Supplementary Information

Practical allylation with unactivated allylic alcohols under mild conditions

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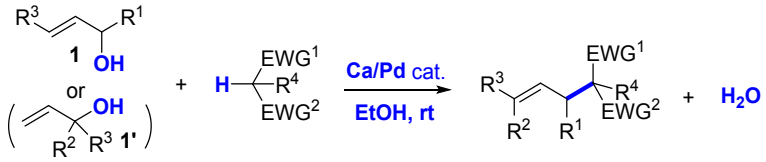
Contents

1. General Information

Unless otherwise noted, all commercially available compounds were used as received. All solvents were purified according to standard procedures. The ^{1}H NMR and spectra was recorded at 400MHz, ^{13}C NMR was recorded at 101MHz, ^{19}F NMR and spectra were recorded at 376 MHz. ^{1}H and ^{13}C NMR Chemical shifts were calibrated to tetramethylsilane as an external reference. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), d (doublet), t (triplet), dd (doublet of doublets), m (multiplet); coupling constants (*J*) are in Hertz (Hz). IR spectra were recorded on a Thermo Scientific Nicolet iS-5 FT-IR spectrometer and are reported in terms of frequency of absorption (cm^{-1}). HRMS were obtained on an IonSpec FT-ICR mass spectrometer with ESI resource. Melting points were measured on a RY-I apparatus and are reported uncorrected. The starting materials **1**[1,2] and **1'**[3,4,5] were readily prepared according to the related literatures, and the Cinnamyl alcohol was purchased from Energy Chemical (Shanghai). The starting materials **2** and the catalyst $Pd(PPh_3)_4$ were purchased from Energy Chemical (Shanghai). The catalyst $Ca(OTf)_2$ was purchased from Tokyo Chemical Industry (TCI) (Shanghai).

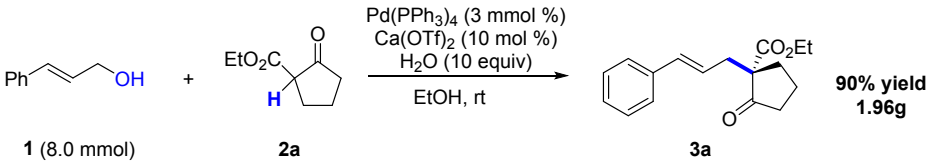
2. General Procedure

2.1 General procedure for preparation of 3



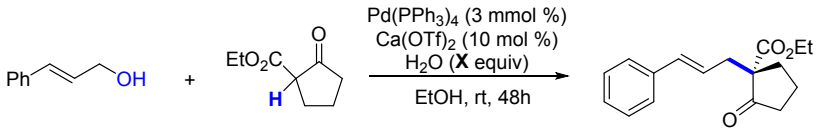
1,3-dicarbonyl compounds **2** (0.60 mmol), allyl alcohols **1** or **1'** (0.30 mmol) was dissolved in EtOH (2.0 mL) at a dried Schlenk tube (10 mL), $Pd(PPh_3)_4$ (0.009-0.03 mmol), $Ca(OTf)_2$ (0.03 mmol) and H_2O (3 mmol) was then added subsequently under nitrogen atmosphere. The reaction was stirred at rt-60 °C (metal bath) for 48h. After complete conversion, the solvent was removed under reduced pressure. The residue was purified by silica gel chromatography or PTLC (for the details, see each compound) to afford the corresponding products **3**. (For compounds **3a**, **3d**, **3e**, **3f**, **3i**, **3m**, **3o**, **3r**, **3s**, **3u** were generated from **1**; for compounds **3b**, **3c**, **3g**, **3h**, **3j - 3l**, **3n**, **3p**, **3q**, **3t**, **3v - 3z**, **3aa**, **3ax**, **3az** were generated from **1'**).

2.2 Procedure for gram scale (8.0 mmol) reaction



(E)-3-phenylprop-2-en-1-ol **1** (8.0 mmol), ethyl 2-oxocyclopentane-1-carboxylate **2a** (16.0 mmol) were dissolved in EtOH (20.0 mL) in Schlenk tube (100 mL), $Pd(PPh_3)_4$ (0.24 mmol), $Ca(OTf)_2$ (0.8 mmol) and H_2O (80 mmol) were then added subsequently under nitrogen. The reaction was stirred at rt for 48h. After complete conversion, the solvent was removed under reduced pressure. The residue was purified via column chromatography (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 50:1) to afford the corresponding products **3a** in 90% yield (1.96g).

2.3 The effect of water loading on this reaction.



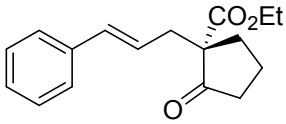
1 **2a** **3a**

Entry	H_2O (X) equiv.	Yield **3a** (%)
1	0	40
2	1.0	50
3	3.0	66
4	10.0	90
5	15.0	90

a Allyl alcohols **1** (0.3 mmol) and **2a** (0.6 mmol) was dissolved in solvent (2.0 mL) in Schlenk tube (10 mL), $Pd(PPh_3)_4$, $Ca(OTf)_2$, and water was then added subsequently. The reaction was stirred at (25-30 °C) (metal bath) for 48h.

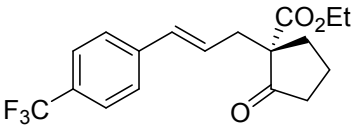
3. Analytical Data for All New Compounds

Ethyl 1-cinnamyl-2-oxocyclopentane-1-carboxylate (3a)



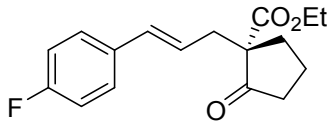
3a was known compounds[6-15]. Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3a** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (73 mg, 90% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 – 7.26 (m, 4H), 7.22 (d, *J* = 6.6 Hz, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.09 (dt, *J* = 15.6, 7.5 Hz, 1H), 4.18 (qd, *J* = 7.1, 1.5 Hz, 2H), 2.82 (dd, *J* = 13.9, 7.4 Hz,1H), 2.56 – 2.39 (m, 3H), 2.30 – 2.20 (m, 1H), 2.04 (dtt, *J* = 13.2, 8.1, 3.8 Hz, 2H), 1.97 – 1.86 (m, 1H), 1.26 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.9, 171.1, 137.1, 134.2, 128.6, 127.6, 126.3, 124.6, 61.6, 60.4, 38.3, 37.1, 32.3, 19.7, 14.3. IR (KBr): 3026.11, 2976.80, 1750.06, 1724.85, 1448.52, 1222.18, 1147.59, 1028.30, 969.54, 747.70, 694.33 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{17}H_{21}O_3$ 273.1491, found 273.1497.

Ethyl (E)-2-oxo-1-(3-(4-(trifluoromethyl)phenyl)allyl)cyclopentane-1-carboxylate (3b)



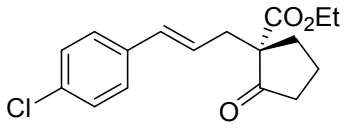
Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3b** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (101 mg, 99% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 7.9 Hz, 2H), 7.39 (d, *J* = 8.1 Hz, 2H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.20 (dt, *J* = 15.5, 7.5 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.81 (dd, *J* = 14.0, 7.3 Hz, 1H), 2.56 – 2.38 (m, 3H), 2.29 – 2.18 (m, 1H), 2.09 – 1.86 (m, 3H), 1.23 (td, *J* = 7.1, 1.3 Hz, 3H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.37. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.6, 171.0, 140.5, 132.8, 129.2 (d, *J* = 32.4 Hz), 127.7, 126.4, 125.6 (q, *J* = 4.1 Hz), 122.9, 61.7, 60.2, 38.1, 37.0, 32.5, 19.7 , 14.2. IR (KBr): 2978.57, 1751.06, 1726.02, 1614.61, 1413.33, 1325.85, 1162.89, 1120.01, 1016.63, 858.64, 755.93, 598.63 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{18}H_{20}F_3O_3$ 341.1365, found 341.1361.

Ethyl (E)-1-(3-(4-fluorophenyl)allyl)-2-oxocyclopentane-1-carboxylate (3c)



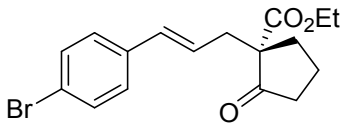
Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3c** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (85 mg, 98% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.32 – 7.26 (m, 2H), 6.98 (t, *J* = 8.7 Hz, 2H), 6.41 (d, *J* = 15.8 Hz, 1H), 6.01 (dt, *J* = 15.3, 7.5 Hz, 1H), 4.18 (qd, *J* = 7.1, 1.3 Hz, 2H), 2.84 – 2.77 (m, 1H), 2.55 – 2.41 (m, 3H), 2.32 – 2.20 (m, 1H), 2.03 (tdt, *J* = 11.9, 7.8, 4.3 Hz, 2H), 1.97 – 1.87 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -114.61. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.8, 171.0, 163.4, 161.0, 133.3 (d, *J* = 3.3 Hz), 132.9, 127.7 (d, *J* = 7.9 Hz), 124.4 (d, *J* = 2.3 Hz), 115.6, 115.3, 61.6, 60.3, 38.1, 37.0, 32.4, 19.7, 14.2. IR (KBr): 2977.82, 1749.94, 1724.93, 1601.35, 1508.67, 1447.93, 1405.57, 1366.53, 1226.83, 1157.86, 1114.59, 1028.42, 971.29, 844.44, 766.88, 567.65, 512.25cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{17}H_{20}FO_3$ 291.1396, found 291.1390.

Ethyl (E)-1-(3-(4-chlorophenyl)allyl)-2-oxocyclopentane-1-carboxylate (3d)



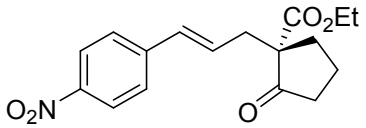
3d was known compounds[12].Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3d** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (75 mg, 82% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.25 (s, 4H), 6.40 (d, *J* = 15.8 Hz, 1H), 6.08 (dt, *J* = 15.7, 7.5, 1H), 4.18 (qd, *J* = 7.1, 1.2 Hz, 2H), 2.81 (ddd, *J* = 14.0, 7.3, 1.3 Hz, 1H), 2.55 – 2.41 (m, 3H), 2.31 – 2.21 (m, 1H), 2.10 – 1.87 (m, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.8, 171.0, 135.6, 133.1, 132.9, 128.8, 127.5, 125.4, 61.7, 60.3, 38.2, 37.0, 32.5, 19.7, 14.2. IR (KBr): 2975.29, 1490.73, 1447.98, 1222.62, 1160.73, 1092.46, 1028.47, 971.23, 815.00, 750.35, 549.74cm^{-1}. HRMS (ESI/[M+Na]$^{+}$) Calcd. for: $C_{17}H_{20}ClO_3$ 307.1101, found 307.1095.

Ethyl (E)-1-(3-(4-bromophenyl)allyl)-2-oxocyclopentane-1-carboxylate (3e)



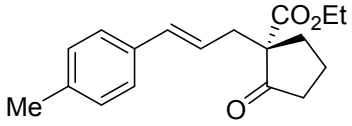
Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3e** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (102 mg, 97% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.40 (dt, *J* = 8.4, 1.5 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H), 6.38 (d, *J* = 15.8 Hz, 1H), 6.10 (dt, *J* = 15.6, 7.5 Hz, 1H), 4.18 (qq, *J* = 7.0, 1.2 Hz, 2H), 2.83 – 2.77 (m, 1H), 2.55 – 2.40 (m, 3H), 2.31 – 2.20 (m, 1H), 2.10 – 1.96 (m, 2H), 1.95 – 1.88 (m, 1H), 1.25 (tt, *J* = 7.1, 1.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.8, 171.0, 136.0, 132.9, 131.7, 127.8, 125.6, 121.2, 61.7, 60.2, 38.2, 37.0, 32.5, 19.7, 14.3. IR (KBr): 2976.15, 1750.36, 1725.26, 1602.79, 1447.42, 1220.40, 1159.32, 1028.89, 970.45, 860.69, 766.76, 694.25, 526.30cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{17}H_{20}BrO_3$ 351.0596, found 351.0598.

Ethyl (E)-1-(3-(4-nitrophenyl)allyl)-2-oxocyclopentane-1-carboxylate (3f)



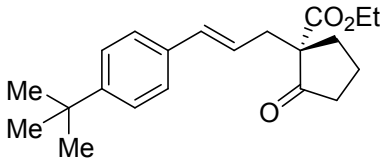
Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3f** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (92 mg, 97% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.10 (dq, *J* = 9.3, 2.4 Hz, 2H), 7.41 (dd, *J* = 8.8, 1.6 Hz, 2H), 6.47 (d, *J* = 15.8 Hz, 1H), 6.35 – 6.26 (m, 1H), 4.14 (qd, *J* = 7.1, 2.0 Hz, 2H), 2.81 (ddt, *J* = 14.0, 7.1, 1.5 Hz, 1H), 2.55 – 2.38 (m, 3H), 2.29 – 2.19 (m, 1H), 2.08 – 1.87 (m, 3H), 1.21 (td, *J* = 7.1, 2.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.4, 170.9, 146.8, 143.5, 132.1, 130.3, 126.8, 124.0, 61.7, 60.1, 38.0, 37.1, 32.8, 19.7, 14.2. IR (KBr): 2975.78, 2360.06, 1750.05, 1724.36, 1596.63, 1516.38, 1448.59, 1342.68, 1161.59, 1028.15, 973.61, 858.90, 744.75, 549.71cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{17}H_{20}NO_5$ 318.1341, found 318.1344.

Ethyl (E)-2-oxo-1-(3-(p-tolyl)allyl)cyclopentane-1-carboxylate (3g)



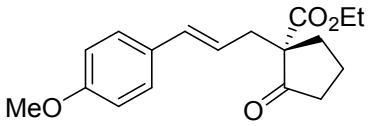
Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3g** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as light yellow solid. (85 mg, 99% yield). Mp: 43-45 °C. ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.22 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.41 (d, *J* = 15.8 Hz, 1H), 6.03 (dt, *J* = 15.4, 7.5 Hz, 1H), 4.17 (qd, *J* = 7.1, 1.9 Hz, 2H), 2.80 (ddd, *J* = 13.9, 7.4, 1.2 Hz, 1H), 2.55 – 2.39 (m, 3H), 2.32 (s, 3H), 2.29 – 2.19 (m, 1H), 2.08 – 1.97 (m, 2H), 1.92 (dddd, *J* = 14.6, 11.6, 6.4, 3.6 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.0, 171.1, 137.3, 134.3, 134.1, 129.3, 126.2, 123.5, 61.6, 60.4, 38.3, 37.1, 32.3, 21.3, 19.7, 14.3. IR (KBr): 2973.69, 2923.52, 1750.68, 1725.29, 1512.60, 1447.89, 1222.69, 1160.26, 1028.95,971.35, 801.31, 750.80, 503.46cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{18}H_{23}O_3$ 287.1647, found 287.1646.

Ethyl (E)-1-(3-(4-(tert-butyl)phenyl)allyl)-2-oxocyclopentane-1-carboxylate (3h)



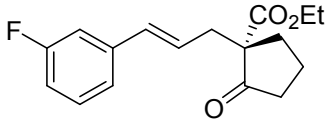
Following the general procedure, the reaction was conducted in 0.3 mmol scale**, 3h** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (78 mg, 79% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.32 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 6.43 (d, *J* = 15.6 Hz, 1H), 6.04 (dt, *J* = 15.1, 7.5 Hz, 1H), 4.18 (qd, *J* = 7.1, 1.7 Hz, 2H), 2.80 (dd, *J* = 13.9, 7.4 Hz, 1H), 2.56 – 2.38 (m, 3H), 2.29 – 2.19 (m, 1H), 2.08 – 1.98 (m, 2H), 1.97 – 1.85 (m, 1H), 1.30 (s, 9H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.9, 171.1, 150.6, 134.4, 134.0, 126.0, 125.5, 123.8, 61.6, 60.4, 38.3, 37.1, 34.6, 32.2, 31.4, 19.7, 14.3. IR (KBr): 2963.17, 1750.49, 1724.74, 1463.32, 1364.20, 1274.31, 1222.89, 1146.26, 1110.04, 1027.10, 971.08, 913.71, 819.11, 764.25, 749.53cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{21}H_{29}O_3$ 329.2117, found 329.2122.

Ethyl (E)-1-(3-(4-methoxyphenyl)allyl)-2-oxocyclopentane-1-carboxylate (3i)



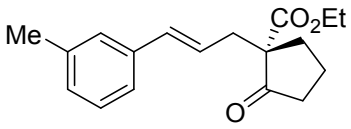
3i was known compounds[12]. Following the general procedure, the reaction was conducted in 0.3 mmol scale, **3i** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (86 mg, 95% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.23 (m, 2H), 6.85 – 6.81 (m, 2H), 6.39 (d, *J* = 15.8 Hz, 1H), 5.98 – 5.89 (m, 1H), 4.18 (qd, *J* = 7.1, 1.9 Hz, 2H), 3.79 (s, 3H), 2.79 (ddd, *J* = 13.9, 7.4, 1.2 Hz, 1H), 2.55 – 2.39 (m, 3H), 2.33 – 2.17 (m, 1H), 2.09 – 1.98 (m, 2H), 2.01 – 1.85 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.9, 171.1, 159.2, 133.6, 129.9, 127.4, 122.3, 114.0, 61.6, 60.4, 55.4, 38.2, 37.1, 32.2, 19.7, 14.2. IR (KBr): 2966.07, 2837.05, 1749.68, 1724.43, 1607.35, 1511.06, 1464.93, 1249.17, 1174.65, 1033.09, 970.61, 838.69,756.99cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{18}H_{23}O_4$ 303.1596, found 303.1597.

Ethyl (E)-1-(3-(3-fluorophenyl)allyl)-2-oxocyclopentane-1-carboxylate (3j)



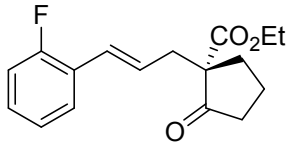
Following the general procedure, the reaction was conducted in 0.3 mmol scale. **3j** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (84 mg, 96% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.25 (td, *J* = 8.1, 6.5 Hz, 1H), 7.08 (d, *J* = 7.8 Hz, 1H), 7.02 (dt, *J* = 10.3, 1.8 Hz, 1H), 6.94 – 6.88 (m, 1H), 6.41 (d, *J* = 15.7 Hz, 1H), 6.12 (dt, *J* = 15.2, 7.5 Hz, 1H), 4.19 (qd, *J* = 7.1, 1.0 Hz, 2H), 2.82 (ddd, *J* = 14.0, 7.3, 1.0 Hz, 1H), 2.55 – 2.41 (m, 3H), 2.32 – 2.22 (m, 1H), 2.10 – 1.96 (m, 2H), 1.93 (ddd, *J* = 14.1, 6.5, 3.6 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.45. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.7 , 171.0 , 163.1 (d, $J_{C\text{-}F}$ = 245.1 Hz), 139.4 (d, *J* = 7.8 Hz), 133.0 (d, *J* = 2.8 Hz), 130.1 (d, *J* = 8.5 Hz), 126.2, 122.2 (d, *J* = 2.9 Hz), 114.3 (d, *J* = 21.4 Hz), 112.7 (d, *J* = 21.7 Hz), 61.7, 60.2, 38.2, 37.0, 32.4, 19.7, 14.2. IR (KBr): 2977.86, 1750.17, 1724.95, 1609.67, 1582.87, 1487.07, 1446.83, 1267.35, 1231.06, 1144.33, 1113.93, 1028.27, 970.82, 870.13, 780.27, 764.72, 750.62, 685.41cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{17}H_{20}FO_3$ 291.1396, found 291.1400.

Ethyl (E)-2-oxo-1-(3-(m-tolyl)allyl)cyclopentane-1-carboxylate (3k)



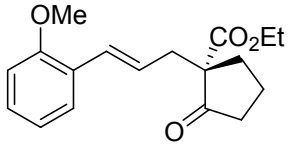
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3k** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (80 mg, 93% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.17 (t, *J* = 7.5 Hz, 1H), 7.15 – 7.10 (m, 2H), 7.02 (d, *J* = 7.3 Hz, 1H), 6.41 (d, *J* = 15.8 Hz, 1H), 6.11 – 6.02 (dt, *J* = 15.7, 7.5 Hz, 1H), 4.17 (qd, *J* = 7.1, 1.4 Hz, 2H), 2.80 (ddd, *J* = 13.9, 7.4, 1.3 Hz, 1H), 2.56 – 2.38 (m, 3H), 2.32 (s, 3H), 2.30 – 2.19 (m, 1H), 2.08 – 1.96 (m, 2H), 1.95 – 1.87 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.0, 171.1, 138.2, 137.0, 134.3, 128.5, 128.3, 127.0, 124.4, 123.5, 61.6, 60.4, 38.3, 37.1, 32.3, 21.5, 19.7, 14.3. IR (KBr): 3026.84, 2975.37, 1749.48, 1724.55, 1587.83, 1486.93, 1401.77, 1222.48, 1160.77, 1072.251028.33, 971.22, 814.40, 750.68, 549.62cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{23}O_3$ 287.1647, found 287.1645.

Ethyl (E)-1-(3-(2-fluorophenyl)allyl)-2-oxocyclopentane-1-carboxylate (3l)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3l** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (84 mg, 97% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (td, *J* = 7.7, 1.6 Hz, 1H), 7.18 (q, *J* = 6.6 Hz, 1H), 7.09 – 6.97 (m, 2H), 6.60 (d, *J* = 15.9 Hz, 1H), 6.22 – 6.14 (dt, *J* = 15.9, 7.9 Hz, 1H), 4.19 (qd, *J* = 7.1, 2.9 Hz, 2H), 2.85 (dd, *J* = 14.0, 7.4 Hz, 1H), 2.58 – 2.40 (m, 3H), 2.32 – 2.22 (m, 1H), 2.10 – 1.97 (m, 2H), 1.97 – 1.88 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -118.42. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.7 , 171.0 , 160.0 (d, $J_{C\text{-}F}$ = 248.8 Hz), 128.8 (d, *J* = 8.4 Hz), 127.4 (dd, *J* = 13.4, 4.4 Hz), 126.5 (d, *J* = 3.8 Hz), 124.8 (d, *J* = 12.3 Hz), 124.1 (d, *J* = 3.7 Hz), 115.7 (d, *J* = 22.1 Hz), 61.6, 60.3, 38.2, 37.5, 32.4, 19.7, 14.2. IR (KBr): 2978.12, 1750.36, 1724.95, 1486.97, 1456.36, 1265.51, 1229.20, 1188.98, 1149.25, 1118.39 1031.37, 972.55, 859.64, 829.62, 755.91cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{17}H_{20}FO_3$ 291.1396, found 291.1390.

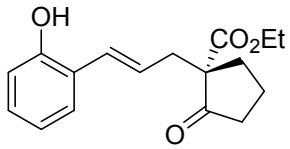
Ethyl (E)-1-(3-(2-methoxyphenyl)allyl)-2-oxocyclopentane-1-carboxylate (3m)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3m** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (89 mg, 98% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.23 – 7.17 (m, 1H), 6.89 (t, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 8.3 Hz, 1H), 6.77 (d, *J* = 15.9 Hz, 1H), 6.07 (dt, *J* = 15.8, 7.5 Hz, 1H), 4.18 (qq, *J* = 7.1, 3.7 Hz, 2H), 3.82 (s, 3H), 2.84 (dd, *J* = 13.9, 7.5 Hz, 1H), 2.57 – 2.39 (m, 3), 2.31 – 2.20 (m, 1H), 2.10 – 1.98 (m, 2H), 1.97 – 1.88 (m, 1H), 1.26 (td, *J* = 7.1, 1.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.9, 171.1, 156.5, 128.9, 128.5, 126.7, 126.2, 125.1, 120.7, 110.9, 61.6, 60.5, 55.5, 38.2, 37.6, 32.3, 19.7, 14.2. IR (KBr): 2965.05, 2837.63, 1749.97, 1724.18, 1597.19, 1488.73, 1463.84,

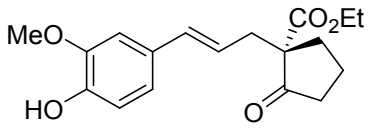
1243.67, 1161.09, 1027.91, 975.99, 859.59, 816.23, 752.80, 653.45, 549.79cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{23}O_4$ 303.1596, found 303.1601.

Ethyl (E)-1-(3-(2-hydroxyphenyl)allyl)-2-oxocyclopentane-1-carboxylate (3n)



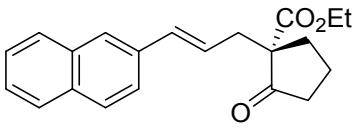
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3n** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (66 mg, 76% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.28 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.09 (td, *J* = 7.7, 1.6 Hz, 1H), 6.88 – 6.78 (m, 2H), 6.68 (d, *J* = 15.9 Hz, 1H), 6.08 (dt, *J* = 15.5, 7.5 Hz, 1H), 6.02 (s, 1H), 4.18 (qd, *J* = 7.1, 1.4 Hz, 2H), 2.81 (ddd, *J* = 13.9, 7.6, 0.9 Hz, 1H), 2.57 (ddd, *J* = 13.9, 7.3, 1.1 Hz, 1H), 2.52 – 2.41 (m, 2H), 2.28 (dt, *J* = 18.4, 7.9 Hz, 1H), 2.11 – 1.98 (m, 2H), 1.98 – 1.88 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.8, 171.4, 153.2, 128.9, 128.6, 127.4, 126.1, 124.3, 120.6, 116.0, 61.9, 60.5, 38.4, 37.6, 32.7, 19.7, 14.2. IR (KBr): 3440.63, 2976.14, 1722.06, 1603.04, 1455.40 1340.84, 1230.34, 1159.02, 1026.86, 977.04, 858.27, 753.51, 653.49, 549.68cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{17}H_{21}O_4$ 289.1440, found 289.1438.

Ethyl (E)-1-(3-(4-hydroxy-3-methoxyphenyl)allyl)-2-oxocyclopentane-1-carboxylate (3o)



Following the general procedure, The reaction was conducted in 0.3 mmol scale with **3o** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (40 mg, 42% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.85 – 6.83 (m, 3H), 6.36 (d, *J* = 15.7 Hz, 1H), 5.92 (dt, *J* = 15.4, 7.5 Hz, 1H), 5.71 (d, *J* = 10.1 Hz, 1H), 4.18 (qd, *J* = 7.1, 1.2 Hz, 2H), 3.89 (d, *J* = 1.5 Hz, 3H), 2.79 (dd, *J* = 14.0, 7.4 Hz, 1H), 2.55 – 2.40 (m, 3H), 2.26 (dt, *J* = 19.5, 8.2 Hz, 1H), 2.10 – 1.99 (m, 2H), 1.97 – 1.88 (m, 1H), 1.26 (td, *J* = 7.1, 0.7 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.1, 171.2, 146.7, 145.4, 134.0, 129.7, 122.1, 120.0, 114.5, 108.2, 61.6, 60.4, 56.0, 38.3, 37.0, 32.3, 19.7, 14.2. IR (KBr): 2975.93, 1747.42, 1722.20, 1597.05 1514.74, 1464.20, 1275.17, 1233.46, 1156.65, 1122.24, 1031.60, 969.31, 858.46, 764.25, 749.99cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{23}O_5$ 319.1545, found 319.1550.

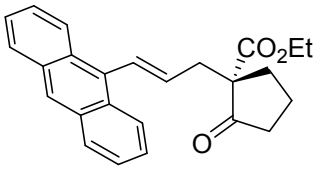
Ethyl (E)-1-(3-(naphthalen-2-yl)allyl)-2-oxocyclopentane-1-carboxylate (3p)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3p** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (95 mg, 98% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.75 (t, *J* = 8.7 Hz, 3H), 7.66 (s, 1H), 7.53 (d, *J* = 8.5 Hz, 1H), 7.46 – 7.37 (m, 2H), 6.59 (d, *J* = 15.7 Hz, 1H), 6.22 (dt, *J* = 15.0, 7.5 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 2.87 (dd, *J* = 13.9, 7.3 Hz, 1H), 2.56 (dd, *J* = 14.0, 7.6 Hz, 1H), 2.45 (ddd, *J* = 29.8, 13.0, 6.9 Hz, 2H), 2.31 – 2.20 (m, 1H), 2.03 (tt, *J* = 15.3, 7.5 Hz, 2H), 1.94 – 1.86 (m, 1H), 1.24 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.9, 171.1, 134.6, 134.3, 133.7, 133.0, 128.3, 128.0, 127.8, 125.9, 125.1,

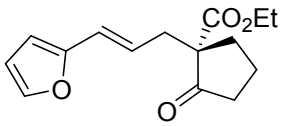
61.7, 60.4, 38.3, 37.2, 32.4, 19.7, 14.3. IR (KBr): 2976.36, 1749.41, 1723.96, 1596.84, 1507.80, 1446.82, 1222.70, 1160.30, 1027.75, 969.32, 860.09, 813.68, 748.14, 476.33cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{21}H_{23}O_3$ 323.1647, found 323.1647.

Ethyl (E)-1-(3-(anthracen-9-yl)allyl)-2-oxocyclopentane-1-carboxylate (3q)



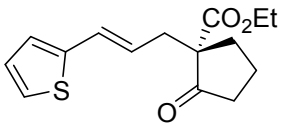
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3q** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (85 mg, 76% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 8.31 (s, 1H), 8.23 (d, *J* = 2.4 Hz, 1H), 8.22 – 8.20 (m, 1H), 7.96 – 7.91 (m, 2H), 7.47 – 7.39 (m, 4H), 7.19 (d, *J* = 16.0 Hz, 1H), 5.91 (dt, *J* = 15.9, 7.4 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 3.12 (ddd, *J* = 13.9, 7.2, 1.3 Hz, 1H), 2.79 (ddd, *J* = 13.9, 7.6, 1.3 Hz, 1H), 2.66 – 2.58 (m, 1H), 2.52 – 2.42 (m, 1H), 2.26 (dt, *J* = 18.5, 8.2 Hz, 1H), 2.19 – 2.01 (m, 2H), 2.01 – 1.89 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.7, 171.0, 133.3, 132.7, 131.5, 130.2, 129.6, 128.8, 126.4, 126.0, 125.5, 125.2, 61.8, 60.5, 38.2, 38.0, 32.8, 19.8, 14.3. IR (KBr): 2984.41, 1748.59, 1723.68, 1672.94, 1593.95, 1455.68, 1275.36, 1260.67, 1026.90, 1026.90, 764.18, 750.04cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{25}H_{25}O_3$ 373.1804, found 373.1798.

Ethyl (E)-1-(3-(furan-2-yl)allyl)-2-oxocyclopentane-1-carboxylate (3r)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3r** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (40 mg, 51% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.31 (d, *J* = 0.8 Hz, 1H), 6.35 (dd, *J* = 3.1, 1.8 Hz, 1H), 6.26 (d, *J* = 15.9 Hz, 1H), 6.17 (d, *J* = 3.2 Hz, 1H), 6.00 (dt, *J* = 15.5, 7.6 Hz, 1H), 4.18 (q, *J* = 7.0 Hz, 2H), 2.79 (dd, *J* = 14.0, 7.5 Hz, 1H), 2.53 – 2.40 (m, 3H), 2.32 – 2.21 (m, 1H), 2.08 – 1.99 (m, 2H), 1.98 – 1.87 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.8, 170.9, 152.5, 141.8, 123.3, 122.5, 111.3, 107.4, 61.6, 60.3, 38.2, 36.9, 32.2, 19.7, 14.2. IR (KBr): 2962.37, 2928.76, 1749.18, 1724.25, 1449.07, 1340.37, 1161.74, 1019.67, 975.33, 921.05, 816.08, 748.55, 653.76, 549.75cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{15}H_{19}O_4$ 263.1283, found 263.1282.

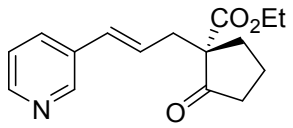
Ethyl (E)-2-oxo-1-(3-(thiophen-2-yl)allyl)cyclopentane-1-carboxylate (3s)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3s** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (78 mg, 94% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.10 (d, *J* = 5.0 Hz, 1H), 6.93 (t, *J* = 4.2 Hz, 1H), 6.89 (s, 1H), 6.57 (d, *J* = 15.6 Hz, 1H), 5.91 (dt, *J* = 15.3, 7.5 Hz, 1H), 4.17 (q, *J* = 7.0 Hz, 2H), 2.78 (dd, *J* = 13.9, 7.4 Hz, 1H), 2.45 (ddt, *J* = 19.5, 13.5, 7.3 Hz, 3H), 2.31 – 2.20 (m, 1H), 2.02 (dt, *J* = 12.4, 5.4 Hz, 2H), 1.97 – 1.86 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.8, 171.0, 142.1, 127.4,

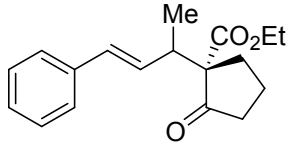
127.3, 125.3, 124.3, 124.0, 61.6, 60.3, 38.2, 36.9, 32.3, 19.7, 14.2. IR (KBr): 2977.08, 1749.15, 1723.98, 1446.67, 1403.78, 1365.74, 1274.83, 1226.39, 1147.61, 1110.27, 1028.00, 960.03, 913.68, 853.99, 764.24, 749.38, 699.54cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{15}H_{19}SO_3$ 279.1055, found 279.1051.

Ethyl (E)-2-oxo-1-(3-(pyridin-3-yl)allyl)cyclopentane-1-carboxylate (3t)



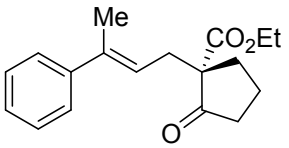
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3t** was isolated by PTLC (Petroleum ether (bp: 60-90 ^{o}C)/ethyl acetate = 2:1) as yellow oil (43 mg, 53% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 8.54 (s, 1H), 8.45 (d, *J* = 4.1 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.23 (dd, *J* = 7.9, 4.8 Hz, 1H), 6.44 (d, *J* = 15.9 Hz, 1H), 6.21 (dt, *J* = 15.8, 7.5 Hz, 1H), 4.19 (q, *J* = 6.9 Hz, 2H), 2.84 (ddd, *J* = 14.0, 7.2, 1.0 Hz, 1H), 2.58 – 2.42 (m, 3H), 2.28 (ddd, *J* = 18.4, 10.6, 4.1 Hz,1H), 2.11 – 1.98 (m, 2H), 1.98 – 1.90 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.6, 170.9, 148.6, 148.2, 132.7, 130.5, 127.3, 123.5, 61.7, 60.2, 38.1, 37.1, 32.6, 19.7, 14.2. IR (KBr): 2966.28, 1748.98, 1723.32, 1447.81, 1414.65, 1338.31, 1225.58, 1160.70, 1024.34, 971.48, 920.34, 815.08, 708.65, 653.33, 549.18cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{20}NO_3$ 274.1443, found 274.1447.

Ethyl 2-oxo-1-((E)-4-phenylbut-3-en-2-yl)cyclopentane-1-carboxylate (3u)



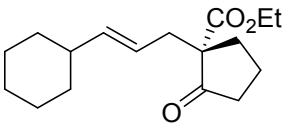
3u was known compounds[16].Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3u** was isolated by PTLC (Petroleum ether (bp: 60-90 ^{o}C)/ethyl acetate = 2:1) as yellow oil (67 mg, 78% yield (mixture of *syn* and *anti*-isomer, major/minor = 70/30 determined by ^{1}H NMR of the crude reaction mixture)). For the major isomer, ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.34 – 7.26 (m, 4H), 7.24 – 7.18 (m, 1H), 6.41 (d, *J* = 15.9 Hz, 1H), 6.06 (dd, *J* = 15.9, 8.0 Hz, 1H), 4.20 (qq, *J* = 10.7, 7.1 Hz, 2H), 3.32 – 3.24 (m, 1H), 2.56 – 2.50 (m, 1H), 2.40 – 2.32 (m, 1H), 2.13 – 2.01 (m, 1H), 2.00 – 1.87 (m, 3H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.11 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.3, 169.9, 137.2, 132.1, 130.0, 128.6, 127.5, 126.3, 65.4, 61.7, 40.9, 39.1, 28.4, 20.0, 15.9, 14.3. IR (KBr): 2974.13, 1749.87, 1719.27, 1447.45, 1259.74, 1223.83, 1123.81, 1028.78, 972.53, 750.27, 694.73cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{23}O_3$ 287.1647, found 287.1638. For the minor isomer, ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.34 – 7.27 (m, 4H), 7.24 – 7.19 (m, 1H), 6.43 (d, *J* = 15.8 Hz, 1H), 6.02 (dd, *J* = 15.8, 8.6 Hz, 1H), 4.24 – 4.08 (m, 2H), 3.32 – 3.24 (m, 1H), 2.54 – 2.39 (m, 2H), 2.16 (ddd, *J* = 18.9, 10.4, 8.7 Hz, 1H), 2.08 – 1.90 (m, 3H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.08 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.6, 170.1, 137.2, 131.4, 130.6, 128.6, 127.5, 126.3, 64.9, 61.7, 41.3, 39.3, 28.9, 19.8, 15.9, 14.3. IR (KBr): 2968.79, 1750.09, 1720.26, 1447.40, 1269.69, 1225.46, 1119.70, 1020.71, 968.55, 759.29, 693.89cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{23}O_3$ 287.1647, found 287.1641.

Ethyl (E)-2-oxo-1-(3-phenylbut-2-en-1-yl)cyclopentane-1-carboxylate (3v)



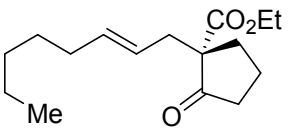
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3v** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) as yellow oil (69 mg, 80% yield) ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.31 (m, 3H), 7.31 – 7.28 (m, 1H), 7.26 – 7.21 (m, 1H), 5.64 (td, *J* = 7.6, 1.4 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 2.85 (dd, *J* = 14.7, 7.8 Hz, 1H), 2.61 – 2.41 (m, 3H), 2.32 – 2.21 (m, 1H), 2.06 (s, 3H), 2.04 – 1.90 (m, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.1, 171.2, 143.6, 138.5, 128.3, 127.1, 125.9, 122.6, 61.6, 60.6, 38.3, 32.7, 32.4, 19.8, 16.3, 14.2. IR (KBr): 2985.54, 1749.18, 1724.05, 1446.37, 1275.33, 1260.66, 1223.86, 1147.86, 1027.12, 750.10, 699.24cm^{-1}. HRMS (ESI/[M+H^{+}) Calcd. for: $C_{18}H_{23}O_3$ 287.1647, found 287.1639.

Ethyl (E)-1-(3-cyclohexylallyl)-2-oxocyclopentane-1-carboxylate (3w)



Following the general procedure, the reaction was conducted at 60 °C in 0.3 mmol scale with **3w** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (62 mg, 74% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 5.45 (dd, *J* = 15.3, 6.8 Hz, 1H), 5.23 (dt, *J* = 15.3, 7.3 Hz, 1H), 4.15 (qd, *J* = 7.0, 1.9 Hz, 2H), 2.58 (dd, *J* = 13.8, 7.2 Hz, 1H), 2.46 – 2.36 (m, 2H), 2.30 (dd, *J* = 13.8, 7.3 Hz, 1H), 2.21 (dt, *J* = 18.4, 7.8 Hz, 1H), 2.04 – 1.85 (m, 4H), 1.73 – 1.59 (m, 5H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.23 – 0.95 (m, 5H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -114.23, -115.28. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.0, 171.1, 141.5, 121.5, 61.4, 60.4, 40.8, 38.3, 36.8, 33.1, 33.1, 32.0, 26.2, 26.1, 19.6, 14.2. IR (KBr): 2924.01, 2851.01, 1752.11, 1725.83, 1448.23, 1224.20, 1159.53, 1029.52, 972.69, 861.70cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{17}H_{27}O_3$ 279.1960, found 279.1964.

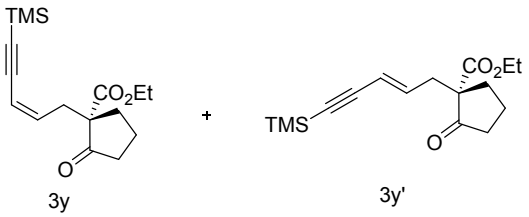
Ethyl 1-(oct-2-en-1-yl)-2-oxocyclopentane-1-carboxylate (3x)



3x was known compounds[12]. Following the general procedure, the reaction was conducted at 60 °C in 0.3 mmol scale with **3x** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (47 mg, 59% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.50 (dt, *J* = 15.0, 6.8 Hz, 1H), 5.27 (dt, *J* = 15.1, 7.3 Hz, 1H), 4.16 (qd, *J* = 7.1, 1.5 Hz, 2H), 2.63 – 2.56 (m, 1H), 2.49 – 2.36 (m, 2H), 2.34 – 2.28 (m, 1H), 2.27 – 2.17 (m, 1H), 2.05 – 1.93 (m, 4H), 1.93 – 1.85 (m, 1H), 1.37 – 1.28 (m, 4H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.23 – 1.18 (m, 2H), 0.88 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.0, 171.1, 135.6, 124.1, 61.4, 60.4, 38.3, 36.8, 32.6, 32.0, 31.4, 29.1, 22.6, 19.6, 14.3, 14.1. IR (KBr): 2958.66, 2926.92, 2856.32, 1752.90, 1726.14, 1465.14, 1223.66, 1160.36, 1029.78, 974.76, 920.90, 844.79, 749,91, 653.60cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{16}H_{27}O_3$ 267.1960, found 267.1960.

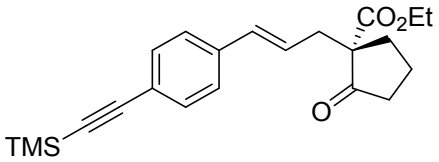
Ethyl (Z)-2-oxo-1-(5-(trimethylsilyl)pent-2-en-4-yn-1-yl)cyclopentane-1-carboxylate (3y)

Ethyl (E)-2-oxo-1-(5-(trimethylsilyl)pent-2-en-4-yn-1-yl)cyclopentane-1-carboxylate (3y')



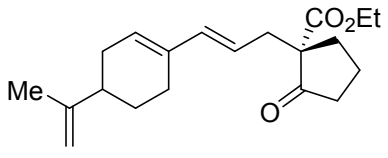
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3y** and **3y'** were isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (77 mg, 88% yield (**3y**/**3y'** = 81/19)). For the **3y**, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.90 (ddd, *J* = 10.8, 8.3, 7.0 Hz, 1H), 5.59 (d, *J* = 10.9 Hz, 1H), 4.14 (qd, *J* = 7.1, 1.3 Hz, 2H), 2.82 (ddd, *J* = 14.1, 7.0, 1.1 Hz, 1H), 2.62 (ddd, *J* = 14.1, 8.3, 1.0 Hz, 1H), 2.45 – 2.34 (m, 2H), 2.33 – 2.23 (m, 1H), 2.05 – 1.91 (m, 3H), 1.22 (t, *J* = 7.1 Hz, 3H), 0.15 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.4, 171.0, 139.3, 113.0 , 101.6, 99.8, 61.6, 60.1, 38.0, 33.5, 32.3, 19.7, 14.1. IR (KBr): 2961.78, 2899.97, 2148.77, 1752,46, 1726.75, 1449.12, 1405.86, 1283.84, 1249.98, 1195.87, 1161.35, 1028.30, 989.58, 844.40, 759.98cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{25}SiO_3$ 293.1573, found 293.1568. For the **3y'**, ^{1}H NMR (401 MHz, Chloroform-*d*) δ 6.06 (dt, *J* = 15.6, 7.6 Hz, 1H), 5.58 (d, *J* = 15.8 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.73 (ddd, *J* = 14.2, 7.4, 1.4 Hz, 1H), 2.50 – 2.39 (m, 2H), 2.39 – 2.34 (m, 1H), 2.32 – 2.21 (m, 1H), 2.10 – 1.98 (m, 1H), 1.98 – 1.88 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H), 0.18 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.3, 170.7, 139.7, 114.0, 103.2, 94.2, 61.7, 59.9, 38.0, 36.9, 32.3, 19.6, 14.2. IR (KBr): 2961.56, 2132.56, 1752.45, 1726.57, 1448.22, 1250.16, 1221.54, 1160.84, 1116.66, 1083.89, 1028.50, 960.64, 844.40, 760.39cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{25}SiO_3$ 293.1573, found 293.1576.

Ethyl (E)-2-oxo-1-(3-(4-((trimethylsilyl)ethynyl)phenyl)allyl)cyclopentane-1-carboxylate (3z)



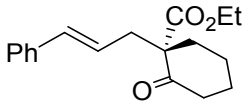
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3z** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 9:1) as yellow oil (105 mg, 95% yield) ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.3 Hz, 2H), 6.38 (d, *J* = 15.8 Hz, 1H), 6.09 (dt, *J* = 15.1, 7.5 Hz, 1H), 4.15 (qd, *J* = 7.1, 1.2 Hz, 2H), 2.79 (ddd, *J* = 14.0, 7.3, 1.0 Hz, 1H), 2.52 – 2.37 (m, 3H), 2.23 (dt, *J* = 18.6, 7.9 Hz, 1H), 2.07 – 1.92 (m, 2H), 1.92 – 1.84 (m, 1H), 1.22 (t, *J* = 7.1 Hz, 3H), 0.22 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.7, 171.0, 137.2, 133.5, 132.2, 126.1, 125.9, 122.0, 105.2, 94.8, 61.7, 60.3, 38.2, 37.1, 32.4, 19.7, 14.2, 0.1. IR (KBr): 2961.87, 2900.06, 2154.54, 1751.51, 1726.21, 1505.44, 1447.45, 1406.91, 1249.89, 1222.64, 1159.24, 1115.63, 1028.48, 971.69, 864.81, 843.56, 760.55, 640.08cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{22}H_{29}SiO_3$ 369.1886, found 369.1892.

Ethyl 2-oxo-1-((E)-3-(4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)allyl)cyclopentane-1-carboxylate (3aa)



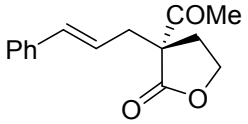
Following the general procedure, the reaction was conducted at 60 °C in 0.3 mmol scale with **3aa** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (88 mg, 93% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.09 (d, *J* = 15.6 Hz, 1H), 5.71 – 5.67 (m, 1H), 5.38 (dt, *J* = 15.3, 7.5 Hz, 1H), 4.73 – 4.70 (m, 2H), 4.17 (qd, *J* = 7.1, 3.2 Hz, 2H), 2.69 (ddd, *J* = 13.8, 7.4, 2.1 Hz, 1H), 2.47 – 2.37 (m, 3H), 2.28 – 2.17 (m, 3H), 2.16 – 2.08 (m, 2H), 2.08 – 1.95 (m, 3H), 1.88 (dddd, *J* = 15.4, 10.0, 4.8, 2.4 Hz, 2H), 1.74 (s, 3H), 1.46 (ddt, *J* = 17.0, 11.5, 5.7 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.0, 171.1, 149.7, 137.3, 135.0, 128.2, 120.5, 108.8, 61.5, 60.4, 41.2, 38.3, 37.0, 32.2, 31.2, 27.4, 25.1, 20.9, 19.7, 14.2. IR (KBr): 2966.14, 2935.11, 1750.14, 1724.72, 1644.31, 1487.02, 1454.41, 1367.12, 1275.02, 1260.80, 1229.10, 1148.90, 1116.75, 1029.51, 971.60, 889.68, 751.60cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{20}H_{29}O_3$ 317.2117, found 317.2108.

Ethyl 1-cinnamyl-2-oxocyclohexane-1-carboxylate (3ab)



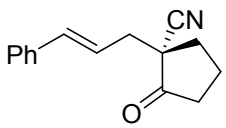
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ab** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 10:1) as yellow oil (64 mg, 75% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.28 (dt, *J* = 15.0, 7.4 Hz, 4H), 7.18 (t, *J* = 7.1 Hz, 1H), 6.37 (d, *J* = 15.8 Hz, 1H), 6.17 (dt, *J* = 15.6, 7.5 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.78 – 2.71 (m, 1H), 2.56 – 2.45 (m, 4H), 2.02 (dq, *J* = 9.2, 2.6 Hz, 1H), 1.80 – 1.60 (m, 3H), 1.56 – 1.48 (m, 1H), 1.21 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.8, 171.7, 137.3, 133.3, 128.6, 127.3, 126.3, 125.2, 61.5, 61.4, 41.3, 38.7, 36.2, 27.6, 22.7, 14.3. IR (KBr): 3025.85, 2940.01, 2865.91, 1713.14, 1495.31, 1448.75, 1308.84, 1223.46, 1191.59, 1134.53, 1095.26, 1021.87, 968.27, 864.35, 744.47, 693.77cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{23}O_3$ 287.1647, found 287.1649.

3-acetyl-3-cinnamyldihydrofuran-2(3H)-one (3ac)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ac** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 10:1) as yellow oil (64 mg, 87% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 – 7.28 (m, 4H), 7.27 – 7.22 (m, 1H), 6.51 (d, *J* = 15.7 Hz, 1H), 5.97 (dt, *J* = 15.6, 8.0 Hz, 1H), 4.28 (td, *J* = 8.9, 3.6 Hz, 1H), 4.18 (td, *J* = 8.9, 7.3 Hz, 1H), 2.92 – 2.79 (m, 3H), 2.38 (s, 3H), 2.17 (dt, *J* = 13.1, 8.8 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 202.3, 175.3, 136.4, 135.2, 128.7, 128.0, 126.4, 122.4, 66.5, 61.4, 38.3, 28.9, 25.9. IR (KBr): 3026.16, 2999.69, 2918.73, 1765.79, 1712.76, 1494.25, 1448.50, 1374.19, 1359.83, 1260.04, 1218.62, 1165.14, 1109.26, 1069.86, 1027.74, 970.64, 753.99, 735.63, 694.52cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{15}H_{17}O_3$ 245.1178, found 245.1176.

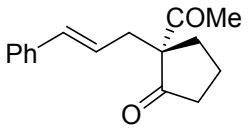
1-cinnamyl-2-oxocyclopentane-1-carbonitrile (3ad)



Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ad** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 10:1) as yellow solid (59 mg, 88% yield). Mp:

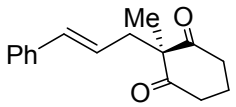
61-63 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.36 (m, 2H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.25 (dd, *J* = 8.3, 5.9 Hz, 1H), 6.54 (d, *J* = 15.7 Hz, 1H), 6.16 (dt, *J* = 15.4, 8.1 Hz, 1H), 2.77 (ddd, *J* = 14.0, 6.7, 1.2 Hz, 1H), 2.55 – 2.31 (m, 4H), 2.18 – 2.06 (m, 2H), 2.01 (dtd, *J* = 13.1, 7.5, 6.5, 1.9 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.1, 136.4, 135.8, 128.8, 128.1, 126.6, 121.8, 119.0, 48.7, 37.3, 36.7, 33.5, 19.3. IR (KBr): 3027.69, 2973.59, 2234.25, 1752.74, 1597.86, 1495.64, 1449.93, 1402.85, 1318.24, 1274.86, 1144.18, 1001.86, 968.26, 821.21, 749.92, 696.28cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{15}H_{16}NO$ 226.1232, found 226.1235.

2-acetyl-2-cinnamylcyclopentan-1-one (3ae)



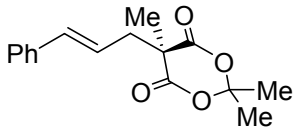
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ae** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 9:1) as yellow oil (65 mg, 90% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.32 – 7.26 (m, 4H), 7.21 (ddd, *J* = 6.1, 5.1, 2.6 Hz, 1H), 6.44 (d, *J* = 15.7 Hz, 1H), 5.94 (dt, *J* = 15.7, 7.4 Hz, 1H), 2.81 (ddd, *J* = 14.3, 7.7, 1.3 Hz, 1H), 2.69 – 2.62 (m, 1H), 2.59 (ddd, *J* = 14.3, 7.1, 1.4 Hz, 1H), 2.41 – 2.24 (m, 2H), 2.24 (s, 3H), 1.93 – 1.82 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.7, 204.0, 136.8, 134.1, 128.7, 127.7, 126.3, 124.0, 68.8, 38.7, 38.4, 30.4, 26.4, 19.5. IR (KBr): 3026.22, 2967.70, 1736.74, 1702.28, 1494.35, 1448.52, 1356.87, 1275.00, 1260.09, 1199.66, 1141.45, 1113.78, 968.87, 913.31, 822.71, 748.21, 695.13cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{19}O_2$ 243.1385, found 243.1379.

