Electronic Supplementary Material (ESI) for ChemComm.

Electrochemical Two-electron Oxygen Reduction Reaction (ORR) Induced Aerobic Oxidation of α- Diazoesters

Supporting Information

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1. General Information

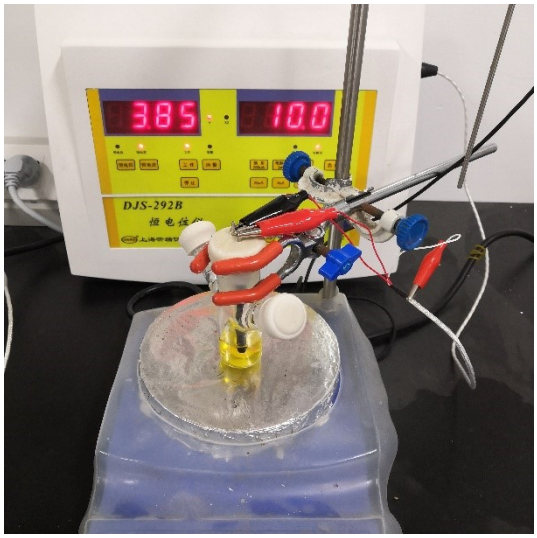
Unless otherwise stated, analytical grade solvents and commercially available reagents were used without further purification. All solvents were analytical reagent or better and were degassed prior to use. The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). The anode electrode is carbon rod electrodes (Φ 6mm) and the cathode electrode is platinum plate electrodes (15 mm×15 mm×0.3 mm). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (boiling point is between 60-90°C). Gradient flash chromatography was conducted eluting with a continuous gradient from petroleum to the indicated solvent, and they are listed as volume/volume ratios. High resolution mass spectra (HRMS) for polypeptides were measured with an Agilent 6224 instrument and accurate masses were reported for the molecular ion + Sodium (M+Na). The ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker Advance III (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. For ^{1}H NMR, chemical shifts (δ) were given in ppm relatives to internal standard (TMS at 0 ppm, $CDCl_3$ at 7.26 ppm). For ^{13}C-NMR, chemical shifts (δ) were reported in ppm using solvent as internal standard ($CDCl_3$ at 77.00 ppm). GC-MS spectra were recorded on a Varian GC-MS 3900-2100T and Shimadzu GCMS-QP2010SE.

1.1 Graphical Guide for the set-up

As experimental set-up, a carbon rod electrode (Φ 6mm), a platinum plate electrode (15 mm×15 mm×0.3 mm), rubber plugs, an undivided three-necked bottle, a dual display potentiostat (DJS-292B) (made in China) and a heating magnetic whisk were used.

a. Assembly of electrode

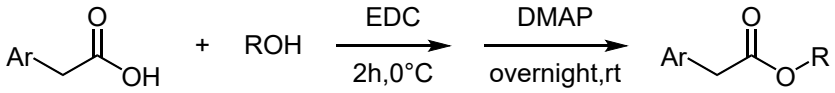
b. Assembly of electrochemical cell



c. Current controlled electrolysis

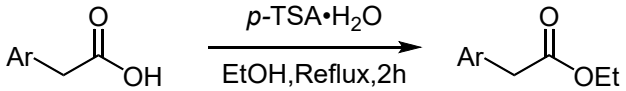
2. General Experimental Procedures

2.1 General Method A - EDC/DCC Ester Synthesis[1]



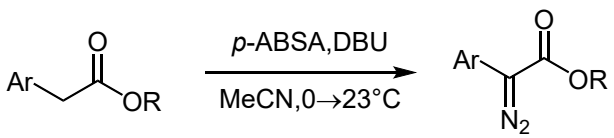
A 100 mL round bottom flask was flame dried and placed under nitrogen. The appropriate carboxylic acid (12 mmol, 1 equiv.) was added followed by anhydrous DCM (25 mL), alcohol (13.2 mmol, 1.1 equiv.), either DCC or EDC (13.2 mmol 1.1 equiv.), and finally DMAP (0.6 mmol, 0.5 equiv.). The reaction was then left to stir under nitrogen overnight and was monitored by TLC. When using DCC, the reaction mixture was filtered through Celite and then added to distilled water and extracted three times with DCM. When using EDC, the reaction mixture was concentrated in vacuo and then the remaining oil was added to distilled water and extracted three times with EtOAc. The rest of the procedure is the same regardless of using DCC or EDC. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. Column chromatography on SiO_2 was performed eluting with 5-10-20% EtOAc/hexanes to provide the product esters.

2.2 General Method B – *p*-TSA Ester Synthesis[2]



A solution of arylphenylacetic acid (12 mmol, 1 equiv.) and *p*-toluenesulfonic acid monohydrate (1.2 mmol, 0.1 equiv.) in ethanol (15 mL) was heated under reflux for 2 h. The solvent was then removed in vacuo. Saturated aq. $NaHCO_3$ (30 mL) was added and the product was extracted with EtOAc (2 x 30 mL). The organic extracts were combined, dried (Na_2SO_4) concentrated in vacuo to afford ester as a colorless oil which was used without further purification.

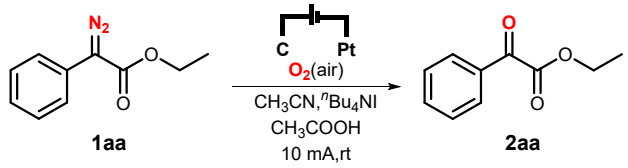
2.3 Diazo Synthesis[3]



To a flame-dried round-bottom flask with a magnetic stir bar were added the aryl acetic acid ester (6.0 mmol, 1 equiv.), *p*-ABSA (7.2 mmol, 1.2 equiv.), and dry acetonitrile (25 mL). The solution was stirred under nitrogen and cooled to 0 °C using an ice water bath. DBU (6.6 mmol, 1.1 equiv.) was added by syringe rapidly in one portion, and the reaction mixture was allowed to warm to 23

°C and stir overnight. Upon completion (as determined by TLC analysis) or after 24 hours, whichever came first, the reaction mixture was quenched with saturated aqueous ammonium chloride (50 mL) and extracted with ether (3 x 50 mL). The combined organic layers were washed with brine (1 x 100 mL), dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude residue was purified by silica gel column chromatography using a mixture of hexanes and ethyl acetate as eluent.

2.4 Ethyl 2-oxo-2-phenylacetate (2aa) Synthesis



In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 1aa (0.2 mmol), CH_3COOH(0.4 mmol) and $^nBu_4NI$ (0.3 mmol) were combined and added. Under the air, CH_3CN (6 mL) were injected into the tubes via syringes. The bottle was equipped with carbon rod (ϕ 6 mm, about 10 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under room temperature for 12 h. After completion of the reaction, as indicated by TLC and GC-MS, the pure product (yield: 80%, 24.28 mg) was obtained by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate= 100:1).

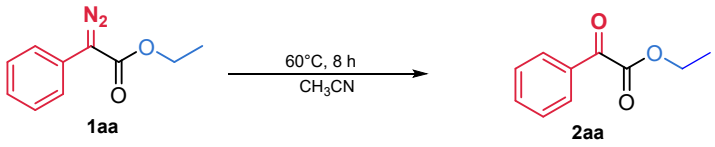
2.5 1 mmol-Scale Experiments

Synthesis of cyclandelate precursor 4: In an oven-dried undivided three-necked bottle (100 mL) equipped with a stir bar, **3** (1 mmol), CH_3COOH (2 mmol) and $^nBu_4NI$ (1.2 mmol) were combined and added. Under the air, CH_3CN (30 mL) were injected into the tubes via syringes. The bottle was equipped with carbon rod (ϕ 6 mm, about 10 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 25 mA under room temperature for 36 h. After completion of the reaction, as indicated by TLC and GC-MS, the pure product (yield: 64%) was obtained by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate= 100:1).

3. Mechanistic experiments.

3.1 H_2O_2 experiments

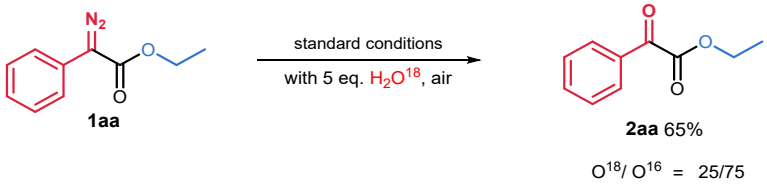
The H_2O_2 experiments in presence of acetic acid were carried out, by employing H_2O_2 and acetic acid instead of electric current to react with **1aa**, in an oven-dried round-bottomed flask (25 mL) equipped with a stir bar, **1aa** (0.2 mmol), CH_3COOH (0.2 mmol) were combined and added. Then H_2O_2 (0.6 mmol), and dry CH_3CN (10 mL) were added successively. The reaction mixture was stirred at 60 °C for 8 hours.31% yield product was produced. Increasing the amount of acetic acid did not improve the yield observably.



With 3 eq. H_2O_2 and 1 eq. CH_3COOH 31%
With 3 eq. H_2O_2 and 3 eq. CH_3COOH 38%

3.2 ^{18}O-labelled experiment with H_2O^{18}

In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, **1aa** (0.2 mmol), CH_3COOH (0.4 mmol) and $^nBu_4NI$ (0.3 mmol) were combined and added. Then H_2O^{18} (1.0 mmol), and dry CH_3CN (6 mL) were added successively. The bottle was equipped with carbon rod (ϕ 6 mm, about 10 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. The reaction mixture was stirred and electrolyzed at constant current under room temperature. GC-MS of the reaction solution showed ~25% ^{18}O-incorporation in the product (Figure S1).



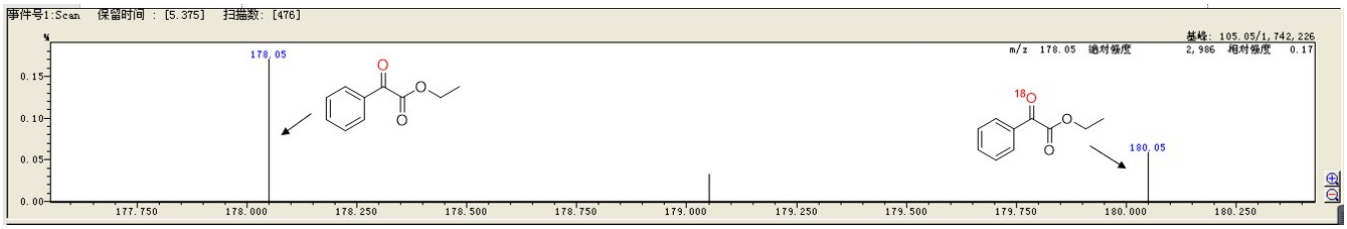


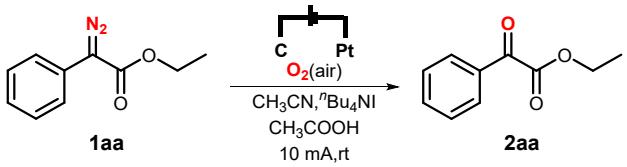
Figure S1. ^{18}O-labelled experiment with H_2O^{18}

3.3 Procedure for electron paramagnetic resonance (EPR) experiments

In an oven-dried undivided three-necked bottle equipped with a stir bar, $^{n}Bu_4NI$ (0.3mmol) were added. The bottle was equipped with carbon rod as the anode and Fe cathode (15 mm x 15 mm x 0.5 mm) as the cathode. Under air conditions , CH_3COOH (0.4 mmol) were injected into the tubes via syringes. The reaction mixture was strong stirred and electrolyzed at a constant current of 10 mA for 15min. When the reaction was finished, the solution sample was taken out into a small tube and analyzed by EPR. After fitting, we proposed that this radical signal belongs to the superoxide radical anion ($g = 2.0065$, $A_N = 13.2$ G, $A_H = 9.5$ G).

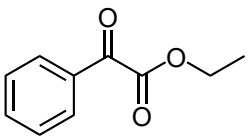
4. Optimization of the reaction conditions

In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 1aa (0.2 mmol), CH_3COOH (0.4 mmol) and $^nBu_4NI$ (0.3 mmol) were combined and added. Under the air, CH_3CN (6 mL) were injected into the tubes via syringes. The bottle was equipped with carbon rod (ϕ 6 mm, about 10 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. The reaction mixture was stirred and electrolyzed at constant current of 10 mA under room temperature for 12 h. When the reaction was finished, the solvent was removed by reduced pressure and the crude product was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate= 100:1). A summary of optimization results is presented in Table S1 below.



entry	Variation from the Standard Conditions	yield (%)
1	$^nBu_4NBF_4$ instead of $^nBu_4NI$	9
2	$^nBu_4NOAc$ instead of $^nBu_4NI$	20
3	$NaBF_4$ instead of $^nBu_4NI$	11
4	KI instead of $^nBu_4NI$	24
5	$^nBu_4NBr$ instead of $^nBu_4NI$	19
6	$LiClO_4$ instead of $^nBu_4NI$	trace
7	H_2O instead of CH_3CN	15
8	Zinc as cathode	19
9	Iron as cathode	9
10	Lead as cathode	17
11	Without electric current	N.D.

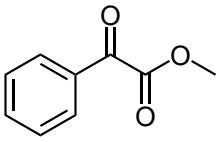
5. Detailed descriptions for products:



Ethyl 2-oxo-2-phenylacetate (2aa): [4]

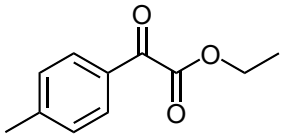
Colorless oil (Yield: 80 %, 24.28 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 8.01 - 7.96 (m, 2H), 7.63 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.8 Hz, 2H), 4.43 (q, J = 7.1 Hz, 2H), 1.40 (t, J = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 186.50 , 163.89 , 134.96 , 132.47 , 130.04 , 128.94 , 62.38 , 14.14 .



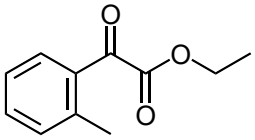
Methyl 2-oxo-2-phenylacetate (2ab):[4]

Colorless oil (Yield: 64 %, 20.99 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 8.07 - 7.93 (m, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 3.98 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.18 , 164.15 , 135.13 , 132.52 , 130.21 , 129.03 , 52.92 .



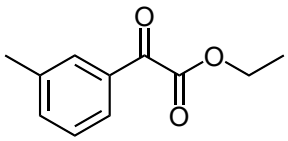
Ethyl 2-oxo-2-(*p*-tolyl)acetate (2ac):[4]

Pale yellow oil (Yield: 84 %, 32.27 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 4.44 (q, *J* = 7.1 Hz, 2H), 2.43 (s, 3H), 1.41 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.22 , 164.16 , 146.35 , 130.27 , 129.74 , 62.34 , 22.02 , 14.23 .



Ethyl 2-oxo-2-(*o*-tolyl)acetate (2ad):[4]

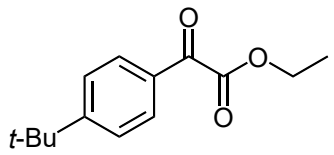
Pale yellow oil (Yield: 63 %, 24.19 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.69 (d, *J* = 7.9 Hz, 1H), 7.52 - 7.47 (m, 1H), 7.32 (t, *J* = 8.0 Hz, 2H), 4.43 (q, *J* = 7.2 Hz, 2H), 2.61 (s, 3H), 1.41 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 188.92 , 164.76 , 141.47 , 133.81 , 132.48 , 132.41 , 131.35 , 126.04 , 62.40 , 21.60 , 14.21 .



Ethyl 2-oxo-2-(*m*-tolyl)acetate (2ae):[4]

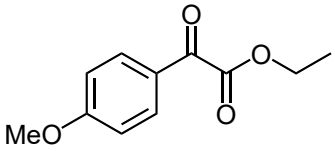
Pale yellow oil (Yield: 80 %, 30.72 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.79 (d, *J* = 7.2

Hz, 2H), 7.46 (d, *J* = 7.3 Hz, 1H), 7.41 - 7.36 (m, 1H), 4.44 (q, *J* = 7.1 Hz, 2H), 2.41 (s, 3H), 1.41 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.79 , 164.11 , 138.94 , 135.86 , 132.54 , 130.38 , 128.87 , 127.45 , 62.36 , 21.36 , 14.20 .



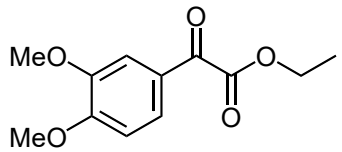
Ethyl 2-(4-(*tert*-butyl)phenyl)-2-oxoacetate (2af):[4]

Pale yellow oil (Yield: 75 %, 35.10 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.96 - 7.93 (m, 2H), 7.54 - 7.51 (m, 2H), 4.44 (q, *J* = 7.1 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H), 1.34 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.23 , 164.17 , 159.18 , 130.16 , 130.05 , 126.05 , 62.34 , 35.51 , 31.09 , 14.26 .



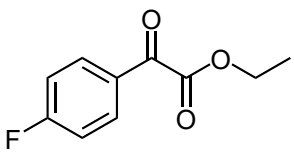
Ethyl 2-(4-methoxyphenyl)-2-oxoacetate (2ag):[5]

Colorless liquid (Yield: 59 %, 24.54 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.99 (d, *J* = 8.9 Hz, 2H), 6.99 - 6.94 (m, 2H), 4.42 (q, *J* = 7.1 Hz, 2H), 3.88 (s, 3H), 1.41 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.00 , 165.13 , 164.28 , 132.69 , 125.64 , 114.35 , 62.28 , 55.76 , 14.23 .



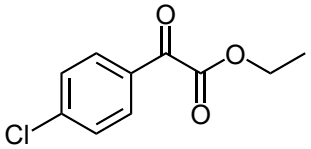
Ethyl 2-(3,4-dimethoxyphenyl)-2-oxoacetate (2ah):[5]

Colorless liquid (Yield: 79 %, 37.60 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.61 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.54 (d, *J* = 2.0 Hz, 1H), 6.91 (d, *J* = 8.5 Hz, 1H), 4.42 (q, *J* = 7.1 Hz, 2H), 3.93 (d, *J* = 11.6 Hz, 6H), 1.40 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.08 , 164.21 , 155.08 , 149.50 , 126.32 , 125.71 , 110.80 , 110.36 , 62.27 , 56.32 , 56.14 , 14.21 .



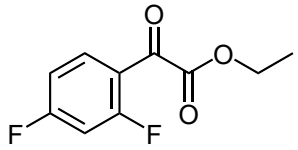
Ethyl 2-(4-fluorophenyl)-2-oxoacetate (2ai):[5]

Colorless liquid (Yield: 79 %, 30.97 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 8.11 - 8.04 (m, 2H), 7.21 - 7.15 (m, 2H), 4.44 (q, *J* = 7.1 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 184.68 , 168.20 , 165.64 , 163.53 , 133.09 (d, *J* = 9.9 Hz), 129.14 (d, *J* = 2.9 Hz), 116.38 (d, *J* = 22.1 Hz), 62.62 , 14.21 . ^{19}F NMR (377 MHz, Chloroform-d) δ -101.26 .



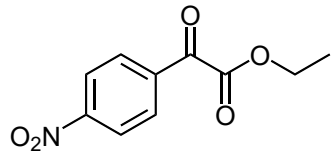
Ethyl 2-(4-chlorophenyl)-2-oxoacetate (2aj):[5]

Colorless liquid (Yield: 60 %, 25.44 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.98 (d, *J* = 8.7 Hz, 2H), 7.49 (d, *J* = 8.7 Hz, 2H), 4.45 (q, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.03 , 163.35 , 141.77 , 131.58 , 131.07 , 129.44 , 62.69 , 14.23 .



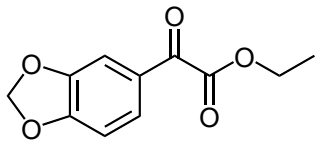
Ethyl 2-(2,4-difluorophenyl)-2-oxoacetate (2ak):

Pale yellow oil (Yield: 76 %, 32.53 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.98 (td, *J* = 8.3, 6.4 Hz, 1H), 7.04 (dddd, *J* = 8.7, 7.7, 2.4, 0.9 Hz, 1H), 6.91 (ddd, *J* = 10.8, 8.6, 2.4 Hz, 1H), 4.43 (q, *J* = 7.2 Hz, 2H), 1.40 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 182.85 , 168.63 (d, *J* = 12.5 Hz), 166.05 (d, *J* = 12.4 Hz), 165.06 (d, *J* = 12.8 Hz), 164.04 , 162.47 (d, *J* = 12.9 Hz), 133.08 (dd, *J* = 11.0, 3.1 Hz), 118.73 - 118.42 (m), 113.06 (dd, *J* = 21.9, 3.5 Hz), 105.10 (t, *J* = 25.5 Hz), 62.81 , 14.05 . ^{19}F NMR (377 MHz, Chloroform-d) δ -97.22 (d, *J* = 13.5 Hz), -106.48 (d, *J* = 13.6 Hz). HRMS (ESI) cald. for $(M+Na)^+$ $C_{10}H_8NaF_2O_3$: 273.0334, found: 273.0332.



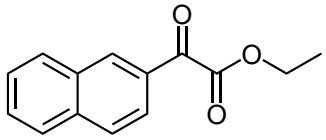
Ethyl 2-(4-nitrophenyl)-2-oxoacetate(2al):[4]

Pale yellow oil (Yield: 48 %, 21.41 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 8.35 (d, *J* = 8.9 Hz, 2H), 8.24 (d, *J* = 8.9 Hz, 2H), 4.48 (q, *J* = 7.1 Hz, 2H), 1.45 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 184.25 , 162.40 , 151.26 , 137.16 , 131.36 , 124.07 , 63.16 , 14.19 .



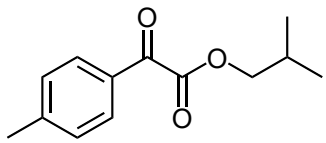
Ethyl 2-(benzo[*d*][1,3]dioxol-5-yl)-2-oxoacetate(2am):[6]

Light yellow oil (Yield: 76 %, 32.53 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.61 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.47 (d, *J* = 1.7 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.08 (s, 2H), 4.42 (q, *J* = 7.1 Hz, 2H), 1.41 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 184.70 , 164.13 , 153.66 , 148.64 , 128.01 , 127.37 , 108.87 , 108.45 , 102.39 , 62.42 , 14.25 .



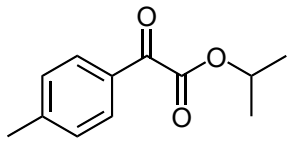
Ethyl 2-(naphthalen-2-yl)-2-oxoacetate(2an):[5]

Colorless liquid (Yield: 68 %, 31.01 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 8.55 (s, 1H), 8.05 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.90 (dd, *J* = 15.9, 8.4 Hz, 2H), 7.65 (ddd, *J* = 8.2, 7.0, 1.3 Hz, 1H), 7.57 (ddd, *J* = 8.1, 7.0, 1.2 Hz, 1H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.45 , 164.05 , 136.47 , 133.61 , 132.39 , 130.11 , 129.93 , 129.68 , 129.06 , 128.04 , 127.27 , 124.09 , 62.53 , 14.26 .



Isobutyl 2-oxo-2-(*p*-tolyl)acetate(2ao):

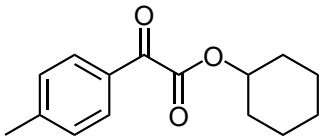
Light yellow oil (Yield: 67 %, 29.48 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.89 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.16 (d, *J* = 6.7 Hz, 2H), 2.43 (s, 3H), 2.08 (dp, *J* = 13.4, 6.7 Hz, 1H), 0.99 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.35 , 164.41 , 146.35 , 130.23 , 129.76 , 72.11 , 27.85 , 22.03 , 19.11 . HRMS (ESI) cald. for $(M+Na)^{+}$ $C_{13}H_{16}NaO_3$: 243.0992, found: 243.0995.



Isopropyl 2-oxo-2-(*p*-tolyl)acetate(2ap):[7]

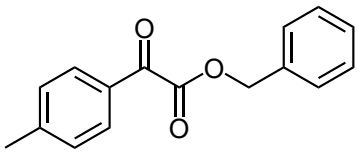
Light yellow oil (Yield: 93 %, 38.32 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.88 (d, *J* = 8.3

Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 5.31 (p, *J* = 6.3 Hz, 1H), 2.43 (s, 3H), 1.40 (d, *J* = 6.3 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.52 , 163.95 , 146.23 , 130.18 , 129.72 , 70.62 , 22.01 , 21.84 .



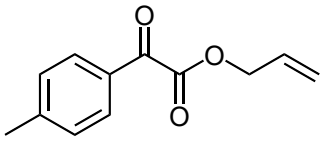
Cyclohexyl 2-oxo-2-(*p*-tolyl)acetate(2aq):[8]

Light yellow oil (Yield: 63 %, 30.99 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.88 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 5.07 (td, *J* = 9.2, 4.6 Hz, 1H), 2.43 (s, 3H), 2.00 (dt, *J* = 12.8, 4.1 Hz, 2H), 1.78 (dq, *J* = 13.2, 4.4 Hz, 2H), 1.64 - 1.53 (m, 3H), 1.46 - 1.37 (m, 2H), 1.29 (dddd, *J* = 15.4, 8.8, 6.1, 2.5 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.61 , 163.98 , 146.20 , 130.19 , 130.16 , 129.71 , 75.38 , 31.53 , 25.27 , 23.73 , 22.00 .



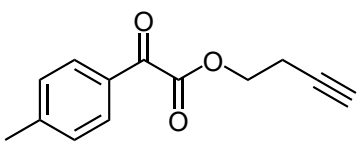
Benzyl 2-oxo-2-(*p*-tolyl)acetate(2ar):[9]

Colorless liquid; (Yield: 44 %, 22.35 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.48 - 7.36 (m, 5H), 7.28 (d, *J* = 8.0 Hz, 2H), 5.41 (s, 2H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.81 , 163.94 , 146.40 , 134.69 , 130.22 , 130.04 , 129.72 , 128.84 , 128.80 , 128.66 , 67.72 , 21.97 .



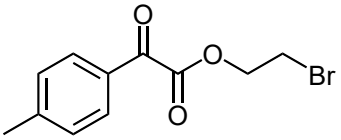
Allyl 2-oxo-2-(*p*-tolyl)acetate(2as):[10]

Light yellow oil (Yield: 62 %, 25.30 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.92 - 7.87 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 6.01 (ddt, *J* = 17.0, 10.4, 5.9 Hz, 1H), 5.47 - 5.31 (m, 2H), 4.86 (dt, *J* = 5.9, 1.3 Hz, 2H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.87 , 163.77 , 146.47 , 130.96 , 130.31 , 130.09 , 129.78 , 120.06 , 66.63 , 22.06 .



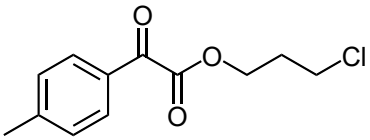
But-3-yn-1-yl 2-oxo-2-(*p*-tolyl)acetate(2at):

Light yellow oil (Yield: 70 %, 30.24 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 4.48 (t, *J* = 6.8 Hz, 2H), 2.68 (td, *J* = 6.8, 2.7 Hz, 2H), 2.43 (s, 3H), 2.05 (t, *J* = 2.7 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.67 , 163.73 , 146.52 , 130.34 , 129.74 , 79.39 , 70.68 , 63.61 , 22.02 , 19.01 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{13}H_{12}NaO_3$: 239.0679, found: 239.0673.



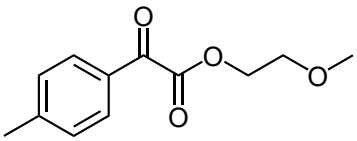
2-bromoethyl 2-oxo-2-(*p*-tolyl)acetate(2au):

Light yellow oil (Yield: 41 %, 22.06 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 4.69 (t, *J* = 6.2 Hz, 2H), 3.64 (t, *J* = 6.2 Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.42 , 163.46 , 146.71 , 130.41 , 129.85 , 65.16 , 27.83 , 22.10 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{11}H_{11}NaBrO_3$: 292.9784, found: 292.9782.



3-chloropropyl 2-oxo-2-(*p*-tolyl)acetate(2av):

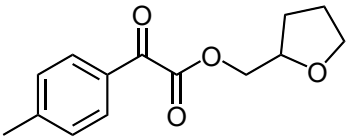
Light yellow oil (Yield: 60 %, 28.80 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 4.53 (t, *J* = 6.1 Hz, 2H), 3.66 (t, *J* = 6.3 Hz, 2H), 2.44 (s, 3H), 2.23 (p, *J* = 6.2 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.80 , 163.93 , 146.57 , 130.25 , 130.01 , 129.81 , 62.72 , 40.92 , 31.32 , 22.04 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{12}H_{13}NaClO_3$: 263.0445, found: 263.0440.



2-methoxyethyl 2-oxo-2-(*p*-tolyl)acetate(2aw):

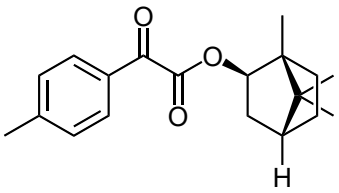
Light yellow oil (Yield: 87 %, 38.63 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 4.53 - 4.50 (m, 2H), 3.72 - 3.69 (m, 2H), 3.40 (s, 3H), 2.42 (s,

3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.92 , 164.09 , 146.39 , 130.30 , 130.03 , 129.71 , 70.01 , 64.82 , 59.10 , 22.00 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{12}H_{14}NaO_4$: 245.0784, found: 245.0780.



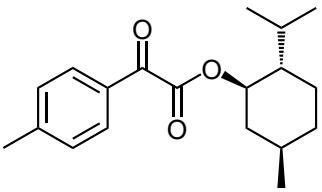
(tetrahydrofuran-2-yl)methyl 2-oxo-2-(*p*-tolyl)acetate(2ba):

Light yellow oil (Yield: 93 %, 46.13 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 4.44 - 4.31 (m, 2H), 4.23 (qd, *J* = 6.8, 3.9 Hz, 1H), 3.92 - 3.77 (m, 2H), 2.42 (s, 3H), 2.09 - 1.87 (m, 3H), 1.75 - 1.65 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.98 , 164.10 , 146.36 , 130.29 , 130.05 , 129.72 , 76.15 , 68.61 , 67.52 , 28.05 , 25.79 , 22.00 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{14}H_{16}NaO_4$: 271.0941, found: 271.0943.



(1*R*,2*R*,4*R*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 2-oxo-2-(*p*-tolyl)acetate(2bb):

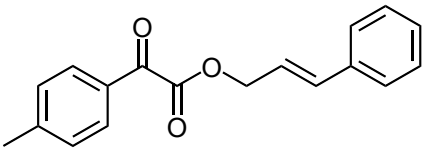
Light yellow oil (Yield: 88%, 52.80 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.87 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.00 - 4.95 (m, 1H), 2.42 (s, 3H), 1.93 (dd, *J* = 5.6, 2.1 Hz, 2H), 1.80 - 1.72 (m, 2H), 1.62 (td, *J* = 12.1, 11.7, 3.8 Hz, 1H), 1.25 - 1.19 (m, 1H), 1.16 - 1.10 (m, 1H), 0.94 (d, *J* = 8.3 Hz, 6H), 0.84 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.51 , 164.15 , 146.17 , 130.11 , 129.70 , 129.20 , 83.33 , 49.21 , 47.12 , 45.15 , 38.69 , 33.76 , 27.07 , 21.99 , 20.12 , 19.93 , 11.62 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{19}H_{24}NaO_3$: 323.1618, found: 323.1615.



(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl 2-oxo-2-(*p*-tolyl)acetate(2bc):

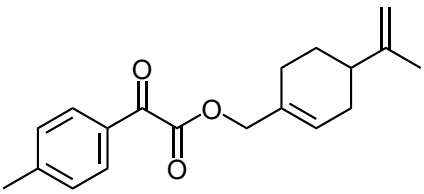
Light yellow oil (Yield: 93%, 56.21 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.87 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 4.99 (td, *J* = 11.0, 4.4 Hz, 1H), 2.43 (s, 3H), 2.21 - 2.13 (m, 1H), 1.95 (ddt, *J* = 13.9, 7.0, 3.5 Hz, 1H), 1.72 (dt, *J* = 15.0, 3.1 Hz, 2H), 1.54 (dddd, *J* = 26.6,

14.5, 6.3, 3.2 Hz, 2H), 1.26 - 1.04 (m, 2H), 0.97 - 0.82 (m, 10H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.61 , 164.21 , 146.21 , 130.13 , 129.74 , 46.90 , 40.72 , 34.16 , 31.63 , 26.23 , 23.41 , 22.07 , 22.00 , 20.78 , 16.24 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{19}H_{26}NaO_3$: 325.1774, found: 325.1774.



