Supporting Information

A Unique 1,2-Acyl Migration for the Construction of Quaternary Carbon by Visible Light Irradiation of Platinum (II) Polypyridyl Complex and Molecular Oxygen

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1. Experimental details

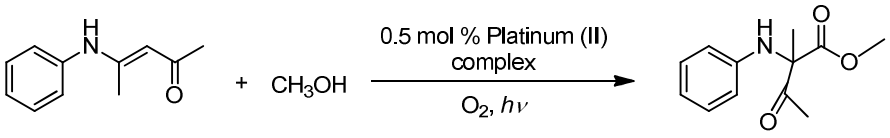
a) General Information

^{1}H NMR spectra were recorded using a Bruker Avance DPX 400 MHz instrument with tetramethylsilane (TMS) as an internal standard. ^{13}C NMR spectra were obtained at 100 MHz and referenced to the internal solvent signals. HRMS (ESI) spectra were recorded on Thermo Scientific Q Exactive Mass Spectrometer. ESR spectra were recorded at room temperature using a Bruker ESP-300E spectrometer at 9.8 GHz, X-band, with 100 Hz field modulation. Solvents were dried by the general methods before use. Photoirradiation was carried out with LEDs (blue light). Commercially available reagents were used without further purification. All of the enamines needed for 1,2-acyl migration reactions were prepared using the reported procedure and were further purified through column chromatography.[1]

b) General procedure for 1,2-acyl migration reaction catalyzed by visible light:

A 10 mL Pyrex tube equipped with a magnetic stir bar was charged with enamine **1** (0.1 mmol), platinum (II) complex (0.0005 mmol) and the corresponding alcohol **2** (2 mL), then the mixture was bubbled with a stream of O_2 for 15 min. and sealed under irradiation with LEDs (blue light) at room temperature for 1.5 h. Then the mixture was evaporated under reduced pressure to remove the solvent and the residue was purified by flash chromatography on silica gel to afford the desired product .

Scheme S1. Gram Scale Reaction

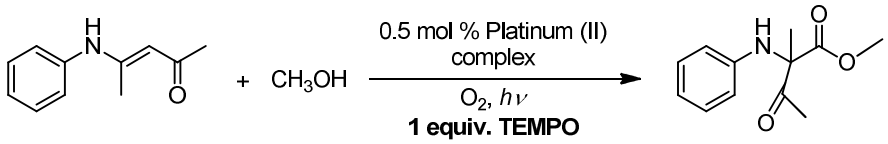


1a (1.35 g)

3a (1.02 g)

yield: 60%

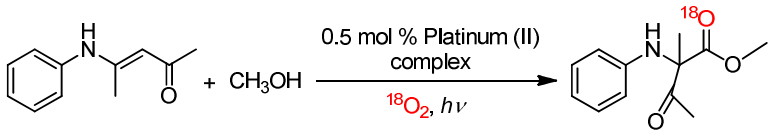
Scheme S2. Radical Captured Experiment



1a

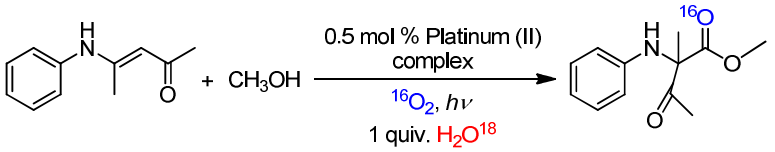
3a, yield: 64%

Scheme S3. Isotope Experiments



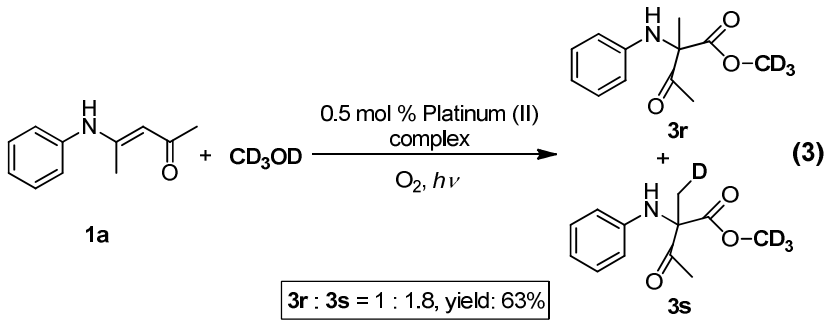
1a

3p, m/z = 223 **(1)**



1a

3a, m/z = 221 **(2)**



1a

3r

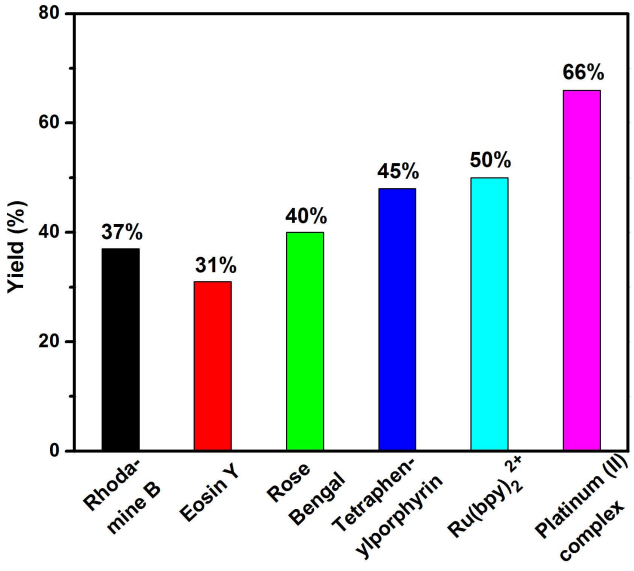
+

3s

3r : **3s** = 1 : 1.8, yield: 63%

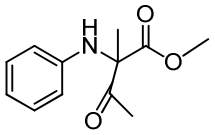
(3)

Figure S1. Effect of Photosensitizer in the 1,2-Acyl Migration Reaction



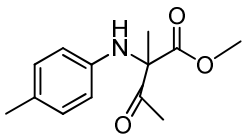
2. Characterization of all products

methyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3a)



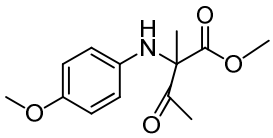
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), oily brown liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (t, J = 7.9 Hz, 2H), 6.74 (t, J = 7.3 Hz, 1H), 6.52 (d, J = 7.8 Hz, 2H), 5.25 (s, 1H), 3.79 (s, 3H), 2.20 (s, 3H), 1.69 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 171.9, 144.0, 129.6, 118.5, 114.3, 69.8, 53.7, 24.9, 18.8. HRMS (ESI) Calcd. for $C_{12}H_{16}NO_3$ [M+H]: 222.1130. Found: 222.1120.

methyl 2-methyl-3-oxo-2-(p-tolylamino)butanoate (3b)



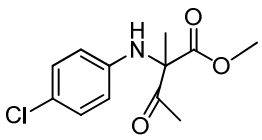
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (d, J = 8.1 Hz, 2H), 6.49 – 6.40 (m, 2H), 5.10 (s, 1H), 3.79 (s, 3H), 2.23 (s, 3H), 2.20 (s, 3H), 1.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 172.0, 141.7, 130.1, 127.9, 114.6, 69.9, 53.7, 25.0, 20.6, 18.8. HRMS (ESI) Calcd. for $C_{13}H_{18}NO_3$ [M+H]: 236.1287. Found: 236.1274.

methyl 2-methyl-3-oxo-2-(p-tolylamino)butanoate (3c)



Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.2), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.74 (dd, J = 6.7, 2.3 Hz, 2H), 6.51 (dd, J = 6.7, 2.3 Hz, 2H), 5.03 (s, 1H), 3.78 (s, 3H), 3.73 (s, 3H), 2.21 (s, 3H), 1.62 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.1, 172.1, 153.2, 137.9, 116.6, 115.1, 70.3, 55.8, 53.6, 25.0, 19.0. HRMS (ESI) Calcd. for $C_{13}H_{18}NO_4$ [M+H]: 252.1236. Found: 252.1223.

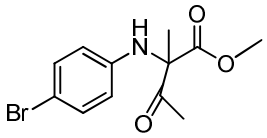
methyl 2-((4-chlorophenyl)amino)-2-methyl-3-oxobutanoate (3d)



Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f =

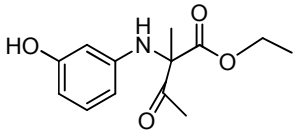
0.3), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (d, J = 8.8 Hz, 2H), 6.46 (d, J = 8.9 Hz, 2H), 3.78 (s, 3H), 2.19 (s, 3H), 1.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.1, 171.7, 142.7, 129.5, 123.4, 115.5, 69.8, 53.9, 24.8, 18.7. HRMS (ESI) Calcd. for $C_{12}H_{15}ClNO_3$ [M+H]: 256.0741. Found: 256.0728.

methyl 2-((4-bromophenyl)amino)-2-methyl-3-oxobutanoate (3e)



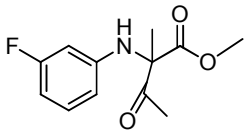
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.3), oily brown liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.18 (m, 2H), 6.44 – 6.35 (m, 2H), 5.34 (s, 1H), 3.78 (s, 3H), 2.18 (s, 3H), 1.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 171.6, 143.2, 132.4, 115.9, 110.4, 69.7, 53.9, 24.8, 18.7. HRMS (ESI) Calcd. for $C_{12}H_{15}BrNO_3$ [M+H]: 300.0235. Found: 300.0222.

ethyl 2-(3-hydroxyphenylamino)-2-methyl-3-oxobutanoate (3f)



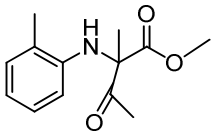
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 4:1, R_f = 0.3), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (t, J = 8.1 Hz, 1H), 6.22 (dd, J = 8.0, 1.8 Hz, 1H), 6.11 (dd, J = 8.1, 1.8 Hz, 1H), 6.02 (s, 1H), 5.30 (s, 1H), 4.25 (q, J = 7.1 Hz, 2H), 2.19 (s, 3H), 1.67 (s, 3H), 1.23 (t, J = 7.2 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.1, 171.3, 157.0, 145.6, 130.6, 107.2, 105.7, 101.2, 69.9, 63.0, 24.9, 18.8, 14.1. HRMS (ESI) Calcd. for $C_{13}H_{18}NO_4$ [M+H]: 252.1236. Found: 252.1224.

methyl 2-(3-fluorophenylamino)-2-methyl-3-oxobutanoate (3g)



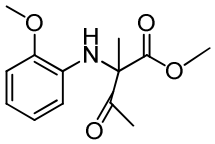
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.2), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08 (dd, J = 15.0, 8.2 Hz, 1H), 6.42 (s, 1H), 6.25 (dd, J = 24.1, 9.8 Hz, 2H), 3.79 (s, 3H), 2.19 (s, 3H), 1.69 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 171.5, 165.2, 162.8, 145.8, 130.7, 109.8, 105.1, 104.9, 101.2, 101.0, 69.7, 53.8, 24.7, 18.7. HRMS (ESI) Calcd. for $C_{12}H_{15}FNO_3$ [M+H]: 240.1036. Found: 240.1024.

methyl 2-methyl-3-oxo-2-(o-tolylamino)butanoate (3h)



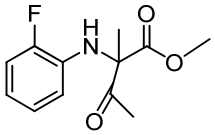
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (d, J = 7.3 Hz, 1H), 7.02 (t, J = 7.3 Hz, 1H), 6.68 (t, J = 7.4 Hz, 1H), 6.29 (d, J = 8.0 Hz, 1H), 5.33 (s, 1H), 3.79 (s, 3H), 2.26 (s, 3H), 2.20 (s, 3H), 1.71 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 172.2, 142.1, 130.9, 127.2, 123.5, 118.1, 111.1, 69.8, 53.8, 24.8, 19.0, 17.9. HRMS (ESI) Calcd. for $C_{13}H_{18}NO_3$ [M+H]: 236.1287. Found: 236.1277.

methyl 2-(2-methoxyphenylamino)-2-methyl-3-oxobutanoate (3i)



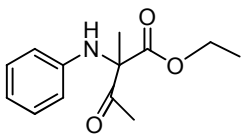
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.3), oily brown liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 – 6.65 (m, 3H), 6.31 (dd, J = 7.7, 1.6 Hz, 1H), 5.88 (s, 1H), 3.88 (s, 3H), 3.79 (s, 3H), 2.19 (s, 3H), 1.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 171.9, 147.7, 133.9, 121.1, 117.8, 111.3, 110.1, 69.6, 55.6, 53.6, 24.9, 18.7. HRMS (ESI) Calcd. for $C_{13}H_{18}NO_4$ [M+H]: 252.1236. Found: 252.1226.

methyl 2-(2-fluorophenylamino)-2-methyl-3-oxobutanoate (3j)



Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (td, J = 11.7, 8.1, 1.3 Hz, 1H), 6.90 (t, J = 7.7 Hz, 1H), 6.73 – 6.59 (m, 1H), 6.49 – 6.35 (m, 1H), 5.57 (s, 1H), 3.80 (s, 3H), 2.21 (s, 3H), 1.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.1, 171.6, 153.5, 151.1, 132.7, 132.6, 124.7, 124.6, 118.3, 118.2, 115.4, 115.2, 113.5, 113.5, 69.5, 53.8, 24.8, 18.9. HRMS (ESI) Calcd. for $C_{12}H_{15}FNO_3$ [M+H]: 240.1036. Found: 240.1026.

