Supporting Information

Iron-Catalyzed Ring-Opening of Cyclic Carboxylic Acids Enabled by Photoinduced Ligand-to-Metal Charge Transfer

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1. General information

^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz) and ^{19}F NMR (376 MHz) spectra were recorded on a Quantum-I Plus 400 NMR spectrometer with $CDCl_3$ as solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts were reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to $CDCl_3$ at 7.26 ppm (for ^{1}H NMR) and 77.16 ppm (for ^{13}C NMR). HR-MS spectra were recorded on a Waters Xevo G2QTOF/UPLC mass spectrometer using electrospray ionization. EPR experiments were conducted using Bruker Elexsys E580 Spectrometer. All commercially available reagents and solvents were used as received unless otherwise specified.

1a, **1b**, **1d**, **1g**, **1h**, **1m**, **1q-1y**, **1aa**, **1ee**, **1gg**, **1hh**, **3a-3k**, **5a-5h** were directly obtained from commercial sources.

2. Photochemical reaction setup

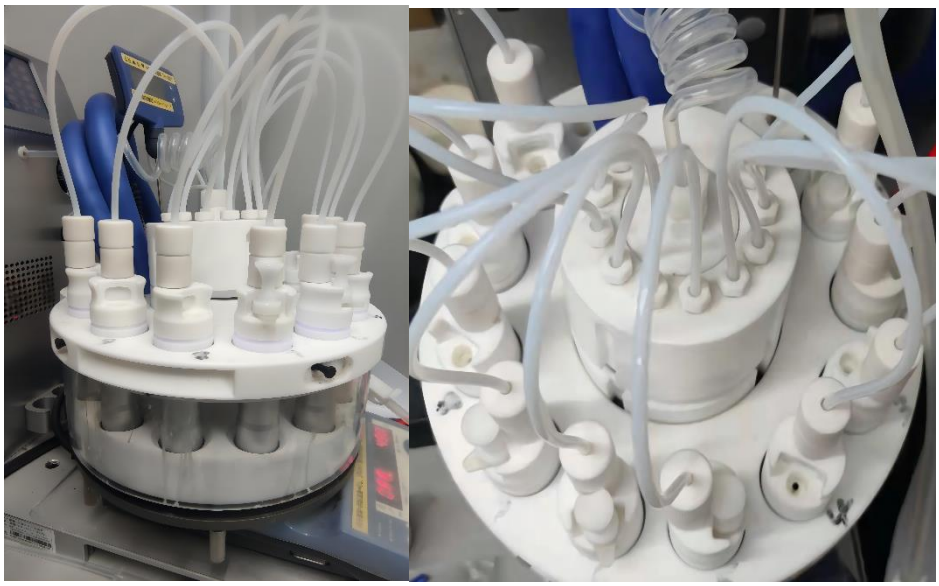
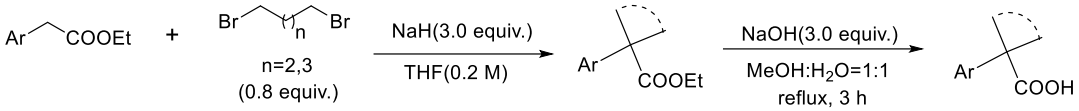


Figure S1: Reaction setup

3. General procedures for synthesis of substrates and Cobalt catalyst

3.1 Synthesis of substrates

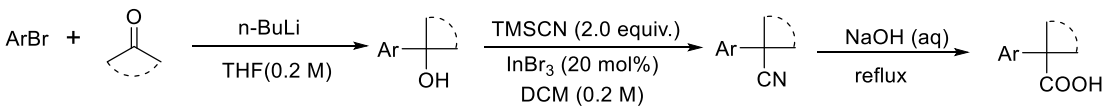
Method A[1]:



1) To a solution of ethyl arylacetate (1.0 equiv.) in dry THF (0.2 M) at 0 ℃, was added NaH (60% dispersion in mineral oil, 3.0 equiv.) in three portions at a 10 min interval. The mixture was stirred at 0 ℃ until no further effervescence was observed, then a solution of dibromoalkanes (0.8 equiv.) in dry THF (10 mL) was added dropwise at 0 ℃. The mixture was stirred at 0 ℃ for 10 min and then, slowly warmed to rt. Stirring was continued at 50 ℃ for 18 h. The mixture was diluted with EtOAc and H_2O and the aqueous layer was extracted with EtOAc. The combined extracts were washed with brine, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by flash chromatography eluting with 10-30% EtOAc / petroleum to give Corresponding ester.

2) A suspension of the ester (1.0 equiv.) in a 1:1 mixture of MeOH/H_2O (40 mL) was treated with NaOH (3.0 equiv.). The mixture was stirred and refluxed for 3 h. The mixture was then diluted with H_2O and the MeOH was mostly removed *in vacuo*. The H_2O solution was washed with ether, acidified to pH 1 with 2 M HCl solution and extracted with EtOAc. The combined EtOAc extracts were washed with brine, dried over $MgSO_4$, and concentrated *in vacuo* to give corresponding carboxylic acid as a white solid.

Method B[2,3]:



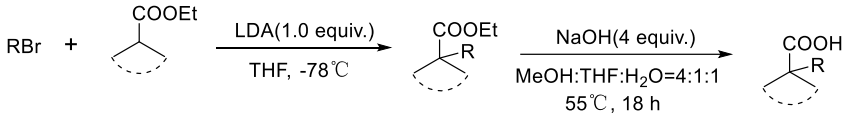
1) To a flame-dried flask was added aryl bromide (5 mmol, 1.0 equiv.) followed by THF (25 ml), the resulting mixture was stirred under nitrogen. *N*-butyl lithium (5.5 mmol, 1.1 equiv.) was added in -78 ℃. The mixture was stirred for 1 h and ketone (5 mmol, 1.0 equiv.) was then added dropwise. After the resulting mixture was stirred at

-78 ℃ for 12 h, the reaction was quenched with saturated aqueous ammonium chloride, and then extracted with EtOAc (30 mL ×2). The phases were separated and the organic phase washed three times with H_2O, and then dried over anhydrous sodium sulfate. The solvent was removed *in vacuo* and the residue purified by silica-gel chromatography (petroleum ether/EtOAc as eluent) to afford the desired alcohol.

2) To a flame-dried flask equipped with a teflon-coated magnetic stirred bar were added $InBr_3$ (0.8 mmol, 0.8 equiv.), CH_2Cl_2 (10 ml) and TMSCN (8 mmol, 2.0 equiv.) sequentially under argon atmosphere. alcohol (4 mmol, 1.0 equiv.) in CH_2Cl_2 (10 ml) was then introduced to the reaction system dropwise with a syringe. The mixture was stirred at room temperature for 30 min. The resulting solution was evaporated *in vacuo* and the residue was submitted to flash column chromatography separation on silica gel with petroleum ether/ethyl acetate (10:1) as eluent to afford the corresponding nitrile compound.

3) A solution of nitrile compound (1.0 equiv.) in ethanol (0.4 M) and 10 N aqueous sodium hydroxide (0.3 M) was refluxed for 48 h, cooled to room temperature, then poured into 1 N aqueous hydrochloric acid, and extracted by ethyl acetate. The organic extracts were washed with brine, dried over sodium sulfate, and concentrated under reduced pressure. The crude product was recrystallized using ethyl acetate to obtain corresponding carboxylic acid.

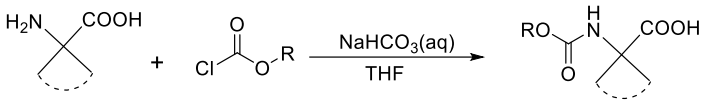
Method C[4,5]



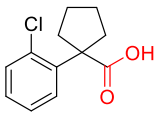
1) To a flame-dried flask was added ester (1.0 equiv.) followed by THF (0.2 M). the resulting mixture was stirred under nitrogen for 1 h. LDA (1.0 equiv.) was added in -78 ℃. The mixture was stirred for 30 min and the bromide (1.5 equiv.) was then added dropwise. After the resulting mixture was stirred at -78 ℃ for 6 h, the reaction was quenched with saturated aqueous ammonium chloride, and then extracted with EtOAc (30 mL ×2). The phases were separated and the organic phase washed three times with H_2O, and then dried over anhydrous sodium sulfate. The solvent was removed *in vacuo* and the residue purified by silica-gel chromatography (petroleum ether/EtOAc as eluent) to afford the desired ester.

2) The ester was added to a mixture of NaOH (2 M, 20 mL) and methanol (30 mL) and stirred overnight at 55 ℃. After removal of methanol in vacuo, the residue was diluted with water and extracted with Et_2O (20 mL × 2). Then the pH value of the water layer was adjusted to 2.0 with HCl (3 M) and extracted with Et_2O (30 mL × 3). The combined organic phases were evaporated *in vacuo* and the residue purified by silica-gel chromatography (petroleum ether/EtOAc as eluent) to gain the acid.

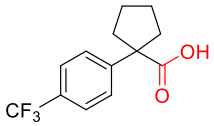
Method D[6]:



To a stirred solution of amino acid (1.0 equiv.) in a mixture of aq. $NaHCO_3$ and THF (20 mL) was added acyl chloride (1.5 equiv.), dropwise and at 0 ℃. The mixture was stirred at room temperature for 12 h and then diluted with ethyl acetate and washed 3 N HCl, dried and the solvent removed *in vacuo* to give Amino acid derivatives, which was used without further purification.

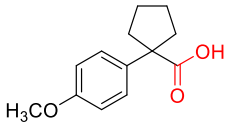


1-(2-chlorophenyl)cyclopentane-1-carboxylic acid (**1c**): Prepared using general procedure A from ethyl 2-(2-chlorophenyl)acetate and 1,4-dibromobutane to give 728 mg of the title compound (White solid, 65% yield, m.p. 108.2 – 109.4 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 11.30 (s, 1H), 7.47 – 7.38 (m, 2H), 7.32 – 7.21 (m, 2H), 2.64 – 2.50 (m, 2H), 2.26 – 2.14 (m, 2H), 1.98 – 1.84 (m, 2H), 1.82 – 1.69 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 182.8, 141.1, 134.4, 130.7, 128.2, 127.3, 126.6, 58.0, 36.6, 24.8; **HRMS (ESI)** calcd $C_{12}H_{14}{}^{35}ClO_2$ $[M + H]^+$: 225.0677, found: 225.0673.

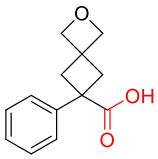


1-(4-(trifluoromethyl)phenyl)cyclopentane-1-carboxylic acid(1e): Prepared using general procedure A from ethyl 2-(4-(trifluoromethyl)phenyl)acetate and 1,4-dibromobutane to give 929 mg of the title compound (White solid, 72% yield, m.p. 142.5 – 143.2 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.61 (d, *J* = 8.3 Hz, 2H), 7.54 (d, *J*

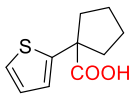
= 8.1 Hz, 2H), 2.79 – 2.64 (m, 2H), 2.04 – 1.90 (m, 2H), 1.87 – 1.74 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 181.3, 146.7, 129.3 (d, *J* = 32.4 Hz), 125.3 (d, *J* = 3.6 Hz), 124.1 (d, *J* = 277.7 Hz), 58.9, 36.1, 23.6; **^{19}F NMR (376 MHz, $CDCl_3$)** δ -62.51 (s, 3F); **HRMS (ESI)** calcd $C_{13}H_{14}F_3O_2$ $[M + H]^+$: 259.0940, found: 259.0939.



1-(4-methoxyphenyl)cyclopentane-1-carboxylic acid (1f): Prepared using general procedure A from ethyl ethyl 2-(4-methoxyphenyl)acetate and 1,4-dibromobutane to give 814 mg of the title compound (White solid, 74% yield, m.p. 153.5 – 154.3 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 2.73 – 2.62 (m, 2H), 1.99 – 1.85 (m, 2H), 1.76 (t, *J* = 6.9 Hz, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 182.5, 158.5, 134.9, 128.3, 113.67, 58.1, 55.3, 36.0, 23.5; **HRMS (ESI)** calcd $C_{13}H_{17}O_3$ $[M + H]^+$: 221.1172, found: 221.1166.

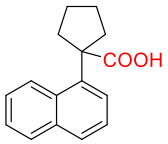


5-phenyl-2-oxaspiro[3.3]heptane-6-carboxylic acid (1i): Prepared using general procedure A from ethyl 2-phenylacetate and 1,1-bis(bromomethyl)cyclobutene to give 578 mg of the title compound (White solid, 53% yield, m.p. 136.8 – 137.2 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.43 – 7.35 (m, 2H), 7.31 (dd, *J* = 9.6, 1.7 Hz, 1H), 7.22 – 7.11 (m, 2H), 4.47 (s, 2H), 3.66 (s, 2H), 2.42 (s, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 175.8, 139.9, 128.4, 127.4, 126.5, 74.7, 64.7, 48.3, 40.8, 35.6; **HRMS (ESI)** calcd $C_{13}H_{15}O_3$ $[M + H]^+$: 219.1016, found: 219.1012.

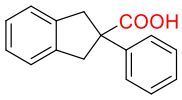


1-(thiophen-2-yl)cyclopentane-1-carboxylic acid (1j): Prepared using general procedure A from ethyl 2-(thiophen-2-yl)acetate and 1,4-dibromobutane to give 608 mg of the title compound (White solid, 62% yield, m.p. 100.6 – 101.2 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.24 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.03 (dd, *J* = 3.6, 1.2 Hz, 1H), 6.99 (dd, *J* = 5.1, 3.6 Hz, 1H), 2.66 – 2.54 (m, 2H), 2.20 – 2.07 (m, 2H), 1.85 – 1.74

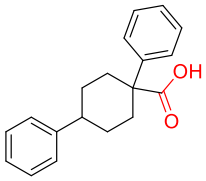
(m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 180.9, 146.6, 126.8, 125.0, 124.3, 56.0, 38.0, 23.8; **HRMS (ESI)** calcd $C_{10}H_{13}O_2S$ $[M + H]^+$: 197.0631, found: 197.0634.



1-(naphthalen-1-yl)cyclopentane-1-carboxylic acid (1k): Prepared using general procedure A from ethyl 2-(naphthalen-1-yl)acetate and 1,4-dibromobutane to give 960 mg of the title compound (White solid, 80% yield, m.p. 156.5 – 157.1 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.97 (d, *J* = 8.3 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.55 (d, *J* = 7.2 Hz, 1H), 7.50 – 7.39 (m, 3H), 2.75 – 2.63 (m, 2H), 2.37 – 2.21 (m, 2H), 1.92 – 1.68 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 183.4, 139.1, 134.5, 131.7, 129.2, 128.2, 126.0, 125.4, 125.1, 124.9, 123.5, 57.7, 37.4, 24.6; **HRMS (ESI)** calcd $C_{16}H_{17}O_2$ $[M + H]^+$: 241.1223, found: 241.1226.

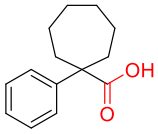


2-phenyl-2,3-dihydro-1H-indene-2-carboxylic acid (1l): Prepared using general procedure A from ethyl 2-phenylacetate and 1,2-bis(bromomethyl)benzene to give 559 mg of the title compound (White solid, 47% yield, m.p. 163.7 – 164.0 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.49 – 7.42 (m, 2H), 7.40 – 7.34 (m, 2H), 7.33 – 7.30 (m, 1H), 7.29 – 7.24 (m, 2H), 7.23 – 7.16 (m, 2H), 4.00 (d, *J* = 15.4 Hz, 2H), 3.35 (d, *J* = 15.5 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 180.7, 141.8, 141.0, 128.6, 127.4, 127.0, 126.8, 124.3, 59.4, 42.4; **HRMS (ESI)** calcd $C_{16}H_{15}O_2$ $[M + H]^+$: 239.1067, found: 239.1072.

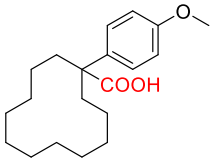


1,4-diphenylcyclohexane-1-carboxylic acid (1n):Prepared using general procedure B from bromobenzene and 4-phenylcyclohexan-1-one to give 588 mg of the title compound (White solid, 42% yield, m.p. 145.6 – 146.2 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.59 – 7.50 (m, 2H), 7.46 – 7.38 (m, 2H), 7.37 – 7.31 (m, 3H), 7.29 – 7.21

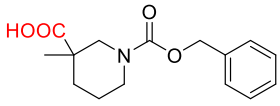
(m, 3H), 2.94 – 2.81 (m, 2H), 2.66 – 2.54 (m, 1H), 2.13 – 1.98 (m, 2H), 1.90 – 1.67 (m, 4H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 180.4, 146.6, 143.6, 128.7, 128.4, 127.3, 126.9, 126.2, 125.8, 50.3, 43.4, 34.9, 31.7; **HRMS (ESI)** calcd $C_{19}H_{21}O_2$ $[M + H]^+$: 281.1536, found: 281.1538.



1-phenylcycloheptane-1-carboxylic acid (1o): Prepared using general procedure B from bromobenzene and cycloheptanone to give 589 mg of the title compound (White solid, 54% yield, m.p. 111.6 – 112.8 ℃); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.45 – 7.33 (m, 4H), 7.30 – 7.25 (m, 1H), 2.52 – 2.39 (m, 2H), 2.20 – 2.07 (m, 2H), 1.80 – 1.52 (m, 8H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 182.9, 144.3, 128.5, 126.8, 126.2, 54.1, 36.6, 29.7, 23.7; **HRMS (ESI)** calcd $C_{14}H_{19}O_2$ $[M + H]^+$: 219.1380, found: 219.1383.

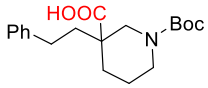


1-(4-methoxyphenyl)cyclododecane-1-carboxylic acid (1p): Prepared using general procedure B from 1-bromo-4-methoxybenzene and cyclododecanone to give 590 mg of the title compound (White solid, 37% yield, m.p. 168.3 – 169.2 ℃); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.27 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H), 2.13 – 2.03 (m, 2H), 2.02 – 1.93 (m, 2H), 1.45 – 1.33 (m, 14H), 1.29 – 1.20 (m, 2H), 1.18 – 1.02 (m, 2H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 182.0, 158.3, 134.7, 127.3, 113.7, 55.2, 52.1, 29.9, 26.4, 26.2, 22.3, 22.0, 19.4; **HRMS (ESI)** calcd $C_{20}H_{31}O_3$ $[M + H]^+$: 319.2268, found: 319.2270.

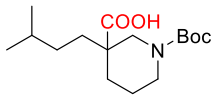


1-((benzyloxy)carbonyl)-3-methylpiperidine-3-carboxylic acid (1y): Prepared using general procedure C from 1-benzyl 3-ethyl piperidine-1,3-dicarboxylate and iodomethane to give 1.08 g of the title compound (White solid, 78% yield, m.p. 124.6 – 125.2 ℃); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.43 – 7.29 (m, 5H), 5.18 (s, 2H), 4.13 – 3.97 (m, 1H), 3.73 – 3.57 (m, 1H), 3.40 – 3.28 (m, 1H), 3.28 – 3.16 (m, 1H), 2.20 –

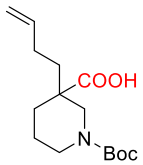
2.05 (m, 1H), 1.73 – 1.60 (m, 2H), 1.58 – 1.46 (m, 1H), 1.26 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 181.9, 155.6, 136.8, 128.5, 128.0, 127.9, 67.2, 50.9, 44.0, 42.6, 33.4, 22.3, 22.0; **HRMS (ESI)** calcd $C_{15}H_{20}NO_4$ $[M + H]^+$: 278.1387, found: 278.1388.



1-(tert-butoxycarbonyl)-3-phenethylpiperidine-3-carboxylic acid (1bb): Prepared using general procedure C from 1-(tert-butyl) 3-ethyl piperidine-1,3-dicarboxylate and (2-bromoethyl)benzene to give 1.33 g of the title compound (White solid, 80% yield, m.p. 118.5 – 119.3 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.33 – 7.27 (m, 2H), 7.25 – 7.18 (m, 3H), 3.98 – 3.84 (m, 1H), 3.60 – 3.30 (m, 3H), 2.79 – 2.54 (m, 2H), 2.18 – 2.07 (m, 1H), 2.06 – 1.93 (m, 1H), 1.90 – 1.77 (m, 1H), 1.73 – 1.59 (m, 3H), 1.50 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 181.0, 154.9, 141.6, 128.5, 128.4, 126.0, 79.9, 46.3, 37.4, 32.4, 30.7, 28.4, 21.8; **HRMS (ESI)** calcd $C_{19}H_{28}NO_4$ $[M + H]^+$: 334.2013, found: 334.2016.

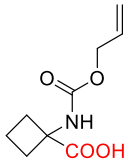


1-(tert-butoxycarbonyl)-3-isopentylpiperidine-3-carboxylic acid (1cc): Prepared using general procedure C from 1-(tert-butyl) 3-ethyl piperidine-1,3-dicarboxylate and 1-bromo-3-methylbutane to give 1.12 g of the title compound (White solid, 75% yield, m.p. 80.2 – 80.5 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 3.94 – 3.75 (m, 1H), 3.54 – 3.42 (m, 1H), 3.36 – 3.16 (m, 2H), 2.09 – 1.98 (m, 1H), 1.68 – 1.58 (m, 3H), 1.57 – 1.48 (m, 3H), 1.46 (s, 9H), 1.27 – 1.09 (m, 2H), 0.89 (d, *J* = 6.6 Hz, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 181.5, 154.8, 79.7, 49.8, 46.2, 43.4, 33.4, 32.9, 32.1, 28.4, 22.6, 22.4, 21.9; **HRMS (ESI)** calcd $C_{16}H_{30}NO_4$ $[M + H]^+$: 300.2169, found: 300.2168.

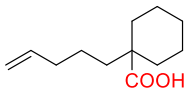


3-(but-3-en-1-yl)-1-(tert-butoxycarbonyl)piperidine-3-carboxylic acid (1dd): Prepared using general procedure C from 1-(tert-butyl) 3-ethyl piperidine-1,3-dicarboxylate and 4-bromobut-1-ene to give 1.33 g of the title

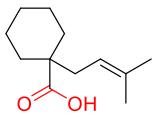
compound (White solid, 80% yield, m.p. 85.5 – 85.9 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 5.89 – 5.73 (m, 1H), 5.11 – 4.94 (m, 2H), 3.95 – 3.80 (m, 1H), 3.59 – 3.43 (m, 1H), 3.40 – 3.16 (m, 2H), 2.21 – 1.98 (m, 3H), 1.80 – 1.68 (m, 1H), 1.68 – 1.53 (m, 4H), 1.48 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 180.7, 154.8, 137.8, 115.0, 79.8, 49.6, 46.1, 34.6, 32.2, 28.4, 21.9; **HRMS (ESI)** calcd $C_{15}H_{26}NO_4$ $[M + H]^+$: 284.1856, found:284.1856.



1-(((allyloxy)carbonyl)amino)cyclobutane-1-carboxylic acid (1ee): Prepared using general procedure D from 1-aminocyclobutane-1-carboxylic acid and allyl carbonochloridate to give 846 mg of the title compound (Colorless liquid, 80% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 5.96 – 5.83 (m, 1H), 5.50 (s, 1H), 5.37 – 5.27 (m, 1H), 5.26 – 5.19 (m, 1H), 4.58 (d, *J* = 3.5 Hz, 2H), 2.74 – 2.61 (m, 2H), 2.48 – 2.24 (m, 2H), 2.17 – 1.98 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.9, 155.4, 132.6, 117.9, 65.8, 58.2, 31.2, 15.1; **HRMS (ESI)** calcd $C_9H_{14}NO_4$ $[M + H]^+$: 200.0917, found: 200.0916.

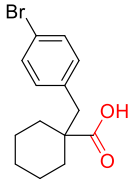


(pent-4-en-1-yl)cyclohexane-1-carboxylic acid (7): Prepared using general procedure C from methyl cyclohexanecarboxylate and 5-bromopent-1-ene to give 706 mg of the title compound (Colorless liquid, 72% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.95 (s, 1H), 5.89 – 5.72 (m, 1H), 5.00 (dd, *J* = 20.7, 13.7 Hz, 2H), 2.17 – 1.95 (m, 4H), 1.66 – 1.52 (m, 5H), 1.51 – 1.35 (m, 4H), 1.33 – 1.21 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 184.1, 138.5, 114.7, 46.8, 39.8, 34.1, 33.9, 25.9, 23.3, 23.2; **HRMS (ESI)** calcd $C_{12}H_{21}O_2$ $[M + H]^+$: 197.1536, found: 197.1537.



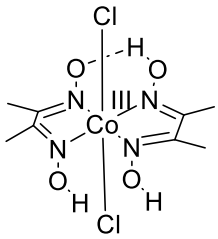
1-(3-methylbut-2-en-1-yl)cyclohexane-1-carboxylic acid (9): Prepared using general procedure C from methyl cyclohexanecarboxylate and

1-bromo-3-methylbut-2-ene to give 666 mg of the title compound (White solid, 68% yield, m.p. 65.2 – 65.4 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 5.15 (t, *J* = 7.7 Hz, 1H), 2.27 (d, *J* = 7.6 Hz, 2H), 2.14 – 2.03 (m, 2H), 1.74 (s, 3H), 1.63 (s, 3H), 1.63 – 1.56 (m, 3H), 1.50 – 1.36 (m, 2H), 1.35 – 1.23 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 183.3, 134.6, 118.9, 47.6, 38.7, 33.5, 26.1, 25.9, 23.3, 17.9; **HRMS (ESI)** calcd $C_{12}H_{21}O_2$ $[M + H]^+$: 197.1536, found: 197.1541.

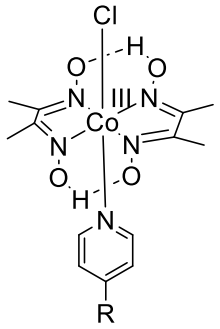


1-(4-bromobenzyl)cyclohexane-1-carboxylic acid (S1): Prepared using general procedure C from 1-bromo-4-(bromomethyl)benzene and methyl cyclohexanecarboxylate to give 1.36 g of the title compound (White solid, 92% yield, m.p. 145.3 – 146.1 ℃); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.41 (d, *J* = 7.0 Hz, 2H), 7.03 (d, *J* = 7.1 Hz, 2H), 2.83 (s, 2H), 2.12 – 2.03 (m, 2H), 1.74 – 1.61 (m, 3H), 1.50 – 1.35 (m, 2H), 1.35 – 1.23 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 182.3, 135.9, 131.7, 131.1, 120.7, 48.5, 46.0, 33.7, 25.7, 23.2; **HRMS (ESI)** calcd $C_{14}H_{18}{}^{79}BrO_2$ $[M + H]^+$: 297.0485, found: 297.0493.

3.2 Synthesis of cobalt catalyst

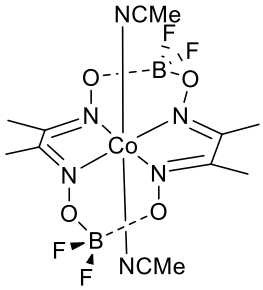


$Co(dmgH)(dmgH_2)Cl_2$[7]: Cobaltous chloride hexahydrate (5.0 g, 0.021 mol) was dissolved in acetone (150 mL) and dimethylglyoxime (4.9 g, 0.042 mol) was added. The mixture was stirred for 10 min and filtered to remove undissolved material. Then the solution was allowed to stand overnight and green crystal was formed, which was further collected and washed with acetone to obtained $Co(dmgH)(dmgH_2)Cl_2$.



R = H ,$Co(dmgH)_2PyCl$
R=CN,$Co(dmgH)_2$(4-CN-Py)Cl
R= $N(CH_3)_2$,$Co(dmgH)_2$(DMAP)Cl

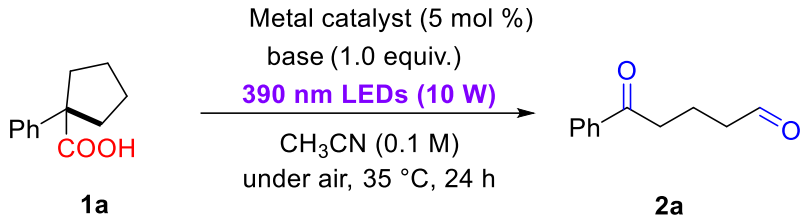
$Co(dmgH)_2$(4-R-Py)Cl[7]: $Co(dmgH)(dmgH_2)Cl_2$ (1.0 equiv.) was suspended in 0.03 M methanol. Then pyridine derivative (1.0 equiv.) was added and the suspension was dissolved. After 1 hour, a brown precipitate was formed, which was filtered and washed with water (10 mL), ethanol (10 mL), and diethyl ether (10 mL) to give a series of cobalt catalysts as shown.



$Co(dmgBF_2)_2(MeCN)_2$[8]: Diethyl ether (150 mL, O_2-free) was added to a flask containing $Co(OAc)_2$•$4H_2O$ (2.0 g, 8 mmol) and $dmgH_2$ (1.9 g, 16 mmol), followed by freshly distilled BF_3•Et_2O (10 mL, an excess). The mixture was stirred for 6 hours under argon. The resulting solid was filtered under argon, washed with ice-cold water (10 mL × 3, O_2-free) and air-dried to produce a brownish-red solid product [$Co(dmgBF_2)_2(H_2O)_2$]. Then this product was stirred in CH_3CN for 1 hour, filtrated, washed with CH_3CN and dried under vacuum to afford $Co(dmgBF_2)_2(CH_3CN)_2$.

4. Optimization of the reaction conditions

Table S1. Optimization of ring opening for cyclic tertiary carboxylic acids



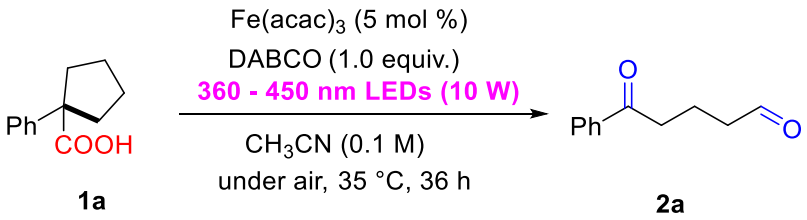
1a **2a**

Entry	Metal catalyst	Base	Solvent	Yield(%)[a]
1	$Cu(acac)_2$	Cs_2CO_3	CH_3CN	N.D
2	$Cu(CF_3SO_3)_2$	Cs_2CO_3	CH_3CN	N.D
3	$Fe(CF_3SO_3)_3$	Cs_2CO_3	CH_3CN	trace
4	$Fe_2(SO_4)_3$	Cs_2CO_3	CH_3CN	0
5	$Fe(Cl)_3$	Cs_2CO_3	CH_3CN	trace
6	$Fe(Br)_3$	Cs_2CO_3	CH_3CN	0
7	$Fe(acac)_3$	Cs_2CO_3	CH_3CN	32
8	$Fe(acac)_3$	KOH	CH_3CN	<5
9	$Fe(acac)_3$	NaOH	CH_3CN	<5
10	$Fe(acac)_3$	LiOH	CH_3CN	N.D
11	$Fe(acac)_3$	MeOK	CH_3CN	<5
12	$Fe(acac)_3$	CH_3COOK	CH_3CN	trace
13	$Fe(acac)_3$	K_3PO_4	CH_3CN	20
14	$Fe(acac)_3$	K_2CO_3	CH_3CN	15
15	$Fe(acac)_3$	CsF	CH_3CN	trace
16	$Fe(acac)_3$	TMG	CH_3CN	trace
17	$Fe(acac)_3$	DIPEA	CH_3CN	25
18	$Fe(acac)_3$	Et_3N	CH_3CN	22
19	$Fe(acac)_3$	Py	CH_3CN	<5
20	$Fe(acac)_3$	DBU	CH_3CN	46
21	$Fe(acac)_3$	DABCO	CH_3CN	49
22	$Fe(acac)_3$	DABCO	CH_3CN	54

Entry	Metal catalyst	Base	Solvent	Yield(%)[a]
23	$Fe(acac)_3$	DABCO	CH_3CN	79
24	$Fe(acac)_3$	DABCO	DMSO	<5
25	$Fe(acac)_3$	DABCO	DMF	trace
26	$Fe(acac)_3$	DABCO	CH_3CN	54
27[b]	$Fe(acac)_3$	DABCO	CH_3CN	79(75[c])
28[b]	$Fe(acac)_3$	DABCO	DMSO	<5
29[b]	$Fe(acac)_3$	DABCO	DMF	trace
30[b]	$Fe(acac)_3$	DABCO	DME	35
31[b]	$Fe(acac)_3$	DABCO	EtOAc	62
32	-	DABCO	CH_3CN	0
33	$Fe(acac)_3$	-	CH_3CN	12
32[bd]	$Fe(acac)_3$	DABCO	CH_3CN	N.D
33[be]	$Fe(acac)_3$	DABCO	CH_3CN	N.D

[a]Reaction conditions: **1a** (0.20 mmol), base (1.0 equiv.), Metal catalyst (5 mol%) in CH_3CN (2 mL, 0.1 M), 35 ℃, 390 nm purple LEDs (10 W), 1.3.5-trimethoxybenzene as internal standard. [b] 36h. [c] Isolated yield. [d] The reaction proceeds in dark. [e]The reaction proceeds under N_2.

Table S2. The effect of the light length on the reaction

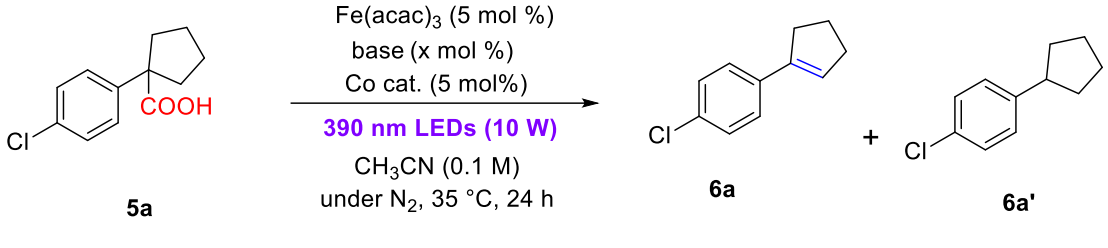


1a **2a**

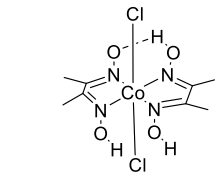
Entry	Wavelength (nm)	Yield (%)[a]
1	360	34
2	380	65
3	390	75
4	410	68
5	450	64

[a]Reaction conditions: **1a** (0.20 mmol), DABCO (1.0 equiv.), $Fe(acac)_3$ (5 mol%) in CH_3CN (2 mL, 0.1 M), 35 ℃, 360 - 450 nm LEDs (10 W), 36 h, Isolated yield.

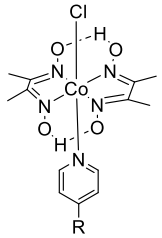
Table S3. Optimization of cyclization of photocobalt synergistic catalysis



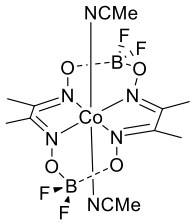
Cobaloxime



Co-1: $Co(dmgH)(dmgH_2)Cl_2$



Co-2: R = H, $Co(dmgH)_2PyCl$
Co-3: R = CN, $Co(dmgH)_2$(4-CN-Py)Cl
Co-4: R = $N(CH_3)_2$, $Co(dmgH)_2$(DMAP)Cl



Co-5: $Co(dmgBF_2)_2(MeCN)_2$

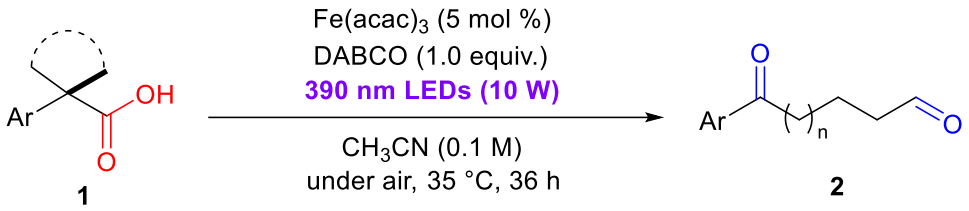
+

entry	Solvent	Cobaltcatalyst	base	yield(%)[a]	5:5'[b]
1	CH_3CN	Co-2	DABCO(100 %)	N.D	-
2	CH_3CN	Co-2	Cs_2CO_3(100%)	24	8:1
3	CH_3CN	Co-2	Cs_2CO_3(50%)	32	8:1
4	CH_3CN	Co-2	Cs_2CO_3(20%)	45	8:1
5	CH_3CN	Co-2	Cs_2CO_3(10%)	55	8:1
6	CH_3CN	Co-2	Cs_2CO_3(5%)	57	8:1
7	CH_3CN	Co-1	Cs_2CO_3 (5%)	42	>1:20
8	CH_3CN	Co-3	Cs_2CO_3 (5%)	59	>20:1
9	CH_3CN	Co-4	Cs_2CO_3 (5%)	46	12:1
10	CH_3CN	Co-5	Cs_2CO_3 (5%)	25	15:1
11	CH_3CN	Co-3	K_3PO_4 (5%)	40	10:1
12	CH_3CN	Co-3	K_2CO_3 (5%)	<5%	-
13	PhMe	Co-3	Cs_2CO_3 (5%)	trace	-
14	EtOAc	Co-3	Cs_2CO_3 (5%)	54	>20:1
15	DCE	Co-3	Cs_2CO_3 (5%)	35	>20:1

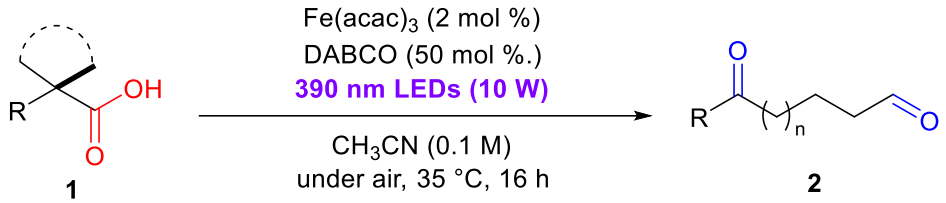
entry	Solvent	Cobaltcatalyst	base	yield(%)[a]	5:5'[b]
16	CH_3CN	-	Cs_2CO_3 (5%)	<5	-
17	CH_3CN	Co-3	-	trace	-
18[c]	CH_3CN	Co-3	Cs_2CO_3 (5%)	N.D	-
19[d]	CH3CN	Co-3	Cs_2CO_3 (5%)	N.D	-

[a]Reaction conditions: **5a** (0.20 mmol), Cs_2CO_3 (5 mol%), $Fe(acac)_3$ (5 mol%), $Co(dmgH)_2$(4-CN-Py)Cl (5 mol%) in CH_3CN (2 mL, 0.1 M), 35 ℃, 390 nm LEDs (10 W), 24 h, isolated yield. [b] ratios determined by the crude ^1H NMR spectra. [c]no $Fe(acac)_3$. [d]The reaction proceeds in dark.

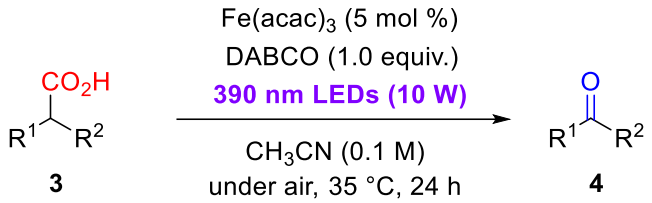
5. General procedures for iron catalyzed LMCT reaction



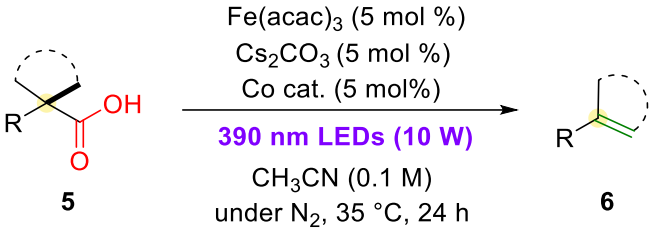
General procedure 1: To a 25 mL quartz tube equipped with a magnetic stir bar, the corresponding aryl cycloformic acid (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (3.6 mg, 5 mol %), DABCO (22.4 mg, 0.2 mmol, 1.0 equiv.) and CH_3CN (2 mL) were added. The resulting mixture was stirred in air under a 10W LEDs and irradiated for 36 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.



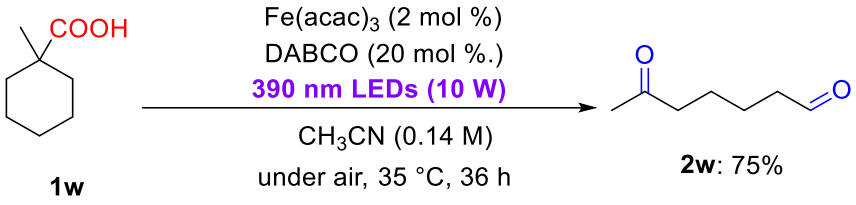
General procedure 2: To a 25 mL quartz tube equipped with a magnetic stir bar, the corresponding alkyl cycloformic acid (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (1.5 mg, 2 mol %), DABCO (11.2 mg, 0.1 mmol, 50 mol %) and CH_3CN (2 mL) were added. The resulting mixture was stirred in air under a 10W LEDs and irradiated for 16 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.



General procedure 3: To a 25 mL quartz tube equipped with a magnetic stir bar, the corresponding acid (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (3.6 mg, 5 mol %), DABCO (22.4 mg, 0.2 mmol, 1.0 equiv.) and CH_3CN (2 mL) were added. The resulting mixture was stirred in air under a 10W LEDs and irradiated for 24 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.



General procedure 4: To a 25 mL quartz tube equipped with a magnetic stir bar, the corresponding acid (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (3.6 mg, 5 mol %), Cs_2CO_3 (3.3 mg, 5 mol %), $Co(dmgH)_2(4\text{-CN-Py})Cl$ (4.3 mg, 5 mol %) was added. The resulting mixture was sealed and then subjected to freeze-pump-thaw for three times. CH_3CN (2 mL) were then added under argon atmosphere. After that, the reaction was placed under a 10W LEDs and irradiated for 24 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel.



Scale-up synthesis of compound 2w

To a 100 mL Single mouth bottle equipped with a magnetic stir bar, 1-Methyl-1-cyclohexanecarboxylic Acid (**1w)** (1.0 g, 7 mmol, 1.0 equiv.), $Fe(acac)_3$ (50 mg, 2 mol %), DABCO (157 mg, 1.4 mmol, 20 mol %) and CH_3CN (50 mL) were added. The resulting mixture was stirred in air under a 390 nm 10W LEDs and irradiated for 36 hours. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography to give **2a** (672 mg, 75% yield) as a yellow oil.

Figure S2: Gram experimental reaction setup

6. Mechanistic studies

6.1 UV-vis absorption study

UV-visible absorption spectra were collected on a SPECORD 200 PLUS. 1-phenylcyclopentane-1-carboxylic acid (**1a**), DABCO, $Fe(acac)_3$ were prepared 1.8×10^{-4} mol/L in CH_3CN (Fig. S3), among which two broad absorption peaks were observed around 360 nm and 440 nm, indicative of photochemical activity under blue LED light irradiation (> 380 nm).

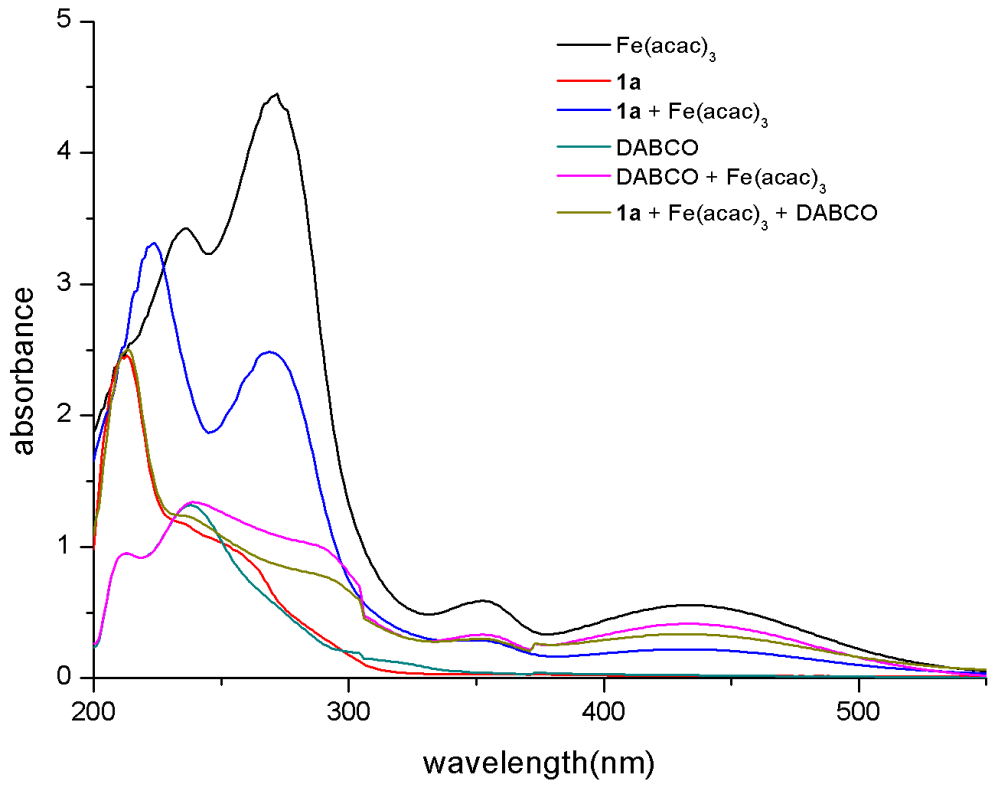
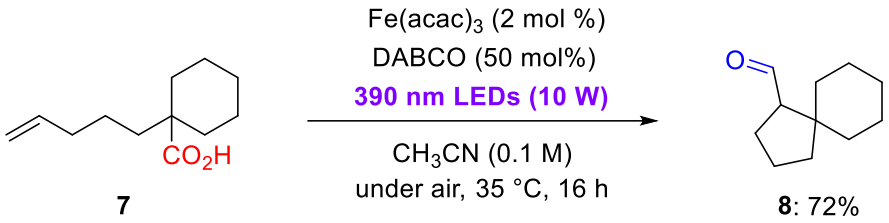


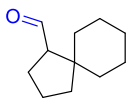
Figure S3: UV-vis absorption

6.2 Intramolecular 5-*exo*-trig cyclization



7 **8**: 72%

To a 25 mL quartz tube equipped with a magnetic stir bar, 1-(pent-4-en-1-yl)cyclohexane-1-carboxylic acid (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (1.5 mg, 2 mol%), DABCO (11.2 mg, 0.1 mmol, 50 mol%) and CH_3CN (2 mL) were added. The resulting mixture was stirred in air under a 10W LEDs and irradiated for 16 hours. The temperature was maintained at 35 ℃ when the LED light was on. After the reaction was finished (monitored by TLC), the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel to give spiro[4.5]decane-1-carbaldehyde as colorless liquid (29 mg, 72%).



spiro[4.5]decane-1-carbaldehyde (8): Followed the general procedure 2 with 1-(pent-4-en-1-yl)cyclohexane-1-carboxylic acid (39 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 29 mg of the title compound (colourless oil; 72% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.77 (d, *J* = 3.5 Hz, 1H), 2.40 (td, *J* = 7.6, 3.5 Hz, 1H), 2.08 – 1.94 (m, 1H), 1.86 – 1.74 (m, 3H), 1.72 – 1.63 (m, 2H), 1.60 – 1.54 (m, 3H), 1.52 – 1.43 (m, 3H), 1.41 – 1.35 (m, 2H), 1.34 – 1.28 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 205.9, 61.4, 49.2, 38.4, 32.9, 26.2, 25.8, 24.5, 23.6, 23.0, 22.7; **HRMS (ESI)** calcd $C_{11}H_{19}$ $[M + H]^+$: 167.1430, found: 167.1434.

6.3 EPR experiments and data

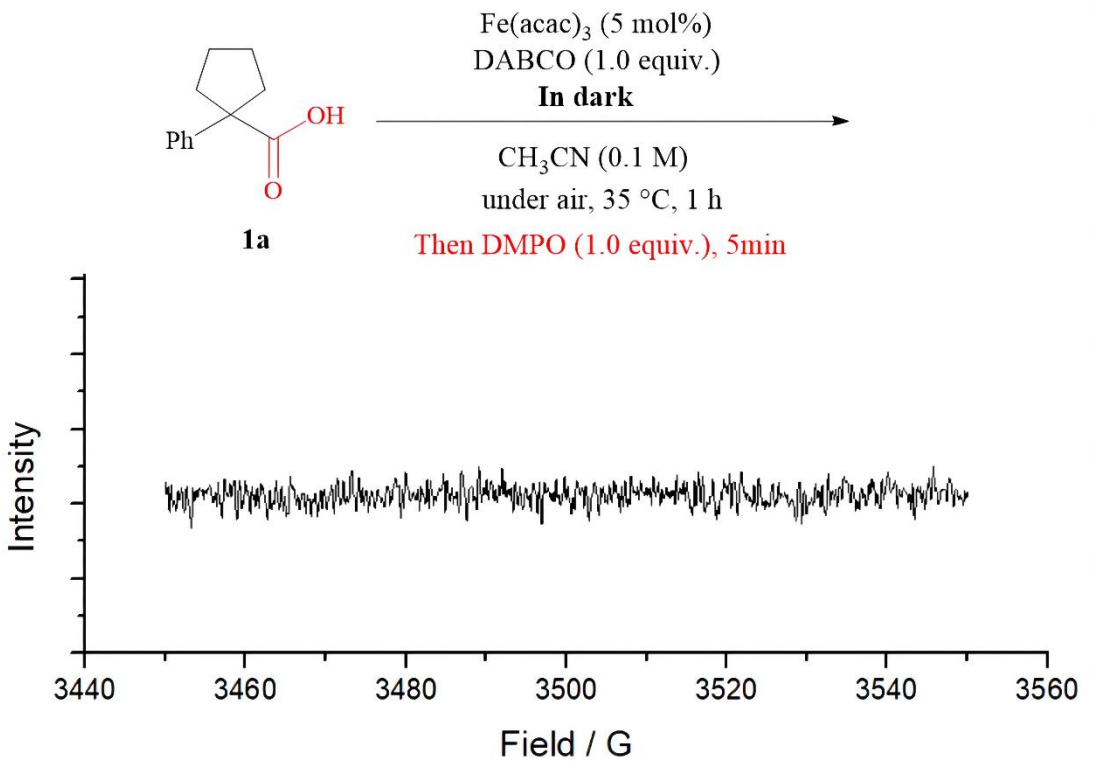


Figure S4: EPR experiments of the reaction proceeds in the dark.

General procedure for EPR studies: To a 25 mL quartz tube equipped with a magnetic stir bar, **1a** (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (3.6 mg, 5 mol%), DABCO (22.4 mg, 0.2 mmol, 1.0 equiv.) and CH_3CN (2 mL) were added. The resulting mixture was stirred in dark for 1h hours. Then, DMPO (5,5-dimethyl-1-pyrroline *N*-oxide, 0.2 mmol, 1.0 equiv.) was added. The reaction solution was continuously excited for 5 minutes. The solution sample was taken out into a small tube for EPR test.

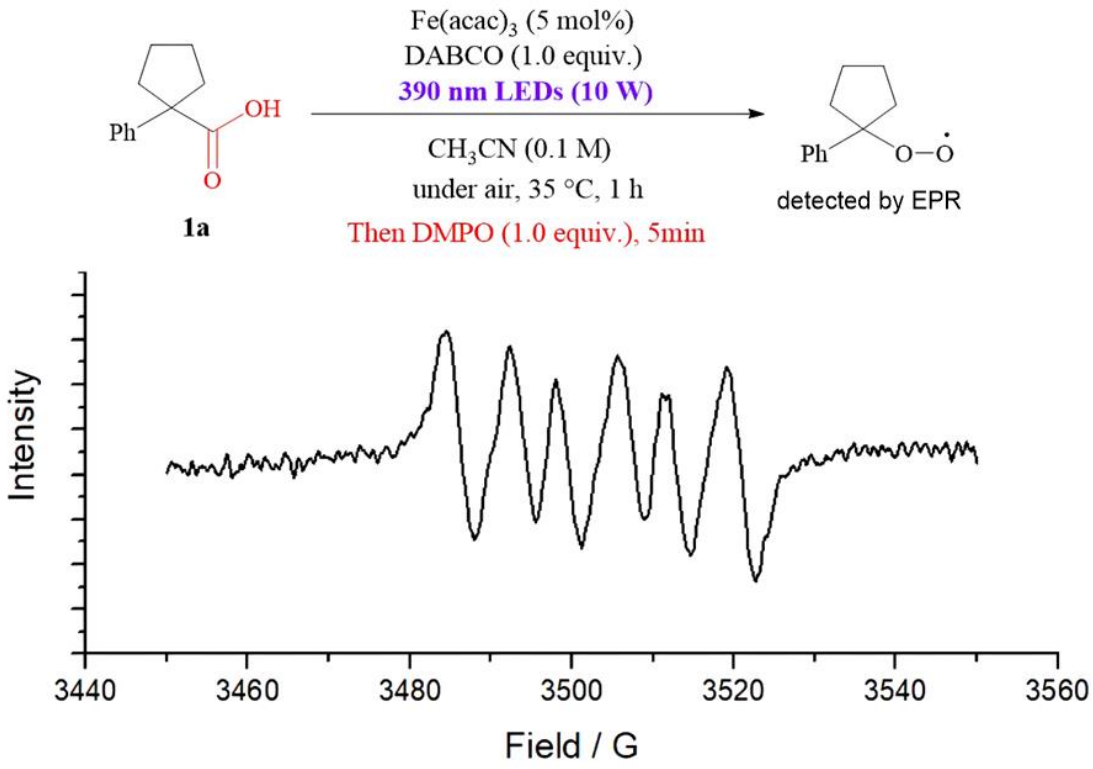


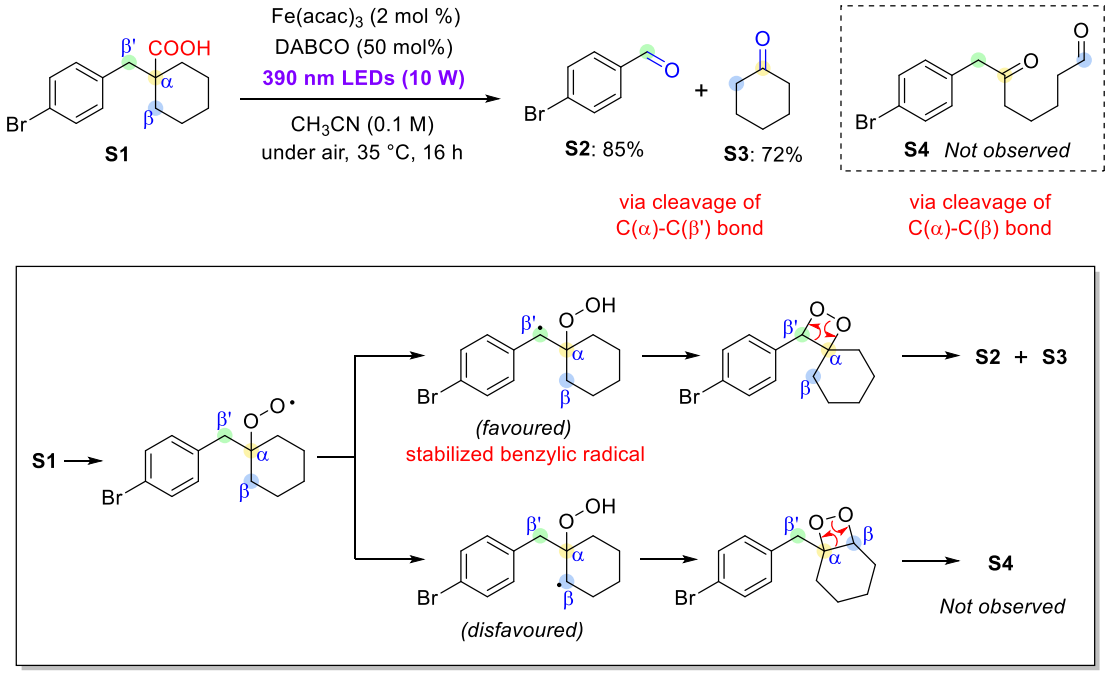
Figure S5: EPR experiments of the reaction proceeds under standard conditions.

General procedure for EPR studies: To a 25 mL quartz tube equipped with a magnetic stir bar, **1a** (0.2 mmol, 1.0 equiv.), $Fe(acac)_3$ (3.6 mg, 5 mol%), DABCO (22.4 mg, 0.2 mmol, 1.0 equiv.) and CH_3CN (2 mL) were added. The resulting mixture was stirred in air under a 10W LEDs and irradiated for 1h hours. Then, DMPO (5,5-dimethyl-1-pyrroline N-oxide, 0.2 mmol, 1.0 equiv.) was added. The reaction solution was continuously excited for 5 minutes. The solution sample was taken out into a small tube for EPR test.

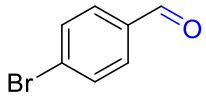
experiment	g value	α_N(G)	α_H(G)
1a+DMPO	2.0084	13.5	7.72

6.4 Study on the C(sp^3)−C(sp^3) bond cleavage and exploration of key β-carbon radical intermediates

In order to gain more insight into the mechanism of carbon-carbon single cleavage, several key substrates have been further investigated under the standard conditions. As shown in Scheme S1, we submitted compound **S1** into the reaction system and unfortunately we did not observe the formation of desired ring-opening product **S4**. Instead, two major products were isolated and identified as 4-bromobenzaldehyde **S2** and cyclohexanone **S3**, which may arise from the cleavage of C(α)−C(β') bond rather than C(α)−C(β) bond. During the investigation of its mechanism we speculated that the generation of **S2** and **S3** should be related to a stabilized C(β')-centered radical species (which is a benzylic radical). Such a process is favoured, followed by the formation of a dioxetane intermediate, which upon ring-opening leads to the formation of **S2** and **S3**. In contrast, the desired ring-opening product **S4** was not observed as the process of generating C(β)-centered radical is disfavoured, .



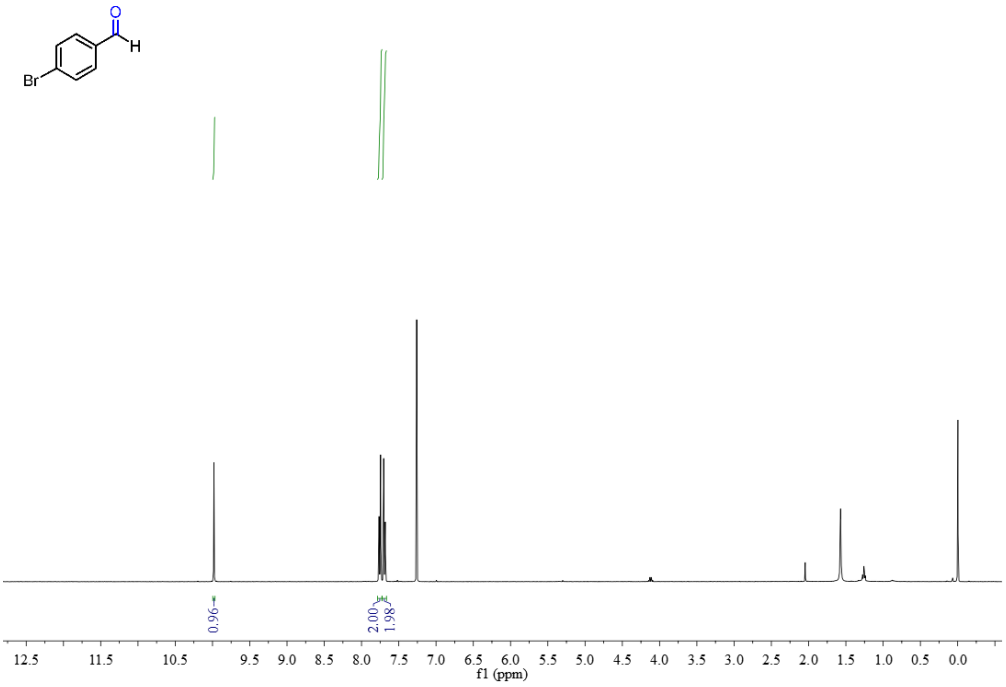
Scheme S1: iron photocatalytic reaction of substrate **S1**

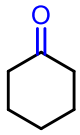


4-bromobenzaldehyde (**S2**): Followed the general procedure 1 with 1-(4-bromobenzyl)cyclohexane-1-carboxylic acid (59 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 20:1) to give 31 mg of the title compound (yellow oil; 85% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.02 (s, 1H), 7.79 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H).

Spectral data is in agreement with the literature.[9]

^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **S2**

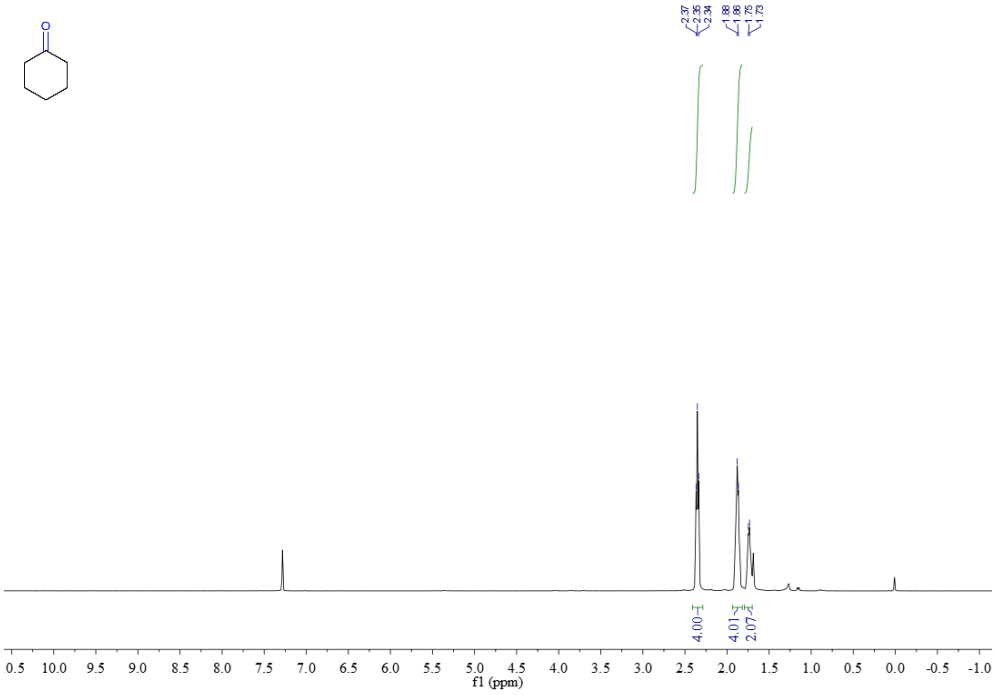




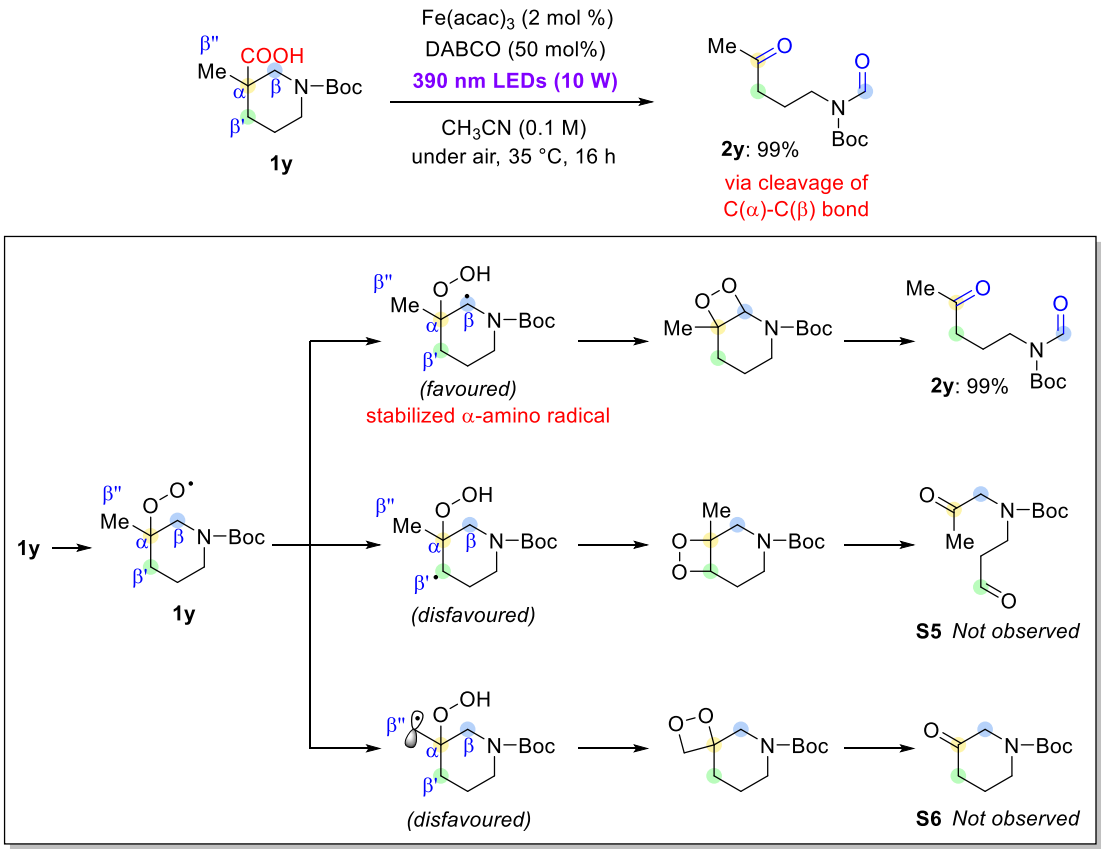
cyclohexanone (**S3**): Followed the general procedure 1 with 1-(4-bromobenzyl)cyclohexane-1-carboxylic acid (59 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 20:1) to give 14 mg of the title compound (colourless oil; 72% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 2.35 (t, *J* = 6.3 Hz, 4H), 1.95 – 1.82 (m, 4H), 1.78 – 1.70 (m, 2H).

