, 119.0, 123.5, 127.0, 128.0, 128.0, 129.5, 133.5, 137.0, 137.8, 138.1, 141.3, 151.3$. HRMS (ESI-TOF). Caled for C$_{19}$H$_{19}$O, [M+H]$^+$ m/z 263.1436, Found 263.1428.

(E)-ethyl 5-((3,7-dimethyloct-6-en-1-yl)oxy)-3-(p-tolyl)pent-3-enoate 9
Colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta = 0.91$ (d, $J = 6.4$ Hz, 3H), 1.19 (t, $J = 7.2$ Hz, 3H), 1.35-1.44 (m, 3H), 1.56-1.68 (m, 8H), 1.95-2.03 (m, 2H), 2.33 (s, 3H), 3.49-3.53 (m, 4H), 4.10 (q, $J = 7.2$ Hz, 2H), 4.20 (d, $J = 6.4$ Hz, 2H), 5.10 (t, $J = 6.4$ Hz, 1H), 6.09 (t, $J = 6.4$ Hz, 1H), 7.13 (d,
$J = 8.0 \text{ Hz, } 2\text{H}$), 7.31 (d, $J = 8.0 \text{ Hz, } 2\text{H}$); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 14.1, 17.6, 19.5, 21.0, 25.5, 25.7, 29.6, 36.4, 36.7, 37.2, 60.8, 67.8, 69.0, 124.8, 125.9, 128.0, 129.0, 131.1, 134.9, 137.1, 138.6, 171.0. HRMS (ESI-TOF). Calcd for C$_{24}$H$_{37}$O$_3$, [M+H]$^+$ m/z 373.2743, Found 373.2739.
5. Structure Analysis X-Ray Crystallography of 6

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6. References


7. $^1$H and $^{13}$C NMR Spectra of Compounds 4-7 and 9

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Product 4b
Product 4c
Product 4d
Product 4e
Product 4f
Product 4g
Product 4i
Product 4j
Product 4k
Product 4m
Product 4o
Product 4p
Product 4q
Product 4r
Product 4s
Product 5a
Product 5b
Product 5c
Product 5d
Product 5e
Product 5h
Product 5i
Product 5j
Product 5l
Product 5m
Product 5n
Product 5o
Product 5p
Product 5q
Product 5r
Product 5s
Product 5t
Product 6
Product 7a
Product 7b
Product 9