Supporting Information

Visible light-induced oxidative esterification of mandelic acid with alcohols: a new synthesis of α-ketoesters

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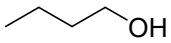
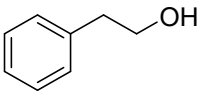
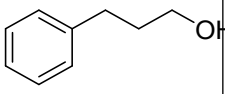
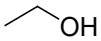
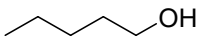
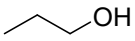
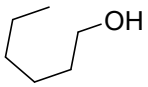
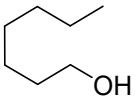
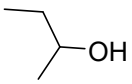
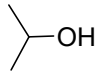
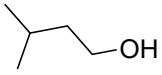
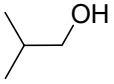
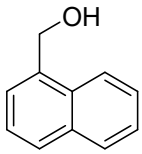
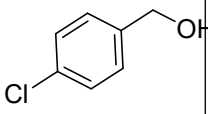
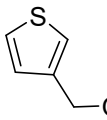
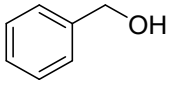
1. General considerations

All reagents were purchased at the highest commercial quality and used without further purification. Reactions were monitored by thin layer chromatography (TLC) using ultra violet light (UV) as the visualizing agent. Flash column chromatography was performed on silica gel (particle size 200−300 mesh) and eluted with petroleum ether/ethyl acetate. Nuclear magnetic resonance spectra (NMR) were recorded on Bruker AV-400 instruments and were calibrated using residual undeuterated solvent as an internal reference (^{1}H NMR: $CHCl_3$ 7.26 ppm, MeOH 3.31 ppm, ^{13}C NMR: $CHCl_3$ 77.16 ppm, MeOH 49.00 ppm). High resolution mass spectra (HRMS) were recorded on a Q-TOF mass spectrometer (Agilent G6545B, Germany). The following abbreviations were used to indicate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet).

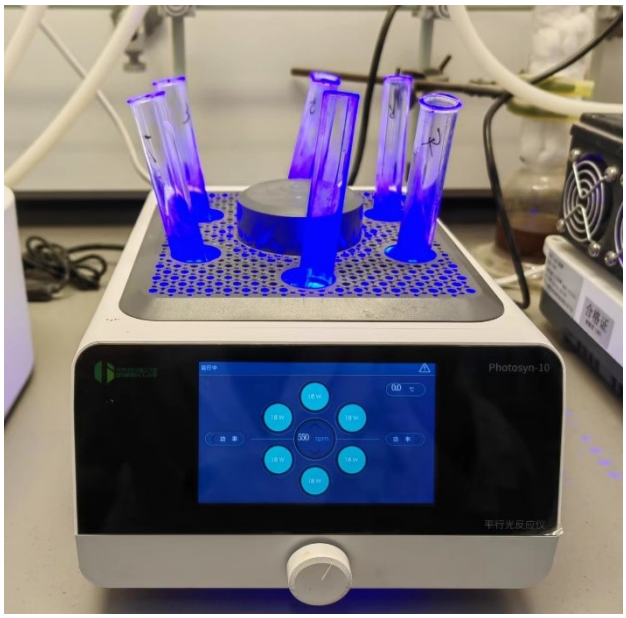
2. List of alcohols

Table S1 List of alcohols

Entry	Alcohol	Compound name	Entry	Alcohol	Compound name
1		**2a**	10		**2j**
2		**2b**	11		**2k**
3		**2c**	12		**2l**
4		**2d**	13		**2m**
5		**2e**	14		**2n**
6		**2f**	15		**2o**
7		**2g**	16		**2p**
8		**2h**	17		**2q**
9		**2i**			

3. Photoreactor set-up

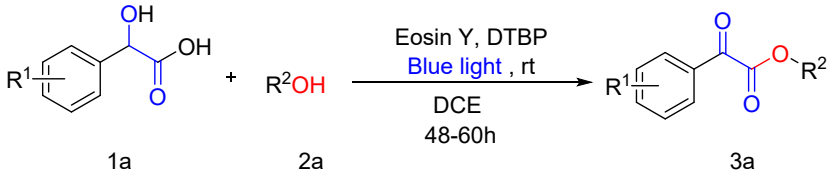
Photochemical experiments were carried out on a Photosyn-10 parallel photoreactor (18w blue LED), 455-460 nm, temperature controlled by fan cooling at the bottom, stirring speed 300-500 r/min).



4. General procedure for the oxidative esterification

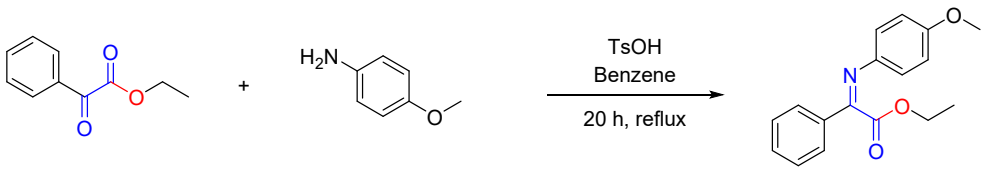
General procedure: A stir bar was added to a 25 mL test tube followed by **1** (0.2 mmol), **2** (0.5 mL or 0.5 mmol), DTBP (1 equiv.), Eosin Y (1 mol%). The reaction mixture was stirred at room temperature under the irradiation by 18 W blue LED for 48-60 h. The reaction was monitored by TLC. The solvent was evaporated under vacuum, and the crude product was purified using column chromatography with silica gel (200-300 mesh) or thin layer chromatography with silica gel (GF254) to give product **3**.

General procedure for the synthesis of of α-ketoesters 3a



25 mL Test tube were charged with mandelic acid **1a** (0.2 mmol), Eosin Y (0.002 mmol), alcohol **2a** (0.5 mL or 0.5 mmol solid alcohol), di-*tert*-butyl peroxide (0.2 mmol). Use DCE as a solvent when the alcohol is in the solid state. The mixture was irradiation with 18 W blue LEDs for 48-60 h at room temperature. Upon completion (TLC), the mixture was moved to the round bottom flask, and the reaction tube was washed additionally with ethyl acetate (5 mL) to fully transfer the residue. The solvent in the flask was then removed at reduced pressure, and the residue was purified by silica gel column chromatography with the elution of mixed ethyl acetate and petroleum ether (v/v = 1:30). For the reaction of solid alcohols, after the reaction completion, water (5 mL) was added to the vessel, and the suspension was extracted with ethyl acetate (3 × 5 mL). The combined organic phase was dried over Na_2SO_4. After filtering, the acquired solution was employed to reduced pressure to remove the organic solvent. And analogous chromatographic purification using mixed ethyl acetate and petroleum (v/v = 1:30) as eluent was executed to obtain corresponding products **3a**.

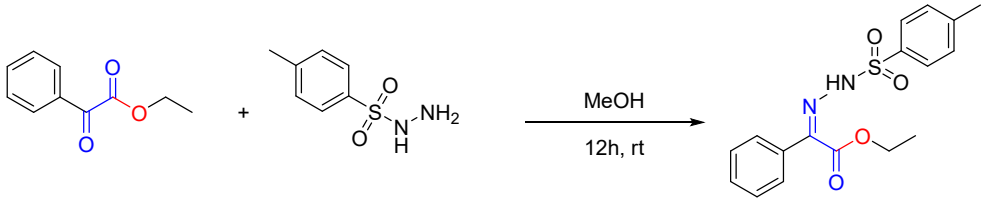
General procedure for the synthesis of 5a



3ac 5a

To a solution of *p*-anisidine (0.129 g, 1.05 mmol) in benzene (3 mL) were added ethyl 2-oxo-2-phenylacetate (0.159 mL, 1 mmol) and *p*-toluenesulfonic acid (9.51 mg, 0.05 mmol). The reaction mixture was heated at reflux temperature for 20 h with azeotropic removal. The solvent was evaporated, and the residue was purified by flash column chromatography (ethyl acetate/ petroleum ether = 1:20) to give the product **5a** in 90% yield.

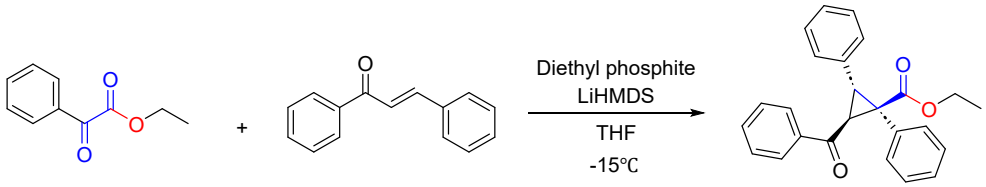
General procedure for the synthesis of 5b



3ac 5b

Ethyl 2-oxo-2-phenylacetate (1.00 mmol) was treated with 4-methylbenzenesulfonhydrazide (1.05 equiv.) in 20 mL of MeOH. The solution was stirred at rt for 12 h, the solvent was removed under reduced pressure giving a pale yellow oil which was dissolved in 20 mL of EtOAc and washed with 3 × 10 mL of water to remove the remaining hydrazide. The organic layer was dried with $MgSO_4$ and the solvent was removed under reduced pressure affording the product **5b** in 95% yield.

General procedure for the synthesis of 5c



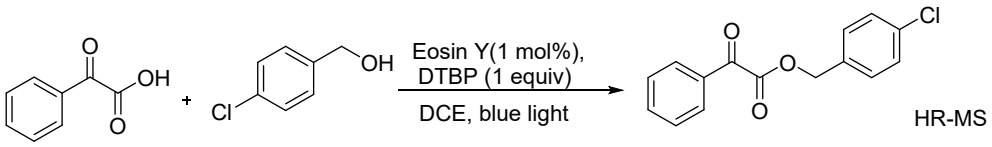
3ac 5c

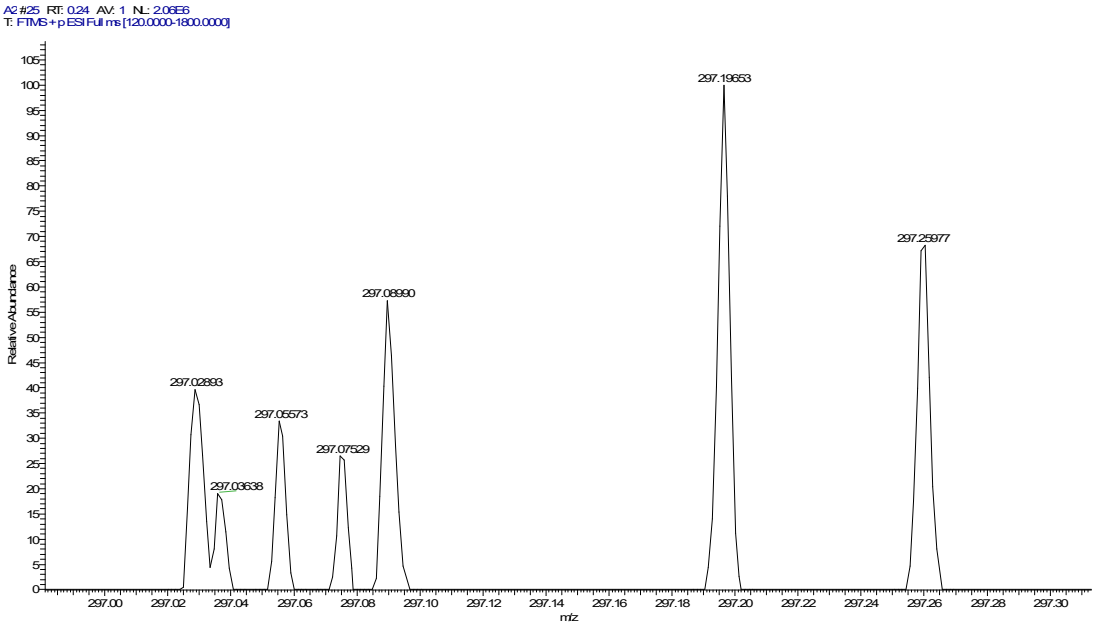
Ethyl 2-oxo-2-phenylacetate (0.60 mmol, 2.0 equiv.) and dimethyl phosphite or diethyl phosphite (0.60 mmol, 2.0 equiv.) in 2.0 mL THF were added to a flame-dried Schlenk flask equipped with a magnetic stirring bar and purged with argon. The

solution was cooled to -15 °C. A THF solution of 1.2 M LiHMDS (0.50 mL, 0.60 mmol, 2.0 equiv.) in 1.0 mL of THF was added dropwise at -15 °C. After another 10 min, a solution of chalcone (0.30 mmol, 1.0 equiv.) in 1.0 mL of THF was added dropwise. The reaction was allowed to proceed at the same temperature and monitored by TLC. Once the chalcone is completely consumed (usually 1.5-2 hours), the reaction mixture is quenched with saturated aqueous ammonium chloride. After being warmed to ambient temperature, the mixture was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography to give the desired product **5c** in 97% yield.

5. Control experiments

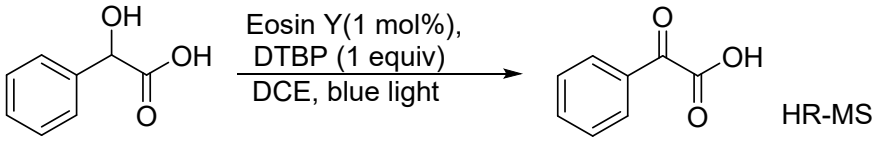
We assumed that phenylglyoxalic acid is the intermediate. Therefore, the reaction of phenylglyoxalic acid with *p*-chlorobenzyl alcohol under standard conditions was found to give the target product by GC-MS.

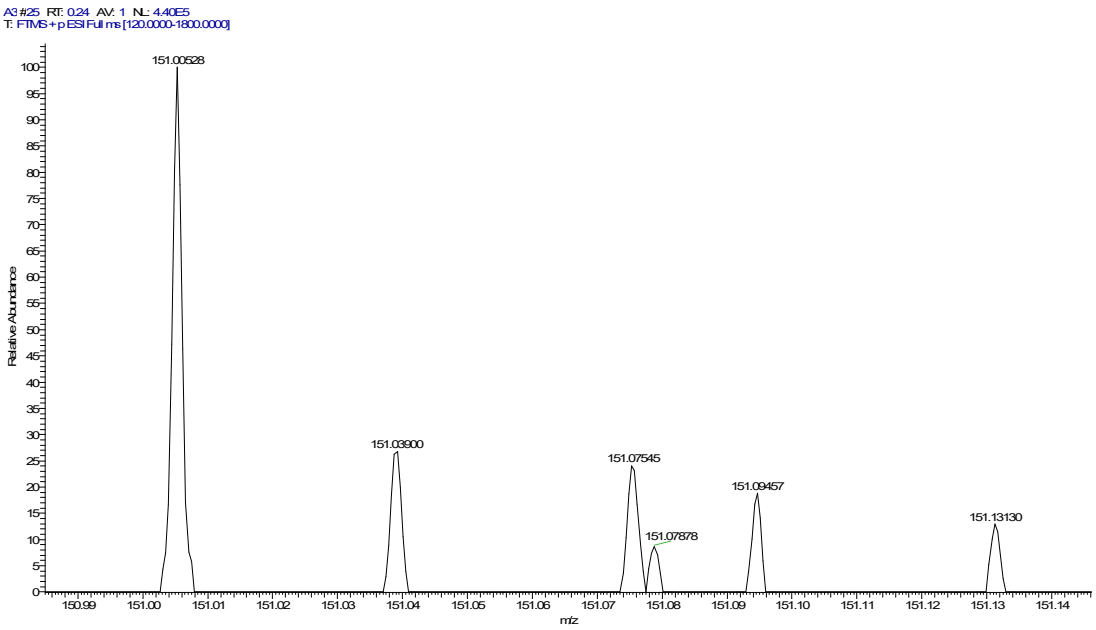




HRMS (EI) m/z: $[M+Na]^+$ Calcd for $C_{15}H_{11}ClO_3$: 297.02889; Found: 297.02893.

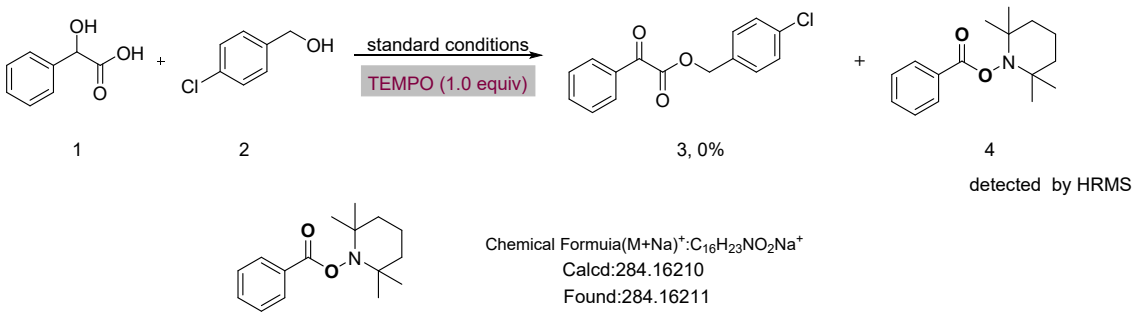
Mandelic acid alone was reacted under standard conditions and the peak of phenylglyoxalic acid was detected by HRMS and its presence was determined.



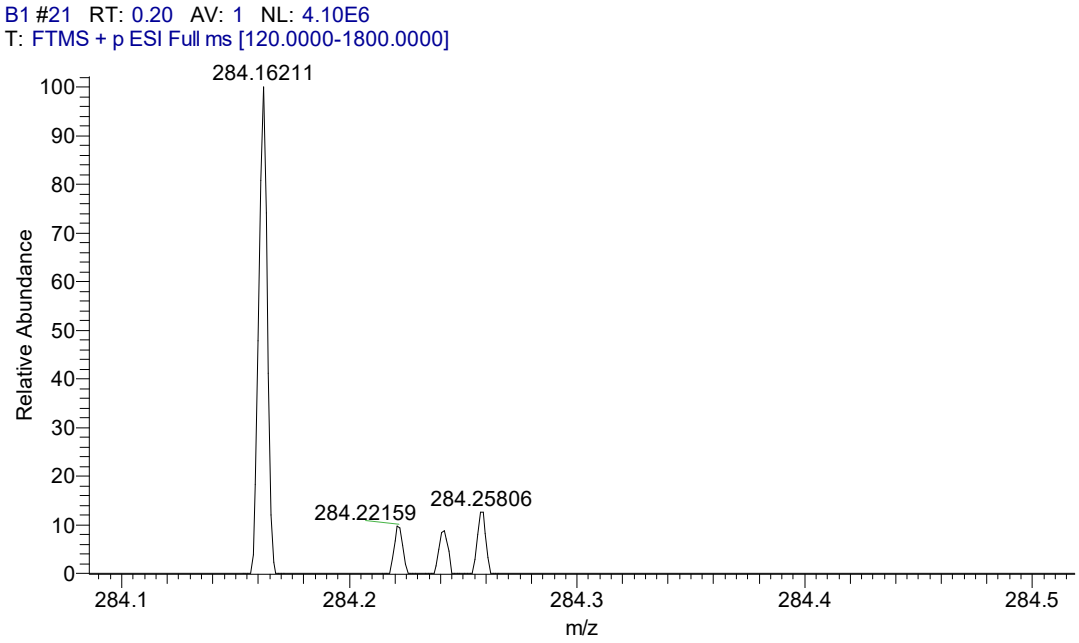


HRMS (EI) m/z: $[M+H]^+$ Calcd for $C_8H_6O_3$: 151.03897; Found: 151.03900.

6. Radical inhibition experiment



Chemical Formuia$(M+Na)^+$:$C_{16}H_{23}NO_2Na^+$
Calcd:284.16210
Found:284.16211



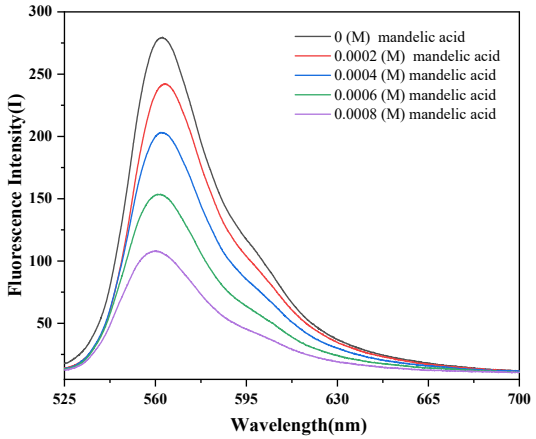
The reaction was nearly completely inhibited by TEMPO. The N-O compound was detected by HRMS, which suggest that a benzophenone radical might be involved in this transformation. These results indicated that the reaction probably proceeded via a free radical process.

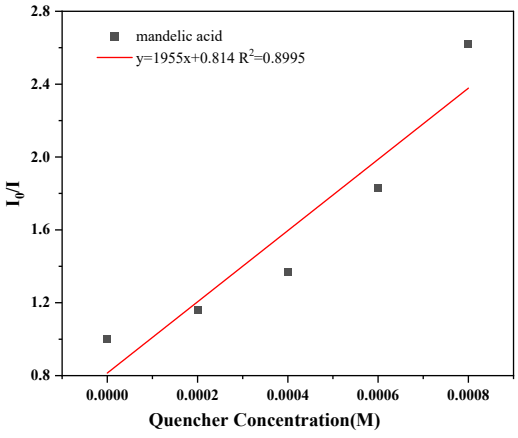
7. Stern-Volmer fluorescence quenching experiments

Formulation solution: 1a (7.6 mg) was dissolved in MeCN in a 5 mL volumetric flask to set the concentration to be 0.01 M. Butyl alcohol (22 μL) was dissolved in MeCN in a 25 mL volumetric flask to set the concentration to be 0.01 M. Dissolve the photocatalyst Eosin Y (1.6 mg) in MeCN in a 25 mL volumetric flask, shake well, take out 5 mL of the solution and make up to volume with MeCN in a 25 mL volumetric flask, setting the concentration to 0.02 mM.

Experimental procedure: The resulting 0.02 mM solution (20 μL) was added to cuvette to obtain different concentrations of catalyst solution. This solution was then diluted to a volume of 2.0 mL by adding MeCN to prepare a 0.2 μM solution. 40.0 μL of a mandelic acid solution was successively added and uniformly stirred and irradiated at 514 nm. Fluorescence emission spectra of 0 μL, 40.0 μL, 80.0 μL, 120.0 μL, 160.0 μL fluorescence intensity was recorded. Follow this method and make changes to the amount to obtain the Stern–Volmer relationship in turn. The solution was excited at λ= 514 nm.

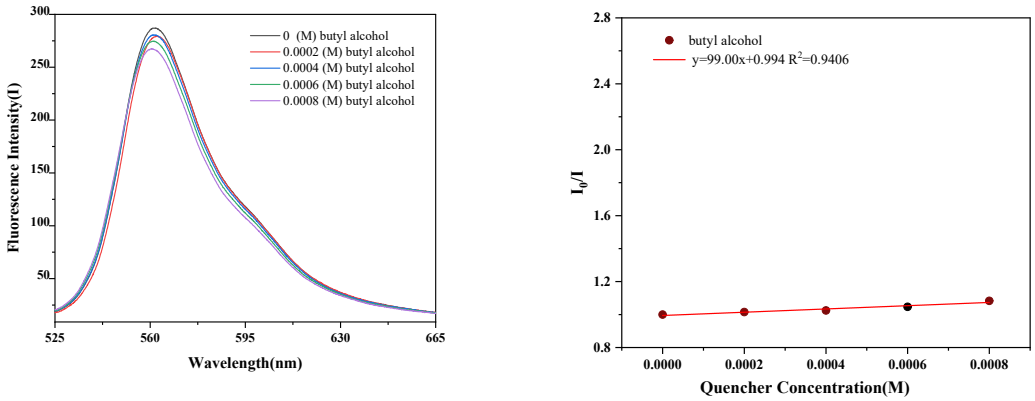
(a) Eosin Y quenched by mandelic acid in MeCN.





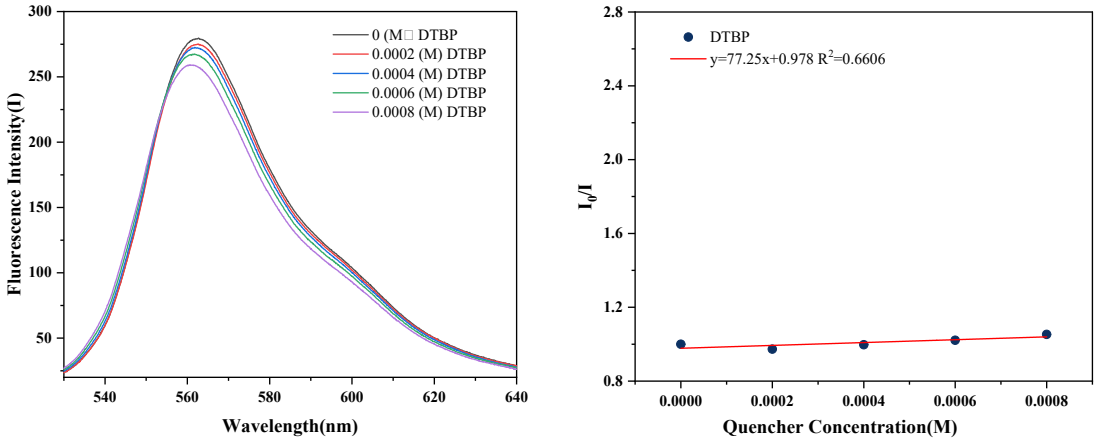
The emission intensity of Eosin Y catalyst solutions was affected by a gradual increase in the amount of mandelic acid.

(b) Eosin Y quenched by butyl alcohol in MeCN



The emission intensity of the Eosin Y catalyst solution affected by the gradual increase of the amount of butyl alcohol.

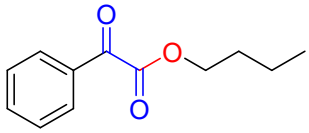
(c) Eosin quenched by DTBP in MeCN



The emission intensity of the Eosin Y catalyst solution affected by the gradual increase of the amount of DTBP.

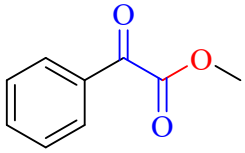
8. Characterization data of products

Butyl 2-oxo-2-phenylacetate (3aa)



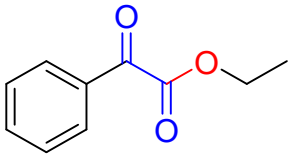
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3aa**, yellow oil, (70%, 28.8 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02-7.98 (m, 2H), 7.69-7.62 (m, 1H), 7.54-7.46 (m, 2H), 4.39 (t, *J* = 6.7 Hz, 2H), 1.80-1.72 (m, 2H), 1.46 (dt, *J* = 15.0, 7.4 Hz, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 186.64, 164.14, 135.02, 132.62, 130.13, 129.03, 66.24, 30.59, 19.16, 13.76. Known compound, the spectroscopic data are consistent with previous report.[1]

Methyl 2-oxo-2-phenylacetate (3ab)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ab**, yellow oil, (73%, 23.9 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05-7.95 (m, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 1H), 3.97 (s, 1H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 186.55, 164.03, 135.52, 131.74, 129.79, 129.76, 129.31, 129.29, 53.03. Known compound, the spectroscopic data are consistent with previous report.[1]

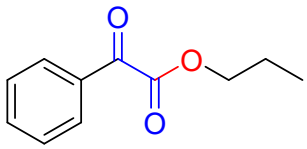
Ethyl 2-oxo-2-phenylacetate (3ac)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ac**, yellow oil, (68%, 24.2 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03-7.93 (m, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 4.43 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* =

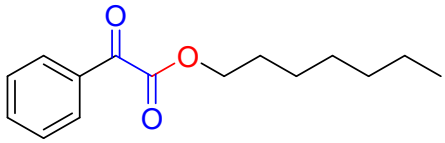
7.1 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 186.54, 164.03, 135.51, 131.74, 129.79, 129.30, 129.28, 53.02. Known compound, the spectroscopic data are consistent with previous report.[1]

Propyl 2-oxo-2-phenylacetate (3ad)



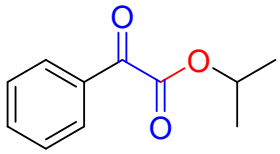
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ad**, yellow oil, (64%, 24.5 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02-7.97 (m, 2H), 7.69-7.61 (m, 1H), 7.55-7.47 (m, 2H), 4.35 (t, *J* = 6.7 Hz, 2H), 1.87-1.74 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H). ^{13} C NMR (101 MHz, DMSO-d_6) δ 186.80, 163.83, 135.51, 131.70, 129.67, 129.33, 21.32, 10.09. Known compound, the spectroscopic data are consistent with previous report.[1]

Heptyl 2-oxo-2-phenylacetate (3ae)



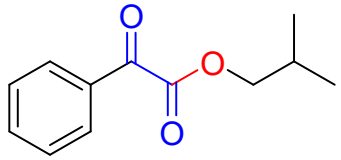
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ae**, yellow oil, (60%, 29.7 mg); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (dt, *J* = 8.5, 1.6 Hz, 2H), 7.70-7.59 (m, 1H), 7.54-7.44 (m, 2H), 4.38 (t, *J* = 6.8 Hz, 2H), 1.84-1.71 (m, 2H), 1.45-1.36 (m, 2H), 1.34-1.24 (m, 6H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 186.78, 163.80, 135.51, 131.70, 129.65, 129.31, 31.10, 28.18, 27.82, 25.18, 21.97, 13.87. HRMS (ESI) Calcd. for $C_{15}H_{20}O_3$ $[M+H]^+$: 249.14852. Found: 249.14778.

Isopropyl 2-oxo-2-phenylacetate (3af)



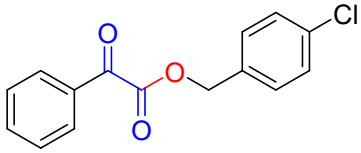
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3af**, yellow oil, (70%, 26.8 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.94 (dd, J = 8.2, 1.1 Hz, 2H), 7.80 (t, J = 7.4 Hz, 1H), 7.64 (t, J = 7.7 Hz, 2H), 5.26 (dt, J = 12.5, 6.3 Hz, 1H), 1.35 (d, J = 6.3 Hz, 6H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 186.96, 163.43, 135.51, 131.66, 129.60, 129.58, 129.40, 70.81, 21.37. Known compound, the spectroscopic data are consistent with previous report.[1]

Isobutyl 2-oxo-2-phenylacetate (3ag)



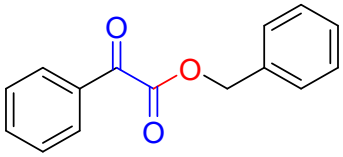
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ag**, yellow oil, (75%, 30.9 mg); ^{1}HNMR (400 MHz, Chloroform-d) δ 8.01 (dd, J = 8.4, 1.3 Hz, 2H), 7.66 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.7 Hz, 2H), 4.18 (d, J = 6.7 Hz, 2H), 2.09 (dp, J = 13.4, 6.7 Hz, 1H), 1.00 (d, J = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 186.80, 163.81, 135.54, 131.69, 129.65, 129.35, 71.64, 27.16, 18.66. Known compound, the spectroscopic data are consistent with previous report.[2]

4-Chlorobenzyl 2-oxo-2-phenylacetate (3ah)



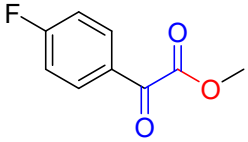
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ah**, white solid, mp: 54.8–57.1 °C, (70%, 38.3 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.98 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.3 Hz, 2H), 7.48 (dd, J = 17.7, 8.3 Hz, 5H), 5.34 (s, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.68, 138.45, 134.94, 132.82, 131.09, 129.92, 128.99, 128.52, 128.28, 65.65. Known compound, the spectroscopic data are consistent with previous report.[3]

Benzyl 2-oxo-2-phenylacetate (3ai)



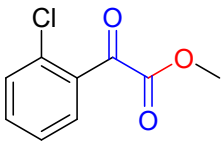
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ai** yellow oil, (71%, 34 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.02 (dt, J = 8.4, 1.5 Hz, 2H), 7.65 (t, J = 7.4 Hz, 1H), 7.54-7.46 (m, 4H), 7.42-7.34 (m, 3H), 5.36 (s, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.98, 136.54, 133.83, 130.01, 129.62, 129.20, 128.93, 128.52, 128.38, 66.59. Known compound, the spectroscopic data are consistent with previous report.[1]

Methyl 2-(4-fluorophenyl)-2-oxoacetate (3bb)



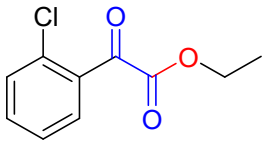
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3bb**, white solid, mp: 50.9-52.6 °C, (38%, 13.8 mg); ^{1}H NMR (600 MHz, Chloroform-d) δ 8.12- 8.07 (m, 2H), 7.21-7.16 (m, 2H), 3.98 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-d) δ 184.28, 167.88, 166.17, 163.76, 133.23, 133.17, 129.14, 129.13, 116.51, 116.37, 53.05. Known compound, the spectroscopic data are consistent with previous report.[4]

Methyl 2-(2-chlorophenyl)-2-oxoacetate (3cb)



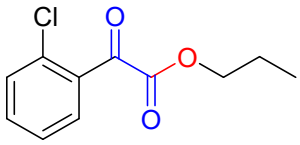
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3cb**, yellow oil, (57%, 22.5 mg); ^{1}H NMR (600 MHz, Chloroform-d) δ 7.75 (dd, J = 7.7, 1.5 Hz, 1H), 7.52 (td, J = 8.0, 1.6 Hz, 1H), 7.44 (d, J = 7.9 Hz, 1H), 7.40 (t, J = 7.6 Hz, 1H), 3.95 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-d) δ 186.35, 163.57, 134.51, 134.04, 133.38, 131.73, 130.70, 127.41, 77.37, 77.16, 76.95, 53.40. Known compound, the spectroscopic data are consistent with previous report.[4]

Ethyl 2-(2-chlorophenyl)-2-oxoacetate (3cc)



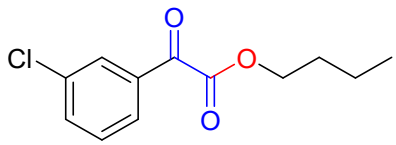
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3cc**, yellow oil, (41%, 17.3 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.76 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.52 (td, *J* = 7.9, 1.7 Hz, 1H), 7.46-7.42 (m, 1H), 7.42-7.38 (m, 1H), 4.42 (d, *J* = 7.2 Hz, 2H), 1.40 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 186.75, 163.23, 134.42, 134.00, 133.50, 131.77, 130.69, 127.39, 62.95, 14.01. Known compound, the spectroscopic data are consistent with previous report.[5]

Propyl 2-(2-chlorophenyl)-2-oxoacetate (3cd)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3cd**, yellow oil, (38%,17.1mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.76 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.52 (td, *J* = 7.9, 1.6 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.40 (td, *J* = 7.7, 0.9 Hz, 1H), 4.31 (t, *J* = 6.7 Hz, 2H), 1.78 (h, *J* = 7.2 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 186.74, 163.38, 134.41, 133.99, 133.52, 131.75, 130.67, 127.38, 68.44, 21.80, 10.42. HRMS (ESI) m/z: Calcd for $C_{11}H_{11}ClO_3$ $[M+H]^+$: 227.04695; Found: 227.04619.