Spectral data is in agreement with the literature.[10]

^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **S3**

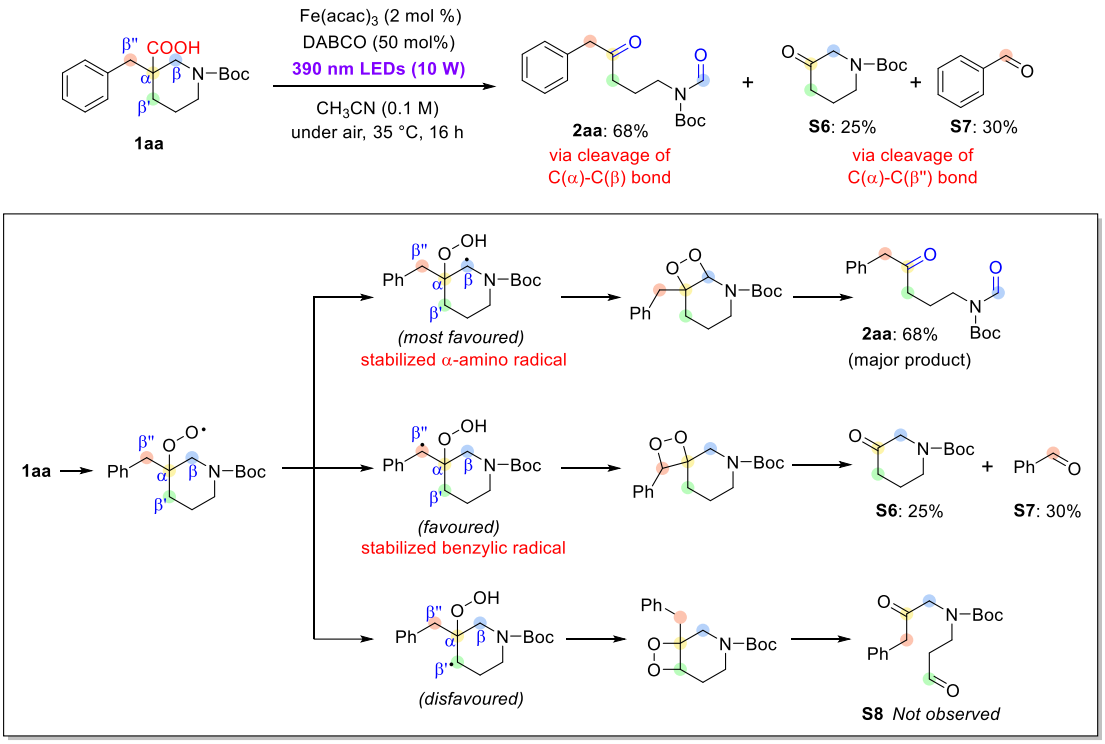


Another example that well explained the existence of a key carbon-centered radical intermediate is compound **1y**, which yielded the ring-opening product **2y** in 99% yield under the standard conditions (Scheme S2). The generation of **2y** may arise from the cleavage of C(α)−C(β) bond rather than C(α)−C(β') bond and C(α)−C(β'') bond. During the investigation of its mechanism we speculated that the generation of **2y** should be related to a stabilized C(β)-centered radical species (which is an α-amino radical). Such a process is favoured, followed by the formation of a dioxetane intermediate, which upon ring-opening leads to the formation of **2y**. In contrast, the formation of **S5** or **S6** was not observed as the process of generating C(β')-centered radical and C(β'')-centered radical is disfavoured.



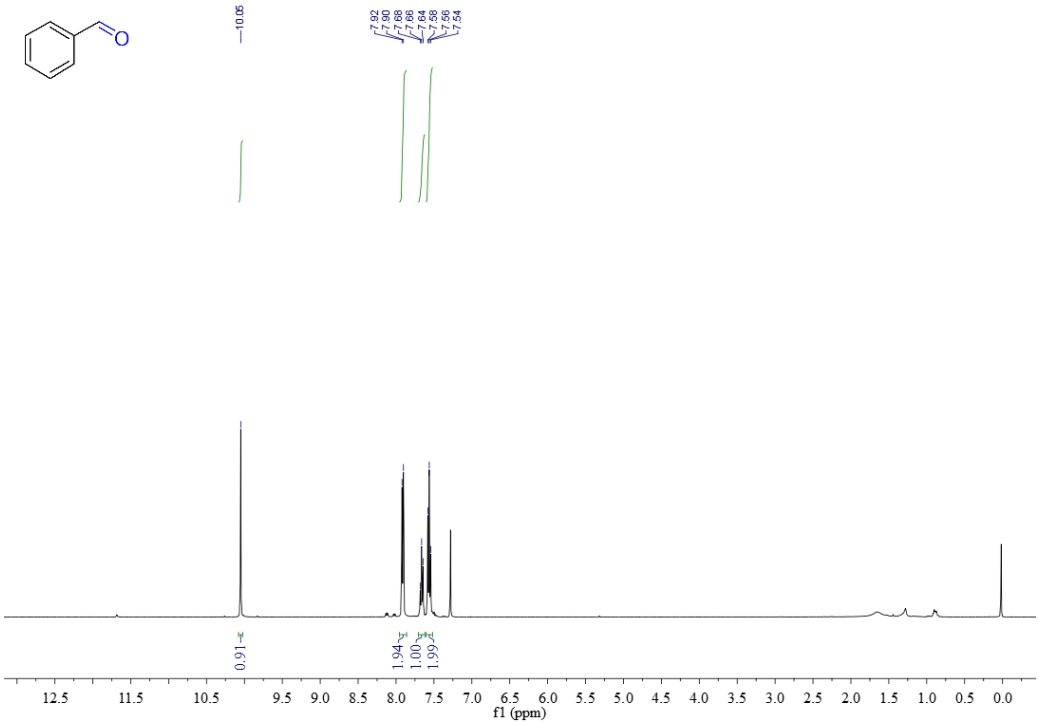
Scheme S2: iron photocatalytic reaction of substrate **1y**

The reaction of **1aa** under the standard conditions was further examined, delivering the desired ring-opening product **2aa** in 68% yield (Scheme S3). Notably, two minor products were also isolated and identified as **S6** and **S7** respectively, which provides evidence for the involvement of C(β'')-centered radical. The generation of **2aa** may arise from the cleavage of C(α)−C(β) bond, and the generation of **S6** and **S7** may arise from the cleavage of C(α)−C(β'') bond. During the investigation of their mechanisms we speculated that the generation of **2aa** should be related to a stabilized C(β)-centered radical species (which is an α-amino radical), and this synthetic route is the most favoured, followed by the formation of a dioxetane intermediate, which upon ring-opening leads to the formation of **2aa**. The generation of **S6** and **S7** should be related to a stabilized C(β'')-centered radical species (which is a benzylic radical), and this route is also favoured. In contrast, the formation of **S8** was not observed as the process of generating C(β')-centered radical is disfavoured.

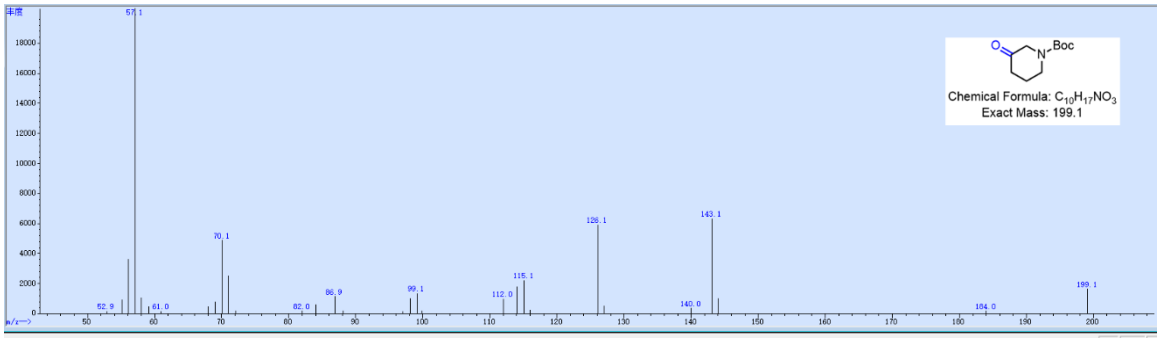


Scheme S3: iron photocatalytic reaction of substrate **1aa**

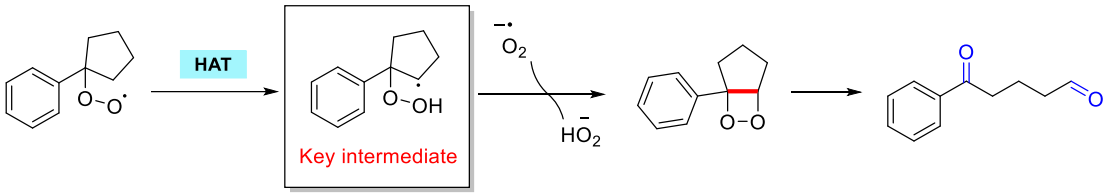
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **S7**



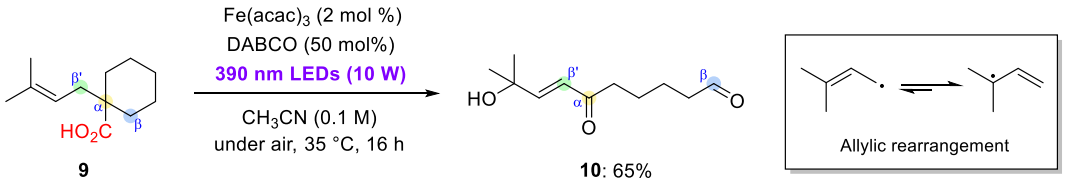
GC-MS spectrum of **S6**



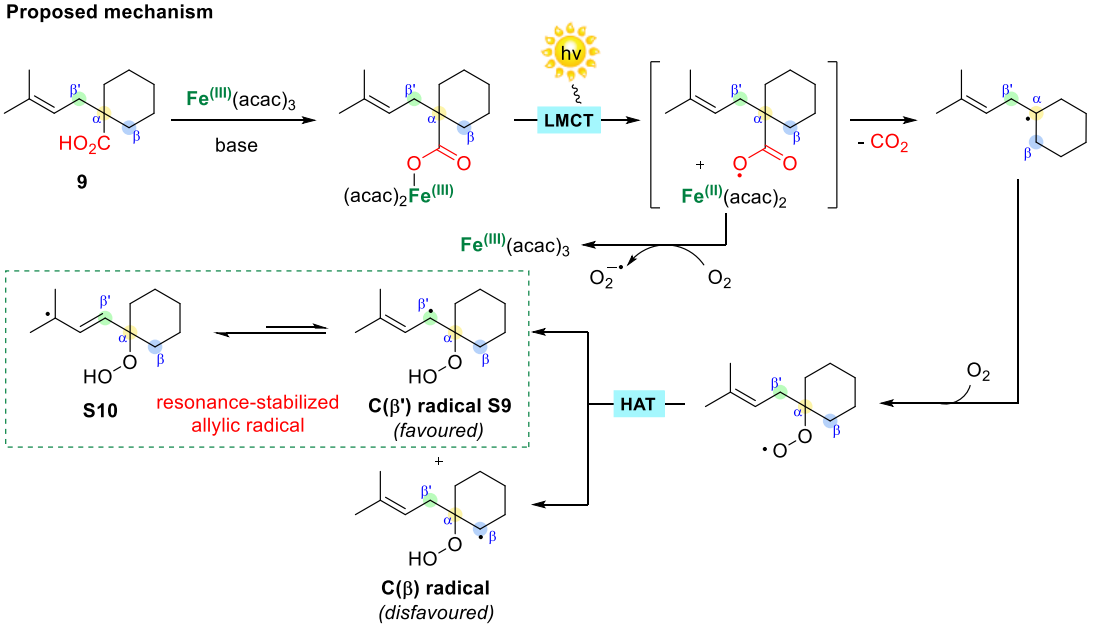
From the above three reactions we speculated that a key carbon-centered radical intermediate may be involved into the C(sp^3)–C(sp^3) cleavage process, and the selectivity is mainly based on the stability of the generated key carbon-centered radical. Therefore, we put forward the following hypothesis for ring-opening process:

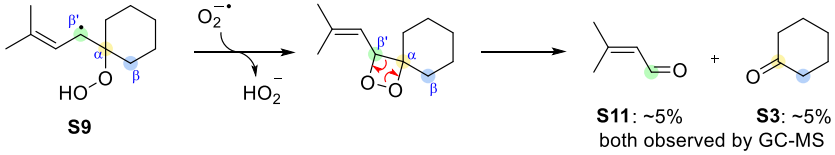


Later, we have tried several radical trapping experiment to directly capture this key carbon-centered radical intermediate (such as EPR experiment, TEMPO trapping reaction, 5-*exo*-trig cyclization, and radical clock reaction), but all the trapping reaction were failed as this carbon radical is very unstable. Meanwhile, we realized that allyl radical species are quite special, and they are prone to undergo allylic rearrangement to give a more stabilized radical species. Thus, we designed the substrate **9** containing an allyl moiety and submitted this compound to our developed iron photocatalytic conditions. The ring-opening product **10** after radical allylic rearrangement was successfully obtained in 65% yield, which further confirmed our hypothesis.



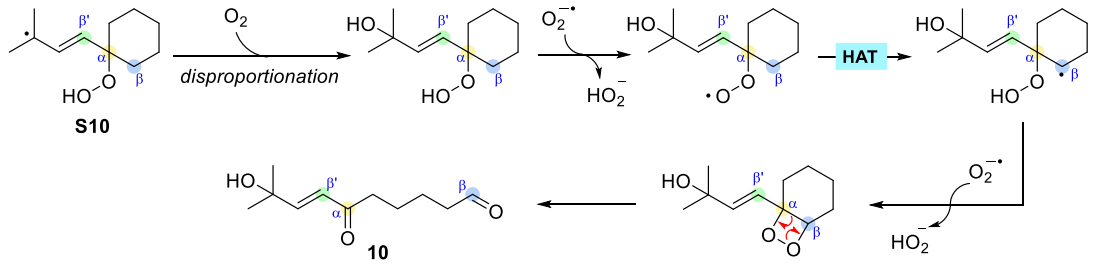
Proposed mechanism



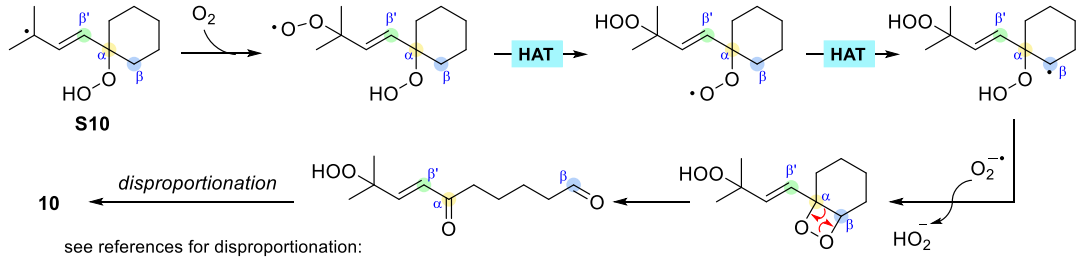


Two possible pathways are proposed for the generation of product **10** from **S10** species

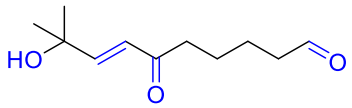
Pathway A



Pathway B

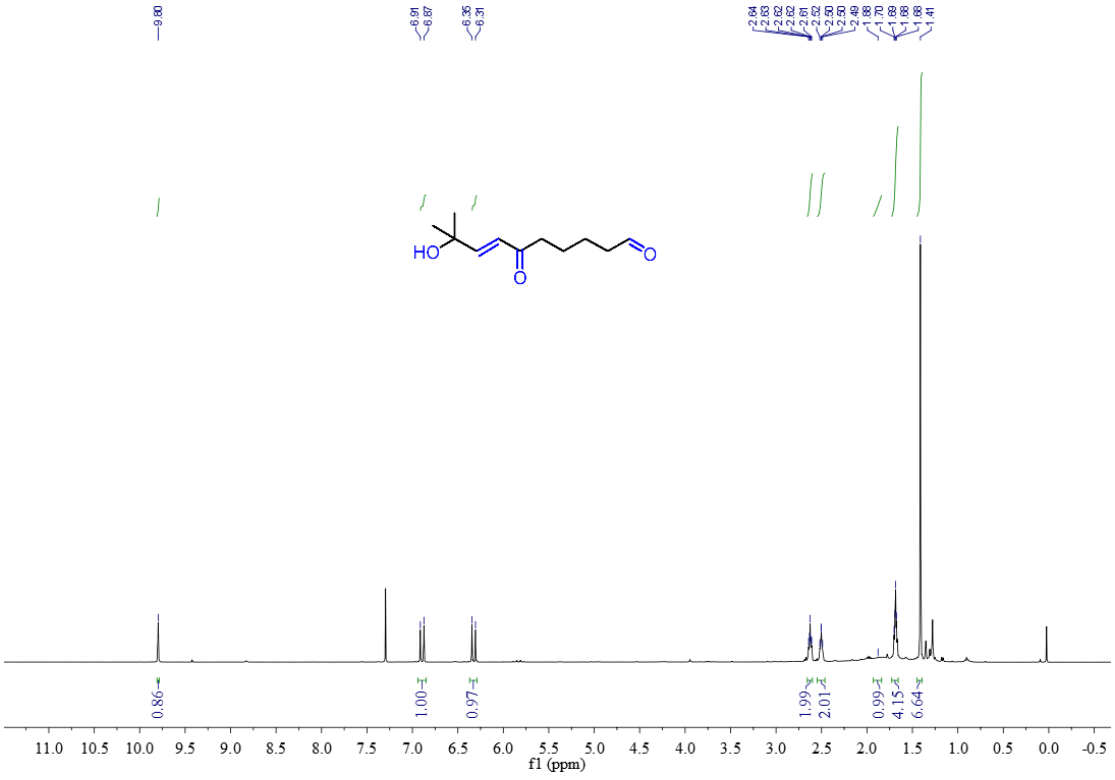


see references for disproportionation:
Adv. Synth. Catal. **2016**, *358*, 74; *J. Org. Chem.* **2016**, *81*, 7250;
Angew. Chem. Int. Ed. **2016**, *55*, 1094; *Chem. Asian J.* **2018**, *13*, 2410; etc.

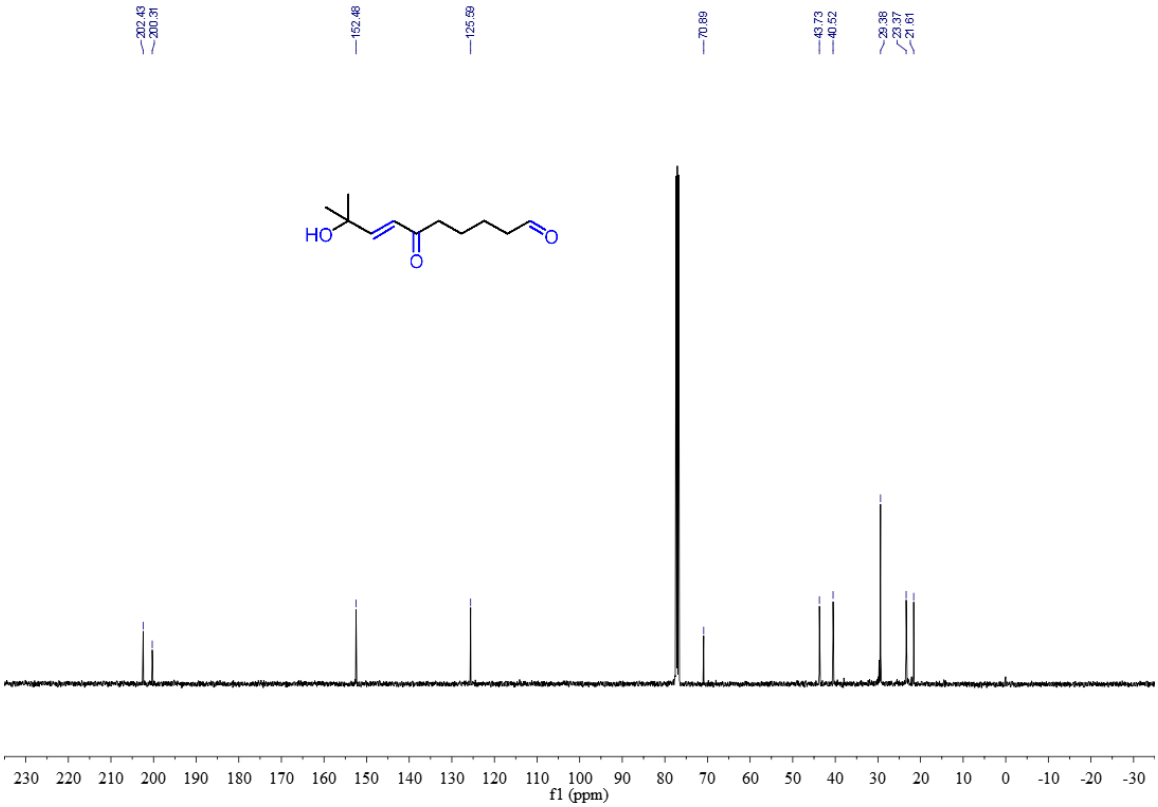


(*E*)-9-hydroxy-9-methyl-6-oxodec-7-enal (**10**): Followed the general procedure 2 with 1-(3-methylbut-2-en-1-yl)cyclohexane-1-carboxylic acid (39 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 26 mg of the title compound (colourless oil; 65% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.80 (s, 1H), 6.89 (d, *J* = 15.9 Hz, 1H), 6.33 (d, *J* = 15.9 Hz, 1H), 2.66 – 2.60 (m, 2H), 2.55 – 2.46 (m, 2H), 1.88 (br s, 1H), 1.73 – 1.66 (m, 4H), 1.41 (s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.4, 200.3, 152.5, 125.6, 70.9, 43.7, 40.5, 29.4, 23.4, 21.6; **HRMS (ESI)** calcd $C_{11}H_{19}O_3$ $[M + H]^+$: 199.1329, found: 199.1329.

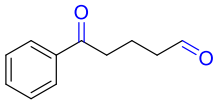
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **10**



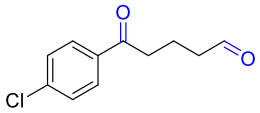
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **10**



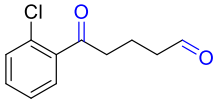
7. Characterization of the products



5-oxo-5-phenylpentanal (2a): Followed the general procedure 1 with 1-phenylcyclopentane-1-carboxylic acid (38 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 26 mg of the title compound (yellow oil; 75% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.84 (t, *J* = 1.3 Hz, 1H), 8.01 – 7.96 (m, 2H), 7.63 – 7.56 (m, 1H), 7.53 – 7.46 (m, 2H), 3.08 (t, *J* = 7.0 Hz, 2H), 2.63 (td, *J* = 7.1, 1.2 Hz, 2H), 2.16 – 2.07 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.1, 199.4, 136.7, 133.2, 128.7, 128.0, 43.1, 37.3, 16.6; **HRMS (ESI)** calcd $C_{11}H_{13}O_2$ $[M + Na]^+$: 177.0910, found: 177.0905.

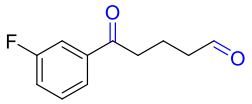


5-(4-chlorophenyl)-5-oxopentanal (2b): Followed the general procedure 1 with 1-(4-chlorophenyl)cyclopentane-1-carboxylic acid (55 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 27 mg of the title compound (yellow oil; 65% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.84 (t, *J* = 1.2 Hz, 1H), 7.97 – 7.87 (m, 2H), 7.51 – 7.42 (m, 2H), 3.05 (t, *J* = 7.0 Hz, 2H), 2.63 (td, *J* = 7.0, 1.2 Hz, 2H), 2.14 – 2.07 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.9, 198.2, 139.7, 135.0, 129.5, 129.0, 43.0, 37.3, 16.5; **HRMS (ESI)** calcd $C_{11}H_{12}{}^{35}ClO_2$ $[M + H]^+$: 211.0520, found: 211.0520.

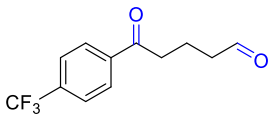


5-(2-chlorophenyl)-5-oxopentanal (2c): Followed the general procedure 1 with 1-(2-chlorophenyl)cyclopentane-1-carboxylic acid (55 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 24 mg of the title compound (yellow oil; 58% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.84 (s, 1H), 7.51 – 7.41 (m, 3H), 7.37 (m, 1H), 3.05 (t, *J* = 7.0 Hz, 2H), 2.63 (t, *J* = 7.1 Hz, 2H), 2.11 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.7, 201.8, 139.3, 131.8, 130.8, 130.6,

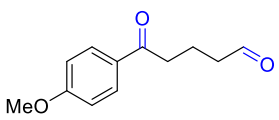
128.8, 127.0, 42.9, 41.7, 16.5; **HRMS (ESI)** calcd $C_{11}H_{12}{}^{35}ClO_2$ $[M + H]^+$: 211.0520 found: 211.0518.



5-(3-fluorophenyl)-5-oxopentanal (2d): Followed the general procedure 1 with 1-(3-fluorophenyl)cyclopentane-1-carboxylic acid (42 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 19 mg of the title compound (yellow oil; 49% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.81 (s, 1H), 7.74 (m, 1H), 7.64 (m, 1H), 7.45 (m, 1H), 7.30 – 7.24 (m, 1H), 3.04 (t, *J* = 7.0 Hz, 2H), 2.61 (t, *J* = 7.0 Hz, 2H), 2.08 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.9, 198.1, 162.9 (d, *J* = 248.0 Hz), 138.8 (d, *J* = 6.0 Hz), 130.4 (d, *J* = 7.6 Hz), 123.8 (d, *J* = 2.9 Hz), 120.2 (d, *J* = 21.4 Hz), 114.8 (d, *J* = 22.3 Hz), 43.0, 37.5, 16.4; **^{19}F NMR (376 MHz, $CDCl_3$)** δ -111.74 (dd, *J* = 15.4, 9.4 Hz); **HRMS (ESI)** calcd $C_{11}H_{12}FO_2$ $[M + H]^+$: 195.0816, found: 195.0818.

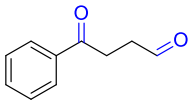


5-oxo-5-(4-(trifluoromethyl)phenyl)pentanal (2e): Followed the general procedure 1 with 1-(4-(trifluoromethyl)phenyl)cyclopentane-1-carboxylic acid (35 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 42 mg of the title compound (yellow oil; 72% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.81 (s, 1H), 8.05 (d, *J* = 8.2 Hz, 2H), 7.73 (d, *J* = 8.3 Hz, 2H), 3.07 (t, *J* = 7.0 Hz, 2H), 2.62 (t, *J* = 6.9, 0.9 Hz, 2H), 2.09 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.8, 198.4, 139.3, 134.5 (q, *J* = 32.7 Hz), 128.4, 125.8 (q, *J* = 3.6 Hz), 123.59 (q, *J* = 272.8 Hz), 42.9, 37.6, 16.3; **^{19}F NMR (376 MHz, $CDCl_3$)** δ -63.10 (s, 3H); **HRMS (ESI)** calcd $C_{12}H_{12}F_3O_2$ $[M + H]^+$: 245.0784, found: 245.0779.

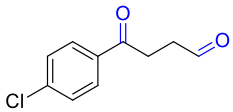


5-(4-methoxyphenyl)-5-oxopentanal (2f): Followed the general procedure 1 with 1-(4-methoxyphenyl)cyclopentane-1-carboxylic acid (44 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 28 mg of the title

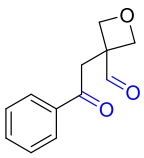
compound (yellow oil; 68% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.84 (t, *J* = 1.2 Hz, 1H), 8.01 – 7.94 (m, 2H), 7.01 – 6.93 (m, 2H), 3.91 (s, 3H), 3.03 (t, *J* = 7.0 Hz, 2H), 2.62 (td, *J* = 7.0, 1.2 Hz, 2H), 2.15 – 2.06 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.2, 198.0, 163.6, 130.3, 129.9, 113.8, 55.5, 43.2, 37.0, 16.8; **HRMS (ESI)** calcd $C_{12}H_{15}O_3$ $[M + H]^+$: 207.1016, found: 207.1017.



4-oxo-4-phenylbutanal (2g): Followed the general procedure 1 with 1-phenylcyclobutane-1-carboxylic acid (35 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 24 mg of the title compound (yellow oil; 75% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.95 (s, 1H), 8.03 (d, *J* = 7.3 Hz, 2H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 3.38 (t, *J* = 6.3 Hz, 2H), 2.98 (t, *J* = 6.3 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.7, 197.9, 136.4, 133.4, 128.7, 128.1, 37.6, 31.1; **HRMS (ESI)** calcd $C_{10}H_{11}O_2$ $[M + H]^+$: 163.0754, found: 163.0758.

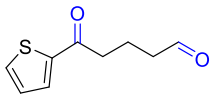


4-(4-chlorophenyl)-4-oxobutanal (2h): Followed the general procedure 1 with 1-(4-chlorophenyl)cyclobutane-1-carboxylic acid (42 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 30 mg of the title compound (yellow oil; 76% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.89 (s, 1H), 7.96 – 7.89 (m, 2H), 7.49 – 7.41 (m, 2H), 3.28 (t, *J* = 6.3 Hz, 2H), 2.94 (t, *J* = 6.3 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.5, 196.7, 139.8, 134.8, 129.5, 129.0, 37.6, 31.0; **HRMS (ESI)** calcd $C_{10}H_{10}{}^{35}ClO_2$ $[M + H]^+$: 197.0364, found: 197.0360.

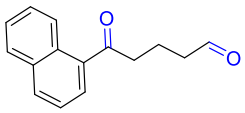


3-(2-oxo-2-phenylethyl)oxetane-3-carbaldehyde (2i): Followed the general procedure 1 with 6-phenyl-2-oxaspiro[3.3]heptane-6-carboxylic acid (44 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to

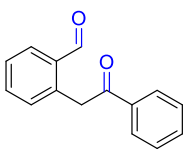
give 30 mg of the title compound (yellow oil; 74% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.21 (s, 1H), 7.97 – 7.92 (m, 2H), 7.63 – 7.57 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 5.02 (d, *J* = 7.0 Hz, 2H), 4.66 (d, *J* = 7.0 Hz, 2H), 3.78 (s, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.3, 196.7, 135.8, 133.9, 128.8, 128.2, 50.3, 42.2; **HRMS (ESI)** calcd $C_{12}H_{13}O_3$ $[M + H]^+$: 205.0859, found: 205.0858.



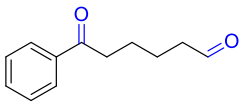
5-oxo-5-(thiophen-2-yl)pentanal (2j): Followed the general procedure 1 with 1-(thiophen-2-yl)cyclopentane-1-carboxylic acid (40 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 23 mg of the title compound (yellow oil; 62% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.83 (t, *J* = 1.2 Hz, 1H), 7.75 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.67 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.16 (dd, *J* = 4.9, 3.8 Hz, 1H), 3.02 (t, *J* = 7.1 Hz, 2H), 2.63 (td, *J* = 7.0, 1.1 Hz, 2H), 2.11 (p, *J* = 7.1 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.9, 192.4, 144.1, 133.7, 132.0, 128.2, 128.1, 43.0, 38.0, 16.9; **HRMS (ESI)** calcd $C_9H_{11}O_2S$ $[M + H]^+$: 183.0474, found: 183.0469.



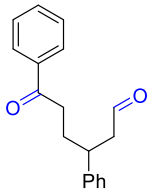
5-(naphthalen-1-yl)-5-oxopentanal (2k): Followed the general procedure 1 with 1-(naphthalen-1-yl)cyclopentane-1-carboxylic acid (48 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 29 mg of the title compound (yellow oil; 64% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.86 (s, 1H), 8.63 (d, *J* = 8.5 Hz, 1H), 8.03 (d, *J* = 8.2 Hz, 1H), 7.91 (dd, *J* = 7.0, 4.1 Hz, 2H), 7.62 (m, 1H), 7.60 – 7.50 (m, 2H), 3.17 (t, *J* = 7.1 Hz, 2H), 2.67 (t, *J* = 7.1, 2H), 2.18 (m, 2H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 203.6, 202.0, 135.7, 134.0, 132.8, 130.1, 128.5, 128.1, 127.7, 126.5, 125.7, 124.4, 43.1, 40.7, 17.0; **HRMS (ESI)** calcd $C_{15}H_{15}O_2$ $[M + H]^+$: 227.1067, found: 227.1063.



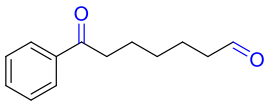
2-(2-oxo-2-phenylethyl)benzaldehyde (2l): Followed the general procedure 1 with 2-phenyl-2,3-dihydro-1H-indene-2-carboxylic acid (37 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 29 mg of the title compound (yellow oil; 82% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.07 (s, 1H), 8.14 – 8.08 (m, 2H), 7.94 – 7.87 (m, 1H), 7.67 – 7.60 (m, 2H), 7.60 – 7.52 (m, 3H), 7.36 – 7.32 (m, 1H), 4.80 (s, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.9, 193.3, 137.1, 136.3, 135.2, 134.5, 133.7, 133.2, 132.8, 128.7, 128.3, 127.8, 43.5; **HRMS (ESI)** calcd $C_{15}H_{13}O_2$ $[M + H]^+$: 225.0910, found: 225.0909.



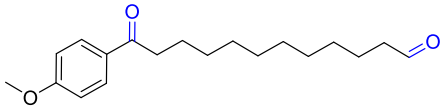
6-oxo-6-phenylhexanal (2m): Followed the general procedure 1 with 1-phenylcyclohexane-1-carboxylic acid (41 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 20:1) to give 25 mg of the title compound (yellow oil; 67% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.82 (s, 1H), 8.02 – 7.96 (m, 2H), 7.60 (t, *J* = 7.3 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 3.05 (t, *J* = 6.8 Hz, 2H), 2.55 (t, *J* = 6.4 Hz, 2H), 1.78 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.4, 199.8, 136.9, 133.1, 128.7, 128.0, 43.8, 38.2, 23.6, 21.8; **HRMS (ESI)** calcd $C_{12}H_{15}O_2$ $[M + H]^+$: 191.1067, found: 191.1077.



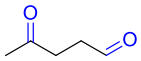
6-oxo-3,6-diphenylhexanal (2n): Followed the general procedure 2 with 1,4-diphenylcyclohexane-1-carboxylic acid (56 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 39 mg of the title compound (white solid; 74% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.71 (s, 1H), 7.86 (d, *J* = 7.5 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 2H), 7.27 (m, 3H), 3.33 (m, 1H), 2.97 – 2.87 (m, 1H), 2.87 – 2.74 (m, 3H), 2.21 (m, 1H), 2.05 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.6, 199.7, 142.9, 136.8, 133.1, 128.9, 128.6, 128.0, 127.6, 127.0, 50.8, 39.4, 36.2, 30.6; **HRMS (ESI)** calcd $C_{18}H_{19}O_2$ $[M + H]^+$: 267.1380, found: 267.1381.



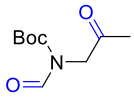
7-oxo-7-phenylheptanal (2o): Followed the general procedure 1 with 1-phenylcycloheptane-1-carboxylic acid (44 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 24 mg of the title compound (yellow oil; 58% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.81 (s, 1H), 8.03 – 7.95 (m, 2H), 7.60 (t, *J* = 7.3 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 3.03 (t, *J* = 7.3 Hz, 2H), 2.50 (t, *J* = 7.3 Hz, 2H), 1.86 – 1.77 (m, 2H), 1.77 – 1.68 (m, 2H), 1.52 – 1.42 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.7, 200.2, 137.0, 133.1, 128.6, 128.06, 43.8, 38.3, 28.8, 23.9, 21.9; **HRMS (ESI)** calcd $C_{13}H_{17}O_2$ $[M + H]^+$: 205.1223, found: 205.1226.



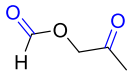
12-(4-methoxyphenyl)-12-oxododecanal (2p): Followed the general procedure 2 with 1-(4-methoxyphenyl)cyclododecane-1-carboxylic acid (64 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 40 mg of the title compound (white solid; 65% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.79 (t, *J* = 1.8 Hz, 1H), 8.01 – 7.95 (m, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 3.90 (s, 3H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.45 (td, *J* = 7.4, 1.8 Hz, 2H), 1.79 – 1.72 (m, 2H), 1.72 – 1.61 (m, 4H), 1.41 – 1.32 (m, 10H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 203.1, 199.3, 163.3, 130.4, 130.2, 113.7, 55.5, 44.0, 38.3, 29.5, 29.4, 29.4, 29.4, 29.2, 24.6, 22.1; **HRMS (ESI)** calcd $C_{19}H_{29}O_3$ $[M + H]^+$: 305.2111, found: 305.2113.



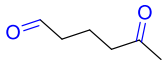
4-oxopentanal (2q): Followed the general procedure 2 with 1-methylcyclobutane-1-carboxylic acid (22 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 20 mg of the title compound (yellow oil; 98% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.74 (s, 1H), 2.70 (s, 4H), 2.15 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 206.5, 200.5, 37.5, 35.5, 29.9; **HRMS (ESI)** calcd $C_5H_9O_2$ $[M + H]^+$: 101.0597, found: 101.0595.



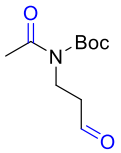
***tert*-butyl formyl(2-oxopropyl)carbamate (2r):** Followed the general procedure 2 with 1-(tert-butoxycarbonyl)-3-methylazetidine-3-carboxylic acid (43 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 40 mg of the title compound (colourless oil; 99% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.22 (s, 1H), 4.41 (s, 2H), 2.20 (s, 3H), 1.53 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.4, 162.4, 151.7, 84.7, 49.5, 27.9, 26.9; **HRMS (ESI)** calcd $C_9H_{16}NO_4$ $[M + H]^+$: 202.1074, found: 202.1072.



2-oxopropyl formate (2s): Followed the general procedure 2 with 3-methyloxetane-3-carboxylic acid (23 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 17 mg of the title compound (yellow oil; 85% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.20 (s, 1H), 4.79 (s, 2H), 2.23 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.5, 159.9, 67.6, 26.1; **HRMS (ESI)** calcd $C_4H_7O_3$ $[M + H]^+$: 103.0390, found: 103.0387.

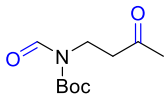


5-oxohexanal (2t): Followed the general procedure 2 with 1-methylcyclopentane-1-carboxylic acid (26 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 20mg of the title compound (yellow oil; 87% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.79 (s, 1H), 2.53 (m, 4H), 2.17 (s, 3H), 1.98 – 1.87 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 208.0, 201.9, 42.94, 42.3, 29.7, 16.0; **HRMS (ESI)** calcd $C_6H_{11}O_2$ $[M + H]^+$: 115.0754, found: 115.0752.

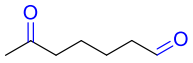


***tert*-butyl acetyl(3-oxopropyl)carbamate (2u):** Followed the general procedure 1 with 1-(tert-butoxycarbonyl)-2-methylpyrrolidine-2-carboxylic acid (46 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 35 mg of the title compound (yellow oil; 82% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.63 (s, 1H), 3.89 (t, *J* = 6.9 Hz, 2H), 2.53 (t, *J* = 6.9 Hz, 2H), 2.32 (s, 3H), 1.39 (s,

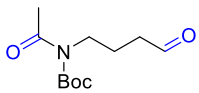
9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 200.5, 173.0, 152.7, 83.8, 43.3, 38.4, 28.0, 26.9; **HRMS (ESI)** calcd $C_{10}H_{18}NO_4$ $[M + H]^+$: 216.1230, found: 216.1225.



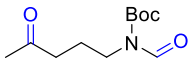
***tert*-butyl formyl(3-oxobutyl)carbamate (2v):** Followed the general procedure 2 with 1-(tert-butoxycarbonyl)-3-methylpyrrolidine-3-carboxylic acid (46 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 42 mg of the title compound (yellow oil; 97% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.12 (s, 1H), 3.91 – 3.78 (m, 2H), 2.71 – 2.64 (m, 2H), 2.15 (s, 3H), 1.54 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 206.2, 162.9, 152.1, 84.4, 41.8, 35.8, 30.0, 28.0; **HRMS (ESI)** calcd $C_{10}H_{18}NO_4$ $[M + H]^+$: 216.1230, found: 216.1231.



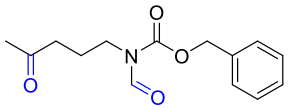
6-oxoheptanal (2w): Followed the general procedure 2 with 1-methylcyclohexane-1-carboxylic acid (28 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 23.1 mg of the title compound (yellow oil; 90% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.79 (s, 1H), 2.49 (m, 4H), 2.17 (s, 3H), 1.71 – 1.57 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 208.6, 202.3, 43.7, 43.3, 30.0, 23.1, 21.5; **HRMS (ESI)** calcd $C_7H_{13}O_2$ $[M + H]^+$: 129.0910, found: 129.0913.



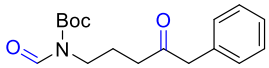
***tert*-butyl acetyl(4-oxobutyl)carbamate (2x):** Followed the general procedure 2 with 1-(tert-butoxycarbonyl)-2-methylpiperidine-2-carboxylic acid (48 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 43 mg of the title compound (yellow oil; 94% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.75 (s, 1H), 3.74 – 3.64 (m, 2H), 2.47 – 2.42 (m, 5H), 1.85 (m, 2H), 1.52 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.5, 173.1, 153.1, 83.3, 43.3, 41.1, 28.0, 26.9, 21.1; **HRMS (ESI)** calcd $C_{11}H_{20}NO_4$ $[M + H]^+$: 230.1387, found: 230.1383.



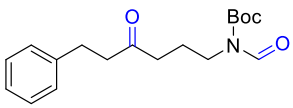
***tert*-butyl formyl(4-oxopentyl)carbamate (2y)**: Followed the general procedure 2 with 1-(tert-butoxycarbonyl)-3-methylpiperidine-3-carboxylic acid (45 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 43 mg of the title compound (yellow oil; 99% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.10 (s, 1H), 3.53 (t, *J* = 6.9 Hz, 2H), 2.39 (t, *J* = 7.2 Hz, 2H), 2.07 (s, 3H), 1.80 – 1.70 (m, 2H), 1.49 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 207.5, 163.1, 152.4, 84.0, 40.4, 39.7, 29.8, 28.0, 22.2; **HRMS (ESI)** calcd $C_{11}H_{20}NO_4$ $[M + H]^+$: 230.1387, found: 230.1386.



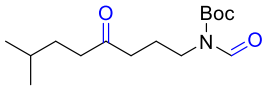
benzyl formyl(4-oxopentyl)carbamate (2z): Followed the general procedure 2 with 1-((benzyloxy)carbonyl)-3-methylpiperidine-3-carboxylic acid (56 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 48 mg of the title compound (yellow oil; 92% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.26 (s, 1H), 7.45 – 7.37 (m, 5H), 5.33 (d, *J* = 3.3 Hz, 2H), 3.69 (t, *J* = 6.9 Hz, 2H), 2.44 (t, *J* = 7.2 Hz, 2H), 2.12 (s, 3H), 1.85 (p, *J* = 7.1 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 207.6, 162.9, 154.0, 134.7, 129.0, 128.9, 128.6, 69.0, 40.4, 40.1, 29.9, 22.3; **HRMS (ESI)** calcd $C_{14}H_{18}NO_4$ $[M + H]^+$: 264.1230, found: 264.1232.



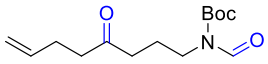
***tert*-butyl formyl(4-oxo-5-phenylpentyl)carbamate (2aa):** Followed the general procedure 2 with 3-benzyl-1-(tert-butoxycarbonyl)piperidine-3-carboxylic acid (64 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 41 mg of the title compound (yellow oil; 68% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.17 (s, 1H), 7.36 (dd, *J* = 10.0, 4.5 Hz, 2H), 7.31 – 7.27 (m, 1H), 7.25 – 7.20 (m, 2H), 3.71 (s, 2H), 3.59 (t, *J* = 7.0 Hz, 2H), 2.48 (t, *J* = 7.2 Hz, 2H), 1.81 (dd, *J* = 14.1, 7.1 Hz, 2H), 1.56 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 207.2, 163.2, 152.4, 134.2, 129.5, 128.8, 127.1, 84.1, 50.2, 39.7, 38.9, 28.1, 22.3; **HRMS (ESI)** calcd $C_{17}H_{23}NO_4Na$ $[M + Na]^+$: 328.1519, found: 328.1528.



***tert*-butyl formyl(4-oxo-6-phenylhexyl)carbamate (2bb)**: Followed the general procedure 2 with 1-(tert-butoxycarbonyl)-3-phenethylpiperidine-3-carboxylic acid (64 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 46 mg of the title compound (yellow oil; 72% yield); **^{1}H NMR (600 MHz, $CDCl_3$)** δ 7.38 (t, *J* = 7.3 Hz, 2H), 7.35 – 7.31 (m, 1H), 7.15 – 7.10 (m, 2H), 7.07 (d, *J* = 7.3 Hz, 2H), 6.86 – 6.79 (m, 2H), 6.78 (d, *J* = 11.5 Hz, 1H), 5.66 (dd, *J* = 11.4, 9.4 Hz, 1H), 5.27 – 5.14 (m, 1H), 4.67 (t, *J* = 8.6 Hz, 1H), 4.21 (dd, *J* = 8.5, 7.4 Hz, 1H), 3.78 (s, 3H); **^{13}C NMR (150 MHz, $CDCl_3$)** δ 157.2, 155.9, 135.3, 135.3, 129.5, 128.9, 128.6, 128.2, 128.0, 123.4, 114.1, 67.1, 55.4, 54.9. **HRMS (ESI)** calcd $C_{19}H_{28}NO_4$ [M + H]$^+$: 320.1856, found: 320.1855.

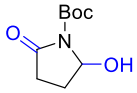


***tert*-butyl formyl(7-methyl-4-oxooctyl)carbamate (2cc)**: Followed the general procedure 2 with 1-(tert-butoxycarbonyl)-3-isopentylpiperidine-3-carboxylic acid (60 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 46 mg of the title compound (yellow oil; 80% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.18 (s, 1H), 3.60 (t, *J* = 7.0 Hz, 2H), 2.46 – 2.36 (m, 4H), 1.87 – 1.78 (m, 2H), 1.56 (s, 9H), 1.55 – 1.41 (m, 5H), 0.89 (d, *J* = 6.4 Hz, 7H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 210.2, 163.2, 152.5, 84.1, 40.8, 39.9, 39.6, 32.6, 28.1, 27.7, 22.4, 22.3; **HRMS (ESI)** calcd $C_{15}H_{28}NO_4$ [M + H]$^+$: 286.2013, found: 286.2013.

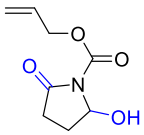


***tert*-butyl formyl(4-oxooct-7-en-1-yl)carbamate (2dd):** Followed the general procedure 2 with 3-(but-3-en-1-yl)-1-(tert-butoxycarbonyl)piperidine-3-carboxylic acid (56 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 42 mg of the title compound (yellow oil; 78% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.18 (s, 1H), 5.86 – 5.77 (m, 1H), 5.04 (d, *J* = 17.2 Hz, 1H), 4.99 (d, *J* = 10.3 Hz, 1H), 3.61 (t, *J* = 6.9 Hz, 2H), 2.51 (t, *J* = 7.3 Hz, 2H), 2.43 (t, *J* = 7.2 Hz, 2H), 2.39 – 2.30 (m, 2H), 1.89 – 1.80 (m, 2H), 1.57 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 208.9, 163.2, 152.4, 137.1, 115.3, 84.1, 41.8, 39.8, 39.7, 28.5,

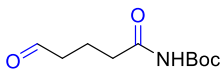
28.1, 27.7, 22.3; **HRMS (ESI)** calcd $C_{14}H_{24}NO_4$ $[M + H]^+$: 270.1700, found: 270.1702.



***tert*-butyl 2-hydroxy-5-oxopyrrolidine-1-carboxylate (2ee)**: Followed the general procedure 2 with 1-((tert-butoxycarbonyl)amino)cyclobutane-1-carboxylic acid (43 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 38 mg of the title compound (colourless oil; 95% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 5.78 – 5.68 (m, 1H), 3.93 (s, 1H), 2.84 – 2.69 (m, 1H), 2.48 – 2.38 (m, 1H), 2.27 – 2.15 (m, 1H), 2.06 – 1.94 (m, 1H), 1.56 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.1, 150.7, 84.1, 82.2, 30.6, 28.1, 25.4; **HRMS (ESI)** calcd $C_9H_{16}NO_4$ $[M + H]^+$: 202.1074, found: 202.1077.

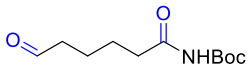


allyl 2-hydroxy-5-oxopyrrolidine-1-carboxylate (2ff): Followed the general procedure 2 with 1-(((allyloxy)carbonyl)amino)cyclobutane-1-carboxylic acid (40 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 29 mg of the title compound (colourless oil; 72% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ6.06 – 5.95 (m, 1H), 5.86 – 5.79 (m, 1H), 5.54 – 5.43 (m, 1H), 5.39 – 5.29 (m, 1H), 4.90 – 4.76 (m, 2H), 3.88 (s, 1H), 2.92 – 2.77 (m, 1H), 2.57 – 2.46 (m, 1H), 2.35 – 2.22 (m, 1H), 2.13 – 2.02 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 172.8, 152.1, 131.0, 119.5, 82.3, 67.6, 30.5, 25.6; **HRMS (ESI)** calcd $C_9H_{14}NO_4$ $[M + H]^+$: 200.0917, found: 200.0916.

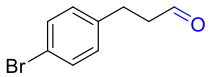


***tert*-butyl (5-oxopentanoyl)carbamate (2gg):** Followed the general procedure 2 with 1-((tert-butoxycarbonyl)amino)cyclopentane-1-carboxylic acid (46 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 35 mg of the title compound (colourless oil; 82% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.80 (s, 1H), 7.50 (s, 1H), 2.82 (t, *J* = 7.2 Hz, 2H), 2.58 (t, *J* = 7.2 Hz, 2H), 2.05 –

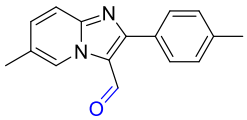
1.95 (m, 2H), 1.51 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 202.0, 174.1, 150.5, 82.7, 43.0, 35.0, 28.0, 16.6; **HRMS (ESI)** calcd $C_{10}H_{18}NO_4$ $[M + H]^+$: 216.1230, found: 216.1234.



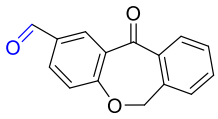
***tert*-butyl (6-oxohexanoyl)carbamate (2hh):** Followed the general procedure 2 with 1-((tert-butoxycarbonyl)amino)cyclohexane-1-carboxylic acid (49 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 30 mg of the title compound (yellow oil; 65% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.79 (s, 1H), 7.46 (s, 1H), 2.85 – 2.76 (m, 2H), 2.61 – 2.46 (m, 2H), 1.84 – 1.65 (m, 4H), 1.51 (s, 9H); **^{13}C NMR (400 MHz, $CDCl_3$)** δ 202.4, 174.4, 150.6, 82.6, 43.6, 35.7, 28.0, 23.6, 21.5; **HRMS (ESI)** calcd $C_{11}H_{20}NO_4$ $[M + H]^+$: 230.1387, found: 230.1389.



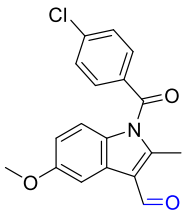
3-(4-bromophenyl)propanal(4a): Followed the general procedure 3 with 5-(4-bromophenyl)pentanoic acid (52 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 15:1) to give 25 mg of the title compound (yellow oil; 58% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.84 (d, *J* = 0.7 Hz, 1H), 7.44 (d, *J* = 8.2 Hz, 2H), 7.11 (d, *J* = 8.2 Hz, 2H), 2.94 (t, *J* = 7.4 Hz, 2H), 2.81 (t, *J* = 7.2 Hz, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 201.1, 139.4, 131.7, 130.1, 120.1, 45.1, 27.5; **HRMS (ESI)** calcd $C_9H_{10}{}^{79}BrO$ $[M + H]^+$: 212.9910, found: 212.9913.



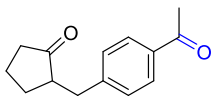
6-methyl-2-(p-tolyl)imidazo[1,2-a]pyridine-3-carbaldehyde (4b): Followed the general procedure 3 with 2-(6-methyl-2-(p-tolyl)imidazo[1,2-a]pyridin-3-yl)acetic acid (56 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 47 mg of the title compound (yellow oil; 95% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.04 (s, 1H), 9.49 (s, 1H), 7.75 – 7.69 (m, 3H), 7.47 – 7.41 (m, 1H), 7.35 (d, *J* = 7.9 Hz, 2H), 2.47 – 2.43 (m, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 179.5, 158.4, 146.7, 139.9, 133.3, 129.7, 129.6, 126.8, 125.4, 120.5, 116.6, 21.4, 18.4; **HRMS (ESI)** calcd $C_{16}H_{15}N_2O$ $[M + H]^+$: 250.1179 found: 251.1176.



11-oxo-6,11-dihydrodibenzo[b,e]oxepine-2-carbaldehyde **(4c):** Followed the general procedure 3 with isoxepac (54 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 47 mg of the title compound (yellow oil; 75% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.03 (s, 1H), 8.76 (d, *J* = 2.0 Hz, 1H), 8.10 – 8.03 (m, 1H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.63 (dd, *J* = 10.7, 4.2 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 7.4 Hz, 1H), 7.20 (d, *J* = 8.6 Hz, 1H), 5.32 (s, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 190.5, 190.3, 165.6, 140.4, 137.5, 134.6, 133.5, 133.3, 131.0, 129.8, 129.4, 128.2, 125.0, 122.2, 73.7; **HRMS (ESI)** calcd $C_{15}H_{12}O_3$ $[M + H]^+$: 239.0703, found: 239.0707.

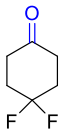


1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indole-3-carbaldehyde (4d): Followed the general procedure 3 with indometacin (71 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 5:1) to give 54 mg of the title compound (yellow oil; 82% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.34 (s, 1H), 7.84 (s, 1H), 7.73 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 8.6 Hz, 2H), 6.76 (d, *J* = 1.4 Hz, 2H), 3.90 (s, 3H), 2.79 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 185.8, 168.3, 157.2, 148.6, 141.0, 132.1, 131.7, 130.6, 129.5, 127.0, 118.4, 114.3, 113.9, 103.3, 55.8, 12.7; **HRMS (ESI)** calcd $C_{18}H_{15}{}^{35}ClNO_3$ $[M + H]^+$: 328.0735, found: 328.0738.

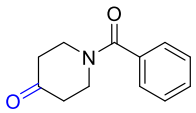


2-(4-acetylbenzyl)cyclopentan-1-one (4e): Followed the general procedure 3 with ioxoprofen (49 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 8:1) to give 40 mg of the title compound (yellow oil; 92% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 7.7 Hz, 2H), 3.18 (dd, *J* = 13.9, 4.3 Hz, 1H), 2.64 – 2.57 (m, 1H), 2.57 (s, 3H), 2.42 – 2.28 (m, 2H),

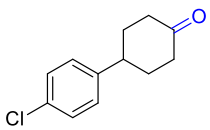
2.14 – 2.02 (m, 2H), 2.00 – 1.92 (m, 1H), 1.81 – 1.65 (m, 1H), 1.59 – 1.45 (m, 1H); **^{13}C NMR (101 MHz, CDCl$_3$)** δ 219.7, 197.9, 145.9, 135.4, 129.2, 128.6, 50.7, 38.1, 35.6, 29.2, 26.6, 20.6; **HRMS (ESI)** calcd $C_{14}H_{17}O_2$ $[M + H]^+$: 217.1223, found: 217.1227.



4,4-difluorocyclohexan-1-one (4f): Followed the general procedure 3 with 4,4-difluorocyclohexane-1-carboxylic acid (33 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 20:1) to give 25 mg of the title compound (yellow oil; 92% yield); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 2.56 (t, *J* = 7.1 Hz, 4H), 2.33 (tt, *J* = 14.0, 7.2 Hz, 4H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 207.3, 121.6 (t, *J* = 241.7 Hz), 36.6 (t, *J* = 5.5 Hz), 32.7 (t, *J* = 26.1 Hz); **^{19}F NMR (376 MHz, CDCl$_3$)** δ -100.29 (m, 2F); **HRMS (ESI)** calcd $C_6H_9F_2O$ $[M + H]^+$: 135.0616, found: 135.0612.

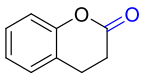


1-benzoylpiperidin-4-one (4g)： Followed the general procedure 3 with 1-benzoylpiperidine-4-carboxylic acid (47 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 8:1) to give 39 mg of the title compound (yellow oil; 95% yield); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.55 – 7.36 (m, 5H), 4.18 – 3.67 (m, 4H), 2.78 – 2.34 (m, 4H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 206.8, 171.0, 135.1, 130.3, 128.7, 127.0, 46.4, 41.2; **HRMS (ESI)** calcd $C_{12}H_{14}NO_2$ $[M + H]^+$: 204.1019, found: 204.1017.

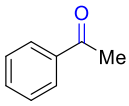


4-(4-chlorophenyl)cyclohexan-1-one (4h)： Followed the general procedure 3 with 4-(4-chlorophenyl)cyclohexane-1-carboxylic acid (48 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 31 mg of the title compound (yellow oil; 75% yield); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.29 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 3.01 (tt, *J* = 12.1, 3.3 Hz, 1H), 2.57 – 2.44 (m, 4H),

2.25 – 2.15 (m, 2H), 2.00 – 1.82 (m, 2H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 210.8, 143.2, 132.3, 128.7, 128.1, 42.2, 41.3, 33.9; **HRMS (ESI)** calcd $C_{12}H_{14}{}^{35}ClO$ $[M + H]^+$: 209.0728, found: 209.0724.

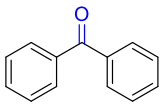


chroman-2-one (4i): Followed the general procedure 3 with chromane-2-carboxylic acid (48 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 10:1) to give 27 mg of the title compound (yellow oil; 90% yield); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.31 (d, *J* = 4.9 Hz, 1H), 7.24 (d, *J* = 7.2 Hz, 1H), 7.18 – 7.06 (m, 2H), 3.05 (t, *J* = 7.2 Hz, 2H), 2.89 – 2.78 (m, 2H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 168.7, 152.0, 128.3, 128.1, 124.4, 122.7, 117.0, 29.3, 23.8; **HRMS (ESI)** calcd $C_9H_9O_2$ $[M + H]^+$: 149.0597, found: 149.0596.



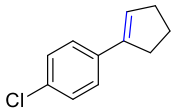
acetophenone (4j): Followed the general procedure 3 with 2-methyl-2-phenylpropanoic acid (33 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 50:1) to give 36 mg of the title compound (yellow oil; 80% yield); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.99 (d, *J* = 7.7 Hz, 2H), 7.63 – 7.56 (m, 1H), 7.49 (t, *J* = 7.1 Hz, 2H), 2.64 (s, 3H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 198.2, 137.1, 133.2, 128.6, 128.4, 26.7.

Spectral data is in agreement with the literature.[11]

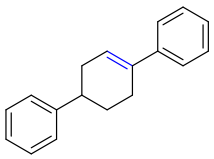


benzophenone (4k): Followed the general procedure 3 with 2,2-diphenylpropanoic acid (45 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 50:1) to give 28 mg of the title compound (yellow oil; 62% yield); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.81 (d, *J* = 8.1 Hz, 2H), 7.63 – 7.54 (m, 1H), 7.53 – 7.43 (m, 2H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 196.8, 137.6, 132.5, 130.1, 128.3.

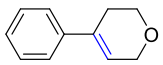
Spectral data is in agreement with the literature.[12]



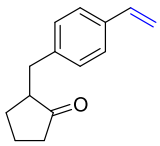
1-chloro-4-(cyclopent-1-en-1-yl)benzene (6a): Followed the general procedure 4 with 1-(4-chlorophenyl)cyclopentane-1-carboxylic acid (45 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 21 mg of the title compound (colourless oil; 59% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.40 (d, *J* = 8.6 Hz, 2H), 7.31 (d, *J* = 7.2 Hz, 2H), 6.24 – 6.18 (m, 1H), 2.76 – 2.67 (m, 2H), 2.62 – 2.51 (m, 2H), 2.10 – 2.01 (m, 2H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.4, 135.3, 132.4, 128.4, 126.9, 126.8, 33.4, 33.2, 23.4; **HRMS (ESI)** calcd $C_{11}H_{12}{}^{35}Cl$ $[M + H]^+$: 179.0622, found: 179.0621.



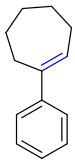
1',2',3',6'-tetrahydro-1,1':4',1''-terphenyl (6b): Followed the general procedure 4 with 1,4-diphenylcyclohexane-1-carboxylic acid (56 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 29 mg of the title compound (white solid; 62% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.52 (d, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 4H), 7.39 – 7.31 (m, 4H), 6.33 – 6.28 (m, 1H), 3.04 – 2.92 (m, 1H), 2.73 – 2.58 (m, 3H), 2.50 – 2.38 (m, 1H), 2.27 – 2.17 (m, 1H), 2.08 – 1.95 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.0, 142.2, 136.6, 128.5, 128.4, 127.0, 126.8, 126.2, 125.1, 124.3, 39.8, 34.2, 30.2, 28.1; **HRMS (ESI)** calcd $C_{18}H_{19}$ $[M + H]^+$: 235.1481, found: 235.1485.



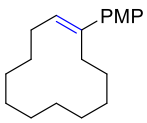
4-phenyl-3,6-dihydro-2H-pyran (6c): Followed the general procedure 4 with 4-phenyltetrahydro-2H-pyran-4-carboxylic acid (41 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 25 mg of the title compound (colourless oil; 77% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.38 (d, *J* = 7.9 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.21 (m, 1H), 6.14 – 6.08 (m, 1H), 4.31 (d, *J* = 2.4 Hz, 2H), 3.92 (t, *J* = 5.4 Hz, 2H), 2.55 – 2.47 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 140.3, 134.2, 128.5, 127.3, 124.7, 122.5, 65.9, 64.5, 27.2; **HRMS (ESI)** calcd $C_{11}H_{13}O$ $[M + H]^+$: 161.0961, found: 161.0957.



2-(4-vinylbenzyl)cyclopentan-1-one (6d): Followed the general procedure 4 with ioxoprofen (49 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 22 mg of the title compound (colourless oil; 56% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 (d, *J* = 7.1 Hz, 2H), 7.16 (d, *J* = 7.2 Hz, 2H), 6.72 (dd, *J* = 17.5, 10.9 Hz, 1H), 5.75 (d, *J* = 17.6 Hz, 1H), 5.24 (d, *J* = 10.8 Hz, 1H), 3.23 – 3.09 (m, 1H), 2.64 – 2.52 (m, 1H), 2.44 – 2.32 (m, 2H), 2.21 – 2.06 (m, 2H), 2.06 – 1.92 (m, 1H), 1.84 – 1.69 (m, 1H), 1.61 – 1.51 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 220.3, 139.7, 136.6, 135.6, 129.1, 126.3, 113.3, 51.0, 38.3, 35.3, 29.1, 20.6; **HRMS (ESI)** calcd $C_{14}H_{17}O$ $[M + H]^+$: 201.1274, found: 201.1274.

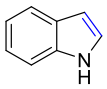


1-phenylcyclohept-1-ene (6e): Followed the general procedure 4 with 1-phenylcycloheptane-1-carboxylic acid (44 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 20 mg of the title compound (colourless oil; 57% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 (d, *J* = 7.1 Hz, 2H), 7.16 (d, *J* = 7.2 Hz, 2H), 6.72 (dd, *J* = 17.5, 10.9 Hz, 1H), 5.75 (d, *J* = 17.6 Hz, 1H), 5.24 (d, *J* = 10.8 Hz, 1H), 3.23 – 3.09 (m, 1H), 2.64 – 2.52 (m, 1H), 2.44 – 2.32 (m, 2H), 2.21 – 2.06 (m, 2H), 2.06 – 1.92 (m, 1H), 1.84 – 1.69 (m, 1H), 1.61 – 1.51 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 220.3, 139.7, 136.6, 135.6, 129.1, 126.3, 113.3, 51.0, 38.3, 35.3, 29.1, 20.6; **HRMS (ESI)** calcd $C_{13}H_{18}$ $[M + H]^+$: 173.1325, found: 173.1329.

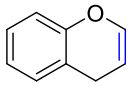


1-(4-methoxyphenyl)cyclododec-1-ene (6f): Followed the general procedure 4 with 1-(4-methoxyphenyl)cyclododecane-1-carboxylic acid (64 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 20 mg of the title compound (white solid; 47% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.28 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 5.54 (t, *J* = 7.8 Hz, 1H), 3.81 (s, 3H), 2.57 (t, *J* =

6.6 Hz, 2H), 2.31 – 2.21 (m, 2H), 1.57 – 1.52 (m, 2H), 1.49 – 1.35 (m, 12H), 1.23 – 1.15 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 158.3, 139.7, 135.9, 129.2, 127.7, 113.5, 55.3, 27.5, 25.6, 25.5, 25.4, 24.9, 24.9, 24.4, 24.2, 22.5; **HRMS (ESI)** calcd $C_{19}H_{30}O$ $[M + H]^+$: 273.2213, found: 273.2215.



1H-indole (6g): Followed the general procedure 4 with indoline-2-carboxylic acid (32 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether/EtOAc = 8:1) to give 17 mg of the title compound (yellow oil; 72% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.19 (s, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.31 – 7.24 (m, 2H), 7.21 – 7.14 (m, 1H), 6.64 – 6.57 (m, 1H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 135.8, 127.9, 124.2, 122.0, 120.8, 119.8, 111.1, 102.7;
Spectral data is in agreement with the literature.[13]



4H-chromene (6h): Followed the general procedure 4 with chromane-2-carboxylic acid (36 mg, 0.2 mmol) and purified using flash chromatography (petroleum ether) to give 20 mg of the title compound (colourless oil; 75% yield); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.15 (t, *J* = 7.3 Hz, 1H), 7.09 – 6.97 (m, 2H), 6.87 (d, *J* = 7.0 Hz, 1H), 6.57 – 6.45 (m, 1H), 5.02 – 4.92 (m, 1H), 3.44 (s, 2H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 151.5, 140.7, 129.4, 127.5, 123.2, 120.0, 116.5, 100.4, 23.1; **HRMS (ESI)** calcd C_9H_9O $[M + H]^+$: 133.0648, found: 133.0657.

