Supporting Information for:

TBHP/I_2-Promoted Oxidative Coupling of Acetophenones and Amines at Room Temperature under Metal-Free and Solvent-Free Conditions for the Synthesis of α-Ketoamides

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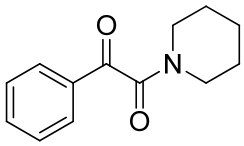
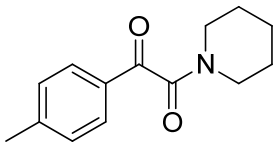
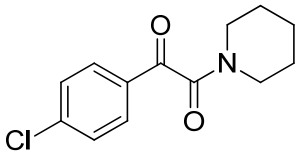
1. Characterization data for the products



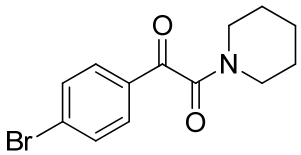
1-Phenyl-2-(piperidin-1-yl)ethane-1,2-dione (3a): Yellowish solid.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95 (d, *J* = 7.6 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 3.71 (s, 2H), 3.29 (t, *J* =5 .2 Hz, 2H), 1.70 (br, s, 4H), 1.55 (br, s 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.86, 165.34, 134.61, 133.04, 129.43, 128.91, 46.93, 42.04, 26.05, 25.31, 24.21.



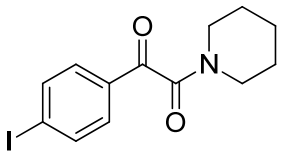
1-(Piperidin-1-yl)-2-(*p*-tolyl)ethane-1,2-dione (3b): Yellowish solid.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 3.68 (br, s, 2H), 3.26 (t, *J* = 5.6 Hz, 2H), 2.42 (s, 3H), 1.68 (t, *J* = 2.8 Hz, 4H), 1.53 (d, *J* = 5.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.64, 165.55, 145.80, 130.74, 129.63, 129.58, 46.93, 41.99, 26.10, 25.36, 24.29, 21.80.



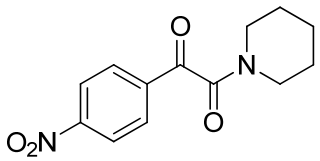
1-(4-Chlorophenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3c): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 3.67–3.65 (m, 2H), 3.25 (t, *J* = 5.6 Hz, 2H), 1.66 (t, *J* = 3.2 Hz, 4H), 1.53–1.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.39, 164.76, 141.06, 131.52, 130.76, 129.25, 46.89, 42.07, 26.09, 25.29, 24.17.



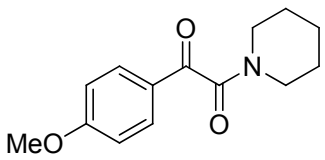
1-(4-Bromophenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3d): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.79 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.8 Hz, 2H), 3.68–3.65 (m, 2H), 3.25 (t, *J* = 5.2 Hz, 2H), 1.67–1.66 (m, 4H), 1.53–1.51 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.61, 164.73, 132.26, 131.93, 130.82, 129.97, 46.91, 42.10, 26.11, 25.31, 24.20.



1-(4-Iodophenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3e): Yellow oil.[2] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88–7.85 (m, 2H), 7.64–7.62 (m, 2H), 3.67 (d, *J* = 2.4 Hz, 2H), 3.27–3.24 (m, 2H), 1.67 (d, *J* = 2.4 Hz, 4H), 1.52 (d, *J* = 3.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.97, 164.74, 138.27, 132.45, 130.61, 103.14, 46.93, 42.12, 26.13, 25.33, 24.22.

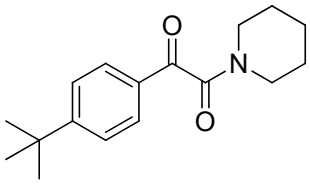


1-(4-Nitrophenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3f): Yellowish solid.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.34 (d, *J* = 8.8 Hz, 2H), 8.13 (d, *J* = 8.4 Hz, 2H), 3.71 (br, s, 2H), 3.31 (t, *J* = 5.6 Hz, 2H), 1.71 (br, s, 4H), 1.57 (br, s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.53, 164.11, 151.05, 137.70, 130.65, 124.12, 47.11, 42.48, 26.30, 25.44, 24.29.

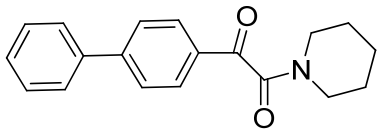


1-(4-Methoxyphenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3g): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 3.66 (t, *J* = 5.2 Hz, 2H), 3.25 (t, *J* = 5.6 Hz, 2H), 1.66 (t, *J* = 2.8 Hz, 4H), 1.51 (br, s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.58, 165.66, 164.66, 131.85, 126.18,

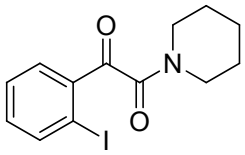
114.18, 55.51, 46.91, 41.93, 26.09, 25.33, 24.24.



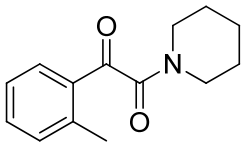
1-(4-(*tert*-Butyl)phenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3h): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 3.67 (br, s, 2H), 3.26 (t, *J* = 5.6 Hz, 2H), 1.66 (br, s, 4H), 1.52 (br, s, 2H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.67, 165.67, 158.68, 130.78, 129.54, 125.99, 47.02, 42.09, 35.33, 30.99, 26.23, 25.45, 24.39. HRMS (EI) ($[M]^+$) Calcd. for $C_{17}H_{23}NO_2$: 273.1728, Found: 273.1729.



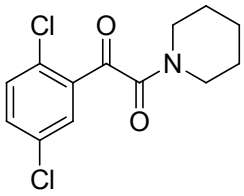
1-([1,1'-Biphenyl]-4-yl)-2-(piperidin-1-yl)ethane-1,2-dione (3i): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.03 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.63 (d, *J* = 7.2 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.2 Hz, 1H), 3.73 (br, s, 2H), 3.33 (t, *J* = 5.2 Hz, 2H), 1.71 (br, s, 4H), 1.57 (br, s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.57, 165.51, 147.37, 139.59, 132.02, 130.18, 129.04, 128.58, 127.66, 127.36, 47.10, 42.20, 26.28, 25.49, 24.42.



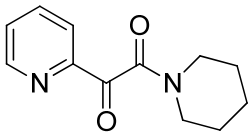
1-(2-Iodophenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3j): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.00–7.95 (m, 1H), 7.71–7.69 (m, 1H), 7.44–7.40 (m, 1H), 7.19–7.15 (m, 1H), 3.61 (d, *J* = 4.4 Hz, 2H), 3.34 (d, *J* = 4.4 Hz, 2H), 1.64–1.58 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.24, 163.85, 141.73, 136.15, 133.88, 132.89, 128.10, 92.90, 46.94, 42.29, 25.61, 24.98, 24.13. HRMS (EI) ($[M]^+$) Calcd. for $C_{13}H_{14}INO_2$: 343.0069, Found: 343.0068.



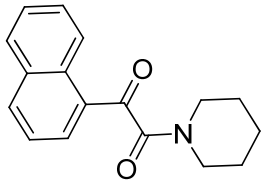
1-(Piperidin-1-yl)-2-(*o*-tolyl)ethane-1,2-dione (3k): Yellowish solid.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69 (d, *J* = 7.6 Hz, 1H), 7.46–7.42 (m, 1H), 7.30–7.26 (m, 2H), 3.66 (br, s, 2H), 3.27 (t, *J* = 5.6 Hz, 2H), 2.64 (s, 3H), 1.66 (br, s, 4H), 1.53 (br, s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.88, 166.13, 141.41, 133.61, 132.68, 132.58, 131.63, 126.13, 47.01, 42.11, 26.07, 25.38, 24.38, 21.81.



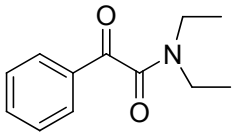
1-(2,5-Dichlorophenyl)-2-(piperidin-1-yl)ethane-1,2-dione (3l): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84 (d, *J* = 2.8 Hz, 1H), 7.47–7.44 (m, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 3.65 (t, *J* = 4.4 Hz, 2H), 3.43 (t, *J* = 5.6 Hz, 2H), 1.70–1.66 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =190.23, 165.18, 134.15, 131.63, 131.01, 130.77, 128.59, 123.06, 46.89, 42.29, 25.58, 25.18, 24.43. HRMS (ESI) ([M]+H) Calcd. for $C_{13}H_{14}Cl_2NO_2$: 286.0402, Found: 286.0400.



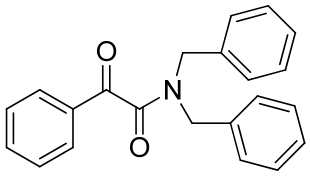
1-(Piperidin-1-yl)-2-(pyridin-2-yl)ethane-1,2-dione (3m): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.71 (d, *J* = 4.4 Hz, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.87 (dt, *J* = 7.6, 1.6 Hz, 1H), 7.51–7.48 (m, 1H), 3.68 (br, s, 2H), 3.23 (t, *J* = 5.6 Hz, 2H), 1.67–1.65 (m, 4H), 1.55 (br, s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.05, 166.04, 151.17, 149.80, 137.07, 127.86, 122.92, 46.86, 41.95, 25.72, 25.05, 24.32.



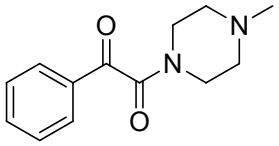
1-(Naphthalen-1-yl)-2-(piperidin-1-yl)ethane-1,2-dione (3n): Yellow oil.[3] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.29 (d, *J* = 8.8 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 8.03 (d, *J* = 7.2 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.69 (t, *J* = 7.6 Hz, 1H), 7.60–7.53 (m, 2H), 3.74 (br, s, 2H), 3.35 (t, *J* = 5.6 Hz, 2H), 1.70 (br, s, 4H), 1.54 (br, s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 194.44, 165.97, 135.86, 134.38, 134.07, 130.94, 129.28, 128.74, 128.55, 126.97, 125.86, 124.55, 47.17, 42.24, 26.11, 25.44, 24.40.



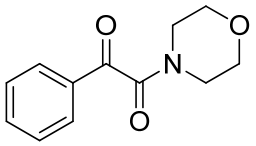
***N,N*-Diethyl-2-oxo-2-phenylacetamide (3o):** Yellow oil.[4] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95–7.93 (m, 2H), 7.66–7.62 (m, 1H), 7.53–7.49 (m, 2H), 3.57 (q, *J* = 7.2 Hz, 2H), 3.25 (q, *J* = 7.2 Hz, 2H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.16 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.56, 166.69, 134.53, 133.21, 129.58, 128.92, 42.06, 38.75, 14.06, 12.80.



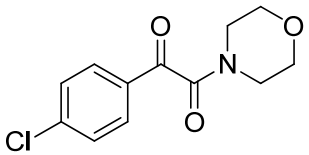
***N,N*-Dibenzyl-2-oxo-2-phenylacetamide (3p):** Yellow oil.[5] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.03–8.01 (m, 2H), 7.68–7.64 (m, 1H), 7.55–7.51 (m, 2H), 7.42–7.40 (m, 2H), 7.38–7.33 (m, 6H), 7.31–7.27 (m, 2H), 4.65 (s, 2H), 4.30 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.30, 167.49, 135.94, 134.80, 134.77, 133.34, 129.77, 129.06, 128.93, 128.87, 128.72, 128.27, 128.19, 127.95, 50.12, 46.08.



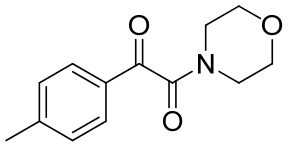
1-(4-Methylpiperazin-1-yl)-2-phenylethane-1,2-dione (3q): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95 (d, *J* = 7.2 Hz, 2H), 7.67–7.63 (m, 1H), 7.54–7.50 (m, 2H), 3.80 (t, *J* = 4.8 Hz, 2H), 3.38 (t, *J* = 4.8 Hz, 2H), 2.53 (t, *J* = 4.8 Hz, 2H), 2.39 (t, *J* = 4.8 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.46, 165.38, 134.79, 133.14, 129.63, 129.03, 54.91, 54.45, 45.99, 45.78, 41.16. HRMS (ESI) ([M]+H) Calcd. for $C_{13}H_{17}N_2O_2$: 233.1290, Found: 233.1285.



1-Morpholino-2-phenylethane-1,2-dione (3r): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.95–7.92 (m, 2H), 7.65–7.61 (m, 1H), 7.52–7.48 (m, 2H), 3.77 (br, s, 4H), 3.63 (t, *J* = 4.4 Hz, 2H), 3.35 (t, *J* = 4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.17, 165,43, 134.94, 133.04, 129.64, 129.10, 66.70, 66.62, 46.24, 41.59.

