Supporting Information

Iron-Catalyzed Stereospecific Arylation of Enol Tosylates with Grignard reagents

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1. General experimental details

$^1$HNMR (400, 500 or 600 MHz) and $^{13}$CNMR (100, 125 or 150 MHz) spectra were recorded on Bruker 400 Hz or 500 Hz and JNM-ECE 400R or 600R spectrometers unless otherwise noted. The chemical shifts (δ) were quoted in parts per million from tetramethylsilane for $^1$H and CDCl$_3$ for $^{13}$C spectroscopy. High resolution mass spectra (HRMS) were obtained with a Bruker microTOF (ESI). Infrared data were acquired using an AVATAR 360 FT-IR spectrophotometer. Melting points were recorded on a TECH X-4 microscopic instrument and uncorrected.

2. General remarks for the preparation of starting materials

All reagents and solvents used for aryl magnesium reagents or lithium reagents and reactions were freshly dehydrated before use. The corresponding glassware was oven dried (120 °C) and cooled under a stream of argon gas. Common aryl Grignard reagents such as phenyl magnesium or 4-methoxyphenyl magnesium bromides were prepared according to standard procedure. Functionalized aryl Grignard reagents such as 2-cyanophenyl magnesium chloride or 4-(ethoxycarbonyl)phenyl magnesium chloride were prepared via iodine-magnesium exchange using i-PrMgCl-LiCl according to Knochel’s method.$^{[1]}$ All the Grignard reagents were titrated before use.$^{[2]}$

All enoltosylates were prepared according to the reported methods$^{[3],[4]}$ and their characterization data were consistent with those reported in the literature.
3. Optimization of reaction conditions

\[
\text{OTs} \quad \text{COOEt} + \quad \text{MgBr} \quad \text{FeCl}_3 (x \text{ eq}) / \text{TMEDA} / L (x \text{ eq}) \quad \text{PhOH} (y \text{ eq}) ; \text{Ti(OEt)}_4 (y \text{ eq}) \quad \text{T} ^\circ \text{C} ; \text{THF} \\
\xrightarrow{1 \text{a} \ n \text{ eq}} \quad \text{OMe} \quad \text{COOEt} \\
\text{Z or E-3aa}
\]

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<th>L</th>
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<td>SIPr-HCl</td>
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\(^a\)The reaction was carried out on 2 mmol scale and the amount of TMEDA was twice that of FeCl\(_3\) unless indicated otherwise. \(^b\)The yields referred to total yields of two isolated products and “>98:2” meant that only a single isomer was observed by TLC check and \(^1\)HNMR of reaction mixtures and isolated products. \(^c\)Total amount of Grignard reagents including those to neutralize phenol as well as NHC-HCl and reduce FeCl\(_3\). \(^d\)SIMes = 1,3-bis(2,4,6-trimethyl-phenyl)imidazolin-2-ylidene; SIPr = 1,3-bis(2,6-di-isopropylphenyl)imidazolin-2-ylidene. \(^e\)The reaction was conducted without either Ti(OEt)\(_4\) or PhOH.
4. General Procedure for method A

4.1 Typical procedure of Method A using common Grignard reagents (Z-3ab)

Under Ar atmosphere, Ti(OEt)₄ (228 mg, 1.0 mmol) and PhOH (94 mg, 1.0 mmol) were dissolved in 3 mL THF and stirred at room temperature for 20 to 30 min. A solution of C₆H₅MgBr (12.0 mL; 12.0 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixture during 10–15 min, and then stirred at room temperature for 30 to 40 min.

To another three-necked round-bottom flask were added ethyl (Z)-3-(tosyloxy)-but-2-enoate (1.42g, 5mmol) and 5 mL THF. Under stirring, FeCl₃ (122 mg; 0.75 mmol), TMEDA (174 mg, 1.5 mmol) and SIPr·HCl (308 mg, 0.75 mmol) were added to the resulting solution and stirred for 10 min at room temperature. The mixture was cooled to 0°C and the above-prepared mixed titanate was added dropwise. The stirring was continued at 0 °C until the reaction finished (monitored by TLC, about 6–8 hr). The reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with CH₂Cl₂. The organic layer was washed with 50 mL saturated NaCl aqueous solution and dried over Na₂SO₄, then concentrated to yield the crude compound, which was purified by column chromatography to afford the desired product Z-3ab (779 mg, 82% yield).

4.2 Typical procedure of Method A using functionalized Grignard reagents (Z-3ag)

Under Ar atmosphere, the solution of 2-IC₆H₄CN (2.75 g, 12 mmol) in 5 mL THF was cooled to -40 °C under stirring. To this solution was added i-PrMgCl-LiCl (12.0 mL; 12 mmol, 1.0 M in THF) dropwise. The stirring was continued at that temperature until the exchange reaction was completed (monitored by TLC).

Under Ar atmosphere, THF (3 mL); Ti(OEt)₄ (228 mg, 1.0 mmol) and PhOH (94 mg, 1.0 mmol) were added to another round-bottom flask, and the mixture was stirred and cooled to -45 ~ -50°C. To this solution, the above-prepared Knochel-type functionalized Grignard reagent was added dropwisethrough a syringe during 10-15 min with the temperature being kept below -40°C. After the addition, the mixture was
allowed to come to room temperature in 2 hr, and stirred at that temperature for 30 min. This mixture could be used directly for the arylation.

In the reactions where the concomitant i-PrI was removed, the solvent as well as i-PrI were taken off the mixture under vacuum (about 10 mmHg) during which the temperature was below 20 °C until the mixture became a paste. THF (10 mL) was added to the paste and the stirring was continued until a solution was formed.

To another three-necked round-bottom flask were added ethyl (Z)-3-(tosyloxy)-but-2-enoate (1.42 g, 5 mmol) and 5 mL THF. Under stirring, FeCl₃ (122 mg, 0.75 mmol), TMEDA (174 mg, 1.5 mmol) and SIPr•HCl (308 mg, 0.75 mmol) were added to the resulting solution and stirred for 10 min at room temperature. The mixture was cooled to 0 °C and the above-prepared mixed titanate was added. The stirring was continued at 0 °C until the reaction finished (monitored by TLC, about 6–8 hr). The reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with CH₂Cl₂. The organic layer was washed with 50 mL saturated NaCl aqueous solution and dried over Na₂SO₄, then concentrated to yield the crude compound, which was purified by column chromatography to afford the desired product Z-3ag (714 mg, 71% yield).

5. Typical procedure of Method B (E-3ab)

Under Ar atmosphere, Ti(OEt)₄ (228 mg, 1.0 mmol) and PhOH (94 mg, 1.0 mmol) were dissolved in 3 mL THF and stirred at room temperature for 20 to 30 min. A solution of C₆H₅MgBr (12 mL; 12.0 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixture during 10–15 min, and then stirred at room temperature for 30 to 40 min.

To another three-necked round-bottom flask were added ethyl (E)-3-(tosyloxy)-but-2-enoate (1.42 g, 5 mmol) and 5 mL THF. Under stirring, FeCl₃ (122 mg, 0.75 mmol), TMEDA (174 mg, 1.5 mmol) were added to the resulting solution and stirred for 10 min at room temperature. The above-prepared mixed titanate was added dropwise. The mixture was heated to 65 °C under stirring. The stirring was continued at 65 °C until the reaction finished (monitored by TLC, about 6 hr). The reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with CH₂Cl₂. The organic layer was washed with 50 mL saturated NaCl aqueous solution and dried over Na₂SO₄, then concentrated to yield the crude compound, which was
purified by column chromatography to afford the desired product **E-3ab** (817 mg, 86% yield).