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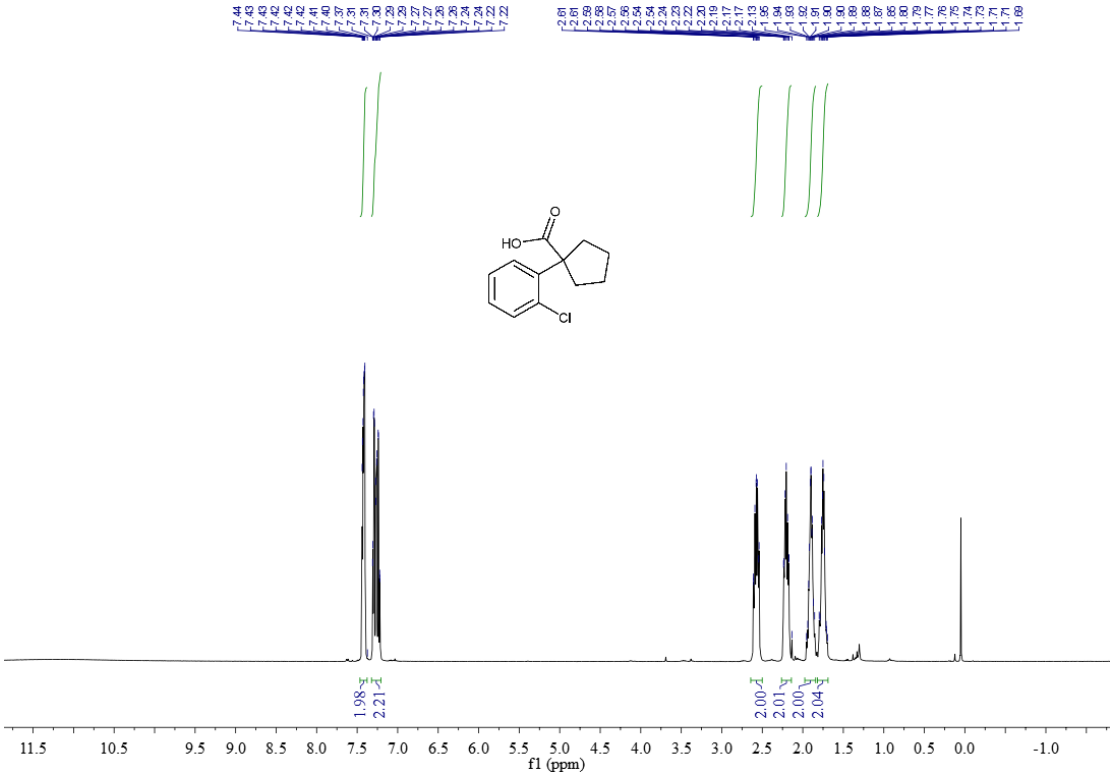
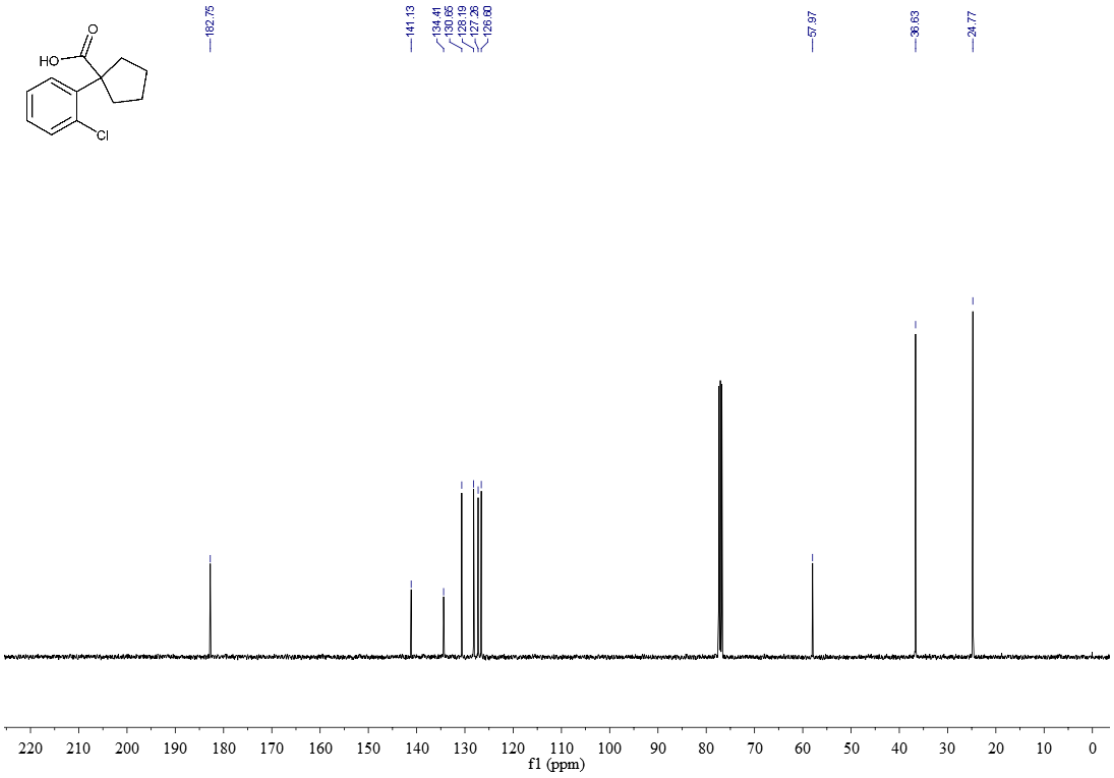
8. NMR Spectra for the substrates and products

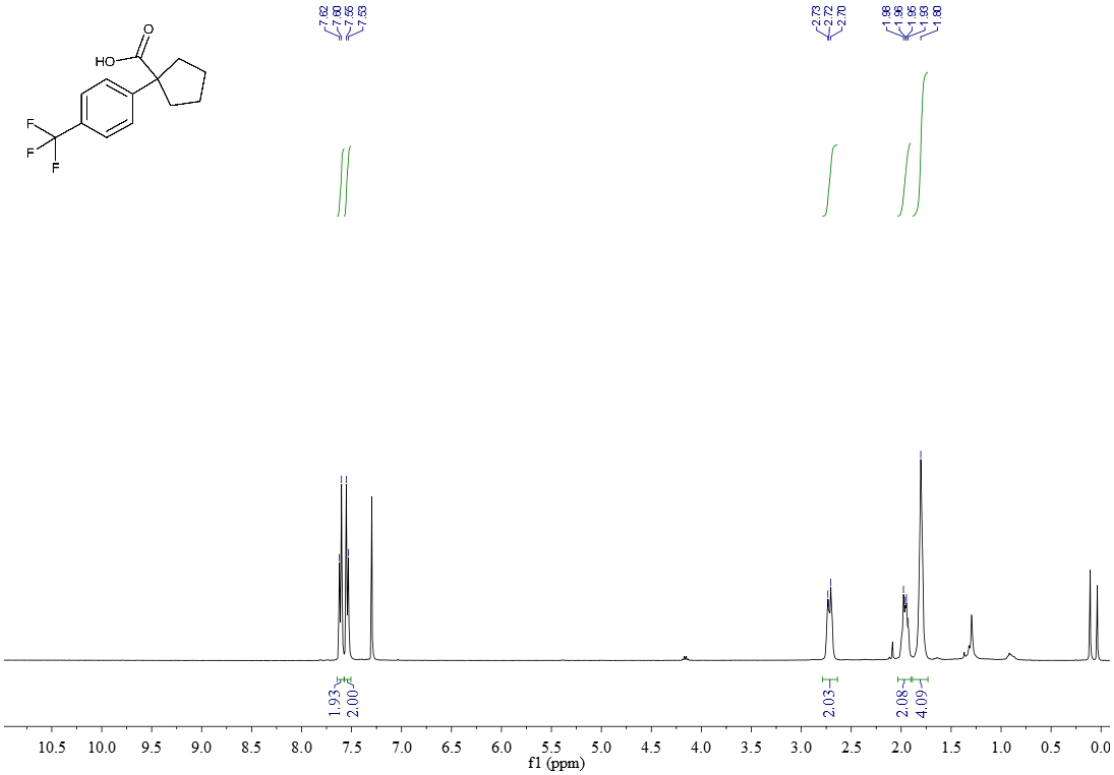
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1c**



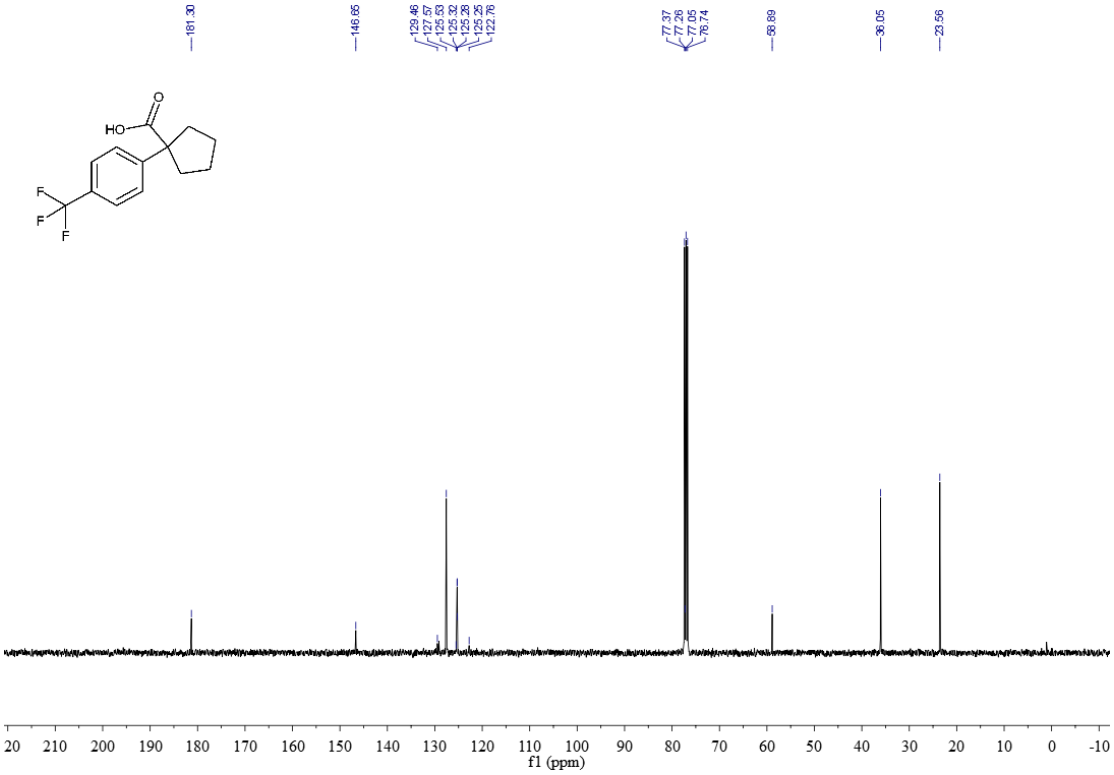
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1c**



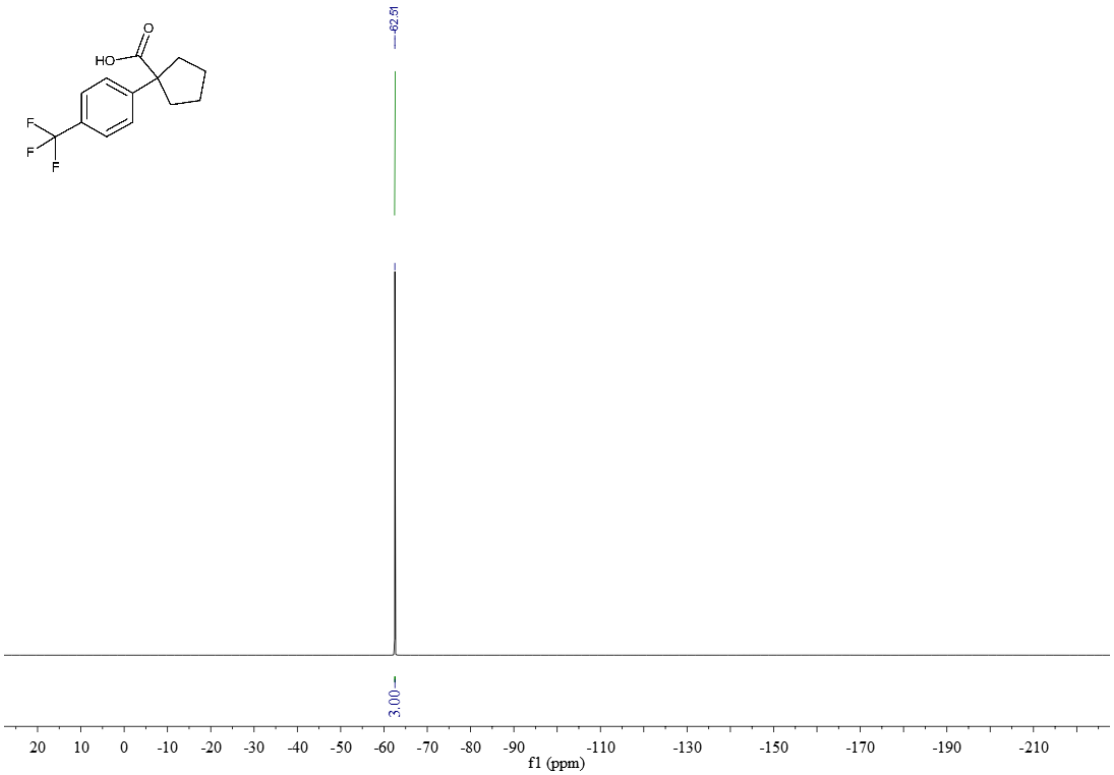
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1e**



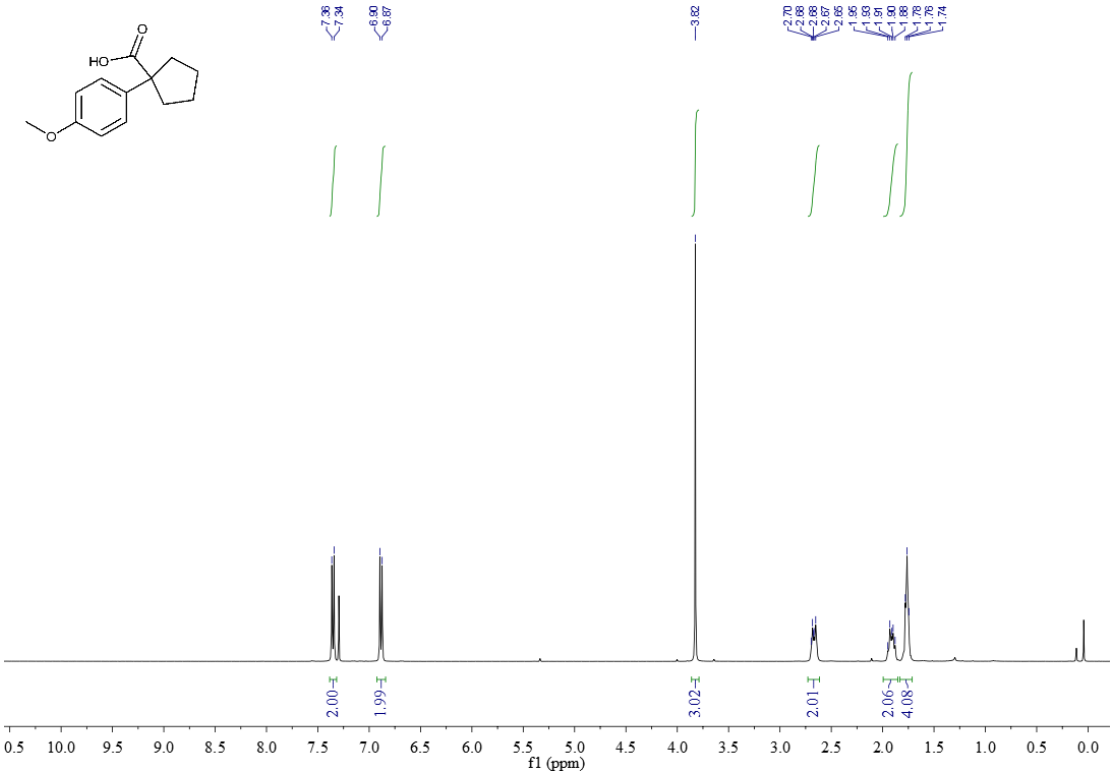
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1e**



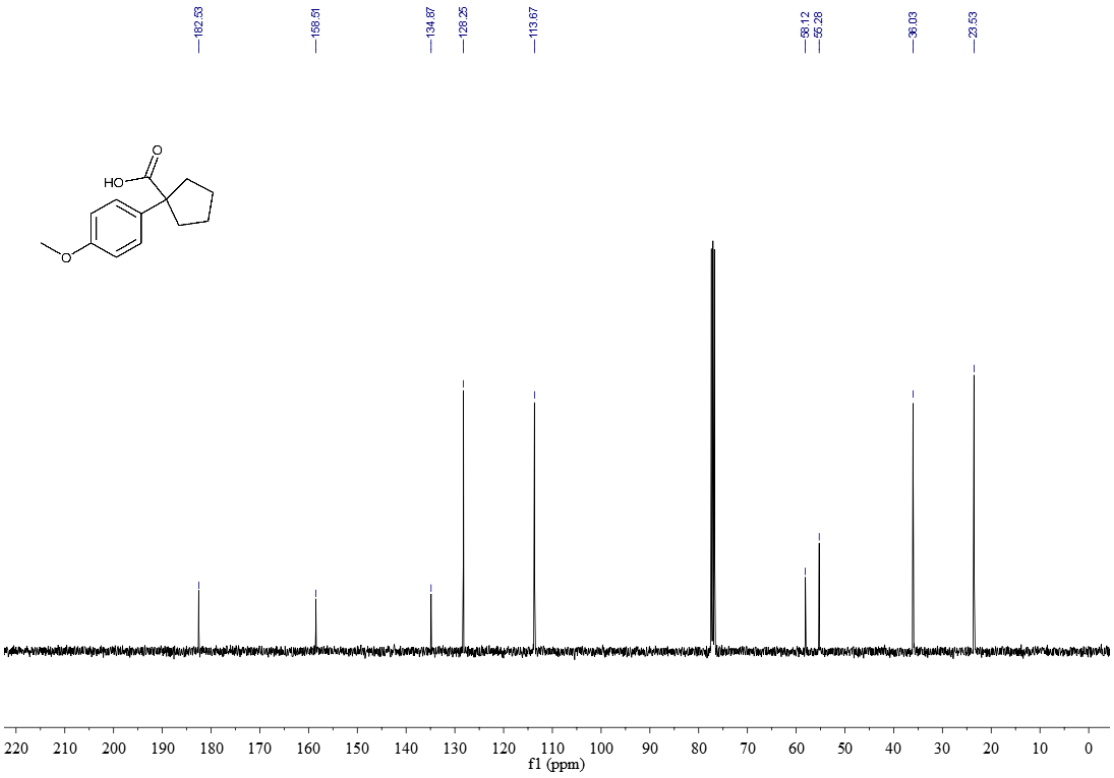
^{19}F NMR spectrum (376 MHz, $CDCl_3$, 23 ℃) of **1e**



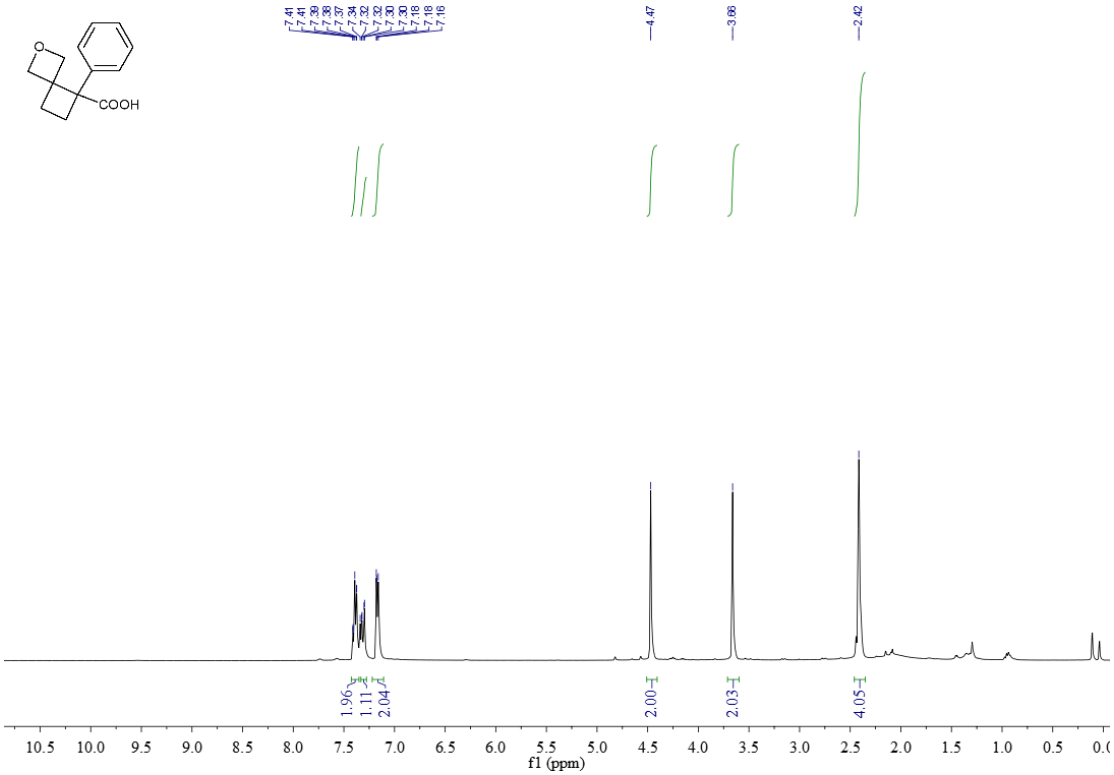
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1f**



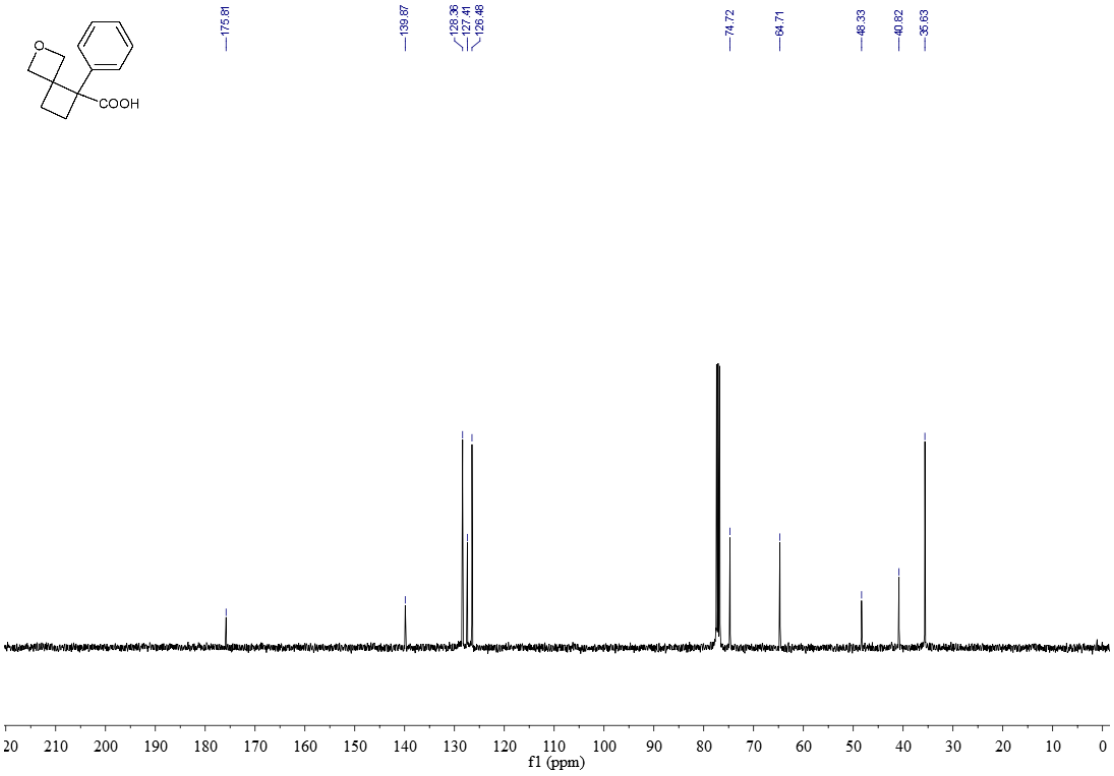
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1f**



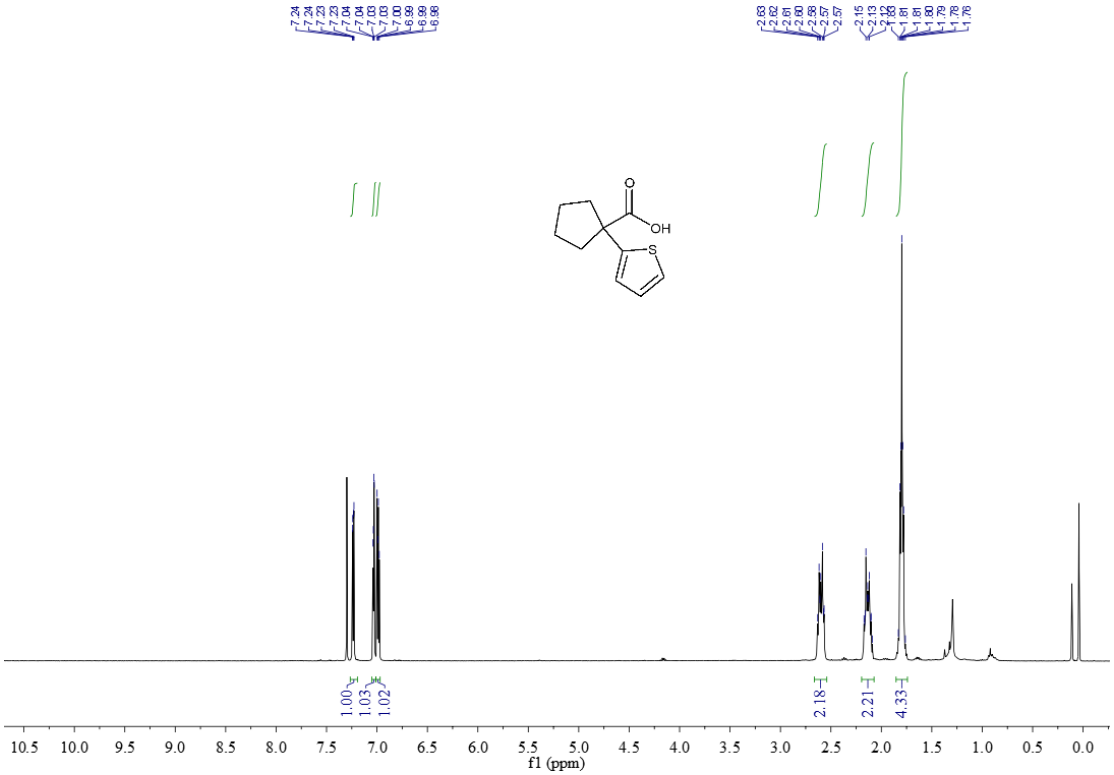
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1i**



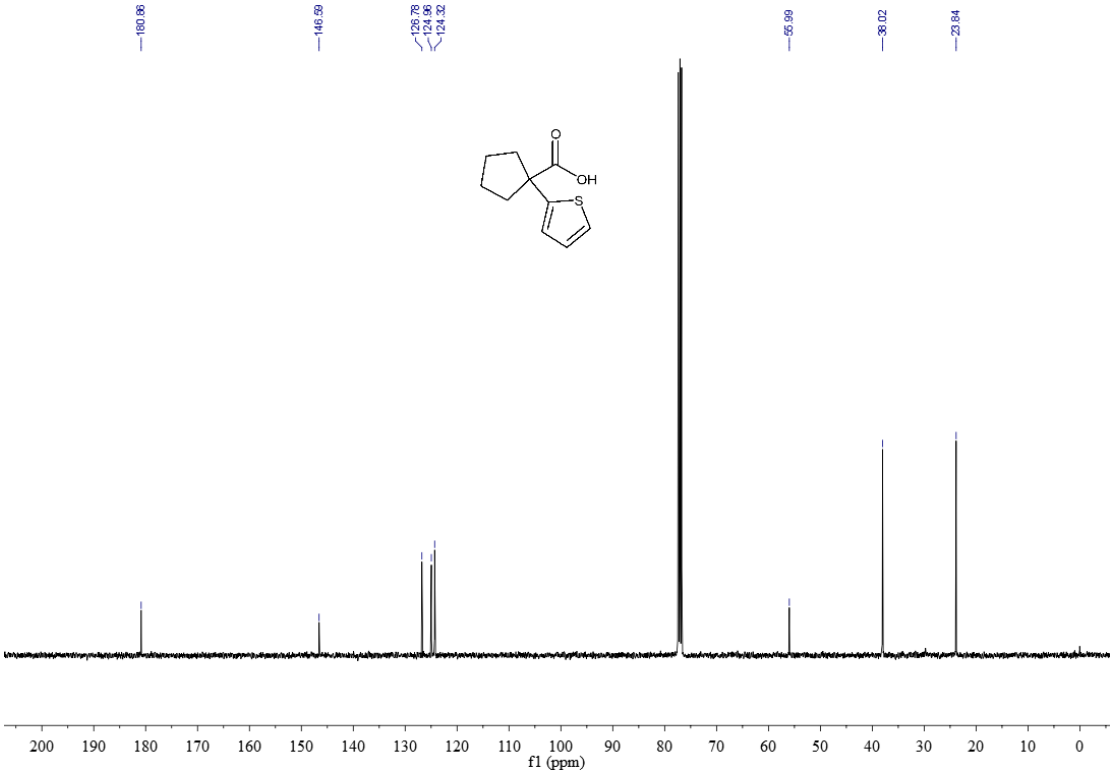
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1i**



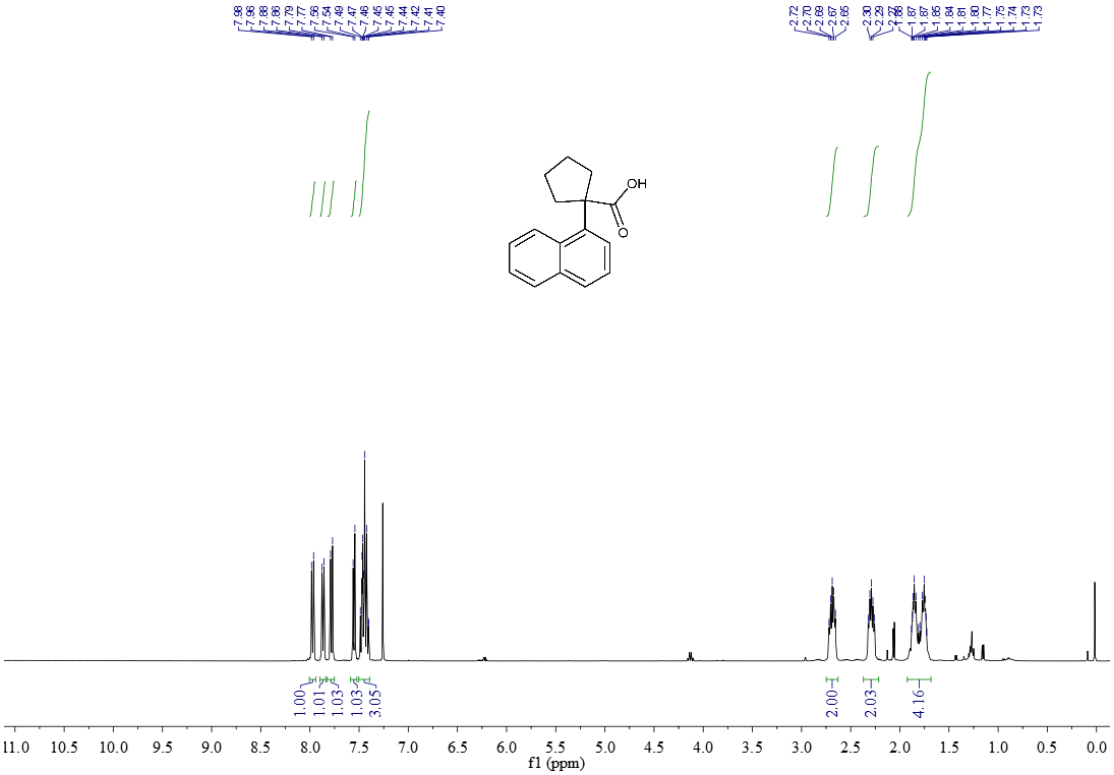
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1j**



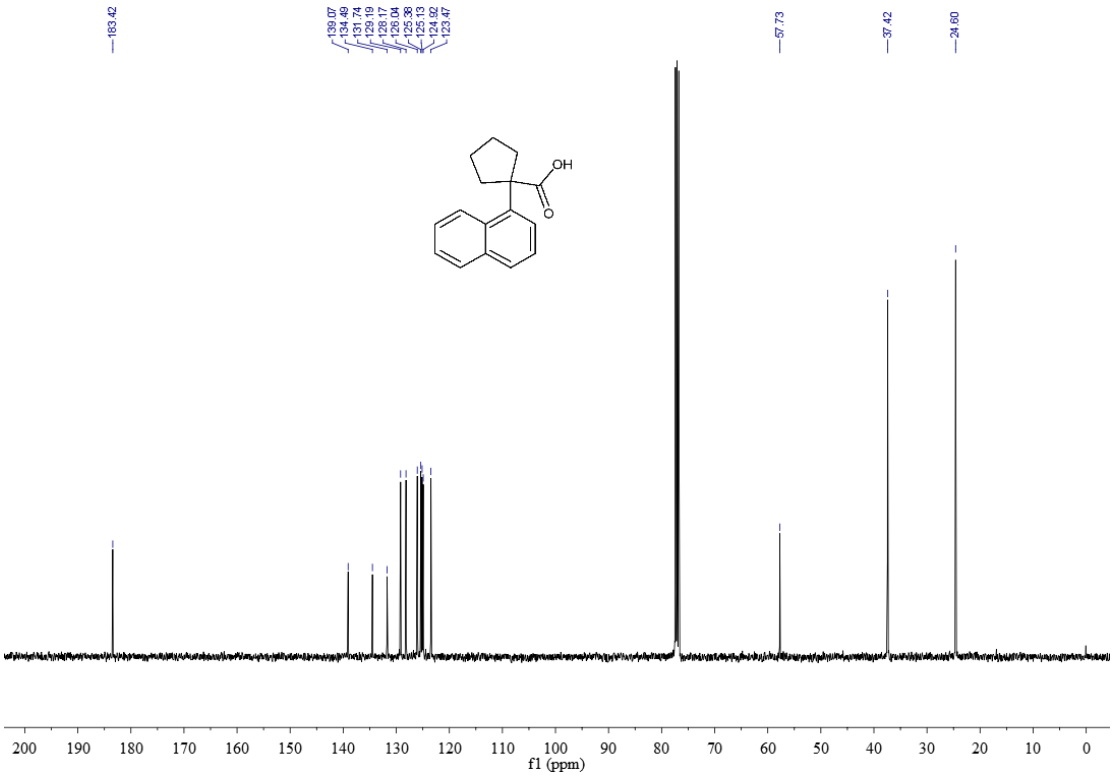
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1j**



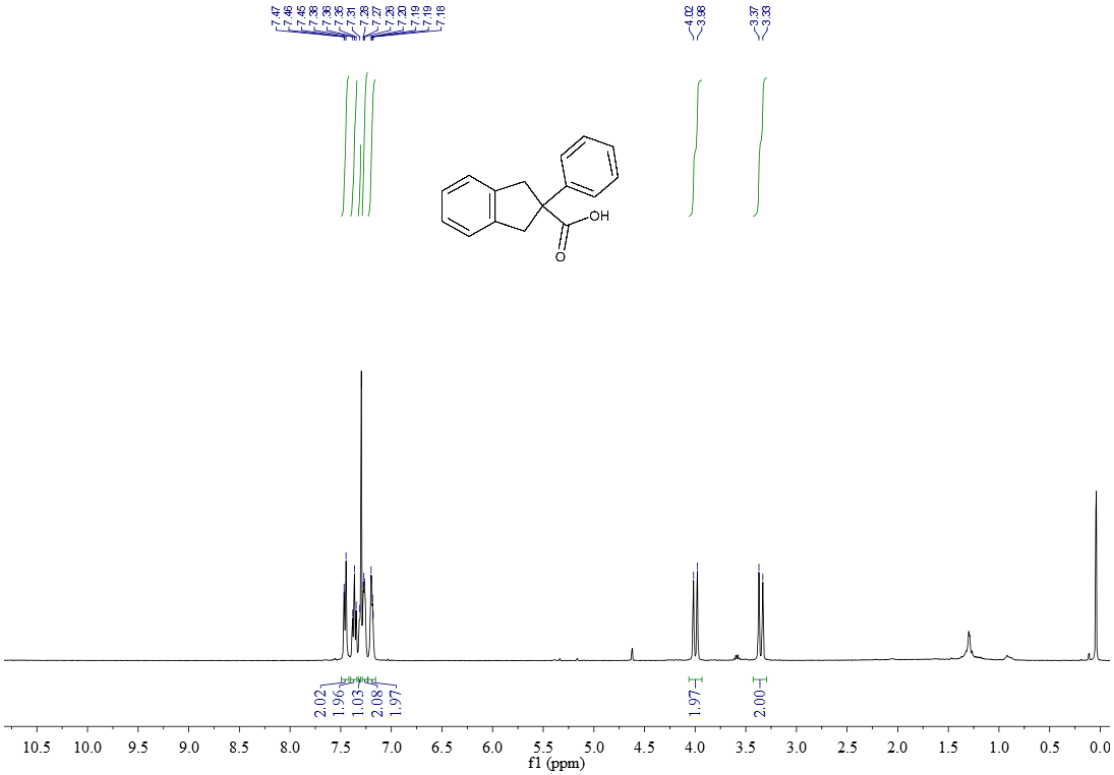
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1k**



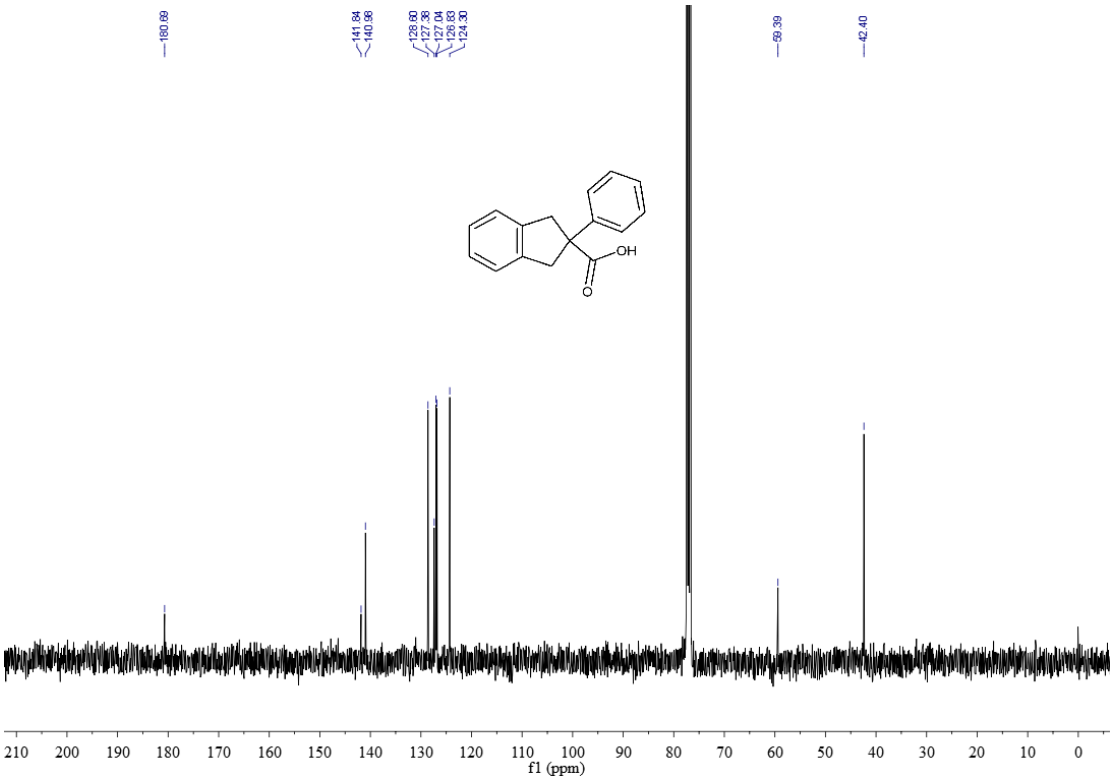
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1k**



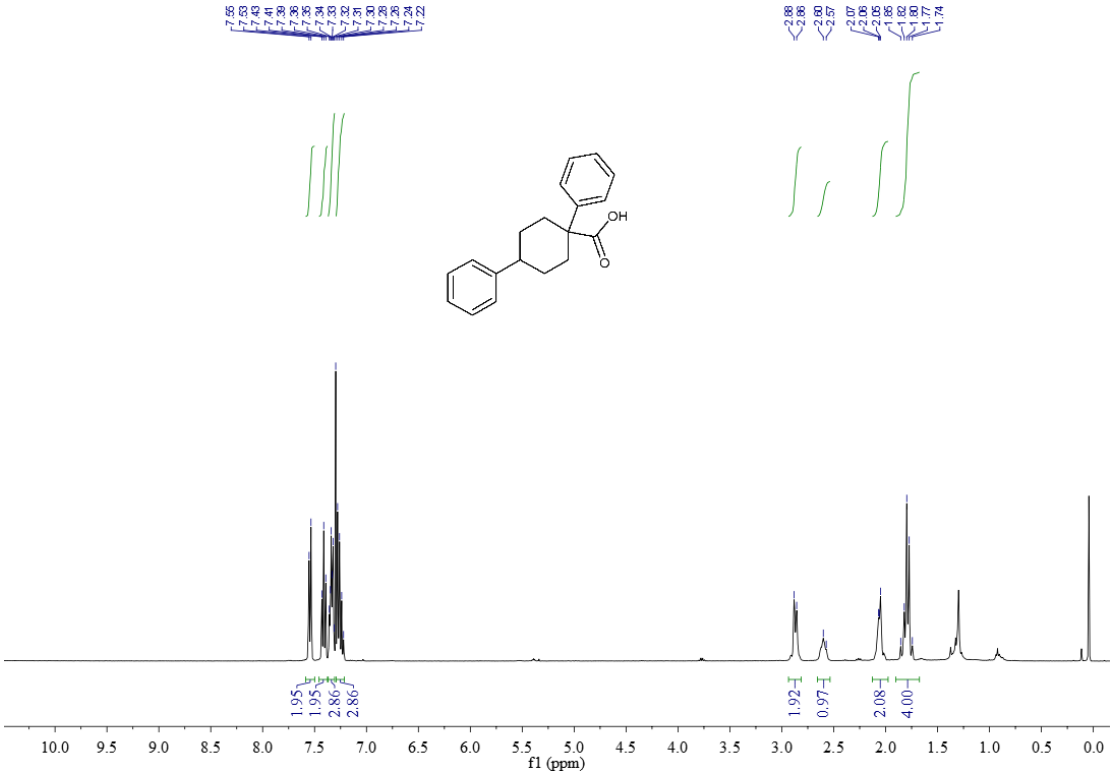
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1l**



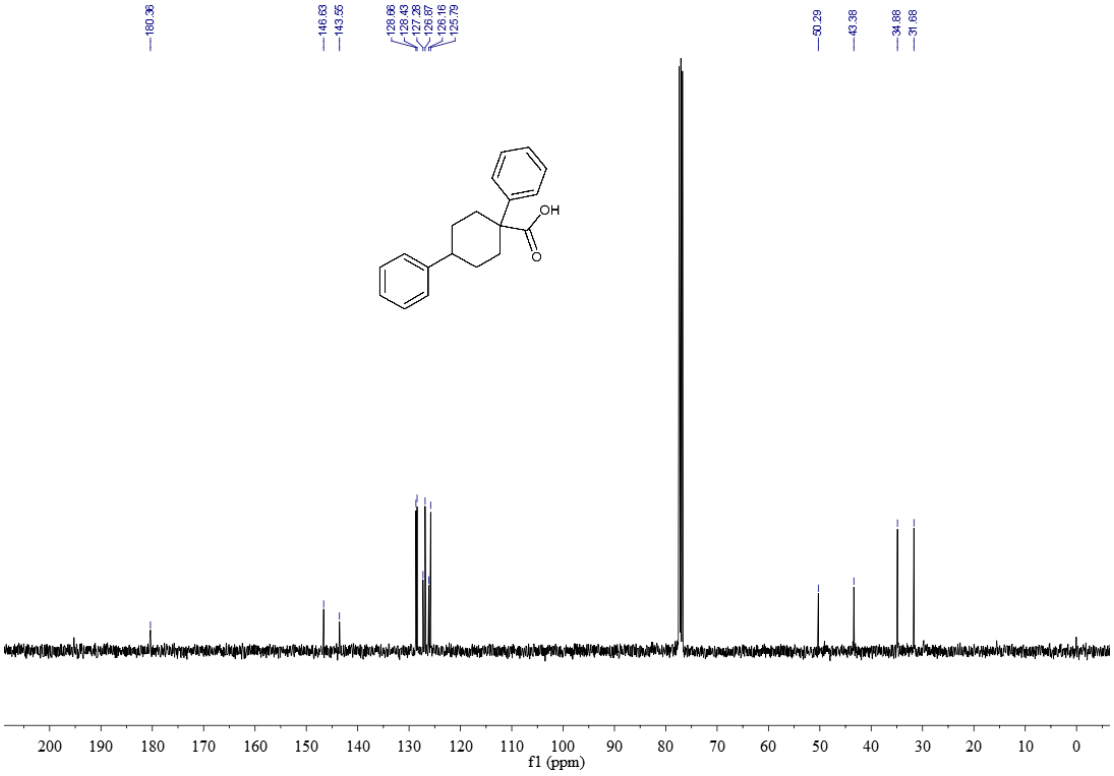
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1l**



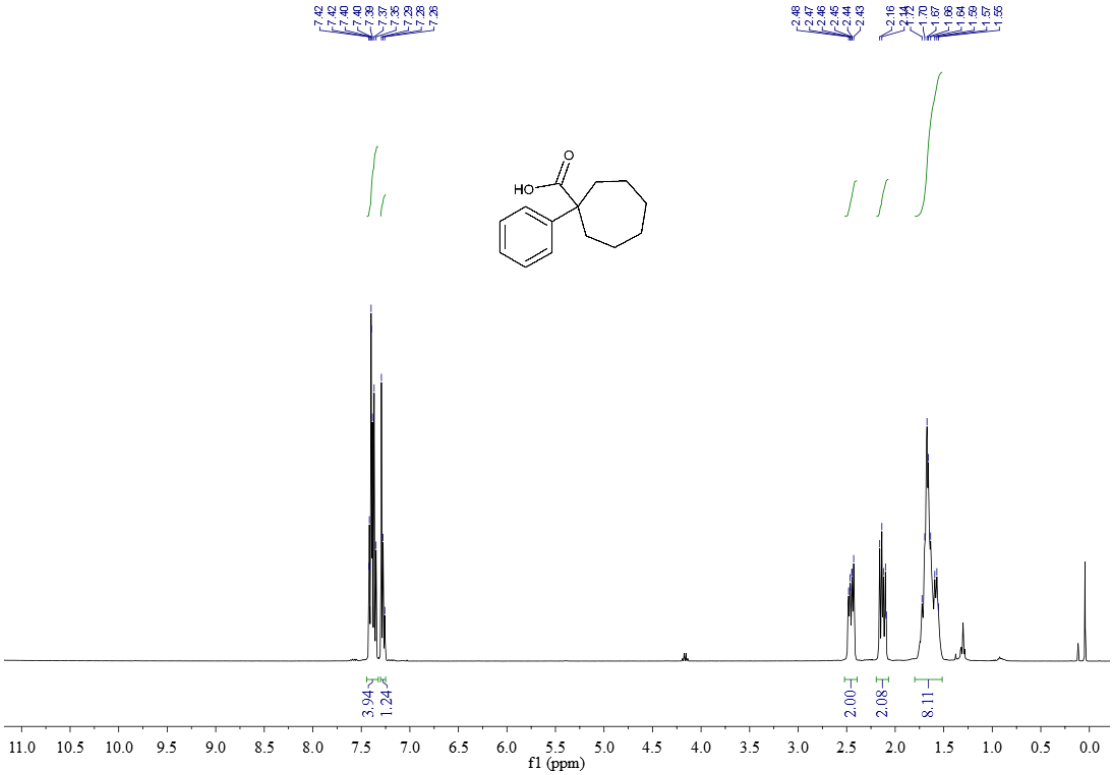
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1n**



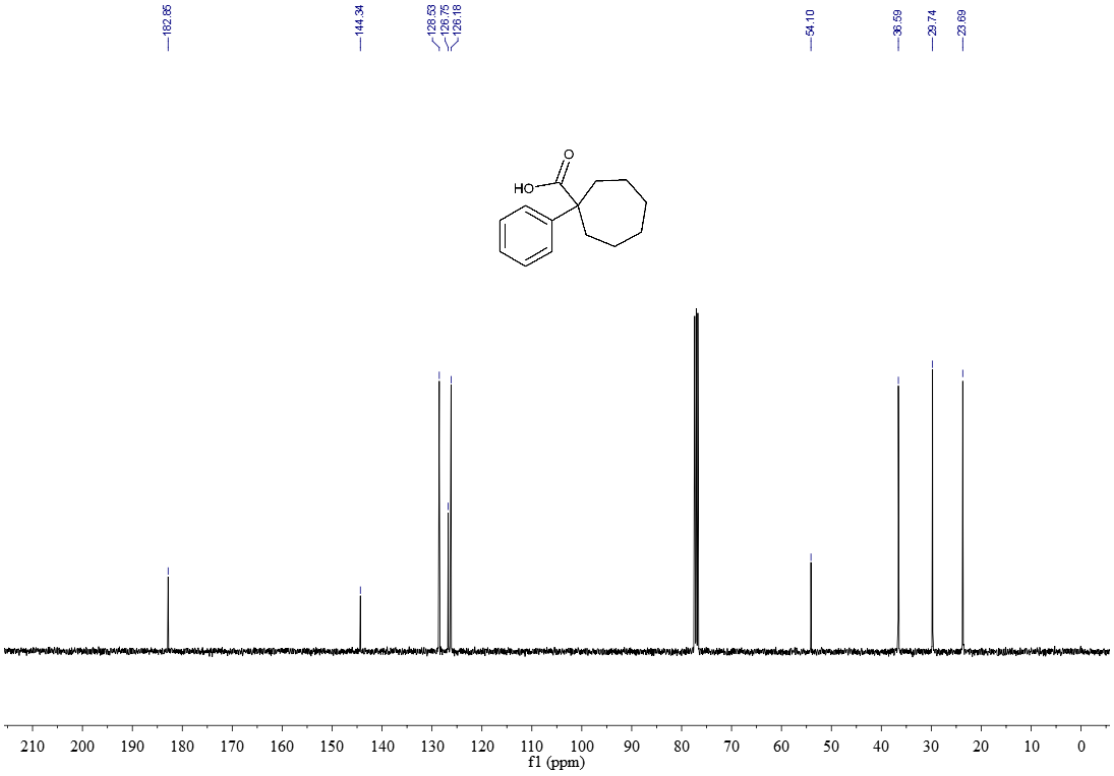
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1n**



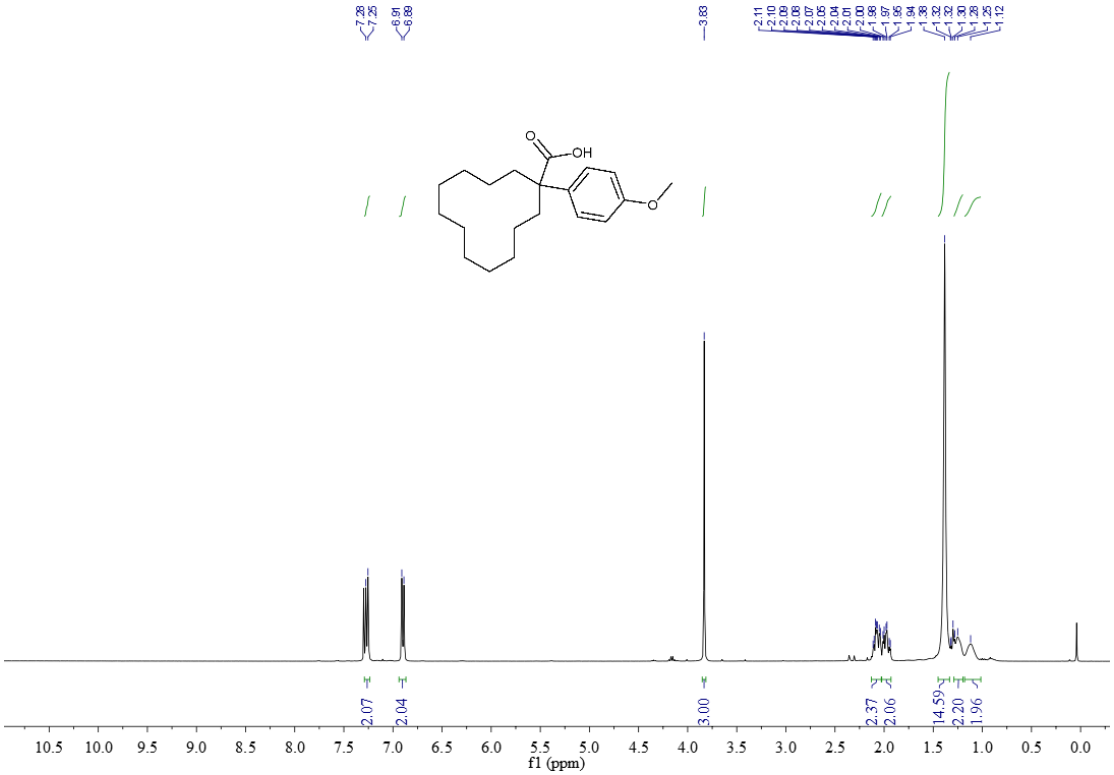
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1o**



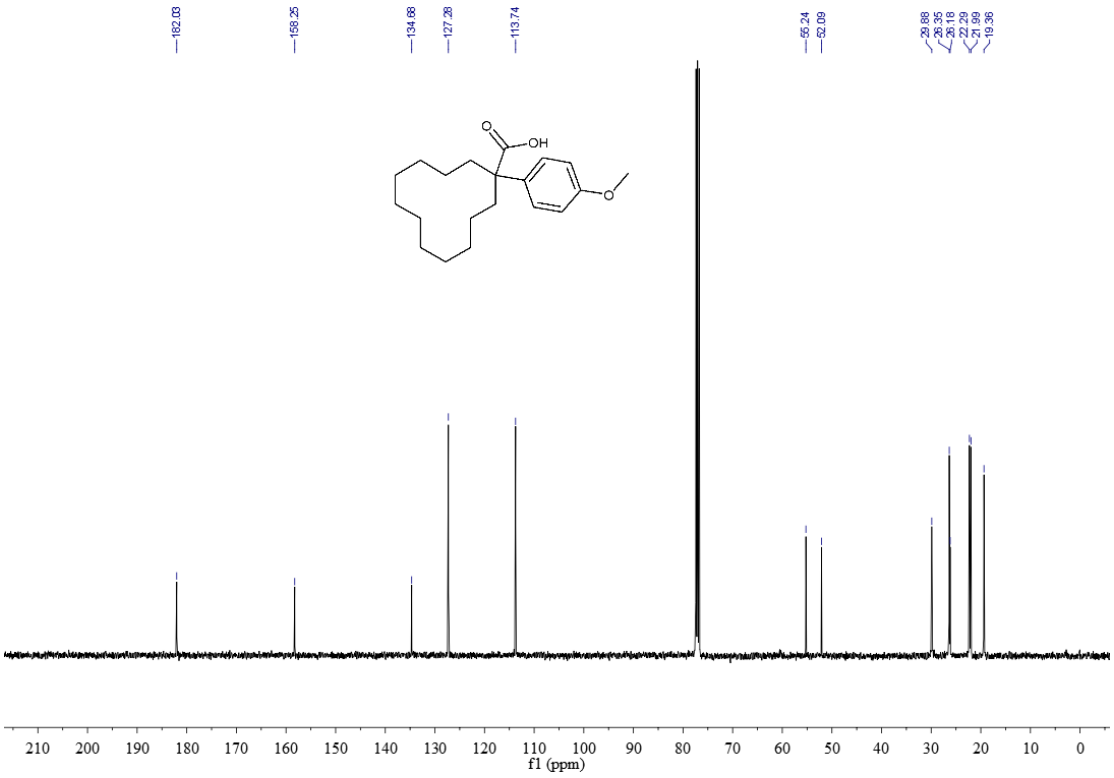
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1o**



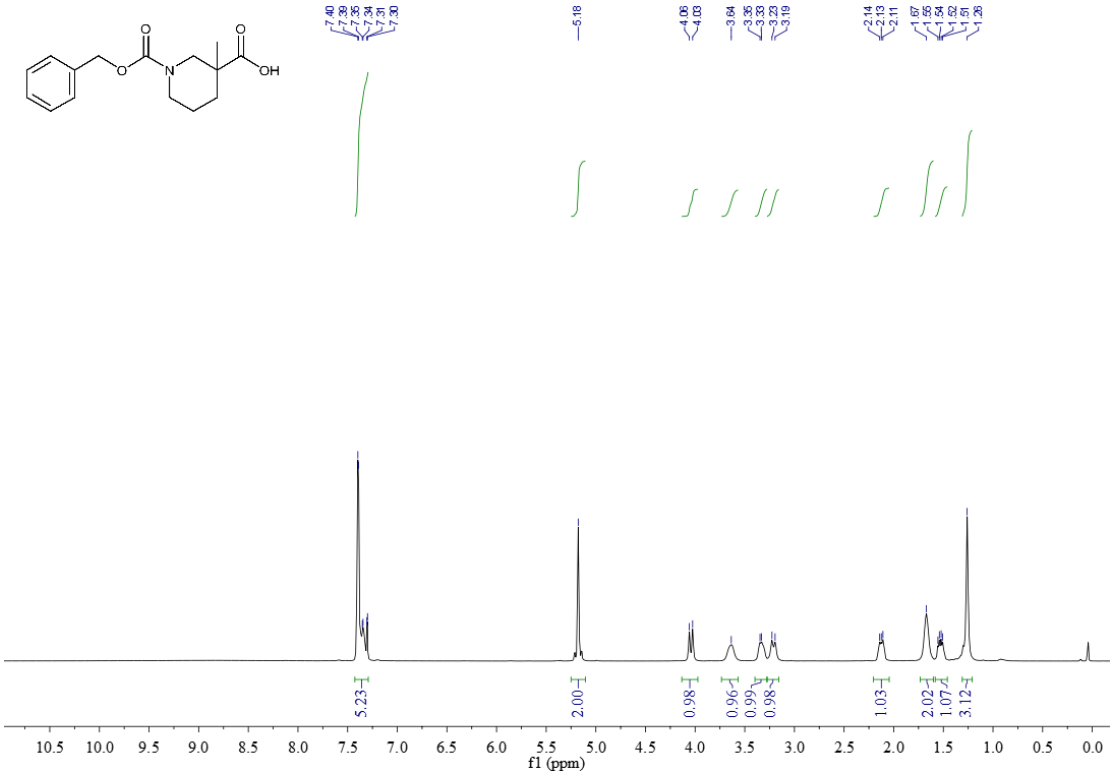
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1p**



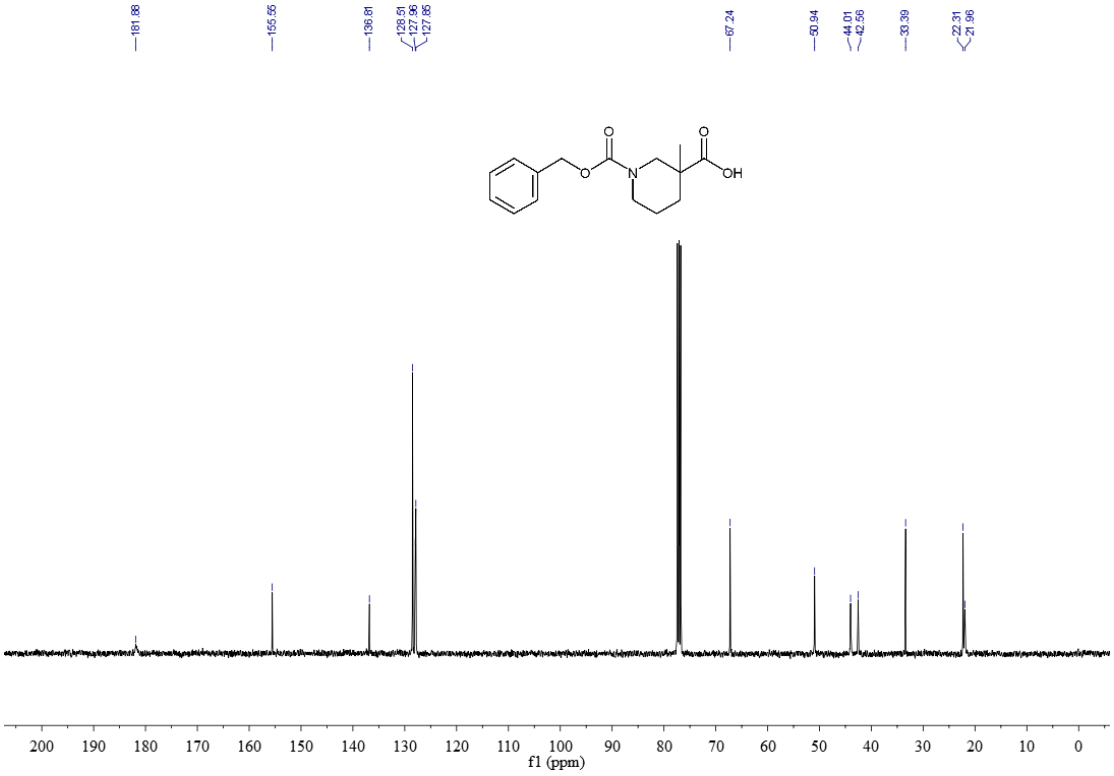
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1p**



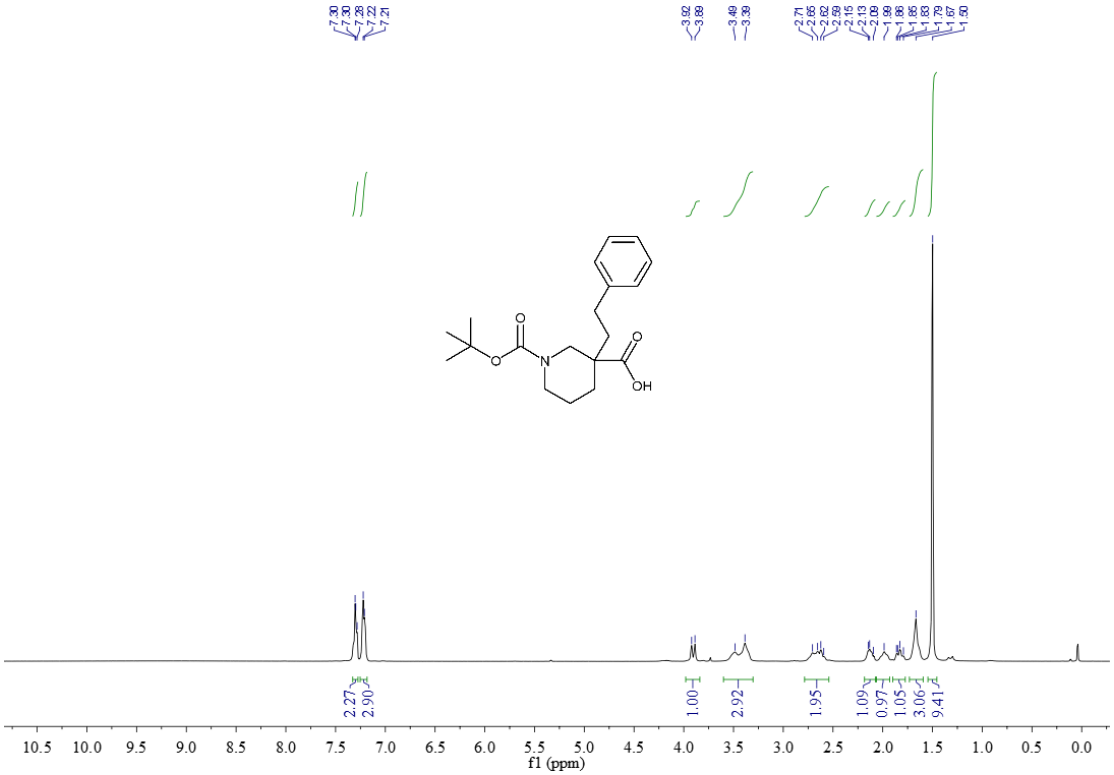
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1z**



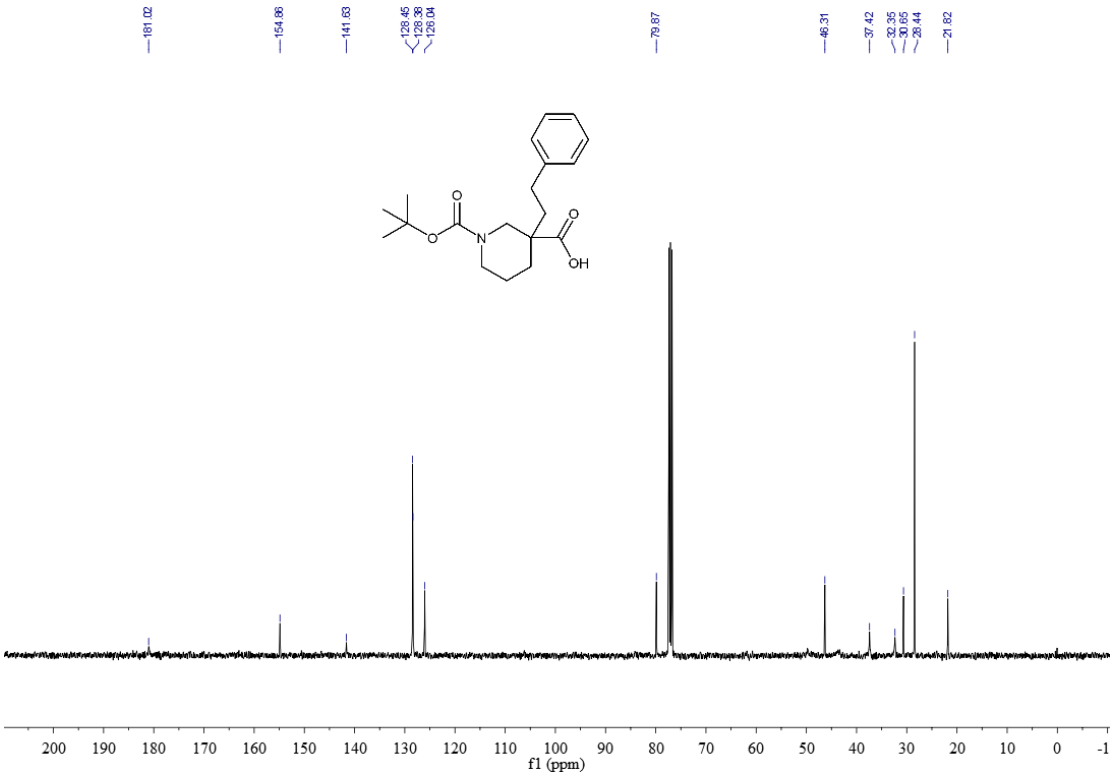
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1z**



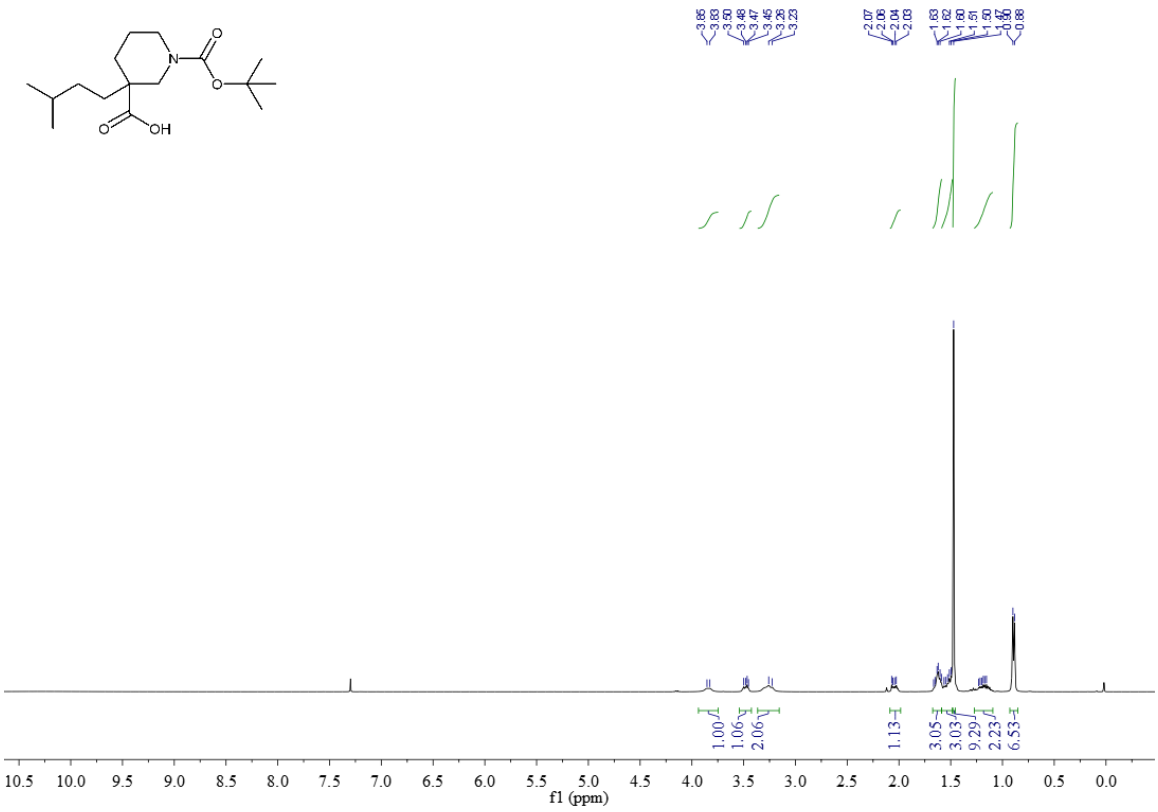
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1bb**



