Supporting Information

Palladium-Catalyzed Cross-Coupling of Enamides with Sterically Hindered *α*-Bromocarbonyls

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Table of Contents

1. General information

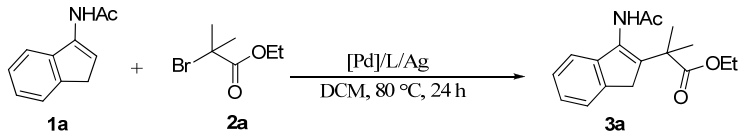
Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. ^{1}H-NMR and ^{13}C-NMR spectra were recorded at 25 ºC on Bruker Advance 400M NMR spectrometers ($CDCl_3$ as solvent). Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of $SiMe_4$ (δ 0.00 singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); dd (doublet of doublets); dt (doublet of triplets); m (multiplets) and *etc*. Coupling constants are reported as a J value in Hz. ^{13}C NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 77.00 triplet). High resolution mass spectral analysis (HRMS) was performed on WaterXEVOG2 Q-TOF (Waters Corporation). IR spectra were recorded on a commercial FT/IR spectrometer. Flash chromatography was performed using 200-300 mesh silica gel with the indicated solvent system.

2. Procedure for the synthesis of compound 3a – 3t, 4a – 4g.

A dry 25-mL Schlenk tube containing a magnetic stirring bar was charged with **1** (0.3 mmol), **2** (2.0 equiv), $Pd(PPh_3)_4$ (5 mol%, 17.3 mg, 0.015 mmol), $(4\text{-MeOPh})_3P$ (20 mol%, 21.12 mg, 0.06 mmol), AgOAc (1.5 eq, 75 mg, 0.45 mmol) and DCM (1.0 mL). Then the mixture was charged with argon and heated at 80 ^{o}C oil bath. After finishing, the reaction mixture was concentrated on a rotary evaporator and the residue was directly subjected to flash column chromatography on silica gel with (50% EtOAc/Petroleum ether) as eluate to furnish the desired product.

3. Optimization of reaction conditions:

Table 1.

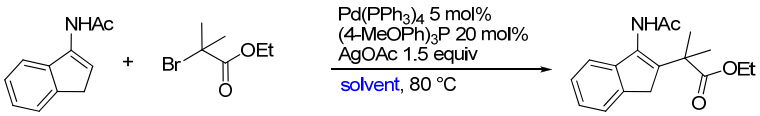


1a 2a 3a

1	$Pd(PPh_3)_4$ (2 mol%)	AgOAc (1.5 eq)	$P(4\text{-MeO-Ph})_3$ (10 mol%)	DCM	43%[c]
2[a]	$Pd(PPh_3)_4$ (5 mol%)	AgOAc (1.5 eq)	$P(4\text{-MeO-Ph})_3$ (20 mol%)	DCM	24%[c]
3[b]	$PPd(PPh_3)_4$ (5 mol%)	AgOAc (1.5 eq)	$P(4\text{-MeO-Ph})_3$ (20 mol%)	DCM	58%[c]

[a] At 60 °C; [b] At 100 °C; [c] Isolated yield

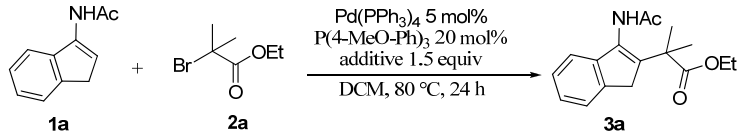
Table 2.



entry	solvent	T (°C)	time (h)	yield[a]
1	$PhCF_3$	80	12	63%
2	dioxane	80	12	70%
3	CH_3CN	80	12	64%
4	DMF	80	12	52%
5	toluene	80	12	60%

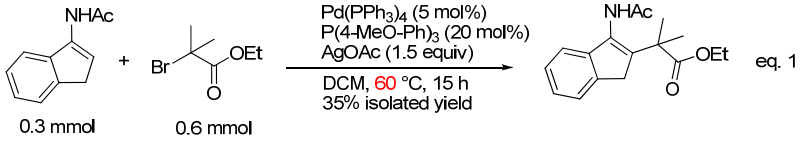
a: isolated yield

Table 3.

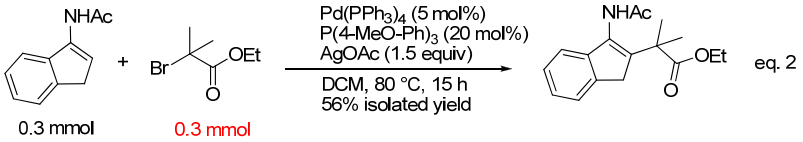


1a 2a 3a

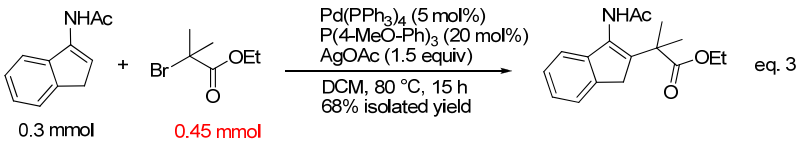
1	NaOAc	NP
2	$Cu(OAc)_2\ H_2O$	71%
3	$Co(OAc)_2$	34
4	$CuBr_2$	NP



0.3 mmol 0.6 mmol

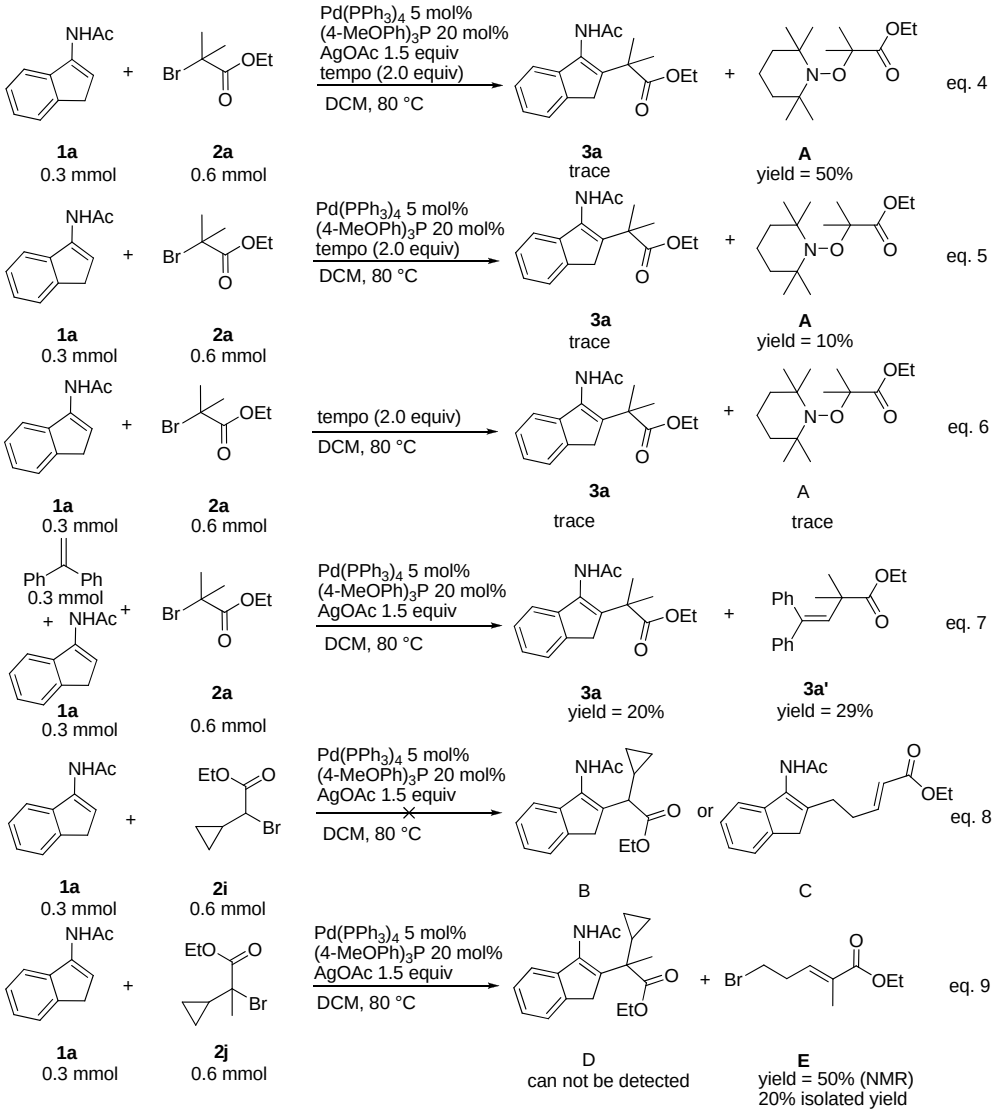


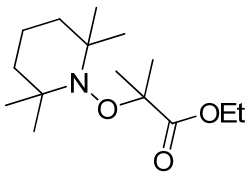
0.3 mmol 0.3 mmol



0.3 mmol 0.45 mmol

4. Mechanistic study of coupling reaction between enamide and *α*-Bromocarbonyls:

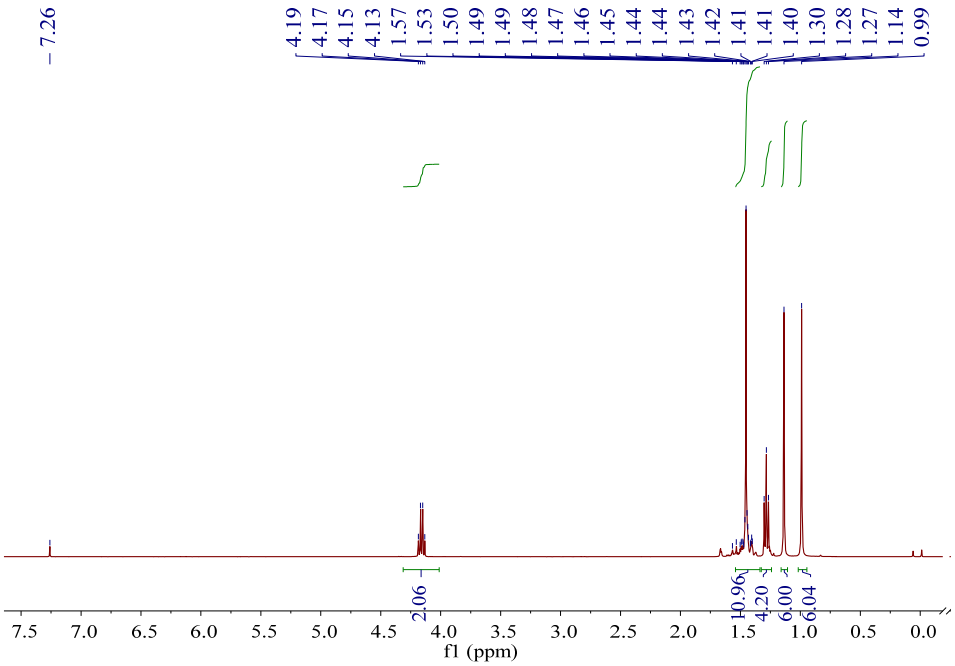


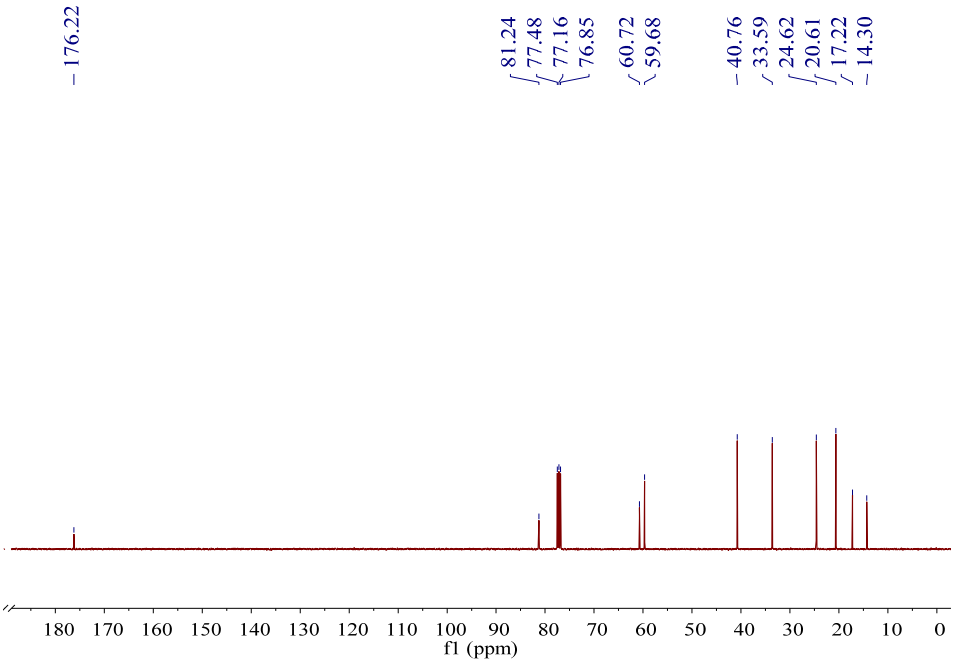


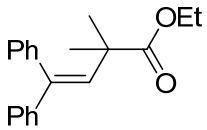
Ethyl 2-methyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propanoate

^{1}H NMR (400 MHz, chloroform-*d*) δ 4.16 (q, *J* = 7.1 Hz, 2H), 1.54 – 1.34 (m, 11H), 1.28 (m, 4H), 1.14 (s, 6H), 0.99 (s, 6H). ^{13}C NMR (100 MHz, chloroform-*d*) δ 176.22, 81.24, 60.72, 59.68, 40.76, 33.59, 24.62, 20.61, 17.22, 14.30.

HRMS (ESI, m/z): calcd for $C_{15}H_{29}NO_3[M+H]^+$ 272.2226, found: 272.2218.



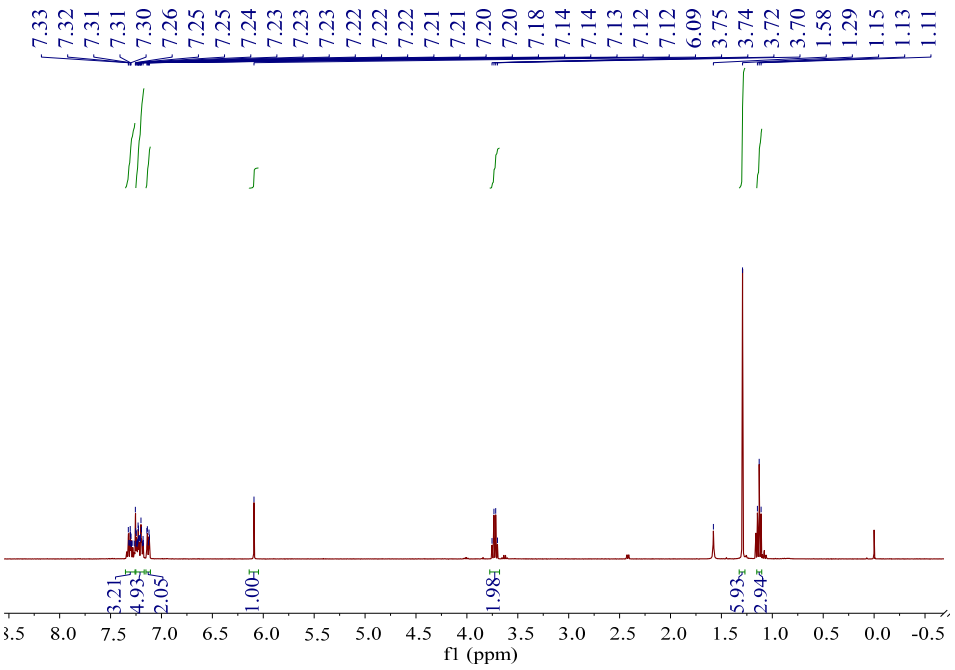


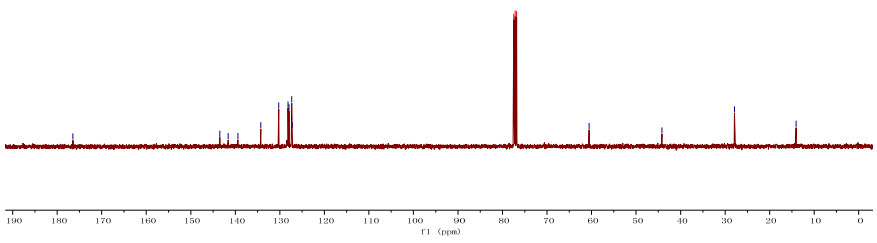


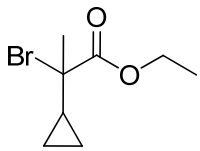
Ethyl 2,2-dimethyl-4,4-diphenylbut-3-enoate

^{1}H NMR (400 MHz, chloroform-*d*) δ 7.35 – 7.26 (m, 3H), 7.25 – 7.17 (m, 5H), 7.15 – 7.11 (m, 2H), 6.09 (s, 1H), 3.73 (q, *J* = 7.2 Hz, 2H), 1.29 (s, 6H), 1.13 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, chloroform-*d*) δ 176.49, 143.48, 141.62, 139.43, 134.29, 130.24, 128.17, 127.96, 127.37, 127.35, 127.24, 60.55, 44.18, 27.90, 14.08.

HRMS (ESI, m/z): calculated for $C_{20}H_{22}O_2Na$ $[M+Na]^+$ 317.1517, found: 317.1519



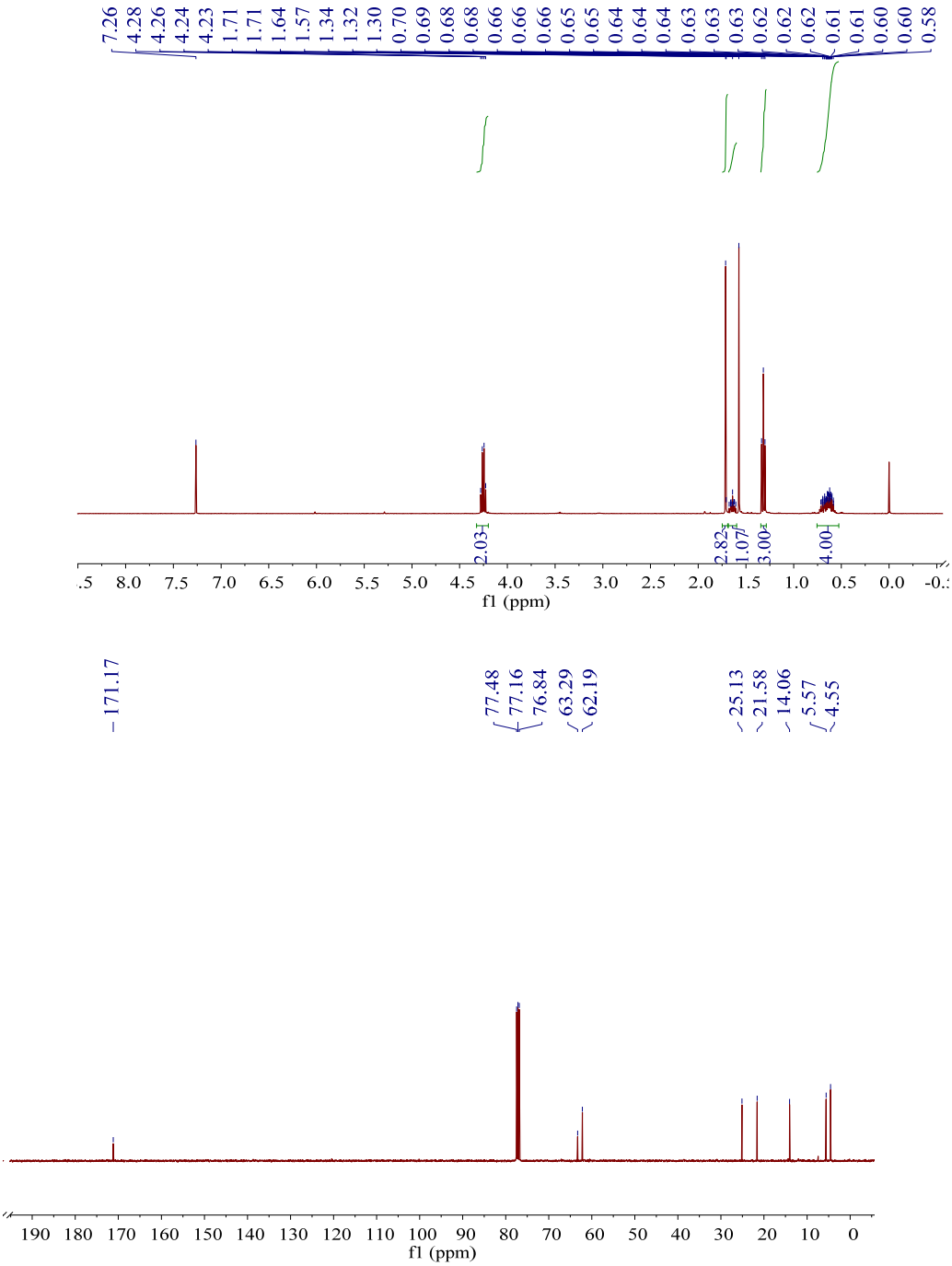


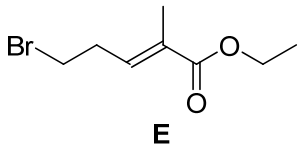


2j

Ethyl 2-bromo-2-cyclopropylpropanoate

^{1}H NMR (400 MHz, chloroform-d) δ 4.25 (q, *J* = 7.1 Hz, 2H), 1.71 (s, 3H), 1.64 (tt, *J* = 8.2, 6.0 Hz, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 0.76 – 0.53 (m, 4H). ^{13}C NMR (100 MHz, chloroform-*d*) δ 171.17, 63.29, 62.19, 25.13, 21.58, 14.06, 5.57, 4.55.



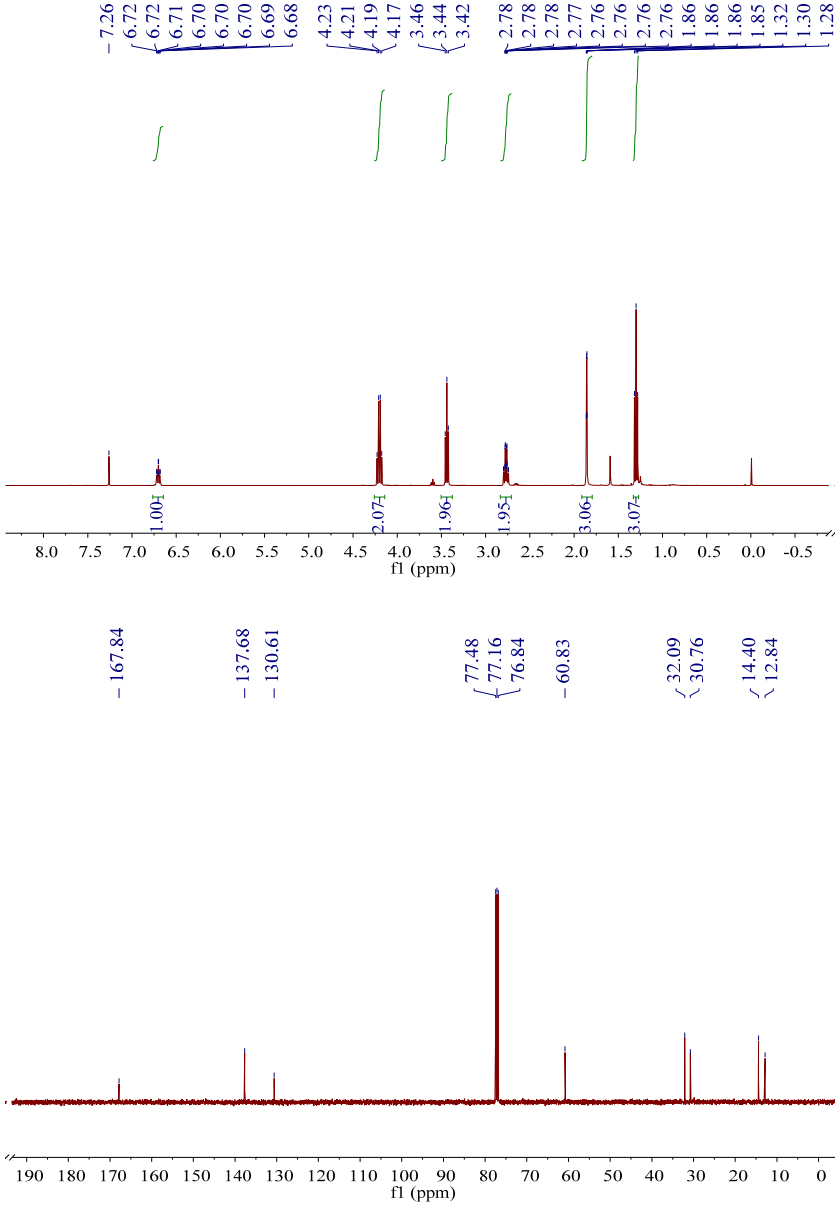


E

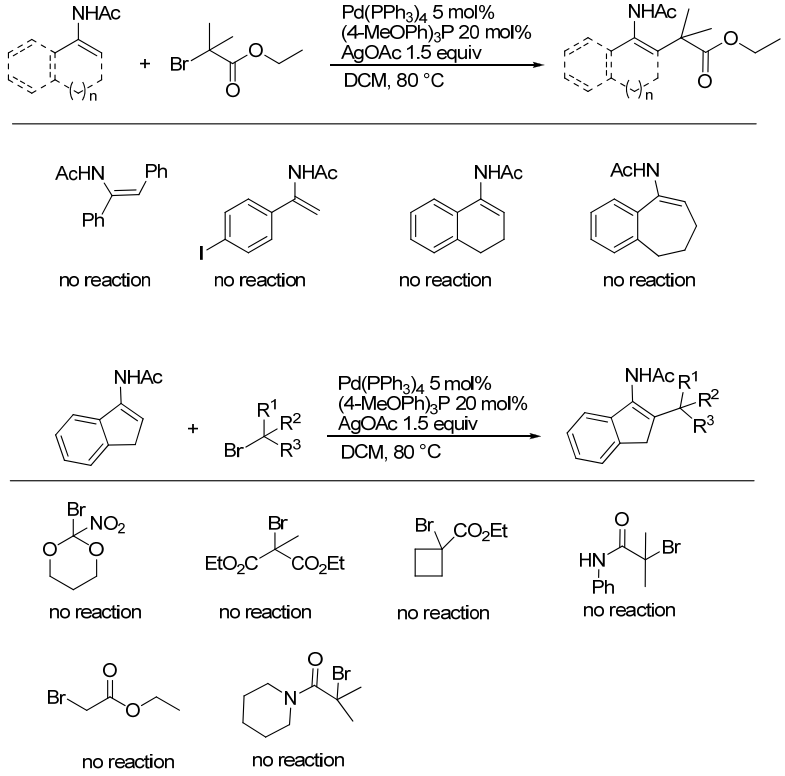
(E)-ethyl 5-bromo-2-methylpent-2-enoate

^{1}H NMR (400 MHz, chloroform-*d*) δ 6.76 – 6.65 (m, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.44 (t, *J* = 6.9 Hz, 2H), 2.77 (dddd, *J* = 8.1, 7.1, 6.1, 1.0 Hz, 2H), 1.86 (q, *J* = 1.1 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, chloroform-*d*) δ 167.84, 137.68, 130.61, 60.83, 32.09, 30.76, 14.40, 12.84.

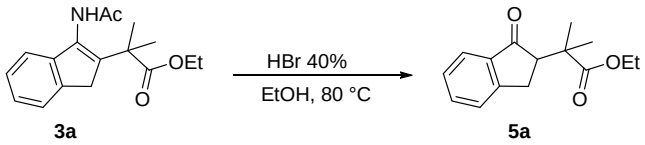
HRMS (ESI, m/z): calculated for $C_8H_{13}BrO_2Na$ $[M+Na]^+$ 242.9997, found: 242.9991.



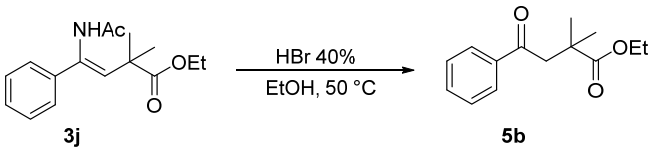
5. Unsuccessful substrates in coupling reaction between enamides and α-bromocarbonyls.



6. Procedure for the synthesis of compound 5a – 5j.

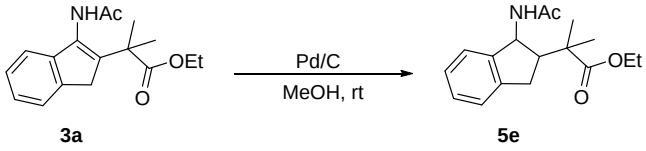


3a (0.3 mmol, 86.4 mg) dissolved in EtOH (2.0 mL) was added 48% aqueous solution of HBr (1.0 mL). The mixture was stirred at 80 °C for 10 hours. After finishing, then the reaction was quenched by addition of saturated aqueous $NaHCO_3$ at 0 °C. The reaction mixture was allowed to warm to room temperature and was extracted with CH_2Cl_2. The organic layer was washed with brine and dried over anhydrous $MgSO_4$. After filtration, the organic solvent was removed under reduced pressure on a rotary evaporator. Then the crude product was purified by column chromatography on silica gel (with 5% EtOAc/Petroleum ether) to afford the desired compound **5a** (61.9 mg, yield = 84%).



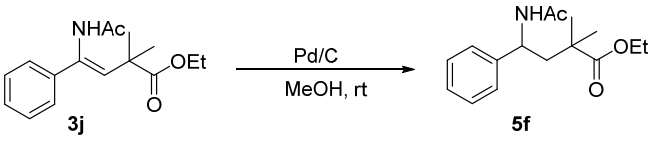
3j **5b**

3j (0.3 mmol, 82.8 mg) dissolved in EtOH (2.0 mL) was added 48% aqueous solution of HBr (1.0 mL). The mixture was stirred at 50 °C for 10 hours. then the reaction was quenched by addition of saturated aqueous $NaHCO_3$ at 0 °C. The reaction mixture was allowed to warm to room temperature and was extracted with CH_2Cl_2. The organic layer was washed with brine and dried over anhydrous $MgSO_4$. After filtration, the organic solvent was removed under reduced pressure on a rotary evaporator. Then the crude product was purified by chromatography on silica gel (with 5% EtOAc/Petroleum ether) to afford the desired compound **5b**. Yield (61.3 mg, 87%).



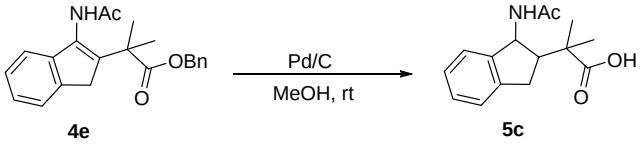
3a **5e**

3a (0.1 g) and Pd/C catalyst (10%, 0.01 g) were added to the MeOH (25 mL) solution in a two-neck round-bottom flask under hydrogen atmosphere. The mixture was stirred for 6 hours and was filtered. The filtrate was evaporated under reduced pressure to give a white solid of **5e** without further purification. Yield (0.093 g, 93%).



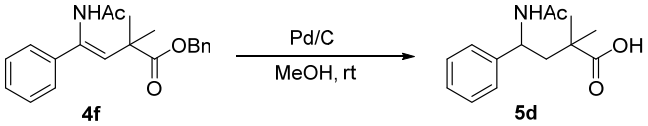
3j **5f**

3j (0.2 g) and Pd/C catalyst (10%, 0.02 g) were added to the MeOH (25 mL) solution in a two-neck round-bottom flask under hydrogen atmosphere. The mixture was stirred for 6 hours and was filtered. The filtrate was evaporated under reduced pressure to give a white solid of **5f** without further purification. Yield (0.183 g, 91%).



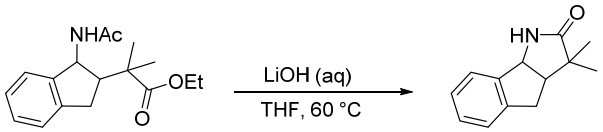
4e **5c**

4e (0.2075 g) and Pd/C catalyst (10%, 0.02 g) were added to the MeOH (25 mL) solution in a two-neck round-bottom flask under hydrogen atmosphere. The mixture was stirred for 6 hours and was filtered. The filtrate was evaporated under reduced pressure, then the crude product was purified by chromatography on silica gel (with 0% - 8% MeOH/$CHCl_3$) to give a white solid of **5c**. Yield (0.1309 g, 85%).



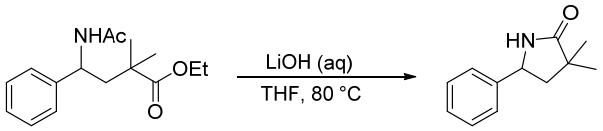
4f **5d**

4f (0.0668 g) and Pd/C catalyst (10%, 0.01 g) were added to the MeOH (25 mL) solution in a two-neck round-bottom flask under hydrogen atmosphere. The mixture was stirred for 6 hours and was filtered. The filtrate was evaporated under reduced pressure, then the crude product was purified by chromatography on silica gel (with 0% - 8% MeOH/$CHCl_3$) to give a white solid of **5d**. Yield (0.04 g, 81%).



5e **5i**

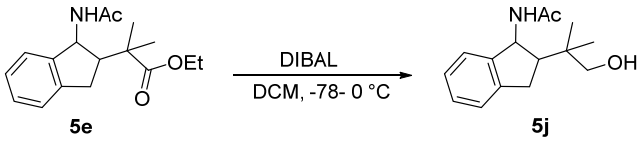
Aqueous lithium hydroxide (1 M, 2 mL) was added to the solution of **5e** (0.0893 g, 0.308 mmol) in THF (2 mL). The mixture was stirred at 60 °C for 10 hours. After finishing, the mixture was diluted with ethyl acetate (10 mL). After separation, the aqueous phase was extracted twice with ethyl acetate (3×10 mL). The combined organic layers were washed with brine and dried over anhydrous sodium sulfate. The organic solvent was removed under reduced pressure, and the crude product was purified by chromatography on silica gel (with 20% - 60% EtOAc/Petroleum ether) to afford the desired compound **5i**. Yield (0.061 g, 92%).



5f **5g**

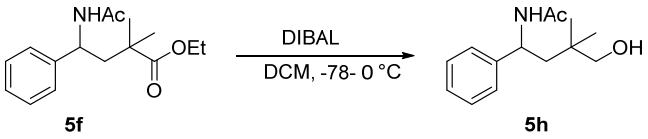
Aqueous lithium hydroxide (1 M, 3 mL) was added to the solution of **5f** (0.148 g, 0.53 mmol) in THF (3 mL). The mixture was stirred at 80 °C for 10 hours. After finishing, the mixture was diluted with ethyl acetate (10 mL). After separation, the aqueous

phase was extracted twice with ethyl acetate (3×10 mL). The combined organic layers were washed with brine and dried over anhydrous sodium sulfate. The organic solvent was removed under reduced pressure, and the crude product was purified by chromatography on silica gel (with 20% - 60% EtOAc/Petroleum ether) to afford the desired compound **5g**. Yield (0.0808 g, 81%).



5e **5j**

Diisobutylaluminum hydride solution (1.0 M in hexane, 0.9 mL, 0.9 mmol) was added dropwise to the solution of **5e** (0.058 g, 0.2 mmol) in dichloromethane (2 mL) at -78 °C. After stirring for 10 minutes at the same temperature, the reaction mixture was warmed to 0 °C and stirred for an additional 30 minutes. The methanol (1 mL) and hydrochloric acid (3 M, 3 mL) were successively added. The reaction mixture was partitioned by dichloromethane, and then the aqueous phase was extracted twice with dichloromethane (3×10 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered and concentrated. The residue was purified by flash column chromatography on silica gel to give **5j** (0.025 g , 51%)

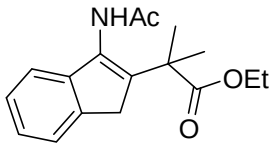


5f **5h**

Diisobutylaluminum hydride solution (1.0 M in hexane, 0.9 mL, 0.9 mmol) was added dropwise to a solution of **5f** (0.0576 g, 0.208 mmol) in dichloromethane (2 mL) at -78 °C. After stirring for 10 minutes at the same temperature, the reaction mixture was warmed to 0 °C and stirred for an additional 30 minutes. The methanol (1mL) and hydrochloric acid (3 M, 3 mL) were successively added. The reaction mixture was partitioned by dichloromethane, and then the aqueous phase was extracted twice with dichloromethane (3×10 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered and concentrated. The residue was purified by flash column chromatography on silica gel to give **5h** (0.020 g , 43%)

7. NMR data and spectra of the products:

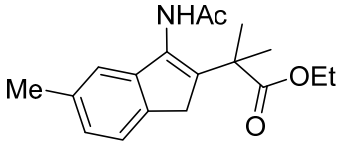
Ethyl 2-(3-acetamido-1*H*-inden-2-yl)-2-methylpropanoate (3a)



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.00 (s, 1H), 7.44 (d, J = 7.2 Hz, 1H), 7.24 (dd, J = 7.4, 1.3 Hz, 1H), 7.21 – 7.14 (m, 1H), 7.09 – 7.03 (m, 1H), 4.07 (q, J = 7.1 Hz, 2H), 3.54 – 3.44 (m, 2H), 2.04 (s, 3H), 1.46 (s, 6H), 1.19 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.35, 169.65, 144.10, 144.06, 140.92, 133.55, 126.74, 125.38, 124.33, 119.79, 61.26, 44.12, 37.58, 26.34, 23.44, 14.82.

