

Visible light-Induced Intermolecular Addition Reactions between Alkyl-Bromocarboxylates and Enamines

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1. General Information

Unless otherwise noted, all reactions were carried out under an atmosphere of nitrogen using standard Schlenk techniques. Materials were purchased from commercial suppliers and used without further purification. Anhydrous DMF, CH₃CN, DMSO, DCM were freshly distilled from calcium hydride, Anhydrous PhMe was freshly distilled from Sodium. ¹H NMR and ¹³C NMR spectra were recorded on a 400 MHz spectrometer. The chemical shifts for ¹H NMR were recorded in ppm downfield from tetramethylsilane (TMS) with the solvent resonance as the internal standard. The chemical shifts for ¹³C NMR were recorded in ppm downfield using the central peak of deuteriochloroform (77.00 ppm) as the internal standard. Coupling constants (*J*) are reported in Hz and refer to apparent peak multiplications. Analytical GC was performed on an Agilent 7890A with FID detector. HRMS were performed under ESI ionization technique on a Waters Micromass Q-TOF Premier Mass Spectrometer. Flash column chromatography was performed on silica gel (300-400 mesh).

2. Preparation of substrates

2.1 Representative procedure for the preparation of enamines. (1a-1l)¹

To a solution of acetophenone (10.0 g, 86.0 mmol) and piperidine (42.5 g, 516.0 mmol) in anhydrous hexane (200 mL), was added TiCl₄ (17.8 g, 86.0 mmol) over 30 min at 0 °C. The reaction mixture was stirred at room temperature for 24 h and filtered. The filtrate was evaporated under vacuum to give colorless oil, which was distilled under reduced pressure (1 mmHg, 99 °C) to give N-(1-styryl)piperidine (**1b**) as a pale yellow liquid (12.0 g, 82% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.45 (m, 2H), 7.33–7.28 (m, 3H), 4.24 (s, 1H), 4.15 (s, 1H), 2.82–2.79 (m, 4H), 1.62–1.53 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 158.0, 140.3, 128.0, 127.7, 127.7, 90.1, 50.5, 26.0, 24.5.

2.2 A General procedure for the preparation of cinnamyl 2-bromopropanoate.⁷

To a mixture of 5.9 g (80.0 mmol) of propionic acid and 4.3 g (16.0 mmol) of tribromophosphine in a 50 ml three-necked flask, bromine (25.6 g, 160.1mmol) was added dropwise at 80 °C over 30 min. After the addition is complete, the solution was heated over a

period of 3 hours. The excess bromine and hydrogen bromide are removed under reduced pressure. To a solution of 4.0 g (30.0 mmol) of cinnamyl alcohol, 0.4 g (3.0 mmol) of DMAP and 4.8 g (60.0 mmol) of pyridine in 30 mL DCM was cooled at 0 °C. The α -bromopropanoyl bromide was added dropwise slowly at such a rate to maintain the internal temperature below 20 °C for 30 min. After completion, the reaction was quenched with H₂O (30 mL) and extracted with DCM (3×30 mL). The combined organic extracts were dried over Na₂SO₄ and the solvent was removed under reduced pressure. The crude product was purified by silica-gel column chromatography to give cinnamyl 2-bromopropanoate as pale yellow oil (5.7 g, 70% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.44–7.24 (m, 5H), 6.71 (d, J = 16.0 Hz, 1H), 6.29 (dt, J = 15.6, 6.4 Hz, 1H), 4.83 (dd, J = 6.0, 0.8 Hz, 2H), 4.42 (q, J = 6.8 Hz, 1H), 1.86 (d, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 169.9, 135.9, 134.8, 128.5, 128.1, 126.6, 122.1, 66.3, 40.0, 21.6.

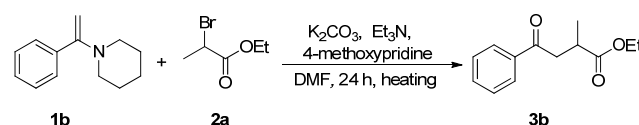
2.3 A General procedure for the preparation of allyl 2-bromo-2-phenylacetate.⁷

To a mixture of 17.7 g (130.0 mmol) of phenylacetic acid and 7.0 g (26.0 mmol) of tribromophosphine in a 100 mL three-necked flask, the bromine (41.6 g, 260.0 mmol) was added dropwise at 100 °C over 30 min. After the addition is complete, the solution was heated over a period of 4 hours. The excess bromine and hydrogen bromide are removed under reduced pressure. To a solution of 7.6 g (130.0 mmol) of allyl alcohol and 13.8 g (136.5 mmol) of Et₃N in 30 mL DCM was cooled at 0 °C. The solution of 2-bromo-2-phenylacetyl bromide in 20 mL was added dropwise slowly at such a rate to maintain the internal temperature below 20 °C for 30 min. After 1h, the reaction was quenched with H₂O (50 mL) and extracted with DCM (3×50 mL). The combined organic extracts were dried over Na₂SO₄ and the solvent was removed under reduced pressure to give colorless oil, which was distilled under reduced pressure (2 mmHg, 100 °C) to give allyl 2-bromo-2-phenylacetate as a colorless oil (17.5 g, 53% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.57–7.53 (m, 2H), 7.38–7.34 (m, 3H), 5.95–5.85 (m, 1H), 5.38 (s, 1H), 5.32 (dq, J = 17.2, 1.6 Hz, 1H), 5.25 (dq, J = 10.4, 1.2 Hz, 1H), 4.69–4.66 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 167.8, 135.6, 131.0, 129.2, 128.7, 128.6, 118.9, 66.7, 46.5.

3. A general procedure for Ru(bpy)₃Cl₂-catalyzed reaction between enamines and alkyl bromocarboxylates under visible light

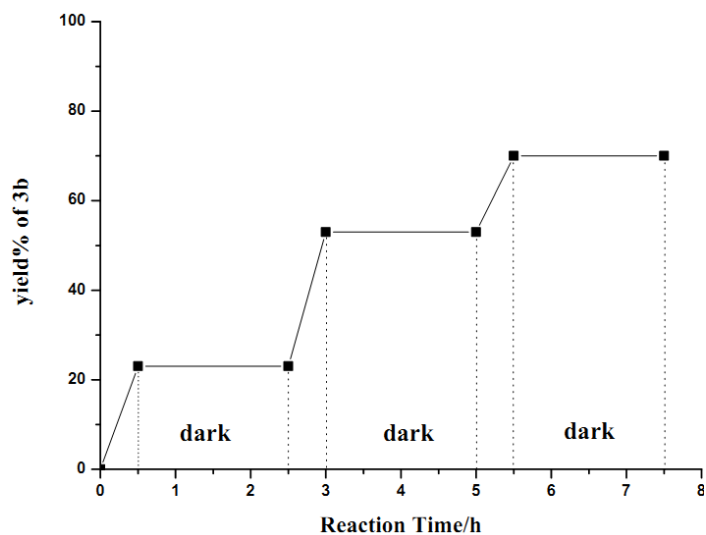
A dried Schlenk tube equipped with a stirrer bar which was evacuated and backfilled with nitrogen was added alkyl bromocarboxylate (1.0 mmol, 181.0 mg), K₂CO₃ (0.5 mmol, 69 mg), Ru(bpy)₃Cl₂ (0.01 mmol, 7.48 mg), Et₃N (0.2 mmol, 20.4 mg), 4-methoxy pyridine (0.2 mmol, 218.2 mg) and enamine (0.5 mmol). Then 2 mL of DMF was added into the reaction tube via a syringe. The reaction mixture was degassed by the freeze-pump-thaw method and then irradiated with a 23W fluorescent household light bulb (distance app. 5 cm) for 24 h. After the completion of the reaction, it was quenched by water and extracted with ethyl acetate (3 x 15 mL). The organic layers were combined and the pure product was obtained by flash column chromatography on silica gel.

4. The control experiment of the reaction conducted in the different temperature in the dark.^a



reaction temp. (°C)	yield ^b (%)
40	5
60	10
80	8
100	5

^aConditions: **1b** (0.5 mmol), **2a** (1 mmol), Et₃N (0.2 mmol), 4-methoxy pyridine (0.2 mmol), base (0.5 mmol), solvent (2 mL), heated from 40 to 100 °C for 24 h. ^bIsolated yields.

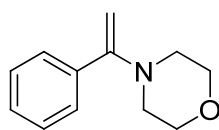


The experiment of turn on and off the light

5. Spectral data for substrates and products

5.1. Spectral data for substrates

N-(1-phenylvinyl)morpholine¹

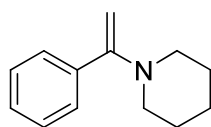


1a

Pale yellow liquid, 86% yield (1 mmHg, 120 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.45 (m, 2H), 7.33–7.31(m, 3H), 4.33 (s, 1H), 4.19 (s, 1H), 3.78–3.76 (m, 4H), 2.84–2.82 (m, 4H).

¹³C NMR (100 MHz, CDCl₃) δ 157.0, 139.0, 128.1, 128.0, 127.7, 91.0, 66.8, 49.7.

N-(1-phenylvinyl)piperidine²

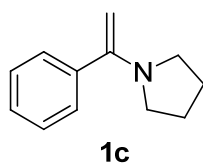


1b

Pale yellow liquid, 82% yield (1 mmHg, 99 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.45 (m, 2H), 7.33–7.28 (m, 3H), 4.24 (s, 1H), 4.15 (s, 1H), 2.82–2.79 (m, 4H), 1.62–1.53 (m, 6H). ¹³C

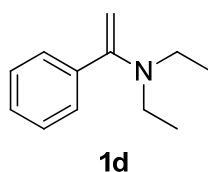
NMR (100 MHz, CDCl₃) δ 158.0, 140.3, 128.0, 127.7, 127.7, 90.1, 50.5, 26.0, 24.5.

N-(1-phenylvinyl)pyrrolidine³



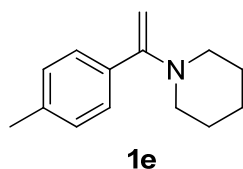
Pale yellow liquid, 88% yield (1 mmHg, 96 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.43–7.41 (m, 2H), 7.36–7.31 (m, 3H), 3.89 (s, 1H), 3.85 (s, 1H), 3.04–3.00 (m, 4H), 1.91–1.88 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 154.3, 140.5, 127.7, 127.7, 127.4, 84.2, 49.1, 24.9.

N,N-diethyl-1-phenylethanamine



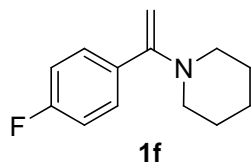
Pale yellow liquid, 85% yield (1 mmHg, 60–61 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.45–7.41 (m, 2H), 7.33–7.28 (m, 3H), 4.15 (s, 1H), 4.06 (s, 1H), 2.99 (q, *J* = 7.2 Hz, 4H), 1.03 (t, *J* = 7.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 155.0, 141.0, 128.5, 128.2, 127.9, 127.9, 127.6, 90.1, 43.1, 11.6.

N-(1-(p-tolyl)vinyl)piperidine⁵



Pale yellow liquid, 79% yield (0.1 mmHg, 78 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.37–7.34 (m, 2H), 7.14–7.12 (m, 2H), 4.21 (s, 1H), 4.10 (s, 1H), 2.81–2.78 (m, 4H), 2.35 (s, 3H), 1.64–1.58 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 158.0, 137.5, 137.4, 128.7, 127.6, 89.5, 50.5, 6.0, 24.5, 21.1.

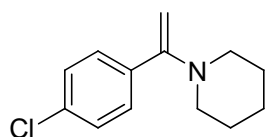
N-(1-(4-fluorophenyl)vinyl)piperidine



Pale yellow liquid, 83% yield (1 mmHg, 125 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.46–7.40 (m, 2H), 7.03–6.97 (m, 2H), 4.20 (s, 1H), 4.13 (s, 1H), 2.76–2.79 (m, 4H), 1.64–1.52 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 163.7, 161.3, 157.0, 136.2, 129.2, 129.1, 114.9, 114.7, 90.3,

50.5, 26.0, 24.4. ^{19}F NMR (376 MHz, CDCl_3) δ -115.23. HRMS-ESI (m/z): Calculated for $\text{C}_{13}\text{H}_{16}\text{FN}$ (M + H) $^+$: 206.1345, Found: 206.1330.

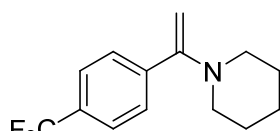
N-(1-(4-chlorophenyl)vinyl)piperidine⁵



1g

Pale yellow liquid, 75% yield (0.06 mmHg, 80–83 °C). ^1H NMR (400 MHz, CDCl_3) δ 7.44–7.40 (m, 2H), 7.32–7.28 (m, 2H), 4.25 (s, 1H), 4.17 (s, 1H), 2.81–2.78 (m, 4H), 1.66–1.57 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 156.9, 138.8, 133.4, 128.9, 128.2, 90.8, 50.5, 25.9, 24.4.

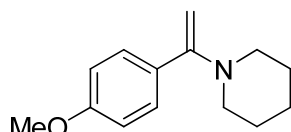
N-(1-(4-(trifluoromethyl)phenyl)vinyl)piperidine



1h

Pale yellow liquid, 79% yield (1 mmHg, 128 °C). ^1H NMR (400 MHz, CDCl_3) δ 7.57 (s, 4H), 4.30 (s, 1H), 4.23 (s, 1H), 2.79–2.77 (m, J = 5.6 Hz, 4H), 1.65–1.52 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 156.8, 144.0, 130.1, 130.0, 129.7, 129.6, 129.4, 129.3, 127.9, 125.59, 125.0, 122.8, 91.9, 50.5, 25.9, 24.4. ^{19}F NMR (376 MHz, CDCl_3) δ -62.86. HRMS-ESI (m/z): Calculated for $\text{C}_{14}\text{H}_{16}\text{F}_3\text{N}$ (M + H) $^+$: 256.1313, Found: 256.1301.

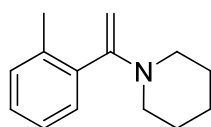
N-(1-(4-methoxyphenyl)vinyl)piperidine⁵



1i

Pale yellow liquid, 72% yield (0.1 mmHg, 92–94 °C). ^1H NMR (400 MHz, CDCl_3) δ 7.40–7.26 (m, 2H), 6.87–6.83 (m, 2H), 4.18 (s, 1H), 4.08 (s, 1H), 3.81 (s, 3H), 2.81–2.78 (m, 4H), 1.63–1.54 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.4, 157.6, 132.7, 128.7, 113.36, 89.0, 77.3, 77.0, 76.7, 55.2, 50.5, 26.0, 24.6.

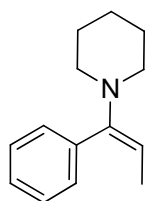
N-(1-(o-tolyl)vinyl)piperidine



1j

Pale yellow liquid, 83% yield (1 mmHg, 125 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.23–7.11 (m, 4H), 4.13 (s, 1H), 3.83 (s, 1H), 2.79–2.77 (m, 4H), 2.35 (s, 3H), 1.56–1.52 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 156.4, 140.2, 136.4, 129.8, 129.6, 127.3, 125.2, 87.6, 48.8, 25.8, 24.5, 19.8. HRMS-ESI (m/z): Calculated for C₁₄H₁₉N (M + H)⁺: 202.1596, Found: 202.1656.

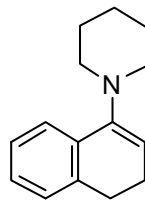
N-(1-phenylprop-1-en-1-yl)piperidine⁶



1k

Pale yellow liquid, 80% yield (1 mmHg, 95–97 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.34–7.33 (m, 3H), 7.29–7.26 (m, 2H), 4.67 (q, *J* = 6.8 Hz, 1H), 2.68–2.66 (m, 4H), 1.59–1.49 (m, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 151.1, 138.6, 129.7, 127.8, 127.1, 99.1, 50.6, 26.2, 24.6, 13.9. HRMS-ESI (m/z): Calculated for C₁₄H₁₉N (M + H)⁺: 202.1596, Found: 202.1664.

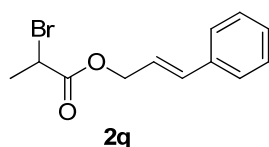
N-(3,4-dihydronaphthalen-1-yl)piperidine⁶



1l

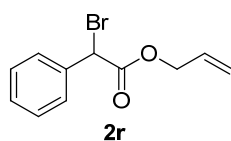
Pale yellow liquid, 70% yield (0.7 mmHg, 120–125 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, *J* = 7.2 Hz, 1H), 7.22–7.17 (m, 1H), 7.14–7.12 (m, 2H), 5.24 (t, *J* = 4.8 Hz, 1H), 2.76 (s, 4H), 2.69–2.65 (m, 2H), 2.26–2.18 (m, 2H), 1.71 (dd, *J* = 11.6, 6.0 Hz, 4H), 1.57 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 148.6, 138.0, 132.9, 127.5, 126.5, 126.0, 123.4, 106.7, 52.0, 28.7, 26.3, 24.7, 22.5. HRMS-ESI (m/z): Calculated for C₁₅H₁₉N (M + H)⁺: 214.1596, Found: 214.1628.

cinnamyl 2-bromopropanoate⁷



Pale yellow oil, 70% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.44–7.24 (m, 5H), 6.71 (d, J = 16.0 Hz, 1H), 6.29 (dt, J = 15.6, 6.4 Hz, 1H), 4.83 (dd, J = 6.0, 0.8 Hz, 2H), 4.42 (q, J = 6.8 Hz, 1H), 1.86 (d, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 135.9, 134.8, 128.5, 128.1, 126.6, 122.1, 66.3, 40.0, 21.6.

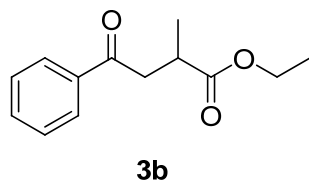
allyl 2-bromo-2-phenylacetate



Colorless oil, 53% yield (2 mmHg, 100 °C). ^1H NMR (400 MHz, CDCl_3) δ 7.57–7.53 (m, 2H), 7.38–7.34 (m, 3H), 5.95–5.85 (m, 1H), 5.38 (s, 1H), 5.32 (dq, J = 17.2, 1.6 Hz, 1H), 5.25 (dq, J = 10.4, 1.2 Hz, 1H), 4.69–4.66 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.8, 135.6, 131.0, 129.2, 128.7, 128.6, 118.9, 66.7, 46.5.

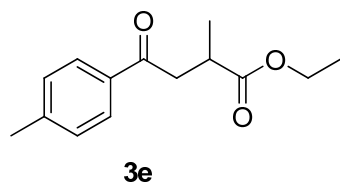
5.2. Spectral data for products

ethyl 2-methyl-4-oxo-4-phenylbutanoate⁸



Yellow liquid, 94% yield, ^1H NMR (400 MHz, CDCl_3) δ 8.00–7.92 (m, 2H), 7.59–7.53 (m, 1H), 7.51–7.41 (m, 2H), 4.15 (q, J = 7.2 Hz, 2H), 3.48 (dd, J = 17.6, 8.0 Hz, 1H), 3.16–3.06 (m, 1H), 3.00 (dd, J = 17.6, 5.6 Hz, 1H), 1.29–1.22 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 198.0, 175.9, 136.7, 133.1, 128.5, 127.9, 60.5, 41.9, 35.0, 17.2, 14.1.