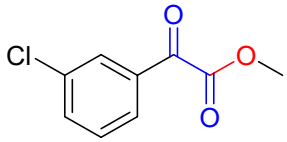
Butyl 2-(3-chlorophenyl)-2-oxoacetate（3da）



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3da**, yellow oil, (39%, 18.7 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (s, 1H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.64–7.61 (m, 1H), 7.46 (t, *J* = 7.9 Hz, 1H), 4.40 (t, *J* = 6.7 Hz, 2H), 1.76 (dt, *J* = 14.6, 6.8 Hz, 2H), 1.48 – 1.43 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR

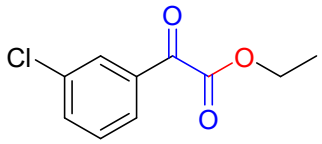
(151 MHz, Chloroform-*d*) δ 185.05, 163.30, 135.38, 134.90, 134.23, 130.34, 129.99, 128.32, 66.52, 30.55, 19.15, 13.74. Known compound, the spectroscopic data are consistent with previous report.[6]

Methyl 2-(3-chlorophenyl)-2-oxoacetate (3db)



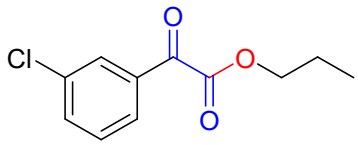
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3db**, white solid, mp: 43.5 – 44.8 °C, (72%, 28.5 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (s, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.64–7.58 (m, 1H), 7.45 (t, *J* = 7.9 Hz, 1H), 3.98 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 186.74, 163.38, 134.41, 133.99, 133.52, 131.75, 130.67, 127.38, 68.44, 21.80, 10.42. Known compound, the spectroscopic data are consistent with previous report.[4]

Ethyl 2-(3-chlorophenyl)-2-oxoacetate (3dc)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3dc**, black oil, (49%, 20.7 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.01 (s, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.65–7.58 (m, 1H), 7.46 (t, *J* = 7.9 Hz, 1H), 4.45 (d, *J* = 7.1 Hz, 2H), 1.42 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 184.97, 163.13, 135.37, 134.91, 134.20, 130.34, 129.99, 128.36, 62.77, 14.20. Known compound, the spectroscopic data are consistent with previous report.[5]

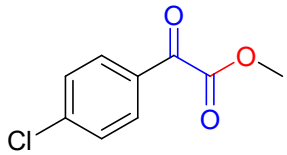
Propyl 2-(3-chlorophenyl)-2-oxoacetate (3dd)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3dd**,

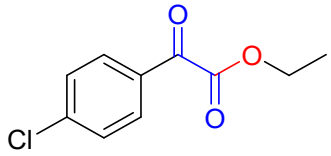
yellow oil, (54%, 24.4 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (s, 1H), 7.90 (d, *J* = 7.8 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 7.9 Hz, 1H), 4.35 (t, *J* = 6.7 Hz, 2H), 1.81 (h, *J* = 7.2 Hz, 2H), 1.01 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 185.04, 163.30, 135.37, 134.90, 134.22, 130.34, 129.96, 128.32, 68.17, 21.98, 10.40. HRMS (ESI) m/z: Calcd for $C_{11}H_{11}ClO_3$ $[M+H]^+$: 227.04695; Found: 227.04617.

Methyl 2-(4-chlorophenyl)-2-oxoacetate (3eb)



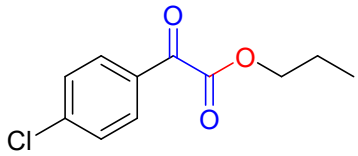
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3eb**, yellow oil, mp: 55.4 – 56.3 °C, (57%, 22.5 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.6 Hz, 1H), 7.49 (d, *J* = 8.6 Hz, 1H), 3.98 (s, 2H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 184.60, 163.54, 141.86, 131.62, 130.99, 129.44, 53.07. Known compound, the spectroscopic data are consistent with previous report.[4]

Ethyl 2-(4-chlorophenyl)-2-oxoacetate (3ec)



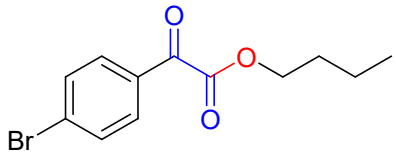
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ec**, yellow oil, (74%, 31.3 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.5 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 4.43 (d, *J* = 7.2 Hz, 2H), 1.40 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 184.96, 163.29, 141.68, 131.52, 131.02, 129.38, 62.62, 14.16. Known compound, the spectroscopic data are consistent with previous report.[1]

Propyl 2-(4-chlorophenyl)-2-oxoacetate (3ed)



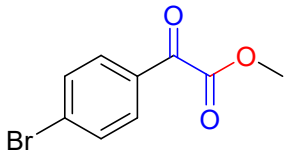
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ed**, yellow oil, (69%, 31.1 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.6 Hz, 2H), 7.47 (d, *J* = 8.6 Hz, 2H), 4.33 (t, *J* = 6.7 Hz, 2H), 1.79 (h, *J* = 7.2 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 185.12, 163.54, 141.77, 131.55, 131.10, 129.47, 68.11, 22.01, 10.43. HRMS (ESI) m/z: Calcd for $C_{11}H_{11}ClO_3$ $[M+H]^+$: 227.04695; Found: 227.04634.

Butyl 2-(4-bromophenyl)-2-oxoacetate (3fa)



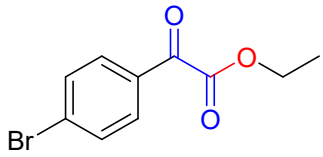
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3fa**, yellow oil, (45%, 25.5 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 8.6 Hz, 2H), 7.65 (d, *J* = 8.6 Hz, 2H), 4.38 (t, *J* = 6.7 Hz, 2H), 1.75 (dt, *J* = 14.5, 6.8 Hz, 2H), 1.44 (h, *J* = 7.4 Hz, 2H), 0.96 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 185.28, 163.45, 132.41, 131.53, 131.46, 130.61, 66.42, 30.54, 19.13, 13.73. Known compound, the spectroscopic data are consistent with previous report.[7]

Methyl 2-(4-bromophenyl)-2-oxoacetate (3fb)



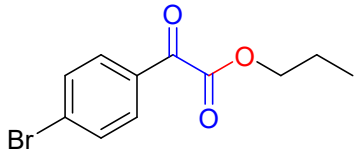
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3fb**, pale yellow solid, mp: 53.6–54.8 °C, (40%, 19.2 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.57 (d, *J* = 8.5 Hz, 2H), 3.90 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.47, 131.83, 131.23, 129.17, 128.16, 52.41. Known compound, the spectroscopic data are consistent with previous report.[8]

Ethyl 2-(4-bromophenyl)-2-oxoacetate (3fc)



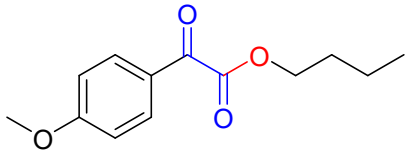
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3fc**, yellow oil, (53%, 27.0 mg), ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.65 (d, *J* = 8.5 Hz, 2H), 4.44 (d, *J* = 7.2 Hz, 2H), 1.41 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 184.97, 163.29, 141.68, 131.52, 131.02, 129.38, 62.62, 14.16. Known compound, the spectroscopic data are consistent with previous report.[1]

Propyl 2-(4-bromophenyl)-2-oxoacetate (3fd)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3fd**, yellow oil, (38%, 20.4 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.92–7.86 (m, 1H), 7.66 (d, *J* = 8.6 Hz, 1H), 4.34 (t, *J* = 6.7 Hz, 1H), 1.80 (h, *J* = 7.2 Hz, 1H), 1.01 (t, *J* = 7.4 Hz, 2H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 185.32, 163.48, 132.44, 131.55, 131.48, 130.65, 68.11, 21.99, 10.42. HRMS (ESI) Calcd. for $C_{11}H_{11}BrO_3$ $[M+H]^+$: 270.99643. Found: 270.99554.

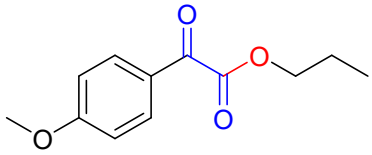
Butyl 2-(4-methoxyphenyl)-2-oxoacetate (3ga)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ga**, yellow oil, (51%, 24.0 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.29 (t, *J* = 6.6 Hz, 2H), 3.86 (s, 3H), 1.82–1.67 (m, 2H), 1.47 (h, *J* = 7.4 Hz, 2H), 0.98 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.61, 163.38, 131.67, 123.14, 113.69, 64.67, 55.55, 30.98, 19.43, 13.92. Known

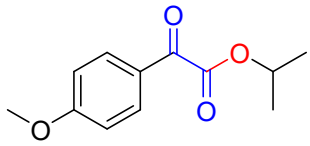
compound, the spectroscopic data are consistent with previous report.[9]

Propyl 2-(4-methoxyphenyl)-2-oxoacetate (3gd)



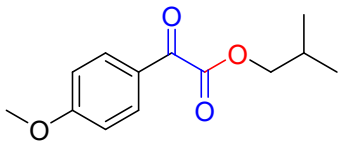
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gd**, yellow oil, (59%, 26.1 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.6 Hz, 2H), 6.91 (d, *J* = 8.6 Hz, 2H), 4.24 (t, *J* = 6.6 Hz, 2H), 3.85 (s, 3H), 1.77 (q, *J* = 7.1 Hz, 2H), 1.02 (t, *J* = 7.4 Hz, 3H).^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.56, 163.37, 131.65, 123.10, 113.66, 66.34, 55.51, 22.28, 10.64. Known compound, the spectroscopic data are consistent with previous report.[9]

Isopropyl 2-(4-methoxyphenyl)-2-oxoacetate (3gf)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gf**, yellow oil, (50%, 22.2 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.9 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 5.22 (p, *J* = 6.3 Hz, 1H), 3.85 (s, 3H), 1.35 (d, *J* = 6.3 Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.02, 163.31, 131.63, 123.52, 113.62, 68.06, 55.54, 22.13. Known compound, the spectroscopic data are consistent with previous report.[10]

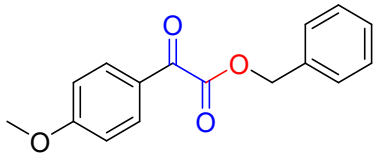
Isobutyl 2-(4-methoxyphenyl)-2-oxoacetate (3gg)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gg**, yellow oil, (53%, 25.0 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.8 Hz,

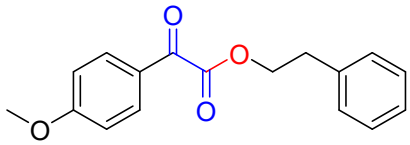
2H), 6.91 (d, J = 8.8 Hz, 2H), 4.07 (d, J = 6.6 Hz, 2H), 3.84 (s, 3H), 2.06 (dp, J = 13.4, 6.7 Hz, 1H), 1.01 (d, J = 6.8 Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.47, 163.37, 131.62, 123.07, 113.66, 70.80, 55.48, 28.03, 19.30. HRMS (ESI) Calcd. for $C_{13}H_{16}O_4$ $[M+H]^+$: 237.11214. Found: 237.11124.

Benzyl 2-(4-methoxyphenyl)-2-oxoacetate (3gi)



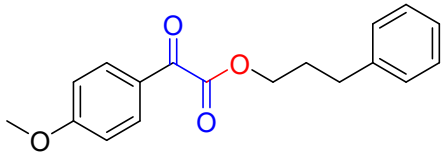
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gi**, yellow oil, (46%, 24.8 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.04 (d, J = 8.9 Hz, 2H), 7.45 (d, J = 7.3 Hz, 2H), 7.39 (t, J = 7.4 Hz, 2H), 7.34 (t, J = 7.3 Hz, 1H), 6.92 (d, J = 8.9 Hz, 2H), 5.35 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.31, 163.57, 136.44, 131.87, 128.69, 128.27, 128.23, 122.68, 113.75, 66.51, 55.54. Known compound, the spectroscopic data are consistent with previous report.[10]

Phenethyl 2-(4-methoxyphenyl)-2-oxoacetate (3gj)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gj**, light grey oil, (57%, 32.5 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 (d, J = 8.8 Hz, 1H), 7.35–7.23 (m, 3H), 6.92 (d, J = 8.8 Hz, 1H), 4.51 (t, J = 7.0 Hz, 1H), 3.86 (s, 2H), 3.08 (t, J = 7.0 Hz, 1H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.39, 163.46, 138.18, 131.71, 129.10, 128.64, 126.66, 122.85, 113.72, 65.31, 55.53, 35.43. Known compound, the spectroscopic data are consistent with previous report.[11]

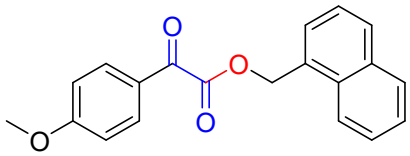
3-Phenylpropyl 2-(4-methoxyphenyl)-2-oxoacetate (3gk)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gk**, yellow oil, (55%, 32.7 mg) ;^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.01 (d, J = 8.8 Hz,

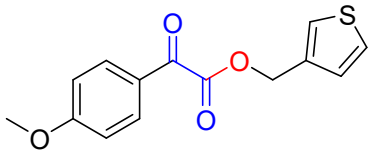
2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.22 (dd, *J* = 15.5, 7.4 Hz, 3H), 6.96–6.91 (m, 2H), 4.33 (t, *J* = 6.5 Hz, 2H), 3.87 (s, 3H), 2.84–2.74 (m, 2H), 2.11 (dt, *J* = 13.8, 6.6 Hz, 2H).^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.46, 163.43, 141.39, 131.68, 128.57, 128.56, 126.11, 122.94, 113.70, 64.08, 55.53, 32.45, 30.48. HRMS (ESI) Calcd. for $C_{18}H_{18}O_4$ $[M+H]^+$: 299.1277. Found: 299.1301.

Naphthalen-1-ylmethyl 2-(4-methoxyphenyl)-2-oxoacetate (3gp)



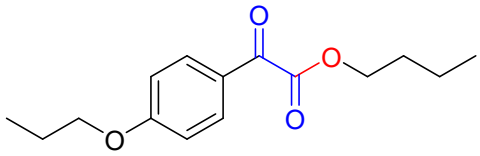
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gp**. yellow oil, (33%, 21.1 mg). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.02 (d, *J* = 8.8 Hz, 2H), 7.36 (d, *J* = 2.5 Hz, 1H), 7.33 (d, *J* = 4.9 Hz, 1H), 7.17 (d, *J* = 4.9 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.34 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.38, 163.59, 133.90, 131.93, 129.39, 128.86, 127.53, 126.72, 126.07, 125.50, 125.46, 123.83, 122.66, 113.76, 64.99, 55.55. HRMS (ESI) Calcd. for $C_{20}H_{16}O_4$ $[M+H]^+$: 321.1121. Found: 321.10928.

Thiophen-3-ylmethyl 2-(4-methoxyphenyl)-2-oxoacetate (3gq)



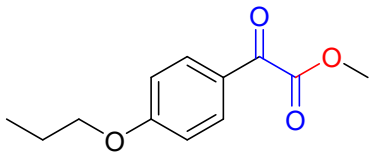
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3gq**. yellow oil, (20%, 11.1 mg). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.02 (d, *J* = 8.8 Hz, 2H), 7.36 (d, *J* = 2.5 Hz, 1H), 7.33 (dd, *J* = 4.9, 3.0 Hz, 1H), 7.17 (d, *J* = 4.9 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 5.34 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 166.28, 163.59, 137.30, 131.88, 127.75, 126.33, 124.24, 122.67, 113.77, 61.72, 55.58. HRMS (ESI) Calcd. for $C_{14}H_{12}O_4S$ $[M+Na]^+$: 299,0348. Found: 299.0347.

Butyl 2-oxo-2-(4-propoxyphenyl)acetate (3ha)



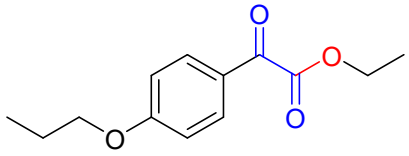
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ha**, yellow oil, (51%, 26.9 mg); ^{1}H NMR (400 MHz, DMSO- d_6) δ 7.91-7.87 (m, 2H), 7.04-7.00 (m, 2H), 4.22 (t, J = 6.5 Hz, 2H), 3.99 (t, J = 6.5 Hz, 2H),1.73-1.63 (m, 4H), 1.45-1.36 (m, 2H), 0.95 (dt, J = 20.5, 7.4 Hz, 6H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.41, 162.54, 131.13, 121.92, 114.36, 69.26, 63.93, 30.30, 21.88, 18.75, 13.57, 10.27. HRMS (ESI) Calcd. for $C_{15}H_{20}O_4$ [M+Na]$^+$: 287.12538. Found: 287.12469.

Methyl 2-oxo-2-(4-propoxyphenyl)acetate (3hb)



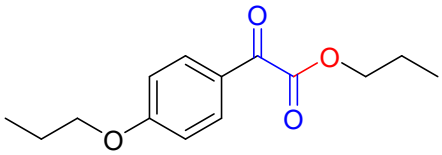
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hb**, yellow oil, (62%, 27.5 mg); ^{1}H NMR (400 MHz, DMSO- d_6) δ 7.91-7.88 (m, 2H), 7.05-6.99 (m, 2H), 4.00 (t, J = 6.5 Hz, 2H) 3.81 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.89, 162.58, 131.21, 121.65, 114.39, 69.28, 51.76, 21.88, 10.27. HRMS (ESI) Calcd. for $C_{12}H_{14}O_4$ [M+Na]$^+$: 245.07843. Found: 245.07793.

Ethyl 2-oxo-2-(4-propoxyphenyl)acetate (3hc)



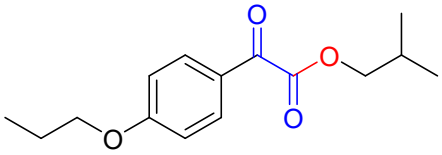
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hc**, yellow oil, (70%, 33 mg); ^{1}H NMR (400 MHz, DMSO- d_6) δ 7.91-7.87 (m, 2H), 7.05-6.95 (m, 2H), 4.27 (q, J = 7.1 Hz, 2H) 3.99 (t, J = 6.5 Hz, 2H), 1.83-1.66 (m, 2H),1.30 (td, J = 7.1, 2.8 Hz, 3H), 0.98 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.38, 162.52, 131.14, 121.93, 114.32, 69.26, 60.24, 21.88, 14.19, 10.26. HRMS (ESI) Calcd. for $C_{13}H_{16}O_4$ [M+Na]$^+$: 259.09408. Found: 259.09406.

Propyl 2-oxo-2-(4-propoxyphenyl)acetate (3hd)



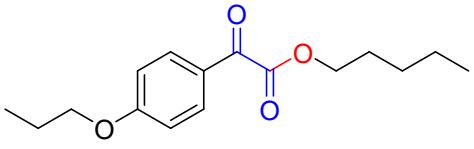
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hd**, yellow oil, (59%, 29.5 mg); ^{1}H NMR (400 MHz, DMSO- d_6) δ 7.92-7.88 (m, 2H), 7.04-7.01 (m, 2H), 4.18 (t, J = 6.6 Hz, 2H),4.00 (t, J = 6.5 Hz, 2H),1.74-1.66 (m, 4H), 0.97 (q, J = 7.6 Hz, 6H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.43, 162.54, 131.15, 121.91, 114.38, 69.27, 65.68, 21.88, 21.66, 10.34, 10.28. HRMS (ESI) m/z: Calcd for $C_{14}H_{18}O_4$ $[M+Na]^+$: 273.10973; Found: 273.10916.

Isobutyl 2-oxo-2-(4-propoxyphenyl)acetate (3hg)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hg**, yellow oil, (55%, 29.0 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.93-7.89 (m, 2H), 7.05-7.03 (m, 2H), 4.03-3.99 (m, 2H), 2.01 (dp, J = 13.2, 6.6 Hz, 1H) ,1.76-1.70 (m, 2H), 1.00-0.96 (m, 9H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.38, 162.57, 131.15, 121.88, 114.43, 70.01, 69.28, 27.43, 21.88, 18.94, 10.29. HRMS (ESI) Calcd. for $C_{15}H_{20}O_4$ $[M+Na]^+$: 287.12538. Found: 287.12473.

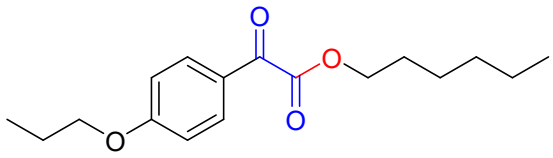
Pentyl 2-oxo-2-(4-propoxyphenyl)acetate (3hl)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hl**, yellow oil, (68%, 37.8 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.96–7.83 (m, 1H), 7.01 (dd, J = 9.0, 2.1 Hz, 1H), 4.21 (t, J = 6.6 Hz, 1H), 3.98 (t, J = 6.5 Hz, 1H), 1.71 (ddt, J = 27.3, 13.7, 6.9 Hz, 2H), 1.34 (h, J = 7.0, 6.5 Hz, 2H), 0.97 (t, J = 7.4 Hz, 1H), 0.88 (t, J = 7.1 Hz, 1H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.38, 162.53, 131.11, 121.92,

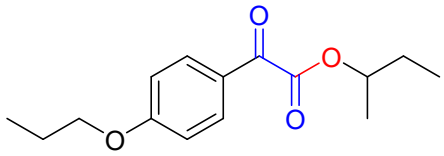
114.32, 69.25, 64.19, 27.93, 27.69, 21.88, 21.79, 13.80, 10.24. HRMS (ESI) m/z: Calcd for $C_{16}H_{22}O_4$ $[M+Na]^+$: 301.14103; Found: 301.14044.

Hexyl 2-oxo-2-(4-propoxyphenyl)acetate (3hm)



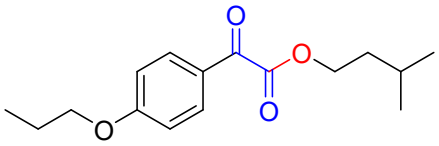
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hm**, yellow oil, (63%, 36.7 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 (d, J = 8.9 Hz, 1H), 7.01 (dd, J = 8.8, 1.4 Hz, 1H), 4.20 (t, J = 6.6 Hz, 1H), 3.98 (t, J = 6.5 Hz, 1H), 1.73 (q, J = 6.9 Hz, 1H), 1.70–1.62 (m, 1H), 1.41–1.32 (m, 1H), 1.29 (dd, J = 4.6, 2.5 Hz, 2H), 0.97 (t, J = 7.4 Hz, 1H), 0.88–0.83 (m, 1H).^{13}C NMR (151 MHz, DMSO-d_6) δ 165.87, 163.01, 131.58, 122.39, 114.84, 114.80, 69.73, 64.68, 31.36, 28.66, 25.63, 22.46, 22.35, 14.29, 10.72. HRMS (ESI) Calcd. for $C_{17}H_{24}O_4$ $[M+H]^+$: 293.1747. Found: 293.1741.

Sec-butyl 2-oxo-2-(4-propoxyphenyl)acetate (3hn)



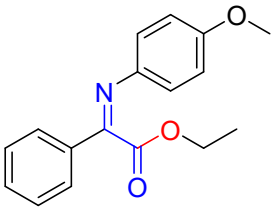
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3hn**, yellow oil, (64%, 33.7 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.91-7.87 (m, 2H), 7.05-7.02 (m, 2H), 4.95 (dd, J = 12.1, 5.9 Hz, 1H), 4.01 (t, J = 6.6 Hz, 2H), 1.76-1.63 (m, 4H), 1.27 (d, J = 6.3 Hz, 3H), 0.98 (t, J = 7.4 Hz, 3H), 0.91 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.86, 163.05, 131.63, 122.36, 114.91, 70.49, 27.91, 22.36, 19.42, 10.77. HRMS (ESI) Calcd. for $C_{15}H_{20}O_4$ $[M+Na]^+$: 287.12538. Found: 287.12476.

Isopentyl 2-oxo-2-(4-propoxyphenyl)acetate (3ho)



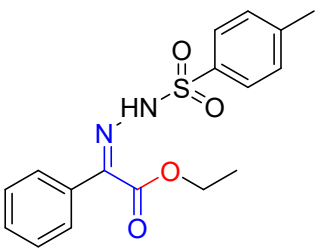
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **3ho**, yellow oil, (59%, 32.8 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.93–7.84 (m, 1H), 7.01 (dd, J = 9.1, 2.5 Hz, 1H), 4.25 (t, J = 6.6 Hz, 1H), 3.98 (t, J = 6.5 Hz, 1H), 1.74 (h, J = 7.1 Hz, 1H), 1.58 (q, J = 6.7 Hz, 1H), 1.05–0.80 (m, 4H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.37, 162.53, 131.11, 121.90, 114.37, 114.34, 69.25, 62.68, 36.95, 24.72, 22.29, 21.87, 10.25. HRMS (ESI) m/z: Calcd for $C_{16}H_{22}O_4$ $[M+Na]^+$: 301.14103; Found: 301.14053.

Ethyl (*Z*)-2-((4-methoxyphenyl)imino)-2-phenylacetate (5a)



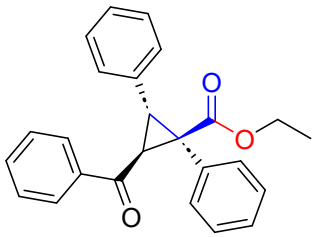
Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **5a**, yellow oil, (90%, 254.7 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.90–7.85 (m, 2H), 7.53–7.43 (m, 3H), 6.96 (d, J = 8.8 Hz, 2H), 6.87 (d, J = 8.8 Hz, 2H), 4.18 (q, J = 7.1 Hz, 2H), 3.81 (s, 3H), 1.08 (t, J = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 165.67, 159.80, 157.43, 143.45, 134.28, 131.68, 128.82, 127.99, 121.34, 114.23, 61.57, 55.61, 14.05. Known compound, the spectroscopic data are consistent with previous report.[12]

Ethyl (*Z*)-2-phenyl-2-(2-tosylhydrazineylidene)acetate (5b)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **5b**, yellow oil, (95%, 328.7 mg); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 11.54 (s, 1H), 7.87 (d, *J* = 8.2 Hz, 2H), 7.53–7.49 (m, 2H), 7.36 (dd, *J* = 11.1, 7.1 Hz, 3H), 7.33–7.30 (m, 2H), 4.35 (q, *J* = 7.1 Hz, 2H), 2.42 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 162.25, 144.53, 138.39, 135.60, 134.14, 129.86, 129.47, 128.67, 128.17, 128.10, 62.52, 21.74, 14.10. Known compound, the spectroscopic data are consistent with previous report.[13]

Ethyl 2-benzoyl-1,3-diphenylcyclopropane-1-carboxylate (5c)



Purification by fast column chromatography (eluent: PE/EA = 30/1, v/v) afforded **5c**, yellow oil, (97%, 215.3 mg). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.14–8.09 (m, 2H), 7.64–7.59 (m, 1H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.23 (ddd, *J* = 17.1, 7.1, 3.3 Hz, 5H), 7.13 (dtd, *J* = 9.2, 4.5, 1.9 Hz, 3H), 6.93 (dd, *J* = 7.2, 2.1 Hz, 2H), 4.15 (dq, *J* = 10.8, 7.1 Hz, 1H), 4.04 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.83 (d, *J* = 6.6 Hz, 1H), 3.76 (d, *J* = 6.6 Hz, 1H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 195.75, 169.98, 137.74, 135.19, 134.44, 133.41, 130.56, 128.88, 128.46, 128.24, 128.09, 127.89, 126.89, 61.71, 48.42, 36.94, 36.63, 14.04. Known compound, the spectroscopic data are consistent with previous report.[14]

9. References

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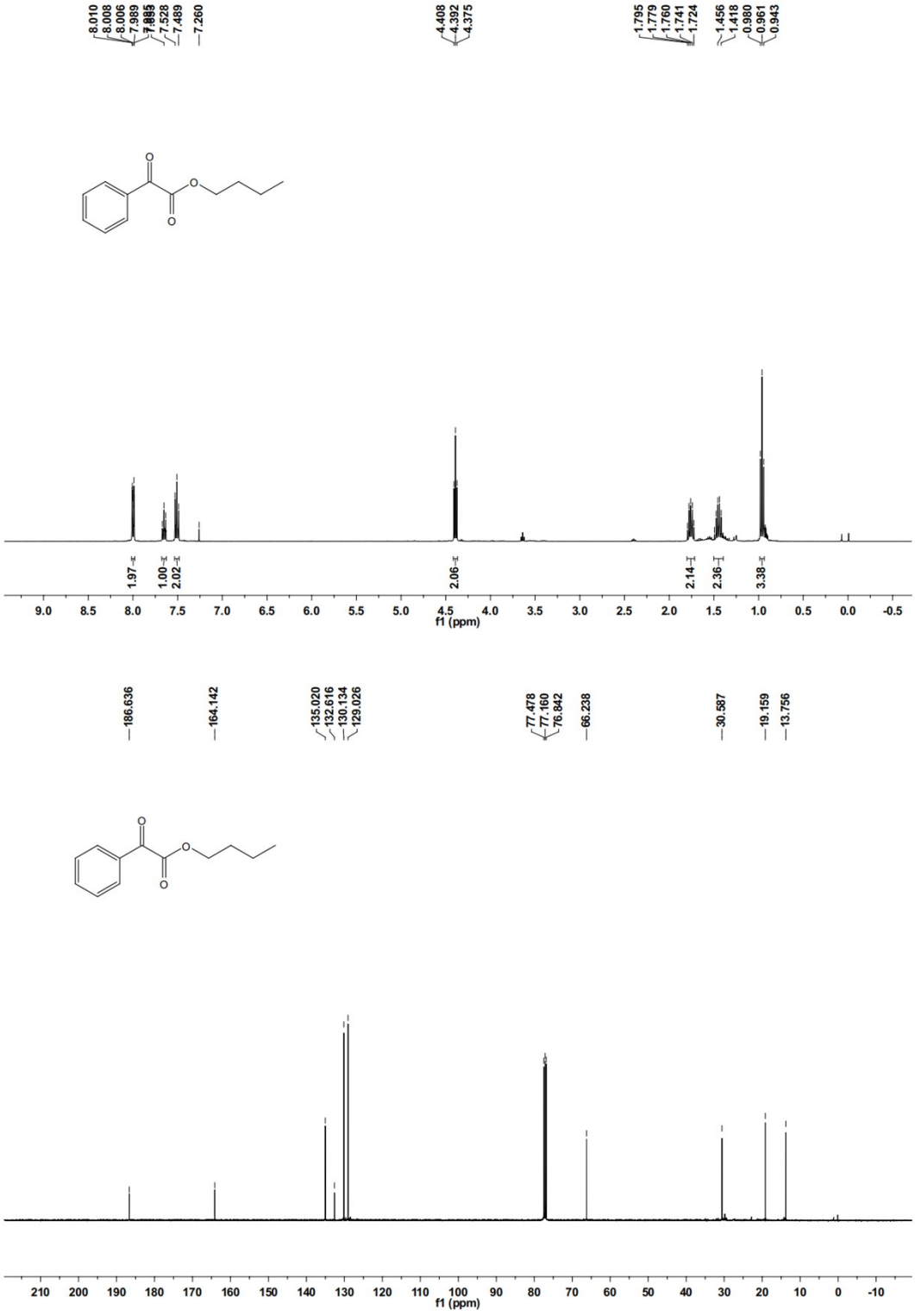
10. ^{1}H and ^{13}C NMR spectra of all products