6. Preparations of 9

![Chemical Structure]

According to typical procedure 3.1, the crude products of arylation reaction were obtained and dissolved in 20 mL CH₂Cl₂. The mixture was stirred and cooled to -78 °C, and BBr₃ (2.5 g, 10 mmol) was added dropwise. After addition, the mixture was allowed to come to room temperature and stirred for 24 h. The reaction was quenched with methanol (20 mL). The product was taken up with CH₂Cl₂ (4 × 50 mL) and washed with saturated NaHCO₃ aqueous solution (50 mL). After dried over Na₂SO₄ and concentrated by rotary evaporator, crude product was obtained, which was purified by column chromatography to afford the desired product **9** (1068 mg, 70% yield).

7. Confirmation of configurational retention in arylation by NMR studies

In this paper, almost all products were synthesized in E/Z isomer pairs. Meanwhile, we also successfully distinguished two isomers in every E/Z pair on TLC (see the *R*₇ values in Characterization data), which allowed us to determine that only one isomer was generated during all our arylations. On this basis, we further confirmed that the arylations using method A proceeded with configuration retention by comparing the **¹H NOESY spectra** of the products in Z/E pairs. Below are **¹H NOESY spectra** for five Z/E pairs of arylation products.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the Z-3ae in CDCl$_3$ at 300K irradiated at δ 5.86 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW =20 ppm; O1P = 4, mixing time D8 = 300 ms) of the E-3ae in CDCl$_3$ at 300K irradiated at $\delta$ 6.26 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the Z-3cj in CDCl$_3$ at 300K irradiated at $\delta$ 5.86 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the E-3cj in CDCl$_3$ at 300K irradiated at $\delta$ 5.69 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the Z-4al in CDCl$_3$ at 300K irradiated at $\delta$ 2.01 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the E-4al in CDCl$_3$ at 300K irradiated at $\delta$ 2.22 ppm.
1D selective $^1$H NOESY spectra (B1 = 400.0 MHz; selnoープ; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **Z-4am** in CDCl$_3$ at 300K irradiated at δ 2.36 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the **E-4am** in CDCl$_3$ at 300K irradiated at $\delta$ 2.16 ppm.
1D selective $^1$H NOESY spectra (BFl = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the Z-6 in CDCl$_3$ at 300K irradiated at $\delta$ 6.44 ppm.
1D selective $^1$H NOESY spectra (BF1 = 400.0 MHz; selnogp; NS = 128; DS = 2; TD = 65536; D1 = 2; SW = 20 ppm; O1P = 4, mixing time D8 = 300 ms) of the E-6 in CDCl$_3$ at 300K irradiated at $\delta$ 6.41 ppm.
8. Characterization data

Ethyl (Z)-3-(2-methoxyphenyl)but-2-enoate\textsuperscript{[5]}

Yellow oil; 88% yield (968 mg); $R_f = 0.22$ (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\textsuperscript{-1}, KBr): 3028, 2951, 1750, 1722, 1333, 1181, 752, 690; \(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz) \(\delta\) (ppm) 7.283-7.27 (m, 1H), 7.011 (dd, \(J = 7.5, 1.8\) Hz, 1H), 6.948-6.907 (m, 1H), 6.889 (d, \(J = 8.3\) Hz, 1H), 5.949-5.945 (m, 1H), 4.001-3.931 (m, 2H), 3.787 (s, 3H), 2.139 (dd, \(J = 1.5, 0.7\) Hz, 3H), 1.040 (td, \(J = 7.1, 0.7\) Hz, 3H); \(^{13}\)C NMR (CDCl\textsubscript{3}, 101 MHz, \(\delta\) (ppm) 165.8, 155.5, 153.3, 130.5, 128.8, 128.1, 120.4, 119.0, 110.8, 59.6, 55.6, 26.2, 14.1.

Ethyl (E)-3-(2-methoxyphenyl)but-2-enoate\textsuperscript{[6]}

Yellow oil; 88% yield (968 mg); $R_f = 0.35$ (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\textsuperscript{-1}, KBr): 3036, 2903, 1756, 1722, 1338, 1177, 970, 762; \(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz) \(\delta\) (ppm) 7.289-7.232 (m, 1H), 7.116 (dd, \(J = 7.5, 1.8\) Hz, 1H), 6.925-6.862 (m, 2H), 5.874-5.869 (m, 1H), 4.206-4.152 (m, 2H), 3.797 (s, 3H), 2.470 (d, \(J = 1.3\) Hz, 3H), 1.280 (td, \(J = 7.1, 0.6\) Hz, 3H); \(^{13}\)C NMR (CDCl\textsubscript{3}, 101 MHz, \(\delta\) (ppm) 166.9, 156.7, 156.4, 133.2, 129.5, 128.9, 120.6, 119.3, 111.1, 59.8, 55.5, 19.9, 14.4.

Ethyl (Z)-3-phenylbut-2-enoate\textsuperscript{[3]}

Yellow oil; 82% yield (779 mg); $R_f = 0.25$ (petroleum ether/ethyl acetate = 50:1, v/v).
IR (cm⁻¹, KBr): 3029, 2891, 1747, 1728, 1250, 1190, 750, 681; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.371-7.289 (m, 3H), 7.220-7.198 (m, 2H), 5.914 (s, 1H), 4.002 (q, J = 7.2 Hz, 2H), 2.184 (s, 3H), 1.081 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 166.0, 155.4, 141.0, 128.0, 127.8, 126.9, 117.0, 59.8, 27.2, 14.0.

Methyl (Z)-3-(p-tolyl)but-2-enoate[^3]

Yellow oil; 74% yield (703 mg); R_f = 0.23 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm⁻¹, KBr): 3030, 2890, 1786, 1728, 1201, 1167, 840, 684; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.169 (d, J = 8.2 Hz, 2H), 7.120 (d, J = 8.0 Hz, 2H), 5.901 (s, 1H), 3.574 (s, 3H), 2.361 (s, 3H), 2.177 (s, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 166.5, 156.2, 137.8, 128.7, 126.9, 116.9, 51.1, 27.3, 21.4.

Methyl (Z)-3-(4-fluorophenyl)but-2-enoate[^7]

Yellow oil; 76% yield (737 mg); R_f = 0.20 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm⁻¹, KBr): 3038, 2904, 1760, 1721, 1248, 1150, 827, 676; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.221-7.171 (m, 2H), 7.068-7.010 (m, 2H), 5.926 (d, J = 1.5 Hz, 1H), 3.572 (s, 3H), 2.170 (d, J = 1.5 Hz, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 166.2, 162.5 (d, J = 245.0 Hz, 1C), 155.0, 136.5, 128.8 (d, J = 8.2 Hz, 1C), 117.6, 115.0 (d, J = 22.0 Hz, 1C), 51.1, 27.3.

Methyl (Z)-3-(thiophen-2-yl)but-2-enoate[^8]

Yellow oil; 71% yield (646 mg); R_f = 0.30 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3029, 2899, 1758, 1722, 1231, 1146, 819, 661; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.479 (dd, J = 3.7, 1.2 Hz, 1H), 7.373 (dd, J = 5.1, 1.2 Hz, 1H),...
7.021 (dd, J = 5.1, 3.7 Hz, 1H), 5.842 (d, J = 1.3 Hz, 1H), 3.674 (s, 3H), 2.276 (d, J = 1.4 Hz, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 166.5, 144.8, 140.2, 129.4, 127.8, 126.8, 116.0, 51.3, 27.6.