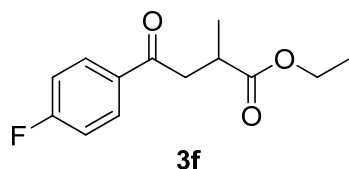
ethyl 2-methyl-4-oxo-4-(p-tolyl)butanoate



Yellow liquid, 92% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.94–7.79 (m, 2H), 7.26–7.24 (m,

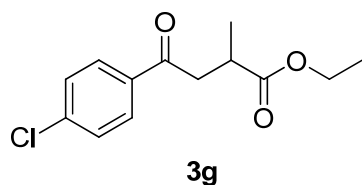
2H), 4.14 (q, $J = 7.2$ Hz, 2H), 3.44 (dd, $J = 17.6, 8.0$ Hz, 1H), 3.15–3.05 (m, 1H), 2.98 (dd, $J = 17.6, 5.6$ Hz, 1H), 2.40 (s, 3H), 1.28–1.22 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 197.4, 175.7, 143.7, 134.1, 129.0, 127.9, 60.3, 41.6, 34.9, 21.4, 17.1, 14.0. HRMS-ESI (m/z): Calculated for $\text{C}_{14}\text{H}_{18}\text{O}_3$ ($\text{M} + \text{H}$) $^+$: 235.1334, Found: 235.1337.

ethyl 4-(4-fluorophenyl)-2-methyl-4-oxobutanoate



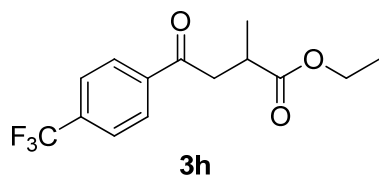
Yellow liquid, 92% yield, ^1H NMR (400 MHz, CDCl_3) δ 8.04–7.95 (m, 2H), 7.17–7.09 (m, 2H), 4.15 (q, $J = 7.2$ Hz, 2H), 3.45 (dd, $J = 17.6, 8.0$ Hz, 1H), 3.16–3.06 (m, 1H), 2.96 (dd, $J = 17.6, 5.6$ Hz, 1H), 1.26 (dd, $J = 14.4, 7.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 196.4, 175.8, 166.9, 164.4, 133.1, 130.6, 130.5, 115.7, 115.5, 60.5, 41.7, 35.0, 17.2, 14.1. ^{19}F NMR (376 MHz, CDCl_3) δ -105.57. HRMS-ESI (m/z): Calculated for $\text{C}_{13}\text{H}_{15}\text{FO}_3$ ($\text{M} + \text{Na}$) $^+$: 261.0903, Found: 261.0707.

ethyl 4-(4-chlorophenyl)-2-methyl-4-oxobutanoate⁹



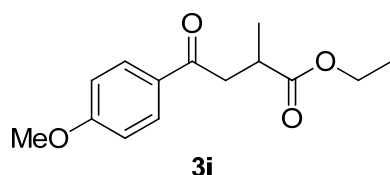
Yellow liquid, 91% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.96–7.88 (m, 2H), 7.47–7.40 (m, 2H), 4.14 (q, $J = 7.2$ Hz, 2H), 3.45 (dd, $J = 17.6, 8.0$ Hz, 1H), 3.15–3.06 (m, 1H), 2.95 (dd, $J = 17.6, 5.2$ Hz, 1H), 1.30–1.22 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 196.8, 175.7, 139.5, 134.9, 129.3, 128.8, 60.5, 41.7, 34.9, 17.2, 14.0.

ethyl 2-methyl-4-oxo-4-(4-(trifluoromethyl)phenyl)butanoate



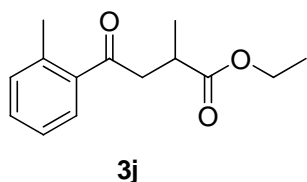
Yellow liquid, 78% yield, ^1H NMR (400 MHz, CDCl_3) δ 8.07 (d, $J = 8.2$ Hz, 2H), 7.73 (d, $J = 8.4$ Hz, 2H), 4.15 (q, $J = 7.2$ Hz, 2H), 3.51 (dd, $J = 17.6, 8.0$ Hz, 1H), 3.18–3.09 (m, 1H), 2.99 (dd, $J = 17.6, 5.2$ Hz, 1H), 1.31–1.23 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 197.2, 175.6, 139.3, 134.5, 134.2, 128.3, 125.6, 125.5, 124.9, 122.2, 60.6, 42.1, 35.0, 17.2, 14.2. ^{19}F NMR (376 MHz, CDCl_3) δ -63.55. HRMS-ESI (m/z): Calculated for $\text{C}_{14}\text{H}_{15}\text{F}_3\text{O}_3$ ($\text{M} + \text{H}$) $^+$: 289.1052, Found: 289.1064.

ethyl 4-(4-methoxyphenyl)-2-methyl-4-oxobutanoate¹⁰



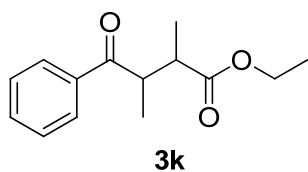
^1H NMR (400 MHz, CDCl_3) δ 7.97–7.93 (m, 2H), 6.95–6.91 (m, 2H), 4.15 (q, $J = 6.8$ Hz, 2H), 3.87 (s, 3H), 3.43 (dd, $J = 17.2, 8.0$ Hz, 1H), 3.15–3.06 (m, 1H), 2.97 (dd, $J = 17.2, 5.6$ Hz, 1H), 1.29–1.22 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 196.5, 176.0, 163.4, 130.2, 129.7, 113.6, 60.4, 55.3, 41.4, 35.0, 17.2, 14.0.

ethyl 2-methyl-4-oxo-4-(o-tolyl)butanoate



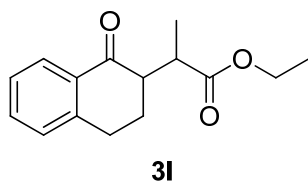
Yellow liquid, 97% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.68 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.36 (td, $J = 7.2, 1.2$ Hz, 1H), 7.28–7.21 (m, 2H), 4.15 (q, $J = 7.2$ Hz, 2H), 3.40 (dd, $J = 17.6, 8.0$ Hz, 1H), 3.14–3.05 (m, 1H), 2.91 (dd, $J = 17.6, 5.6$ Hz, 1H), 2.48 (s, 3H), 1.25 (t, $J = 7.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 202.0, 175.8, 138.0, 137.6, 131.8, 131.2, 128.4, 125.6, 60.5, 44.7, 35.2, 21.2, 17.2, 14.1. HRMS-ESI (m/z): Calculated for $\text{C}_{14}\text{H}_{18}\text{O}_3$ ($\text{M} + \text{Na}$) $^+$: 257.1154, Found: 257.1155.

ethyl 2,3-dimethyl-4-oxo-4-phenylbutanoate⁸



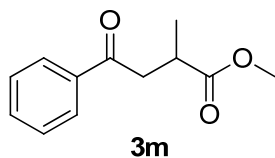
Yellow liquid, 66% yield, dr = 11:1 ¹H NMR (400 MHz, CDCl₃) δ 8.02–7.92 (m, 2H), 7.60–7.53 (m, 1H), 7.51–7.42 (m, 2H), 4.15–3.96 (m, 2H), 3.80–3.72 (m, 1H), 3.02–2.89 (m, 1H), 1.29–1.12 (m, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 203.4, 175.7, 136.0, 132.9, 128.6, 128.3, 60.5, 43.0, 41.7, 14.5, 14.1, 14.0.

ethyl 2-(1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl)propanoate⁸



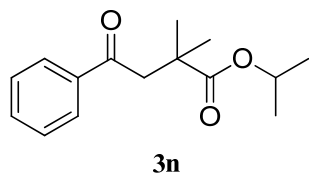
Yellow liquid, 33% yield, dr = 3:1 ¹H NMR (400 MHz, CDCl₃) for major δ 8.02 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.47 (td, *J* = 11.2, 1.2 Hz, 1H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 4.23–4.16 (m, 2H), 3.22–2.98 (m, 4H), 2.21–2.15 (m, 1H), 1.99–1.88 (m, 1H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.17 (d, *J* = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 198.0, 176.0, 143.9, 133.3, 132.3, 128.6, 127.4, 126.6, 60.5, 50.2, 38.7, 29.4, 25.2, 14.2, 13.1.

methyl 2-methyl-4-oxo-4-phenylbutanoate¹¹



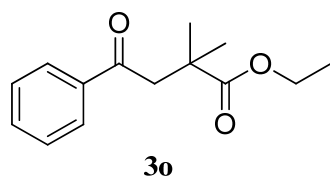
Yellow liquid, 90% yield, ¹H NMR (400 MHz, CDCl₃) δ 7.98–7.95 (m, 2H), 7.59–7.53 (m, 1H), 7.51–7.41 (m, 2H), 3.70 (s, 3H), 3.51–3.44 (m, 1H), 3.14 (qd, *J* = 7.2, 1.6 Hz, 1H), 3.02 (dd, *J* = 17.6, 5.6 Hz, 1H), 1.29–1.27 (m, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 197.9, 176.3, 136.5, 133.0, 128.5, 127.9, 51.8, 41.9, 34.8, 17.2.

isopropyl 2,2-dimethyl-4-oxo-4-phenylbutanoate



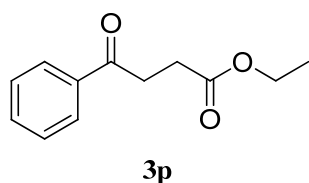
Yellow liquid, 78% yield, ^1H NMR (400 MHz, CDCl_3) δ 8.03–8.00 (m, 2H), 7.60–7.55 (m, 1H), 7.51–7.45 (m, 2H), 4.36–4.28 (m, 1H), 4.13–4.04 (m, 4H), 2.83 (dd, $J = 16.4, 7.2$ Hz, 2H), 2.51 (dd, $J = 16.4, 6.8$ Hz, 2H), 1.19 (t, $J = 7.2$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 201.0, 171.4, 135.8, 133.2, 128.7, 128.5, 60.8, 38.7, 36.0, 29.7, 14.0. HRMS-ESI (m/z): Calculated for $\text{C}_{15}\text{H}_{20}\text{O}_3$ ($M + \text{H}$) $^+$: 249.1491, Found: 249.1482.

ethyl 2,2-dimethyl-4-oxo-4-phenylbutanoate¹²



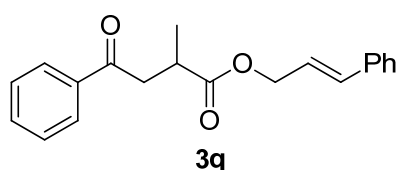
Yellow liquid, 62% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.95–7.92 (m, 2H), 7.57–7.53 (m, 1H), 7.47–7.43 (m, 2H), 4.13 (q, $J = 7.2$ Hz, 2H), 3.28 (s, 2H), 1.32 (s, 6H), 1.20 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 197.6, 177.2, 137.0, 132.9, 128.5, 127.8, 60.4, 48.4, 40.0, 29.6, 25.7, 14.0.

ethyl 4-oxo-4-phenylbutanoate¹³



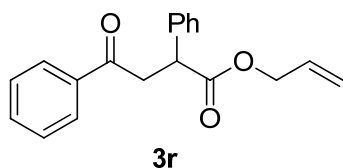
Yellow liquid, 66% yield, ^1H NMR (400 MHz, CDCl_3) δ 8.01–7.92 (m, 2H), 7.59–7.52 (m, 1H), 7.50–7.42 (m, 2H), 4.16 (q, $J = 7.2$ Hz, 2H), 3.31 (t, $J = 6.4$ Hz, 2H), 2.75 (t, $J = 6.4$ Hz, 2H), 1.26 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 198.0, 172.7, 136.5, 128.5, 127.9, 60.5, 33.3, 28.2, 14.0.

cinnamyl 2-methyl-4-oxo-4-phenylbutanoate



Yellow liquid, 68% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.98–7.96 (m, 2H), 7.58–7.24 (m, 9H), 6.65 (d, $J = 16.0$ Hz, 1H), 6.27 (dt, $J = 15.6, 6.4$ Hz, 1H), 4.76 (dt, $J = 6.4$ Hz, 1.6, 2H), 3.51 (dd, $J = 17.6, 8.0$ Hz, 1H), 3.22–3.16 (m, 1H), 3.05 (dd, $J = 17.6, 5.6$ Hz, 1H), 1.32 (d, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 197.9, 175.6, 136.5, 136.2, 133.8, 133.1, 128.5, 128.5, 127.9, 127.9, 126.5, 123.1, 65.1, 41.8, 35.0, 17.3. HRMS-ESI (m/z): Calculated for $\text{C}_{20}\text{H}_{20}\text{O}_3$ ($M + \text{H}$) $^+$: 309.1491, Found: 309.1481.

allyl 4-oxo-2,4-diphenylbutanoate¹⁴



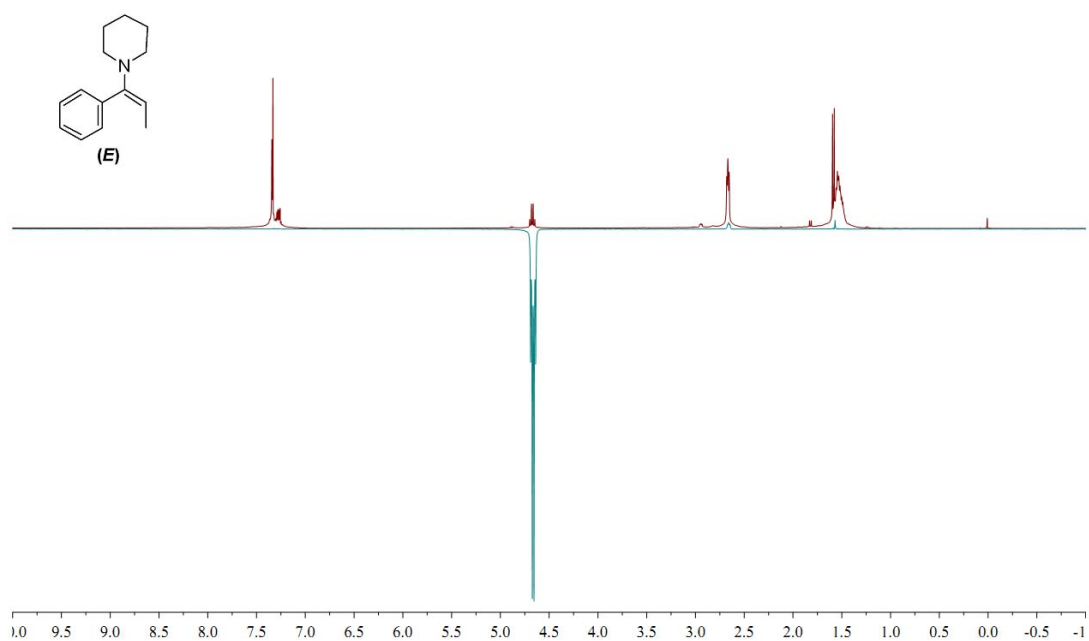
Yellow liquid, 77% yield, ^1H NMR (400 MHz, CDCl_3) δ 7.98 (m, 2H), 7.60–7.26 (m, 8H), 5.92–5.78 (m, 1H), 5.24–5.14 (m, 2H), 4.67–4.55 (m, 2H), 4.33 (dd, $J = 10.4, 4.4$ Hz, 1H), 3.96 (dd, $J = 18.0, 10.4$ Hz, 1H), 3.29 (dd, $J = 18.0, 4.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 197.5, 173.0, 138.2, 136.3, 133.3, 131.9, 128.8, 128.5, 128.0, 127.8, 127.5, 117.8, 65.5, 46.4, 42.7.

6. Reference

- 1 W. J. Zhao, M. Yan, D. Huang and S. J. Ji, *Tetrahedron*, 2005, **61**, 5585.
- 2 J. Paleček, O. Paleta, *Synthesis*, 2004, **4**, 521.
- 3 K. Blau, L. Burgemeister, J. Grasnack, V. Voerckel, *Journal für Praktische Chemie (Leipzig)*, 1991, **333**, 455.
- 4 F. Ando, K. Ohashi, J. Koketsu, *Bull. Chem. Soc. Jpn.*, 1976, **49**, 727.
- 5 M. K. Meilahn, B. Cox, M. E. Munk, *J. Org. Chem.*, 1975, **40**, 819.
- 6 R. F. Parcell, F. P. Hauck Jr, *J. Org. Chem.*, 1963, **28**, 3468.
- 7 J. Ishihara, Y. Watanabe, N. Koyama, Y. Nishino, K. Takahashi, S. Hatakeyama, *Tetrahedron*, 2011, **67**, 3659.
- 7 N. Fujisawa, A. Hosomi, K. Miura, H. Saito, D. Wang, *Org. Lett.*, 2001, **3**, 2591.
- 8 C. G. Daniliuc, F. Glorius, N. E. Wurcz, *Chem. Eur. J.*, 2012, **18**, 16297.

