Electronic Supplementary Material (ESI) for RSC Advances.

Room temperature copper-catalyzed oxidative amidation of terminal alkynes for synthesis of *α*-ketoamides using *O*-benzoyl hydroxylamines as aminating reagent and oxidant

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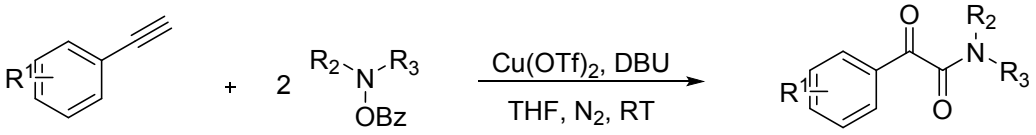
Contents

1. General information

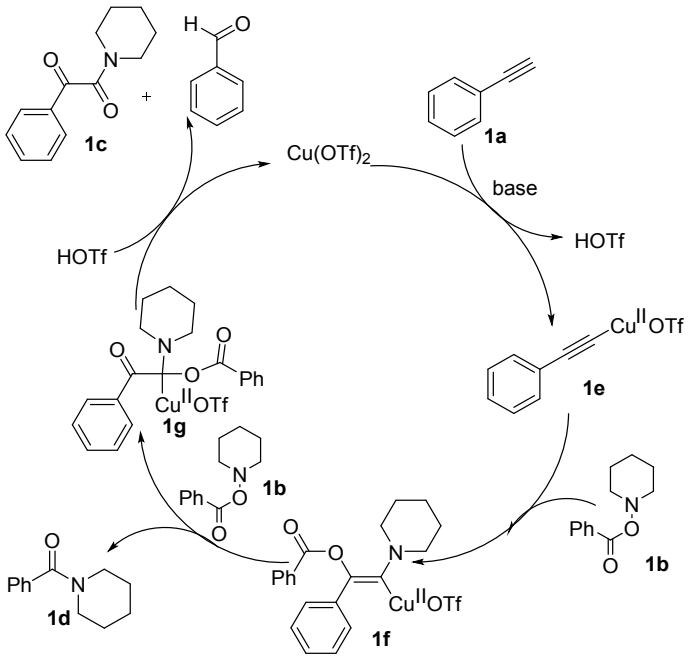
All reagents and solvents were pure analytical grade materials purchased from commercial sources and were used without further purification, if not stated otherwise. THF was anhydrous before using. All starting substrates were prepared according to the known literatures. The NMR spectra were recorded in $CDCl_3$ or d_6-DMSO on a 400 MHz instrument with TMS as internal standard. High-resolution mass spectra (HRMS) were obtained with a Q-TOF Premier (ESI). TLC was carried out with 0.2 mm thick silica gel plates (GF254). Visualization was accomplished by UV light. Column chromatography was hand packed with silica gel (200-300 mesh). All reactions were carried out in an over-dried Schlenk tube equipped with a magnetic stir bar.

2. General procedure and possible reaction mechanism for the synthesis of *α*-ketoamides



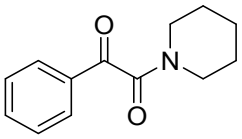
a **b** **c**

An oven-dried Schlenk tube equipped with a Teflon valve was charged with a magnetic stir bar, copper(II) trifluoromethanesulfonate (0.05 mmol, 0.018g), *N*-(benzoyloxy)piperidine **1b** (0.5 mmol, 0.103g). The tube was placed under vacuum for twenty minutes and backfilled with N_2. Then anhydrous THF (3.0 mL), phenylacetylene **1a** (2.0 mmol, 0.204g) and DBU (1.0 mmol, 0.152g) were added under N_2 atmosphere. The reaction mixture was stirred for 12 h at room temperature. The reaction was monitored by TLC. When *N*-(benzoyloxy)piperidine **1b** consumed completely, the resulting suspension was filtered through a pad of filter paper with 20 mL of ethyl acetate for 3 times. After evaporating the solvent under reduced pressure, the residue was purified by column chromatography on silica gel to give the product **1c** in 84% yield (46 mg).

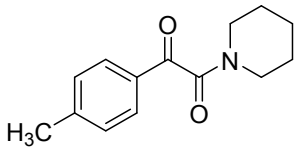


Based on the above information, a plausible mechanism for this reaction is proposed. The Cu(II)-acetylide species **1e** was formed by the reaction of the copper(II) trifluoromethanesulfonate with phenylacetylene **1a** in the presence of DBU. The addition reaction of the Cu(II)-acetylide species **1e** with *N*-(benzoyloxy)piperidine **1b** would lead to the formation of the intermediate **1f** and some **1d**. Then another *N*-(benzoyloxy)piperidine **1b** attack the intermediate **1f** to get the intermediate **1g**, the obtained intermediates **1g** finally converted into the products **1c**.

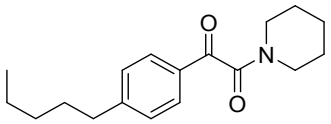
3. Date of the products



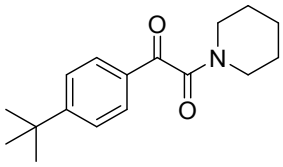
1-phenyl-2-(piperidin-1-*yl*)ethane-1,2-dione (**1c**).[1] Mp: 104-107 ^{o}C. Compound **1c** (46 mg, 84% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow solid. ^{1}H NMR (400M Hz, $CDCl_3$/TMS): δ 7.93 (d, *J* = 8.0 Hz, 2H), δ 7.62 (t, *J* = 8.0 Hz, 1H), δ 7.49 (t, *J* = 8.0 Hz, 2H), δ 3.69 (m, 2H), δ 3.27 (m, 2H), δ 1.68-1.53 (m, 6H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 192.04, 165.53, 134.76, 133.32, 129.65, 129.09, 47.13, 42.24, 26.29, 25.54, 24.46. HRMS (ESI): *m/z* calcd for $C_{13}H_{16}NO_2$ $[M+H]^+$: 218.1181, found: 218.1175.



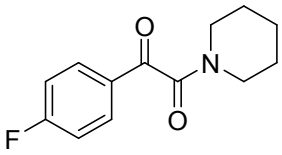
1-(piperidin-1-*yl*)-2-(p-tolyl)ethane-1,2-dione (**2c**).[2] Compound **2c** (47 mg, 82% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.83 (d, *J* = 8.0 Hz, 2H), δ 7.30 (d, *J* = 8.0 Hz, 2H), δ 3.69 (m, 2H), δ 3.27 (m, 2H), δ 1.68-1.53 (m, 6H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.83, 165.76, 146.01, 130.94, 130.10, 129.81, 47.15, 42.21, 26.32, 25.57, 24.51, 22.04. HRMS (ESI): *m/z* calcd for $C_{14}H_{18}NO_2$ $[M+H]^+$: 232.1338, found: 232.1339.



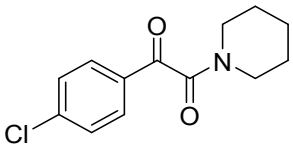
1-(4-pentylphenyl)-2-(piperidin-1-*yl*)ethane-1,2-dione (**3c**). Compound **3c** (46 mg, 64% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.84 (d, *J* = 8.0 Hz, 2H), δ 7.30 (d, *J* = 8.0 Hz, 2H), δ 3.69 (m, 2H), δ 3.28 (m, 2H), δ 2.66 (t, *J* = 8.0 Hz, 2H), δ 1.68-1.54 (m, 8H), δ 1.31 (m, 4H), δ 0.88 (t, *J* = 7.8 Hz, 3H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.85, 165.78, 150.96, 131.04, 129.85, 129.21, 47.16, 42.20, 36.28, 31.50, 30.85, 26.33, 25.57, 24.51, 22.61, 14.14. HRMS (ESI): *m/z* calcd for $C_{18}H_{26}NO_2$ $[M+H]^+$: 288.1964, found: 288.1960.



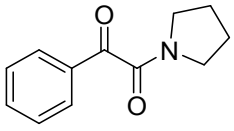
1-(4-(tert-butyl)phenyl)-2-(piperidin-1-*yl*)ethane-1,2-dione (**4c**).[1] Compound **4c** (49 mg, 72% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.87 (d, *J* = 8.0 Hz, 2H), δ 7.51 (d, *J* = 8.0 Hz, 2H), δ 3.69 (m, 2H), δ 3.28 (m, 2H), δ 1.68-1.54 (m, 6H), δ 1.33 (s, 9H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.81, 165.77, 158.83, 130.80, 129.67, 126.13, 47.16, 42.21, 35.48, 31.12, 26.36, 25.58, 24.51. HRMS (ESI): *m/z* calcd for $C_{17}H_{24}NO_2$ $[M+H]^+$: 274.1807, found: 274.1809.



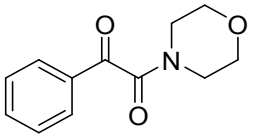
1-(4-fluorophenyl)-2-(piperidin-1-*yl*)ethane-1,2-dione (**5c**). Compound **5c** (47 mg, 80% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.99-7.95 (m, 2H), δ 7.20-7.15 (m, 2H), δ 3.68 (m, 2H), δ 3.28 (m, 2H), δ 1.68-1.54 (m, 6H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 190.35, 167.99, 165.31 (d, J = 25 Hz), 132.48 (d, J = 10Hz), 129.83, 116.45 (d, J = 22Hz), 47.16, 42.30, 26.33, 25.54, 24.43. HRMS (ESI): *m/z* calcd for $C_{13}H_{15}FNO_2$ $[M+H]^+$: 236.1087, found: 236.1081.



1-(4-chlorophenyl)-2-(piperidin-1-*yl*)ethane-1,2-dione (**6c**).[1] Compound **6c** (49 mg, 78% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.89 (d, J = 8.0 Hz, 2H), δ 7.48 (d, J = 8.0 Hz, 2H), δ 3.69 (m, 2H), δ 3.27 (m, 2H), δ 1.69-1.55 (m, 6H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 190.67, 165.03, 141.38, 131.75, 131.05, 129.53, 47.19, 42.37, 26.38, 25.58, 24.37. HRMS (ESI): *m/z* calcd for $C_{13}H_{15}ClNO_2$ $[M+H]^+$: 252.0791, found: 252.0785.

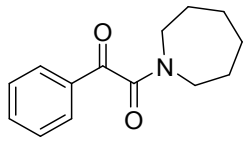


1-phenyl-2-(pyrrolidin-1-*yl*)ethane-1,2-dione (**7c**).[3] Compound **7c** (41 mg, 80% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow solid. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 8.00 (d, J = 7.2 Hz, 2H), δ 7.64 (t, J = 7.2 Hz, 1H), δ 7.51 (t, J = 7.2 Hz, 2H), δ 3.66 (m, 2H), δ 3.43 (m, 2H), δ 1.96 (m, 4H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.70, 165.05, 134.73, 133.06, 130.04, 129.06, 46.83, 45.40, 26.07, 24.18. HRMS (ESI): *m/z* calcd for $C_{12}H_{14}NO_2$ $[M+H]^+$: 204.1025, found: 204.1022.

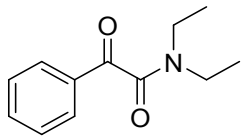


1-morpholino-2-phenylethane-1,2-dione (**8c**).[3] Mp: 50-51 °C. Compound **8c** (39 mg, 72% yield) was purified by flash column chromatography

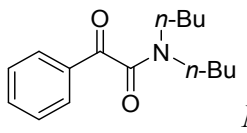
(PE/Et_3N = 5:1) as yellow solid. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.96 (d, *J* = 8.0 Hz, 2H), δ 7.66 (t, *J* = 8.0 Hz, 1H), δ 7.53 (t, *J* = 8.0 Hz, 2H), δ 3.80 (m, 4H), δ 3.65 (m, 2H), δ 3.38 (m, 2H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.30, 165.55, 135.13, 133.08, 129.81, 129.25, 66.87, 66.80, 46.38, 41.72. HRMS (ESI): *m/z* calcd for $C_{12}H_{14}NO_3$ $[M+H]^+$: 220.0974, found: 220.0970.