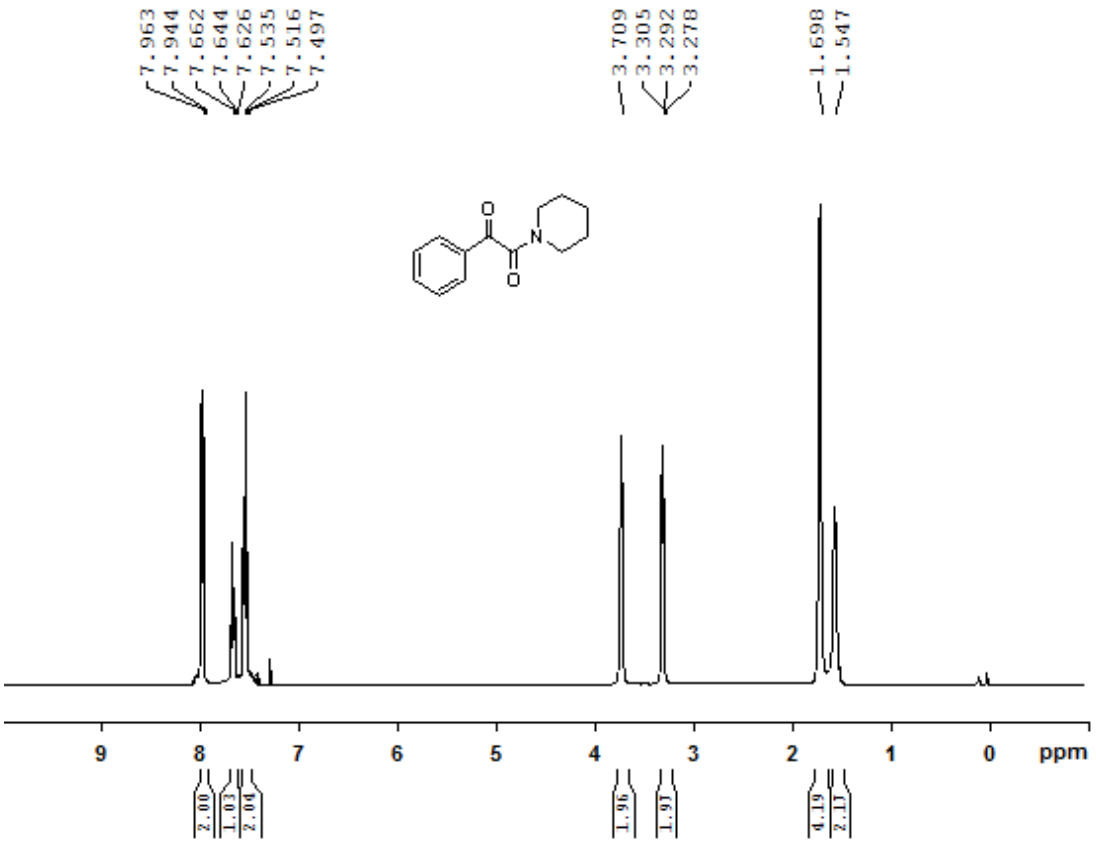


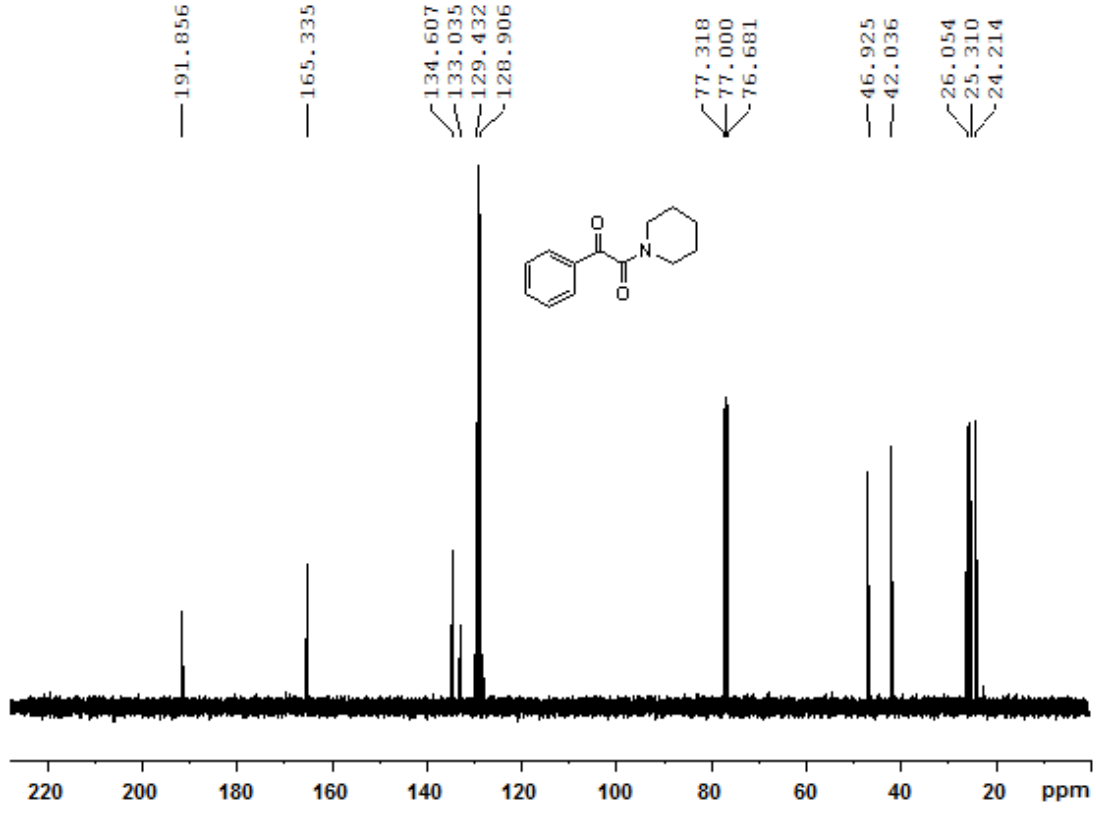
1-(4-Chlorophenyl)-2-morpholinoethane-1,2-dione (3s): Yellow oil.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89 (d, *J* = 8.8 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 3.80–3.76 (m, 4H), 3.65 (t, *J* = 4.8 Hz, 2H), 3.37 (t, *J* = 4.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 189.70, 164.89, 141.59, 131.48, 131.04, 129.50, 66.73, 66.63, 46.28, 41.70.

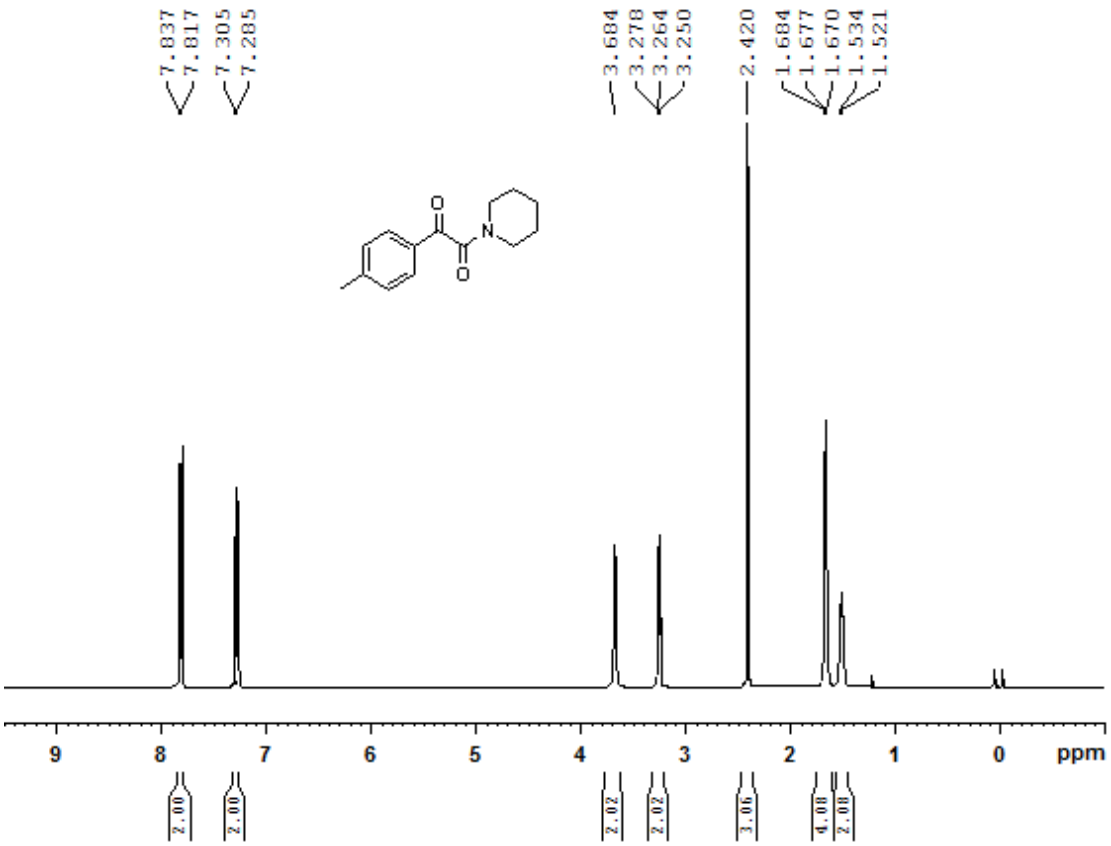


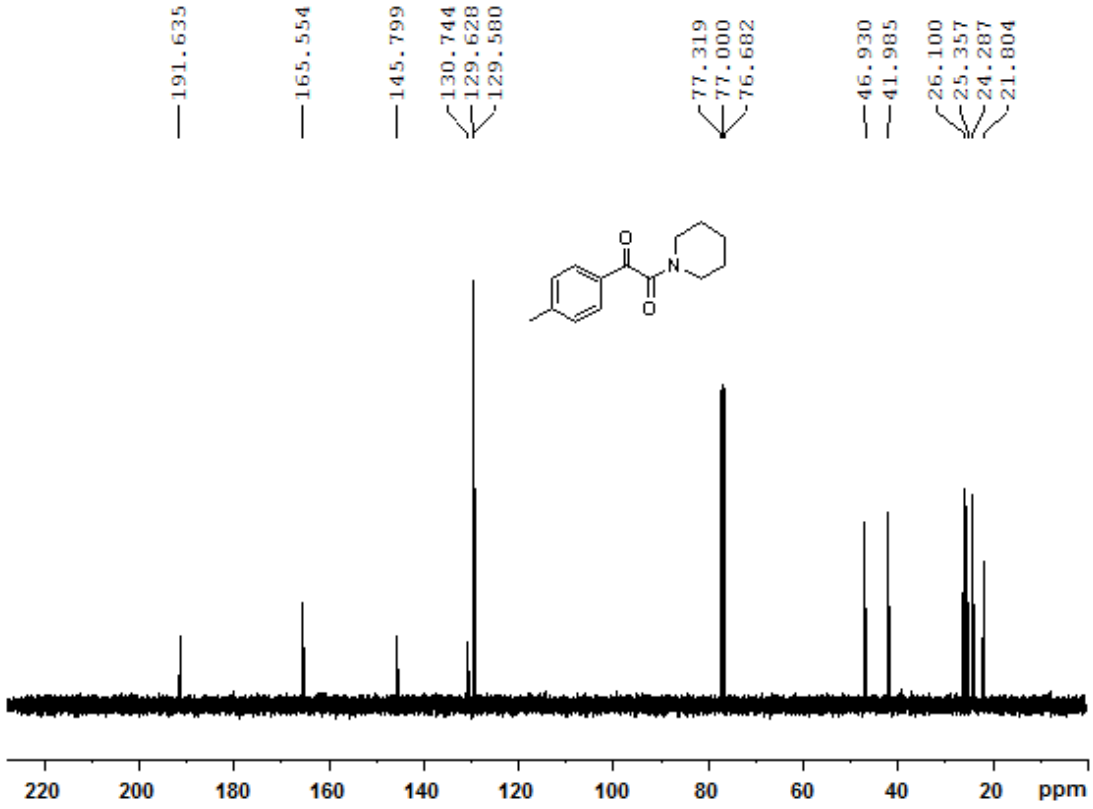
1-Morpholino-2-(*p*-tolyl)ethane-1,2-dione (3t): Yellow oil.[4] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.78 (br, s, 4H), 3.64 (t, *J* = 4.4 Hz, 2H), 3.36 (t, *J* = 4.4 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 190.90, 165.67, 146.28, 130.66, 129.83, 129.79, 66.75, 66.68, 46.27, 41.57, 21.92.