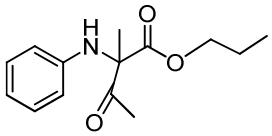
ethyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3k)



Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.3), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (t, J = 7.9 Hz, 4H), 6.73 (t, J = 7.3 Hz, 2H), 6.53 (d, J = 7.8 Hz, 4H), 5.29 (s, 2H), 4.25 (q, J = 7.1 Hz, 4H), 2.20 (s, 6H), 1.68 (s, 6H),

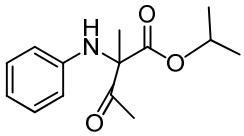
1.23 (t, J = 7.1 Hz, 7H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 171.3, 144.2, 129.6, 118.5, 114.4, 69.8, 62.9, 24.9, 18.8, 14.2. HRMS (ESI) Calcd. for $C_{13}H_{18}NO_3$ [M+H]: 236.1287. Found: 236.1276.

propyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3l)



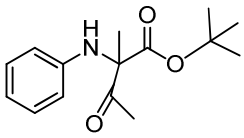
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.3), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (t, J = 7.9 Hz, 2H), 6.73 (t, J = 7.3 Hz, 1H), 6.53 (d, J = 7.8 Hz, 2H), 5.30 (s, 1H), 4.15 (t, J = 6.6 Hz, 2H), 2.20 (s, 3H), 1.69 (s, 3H), 1.68 – 1.55 (m, 2H), 0.87 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8, 171.4, 144.2, 129.5, 118.5, 114.3, 69.9, 68.4, 24.8, 21.9, 18.8, 10.3. HRMS (ESI) Calcd. for $C_{14}H_{20}NO_3$ [M+H]: 250.1443. Found: 250.1431.

isopropyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3m)



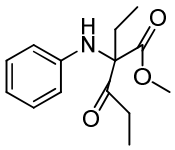
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.3), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (t, J = 8.0 Hz, 2H), 6.72 (t, J = 7.3 Hz, 1H), 6.53 (d, J = 7.7 Hz, 2H), 5.29 (s, 1H), 5.10 (dt, J = 12.5, 6.3 Hz, 1H), 2.18 (s, 3H), 1.66 (s, 3H), 1.21 (dd, J = 33.3, 6.3 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 170.8, 144.3, 129.5, 118.5, 114.4, 70.7, 69.9, 24.8, 21.6, 21.5, 18.7. HRMS (ESI) Calcd. for $C_{14}H_{20}NO_3$ [M+H]: 250.1443. Found: 250.1433.

tert-butyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3n)



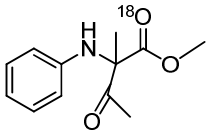
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.5), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.10 (m, 2H), 6.72 (t, J = 7.3 Hz, 1H), 6.53 (dd, J = 8.6, 0.9 Hz, 2H), 2.18 (s, 3H), 1.63 (s, 3H), 1.43 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.3, 170.3, 144.5, 129.6, 118.4, 114.4, 83.8, 70.3, 27.9, 24.9, 18.6. HRMS (ESI) Calcd. for $C_{15}H_{22}NO_3$ [M+H]: 264.1600. Found: 264.1587.

methyl 2-ethyl-3-oxo-2-(phenylamino)pentanoate (3o)



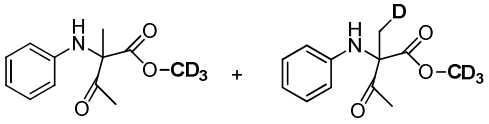
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), colorless oily liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.08 (m, 2H), 6.72 (t, J = 7.3 Hz, 1H), 6.50 (d, J = 7.7 Hz, 2H), 3.77 (s, 3H), 2.51 (dd, J = 50.1, 18.0 Hz, 2H), 2.33 (q, J = 7.4 Hz, 2H), 1.04 (t, J = 7.3 Hz, 3H), 0.66 (t, J = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.8, 171.3, 144.0, 129.5, 118.3, 114.1, 73.5, 53.5, 30.8, 22.9, 8.3, 7.5. HRMS (ESI) Calcd. for $C_{14}H_{20}NO_3$ [M+H]: 250.1443. Found: 250.1431.

(^{18}O) methyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3p)



Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), oily brown liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (t, J = 7.8 Hz, 2H), 6.74 (t, J = 7.2 Hz, 1H), 6.52 (d, J = 8.4 Hz, 2H), 3.79 (s, 3H), 2.20 (s, 3H), 1.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 171.8, 144.0, 129.5, 118.5, 114.3, 69.8, 53.7, 24.8, 18.8. HRMS (ESI) Calcd. for $C_{12}H_{16}NO_2{}^{18}O$ [M+H]: 224.1173. Found: 224.1164.

Deuterated-methyl 2-methyl-3-oxo-2-(phenylamino)butanoate (3r) & Deuterated-methyl 2-mono-deuterated methyl-3-oxo-2-(phenylamino)butanoate (3s)



3r **3s**

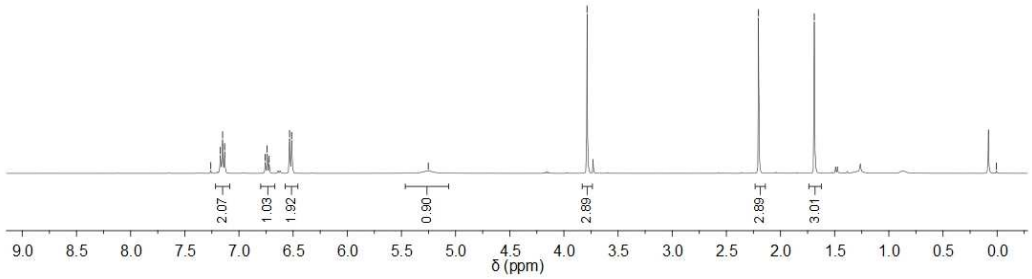
Isolated by column chromatography on silica gel (eluting with hexane/ethyl acetate = 16:1, R_f = 0.4), oily brown liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (t, J = 7.9 Hz, 2H), 6.74 (t, J = 7.3 Hz, 1H), 6.52 (d, J = 8.1 Hz, 2H), 5.28 (s, 1H), 2.20 (s, 3H), 1.71 – 1.63 (m, 2.36H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 171.9, 144.1, 129.6, 118.6, 114.3, 69.8, 69.7 (t, J = 4.4 Hz), 52.8 (m), 24.8, 18.8, 18.5 (t, J = 20.0 Hz). HRMS (ESI) Calcd. for $C_{12}H_{13}D_3NO_3$ [**3r**+H]: 225.1318. Found: 225.1308, for $C_{12}H_{12}D_4NO_3$ [**3s**+H]: 226.1381. Found: 226.1362.

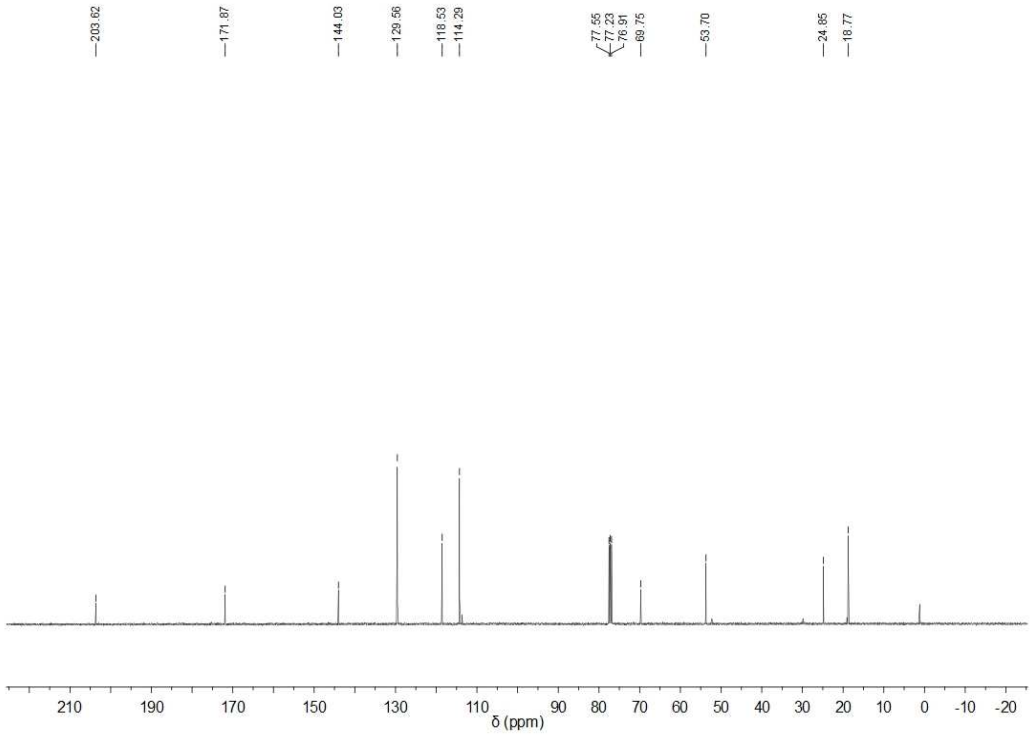
Reference

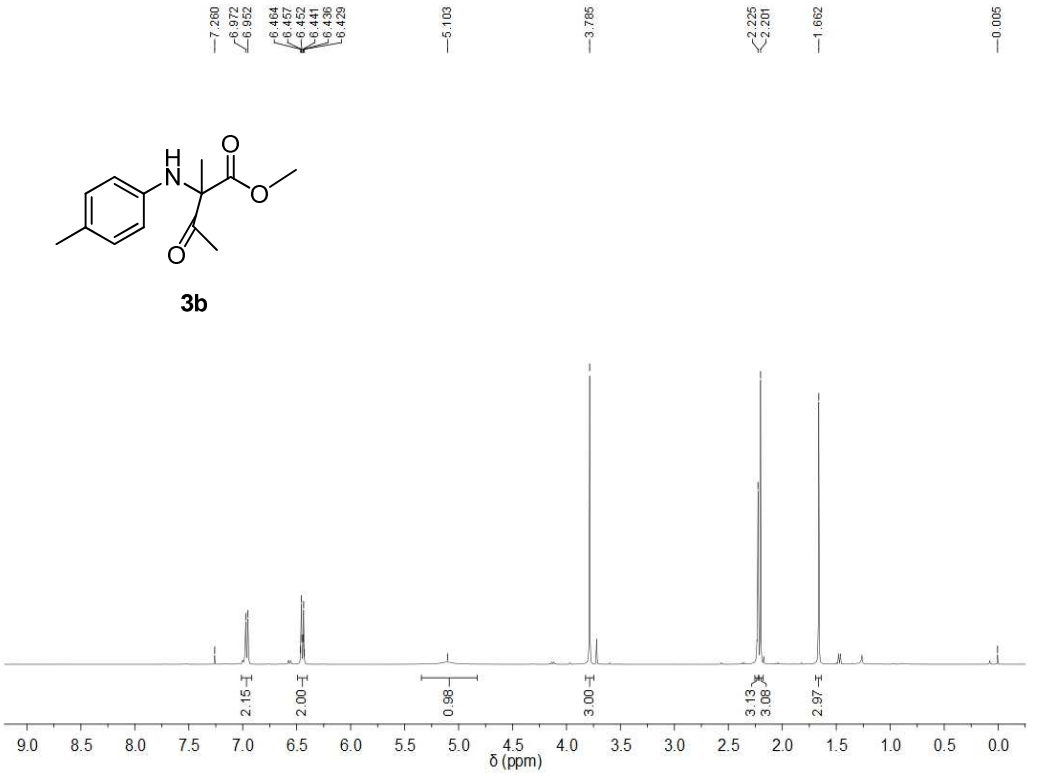
[1]. Bartoli, G.; Bosco, M.; Locatelli, M.; Marcantoni, E.; Melchiorre, P.; Sambri, L. *Synlett* **2004**, *2004*, 239.

