Supporting Information

One-Pot Cascade Synthesis of α-Diketones from Aldehydes and Ketones in Water Catalyzed by A Bifunctional Iron Nanocomposites Catalyst

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Table Contents

1. General information

Unless otherwise noted, all reagents were purchased commercially from Sigma-Aldrich, or Aladdin and used as received without further purification. The fresh bamboo shoots were obtained from Anhui Taiping Test Centre, International Centre for Bamboo and Rattan, Anhui Province, China. All operations were carried out in an argon atmosphere using glovebox and Schlenk techniques unless otherwise specified. Gas chromatography analysis was performed on an Agilent HP-7890 instrument with a flame ionization detector (FID) and an HP-5MS capillary column (30 m, 0.25 mm i.d., 0.25 μm film thicknesses) using helium as the carrier gas. Gas chromatography-mass spectrometry analysis was carried out on an Agilent HP-7890 instrument with an Agilent HP-5975 with triple-axis detector and HP-5 capillary column using helium carrier gas. NMR spectra were from a Bruker DRX-400, or DRX-600, instrument and calibrated using residual non-deuterated solvent ($CDCl_3$: δ_H = 7.26 ppm, δ_C = 77.16 ppm; DMSO-d_6, δ_H = 2.50 ppm, δ_C = 39.60 ppm) as an internal reference. High-resolution mass data were recorded on Bruke Maxis UHR TOF mass spectrometers in ESI mode.

2. General procedures for the synthesis of α-Diketones

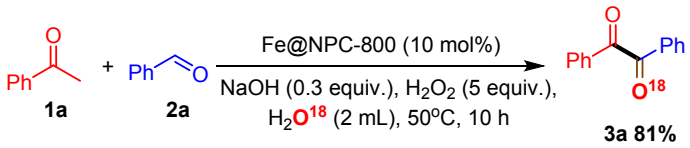
A 25 mL sealing tube was charged with a magnetic stirring, ketone (0.2 mmol), aldehyde (0.22 mmol), Fe@NPC-800 (10 mol% of Fe), H_2O_2 30wt% in water (5 eq.), H_2O (2 mL). The reaction was stirred for 10 h at 50 °C. After completion of the reaction, the reaction mixture was cooled to room temperature and the liquid was extracted by ethyl acetate and the organic phase was analyzed by GC and GC-MS to determine the conversion and selectivity using dodecane as an internal standard. The products were purified by column chromatography and structurally confirmed by NMR.

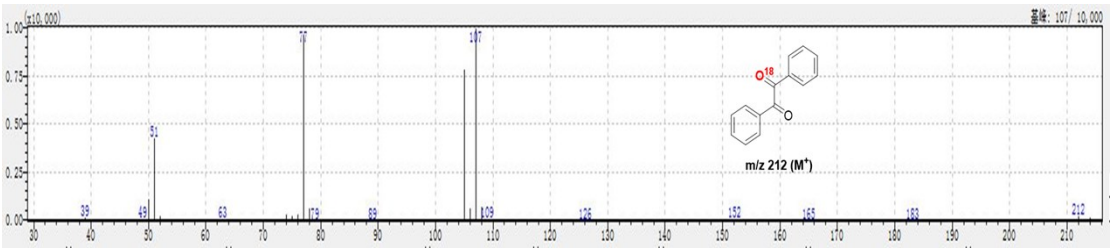
3. Recyclability of the catalyst

The model reaction was chosen to investigate the recyclability of the Fe@NPC-

800 catalyst. A 25 mL sealing tube was charged with a magnetic stirring, acetophenone (0.2 mmol), benzaldehyde (0.22 mmol), catalyst (10 mol% of Fe), H_2O_2 30wt% in water (5 eq.), H_2O (2 mL). The reaction was stirred for 10 h at 50 °C. After completion of the reaction, the reaction mixture was cooled to room temperature and the liquid was extracted by ethyl acetate and the organic phase was analyzed by GC and GC-MS to determine the conversion and selectivity using dodecane as an internal standard. The residue was dispersed in ethanol and water (5 mL v/v=1/1) and the resulting mixture was stirred for 10 min, the catalyst was separated by centrifugation. Such operation was repeated for 3 times. Finally, the obtained black solid was dried under vacuum at 40°C overnight for successive use.

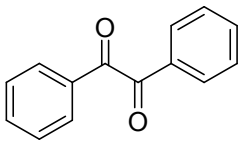
4. The O^{18}-labeling experiment



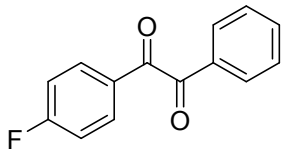


Scheme 1. GC-MS result for O^{18}-Labeling Studies

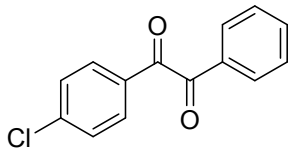
5. Characterization of products



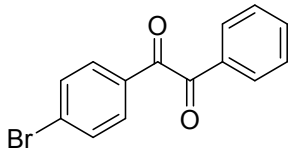
benzil (3a), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05-7.85 (m, 4H), 7.73 -7.60 (m, 2H), 7.58-7.39 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.6, 134.9, 133.0, 129.9, 129.1. The spectroscopic data matched that previously report.[1]



1-(4-fluorophenyl)-2-phenylethane-1,2-dione (3b), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00-7.91 (m, 2H), 7.91-7.82 (m, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.8 Hz, 2H), 7.16-7.04 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.1, 192.8, 167.12 (d, J = 259 Hz), 135.0, 132.9, 132.76 (d, $J_{C\text{-}F}$ = 7.6 Hz), 129.9, 129.5 (d, $J_{C\text{-}F}$ = 2.9 Hz), 129.1, 116.43 (d, $J_{C\text{-}F}$ = 22.1 Hz). The spectroscopic data matched that previously report.[1]

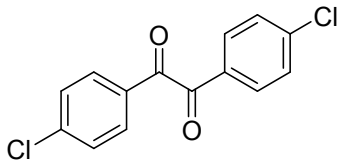


1-(4-chlorophenyl)-2-phenylethane-1,2-dione (3c), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92-7.82 (m, 4H), 7.59 (d, J = 7.4 Hz, 1H), 7.49-7.38 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.9, 193.1, 141.6, 135.1, 132.8, 131.4, 131.3, 129.9, 129.5, 129.1. The spectroscopic data matched that previously report.[1]

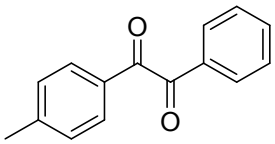


1-(4-bromophenyl)-2-phenylethane-1,2-dione (3d), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$):

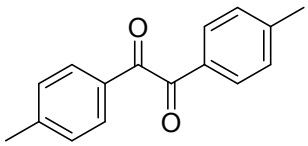
δ 7.89 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.81-7.73 (m, 2H), 7.67-7.57 (m, 3H), 7.50-7.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.9, 193.3, 135.1, 132.8, 132.5, 131.8, 131.3, 130.5, 129.9, 129.1. The spectroscopic data matched that previously report.[1]



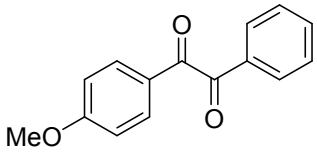
1-(4-chlorophenyl)-2-phenylethane-1,2-dione (3e), Yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.2 Hz, 4H), 7.50 (d, J = 8.2 Hz, 4H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 192.4, 141.8, 131.3, 131.1, 129.5. The spectroscopic data matched that previously report.[1]



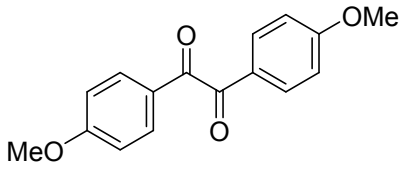
1-phenyl-2-(p-tolyl)ethane-1,2-dione (3f), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96-7.83 (m, 2H), 7.78 (d, *J* = 8.2 Hz, 2H), 7.62 -7.49 (m, 1H), 7.40 (dd, *J* = 10.7, 4.8 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.8, 194.3, 146.3, 134.8, 133.1, 130.6, 130.0, 129.9, 129.8, 129.1, 21.9. The spectroscopic data matched that previously report.[1]



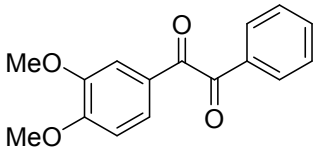
1,2-di-p-tolylethane-1,2-dione (3g), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL).^{1}H NMR (600 MHz, $CDCl_3$): δ 7.86 (d, J = 8.2 Hz, 4H), 7.30 (d, J = 8.2 Hz, 4H), 2.43 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$):δ 194.5, 146.2, 130.7, 130.0, 129.7, 21.9. The spectroscopic data matched that previously report.[1]



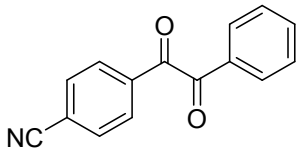
1-(4-methoxyphenyl)-2-phenylethane-1,2-dione (3h), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 5:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98-7.72 (m, 4H), 7.61-7.45 (m, 1H), 7.40 (dd, *J* = 10.7, 4.8 Hz, 2H), 6.99-6.80 (m, 2H), 3.78 (s, 3H). ^{13}C NMR (400 MHz, $CDCl_3$): δ 194.9, 193.2, 165.0, 134.8, 133.2, 132.4, 129.9, 128.9, 126.1, 114.4, 55.7. The spectroscopic data matched that previously report.[1]



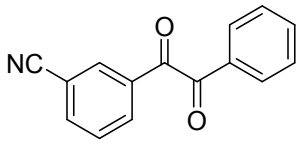
1,2-di-p-tolylethane-1,2- dione (3i), white solid; Purified by column chromatography (petroleum ether/EtOAc, 5:1; 200 mL). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.9 Hz, 4H), 6.96 (d, J = 8.9 Hz, 4H), 3.88 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 193.5, 164.9, 132.4, 126.3, 114.3, 55.6. The spectroscopic data matched that previously report.[1]



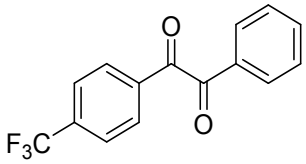
1-phenyl-2-(p-tolyl)ethane-1,2-dione (3j), white solid; Purified by column chromatography (petroleum ether/EtOAc, 5:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20-7.78 (m, 2H), 7.67-7.47 (m, 2H), 7.49-7.32 (m, 3H), 6.80 (d, *J* = 8.4 Hz, 1H), 3.86 (s, 3H), 3.86 (s, 3H). ^{13}C NMR (400 MHz, $CDCl_3$): δ 193.7, 192.3, 153.9, 148.6, 133.3, 132.5, 128.9, 127.9, 125.3, 125.2, 109.4, 109.2, 55.2, 55.1. The spectroscopic data matched that previously report.[2]



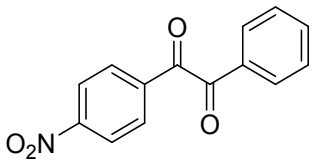
4-(2-oxo-2-phenylacetyl)benzonitrile (3k), white solid; Purified by column chromatography (petroleum ether/EtOAc, 7:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13-8.06 (m, 2H), 8.01-7.93 (m, 2H), 7.85-7.79 (m, 2H), 7.70 (d, *J* = 7.5 Hz, 1H), 7.55 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (400 MHz, $CDCl_3$): δ 193.0, 192.4, 135.9, 135.4, 132.8, 132.5, 130.2, 130.0, 129.2, 117.9, 117.6. The spectroscopic data matched that previously report.[1]



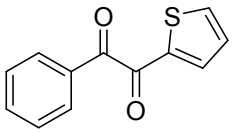
3-(2-oxo-2-phenylacetyl)benzonitrile (3l), white solid; Purified by column chromatography (petroleum ether/EtOAc, 7:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (d, *J* = 1.5 Hz, 1H), 8.17-8.10 (m, 1H), 7.91 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.86-7.80 (m, 1H), 7.68-7.56 (m, 2H), 7.47 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (400 MHz, $CDCl_3$): δ 192.9, 191.7, 137.4, 135.5, 133.8, 133. 7, 133.4, 132.4, 130.09, 130.07, 129.2, 117.4, 113.7. The spectroscopic data matched that previously report.[3]



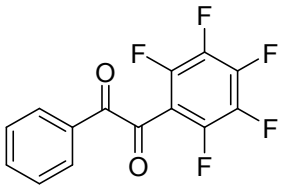
1-phenyl-2-(4-(trifluoromethyl)phenyl)ethane-1,2-dione (3m), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.2 Hz, 2H), 7.95-7.86 (m, 2H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.51-7.40 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 193.5, 193.0, 135.9 (d, J_{C-F} = 33.1 Hz), 135.3, 132.6, 130.2, 130.0, 129.2, 126.07 (d, J_{C-F} = 3.7 Hz), 123.33 (d, J_{C-F} = 271.0 Hz). The spectroscopic data matched that previously report.[4]



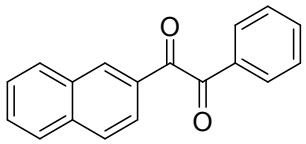
1-(4-nitrophenyl)-2-phenylethane-1,2-dione (3n), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 5:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40-8.20 (m, 2H), 8.20-8.05 (m, 2H), 7.98-7.85 (m, 2H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 192.8, 192.1, 151.2, 137.3, 135.5, 132.4, 130.9, 130.1, 129.3, 124.2. The spectroscopic data matched that previously report.[4]



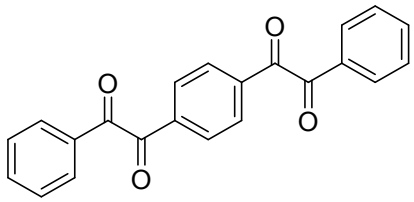
1-phenyl-2-(thiophen-2-yl)ethane-1,2-dione (3o), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (dd, *J* = 2.8, 1.1 Hz, 1H), 8.08-7.98 (m, 2H), 7.71-7.61 (m, 2H), 7.51 (dd, *J* = 10.8, 4.7 Hz, 2H), 7.41 (dd, *J* = 5.1, 2.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 193.3, 187.3, 138.1, 137.0, 134.8, 132.7, 130.2, 128.9, 127.2, 127.1. The spectroscopic data matched that previously report.[5]



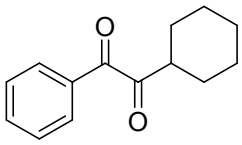
1-(perfluorophenyl)-2-phenylethane-1,2-dione (3p), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11-7.98 (m, 2H), 7.72 (dd, *J* = 10.6, 4.3 Hz, 1H), 7.57 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 188.8, 184.9, 147.85–147.06 (m), 145.19-144.40 (m), 139.15-138.43 (m), 136.93-136.08 (m), 135.4, 130.9, 130.5, 129.2. The spectroscopic data matched that previously report.[6]



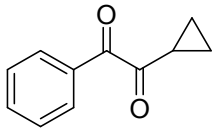
1-(naphthalen-2-yl)-2-phenylethane-1,2-dione (3q), white solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 8.32 (s, 1H), 8.01 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.98-7.89 (m, 2H), 7.87 (d, *J* = 8.7 Hz, 1H), 7.81 (t, J = 8.0 Hz, 2H), 7.62-7.51 (m, 2H), 7.45 (dt, *J* = 15.5, 4.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 194.7, 136.4, 134.9, 133.6, 133.2, 132.4, 130.4, 130.0, 129.9, 129.6, 129.2, 129.1, 128.0, 127.2, 123.7. The spectroscopic data matched that previously report.[7]



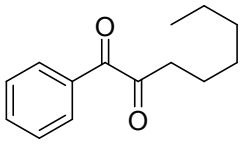
2,2'-(1,4-phenylene)bis(1-phenylethane-1,2-dione) (3r), white solid; Purified by column chromatography (petroleum ether/EtOAc, 3:1; 200 mL).^{1}H NMR (600 MHz, $CDCl_3$): δ 8.11 (s, 4H), 7.97 (dd, J = 8.2, 1.0 Hz, 4H), 7.69 (t, J = 7.4 Hz, 2H), 7.54 (t, J = 7.8 Hz, 4H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 193.5, 193.3, 137.2, 135.3, 132.6, 130.3, 130.0, 129.2. The spectroscopic data matched that previously report.[1]



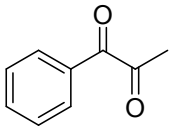
1-cyclohexyl-2-phenylethane-1,2-dione (3s), white solid; Purified by column chromatography (petroleum ether/EtOAc, 20:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99-7.74 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.52-7.35 (m, 2H), 3.004-2.98 (m, 1H), 1.91-1.81 (m, 2H), 1.77-1.67 (m, 2H), 1.66-1.56 (m, 1H), 1.41-1.09 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 206.1, 194.3, 134.6, 132.6, 129.9, 128.9, 45.9, 27.2, 25.8, 25.4. The spectroscopic data matched that previously report.[4]



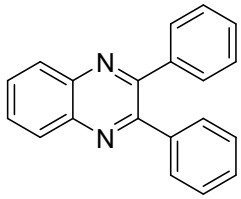
1-cyclopropyl-2-phenylethane-1,2-dione (3t), white solid; Purified by column chromatography (petroleum ether/EtOAc, 20:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 5.1, 3.3 Hz, 2H), 7.54 (d, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 2.55-2.37 (m, 1H), 1.33-1.15 (m, 2H), 1.13-1.08 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 202.7, 192.4, 134.5, 132.2, 130.3, 128.8, 18.6, 13.3. The spectroscopic data matched that previously report.[4]



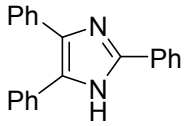
1-phenyloctane-1,2-dione (3u), white solid; Purified by column chromatography (petroleum ether/EtOAc, 20:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13-7.86 (m, 2H), 7.71-7.56 (m, 1H), 7.50 (dd, *J* = 10.8, 4.8 Hz, 2H), 2.87 (t, *J* = 7.4 Hz, 2H), 1.69 (dd, *J* = 14.9, 7.5 Hz, 2H), 1.39-1.26 (m, 6H), 0.89 (dd, *J* = 6.9, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 203.6, 192.6, 134.6, 132.0, 130.2, 128.9, 38.8, 31.5, 28.8, 22.8, 22.5, 14.0. The spectroscopic data matched that previously report.[4]