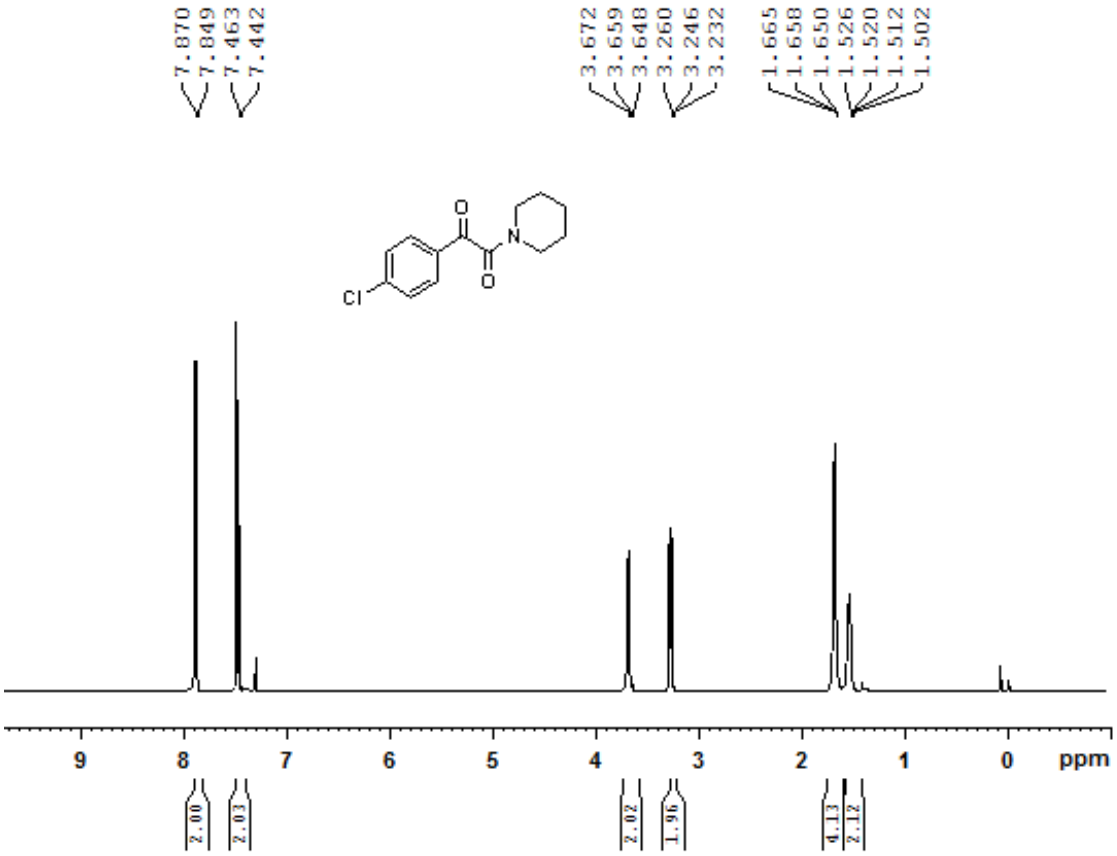
2. ^{1}H, ^{13}C NMR and HRMS spectra

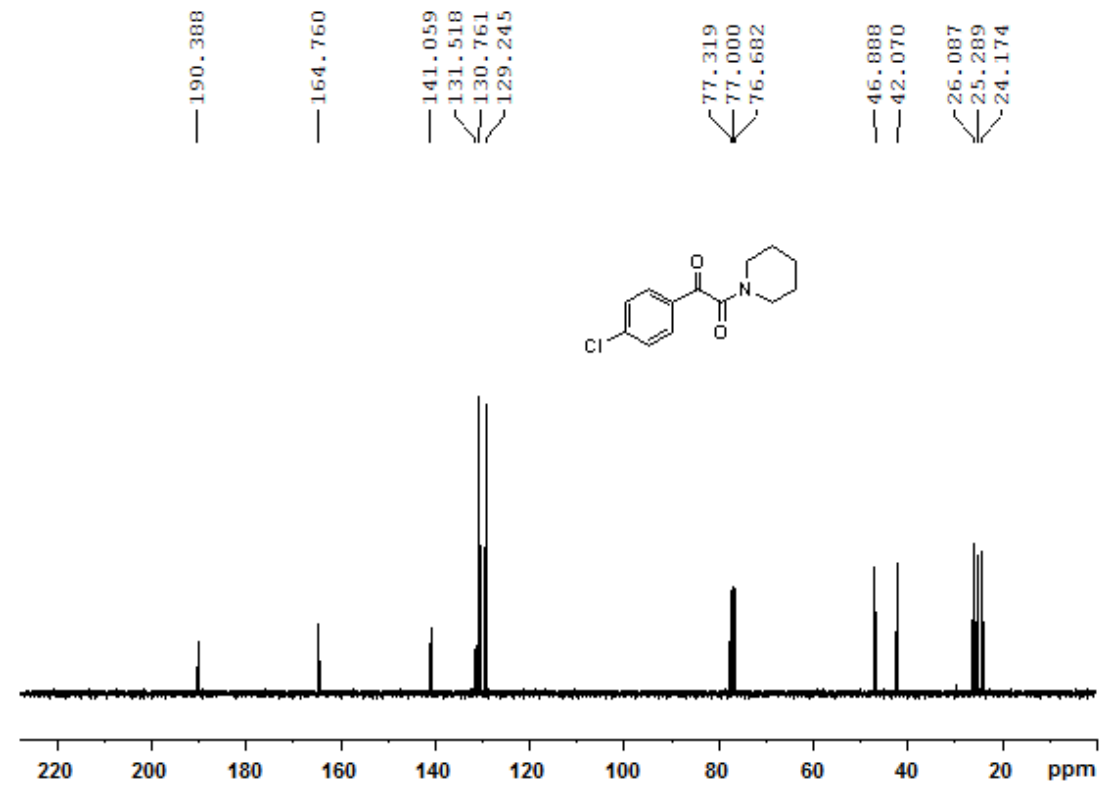


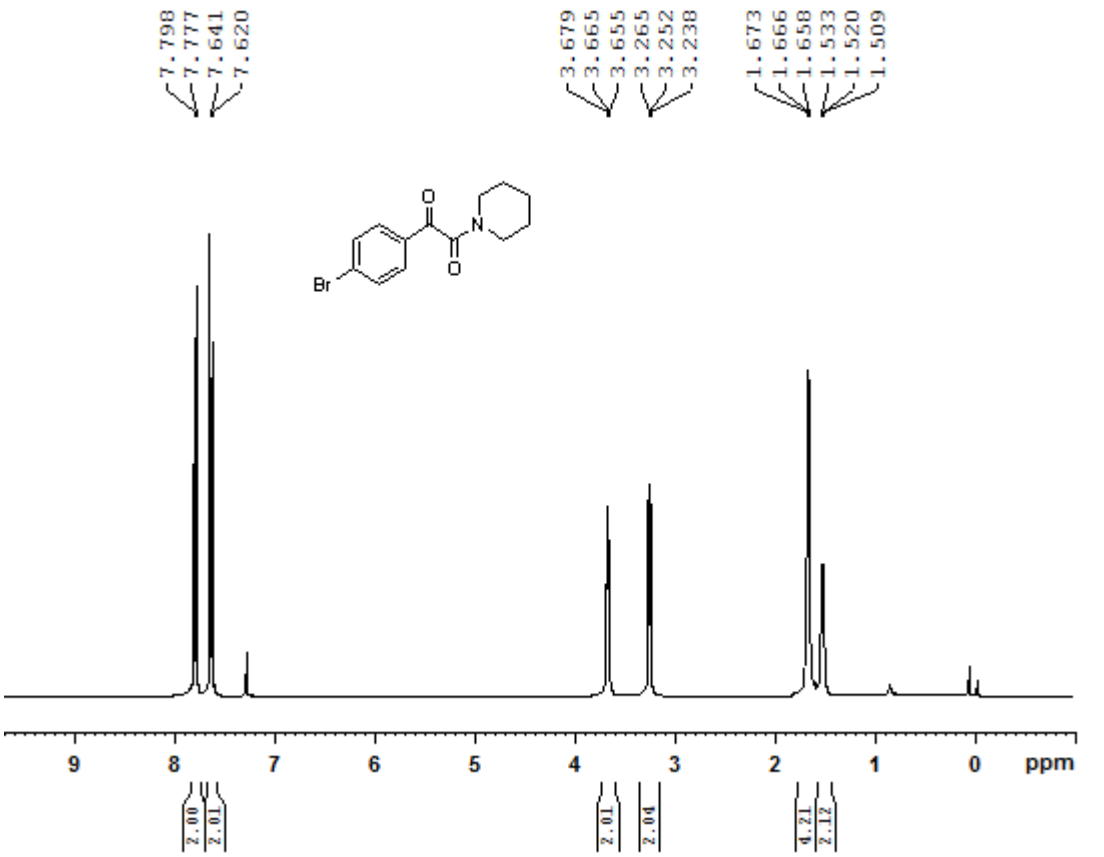


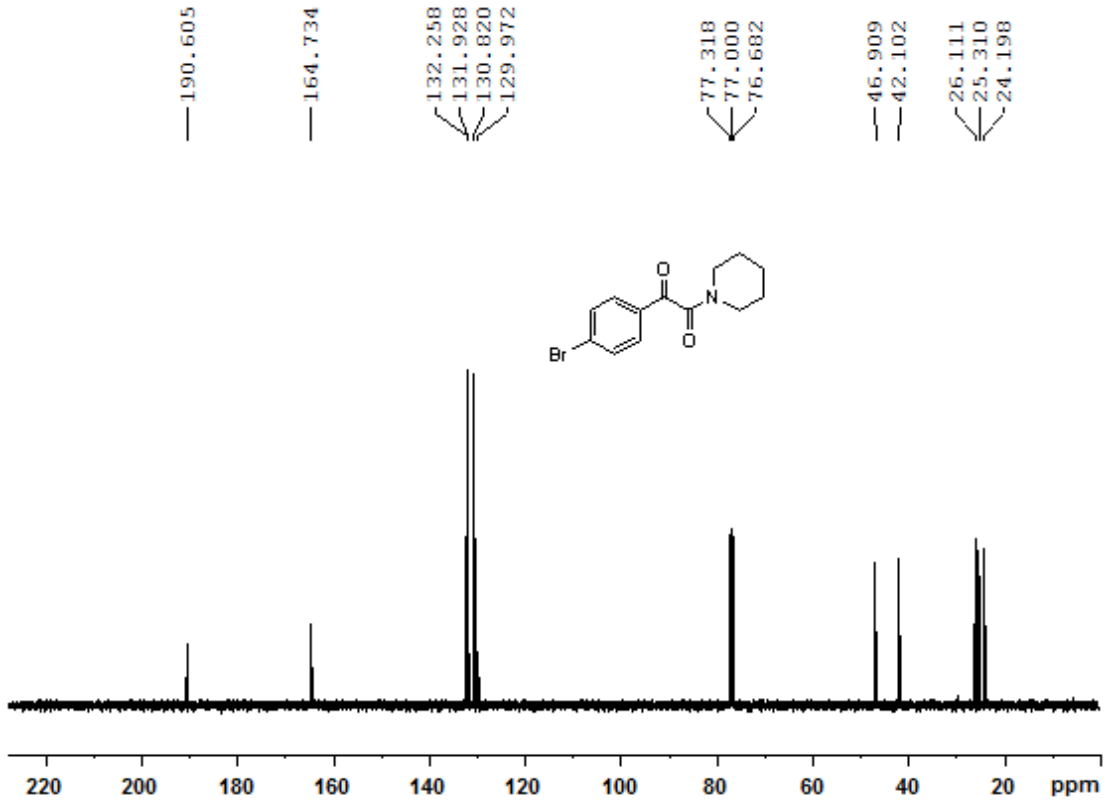


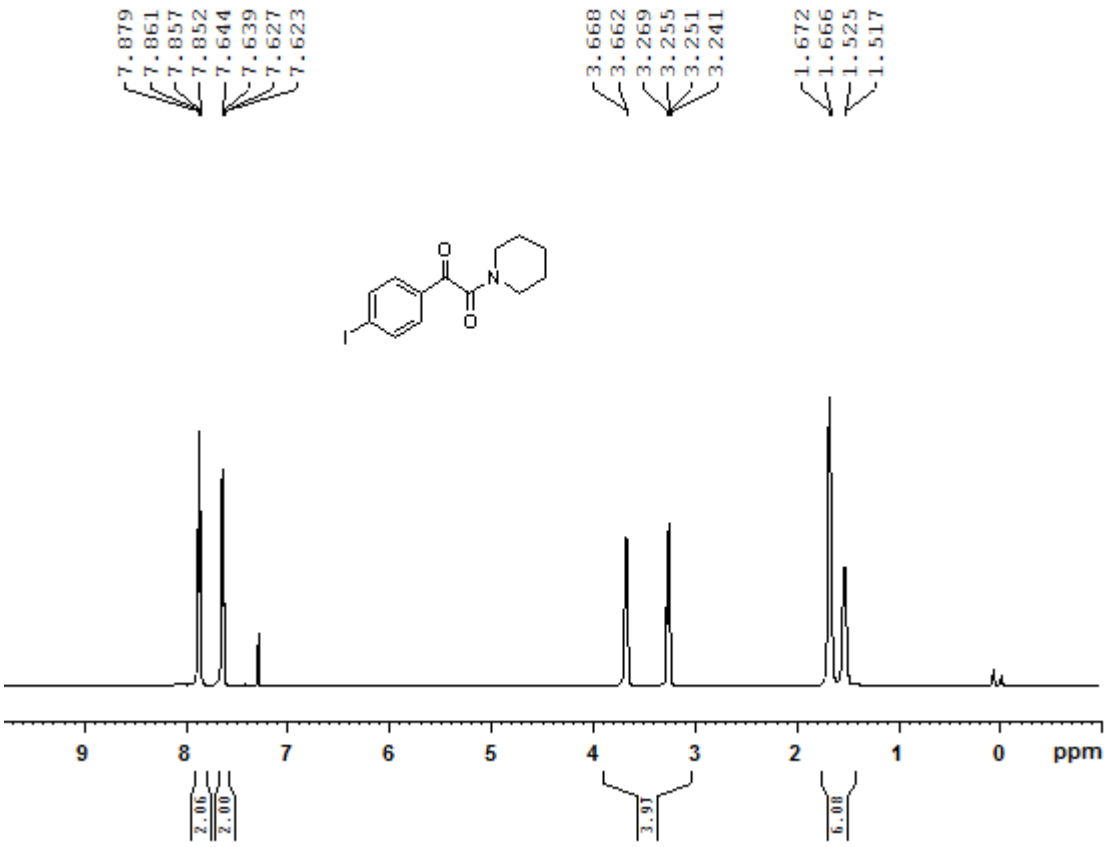








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3.251
3.241
1.672
1.666
1.525
1.517
O
N
O
I
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8
7
6
5
4
3
2
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0
ppm
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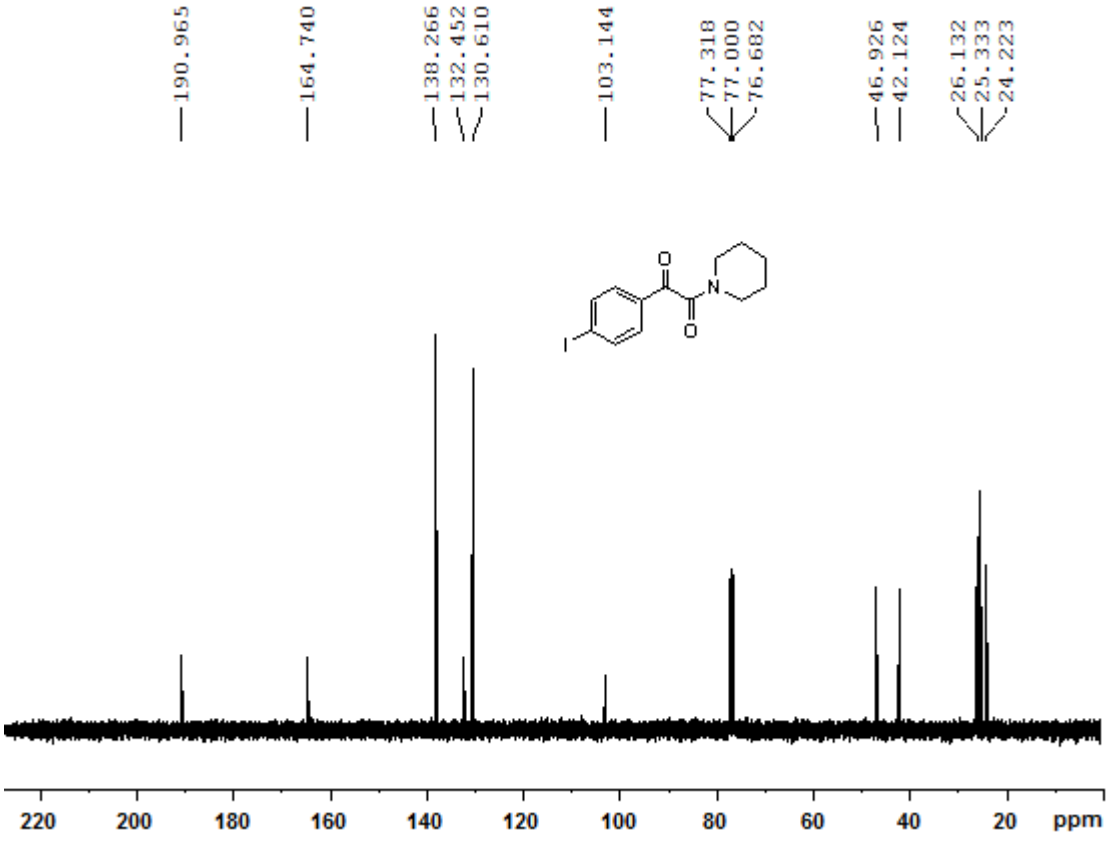
2.00
3.97
6.08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130.610
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77.318
77.000
76.682
46.926
42.124
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25.333
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O
N
O
I
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140
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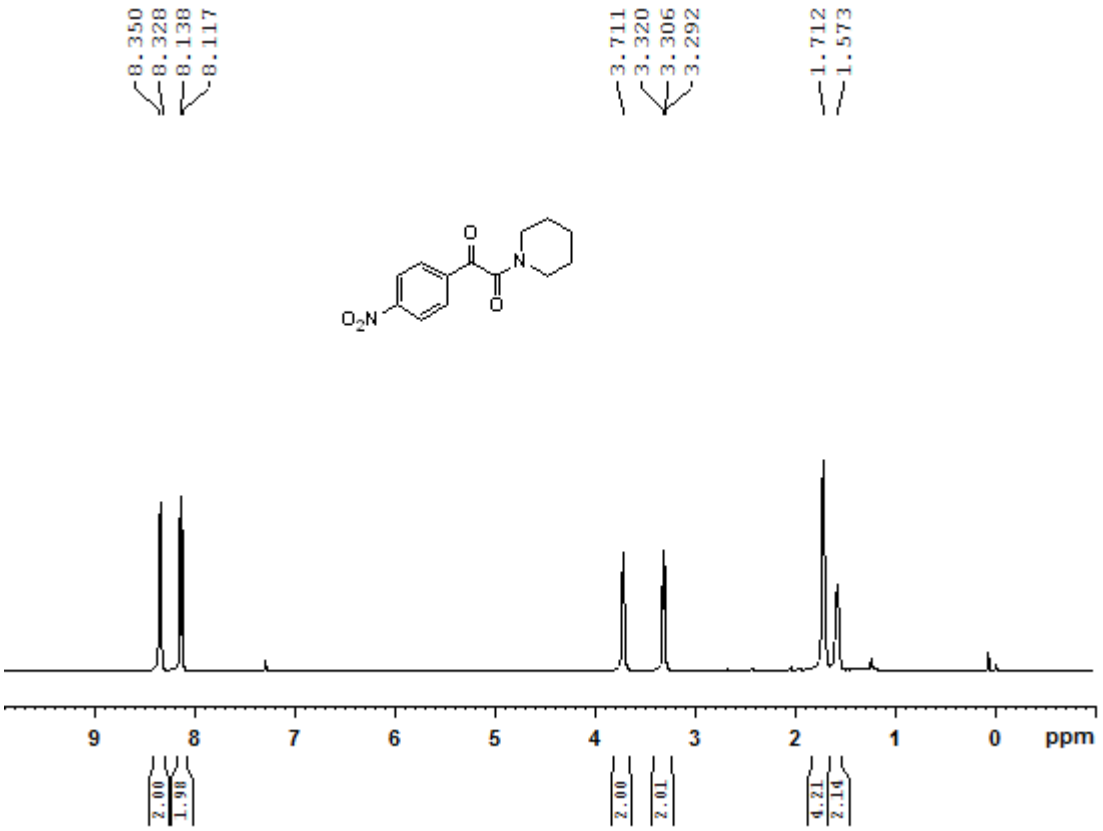
60
40
20
ppm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3.306
3.292
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1.573
O
N
O
O2N
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8
7
6
5
4
3
2
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2.00
2.01
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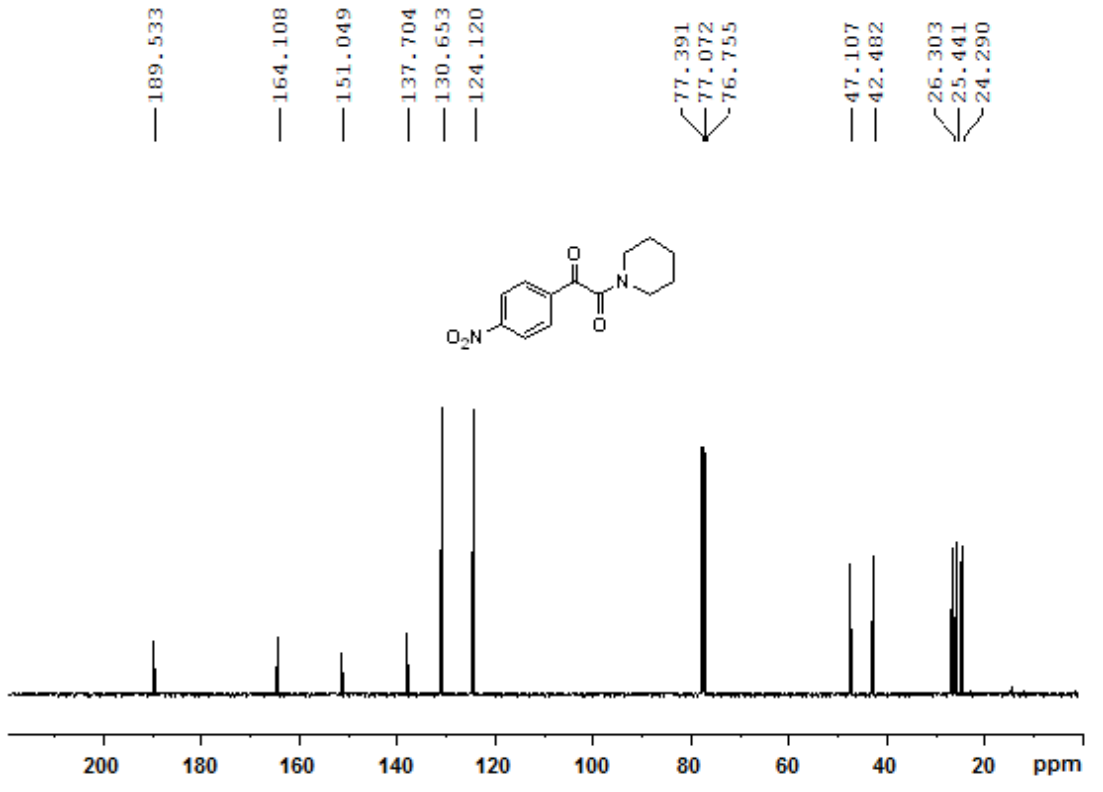
2.14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47.107
42.482
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O
N
O
O2N
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180
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140
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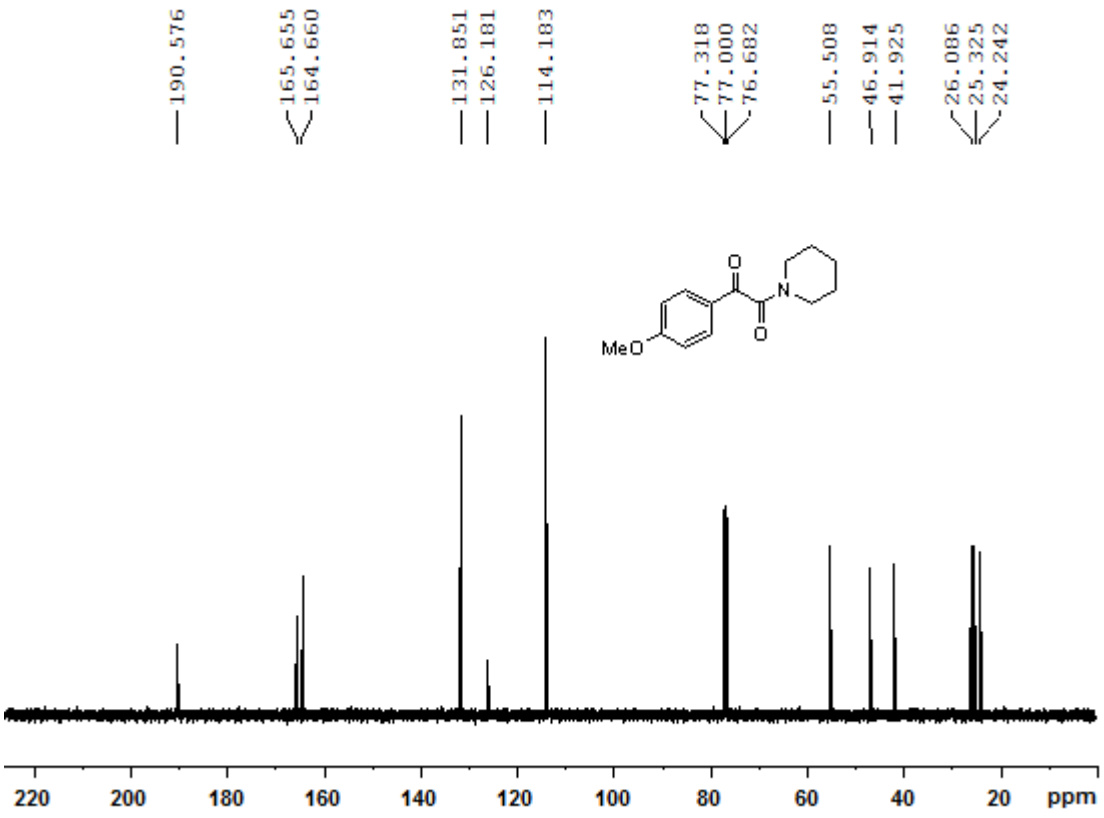
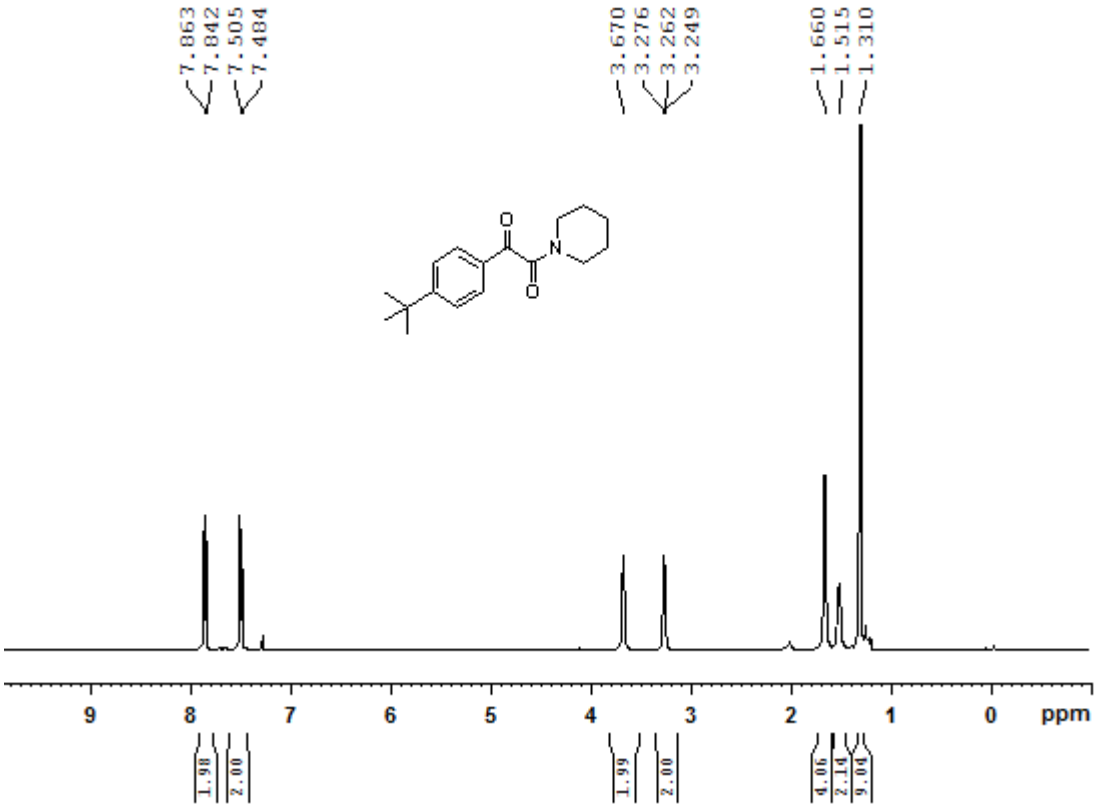
40
20
ppm