1-(azepan-1-*yl*)-2-phenylethane-1,2-dione (**9c**).[4] Compound **9c** (36 mg, 62% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.93 (d, *J* = 8.0 Hz, 2H), δ 7.62 (t, *J* = 8.0 Hz, 1H), δ 7.49 (t, *J* = 8.0 Hz, 2H), δ 3.67 (t, *J* = 6.0 Hz, 2H), δ 3.32 (t, *J* = 6.0 Hz, 2H), δ 1.85-1.80 (m, 2H), δ 1.68-1.57 (m, 6H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.95, 167.26, 134.70, 133.24, 129.72, 129.06, 48.03, 45.21, 29.09, 27.70, 27.28, 26.62. HRMS (ESI): *m/z* calcd for $C_{14}H_{18}NO_2$ $[M+H]^+$: 232.1338, found: 232.1339.

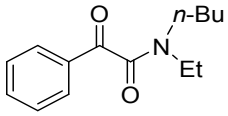


N, *N*-diethyl-2-oxo-2-phenylacetamide (**10c**).[1] Compound **10c** (35 mg, 68% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.93 (d, *J* = 8.0 Hz, 2H), δ 7.63 (t, *J* = 8.0 Hz, 1H), δ 7.50 (t, *J* = 8.0 Hz, 2H), δ 3.56 (q, *J* = 8.0 Hz, 2H), δ 3.24 (q, *J* = 8.0 Hz, 2H), δ 1.28 (t, *J* = 8.0 Hz, 3H), δ 1.15 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.73, 166.84, 134.72, 133.33, 129.75, 129.09, 42.22, 38.89, 14.25, 12.99. HRMS (ESI): *m/z* calcd for $C_{12}H_{16}NO_2$ $[M+H]^+$: 206.1181, found: 206.1185.

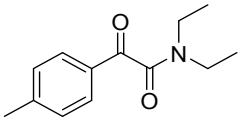


N, *N*-dibutyl-2-oxo-2-phenylacetamide (**11c**).[2] Compound **11c** (50 mg, 76% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^1H NMR (400M Hz, $CDCl_3$/TMS): δ 7.93 (d, *J* = 8.0 Hz, 2H), δ 7.63 (t, *J* = 8.0 Hz, 1H), δ 7.50 (t, *J* = 8.0 Hz, 2H), δ 3.49 (t, *J* = 8.0 Hz, 2H), δ 3.14 (t, *J* = 8.0 Hz, 2H), δ 1.68-1.63 (m, 2H), δ 1.55-1.49 (m, 2H), δ 1.44-1.38 (m, 2H), δ 1.21-1.15

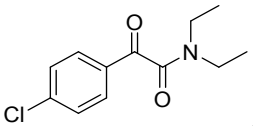
(m, 2H), δ 0.99 (q, *J* = 8.0 Hz, 3H), δ 0.81 (q, *J* = 8.0 Hz, 3H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.73, 167.19, 134.63, 133.49, 129.75, 129.06, 47.57, 44.15, 30.76, 29.59, 20.40, 19.91, 14.01, 13.71. HRMS (ESI): *m/z* calcd for $C_{16}H_{24}NO_2$ $[M+H]^+$: 262.1807, found: 262.1801.



N-butyl-*N*-ethyl-2-oxo-2-phenylacetamide (**12c**). Compound **12c** (35 mg, 60% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^{1}H NMR (400M Hz, $CDCl_3$/TMS): δ 7.92 (d, *J* = 8.0 Hz, 4H), δ 7.62 (t, *J* = 8.0 Hz, 2H), δ 7.49 (t, *J* = 8.0 Hz, 4H), δ 3.55 (q, *J* = 8.0 Hz, 2H), δ 3.45 (t, *J* = 8.0 Hz, 2H), δ 3.22 (q, *J* = 8.0 Hz, 2H), δ 3.14 (t, *J* = 8.0 Hz, 2H), δ 1.70-1.62 (m, 2H), δ 1.53-1.49 (m, 2H), δ 1.44-1.36 (m, 4H), δ 1.27 (t, *J* = 8.0 Hz, 3H), δ 1.14 (t, *J* = 8.0 Hz, 3H), δ 0.98 (t, *J* = 8.0 Hz, 3H), δ 0.80 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.75, 191.64, 167.08, 166.95, 134.64, 134.63, 133.45, 133.37, 129.72, 129.69, 129.05, 129.04, 47.33, 43.69, 42.42, 39.35, 30.81, 29.66, 20.35, 19.86, 14.11, 13.96, 13.66, 12.84. HRMS (ESI): *m/z* calcd for $C_{14}H_{20}NO_2$ $[M+H]^+$: 234.1494, found: 234.1490.



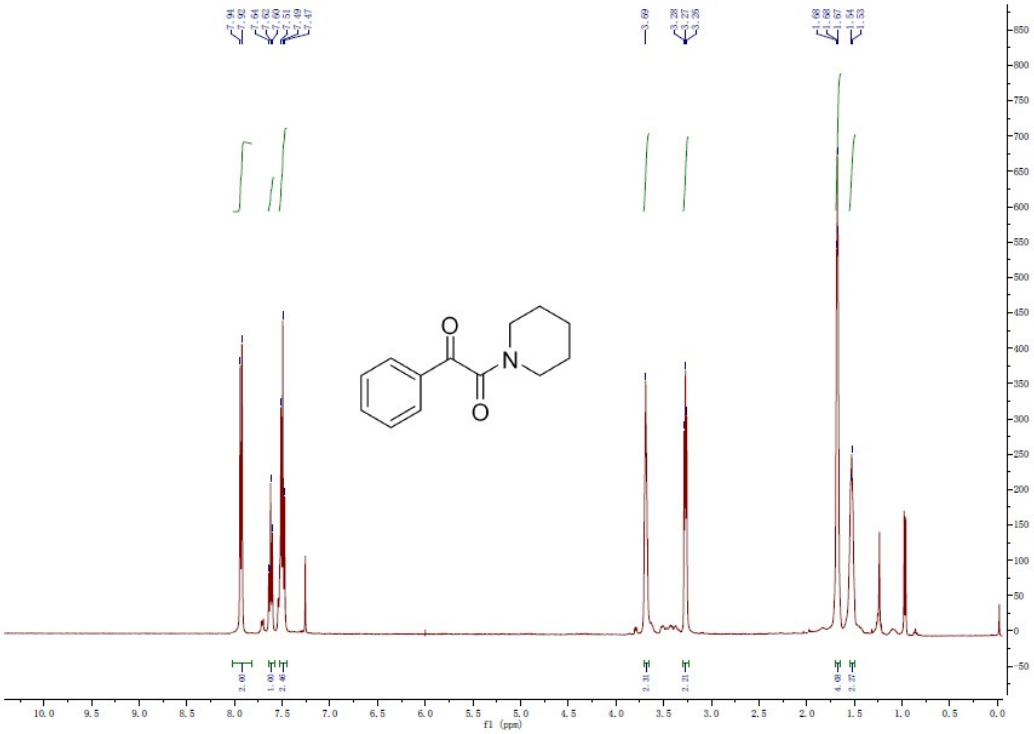
N, *N*-diethyl-2-oxo-2-(*p*-tolyl)acetamide (**13c**). Compound **13c** (36 mg, 65% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^{1}H NMR (400M Hz, $CDCl_3$/TMS): δ 7.82 (d, *J* = 8.0 Hz, 2H), δ 7.29 (d, *J* = 8.0 Hz, 2H) , δ 3.55 (q, *J* = 8.0 Hz, 2H), δ 3.23 (q, *J* = 8.0 Hz, 2H), δ 2.42 (s, 3H), δ 1.27 (t, *J* = 8.0 Hz, 3H), δ 1.14 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 191.50, 167.04, 145.91, 130.92, 129.87, 129.80, 42.21, 38.83, 22.04, 14.24, 12.98. HRMS (ESI): *m/z* calcd for $C_{13}H_{18}NO_2$ $[M+H]^+$: 220.1338, found: 220.1339.

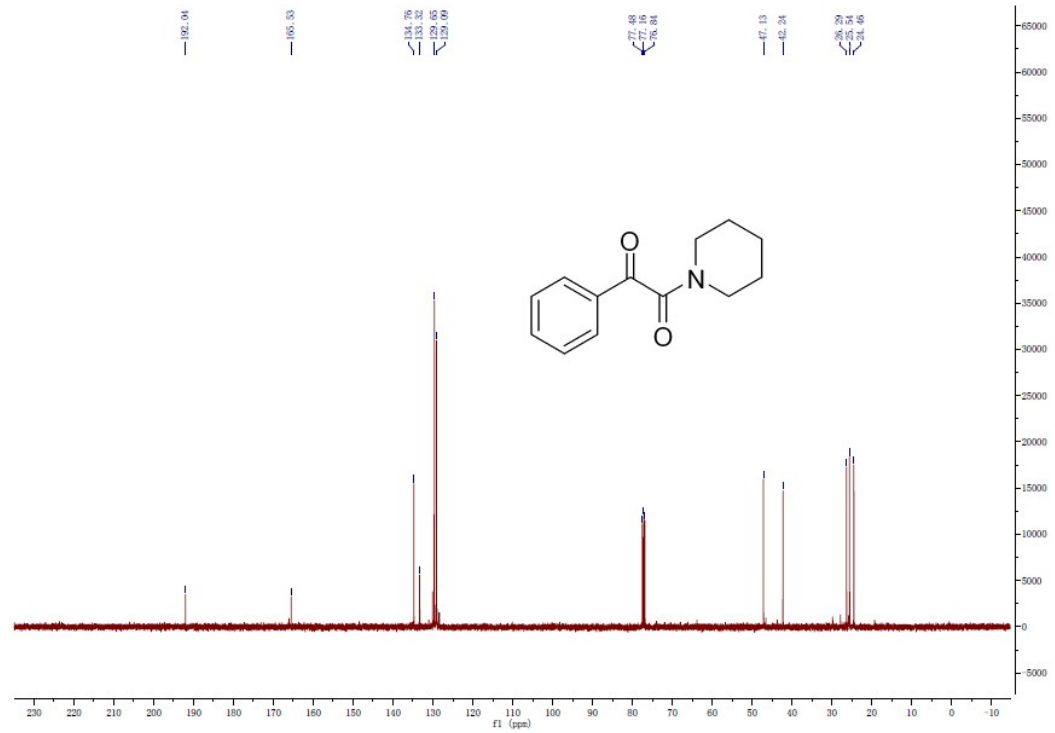


2-(4-chlorophenyl)-*N*, *N*-diethyl-2-oxoacetamide (**14c**). Compound **14c** (36 mg, 61% yield) was purified by flash column chromatography (PE/Et_3N = 5:1) as yellow oil. ^{1}H NMR (400M Hz, $CDCl_3$/TMS): δ 7.88 (d, *J* = 12.0

Hz, 2H), δ 7.48 (d, *J* = 12.0 Hz, 2H), δ 3.55 (q, *J* = 8.0 Hz, 2H), δ 3.23 (q, *J* = 8.0 Hz, 2H), δ 1.27 (t, *J* = 8.0 Hz, 3H), δ 1.15 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100M Hz, $CDCl_3$/TMS): δ 190.30, 166.32, 141.29, 131.74, 131.10, 129.49, 42.27, 39.04, 14.32, 12.98. HRMS (ESI): *m/z* calcd for $C_{12}H_{15}ClNO_2$ $[M+H]^+$: 240.0791, found: 240.0799.