HRMS (ESI, m/z): calculated for $C_{17}H_{21}NO_3$ $[M+Na]^+$ 310.1419, found:310.1416.

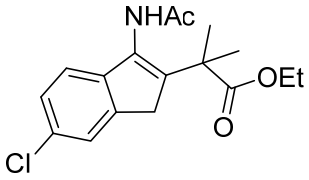
Ethyl 2-(3-acetamido-5-methyl-1*H*-inden-2-yl)-2-methylpropanoate (3b)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.95 (s, 1H), 7.31 (d, J = 7.5 Hz, 1H), 7.01 (d, J = 7.5 Hz, 1H), 6.88 (d, J = 3.0 Hz, 1H), 4.07 (q, J = 7.1 Hz, 2H), 3.43 (s, 2H), 2.34 (s, 3H), 2.04 (d, J = 2.2 Hz, 3H), 1.45 (d, J = 2.3 Hz, 6H), 1.19 (t, J = 7.1 Hz, 2H). ^{13}C NMR (101 MHz, DMSO) δ 176.35, 169.62, 144.35, 144.31, 138.02, 135.72, 133.50, 126.16, 124.04, 120.21, 61.22, 44.15, 37.18, 26.31, 23.45, 22.08, 14.81.

HRMS (ESI, m/z): calculated for $C_{18}H_{23}NO_3$ $[M+Na]^+$ 324.1576, found: 324.1573.

Ethyl 2-(3-acetamido-6-chloro-1*H*-inden-2-yl)-2-methylpropanoate (3c)

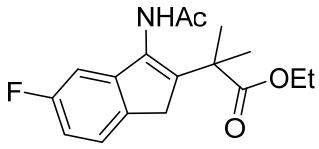


^{1}H NMR (400 MHz, DMSO-d_6) δ 9.05 (s, 1H), 7.50 (d, J = 2.0 Hz, 1H), 7.31 (dd, J = 8.2, 2.0 Hz, 1H), 7.05 (d, J = 8.1 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 3.53 (s, 2H), 2.04 (s, 3H), 1.45 (s, 6H), 1.18 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.16,

169.71, 144.81, 143.06, 142.96, 132.95, 130.14, 126.84, 124.55, 121.19, 61.33, 44.16, 37.57, 26.26, 23.41, 14.80.

HRMS (ESI, m/z): calculated for $C_{17}H_{20}NClO_3$ $[M+Na]^+$344.1029, found: 344.1014.

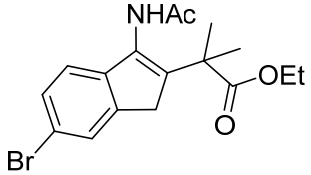
Ethyl 2-(3-acetamido-5-fluoro-1*H*-inden-2-yl)-2-methylpropanoate (3d)



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.05 (s, 1H), 7.43 (dd, *J* = 8.2, 5.1 Hz, 1H), 7.00 (ddd, *J* = 9.9, 8.2, 2.5 Hz, 1H), 6.83 (dd, *J* = 9.2, 2.5 Hz, 1H), 4.07 (q, *J* = 7.1 Hz, 2H), 3.49 (s, 2H), 2.05 (s, 3H), 1.45 (s, 6H), 1.18 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.16, 169.78, 162.44 (*J* = 242 Hz), 146.74, 146.31 (*J* = 9 Hz), 136.62, 133.12, 125.57 (*J* = 9 Hz), 111.98 (*J* = 23 Hz), 106.79 (*J* = 24 Hz), 61.33, 44.24, 37.09, 26.28, 23.43, 14.80.

HRMS (ESI, m/z): calculated for $C_{17}H_{20}NFO_3$ $[M+Na]^+$ 328.1325, found: 328.1318.

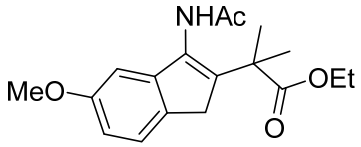
Ethyl 2-(3-acetamido-6-bromo-1*H*-inden-2-yl)-2-methylpropanoate (3e)



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.05 (s, 1H), 7.63 (d, *J* = 1.8 Hz, 1H), 7.44 (dd, *J* = 8.1, 1.9 Hz, 1H), 7.00 (d, *J* = 8.1 Hz, 1H), 4.06 (q, *J* = 7.1 Hz, 2H), 3.53 (s, 2H), 2.03 (s, 3H), 1.45 (s, 6H), 1.18 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.13, 169.71, 144.83, 143.45, 143.33, 133.02, 129.63, 127.36, 121.64, 118.50, 61.33, 44.14, 37.59, 26.25, 23.40, 14.80.

HRMS (ESI, m/z): calculated for $C_{17}H_{20}NBrO_3$ $[M+Na]^+$ 388.0524, found: 388.0527.

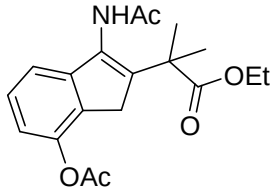
Ethyl 2-(3-acetamido-5-methoxy-1*H*-inden-2-yl)-2-methylpropanoate (3f)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.97 (s, 1H), 7.32 (d, J = 8.1 Hz, 1H), 6.77 (dd, J = 8.1, 2.4 Hz, 1H), 6.61 (d, J = 2.4 Hz, 1H), 4.07 (q, J = 7.1 Hz, 2H), 3.76 (s, 3H), 3.41 (d, J = 1.7 Hz, 2H), 2.04 (d, J = 2.7 Hz, 3H), 1.45 (s, 6H), 1.19 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.31, 169.67, 159.27, 145.61, 145.56, 133.45, 132.95, 124.87, 111.34, 105.46, 61.25, 56.14, 44.21, 36.81, 26.29, 23.46, 14.81.

HRMS (ESI, m/z): calculated for $C_{18}H_{23}NO_4$ $[M+Na]^+$ 340.1525, found: 340.1526.

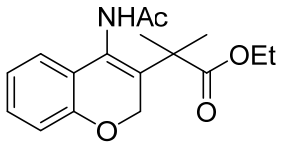
Ethyl 2-(3-acetamido-7-acetoxy-1*H*-inden-2-yl)-2-methylpropanoate (3g)



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.01 (s, 1H), 7.27 (t, J = 7.8 Hz, 1H), 6.93 (dd, J = 12.6, 7.7 Hz, 2H), 4.03 (q, J = 7.1 Hz, 2H), 3.34 (s, 2H), 2.33 (s, 3H), 2.01 (s, 3H), 1.41 (s, 6H), 1.15 (t, J = 7.1 Hz, 3H).^{13}C NMR (101 MHz, DMSO) δ 176.15, 169.76, 169.60, 146.90, 146.36, 144.53, 133.40, 132.12, 128.47, 119.13, 117.80, 61.32, 44.09, 35.03, 26.28, 23.40, 21.66, 14.78.

HRMS (ESI, m/z): calculated for $C_{19}H_{23}NO_5$ $[M+Na]^+$ 368.1474, found: 368.1472.

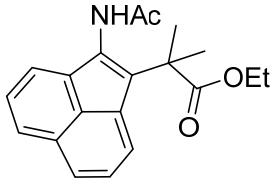
Ethyl 2-(4-acetamido-2*H*-chromen-3-yl)-2-methylpropanoate (3h)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.70 (s, 1H), 7.16 (td, J = 7.6, 1.7 Hz, 1H), 7.02 (dd, J = 7.7, 1.7 Hz, 1H), 6.92 (td, J = 7.5, 1.2 Hz, 1H), 6.83 (dd, J = 8.1, 1.2 Hz, 1H), 4.85 (s, 2H), 4.09 (t, J = 7.1 Hz, 2H), 1.97 (s, 3H), 1.32 (s, 6H), 1.22 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.23, 170.33, 154.48, 133.93, 129.79, 126.21, 124.18, 123.61, 122.07, 116.02, 66.26, 61.37, 45.44, 24.88, 23.33, 14.83.

HRMS (ESI, m/z): calculated for $C_{17}H_{21}NO_4$ $[M+Na]^+$ 326.1368, found: 326.1360.

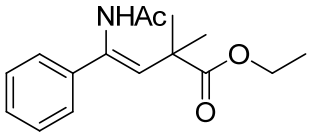
Ethyl 2-(2-acetamidoacenaphthylen-1-yl)-2-methylpropanoate (3i)



^{1}H NMR (400 MHz, DMSO-d6) δ 9.47 (s, 1H), 8.00 – 7.79 (m, 2H), 7.57 (t, *J* = 21.2 Hz, 4H), 4.11 (d, *J* = 6.3 Hz, 2H), 2.13 (s, 3H), 1.70 (s, 6H), 1.11 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.97, 170.35, 138.02, 137.79, 137.26, 134.65, 128.53, 128.46, 128.34, 128.06, 127.82, 126.67, 124.27, 123.35, 61.39, 44.89, 26.74, 23.71, 14.80.

HRMS (ESI, m/z): calculated for $C_{20}H_{21}NO_3$ $[M+Na]^+$ 346.1419, found: 346.1415.

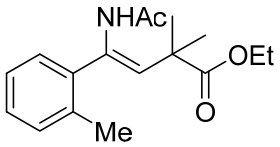
Ethyl (Z)-4-acetamido-2,2-dimethyl-4-phenylbut-3-enoate (3j)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.84 (s, 1H), 7.45 – 7.26 (m, 5H), 5.98 (d, *J* = 1.3 Hz, 1H), 4.07 (q, *J* = 7.1 Hz, 2H), 1.95 (s, 3H), 1.36 (s, 6H), 1.20 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.62, 169.73, 139.30, 134.89, 131.90, 129.09, 128.41, 126.14, 61.12, 43.55, 27.10, 23.45, 14.86.

HRMS (ESI, m/z): calculated for $C_{16}H_{21}NO_3$ $[M+Na]^+$ 298.1419, found: 298.1413.

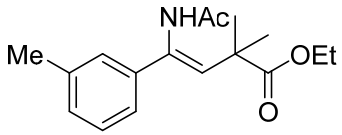
Ethyl (Z)-4-acetamido-2,2-dimethyl-4-(o-tolyl)but-3-enoate (3k)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.85 (s, 1H), 7.22 – 7.12 (m, 4H), 5.27 (d, *J* = 1.2 Hz, 1H), 4.06 (q, *J* = 7.1 Hz, 2H), 2.35 (s, 3H), 1.86 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.73, 168.93, 140.78, 136.18, 134.98, 132.74, 130.90, 129.59, 127.97, 126.12, 61.15, 43.42, 26.99, 23.43, 20.97, 14.81.

HRMS (ESI, m/z): calculated for $C_{17}H_{23}NO_3$ $[M+Na]^+$ 312.1576, found: 312.1578.

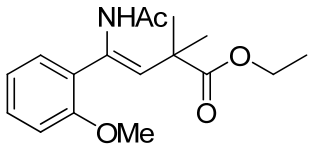
Ethyl (Z)-4-acetamido-2,2-dimethyl-4-(m-tolyl)but-3-enoate (3l)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.80 (s, 1H), 7.26 – 7.19 (m, 2H), 7.17 (d, J = 7.8 Hz, 1H), 7.11 (d, J = 7.4 Hz, 1H), 5.95 (d, J = 1.3 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 2.34 (s, 3H), 1.94 (s, 3H), 1.35 (s, 6H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.62, 169.69, 139.34, 138.08, 134.93, 131.74, 129.08, 128.97, 126.70, 123.38, 61.10, 43.52, 27.13, 23.46, 22.01, 14.86.

HRMS (ESI, m/z): calculated for $C_{17}H_{23}NO_3$ $[M+Na]^+$312.1576, found: 312.1576.

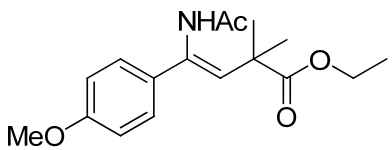
Ethyl (*Z*)-4-acetamido-4-(2-methoxyphenyl)-2,2-dimethylbut-3-enoate (3m)



^{1}H NMR (400 MHz, DMSO-d6) δ 8.62 (s, 1H), 7.28 – 7.13 (m, 2H), 7.02 – 6.90 (m, 2H), 5.75 (d, J = 1.3 Hz, 1H), 4.07 (q, J = 7.1 Hz, 2H), 3.80 (s, 3H), 1.87 (s, 3H), 1.36 (s, 6H), 1.21 (t, J = 7.1 Hz, 3H).^{13}C NMR (101 MHz, DMSO) δ 176.75, 169.24, 157.47, 133.67, 132.84, 130.33, 129.47, 128.91, 121.11, 112.74, 61.09, 56.59, 43.57, 27.08, 23.48, 14.88.

HRMS (ESI, m/z): calculated for $C_{17}H_{23}NO_4$ $[M+Na]^+$ 328.1525, found: 328.1520.

Ethyl (*Z*)-4-acetamido-4-(4-methoxyphenyl)-2,2-dimethylbut- 3-enoate (3n)

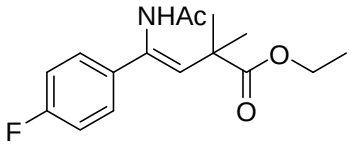


^{1}H NMR (400 MHz, DMSO-d_6) δ 8.76 (s, 1H), 7.32 (d, J = 8.7 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 6.32 (s, 0H), 5.86 (d, J = 1.2 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 3.78 (s, 3H), 1.94 (s, 3H), 1.35 (s, 6H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO)

δ 176.71, 169.66, 159.77, 134.50, 131.74, 129.96, 127.40, 114.45, 61.07, 56.05, 43.47, 27.19, 23.47, 14.87.

HRMS (ESI, m/z): calculated for $C_{17}H_{23}NO_4$ $[M+Na]^+$ 328.1525, found: 328.1523.

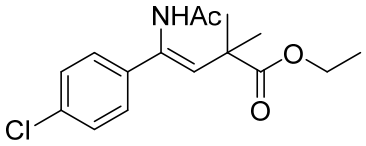
Ethyl (*Z*)-4-acetamido-4-(4-fluorophenyl)-2,2-dimethylbut-3-enoate (3o)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.86 (s, 1H), 7.42 (dd, J = 8.8, 5.6 Hz, 2H), 7.18 (t, J = 8.8 Hz, 2H), 5.94 (d, J = 1.3 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 1.95 (s, 3H), 1.35 (s, 6H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.56, 169.74, 162.58 (J = 242 Hz), 135.83, 135.80, 134.01, 131.78, 128.14 (J = 8 Hz), 115.85 (J = 22 Hz), 61.14, 43.54, 27.09, 23.44, 14.85.

HRMS (ESI, m/z): calculated for $C_{16}H_{20}FNO_3$ $[M+Na]^+$ 316.1325, found: 316.1326.

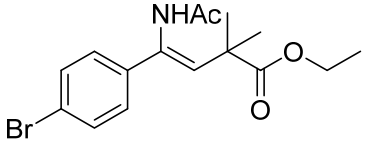
Ethyl (*Z*)-4-acetamido-4-(4-chlorophenyl)-2,2-dimethylbut-3-enoate (3p)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.88 (s, 1H), 7.41 (s, 4H), 4.06 (q, J = 7.1 Hz, 2H), 1.95 (s, 3H), 1.36 (s, 6H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.49, 169.77, 138.25, 133.97, 132.88, 132.50, 129.06, 127.93, 61.17, 43.58, 27.05, 23.43, 14.84.

HRMS (ESI, m/z): calculated for $C_{16}H_{20}ClNO_3$ $[M+Na]^+$ 332.1029, found: 332.1032

Ethyl (*Z*)-4-acetamido-4-(4-bromophenyl)-2,2-dimethylbut-3-enoate (3q)

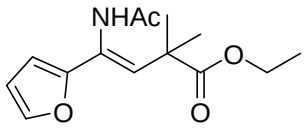


^{1}H NMR (400 MHz, DMSO-d_6) δ 8.89 (s, 1H), 7.54 (d, J = 8 Hz, 2H), 7.34 (d, J = 8Hz, 2H), 6.01 (d, J = 1.3 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 1.95 (s, 3H), 1.36 (s, 6H),

1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.48, 169.77, 138.65, 134.06, 132.51, 131.97, 128.27, 121.43, 61.17, 43.59, 27.04, 23.42, 14.84.

HRMS (ESI, m/z): calculated for $C_{16}H_{20}BrNO_3$ $[M+Na]^+$ 376.0524, found: 376.0526.

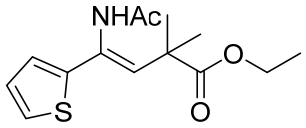
Ethyl (*Z*)-4-acetamido-4-(furan-2-yl)-2,2-dimethylbut-3-enoate (3r)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.83 (s, 1H), 7.64 (dd, J = 1.9, 0.8 Hz, 1H), 6.49 (dd, J = 3.4, 1.8 Hz, 1H), 6.28 (dd, J = 3.3, 0.8 Hz, 1H), 6.04 (d, J = 1.1 Hz, 1H), 4.06 (q, J = 7.1 Hz, 2H), 1.93 (s, 3H), 1.33 (s, 6H), 1.20 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, DMSO) δ 176.30, 170.07, 152.72, 143.59, 129.90, 126.48, 112.46, 107.73, 61.22, 43.14, 26.95, 23.42, 14.84.

HRMS (ESI, m/z): calculated for $C_{14}H_{19}NO_4$ $[M+Na]^+$ 288.1212, found: 288.1207

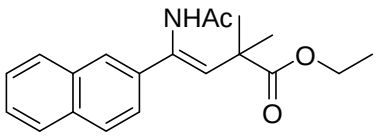
Ethyl (*Z*)-4-acetamido-2,2-dimethyl-4-(thiophen-2-yl)but-3-enoate (3s)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.93 (s, 1H), 7.38 (dd, J = 4.9, 1.4 Hz, 1H), 7.03 – 6.93 (m, 2H), 5.95 (d, J = 1.3 Hz, 1H), 4.03 (q, J = 7.1 Hz, 2H), 1.90 (s, 3H), 1.30 (s, 6H), 1.17 (t, J = 7.1 Hz, 3H).^{13}C NMR (101 MHz, DMSO) δ 176.28, 169.85, 144.36, 130.99, 129.93, 128.46, 125.91, 124.64, 61.22, 43.49, 26.97, 23.39, 14.86.

HRMS (ESI, m/z): calculated for $C_{14}H_{19}NSO_3$ $[M+Na]^+$ 304.0983, found: 304.0982

Ethyl (*Z*)-4-acetamido-2,2-dimethyl-4-(naphthalen-2-yl)but-3-enoate (3t)

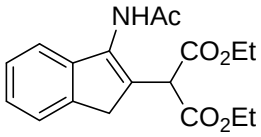


^{1}H NMR (400 MHz, DMSO-d_6) δ 8.96 (s, 1H), 7.98 – 7.85 (m, 4H), 7.67 – 7.59 (m, 1H), 7.58 – 7.48 (m, 2H), 6.17 (s, 1H), 4.09 (q, J = 7.1 Hz, 2H), 2.02 (s, 3H), 1.41 (s,

6H), 1.22 (t, J = 7.1 Hz, 3H).^{13}C NMR (101 MHz, DMSO) δ 176.64, 169.88, 136.75, 134.91, 133.77, 133.34, 132.62, 128.99, 128.55, 128.29, 127.21, 126.88, 124.77, 124.62, 61.18, 43.68, 27.19, 23.54, 14.88.

HRMS (ESI, m/z): calculated for $C_{20}H_{23}NO_3$ $[M+Na]^+$ 348.1576, found: 348.1577.

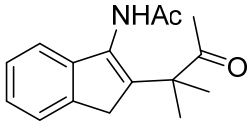
Diethyl 2-(3-acetamido-1*H*-inden-2-yl)malonate (4a)



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.72 (s, 1H), 7.50 (d, J = 7.2 Hz, 1H), 7.40 – 7.24 (m, 3H), 4.83 (s, 1H), 4.19 (tq, J = 7.1, 3.0 Hz, 4H), 3.56 (s, 2H), 2.13 (s, 3H), 1.23 (t, J = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, DMSO) δ 169.34, 168.35, 142.37, 142.00, 137.05, 127.39, 126.86, 126.28, 124.63, 120.47, 62.20, 52.53, 37.80, 23.82, 14.85.

HRMS (ESI, m/z): calculated for $C_{18}H_{21}NO_3$ $[M+Na]^+$354.1317, found: 354.1318.

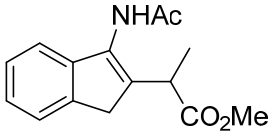
***N*-(2-(2-methyl-3-oxobutan-2-yl)-1*H*-inden-3-yl)acetamide (4b)**



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.02 (s, 1H), 7.46 (d, J = 7.2 Hz, 1H), 7.23 (ddd, J = 15.7, 7.3, 1.3 Hz, 2H), 7.07 (dd, J = 7.0, 1.2 Hz, 1H), 3.57 (d, J = 1.7 Hz, 2H), 2.06 (s, 3H), 2.03 (s, 3H), 1.37 (s, 6H). ^{13}C NMR (101 MHz, DMSO) δ 210.51, 169.98, 144.58, 143.98, 141.31, 134.19, 126.75, 125.43, 124.38, 119.81, 50.12, 37.77, 26.33, 25.15, 23.34.

HRMS (ESI, m/z): calculated for $C_{16}H_{19}NO_2$[M+Na]$^+$ 280.1313, found: 280.1316.

Methyl 2-(3-acetamido-1*H*-inden-2-yl)propanoate (4c)

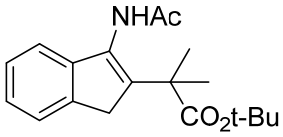


^{1}H NMR (400 MHz, DMSO-d6) δ 9.50 (s, 1H), 7.44 (d, J = 7.2 Hz, 1H), 7.32 – 7.16 (m, 3H), 3.85 (q, J = 7.2 Hz, 1H), 3.63 (s, 3H), 3.47 (d, J = 22.9 Hz, 1H), 3.36 (d, J =

22.9 Hz, 1H), 2.11 (s, 3H), 1.38 (d, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 174.73, 169.38, 143.01, 141.83, 136.65, 134.45, 126.77, 125.57, 124.51, 120.12, 52.66, 38.94, 36.39, 23.71, 17.74.

HRMS (ESI, m/z): calculated for $C_{15}H_{17}NO_3$ $[M+Na]^+$ 282.1106, found: 280.1110.

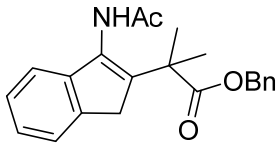
***Tert*-butyl 2-(3-acetamido-1*H*-inden-2-yl)-2-methylpropanoate (4d)**



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.00 (s, 1H), 7.43 (d, J = 7.2 Hz, 1H), 7.25 (td, J = 7.5, 1.2 Hz, 1H), 7.18 (td, J = 7.3, 1.3 Hz, 1H), 7.09 – 7.04 (m, 1H), 3.46 (d, J = 1.6 Hz, 2H), 2.05 (s, 3H), 1.44 (s, 6H), 1.42 (s, 9H). ^{13}C NMR (101 MHz, DMSO) δ 175.43, 169.62, 144.21, 143.67, 140.95, 133.45, 126.68, 125.30, 124.28, 119.88, 80.71, 45.16, 37.96, 28.39, 26.42, 23.67.

HRMS (ESI, m/z): calculated for $C_{19}H_{25}NO_3$ $[M+Na]^+$ 338.1732, found: 338.1735.

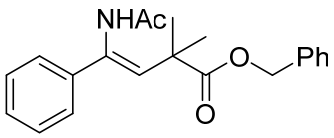
Benzyl 2-(3-acetamido-1*H*-inden-2-yl)-2-methylpropanoate (4e)



^{1}H NMR (400 MHz, DMSO-d_6) δ 9.05 (s, 1H), 7.44 – 7.34 (m, 6H), 7.26 (td, J = 7.4, 1.2 Hz, 1H), 7.19 (td, J = 7.4, 1.3 Hz, 1H), 7.12 – 7.07 (m, 1H), 5.12 (s, 2H), 3.49 (d, J = 1.7 Hz, 2H), 2.07 (s, 3H), 1.50 (s, 6H). ^{13}C NMR (101 MHz, DMSO) δ 176.09, 169.81, 144.09, 143.97, 140.93, 137.23, 133.70, 129.28, 128.77, 128.59, 126.77, 125.45, 124.33, 119.73, 66.71, 44.28, 37.63, 26.23, 23.46.

HRMS (ESI, m/z): calculated for $C_{22}H_{23}NO_3$ $[M+Na]^+$ 372.1576, found: 372.1578.

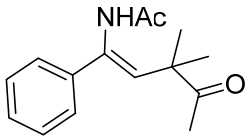
Benzyl (Z)-4-acetamido-2,2-dimethyl-4-phenylbut-3-enoate (4f)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.91 (s, 1H), 7.44 – 7.32 (m, 10H), 6.09 – 5.97 (m, 1H), 5.11 (s, 2H), 1.98 (d, J = 1.6 Hz, 3H), 1.40 (s, 6H). ^{13}C NMR (101 MHz, DMSO) δ 176.39, 169.90, 139.23, 137.41, 135.07, 131.81, 129.29, 129.11, 128.74, 128.53, 128.48, 126.15, 66.59, 43.68, 27.02, 23.49.

HRMS (ESI, m/z): calculated for $C_{21}H_{23}NO_3$ $[M+Na]^+$ 360.1576, found: 360.1579.

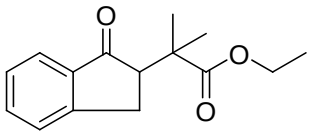
(*Z*)-*N*-(3,3-dimethyl-4-oxo-1-phenylpent-1-en-1-yl) acetamide (4g)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.91 (s, 1H), 7.45 – 7.39 (m, 2H), 7.39 – 7.33 (m, 2H), 7.33 – 7.27 (m, 1H), 6.06 (d, J = 1.3 Hz, 1H), 2.10 (s, 3H), 1.92 (s, 3H), 1.28 (s, 6H). ^{13}C NMR (101 MHz, DMSO) δ 210.78, 170.00, 139.30, 135.78, 132.31, 129.10, 128.50, 126.23, 49.48, 26.50, 26.12, 23.30.

HRMS (ESI, m/z): calculated for $C_{15}H_{19}NO_2$ $[M+Na]^+$ 268.1313, found: 268.1314.

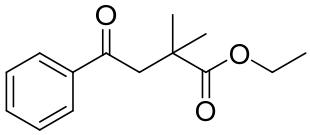
Ethyl 2-methyl-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl) propanoate (5a)



^{1}H NMR (400 MHz, Chloroform-d) δ 7.72 (ddd, J = 7.6, 1.3, 0.7 Hz, 1H), 7.57 (td, J = 7.5, 1.2 Hz, 1H), 7.44 (dt, J = 7.6, 1.0 Hz, 1H), 7.35 (td, J = 7.4, 0.9 Hz, 1H), 4.13 (qd, J = 7.2, 0.7 Hz, 2H), 3.34 – 3.22 (m, 1H), 3.04 (dd, J = 8.1, 4.7 Hz, 1H), 2.90 (dd, J = 17.1, 4.7 Hz, 1H), 1.90 (s, 3H), 1.31 (s, 3H), 1.30 (s, 3H), 1.18 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.36, 176.77, 152.90, 137.68, 134.71, 127.51, 126.41, 123.82, 60.89, 53.73, 44.83, 30.42, 23.45, 22.66, 14.15.

HRMS (ESI, m/z): calculated for $C_{15}H_{18}O_3$ $[M+Na]^+$ 269.1154, found: 269.1154.

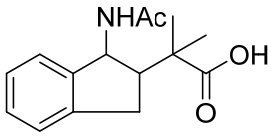
Ethyl 2,2-dimethyl-4-oxo-4-phenylbutanoate (5b)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.91 (m, 2H), 7.57 – 7.53 (m, 1H), 7.48 – 7.41 (m, 2H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.28 (s, 2H), 1.32 (s, 6H), 1.20 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.77, 177.42, 137.24, 133.15, 128.66, 128.04, 60.63, 48.58, 40.21, 25.89, 14.22.

HRMS (ESI, m/z): calculated for $C_{14}H_{18}O_3$ $[M+Na]^+$ 257.1154, found: 257.1153

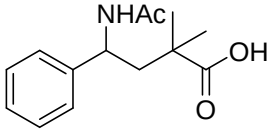
2-(1-acetamido-2,3-dihydro-1*H*-inden-2-yl)-2-methylpropanoic acid (5c)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 (d, *J* = 7.3 Hz, 1H), 7.24 – 7.14 (m, 3H), 6.37 (d, *J* = 9.9 Hz, 1H), 5.67 (dd, *J* = 10.0, 7.5 Hz, 1H), 3.43 (dd, *J* = 16.0, 9.9 Hz, 1H), 2.98 (dd, *J* = 16.0, 8.2 Hz, 1H), 2.69 (dt, *J* = 9.5, 7.9 Hz, 1H), 1.90 (s, 3H), 1.36 (s, 3H), 1.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.85, 170.33, 143.01, 142.60, 128.44, 127.16, 125.00, 124.75, 54.59, 51.93, 42.74, 32.85, 25.94, 25.65, 23.10.

HRMS (ESI, m/z): calculated for $C_{15}H_{19}NO_3$ $[M+Na]^+$ 284.1263, found: 284.1269

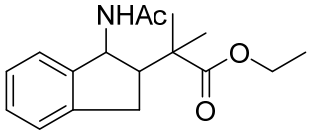
4-Acetamido-2,2-dimethyl-4-phenylbutanoic acid (5d)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 (d, *J* = 5.8 Hz, 4H), 7.23 (dd, *J* = 6.2, 2.6 Hz, 1H), 6.48 (d, *J* = 8.9 Hz, 1H), 5.18 (ddd, *J* = 11.1, 8.9, 3.9 Hz, 1H), 2.38 (dd, *J* = 14.5, 11.1 Hz, 1H), 1.92 (s, 3H), 1.77 (dd, *J* = 14.5, 3.9 Hz, 1H), 1.28 (s, 3H), 1.25 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.85, 170.33, 142.96, 128.77, 127.48, 126.40, , 50.66, 46.45, 41.24, 28.46, 23.17, 23.07.

HRMS (ESI, m/z): calculated for $C_{14}H_{19}NO_3$ $[M+Na]^+$ 272.1263, found: 272.1269

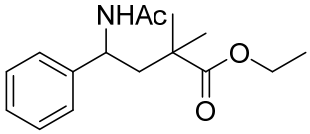
Ethyl 2-(1-acetamido-2,3-dihydro-1*H*-inden-2-yl)-2-methylpropanoate (5e)



^{1}H NMR (400 MHz, Chloroform-d) δ 7.38 – 7.31 (m, 1H), 7.24 – 7.15 (m, 3H), 6.19 (d, *J* = 9.8 Hz, 1H), 5.65 (dd, *J* = 9.9, 7.6 Hz, 1H), 4.18 – 4.02 (m, 2H)，3.46 (dd, *J* = 15.9, 9.7 Hz, 1H), 2.97 (dd, *J* = 15.9, 8.1 Hz, 1H), 2.63 (dt, *J* = 9.7, 7.9 Hz, 1H), 1.90 (s, 3H), 1.35 (s, 3H), 1.30 – 1.24 (m, 6H). ^{13}C NMR (101 MHz, CDCl3) δ 178.68, 169.08, 143.69, 142.45, 128.23, 127.08, 124.95, 124.68, 60.80, 54.39, 51.95, 42.90, 33.03, 26.45, 25.47, 23.59, 14.26.

HRMS (ESI, m/z): calculated for $C_{17}H_{23}NO_3$ $[M+Na]^+$ 312.1576, found: 312.1574.

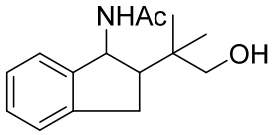
Ethyl 4-acetamido-2, 2-dimethyl-4-phenylbutanoate (5f)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.20 (m, 5H), 6.00 (d, *J* = 8.5 Hz, 1H), 5.09 (ddd, *J* = 10.9, 8.4, 4.4 Hz, 1H), 4.12 – 4.01 (m, 2H), 2.38 (dd, *J* = 14.5, 10.9 Hz, 1H), 1.90 (s, 3H), 1.75 (dd, *J* = 14.5, 4.5 Hz, 1H), 1.29 – 1.24 (m, 6H), 1.23 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.87, 169.01, 143.18, 128.72, 127.39, 126.44, 60.92, 50.78, 45.94, 41.31, 28.36, 23.45, 23.39, 14.27.

HRMS (ESI, m/z): calculated for $C_{16}H_{23}O_3$ $[M+Na]^+$ 300.1576, found: 300.1575

***N*-(2-(1-hydroxy-2-methylpropan-2-yl)-2,3-dihydro-1*H*-inden-1-yl)acetamide (5g)**

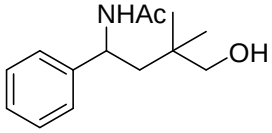


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.39 (m, 1H), 7.24 – 7.14 (m, 3H), 6.69 (s, 1H), 5.45 (t, *J* = 7.8 Hz, 1H), 3.58 (d, *J* = 10.6 Hz, 1H), 3.47 (d, *J* = 10.7 Hz, 1H), 3.04 (dd, *J* = 15.5, 11.4 Hz, 1H), 2.87 (dd, *J* = 15.5, 7.5 Hz, 1H), 2.51 (dt, *J* = 11.4, 7.1 Hz, 1H), 1.92 (s, 3H), 1.10 (s, 3H), 1.05 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ

169.40, 144.61, 142.62, 128.08, 127.08, 125.32, 124.69, 70.89, 54.88, 50.93, 36.86, 32.01, 24.79, 24.74, 23.92, 23.76.

HRMS (ESI, m/z): calculated for $C_{15}H_{21}O_2$ $[M+Na]^+$ 270.1470, found: 270.1476

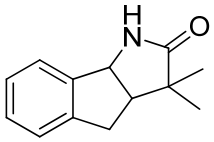
_N_-(4-hydroxy-3,3-dimethyl-1-phenylbutyl)acetamide (5h)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.21 (m, 6H), 6.44 (s, 1H), 4.91 (q, *J* = 6.6 Hz, 1H), 3.56 (d, *J* = 10.4 Hz, 1H), 3.23 (d, *J* = 11.5 Hz, 2H), 1.98 (dd, *J* = 14.7, 7.1 Hz, 1H), 1.93 (q, *J* = 0.9 Hz, 3H), 1.72 – 1.64 (m, 1H), 0.91 (s, 3H), 0.76 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.82, 143.69, 128.89, 127.52, 126.84, 70.40, 51.43, 44.52, 35.44, 27.03, 23.99, 23.41.

HRMS (ESI, m/z): calculated for $C_{14}H_{21}NO_2$ $[M+Na]^+$ 258.1470, found: 258.1475

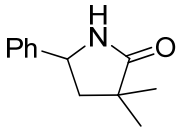
3, 3-Dimethyl-3, 3a, 4, 8b-tetrahydroindeno[1,2-b]pyrrol-2(1*H*)-one (5i)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.15 (m, 4H), 6.39 (s, 1H), 4.90 (d, *J* = 6.4 Hz, 1H), 3.06 – 2.88 (m, 3H), 1.29 (s, 3H), 1.11 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.15, 143.97, 141.47, 128.77, 127.06, 125.01, 124.78, 60.04, 51.25, 43.09, 33.13, 26.81, 20.69.

HRMS (ESI, m/z): calculated for $C_{13}H_{15}O$ $[M+Na]^+$ 224.1051, found: 224.1051.

3,3-Dimethyl-5-phenylpyrrolidin-2-one (5j)

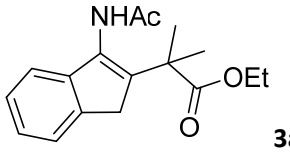


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 – 7.28 (m, 5H), 5.88 (s, 1H), 4.69 (dd, *J* = 8.5, 6.9 Hz, 1H), 2.38 (dd, *J* = 12.8, 6.9 Hz, 1H), 1.85 (dd, *J* = 12.8, 8.6 Hz, 1H), 1.26

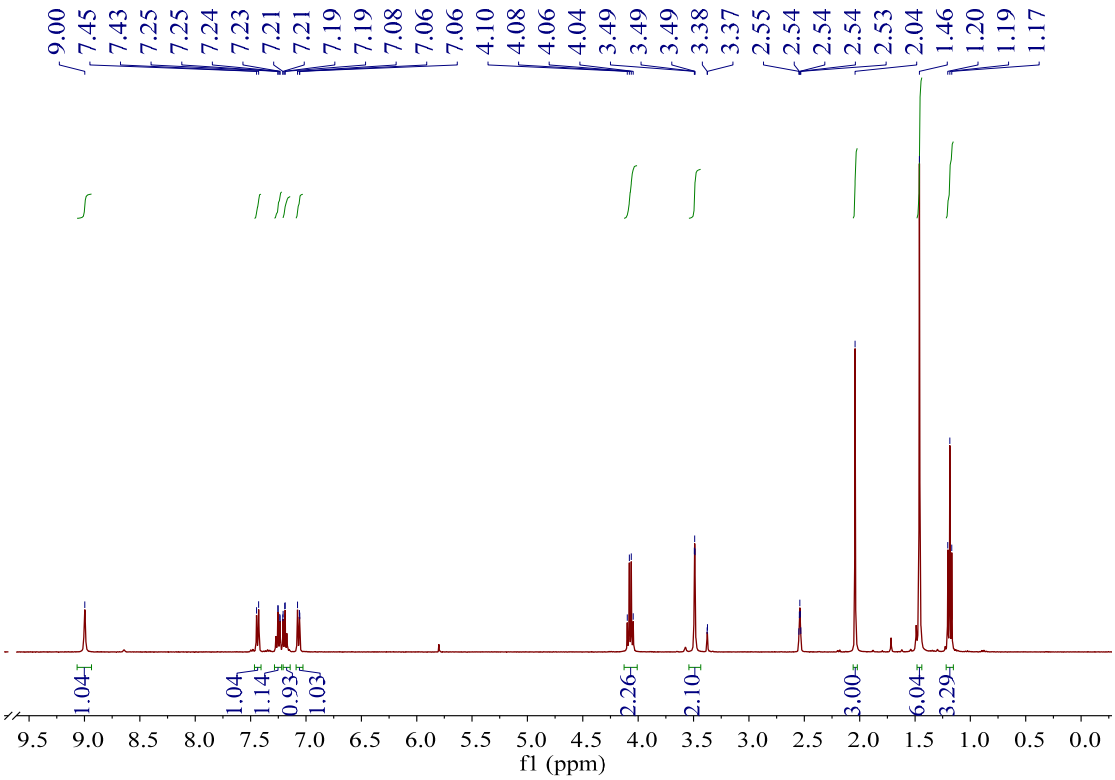
(s, 3H), 1.23 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 182.92, 142.44, 129.03, 128.03, 125.89, 54.88, 47.50, 41.05, 25.23, 24.65.