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3.249
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1.515
1.310
9
8
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3
2
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1.99
2.00
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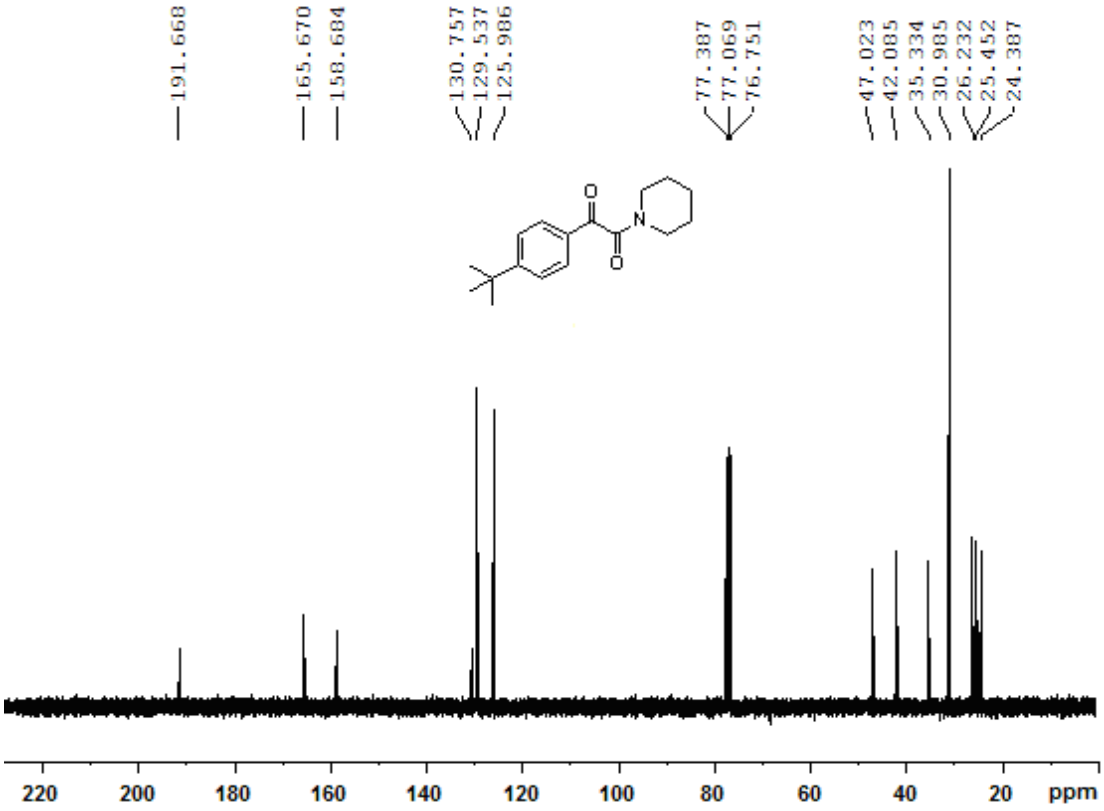
2.14
9.04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77.069
76.751
47.023
42.085
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25.452
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140
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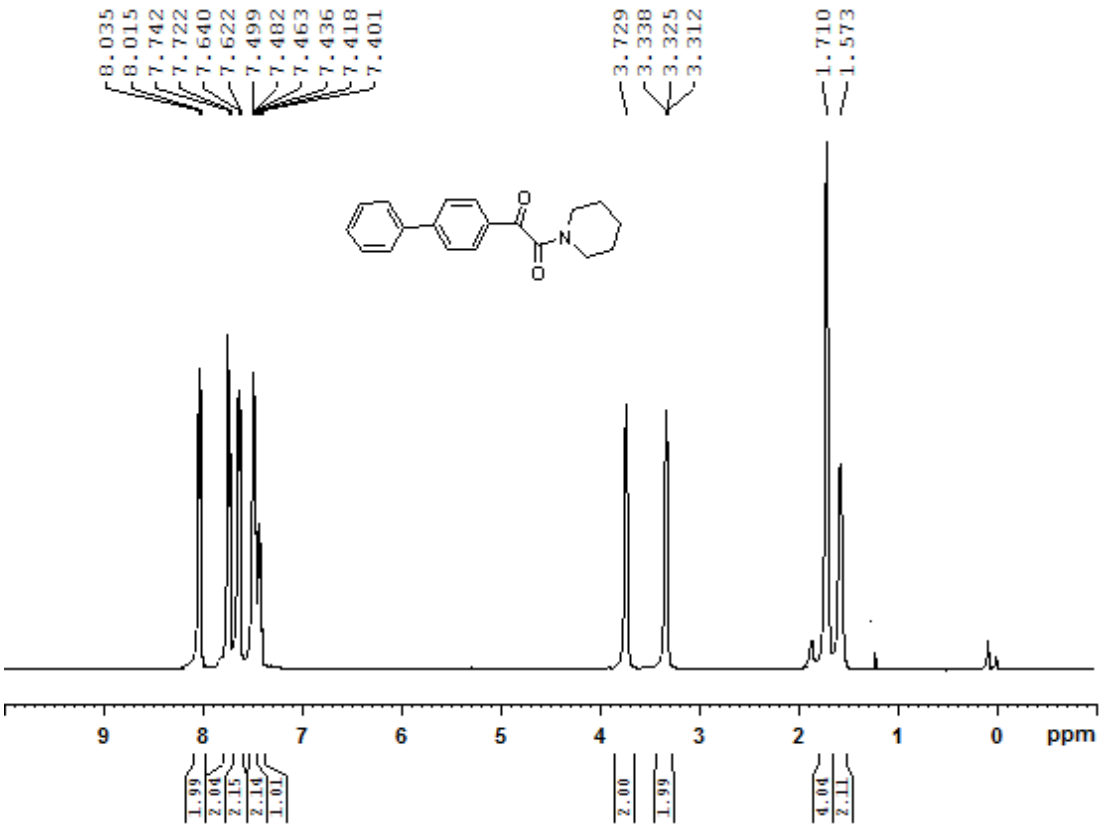
60
40
20
ppm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7.640
7.622
7.499
7.482
7.463
7.436
7.418
7.401
3.729
3.338
3.325
3.312
1.710
1.573
O
N
O
9
8
7
6
5
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ppm
1.99
2.04
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2.14
1.01
2.00
1.99
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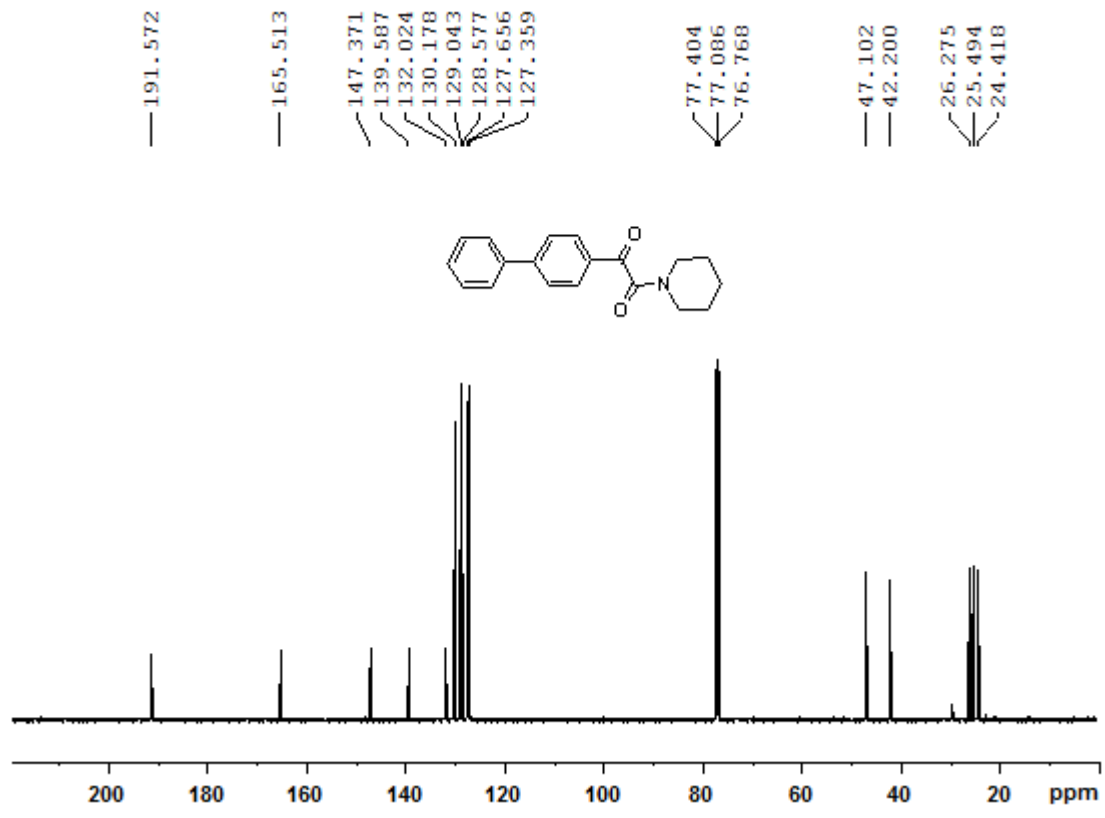
2.11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130.178
129.043
128.577
127.656
127.359
77.404
77.086
76.768
47.102
42.200
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24.418
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O
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140
120
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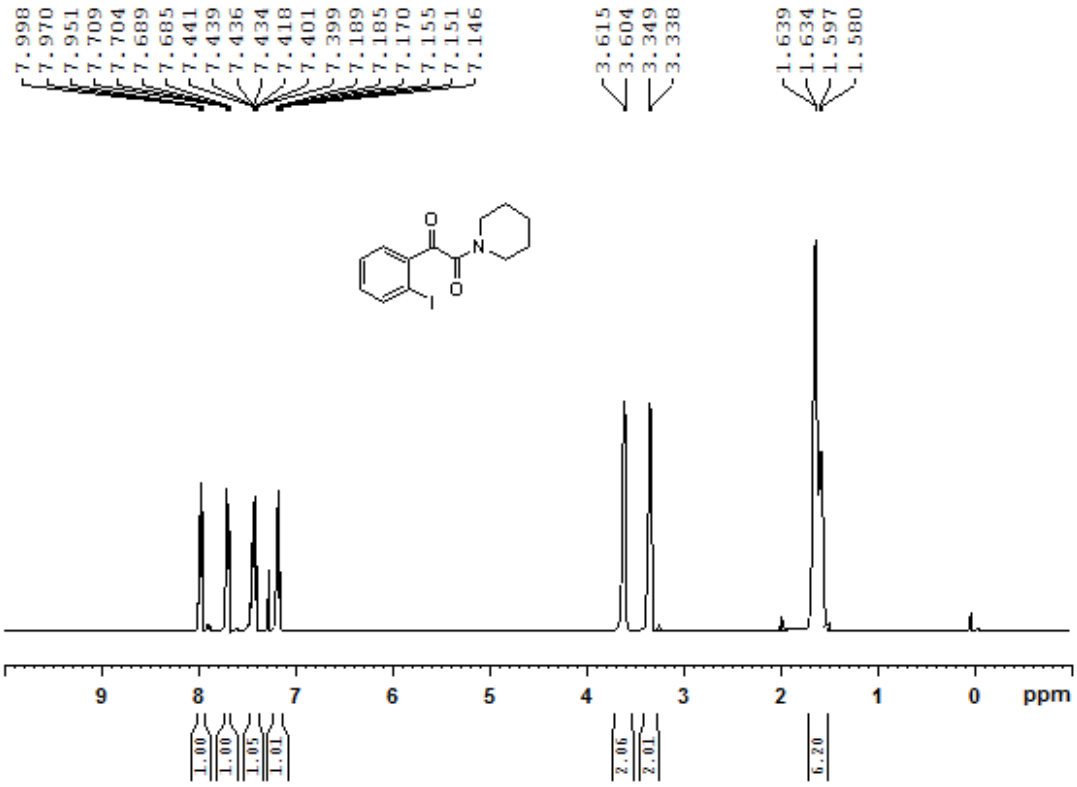
80
60
40
20
ppm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7.951
7.709
7.704
7.689
7.685
7.441
7.439
7.436
7.434
7.418
7.401
7.399
7.189
7.185
7.170
7.155
7.151
7.146
3.615
3.604
3.349
3.338
1.639
1.634
1.597
1.580
O
N
O
I
9
8
7
6
5
4
3
2
1
0
ppm
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1.00
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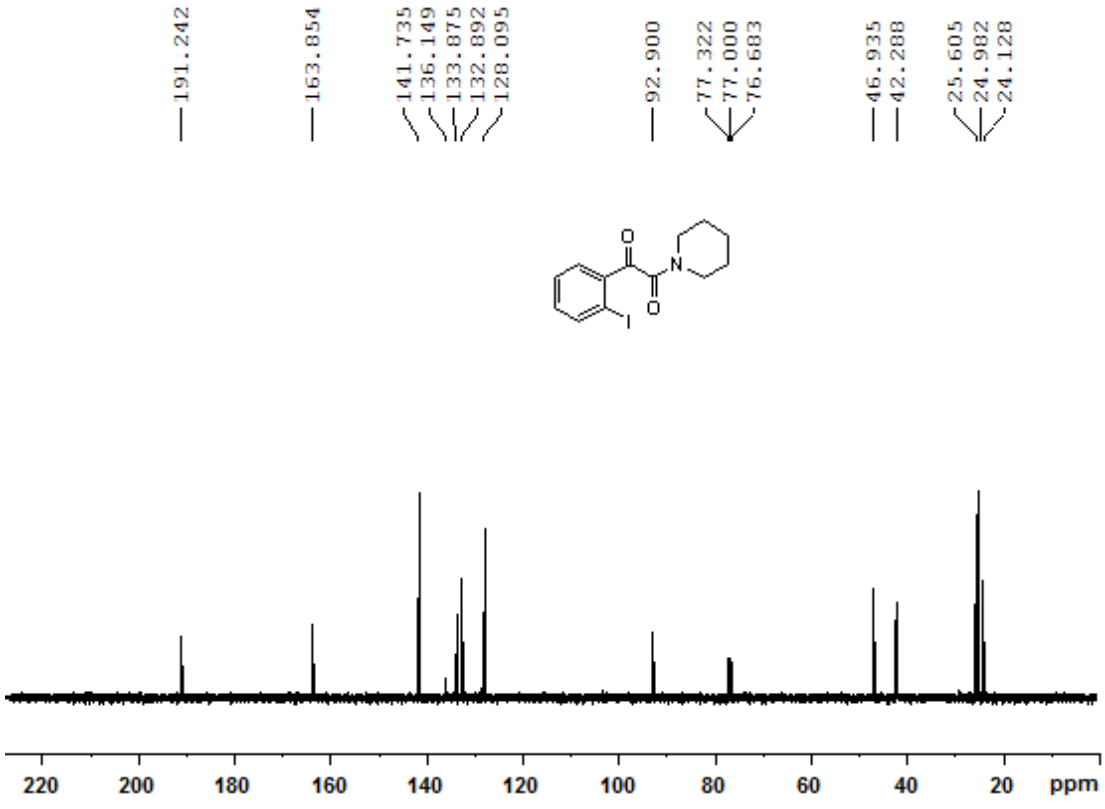
1.01
2.06
2.01
6.20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77.000
76.683
46.935
42.288
25.605
24.982
24.128
O
N
O
I
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200
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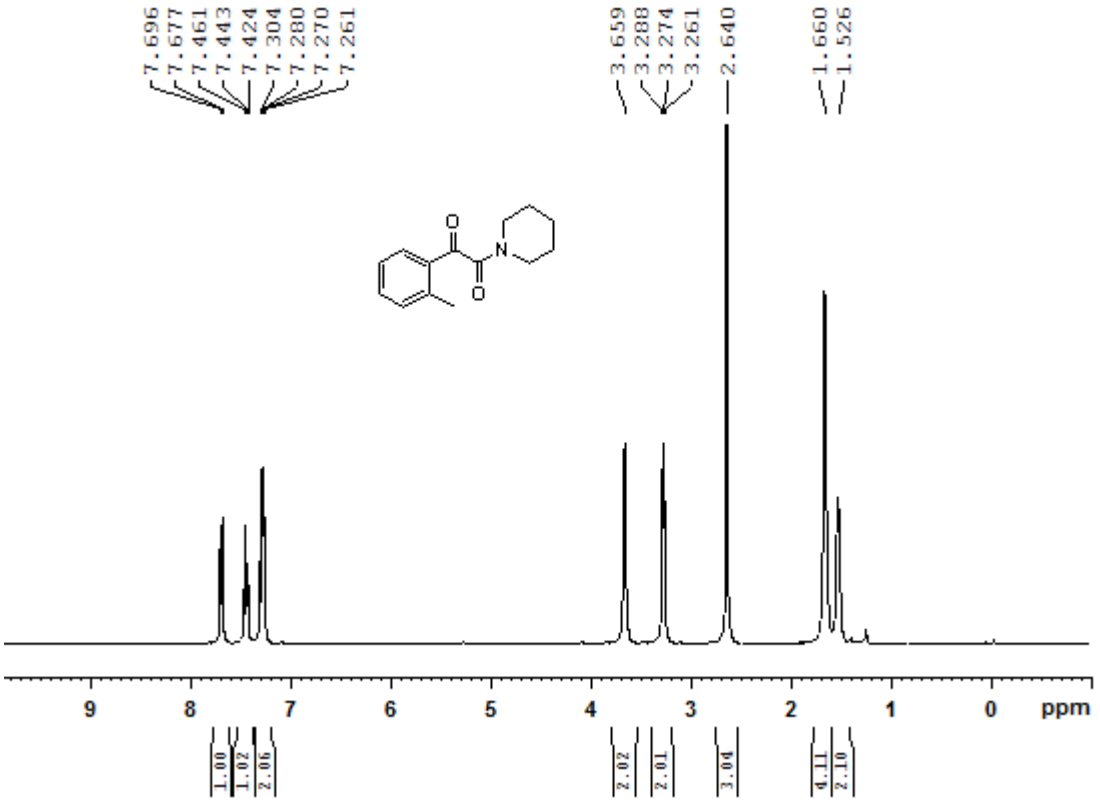
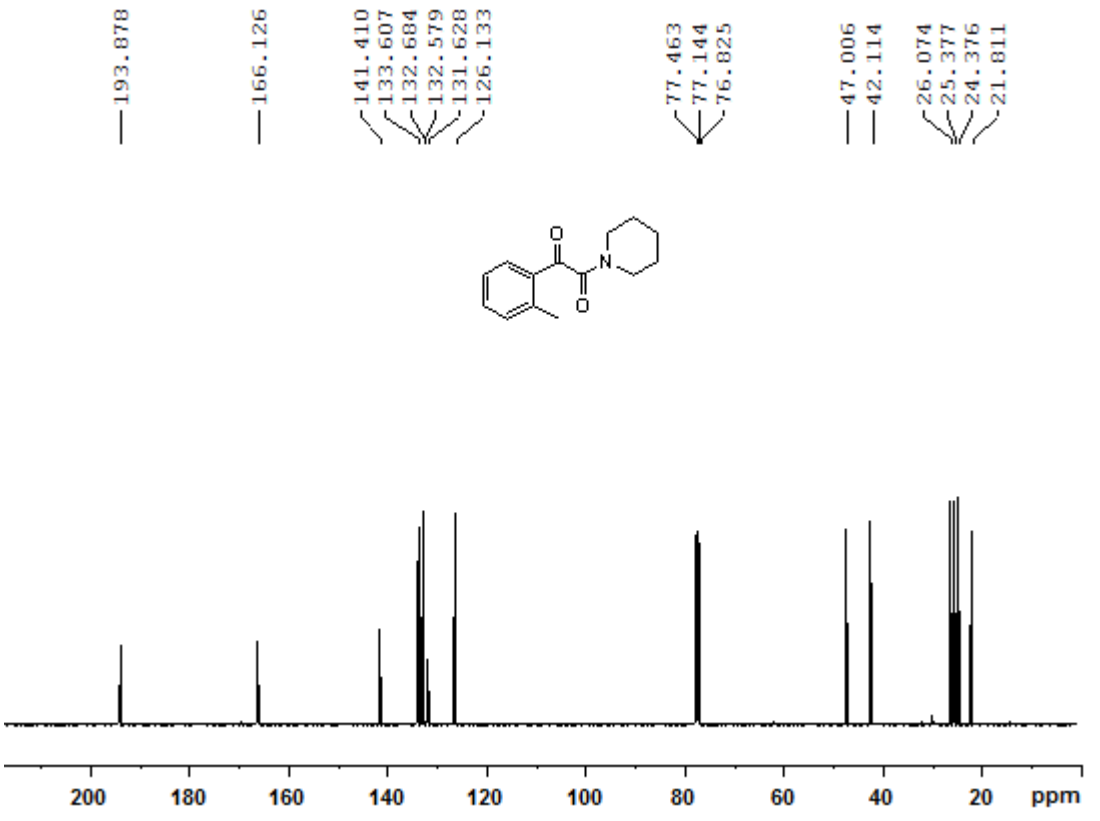
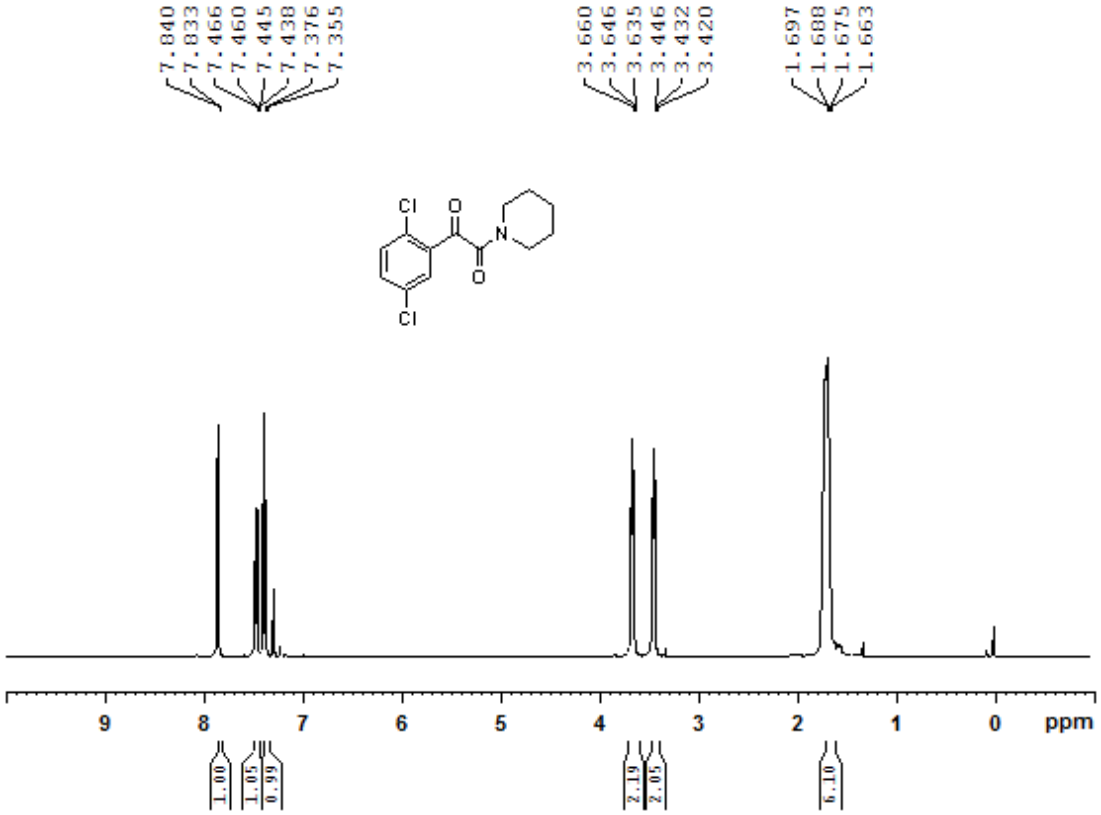
160
140
120
100
80
60
40
20
ppm