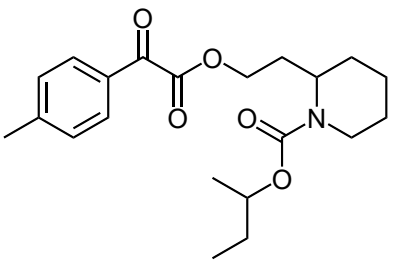
Cinnamyl 2-oxo-2-(*p*-tolyl)acetate(2bd):

Light yellow oil (Yield: 50%, 28.32 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.42 (dd, *J* = 8.2, 1.1 Hz, 2H), 7.37 - 7.28 (m, 5H), 6.78 (d, *J* = 15.9 Hz, 1H), 6.38 (dt, *J* = 15.9, 6.6 Hz, 1H), 5.03 (dd, *J* = 6.6, 1.2 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 185.88 , 163.87 , 146.46 , 135.98 , 130.34 , 130.12 , 129.79 , 128.79 , 128.53 , 126.89 , 121.72 , 66.72 , 22.06 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{18}H_{16}NaO_3$: 303.0992, found: 303.0999.



(4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl 2-oxo-2-(*p*-tolyl)acetate(2be):

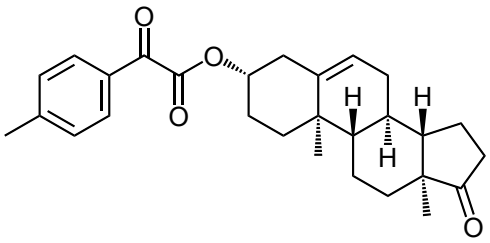
Light yellow oil (Yield: 53%, 31.59 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.91 - 7.87 (m, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 5.88 (d, *J* = 5.1 Hz, 1H), 4.77 - 4.71 (m, 4H), 2.44 (s, 3H), 2.19 - 2.12 (m, 4H), 1.87 (ddt, *J* = 12.2, 4.2, 2.3 Hz, 1H), 1.73 (s, 3H), 1.67 (d, *J* = 14.2 Hz, 1H), 1.50 (ddt, *J* = 13.9, 8.4, 2.5 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.15 , 164.20 , 149.48 , 146.37 , 131.67 , 130.27 , 130.17 , 129.76 , 127.82 , 122.57 , 109.03 , 70.09 , 67.38 , 41.24 , 40.74 , 30.59 , 27.57 , 27.32 , 26.49 , 26.22 , 22.05 , 20.91 , 20.88 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{19}H_{22}NaO_3$: 321.1461, found: 321.1466.



Sec-butyl 2-(2-(2-oxo-2-(*p*-tolyl)acetoxy)ethyl)piperidine-1-carboxylate(2bf):

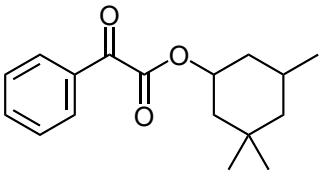
Light yellow oil (Yield: 53%, 39.75 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.91 (d, *J* = 8.1

Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 4.73 (ddt, *J* = 9.0, 6.3, 2.7 Hz, 1H), 4.50 - 4.30 (m, 3H), 4.10 - 4.02 (m, 1H), 2.83 (t, *J* = 13.0 Hz, 1H), 2.43 (s, 3H), 2.24 (dtd, *J* = 12.7, 10.0, 6.4 Hz, 1H), 1.92 - 1.81 (m, 1H), 1.69 - 1.46 (m, 8H), 1.18 (d, *J* = 6.2 Hz, 3H), 0.87 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.04 , 164.07 , 155.67 , 146.36 , 130.37 , 130.12 , 129.73 , 129.31 (d, *J* = 8.5 Hz), 73.24 (d, *J* = 4.6 Hz), 63.92 , 47.79 , 39.03 (d, *J* = 9.3 Hz), 29.81 , 29.17 – 29.14 (m), 28.87 (d, *J* = 5.2 Hz), 25.57 (d, *J* = 4.5 Hz), 22.04 , 19.86 , 19.18 , 9.84 (d, *J* = 4.6 Hz). HRMS (ESI) cald. for $(M+Na)^+$ $C_{21}H_{29}NaNO_5$: 398.1938, found: 398.1938.



(3*S*,8*R*,9*S*,10*R*,13*S*,14*S*)-10,13-dimethyl-17-oxo-2,3,4,7,8,9,10,11,12,13,14,15,16,17 - tetradecahydro-1*H*-cyclopenta[a]phenanthren-3-yl 2-oxo-2-(*p*-tolyl)acetate(2bg):

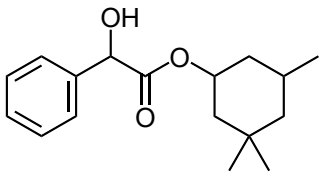
White solid (Yield: 70%, 60.67 mg). ^{1}H NMR (400 MHz, Chloroform-d) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.47 (d, *J* = 4.8 Hz, 1H), 4.92 (tdd, *J* = 11.4, 7.1, 4.6 Hz, 1H), 2.50 (d, *J* = 8.1 Hz, 2H), 2.43 (s, 3H), 2.16 - 2.01 (m, 3H), 1.94 (dt, *J* = 13.2, 3.4 Hz, 2H), 1.89 - 1.77 (m, 2H), 1.70 (ddd, *J* = 16.1, 11.2, 7.1 Hz, 4H), 1.62 - 1.43 (m, 3H), 1.35 - 1.14 (m, 4H), 1.04 (d, *J* = 11.8 Hz, 4H), 0.88 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 221.07 , 186.35 , 163.75 , 146.29 , 139.46 , 130.20 (d, *J* = 6.2 Hz), 129.75 , 122.67 , 77.48 , 51.79 , 50.22 , 47.63 , 37.94 , 36.92 (d, *J* = 13.5 Hz), 35.94 , 31.53 (d, *J* = 5.3 Hz), 30.90 , 27.68 , 22.01 (d, *J* = 5.3 Hz), 20.45 , 19.42 , 13.66 . HRMS (ESI) cald. for $(M+Na)^+$ $C_{28}H_{34}NaO_4$: 457.2349, found: 457.2344. MP：227.1℃.



3,3,5-trimethylcyclohexyl 2-oxo-2-phenylacetate(4):[11]

Colorless liquid (Yield: 75%, 41.12 mg).^{1}H NMR (400 MHz, Chloroform-d) δ 7.99 (ddd, *J* = 7.0, 3.9, 1.8 Hz, 2H), 7.66 - 7.61 (m, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 5.43 (p, *J* = 3.0 Hz, 0.8H), 5.21 (tt, *J* = 11.6, 4.5 Hz, 0.2H), 2.17 - 2.10 (m, 0.25H), 2.01 - 1.80 (m, 2.75H), 1.46 (q, *J* = 2.2 Hz, 0.4H), 1.42 (dt, *J* = 8.5, 2.9 Hz, 0.9H), 1.37 (d, *J* = 3.6 Hz, 0.4H), 1.34 - 1.24 (m, 0.5H), 1.15 (ddd, *J* =

14.4, 12.0, 3.0 Hz, 0.8H), 0.99 (s, 3H), 0.94 (d, *J* = 6.5 Hz, 0.9H), 0.92 - 0.88 (m, 4.8H), 0.87 - 0.82 (m, 1.5H). ^{13}C NMR (101 MHz, Chloroform-d) δ 186.78 (d, *J* = 9.4 Hz), 163.74 (d, *J* = 11.4 Hz), 134.86 , 132.59 (d, *J* = 5.1 Hz), 130.03 , 129.39 , 128.95 , 128.55 , 74.07 (d, *J* = 6.9 Hz), 47.97 , 47.40 , 43.78 , 41.39 , 40.18 , 38.32 , 33.92 , 33.02 , 32.54 , 30.72 , 27.35 (d, *J* = 18.3 Hz), 25.57 , 23.38 , 22.37 (d, *J* = 13.3 Hz).



Cyclandelate(5):[12]

Prepared from 3,3,5-trimethylcyclohexyl 2-oxo-2-phenylacetate(2bf, 0.2mmol) according to general procedure.[13] The crude residue was purified by short flash column chromatography to yield 7q as a mixture of R/S (33.14 mg, 60 %).^{1}H NMR (400 MHz, Chloroform-d) δ 7.43 - .28 (m, 5H), 5.16 (h, *J* = 3.2 Hz, 0.8H), 5.10 (s, 1H), 4.95 (ttd, *J* = 11.6, 4.4, 2.8 Hz, 0.2H), 3.56 (d, *J* = 47.4 Hz, 1H), 1.88 - 1.65 (m, 1.8H), 1.56 (ddt, *J* = 14.3, 5.2, 2.7 Hz, 0.4H), 1.45 (dq, *J* = 15.2, 2.2 Hz, 0.6H), 1.32 (ddd, *J* = 13.2, 7.8, 3.3 Hz, 1.6H), 1.27 - 1.16 (m, 0.8H), 1.08 - 0.99 (m, 0.6H), 0.95 - 0.83 (m, 6H), 0.75 (d, *J* = 5.2 Hz, 2H), 0.71 (d, *J* = 6.4 Hz, 1H), 0.46 (s, 1.3H). ^{13}C NMR (101 MHz, Chloroform-d) δ 173.38 (d, *J* = 11.6 Hz), 128.71 – 128.28 (m), 126.96 , 126.80 – 126.43 (m), 73.98 – 73.56 (m), 73.30 (d, *J* = 4.3 Hz), 47.96 , 41.29 (d, *J* = 15.9 Hz), 38.43 , 33.89 (d, *J* = 17.2 Hz), 30.32 , 27.44 – 27.00 (m), 26.71 , 23.13 , 22.59 – 22.16 (m). HRMS (ESI) cald. for $(M+Na)^+$ $C_{21}H_{29}NaNO_5$: 299.1618, found: 299.1618.The ^{1}H NMR and ^{13}C NMR data of the mixture compound 3bf are consistent with that of known cyclandelate which was purchased from Adamas.

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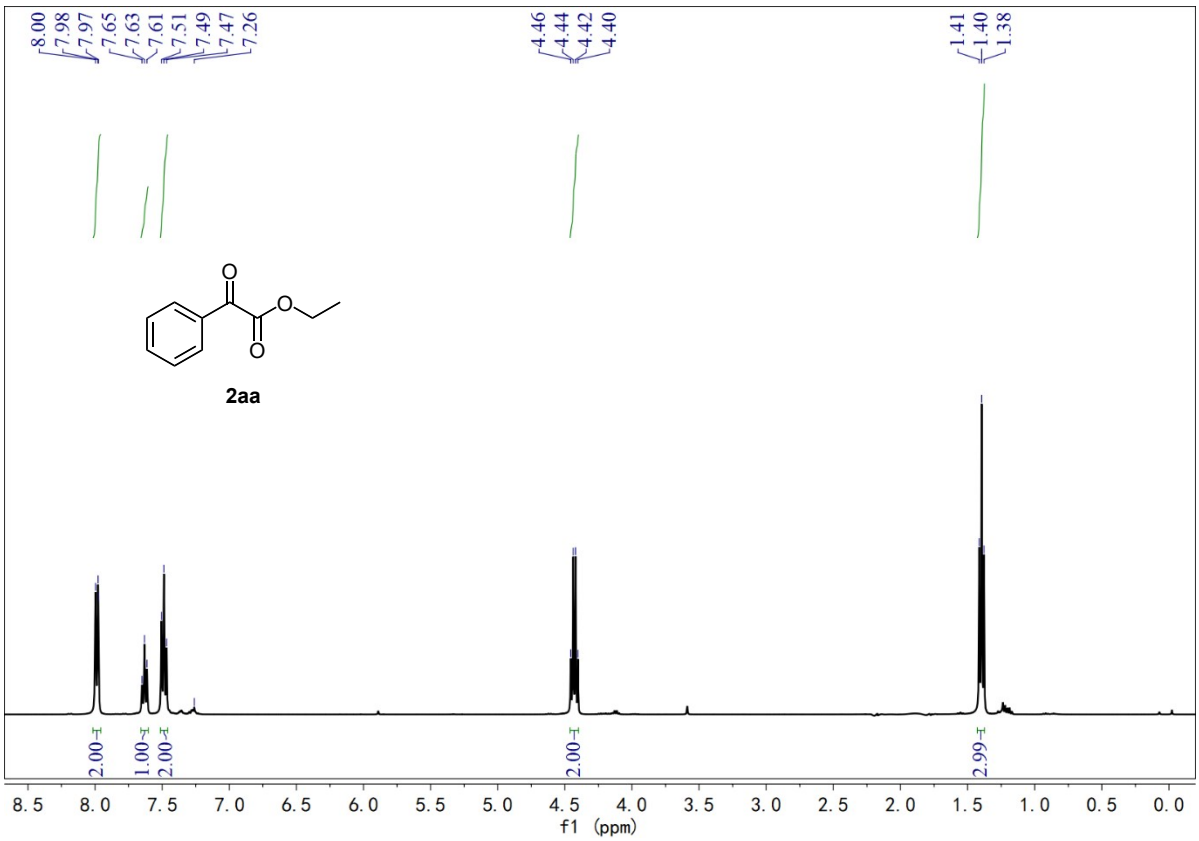
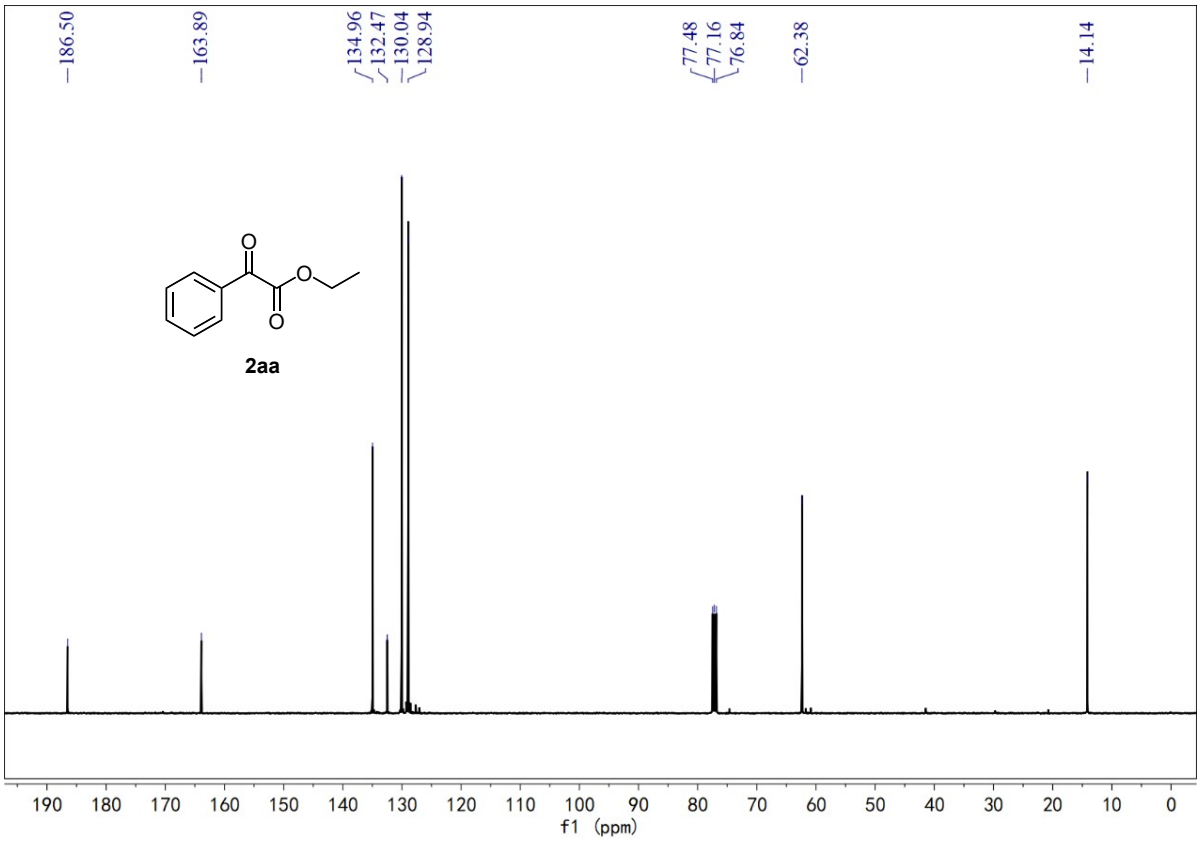
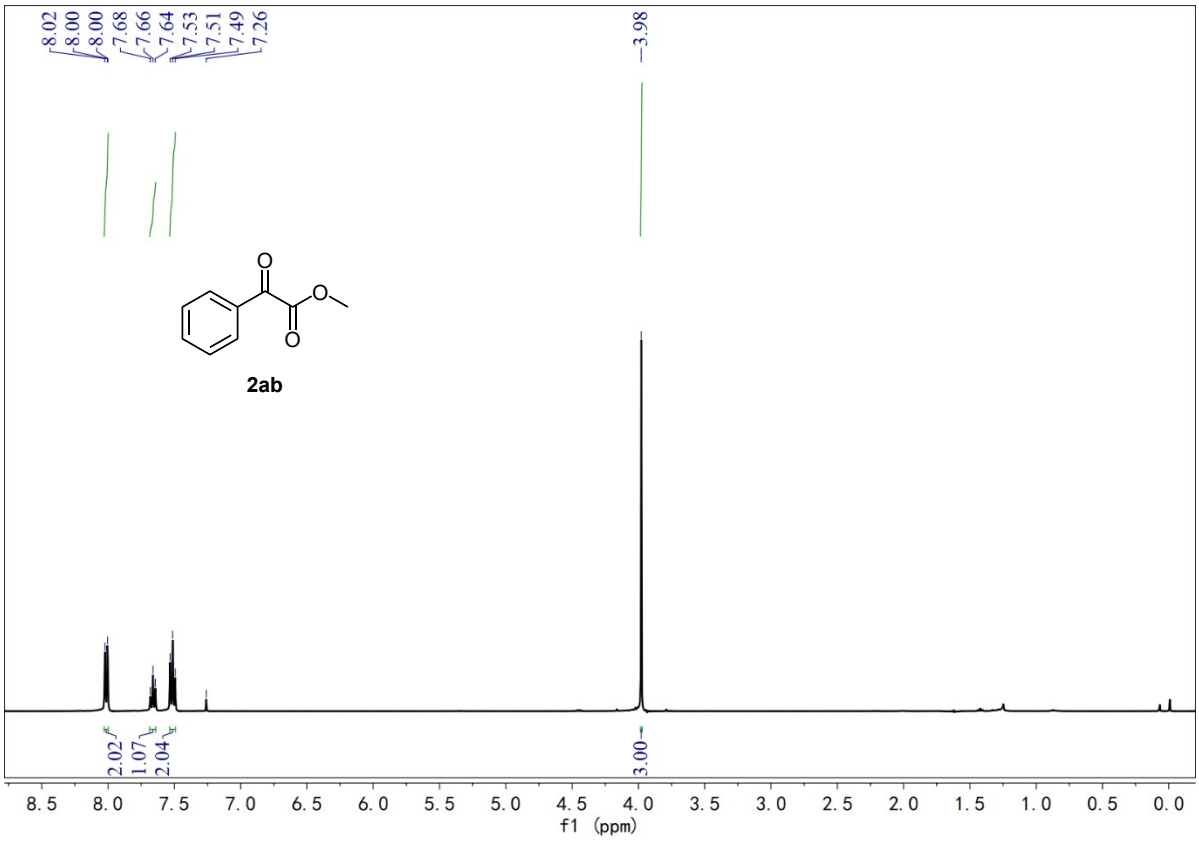
6. NMR spectra of all products

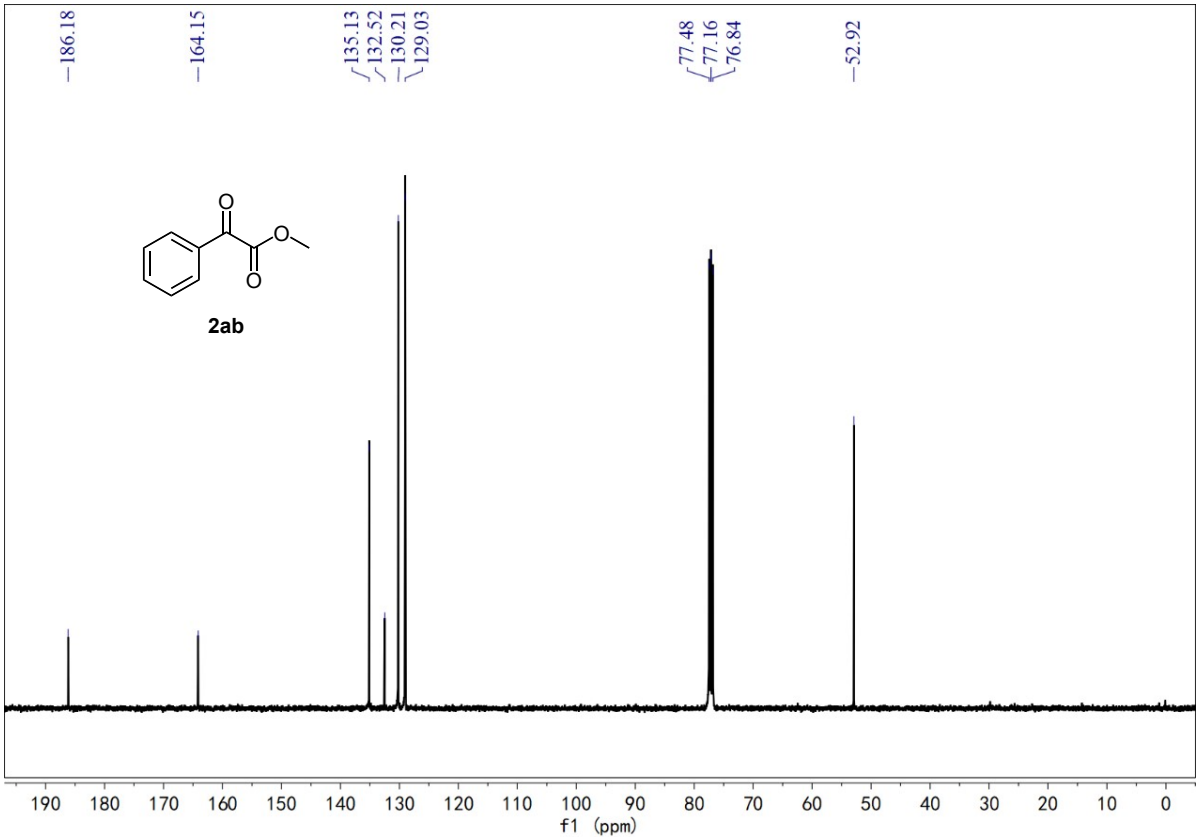
^{1}H and ^{13}C NMR spectra of **2aa**



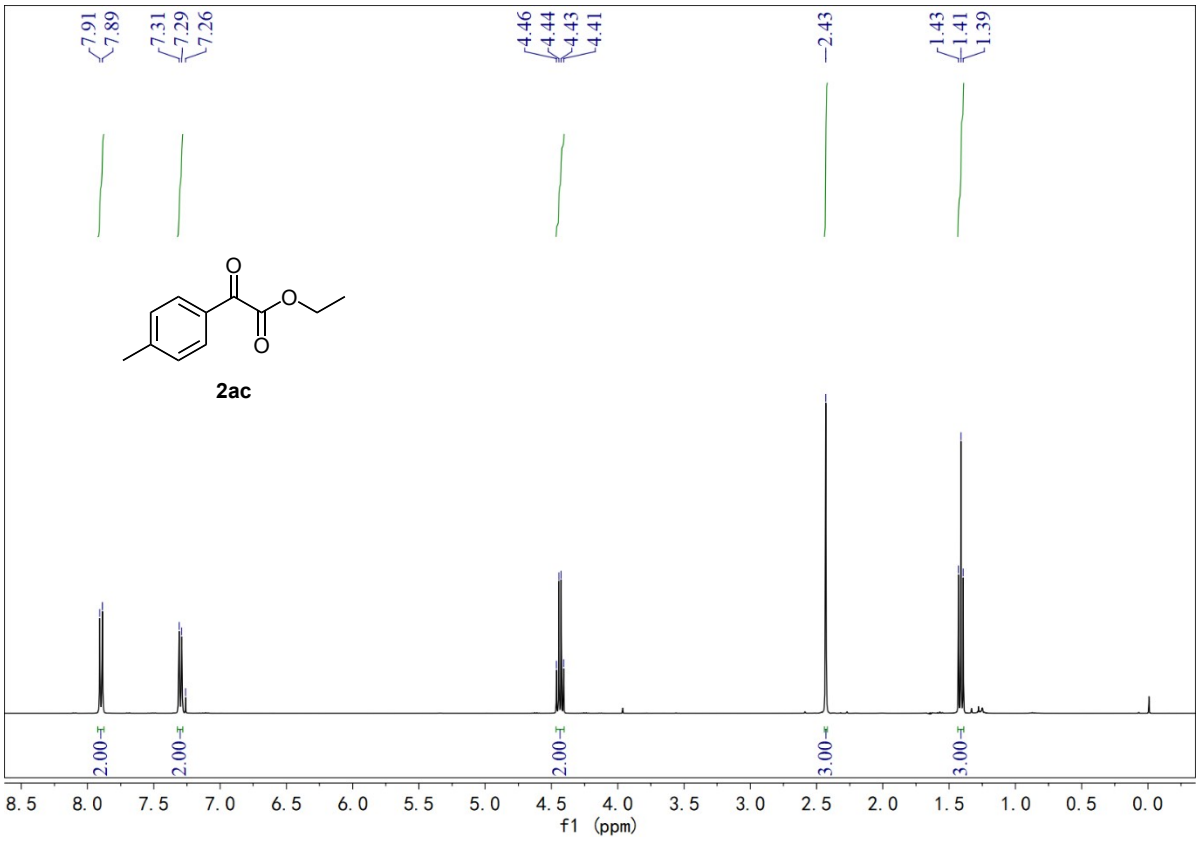


^{1}H and ^{13}C NMR spectra of **2ab**

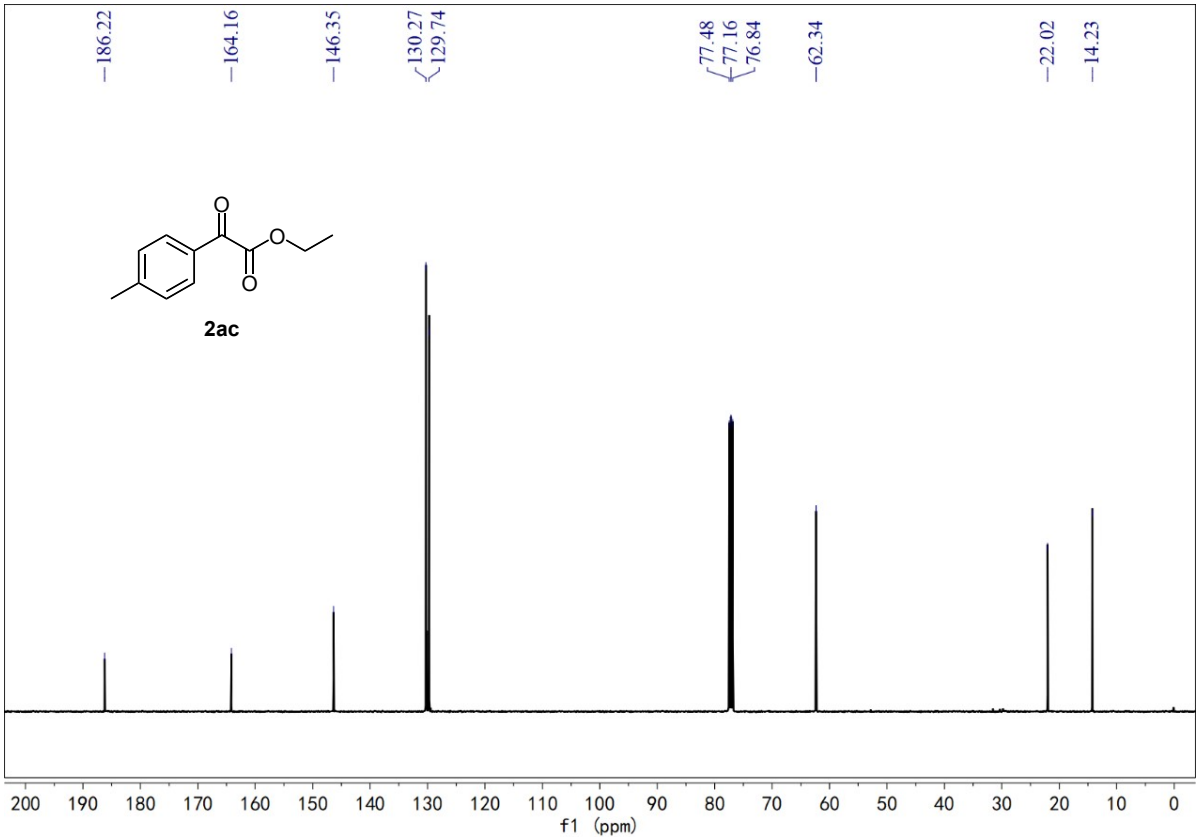




^{1}H and ^{13}C NMR spectra of **2ac**



2ac



2ac

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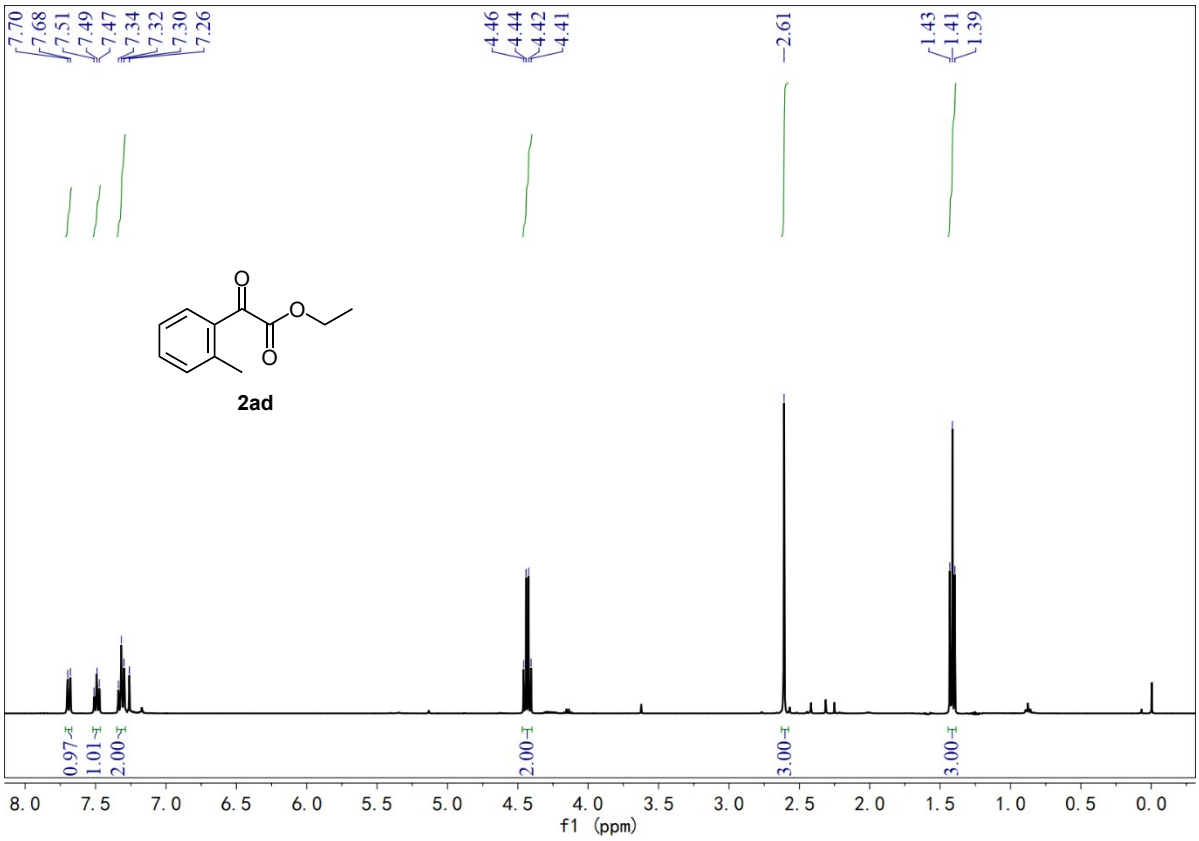