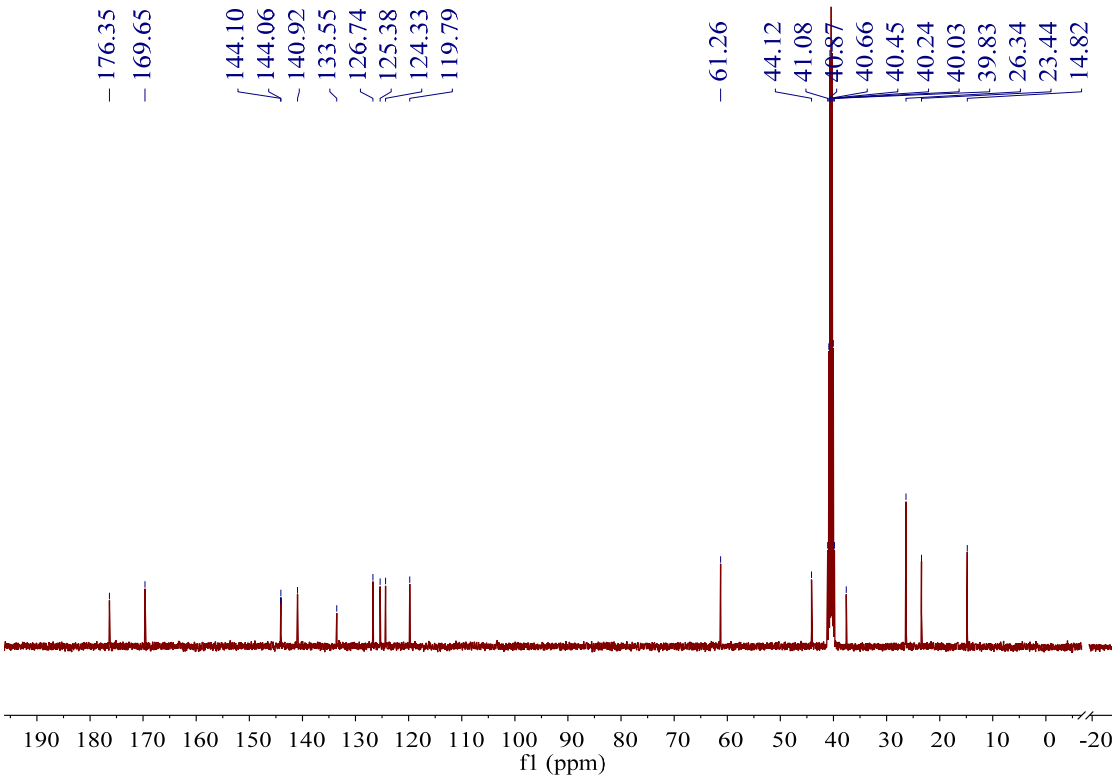
HRMS (ESI, m/z): calculated for $C_{12}H_{15}NO$ $[M+Na]^+$ 212.1051, found: 212.1050

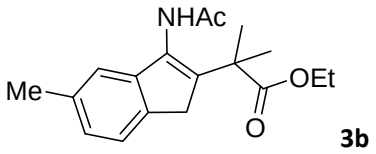
NMR spectra



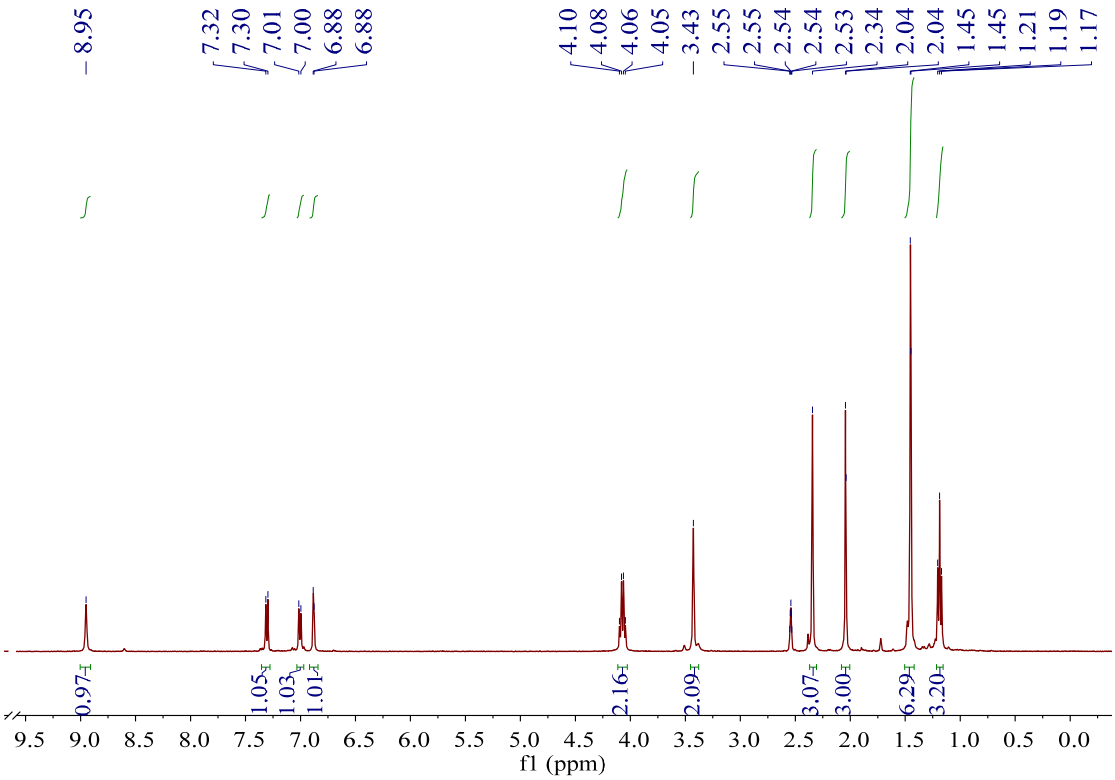
3a

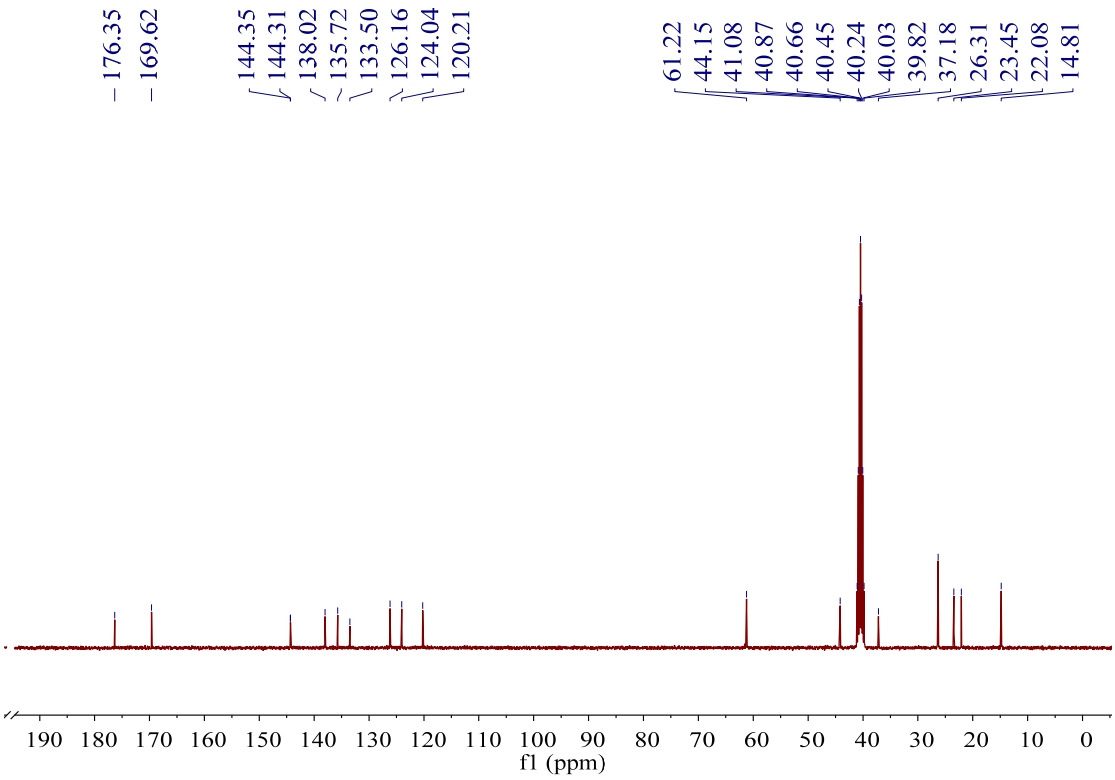


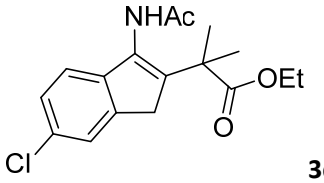



NHAc
Me
OEt
O

3b


8.95
7.32
7.30
7.01
7.00
6.88
6.88
4.10
4.08
4.06
4.05
3.43
2.55
2.55
2.54
2.54
2.53
2.34
2.04
2.04
1.45
1.45
1.21
1.19
1.17
0.97
1.05
1.03
1.01
2.16
2.09
3.07
3.00
6.29
3.20
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
f1 (ppm)


176.35
169.62
144.35
144.31
138.02
135.72
133.50
126.16
124.04
120.21
61.22
44.15
41.08
40.87
40.66
40.45
40.24
40.03
39.82
37.18
26.31
23.45
22.08
14.81
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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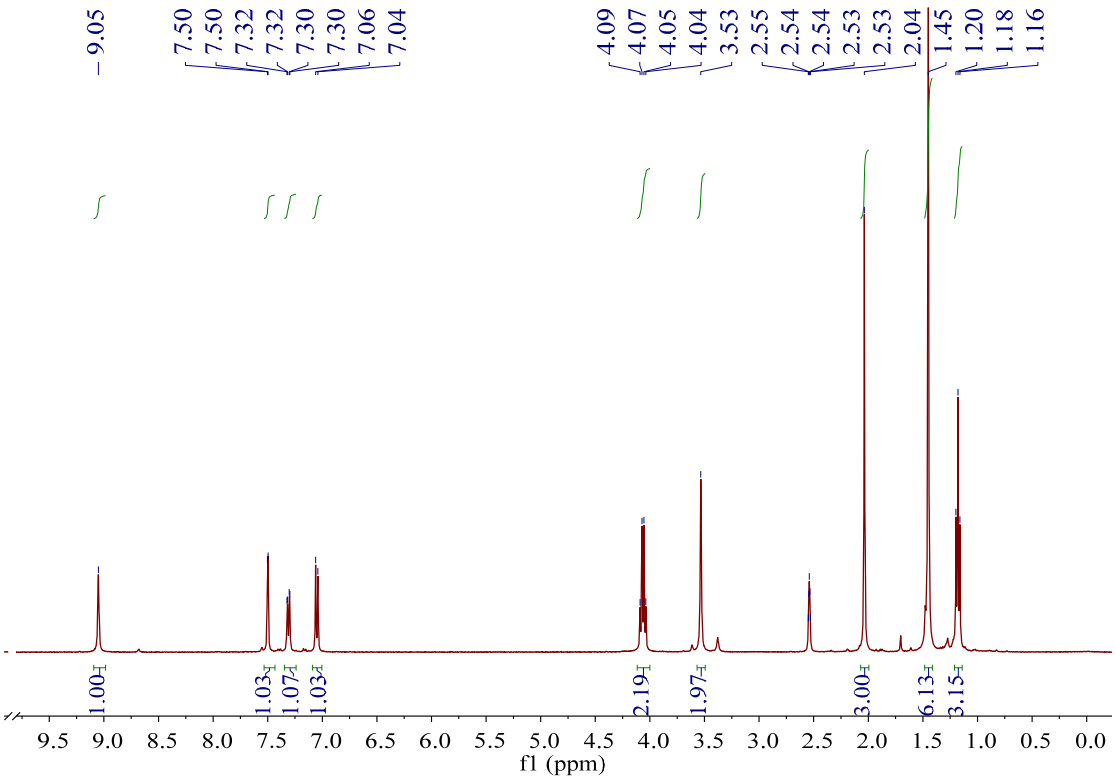
OEt
O
Cl

3c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7.04
4.09
4.07
4.05
4.04
3.53
2.55
2.54
2.54
2.53
2.53
2.04
1.45
1.20
1.18
1.16
1.00
1.03
1.07
1.03
2.19
1.97
3.00
6.13
3.15
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
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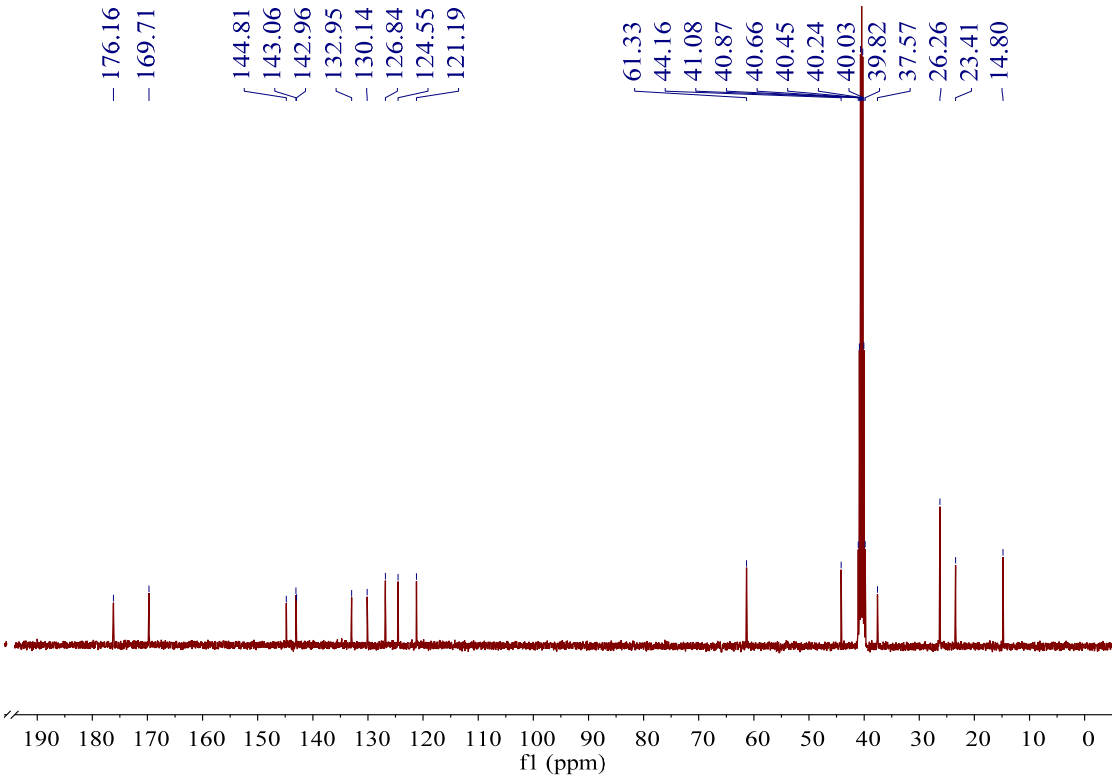
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124.55
121.19
61.33
44.16
41.08
40.87
40.66
40.45
40.24
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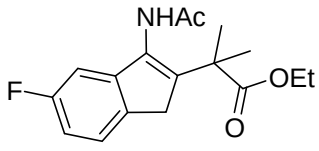
37.57
26.26
23.41
14.80
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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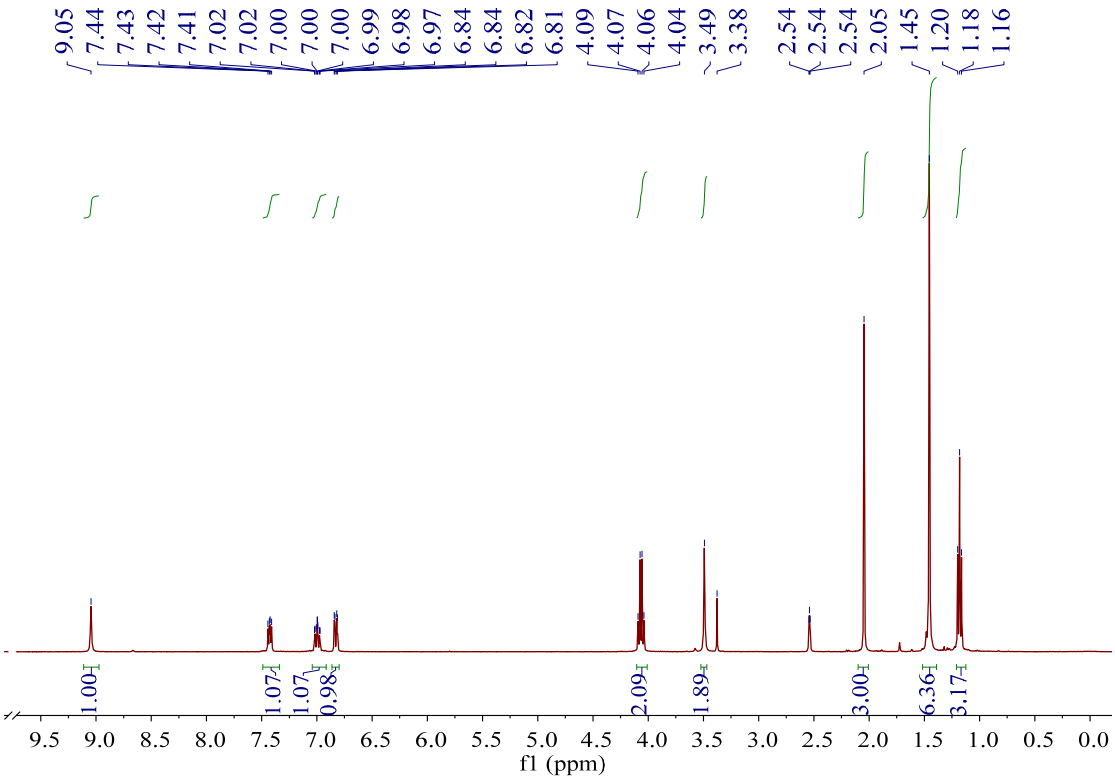
F
OEt
O

3d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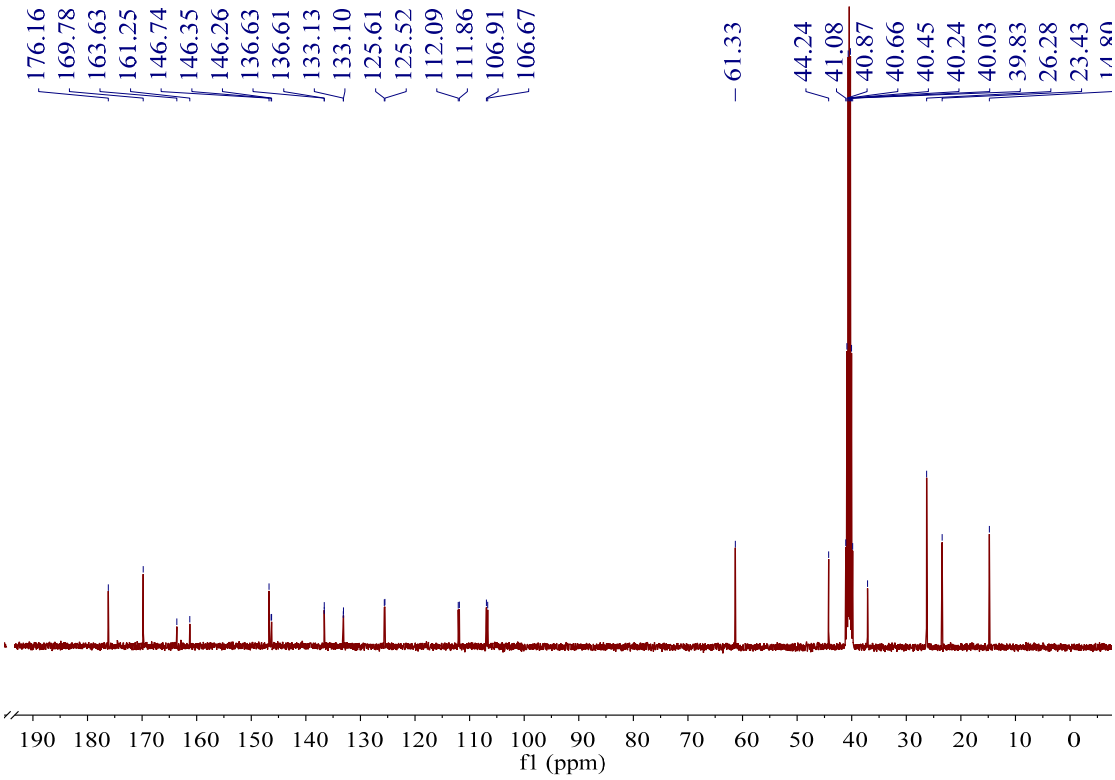
7.02
7.00
7.00
7.00
6.99
6.98
6.97
6.84
6.84
6.82
6.81
4.09
4.07
4.06
4.04
3.49
3.38
2.54
2.54
2.54
2.05
1.45
1.20
1.18
1.16
1.00
1.07
1.07
0.98
2.09
1.89
3.00
6.36
3.17
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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136.63
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133.10
125.61
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111.86
106.91
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61.33
44.24
41.08
40.87
40.66
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40.03
39.83
26.28
23.43
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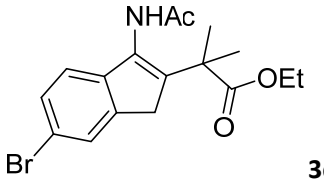
f1 (ppm)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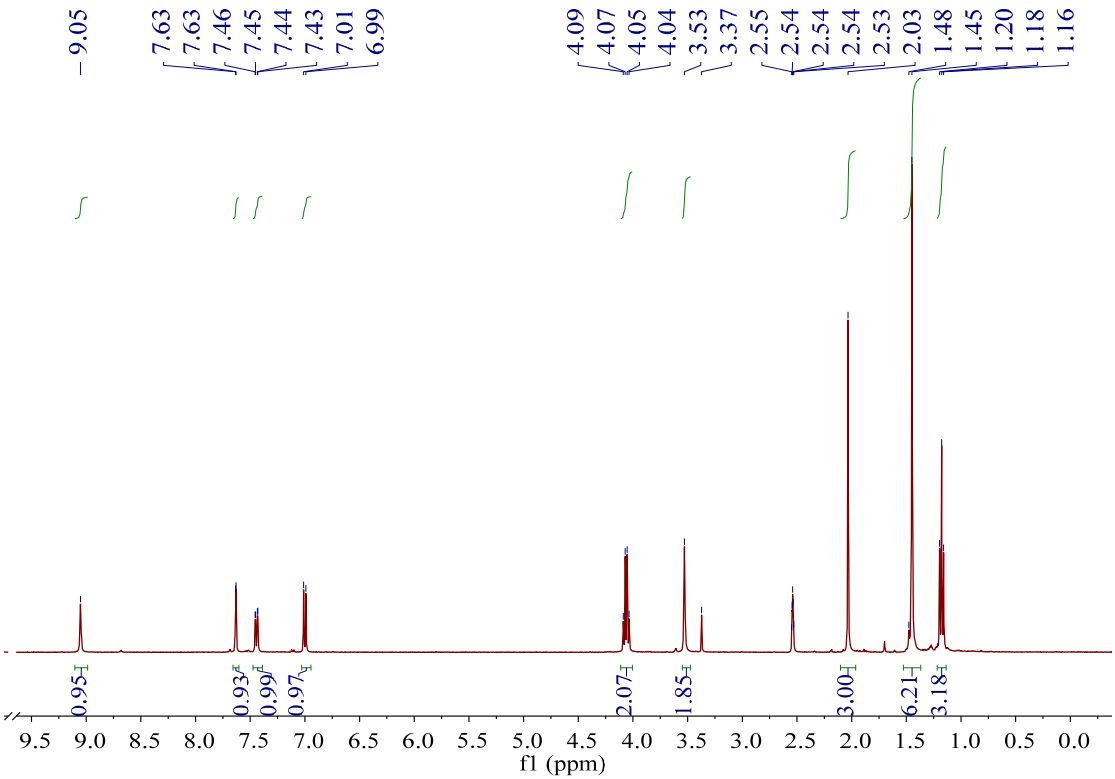
OEt
O
Br

3e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7.43
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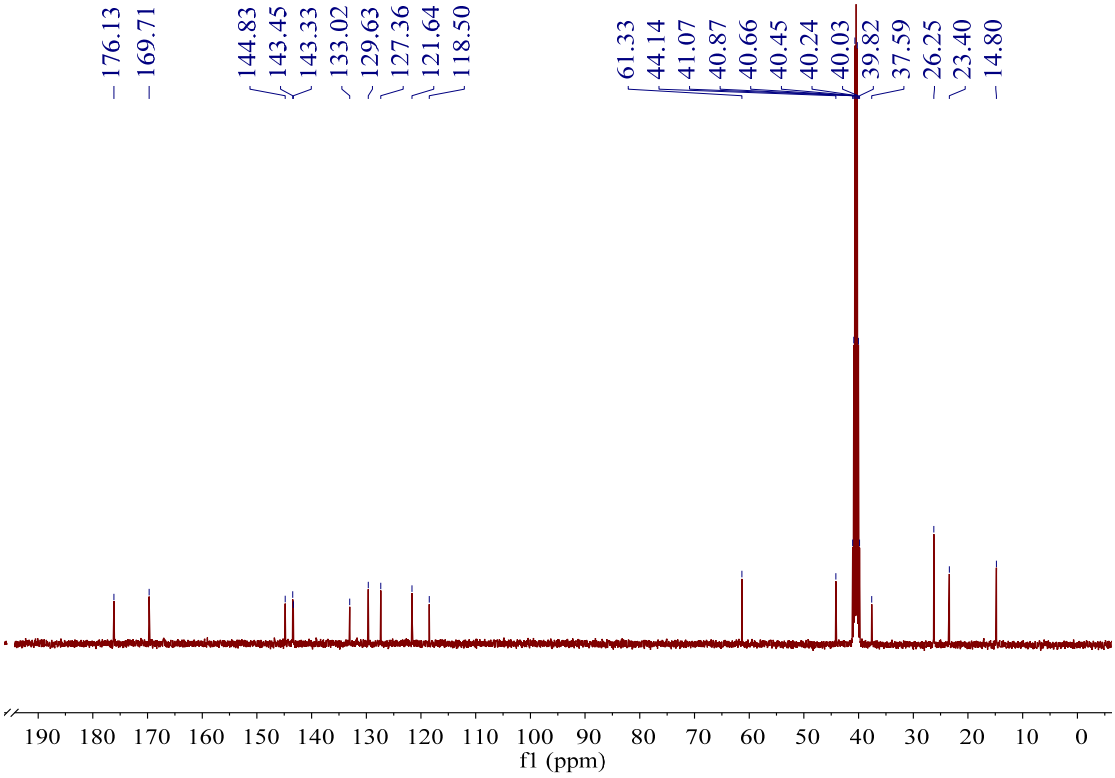
4.09
4.07
4.05
4.04
3.53
3.37
2.55
2.54
2.54
2.54
2.53
2.03
1.48
1.45
1.20
1.18
1.16
0.95
0.93
0.99
0.97
2.07
1.85
3.00
6.21
3.18
f1 (ppm)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127.36
121.64
118.50
61.33
44.14
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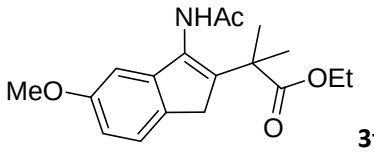
37.59
26.25
23.40
14.80
f1 (ppm)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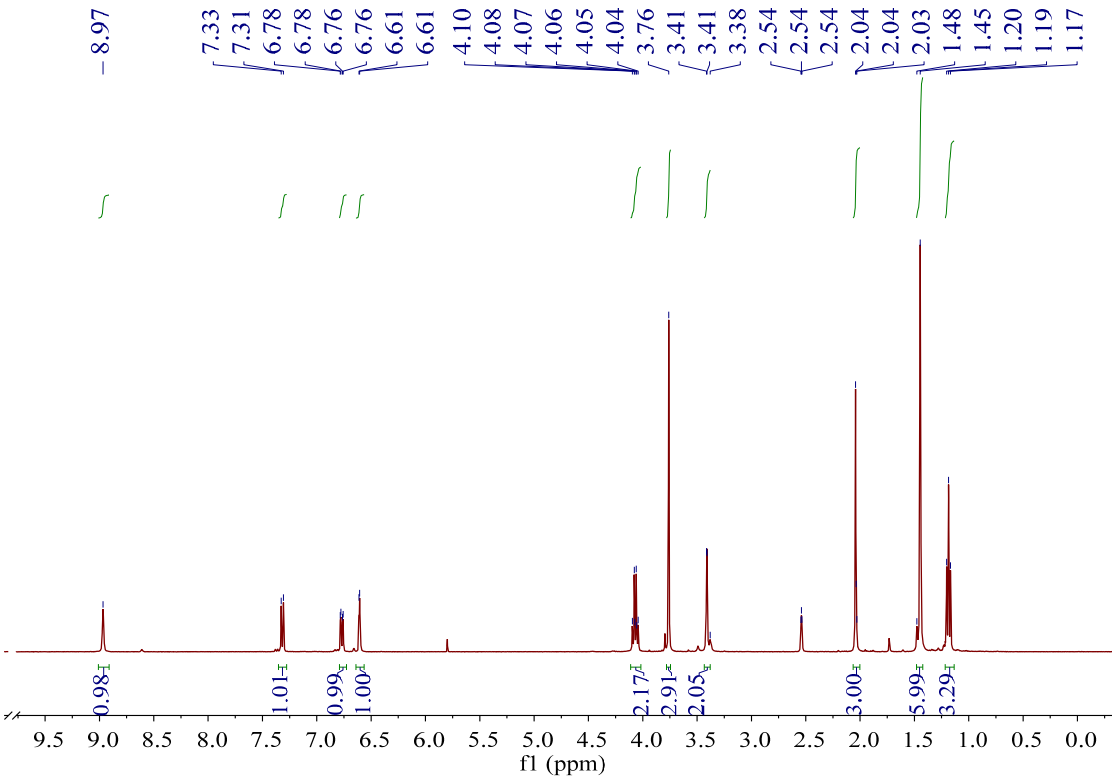
MeO
OEt
O

3f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6.76
6.76
6.61
6.61
4.10
4.08
4.07
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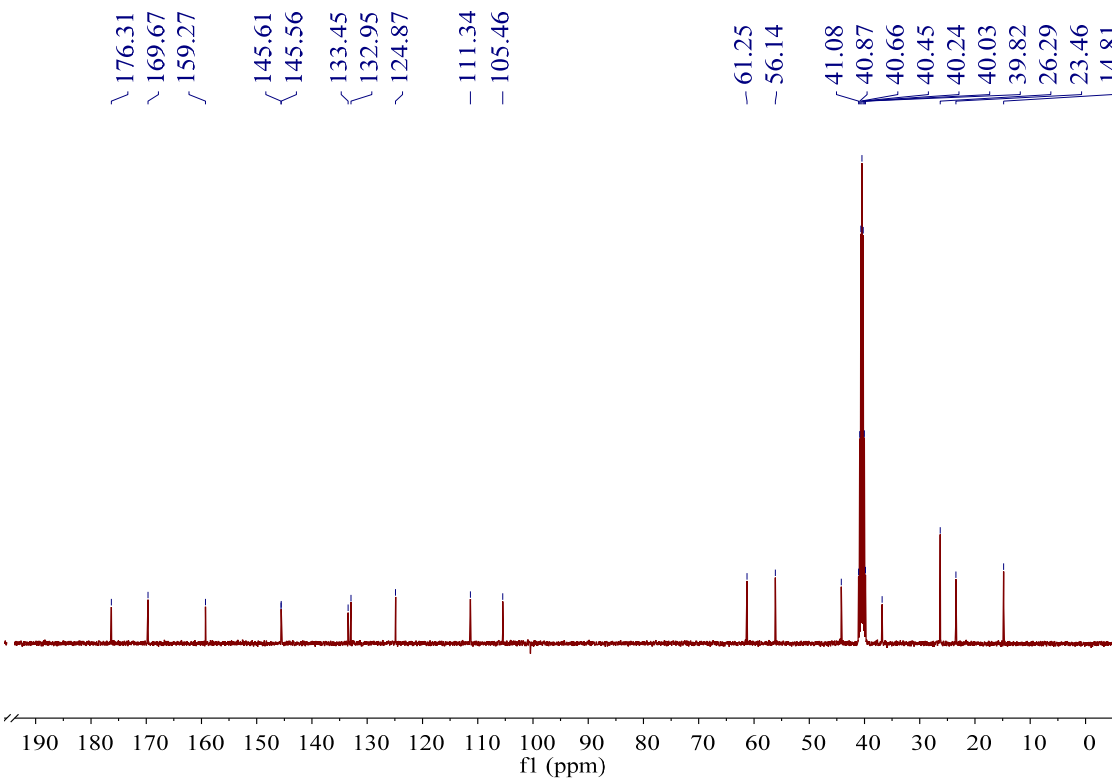
4.05
4.04
3.76
3.41
3.41
3.38
2.54
2.54
2.54
2.04
2.04
2.03
1.48
1.45
1.20
1.19
1.17
0.98
1.01
0.99
1.00
2.17
2.91
2.05
3.00
5.99
3.29
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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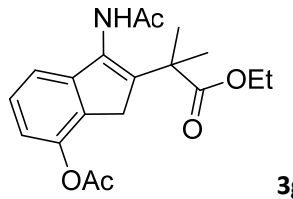
132.95
124.87
111.34
105.46
61.25
56.14
41.08
40.87
40.66
40.45
40.24
40.03
39.82
26.29
23.46
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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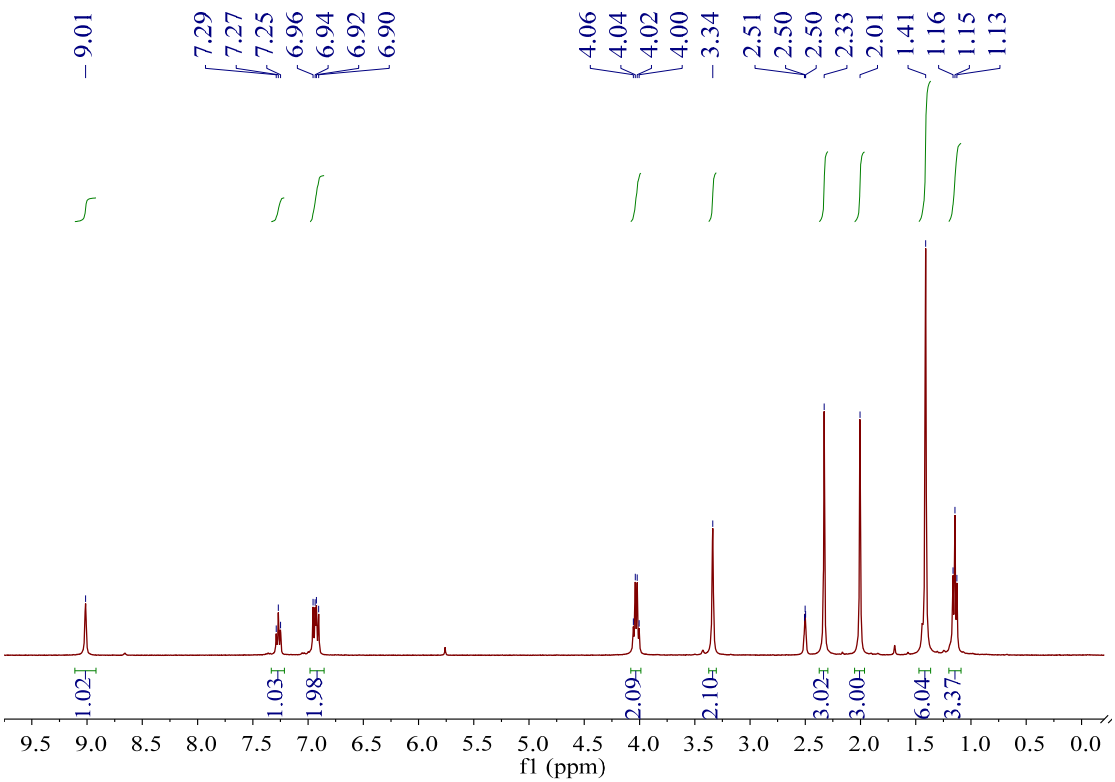
OEt
O
OAc