2-cinnamyl-2-methylcyclohexane-1,3-dione (3af)



Following the general procedure, $Pd(PPh_3)_4$ (5 mol%) was used. The reaction was conducted at 60 ºC in 0.3 mmol scale with **3af** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 9:1) as yellow oil (60 mg, 82% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31 – 7.28 (m, 3H), 7.27 (d, *J* = 0.8 Hz, 1H), 7.23 – 7.18 (m, 1H), 6.40 (d, *J* = 15.7 Hz, 1H), 5.97 (dt, *J* = 15.7, 7.5 Hz, 1H), 2.69 (dd, *J* = 7.5, 1.3 Hz, 2H), 2.67 – 2.63 (m, 4H), 2.03 – 1.84 (m, 2H), 1.29 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.3, 136.9, 134.2, 128.6, 127.6, 126.4, 123.8, 65.5, 40.4, 38.4, 20.4, 17.6. IR (KBr): 2964.76, 1725.58, 1695.93, 1449.57, 1318.34, 1274.86, 1224.99, 1025.95, 968.70, 911.56, 749.23, 693.95cm^{-1}. HRMS (ESI/[M+Na]$^+$) Calcd. for: $C_{16}H_{19}O_2$ 243.1385, found 243.1387.

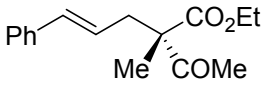
5-cinnamyl-2,2,5-trimethyl-1,3-dioxane-4,6-dione (3ag)



Following the general procedure, the reaction was conducted at 60 ºC in 0.3 mmol scale with **3ag** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 10:1) as yellow solid (45 mg, 55% yield). Mp: 56-58 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.28 (m, 4H), 7.26 – 7.21 (m, 1H), 6.52 (d, *J* = 15.8 Hz, 1H), 6.04 (dt, *J* = 15.6, 7.7 Hz, 1H), 2.91 (dd, *J* = 7.7, 1.0 Hz, 2H), 1.72 (s, 3H), 1.69 (s, 3H), 1.63 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.1, 136.5, 135.9, 128.7, 128.0, 126.5, 121.9, 105.3, 50.5, 43.4, 29.7, 29.0, 24.5. IR (KBr): 3027.44, 3000.07, 2940.77, 1744.54,

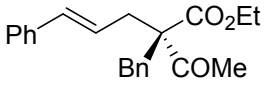
1598.34, 1451.85, 1380.77, 1277.90, 1203.61, 1144.03, 1050.06, 974.47, 944.07, 843.27. 742.92, 692.95, 625.26cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{19}O_4$ 275.1283, found 275.1278.

Ethyl (E)-2-acetyl-2-methyl-5-phenylpent-4-enoate (3ah)



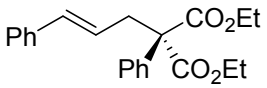
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ah** was isolated by PTLC (Petroleum ether (bp: 60-90 ^{o}C)/ethyl acetate = 10:1) as yellow oil (62 mg, 80% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.25 (m, 4H), 7.23 – 7.18 (m, 1H), 6.43 (d, *J* = 15.7 Hz, 1H), 6.05 (dt, *J* = 15.7, 7.6 Hz, 1H), 4.20 (qd, *J* = 7.1, 1.6 Hz, 2H), 2.79 (ddd, *J* = 14.1, 7.3, 1.3 Hz, 1H), 2.65 (ddd, *J* = 14.1, 7.7, 1.2 Hz, 1H), 2.18 (s, 3H), 1.38 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 205.3, 172.6, 137.1, 134.0, 128.6, 127.5, 126.3, 124.3, 61.5, 59.9, 38.7, 26.5, 19.3, 14.2. IR (KBr): 2982.89, 1711.50, 1597.12, 1448.24, 1275.19, 1260.94, 1095.89, 1020.97, 967.04, 858.58, , 764.20, 749.44, 693.00cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{21}O_3$ 261.1491, found 261.1482.

Ethyl (E)-2-acetyl-2-benzyl-5-phenylpent-4-enoate (3ai)



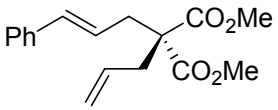
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ai** was isolated by PTLC (Petroleum ether (bp: 60-90 ^{o}C)/ethyl acetate = 10:1) as light yellow oil (89 mg, 88% yield). MP: 56-58^{o}C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 – 7.31 (m, 3H), 7.31 – 7.26 (m, 2H), 7.25 – 7.20 (m, 3H), 7.11 (dd, *J* = 7.9, 1.6 Hz, 2H), 6.45 (d, *J* = 15.7 Hz, 1H), 6.05 (dt, *J* = 15.6, 7.4 Hz, 1H), 4.29 – 4.08 (m, 2H), 3.32 – 3.17 (m, 2H), 2.71 (dd, *J* = 7.4, 1.4 Hz, 2H), 2.15 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.7, 171.7, 137.1, 136.3, 134.3, 130.1, 128.7, 128.5, 127.7, 127.1, 126.3, 123.9, 65.1, 61.6, 38.1, 35.6, 27.8, 14.2. IR (KBr): 3028.72, 2979.86, 2929.35, 1712.96, 1599.95, 1494.92, 1448.24, 1355.45, 1261.25, 1229.30, 1189.19, 1156.41, 1094.78, 1019.36, 966.85, 860.49, 746.12, 701.34cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{22}H_{25}O_3$ 337.1804, found 337.1808.

Diethyl 2-cinnamyl-2-phenylmalonate (3aj)



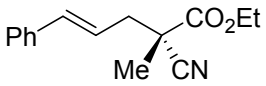
Following the general procedure, $Pd(PPh_3)_4$ (10 mol%) was used. The reaction was conducted at 60 ^{o}C in 0.3 mmol scale with **3aj** was isolated by PTLC (Petroleum ether (bp: 60-90 ^{o}C)/ethyl acetate = 9:1) as yellow oil (61 mg, 58% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.39 – 7.34 (m, 2H), 7.33 – 7.30 (m, 1H), 7.29 – 7.26 (m, 4H), 7.22 – 7.17 (m, 1H), 6.41 (d, *J* = 15.8 Hz, 1H), 6.18 (dt, *J* = 15.8, 7.3 Hz, 1H), 4.29 – 4.16 (m, 4H), 3.23 (dd, *J* = 7.3, 1.2 Hz, 2H), 1.23 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5, 137.3, 137.0, 133.9, 128.6, 128.3, 128.2, 127.7, 127.4, 126.3, 124.8, 63.2, 61.8, 39.8, 14.2. IR (KBr): 2979.62, 2318.16, 1730.63, 1698.37, 1627.53, 1446.88, 1367.63, 1292.12, 1256.97, 1241.79, 1200.16, 1142.38, 1125.37, 1062.79, 1009.79, 964.61, 941.00, 836.91, 825.47, 749.77, 693.28cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{22}H_{25}O_4$ 353.1753, found 353.1758.

Dimethyl 2-allyl-2-cinnamylmalonate (3ak)



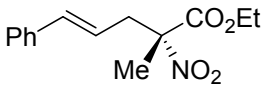
Following the general procedure, $Pd(PPh_3)_4$ (5 mol%) was used. The reaction was conducted at 60 °C in 0.3 mmol scale with **3ak** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 9:1) as yellow oil (56 mg, 65% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 (td, *J* = 8.2, 6.4 Hz, 4H), 7.25 – 7.19 (m, 1H), 6.44 (d, *J* = 15.7 Hz, 1H), 6.02 (dt, *J* = 15.4, 7.6 Hz, 1H), 5.69 (ddt, *J* = 18.8, 9.2, 7.3 Hz, 1H), 5.15 (d, *J* = 4.3 Hz, 1H), 5.12 (s, 1H), 3.73 (s, 6H), 2.80 (d, *J* = 6.7 Hz, 2H), 2.69 (d, *J* = 7.4 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.3, 137.1, 134.2, 132.3, 128.6, 127.6, 126.3, 123.9, 119.5, 58.1, 52.6, 37.3, 36.4. IR (KBr): 2952.98, 1731.92, 1508.31, 1490.07, 1435.86, 1300.57, 1251.83, 1211.98, 1132.56, 1067.69, 1029.52, 968.06, 924.67, 837.21, 745.74, 692.57cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{17}H_{21}O_4$ 289.1440, found 289.1446.

Ethyl (E)-2-cyano-2-methyl-5-phenylpent-4-enoate (3al)



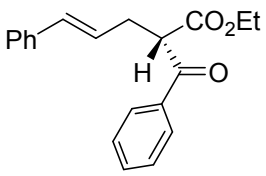
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3al** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 3:1) as yellow oil (61 mg, 84% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.35 (m, 2H), 7.31 (dd, *J* = 8.2, 6.6 Hz, 2H), 7.27 – 7.22 (m, 1H), 6.55 (d, *J* = 15.7 Hz, 1H), 6.19 (dt, *J* = 15.3, 7.5 Hz, 1H), 4.25 (qd, *J* = 7.1, 1.2 Hz, 2H), 2.82 (ddd, *J* = 13.8, 7.5, 0.9 Hz, 1H), 2.67 (ddd, *J* = 13.8, 7.4, 1.0 Hz, 1H), 1.62 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.0, 136.5, 135.8, 128.7, 128.1, 126.6, 121.8, 119.8, 63.0, 44.1, 41.6, 22.9, 14.2. IR (KBr): 3027.86, 2985.17, 2940.89, 2243.49, 1742.85, 1449.86, 1380.64, 1242.73, 1126.92, 1016.34, 969.30, 859.20, 745.10, 694.24cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{15}H_{18}NO_2$ 244.1338, found 244.1344.

Ethyl (E)-2-methyl-2-nitro-5-phenylpent-4-enoate (3am)



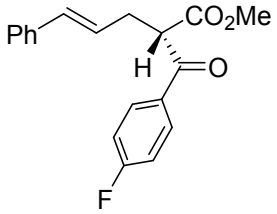
Following the general procedure, $Pd(PPh_3)_4$ (10 mol%) was used. The reaction was conducted at 60 °C in 0.3 mmol scale with **3am** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (73 mg, 92% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 – 7.27 (m, 4H), 7.29 – 7.18 (m, 1H), 6.51 (d, *J* = 15.7 Hz, 1H), 6.02 (dt, *J* = 15.5, 7.5 Hz, 1H), 4.27 (qd, *J* = 7.1, 1.3 Hz, 2H), 3.14 (dd, *J* = 14.3, 7.4 Hz, 1H), 3.02 (dd, *J* = 14.3, 7.7 Hz, 1H), 1.80 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.1, 136.4, 136.2, 128.6, 128.0, 126.4, 120.7, 92.3, 62.9, 40.3, 21.3, 13.9. IR (KBr): 2983.99, 2938.11, 1748.98, 1698.35, 1627.30, 1552.63, 1448.05, 1385.50, 1349.46, 1298.93, 1255.26, 1200.43, 1126.74, 1016.92, 970.04, 857.89, 836.93, 745.95, 692.93cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{14}H_{18}NO_4$ 264.1236, found 264.1239.

Ethyl (E)-2-benzoyl-5-phenylpent-4-enoate (3ao)



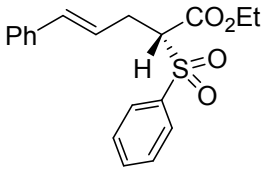
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ao** was isolated by PTLC (Petroleum ether (bp: 60-90 ℃)/ethyl acetate = 10:1) as light yellow solid (79 mg, 85% yield). Mp: 44-46 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (dd, *J* = 8.5, 1.3 Hz, 2H), 7.60 – 7.55 (m, 1H), 7.49 – 7.44 (m, 2H), 7.31 – 7.23 (m, 4H), 7.21 – 7.16 (m, 1H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.20 (dt, *J* = 15.7, 7.2 Hz, 1H), 4.46 (t, *J* = 7.2 Hz, 1H), 4.14 (qd, *J* = 7.1, 2.8 Hz, 2H), 2.91 (hd, *J* = 7.2, 1.3 Hz, 2H), 1.15 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 194.6, 169.5, 137.2, 136.2, 133.7, 132.8, 128.9, 128.8, 128.6, 127.5, 126.3, 126.2, 61.6, 54.4, 32.5, 14.2. IR (KBr): 2985.73, 1734.98, 1686.20, 1596.26, 1447.84, 1275.36, 1260.76, 965.94, 764.26, 749.65, 692.01cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{20}H_{21}O_3$ 309.1491, found 309.1494.

Methyl (E)-2-(4-fluorobenzoyl)-5-phenylpent-4-enoate (3ap)



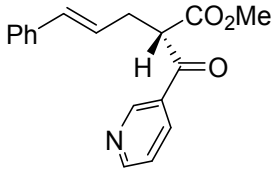
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ap** was isolated by PTLC (Petroleum ether (bp: 60-90 ℃)/ethyl acetate = 10:1) as yellow oil (70 mg, 75% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 8.01 (m, 2H), 7.31 – 7.24 (m, 4H), 7.22 – 7.17 (m, 1H), 7.17 – 7.12 (m, 2H), 6.47 (d, *J* = 15.8 Hz, 1H), 6.17 (dt, *J* = 15.7, 7.2 Hz, 1H), 4.45 (t, *J* = 7.2 Hz, 1H), 3.69 (s, 3H), 2.98 – 2.84 (m, 2H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -103.75. ^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.9, 169.8, 166.2 (d, $J_{C\text{-}F}$ = 256.1 Hz), 137.0, 133.0, 132.6 (d, *J* = 3.1 Hz), 131.5 (d, *J* = 9.5 Hz), 128.6, 127.6, 126.3, 125.9, 116.1 (d, *J* = 22.0 Hz), 54.1, 52.8, 32.5. IR (KBr): 3026.49, 2953.08, 1740.93, 1685.66, 1597.72, 1507.20, 1494.47, 1435.26, 1409.87, 1274.73, 1261.65, 1233.60, 1157.63, 1011.94, 966.61, 846.43, 748.88, 693.75, 583.73cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{19}H_{18}FO_3$ 313.1240, found 313.1244.

Ethyl (E)-5-phenyl-2-(phenylsulfonyl)pent-4-enoate (3aq)



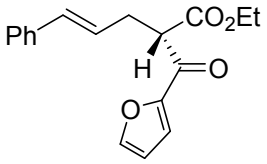
Following the general procedure, $Pd(PPh_3)_4$ (10 mol%) was used. The reaction was conducted at 60 ℃ in 0.3 mmol scale with **3aq** was isolated by PTLC (Petroleum ether (bp: 60-90 ℃)/ethyl acetate = 4:1) as yellow oil (54 mg, 52% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.93 – 7.90 (m, 2H), 7.72 – 7.68 (m, 1H), 7.58 (ddd, *J* = 8.0, 6.8, 1.1 Hz, 2H), 7.28 (d, *J* = 4.3 Hz, 4H), 7.25 – 7.19 (m, 1H), 6.46 (d, *J* = 15.8 Hz, 1H), 6.01 (ddd, *J* = 15.7, 7.8, 6.6 Hz, 1H), 4.12 – 4.05 (m, 3H), 2.97 (dddd, *J* = 13.9, 6.5, 3.9, 1.5 Hz, 1H), 2.86 (dddd, *J* = 14.0, 11.3, 7.8, 1.2 Hz, 1H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 165.4, 137.1, 136.6, 134.6, 134.4, 129.5, 129.3, 128.7, 127.9, 126.4, 122.8 , 70.3, 62.4, 30.4, 14.0. IR (KBr): 2981.50, 2934.09, 1737.24, 1598.93, 1584.04, 1493.88, 1447.51, 1369.21, 1325.23, 1256.95, 1230.67, 1197.24, 1146.72, 1083.29, 1028.26, 968.39, 853.55, 819.45, 743.76, 724.24, 690.20cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{19}H_{21}SO_4$ 345.1161, found 345.1167.

Methyl (E)-2-nicotinoyl-5-phenylpent-4-enoate (3ar)



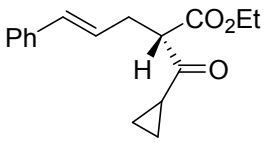
Following the general procedure, the reaction was conducted at 60 °C in 0.3 mmol scale with **3ar** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1:1) as yellow oil (84 mg, 95% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.23 (s, 1H), 8.80 (d, *J* = 3.6 Hz, 1H), 8.27 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.46 – 7.41 (m, 1H), 7.31 – 7.25 (m, 4H), 7.20 (ddd, *J* = 7.2, 5.4, 2.7 Hz, 1H), 6.48 (d, *J* = 15.8 Hz, 1H), 6.17 (dt, *J* = 15.7, 7.2 Hz, 1H), 4.48 (t, *J* = 7.2 Hz, 1H), 3.70 (s, 3H), 2.93 (tdd, *J* = 7.2, 3.7, 1.3 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 193.6, 169.3, 154.1, 150.1, 136.9, 136.1, 133.2, 131.5, 128.6, 127.6, 126.3, 125.5, 123.9, 54.4, 52.9, 32.2. IR (KBr): 3026.77, 2952.81, 1742.65, 1691.09, 1584.80, 1494.02, 1435.06, 1419.27, 1236.22, 1162.53, 967.21, 815.85, 747.09, 700.31, 620.75cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{18}NO_3$ 296.1287, found 296.1291.

Ethyl (E)-2-(furan-2-carbonyl)-5-phenylpent-4-enoate (3as)



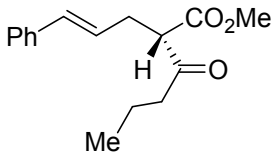
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3as** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 10:1) as light yellow solid (83 mg, 93% yield). Mp: 47-49 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61 (dd, *J* = 1.7, 0.7 Hz, 1H), 7.32 – 7.24 (m, 5H), 7.21 – 7.16 (m, 1H), 6.55 (dd, *J* = 3.6, 1.7 Hz, 1H), 6.47 (d, *J* = 15.8 Hz, 1H), 6.18 (dt, *J* = 15.7, 7.2 Hz, 1H), 4.25 (t, *J* = 7.3 Hz, 1H), 4.16 (qd, *J* = 7.1, 1.0 Hz, 2H), 2.89 (tdd, *J* = 7.2, 2.3, 1.4 Hz, 2H), 1.19 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.3, 169.1, 152.0, 147.2, 137.1, 132.8, 128.6 , 127.5, 126.3, 126.0, 118.8, 112.8, 61.7, 54.5, 32.0, 14.2. IR (KBr): 2981.55, 2929.60, 1738.52, 1680.43, 1567.13, 1465.09, 1392.19, 1252.39, 1161.87, 1085.27, 1028.29, 966.26, 883.01, 746.74, 694.13cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{18}H_{19}O_4$ 299.1283, found 299.1284.

Ethyl (E)-2-(cyclopropanecarbonyl)-5-phenylpent-4-enoate (3at)



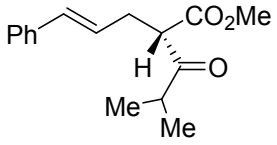
Following the general procedure, the reaction was conducted at 60 °C in 0.3 mmol scale with **3at** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 10:1) as yellow oil (64 mg, 78% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.25 (m, 4H), 7.20 (t, *J* = 6.9 Hz, 1H), 6.46 (d, *J* = 15.8 Hz, 1H), 6.14 (dt, *J* = 15.8, 7.8 Hz, 1H), 4.21 (qq, *J* = 6.7, 3.7 Hz, 2H), 3.74 (t, *J* = 7.4 Hz, 1H), 2.78 (t, *J* = 7.2 Hz, 2H), 2.09 (tt, *J* = 7.9, 4.5 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H), 1.09 (t, *J* = 3.9 Hz, 2H), 0.96 – 0.91 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.8, 169.4, 137.2, 132.7, 128.6, 127.4, 126.2, 126.1, 61.5, 59.9, 31.7, 20.1, 14.3, 12.1, 11.8. IR (KBr): 2981.96, 2936.17, 1737.48, 1702.57, 1494.06, 1447.74, 1382.45, 1260.42, 1159.21, 1075.13, 1028.43, 965.97, 746.35, 693.97cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{17}H_{21}O_3$ 273.1491, found 273.1497.

Methyl 2-cinnamyl-3-oxohexanoate (3au)



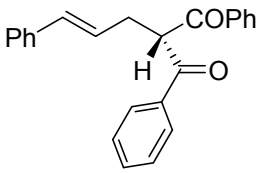
Following the general procedure, the reaction was conducted at in 0.3 mmol scale with **3au** was isolated by PTLC (Petroleum ether (bp: 60-90 ℃)/ethyl acetate = 2:1) as yellow oil (55 mg, 70% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.26 (m, 4H), 7.23 – 7.19 (m, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.10 (dt, *J* = 15.8, 7.2 Hz, 1H), 3.73 (s, 3H), 3.62 (t, *J* = 7.4 Hz, 1H), 2.75 (td, *J* = 7.3, 1.3 Hz, 2H), 2.62 – 2.43 (m, 2H), 1.62 (h, *J* = 7.3 Hz, 2H), 0.90 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 204.7, 169.8, 137.1, 132.8, 128.6, 127.5, 126.2, 125.9, 58.8, 52.6, 44.3, 31.7, 17.0, 13.6. IR (KBr): 2961.93, 1745.28, 1715.07, 1494.01, 1435.14, 1260.89, 1206.49, 1163.53, 967.12, 747.32, 693.88cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{21}O_3$ 261.1491, found 261.1488.

Methyl (E)-2-isobutyryl-5-phenylpent-4-enoate (3av)



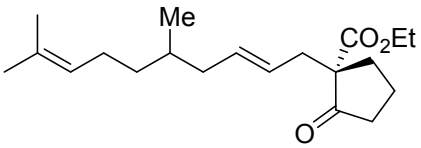
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3av** was isolated by PTLC (Petroleum ether (bp: 60-90 ℃)/ethyl acetate = 10:1) as yellow oil (63 mg, 81% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 – 7.23 (m, 4H), 7.20 (ddt, *J* = 8.4, 5.7, 2.0 Hz, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.10 (dt, *J* = 15.7, 7.3 Hz, 1H), 3.80 (t, *J* = 7.3 Hz, 1H), 3.71 (s, 3H), 2.86 – 2.76 (m, 1H), 2.74 (t, *J* = 7.4 Hz, 2H), 1.12 (d, *J* = 6.8 Hz, 3H), 1.09 (d, *J* = 7.0 Hz, 3H).^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.5, 169.8, 137.1, 132.8, 128.6, 127.5, 126.2, 126.0, 56.7, 52.6, 41.1, 32.0, 18.3, 18.0. IR (KBr): 2978.80, 2318.52, 1734.81, 1493.98, 1448.29, 1275.34, 1260.74, 1027.00, 966.08, 764.17, 749.41, 692.90cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{16}H_{21}O_3$ 261.1491, found 261.1499.

2-cinnamyl-1,3-diphenylpropane-1,3-dione (3aw)



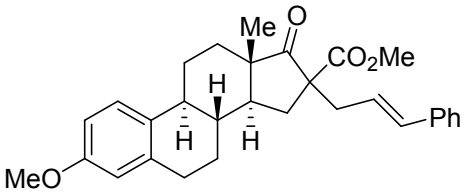
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3aw** was isolated by PTLC (Petroleum ether (bp: 60-90 ℃)/ethyl acetate = 10:1) as light yellow solid (88 mg, 86% yield). Mp: 76-78 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.2 Hz, 4H), 7.55 (t, *J* = 7.4 Hz, 2H), 7.43 (t, *J* = 7.7 Hz, 4H), 7.25 (s, 2H), 7.24 (d, *J* = 2.3 Hz, 2H), 7.20 – 7.14 (m, 1H), 6.45 (d, *J* = 15.8 Hz, 1H), 6.25 (dt, *J* = 15.7, 7.2 Hz, 1H), 5.38 (t, *J* = 6.7 Hz, 1H), 3.01 (td, *J* = 7.1, 1.1 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 195.7, 137.1, 136.0, 133.8, 132.6, 129.1, 128.8, 128.6, 127.5, 126.9, 126.3, 57.1, 33.1. IR (KBr): 3059.20, 3026.03, 1695.56, 1671.58, 1595.91, 1579.66, 1492.98, 1447.74, 1329.54, 1262.98, 1231.12, 1197.90, 1180.92, 1000.52, 966.06, 939.38, 748.26, 691.96, 598.29cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{24}H_{21}O_2$ 341.1542, found 341.1547.

Ethyl 1-((E)-5,9-dimethyldeca-2,8-dien-1-yl)-2-oxocyclopentane-1-carboxylate (3ax)



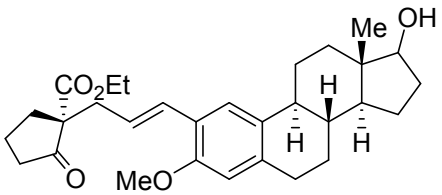
Following the general procedure, $Pd(PPh_3)_4$ (10 mol%) was used. The reaction was conducted at 60 °C in 0.3 mmol scale with **3ax** was isolated by flash column chromatography (petroleum ether/ethyl acetate = 50/1~30/1). as yellow oil (78 mg, 81% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 5.48 (dt, *J* = 14.6, 7.1 Hz, 1H), 5.27 (dt, *J* = 15.1, 7.3 Hz, 1H), 5.08 (t, *J* = 7.1 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.61 (dd, *J* = 13.9, 7.1 Hz, 1H), 2.49 – 2.28 (m,3H), 2.28 – 2.17 (m, 1H), 2.05 – 1.88 (m, 6H), 1.82 (dt, *J* = 15.0, 7.0 Hz, 1H), 1.68 (s, 3H), 1.60 (s, 3H), 1.45 (dq, *J* = 13.2, 6.6 Hz, 1H), 1.36 – 1.27 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.11 (td, *J* = 13.9, 8.5 Hz, 1H), 0.84 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.9, 171.0, 134.0, 131.1, 125.3, 124.8, 61.4, 60.3, 39.9, 38.2, 36.8, 36.6, 32.6, 32.0, 25.7, 25.5, 19.5, 19.4, 17.6, 14.1. IR (KBr): 2963.87, 2912.50, 1752.89, 1726.05, 1447.91, 1405.75, 1376.90, 1314.77, 1277.85, 1223.56, 1147.57, 1029.35, 973.66, 921.56, 860.09cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{20}H_{33}O_3$ 321.2430, found 321.2437.

Methyl-(8R,9S,13S,14S)-16-cinnamyl-3-methoxy-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthrene-16-carboxylate (3ay)



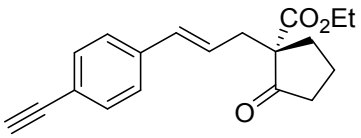
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3ay** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (122 mg, 89% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.30 (dt, *J* = 15.2, 7.5 Hz, 4H), 7.21 (d, *J* = 6.1 Hz, 1H), 7.17 (d, *J* = 8.7 Hz, 1H), 6.74 – 6.68 (m, 1H), 6.63 (s, 1H), 6.46 (d, *J* = 15.7 Hz, 1H), 6.06 (dt, *J* = 15.2, 7.3 Hz, 1H), 3.76 (s, 3H), 3.74 (s, 3H), 2.94 (dt, *J* = 15.9, 7.9 Hz, 1H), 2.87 (d, *J* = 6.3 Hz, 2H), 2.48 (dd, *J* = 13.8, 8.0 Hz, 1H), 2.39 (t, *J* = 13.1 Hz, 2H), 2.18 (td, *J* = 16.1, 13.1, 7.7 Hz, 2H), 1.97 (d, *J* = 8.9 Hz, 2H), 1.65 – 1.56 (m, 1H), 1.55 – 1.45 (m, 3H), 1.43 – 1.32 (m, 1H), 0.98 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 213.8, 171.5, 157.8, 137.8, 137.1, 134.2, 131.9, 128.7, 127.7, 126.4, 126.3, 124.9, 114.0, 111.8, 60.3, 55.3, 53.0, 49.7, 46.0, 44.2, 38.9, 37.9, 32.3, 30.4, 29.7, 26.7, 25.9, 14.3. IR (KBr): 2932.00, 2858.83, 2253.48, 1749.90, 1723.65, 1609.05, 1575.82, 1499.24, 1452.09, 1434.56, 1376.28, 1280.95, 1238.40, 1177.91, 1130.01, 1103.61, 1039.05, 1015.36, 983.90, 969.16, 911.36, 876.50, 810.17, 742.45, 693.65cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{30}H_{35}O_4$ 459.2535, found 459.2539.

Ethyl-1-((E)-3-((8R,9S,13S,14S)-17-hydroxy-3-methoxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-2-yl)allyl)-2-oxocyclopentane-1-carboxylate (3az)



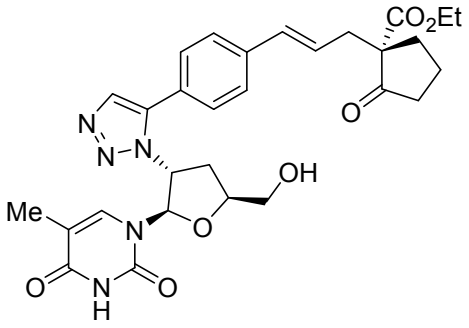
Following the general procedure, the reaction was conducted in 0.3 mmol scale with **3az** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 4:1) as yellow oil (72 mg, 50% yield). ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.28 (s, 1H), 6.71 (d, *J* = 15.9 Hz, 1H), 6.56 (s, 1H), 6.01 (dt, *J* = 15.7, 7.5 Hz, 1H), 4.19 (qt, *J* = 7.1, 3.8 Hz, 2H), 3.79 (s, 3H), 3.72 (t, *J* = 8.5 Hz, 1H), 2.87 – 2.78 (m, 3H), 2.57 – 2.39 (m, 3H), 2.36 – 2.29 (m, 1H), 2.24 (dd, *J* = 18.9, 8.2 Hz, 1H), 2.19 – 1.99 (m, 4H), 1.99 – 1.84 (m, 4H), 1.68 (ddd, *J* = 11.9, 9.5, 5.2 Hz, 1H), 1.55 – 1.30 (m, 6H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.18 (td, *J* = 11.7, 7.2 Hz, 1H), 0.77 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.3, 171.2, 154.5 , 137.3, 132.4, 129.3, 124.0, 123.7, 123.5, 111.3, 81.9, 61.6, 60.5, 55.6, 50.1, 44.0, 43.3, 39.0, 38.3, 37.7, 36.8, 32.2, 30.6, 29.9, 27.3, 26.5, 23.2, 19.7, 14.2, 11.2. IR (KBr): 2930.28, 1722.29, 1639.33, 1608.78, 1584.26, 1495.48, 1438.58, 1340.67, 1301.83, 1252.96, 1131.76, 1067.74, 1028.68, 959.10, 912.45, 837.40, 787.66, 744.82, 689.73cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{30}H_{41}O_5$ 481.2954, found 481.2951.

Ethyl (E)-1-(3-(4-ethynylphenyl)allyl)-2-oxocyclopentane-1-carboxylate (4)



The 3**z** (147 mg, 0.4 mmol) was dissolved in anhydrous THF (4 mL) in a 10 mL Schlenk flask. Then, H_2O (288 mg, 16.0 mmol) and TBAF (0.8 mL, 0.8 mmol, 1 M in THF) were added into the solution slowly at 0 ºC (low temperature magnetic stirrer, with ethylene glycol bath), and the mixture was stirred for 2 h. The reaction mixture was poured into water (5 mL) and extracted with EtOAc (10 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated under reduced pressure to give the crude product. The product 4 was isolated from the crude mixture by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 9:1) as yellow oil (78 mg, 88% yield). ^{1}H NMR (401 MHz, Chloroform-d) δ 7.42 (d, J = 8.3 Hz, 2H), 7.28 (d, *J* = 7.6 Hz, 2H), 6.43 (d, *J* = 15.8 Hz, 1H), 6.18 – 6.09 (dt, *J* = 15.3, 7.5 Hz, 1H), 4.18 (qd, *J* = 7.1, 1.4 Hz, 2H), 3.11 (s, 1H), 2.83 (ddd, *J* = 14.0, 7.3, 1.3 Hz, 1H), 2.56 – 2.41 (m, 3H), 2.31 – 2.22 (m, 1H), 2.10 – 1.98 (m, 2H), 1.97 – 1.88 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 214.8, 171.0, 137.5, 133.4, 132.4, 126.2, 126.1, 121.0, 83.7, 77.8, 61.7, 60.3, 38.2, 37.1, 32.5, 19.7, 14.3. IR (KBr): 3286.72, 2926.90, 1748.85, 1722.94, 1604.22, 1505.32, 1447.17, 1366.87, 1257.99, 1222.43, 1143.53, 1026.07, 971.04, 837.20, 749.11cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{19}H_{21}O_3$ 297.1491, found 297.1494.

Ethyl-1-((E)-3-(4-(1-((2R,3R,5S)-5-(hydroxymethyl)-2-(5-methyl-2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)tetrahydrofuran-3-yl)-1H-1,2,3-triazol-5-yl)phenyl)allyl)-2-oxocyclopentane-1-carboxylate (5)



Under argon atmosphere, a flame-dried 10 mL schlenk tube was charged with compound 4 (0.3 mmol, 1.0 equiv.), zidovudine (0.33 mmol, 1.1 equiv.), ^{t}BuOH (2.0 mL) and a stir bar was added a freshly prepared solution of $CuSO_4 \cdot 5H_2O$ (0.5 equiv.) and sodium ascorbate (0.5 equiv.) in H_2O (1 mL). the resulting solution was stirred at room temperature for 12 h. The reaction mixture was concentrated and subjected to PTLC (ethanol/ dichloromethane = 1:20) to give 5 as a white solid (144 mg, 85% yield). Mp: 143-145 ºC. ^{1}H NMR (401 MHz, DMSO-d6) δ 11.36 (s, 1H), 8.77 (s, 1H), 7.83 (s, 1H), 7.77 (d, *J* = 7.6 Hz, 2H), 7.44 (d, *J* = 7.7 Hz, 2H), 6.46 (d, *J* = 15.7 Hz, 1H), 6.42 (d, *J* = 6.6 Hz, 1H), 6.16 (dt, *J* = 15.0, 7.1 Hz, 1H), 5.41 – 5.32 (m, 2H), 4.24 (s, 1H), 4.07 (q, J = 6.8 Hz, 2H), 3.67 (q, *J* = 11.8 Hz, 2H), 2.77 (dt, *J* = 11.6, 5.4 Hz, 1H), 2.67 (dd, *J* = 13.9, 7.0 Hz, 2H), 2.44 – 2.39 (m, 1H), 2.37 – 2.24 (m, 3H), 1.98 (dt, *J* = 13.3, 7.3 Hz, 1H), 1.92 – 1.83 (m, 2H), 1.78 (s, 3H), 1.13 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 214.7, 171.0, 164.3, 151.0, 146.9, 136.9, 133.5, 130.0, 127.2, 125.9, 125.7, 121.6, 110.2, 85.0, 84.4, 61.5, 61.3, 60.3, 59.9, 38.0, 37.7, 36.9, 32.3, 19.7, 14.5, 12.8. IR (KBr): 2926.51, 2854.69, 2251.62, 2124.94, 1714.53, 1454.27, 1369.44, 1300.02, 1256.76, 1131.38, 1058.42, 1027.74, 1008.31, 972.46, 819.70, 757.14, 692.35cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{29}H_{34}N_5O_7$ 564.2458, found 564.2462.

4. References

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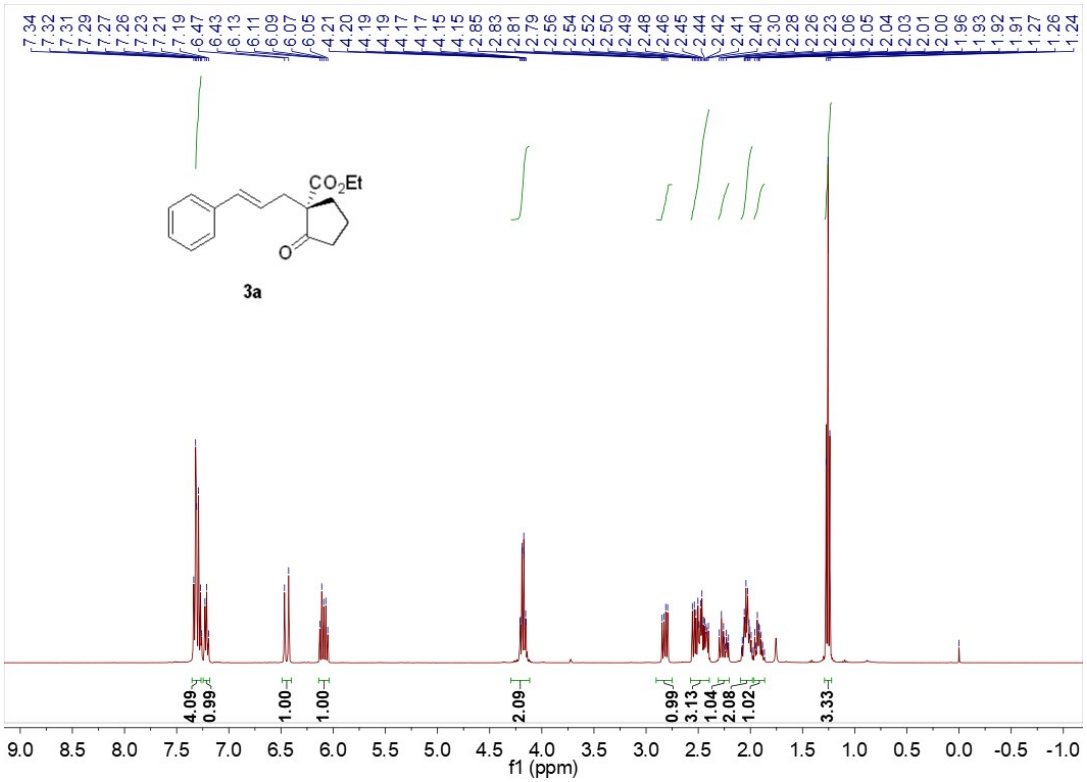
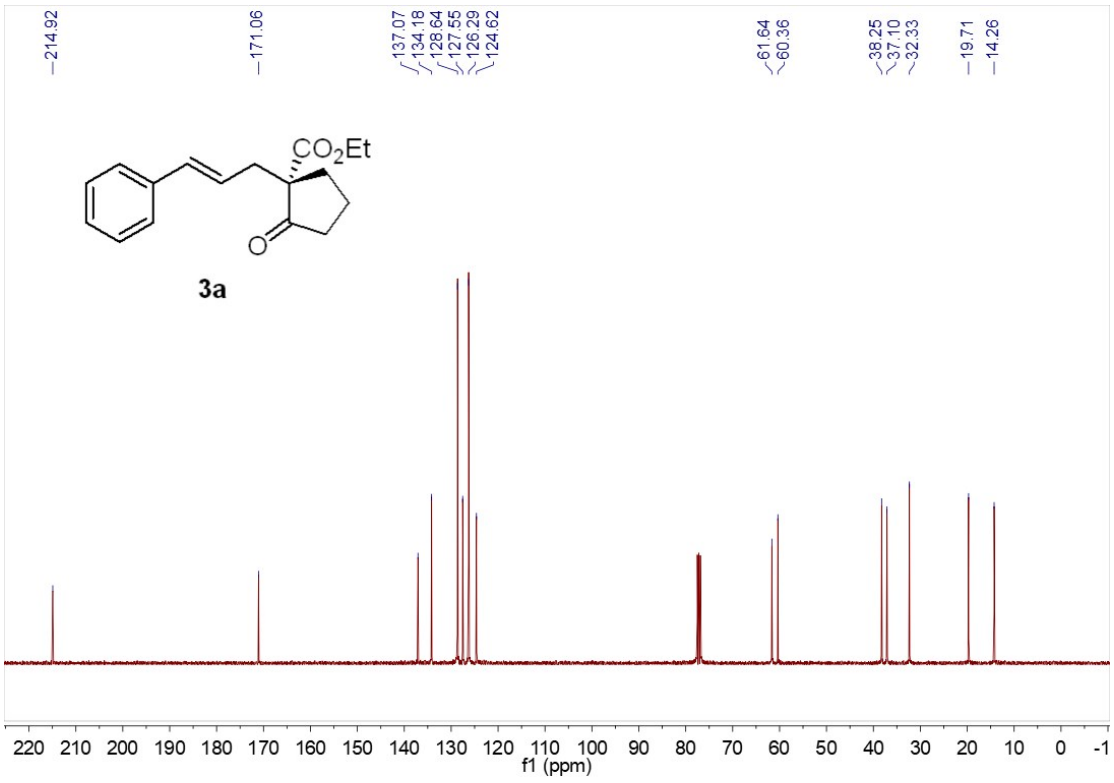
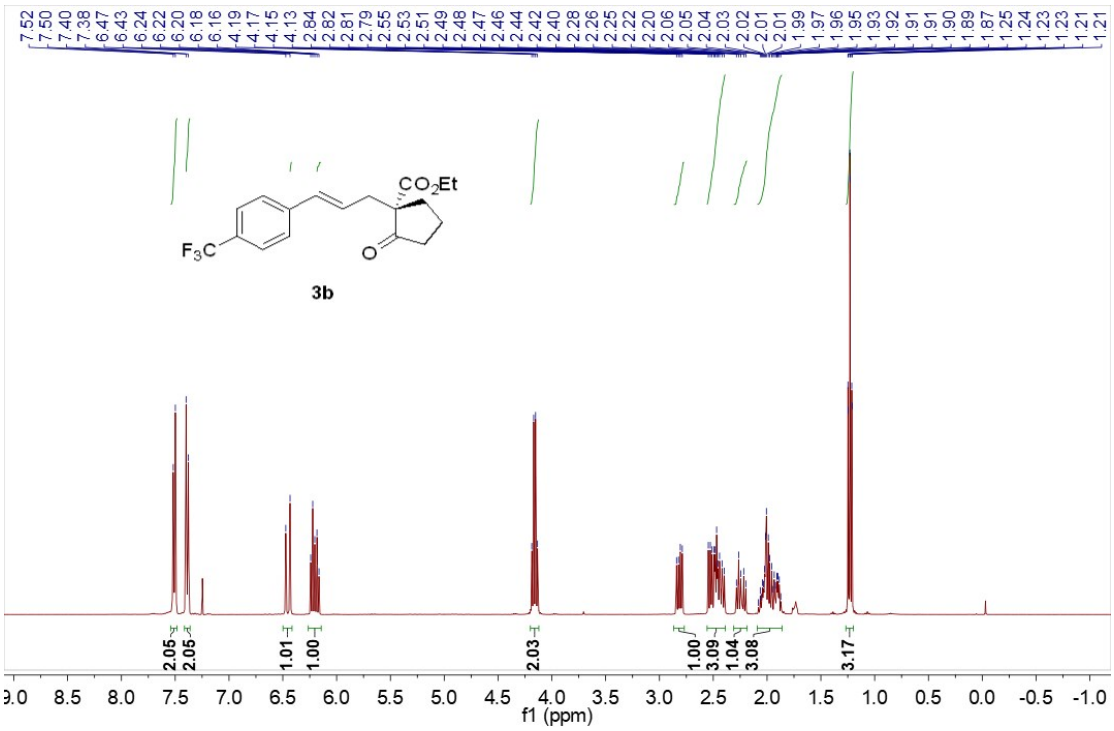
5. NMR Spectra for New Compounds