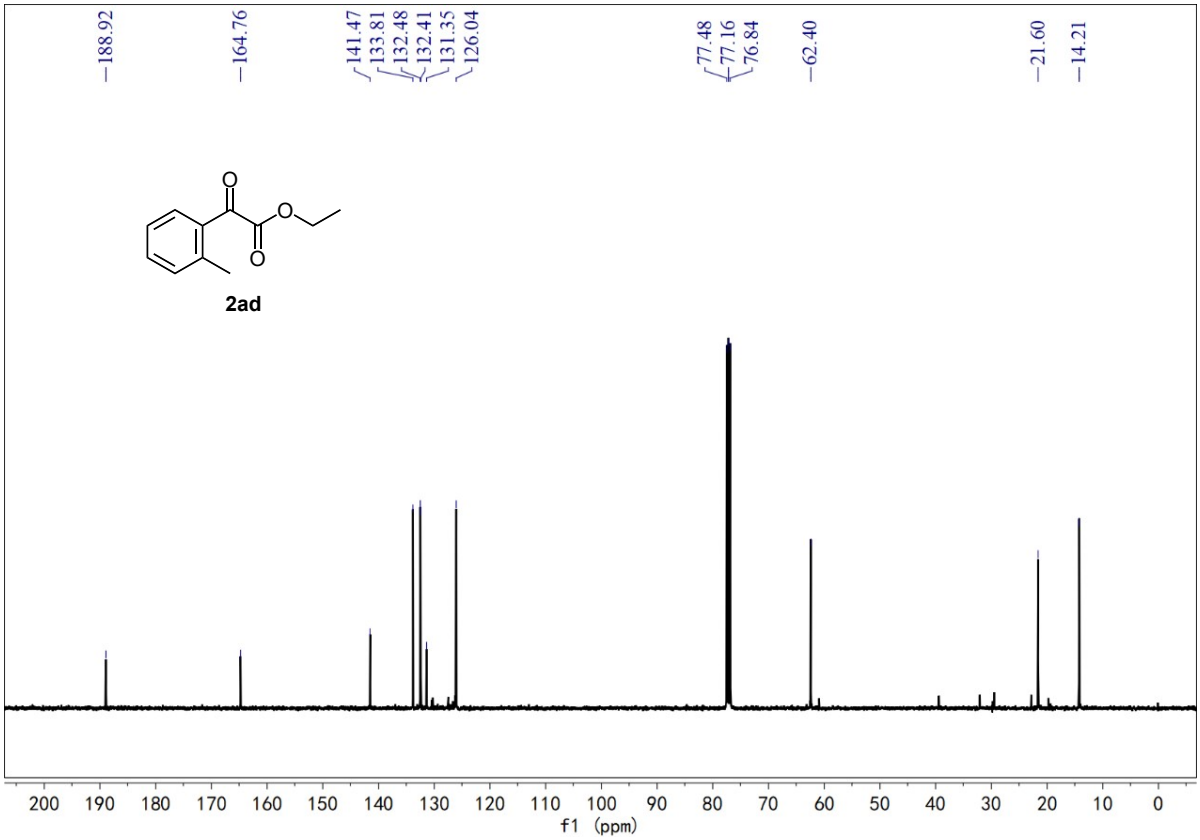

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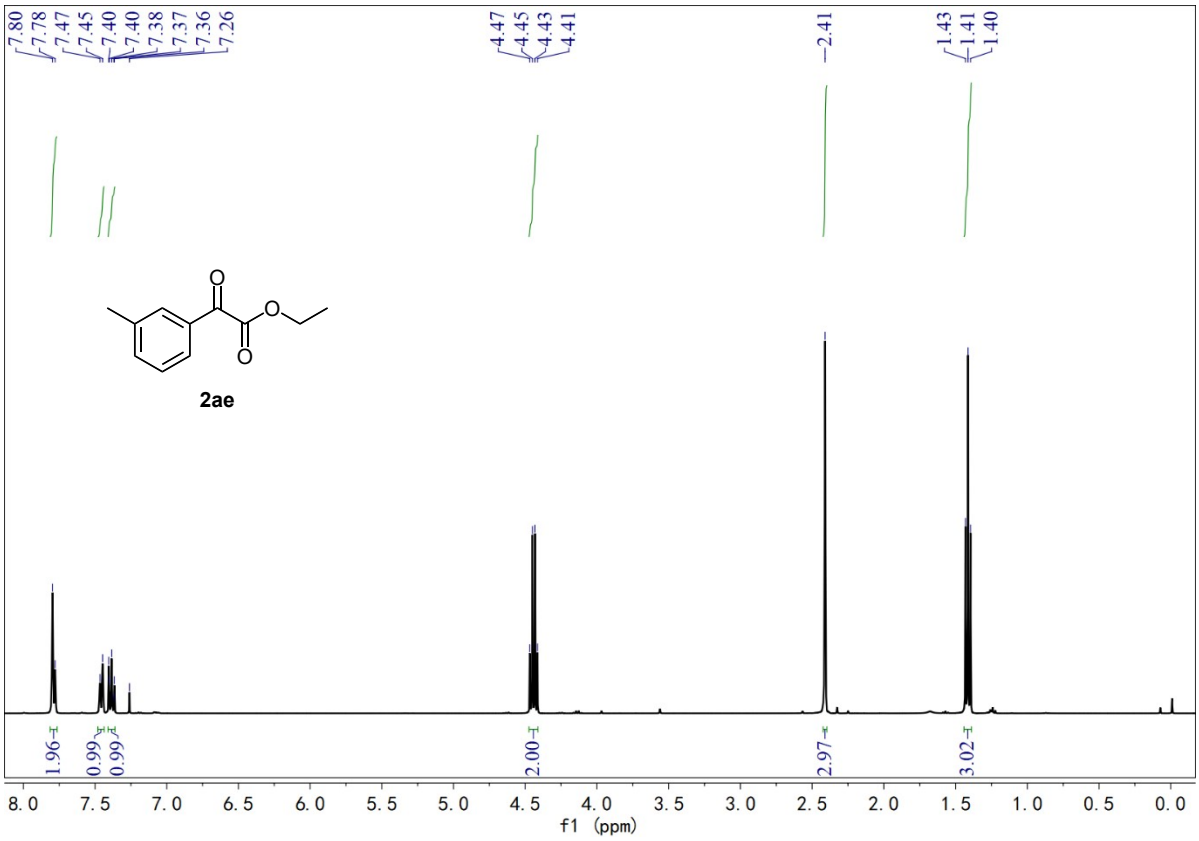

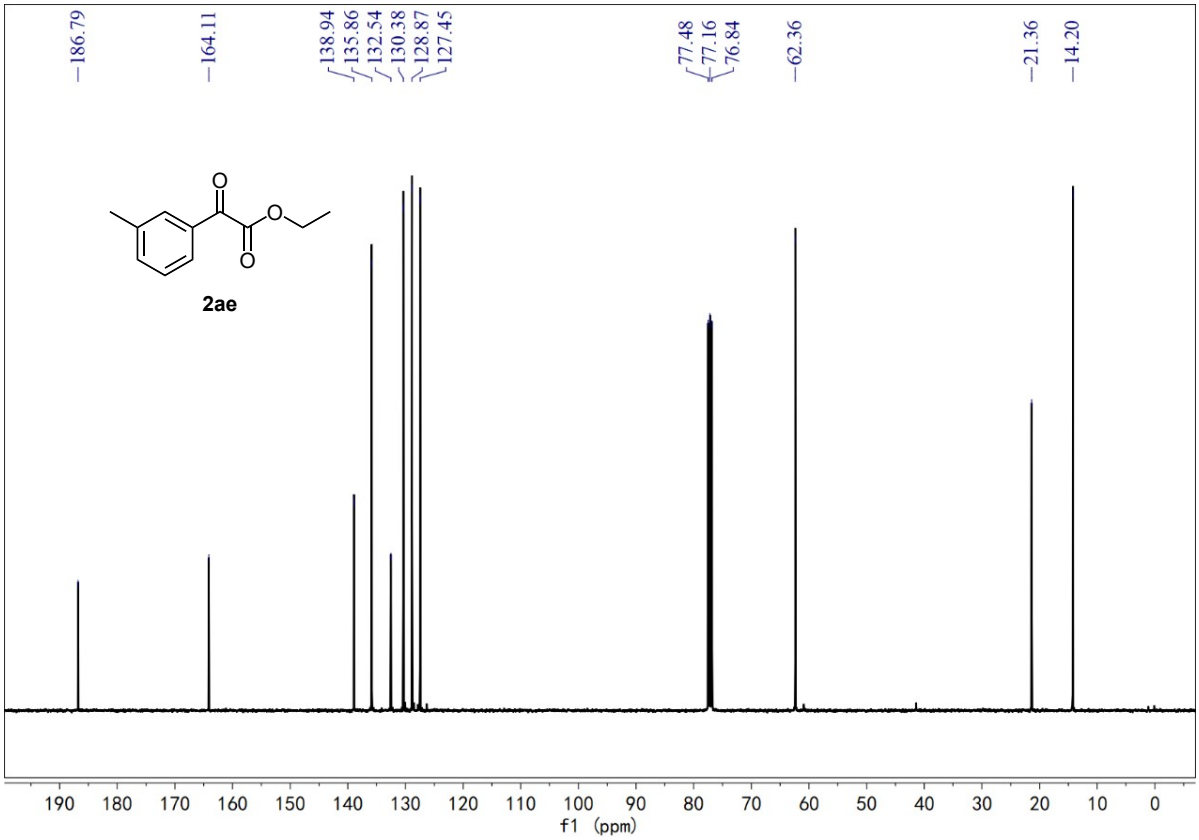

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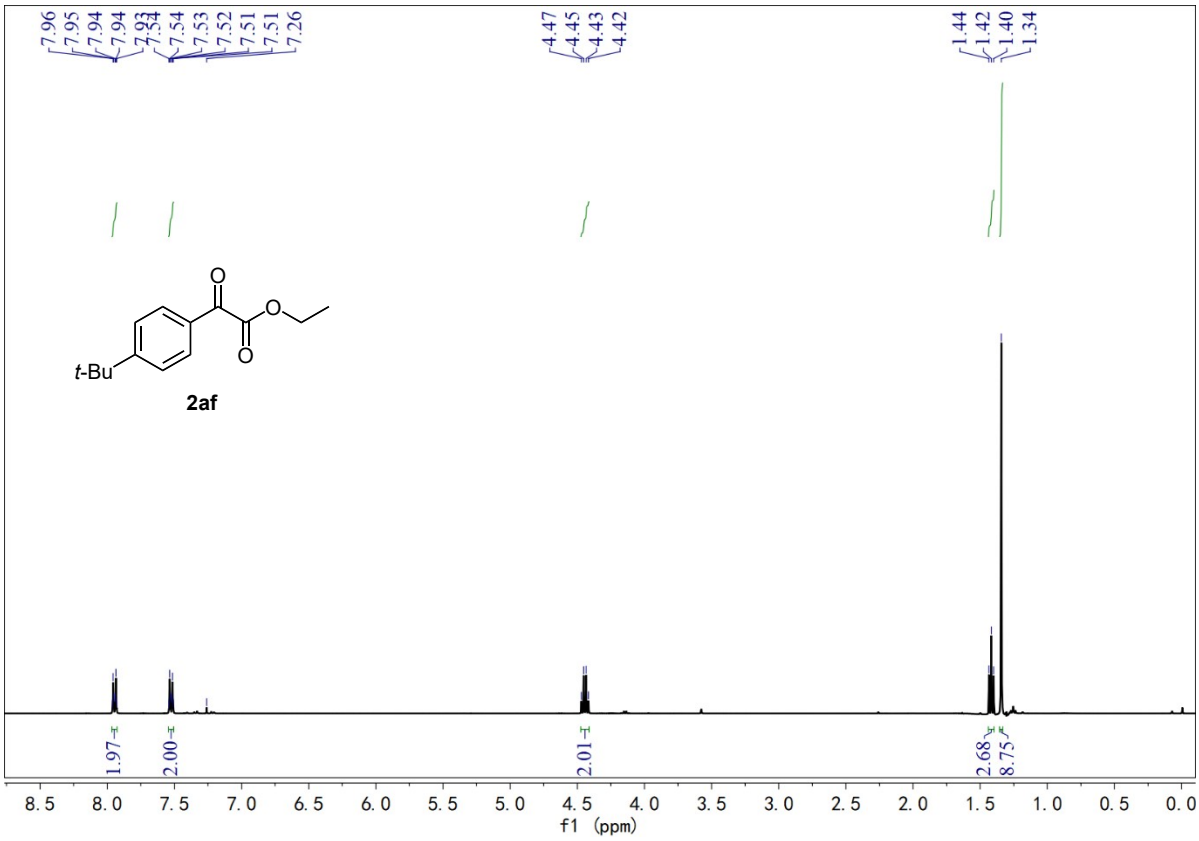
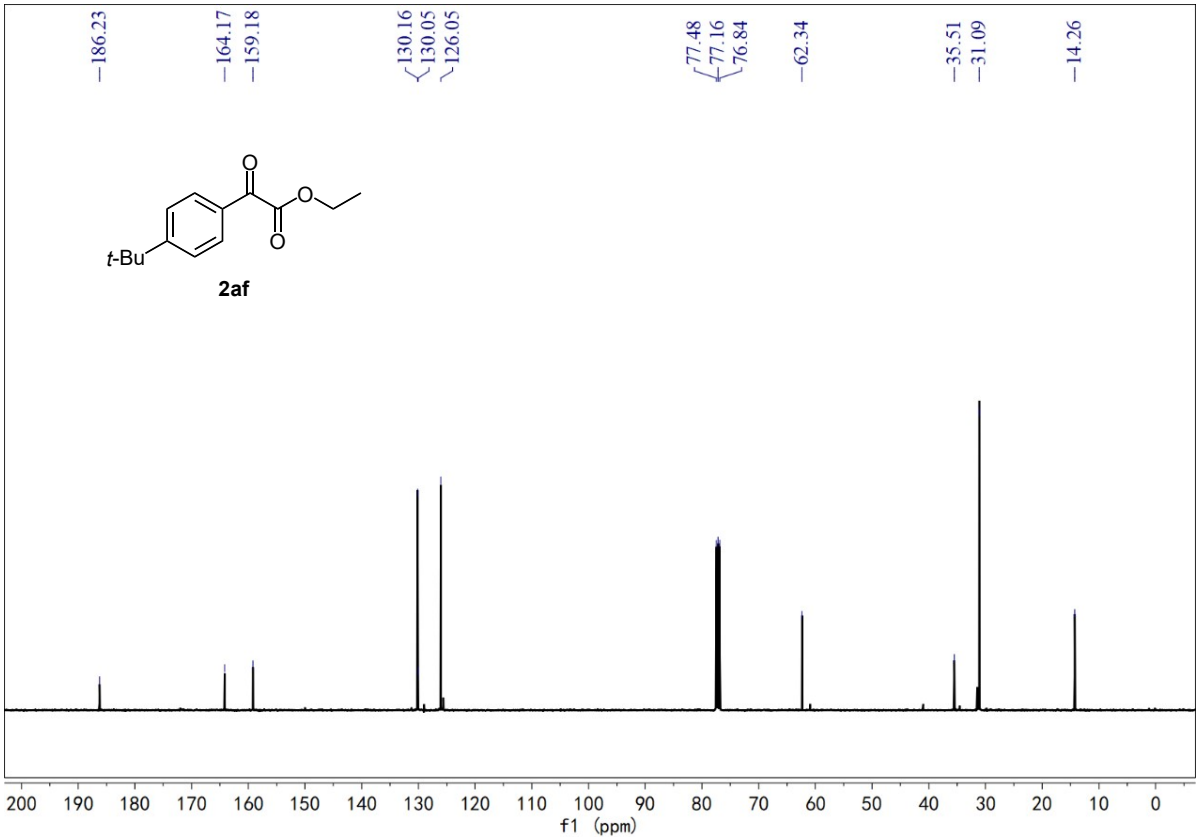
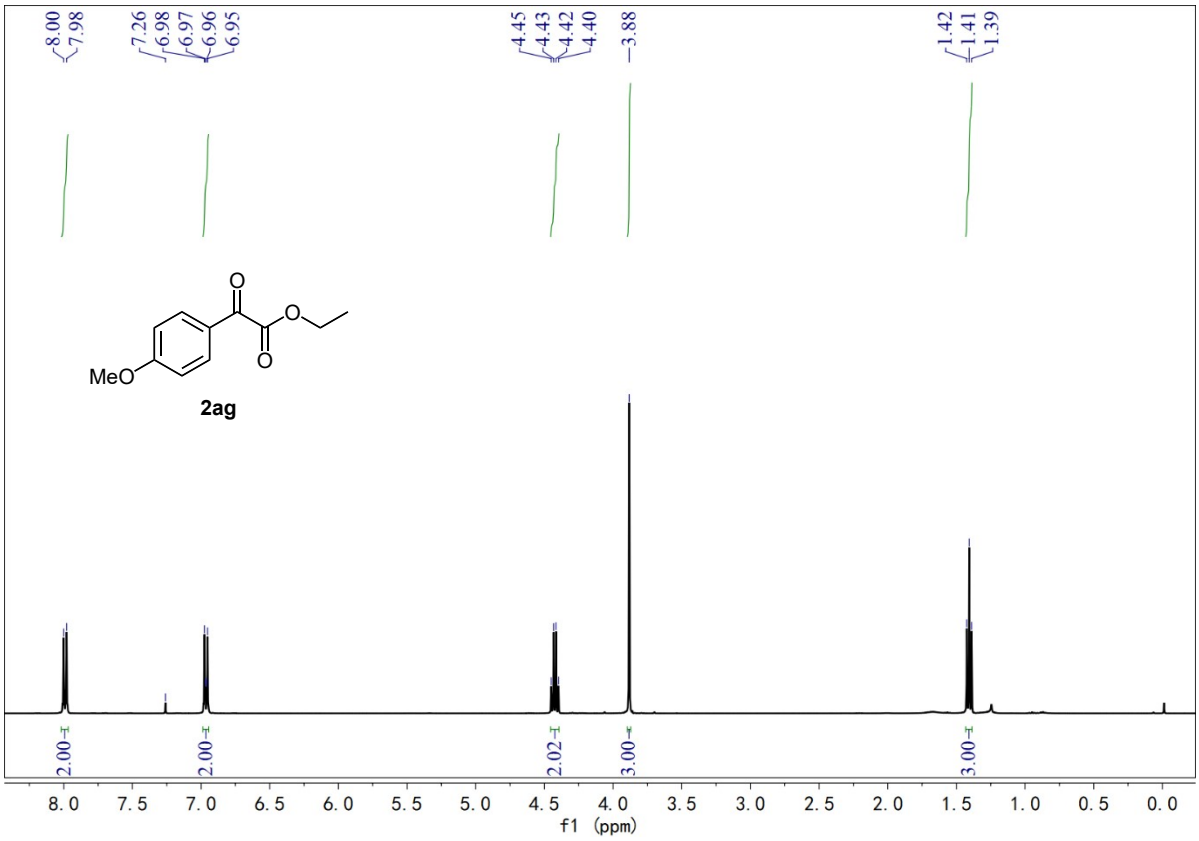
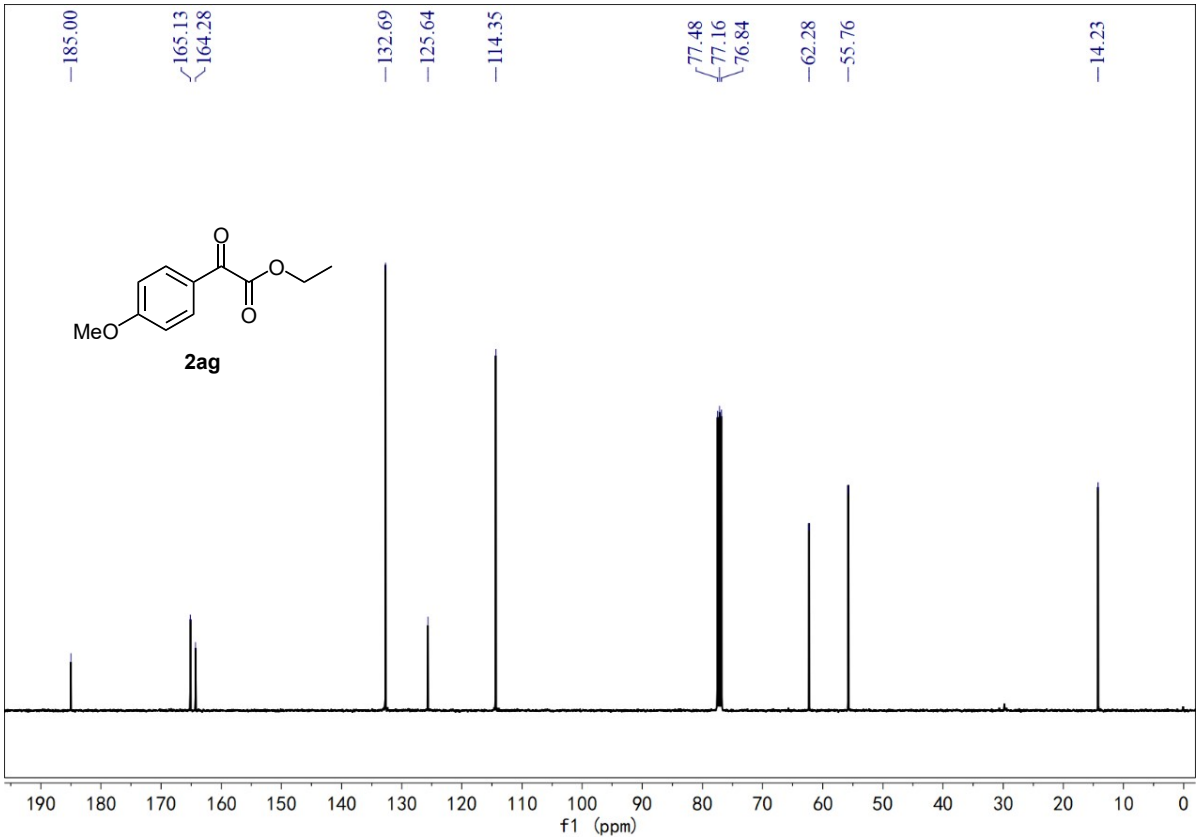