3g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7.25
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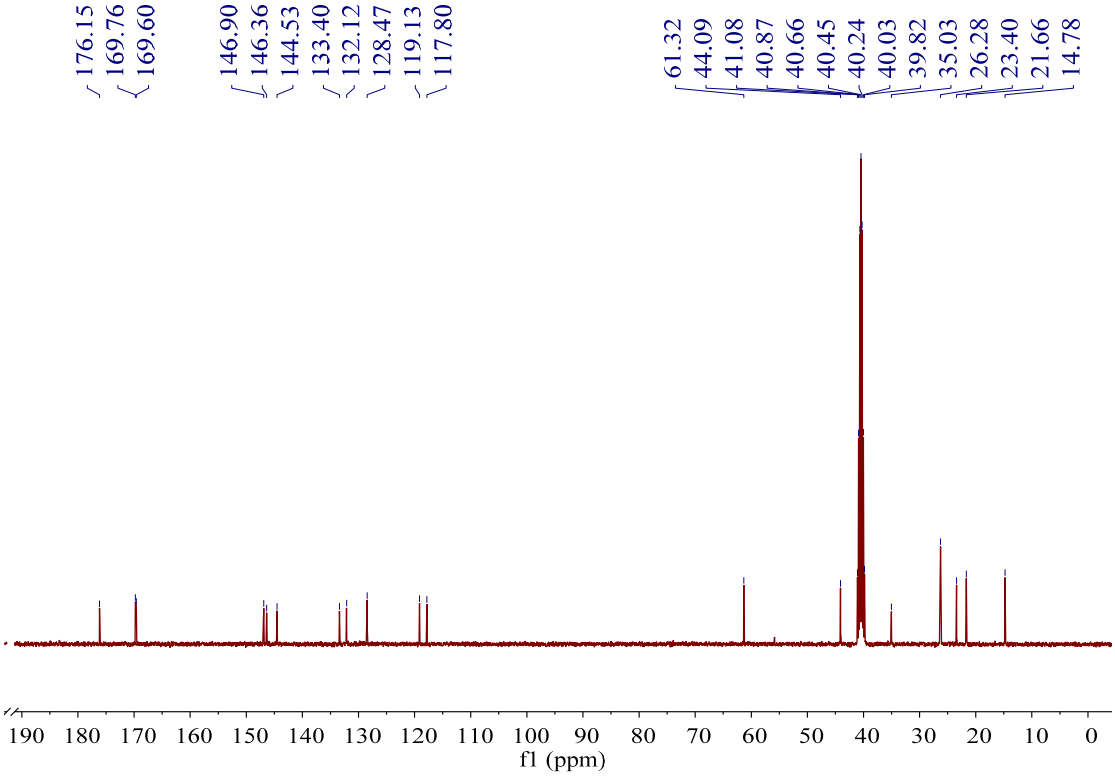
6.94
6.92
6.90
4.06
4.04
4.02
4.00
3.34
2.51
2.50
2.50
2.33
2.01
1.41
1.16
1.15
1.13
1.02
1.03
1.98
2.09
2.10
3.02
3.00
6.04
3.37
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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144.53
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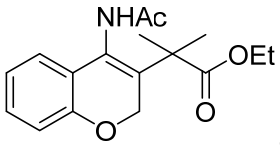
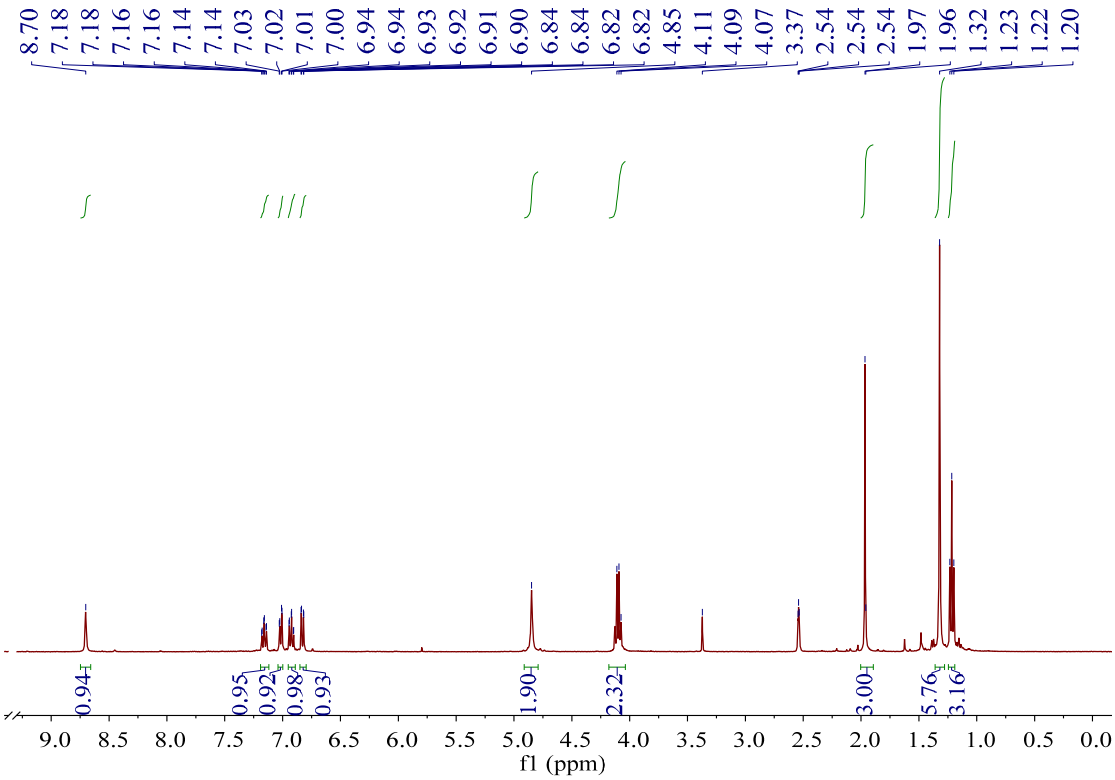
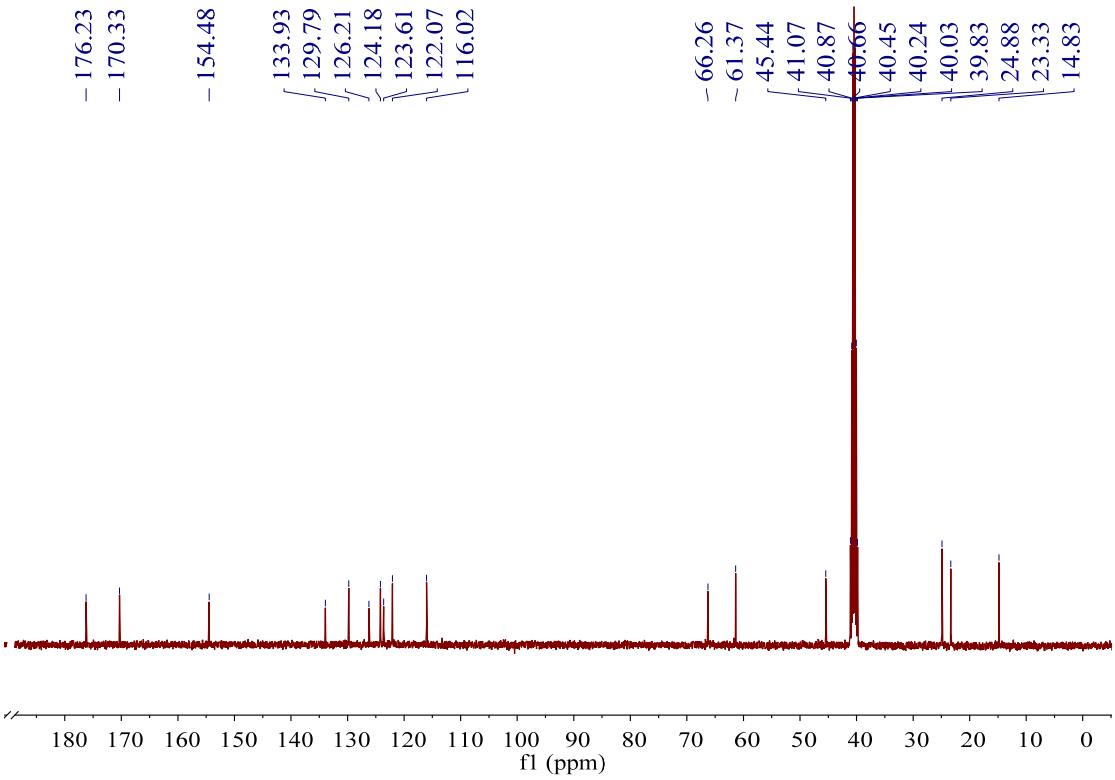
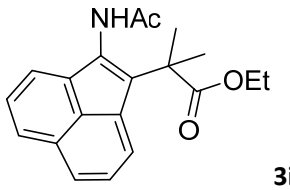
132.12
128.47
119.13
117.80
61.32
44.09
41.08
40.87
40.66
40.45
40.24
40.03
39.82
35.03
26.28
23.40
21.66
14.78
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O
O

3h


8.70 7.18 7.18 7.16 7.16 7.14 7.14 7.03 7.02 7.01 7.00 6.94 6.94 6.93 6.92 6.91 6.90 6.84 6.84 6.82 6.82 4.85 4.11 4.09 4.07 3.37 2.54 2.54 2.54 1.97 1.96 1.32 1.23 1.22 1.20
0.94 0.95 0.92 0.98 0.93 1.90 2.32 3.00 5.76 3.16
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
f1 (ppm)


176.23 170.33 154.48 133.93 129.79 126.21 124.18 123.61 122.07 116.02 66.26 61.37 45.44 41.07 40.87 40.66 40.45 40.24 40.03 39.83 24.88 23.33 14.83
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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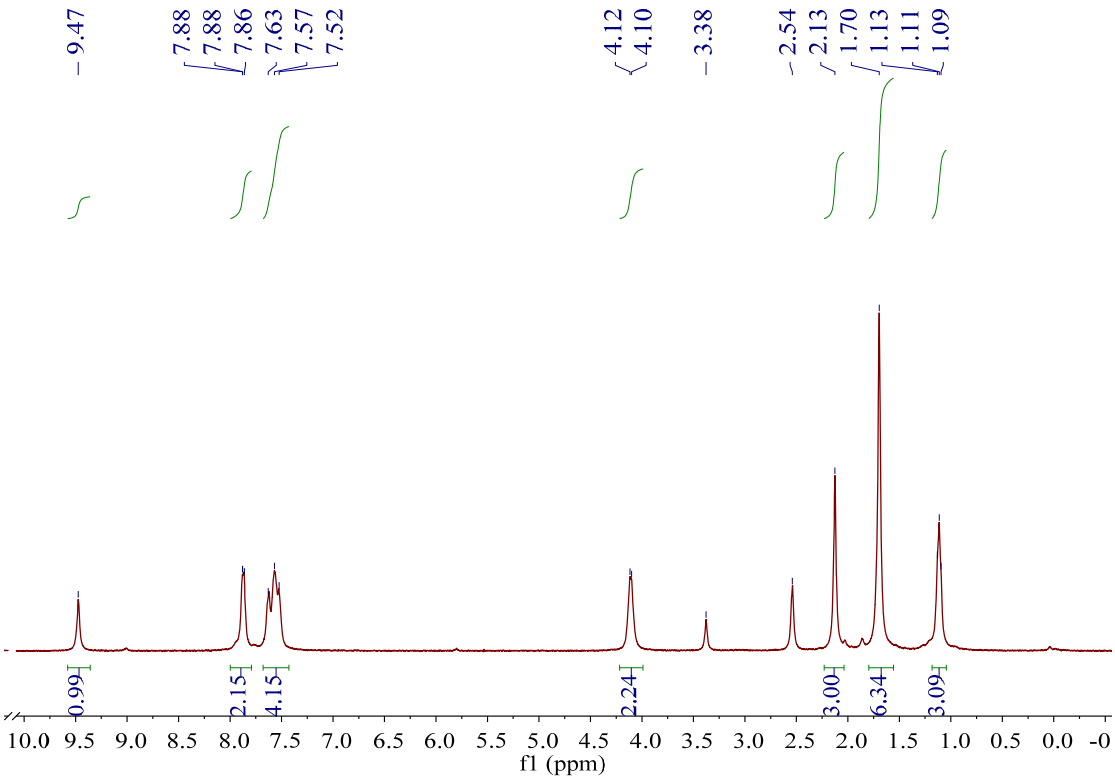
OEt
O

3i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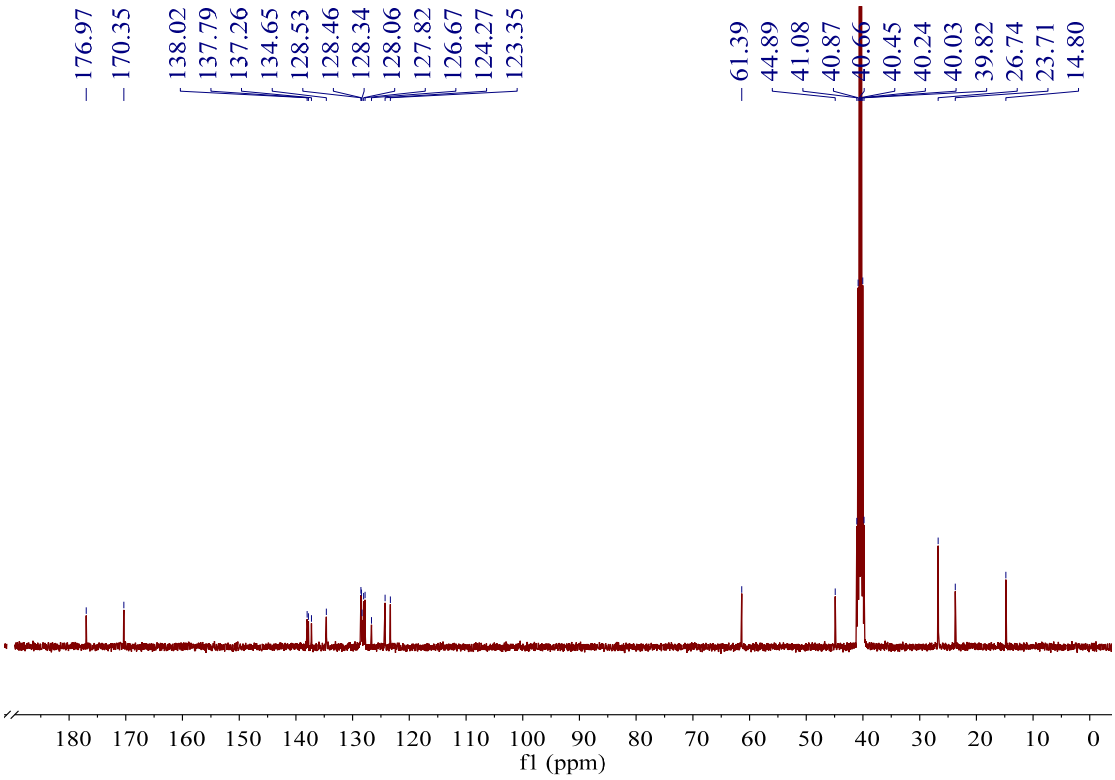
7.57
7.52
4.12
4.10
3.38
2.54
2.13
1.70
1.13
1.11
1.09
0.99
2.15
4.15
2.24
3.00
6.34
3.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)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128.46
128.34
128.06
127.82
126.67
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123.35
61.39
44.89
41.08
40.87
40.66
40.45
40.24
40.03
39.82
26.74
23.71
14.80
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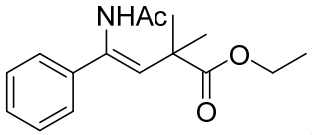
f1 (ppm)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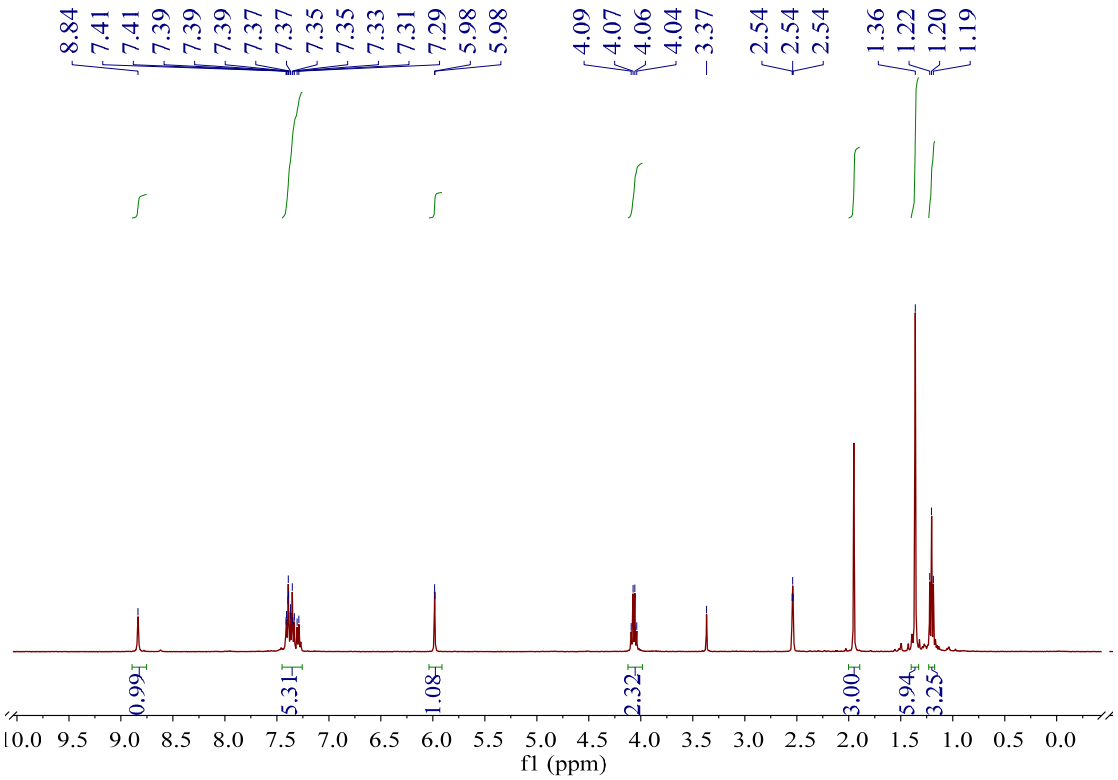
O
O

3j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7.35
7.33
7.31
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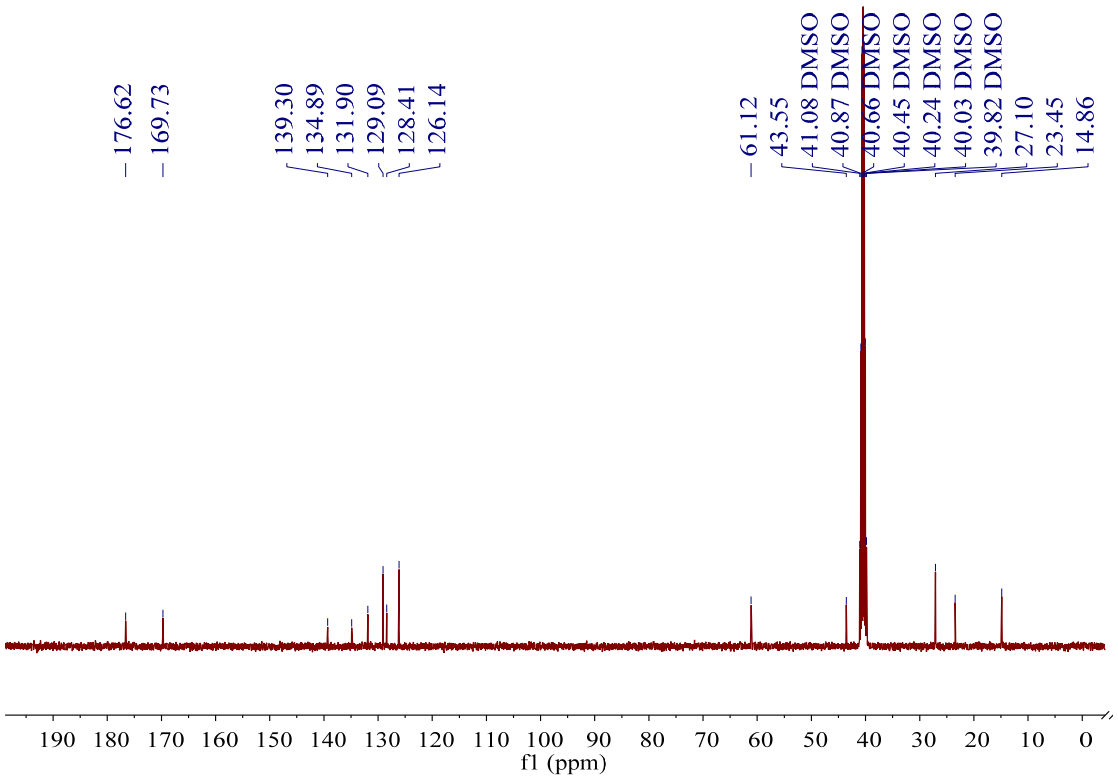
5.98
4.09
4.07
4.06
4.04
3.37
2.54
2.54
2.54
1.36
1.22
1.20
1.19
0.99
5.31
1.08
2.32
3.00
5.94
3.25
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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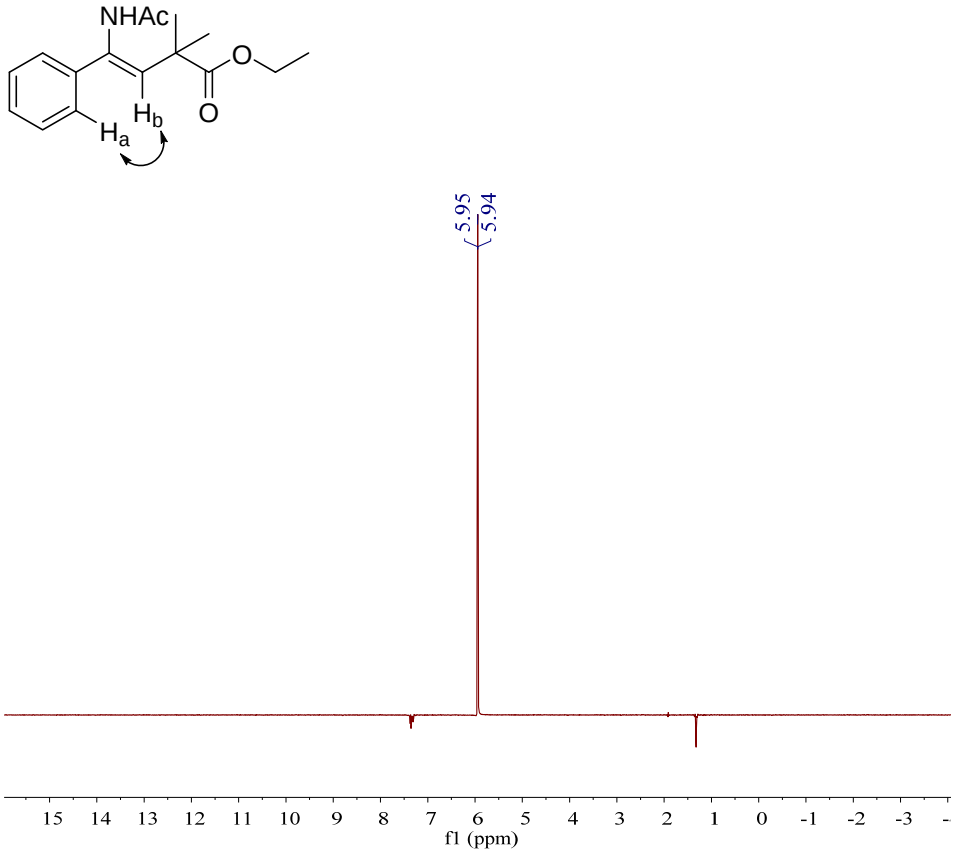
131.90
129.09
128.41
126.14
61.12
43.55
41.08 DMSO
40.87 DMSO
40.66 DMSO
40.45 DMSO
40.24 DMSO
40.03 DMSO
39.82 DMSO
27.10
23.45
14.86
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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O
O
Hb
Ha
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5.94
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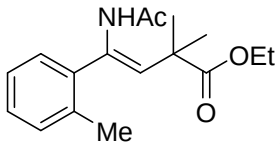
f1 (ppm)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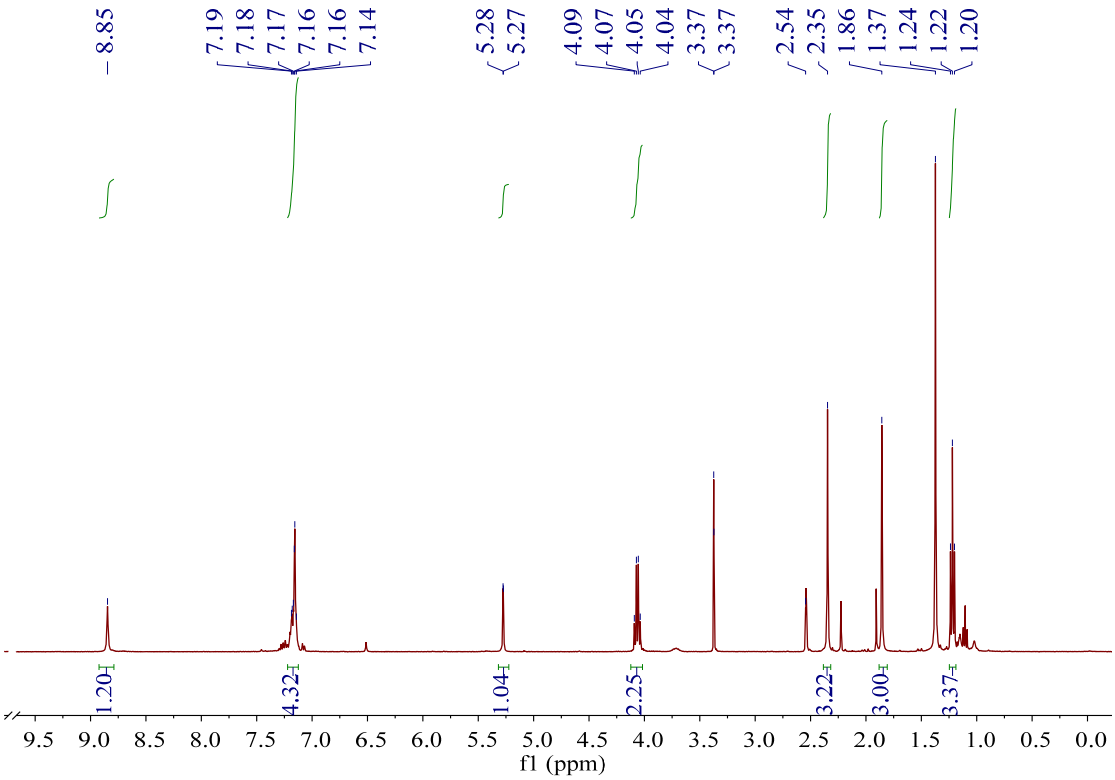
OEt
O
Me

3k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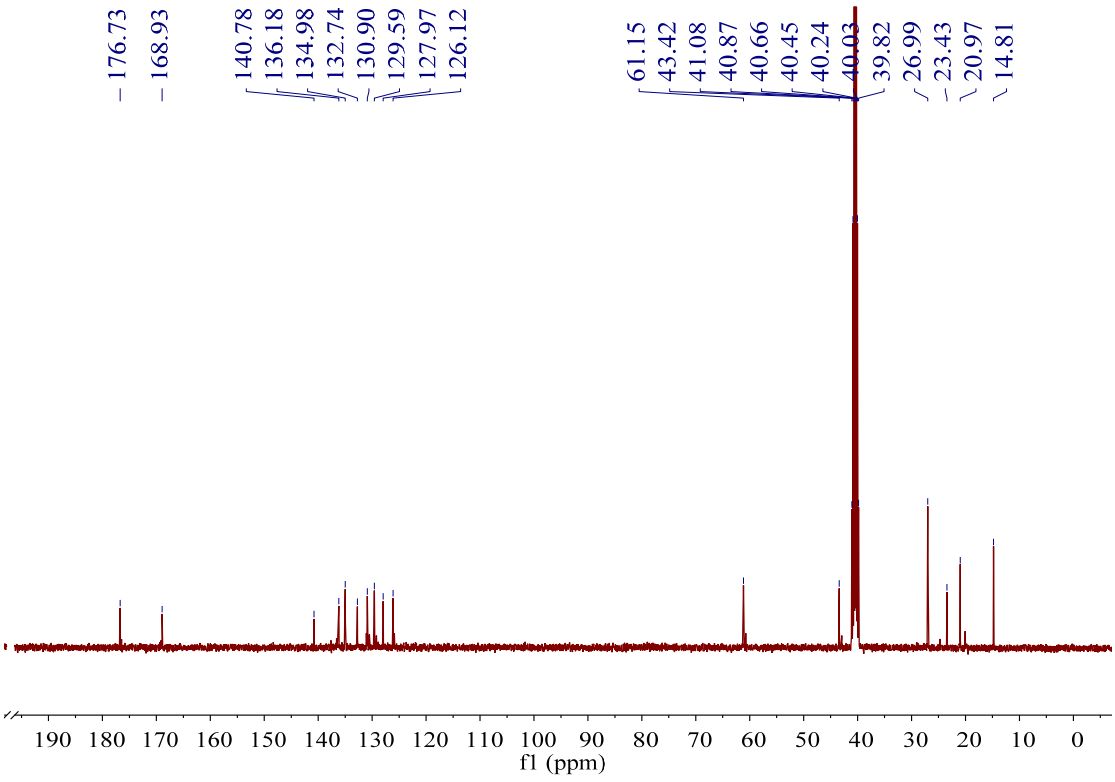
4.09
4.07
4.05
4.04
3.37
3.37
2.54
2.35
1.86
1.37
1.24
1.22
1.20
1.20
4.32
1.04
2.25
3.22
3.00
3.37
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.59
127.97
126.12
61.15
43.42
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40.87
40.66
40.45
40.24
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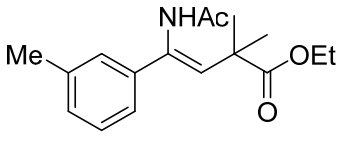
26.99
23.43
20.97
14.81
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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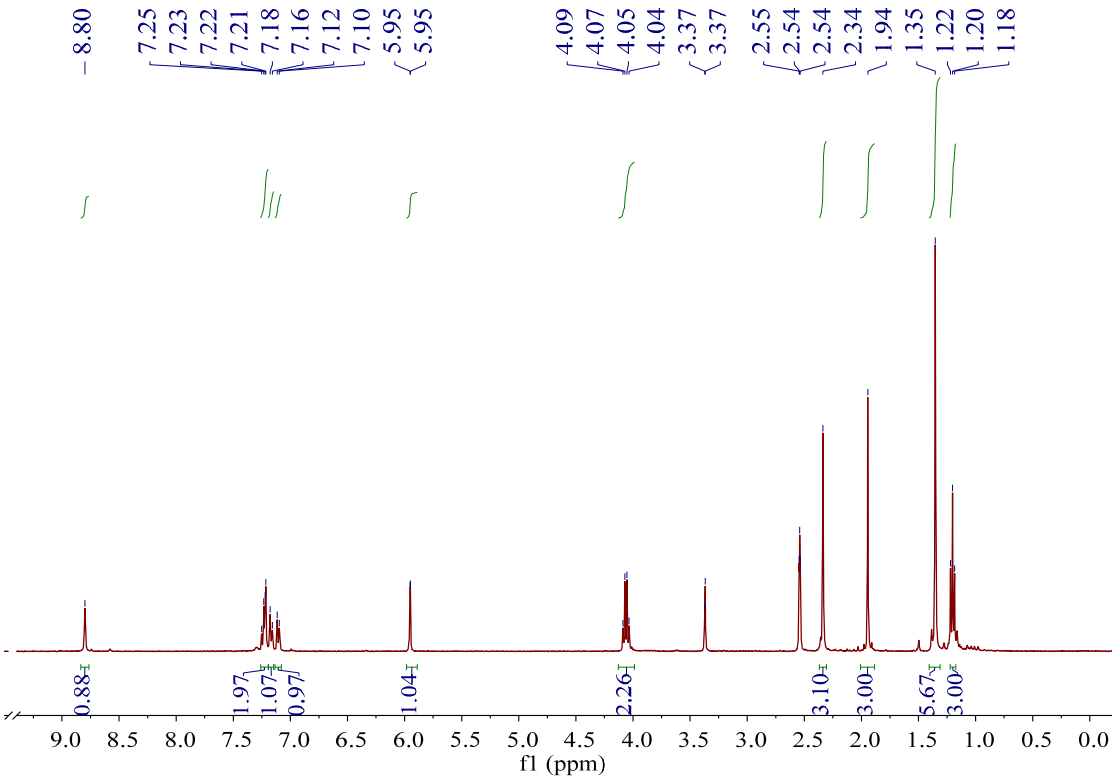
Me
OEt
O