^{1}H and ^{13}C NMR spectra of **3aa**



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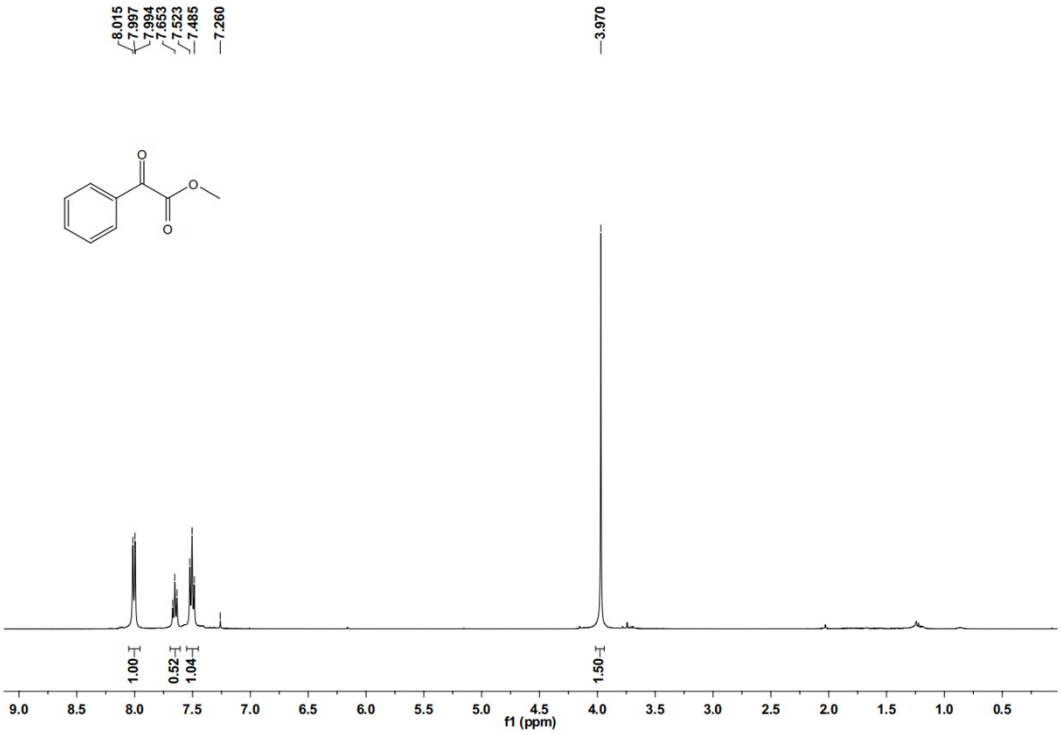

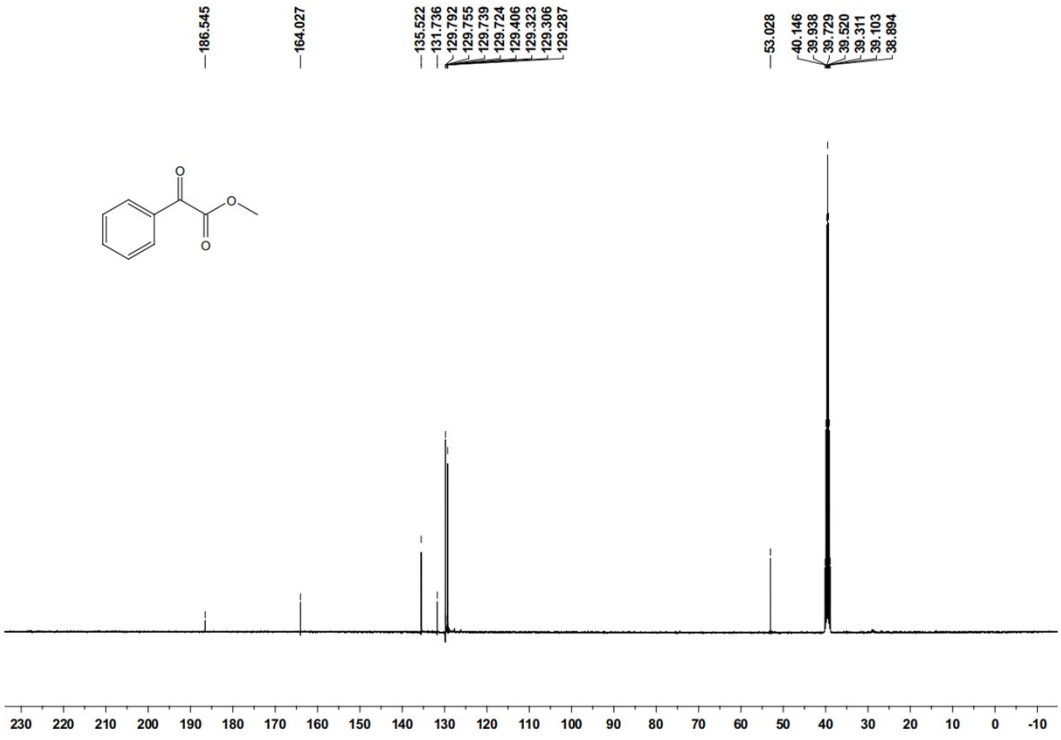

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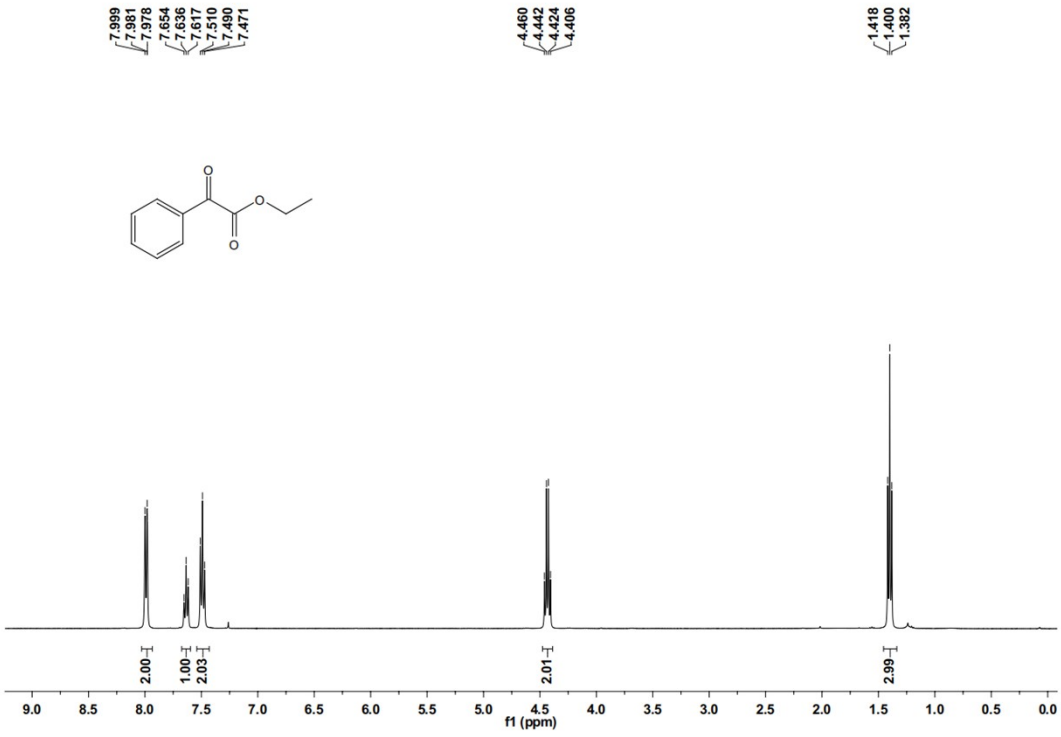

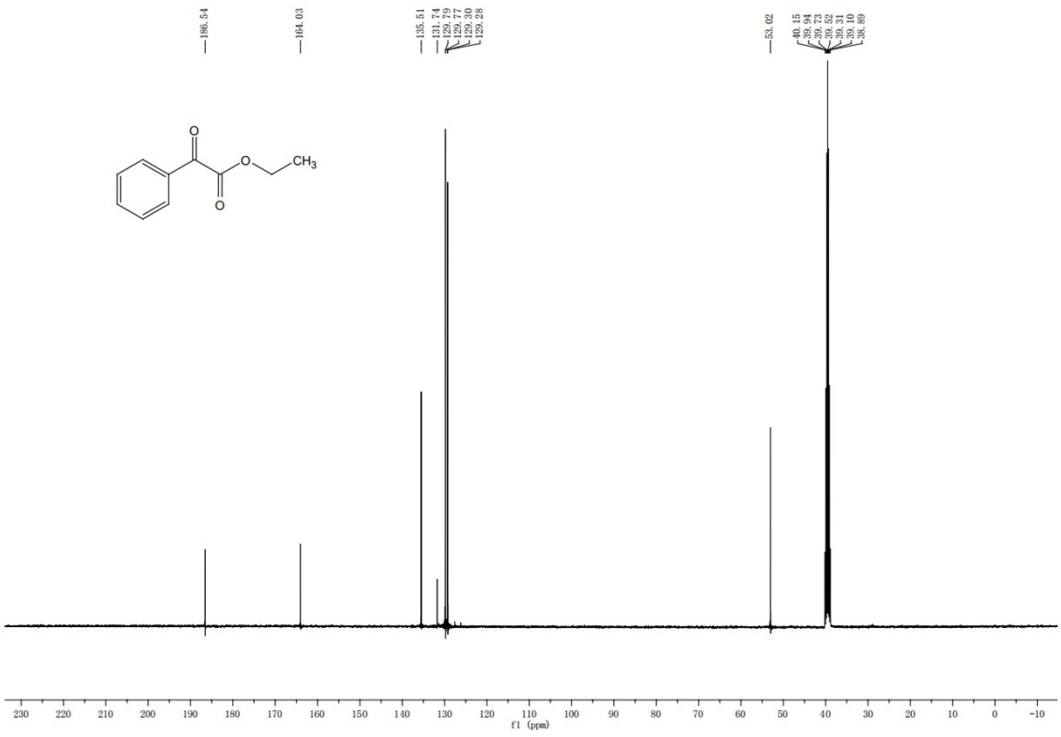

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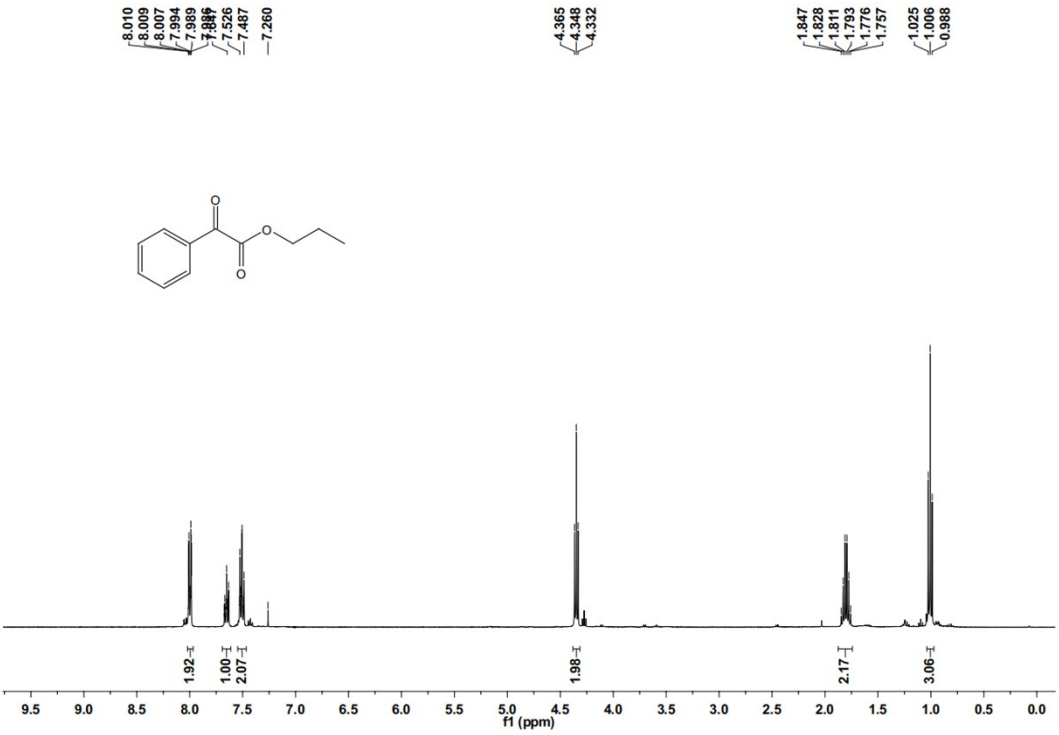

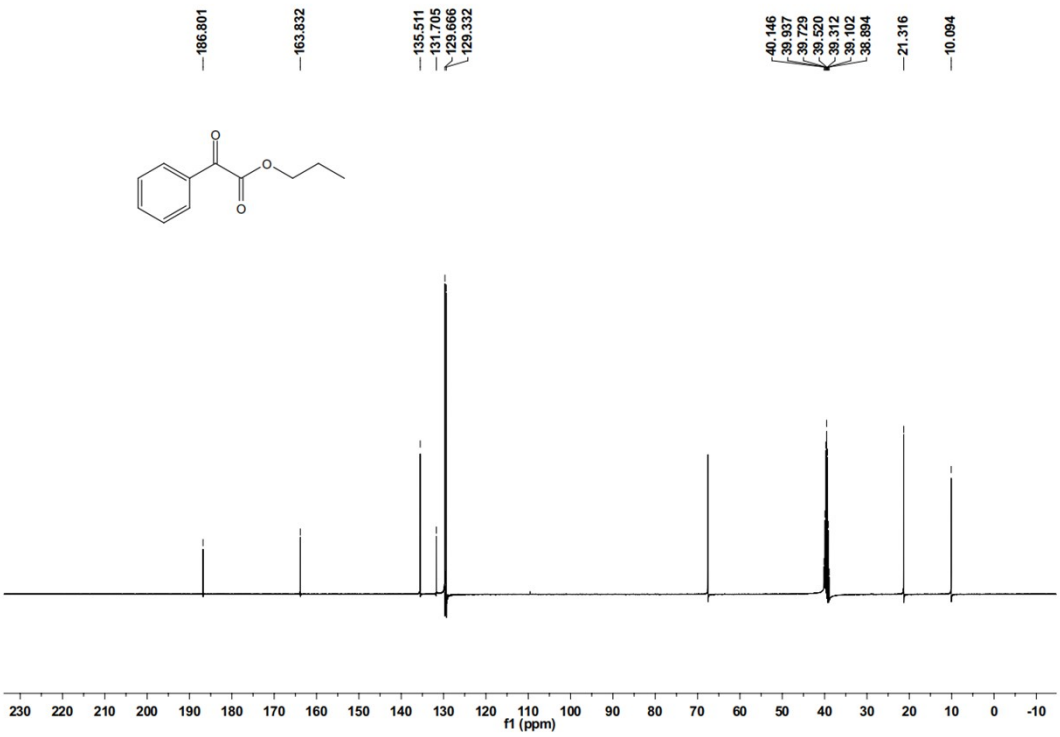

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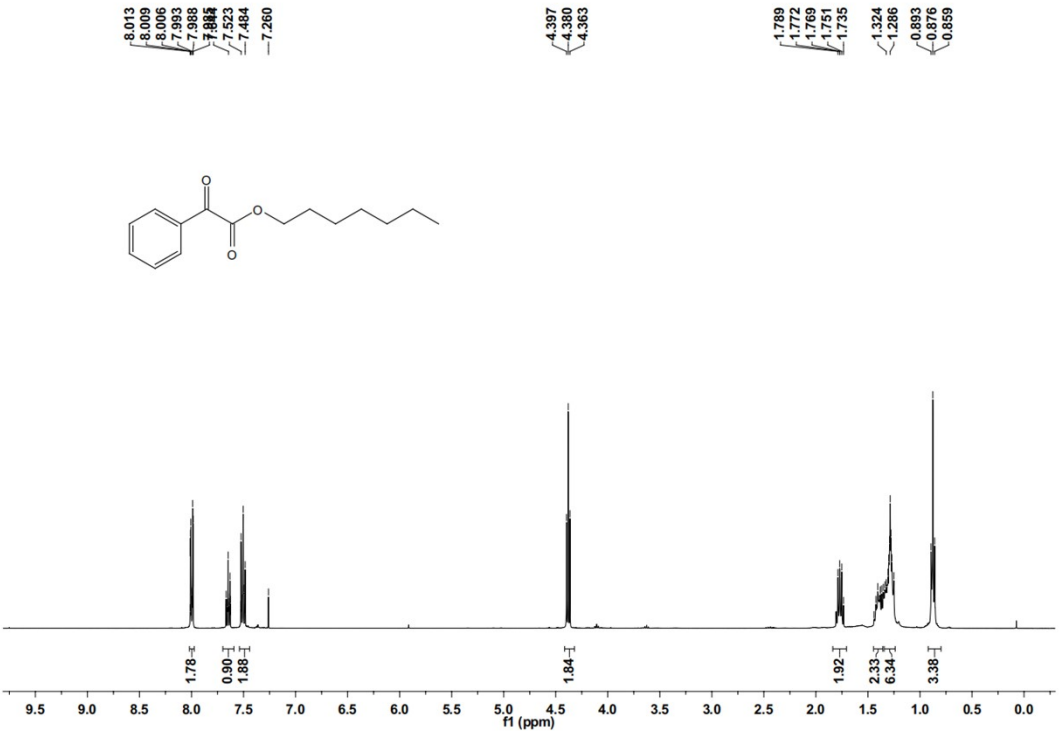

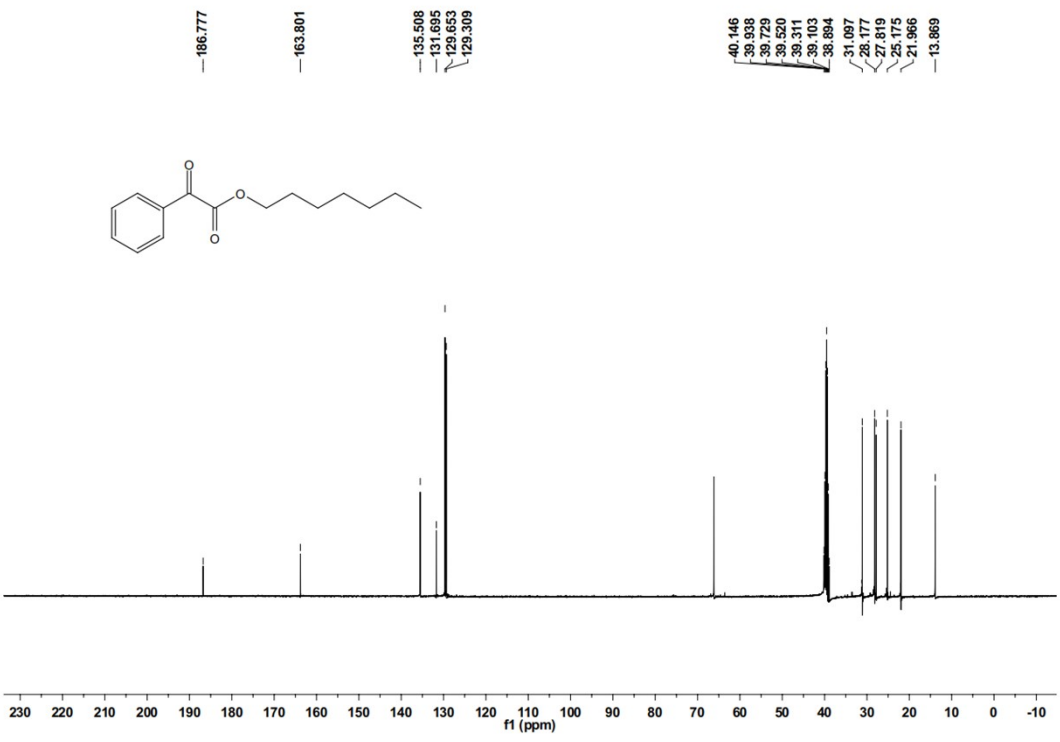

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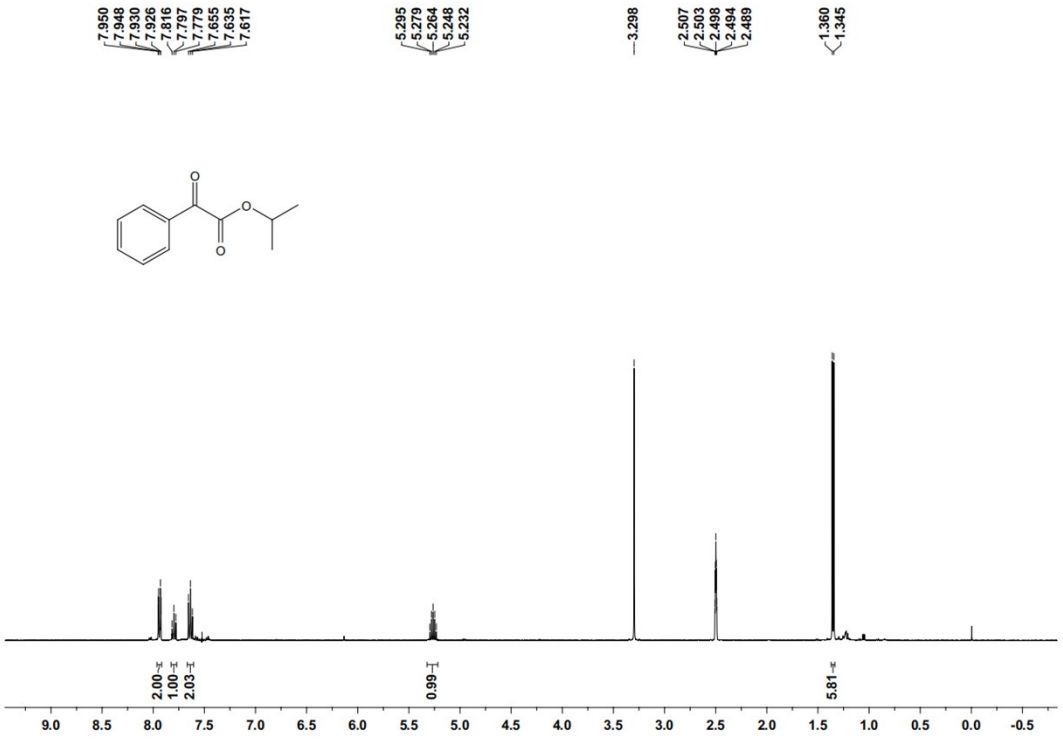

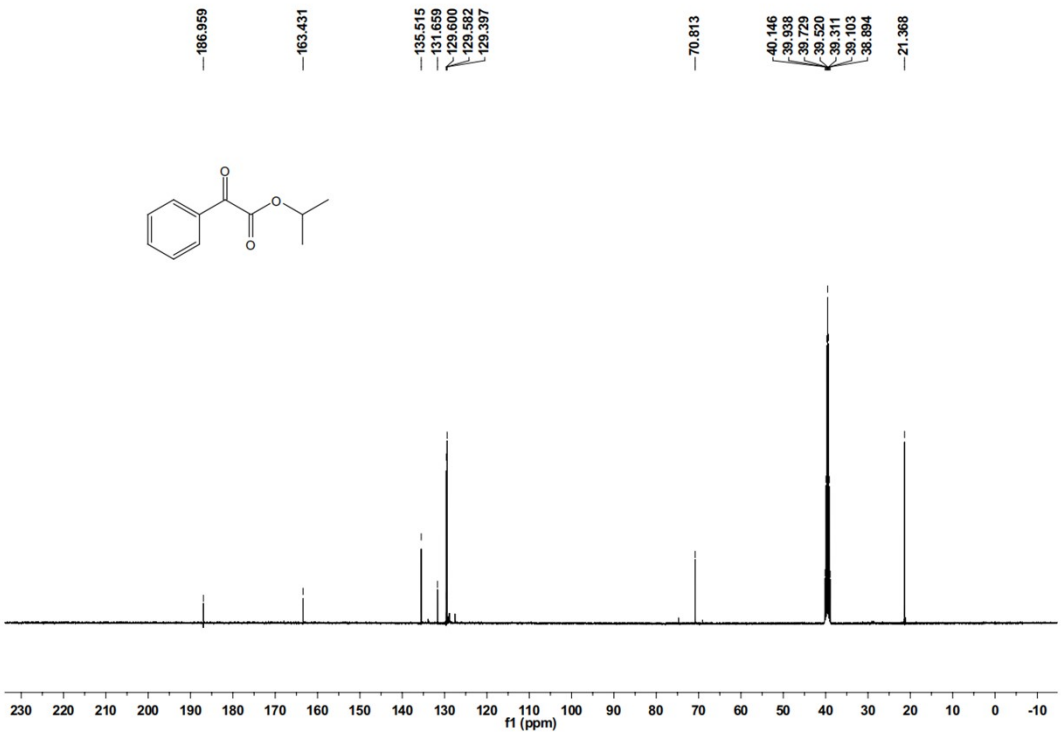

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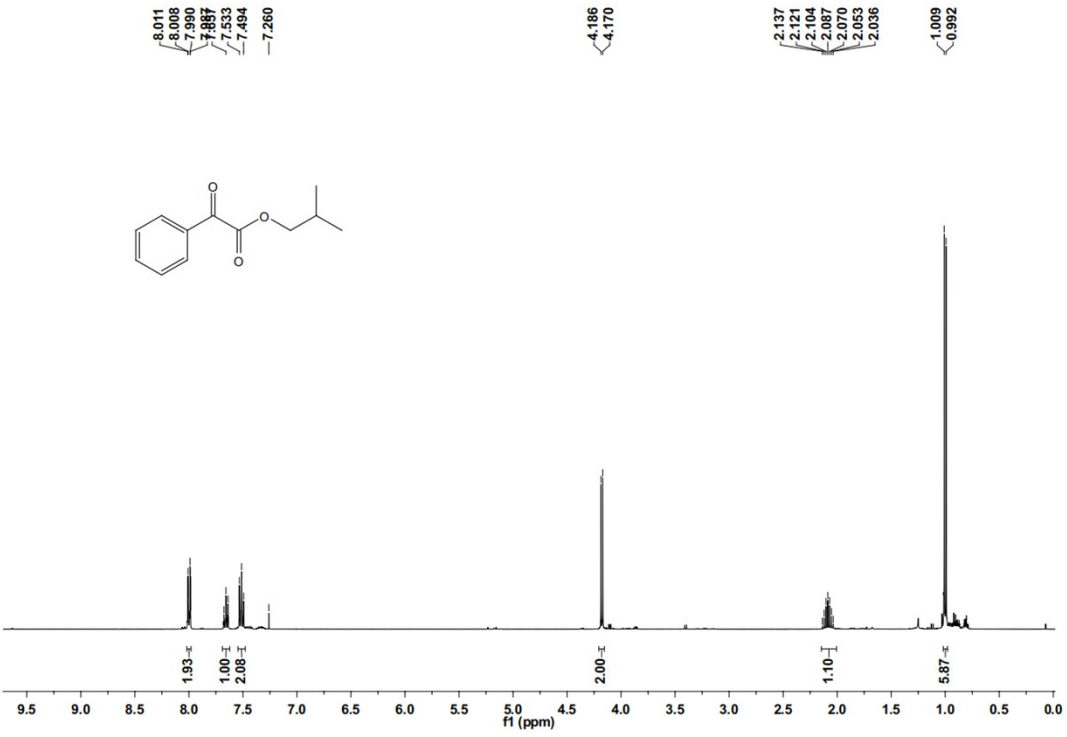
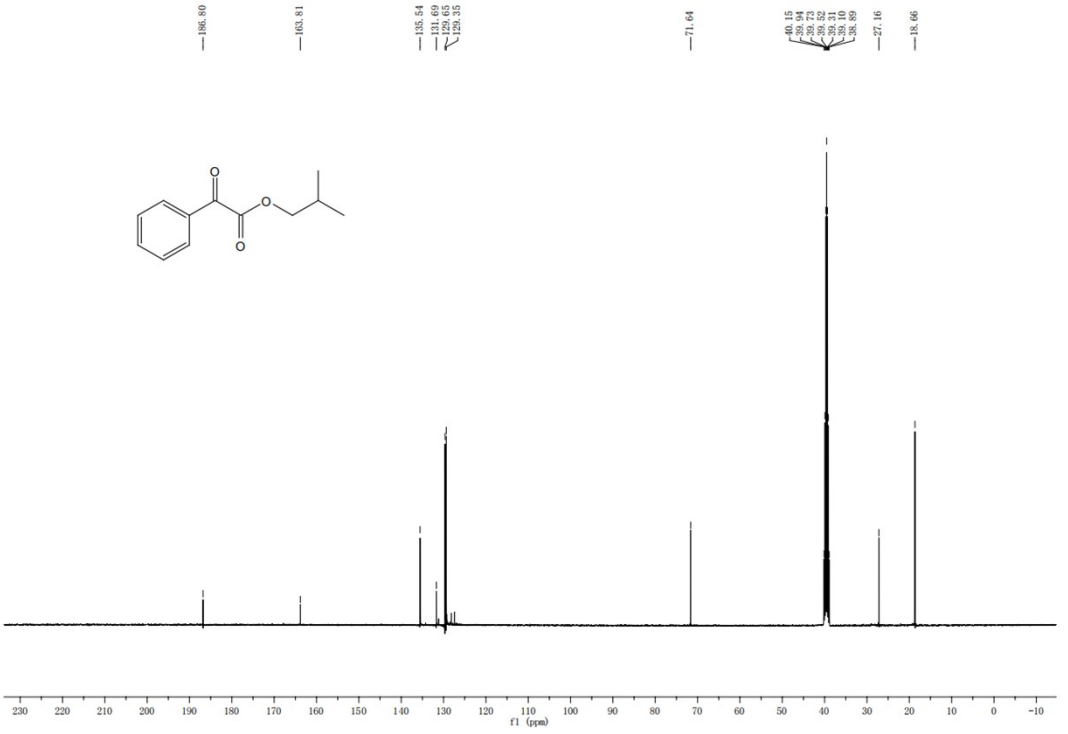
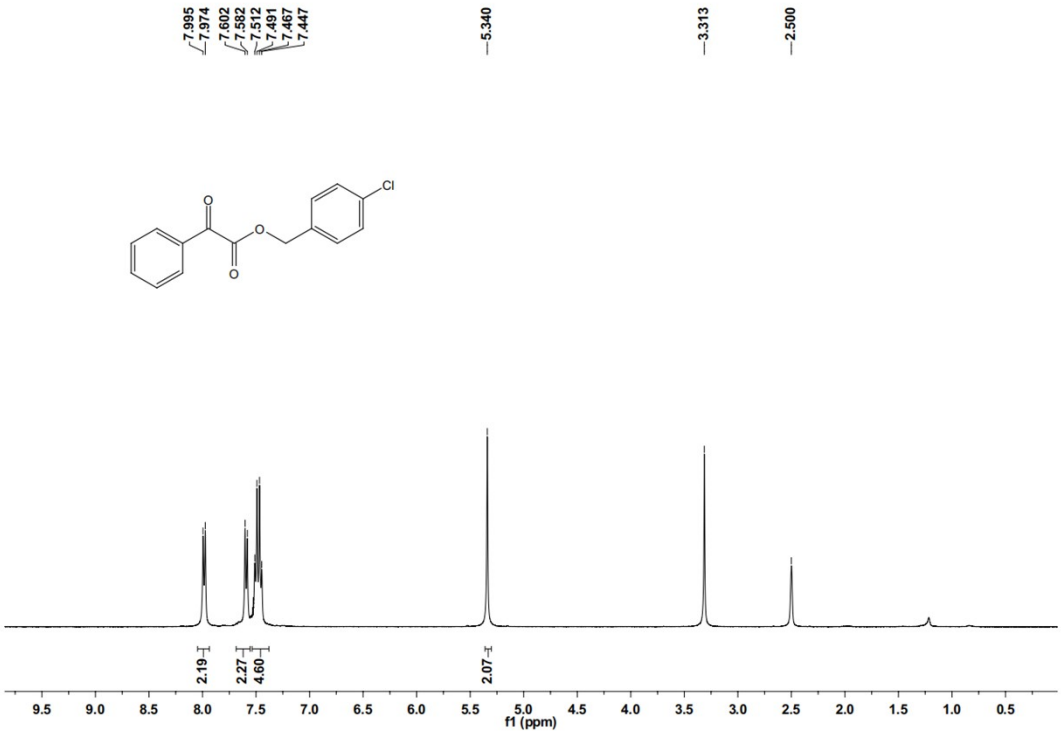
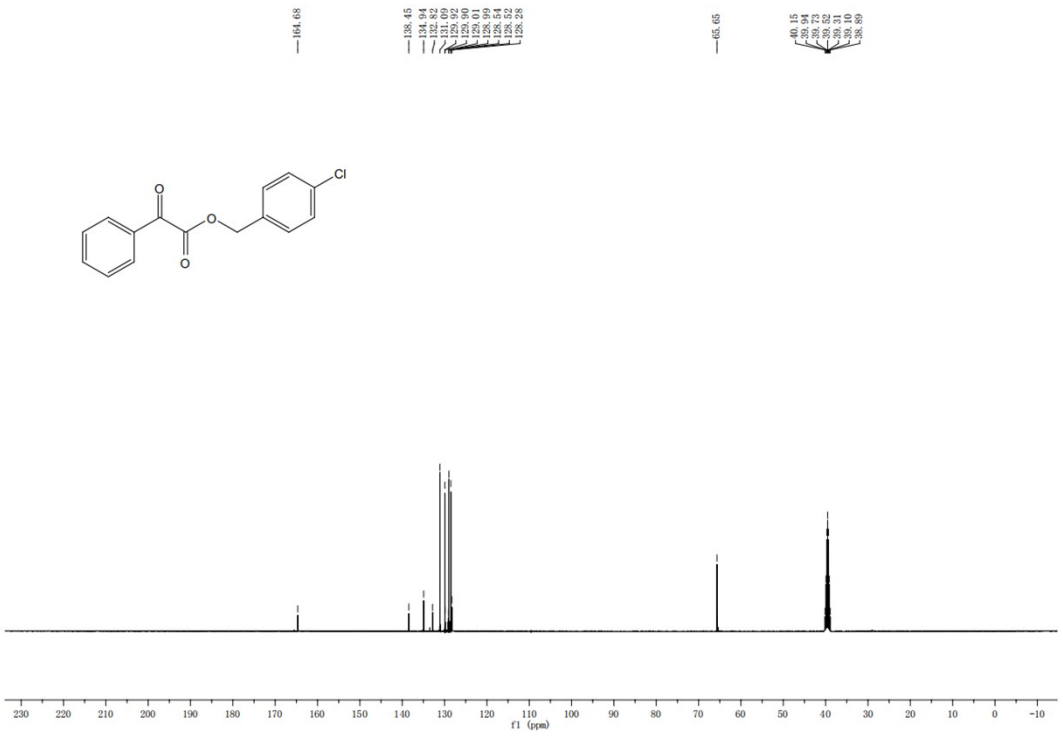