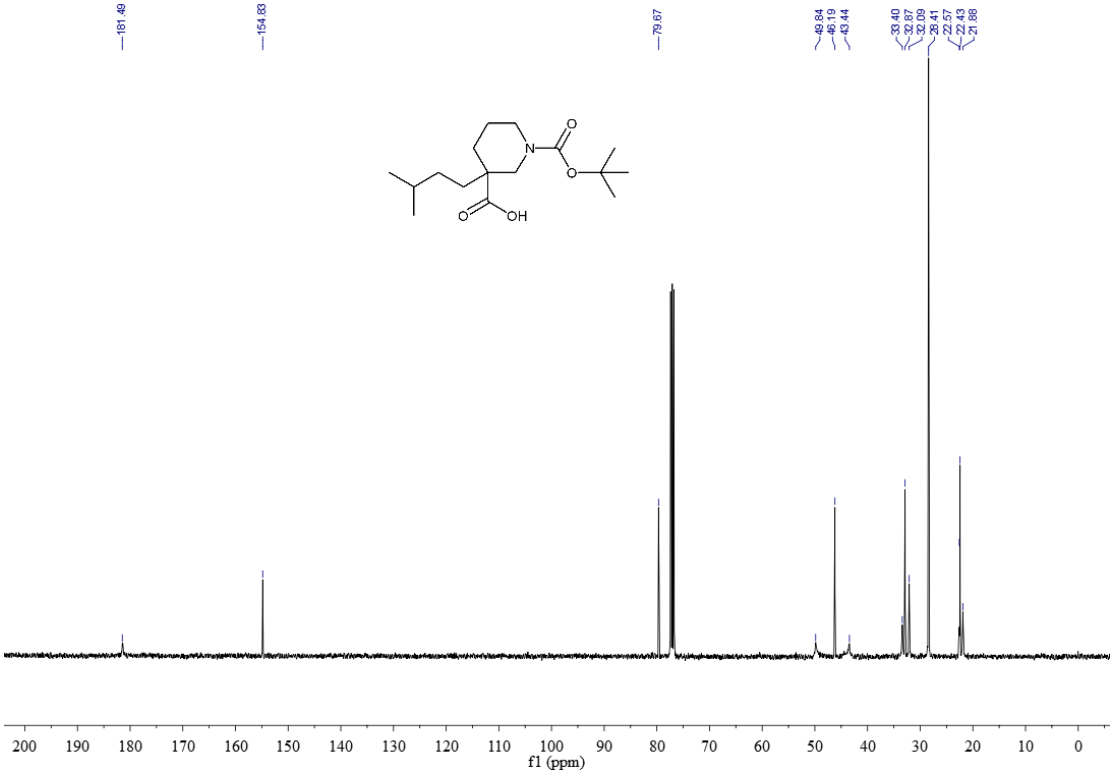
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1bb**



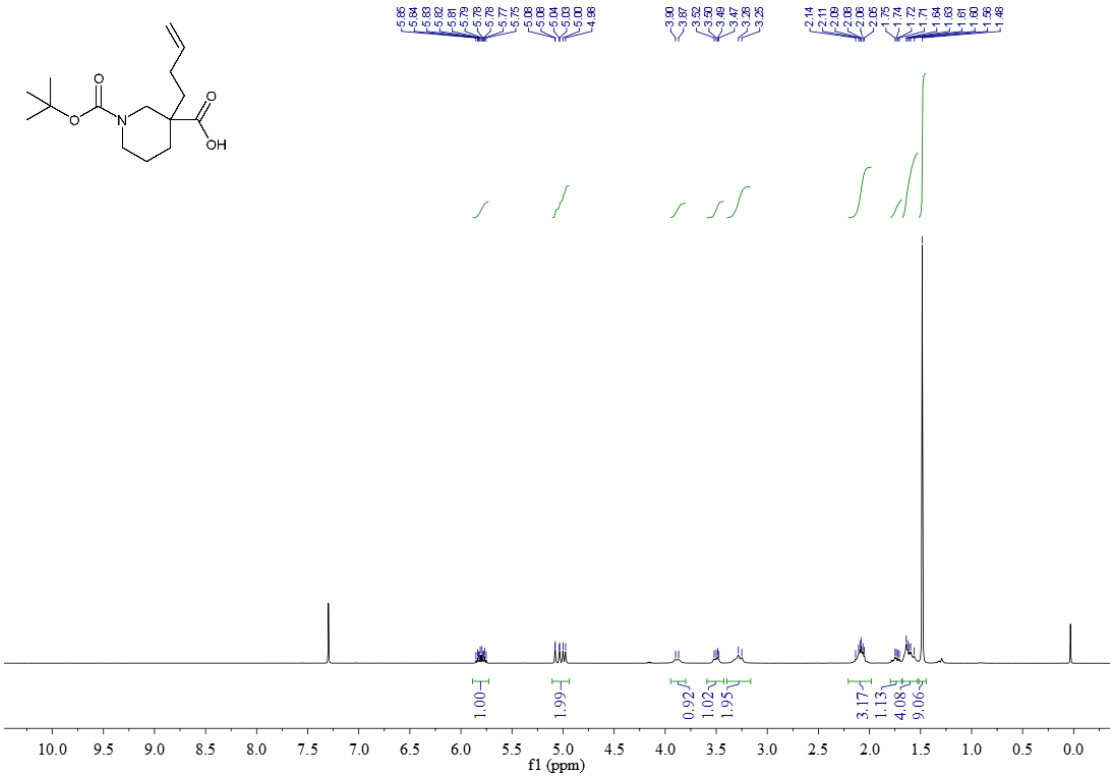
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1cc**



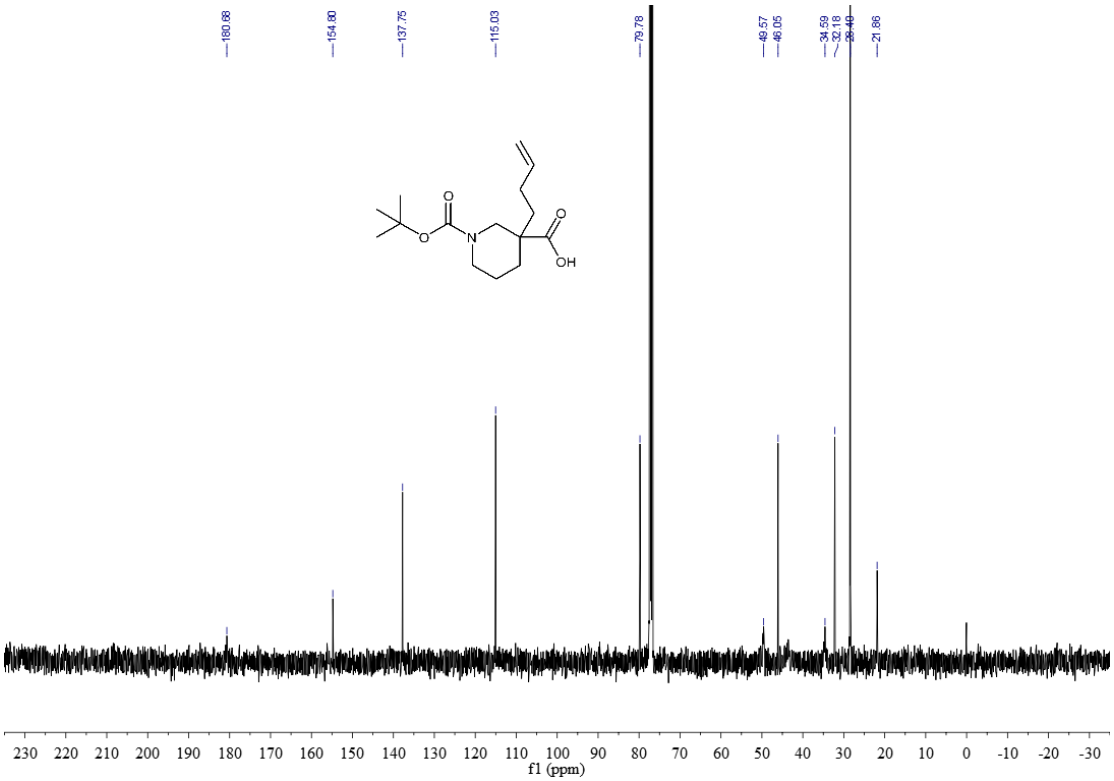
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1cc**



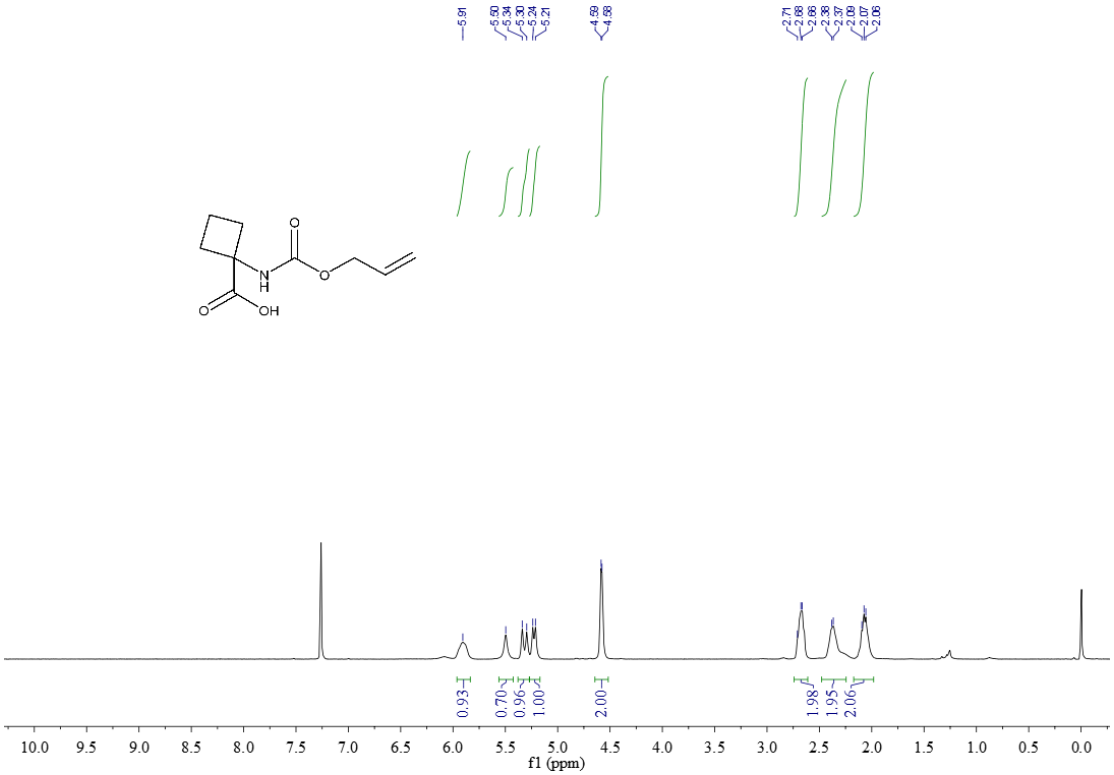
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1dd**



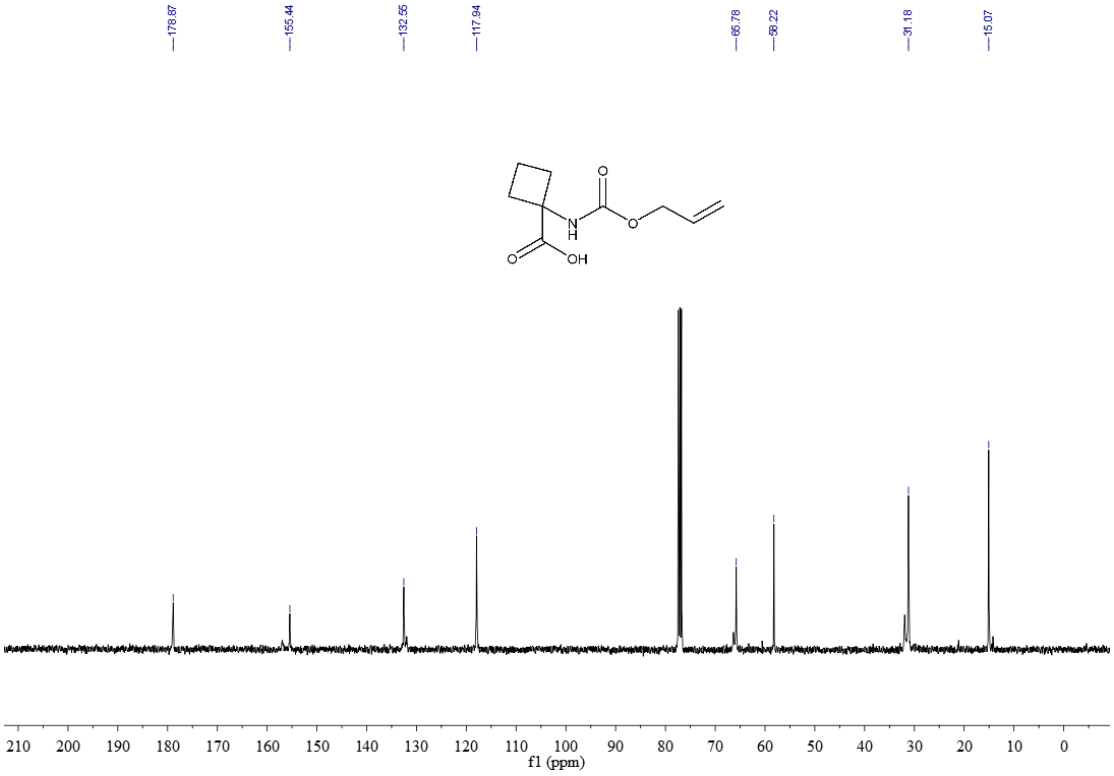
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1dd**



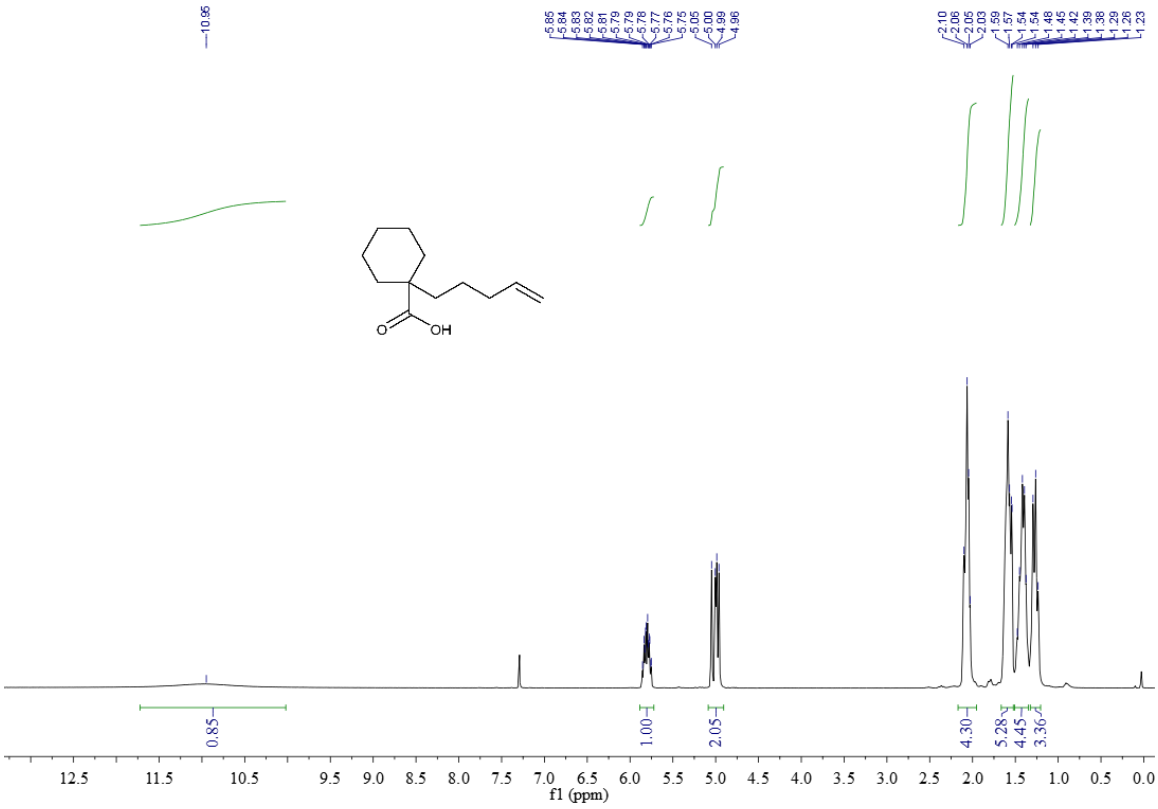
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **1ee**



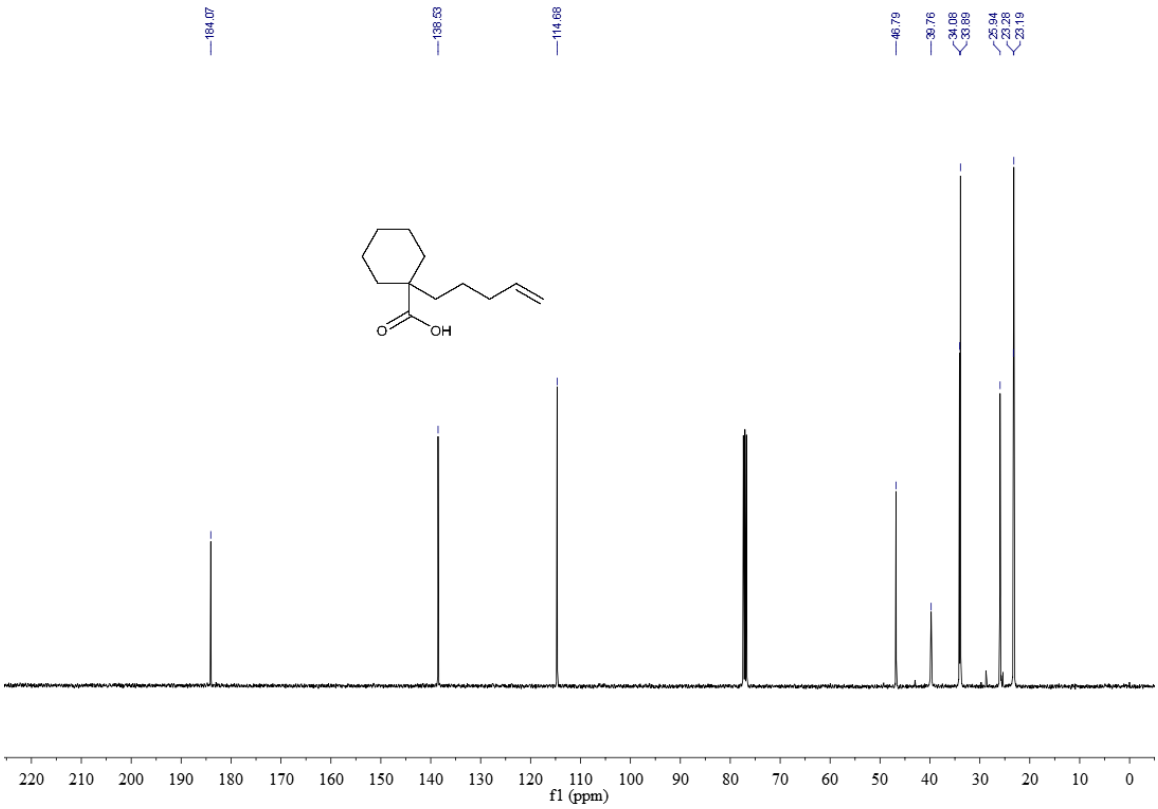
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **1ee**



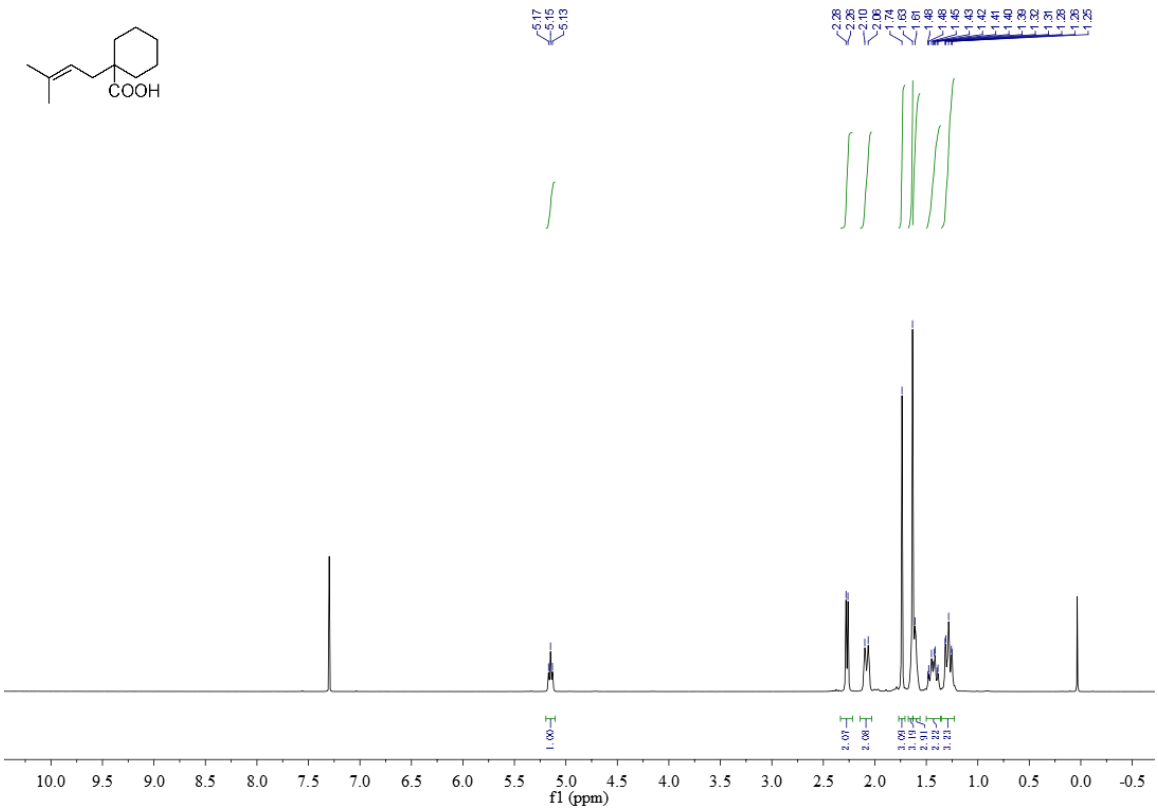
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **7**



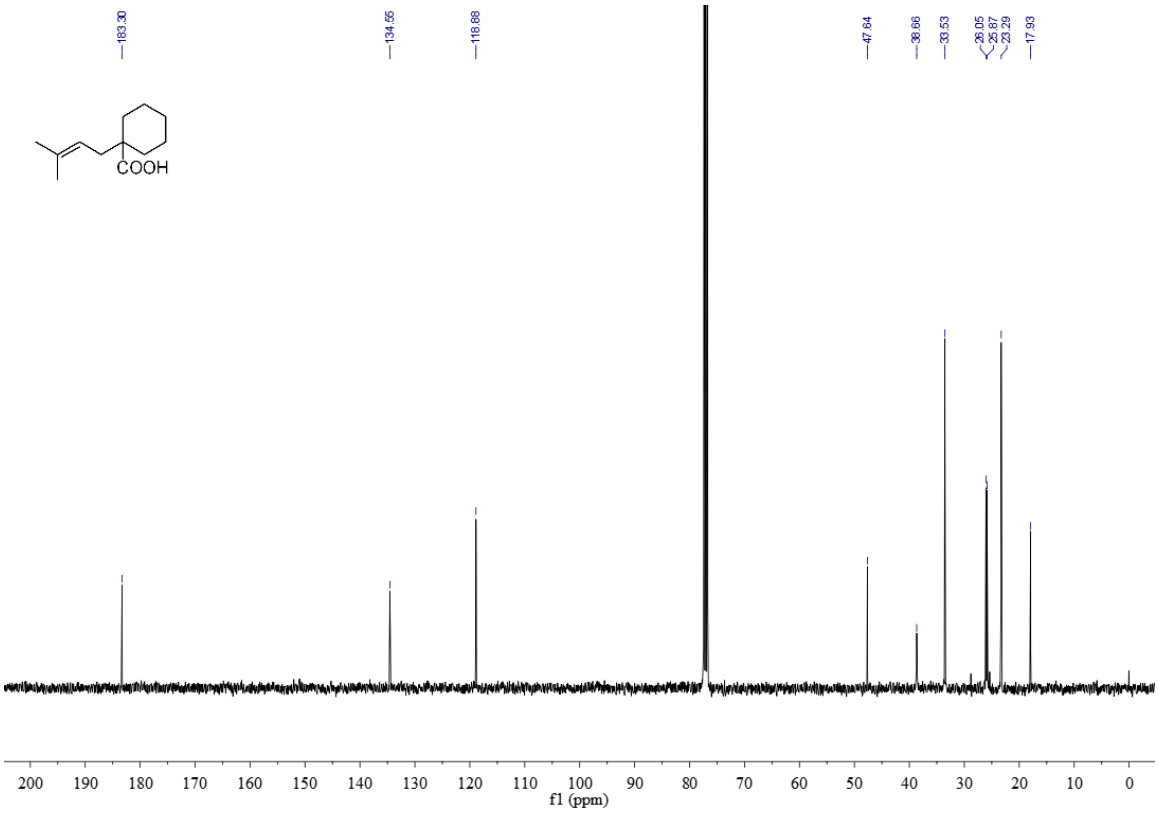
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **7**



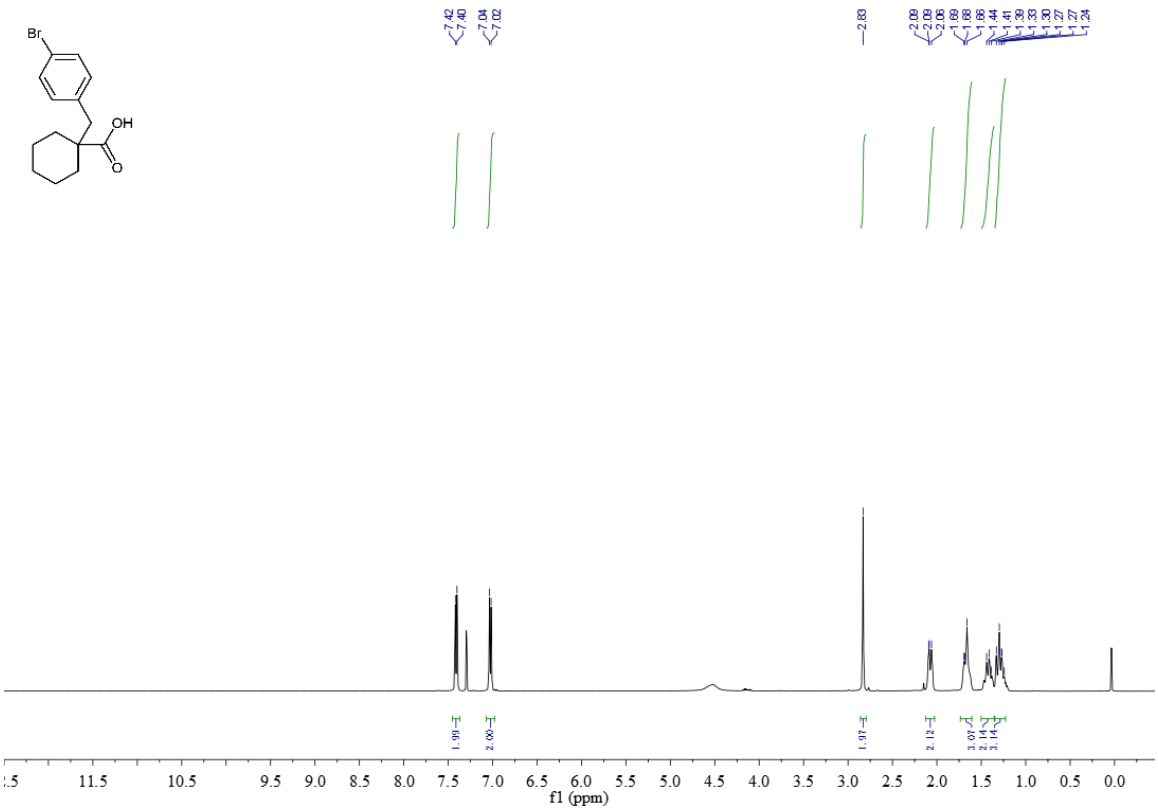
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **9**



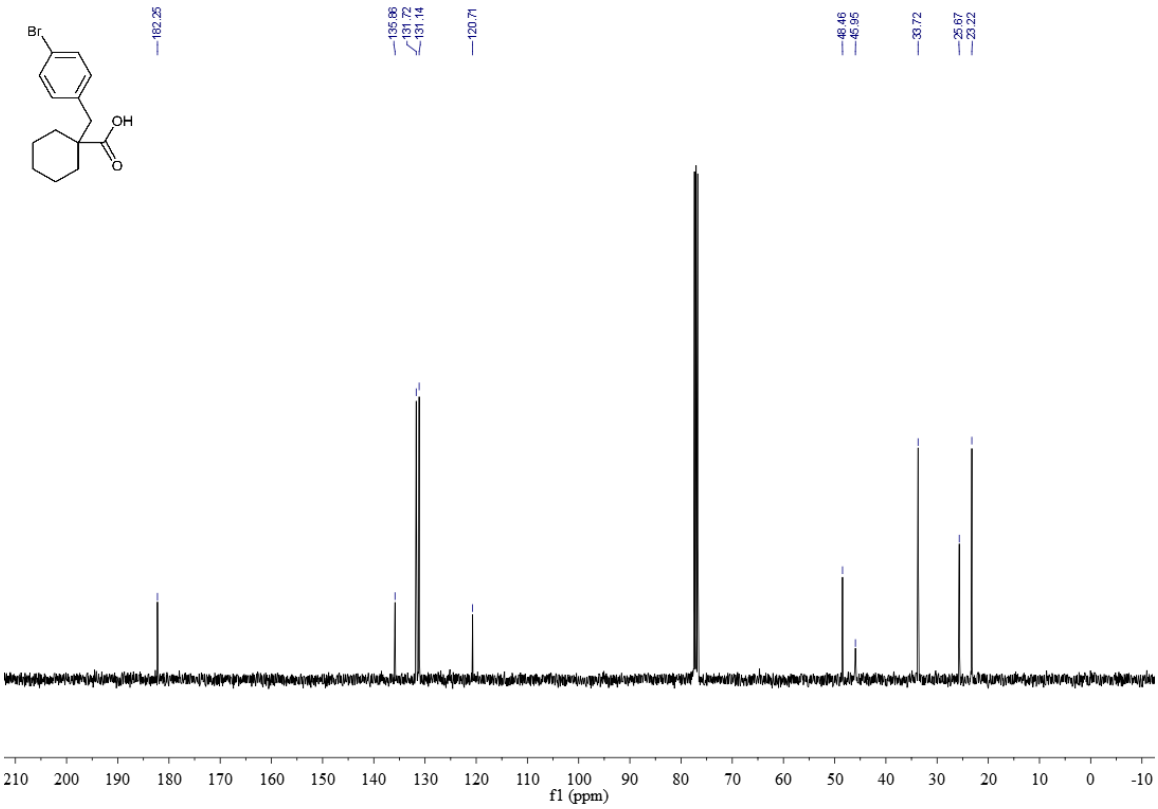
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **9**



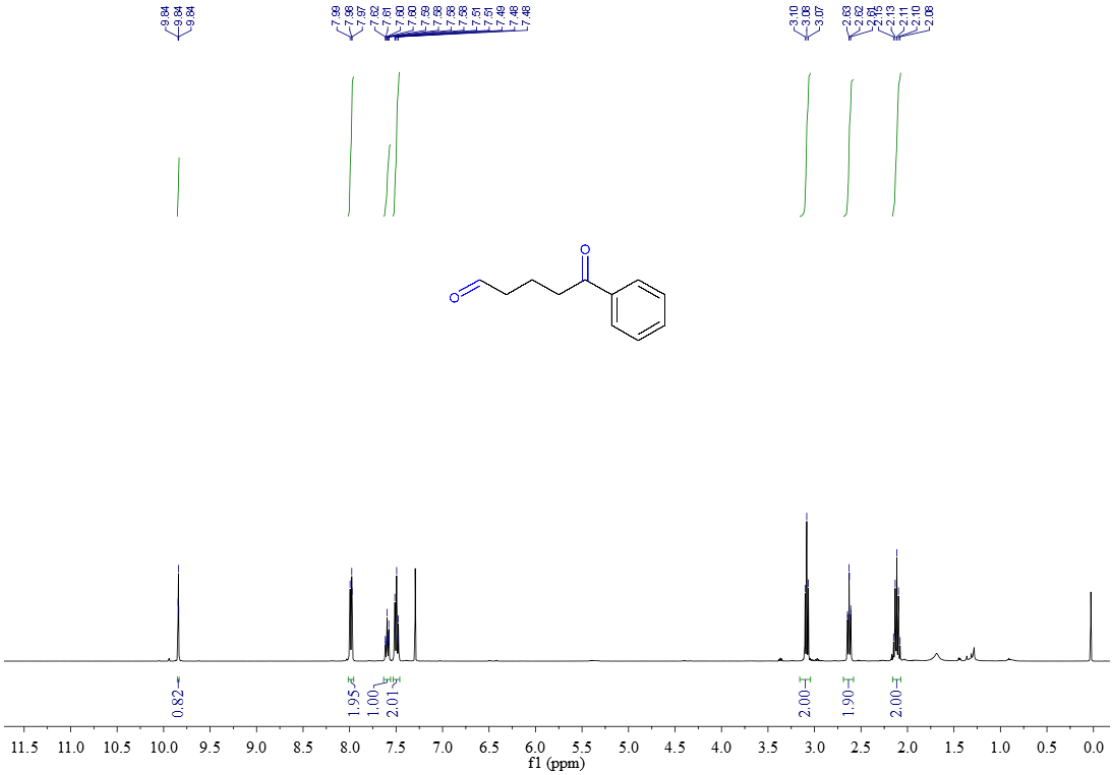
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **S1**



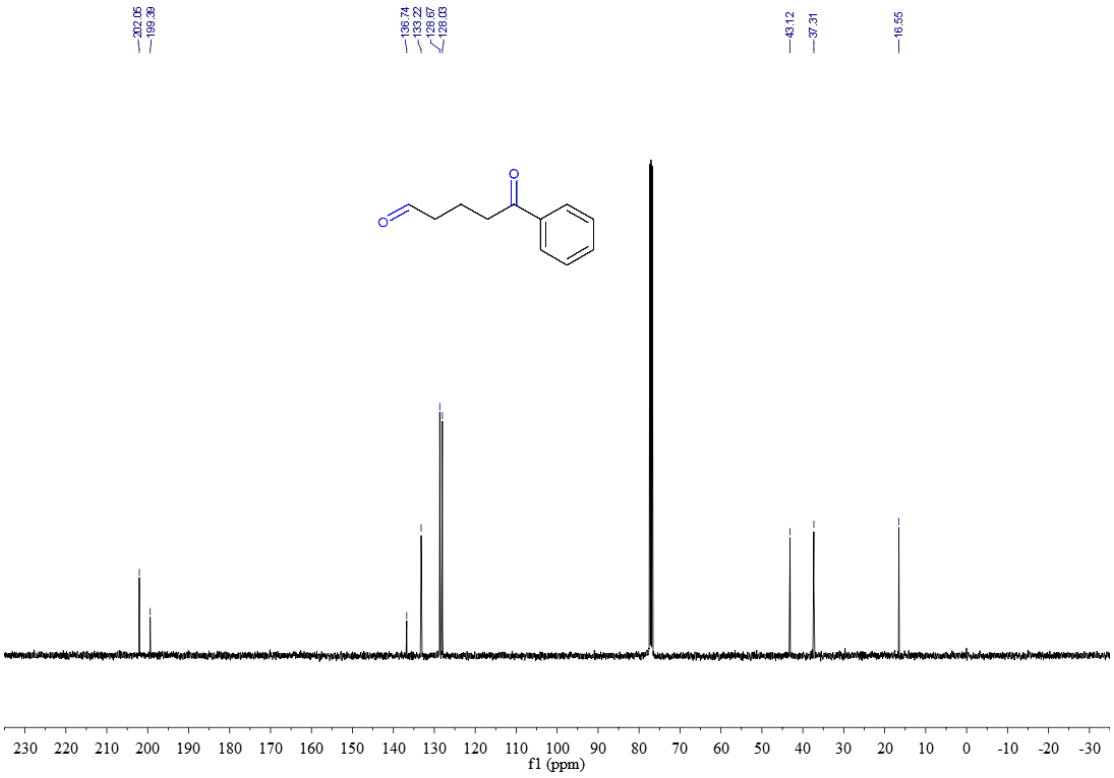
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **S1**



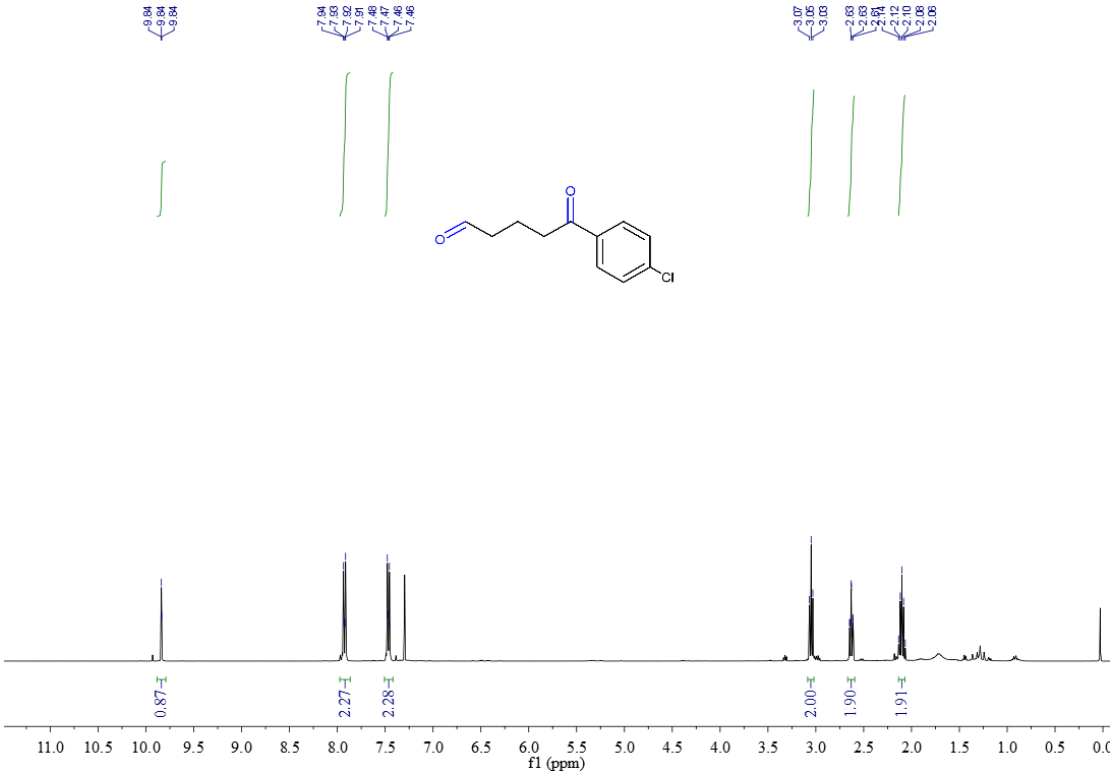
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2a**



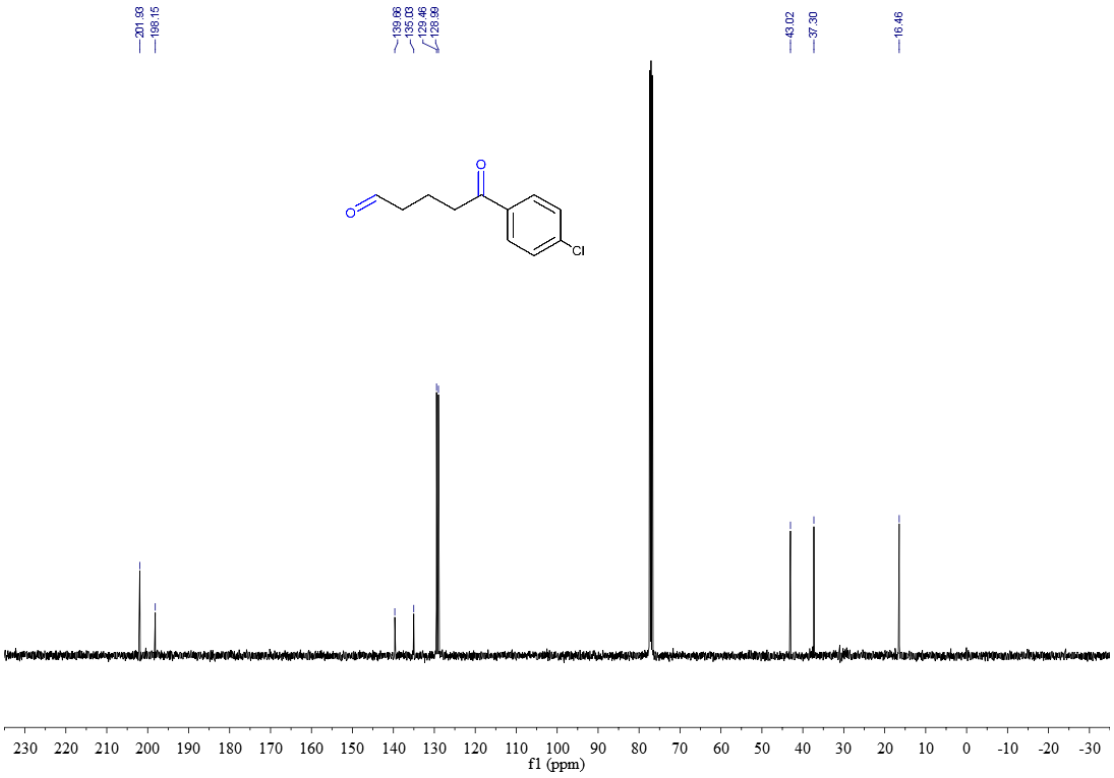
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2a**



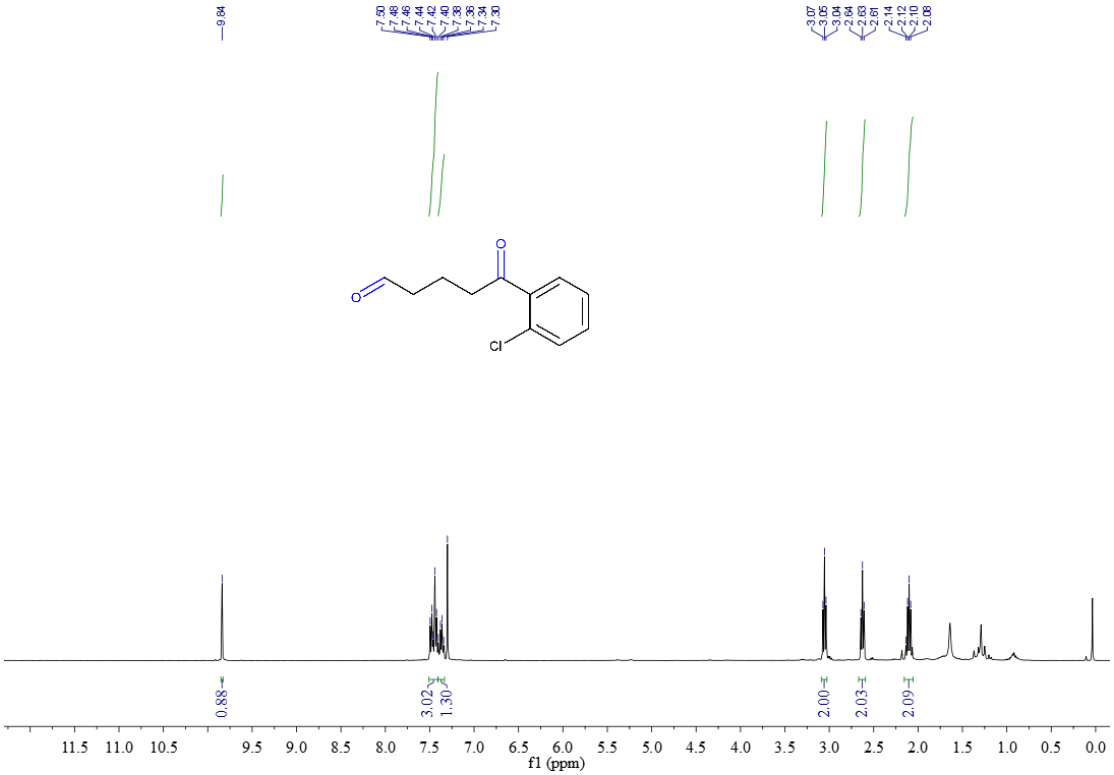
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2b**



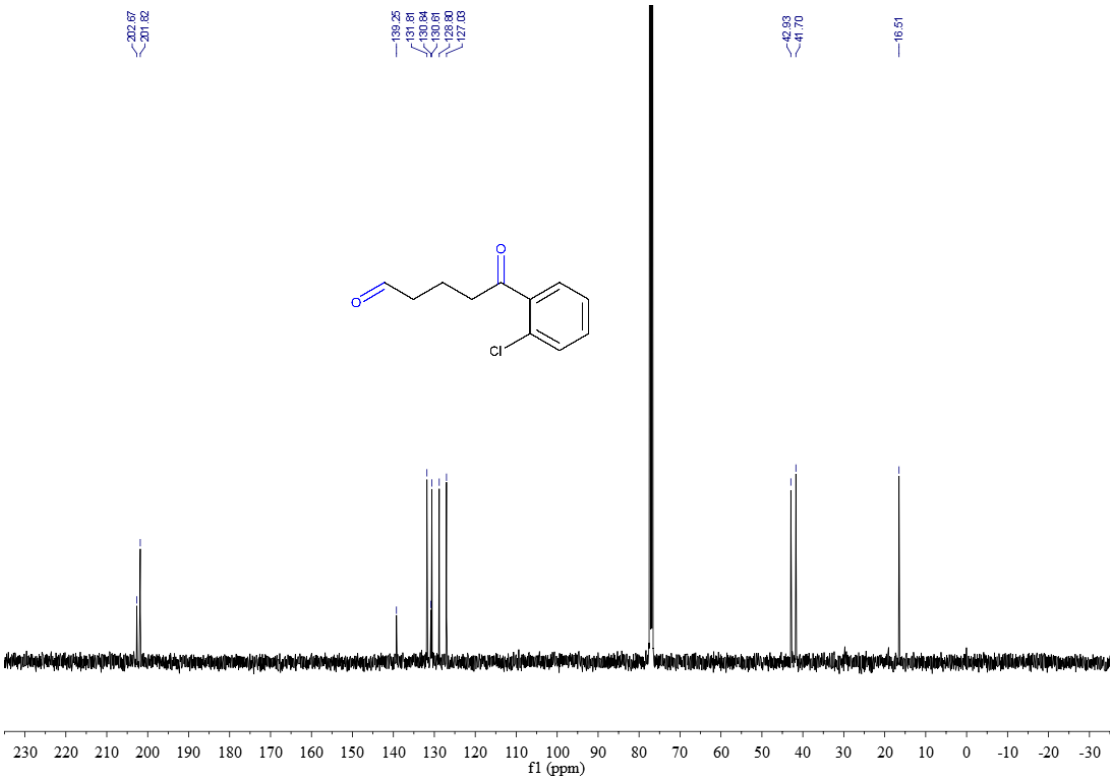
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2b**



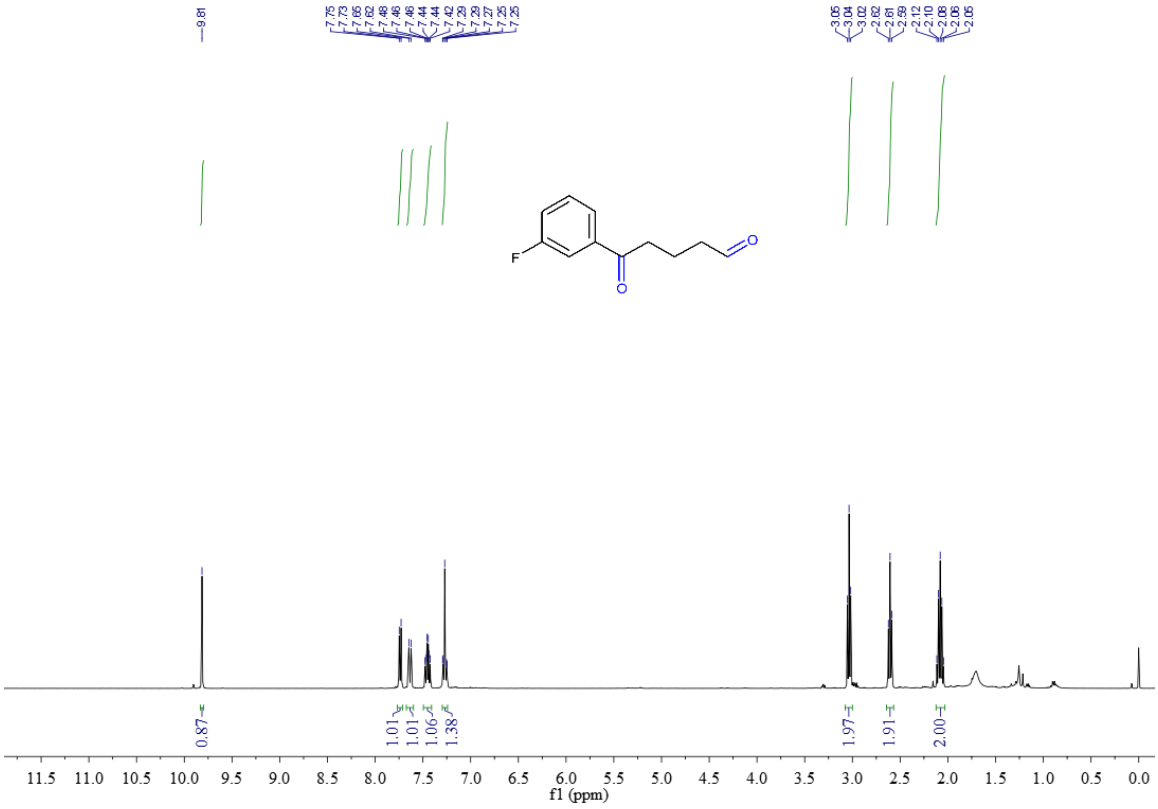
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2c**



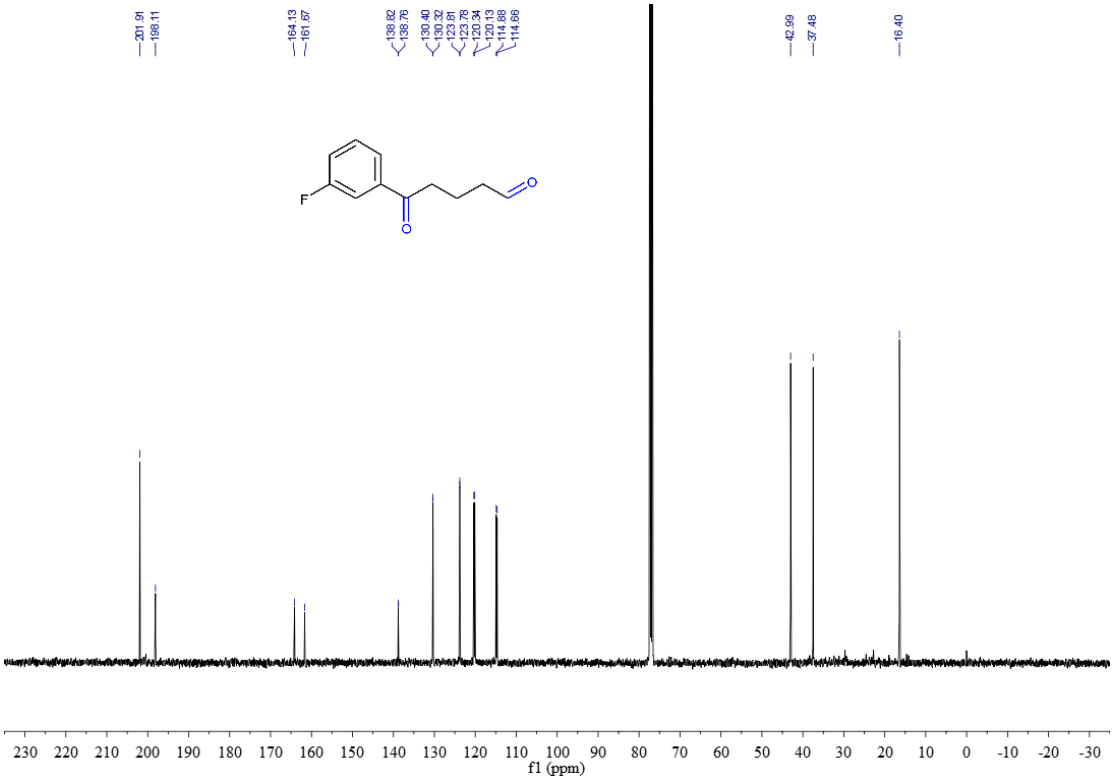
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2c**



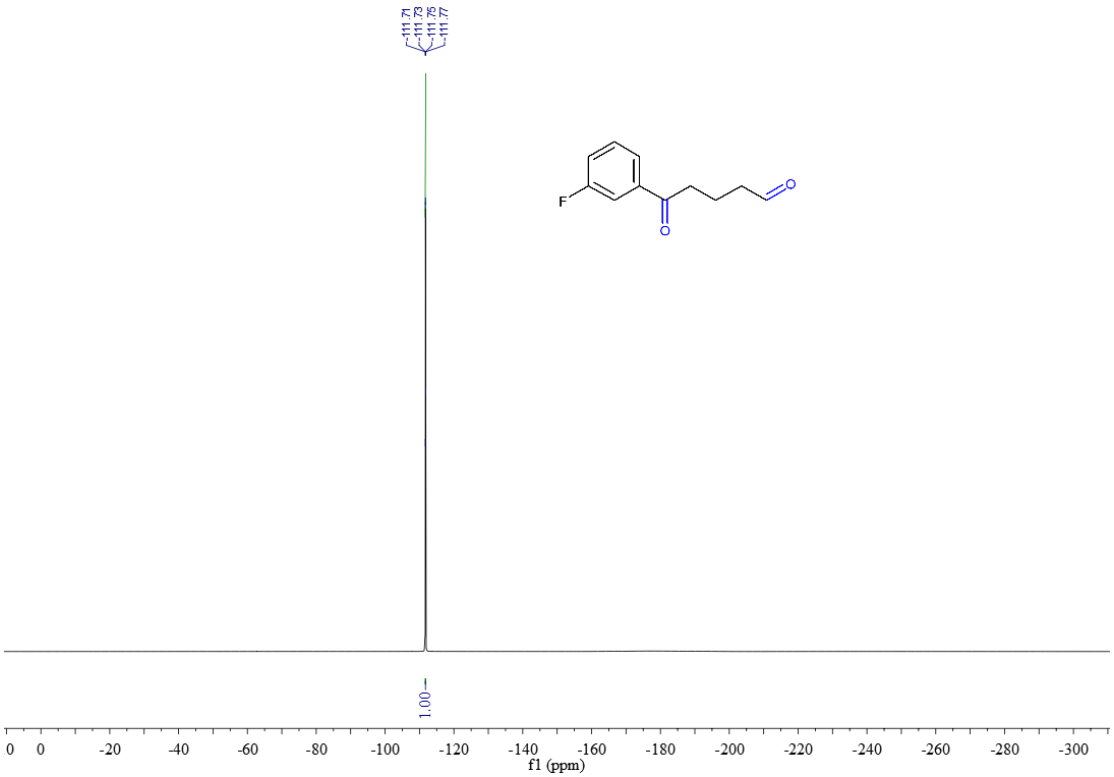
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2d**



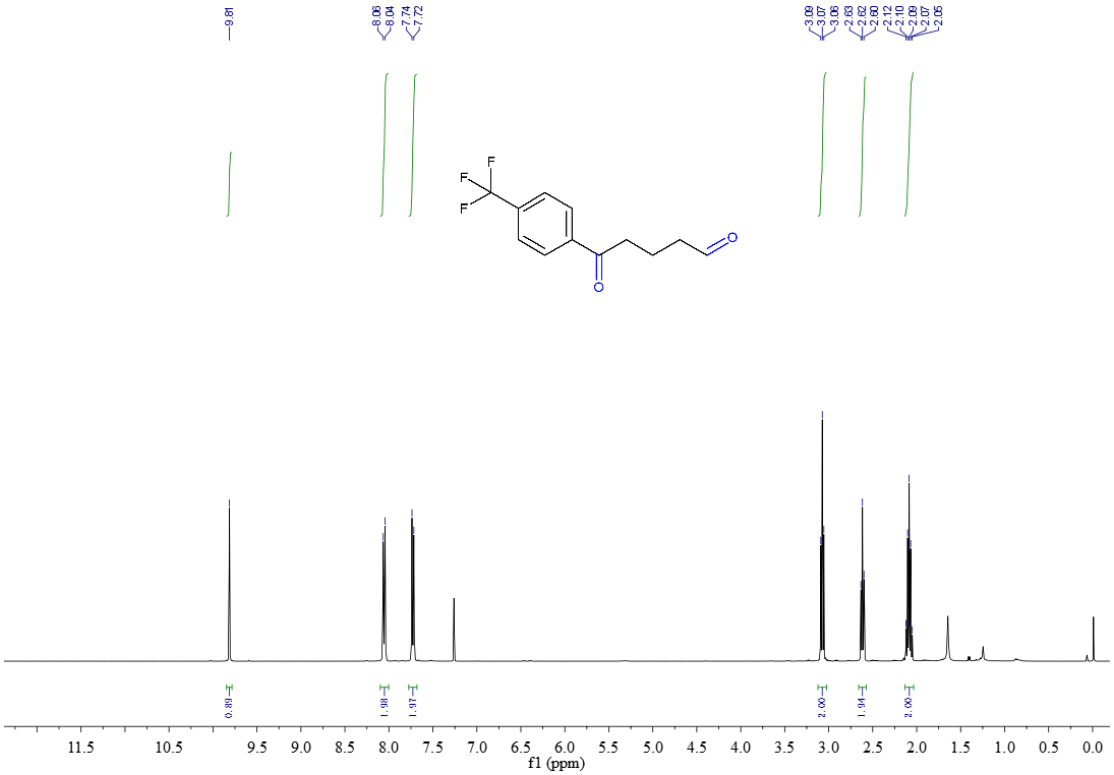
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2d**



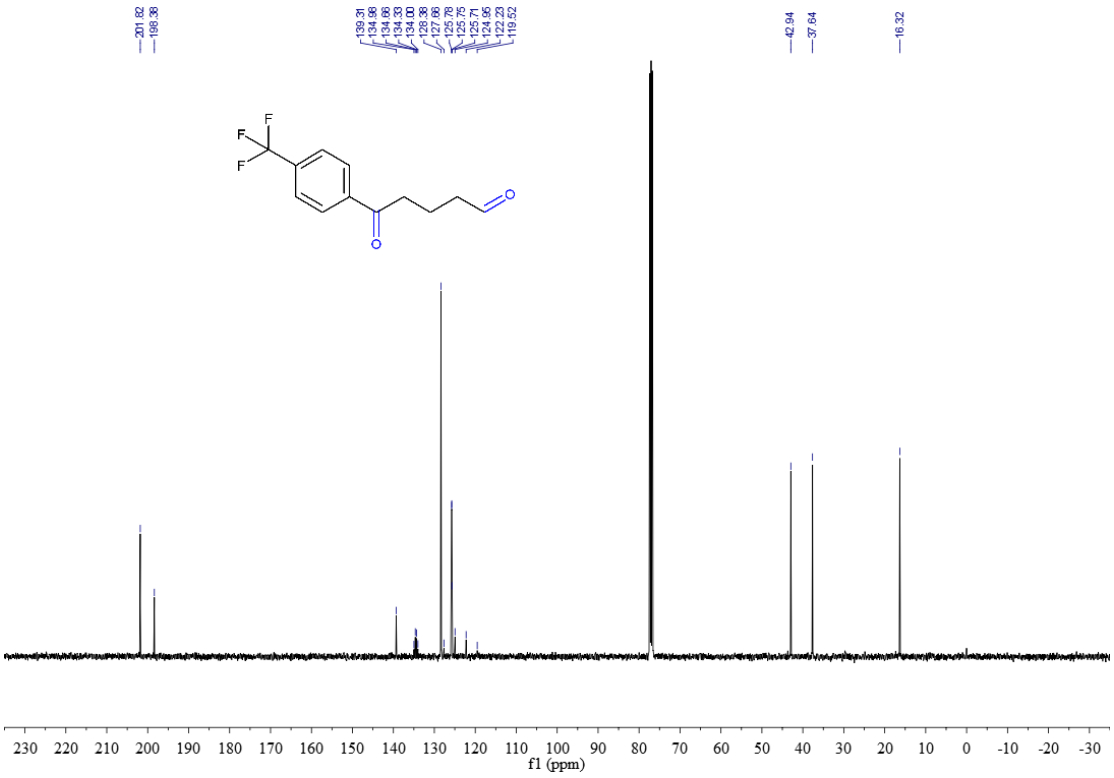
^{19}F NMR spectrum (376 MHz, $CDCl_3$, 23 ℃) of **2d**



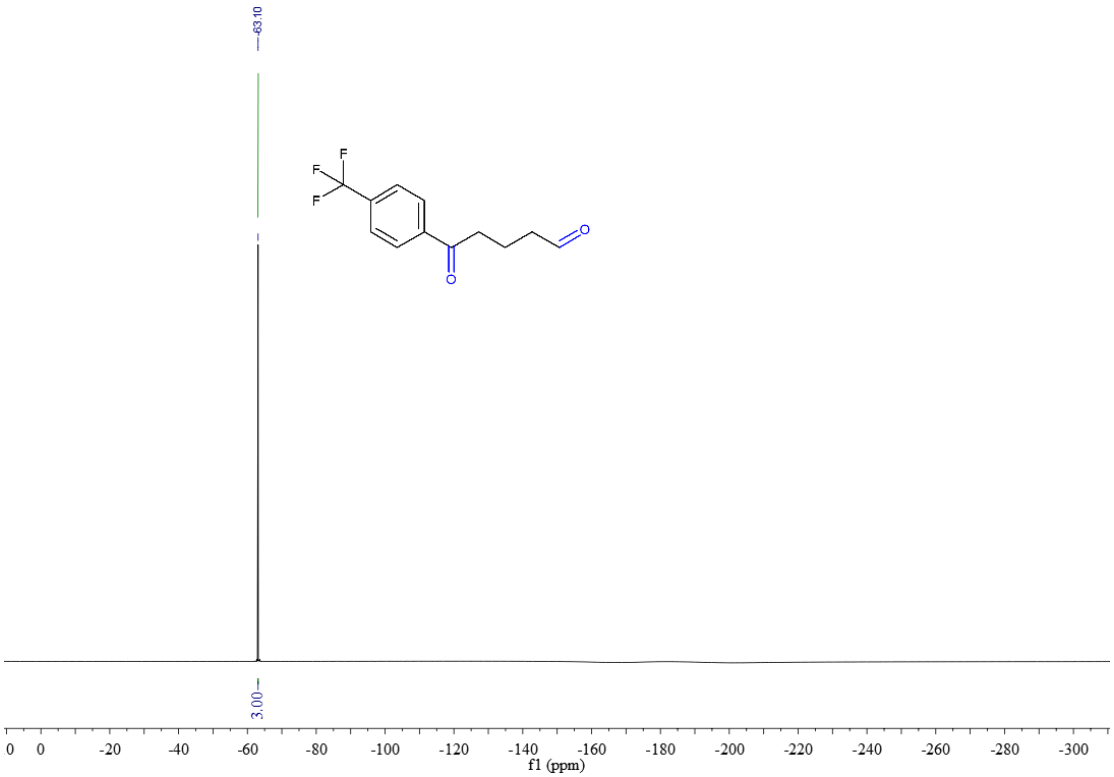
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2e**



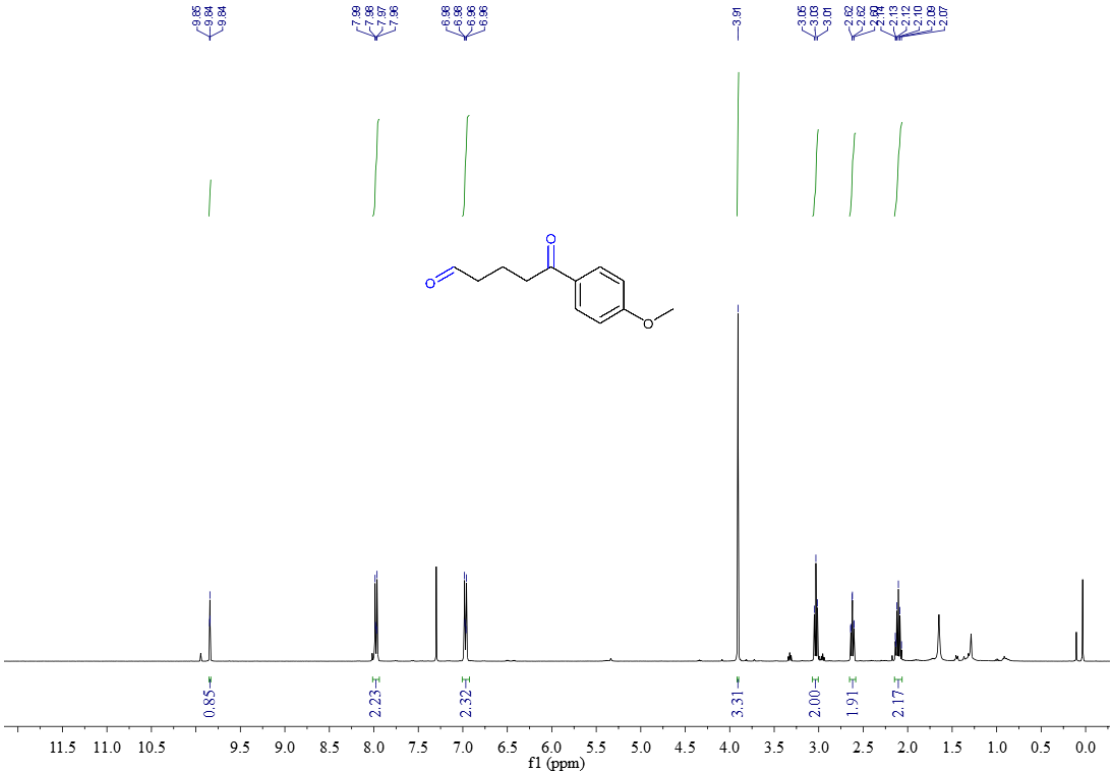
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2e**