1-phenylpropane-1,2-dione (3v), white solid; Purified by column chromatography (petroleum ether/EtOAc, 20:1; 200 mL). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, *J* = 7.9 Hz, 2H), 7.75-7.57 (m, 1H), 7.56-7.37 (m, 2H), 2.52 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 200.6, 191.4, 134.6, 131.8, 130.3, 128.8, 26.4. The spectroscopic data matched that previously report.[4]

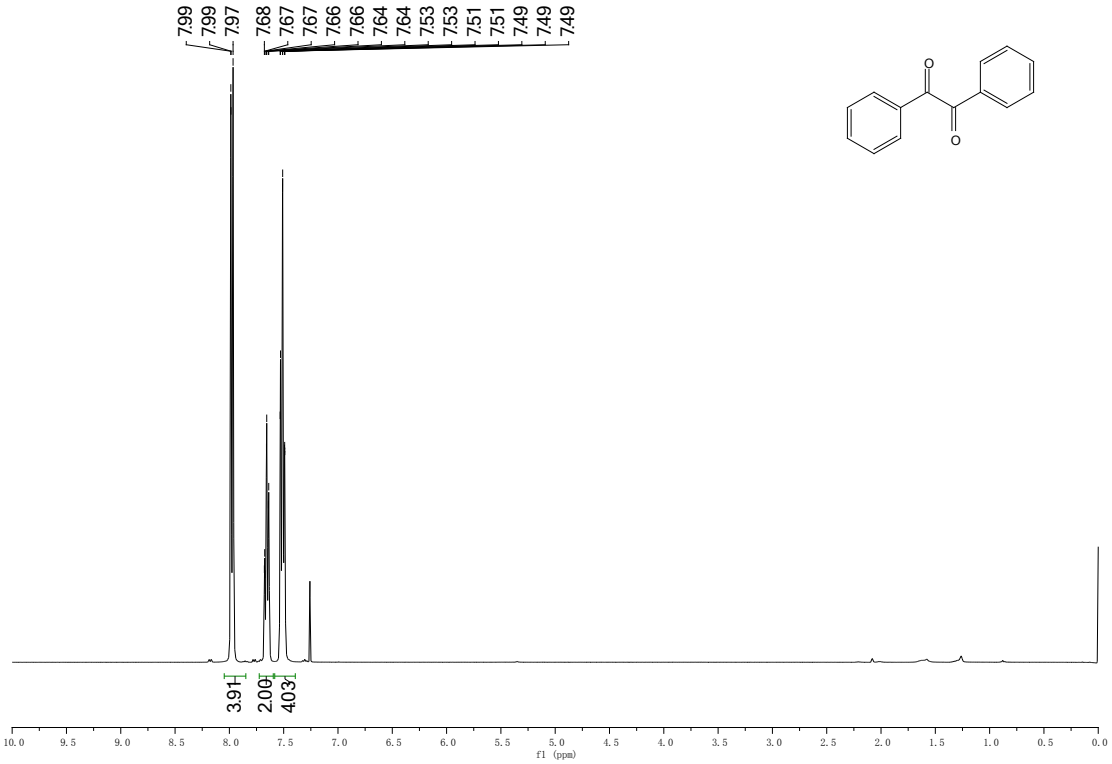


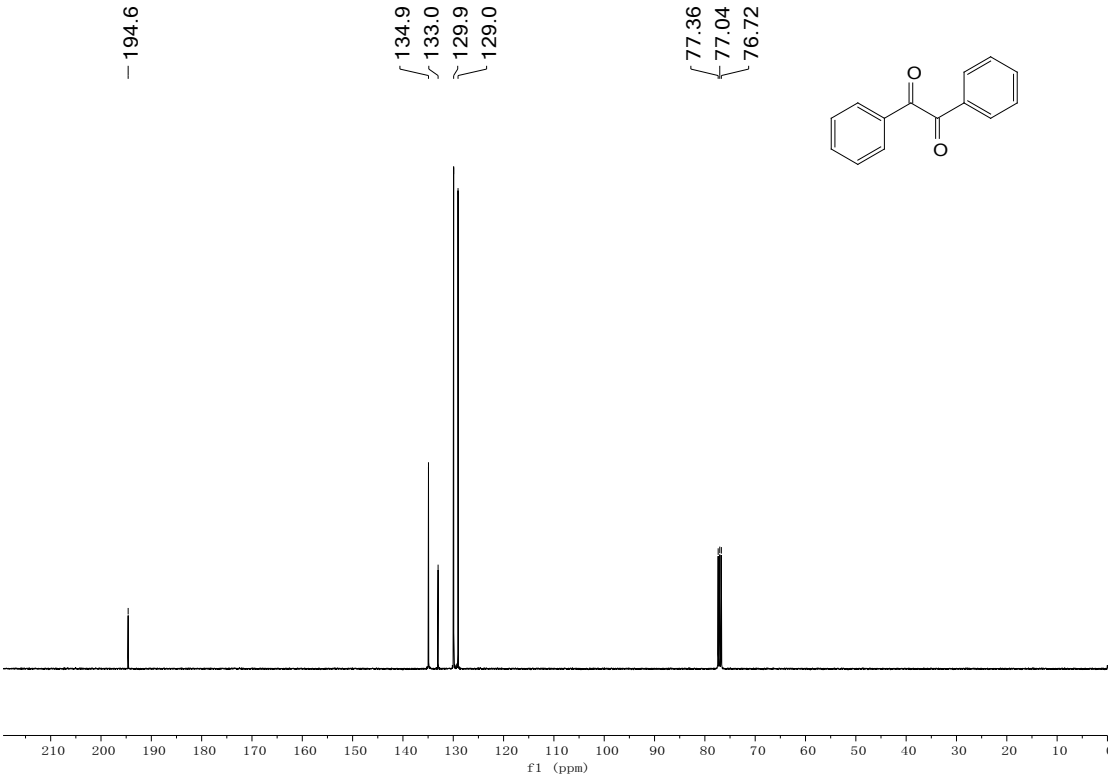
2,3-diphenylquinoxaline (4a), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 10:1; 200 mL).^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (dd, *J* = 6.4, 3.4 Hz, 2H), 7.78 (dd, *J* = 6.4, 3.4 Hz, 2H), 7.52 (dd, *J* = 7.6, 1.6 Hz, 4H), 7.35 (dd, *J* = 8.8, 4.6 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 153.5, 141.3, 139.1, 129.9, 129.8, 129.2, 128.8, 128.3. The spectroscopic data matched that previously report.[8]

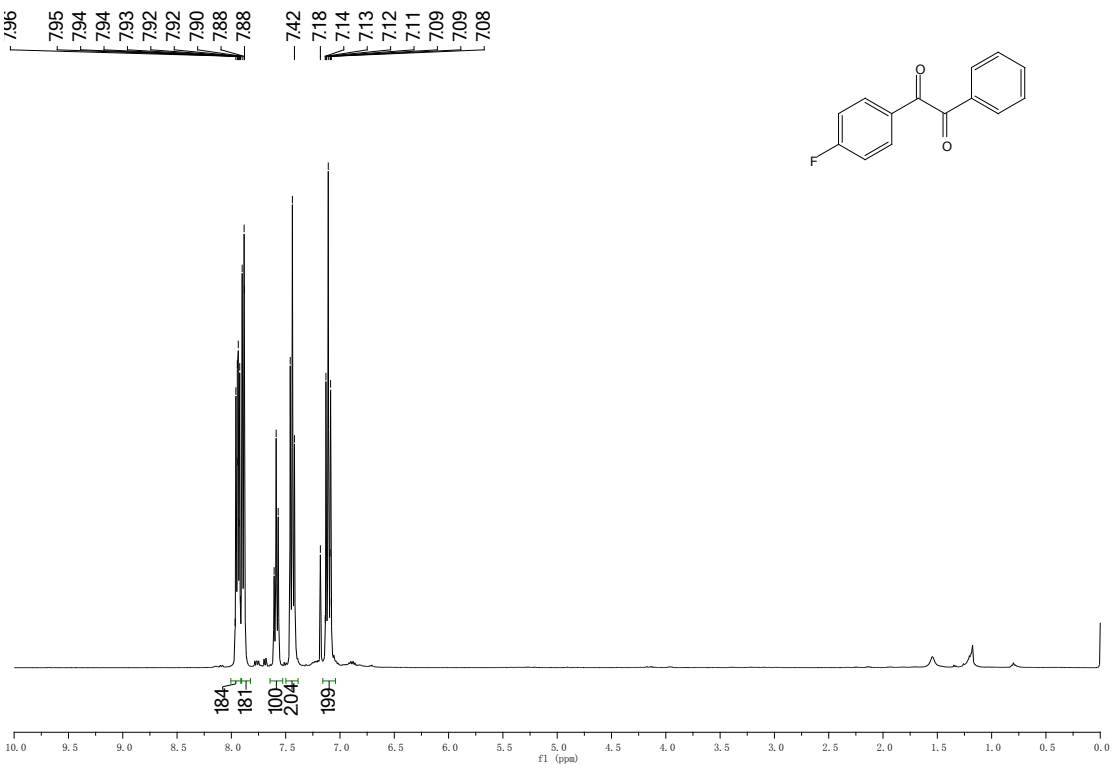


2,4,5-triphenyl-1H-imidazole (5a), yellow solid; Purified by column chromatography (petroleum ether/EtOAc, 3:1; 200 mL). ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.71 (s, 1H), 8.12 (d, *J* = 7.6 Hz, 2H), 7.71-7.08 (m, 13H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 146.0, 137.6, 135.7, 131.6, 130.8, 129.2, 128.9, 128.7, 128.2, 127.6, 126.9, 125.7. The spectroscopic data matched that previously report.[9]

6. ^{1}H and ^{13}C NMR spectra of products



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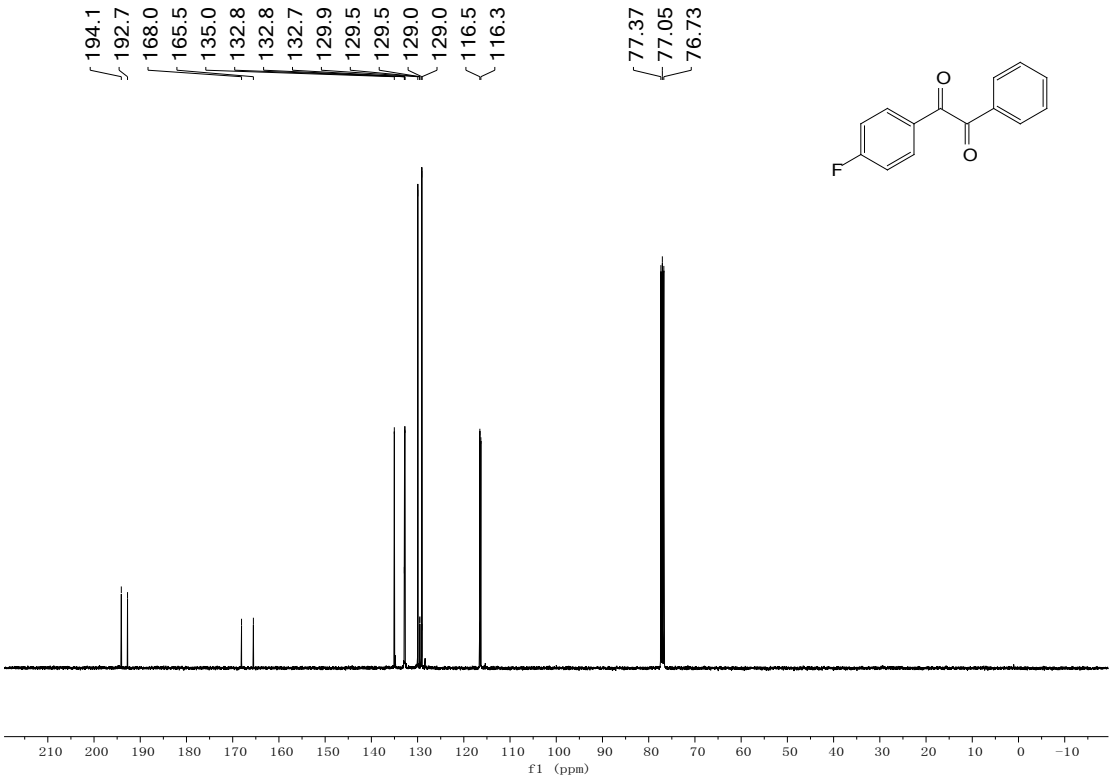
7.14
7.13
7.12
7.11
7.09
7.09
7.08
O
O
F
1.84
1.81
1.00
2.04
1.99
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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192.7
168.0
165.5
135.0
132.8
132.8
132.7
129.9
129.5
129.5
129.0
129.0
116.5
116.3
77.37
77.05
76.73
O
O
F
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)