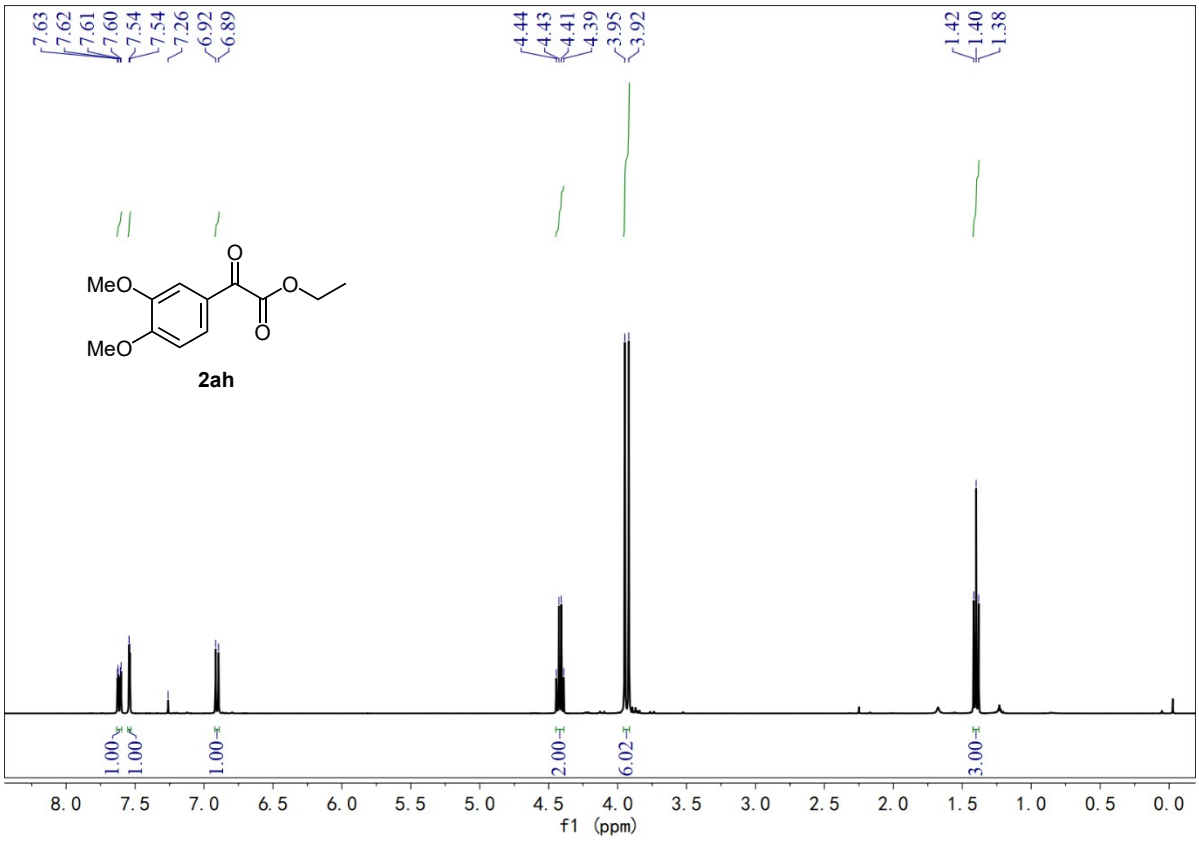


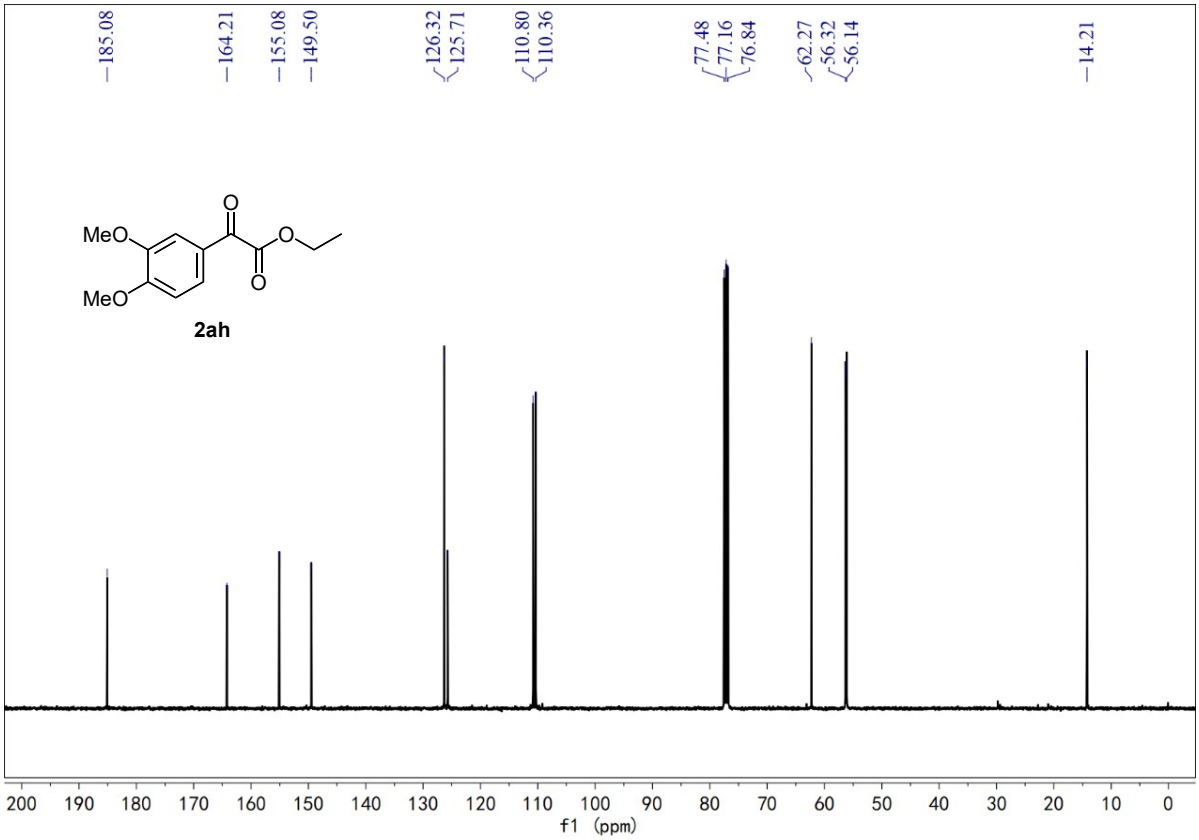
^{1}H and ^{13}C NMR spectra of **2ag**



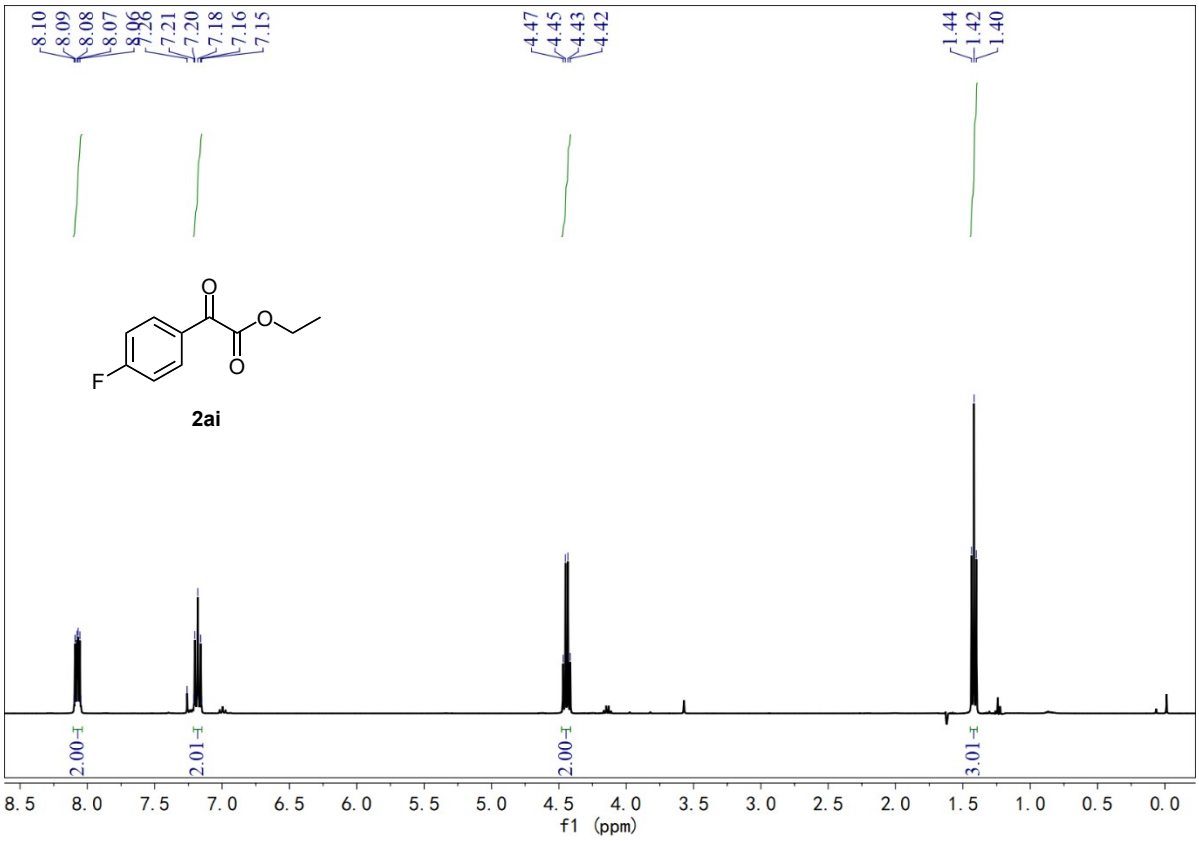


^{1}H and ^{13}C NMR spectra of **2ah**

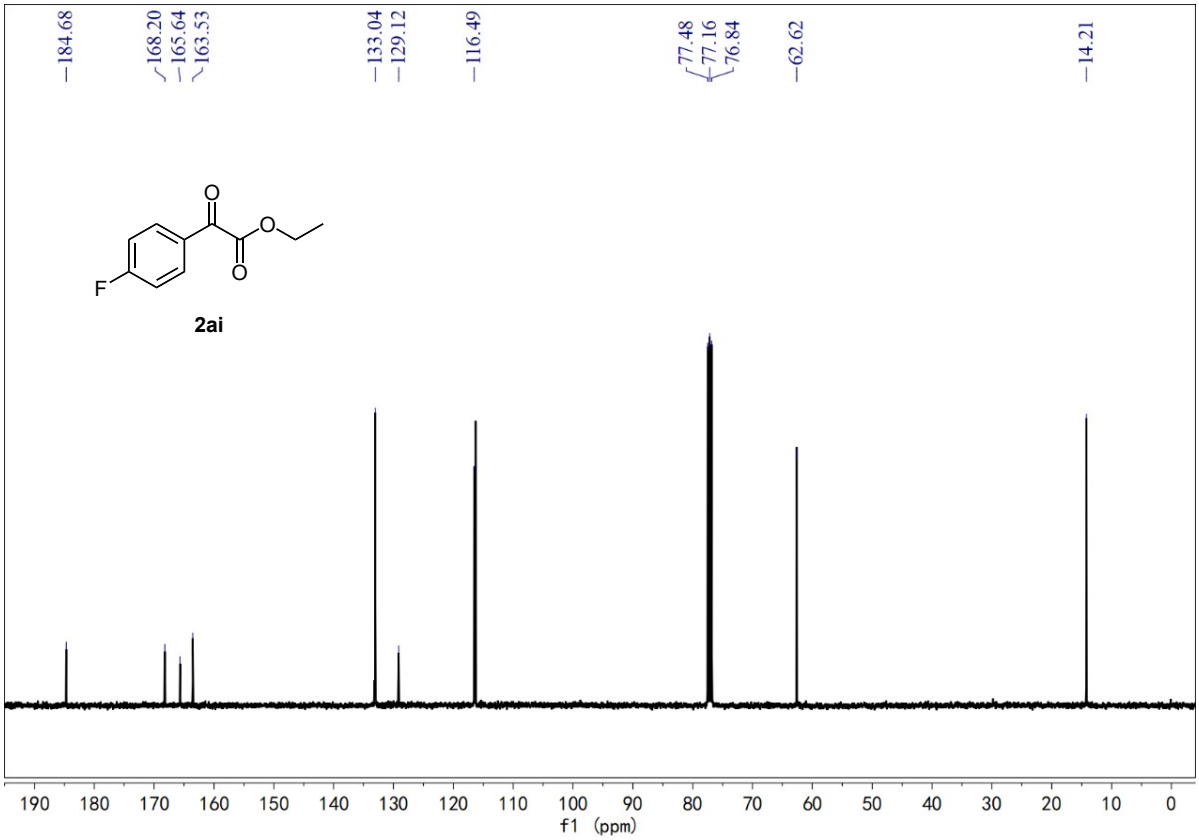




^{1}H and ^{13}C NMR and ^{19}F NMR spectra of **2ai**



2ai



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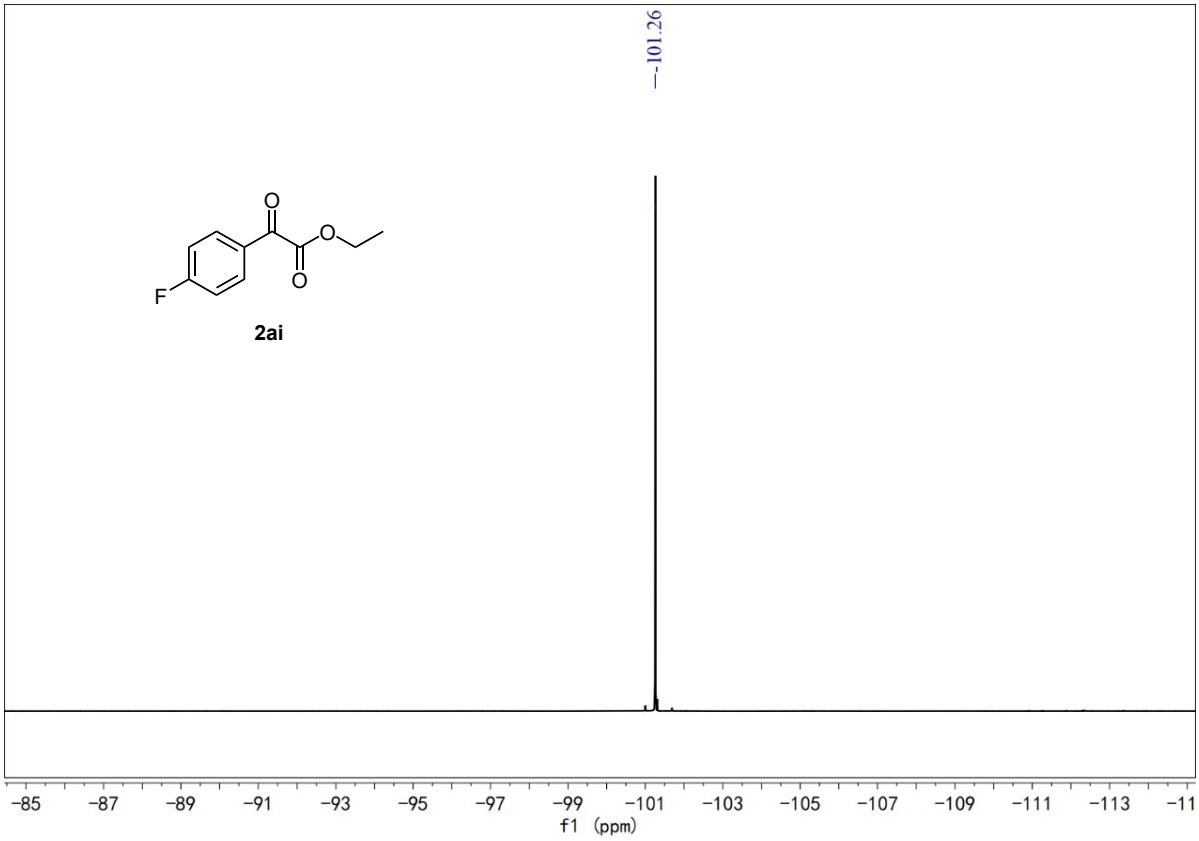
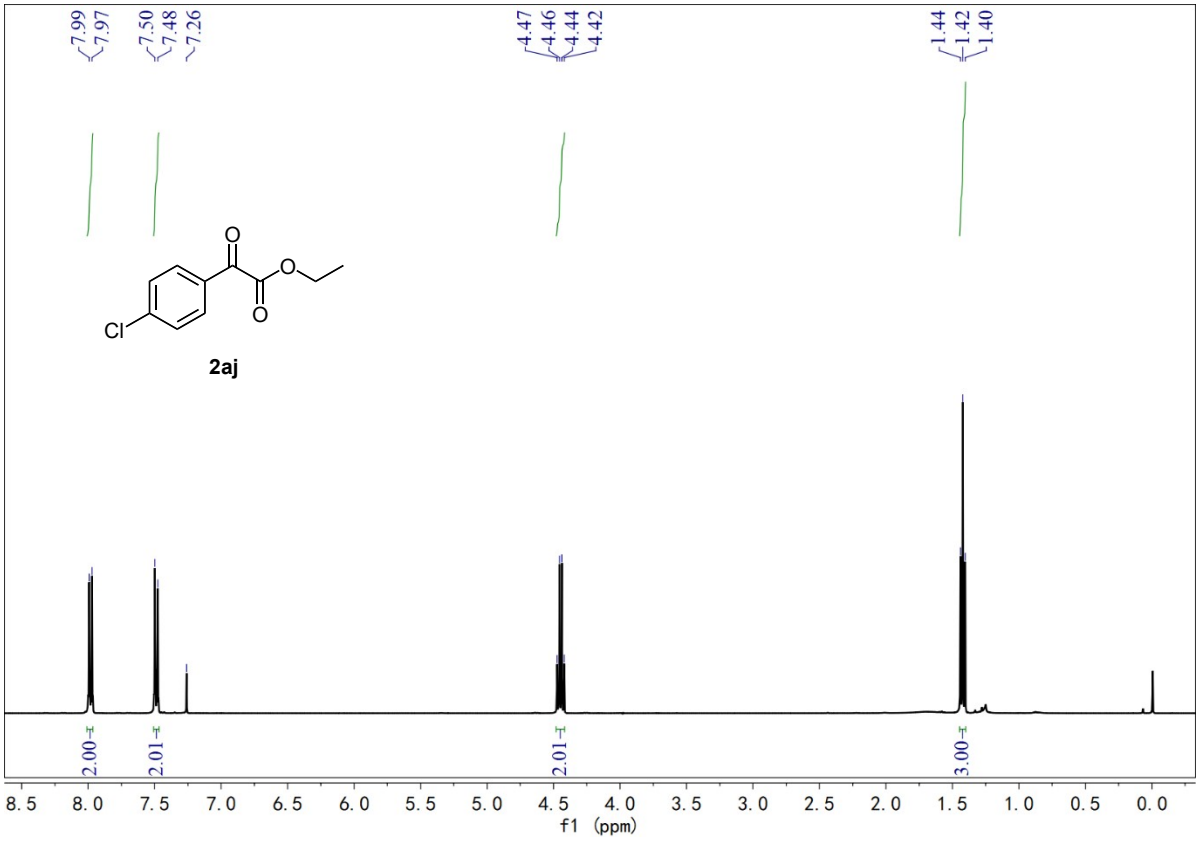
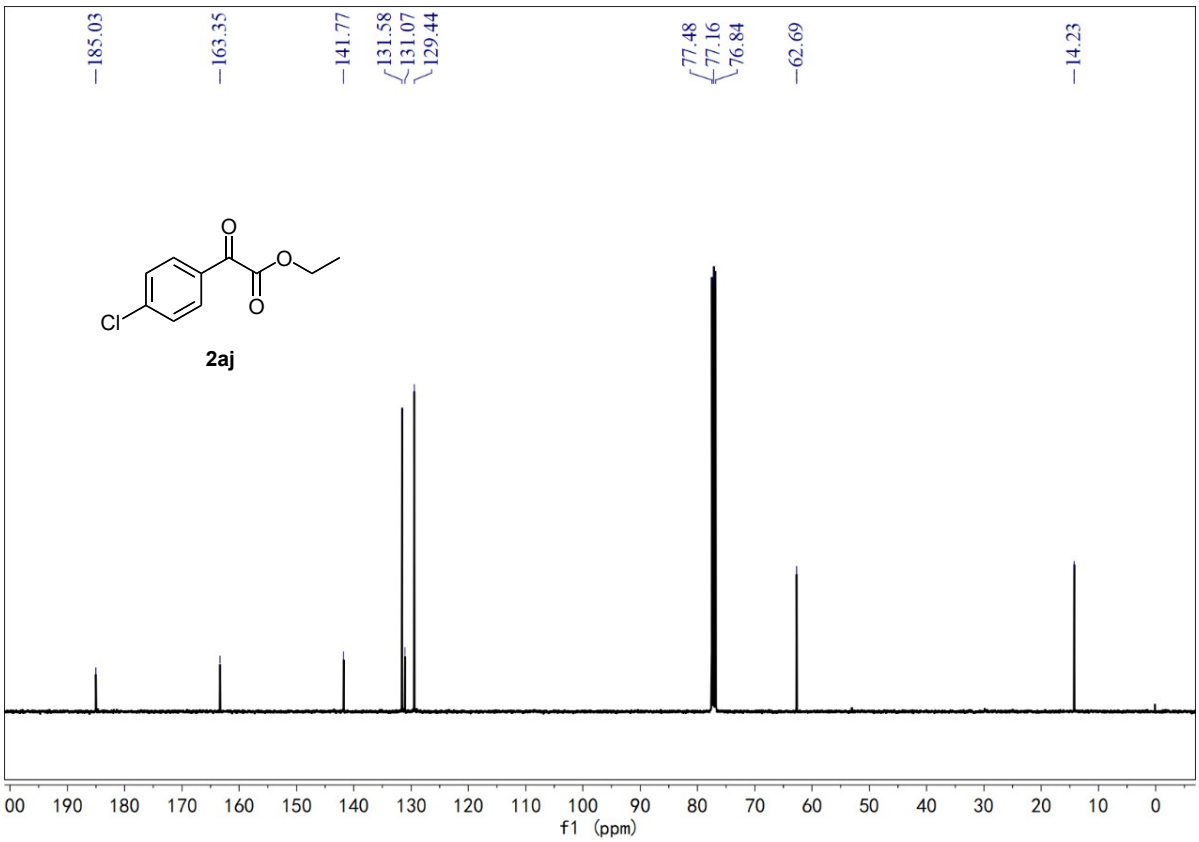
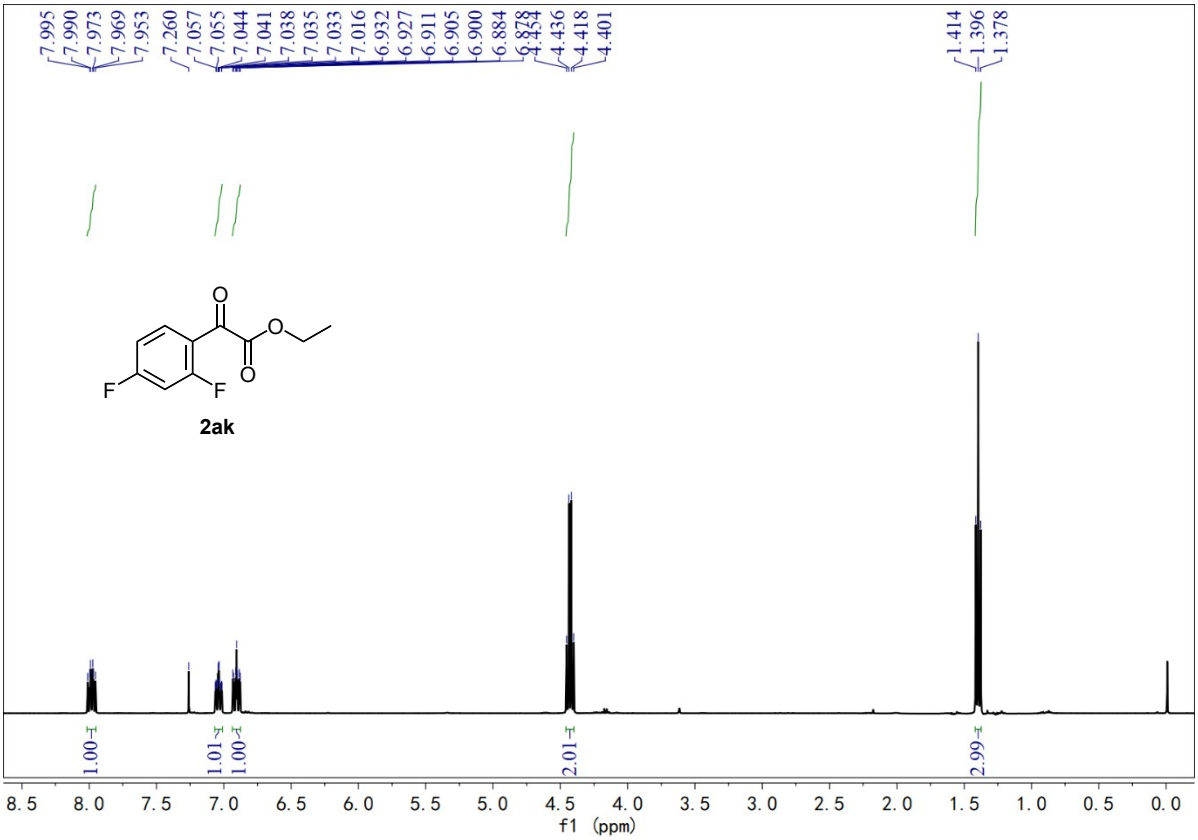


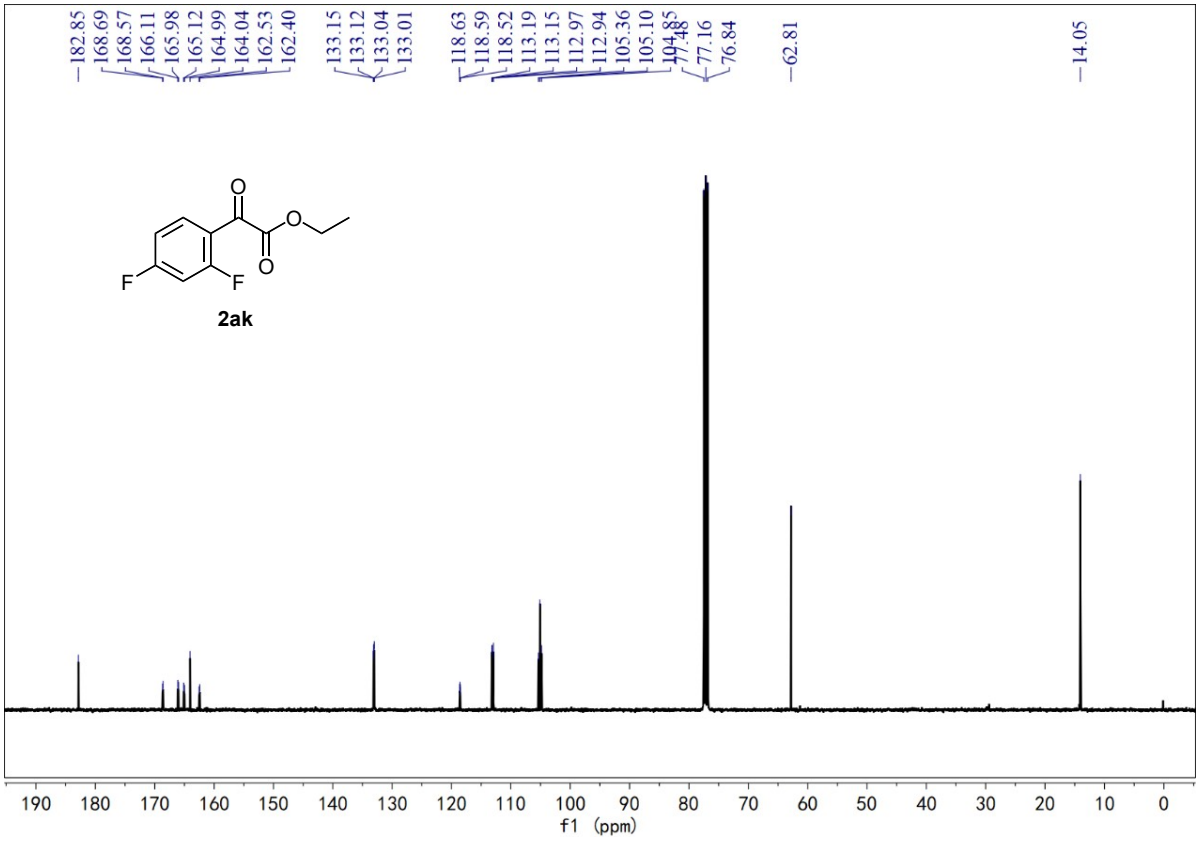
^{1}H and ^{13}C NMR and spectra of **2aj**

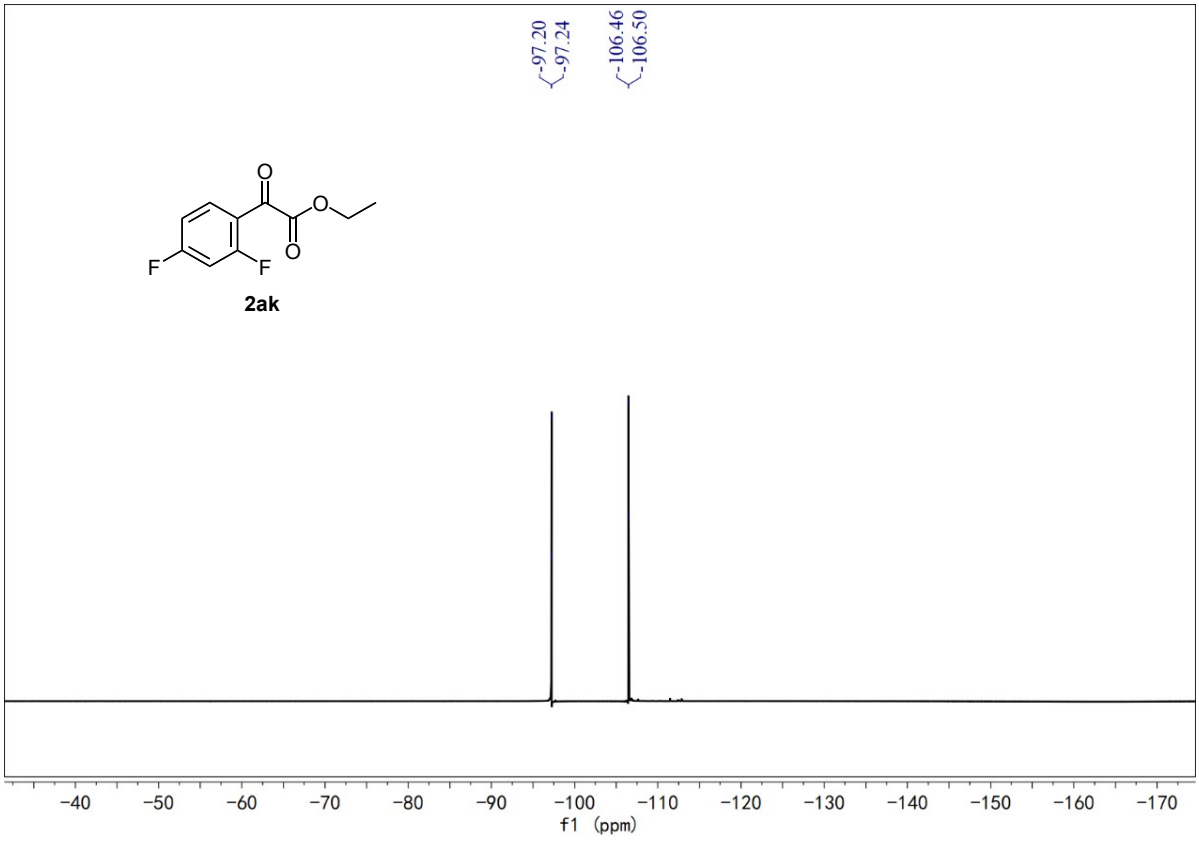




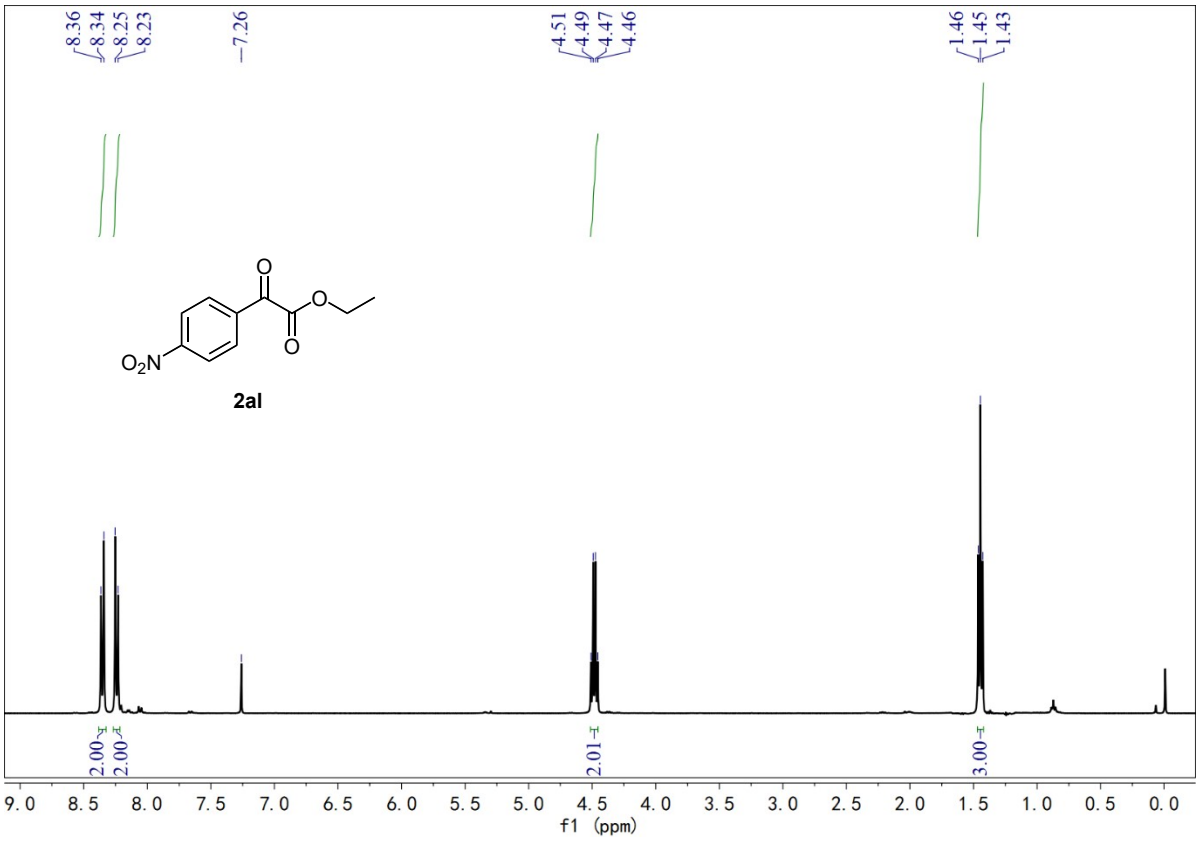
^{1}H and ^{13}C NMR and ^{19}F NMR spectra of **2ak**

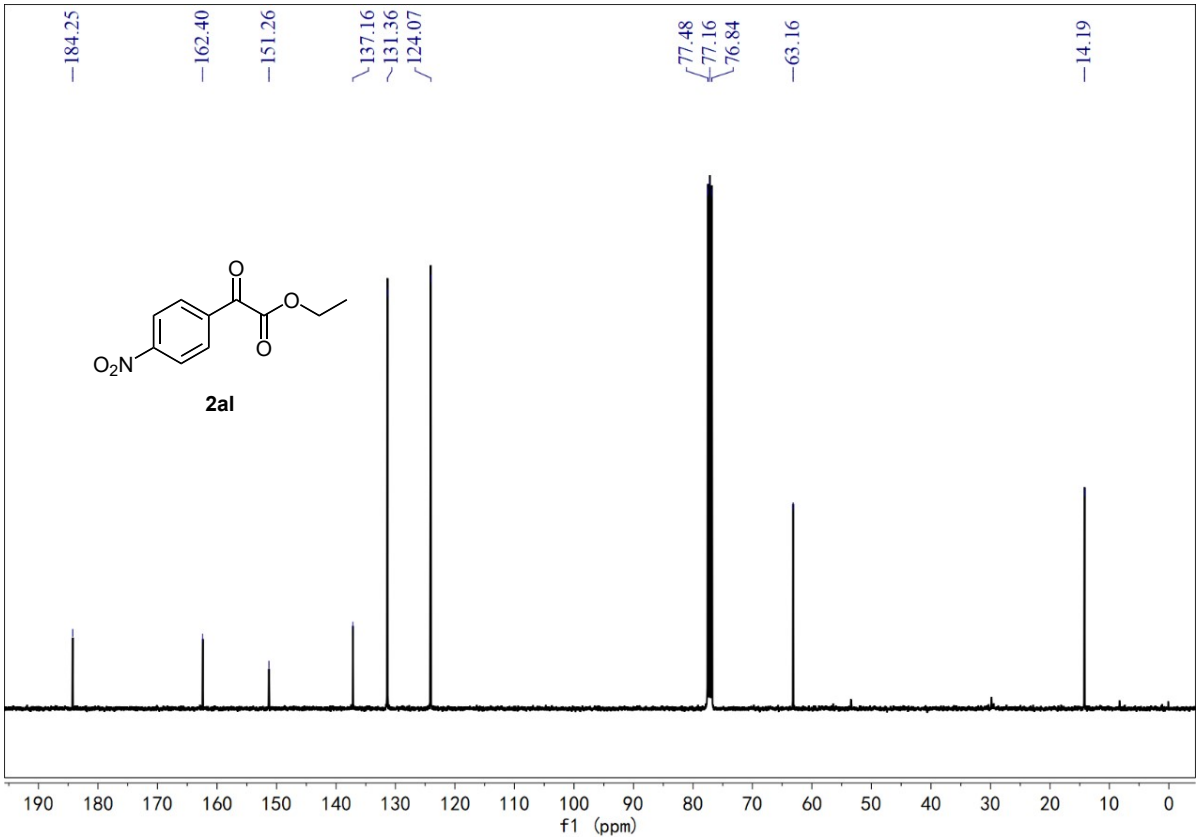



182.85
168.69
168.57
166.11
165.98
165.12
164.99
164.04
162.53
162.40
133.15
133.12
133.04
133.01
118.63
118.59
118.52
113.19
113.15
112.97
112.94
105.36
105.10
77.48
77.16
76.84
62.81
14.05
O
O
O
F
F
2ak
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

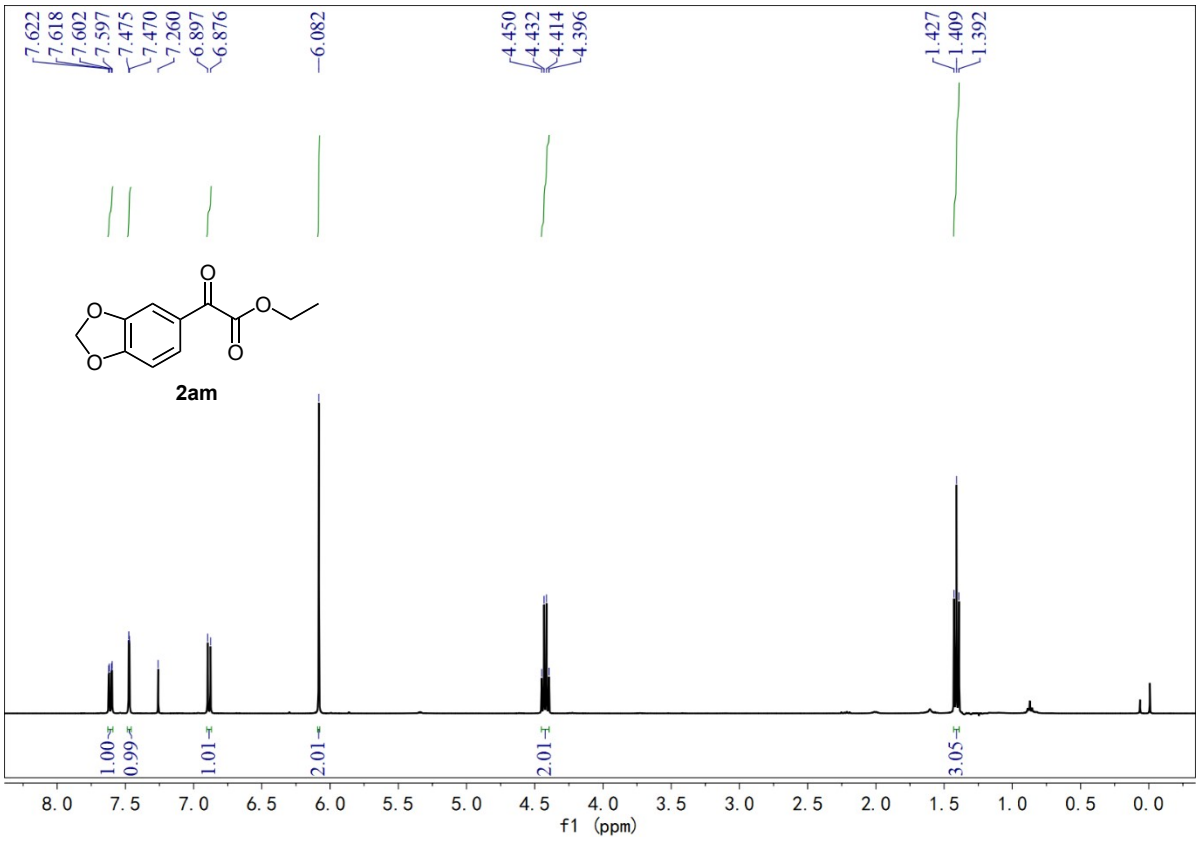

-97.20
-97.24
-106.46
-106.50
O
O
O
F
F
2ak
-40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170
f1 (ppm)