![Z-3af](image)

**Ethyl (Z)-3-(2-(trifluoromethyl)phenyl)but-2-enolate**

Yellow oil; 75% yield (968 mg); Rᵣ = 0.29 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm⁻¹, KBr): 3011, 2891, 1761, 1710, 1130, 764, 701; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.536 (d, J = 5.3 Hz, 1H), 7.427 (d, J = 5.0 Hz, 1H), 7.330 (t, J = 5.1 Hz, 1H), 7.220 (d, J = 5.1 Hz, 1H), 7.084 (d, J = 0.8 Hz, 1H), 4.012 (q, J = 4.7 Hz, 2H), 2.354 (s, 3H), 1.297 (d, J = 4.7 Hz, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 165.8, 156.3, 155.1, 140.5 (d, J = 5.3 Hz, 1C), 131.3, 128.9, 128.6, 125.9 (q, J = 13.7 Hz, 1C), 120.1 (d, J = 4.1 Hz, 1C), 118.3, 111.4, 59.9, 27.4, 14.0. HRMS calcd for C₁₃H₁₃F₃NaO₂⁺ [M+Na⁺] 281.0765, Found 281.0758.

![Z-3ag](image)

**Methyl (Z)-3-(2-cyanophenyl)but-2-enolate**

Yellow oil; 71% yield (714 mg); Rᵣ = 0.30 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3020, 2890, 1751, 1720, 1198, 1120, 771, 693; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.720 (ddd, J = 8.1, 1.8, 0.8 Hz, 1H), 7.609 (dt, J = 7.9, 1.5 Hz, 1H), 7.365 (q, J = 6.3 Hz, 1H), 7.277 (td, J = 7.8, 1.8 Hz, 1H), 5.902 (s, 1H), 3.572 (s, 3H), 2.192 (s, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 166.3, 157.3, 136.8, 128.2, 126.5, 126.3, 126.1, 124.3, 118.3, 111.4, 51.2, 27.1. HRMS calcd for C₁₂H₁₂NO₂⁺ [M+H]⁺ 202.0866, Found 202.0866.

![Z-3bb](image)

**Methyl (Z)-3-phenylhex-2-enolate**

Yellow oil; 74% yield (755 mg); Rᵣ = 0.24 (petroleum ether/ethyl acetate = 50:1, v/v).
IR (cm$^{-1}$, KBr): 3033, 2948, 1749, 1710, 1233, 1109, 741, 689; $^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.369-7.307 (m, 3H), 7.168-7.144 (m, 2H), 5.885 (s, 1H), 3.544 (s, 3H), 2.445-2.405 (m, 2H), 1.415 (dd, $J = 14.9, 7.4$ Hz, 2H), 0.912 (t, $J = 7.4$ Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 166.5, 160.3, 140.2, 127.9, 127.7, 127.1, 116.8, 51.1, 42.6, 20.6, 13.6. HRMS calcd for C$_{13}$H$_{16}$NaO$_2^+$ [M+Na]$^+$ 227.1048, Found 227.1045.

Methyl (Z)-3-(2-methoxyphenyl)hex-2-enoate
Yellow oil; 72% yield (842 mg); $R_f = 0.20$ (petroleum ether/ethyl acetate = 20:1, v/v).
IR (cm$^{-1}$, KBr): 3021, 2910, 1762, 1728, 1204, 1131, 752, 679; $^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.268-7.242 (m, 1H), 6.952 (ddd, $J = 7.9, 7.3, 1.4$ Hz, 2H), 6.893 (d, $J = 8.3$ Hz, 1H), 5.914 (s, 3H), 3.721 (s, 3H), 3.500 (s, 3H), 2.420-2.385 (m, 2H), 1.405 (dd, $J = 15.0, 7.4$ Hz, 2H), 0.904 (t, $J = 7.4$ Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 166.3, 157.9, 155.7, 129.5, 128.5, 120.3, 117.7, 110.8, 55.6, 50.6, 41.5, 20.6, 13.8. HRMS calcd for C$_{14}$H$_{18}$NaO$_3^+$ [M+Na]$^+$ 257.1154, Found 257.1150.

Methyl (Z)-3-(pyridin-3-yl)hex-2-enoate
Yellow oil; 75% yield (768 mg); $R_f = 0.28$ (petroleum ether/ethyl acetate = 10:1, v/v).
IR (cm$^{-1}$, KBr): 3033, 2956, 1762, 1722, 1433, 1289, 1128, 680; $^{1}$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 8.684 (d, $J = 0.9$ Hz, 1H), 8.605-8.588 (m, 1H), 7.740-7.717 (m, 1H), 7.323 (dd, $J = 5.7, 1.5$ Hz, 1H), 6.057 (s, 1H), 3.758 (s, 3H), 3.088 (t, $J = 7.7$ Hz, 2H), 1.405 (dd, $J = 15.2, 7.6$ Hz, 2H), 0.936 (t, $J = 8.6$ Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 166.3, 157.4, 149.9, 147.8, 136.9, 134.0, 123.3, 118.5, 51.2, 32.6, 22.1, 13.9. HRMS calcd for C$_{12}$H$_{16}$NO$_2^+$ [M+H]$^+$ 206.1181, Found 206.1178.
Methyl (Z)-4-methyl-3-(3-(trifluoromethyl)phenyl)pent-2-enoate

Yellow oil; 80% yield (1088 mg); \( R_f = 0.3 \) (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm\(^{-1}\), KBr): 3029, 2899, 1768, 1720, 1281, 1170, 776, 683; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \( \delta \) (ppm) 7.570 (ddd, \( J = 3.5, 1.5, 0.9 \) Hz, 1H), 7.489-7.449 (m, 1H), 7.353 (s, 1H), 7.300-7.281 (m, 1H), 5.939 (d, \( J = 1.2 \) Hz, 1H), 3.516 (s, 3H), 2.690-2.619 (m, 1H), 1.088 (d, \( J = 6.8 \) Hz, 6H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz,) \( \delta \) (ppm) 166.4, 164.1, 140.9, 130.9 (d, \( J = 1.5 \) Hz, 1C), 130.3 (d, \( J = 32.0 \) Hz, 1C), 128.2, 125.6, 124.2 (q, \( J = 7.9 \) Hz, 1C), 122.9, 116.3, 51.2, 37.3, 21.1.HRMS calcd for C\(_{14}\)H\(_{16}\)F\(_3\)O\(_2\)\(^+\) [M+H]\(^+\) 273.1102, Found 273.1100.

Methyl (Z)-3-(4-(dimethylcarbamoyl)phenyl)-4-methylpent-2-enoate

Yellow solid; 70% yield (962.5 mg); \( R_f = 0.29 \) (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm\(^{-1}\), KBr): 3018, 2860, 1766, 1723, 1630, 1319, 1277, 1163, 821, 692; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \( \delta \) (ppm) 7.373-7.350 (m, 2H), 7.087 (dd, \( J = 7.9, 0.5 \) Hz, 2H), 5.838 (s, 1H), 3.474-3.470 (m, 3H), 3.019 (d, \( J = 35.2 \) Hz, 6H), 2.590 (hept, \( J = 6.8 \) Hz, 1H), 1.016 (dd, \( J = 6.8, 0.5 \) Hz, 6H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz,) \( \delta \) (ppm) 171.5, 166.6, 165.0, 141.6, 135.2, 127.3, 126.7, 115.6, 51.1, 39.8, 37.4, 35.4, 21.1.HRMS calcd for C\(_{16}\)H\(_{21}\)NNaO\(_3\)\(^+\) [M+Na]\(^+\) 298.1419, Found 298.1416.