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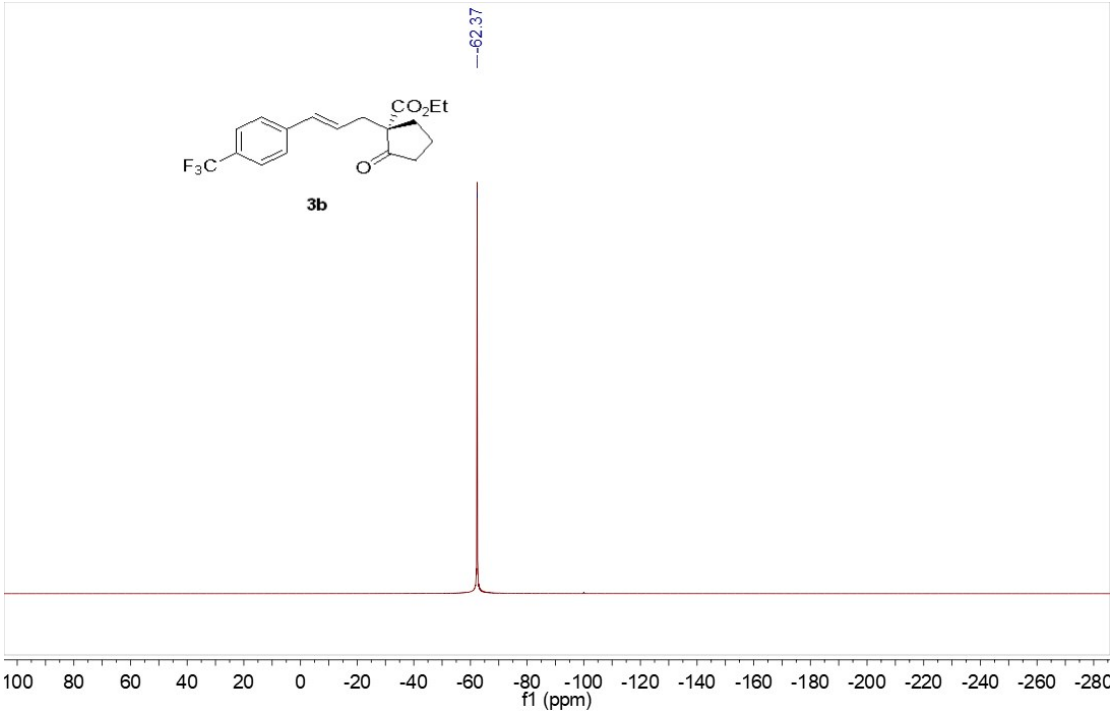
CO2Et
F3C
O
3b
2.05
2.05
1.01
1.00
2.03
1.00
3.09
1.04
3.08
3.17
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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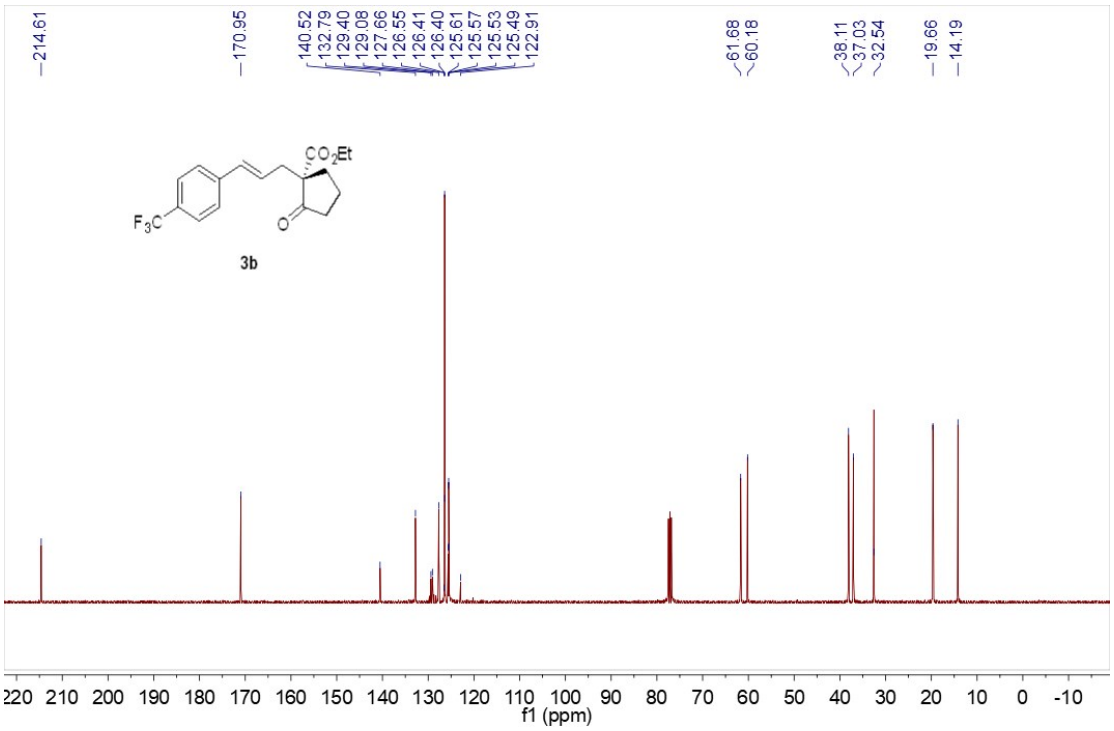
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125.53
125.49
122.91
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37.03
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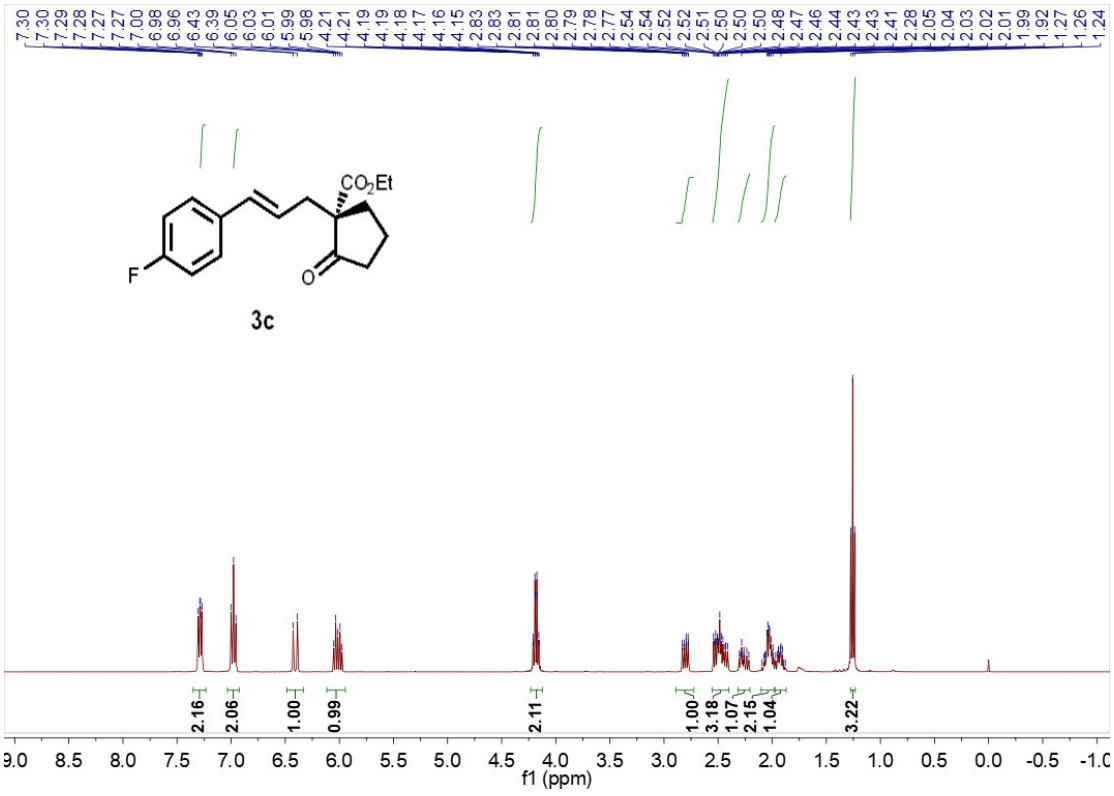
CO2Et
F3C
O
3b
f1 (ppm)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F
O
3c
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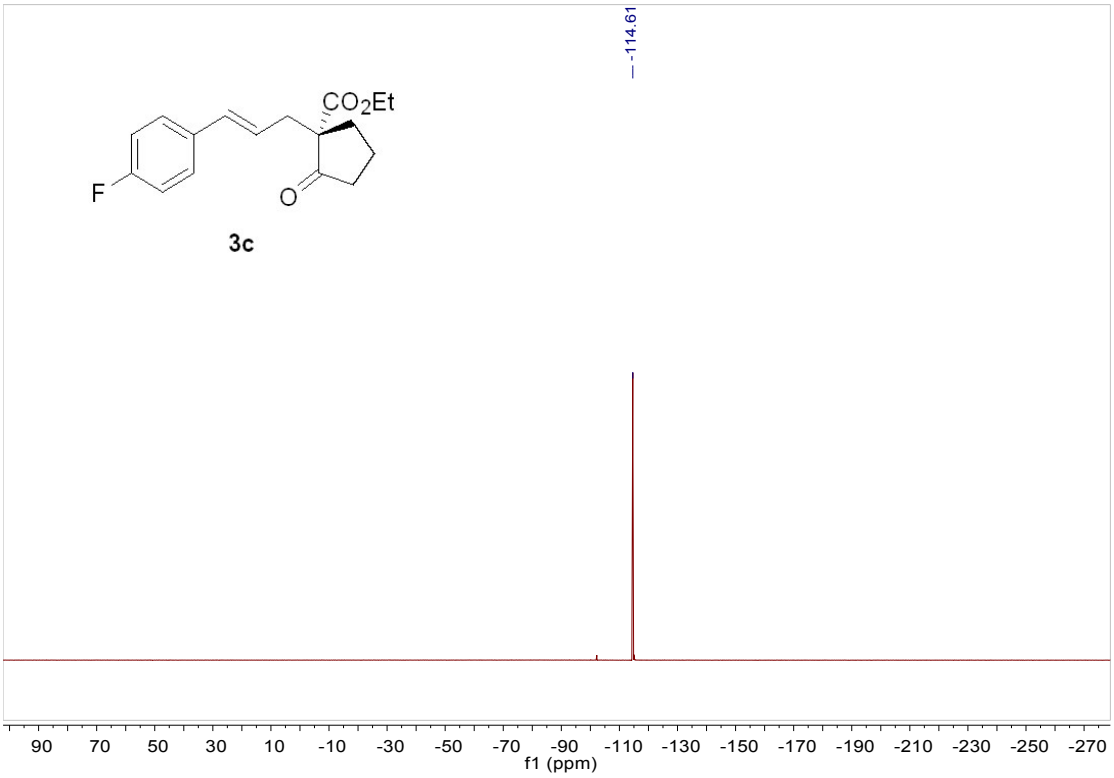
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F
O
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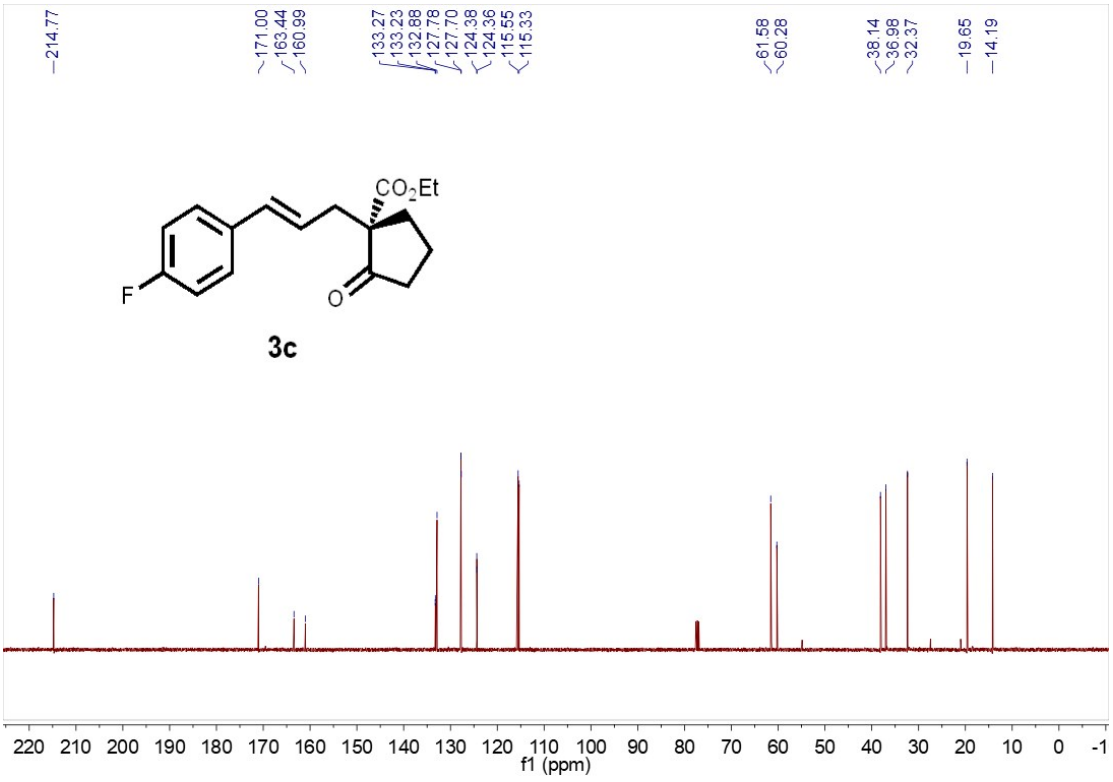
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124.36
115.55
115.33
61.58
60.28
38.14
36.98
32.37
19.65
14.19
CO2Et
F
O
3c
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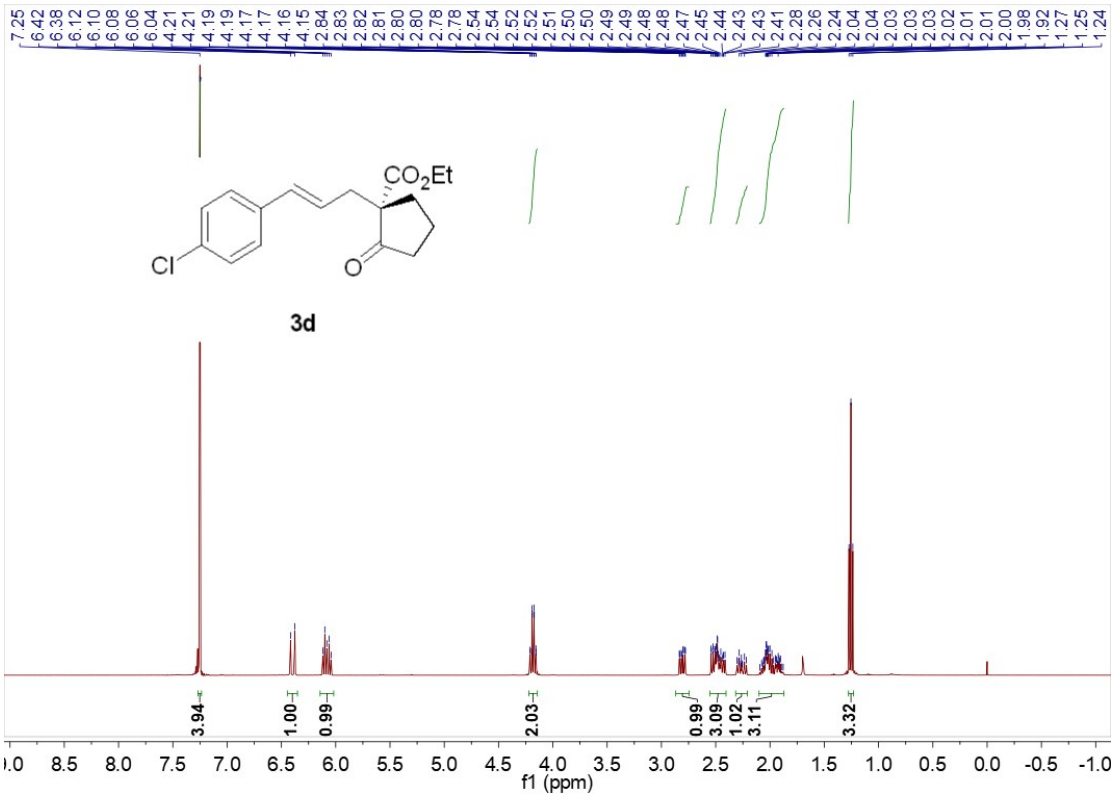
f1 (ppm)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CO2Et
Cl
O
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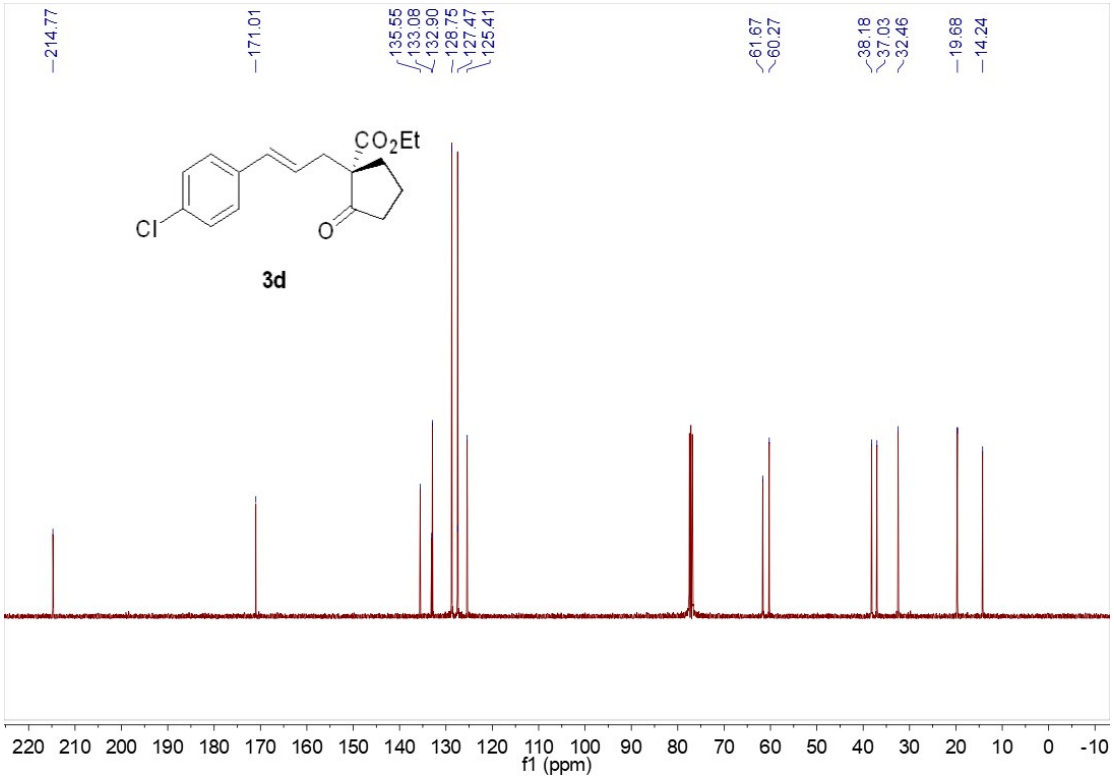
0.99
2.03
0.99
3.09
1.02
3.11
3.32
f1 (ppm)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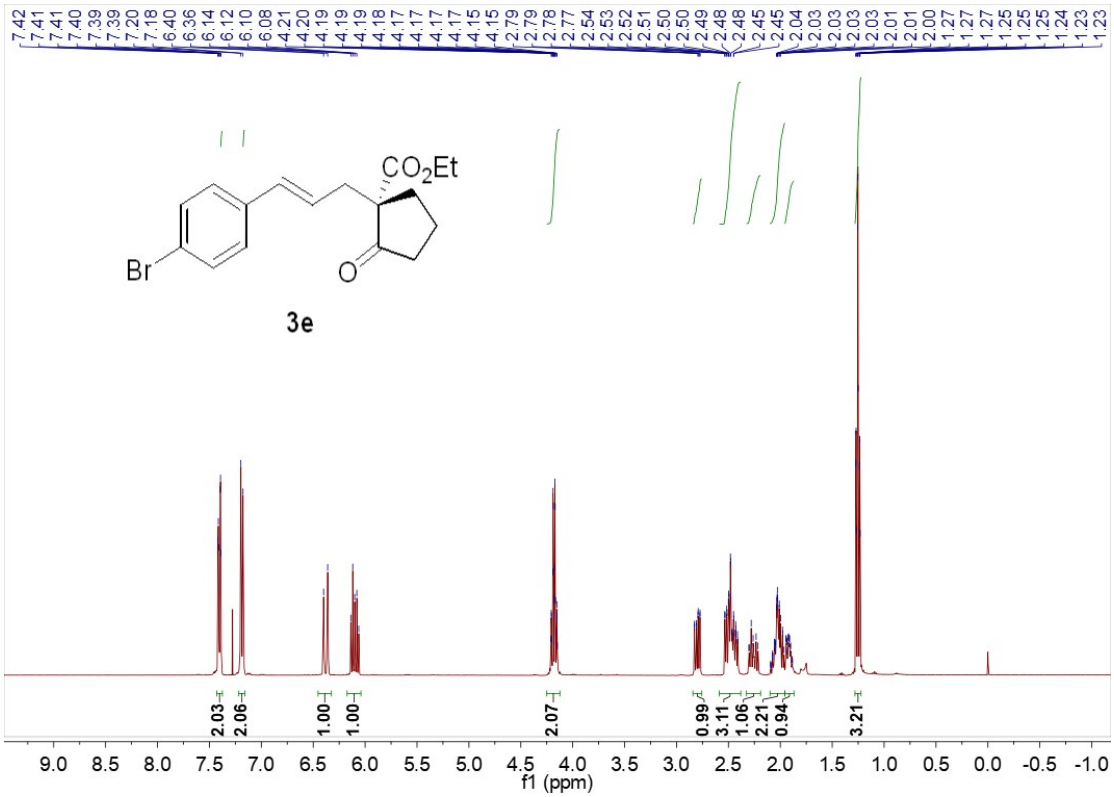
14.24
CO2Et
Cl
O
3d
f1 (ppm)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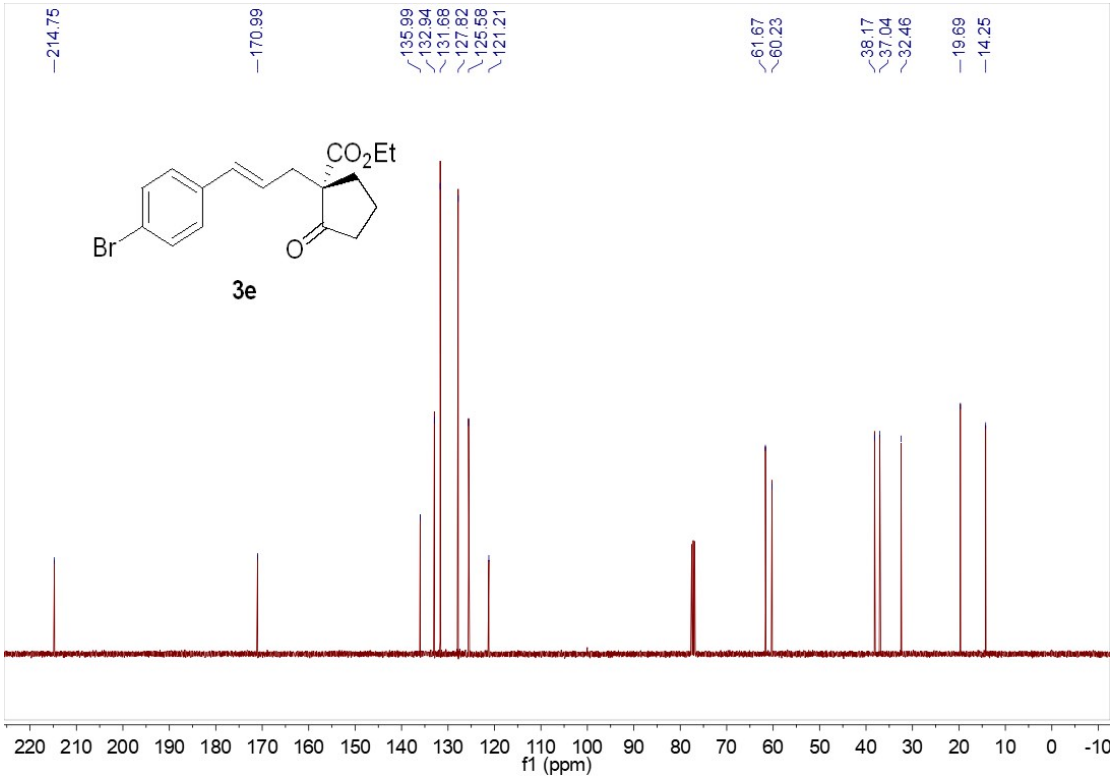
Br
O
3e
f1 (ppm)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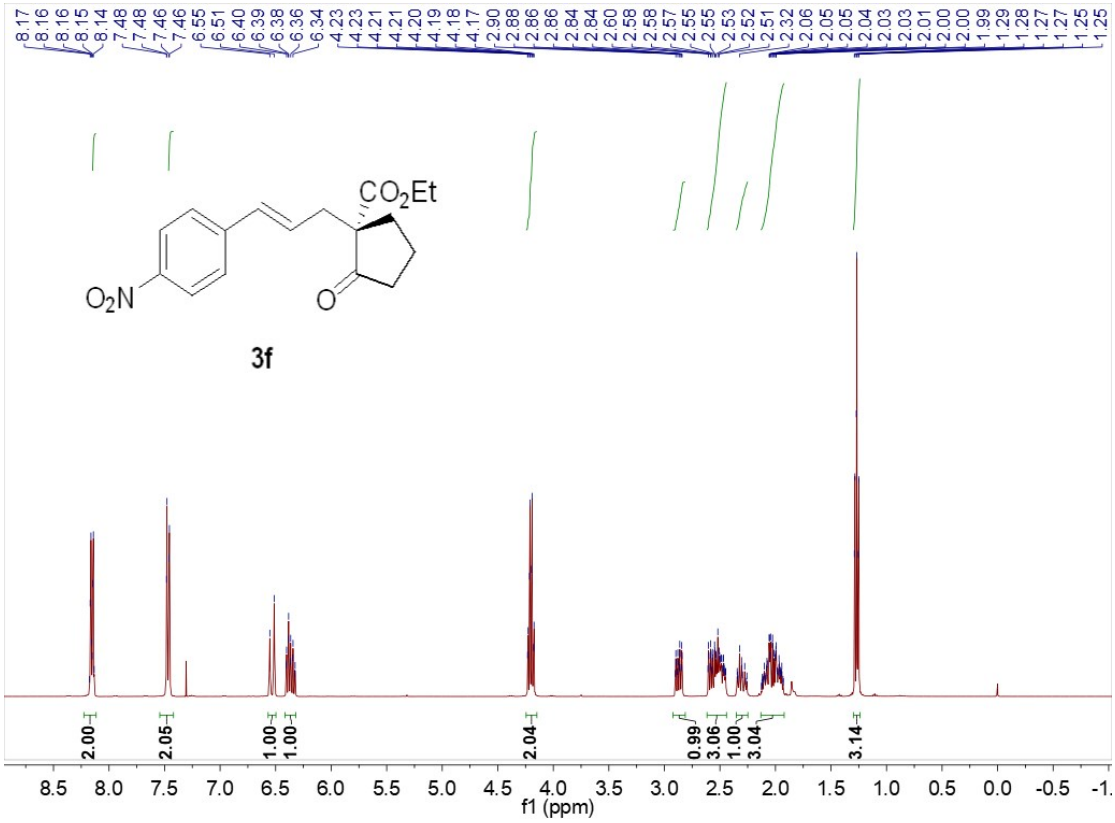
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CO2Et
O2N
O
3f
2.00 2.05 1.00 1.00 2.04 0.99 3.06 1.00 3.04 3.14
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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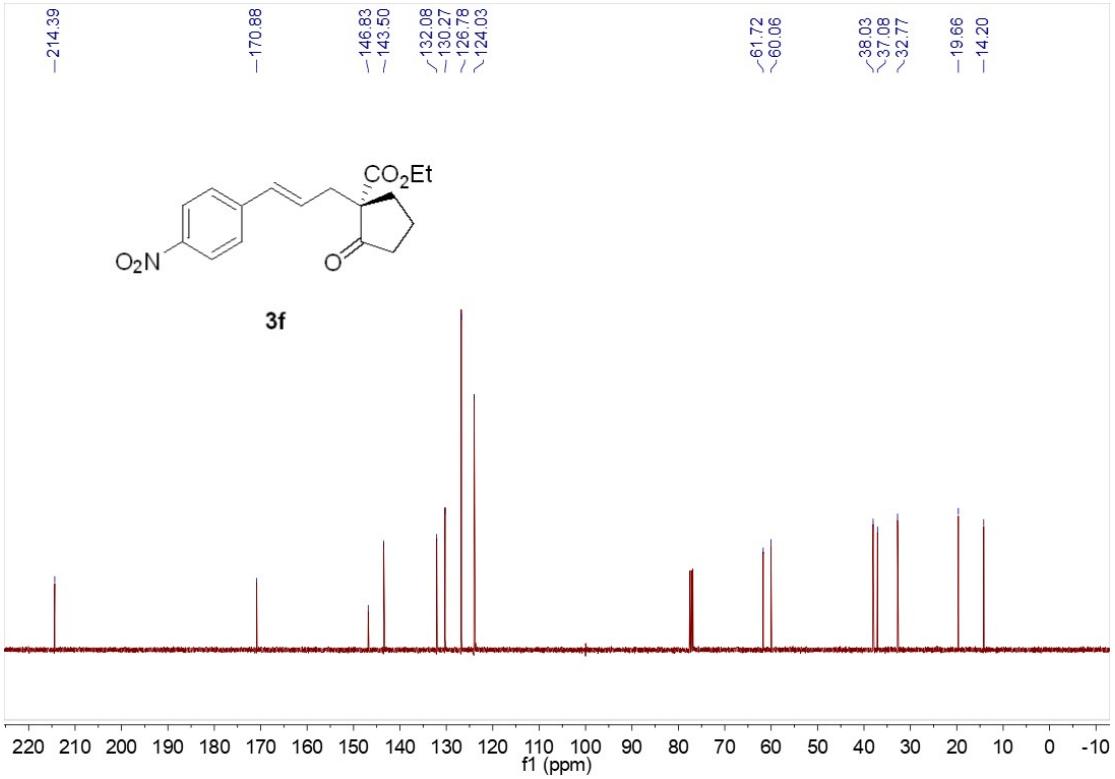
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3f
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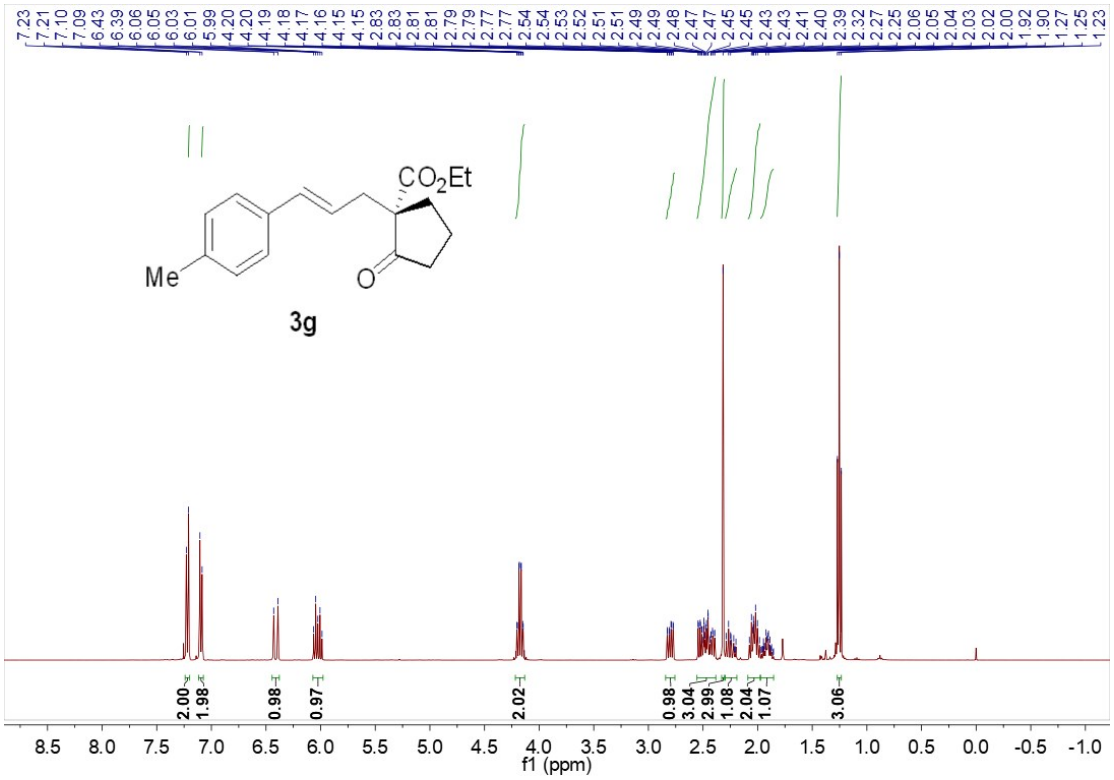
f1 (ppm)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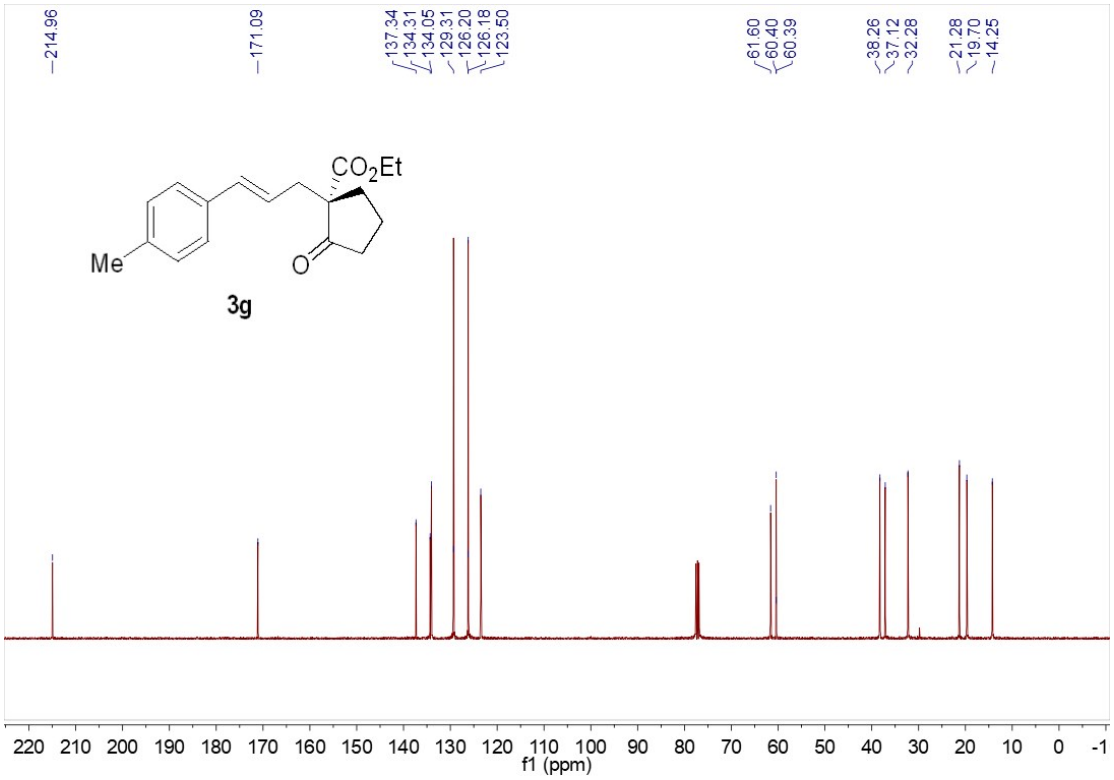
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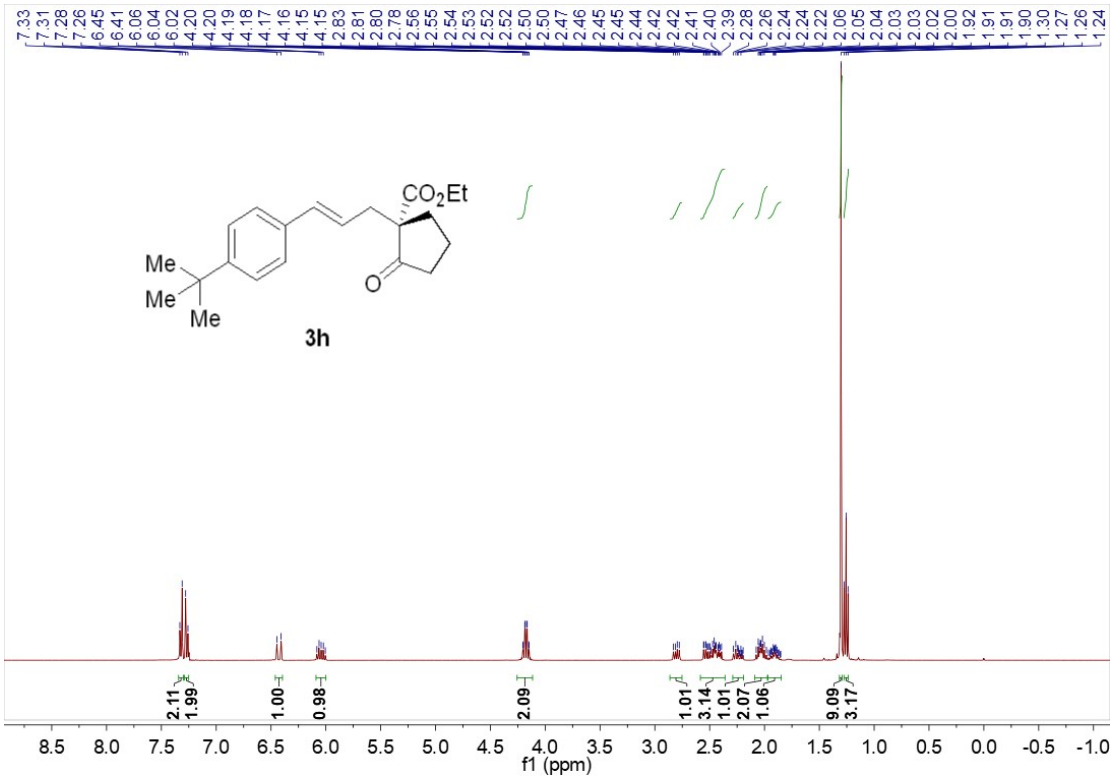
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Me
Me
O
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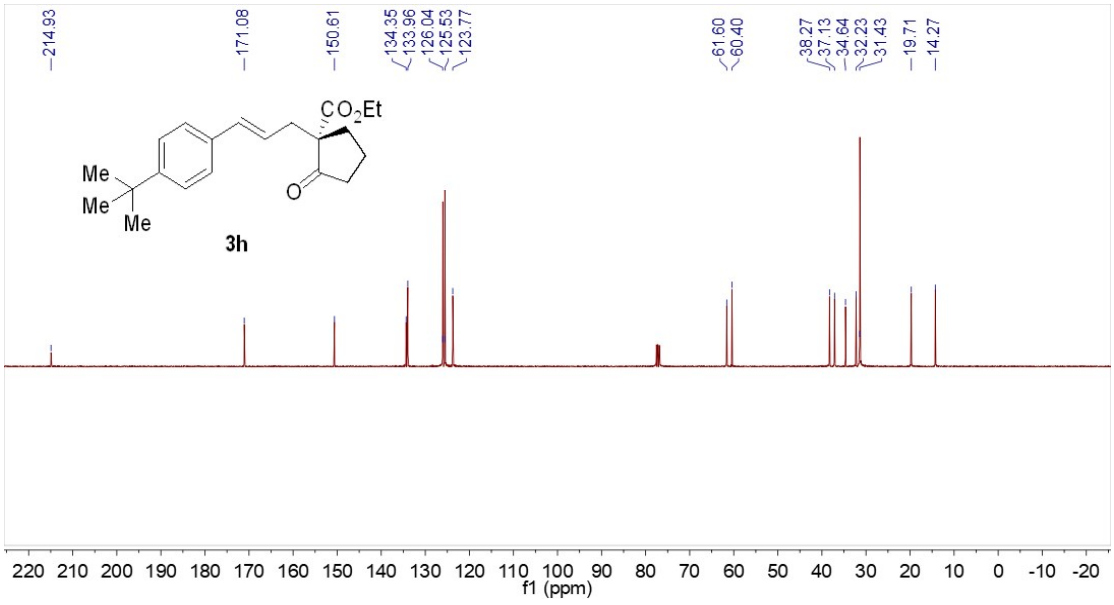
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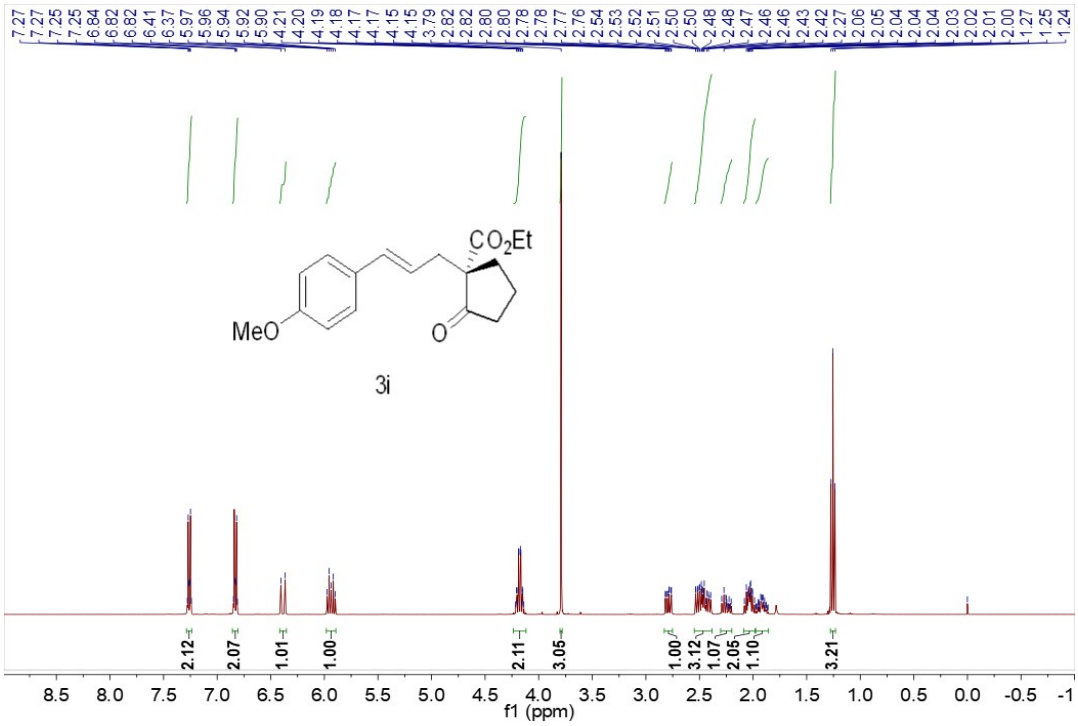
O
3h
f1 (ppm)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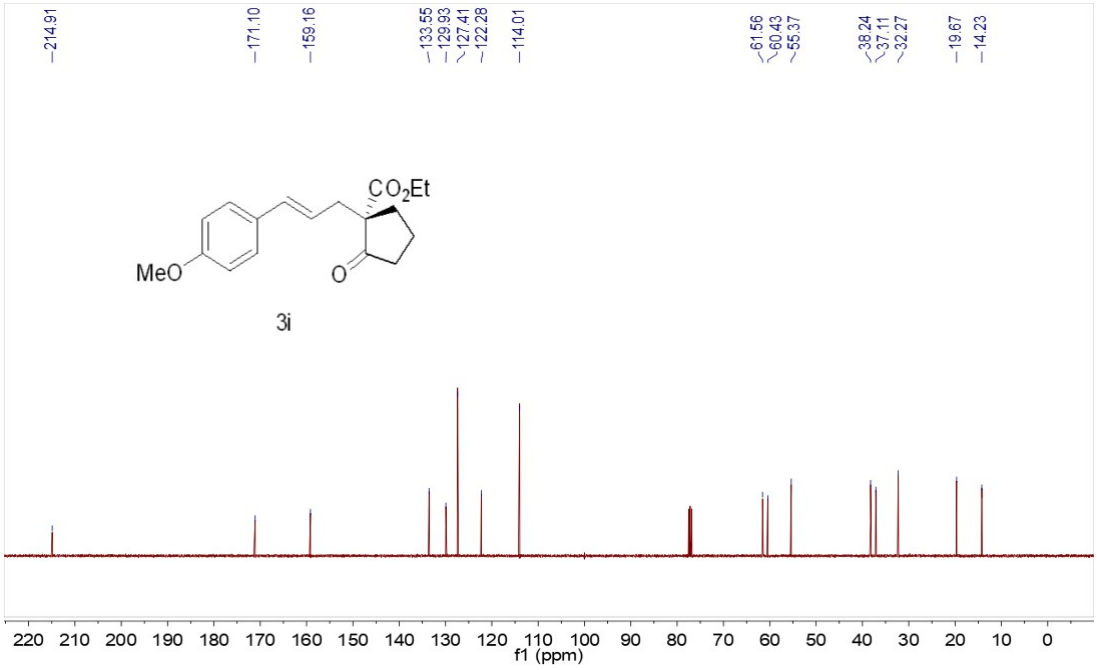
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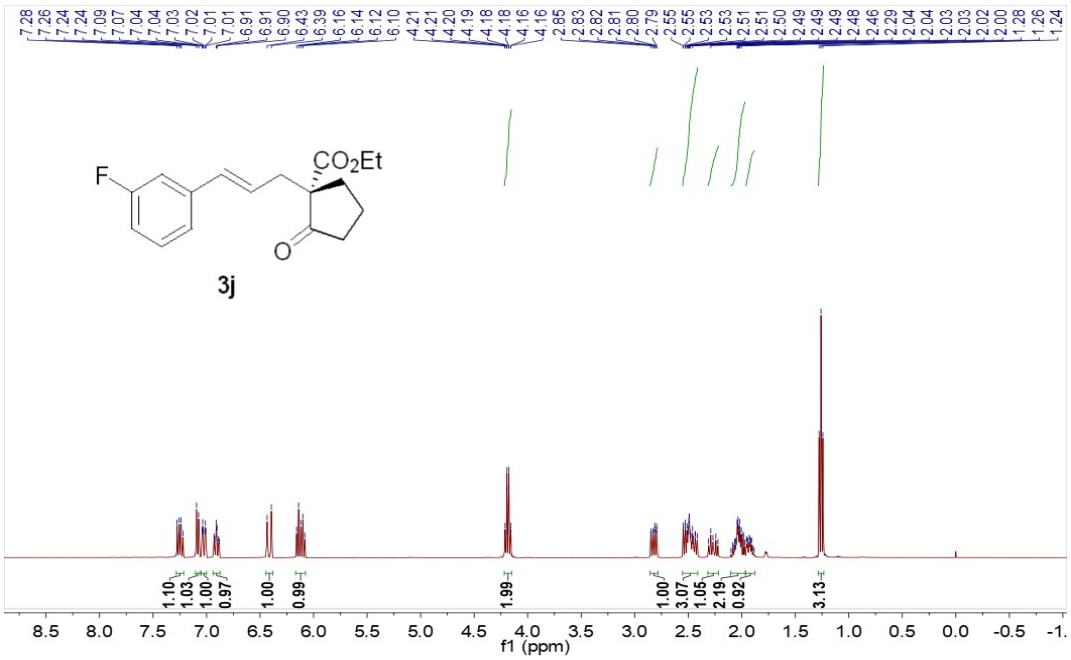
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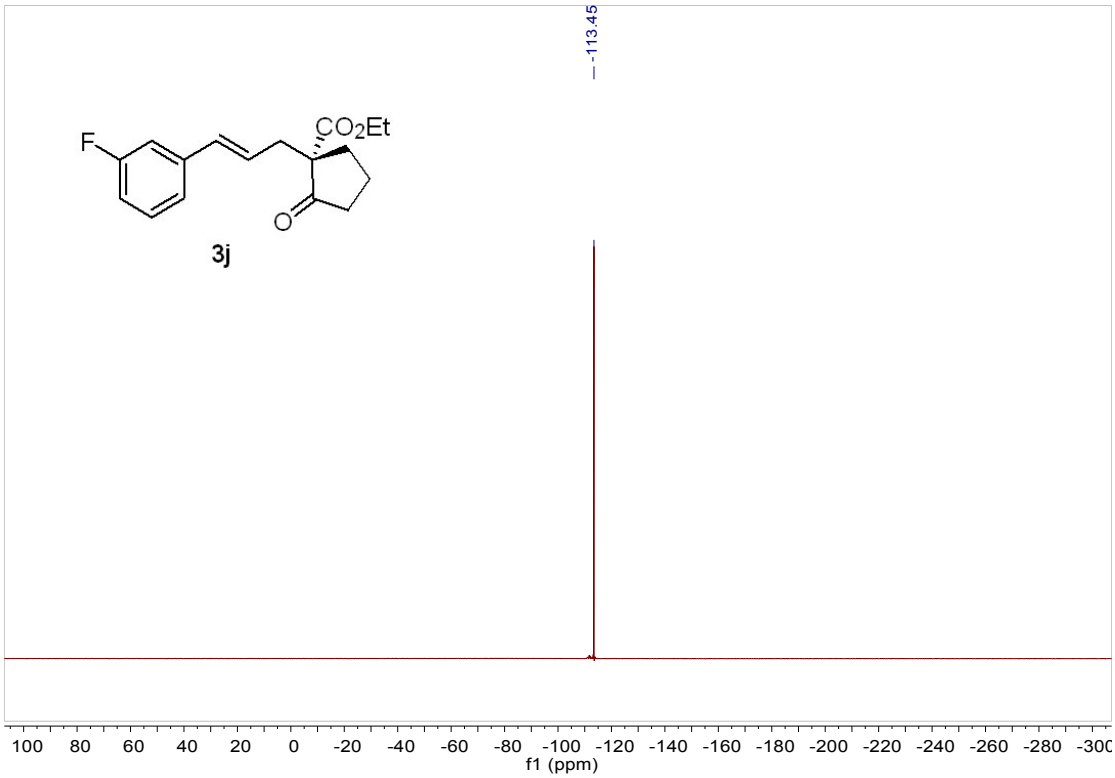
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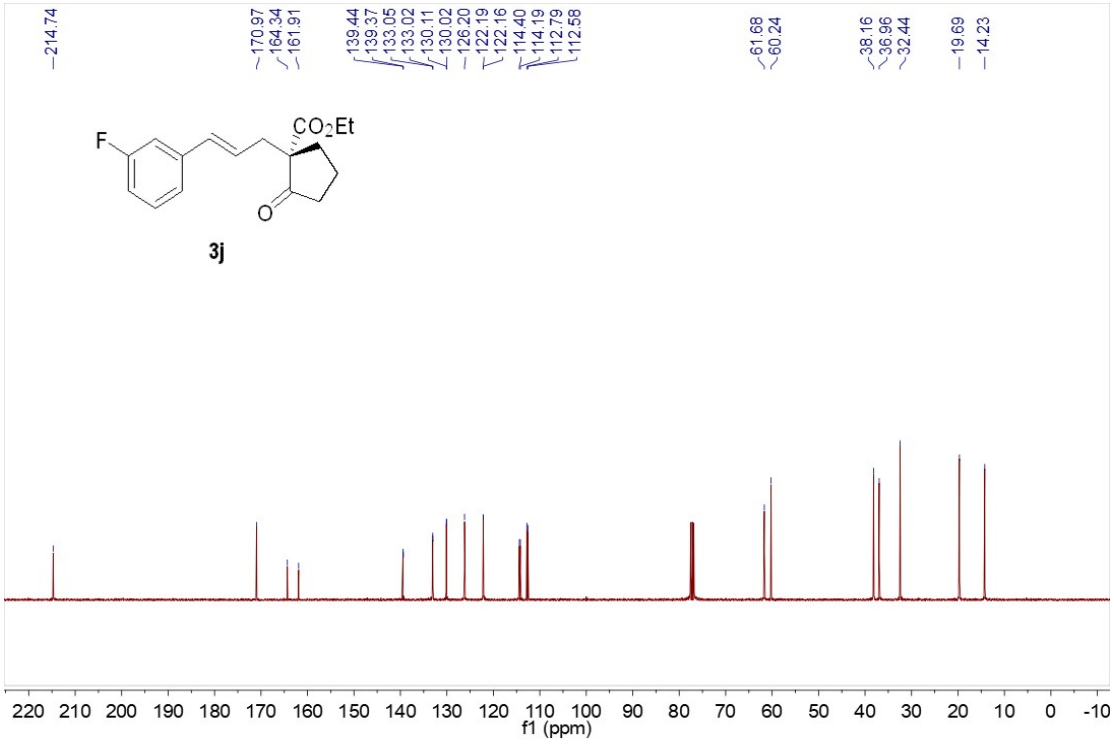
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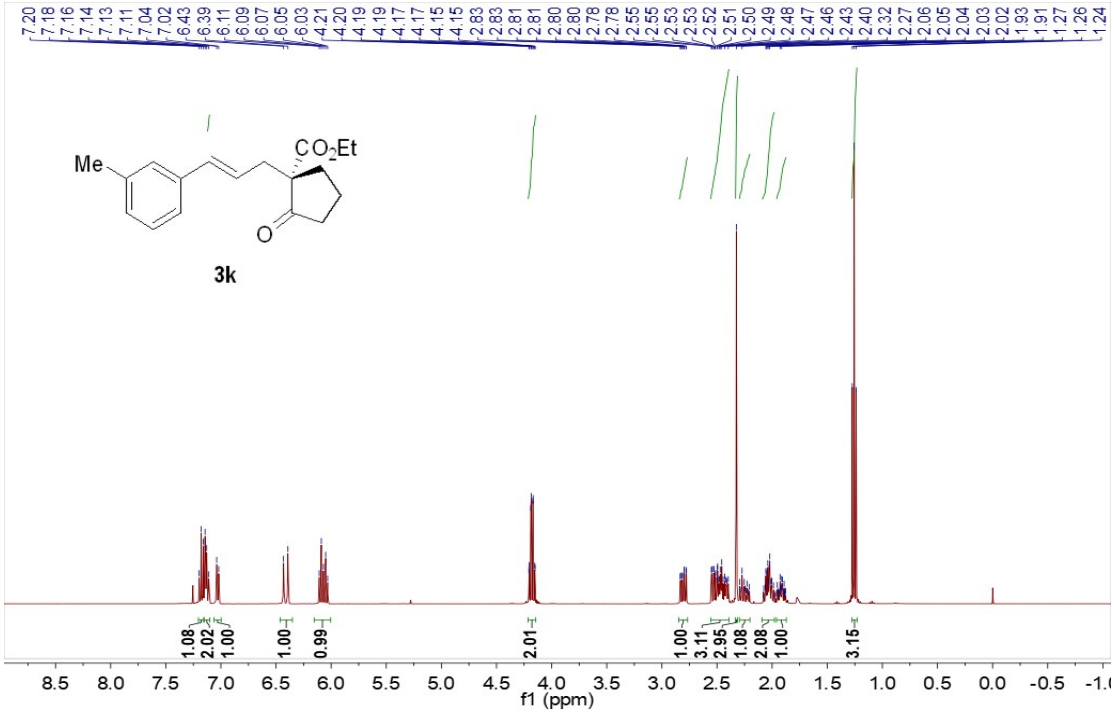
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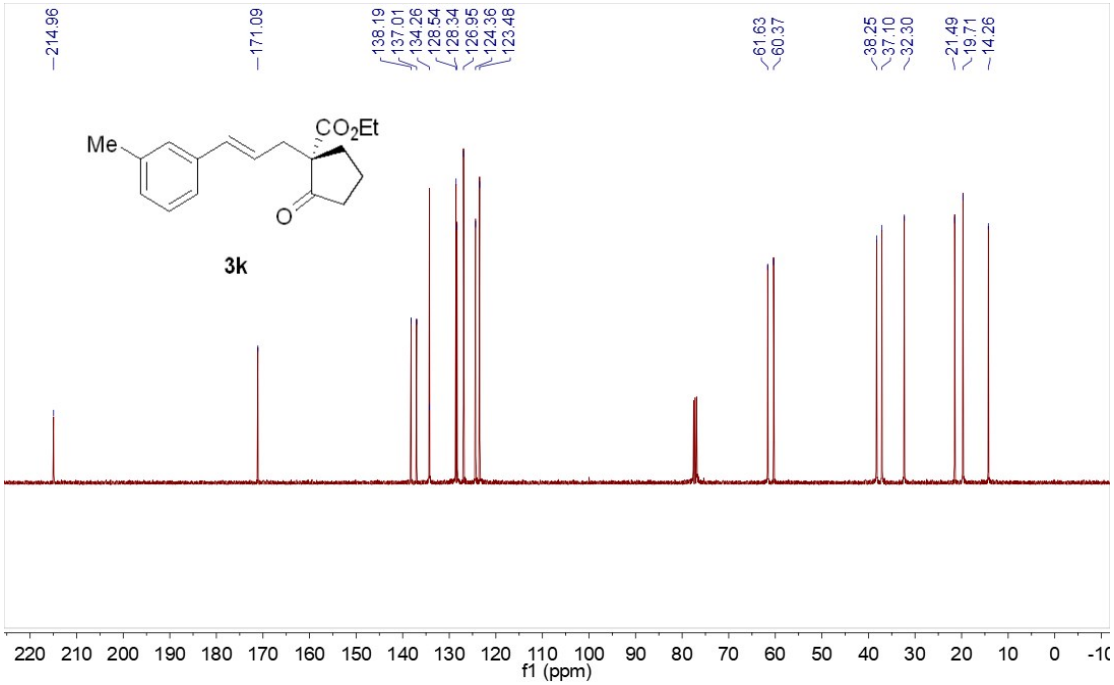
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O
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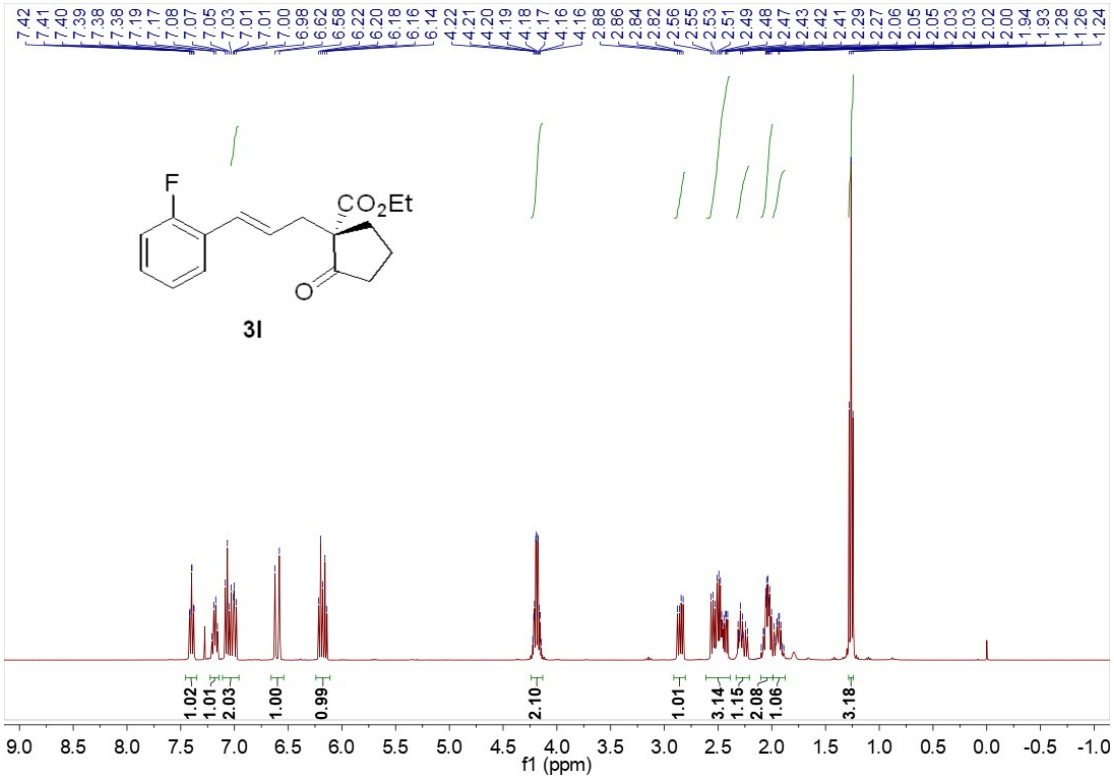
f1 (ppm)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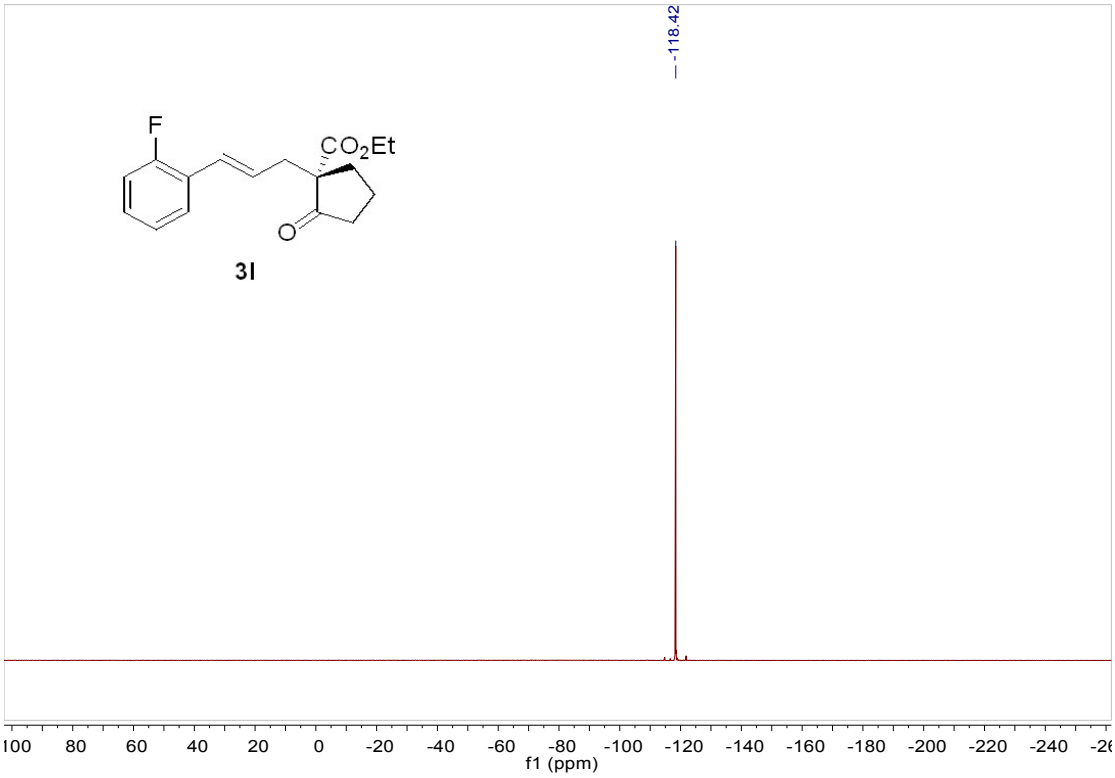
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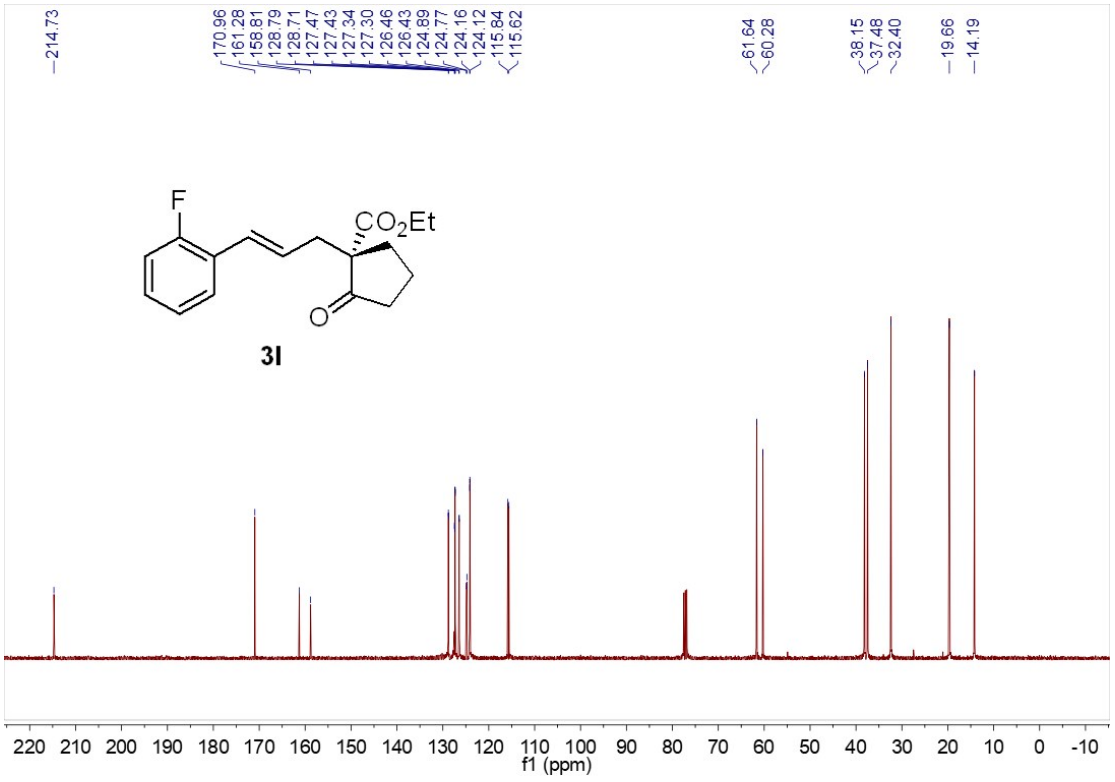
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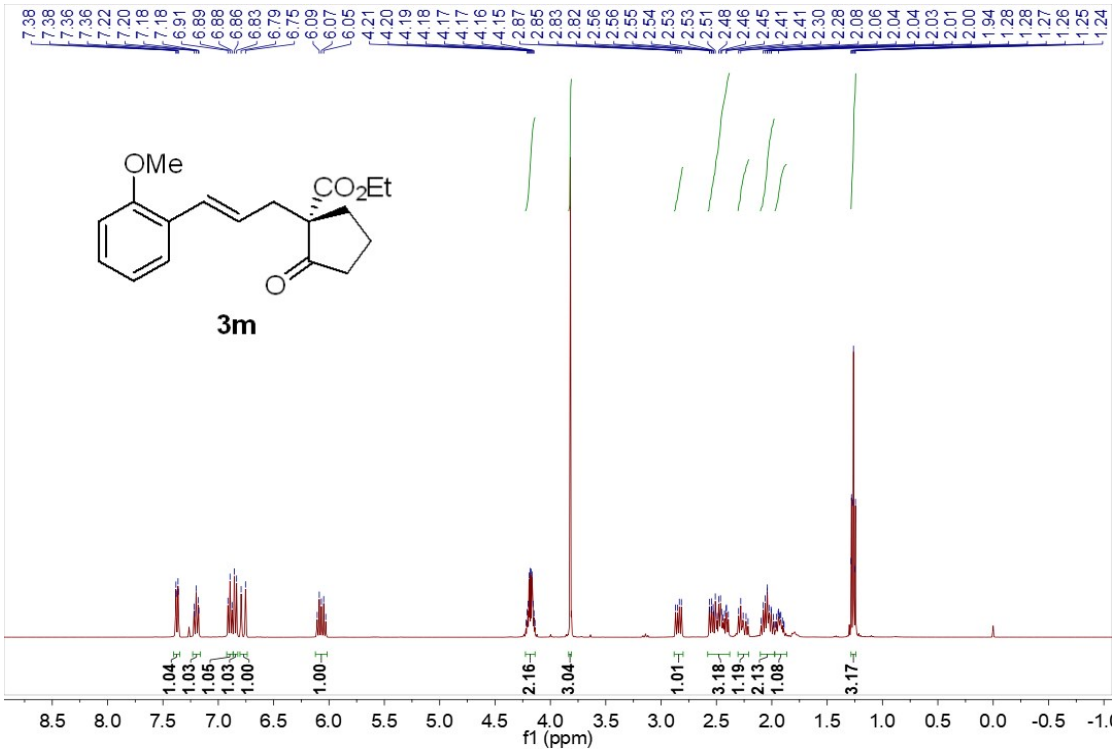
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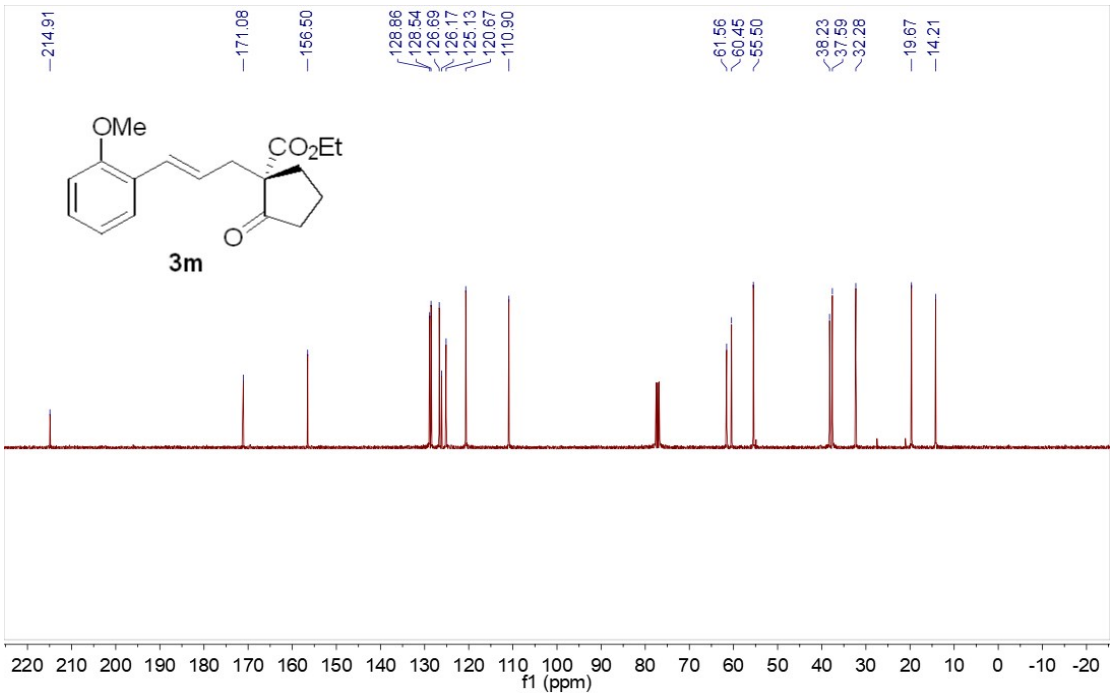
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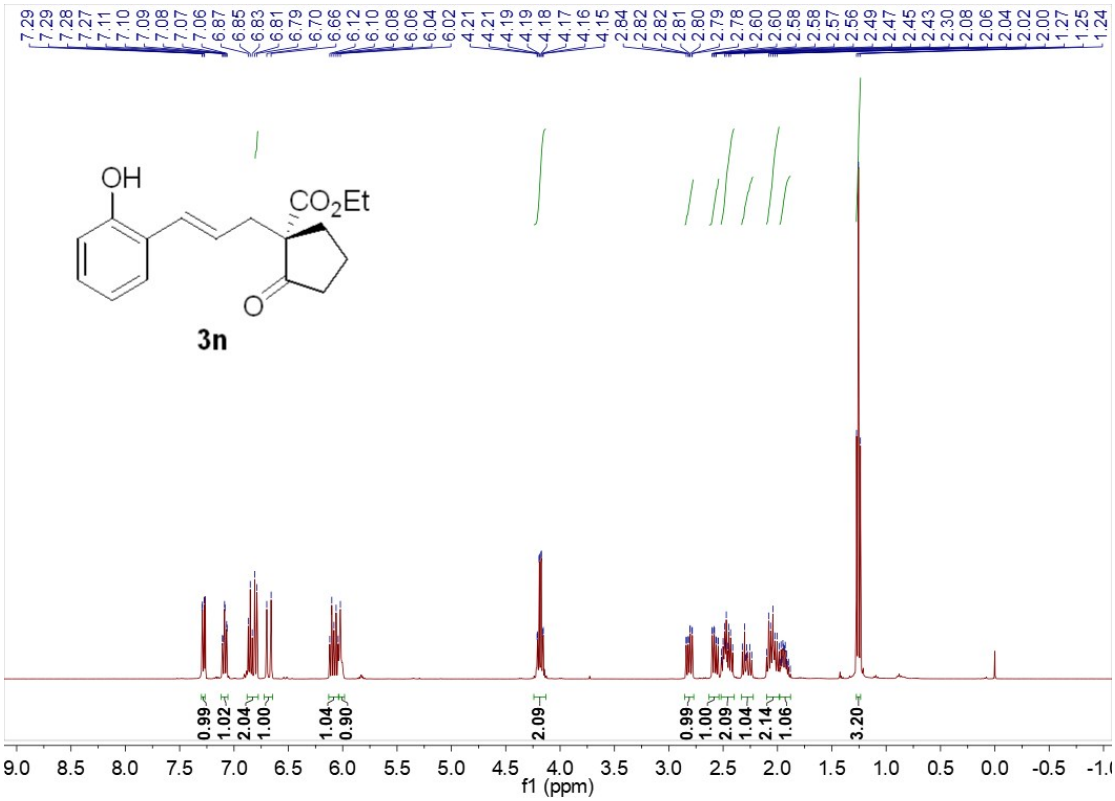
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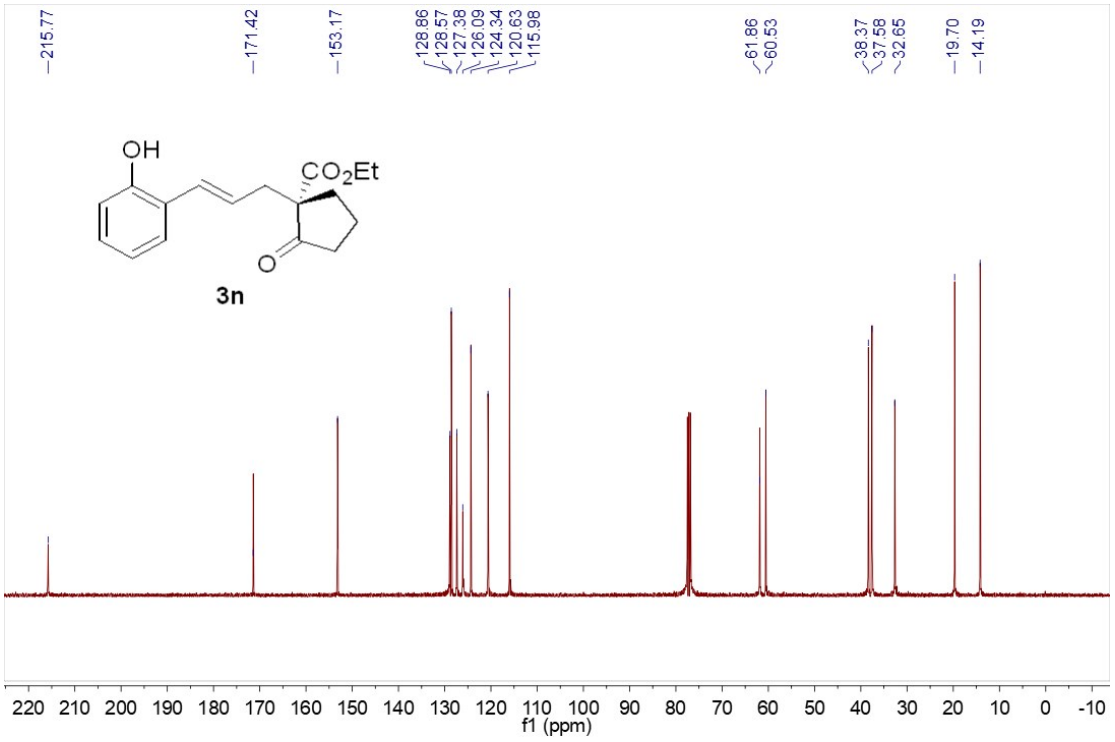
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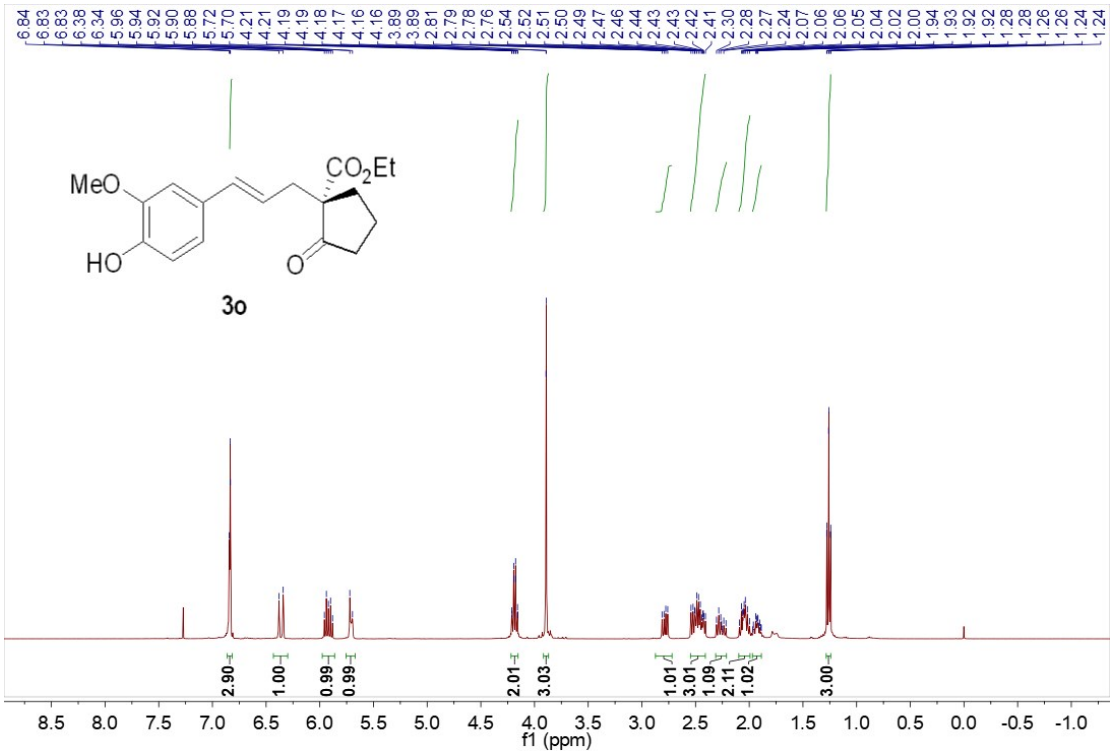
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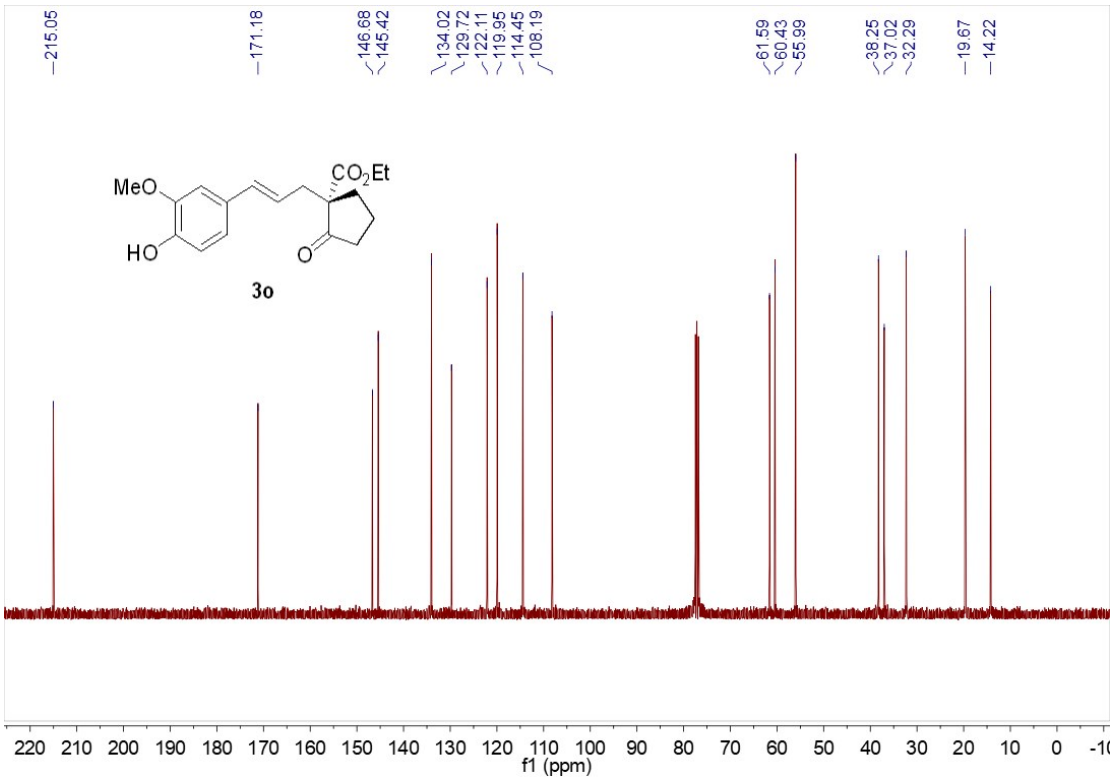
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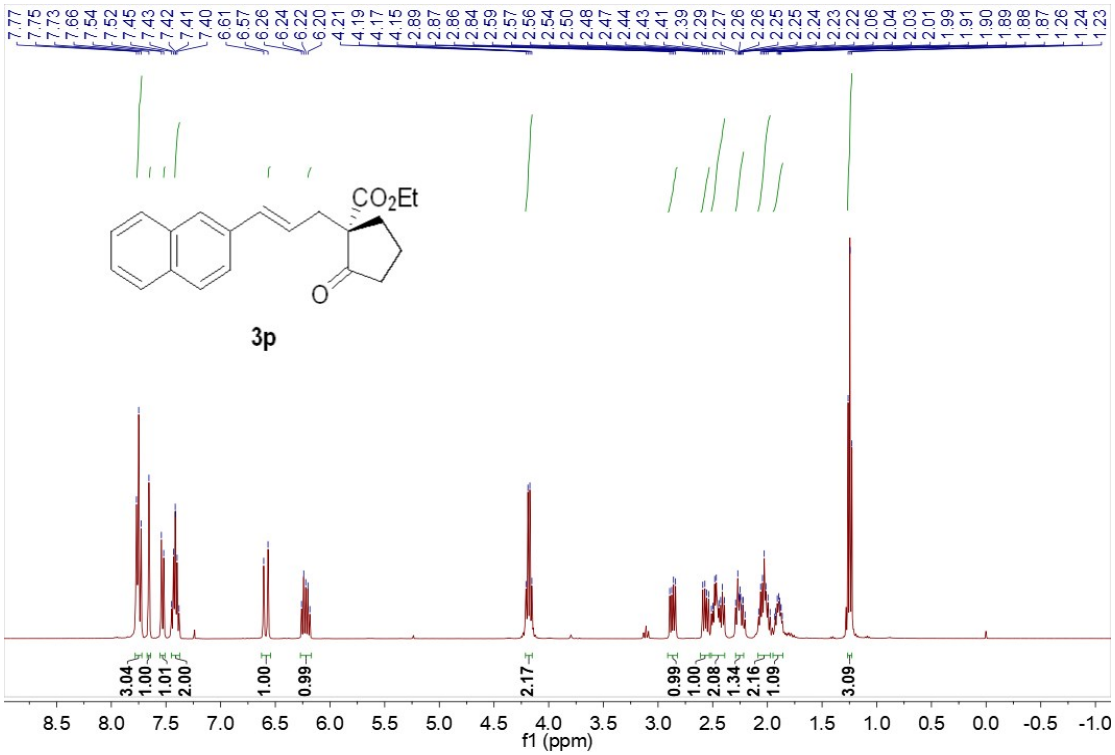
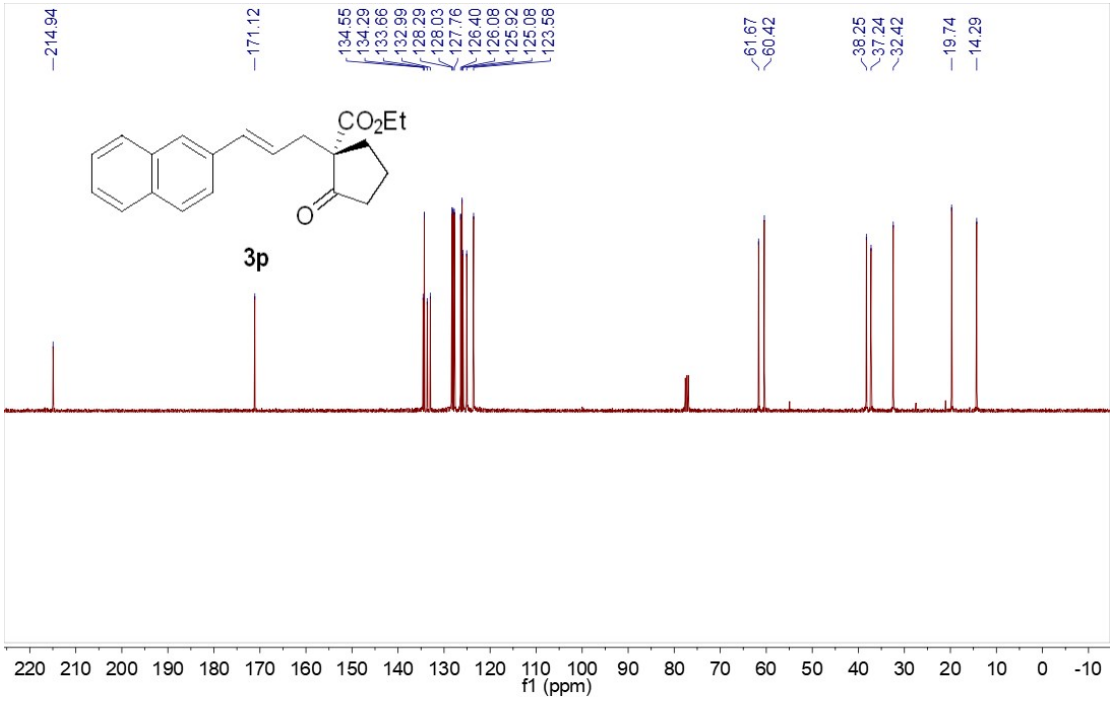
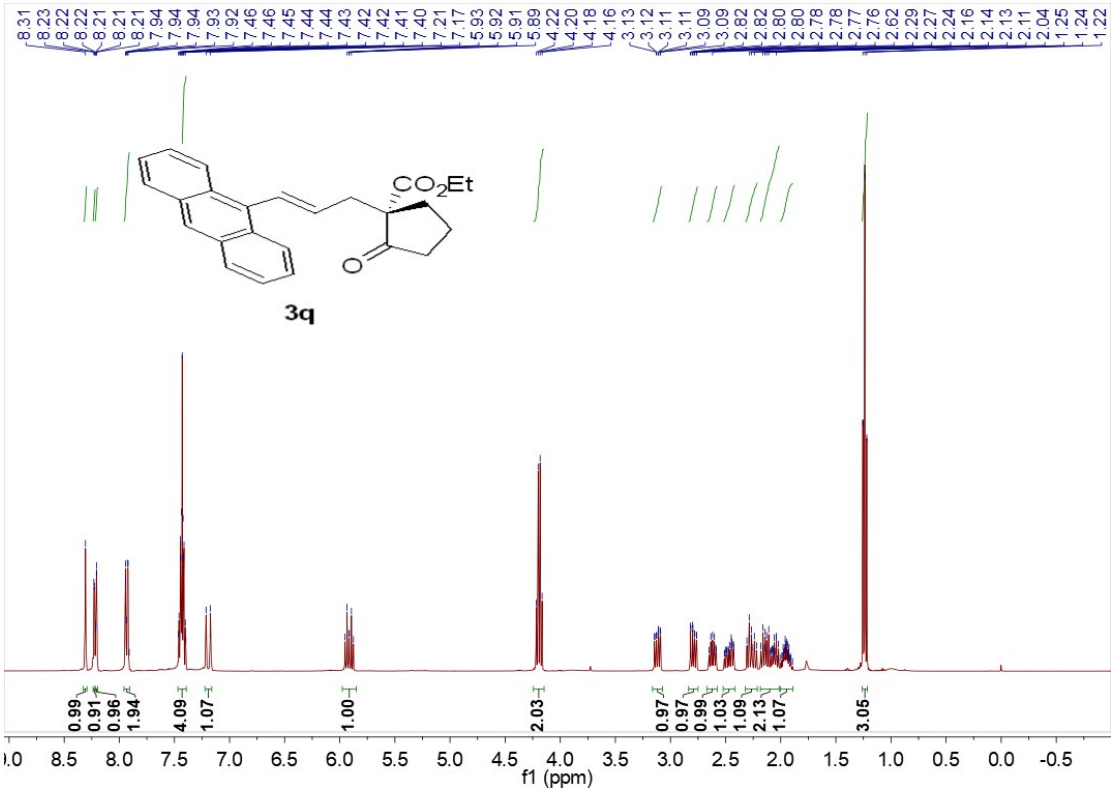
f1 (ppm)