3l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5.95
5.95
4.09
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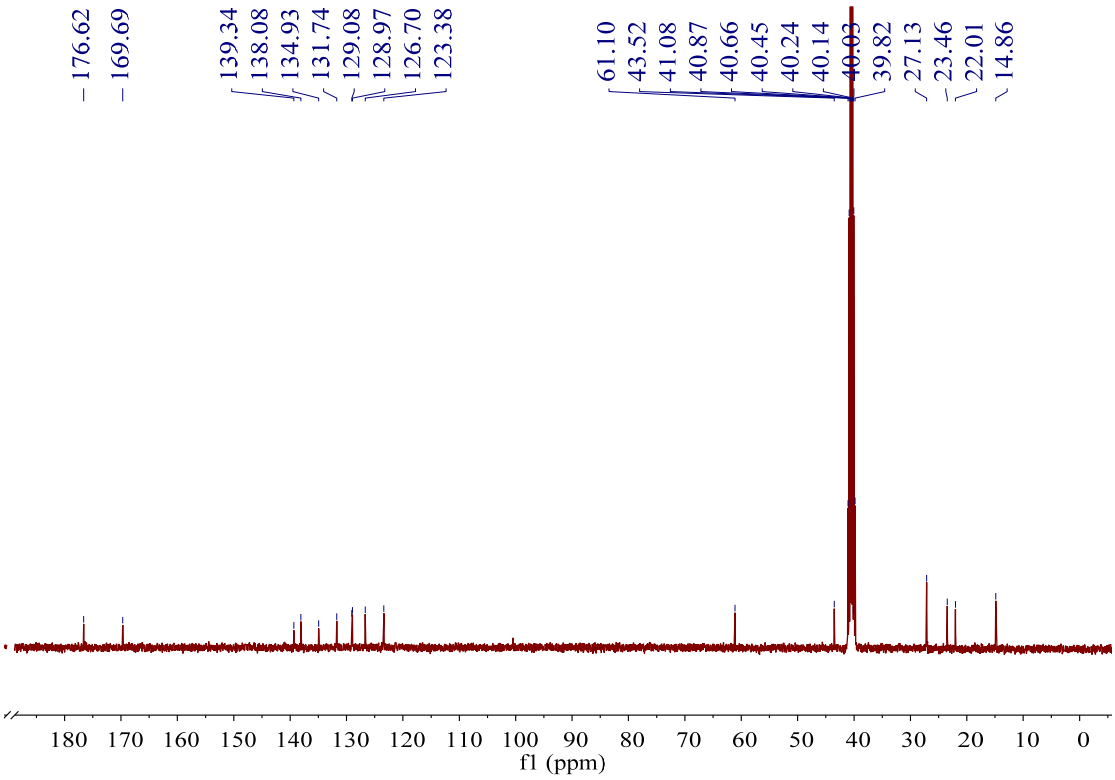
4.04
3.37
3.37
2.55
2.54
2.54
2.34
1.94
1.35
1.22
1.20
1.18
0.88
1.97
1.07
0.97
1.04
2.26
3.10
3.00
5.67
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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123.38
61.10
43.52
41.08
40.87
40.66
40.45
40.24
40.14
40.03
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27.13
23.46
22.01
14.86
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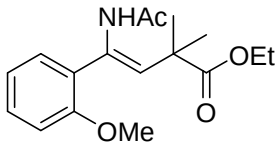
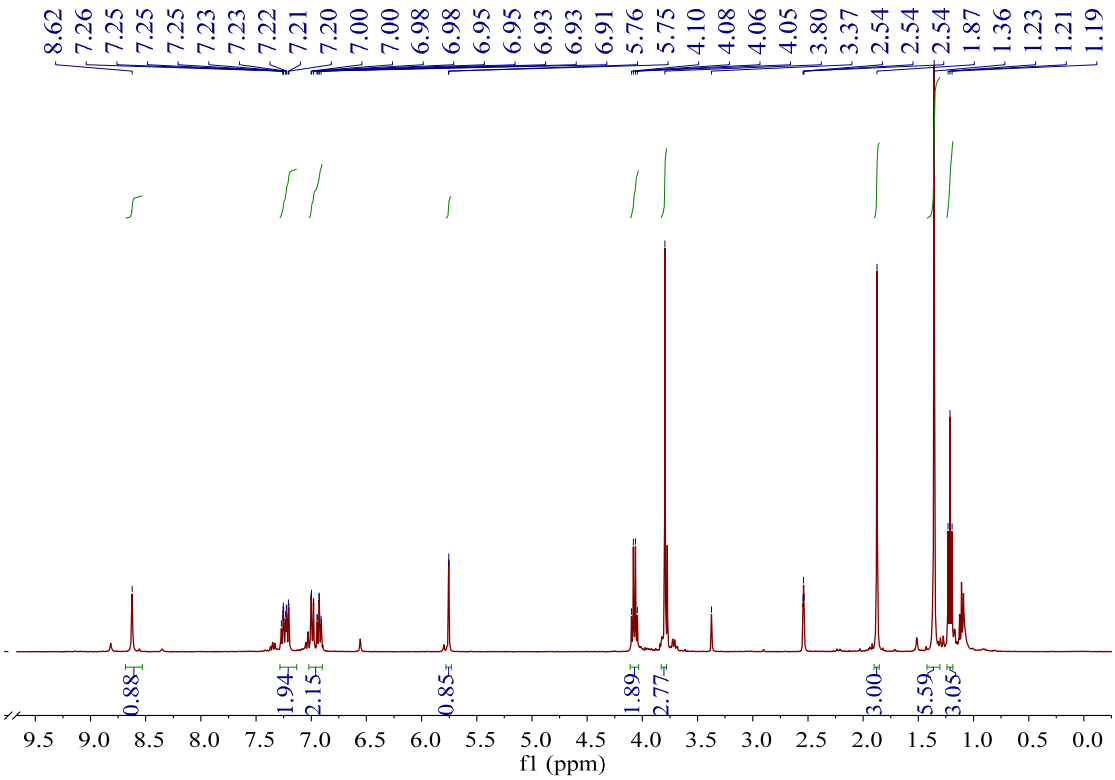
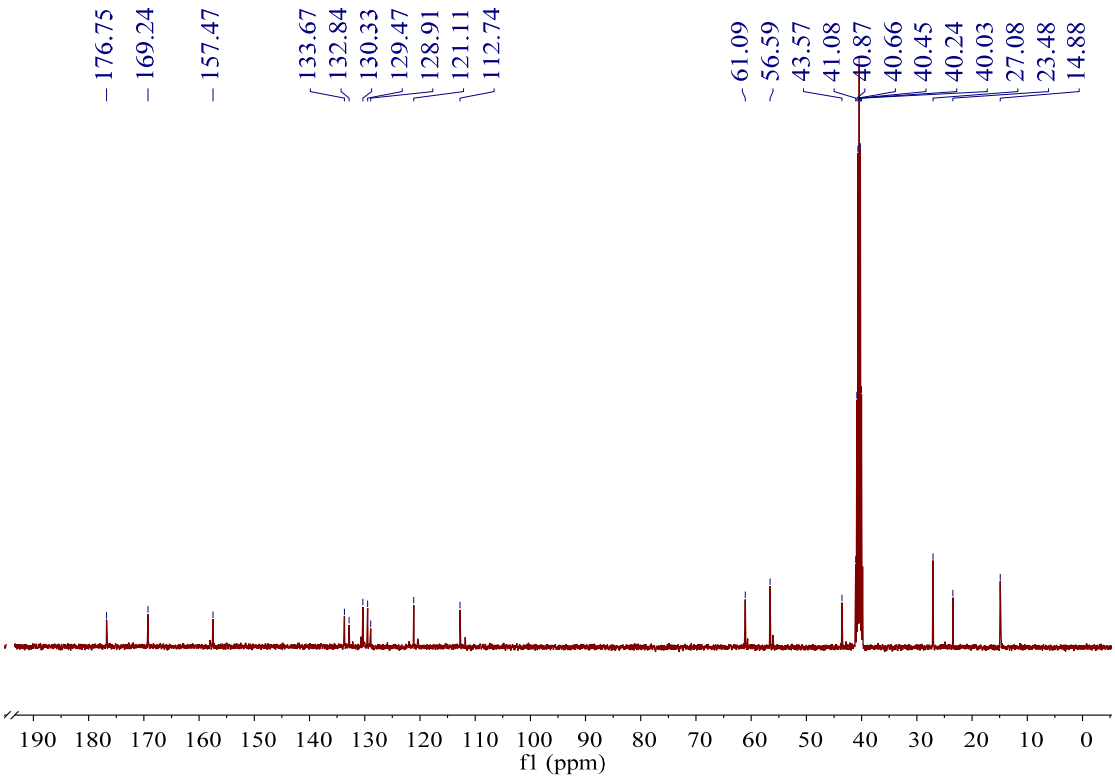
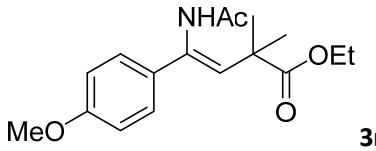
f1 (ppm)


NHAc
OEt
O
OMe

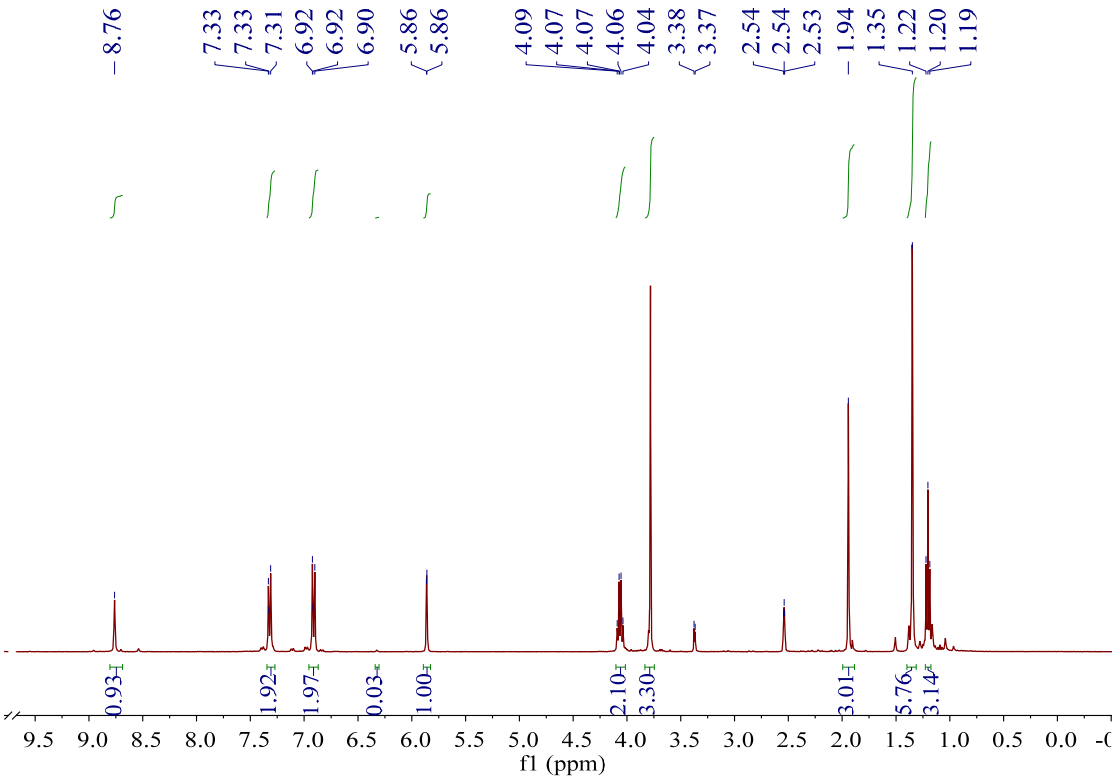
3m

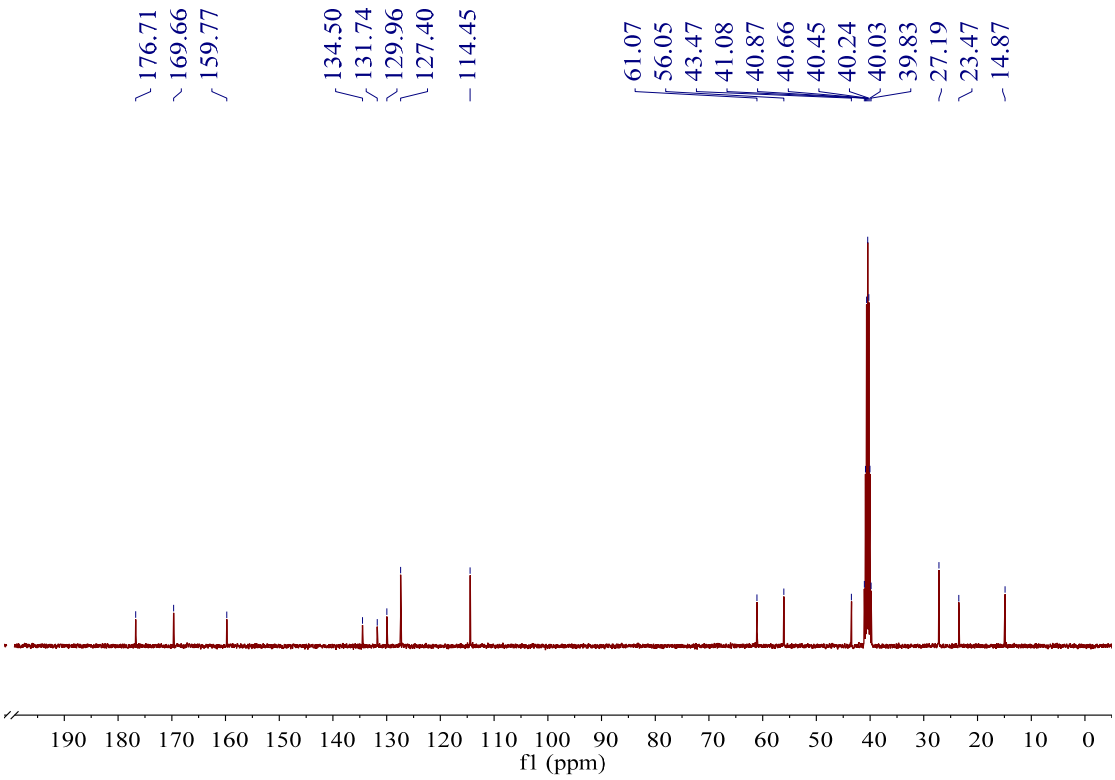

8.62 7.26 7.25 7.25 7.25 7.23 7.23 7.22 7.21 7.20 7.00 7.00 6.98 6.98 6.95 6.95 6.93 6.93 6.91 5.76 5.75 4.10 4.08 4.06 4.05 3.80 3.37 2.54 2.54 2.54 1.87 1.36 1.23 1.21 1.19
0.88 1.94 2.15 0.85 1.89 2.77 3.00 5.59 3.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


176.75 169.24 157.47 133.67 132.84 130.33 129.47 128.91 121.11 112.74 61.09 56.59 43.57 41.08 40.87 40.66 40.45 40.24 40.03 27.08 23.48 14.88
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O
MeO

3n


8.76
7.33
7.33
7.31
6.92
6.92
6.90
5.86
5.86
4.09
4.07
4.07
4.06
4.04
3.38
3.37
2.54
2.54
2.53
1.94
1.35
1.22
1.20
1.19
0.93
1.92
1.97
0.03
1.00
2.10
3.30
3.01
5.76
3.14
f1 (ppm)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169.66
159.77
134.50
131.74
129.96
127.40
114.45
61.07
56.05
43.47
41.08
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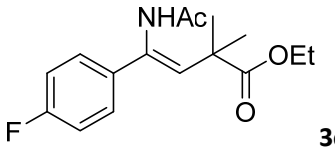
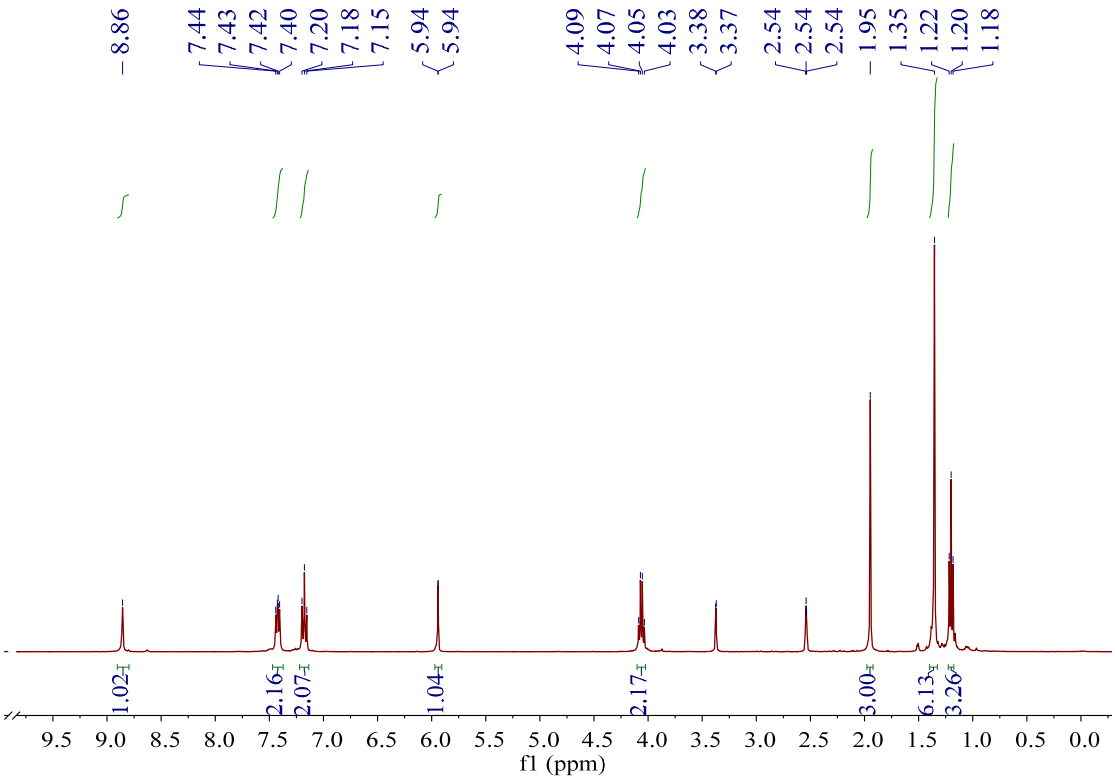
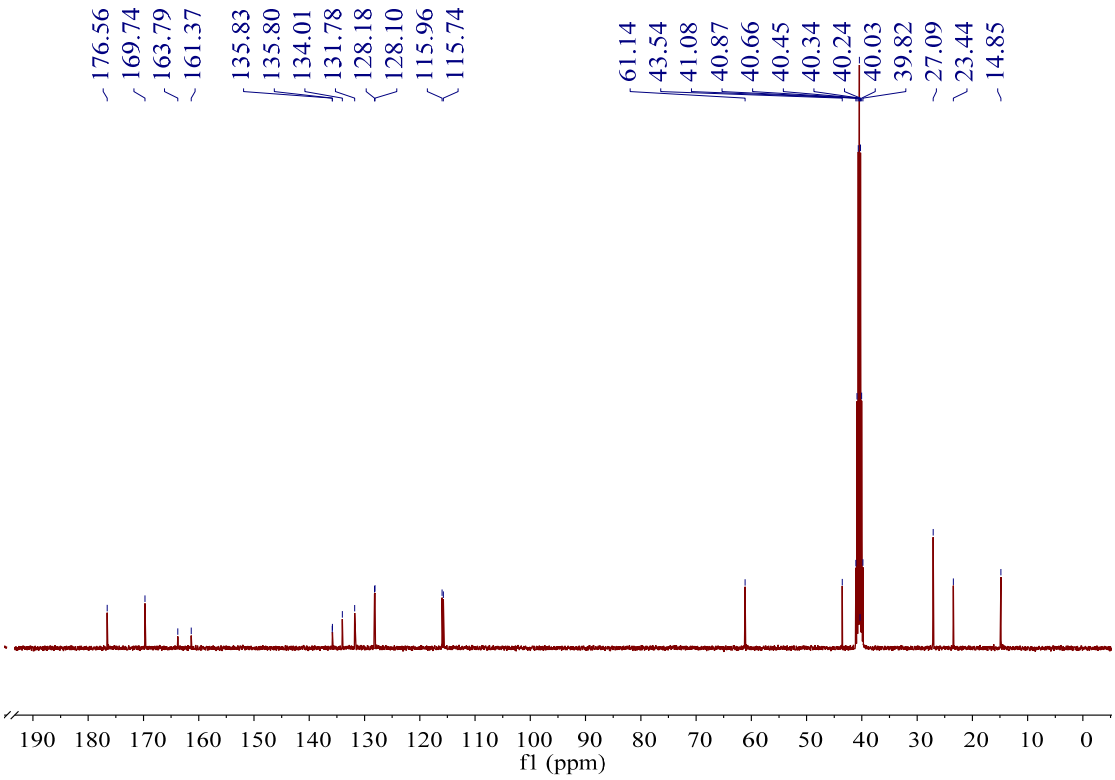
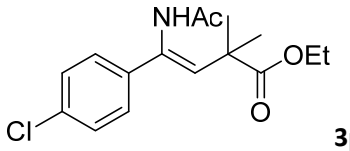
40.03
39.83
27.19
23.47
14.87
f1 (ppm)


NHAc
OEt
O
F

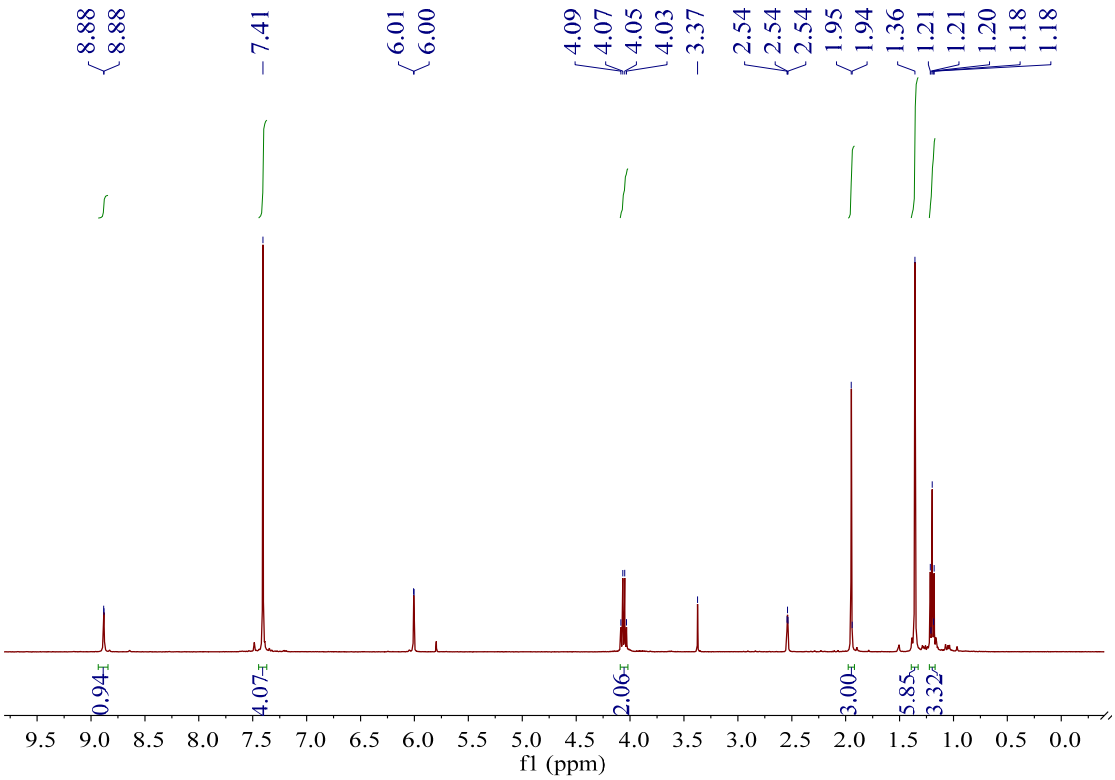
3o

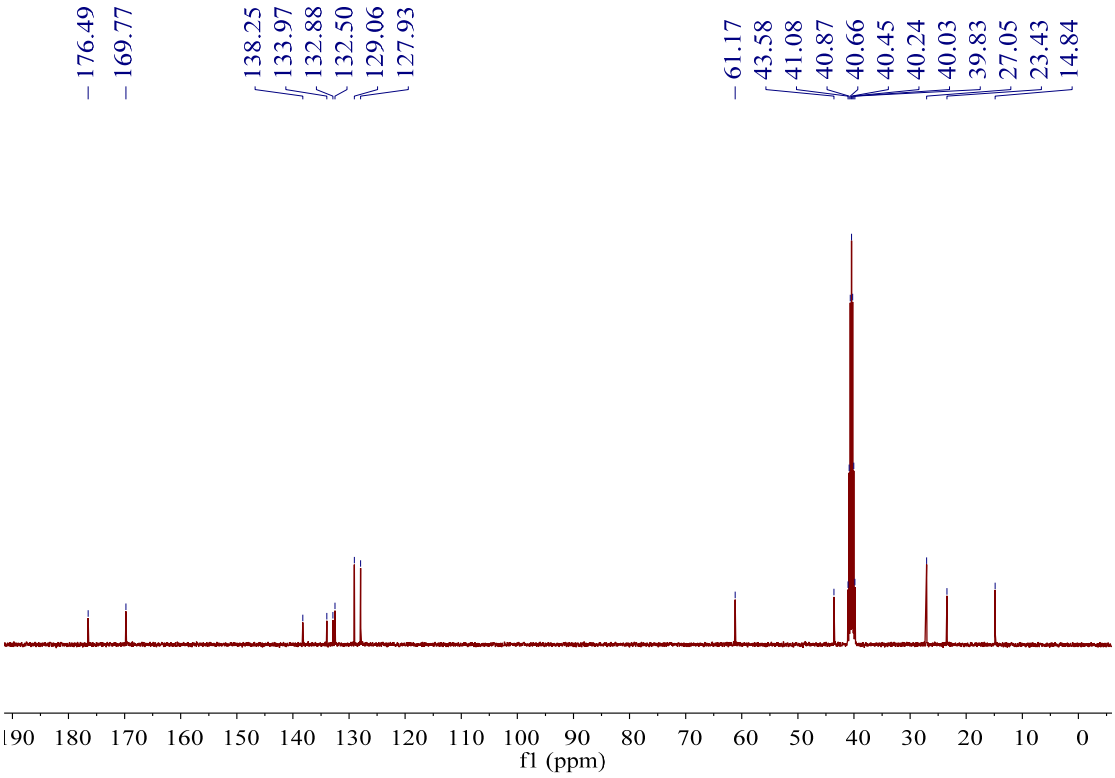

8.86
7.44
7.43
7.42
7.40
7.20
7.18
7.15
5.94
5.94
4.09
4.07
4.05
4.03
3.38
3.37
2.54
2.54
2.54
1.95
1.35
1.22
1.20
1.18
1.02
2.16
2.07
1.04
2.17
3.00
6.13
3.26
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


176.56
169.74
163.79
161.37
135.83
135.80
134.01
131.78
128.18
128.10
115.96
115.74
61.14
43.54
41.08
40.87
40.66
40.45
40.34
40.24
40.03
39.82
27.09
23.44
14.85
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O
Cl

3p


8.88
8.88
7.41
6.01
6.00
4.09
4.07
4.05
4.03
3.37
2.54
2.54
2.54
1.95
1.94
1.36
1.21
1.21
1.20
1.18
1.18
0.94
4.07
2.06
3.00
5.85
3.32
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
f1 (ppm)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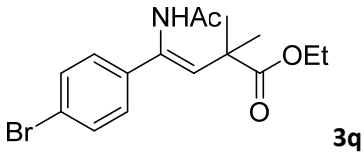
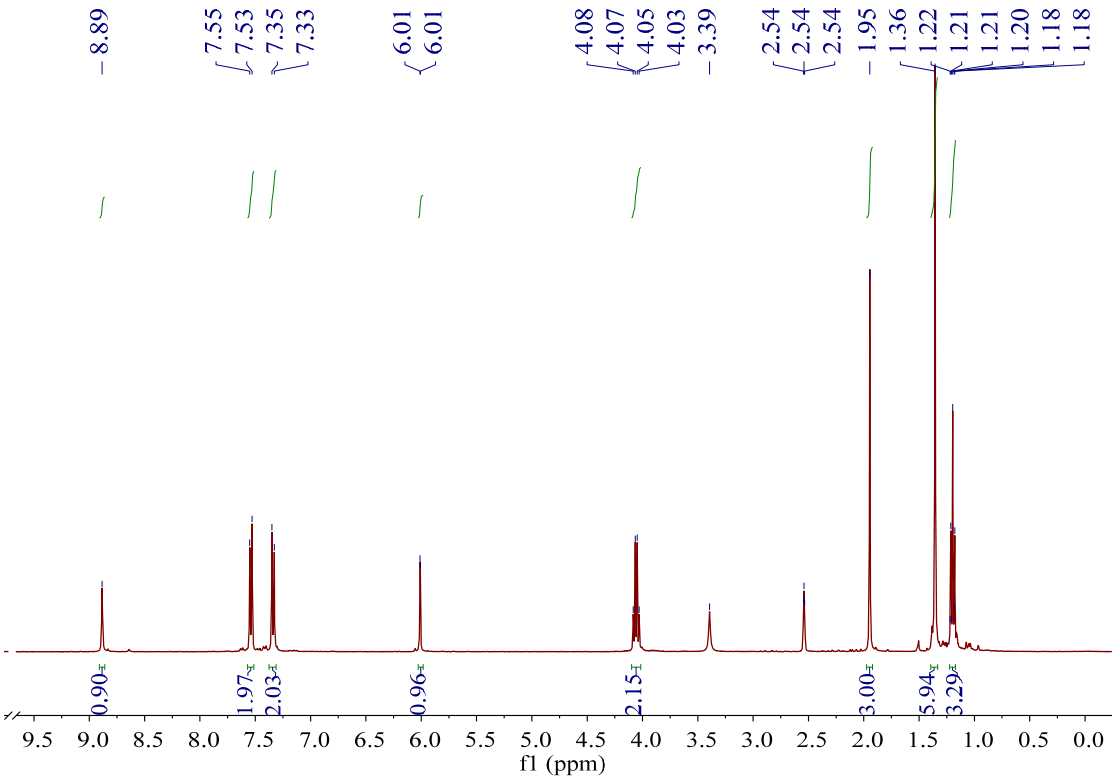
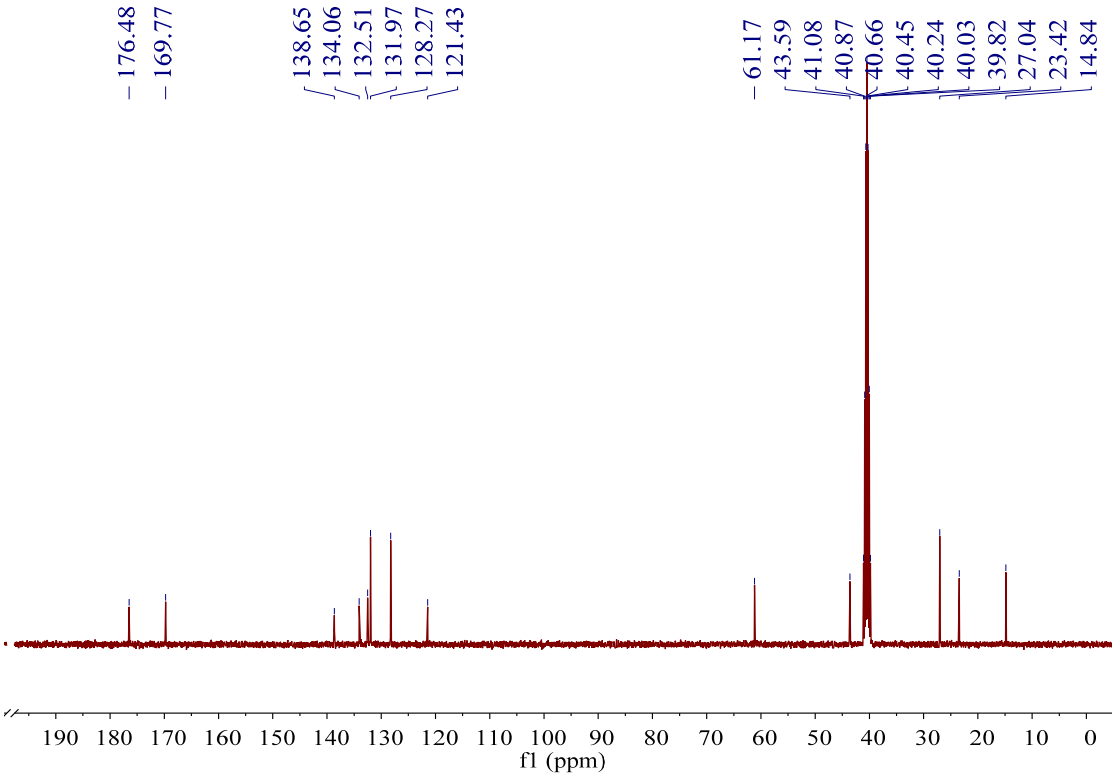
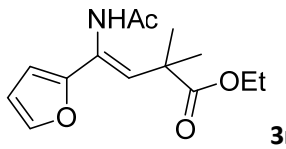
169.77
138.25
133.97
132.88
132.50
129.06
127.93
61.17
43.58
41.08
40.87
40.66
40.45
40.24
40.03
39.83
27.05
23.43
14.84
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O
Br

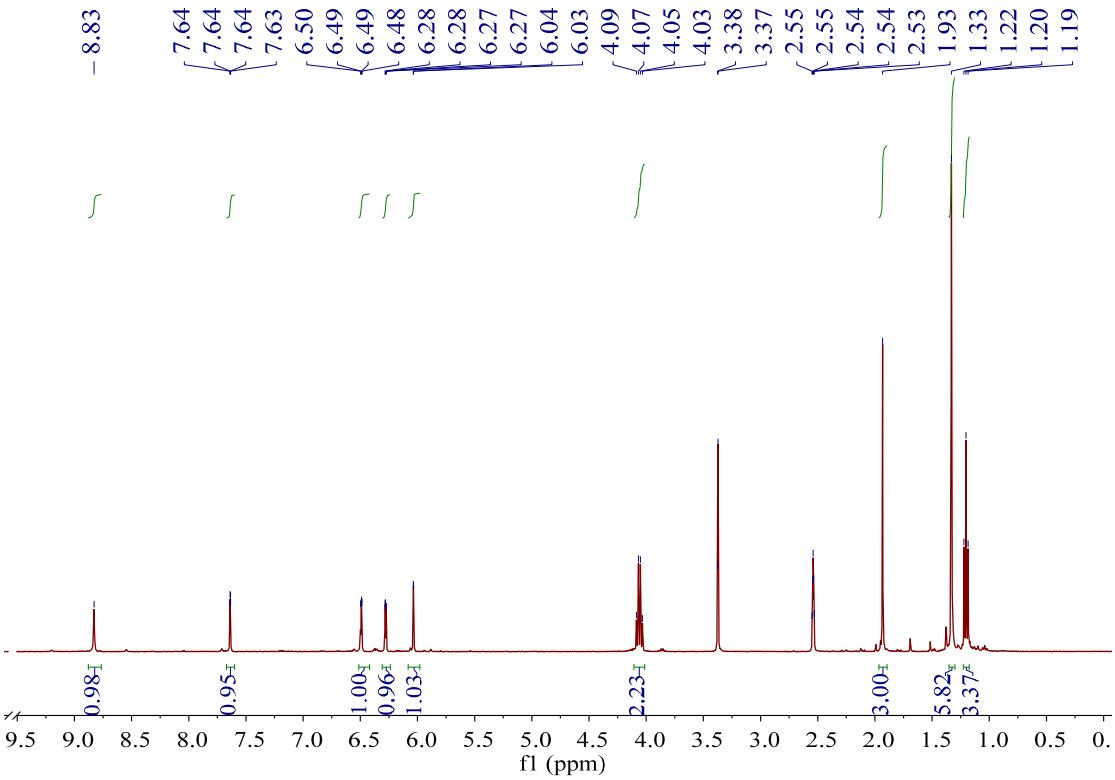
3q

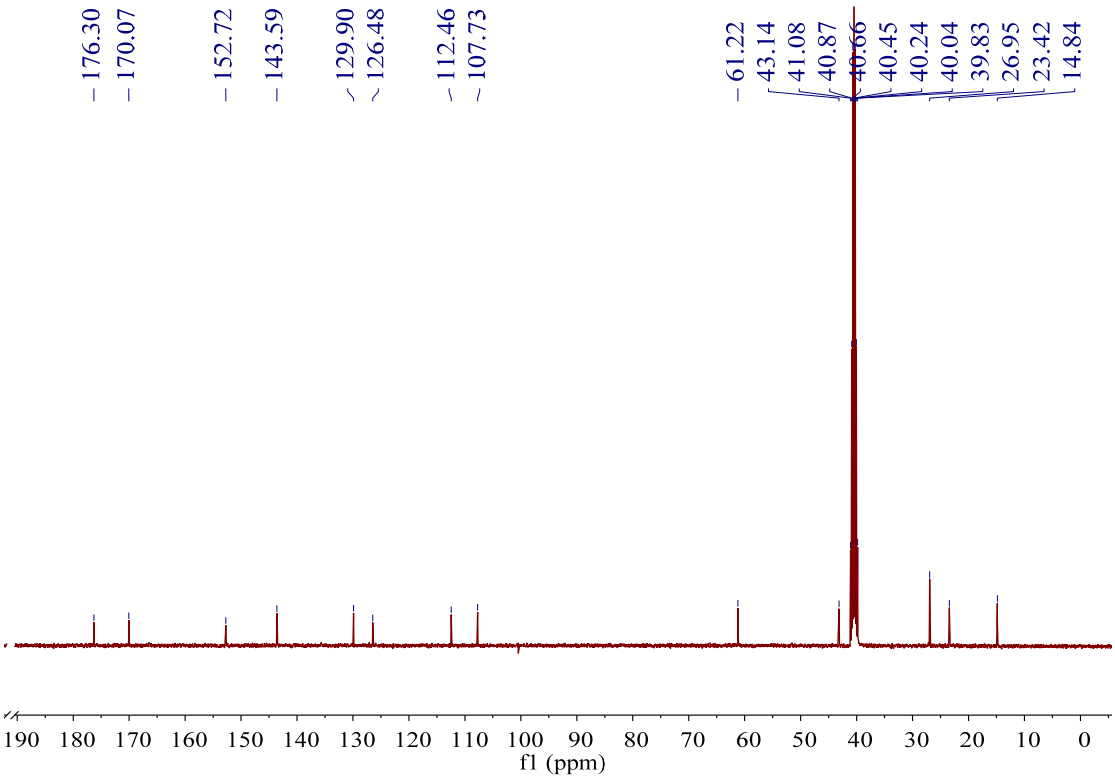

8.89
7.55
7.53
7.35
7.33
6.01
6.01
4.08
4.07
4.05
4.03
3.39
2.54
2.54
2.54
1.95
1.36
1.22
1.21
1.21
1.20
1.18
1.18
0.90
1.97
2.03
0.96
2.15
3.00
5.94
3.29
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

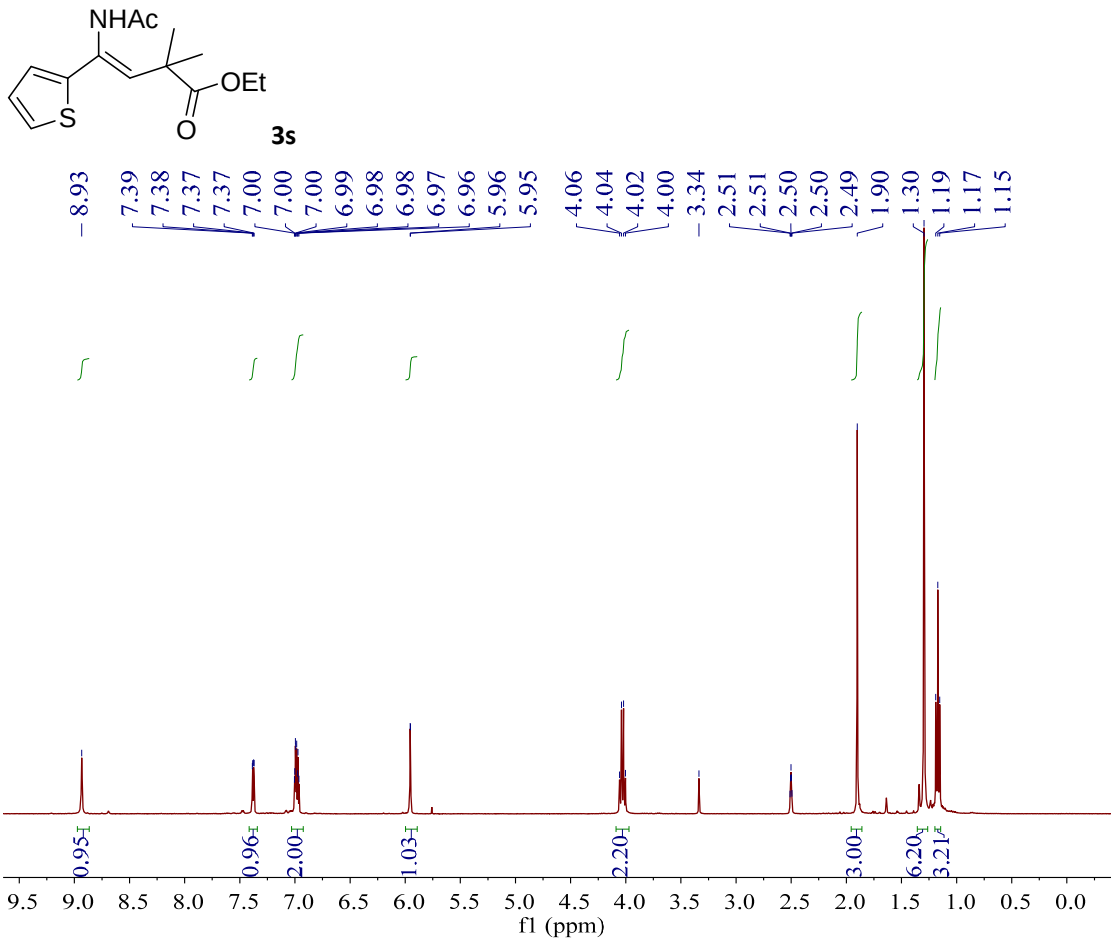

176.48
169.77
138.65
134.06
132.51
131.97
128.27
121.43
61.17
43.59
41.08
40.87
40.66
40.45
40.24
40.03
39.82
27.04
23.42
14.84
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