Ethyl (Z)-3-(2,4-dimethylphenyl)-3-phenylacrylate
Yellow oil; 85% yield (1190 mg); R$_f$ = 0.31 (petroleum ether/ethyl acetate = 50:1, v/v).
IR (cm$^{-1}$, KBr): 3022, 2871, 1760, 1719, 1238, 1154, 870, 752, 701; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.545-7.299 (m, 5H), 7.061-7.036 (m, 2H), 6.958 (d, $J$ = 7.6 Hz, 1H), 6.506 (s, 1H), 4.043 (q, $J$ = 7.1 Hz, 2H), 2.367 (s, 3H), 2.046 (s, 3H), 1.118 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 166.0, 156.4, 139.8, 137.5, 135.8, 135.3, 130.8, 129.5, 128.7, 128.5, 127.6, 126.3, 117.7, 60.1, 21.4, 19.7, 14.2.HRMS calcd for C$_{19}$H$_{20}$NaO$_2$ $^{+}$ [M+Na]$^{+}$ 303.1361, Found 303.1354.

Ethyl (Z)-3-phenyl-3-(pyridin-3-yl)acrylate
Yellow oil; 70% yield (885 mg); R$_f$ = 0.22 (petroleum ether/ethyl acetate = 10:1, v/v).
IR (cm$^{-1}$, KBr): 3029, 2944, 1768, 1721, 1233, 1180, 742, 691; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 8.620 (dd, $J$ = 4.8, 1.5 Hz, 1H), 8.478 (d, $J$ = 1.7 Hz, 1H), 7.549-7.520 (m, 1H), 7.373-7.263 (m, 6H), 6.458 (s, 1H), 4.063 (q, $J$ = 7.1 Hz, 2H), 1.127 (t, $J$ = 7.1 Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 165.7, 153.0, 149.8, 149.2, 140.0, 136.7, 134.9, 129.9, 128.7, 128.2, 122.8, 119.0, 60.4, 14.1.HRMS calcd for C$_{16}$H$_{16}$NO$_2$ $^{+}$ [M+H]$^{+}$ 254.1181, Found 254.1180.

Methyl (Z)-3-(4-bromophenyl)-3-(thiophen-2-yl)acrylate
Yellow oil; 75% yield (1211 mg); R$_f$ = 0.36 (petroleum ether/ethyl acetate = 20:1, v/v).
IR (cm$^{-1}$, KBr): 3026, 2913, 1763, 1728, 1288, 1162, 868, 692; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm)7.668-7.637 (m, 2H), 7.456 (dd, $J$ = 5.1, 1.2 Hz, 1H), 7.355 (dd, $J$ = 5.1, 1.2 Hz, 1H), 7.284 (dd, $J$ = 5.2, 3.6 Hz, 1H), 7.096-7.060 (m, 2H), 6.224 (s, 1H), 3.698 (s, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 166.3, 148.4, 143.5, 140.6, 135.7, 129.2, 128.3, 126.9, 125.7, 123.8, 117.6, 51.6. HRMS calcd for C$_{14}$H$_{11}$BrNaO$_2$S$^{+}$ [M+Na]$^{+}$ 344.9561, Found 344.9552.
Ethyl (E)-3-phenylbut-2-enoate$^3$

Yellow oil; 83% yield by method A (788 mg); 86% yield by method B (817 mg); R$_f$ = 0.30 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm$^{-1}$, KBr): 3026, 2889, 1751, 1722, 1248, 1177, 966, 751; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.491-7.467 (m, 2H), 7.388-7.351 (m, 3H), 6.141 (d, $J = 1.3$ Hz, 1H), 4.222 (q, $J = 7.1$ Hz, 2H), 2.584 (d, $J = 1.3$ Hz, 3H), 1.322 (t, $J = 7.1$ Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz.) $\delta$ (ppm) 166.9, 155.6, 142.4, 129.0, 128.6, 126.4, 117.3, 59.9, 18.0, 14.4.

Methyl (E)-3-(p-tolyl)but-2-enoate$^3$

Yellow oil; 74% yield by method A (703 mg); 82% yield by method B (779 mg); R$_f$ = 0.31 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm$^{-1}$, KBr): 3031, 2892, 1783, 1723, 1244, 1166, 962, 842; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.387 (d, $J = 8.2$ Hz, 2H), 7.185 (d, $J = 8.5$ Hz, 2H), 6.167 (s, 1H), 3.752 (s, 3H), 2.574 (d, $J = 1.3$ Hz, 3H), 2.371 (s, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz.) $\delta$ (ppm) 167.5, 155.9, 139.3, 129.3, 126.3, 115.9, 51.1, 21.3, 17.9.

Methyl (E)-3-(4-fluorophenyl)but-2-enoate$^7$

Yellow oil; 75% yield by method A (727 mg); 80% yield by method B (776 mg); R$_f$ = 0.28 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm$^{-1}$, KBr): 3032, 2910, 1760, 1722, 1245, 1155, 976, 827; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.454 (dd, $J = 9.0$, 5.3 Hz, 2H), 7.074-7.031 (m, 2H), 6.092 (d, $J = 1.3$ Hz, 1H), 3.748 (s, 3H), 2.556 (d, $J = 1.3$ Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz.) $\delta$ (ppm) 167.2, 163.3 (d, $J = 248.1$ Hz, 1C), 154.7, 138.22 (d, $J = 3.4$ Hz, 1C), 128.19 (d, $J = 8.3$ Hz, 1C), 116.69 (d, $J = 1.5$ Hz, 1C), 115.5 (d, $J = 21.2$ Hz, 1C), 51.2, 18.1.
Methyl (E)-3-(thiophen-2-yl)but-2-enoate

Yellow oil; 70% yield by method A (637 mg); 76% yield by method B (692 mg); Rf = 0.33 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3022, 2896, 1752, 1711, 1233, 1144, 960, 817; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.288 (d, J = 4.2 Hz, 2H), 7.012 (dd, J = 4.7, 4.1 Hz, 1H), 6.242 (d, J = 1.2 Hz, 1H), 3.716 (s, 3H), 2.587 (d, J = 1.2 Hz, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 167.2, 148.1, 145.6, 128.0, 127.2, 126.9, 113.9, 51.2, 17.4.

Ethyl (E)-3-(2-(trifluoromethyl)phenyl)but-2-enoate

Yellow oil; 77% yield by method A (993 mg); 82% yield by method B (1058 mg); Rf = 0.34 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm⁻¹, KBr): 3013, 2893, 1762, 1712, 1221, 1130, 867, 764; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.677-7.65 (m, 1H), 7.533 (ddd, J = 7.6, 3.8, 0.6 Hz, 1H), 7.439-7.41 (m, 1H), 7.223-7.203 (m, 1H), 5.768 (d, J = 1.4 Hz, 1H), 4.217 (q, J = 7.1 Hz, 2H), 2.468-2.465 (m, 3H), 1.309 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 168.2, 166.2, 162.4, 155.1, 142.8 (d, J = 2.3 Hz, 1C), 137.0, 132.5 (d, J = 148.0 Hz, 1C), 128.9 (d, J = 86.5 Hz, 1C), 126.3 (d, J = 5.0 Hz, 1C), 120.44 (q, J = 2.1 Hz, 1C), 60.1, 21.3, 14.3. HRMS calcd for C₁₃H₁₄F₃O₂⁺ [M+H]⁺ 259.0946, Found 259.0938.