7.90
7.88
7.88
7.86
7.86
7.84
7.84
7.60
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7.43
7.42
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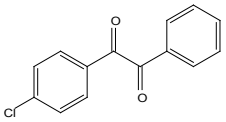
7.40
7.40
7.18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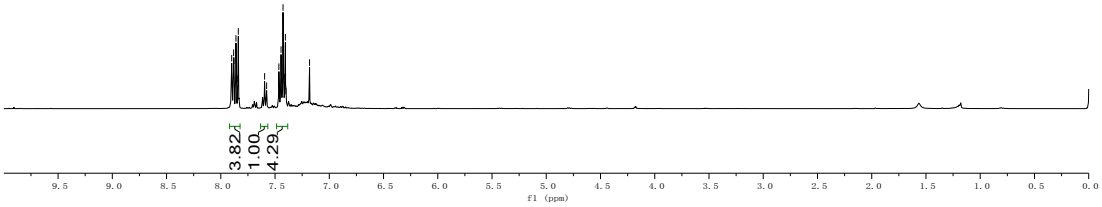
O
Cl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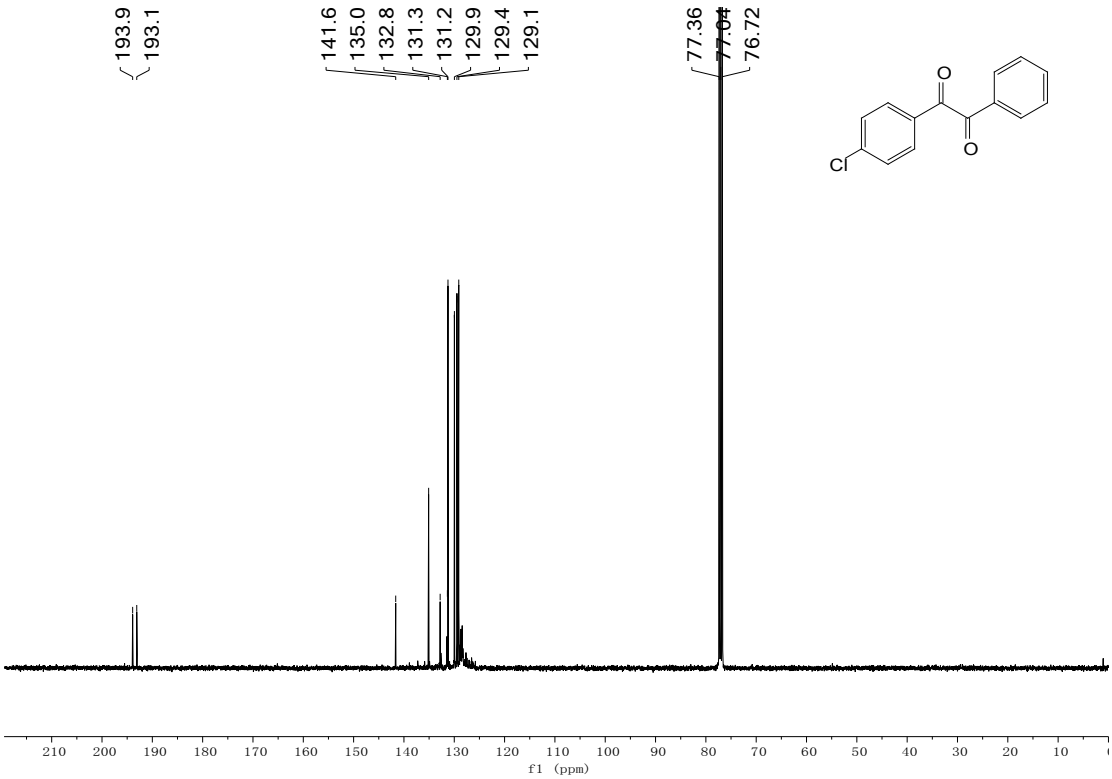
f1 (ppm)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193.1
141.6
135.0
132.8
131.3
131.2
129.9
129.4
129.1
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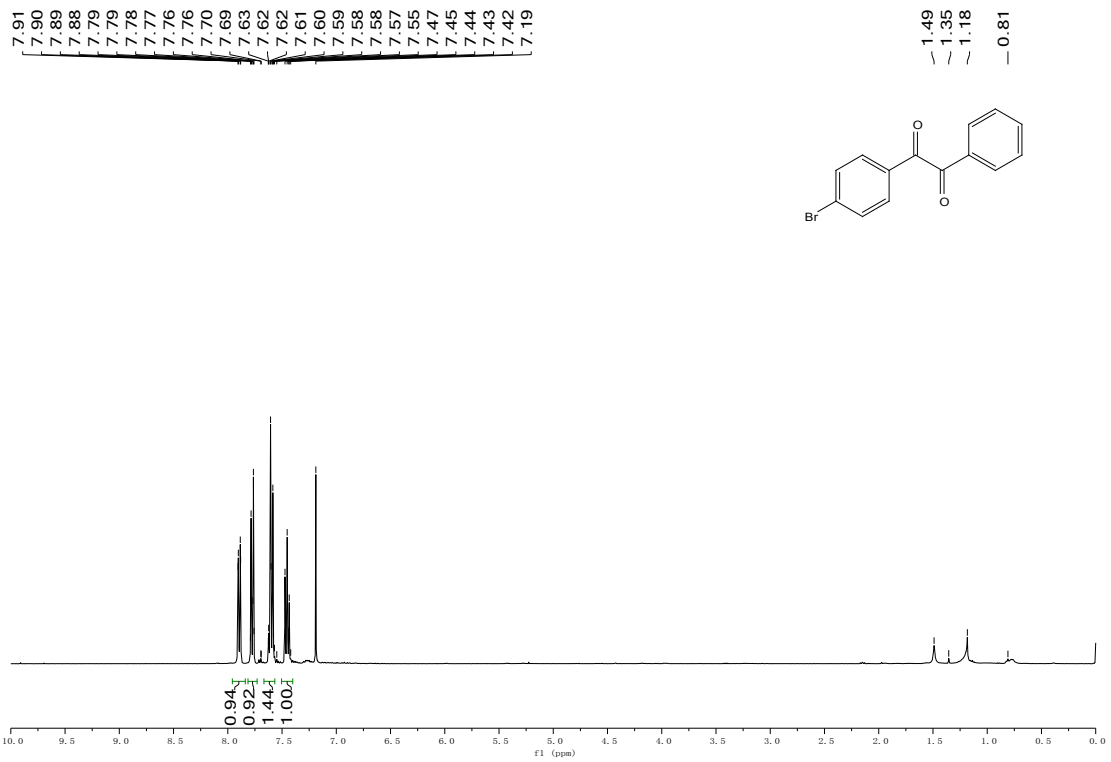
77.04
76.72
O
O
Cl
f1 (ppm)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7.57
7.55
7.47
7.45
7.44
7.43
7.42
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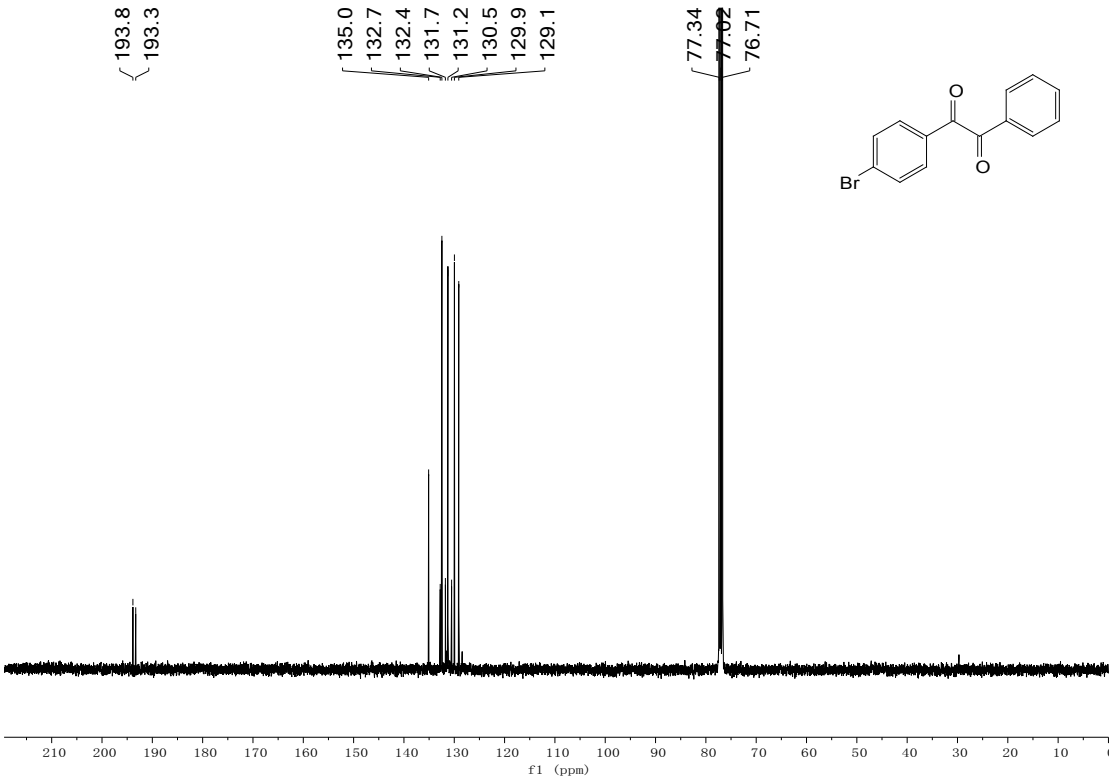
f1 (ppm)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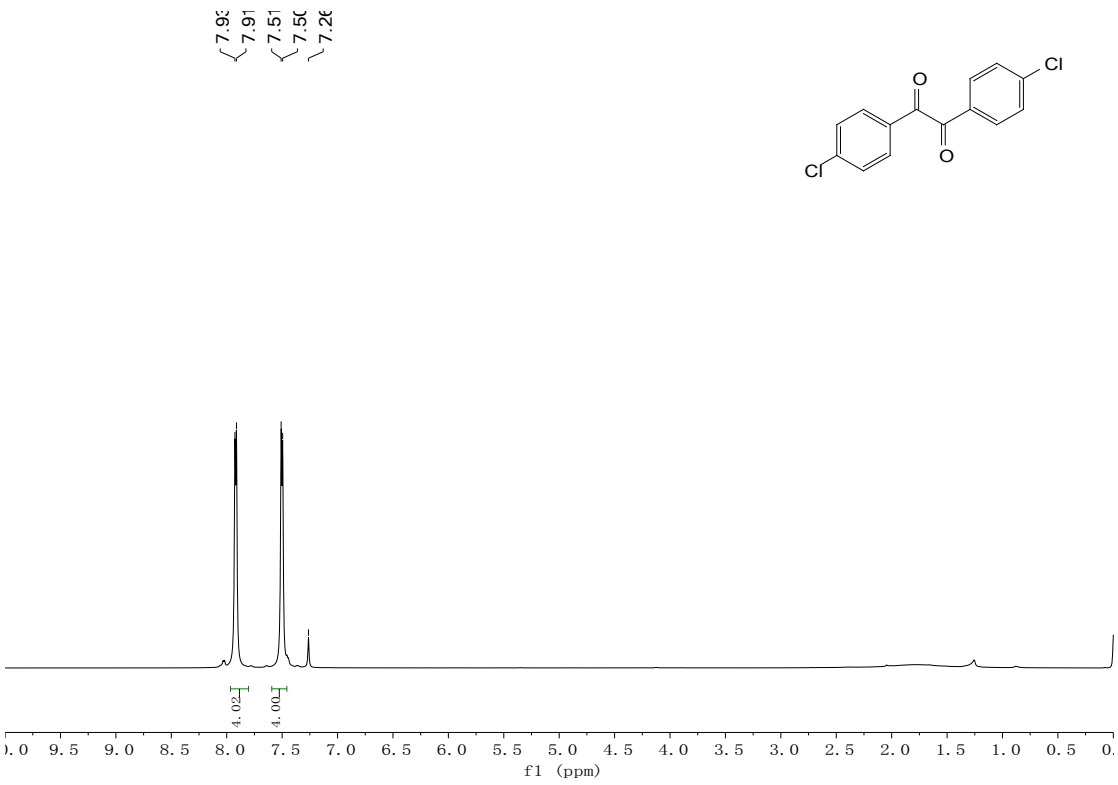
Br
f1 (ppm)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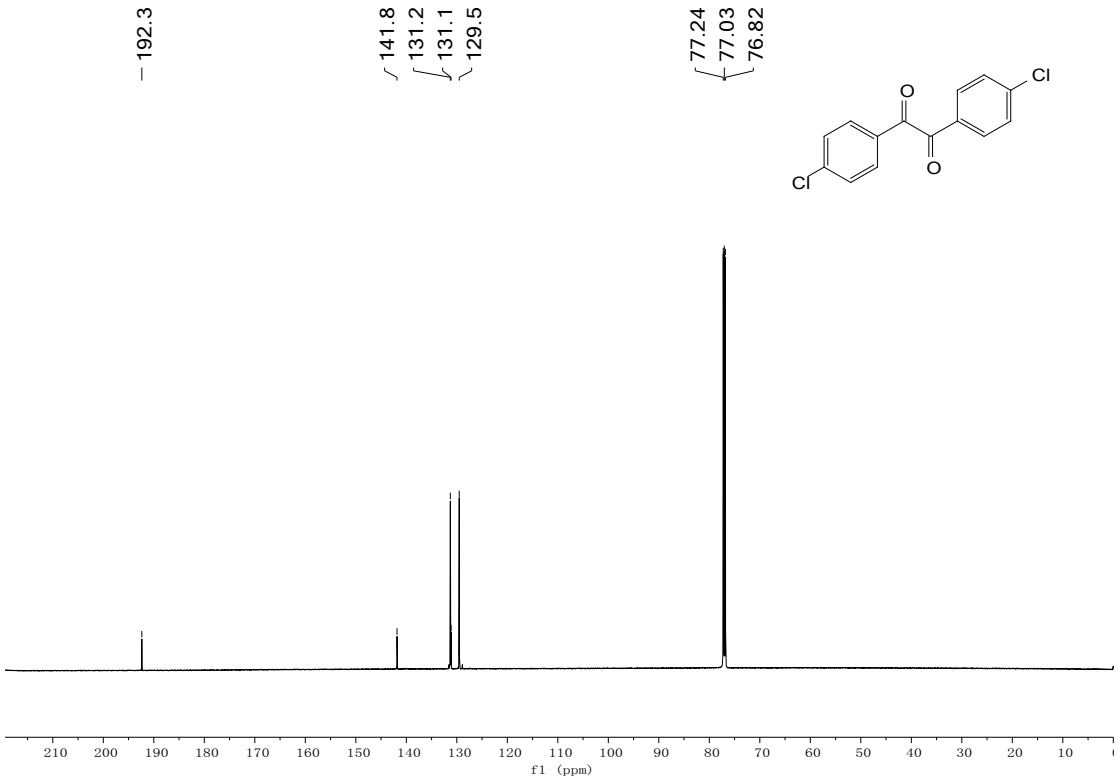
Cl
4.02
4.00
f1 (ppm)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141.8
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131.1
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76.82
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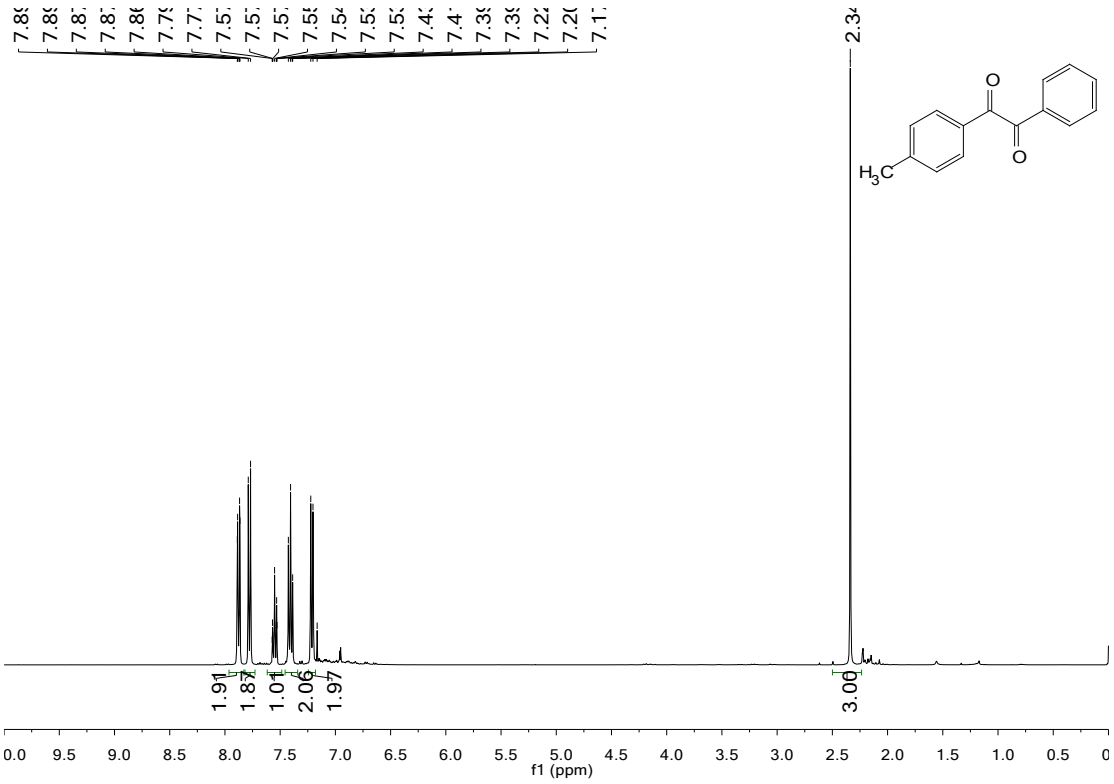
O
O
Cl
f1 (ppm)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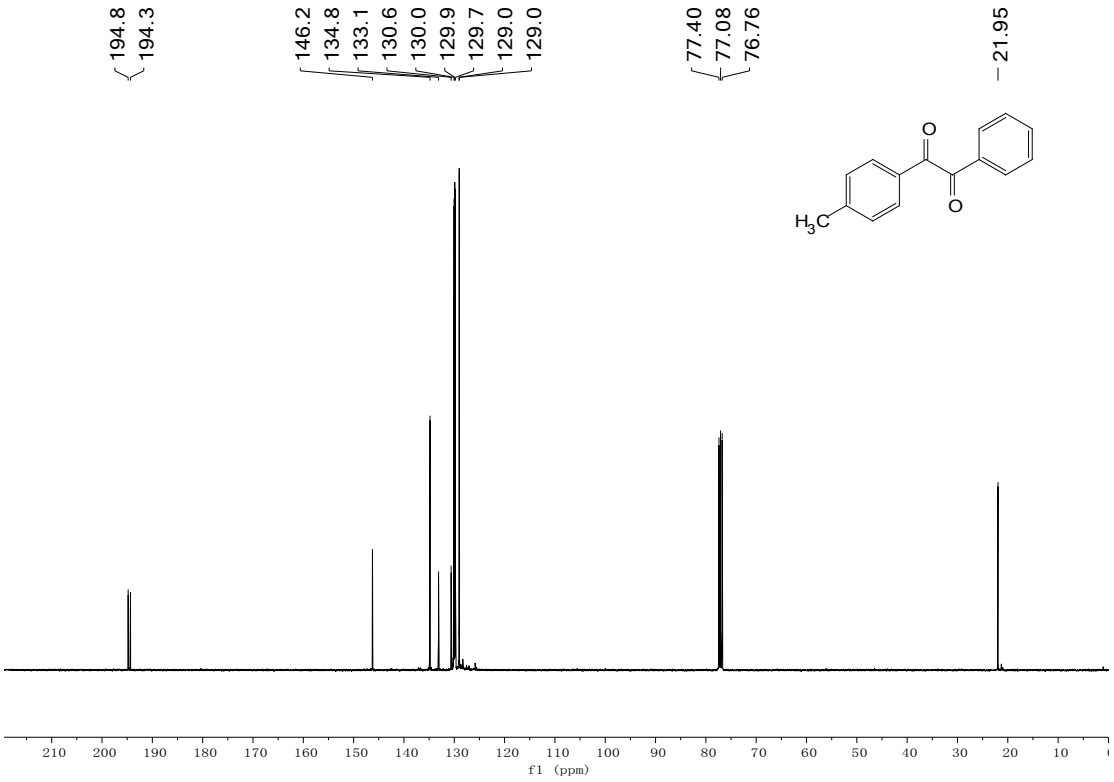
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194.3
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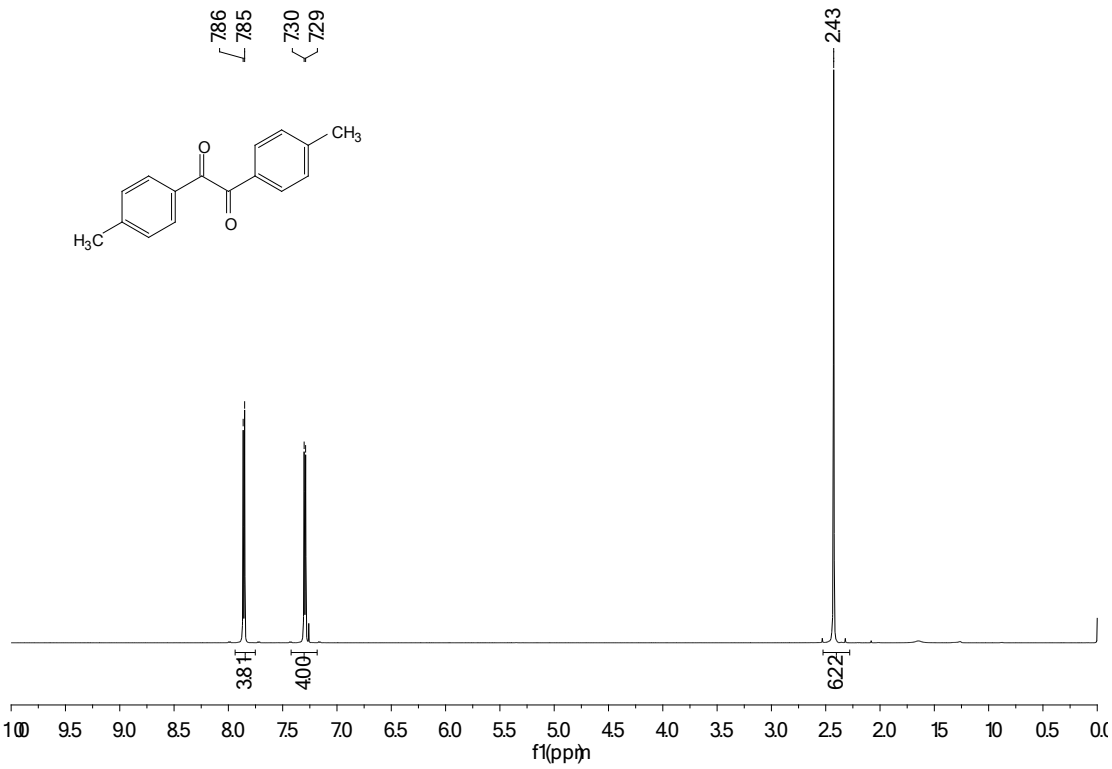
H3C
f1 (ppm)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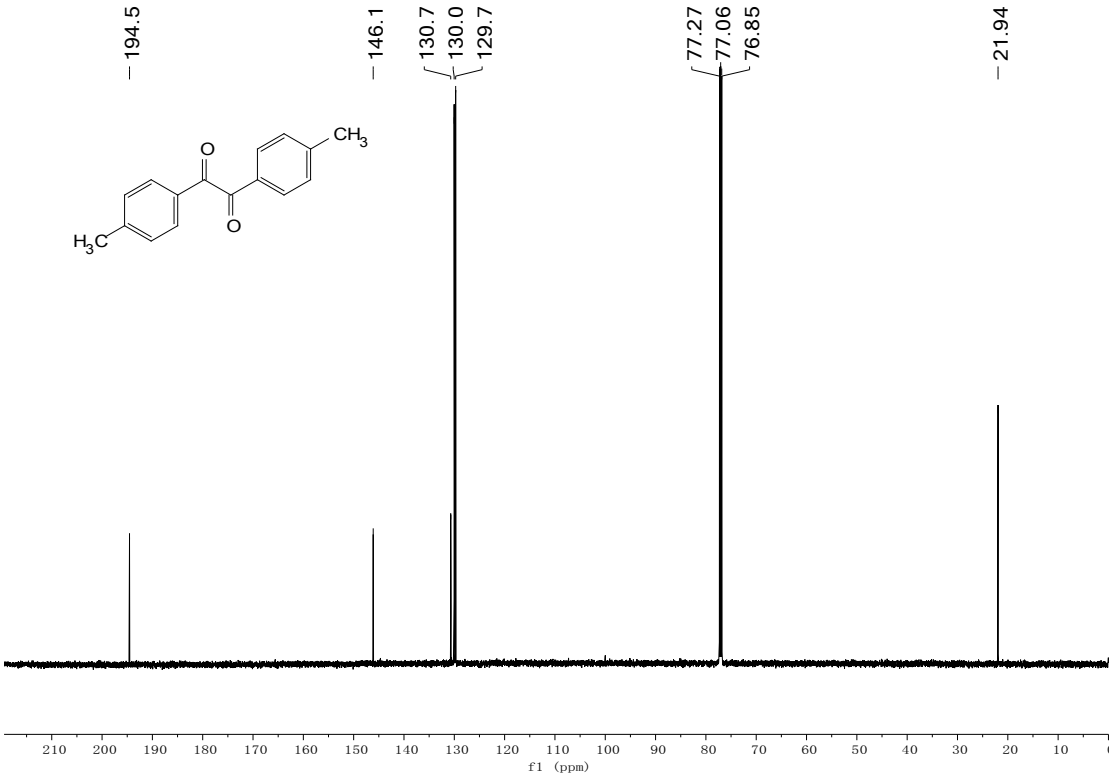
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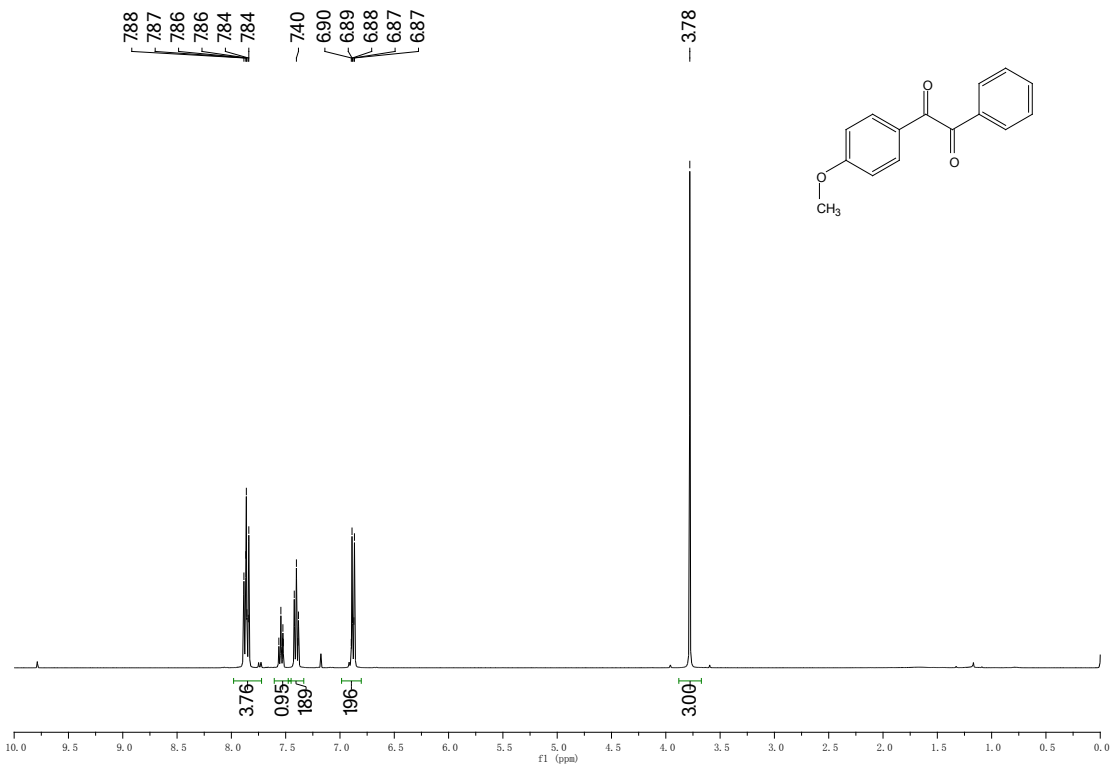
O
H3C
f1 (ppm)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1.89
1.96
3.00
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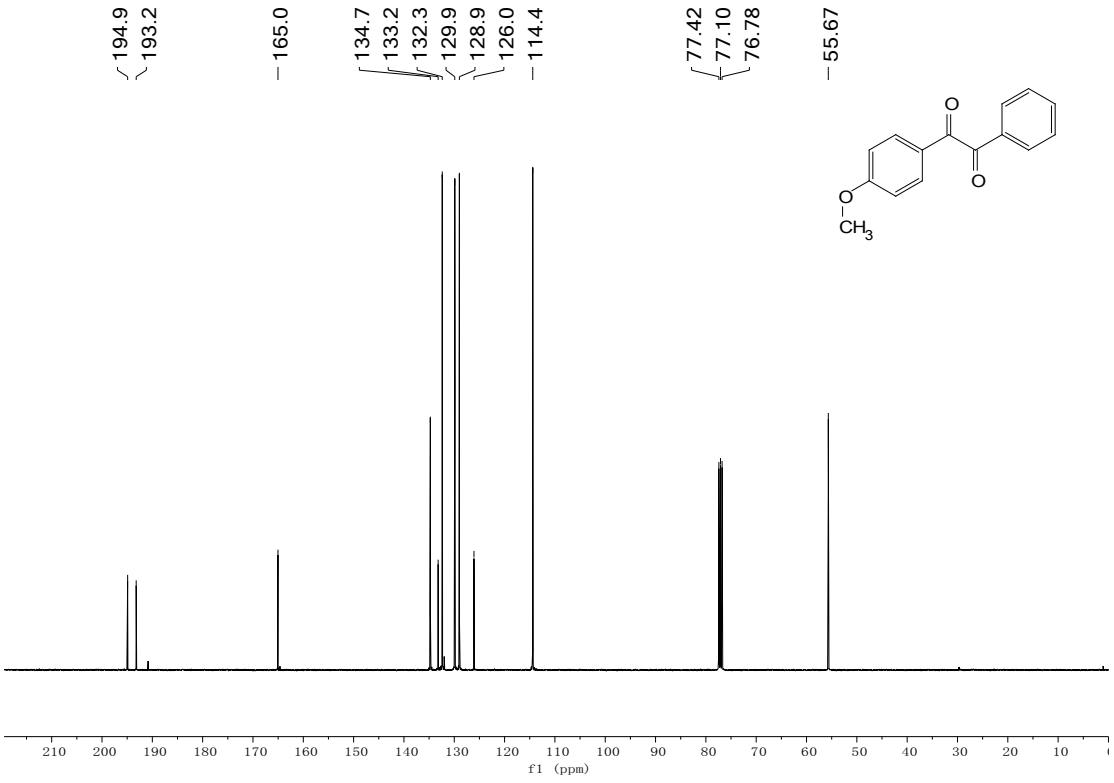
f1 (ppm)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O
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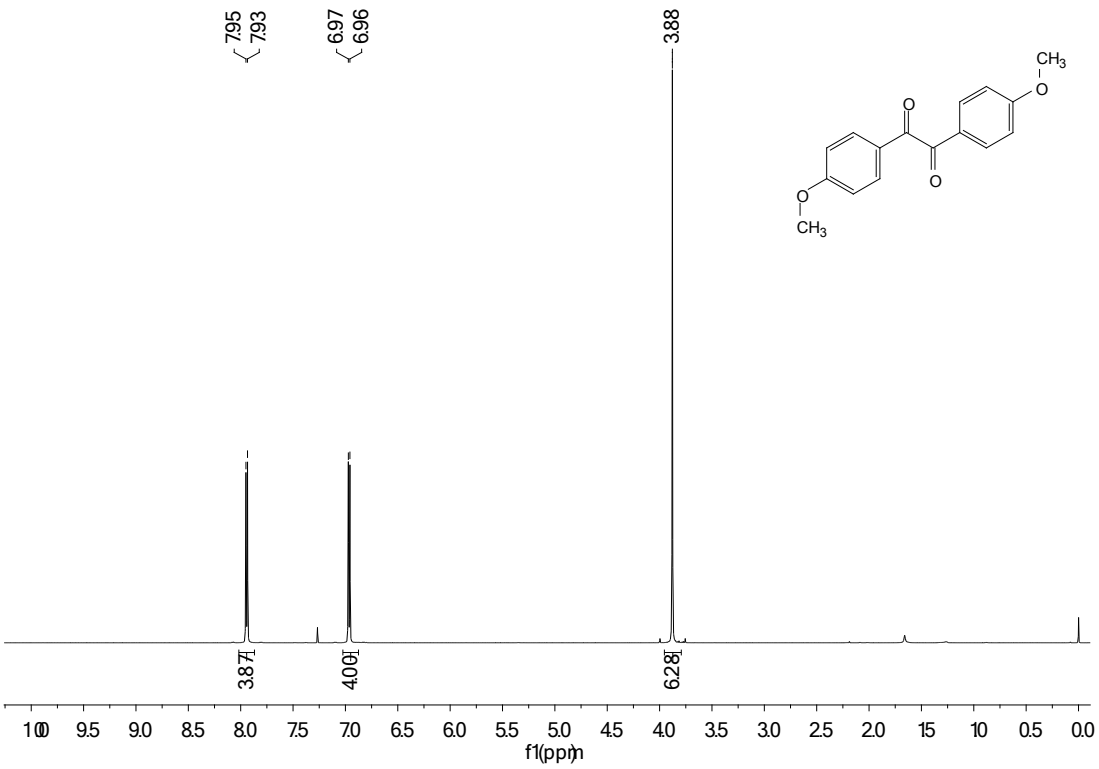
f1 (ppm)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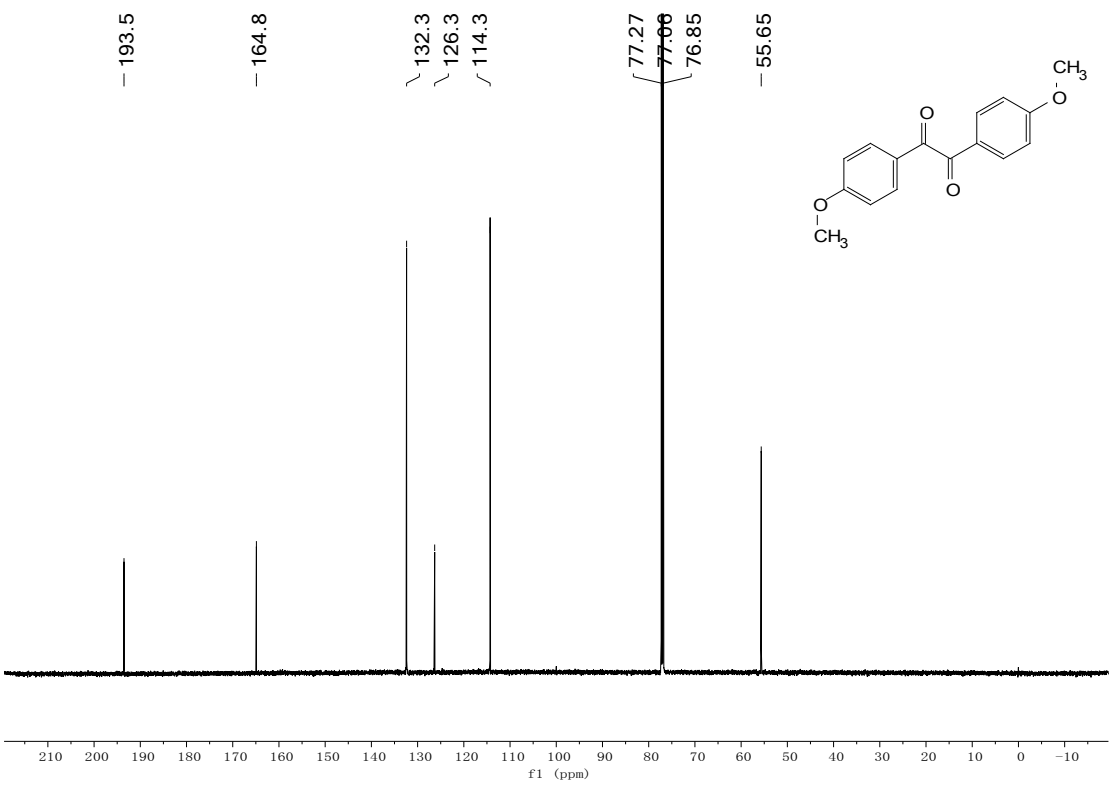
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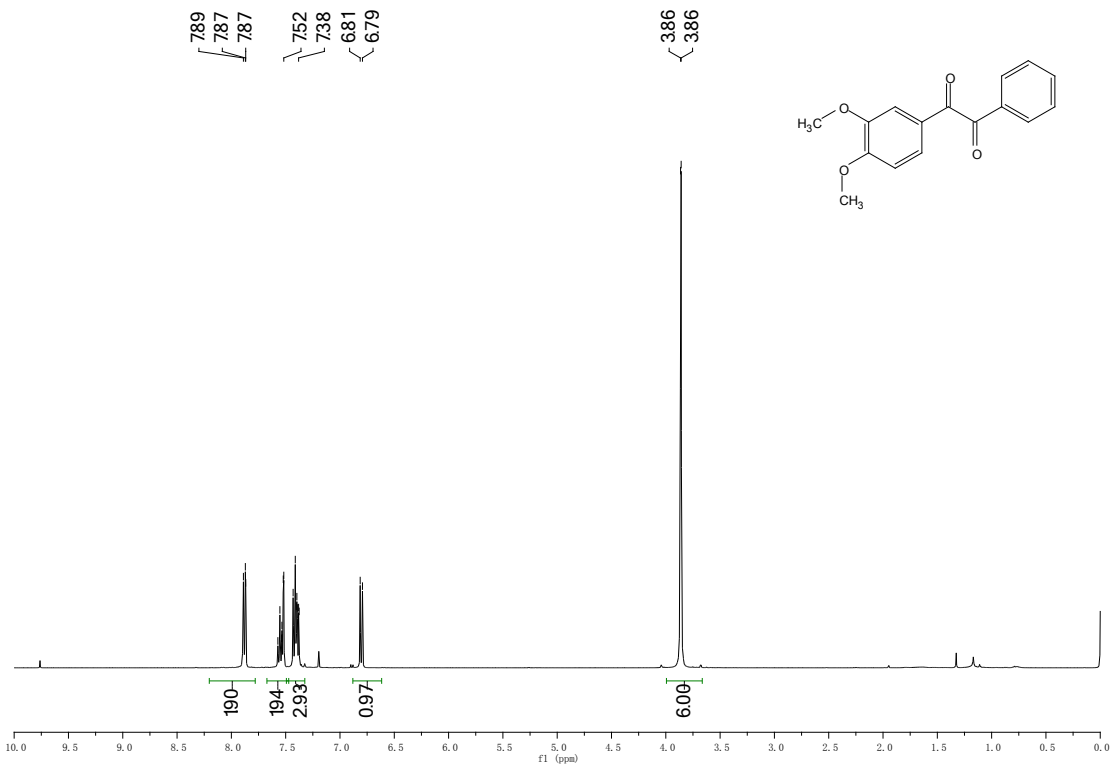
76.85
55.65
f1 (ppm)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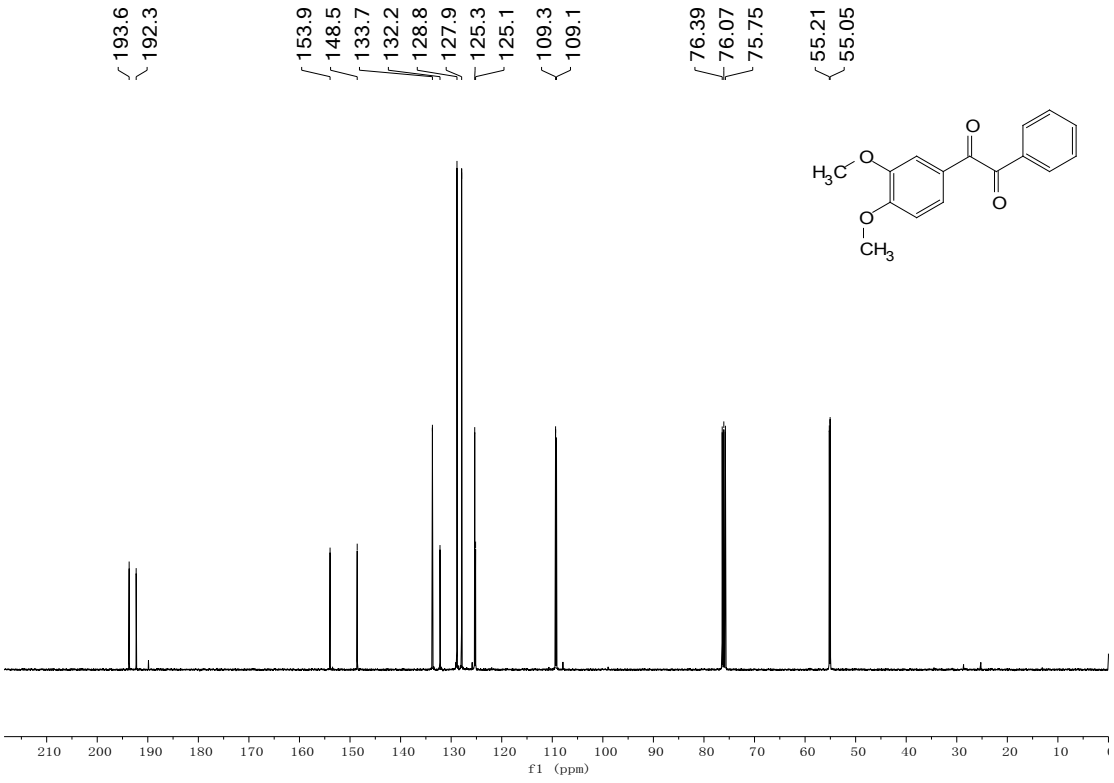
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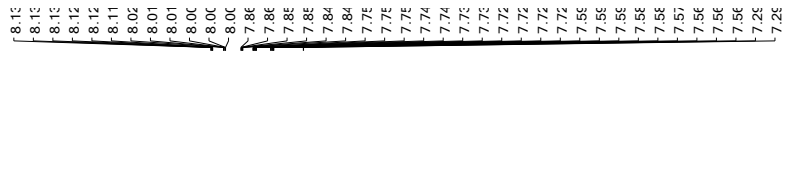