4. Copies of NMR spectra

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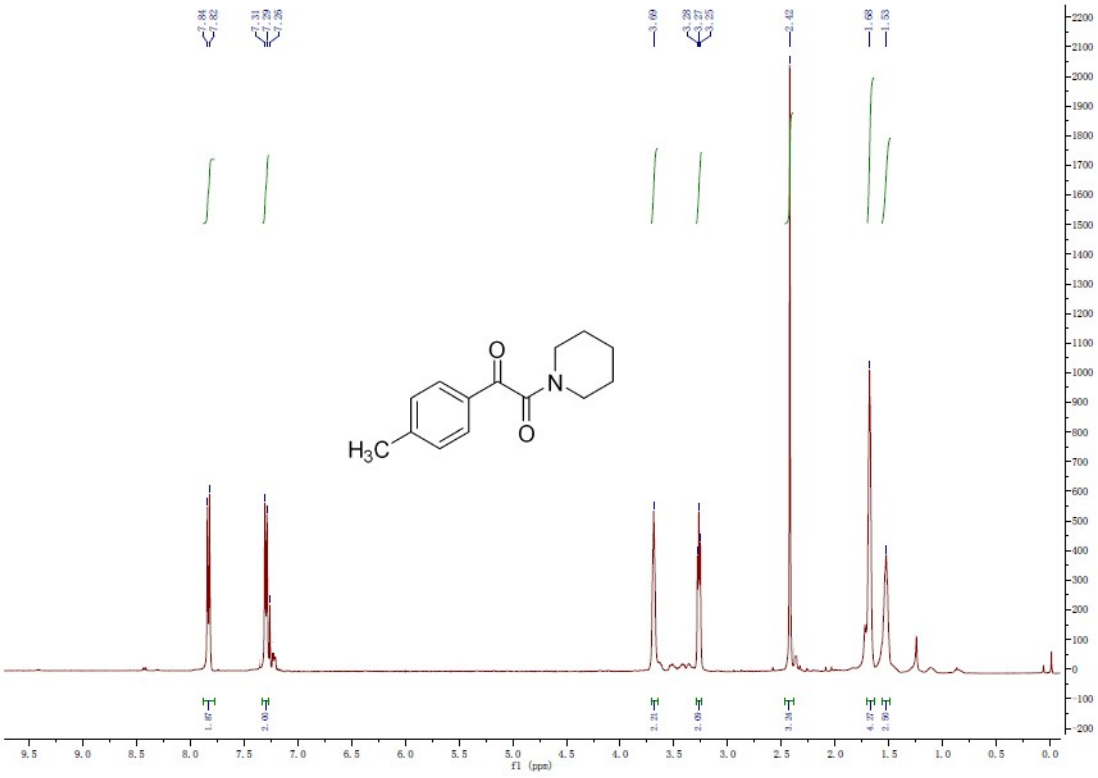
N
O
f1 (ppm)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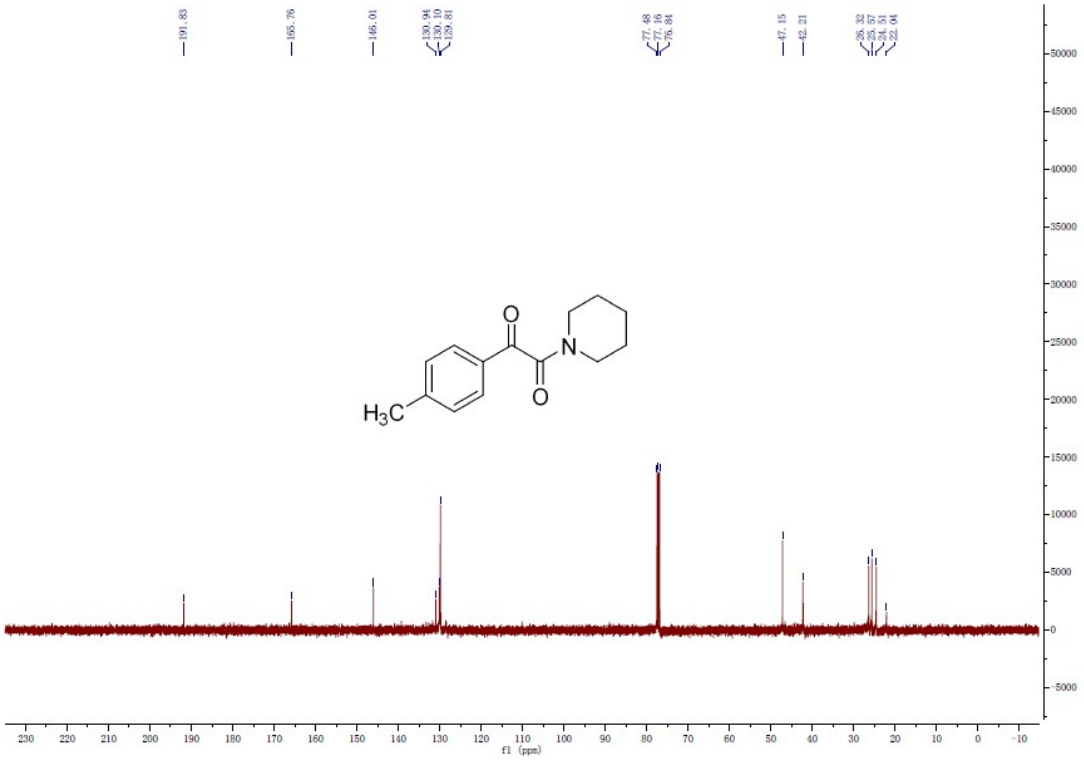
2.90
f1 (ppm)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130.10
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47.15
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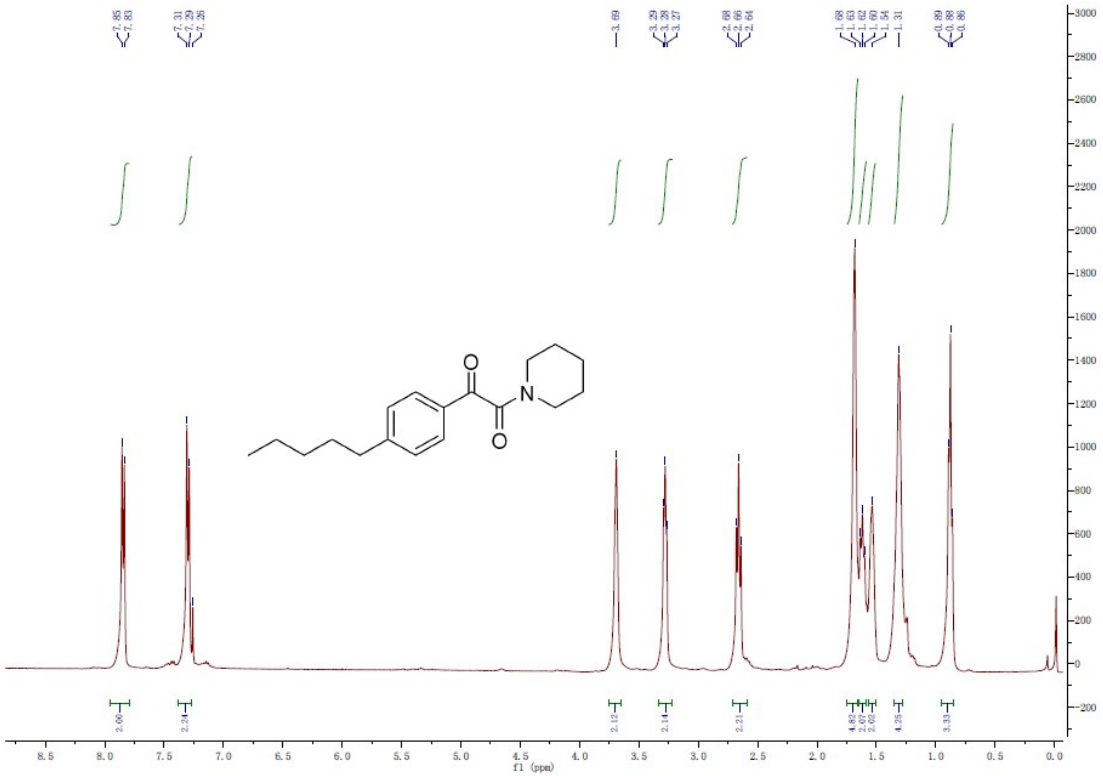
24.51
22.04
O
N
O
H3C
f1 (ppm)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7.83
7.31
7.29
7.25
3.69
3.29
3.28
3.27
2.68
2.66
2.64
1.68
1.63
1.62
1.60
1.54
1.31
0.89
0.88
0.86
O
N
O
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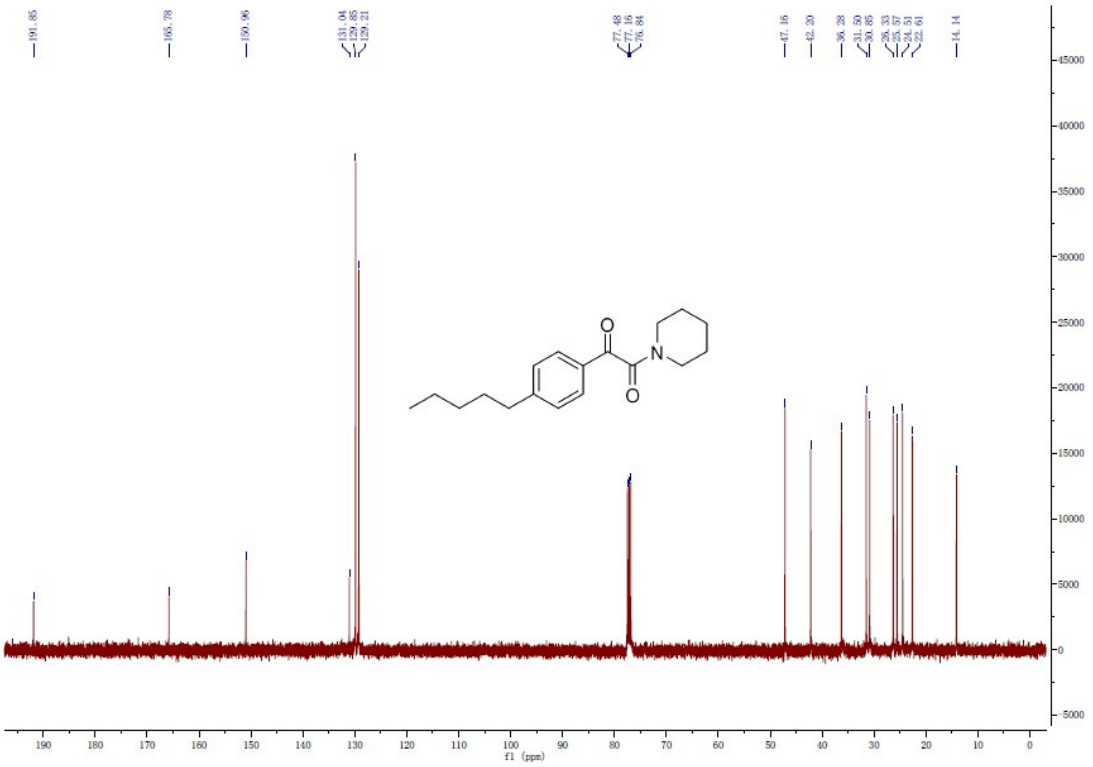
2.04
2.12
2.14
2.21
4.82
2.07
2.00
4.35
3.33
f1 (ppm)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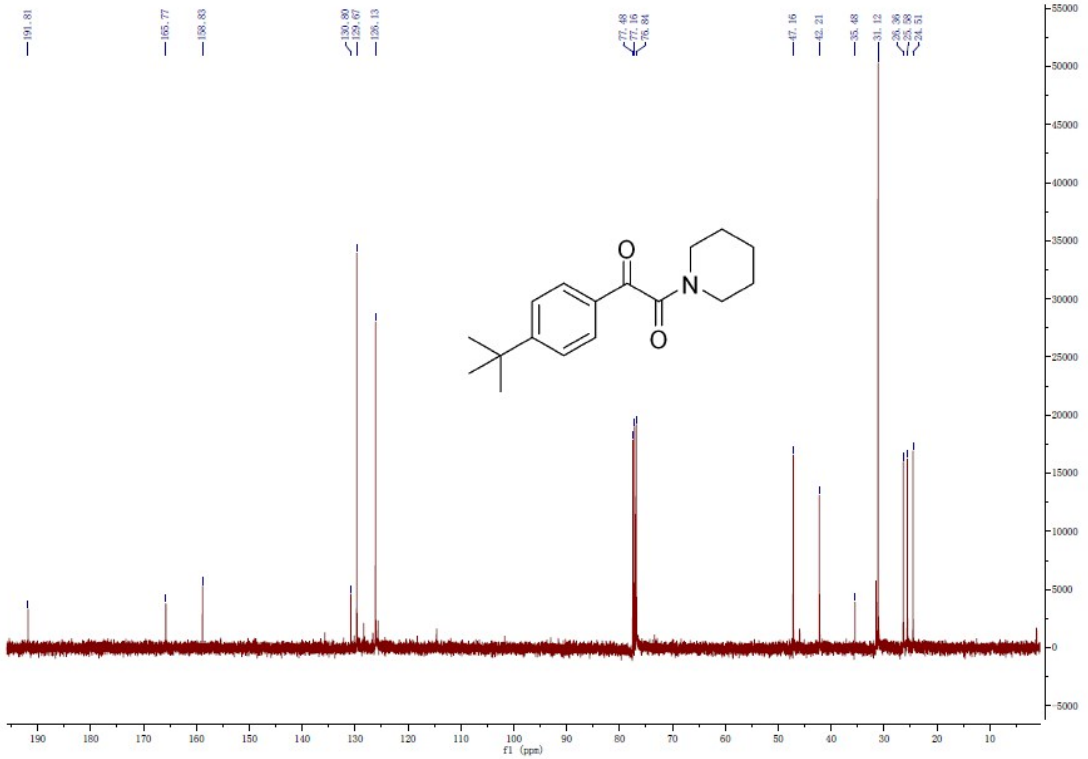
150.96
131.04
129.95
129.21
77.48
77.16
76.84
67.16
62.20
36.28
31.50
30.85
26.33
25.57
24.51
22.61
14.14
O
N
O
f1 (ppm)