3. ^{1}H and ^{13}C NMR

3a



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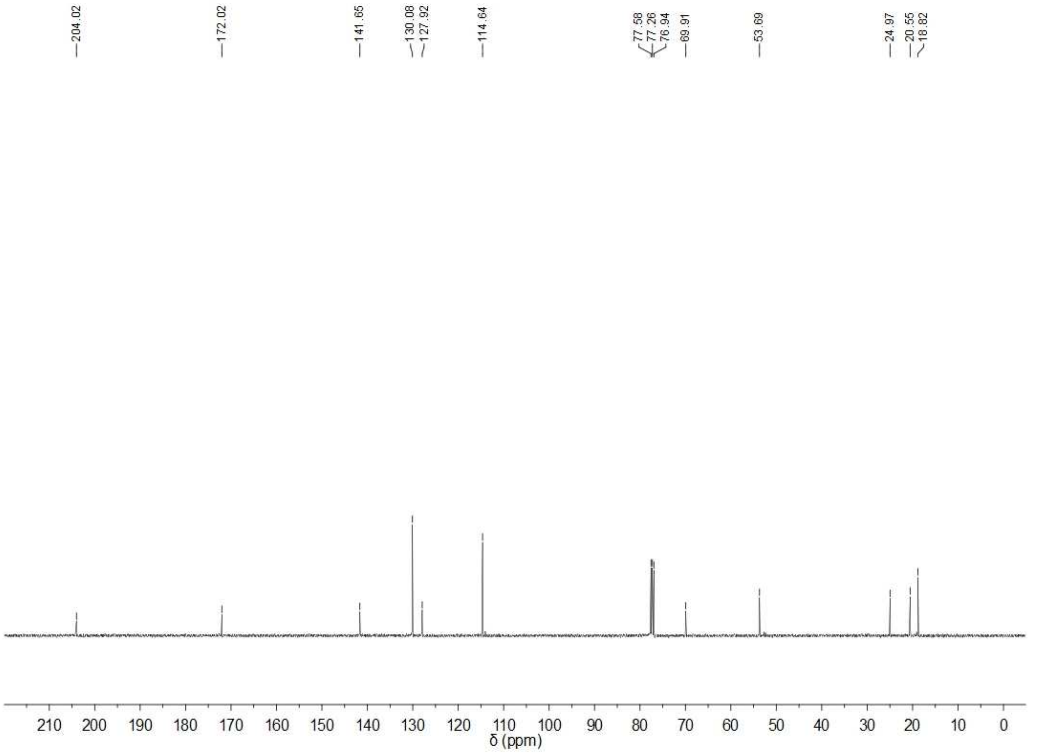
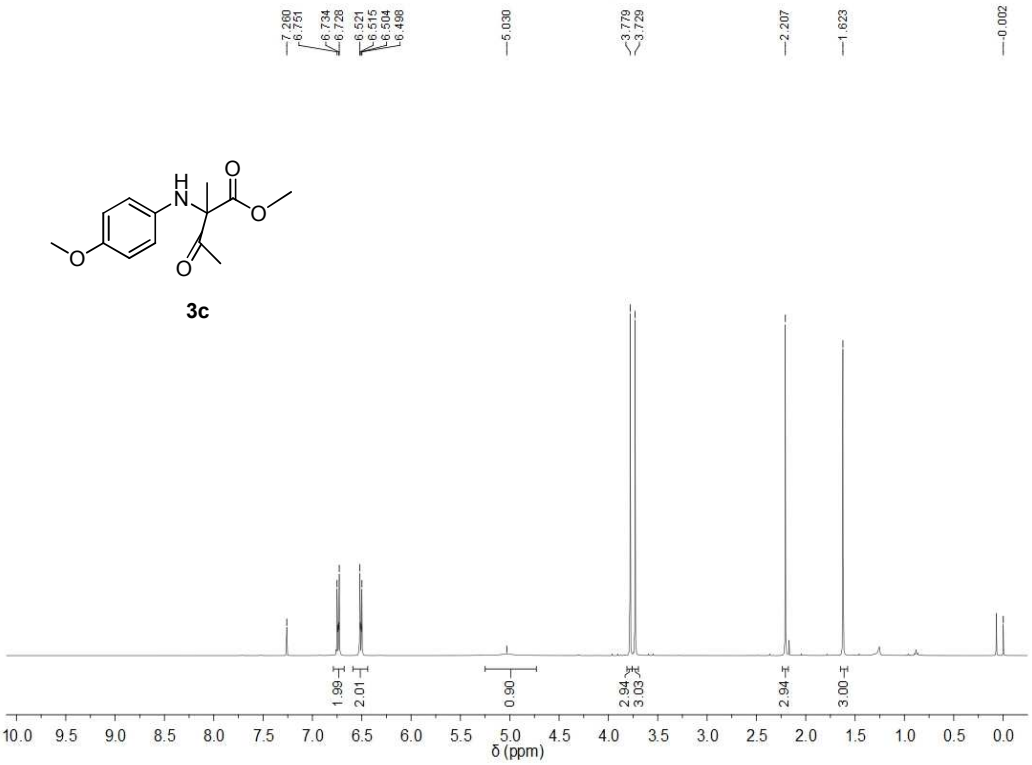
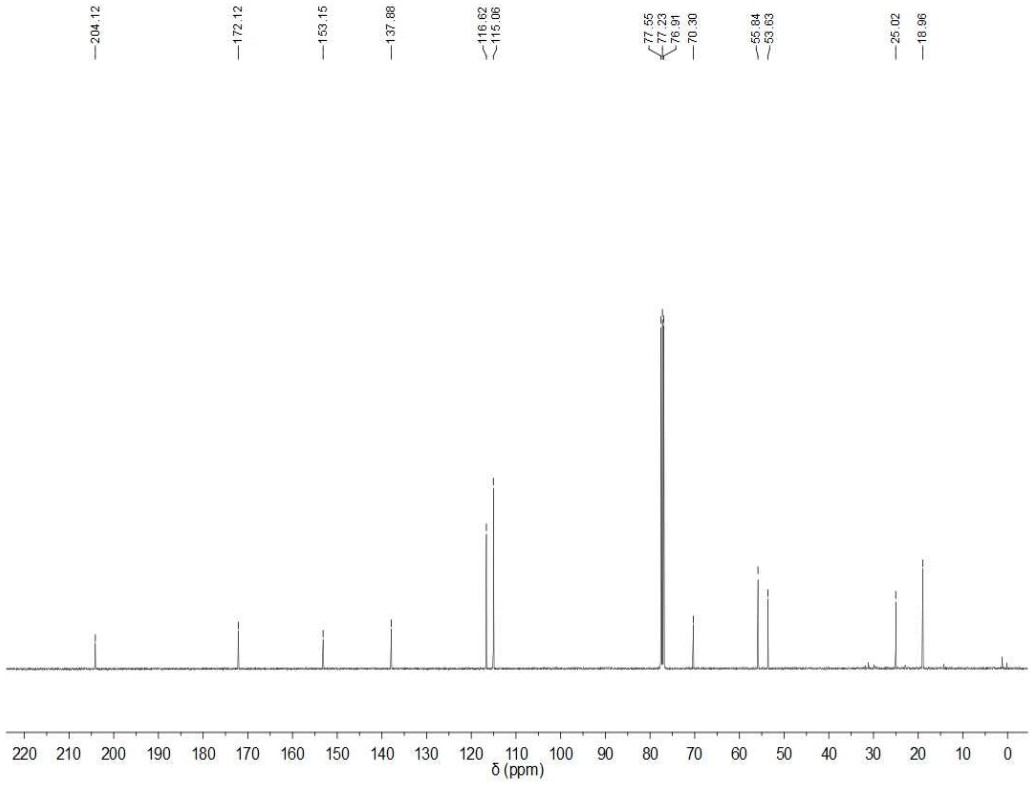
6.972
6.952
6.464
6.457
6.452
6.441
6.436
6.429
5.103
3.785
2.225
2.201
1.662
0.005
2.15
2.00
0.98
3.00
3.13
3.08
2.97
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)

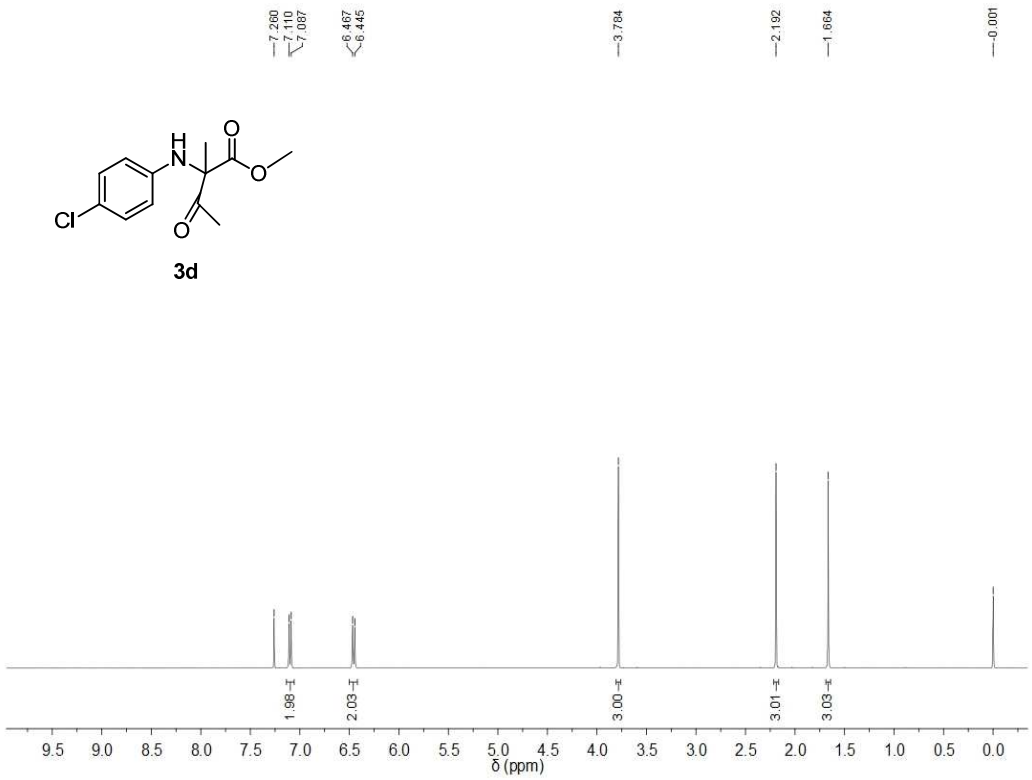
3b


204.02
172.02
141.65
130.08
127.92
114.64
77.58
77.26
76.94
69.91
53.69
24.97
20.55
18.82
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

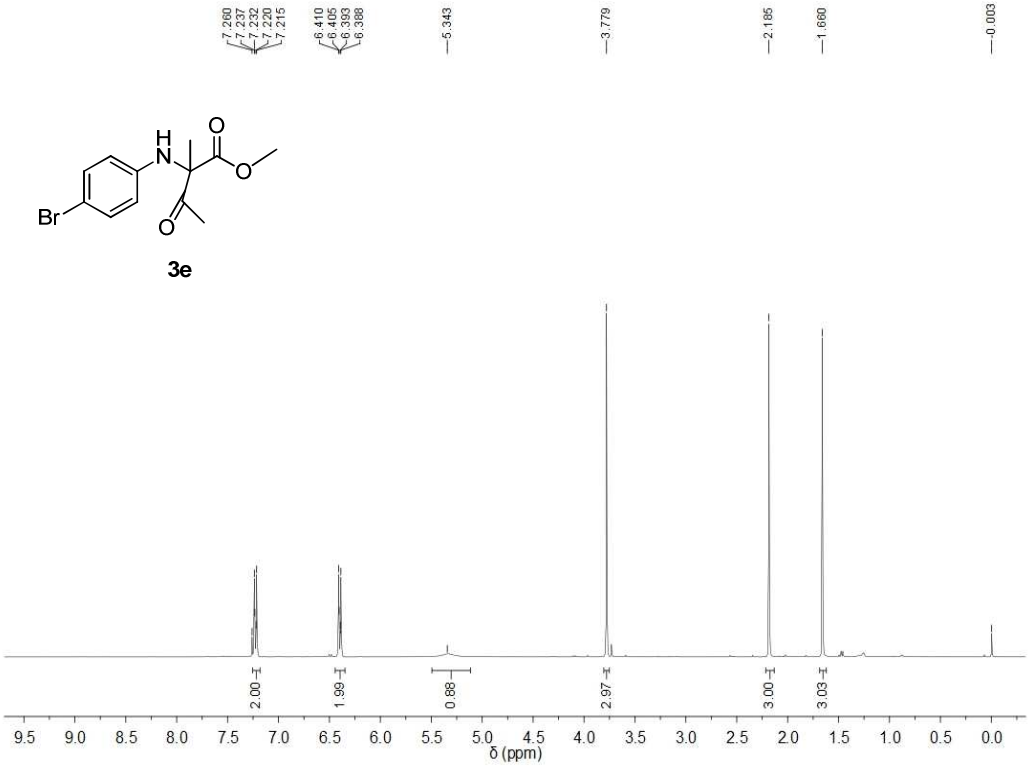

7.260
6.751
6.734
6.728
6.521
6.515
6.504
6.498
5.030
3.779
3.729
2.207
1.623
0.002
1.99
2.01
0.90
2.94
3.03
2.94
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)