Methyl (E)-3-(2-cyanophenyl)but-2-enoate

Yellow oil; 68% yield by method A (683 mg); 70% yield by method B (704 mg); Rf = 0.35 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3022, 2889, 1765, 1720, 1179, 1123, 871, 771; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.644 (ddd, J = 8.1, 1.8, 0.8 Hz, 1H), 7.539 (dt, J = 7.9, 1.5 Hz, 1H), 7.355 (t, J = 4.0 Hz, 1H), 7.288 (q, J = 6.1 Hz, 1H), 6.124 (s, 1H), 3.763 (s, 3H), 2.573 (s, 3H); ¹³C NMR (CDCl₃, 101 MHz) δ (ppm) 167.5, 155.8, 139.3, 129.2, 128.3, 127.2, 126.3, 126.2, 116.4, 111.3, 51.2, 17.3. HRMS calcd for C₁₂H₁₂NO₂⁺ [M+H]⁺ 202.0868, Found 202.0867.
Methyl (E)-3-phenylhex-2-enoate\(^9\)

Yellow oil; 74% yield by method A (755 mg); 80% yield by method B (816 mg); \(R_f = 0.30\) (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm\(^{-1}\), KBr): 3032, 2945, 1721, 1707, 1109, 877, 741; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.443 – 7.418 (m, 2H), 7.378 – 7.350 (m, 2H), 6.051 (s, 1H), 3.747 (s, 3H), 3.101 (dd, \(J = 8.6, 6.9\) Hz, 2H), 1.518 – 1.424 (m, 2H), 0.946 (t, \(J = 7.4\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 167.0, 161.2, 141.5, 128.9, 128.6, 126.8, 117.1, 51.1, 32.9, 22.3, 14.1.

Methyl (E)-3-(2-methoxyphenyl)hex-2-enoate

Yellow oil; 73% yield by method A (854 mg); 78% yield by method B (913 mg); \(R_f = 0.31\) (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3021, 2913, 1766, 1728, 1204, 1131, 869, 752; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.304 – 7.260 (m, 1H), 7.086 (dd, \(J = 7.4, 1.8\) Hz, 1H), 6.927 (dt, \(J = 7.5, 3.7\) Hz, 1H), 6.881 (d, \(J = 8.3\) Hz, 1H), 5.819 (s, 1H), 3.798 (s, 3H), 3.719 (s, 3H), 3.039 – 3.000 (m, 2H), 1.382 – 1.325 (m, 2H), 0.880 (t, \(J = 7.4\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 167.0, 161.2, 156.4, 131.8, 129.5, 129.4, 120.5, 118.9, 110.9, 55.5, 51.1, 34.1, 21.8, 14.2. HRMS calcd for C\(_{14}\)H\(_{18}\)NaO\(_3\)\(^+\) [M+Na]\(^+\) 257.1154, Found 257.1150.

Methyl (E)-3-(pyridin-3-yl)hex-2-enoate

Yellow oil; 77% yield by method A (789 mg); 81% yield by method B (830 mg); \(R_f = 0.27\) (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm\(^{-1}\), KBr): 3033, 2953, 1732, 1718, 1430, 1286, 1128, 879; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 8.472 (dt, \(J = 4.8, 1.6\) Hz, 1H), 8.335 (s, 1H), 7.442 – 7.418 (m, 1H), 7.217 – 7.192 (m, 1H), 5.904 (s, 1H), 3.478 (s, 3H), 2.358 (td, \(J = 7.8, 1.3\) Hz, 2H), 1.373-
1.314 (m, 2H), 0.862-0.822 (m, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz) $\delta$ (ppm) 165.9, 156.4, 148.7, 147.9, 135.7, 134.7, 122.7, 118.4, 51.1, 42.2, 20.4, 13.4. HRMS calcd for C$_{12}$H$_{16}$NO$_2^+$ [M+H]$^+$ 206.1181, Found 206.1178.

Methyl (E)-4-methyl-3-(3-(trifluoromethyl)phenyl)pent-2-enoate

Yellow oil; 81% yield by method A (1101 mg); 86% yield by method B (1034 mg); R$_f$ = 0.36 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm$^{-1}$, KBr): 3032, 2899, 1761, 1720, 1281, 1170, 776; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.589 (d, $J = 7.8$ Hz, 1H), 7.481-7.440 (m, 2H), 7.381 (d, $J = 7.7$ Hz, 1H), 5.709 (s, 1H), 4.137 (dq, $J = 13.7$, 6.8 Hz, 1H), 3.751 (s, 3H), 1.081 (d, $J = 7.0$ Hz, 6H); $^{13}$C NMR (CDCl$_3$, 101 MHz) $\delta$ (ppm) 166.3, 165.6, 141.4, 131.1, 124.5 (d, $J = 2.9$ Hz, 1C), 119.2, 100.0, 51.3, 29.6, 21.3. HRMS calcd for C$_{14}$H$_{16}$F$_3$O$_2^+$ [M+H]$^+$ 273.1102, Found 273.1100.

Methyl (E)-3-(4-(dimethylcarbamoyl)phenyl)-4-methylpent-2-enoate

Yellow oil; 71% yield by method A (976 mg); 71% yield by method B (976 mg); R$_f$ = 0.32 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3019, 2877, 1769, 1722, 1639, 1316, 1273, 1163, 866, 821; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.392-7.370 (m, 2H), 7.233-7.204 (m, 2H), 5.686 (d, $J = 0.5$ Hz, 1H), 4.162-4.058 (m, 1H), 3.731 (s, 3H), 3.053 (s, 6H), 1.072 (d, $J = 7.0$ Hz, 6H); $^{13}$C NMR (CDCl$_3$, 101 MHz) $\delta$ (ppm) 171.3, 166.6, 166.5, 142.0, 135.7, 127.8, 126.7, 118.5, 51.2, 39.7, 35.5, 29.6, 21.4. HRMS calcd for C$_{16}$H$_{22}$NO$_3^+$ [M+H]$^+$ 276.1600, Found 276.1591.
Methyl (E)-3-(4-bromophenyl)-3-(thiophen-2-yl)acrylate

Yellow oil; 76% yield by method A (1227 mg); 78% yield by method B (1260 mg); R_f =0.39 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3020, 2918, 1760, 1720, 1285, 1162, 877, 868; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.560-7.539 (m, 2H), 7.378 (dd, \(J = 5.1, 1.2\) Hz, 1H), 7.173-7.151 (m, 2H), 6.984 (dd, \(J = 5.1, 3.8\) Hz, 1H), 6.842 (dd, \(J = 3.7, 1.2\) Hz, 1H), 6.409 (s, 1H), 3.612 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 165.9, 149.4, 144.5, 137.0, 131.3, 130.41, 130.36, 128.6, 128.1, 122.7, 114.4, 51.4. HRMS calcd for C\(_{14}\)H\(_{11}\)BrNaO\(_2\)S\(^+\) [M+Na\(^+\)] 344.9561, Found 344.9553.