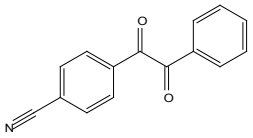
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O
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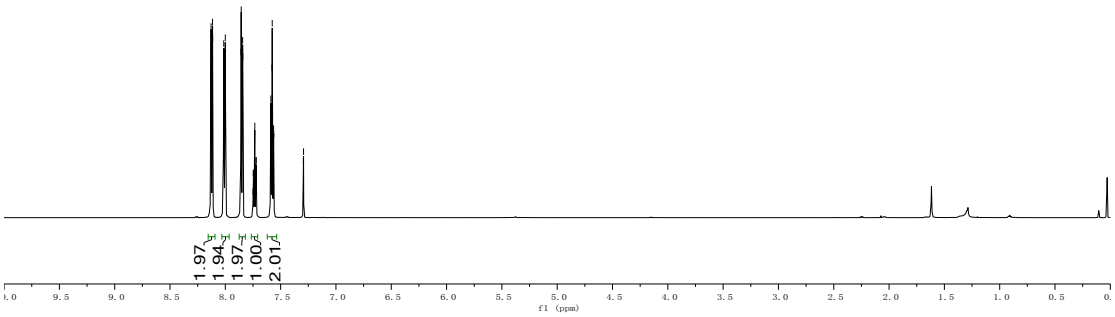
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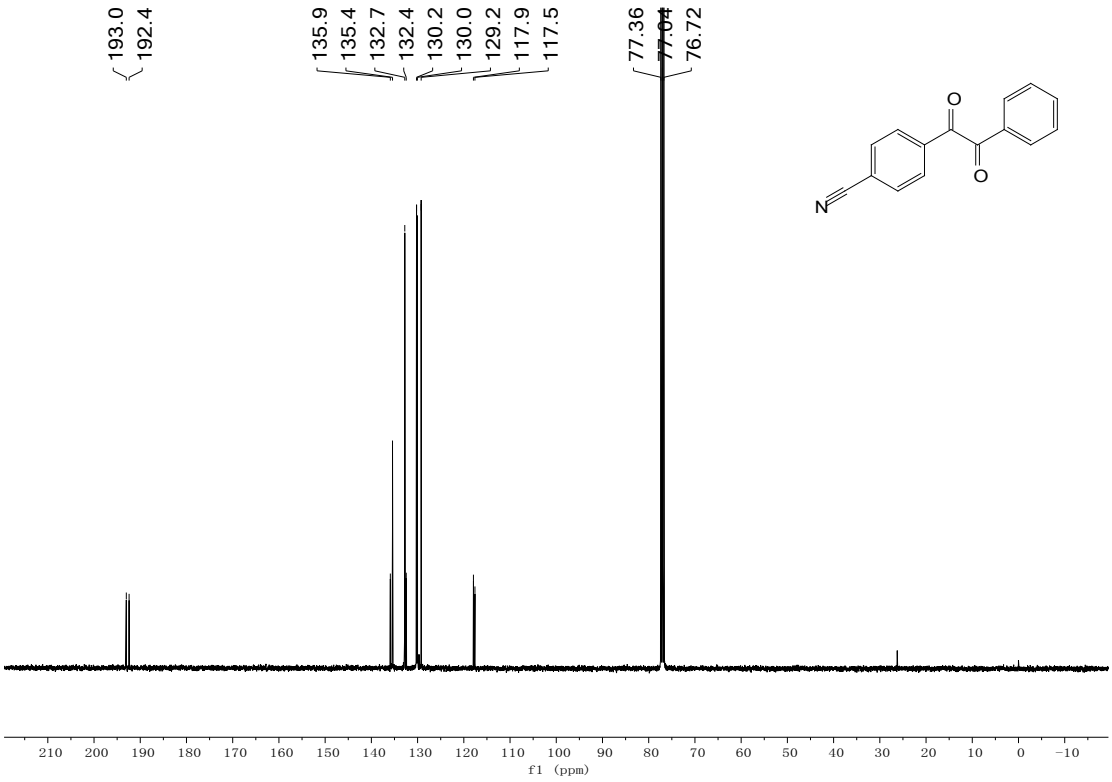
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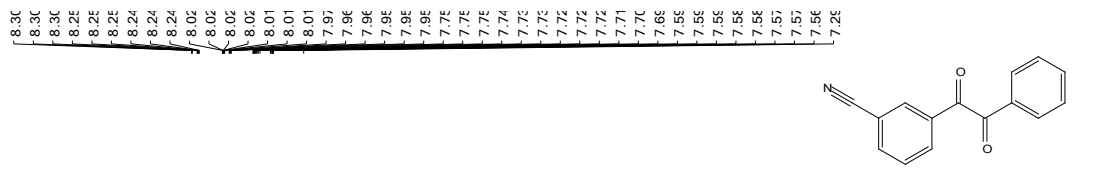
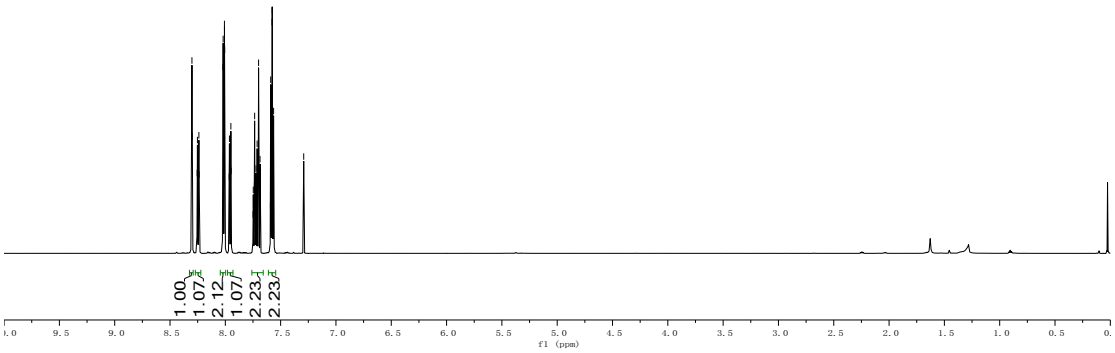
O
O
N