3c

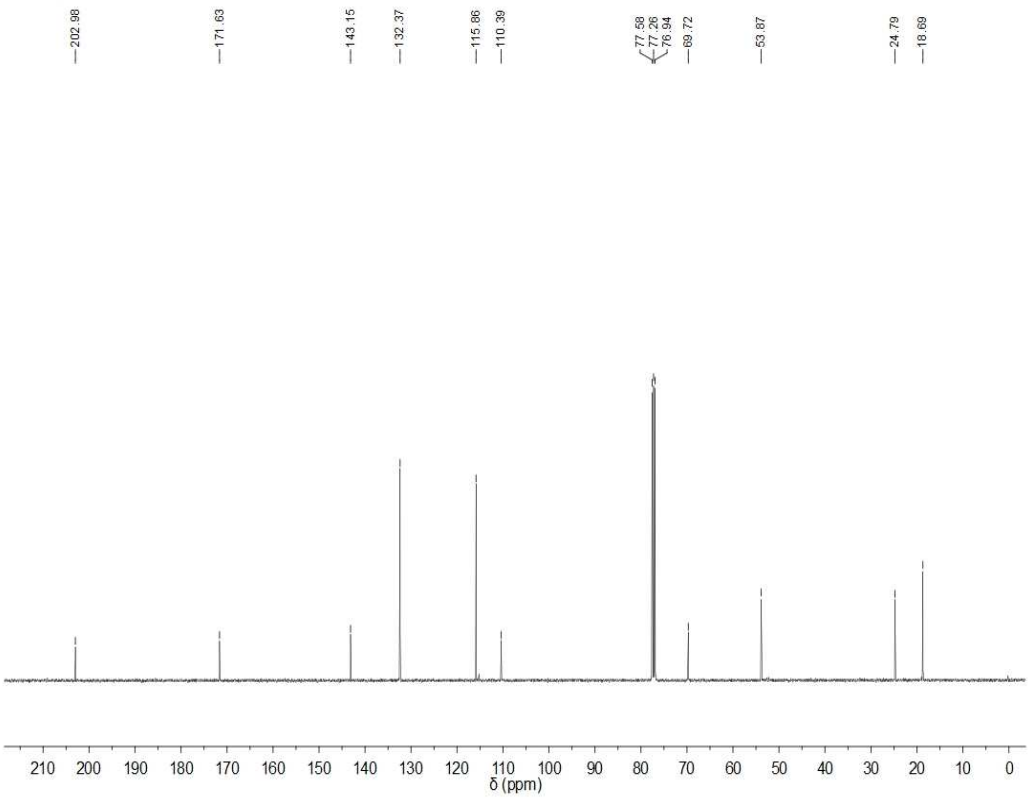

204.12
172.12
153.15
137.88
116.62
115.06
77.55
77.23
76.91
70.30
55.84
53.63
25.02
18.96
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

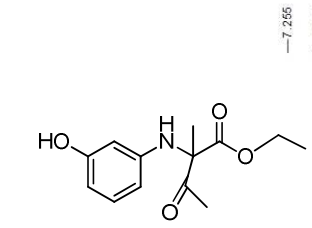

7.260
7.110
7.097
6.467
6.445
3.784
2.192
1.664
0.001
Cl
H
N
O
O
O
1.98
2.03
3.00
3.01
3.03
δ (ppm)
203.07
171.68
142.68
129.51
123.43
115.49
77.58
77.00
76.94
69.82
53.87
24.83
18.74
δ (ppm)

3d


7.260
7.237
7.232
7.220
7.215
6.410
6.405
6.393
6.388
5.343
3.779
2.185
1.660
0.003
Br
2.00
1.99
0.88
2.97
3.00
3.03
δ (ppm)

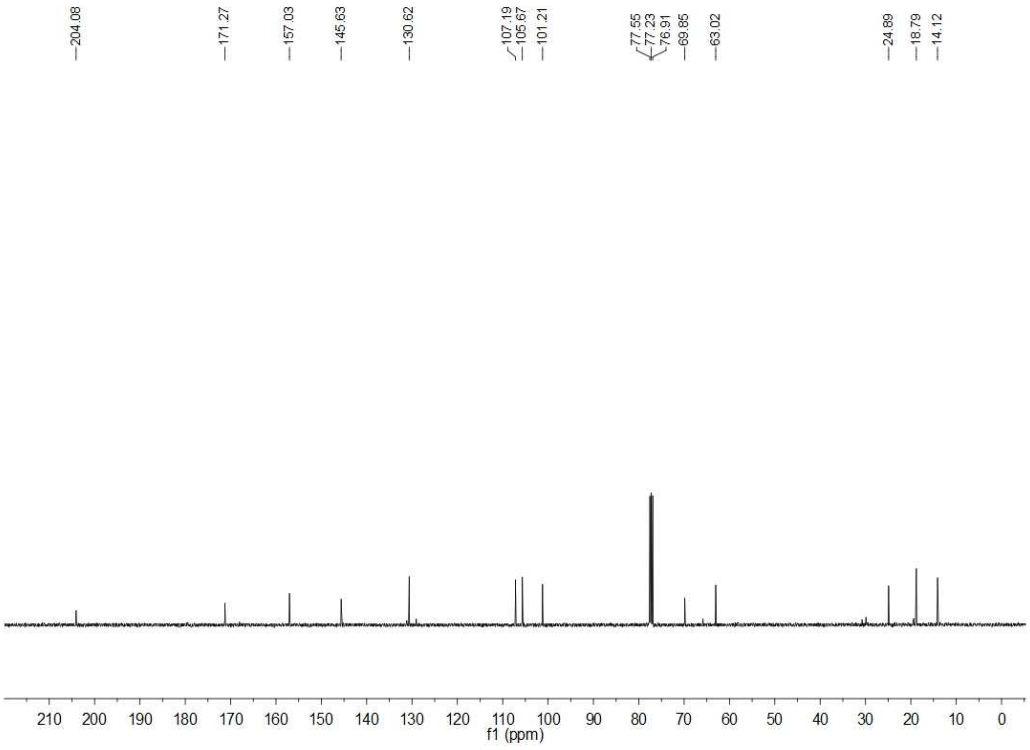
3e

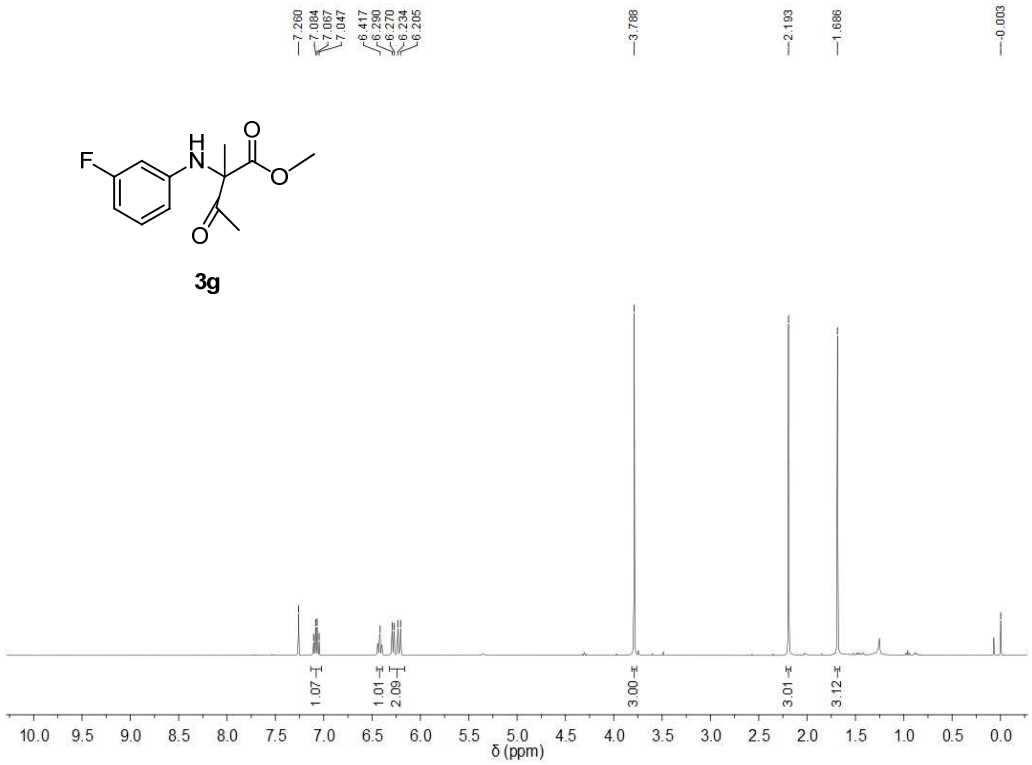

202.98
171.63
143.15
132.37
115.86
110.39
77.58
77.26
76.94
69.72
53.87
24.79
18.69
δ (ppm)