7.88
7.86
7.52
7.50
7.25
3.69
3.30
3.28
3.27
1.68
1.68
1.54
1.33
O
N
O
2.00
2.00
2.10
2.04
4.21
2.28
9.47
f1 (ppm)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77.16
76.84
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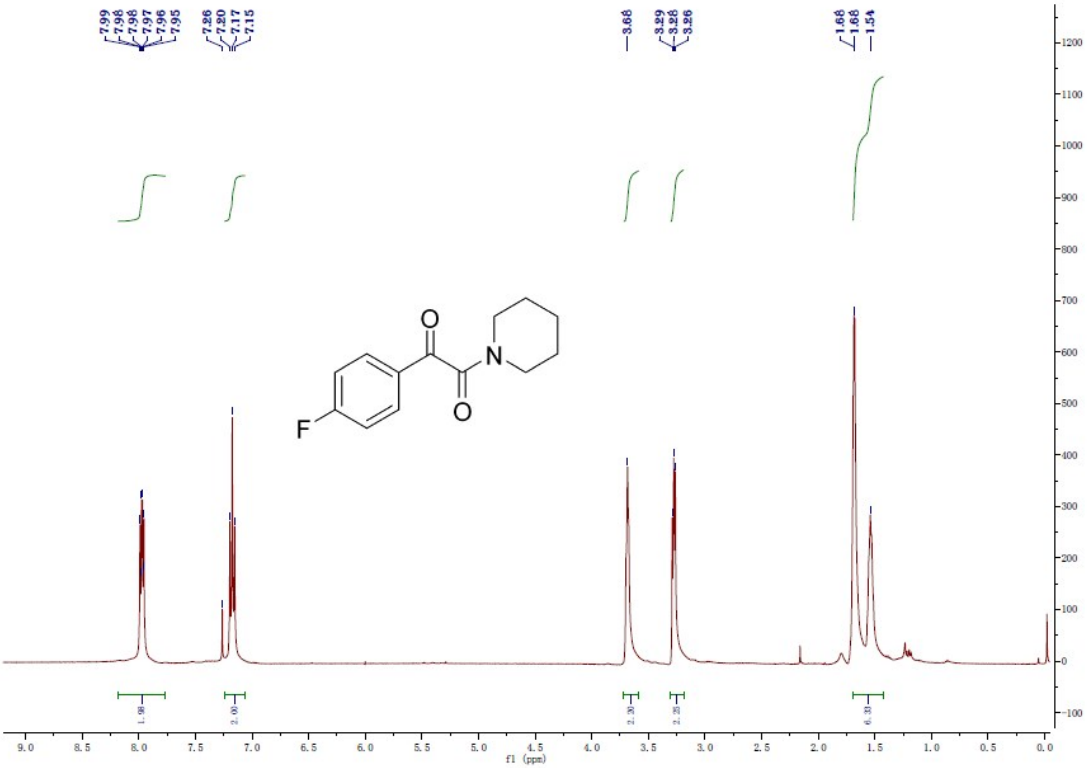
42.21
35.48
31.12
26.36
25.89
24.51
O
N
O
f1 (ppm)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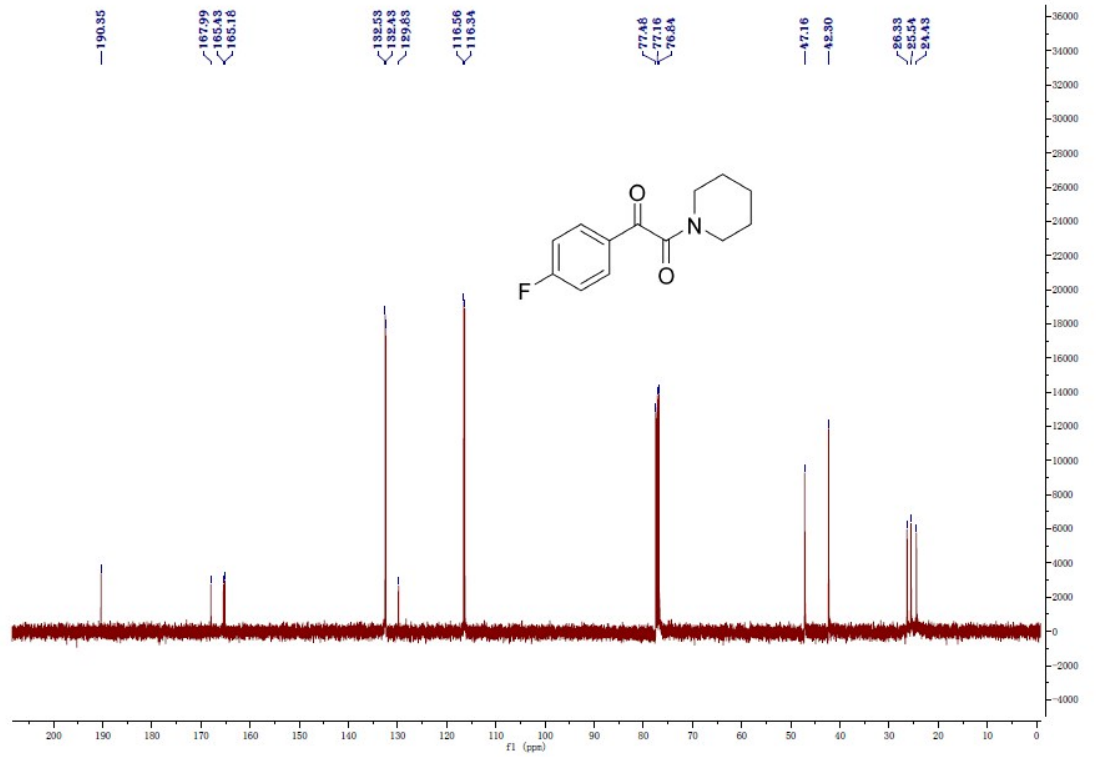
7.96
7.95
7.26
7.20
7.17
7.15
3.68
3.29
3.26
3.26
1.66
1.66
1.54
O
N
O
F
1.98
2.00
2.30
2.25
6.30
f1 (ppm)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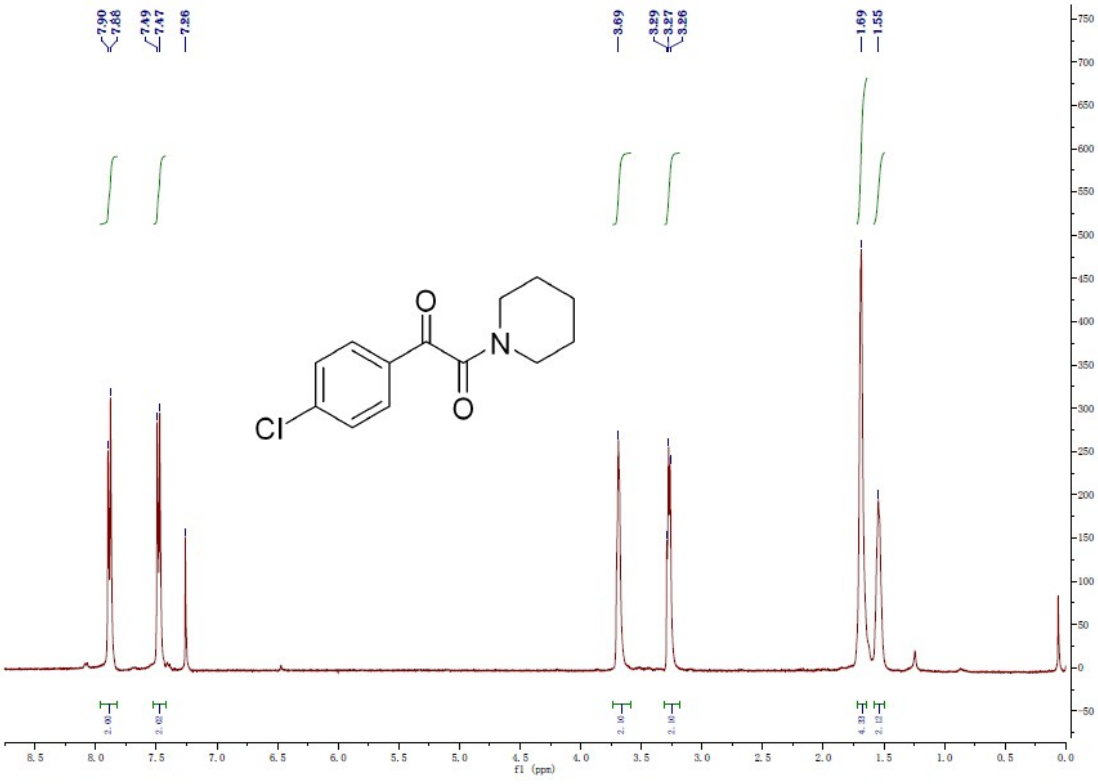
167.99
165.43
165.18
132.53
132.43
129.83
116.56
116.34
77.48
77.16
76.84
47.16
42.30
26.33
25.54
24.43
F
O
N
O
f1 (ppm)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7.88
7.49
7.47
7.26
3.69
3.89
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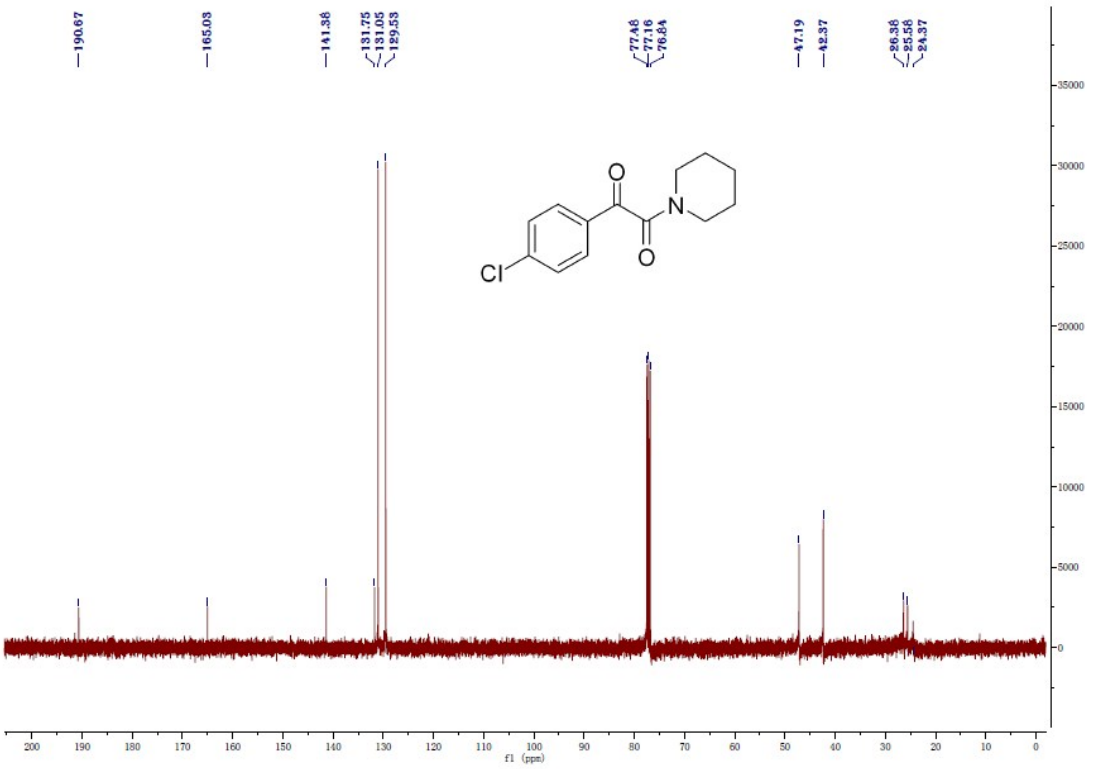
