N-Haloimide Enabled Halogenation via Halogen-Bond-Assisted C-C Activation of Alkanols

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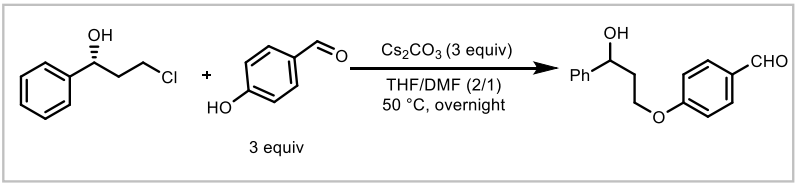
I. General Information

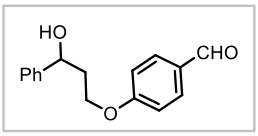
All catalytic reactions were carried out under a nitrogen atmosphere in flame-dried vials sealed with the appropriate caps (up to 8 mL total volume). All anhydrous solvents were commercially supplied. All reagents were obtained from commercial sources (Energy Chemical, Bidepharmatech, or Adamas) and used without further purification unless otherwise stated. Flash column chromatographical purifications were performed using SiO_2 60 (200-300 or 300-400 mesh ASTM). The thin layer chromatography (TLC) was performed using glass plates covered with SiO_2. Spots were visualized by UV light irradiation or by staining of the TLC plate with phosphomolybdic acid stain. Unless otherwise stated, all NMR spectra were recorded on Bruker WH 400 instrument at room temperature (20 ℃ - 25 ℃). Chemical shifts (δ) are reported in parts per million (ppm). Coupling constants (*J*) are given in Hertz (Hz) and refer to apparent multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, hex = hextet, h = heptet, m = multiplet, br s = broad signal, dd = doublet of doublets, ddd = doublet of doublet of doublets). HRMS data were obtained on a Thermo Scientific LTQ Orbitrap Discovery spectrometer (Bremen, Germany). GC-MS spectra were recorded on Agilent 8860-5977B. UV-vis spectra was collected on an Agilent Cary 300 spectrophotometer. Compound names are generated by ChemDraw Professional 18.0 software (PerkinElmer), following the IUPAC nomenclature. The photo reactors (RLH-18-22) used in this study were purchased from Beijing Roger Tech Ltd.

II. Preparation of alkanol substrates



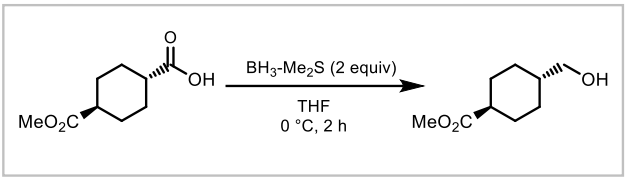
In a dry 100 mL round-bottom flask, hydroxybenzaldehyde (3 equiv) and Cs_2CO_3 (3 equiv) was dissolved in a mixture of anhydrous THF/DMF (2/1). The solution was stirred at 50 °C for 1 h. 3-Chloro-1-phenylpropan-1-ol was added and stirred overnight at 50 °C. After completion of reaction as determined by TLC, the reaction was cooled, diluted with EtOAc (3 × 30mL) and washed with saturated aqueous $NaHCO_3$. The combine organic layers were then washed with 1 M aqueous NaOH (3 × 25 ml), dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification by flash column

chromatography with PE/EA (1/1) as eluent to afford the title compound (0.87 g, 68% yield) as a white solid.

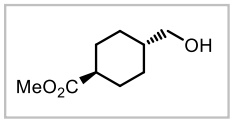


4-(3-hydroxy-3-phenylpropoxy)benzaldehyde

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (s, 1H), 7.85 (d, *J* = 8.7 Hz, 2H), 7.46 – 7.28 (m, 5H), 7.03 (d, *J* = 8.6 Hz, 2H), 5.03 (dd, *J* = 8.3, 4.8 Hz, 1H), 4.33 – 4.26 (m, 1H), 4.18 – 4.21 (m, 1H), 2.33 – 2.20 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.8, 163.9, 144.0, 132.0, 130.1, 128.7, 127.9, 125.8, 114.8, 71.6, 65.5, 38.2. (Known compound*: Eur. J. Med. Chem.* **2016**, *109*, 157).

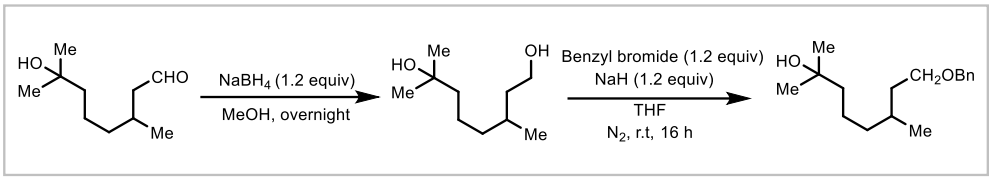


In a dry 100 mL round-bottom flask, trans-4-(methoxycarbonyl)cyclo-hexanecarboxy-lic acid in THF (15 mL) was added a solution of BH_3-Me_2S (2 equiv) dropwise at 0 °C under nitrogen atmosphere. The resulting solution was stirred for 2 h at room temperature. On completion, the reaction was quenched with MeOH (30 mL) at 0 °C. The resulting solution was concentrated under reduced pressure. Purification by flash column chromatography with PE/EA (3/1) as eluent afforded the title compound (0.72 g, 84% yield) as a colorless liquid.

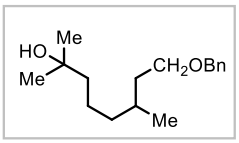


methyl (1r,4r)-4-(hydroxymethyl)cyclohexane-1-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.66 (s, 3H), 3.45 (d, *J* = 6.3 Hz, 2H), 2.45 – 2.21 (m, 1H), 2.09 – 1.97 (m, 2H), 1.89 – 1.83 (m, 3H), 1.53 – 1.38 (m, 3H), 1.05 – 0.93 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 68.2, 51.5, 43.3, 39.7, 28.5, 28.4. (Known compound: *ChemMedChem*. **2016**, *11*, 31).



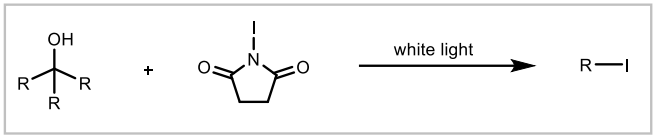
In a dry 100 mL round-bottom flask, 7-hydroxy-3,7-dimethyloctanal (5 mmol) and $NaBH_4$ (1.2 equiv) was dissolved in MeOH (15 mL) at 0 °C. The solution was allowed to warm to room temperature and stirred for 2 h. After completion of reaction as determined by TLC, the reaction was cooled to 0 °C and quenched with saturated NH_4Cl (30 mL). Upon separation of the two layers, the aqueous layer was extracted with EtOAc (3 × 30 mL). The combined organic layers were then dried over Na_2SO_4, filtered, and concentrated under reduced pressure to afford 3,7-dimethyloctane-1,7-diol. In a dry 100 mL round-bottom flask, 3,7-dimethyloctane-1,7-diol (5 mmol), NaH (1.2 equiv) was dissolved in THF (15 mL) under nitrogen atmosphere. After cooling the solution to 0 °C, Benzyl bromide (1.2 equiv) was added dropwise over 1 hour. The solution was allowed to warm to room temperature and stirred for 16 h. After completion of reaction as determined by TLC, the reaction was cooled to 0 °C and quenched with saturated NH_4Cl (30 mL). Upon separation of the two layers, the aqueous layer was extracted with EtOAc (3 × 30 mL). The combined organic layers were then dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification by flash column chromatography with PE/EA (3/1) as eluent to afford the title compound (0.85 g, 64% yield) as a colorless liquid.



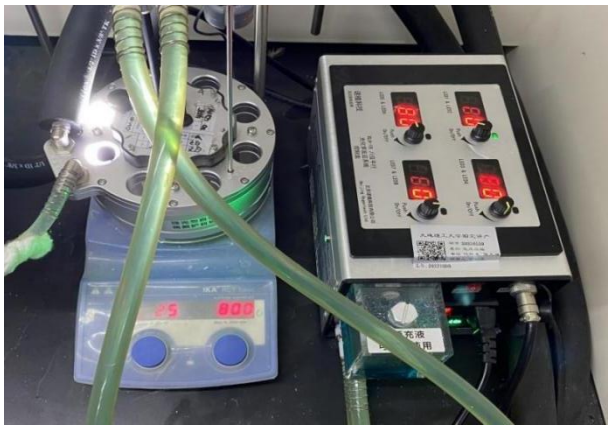
8-(benzyloxy)-2,6-dimethyloctan-2-ol

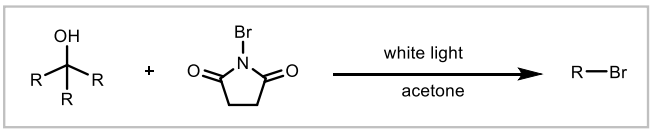
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.34 (m, 4H), 7.32 – 7.28 (m, 1H), 4.52 (s, s, 2H), 3.56 – 3.49 (m, 2H), 1.74 – 1.61 (m, 2H), 1.49 – 1.43 (m, 4H), 1.39 – 1.27 (m, 3H), 1.23 (s, 6H), 0.91 (d, J = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 128.4, 127.6, 127.5, 72.9, 71.0, 68.7, 44.2, 37.6, 36.8, 29.9, 29.3, 29.2, 21.7, 19.7. GCMS (EI): Calcd. For $C_{17}H_{28}O_2$ m/z 264.2, found m/z 264.2 $[M]^+$

III. General procedure for Halogenation

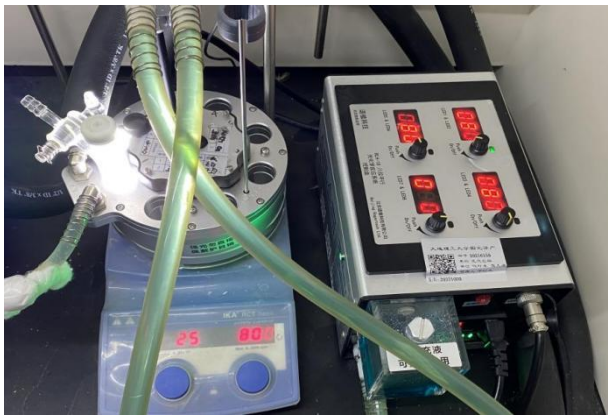


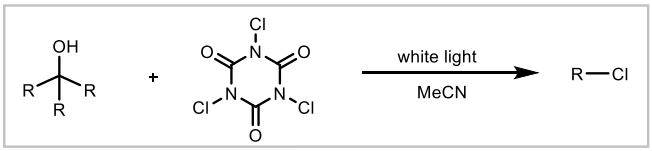
Procedure A: To a dried 8 mL vial was added alkanol (0.3 mmol), 1-iodopyrrolidine -2,5-dione (1.0-1.5 equiv). The vial was transferred to a glovebox, where 3 mL solvent was added. The vial was sealed and stirred under white light for 2 h (8 W, 25 °C). After that, the reaction was concentrated under reduced pressure. Purification by flash column chromatography with PE/EA as an eluent gave the product.



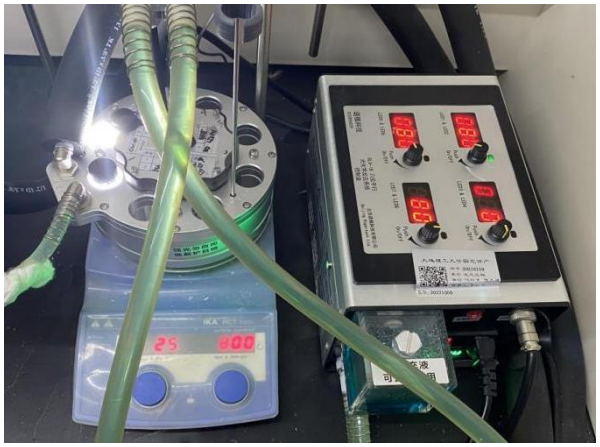


Procedure B: To a Schlenk tube was added the alkanol (0.3 mmol), 1-bromopyrrolidine-2,5-dione (1.2 equiv) and K_3PO_4 (50 mol%) in 3 mL acetone under nitrogen atmosphere. The resulting solution was stirred under white light for 2 h (8 W, 25 °C). After 2 h, additional 1-bromopyrrolidine-2,5-dione (1.2 equiv) was added under nitrogen atmosphere and stirred for 2 h (25 °C). After that, the reaction was concentrated under reduced pressure. Purification by flash column chromatography with PE/EA as an eluent gave the product.

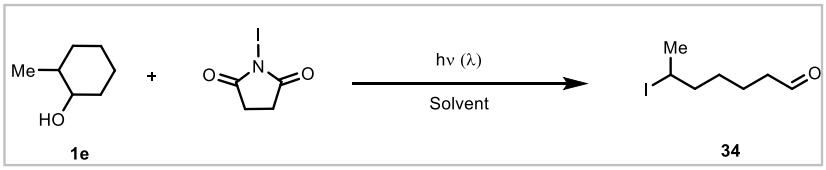




Procedure C: To a dried 8 mL vial was added alkanol (0.3 mmol), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol) and K_3PO_4 (1.5 mmol). The vial was transferred to a glovebox, where 3 mL MeCN was added. The vial was sealed and stirred at white light for 10 h (8 W, 25 °C). After that, the reaction was concentrated under reduced pressure. Purification by flash column chromatography with PE/EA as an eluent gave the product.



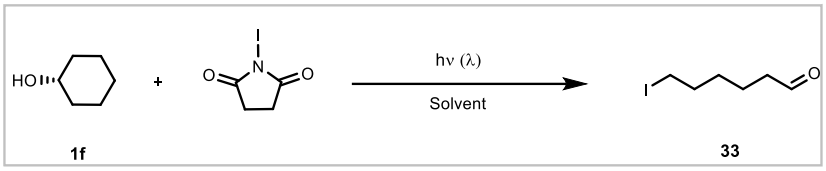
IV. Optimization of conditions



Entry	Additive	Solvent	λ/nm	Yield/%[b]

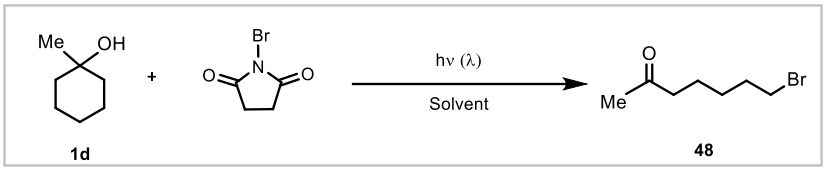
1	K_3PO_4/30%	MeCN	White light	50
2	K_3PO_4/100%	MeCN	White light	43
3	K_2CO_3/30%	MeCN	White light	58
4	Cs_2CO_3/30%	MeCN	White light	38
5	K_2CO_3/30%	acetone	White light	40
6	K_2CO_3/30%	DCE	White light	64
7	K_2CO_3/30%	$PhCF_3$	White light	47
8	K_2CO_3/30%	HFIP	White light	78
9	K_2CO_3/30%	$CHCl_3$	White light	44

[a] Conditions: Alkanol (0.3 mmol), NIS (0.45 mmol), Solvent (3 ml), 25 ˚C, N_2, 2 h. [b] GC relative yield determined with naphthalene as an internal standard.



Entry	Additive	Solvent	λ/nm	Yield/%[b]
1	K_3PO_4/30%	MeCN	White light	23
2	K_3PO_4/60%	MeCN	White light	23
3	K_2CO_3/60%	MeCN	White light	24
4	Cs_2CO_3/60%	MeCN	White light	14
5	K_2CO_3/60%	acetone	White light	< 10
6	K_2CO_3/60%	DCE	White light	60
7	K_2CO_3/60%	$PhCF_3$	White light	< 10
8	K_2CO_3/60%	HFIP	White light	69
9	K_2CO_3/60%	$CHCl_3$	White light	33
10	K_2CO_3/30%	HFIP	White light	< 10
11	K_2CO_3/100%	HFIP	White light	61
12[c]	K_2CO_3/60%	HFIP	White light	79
13[c, d]	K_2CO_3/60%	HFIP	White light	74

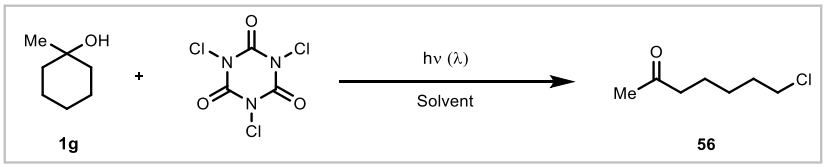
[a] Conditions: Alkanol (0.3 mmol), NIS (0.45 mmol), Solvent (3 ml), 25 ˚C, N_2, 2 h. [b] GC relative yield determined with naphthalene as an internal standard. [c] 4 h. [d] NIS (0.6 mmol).



1d 48

Entry	Additive	Solvent	λ/nm	Yield/%[b]
1	K_3PO_4/30%	MeCN	White light	35
2	K_3PO_4/50%	MeCN	White light	29
3	K_3PO_4/100%	MeCN	White light	25
4	K_2CO_3/30%	MeCN	White light	33
5	Cs_2CO_3/30%	MeCN	White light	23
6	K_3PO_4/30%	acetone	White light	56
7	K_3PO_4/30%	DCE	White light	< 10
8	K_3PO_4/30%	$PhCF_3$	White light	< 10
9	K_3PO_4/30%	HFIP	White light	< 10
10	K_3PO_4/30%	$CHCl_3$	White light	< 10
11[c]	K_3PO_4/30%	acetone	White light	45
12[d]	K_3PO_4/30%	acetone	White light	46
13[e]	K_3PO_4/30%	acetone	White light	89

[a] Conditions: Alkanol (0.3 mmol), NBS (0.45 mmol), Solvent (3 ml), 25 °C, N_2, 4 h. [b] GC relative yield determined with naphthalene as an internal standard. [c] air. [d] 12 h. [e] NBS (0.36 + 0.36 mmol).



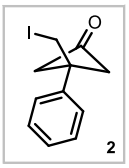
1g 56

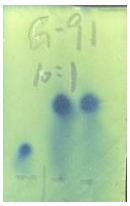
Entry	Additive	Solvent	λ/nm	Yield/%[b]
1	K_3PO_4/30%	MeCN	White light	< 5
2	-	MeCN	White light	N.R.
3	K_3PO_4/200%	MeCN	White light	12
4	K_3PO_4/500%	MeCN	White light	29
5	K_3PO_4/1000%	MeCN	White light	25
6[c]	K_3PO_4/500%	MeCN	White light	23
7[d, e]	K_3PO_4/500%	MeCN	White light	66

8[d, f]	K_3PO_4/500%	MeCN	White light	81
9[d, g]	K_3PO_4/500%	MeCN	White light	75
10[d, f]	K_3PO_4/200%	MeCN	White light	51

[a] Conditions: Alkanol (0.3 mmol), TCCA (0.45 mmol), Solvent (3 ml), 25 °C, N_2, 2 h. [b] GC relative yield determined with naphthalene as an internal standard. [C] 8h. [d] 10 h. [e] 0.15 mmol TCCA. [f] 0.2 mmol TCCA. [g] 0.3 mmol TCCA.

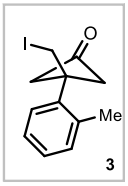
V. Compound characterization



3-(iodomethyl)-3-phenylcyclobutan-1-one 

According to general procedure A: using 3-phenylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 48 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as yellow liquid (83.2 mg, 97% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.39 (m, 2H), 7.39 – 7.29 (m, 3H), 3.69 (s, 2H), 3.63 – 3.54 (m, 2H), 3.49 – 3.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.5, 143.7, 128.6, 127.3, 126.9, 57.7, 38.6, 21.5. HRMS (ESI): Calcd. for $C_{11}H_{11}INaO$ m/z 308.9747, found m/z 308.9747 $[M+Na]^+$

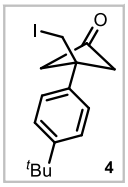


3-(iodomethyl)-3-(*o*-tolyl)cyclobutan-1-one

According to general procedure A: using 3-(*O*-tolyl)bicyclo[1.1.1]pentan-1-ol (0.3 mmol, 52 mg), 1-iodopyrrolidine-2,5-dione (0.3 mmol, 67 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as colorless liquid (82.5 mg, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 7.24 – 7.19 (m, 2H), 3.72 (s, 2H), 3.68 – 3.59 (m, 2H), 3.49 – 3.40 (m, 2H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ

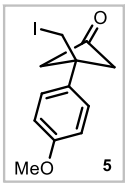
204.4, 140.6, 135.4, 132.0, 128.8, 127.9, 125.9, 58.1, 38.5, 20.4, 18.5. HRMS (ESI): Calcd. for $C_{12}H_{13}INaO$ m/z 322.9903, found m/z 322.9900 $[M+Na]^+$



3-(4-(*tert*-butyl)phenyl)-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-(4-(*tert*-butyl)phenyl)bicyclo[1.1.1]pentan -1-ol (0.3 mmol, 65 mg), 1-iodopyrrolidine-2,5-dione (0.3 mmol, 67 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (90.5 mg, 88% yield).

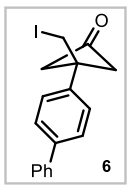
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.42 (m, 2H), 7.29 – 7.24 (m, 2H), 3.70 (s, 2H), 3.60 – 3.53 (m, 2H), 3.47 – 3.39 (m, 2H), 1.37 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.7, 150.2, 140.5 126.6, 125.5, 57.9, 38.2, 34.6, 31.4, 21.5. HRMS (ESI): Calcd. for $C_{15}H_{19}INaO$ m/z 365.0373, found m/z 365.0373 $[M+Na]^+$



3-(iodomethyl)-3-(4-methoxyphenyl)cyclobutan-1-one

According to general procedure A: using 3-(4-methoxyphenyl)bicyclo[1.1.1]pentan-1 -ol (0.3 mmol, 57 mg), 1-iodopyrrolidine-2,5-dione (0.3 mmol, 67 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as white powder (89 mg, 94% yield).

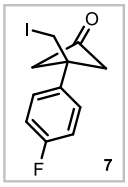
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 2H), 6.96 – 6.90 (m, 2H), 3.84 (s, 3H), 3.68 (s, 2H), 3.58 – 3.49 (m, 2H), 3.44 – 3.34 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 158.7, 135.6, 128.1, 113.9, 57.8, 55.45, 38.1, 21.9. HRMS (ESI): Calcd. for $C_{12}H_{13}INaO_2$ m/z 338.9852, found m/z 338.9853 $[M+Na]^+$



3-([1,1'-biphenyl]-4-yl)-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-([1,1'-biphenyl]-4-yl)bicyclo[1.1.1]pentan-1-ol (0.3 mmol, 71 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as white solid (93.1 mg, 86% yield).

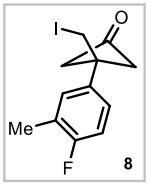
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 – 7.63 (m, 4H), 7.50 (t, *J* = 7.4 Hz, 2H), 7.43 – 7.39 (m, 3H), 3.74 (s, 2H), 3.65 – 3.59 (m, 2H), 3.49 – 3.42 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.3, 142.6, 140.4, 140.2, 128.9, 127.6, 127.4, 127.3, 127.1, 57.8, 38.5, 21.3. HRMS (ESI): Calcd. for $C_{17}H_{15}INaO$ m/z 385.0060, found m/z 385.0061 $[M+Na]^+$



3-(4-fluorophenyl)-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-(4-fluorophenyl)bicyclo[1.1.1]pentan-1-ol (0.3 mmol, 53 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (80.4 mg, 88% yield).

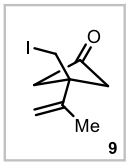
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 2H), 7.13 – 7.06 (m, 2H), 3.67 (s, 2H), 3.59 – 3.51 (m, 2H), 3.45 – 3.36 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.8, 161.9 (d, *J* = 245.1 Hz), 139.5 (d, *J* = 3.1 Hz), 128.7 (d, *J* = 8.1 Hz), 115.4 (d, *J* = 21.3 Hz), 57.8, 38.3, 21.5. ^{19}F NMR (400 MHz, $CDCl_3$) δ -114.9. HRMS (ESI): Calcd. for $C_{11}H_{10}FINaO$ m/z 326.9653, found m/z 326.9657 $[M+Na]^+$



3-(4-fluoro-3-methylphenyl)-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-(4-fluoro-3-methylphenyl)bicyclo[1.1.1] pentan-1-ol (0.3 mmol, 58 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (89.4 mg, 94% yield).

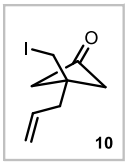
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 – 7.13 (m, 1H), 6.97 – 6.89 (m, 2H), 3.69 (s, 2H), 3.64 – 3.55 (m, 2H), 3.47 – 3.38 (m, 2H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 161.9 (d, *J* = 225.0 Hz), 137.9 (d, *J* = 7.5 Hz), 136.5 (d, *J* =3.1 Hz), 130.6 (d, *J* = 8.5 Hz), 118.4 (d, *J* = 20.9 Hz), 112.5 (d, *J* = 20.8 Hz), 58.1, 38.2, 20.5, 18.6. ^{19}F NMR (400 MHz, $CDCl_3$) δ -115.4. HRMS (ESI): Calcd. for $C_{12}H_{12}FINaO$ m/z 340.9809, found m/z 340.9816 $[M+Na]^+$



3-(iodomethyl)-3-(prop-1-en-2-yl)cyclobutan-1-one

According to general procedure A: using 3-(prop-1-en-2-yl)bicyclo[1.1.1]pentan-1-ol (0.3 mmol, 37 mg), 1-iodopyrrolidine-2,5-dione (0.3 mmol, 67 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as white powder (62.7 mg, 84% yield).

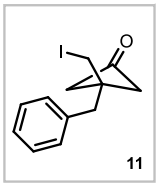
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.14 (s, 1H), 4.92 (s, 1H), 3.58 (s, 2H), 3.32 – 3.24 (m, 2H), 3.04 – 2.96 (m, 2H), 1.82 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.7, 144.7, 114.1, 55.5, 39.9, 18.7, 16.9. HRMS (ESI): Calcd. for $C_8H_{11}INaO$ m/z 272.9747, found m/z 272.9750 $[M+Na]^+$



3-allyl-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-allylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 37 mg), 1-iodopyrrolidine-2,5-dione (0.3 mmol, 67 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as yellow solid (65.9 mg, 88% yield).

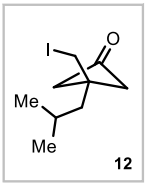
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.79 – 5.67 (m, 1H), 5.32 – 5.19 (m, 2H), 3.50 (s, 2H), 2.95 (s, 4H), 2.54 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 132.7, 119.7, 55.6, 42.4, 33.3, 18.0. HRMS (ESI): Calcd. for $C_8H_{11}INaO$ m/z 272.9747, found m/z 272.9750 $[M+Na]^+$



3-benzyl-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-benzylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 52 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid (82.8 mg, 92% yield).

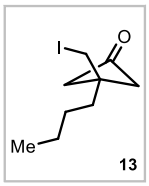
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 5H), 3.44 (s, 2H), 3.14 – 3.07 (m, 4H), 2.97 – 2.90 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8, 137.3, 129.5, 128.7, 127.2, 55.7, 43.3, 34.9, 18.6. HRMS (ESI): Calcd. for $C_{12}H_{13}INaO$ m/z: 322.9903, found m/z 322.9910 $[M+Na]^+$



3-(iodomethyl)-3-isobutylcyclobutan-1-one

According to general procedure A: using 3-isobutylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 42 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 100/1) gave the title compound as colorless liquid (73.4 mg, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.51 (s, 2H), 2.96 (s, 4H), 1.75 – 1.69 (m, 3H), 0.97 (d, J = 6.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 57.5, 47.1, 33.2, 25.6, 23.2, 17.5. HRMS (ESI): Calcd. for $C_9H_{15}INaO$ m/z 289.0060, found m/z 289.0061 $[M+Na]^+$



3-butyl-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-butylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 42 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification

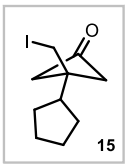
by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (75.1 mg, 94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.50 (s, 2H), 2.98 – 2.91 (m, 2H), 2.90 – 2.82 (m, 2H), 1.80 – 1.73 (m, 2H), 1.44 – 1.34 (m, 2H), 1.28 – 1.20 (m, 2H), 0.95 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7, 56.0, 38.4, 33.7, 27.1, 22.8, 17.9, 14.1. HRMS (ESI): Calcd. for $C_9H_{15}INaO$ m/z 289.0060, found m/z 289.0064 $[M+Na]^+$

3-cyclopropyl-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-cyclopropylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 37 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as yellow liquid. (68.3mg, 91% yield).

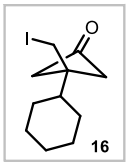
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.61 (s, 2H), 2.92 – 2.82 (m, 2H), 2.72 – 2.63 (m, 2H), 1.38 – 1.28 (m, 1H), 0.66 – 0.58 (m, 2H), 0.28 – 0.20 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.1, 53.6, 33.9, 19.9, 17.5, 1.9. HRMS (ESI): Calcd. for $C_8H_{11}INaO$ m/z 272.9747, found m/z 272.9751 $[M+Na]^+$



3-cyclopentyl-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-cyclopentylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 46 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (73.3 mg, 88% yield).

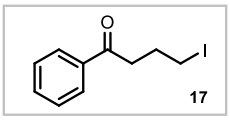
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.58 – 3.51 (m, 2H), 3.01 – 2.80 (m, 4H), 2.54 – 2.42 (m, 1H), 1.86 – 1.76 (m, 2H), 1.66 (s, 4H), 1.22 – 1.08 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.1, 53.5, 44.9, 36.1, 28.1, 25.8, 20.7. HRMS (ESI): Calcd. for $C_{10}H_{15}INaO$ m/z 301.0060, found m/z 301.0063 $[M+Na]^+$



3-cyclohexyl-3-(iodomethyl)cyclobutan-1-one

According to general procedure A: using 3-cyclohexylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 50 mg), 1-iodopyrrolidine-2,5-dione (0.36 mmol, 81 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (79 mg, 90% yield).

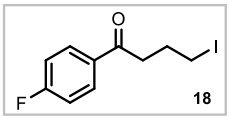
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.57 (s, 2H), 3.04 – 2.96 (m, 2H), 2.91 – 2.83 (m, 2H), 1.87 – 1.80 (m, 2H), 1.80 – 1.68 (m, 4H), 1.37 – 1.24 (m, 2H), 1.20 – 1.10 (tt, *J* = 13.0, 3.4 Hz, 1H), 1.09 – 0.96 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.1, 54.3, 43.1, 36.3, 27.5, 26.4, 26.1, 18.3. HRMS (ESI): Calcd. for $C_{11}H_{17}INaO$ m/z 315.0216, found m/z 315.0220 $[M+Na]^+$



4-iodo-1-phenylbutan-1-one

According to general procedure A: using 1-phenylcyclobutan-1-ol (0.3 mmol, 44 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 120/1) gave the title compound as yellow liquid (58.4 mg, 71% yield).

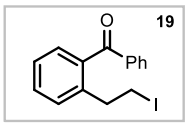
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.95 (m, 2H), 7.64 – 7.57 (m, 1H), 7.56 – 7.44 (m, 2H), 3.36 (t, *J* = 6.6 Hz, 2H), 3.17 (t, *J* = 6.9 Hz, 2H), 2.35 – 2.24 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.7, 136.7, 133.3, 128.7, 128.1, 38.9, 27.5, 6.9. (Known compound: *Org. Lett.* **2016**, *18*, 684).



1-(4-fluorophenyl)-4-iodobutan-1-one

According to general procedure A: using 1-(4-fluorophenyl)cyclobutan-1-ol (0.3 mmol, 50 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 70/1) gave the title compound as yellow liquid (69.8 mg, 80% yield).

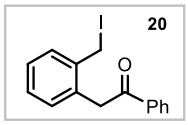
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 7.96 (m, 2H), 7.16 (t, *J* = 8.5 Hz, 2H), 3.34 (t, *J* = 6.5 Hz, 2H), 3.13 (t, *J* = 6.9 Hz, 2H), 2.33 – 2.22 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 165.8 (d, *J* = 253.4 Hz), 133.2, 130.7 (d, *J* = 9.3 Hz), 115.8 (d, *J* = 21.7 Hz), 38.8, 27.4, 6.8. ^{19}F NMR (400 MHz, $CDCl_3$) δ -104.9. (Known compound: *Angew. Chem. Int. Ed.* **2020**, *59*, 8099).



(2-(2-iodoethyl)phenyl)(phenyl)methanone

According to general procedure A: using 1-phenyl-2,3-dihydro-*1H*-inden-1-ol (0.3 mmol, 63 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 70/1) gave the title compound as yellow liquid (60.5 mg, 60% yield).

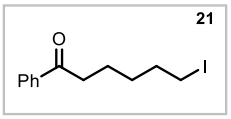
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.7 Hz, 2H), 7.65 – 7.59 (m, 1H), 7.53 – 7.45 (m, 3H), 7.41 – 7.33 (m, 3H), 3.43 – 3.35 (m, 2H), 3.32 – 3.24 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.0, 139.9, 138.1, 137.7, 133.4, 130.8, 130.7, 130.4, 129.5, 128.5, 126.3, 37.9, 5.6. HRMS (ESI): Calcd. for $C_{15}H_{13}INaO$ m/z 358.9903, found m/z 358.9907 $[M+Na]^+$



2-(2-(iodomethyl)phenyl)-1-phenylethan-1-one

According to general procedure A: using 2-phenyl-2,3-dihydro-*1H*-inden-2-ol (0.3 mmol, 63 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 70/1) gave the title compound as white powder (70.2 mg, 70% yield).

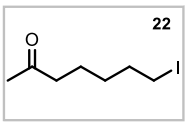
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 7.4 Hz, 2H), 7.68 – 7.61 (m, 1H), 7.57 – 7.51 (m, 2H), 7.45 – 7.37 (m, 1H), 7.30 – 7.25 (m, 2H), 7.19 – 7.09 (m, 1H), 4.48 (s, 2H), 4.44 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 137.6 136.6, 133.5, 131.5, 130.1, 128.8, 128.7, 128.5, 127.9, 42.7, 5.2 (one signal is missing due to overlapping). HRMS (ESI): Calcd. for $C_{15}H_{13}INaO$ m/z 358.9903, found m/z 358.9906 $[M+Na]^+$



6-iodo-1-phenylhexan-1-one

According to general procedure A: using 1-phenylcyclohexan-1-ol (0.3 mmol, 53 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as yellow liquid (85.2 mg, 94% yield).

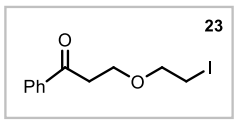
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.94 (m, 2H), 7.60 – 7.54 (m, 1H), 7.50 – 7.44 (m, 2H), 3.21 (t, *J* = 7.0 Hz, 2H), 3.00 (t, *J* = 7.2 Hz, 2H), 1.94 – 1.84 (m, 2H), 1.82 – 1.73 (m, 2H), 1.55 – 1.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 136.9, 133.0, 128.6, 128.0, 38.3, 33.4, 30.2, 23.1, 6.8. (Known compound: *Org. Lett.* **2018**, *20*, 1228).



7-iodoheptan-2-one

According to general procedure A: using 1-methylcyclohexan-1-ol (0.3 mmol, 34 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as yellow liquid (69.1 mg, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.15 (t, *J* = 7.0 Hz, 2H), 2.42 (t, *J* = 7.3 Hz, 2H), 2.10 (s, 3H), 1.85 – 1.75 (m, 2H), 1.61 – 1.50 (m, 2H), 1.41 – 1.31 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.6, 43.3, 33.2, 29.9, 22.6, 6.8 (one signal is missing due to overlapping). (Known compound: *Angew. Chem. Int. Ed.* **2001**, *40*, 3389).

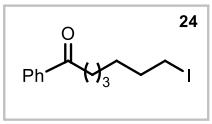


3-(2-iodoethoxy)-1-phenylpropan-1-one

According to general procedure A: using 4-phenyltetrahydro-*2H*-pyran-4-ol (0.3 mmol, 53 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid. (82.5 mg, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 – 7.92 (m, 2H), 7.63 – 7.55 (m, 1H), 7.53 – 7.43 (m, 2H), 3.95 (t, *J* = 6.4 Hz, 2H), 3.77 (t, *J* = 6.8 Hz, 2H), 3.34 – 3.22 (m, 4H). ^{13}C

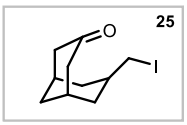
NMR (100 MHz, $CDCl_3$) δ 198.2 136.9, 133.3, 128.7, 128.2, 71.8, 66.0, 38.7, 3.0. (Known compound: *Org. Chem. Front.* **2022**, *9*, 3692).



7-iodo-1-phenylpentan-1-one

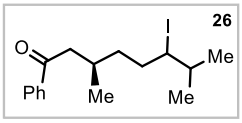
According to general procedure A: using 1-phenylcycloheptan-1-ol (0.3 mmol, 57 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as white solid (82.1 mg, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.95 (m, 2H), 7.59 – 7.54 (m, 1H), 7.50 – 7.44 (m, 2H), 3.20 (t, *J* = 7.0 Hz, 2H), 2.99 (t, *J* = 7.3 Hz, 2H), 1.89 – 1.72 (m, 4H), 1.51 – 1.37 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.3, 137.0, 133.0, 128.6, 128.1, 38.4, 33.3, 30.4, 28.2, 24.0, 7.4. (Known compound: *Chem. Commun.* **2020,** *56*, 5002).



7-(iodomethyl)bicyclo[3.3.1]nonan-3-one

According to general procedure A: using 1-Adamantanol (0.3 mmol, 46 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg) and 3 mL MeCN. The resulting solution was stirred for 12 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as white powder (57.6 mg, 69% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.00 (d, *J* = 7.0 Hz, 2H), 2.55 (s, 2H), 2.49 – 2.41 (m, 2H), 2.35 – 2.27 (m, 2H), 2.27 – 2.16 (m, 2H), 1.93 – 1.75 (m, 2H), 1.67 – 1.59 (m, 1H), 0.98 – 0.88 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 212.1, 50.3, 34.6, 32.3, 28.51, 28.4, 14.1. (Known compound: *J. Med. Chem.* **2017**, *60*, 1716).

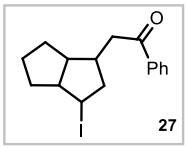


(3*R*)-6-iodo-3,7-dimethyl-1-phenyloctan-1-one

According to general procedure A: using (1S,2R,5R)-5-methyl-2-(1-methylethyl)-1-phenylcyclohexanol (0.3 mmol, 70 mg, dr > 20:1), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash

column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid. The dr was determined by ^{1}H NMR (100.5 mg, 94% yield, dr = 1.9:1).

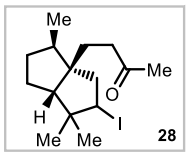
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.94 (m, 2H), 7.60 – 7.54 (m, 1H), 7.51 – 7.44 (m, 2H), 4.18 – 4.09 (m, 1H), 2.98 (t, J = 5.4 Hz, 0.34 H), 2.94 (t, J = 5.4 Hz, 0.64 H), 2.85 (dd, J = 7.7, 3.9 Hz, 0.64 H), 2.81 (dd, J = 7.7, 3.9 Hz, 0.34 H), 2.28 – 2.17 (m, 1H), 2.06 – 1.92 (m, 1H), 1.84 – 1.62 (m, 2H), 1.57 – 1.49 (m, 1H), 1.28 – 1.22 (m, 1H), 1.03 – 0.97 (m, 6H), 0.97 – 0.92 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 137.3, 133.0, 128.6, 128.1, 52.3, 45.8, 37.3, 36.1, 35.0, 29.0, 23.0, 20.0, 19.8. HRMS (ESI): Calcd. for $C_{16}H_{23}INaO$ m/z 381.0686, found m/z 381.0688 $[M+Na]^+$



2-(3-iodooctahydropentalen-1-yl)-1-phenylethan-1-one

According to general procedure A: using 5-phenyloctahydro-*1H*-4,7-methanoinden -5-ol (0.3 mmol, 68 mg, dr > 20:1), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 100/1) gave the title compound as colorless liquid. The dr and rr ratios were determined by ^{1}H NMR (99.1 mg, 93% yield, dr = 1.6:1, rr = 1.7:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06-7.89 (m, 2H), 7.66-7.59 (m, 1H), 7.54-7.41 (m, 2H), 4.68 – 4.45 (m, 0.27H), 3.73-3.59 (m, 0.46H), 3.47 (dt, J = 11.3, 6.9 Hz, 0.29H), 3.38 (dd, J = 9.7, 5.2 Hz, 0.27H), 3.24 – 2.88 (m, 2.5H), 2.69 – 2.18 (m, 2.7H), 2.07-1.88 (m, 1.4H), 1.88 – 1.45 (m, 6.9H). ^{13}C NMR (major) (100 MHz, $CDCl_3$) δ 199.5, 137.0, 133.1, 128.7, 128.1, 56.7, 49.5, 47.6, 43.8, 36.3, 33.1, 32.4, 30.8, 24.8. HRMS (ESI): Calcd. for $C_{16}H_{19}INaO$ m/z 377.0373, found m/z 377.0382 $[M+Na]^+$

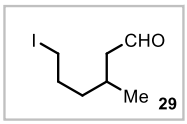


4-((3a*S*,4*R*,6a*R*)-2-iodo-1,1,4-trimethylhexahydropentalen-3a(1*H*)-yl)butan-2-one

According to general procedure A: using cedrol (0.3 mmol, 67 mg), 1-iodopyrroli-

dine-2,5-dione (0.45 mmol, 101 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as yellow liquid. The dr ratio was determined by ^{1}H NMR (84.6 mg, 81% yield, dr = 1.2:1).

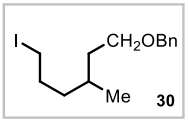
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.05 – 3.99 (m, 1H), 2.50 – 2.36 (m, 2H), 2.20 – 2.14 (m, 3H), 2.10 – 1.93 (m, 2H), 1.89 – 1.80 (m, 2H), 1.70 – 1.63 (m, 2H), 1.51 – 1.45 (m, 1H), 1.29 – 1.10 (m, 2H), 1.05 (s, 1H), 0.99 – 0.83 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.8, 208.6, 54.5, 53.6, 47.5, 45.9, 44.3, 40.9, 40.0, 35.5, 31.0, 28.8, 28.5, 24.6, 23.4, 14.3. HRMS (ESI): Calcd. for $C_{15}H_{25}INaO$ m/z 371.0842, found m/z 371.0845 $[M+Na]^+$



6-iodo-3-methylhexanal

According to general procedure A: using 7-hydroxy-3,7-dimethyloctanal (0.3 mmol, 52 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid. (34.4 mg, 48% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 – 9.75 (m, 1H), 3.23 – 3.16 (m, 2H), 2.48 – 2.40 (m, 1H), 2.35 – 2.25 (m, 1H), 2.19 – 2.05 (m, 1H), 1.96 – 1.77 (m, 2H), 1.51 – 1.43 (m, 1H), 1.39 – 1.30 (m, 1H), 1.02 – 0.97 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 50.9, 37.6, 31.0, 27.3, 19.9, 6.6. HRMS (ESI): Calcd. for $C_7H_{13}INaO$ m/z 262.9903, found m/z 262.9912 $[M+Na]^+$

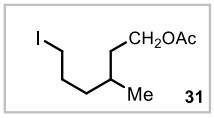


(((6-iodo-3-methylhexyl)oxy)methyl)benzene

According to general procedure A: using 8-(benzyloxy)-2,6-dimethyloctan-2-ol (0.3 mmol, 79 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification

by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as yellow liquid (73.1 mg, 73% yield).

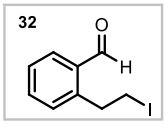
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 5H), 4.54 (s, 2H), 3.59 – 3.49 (m, 2H), 3.24 – 3.14 (m, 2H), 1.97 – 1.78 (m, 2H), 1.74 – 1.62 (m, 2H), 1.51 – 1.40 (m, 2H), 1.30 – 1.21 (m, 1H), 0.93 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 128.4, 127.7, 127.6, 73.0, 68.4, 37.8, 36.6, 31.2, 29.2, 19.6, 7.5. HRMS (ESI): Calcd. for $C_{14}H_{21}INaO$ m/z 355.0529, found m/z 355.0530 $[M+Na]^+$



6-iodo-3-methylhexyl acetate

According to general procedure A: using 7-hydroxy-3,7-dimethyloctyl acetate (0.3 mmol, 65 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid (53.2 mg, 62% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.28 – 3.99 (m, 2H), 3.19 (td, *J* = 7.0, 2.6 Hz, 2H), 2.06 (s, 3H), 1.95 – 1.76 (m, 2H), 1.71 – 1.56 (m, 2H), 1.52 – 1.40 (m, 2H), 1.31 – 1.24 (m, 1H), 0.94 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 62.8, 37.7, 35.4, 31.0, 29.1, 21.0, 19.4, 7.0. HRMS (ESI): Calcd. for $C_9H_{18}IO_2$ m/z 285.0346, found m/z 285.0342 $[M+H]^+$

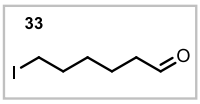


2-(2-iodoethyl)benzaldehyde

According to general procedure A: using 2,3-dihydro-*1H*-inden-1-ol (0.3 mmol, 40 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/DCM = 10/1) gave the title compound as yellow liquid (53.4 mg, 68% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.63 – 7.48 (m, 2H), 7.34 (d, *J* = 7.4 Hz, 1H), 3.60 (t, *J* = 7.4 Hz, 2H), 3.41 (t, *J* = 7.4 Hz, 2H). ^{13}C

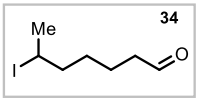
NMR (100 MHz, $CDCl_3$) δ 192.8, 142.2, 134.5, 133.9, 133.7, 131.7, 127.7, 37.2, 5.3. HRMS (ESI): Calcd. for C_9H_9INaO m/z 282.9590, found m/z 282.9593 $[M+Na]^+$



6-iodohexanal

According to general procedure A: using cyclohexanol (0.3 mmol, 30 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as colorless liquid (53.3 mg, 79% yield).

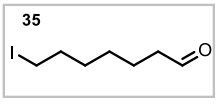
^1H NMR (400 MHz, $CDCl_3$) δ 9.80 (t, *J* = 1.6 Hz, 1H), 3.21 (t, *J* = 6.9 Hz, 2H), 2.48 (dt, *J* = 1.6, 7.3 Hz, 2H), 1.91 – 1.82 (m, 2H), 1.72 – 1.63 (m, 2H), 1.51 – 1.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.2, 43.6, 33.1, 30.0, 21.0, 6.4 (Known compound: *Science*, **2018,** *362*, 225).



6-iodoheptanal

According to general procedure A: using 2-methylcyclohexan-1-ol (0.3 mmol, 34 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (30 mol%, 12.4 mg) and 3 mL HFIP. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid (54.6 mg, 76% yield).

^1H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 4.22 – 4.13 (m, 1H), 2.47 (t, *J* = 7.2 Hz, 2H), 1.92 (d, *J* = 6.8 Hz, 3H), 1.88 – 1.80 (m, 1H), 1.71 – 1.60 (m, 3H), 1.57 – 1.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.3, 43.7, 42.5, 29.9, 29.3, 28.9, 21.1 (Known compound: *Chem. Eur. J.* **2004**, *10*, 4206).

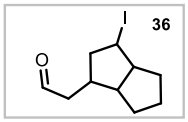


7-iodoheptanal

According to general procedure A: using cycloheptanol (0.3 mmol, 34 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column

chromatography (eluent: PE/EA = 30/1) gave the title compound as colorless liquid (53.8 mg, 75% yield).

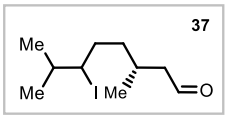
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 3.19 (t, *J* = 6.9 Hz, 2H), 2.45 (t, *J* = 7.3 Hz, 2H), 1.88 – 1.79 (m, 2H), 1.71 – 1.60 (m, 2H), 1.48 – 1.32 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.6, 43.8, 33.2, 30.2, 28.0, 21.8, 7.0 (Known compound: *PCT Int. Appl.* **2021,** 2021051034).



2-(3-iodooctahydropentalen-1-yl)acetaldehyde

According to general procedure A: using octahydro-*1H*-4,7-methanoinden-5-ol (0.3 mmol, 46 mg, dr > 20:1), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (30 mol%, 12.4 mg) and 3 mL HFIP. Purification by flash column chromatography (eluent: PE/EA = 100/1) gave the title compound as colorless liquid. The dr and rr ratios were determined by ^{1}H NMR (50.8 mg, 61% yield, dr = 4.5:1, rr = 6.2:1).

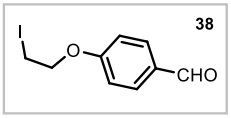
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.10-9.77 (m, 0.8H), 9.65 (d, *J* = 3.0 Hz, 0.13H), 4.51 (q, *J* = 7.1 Hz, 0.15H), 3.68 – 3.57 (m, 0.64H), 3.36 (dd, *J* = 9.8, 5.1 Hz, 0.15H), 3.24 – 3.14 (m, 0.15H), 3.00 – 2.71 (m, 0.92H), 2.65 (dd, *J* = 16.7, 5.3 Hz, 0.77H), 2.60 – 2.44 (m, 2H), 2.44 – 2.21 (m, 0.81H), 2.20 – 2.07 (m, 1.19H), 2.04 – 1.75 (m, 2H), 1.68 – 1.43 (m, 5.38H). ^{13}C NMR (major) (100 MHz, $CDCl_3$) δ 201.5, 56.5, 49.4, 49.1, 47.2, 40.8, 32.3, 30.8, 28.8, 24.7. HRMS (ESI): Calcd. for $C_{10}H_{15}INaO$ m/z 301.0060, found m/z 301.0069 $[M+Na]^+$



(3R)-6-iodo-3,7-dimethyloctanal

According to general procedure A: using (-)-Menthol (0.3 mmol, 47 mg, dr > 20:1), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (30 mol%, 12.4 mg) and 3 mL HFIP. Purification by flash column chromatography (eluent: PE/EA = 100/1) gave the title compound as colorless liquid. The dr was determined by ^{1}H NMR (54.8 mg, 65% yield, dr = 2.0:1).

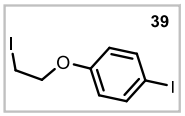
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 4.15 – 4.09 (m, 1H), 2.48 – 2.25 (m, 2H), 2.17 – 2.07 (m, 1H), 2.01 – 1.90 (m, 1H), 1.78 – 1.61 (m, 2H), 1.55 – 1.45 (m, 1H), 1.29 – 1.25 (m, 1H), 1.02 – 0.98 (m, 6H), 0.97 – 0.94 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 51.8, 51.0, 37.0, 36.0, 34.9, 27.6, 23.1, 20.1, 19.8. HRMS (ESI): Calcd. for $C_{10}H_{19}INaO$ m/z 305.0373, found m/z 305.0377 $[M+Na]^+$



4-(2-iodoethoxy)benzaldehyde

According to general procedure A: using 4-(3-hydroxy-3-phenylpropoxy)benzaldehyde (0.3 mmol, 77 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as yellow powder (72.4 mg, 87% yield).

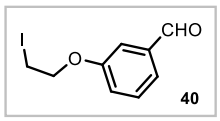
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.90 (s, 1H), 7.85 (d, *J* = 7.3 Hz, 2H), 7.02 (d, *J* = 7.6 Hz, 2H), 4.34 (t, *J* = 6.7 Hz, 2H), 3.46 (t, *J* = 6.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.8, 162.9, 132.1, 130.4, 114.9, 68.7, 0.2. (Known compound: *Org. Lett.* **2021**, *23*, 4002).



1-iodo-4-(2-iodoethoxy)benzene

According to general procedure A: using 3-phenoxy-1-phenylpropan-1-ol (0.3 mmol, 68 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 150/1) gave the title compound as white solid (45.8 mg, 41% yield).

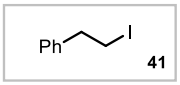
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.9 Hz, 2H), 6.71 (d, *J* = 8.9 Hz, 2H), 4.23 (t, *J* = 6.8 Hz, 2H), 3.43 (t, *J* = 6.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 138.4, 117.2, 83.6, 68.7, 0.8. (Known compound: *J. Med. Chem.* **2011**, *54*, 6563).



3-(2-iodoethoxy)benzaldehyde

According to general procedure A: using 3-(3-hydroxy-3-phenylpropoxy)benzaldehyde (0.3 mmol, 77 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as yellow liquid (54.6 mg, 66% yield).

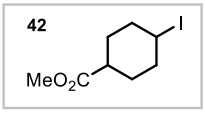
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.99 (s, 1H), 7.54 – 7.45 (m, 2H), 7.39 (s, 1H), 7.25 – 7.17 (m, 1H), 4.32 (t, *J* = 6.7 Hz, 2H), 3.46 (t, *J* = 6.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.0, 158.5, 137.8, 130.3, 124.2, 122.1, 112.9, 68.7, 0.7. HRMS (ESI): Calcd. for $C_9H_{10}IO_2$ m/z 276.9720, found m/z 276.9717 $[M+H]^+$



(2-iodoethyl)benzene

According to general procedure A: using 1-(4-fluorophenyl)-3-phenylpropan-1-ol (0.3 mmol, 69 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), K_2CO_3 (60 mol%, 24.9 mg) and 3 mL HFIP. The resulting solution was stirred for 4 h at 25 °C. Purification by flash column chromatography (eluent: PE) gave the title compound as colorless liquid (45.5 mg, 65% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.33 (m, 2H), 7.33 - 7.28 (m, 1H), 7.23 (d, *J* = 7.0 Hz, 2H), 3.39 (t, *J* = 7.8 Hz, 2H), 3.22 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 128.7, 128.4, 126.9, 40.4, 5.8. (Known compound: *React. Chem. Eng*. **2022**, *7*, 1650).

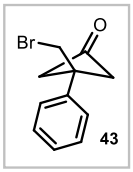


methyl 4-iodocyclohexane-1-carboxylate

According to general procedure A: using trans-methyl-4-(hydroxymethyl)cyclohex ane-1-carboxylate (0.3 mmol, 52 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg), and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA =

50/1) gave the title compound as colorless liquid. The dr was determined by ^{1}H NMR (25.4 mg, 32% yield, dr = 2.0:1).

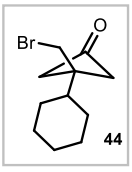
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.71-4.63 (m, 0.3 H), 4.21-4.10 (m, 0.61 H), 3.72 (s, 0.93 H), 3.68 (s, 1.89 H), 2.50-2.36 (m, 2.18 H), 2.19-2.10 (m, 0.67 H), 2.05 – 1.88 (m, 3.21H), 1.85 – 1.72 (m, 1.35H), 1.62 – 1.49 (m, 1.28 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.7, 51.7, 41.4, 38.8, 35.9, 30.6 (Known compound*: Angew. Chem. Int. Ed.* **2018**, *57*, 5492).



3-(bromomethyl)-3-phenylcyclobutan-1-one

According to general procedure B: using 3-phenylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 48 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 20/1) gave the title compound as colorless liquid (65.8 mg, 92% yield).

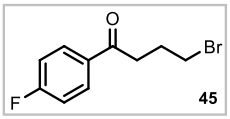
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.40 (m, 2H), 7.38 – 7.30 (m, 3H), 3.79 (s, 2H), 3.58 – 3.45 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.7, 143.2, 128.6, 127.4, 127.1, 56.9, 44.2, 38.9. HRMS (ESI): Calcd. for $C_{11}H_{11}BrNaO$ m/z 260.9885, found m/z 260.9888 $[M+Na]^+$



3-(bromomethyl)-3-cyclohexylcyclobutan-1-one

According to general procedure B: using 3-cyclohexylbicyclo[1.1.1]pentan-1-ol (0.3 mmol, 50 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid (66.4 mg, 91% yield).

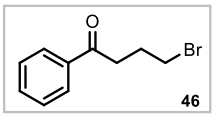
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.74 – 3.66 (m, 2H), 3.01 – 2.92 (m, 4H), 1.87 – 1.72 (m, 6H), 1.33 – 1.24 (m, 2H), 1.19 – 1.03 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 53.6, 43.1, 41.0, 36.9, 27.6, 26.5, 26.1. HRMS (ESI): Calcd. for $C_{11}H_{17}BrNaO$ m/z 267.0355, found m/z 267.0358 $[M+Na]^+$



4-bromo-1-(4-fluorophenyl)butan-1-one

According to general procedure B: using 1-(4-fluorophenyl)cyclobutan-1-ol (0.3 mmol, 50 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 80/1) gave the title compound as colorless liquid (67.9 mg, 93% yield).

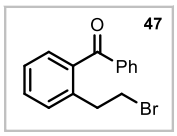
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 7.95 (m, 2H), 7.23 – 7.07 (m, 2H), 3.64 – 3.51 (m, 2H), 3.25 – 3.11 (m, 2H), 2.40 – 2.25 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.2, 165.8 (d, *J* = 254.7 Hz), 133.2 (d, *J* = 3.0 Hz), 130.7 (d, *J* = 9.2 Hz), 115.8 (d, *J* = 21.9 Hz), 36.5, 33.6, 26.8. ^{19}F NMR (400 MHz, $CDCl_3$) δ -105.0 (Known compound*: Green Chem.* **2020**, *22*, 4357).



4-bromo-1-phenylbutan-1-one

According to general procedure B: using 1-phenylcyclobutan-1-ol (0.3 mmol, 44 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol,128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as yellow liquid (63.9 mg, 94% yield).

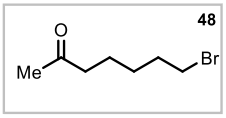
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.4 Hz, 2H), 7.63 – 7.56 (m, 1H), 7.53 – 7.46 (m, 2H), 3.57 (t, *J* = 6.3 Hz, 2H), 3.21 (t, *J* = 6.9 Hz, 2H), 2.39 – 2.28 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.8, 136.7, 133.3, 128.7, 128.0, 36.6, 33.7, 26.9. (Known compound*: Org. Lett.* **2018**, *20*, 1228).



(2-(2-bromoethyl)phenyl)(phenyl)methanone

According to general procedure B: using 1-phenyl-2,3-dihydro-*1H*-inden-1-ol (0.3 mmol, 63 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (100 mol%, 63.7 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 100/1) gave the title compound as yellow liquid (56.9 mg, 66% yield).

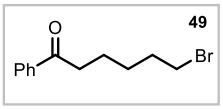
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.80 (m, 2H), 7.66 – 7.59 (m, 1H), 7.52 – 7.47 (m, 3H), 7.44 – 7.40 (m, 1H), 7.39 – 7.32 (m, 2H), 3.62 (t, *J* = 7.4 Hz, 2H), 3.28 (t, *J* = 7.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 138.4, 138.3, 137.6, 133.4, 131.2, 130.6, 130.4, 129.4, 128.5, 126.3, 36.9, 33.1. HRMS (ESI): Calcd. for $C_{15}H_{14}BrO$ m/z 289.0223, found m/z 289.0212 $[M+H]^+$



7-bromoheptan-2-one

According to general procedure B: using 1-methylcyclohexan-1-ol (0.3 mmol, 34 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (30 mol%, 19.1 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as colorless liquid (51.5 mg, 89% yield).

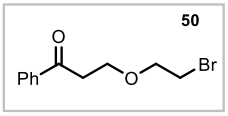
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.42 (t, *J* = 6.7 Hz, 2H), 2.46 (t, *J* = 7.2 Hz, 2H), 2.15 (s, 3H), 1.92 – 1.83 (m, 2H), 1.66 – 1.56 (m, 2H), 1.49 – 1.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.8, 43.4, 33.6, 32.5, 30.0, 27.7, 22.8 (Known compound*: J. Org. Chem.* **2021**, *86*, 16177).



6-bromo-1-phenylhexan-1-one

According to general procedure B: using 1-phenylcyclohexan-1-ol (0.3 mmol, 53 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as colorless liquid (54.6 mg, 72% yield).

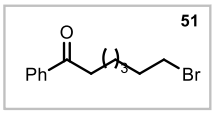
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 – 7.91 (m, 2H), 7.62 – 7.54 (m, 1H), 7.53 – 7.43 (m, 2H), 3.45 (t, *J* = 6.7 Hz, 2H), 3.02 (t, *J* = 7.3 Hz, 2H), 2.00 – 1.88 (m, 2H), 1.85 – 1.74 (m, 2H), 1.61 – 1.50 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 136.9, 133.0, 128.6, 128.0 38.3, 33.7, 32.6, 27.9, 23.3 (Known compound*: J. Org. Chem.* **2021**, *86*, 16177).



3-(2-bromoethoxy)-1-phenylpropan-1-one

According to general procedure B: using 4-phenyltetrahydro-*2H*-pyran-4-ol (0.3 mmol, 53 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 30/1) gave the title compound as colorless liquid (54.6 mg, 71% yield).

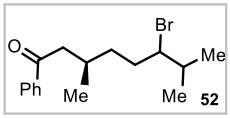
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 7.89 (m, 2H), 7.65 – 7.55 (m, 1H), 7.55 – 7.44 (m, 2H), 3.97 (t, *J* = 6.4 Hz, 2H), 3.84 (t, *J* = 6.1 Hz, 2H), 3.48 (t, *J* = 6.1 Hz, 2H), 3.31 (t, *J* = 6.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 136.9, 133.3, 128.7, 128.1, 71.1, 66.3, 38.7, 30.4 (Known compound*: J. Org. Chem.* **2021**, *86*, 16177).



7-bromo-1-phenylheptan-1-one

According to general procedure B: using 1-phenylcycloheptan-1-ol (0.3 mmol, 57 mg), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid (61.7 mg, 77% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.94 (m, 2H), 7.61 – 7.55 (m, 1H), 7.52 – 7.45 (m, 2H), 3.44 (t, *J* = 6.8 Hz, 2H), 3.00 (t, *J* = 7.3 Hz, 2H), 1.95 – 1.86 (m, 2H), 1.83 – 1.73 (m, 2H), 1.57 – 1.39 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.3, 137.0, 133.0, 128.6, 128.1, 38.4, 33.9, 32.6, 28.5, 28.0, 24.0 (Known compound*: Org. Chem. Front.* **2022**, *9*, 3692).

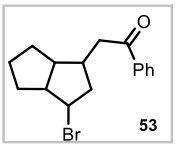


(3*R*)-6-bromo-3,7-dimethyl-1-phenyloctan-1-one

According to general procedure B: using (1S,2R,5R)-5-methyl-2-(1-methylethyl)-1-phenylcyclohexanol (0.3 mmol, 70 mg, dr > 20:1), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. After adding each portion of 1-bromopyrrolidine-2,5-dione, the resulting solution was stirred at 25 °C for 3.5 h (totally 7 h). Purification by flash column chromatography (eluent: PE/DCM

= 5/1) gave the title compound as colorless liquid. The dr ratio was determined by ^{1}H NMR (64.5 mg, 58% yield, dr = 1.7:1).

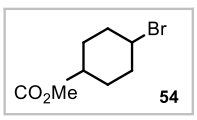
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 7.7 Hz, 2H), 7.61 – 7.55 (m, 1H), 7.51 – 7.45 (m, 2H), 4.08 – 3.94 (m, 1H), 2.97 (ddd, J = 16.2, 5.9, 2.6 Hz, 1H), 2.89 – 2.79 (m, 1H), 2.28 – 2.17 (m, 1H), 1.97 – 1.81 (m, 3H), 1.77 – 1.65 (m, 1H), 1.58 – 1.51 (m, 1H), 1.06 – 0.97 (m, 9H). ^{13}C NMR (major) (100 MHz, $CDCl_3$) δ 200.0, 137.4, 133.0, 128.6, 128.1, 67.2, 46.0, 35.5, 34.6, 34.2, 29.2, 21.0, 19.8, 17.9. HRMS (ESI): Calcd. for $C_{16}H_{23}BrNaO$ m/z 333.0824 found m/z 333.0825 $[M+Na]^+$



2-(3-bromooctahydropentalen-1-yl)-1-phenylethan-1-one

According to general procedure B: using 5-phenyloctahydro-*1H*-4,7-methanoinden-5-ol (0.3 mmol, 68 mg, dr > 20:1), 1-bromopyrrolidine-2,5-dione (0.36 + 0.36 mmol, 128 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as yellow liquid. The dr and rr ratios were determined by ^{1}H NMR. (71.7 mg, 78% yield, dr = 1.9:1, rr = 2.8:1).

1H NMR (400 MHz, $CDCl_3$) δ 8.05-7.92 (m, 1H), 7.64-7.54 (m, 1H), 7.53-7.45 (m, 2H), 4.58 (q, J = 6.1 Hz, 0.2H), 3.76 (ddd, J = 10.7, 7.9, 5.7 Hz, 0.55H), 3.55 (dd, J = 9.9, 5.5 Hz, 0.29H), 3.51 – 3.44 (m, 0.28H), 3.44-3.35 (m, 0.34H), 3.31 – 2.91 (m, 1.8H), 2.86 – 2.66 (m, 0.8H), 2.60 – 2.18 (m, 2.5H), 2.17 – 1.90 (m, 1.1H), 1.91 – 1.41 (m, 7.2H). ^{13}C NMR (major) (100 MHz, $CDCl_3$) δ 199.5, 137.0, 133.2, 128.7, 128.1, 54.7, 54.5, 49.4, 45.2, 44.1, 41.3, 32.3, 31.0, 25.1. HRMS (ESI): Calcd. for $C_{16}H_{19}BrNaO$ m/z 329.0511, found m/z 329.0513 $[M+Na]^+$

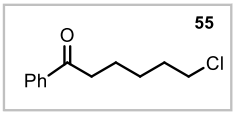


methyl 4-bromocyclohexane-1-carboxylate

According to general procedure B: using trans-methyl-4-(hydroxymethyl)cyclohexane-1-carboxylate (0.3 mmol, 52 mg), 1-bromopyrrolidine-2,5-dione (0.45 mmol, 77.8 mg), K_3PO_4 (50 mol%, 31.8 mg) and 3 mL acetone. The resulting solution was stirred

for 2 h at 25 °C. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid. The dr ratio was determined by ^{1}H NMR (20.5 mg, 31% yield, dr = 2.4:1).

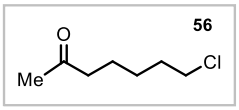
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.58-4.45 (m, 0.7 H), 4.13-3.94 (m, 0.26 H), 3.72 (s, 2.0 H), 3.69 (s, 0.91), 2.46 – 2.32 (m, 1.48 H), 2.17-2.00 (m, 3.58 H), 1.97-1.76 (m, 3.63 H), 1.66-1.53 (m, 0.4 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.3, 52.3, 51.7, 41.4, 34.1, 24.6. HRMS (ESI): Calcd. for $C_8H_{14}BrO_2$ m/z 221.0172, found m/z 221.0171 $[M+H]^+$



6-chloro-1-phenylhexan-1-one

According to general procedure C: using 1-phenylcyclohexan-1-ol (0.3 mmol, 53 mg), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol, 46.5 mg), K_3PO_4 (1.5 mmol, 318.4 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 40/1) gave the title compound as colorless liquid (59.4 mg, 94% yield).

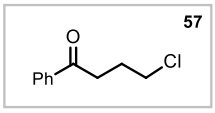
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.92 (m, 2H), 7.60 – 7.54 (m, 1H), 7.50 – 7.44 (m, 2H), 3.57 (t, *J* = 6.6 Hz, 2H), 3.00 (t, *J* = 7.3 Hz, 2H), 1.87 – 1.75 (m, 4H), 1.60 – 1.50 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.0, 137.0, 133.0, 128.6, 128.0, 44.9, 38.3, 32.5, 26.6, 23.5. (Known compound*: J. Org. Chem.* **2013**, *78*, 9181).



7-chloroheptan-2-one

According to general procedure C: using 1-methylcyclohexan-1-ol (0.3 mmol, 34 mg), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol, 46.5 mg), K_3PO_4 (1.5 mmol, 318.4 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 25/1) gave the title compound as colorless liquid (35.1 mg, 79% yield).

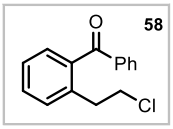
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.55 (t, *J* = 6.6 Hz, 2H), 2.47 (t, *J* = 7.3 Hz, 2H), 2.16 (s, 3H), 1.83 – 1.76 (m, 2H), 1.65 – 1.59 (m, 2H), 1.49 – 1.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.7, 44.8, 43.4, 32.4, 29.9, 26.4, 23.0. (Known compound*: Angew. Chem. Int. Ed.* **2021**, *60*, 7132).



4-chloro-1-phenylbutan-1-one

According to general procedure C: using 1-phenylcyclobutan-1-ol (0.3 mmol, 44 mg), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol, 46.5 mg), K_3PO_4 (1.5 mmol, 318.4 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless liquid (38.8 mg, 71% yield).

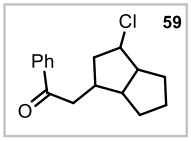
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.98 (m, 2H), 7.62 – 7.56 (m, 1H), 7.52 – 7.47 (m, 2H), 3.70 (t, *J* = 6.2 Hz, 2H), 3.21 (t, *J* = 6.9 Hz, 2H), 2.31 – 2.21 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.0, 136.8, 133.2, 128.7, 128.0, 44.7, 35.3, 26.8. (Known compound*: Org. Lett.* **2019**, *21*, 9241).



(2-(2-chloroethyl)phenyl)(phenyl)methanone

According to general procedure C: using 1-phenyl-2,3-dihydro-*1H*-inden-1-ol (0.3 mmol, 63 mg), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol, 46.5 mg), K_3PO_4 (1.5 mmol, 318.4 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 75/1) gave the title compound as colorless liquid (44.7 mg, 61% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.78 (m, 2H), 7.65 – 7.59 (m, 1H), 7.52 – 7.45 (m, 3H), 7.45 – 7.41 (m, 1H), 7.40 – 7.32 (m, 2H), 3.77 (t, *J* = 7.2 Hz, 2H), 3.19 (t, *J* = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 138.6, 137.7, 137.5, 133.4, 131.4, 130.6, 130.4, 129.3, 128.5, 126.3, 45.1, 36.6. HRMS (ESI): Calcd. for $C_{15}H_{14}ClO$m/z 245.0728, found m/z 245.0726 $[M+H]^+$

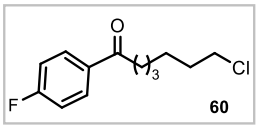


2-(3-chlorooctahydropentalen-1-yl)-1-phenylethan-1-one

According to general procedure C: using 5-phenyloctahydro-*1H*-4,7-methanoinden -5-ol (0.3 mmol, 68 mg, dr > 20:1), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol, 46.5 mg), K_3PO_4 (1.5 mmol, 318.4 mg) and 3 mL MeCN. Purification by flash

column chromatography (eluent: PE/EA = 50/1) gave the title compound as yellow liquid. The dr and rr ratios were determined by ^{1}H NMR (68.1 mg, 87% yield, dr = 1.5:1, rr = 1.9:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.93 (m, 1.95H), 7.65-7.55 (m, 1H), 7.54-7.40 (m, 2H), 4.49 (dt, J = 6.9, 5.1 Hz, 0.22H), 3.76 (ddd, J = 10.1, 7.2, 5.7 Hz, 0.41H), 3.66 (dd, J = 10.7, 5.6 Hz, 0.33H), 3.59 – 3.41 (m, 0.67H), 3.29 – 2.91 (m, 1.59H), 2.84 – 2.59 (m, 0.73H), 2.50 – 1.98 (m, 3.1H), 1.90 – 1.41 (m, 7H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 137.1, 133.1, 128.7, 128.1, 64.2, 54.0, 50.3, 49.2, 48.1, 44.2, 40.7, 31.1, 25.2. HRMS (ESI): Calcd. for $C_{16}H_{20}ClO$ m/z 263.1197, found m/z 263.1192 $[M+H]^+$

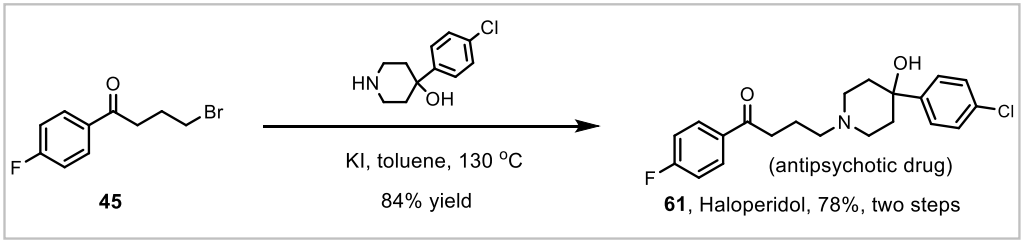


7-chloro-1-(4-fluorophenyl)heptan-1-one

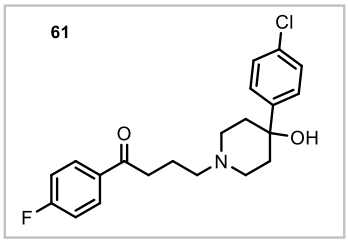
According to general procedure C: using 1-(4-fluorophenyl)cycloheptan-1-ol (0.3 mmol, 62 mg), 1,3,5-trichloro-1,3,5-triazinane-2,4,6-trione (0.2 mmol, 46.5 mg), K_3PO_4 (1.5 mmol, 318.4 mg) and 3 mL MeCN. Purification by flash column chromatography (eluent: PE/EA = 100/1) gave the title compound as colorless liquid (57.4 mg, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.94 (m, 2H), 7.14 (t, *J* = 8.5 Hz, 2H), 3.55 (t, *J* = 6.7 Hz, 2H), 2.96 (t, *J* = 7.3 Hz, 2H), 1.85 – 1.72 (m, 4H), 1.54 – 1.38 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.6, 165.7 (d, *J* = 254.3 Hz), 133.5 (d, *J* = 3.0 Hz), 130.6 (d, *J* = 9.4 Hz), 115.7 (d, *J* = 21.8 Hz), 45.0, 38.3, 32.4, 28.5, 26.7, 24.0. ^{19}F NMR (400 MHz, $CDCl_3$) δ -105.4. HRMS (ESI): Calcd. for $C_{13}H_{17}ClFO$ m/z 243.0946, found m/z 243.0946 $[M+H]^+$

VI. Application of the methodology

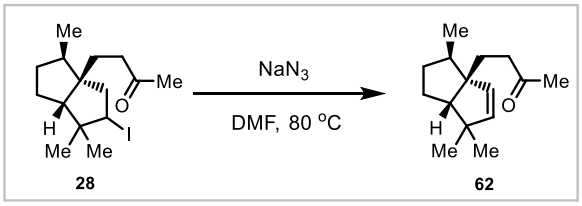


The compound **45** (0.3 mmol, 73 mg), 4-(4-chloro-phenyl) piperidin-4-ol (0.6 mmol, 127 mg), and KI (0.009 mmol, 1.5 mg,) were dissolved in anhydrous toluene (3.0 mL). The reaction mixture was stirred at 130 °C for 60 h. After completion of the reaction, the solvent was removed under reduced pressure. Purification by flash column chromatography (eluent: EA/TEA) gave the title compound as white solid (94.7 mg, 84% yield).