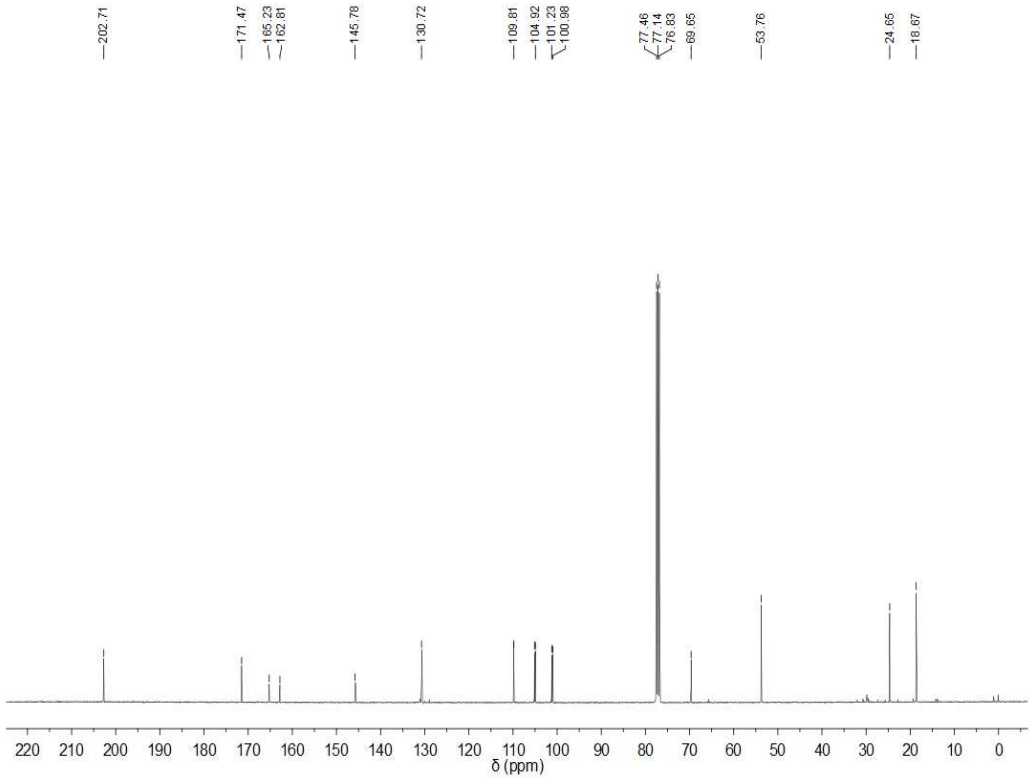
3f

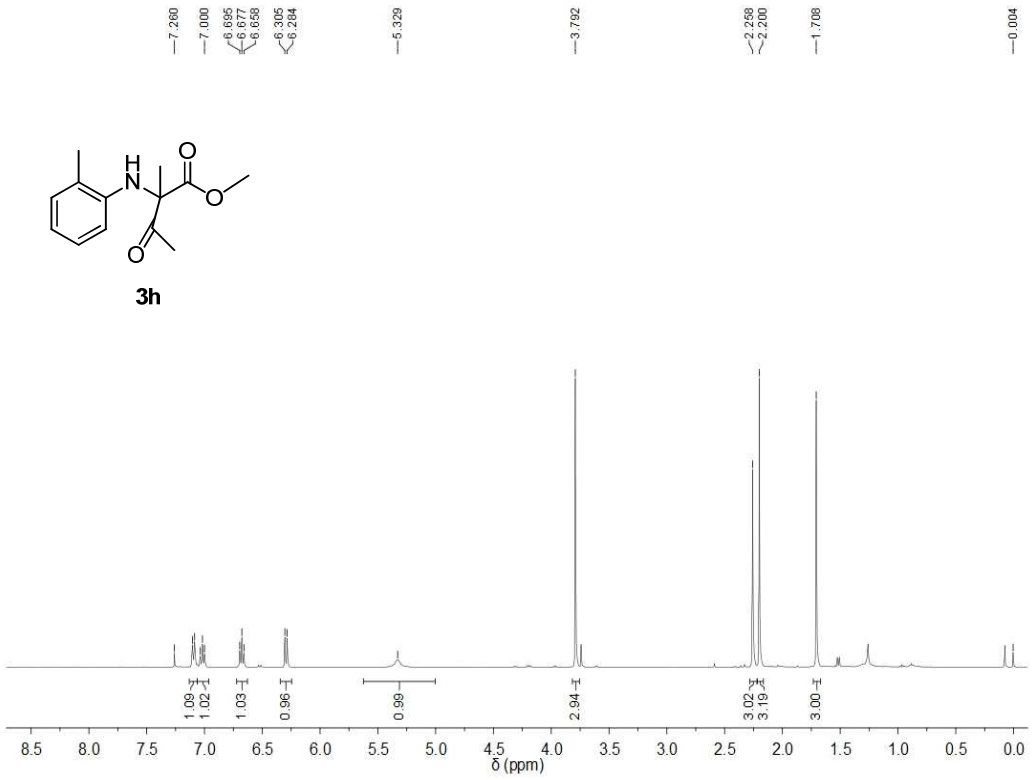
7.255
6.984
6.964
6.234
6.210
6.119
6.115
6.099
6.095
6.019
5.303
4.274
4.257
4.239
4.221
2.185
1.670
1.245
1.227
1.209
0.009
1.00
1.04
1.01
0.96
1.37
1.96
3.02
2.96
3.46
f1 (ppm)


204.08
171.27
157.03
145.63
130.62
107.19
105.67
101.21
77.55
77.23
76.91
69.85
63.02
24.89
18.79
14.12
f1 (ppm)

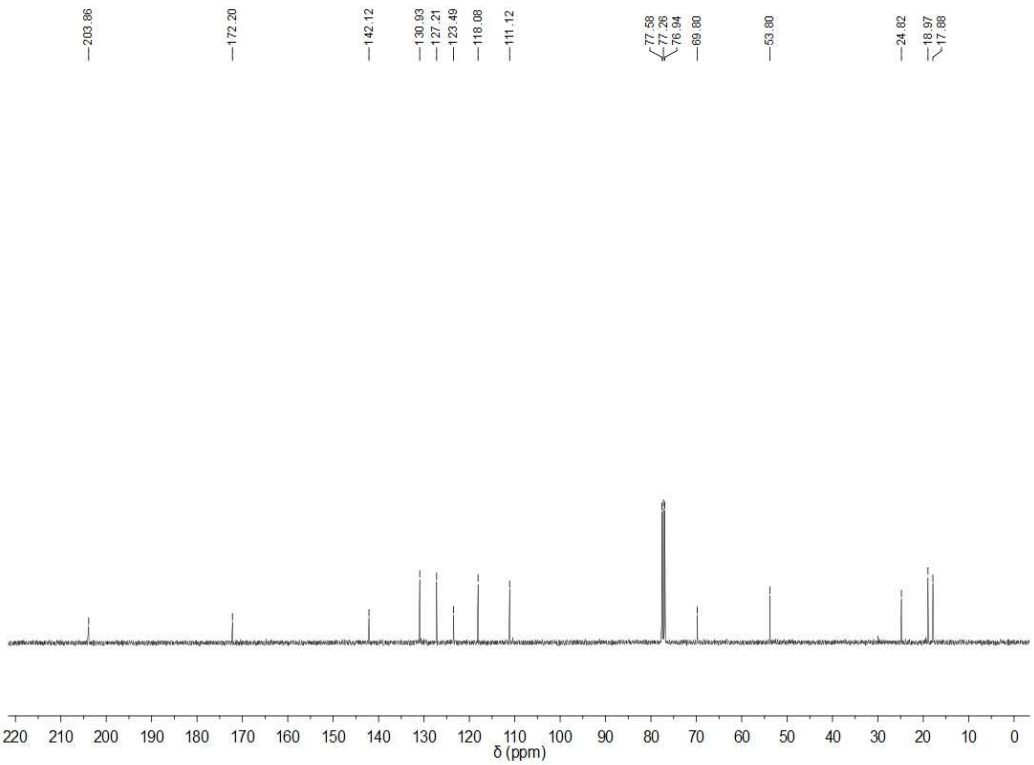

7.260
7.084
7.067
7.047
6.417
6.290
6.270
6.234
6.205
3.788
2.193
1.686
0.003
F
H
N
O
O
O
1.07
1.01
2.09
3.00
3.01
3.12
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)

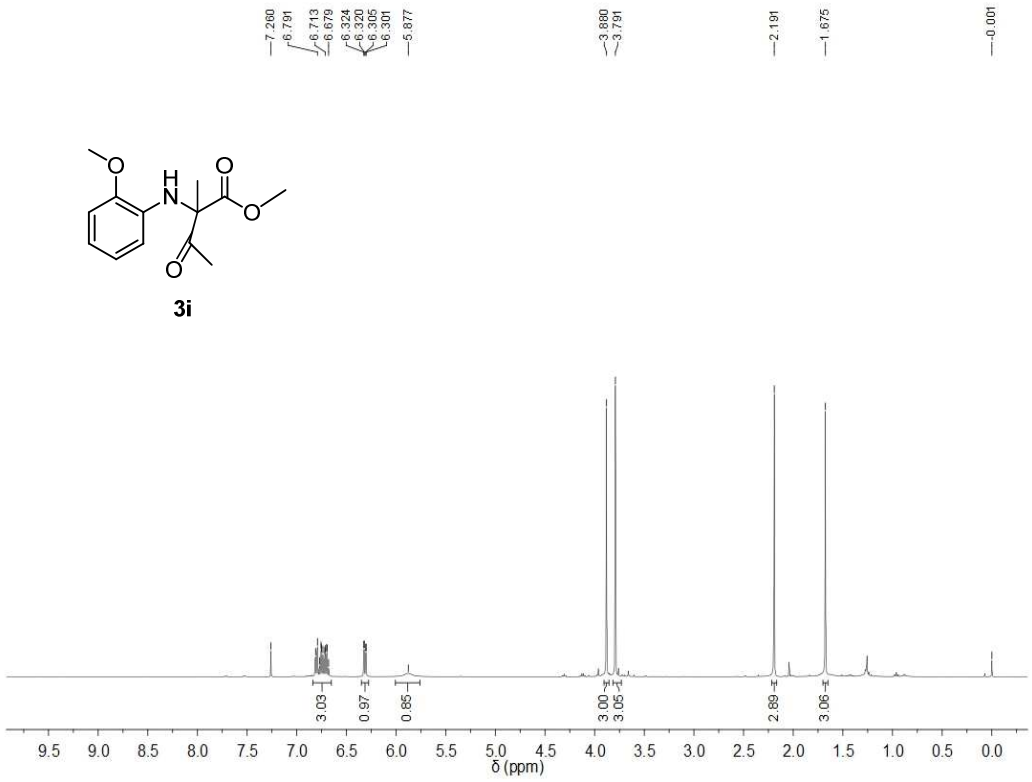
3g


202.71
171.47
165.23
162.81
145.78
130.72
109.81
104.92
101.23
100.98
77.46
77.14
76.83
69.65
53.76
24.65
18.67
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

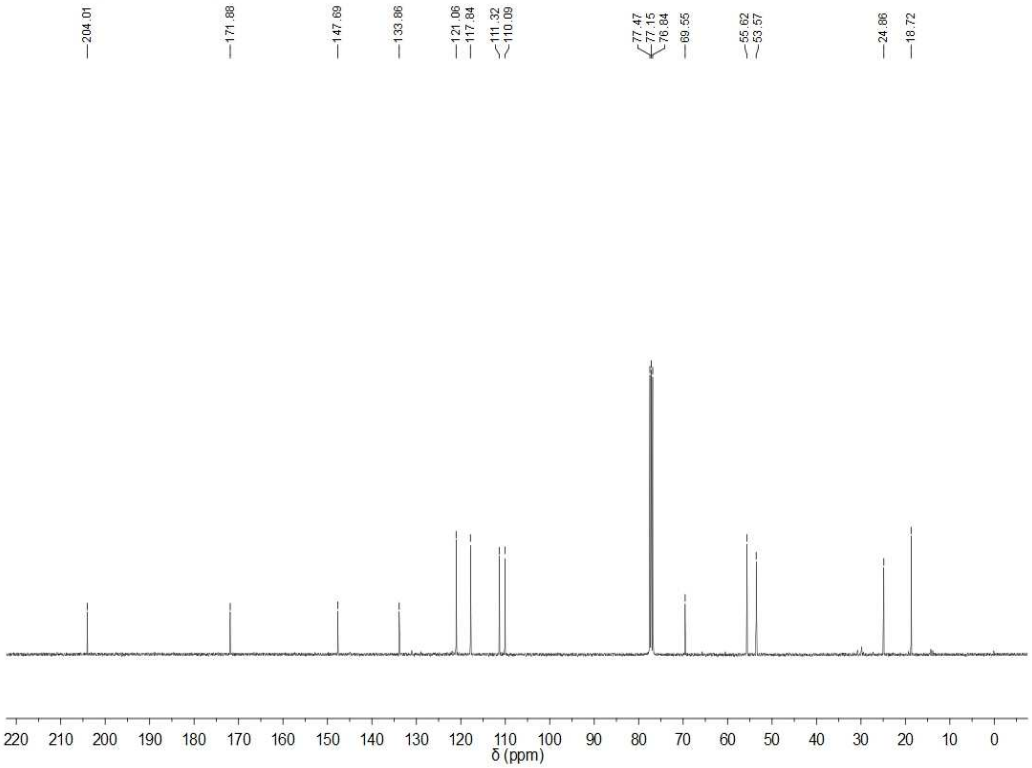

7.260
7.000
6.695
6.677
6.658
6.305
6.284
5.329
3.792
2.258
2.200
1.706
0.004
1.09
1.02
1.03
0.96
0.99
2.94
3.02
3.19
3.00
δ (ppm)