^{1}H and ^{13}C NMR spectra of **3ah**





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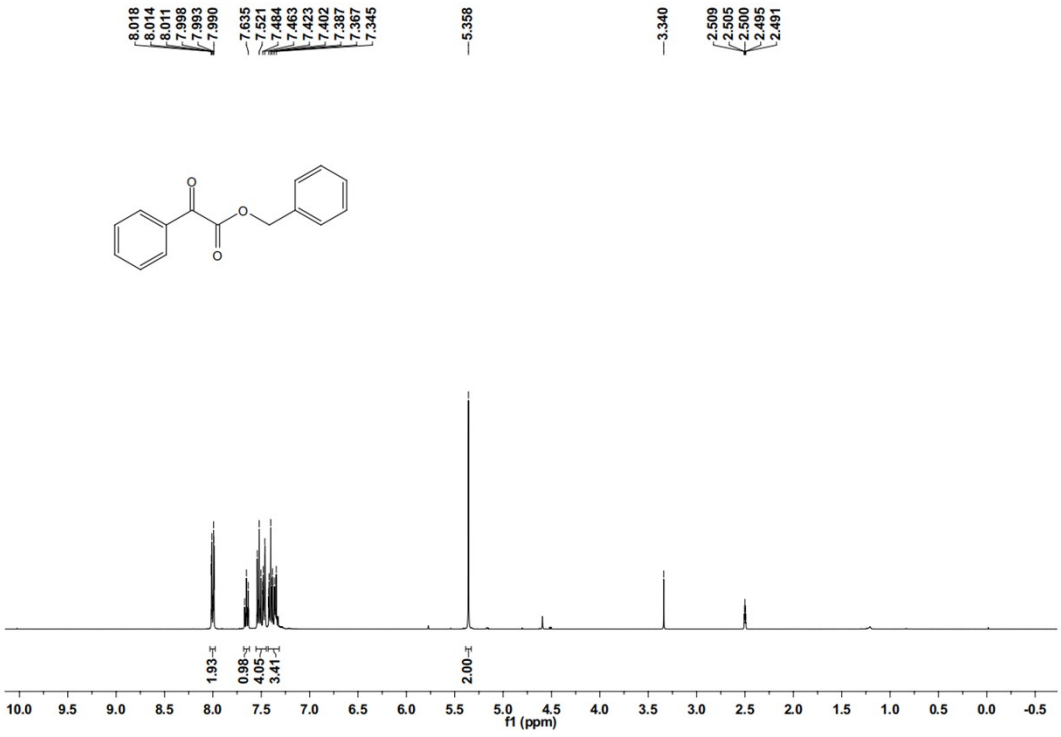
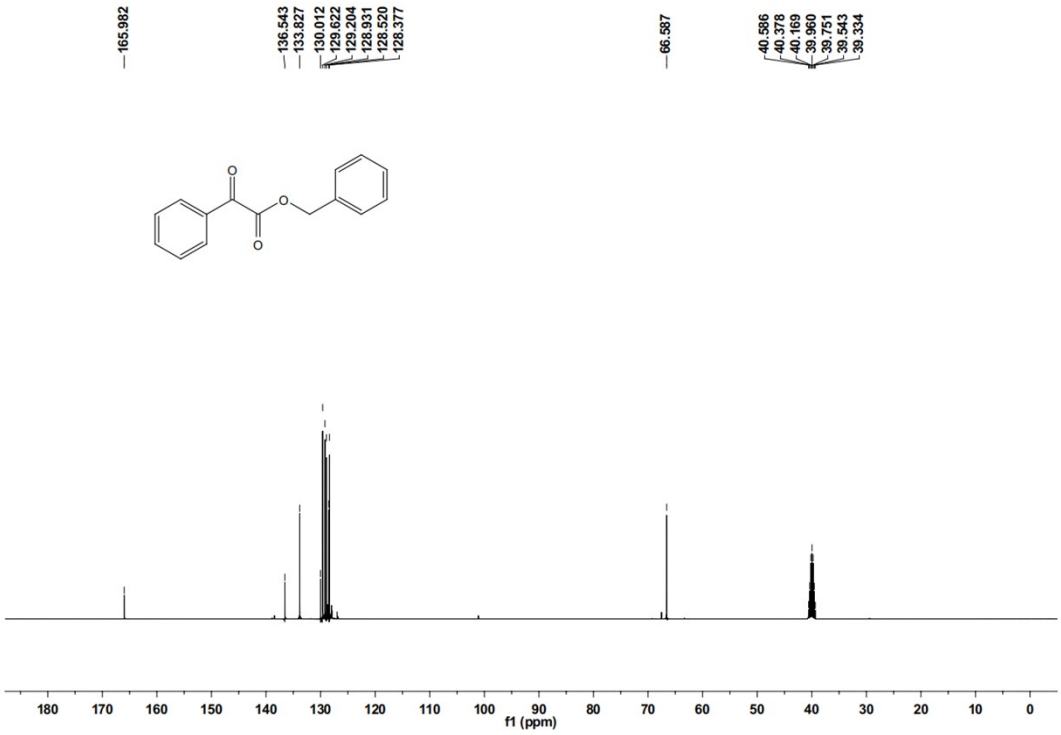
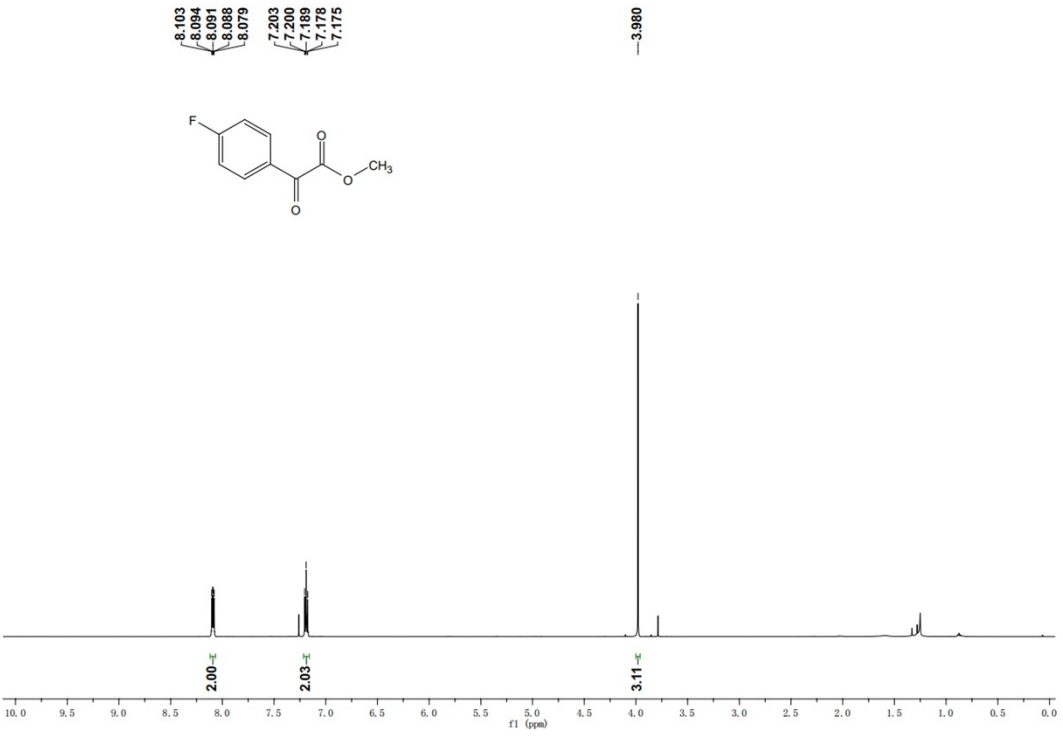
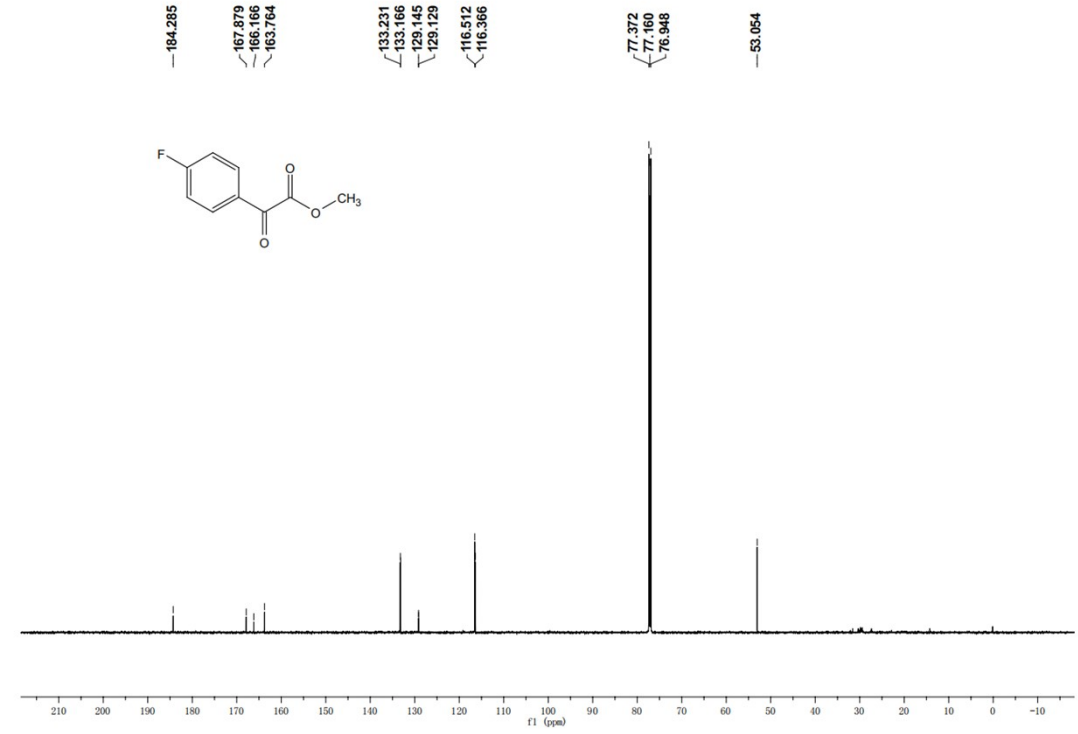


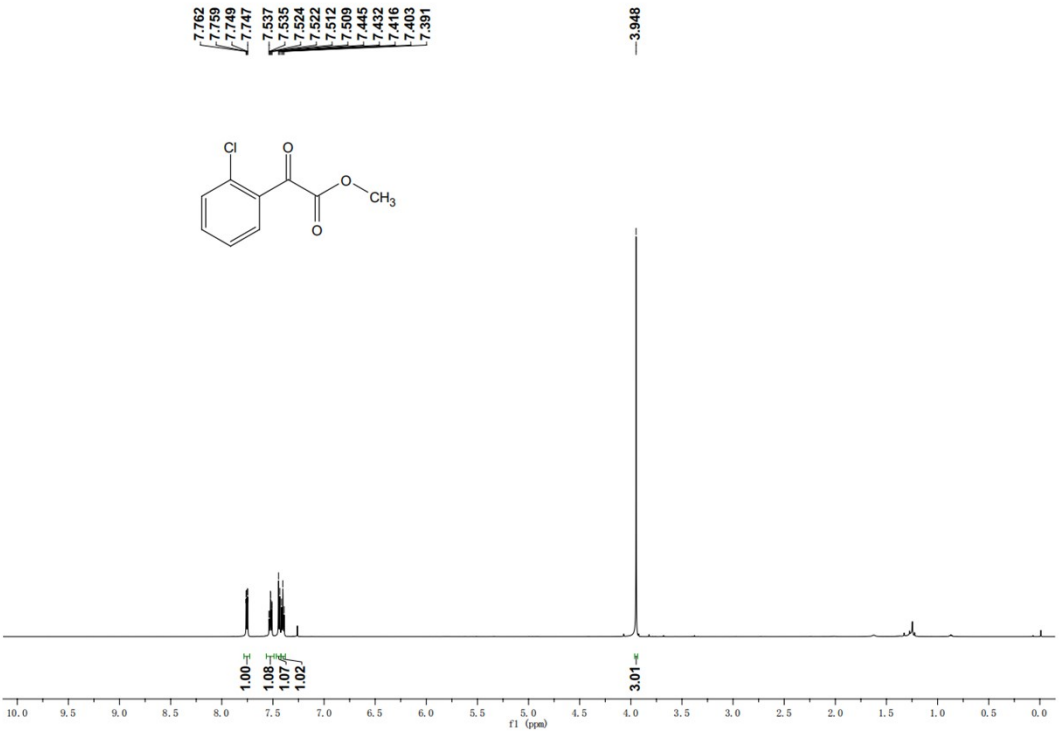


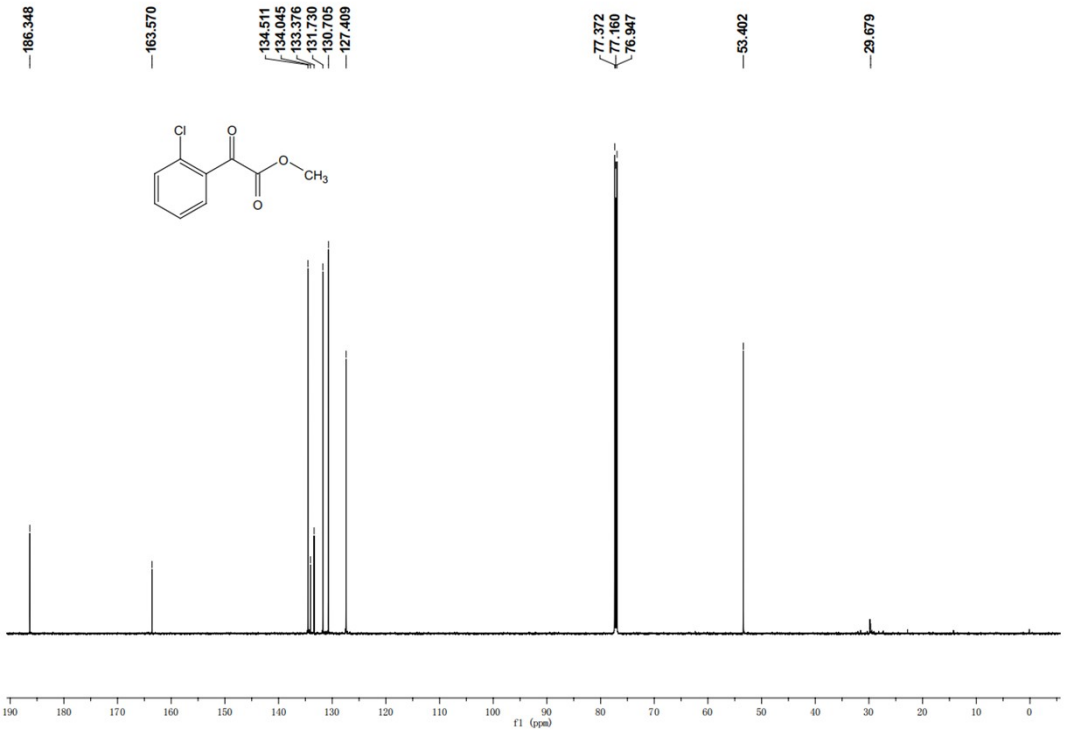
^{1}H and ^{13}C NMR spectra of **3bb**





^{1}H and ^{13}C NMR spectra of **3cb**





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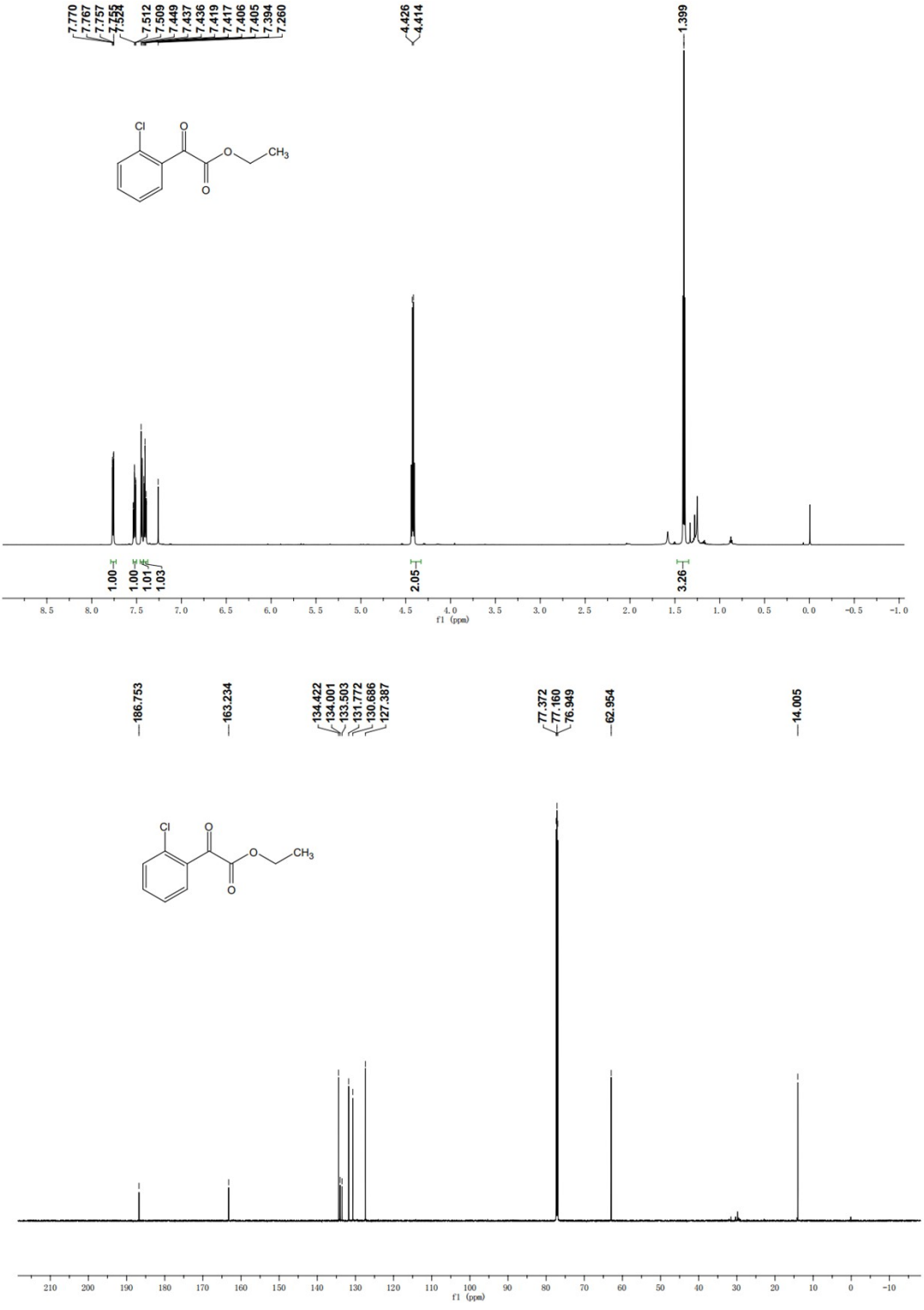

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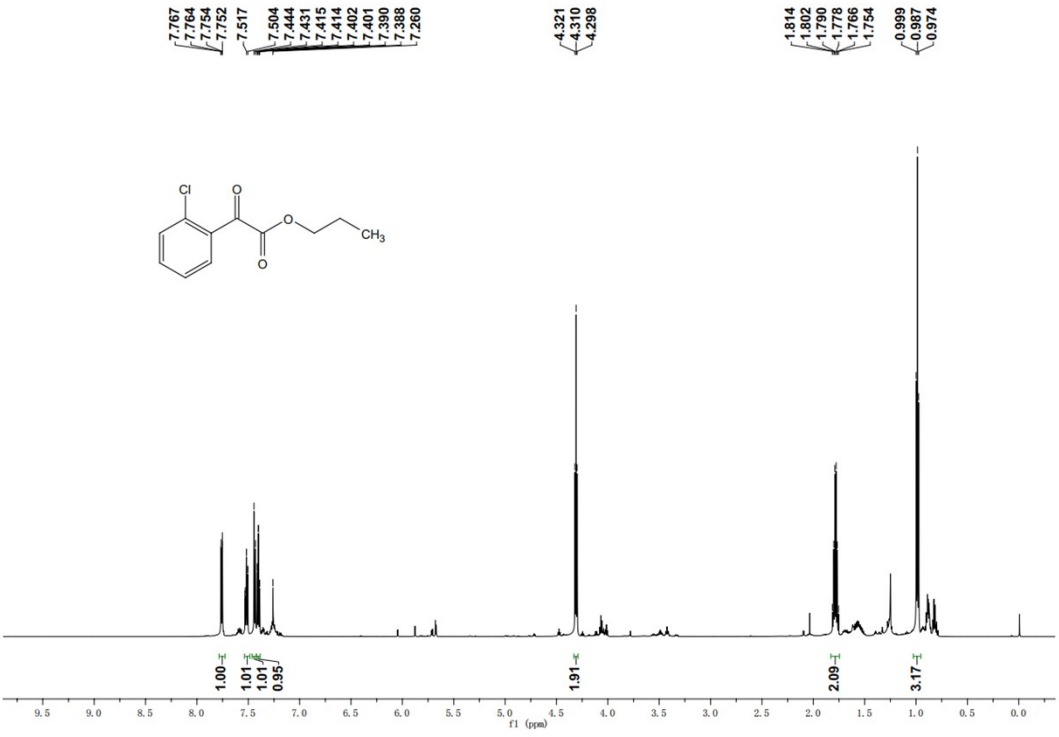
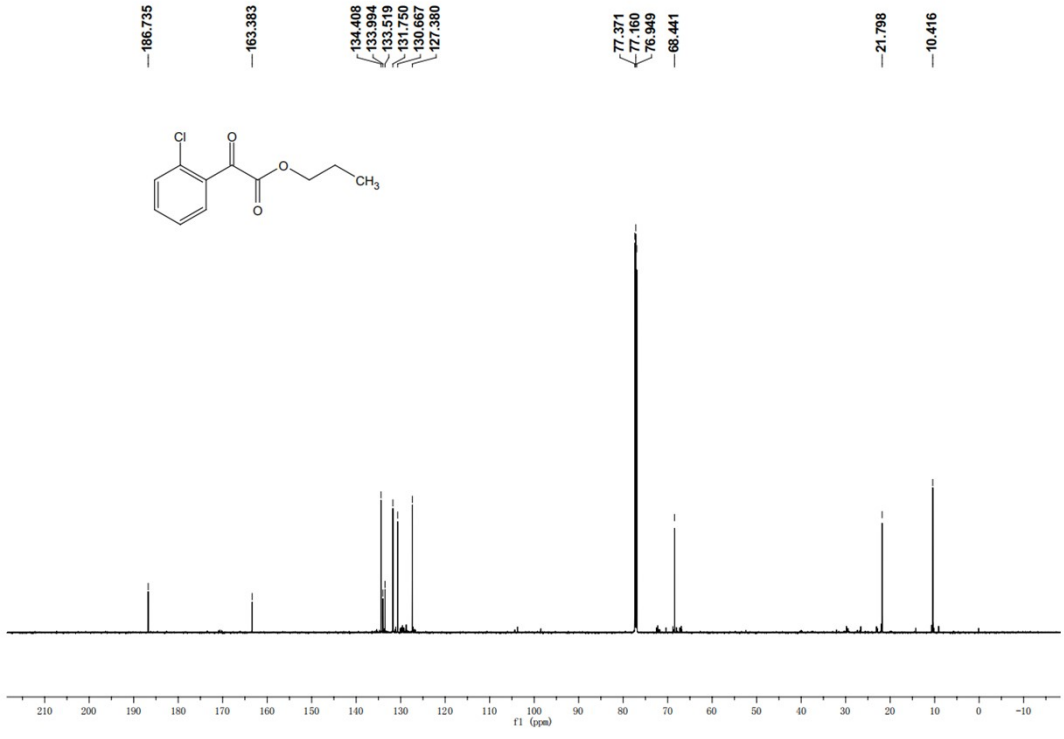
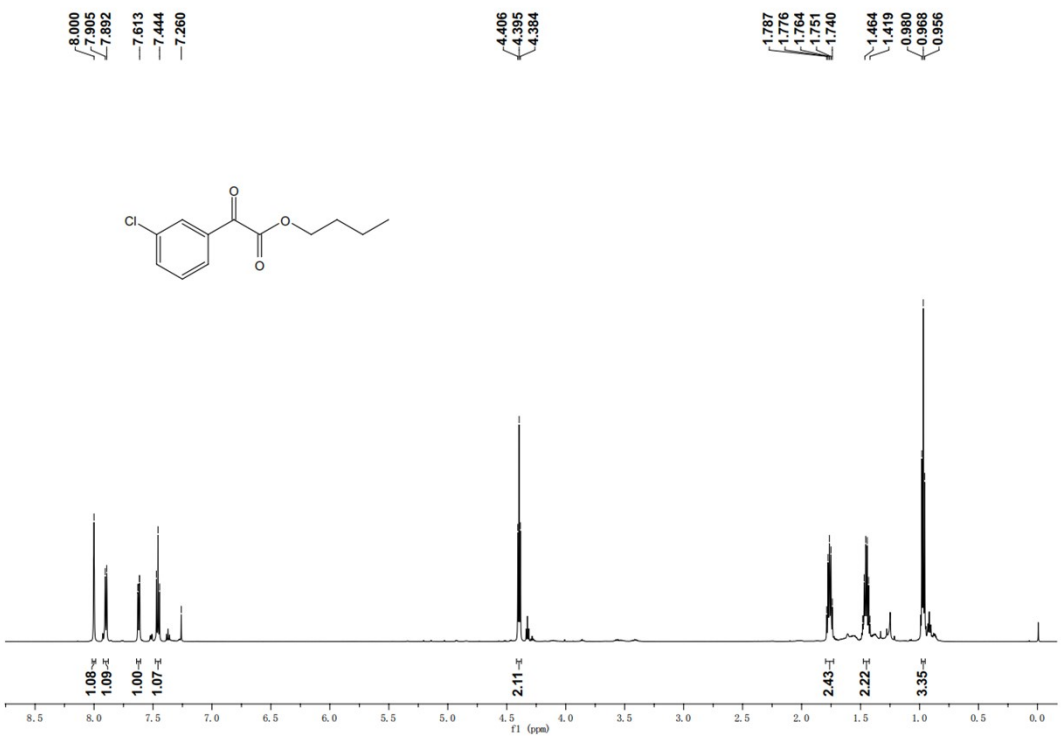
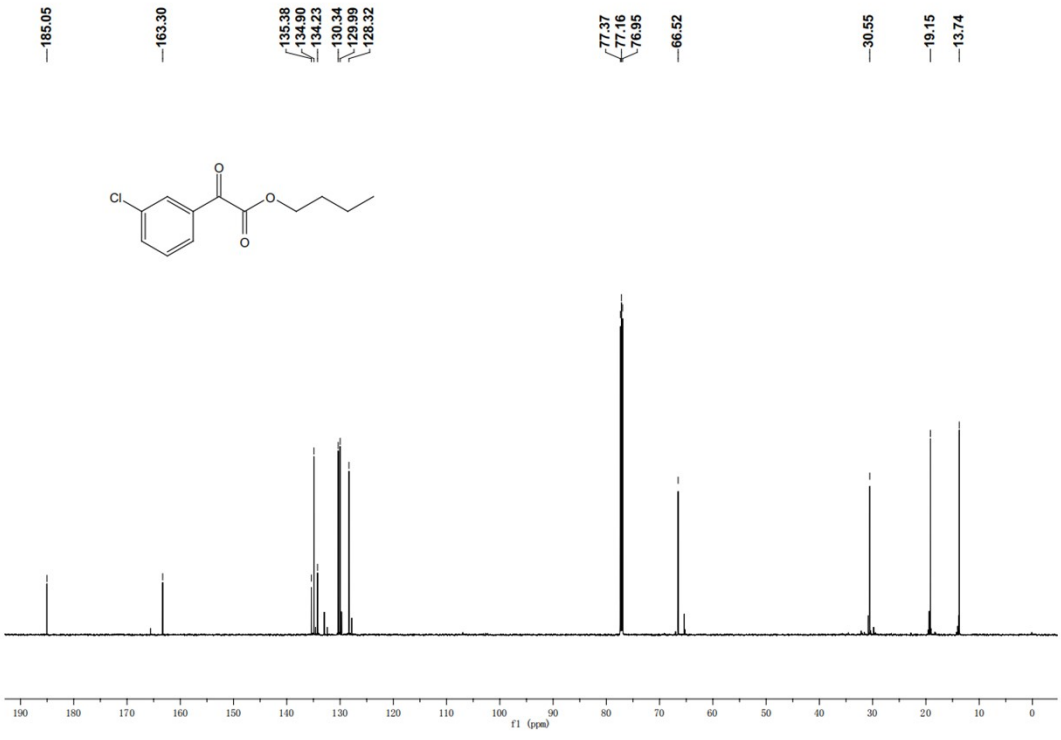




^{1}H and ^{13}C NMR spectra of **3da**





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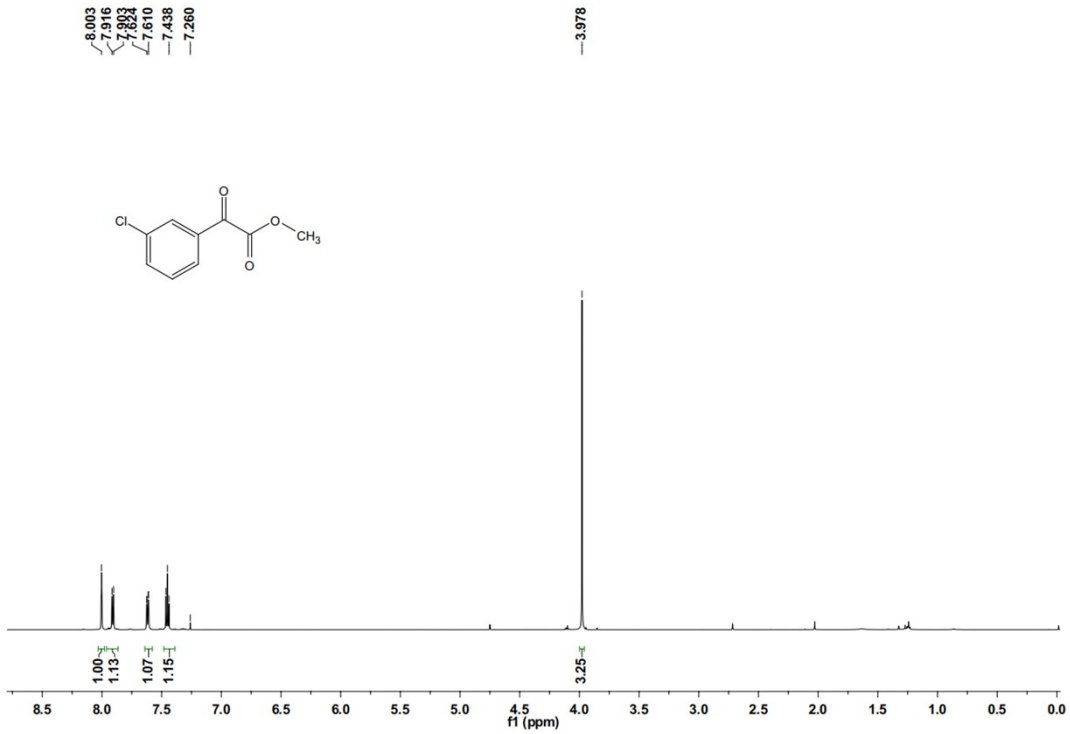