Ethyl (Z)-3-(4-methoxyphenyl)-2-methylbut-2-enoate

Yellow oil; 67% yield (784 mg); R_f = 0.23 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3018, 2950, 2825, 1766, 1721, 1127, 860; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.074 (d, \(J = 8.8\) Hz, 2H), 6.822 (d, \(J = 8.8\) Hz, 2H), 3.886 (q, \(J = 7.1\) Hz, 2H), 3.787 (s, 3H), 2.063 (d, \(J = 1.1\) Hz, 3H), 2.004 (d, \(J = 1.1\) Hz, 3H), 0.900 (t, \(J = 7.1\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 171.0, 158.8, 142.2, 136.5, 128.2, 125.8, 113.5, 60.2, 55.3, 21.7, 16.6, 13.8.

Ethyl (Z)-2-methyl-3-(naphthalen-1-yl)but-2-enoate

Yellow oil; 66% yield (838 mg); R_f = 0.22 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm\(^{-1}\), KBr): 3010, 2980, 1788, 1723, 1129, 760; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.873-7.849 (m, 1H), 7.783-7.742 (m, 2H), 7.487-7.442 (m, 3H), 7.188 (dd, \(J = 5.1, 1.2\) Hz, 1H), 7.151-7.128 (m, 2H), 6.842 (dd, \(J = 3.7, 1.2\) Hz, 1H), 6.409 (s, 1H), 3.612 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 165.9, 149.4, 144.5, 137.0, 131.3, 130.41, 130.36, 128.6, 128.1, 122.7, 114.4, 51.4. HRMS calcd for C\(_{14}\)H\(_{11}\)BrNaO\(_2\)S\(^+\) [M+Na\(^+\)] 344.9561, Found 344.9553.
7.0, 1.2 Hz, 1H), 4.309 (q, J = 7.1 Hz, 2H), 2.345 (s, 3H), 1.571 (s, 3H), 1.376 (t, J = 7.1 Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz, $\delta$ (ppm) 169.6, 145.0, 141.5, 133.8, 129.8, 128.5, 127.3, 126.7, 126.4, 126.0, 125.7, 125.1, 124.0, 60.5, 23.5, 17.4, 14.5. HRMS calcd for C$_{17}$H$_{18}$NaO$_2$$^+$ [M+Na]$^+$ 277.1204, Found 277.1201.

![Z-4bn](image)

**Ethyl (Z)-3-(2,4-dimethylphenyl)-2-methyl-3-phenylacrylate**

Yellow oil; 77% yield (1132 mg); R$_f$ = 0.31 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm$^{-1}$, KBr): 3021, 2868, 1760, 1719, 1234, 1154, 873, 751; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.210-7.174 (m, 3H), 7.180 (dd, J = 7.7, 1.8 Hz, 2H), 7.029-6.984 (m, 3H), 3.973 (qd, J = 7.1, 1.4 Hz, 2H), 2.305 (s, 3H), 2.099 (s, 3H), 1.819 (s, 3H), 0.925 (t, J = 7.1 Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz, $\delta$ (ppm) 171.3, 145.8, 141.6, 137.5, 137.3, 135.5, 131.2, 129.4, 129.0, 128.8, 128.3, 127.9, 127.2, 126.6, 60.6, 21.2, 19.6, 18.2, 13.7. HRMS calcd for C$_{20}$H$_{22}$NaO$_2$$^+$ [M+Na]$^+$ 317.1517, Found 317.1510.

![Z-4bo](image)

**Ethyl (Z)-3-(3-cyanophenyl)-2-methyl-3-phenylacrylate**

Yellow oil; 65% yield (946 mg); R$_f$ =0.32 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm$^{-1}$, KBr): 3089, 2879, 1762, 1720, 1179, 1122, 781; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.446-7.394 (m, 5H), 7.343 (d, J = 7.6 Hz, 1H), 7.302 (dd, J = 10.3, 4.7 Hz, 2H), 7.238 (d, J = 4.2 Hz, 1H), 3.856 (q, J = 3.9 Hz, 2H), 2.212 (s, 3H), 1.055 (t, J = 3.9 Hz, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz, $\delta$ (ppm) 169.3, 145.8, 144.6, 132.5, 128.4, 127.6, 126.6, 126.4, 126.2, 126.1, 125.4, 122.5, 122.1, 111.6, 60.1, 27.5, 13.7. HRMS calcd for C$_{19}$H$_{18}$NO$_2$$^+$ [M+H]$^+$ 292.1337, Found 292.1339.

![Z-4ce](image)

**Ethyl (Z)-2,3-diphenyl-3-(thiophen-2-yl)acrylate**

S29
Yellow oil; 71% yield (1186 mg); R_f = 0.31 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3042, 3011, 2881, 1769, 1720, 1179, 1123, 772, 741; ^1H NMR (CDCl₃, 400 MHz) δ (ppm) 7.372–7.340 (m, 3H), 7.181–7.094 (m, 9H), 6.992 (dd, J = 5.1, 3.6 Hz, 1H), 4.175 (q, J = 7.1 Hz, 2H), 1.148 (t, J = 7.1 Hz, 3H); ^13C NMR (CDCl₃, 101 MHz) δ (ppm) 170.5, 144.4, 140.2, 136.9, 130.9, 130.0, 129.7, 129.4, 128.9, 128.6, 128.2, 128.1, 127.9, 127.6, 127.4, 126.9, 100.0, 61.4, 13.9. HRMS calcd for C₂₁H₁₈Na₂O₂S⁺ [M+Na⁺]⁺ 357.0925, Found 357.0920.

Ethyl (Z)-3-(3-ethoxy-3-oxo-1,2-diphenylprop-1-en-1-yl)benzoate
Yellow oil; 71% yield (1420 mg); R_f = 0.34 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3043, 3011, 2881, 1769, 1720, 1179, 1123, 772, 741; ^1H NMR (CDCl₃, 400 MHz) δ (ppm) 8.313 (s, 2H), 8.066 (d, J = 7.7 Hz, 2H), 7.969 (d, J = 7.6 Hz, 1H), 7.810 (d, J = 7.6 Hz, 2H), 7.536 (t, J = 7.7 Hz, 3H), 7.439-7.408 (m, 2H), 7.376-7.266 (m, 2H), 4.429 (q, J = 7.1 Hz, 4H), 1.429 (t, J = 7.1 Hz, 6H); ^13C NMR (CDCl₃, 101 MHz) δ (ppm) 168.9, 166.5, 140.6, 133.6, 131.59, 131.56, 130.3, 129.7, 129.3, 129.04, 128.99, 128.89, 128.85, 128.5, 128.3, 128.1, 128.0, 61.2, 60.7, 14.4, 14.1. HRMS calcd for C₂₆H₂₅O₄⁺ [M+H⁺]⁺ 401.1753, Found 401.1751.