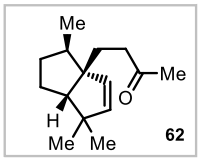


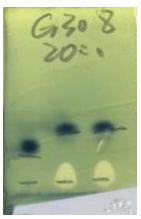
4-(4-(4-chlorophenyl)-4-hydroxypiperidin-1-yl)-1-(4-fluorophenyl)butan-1-one

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 – 7.94 (m, 2H), 7.45 – 7.27 (m, 4H), 7.24 – 7.09 (m, 2H), 3.08 – 2.93 (m, 2H), 2.80 (d, *J* = 8.1 Hz, 2H), 2.58 – 2.38 (m, 4H), 2.10 – 1.94 (m, 4H), 1.74 – 1.65 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.4, 166.9, 164.3, 147.0, 133.7 (d, *J* = 3.0 Hz), 132.7, 130.7 (d, *J* = 9.3 Hz), 128.4, 126.1, 115.6 (d, *J* = 21.8 Hz), 71.1, 57.9, 49.3, 38.4, 36.3, 21.9. ^{19}F NMR (400 MHz, $CDCl_3$) δ -105.6 (Known compound*: J. Am. Chem. Soc.* **2019**, *141*, 5034).



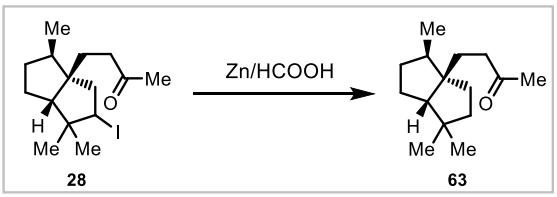
The compound **28** (0.2 mmol, 70 mg) was dissolved in DMF (6 ml) and NaN_3(2 mmol, 130 mg) was added. The reaction mixture was stirred for 48 h at 80 °C. After completion of the reaction the mixture was poured onto 15 ml water and extracted with DCM for three times. The combined organic phases were dried over Na_2SO_4 and concentrated under reduced pressure. Purification by flash column chromatography (eluent: PE/EA = 70/1) gave the title compound as colorless solid (41.8 mg, 95% yield).



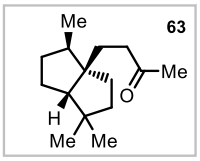


4-((3*R*,3a*R*,6a*S*)-3,6,6-trimethyl-2,3,6,6a-tetrahydropentalen-3a(1*H*)-yl)butan-2-one

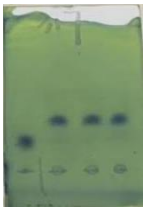
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39 – 5.23 (m, 2H), 2.44 – 2.34 (m, 2H), 2.15 (s, 3H), 1.93 – 1.88 (m, 1H), 1.86 – 1.64 (m, 5H), 1.59 – 1.56 (m, 1H), 1.33 – 1.28 (m, 1H), 1.08 (s, 3H), 0.99 – 0.91 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.8, 140.7, 133.7, 63.5, 56.3, 46.8, 43.4, 40.8, 34.5, 31.9, 30.1, 28.5, 26.0, 25.5, 14.9. HRMS (ESI): Calcd. for $C_{15}H_{25}O$ m/z 221.1900, found m/z 221.1894 $[M+H]^+$



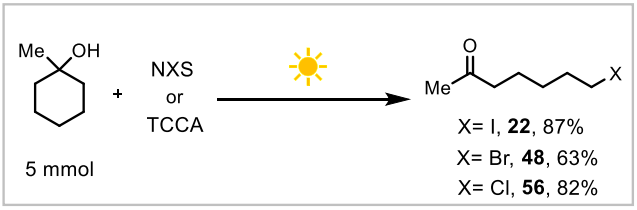
The compound **28** (0.1 mmol, 35 mg) with activated zinc powder (1.5 mmol, 98 mg) in HCOOH (10 mL) at Ambient temperature for 10 h. Quench the reaction with saturated $NaHCO_3$ and extract with DCM for three times. Dry the combine organic layers over Na_2SO_4 and concentrate under reduced pressure. Purification by flash column chromatography (eluent: PE/EA = 50/1) gave the title compound as colorless solid (21.3 mg, 96% yield).



4-((3a*R*,4*R*,6a*S*)-1,1,4-trimethylhexahydropentalen-3a(1*H*)-yl)butan-2-one

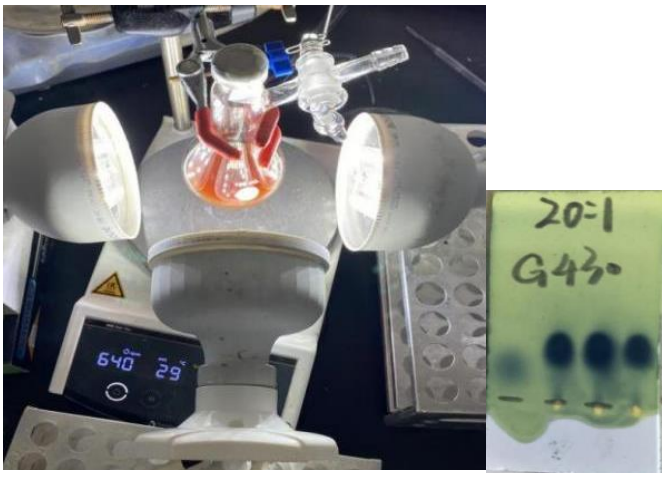


^{1}H NMR (400 MHz, $CDCl_3$) δ 2.52 – 2.44 (m, 2H), 2.17 (s, 3H), 1.70 – 1.59 (m, 5H), 1.57 – 1.44 (m, 3H), 1.36 – 1.27 (m, 3H), 1.25 – 1.18 (m, 1H), 0.98 – 0.91 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.9, 58.7, 54.6, 45.4, 41.2, 41.0, 39.2, 34.8, 34.5, 30.9, 30.0, 29.7, 27.6, 25.2, 14.3 (Known compound*: J. Am. Chem. Soc.* **2019**, *141*, 1457).

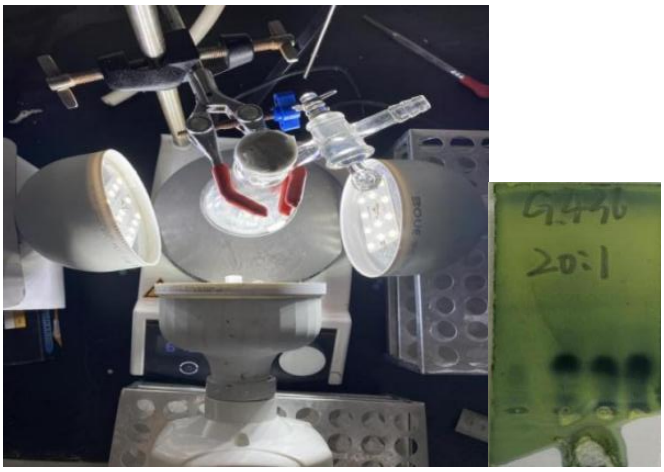


In a dry 100 mL round-bottom flask was added 1-methylcyclohexan-1-ol (5 mmol), 1-iodopyrrolidine-2,5-dione (7.5 mmol) in 20 mL MeCN under nitrogen atmosphere. The resulting solution was stirred for 2 h at room temperature under three white lights (30 W, see the apparatus below). On completion, the resulting solution was

concentrated under reduced pressure. Purification by flash column chromatography with PE/EA (30/1) as eluent gave the compound **22** as yellow liquid (1.04 g, 87% yield).

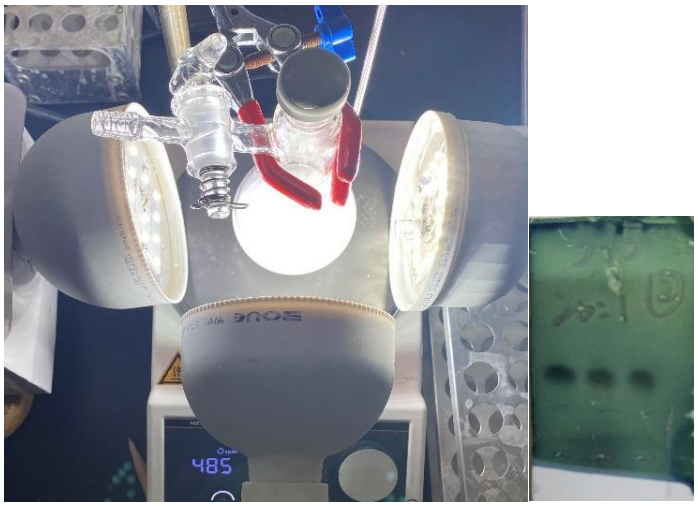


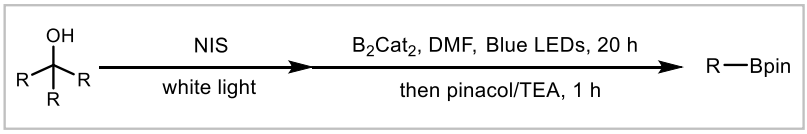
In a dry 100 mL round-bottom flask was added 1-methylcyclohexan-1-ol (5 mmol), 1-bromopyrrolidine-2,5-dione (6 mmol), K_3PO_4 (30 mol%) in 20 mL acetone under nitrogen atmosphere. The resulting solution was stirred for 2 h at room temperature under three white lights (30 W, see the apparatus below). After 2 h, 1-bromopyrrolidine-2,5-dione (6 mmol) was added under nitrogen atmosphere and stirred for additional 2 h. On completion, the resulting solution was concentrated under reduced pressure. Purification by flash column chromatography with PE/EA (30/1) as eluent gave the compound **48** as yellow liquid (0.61 g, 63% yield).



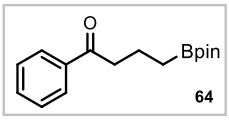
In a dry 100 mL round-bottom flask was added 1-methylcyclohexan-1-ol (5 mmol), TCCA (3.3 mmol), K_3PO_4 (25 mmol) in 50 mL MeCN under nitrogen atmosphere. The resulting solution was stirred for 8 h at room temperature under three white lights (30 W, see the apparatus below). After that, TCCA (2.5 mmol) was added under nitrogen

atmosphere and stirred for 12 h. On completion, the resulting solution was concentrated under reduced pressure to remove MeCN. Then quenched with saturated NH_4Cl (30 mL). Upon separation of the two layers, the aqueous layer was extracted with DCM (3 × 30 mL). The combined organic layers were then dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification by flash column chromatography with PE/EA (25/1) as eluent gave the compound **56** as yellow liquid (0.61 g, 82% yield).





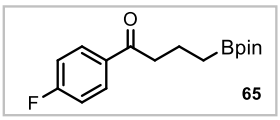
To a Schlenk tube was added the alkanol (0.3 mmol), 1-iodopyrrolidine-2,5-dione (0.45 mmol). The Schlenk tube was transferred to a glovebox, where 3 mL MeCN was added. Under nitrogen atmosphere, the reaction mixture was stirred and irradiated at white light for 2 h. On the occasion that the iodination step completed, removing MeCN, adding B_2Cat_2 (0.9 mmol) and 3 ml DMF. The reaction mixture was stirred under 455 nm irradiation at room temperature for 20 h. Then a solution of pinacol (2 mmol) in triethylamine (1 mL) was added to the mixture. After 1 hour, water (15 mL) was added and the aqueous layer was extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The product was purified by flash column chromatography on silica gel with PE/EA as eluent gave the corresponding product.



1-phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butan-1-one 

Following the general procedure using 1-phenylcyclobutan-1-ol (0.3 mmol, 44 mg) provided the product **64** (35.4 mg, 43% yield) as a colorless oil after purification by flash chromatography (eluent: PE/EA = 20/1).

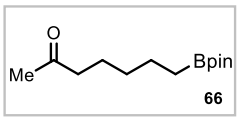
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.96 (m, 2H), 7.59 – 7.54 (m, 1H), 7.50 – 7.44 (m, 2H), 2.97 (t, *J* = 7.5 Hz, 2H), 1.91 – 1.85 (m, 2H), 1.27 (s, 12H), 0.93 – 0.89 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.6, 137.1, 132.8, 128.5, 128.2, 83.1, 41.0, 24.9, 19.3. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.4 (Known compound*: J. Org. Chem.* **2009**, *74*, 3196).



1-(4-fluorophenyl)-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butan-1-one

Following the general procedure using 1-(4-fluorophenyl)cyclobutan-1-ol (0.3 mmol, 50 mg) provided the product **65** (43.8 mg, 50% yield) as a colorless oil after purification by flash chromatography (eluent: PE/EA = 25/1).

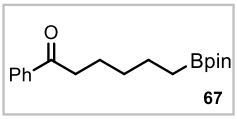
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.6, 5.6 Hz, 2H), 7.14 (t, *J* = 8.6 Hz, 2H), 2.97 (t, *J* = 7.5 Hz, 2H), 1.91- 1.83 (m, 2H), 1.27 (s, 12H), 0.90 (t, *J* = 7.7 Hz, 2H). ^{13}CNMR (100 MHz, $CDCl_3$) δ 199.0, 165.6 (d, *J* = 253.9 Hz), 133.5 (d, *J* = 3.0 Hz), 130.8 (d, *J* = 9.3 Hz), 115.6 (d, *J* = 21.8 Hz), 83.1, 40.9, 24.8, 19.3. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.4. ^{19}F NMR (400 MHz, $CDCl_3$) δ -105.9 (Known compound*: J. Org. Chem.* **2009**, *74*, 3196).



7-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptan-2-one

Following the general procedure using 1-methylcyclohexan-1-ol (0.3 mmol, 34 mg) provided the product **66** (54.7 mg, 76% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 20/1).

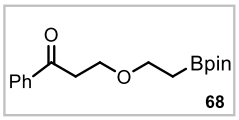
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.40 (t, *J* = 7.2 Hz, 2H), 2.17 – 2.07 (m, 3H), 1.61 – 1.51 (m, 2H), 1.44 – 1.36 (m, 2H), 1.31 – 1.26 (m, 2H), 1.23 (s, 12H), 0.75 (t, *J* = 7.1 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.4, 82.9, 43.7, 31.8, 29.8, 24.8, 23.7 (d, *J* = 5.0 Hz). ^{11}B NMR (128 MHz, $CDCl_3$) δ 33.9. (Known compound*: Org. Lett.* **2020**, *22*, 7213).



1-phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexan-1-one

Following the general procedure using 1-phenylcyclohexan-1-ol (0.3 mmol, 53 mg) provided the product **67** (72 mg, 79% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 20/1).

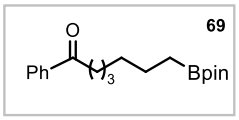
^{1}H NMR (400 MHz, CDCl3) δ 7.96 (d, J = 8.0 Hz, 2H), 7.58 – 7.53 (m, 1H), 7.49 – 7.43 (m, 2H), 2.97 (t, J = 7.4 Hz, 2H), 1.79 – 1.71 (m, 2H), 1.50 – 1.37 (m, 4H), 1.25 (s, 12H), 0.80 (t, J = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, CDCl3) δ 200.6, 137.1, 132.9, 128.6, 128.1, 82.9, 38.6, 32.1, 24.8, 24.12, 23.8. ^{11}B NMR (128 MHz, CDCl3) δ 32.5. (Known compound: *Org. Lett.* **2019**, 21, 2477).

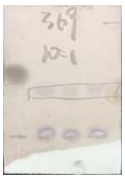


1-phenyl-3-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethoxy)propan-1-one

Following the general procedure using 4-phenyltetrahydro-*2H*-pyran-4-ol (0.3 mmol, 53 mg) provided the product **68** (62.9 mg, 69% yield) as a yellow liquid after purification by flash chromatography (eluent: PE/EA = 15/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.4 Hz, 2H), 7.60 – 7.53 (m, 1H), 7.49 – 7.43 (m, 2H), 3.87 (t, *J* = 6.7 Hz, 2H), 3.63 (t, *J* = 7.9 Hz, 2H), 3.26 (t, *J* = 6.7 Hz, 2H), 1.24 (s, 12H), 0.93 – 0.83 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 137.0, 133.1, 128.6, 128.1, 83.2, 67.8, 65.7, 38.9, 24.78. ^{11}B NMR (128 MHz, $CDCl_3$) δ 33.2 (Known compound*: Org. Lett.* **2019**, *21*, 2477).

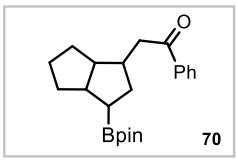




1-phenyl-7-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptan-1-one

Following the general procedure using 1-phenylcycloheptan-1-ol (0.3 mmol, 57 mg) provided the product **69** (77.7 mg, 82% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 30/1).

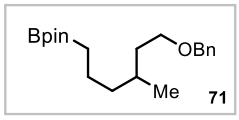
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.95 (m, 2H), 7.59 – 7.54 (m, 1H), 7.50 – 7.45 (m, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.78 – 1.72 (m, 2H), 1.47 – 1.35 (m, 6H), 1.26 (s, 12H), 0.79 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.7, 137.1, 132.9, 128.6, 128.1, 82.9, 38.7, 32.2, 29.2, 24.9, 24.4, 23.9. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.38 (Known compound*: Org. Lett.* **2019**, *21*, 2477).



1-phenyl-2-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)octahydropentalen-1-yl)ethan-1-one

Following the general procedure using 5-phenyloctahydro-*1H*-4,7-methanoinden-5-ol (0.3 mmol, 68 mg) provided the product **70** (84.7 mg, 80% yield, rr = 3.0:1) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 35/1).

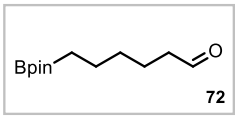
^{1}H NMR (400 MHz, Chloroform-d) δ 8.15 – 7.88 (m, 2H), 7.59-7.52 (m, 1H), 7.50-7.41 (m, 2H), 3.39 (ddd, J = 12.1, 8.0, 5.9 Hz, 0.25H), 3.23 – 2.92 (m, 1.61H), 2.92 – 2.75 (m, 0.29H), 2.61-2.30 (m, 0.88H), 2.36 – 1.68 (m, 3.5H), 1.72 – 1.36 (m, 4.3H), 1.24 (s, 12H), 1.17 – 0.78 (m, 3.2H). ^{13}C NMR (major) (100 MHz, Chloroform-d) δ 200.7, 137.3, 128.6, 128.2, 82.8, 50.7, 46.6, 46.0, 44.3, 37.5, 33.3, 32.3, 25.0, 24.8, 24.7. HRMS (ESI): Calcd. for $C_{22}H_{32}BO_3$ m/z 355.2439, found m/z 355.2432 $[M+H]^+$



2-(6-(benzyloxy)-4-methylhexyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane

Following the general procedure using 8-(benzyloxy)-2,6-dimethyloctan-2-ol (0.3 mmol, 79 mg) provided the product **71** (41.8 mg, 42% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 80/1).

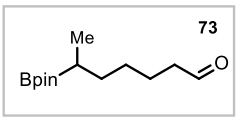
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.31 (m, 5H), 4.53 – 4.51 (m, 2H), 3.54 – 3.49 (m, 2H), 1.74 – 1.55 (m, 3H), 1.49 – 1.38 (m, 4H), 1.26 (s, 12H), 0.90 (d, *J* = 6.6 Hz, 3H), 0.78 (t, *J* = 7.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 128.4, 127.6, 127.5, 82.9, 72.9, 68.8, 40.0, 36.8, 29.8, 24.9, 21.4, 19.7. ^{11}B NMR (128 MHz, $CDCl_3$) δ 33.9. HRMS (ESI): Calcd. for $C_{20}H_{34}BO_3$ m/z 333.2596, found m/z 333.2588 $[M+H]^+$



6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanal

Following the general procedure using cyclohexanol (0.3 mmol, 30 mg) provided the product **72** (21.1 mg, 31% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 30/1).

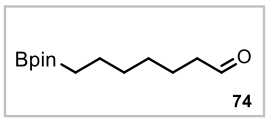
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (t, *J* = 1.8 Hz, 1H), 2.43 (td, *J* = 7.4, 1.8 Hz, 2H), 1.69 – 1.63 (m, 4H), 1.46 – 1.43 (m, 2H), 1.26 (s, 12H), 0.80 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.1, 82.9, 43.8, 31.8, 24.8, 23.7, 21.9. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.4 (Known compound*: Angew. Chem. Int. Ed.* **2018**, *57*, 16832).



6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptanal

Following the general procedure using 2-methylcyclohexan-1-ol (0.3 mmol, 34 mg) provided the product **73** (27.4 mg, 38% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 30/1).

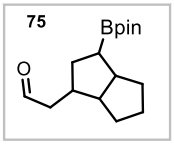
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (t, *J* = 1.7 Hz, 1H), 2.44 (td, *J* = 7.4, 1.8 Hz, 2H), 1.67 – 1.60 (m, 3H), 1.39 – 1.33 (m, 4H), 1.25 (s, 12H), 0.99 – 0.96 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 82.9, 43.9, 32.8, 28.5, 24.7 (d, *J* = 4.6 Hz), 22.3, 15.5. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.6. HRMS (ESI): Calcd. for $C_{13}H_{26}BO_3$ m/z 241.1970, found m/z 241.1964 $[M+H]^+$



7-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptanal

Following the general procedure using cycloheptanol (0.3 mmol, 34 mg) provided the product **74** (30.9 mg, 43% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 25/1).

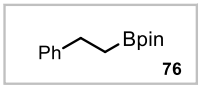
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 – 9.72 (m, 1H), 2.43 (t, *J* = 7.3 Hz, 2H), 1.67 – 1.60 (m, 4H), 1.46 – 1.40 (m, 2H), 1.35 – 1.33 (m, 2H), 1.26 (s, 12H), 0.79 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 82.9, 43.9, 32.0, 28.9, 24.8, 23.8, 22.0. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.4. HRMS (ESI): Calcd. for $C_{13}H_{26}BO_3$ m/z 241.1970, found m/z 241.1964 $[M+H]^+$



2-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)octahydropentalen-1-yl)acetaldehyde

Following the general procedure using octahydro-*1H*-4,7-methanoinden-5-ol (0.3 mmol, 46 mg) provided the product **75** (30.9 mg, 37% yield, rr = 6.3:1) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 25/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (t, *J* = 2.4 Hz, 1H), 2.58 – 2.52 (m, 1H), 2.46 – 2.38 (m, 1H), 2.11 – 1.94 (m, 3H), 1.89 – 1.78 (m, 1H), 1.60 – 1.49 (m, 5H), 1.45 – 1.37 (m, 3H), 1.25 (s, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.1, 82.9, 50.5, 49.5, 46.5, 44.1, 37.3, 33.2, 32.2, 29.7, 24.76, 24.70. ^{11}B NMR (128 MHz, $CDCl_3$) δ 34.1. HRMS (ESI): Calcd. for $C_{16}H_{28}BO_3$ m/z 279.2126, found m/z 279.2121 $[M+H]^+$

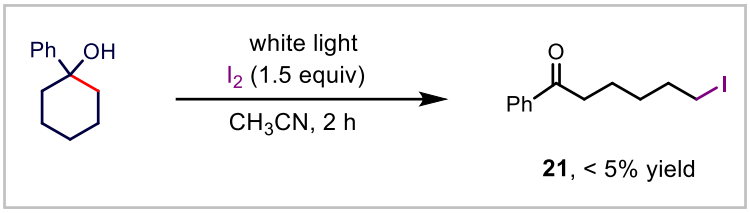


4,4,5,5-tetramethyl-2-phenethyl-1,3,2-dioxaborolane

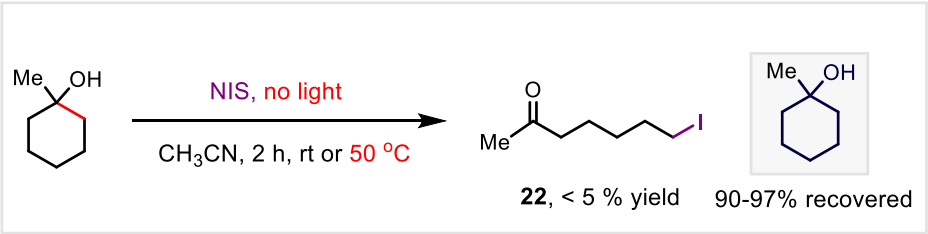
Following the general procedure using 1-(4-fluorophenyl)-3-phenylpropan-1-ol (0.3 mmol, 69 mg) provided the product **76** (27.8 mg, 40% yield) as a colorless liquid after purification by flash chromatography (eluent: PE/EA = 100/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 4H), 7.21 – 7.15 (m, 1H), 2.81 – 2.75 (t, *J* = 8.1 Hz, 2H), 1.25 (s, 12H), 1.17 (t, *J* = 8.1 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 128.2, 128.0, 125.5, 83.1, 30.07, 24.8. ^{11}B NMR (128 MHz, $CDCl_3$) δ 33.7 (Known compound*: J. Am. Chem. Soc.* **2022**, *144*, 2460).

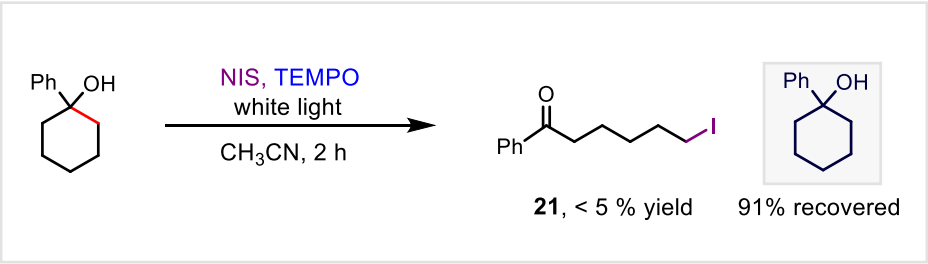
VII. Mechanistic studies



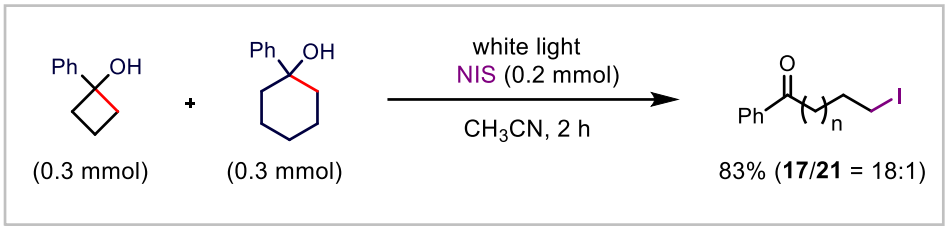
An oven-dried vial was charged with 1-phenylcyclohexan-1-ol (0.3 mmol, 53 mg), MeCN (3 mL), and I_2 (114 mg) under inert atmosphere. The sample was stirred and irradiated (white light, 8W) for 2 h. After that, starting material was decomposed and no desired product was detected.



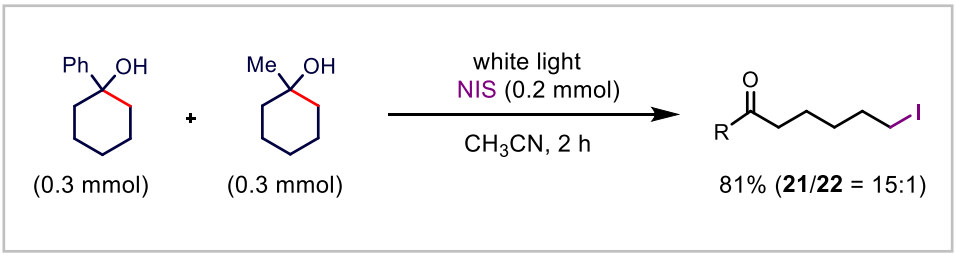
An oven-dried vial was charged with 1-methylcyclohexan-1-ol (0.3 mmol, 34 mg), 1-iodopyrrolidine-2,5-dione (0.45 mmol, 101 mg) and MeCN (3 mL) under inert atmosphere. The sample was stirred in the dark at room temperature or was heated at 50 °C for 2 h in the dark. After that, starting material was recovered in 90% and 97% yield, respectively.



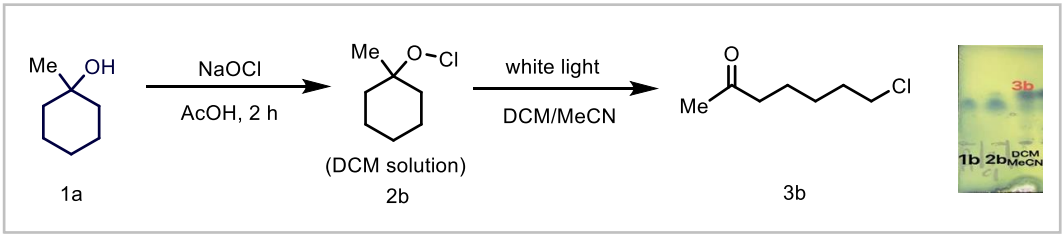
An oven-dried vial was charged with 1-phenylcyclohexan-1-ol (0.2 mmol, 35 mg), 1-iodopyrrolidine-2,5-dione (0.3 mmol, 68 mg), TEMPO (0.3 mmol, 47 mg) and MeCN (3 mL) under inert atmosphere. The sample was stirred and irradiated (white light, 8 W) for 2 h. After that, no product was detected with recovery of starting material in 91% yield.



An oven-dried vial was charged with 1-phenylcyclobutan-1-ol (0.3 mmol, 44 mg), 1-phenylcyclohexan-1-ol (0.3 mmol, 35 mg), 1-iodopyrrolidine-2,5-dione (0.2 mmol, 45 mg) and MeCN (3 mL) under inert atmosphere. The sample was stirred and irradiated (white light, 8 W) for 2 h. Purification by flash column chromatography gave the mixture compounds, which was analyzed by ^{1}H NMR.



An oven-dried vial was charged with 1-phenylcyclohexan-1-ol (0.3 mmol, 53 mg), 1-methylcyclohexan-1-ol (0.3 mmol, 34 mg), 1-iodopyrrolidine-2,5-dione (0.2 mmol, 45 mg) and MeCN (3 mL) under inert atmosphere. The sample was stirred and irradiated (white light, 8 W) for 2 h. Purification by flash column chromatography gave the mixture compounds, which was analyzed by ^{1}H NMR.

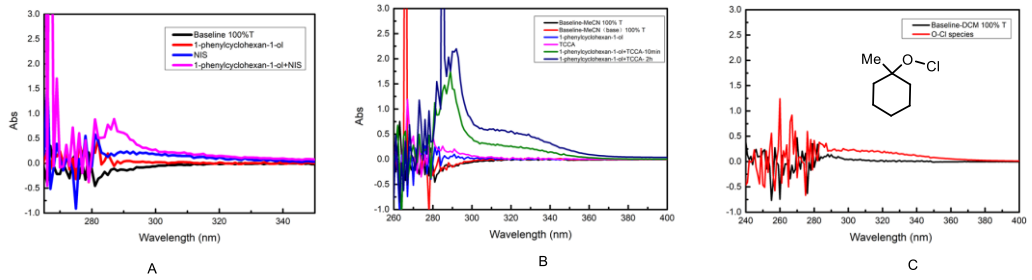


To a 8% aqueous sodium hypochlorite solution（0.9 mmol）at 0 ℃ was added 0.9 mmol of 1-methylcyclohexan-1-ol in portions into the flask, followed by the addition of acetic acid (0.9 mmol). Stir the reaction mixture for 2 hours at 0 ℃, and extract the reaction mixture with DCM (9 mL). Wash the organic phase with a saturated solution of sodium bicarbonate, and dry the organic layer over sodium sulfate. An oven-dried vial was

charged with the resulted DCM solution of hypochlorite (3 mL) and additional MeCN (3 mL). Under air atmosphere, the sample was stirred and irradiated (white light, 8 W) for 10 h. Then purification by flash column chromatography gave the title compound as colorless liquid (20.5 mg, 46% yield).

UV-Vis Characterization

The monitor of the reaction by UV-Vis was performed (10^{-5} mol/L). The related results were listed below: A) the mixture of alcohol and NIS; B) the mixture of alcohol, TCCA, and K_2PO_4; C) the mixture of pre-prepared hypochlorite.



Quantum Yield Determination

a) Determination of Photon Flux using Ferrioxalate Actinometry

Standard ferrioxalate actinometry was used to determine the photon flux of the spectrophotometer at 455 nm.

Preparation of solutions: Two stock solutions of H_2SO_4 (0.05 M and 0.5 M) were prepared in volumetric flasks. A 0.15 M solution of ferrioxalate (**Solution A**) was prepared by dissolving potassium ferrioxalate hydrate (2.21 g, 4.5 mmol) in 30 mL of 0.05 M aq H_2SO_4 in a volumetric flask wrapped in aluminum foil. A buffered solution of phenanthroline (**Solution B**) was prepared by dissolving phenanthroline (0.05 g, 0.28 mmol) and NaOAc (11.25 g, 0.14 mmol) in 50 mL of 0.5 M aq H_2SO_4 in a volumetric flask wrapped in aluminum foil. Both solutions were stored in the dark. *Absorbance of non-irradiated sample:* In a dark room, 2.0 mL of **Solution A** and 0.35 mL of **Solution B** were added via syringes to a vial. The vial was capped and allowed to sit in the dark for 1 h, after which time it was transferred to a cuvette, and the absorbance at 510 nm measured.

Absorbance of the irradiate sample: In a dark room, 2.0 mL of **Solution A** was placed in a vial and irradiated for 90.0 seconds at λ = 455 nm. After irradiation, 0.35 mL of **Solution B** was added to the vial. The solution was then allowed to rest for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline, after which time it was transferred to a cuvette, and the absorbance at 510 nm measured.

Calculation of photon flux: The following equations were used to calculate the photon flux.

$$\text{mol Fe}^{2+} = \frac{V \cdot \Delta A}{l \cdot \varepsilon}$$

Where V is the total volume of the solution, ΔA is the difference in absorption between the irradiated and non-irradiated solutions, ι is the path length (1.000 cm), and ε is the molar absorptivity at 510 nm (11,110 L mol^{-1} cm^{-1}).

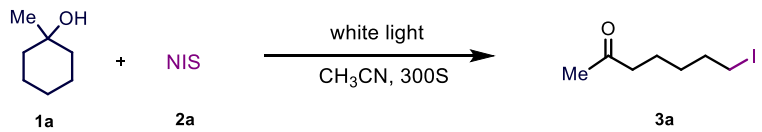
$$\text{photon flux} = \frac{\text{mol } Fe^{2+}}{\Phi \cdot t \cdot f}$$

Where Φ is the quantum yield for the ferrioxalate actinometer (0.845 for a 0.15 M solution at λ = 457 nm), t is the time (90.0 s), and f is the fraction of light absorbed at λ = 455nm.

$$\text{mol Fe}^{2+} = \frac{0.00235\text{L} \cdot 0.764}{1.000\,\text{cm} \cdot 11{,}100\,\text{L mol}^{-1}\text{cm}^{-1}} = 1.62 \times 10^{-7}\ \text{mol}$$

$$\text{photon flux} = \frac{1.62 \times 10^{-7}\,\text{mol}}{0.845 \cdot 90\,\text{s} \cdot 0.97593} = 2.18 \times 10^{-9}\,\text{einstein s}^{-1}$$

b) Measurement of quantum yield



An oven-dried vial was charged with 1-methylcyclohexan-1-ol 1a (0.2 mmol, 22.8 mg), 1-iodopyrrolidine-2,5-dione 2a (0.3 mmol, 67.5 mg). The vial was put on vacuum and backfilled with nitrogen three times. Afterwards MeCN (2 mL) was added by syringe.

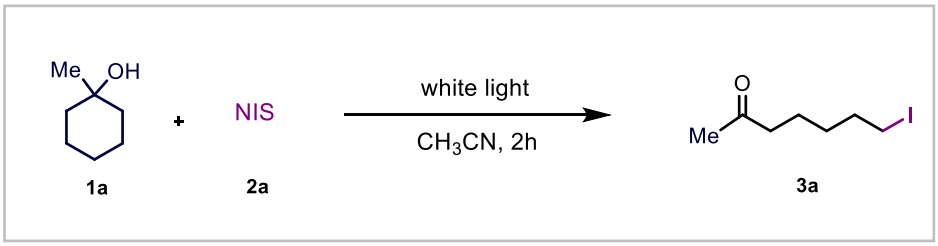
Under nitrogen atmosphere, the sample was stirred and irradiated (λ = 455 nm) for 300s. After irradiation, the reaction was transferred to a foil lined flask and concentrated under reduced pressure to give a yellow oil. The yield of 3a was determined by 1H NMR using 1,1,2,2-tetrachloroethane as an external standard. The quantum yield calculation is then as following:

$$\Phi = \frac{\text{moles of product}}{\text{moles of absorbed photons}} = \frac{\text{moles of product}}{\text{flux} \cdot t \cdot f}$$

Where flux is the photon flux determined by ferrioxalate actinometry (2.18×10^{-9} einstein s^{-1}), *t* is the time (300 s), and *f* is the fraction of light absorbed by reaction system at 455 nm. The absorbance of the solution at 455 nm was calculated: $f = 1\text{-}10^{-A}$. Where A is the absorbance of the solution at 455 nm, which was found to be 0.054. $f = 1 - 10^{-0.054} = 0.1169$.

$$\Phi = \frac{1 \times 10^{-5}\,\text{mol}}{2.18 \times 10^{-9}\,\text{einstein}\,\text{s}^{-1} \cdot 300\,\text{s} \cdot 0.1169} = 131.38$$

On/off studies



An oven-dried 10 mL Schlenk tube was charged with 1-methylcyclohexan-1-ol **1a** (0.3 mmol, 34 mg), 1-iodopyrrolidine-2,5-dione **2a** (0.45 mmol, 101 mg) and naphthalene (20.7 mg). The Schlenk tube was put on vacuum and backfilled with nitrogen three times. Afterwards MeCN (2 mL) was added by syringe. Under nitrogen atmosphere, the sample was stirred and irradiated (white light) for 2 h. The reaction was alternatively irradiated in the LED reactor and kept in the dark in five minutes intervals. For each indicated time, reaction mixture was taken from the Schlenk tube under a flow of nitrogen. After 2 h, the yield of product **3a** was determined by GC-MS using naphthalene as internal standard.

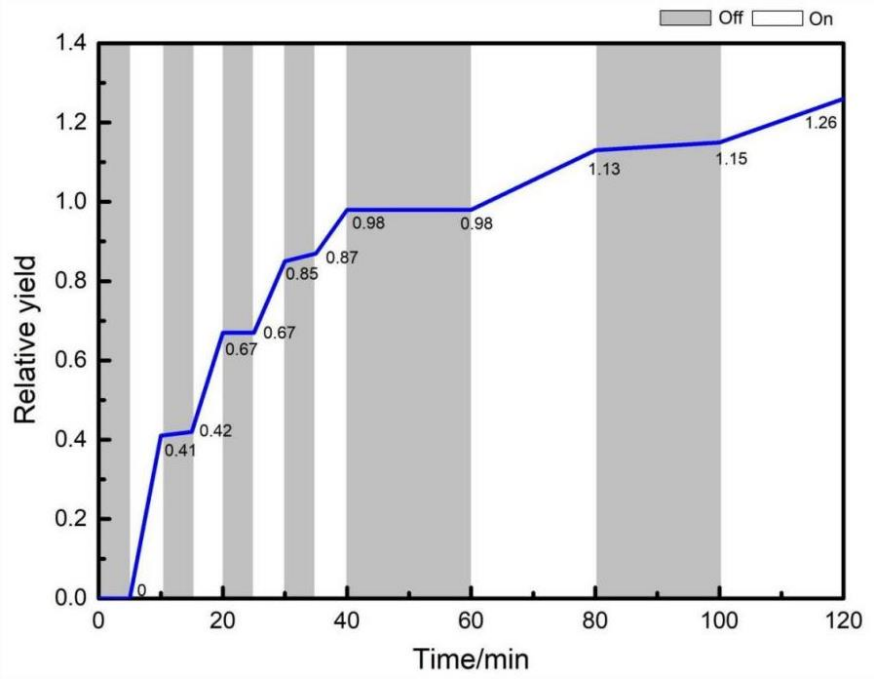
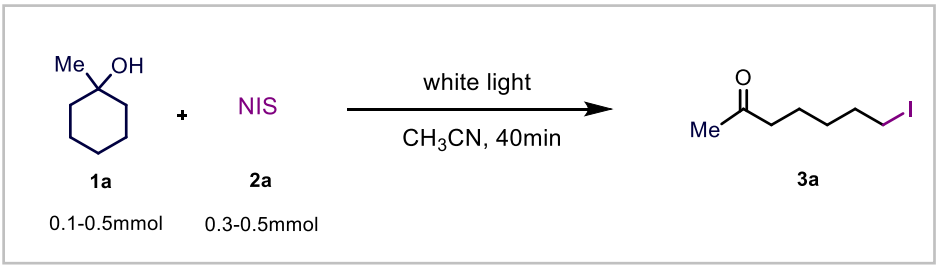
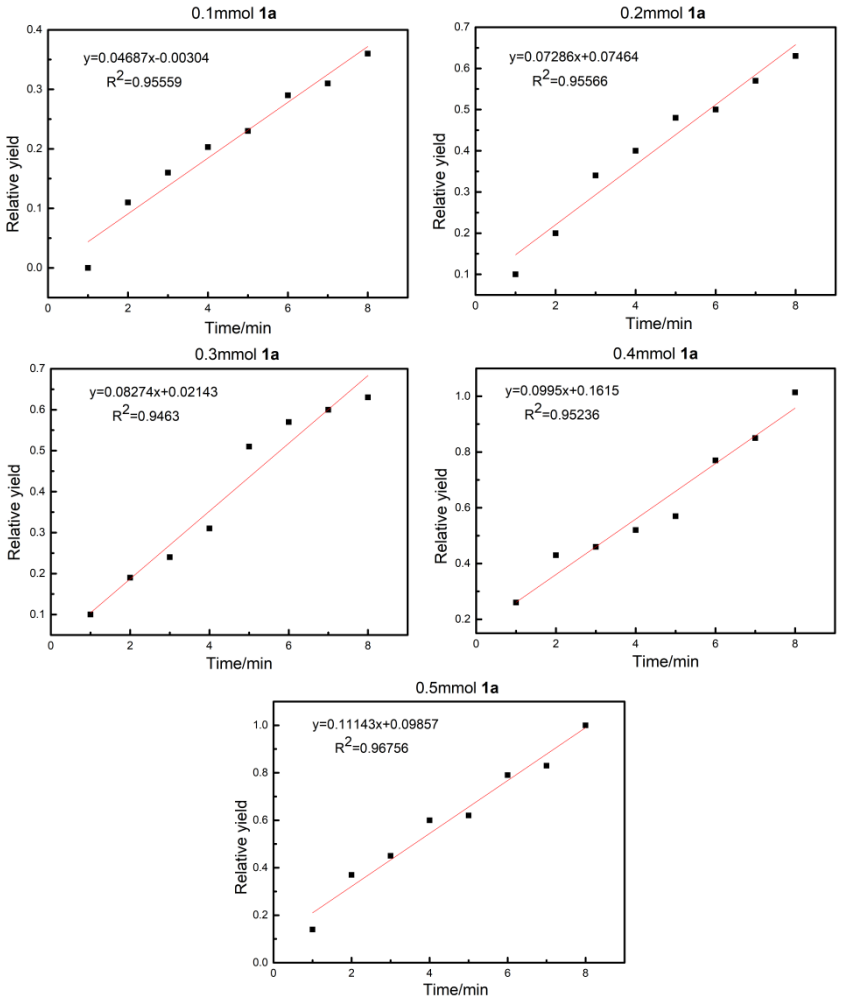


Fig .1 Plot of Effect of Light Source on Reaction Rate Over Time

Kinetic experiments



An oven-dried 10 mL Schlenk tube was charged with 1-methylcyclohexan-1-ol **1a**, 1-iodopyrrolidine-2,5-dione **2a** and naphthalene (20 mg). The Schlenk tube was put on vacuum and backfilled with nitrogen three times. Afterwards MeCN (3 mL) was added by syringe. Under nitrogen atmosphere, the sample was stirred and irradiated (white light) for 40 min. For each indicated time, the reaction mixture was taken from the Schlenk tube under a flow of nitrogen. The yield of product **3a** was determined by GC-MS using naphthalene as internal standard.



1a M/L	k_{obs}（mol • $L^{-1}min^{-1}$）
0.05	0.04687
0.1	0.07286
0.15	0.08274
0.2	0.09951
0.25	0.11143

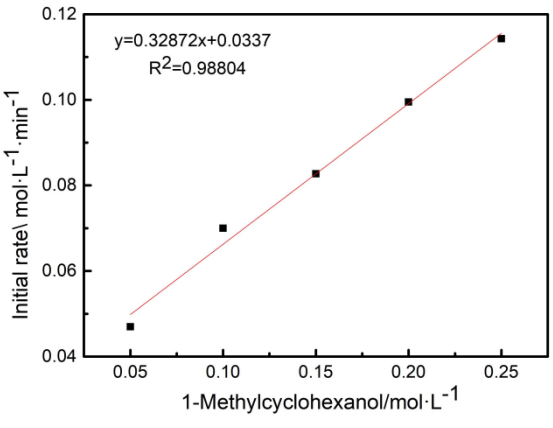
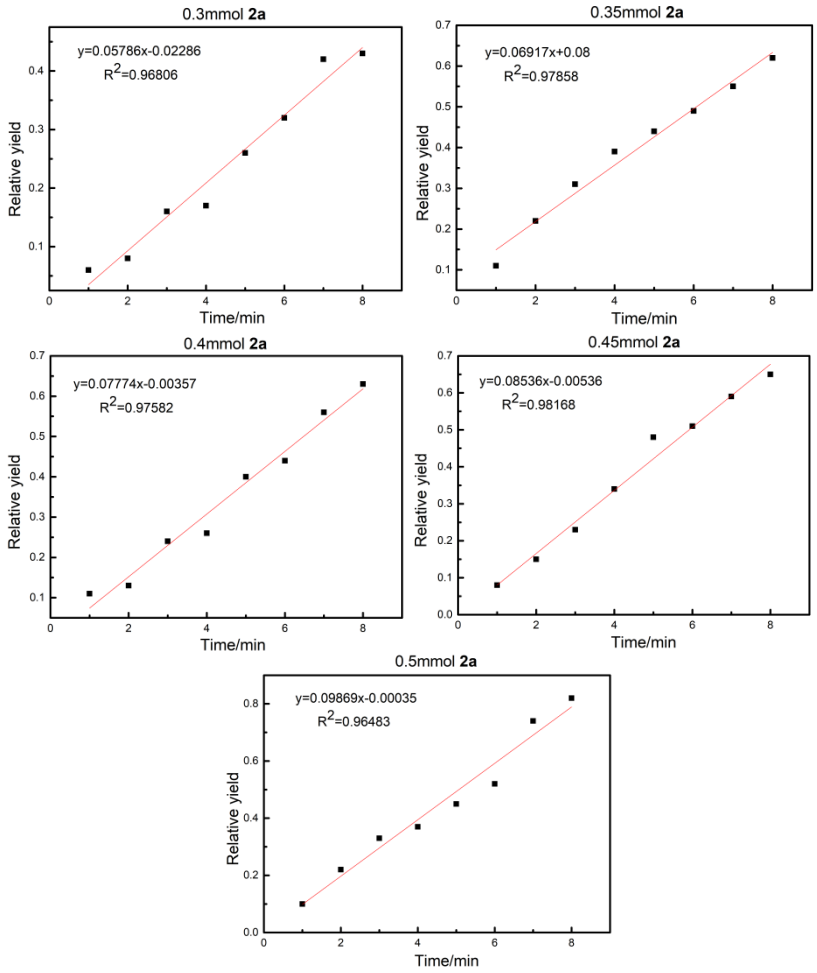


Fig .2 Dependence of the reaction rate on concentration of 1a

Kinetic profiles of different initial concentrations of **1a** (from 0.1 mmol to 0.5 mmol).



2a M/L	k_{obs}（mol • $L^{-1}min^{-1}$）
0.15	0.05786
0.175	0.06917
0.2	0.07774
0.2225	0.08536
0.25	0.09869

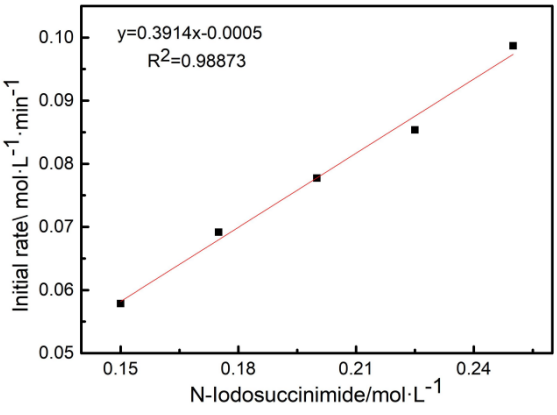
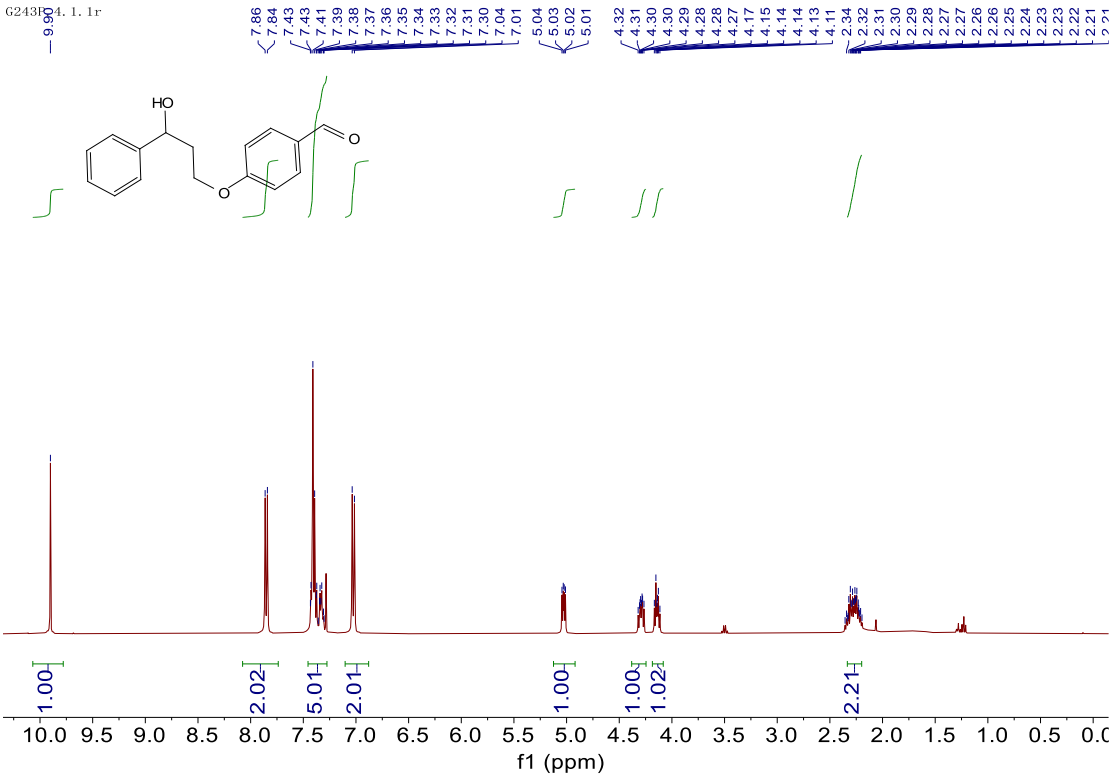


Fig .3 Dependence of the reaction rate on concentration of 2a

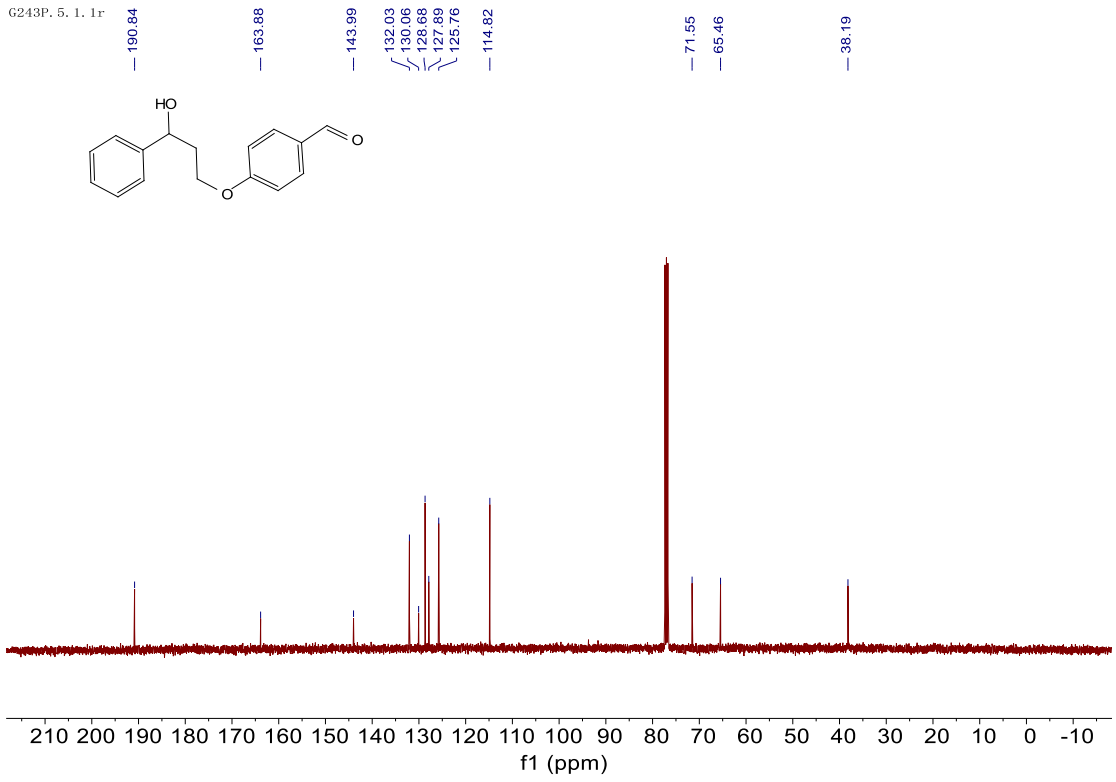
Kinetic profiles of different initial concentrations of **2a** (from 0.3 mmol to 0.5 mmol).