7.77 7.75 7.73 7.66 7.54 7.52 7.45 7.43 7.42 7.41 7.40 6.61 6.57 6.26 6.24 6.22 6.20 4.21 4.19 4.17 4.15 2.89 2.87 2.86 2.84 2.59 2.57 2.56 2.54 2.50 2.48 2.47 2.44 2.43 2.41 2.39 2.29 2.27 2.26 2.26 2.25 2.25 2.24 2.23 2.22 2.06 2.04 2.03 2.01 1.99 1.91 1.90 1.89 1.88 1.87 1.26 1.24 1.23
CO2Et
O
3p
3.04 1.00 1.01 2.00 1.00 0.99 2.17 0.99 1.00 2.08 1.34 2.16 1.09 3.09
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


214.94 171.12 134.55 134.29 133.66 132.99 128.29 128.03 127.76 126.40 126.08 125.92 125.08 123.58 61.67 60.42 38.25 37.24 32.42 19.74 14.29
CO2Et
O
3p
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.31 8.23 8.22 8.22 8.21 8.21 8.21 8.21 7.94 7.94 7.94 7.93 7.92 7.46 7.46 7.45 7.44 7.44 7.43 7.42 7.42 7.41 7.40 7.21 7.17 5.93 5.92 5.91 5.89 4.22 4.20 4.18 4.16 3.13 3.12 3.11 3.11 3.09 3.09 2.82 2.82 2.80 2.80 2.78 2.78 2.77 2.76 2.62 2.29 2.27 2.24 2.16 2.14 2.13 2.11 2.04 1.25 1.24 1.22
CO2Et
O
3q
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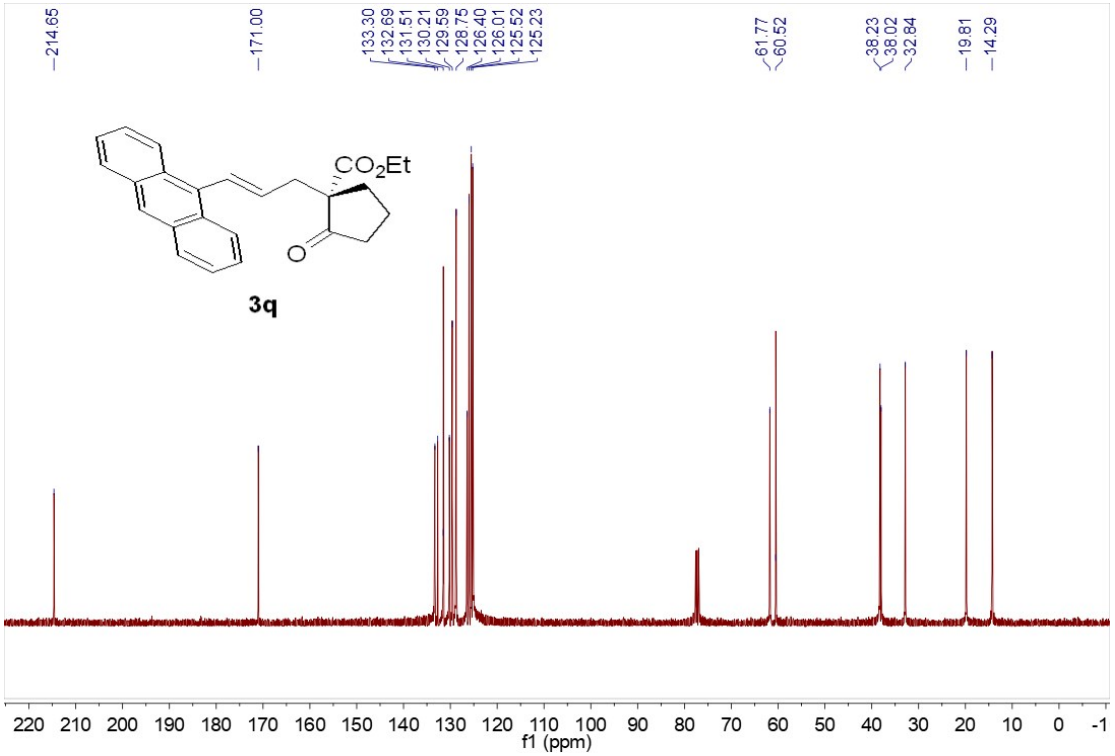
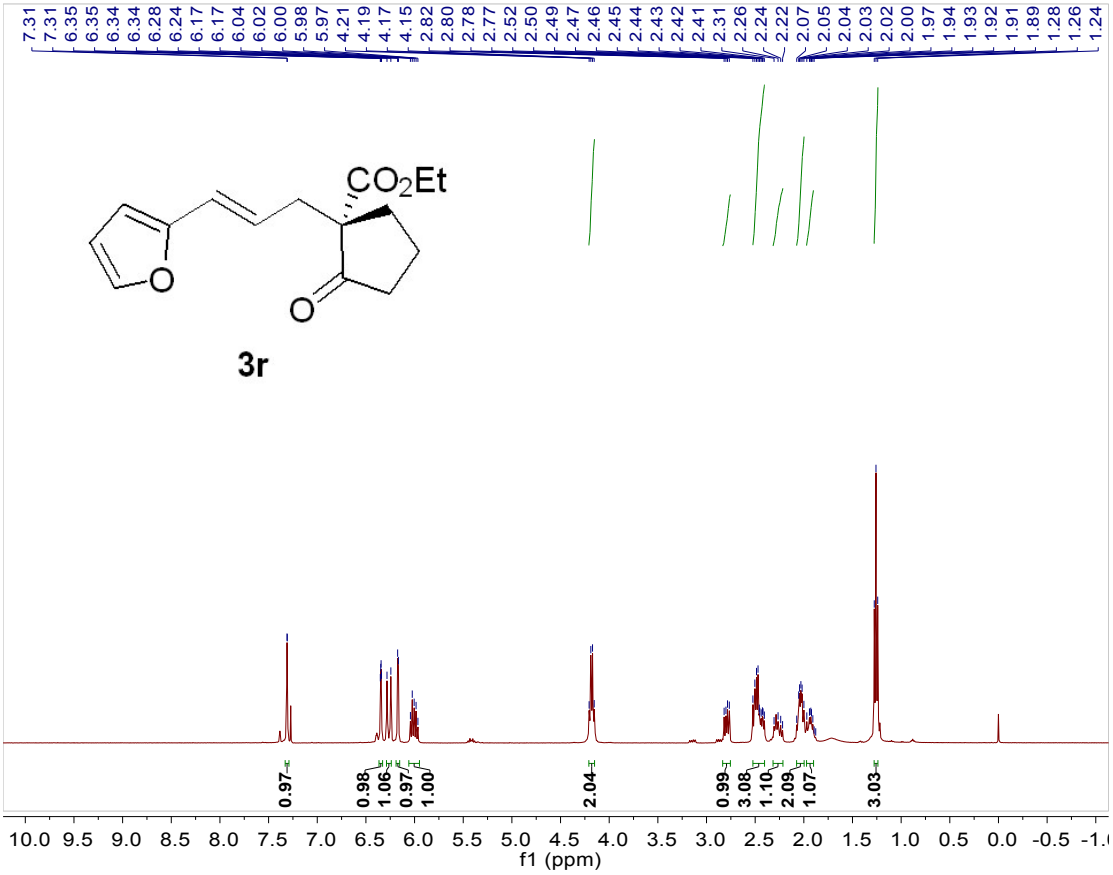
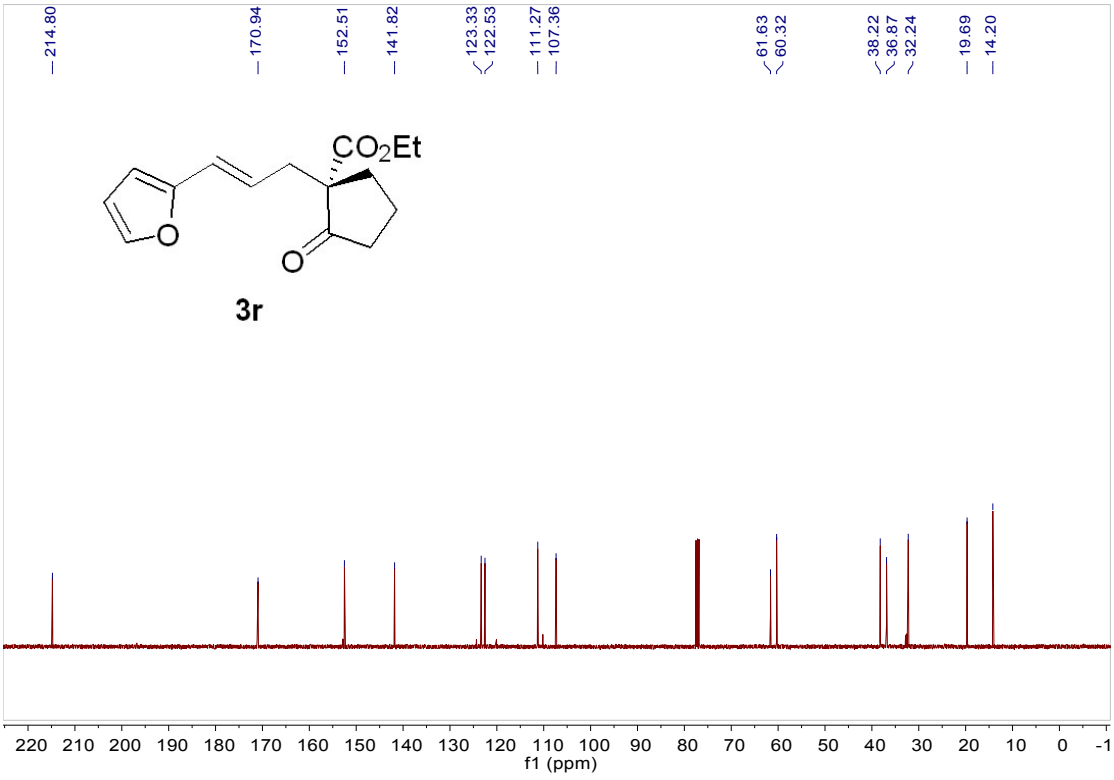
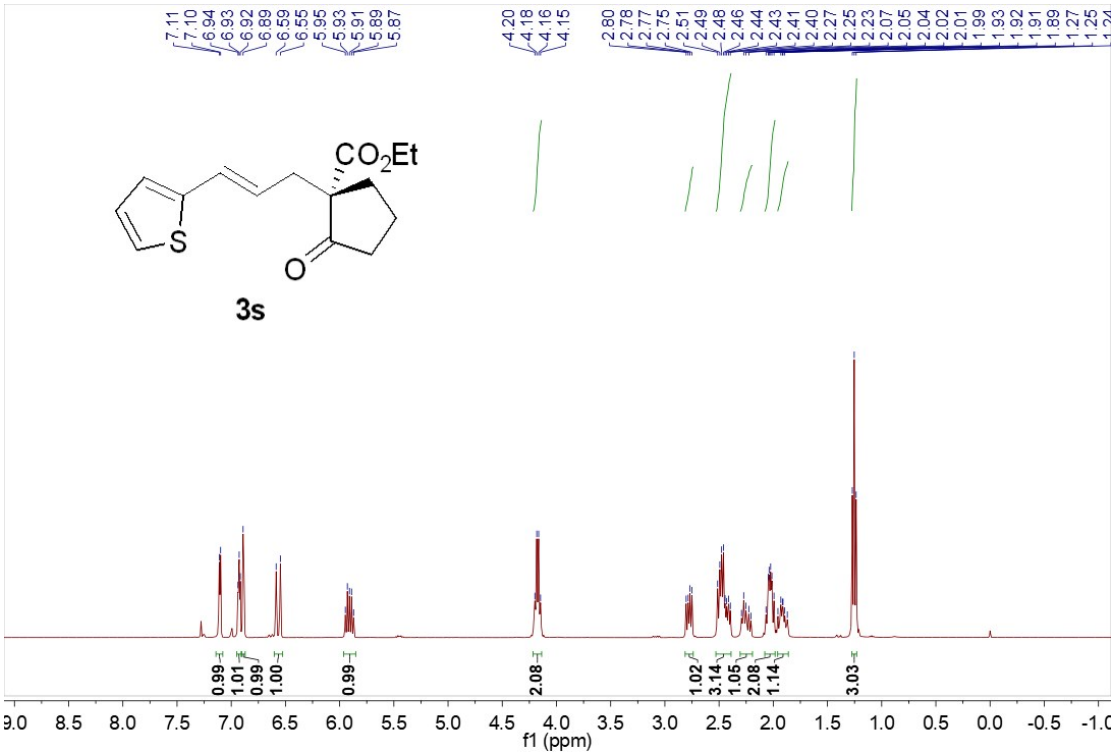
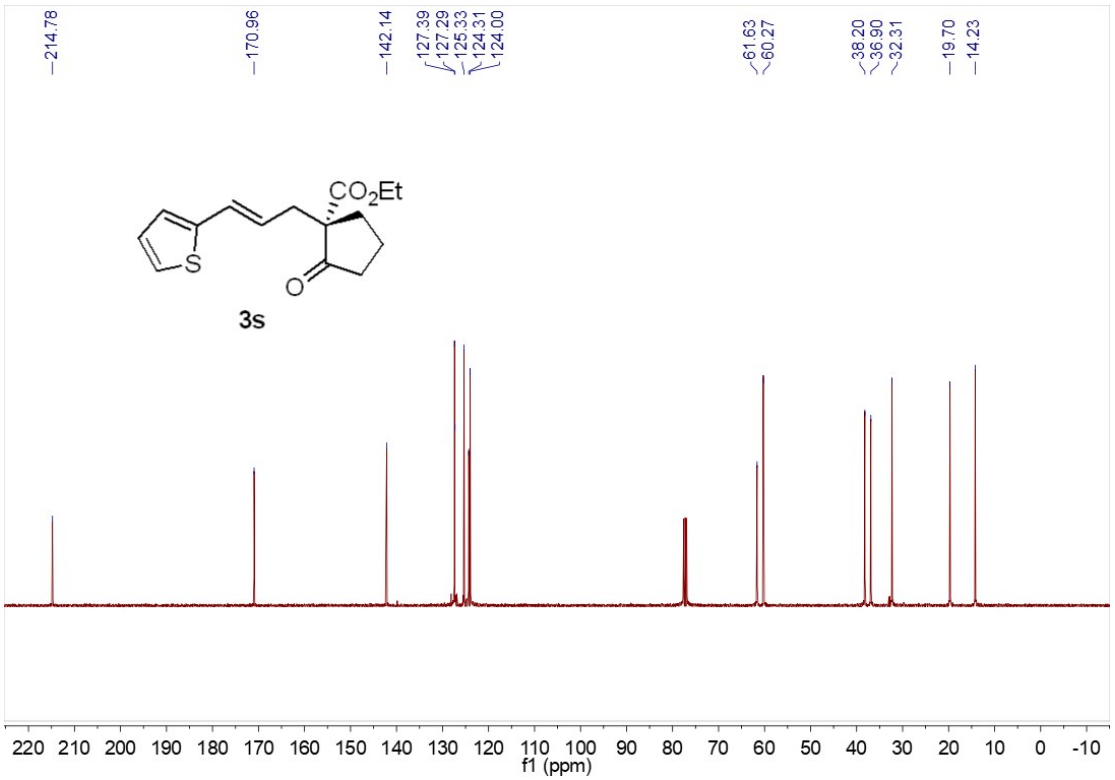
f1 (ppm)

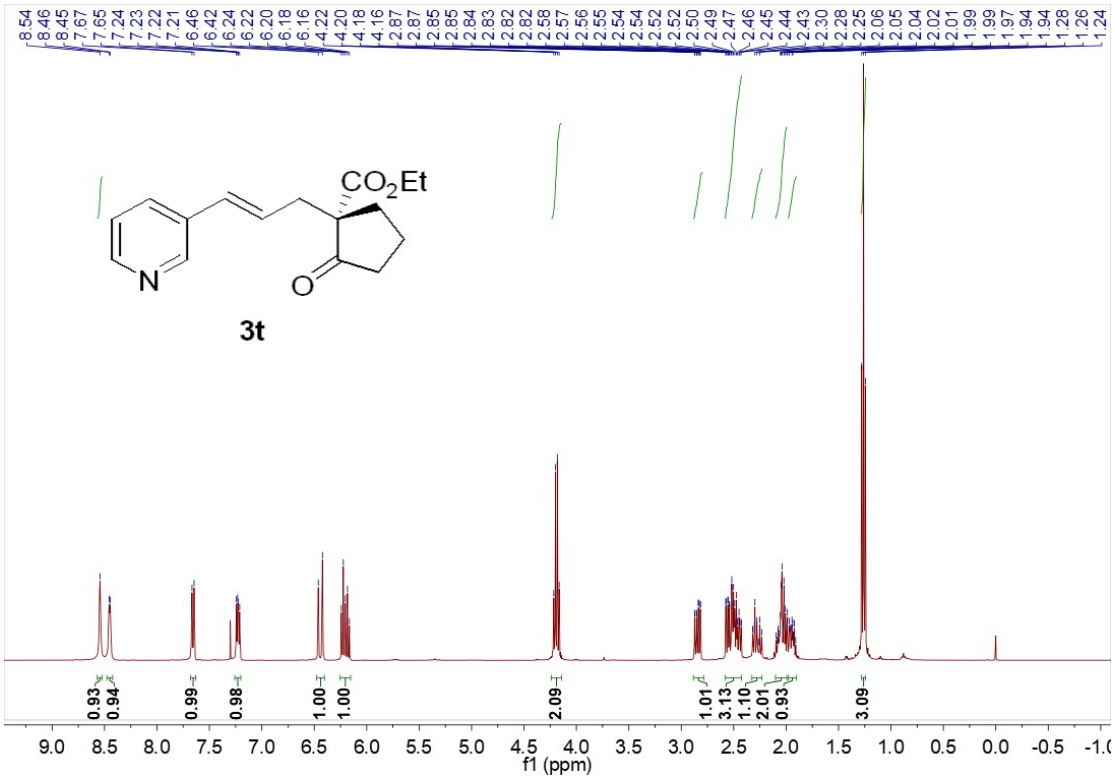

214.65 171.00 133.30 132.69 131.51 130.21 129.59 128.75 126.40 126.01 125.52 125.23 61.77 60.52 38.23 38.02 32.84 19.81 14.29
CO2Et
O
3q
f1 (ppm)

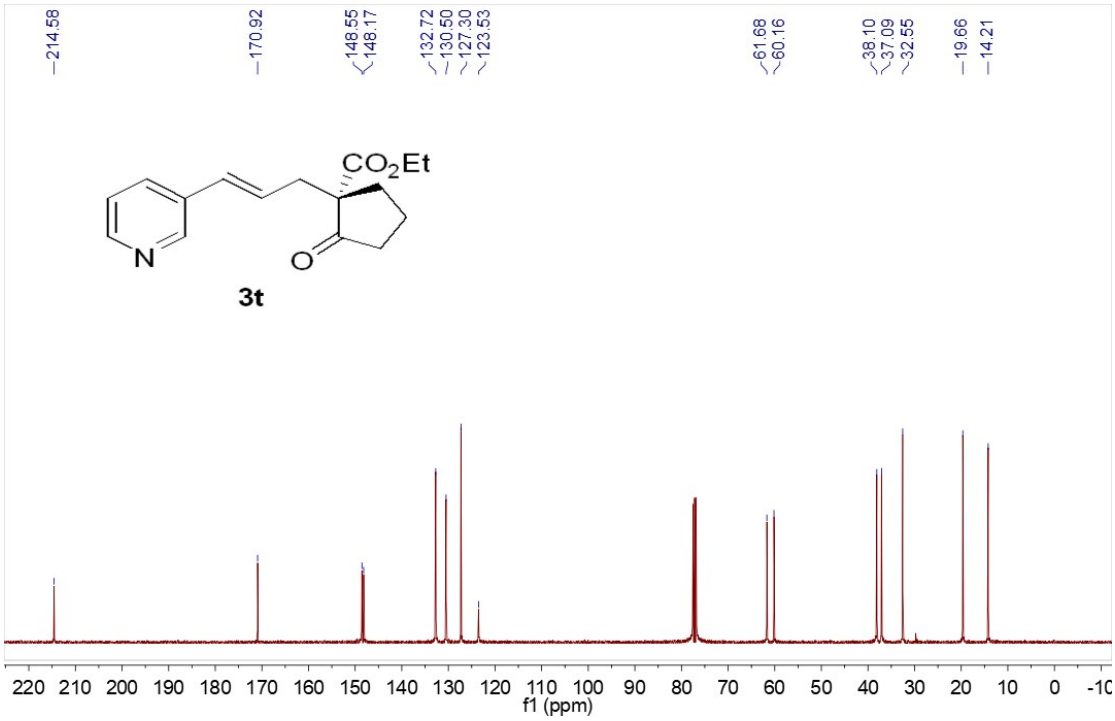

7.31 7.31 6.35 6.35 6.34 6.34 6.28 6.24 6.17 6.17 6.04 6.02 6.00 5.98 5.97 4.21 4.19 4.17 4.15 2.82 2.80 2.78 2.77 2.52 2.50 2.49 2.47 2.46 2.45 2.44 2.43 2.42 2.41 2.31 2.26 2.24 2.22 2.07 2.05 2.04 2.03 2.02 2.00 1.97 1.94 1.93 1.92 1.91 1.89 1.28 1.26 1.24
CO2Et
O
O
3r
0.97 0.98 1.06 0.97 1.00 2.04 0.99 3.08 1.10 2.09 1.07 3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

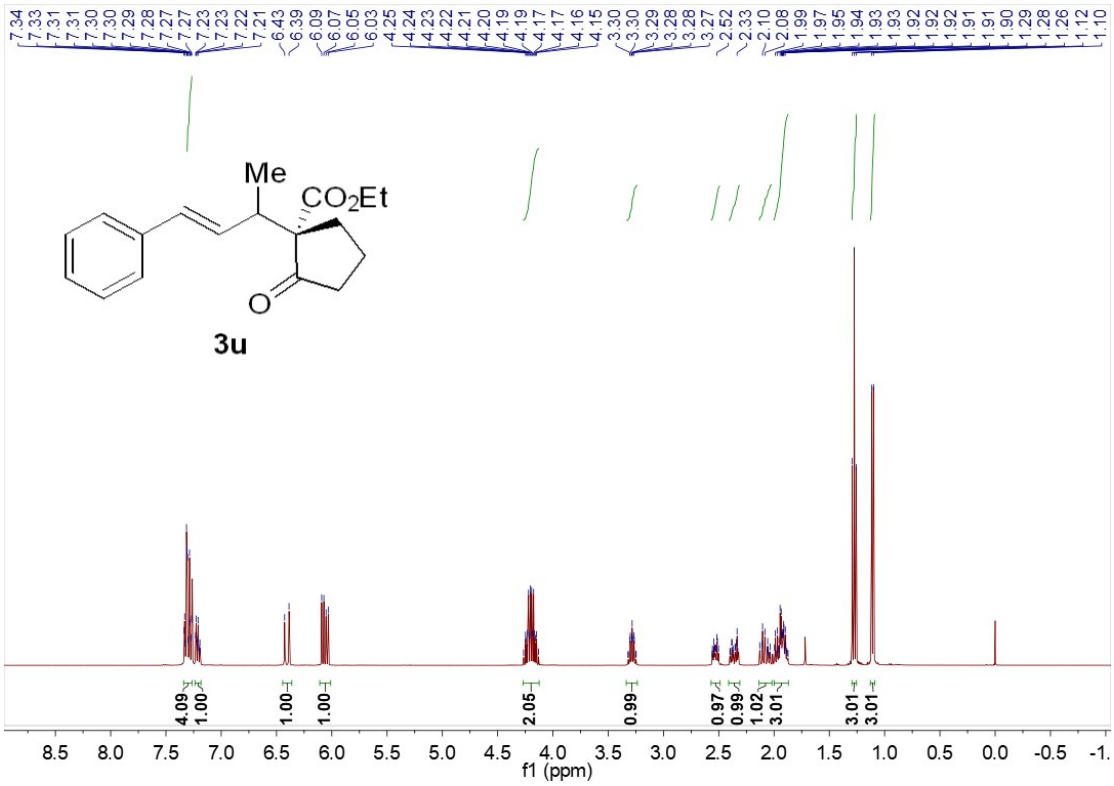

214.80 170.94 152.51 141.82 123.33 122.53 111.27 107.36 61.63 60.32 38.22 36.87 32.24 19.69 14.20
CO2Et
O
O
3r
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