Ethyl (E)-3-(4-methoxyphenyl)-2-methylbut-2-enoate[^10]
Yellow oil; 70% yield (819 mg); R_f = 0.28 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3029, 2951, 2825, 1762, 1722, 1134, 862; ^1H NMR (CDCl₃, 400 MHz) δ (ppm) 7.085-7.070 (m, 2H), 6.887-6.873 (m, 2H), 4.247 (q, J = 4.7 Hz, 2H), 3.809 (s, 3H), 2.225 (s, 3H), 1.771 (s, 3H), 1.331 (d, J = 4.8 Hz, 3H); ^13C NMR (CDCl₃, 101 MHz) δ (ppm) 170.3, 158.7, 145, 135.8, 128.7, 124.8, 113.7, 60.4, 55.3, 23.3, 17.5, 14.4.
Ethyl (E)-2-methyl-3-(naphthalen-1-yl)but-2-enate

Yellow oil; 68% yield (864 mg); R_f = 0.27 (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm⁻¹, KBr): 3023, 2989, 1783, 1726, 1139, 758;¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.826 (dd, J = 6.2, 3.3 Hz, 1H), 7.761 (dd, J = 6.1, 3.5 Hz, 1H), 7.733 (d, J = 8.2 Hz, 1H), 7.447 (dd, J = 6.3, 3.3 Hz, 2H), 7.392 (dd, J = 8.0, 7.2 Hz, 1H), 7.135 (d, J = 6.9 Hz, 1H), 3.629-3.538 (m, 2H), 2.193 (s, 3H), 2.166 (s, 3H), 0.375 (t, J = 7.1 Hz, 3H);¹³C NMR (CDCl₃, 101 MHz,) δ (ppm) 169.4, 143.4, 142.8, 133.6, 130.9, 128.3, 127.7, 126.9, 125.9, 125.7, 125.5, 125.4, 123.6, 59.8, 22.8, 15.9, 13.1.HRMS calcd for C₁₇H₁₈NaO₂⁺ [M+Na]⁺ 277.1204, Found 277.1202.

Ethyl (E)-3-(2,4-dimethylphenyl)-2-methyl-3-phenylacrylate

Yellow oil; 76% yield (1117 mg); R_f =0.33 (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm⁻¹, KBr): 3018, 2865, 1766, 1713, 1230, 1152, 748;¹H NMR (CDCl₃, 400 MHz) δ (ppm) 7.313-7.275 (m, 2H), 7.246 (dd, J = 5.1, 3.6 Hz, 1H), 7.198-7.174 (m, 2H), 6.995 (d, J = 7.5 Hz, 1H), 6.928 (d, J = 8.3 Hz, 2H), 3.878 (q, J = 7.1 Hz, 2H), 2.287 (s, 3H), 2.096 (s, 3H), 2.044 (s, 3H), 0.843 (t, J = 7.1 Hz, 3H);¹³C NMR (CDCl₃, 101 MHz,) δ (ppm)170.7, 146.8, 140.1, 139.5, 137.0, 135.8, 130.8, 129.4, 128.8, 128.7, 128.0, 127.5, 126.0, 60.3, 21.2, 19.9, 17.7, 13.5.HRMS calcd for C₂₀H₂₂NaO₂⁺ [M+Na]⁺ 317.1517, Found 317.1509.

Ethyl (E)-3-(3-cyanophenyl)-2-methyl-3-phenylacrylate

Yellow oil; 66% yield (960 mg); R_f = 0.36 (petroleum ether/ethyl acetate = 20:1, v/v).
IR (cm\(^{-1}\), KBr): 3086, 2873, 1764, 1718, 1176, 1122, 779; \(^{1}\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.407-7.356 (m, 5H), 7.305 (dt, \(J = 13.3, 7.7\) Hz, 3H), 7.220 (s, 1H), 4.042 (q, \(J = 4.3\) Hz, 2H), 2.100 (s, 3H), 1.041 (t, \(J = 4.1\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 170.3, 145.7, 144.6, 132.6, 128.3, 127.7, 126.6, 126.4, 126.2, 126.1, 125.3, 122.3, 122.2, 111.6, 59.3, 26.4, 14.1. HRMS calcd for C\(_{19}\)H\(_{18}\)NO\(_2\)\([\text{M+H]}^+\) 292.1337, Found 292.1339.

![E-4ce](image1)

**Ethyl (E)-2,3-diphenyl-3-(thiophen-2-yl)acrylate**

Yellow oil; 69% yield (1152 mg); \(R_f=0.33\) (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3048, 3012, 2871, 1766, 1720, 1176, 1123, 778, 739; \(^{1}\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.385-7.354 (m, 5H), 7.352-7.323 (m, 5H), 7.177 (dd, \(J = 5.1, 1.2\) Hz, 1H), 6.768 (dd, \(J = 5.1, 3.7\) Hz, 1H), 6.557 (dd, \(J = 3.7, 1.2\) Hz, 1H), 3.895 (q, \(J = 7.1\) Hz, 2H), 0.862 (t, \(J = 7.1\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 169.5, 143.0, 141.8, 139.7, 137.4, 131.4, 130.9, 130.0, 129.4, 128.9, 128.5, 128.3, 128.1, 126.3, 100.0, 60.9, 13.7. HRMS calcd for C\(_{21}\)H\(_{18}\)NaO\(_2\)S\([\text{M+Na]}^+\) 357.0925, Found 357.0921.

![E-4cp](image2)

**Ethyl (E)-3-(3-ethoxy-3-oxo-1,2-diphenylprop-1-en-1-yl)benzoate**

Yellow oil; 72% yield (1440 mg); \(R_f=0.36\) (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3043, 3010, 2871, 1766, 1720, 1179, 1113, 769, 731; \(^{1}\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 8.023 (s, 1H), 7.693 (d, \(J = 7.0\) Hz, 1H), 7.492-7.266 (m, 4H), 7.150 (dd, \(J = 16.3, 6.5\) Hz, 7H), 6.993 (d, \(J = 6.6\) Hz, 1H), 4.392 (q, \(J = 6.9\) Hz, 2H), 4.037 (q, \(J = 6.9\) Hz, 2H), 1.382 (t, \(J = 7.1\) Hz, 3H), 1.007 (t, \(J = 5.6\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 170.1, 166.4, 144.9, 137.3, 137.2, 135.2, 133.7, 132.0, 130.9, 130.1, 130.0, 129.9, 129.2, 128.4, 128.34, 128.29, 128.0, 127.9, 61.1, 61.0, 14.4, 13.8. HRMS calcd for C\(_{26}\)H\(_{25}\)O\(_4\)\([\text{M+H]}^+\) 401.1753, Found 401.1754.
**Ethyl 2’-(trifluoromethyl)-3,4,5,6-tetrahydro-[1,1’-biphenyl]-2-carboxylate**

Yellow oil; 79% yield by method A (1177 mg); 70% yield by method B (1043 mg); \( R_f = 0.33 \) (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm\(^{-1}\), KBr): 3029, 2896, 1759, 1720, 1167, 873, 776; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \( \delta \) (ppm) 7.61 (d, \( J = 7.9 \) Hz, 1H), 7.485 (t, \( J = 7.5 \) Hz, 1H), 7.351 (t, \( J = 7.7 \) Hz, 1H), 7.120 (d, \( J = 7.7 \) Hz, 1H), 3.824 (qd, \( J = 7.1, 1.3 \) Hz, 2H), 2.502-2.269 (m, 4H), 1.73 (dd, \( J = 6.9, 4.4 \) Hz, 4H), 0.797 (t, \( J = 7.1 \) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \( \delta \) (ppm) 167.5, 146.3, 143.4 (d, \( J = 2.4 \) Hz, 1C), 131.3, 128.8, 127.8, 126.5, 125.9 (q, \( J = 49.6 \) Hz, 1C), 125.3, 123.5, 59.8, 34.6, 26.0, 22.3, 22.0, 13.5.HRMS calcd for C\(_{16}H_{17}F_3NaO_2\) \([M+Na]^+\) 321.1078, Found 321.1072.