VIII.NMR spectra

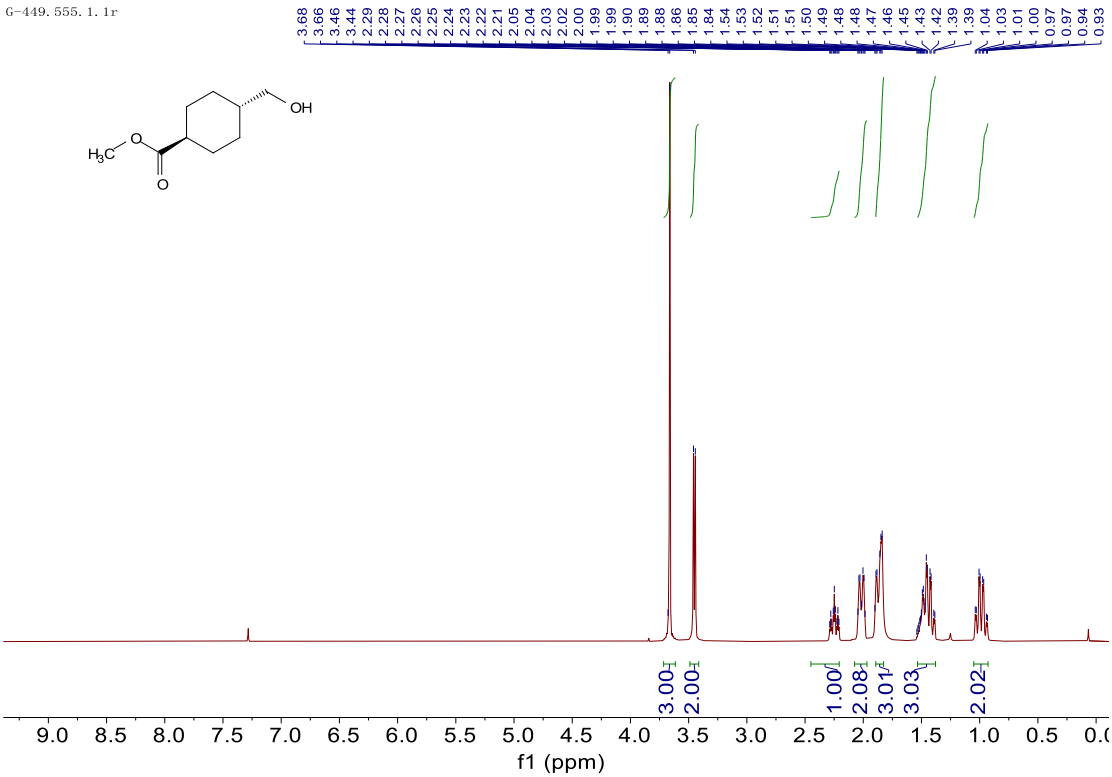
^{1}H NMR



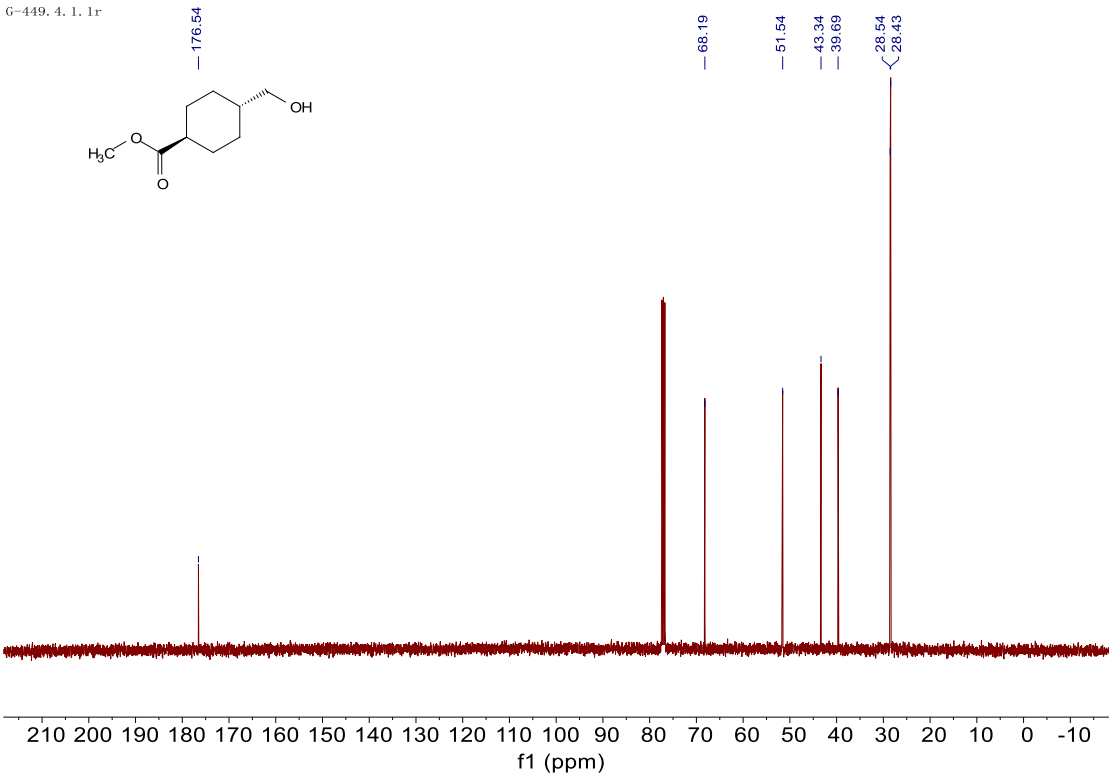
^{13}C NMR



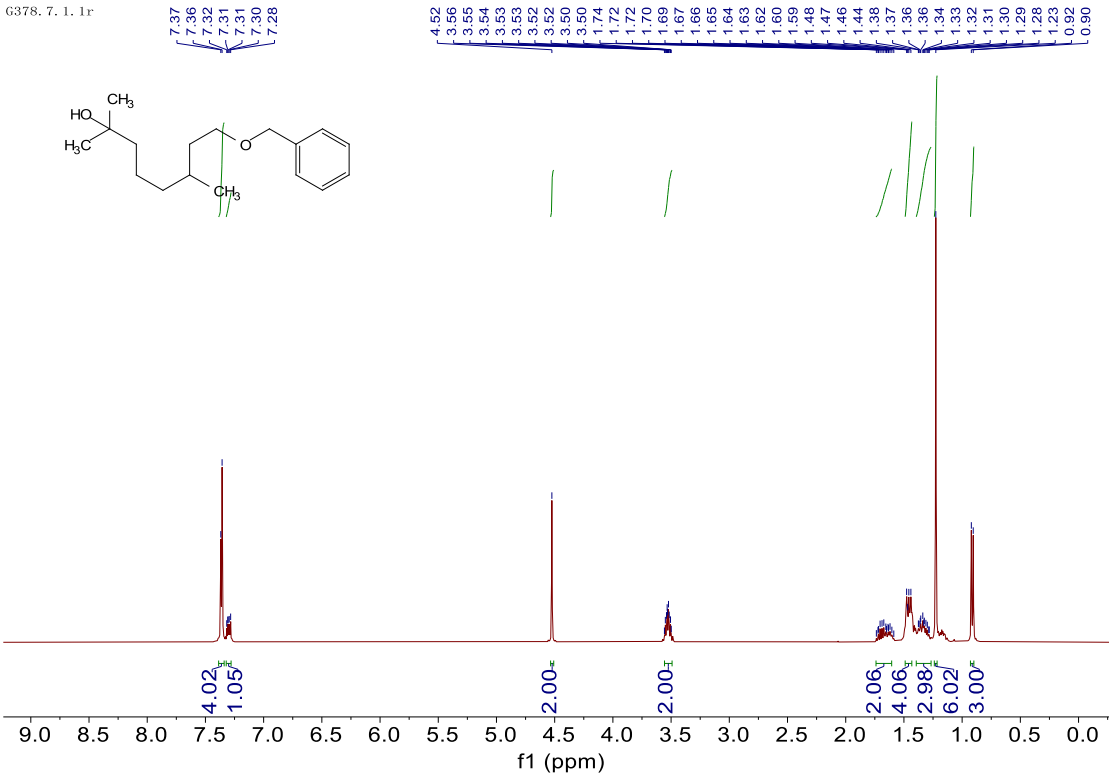
^{1}H NMR



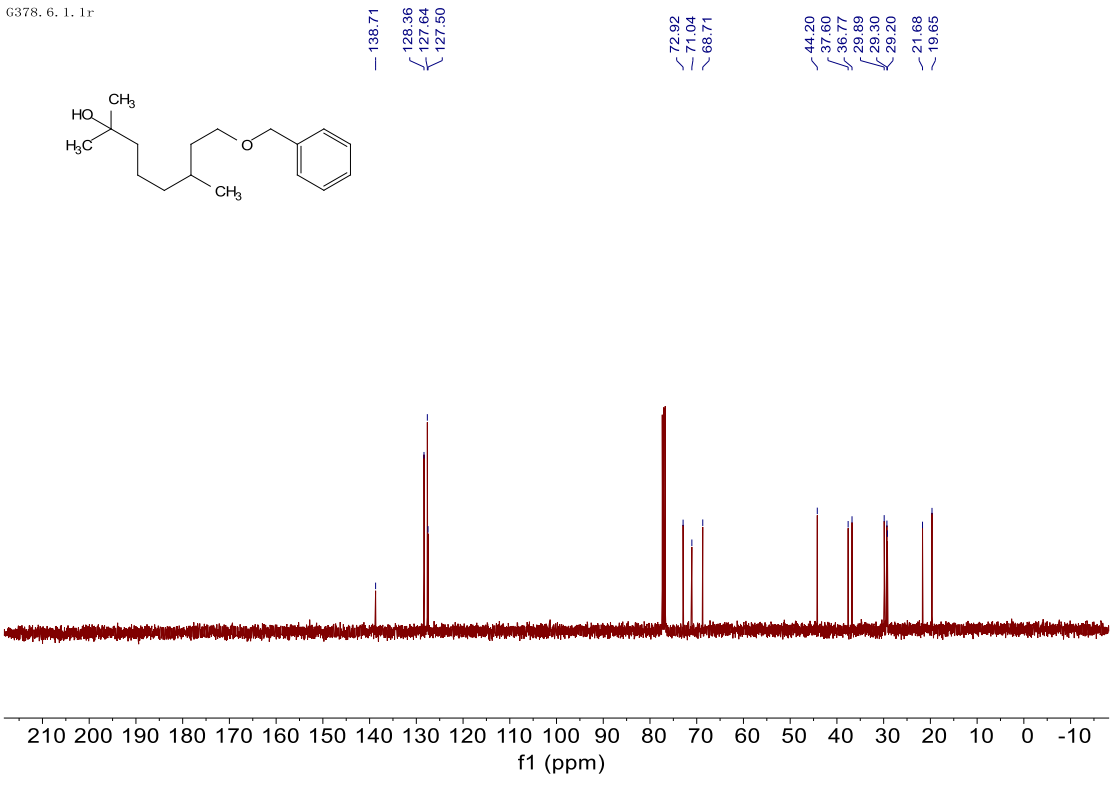
^{13}C NMR



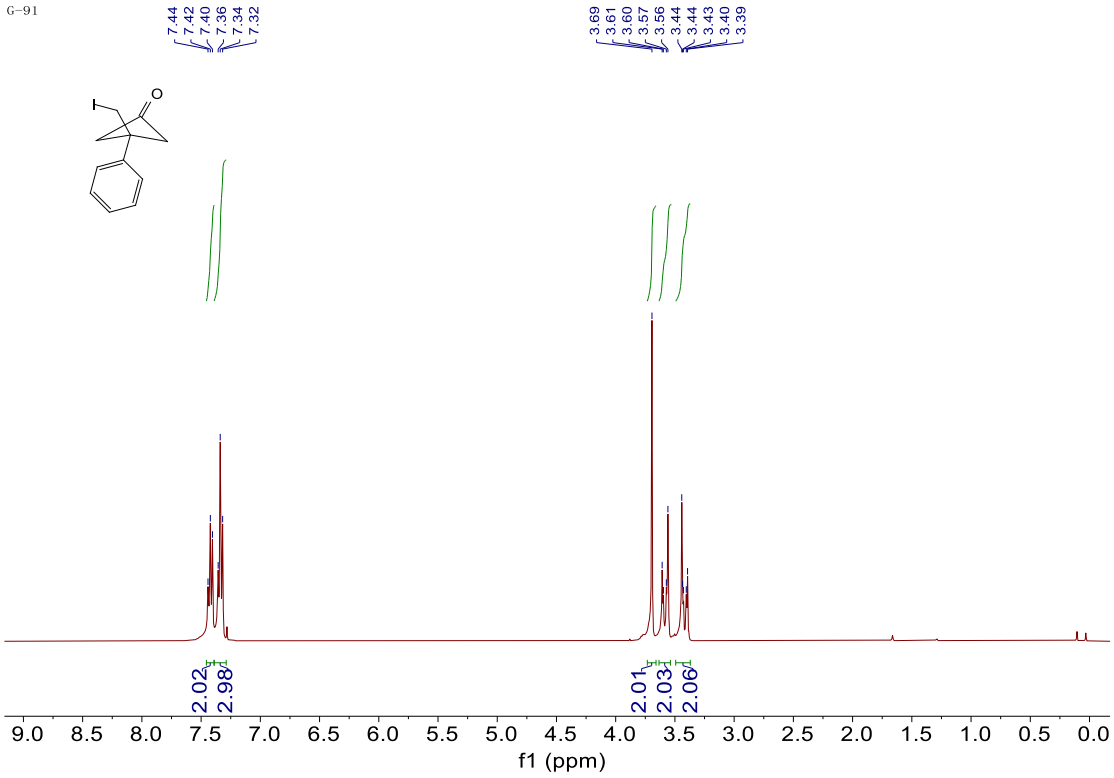
^{1}H NMR



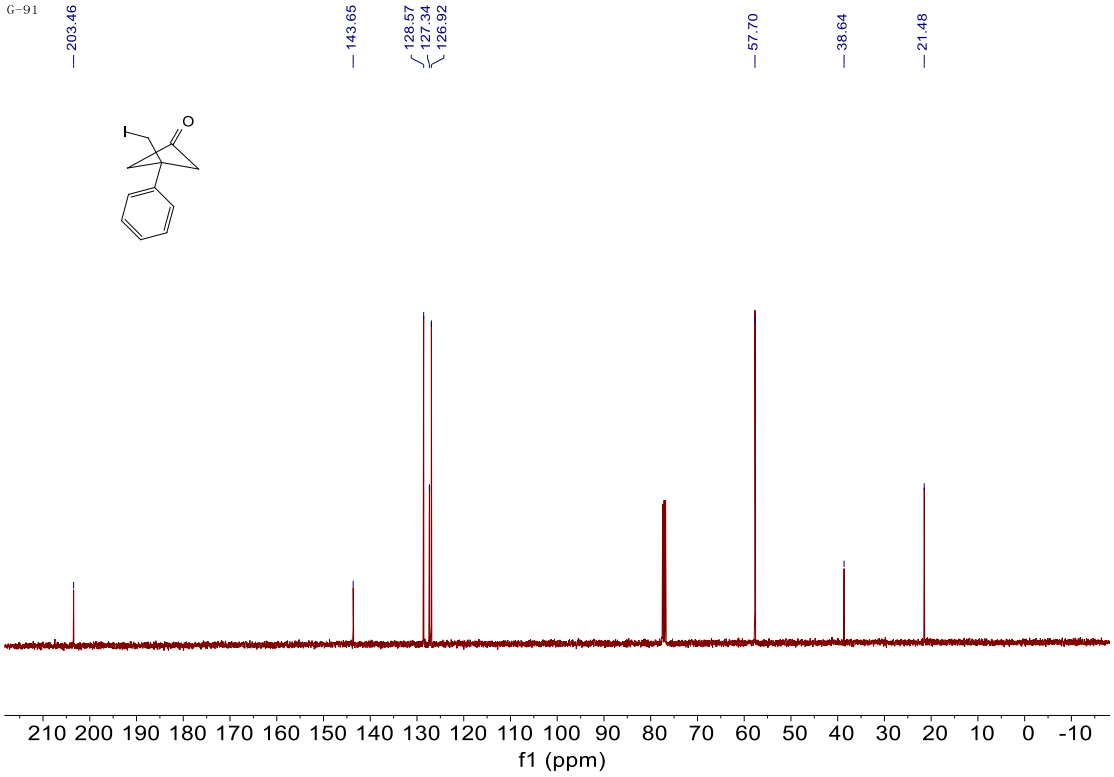
^{13}C NMR



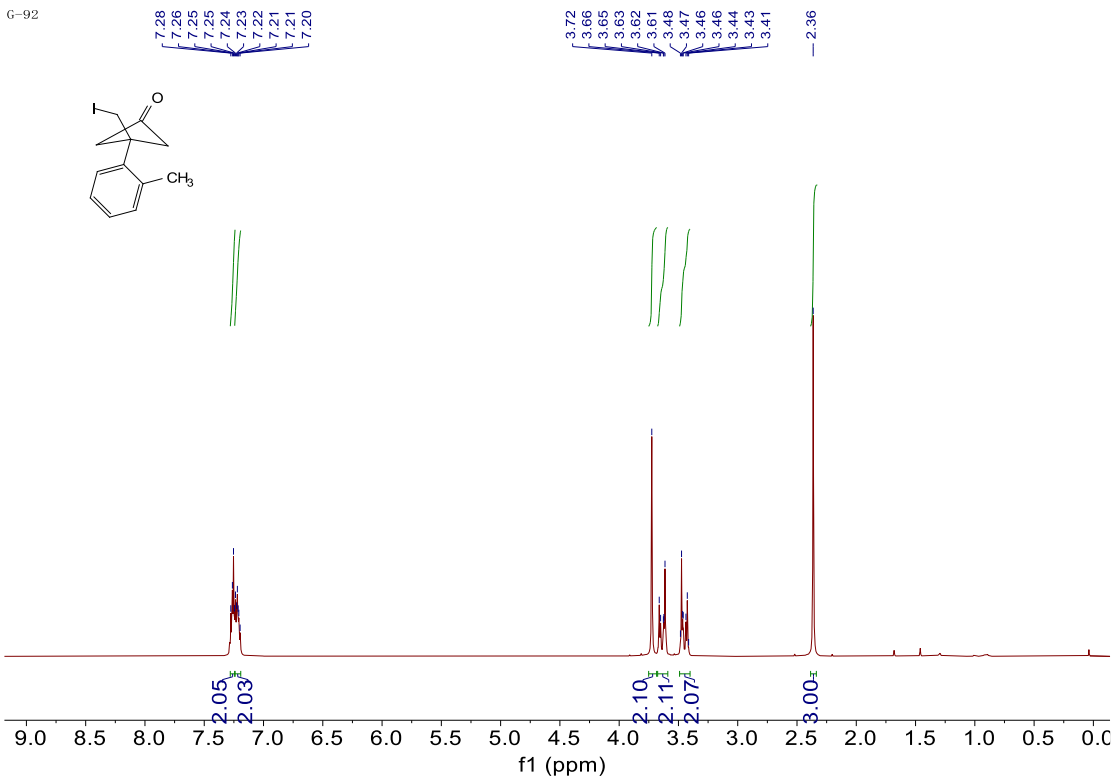
^{1}H NMR of compound **2**



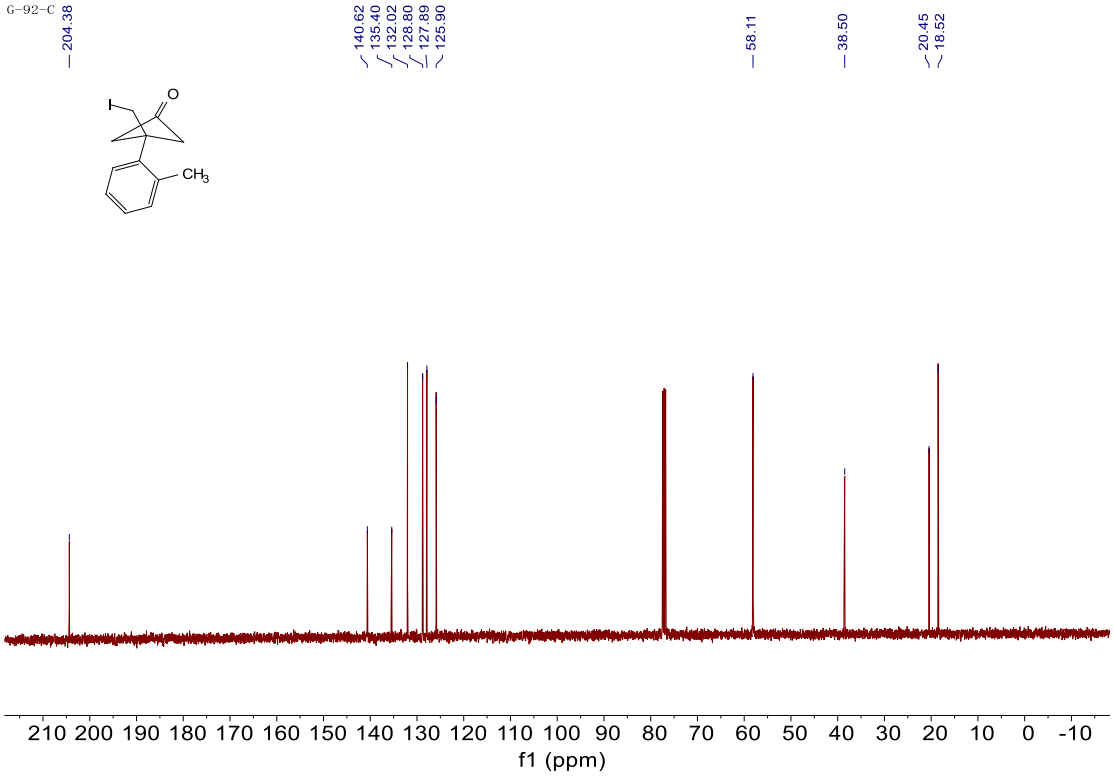
^{13}C NMR of compound **2**



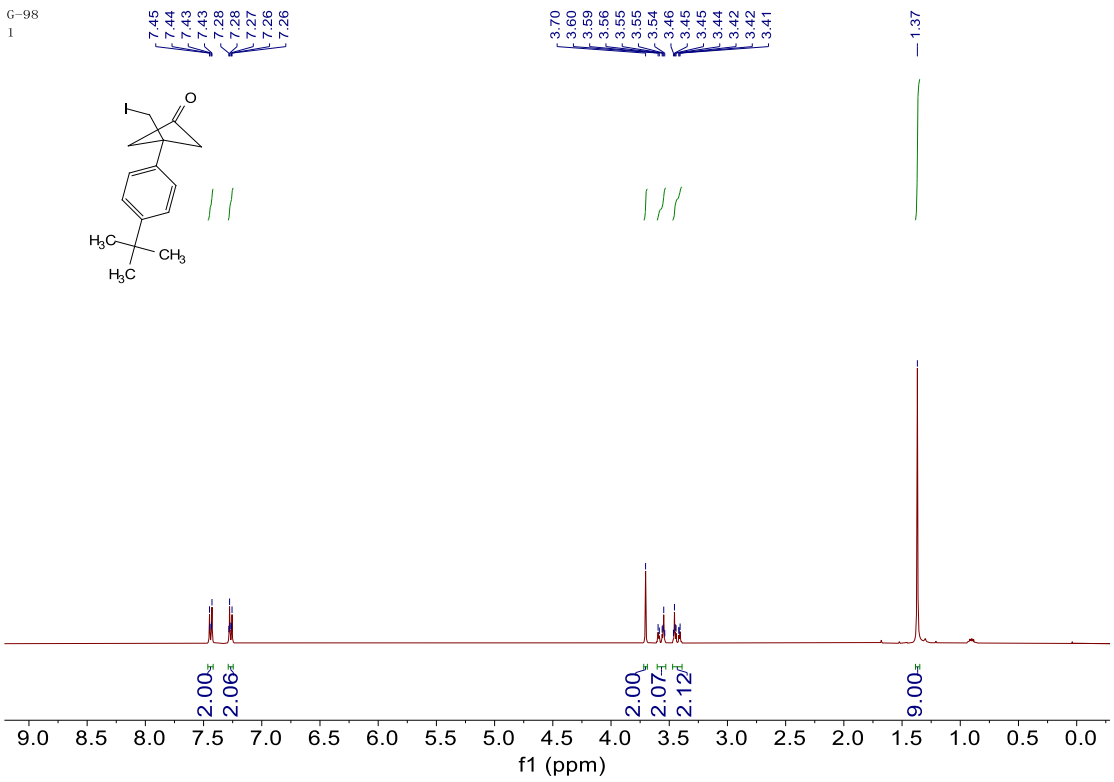
^{1}H NMR of compound **3**



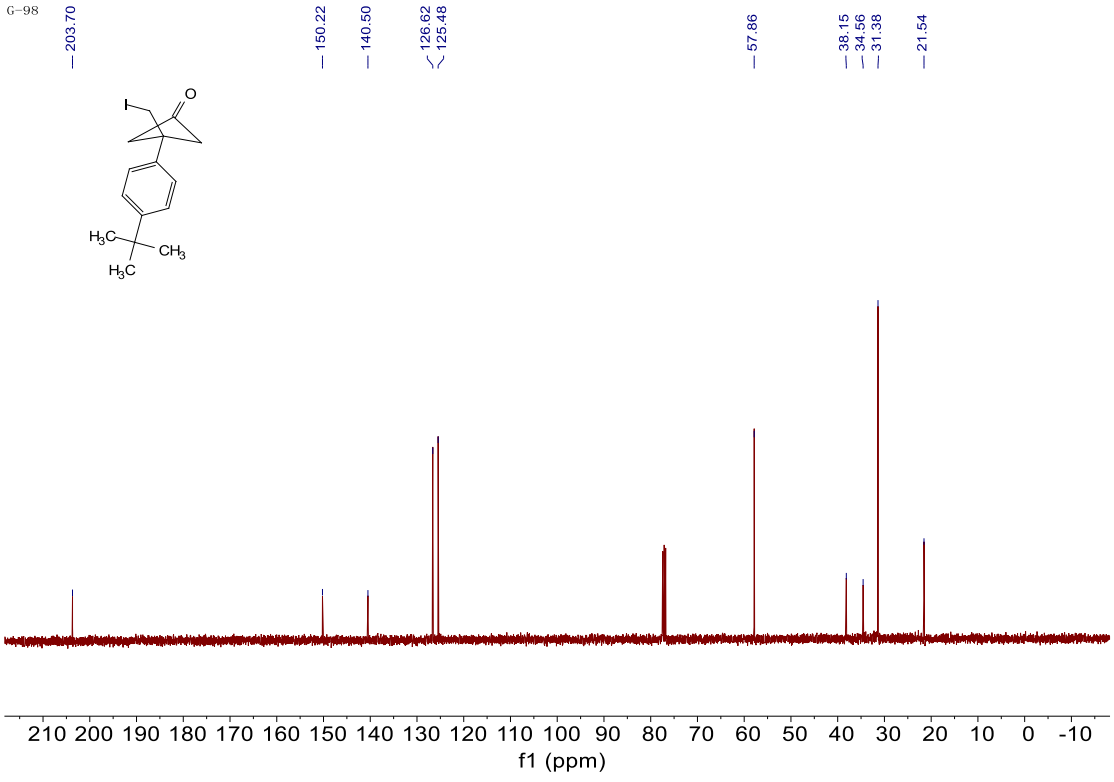
^{13}C NMR of compound **3**



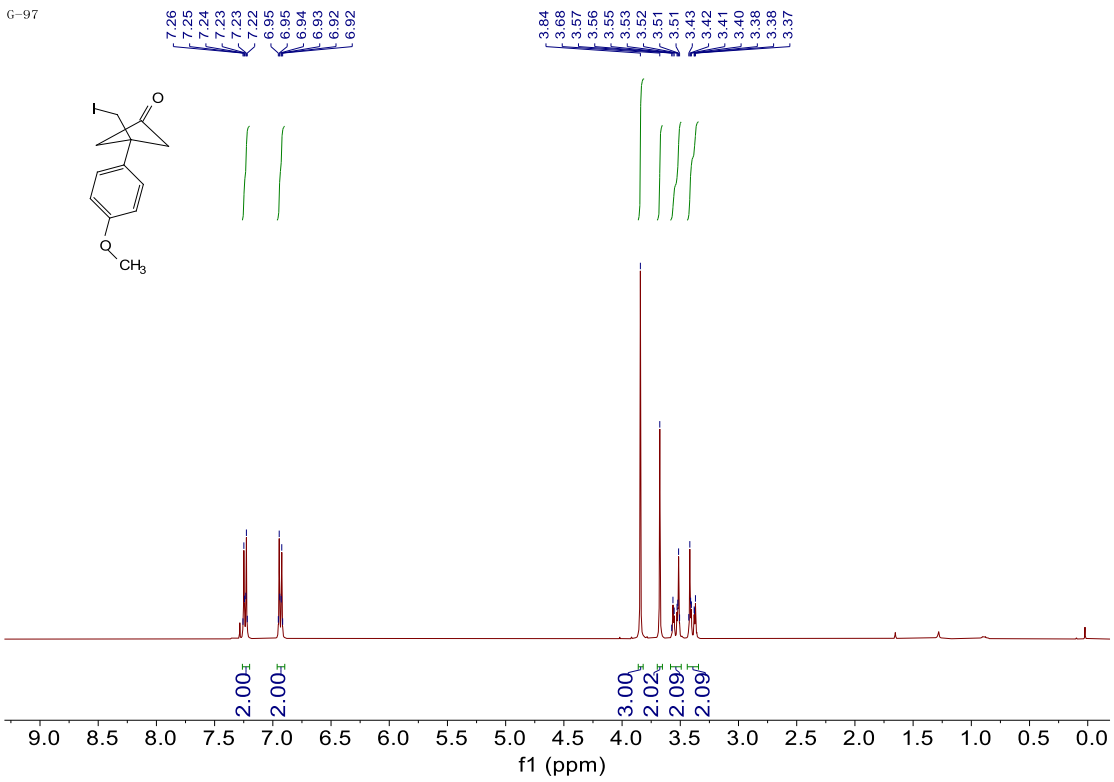
^{1}H NMR of compound **4**



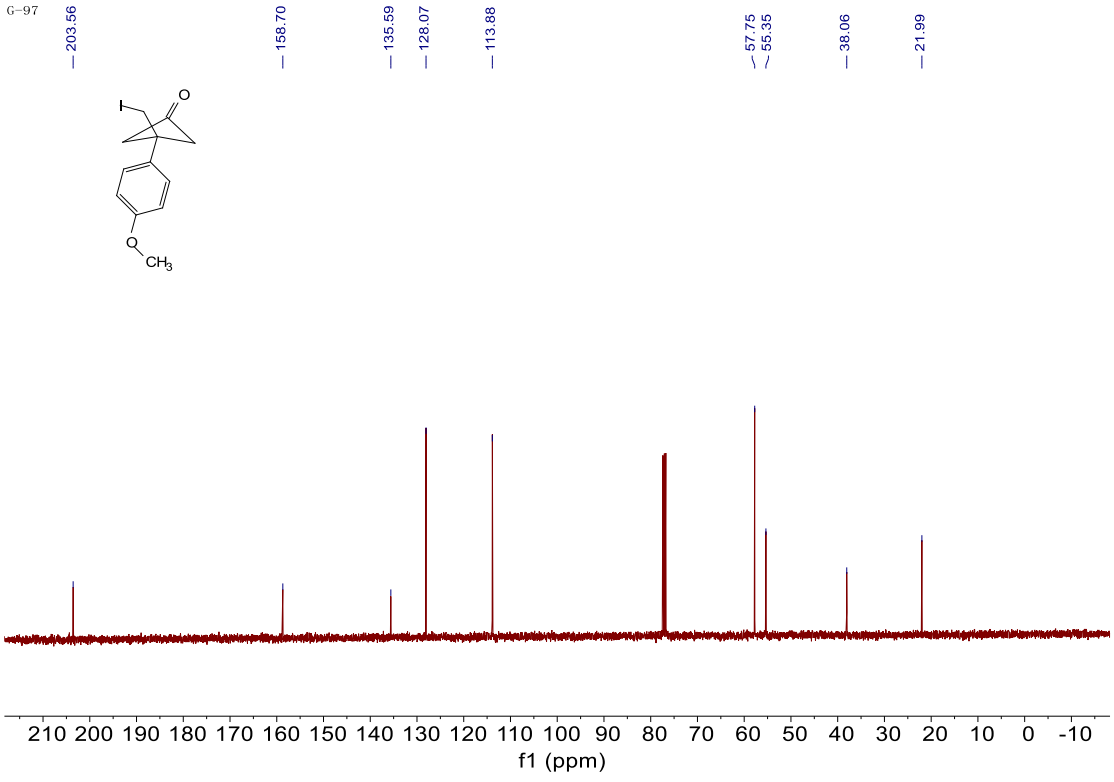
^{13}C NMR of compound **4**



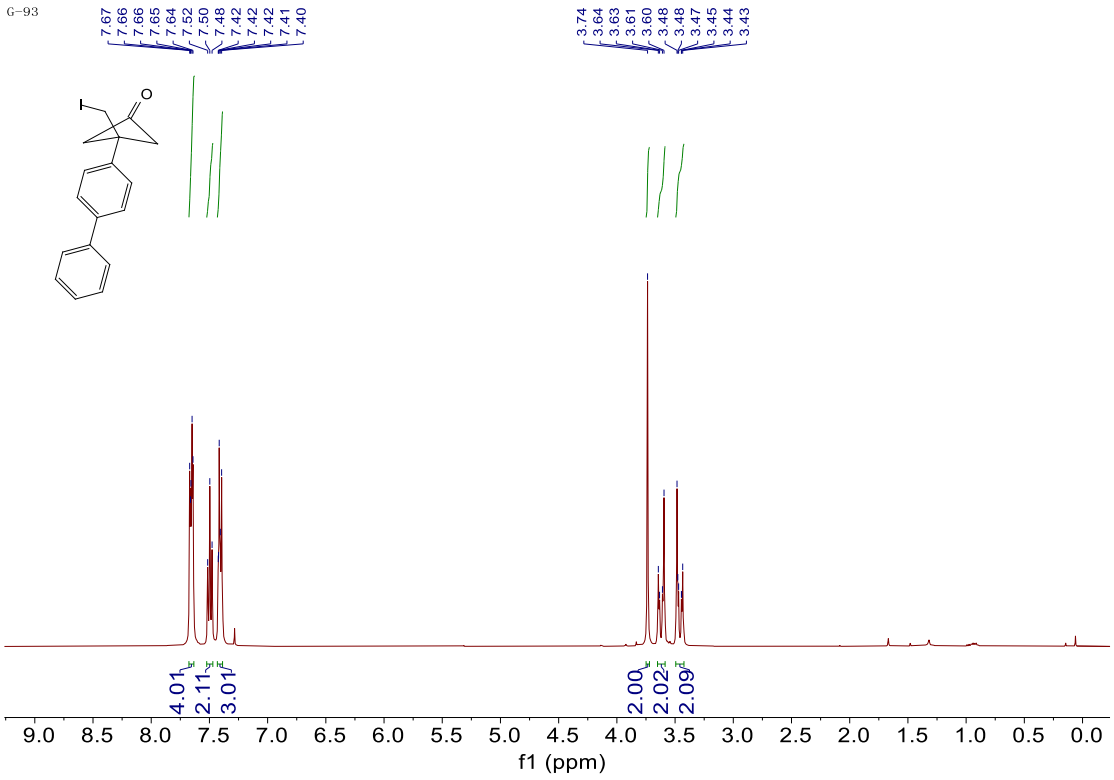
^{1}H NMR of compound **5**



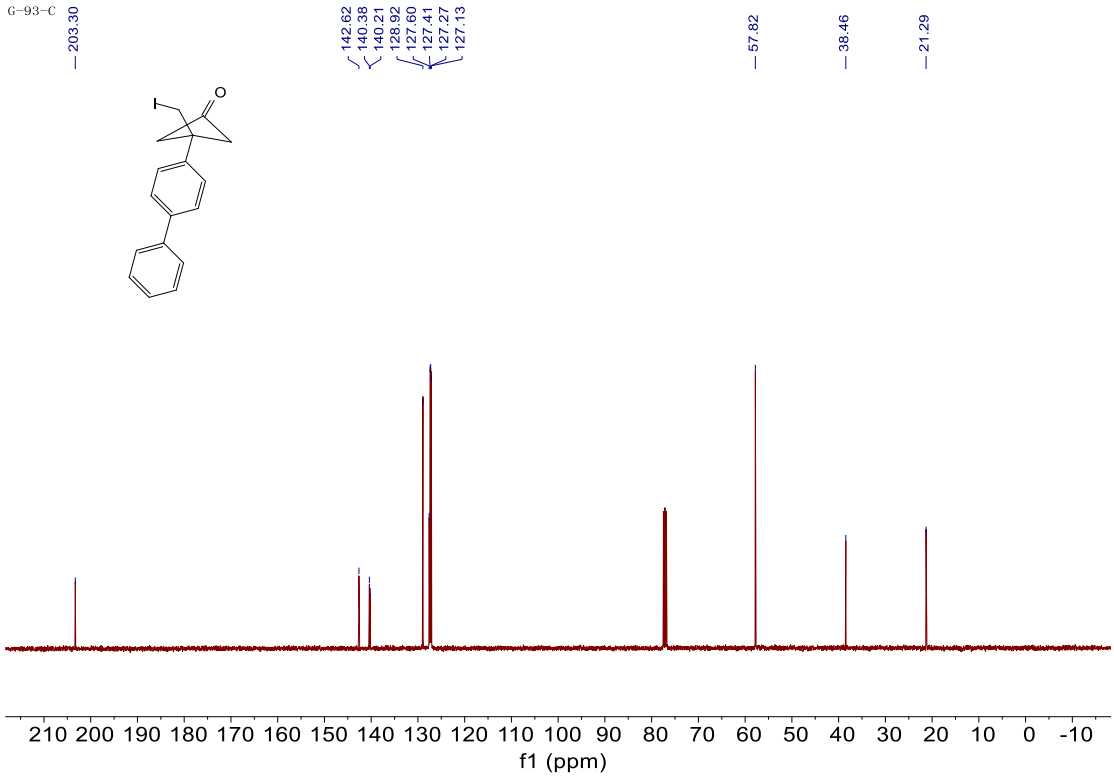
^{13}C NMR of compound **5**



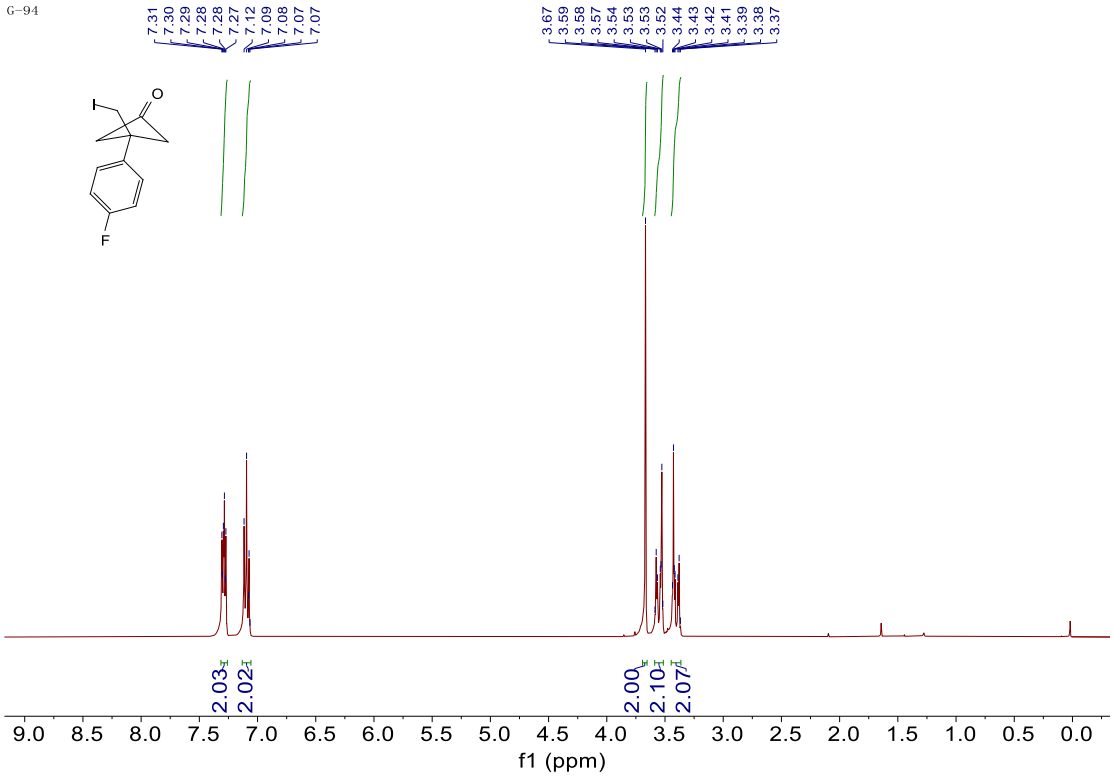
^{1}H NMR of compound **6**



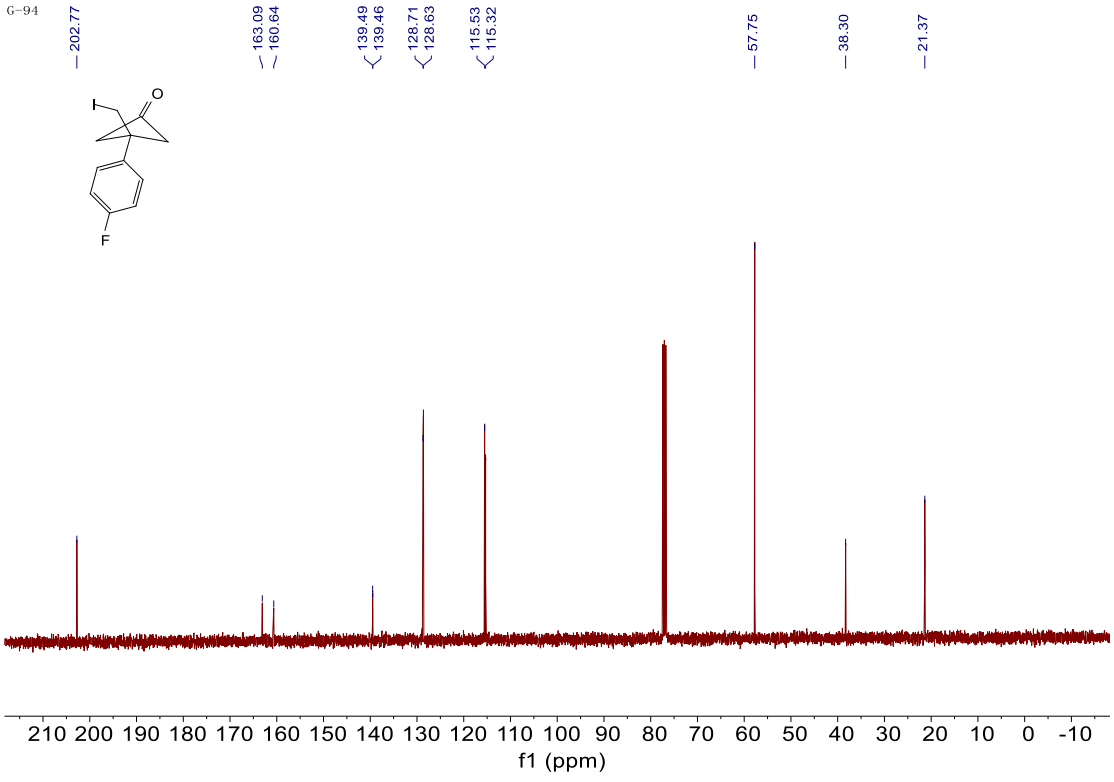
^{13}C NMR of compound **6**



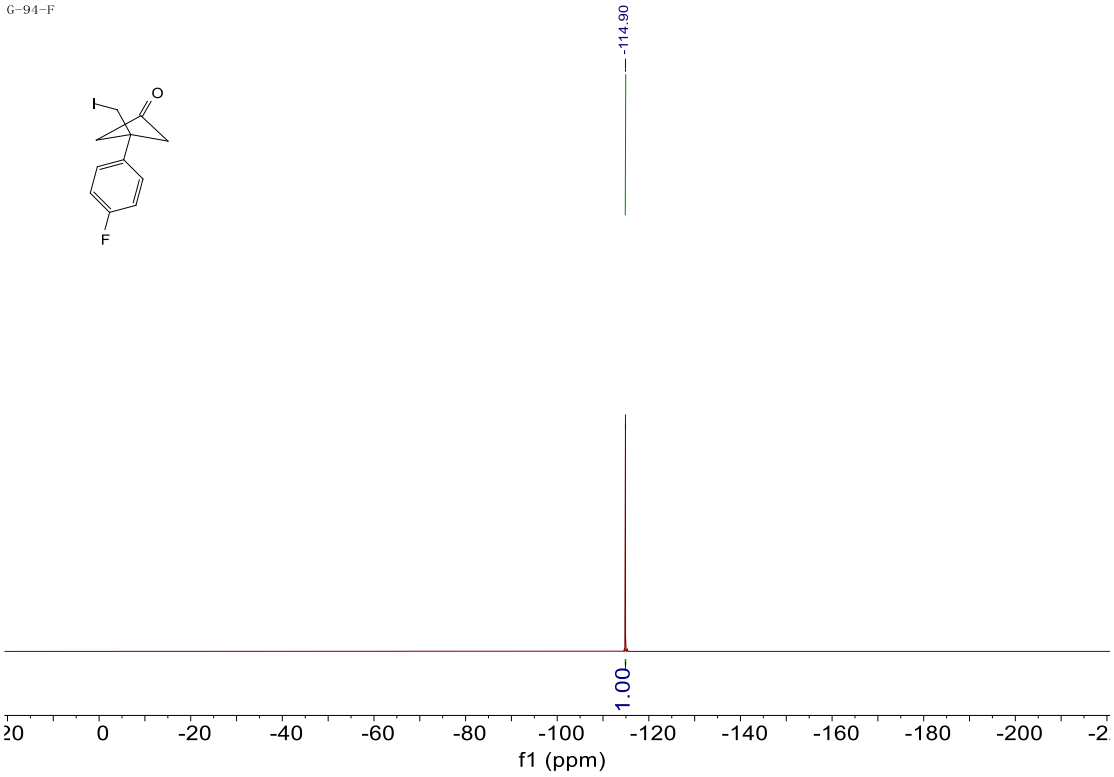
^{1}H NMR of compound **7**



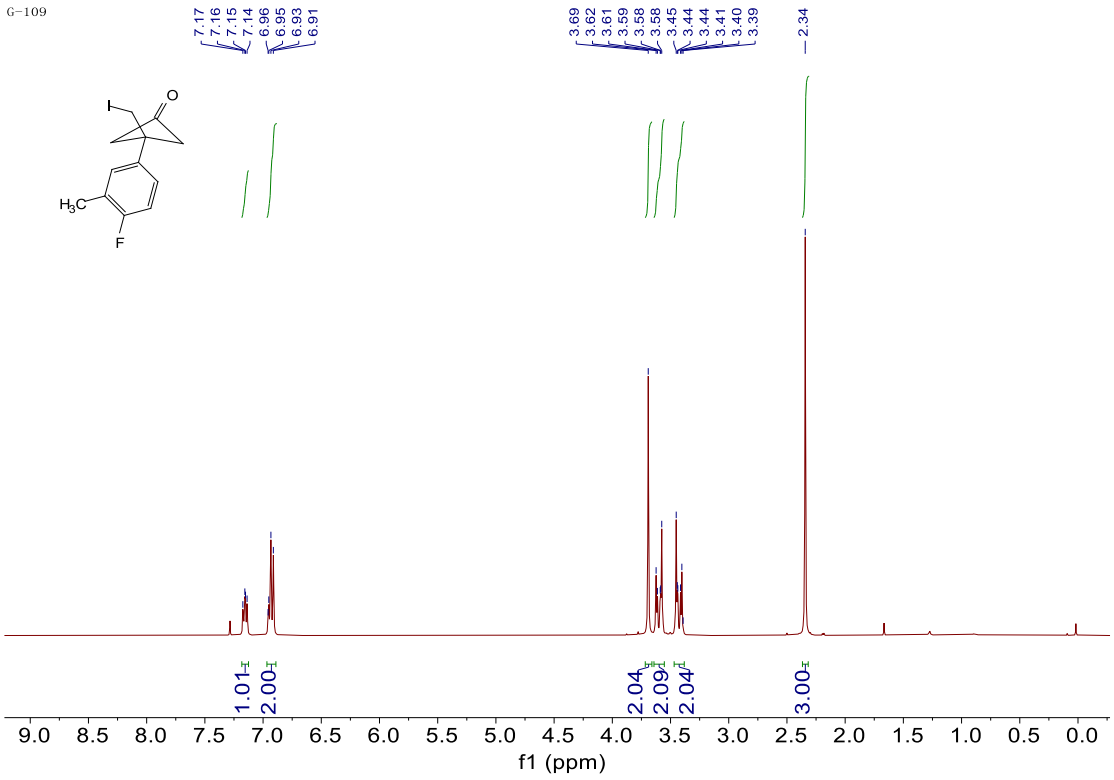
^{13}C NMR of compound **7**



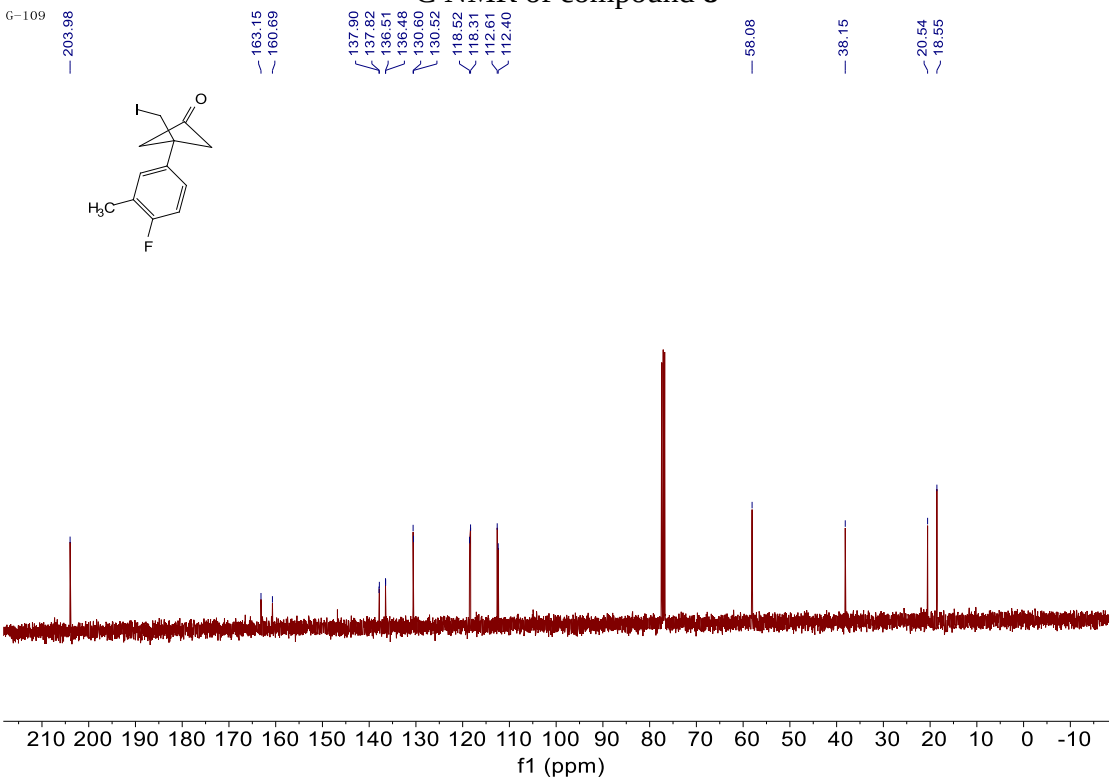
^{19}F NMR of compound **7**



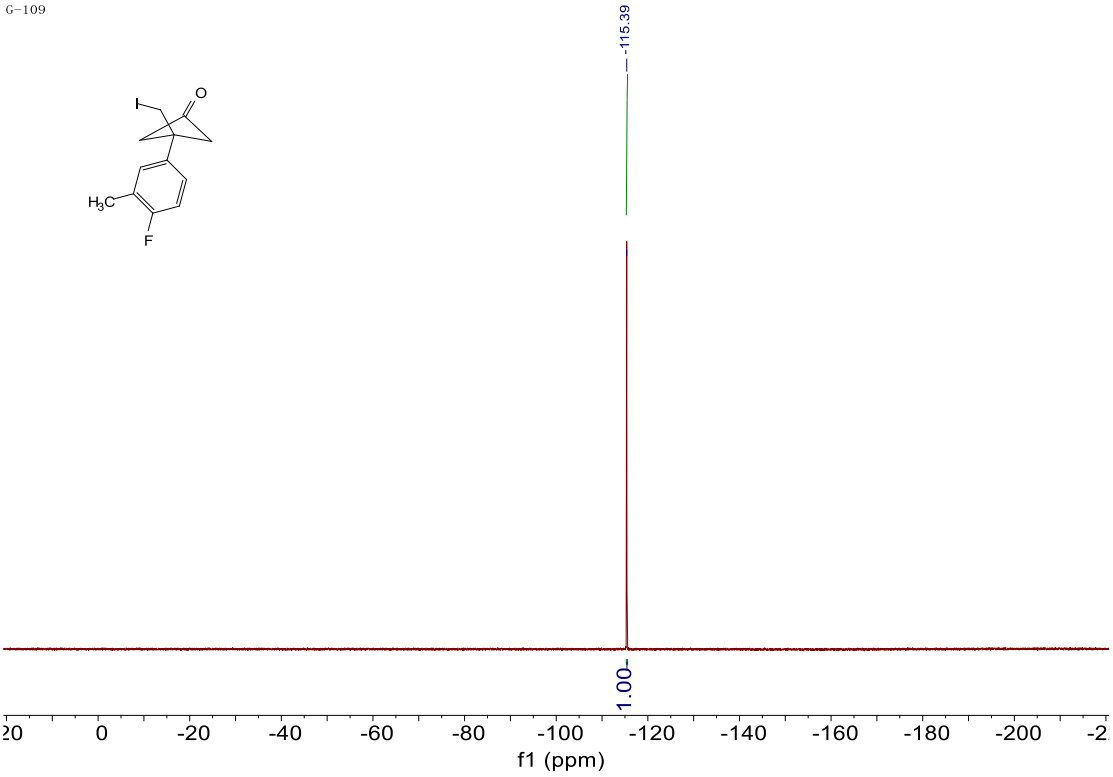
^{1}H NMR of compound **8**



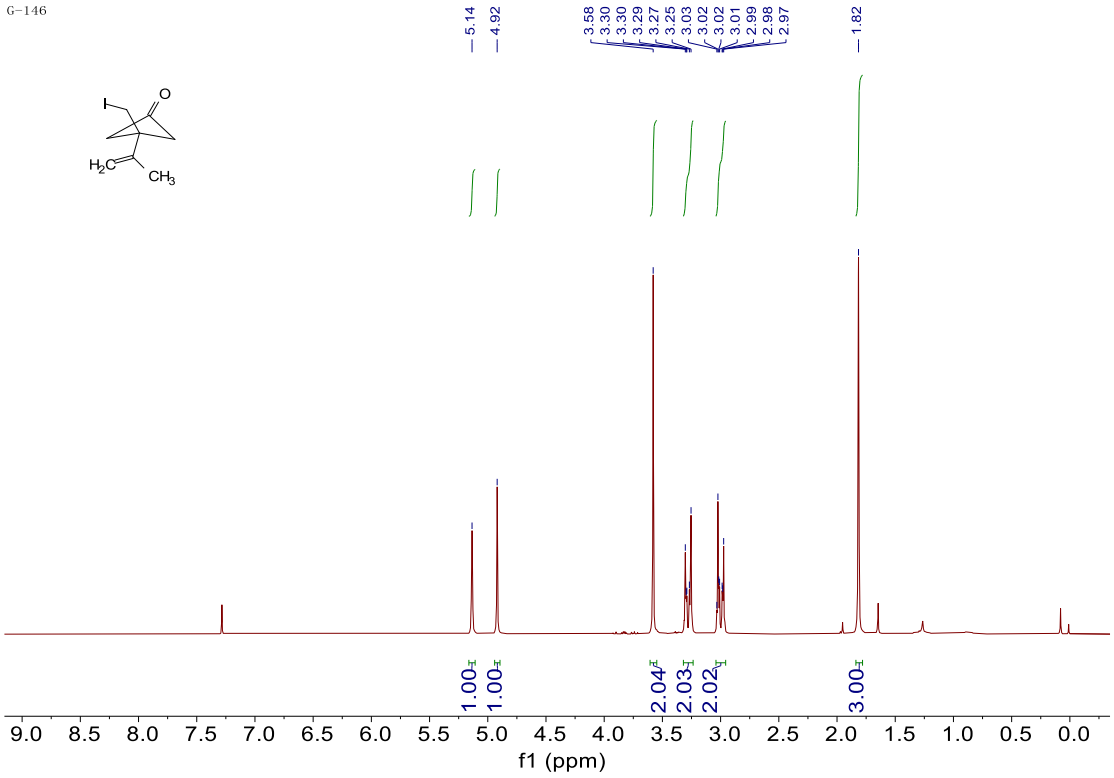
^{13}C NMR of compound **8**



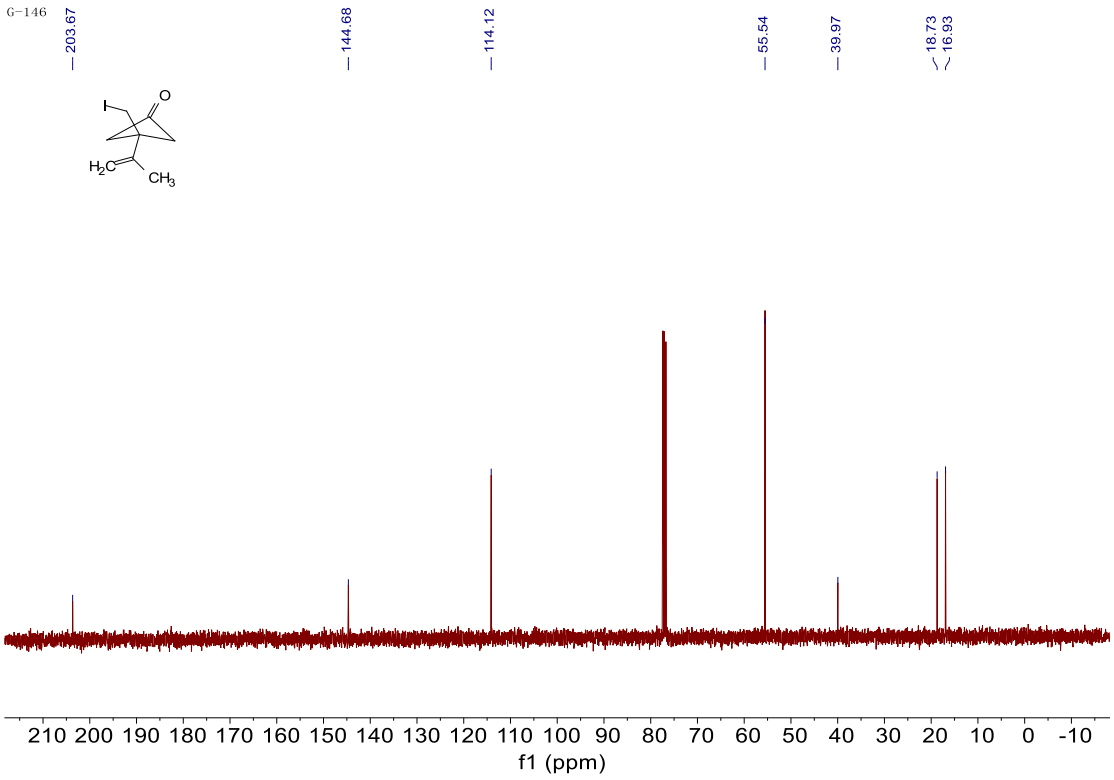
^{19}F NMR of compound **8**



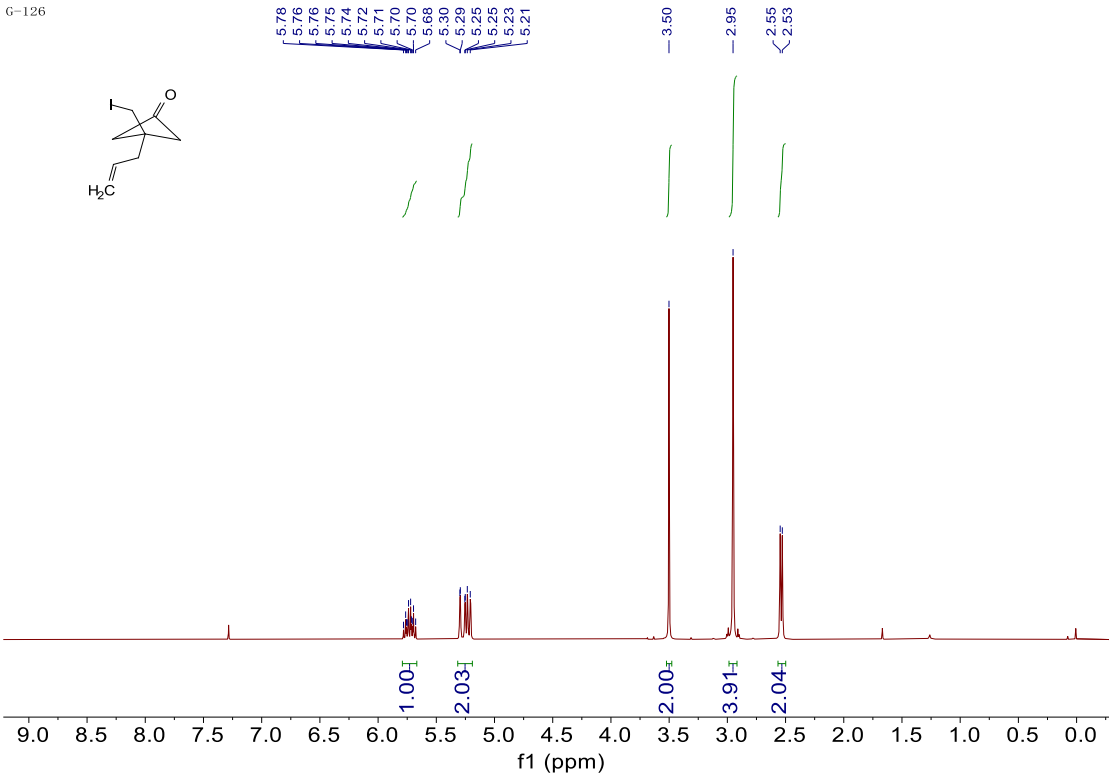
^{1}H NMR of compound **9**



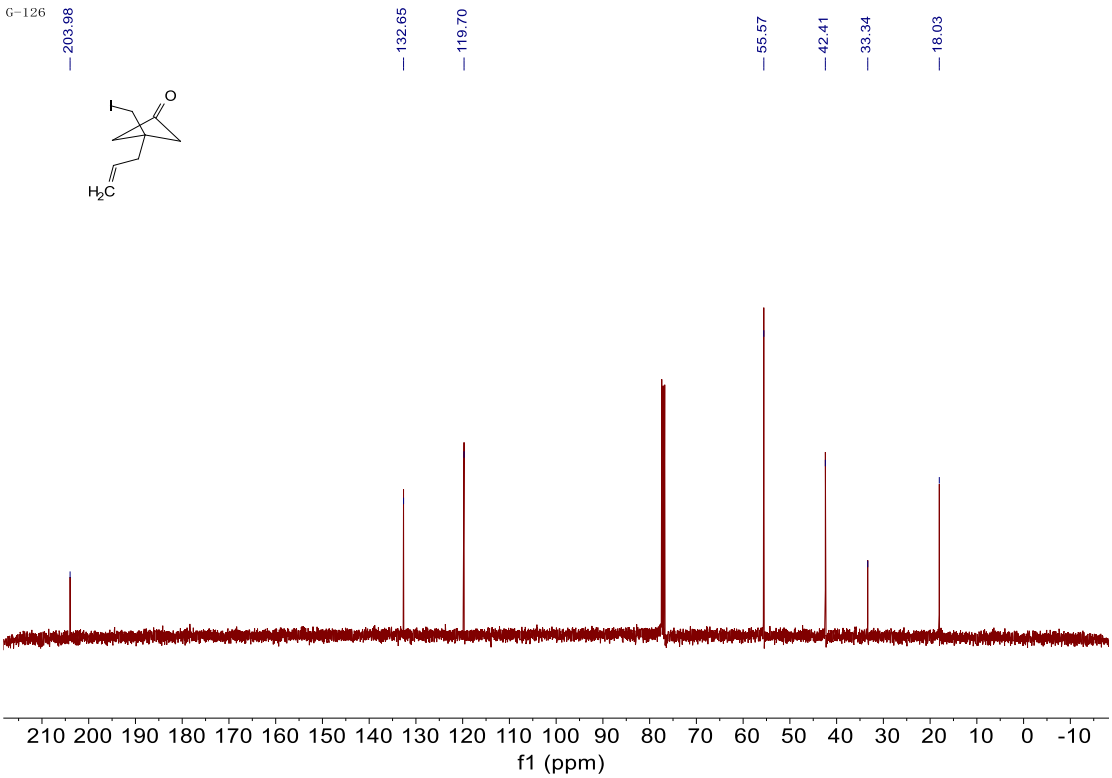
^{13}C NMR of compound **9**



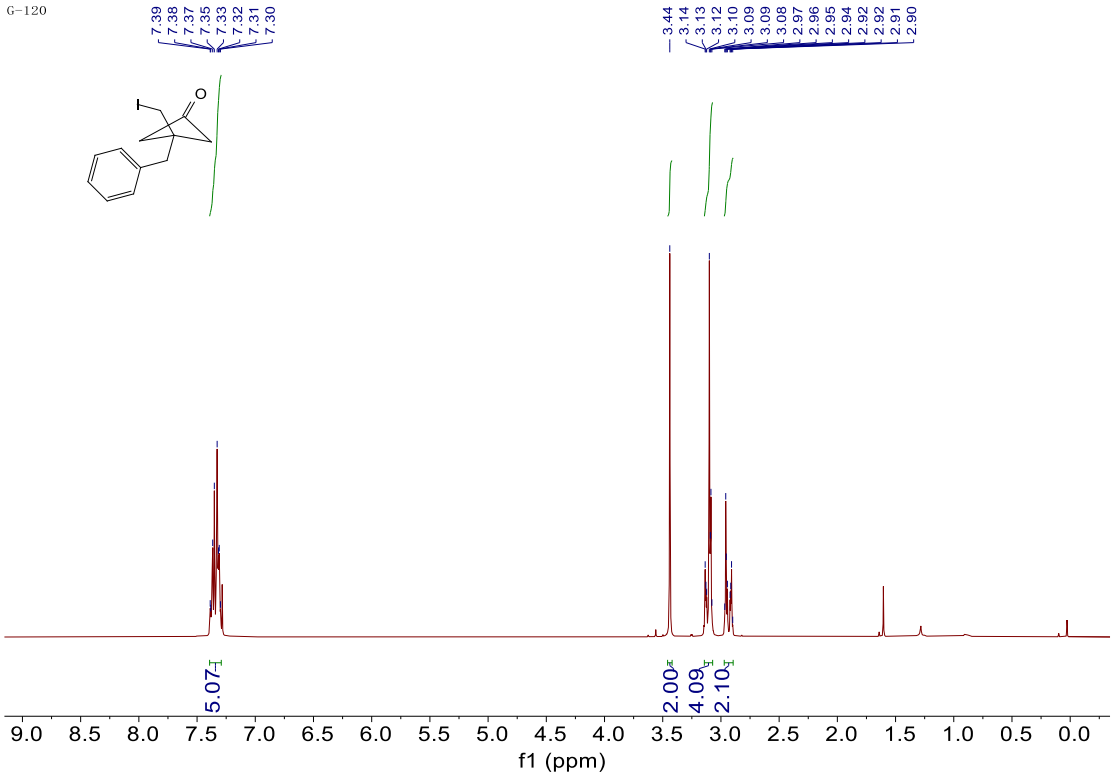
^{1}H NMR of compound **10**



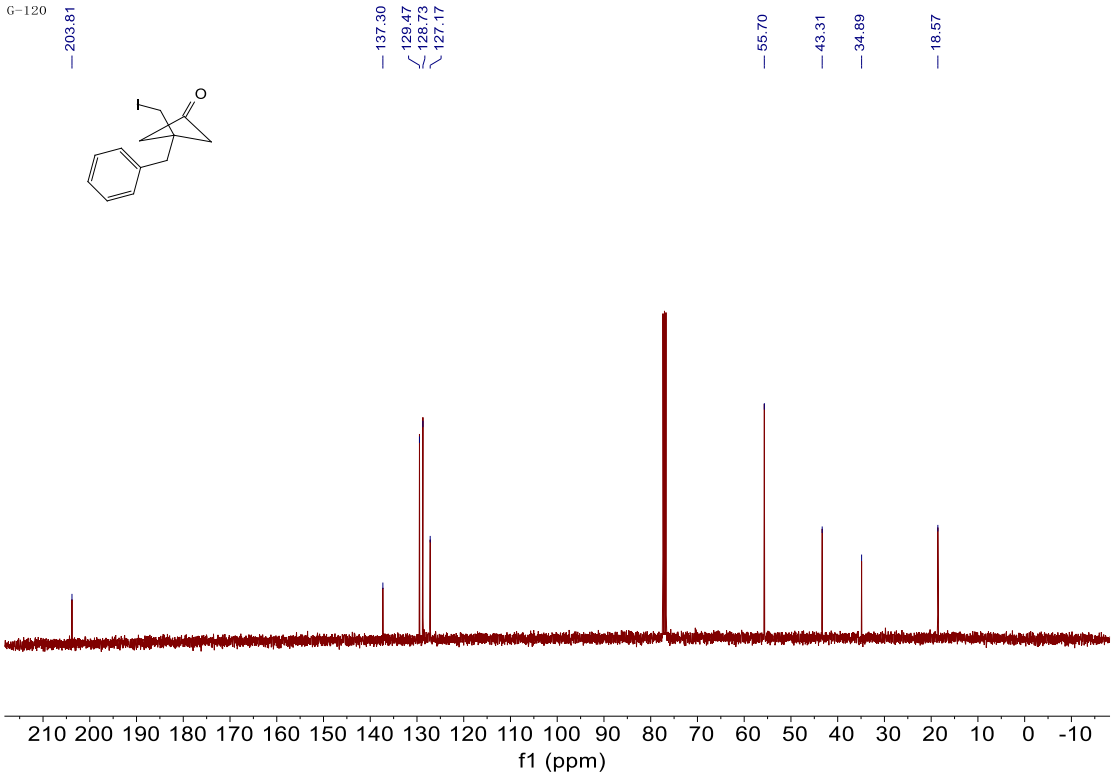
^{13}C NMR of compound **10**



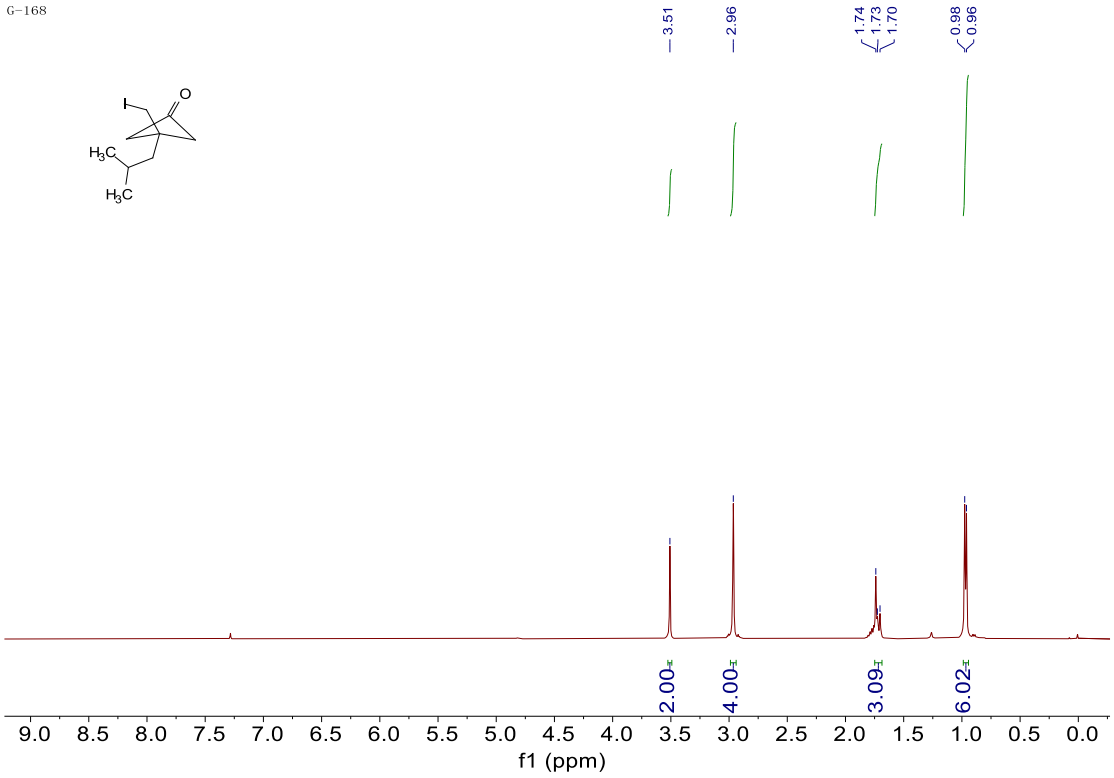
^{1}H NMR of compound **11**



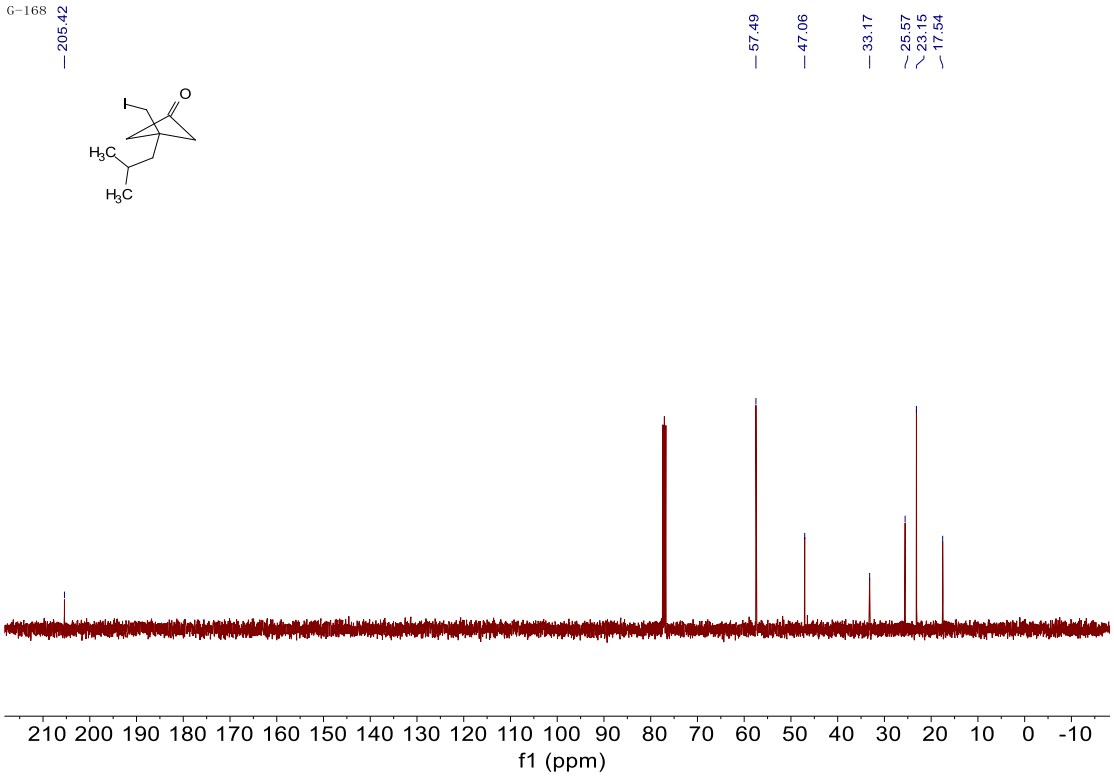
^{13}C NMR of compound **11**



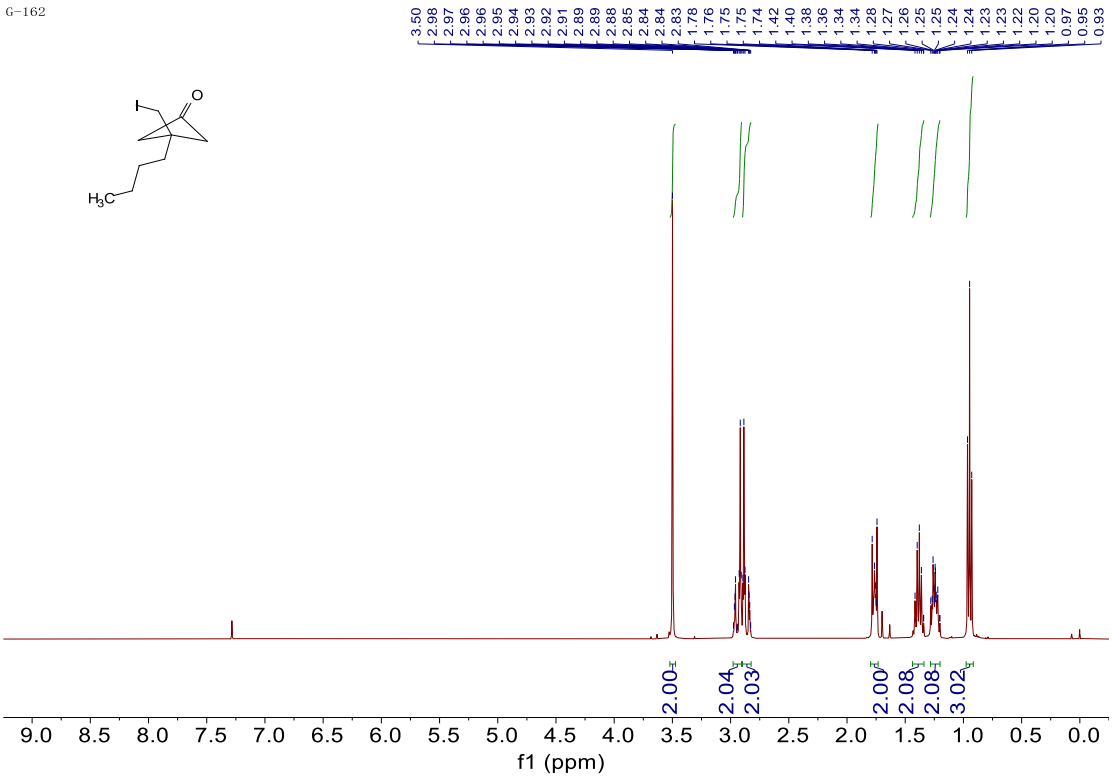
^{1}H NMR of compound **12**



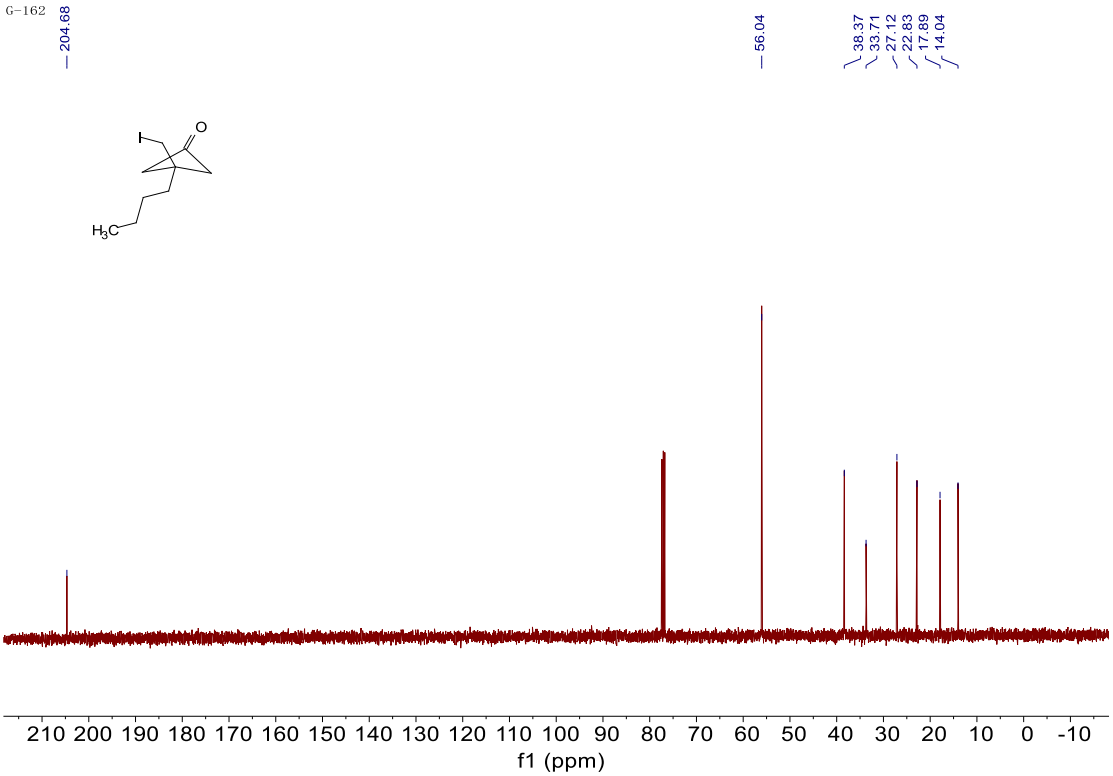