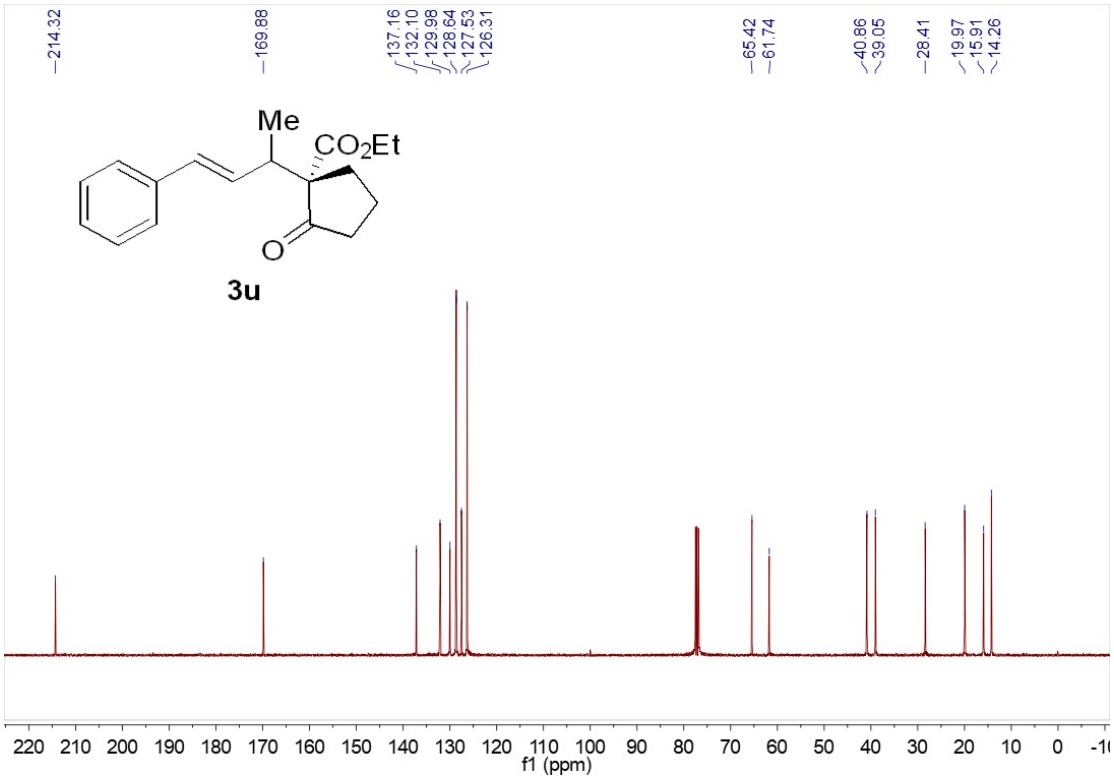

7.11
7.10
6.94
6.93
6.92
6.89
6.59
6.55
5.95
5.93
5.91
5.89
5.87
4.20
4.18
4.16
4.15
2.80
2.78
2.77
2.75
2.51
2.49
2.48
2.46
2.44
2.43
2.41
2.40
2.27
2.25
2.23
2.07
2.05
2.04
2.02
2.01
1.99
1.93
1.92
1.91
1.89
1.27
1.25
CO2Et
S
O
3s
0.99
1.01
0.99
1.00
0.99
2.08
1.02
3.14
1.05
2.08
1.14
3.03
f1 (ppm)

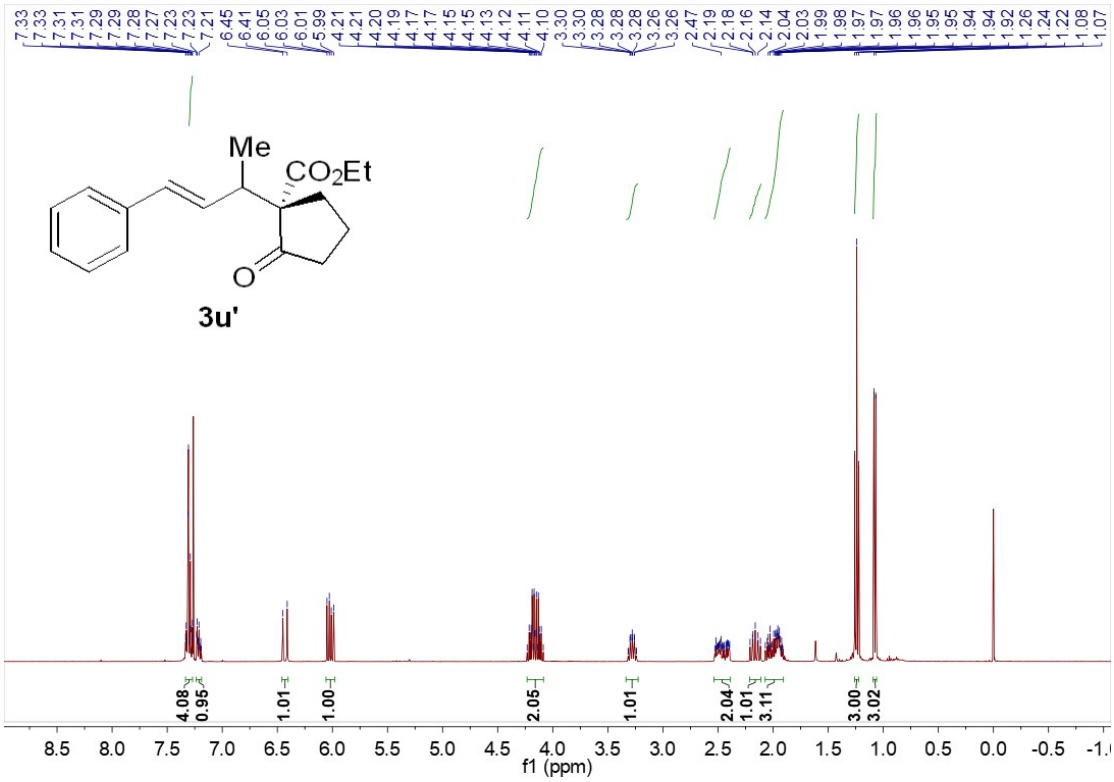

214.78
170.96
142.14
127.39
127.29
125.33
124.31
124.00
61.63
60.27
38.20
36.90
32.31
19.70
14.23
CO2Et
S
O
3s
f1 (ppm)

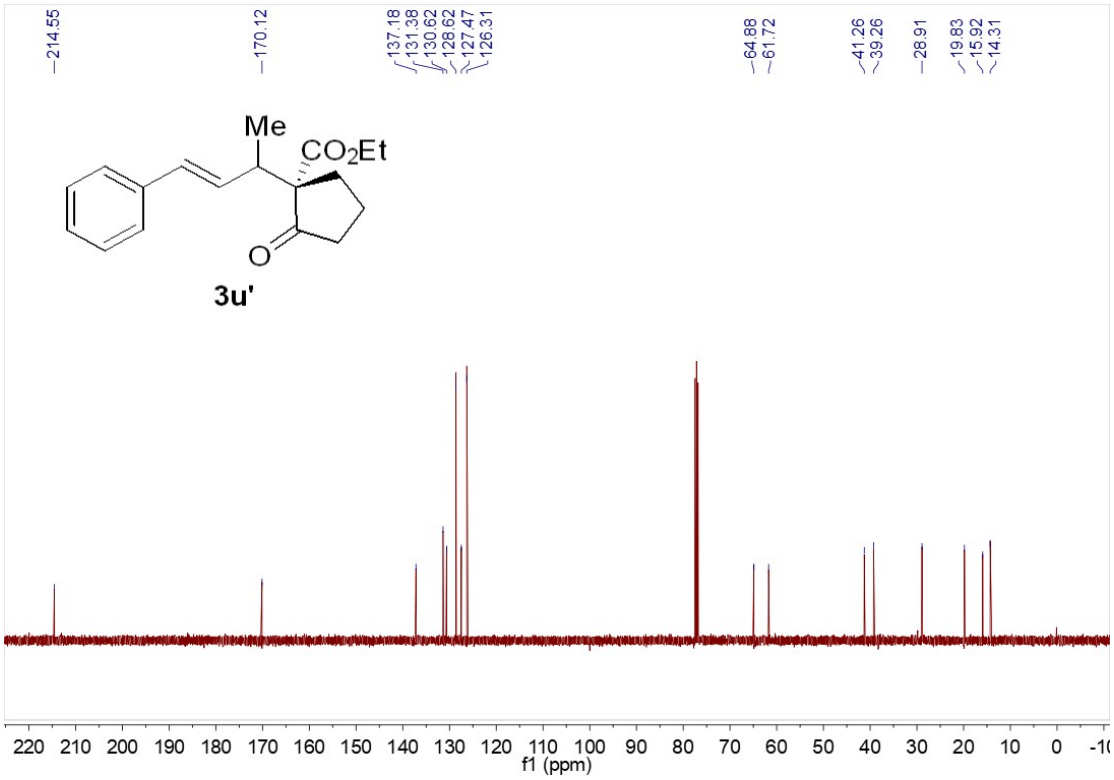

8.54 8.46 8.45 7.67 7.65 7.24 7.23 7.22 7.21 6.46 6.42 6.24 6.22 6.20 6.18 6.16 4.22 4.20 4.18 4.16 2.87 2.85 2.85 2.84 2.83 2.82 2.82 2.58 2.57 2.56 2.55 2.54 2.54 2.52 2.52 2.50 2.49 2.47 2.46 2.45 2.44 2.43 2.30 2.28 2.25 2.06 2.05 2.04 2.02 2.01 1.99 1.99 1.97 1.94 1.94 1.28 1.26 1.24
CO2Et
N
O
3t
0.93 0.94 0.99 0.98 1.00 1.00 2.09 1.01 3.13 1.10 2.01 0.93 3.09
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

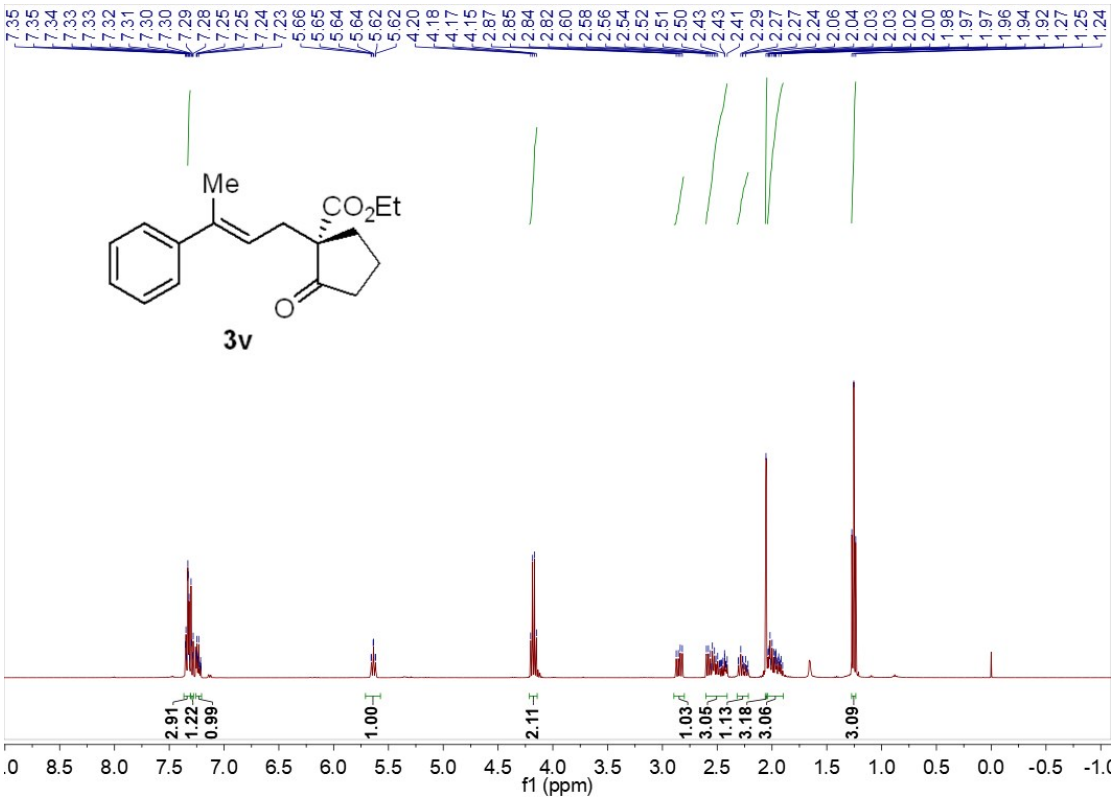

214.58 170.92 148.55 148.17 132.72 130.50 127.30 123.53 61.68 60.16 38.10 37.09 32.55 19.66 14.21
CO2Et
N
O
3t
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

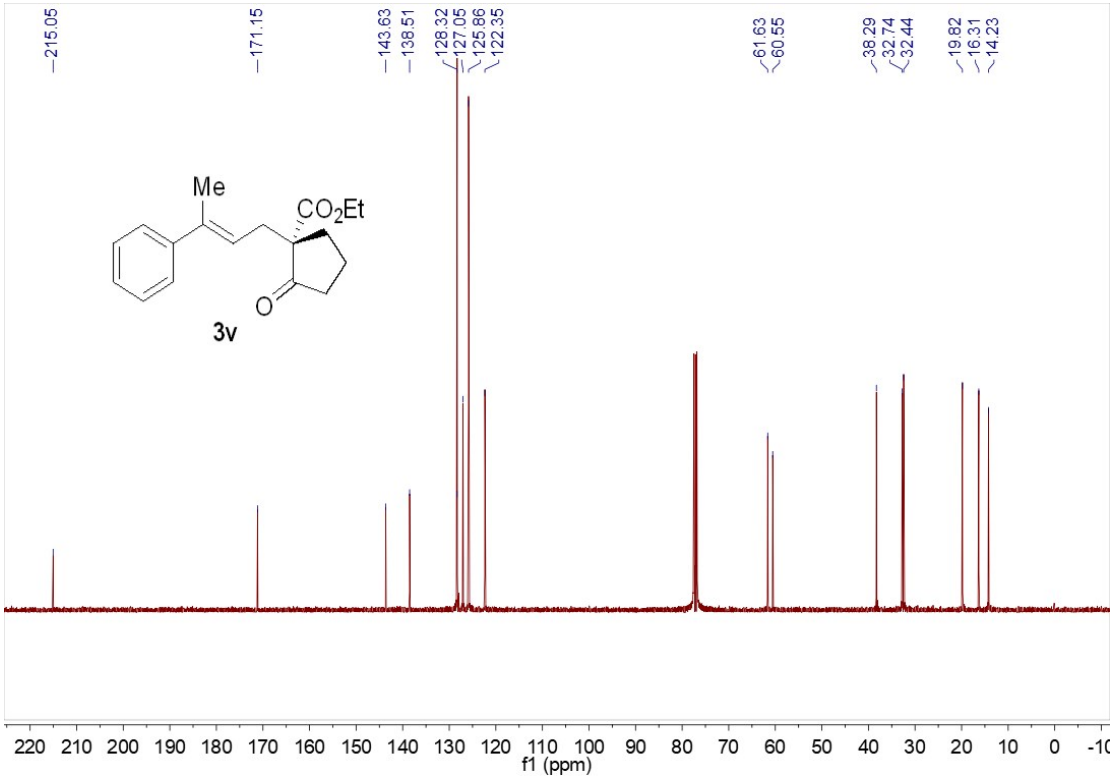

Me
CO2Et
O
3u
f1 (ppm)

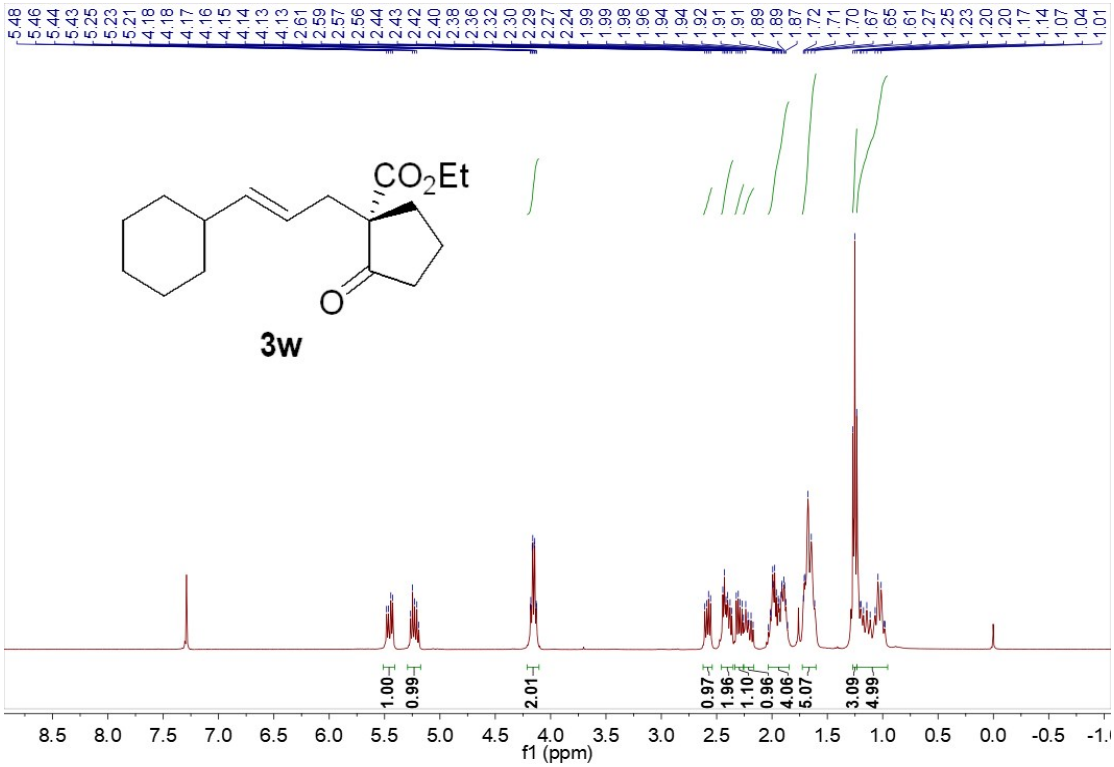

Me
CO2Et
O
3u
f1 (ppm)

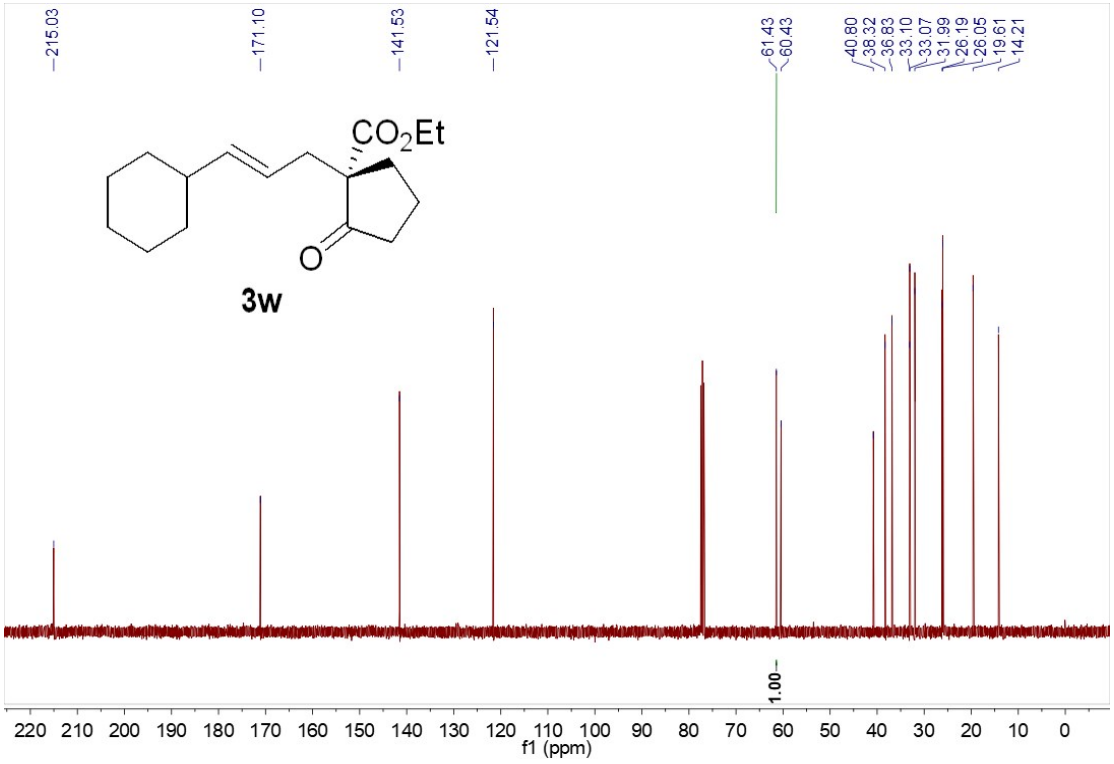

Me
CO2Et
O
3u'
f1 (ppm)

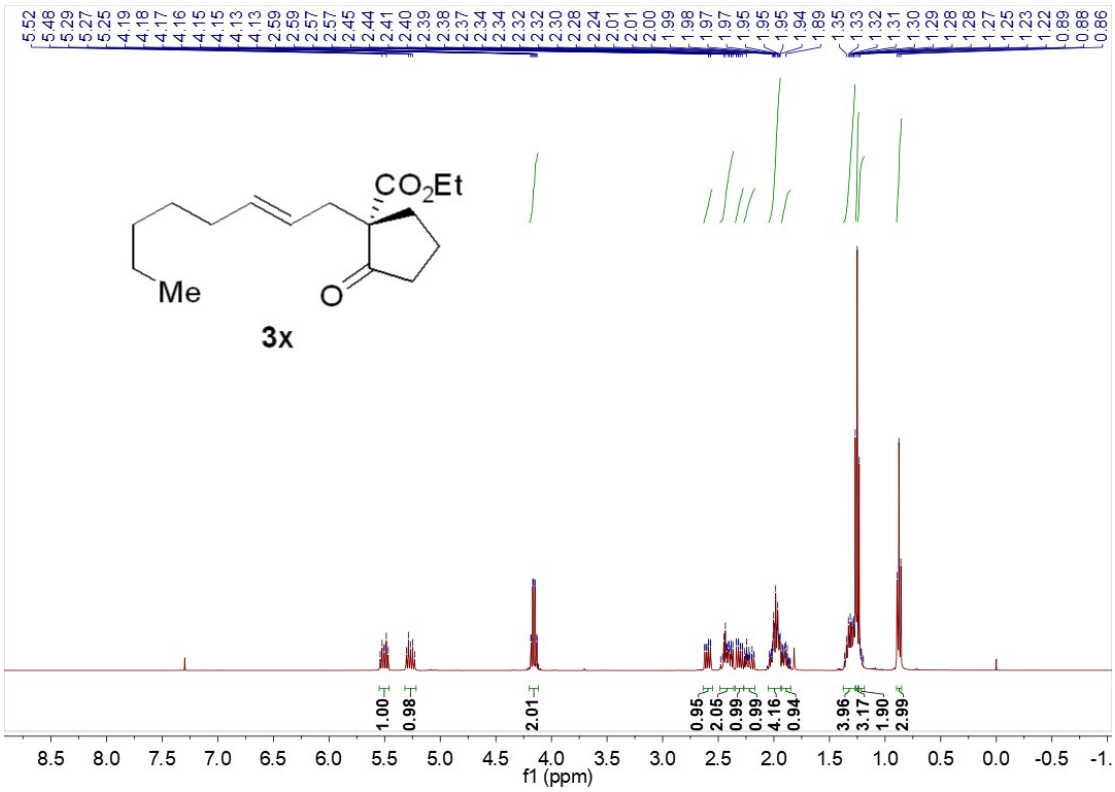

Me
CO2Et
O
3u'
f1 (ppm)

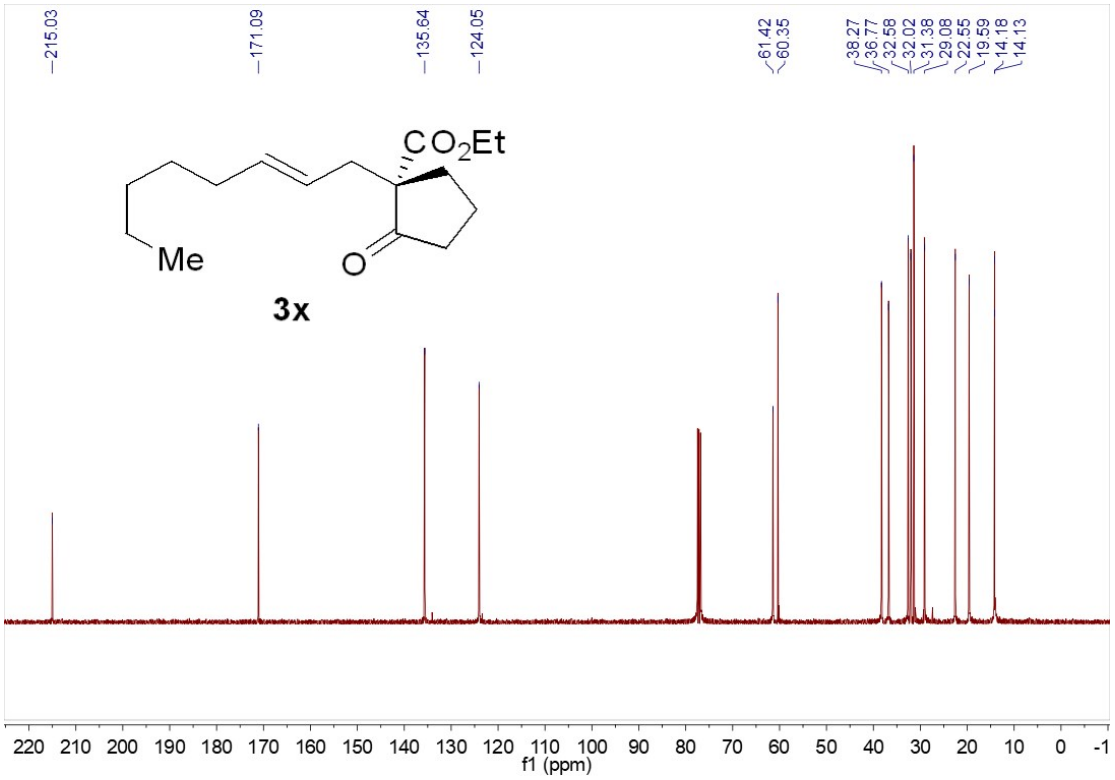

Me
CO2Et
O
3v
f1 (ppm)

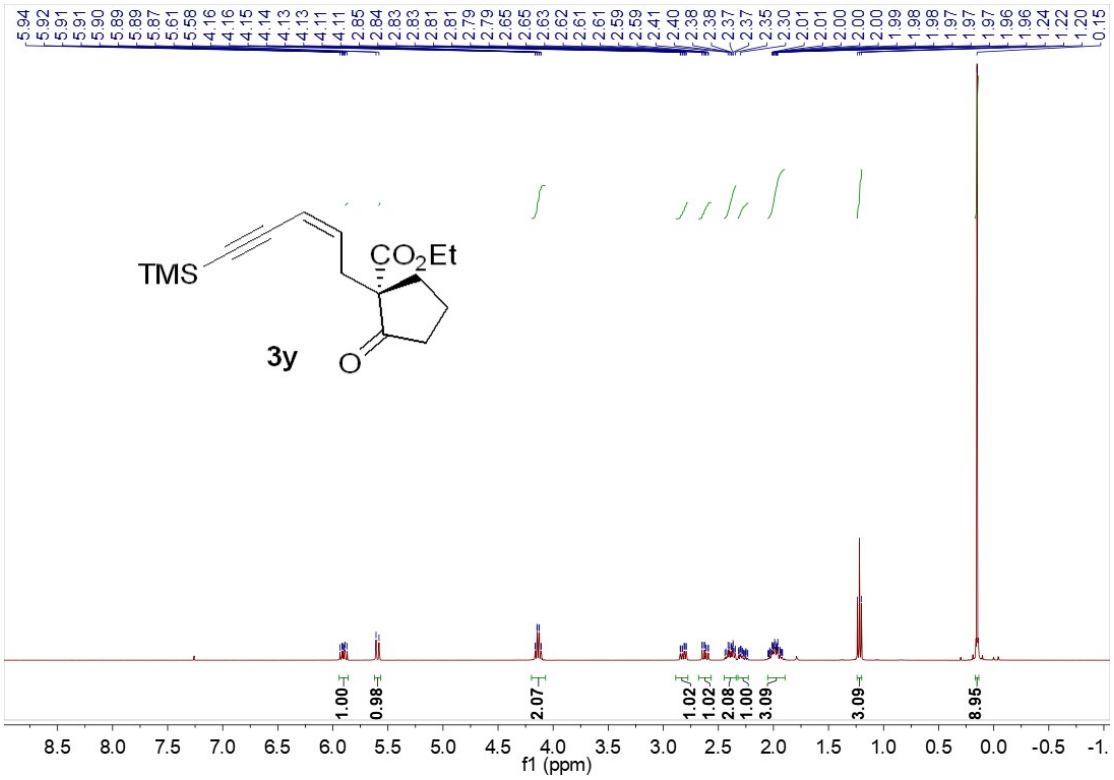

Me
CO2Et
O
3v
f1 (ppm)

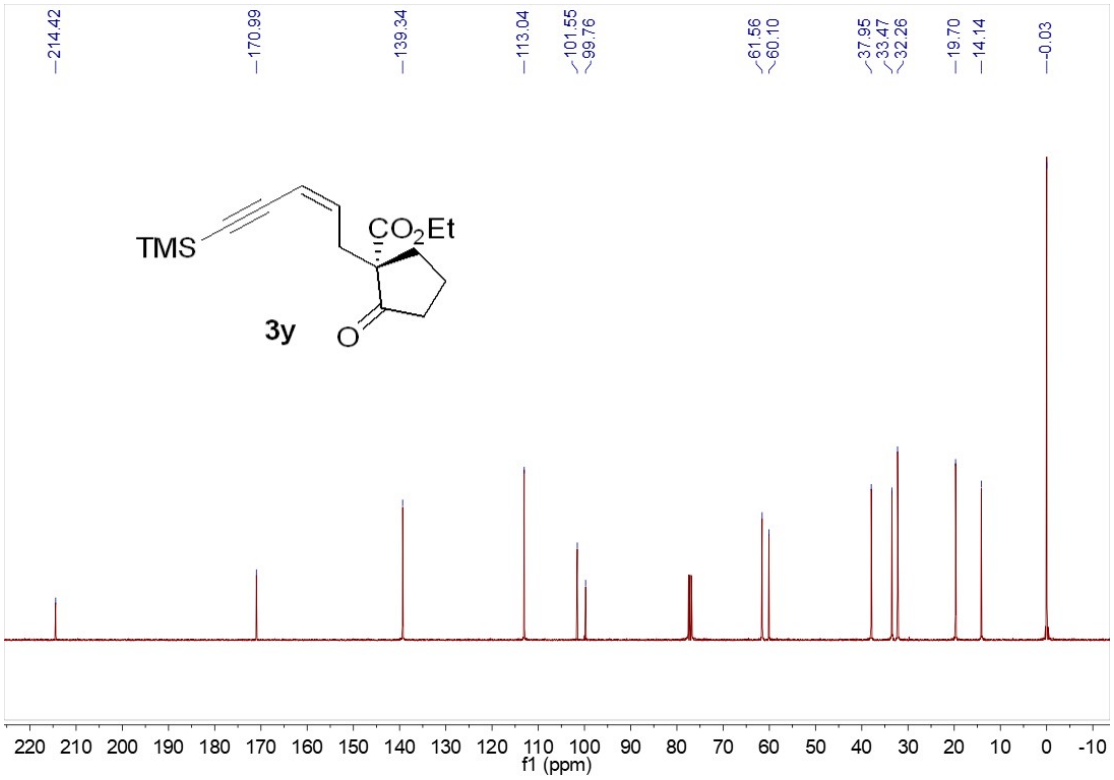

CO2Et
O
3w
1.00
0.99
2.01
0.97
1.96
1.10
0.96
4.06
5.07
3.09
4.99
f1 (ppm)

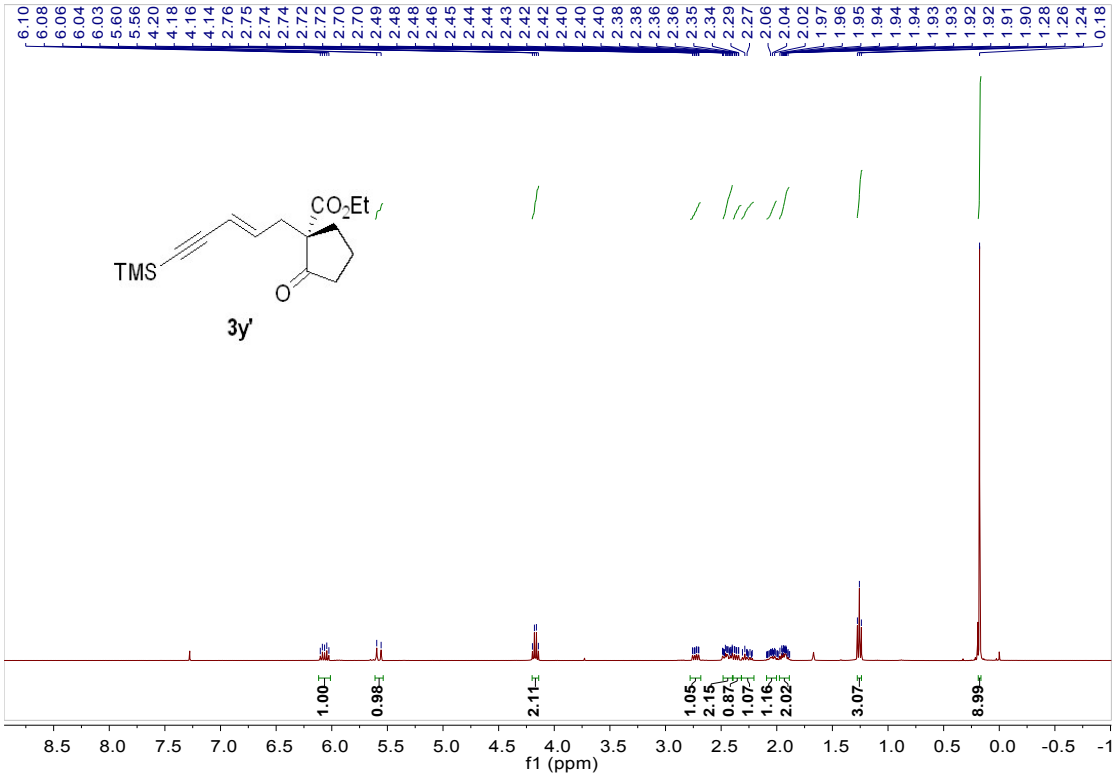

215.03
171.10
141.53
121.54
61.43
60.43
40.80
38.32
36.83
33.10
33.07
31.99
26.19
26.05
19.61
14.21
CO2Et
O
3w
1.00
f1 (ppm)

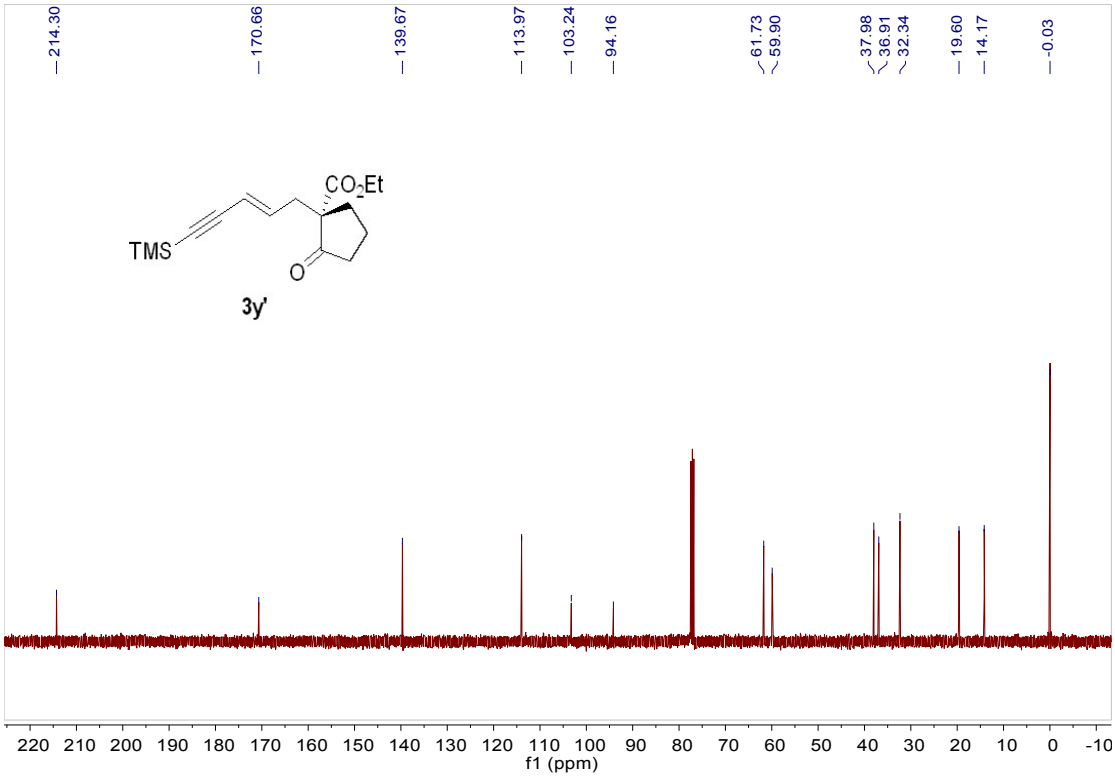

CO2Et
Me
O
3x
f1 (ppm)

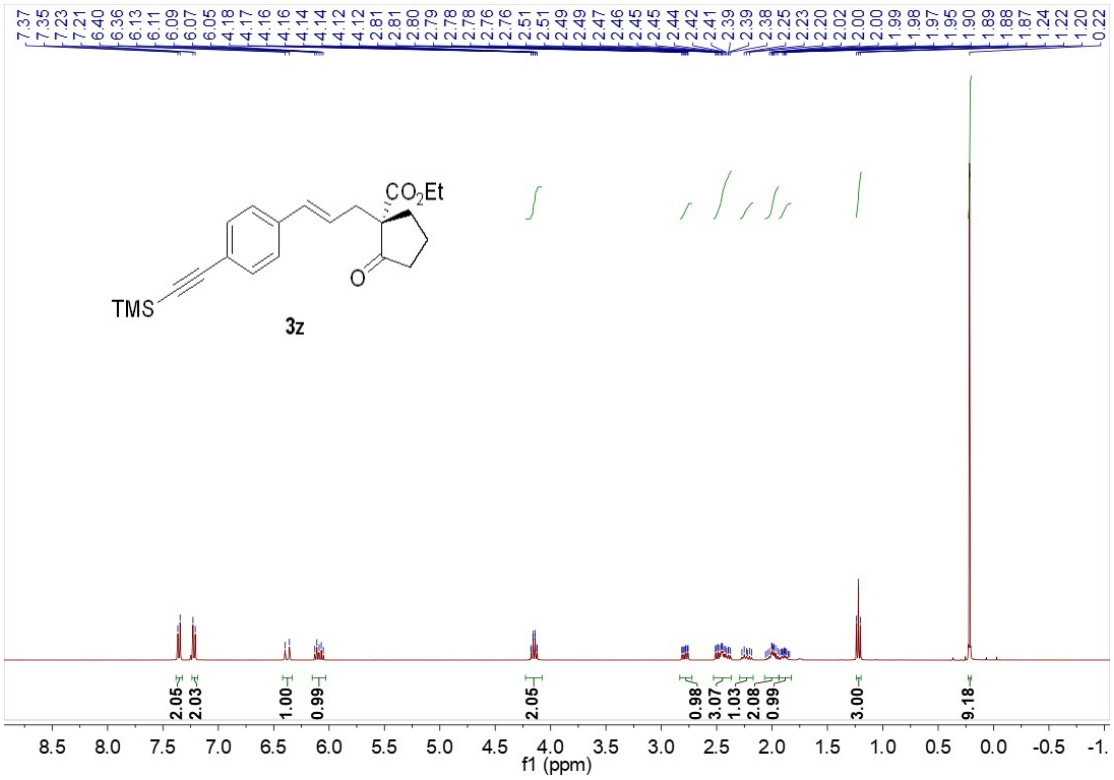

CO2Et
Me
O
3x
f1 (ppm)

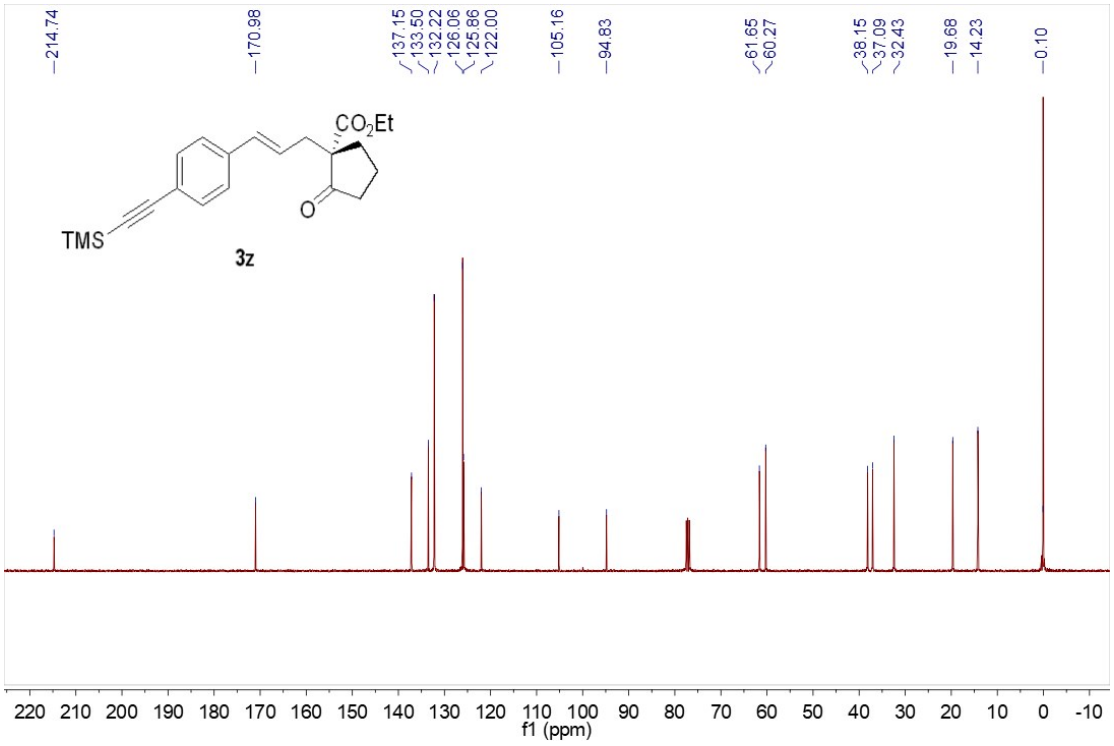

TMS
CO2Et
O
3y
f1 (ppm)

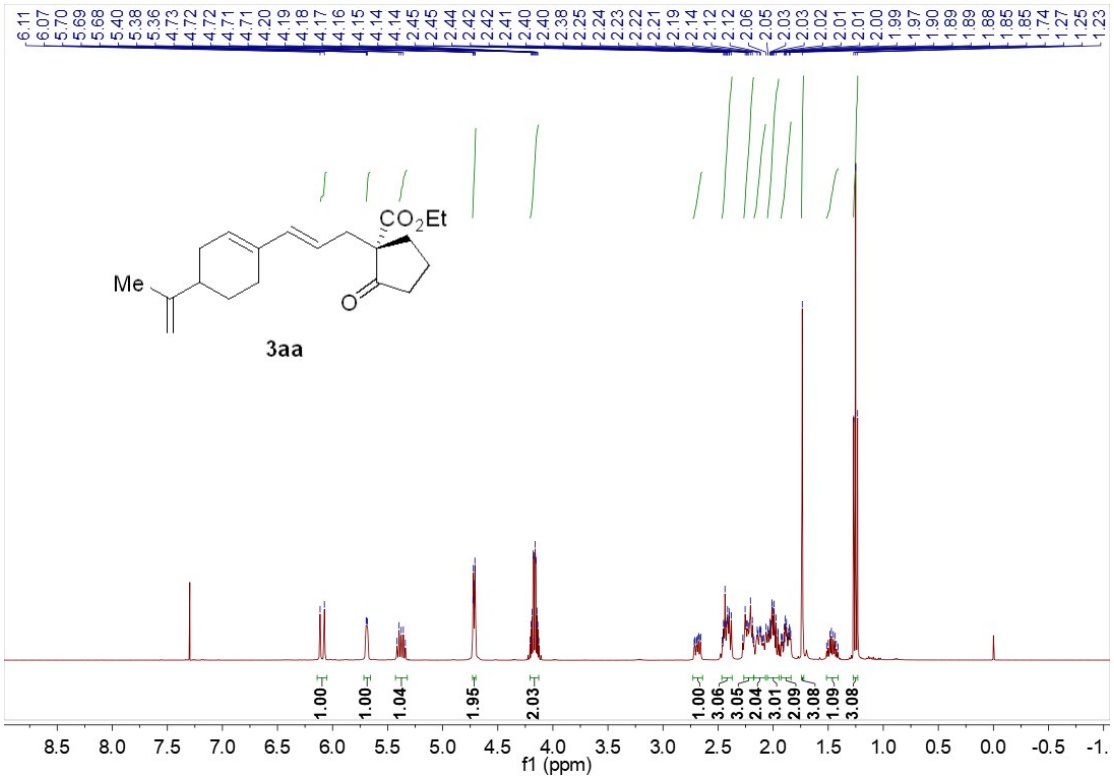

TMS
CO2Et
O
3y
f1 (ppm)

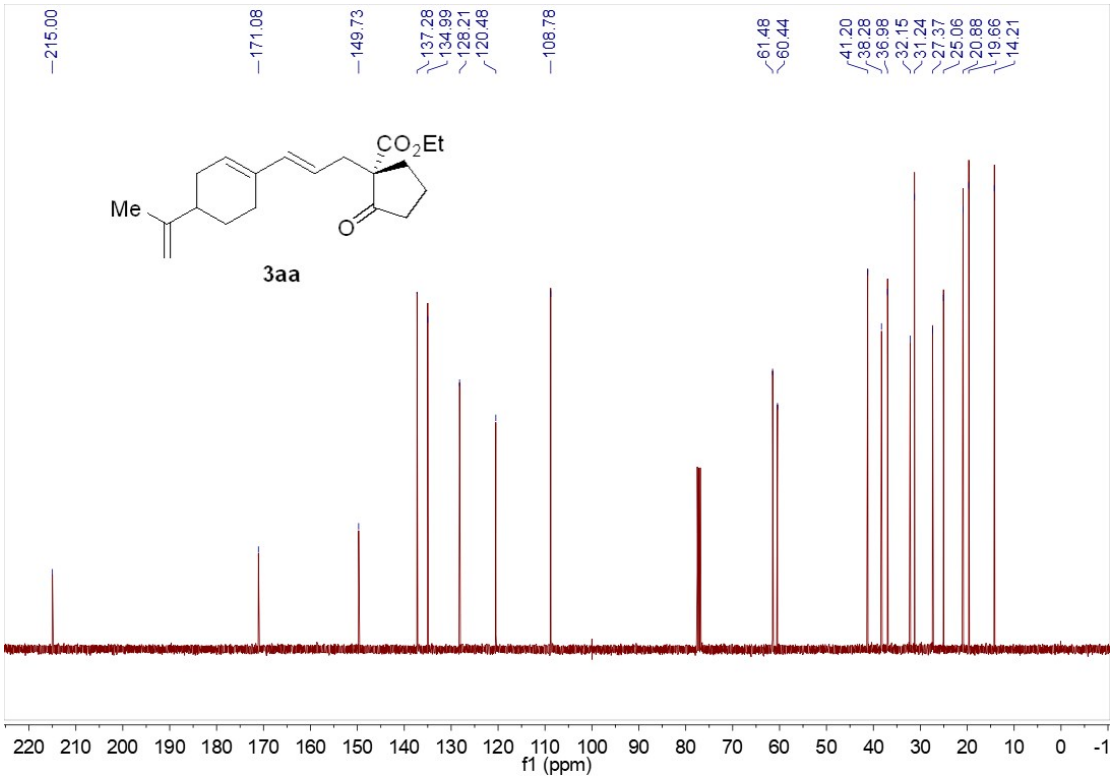

CO2Et
TMS
O
3y'
f1 (ppm)

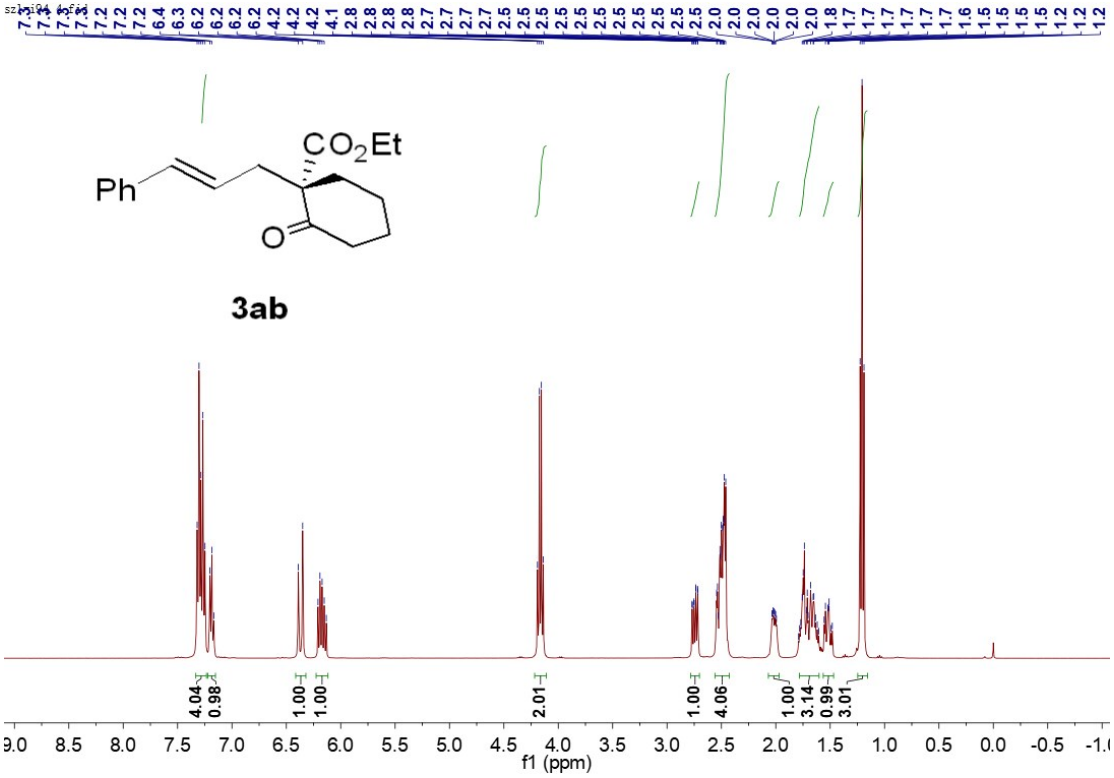

CO2Et
TMS
O
3y'
f1 (ppm)