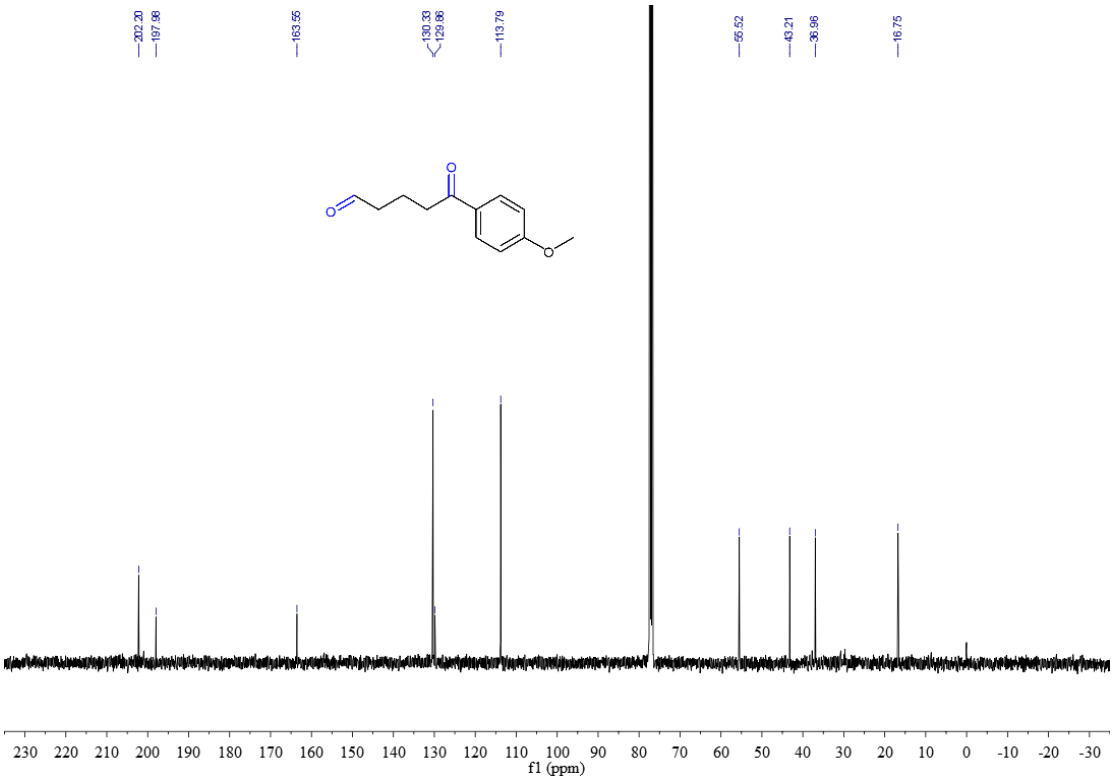
^{19}F NMR spectrum (376 MHz, $CDCl_3$, 23 ℃) of **2e**



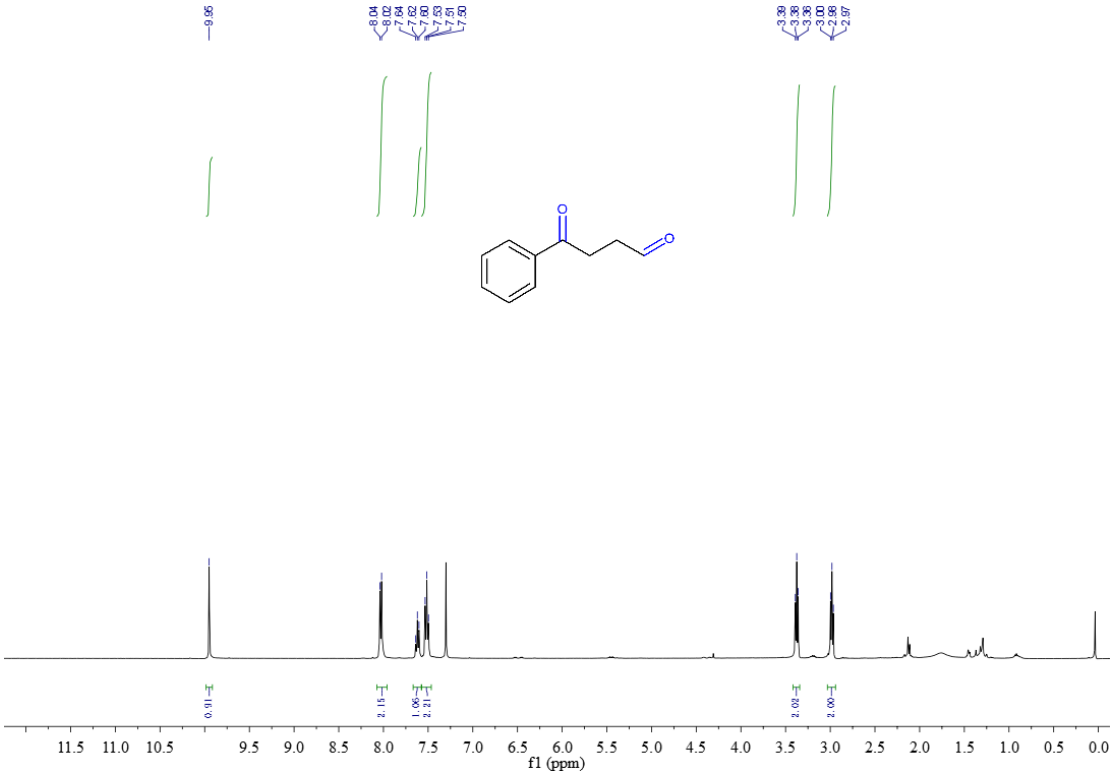
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2f**



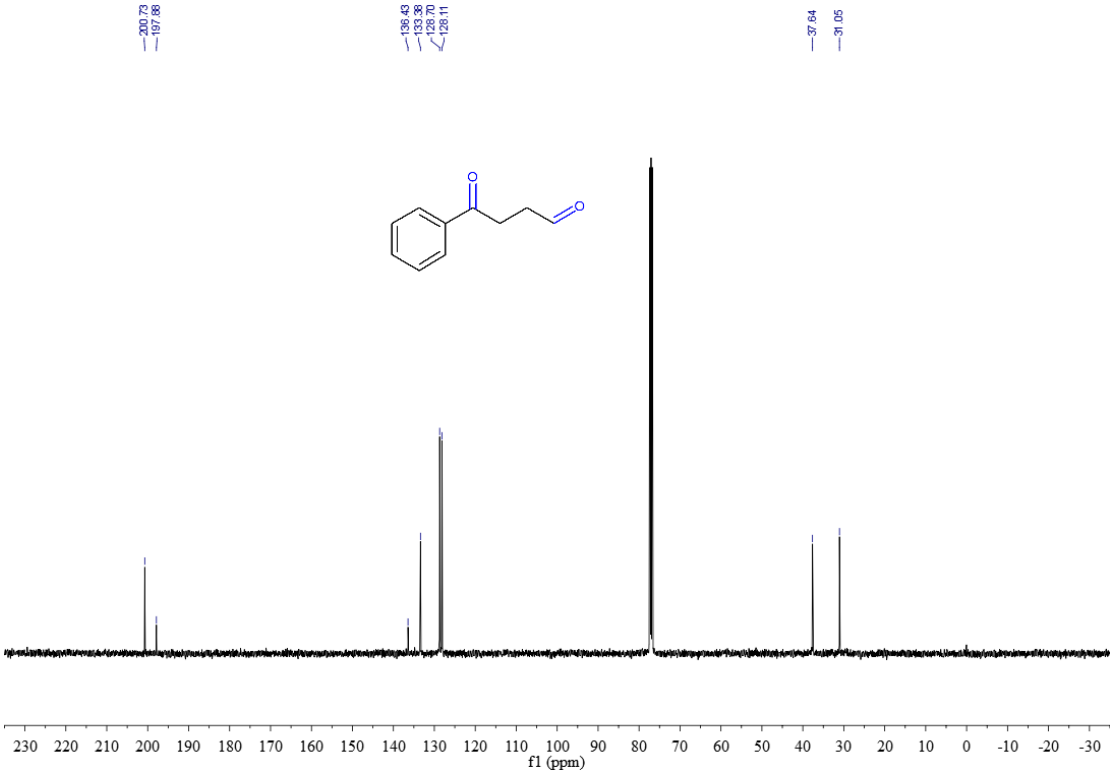
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2f**



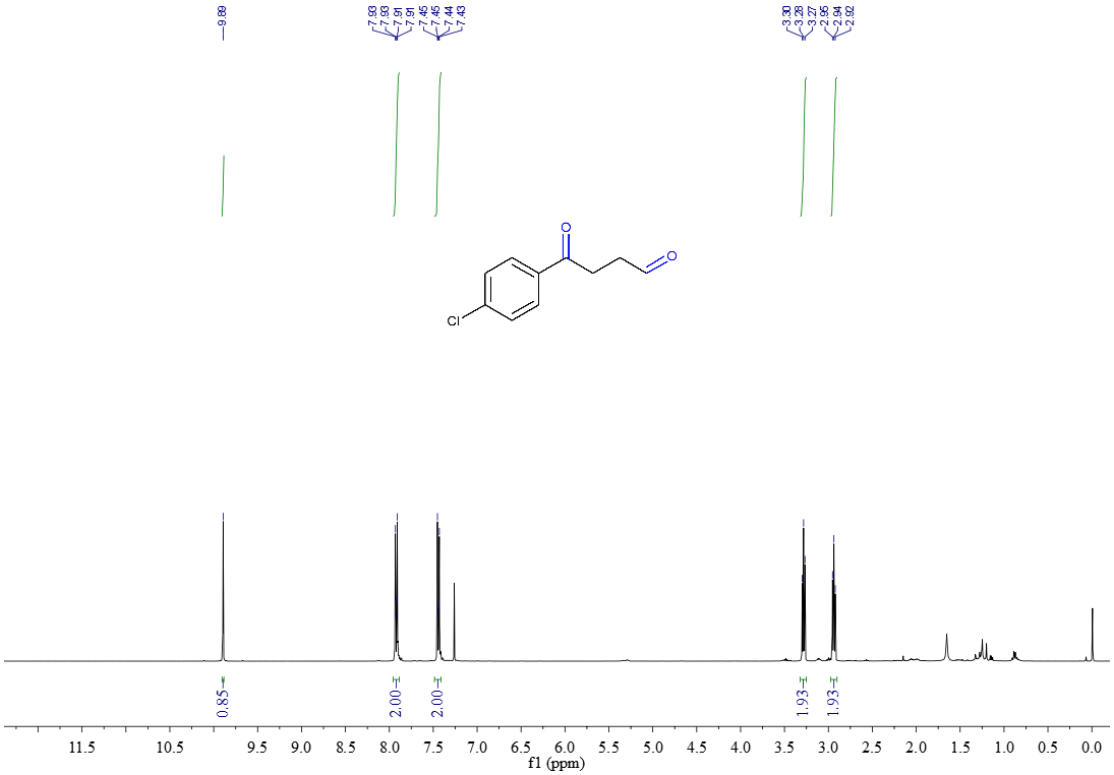
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2g**



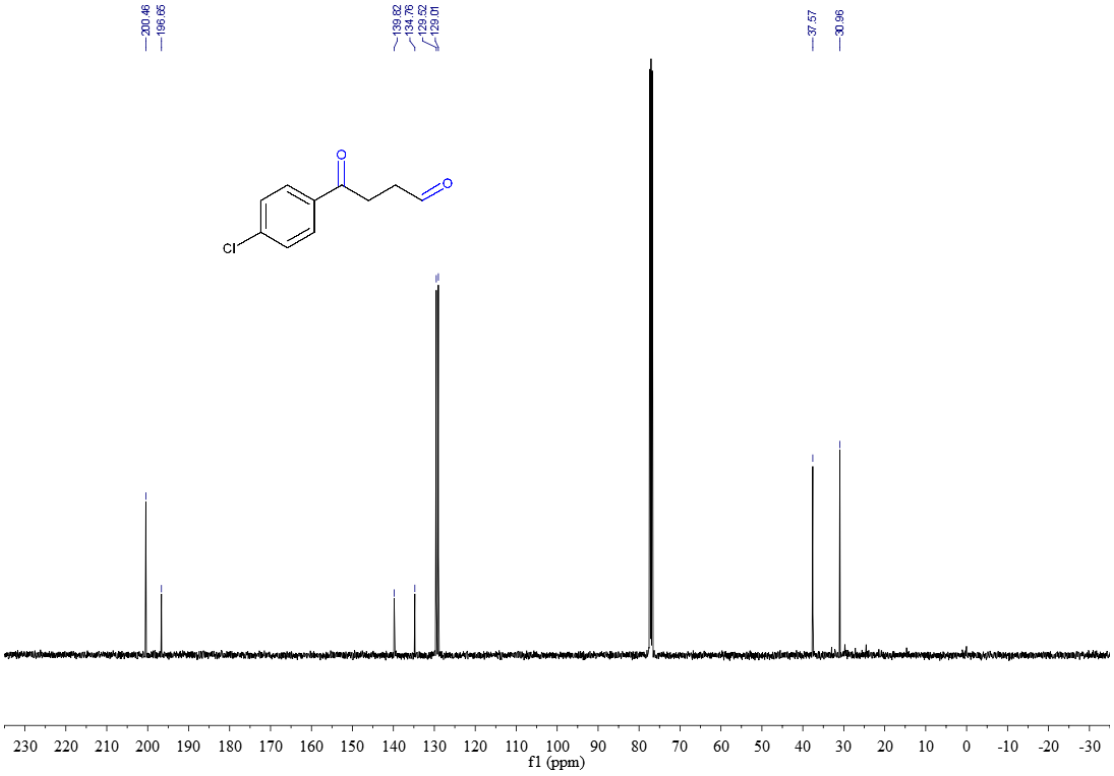
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2g**



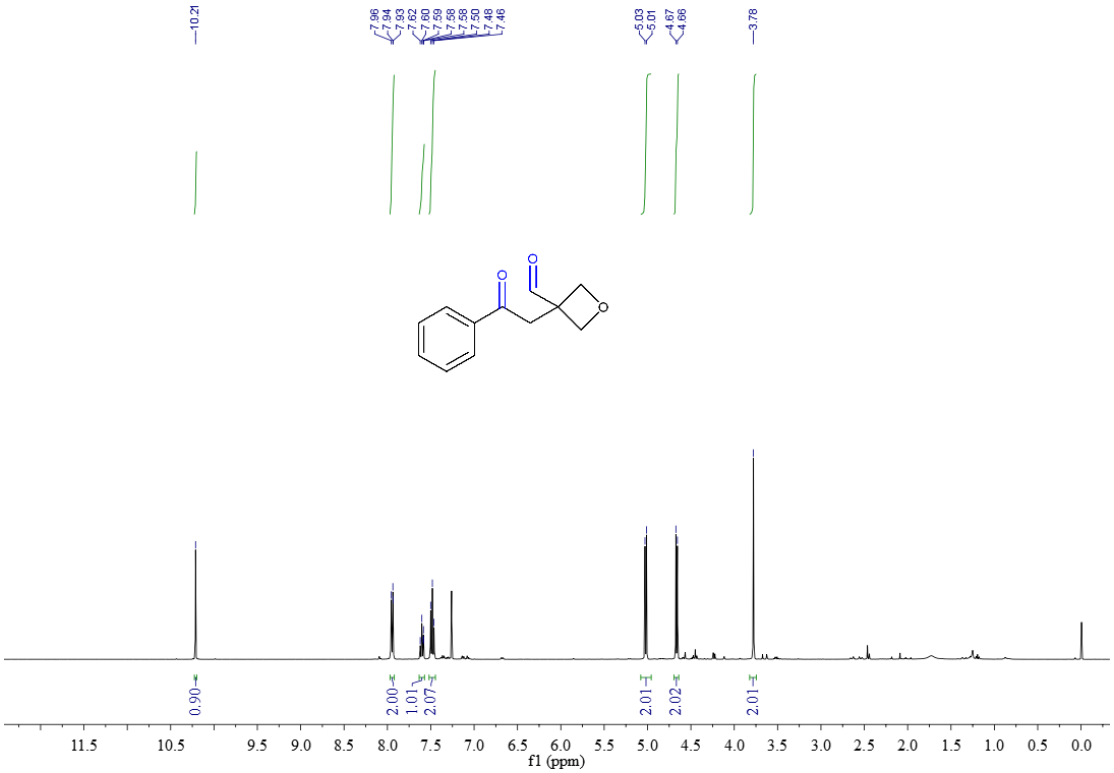
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2h**



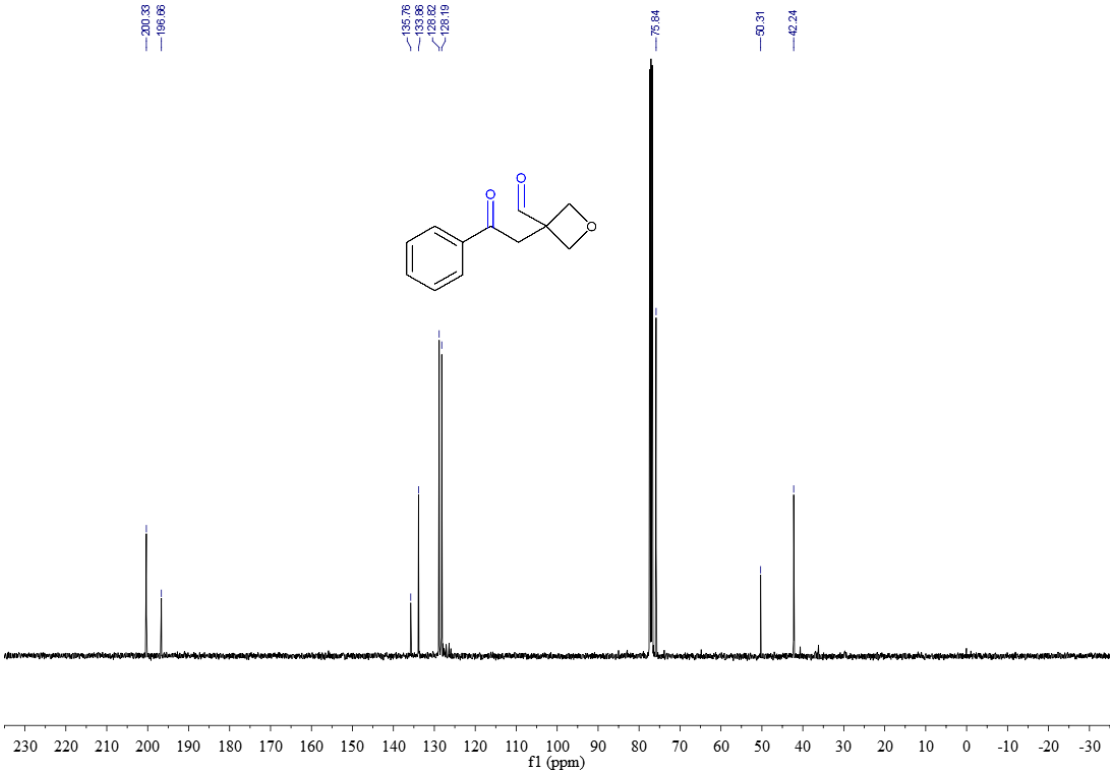
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2h**



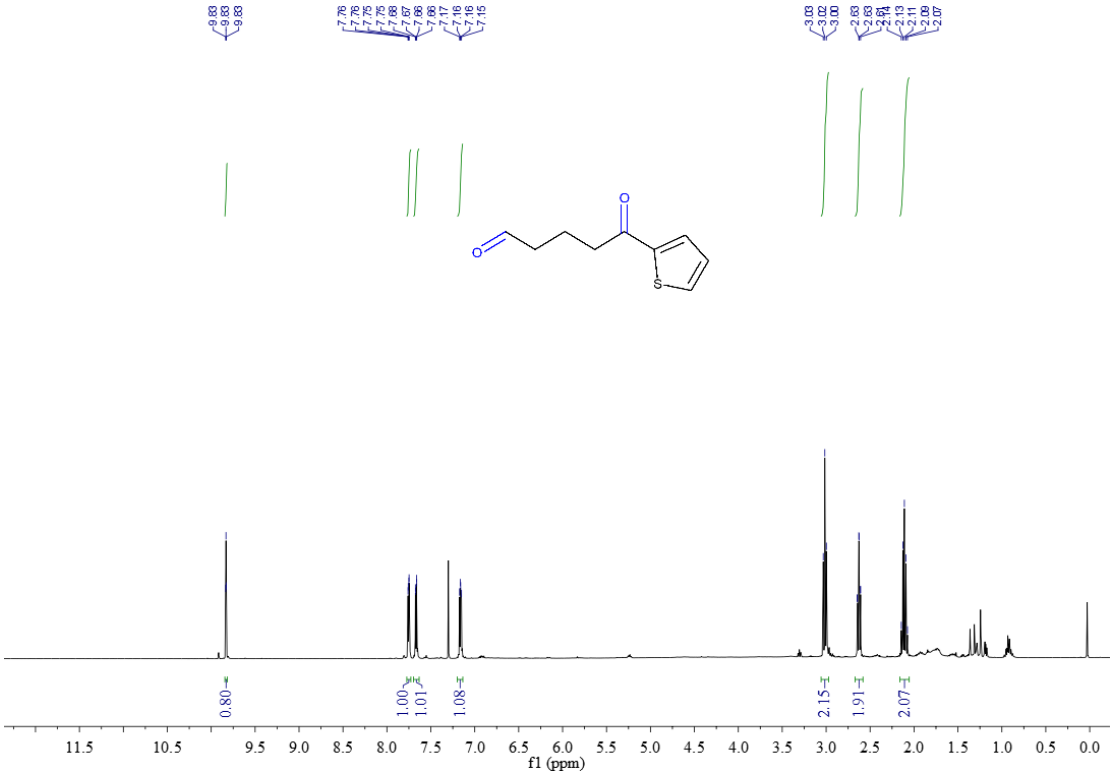
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2i**



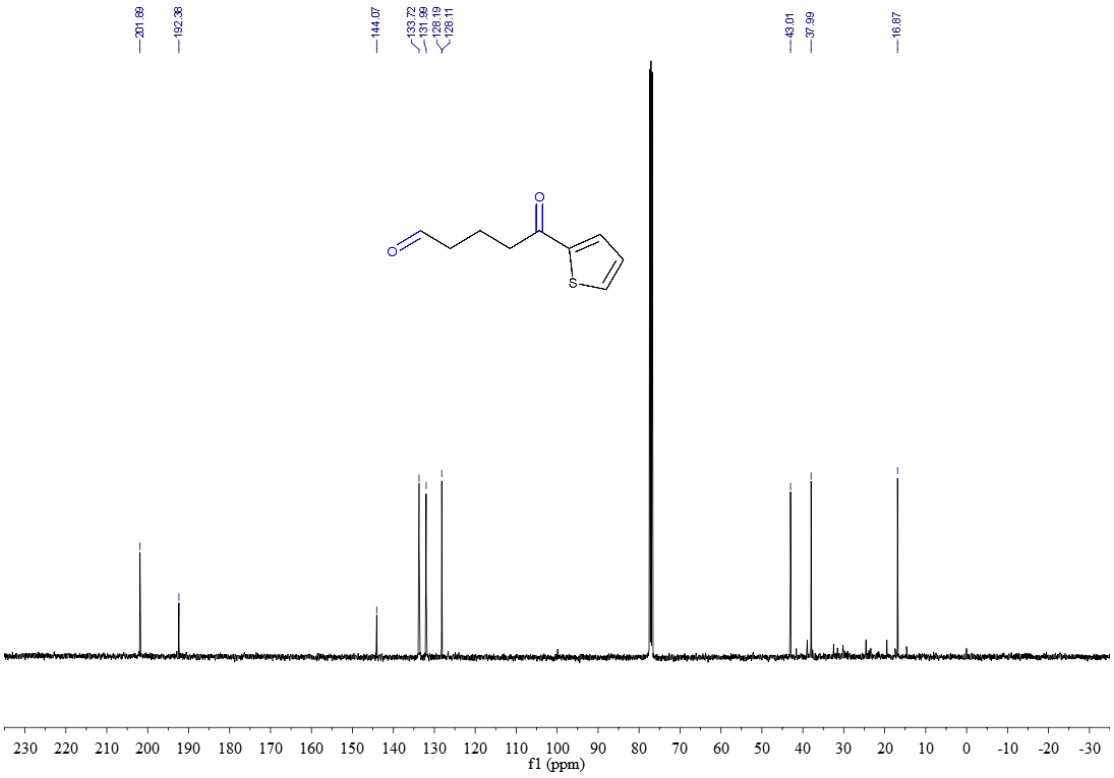
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2i**



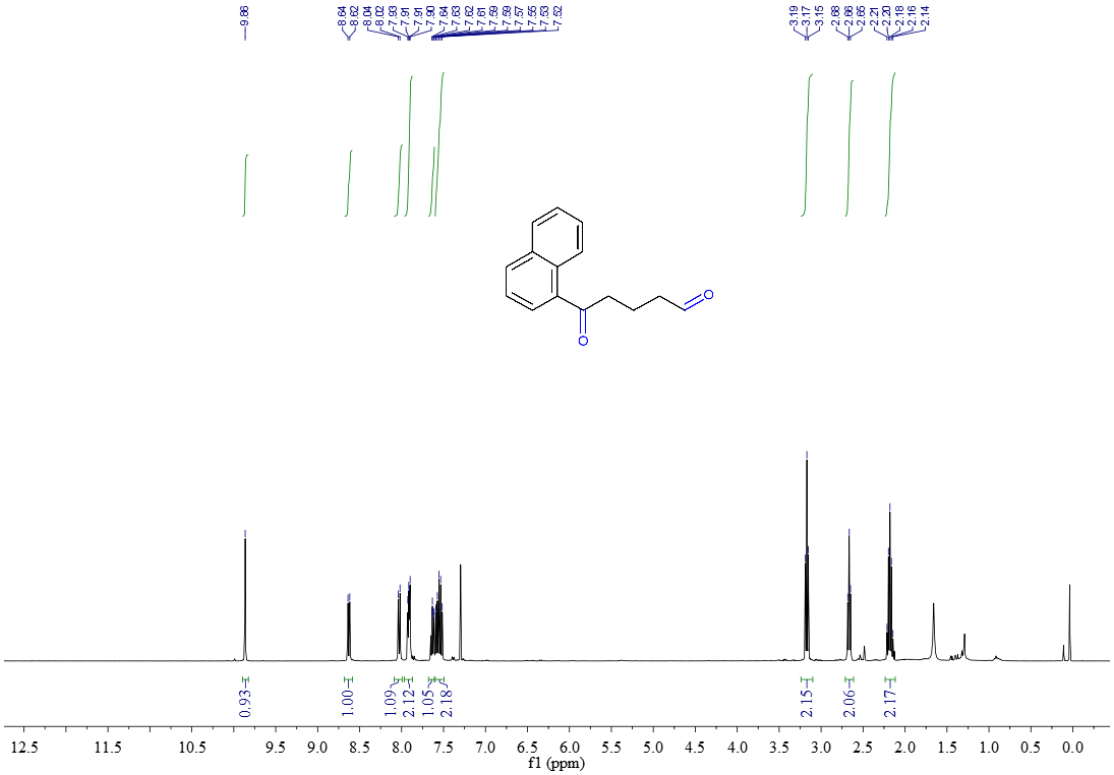
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2j**



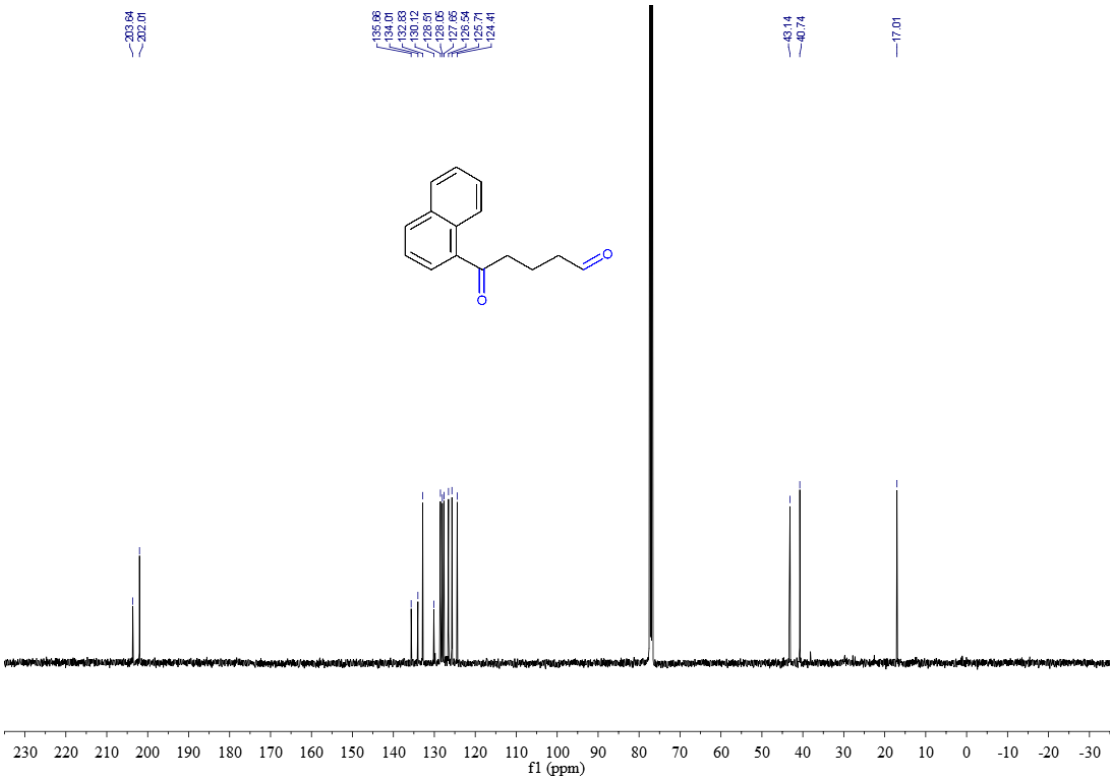
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2j**



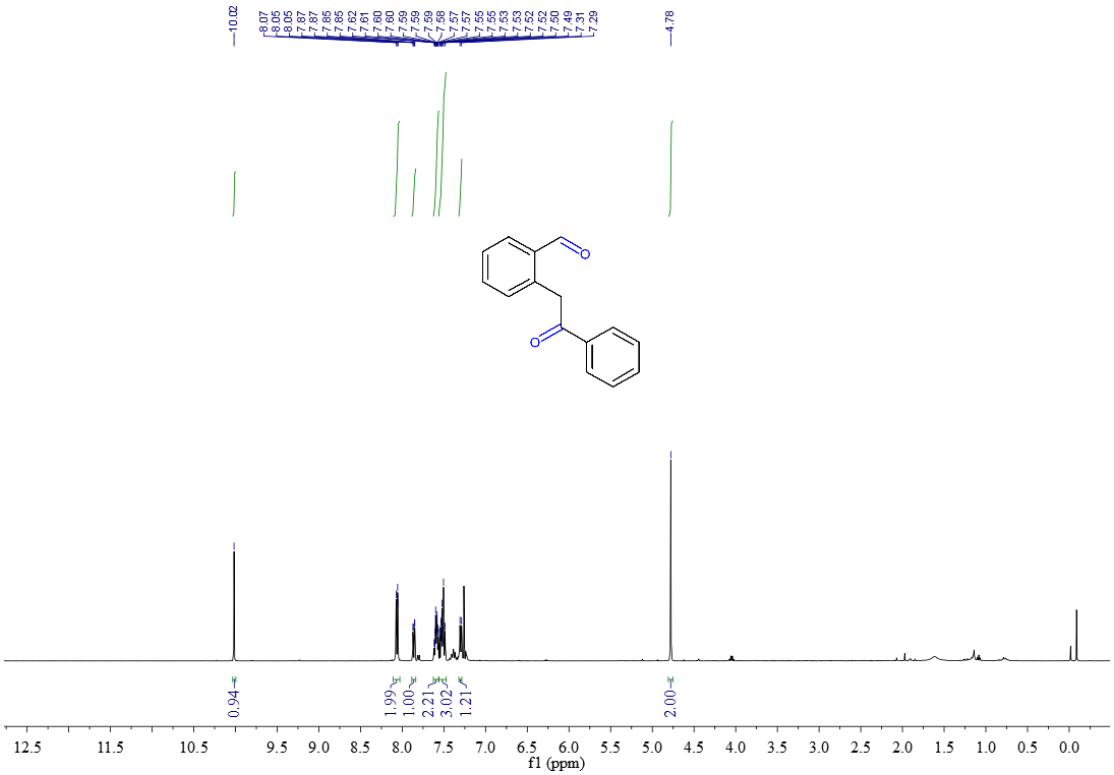
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2k**



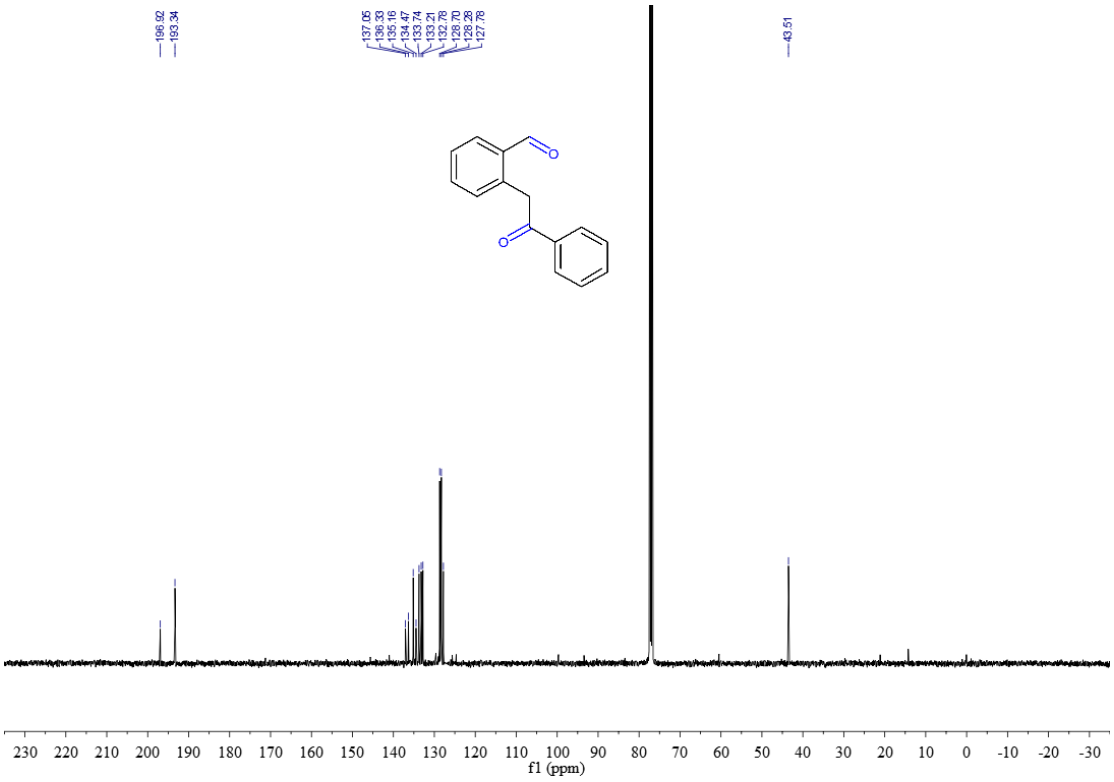
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2k**



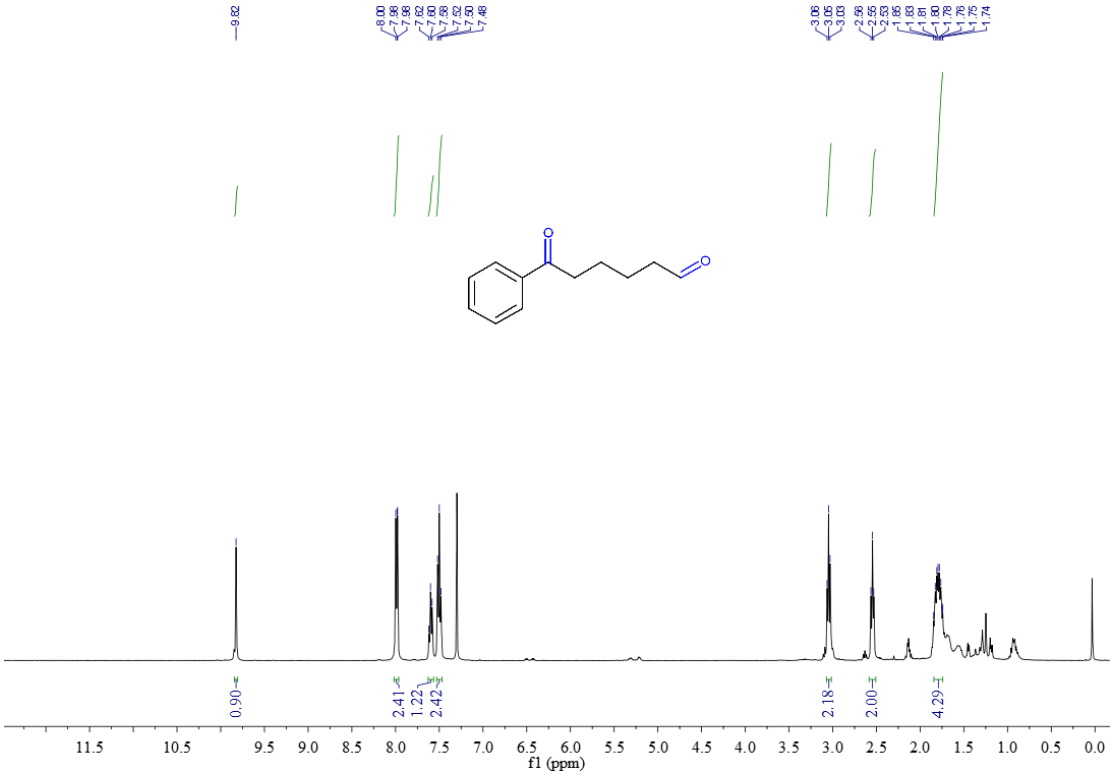
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2l**



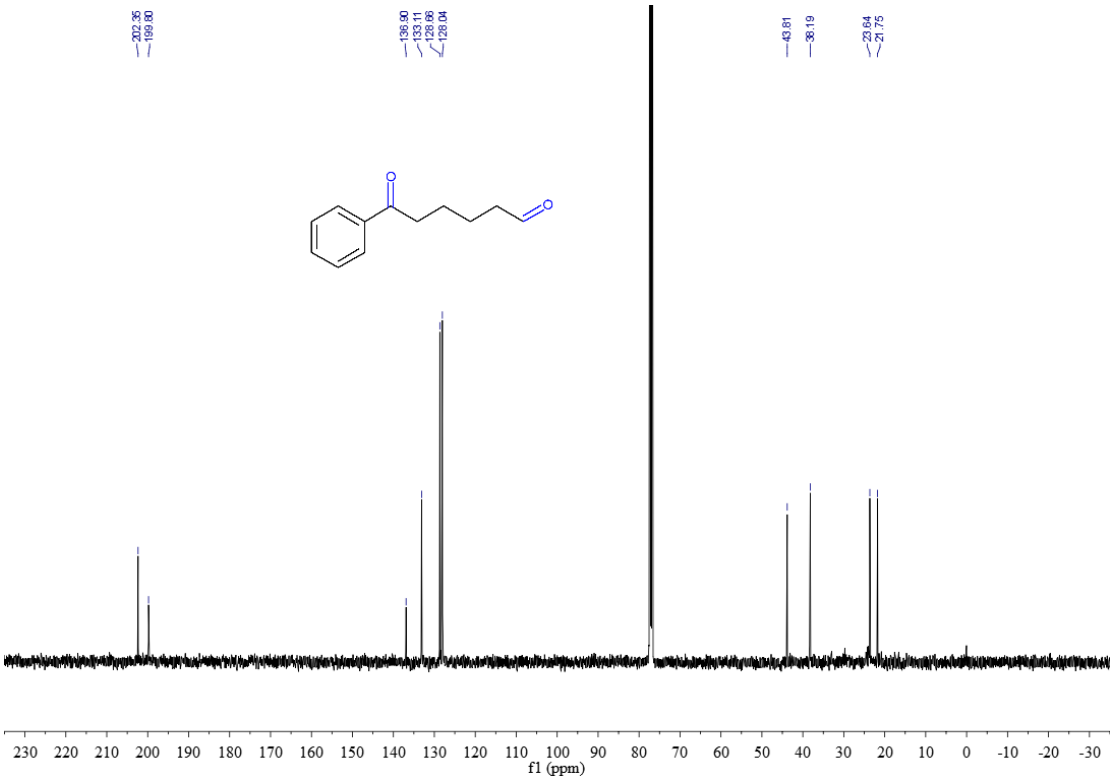
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2l**



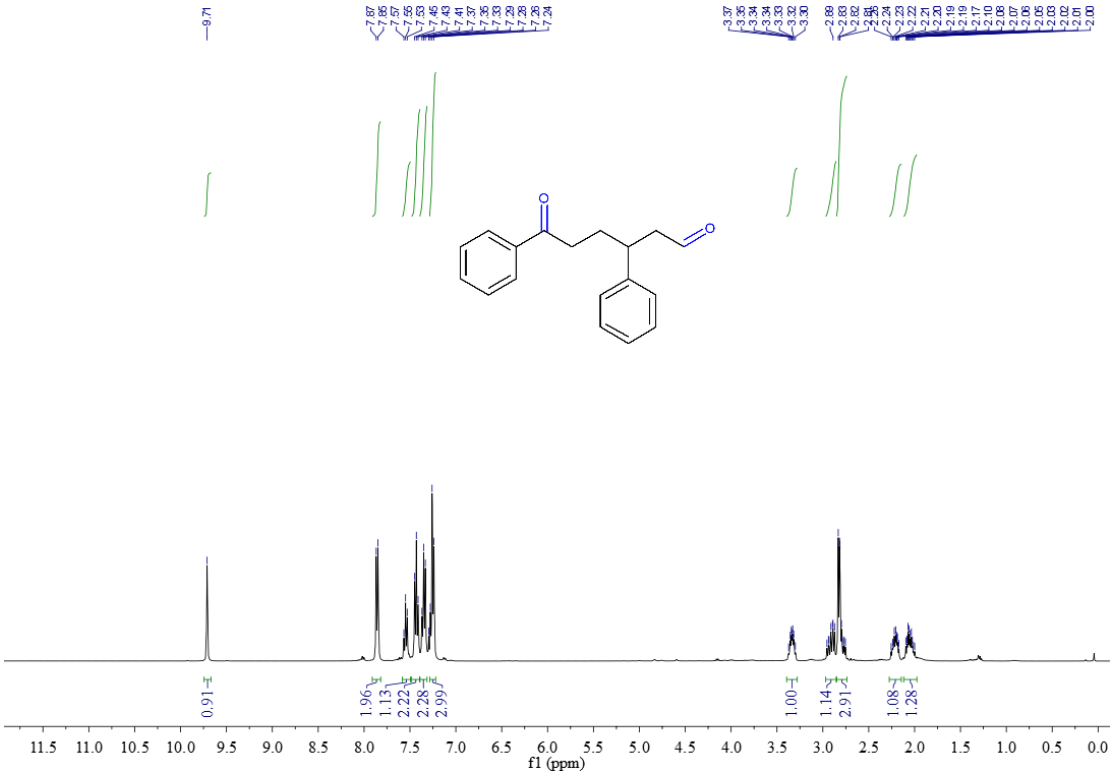
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2m**



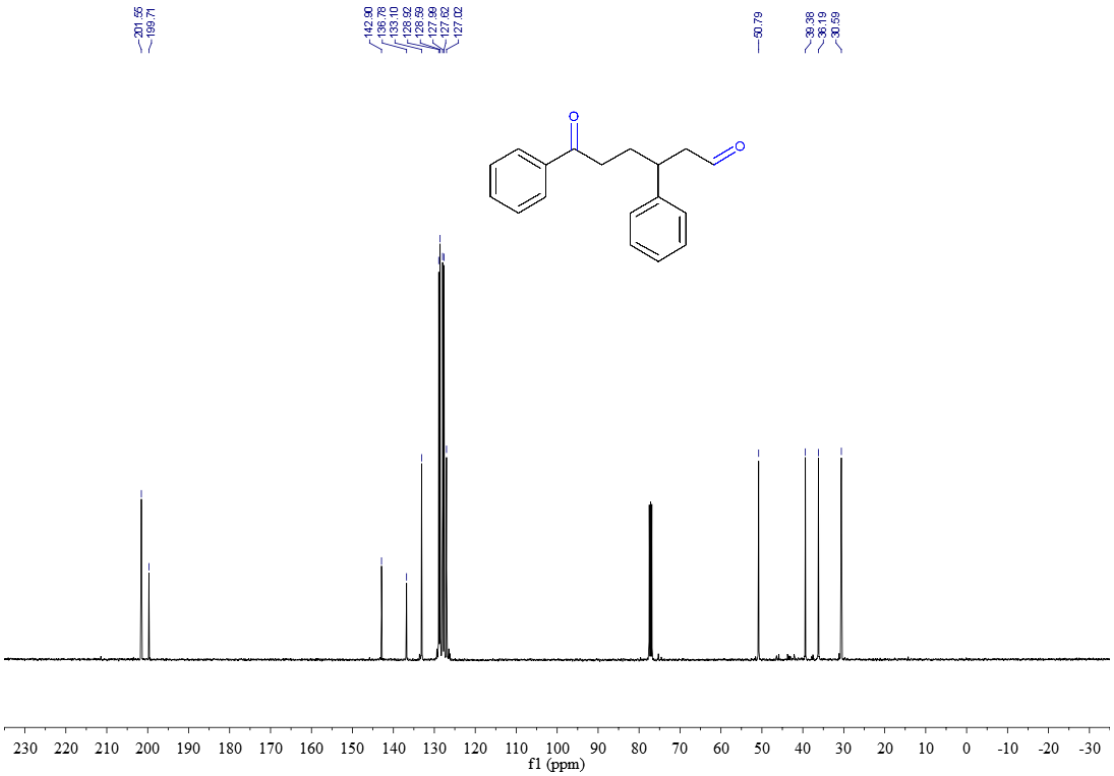
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2m**



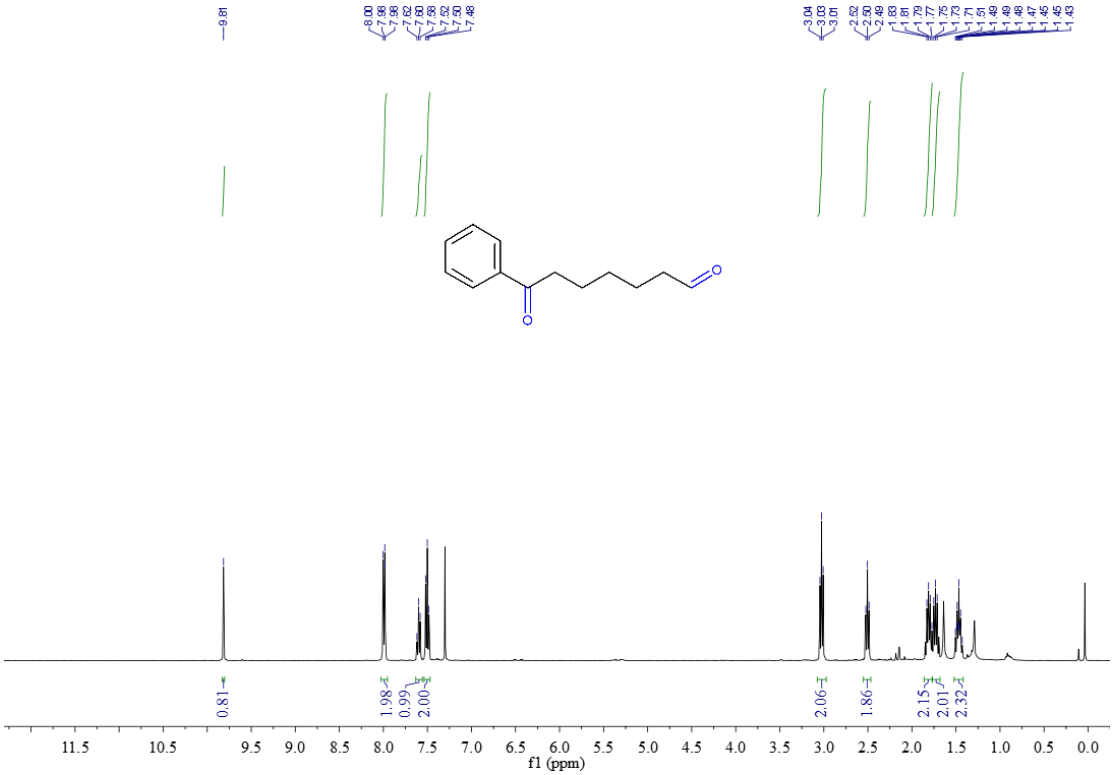
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2n**



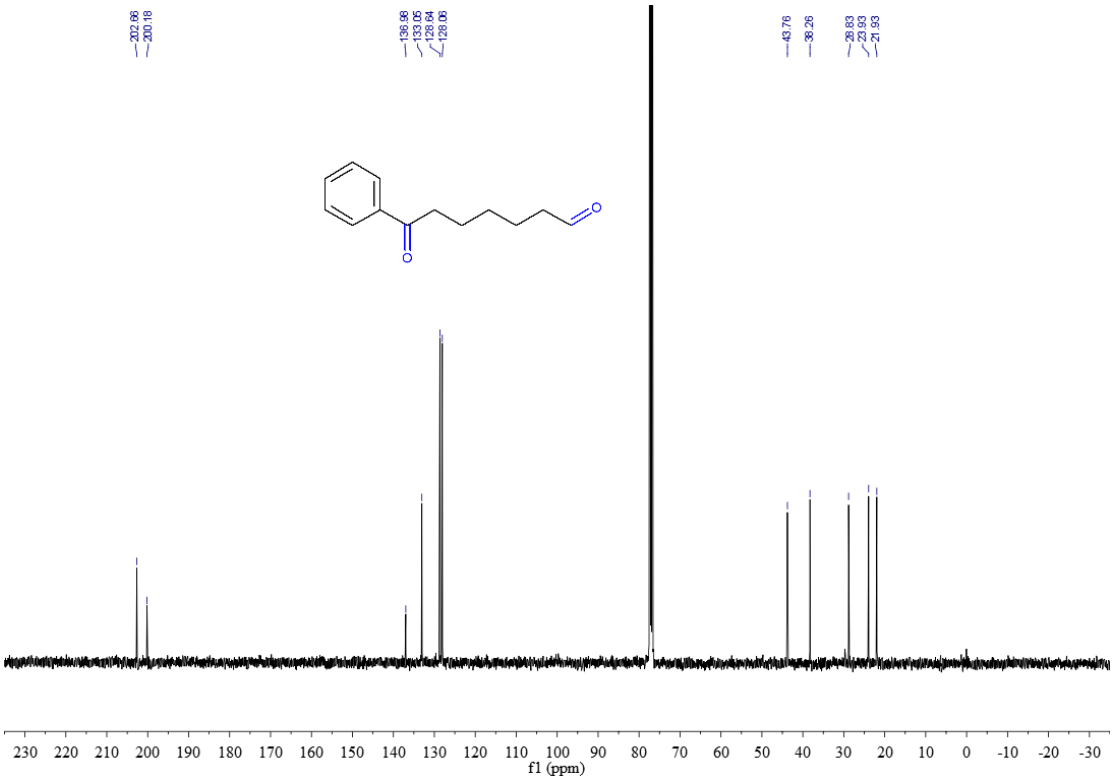
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2n**



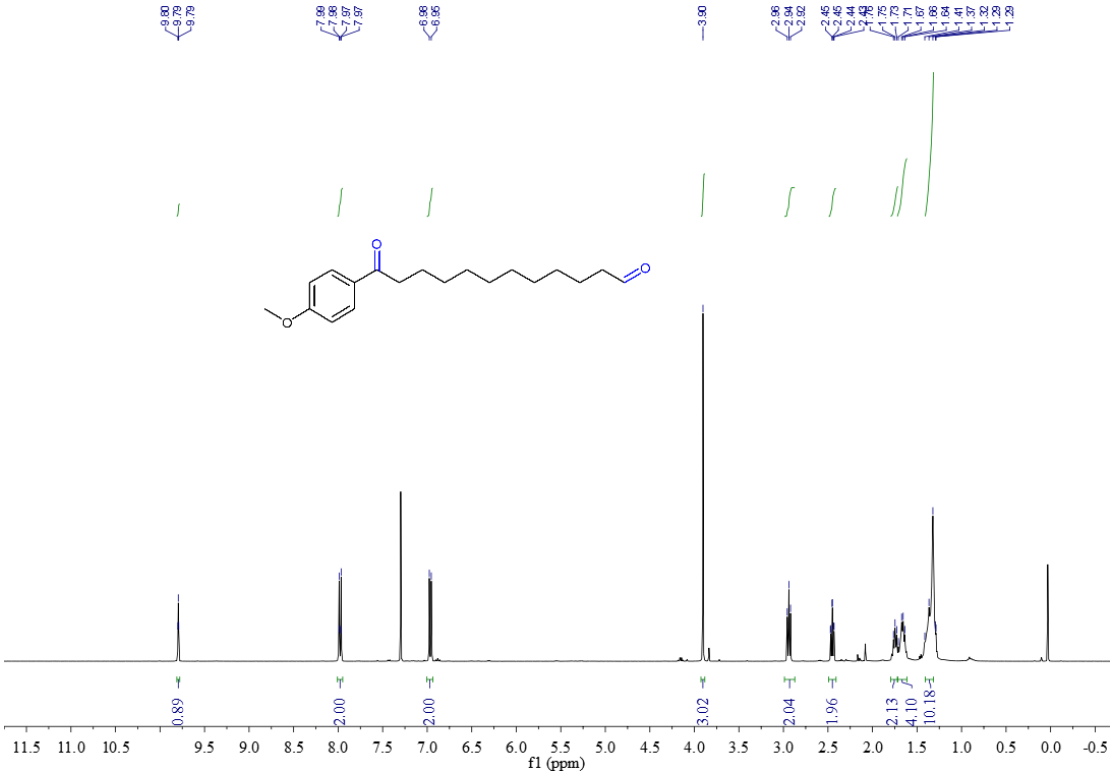
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2o**



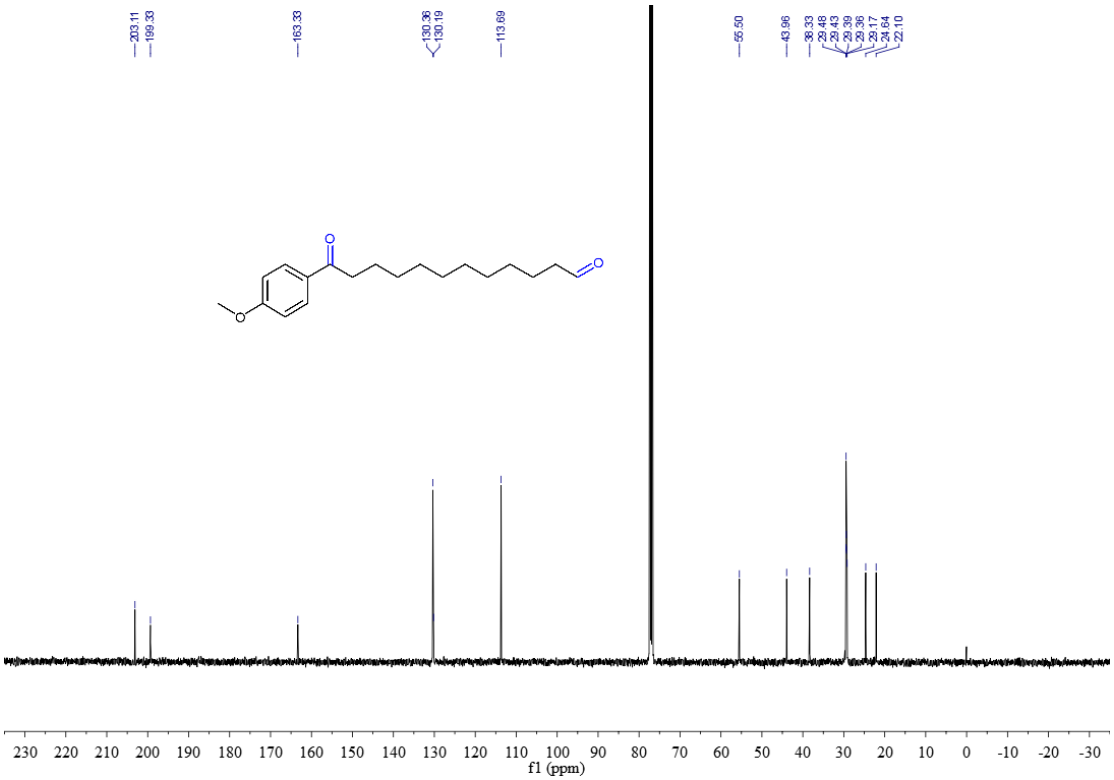
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2o**



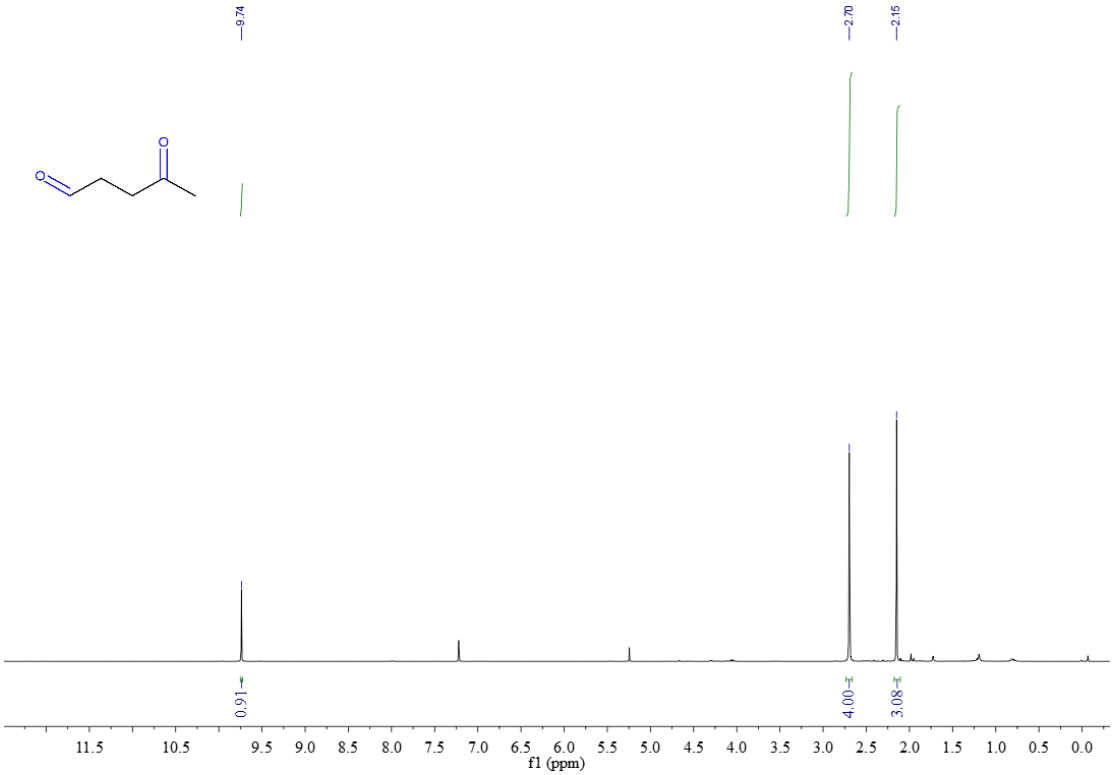
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2p**



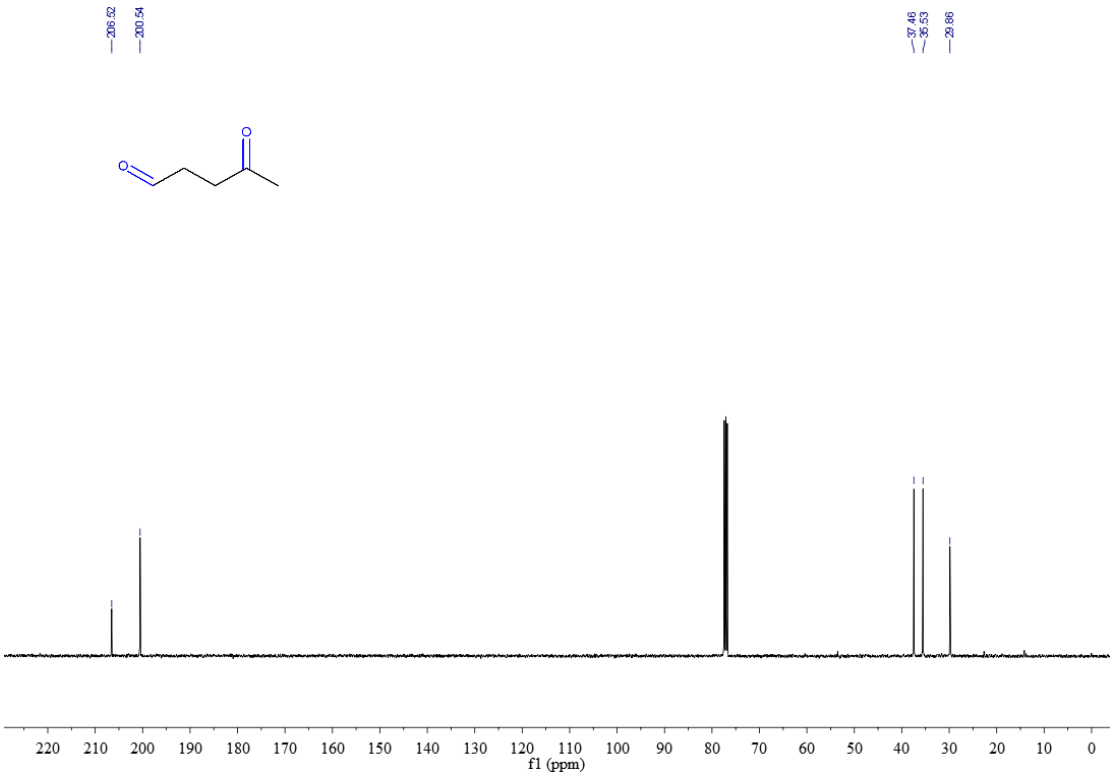
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2p**



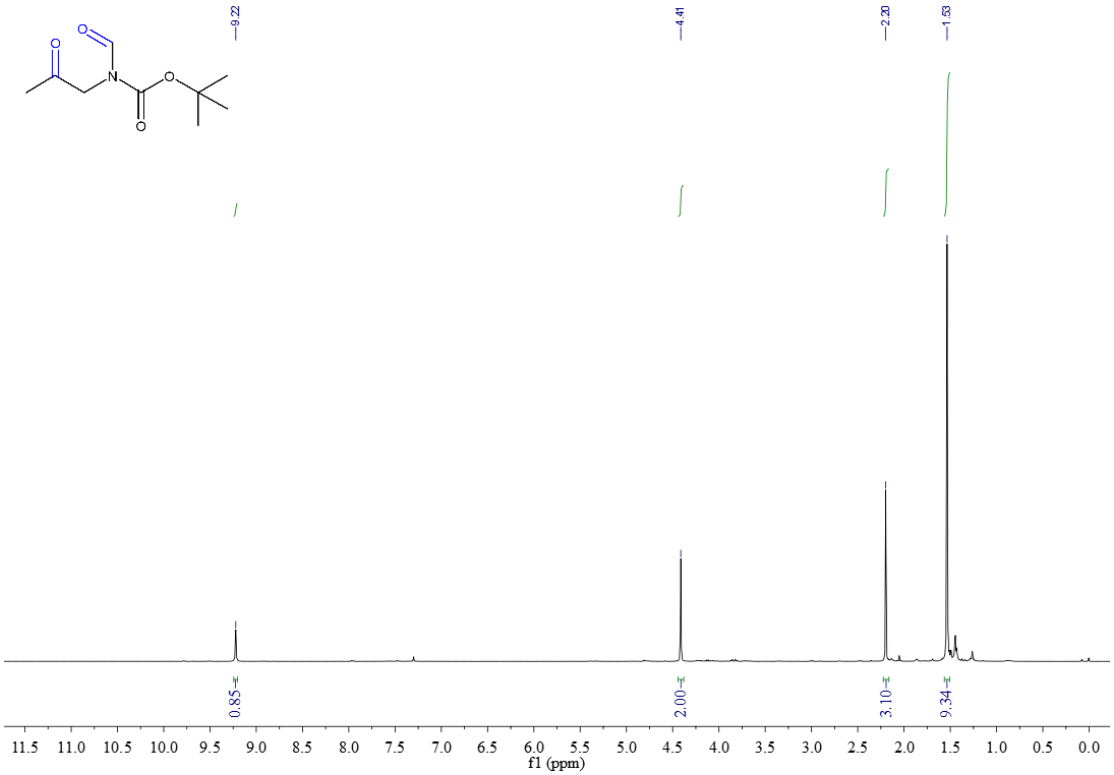
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2q**



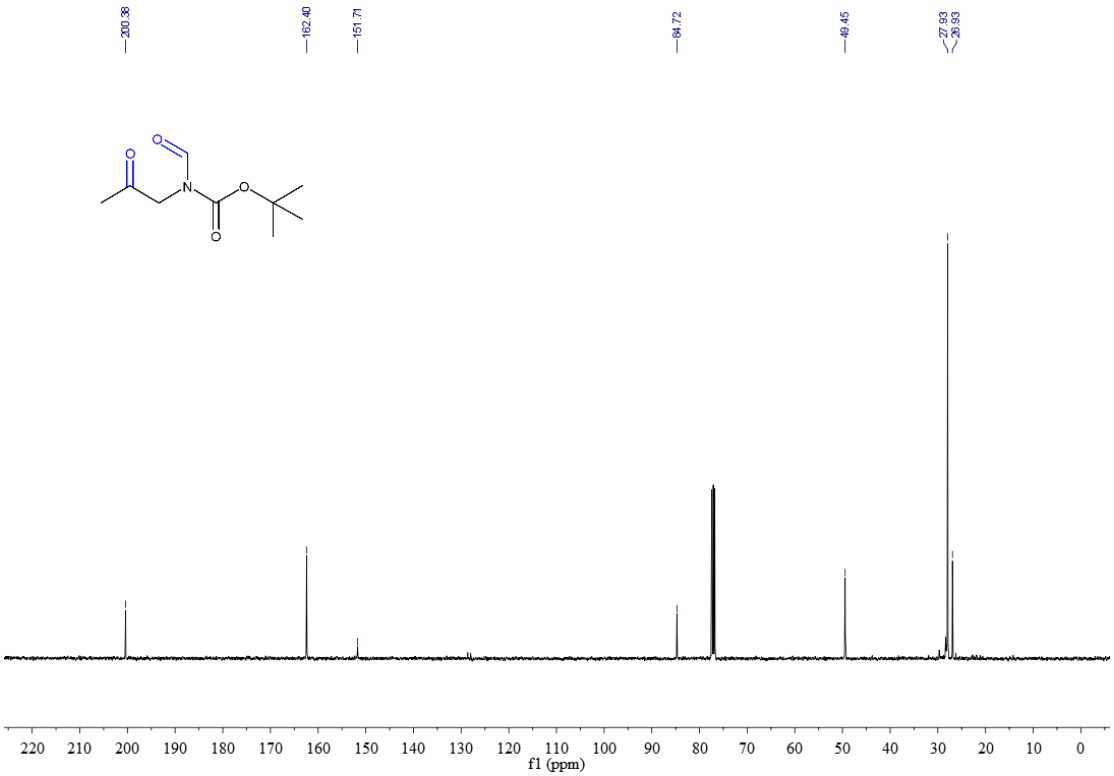
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2q**