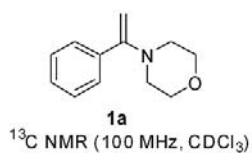
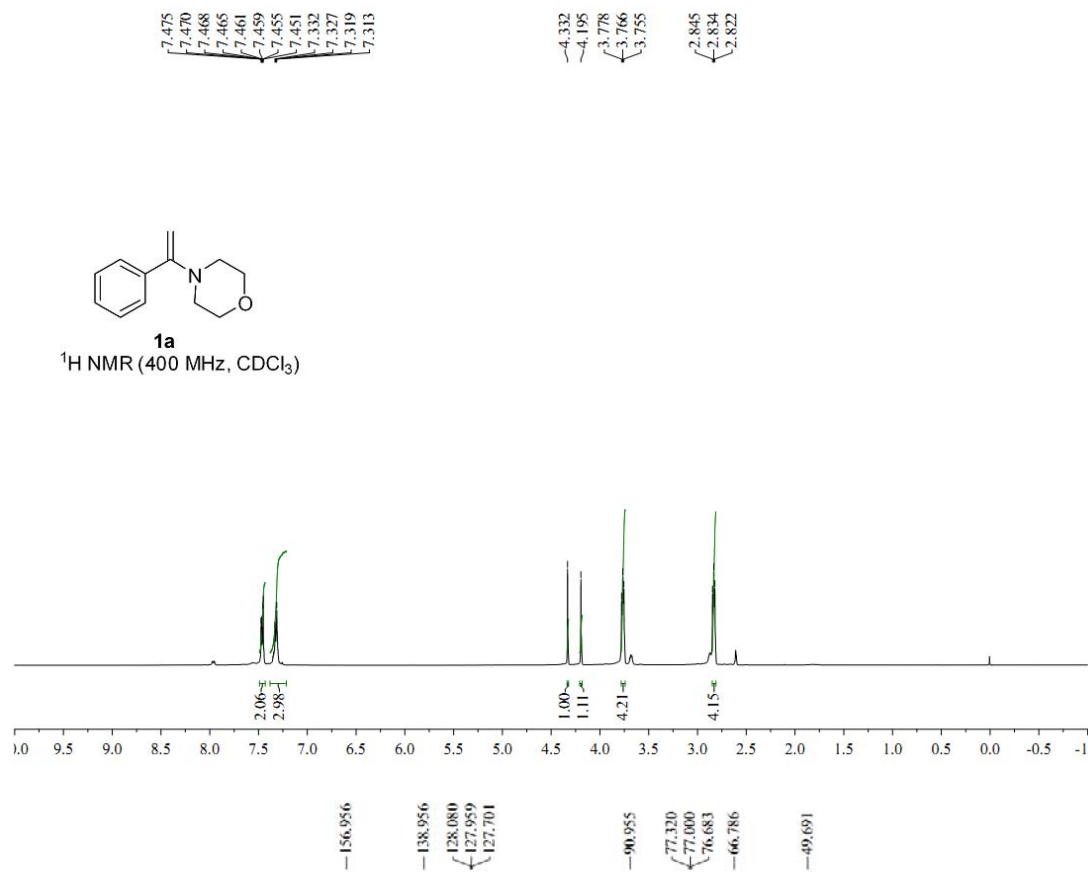
- 9 T. Fujimura, S. Aoki, E. Nakamura, *J. Org. Chem.*, 1991, **56**, 2809.
- 10 E. A. Jo, C. H. Jun, *Eur. J. Org. Chem.*, 2006, **11**, 2504.
- 11 S. A. Barnett, Y. D. Cai, B. P. Roberts, D. A. Tocher, *Org. Biomol. Chem.*, 2004, **2**, 2517.
- 12 L. H. P. Meijer, U. K. Pandit, *Tetrahedron*, 1985, **41**, 467.
- 13 G. N. Gururaja, I. N. N. Namboothiri, S. M. Mobin, *Eur. J. Org. Chem.*, 2011, **11**, 2048.

7. The NOE experiment of N-(1-phenylprop-1-en-1-yl)piperidine (**11**)



8. NMR spectra of the products

N-(1-phenylvinyl)morpholine (1a)

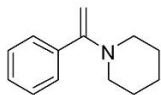


N-(1-phenylvinyl)piperidine (1b)

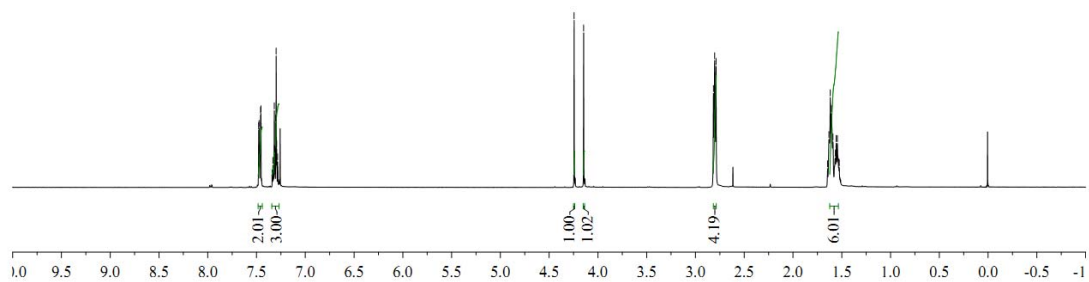
7.478
7.473
7.458
7.454
7.332
7.318
7.313
7.303
7.299
7.294
7.284

4.244
4.147

2.816
2.803
2.789
1.645
1.631
1.618
1.605
1.591
1.566
1.558
1.545
1.532
1.526



1b
 $^1\text{H NMR}$ (400 MHz, CDCl_3)

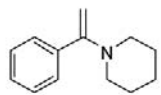


158.037
140.289
128.003
127.727
127.661

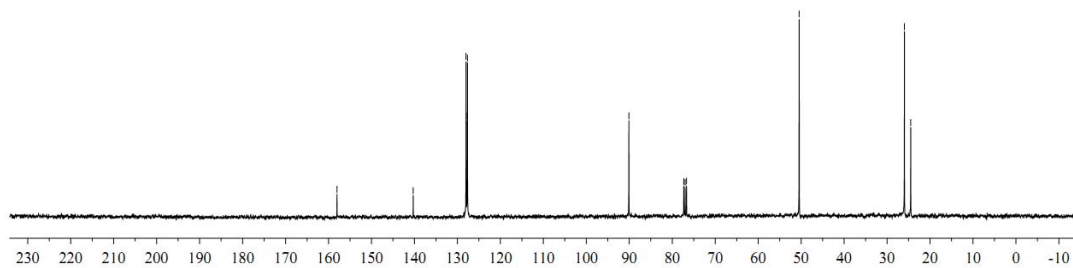
90.095
77.318
77.000
76.682

50.459

25.969
24.499



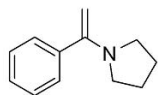
1b
 $^{13}\text{C NMR}$ (100 MHz, CDCl_3)



N-(1-phenylvinyl)pyrrolidine (1c)

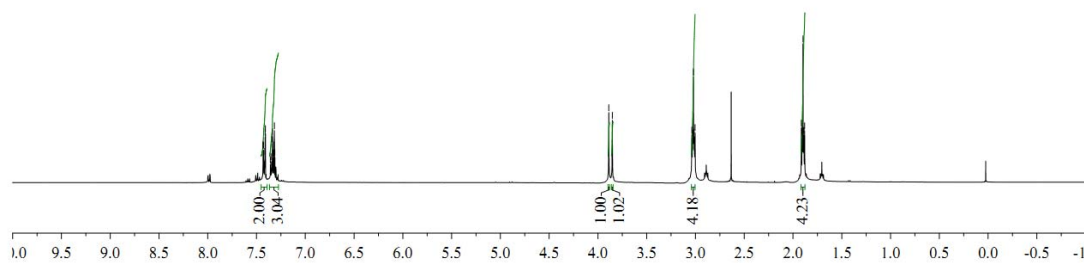
7.433
7.428
7.415
7.413
7.409
7.359
7.352
7.338
7.336
7.333
7.323
7.322
7.317
7.312

3.889
3.853
3.038
3.027
3.021
3.015
3.005
1.914
1.907
1.905
1.898
1.890
1.889
1.881



1c

¹H NMR (400 MHz, CDCl₃)



154.335

140.506

127.744

127.697

127.386

84.221

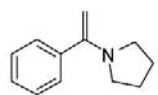
77.319

77.000

76.683

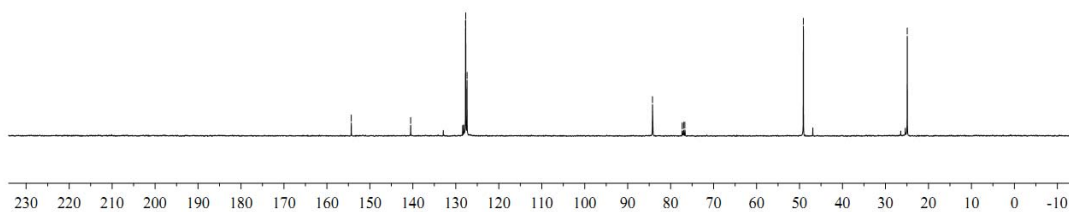
49.114

24.931

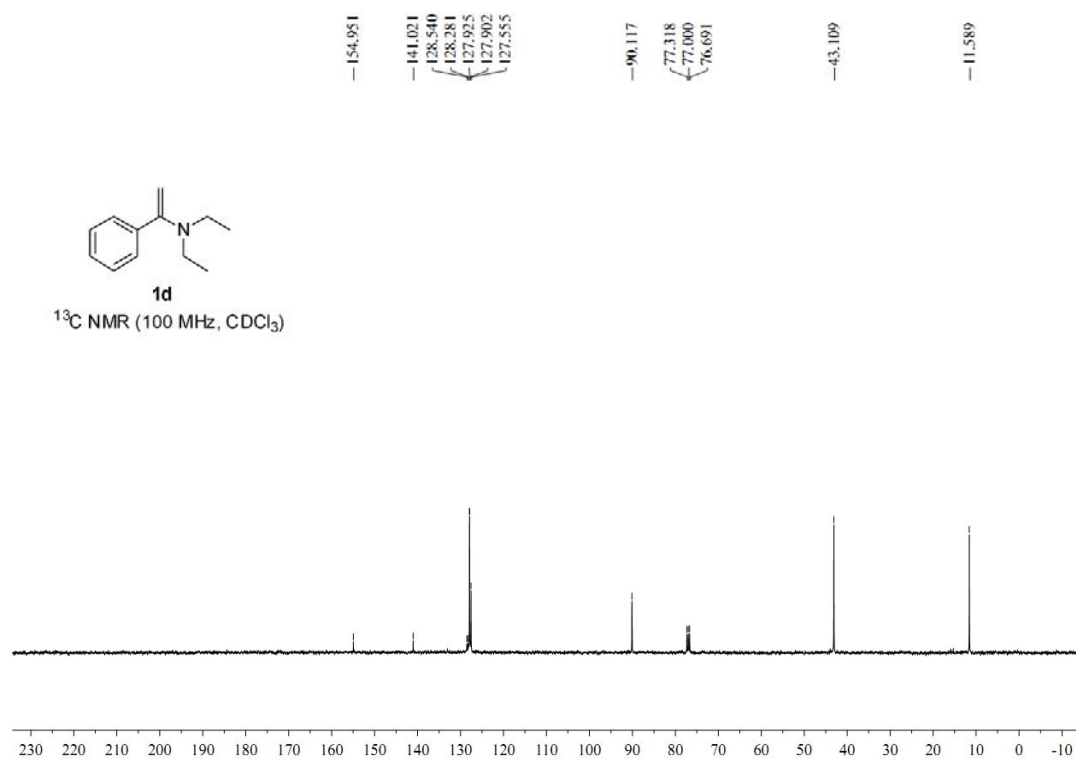
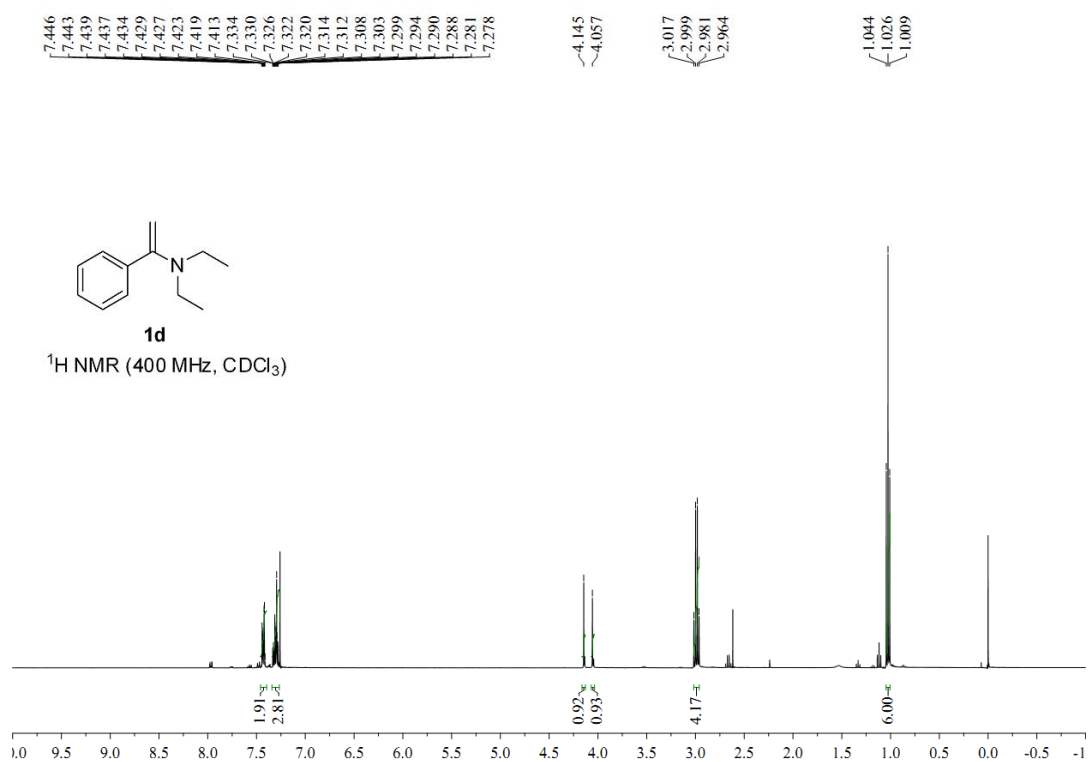


1c

¹³C NMR (100 MHz, CDCl₃)

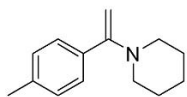


N,N-diethyl-1-phenylethanamine (1d)



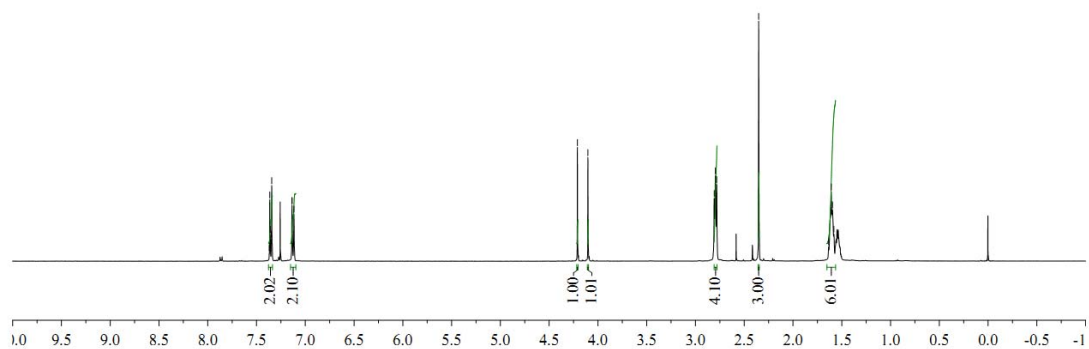
N-(1-(p-tolyl)vinyl)piperidine (1e)

7.369
7.364
7.360
7.349
7.344
7.137
7.136
7.116
4.211
4.103
2.808
2.796
2.782
2.352
1.635
1.608
1.596
1.581

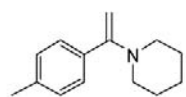


1e

¹H NMR (400 MHz, CDCl₃)

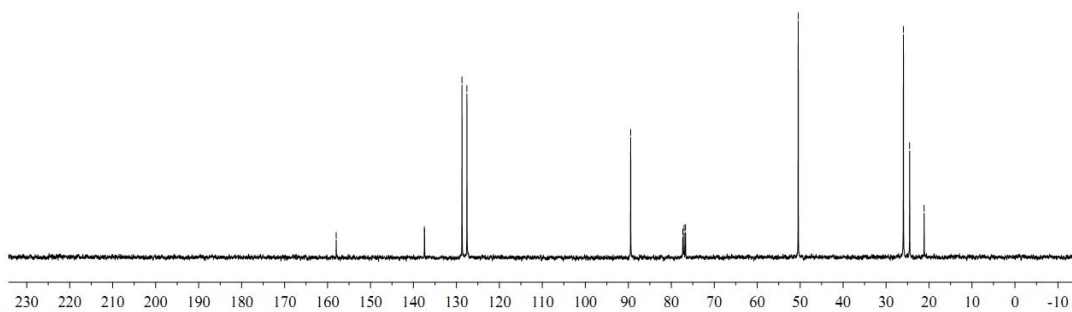


157.977
137.457
137.407
128.710
127.561
89.453
77.317
77.000
76.683
50.458
25.979
24.523
21.141



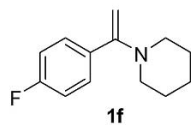
1e

¹³C NMR (100 MHz, CDCl₃)

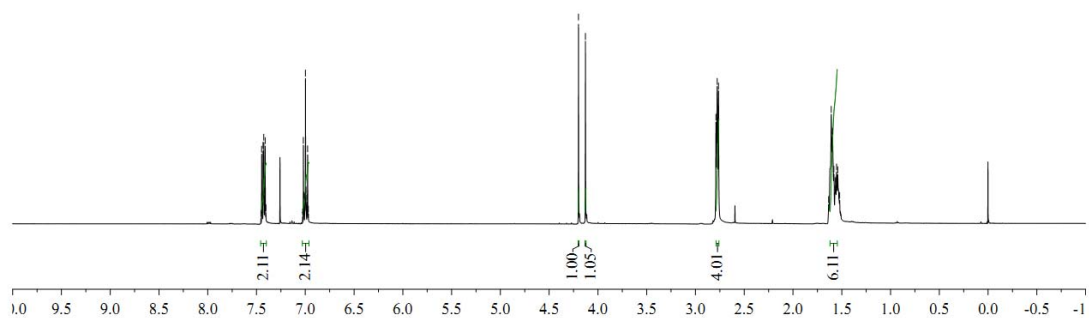


N-(1-(4-fluorophenyl)vinyl)piperidine (1f)

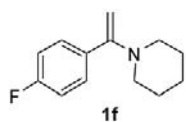
7.455, 7.447, 7.442, 7.433, 7.425, 7.417, 7.411, 7.404, 7.027, 7.020, 7.014, 7.003, 6.998, 6.993, 6.981, 6.976, 6.969, 4.199, 4.128, 2.790, 2.778, 2.764, 1.635, 1.608, 1.595, 1.581, 1.570, 1.565, 1.562, 1.553, 1.541, 1.522



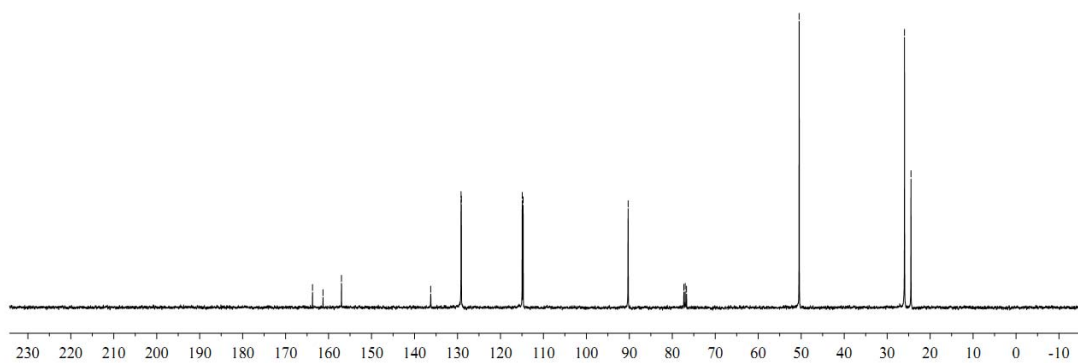
¹H NMR (400 MHz, CDCl₃)



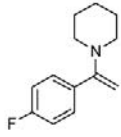
163.742, 161.297, 157.002, 136.233, 129.179, 129.101, 114.933, 114.721, 90.275, 77.316, 77.000, 76.684, 50.473, 25.959, 24.455