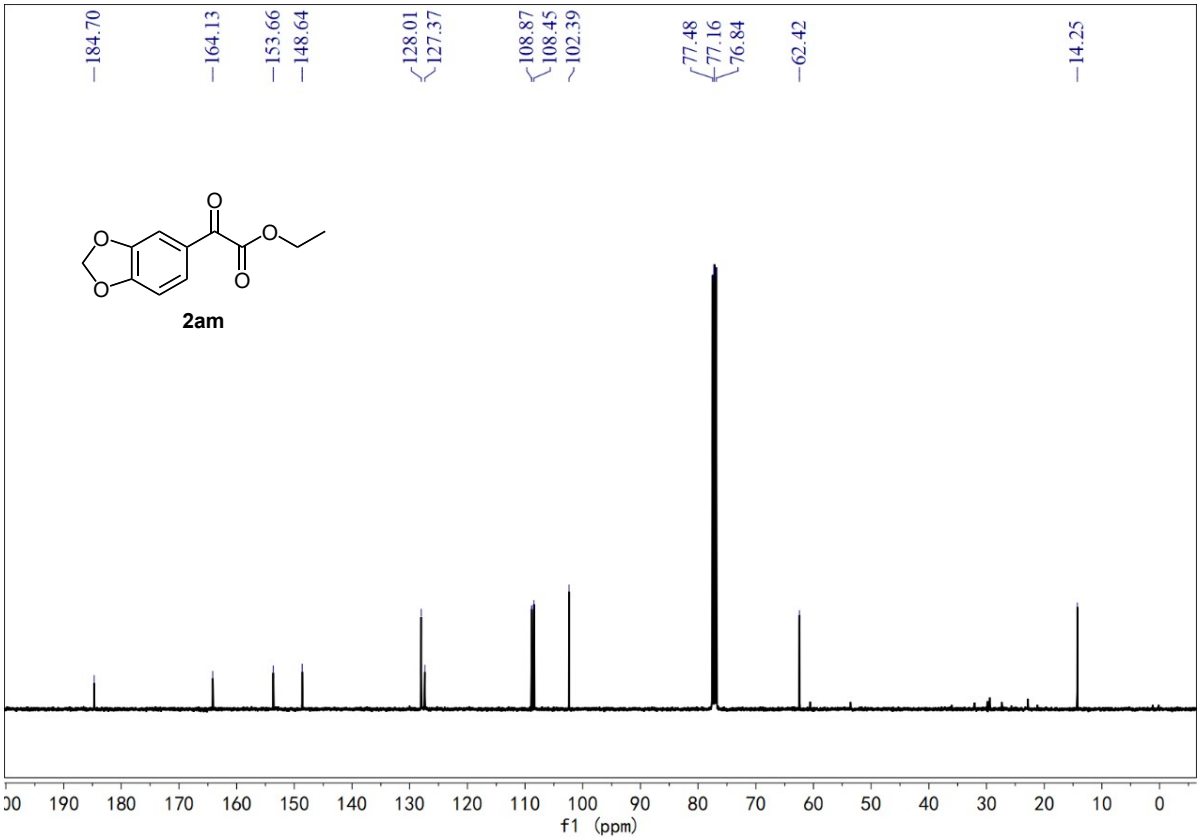
^{1}H and ^{13}C NMR spectra of **2al**



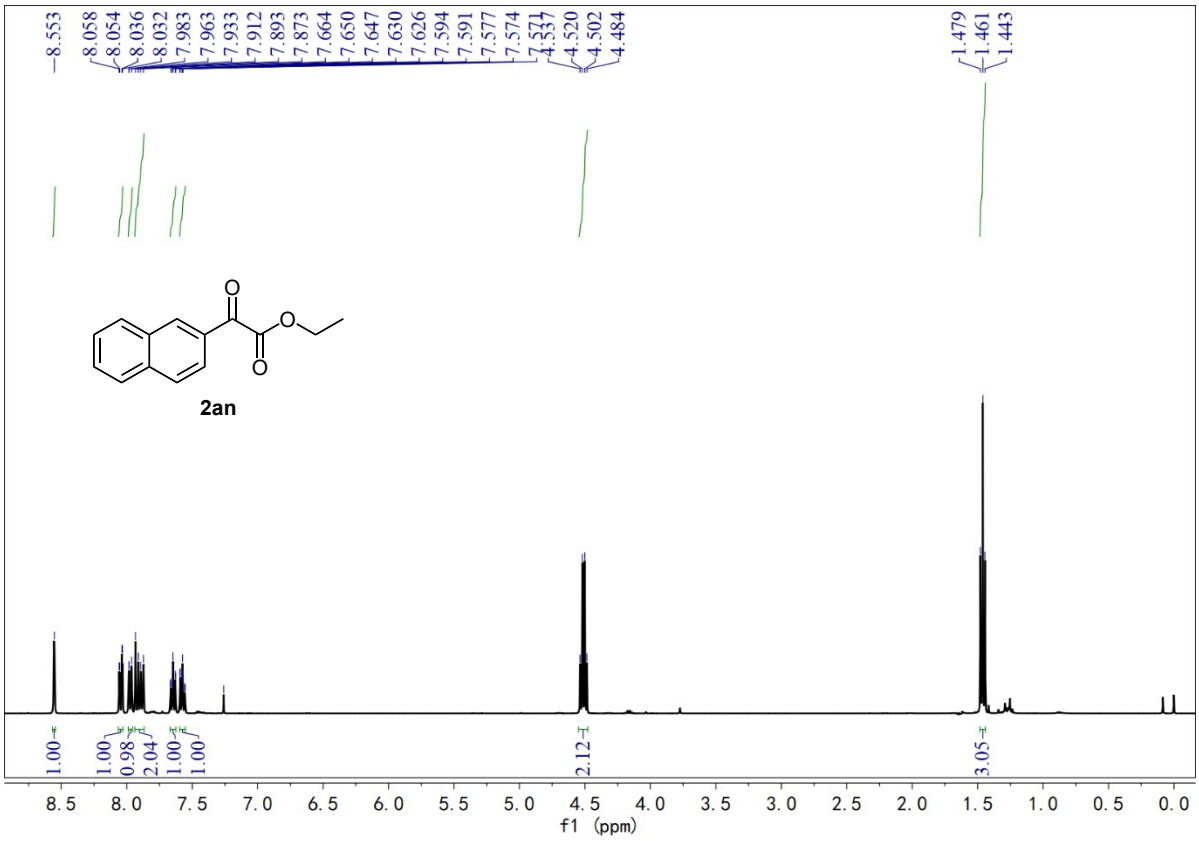


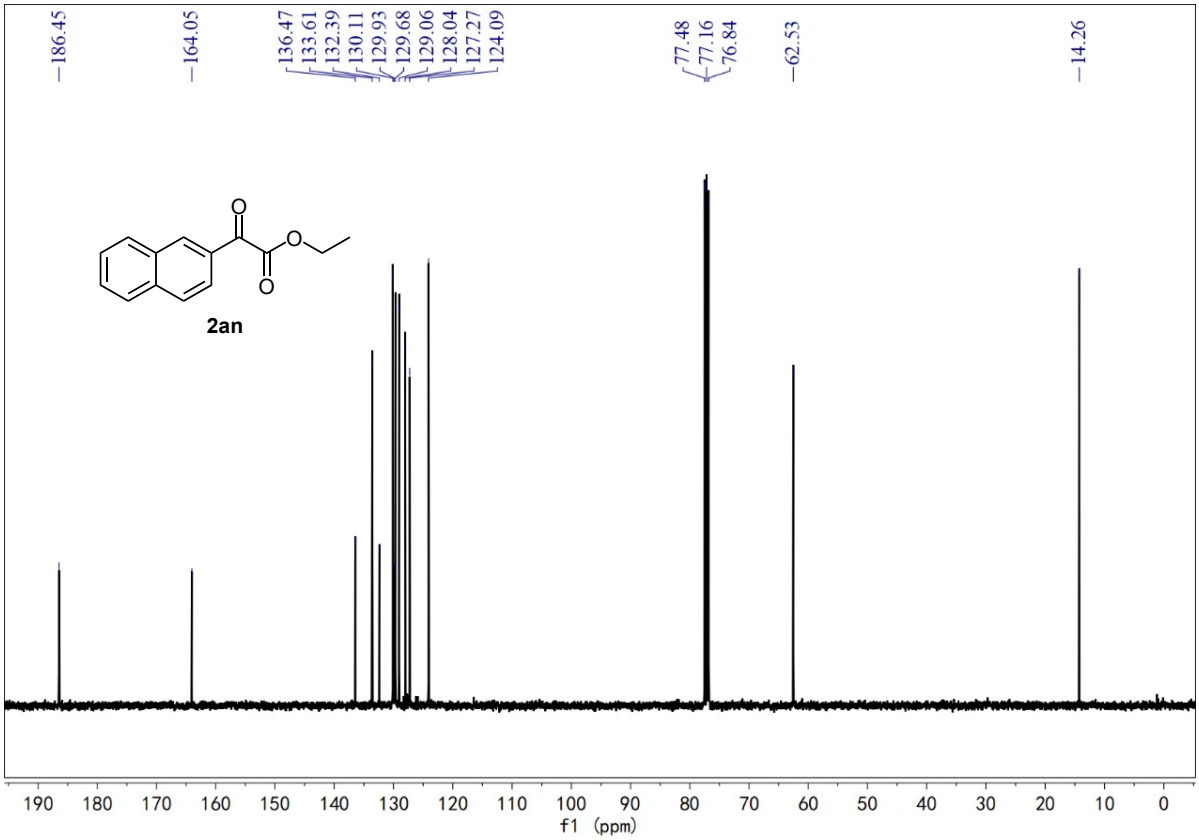
^{1}H and ^{13}C NMR spectra of **2am**



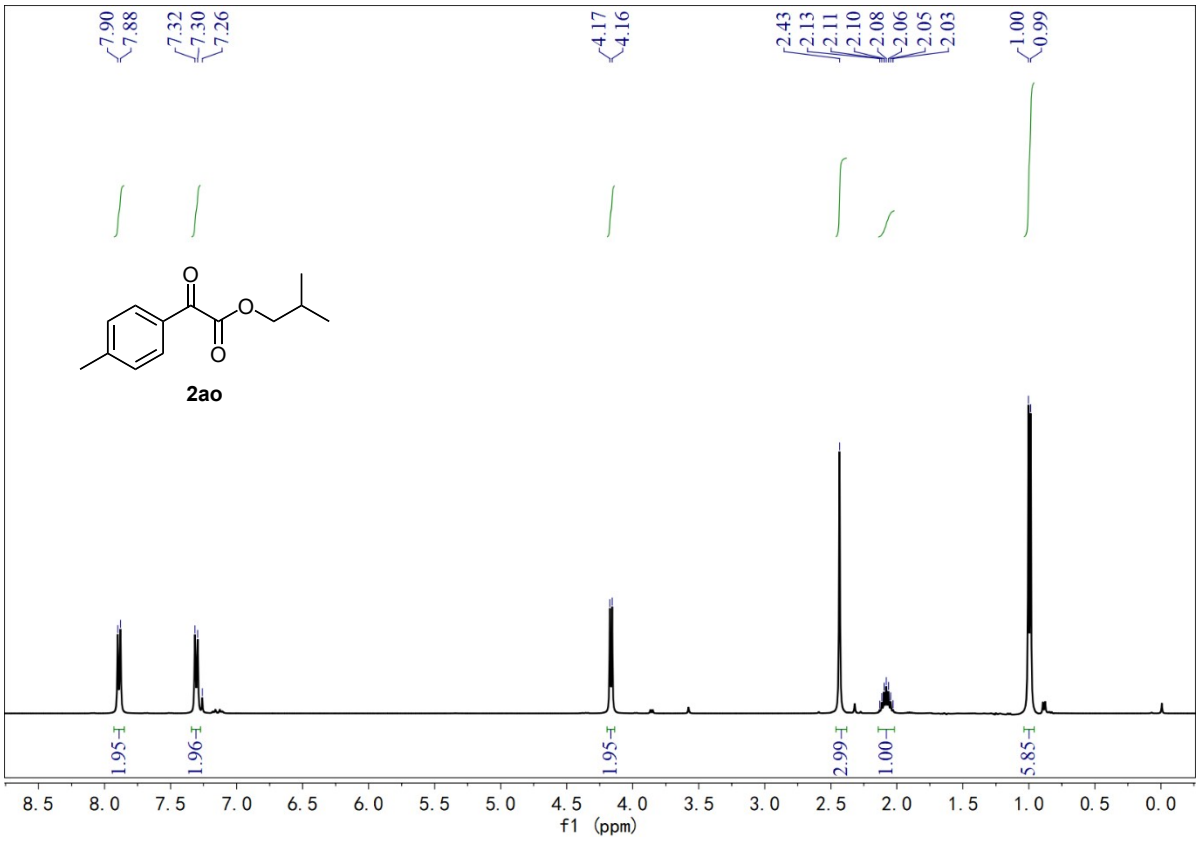


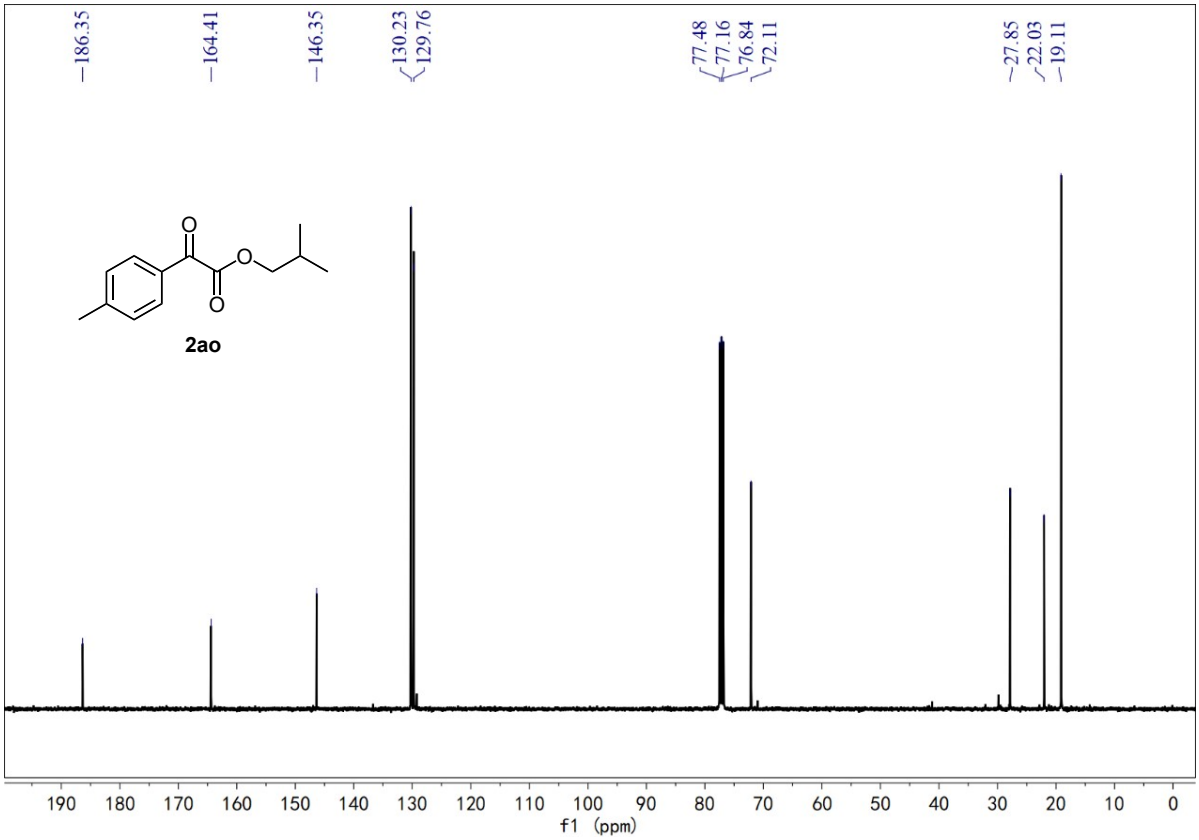
^{1}H and ^{13}C NMR spectra of **2an**



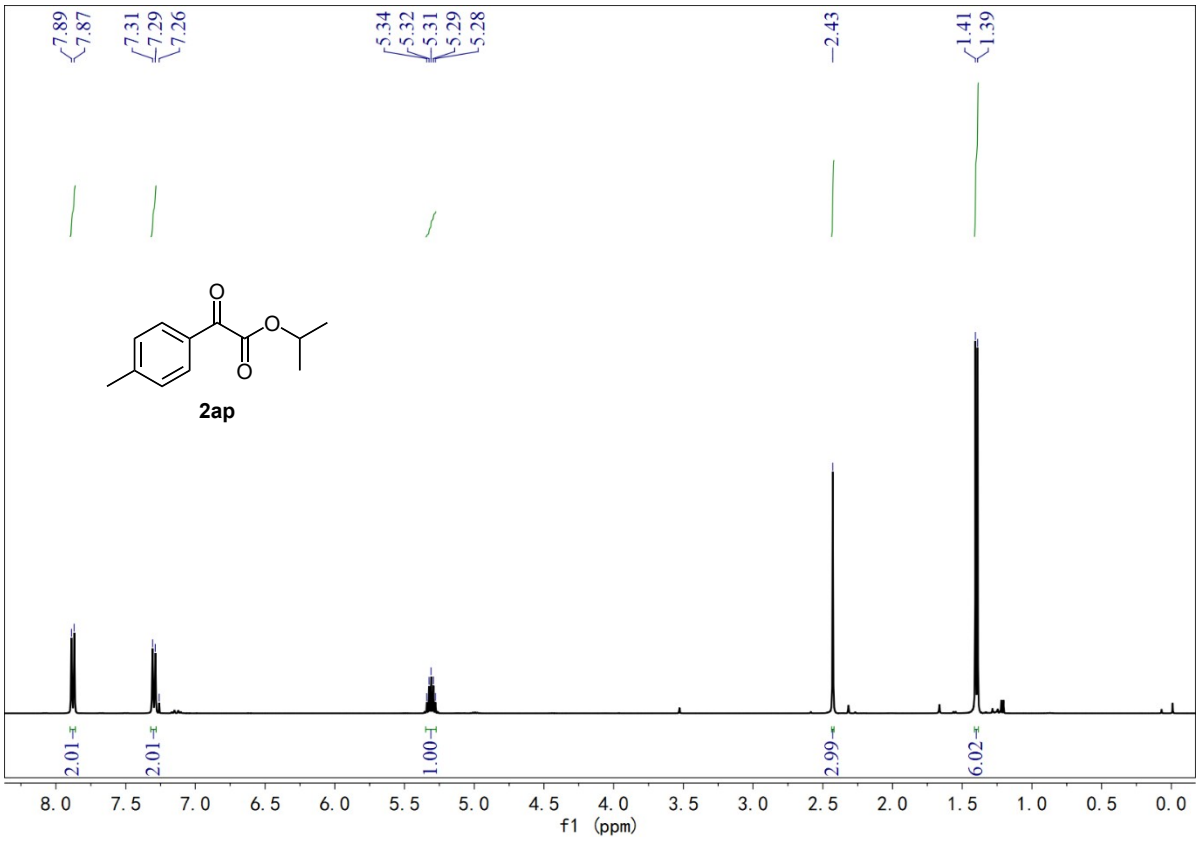


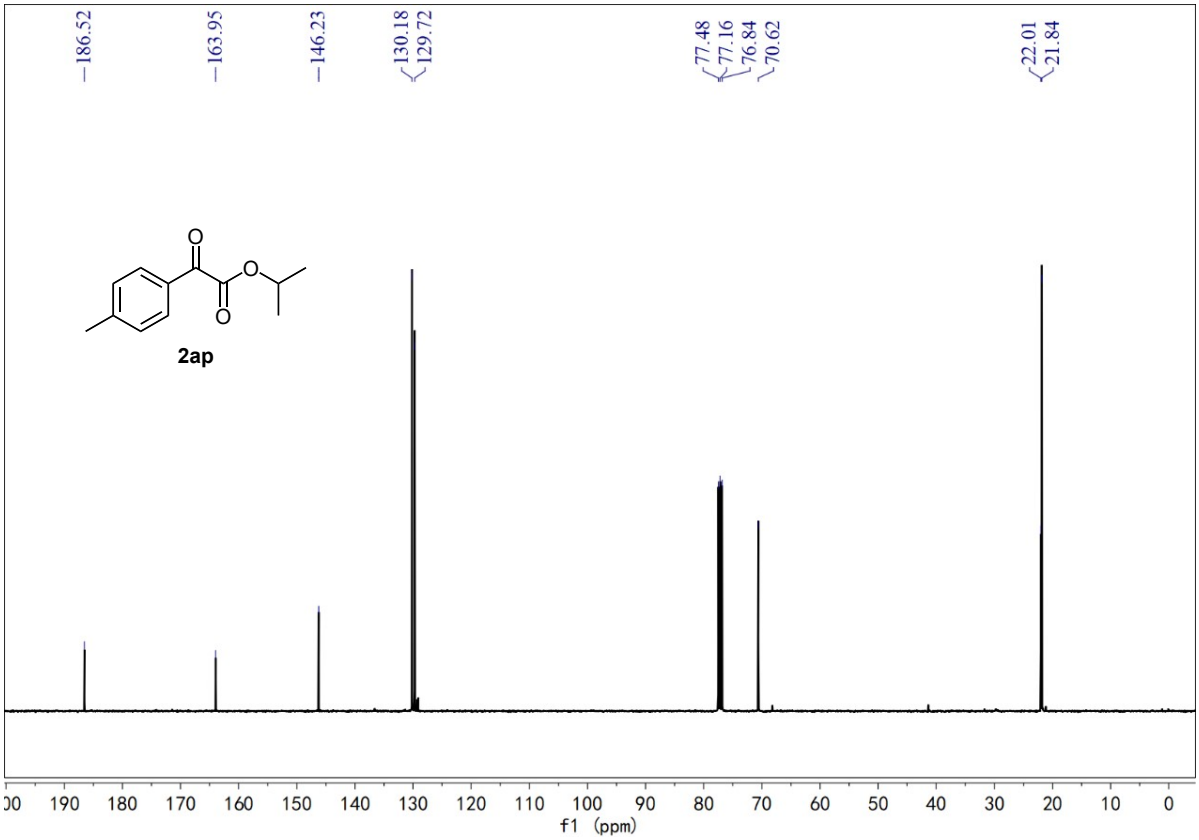
^{1}H and ^{13}C NMR spectra of **2ao**



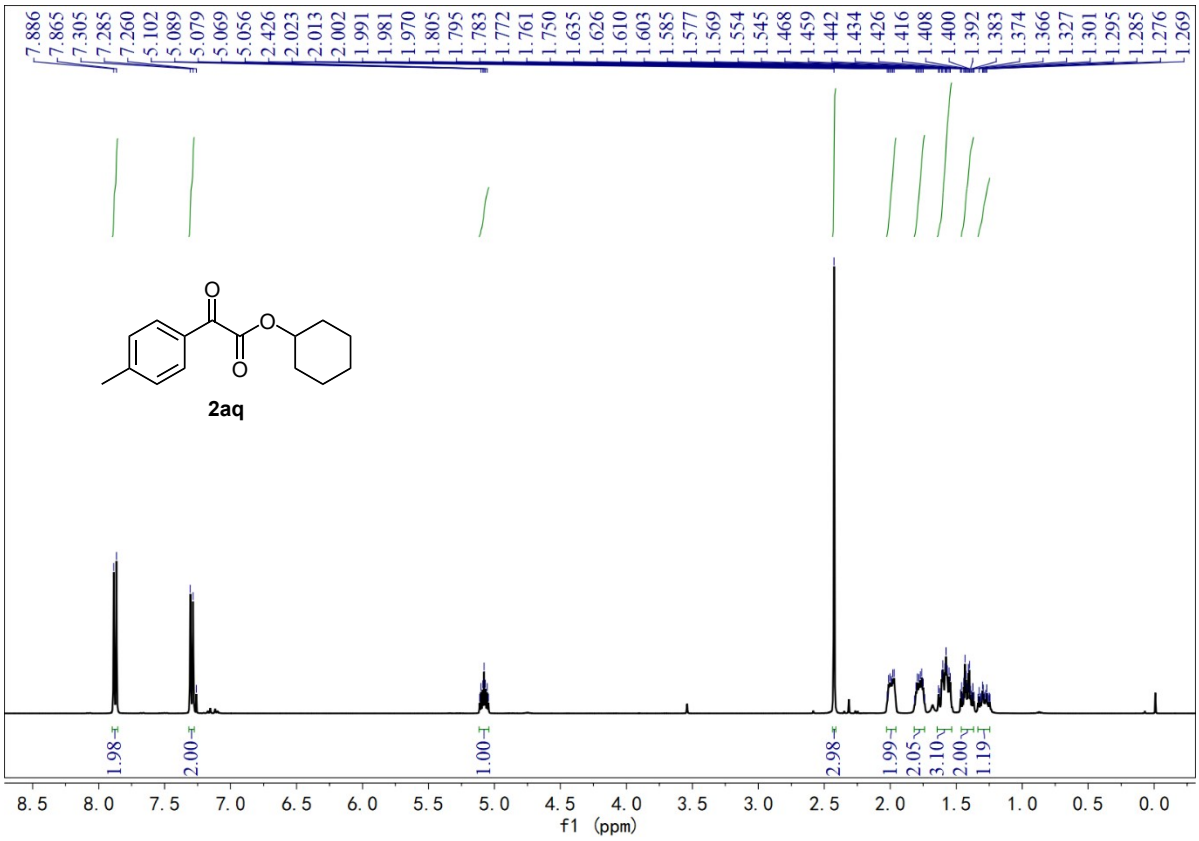


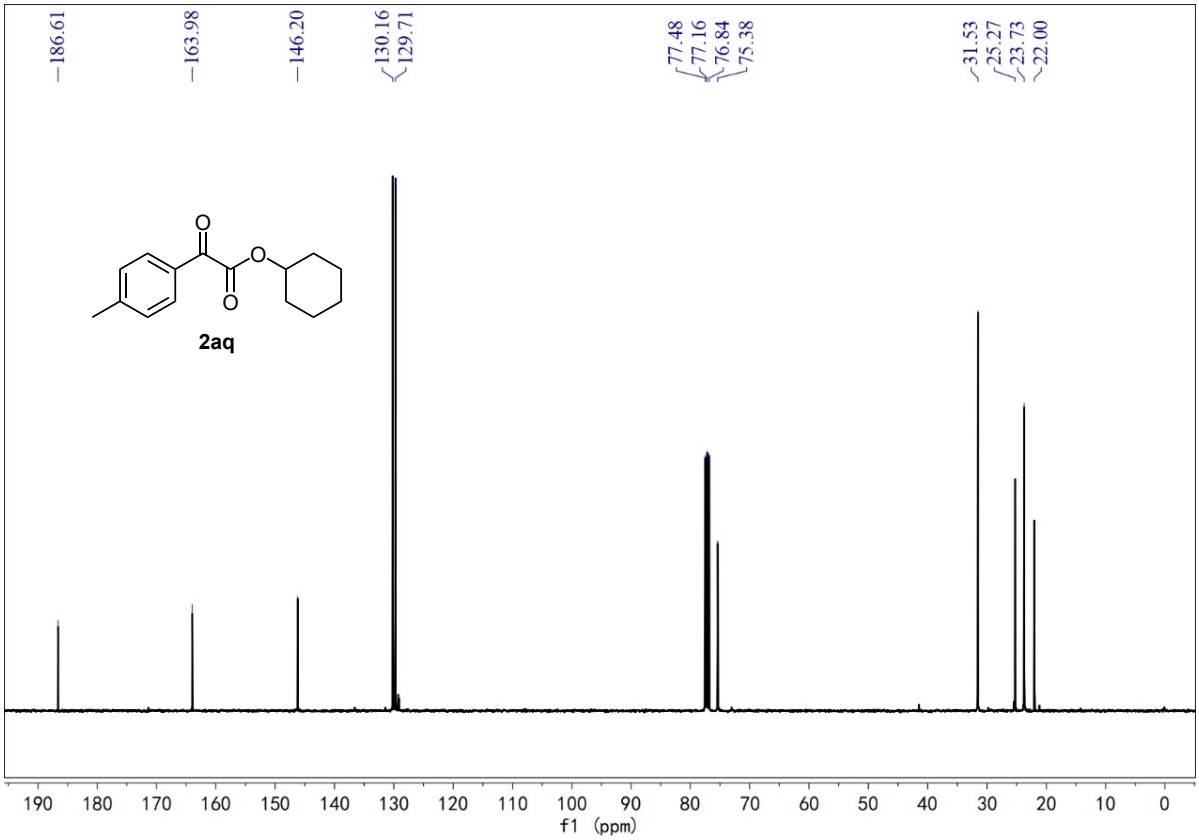
^{1}H and ^{13}C NMR spectra of **2ap**



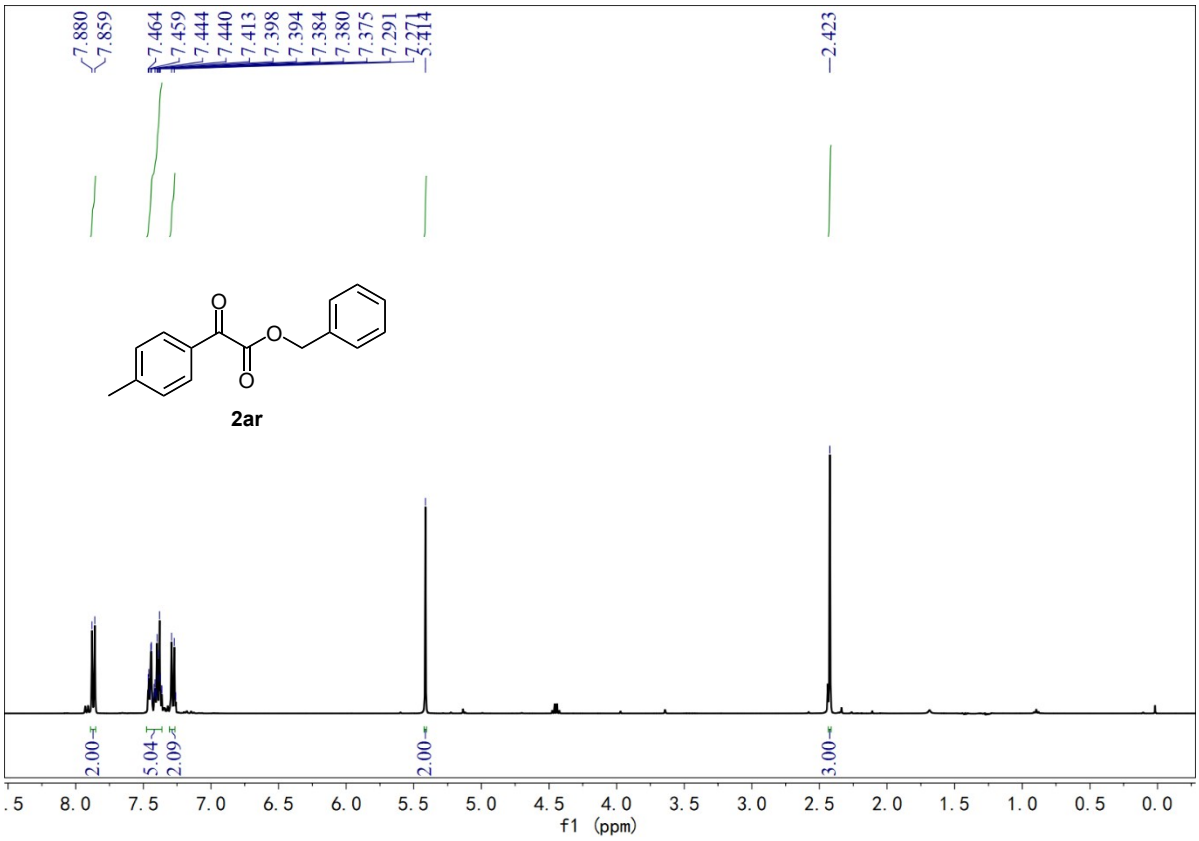


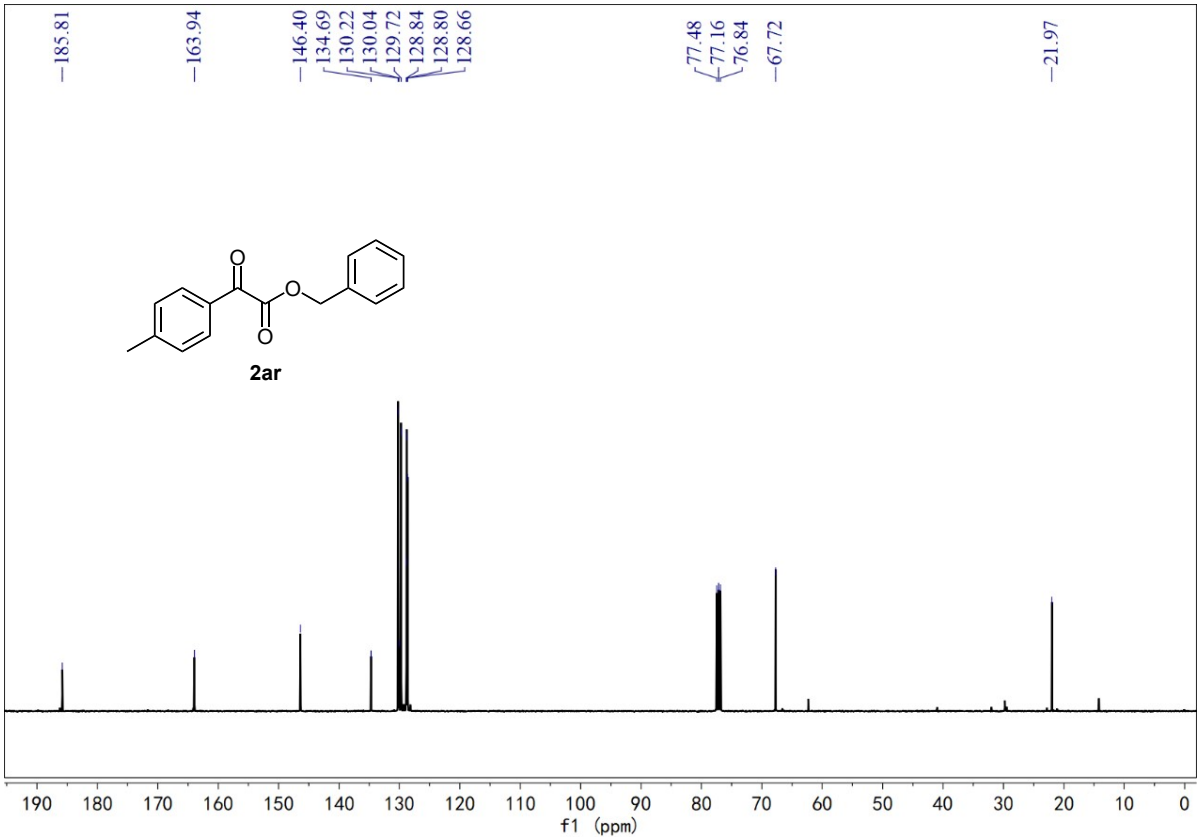
^{1}H and ^{13}C NMR spectra of **2aq**



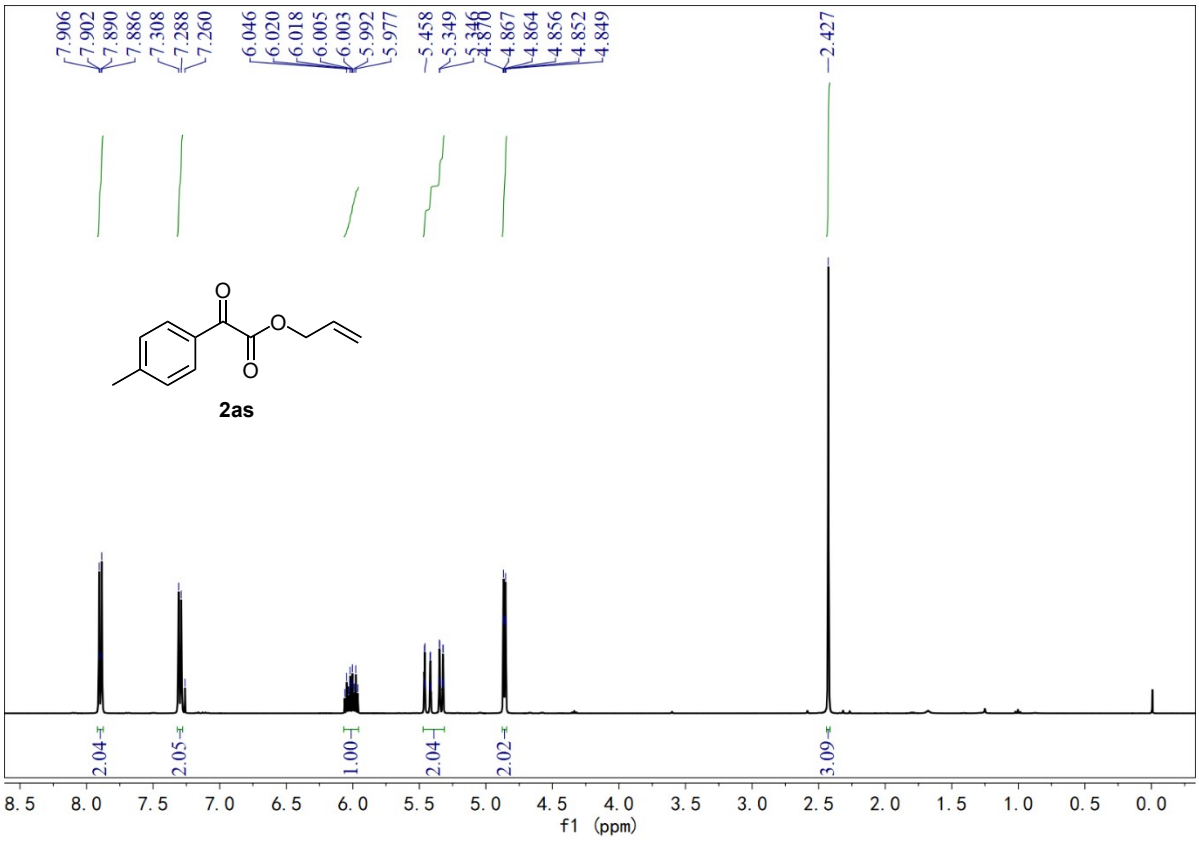


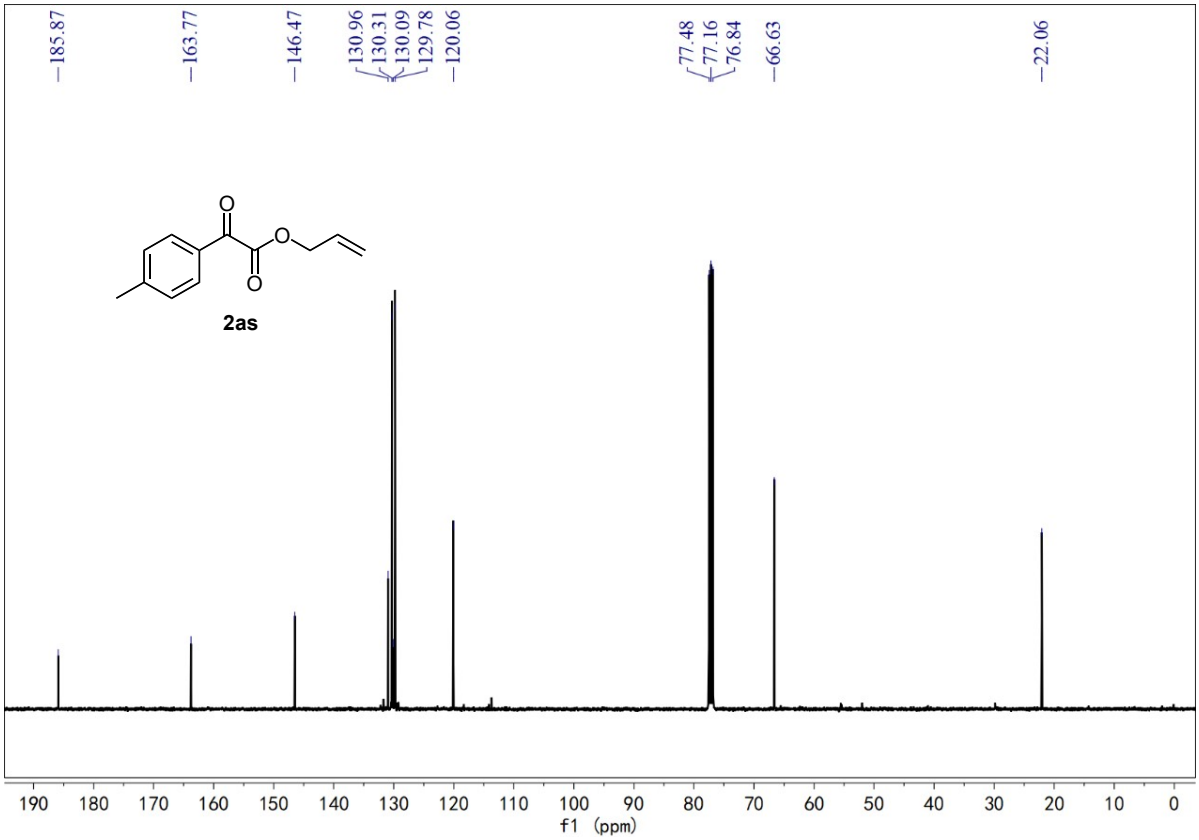
^{1}H and ^{13}C NMR spectra of **2ar**



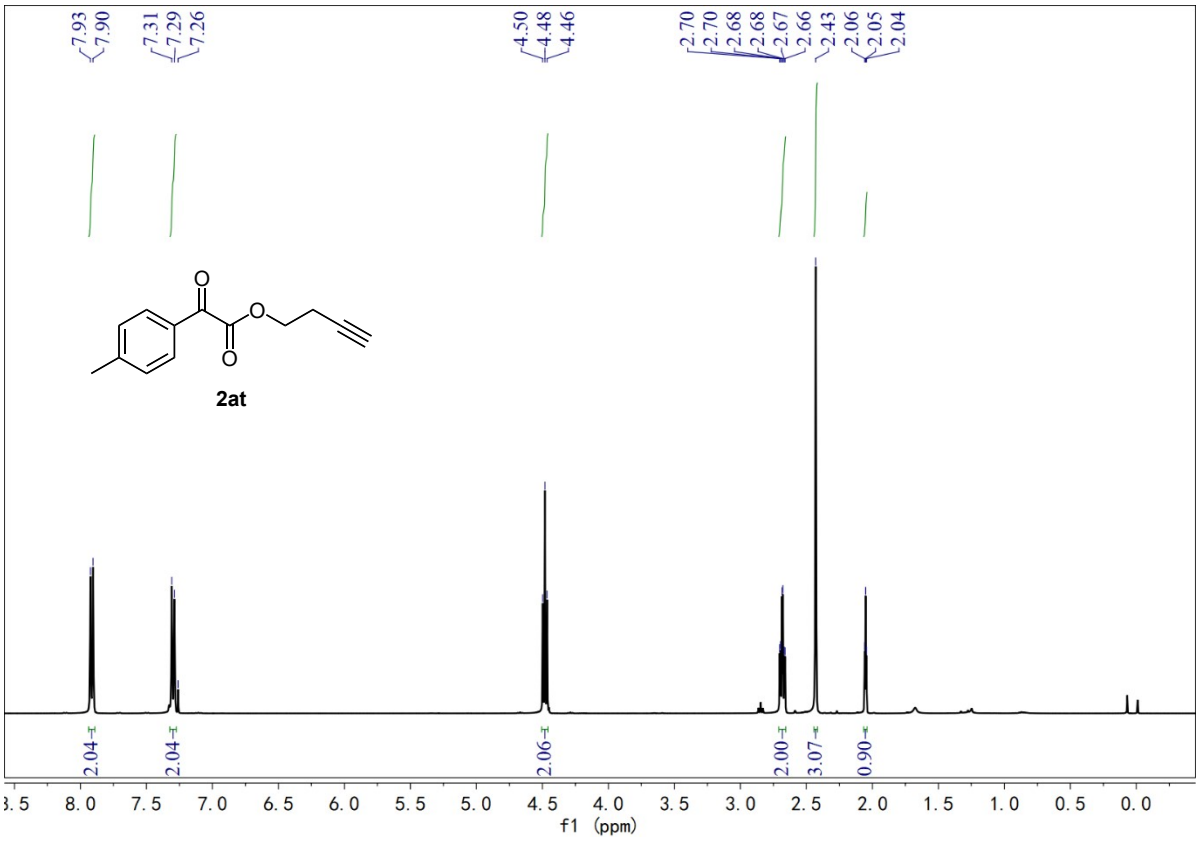


^{1}H and ^{13}C NMR spectra of **2as**

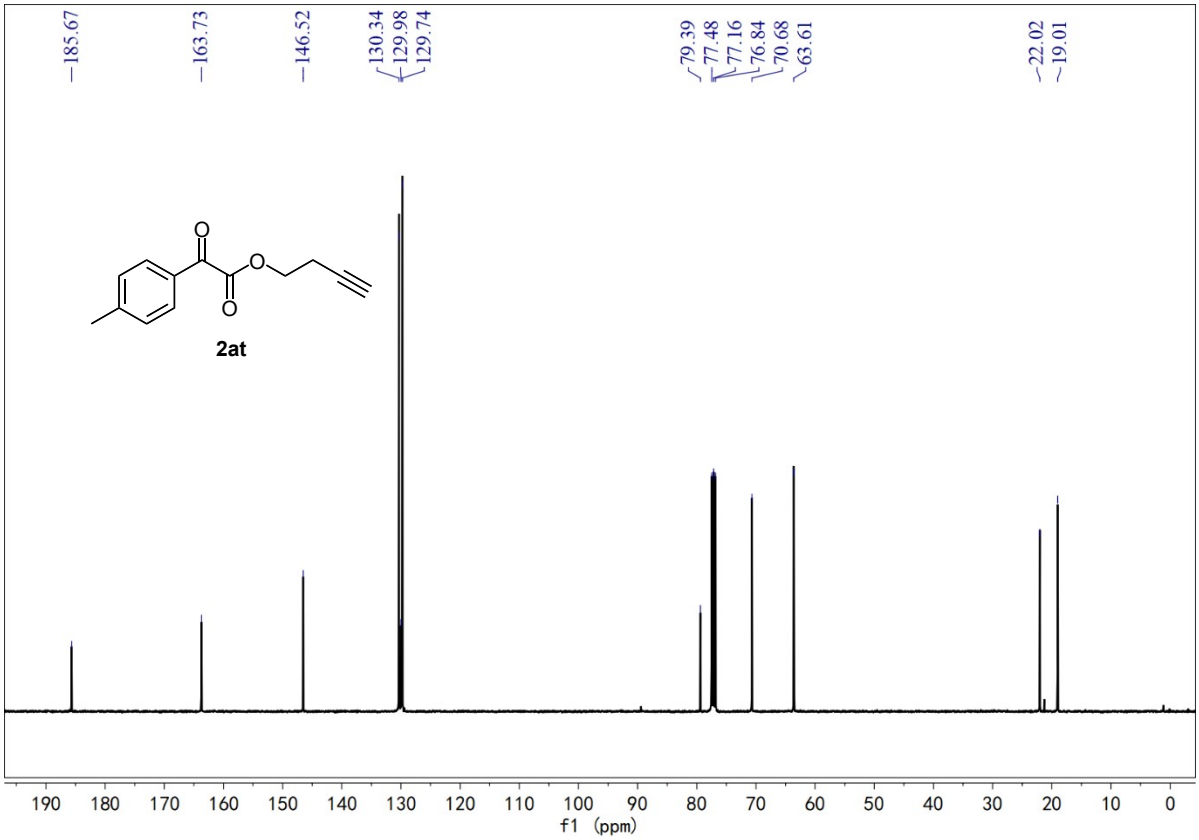




^{1}H and ^{13}C NMR spectra of **2at**

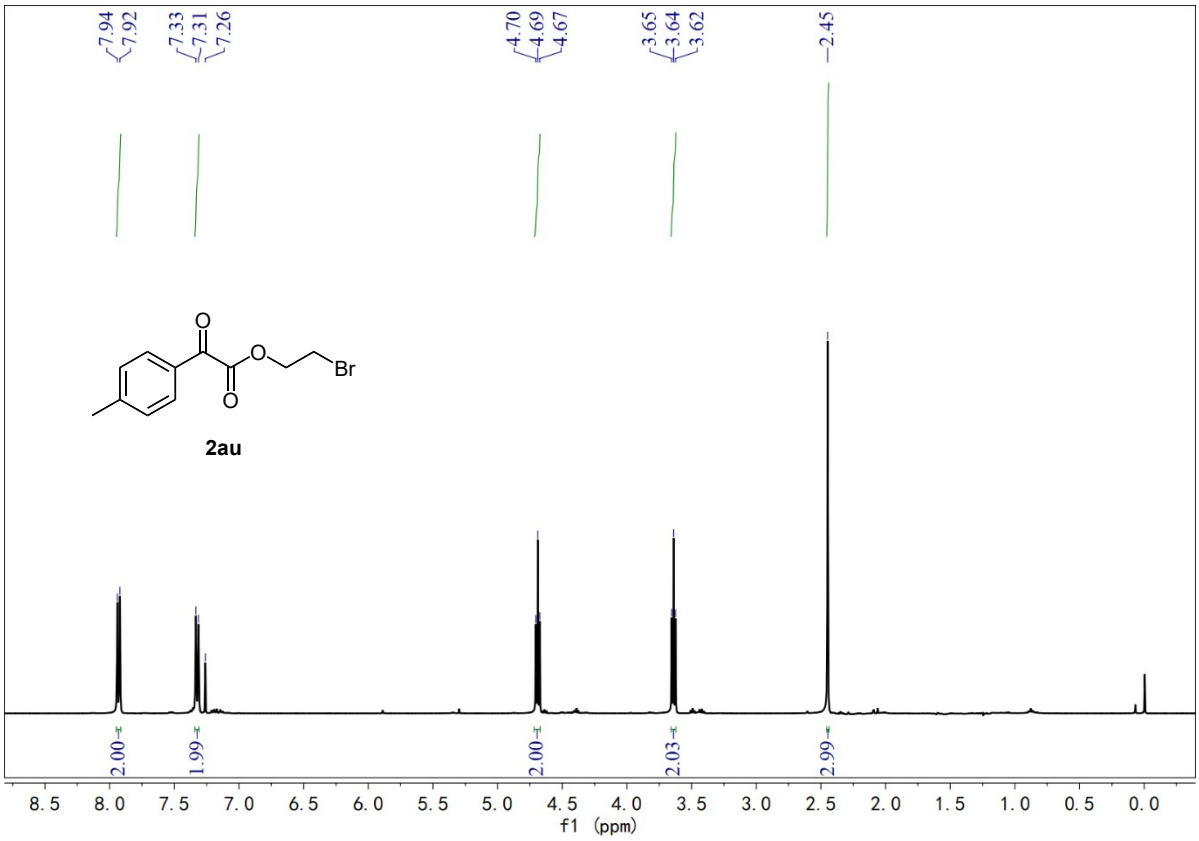


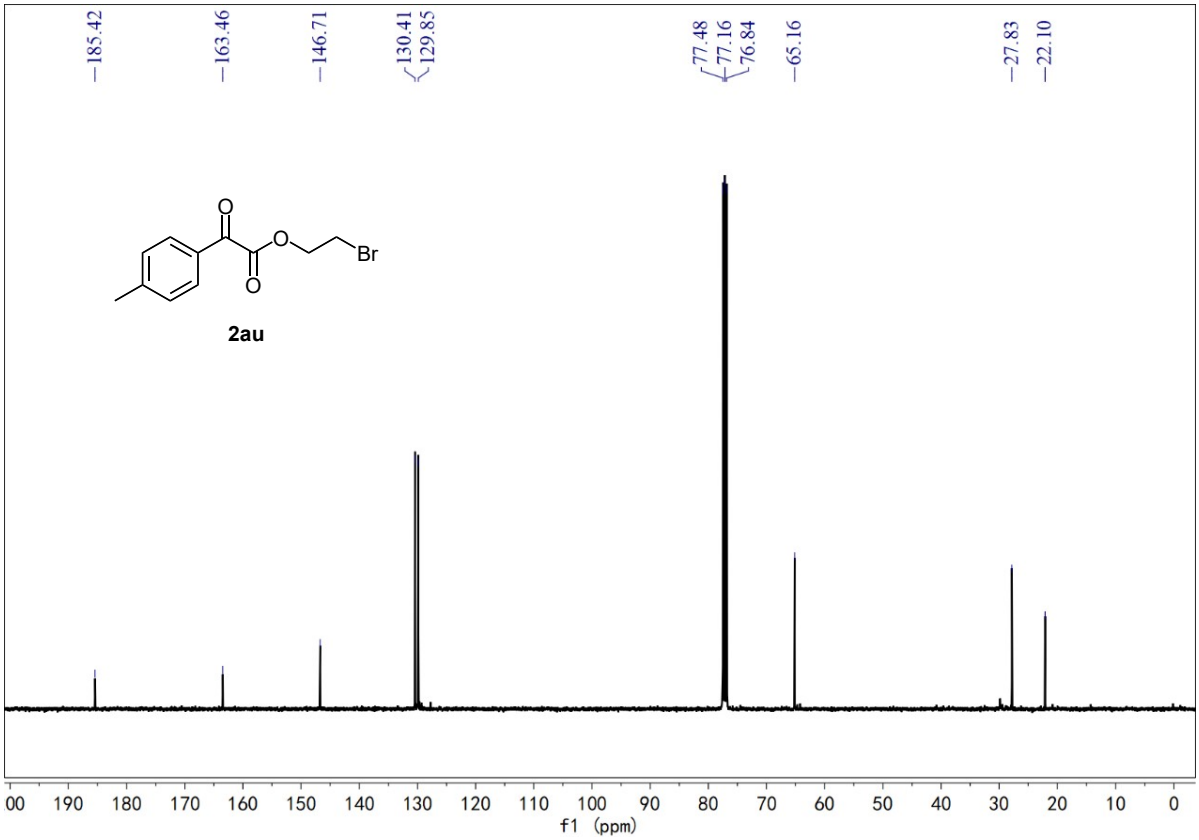
2at



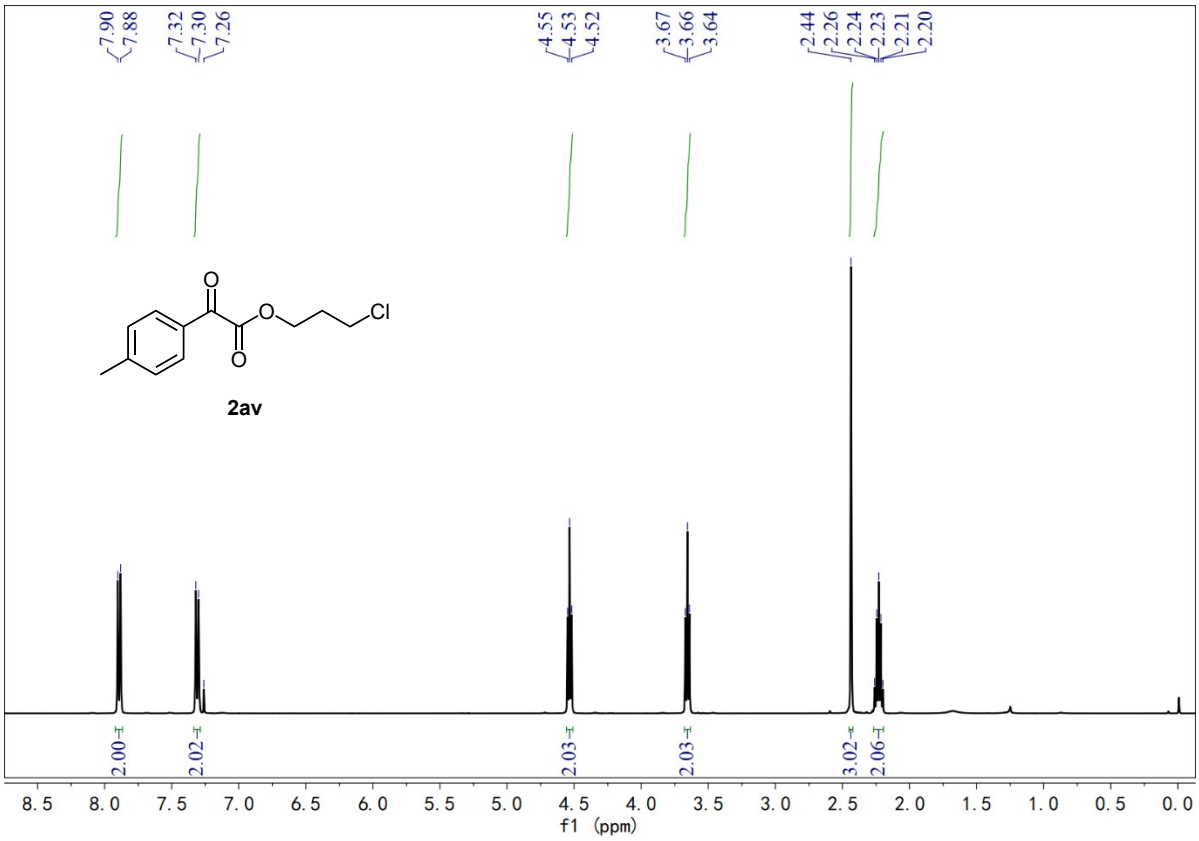
2at

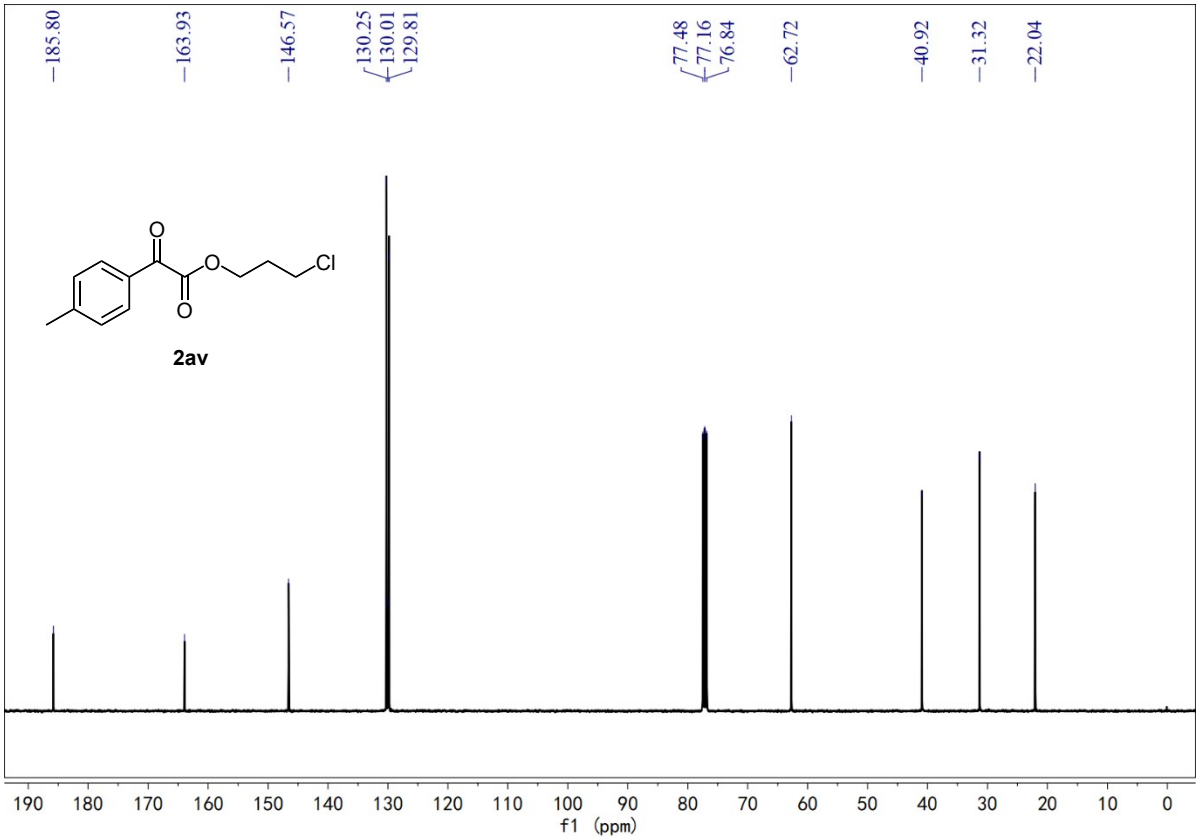
^{1}H and ^{13}C NMR spectra of **2au**



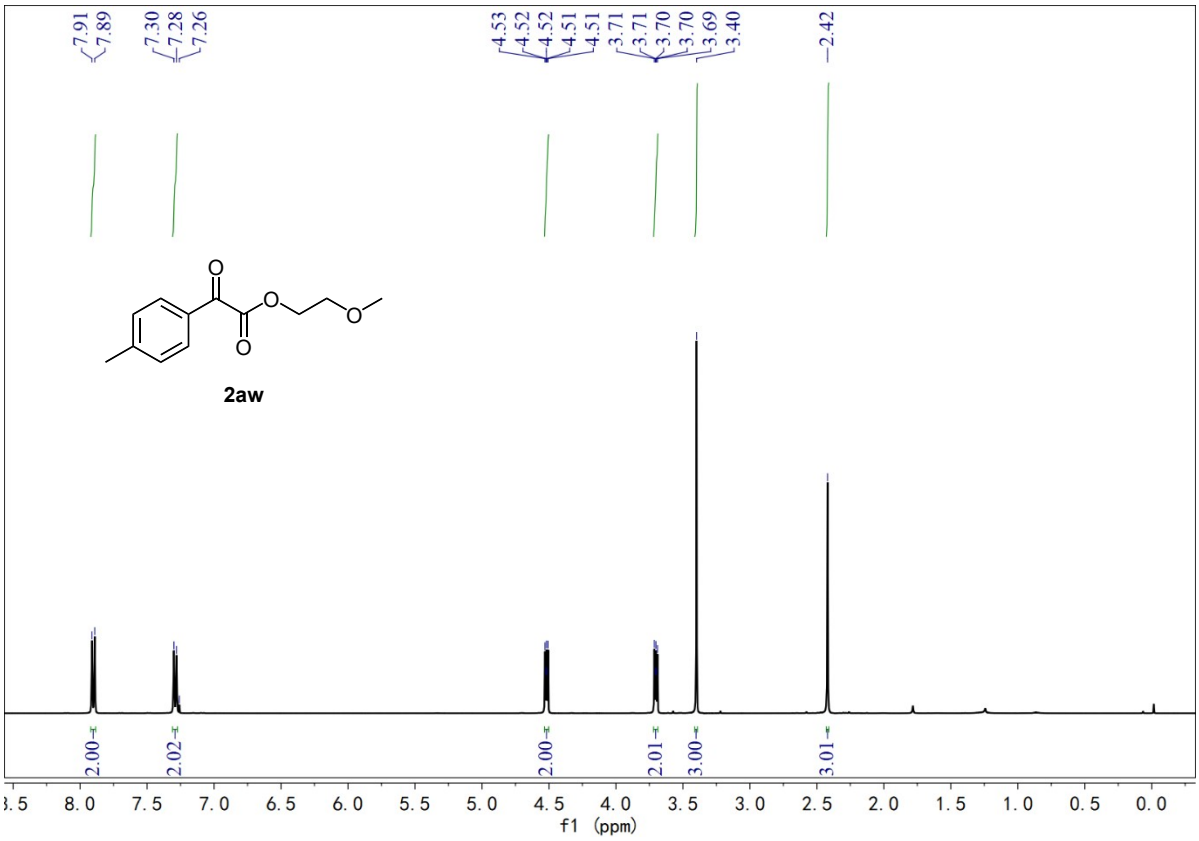


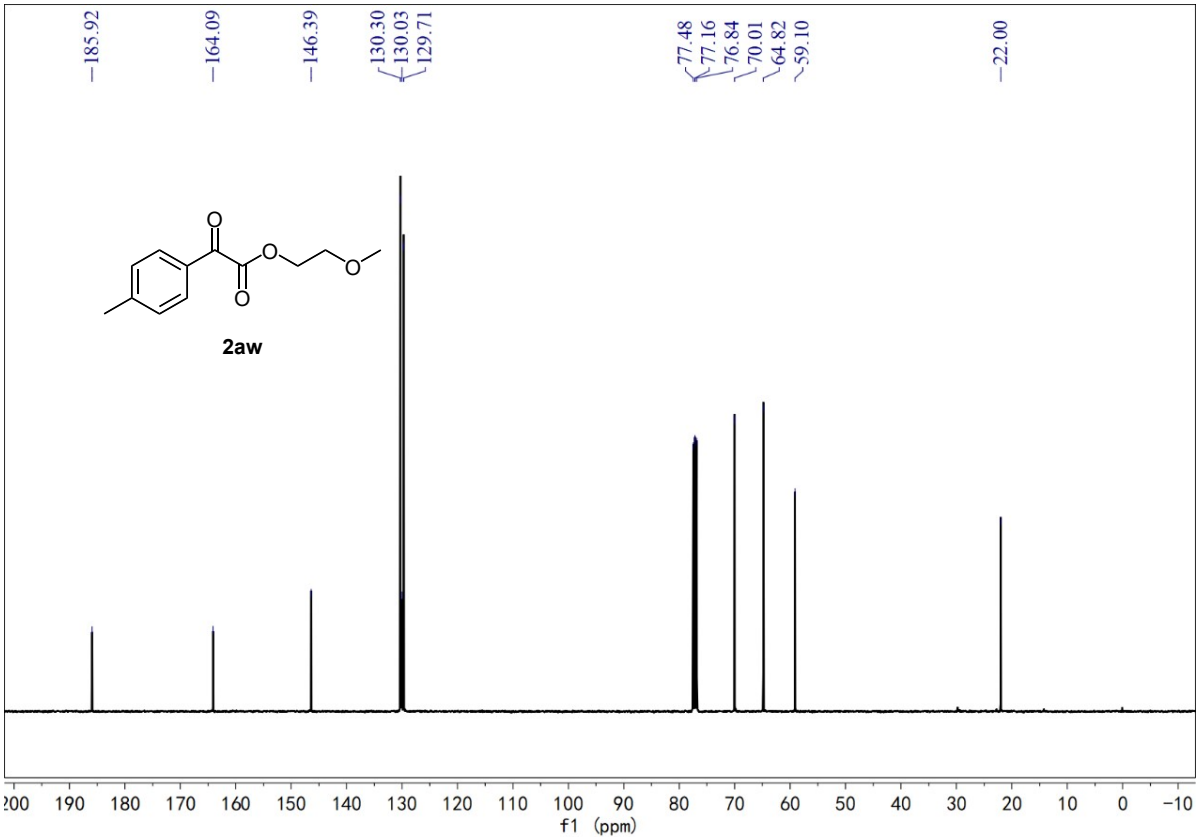
^{1}H and ^{13}C NMR spectra of **2av**



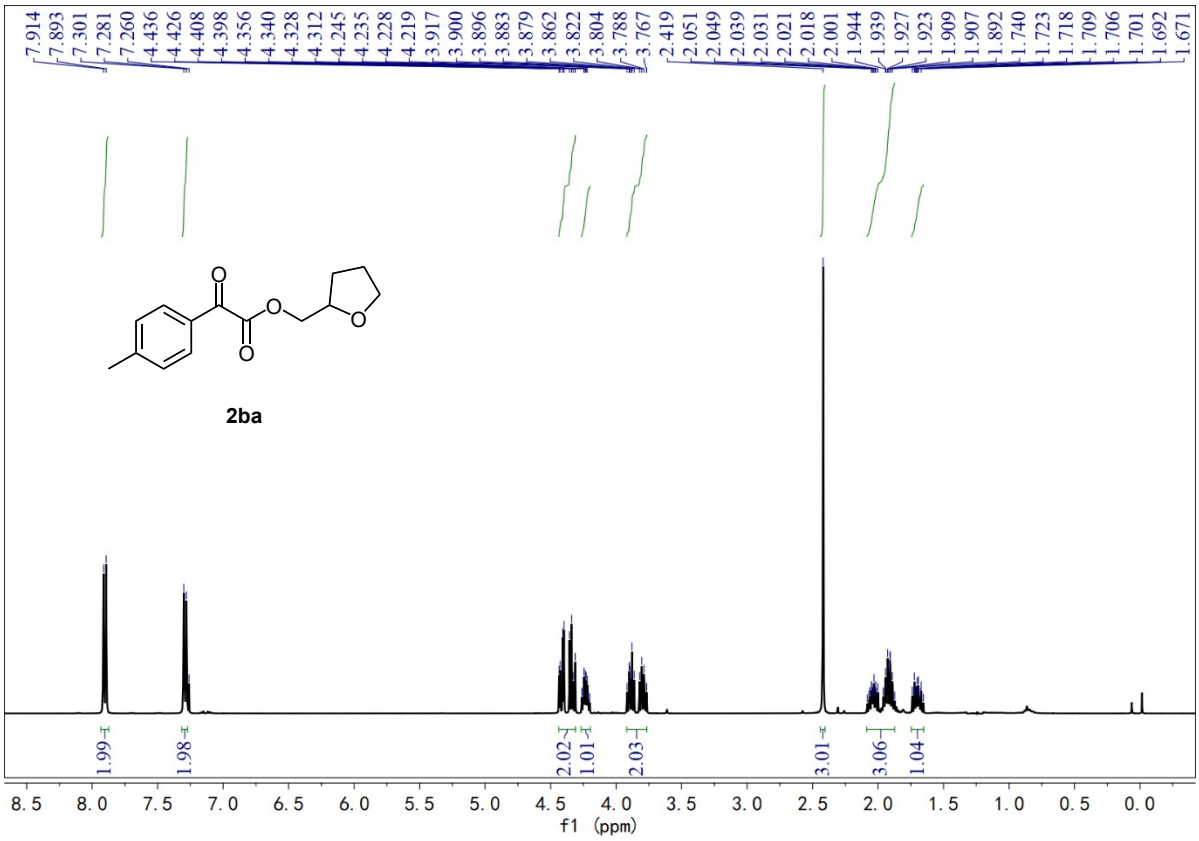


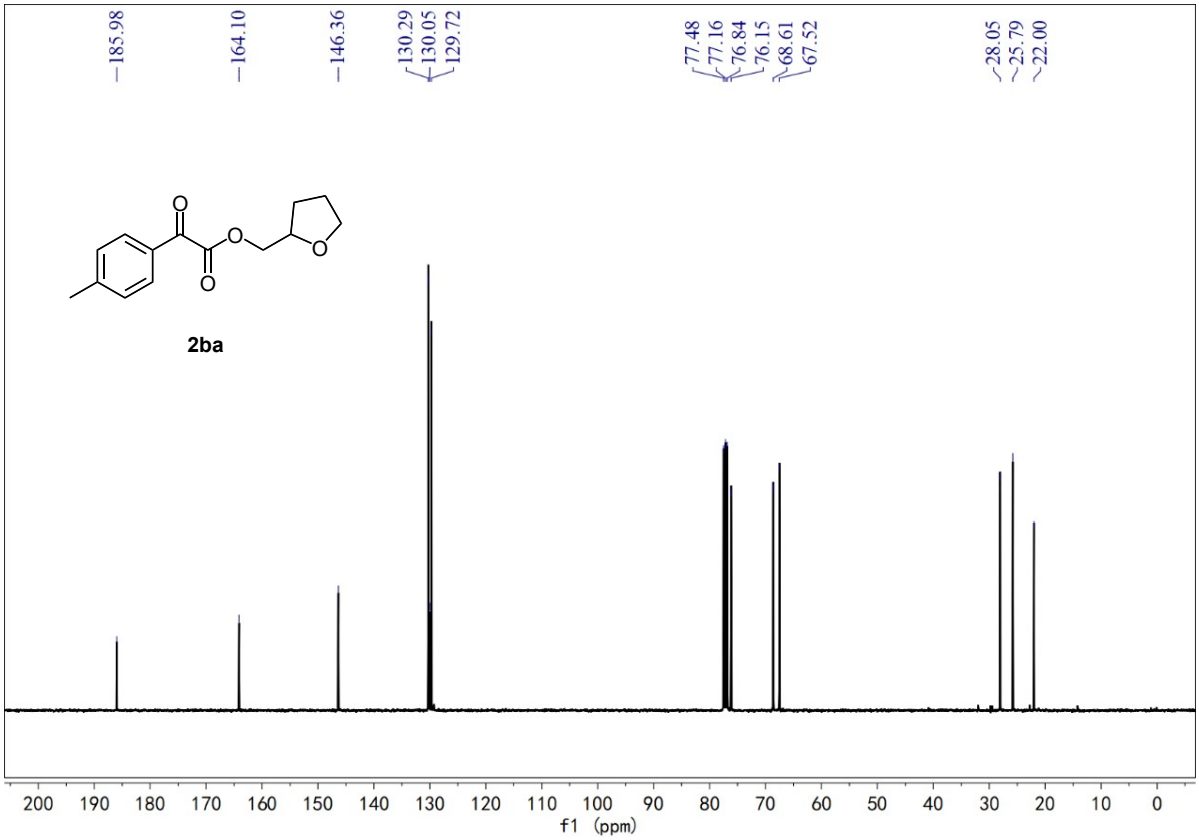
^{1}H and ^{13}C NMR spectra of **2aw**



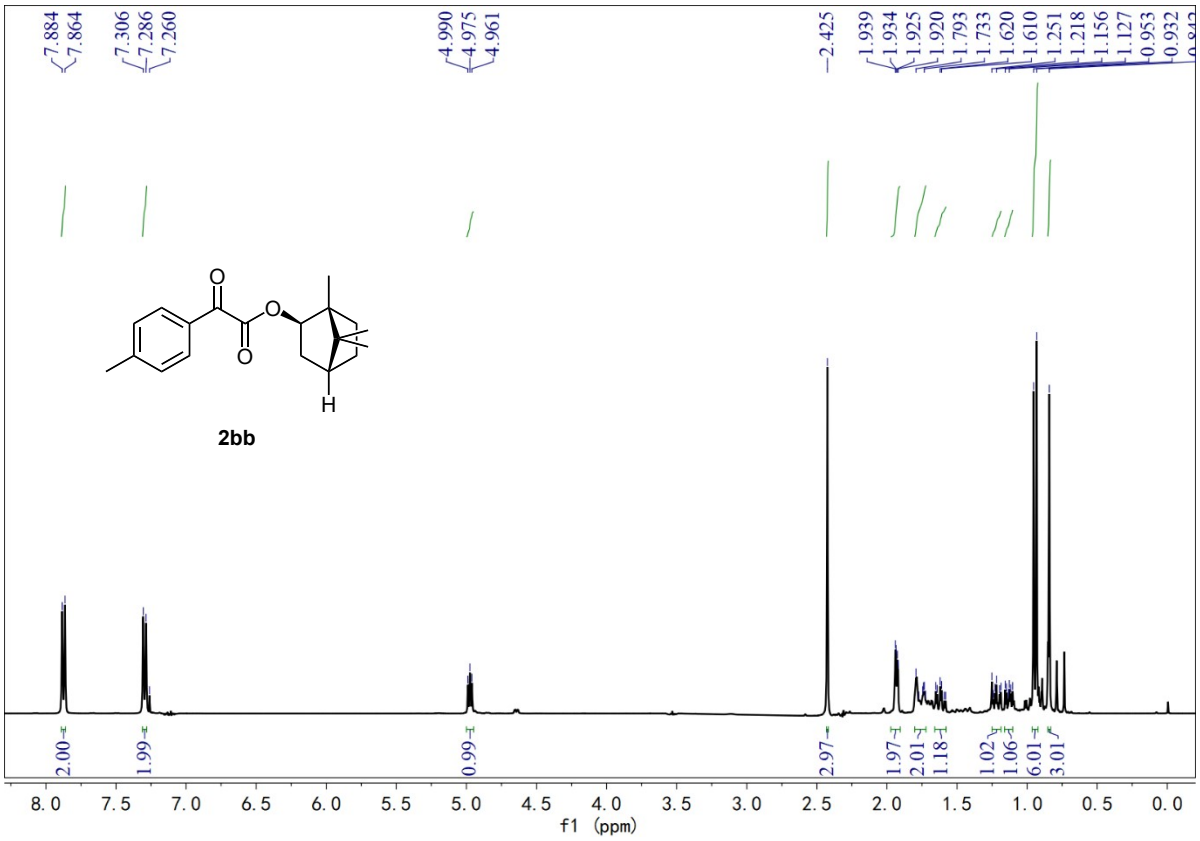