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7.355
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3.646
3.635
3.446
3.432
3.420
1.697
1.688
1.675
1.663
Cl
O
N
O
Cl
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8
7
6
5
4
3
2
1
0
ppm
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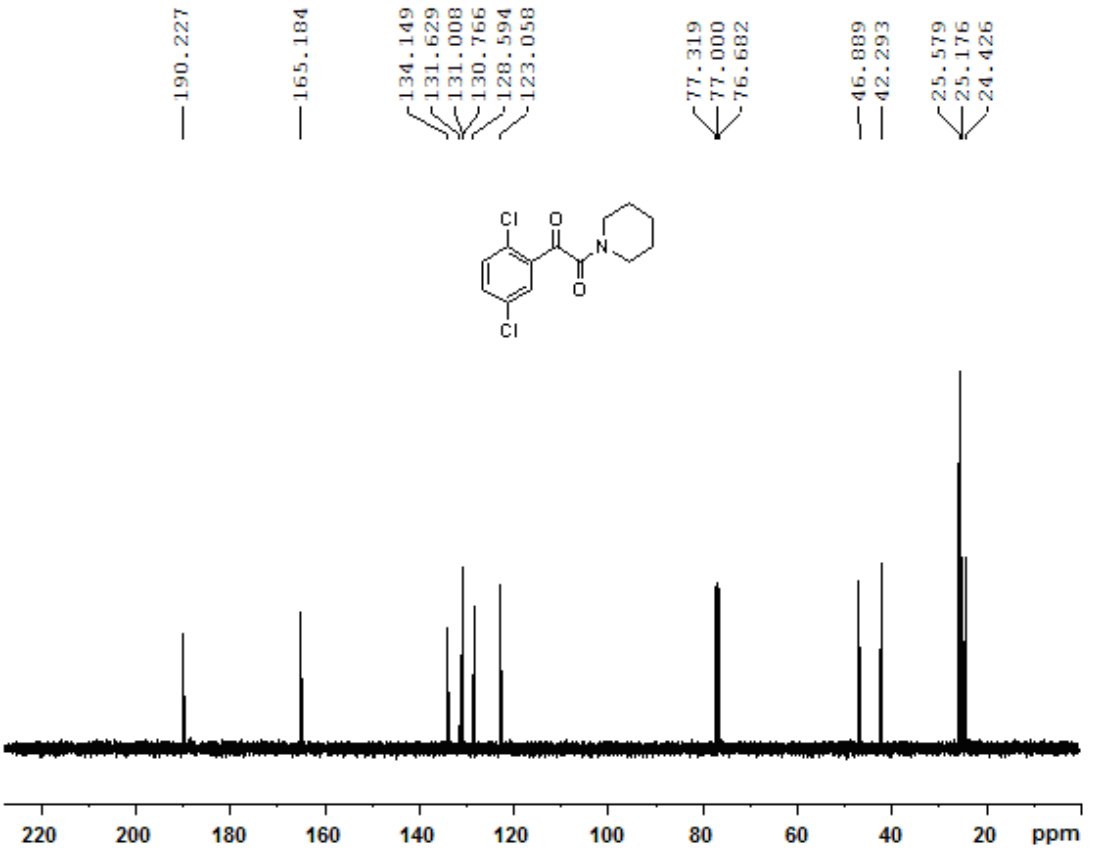
1.05
0.99
2.19
2.05
6.10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77.000
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42.293
25.579
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O
Cl
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140
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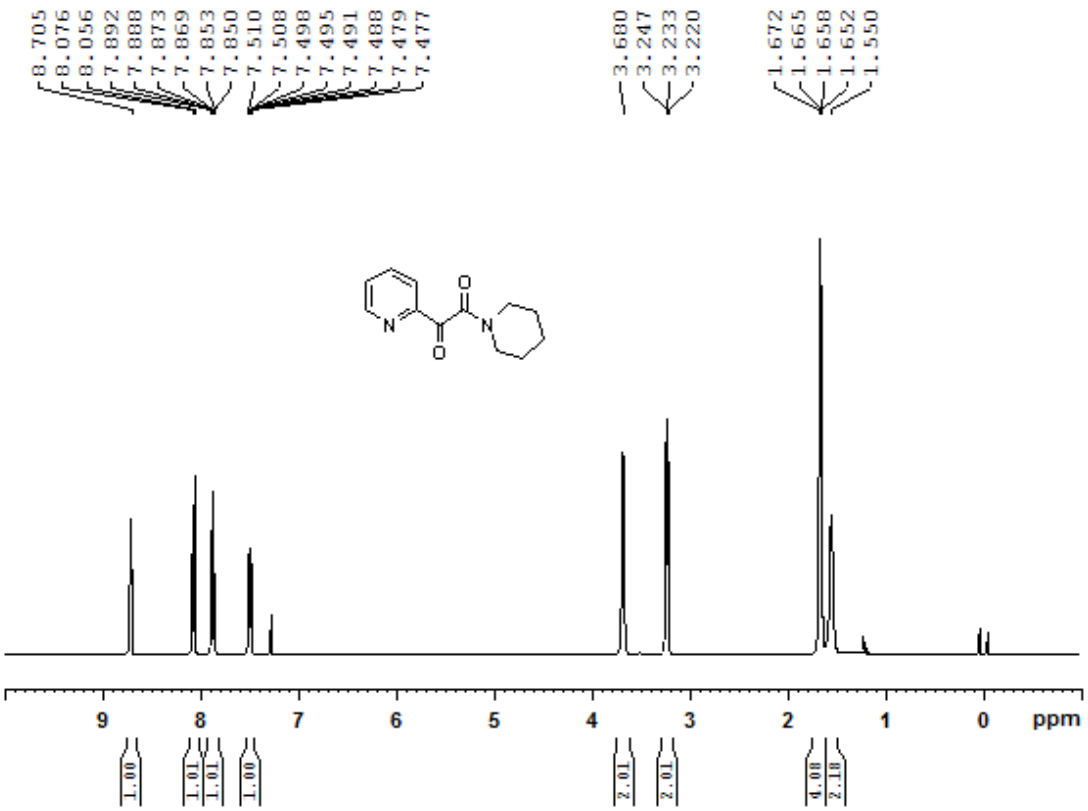
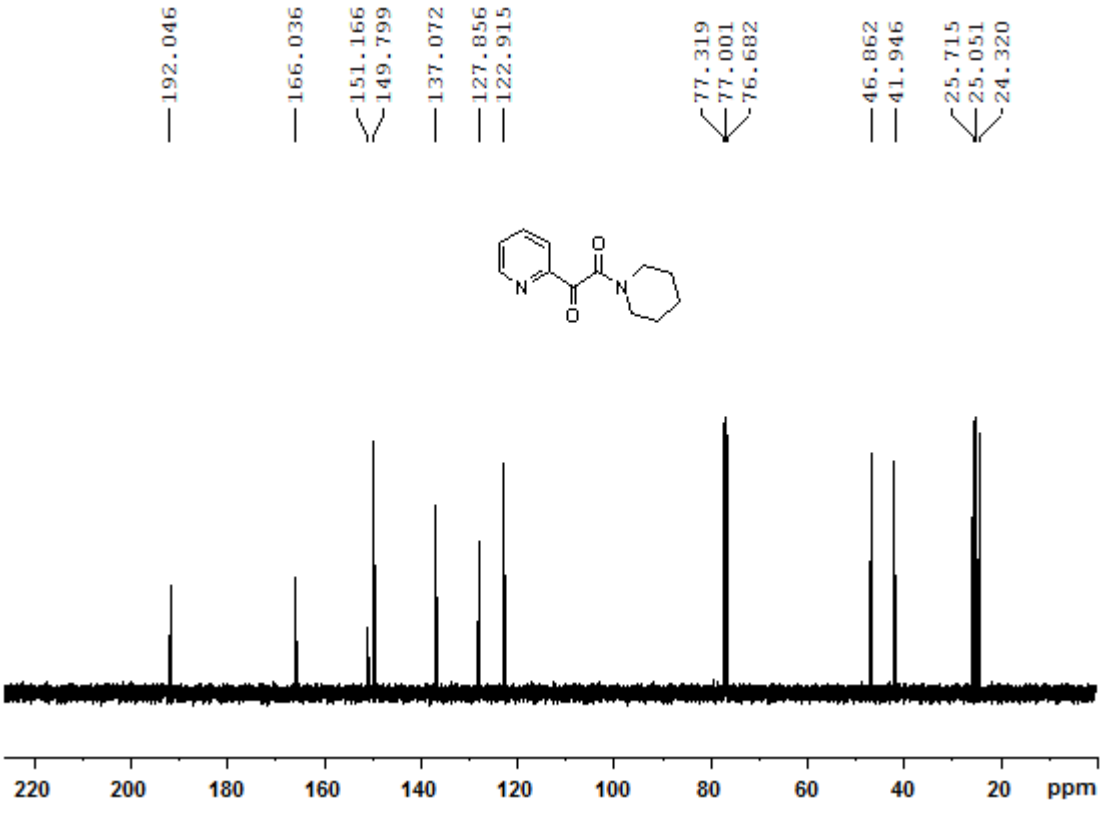
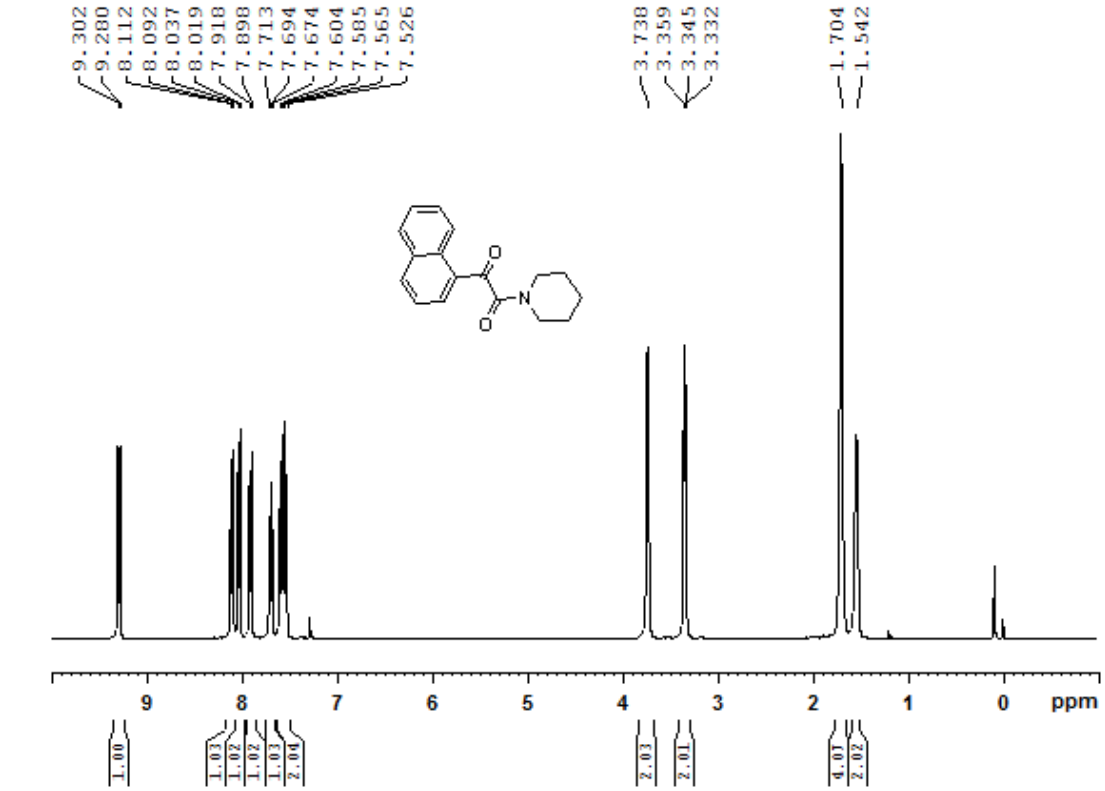
100
80
60
40
20
ppm