¹³C NMR (100 MHz, CDCl₃)

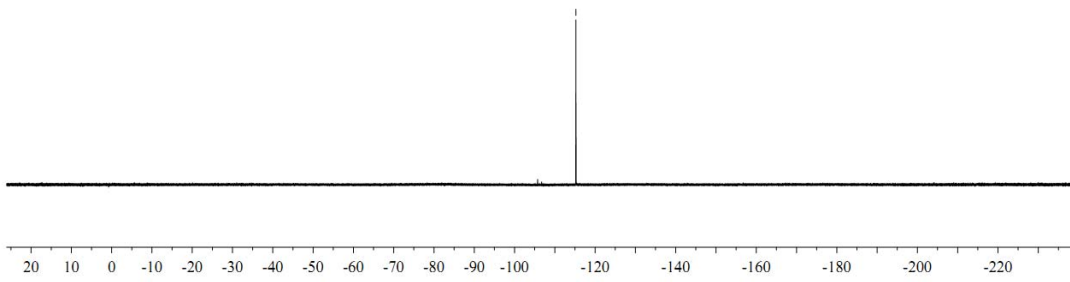


-115.228



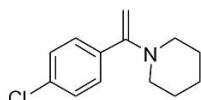
1f

^{19}F NMR (376 MHz, CDCl_3)



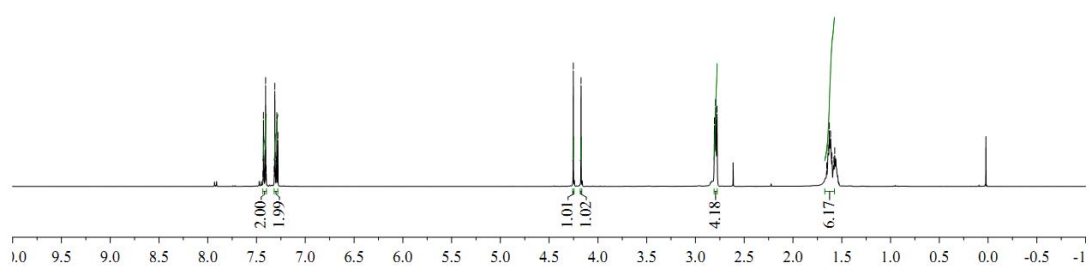
N-(1-(4-chlorophenyl)vinyl)piperidine (1g)

7.435, 7.429, 7.424, 7.412, 7.407, 7.401, 7.319, 7.313, 7.308, 7.296, 7.291, 7.285, 7.281, 4.252, 4.173, 2.806, 2.793, 2.779, 1.655, 1.641, 1.627, 1.614, 1.601, 1.581, 1.573

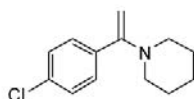


1g

¹H NMR (400 MHz, CDCl₃)

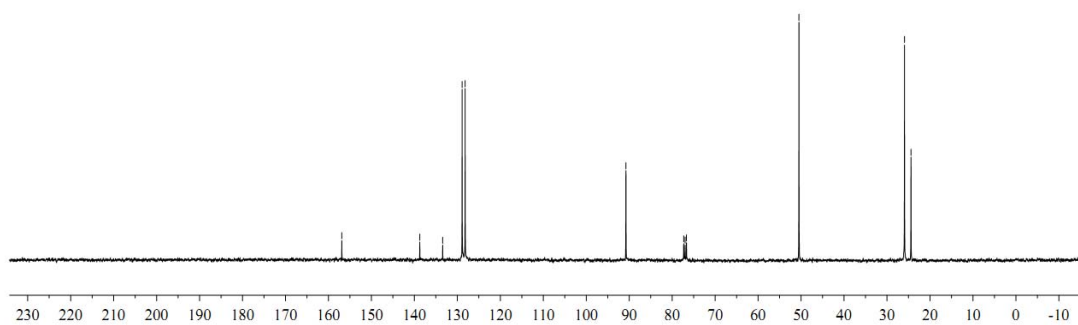


156.896, 138.750, 133.416, 128.897, 128.198, 90.767, 77.316, 77.000, 76.682, 50.496, 25.937, 24.418

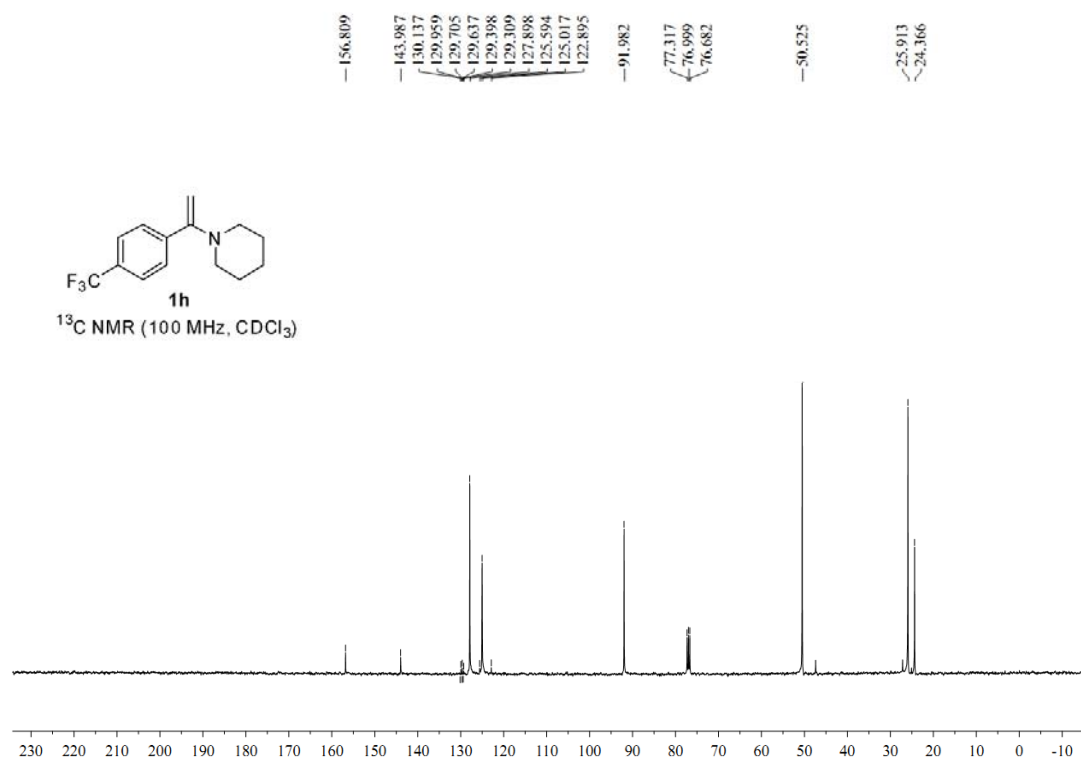
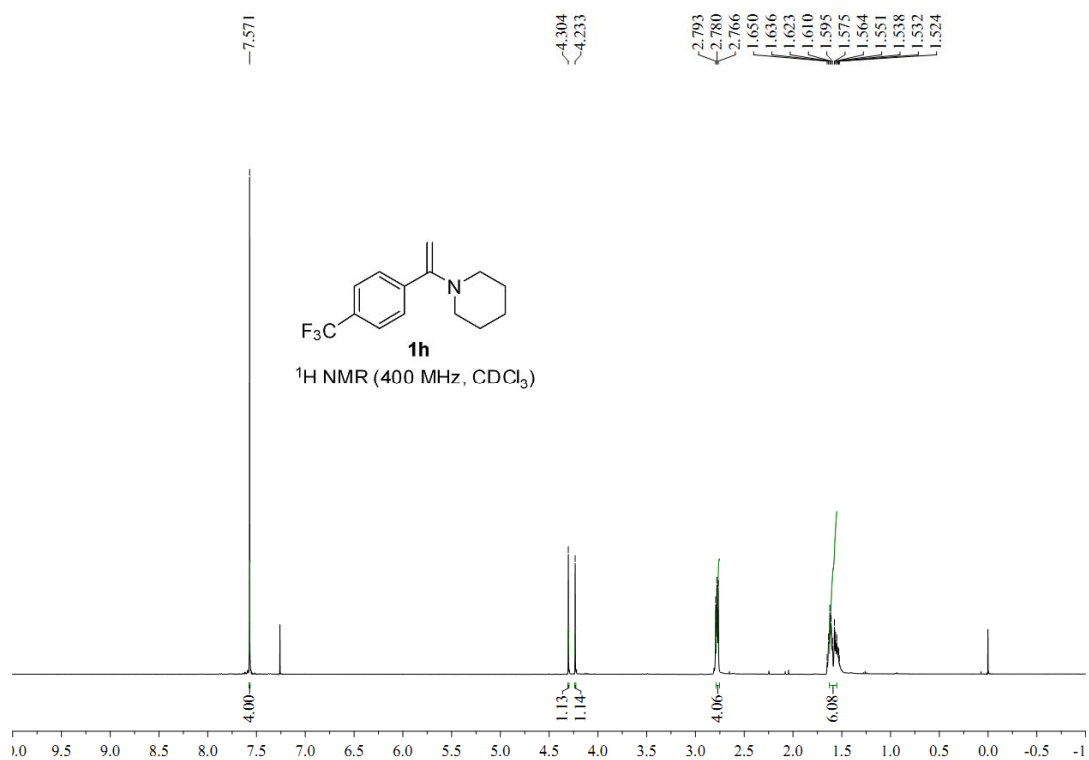


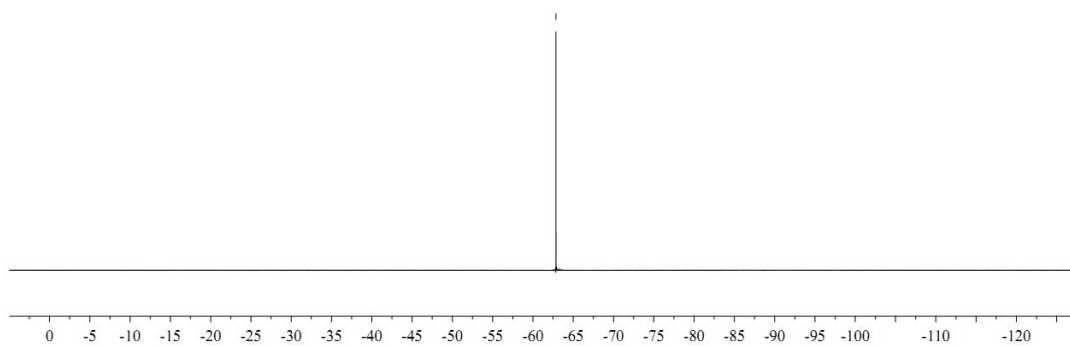
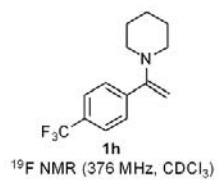
1g

¹³C NMR (100 MHz, CDCl₃)

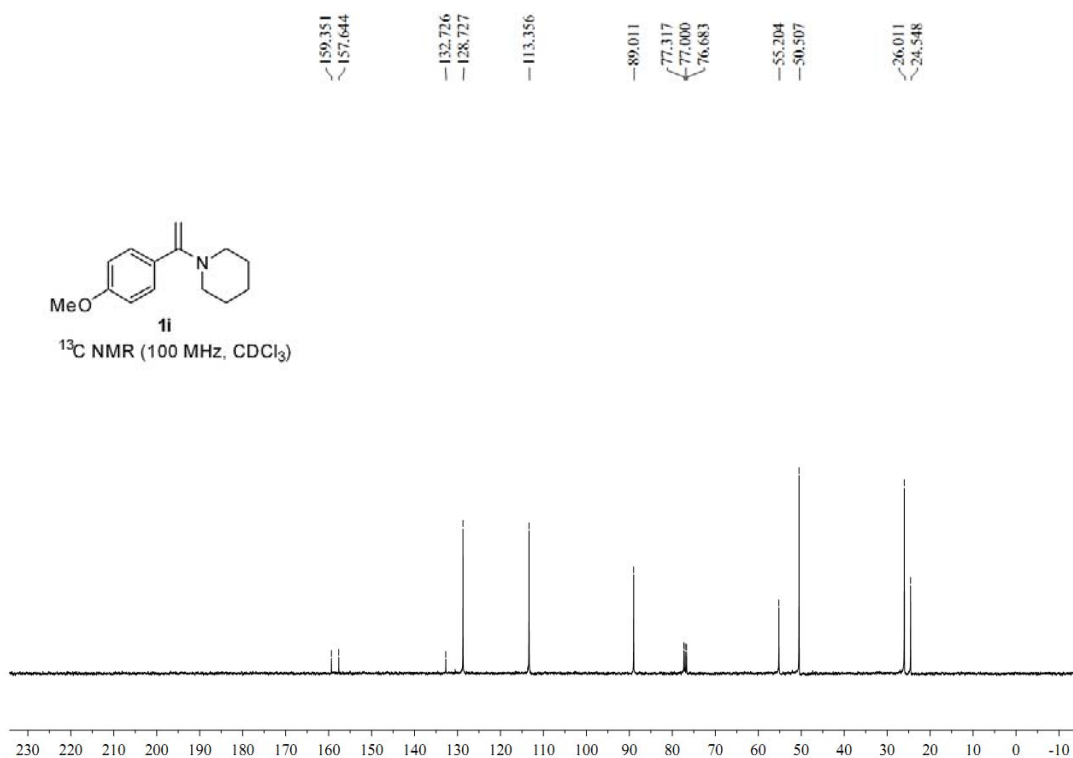
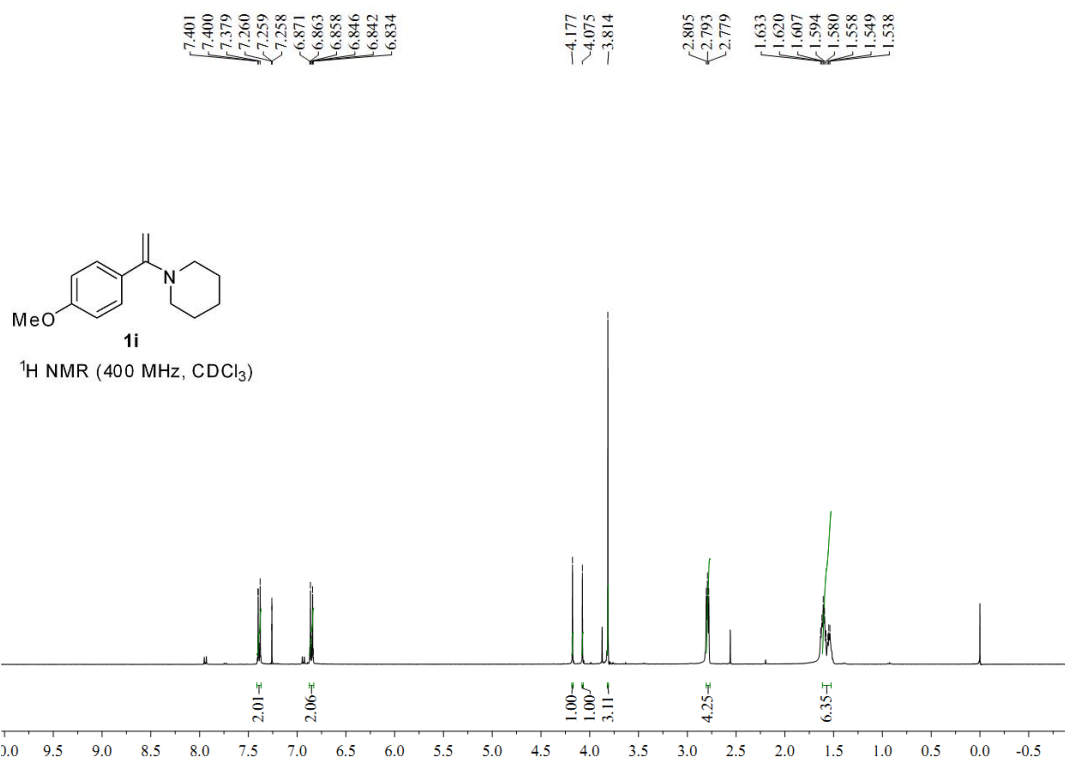


N-(1-(4-(trifluoromethyl)phenyl)vinyl)piperidine (1h)

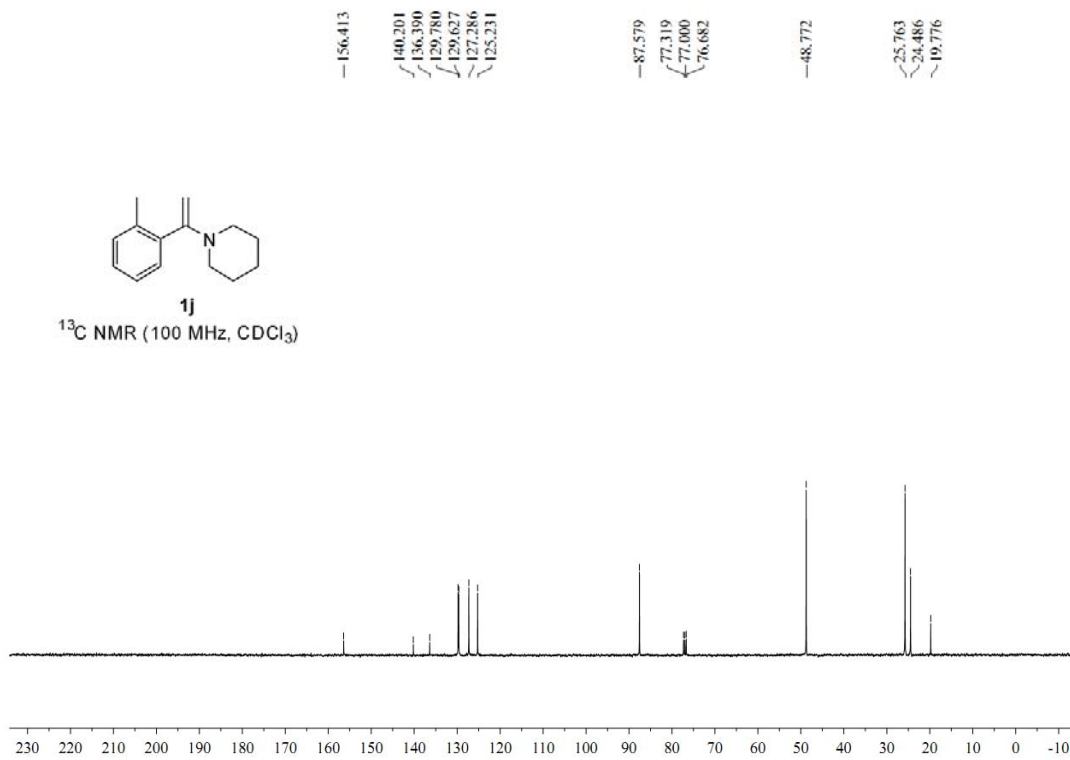
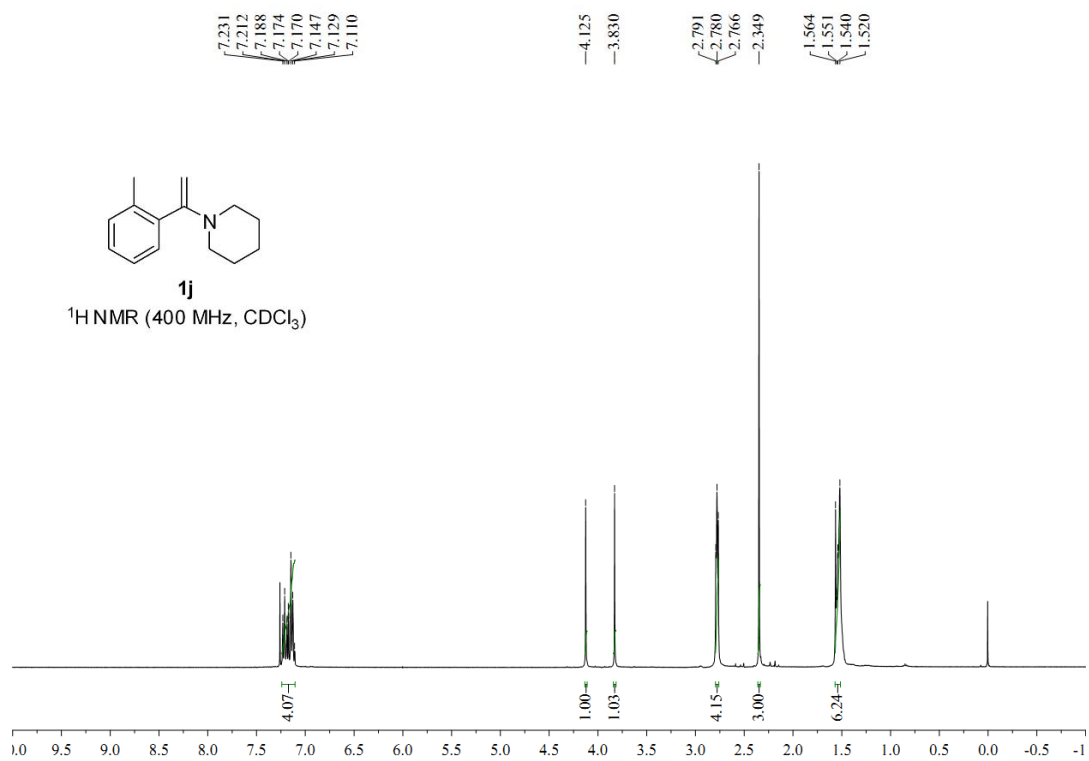