3.26
1.69
1.55
Cl
O
N
O
f1 (ppm)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141.38
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131.05
129.53
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77.16
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47.19
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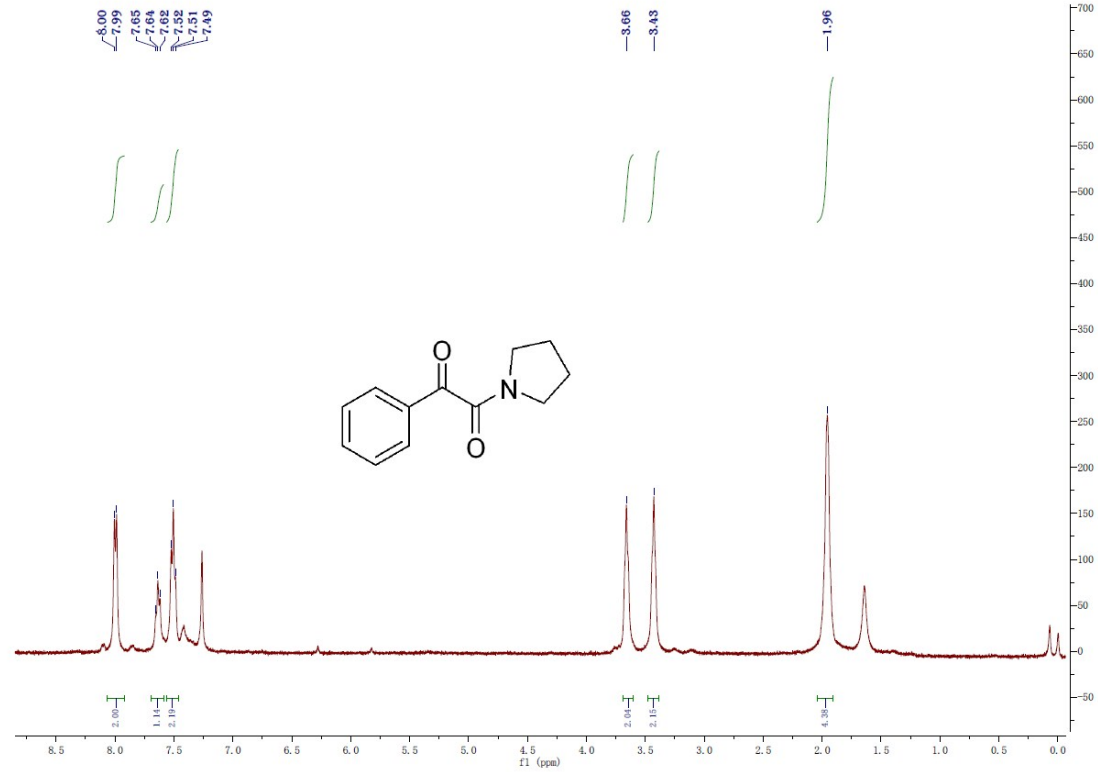
26.38
25.56
24.37
O
N
O
Cl
f1 (ppm)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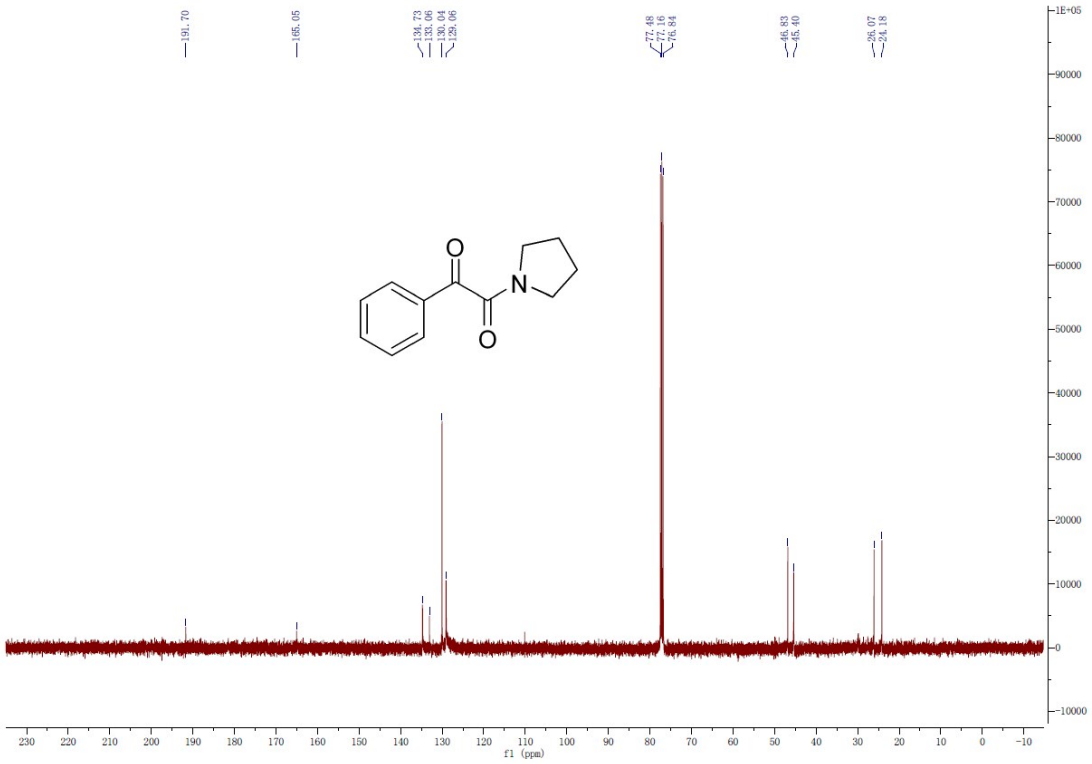
7.99
7.65
7.64
7.62
7.52
7.51
7.49
3.66
3.43
1.96
O
N
O
2.00
1.14
2.10
2.04
2.15
4.38
f1 (ppm)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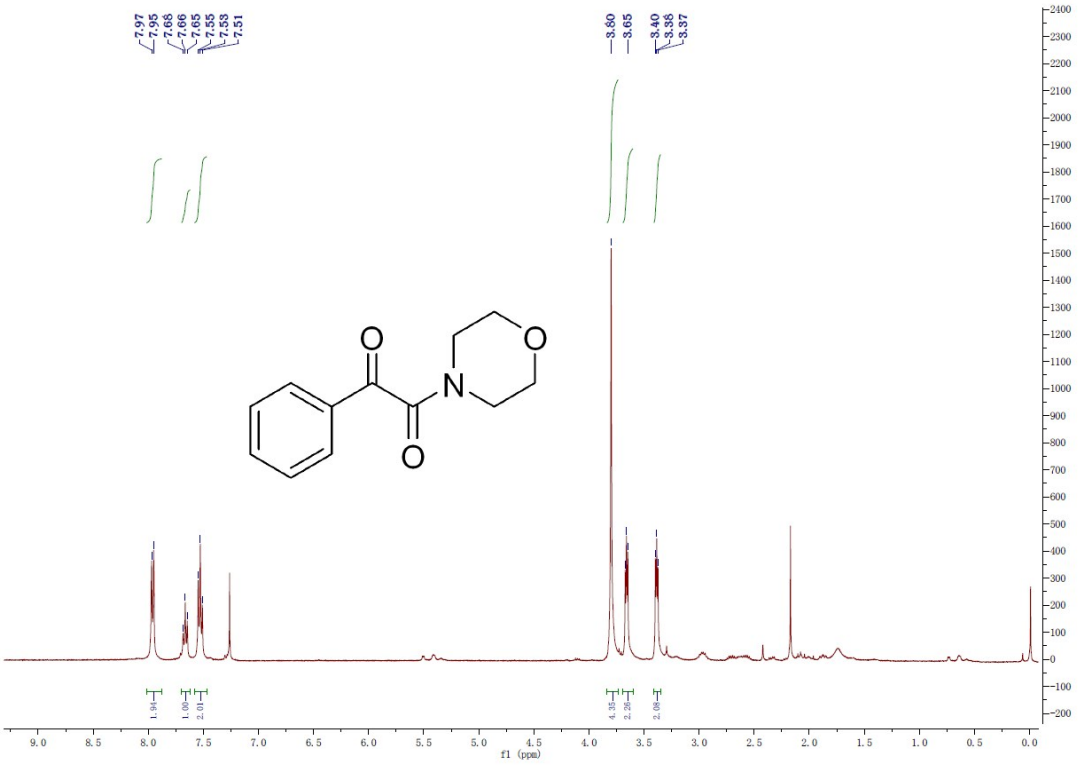
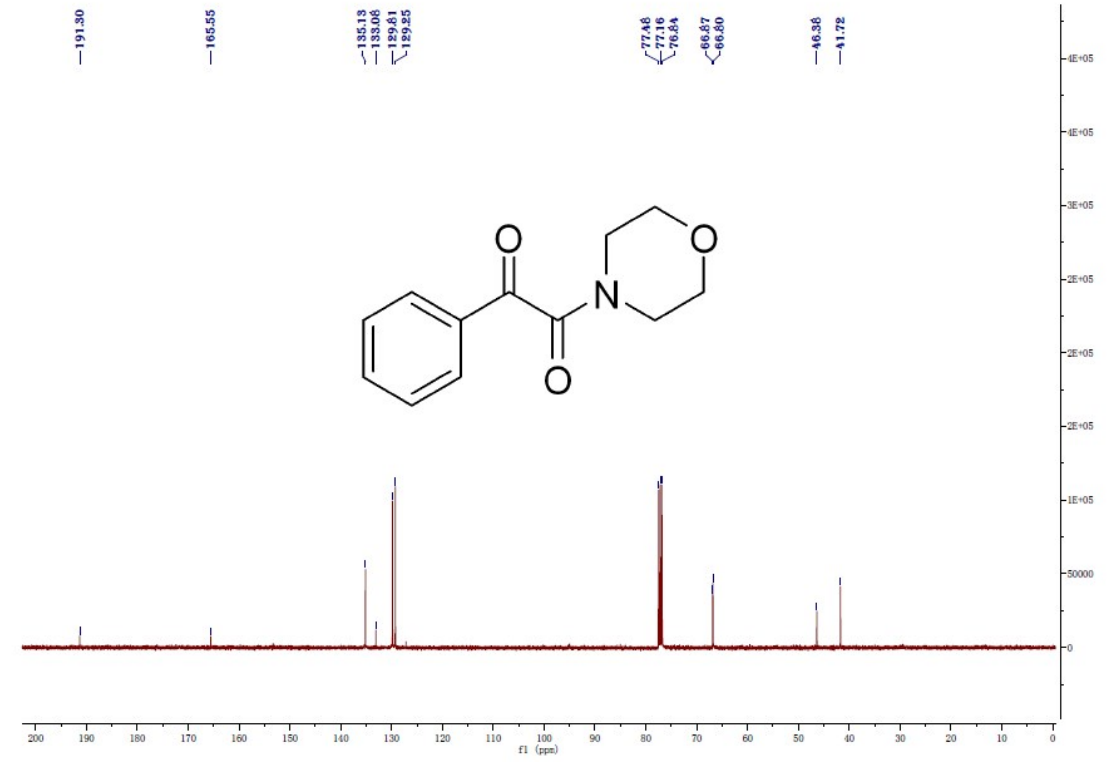
165.05
134.73
133.06
130.04
129.06
77.48
77.16
76.84
46.83
45.40
26.07
24.18
O
N
O
f1 (ppm)