^{13}C NMR of compound **12**



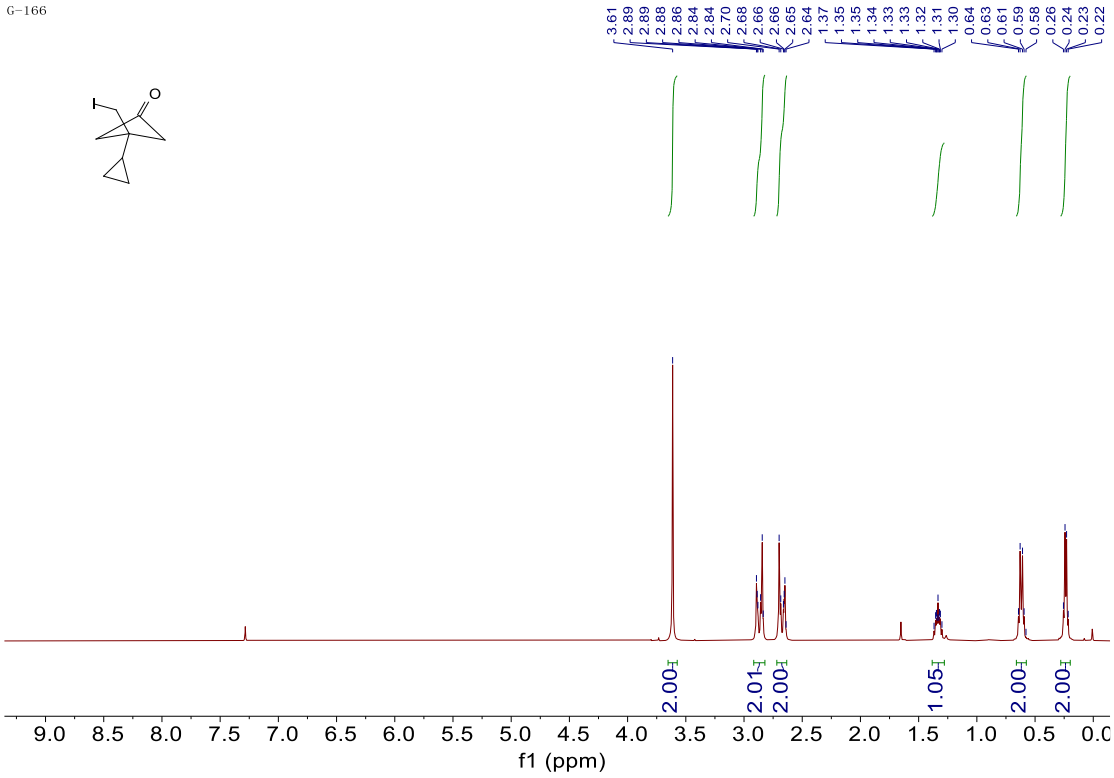
^{1}H NMR of compound **13**



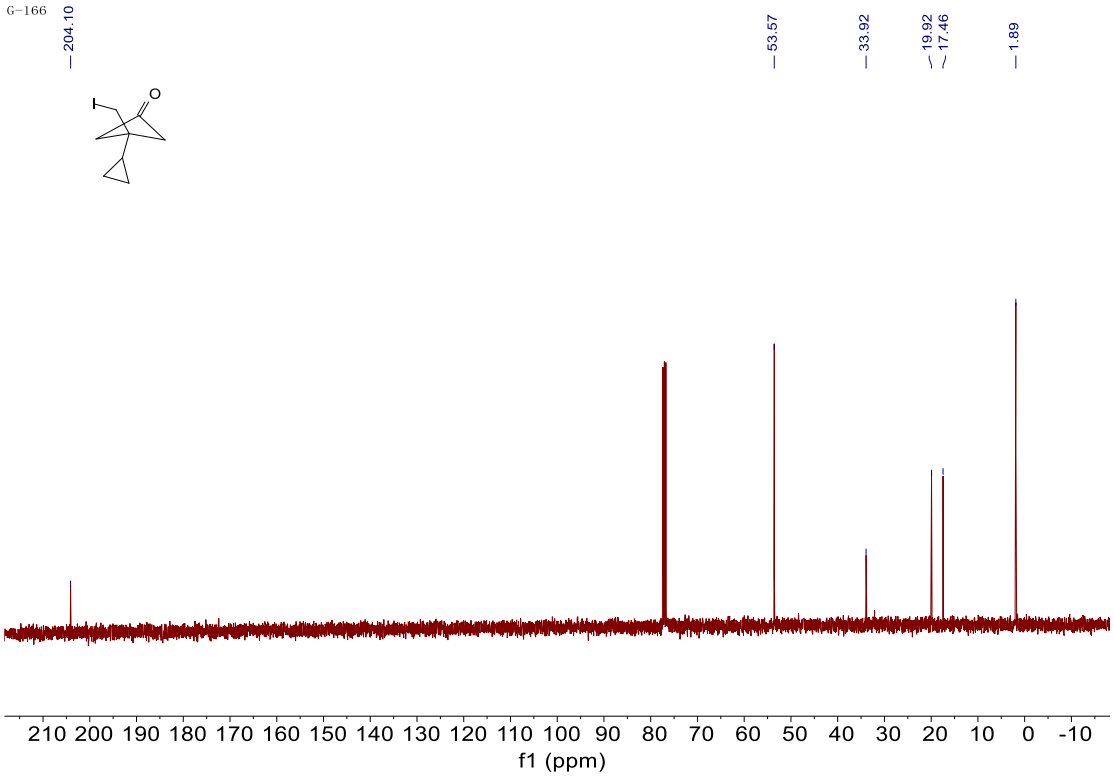
^{13}C NMR of compound **13**



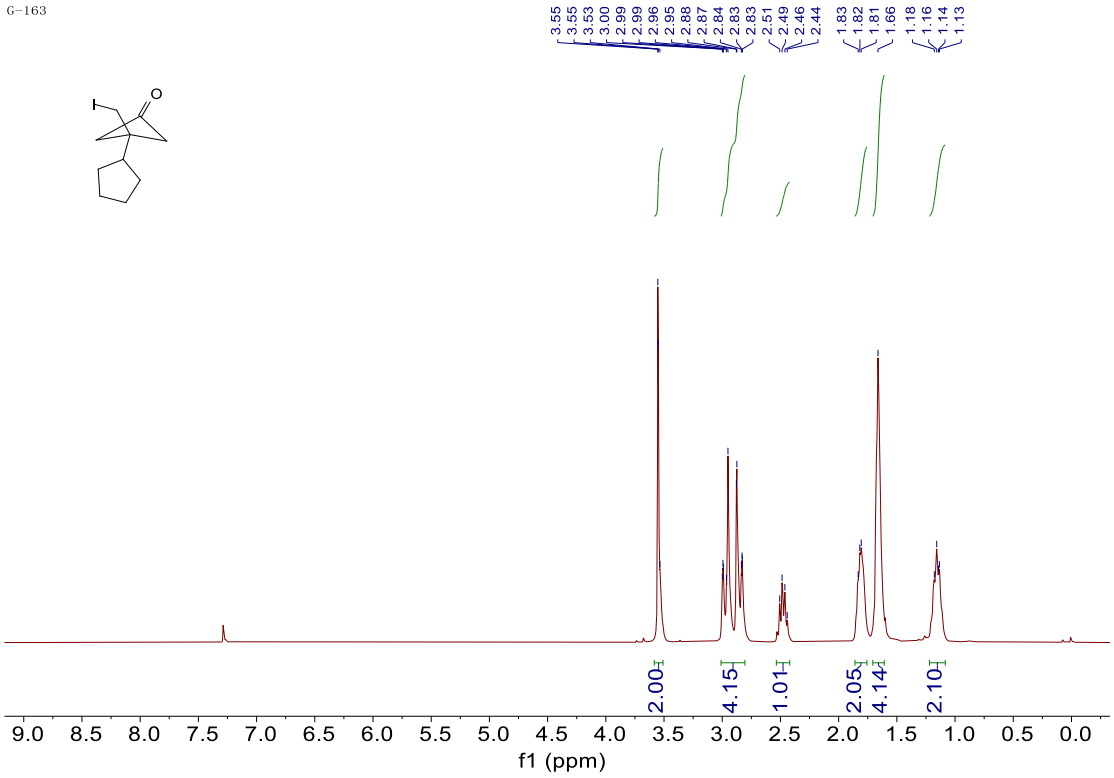
^{1}H NMR of compound **14**



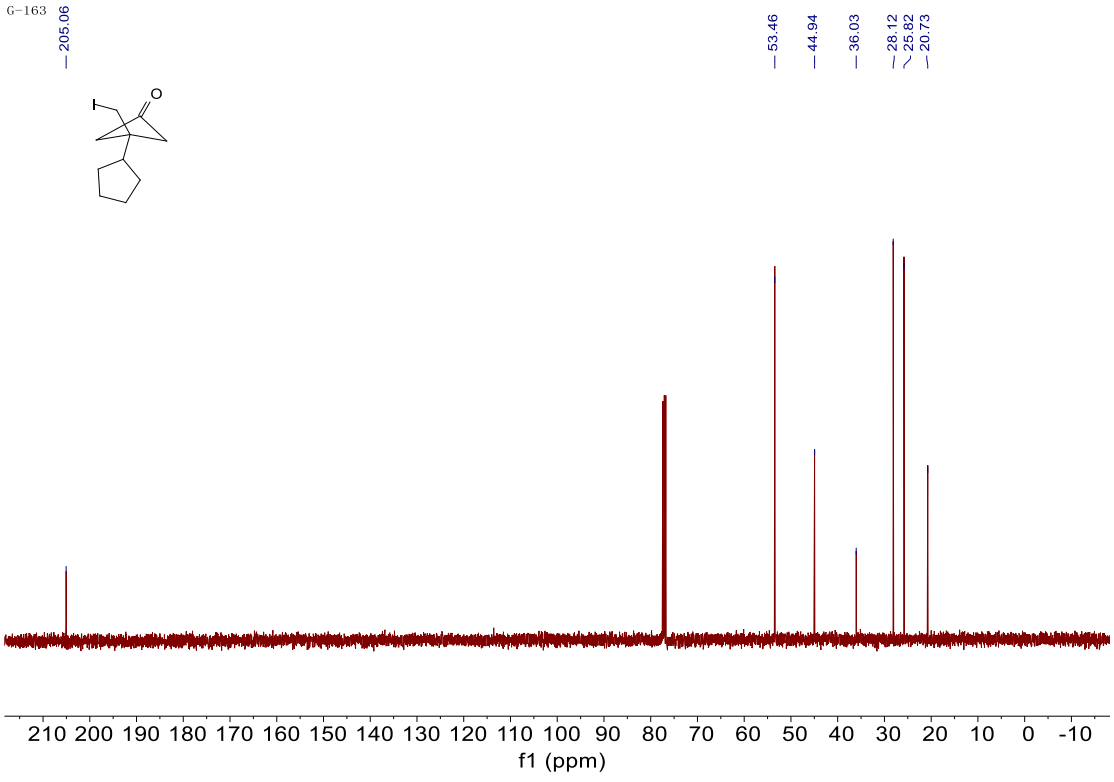
^{13}C NMR of compound **14**



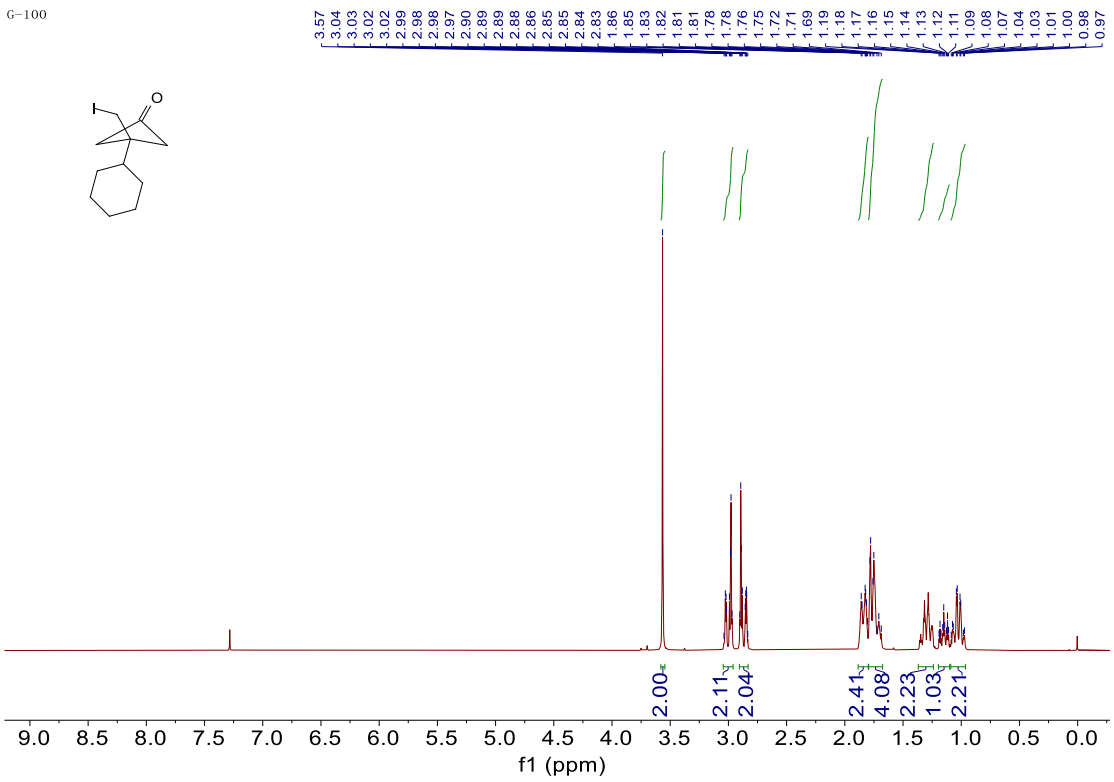
^{1}H NMR of compound **15**



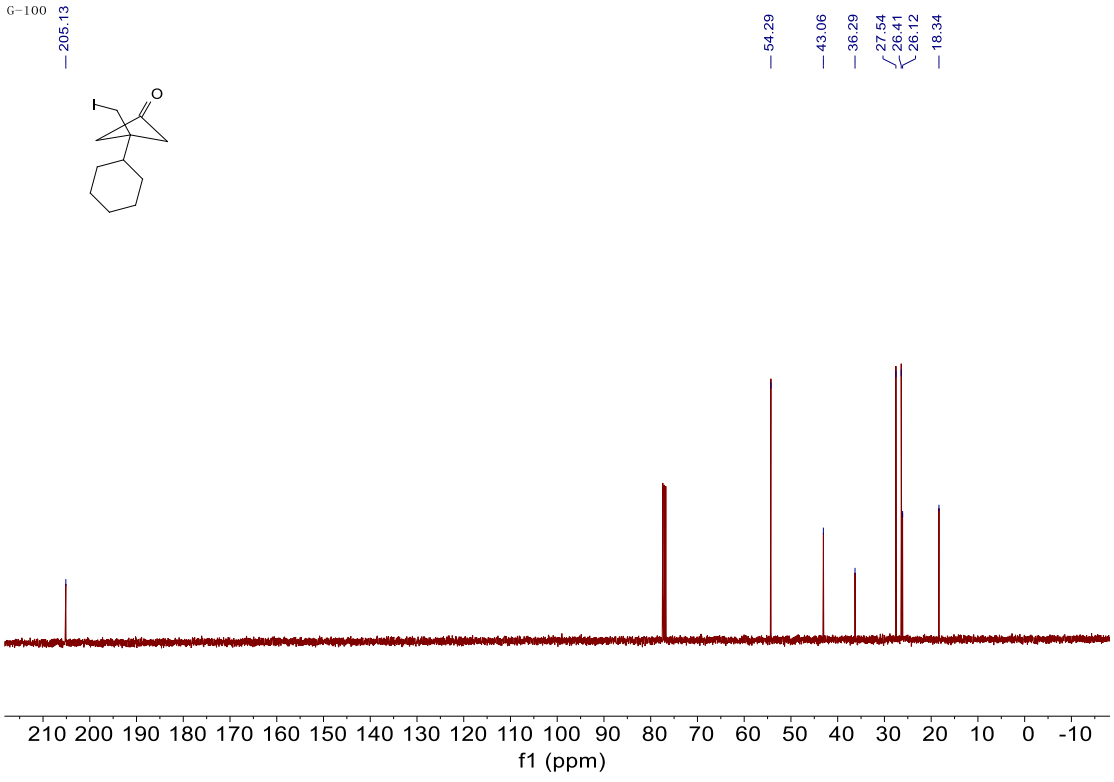
^{13}C NMR of compound **15**



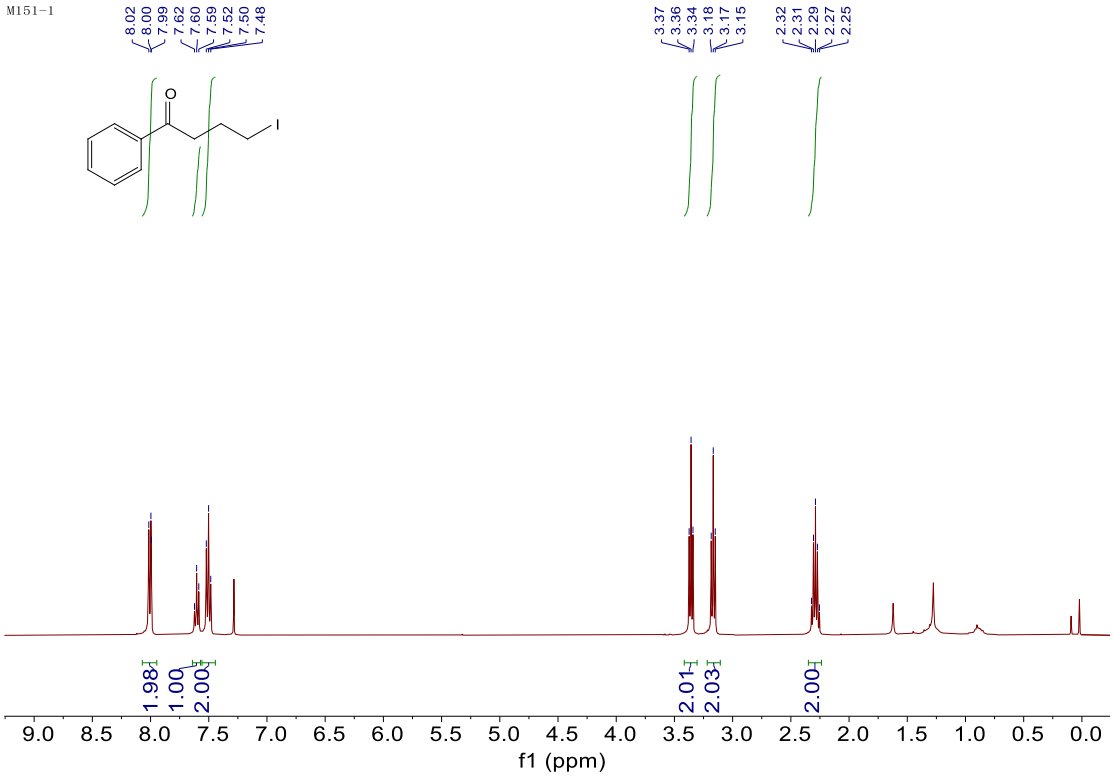
^{1}H NMR of compound **16**



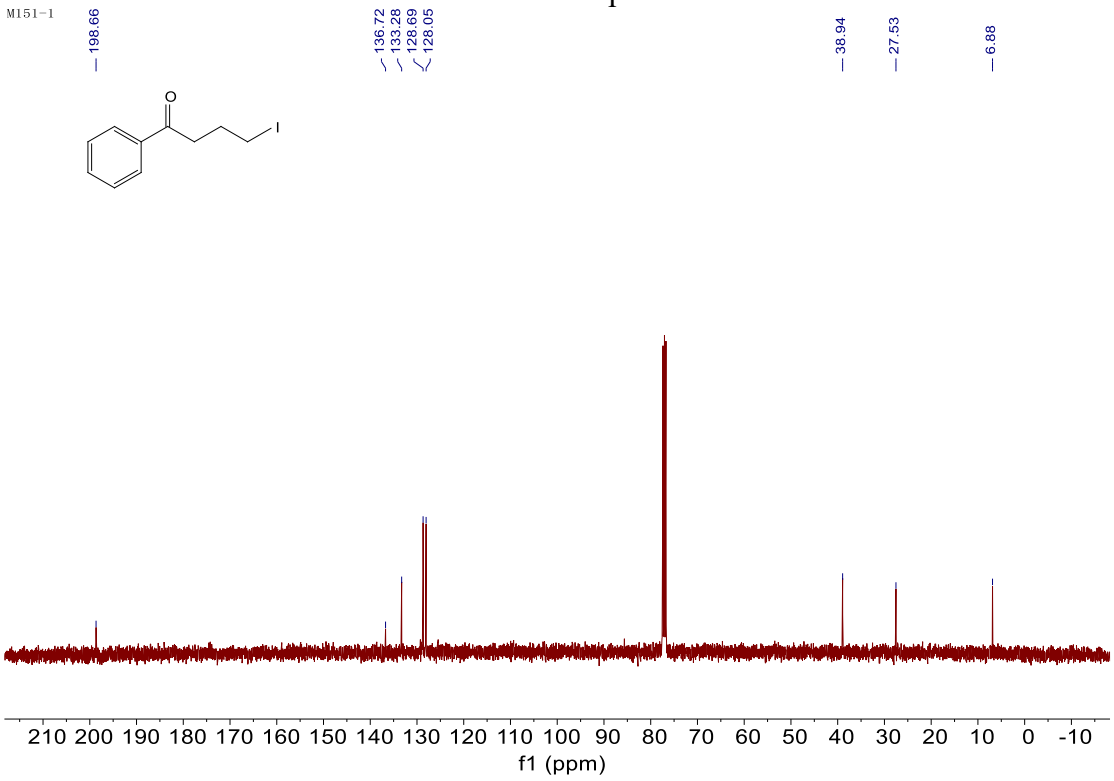
^{13}C NMR of compound **16**



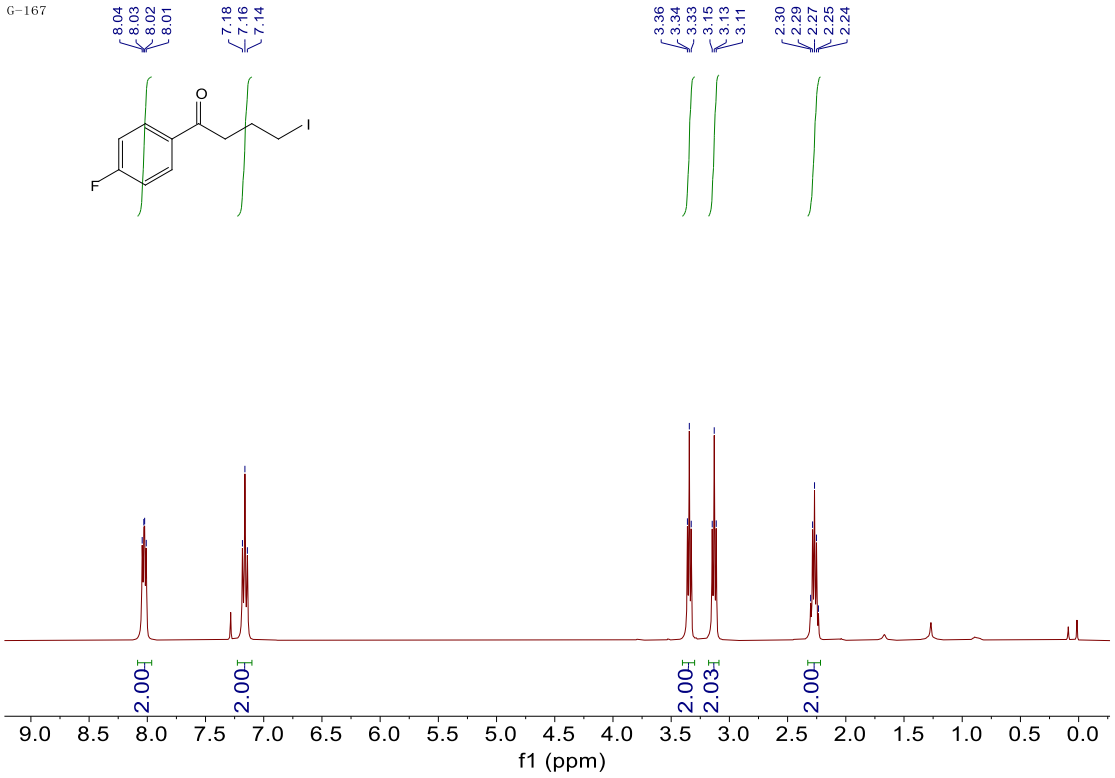
^{1}H NMR of compound **17**



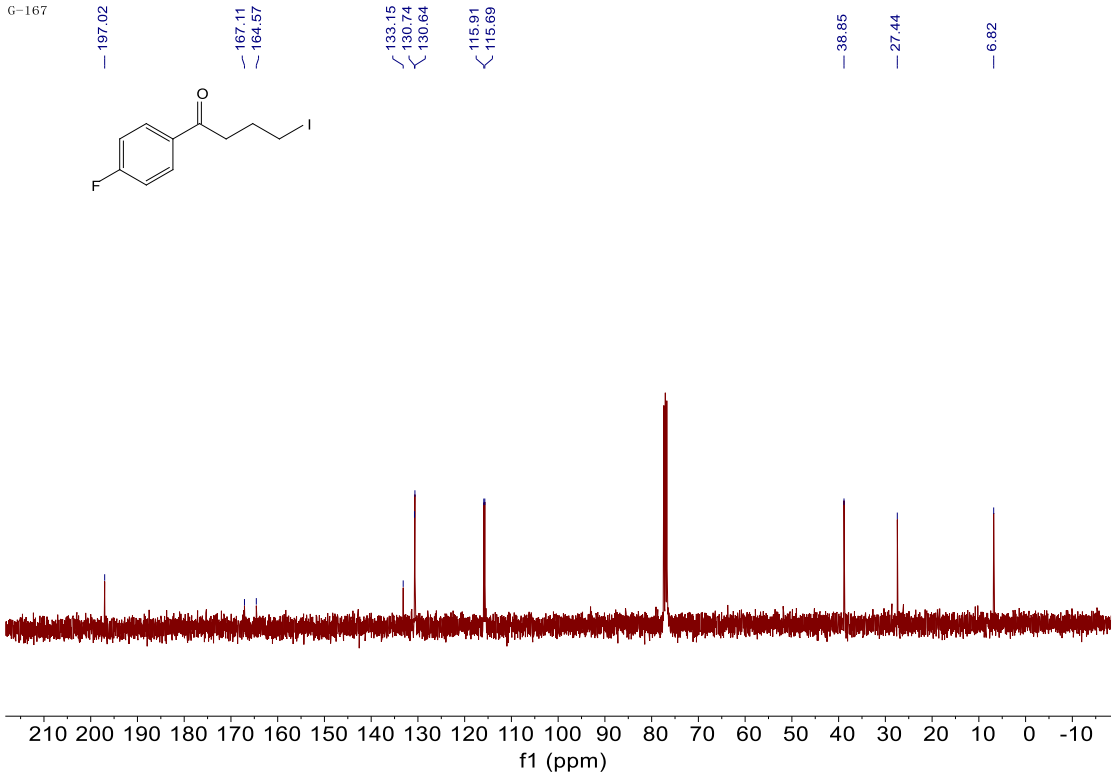
^{13}C NMR of compound **17**



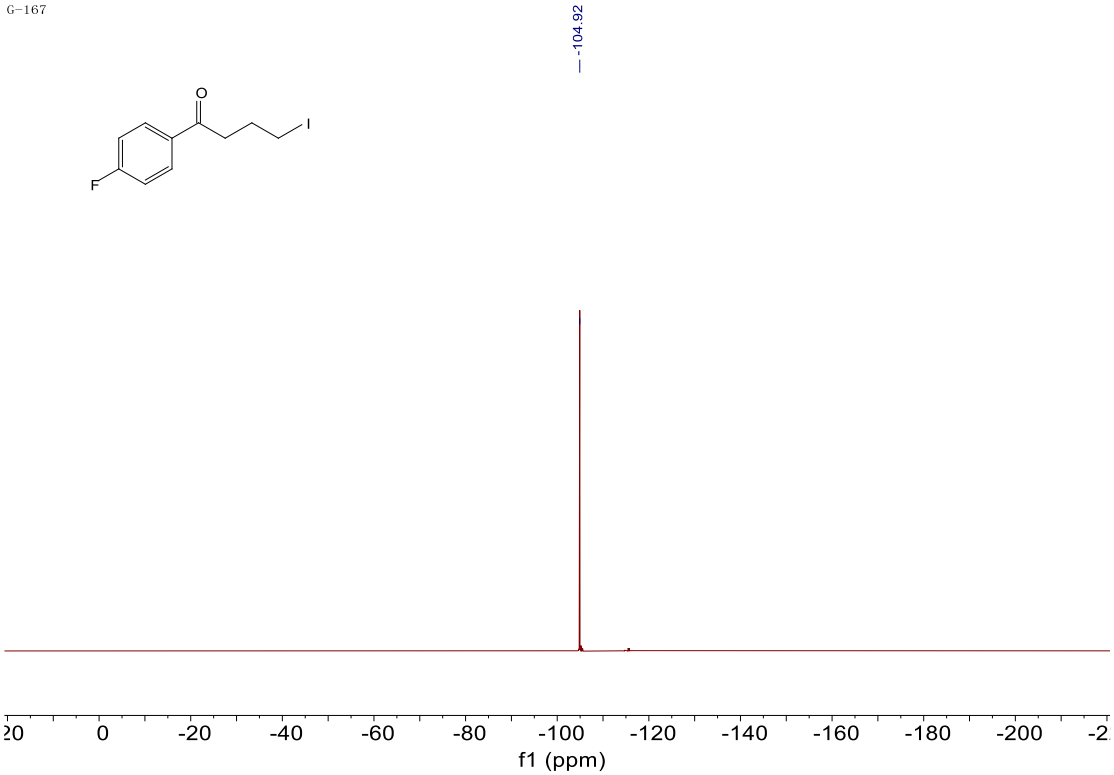
^{1}H NMR of compound **18**



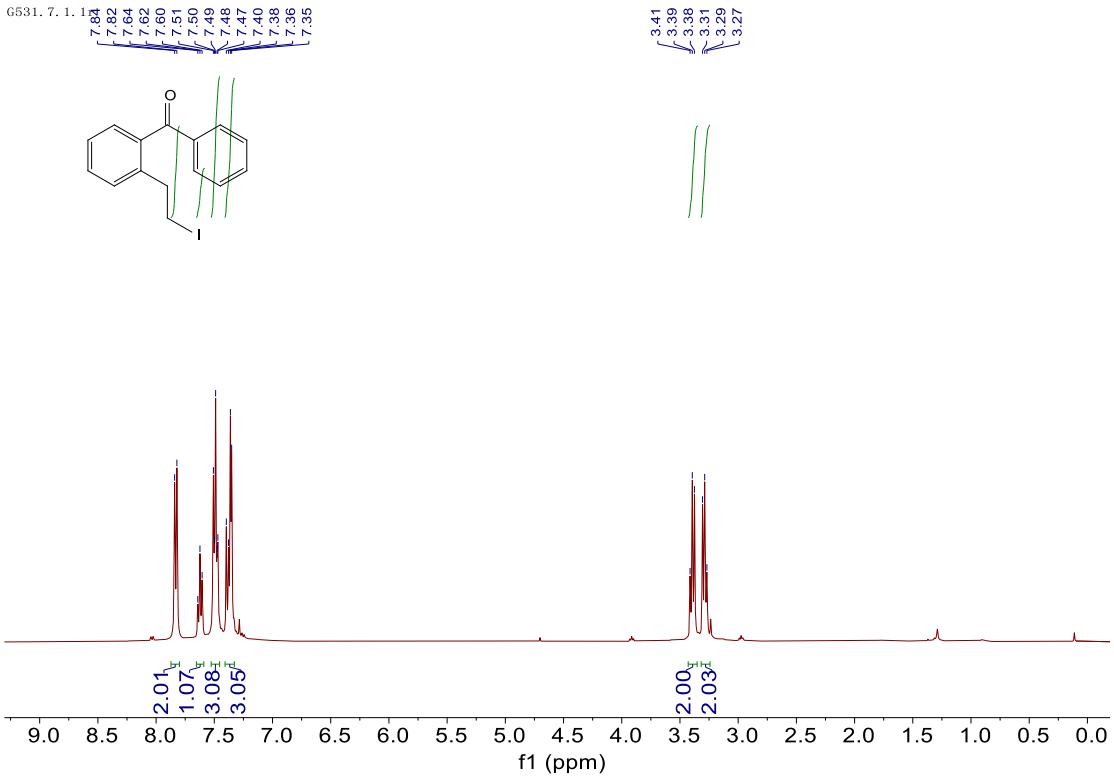
^{13}C NMR of compound **18**



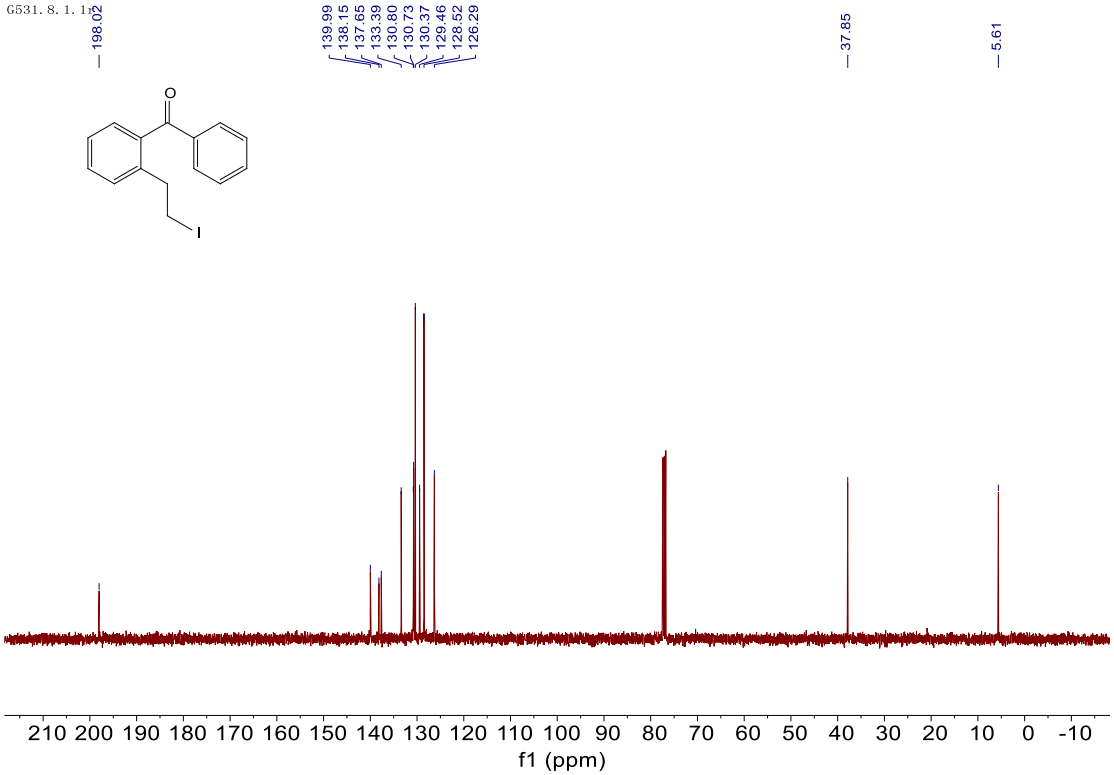
^{19}F NMR of compound **18**



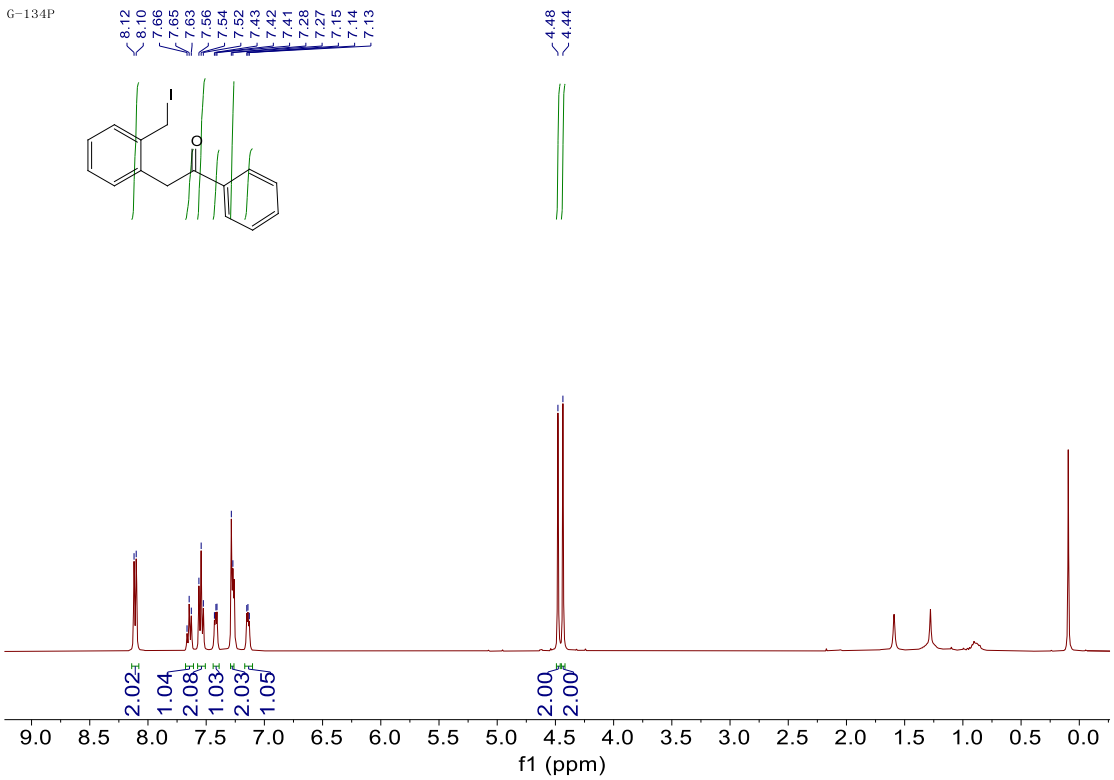
^{1}H NMR of compound **19**



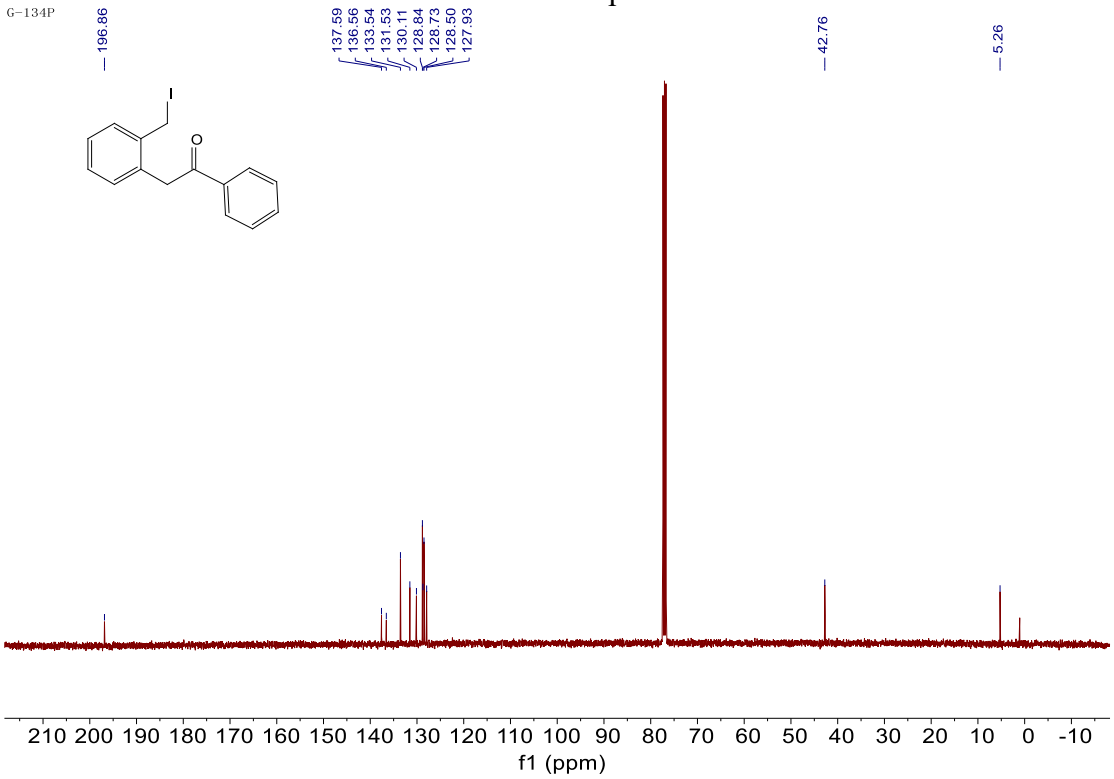
^{13}C NMR of compound **19**



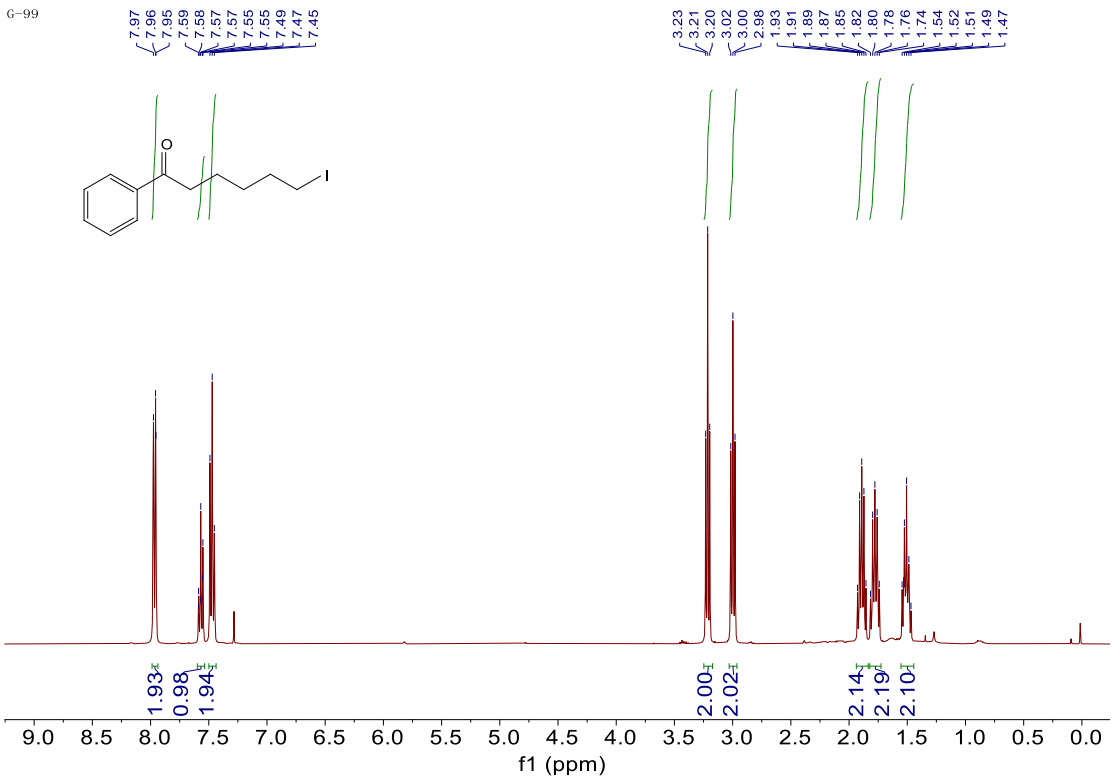
^{1}H NMR of compound **20**



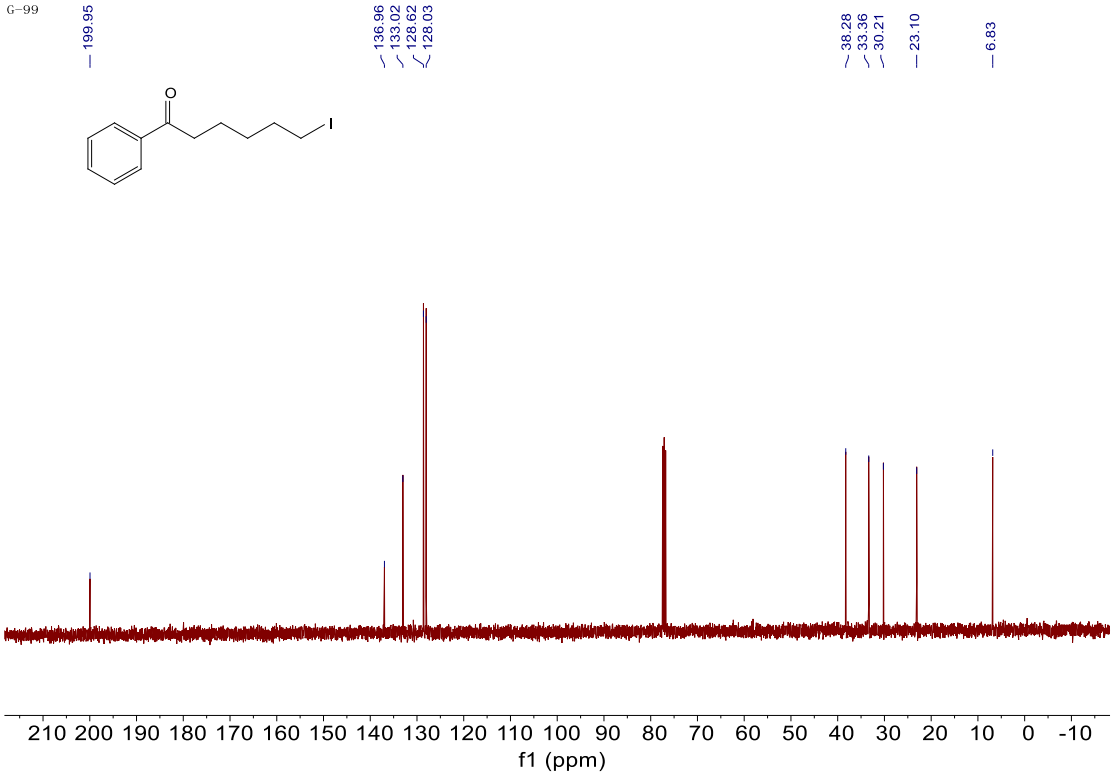
^{13}C NMR of compound **20**



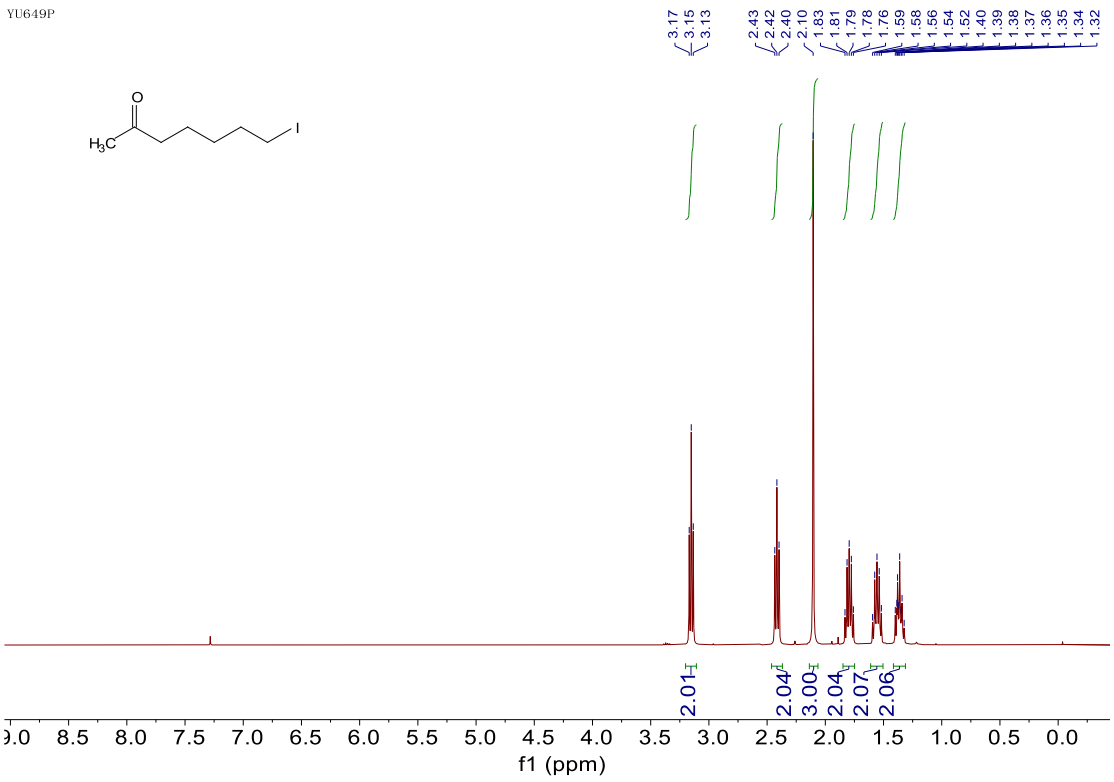
^{1}H NMR of compound **21**



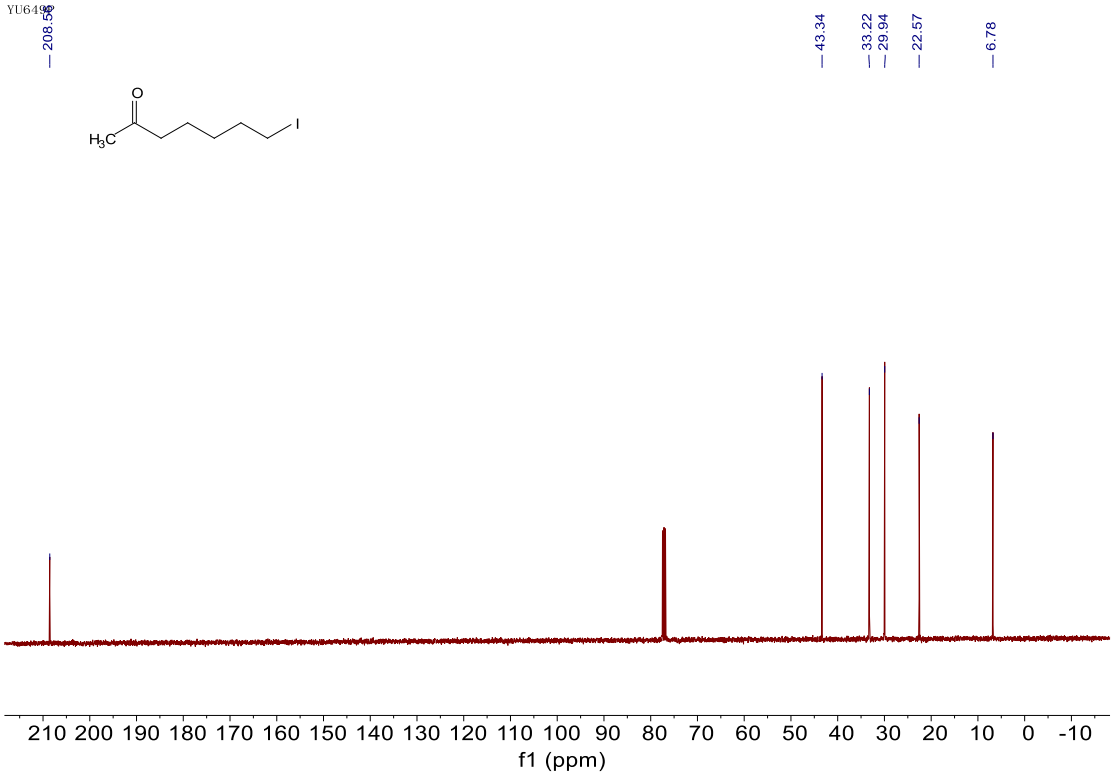
^{13}C NMR of compound **21**



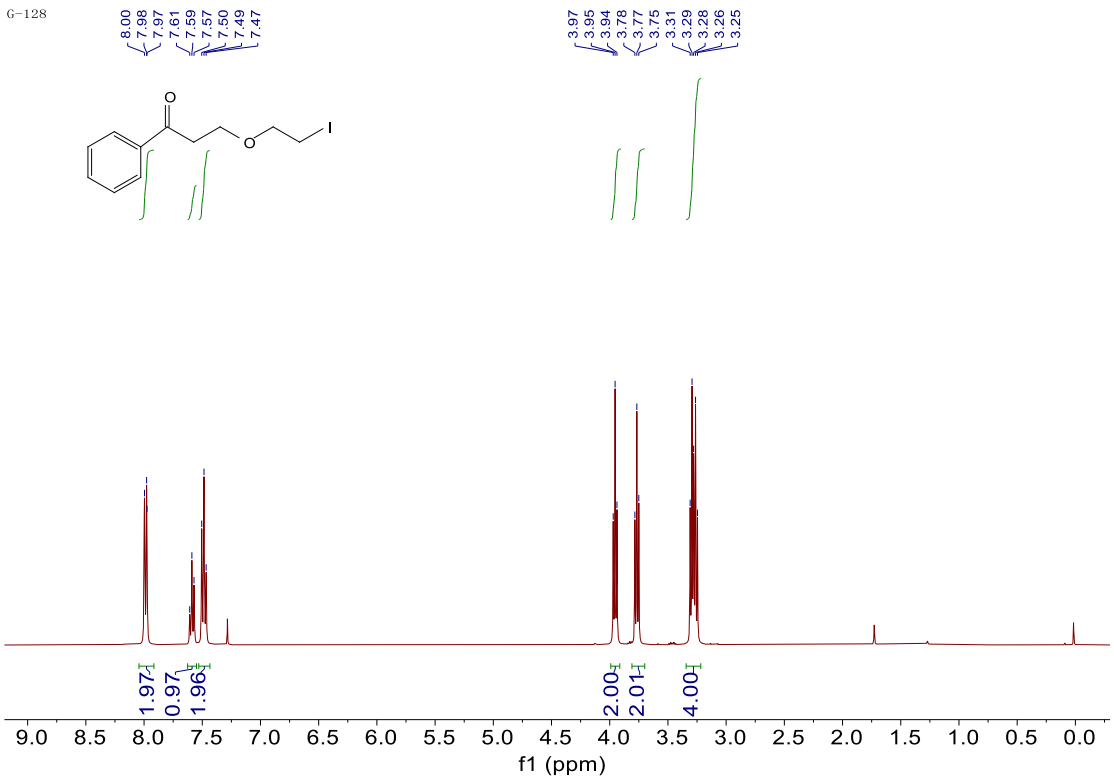
^{1}H NMR of compound **22**



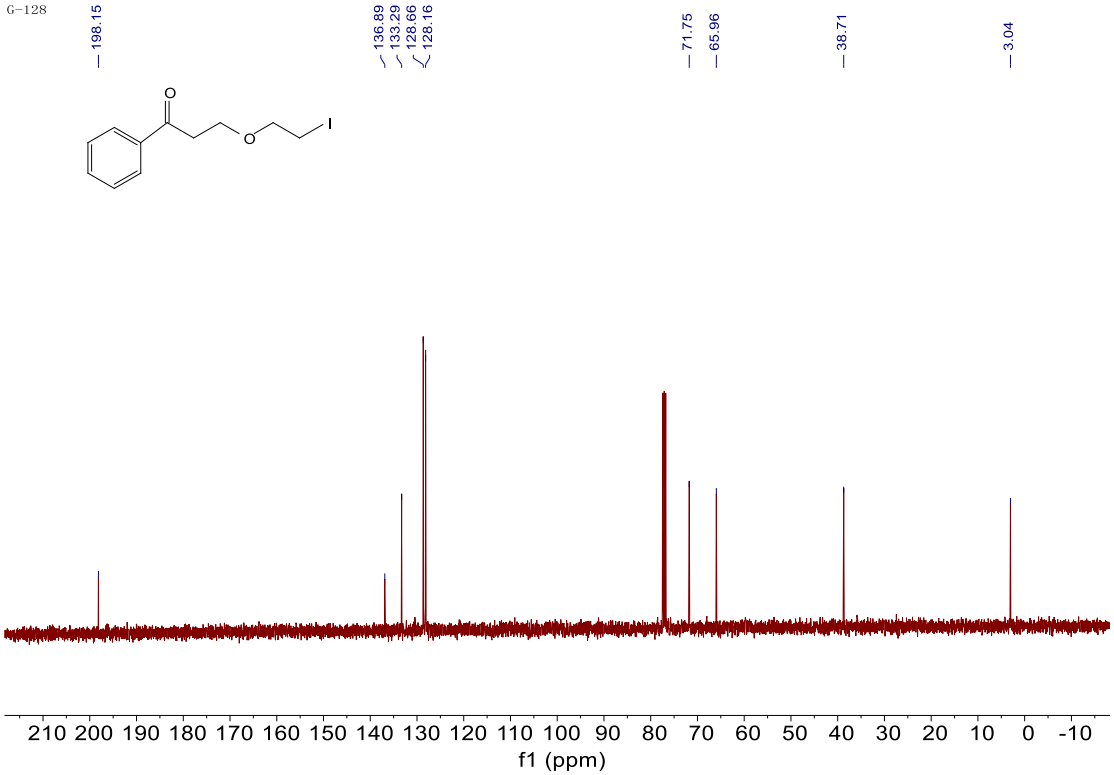
^{13}C NMR of compound **22**



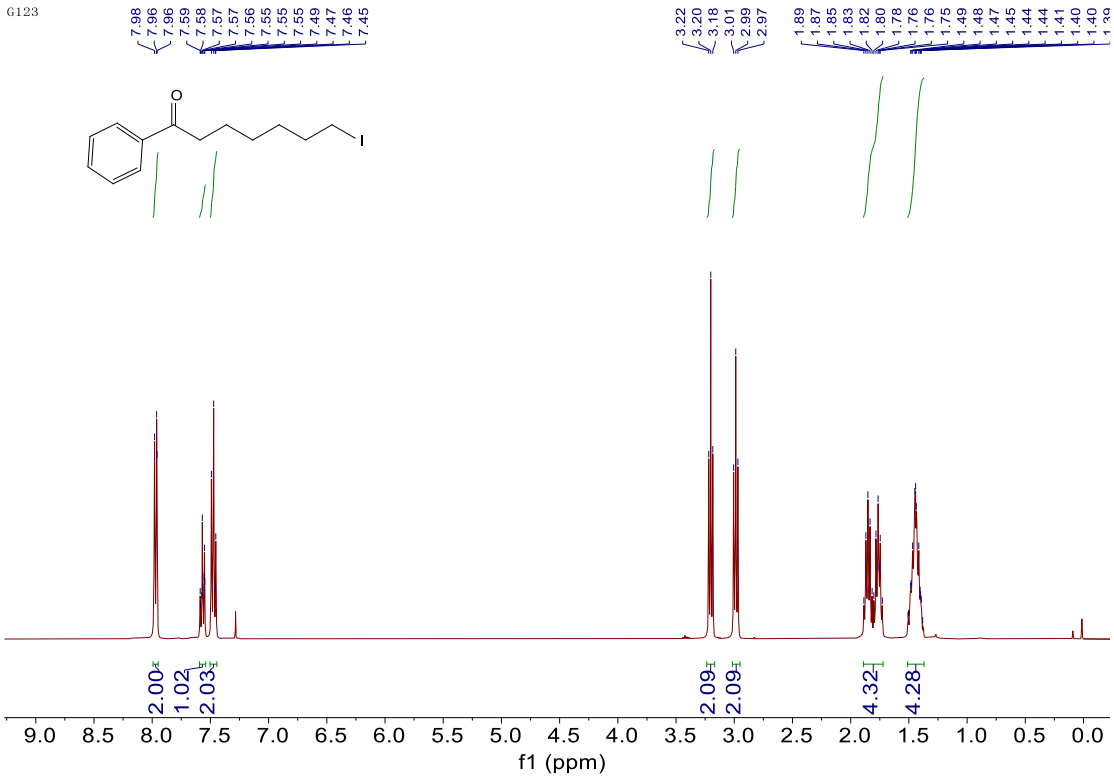
^{1}H NMR of compound **23**



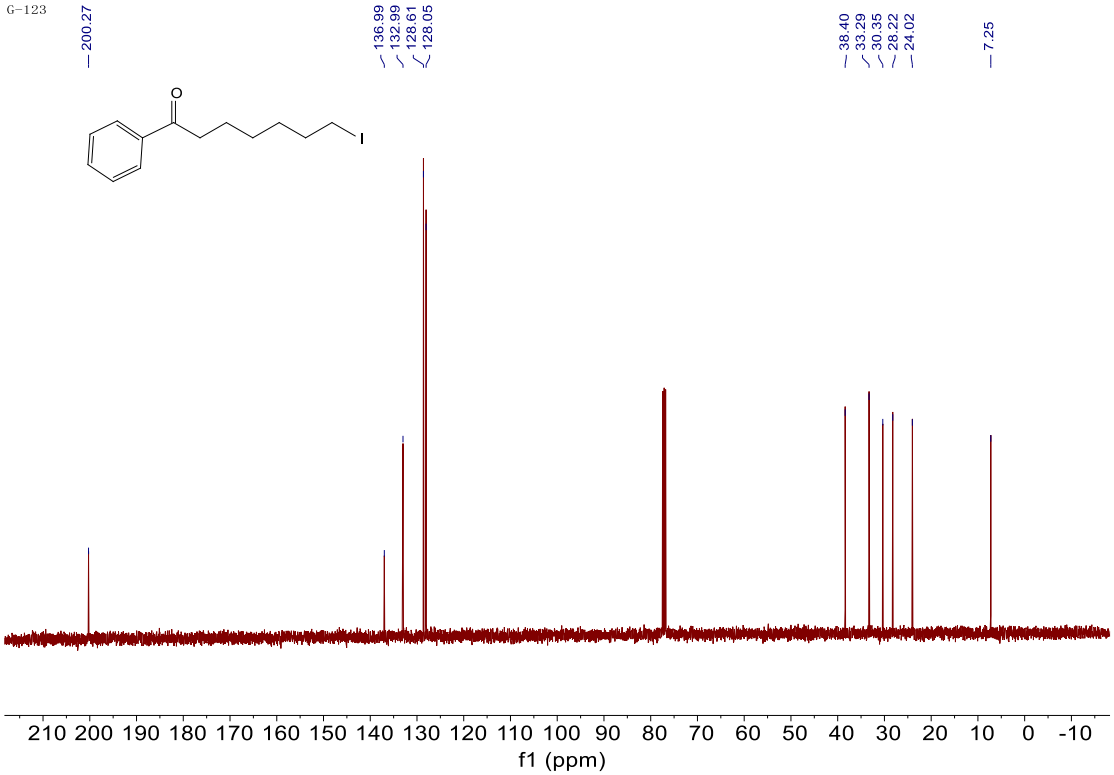
^{13}C NMR of compound **23**



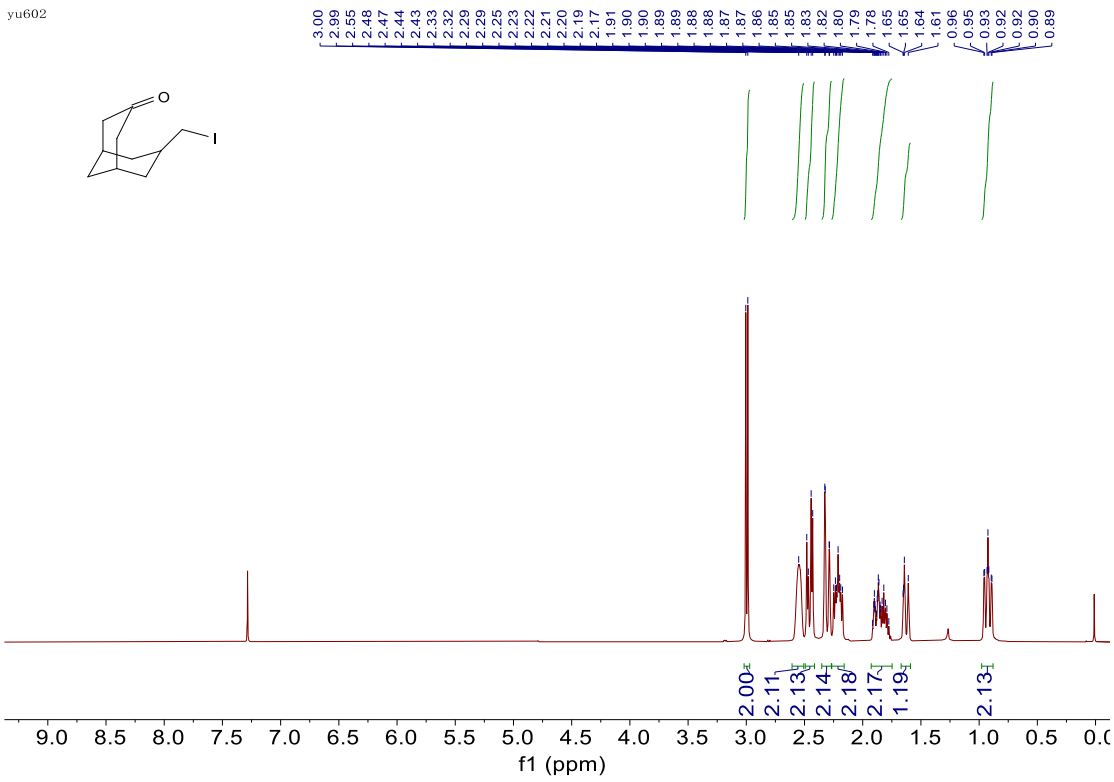
^{1}H NMR of compound **24**



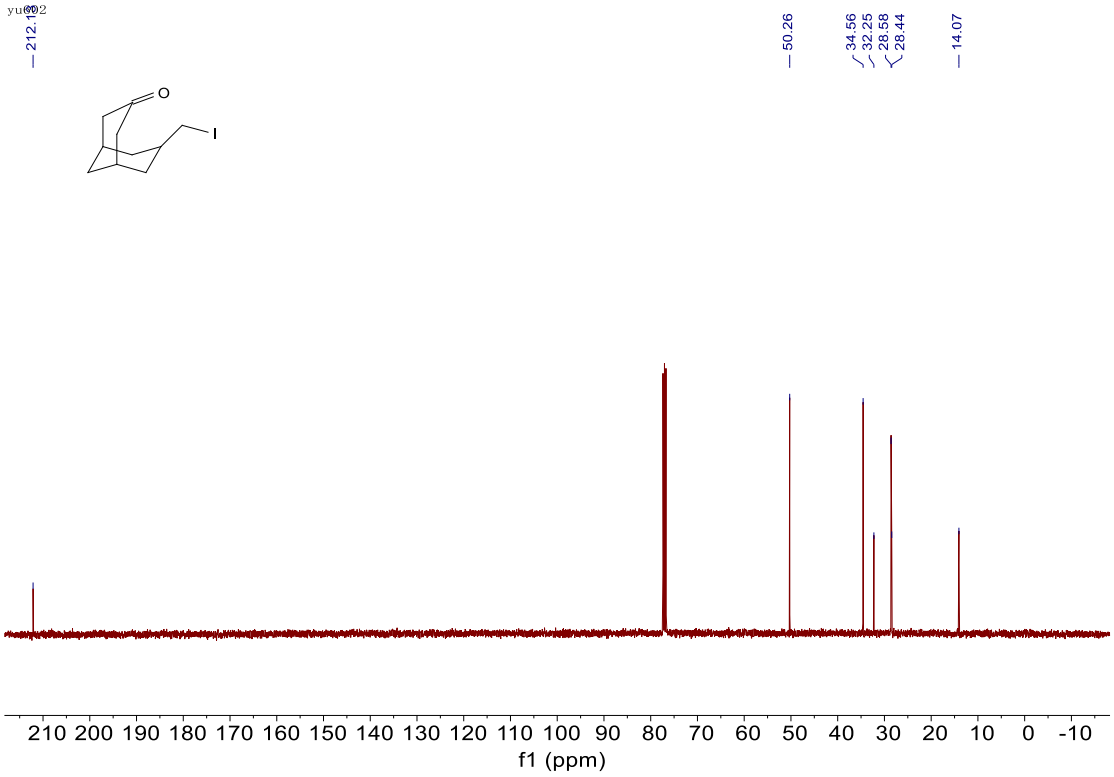
^{13}C NMR of compound **24**



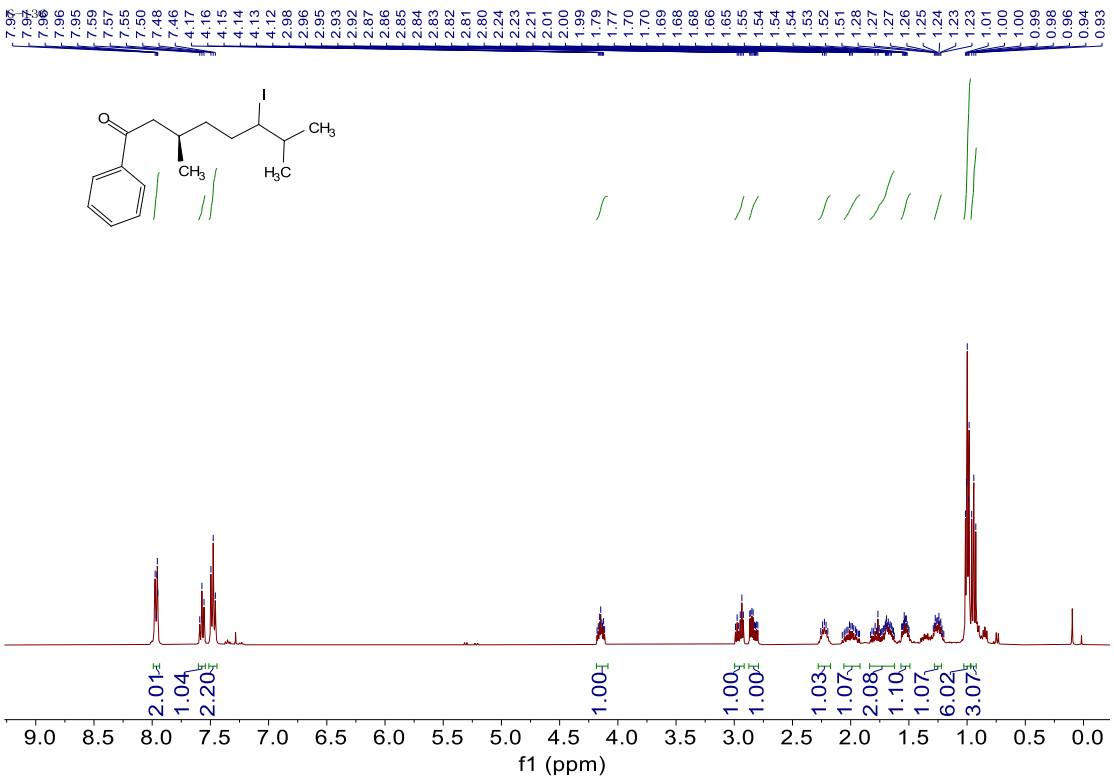
^{1}H NMR of compound **25**



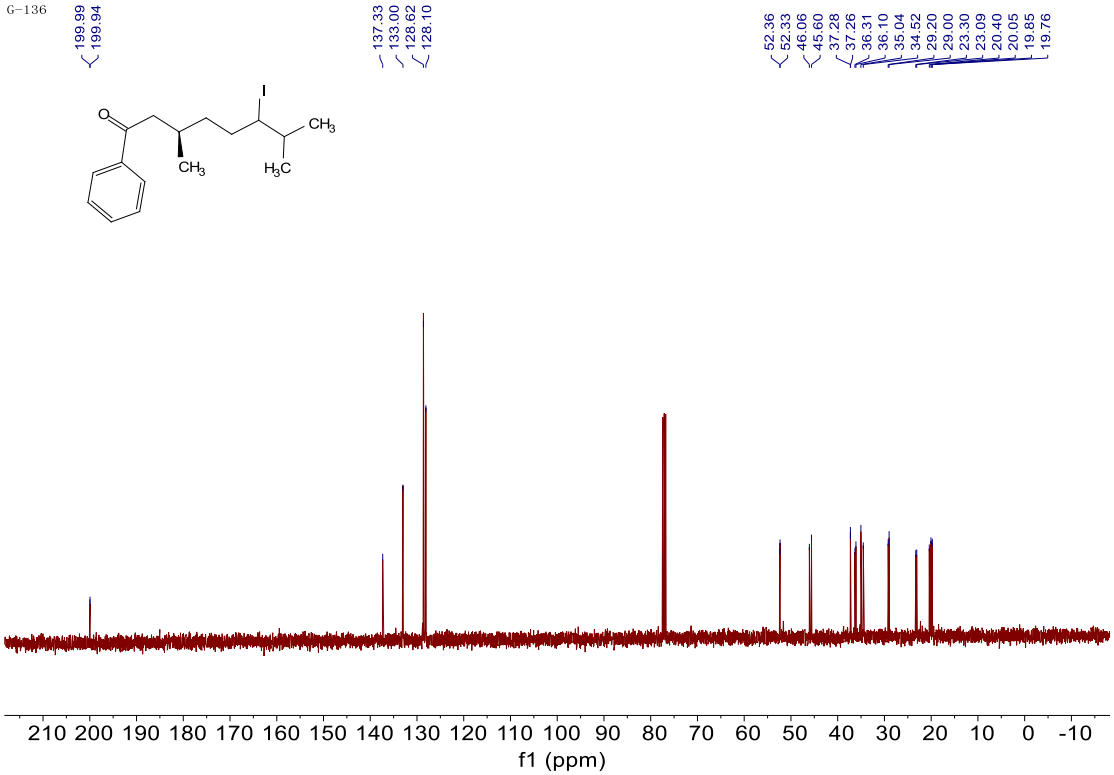
^{13}C NMR of compound **25**



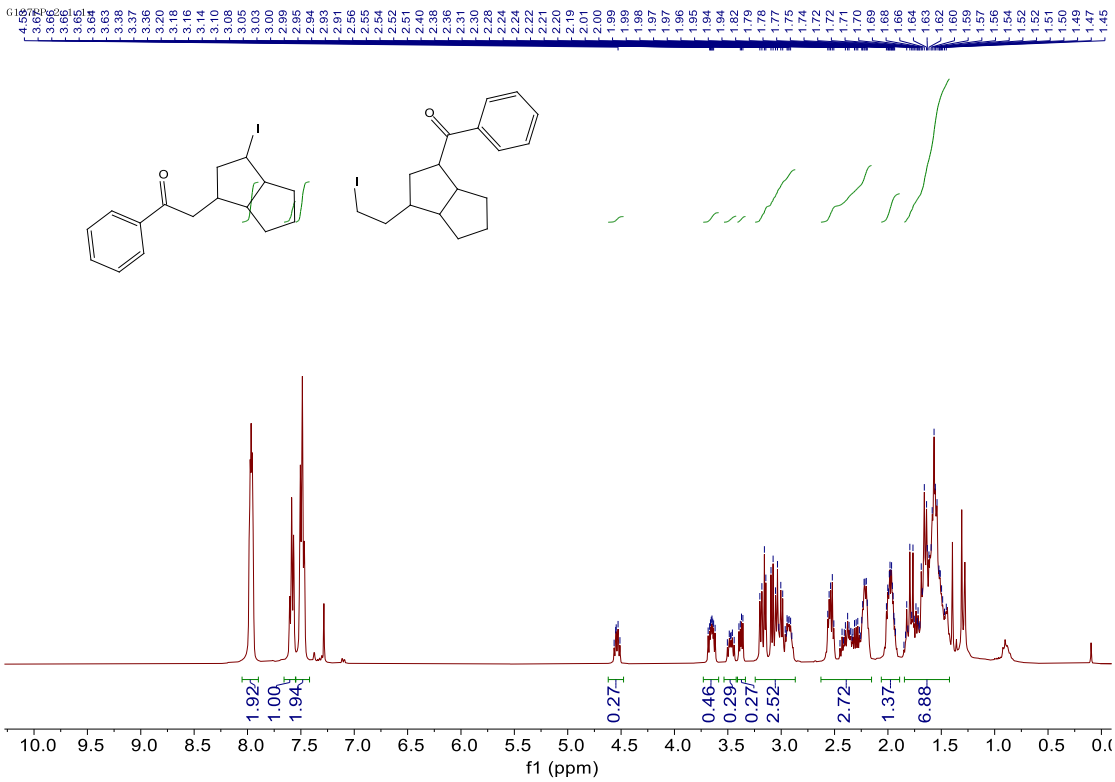