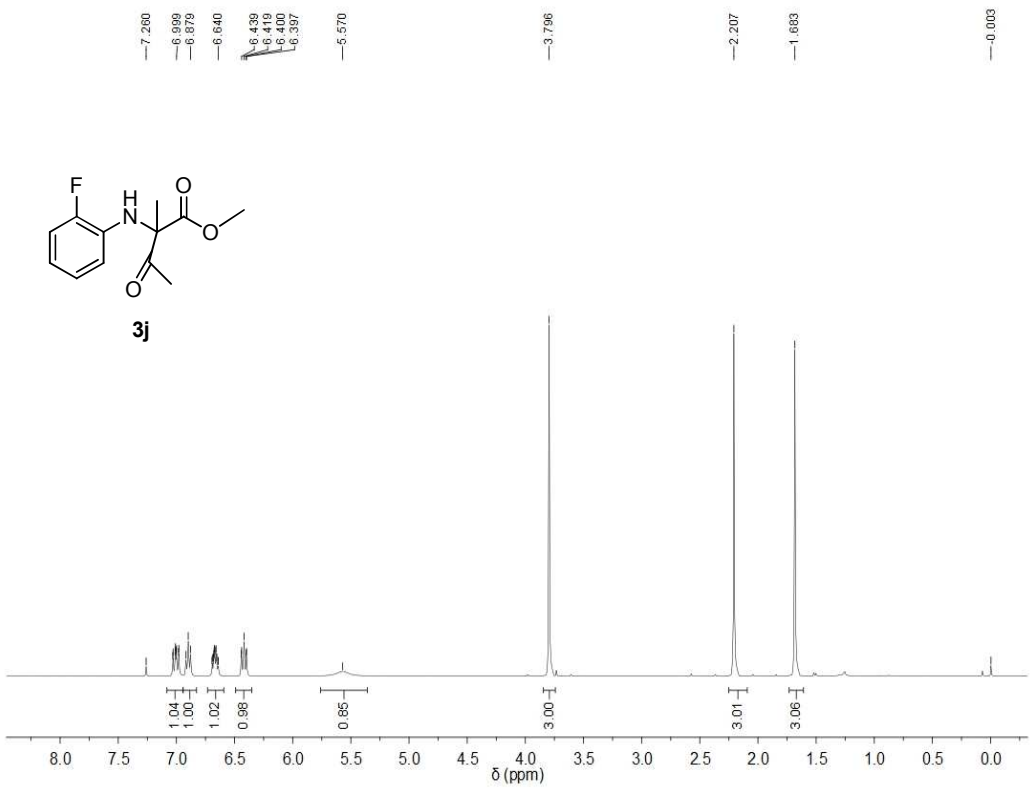
3h


203.86
172.20
142.12
130.93
127.21
123.49
118.08
111.12
77.58
77.26
76.94
69.80
53.80
24.82
18.97
17.88
δ (ppm)


7.260
6.791
6.713
6.679
6.324
6.320
6.305
6.301
5.877
3.880
3.791
2.191
1.675
0.001
3.03
0.97
0.85
3.00
3.05
2.89
3.06
δ (ppm)

3i


204.01
171.88
147.69
133.86
121.06
117.84
111.32
110.09
77.47
77.15
76.84
69.55
55.62
53.57
24.88
18.72
δ (ppm)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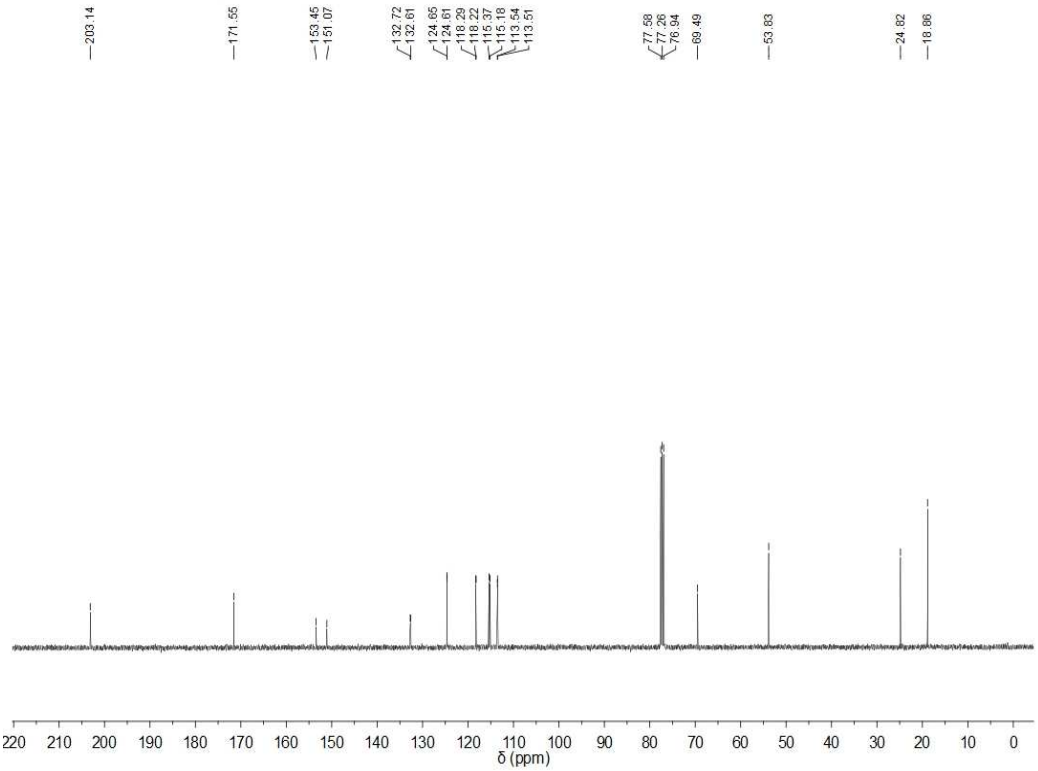
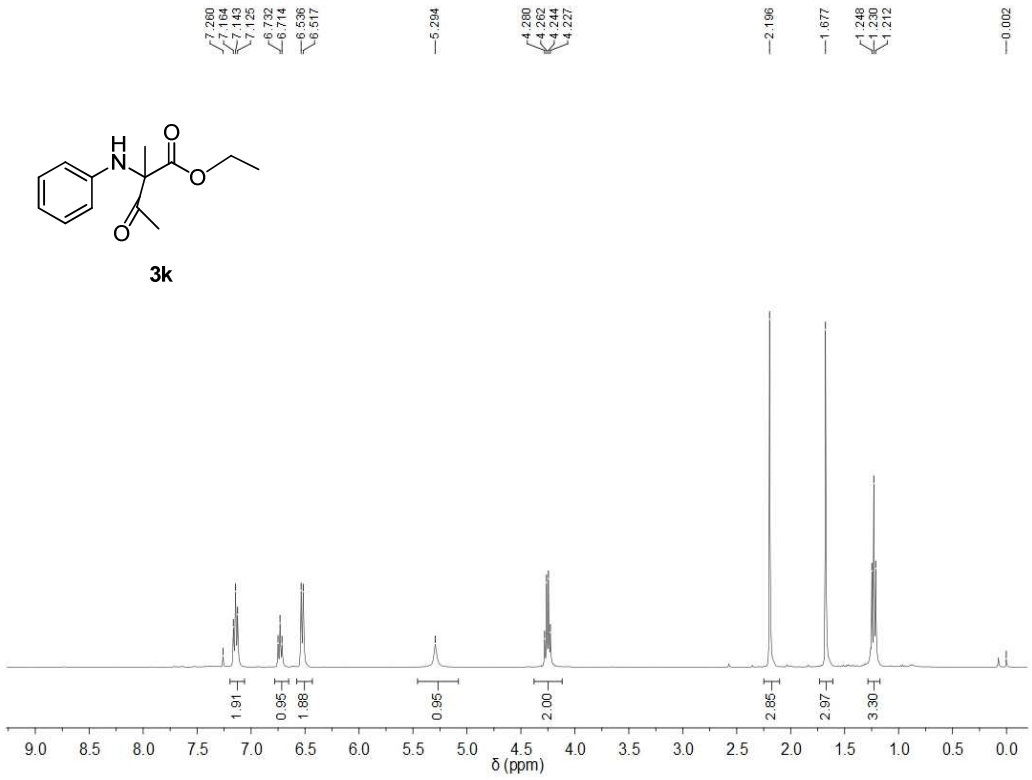
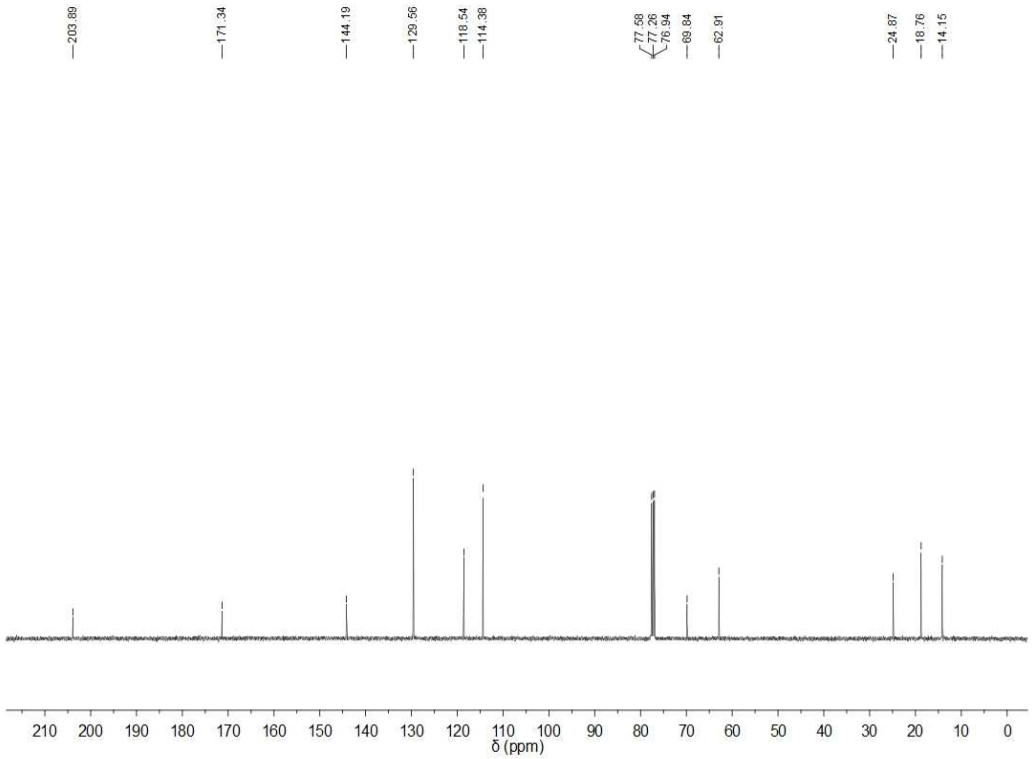
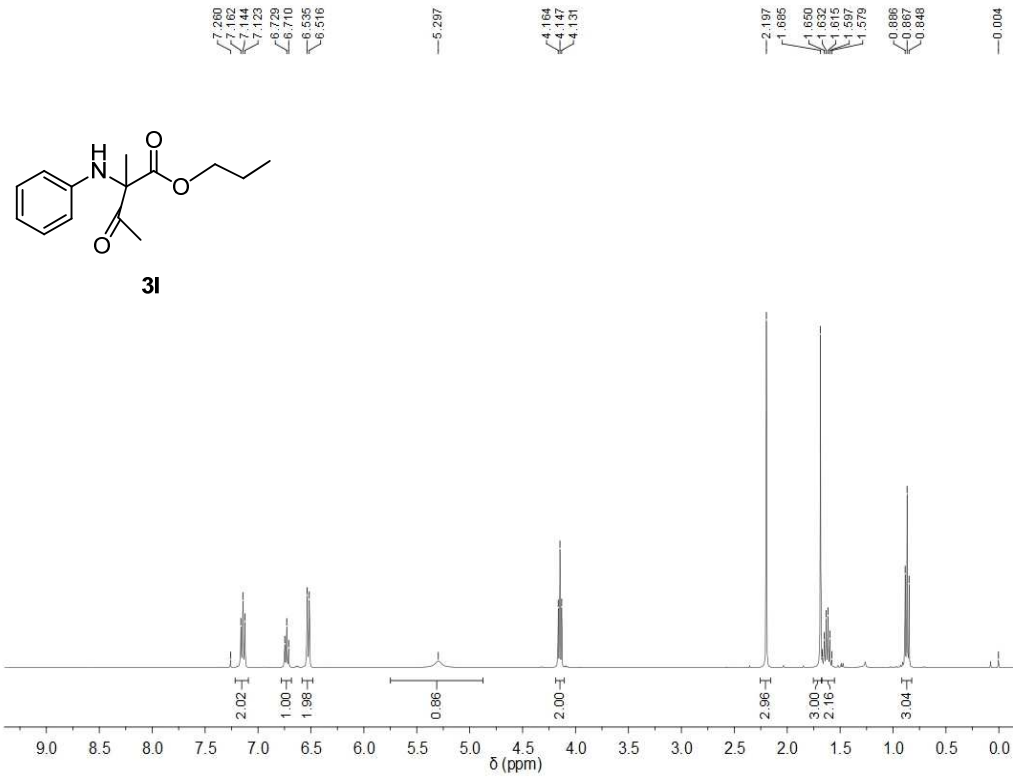
6.999
6.879
6.640
6.439
6.419
6.400
6.397
5.570
3.796
2.207
1.683
0.003
F
H
N
O
O
O
3j
1.04
1.00
1.02
0.98
0.85
3.00
3.01
3.06
δ (ppm)