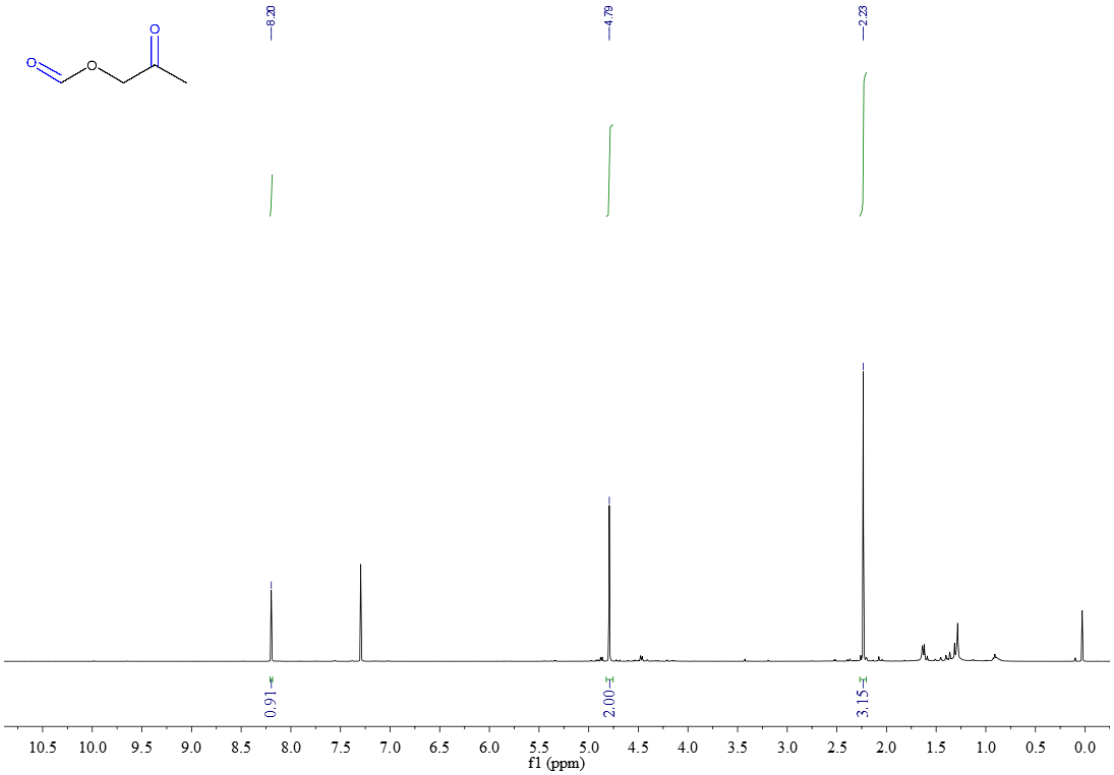
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2r**



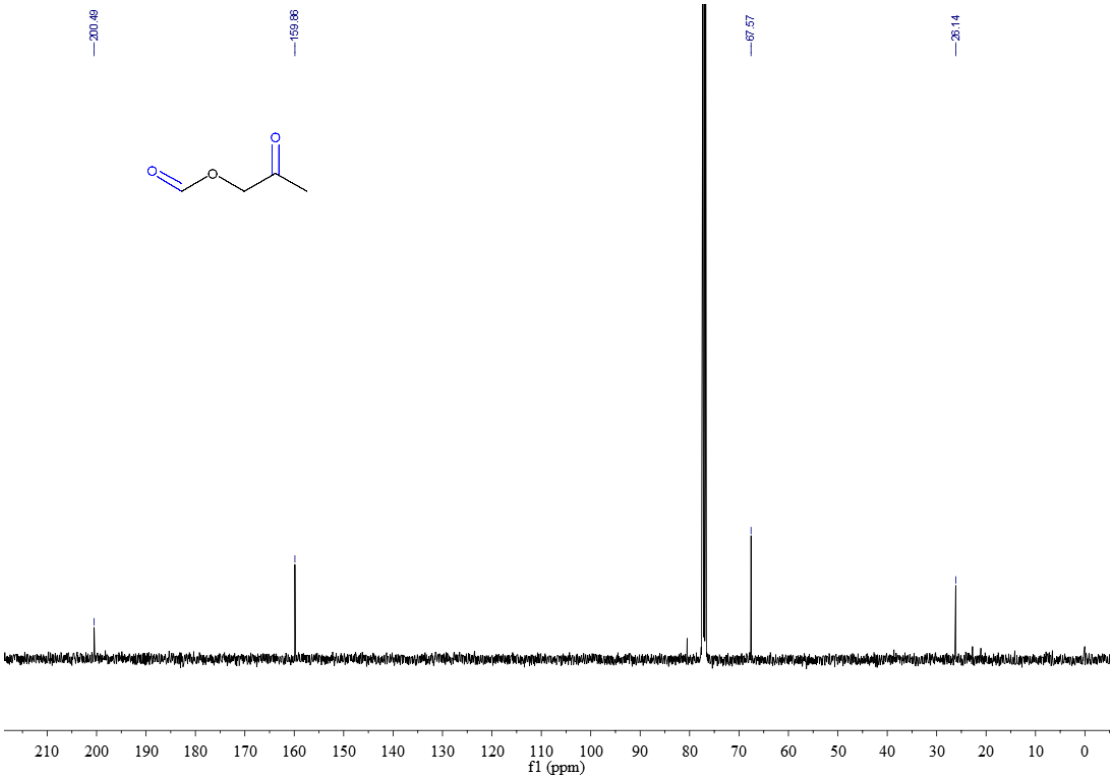
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2r**



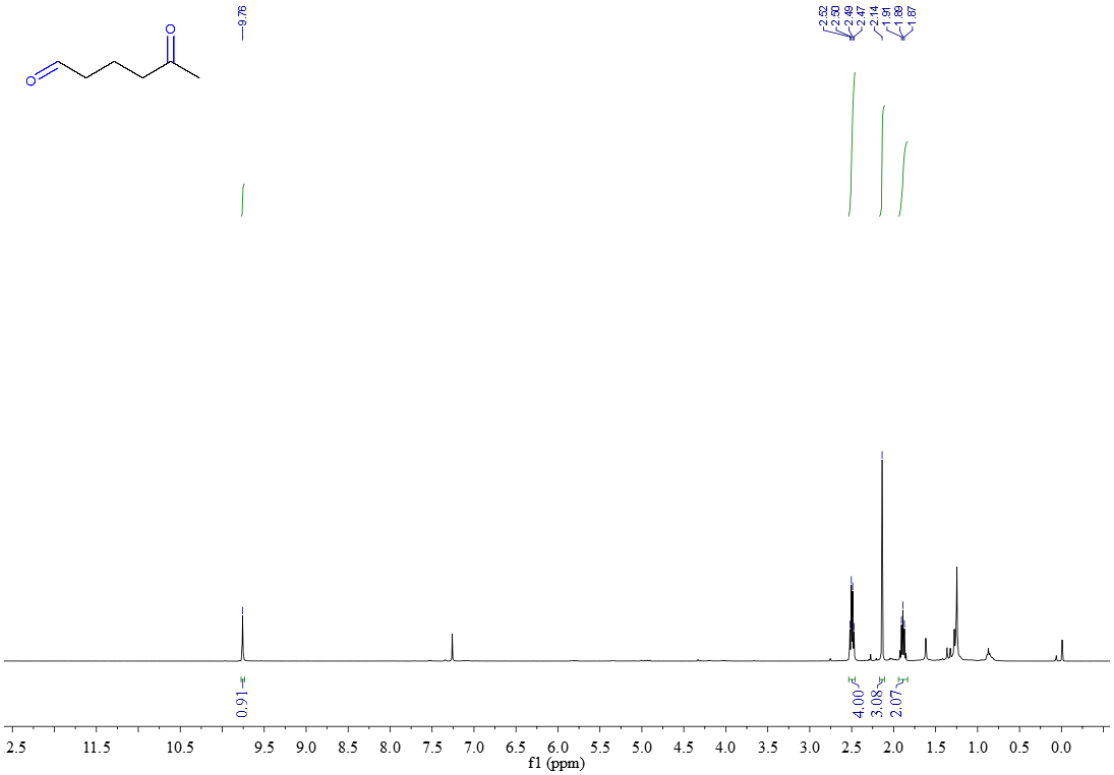
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2s**



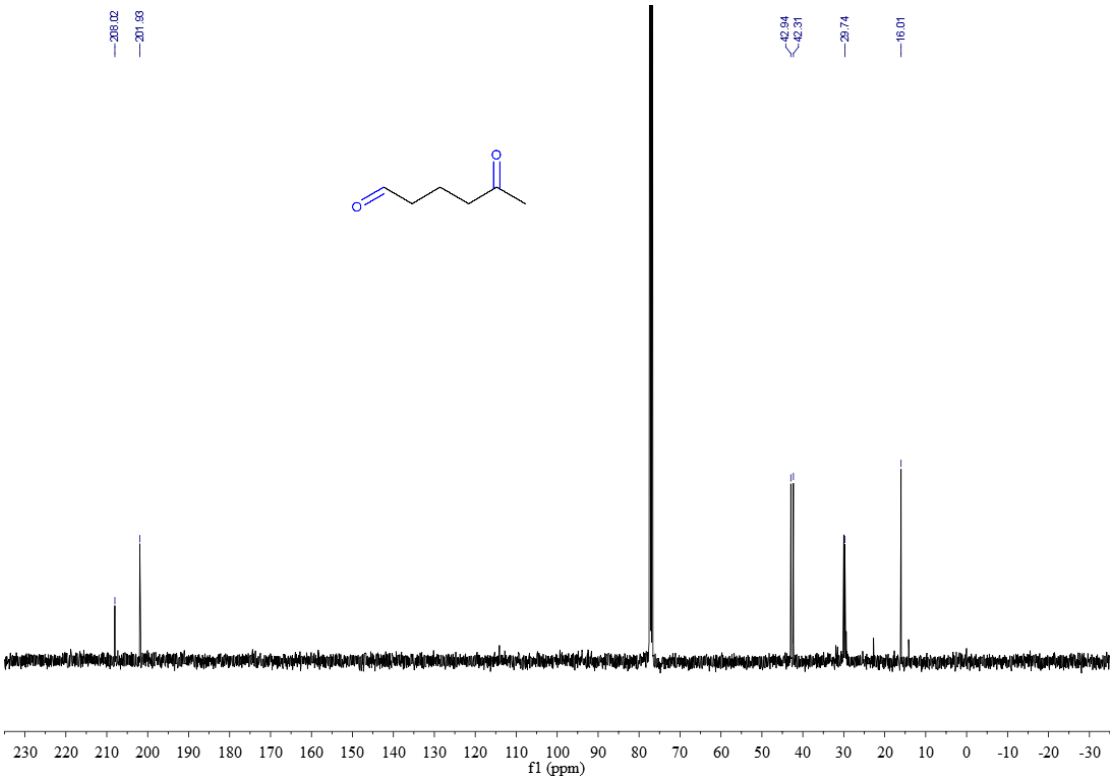
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2s**



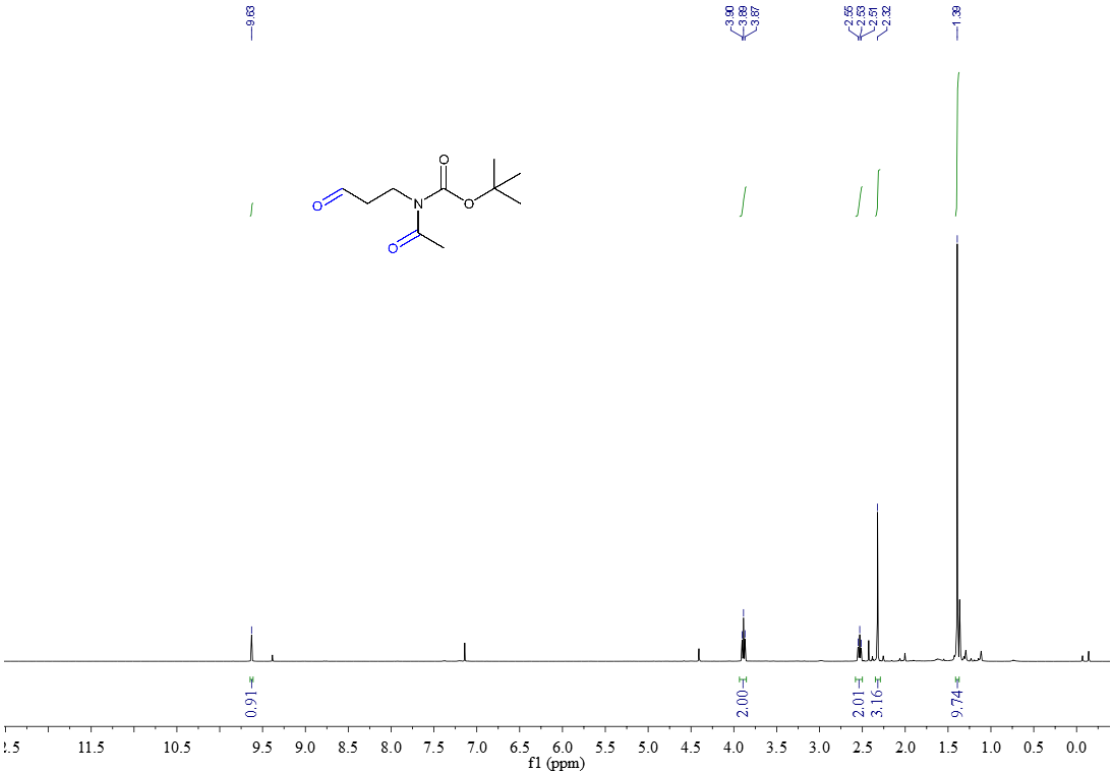
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2t**



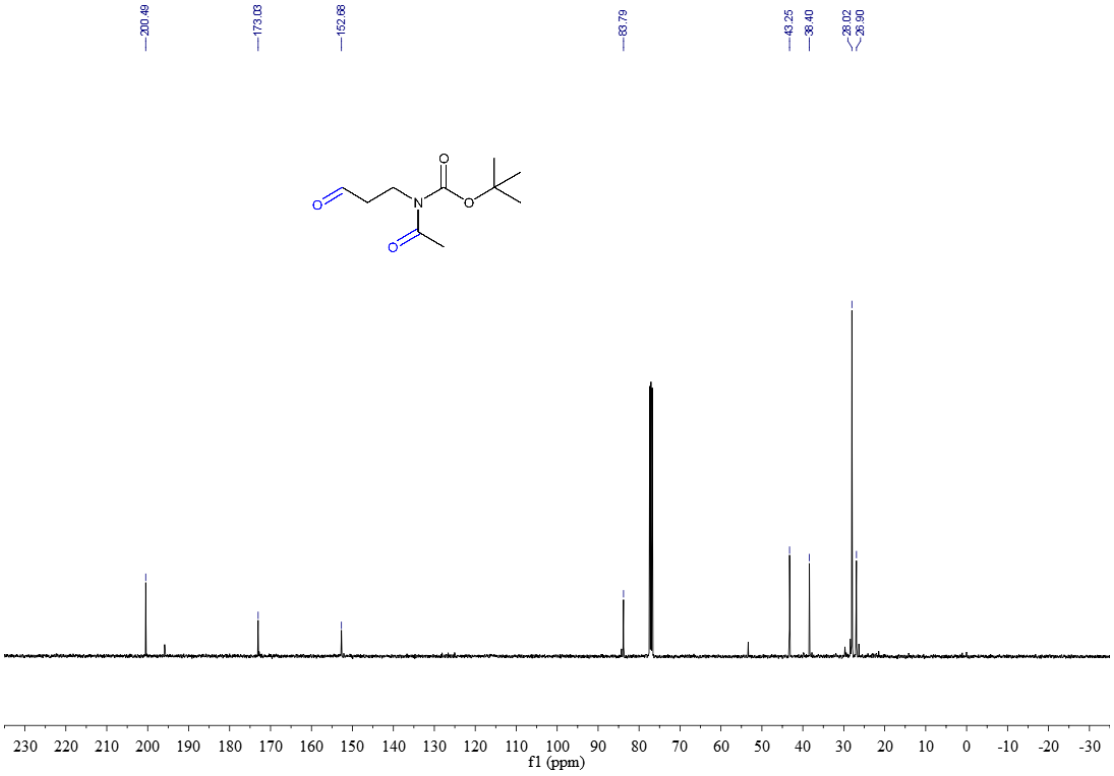
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2t**



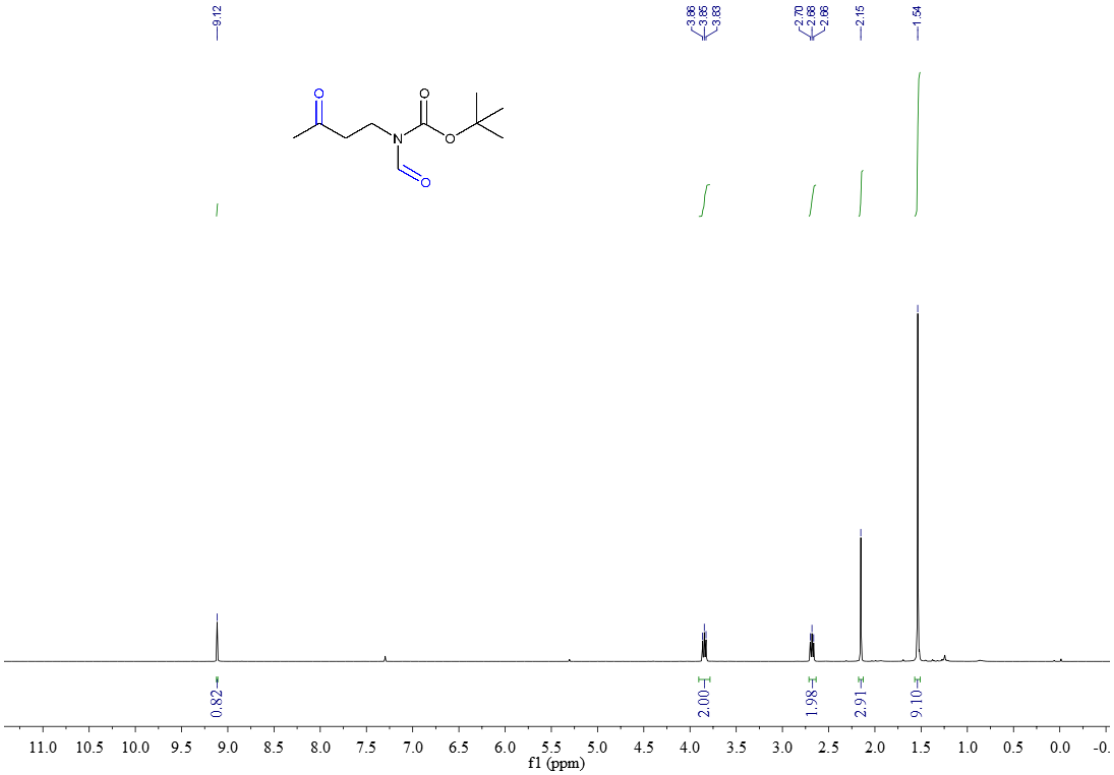
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2u**



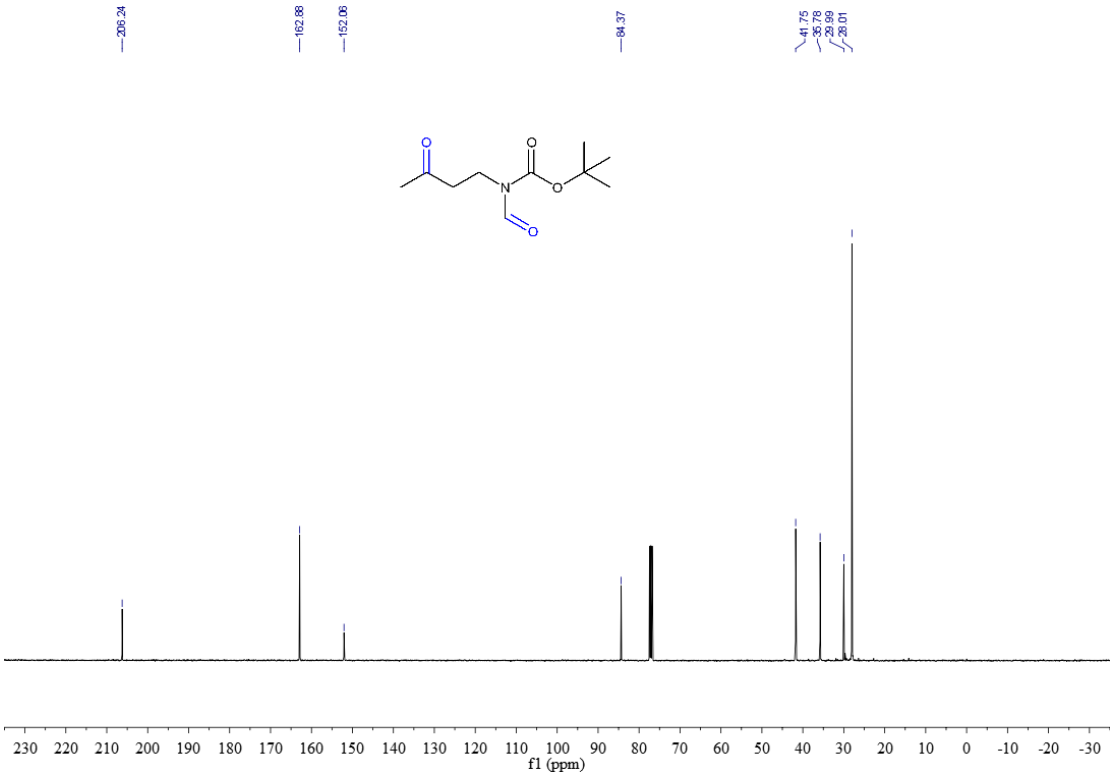
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2u**



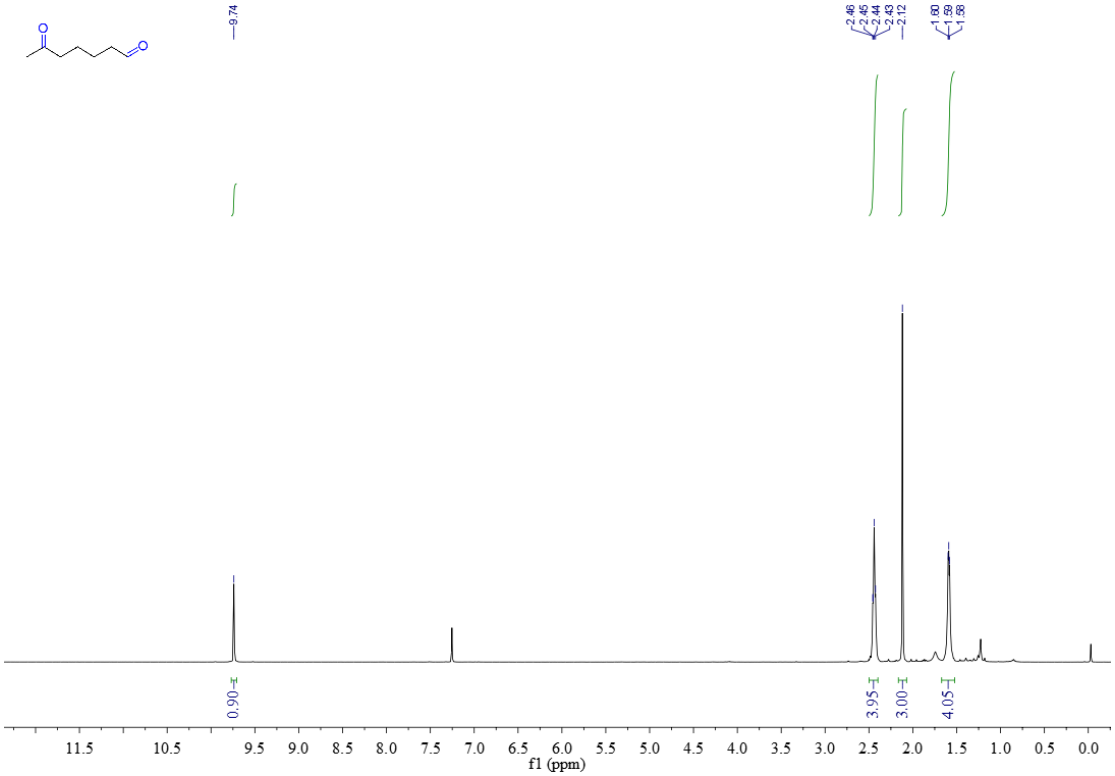
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2v**



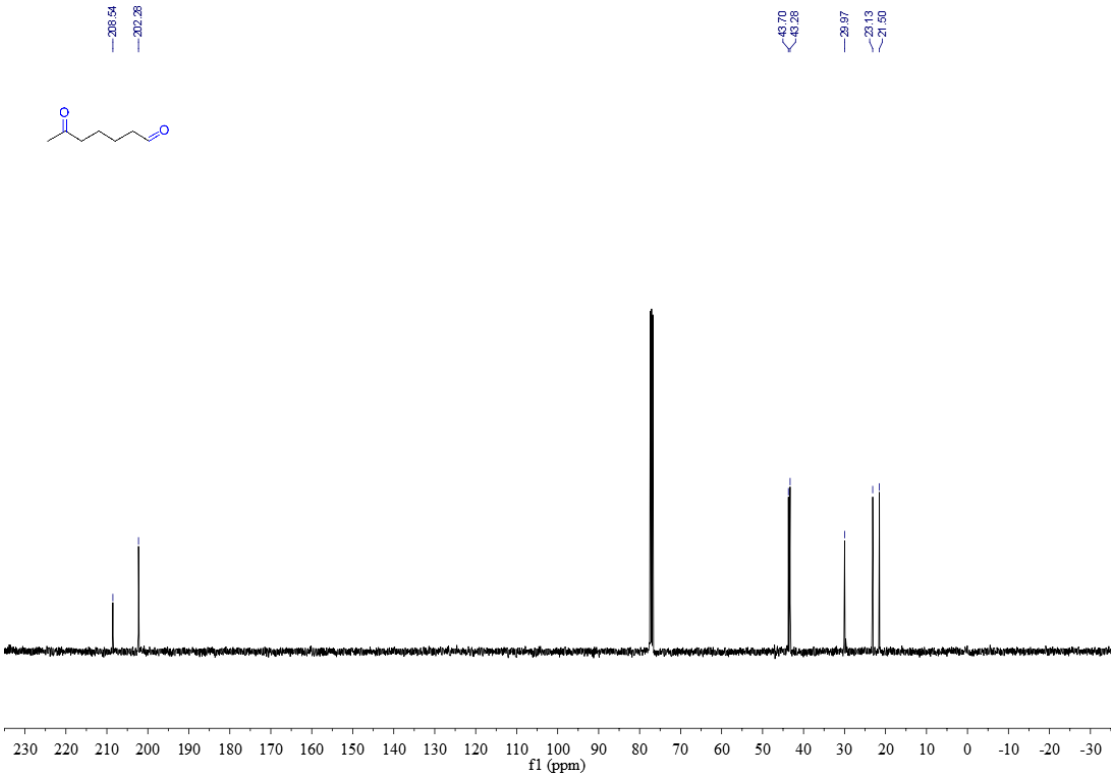
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2v**



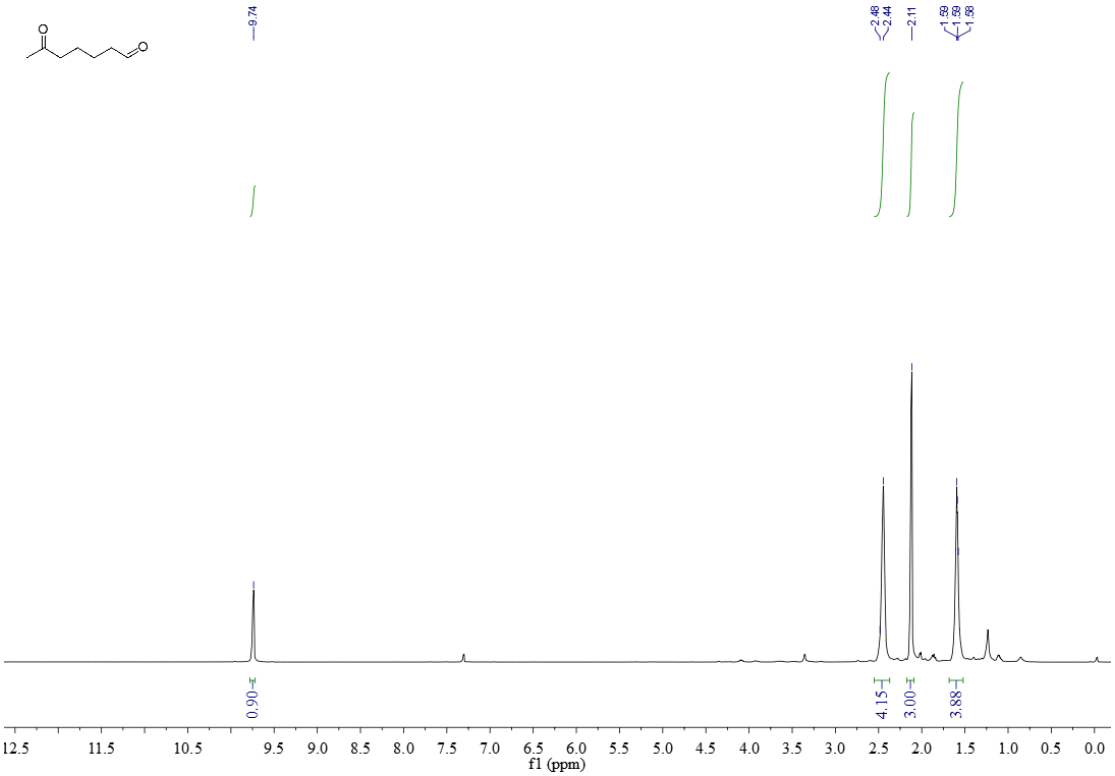
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2w**



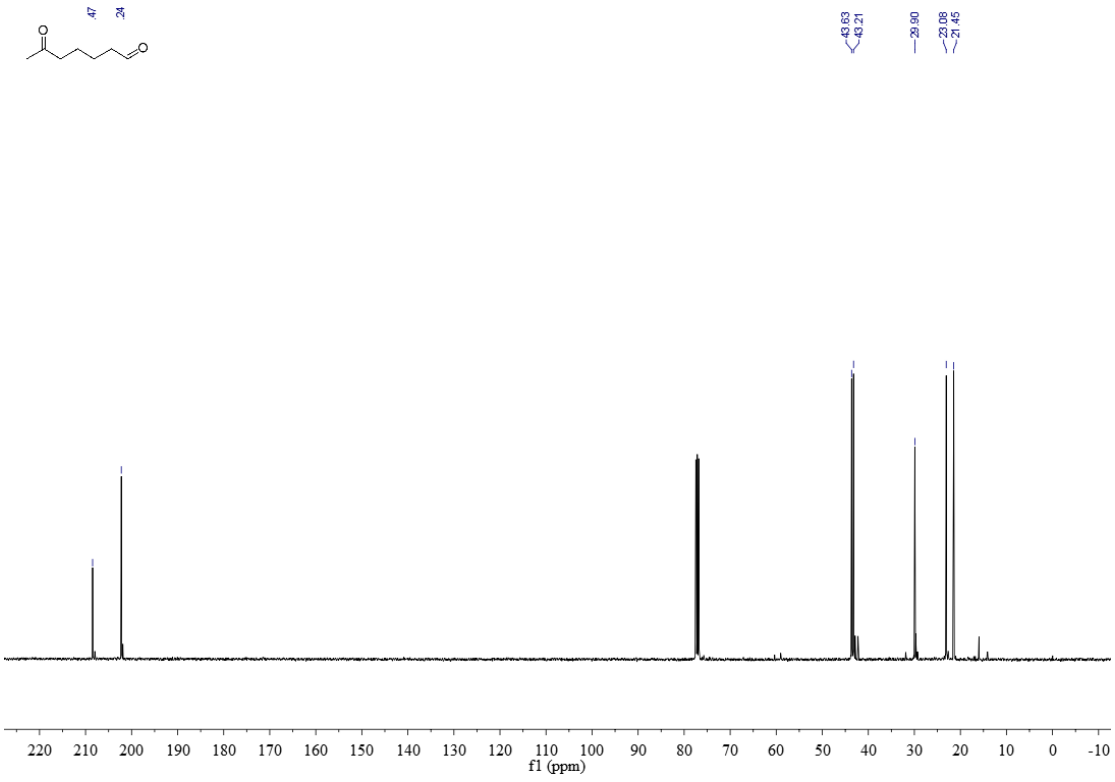
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2w**



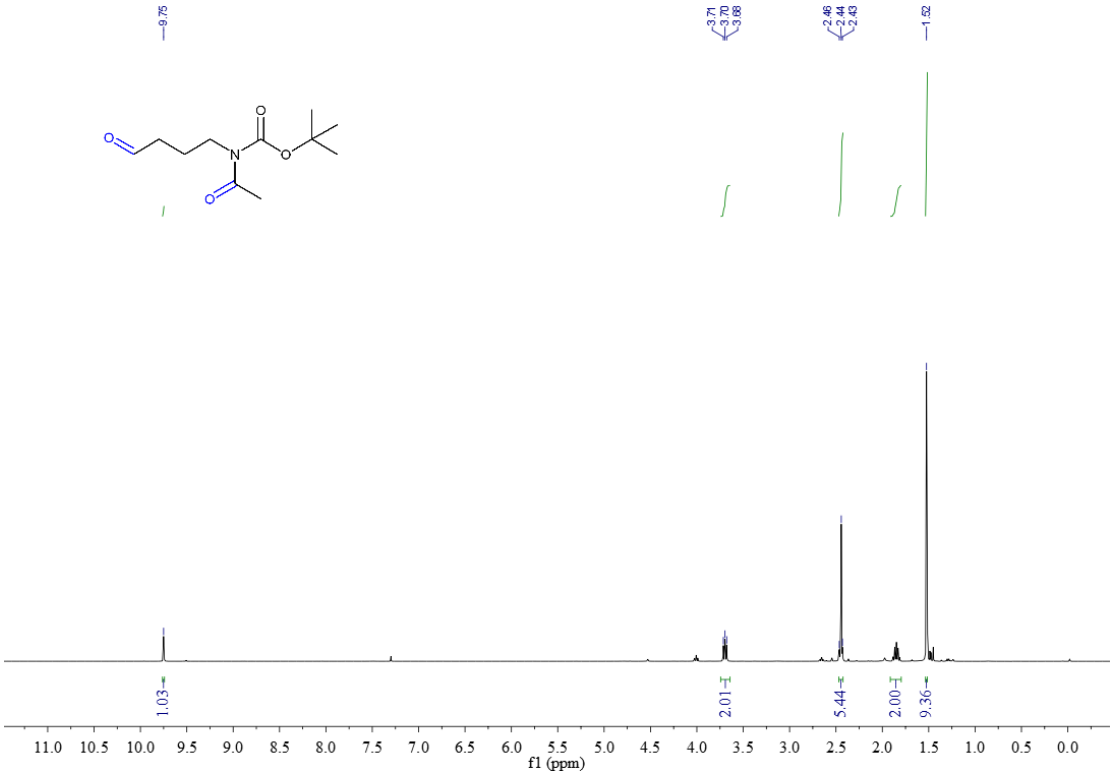
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2w** (for gram-scale reaction)



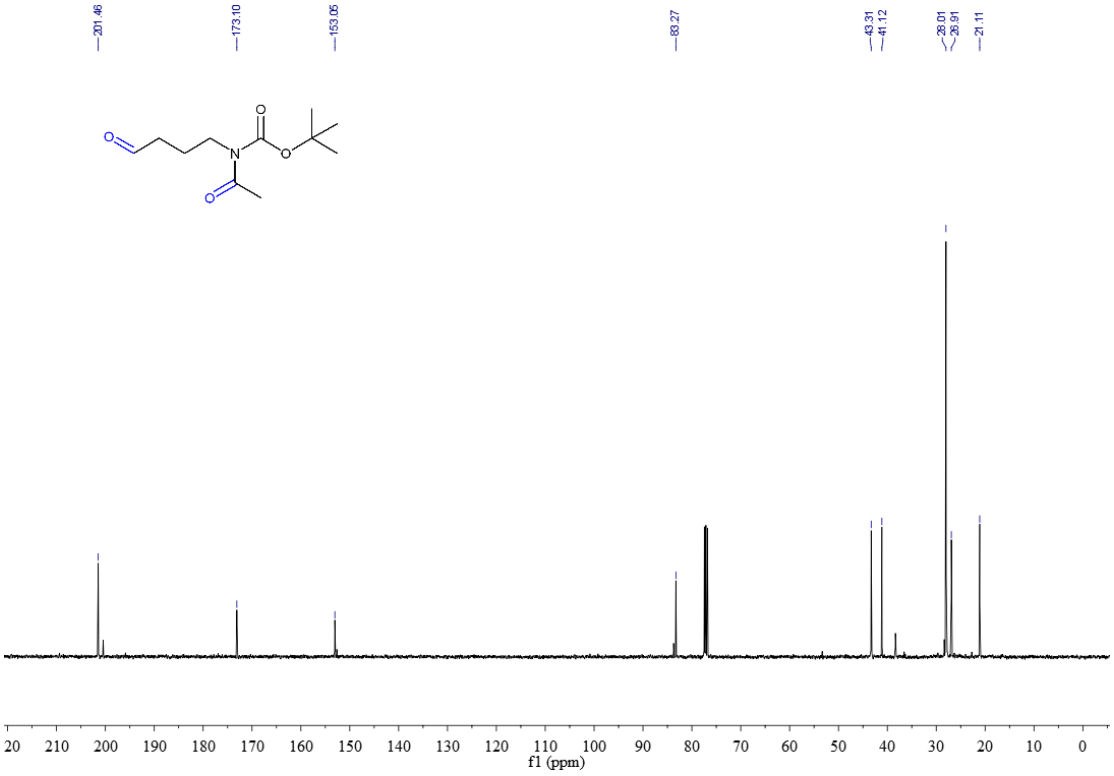
^{13}C NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2w** (for gram-scale reaction)



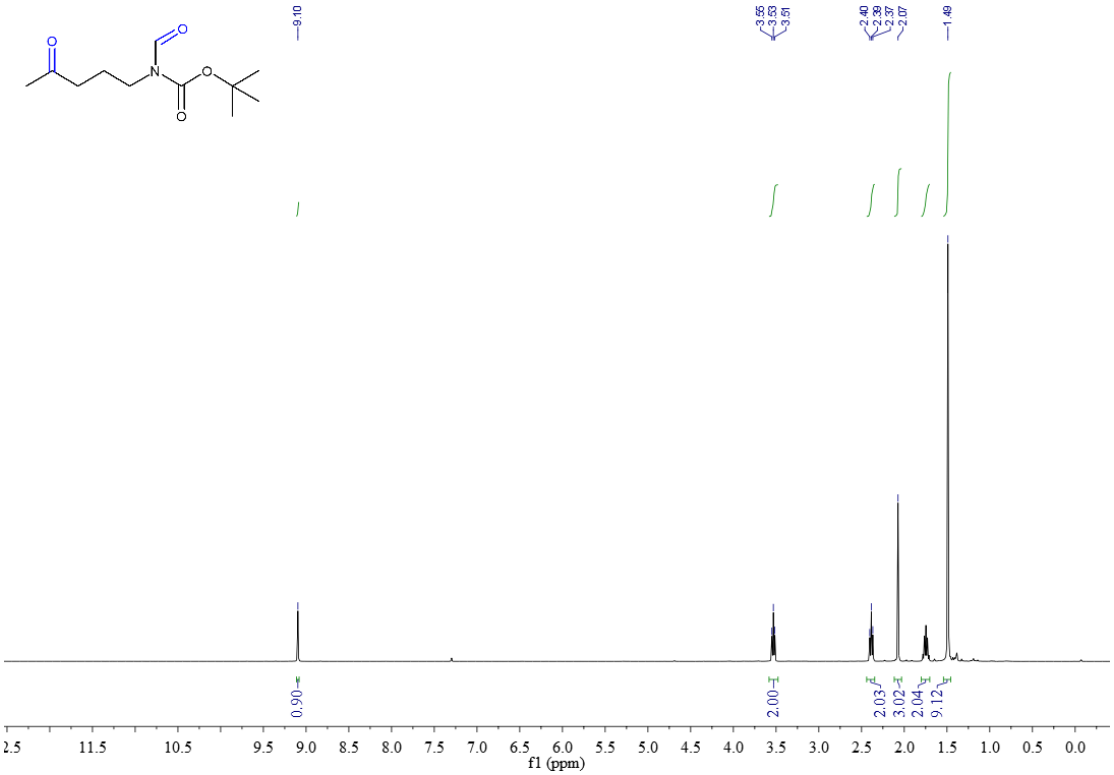
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2x**



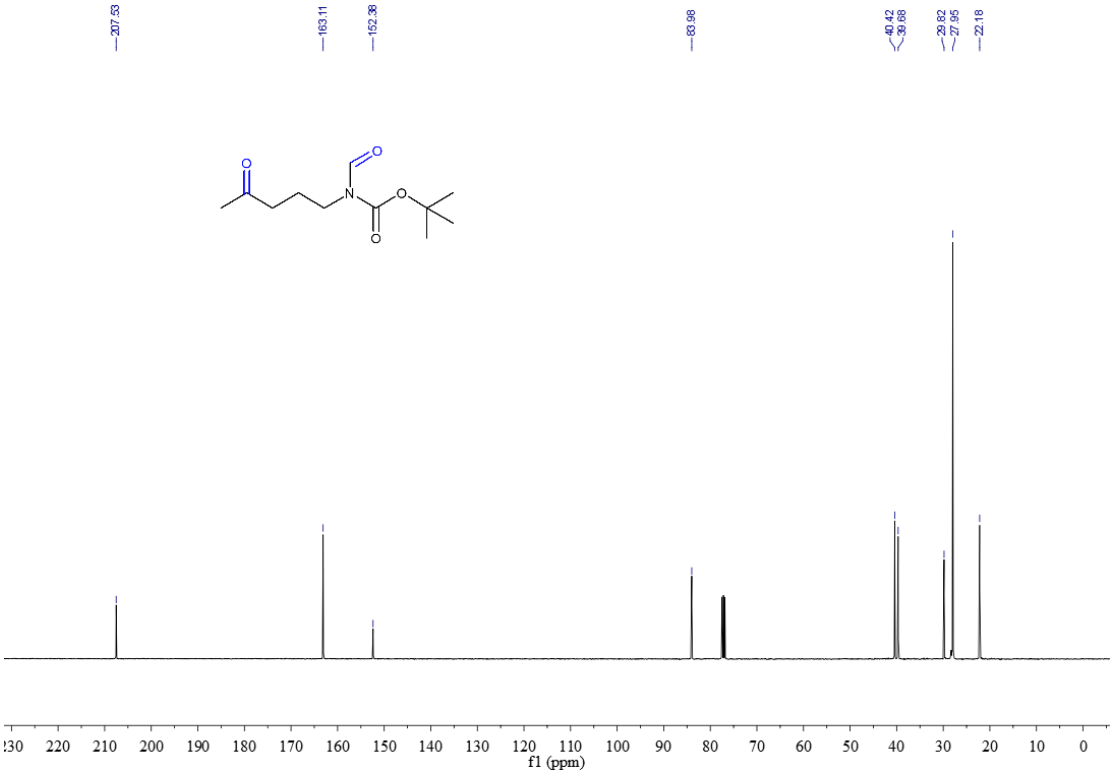
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2x**



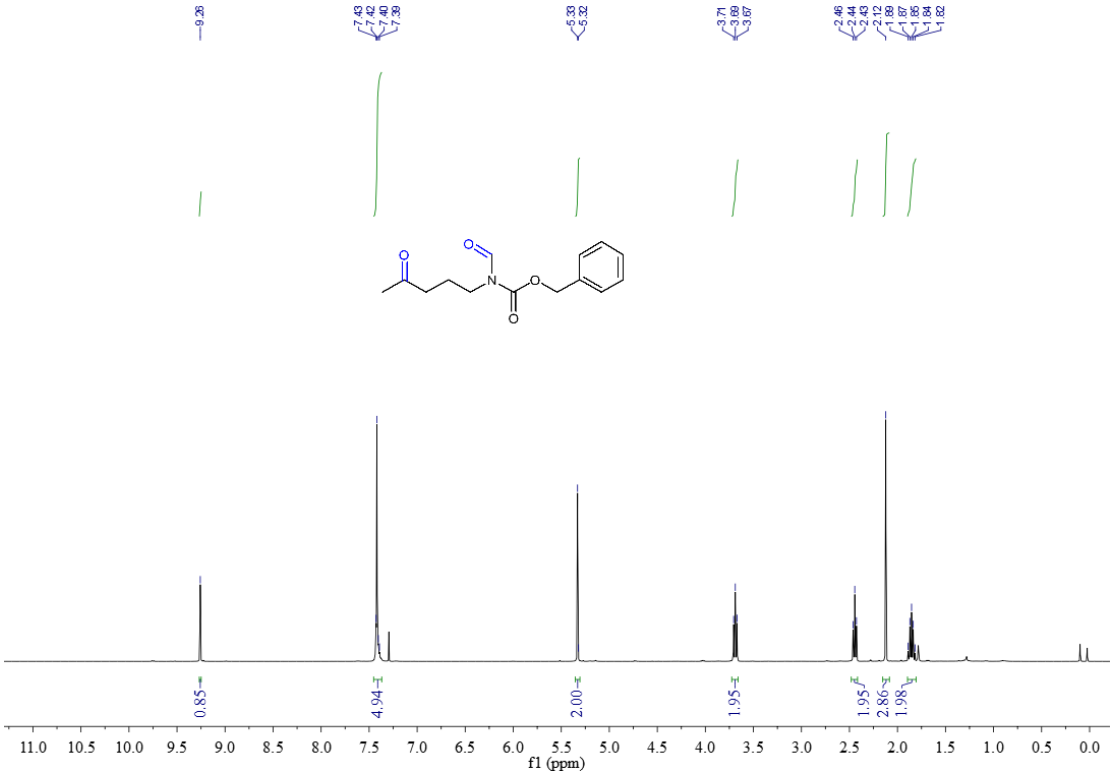
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2y**



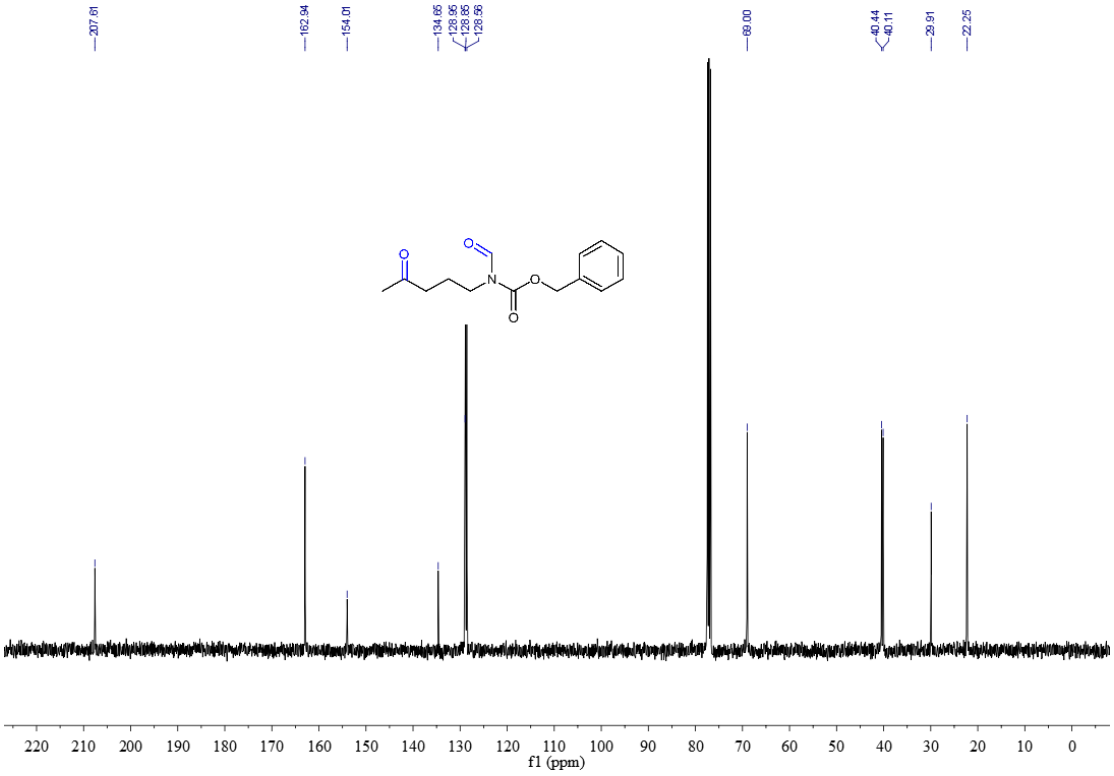
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2y**



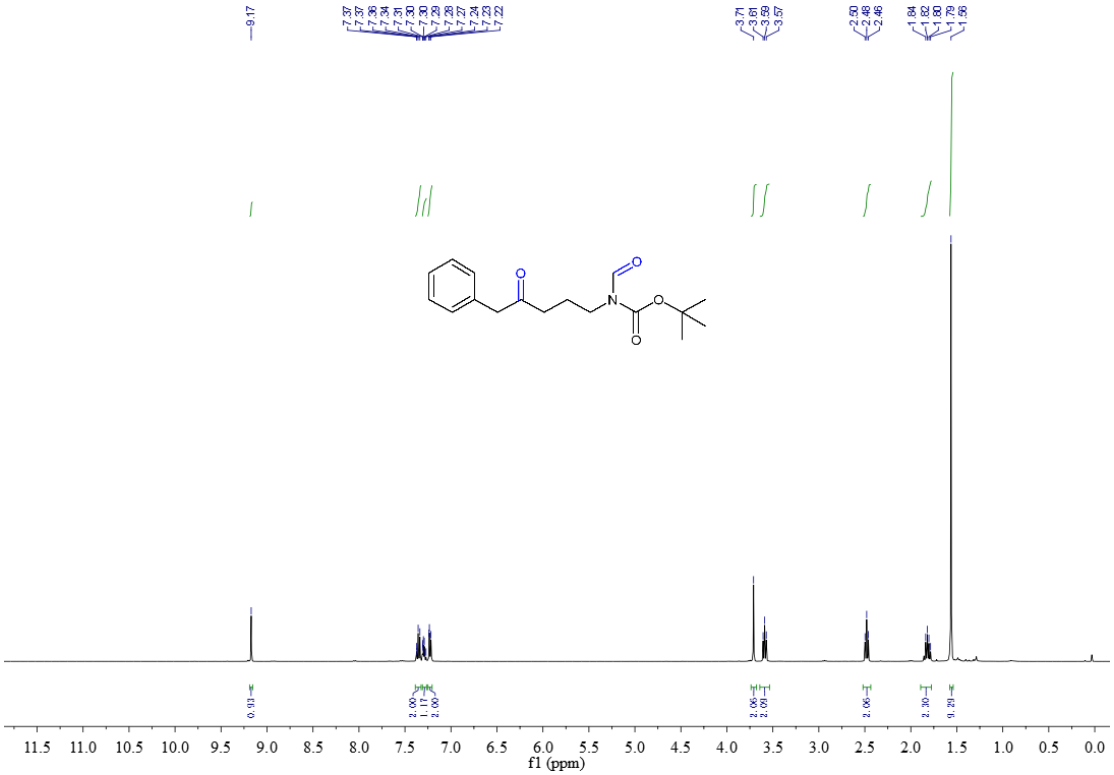
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2z**



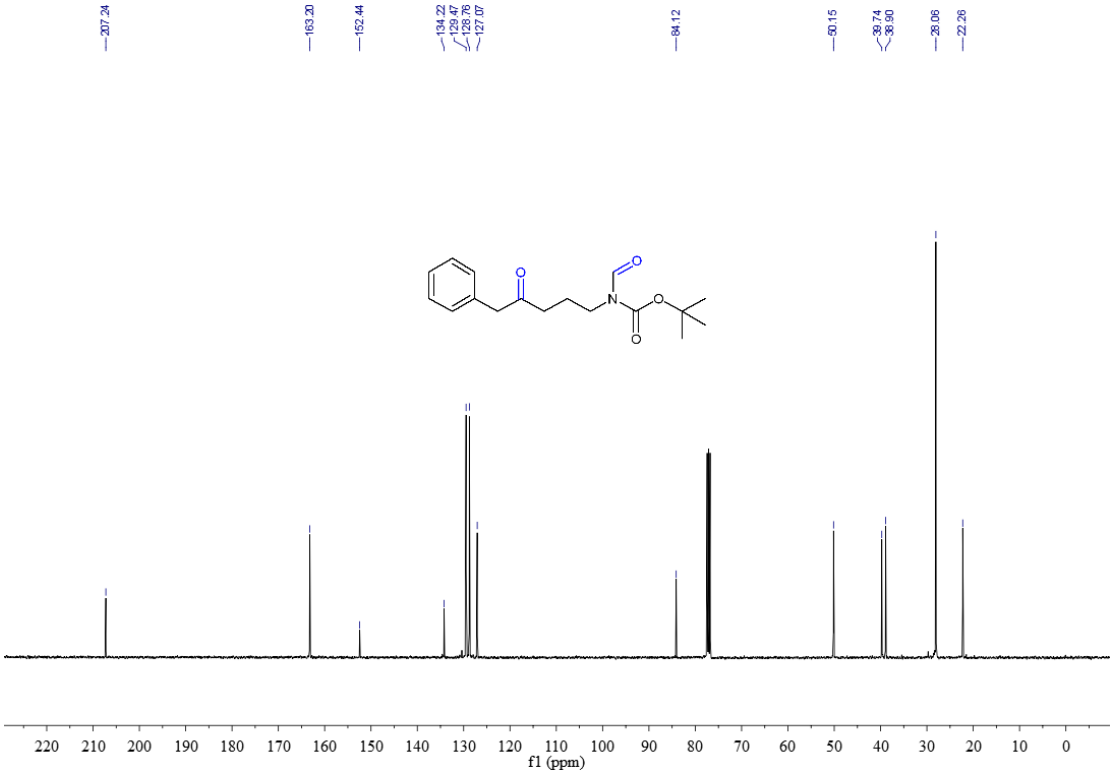
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2z**



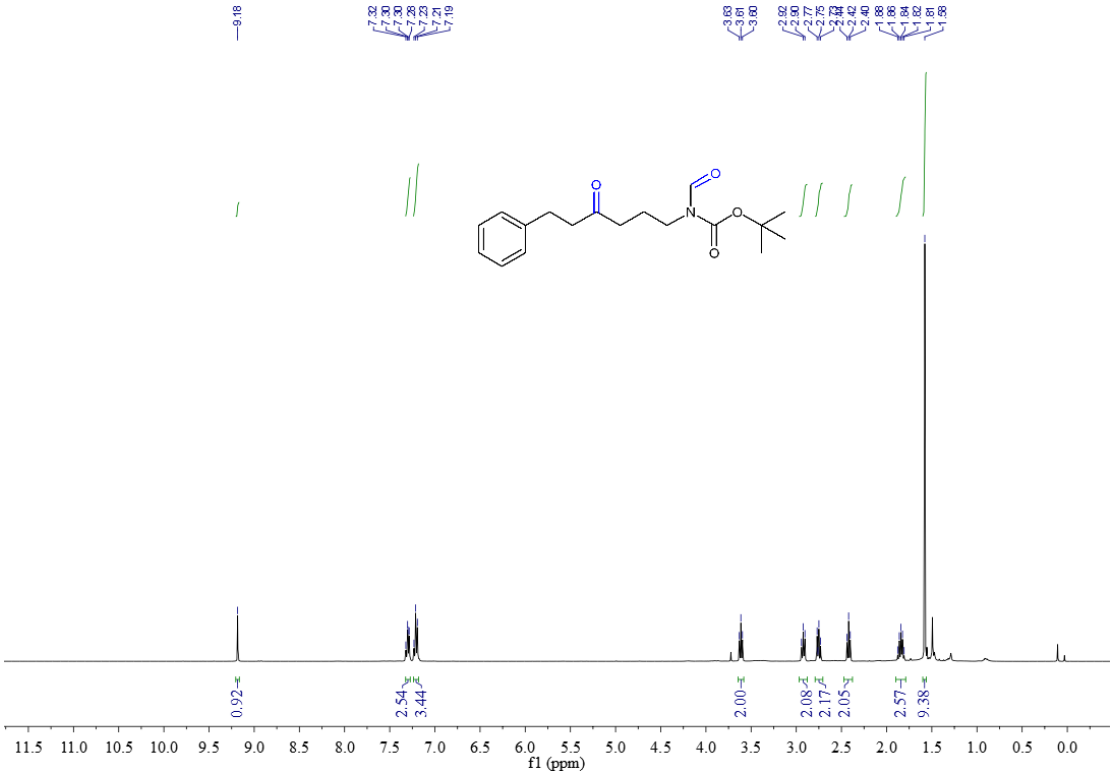
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2aa**



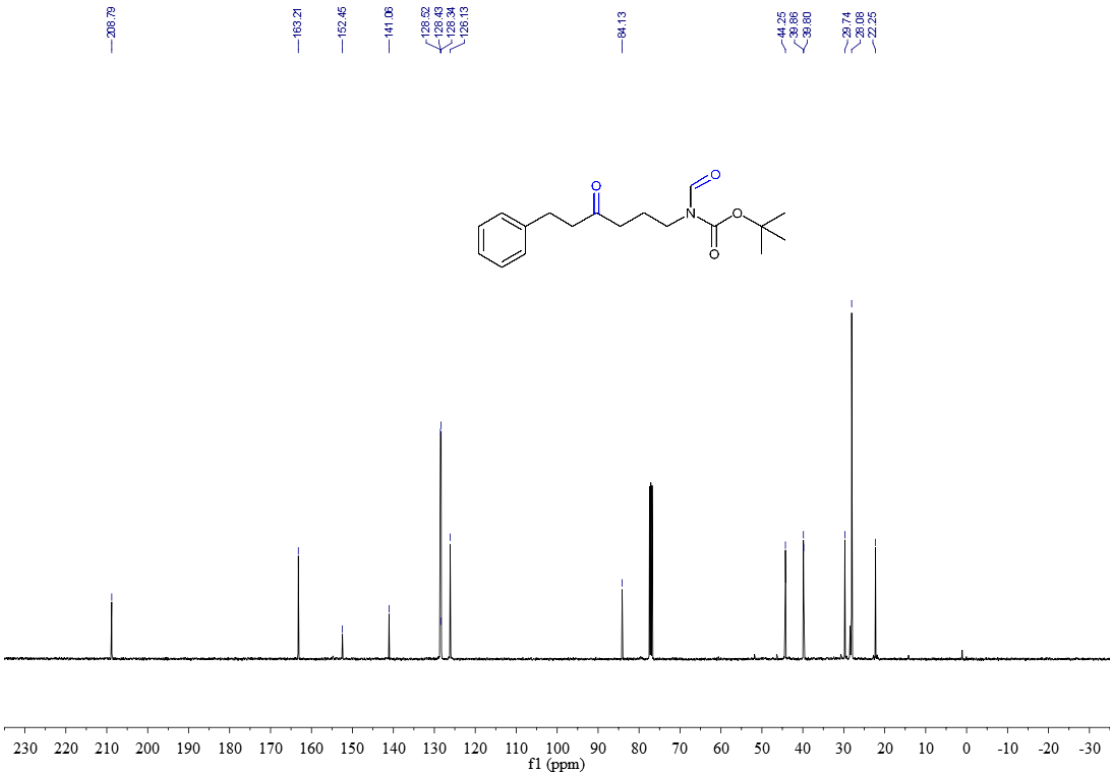
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2aa**



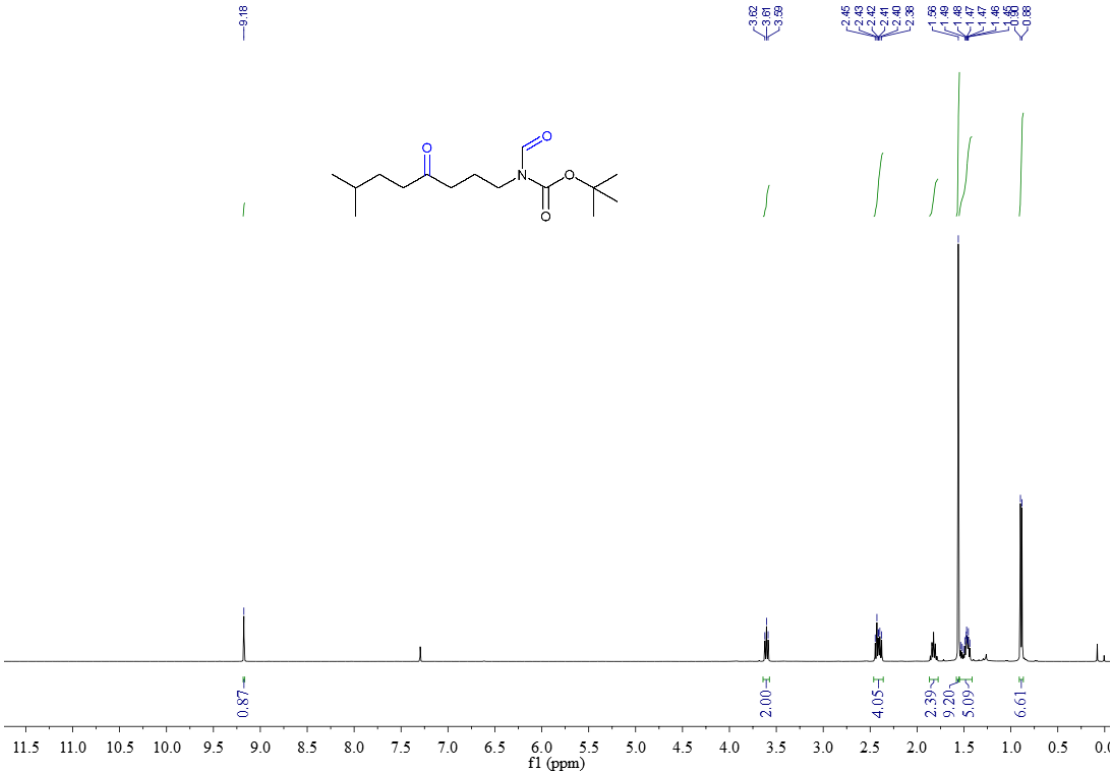
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2bb**



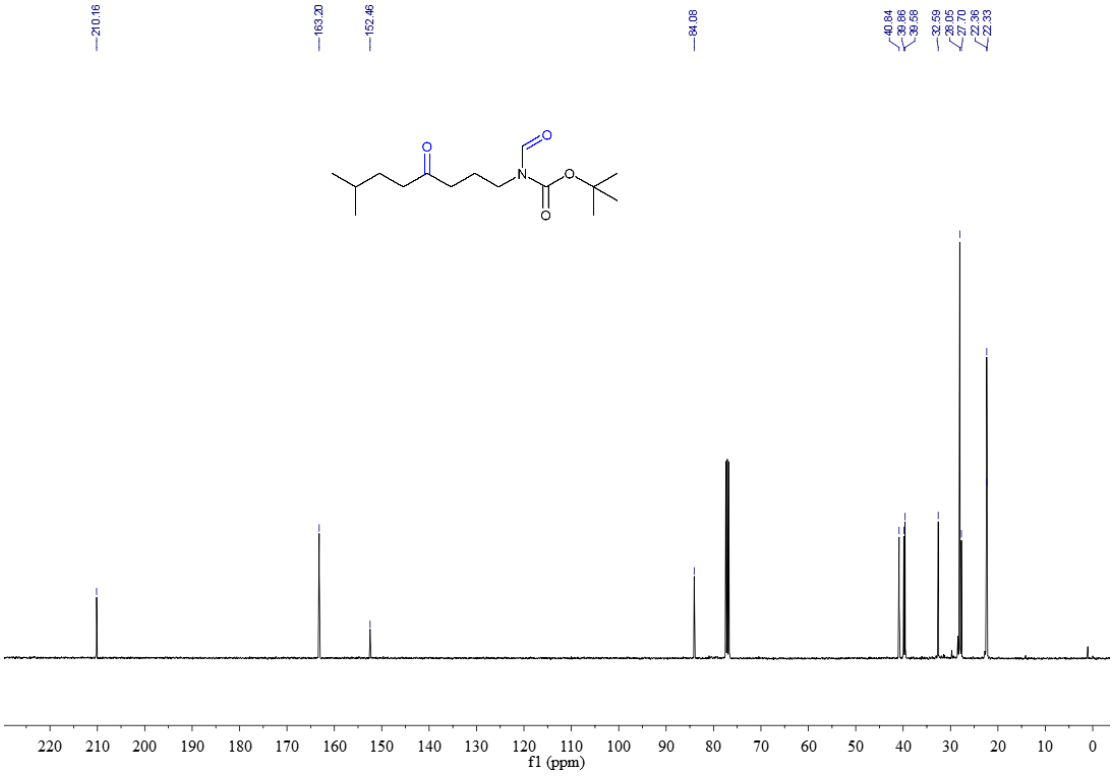
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2bb**



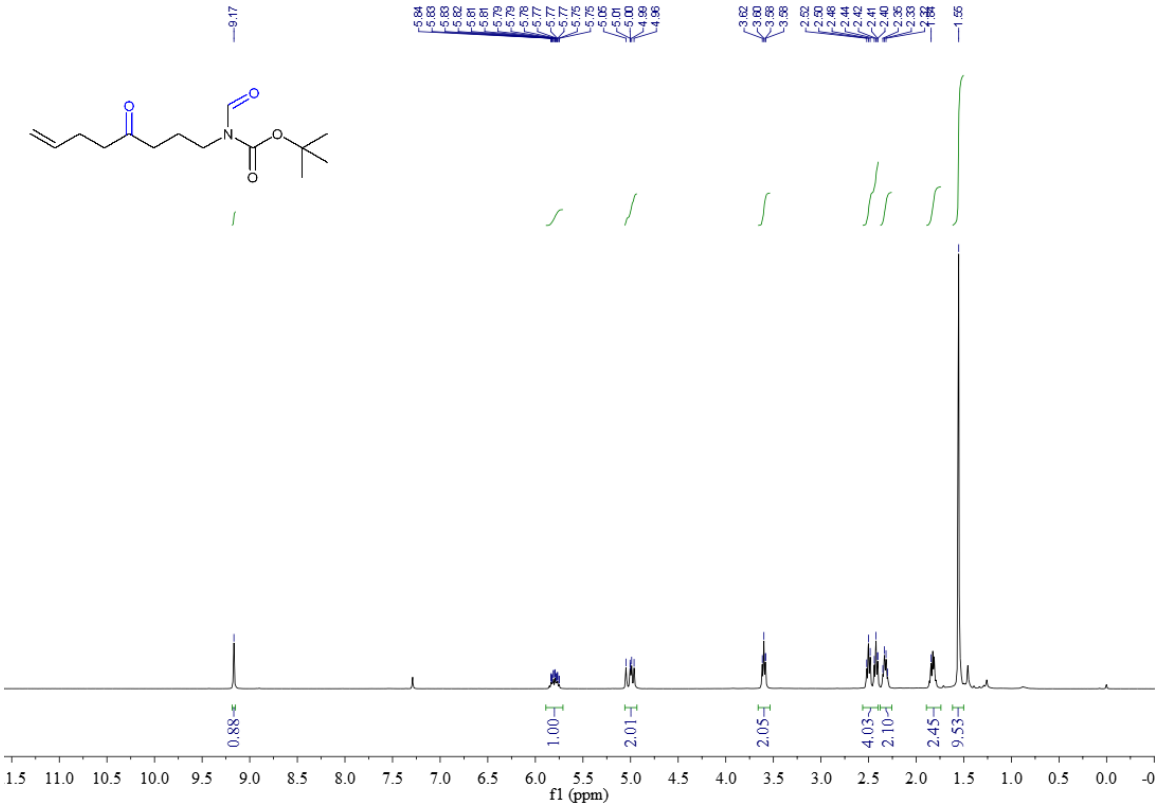
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2cc**