7.97
7.95
7.68
7.66
7.65
7.55
7.53
7.51
3.80
3.65
3.40
3.38
3.37
O
O
N
O
1.94
1.00
2.01
4.35
2.26
2.08
f1 (ppm)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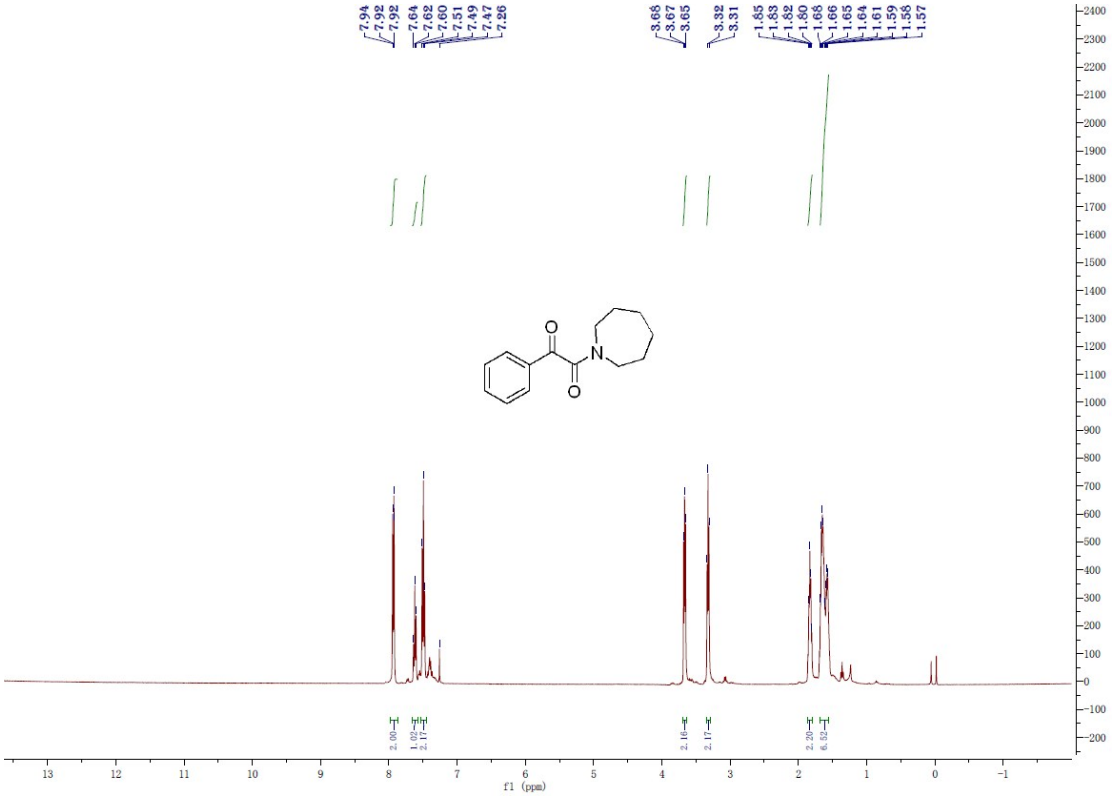
76.84
66.87
66.80
46.36
41.72
f1 (ppm)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7.49
7.47
7.26
3.68
3.67
3.65
3.32
3.31
1.85
1.83
1.82
1.80
1.68
1.66
1.65
1.64
1.61
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1.57
2.00
1.02
2.17
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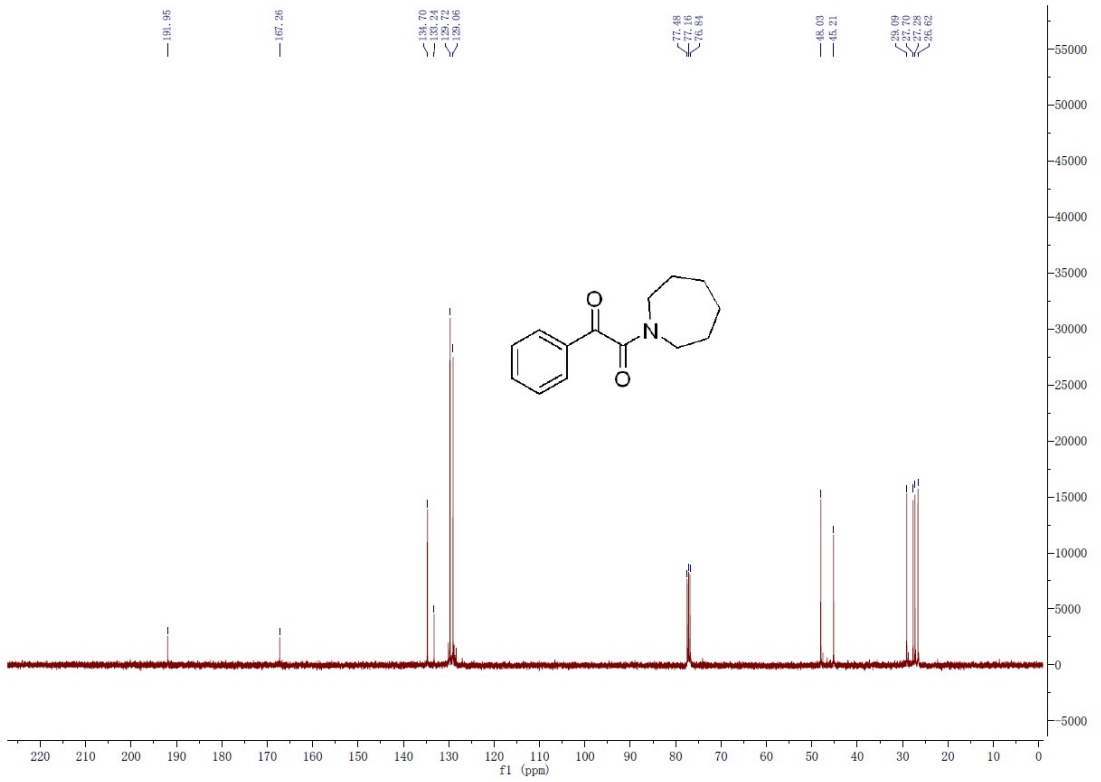
2.17
2.20
6.52
f1 (ppm)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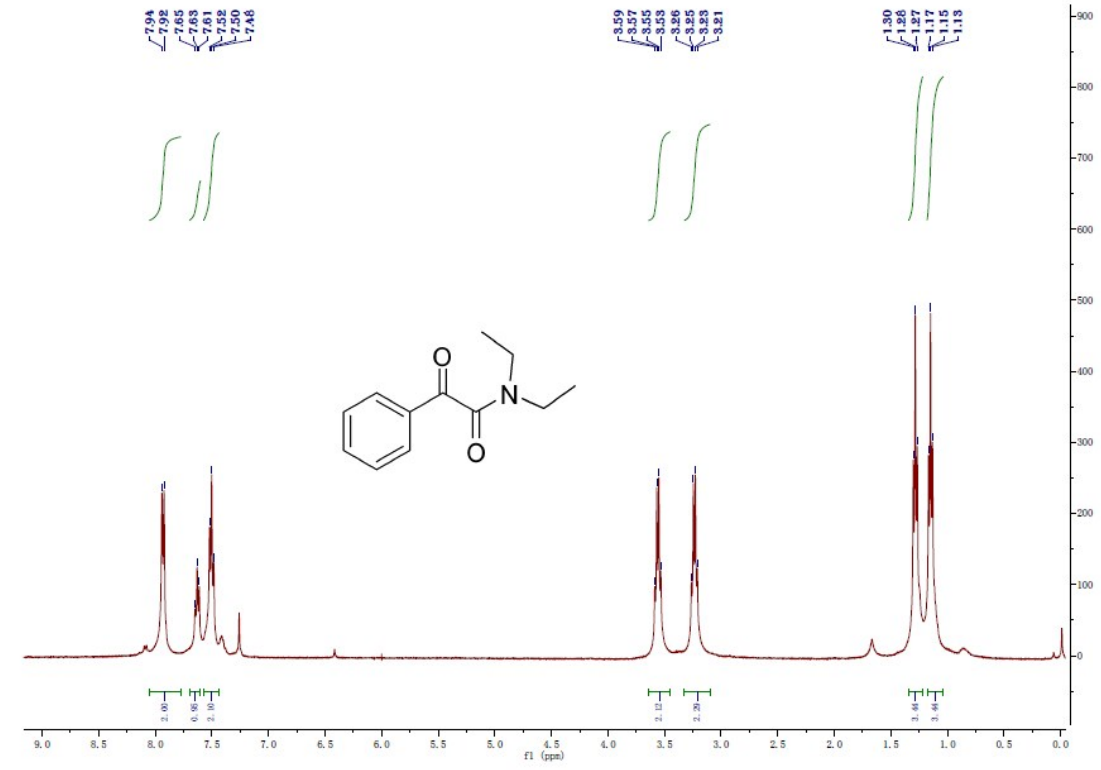
134.70
133.24
128.72
128.06
77.48
77.16
76.84
48.03
45.21
29.09
27.70
27.28
26.62
O
N
O
f1 (ppm)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7.61
7.58
7.50
7.48
3.59
3.57
3.55
3.53
3.26
3.25
3.23
3.21
1.30
1.28
1.27
1.17
1.15
1.13
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N
O
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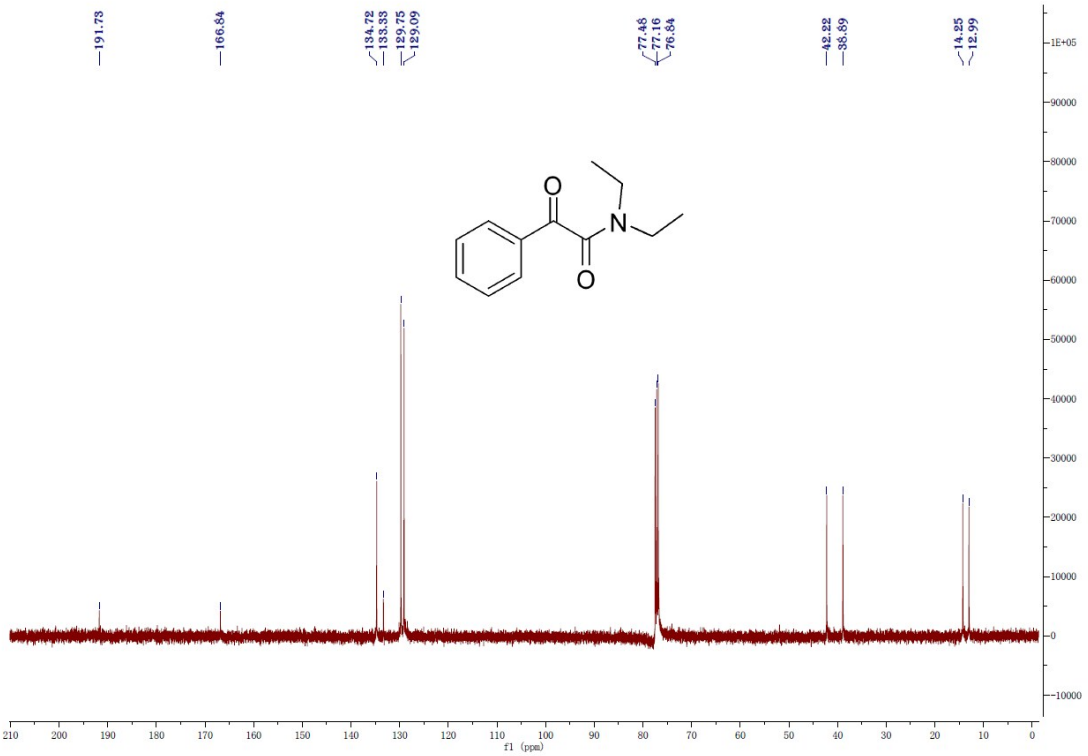
2.10
2.12
2.20
3.44
3.44