N-(1-(4-methoxyphenyl)vinyl)piperidine (1i)

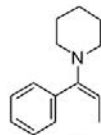


N-(1-(o-tolyl)vinyl)piperidine (1j)



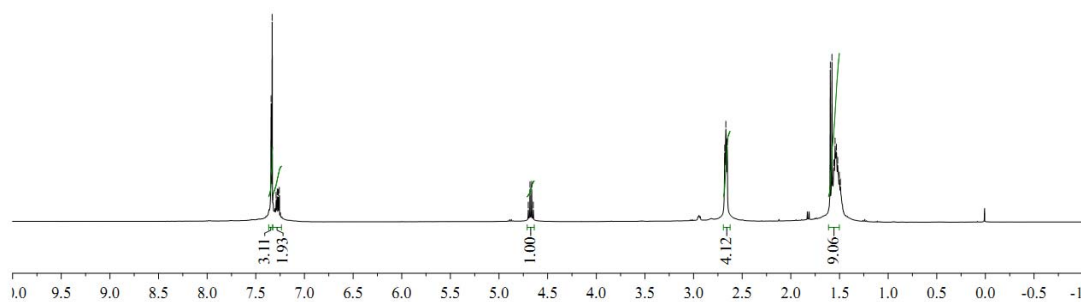
N-(1-phenylprop-1-en-1-yl)piperidine (1k)

7.343, 7.338, 7.332, 7.325, 7.291, 7.280, 7.269, 7.259, 4.699, 4.682, 4.664, 4.647, 2.680, 2.668, 2.655, 1.594, 1.577, 1.572, 1.557, 1.545, 1.532, 1.519, 1.502, 1.491

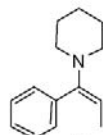


1k

¹H NMR (400 MHz, CDCl₃)

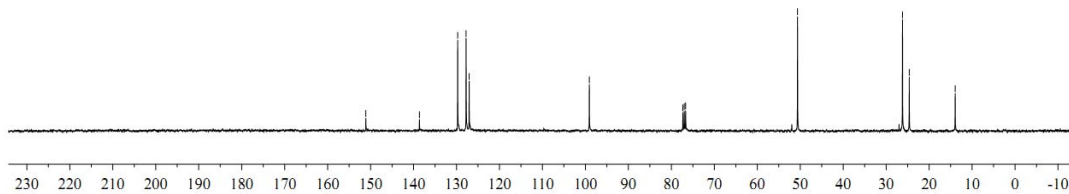


151.114, 138.629, 129.733, 127.778, 127.060, 99.089, 77.319, 77.000, 76.684, 50.641, 26.207, 24.633, 13.941



1k

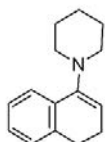
¹³C NMR (100 MHz, CDCl₃)



N-(3,4-dihydronaphthalen-1-yl)piperidine (11)

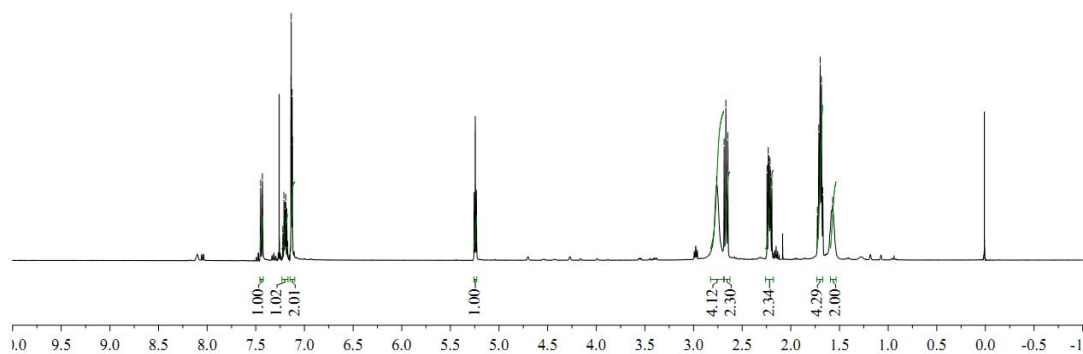
7.450
7.432
7.222
7.214
7.211
7.200
7.196
7.193
7.181
7.172
7.139
7.136
7.128
7.127
7.124

2.763
2.686
2.668
2.648
2.233
2.225
1.727
1.712
1.698
1.685
1.672
1.570



11

¹H NMR (400 MHz, CDCl₃)



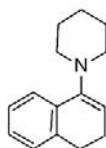
148.641
138.034
132.850
127.374
126.461
125.987
123.430

106.745

77.318
77.000
76.682

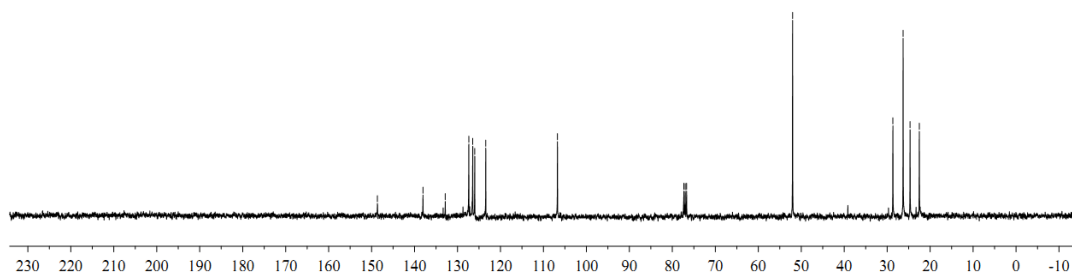
52.021

28.669
26.294
24.672
22.517



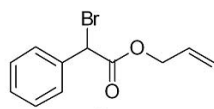
11

¹³C NMR (100 MHz, CDCl₃)



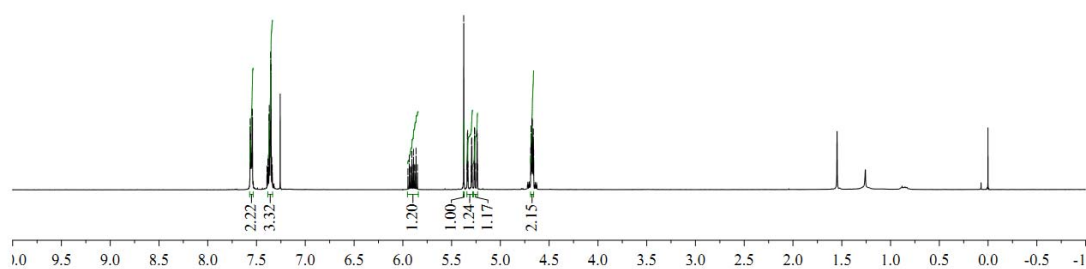
allyl 2-bromo-2-phenylacetate (**2r**)

7.566
7.560
7.555
7.551
7.546
7.542
7.383
7.379
7.372
7.370
7.366
7.361
7.357
7.352
7.343
7.338
5.934
5.908
5.905
5.891
5.865
5.376
5.266
4.689
4.686
4.682
4.680
4.676
4.672
4.668
4.666
4.662
4.659



2r

¹H NMR (400 MHz, CDCl₃)



167.844

135.568

131.008

129.200

128.715

128.574

118.896

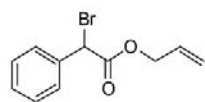
77.318

77.000

76.682

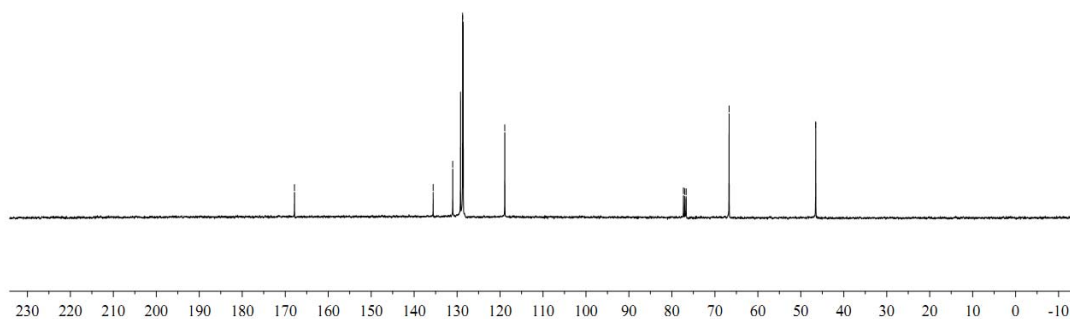
66.690

46.529



2r

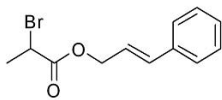
¹³C NMR (100 MHz, CDCl₃)



cinnamyl 2-bromopropanoate (2q)

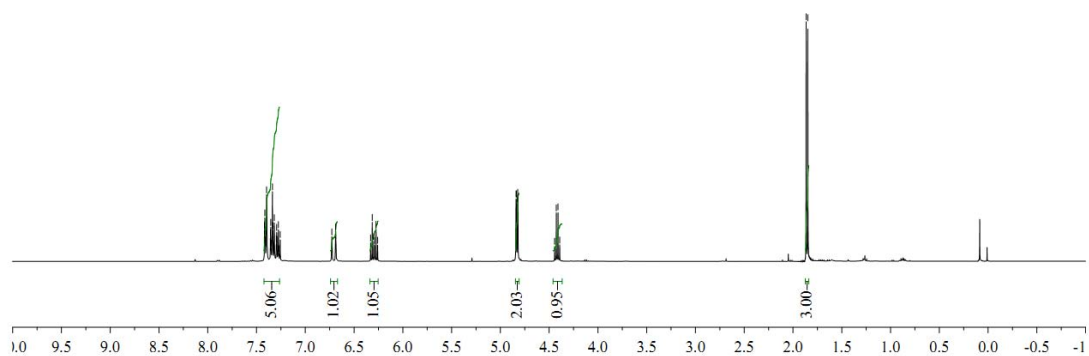
7.419
7.415
7.410
7.397
7.358
7.355
7.350
7.337
7.333
7.318
7.297
7.294
7.290
7.282
7.276
7.258
7.258
6.688
6.330
6.314
6.298
6.291
6.275
6.258
4.839
4.837
4.824
4.823
4.444
4.427
4.409
4.392

1.866
1.848



2q

¹H NMR (400 MHz, CDCl₃)



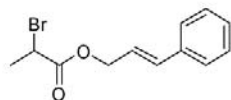
169.853

135.890
134.759
128.519
128.121
126.571
122.069

77.318
77.000
76.682
66.294

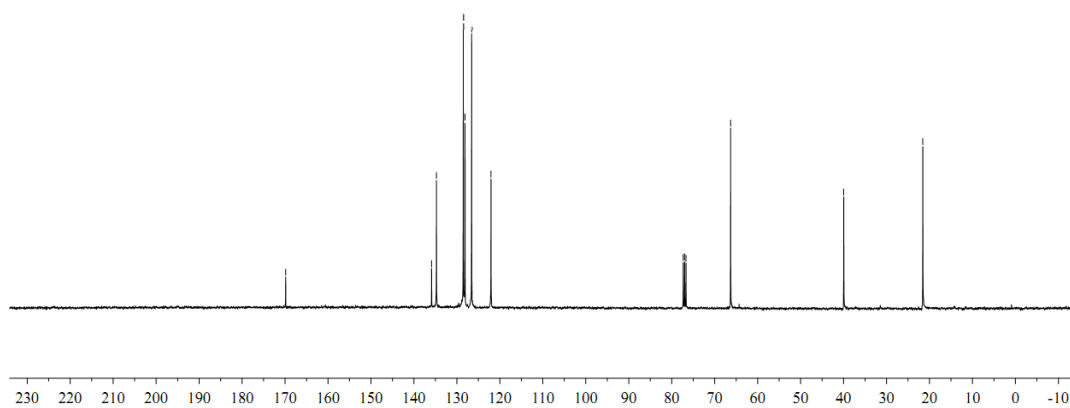
39.965

21.564

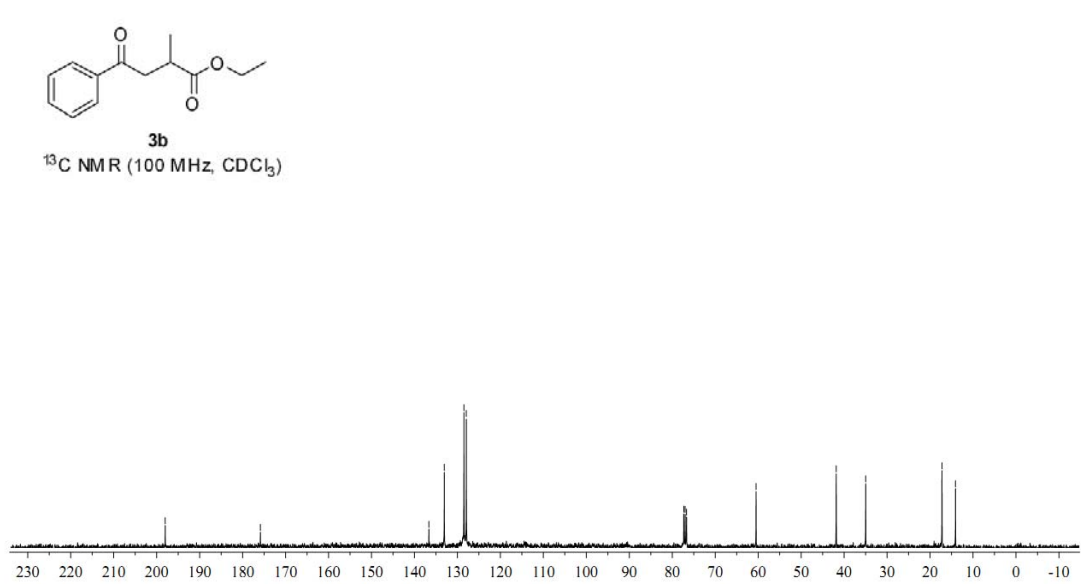
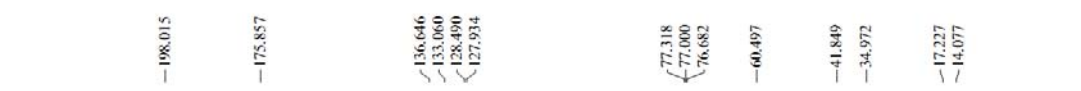
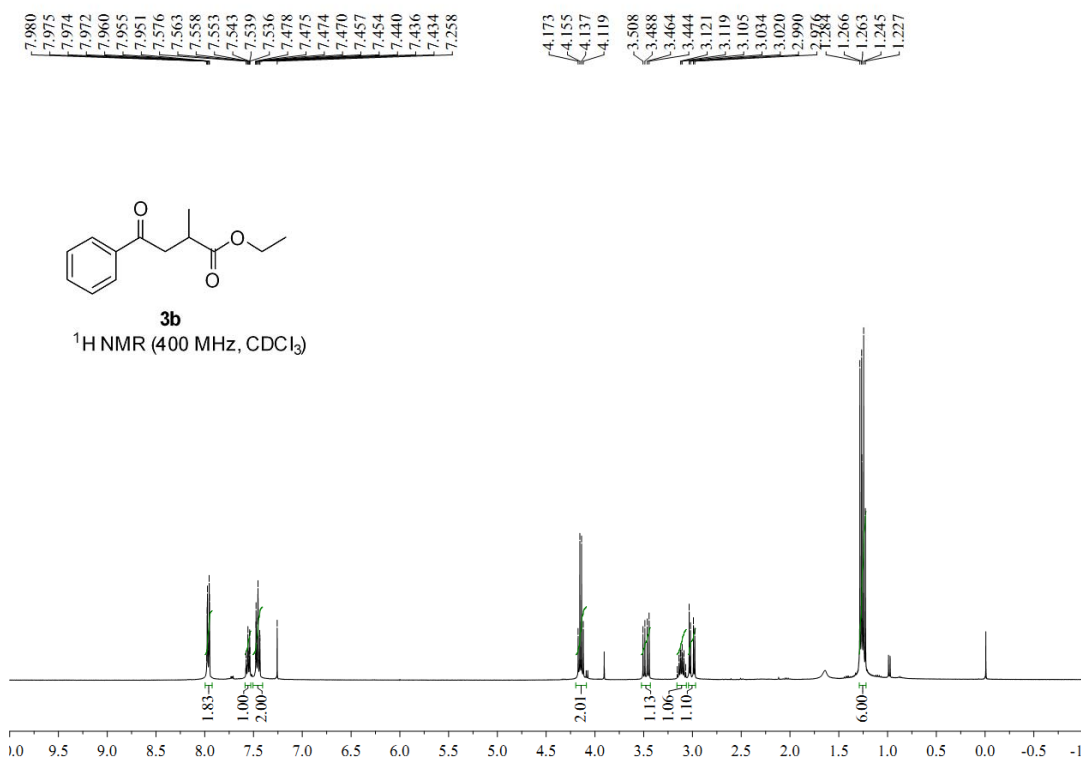