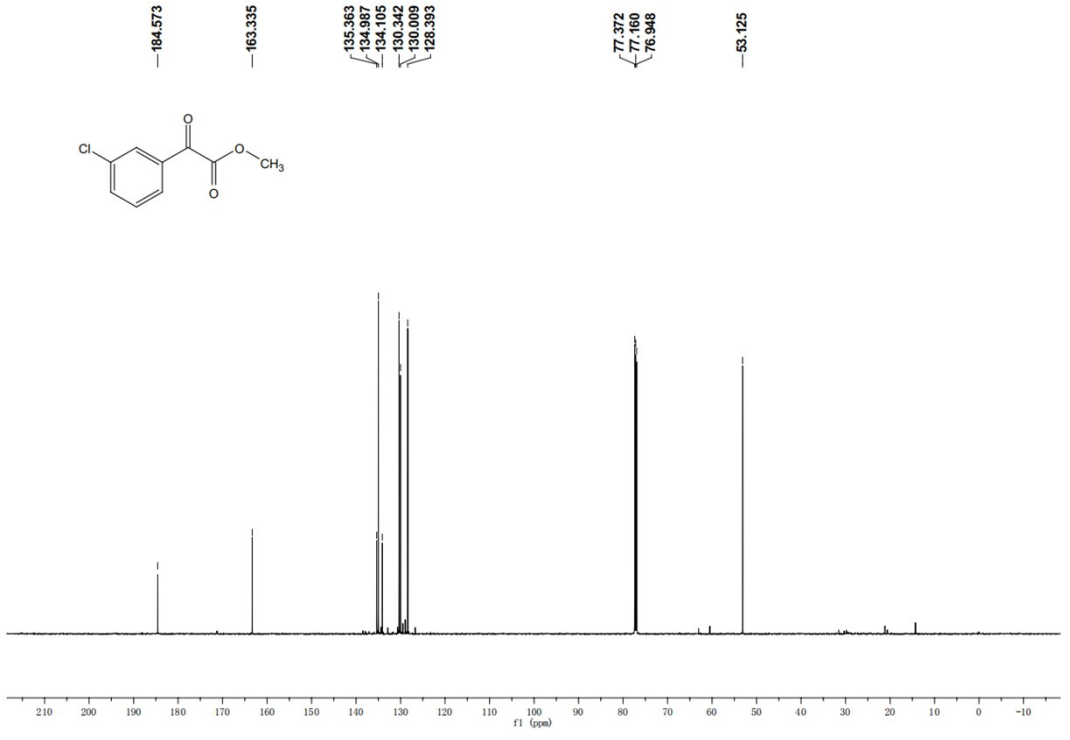

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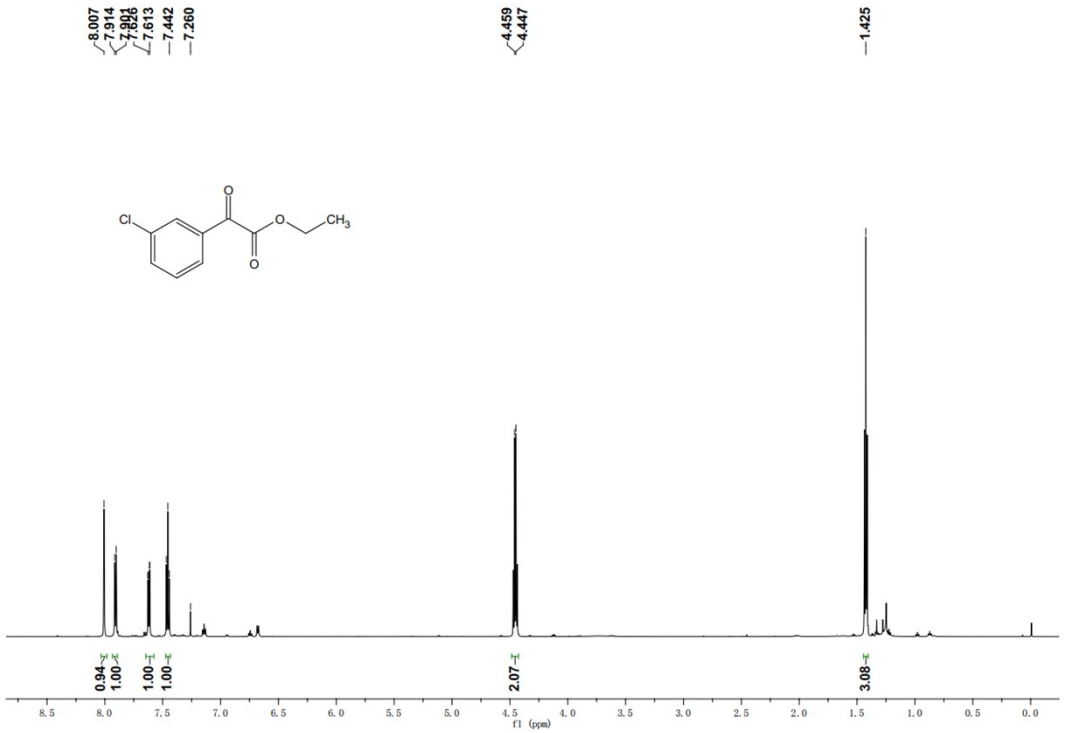
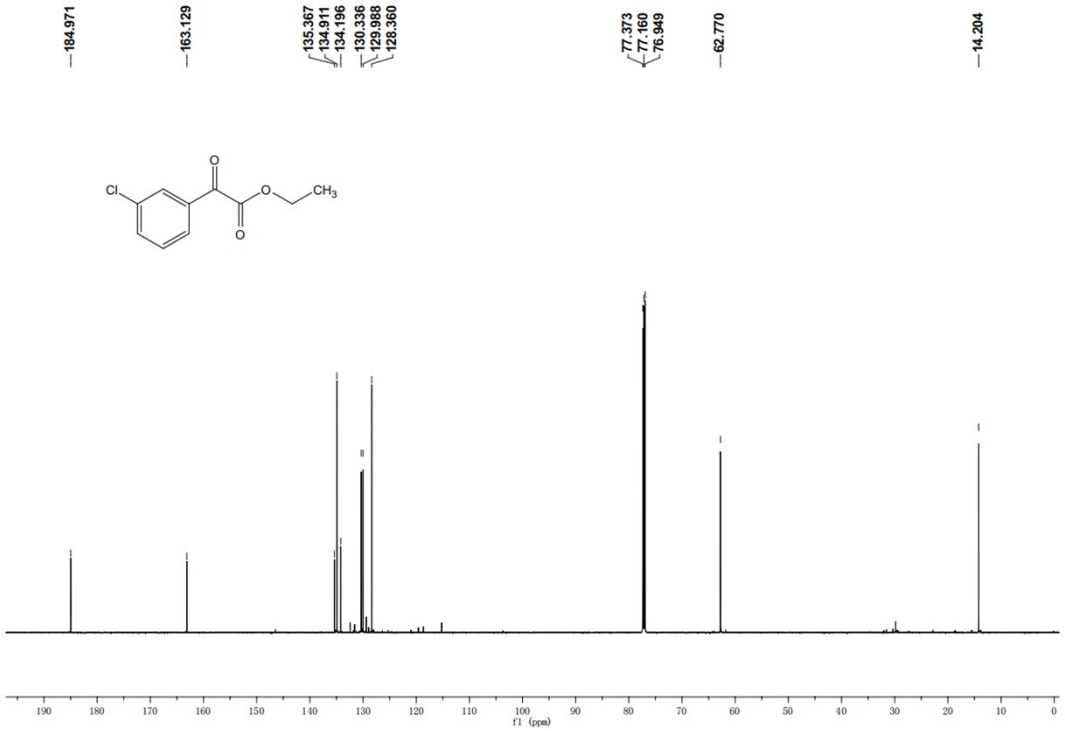
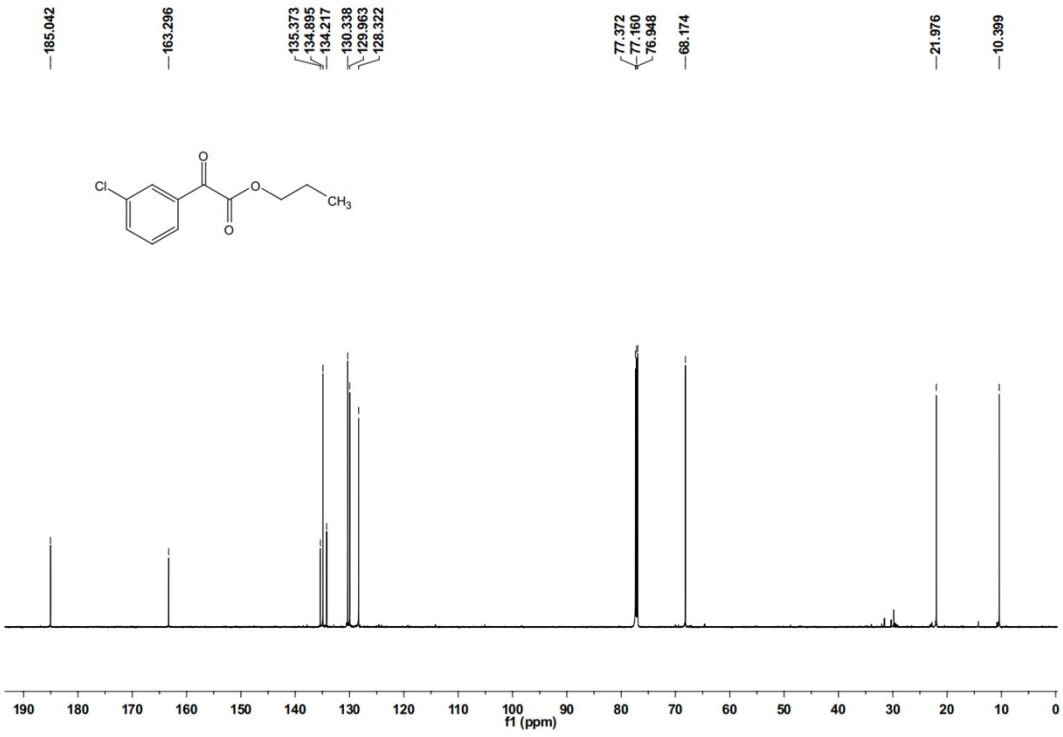




^{1}H and ^{13}C NMR spectra of **3dd**



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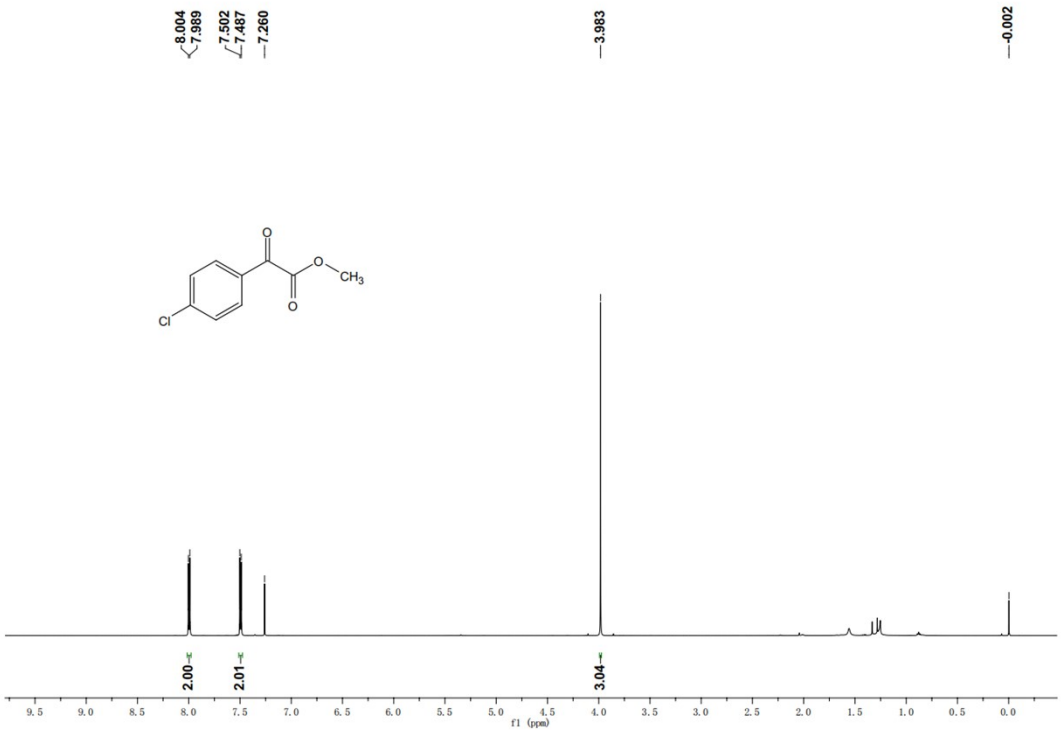

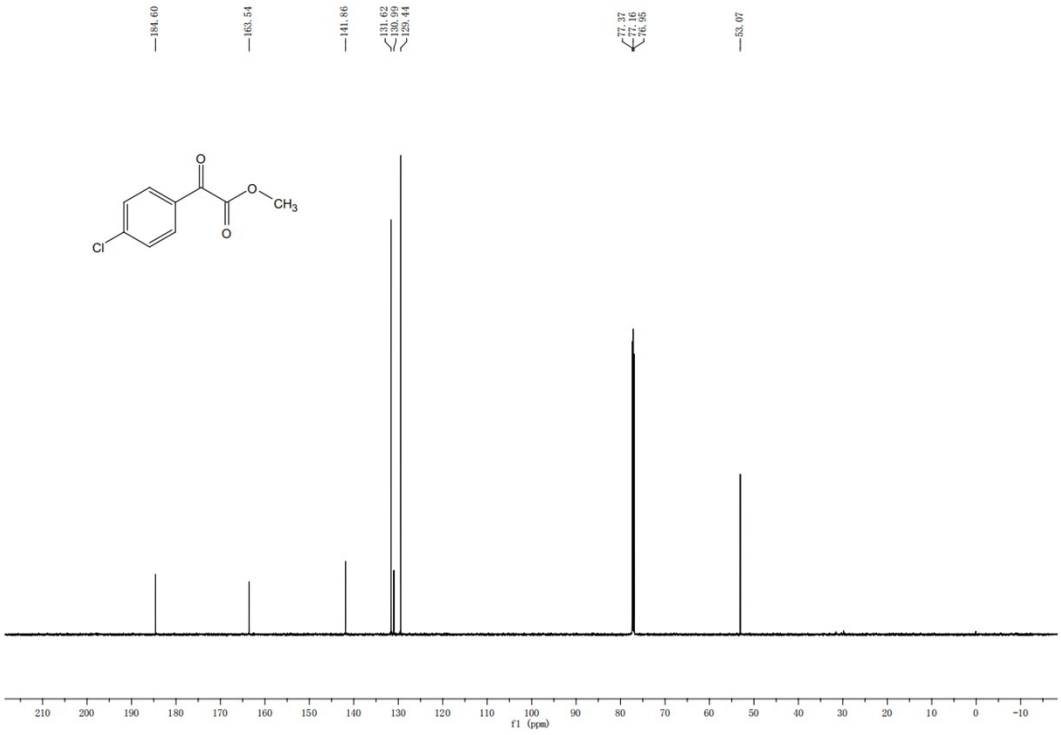

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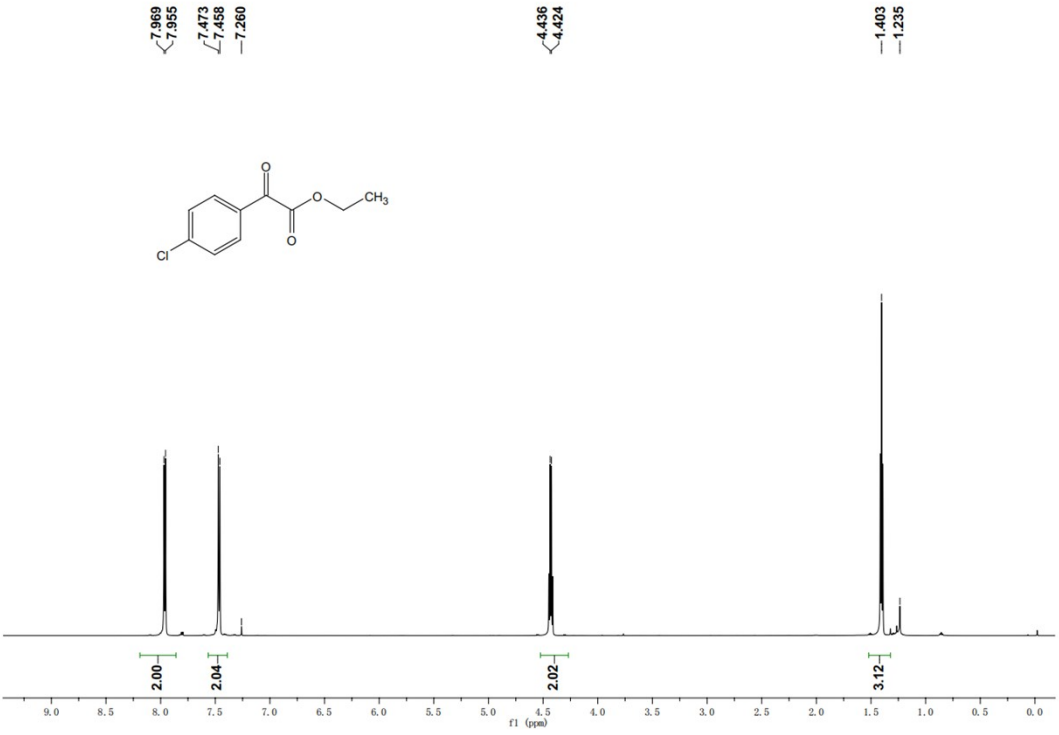

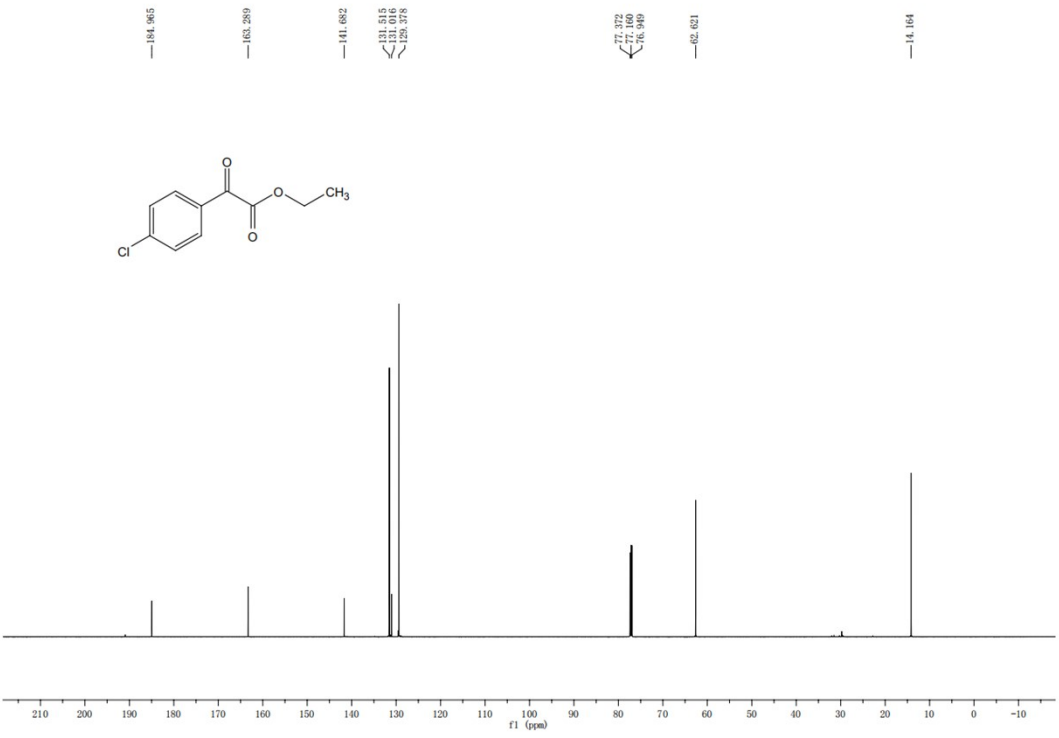

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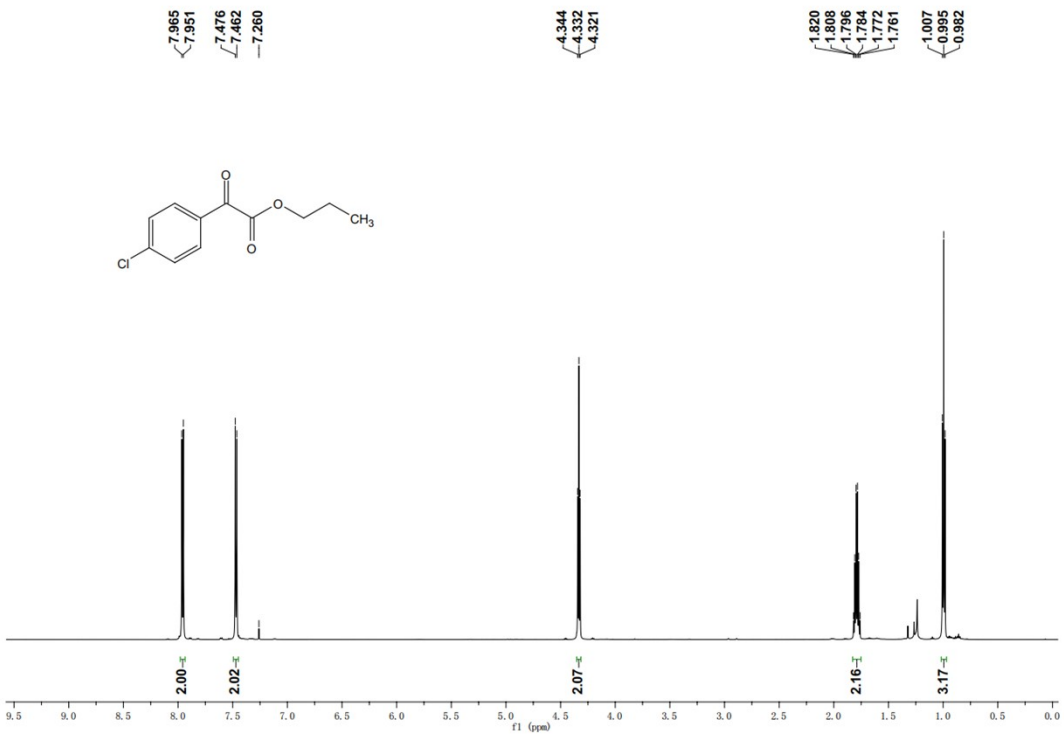

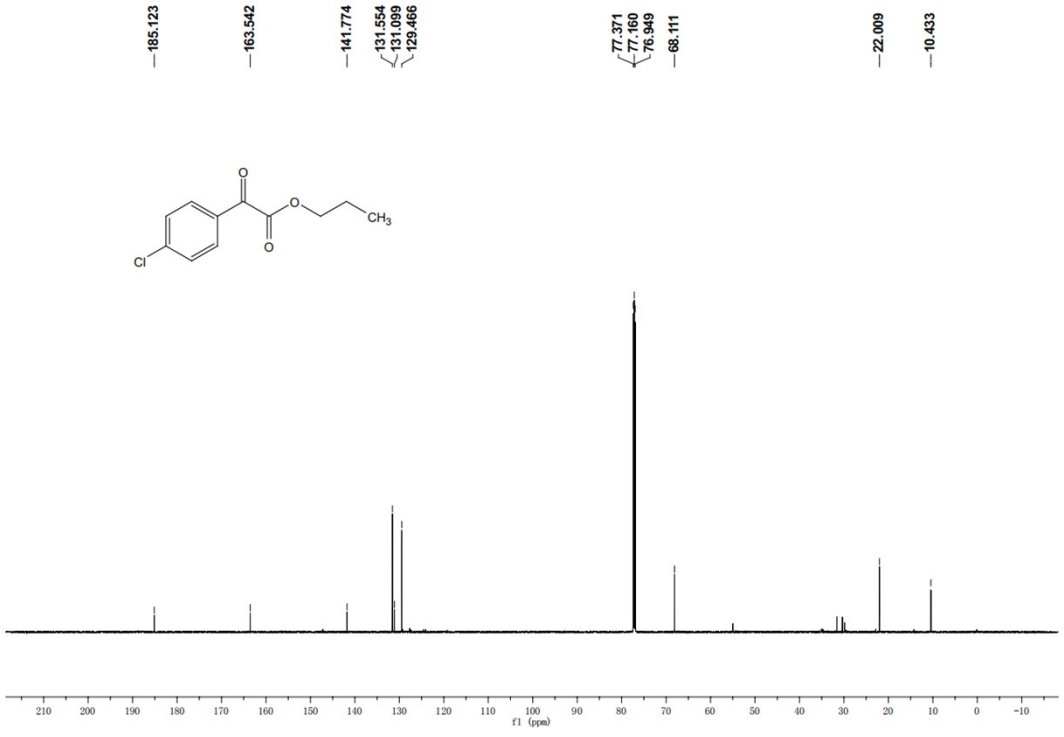

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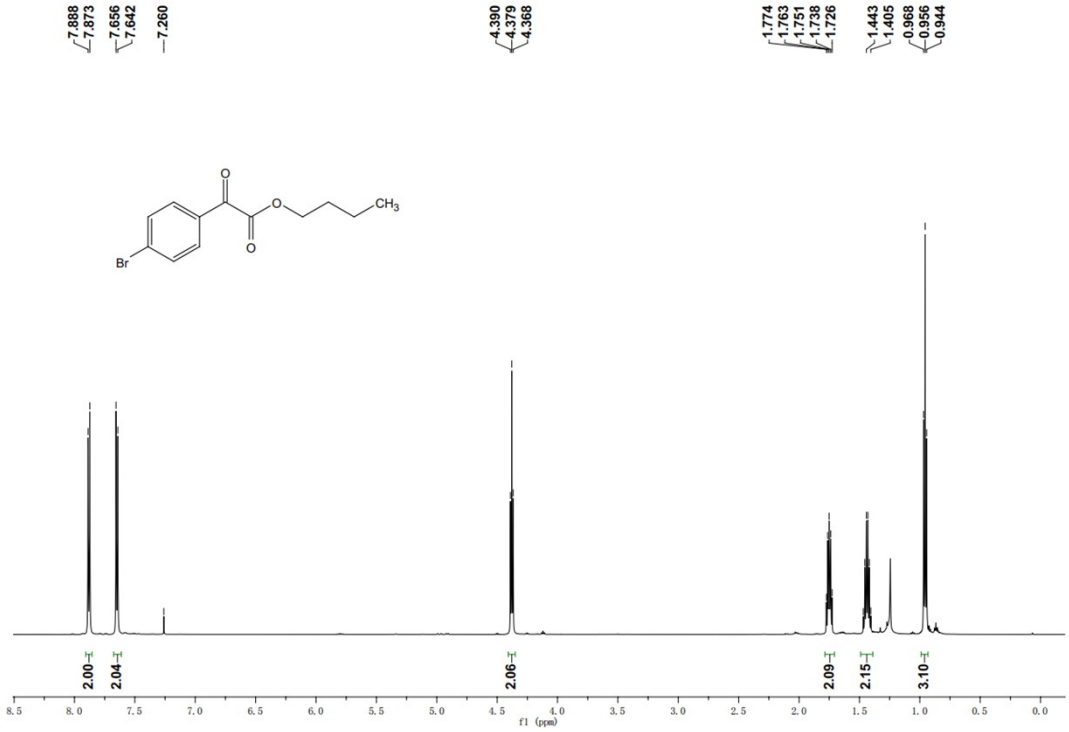

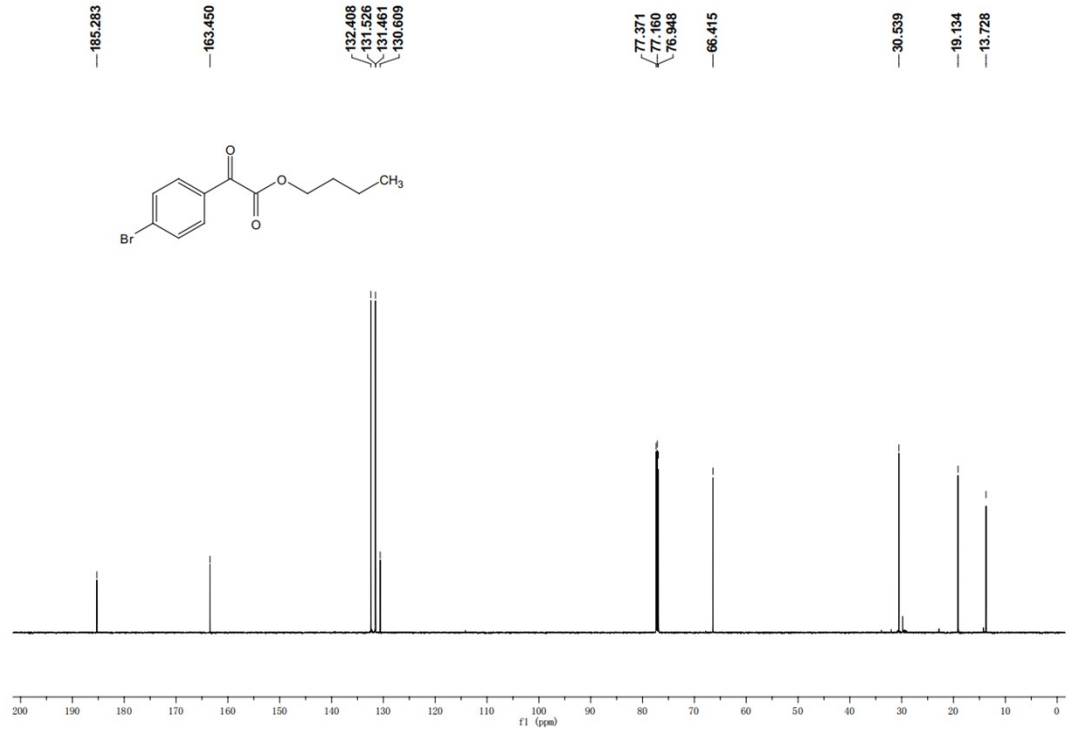

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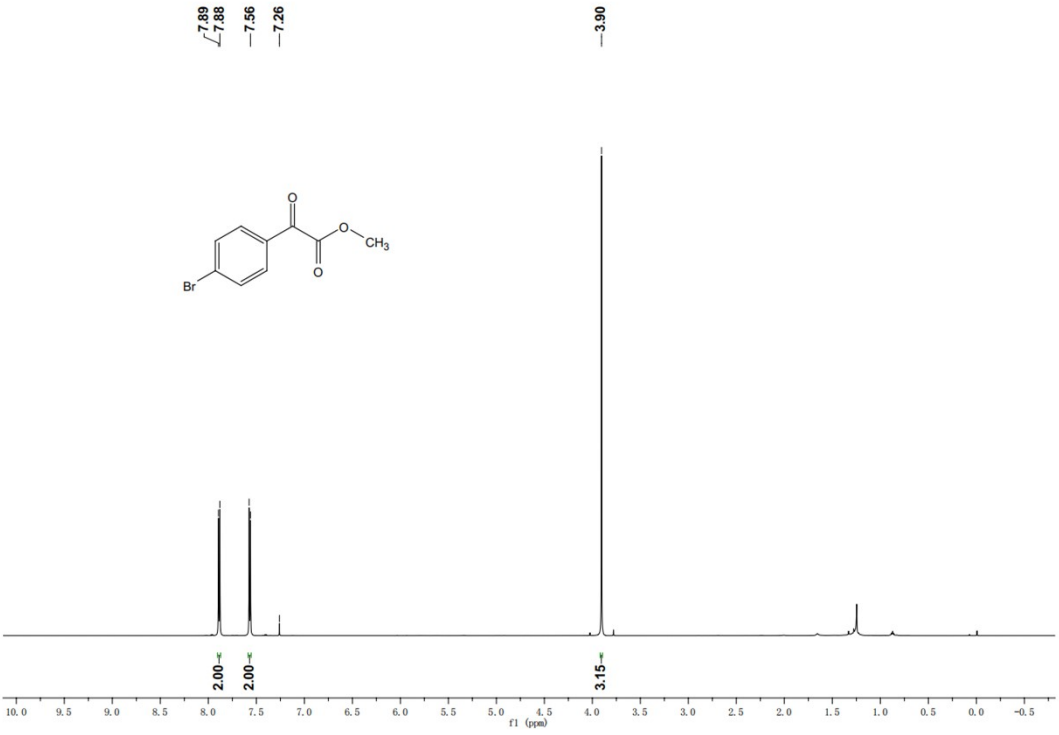