203.14
171.55
153.45
151.07
132.72
132.61
124.65
124.61
118.29
118.22
115.37
115.18
113.54
113.51
77.58
77.26
76.94
69.49
53.83
24.82
18.86
δ (ppm)


7.260
7.164
7.143
7.125
6.732
6.714
6.536
6.517
5.294
4.280
4.262
4.244
4.227
2.196
1.677
1.248
1.230
1.212
0.002
1.91
0.95
1.88
0.95
2.00
2.85
2.97
3.30
δ (ppm)

3k


203.89
171.34
144.19
129.56
118.54
114.38
77.58
77.26
76.94
69.84
62.91
24.87
18.76
14.15
δ (ppm)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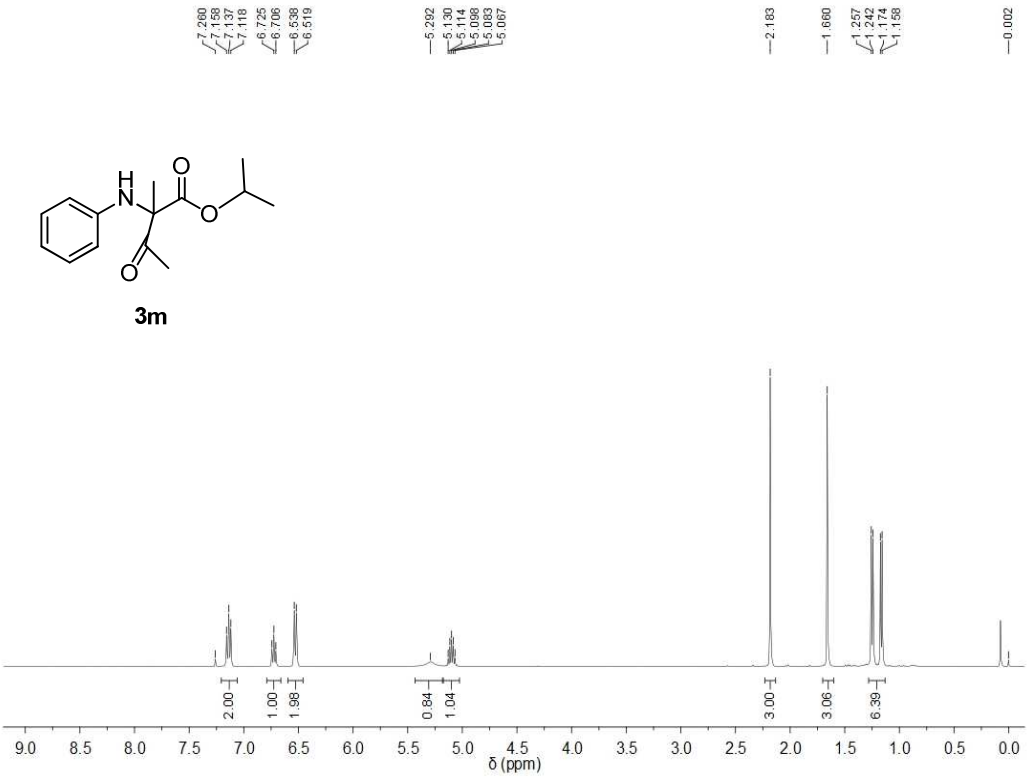
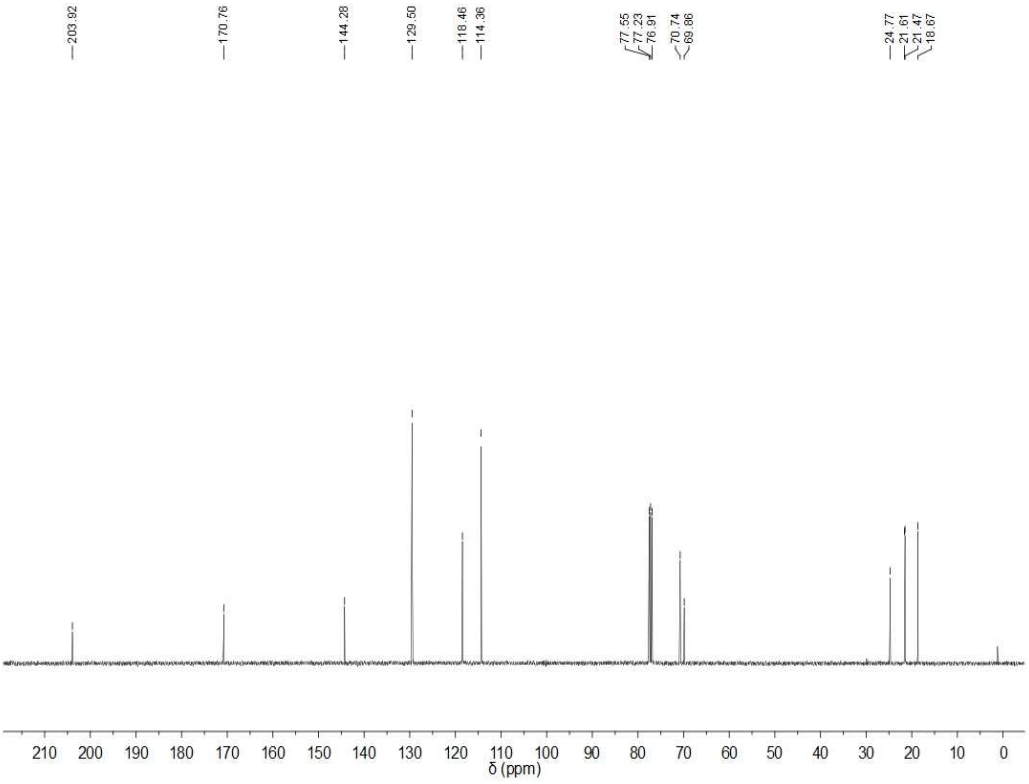
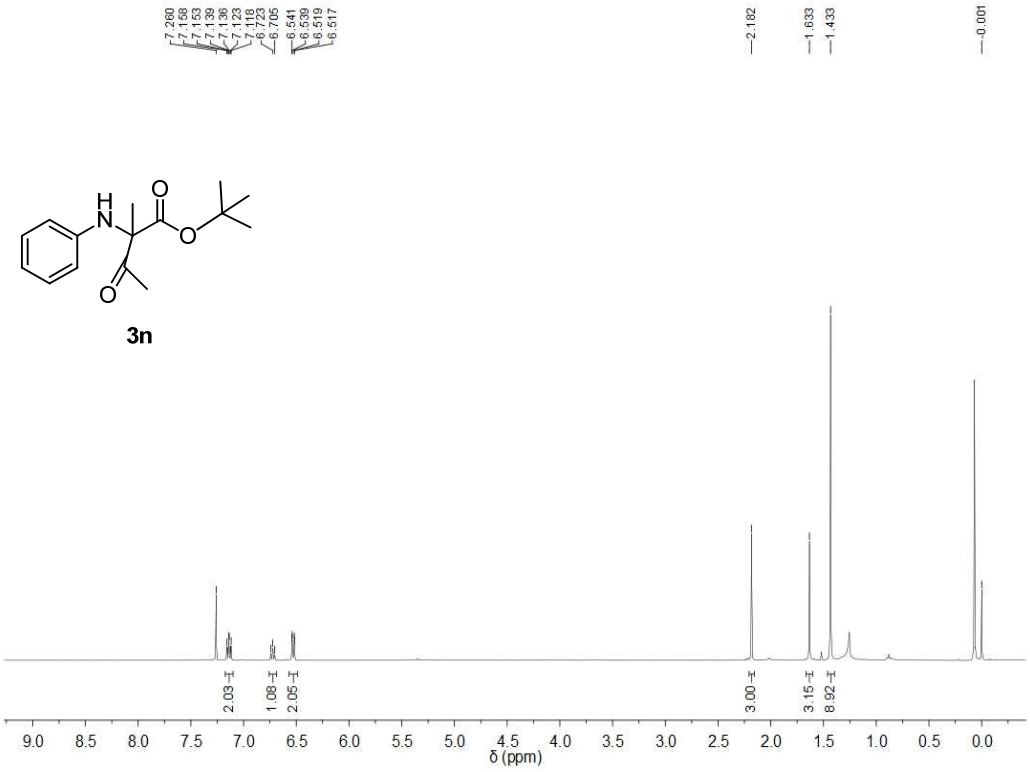
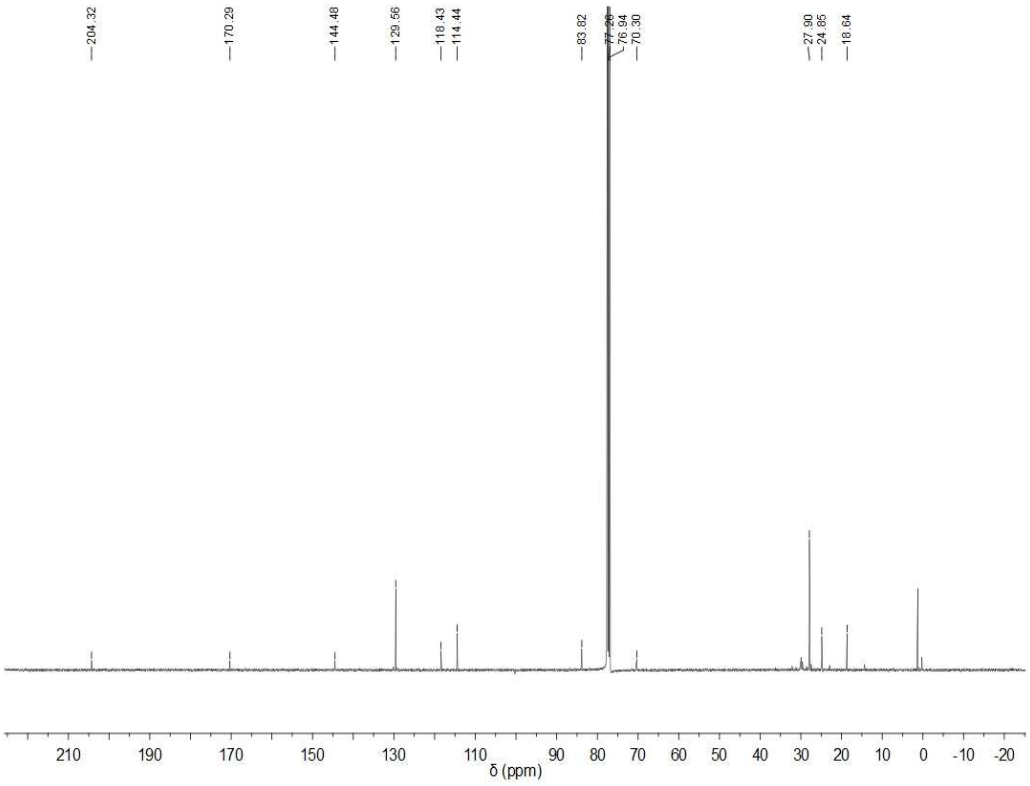
7.162
7.144
7.123
6.729
6.710
6.535
6.516
5.297
4.164
4.147
4.131
2.197
1.685
1.650
1.632
1.615
1.597
1.579
0.886
0.867
0.848
0.004
3I
2.02
1.00
1.98
0.86
2.00
2.96
3.00
2.16
3.04
δ (ppm)
203.801
171.375
144.173
129.519
118.484
114.310
77.548
77.230
76.913
69.855
68.400
24.845
21.956
18.769
10.290
f1 (ppm)