f1 (ppm)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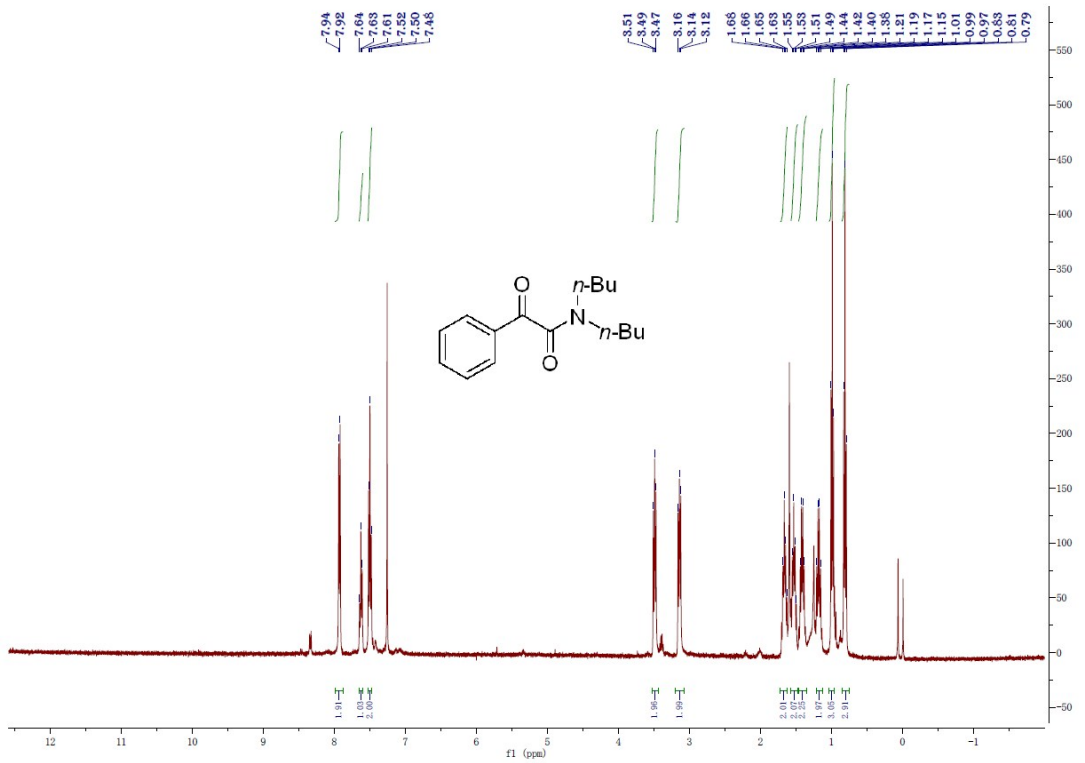
12.99
O
N
O
f1 (ppm)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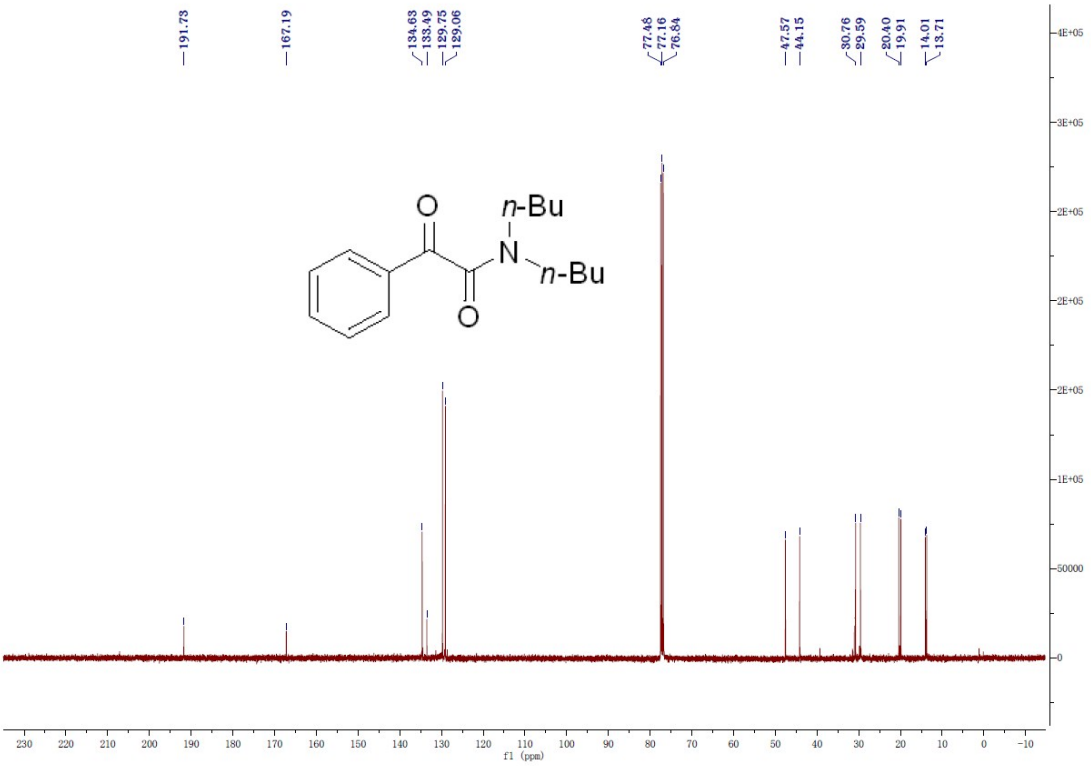
3.49
3.47
3.16
3.14
3.12
1.68
1.66
1.65
1.63
1.55
1.53
1.51
1.49
1.44
1.42
1.40
1.38
1.21
1.19
1.17
1.15
1.01
0.99
0.97
0.83
0.81
0.79
O
n-Bu
N
n-Bu
O
1.91
1.03
2.00
1.98
1.99
2.01
2.03
2.25
1.97
3.06
2.91
f1 (ppm)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129.06
77.48
77.16
76.84
47.57
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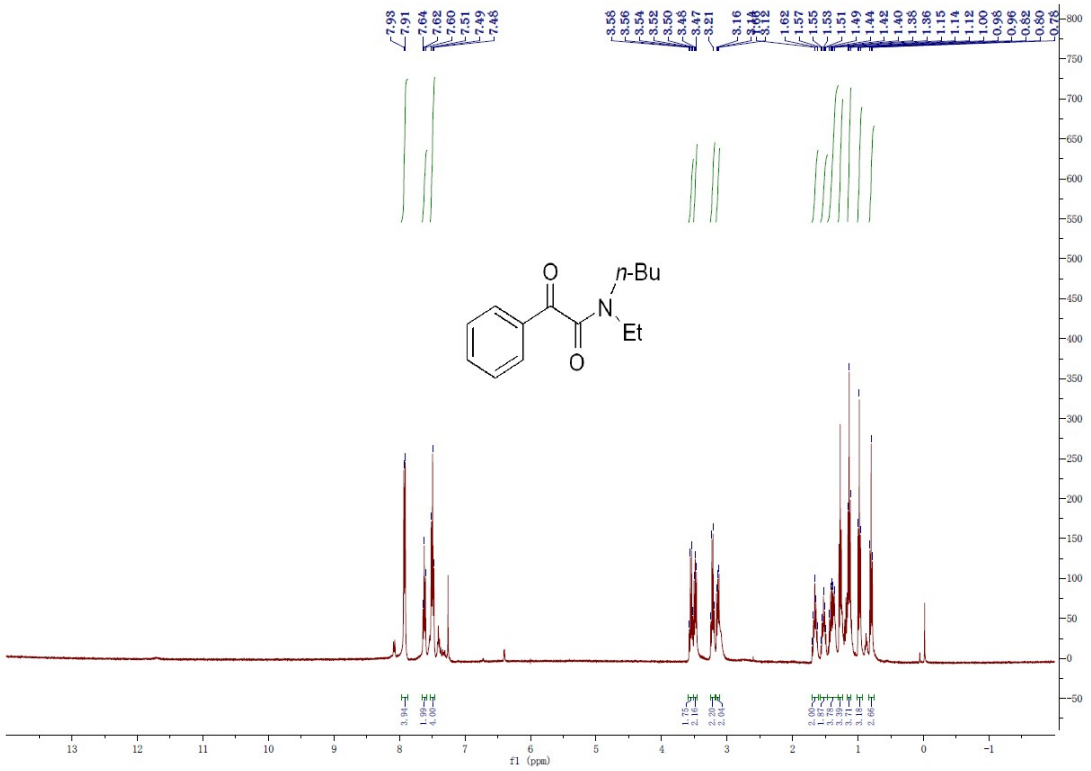
20.40
19.91
14.01
13.71
O
n-Bu
N
n-Bu
O
f1 (ppm)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7.48
3.56
3.54
3.53
3.50
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1.44
1.42
1.40
1.38
1.36
1.15
1.14
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0.96
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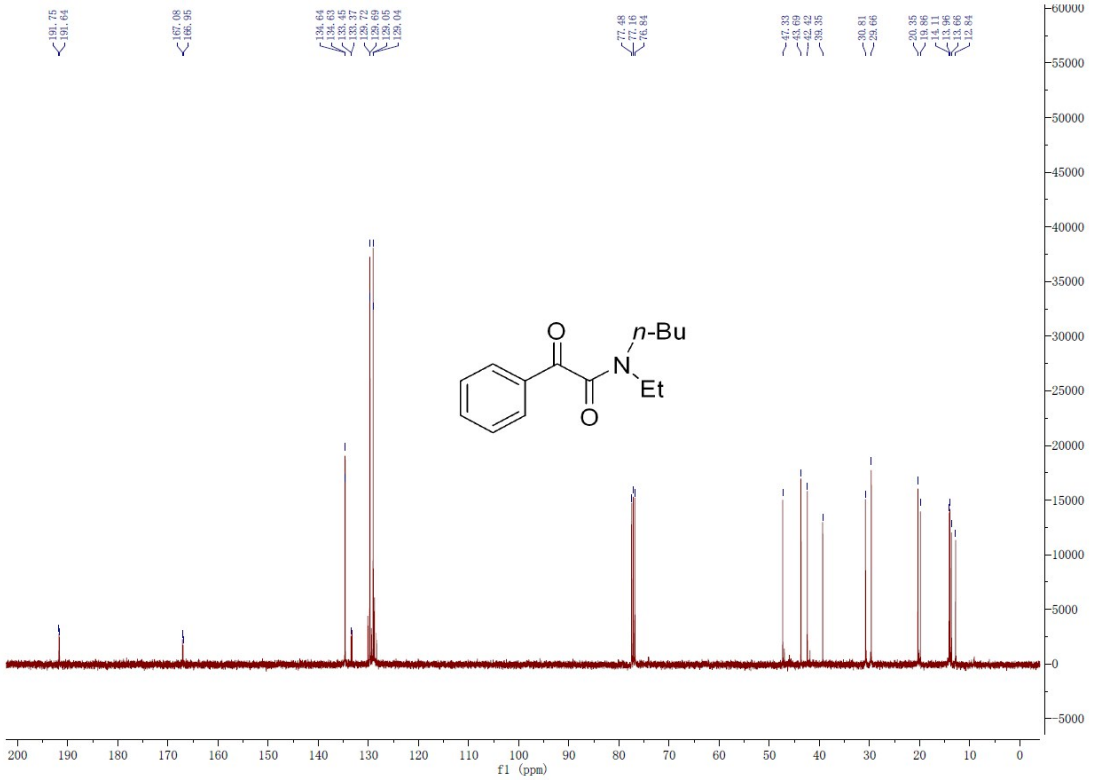
0.80
0.78
O
n-Bu
N
Et
O
f1 (ppm)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128.04
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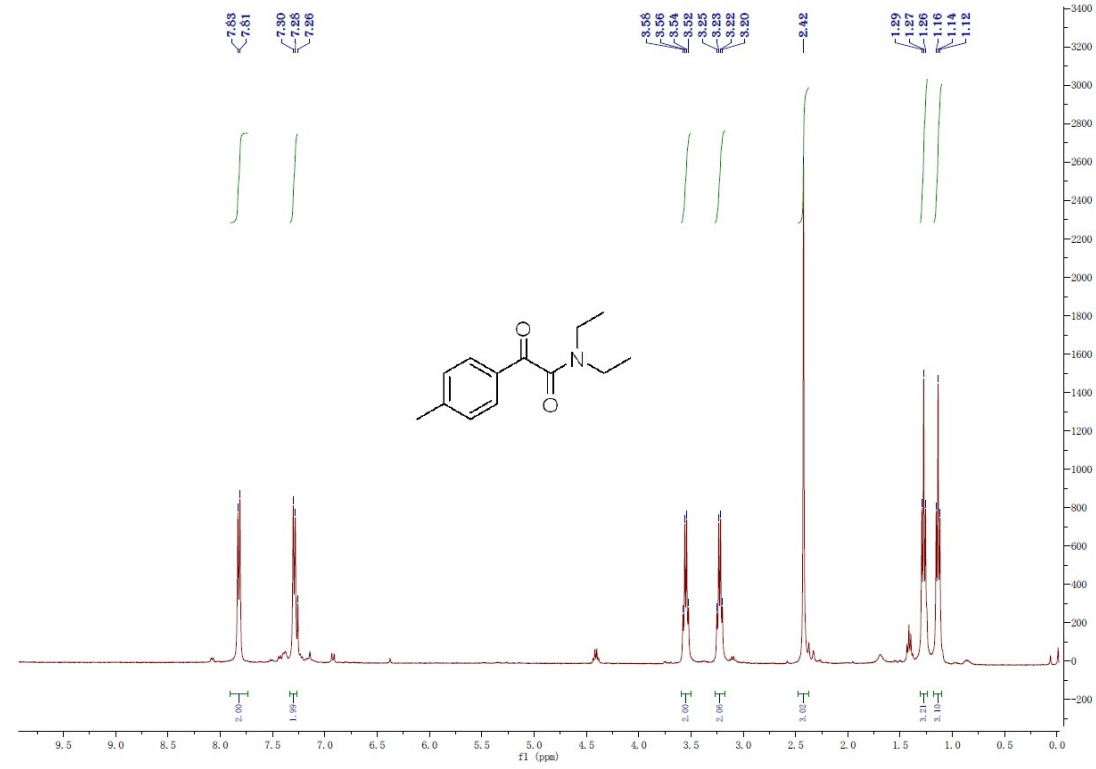
77.16