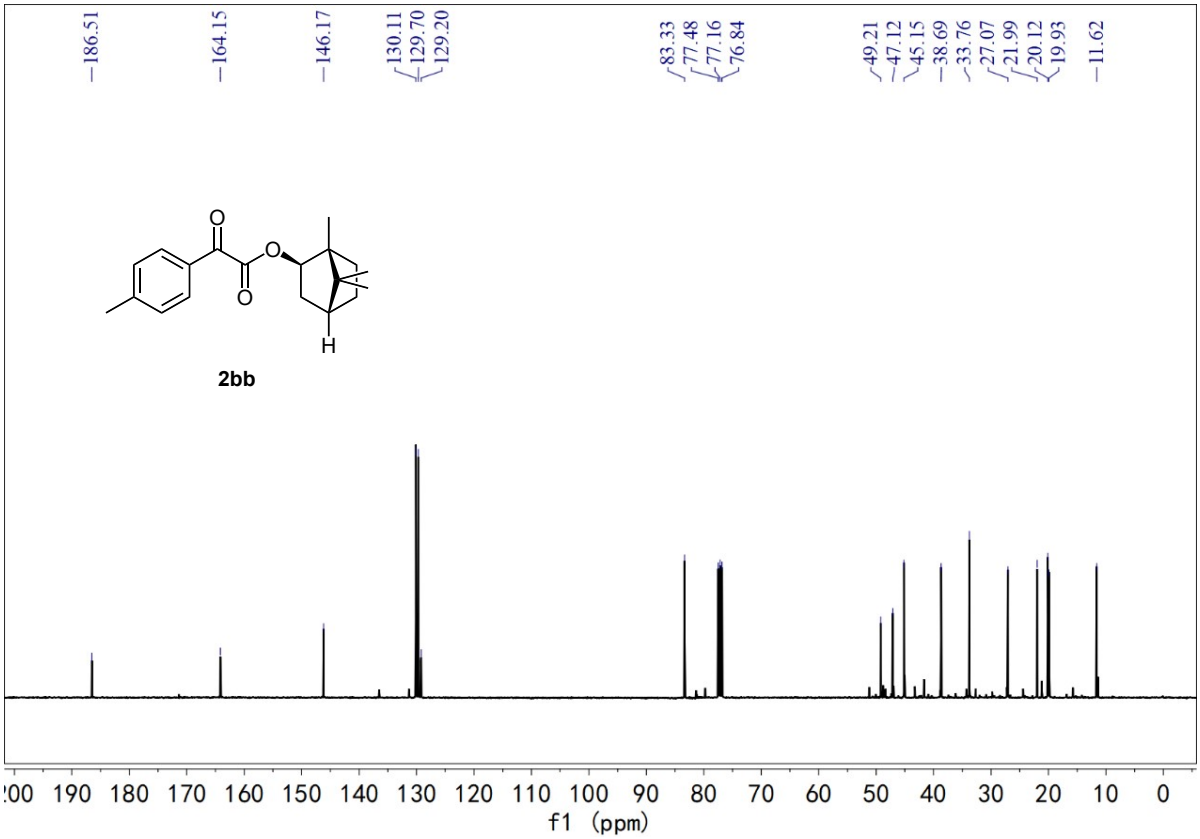
^{1}H and ^{13}C NMR spectra of **2ba**



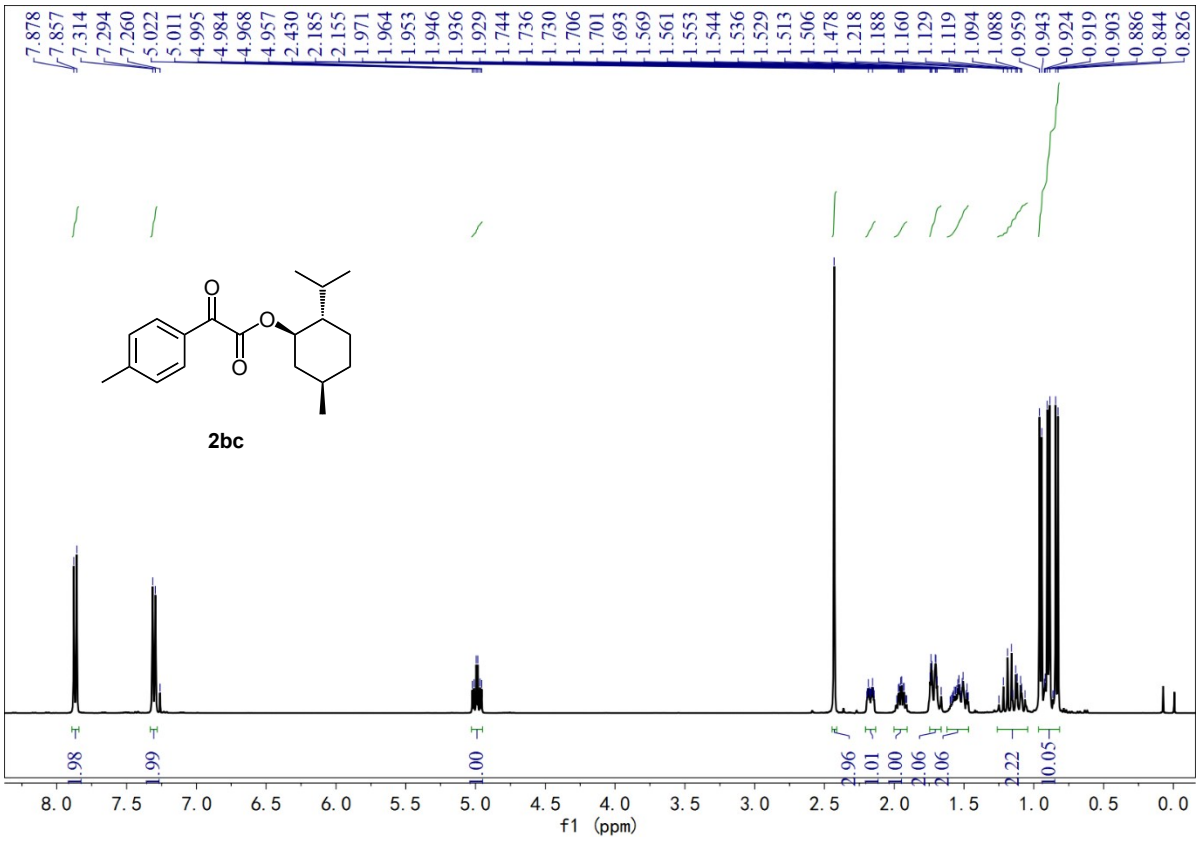


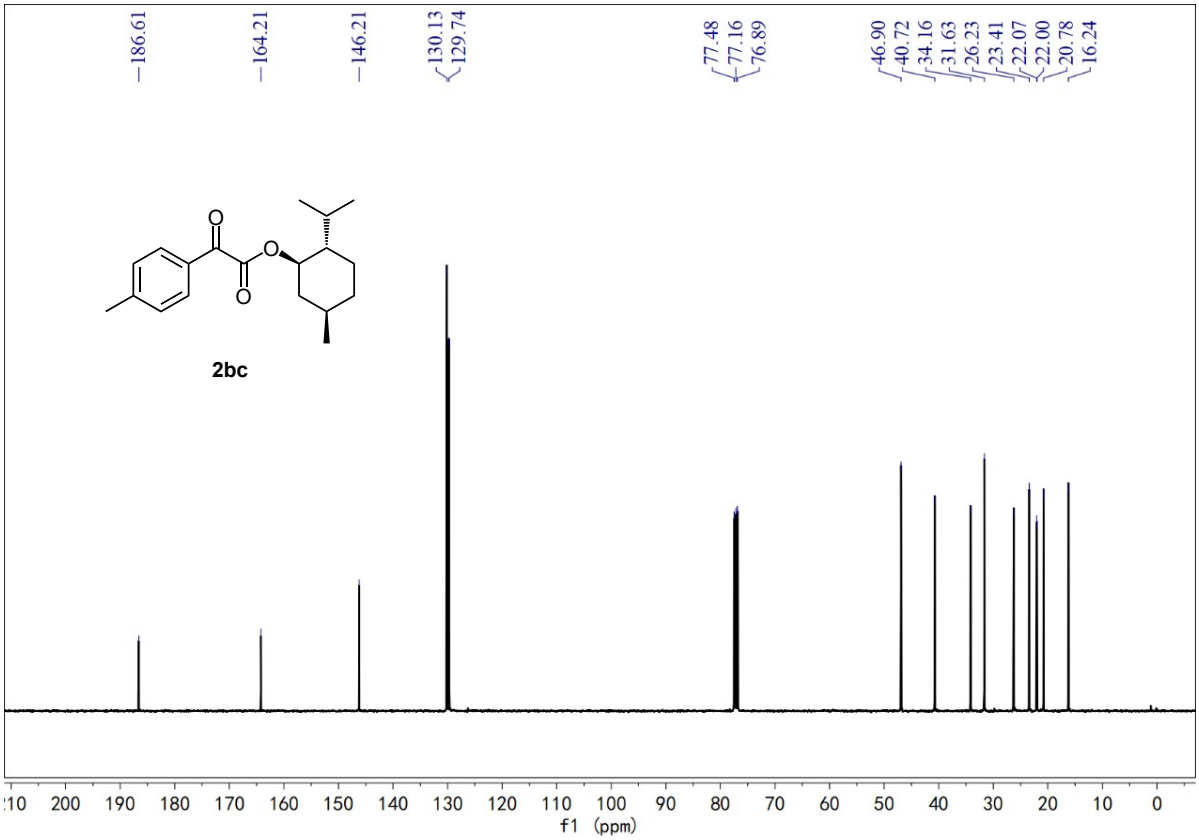
^{1}H and ^{13}C NMR spectra of **2bb**



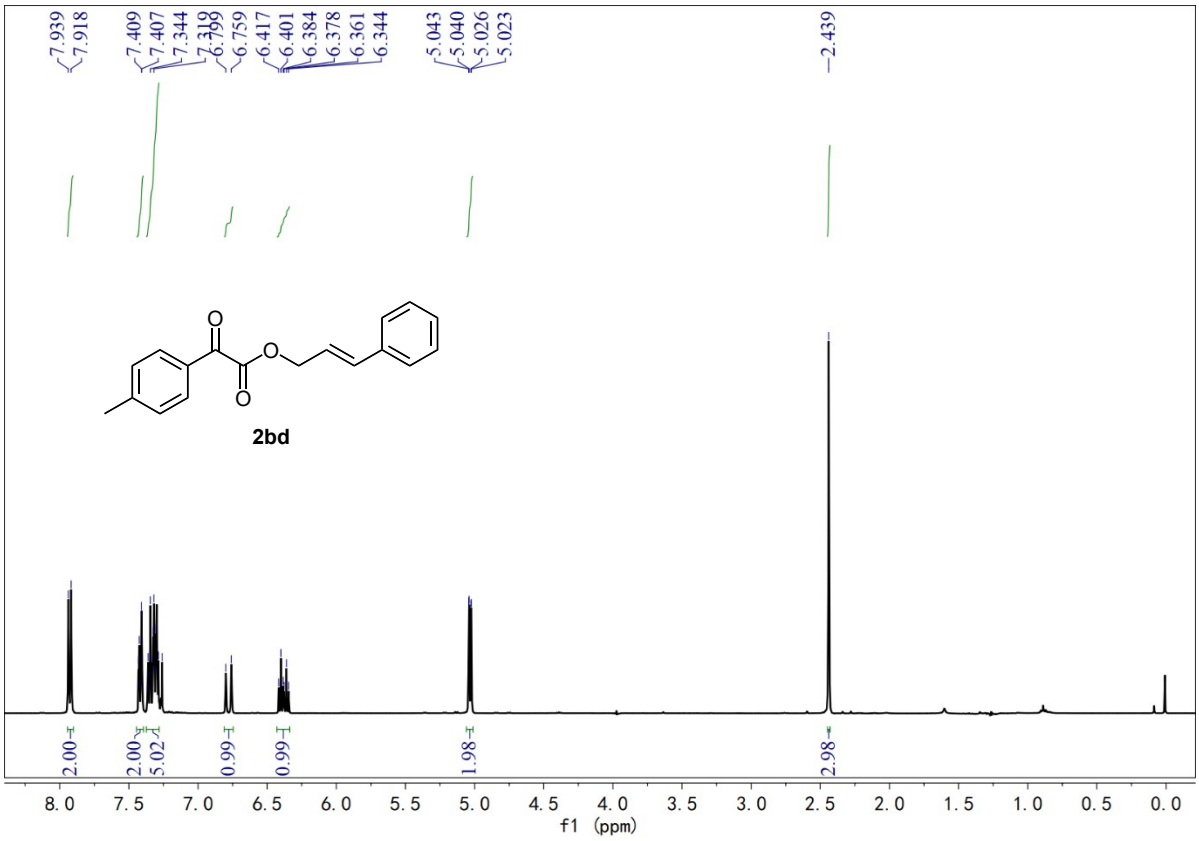


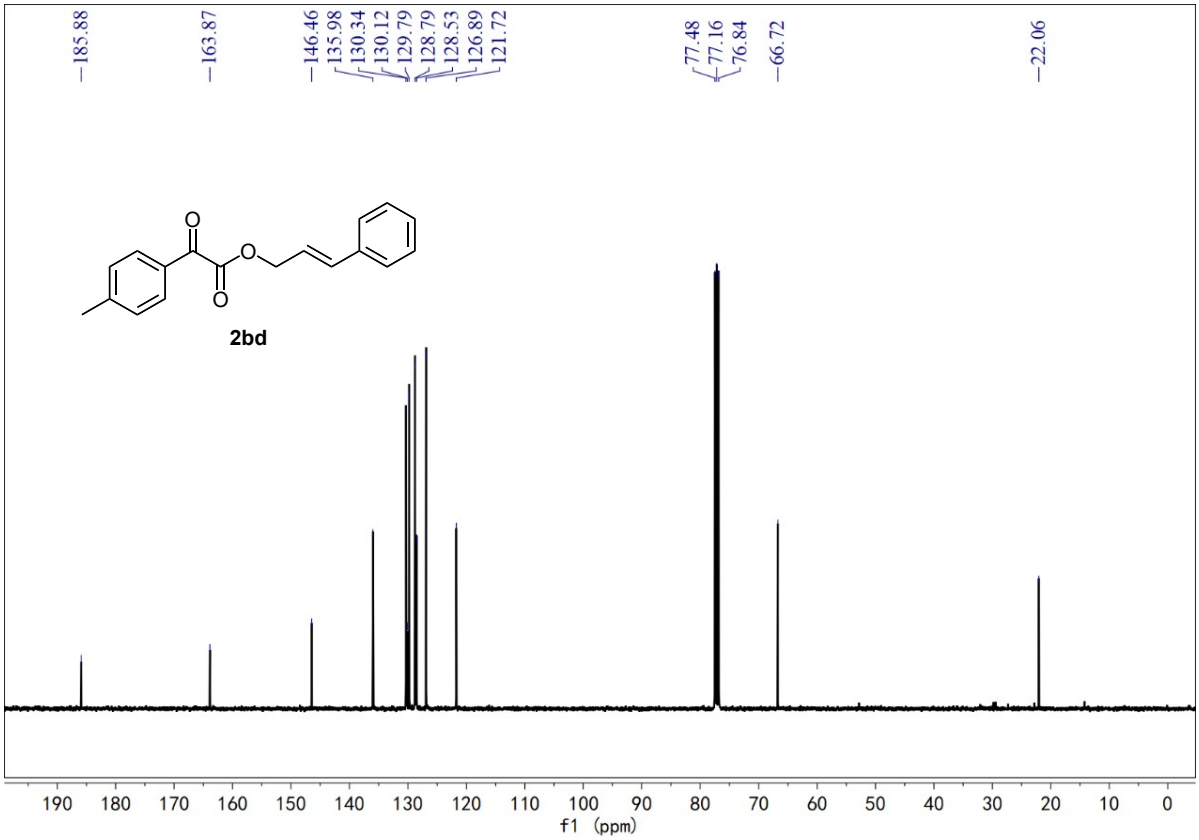
^{1}H and ^{13}C NMR spectra of **2bc**



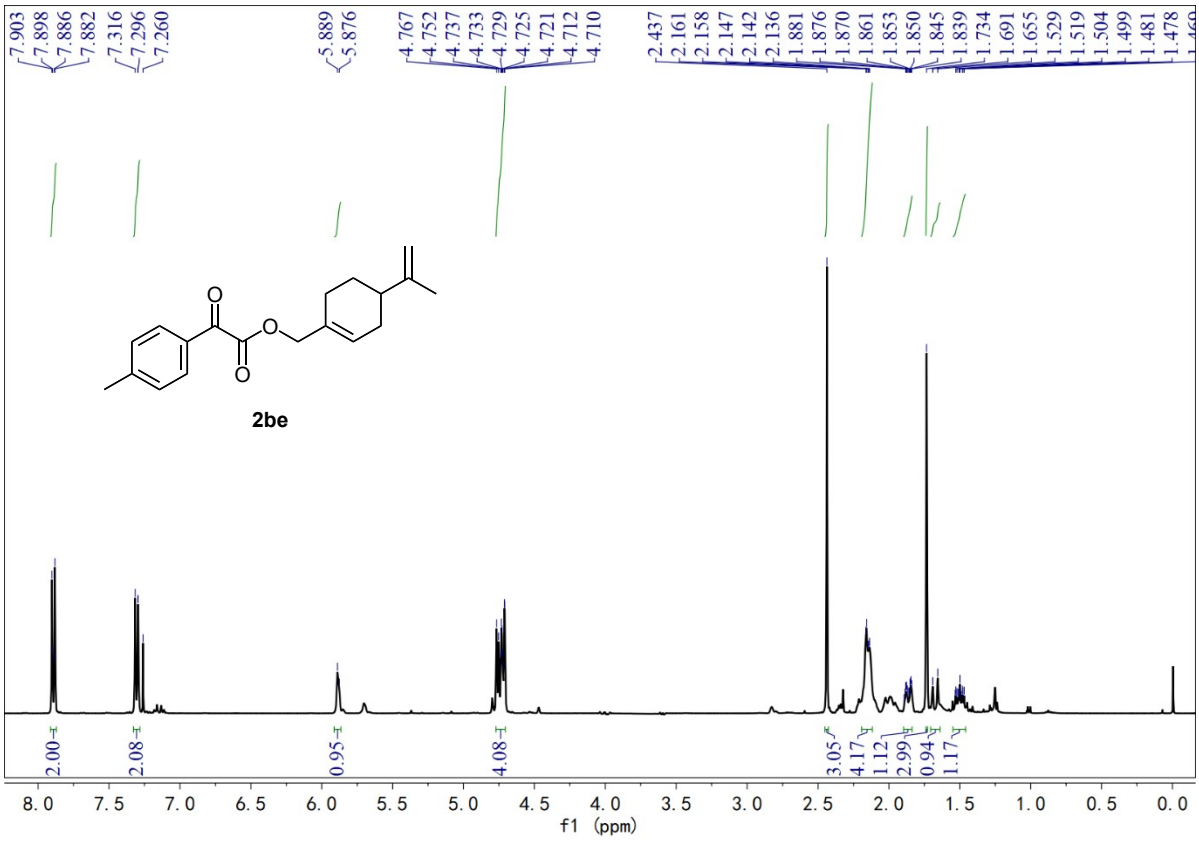


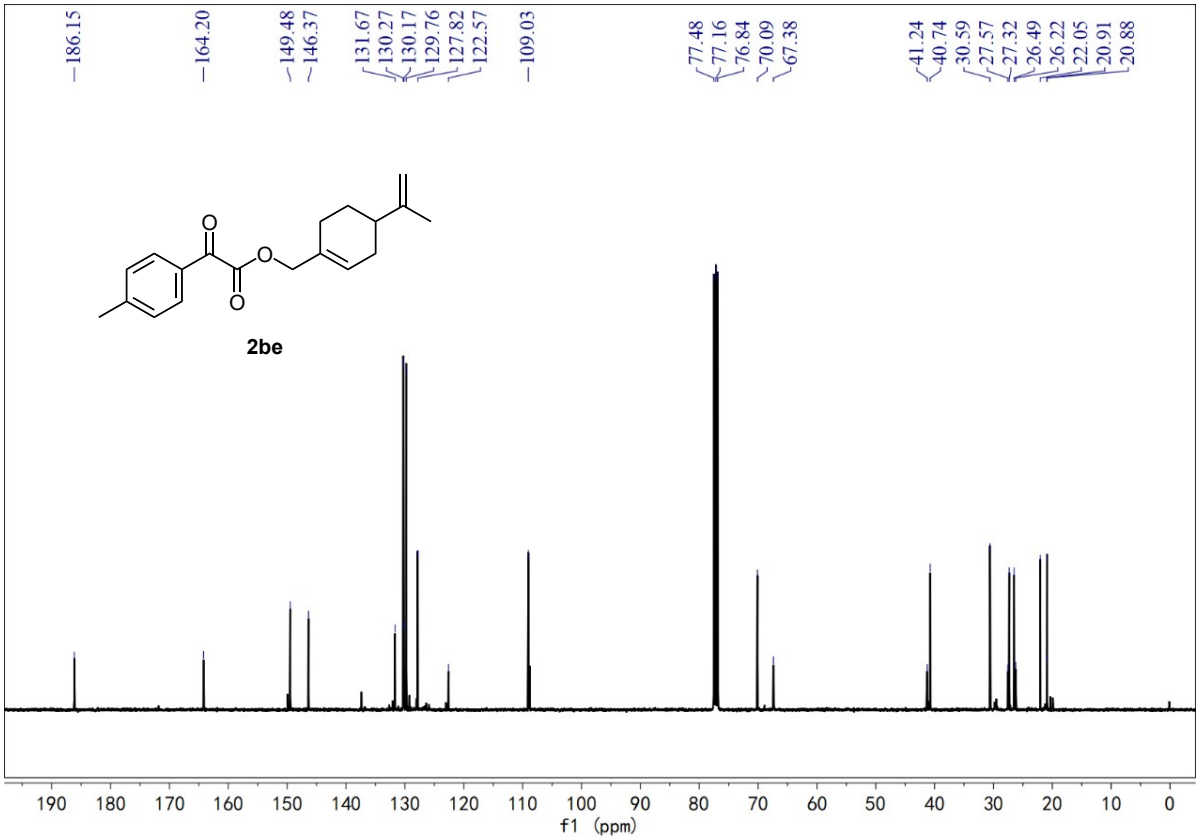
^{1}H and ^{13}C NMR spectra of **2bd**



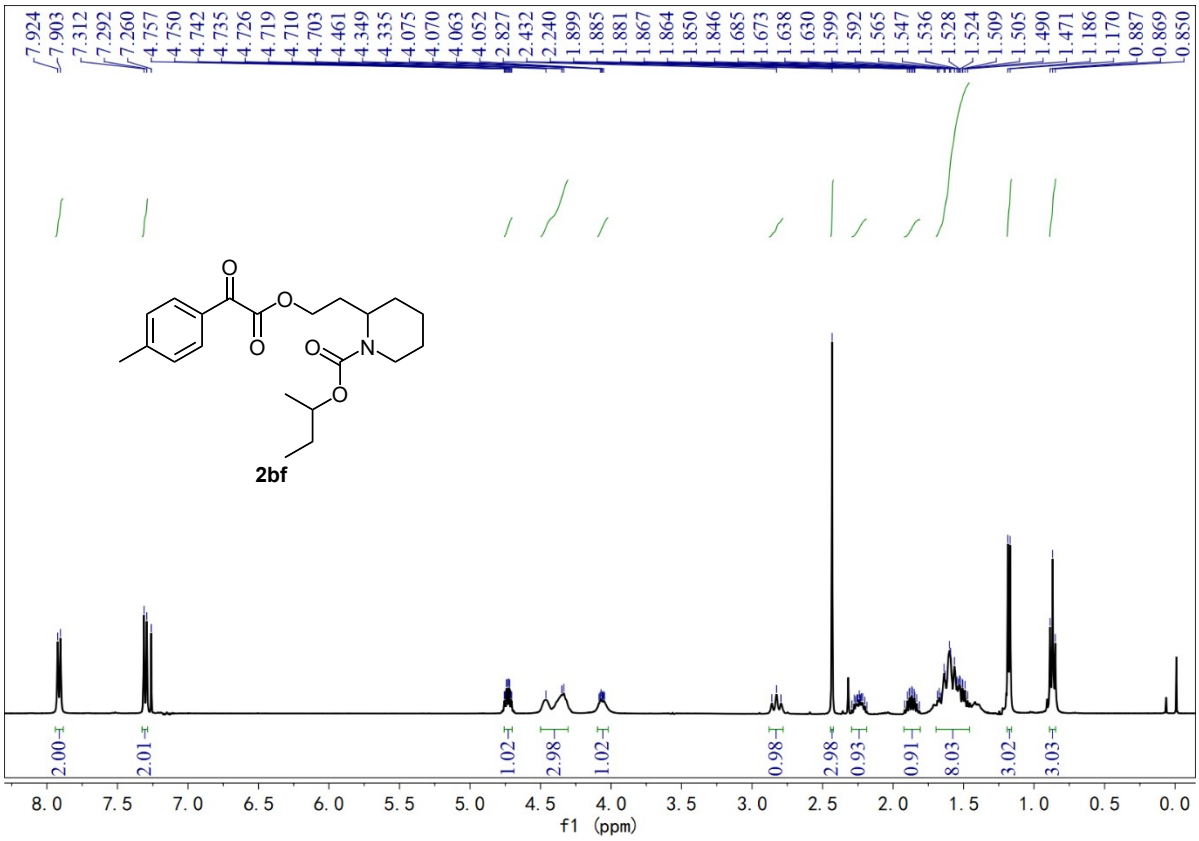


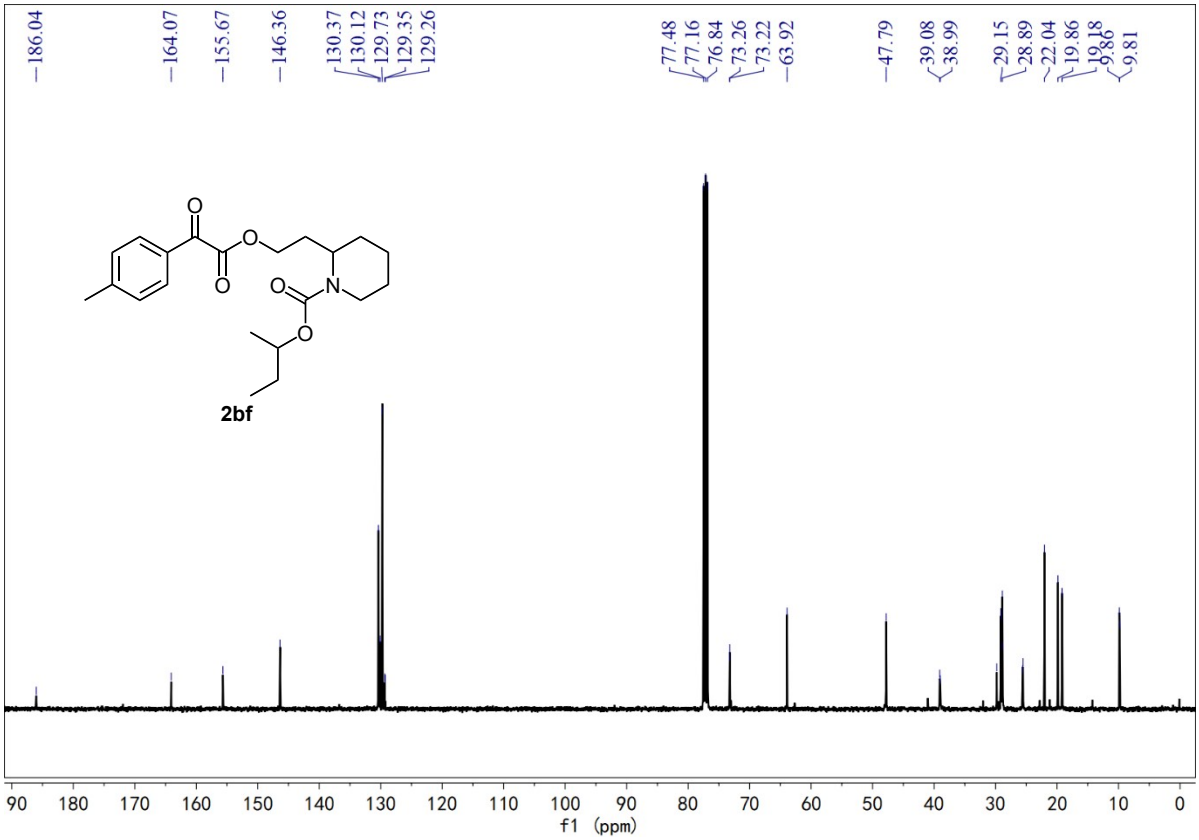
^{1}H and ^{13}C NMR spectra of **2be**



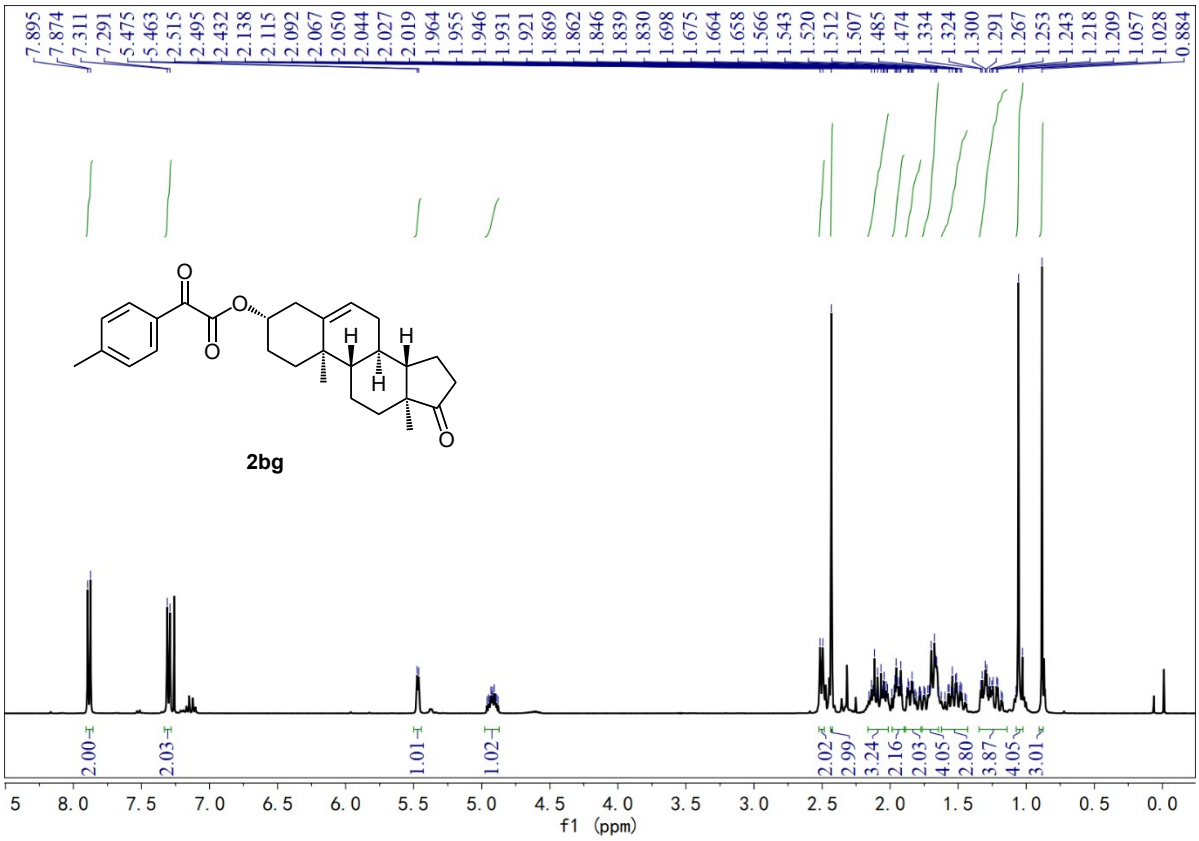


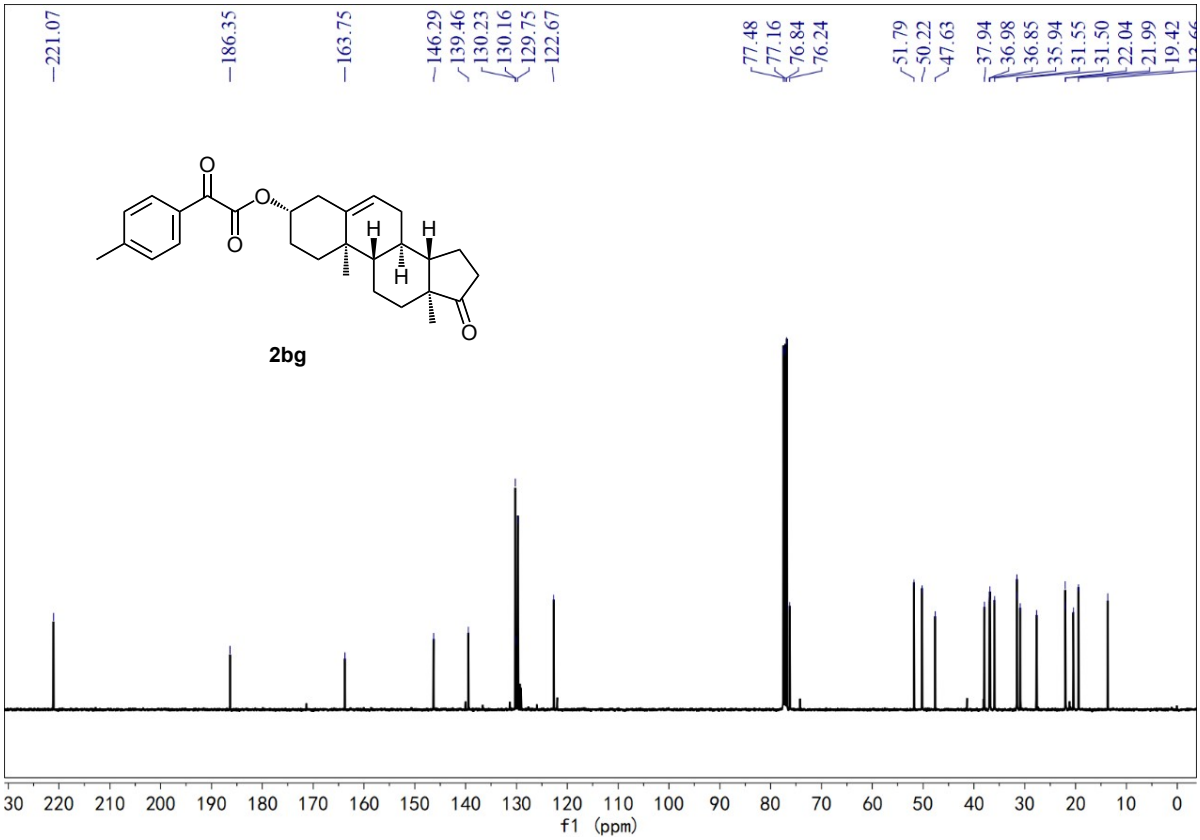
^{1}H and ^{13}C NMR spectra of **2bf**



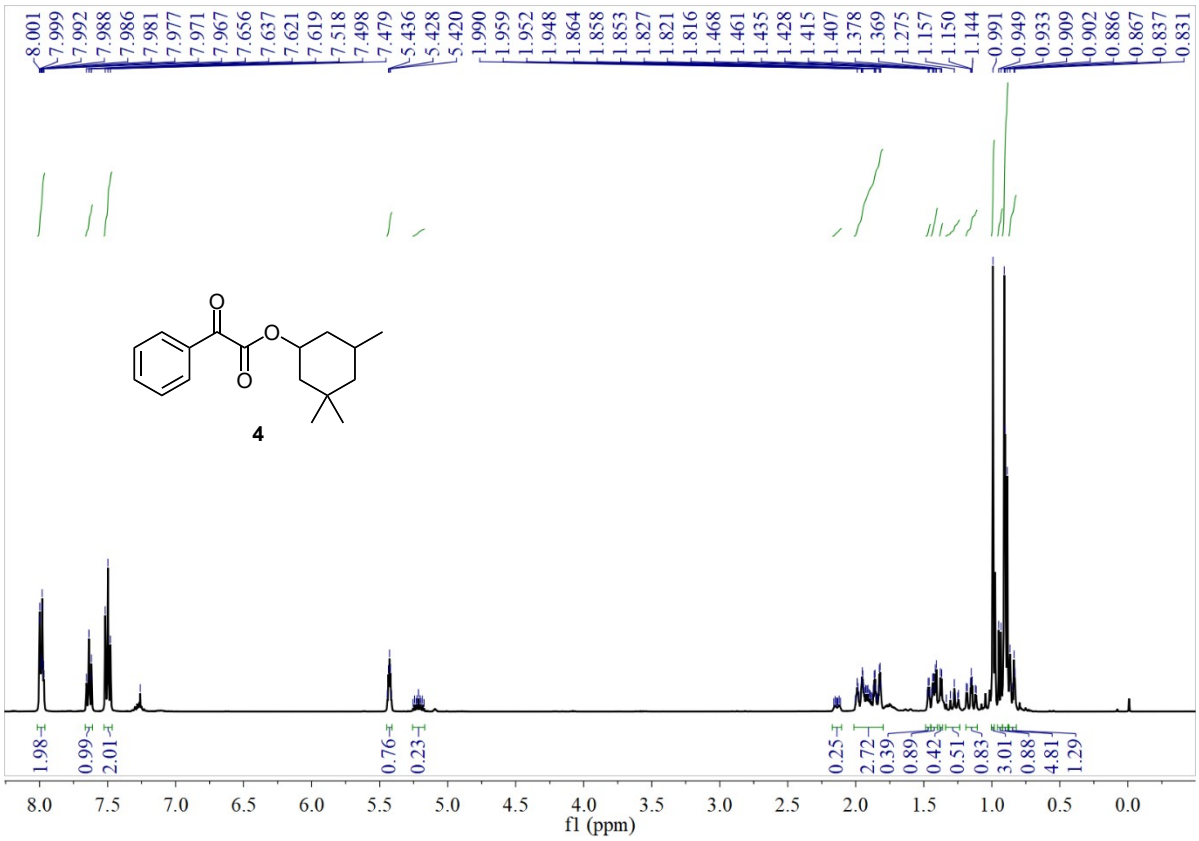


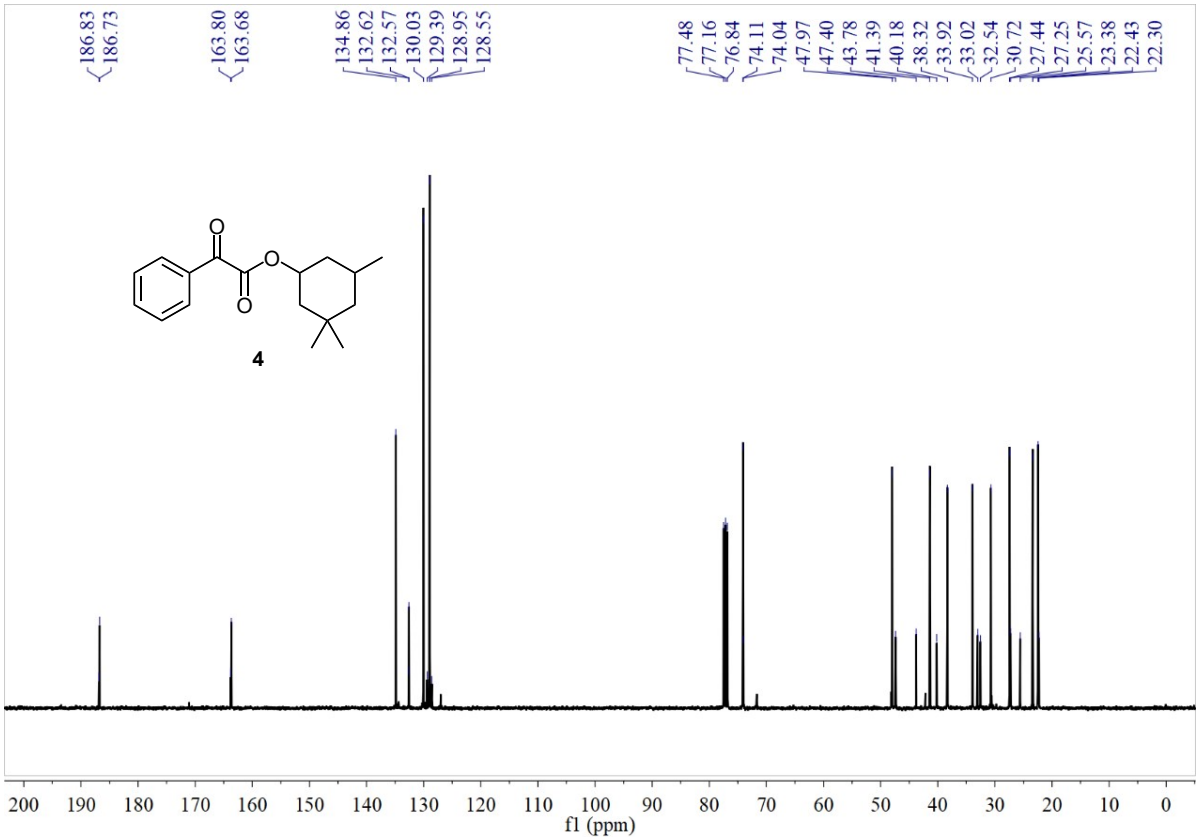
^{1}H and ^{13}C NMR spectra of **2bg**



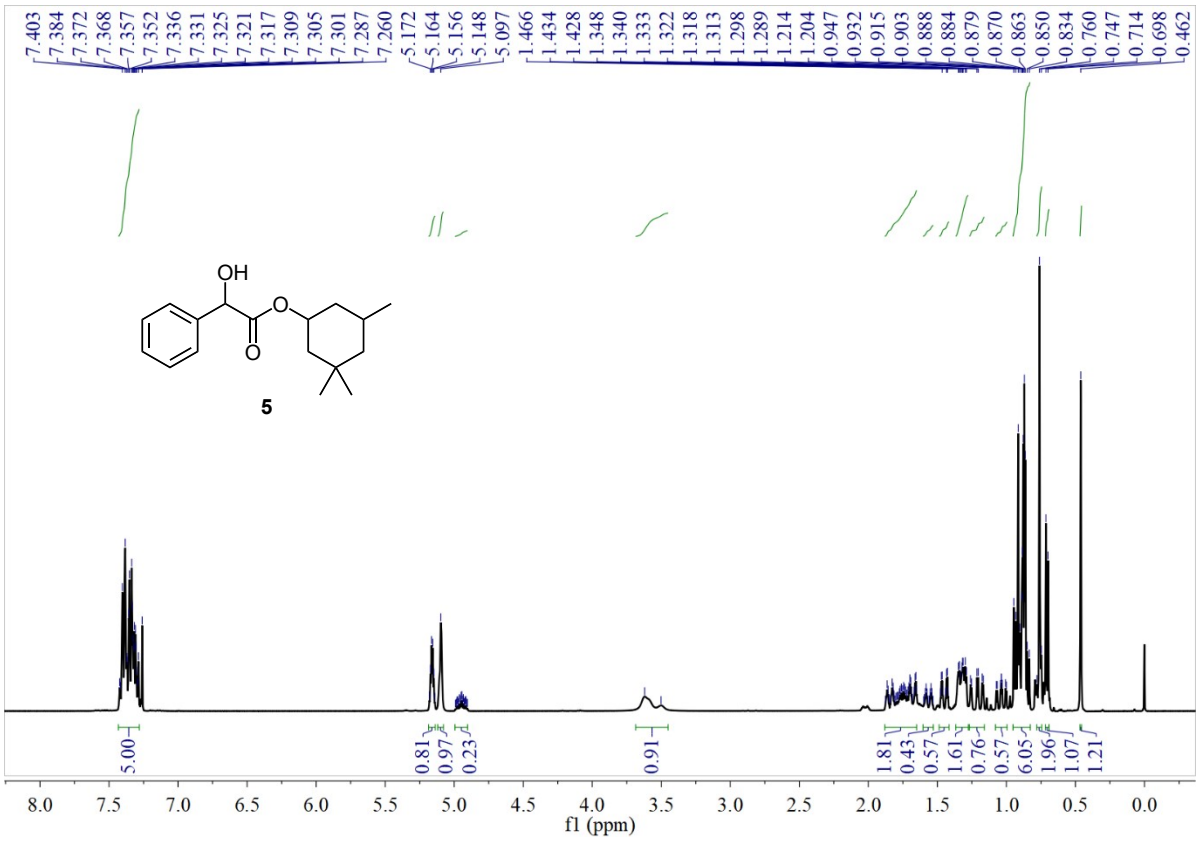


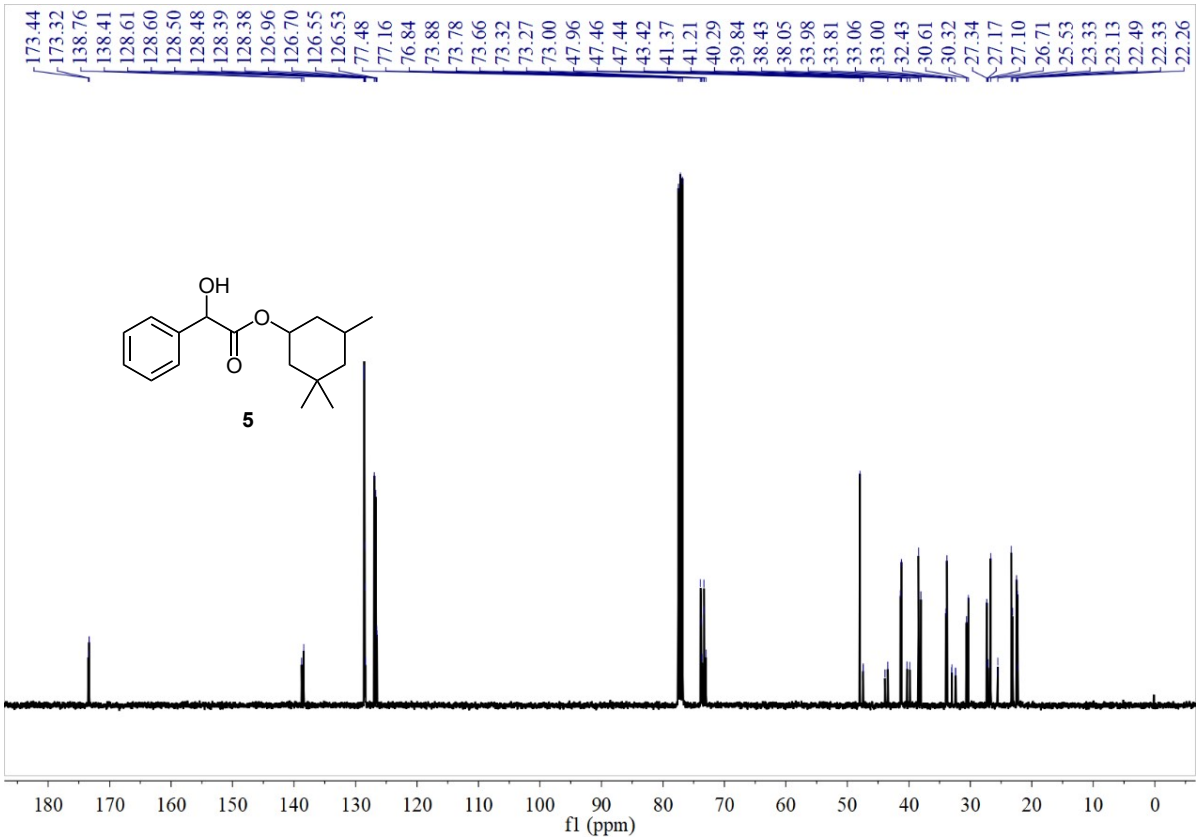
^{1}H and ^{13}C NMR spectra of **4**



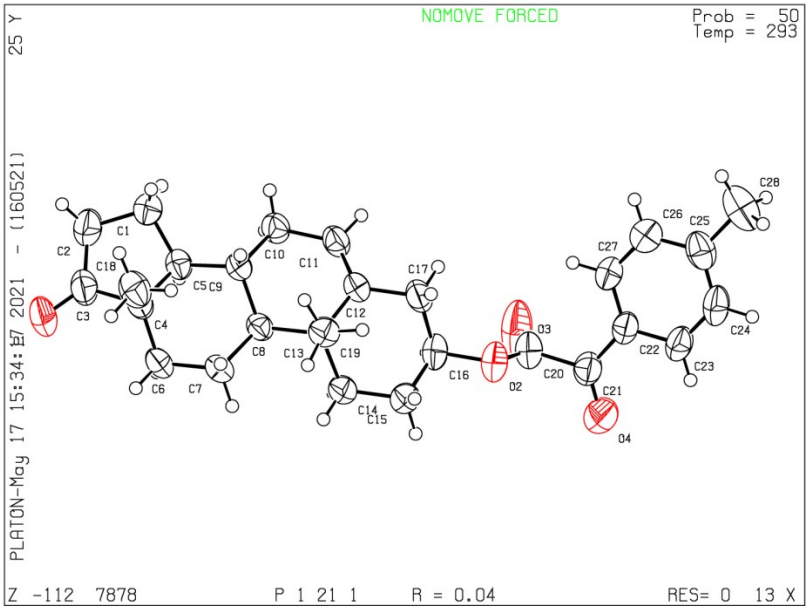


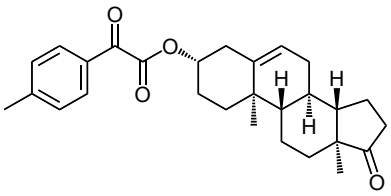
^{1}H and ^{13}C NMR spectra of **5**





7. X-ray single-crystal data





2bg

2bg : CCDC-2086255

CCDC-2086255	
Formula	$C_{28}H_{34}O_4$
Formula weight	434.55
Temperature / K	293
Crystal system	Monoclinic
space group	*P 1 21 1*
a / Å	6.7004(2)
b / Å	18.4346(3)
c / Å	10.0905(2)
a / °	90
β / °	107.339(2)
γ/ °	90
V / Å^3	1189.73(5)
Z	2
Dx (g/cm^3)	1.213
μ / mm^{-1}	0.631
F (000)	468.0
Reflections collected	21091

Independent reflections	4507
*R*int	0.0329
GOF	1.043