2q

¹³C NMR (100 MHz, CDCl₃)



ethyl 2-methyl-4-oxo-4-phenylbutanoate (3b)



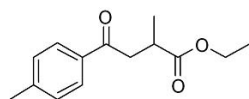
ethyl 2-methyl-4-oxo-4-(p-tolyl)butanoate (3e)

7.872
7.868
7.856
7.851
7.258
7.239

4.171
4.153
4.135
4.117

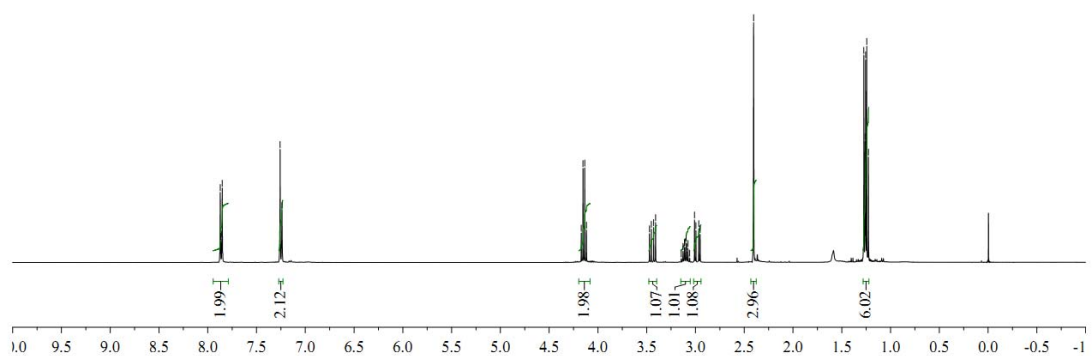
3.452
3.428
3.409
3.010
2.996
2.969

1.274
1.262
1.256
1.244
1.226



3e

¹H NMR (400 MHz, CDCl₃)



197.440

175.739

143.666

134.119

129.028

127.927

77.315

77.000

76.646

60.289

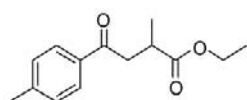
41.604

34.885

21.376

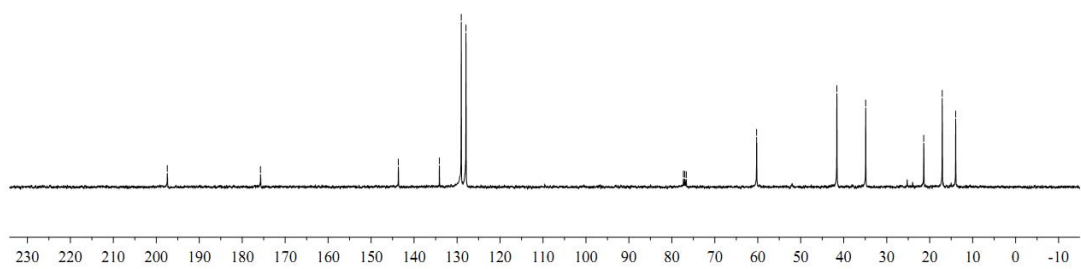
17.083

13.950

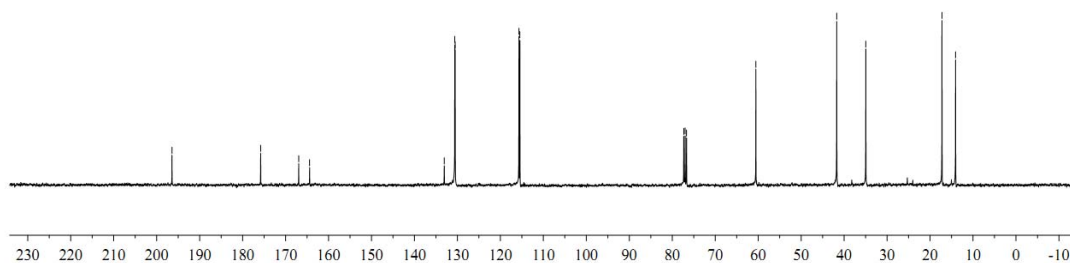
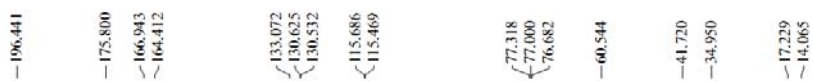
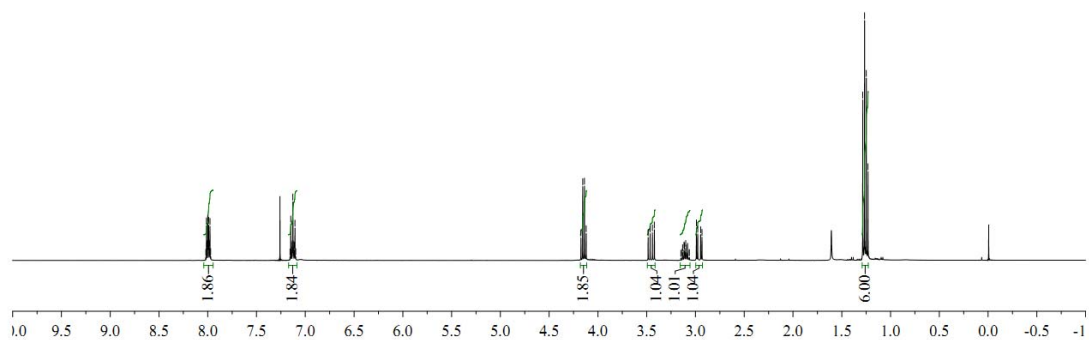
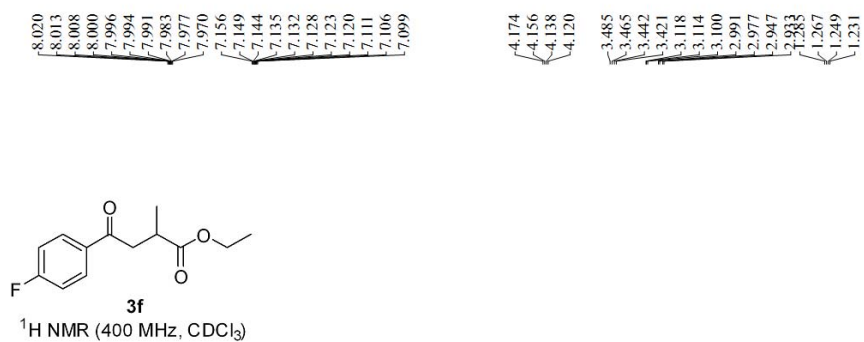


3e

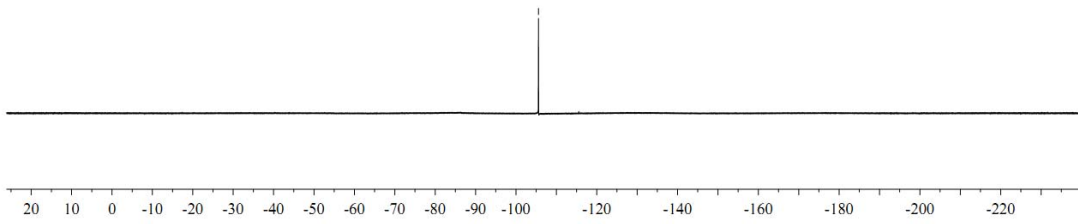
¹³C NMR (100 MHz, CDCl₃)



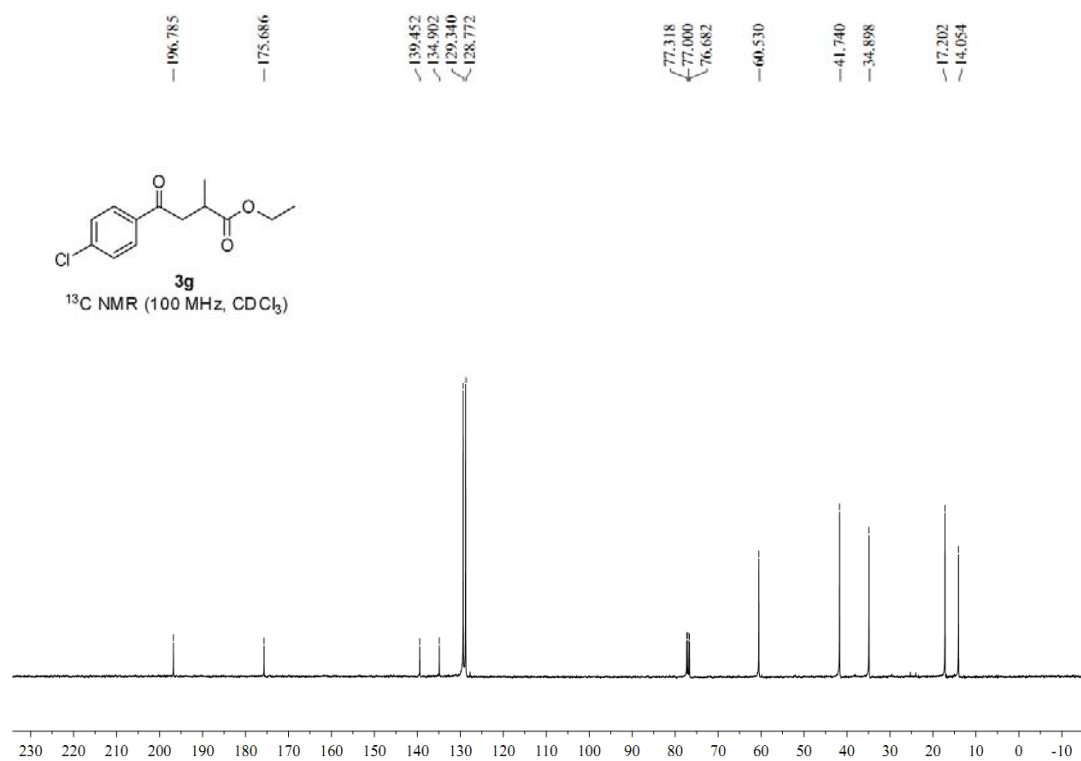
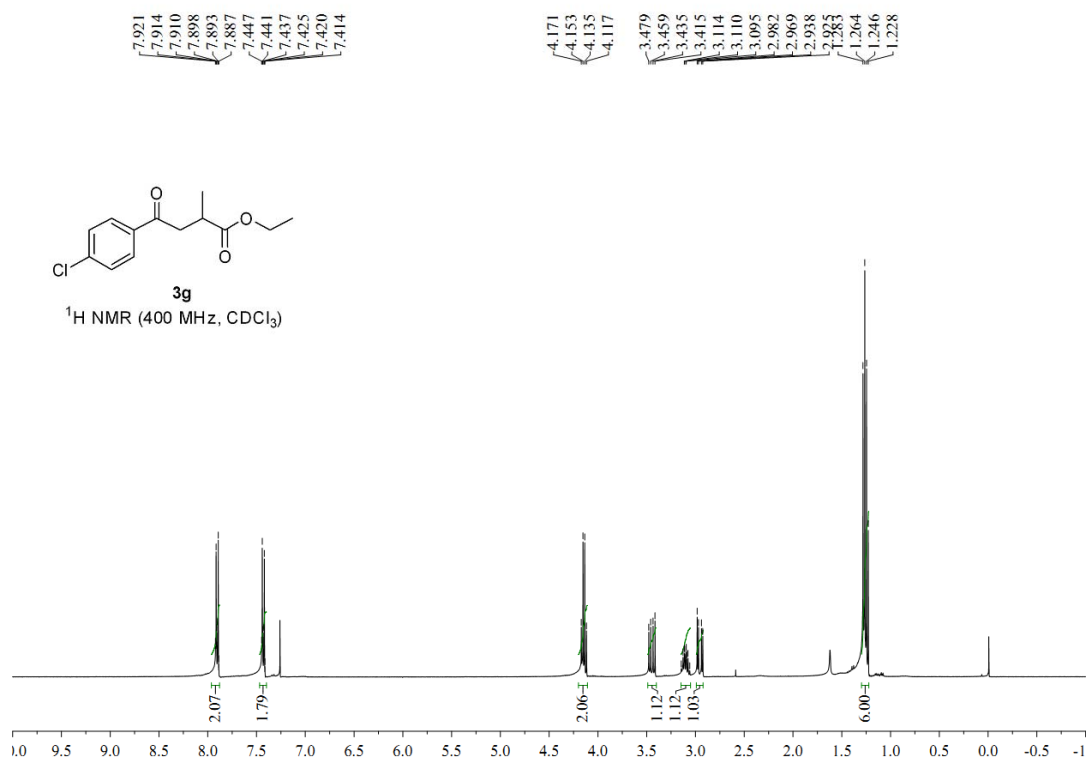
ethyl 4-(4-fluorophenyl)-2-methyl-4-oxobutanoate (3f)



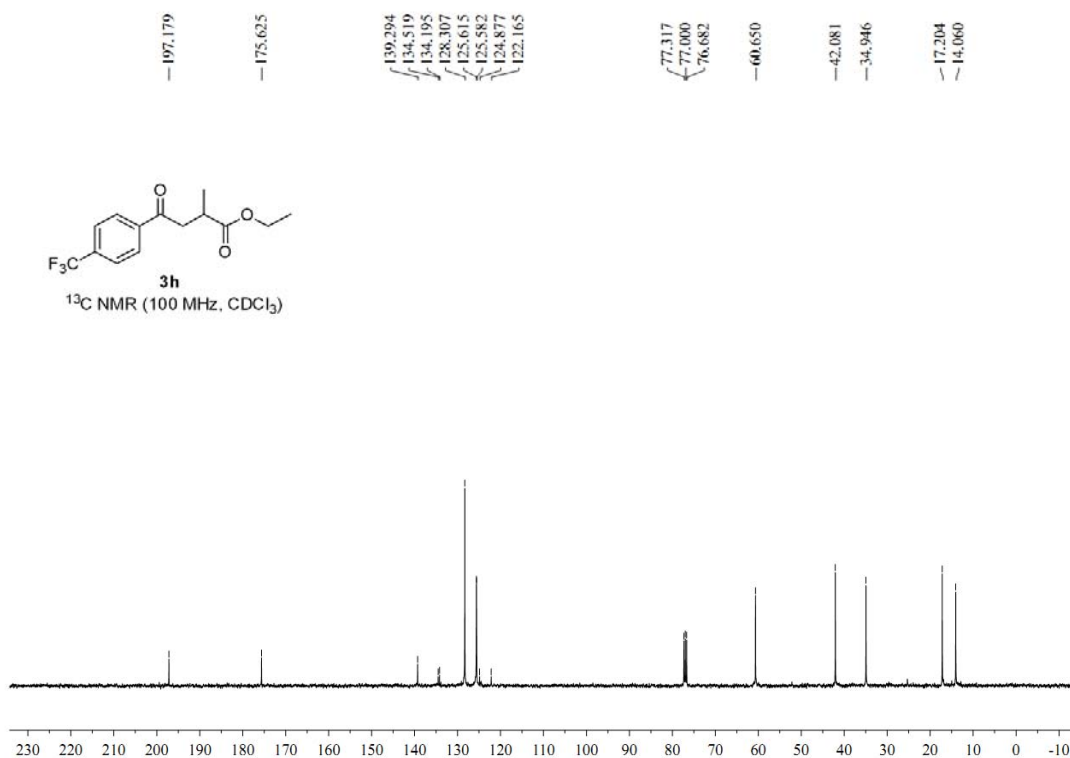
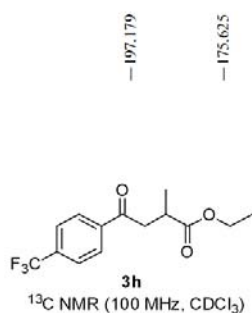
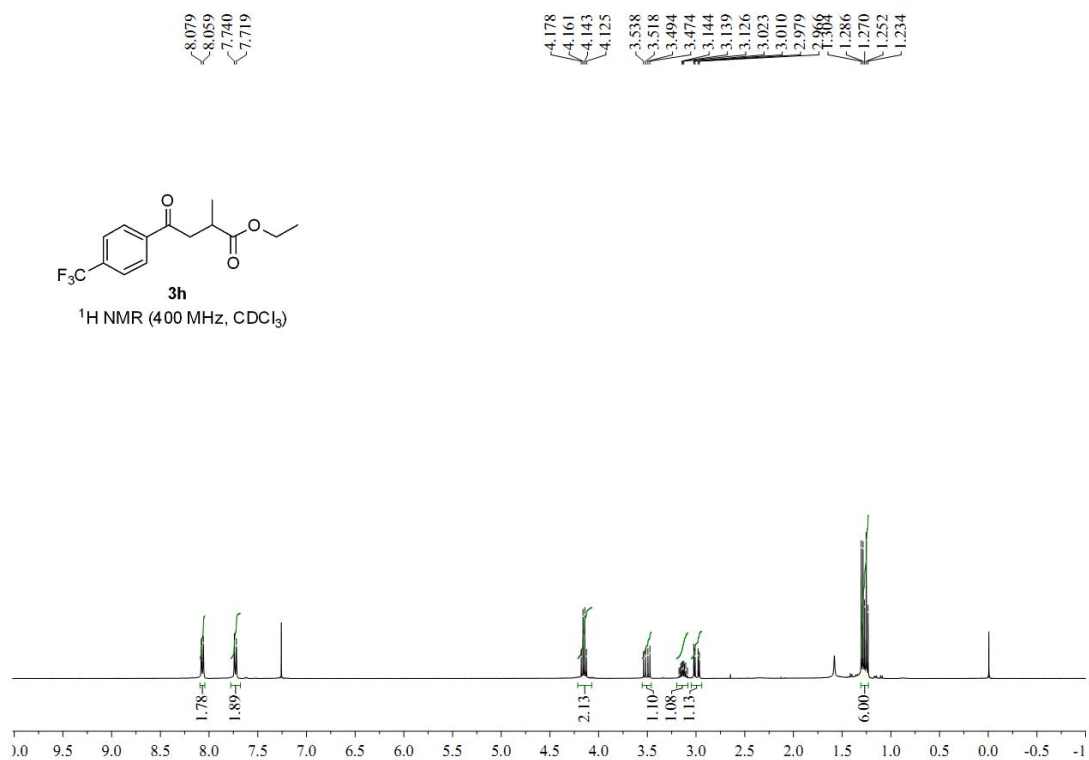
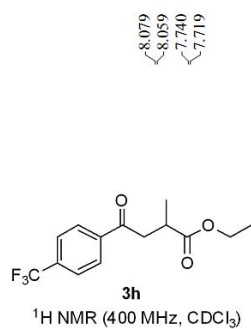
--105.571