^{1}H NMR of compound **26**



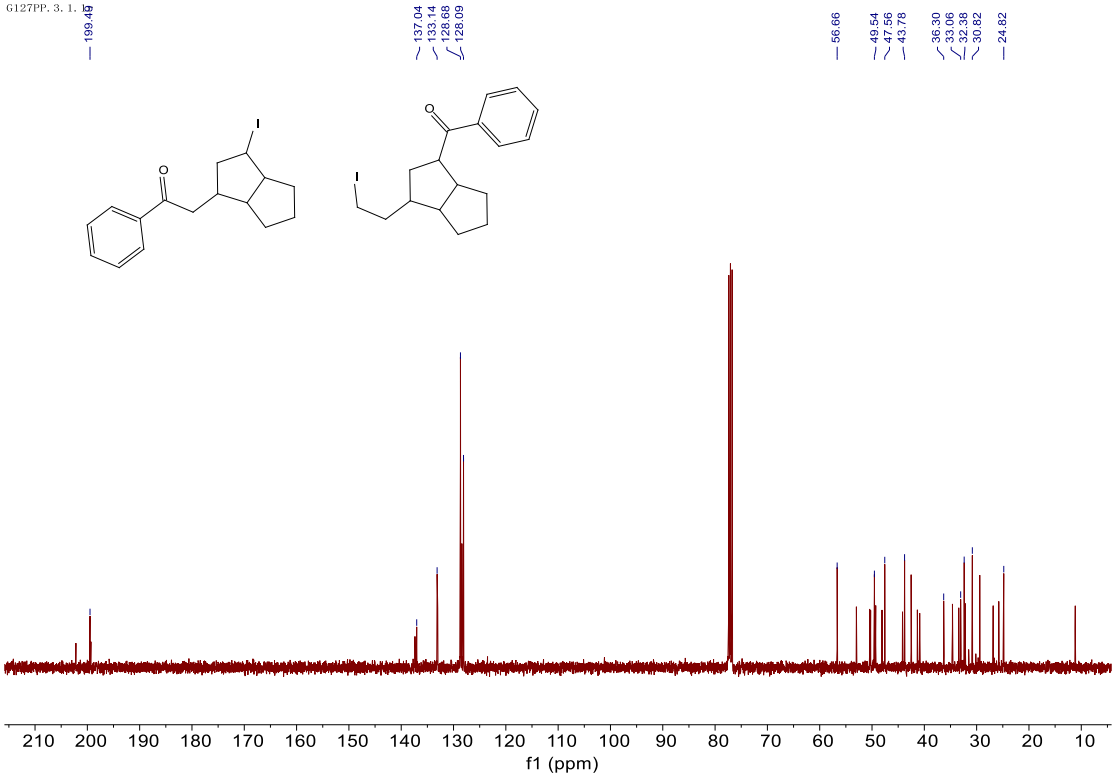
^{13}C NMR of compound **26**



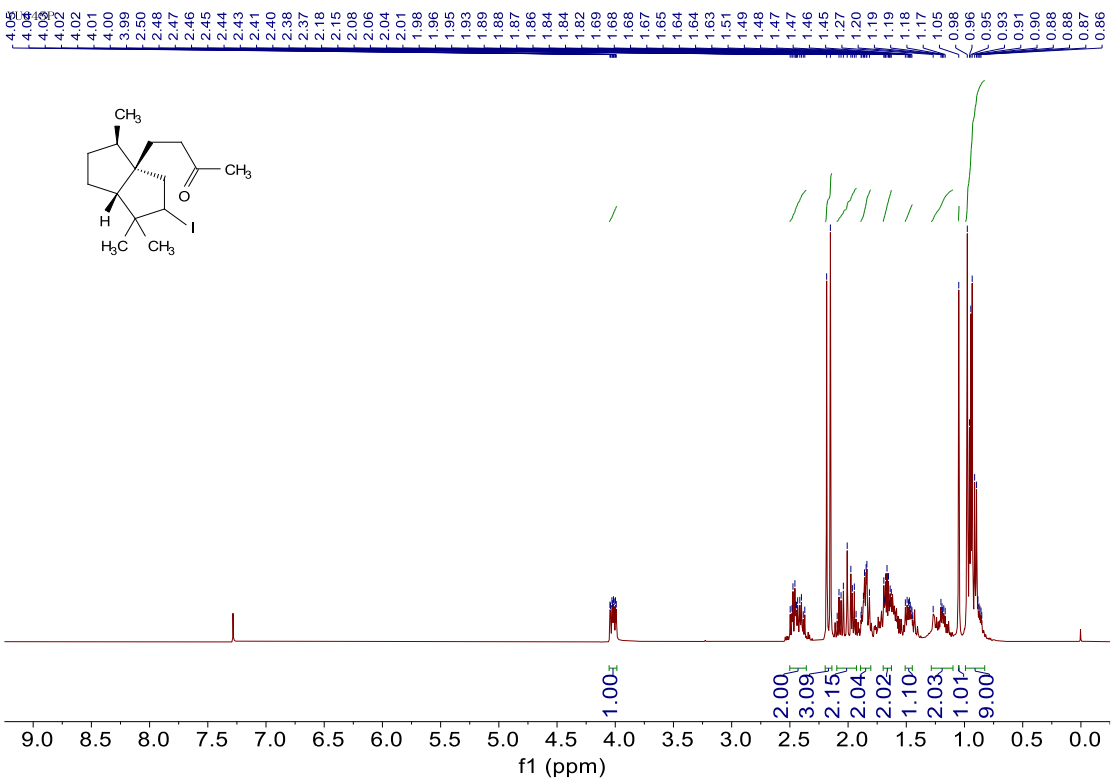
^{1}H NMR of compound **27**



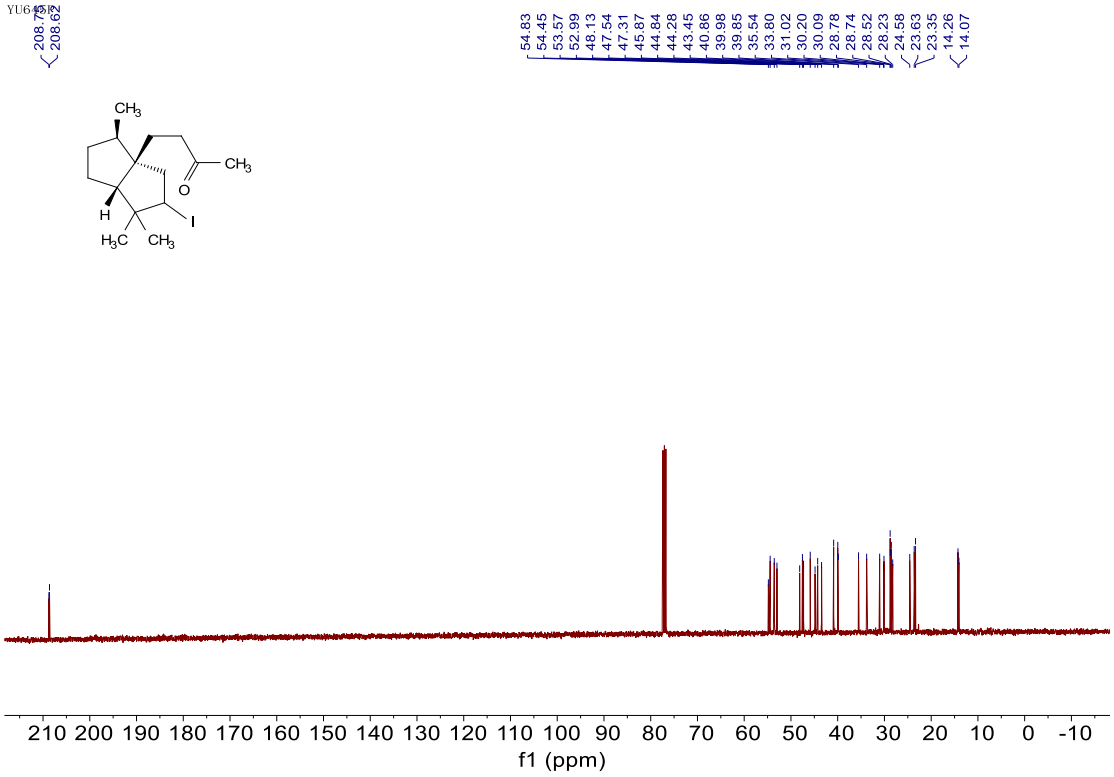
^{13}C NMR of compound **27**



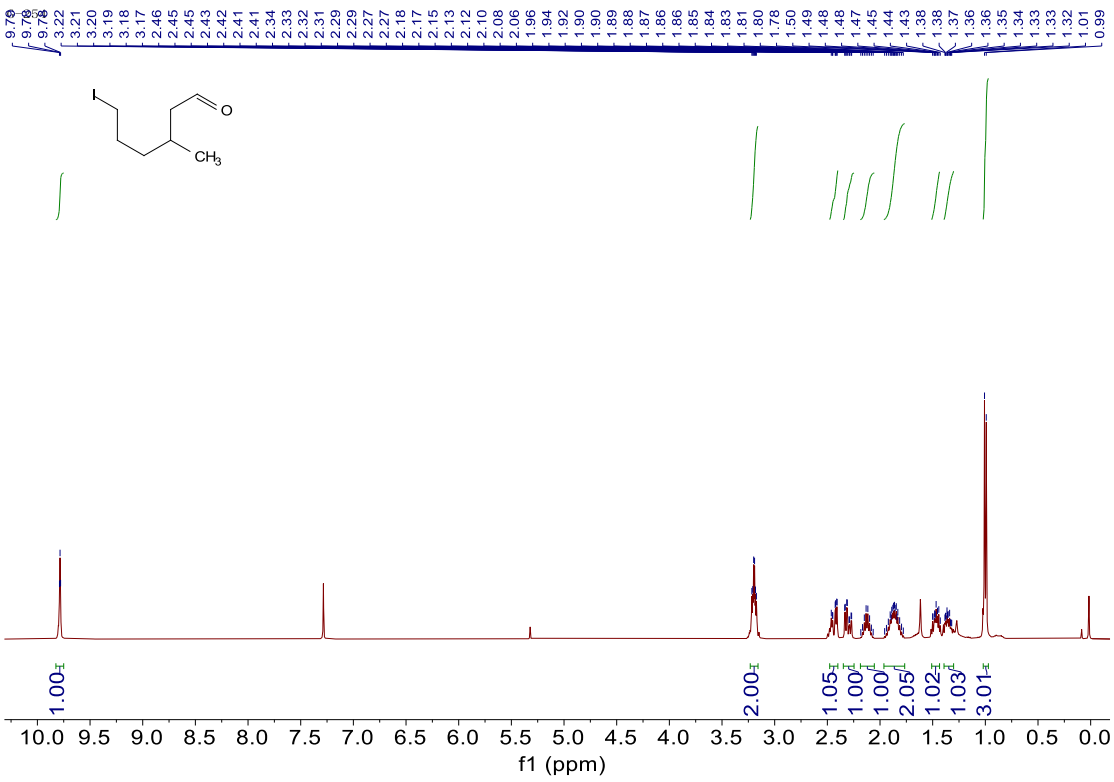
^{1}H NMR of compound **28**



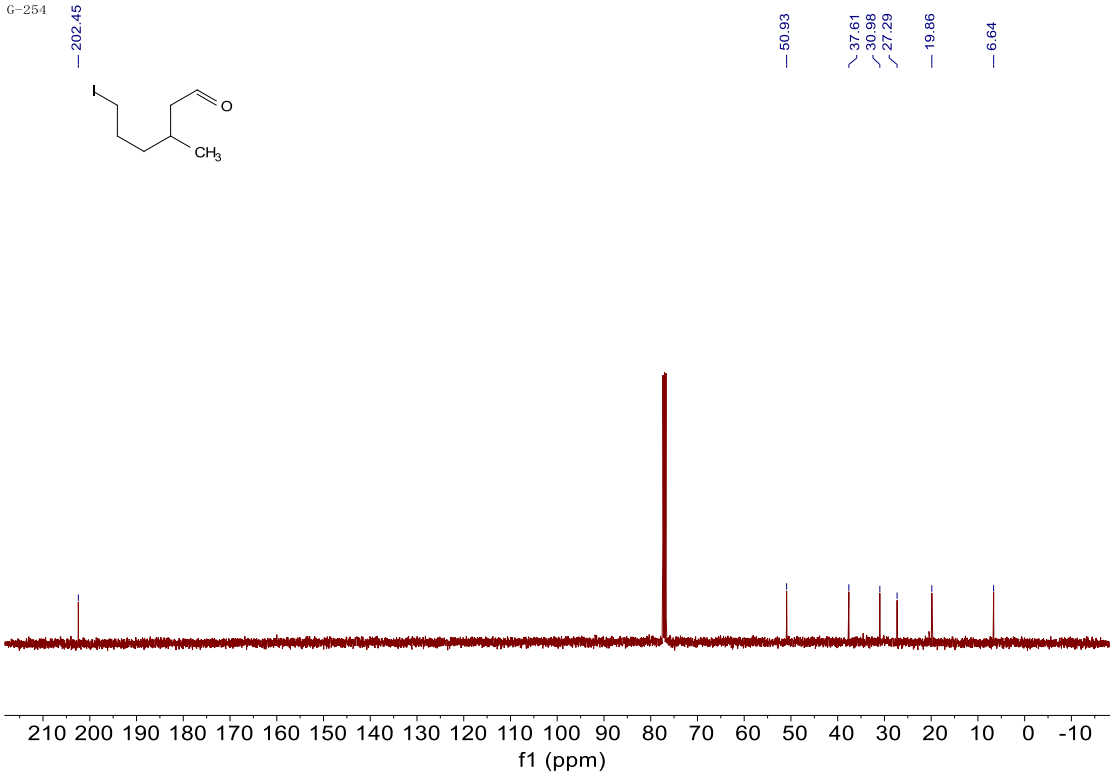
^{13}C NMR of compound **28**



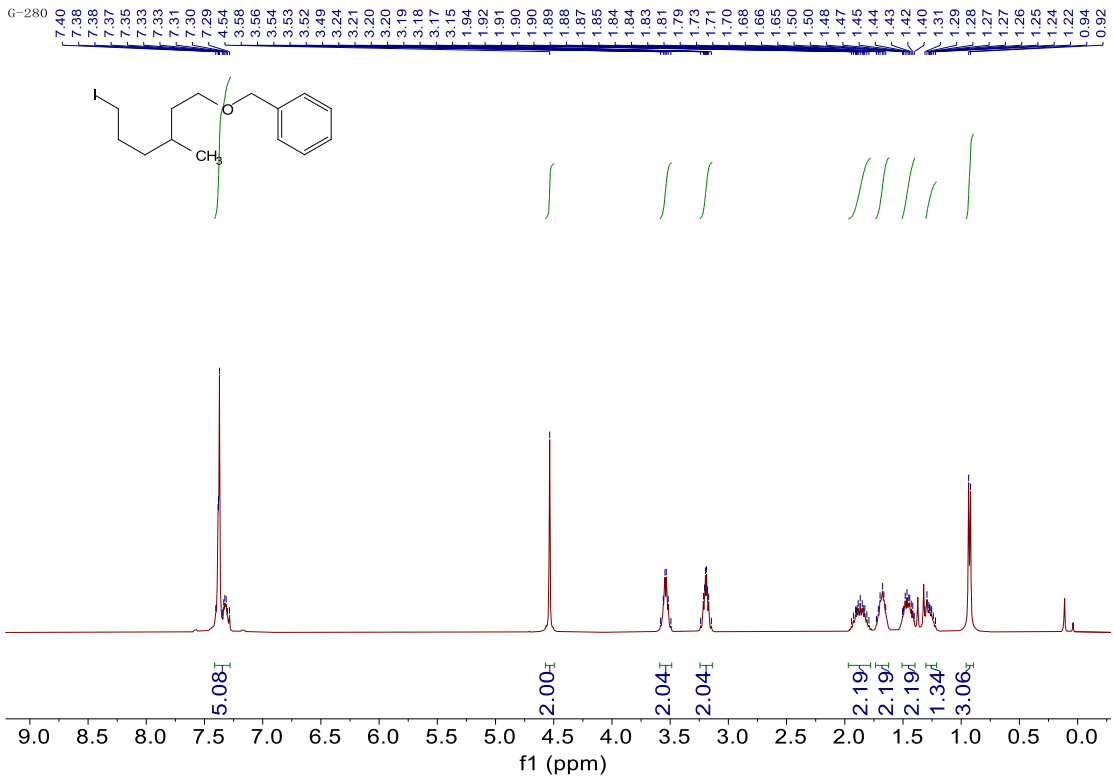
^{1}H NMR of compound **29**



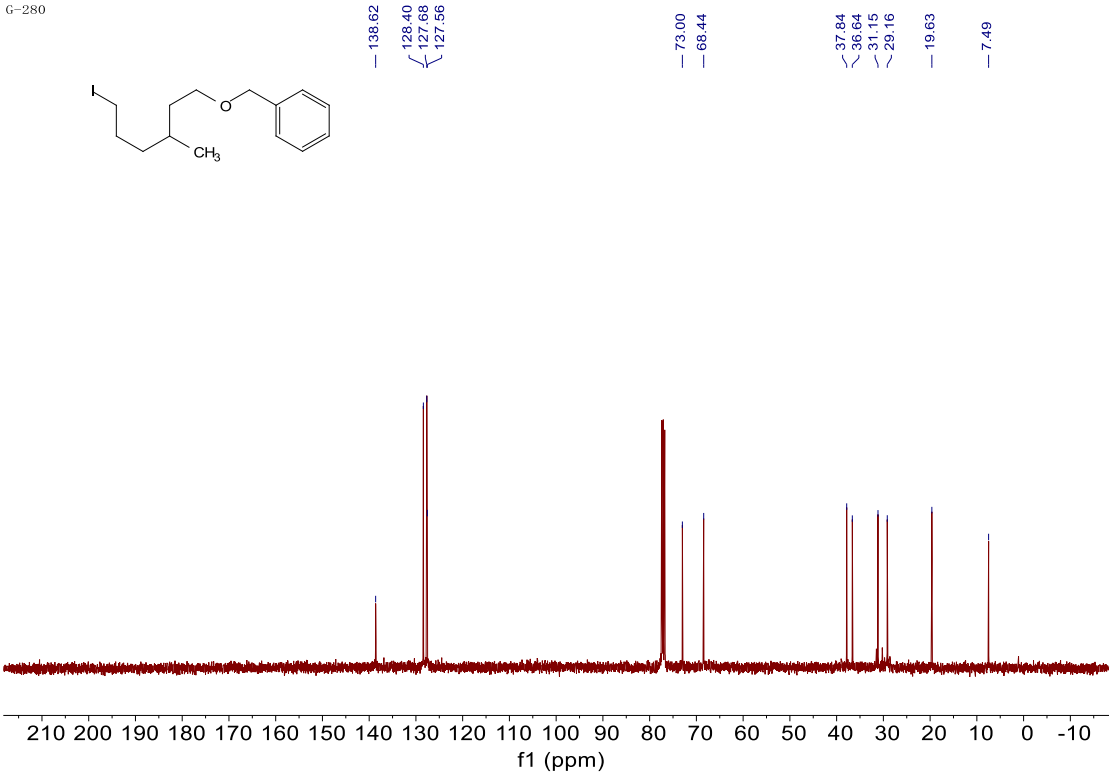
^{13}C NMR of compound **29**



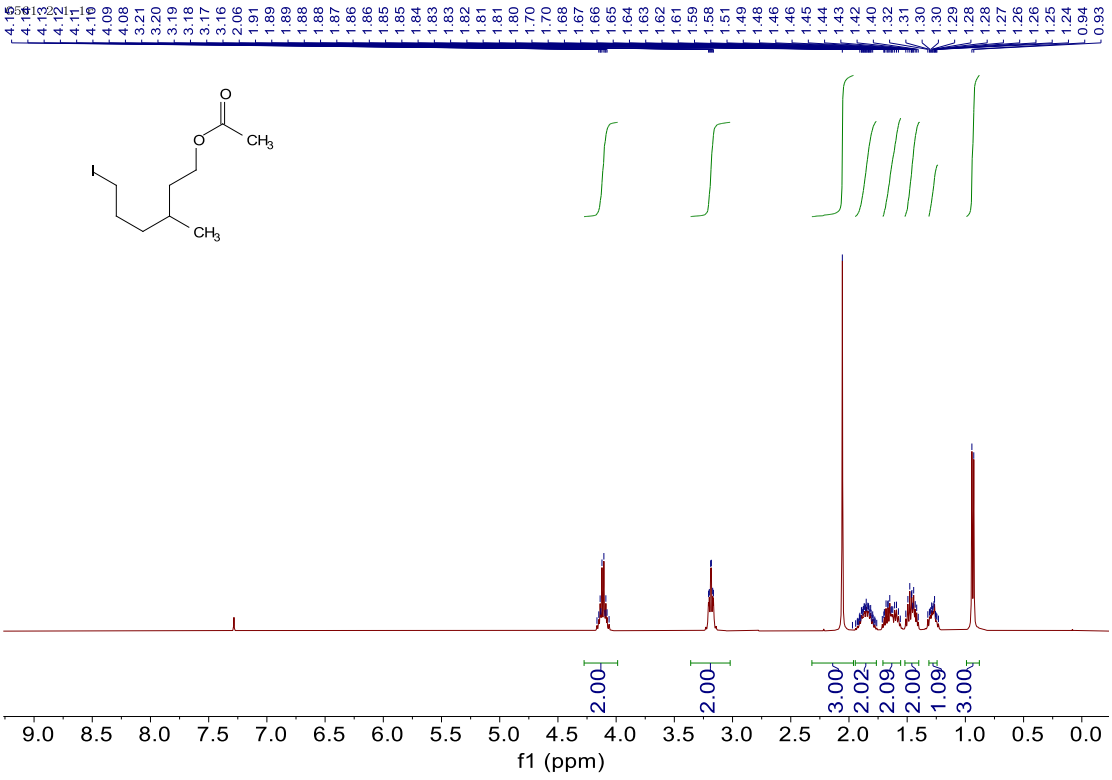
^{1}H NMR of compound **30**



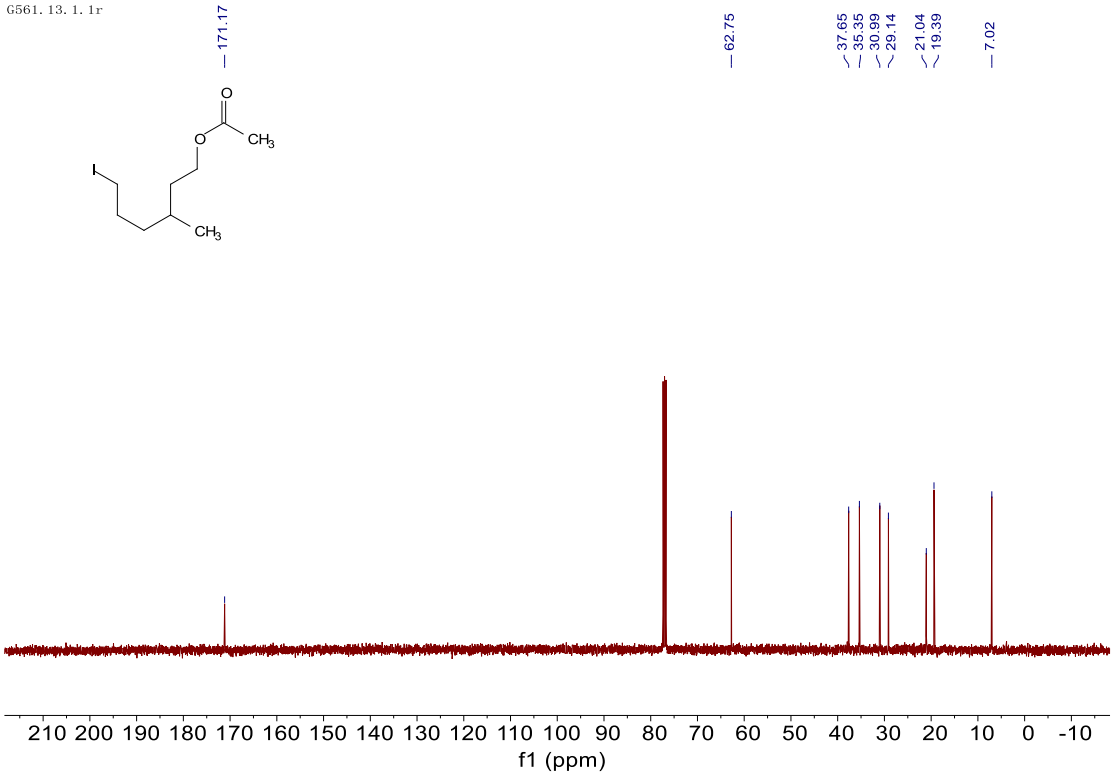
^{13}C NMR of compound **30**



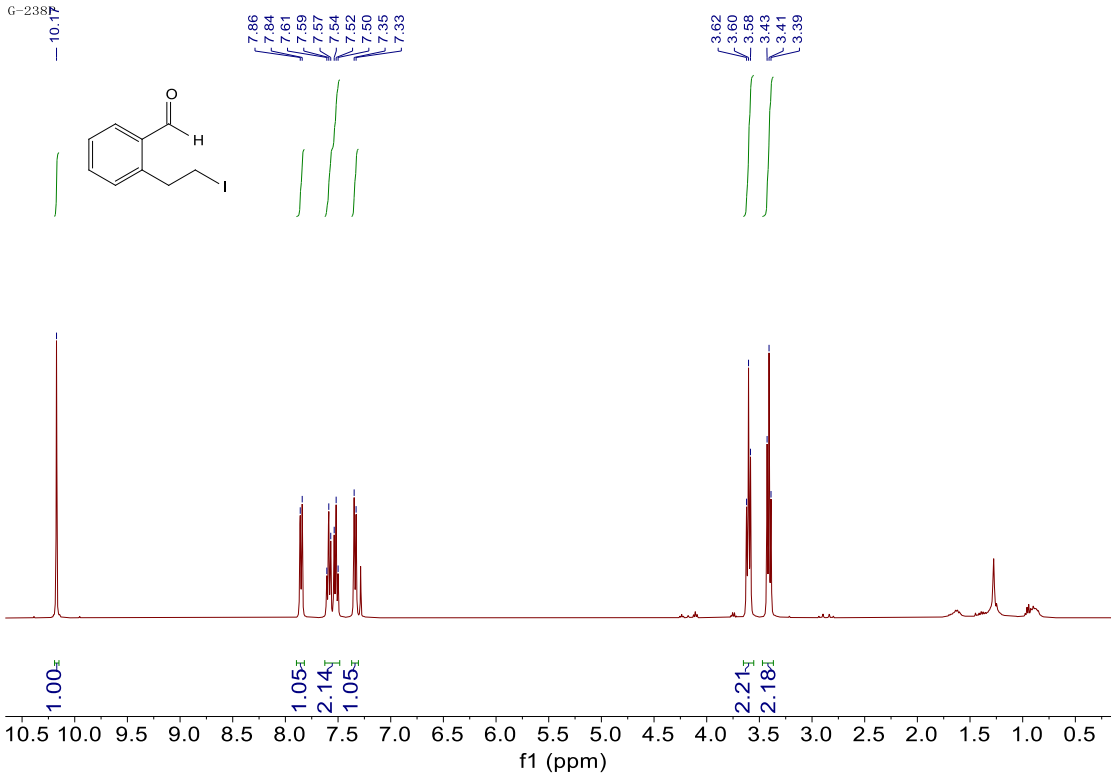
^{1}H NMR of compound **31**



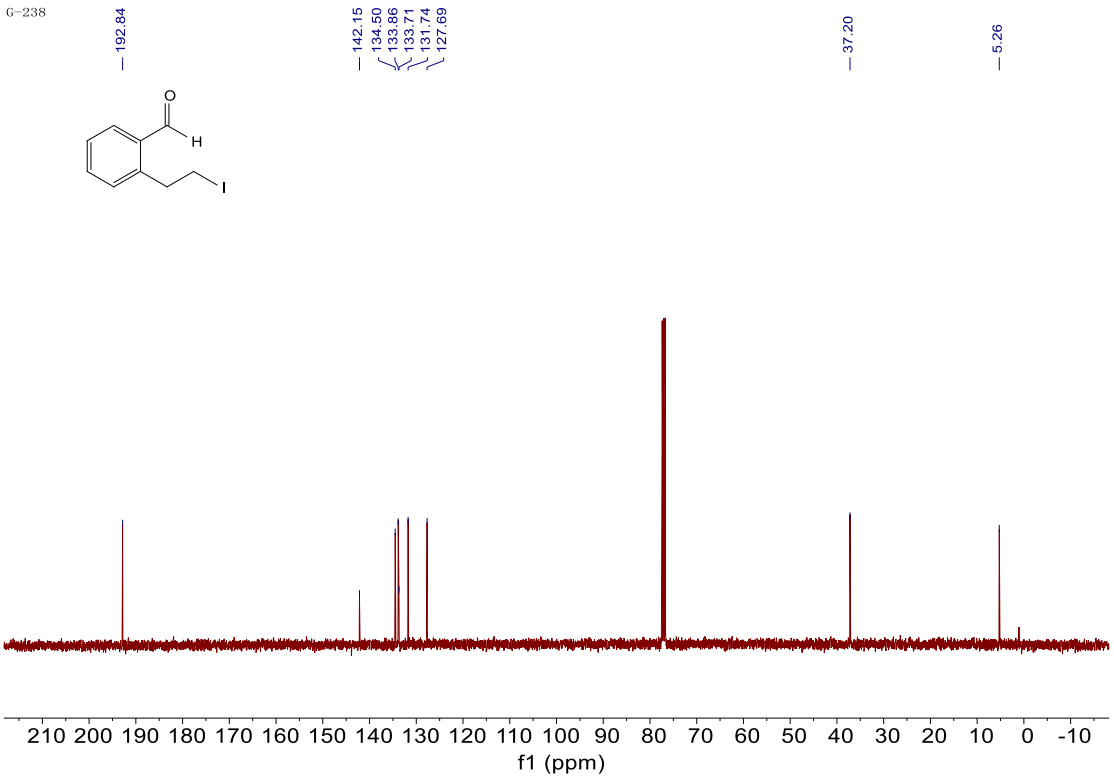
^{13}C NMR of compound **31**



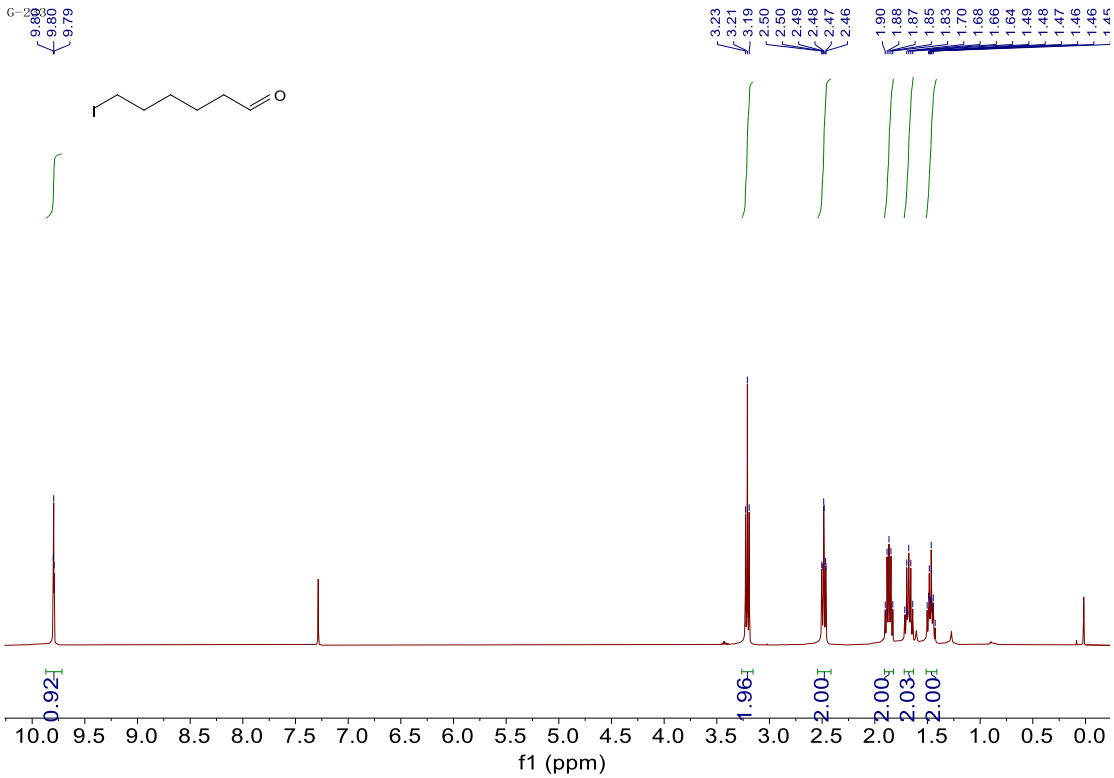
^{1}H NMR of compound **32**



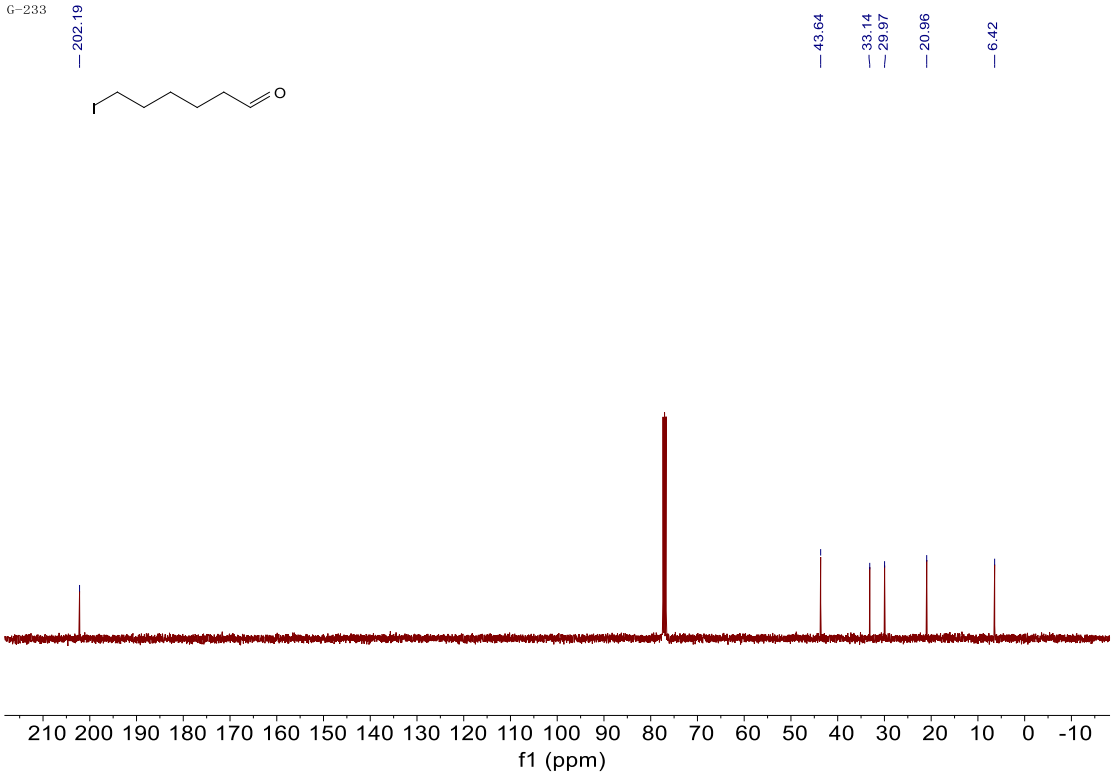
^{13}C NMR of compound **32**



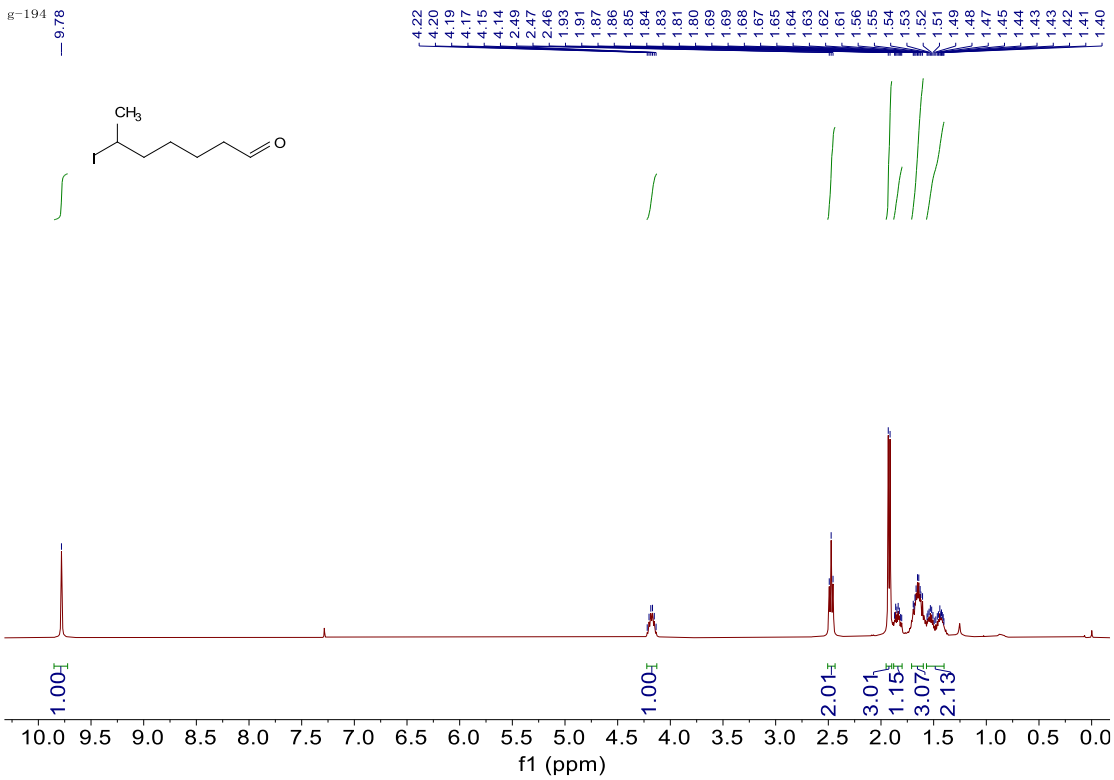
^{1}H NMR of compound **33**



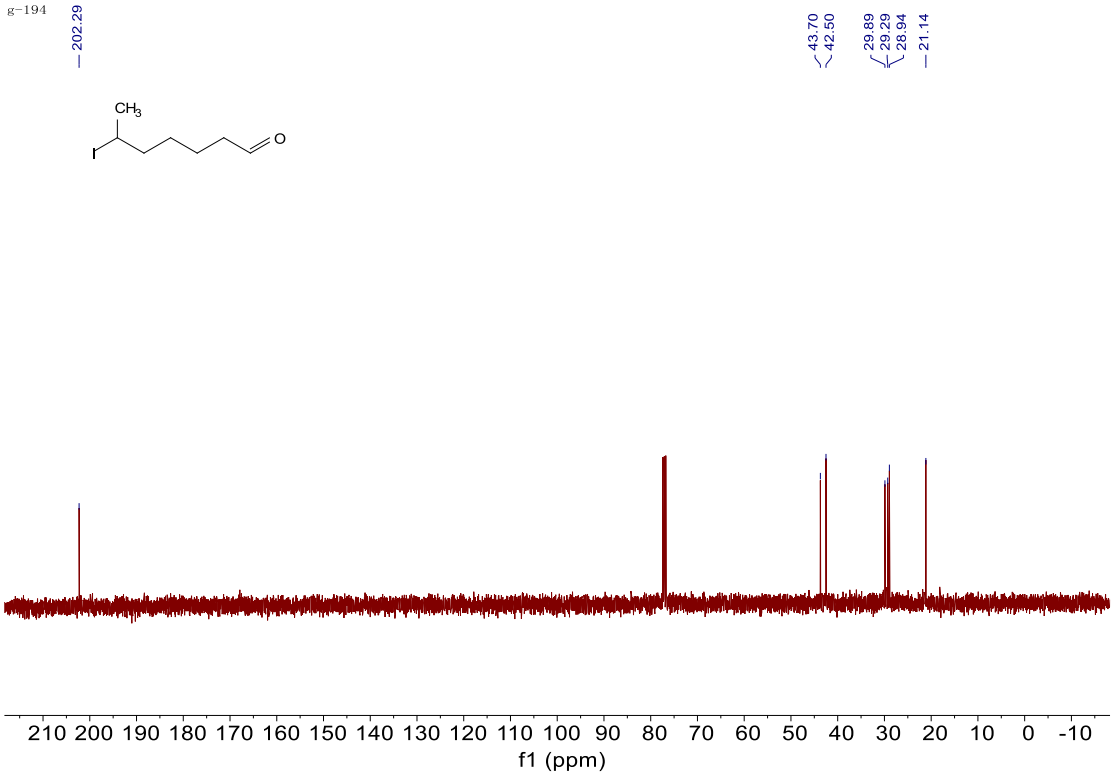
^{13}C NMR of compound **33**



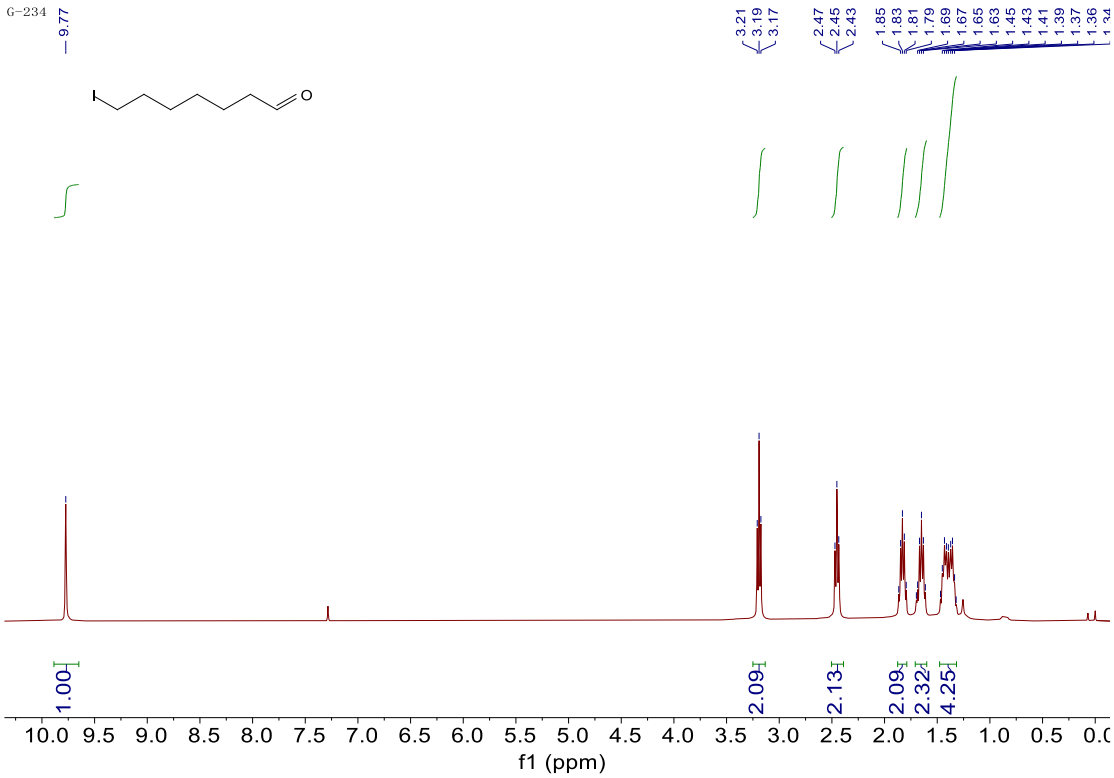
^{1}H NMR of compound **34**



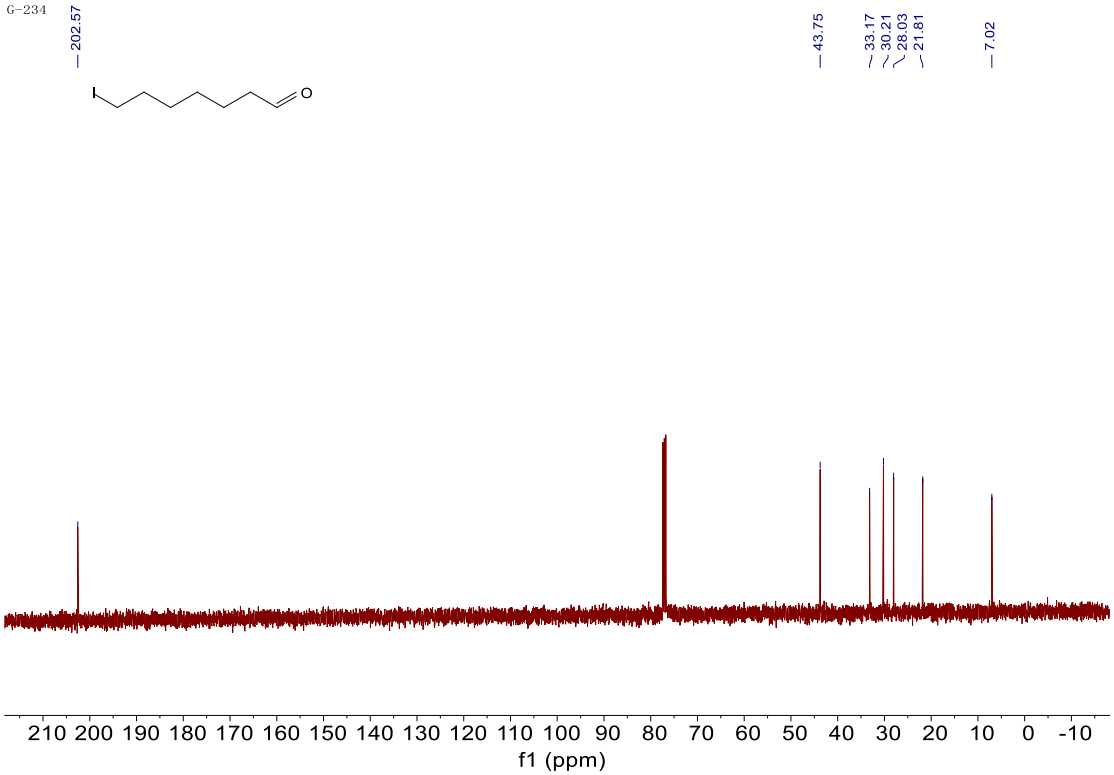
^{13}C NMR of compound **34**



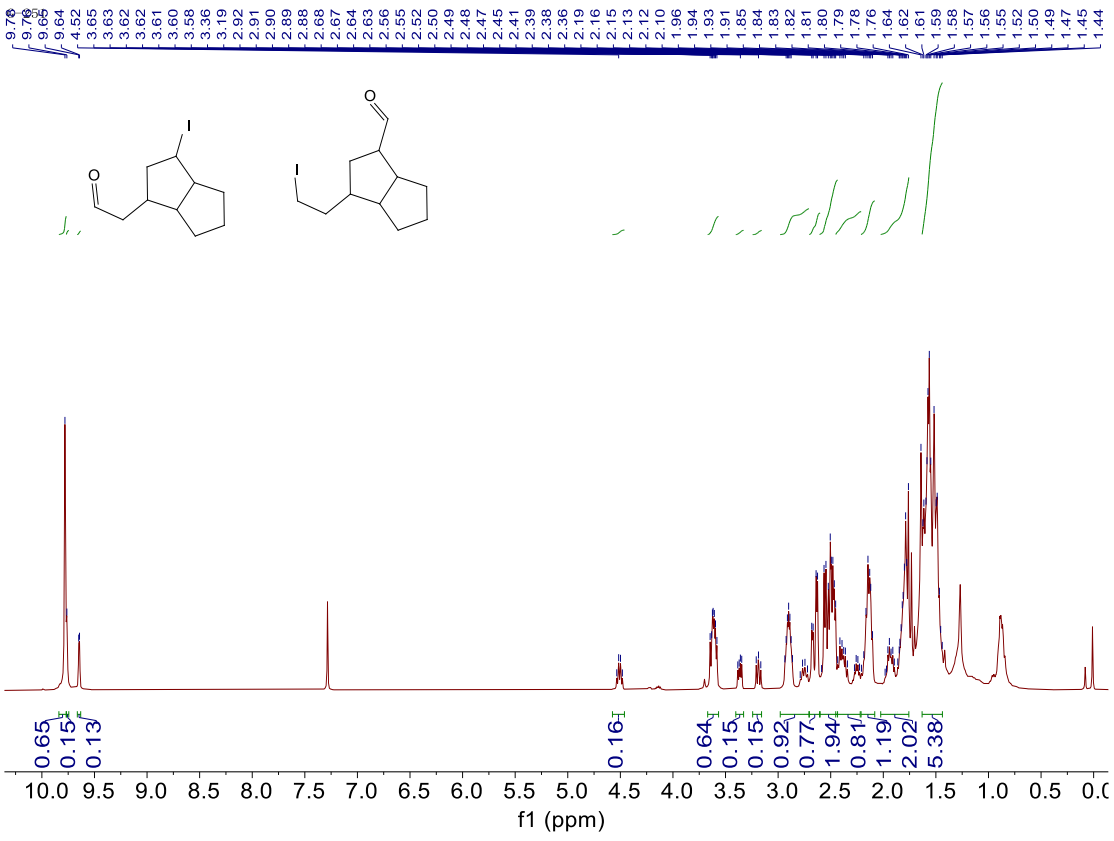
^{1}H NMR of compound **35**



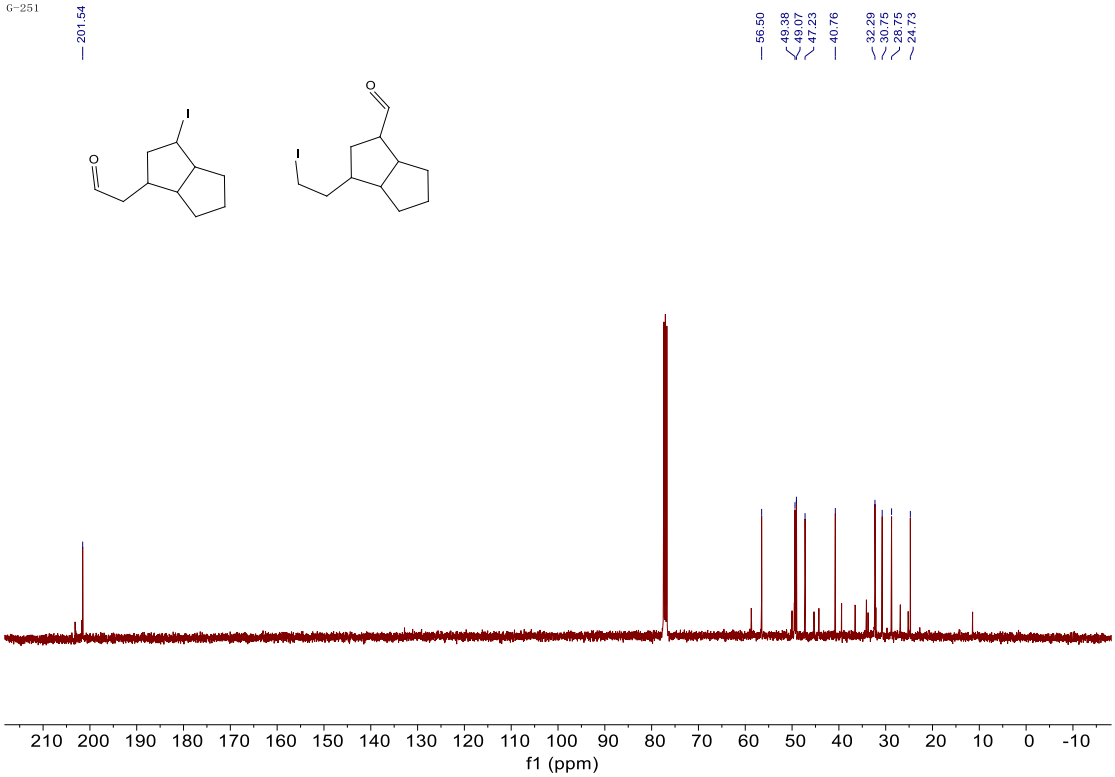
^{13}C NMR of compound **35**



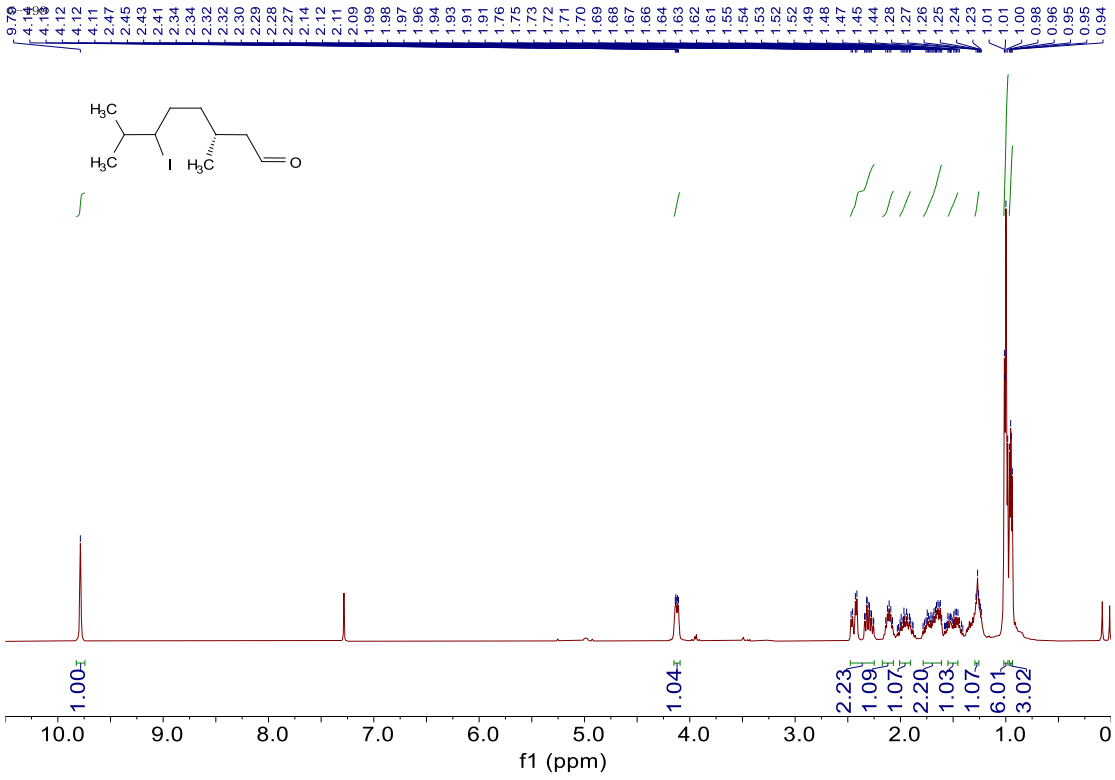
^{1}H NMR of compound **36**



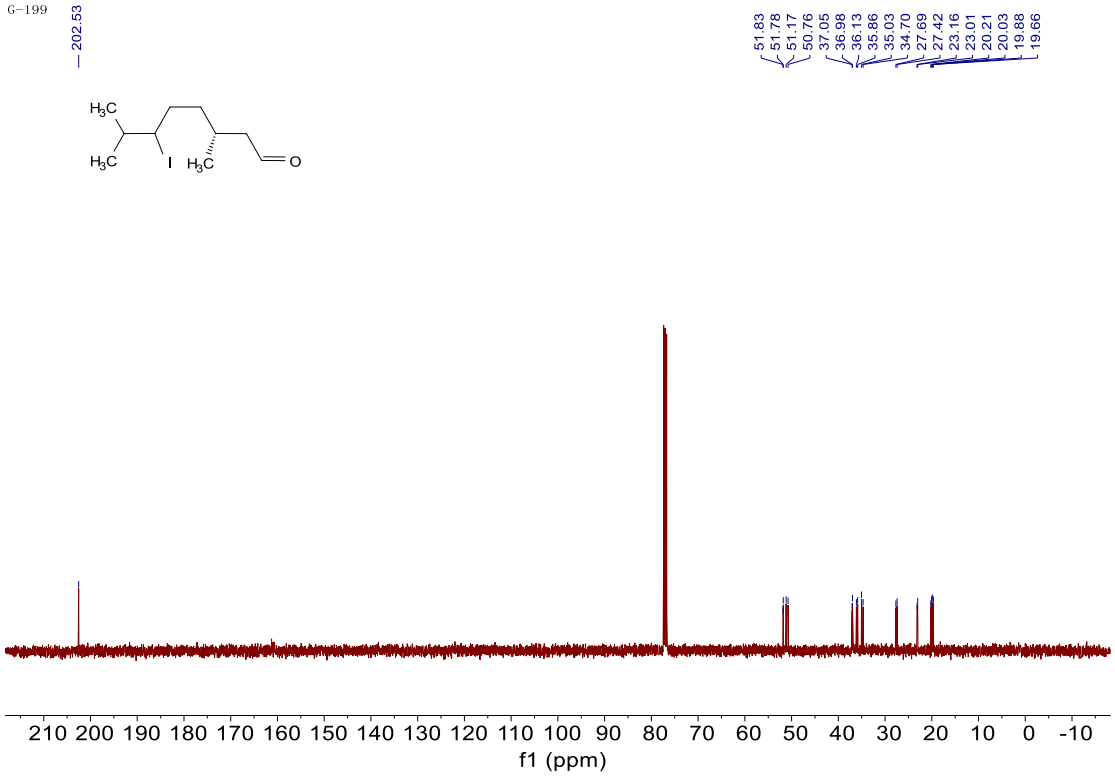
^{13}C NMR of compound **36**



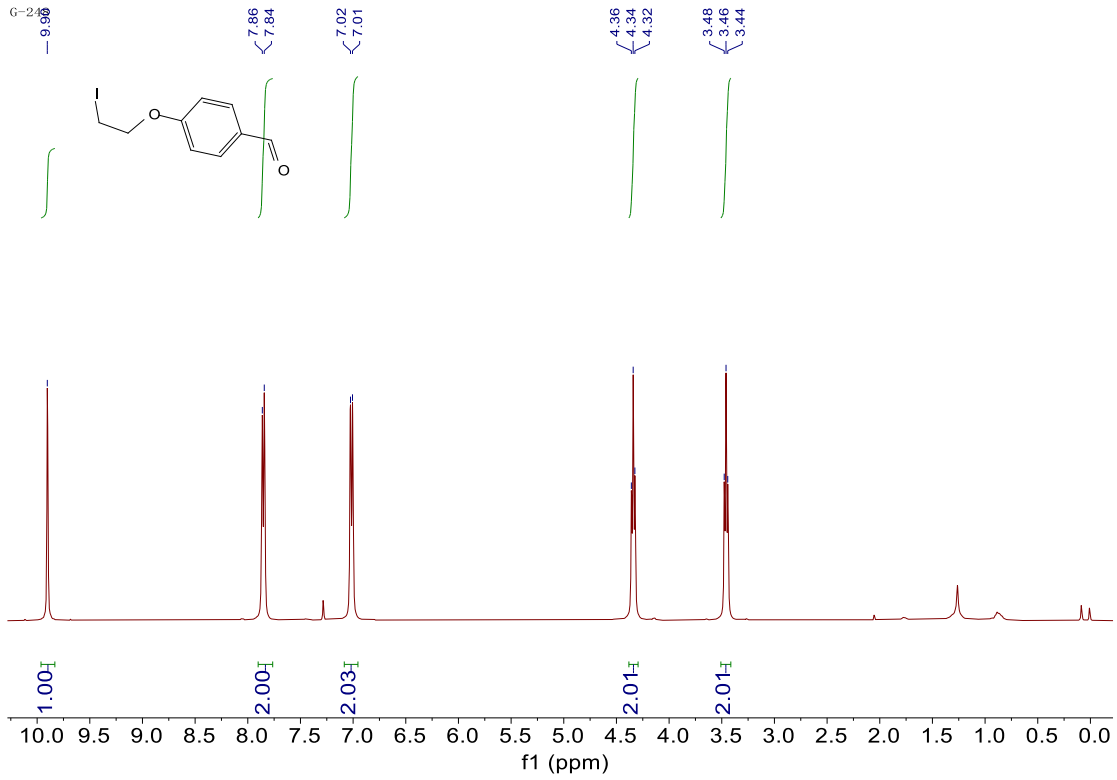
^{1}H NMR of compound **37**



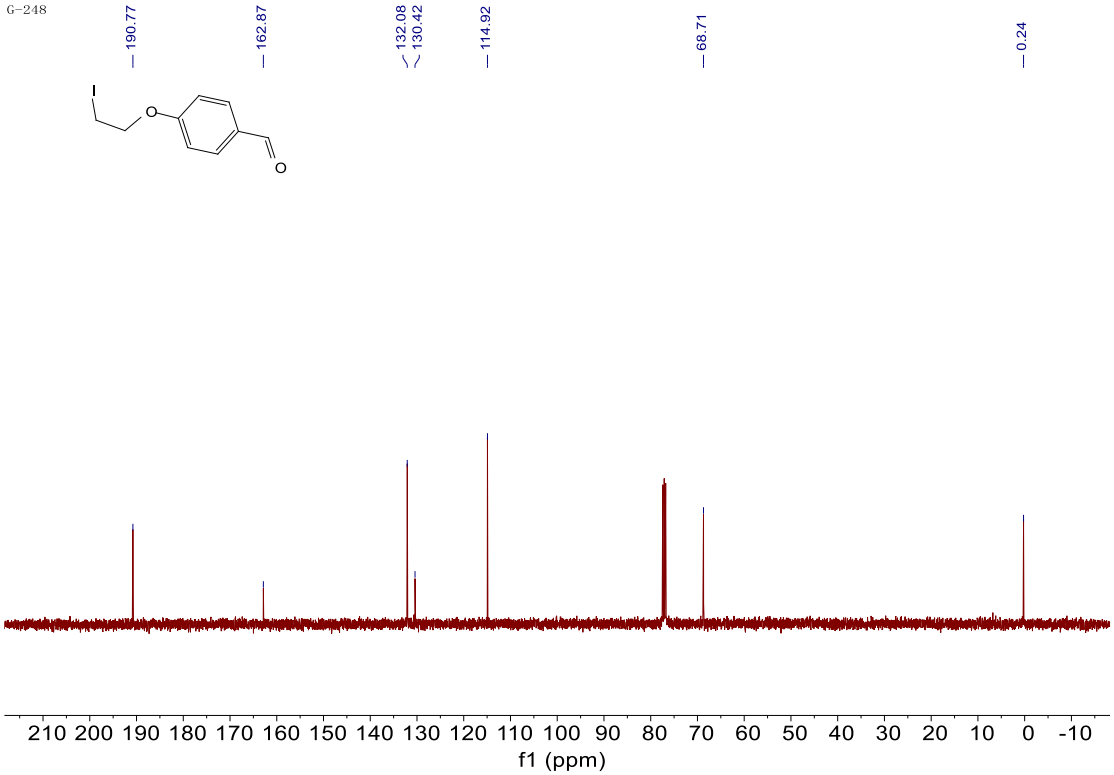
^{13}C NMR of compound **37**



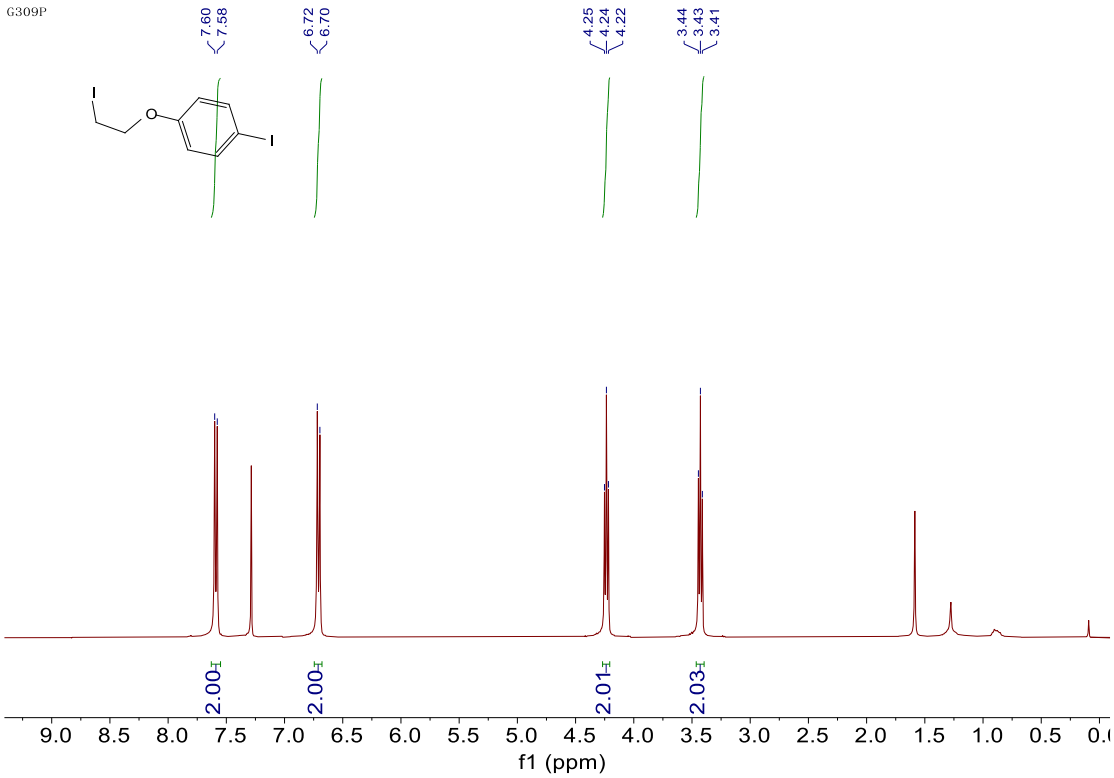
^{1}H NMR of compound **38**



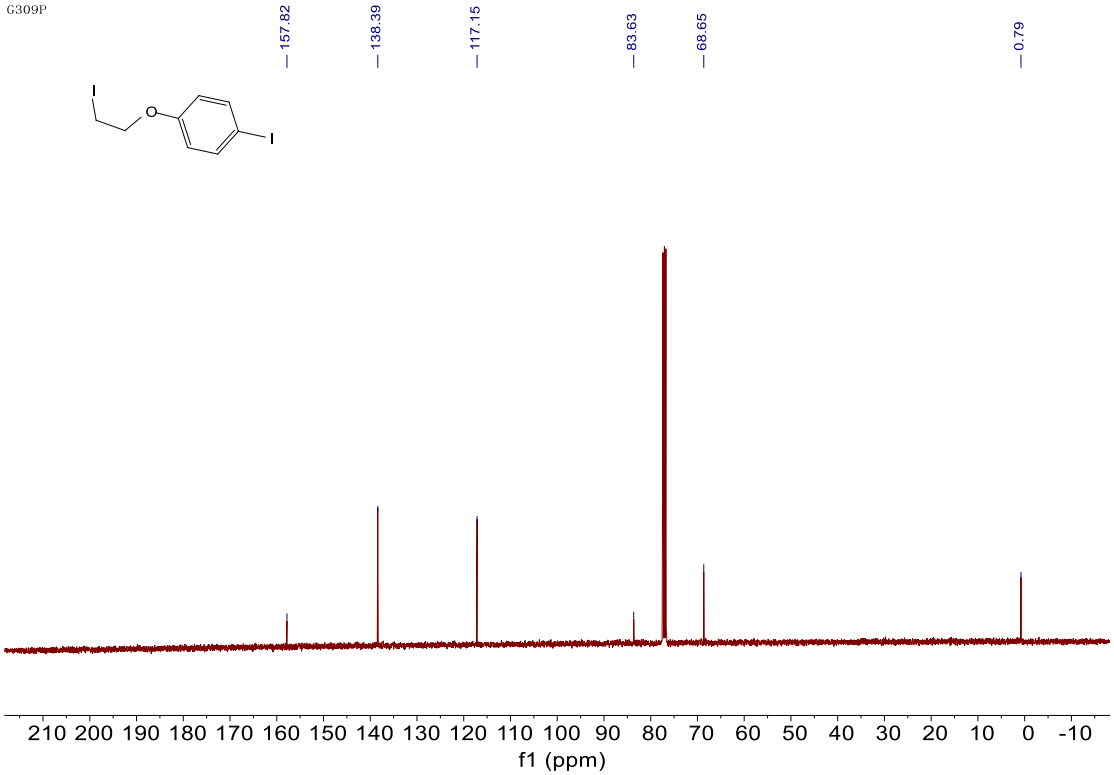
^{13}C NMR of compound **38**



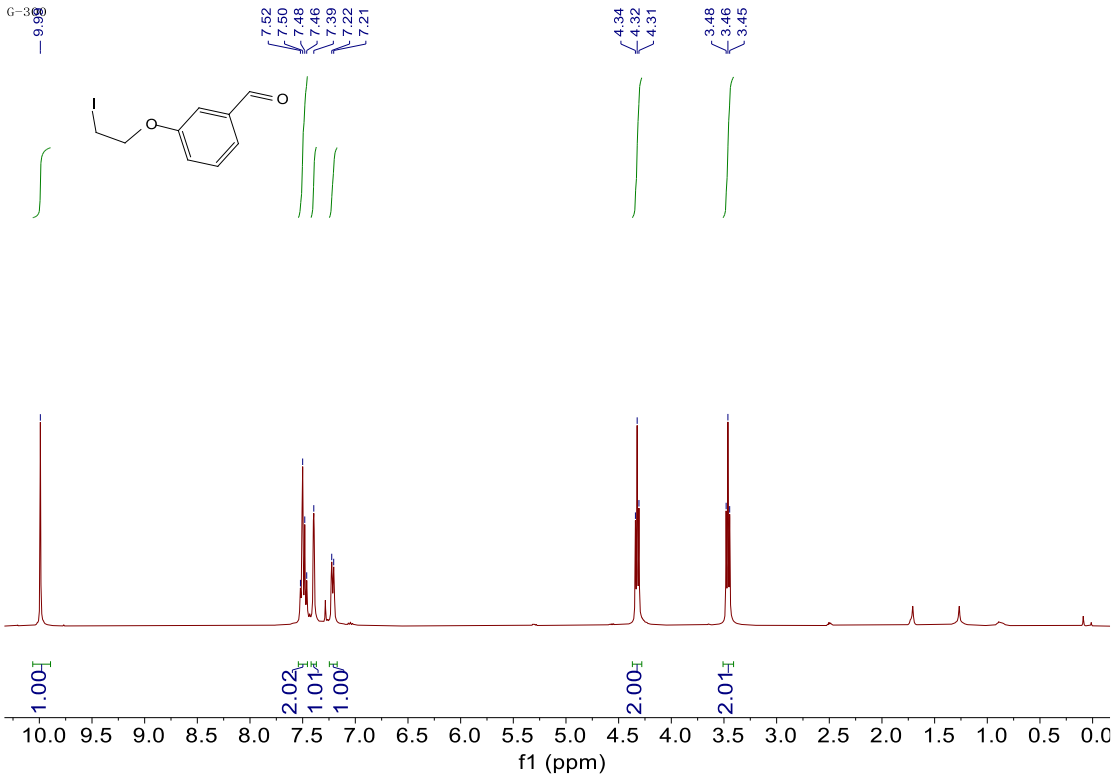
^{1}H NMR of compound **39**



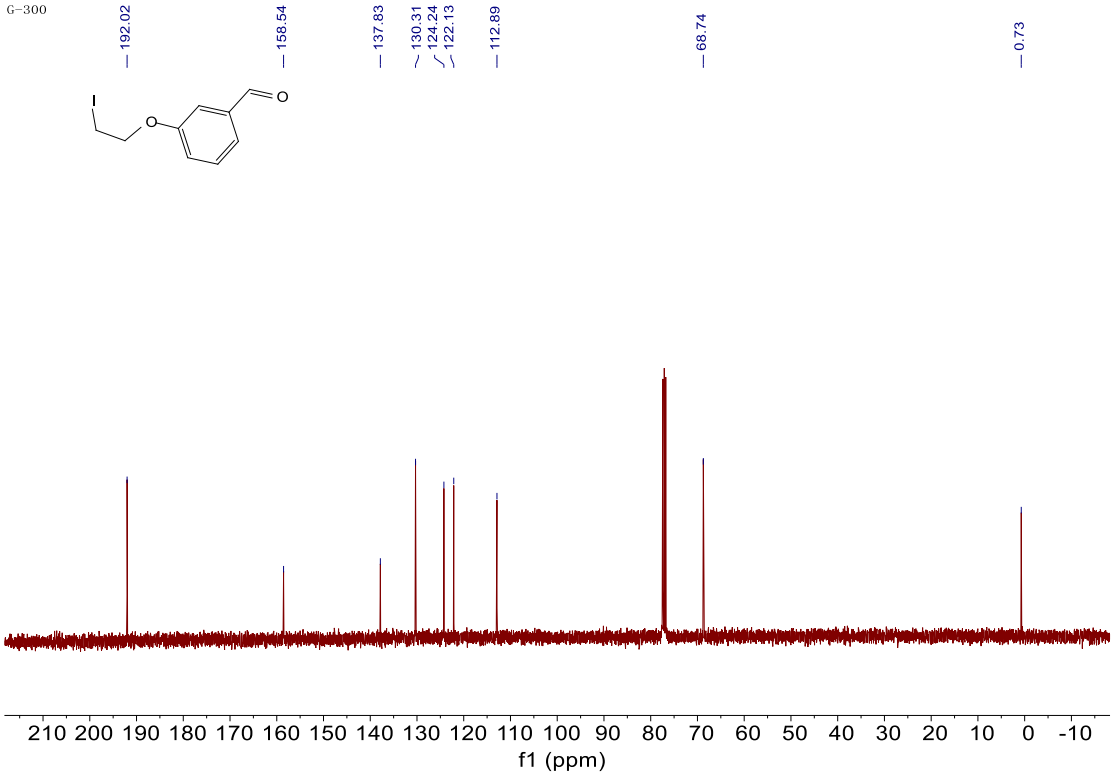
^{13}C NMR of compound **39**



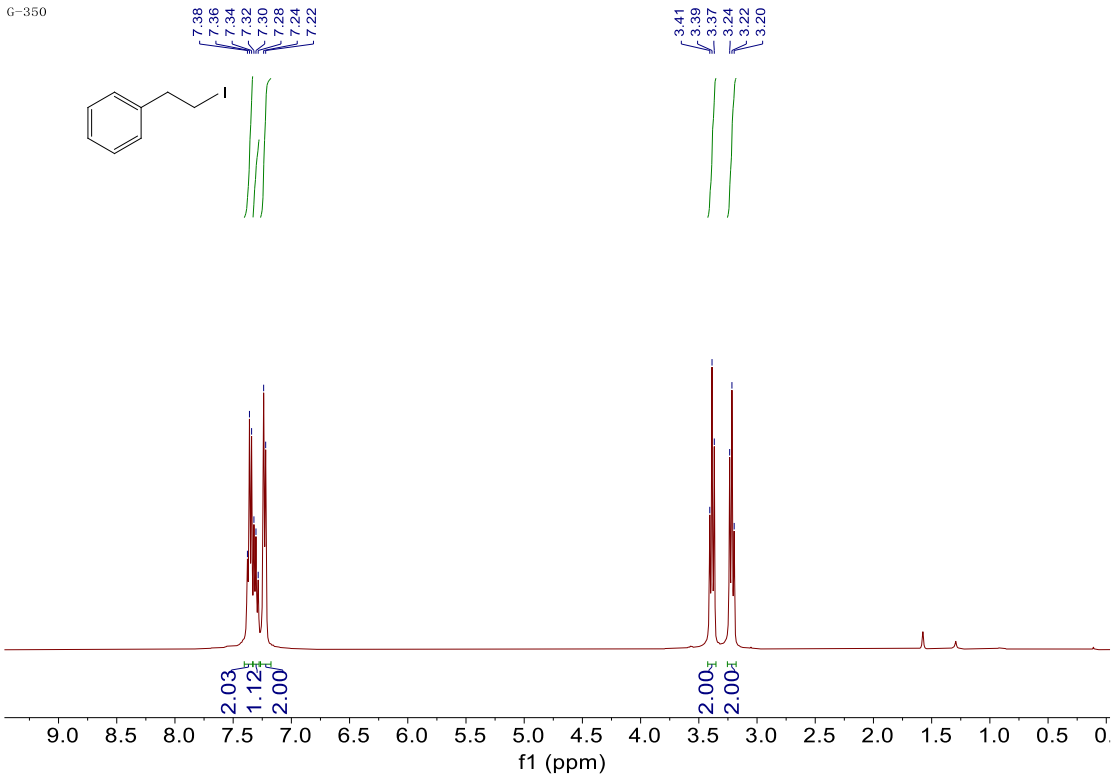
^{1}H NMR of compound **40**