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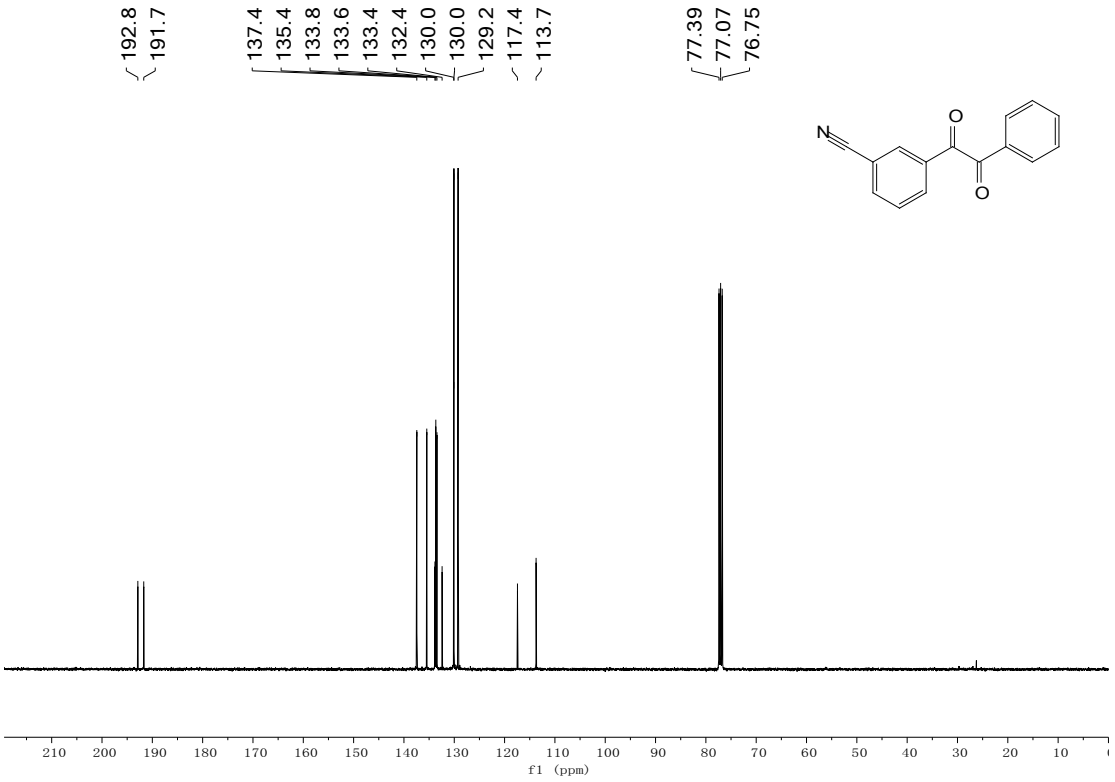
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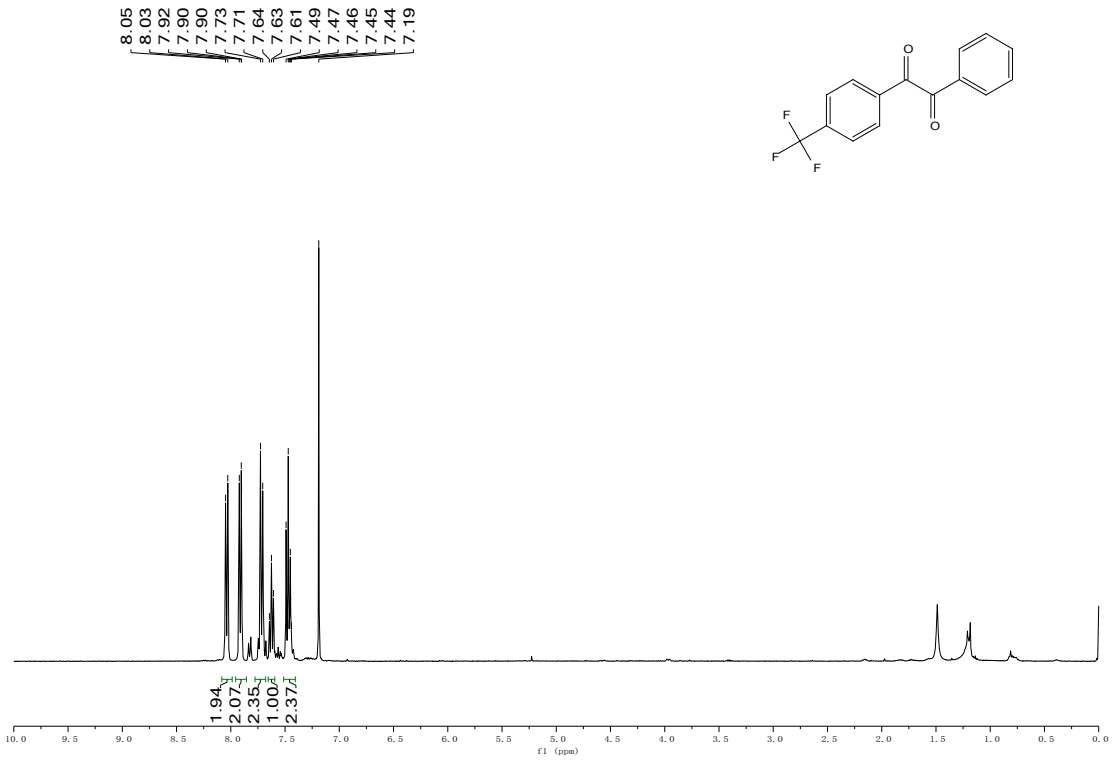
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O
F
F
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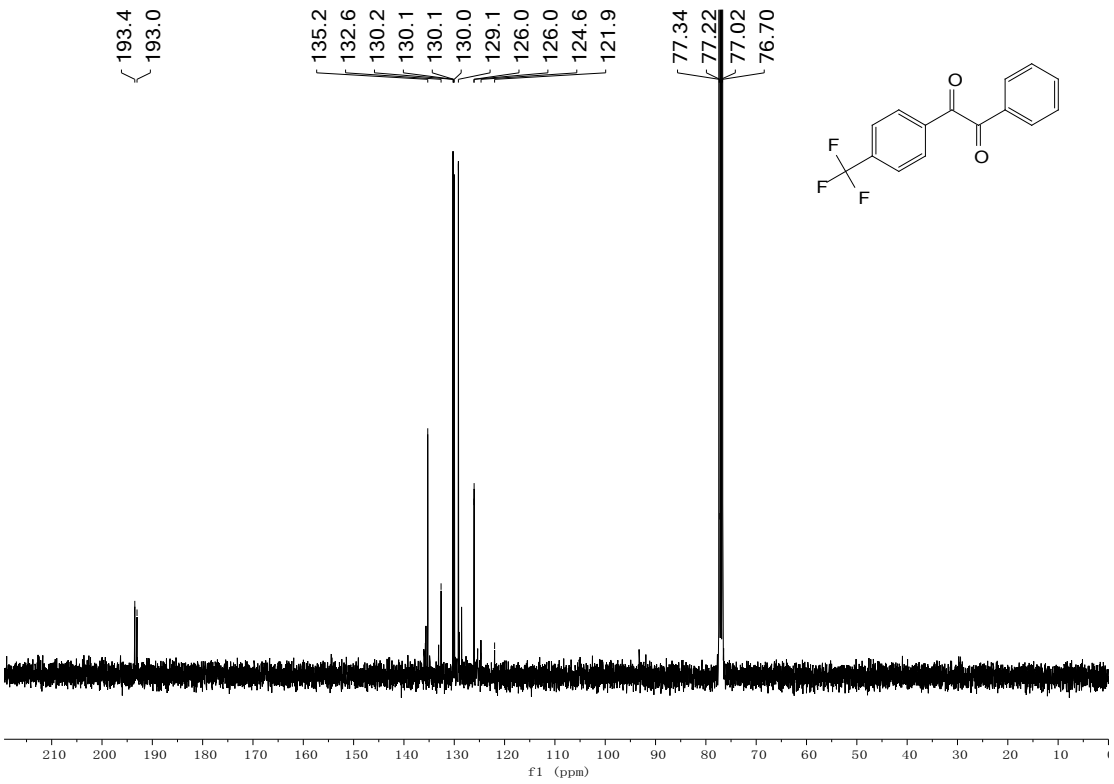
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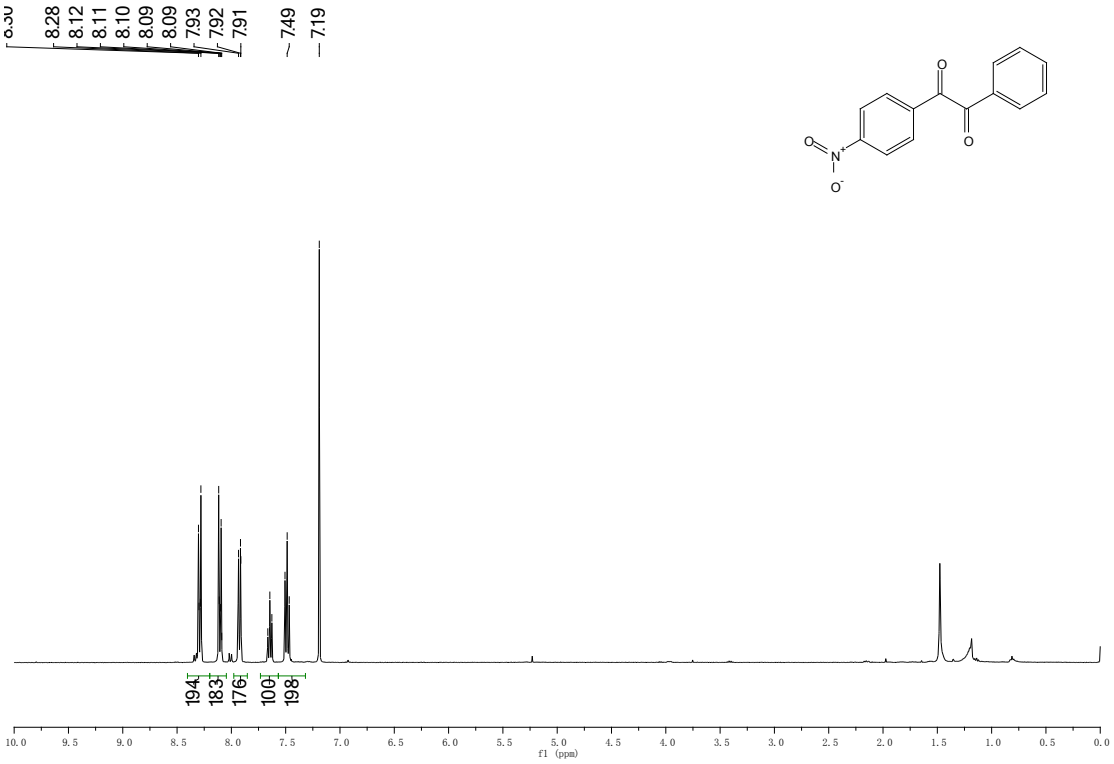
F
F
F
f1 (ppm)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1.98
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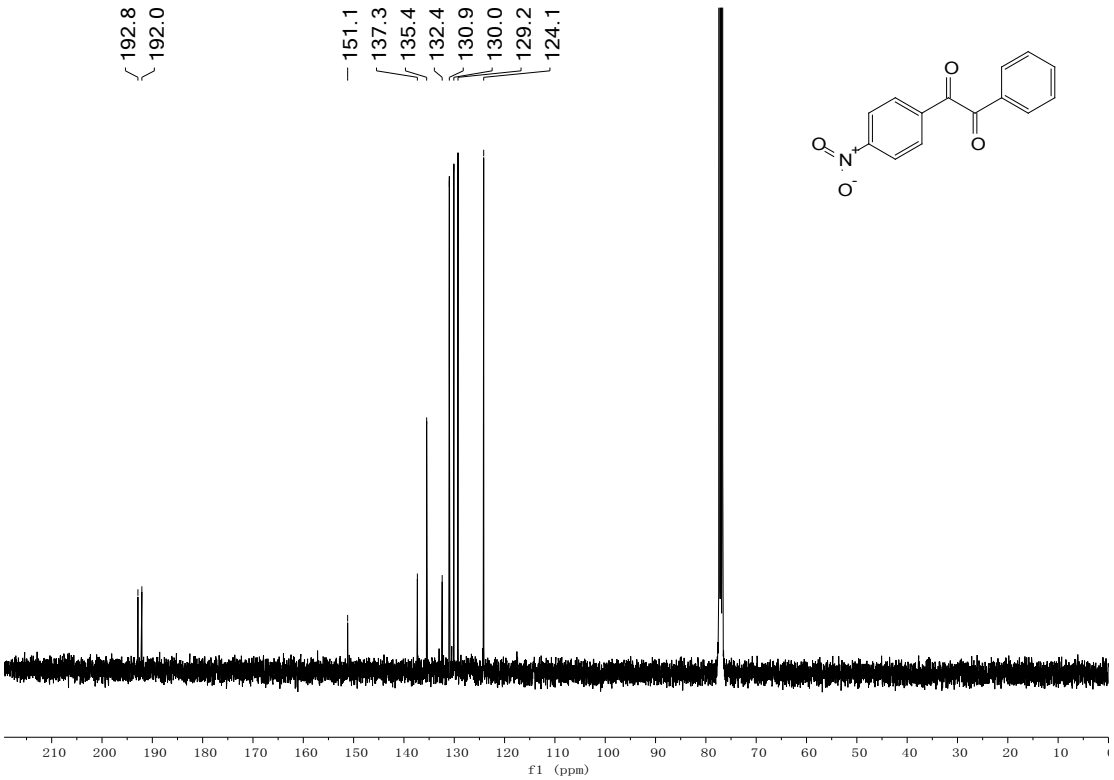
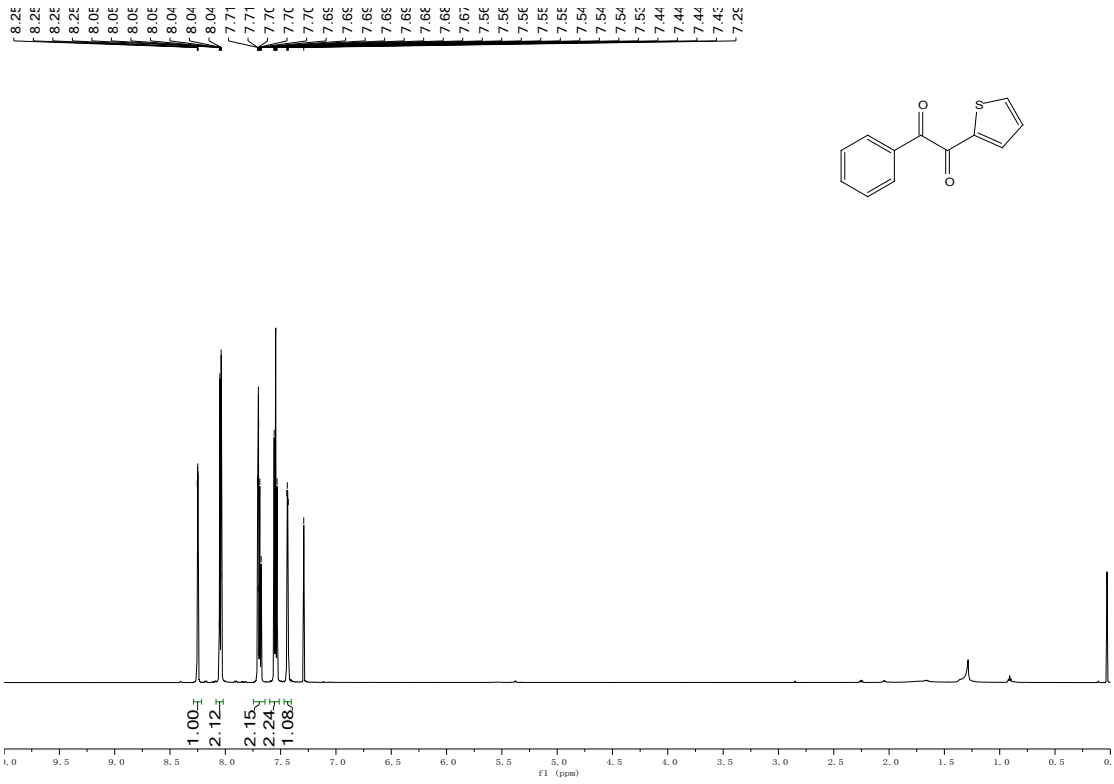
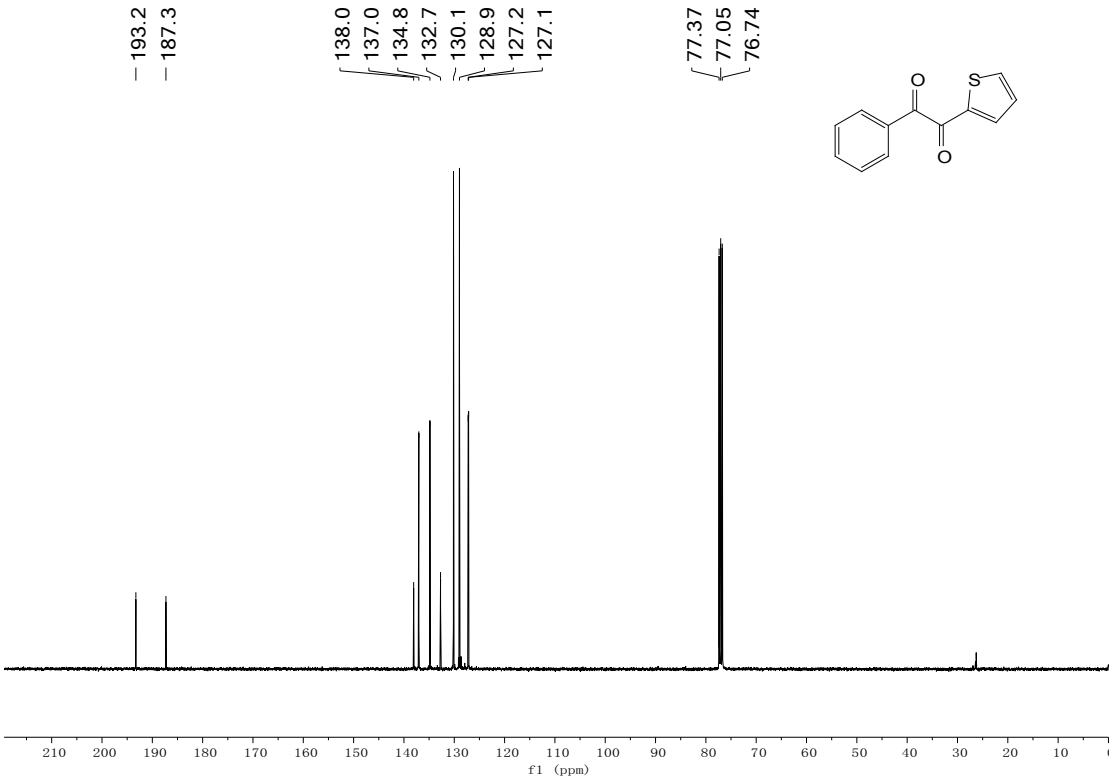
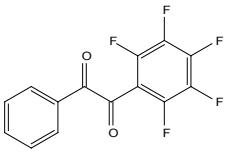
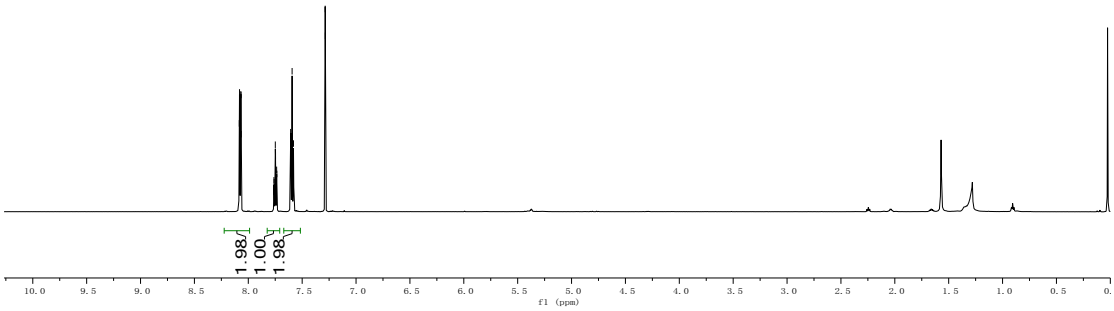
f1 (ppm)