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7.604
7.585
7.565
7.526
3.738
3.359
3.345
3.332
1.704
1.542
O
O
N
9
8
7
6
5
4
3
2
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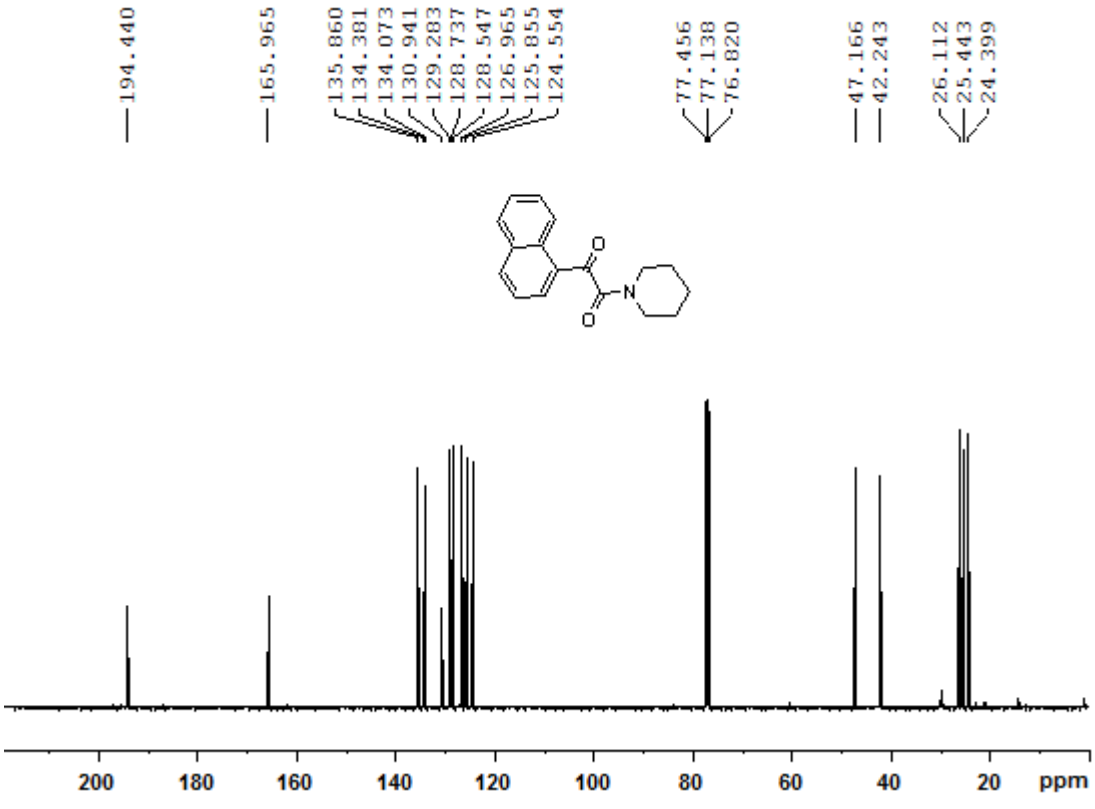
1.03
1.02
1.02
1.03
2.04
2.03
2.01
4.07
2.02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76.820
47.166
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O
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140
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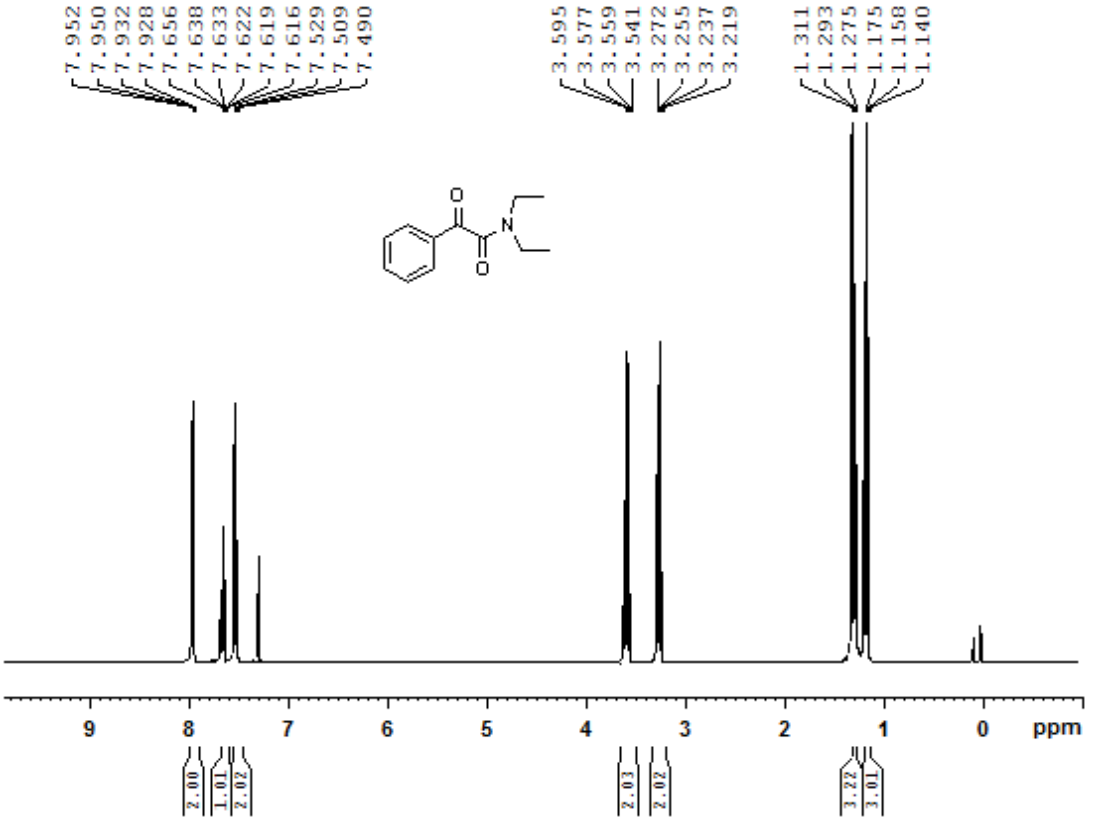
40
20
ppm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7.490
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3.577
3.559
3.541
3.272
3.255
3.237
3.219
1.311
1.293
1.275
1.175
1.158
1.140
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N
O
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8
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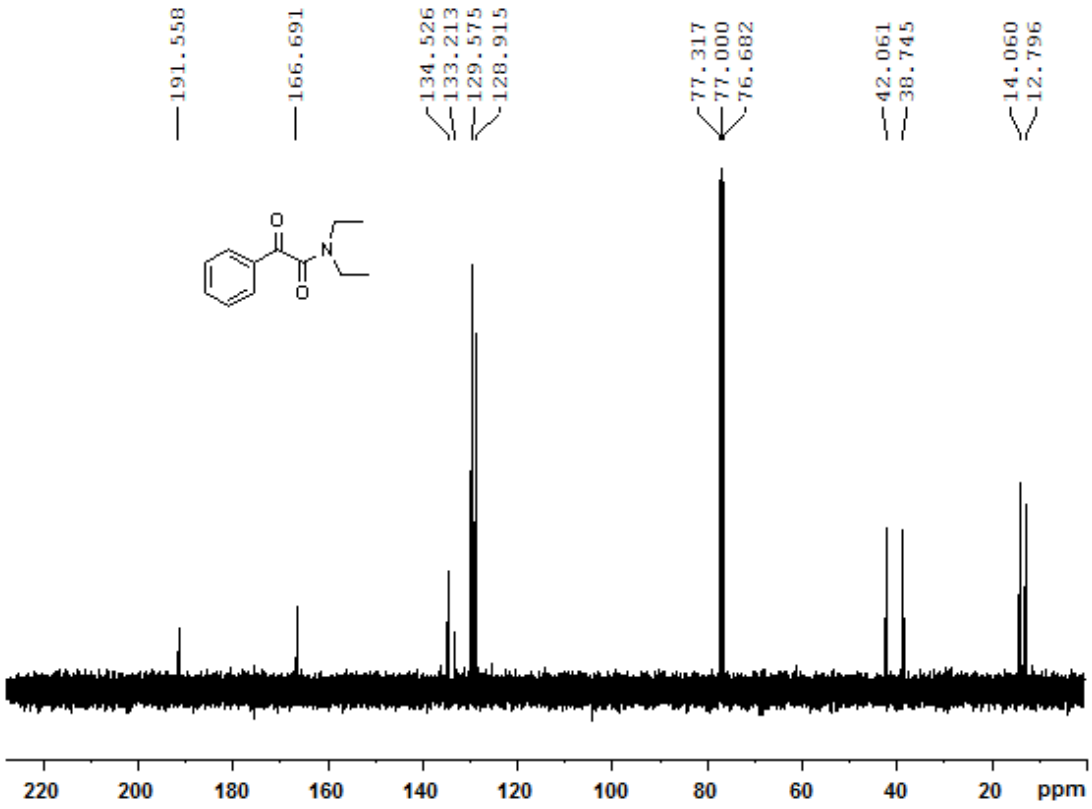
3.01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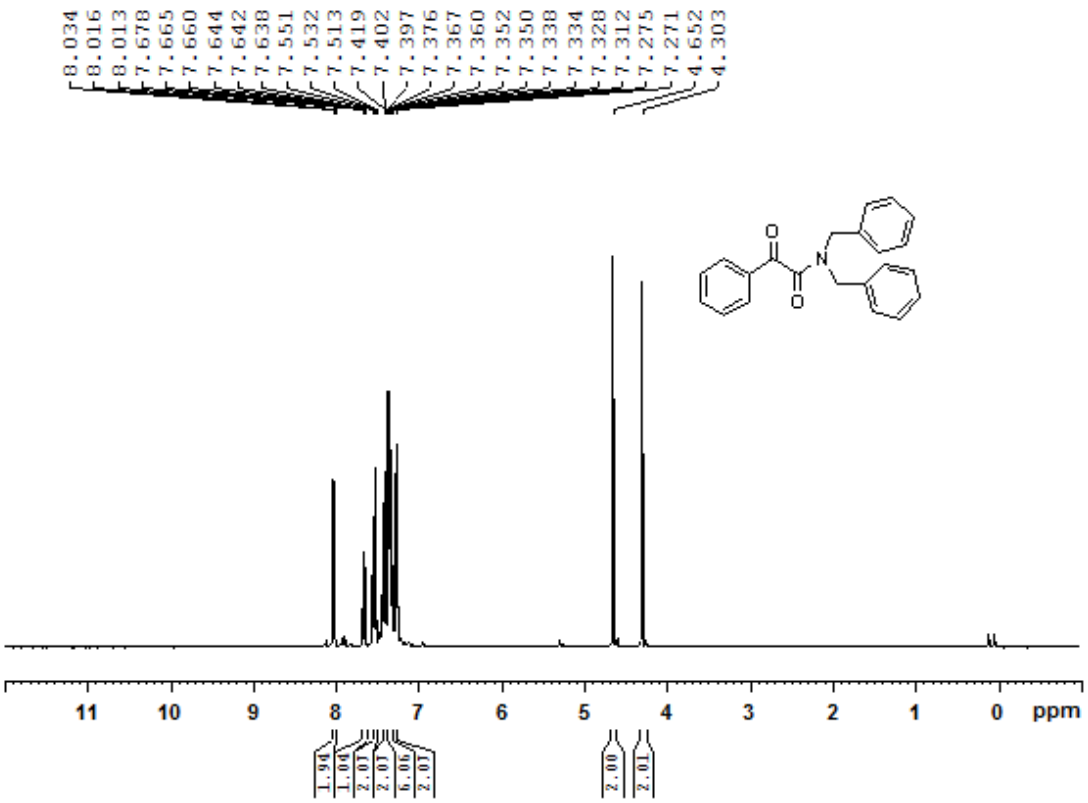
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7.360
7.352
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7.334
7.328
7.312
7.275
7.271
4.652
4.303
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8
7
6
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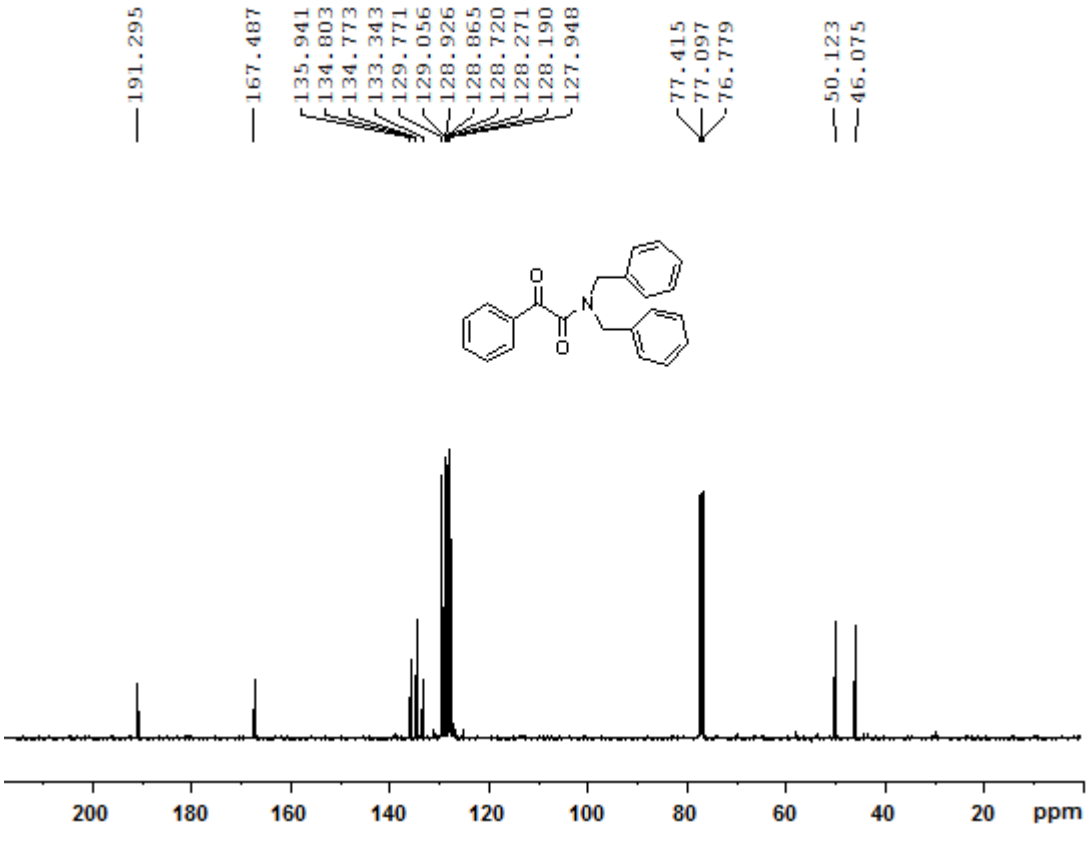
2.01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127.948
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140
120