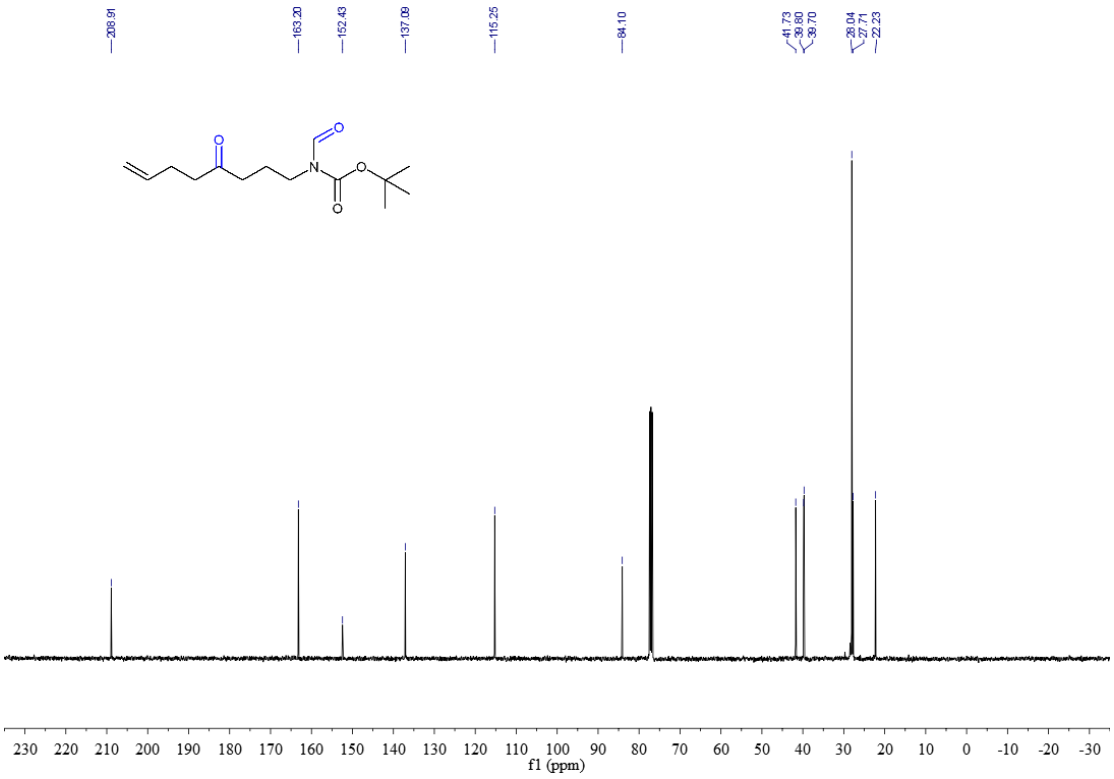
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2cc**



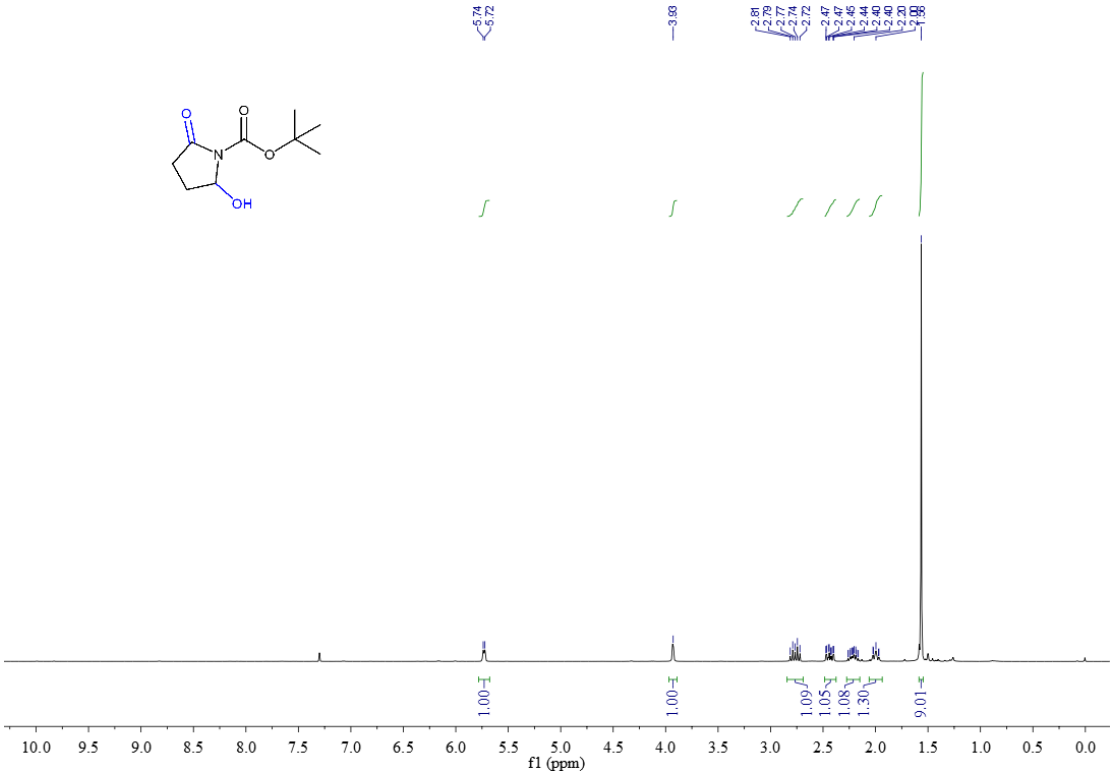
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2dd**



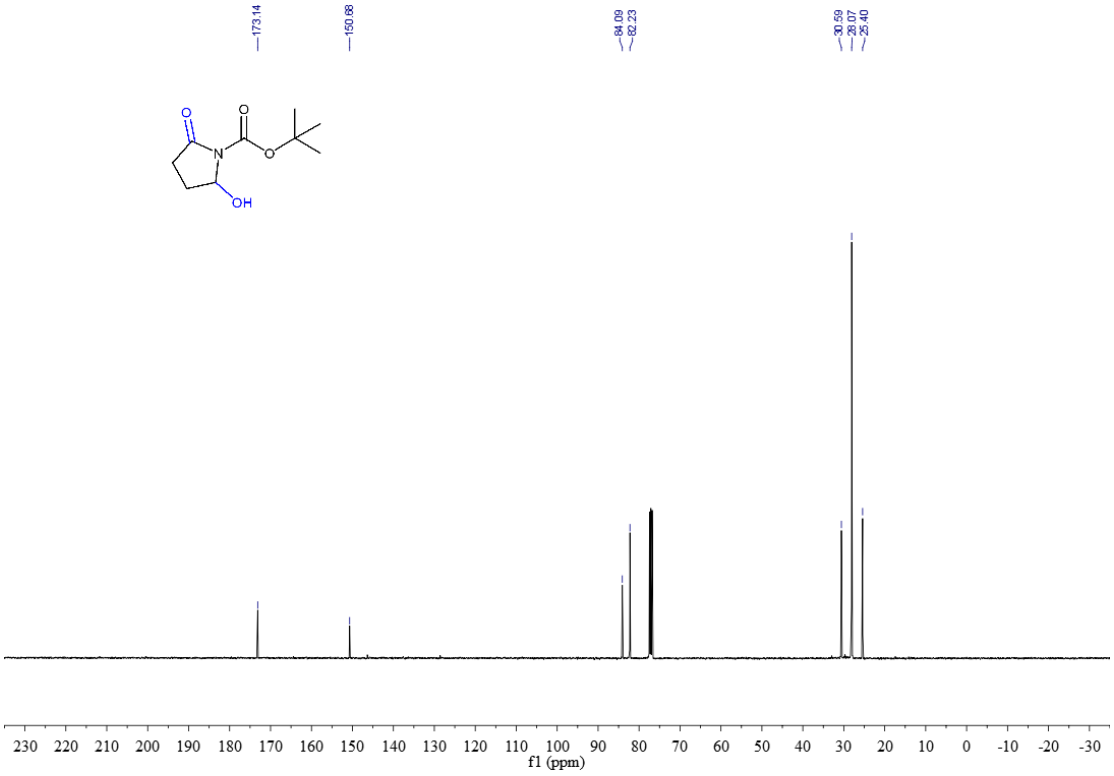
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2dd**



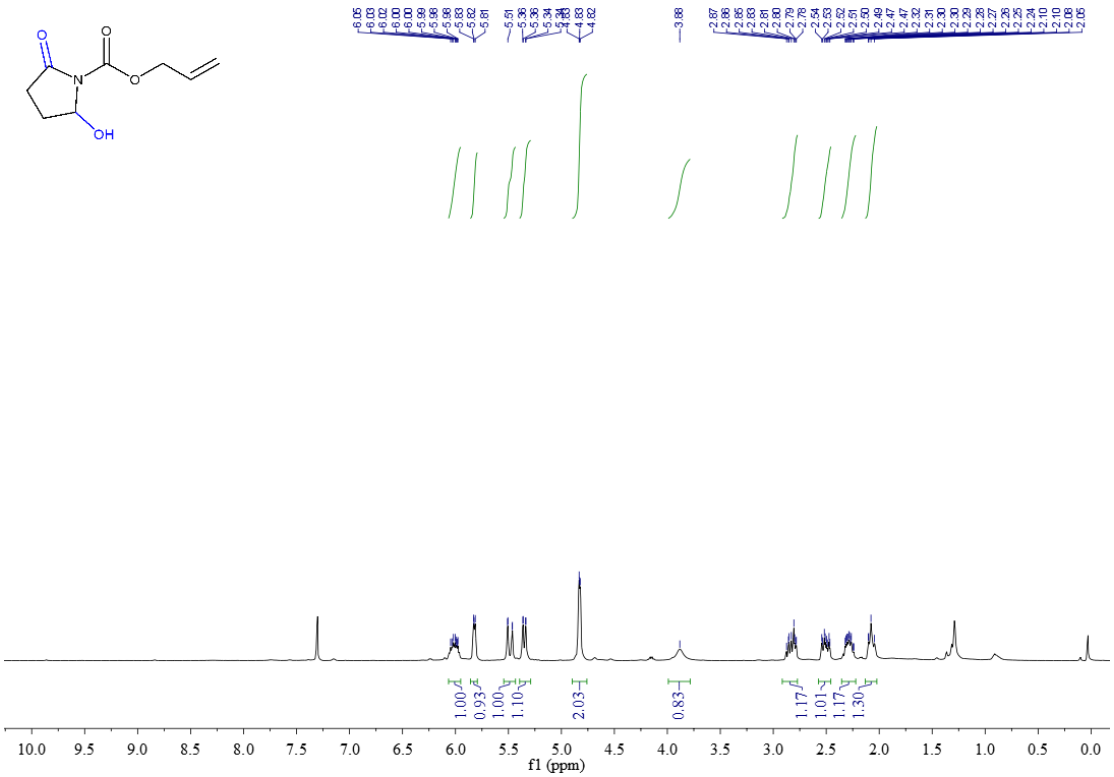
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2ee**



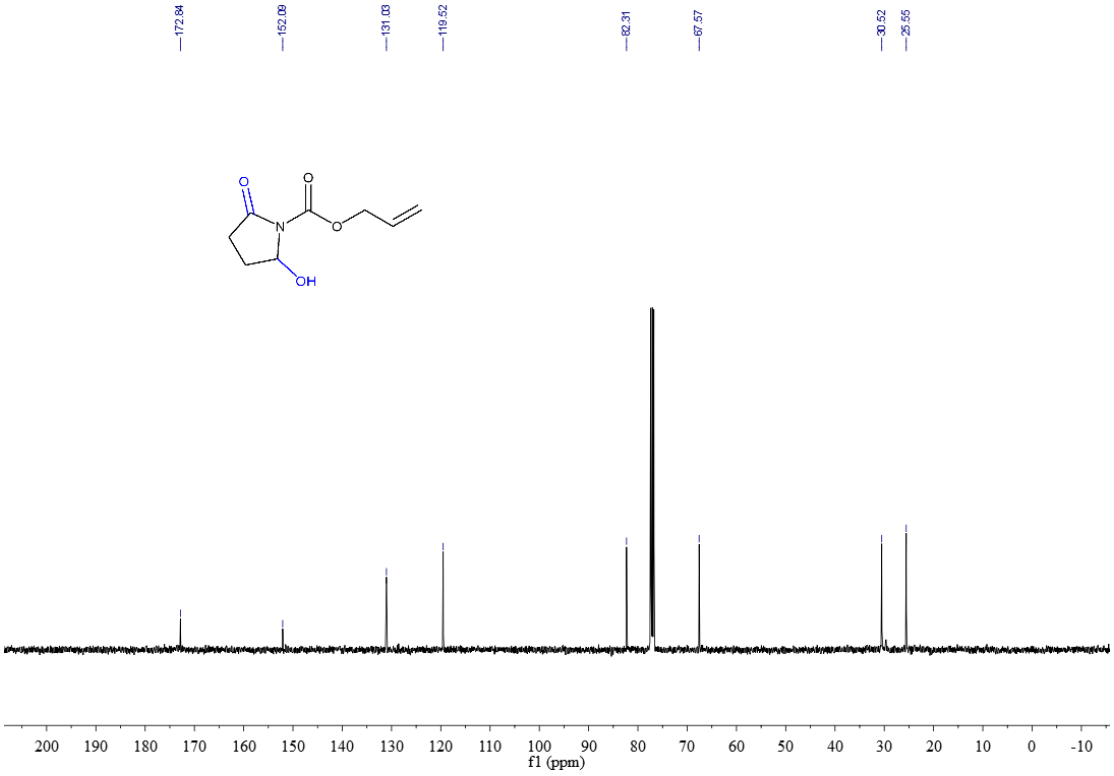
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2ee**



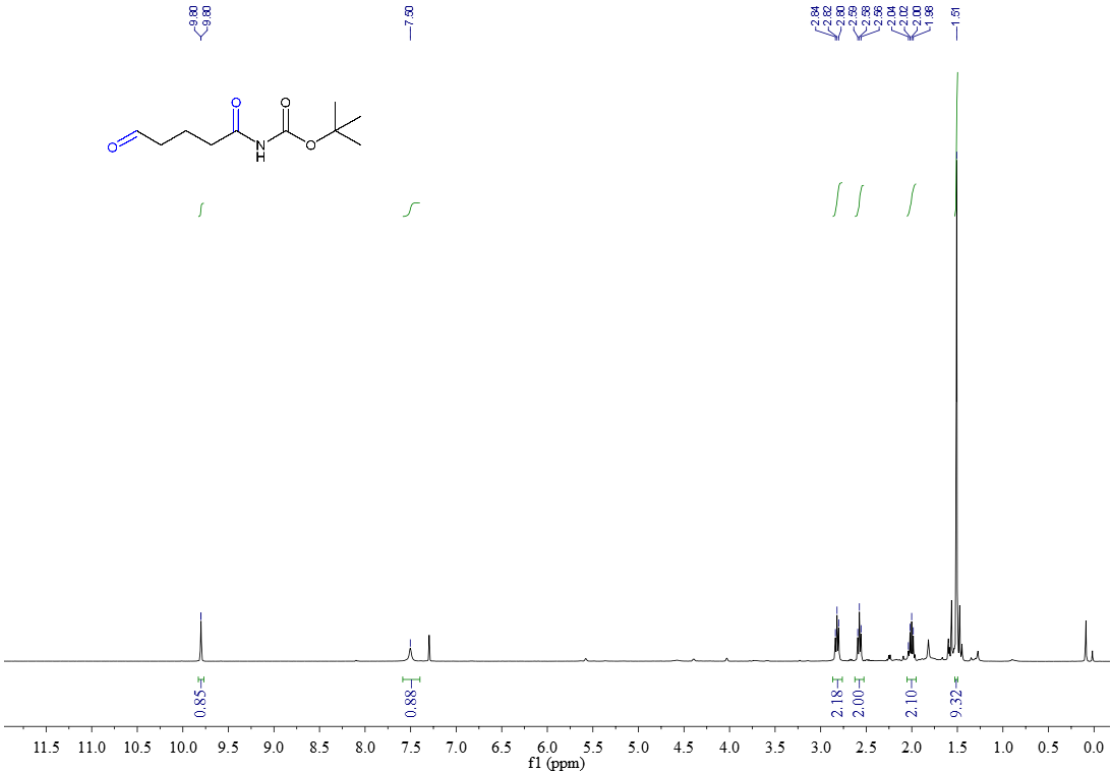
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2ff**



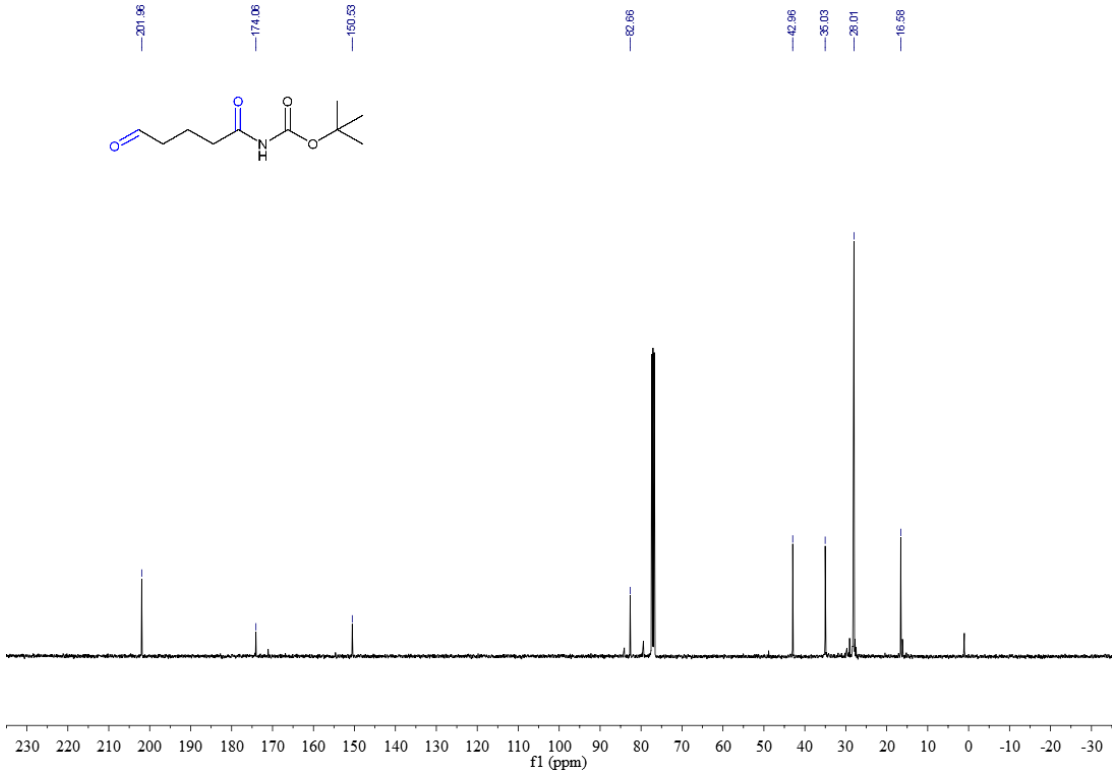
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2ff**



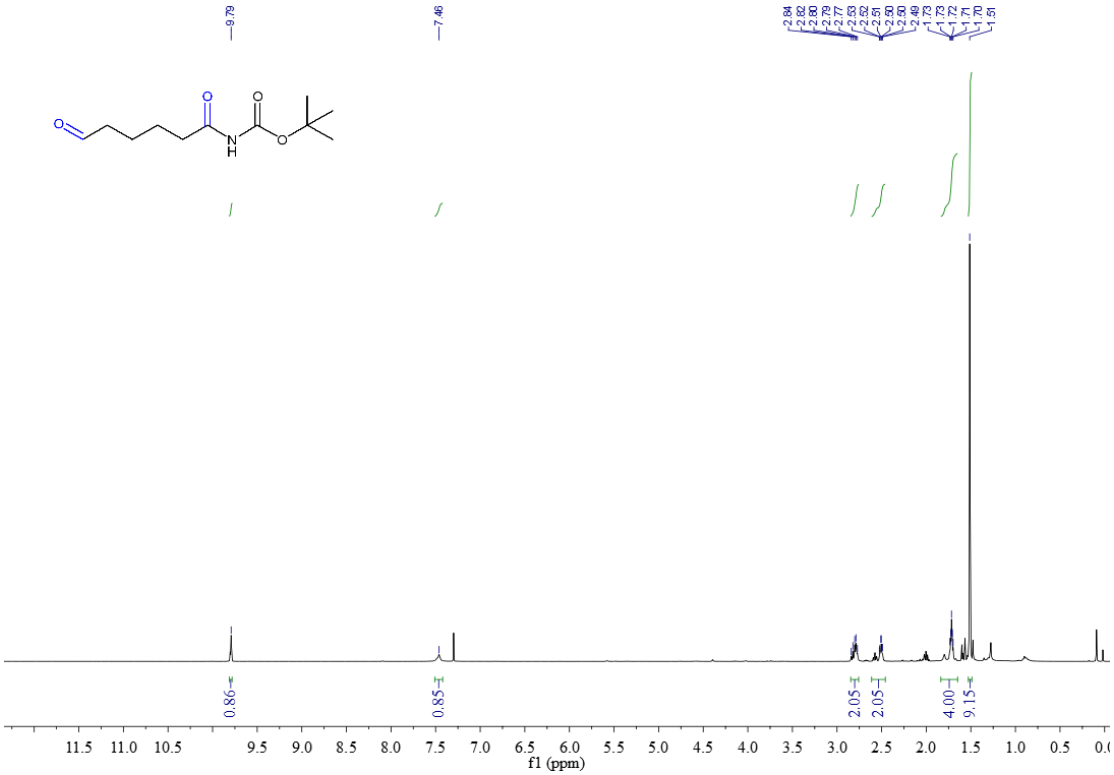
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2gg**



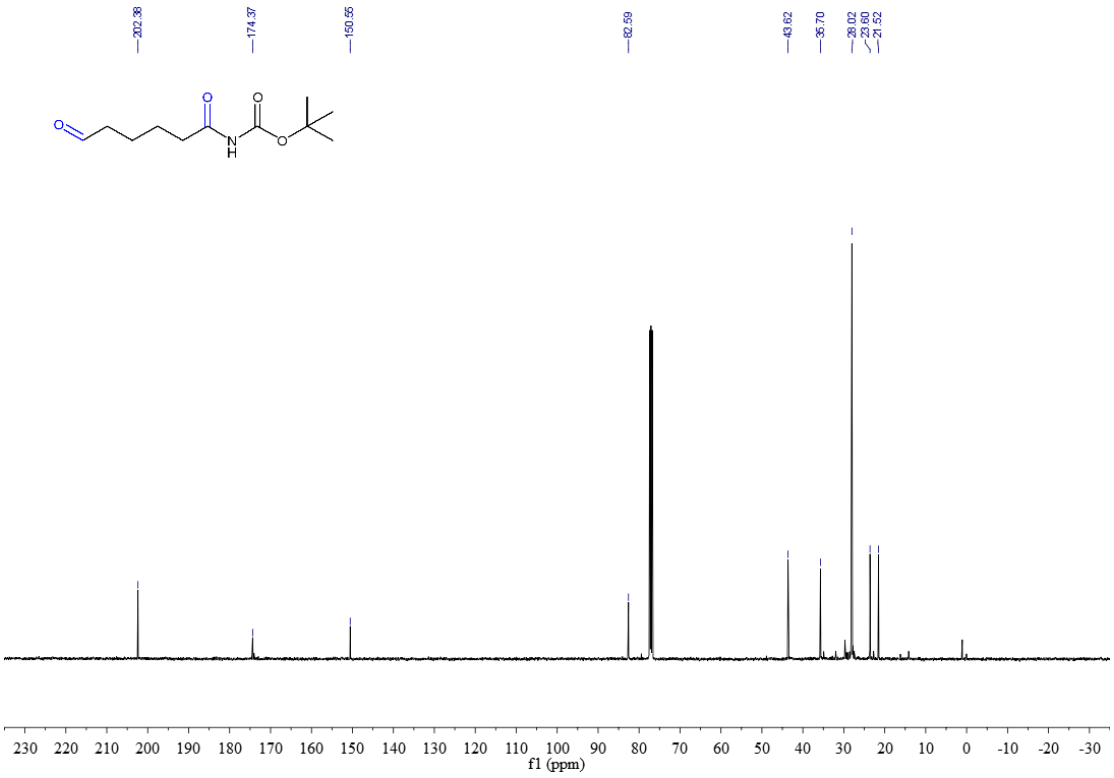
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2gg**



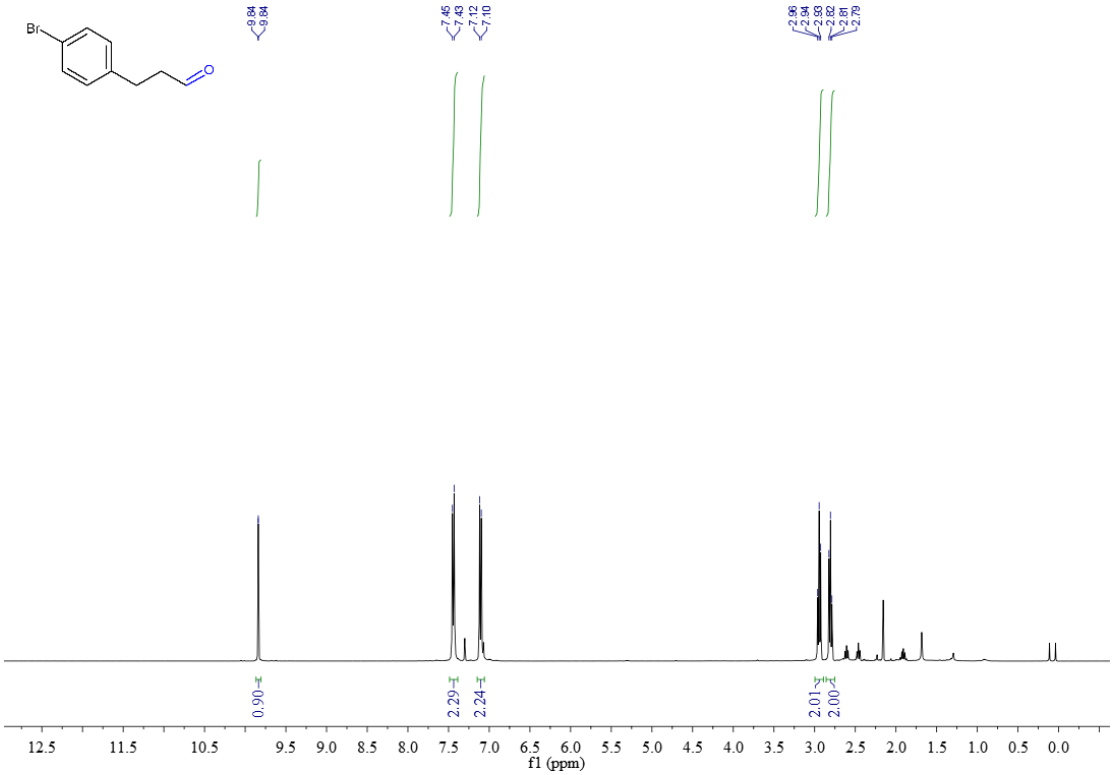
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **2hh**



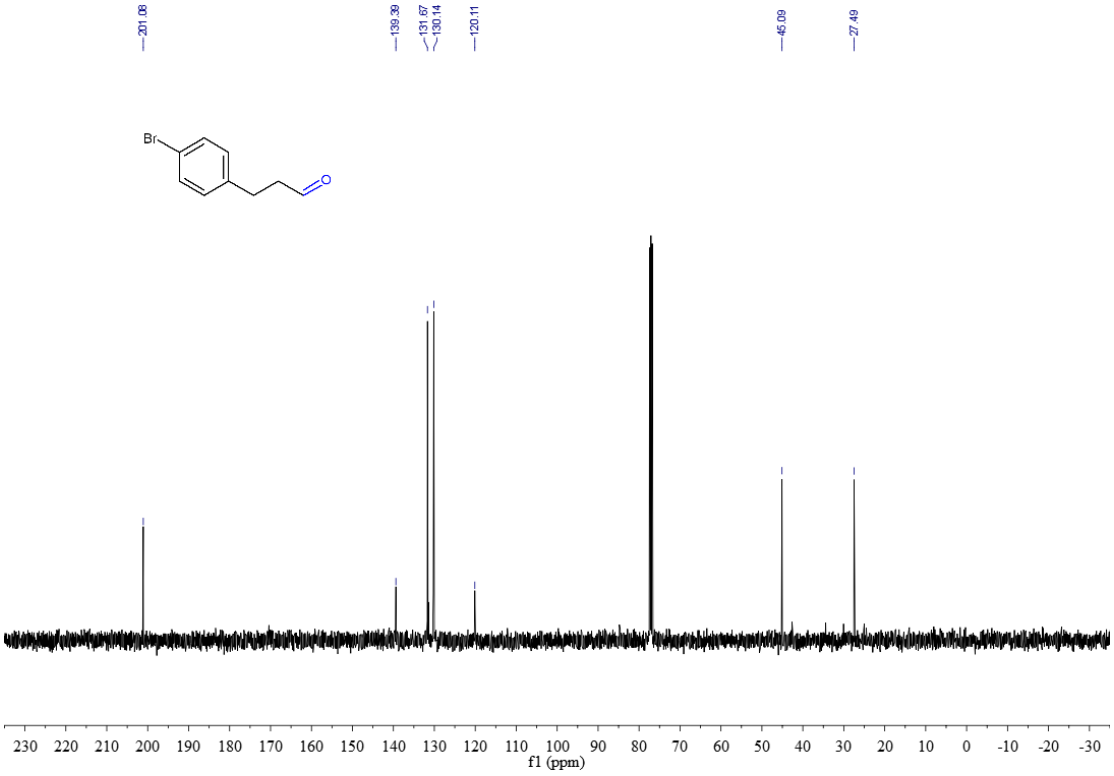
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **2hh**



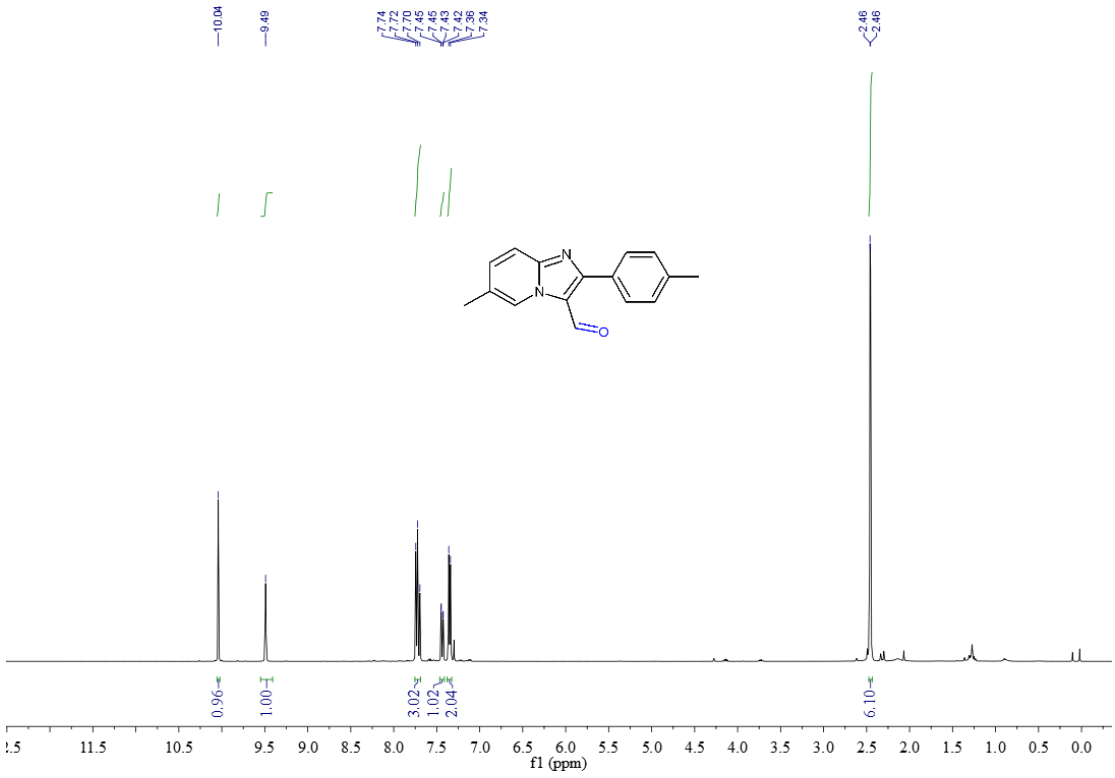
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4a**



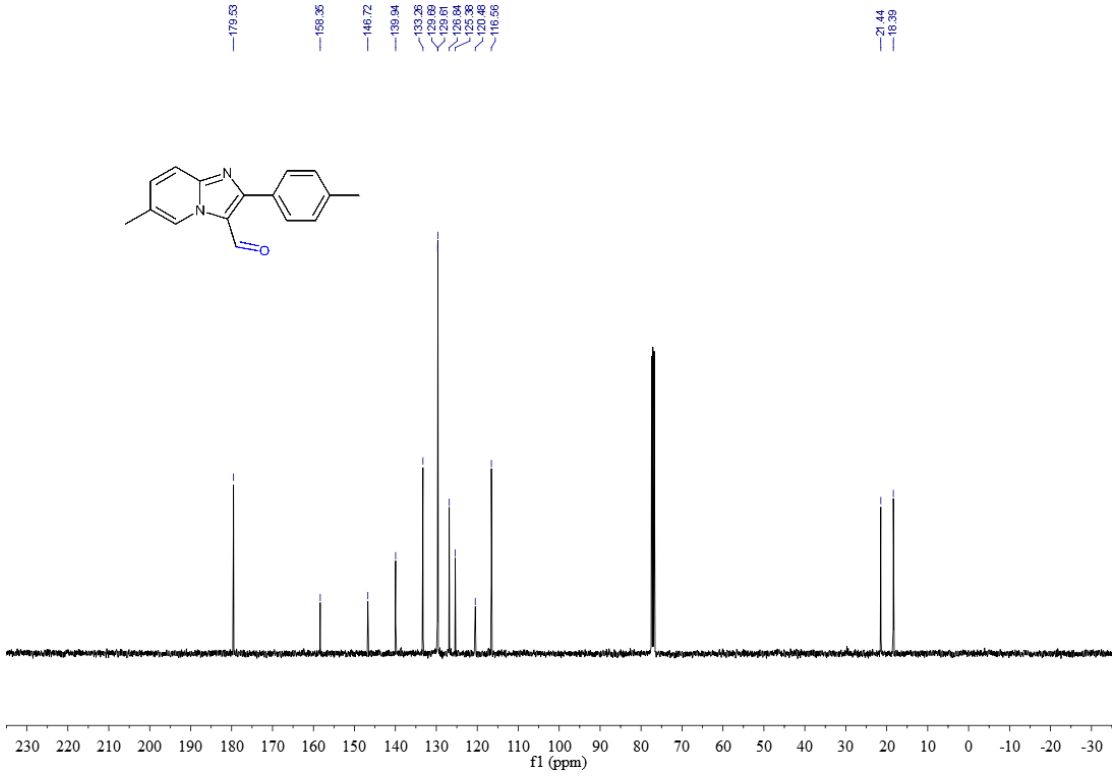
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4a**



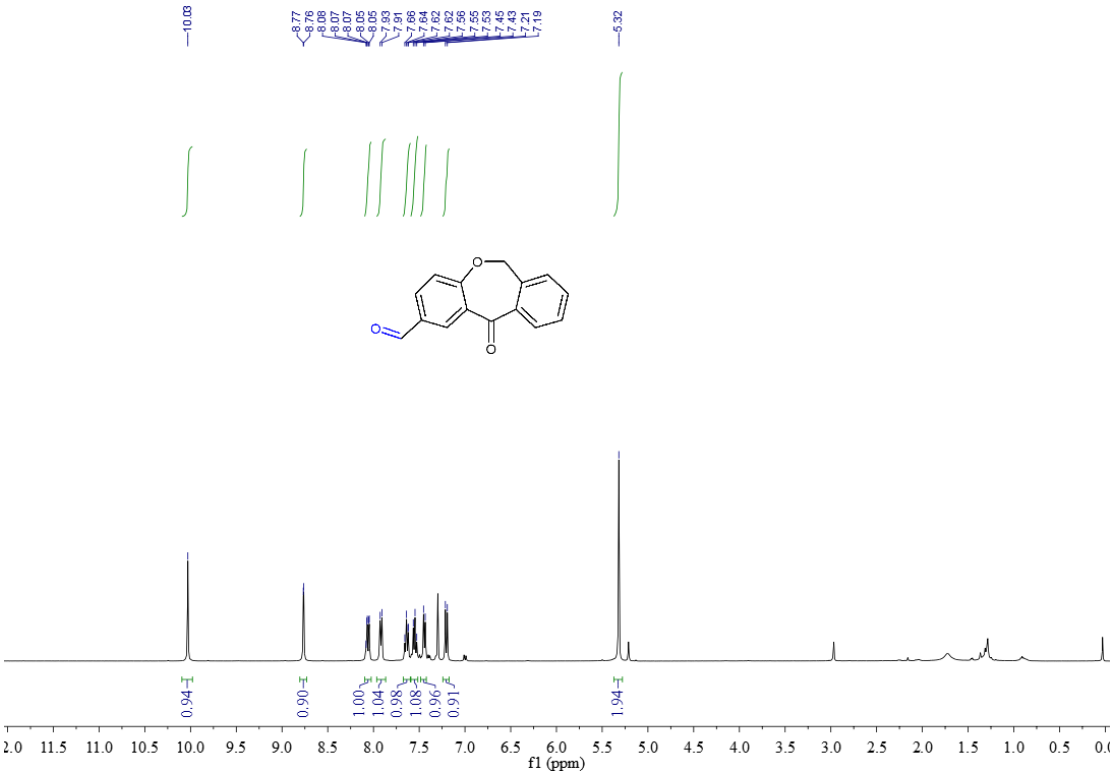
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4b**



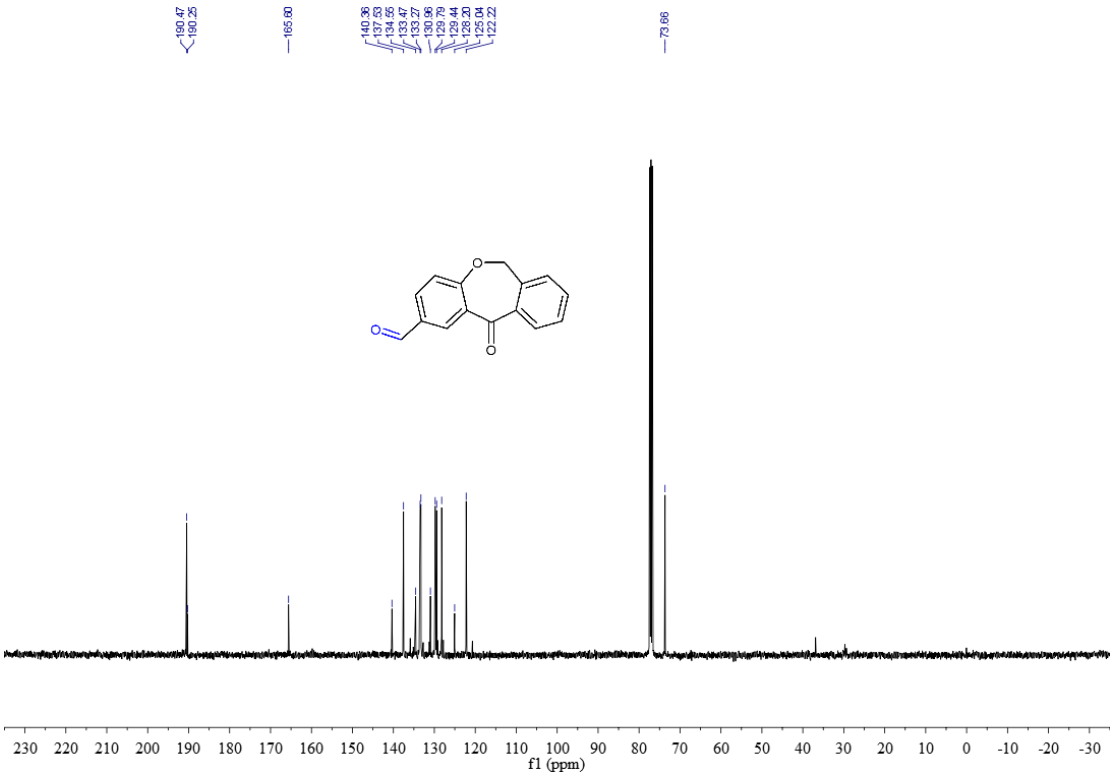
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4b**



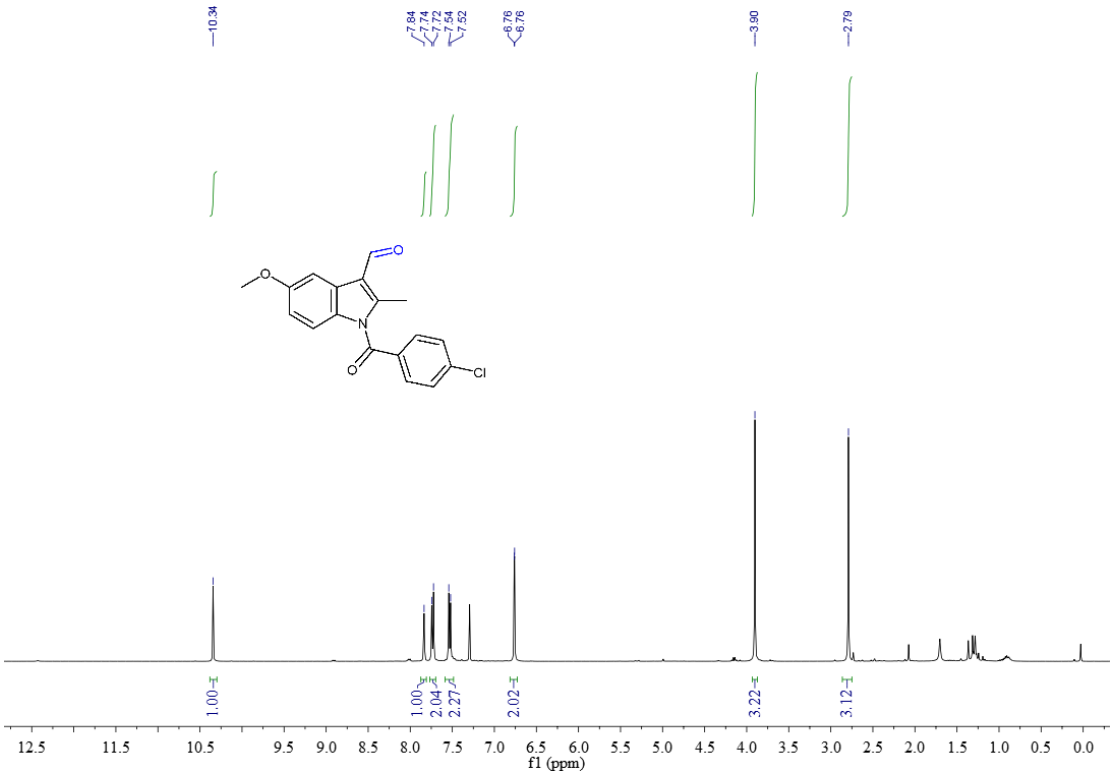
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4c**



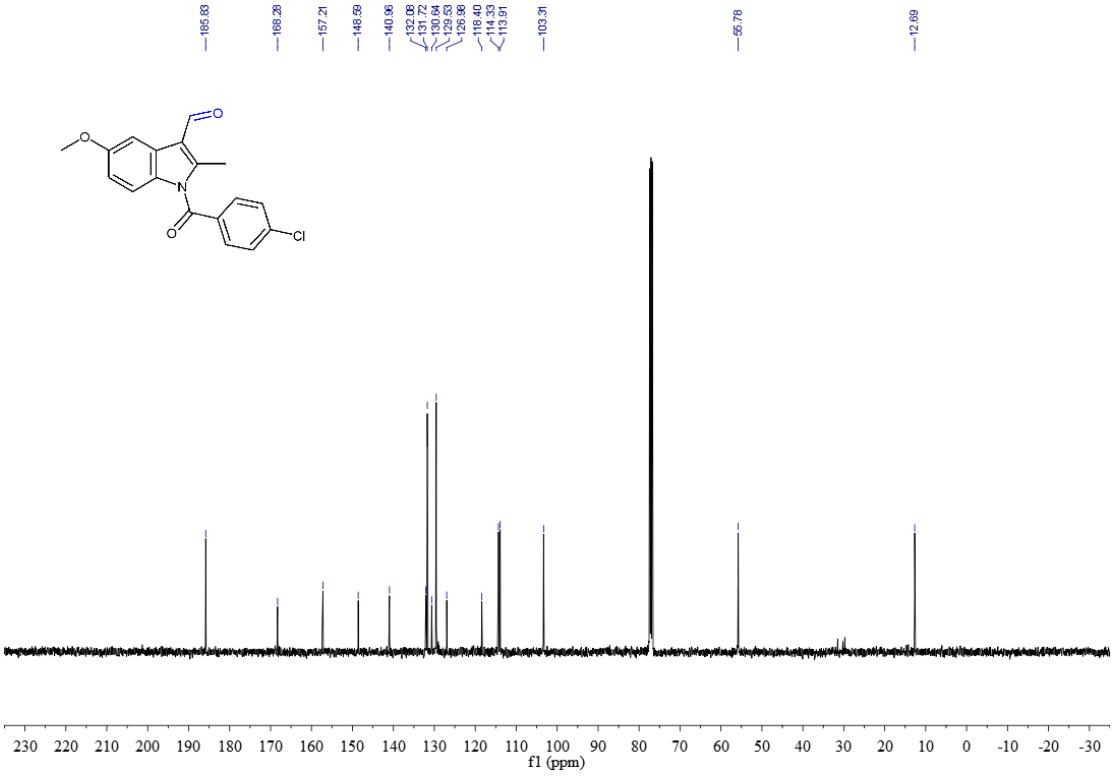
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4c**



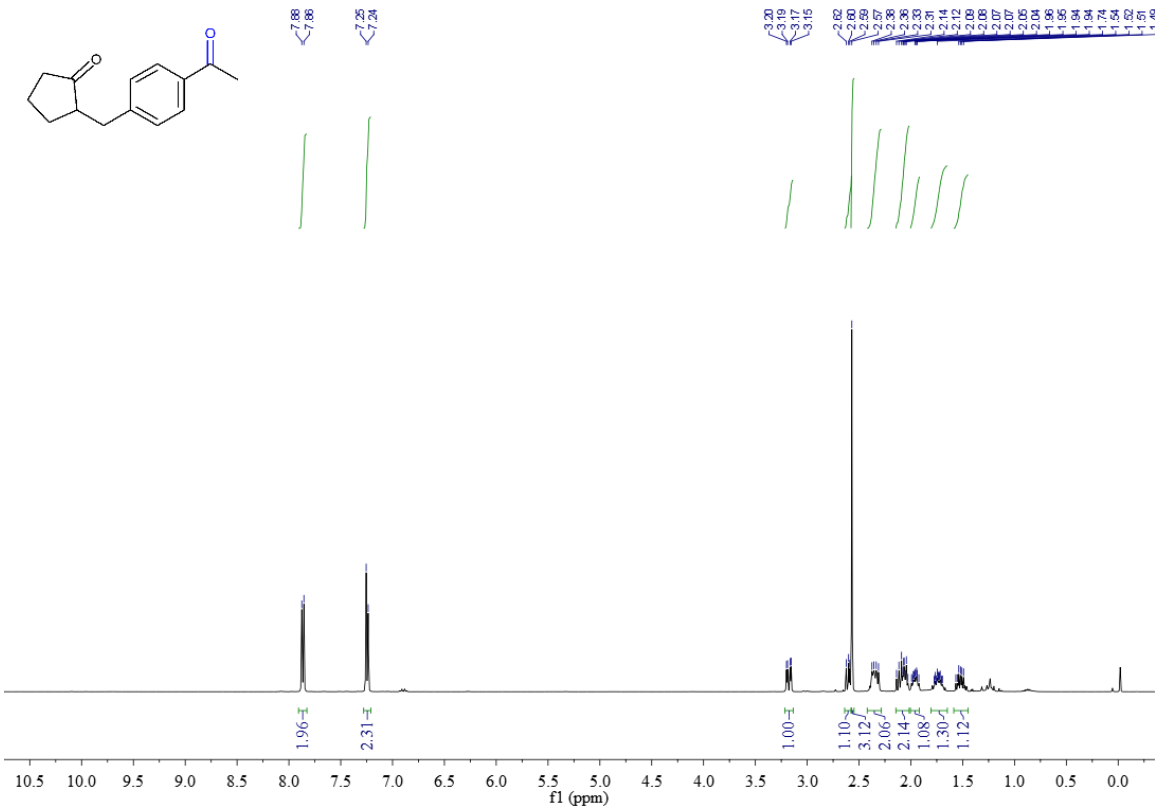
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4d**



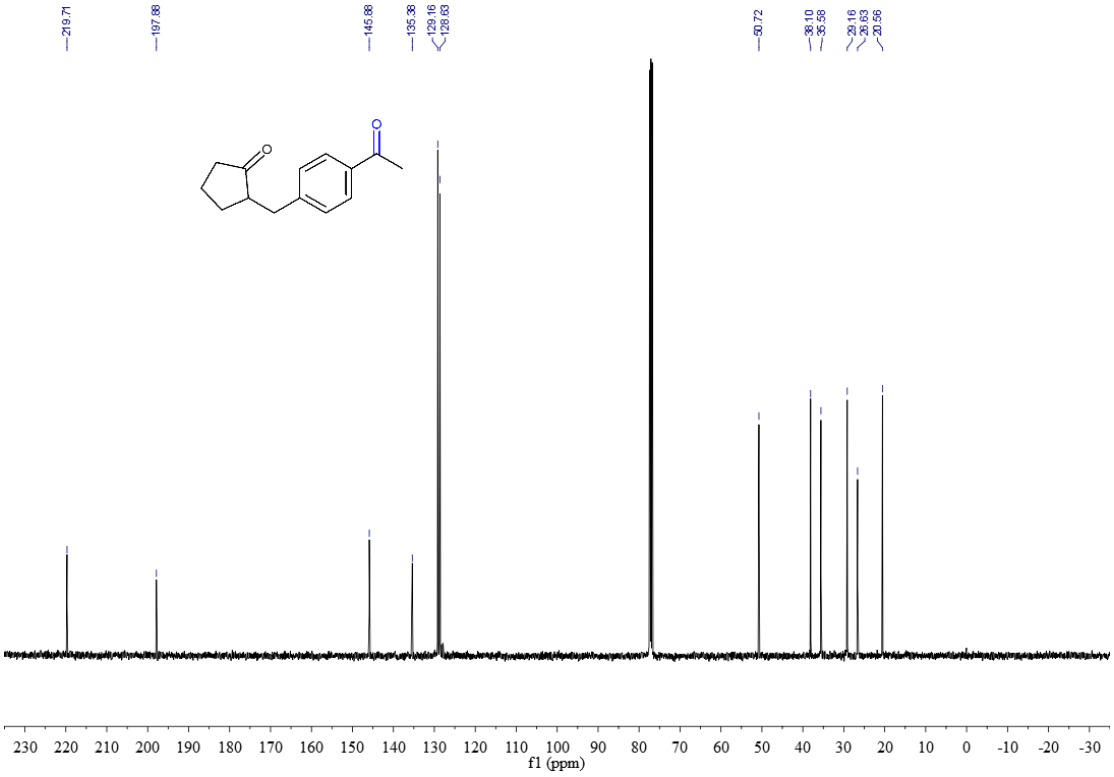
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4d**



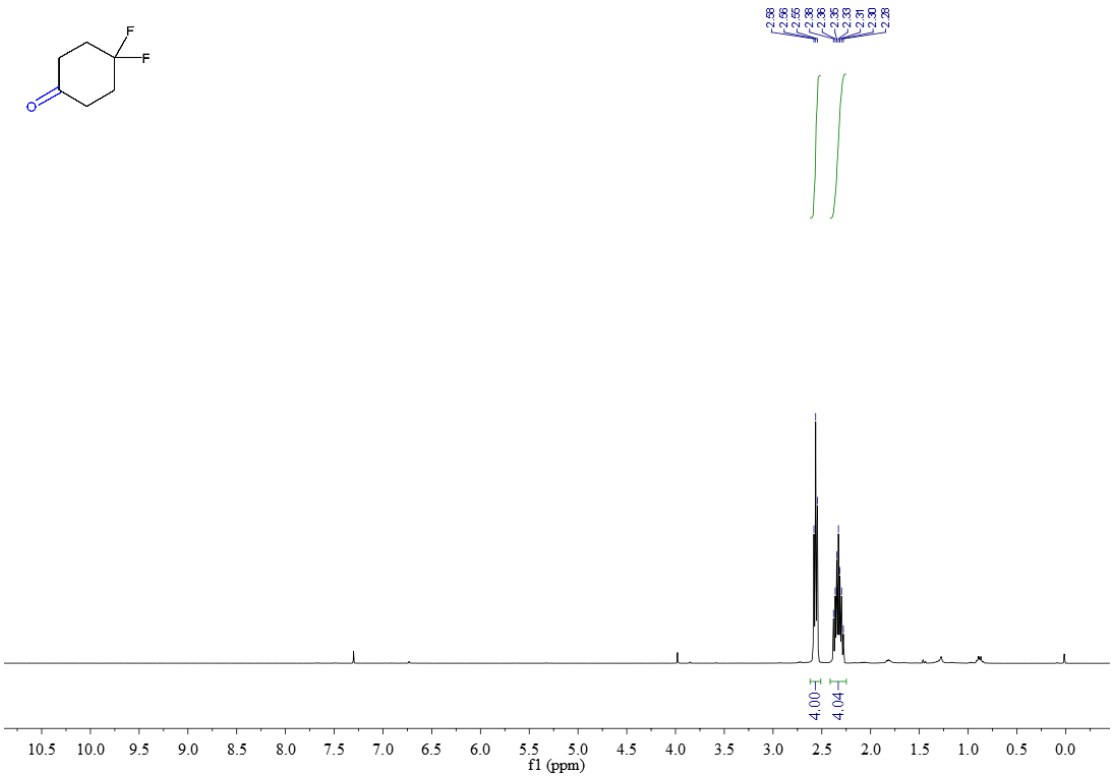
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4e**



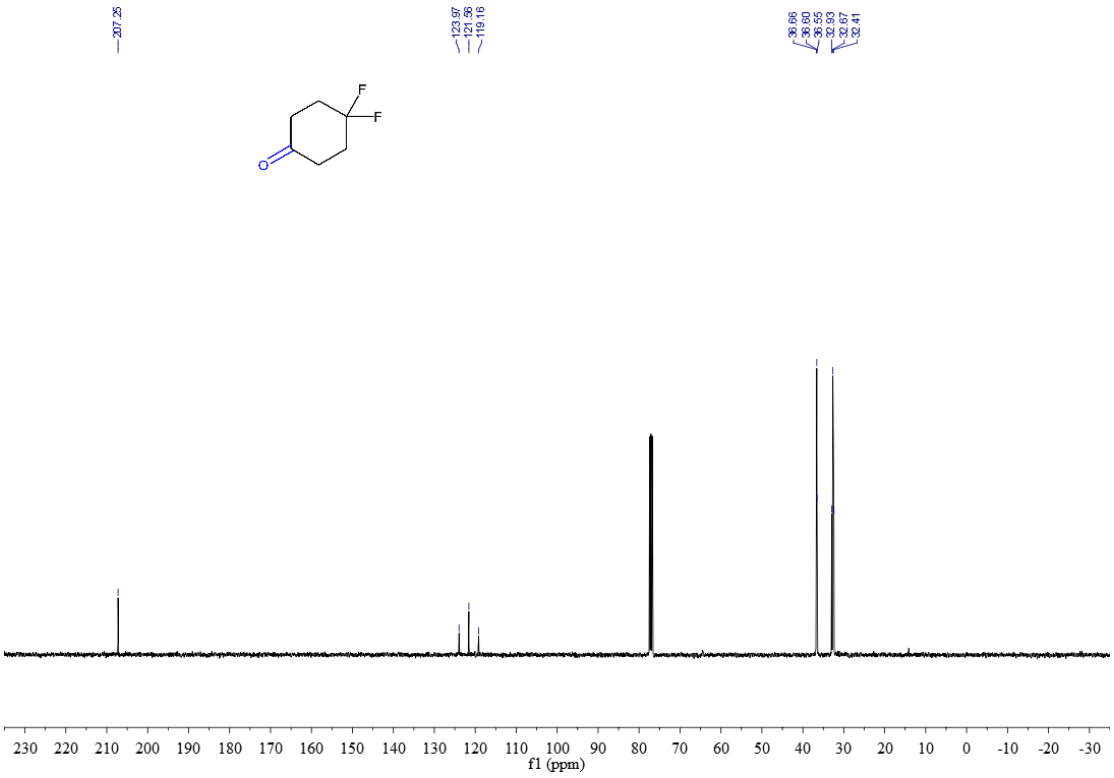
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4e**



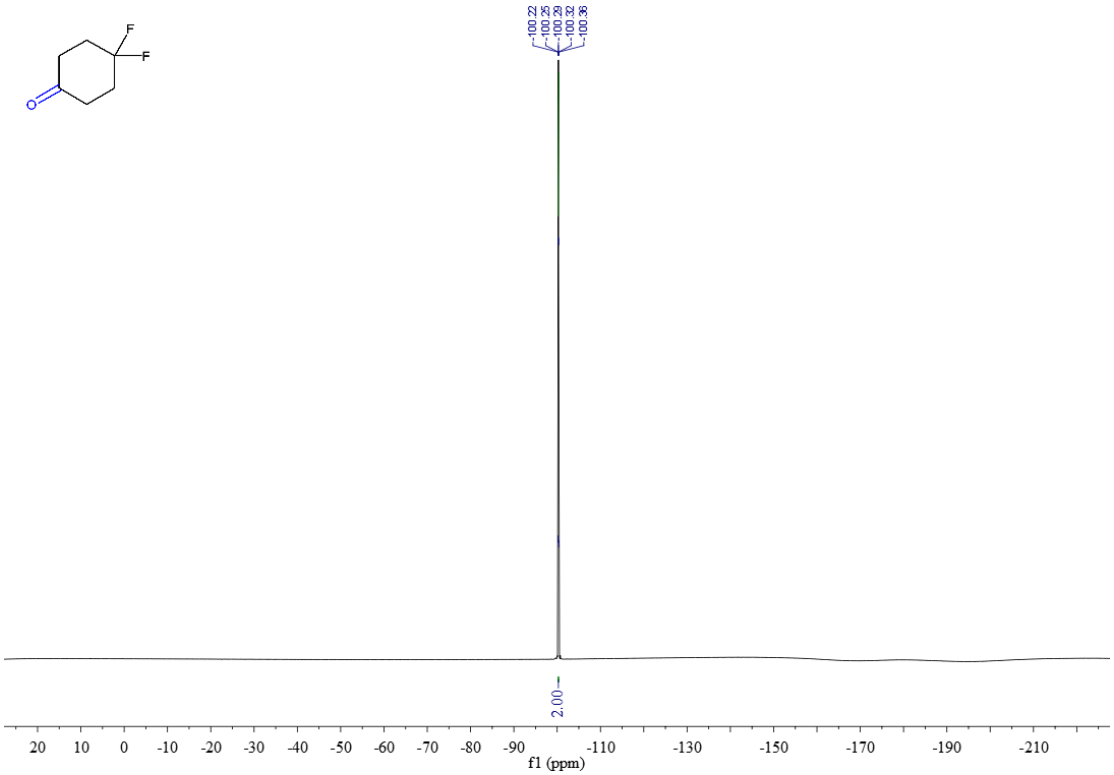
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4f**



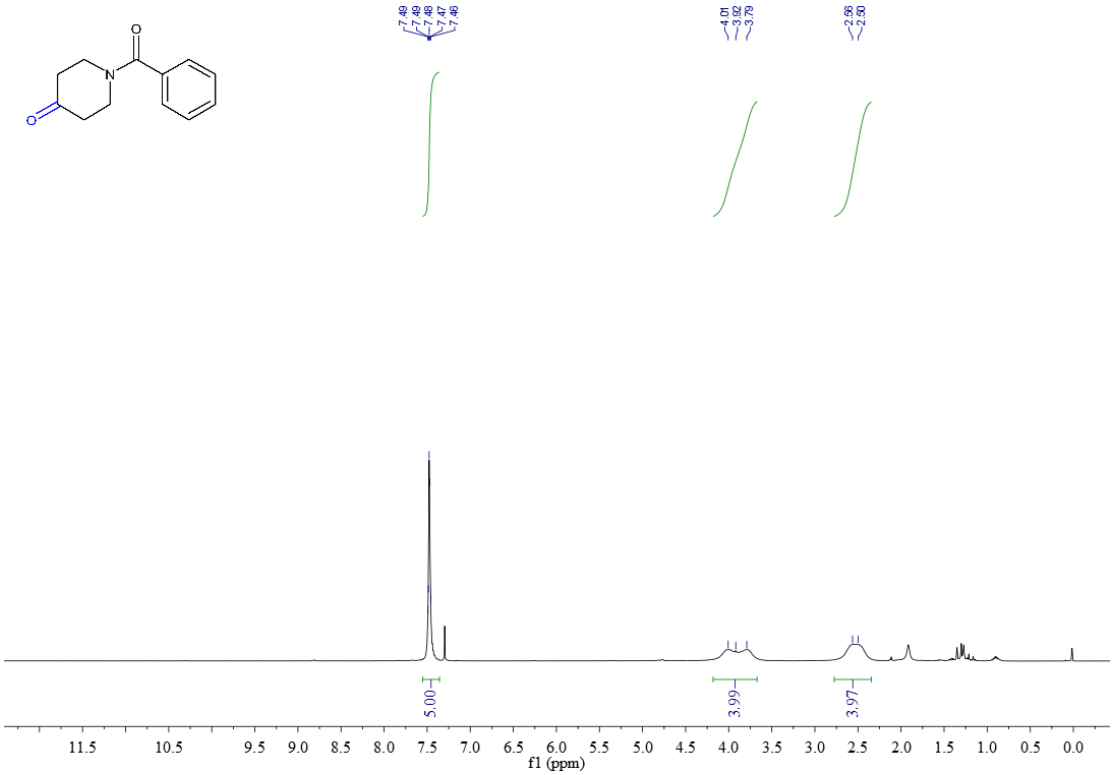
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4f**



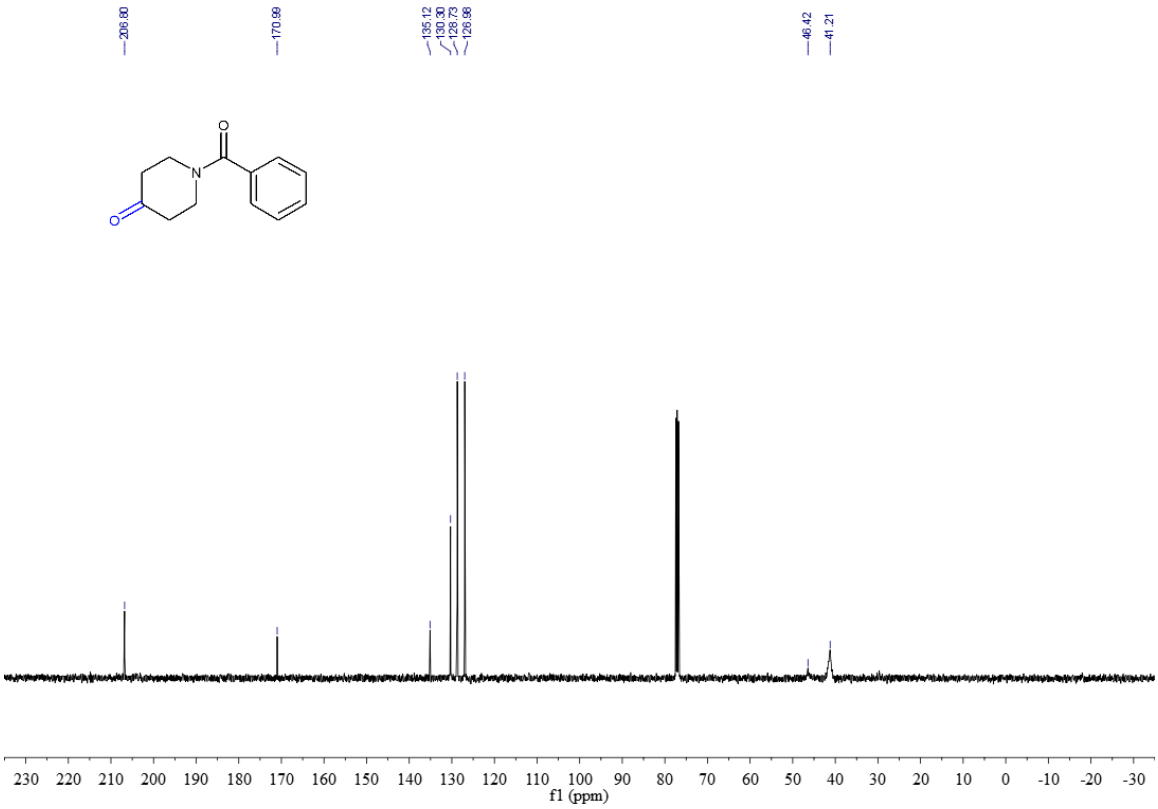
^{19}F NMR spectrum (376 MHz, $CDCl_3$, 23 ℃) of **4f**