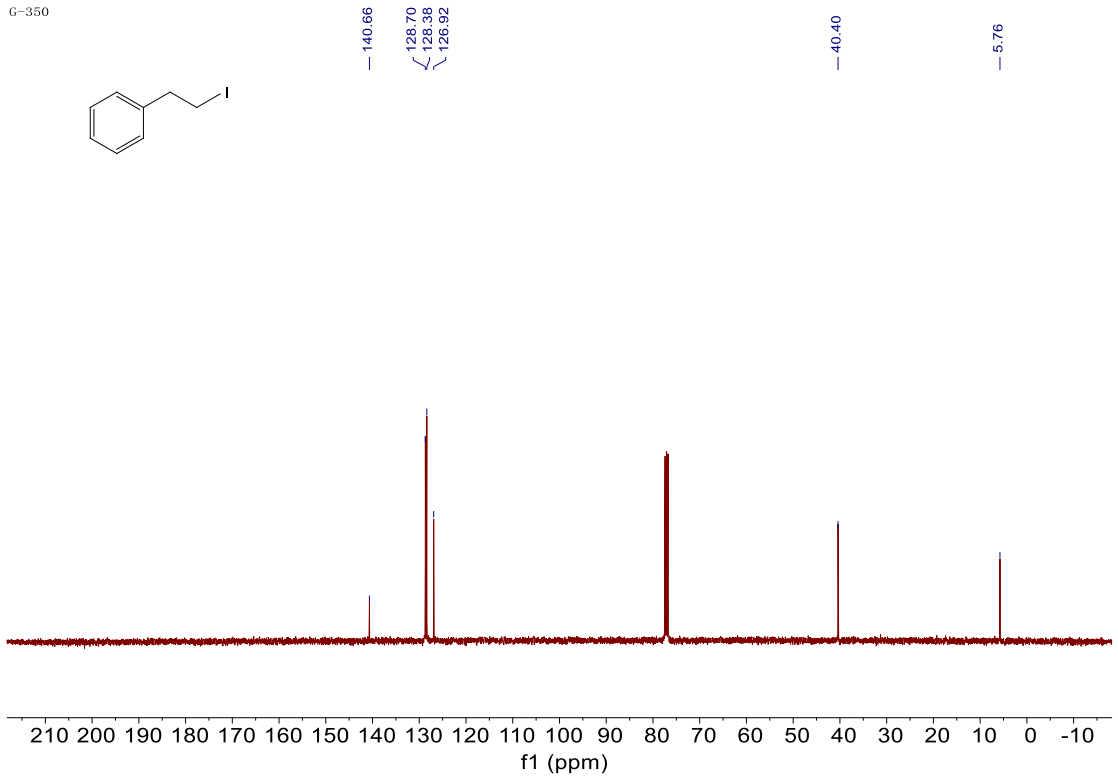
^{13}C NMR of compound **40**



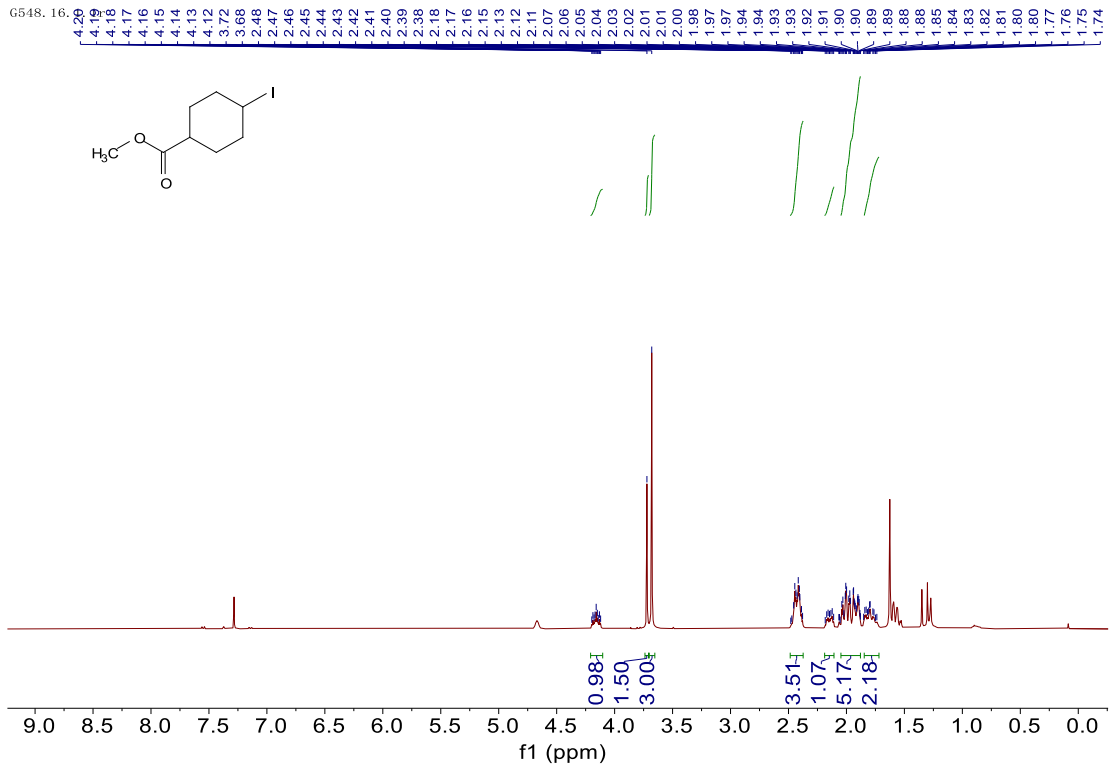
^{1}H NMR of compound **41**



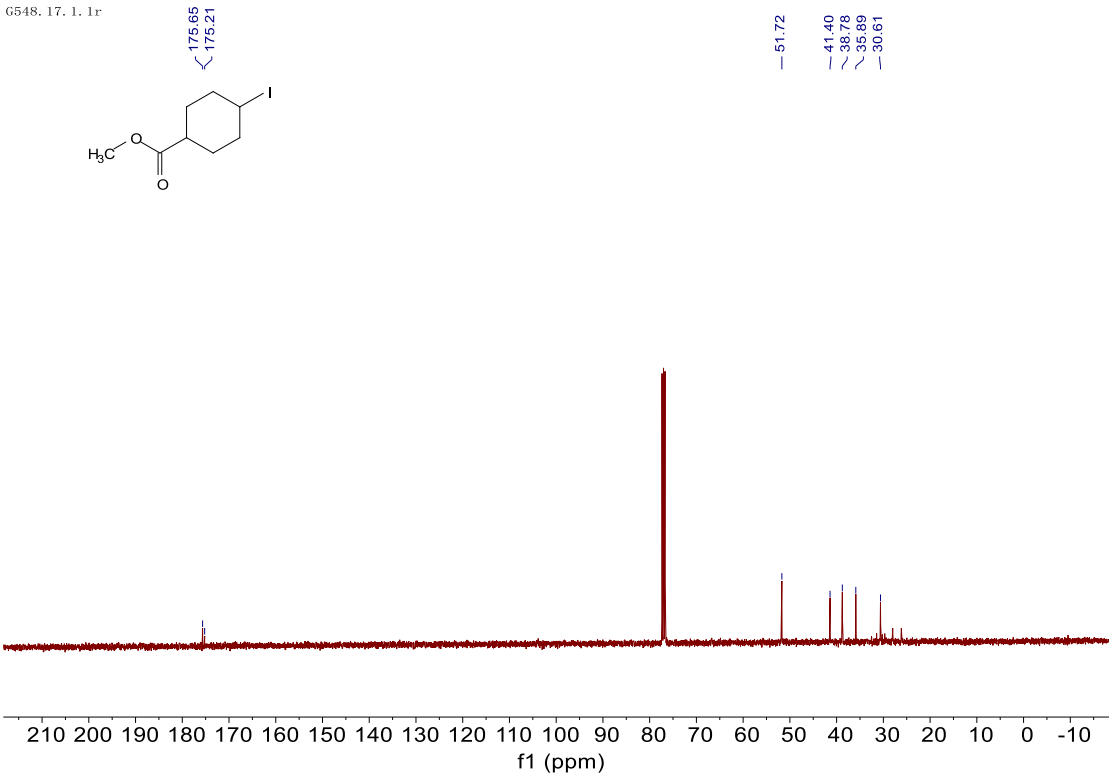
^{13}C NMR of compound **41**



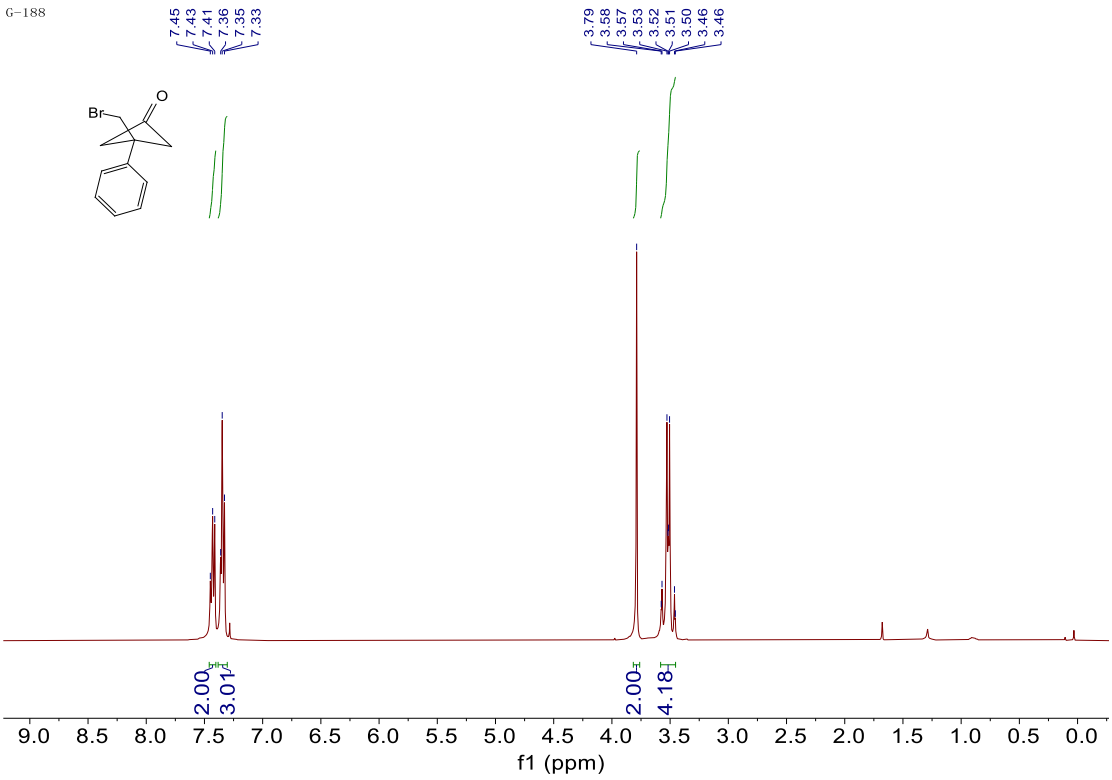
^{1}H NMR of compound **42**



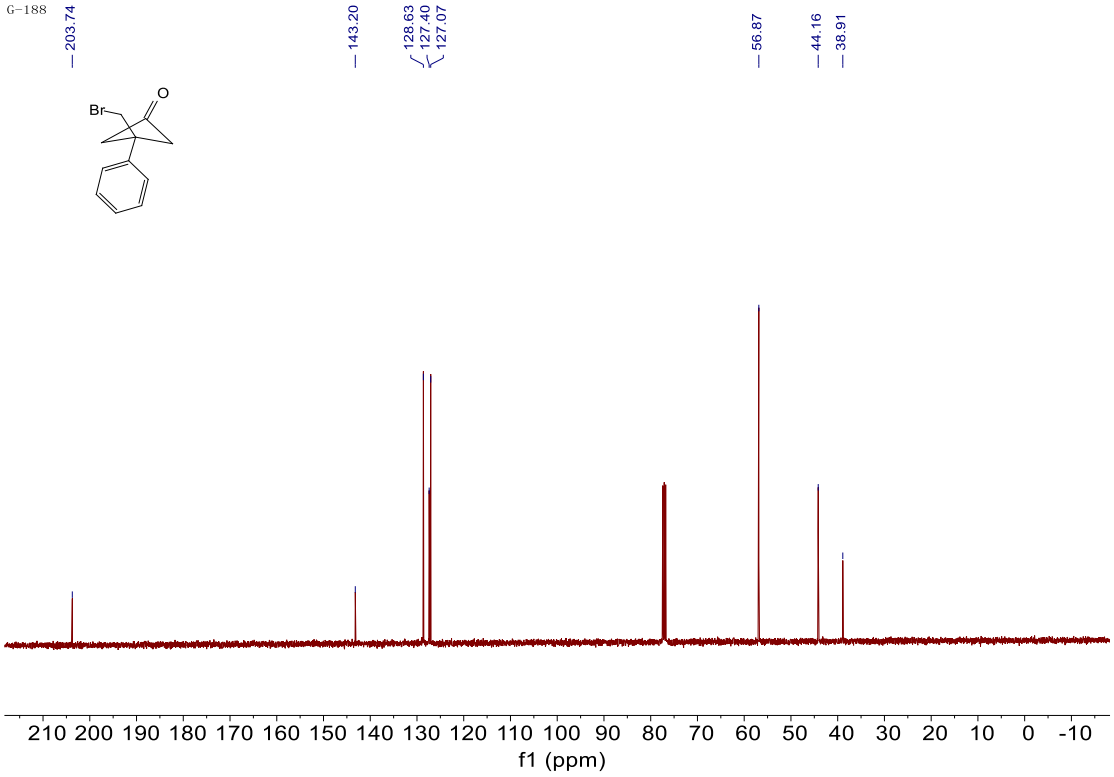
^{13}C NMR of compound **42**



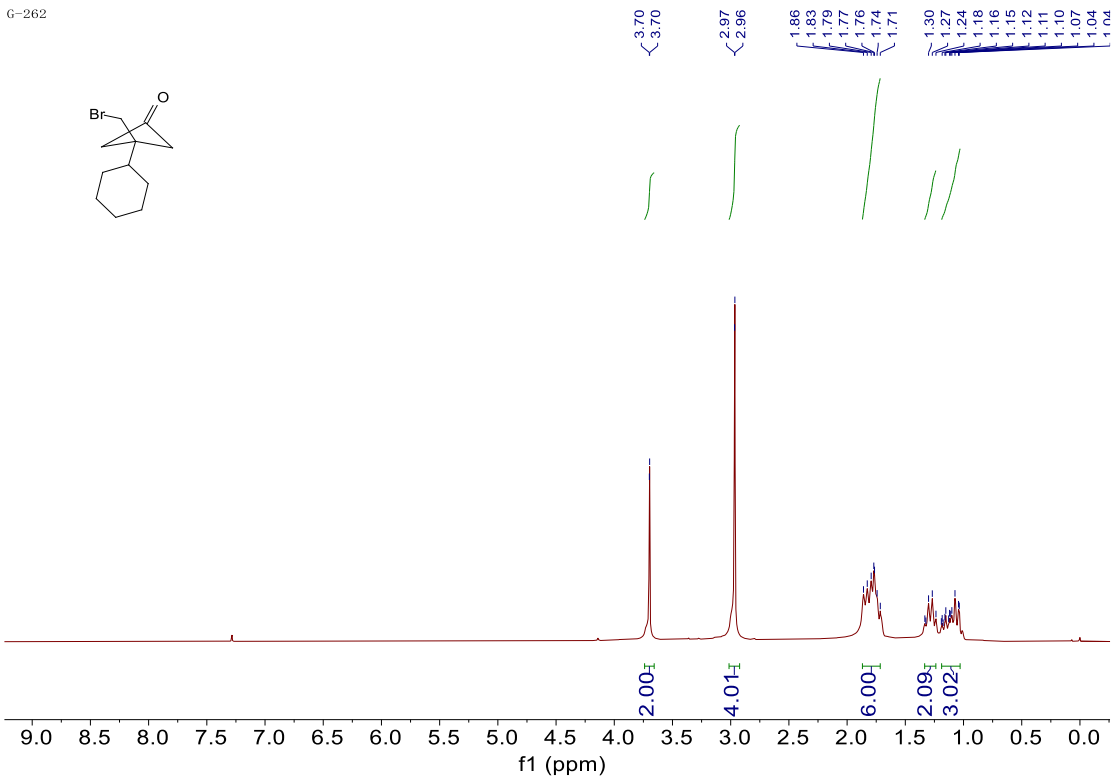
^{1}H NMR of compound **43**



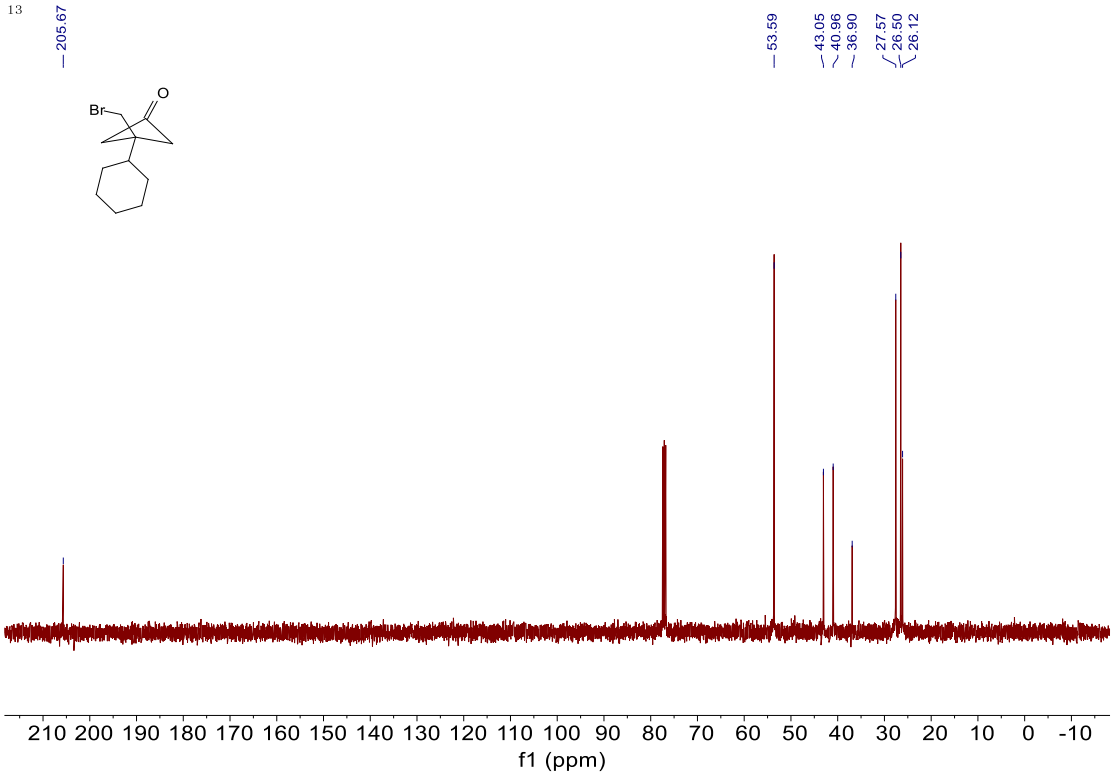
^{13}C NMR of compound **43**



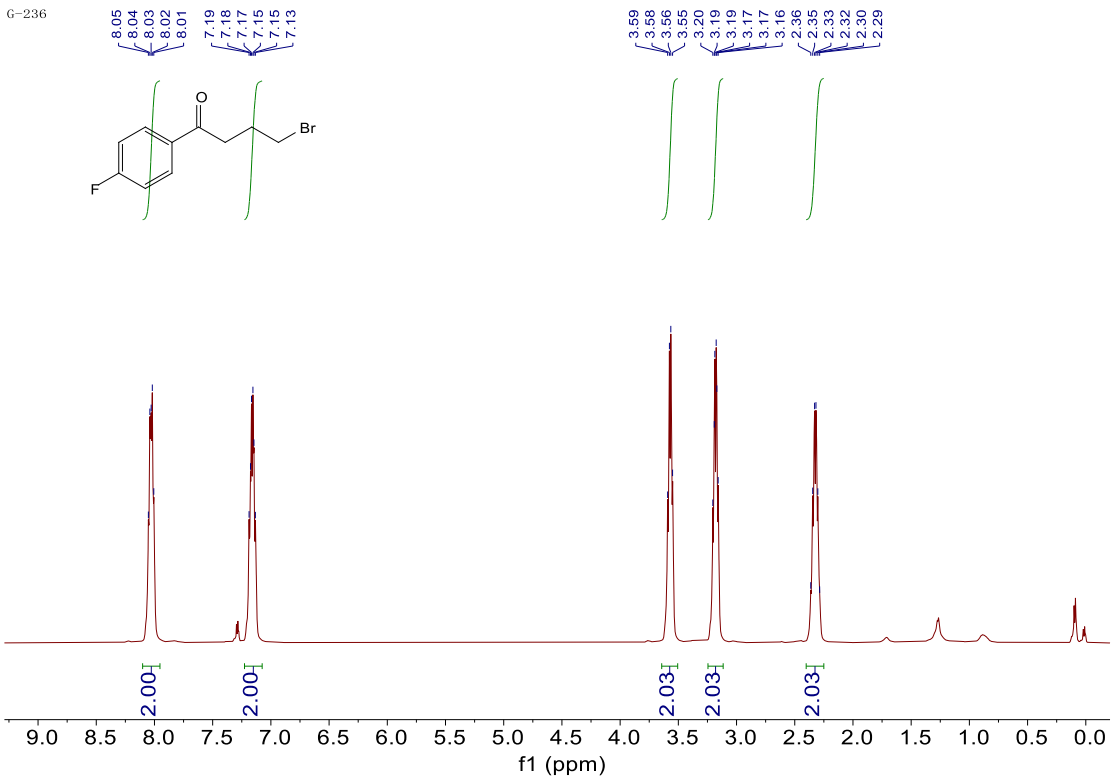
^{1}H NMR of compound **44**



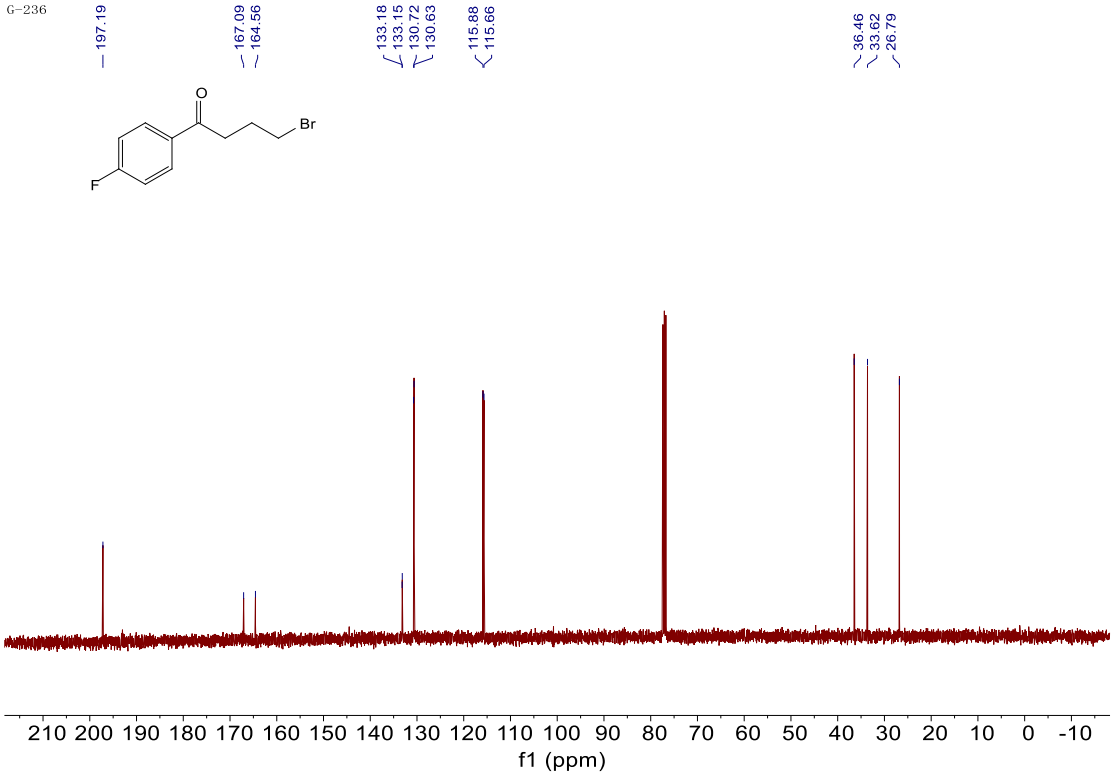
^{13}C NMR of compound **44**



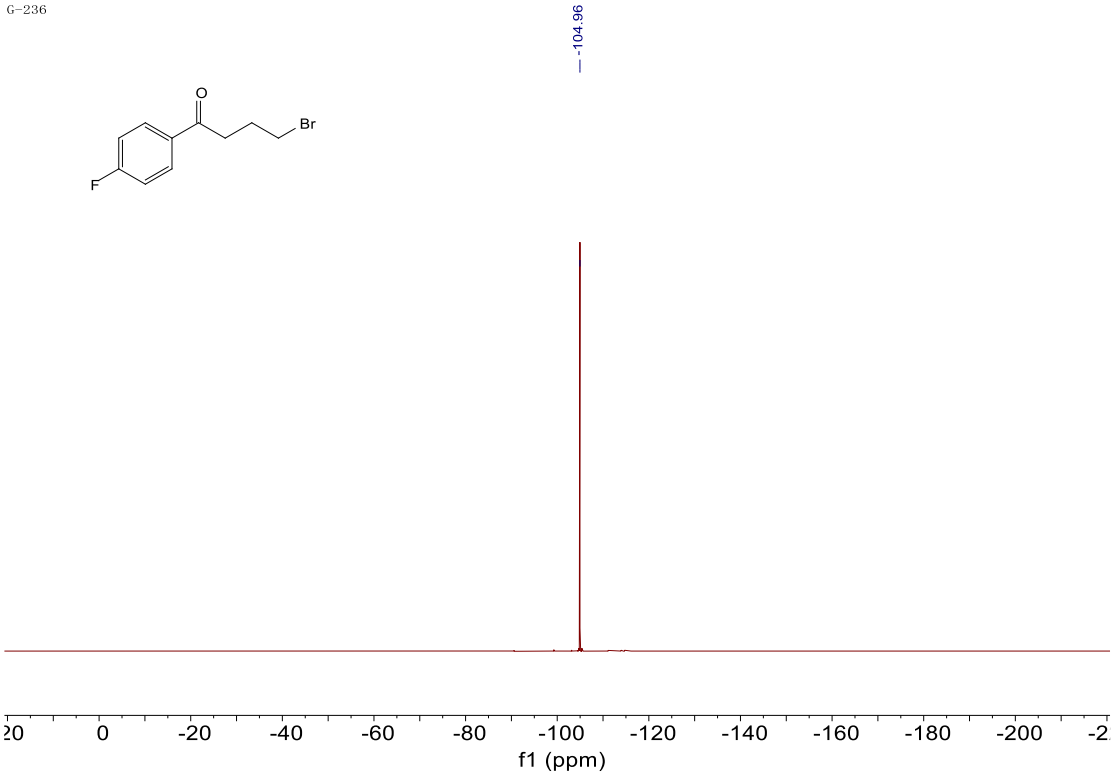
^{1}H NMR of compound **45**



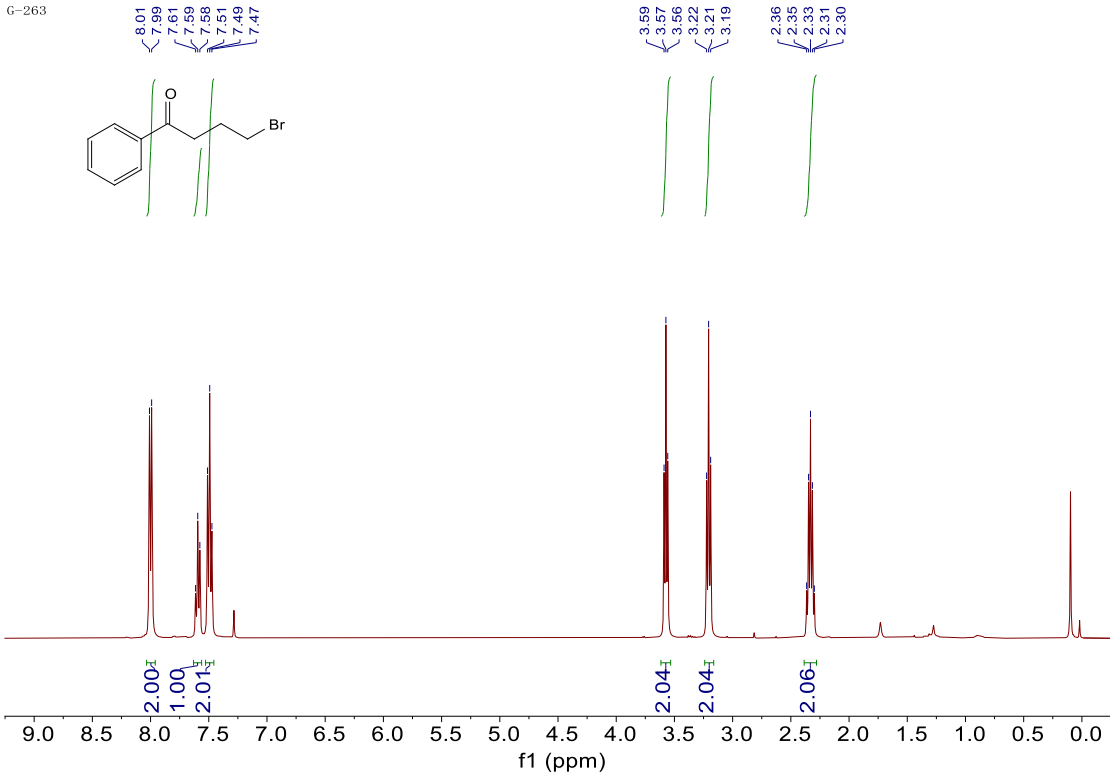
^{13}C NMR of compound **45**



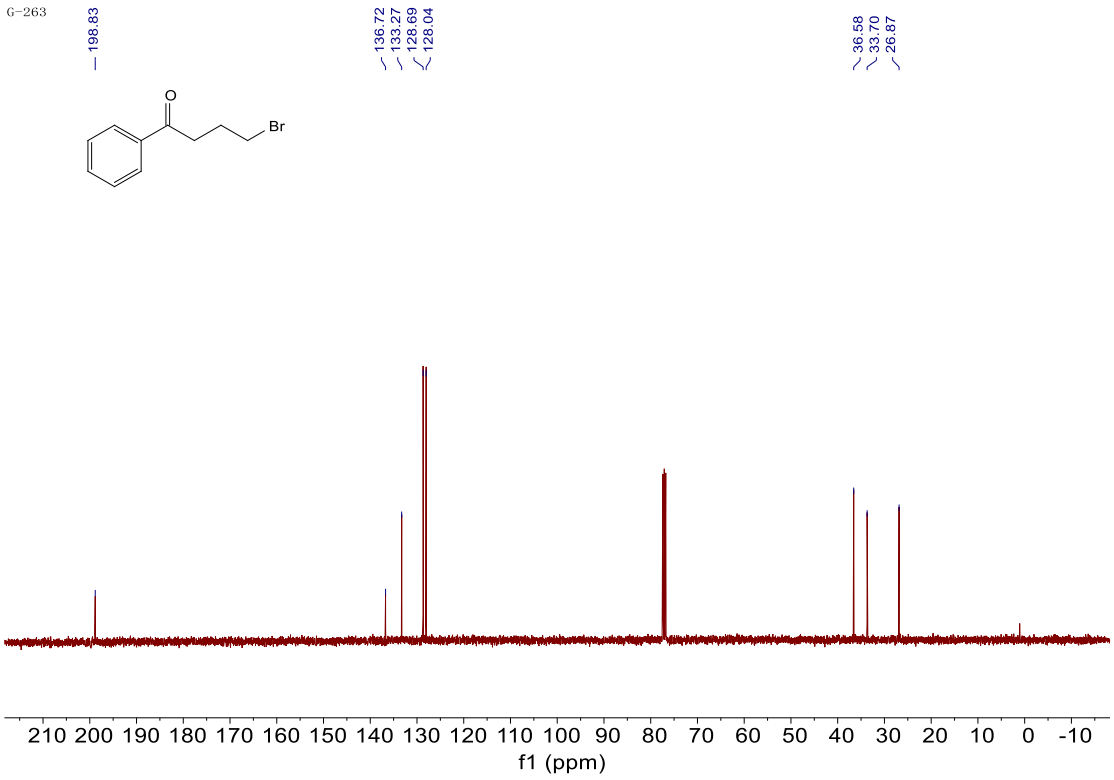
^{19}F NMR of compound **45**



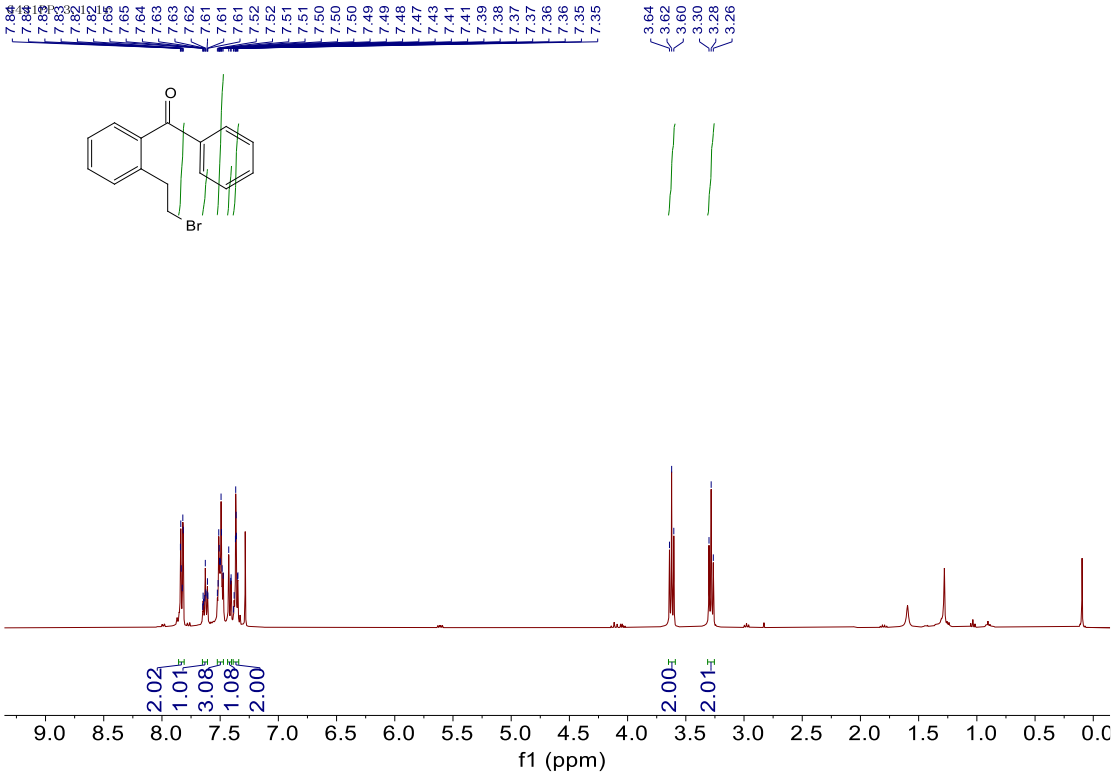
^{1}H NMR of compound **46**



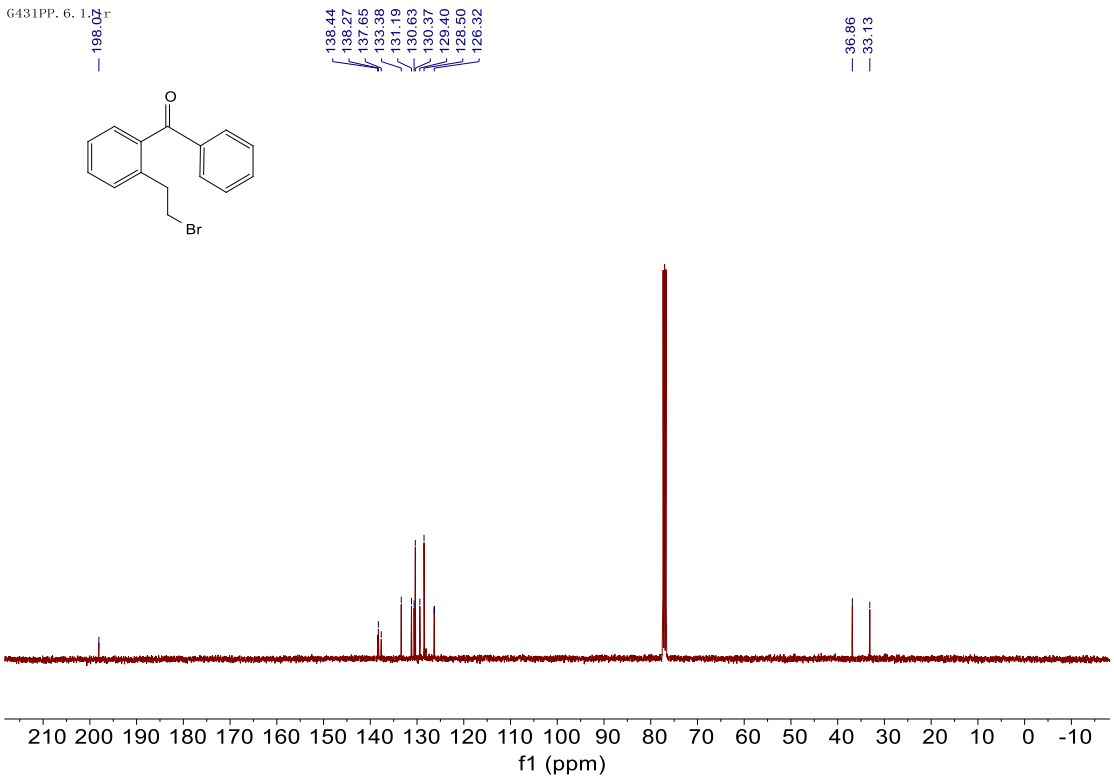
^{13}C NMR of compound **46**



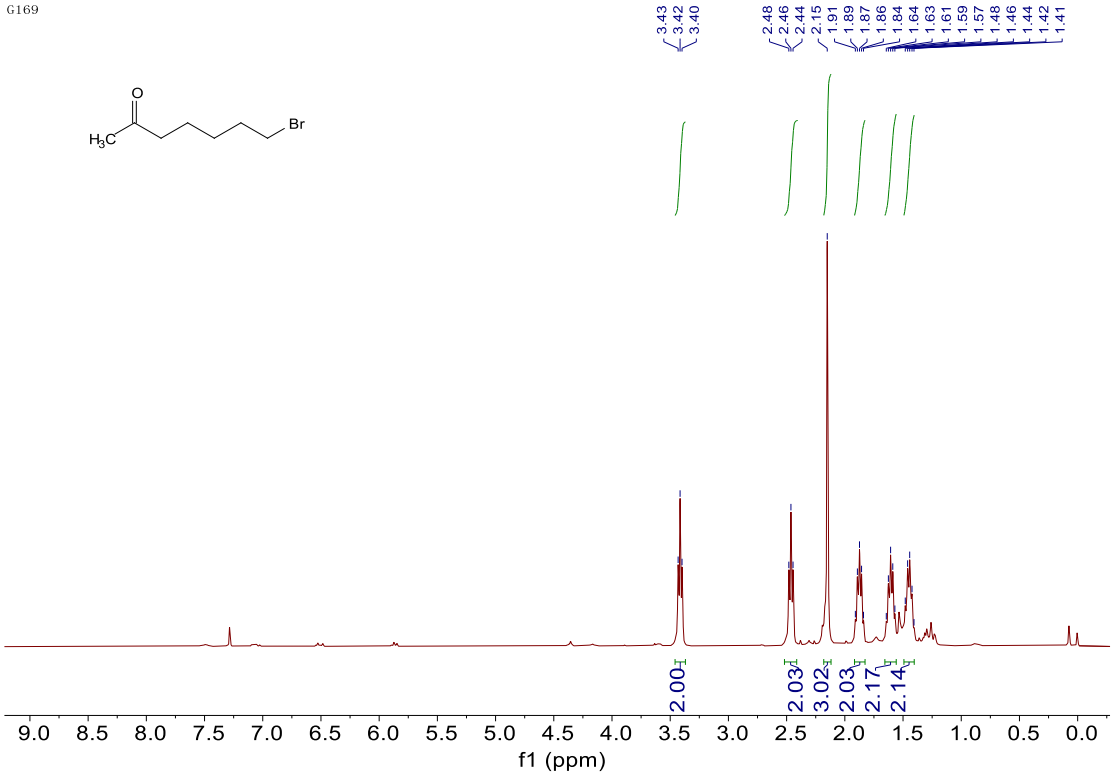
^{1}H NMR of compound **47**



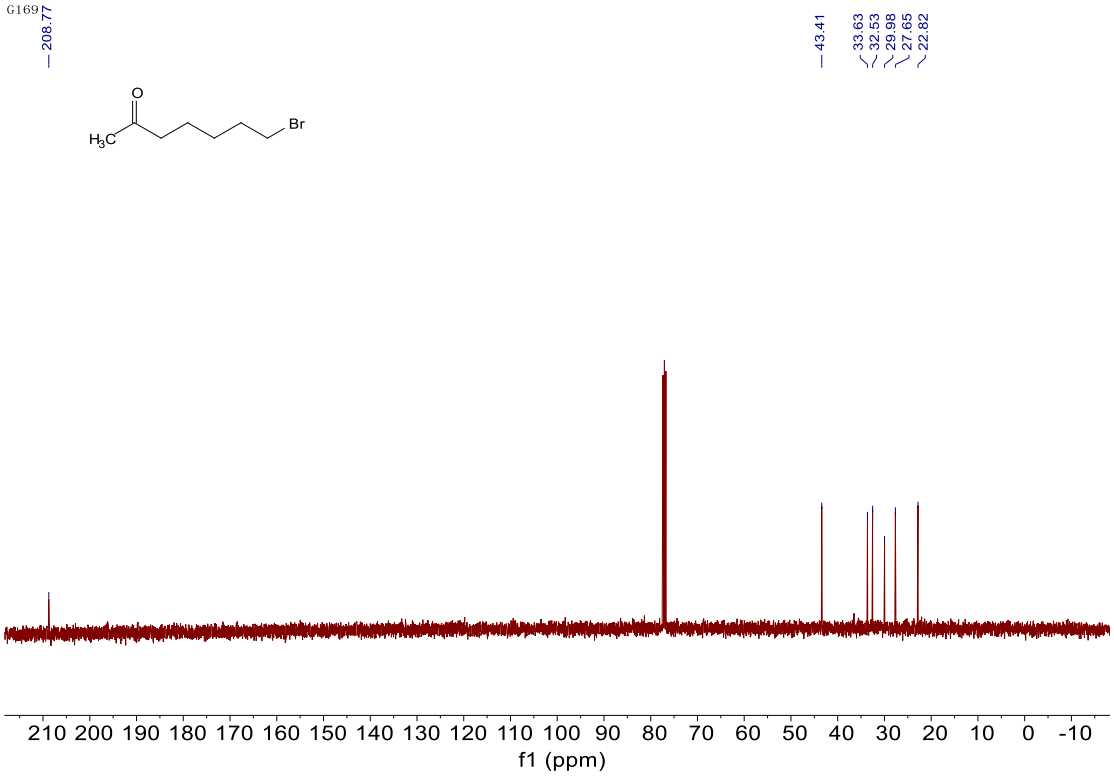
^{13}C NMR of compound **47**



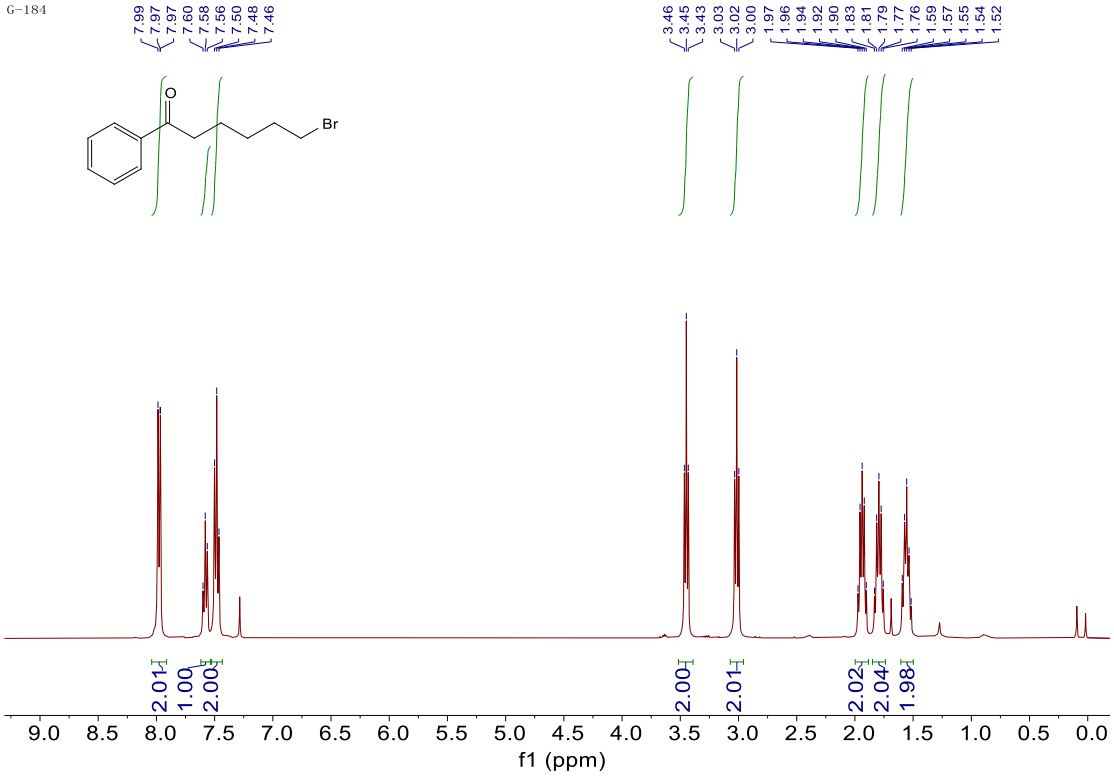
^{1}H NMR of compound **48**



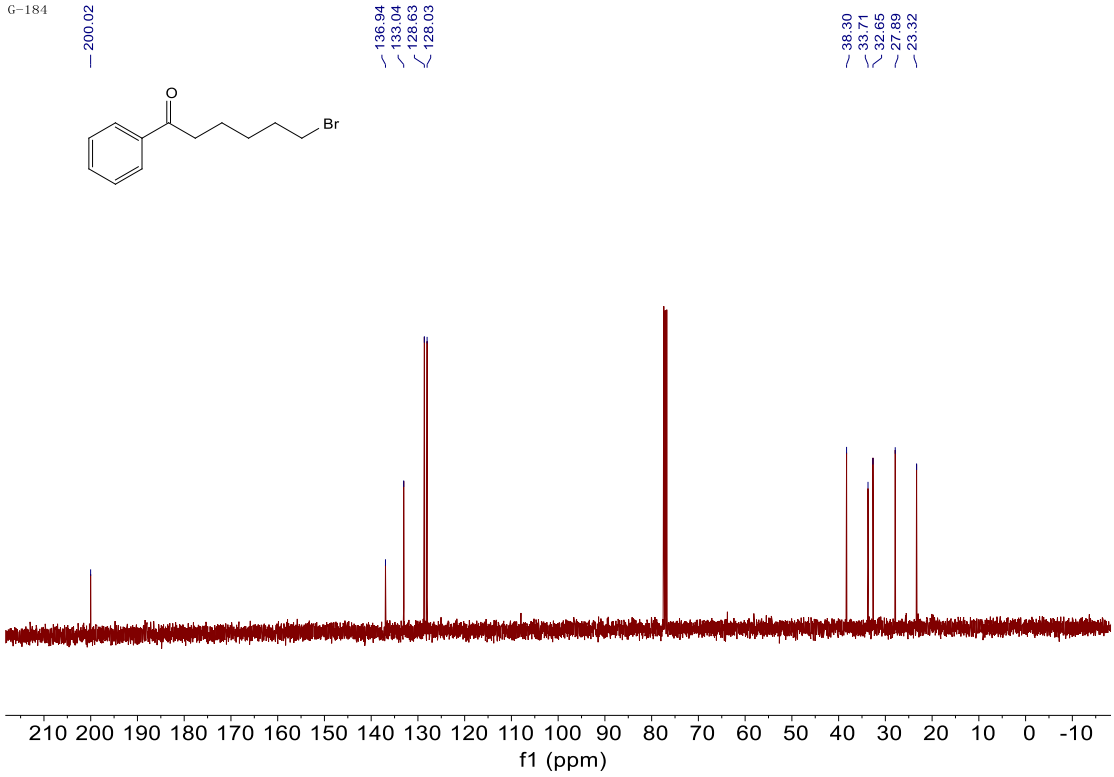
^{13}C NMR of compound **48**



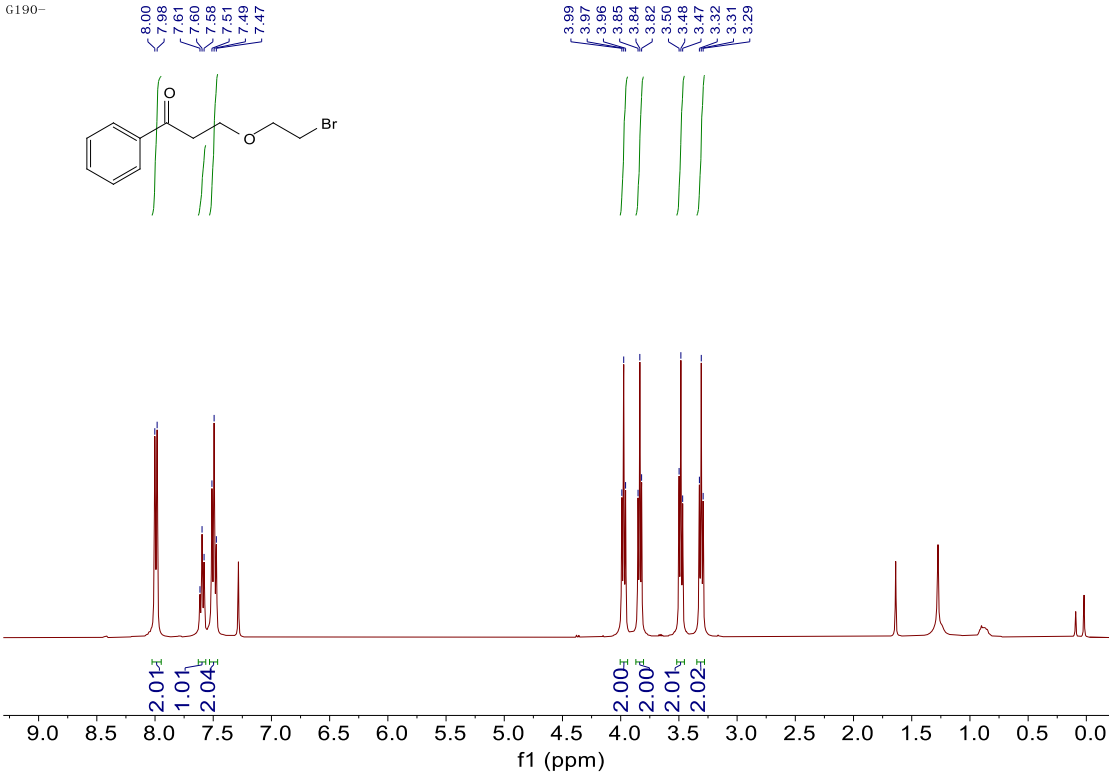
^{1}H NMR of compound **49**



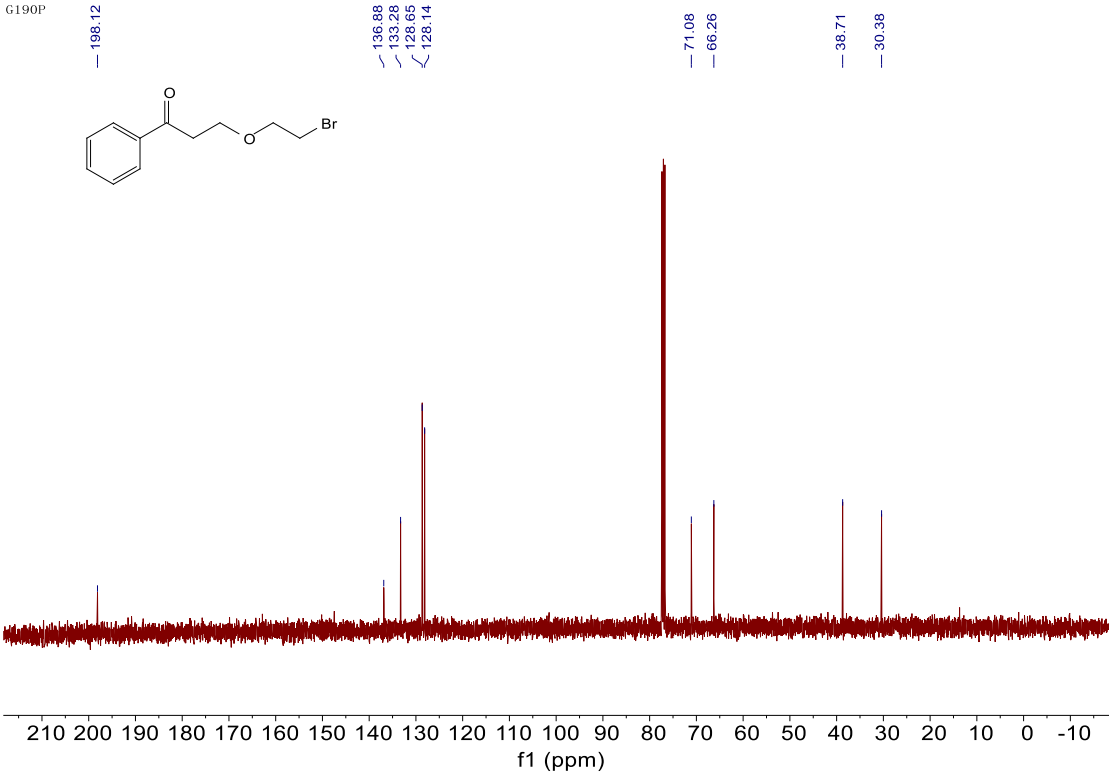
^{13}C NMR of compound **49**



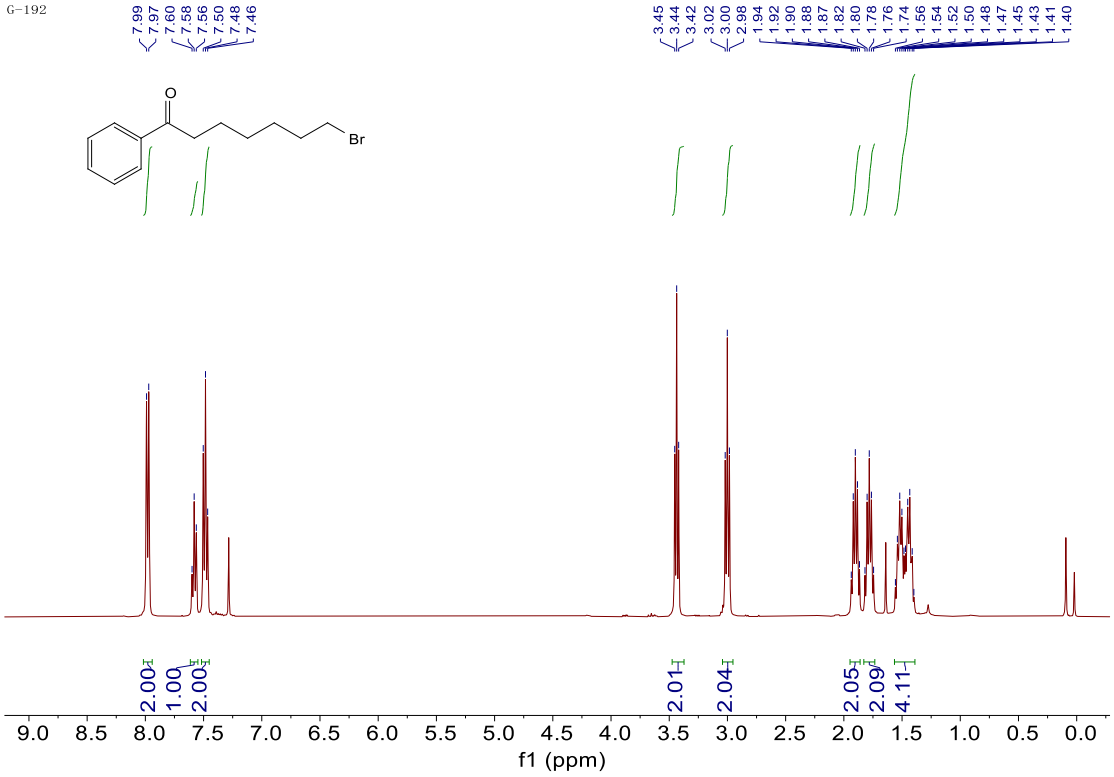
^{1}H NMR of compound **50**



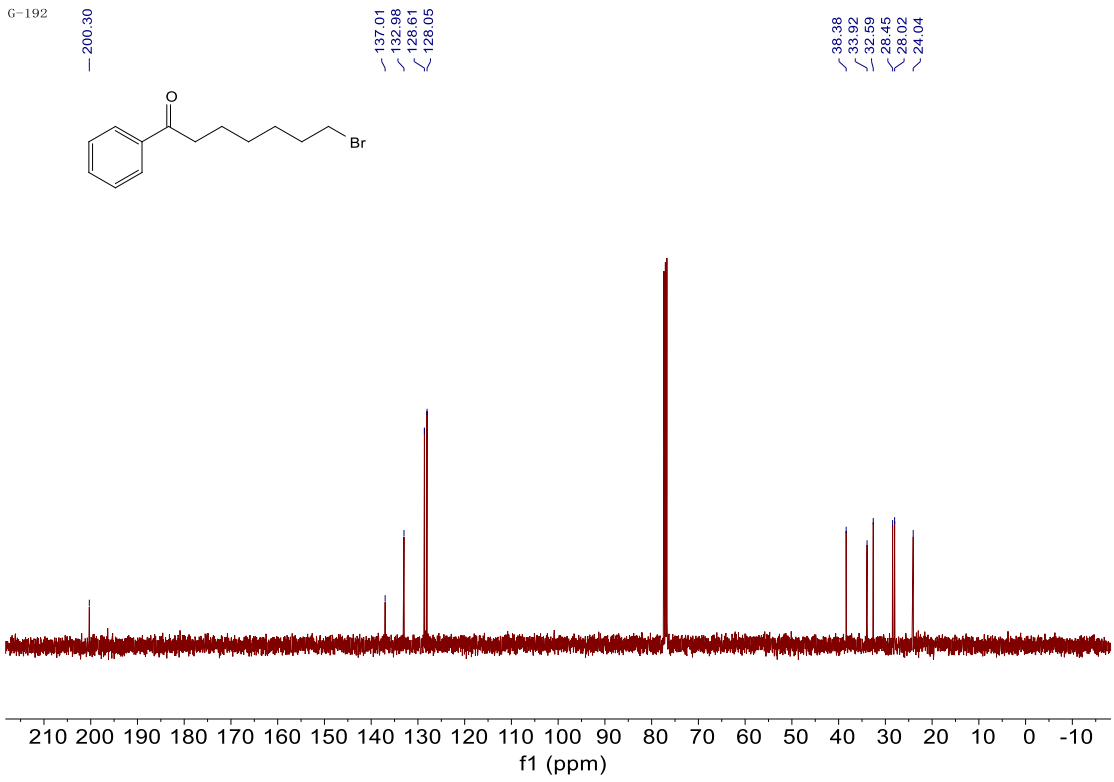
^{13}C NMR of compound **50**



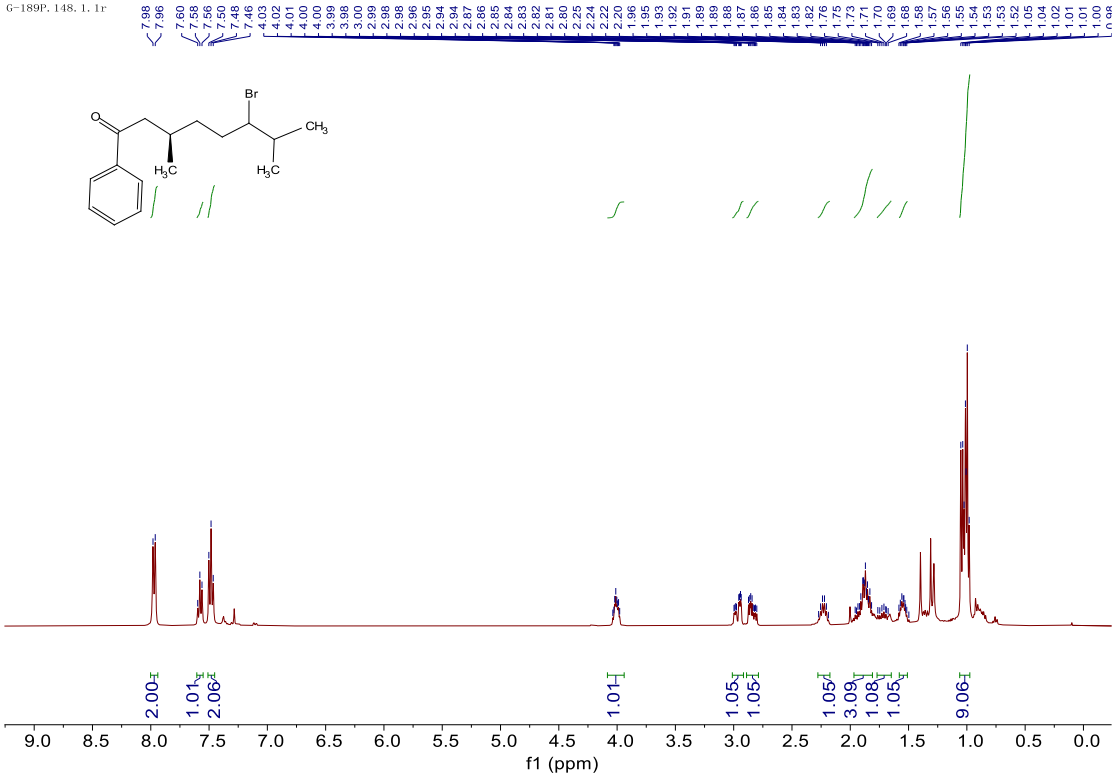
^{1}H NMR of compound **51**



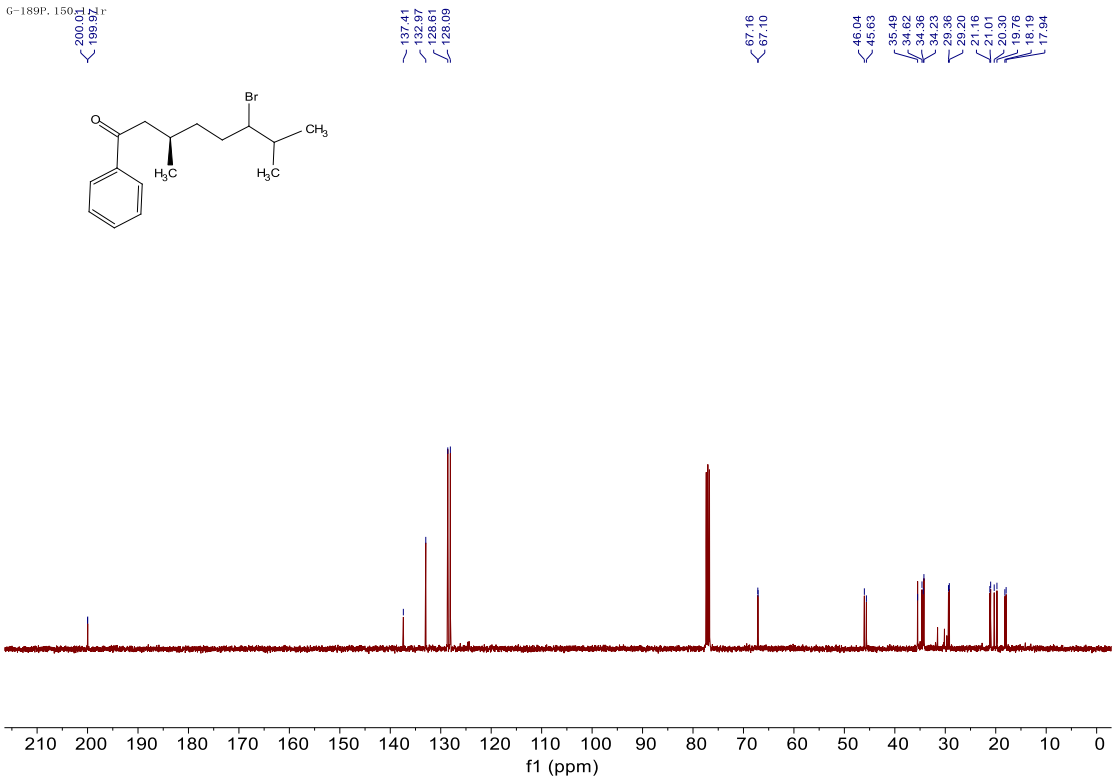
^{13}C NMR of compound **51**



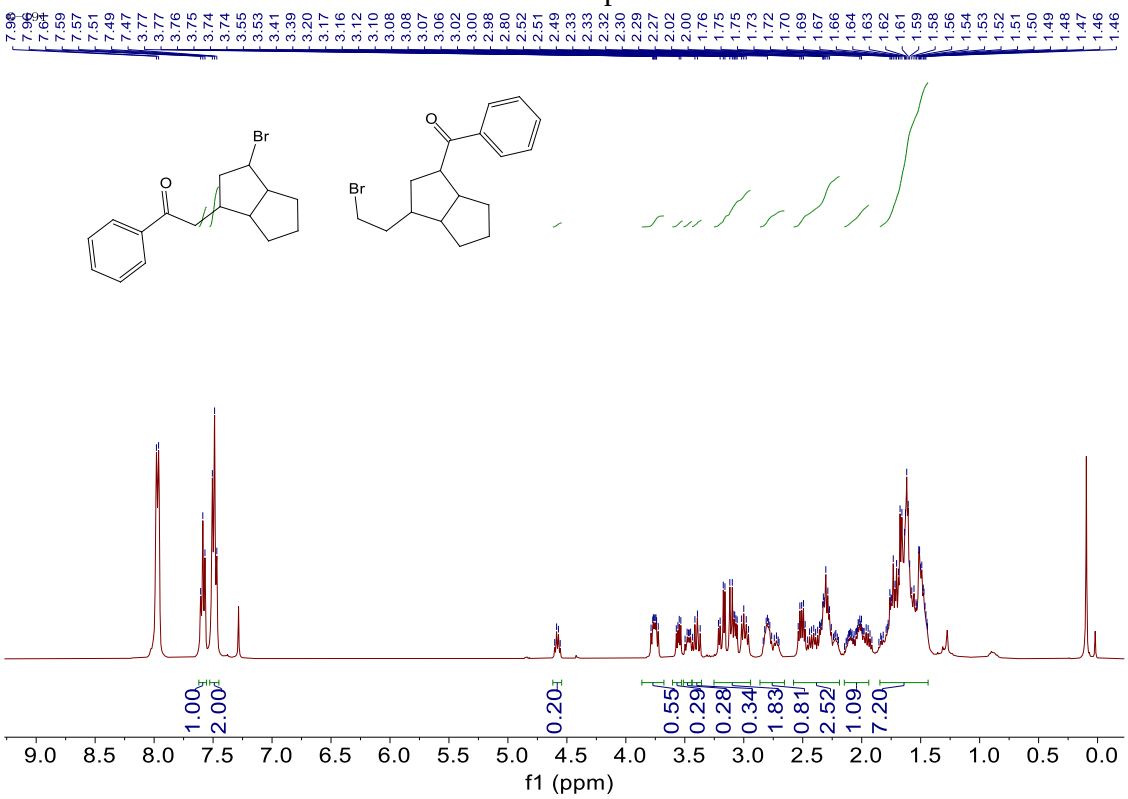
^{1}H NMR of compound **52**



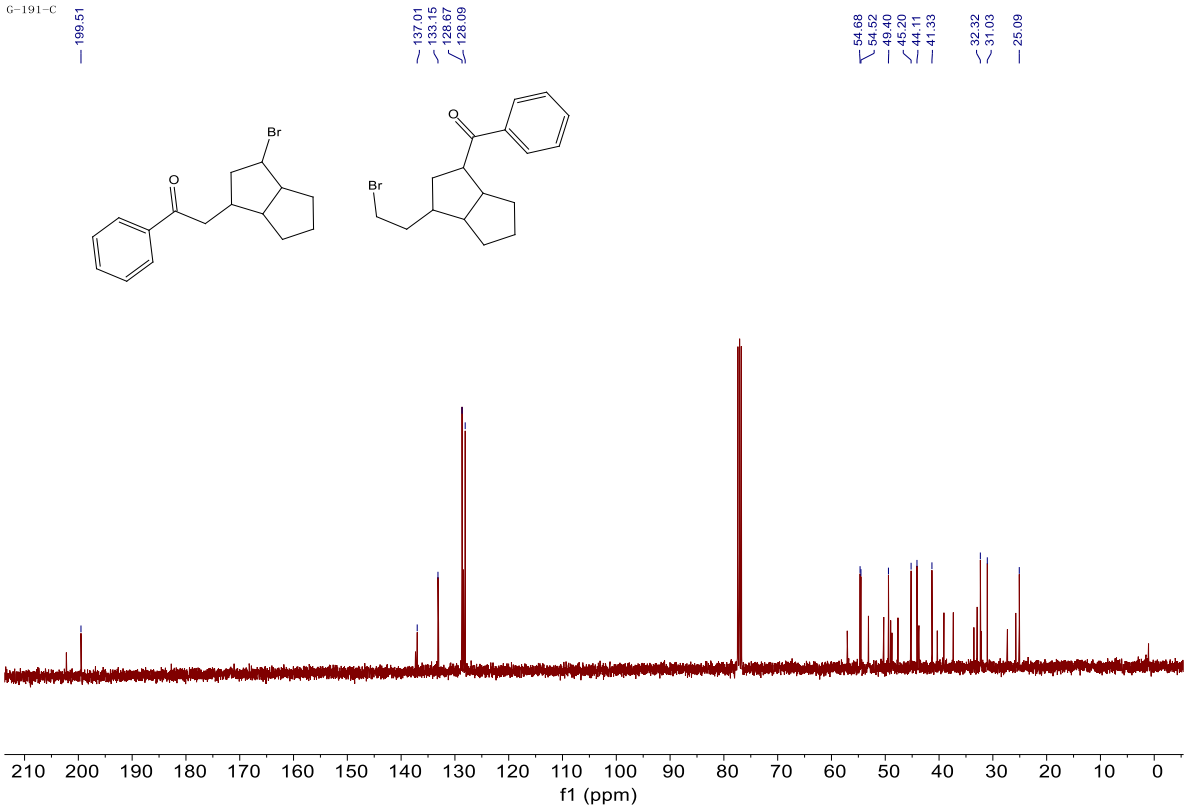
^{13}C NMR of compound **52**



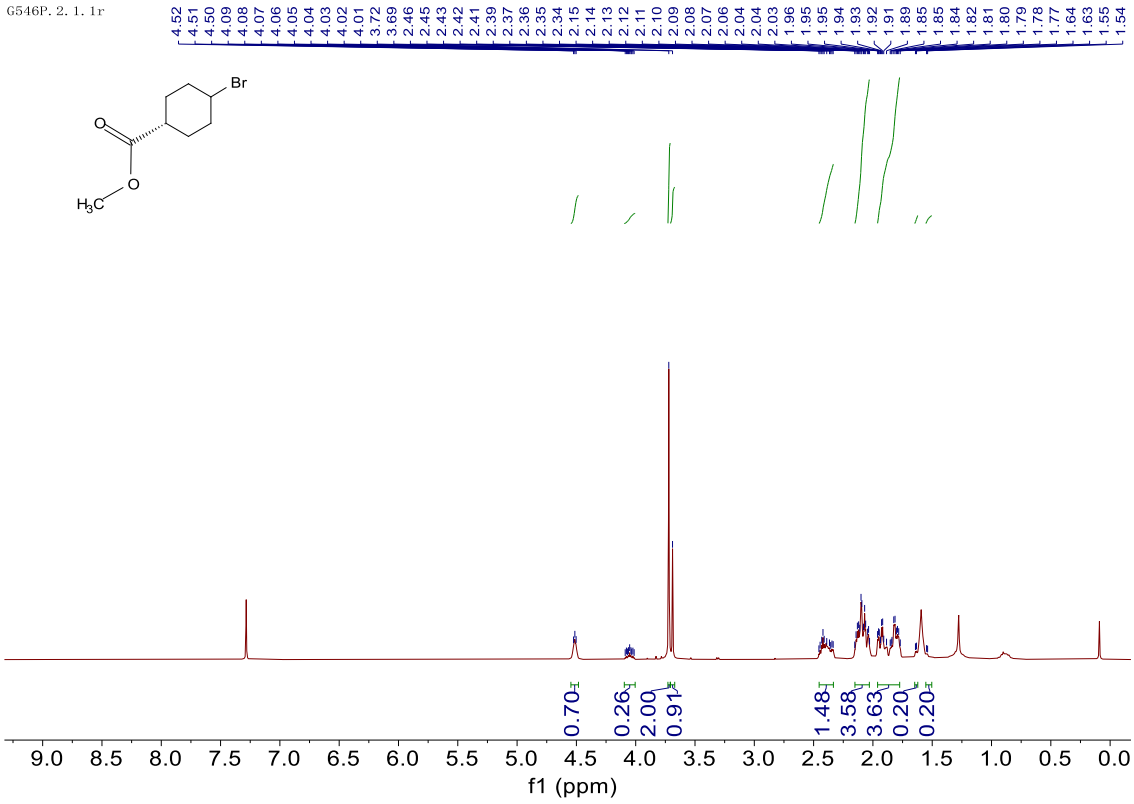
^{1}H NMR of compound **53**



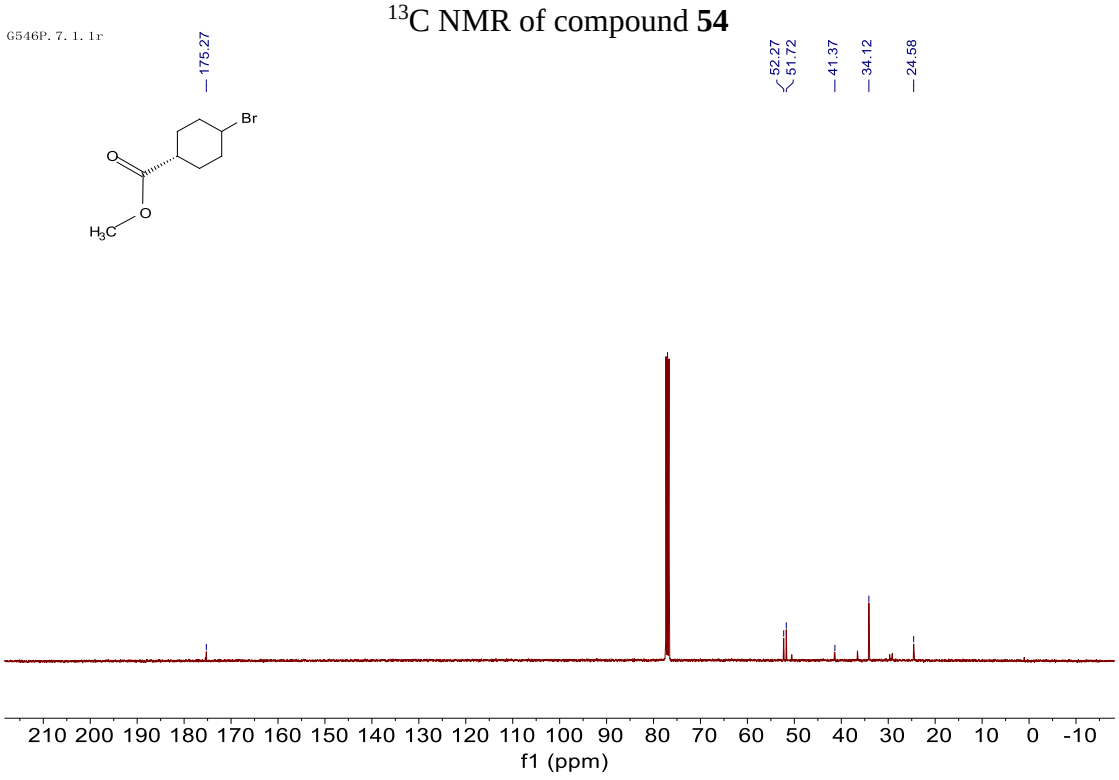
^{13}C NMR of compound **53**