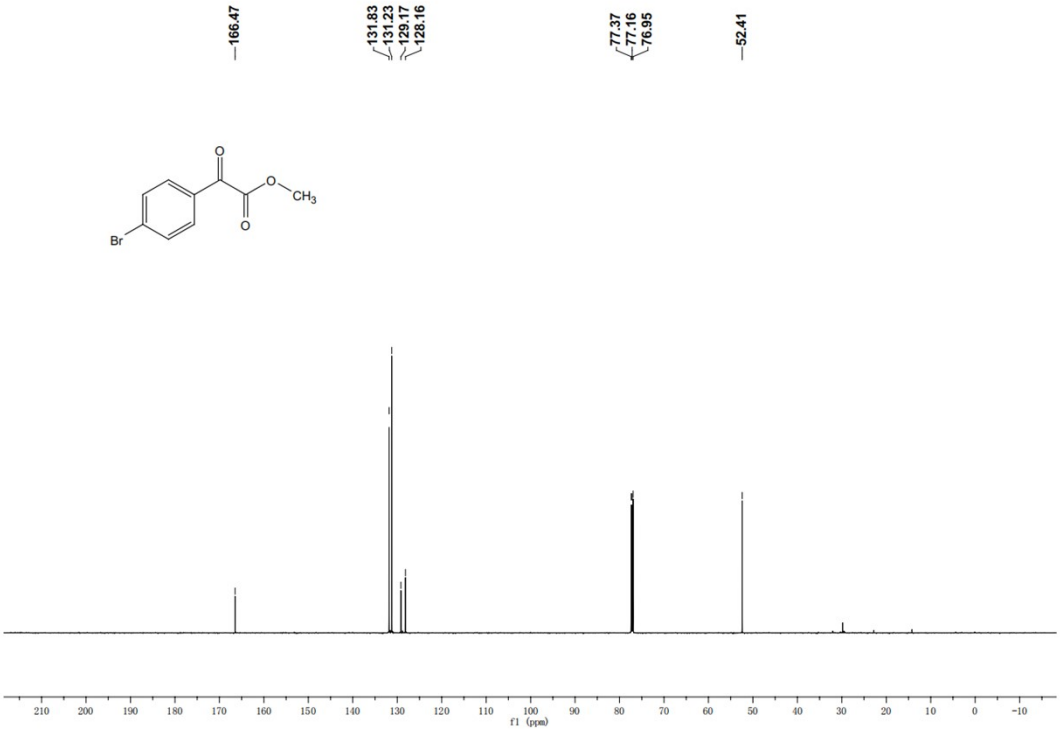

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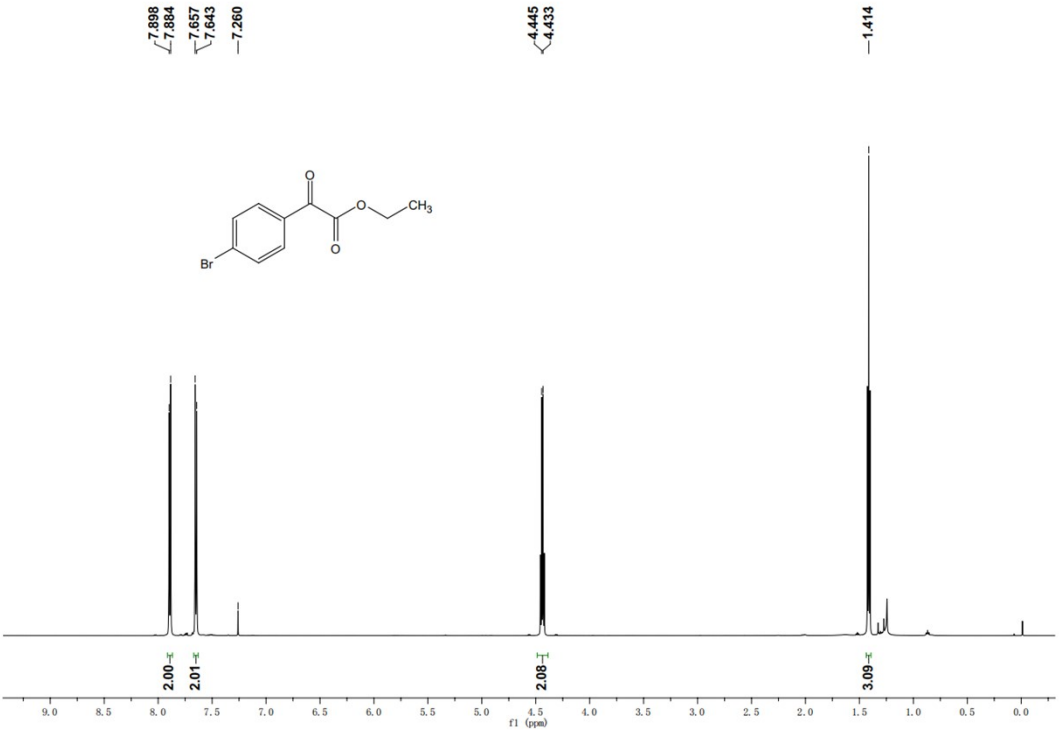

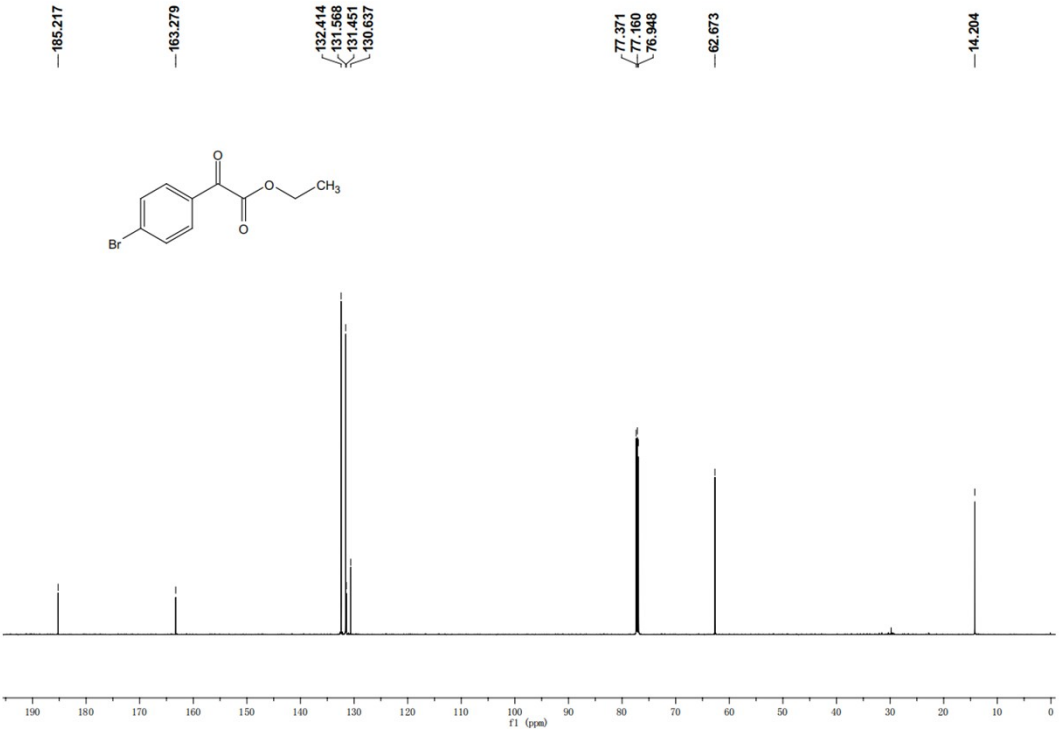

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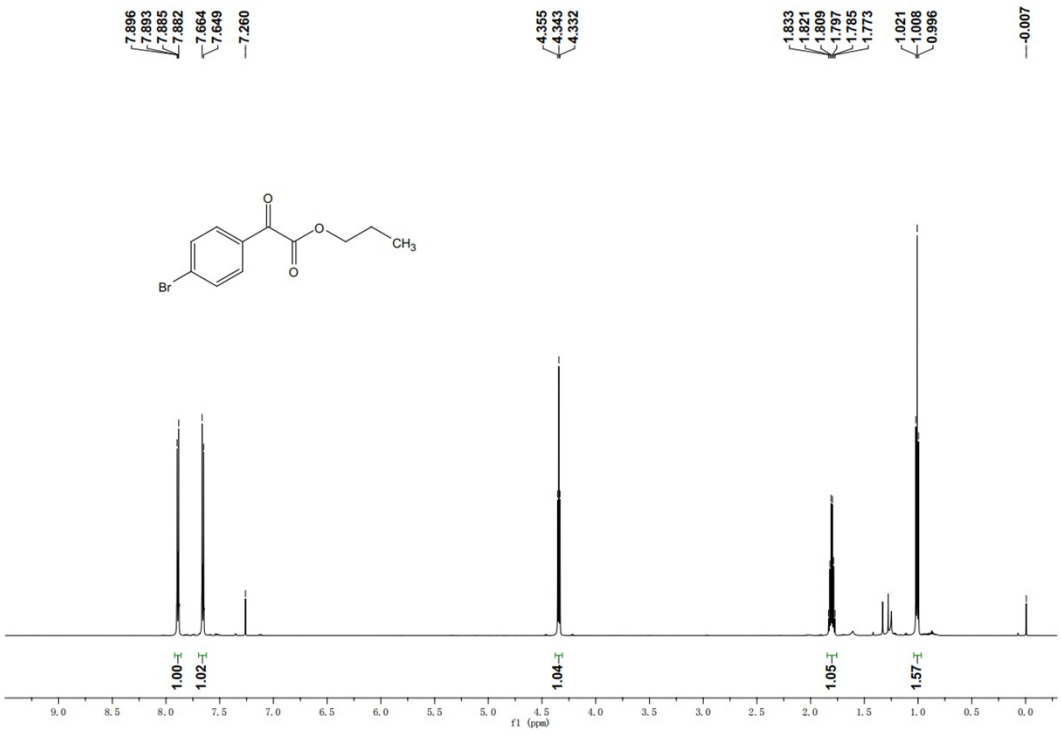
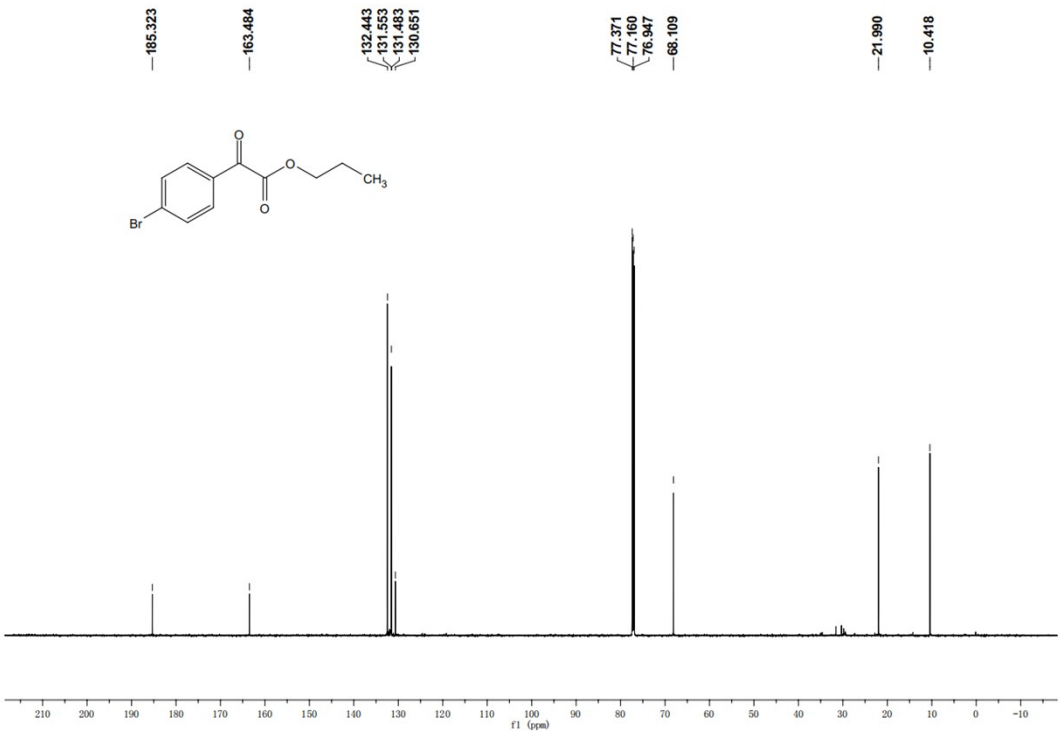
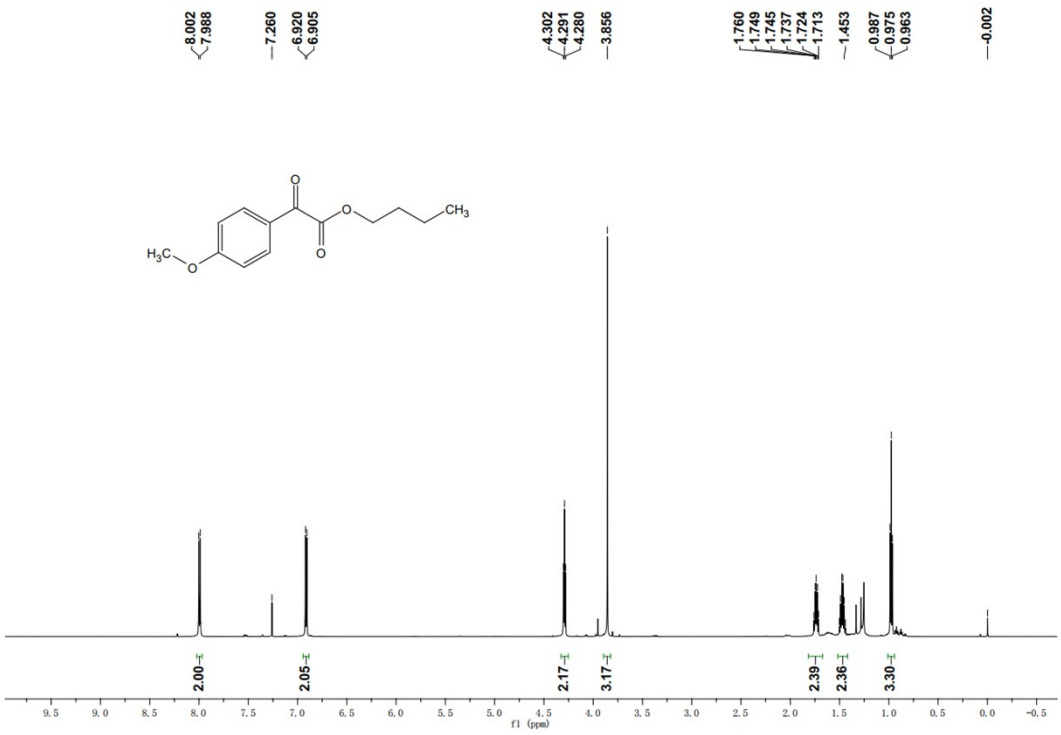
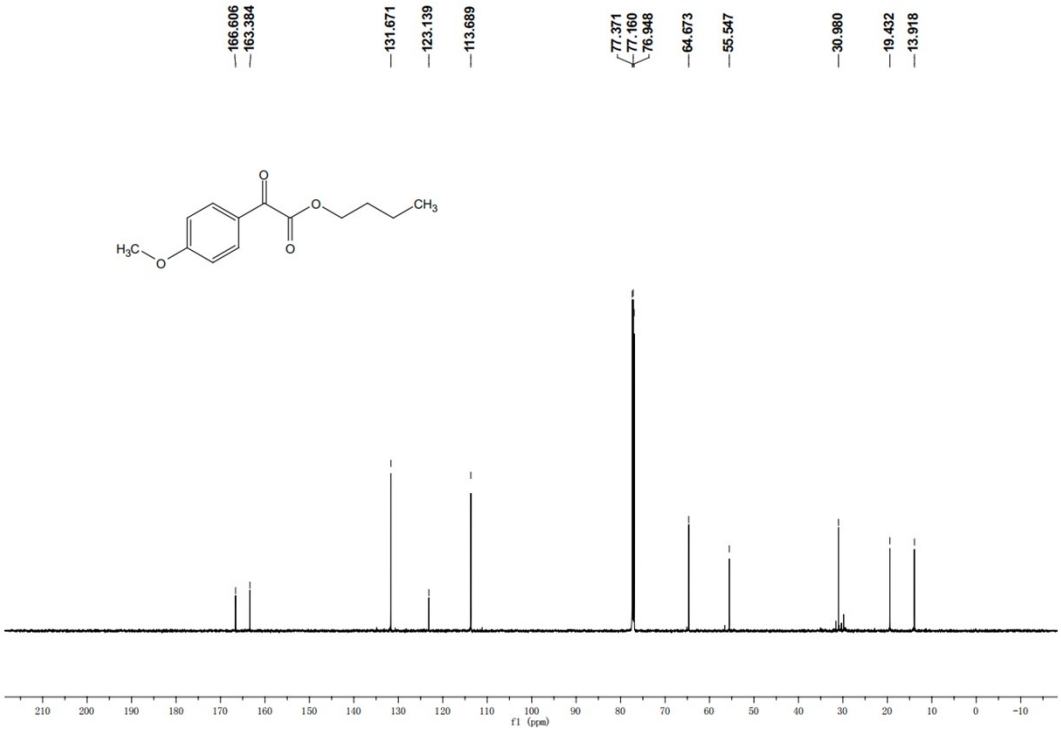