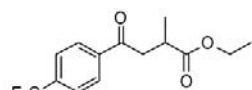
ethyl 4-(4-chlorophenyl)-2-methyl-4-oxobutanoate (**3g**)



ethyl 2-methyl-4-oxo-4-(4-(trifluoromethyl)phenyl)butanoate (3h)

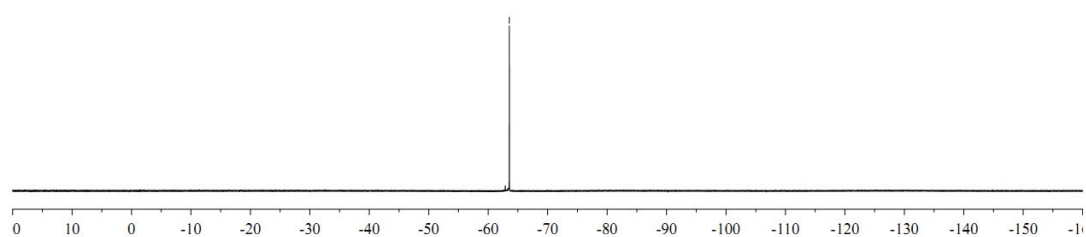


-63.550

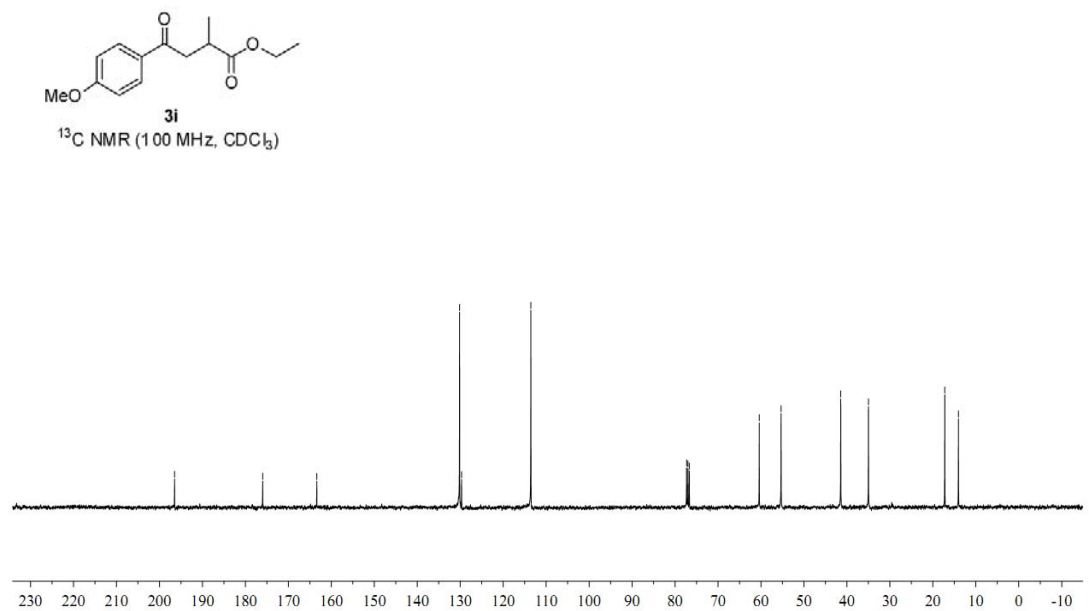
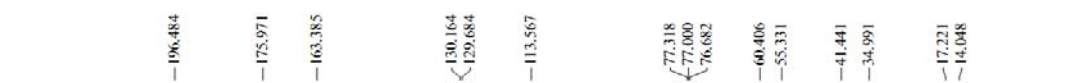
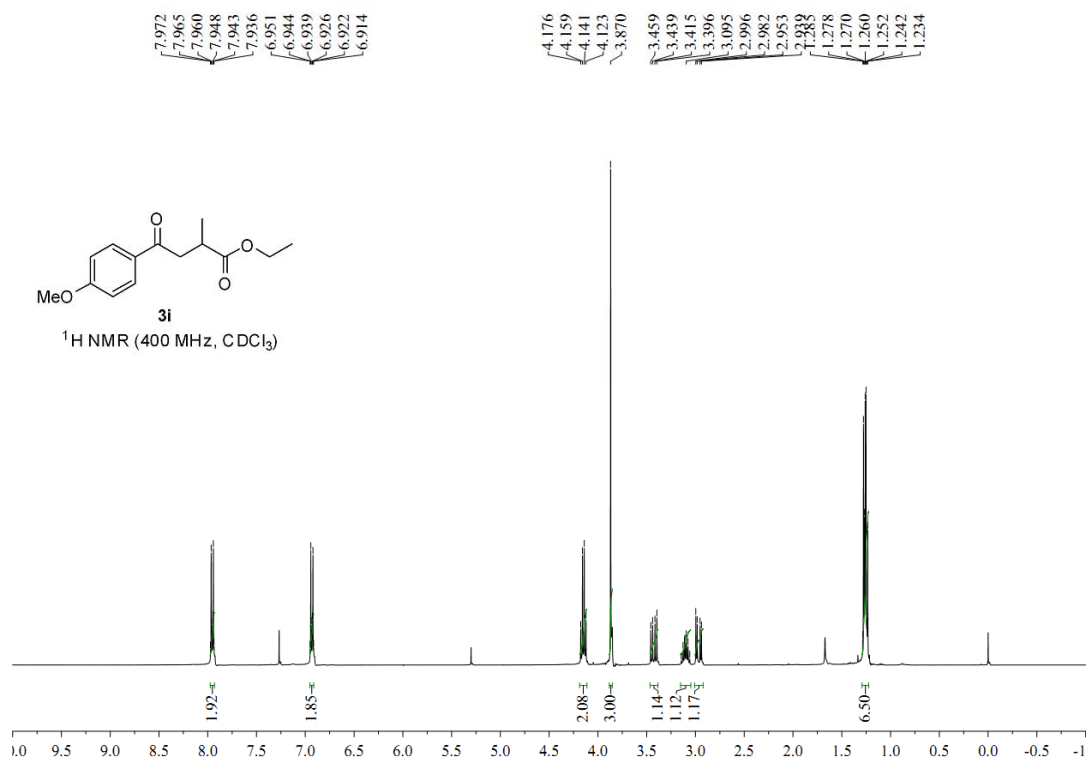


3h

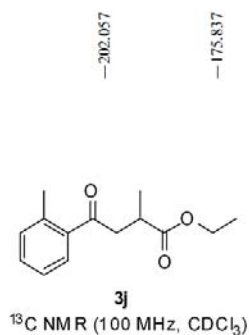
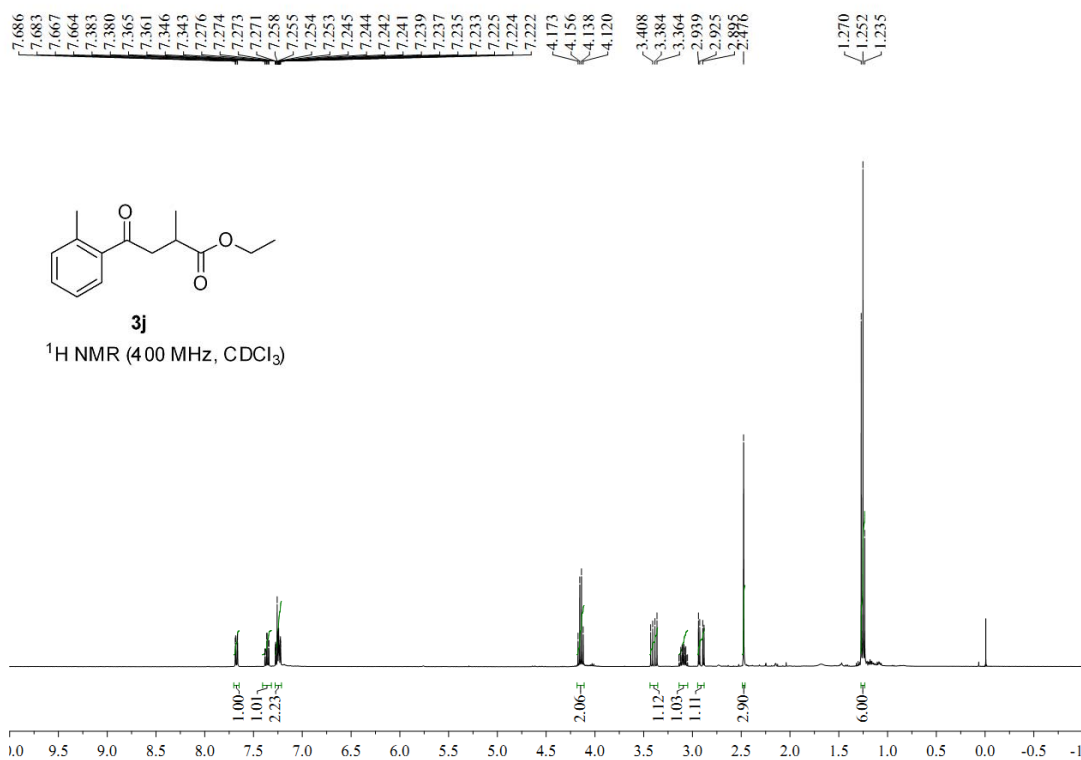
¹⁹F NMR (376 MHz, CDCl₃)



ethyl 4-(4-methoxyphenyl)-2-methyl-4-oxobutanoate (3i)

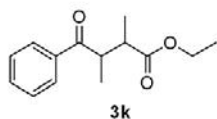


ethyl 2-methyl-4-oxo-4-(o-tolyl)butanoate (**3j**)

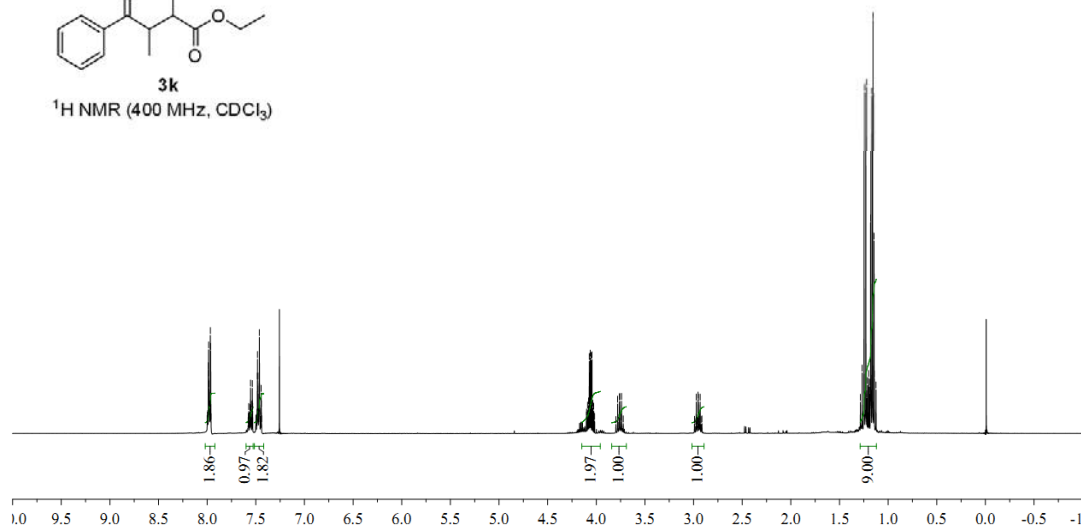


ethyl 2,3-dimethyl-4-oxo-4-phenylbutanoate (3k)

7.988
7.987
7.985
7.981
7.976
7.973
7.968
7.964
7.872
7.559
7.554
7.549
7.539
7.535
7.532
7.487
7.484
7.483
7.479
7.471
7.467
7.463
7.449
7.445
7.443
4.099
4.090
4.080
4.072
4.062
4.054
4.044
4.036
4.027
3.781
3.763
3.759
3.741
2.972
2.954
2.951
2.932
1.286
1.268
1.248
1.230
1.219
1.212
1.201
1.192
1.183
1.180
1.177
1.163
1.159
1.145
1.142
1.125



¹H NMR (400 MHz, CDCl₃)



-203.437

-175.716

136.013

132.884

128.583

128.329

77.318

77.000

76.682

-60.522

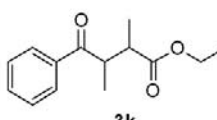
43.017

41.725

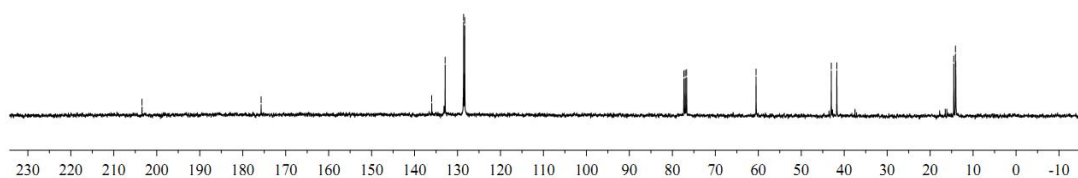
14.500

14.112

14.033

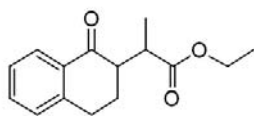


¹³C NMR (100 MHz, CDCl₃)



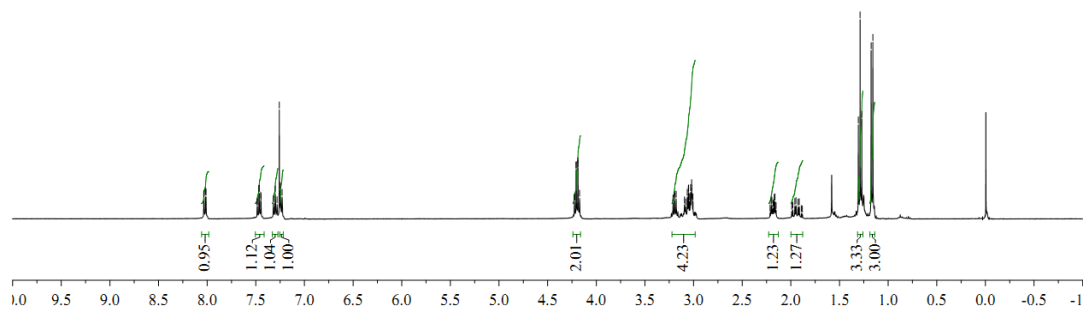
ethyl 2-(1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl)propanoate (31)

8.035, 8.032, 8.015, 8.012, 7.487, 7.484, 7.469, 7.465, 7.450, 7.446, 7.319, 7.300, 7.281, 7.259, 7.249, 7.230, 4.226, 4.224, 4.208, 4.206, 4.190, 4.188, 4.172, 3.054, 3.052, 3.031, 3.020, 3.017, 2.203, 2.195, 2.170, 2.163, 1.988, 1.986, 1.289, 1.271, 1.175, 1.157

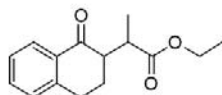


31

¹H NMR (400 MHz, CDCl₃)

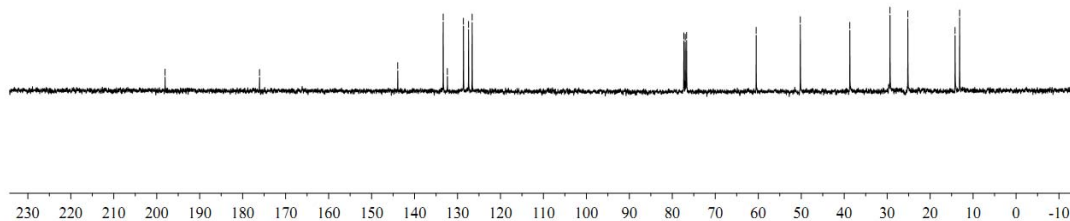


196.060, 176.072, 143.911, 133.332, 132.350, 128.617, 127.433, 126.589, 77.318, 77.000, 76.683, 60.483, 50.215, 38.692, 29.346, 25.192, 14.194, 13.140

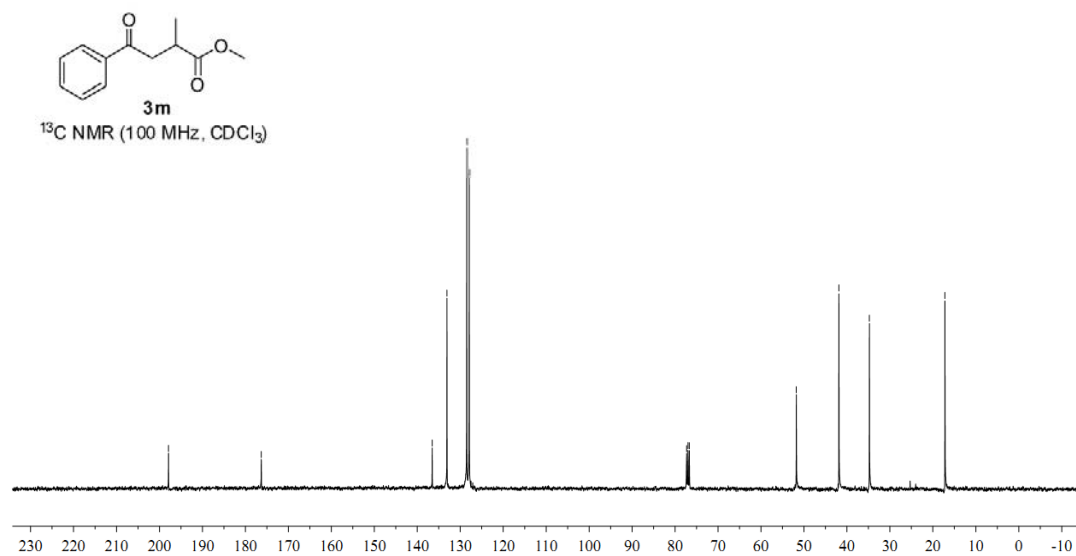
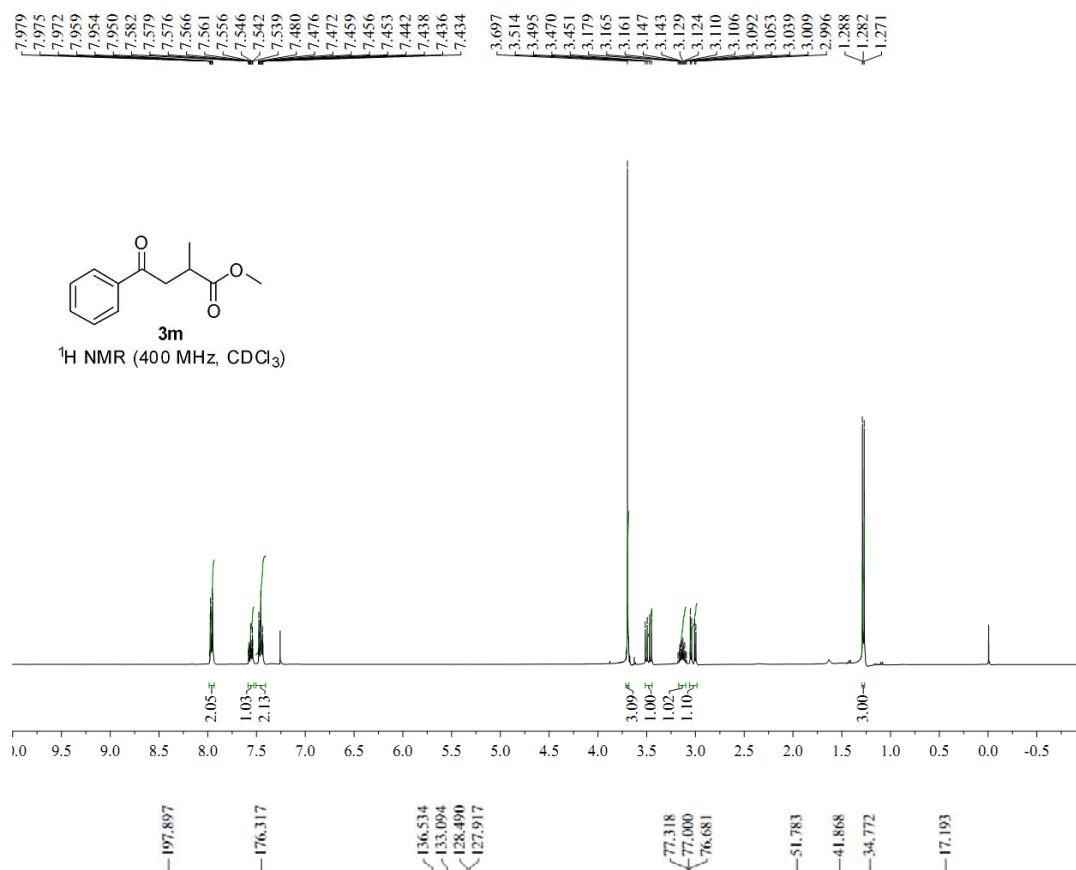