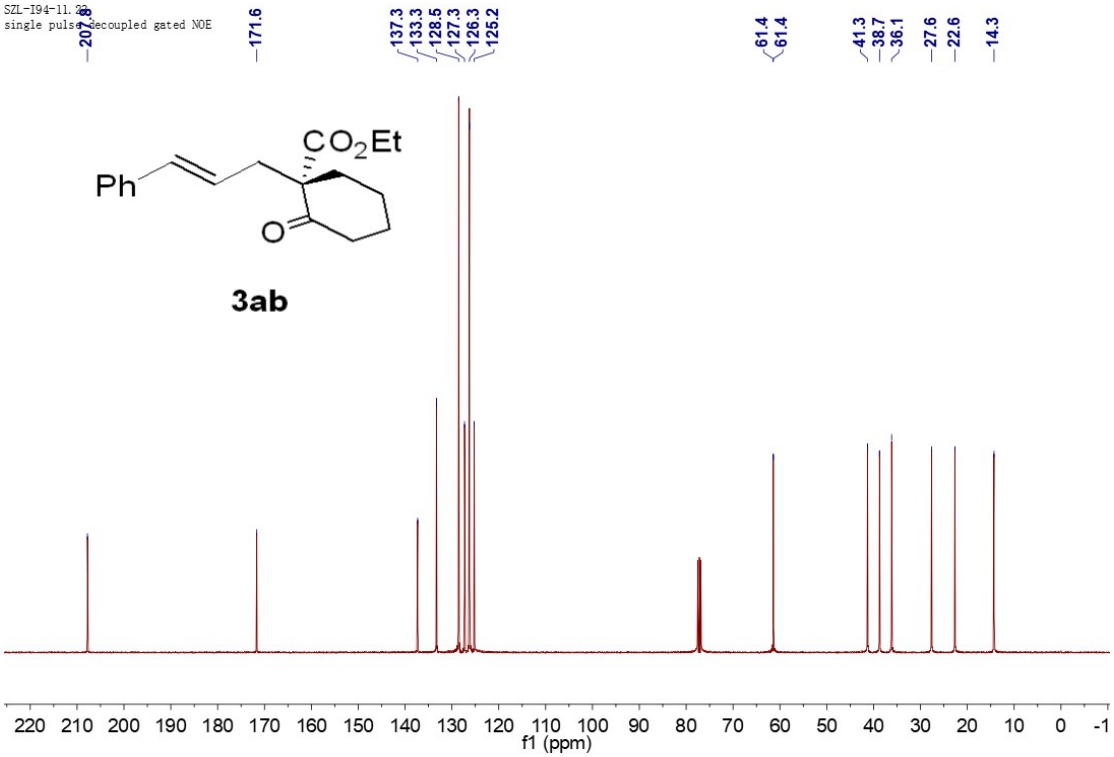

7.37 7.35 7.23 7.21 6.40 6.36 6.13 6.11 6.09 6.07 6.05 4.18 4.17 4.16 4.16 4.14 4.14 4.12 4.12 2.81 2.81 2.80 2.79 2.78 2.78 2.76 2.76 2.51 2.51 2.49 2.49 2.47 2.46 2.45 2.45 2.44 2.42 2.41 2.39 2.39 2.38 2.25 2.23 2.20 2.02 2.00 2.00 1.99 1.98 1.97 1.95 1.90 1.89 1.88 1.87 1.24 1.22 1.20 0.22
CO2Et
TMS
O
3z
2.05 2.03 1.00 0.99 2.05 0.98 3.07 1.03 2.08 0.99 3.00 9.18
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

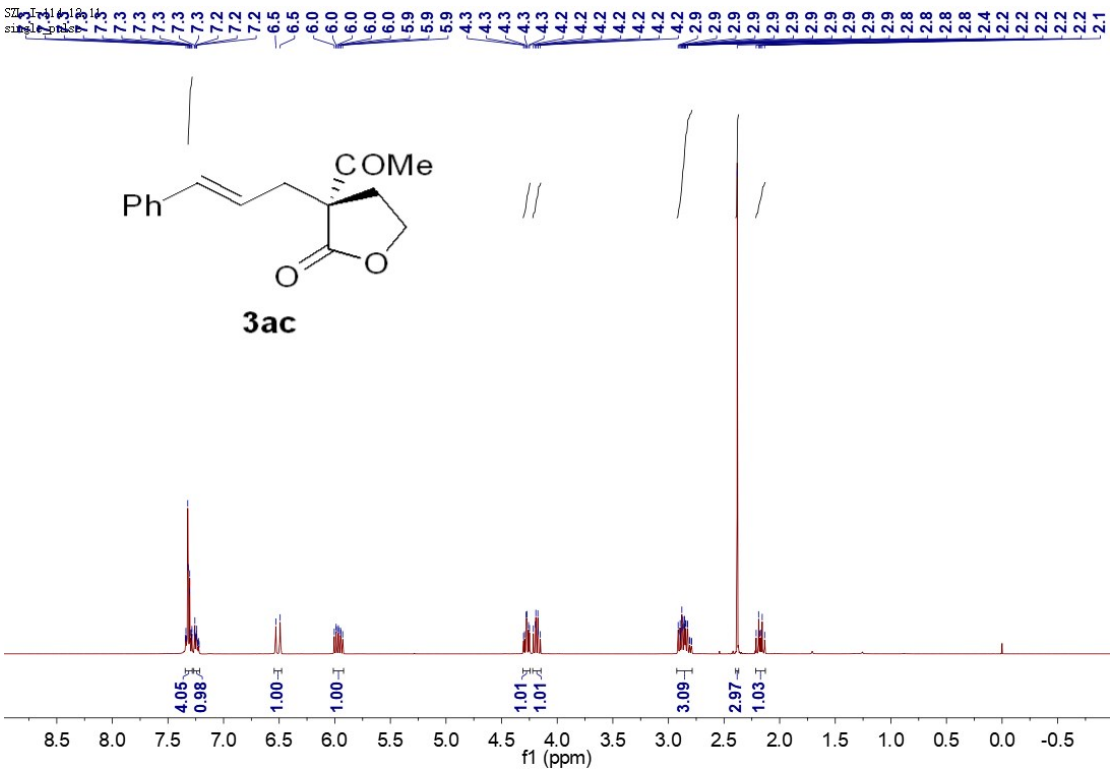

214.74 170.98 137.15 133.50 132.22 126.06 125.86 122.00 105.16 94.83 61.65 60.27 38.15 37.09 32.43 19.68 14.23 0.10
CO2Et
TMS
O
3z
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

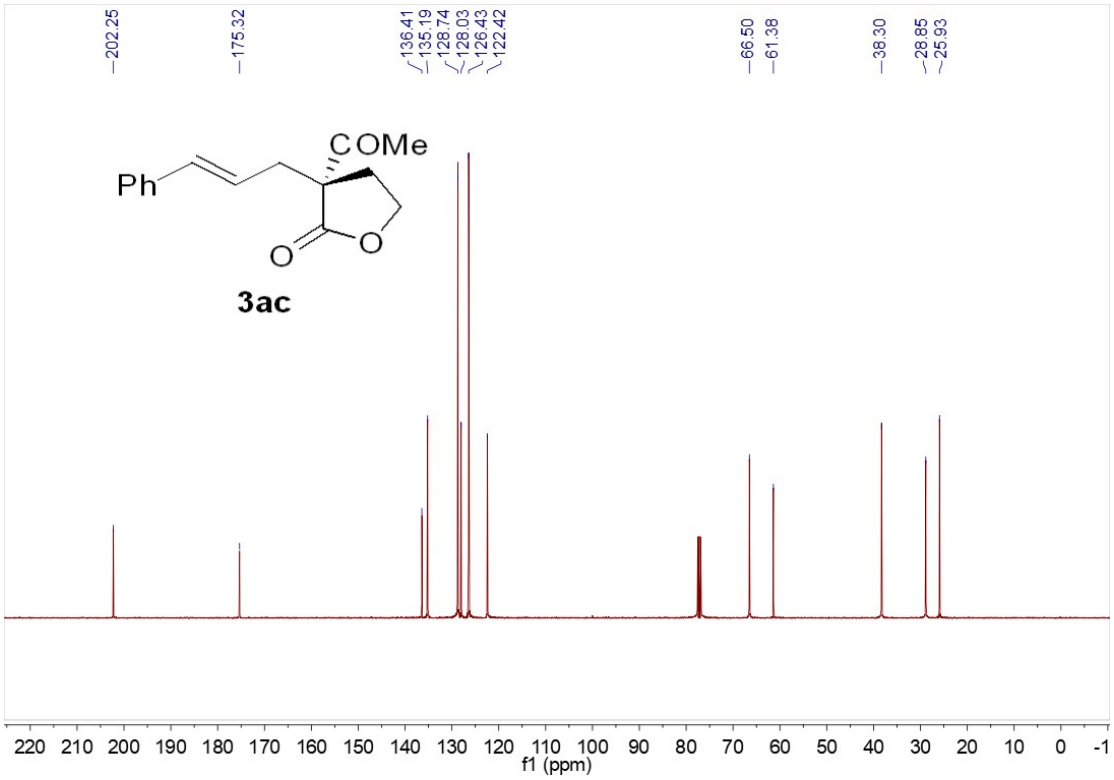

CO2Et
Me
O
3aa
f1 (ppm)

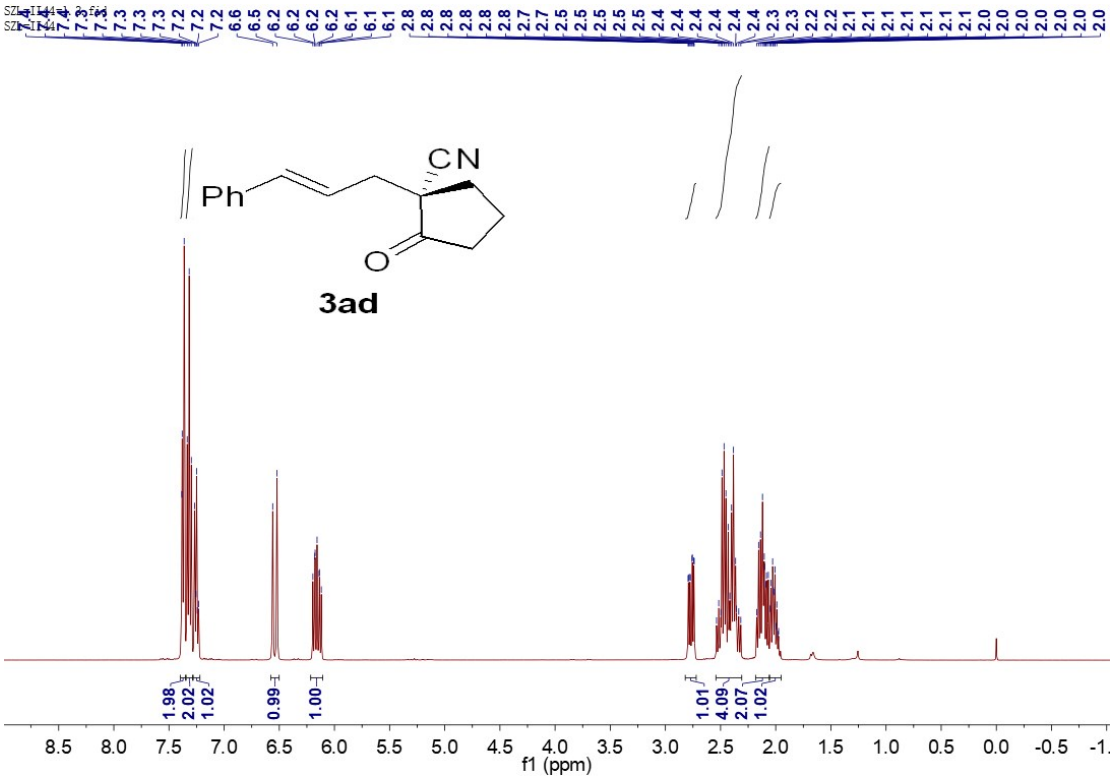

CO2Et
Me
O
3aa
f1 (ppm)


CO2Et
Ph
O
3ab
4.04
0.98
1.00
1.00
2.01
1.00
4.06
1.00
3.14
0.99
3.01
f1 (ppm)


SZL-I94-11.32
single pulse decoupled gated NOE
207.8
171.6
137.3
133.3
128.5
127.3
126.3
125.2
61.4
61.4
41.3
38.7
36.1
27.6
22.6
14.3
CO2Et
Ph
O
3ab
f1 (ppm)


COMe
Ph
O
O
3ac
4.05
0.98
1.00
1.00
1.01
1.01
3.09
2.97
1.03
f1 (ppm)


202.25
175.32
136.41
135.19
128.74
128.03
126.43
122.42
66.50
61.38
38.30
28.85
25.93
COMe
Ph
O
O
3ac
f1 (ppm)


CN
Ph
O
3ad
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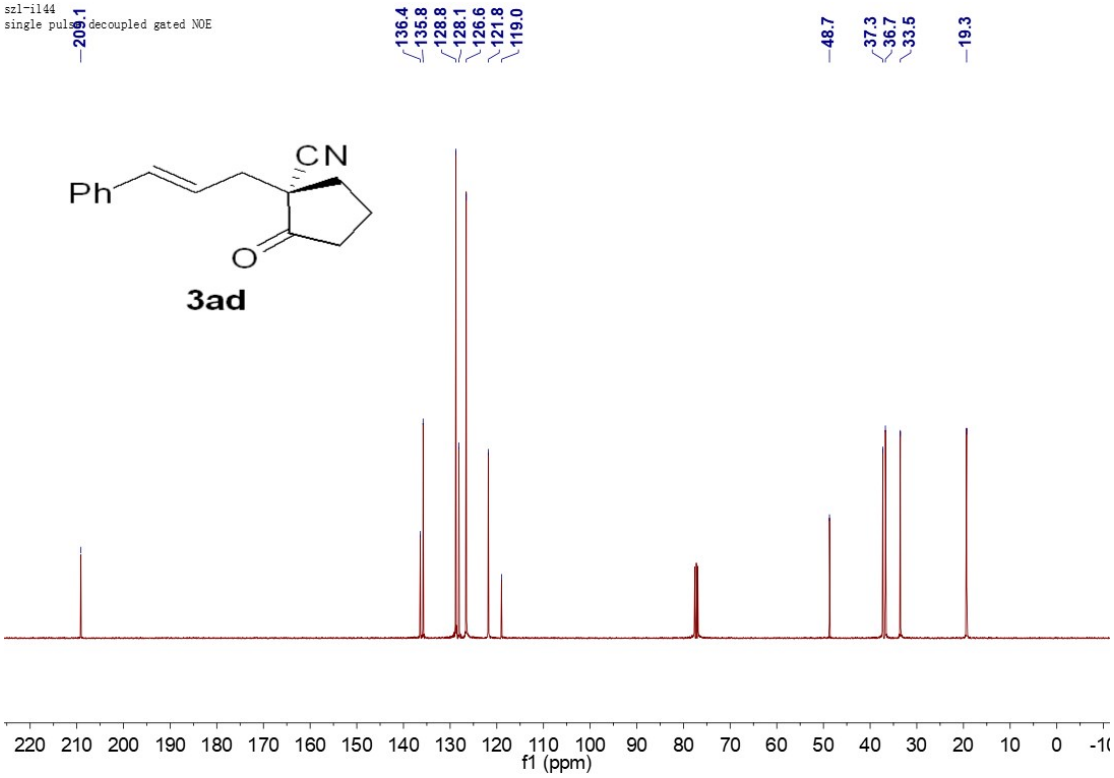
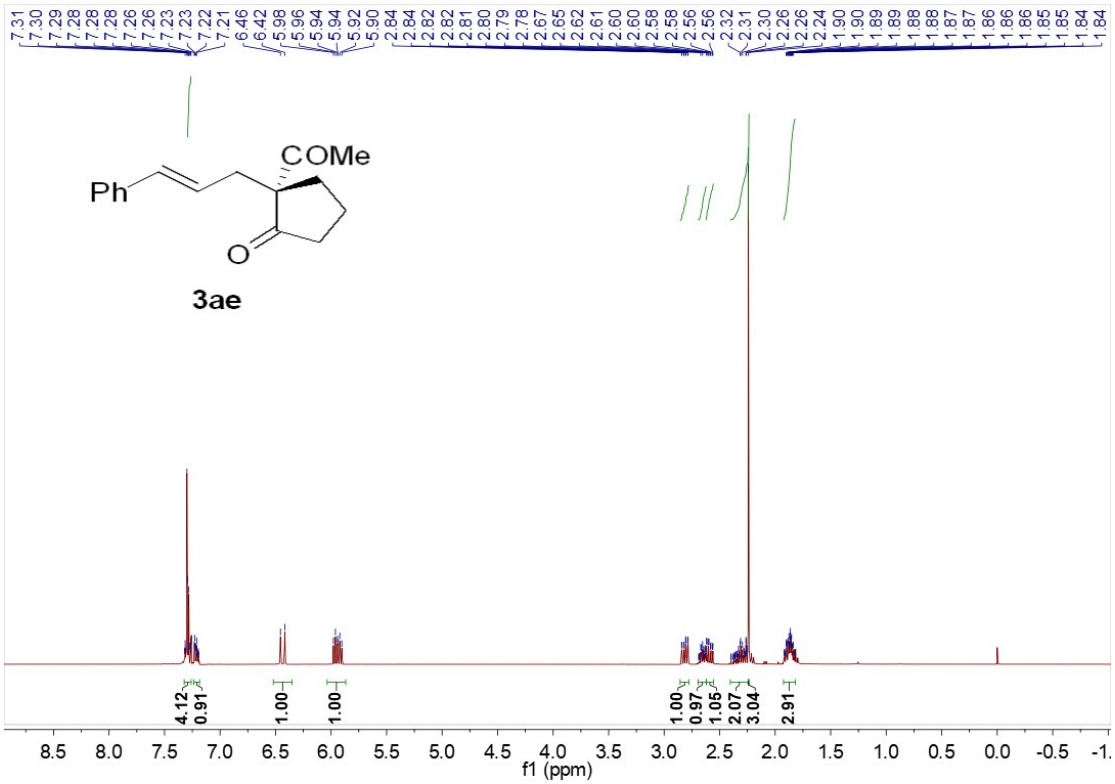
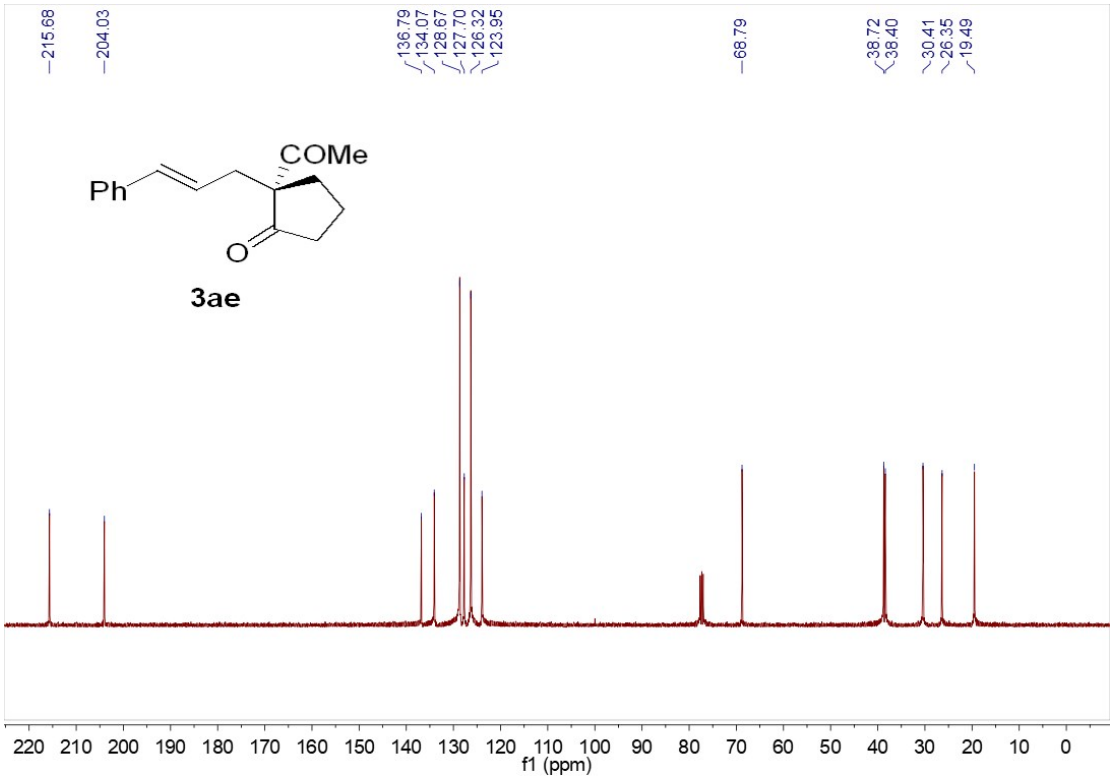
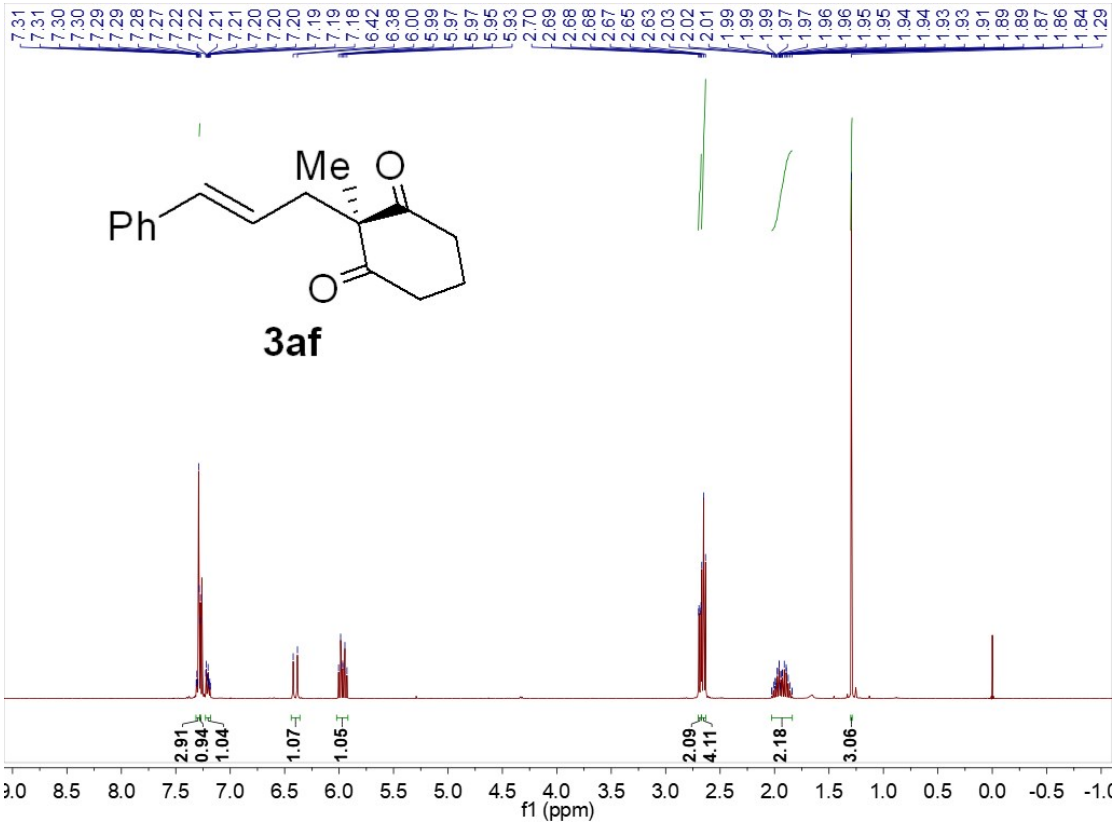
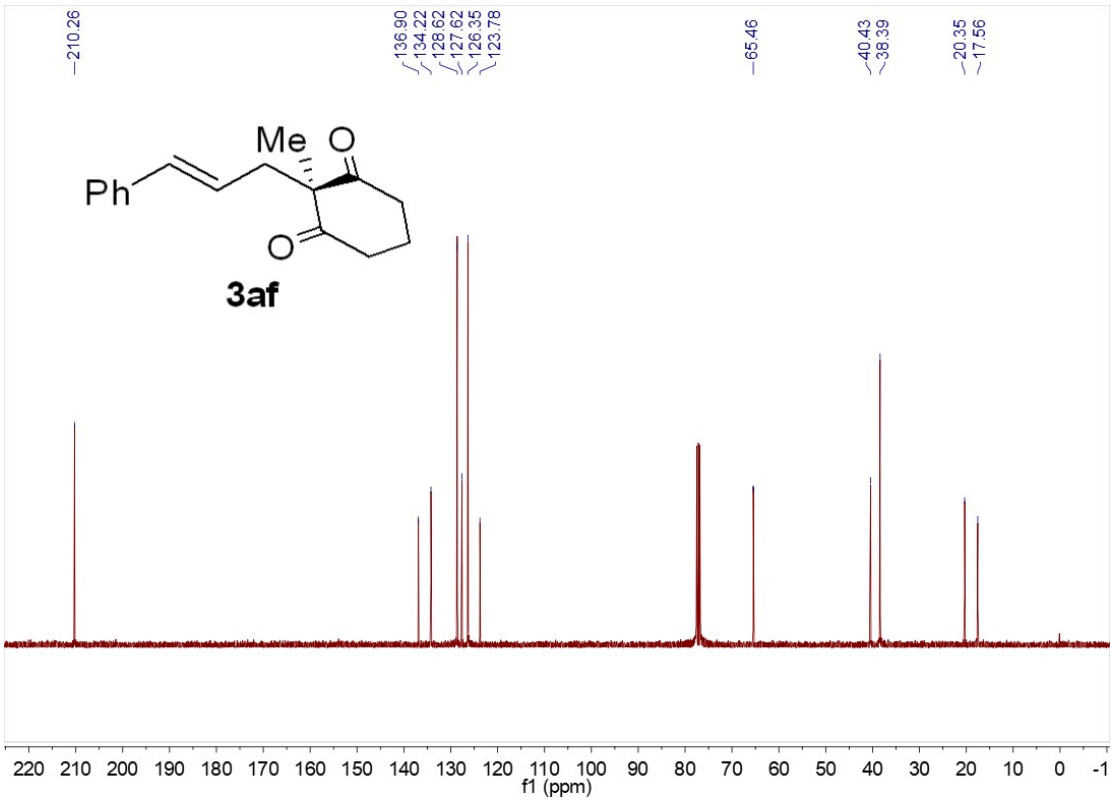
2.02
1.02
0.99
1.00
1.01
4.09
2.07
1.02
f1 (ppm)

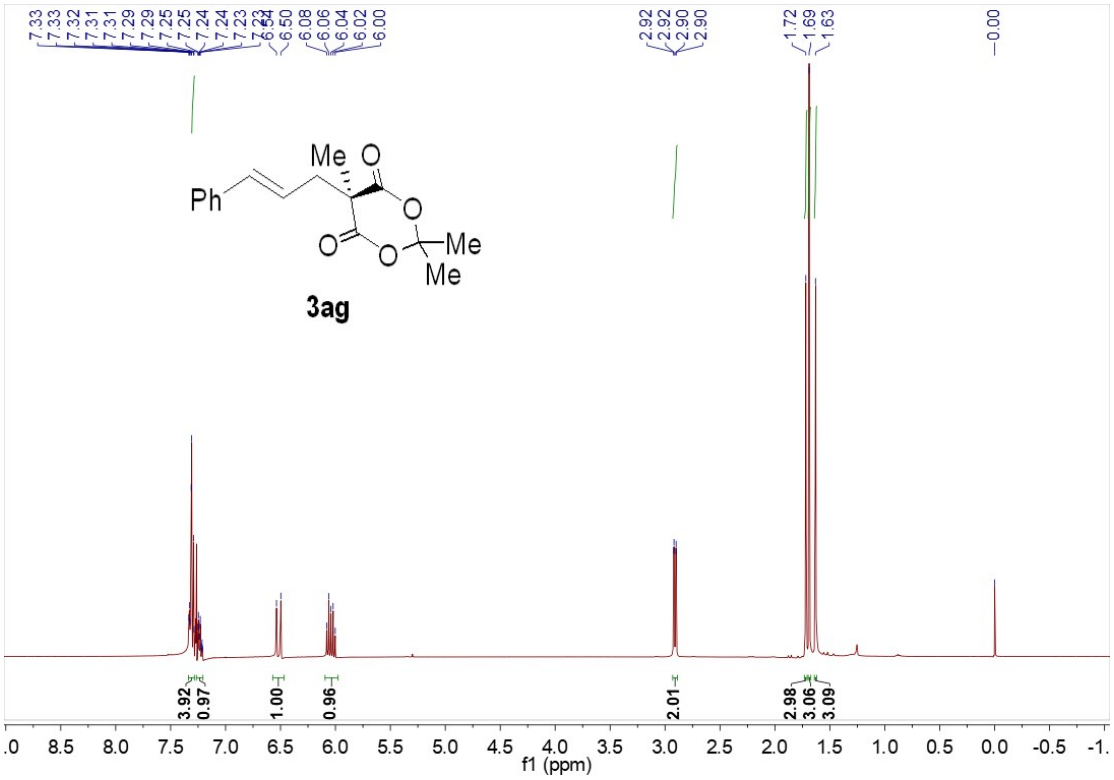

szl-il44
single pulse decoupled gated NOE
209.1
136.4
135.8
128.8
128.1
126.6
121.8
119.0
48.7
37.3
36.7
33.5
19.3
CN
Ph
O
3ad
f1 (ppm)

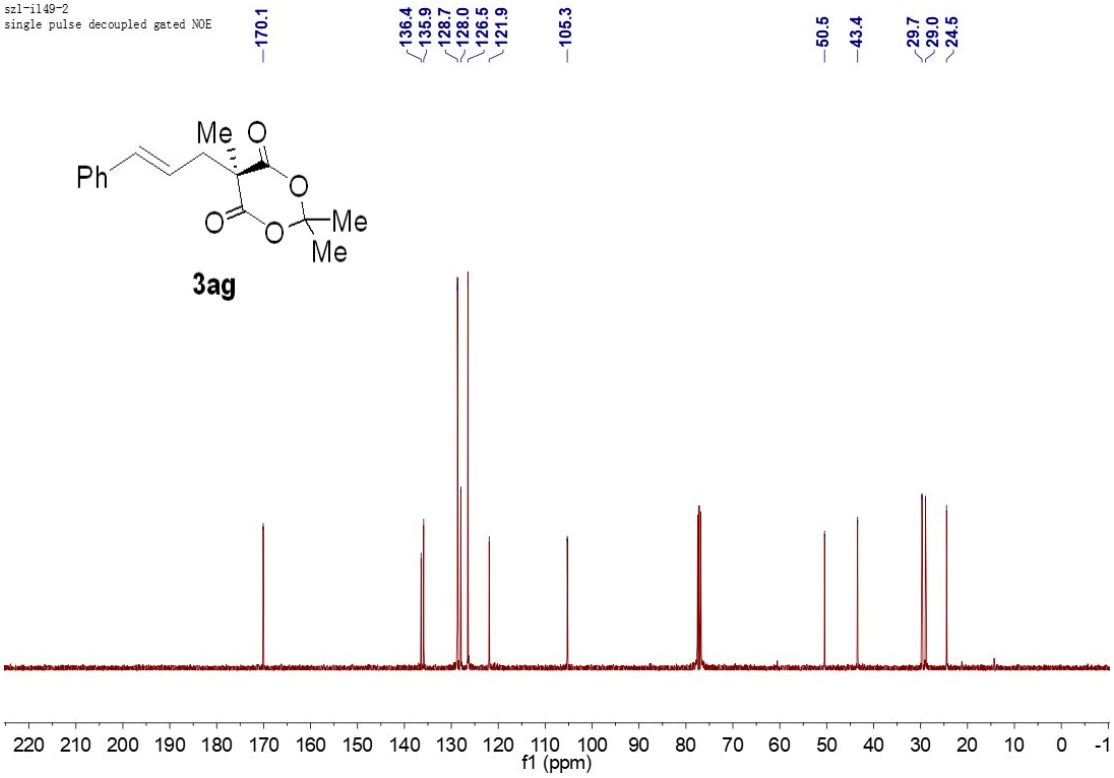

7.31 7.30 7.29 7.28 7.28 7.28 7.26 7.26 7.23 7.23 7.22 7.21 6.46 6.42 5.98 5.96 5.94 5.94 5.92 5.90 2.84 2.84 2.82 2.82 2.81 2.80 2.79 2.78 2.67 2.65 2.62 2.61 2.60 2.60 2.58 2.58 2.56 2.56 2.32 2.31 2.30 2.26 2.26 2.24 1.90 1.90 1.89 1.89 1.88 1.88 1.87 1.87 1.86 1.86 1.86 1.85 1.85 1.84 1.84
COMe
Ph
O
3ae
4.12
0.91
1.00
1.00
1.00
0.97
1.05
2.07
3.04
2.91
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

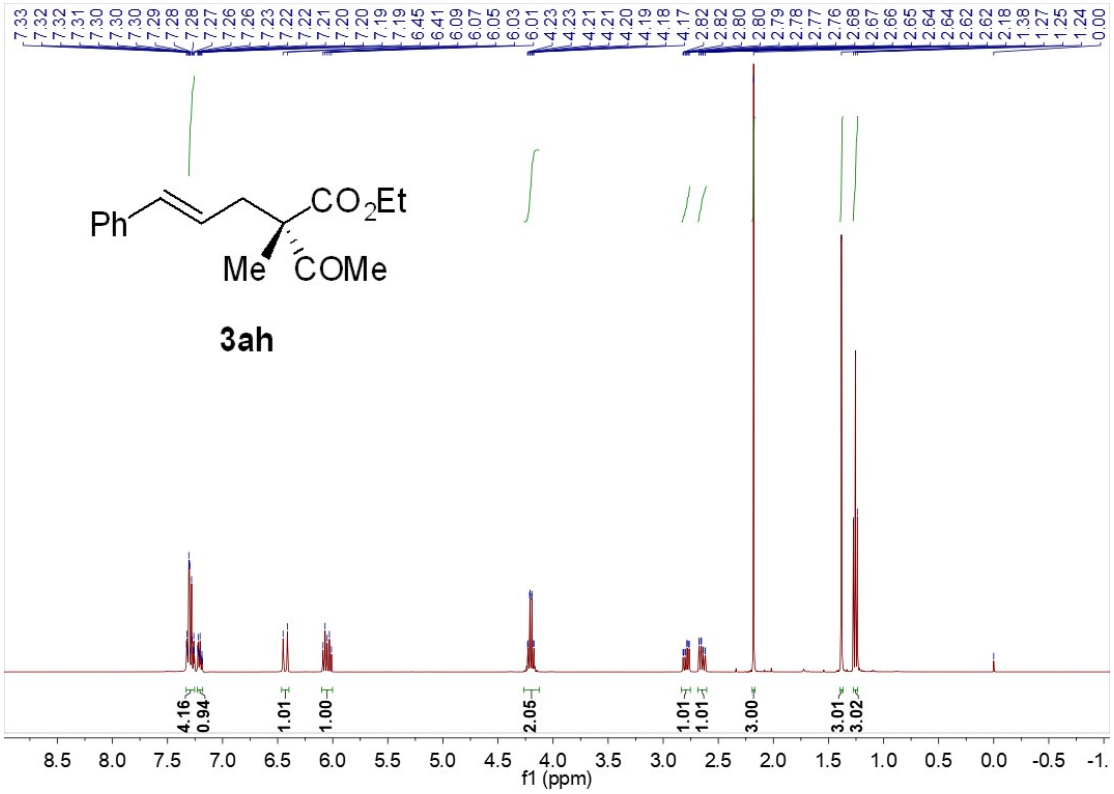

215.68
204.03
136.79
134.07
128.67
127.70
126.32
123.95
68.79
38.72
38.40
30.41
26.35
19.49
COMe
Ph
O
3ae
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

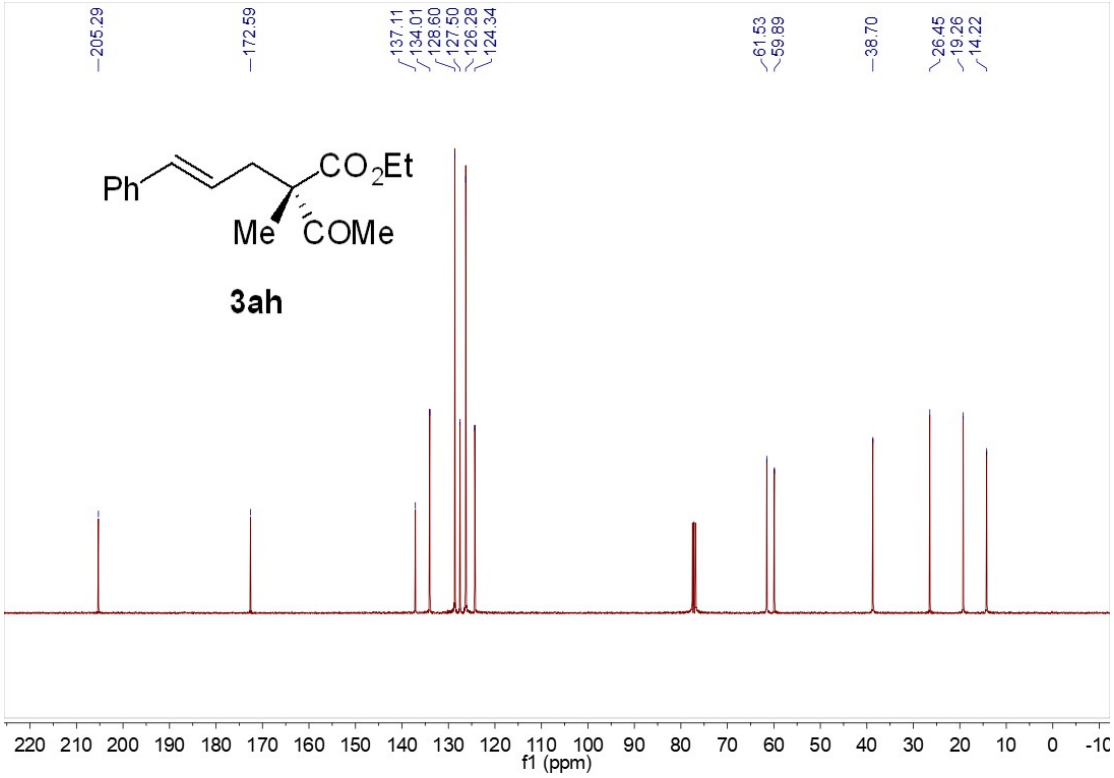

Me
O
Ph
O
3af
f1 (ppm)

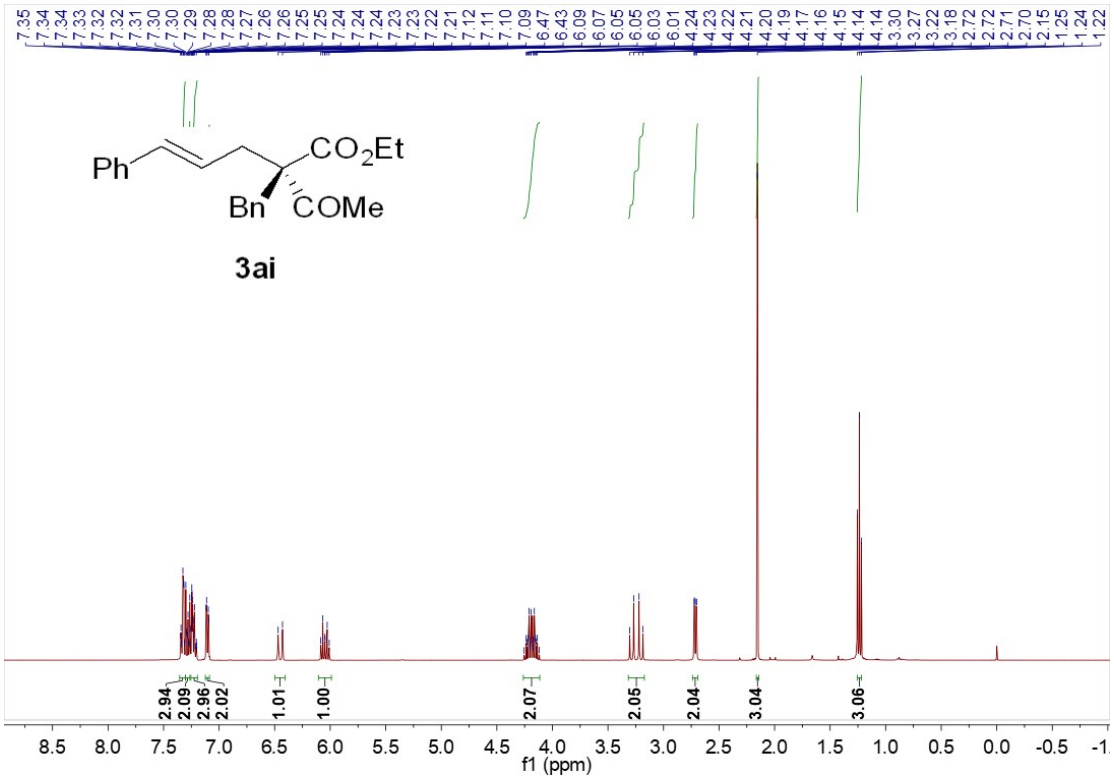

Me
O
Ph
O
3af
f1 (ppm)

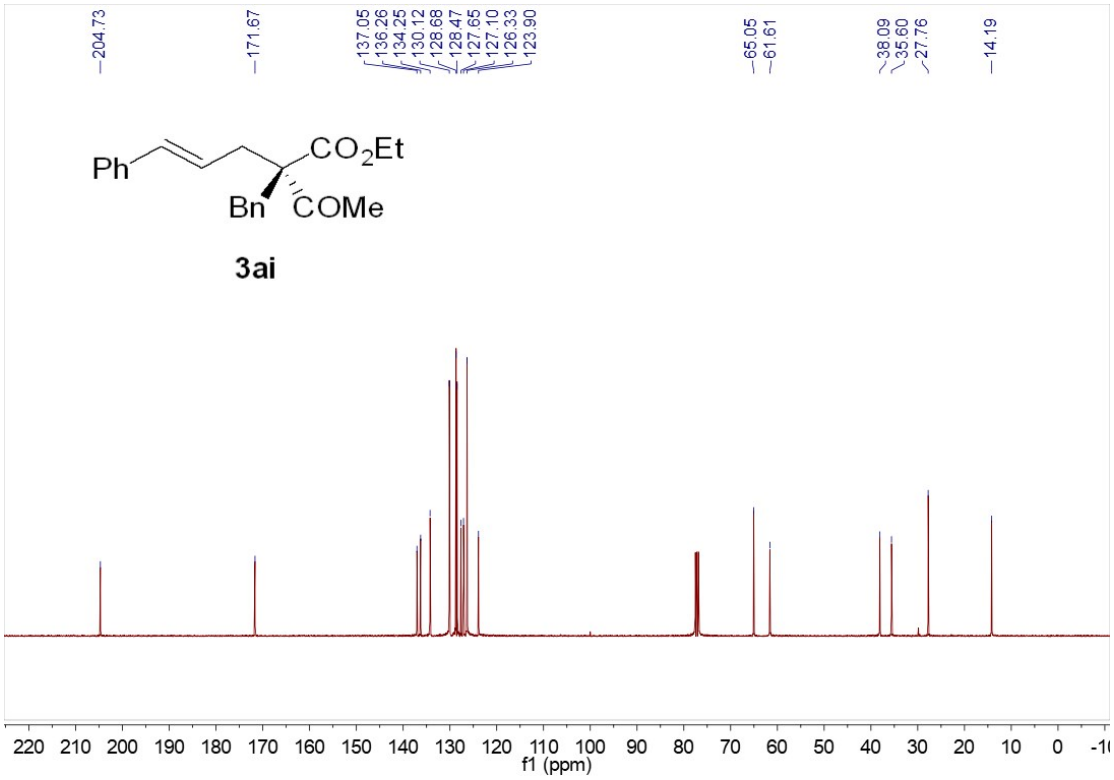

7.33
7.33
7.32
7.31
7.31
7.29
7.29
7.25
7.25
7.24
7.24
7.23
6.54
6.50
6.08
6.06
6.04
6.02
6.00
2.92
2.92
2.90
2.90
1.72
1.69
1.63
0.00
Me
O
Ph
O
O
O
Me
Me
3ag
3.92
0.97
1.00
0.96
2.01
2.98
3.06
3.09
f1 (ppm)

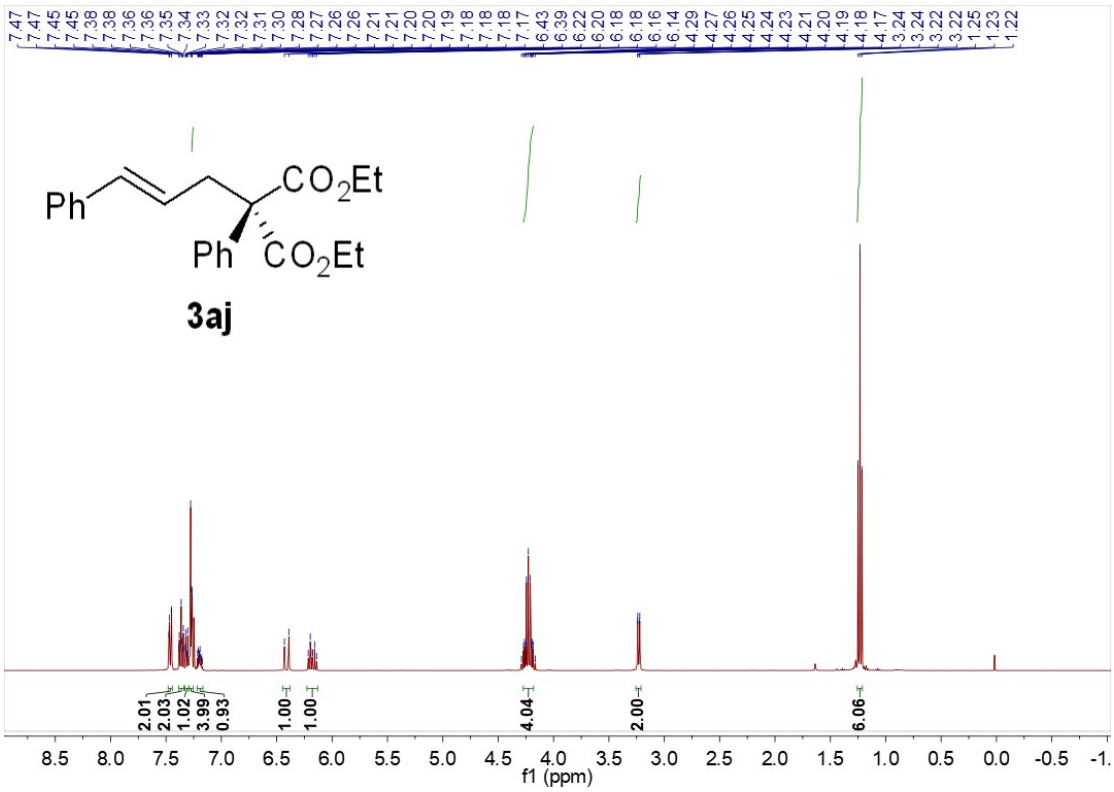

sz1-i149-2
single pulse decoupled gated NOE
170.1
136.4
135.9
128.7
128.0
126.5
121.9
105.3
50.5
43.4
29.7
29.0
24.5
Me
O
Ph
O
O
O
Me
Me
3ag
f1 (ppm)

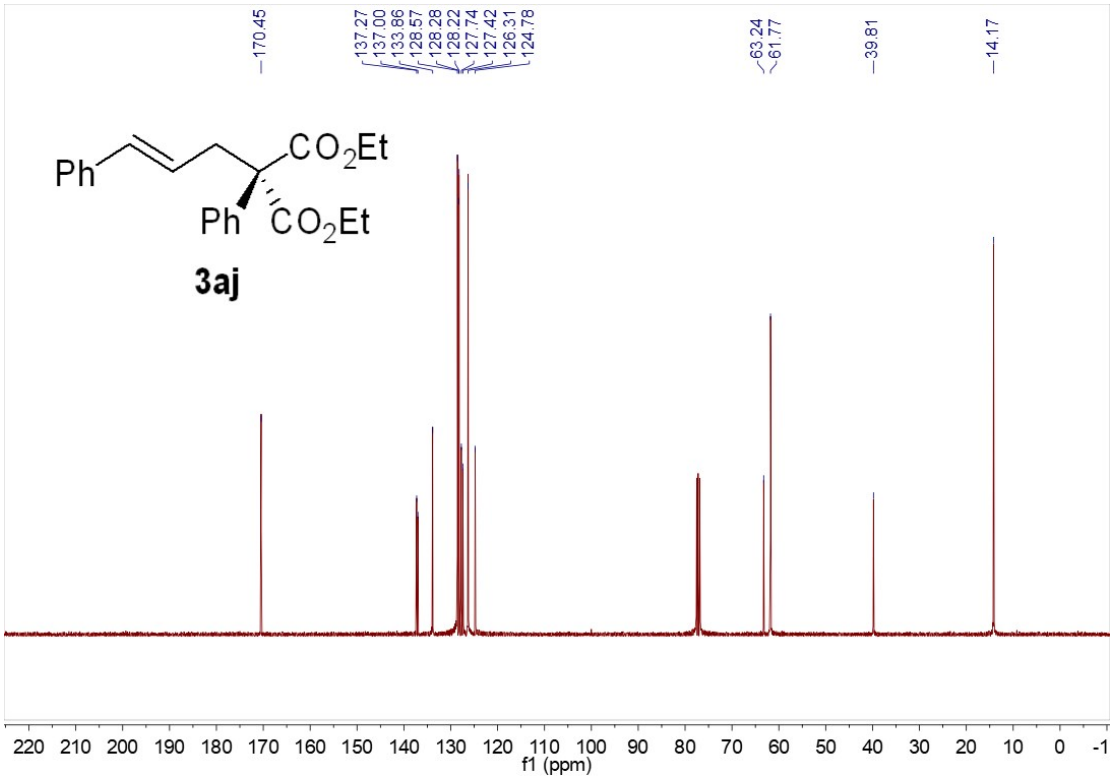

Ph
CO2Et
Me
COMe
3ah
f1 (ppm)

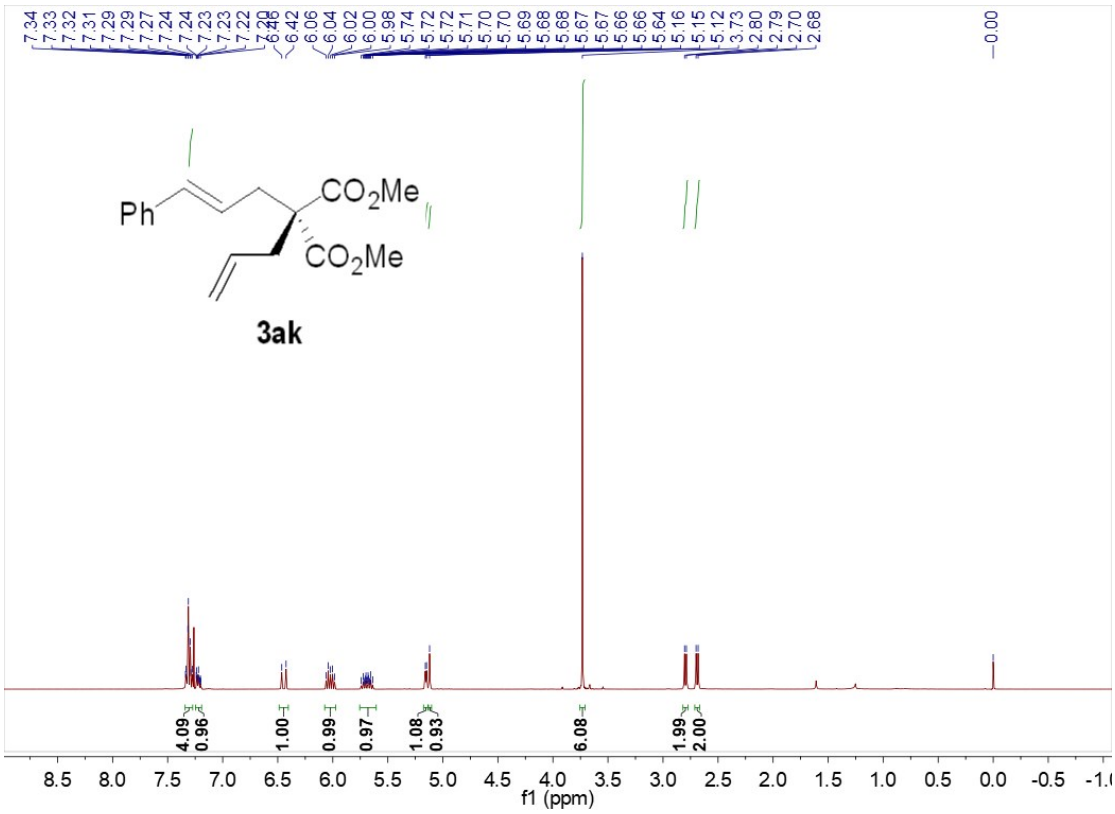

Ph
CO2Et
Me
COMe
3ah
f1 (ppm)