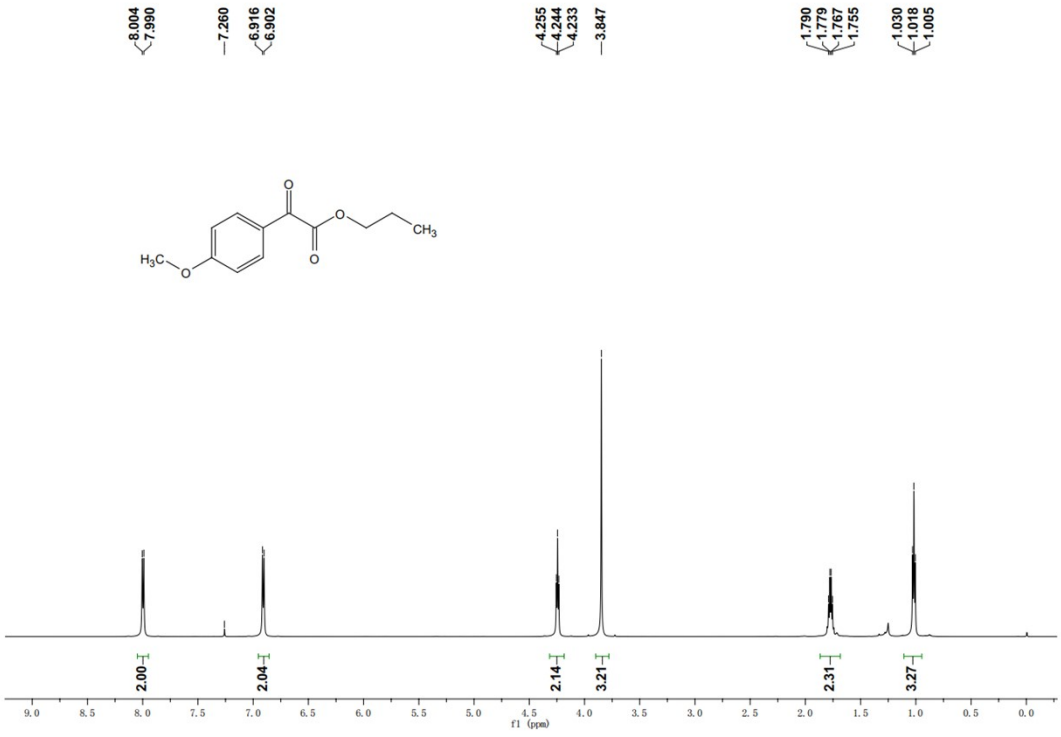


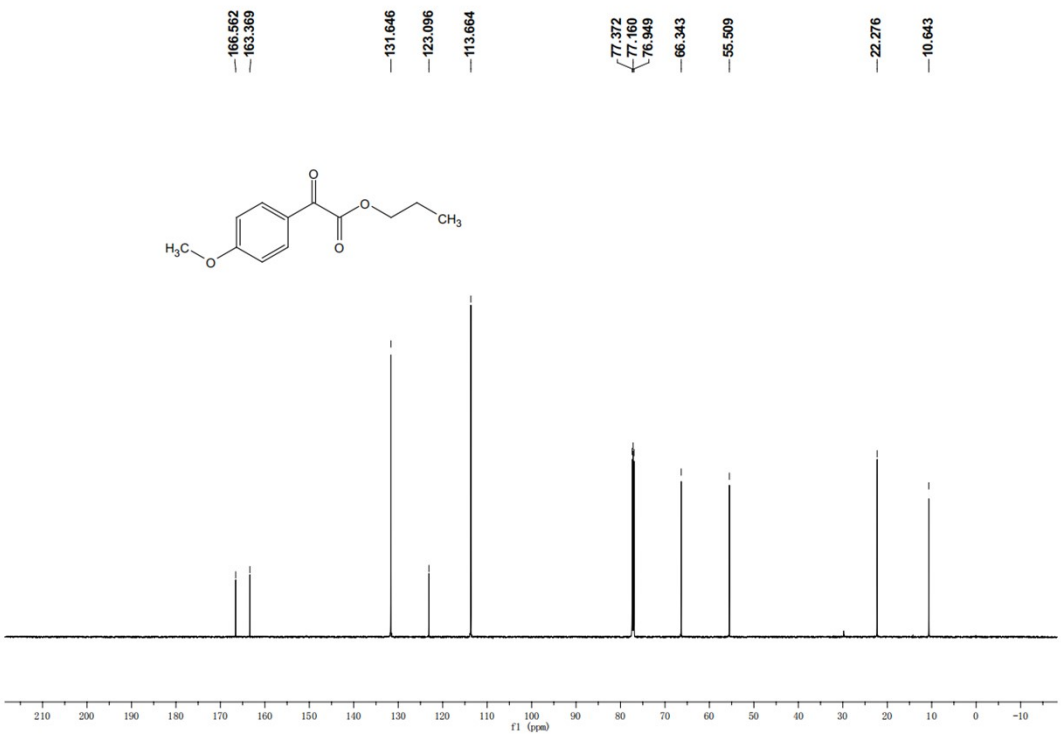
^{1}H and ^{13}C NMR spectra of **3ga**



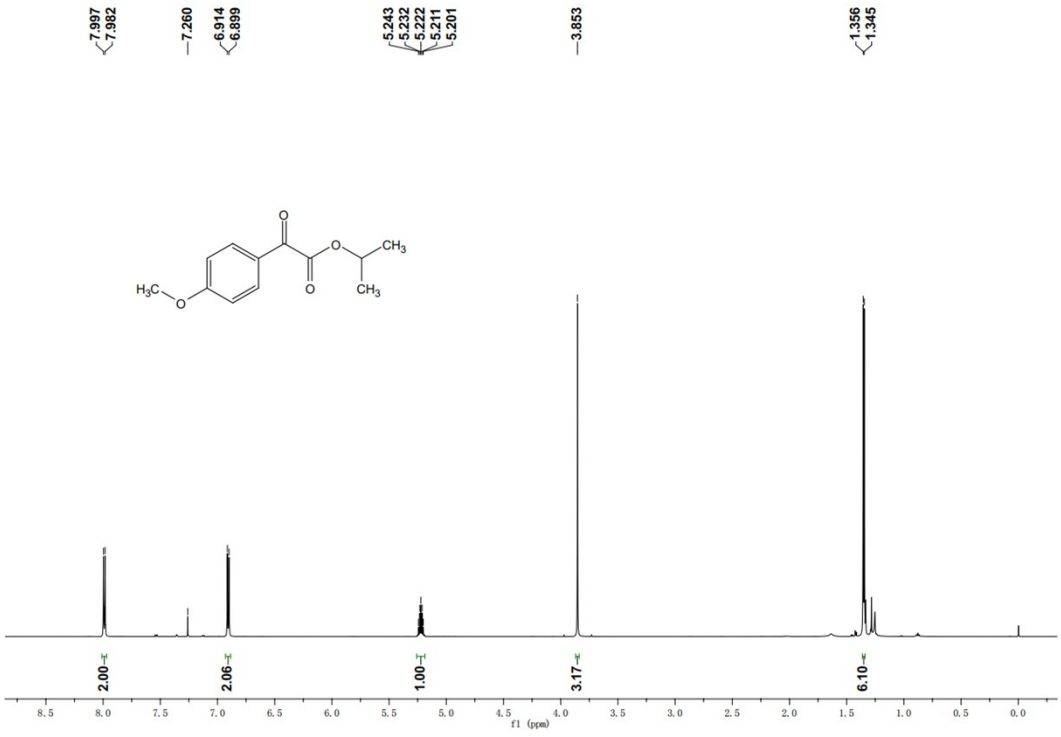


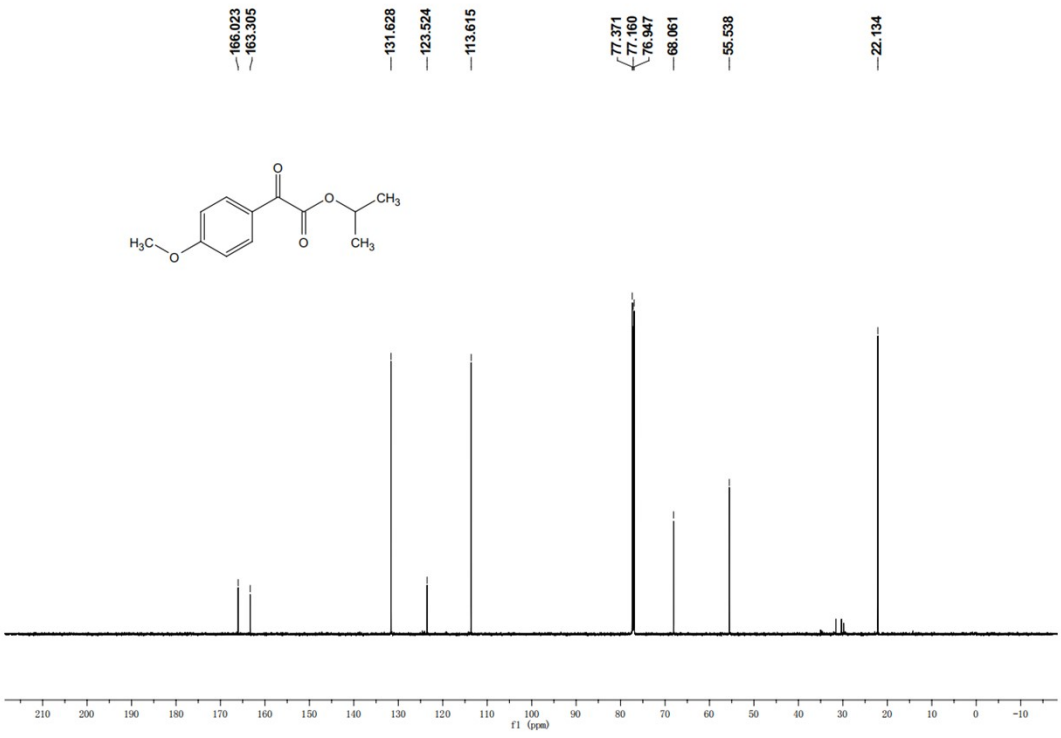
^{1}H and ^{13}C NMR spectra of **3gd**



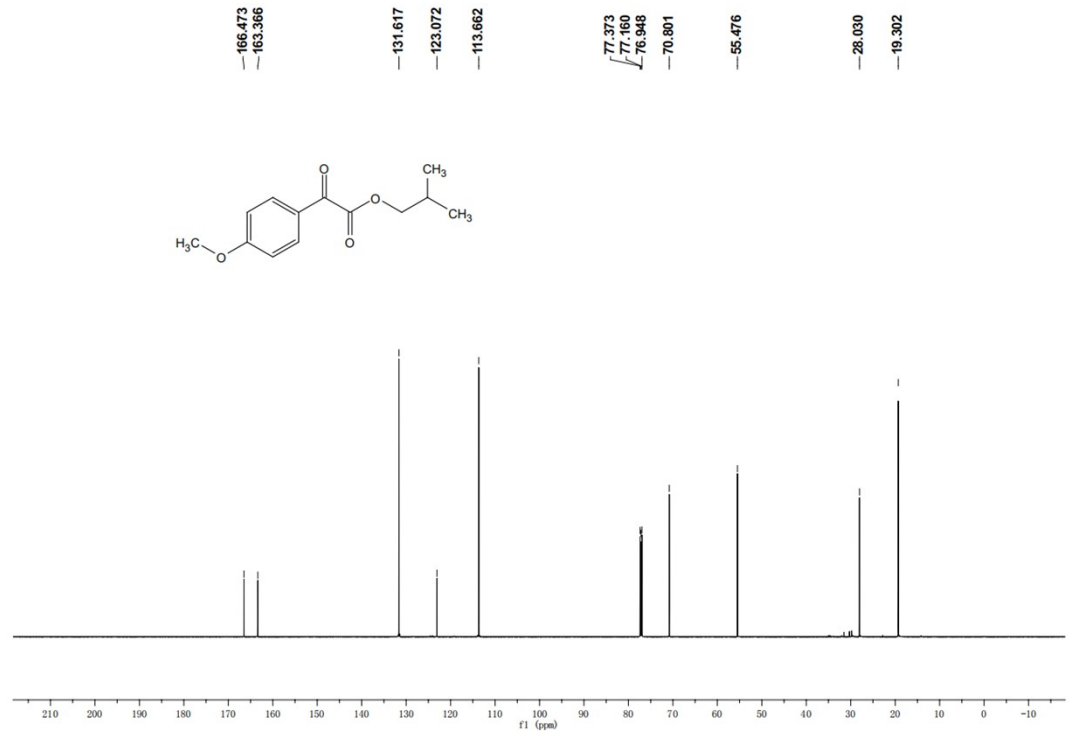


^{1}H and ^{13}C NMR spectra of **3gf**

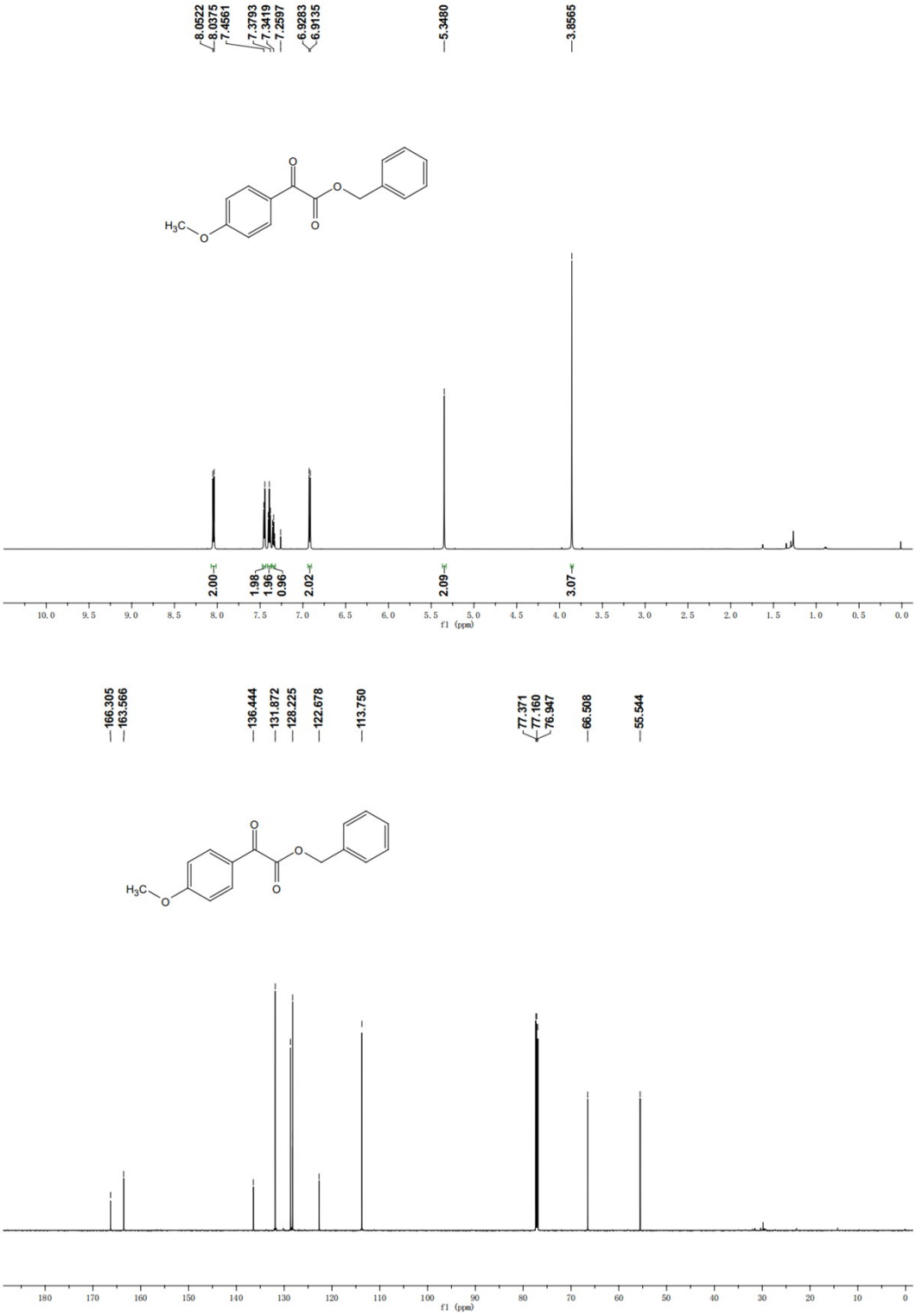




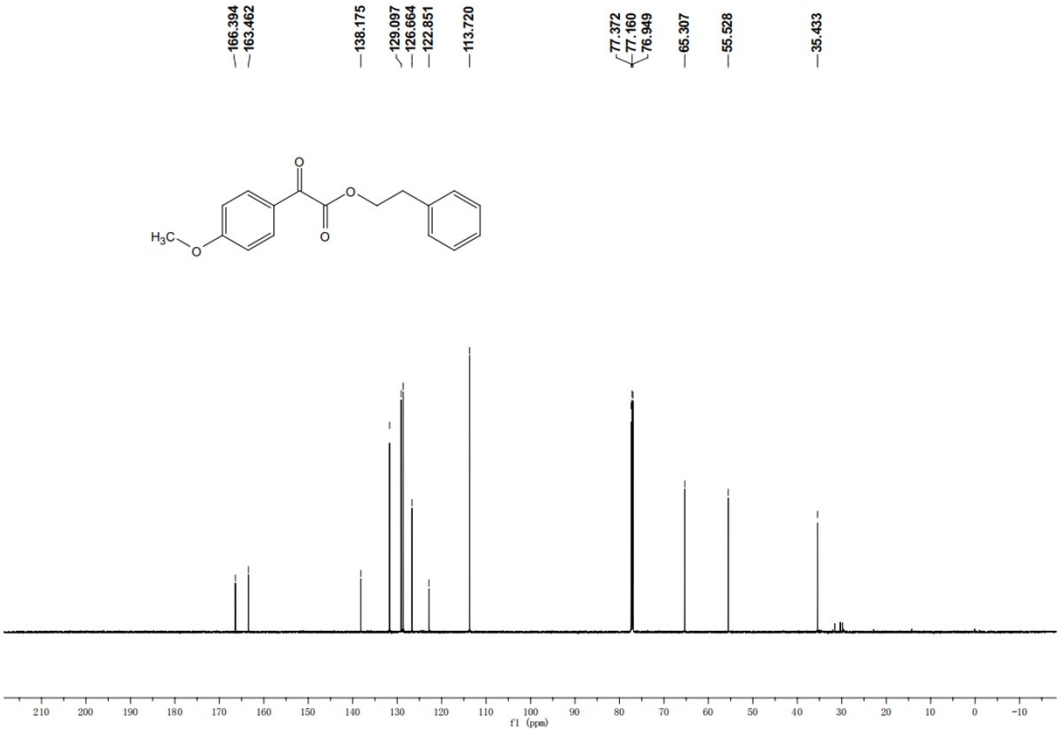
^{1}H and ^{13}C NMR spectra of **3gg**



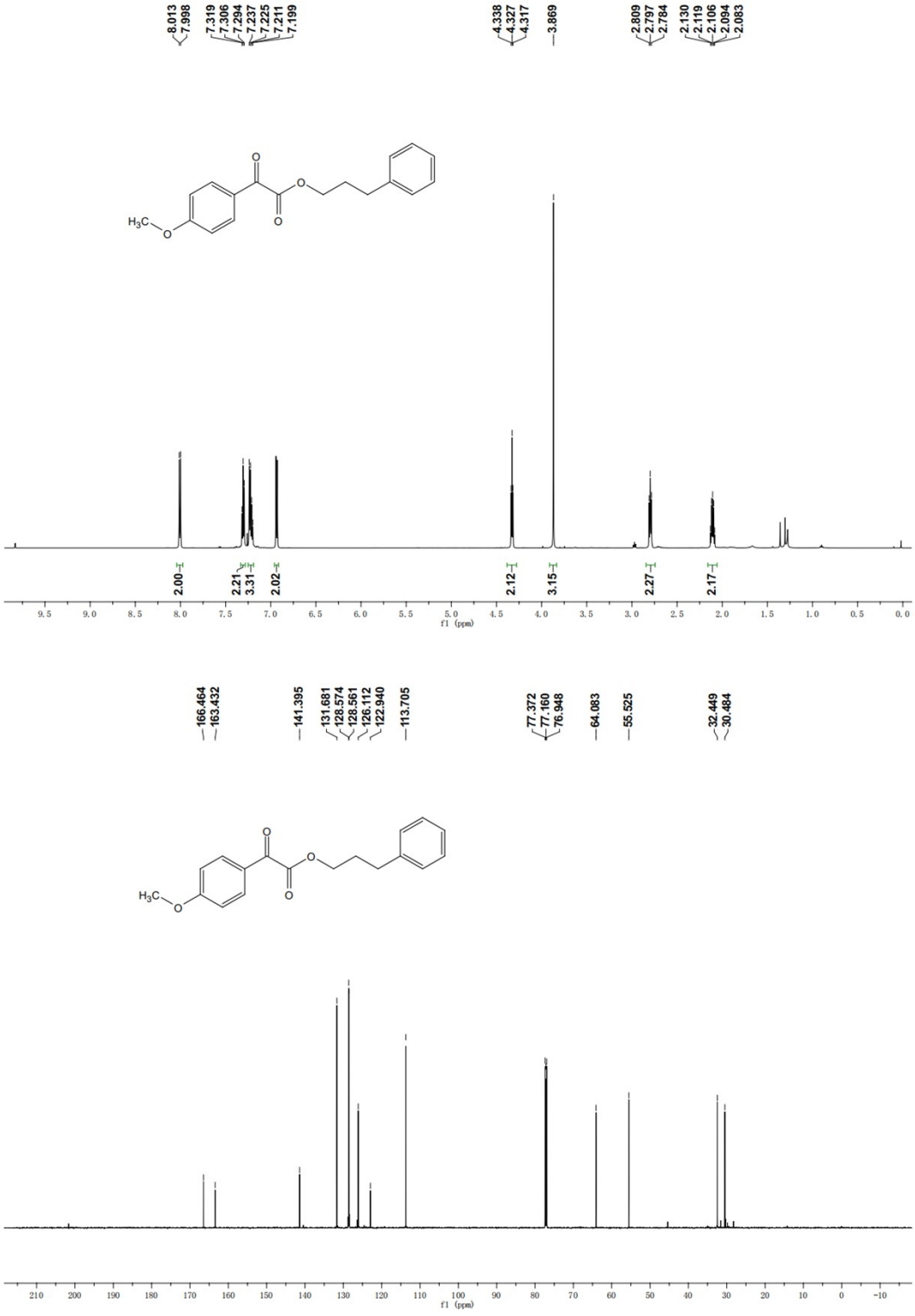
^{1}H and ^{13}C NMR spectra of **3gi**



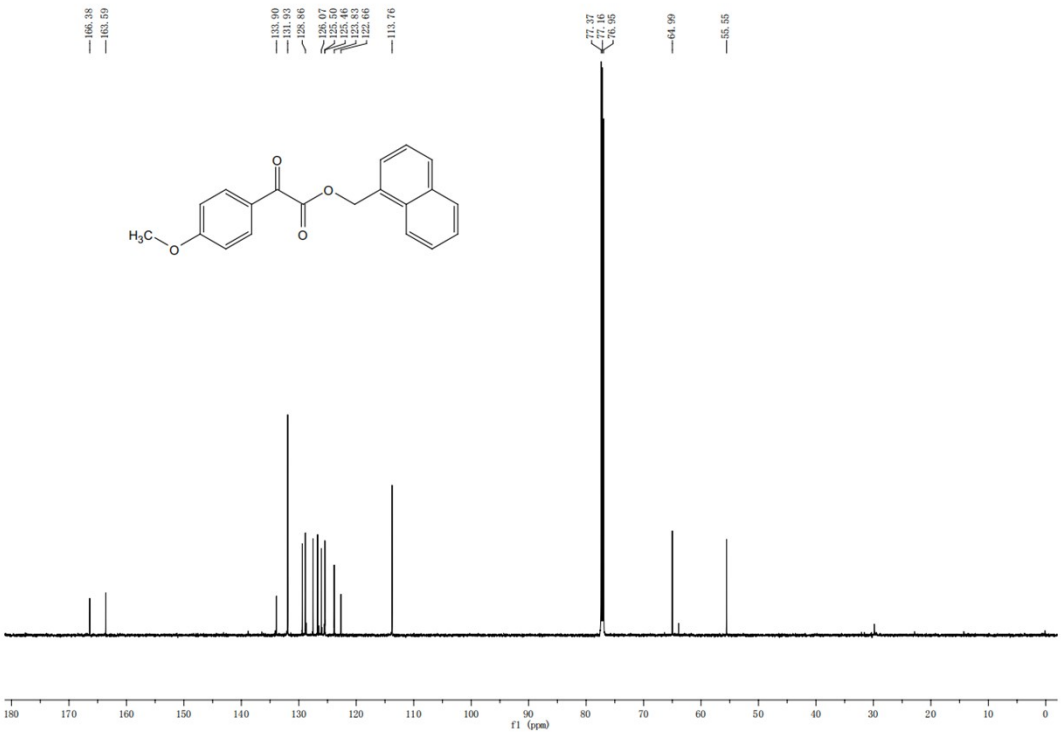
^{1}H and ^{13}C NMR spectra of **3gj**



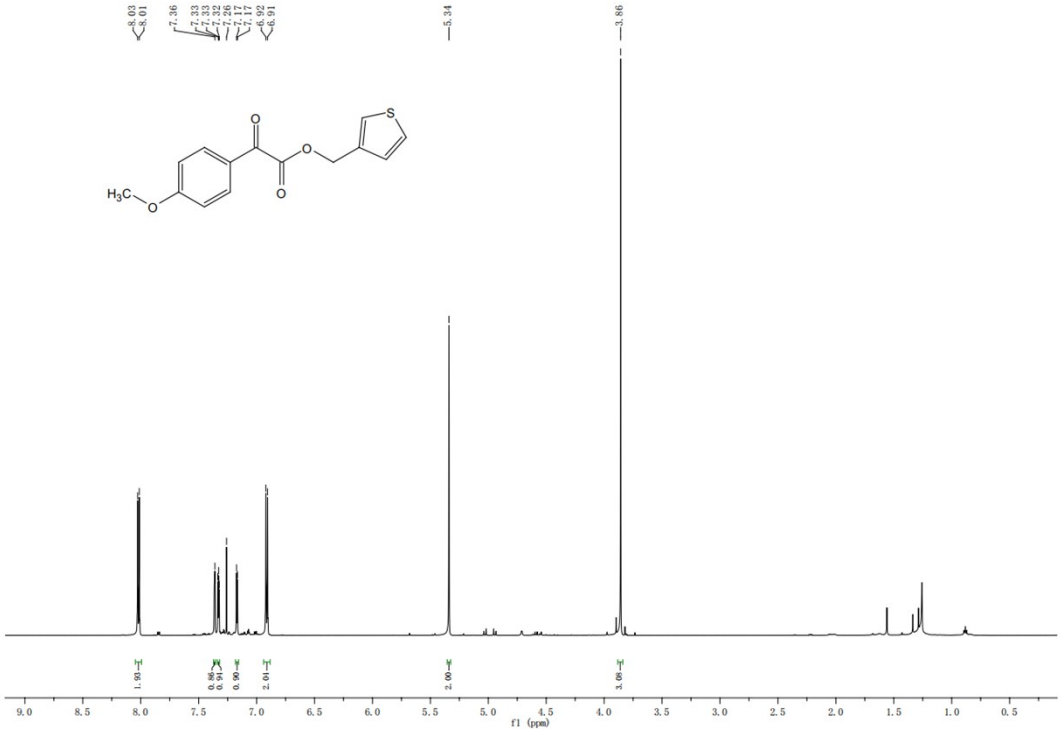
^{1}H and ^{13}C NMR spectra of **3gk**

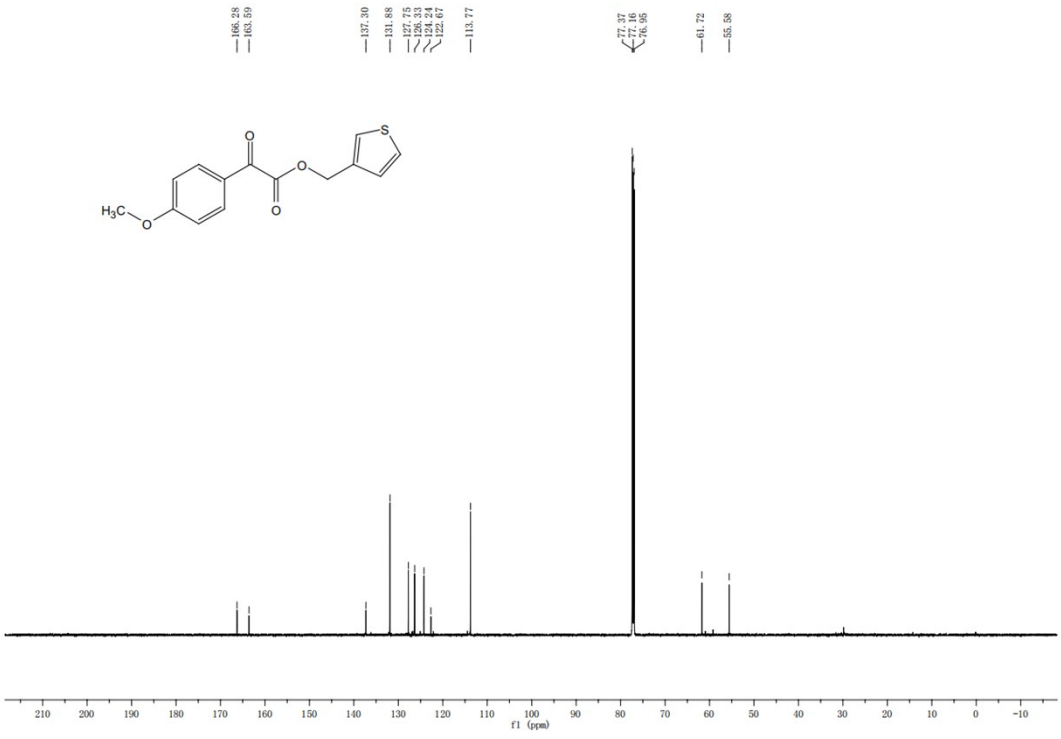


^{1}H and ^{13}C NMR spectra of **3gp**

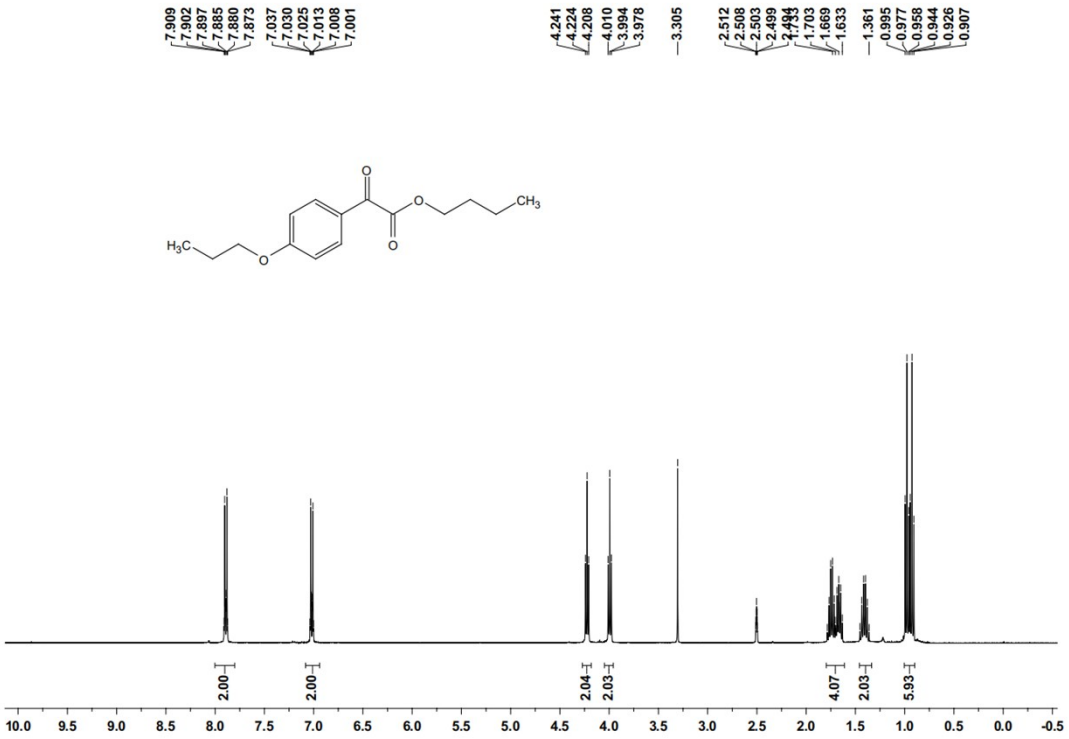


^{1}H and ^{13}C NMR spectra of **3gq**

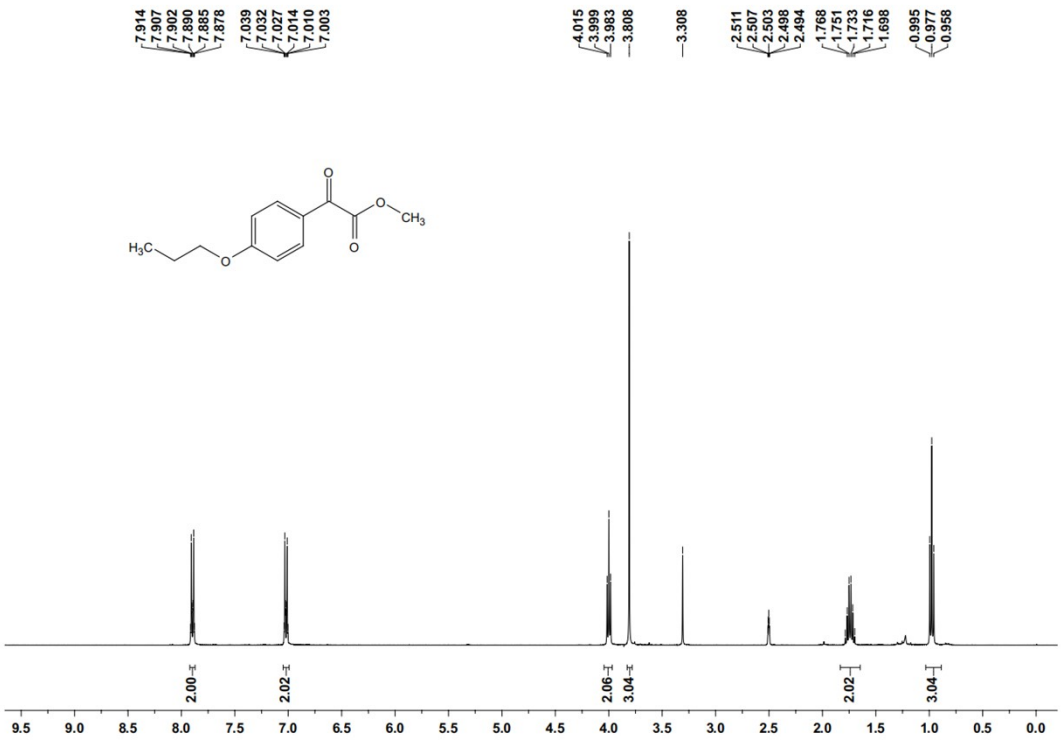


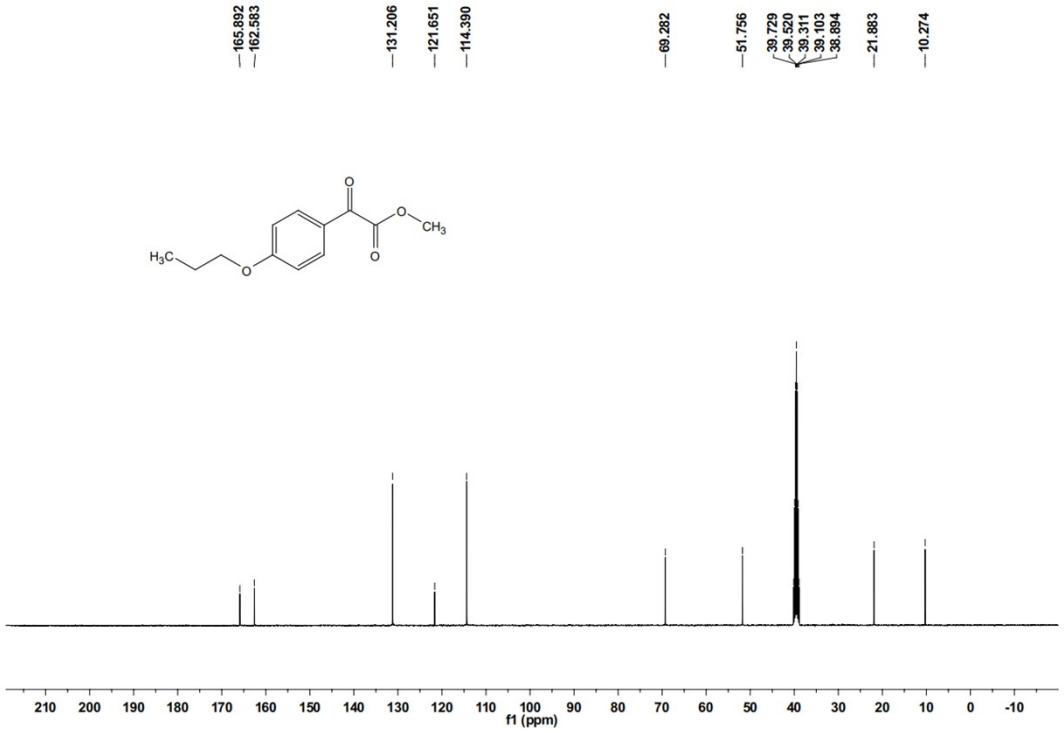


^{1}H and ^{13}C NMR spectra of **3ha**

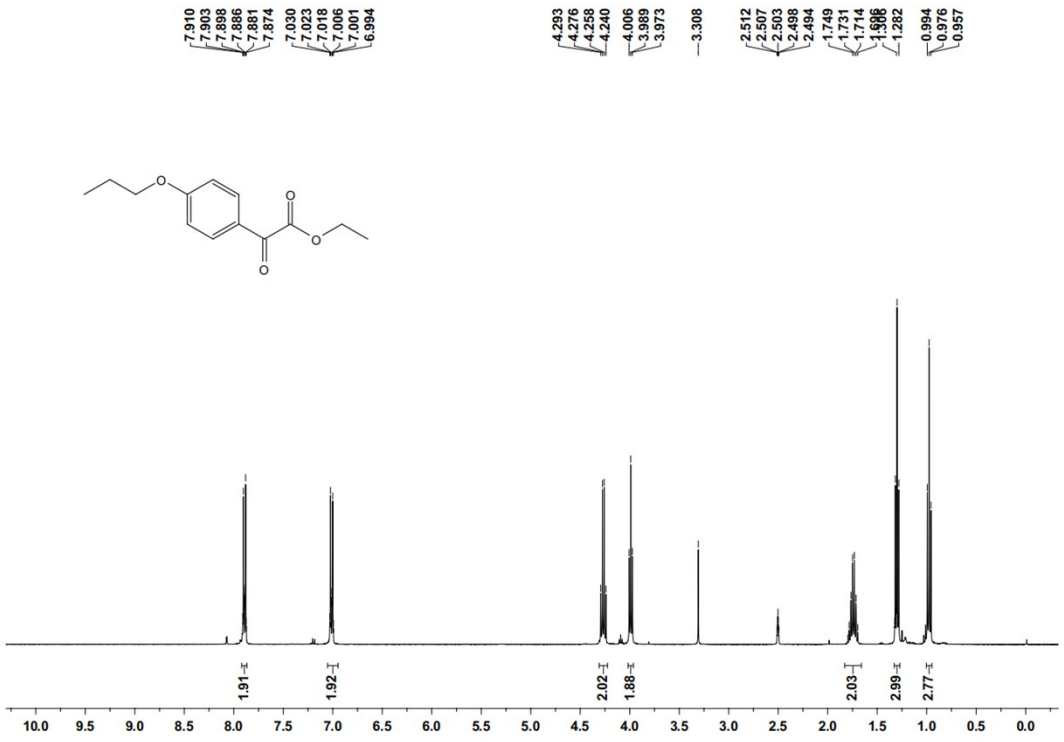


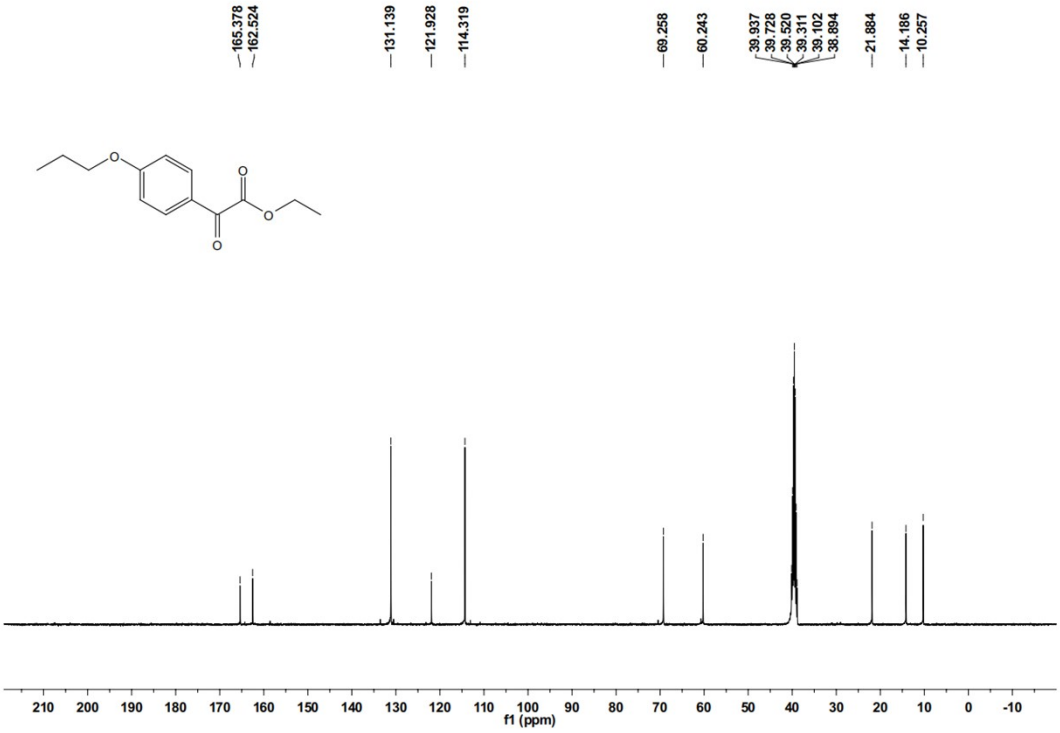
^{1}H and ^{13}C NMR spectra of **3hb**



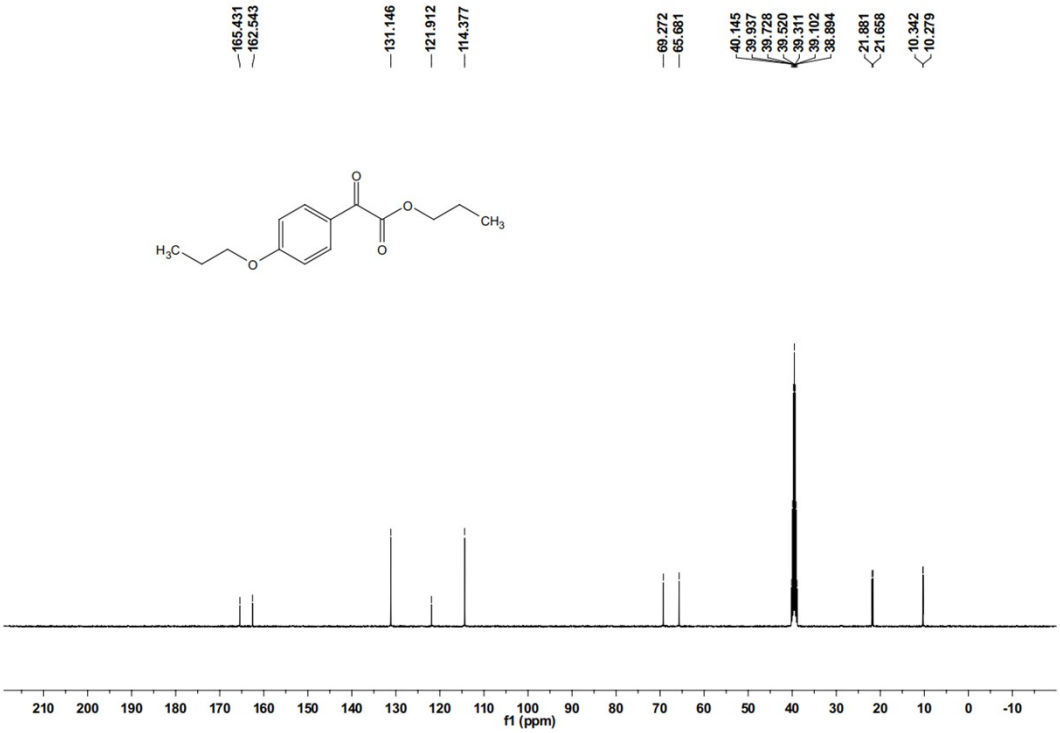


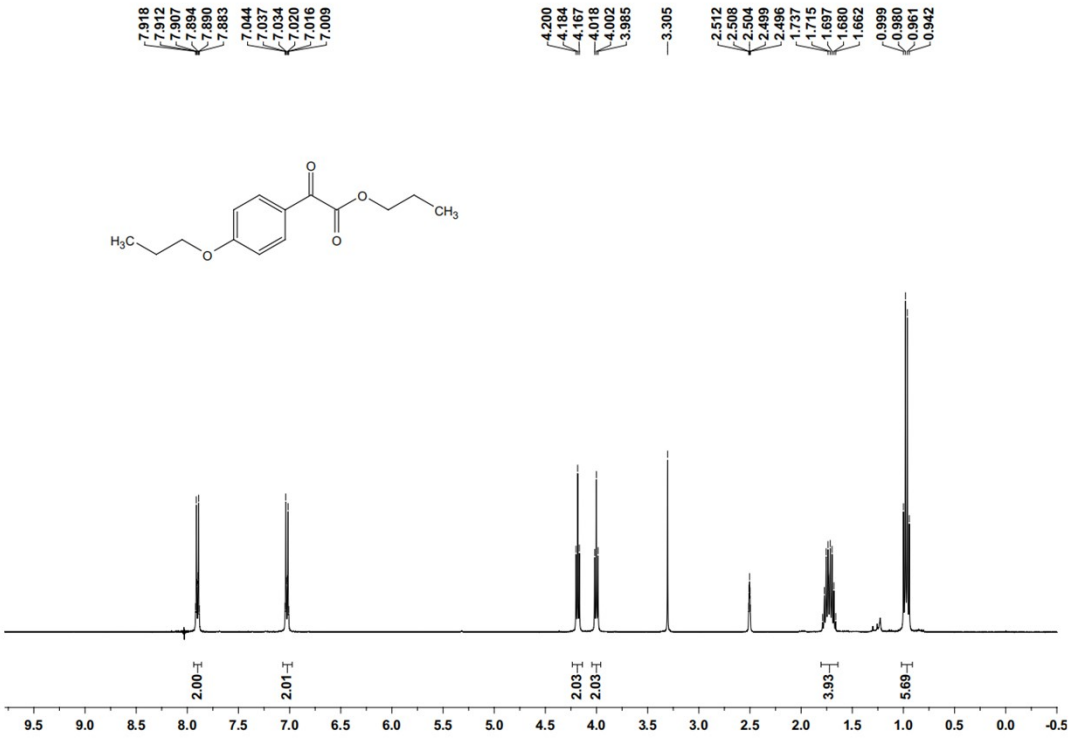
^{1}H and ^{13}C NMR spectra of **3hc**



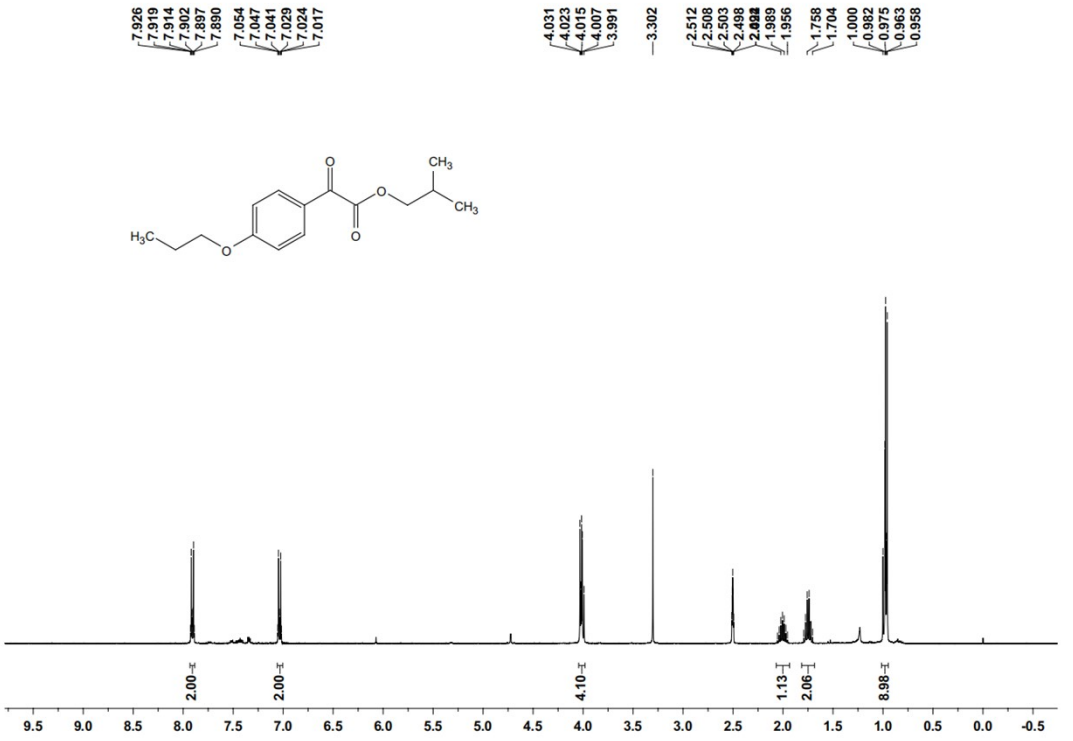