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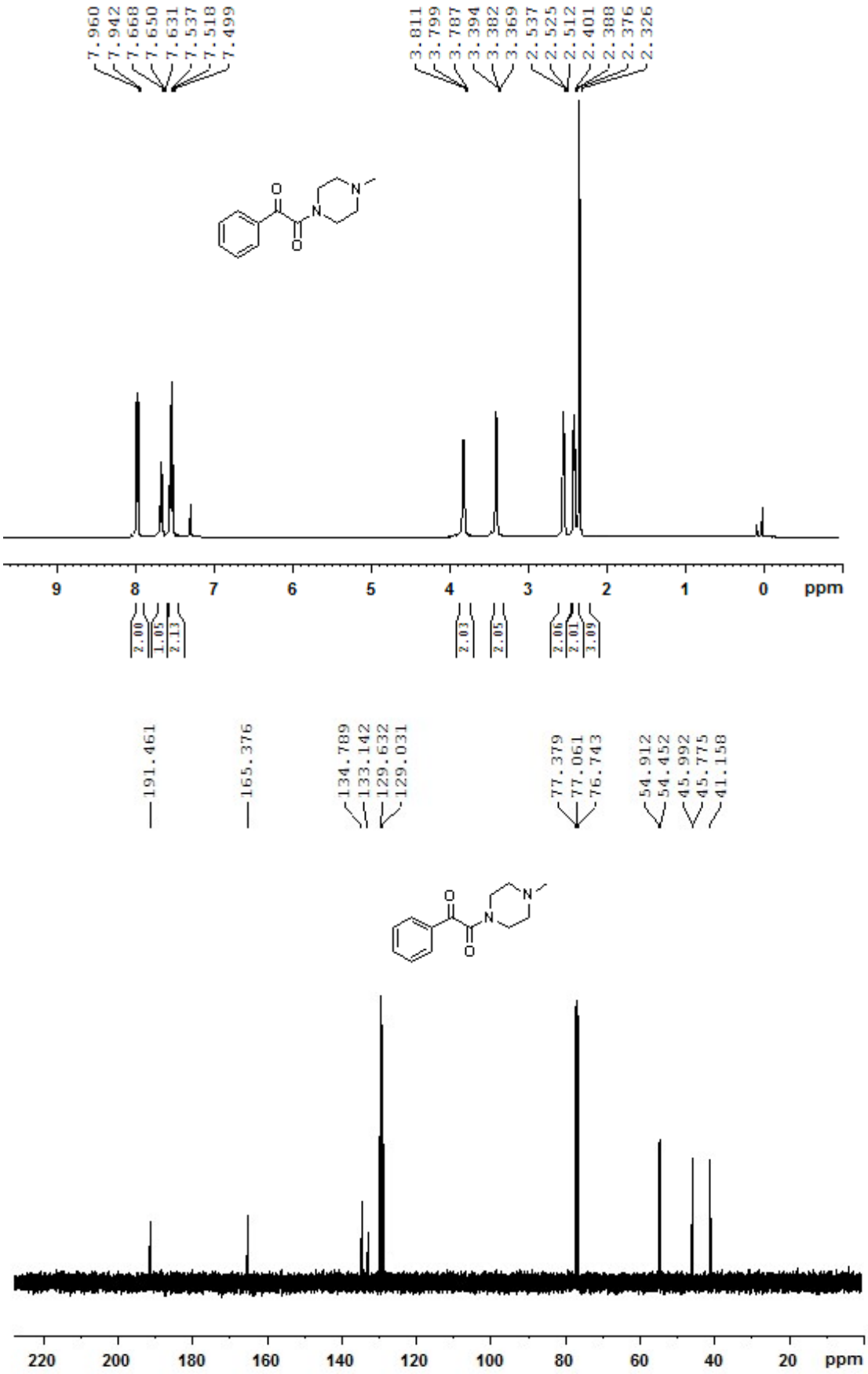
80
60
40
20
ppm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3.394
3.382
3.369
2.537
2.525
2.512
2.401
2.388
2.376
2.326
O
N
N
O
9
8
7
6
5
4
3
2
1
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ppm
2.00
1.05
2.13
2.03
2.05
2.06
2.01
3.09
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133.142
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77.061
76.743
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54.452
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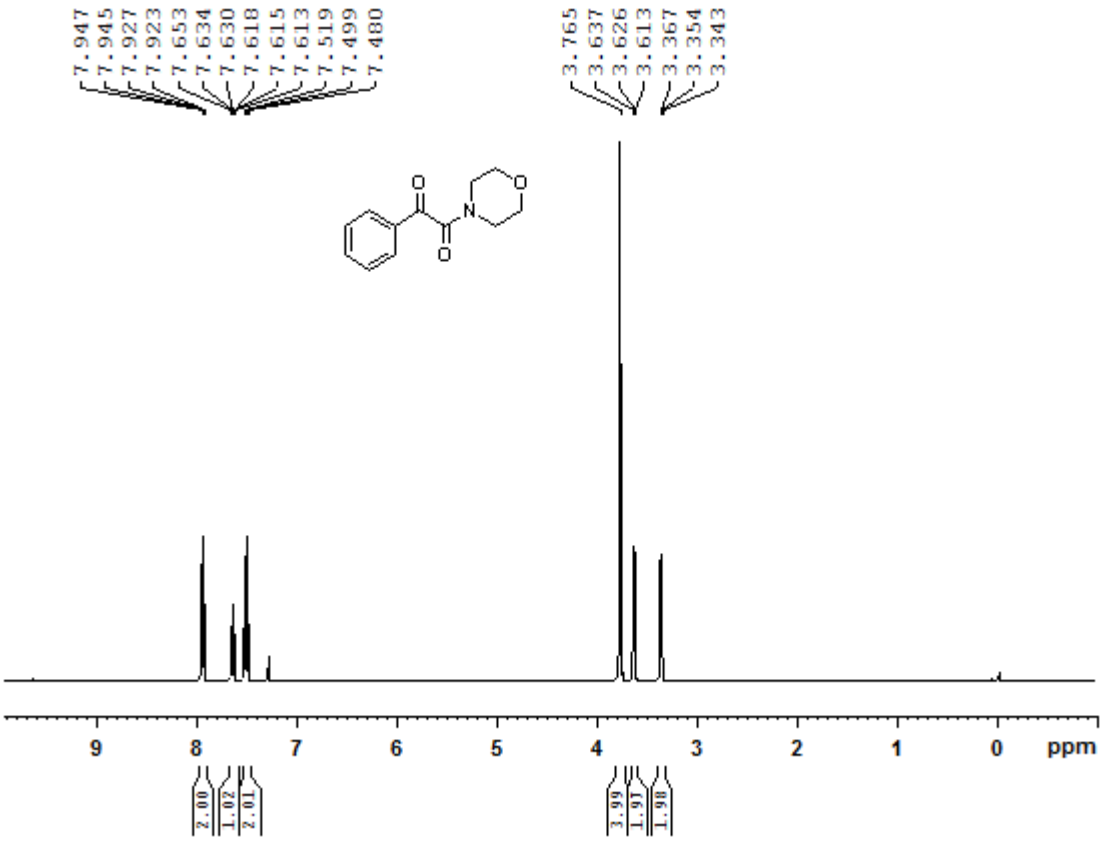
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7.613
7.519
7.499
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3.765
3.637
3.626
3.613
3.367
3.354
3.343
O
O
N
O
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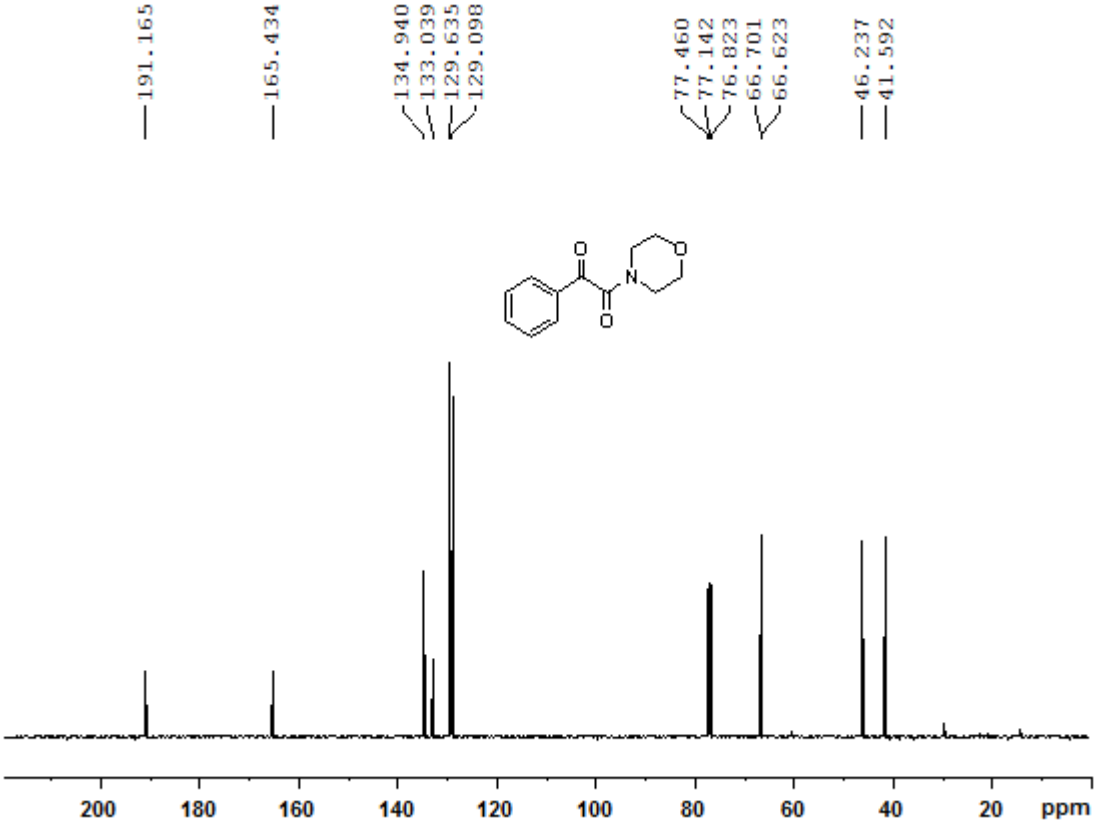
1.98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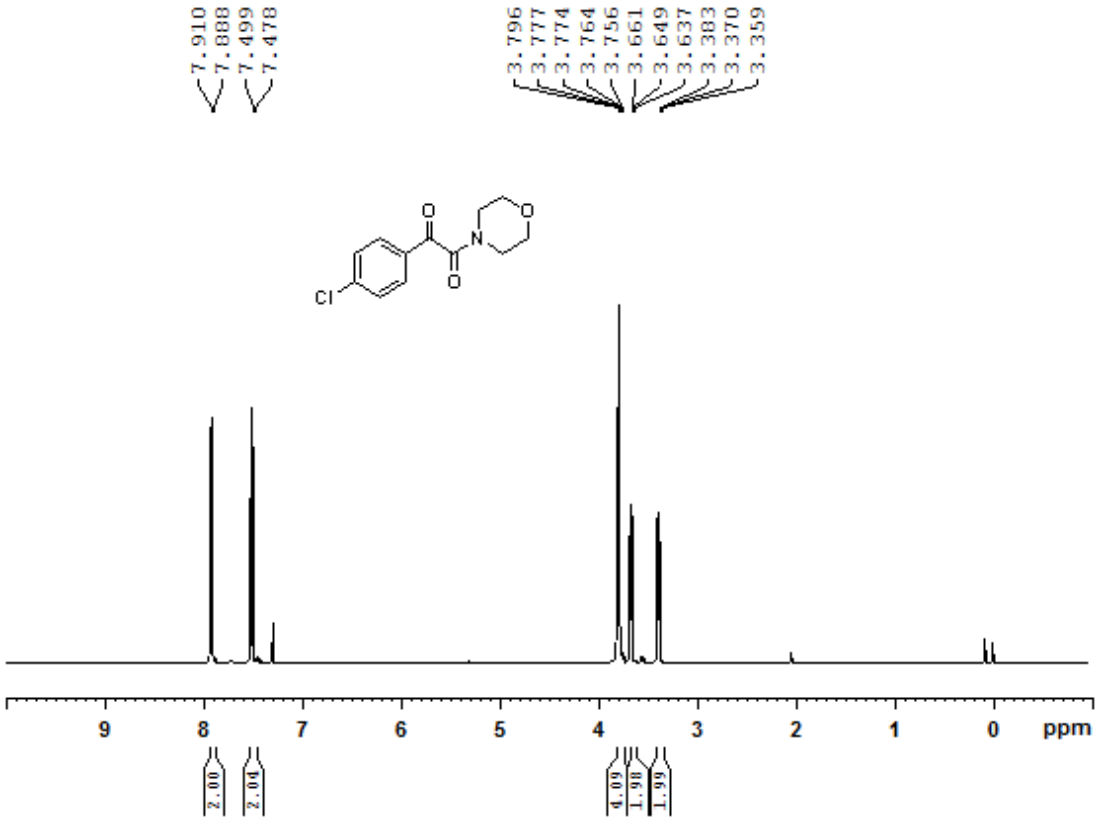
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3.370
3.359
Cl
O
O
N
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6
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4
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2
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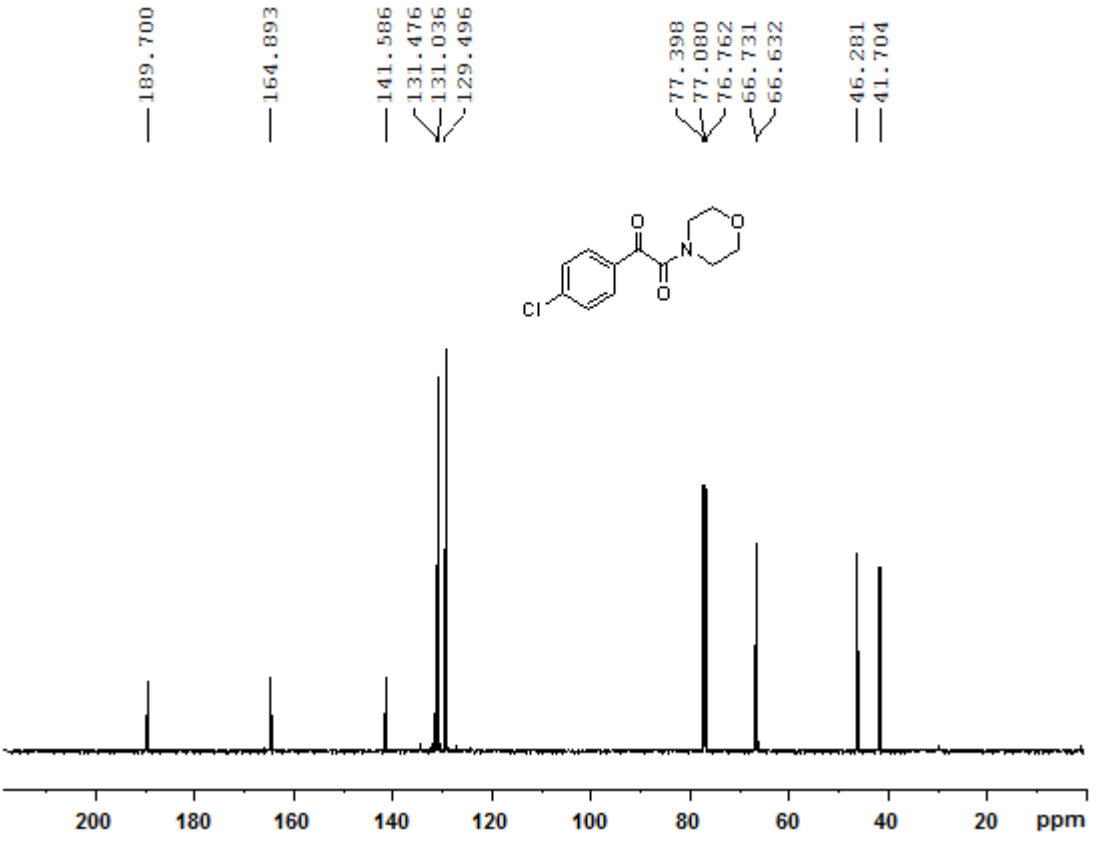
ppm
2.00
2.04
4.09
1.98
1.99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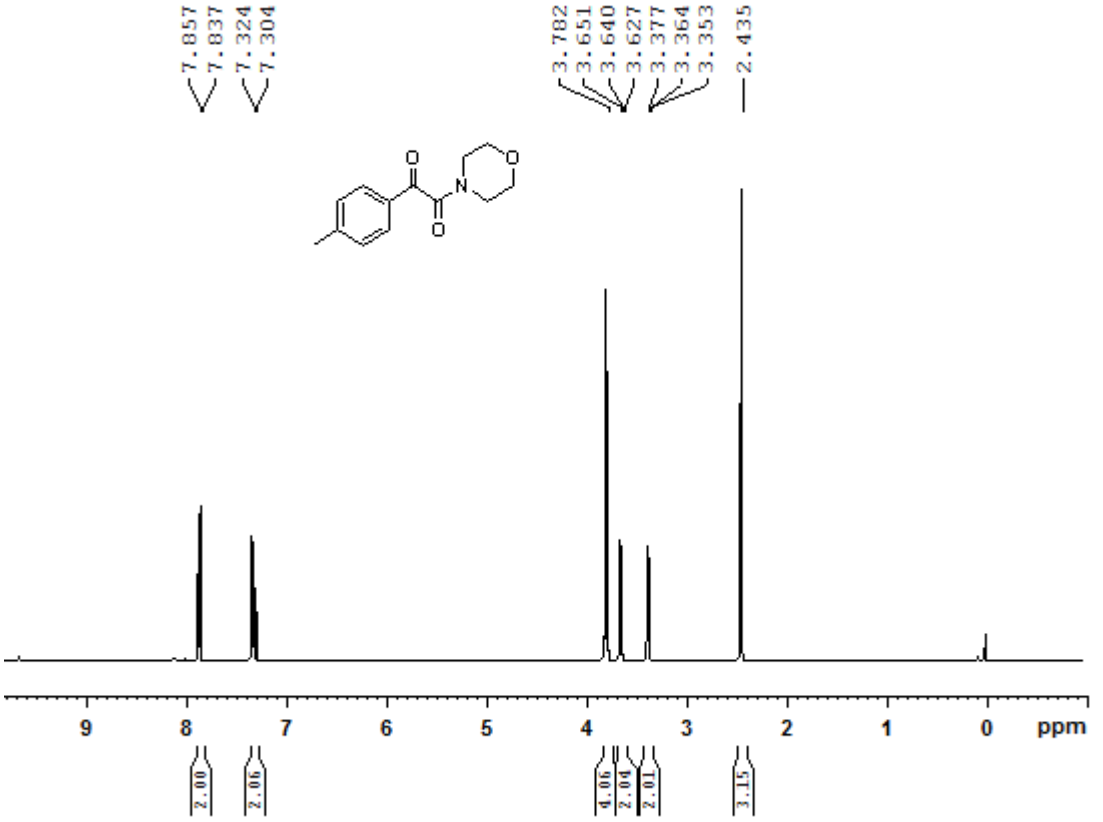
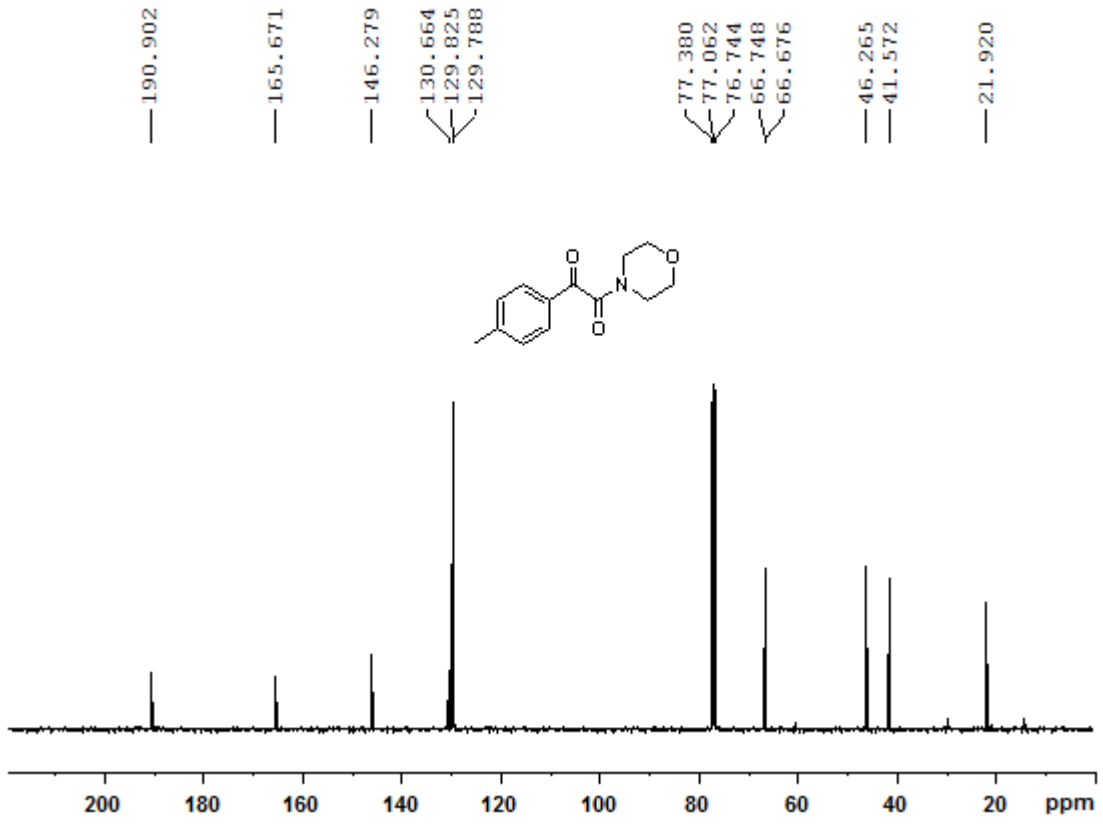
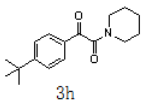
ppm