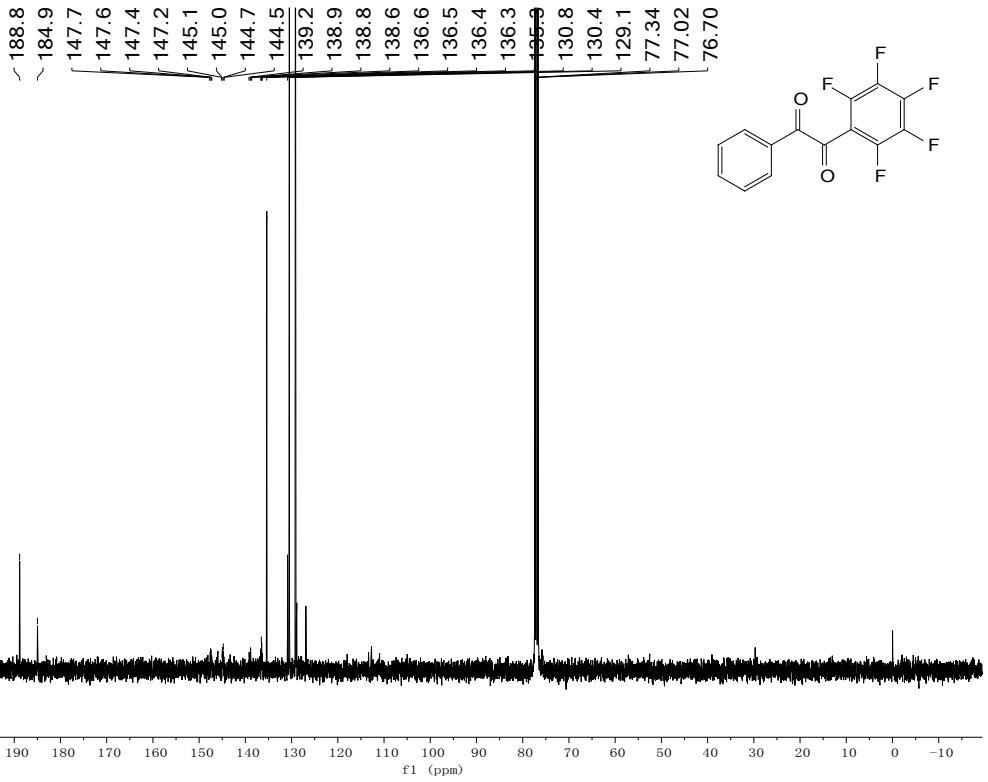

192.8
192.0
151.1
137.3
135.4
132.4
130.9
130.0
129.2
124.1
O
O
O
N+
O-
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


8.25
8.25
8.25
8.25
8.05
8.05
8.05
8.05
8.04
8.04
8.04
7.71
7.71
7.70
7.70
7.70
7.68
7.68
7.68
7.68
7.68
7.68
7.67
7.56
7.56
7.56
7.55
7.55
7.55
7.54
7.54
7.54
7.53
7.44
7.44
7.44
7.25
O
S
O
1.00
2.12
2.15
2.24
1.08
f1 (ppm)


193.2
187.3
138.0
137.0
134.8
132.7
130.1
128.9
127.2
127.1
77.37
77.05
76.74
O
S
O
f1 (ppm)


F
F
F
F
O
O
F


1.98
1.00
1.98
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
f1 (ppm)


188.8
184.9
147.7
147.6
147.4
147.2
145.1
145.0
144.7
144.5
139.2
138.9
138.8
138.6
136.6
136.5
136.4
136.3
130.8
130.4
129.1
77.34
77.02
76.70
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F
F
F
O
O
F
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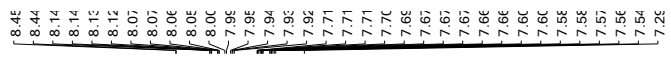
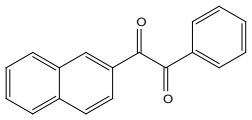
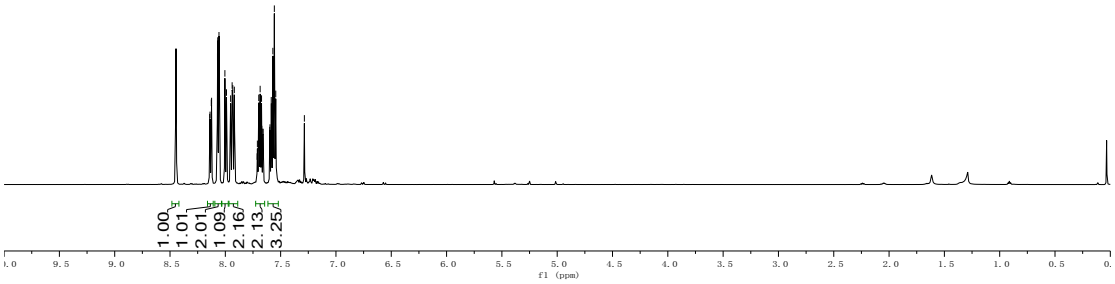
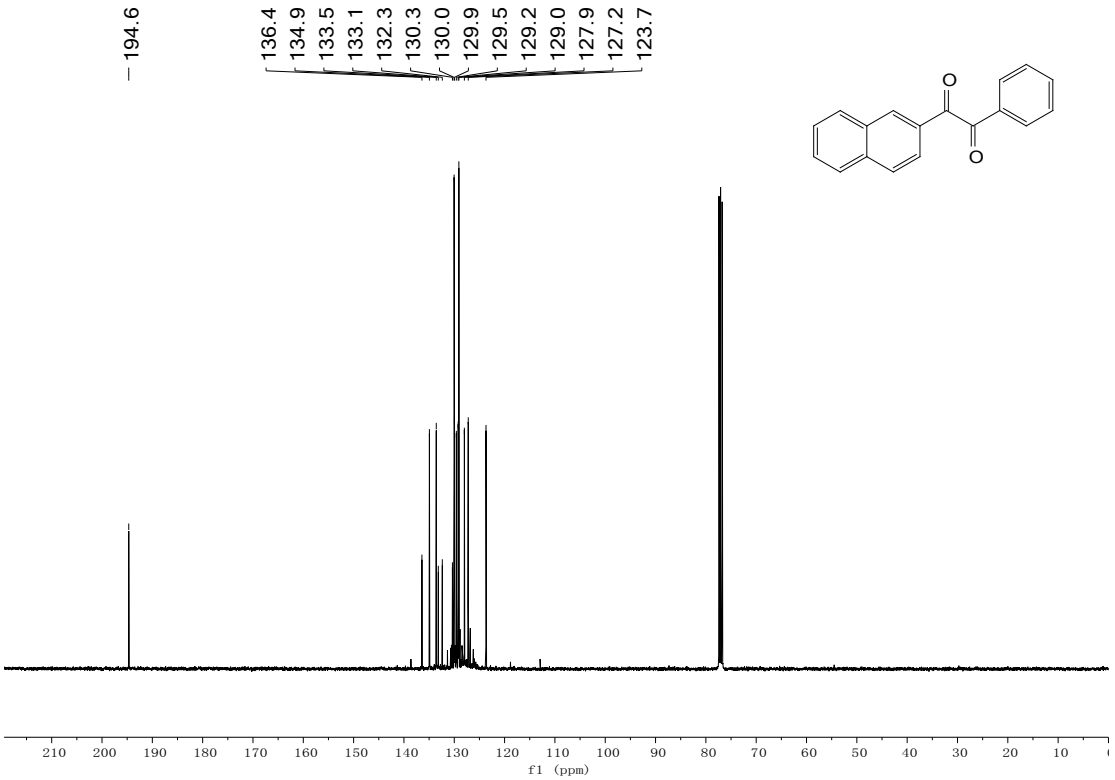
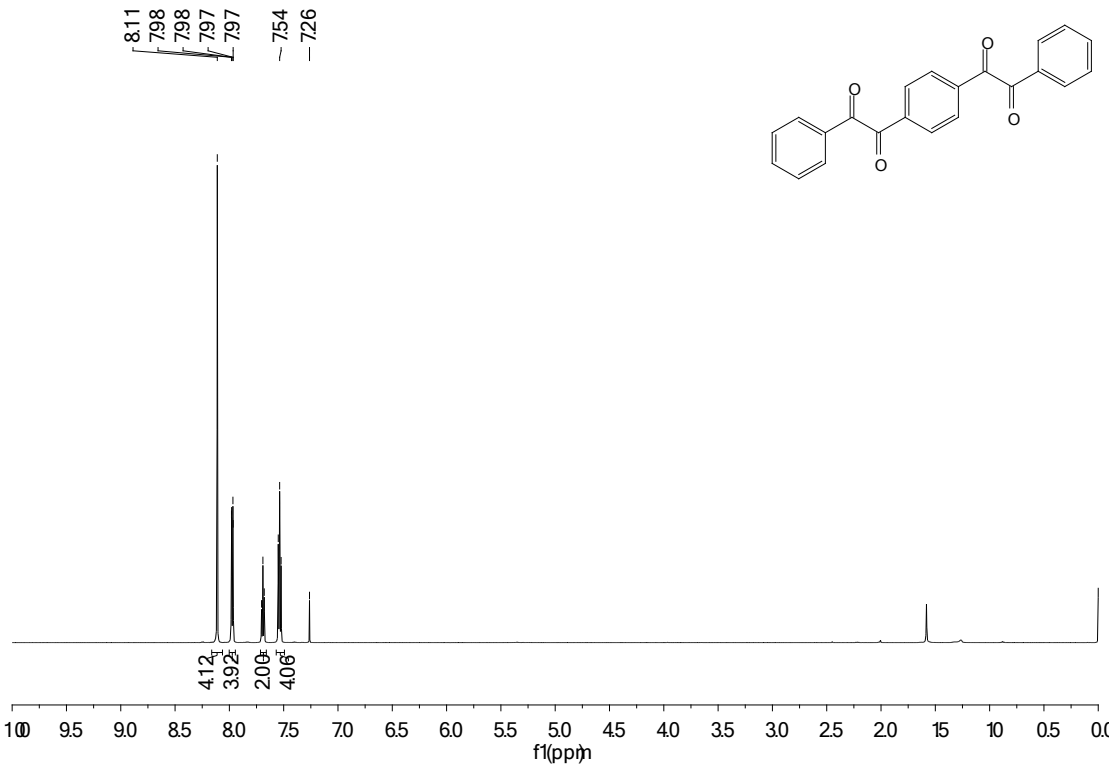
f1 (ppm)