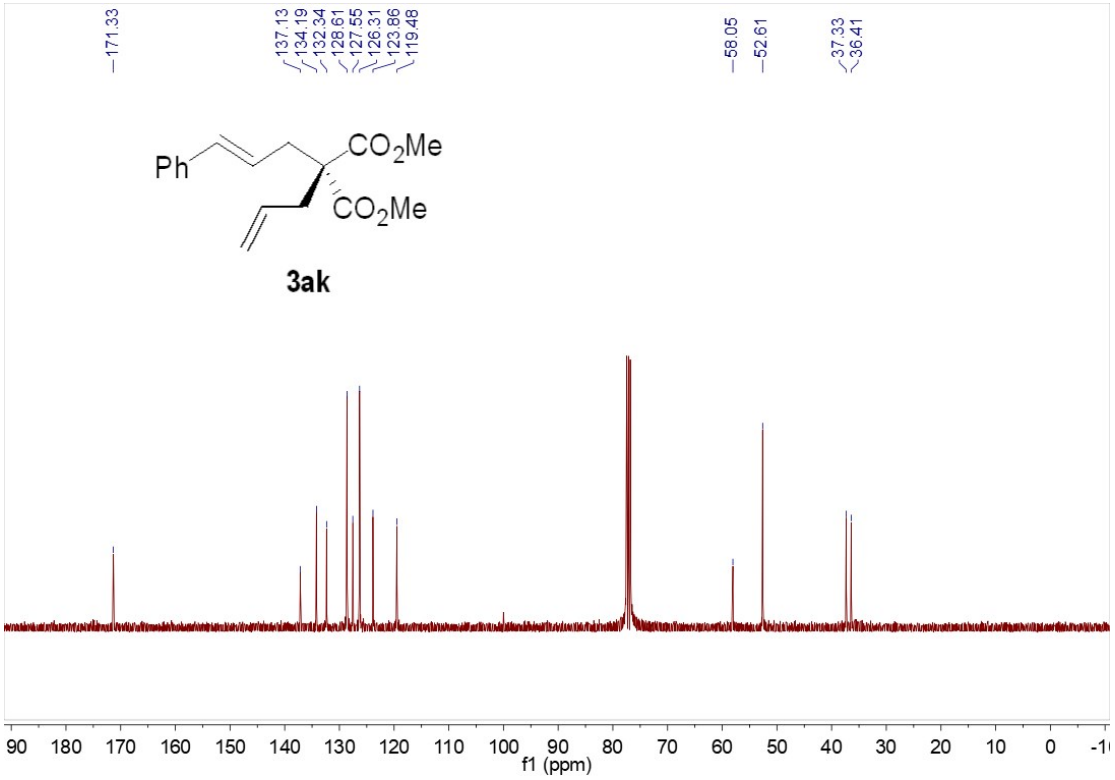

Ph
CO2Et
Bn
COMe
3ai
f1 (ppm)

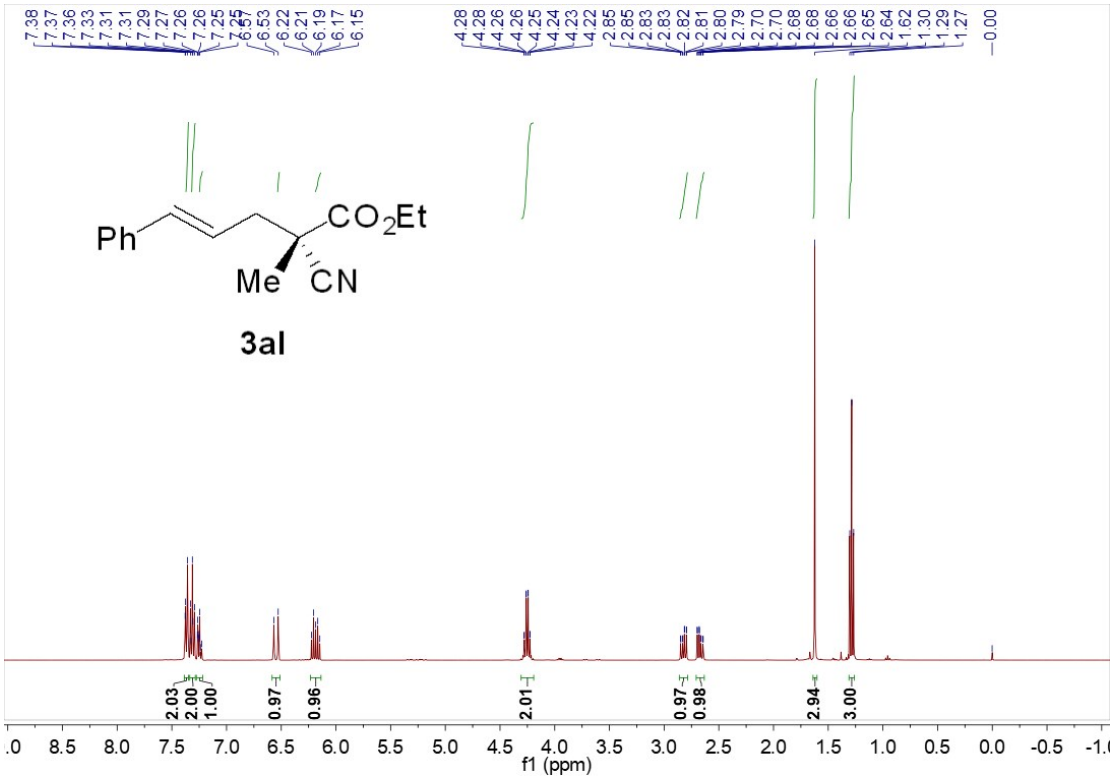

Ph
CO2Et
Bn
COMe
3ai
f1 (ppm)

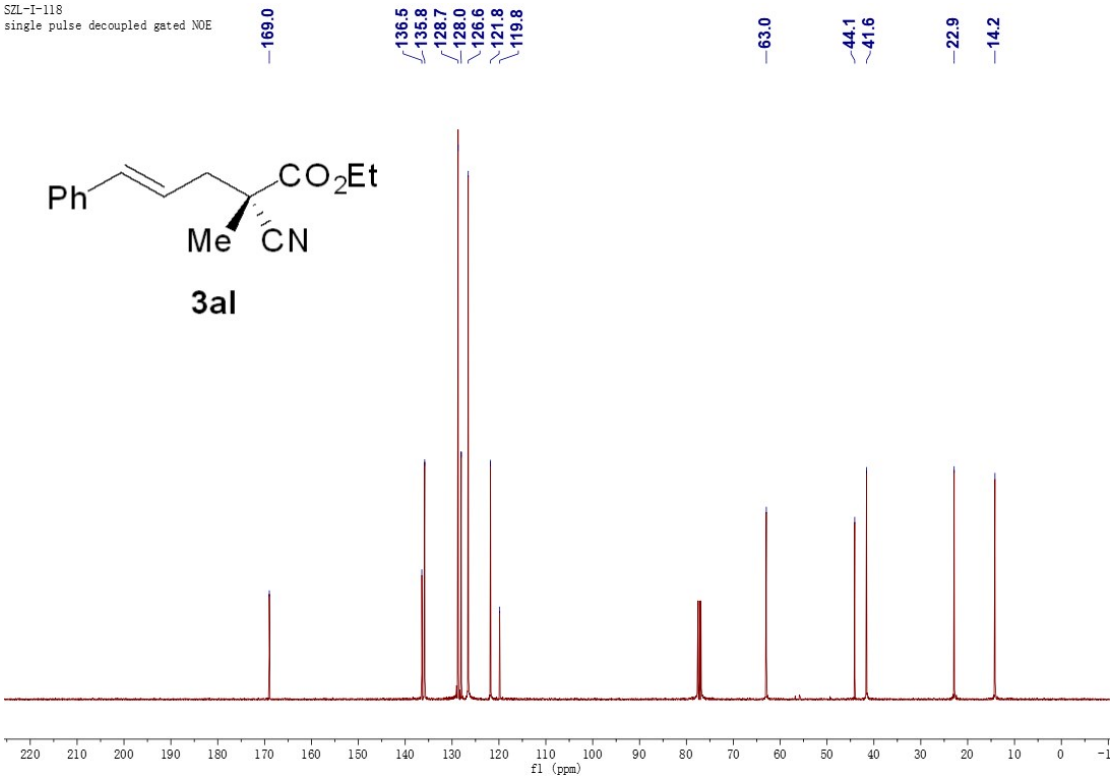

Ph
CO2Et
Ph
CO2Et
3aj
f1 (ppm)

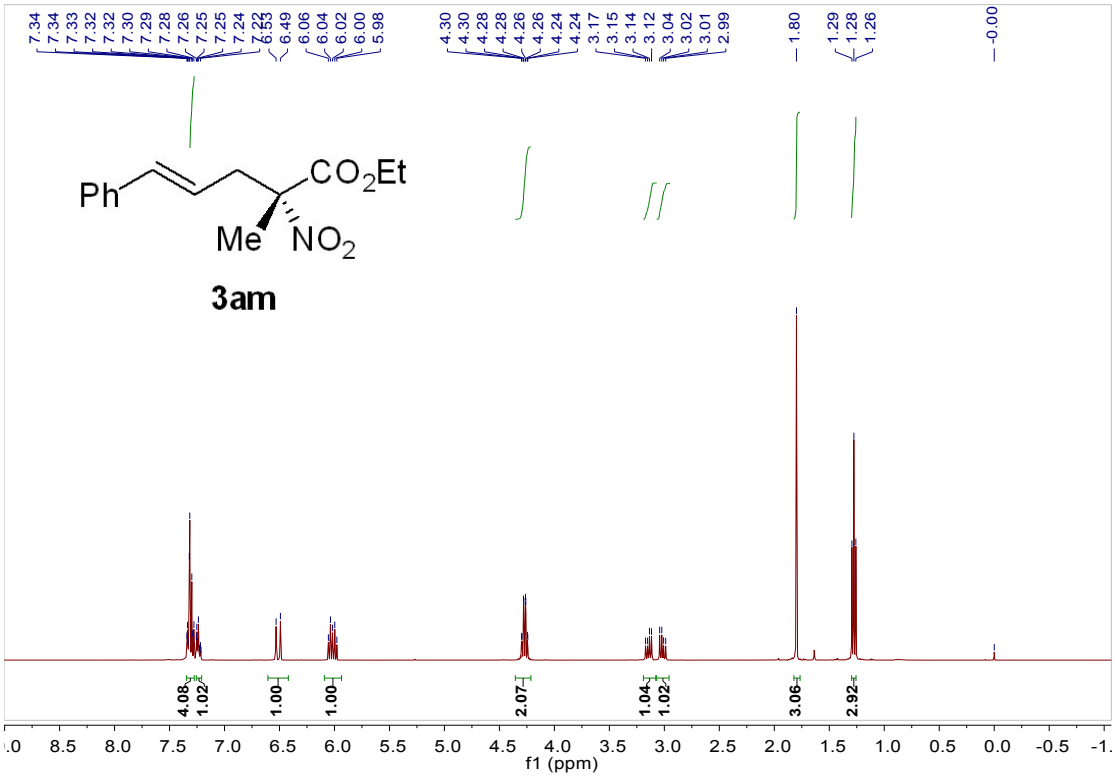

Ph
CO2Et
Ph
CO2Et
3aj
f1 (ppm)

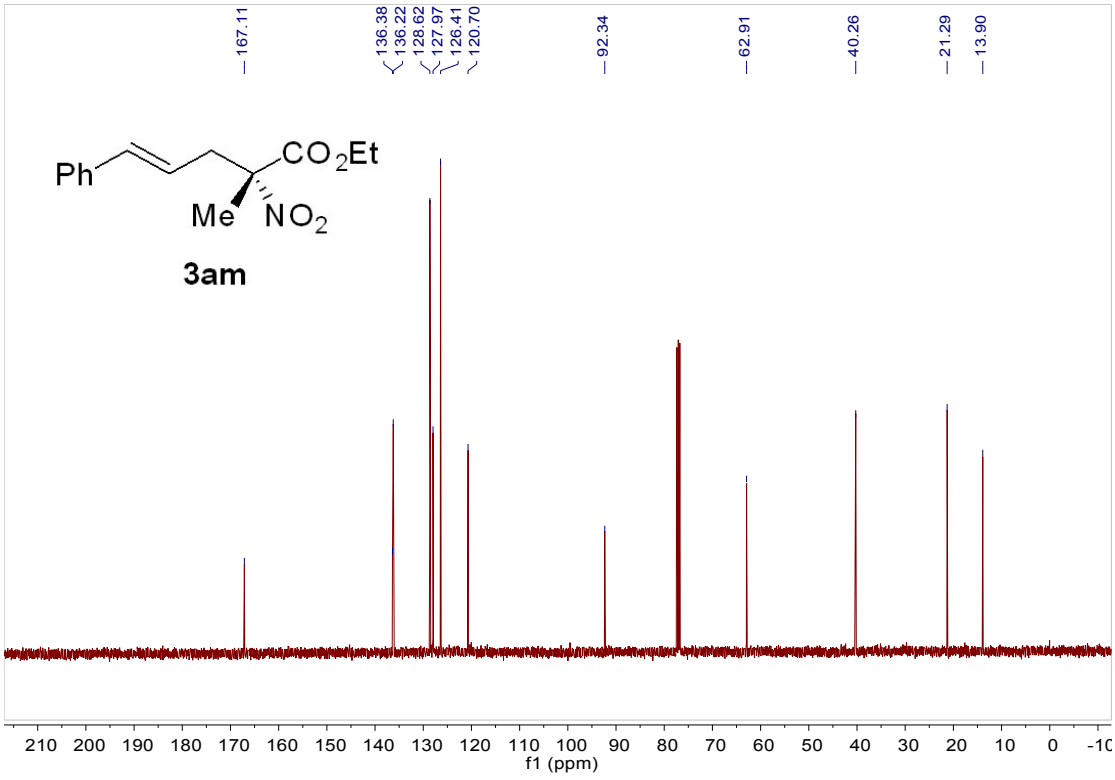

7.34
7.33
7.32
7.31
7.29
7.29
7.27
7.24
7.24
7.23
7.23
7.22
6.42
6.06
6.04
6.02
6.00
5.98
5.74
5.72
5.72
5.71
5.70
5.70
5.69
5.68
5.68
5.67
5.67
5.66
5.66
5.64
5.16
5.15
5.12
3.73
2.80
2.79
2.70
2.68
0.00
Ph
CO2Me
CO2Me
3ak
4.09
0.96
1.00
0.99
0.97
1.08
0.93
6.08
1.99
2.00
f1 (ppm)

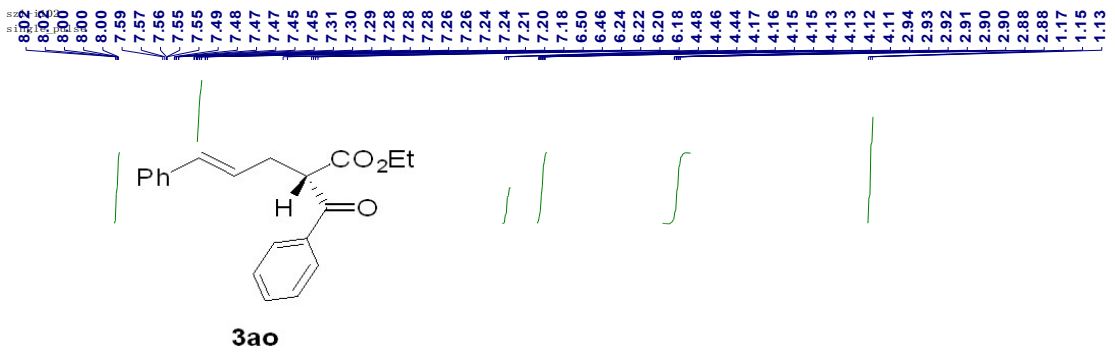

171.33
137.13
134.19
132.34
128.61
127.55
126.31
123.86
119.48
58.05
52.61
37.33
36.41
Ph
CO2Me
CO2Me
3ak
f1 (ppm)


Ph
CO2Et
Me
CN
3aI
f1 (ppm)

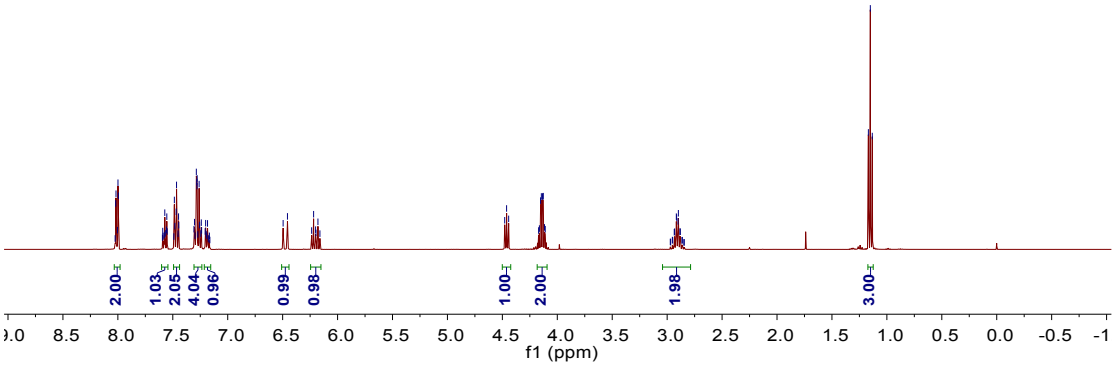

SZL-I-118
single pulse decoupled gated NOE
Ph
CO2Et
Me
CN
3aI
f1 (ppm)

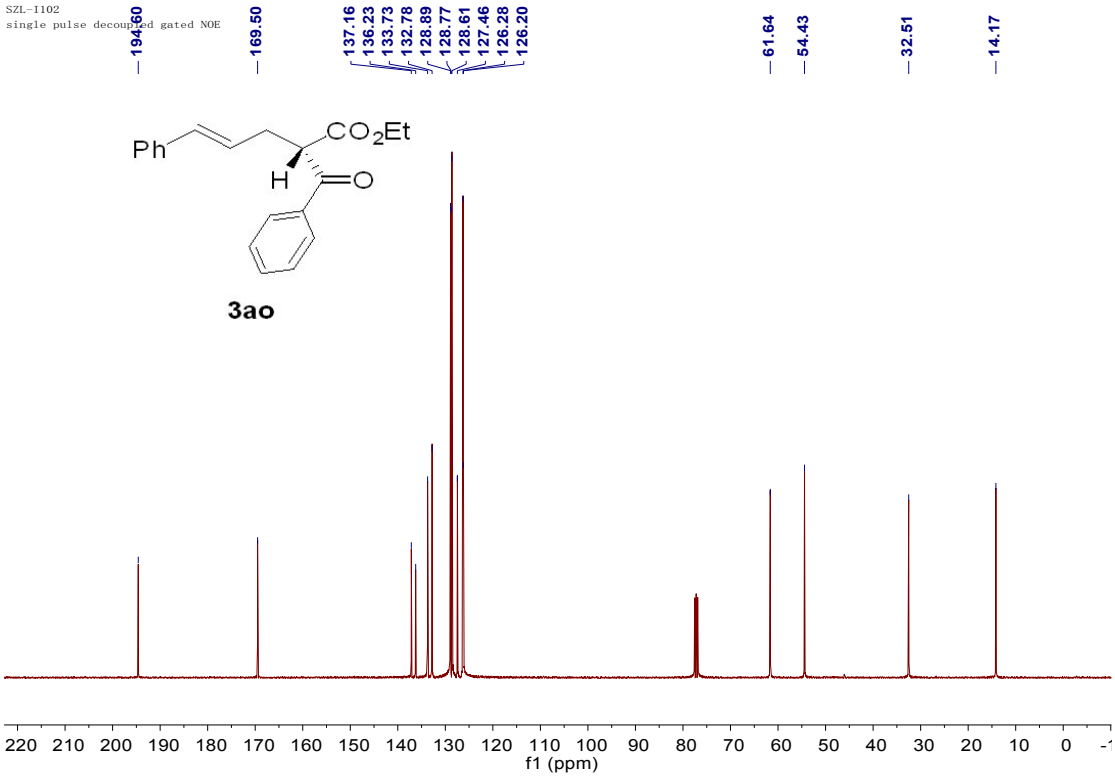

7.34
7.34
7.33
7.32
7.32
7.30
7.29
7.28
7.26
7.25
7.25
7.24
6.49
6.06
6.04
6.02
6.00
5.98
4.30
4.30
4.28
4.28
4.26
4.26
4.24
4.24
3.17
3.15
3.14
3.12
3.04
3.02
3.01
2.99
1.80
1.29
1.28
1.26
-0.00
Ph
CO2Et
Me
NO2
3am
4.08
1.02
1.00
1.00
2.07
1.04
1.02
3.06
2.92
f1 (ppm)


167.11
136.38
136.22
128.62
127.97
126.41
120.70
92.34
62.91
40.26
21.29
13.90
Ph
CO2Et
Me
NO2
3am
f1 (ppm)

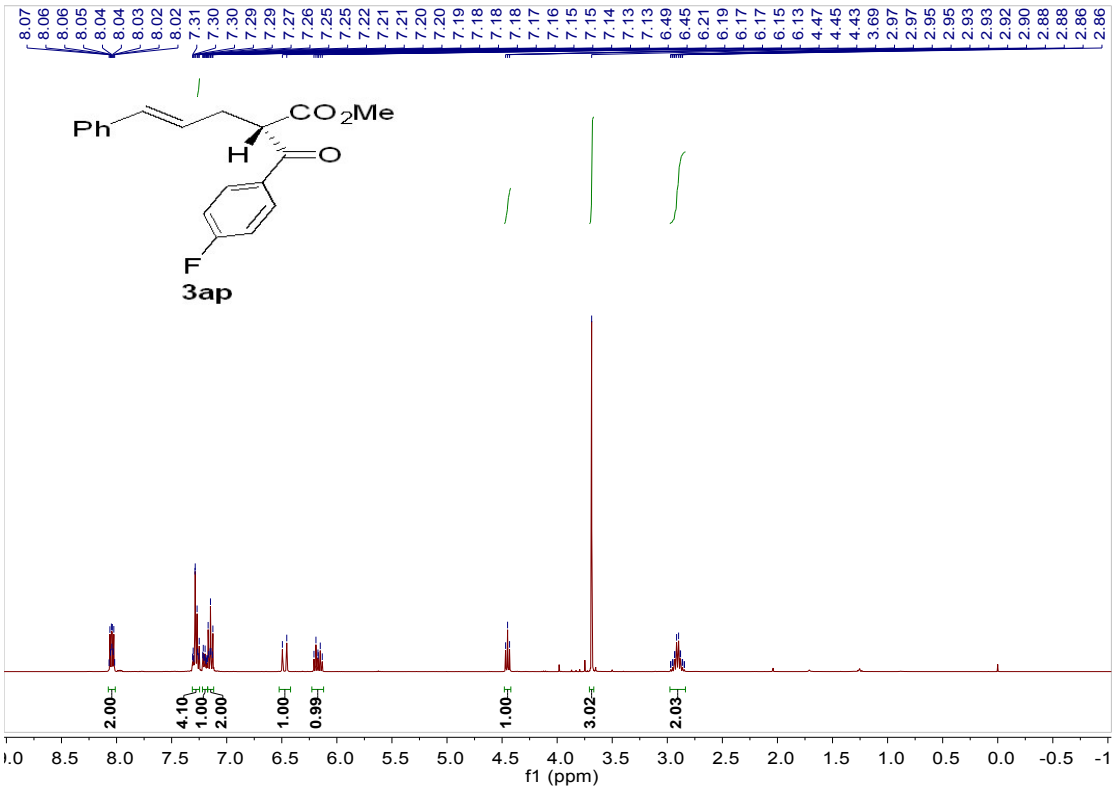

Ph
CO2Et
H
O

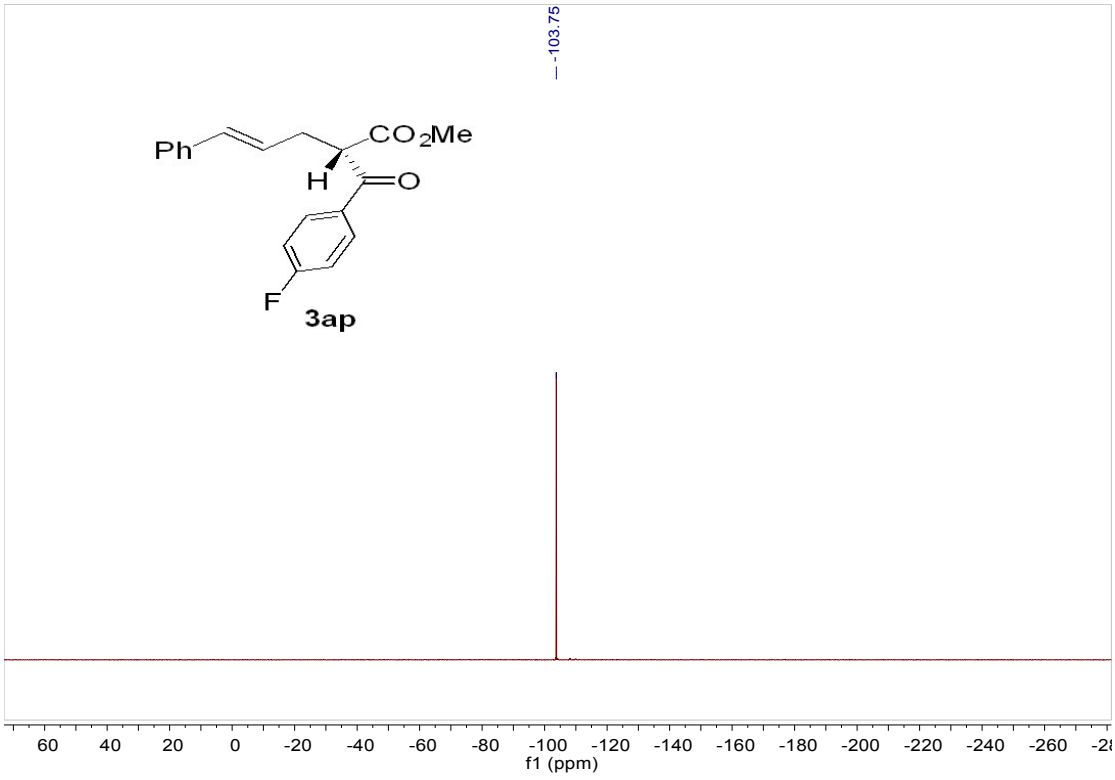
3ao

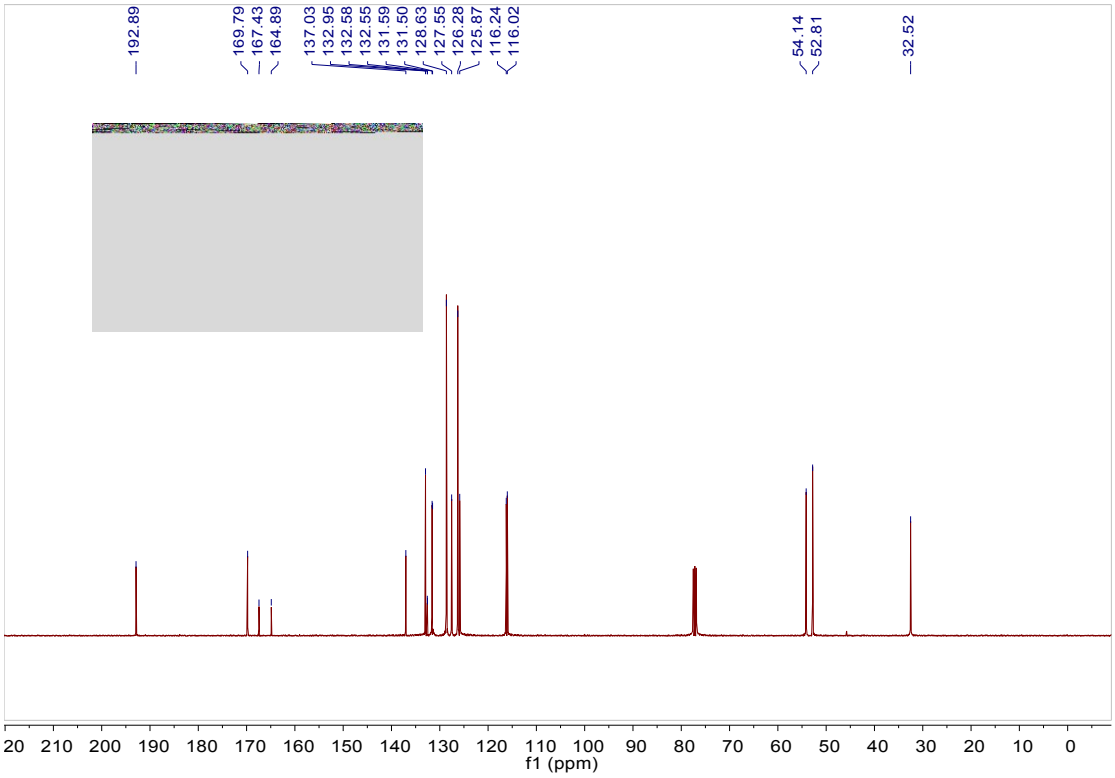

2.00
1.03
2.05
4.04
0.96
0.99
0.98
1.00
2.00
1.98
3.00
f1 (ppm)

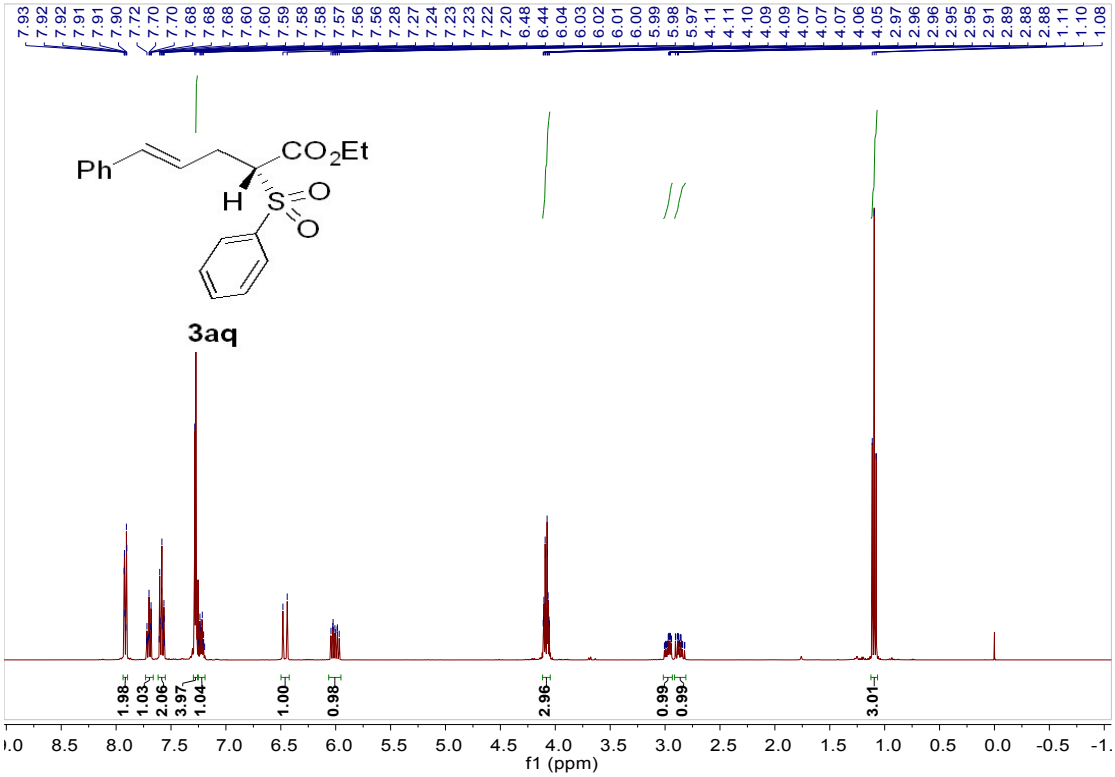

SZL-I102
single pulse decoupled gated NOE
194.60
169.50
137.16
136.23
133.73
132.78
128.89
128.77
128.61
127.46
126.28
126.20
61.64
54.43
32.51
14.17
Ph
CO2Et
H
O
f1 (ppm)

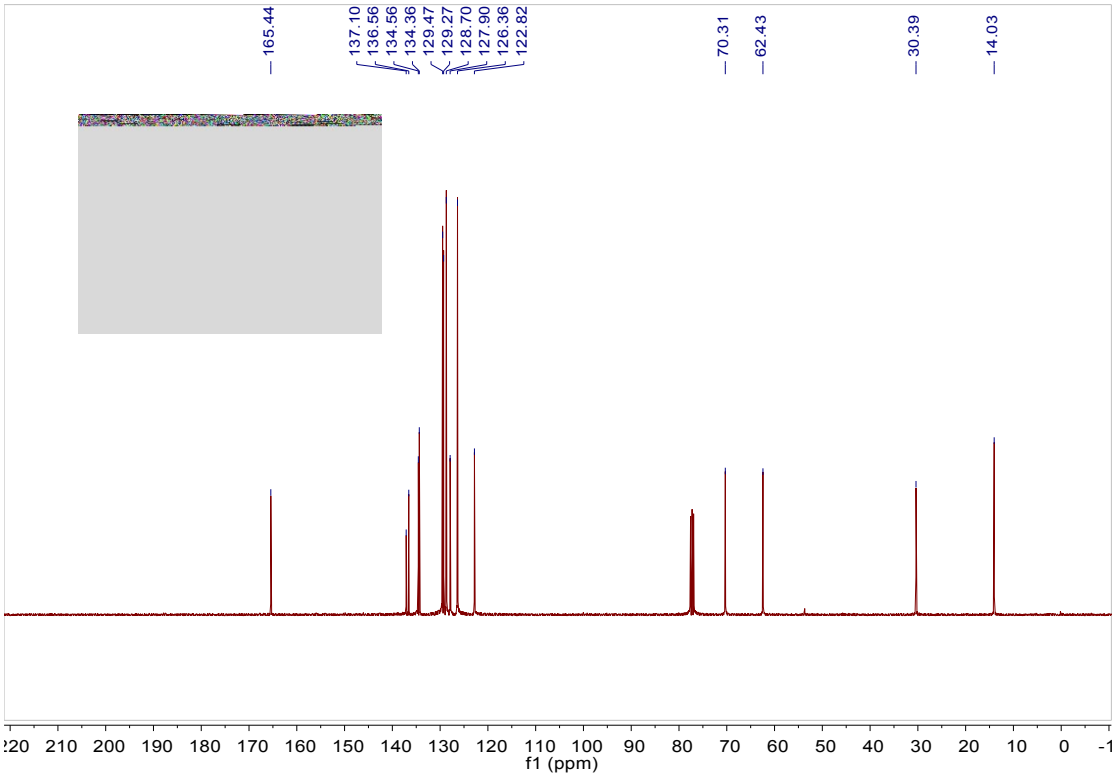
3ao

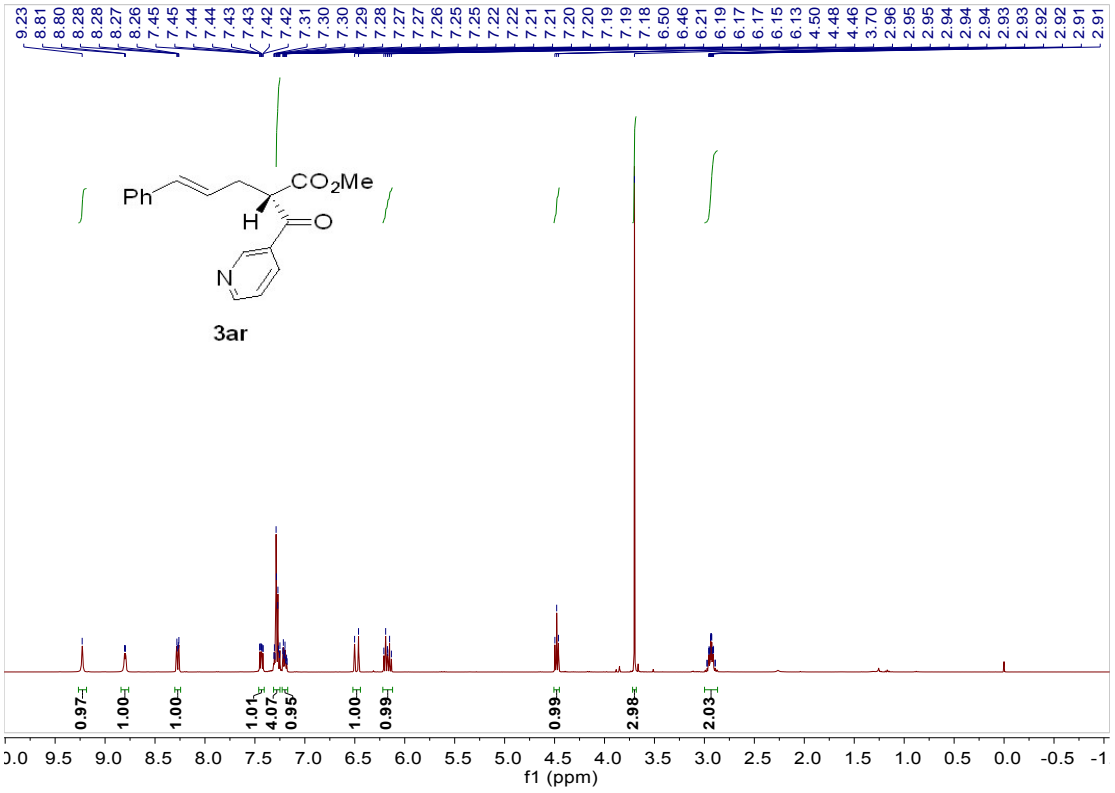

8.07 8.06 8.06 8.05 8.04 8.04 8.03 8.02 8.02 7.31 7.30 7.30 7.29 7.29 7.27 7.26 7.25 7.25 7.22 7.21 7.21 7.20 7.20 7.19 7.18 7.18 7.18 7.17 7.16 7.15 7.15 7.14 7.13 7.13 6.49 6.45 6.21 6.19 6.17 6.17 6.15 6.13 4.47 4.45 4.43 3.69 2.97 2.97 2.95 2.95 2.93 2.93 2.92 2.90 2.88 2.88 2.86 2.86
Ph
CO2Me
H
O
F
3ap
2.00
4.10
1.00
2.00
1.00
0.99
1.00
3.02
2.03
f1 (ppm)

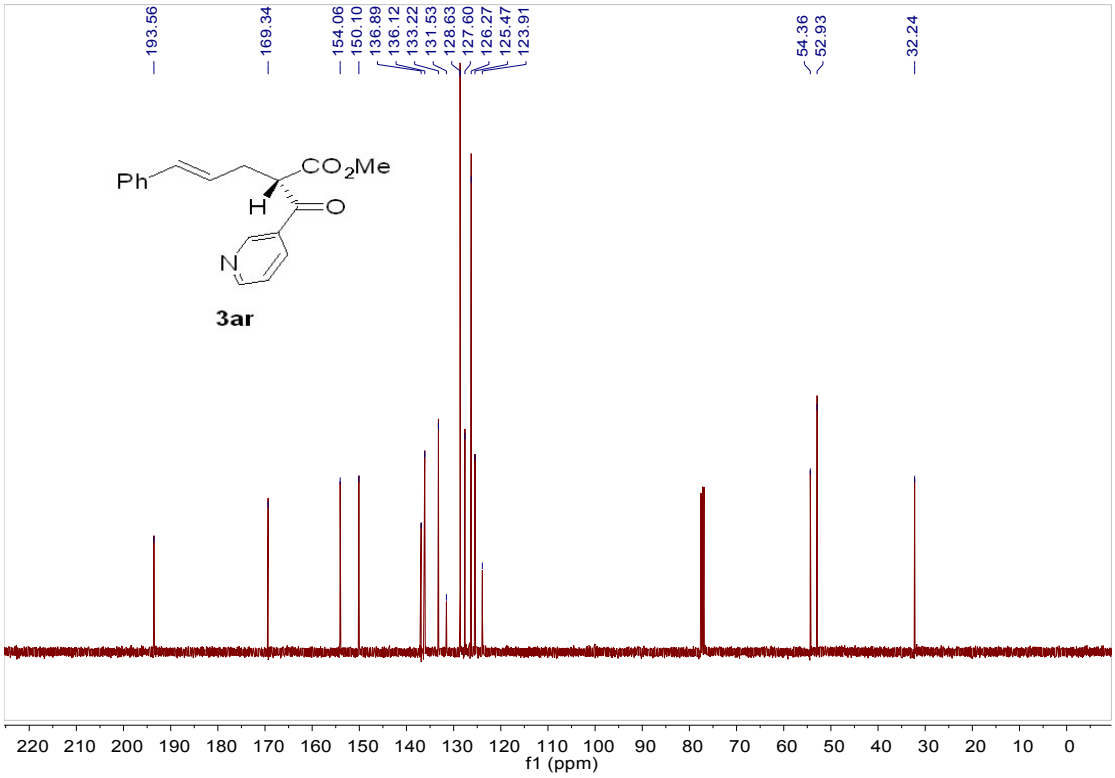

-103.75
Ph
CO2Me
H
O
F
3ap
f1 (ppm)

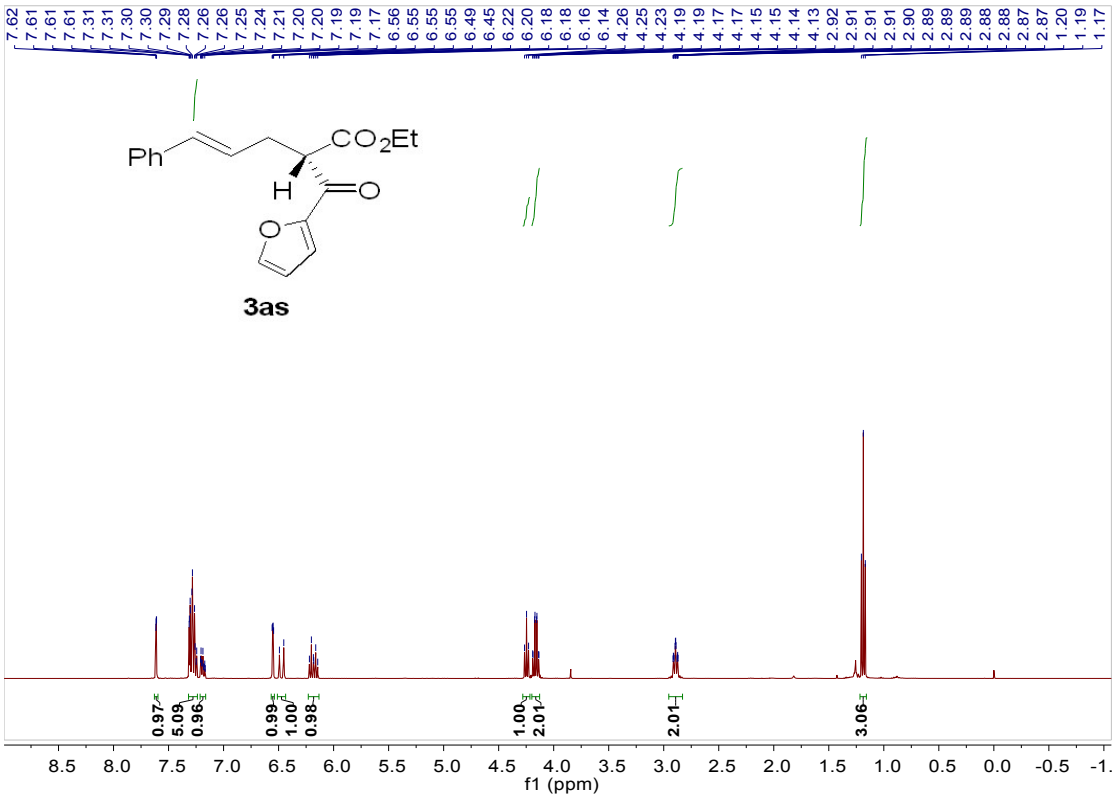

192.89
169.79
167.43
164.89
137.03
132.95
132.58
132.55
131.59
131.50
128.63
127.55
126.28
125.87
116.24
116.02
54.14
52.81
32.52
f1 (ppm)

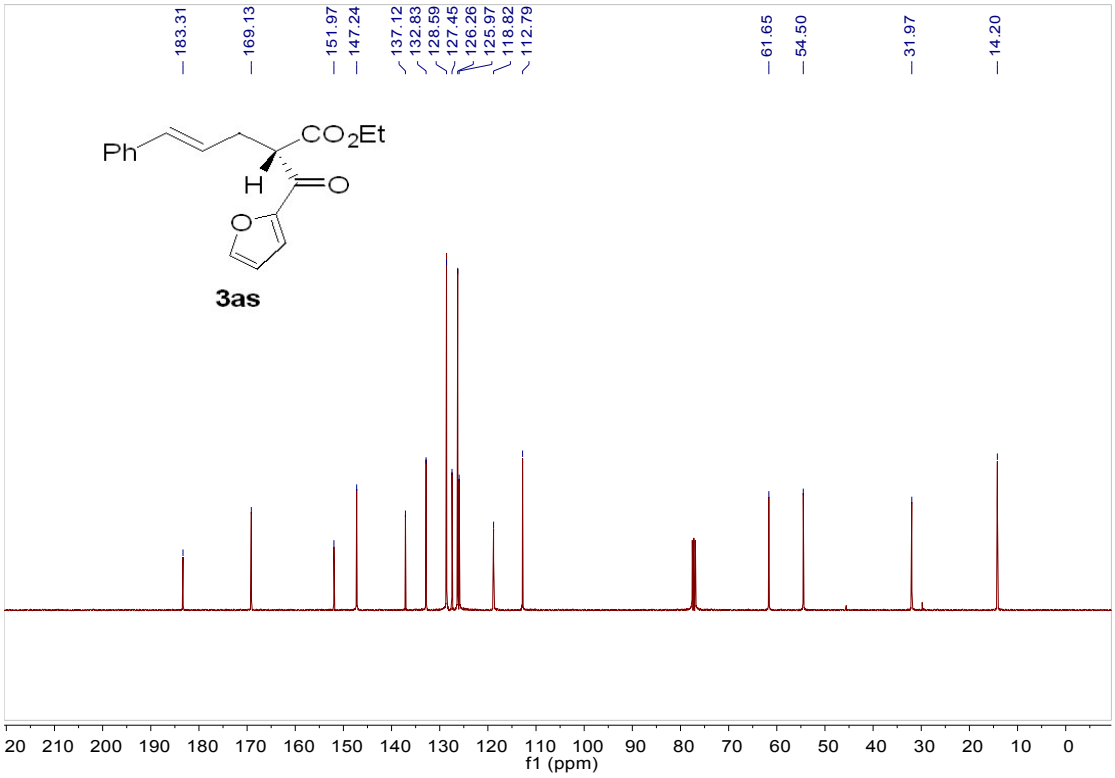

Ph
CO2Et
H
S
O
O
3aq
1.98
1.03
2.06
3.97
1.04
1.00
0.98
2.96
0.99
0.99
3.01
f1 (ppm)

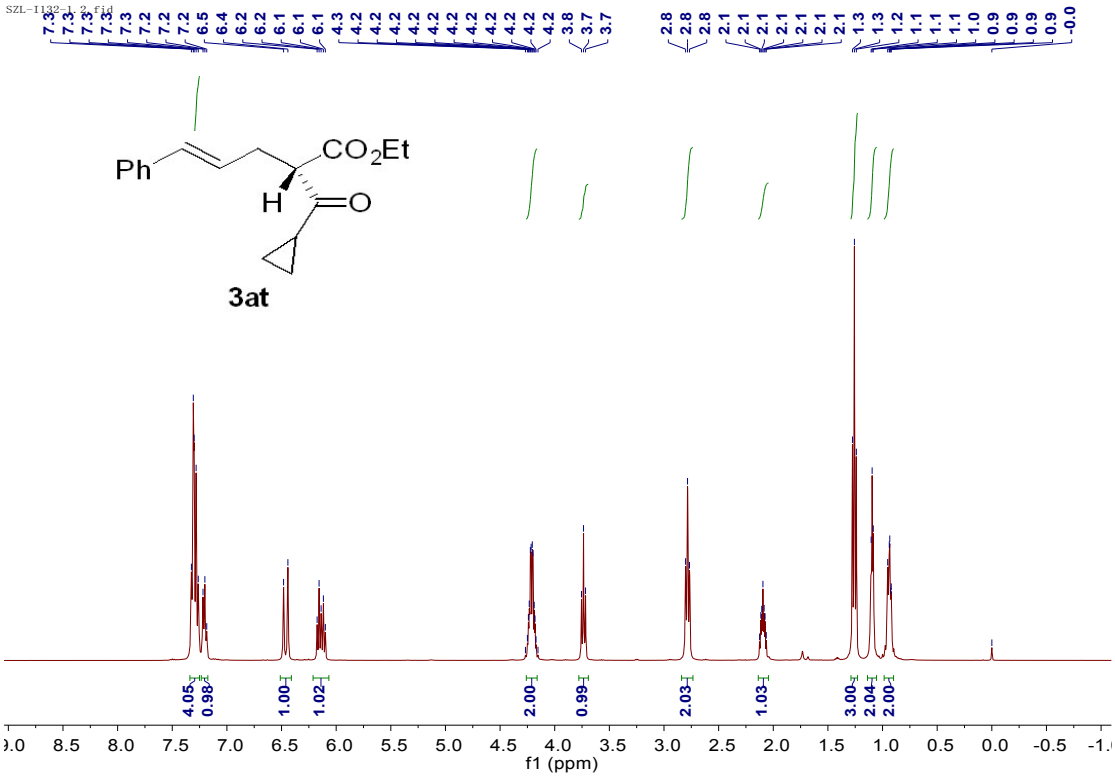

165.44
137.10
136.56
134.56
134.36
129.47
129.27
128.70
127.90
126.36
122.82
70.31
62.43
30.39
14.03
f1 (ppm)

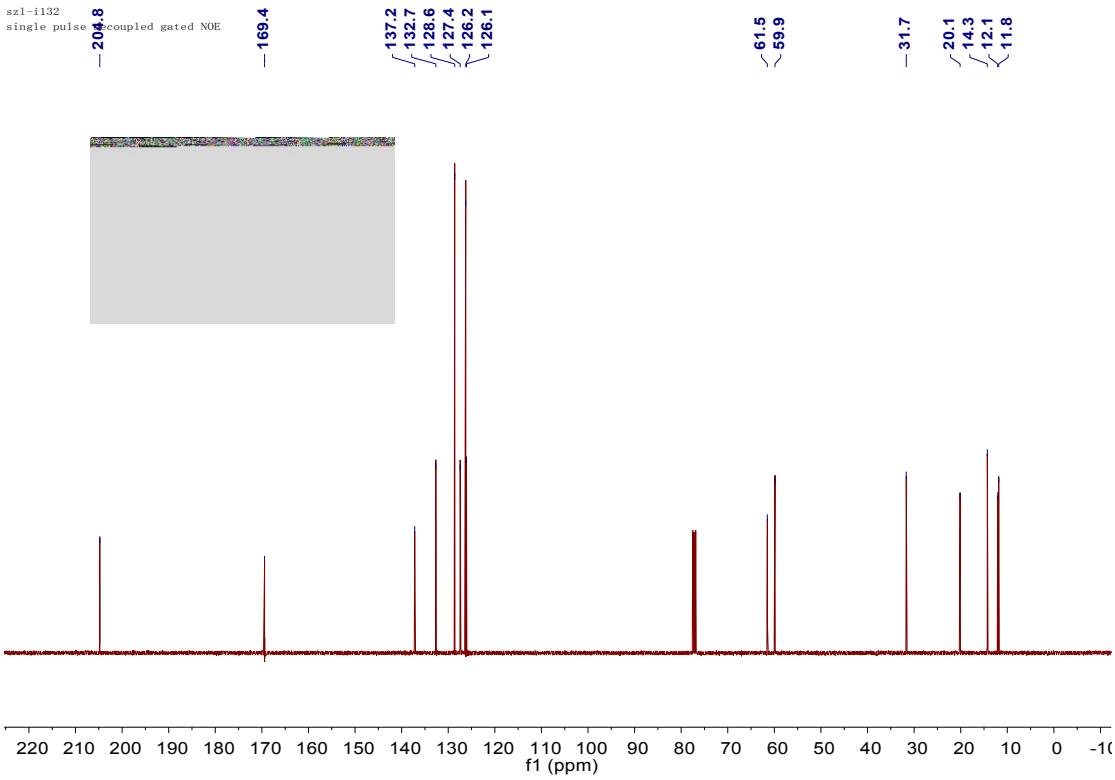

Ph
CO2Me
H
O
N
3ar
0.97
1.00
1.00
1.01
4.07
0.95
1.00
0.99
0.99
2.98
2.03
f1 (ppm)