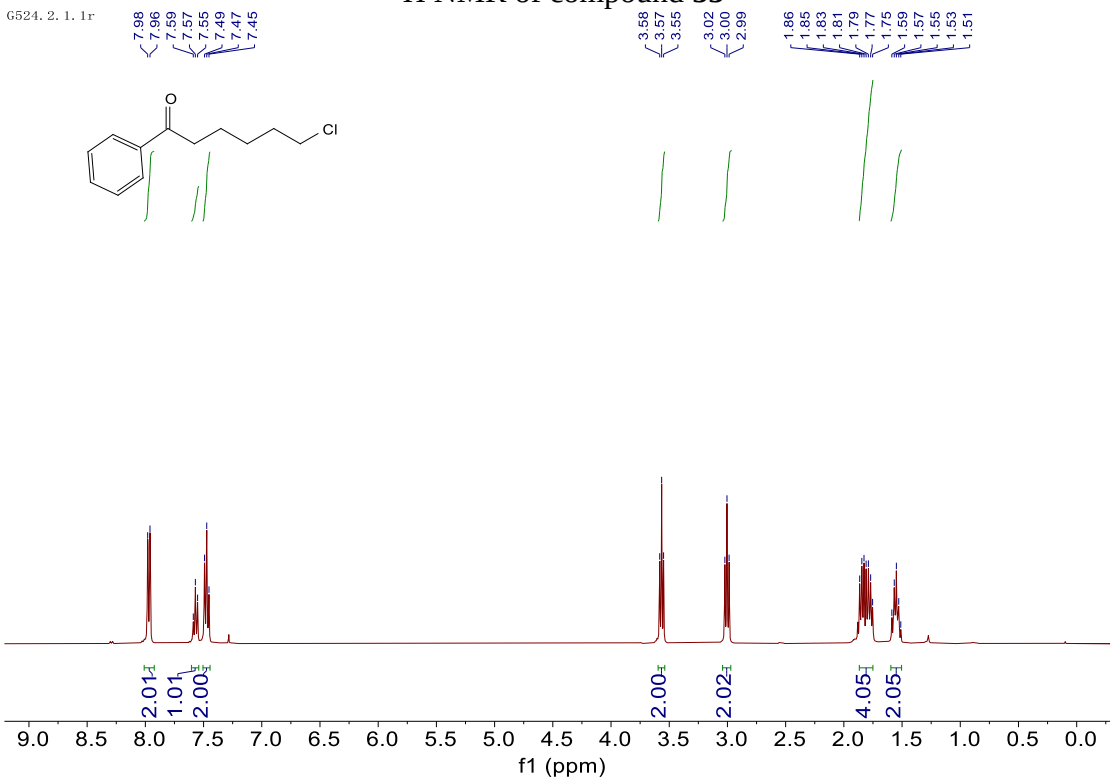
^{1}H NMR of compound **54**



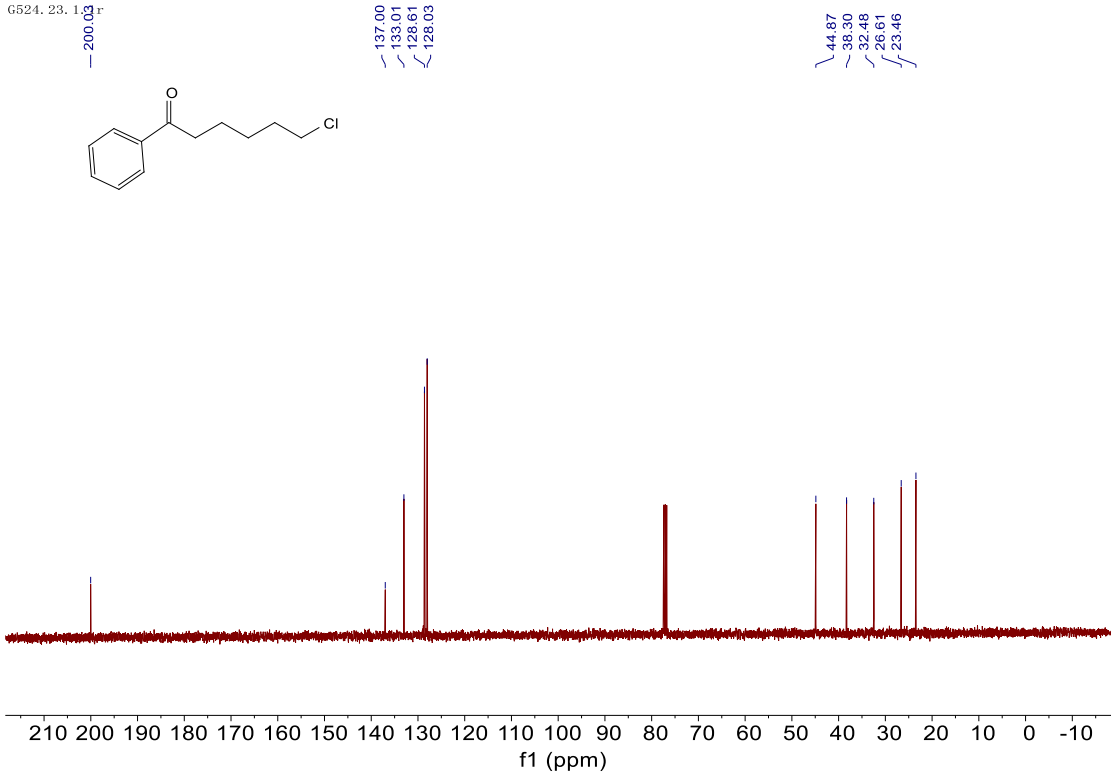
^{13}C NMR of compound **54**



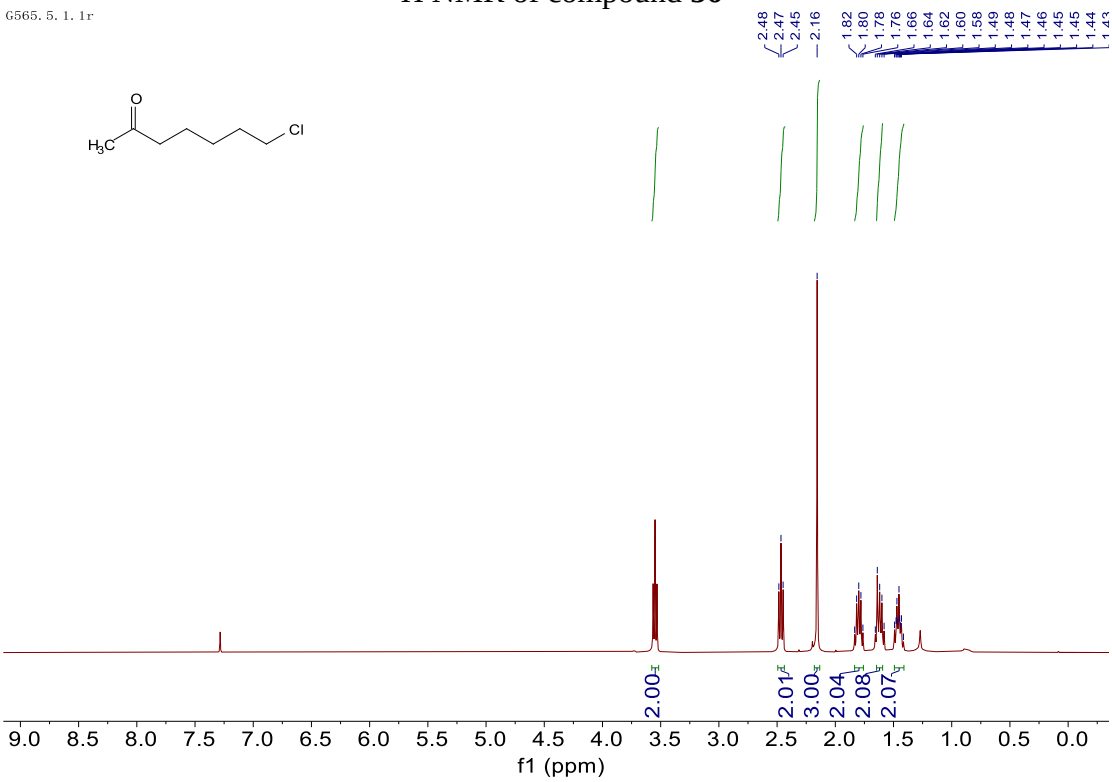
^{1}H NMR of compound **55**



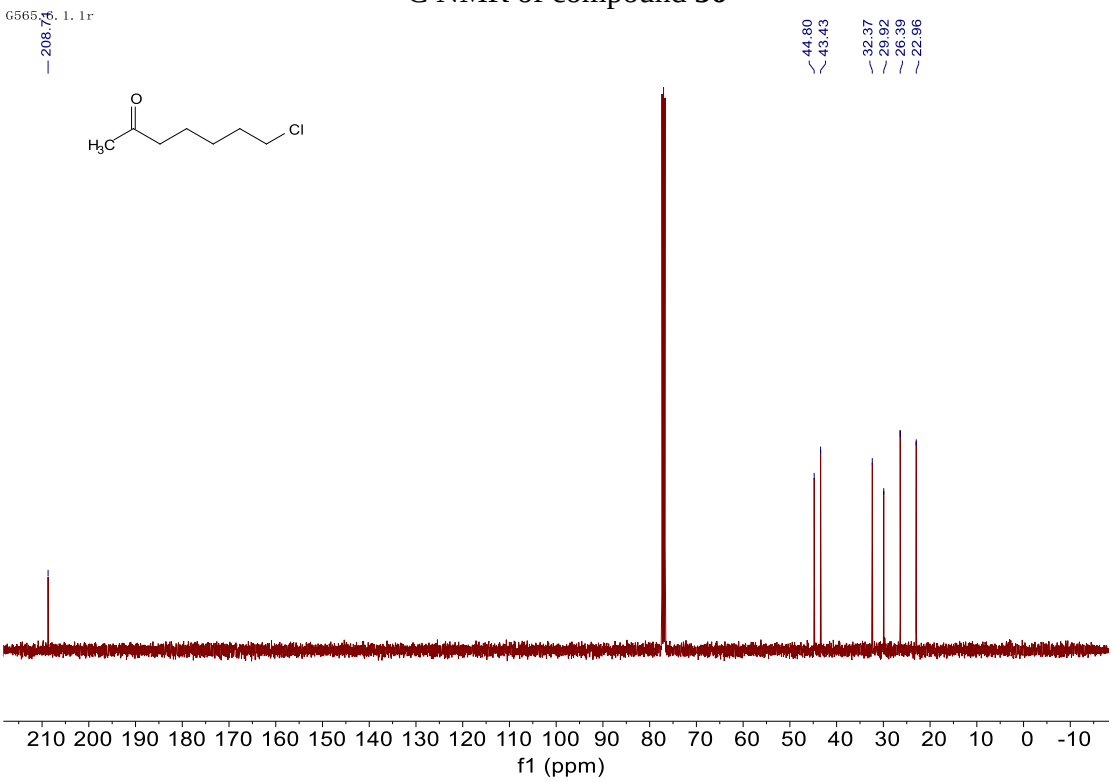
^{13}C NMR of compound **55**



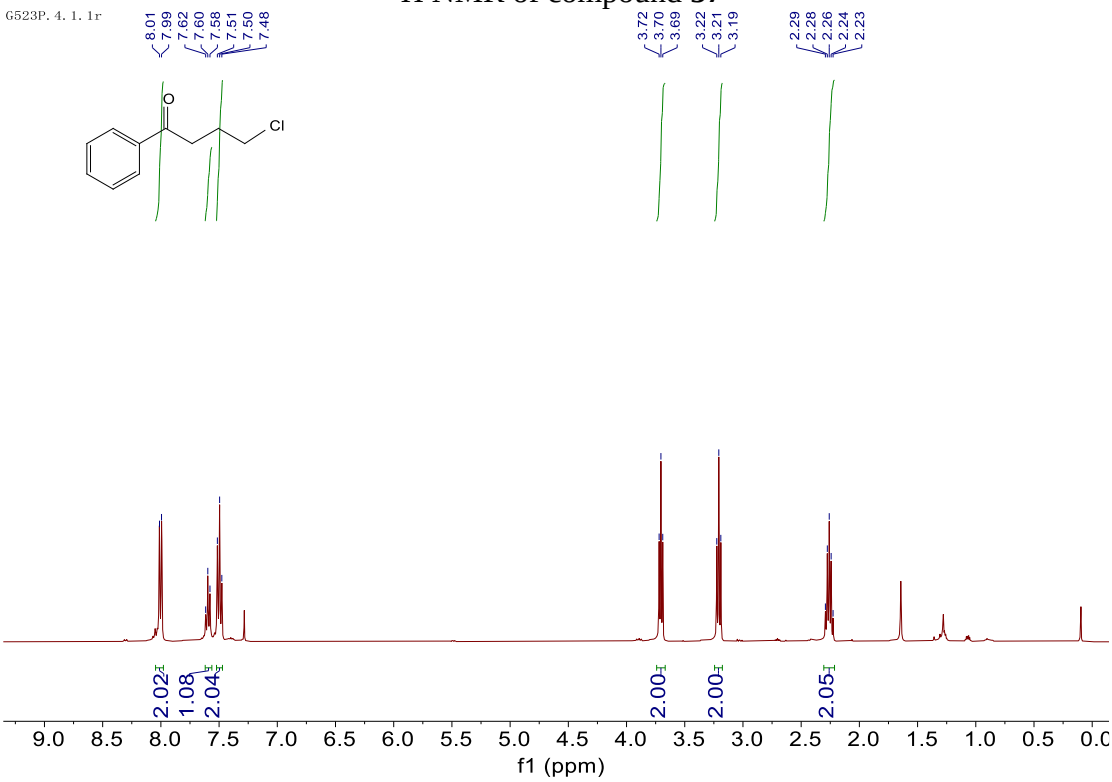
^{1}H NMR of compound **56**



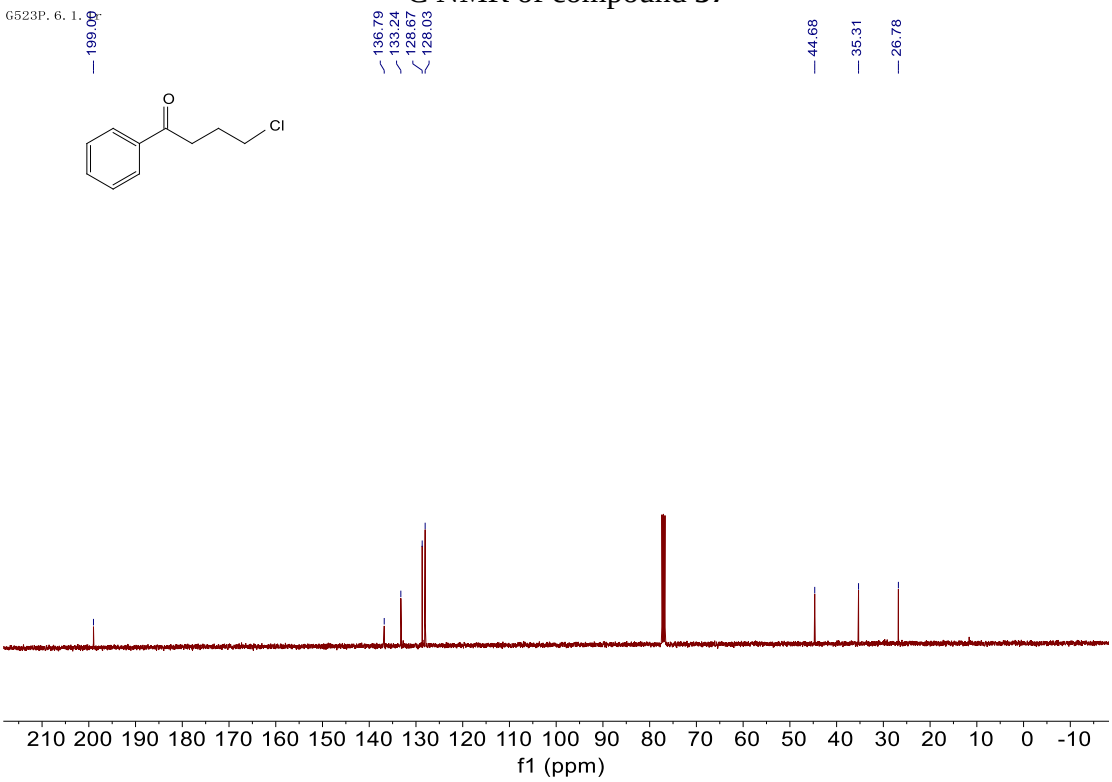
^{13}C NMR of compound **56**



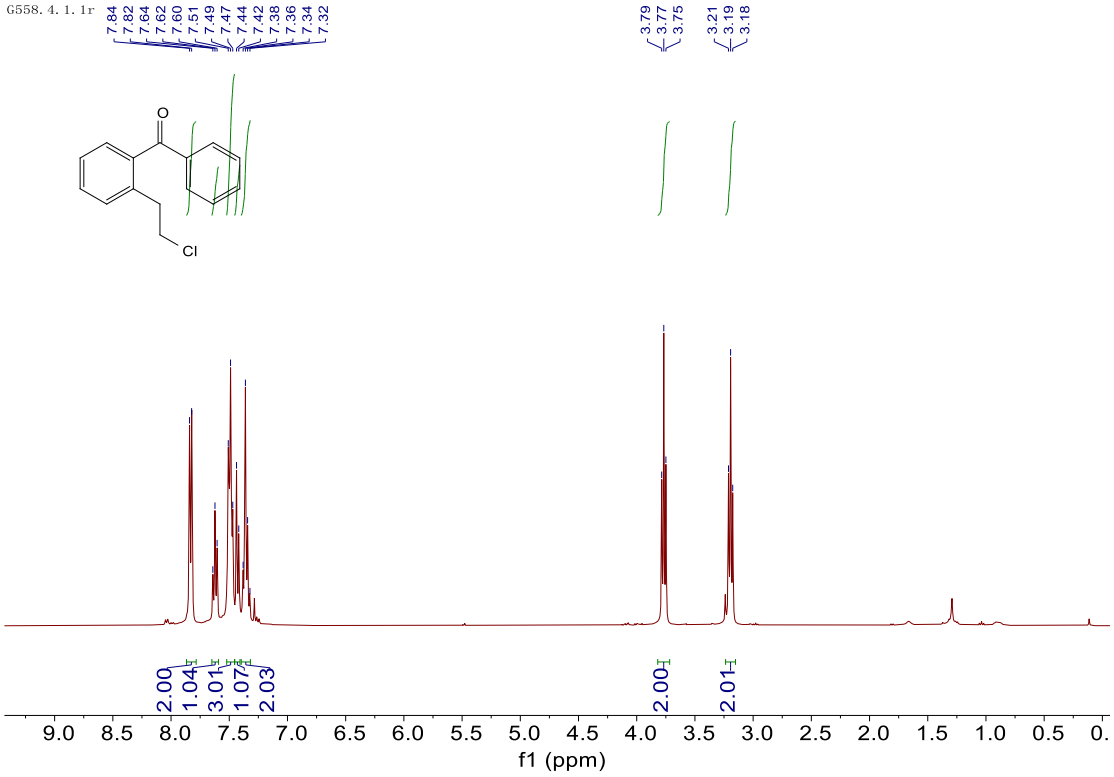
^{1}H NMR of compound **57**



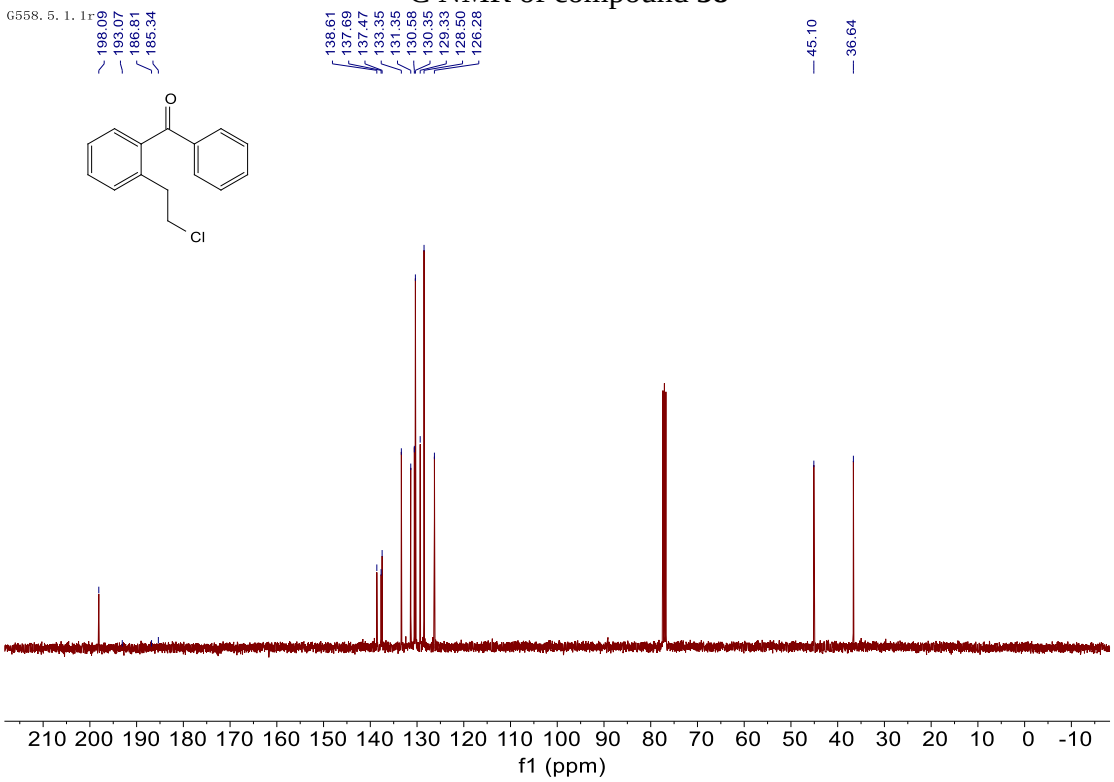
^{13}C NMR of compound **57**



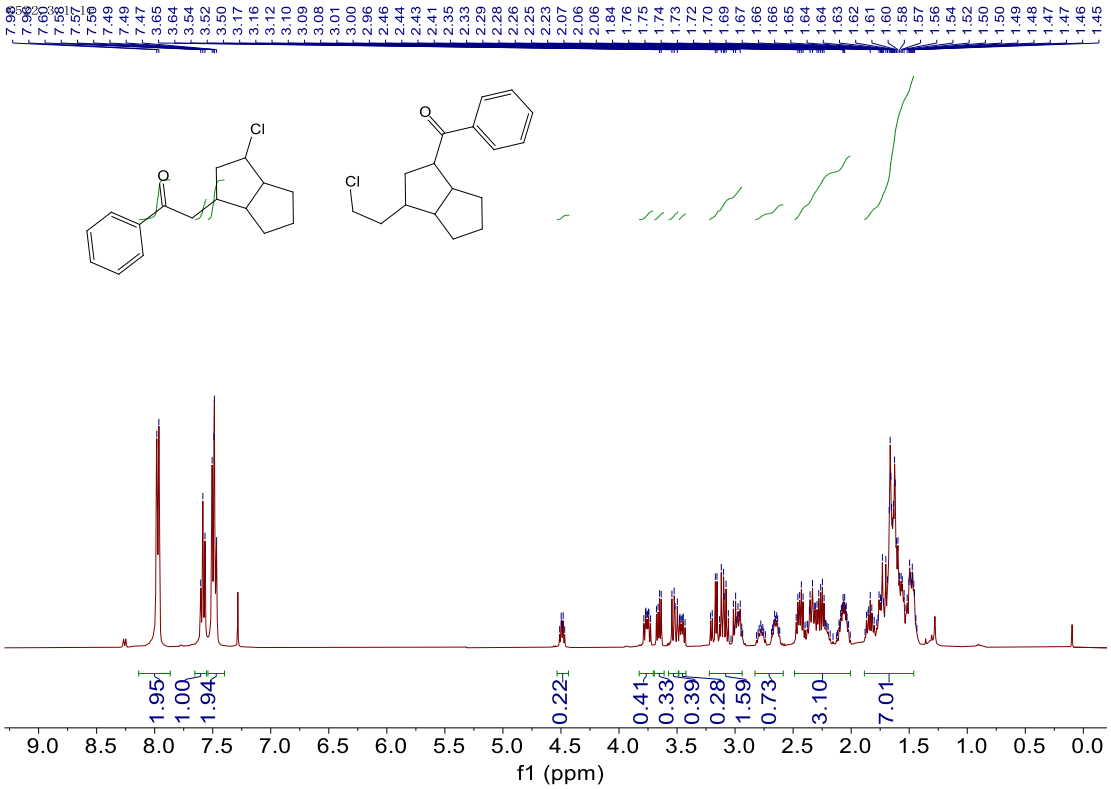
^{1}H NMR of compound **58**



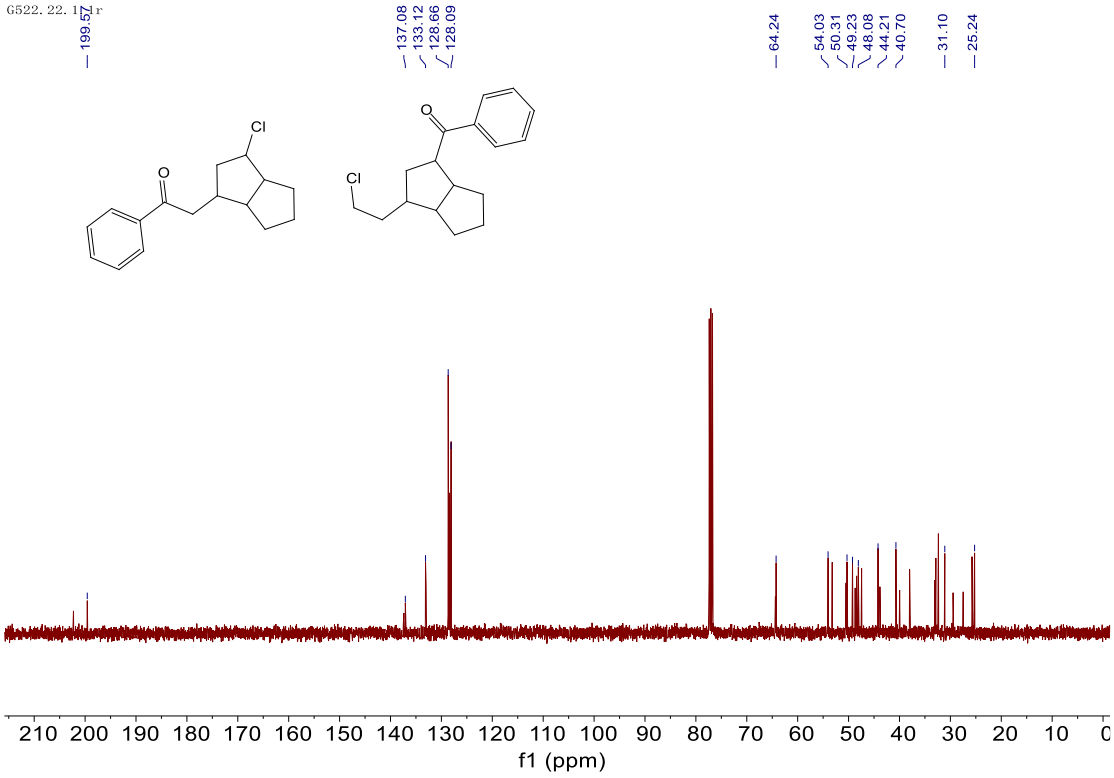
^{13}C NMR of compound **58**



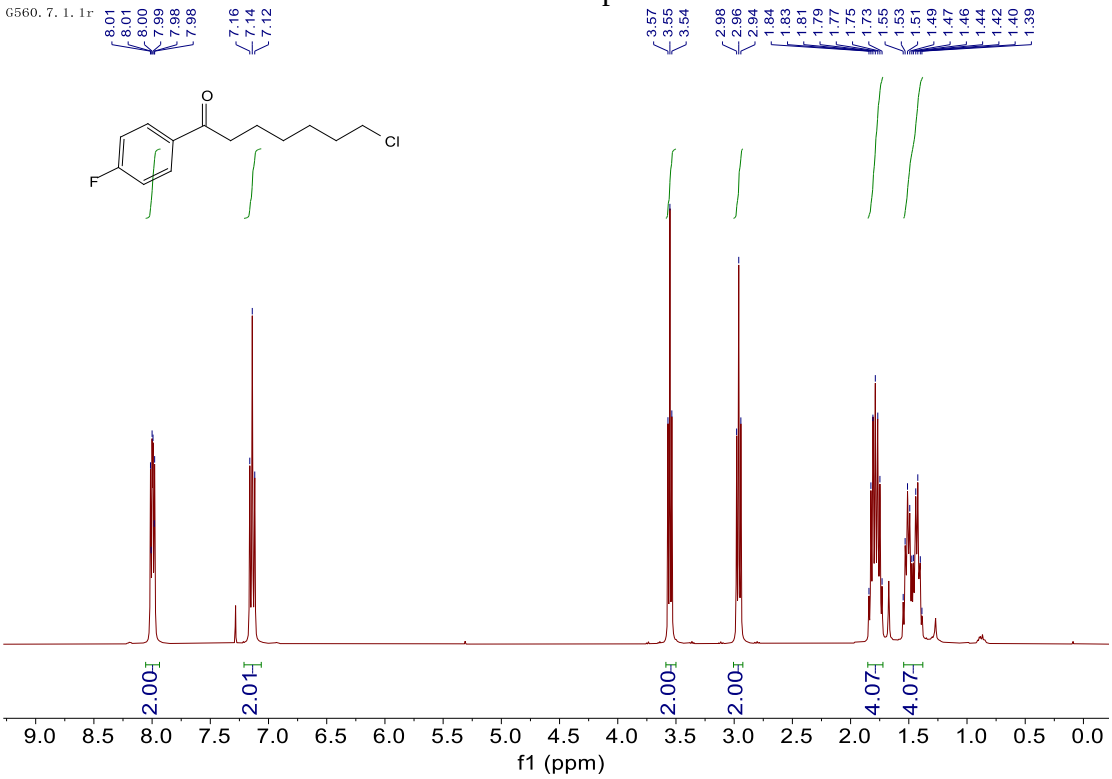
^{1}H NMR of compound **59**



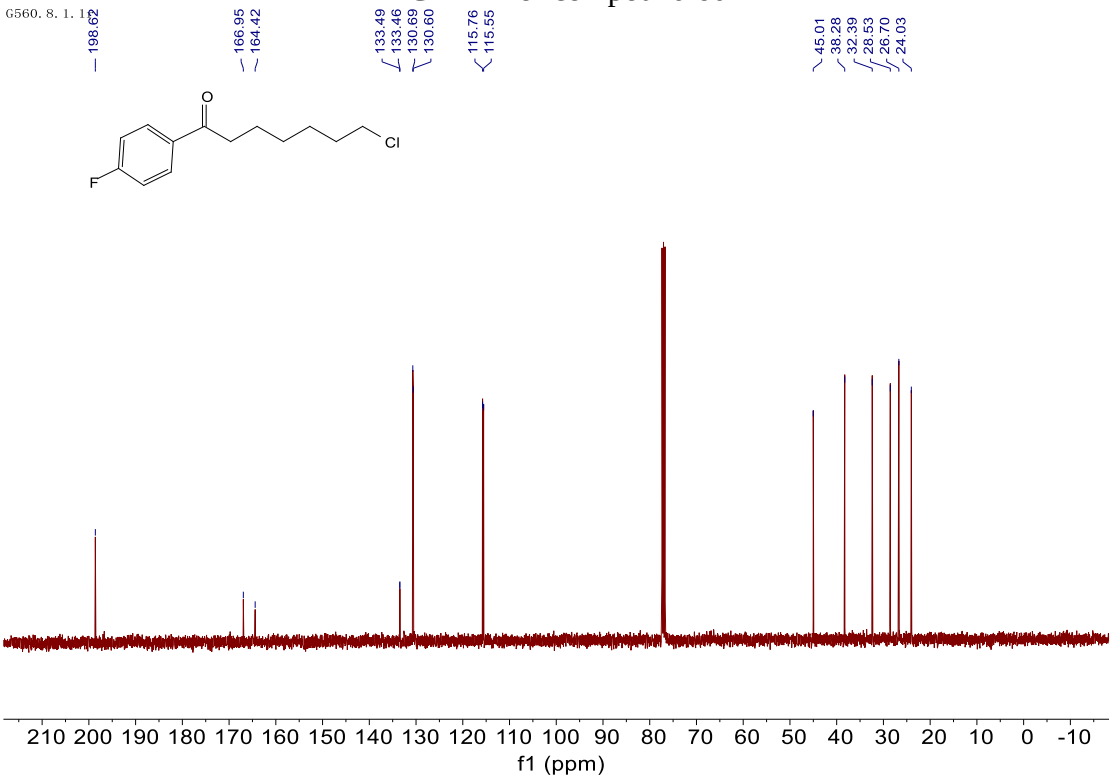
^{13}C NMR of compound **59**



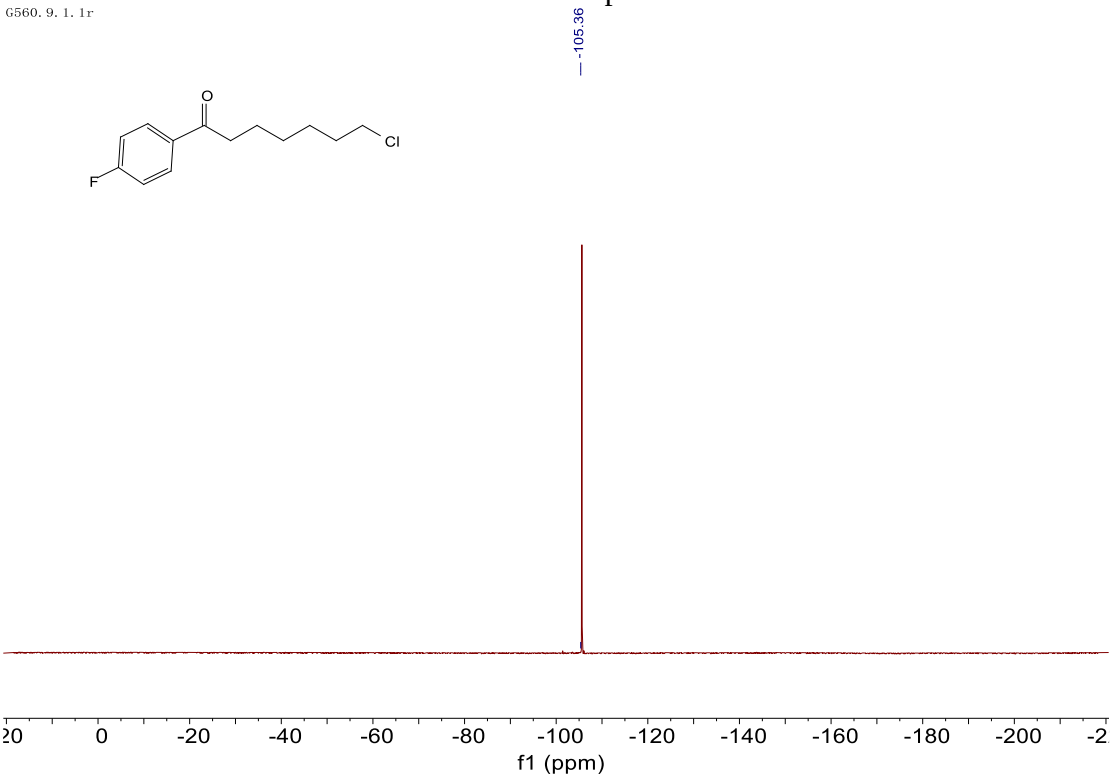
^{1}H NMR of compound **60**



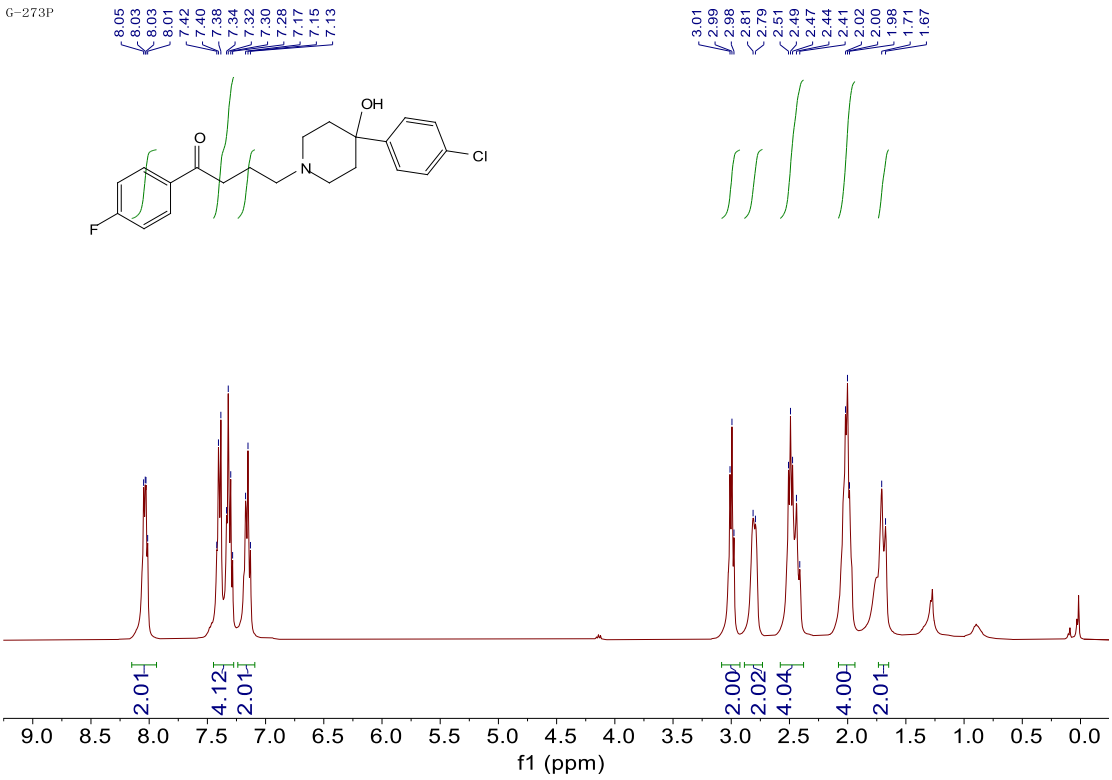
^{13}C NMR of compound **60**



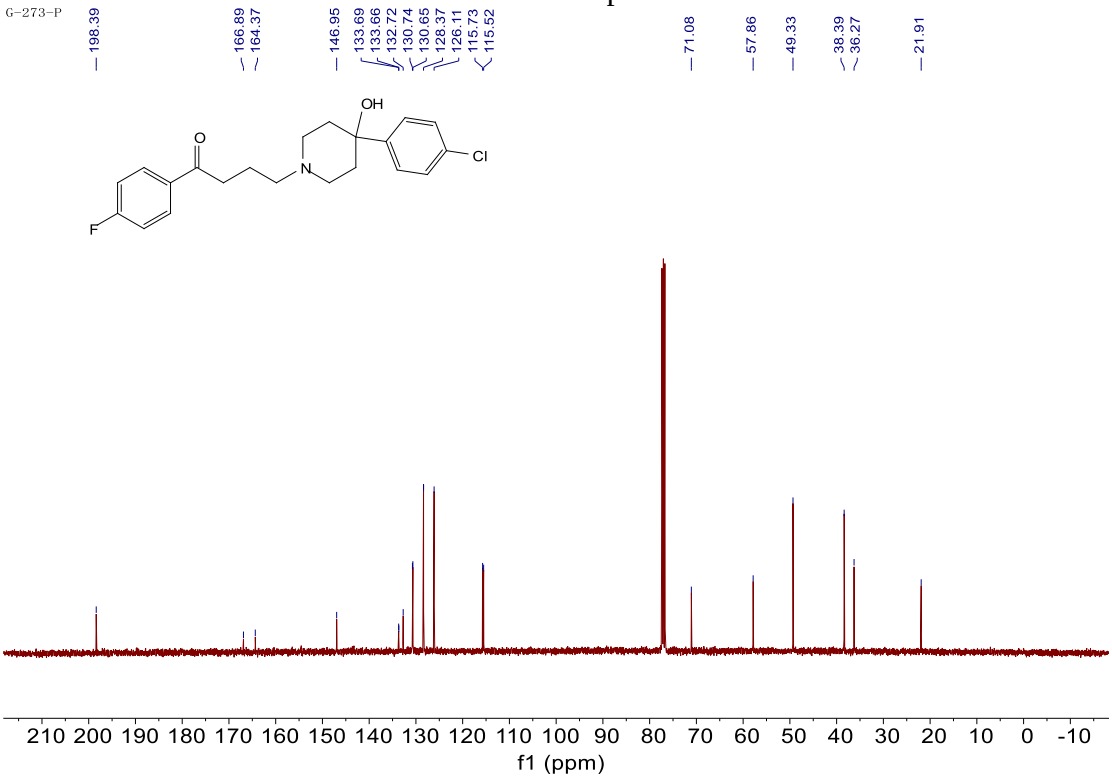
^{19}F NMR of compound **60**



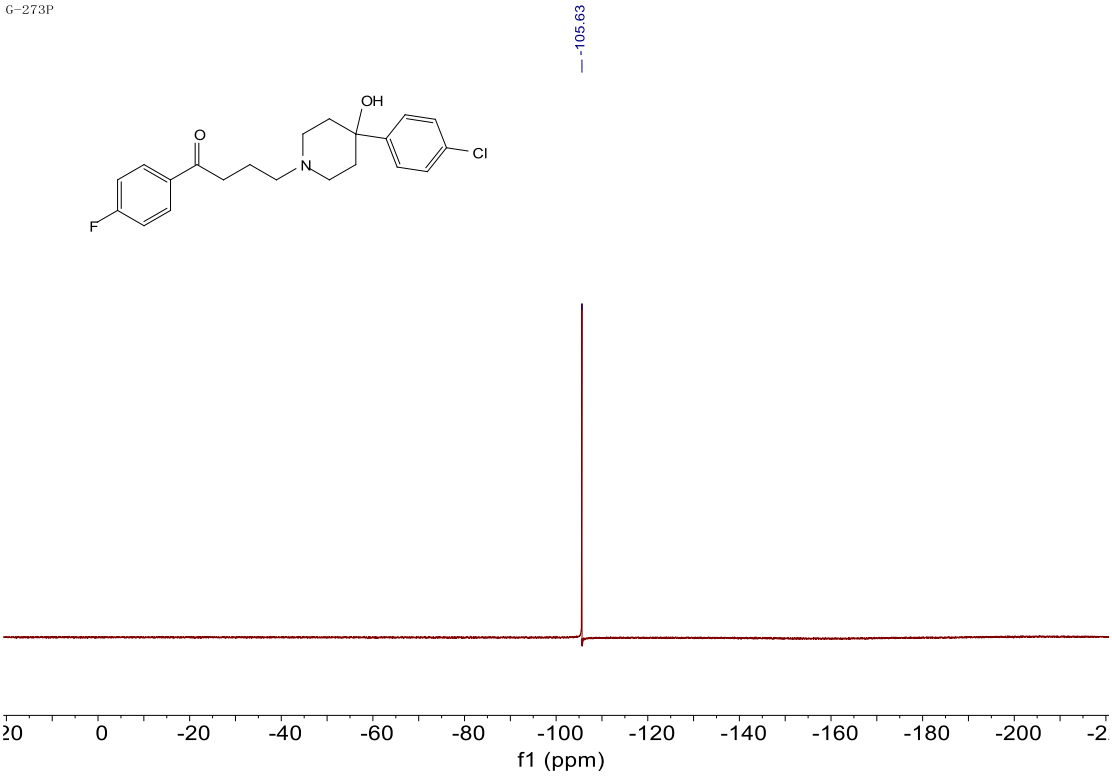
^{1}H NMR of compound **61**



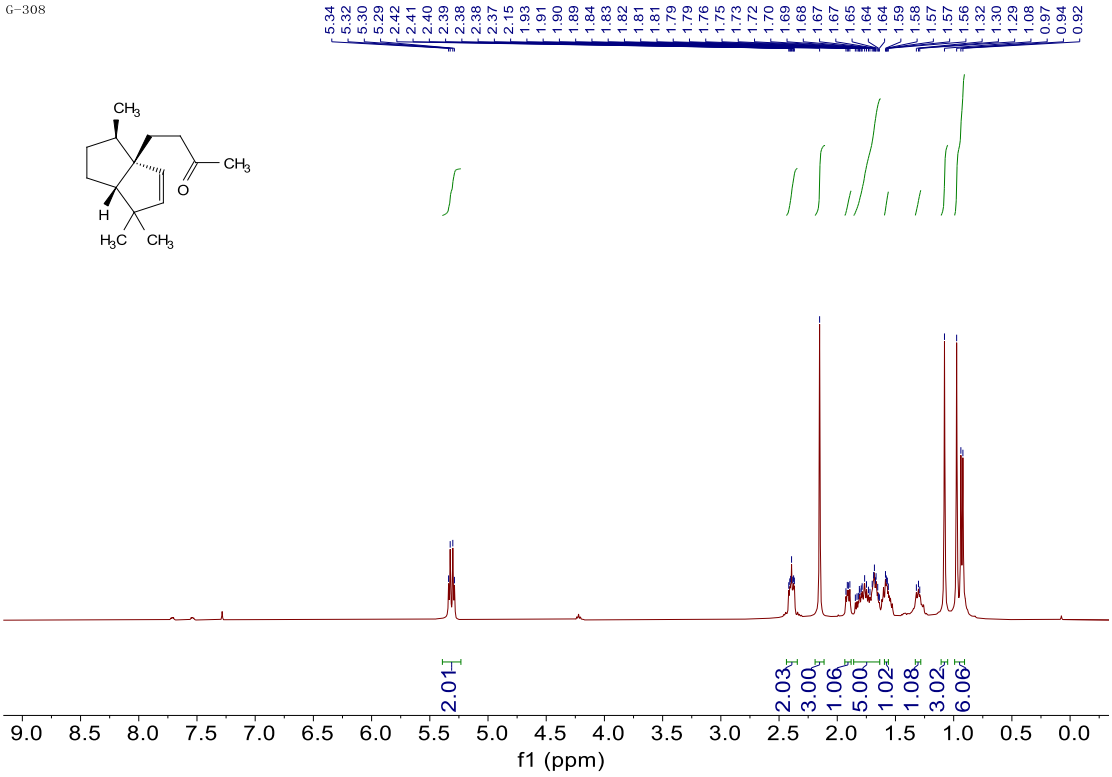
^{13}C NMR of compound **61**



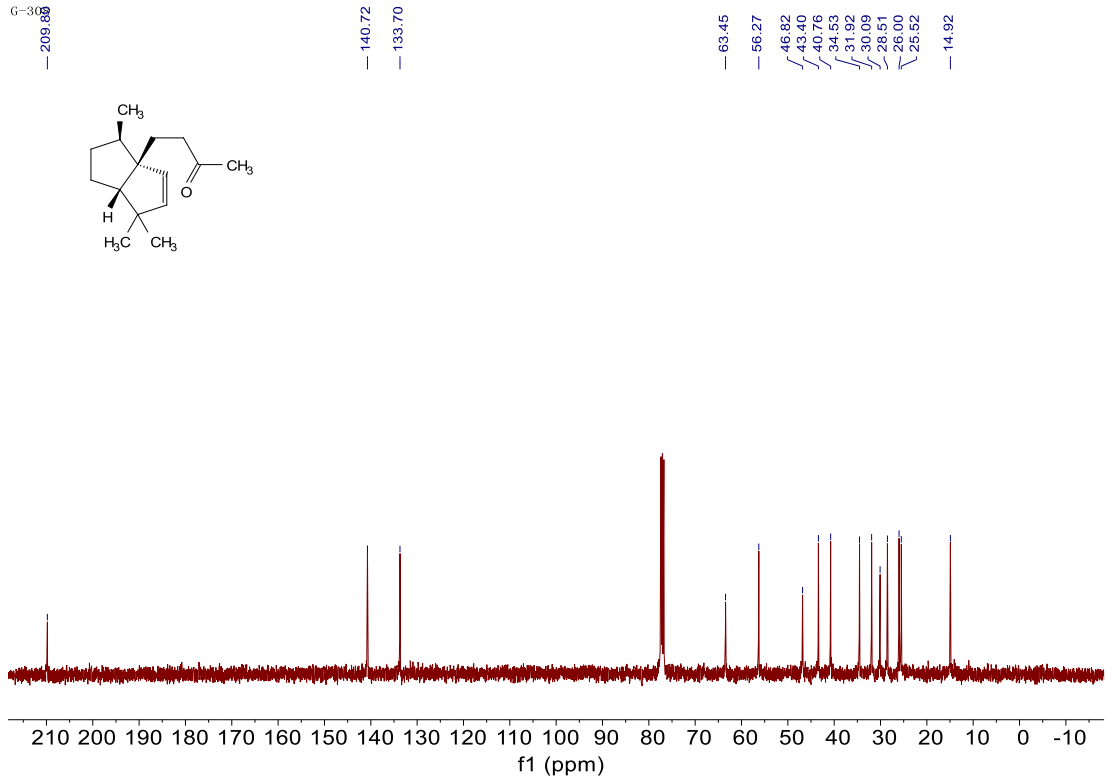
^{19}F NMR of compound **61**



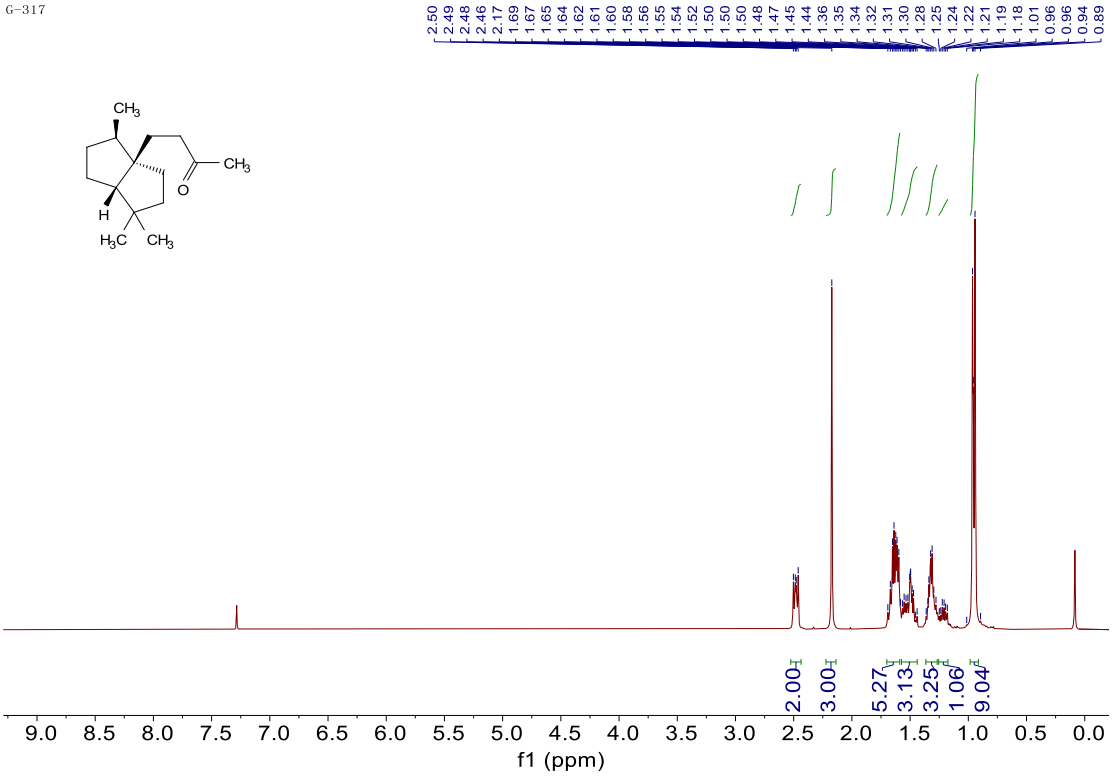
^{1}H NMR of compound **62**



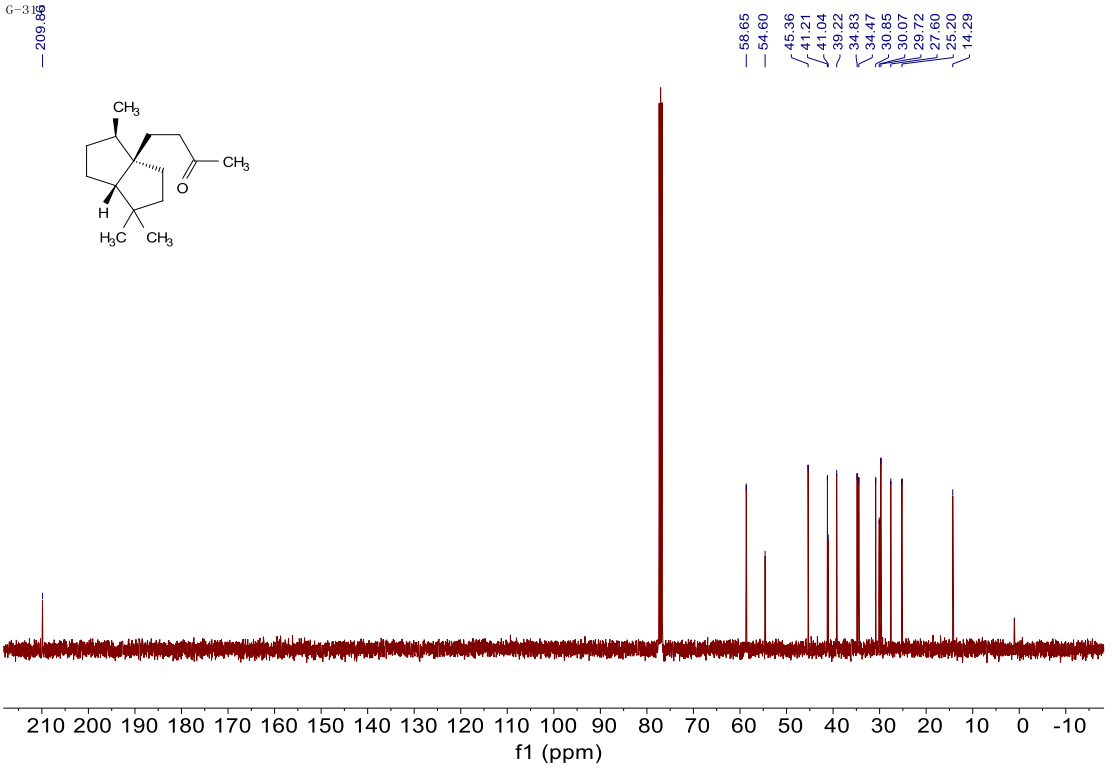
^{13}C NMR of compound **62**



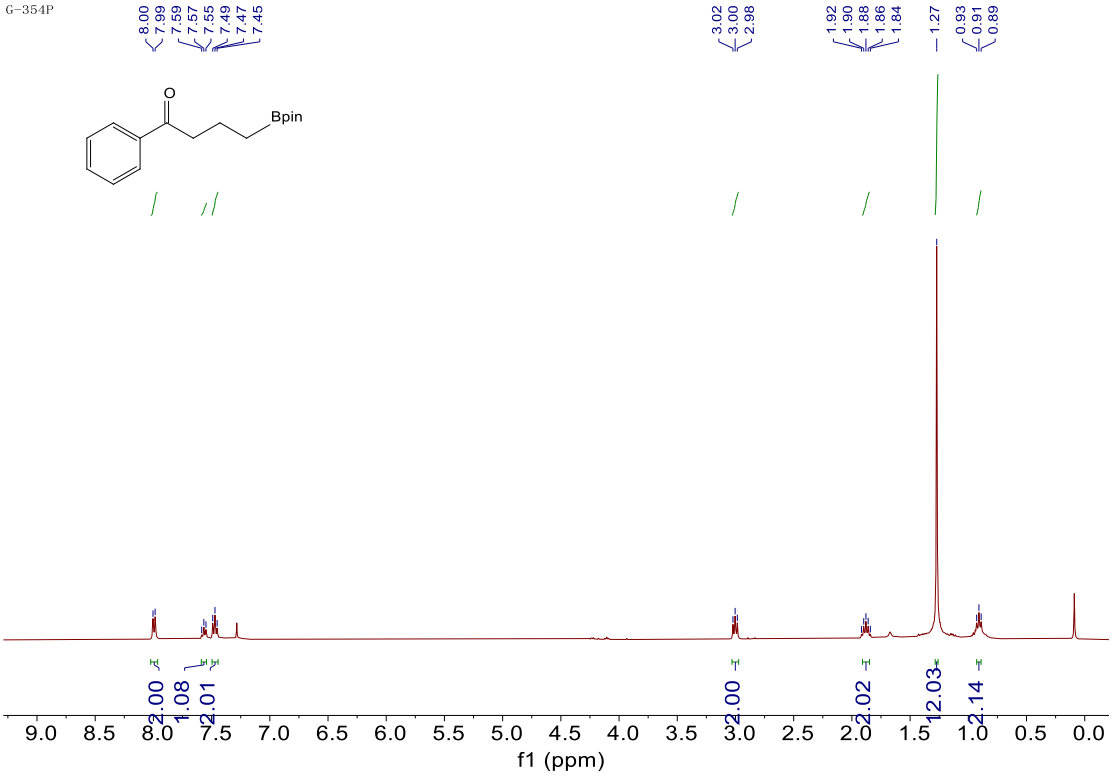
^{1}H NMR of compound **63**



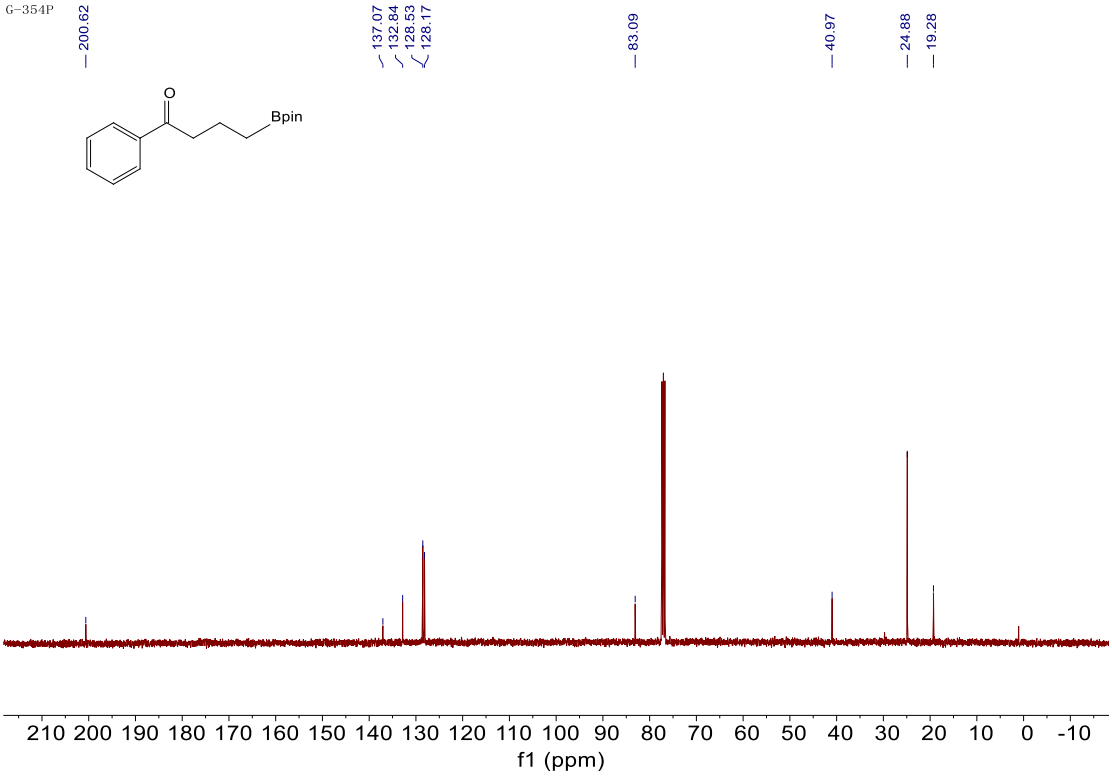
^{13}C NMR of compound **63**



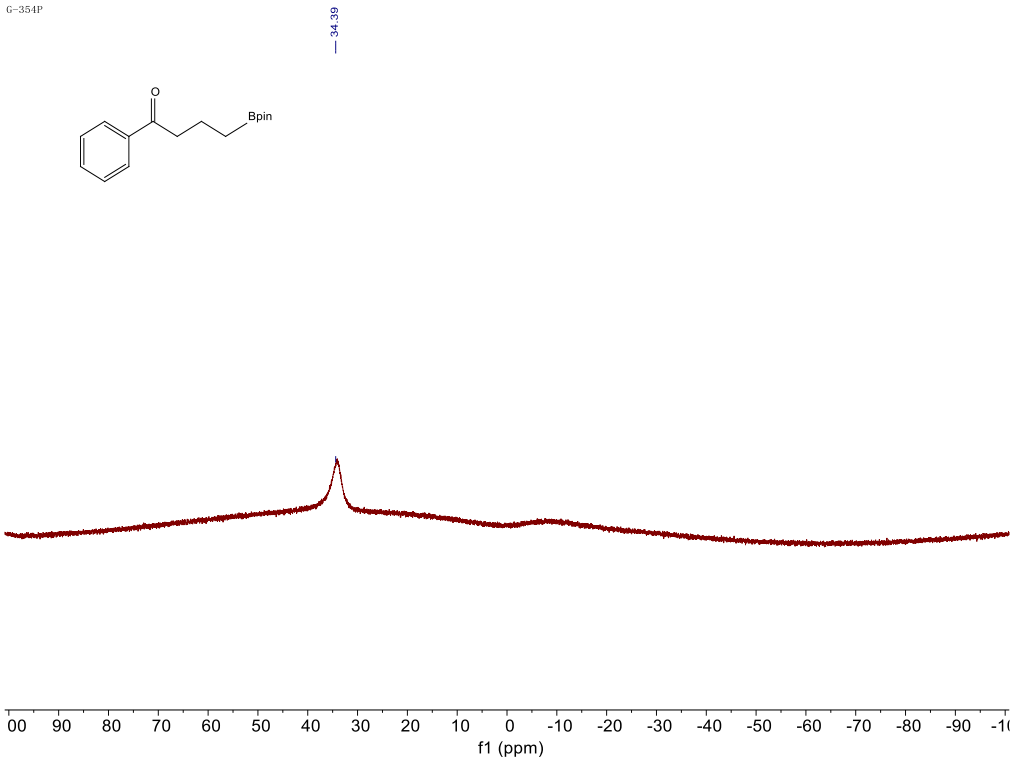
^{1}H NMR of compound **64**



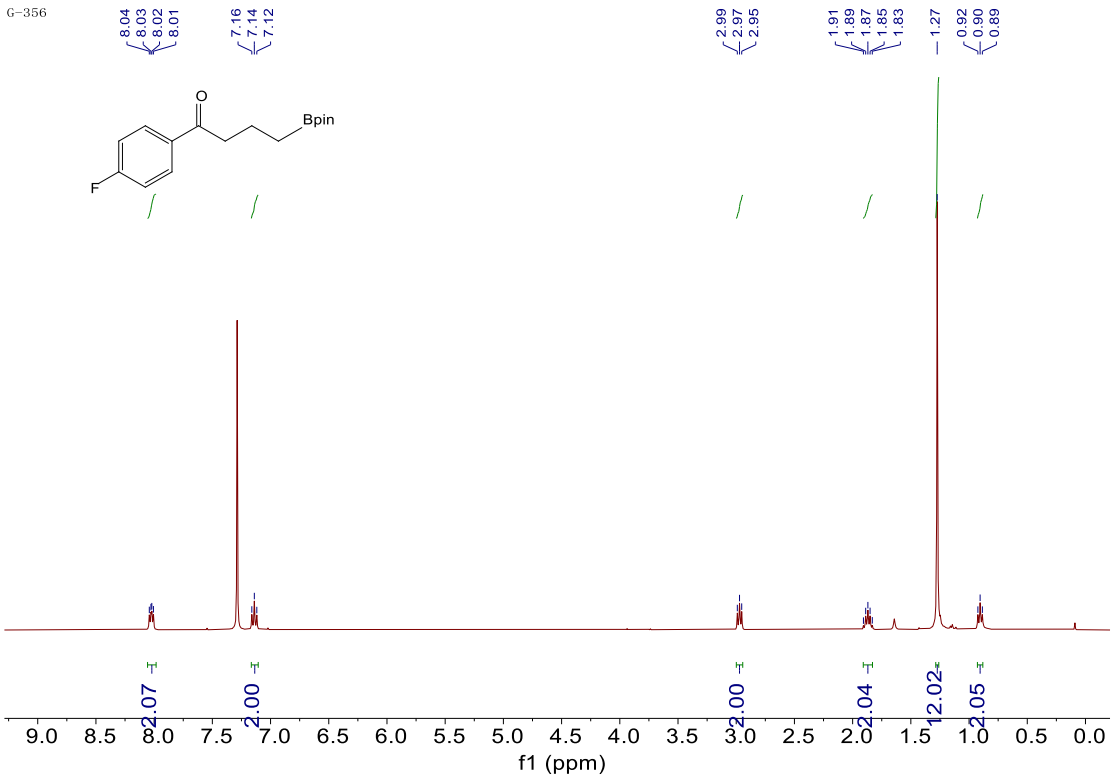
^{13}C NMR of compound **64**



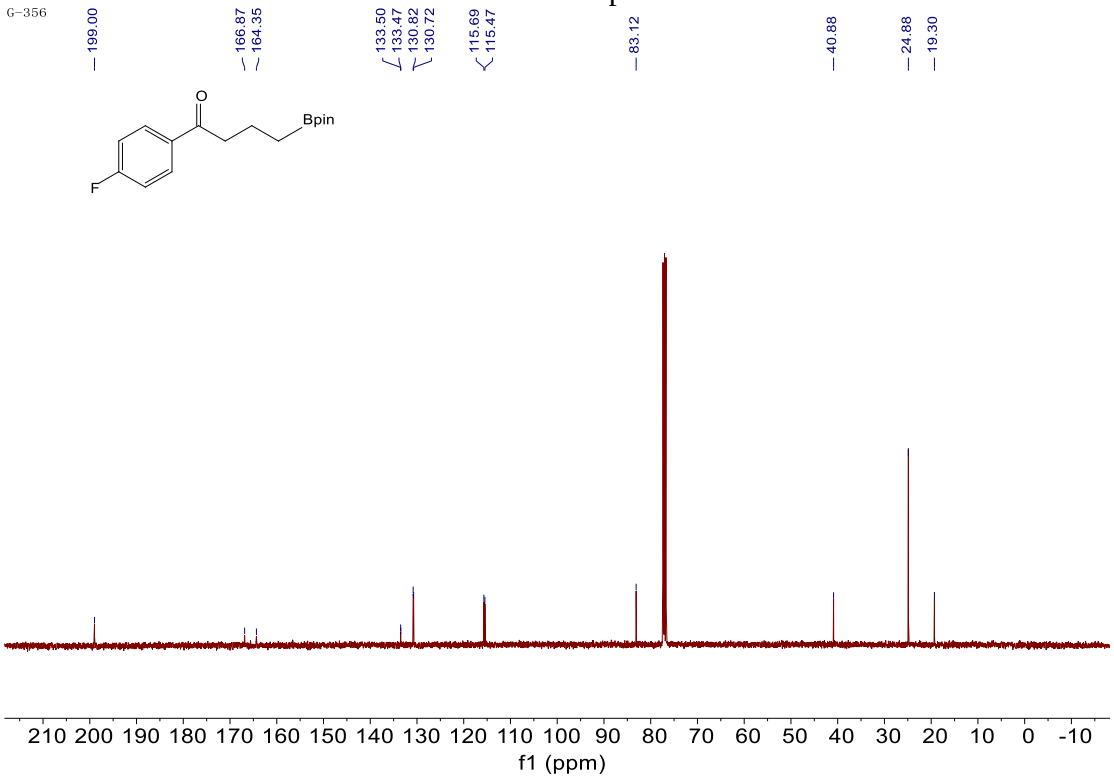
^{11}B NMR of compound **64**



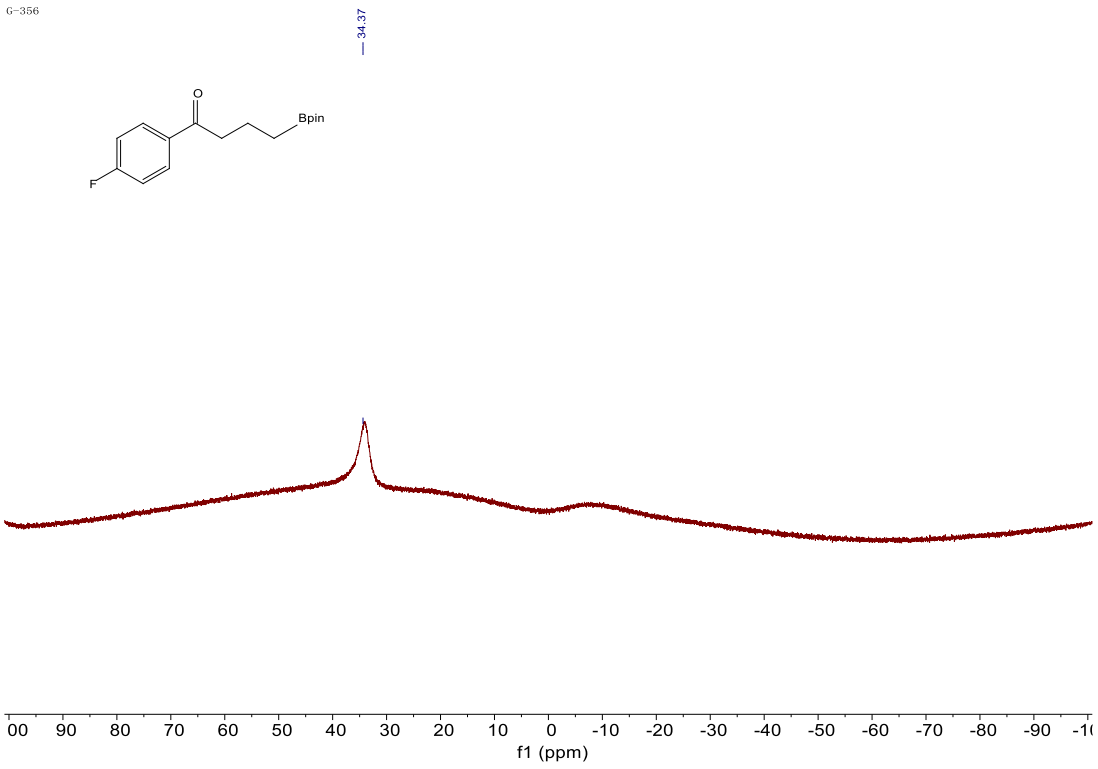
^{1}H NMR of compound **65**



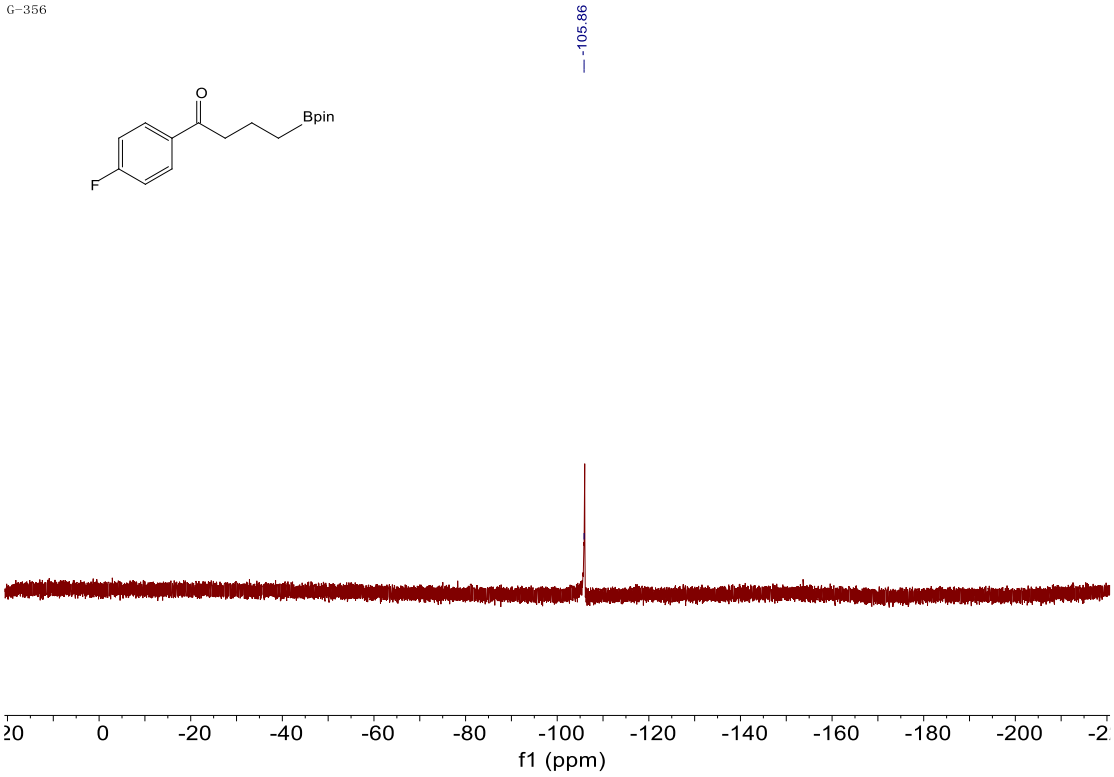
^{13}C NMR of compound **65**



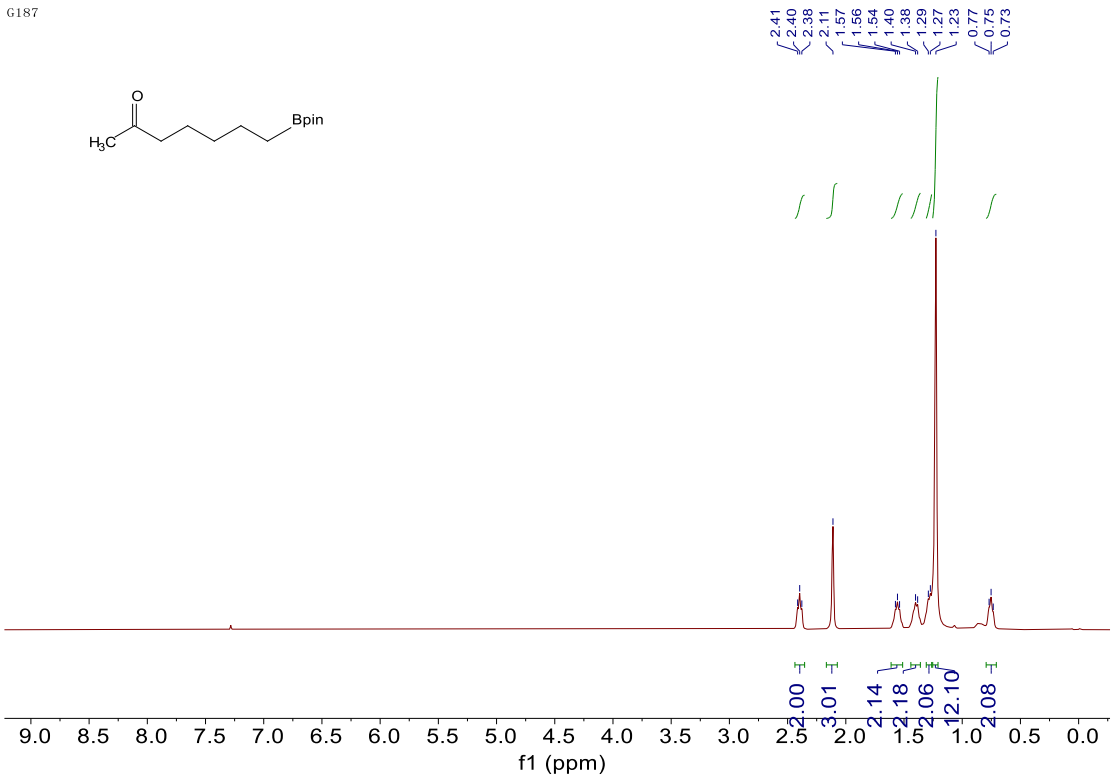
^{11}B NMR of compound **65**