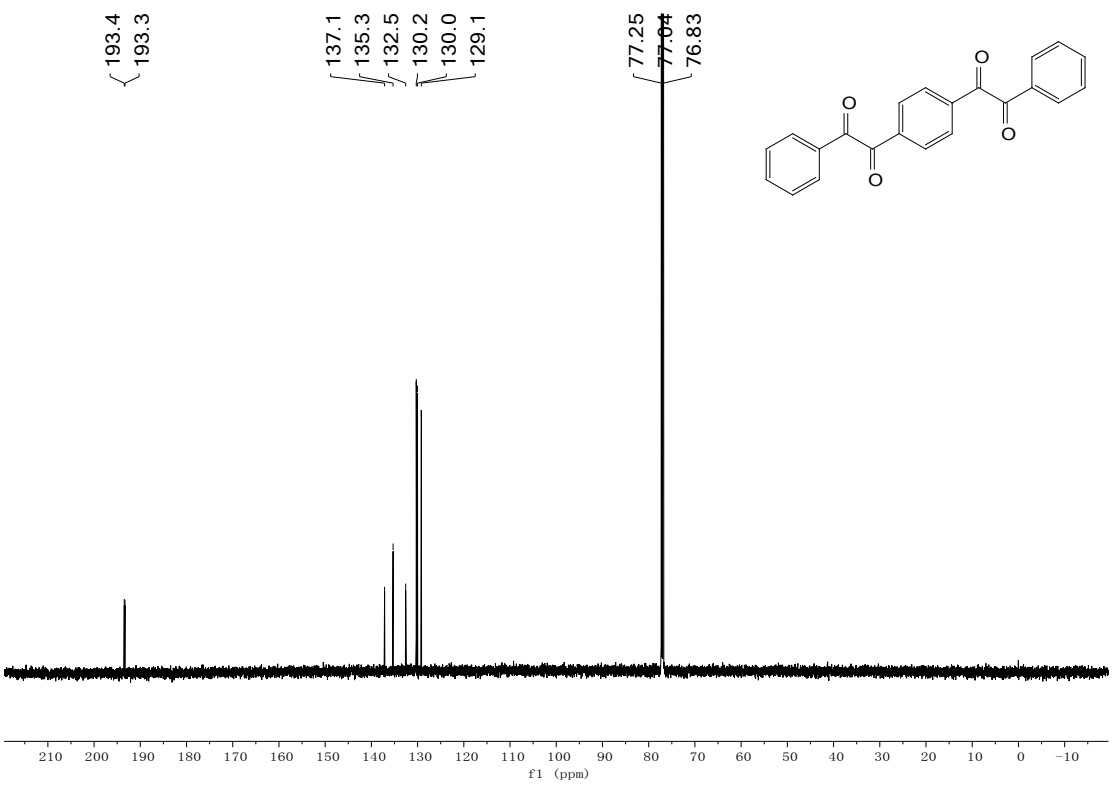


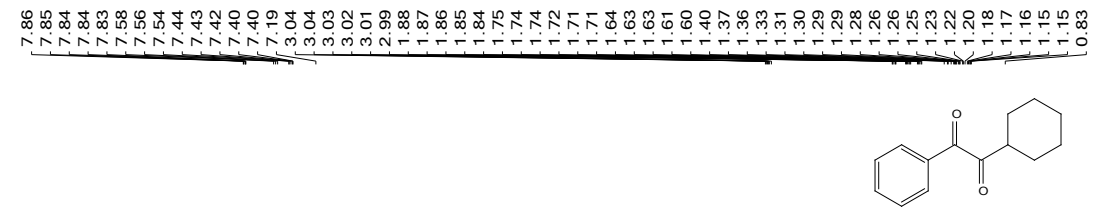



1.00
1.01
2.01
1.09
2.16
2.13
3.25
f1 (ppm)

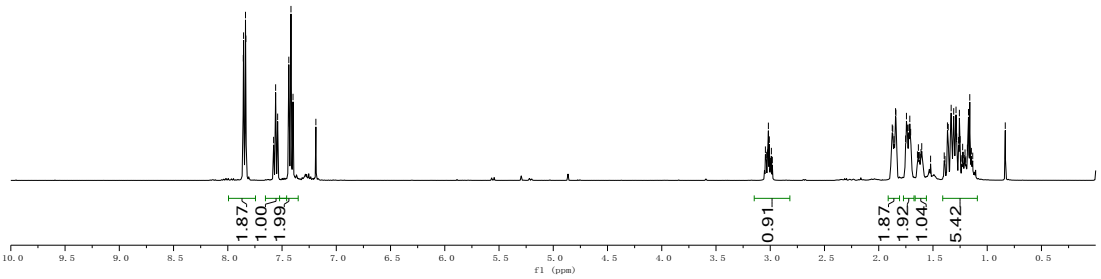

194.6
136.4
134.9
133.5
133.1
132.3
130.3
130.0
129.9
129.5
129.2
129.0
127.9
127.2
123.7
f1 (ppm)

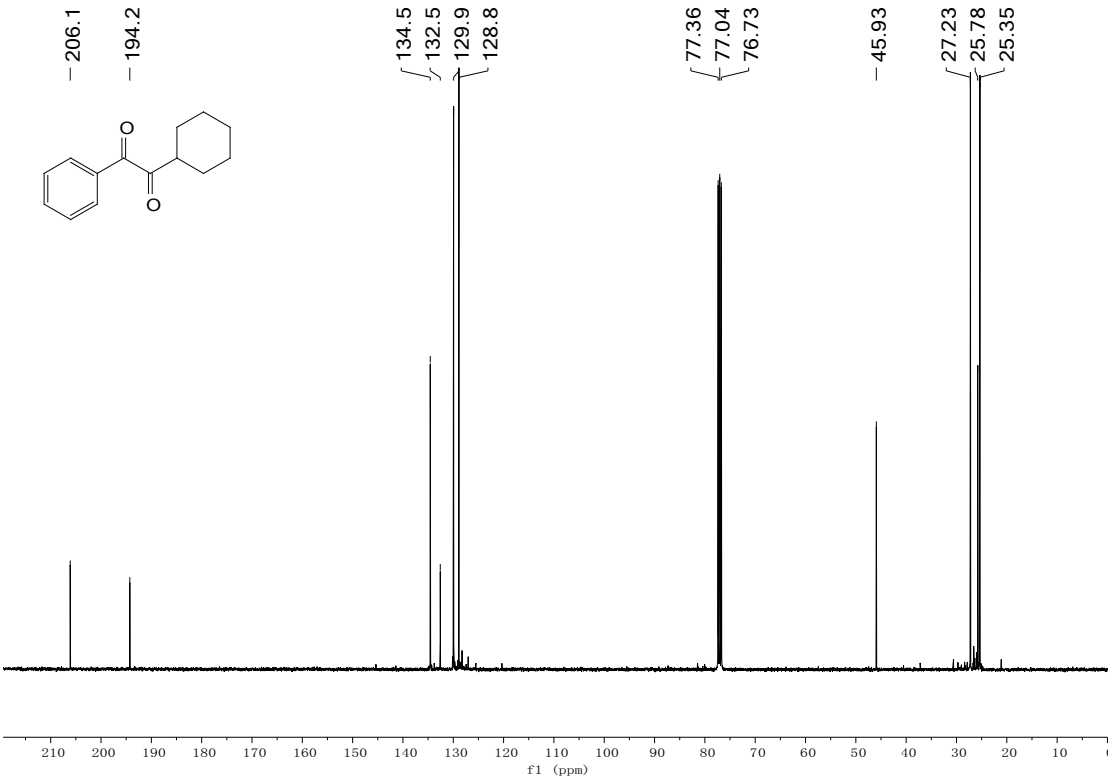

8.11
7.98
7.98
7.97
7.97
7.54
7.26
4.12
3.92
2.00
4.06
f1(ppm)

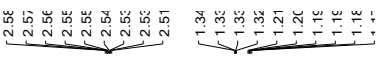

193.4
193.3
137.1
135.3
132.5
130.2
130.0
129.1
77.25
77.04
76.83
f1 (ppm)

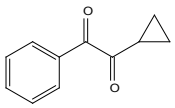

7.86
7.85
7.84
7.84
7.83
7.58
7.56
7.54
7.44
7.43
7.42
7.40
7.40
7.19
3.04
3.04
3.03
3.02
3.01
2.99
1.88
1.87
1.86
1.85
1.84
1.75
1.74
1.74
1.72
1.71
1.71
1.64
1.63
1.63
1.61
1.60
1.40
1.37
1.36
1.33
1.31
1.30
1.29
1.29
1.28
1.26
1.26
1.25
1.23
1.22
1.20
1.18
1.17
1.16
1.15
1.15
0.83

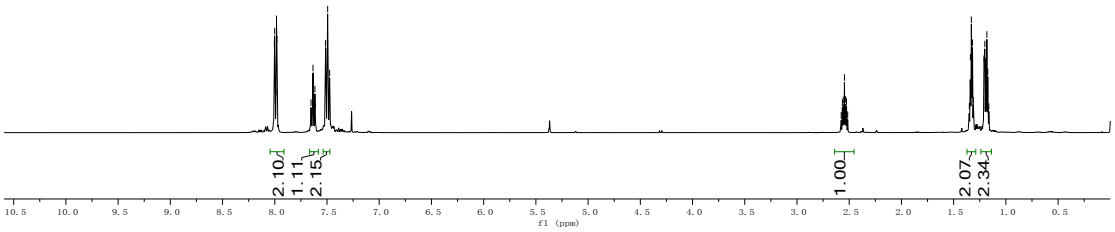
O
O

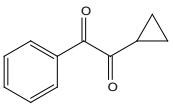

1.87
1.00
1.99
0.91
1.87
1.92
1.04
5.42
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

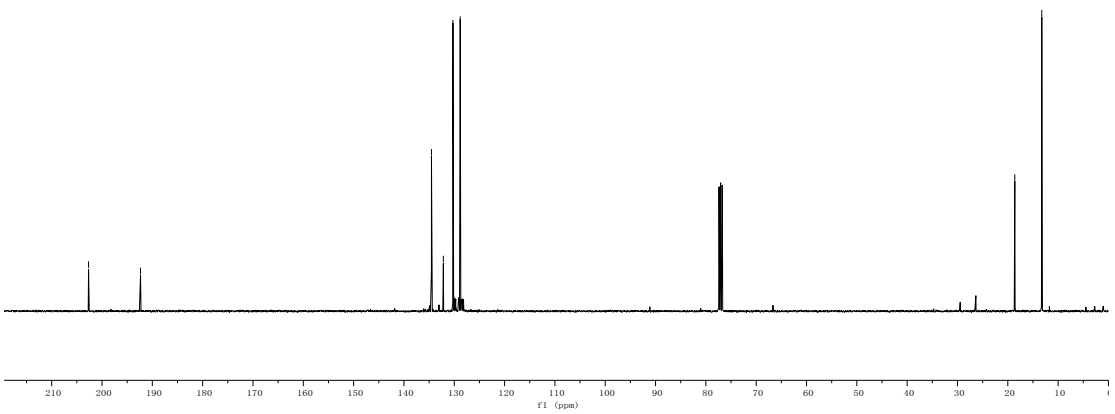

206.1
194.2
134.5
132.5
129.9
128.8
77.36
77.04
76.73
45.93
27.23
25.78
25.35
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