Final R indices ($I > 2\sigma(I)$)	0.0371, 0.0968
R indices (all data)	0.0401, 0.1018

Datablock:

Bond precision: C-C = 0.0037 A Wavelength=1.54184

```
Cell:           a=6.7004(2)    b=18.4346(3)        c=10.0905(2)
                alpha=90       beta=107.339(2)     gamma=90
Temperature:    293 K

                Calculated                 Reported
Volume          1189.73(5)                 1189.73(5)
Space group     P 21                       P 1 21 1
Hall group      P 2yb                      P 2yb
Moiety formula  C28 H34 O4                 C28 H34 O4
Sum formula     C28 H34 O4                 C28 H34 O4
Mr              434.55                     434.55
Dx,g cm-3       1.213                      1.213
Z               2                          2
Mu (mm-1)       0.631                      0.631
F000            468.0                      468.0
F000'           469.35
h,k,lmax        8,22,12                    11,25,20
Nref            4834[ 2496]                4507
Tmin,Tmax       0.970,0.981                0.648,1.000
Tmin'           0.969

Correction method= # Reported T Limits: Tmin= 0.648 Tmax=1.000
AbsCorr = MULTI-SCAN

Correction method= # Reported T Limits: Tmin= 0.648 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.81/0.93          Theta(max)= 74.021

R(reflections)= 0.0371( 4178)         wR2(reflections)= 0.1018( 4507)

S = 1.043                  Npar= 292
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C20 Check

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 3 Report