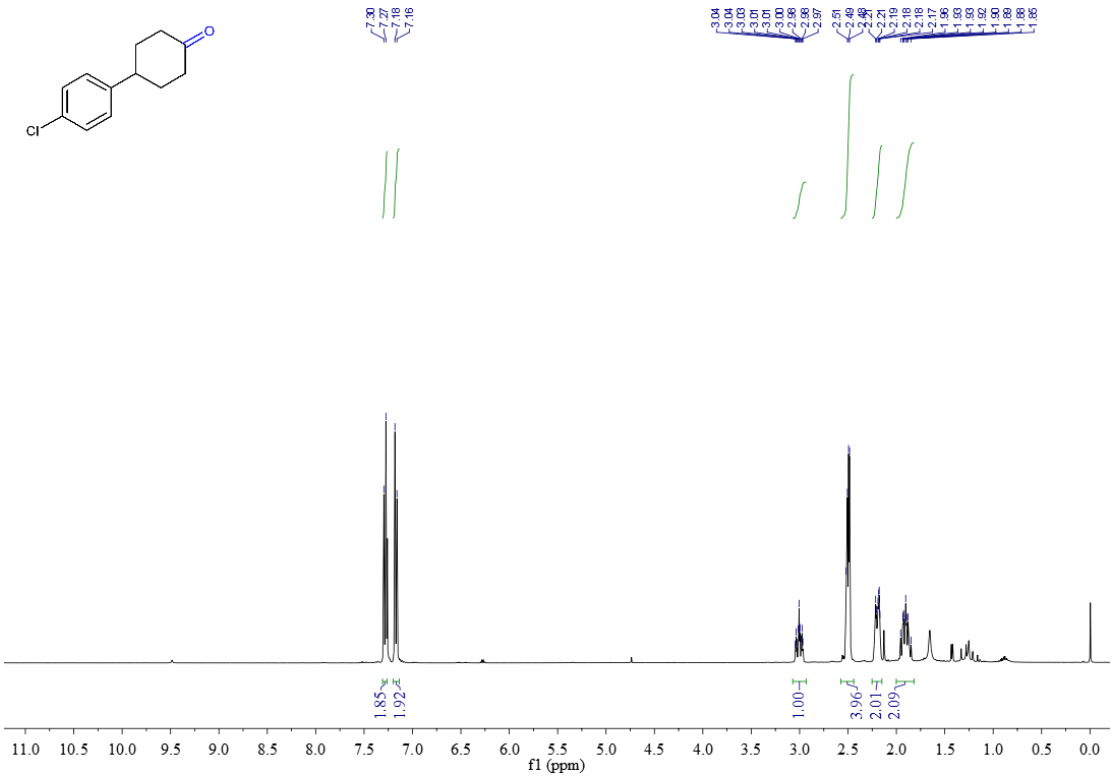
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4g**



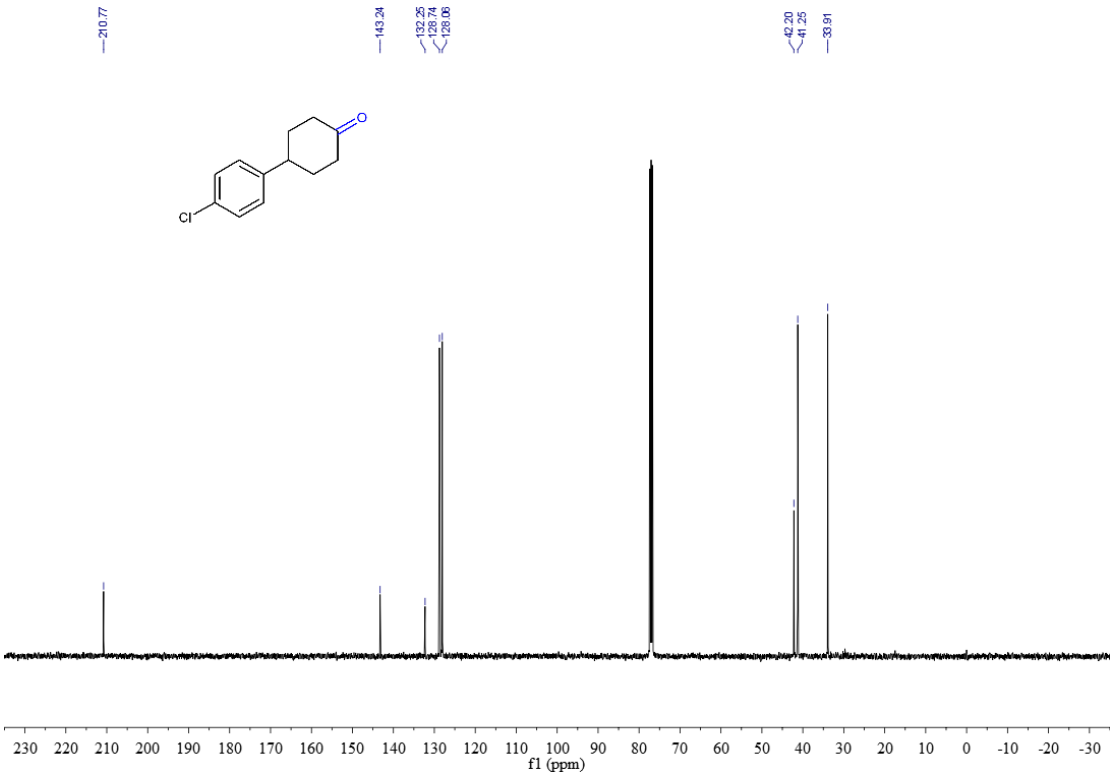
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4g**



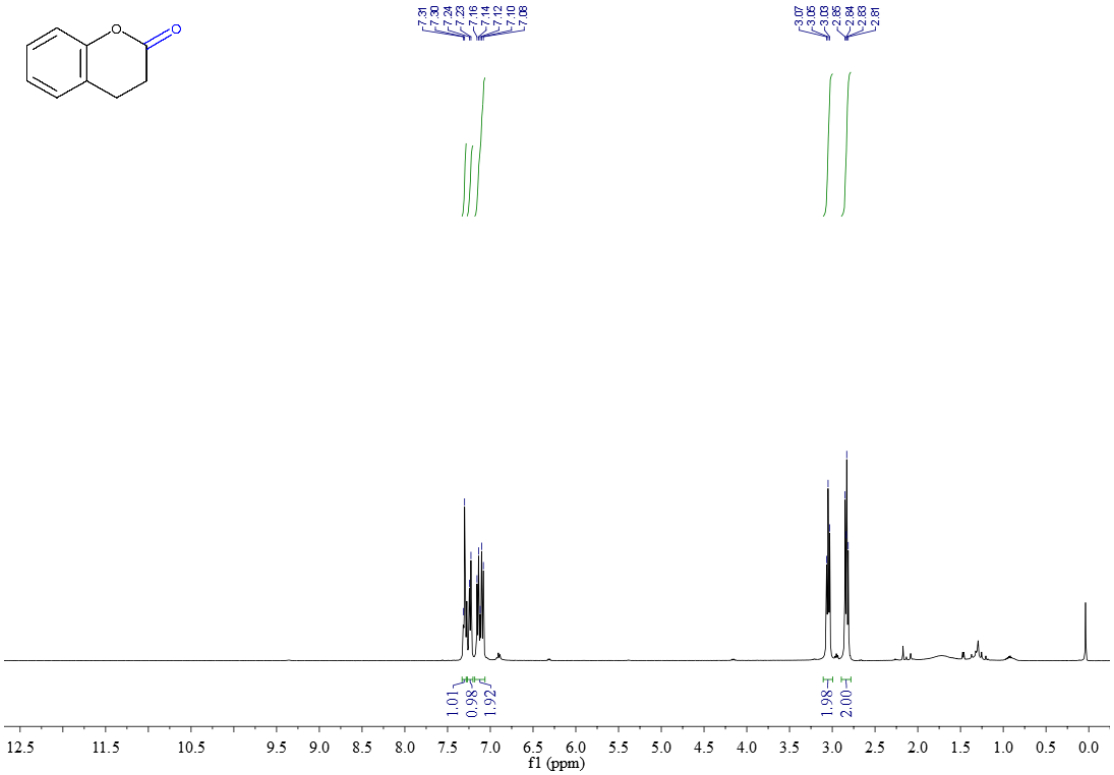
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4h**



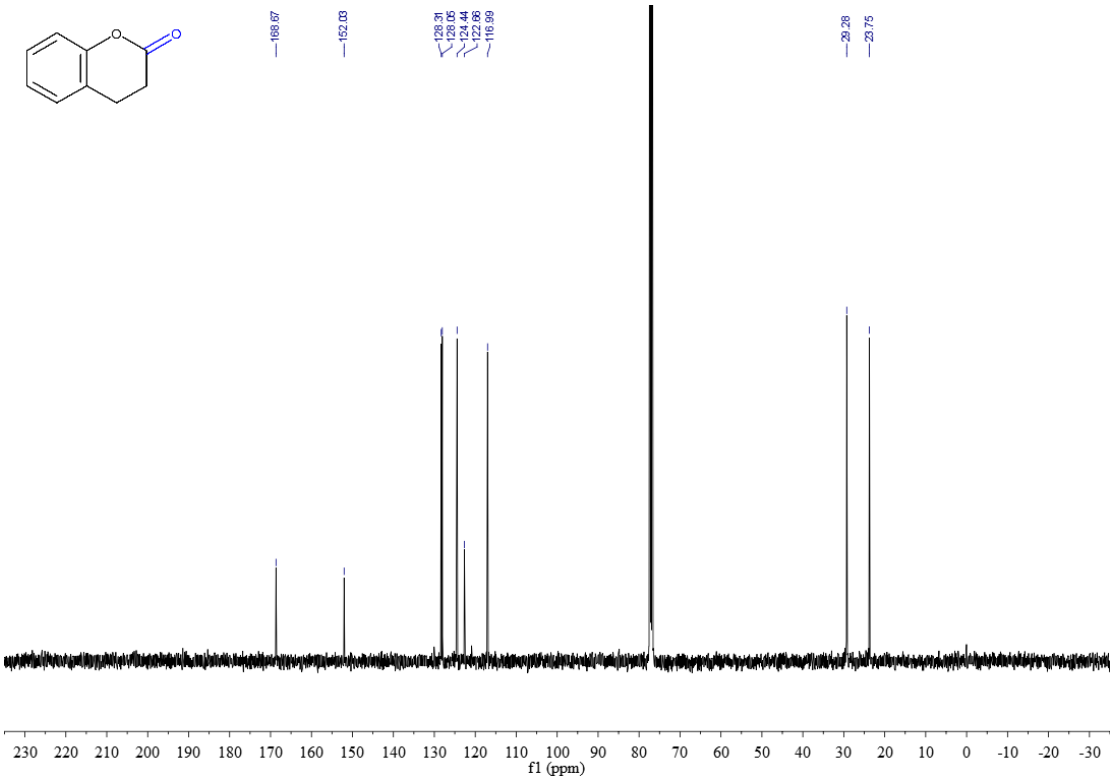
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4h**



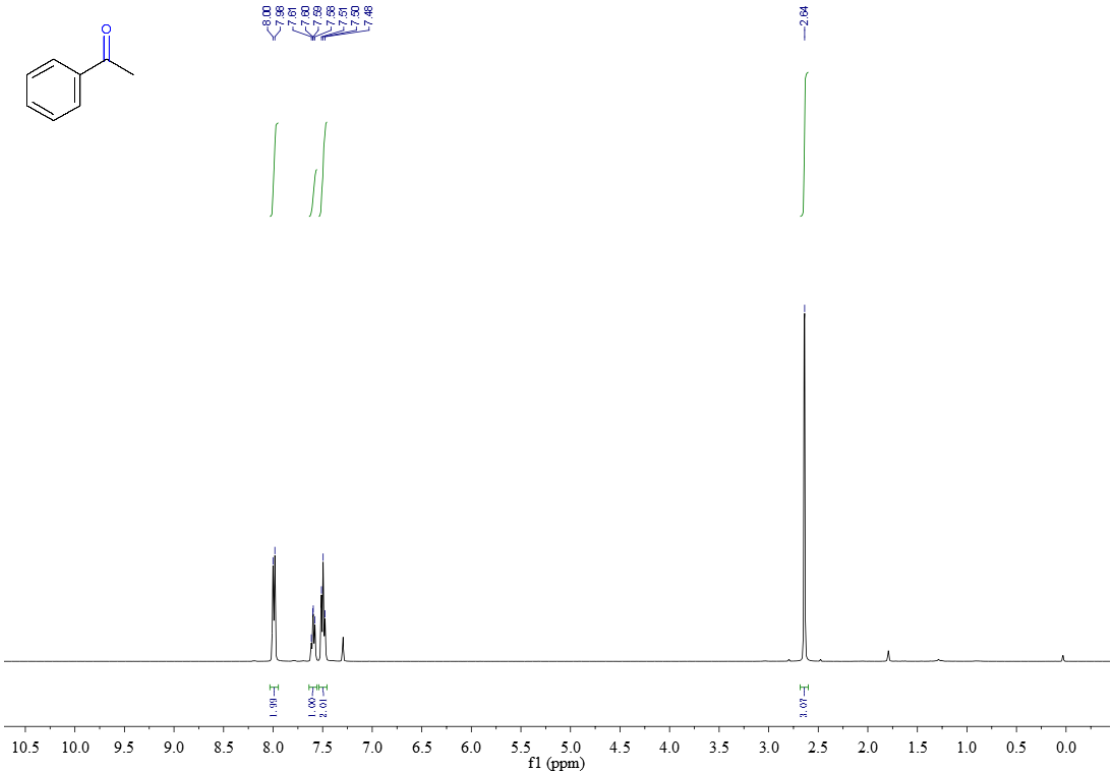
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4i**



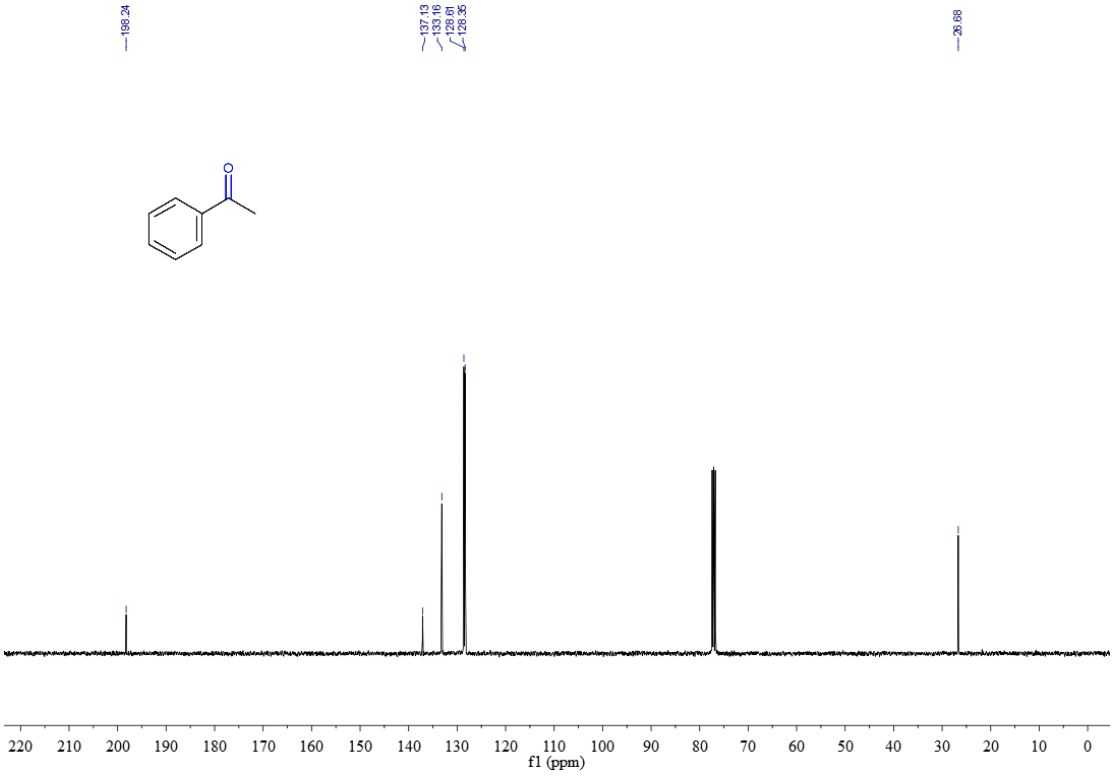
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4i**



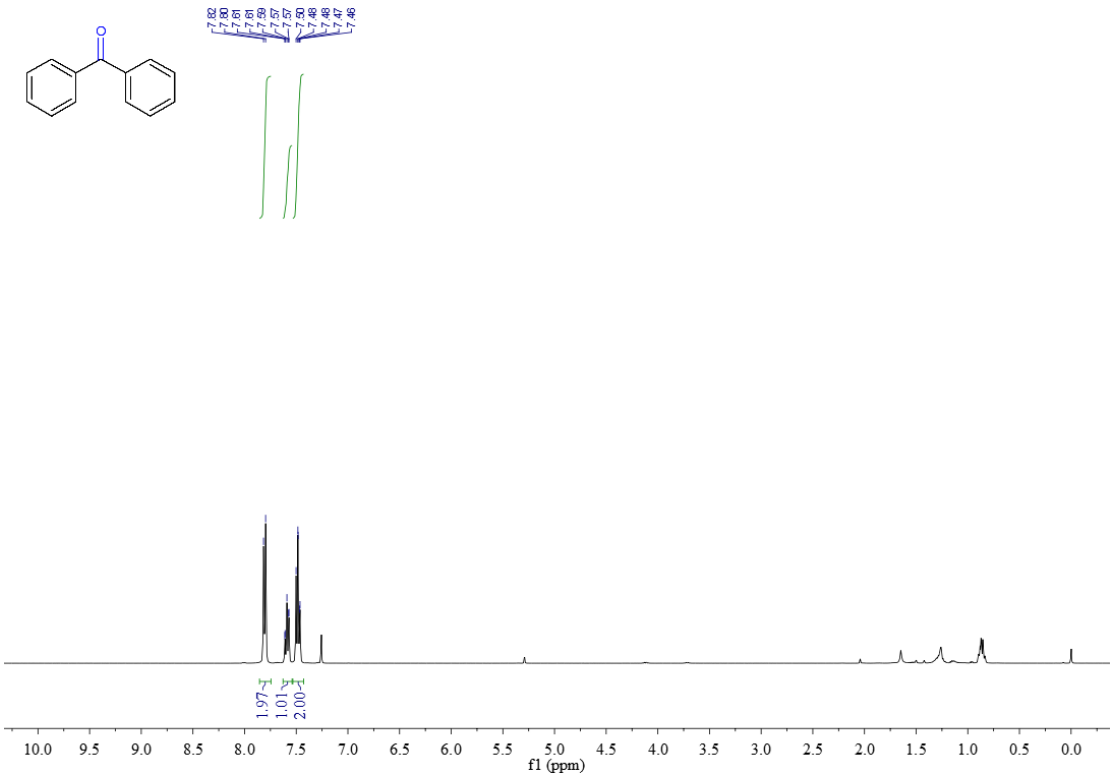
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4j**



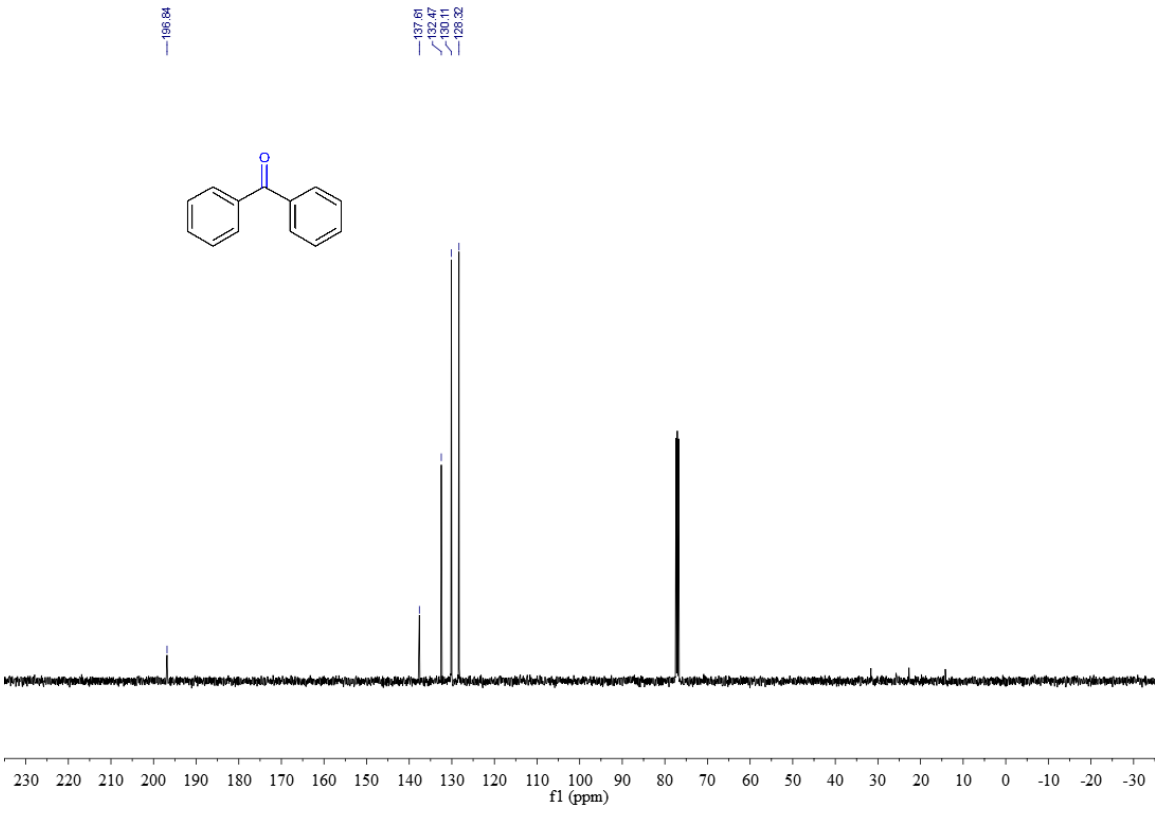
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4j**



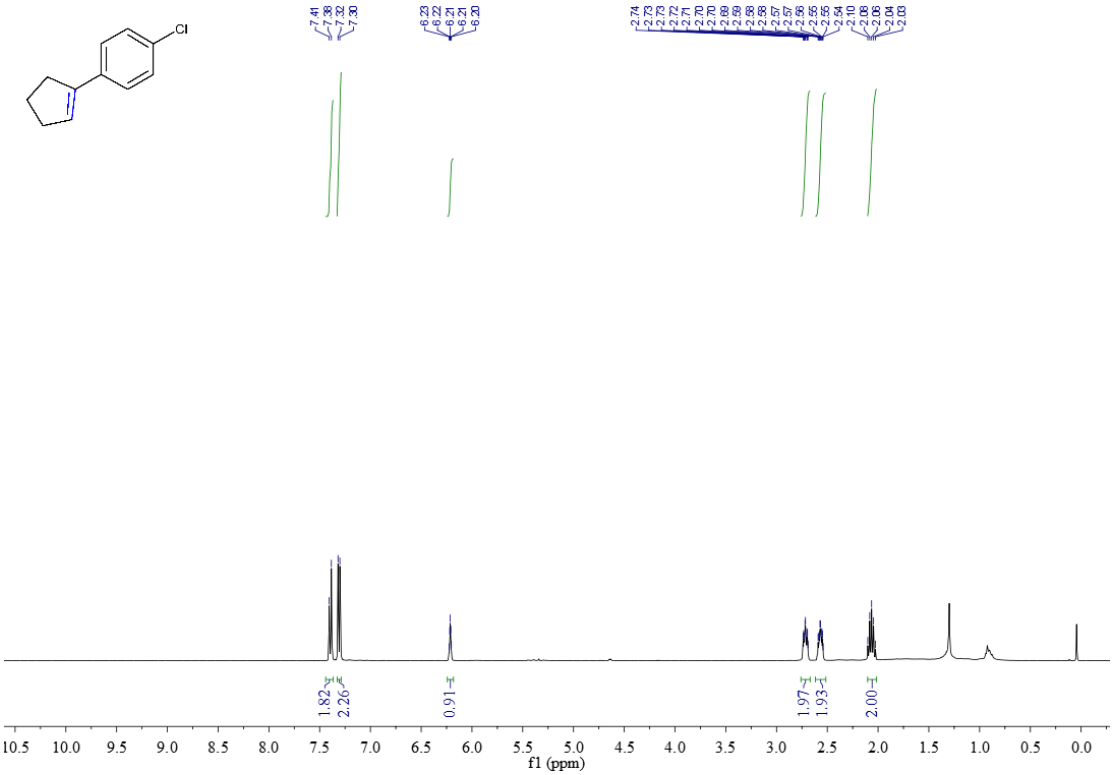
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **4k**



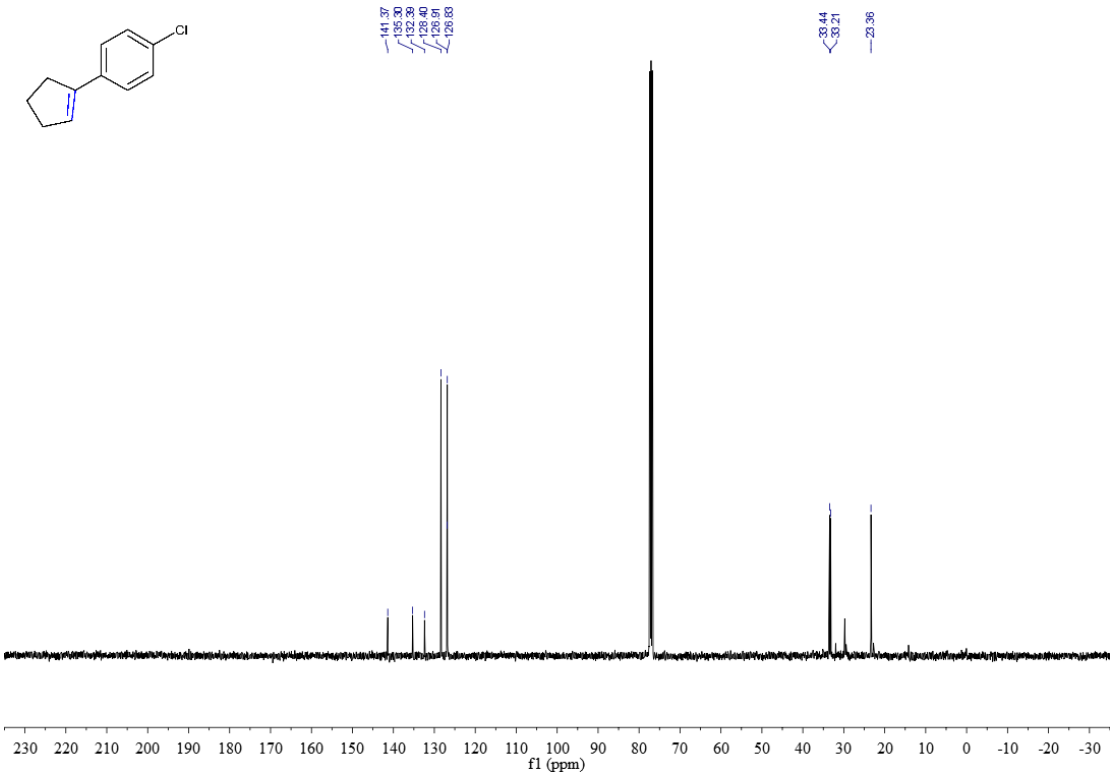
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **4k**



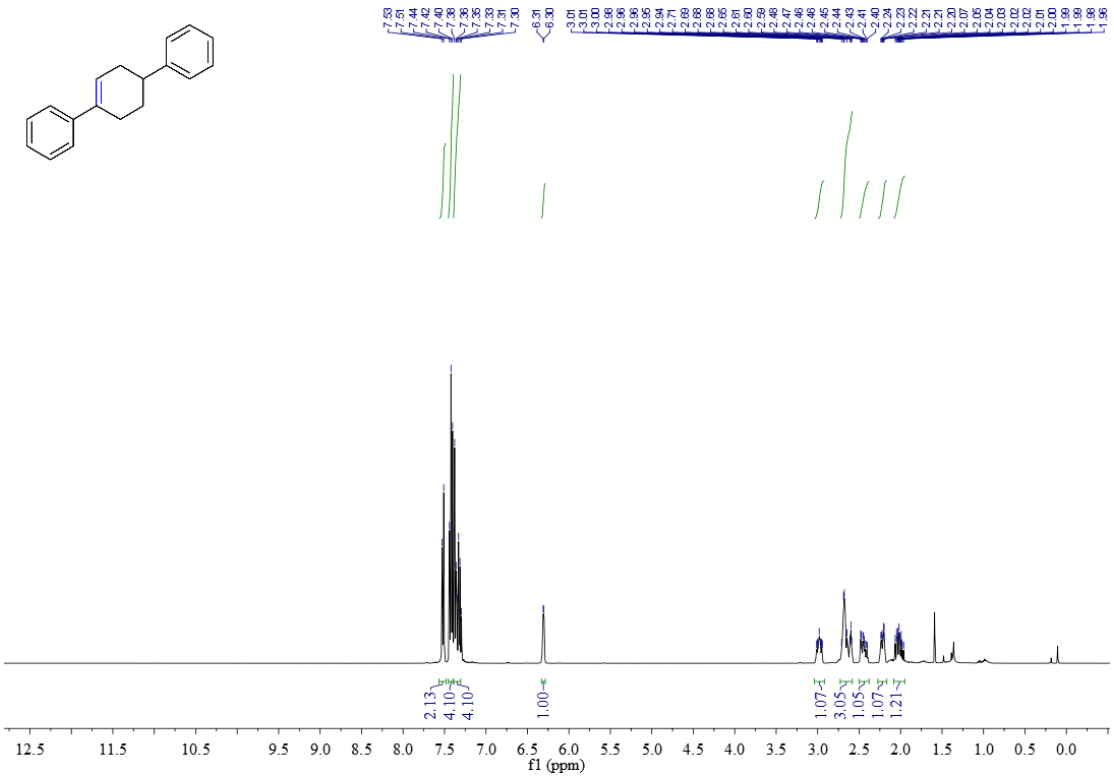
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6a**



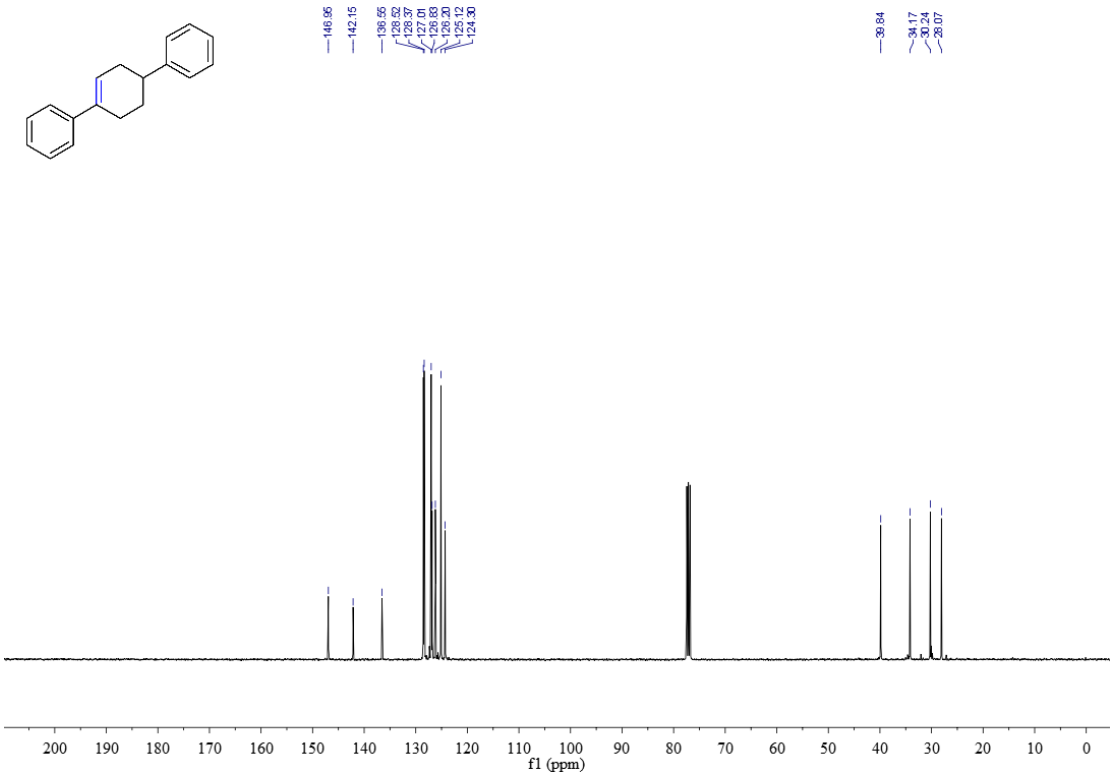
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6a**



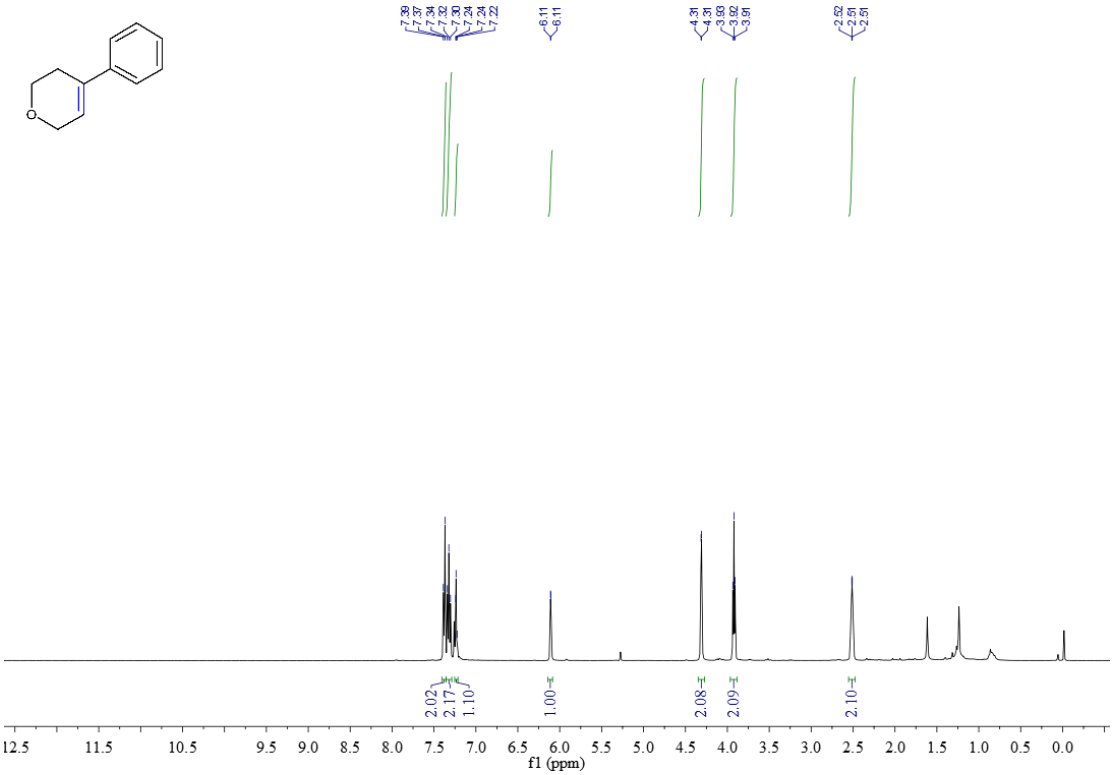
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6b**



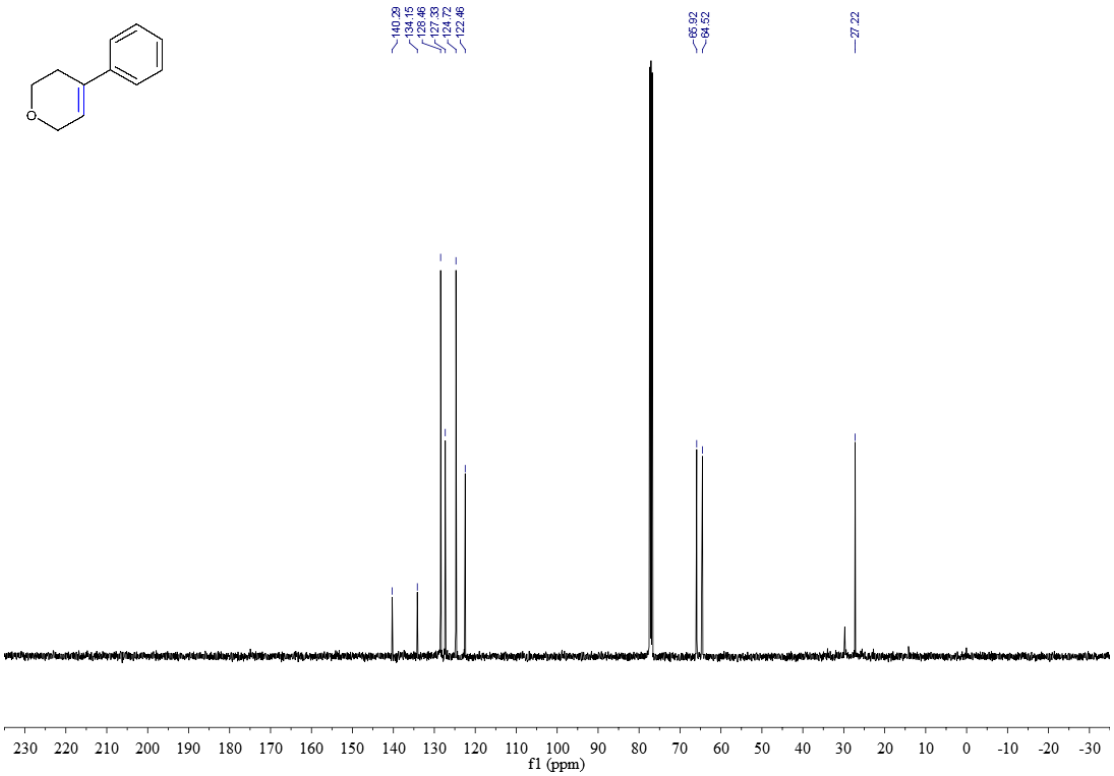
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6b**



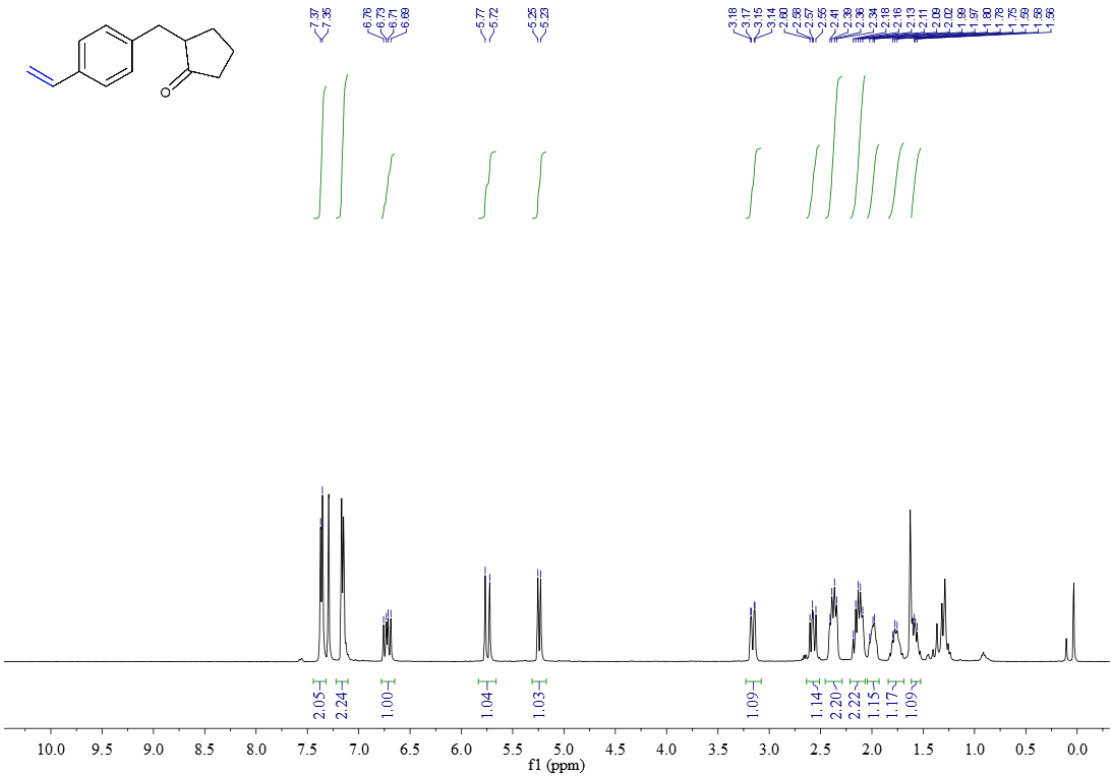
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6c**



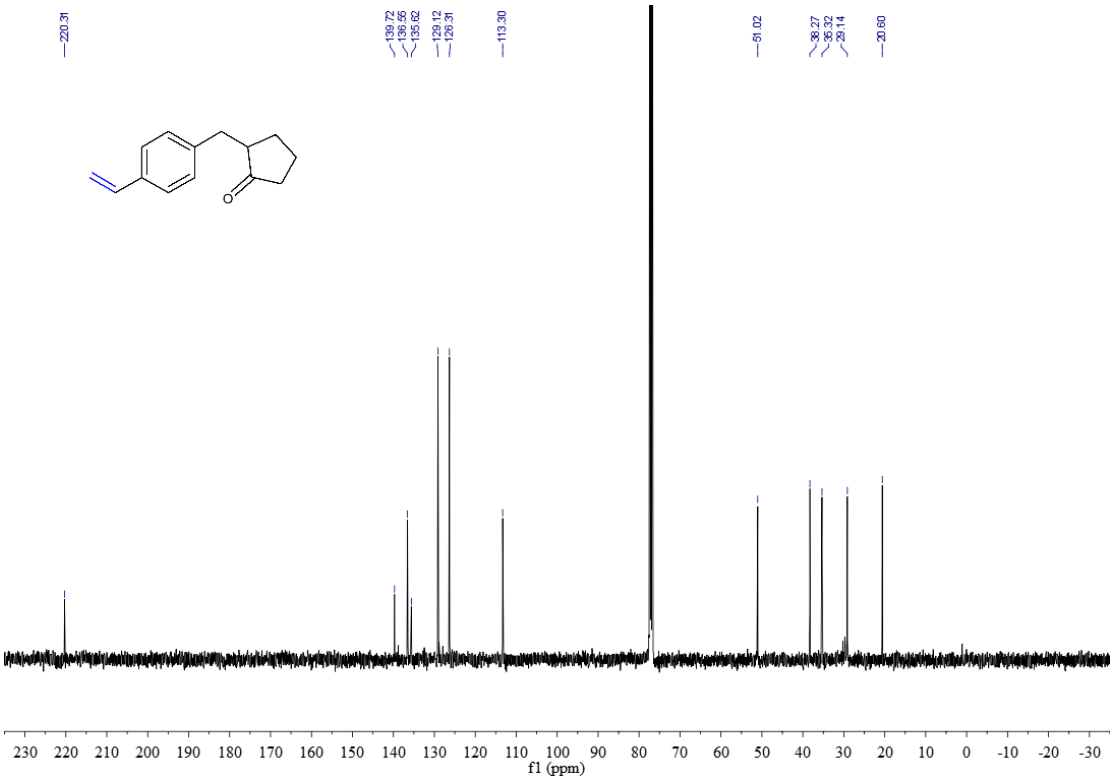
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6c**



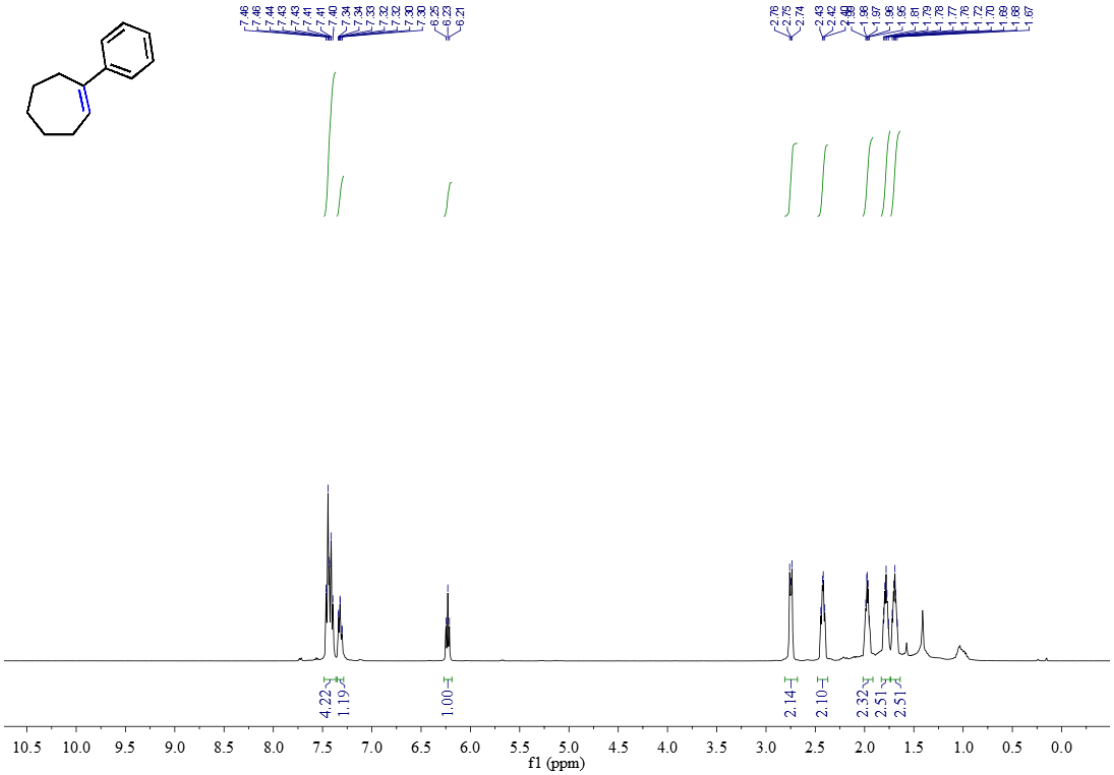
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6d**



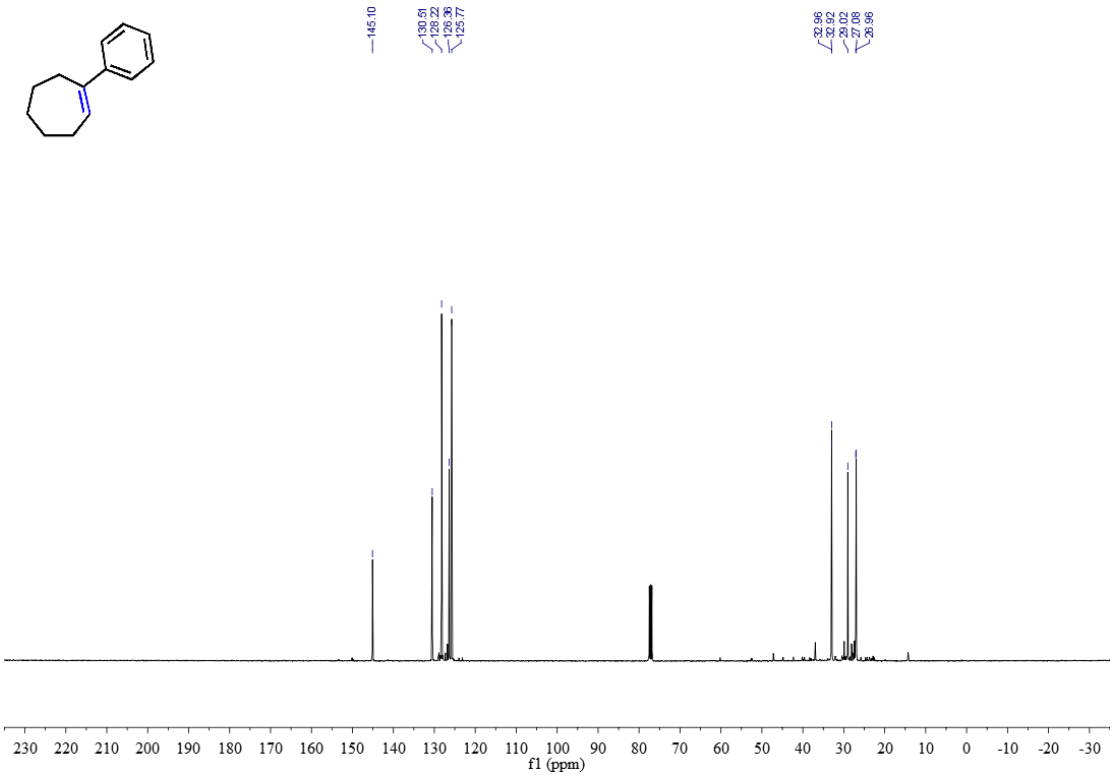
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6d**



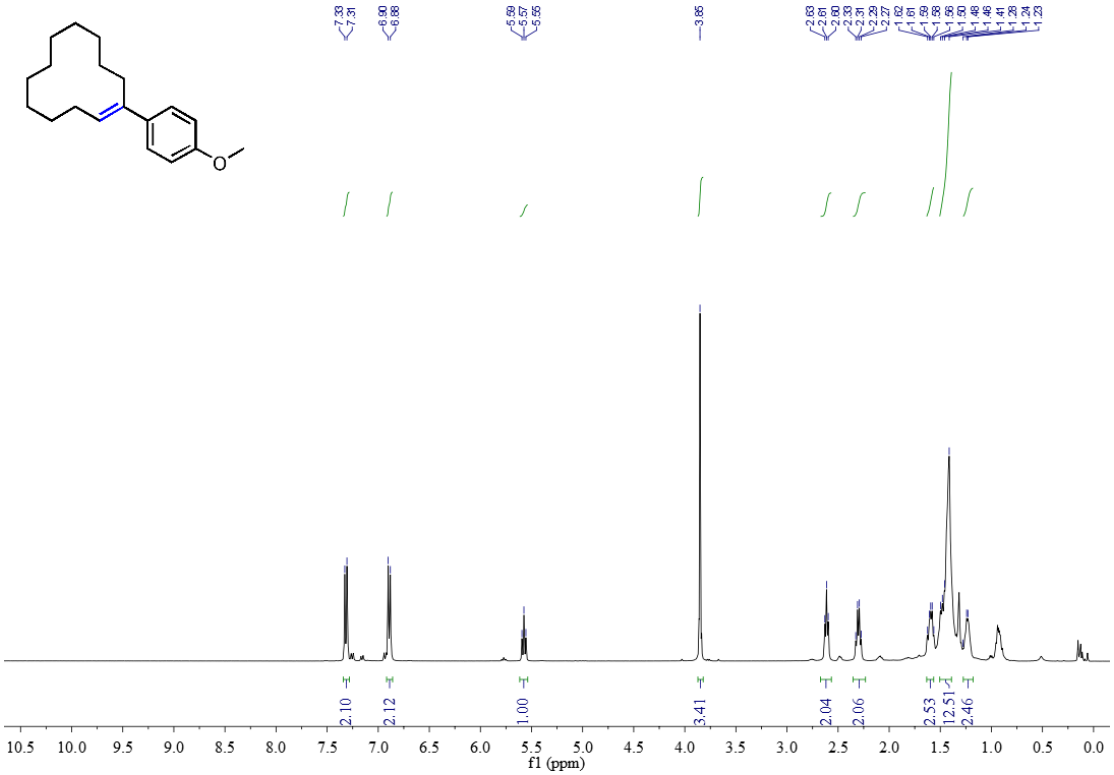
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6e**



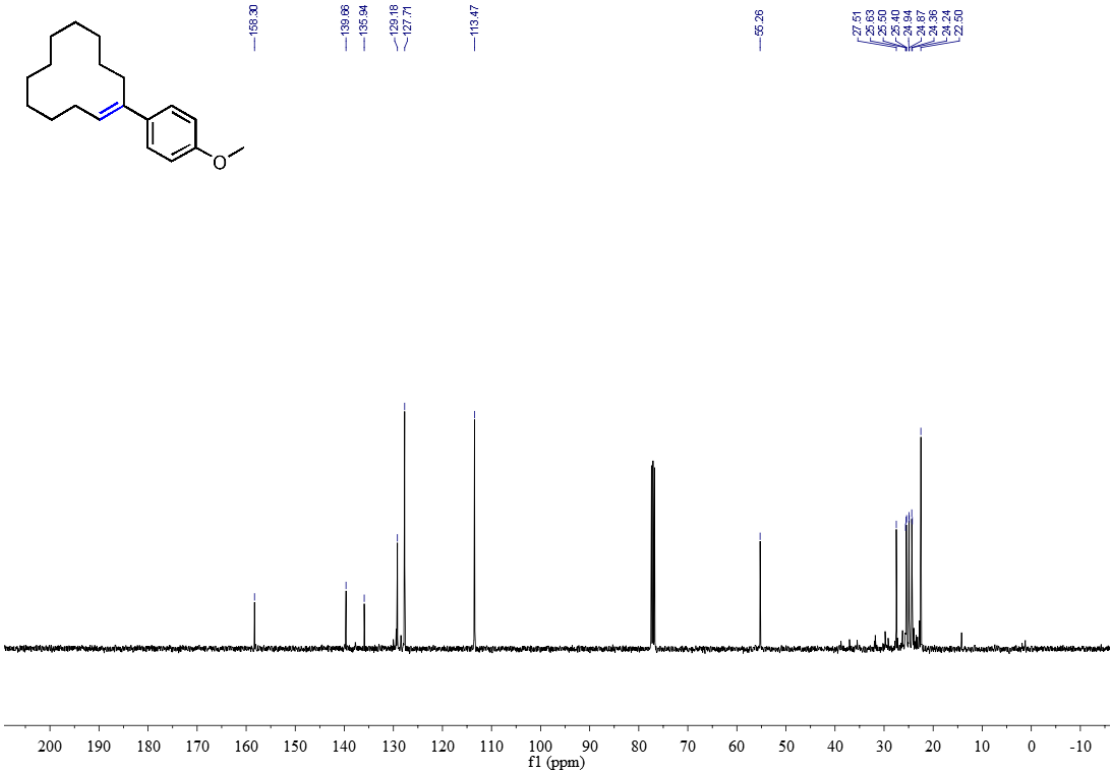
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6e**



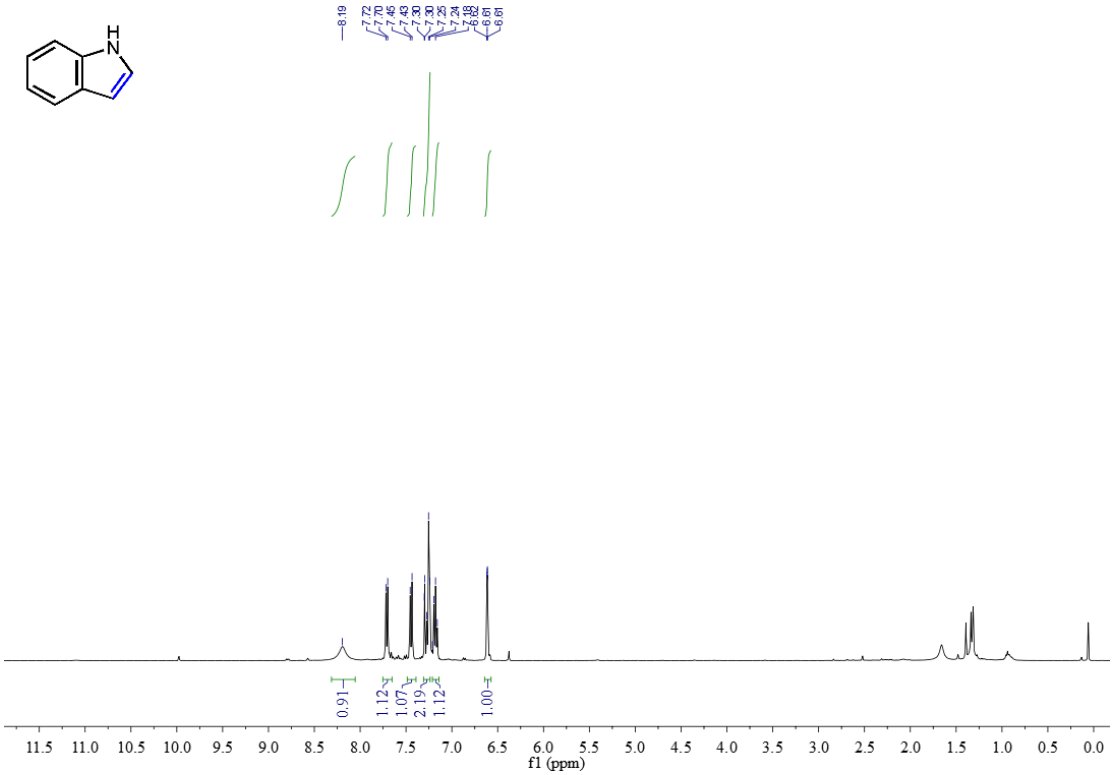
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6f**



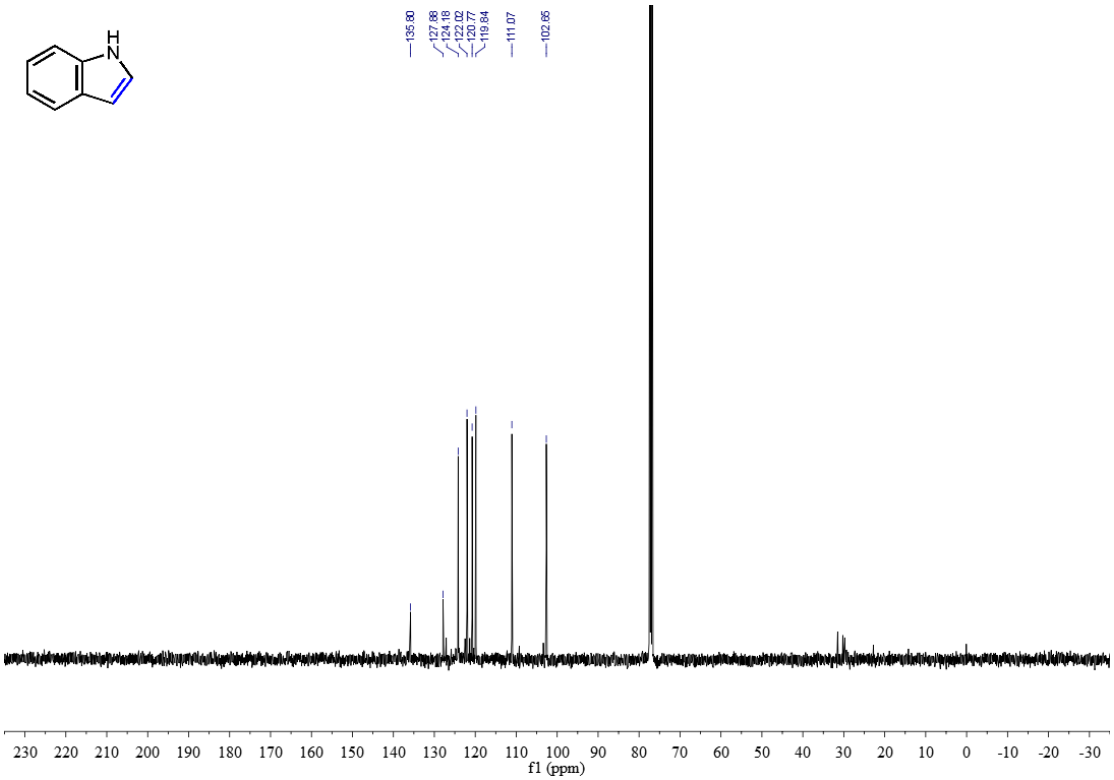
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6f**



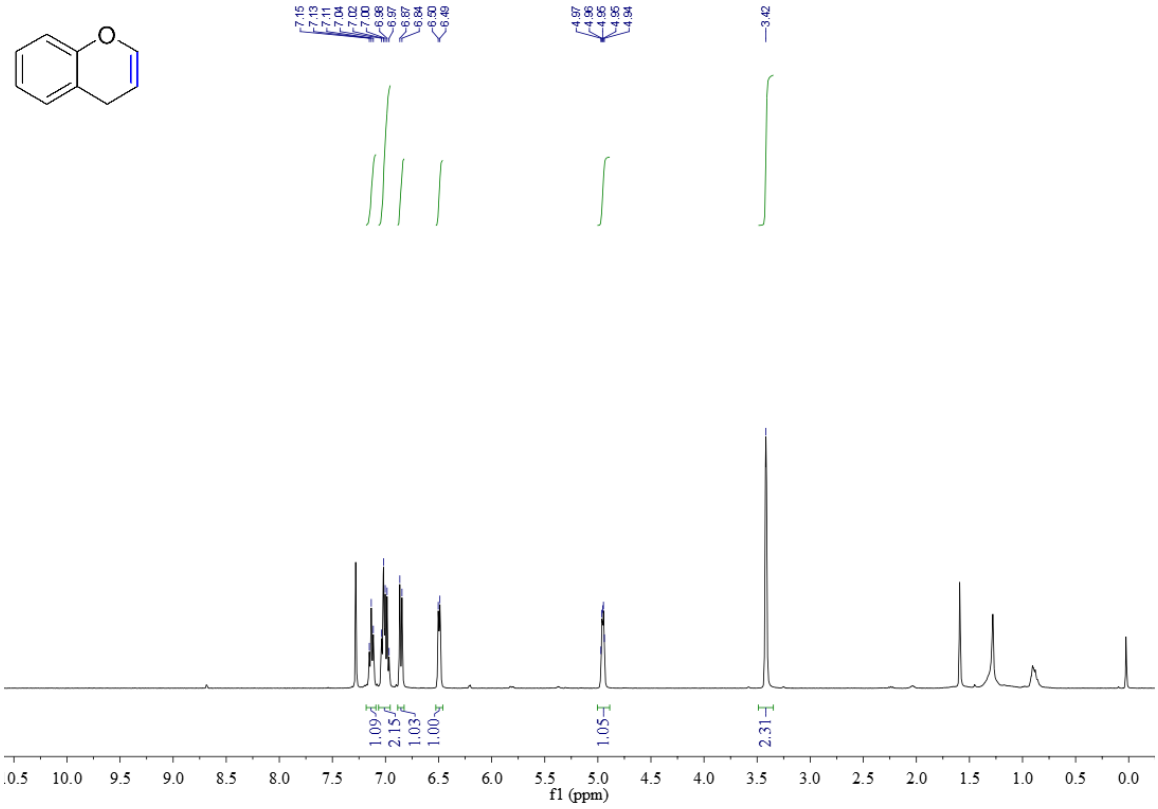
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6g**



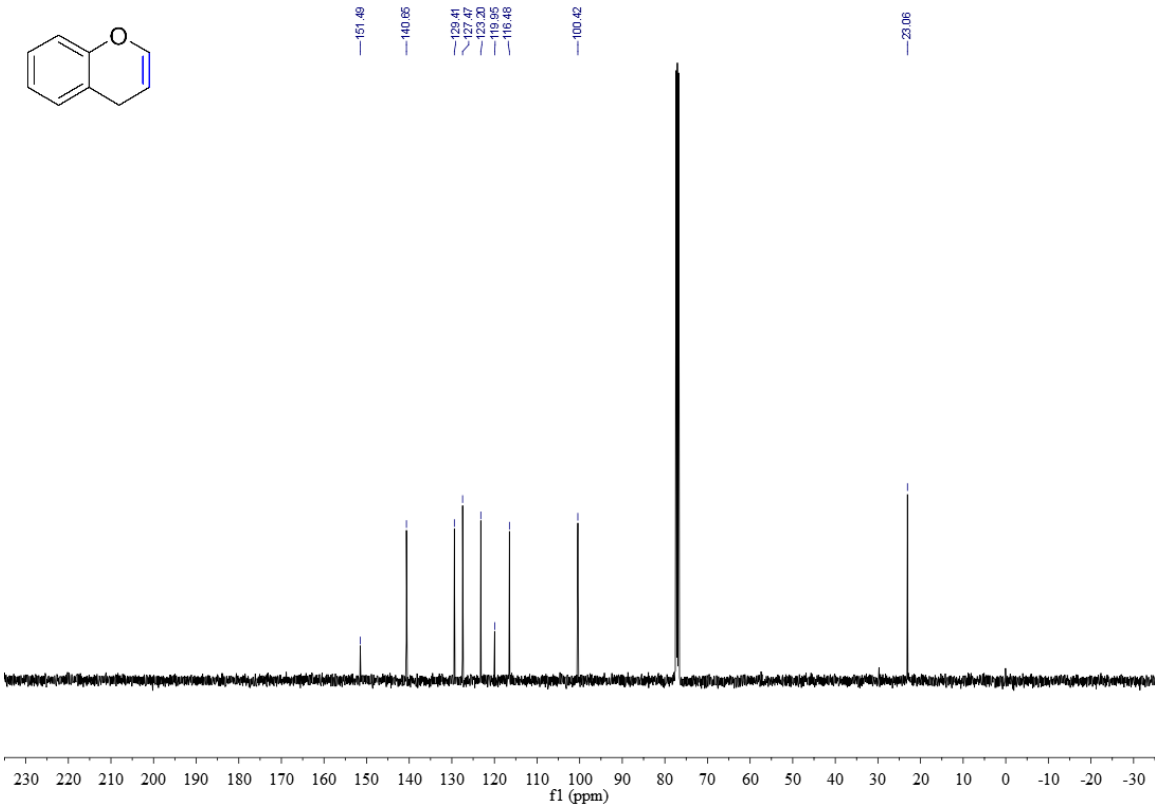
^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6g**



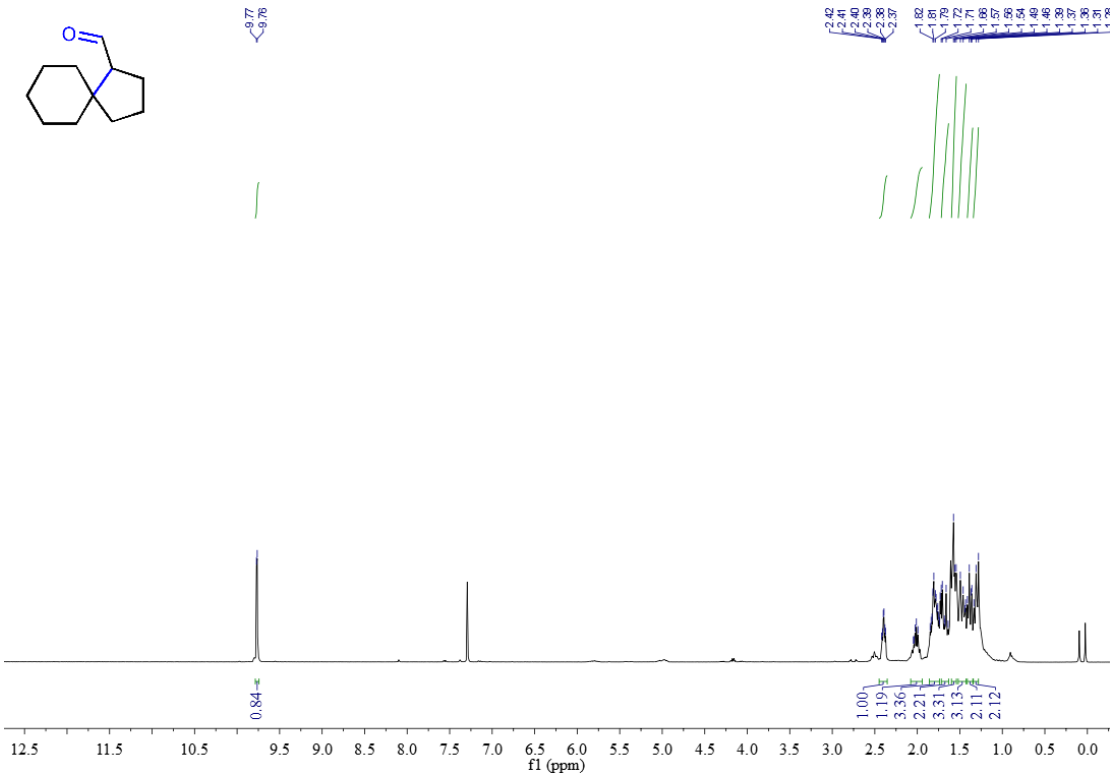
^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **6h**



^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **6h**



^{1}H NMR spectrum (400 MHz, $CDCl_3$, 23 ℃) of **8**



^{13}C NMR spectrum (100 MHz, $CDCl_3$, 23 ℃) of **8**