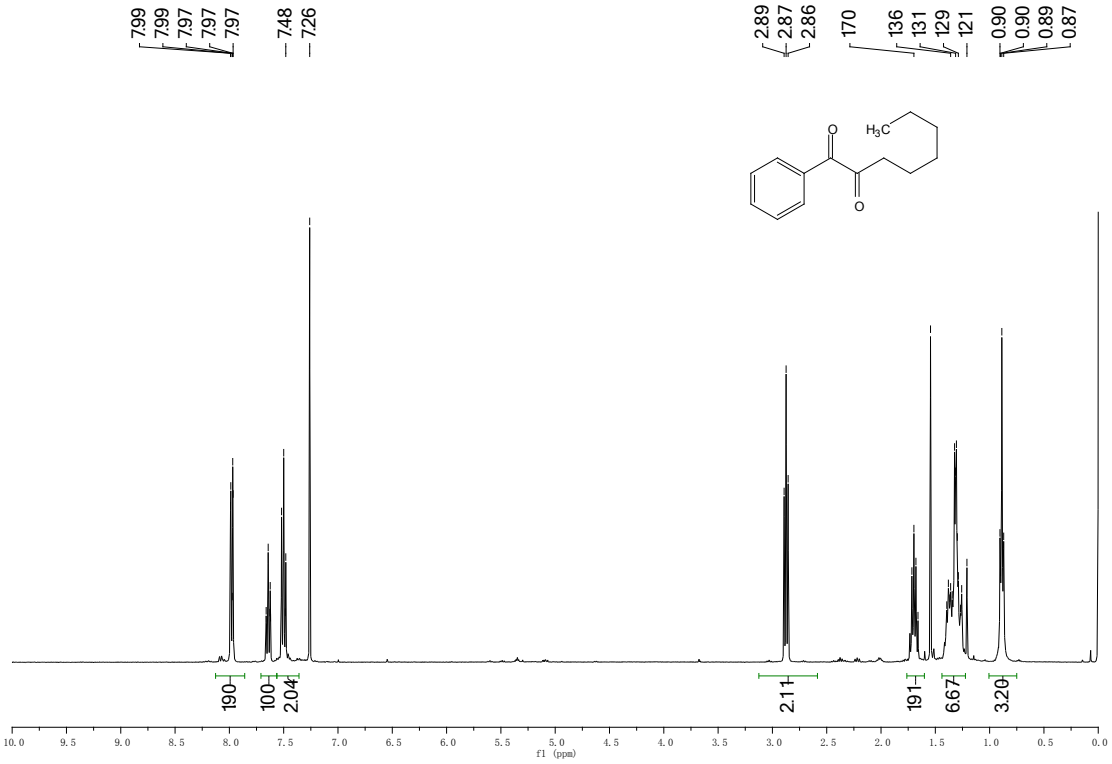


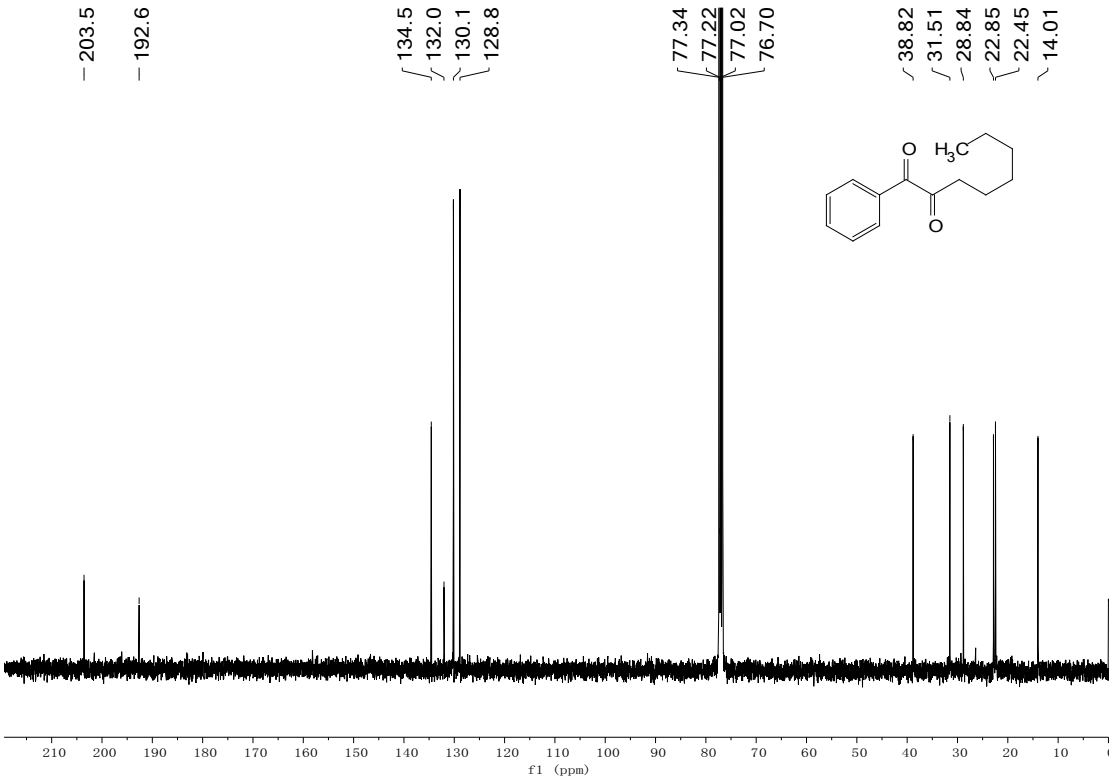


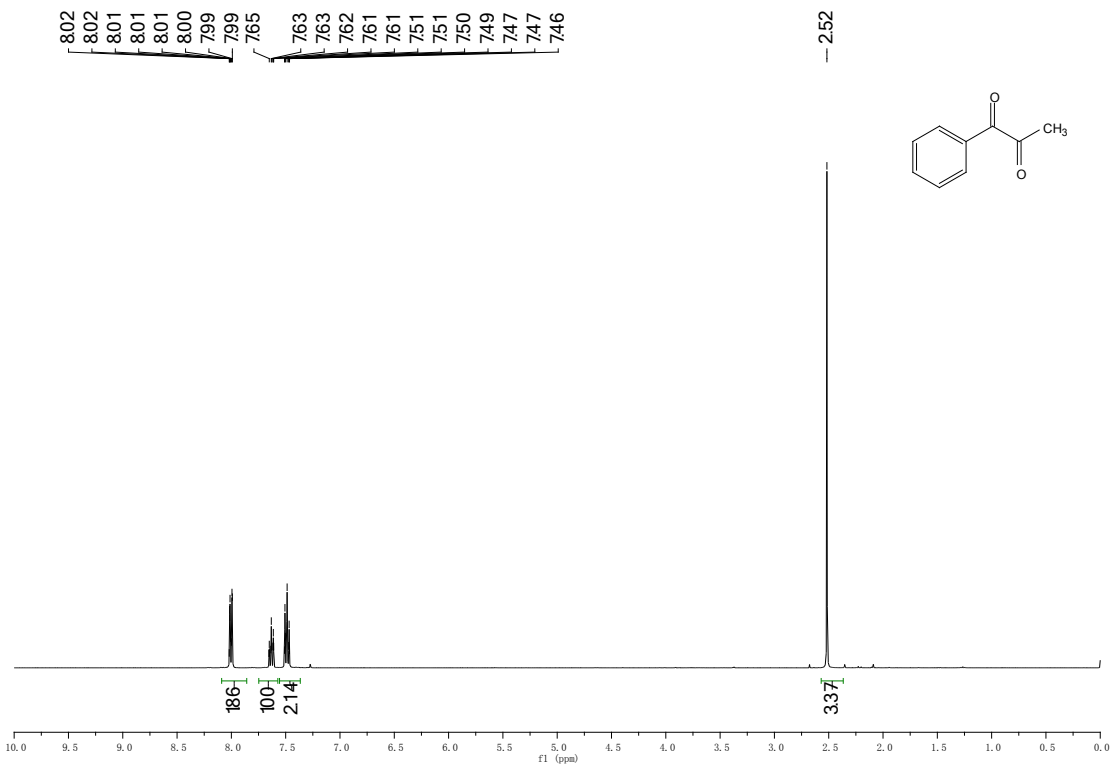

2.10
1.11
2.15
1.00
2.07
2.34
f1 (ppm)



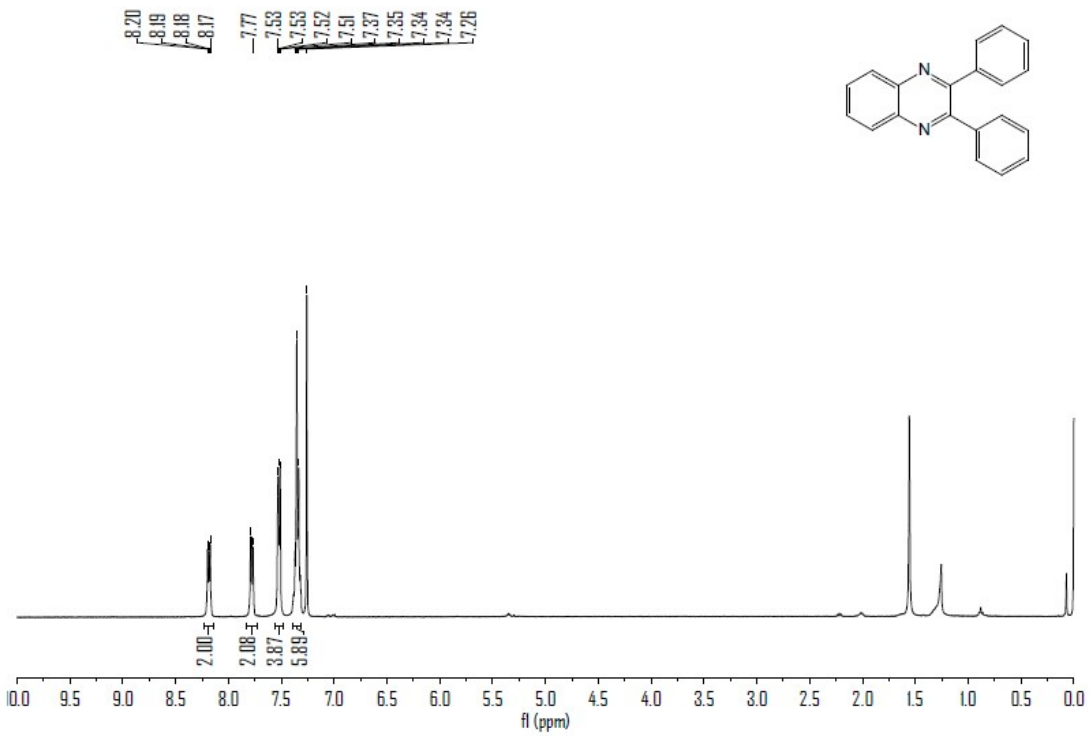

f1 (ppm)

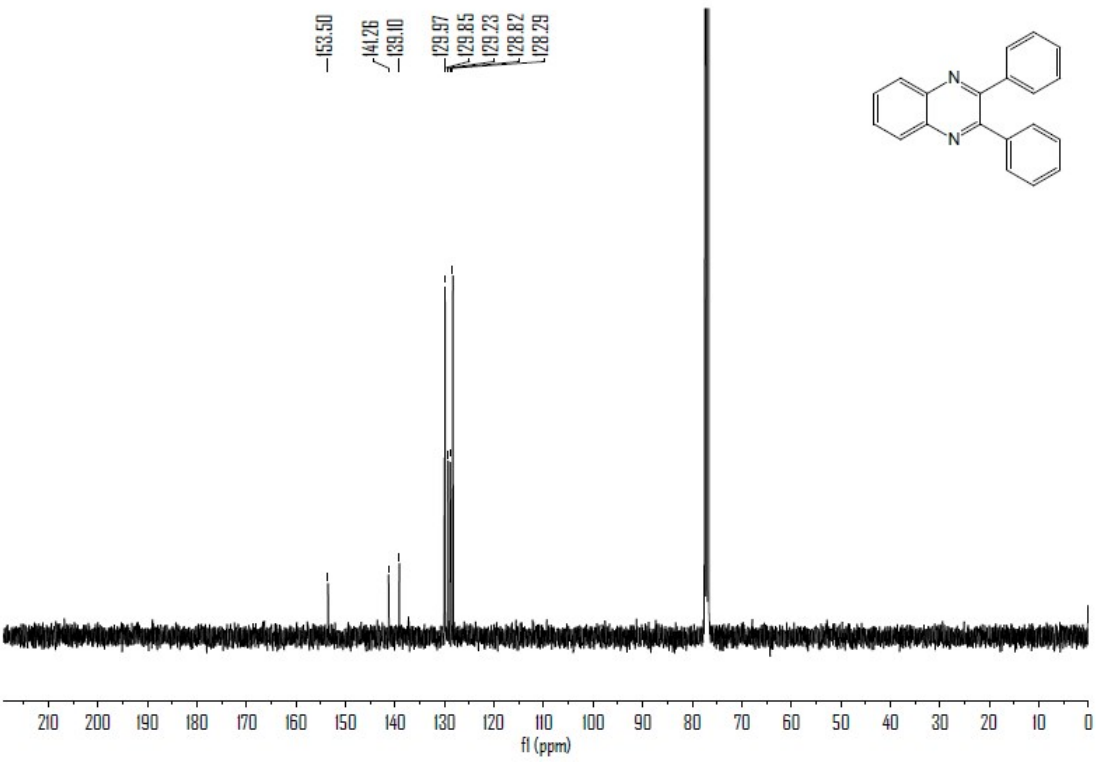

7.99
7.99
7.97
7.97
7.97
7.48
7.26
2.89
2.87
2.86
1.70
1.36
1.31
1.29
1.21
0.90
0.90
0.89
0.87
O
H3C
O
1.90
1.00
2.04
2.11
1.91
6.67
3.20
f1 (ppm)

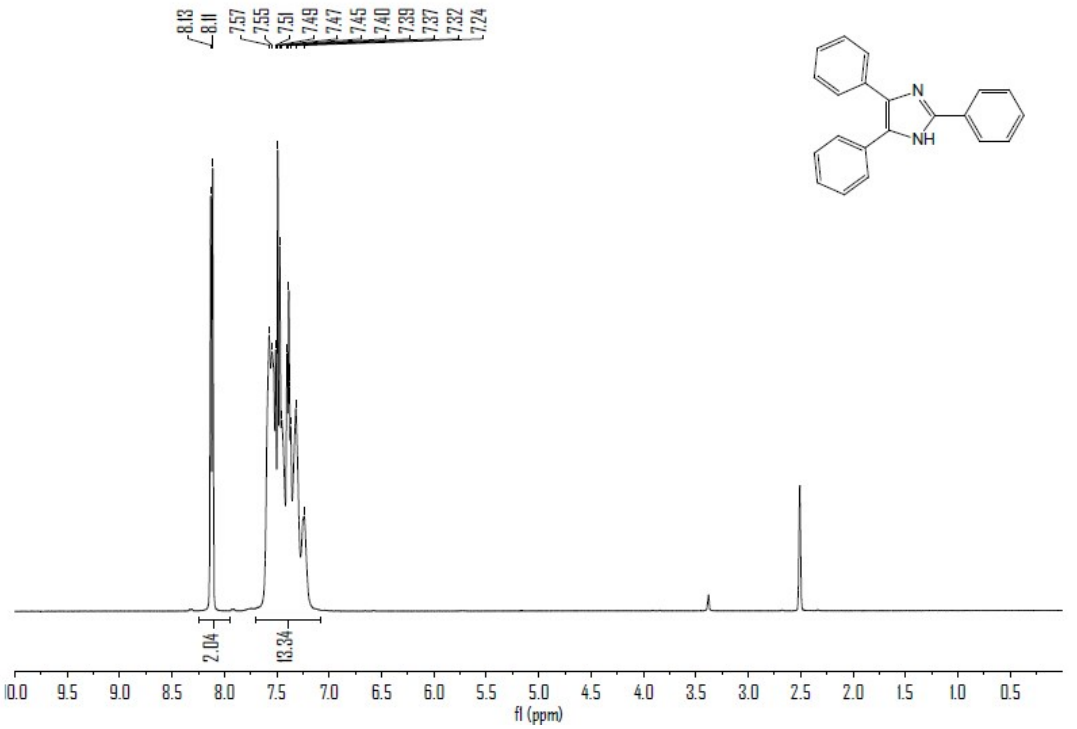

203.5
192.6
134.5
132.0
130.1
128.8
77.34
77.22
77.02
76.70
38.82
31.51
28.84
22.85
22.45
14.01
O
H3C
O
f1 (ppm)

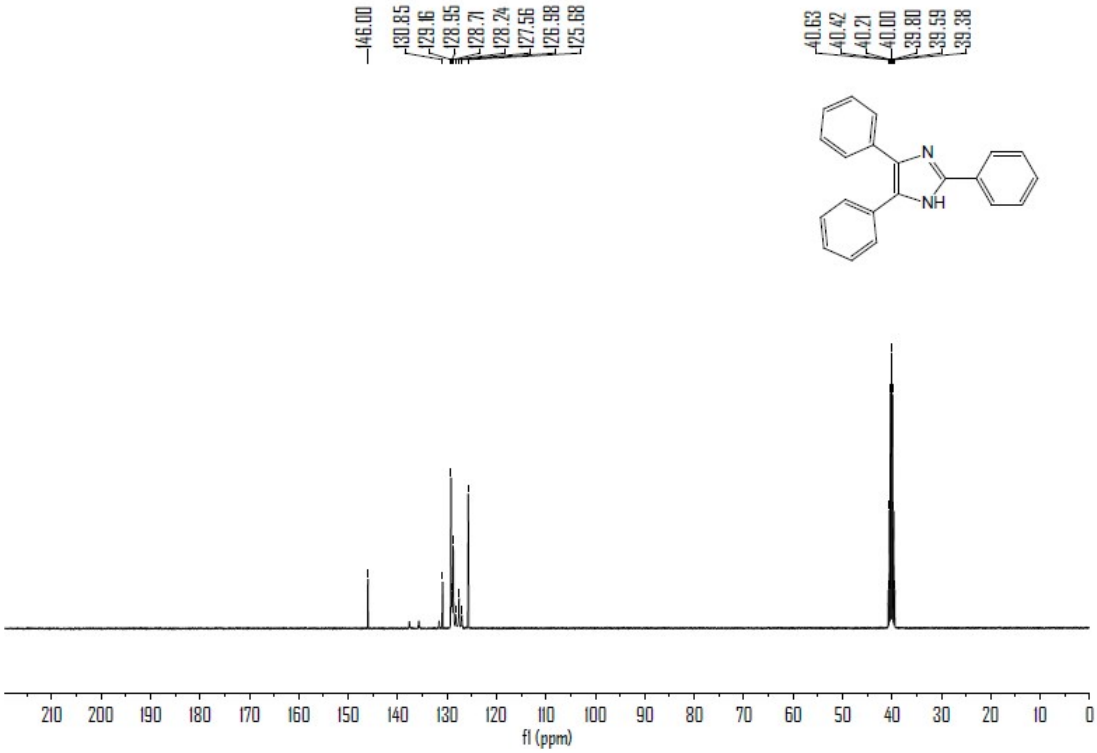

8.02
8.02
8.01
8.01
8.01
8.00
7.99
7.99
7.65
7.63
7.63
7.62
7.61
7.61
7.51
7.51
7.50
7.49
7.47
7.47
7.46
2.52
O
CH3
O
1.86
1.00
2.14
3.37
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


200.5
191.3
134.5
131.7
130.3
128.8
77.39
77.07
76.76
26.35
O
CH3
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


8.20
8.19
8.18
8.17
7.77
7.53
7.53
7.52
7.51
7.37
7.35
7.34
7.34
7.26
N
N
2.00
2.08
3.87
5.89
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)


153.50
141.26
139.10
129.97
129.85
129.23
128.82
128.29
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.13
8.11
7.57
7.55
7.51
7.49
7.47
7.45
7.40
7.39
7.37
7.32
7.24
N
NH
2.04
13.34
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
f1 (ppm)


146.00
130.85
129.16
128.95
128.71
128.24
127.56
126.98
125.68
40.63
40.42
40.21
40.00
39.80
39.59
39.38
N
NH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

6. Reference

1. Vellakkaran, M.; Andappan, M. M. S.; Kommu, N. Replacing a stoichiometric silver oxidant with air: ligated Pd (II)-catalysis to β-aryl carbonyl derivatives with improved chemoselectivity. *Green Chem.,* **2014***, 16,* 2788.
2. Chebolu, R.; Bahuguna, A.; Sharma, R.; Mishra, V. K.; Ravikumar, P. C. An unusual chemoselective oxidation strategy by an unprecedented exploration of an electrophilic center of DMSO: a new facet to classical DMSO oxidation. *Chem. Commun*., **2015**, *51*, 15438.
3. Cumming, J. N.; Smith, E. M.; Wang, L.; Misiaszek, J.; Durkin, J.; Pan, J.; Iserloh, U.; Wu, Y.; Zhu, Z.; Strickland, C.; Voigt, J.; Chen, X.; Kennedy, M. E.; Kuvelkar, R.; Kathleen, L. A. H.; Favreau, C. L.; Czarniecki, M. F.; Stamford, A. W. Structure based design of iminohydantoin BACE1 inhibitors: identification of an orally available, centrally active BACE1 inhibitor. *Bio. Med. Chem. Lett.,* **2012**, *22*, 2444.
4. Ruengsangtongkul, S.; Chaisan, N.; Thongsornkleeb, C.; Tummatorn, J.; Ruchirawat, S. Rate Enhancement in CAN-Promoted Pd $(PPh_3)_2Cl_2$-Catalyzed Oxidative Cyclization: Synthesis of 2-Ketofuran-4-carboxylate Esters. *Org. Lett*., **2019**, *21*, 2514.
5. Chen, H.; Zhang, L. A Desulfonylative Approach in Oxidative Gold Catalysis: Regiospecific Access to Donor‐Substituted Acyl Gold Carbenes. *Angew. Chem. Int. Ed.,* **2015**, *54*, 11775.
6. Chen, S.; Liu, Z.; Shi, E.; Chen, L.; Wei, W.; Li, H.; Cheng, Y.; Wan, X. Ruthenium-catalyzed oxidation of alkenes at room temperature: a practical and concise approach to α-diketones. *Org. Lett*., **2011**, *13*, 2274.
7. Wang, F., Cao, X., Mei, L., Zhang, X., Hu, J., & Tao, Y. (2018). Twisted penta‐Carbazole/Benzophenone Hybrid Compound as Multifunctional Organic Host, Dopant or Non‐doped Emitter for Highly Efficient Solution‐Processed Delayed Fluorescence OLEDs. *Chin. J. Chem.*, **2018**, *36*, 241.
8. Bansode, A. H.; Suryavanshi, G. Iodine-mediated oxidative rearrangement of α, β-unsaturated diaryl ketones: a facile access to 1, 2-diaryl diketones. *ACS Omega*, **2019**, *4*, 9636.
9. Zarnegar, Z.; Safari, J. Catalytic activity of Cu nanoparticles supported on Fe_3O_4-polyethylene glycol nanocomposites for the synthesis of substituted imidazoles *New J. Chem.* **2014**, *38*, 4555.