31

¹³C NMR (100 MHz, CDCl₃)

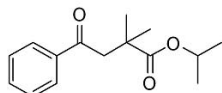


methyl 2-methyl-4-oxo-4-phenylbutanoate (3m)



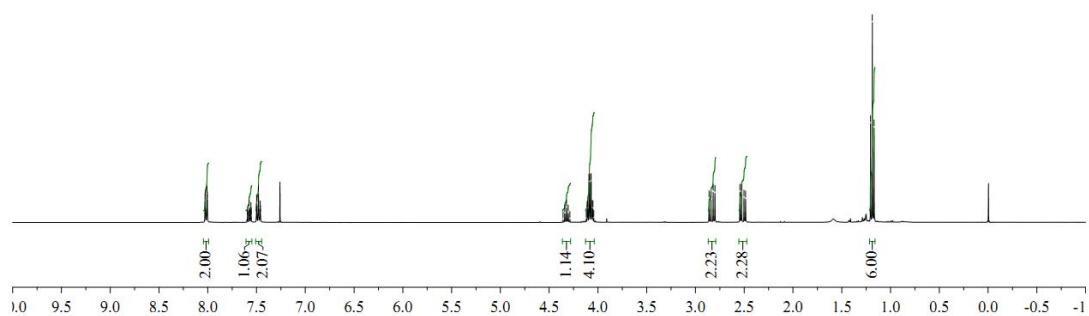
isopropyl 2,2-dimethyl-4-oxo-4-phenylbutanoate (3n)

8.027
8.025
8.008
8.004
7.576
7.561
7.558
7.554
7.499
7.496
7.483
7.480
7.462
7.460
4.340
4.322
4.305
4.110
4.105
4.101
4.092
4.087
4.074
4.070
4.056
4.052
4.043
2.842
2.819
2.800
2.544
2.527
2.503
2.486
1.205
1.187
1.169

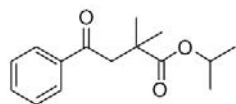


3n

¹H NMR (400 MHz, CDCl₃)

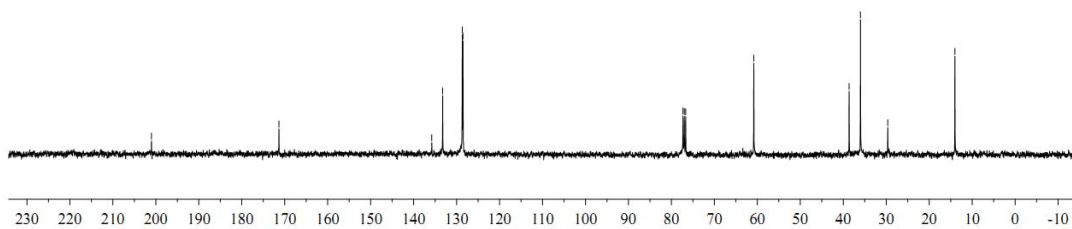


201.022
171.349
135.778
133.240
128.679
128.512
77.318
77.000
76.681
60.829
38.645
36.005
29.646
14.023

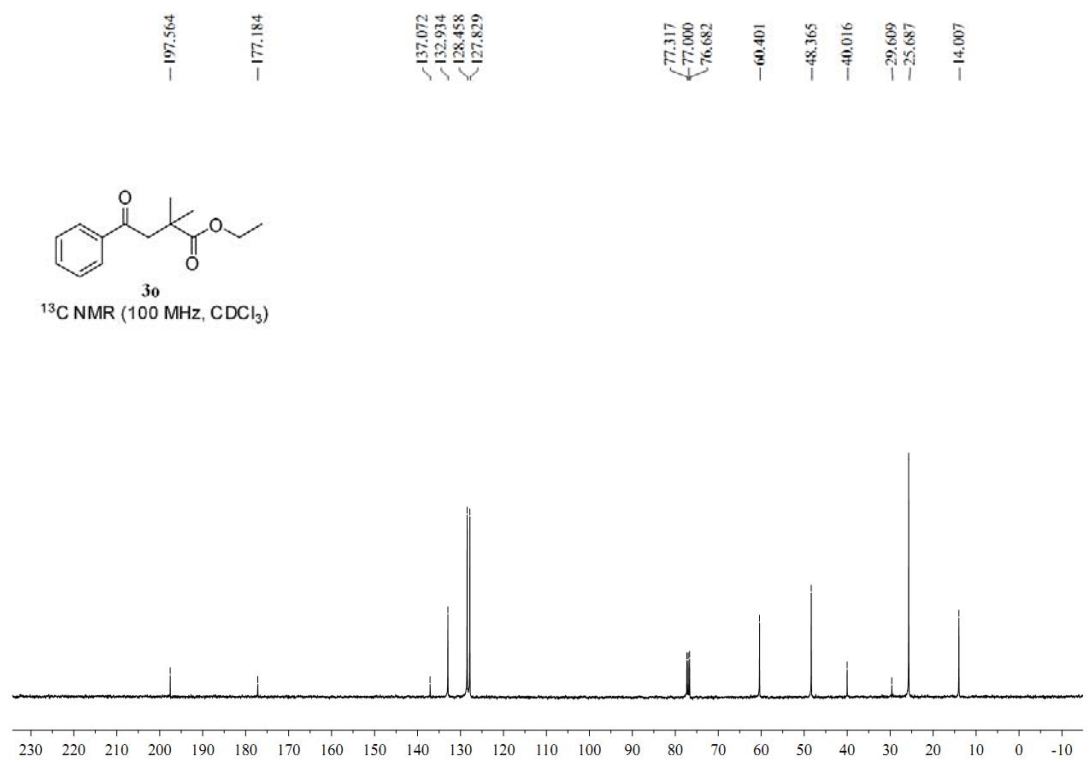
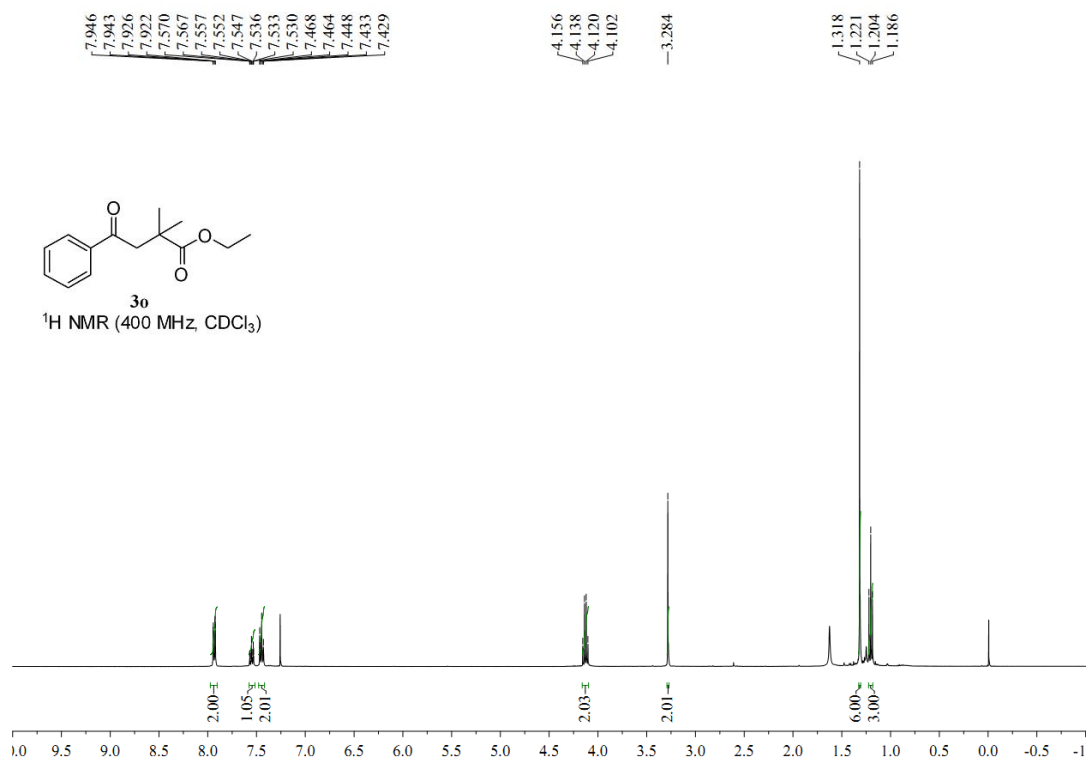


3n

¹³C NMR (100 MHz, CDCl₃)

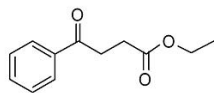


ethyl 2,2-dimethyl-4-oxo-4-phenylbutanoate (**3o**)



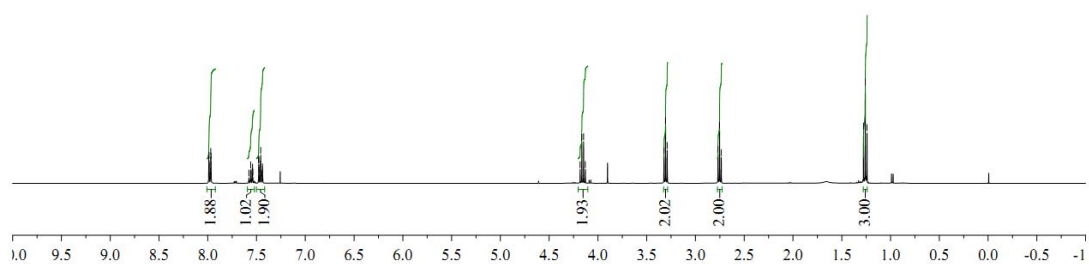
ethyl 4-oxo-4-phenylbutanoate (3p)

7.990, 7.987, 7.985, 7.975, 7.970, 7.966, 7.578, 7.560, 7.555, 7.544, 7.541, 7.538, 7.478, 7.477, 7.473, 7.460, 7.457, 7.454, 7.443, 7.439, 7.437, 4.183, 4.165, 4.147, 4.129, 3.323, 3.307, 3.290, 2.769, 2.753, 2.736, 1.277, 1.259, 1.241

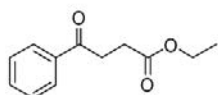


3p

¹H NMR (400 MHz, CDCl₃)

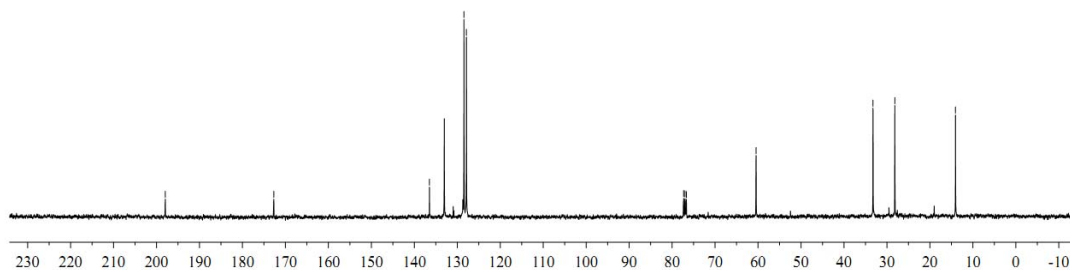


197.959, 172.697, 136.480, 128.451, 127.870, 77.317, 77.000, 76.685, 60.455, 33.247, 28.165, 14.045



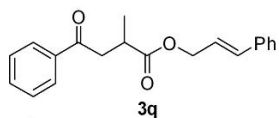
3p

¹³C NMR (100 MHz, CDCl₃)

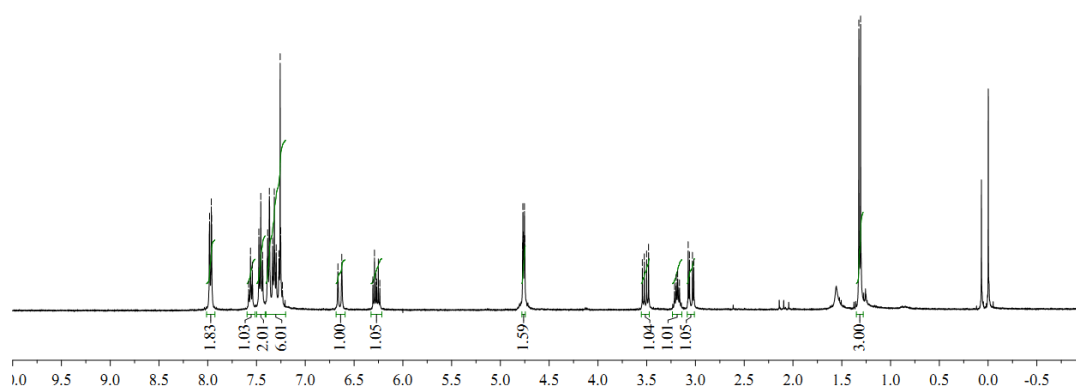


cinnamyl 2-methyl-4-oxo-4-phenylbutanoate (3q)

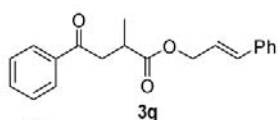
7.982
7.964
7.960
7.456
7.369
7.318
7.288
7.268
6.626
6.307
6.291
6.275
6.268
6.252
6.236
4.773
4.769
4.765
4.757
4.753
4.750
3.545
3.525
3.501
3.481
3.215
3.196
3.182
3.164
3.075
3.061
3.031
3.017
1.325
1.307



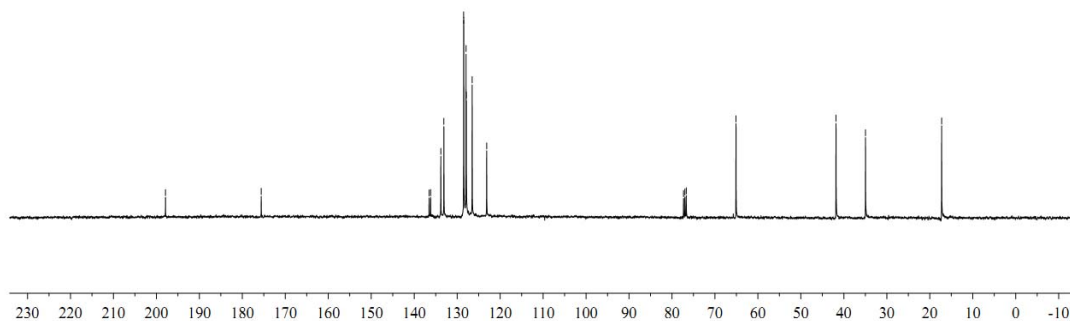
¹H NMR (400 MHz, CDCl₃)



197.879
175.603
136.531
136.162
133.804
133.093
128.487
128.464
127.935
127.878
126.510
123.138
77.318
77.000
76.682
65.110
41.831
34.966
17.245

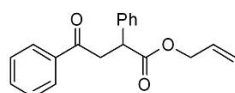


¹³C NMR (100 MHz, CDCl₃)



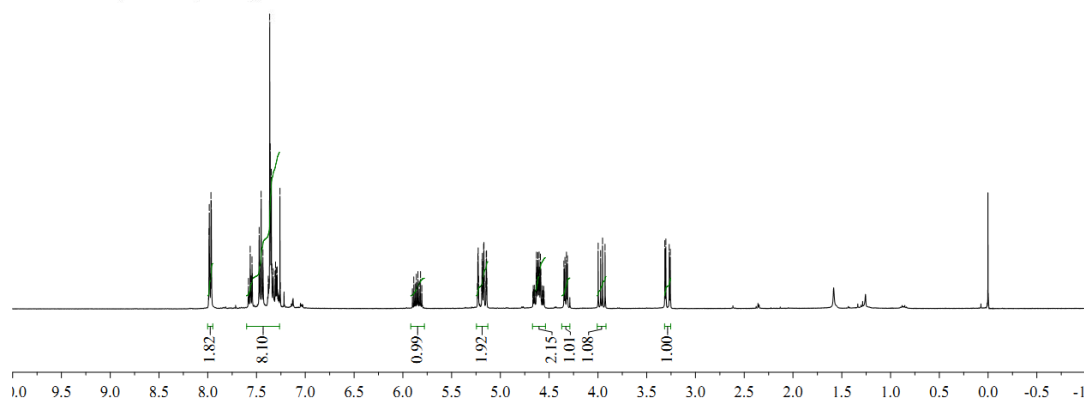
allyl 4-oxo-2,4-diphenylbutanoate (3r)

7.987
7.984
7.972
7.967
7.963
7.546
7.543
7.473
7.469
7.453
7.435
7.365
7.359
7.349
7.343
7.329
7.311
7.305
7.297
7.289
7.260
5.862
5.845
5.819
5.819
5.228
5.188
5.185
5.172
5.168
5.165
5.146
5.142
4.633
4.630
4.626
4.620
4.616
4.612
4.607
4.603
4.599
4.593
4.589
4.585
4.349
4.338
4.323
4.313
3.998
3.972
3.953
3.927
3.314
3.304
3.269
3.259

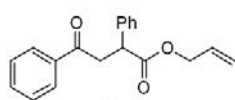


3r

¹H NMR (400 MHz, CDCl₃)



197.508
172.956
138.229
136.300
133.252
131.899
128.834
128.535
128.015
127.792
127.498
117.825
77.322
77.004
76.686
65.510
46.387
42.677



3r

¹³C NMR (100 MHz, CDCl₃)