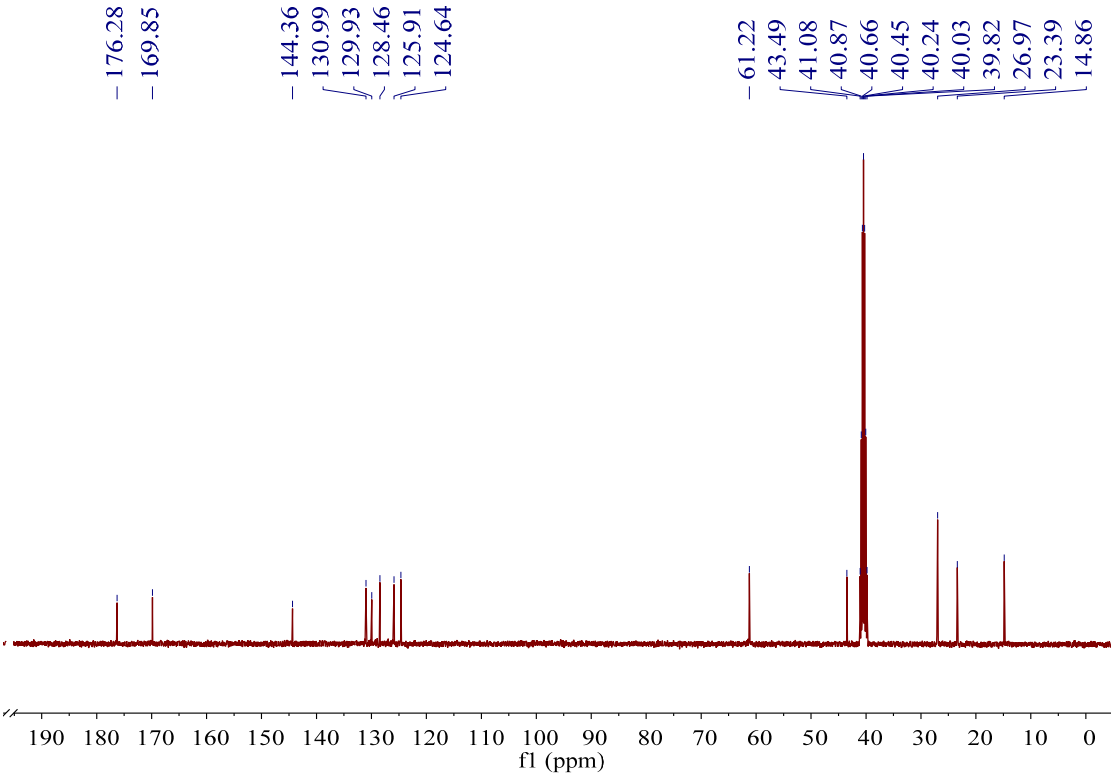

NHAc
OEt
O
O

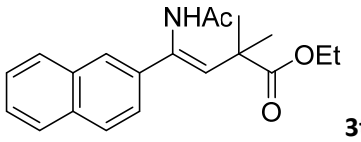
3r


8.83
7.64
7.64
7.64
7.63
6.50
6.49
6.49
6.48
6.28
6.28
6.27
6.27
6.04
6.03
4.09
4.07
4.05
4.03
3.38
3.37
2.55
2.55
2.54
2.54
2.53
1.93
1.33
1.22
1.20
1.19
0.98
0.95
1.00
0.96
1.03
2.23
3.00
5.82
3.37
f1 (ppm)

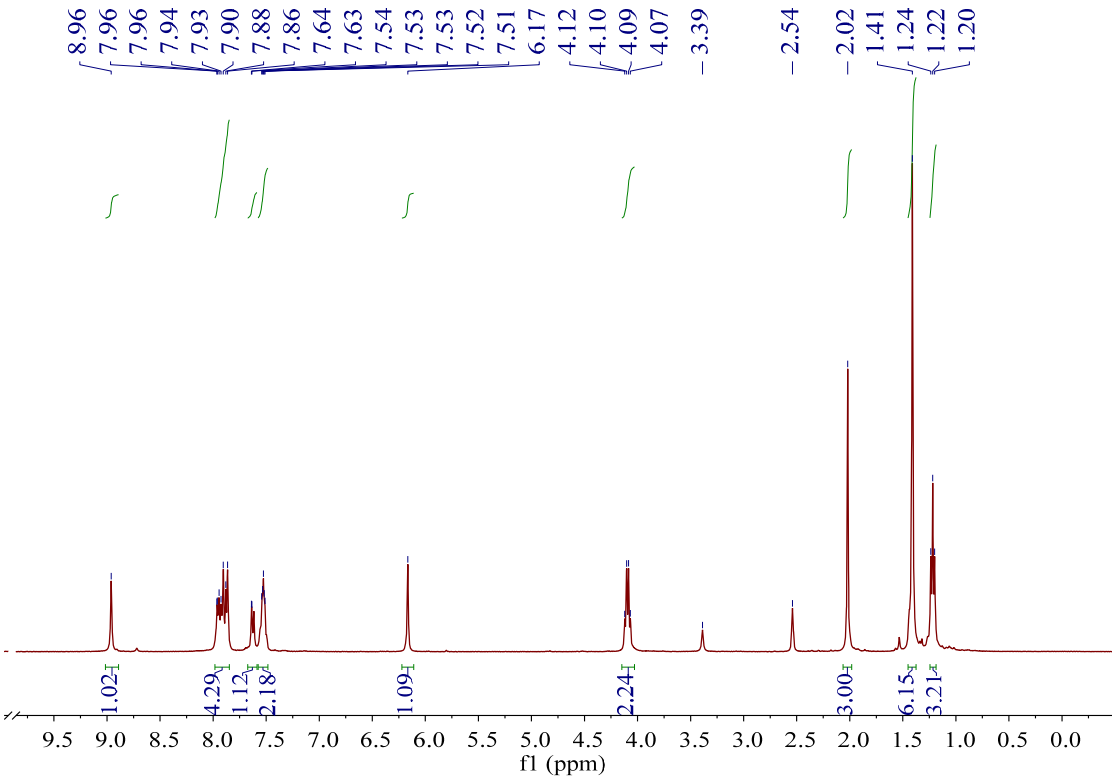

176.30
170.07
152.72
143.59
129.90
126.48
112.46
107.73
61.22
43.14
41.08
40.87
40.66
40.45
40.24
40.04
39.83
26.95
23.42
14.84
f1 (ppm)

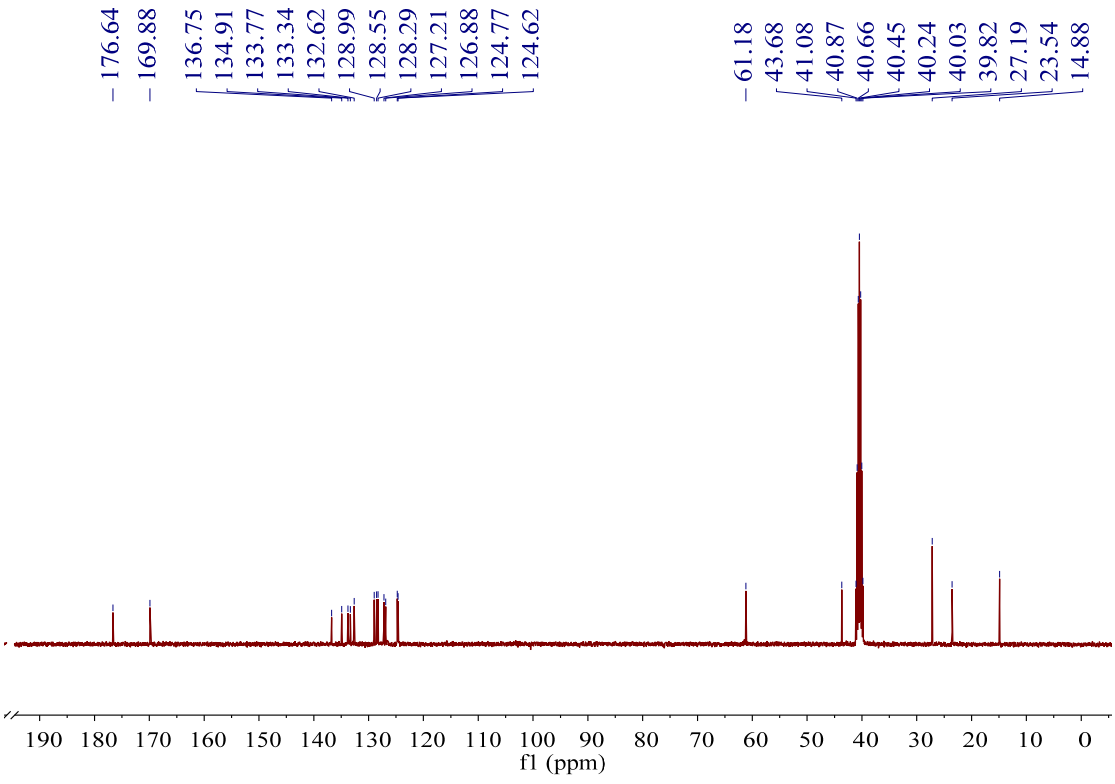

NHAc
OEt
S
O
3s
8.93
7.39
7.38
7.37
7.37
7.00
7.00
7.00
6.99
6.98
6.98
6.97
6.96
5.96
5.95
4.06
4.04
4.02
4.00
3.34
2.51
2.51
2.50
2.50
2.49
1.90
1.30
1.19
1.17
1.15
0.95
0.96
2.00
1.03
2.20
3.00
6.20
3.21
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


176.28
169.85
144.36
130.99
129.93
128.46
125.91
124.64
61.22
43.49
41.08
40.87
40.66
40.45
40.24
40.03
39.82
26.97
23.39
14.86
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O

3t


8.96
7.96
7.96
7.94
7.93
7.90
7.88
7.86
7.64
7.63
7.54
7.53
7.53
7.52
7.51
6.17
4.12
4.10
4.09
4.07
3.39
2.54
2.02
1.41
1.24
1.22
1.20
1.02
4.29
1.12
2.18
1.09
2.24
3.00
6.15
3.21
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
f1 (ppm)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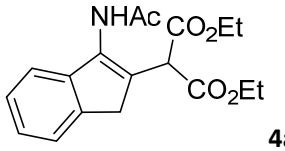
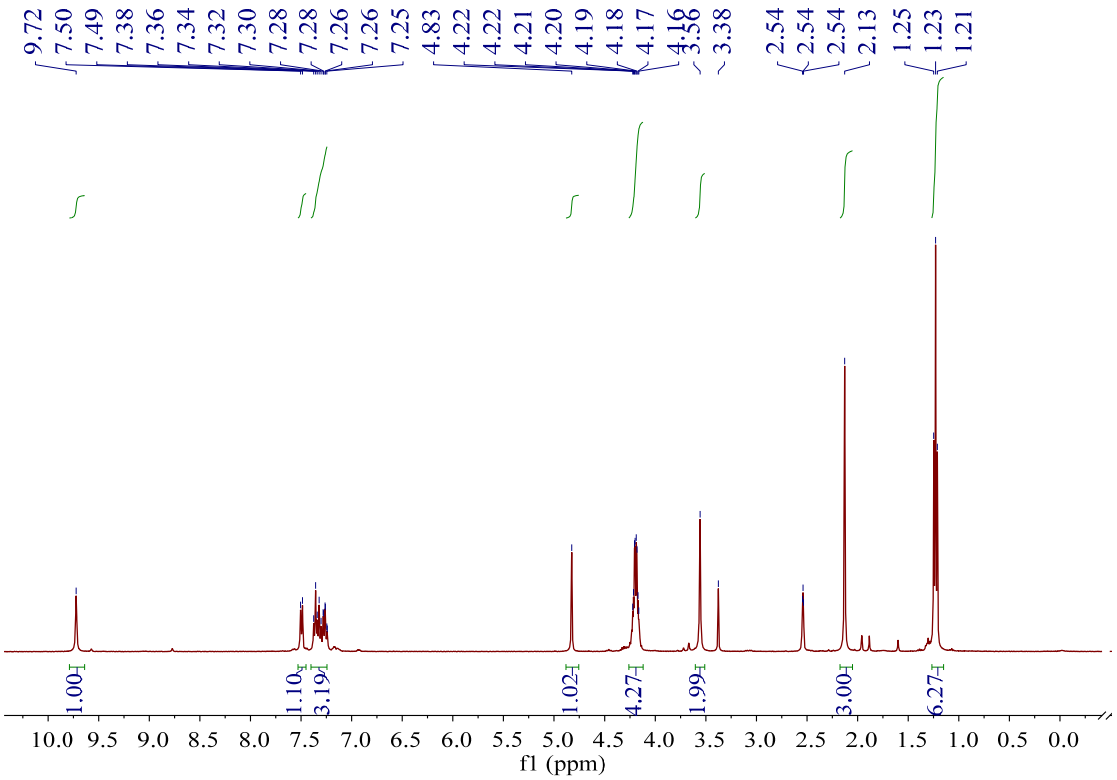
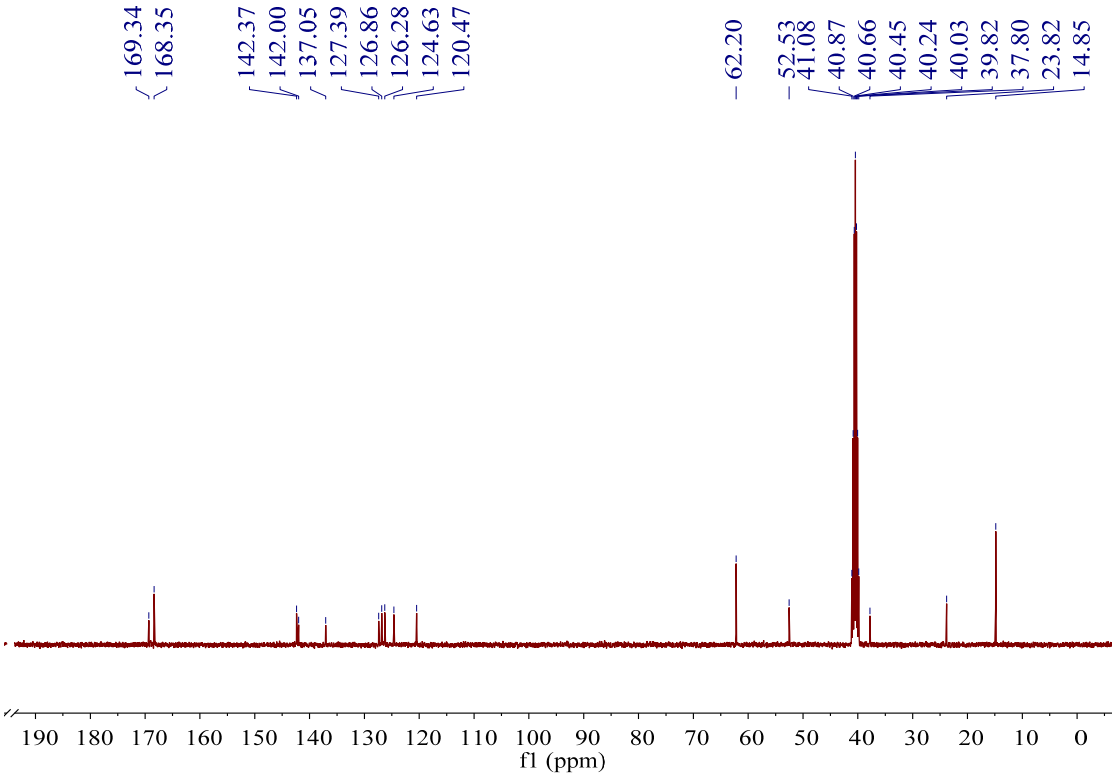
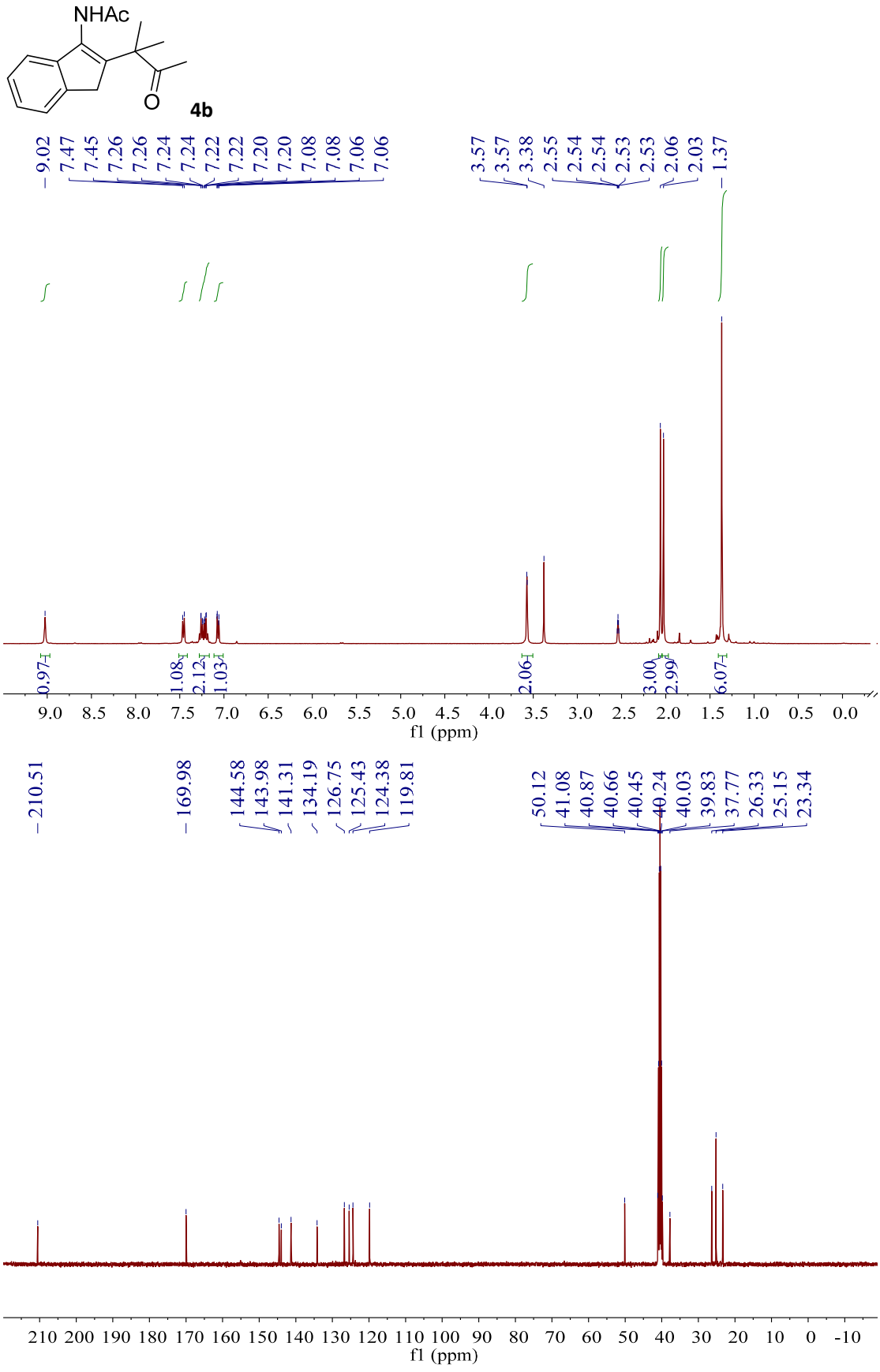
136.75
134.91
133.77
133.34
132.62
128.99
128.55
128.29
127.21
126.88
124.77
124.62
61.18
43.68
41.08
40.87
40.66
40.45
40.24
40.03
39.82
27.19
23.54
14.88
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

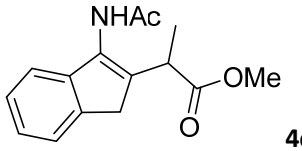

NHAc
CO2Et
CO2Et

4a

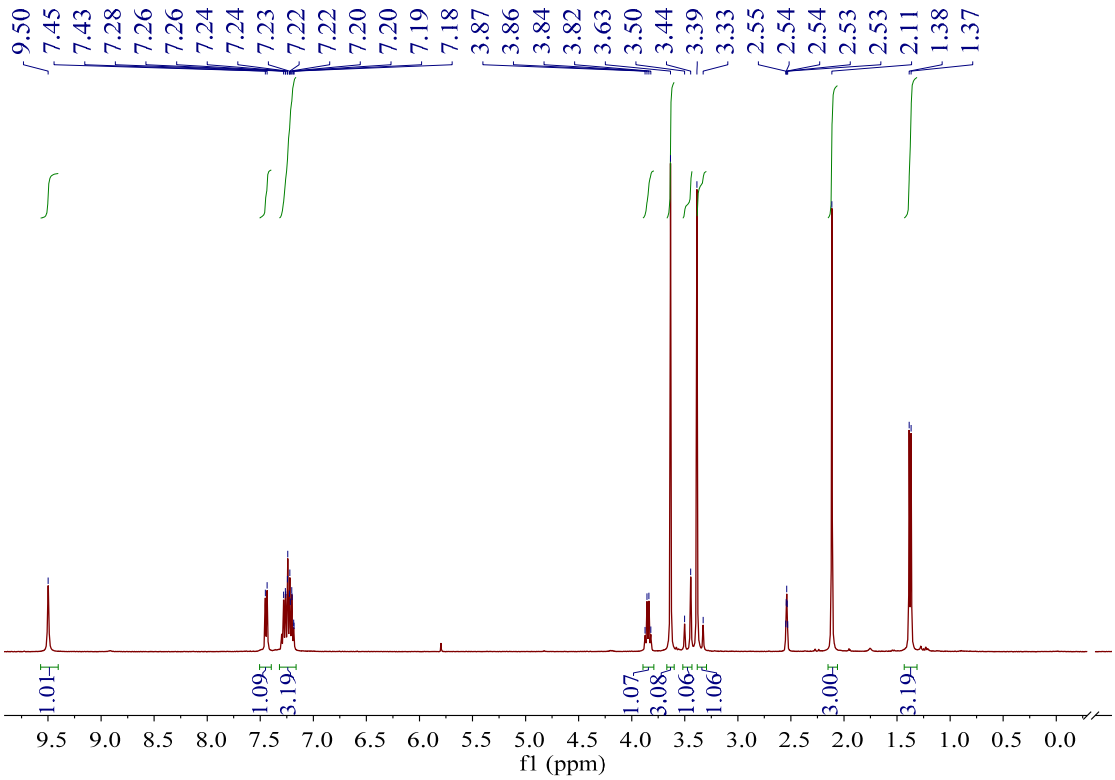

9.72 7.50 7.49 7.38 7.36 7.34 7.32 7.30 7.28 7.28 7.26 7.26 7.25 4.83 4.22 4.22 4.21 4.20 4.19 4.18 4.17 4.16 3.56 3.38 2.54 2.54 2.54 2.13 1.25 1.23 1.21
1.00 1.10 3.19 1.02 4.27 1.99 3.00 6.27
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

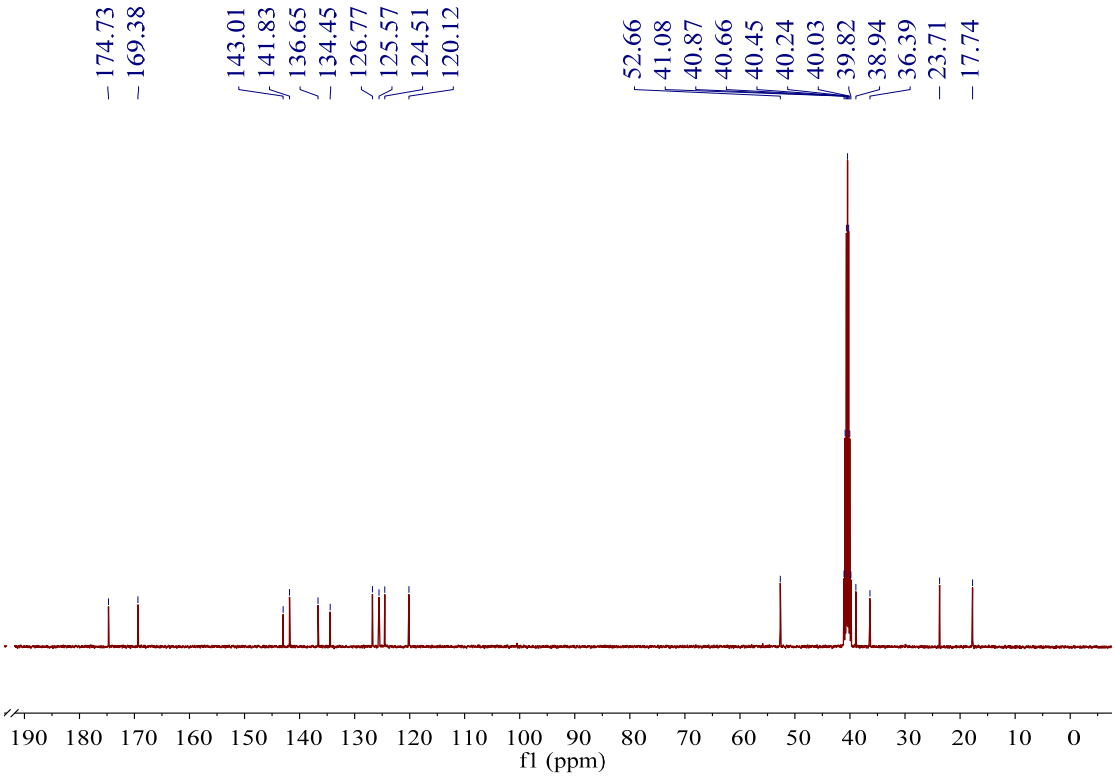

169.34 168.35 142.37 142.00 137.05 127.39 126.86 126.28 124.63 120.47 62.20 52.53 41.08 40.87 40.66 40.45 40.24 40.03 39.82 37.80 23.82 14.85
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

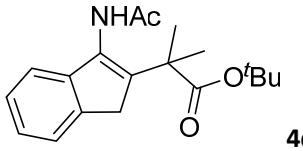

NHAc
O
4b
9.02
7.47
7.45
7.26
7.26
7.24
7.24
7.22
7.22
7.20
7.20
7.08
7.08
7.06
7.06
3.57
3.57
3.38
2.55
2.54
2.54
2.53
2.53
2.06
2.03
1.37
0.97
1.08
2.12
1.03
2.06
3.00
2.99
6.07
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
210.51
169.98
144.58
143.98
141.31
134.19
126.75
125.43
124.38
119.81
50.12
41.08
40.87
40.66
40.45
40.24
40.03
39.83
37.77
26.33
25.15
23.34
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)


NHAc
OMe
O

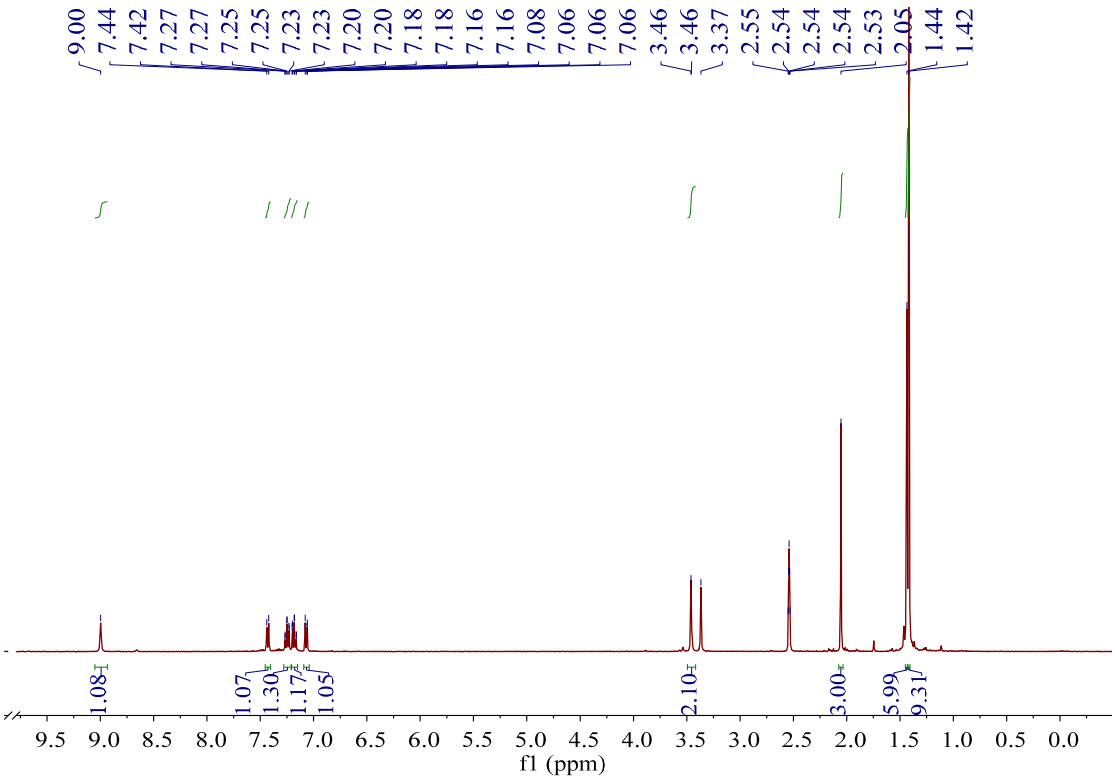
4c

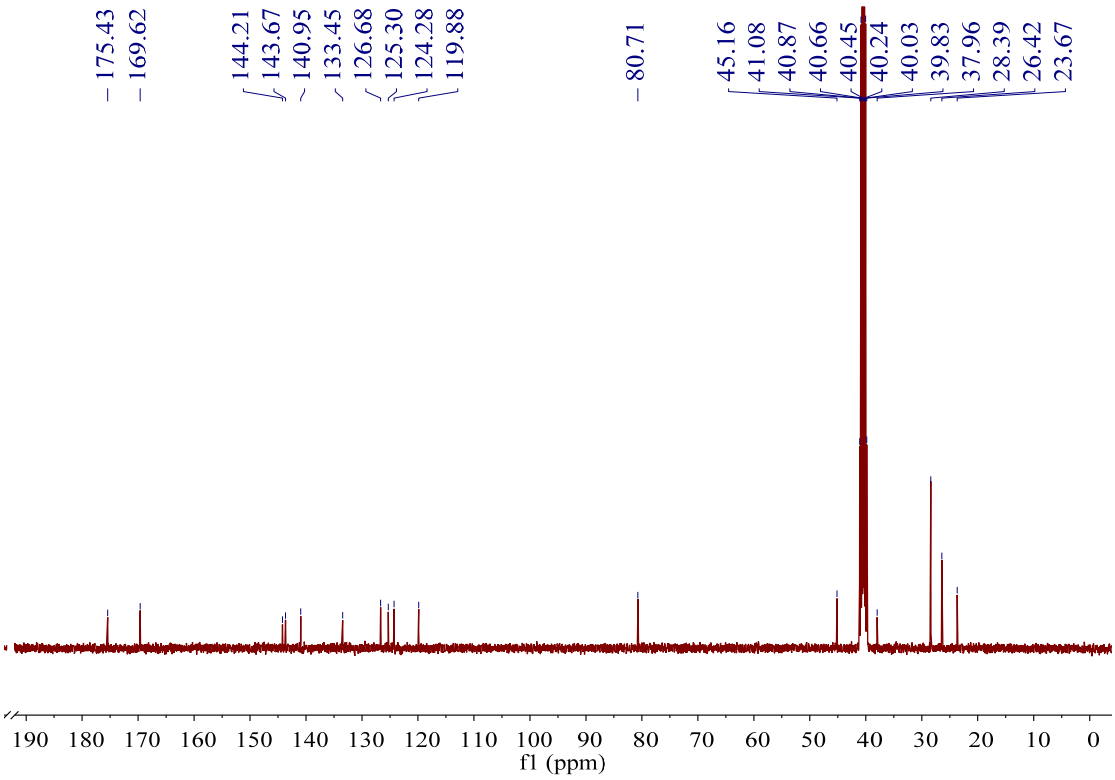

9.50 7.45 7.43 7.28 7.26 7.26 7.24 7.24 7.23 7.22 7.22 7.20 7.20 7.19 7.18 3.87 3.86 3.84 3.82 3.63 3.50 3.44 3.39 3.33 2.55 2.54 2.54 2.53 2.53 2.11 1.38 1.37
1.01 1.09 3.19 1.07 3.08 1.06 1.06 3.00 3.19
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

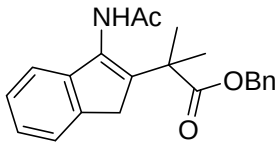

174.73 169.38 143.01 141.83 136.65 134.45 126.77 125.57 124.51 120.12 52.66 41.08 40.87 40.66 40.45 40.24 40.03 39.82 38.94 36.39 23.71 17.74
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
O^{t}Bu
O

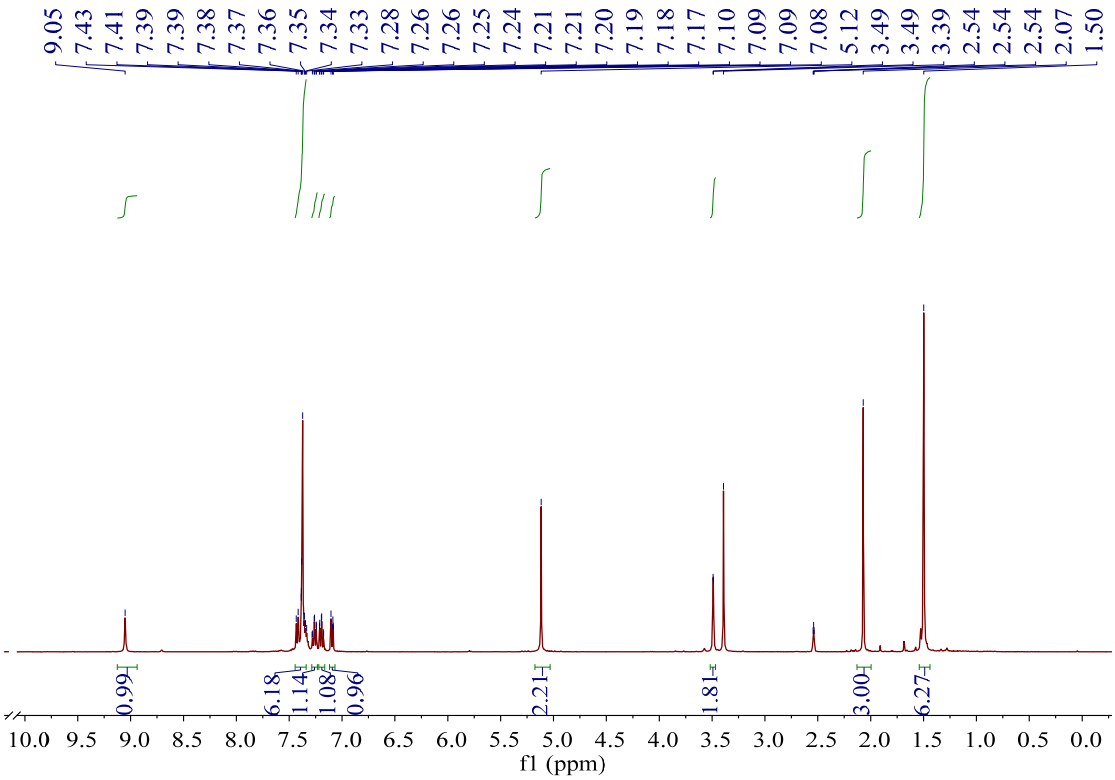
4d

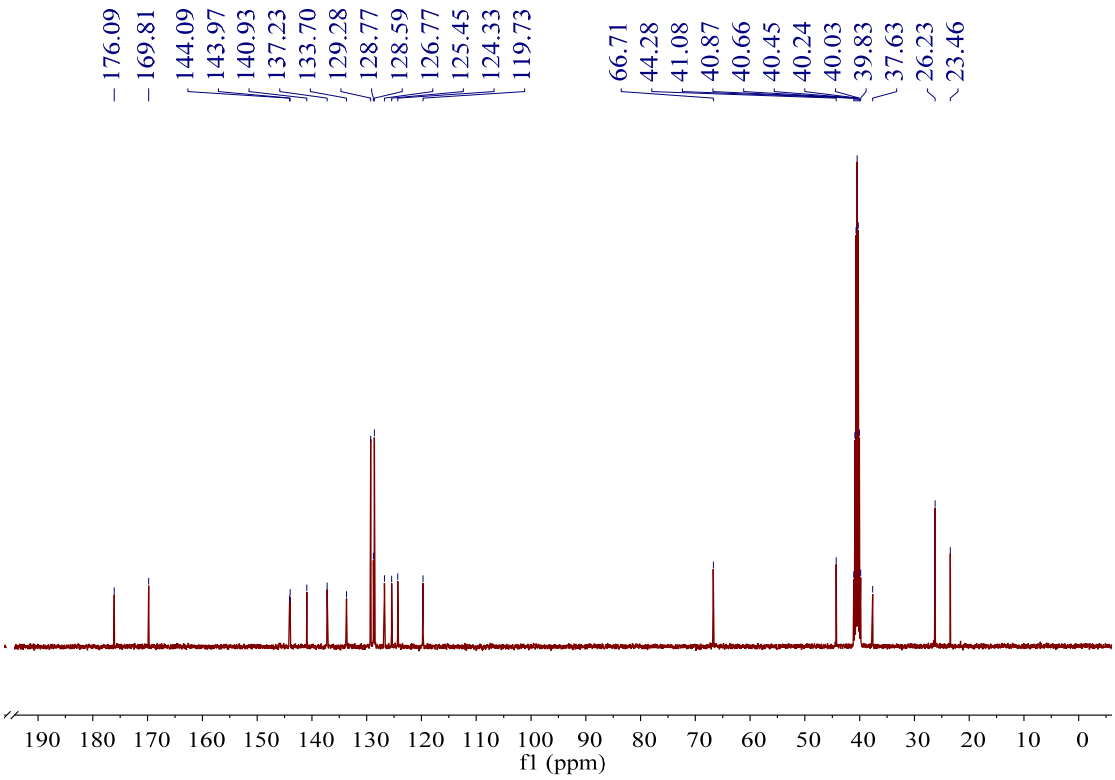

9.00 7.44 7.42 7.27 7.27 7.25 7.25 7.23 7.23 7.20 7.20 7.18 7.18 7.16 7.16 7.08 7.06 7.06 7.06 3.46 3.46 3.37 2.55 2.54 2.54 2.54 2.53 2.05 1.44 1.42
1.08 1.07 1.30 1.17 1.05 2.10 3.00 5.99 9.31
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