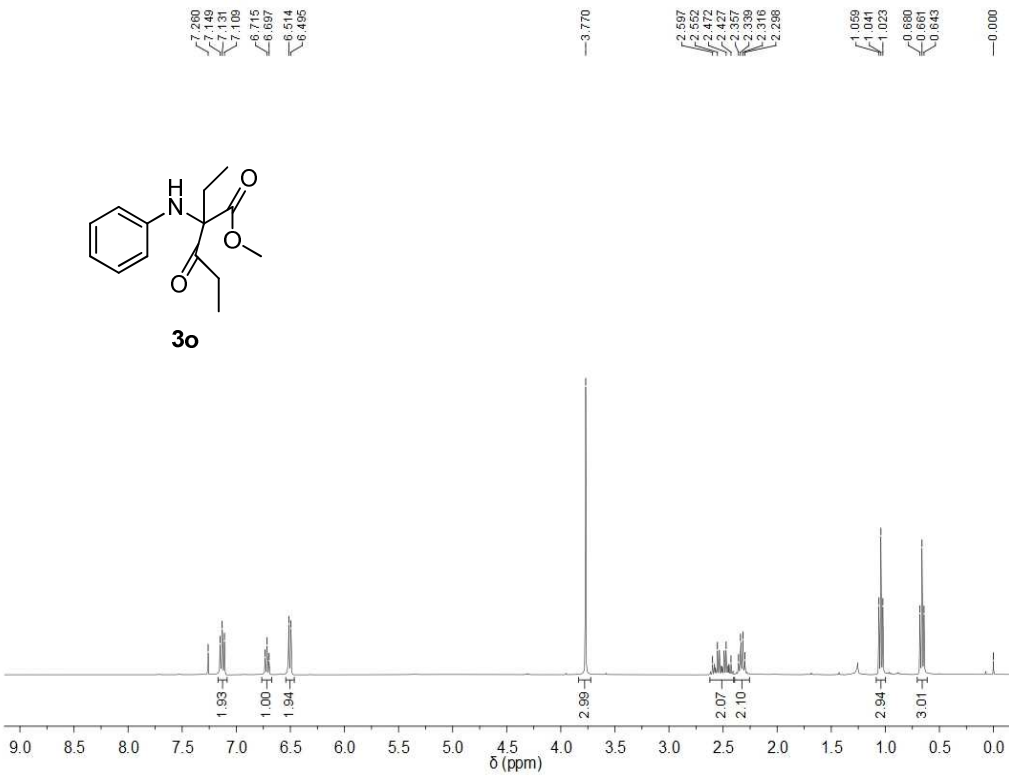

7.280
7.158
7.137
7.118
6.725
6.706
6.538
6.519
5.292
5.130
5.114
5.098
5.083
5.067
2.183
1.660
1.257
1.242
1.174
1.158
0.002
2.00
1.00
1.98
0.84
1.04
3.00
3.06
6.39
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)

3m

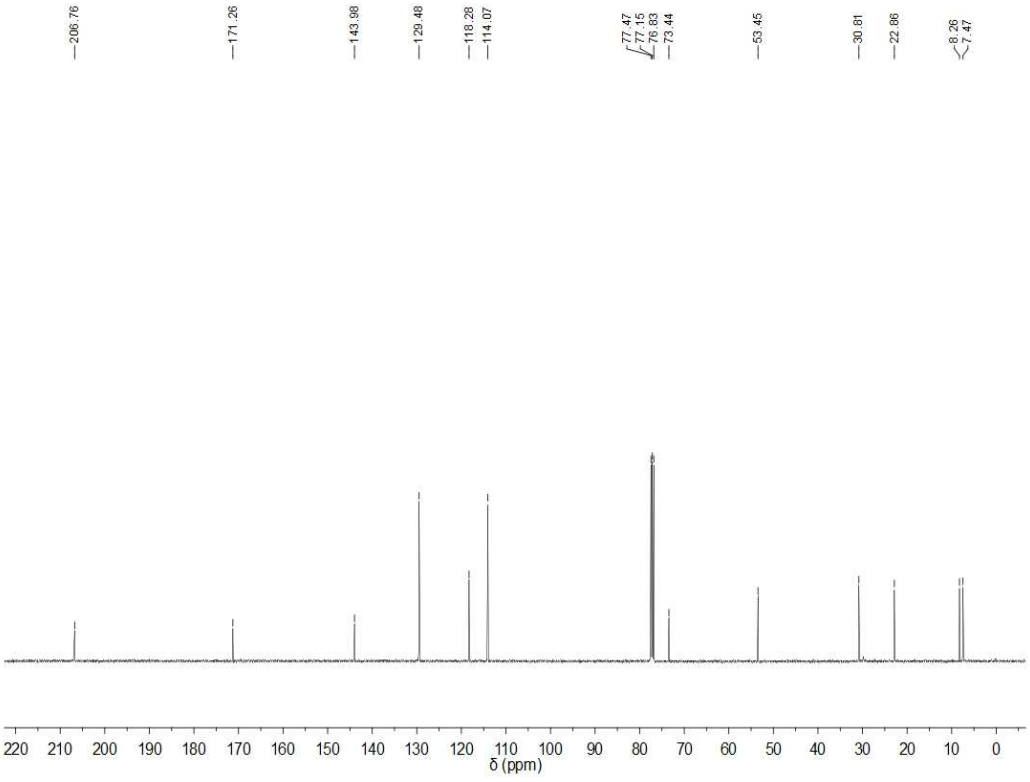

203.92
170.76
144.28
129.50
118.46
114.36
77.55
77.23
76.91
70.74
69.86
24.77
21.61
21.47
18.67
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

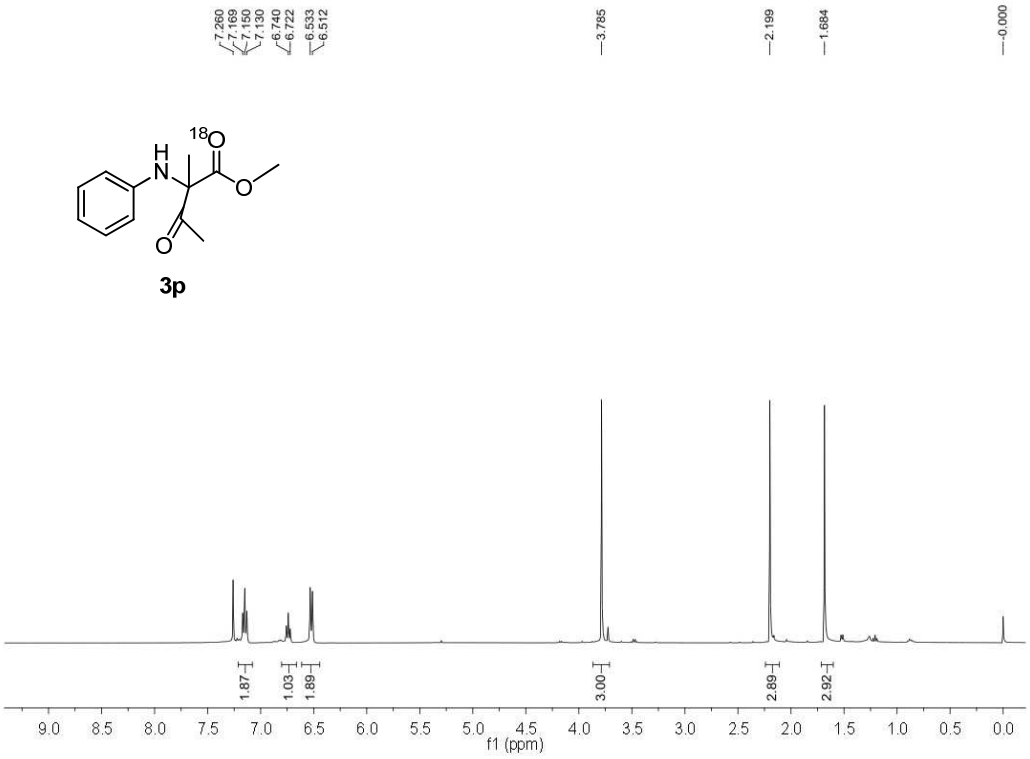

7.260
7.158
7.153
7.139
7.136
7.123
7.118
6.723
6.705
6.541
6.539
6.519
6.517
2.162
1.633
1.433
0.001
3n
2.03
1.08
2.05
3.00
3.15
8.92
δ (ppm)


204.32
170.29
144.48
129.56
119.43
114.44
83.82
77.26
76.94
70.30
27.90
24.85
18.64
δ (ppm)


7.260
7.149
7.131
7.109
6.715
6.697
6.514
6.495
3.770
2.597
2.552
2.472
2.457
2.357
2.339
2.316
2.298
1.059
1.041
1.023
0.680
0.661
0.643
0.000
1.93
1.00
1.94
2.99
2.07
2.10
2.94
3.01
δ (ppm)

3o


206.76
171.26
143.98
129.48
118.28
114.07
77.47
77.15
76.83
73.44
53.45
30.81
22.86
8.26
7.47
δ (ppm)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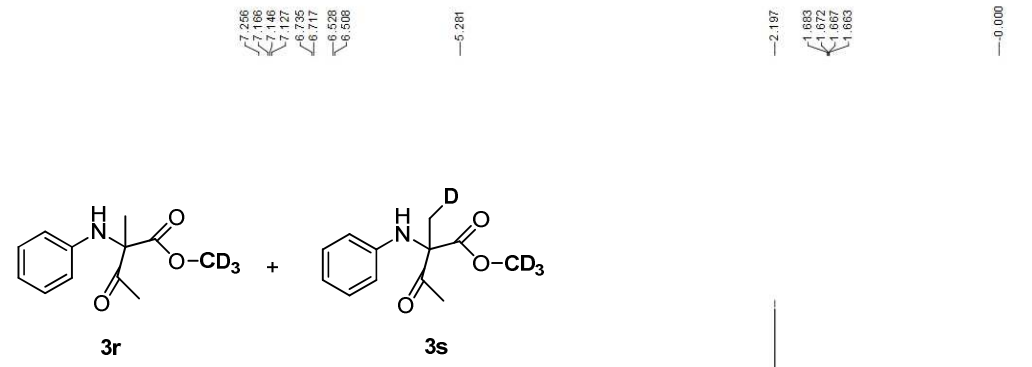
7.169
7.150
7.130
6.740
6.722
6.533
6.512
3.785
2.199
1.684
0.000
18O
H
N
O
O
O
3p
1.87
1.03
1.89
3.00
2.89
2.92
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
203.56
171.84
144.03
129.53
118.53
114.30
77.55
77.23
76.91
69.75
53.65
24.80
18.77
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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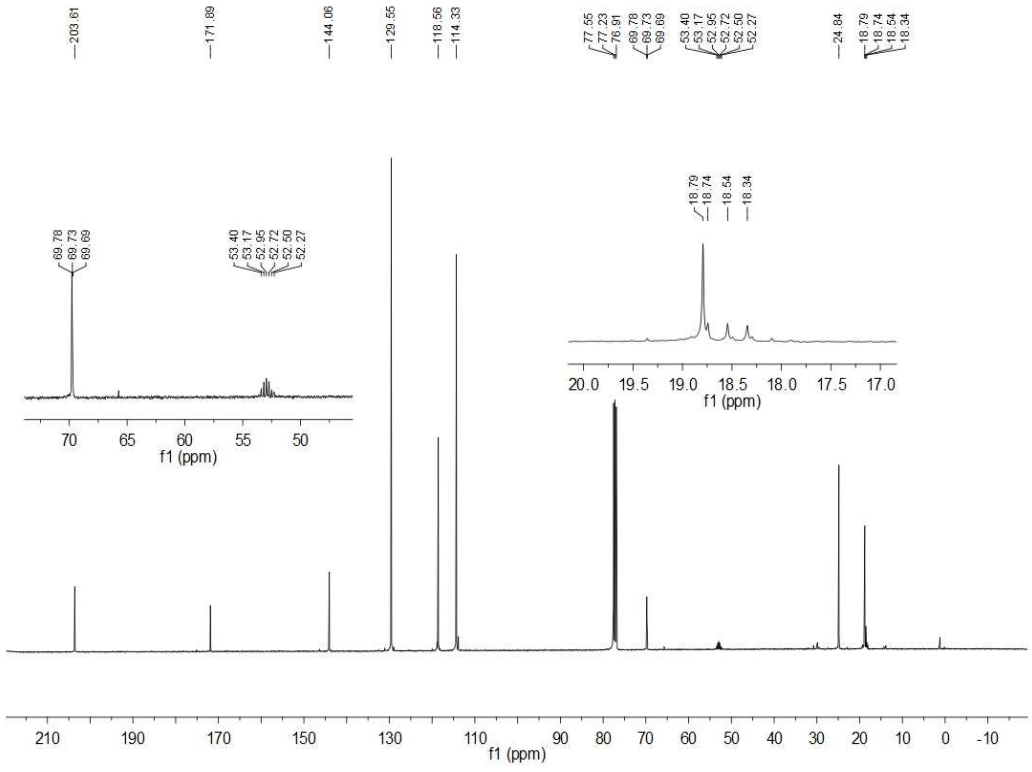
7.166
7.146
7.127
6.735
6.717
6.528
6.508
5.281
2.197
1.693
1.672
1.667
1.663
0.000
3r
3s
2.11
1.05
2.02
0.96
3.00
2.36
f1 (ppm)


203.61
171.89
144.06
129.55
118.56
114.33
77.55
77.23
76.91
69.78
69.73
69.69
53.40
53.17
52.95
52.72
52.50
52.27
24.84
18.79
18.74
18.54
18.34
f1 (ppm)