**Ethyl 4’-(dimethylcarbamoyl)-3,4,5,6-tetrahydro-[1,1’-biphenyl]-2-carboxylate**

Yellow oil; 73% yield by method A (1099 mg); 66% yield by method B (993 mg); \( R_f = 0.23 \) (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm\(^{-1}\), KBr): 3022, 2893, 1769, 1720, 1644, 1267, 1162, 863, 767; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \( \delta \) (ppm) 7.358-7.330 (m, 2H), 7.160-7.132 (m, 2H), 3.872-3.846 (m, 2H), 3.032 (d, \( J = 36.7 \) Hz, 6H), 2.408-2.333 (m, 4H), 1.723 (d, \( J = 3.0 \) Hz, 4H), 0.877-0.833 (m, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \( \delta \) (ppm) 171.5, 169.7, 145.1, 144.8, 134.8, 128.6, 127.0, 126.9, 60.2, 39.7, 35.4, 32.6, 26.7, 22.5, 21.9, 13.7.HRMS calcd for C\(_{18}H_{23}N_2NaO_3\) \([M+Na]^+\) 324.1576, Found 324.1573.

**Methyl (Z)-3-(4-bromophenyl)-3-(pyridin-3-yl)acrylate**

Yellow oil; 76% yield (1205 mg); \( R_f = 0.24 \) (petroleum ether/ethyl acetate = 10:1, v/v).
IR (cm\(^{-1}\), KBr): 3030, 2950, 1735, 1716, 1430, 1286, 1128, 840; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 8.631 (dd, \(J = 4.9, 1.7\) Hz, 1H), 8.442 (dd, \(J = 2.2, 0.8\) Hz, 1H), 7.536-7.526 (m, 1H), 7.489-7.478 (m, 1H), 7.466-7.455 (m, 1H), 7.337 (ddd, \(J = 7.8, 4.9, 0.9\) Hz, 1H), 7.142-7.120 (m, 2H), 6.430 (s, 1H), 3.618 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 165.8, 152.3, 149.7, 149.5, 138.9, 136.7, 134.2, 132.0, 129.8, 124.6, 123.0, 118.8, 51.5. HRMS calcd for C\(_{15}\)H\(_{13}\)BrNO\(_2\) \([M+H]^+\) 318.0130, Found 318.0121.

**Methyl (E)-3-(4-bromophenyl)-3-(pyridin-3-yl)acrylate**

Yellow oil; 78% yield (1236 mg); \(R_f = 0.28\) (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm\(^{-1}\), KBr): 3033, 2953, 1732, 1718, 1430, 1286, 1125, 876; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 8.607 (dd, \(J = 5.6, 1.8\) Hz, 2H), 7.537 (ddd, \(J = 6.4, 4.5, 1.3\) Hz, 3H), 7.283 (d, \(J = 5.1\) Hz, 1H), 7.113-7.092 (m, 2H), 6.406 (s, 1H), 3.661 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 165.7, 152.7, 150.6, 149.0, 136.6, 136.2, 135.7, 131.5, 130.9, 123.4, 123.2, 118.7, 51.6. HRMS calcd for C\(_{15}\)H\(_{13}\)BrNO\(_2\) \([M+H]^+\) 318.0130, Found 318.0125.

**Ethyl (Z)-3-(4-methoxyphenyl)-2,3-diphenylacrylate\(^{[11]}\)**

Yellow oil; 76% yield (1360 mg); \(R_f = 0.30\) (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3023, 2954, 2821, 1759, 1719, 1130, 861, 749; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm) 7.223-7.201 (m, 2H), 7.174-7.091 (m, 8H), 7.015 (ddd, \(J = 7.2, 2.0, 0.8\) Hz, 2H), 6.868-6.846 (m, 2H), 4.081 (q, \(J = 7.1\) Hz, 2H), 3.822 (s, 3H), 1.048 (t, \(J = 7.1\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz) \(\delta\) (ppm) 170.9, 159.7, 145.7, 141.0, 137.9, 135.0, 133.0, 131.1, 130.6, 130.0, 128.2, 127.9, 127.7, 127.3, 113.6, 61.0, 55.4, 14.0.
Ethyl (E)-3-(4-methoxyphenyl)-2,3-diphenylacrylate\textsuperscript{[11]}

Yellow oil; 75\% yield (1342 mg); \( R_f = 0.35 \) (petroleum ether/ethyl acetate = 20:1, v/v).

IR (cm\(^{-1}\), KBr): 3030, 2954, 2824, 1759, 1729, 1130, 861, 746; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \( \delta \) (ppm) 7.318 (dd, \( J = 4.4, 2.0 \) Hz, 3H), 7.281-7.260 (m, 2H), 7.201-7.170 (m, 3H), 7.139-7.123 (m, 2H), 6.913-6.899 (m, 2H), 6.654-6.640 (m, 2H), 3.997 (q, \( J = 7.1 \) Hz, 2H), 3.737 (s, 3H), 0.941 (t, \( J = 7.1 \) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz,) \( \delta \) (ppm) 170.8, 159.2, 146.0, 143.0, 138.1, 133.0, 132.5, 130.0, 129.3, 128.3, 128.1, 128.1, 127.3, 113.3, 60.9, 55.2, 13.8.

Ethyl 2-(naphthen-2-yl)cyclohex-1-ene-1-carboxylate

Yellow oil; 77\% yield (1078 mg); \( R_f = 0.37 \) (petroleum ether/ethyl acetate = 50:1, v/v).

IR (cm\(^{-1}\), KBr): 3028, 2976, 1780, 1736, 1137, 758; \(^1\)H NMR (CDCl\(_3\), 400 MHz) \( \delta \) (ppm) 7.856-7.804 (m, 2H), 7.766-7.745 (m, 1H), 7.471-7.413 (m, 3H), 7.180 (dd, \( J = 7.0, 1.0 \) Hz, 1H), 3.680-3.580 (m, 2H), 2.615-2.405 (m, 4H), 1.922-1.833 (m, 4H), 0.411 (t, \( J = 7.1 \) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\), 101 MHz,) \( \delta \) (ppm) 168.7, 146.2, 142.2, 133.6, 131.0, 129.4, 128.3, 126.8, 125.9, 125.7, 125.4, 125.2, 123.6, 59.7, 34.1, 26.3, 22.7, 22.4, 13.2; HRMS calcd for C\(_{19}\)H\(_{20}\)NaO\(_2\)\(^+\) [M+Na]\(^+\) 303.1361, Found 303.1356.

1,2,3,4-Tetrahydro-8-methyl-3-(phenylmethyl)-5H-[1]benzopyran-3,4-c pyridin-5-one\textsuperscript{[12]}

Yellow solid; 70\% yield (1068 mg); \( R_f = 0.3 \) (petroleum ether/ethyl acetate = 20:1, v/v).
IR (cm$^{-1}$, KBr): 3021, 2981, 1786, 1726, 1619, 1453, 1139, 758; $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ (ppm) 7.586 (d, $J = 7.4$ Hz, 1H), 7.364-7.193 (m, 7H), 3.710 (s, 2H), 3.266 (s, 2H), 2.898 (t, $J = 14.8$ Hz, 2H), 2.750 (t, $J = 14.6$ Hz, 2H), 2.406 (s, 3H); $^{13}$C NMR (CDCl$_3$, 101 MHz,) $\delta$ (ppm) 170.1, 167.1, 156.2, 144.7, 138.4, 129.4, 129.1, 128.6, 128.4, 128.0, 127.2, 121.1, 111.6, 60.4, 59.6, 56.3, 28.8, 20.1.
9. References


10. $^1$H and $^{13}$C NMR spectra for products
Z-3ag
Z-3bb
S76
EtOOC—\(\text{Ph}^{\text{Ph}}\text{Ph}\text{COOEt}\)

Z-4cp
\text{S121}