175.43 169.62 144.21 143.67 140.95 133.45 126.68 125.30 124.28 119.88 80.71 45.16 41.08 40.87 40.66 40.45 40.24 40.03 39.83 37.96 28.39 26.42 23.67
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OBn
O

4e


9.05 7.43 7.41 7.39 7.39 7.38 7.37 7.36 7.35 7.34 7.33 7.28 7.26 7.26 7.25 7.24 7.21 7.21 7.20 7.19 7.18 7.17 7.10 7.09 7.09 7.08 5.12 3.49 3.49 3.39 2.54 2.54 2.54 2.07 1.50
0.99 6.18 1.14 1.08 0.96 2.21 1.81 3.00 6.27
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
f1 (ppm)


176.09 169.81 144.09 143.97 140.93 137.23 133.70 129.28 128.77 128.59 126.77 125.45 124.33 119.73
66.71 44.28 41.08 40.87 40.66 40.45 40.24 40.03 39.83 37.63 26.23 23.46
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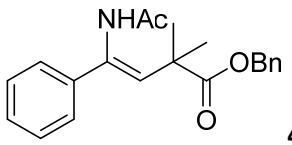
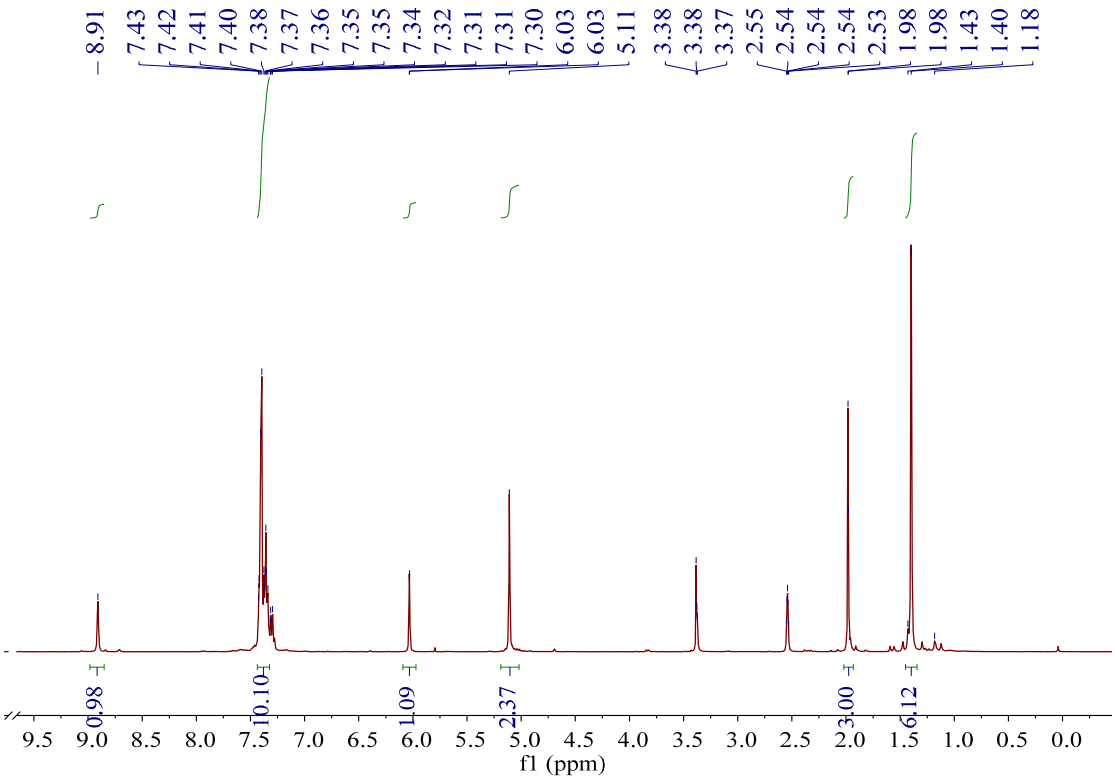
f1 (ppm)


NHAc
OBn
O

4f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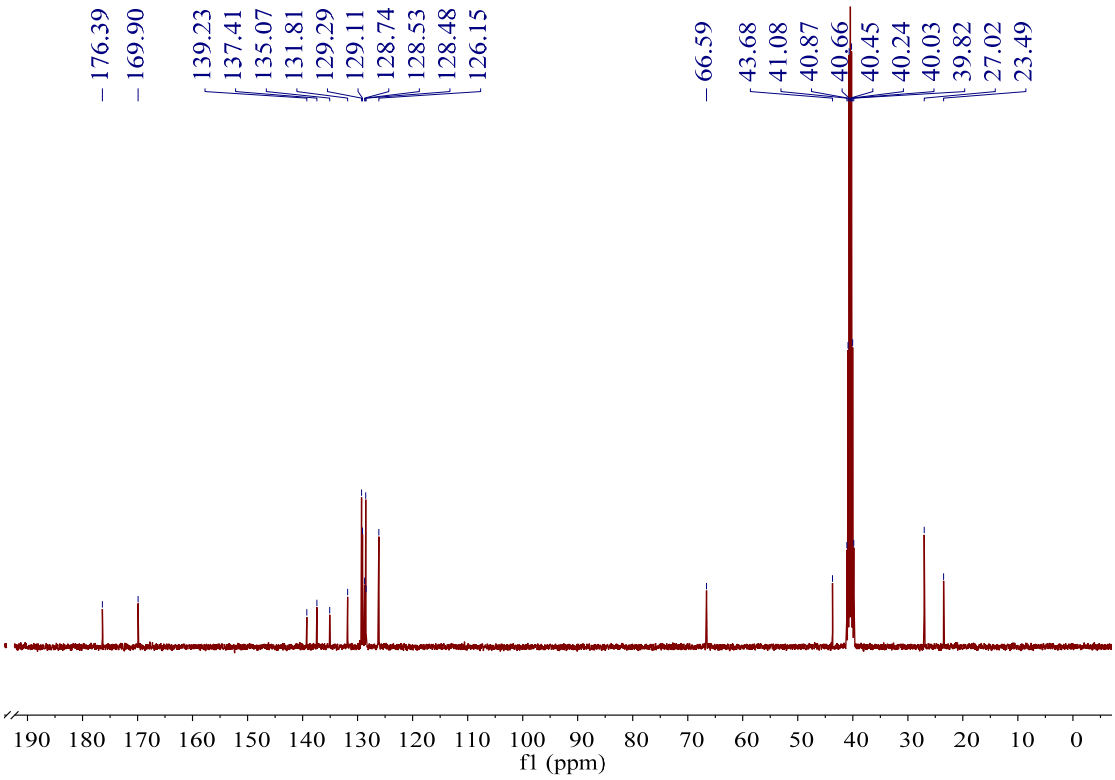
7.43
7.42
7.41
7.40
7.38
7.37
7.36
7.35
7.35
7.34
7.32
7.31
7.31
7.30
6.03
6.03
5.11
3.38
3.38
3.37
2.55
2.54
2.54
2.54
2.53
1.98
1.98
1.43
1.40
1.18
0.98
10.10
1.09
2.37
3.00
6.12
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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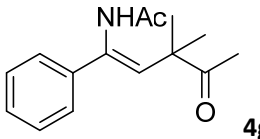
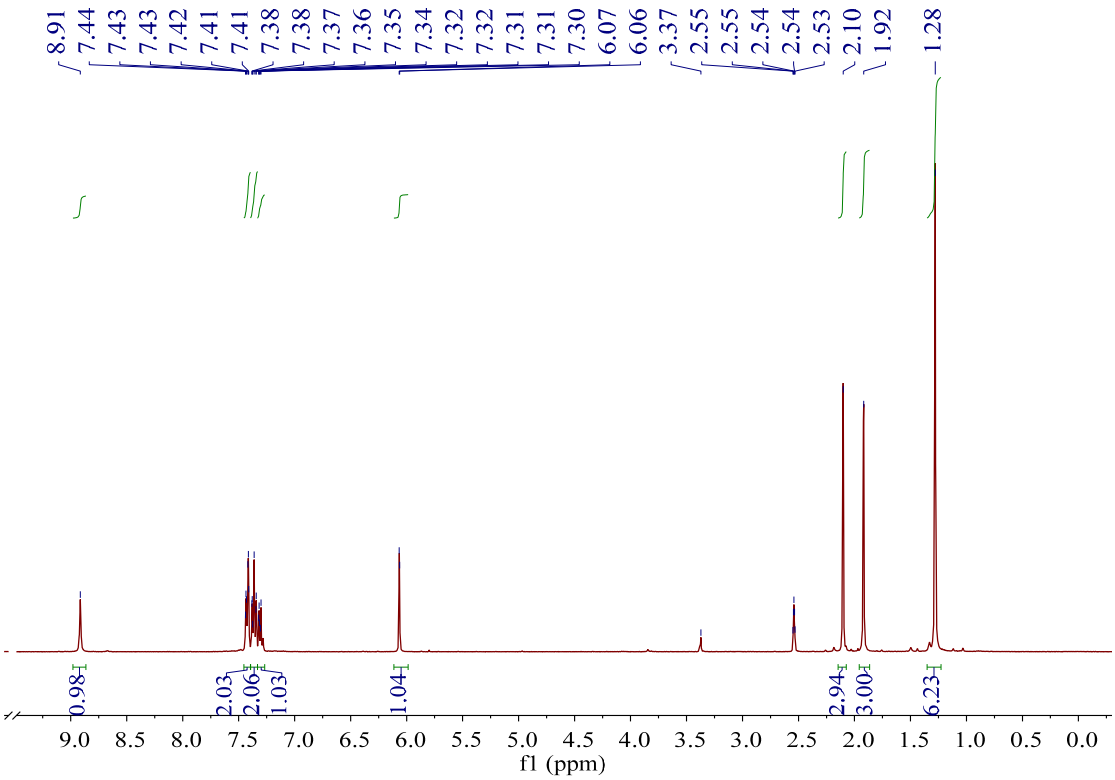
169.90
139.23
137.41
135.07
131.81
129.29
129.11
128.74
128.53
128.48
126.15
66.59
43.68
41.08
40.87
40.66
40.45
40.24
40.03
39.82
27.02
23.49
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
O

4g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7.44
7.43
7.43
7.42
7.41
7.41
7.38
7.38
7.37
7.36
7.35
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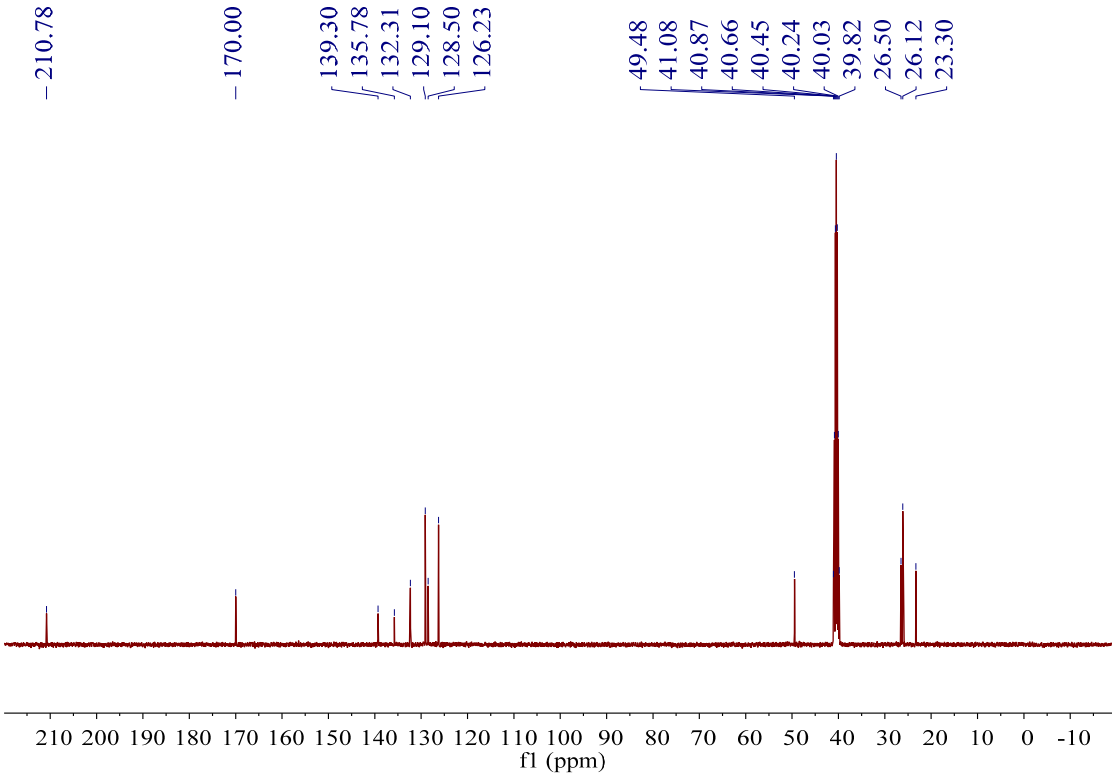
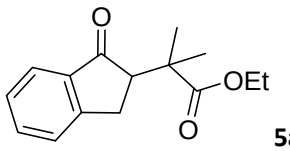
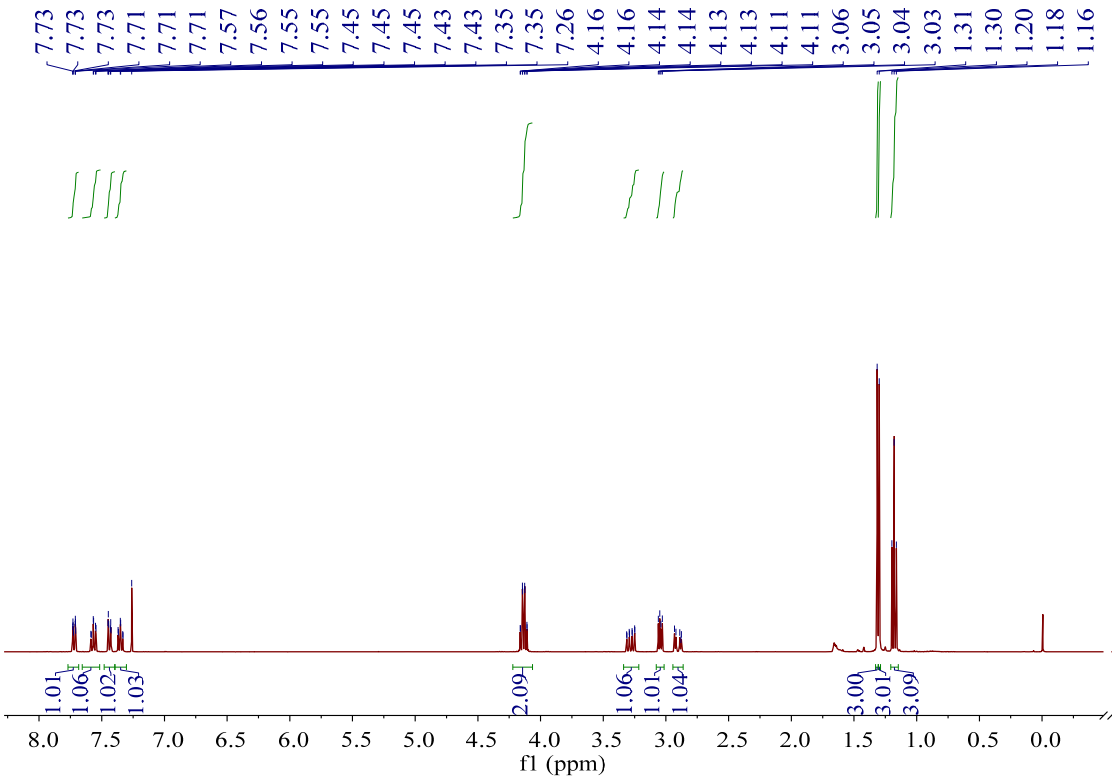
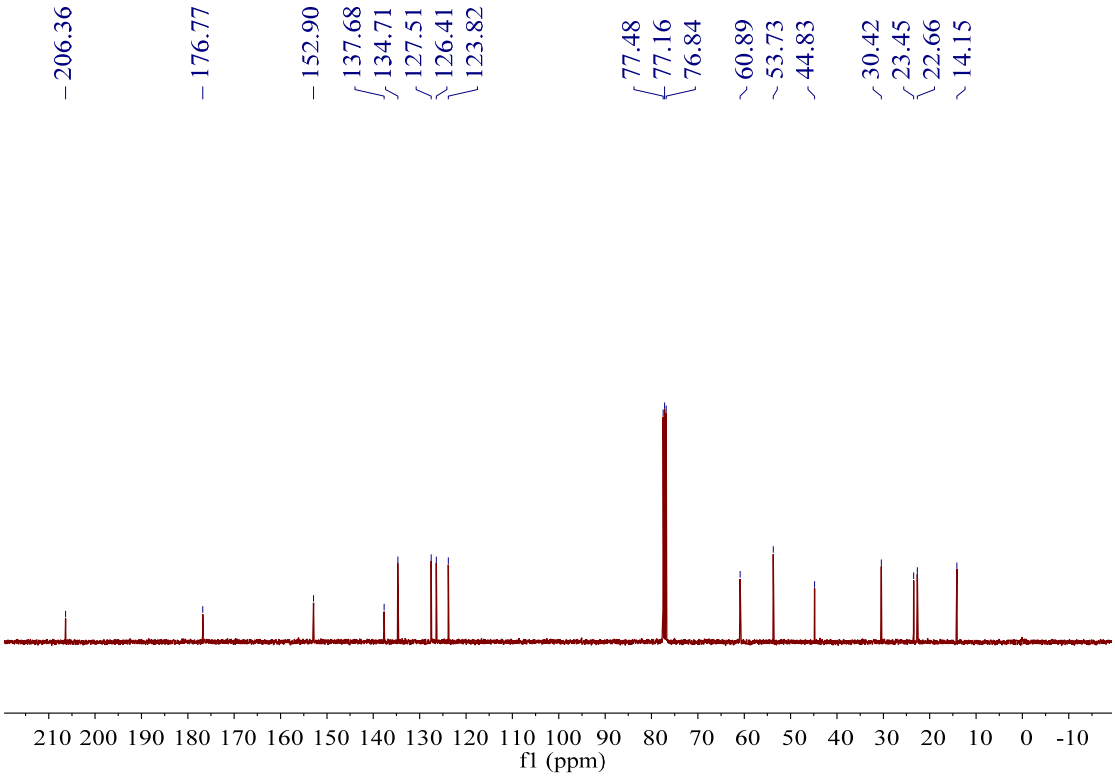
7.32
7.32
7.31
7.31
7.30
6.07
6.06
3.37
2.55
2.55
2.54
2.54
2.53
2.10
1.92
1.28
0.98
2.03
2.06
1.03
1.04
2.94
3.00
6.23
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


210.78
170.00
139.30
135.78
132.31
129.10
128.50
126.23
49.48
41.08
40.87
40.66
40.45
40.24
40.03
39.82
26.50
26.12
23.30
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)


O
OEt
O

5a


7.73 7.73 7.73 7.71 7.71 7.71 7.57 7.56 7.55 7.55 7.45 7.45 7.45 7.43 7.43 7.35 7.35 7.26 4.16 4.16 4.14 4.14 4.13 4.13 4.11 4.11 3.06 3.05 3.04 3.03 1.31 1.30 1.20 1.18 1.16
1.01 1.06 1.02 1.03 2.09 1.06 1.01 1.04 3.00 3.01 3.09
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
f1 (ppm)


206.36 176.77 152.90 137.68 134.71 127.51 126.41 123.82 77.48 77.16 76.84 60.89 53.73 44.83 30.42 23.45 22.66 14.15
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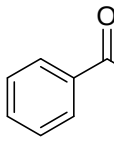
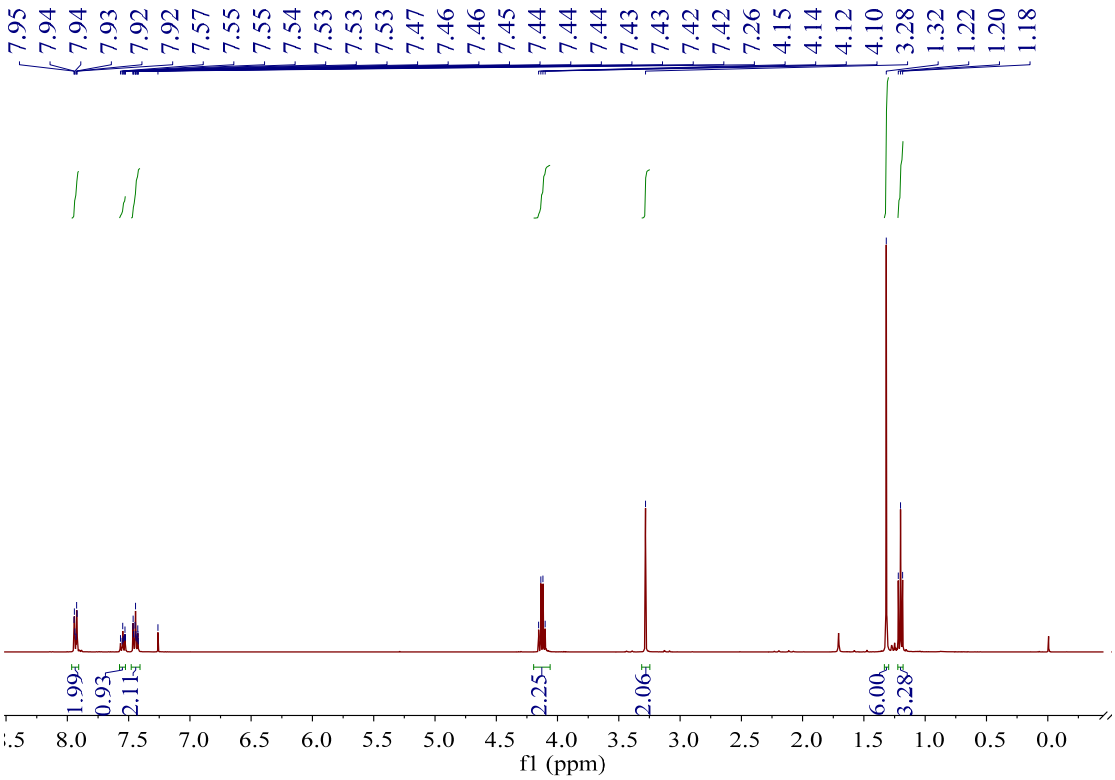
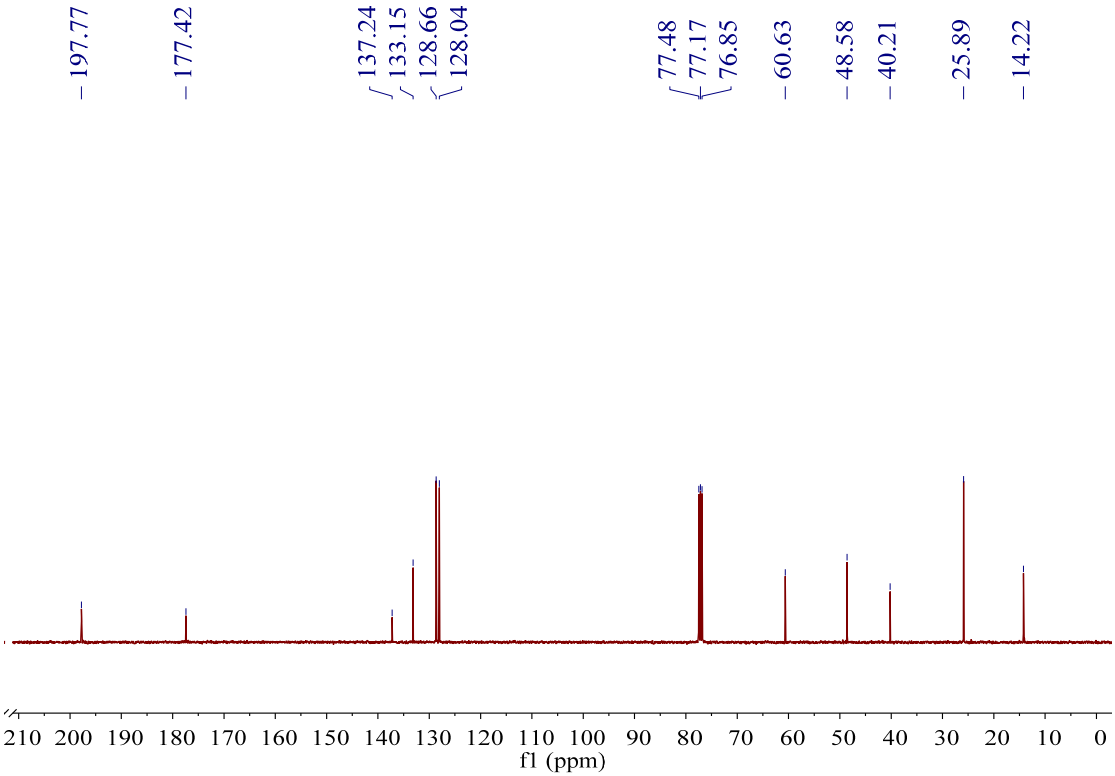
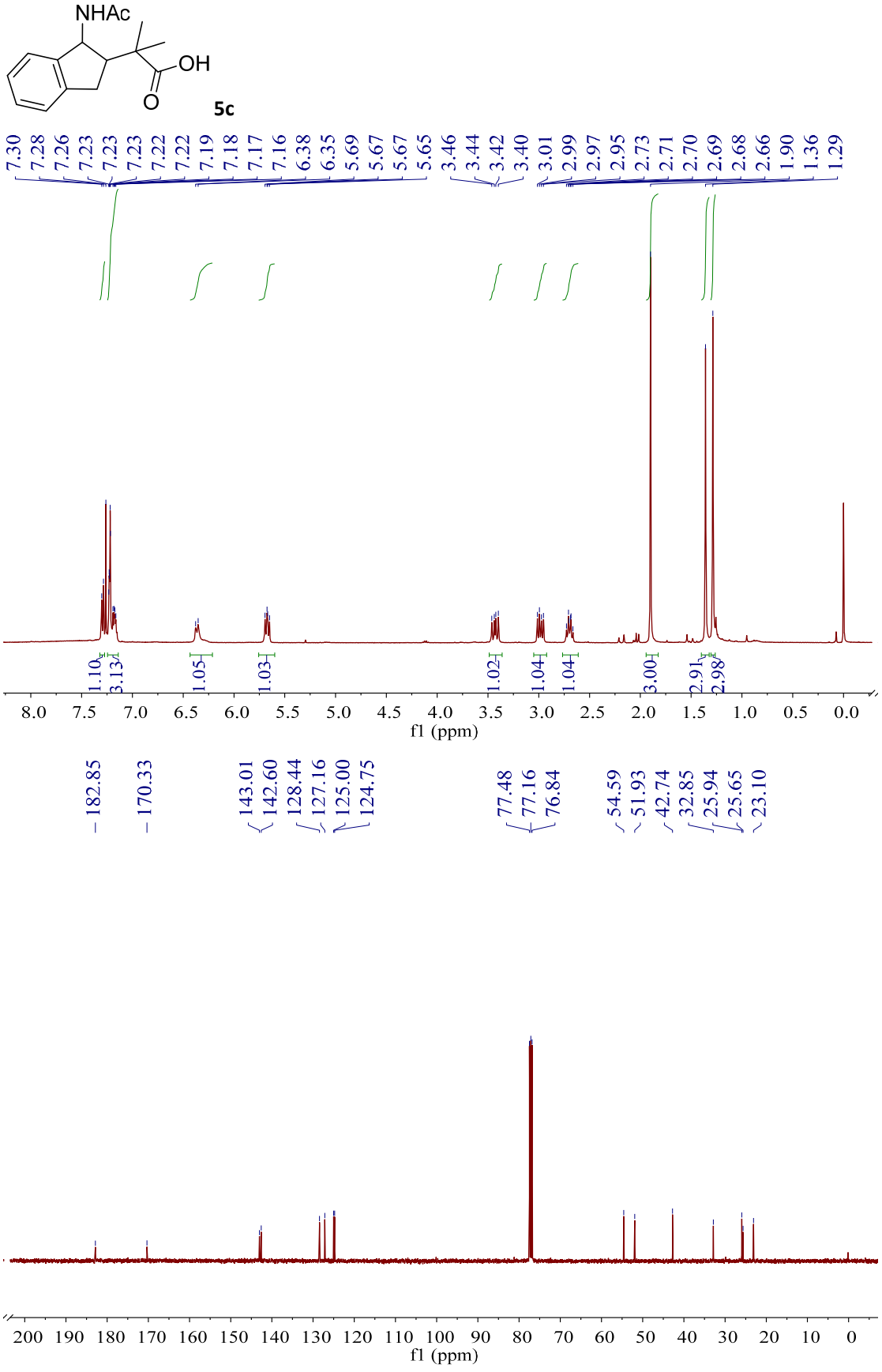
f1 (ppm)

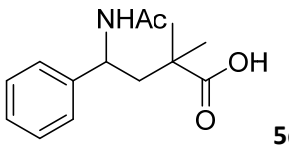

O
OEt
O

5b

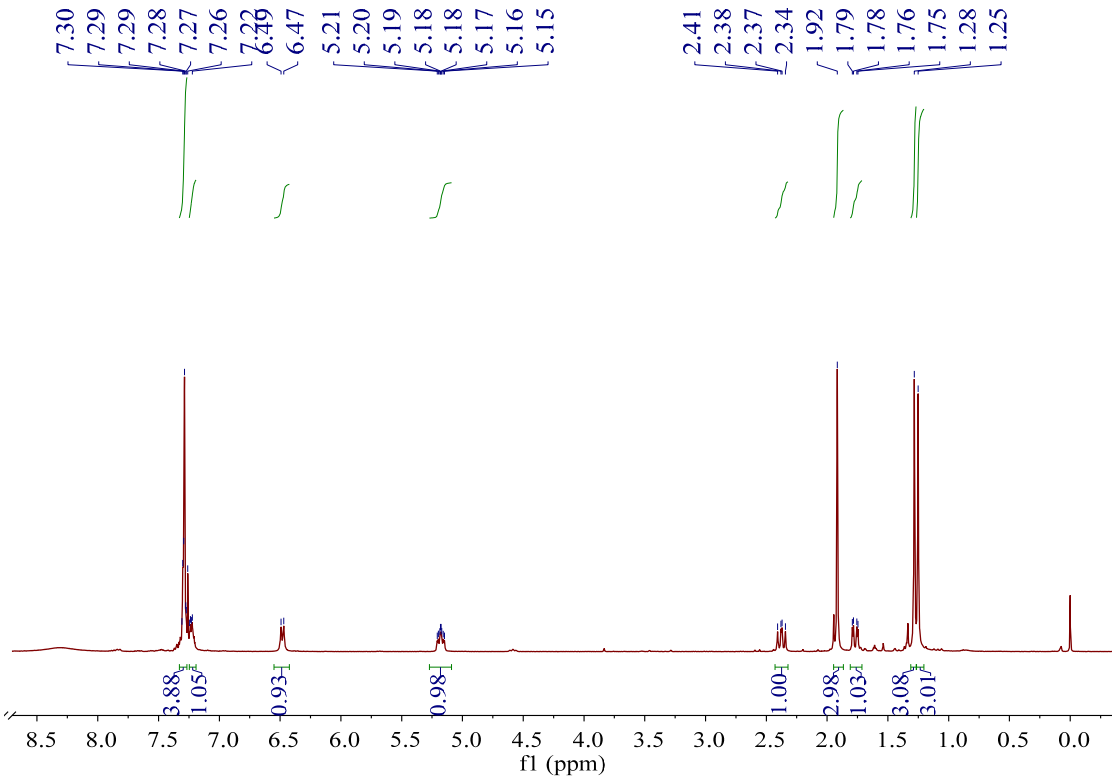

7.95 7.94 7.94 7.93 7.92 7.92 7.57 7.55 7.55 7.54 7.53 7.53 7.53 7.47 7.46 7.46 7.45 7.44 7.44 7.44 7.43 7.43 7.42 7.42 7.26 4.15 4.14 4.12 4.10 3.28 1.32 1.22 1.20 1.18
1.99 0.93 2.11 2.25 2.06 6.00 3.28
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


197.77 177.42 137.24 133.15 128.66 128.04 77.48 77.17 76.85 60.63 48.58 40.21 25.89 14.22
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OH
O
5c
7.30 7.28 7.26 7.23 7.23 7.23 7.22 7.22 7.19 7.18 7.17 7.16 6.38 6.35 5.69 5.67 5.67 5.65
3.46 3.44 3.42 3.40 3.01 2.99 2.97 2.95 2.73 2.71 2.70 2.69 2.68 2.66 1.90 1.36 1.29
1.10 3.13 1.05 1.03 1.02 1.04 1.04 3.00 2.91 2.98
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
f1 (ppm)
182.85 170.33 143.01 142.60 128.44 127.16 125.00 124.75 77.48 77.16 76.84 54.59 51.93 42.74 32.85 25.94 25.65 23.10
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OH
O