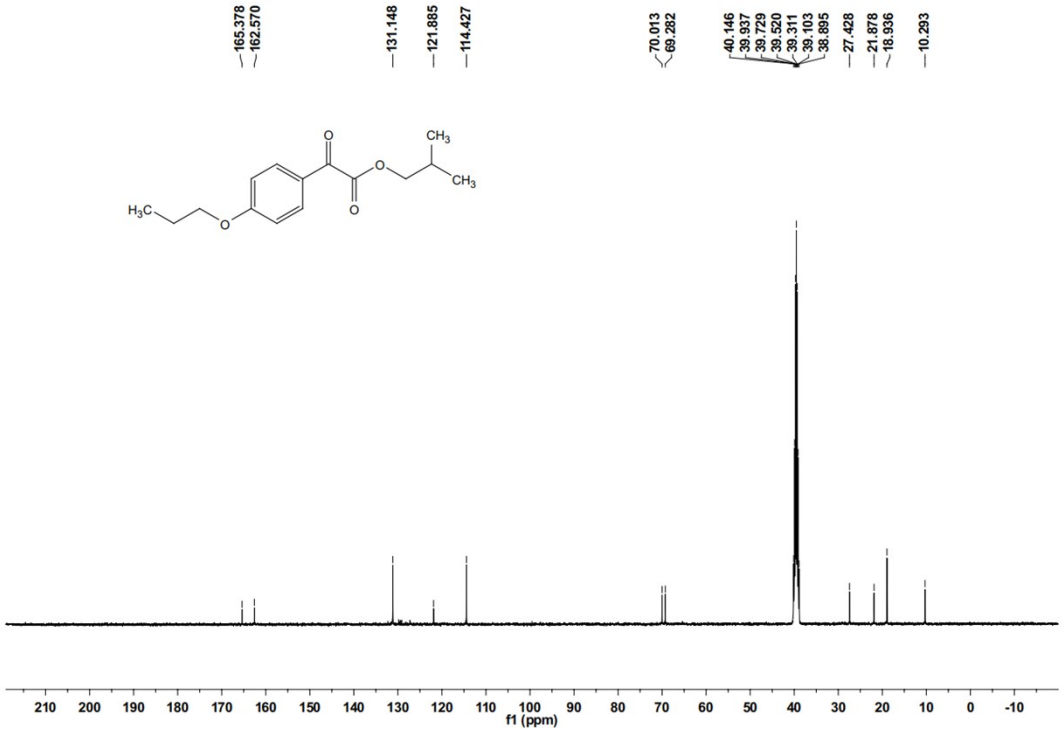
^{1}H and ^{13}C NMR spectra of **3hd**



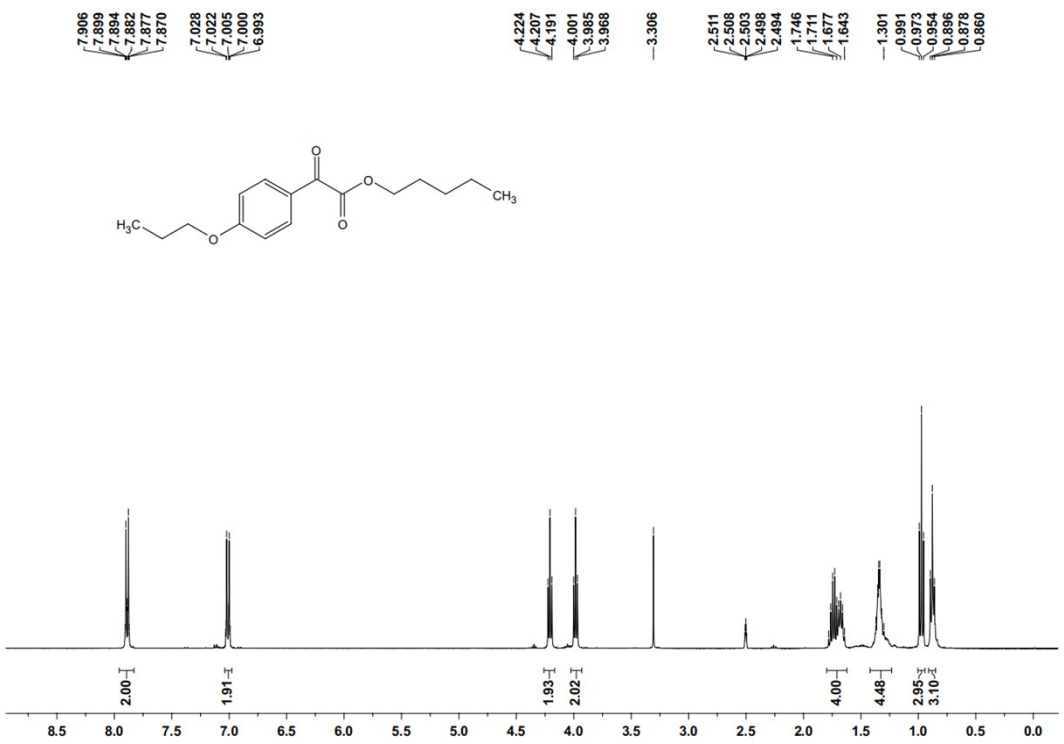


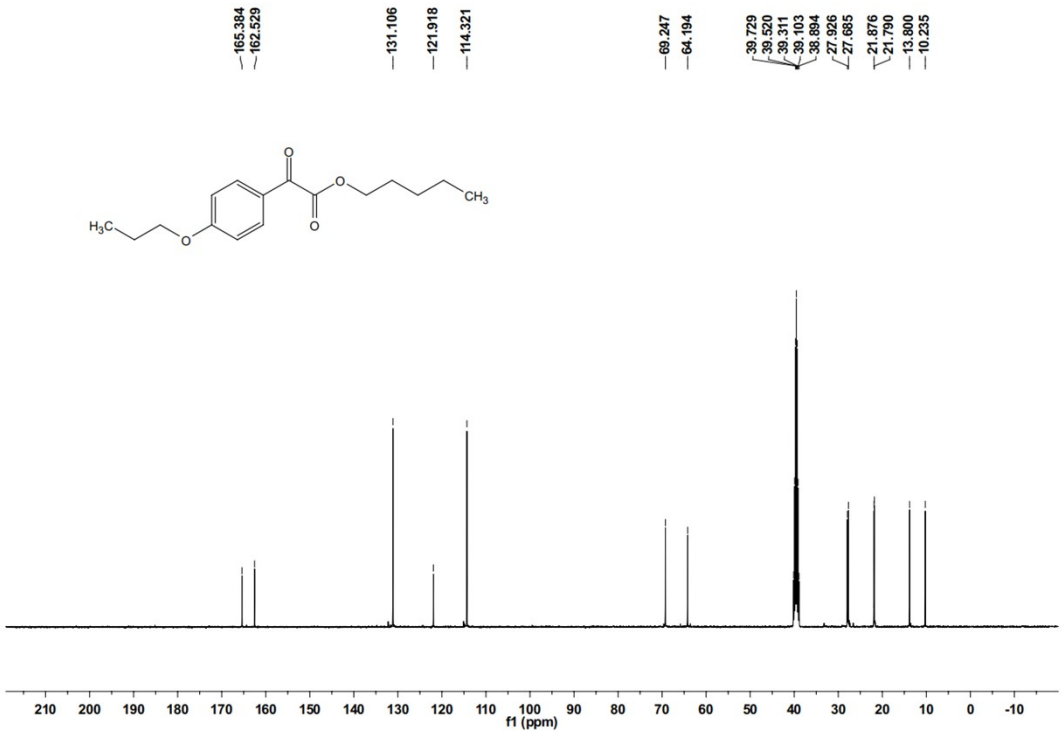
^{1}H and ^{13}C NMR spectra of **3hg**



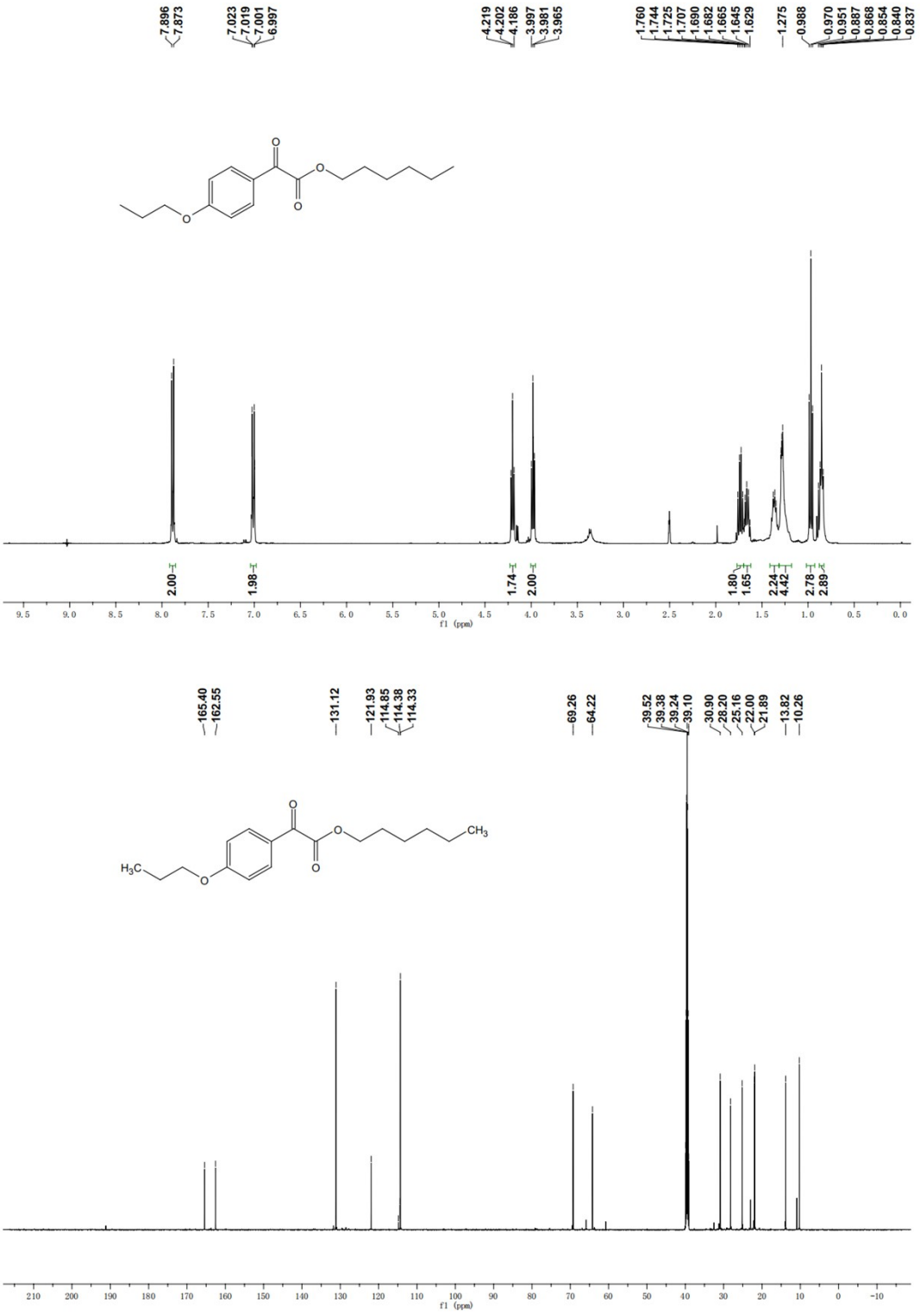


^{1}H and ^{13}C NMR spectra of **3hl**

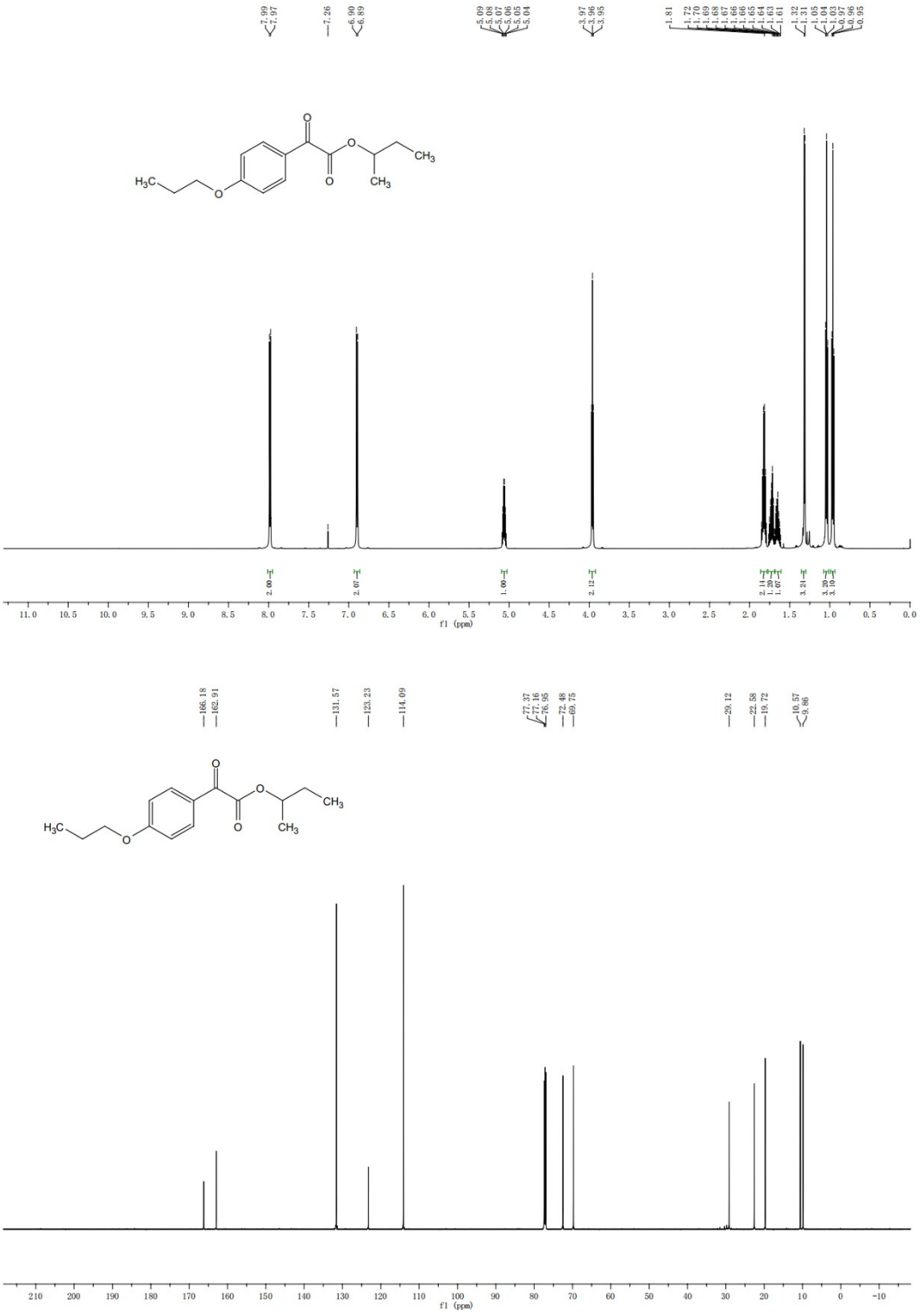




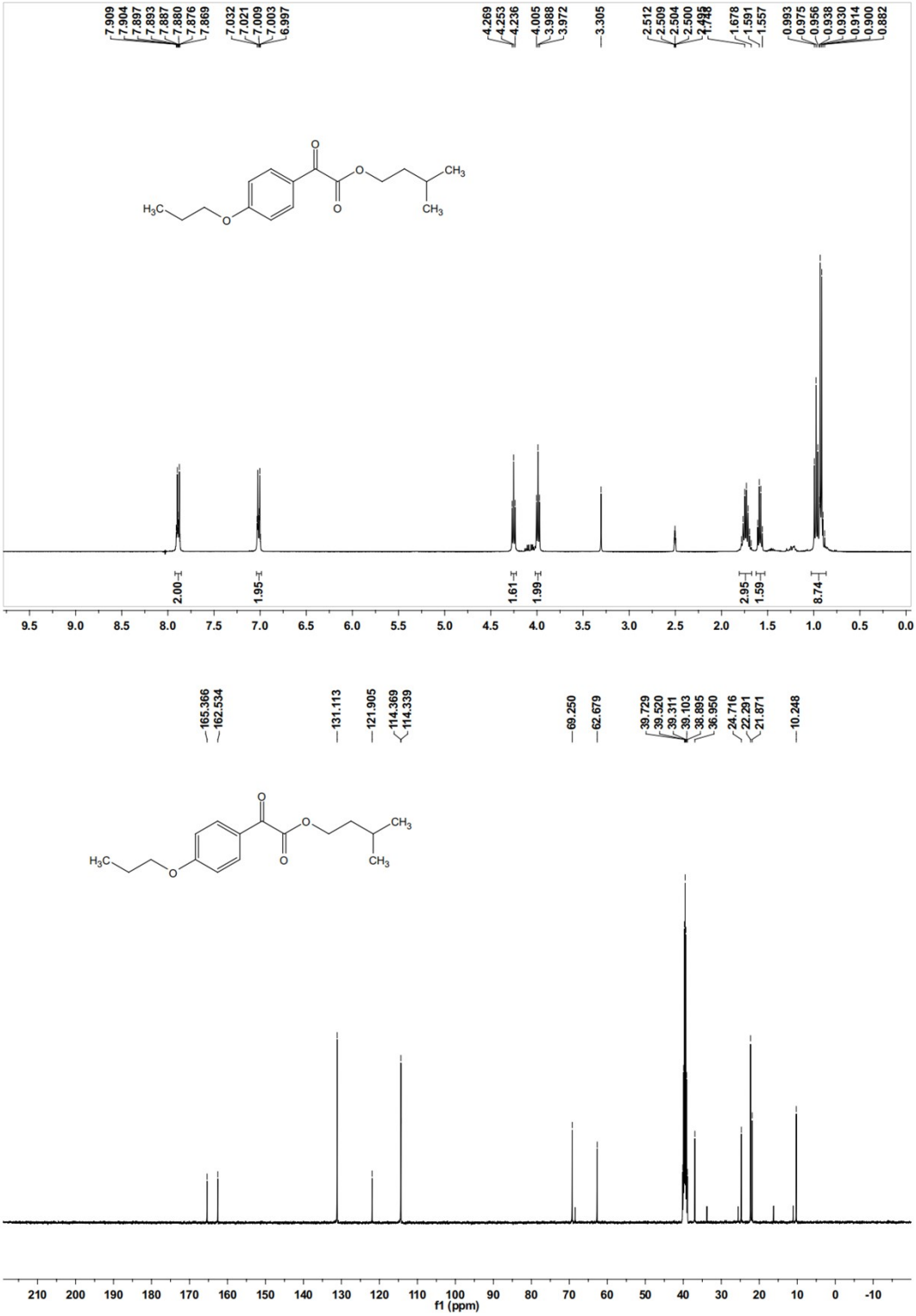
^{1}H and ^{13}C NMR spectra of **3hm**



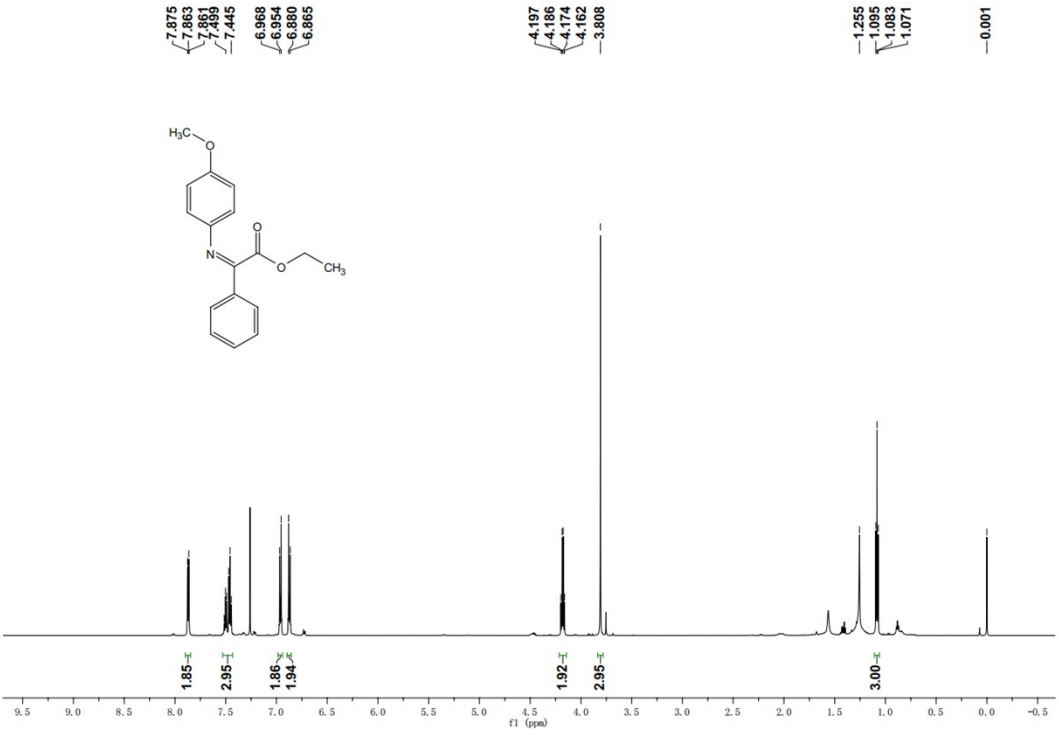
^{1}H and ^{13}C NMR spectra of **3hn**

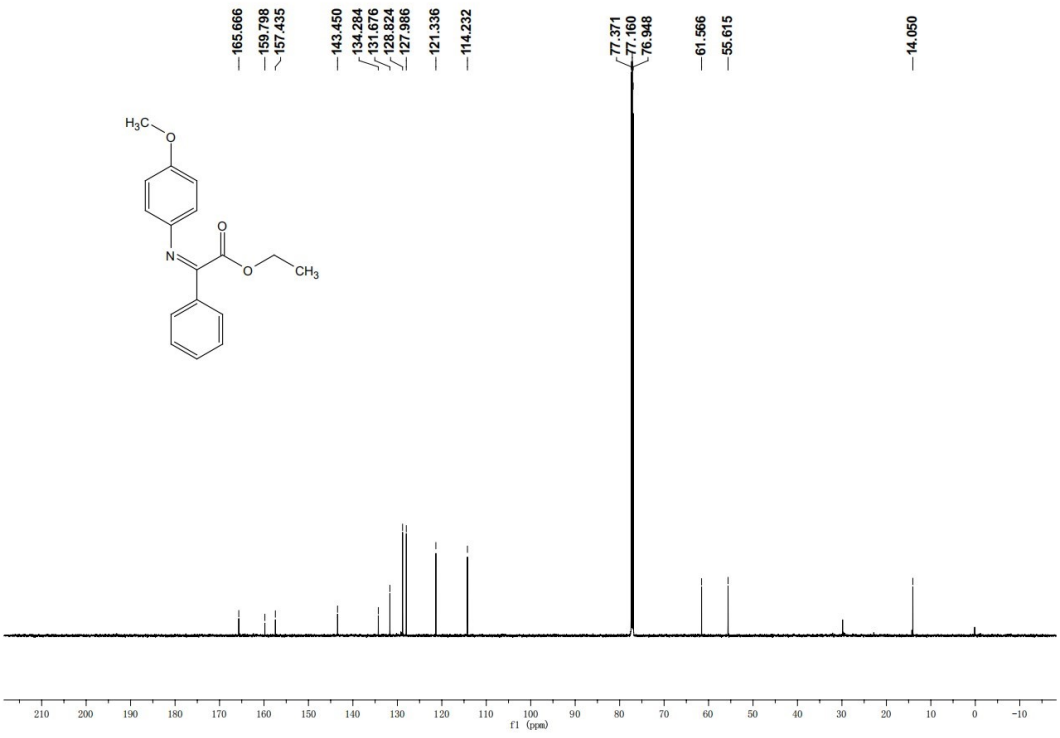


^{1}H and ^{13}C NMR spectra of **3ho**

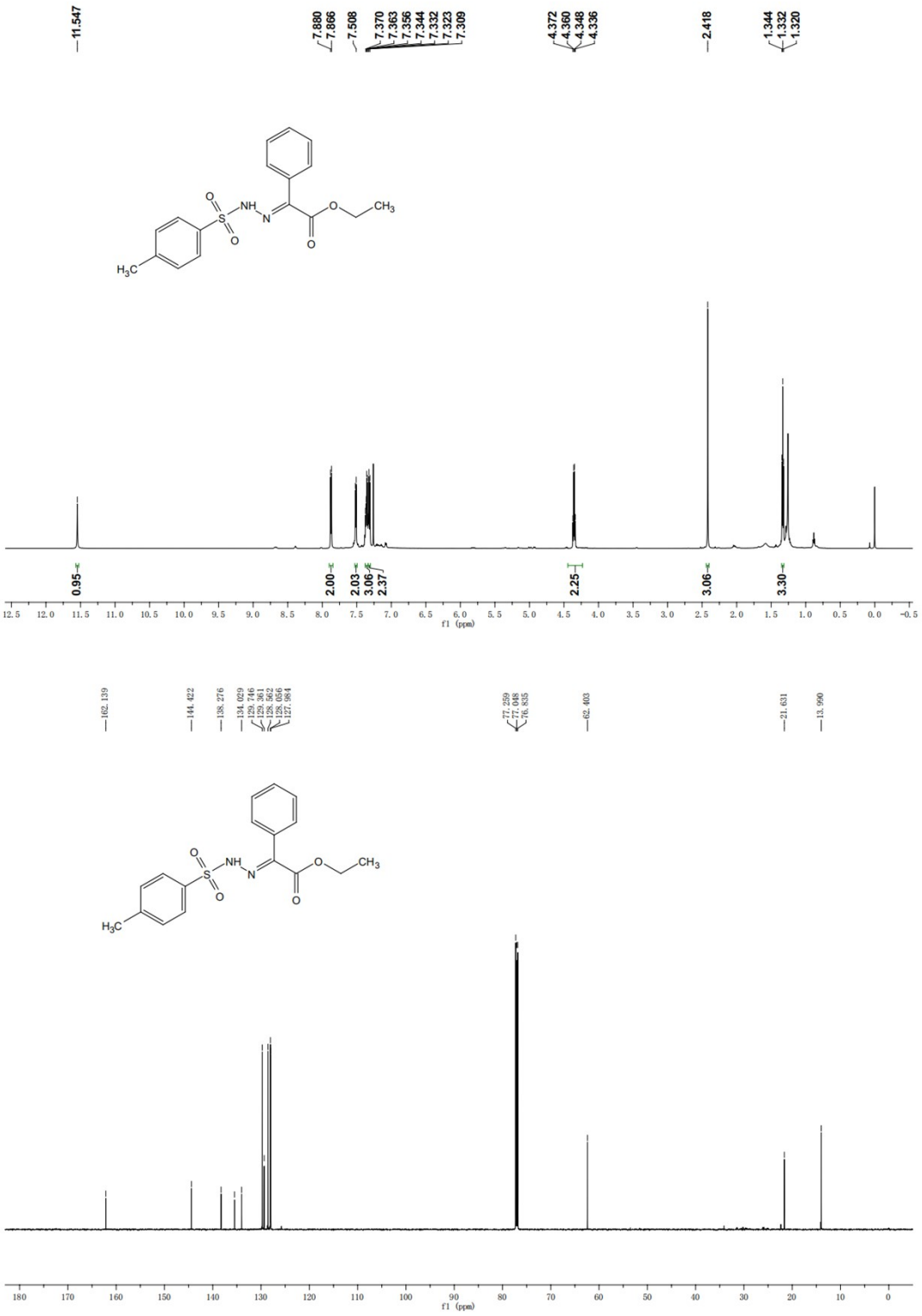


^{1}H and ^{13}C NMR spectra of **5a**

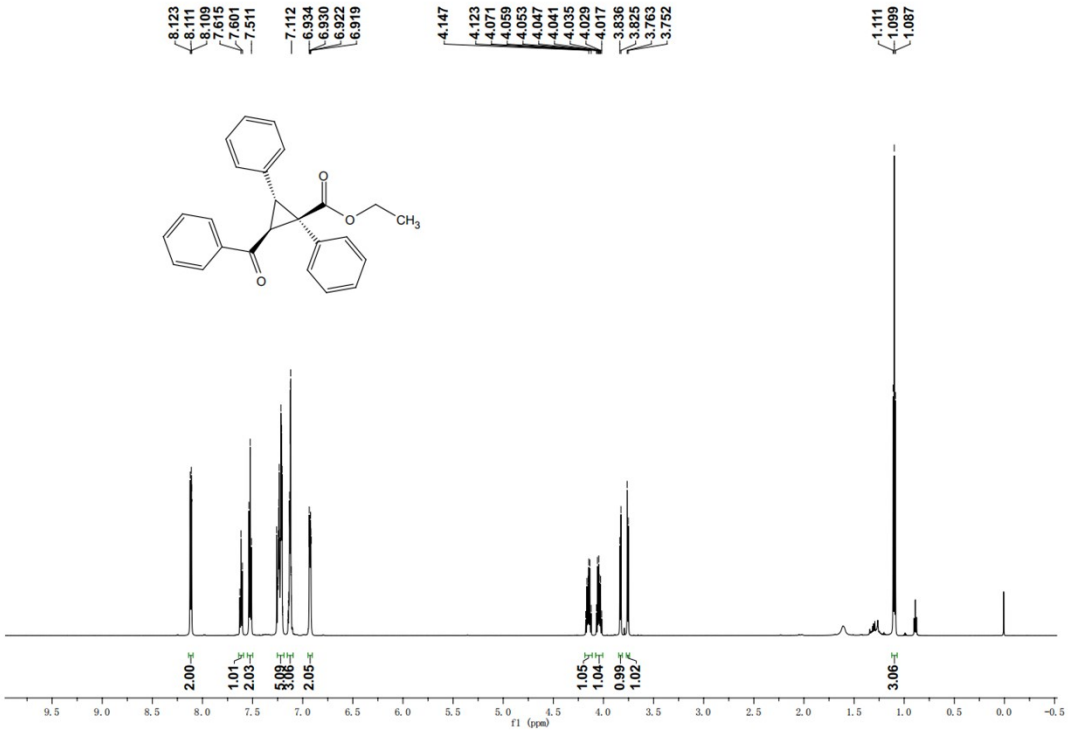




^{1}H and ^{13}C NMR spectra of **5b**



^{1}H and ^{13}C NMR spectra of **5c**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8.109
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7.112
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6.922
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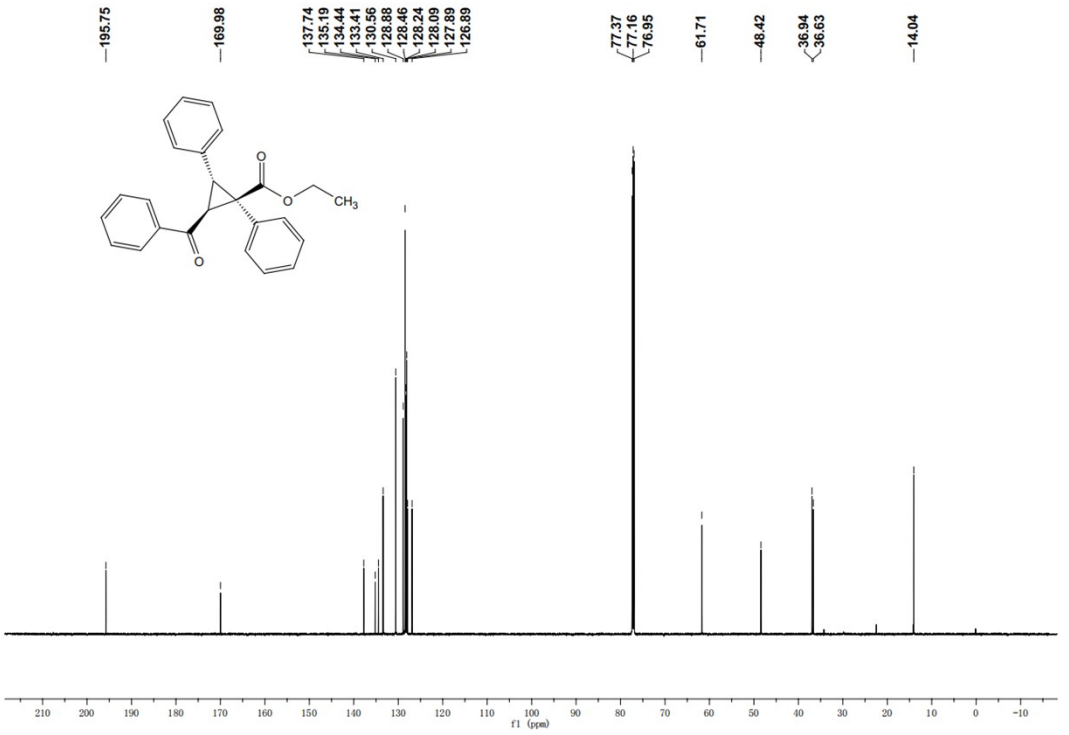
4.123
4.071
4.059
4.053
4.047
4.041
4.035
4.029
4.017
3.836
3.825
3.763
3.752
1.111
1.099
1.087
O
O
O
CH3
2.00
1.01
2.03
5.09
3.08
2.05
1.05
1.04
0.99
1.02
3.06
f1 (ppm)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76.95
61.71
48.42
36.94
36.63
14.04
O
O
O
CH3
f1 (ppm)