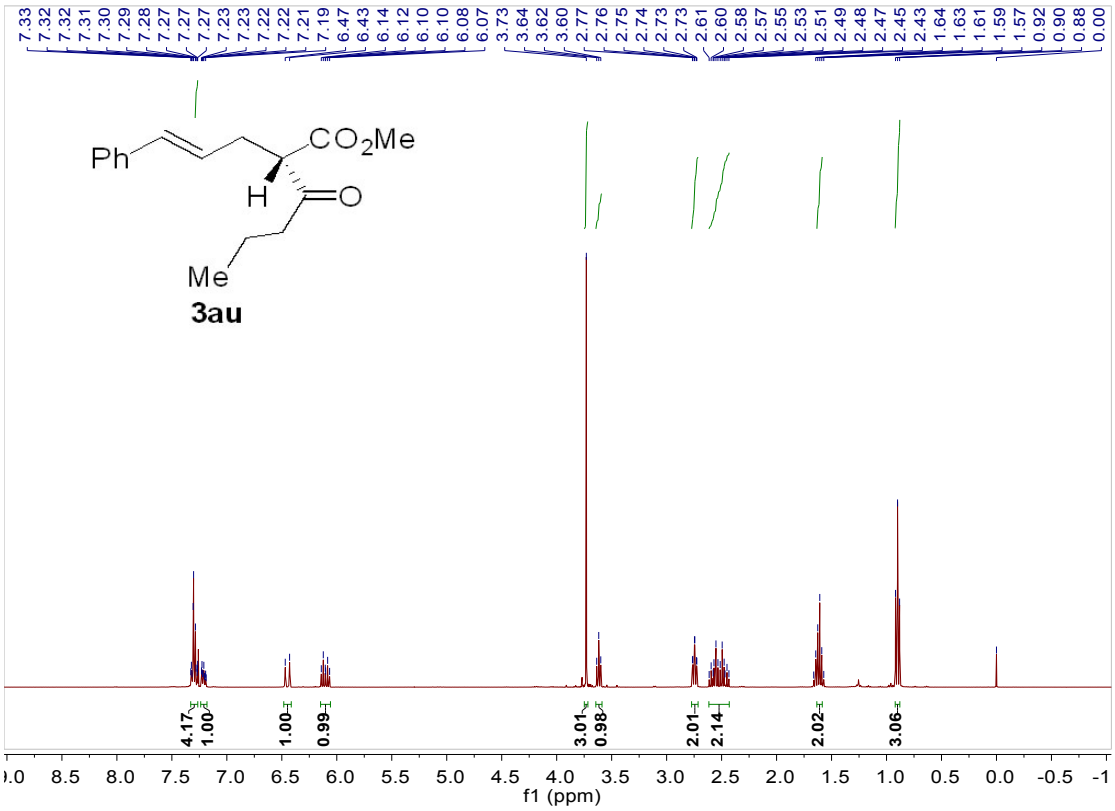

193.56
169.34
154.06
150.10
136.89
136.12
133.22
131.53
128.63
127.60
126.27
125.47
123.91
54.36
52.93
32.24
Ph
CO2Me
H
O
N
3ar
f1 (ppm)

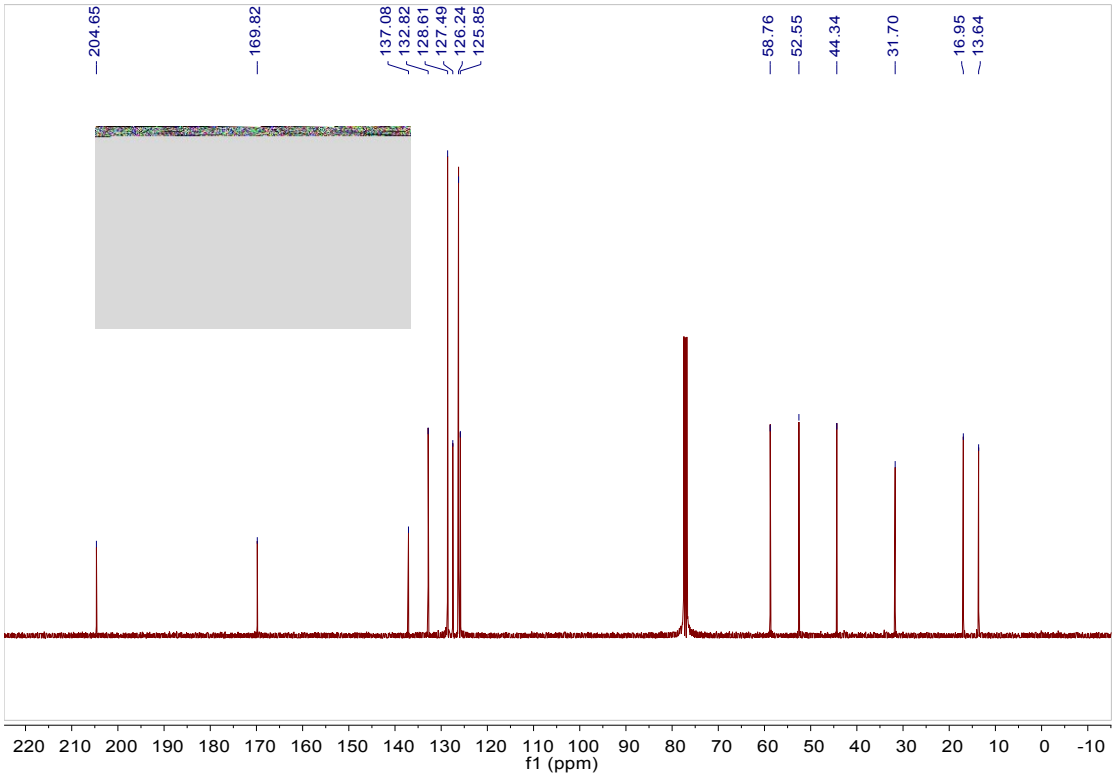

Ph
CO2Et
H
O
O
3as
0.97
5.09
0.96
0.99
1.00
0.98
1.00
2.01
2.01
3.06
f1 (ppm)

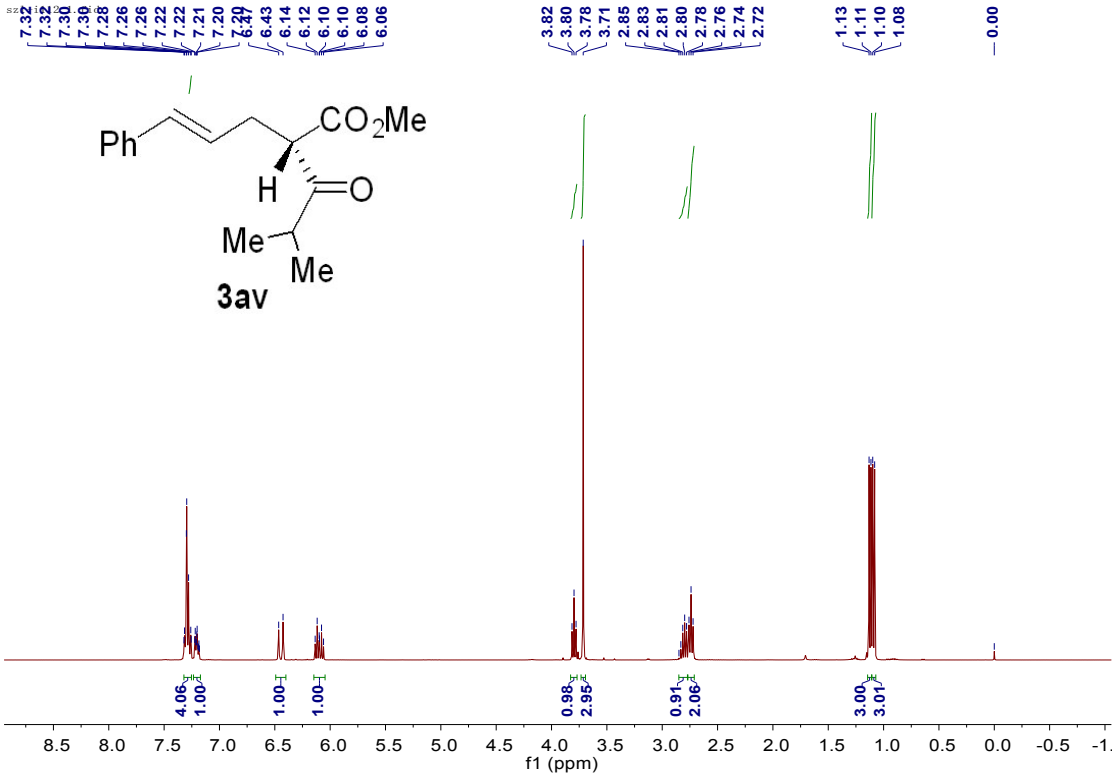

183.31
169.13
151.97
147.24
137.12
132.83
128.59
127.45
126.26
125.97
118.82
112.79
61.65
54.50
31.97
14.20
Ph
CO2Et
H
O
O
3as
f1 (ppm)

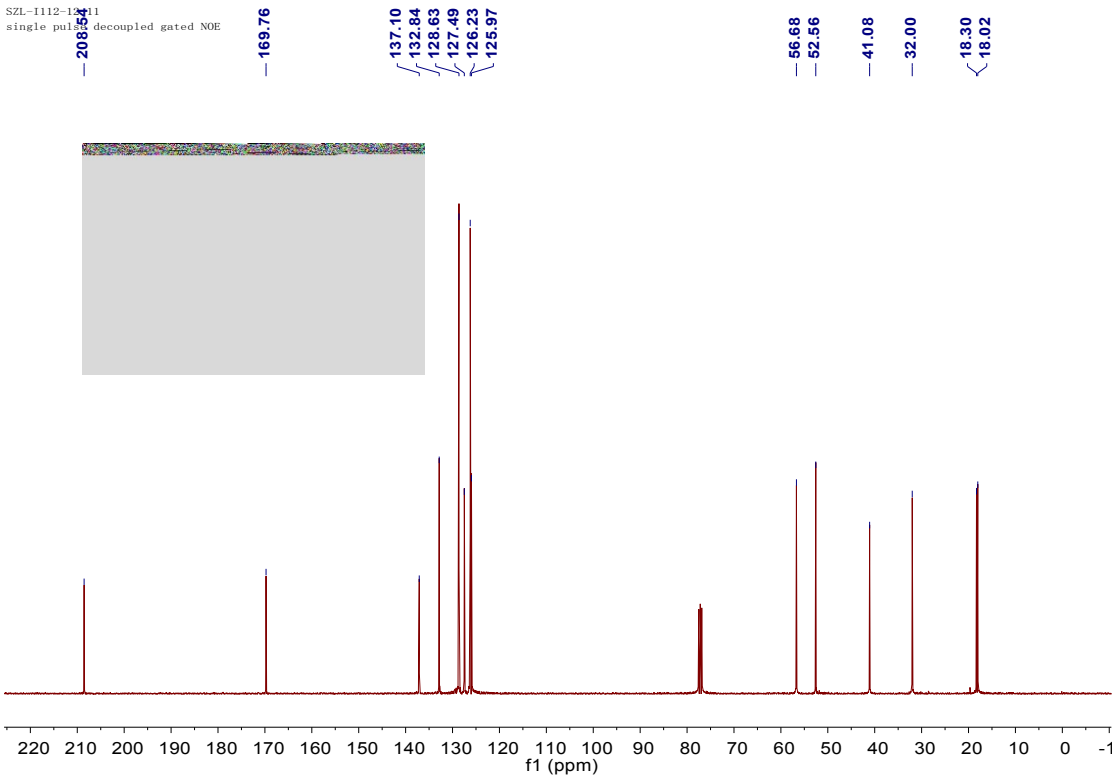

SZL-1132-1-2.fid
Ph
CO2Et
H
O
3at
4.05
0.98
1.00
1.02
2.00
0.99
2.03
1.03
3.00
2.04
2.00
f1 (ppm)

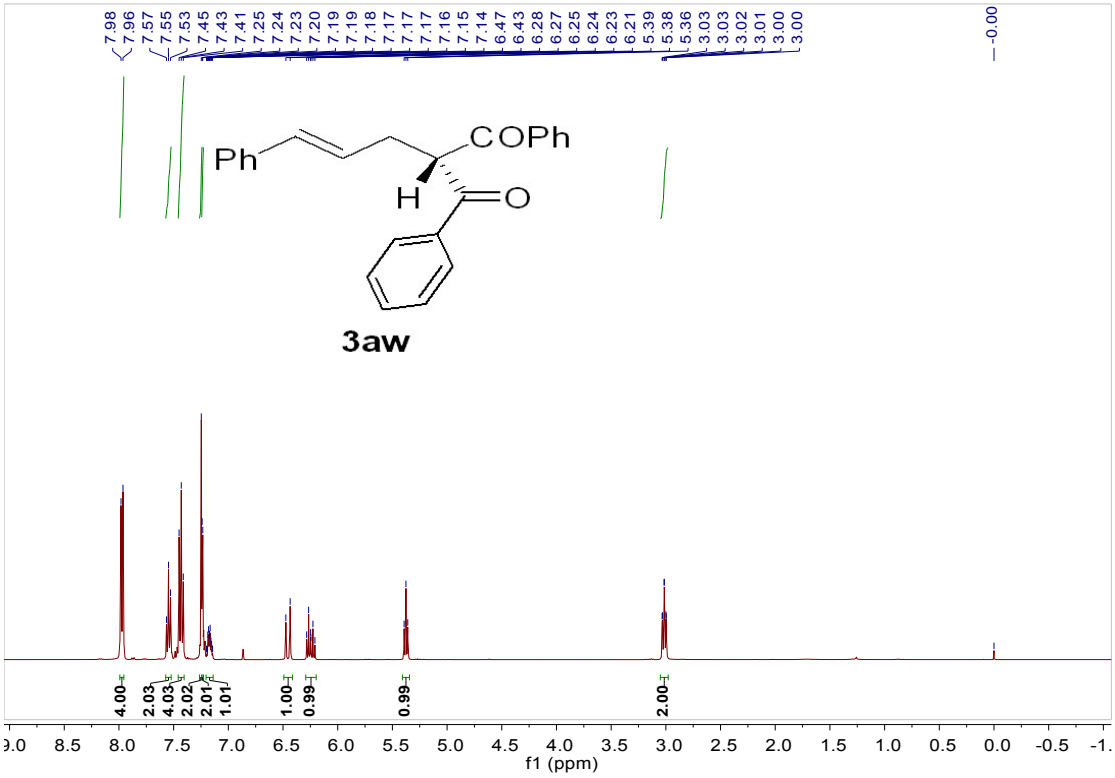

sz1-i132
single pulse decoupled gated NOE
204.8
169.4
137.2
132.7
128.6
127.4
126.2
126.1
61.5
59.9
31.7
20.1
14.3
12.1
11.8
f1 (ppm)

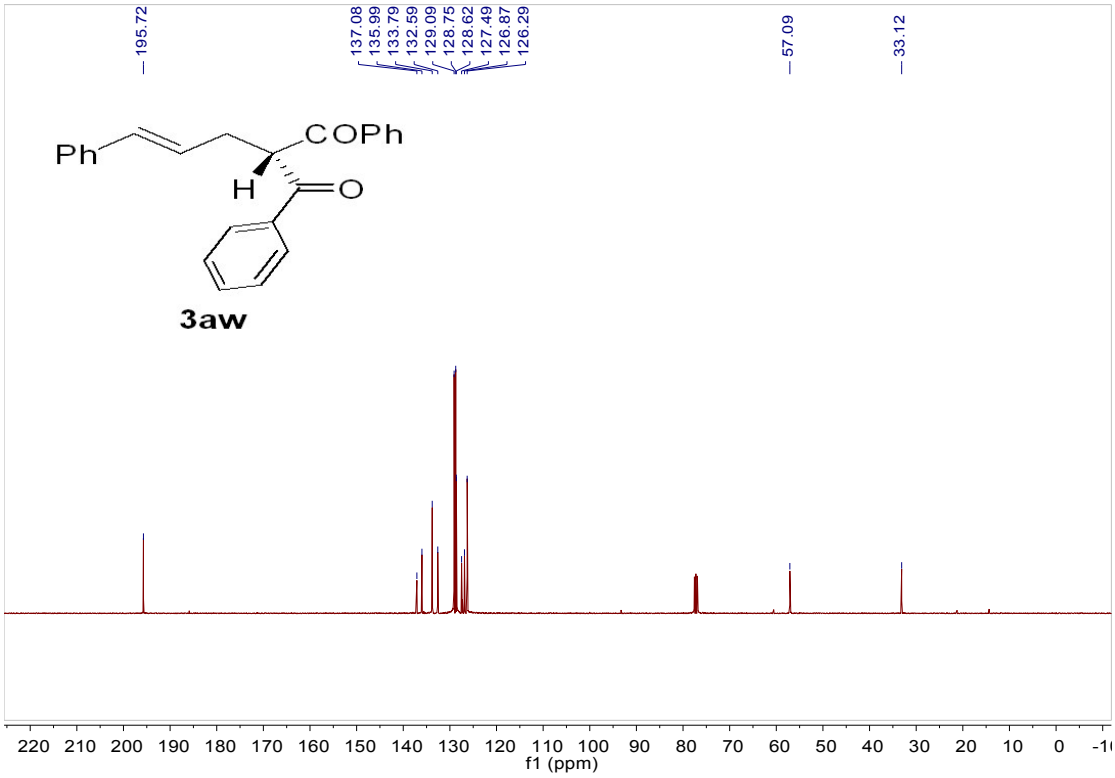

Ph
CO2Me
H
O
Me
3au
4.17
1.00
1.00
0.99
3.01
0.98
2.01
2.14
2.02
3.06
f1 (ppm)

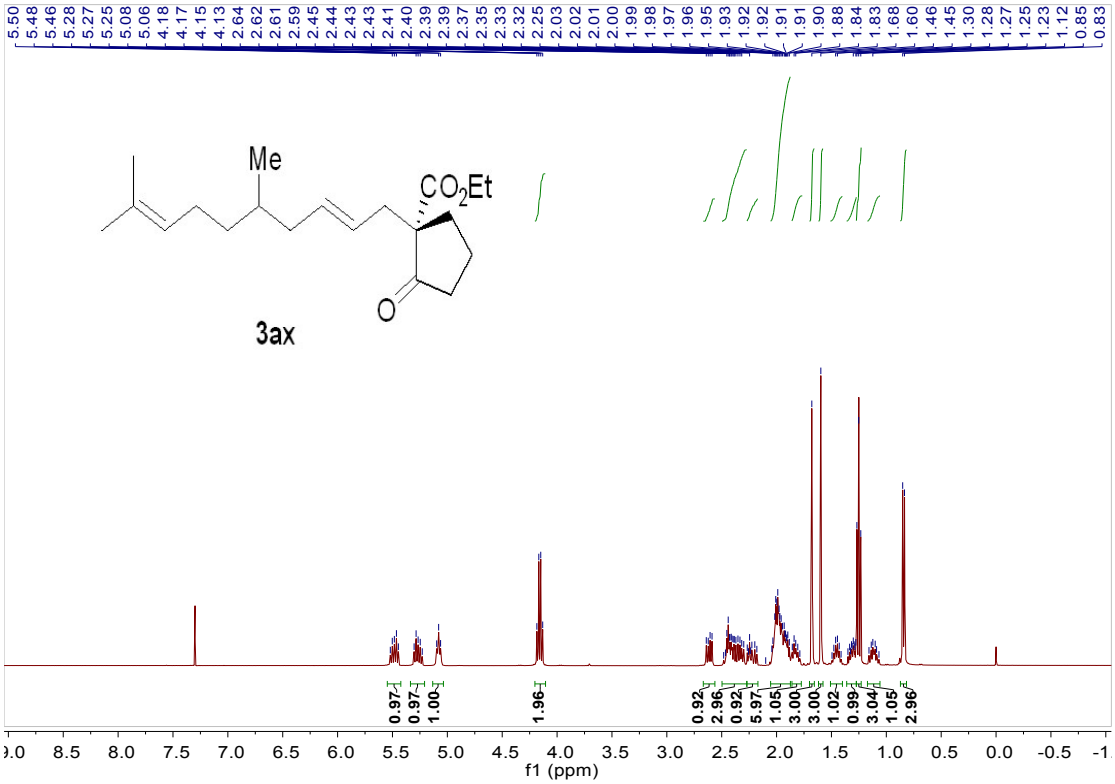

204.65
169.82
137.08
132.82
128.61
127.49
126.24
125.85
58.76
52.55
44.34
31.70
16.95
13.64
f1 (ppm)

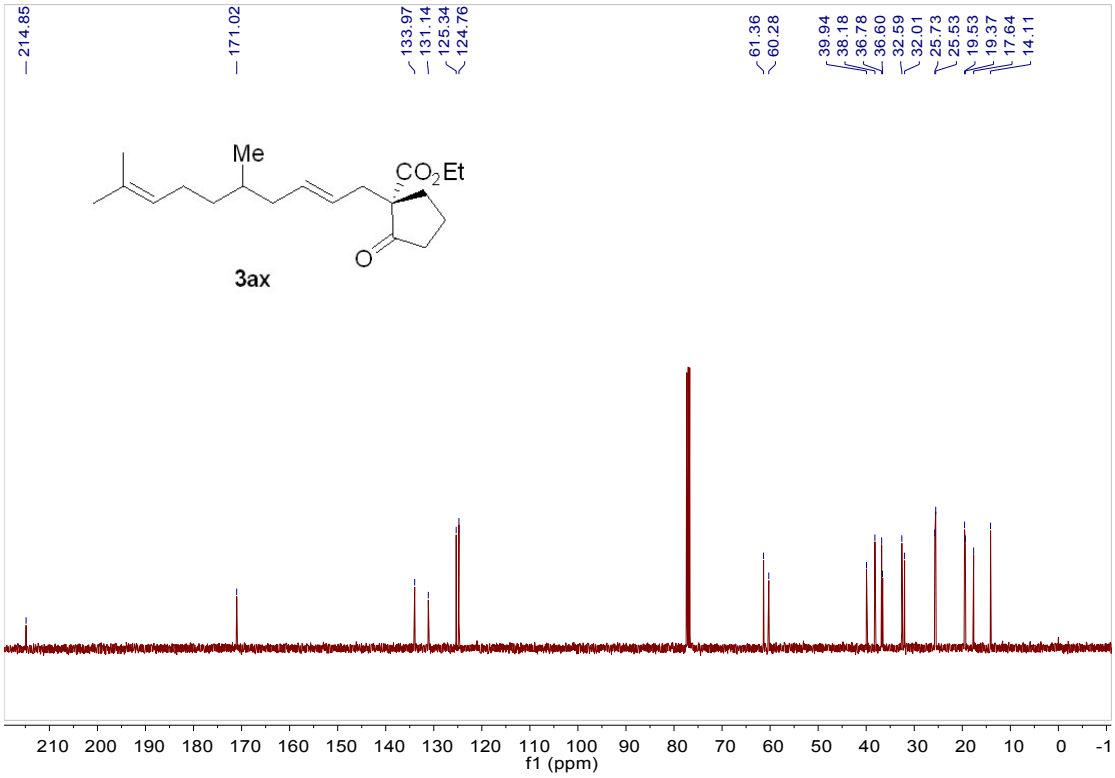

7.32
7.32
7.30
7.30
7.28
7.26
7.26
7.22
7.22
7.21
7.20
7.20
6.47
6.43
6.14
6.12
6.10
6.10
6.08
6.06
3.82
3.80
3.78
3.71
2.85
2.83
2.81
2.80
2.78
2.76
2.74
2.72
1.13
1.11
1.10
1.08
0.00
Ph
CO2Me
H
O
Me
Me
3av
4.06
1.00
1.00
1.00
0.98
2.95
0.91
2.06
3.00
3.01
f1 (ppm)


SZL-I112-13-11
single pulse decoupled gated NOE
208.54
169.76
137.10
132.84
128.63
127.49
126.23
125.97
56.68
52.56
41.08
32.00
18.30
18.02
f1 (ppm)


7.98
7.96
7.57
7.55
7.53
7.45
7.43
7.41
7.25
7.24
7.23
7.20
7.19
7.19
7.18
7.17
7.17
7.17
7.16
7.15
7.14
6.47
6.43
6.28
6.27
6.25
6.24
6.23
6.21
5.39
5.38
5.36
3.03
3.03
3.02
3.01
3.00
3.00
-0.00
Ph
COPh
H
O
3aw
4.00
2.03
4.03
2.02
2.01
1.01
1.00
0.99
0.99
2.00
f1 (ppm)


195.72
137.08
135.99
133.79
132.59
129.09
128.75
128.62
127.49
126.87
126.29
57.09
33.12
Ph
COPh
H
O
3aw
f1 (ppm)


Me
CO2Et
O
3ax
0.97
0.97
1.00
1.96
0.92
2.96
0.92
5.97
1.05
3.00
3.00
1.02
0.99
3.04
1.05
2.96
f1 (ppm)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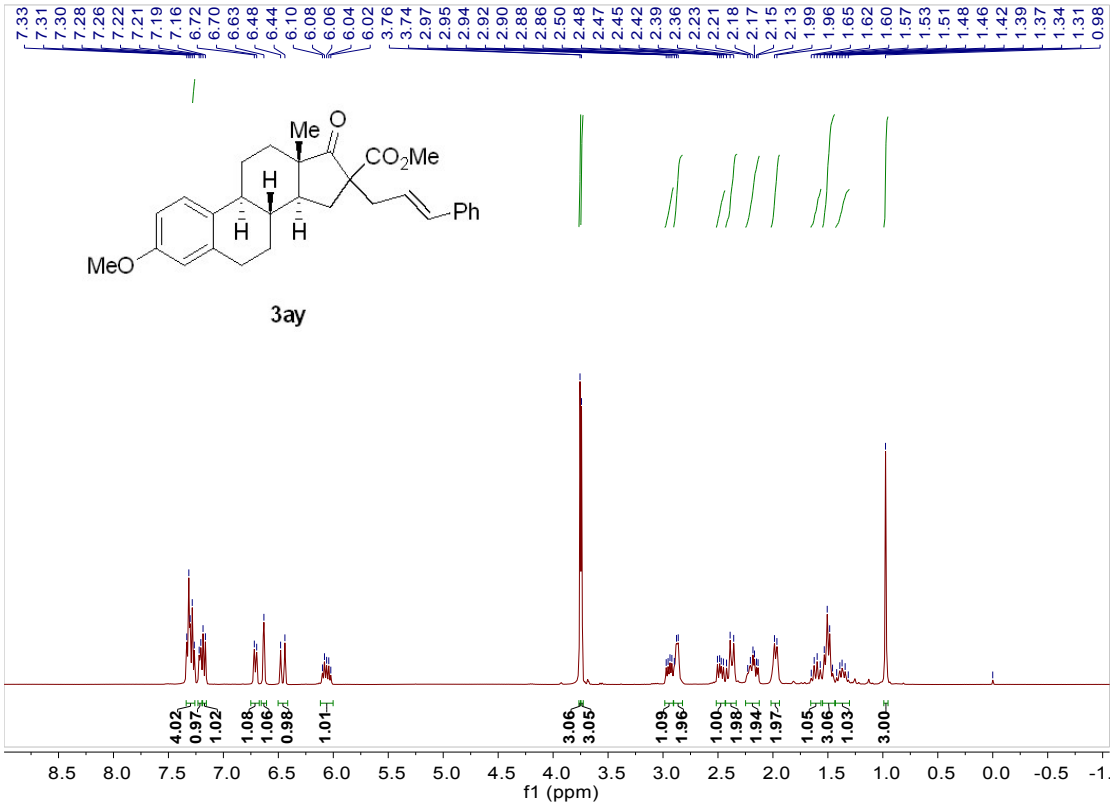
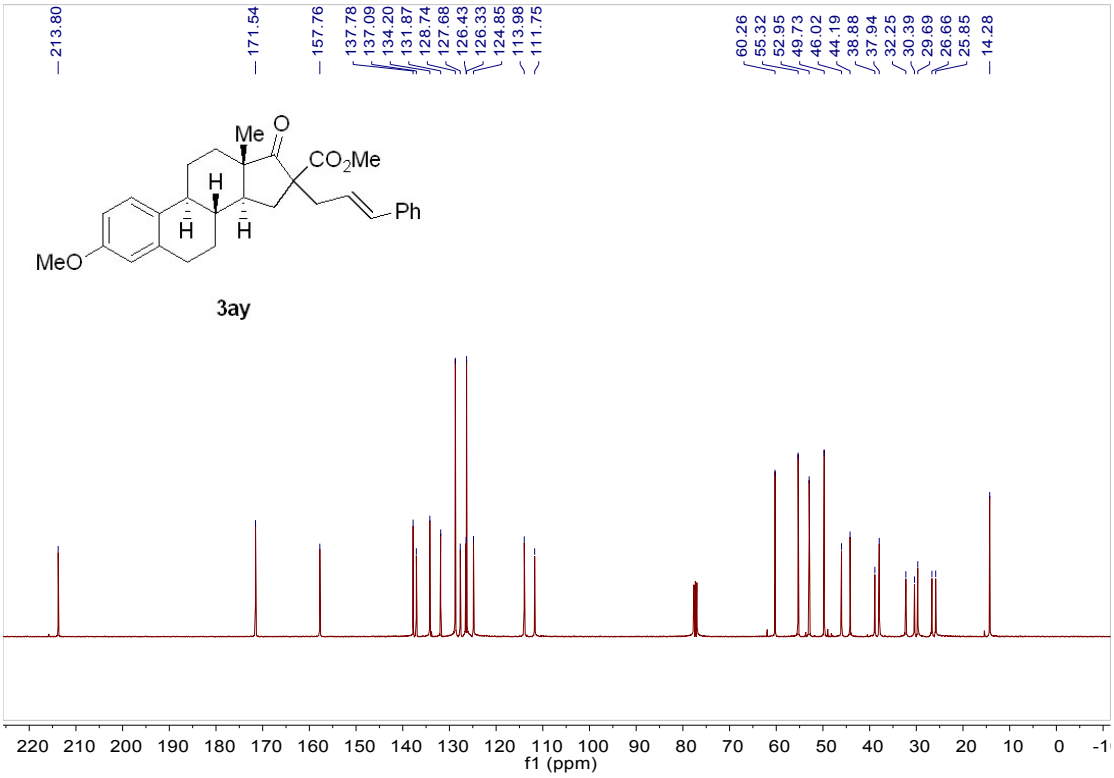
171.02
133.97
131.14
125.34
124.76
61.36
60.28
39.94
38.18
36.78
36.60
32.59
32.01
25.73
25.53
19.53
19.37
17.64
14.11
Me
CO2Et
O
3ax
f1 (ppm)


7.33
7.31
7.30
7.28
7.26
7.22
7.21
7.19
7.16
6.72
6.70
6.63
6.48
6.44
6.10
6.08
6.06
6.04
6.02
3.76
3.74
2.97
2.95
2.94
2.92
2.90
2.88
2.86
2.50
2.48
2.47
2.45
2.42
2.39
2.36
2.23
2.21
2.18
2.17
2.15
2.13
1.99
1.96
1.65
1.62
1.60
1.57
1.53
1.51
1.48
1.46
1.42
1.39
1.37
1.34
1.31
0.98
Me
O
CO2Me
H
H
H
Ph
MeO
3ay
4.02
0.97
1.02
1.08
1.06
0.98
1.01
3.06
3.05
1.09
1.96
1.00
1.98
1.94
1.97
1.05
3.06
1.03
3.00
f1 (ppm)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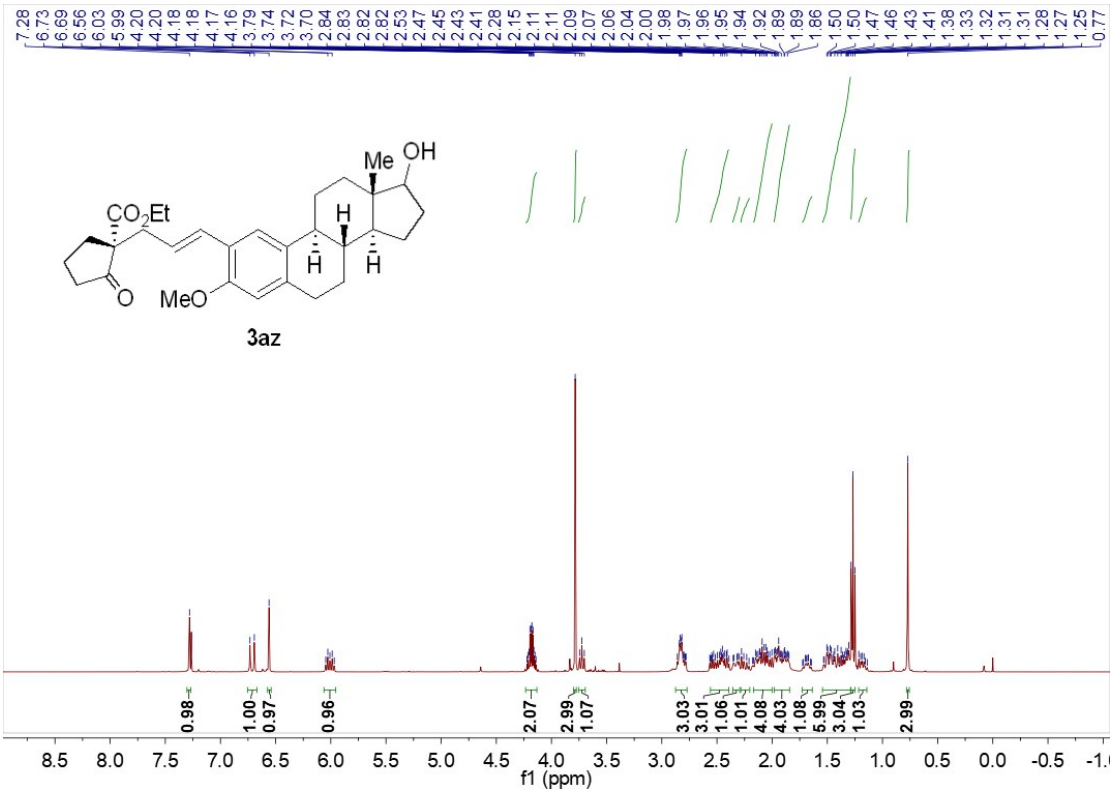
128.74
127.68
126.43
126.33
124.85
113.98
111.75
60.26
55.32
52.95
49.73
46.02
44.19
38.88
37.94
32.25
30.39
29.69
26.66
25.85
14.28
Me
O
CO2Me
H
Ph
H
H
MeO
3ay
f1 (ppm)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6.73
6.69
6.56
6.03
5.99
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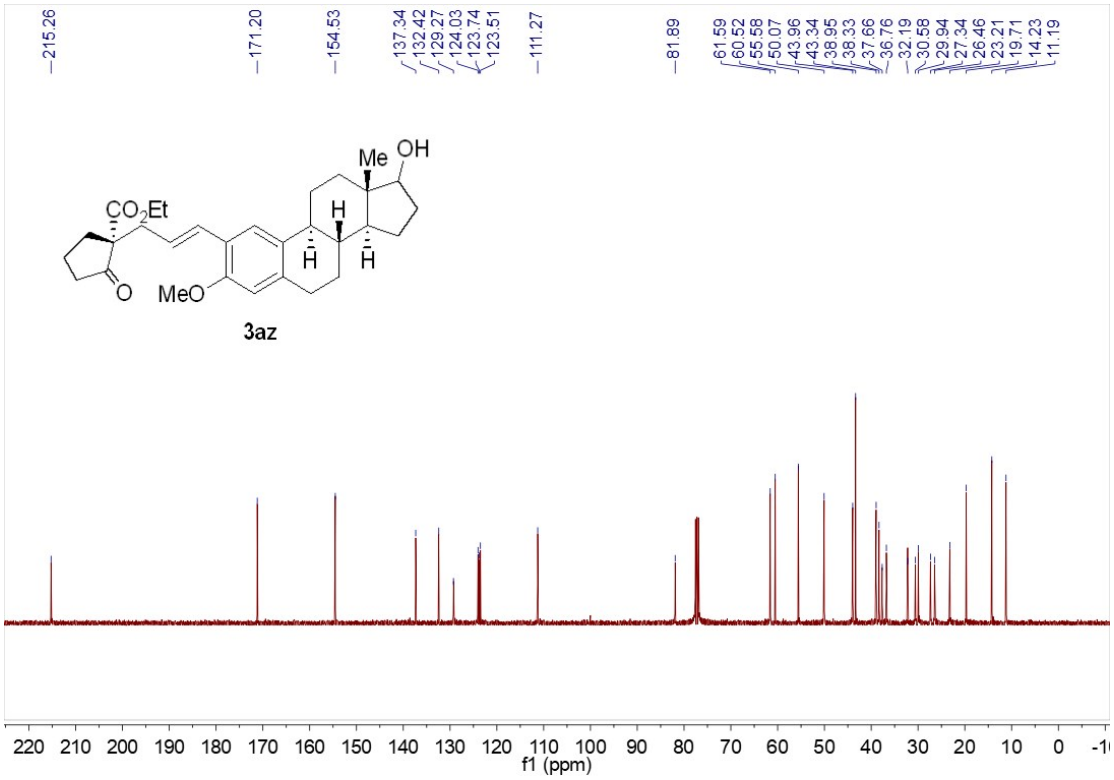
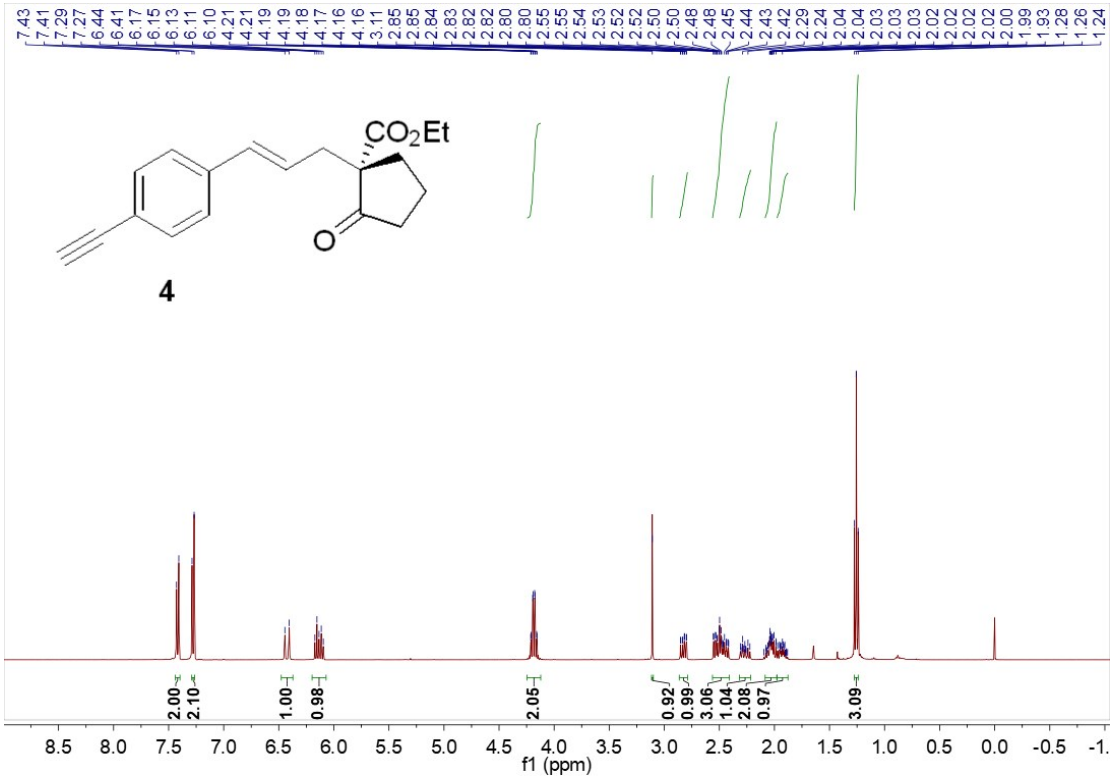
4.20
4.18
4.18
4.17
4.16
3.79
3.74
3.72
3.70
2.84
2.83
2.82
2.82
2.53
2.47
2.45
2.43
2.41
2.28
2.15
2.11
2.11
2.09
2.07
2.06
2.04
2.00
1.98
1.97
1.96
1.95
1.94
1.92
1.89
1.89
1.86
1.50
1.50
1.47
1.46
1.43
1.41
1.38
1.33
1.32
1.31
1.31
1.28
1.27
1.25
0.77
Me
OH
CO2Et
H
H
H
O
MeO
3az
0.98
1.00
0.97
0.96
2.07
2.99
1.07
3.03
3.01
1.06
1.01
4.08
4.03
1.08
5.99
3.04
1.03
2.99
f1 (ppm)


215.26
171.20
154.53
137.34
132.42
129.27
124.03
123.74
123.51
111.27
81.89
61.59
60.52
55.58
50.07
43.96
43.34
38.95
38.33
37.66
36.76
32.19
30.58
29.94
27.34
26.46
23.21
19.71
14.23
11.19
Me
OH
CO2Et
H
H
H
O
MeO
3az
f1 (ppm)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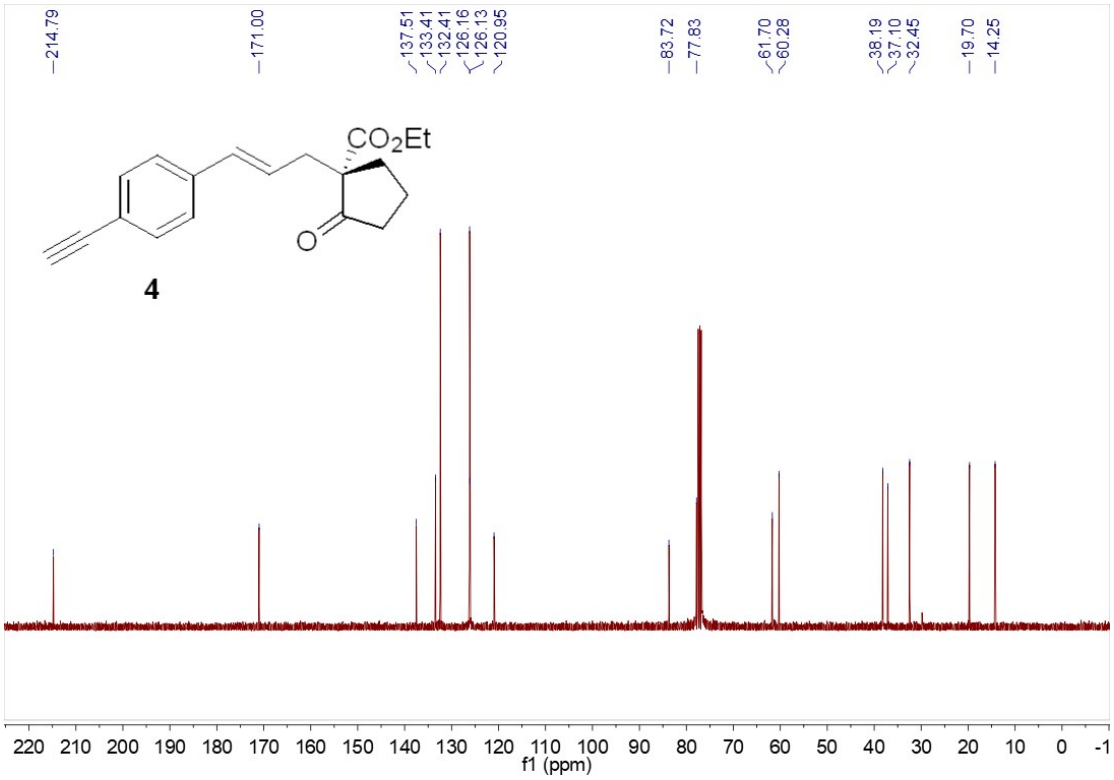
7.41
7.29
7.27
6.44
6.41
6.17
6.15
6.13
6.11
6.10
4.21
4.19
4.19
4.18
4.17
4.16
4.16
3.11
2.85
2.85
2.84
2.83
2.82
2.82
2.80
2.80
2.55
2.55
2.54
2.53
2.52
2.52
2.50
2.50
2.48
2.48
2.45
2.44
2.43
2.42
2.29
2.24
2.04
2.04
2.03
2.03
2.03
2.02
2.02
2.02
2.02
2.00
1.99
1.93
1.28
1.26
1.24
CO2Et
O
4
2.00
2.10
1.00
0.98
2.05
0.92
0.99
3.06
1.04
2.08
0.97
3.09
f1 (ppm)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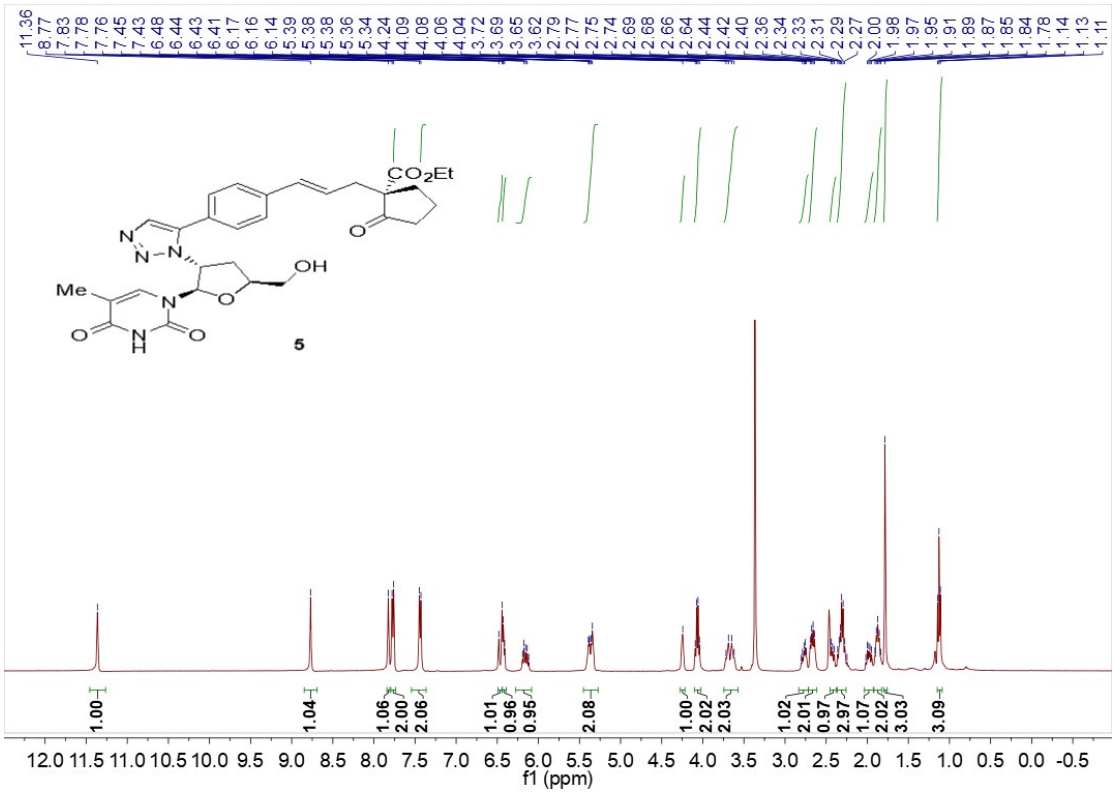
133.41
132.41
126.16
126.13
120.95
83.72
77.83
61.70
60.28
38.19
37.10
32.45
19.70
14.25
CO2Et
O
4
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)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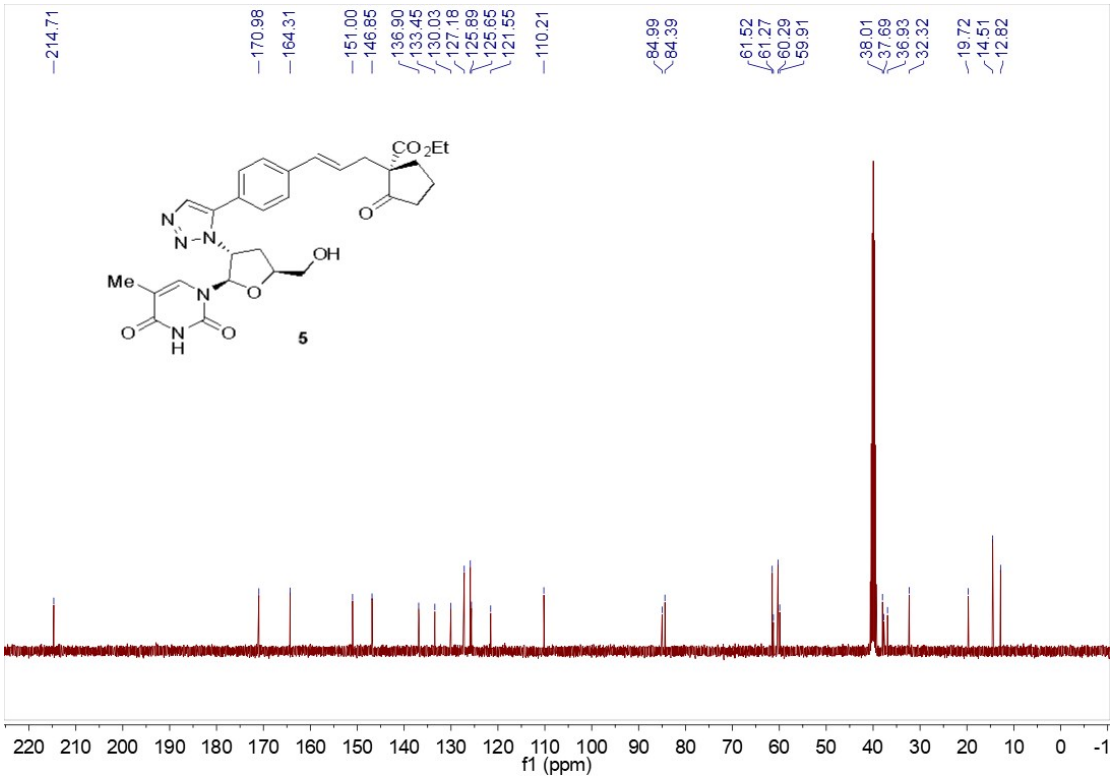
8.77
7.83
7.78
7.76
7.45
7.43
6.48
6.44
6.43
6.41
6.17
6.16
6.14
5.39
5.38
5.38
5.36
5.34
4.24
4.09
4.08
4.06
4.04
3.72
3.69
3.65
3.62
2.79
2.77
2.75
2.74
2.69
2.68
2.66
2.64
2.44
2.42
2.40
2.36
2.34
2.33
2.31
2.29
2.27
2.00
1.98
1.97
1.95
1.91
1.89
1.87
1.85
1.84
1.78
1.14
1.13
1.11
CO2Et
O
N
N
N
OH
Me
N
O
O
N
H
O
5
1.00
1.04
1.06
2.00
2.06
1.01
0.96
0.95
2.08
1.00
2.02
2.03
1.02
2.01
0.97
2.97
1.07
2.02
3.03
3.09
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


214.71
170.98
164.31
151.00
146.85
136.90
133.45
130.03
127.18
125.89
125.65
121.55
110.21
84.99
84.39
61.52
61.27
60.29
59.91
38.01
37.69
36.93
32.32
19.72
14.51
12.82
CO2Et
O
N
N
N
OH
Me
N
O
O
N
H
O
5
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)