PLAT934_ALERT_3_C Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers .. 1 Check

Alert level G

PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 293 Check

PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 293 Check

PLAT760_ALERT_1_G CIF Contains no Torsion Angles ..... ? Info

PLAT791_ALERT_4_G Model has Chirality at C4 (Sohnke SpGr) S Verify

PLAT791_ALERT_4_G Model has Chirality at C5 (Sohnke SpGr) S Verify

PLAT791_ALERT_4_G Model has Chirality at C8 (Sohnke SpGr) S Verify

PLAT791_ALERT_4_G Model has Chirality at C9 (Sohnke SpGr) R Verify

PLAT791_ALERT_4_G Model has Chirality at C13 (Sohnke SpGr) R Verify

PLAT791_ALERT_4_G Model has Chirality at C16 (Sohnke SpGr) S Verify

PLAT795_ALERT_4_G C-Atom in CIF Coordinate List Out-of-Sequence .. C9 Note

PLAT796_ALERT_4_G O-Atom in CIF Coordinate List Out-of-Sequence .. O1 Note

PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 99 Note

PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info

PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by 2 Check

0 **ALERT level A** = Most likely a serious problem - resolve or explain

0 **ALERT level B** = A potentially serious problem, consider carefully

0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight

14 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

2 ALERT type 2 Indicator that the structure model may be wrong or deficient

2 ALERT type 3 Indicator that the structure quality may be low

9 ALERT type 4 Improvement, methodology, query or suggestion

1 ALERT type 5 Informative message, check