76.84
47.33
43.69
42.42
38.35
30.81
29.66
20.35
19.86
14.11
13.96
13.66
12.84
O
n-Bu
N
Et
O
f1 (ppm)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3.23
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3.20
2.42
1.29
1.27
1.26
1.16
1.14
1.12
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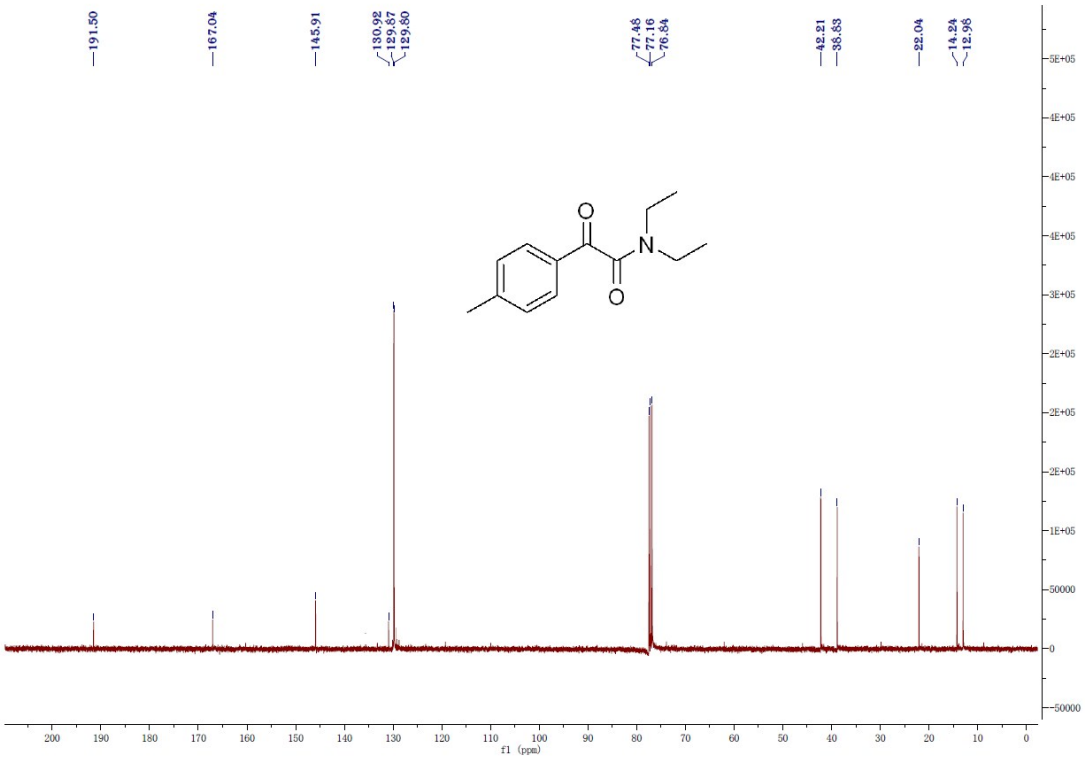
N
O
2.00
1.90
2.00
2.08
3.02
3.21
3.10
f1 (ppm)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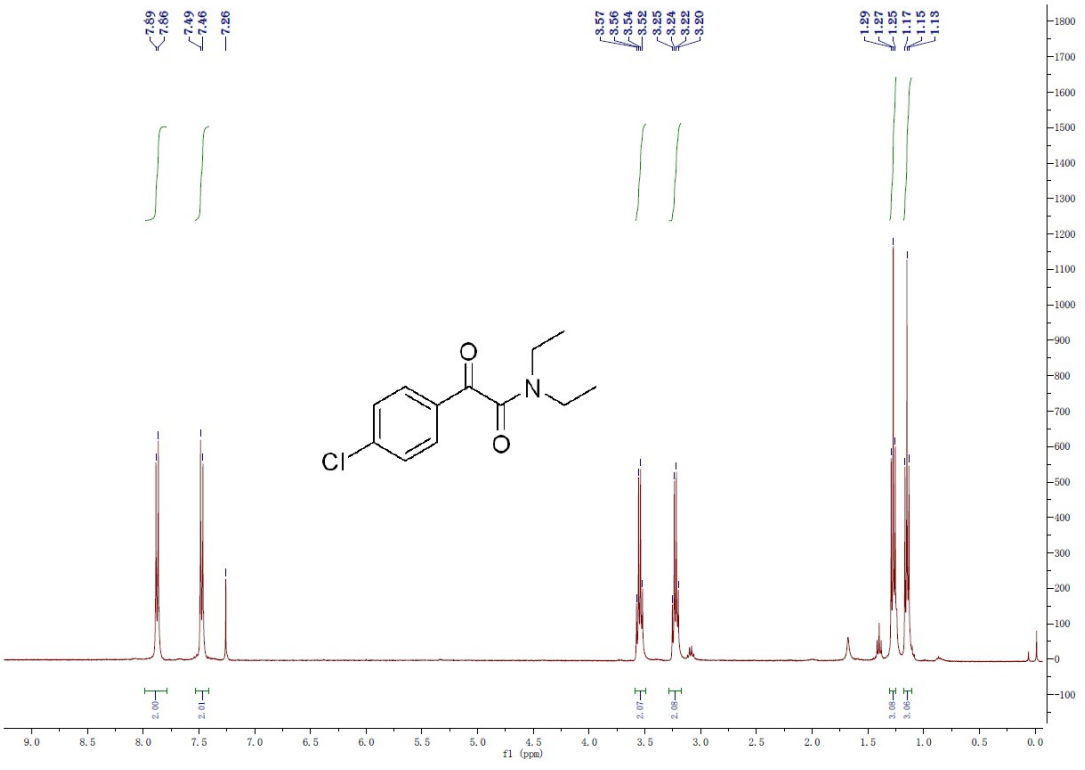
22.04
14.24
12.98
O
N
O
f1 (ppm)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3.52
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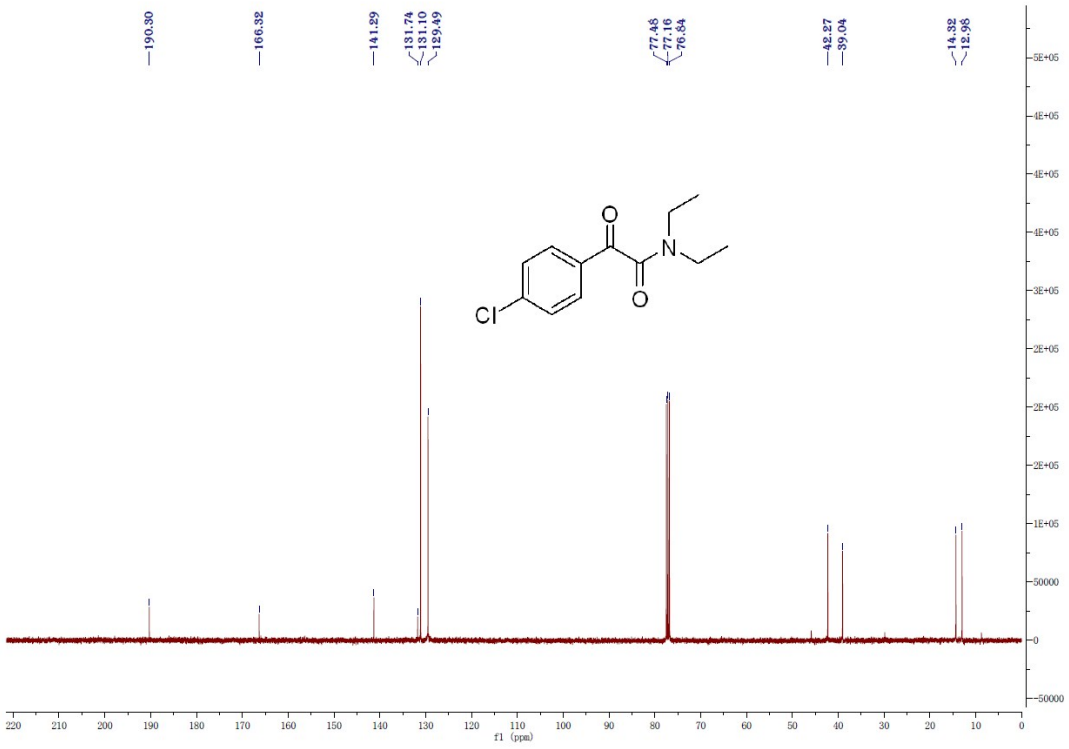
3.24
3.22
3.20
1.29
1.27
1.25
1.17
1.15
1.13
O
N
O
Cl
2.00
2.01
2.07
2.08
3.08
3.00
f1 (ppm)



5. Notes and references

(1) X. Zhang and L. Wang, *Green Chem.*, 2012, **14**, 2141;
(2) F. T. Du and J. X. Ji, *Chem. Sci.*, 2012, **3**, 460;
(3) C. K. Liu, Z. Fang, Z. Yang, Q. W. Li, S. Y. Guo and K. Guo, *RSC Adv.*, 2016, **6**, 25167.
(4) B. F. Chen, F. B. Li, Z. J. Huang, T. Lu and G. Q. Yuan, *Applied Catalysis, A: General*, 2014, **481**, 54.