3h

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

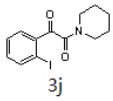
Card Serial Number: GCT-P-T12-04-OS0258
Sample Serial Number: HBSF-Li007
Operator: Li
Date: 2012/03/27
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
846 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-2 O: 0-5 S: 0-2 Cl: 0-2 Br: 0-3

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
273.1728	273.1729	-0.1	-0.4	7.0	2774018.3	C17 H23 N O2
	273.1734	-0.6	-2.2	2.5	2774844.5	C14 H26 N2 O Cl

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS Data Report

Instrument: Waters Micromass GCT Premier Ionisation Mode: EI+ Electron Energy: 70eV

Card Serial Number: GCT-P-T12-03-OS0227
Sample Serial Number: HBSF-LiM2-S2
Operator: Li
Date: 2012/03/27
Elemental Composition Report
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Odd and Even Electron Ions
249 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-60 H: 0-80 N: 0-4 O: 0-6 I: 0-1



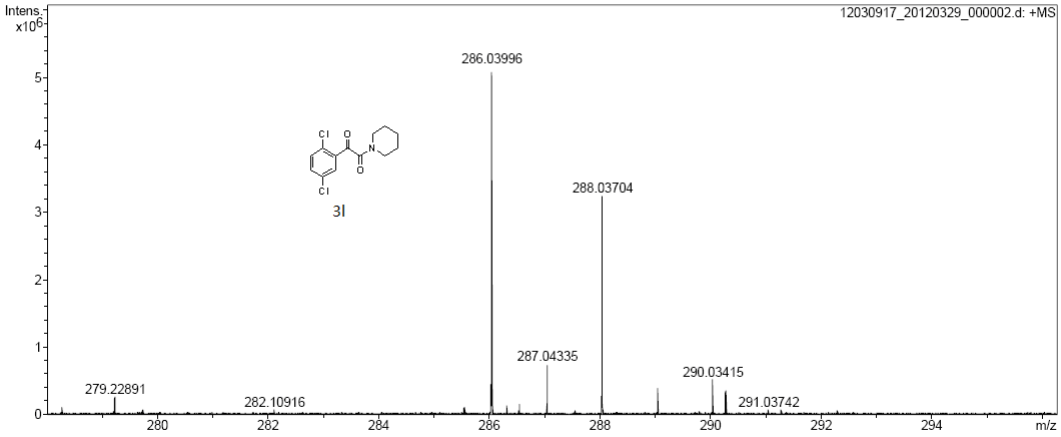
3j

Minimum:				-1.5		
Maximum:		2.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
343.0068	343.0069	-0.1	-0.3	7.0	2773015.3	$C_{13}H_{14}NO_2I$
	343.0058	1.0	2.9	27.0	2773025.0	$C_{26}HNO$
	343.0056	1.2	3.5	7.5	2773014.8	$C_{11}H_{12}N_4OI$

Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	12030917_20120329_000002.d	Acquisition Date	3/29/2012 6:40:40 PM
Sample	wx-zxb-5	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

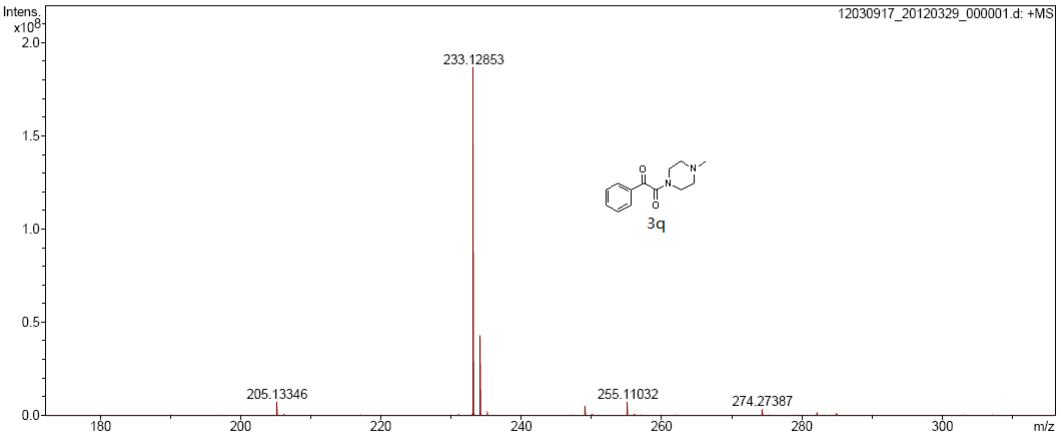


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
286.03996	1	C 13 H 14 Cl 2 N O 2	100.00	286.03961	-0.3	-1.2	12.3	6.5	even	ok

Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	12030917_20120329_000001.d	Acquisition Date	3/29/2012 6:38:28 PM
Sample	wx-zxb-4	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
233.12853	1	C 13 H 17 N 2 O 2	100.00	233.12845	-0.1	-0.3	52.4	6.5	even	ok

3. Reference

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