5d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7.29
7.28
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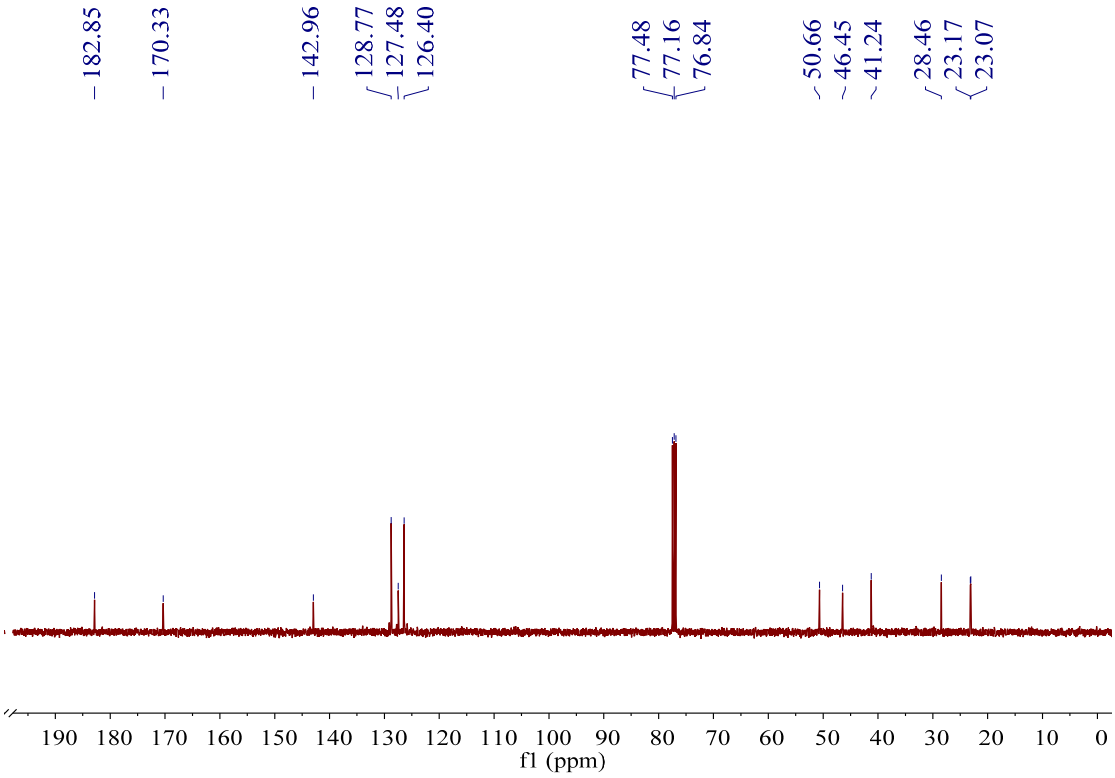
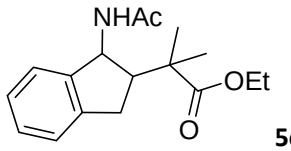
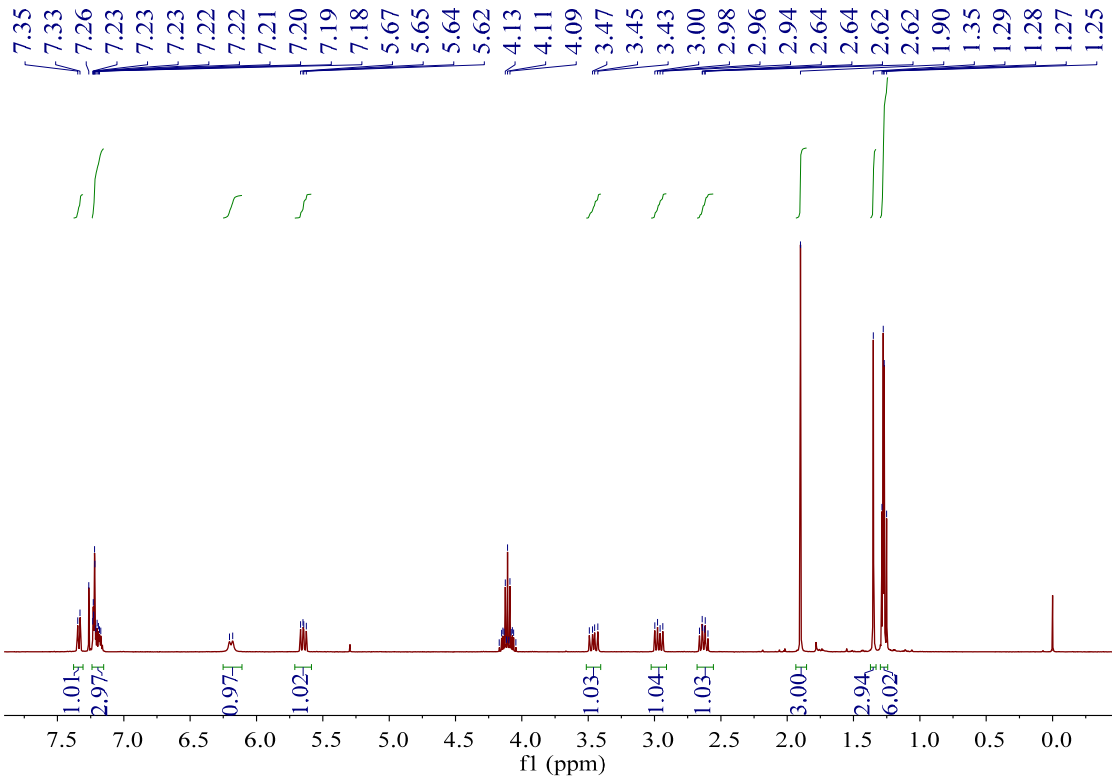
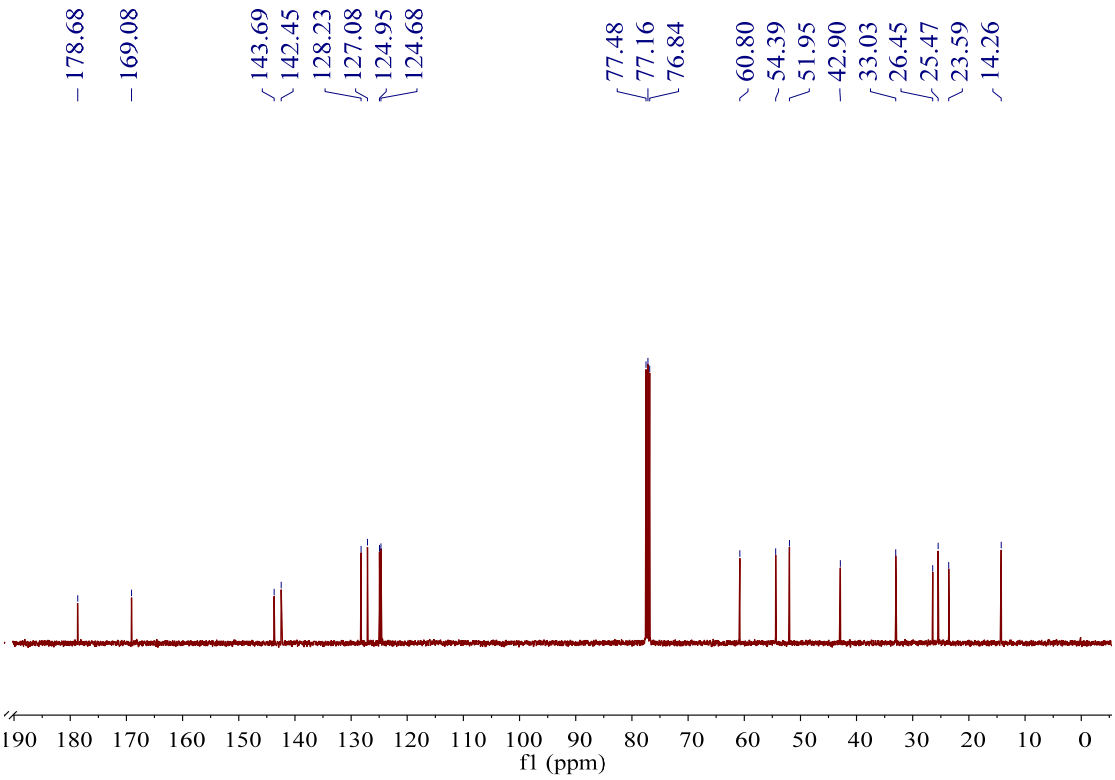
7.22
6.49
6.47
5.21
5.20
5.19
5.18
5.18
5.17
5.16
5.15
2.41
2.38
2.37
2.34
1.92
1.79
1.78
1.76
1.75
1.28
1.25
3.88
1.05
0.93
0.98
1.00
2.98
1.03
3.08
3.01
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


182.85
170.33
142.96
128.77
127.48
126.40
77.48
77.16
76.84
50.66
46.45
41.24
28.46
23.17
23.07
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O

5e


7.35 7.33 7.26 7.23 7.23 7.23 7.22 7.22 7.21 7.20 7.19 7.18 5.67 5.65 5.64 5.62 4.13 4.11 4.09 3.47 3.45 3.43 3.00 2.98 2.96 2.94 2.64 2.64 2.62 2.62 1.90 1.35 1.29 1.28 1.27 1.25
1.01 2.97 0.97 1.02 1.03 1.04 1.03 3.00 2.94 6.02
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
f1 (ppm)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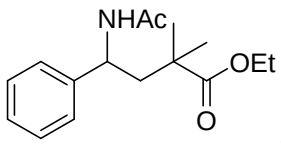
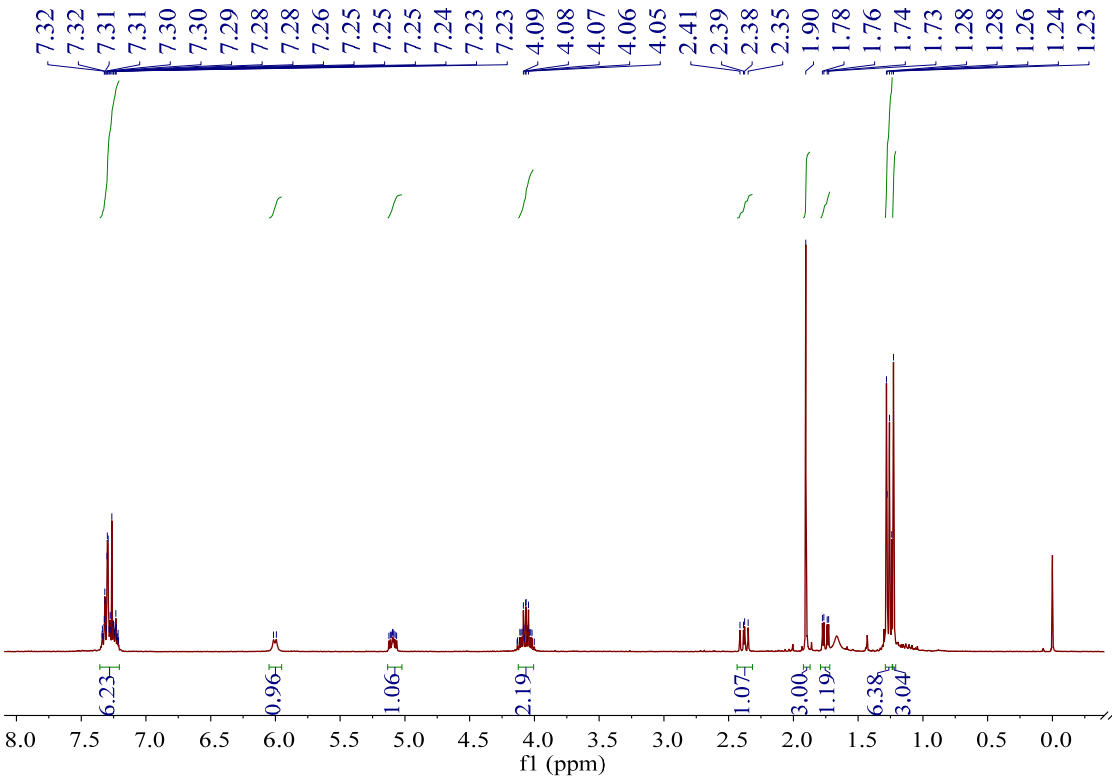
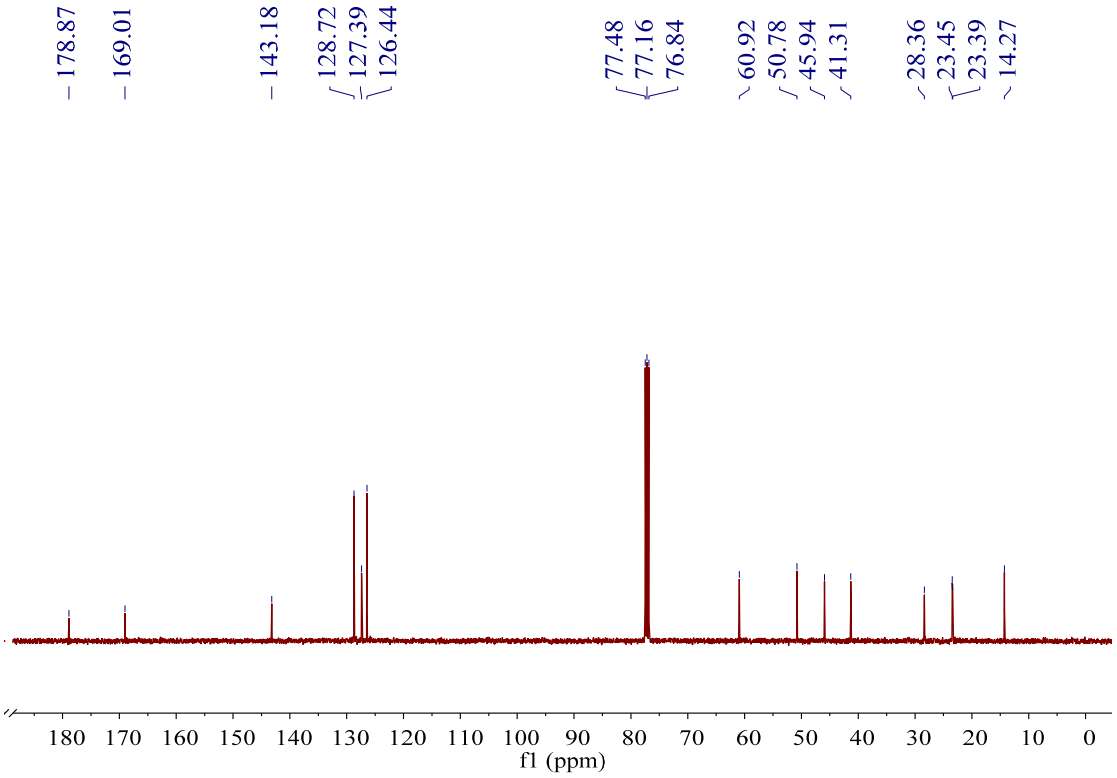
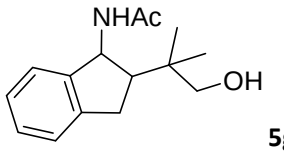
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OEt
O

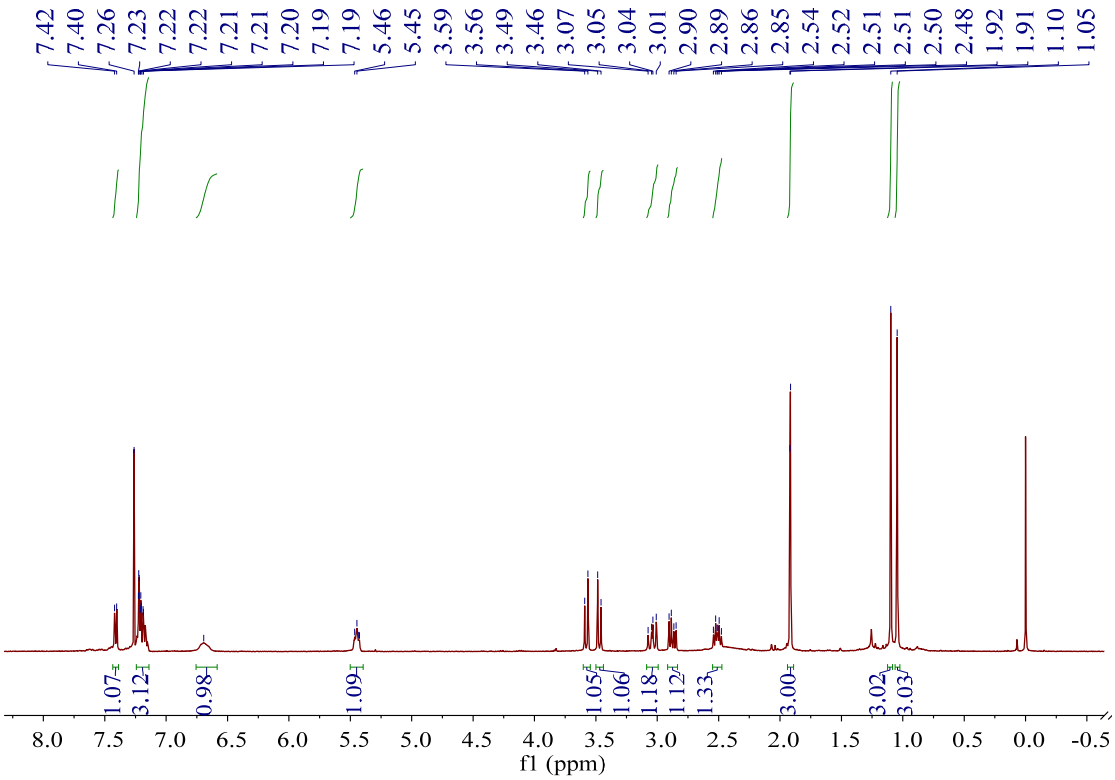
5f


7.32 7.32 7.31 7.31 7.30 7.30 7.29 7.28 7.28 7.26 7.25 7.25 7.25 7.24 7.23 7.23
4.09 4.08 4.07 4.06 4.05
2.41 2.39 2.38 2.35
1.90 1.78 1.76 1.74 1.73 1.28 1.28 1.26 1.24 1.23
6.23 0.96 1.06 2.19 1.07 3.00 1.19 6.38 3.04
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
f1 (ppm)


178.87 169.01 143.18 128.72 127.39 126.44 77.48 77.16 76.84 60.92 50.78 45.94 41.31 28.36 23.45 23.39 14.27
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OH

5g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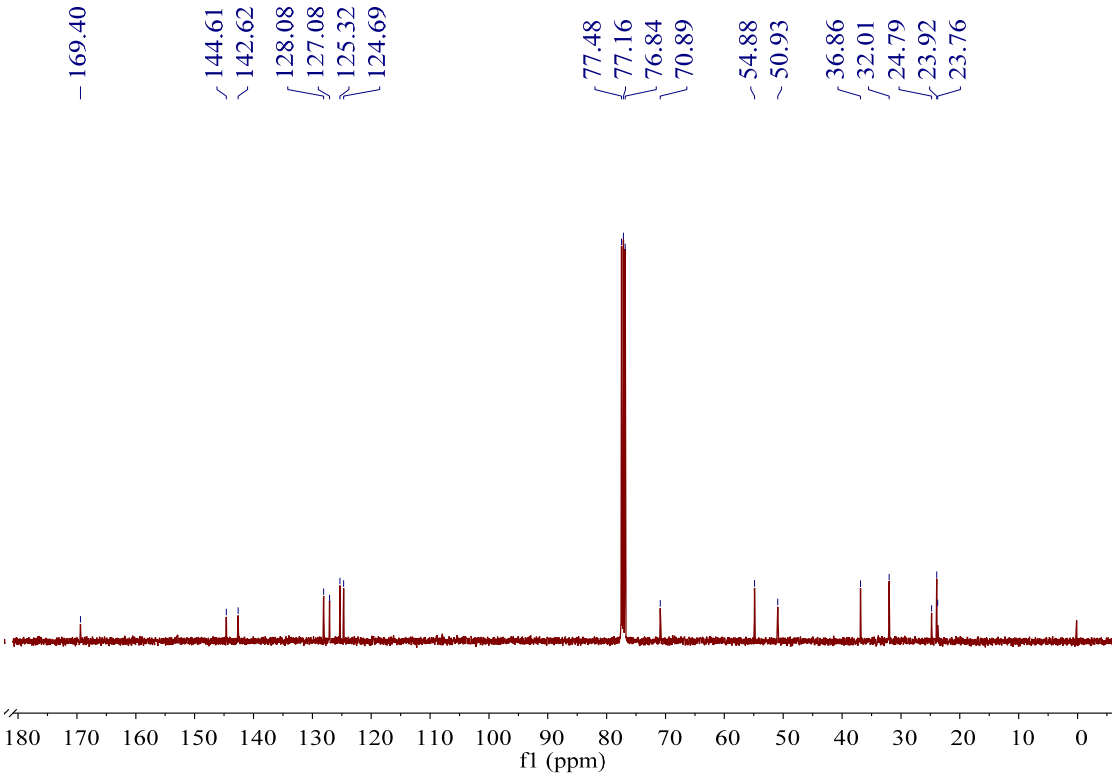
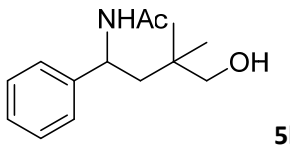
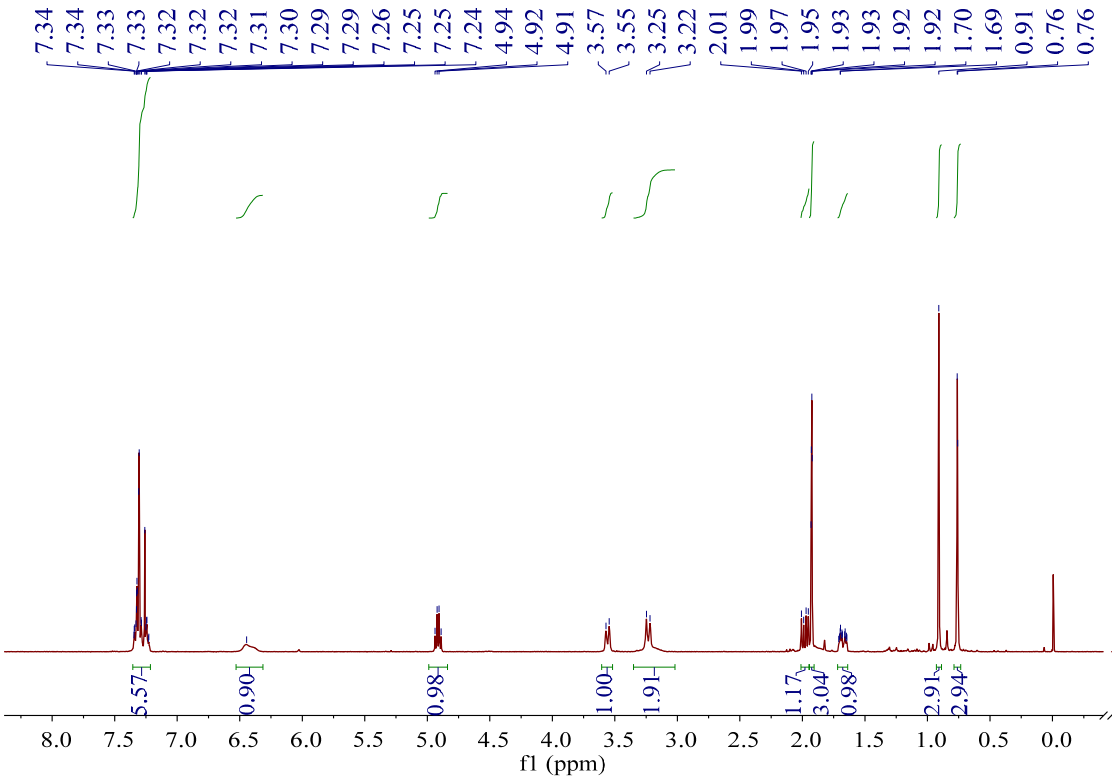
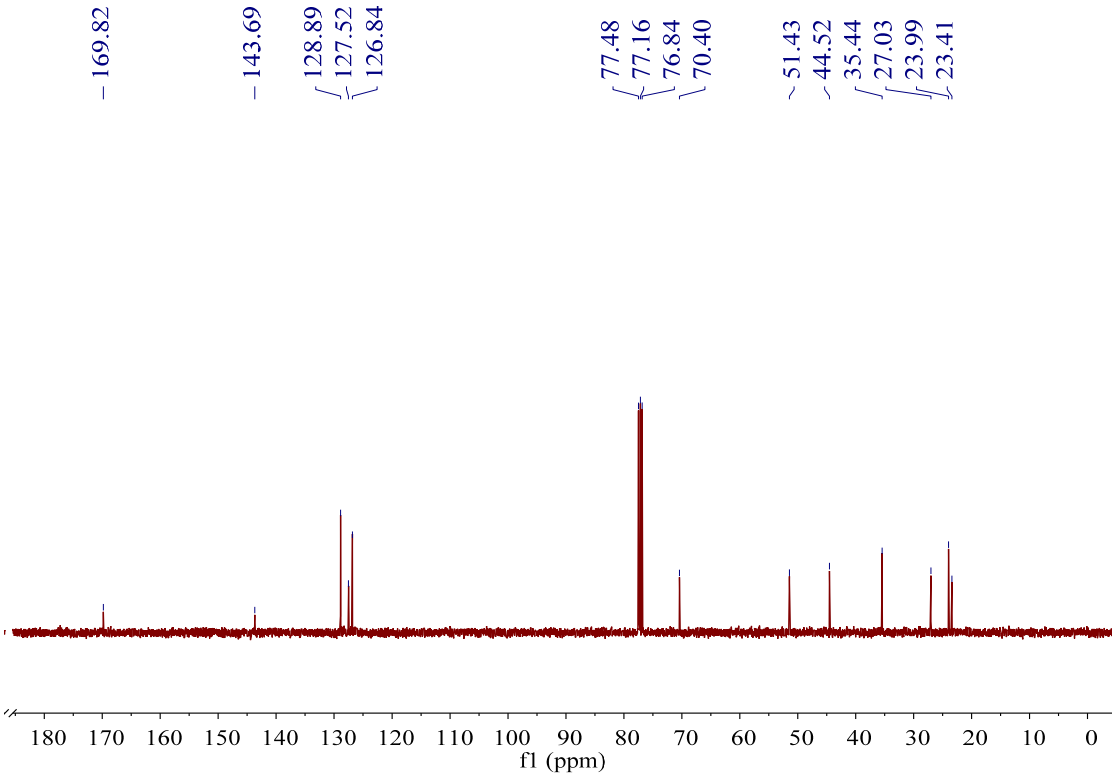
1.07 3.12 0.98 1.09 1.05 1.06 1.18 1.12 1.33 3.00 3.02 3.03
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

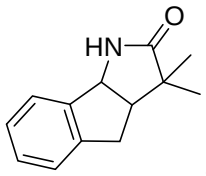

169.40
144.61 142.62 128.08 127.08 125.32 124.69
77.48 77.16 76.84 70.89 54.88 50.93 36.86 32.01 24.79 23.92 23.76
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


NHAc
OH

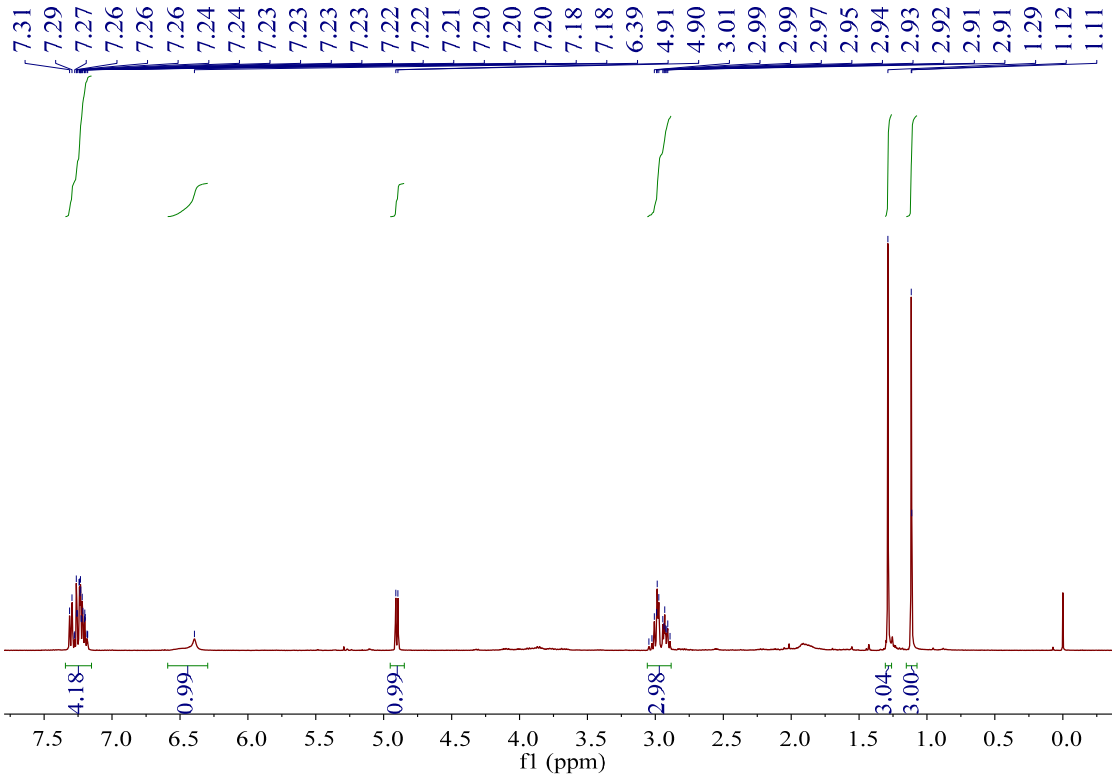
5h

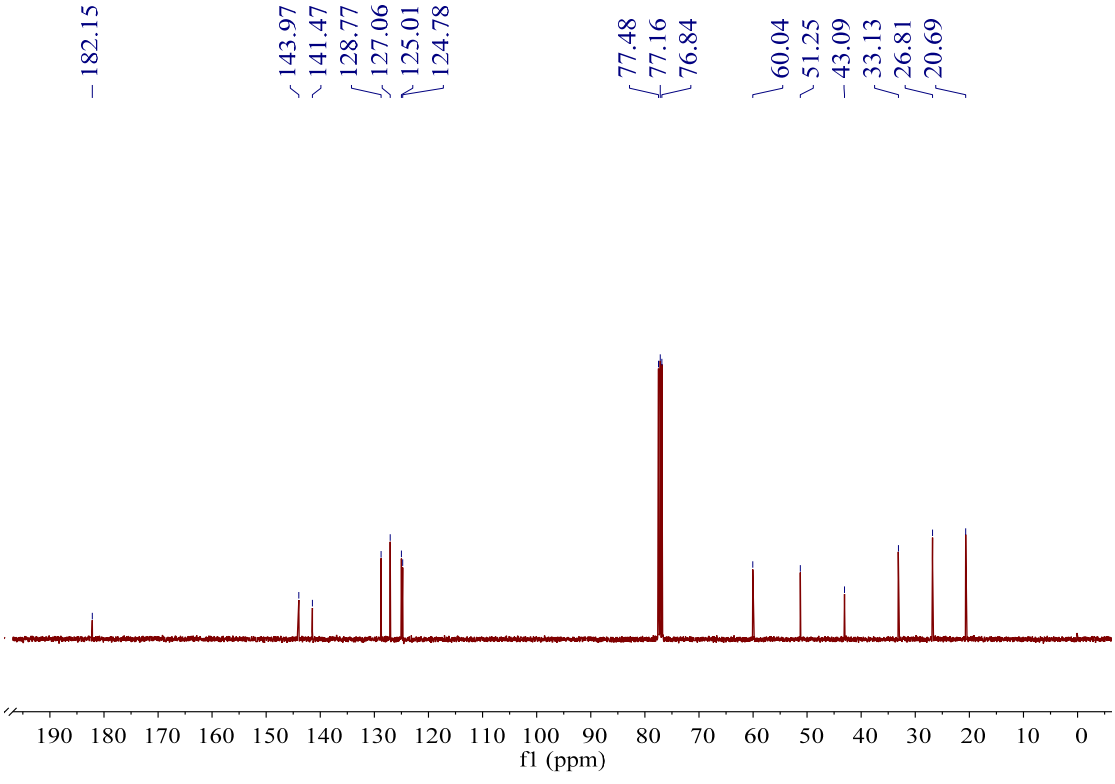

7.34 7.34 7.33 7.33 7.32 7.32 7.32 7.31 7.30 7.29 7.29 7.26 7.25 7.25 7.24 4.94 4.92 4.91 3.57 3.55 3.25 3.22 2.01 1.99 1.97 1.95 1.93 1.93 1.92 1.92 1.70 1.69 0.91 0.76 0.76
5.57 0.90 0.98 1.00 1.91 1.17 3.04 0.98 2.91 2.94
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

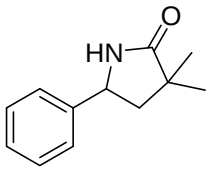

169.82 143.69 128.89 127.52 126.84 77.48 77.16 76.84 70.40 51.43 44.52 35.44 27.03 23.99 23.41
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


HN
O

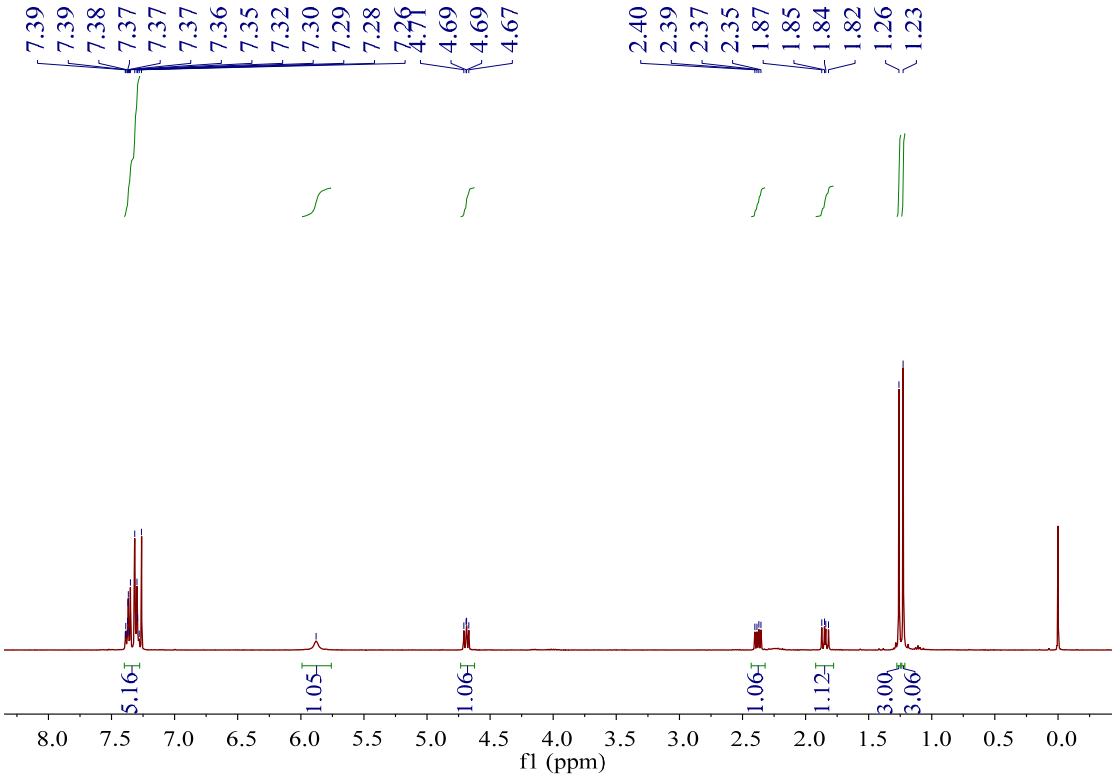
5i

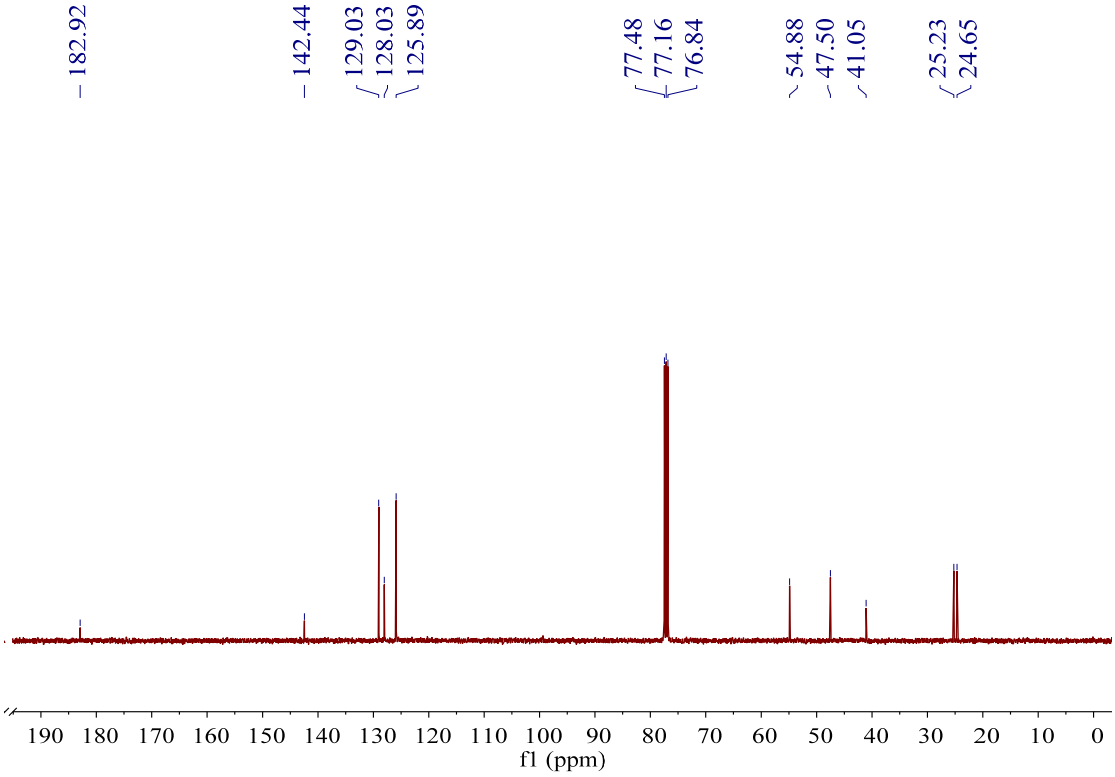

7.31 7.29 7.27 7.26 7.26 7.26 7.24 7.24 7.23 7.23 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.20 7.18 7.18 6.39 4.91 4.90 3.01 2.99 2.99 2.97 2.95 2.94 2.93 2.92 2.91 2.91 1.29 1.12 1.11
4.18 0.99 0.99 2.98 3.04 3.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


182.15
143.97 141.47 128.77 127.06 125.01 124.78
77.48 77.16 76.84
60.04 51.25 43.09 33.13 26.81 20.69
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
HN

5j


7.39
7.39
7.38
7.37
7.37
7.37
7.36
7.35
7.32
7.30
7.29
7.28
7.26
4.71
4.69
4.69
4.67
2.40
2.39
2.37
2.35
1.87
1.85
1.84
1.82
1.26
1.23
5.16
1.05
1.06
1.06
1.12
3.00
3.06
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
f1 (ppm)


182.92
142.44
129.03
128.03
125.89
77.48
77.16
76.84
54.88
47.50
41.05
25.23
24.65
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)