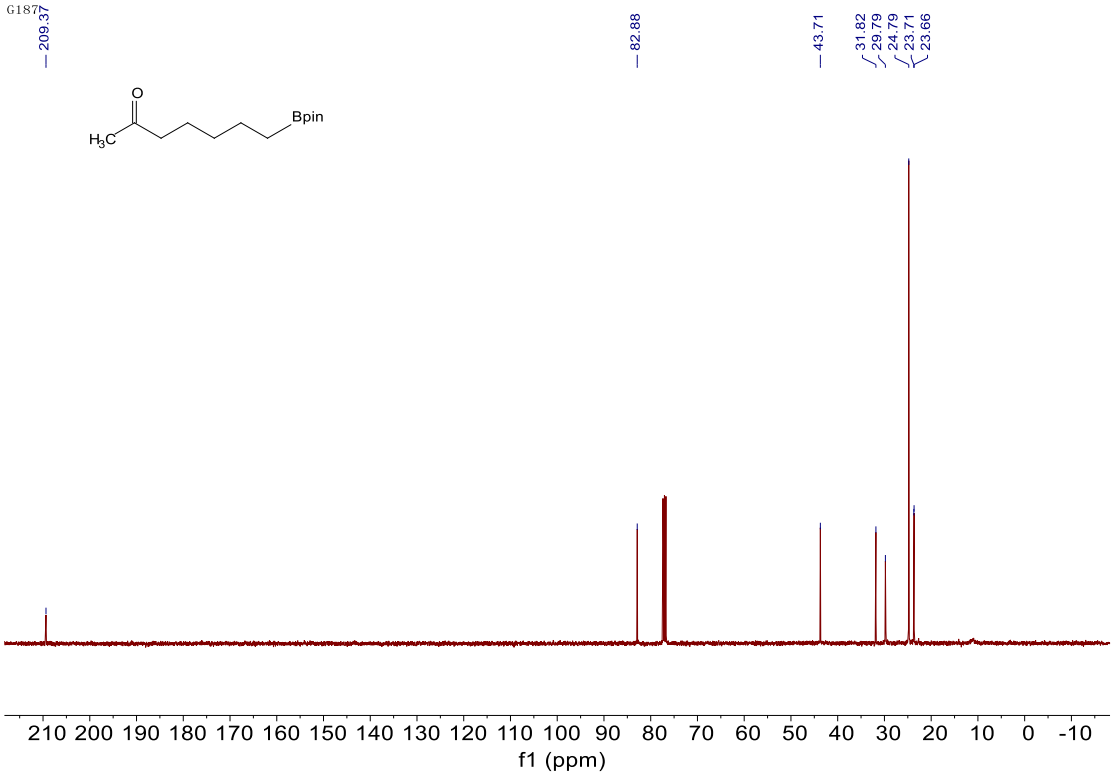
^{19}F NMR of compound **65**



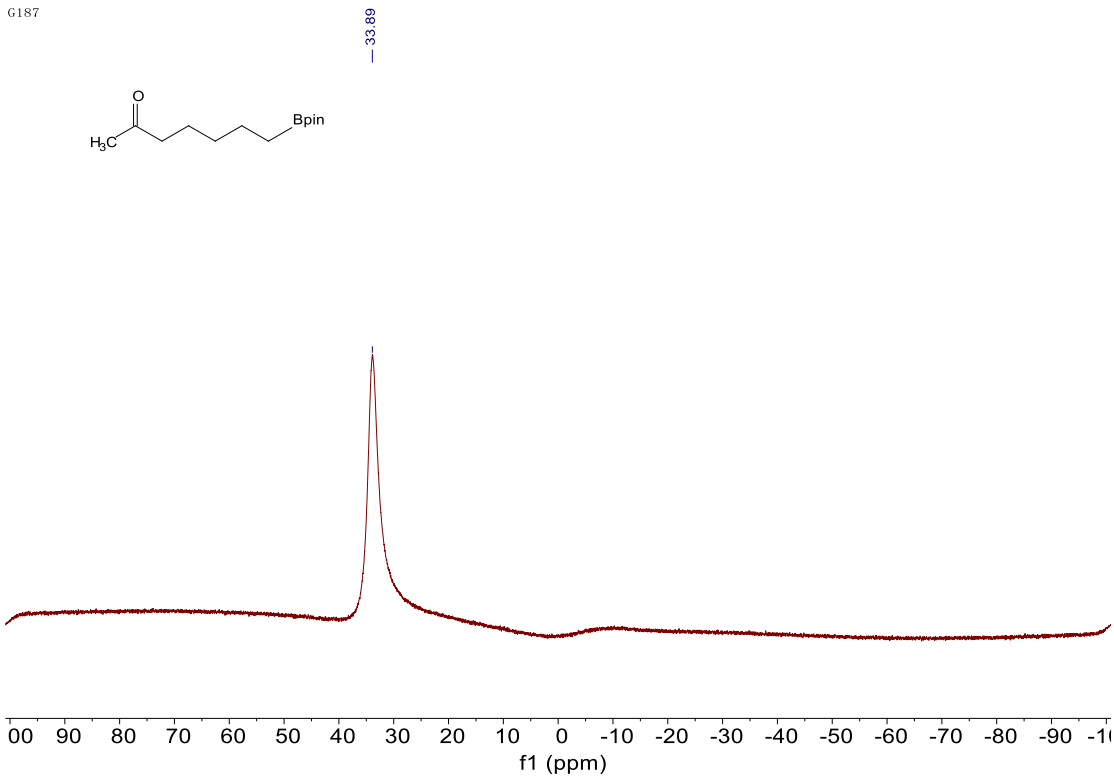
^{1}H NMR of compound **66**



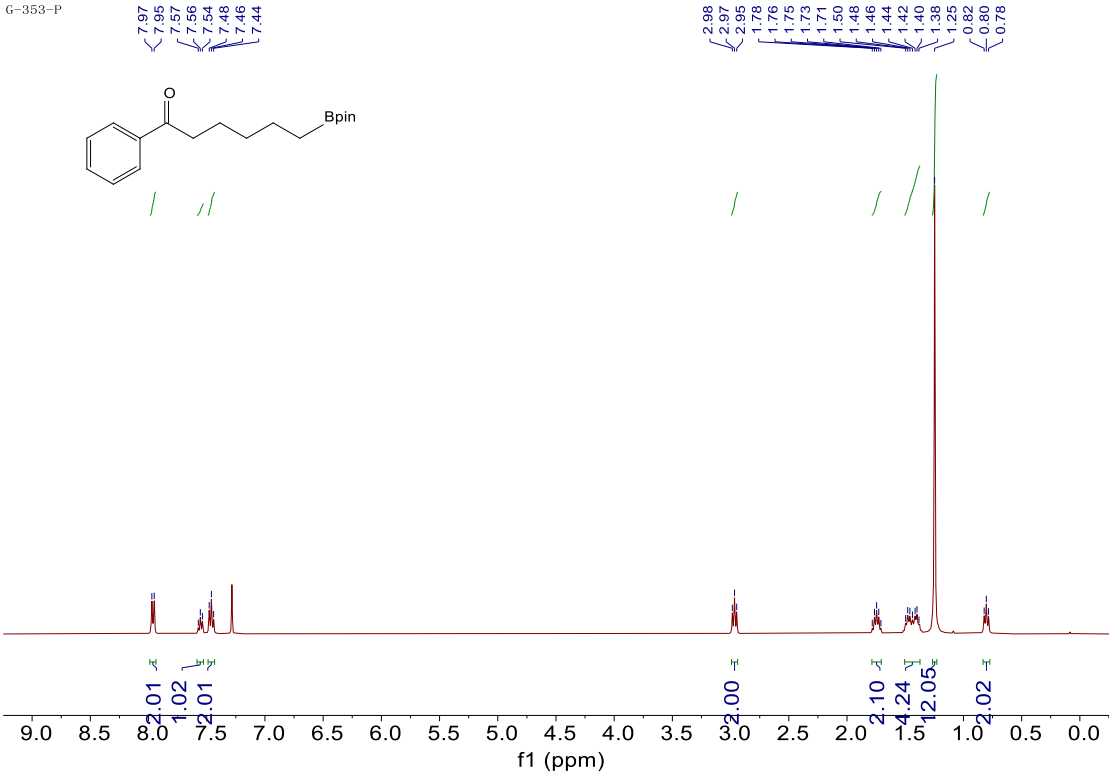
^{13}C NMR of compound **66**



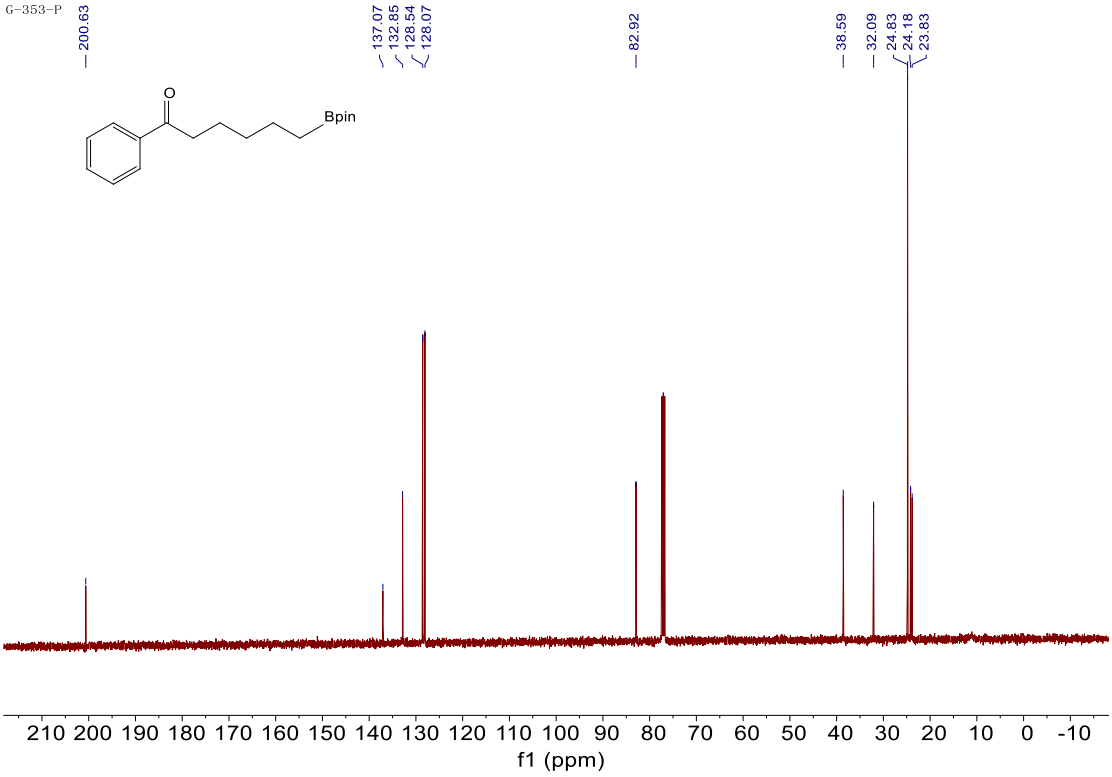
^{11}B NMR of compound **66**



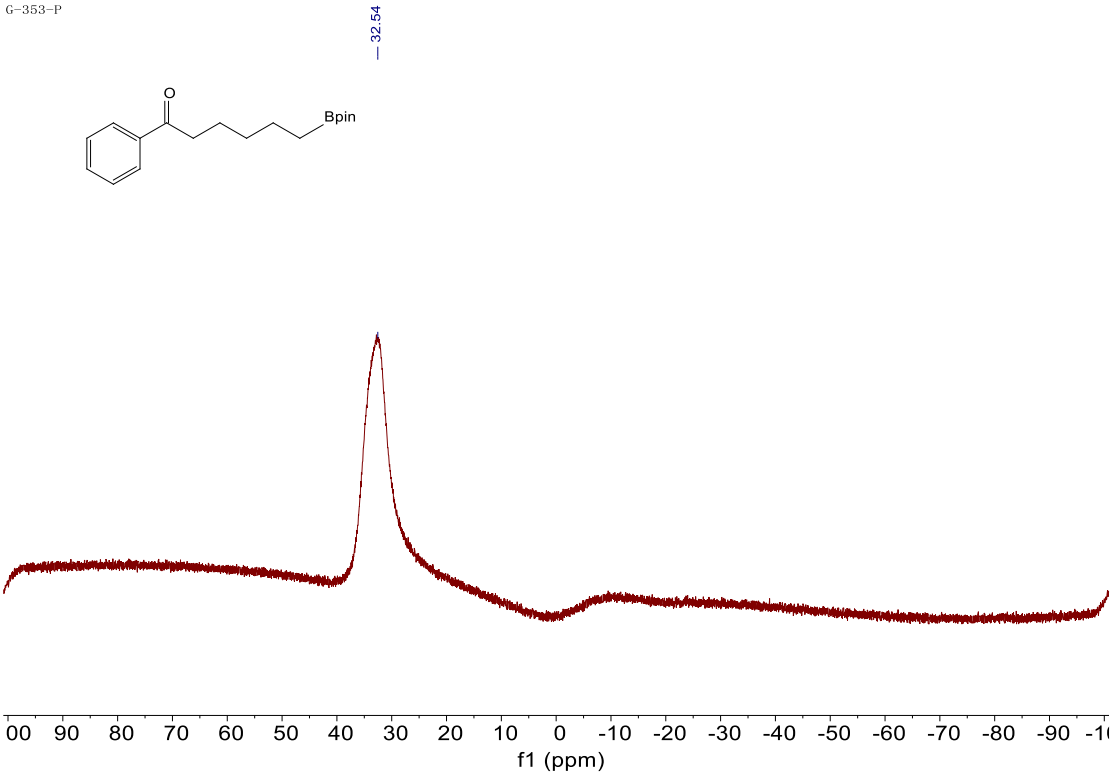
^{1}H NMR of compound **67**



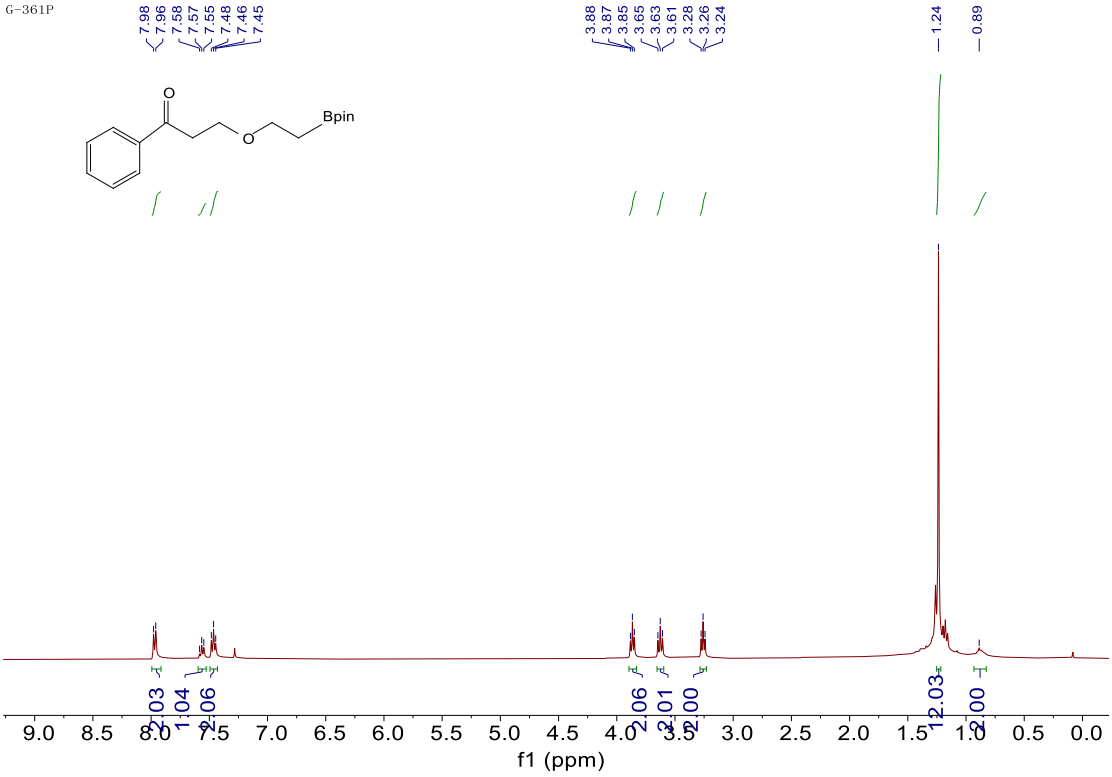
^{13}C NMR of compound **67**



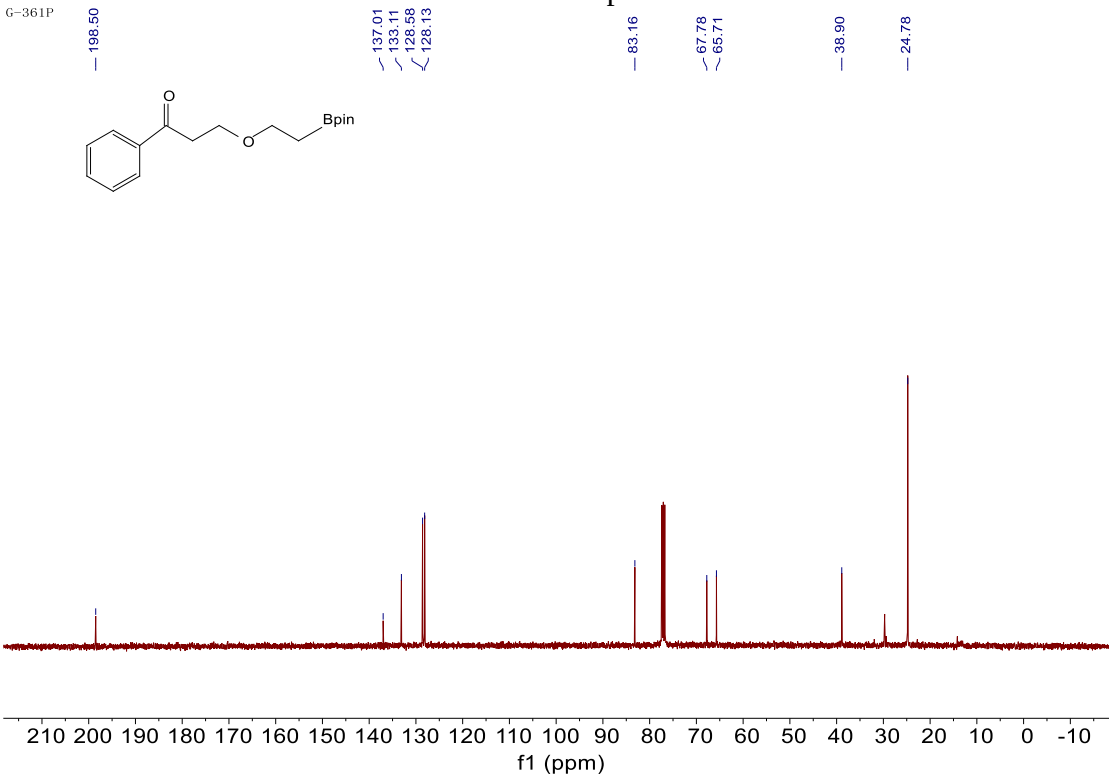
^{11}B NMR of compound **67**



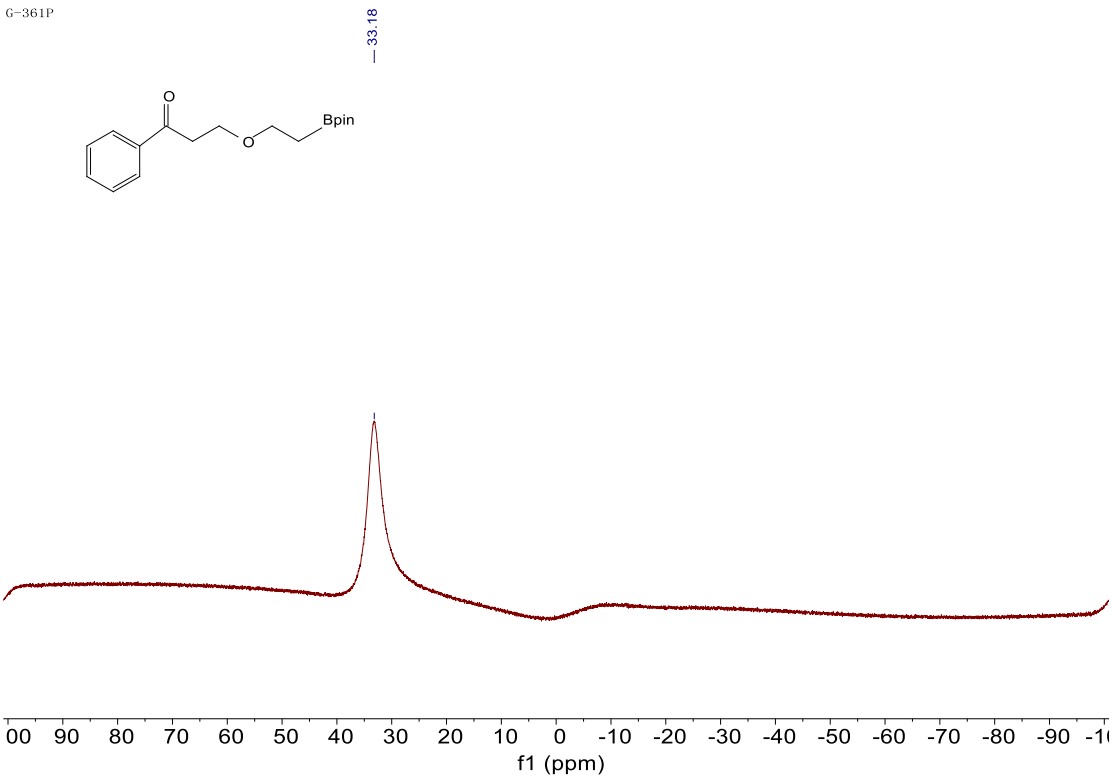
^{1}H NMR of compound **68**



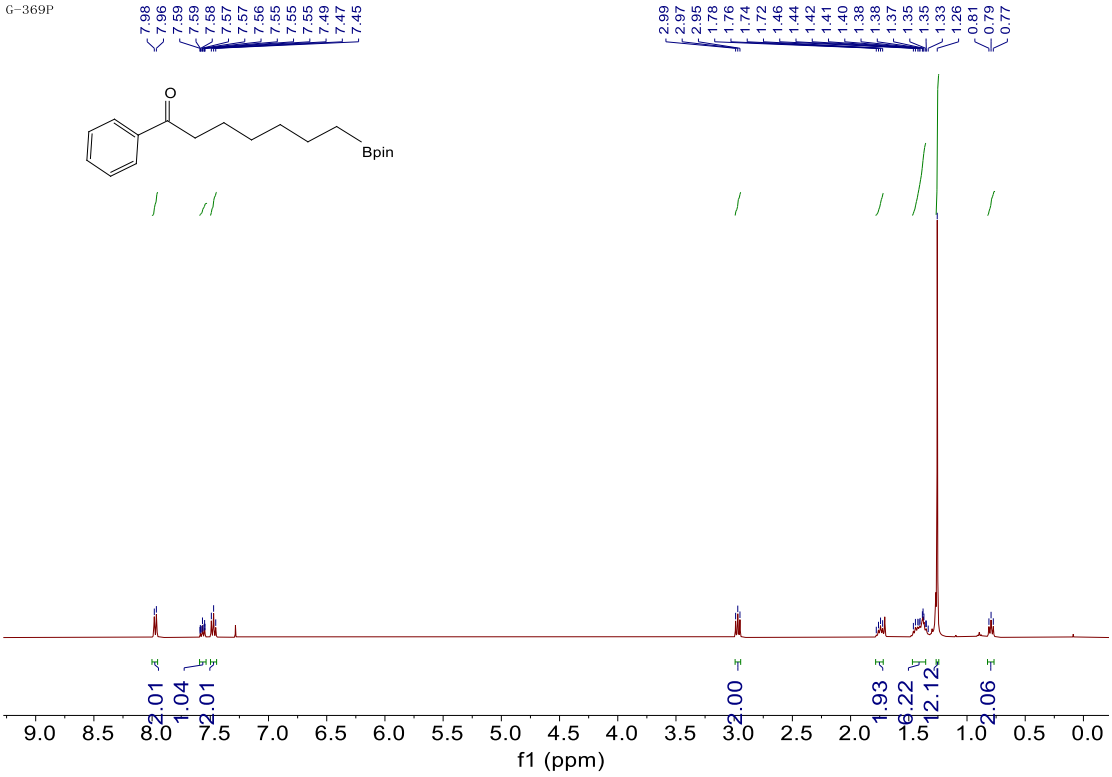
^{13}C NMR of compound **68**



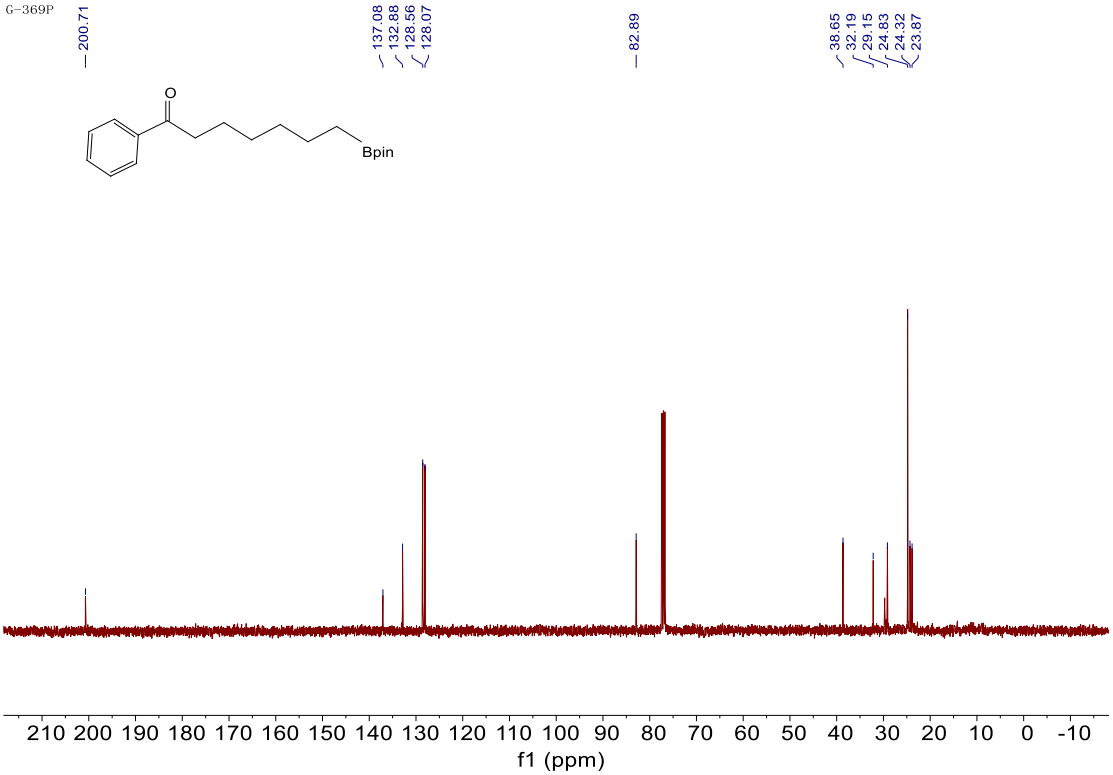
^{11}B NMR of compound **68**



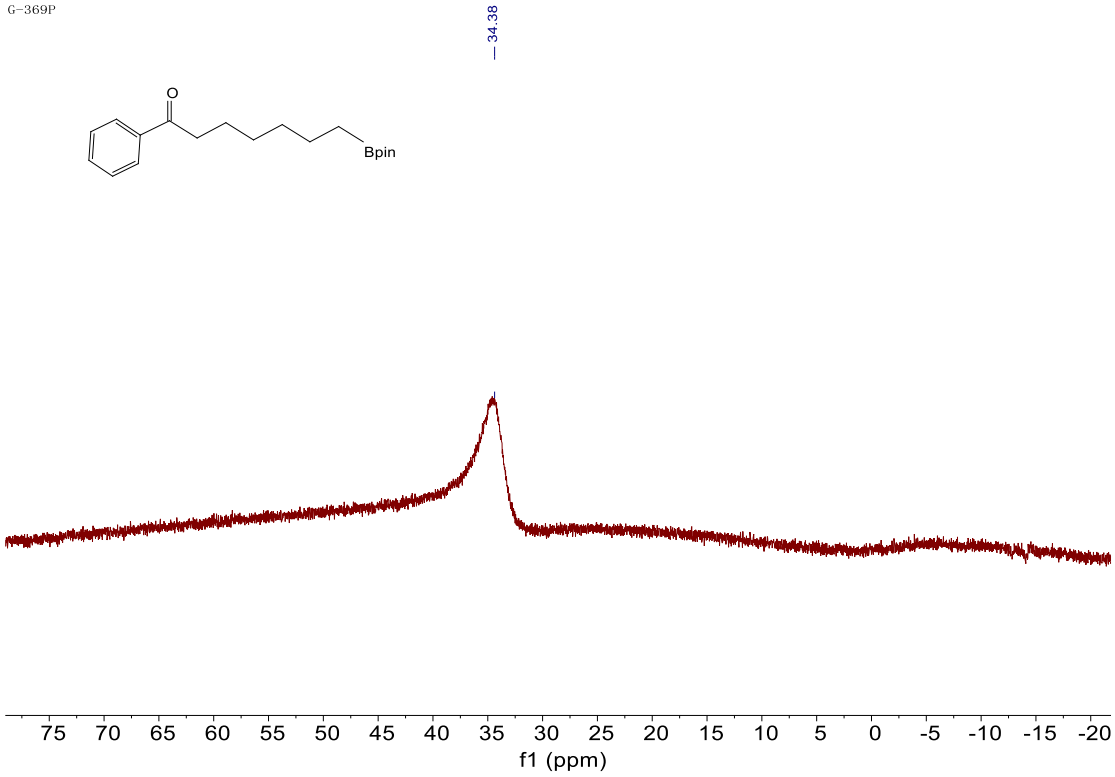
^{1}H NMR of compound **69**



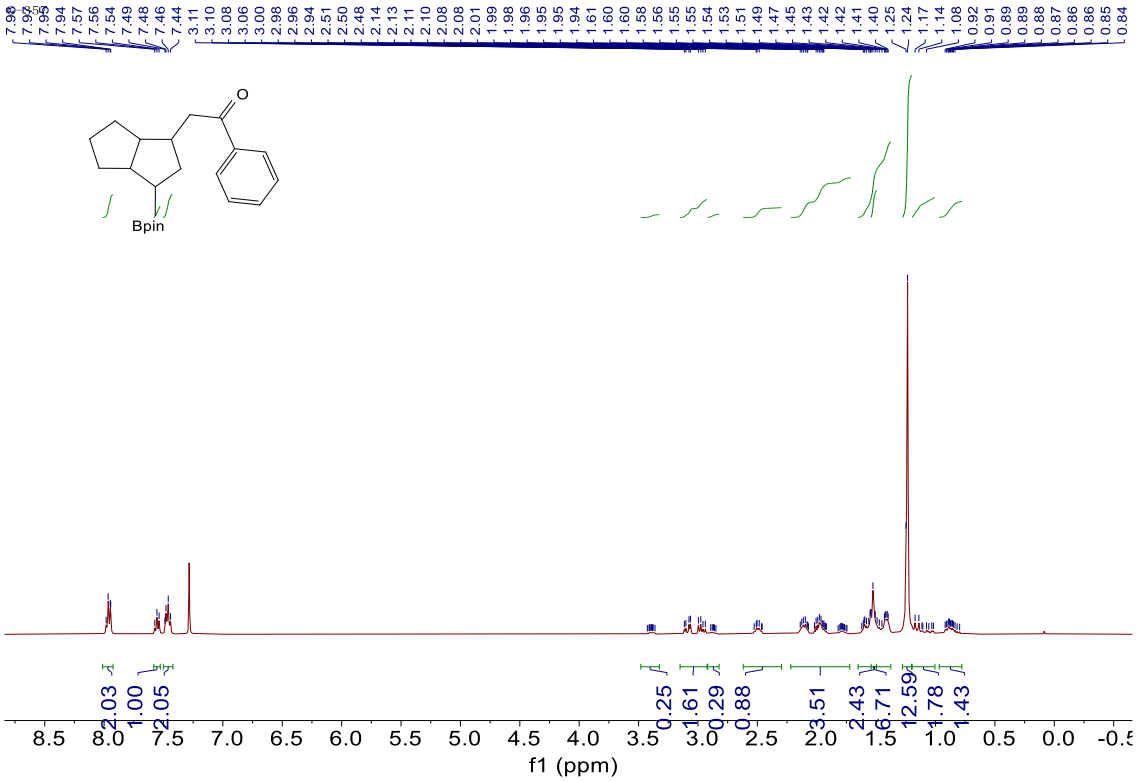
^{13}C NMR of compound **69**



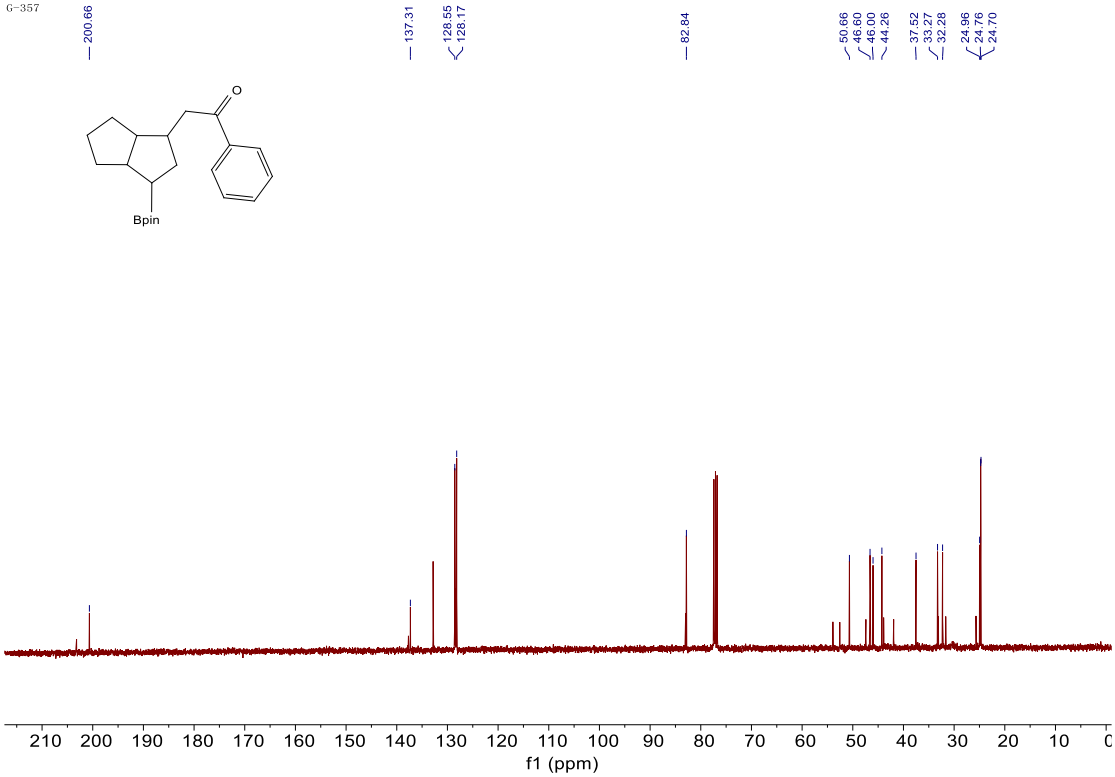
^{11}B NMR of compound **69**



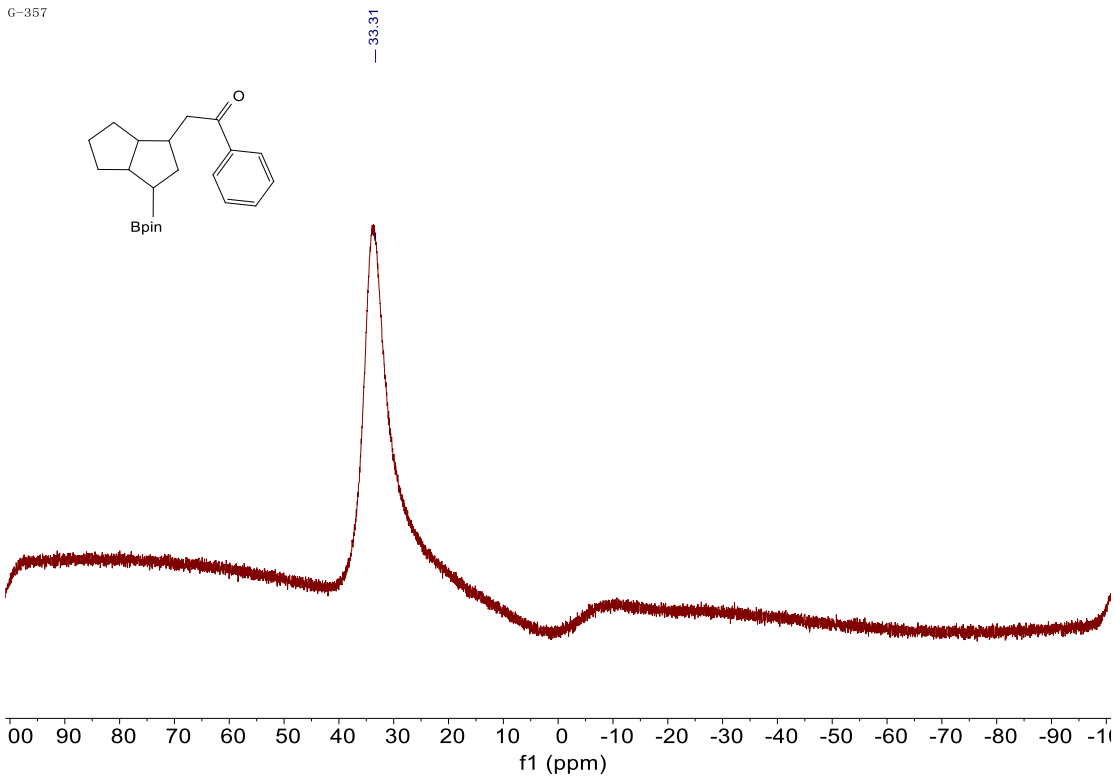
^{1}H NMR of compound **70**



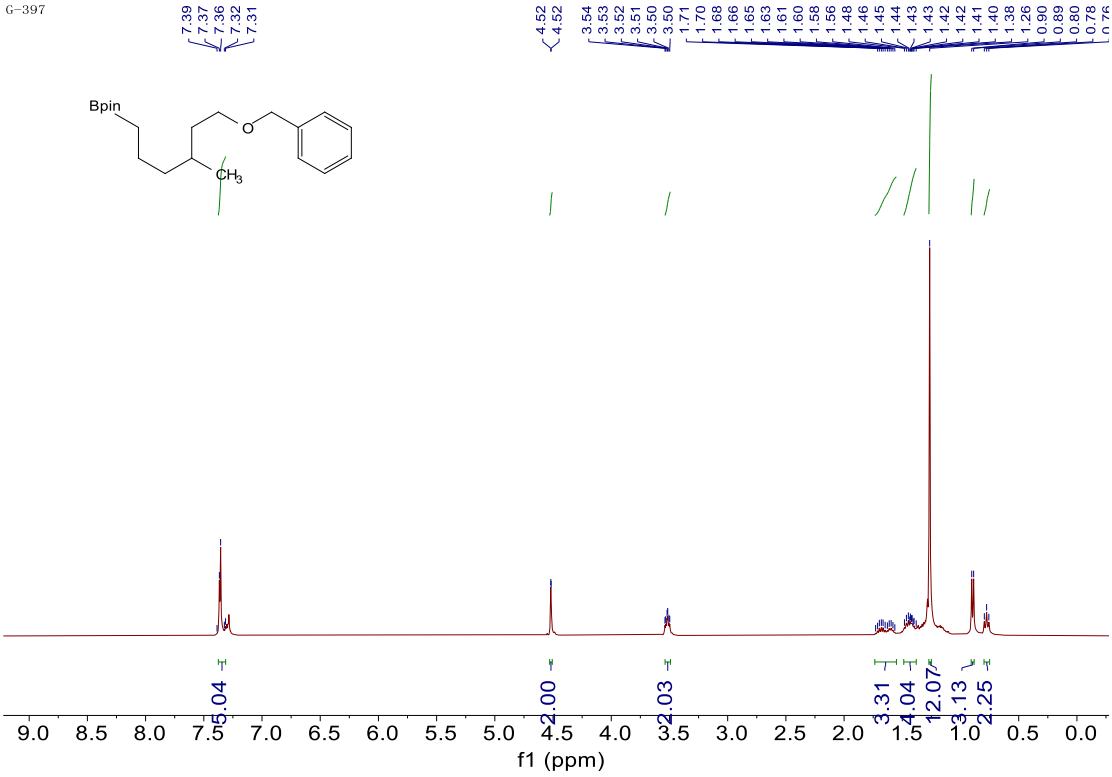
^{13}C NMR of compound **70**



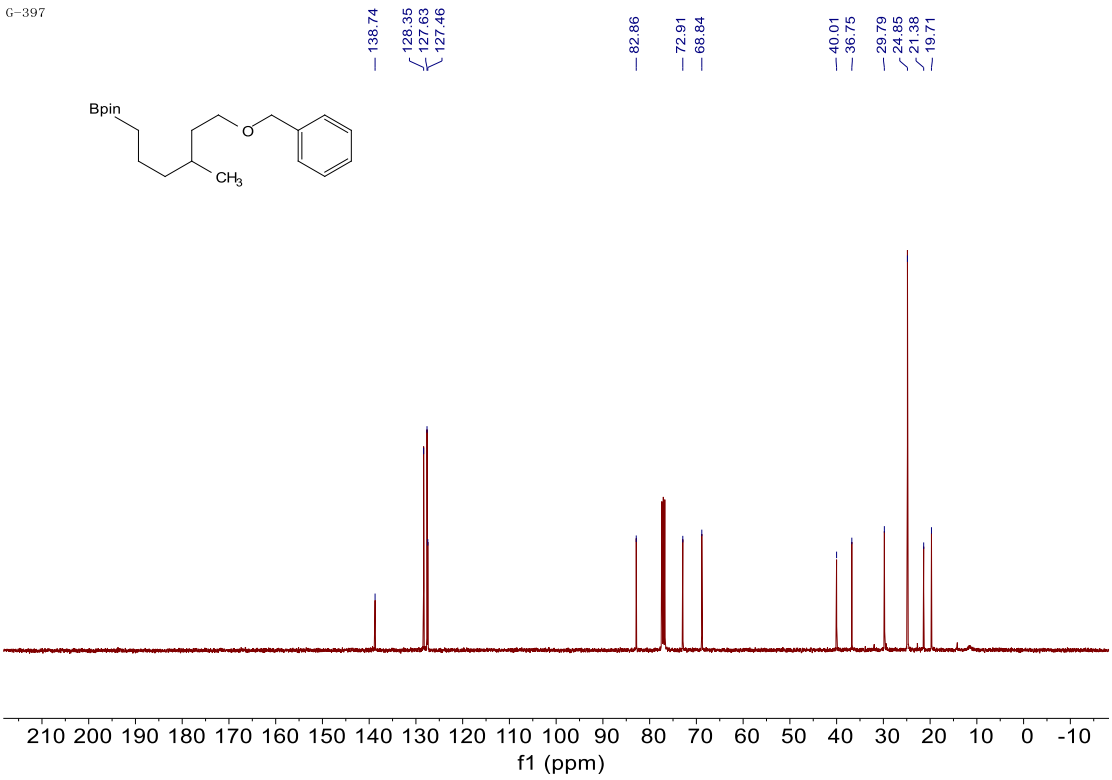
^{11}B NMR of compound **70**



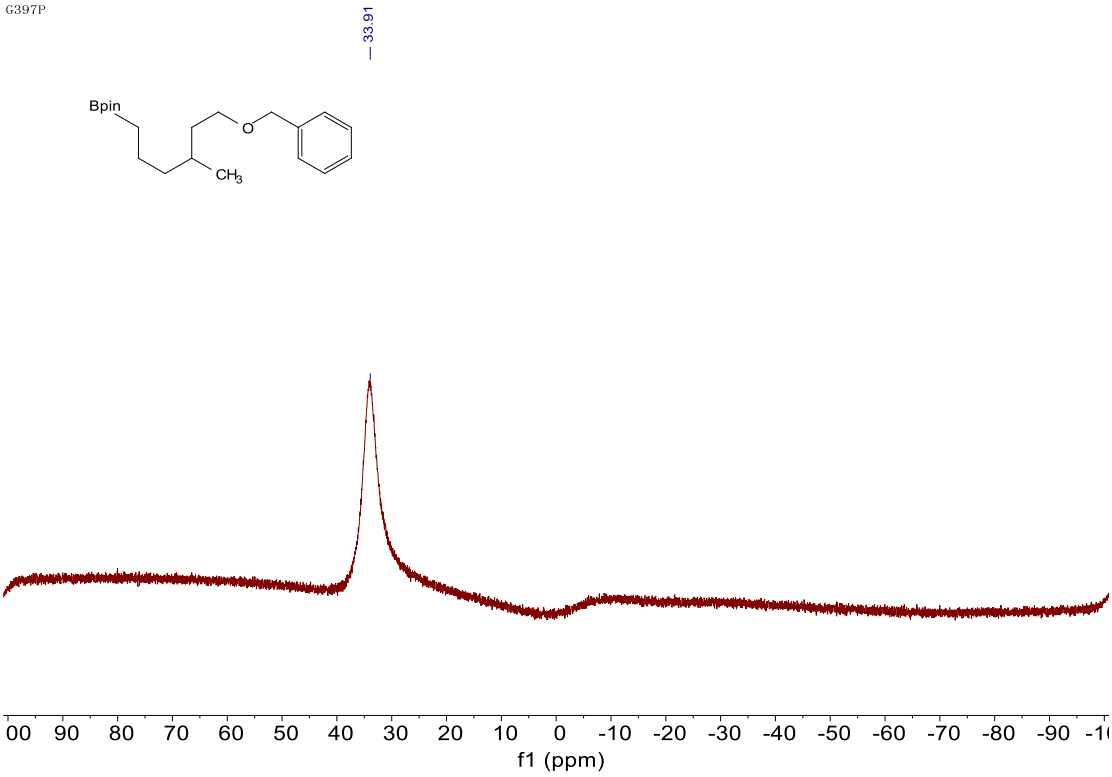
^{1}H NMR of compound **71**



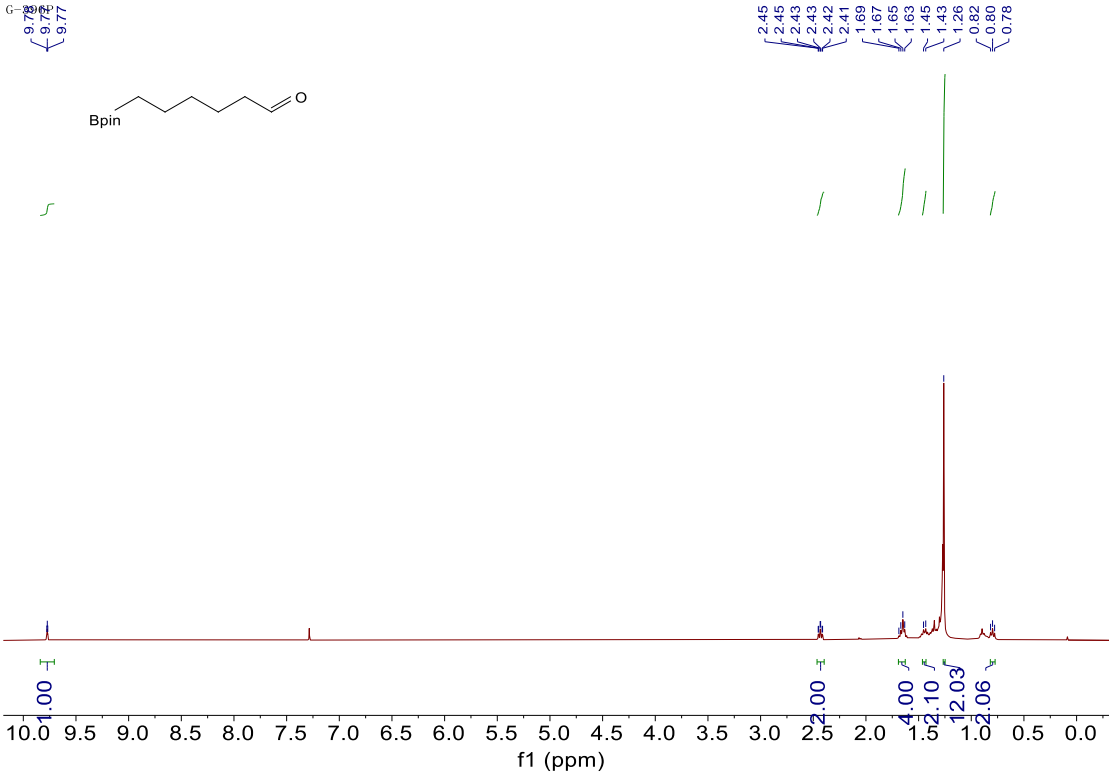
^{13}C NMR of compound **71**



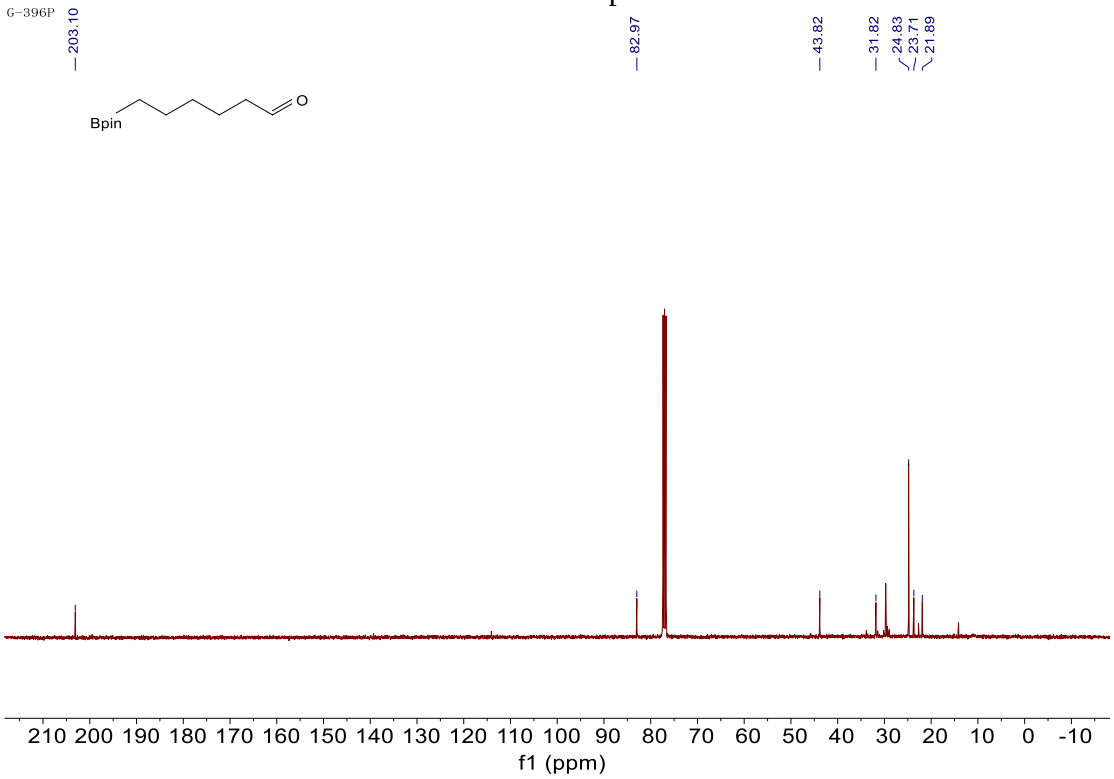
^{11}B NMR of compound **71**



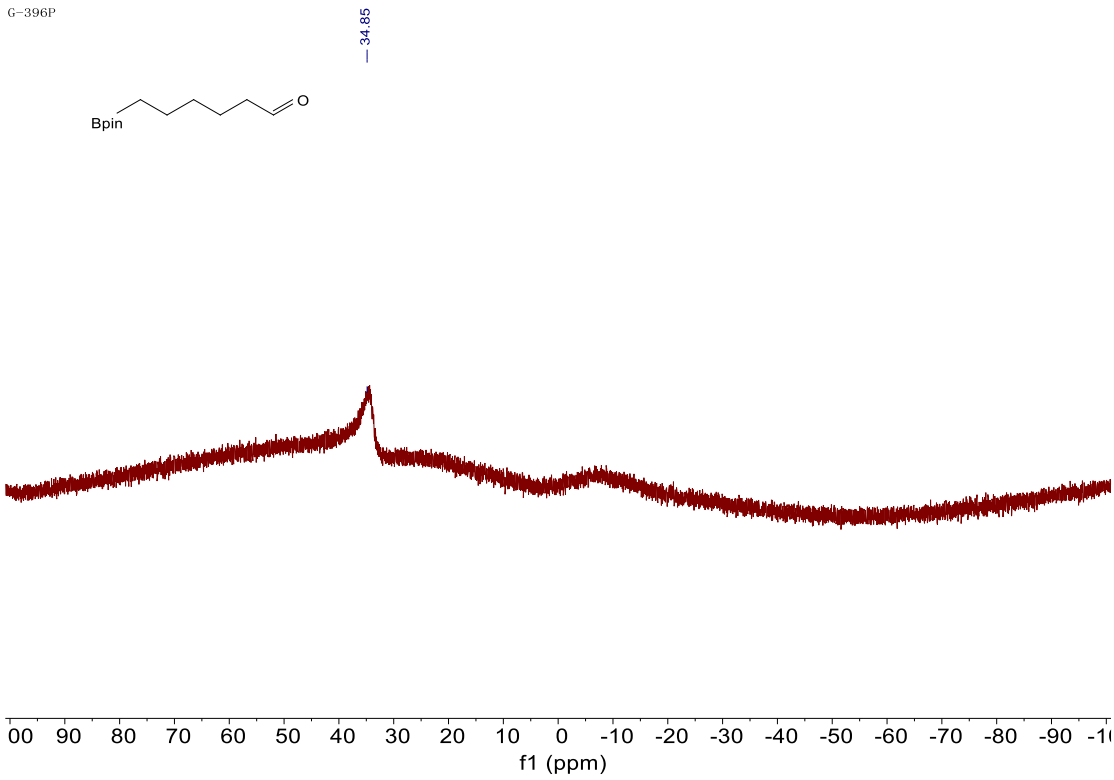
^{1}H NMR of compound **72**



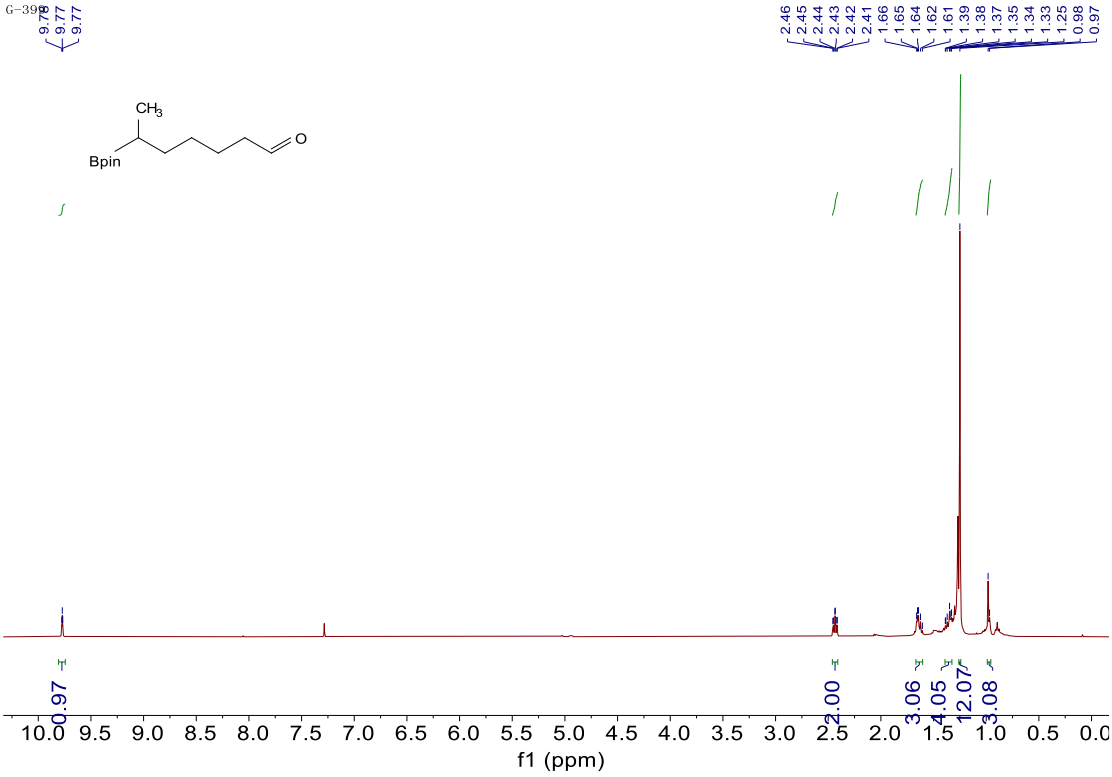
^{13}C NMR of compound **72**



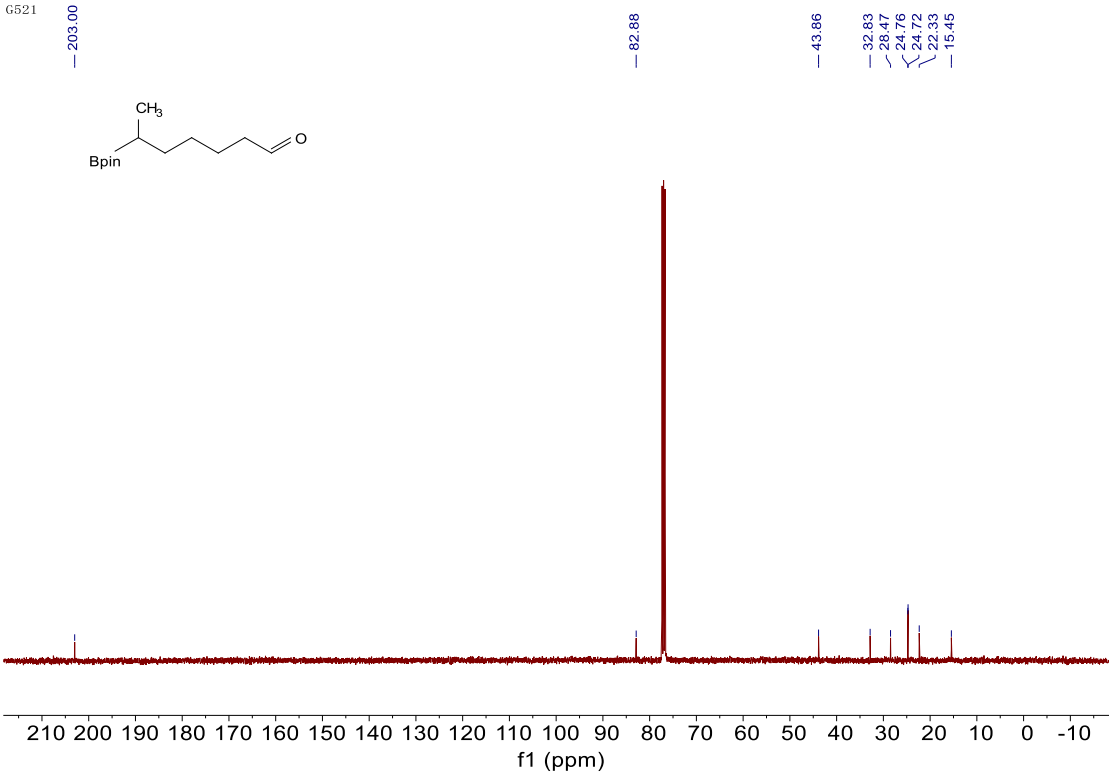
^{11}B NMR of compound **72**



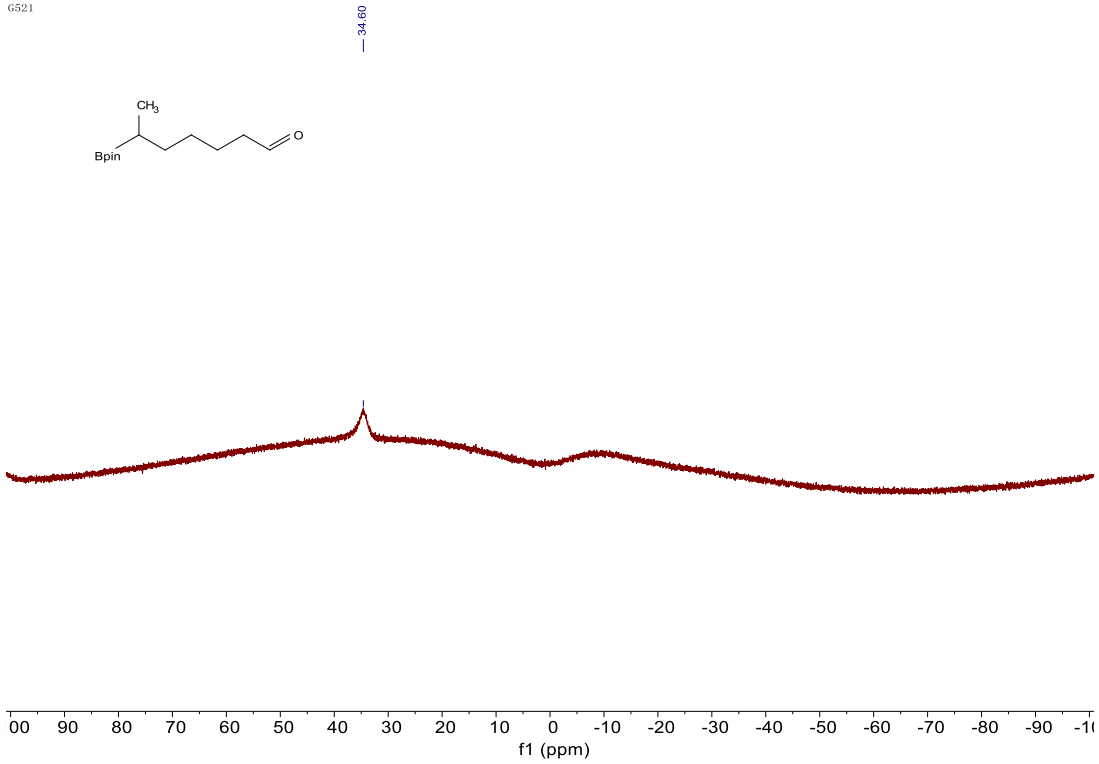
^{1}H NMR of compound **73**



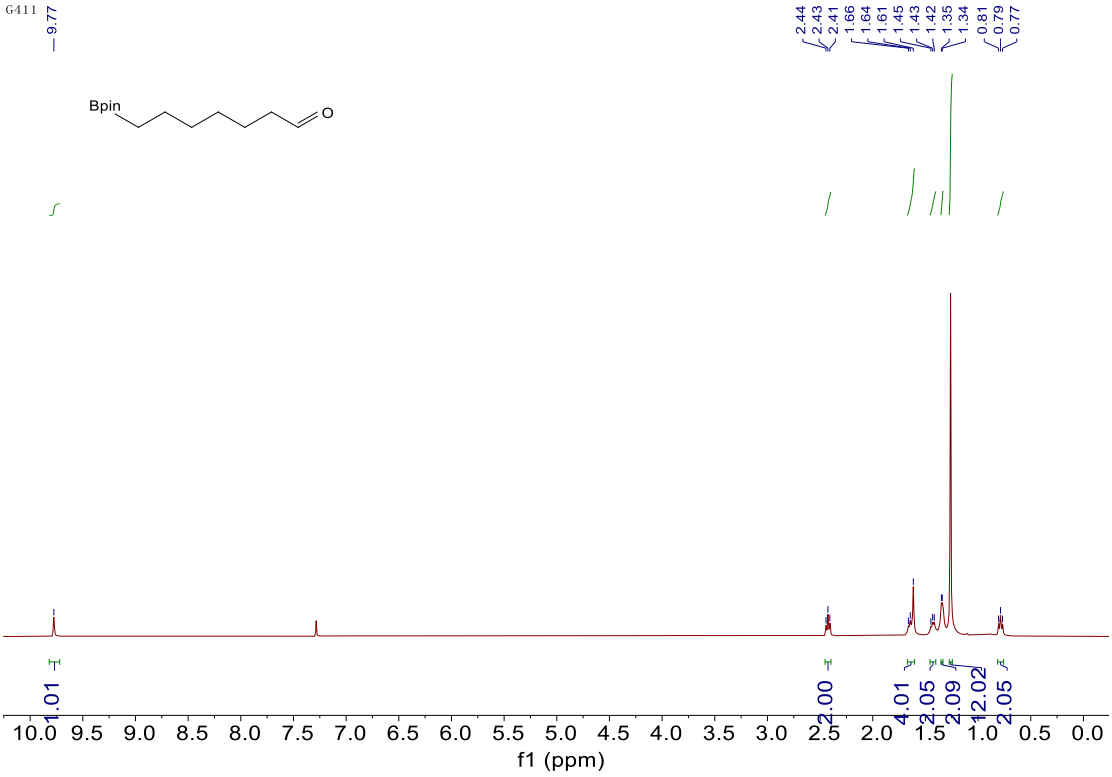
^{13}C NMR of compound **73**



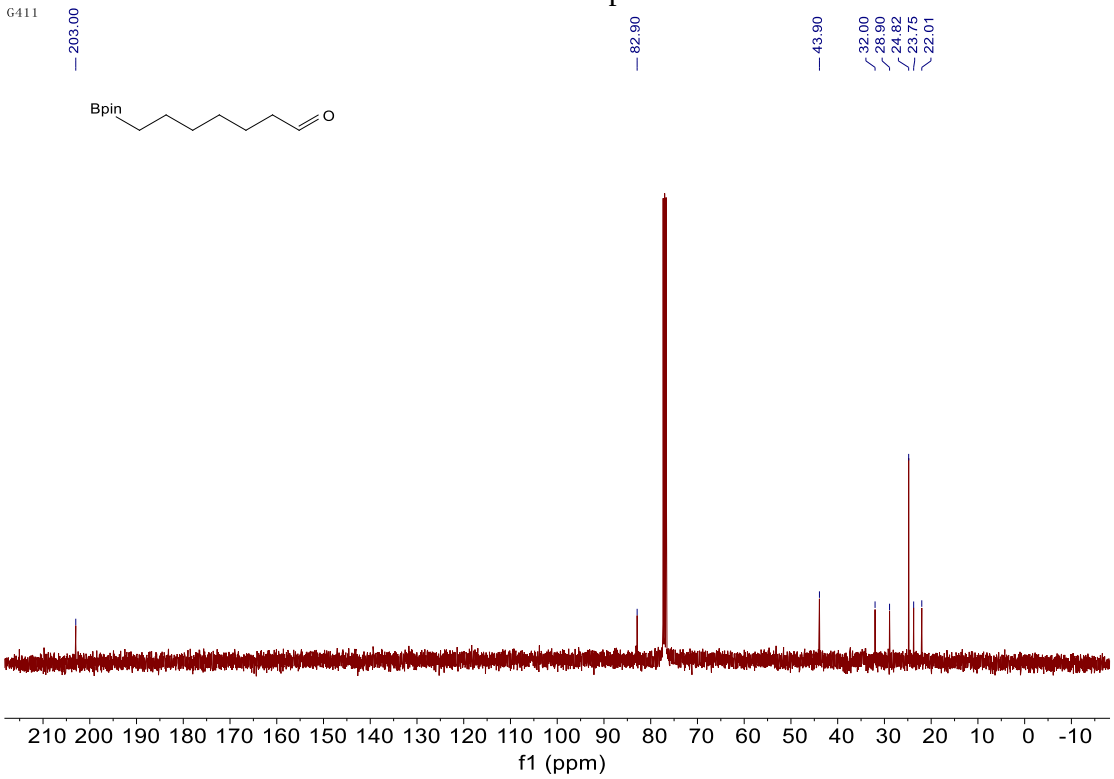
^{11}B NMR of compound **73**



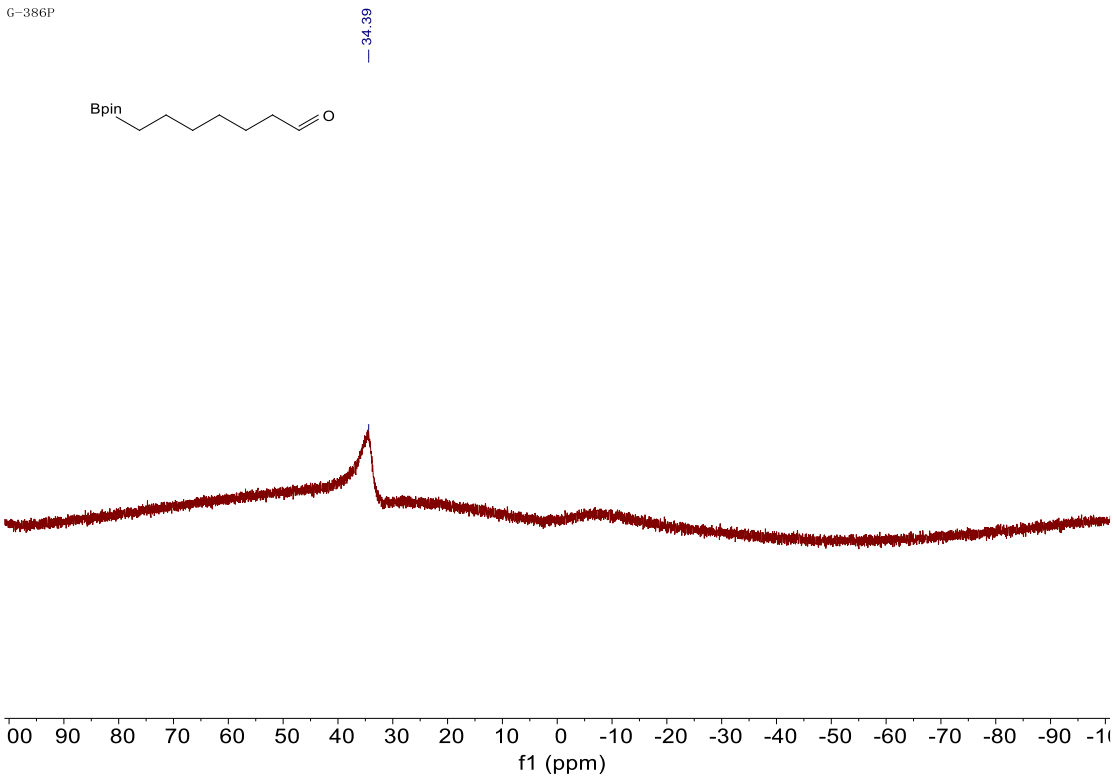
^{1}H NMR of compound **74**



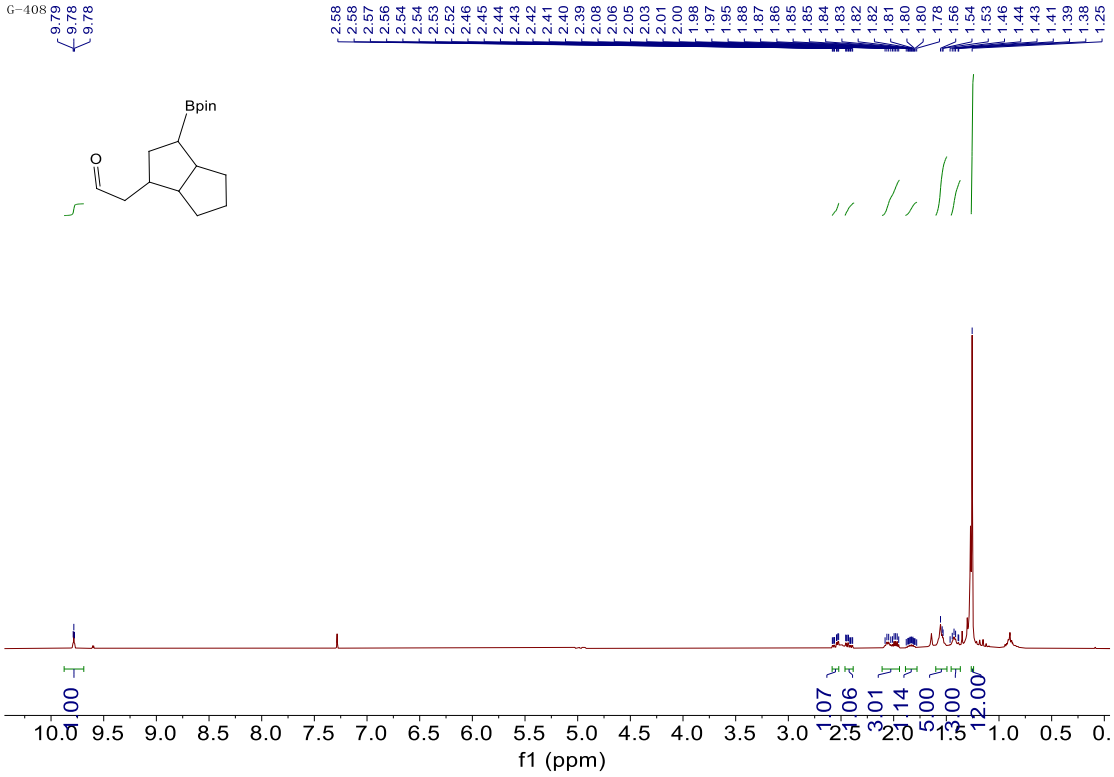
^{13}C NMR of compound **74**



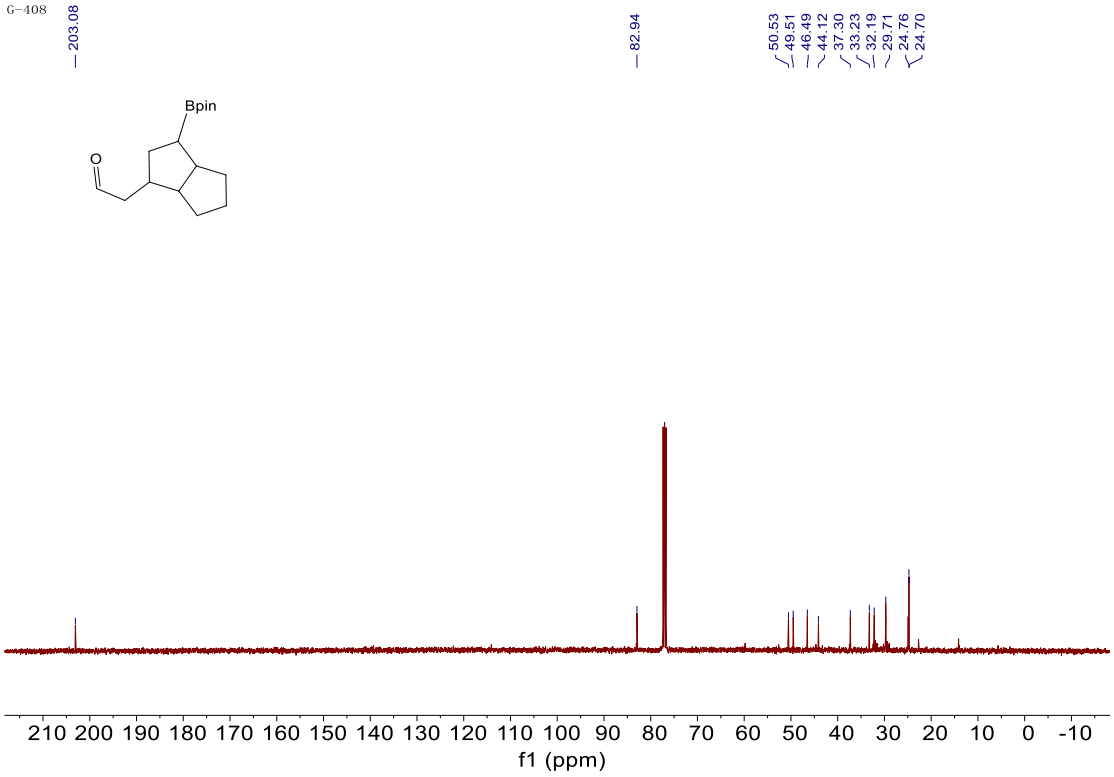
^{11}B NMR of compound **74**



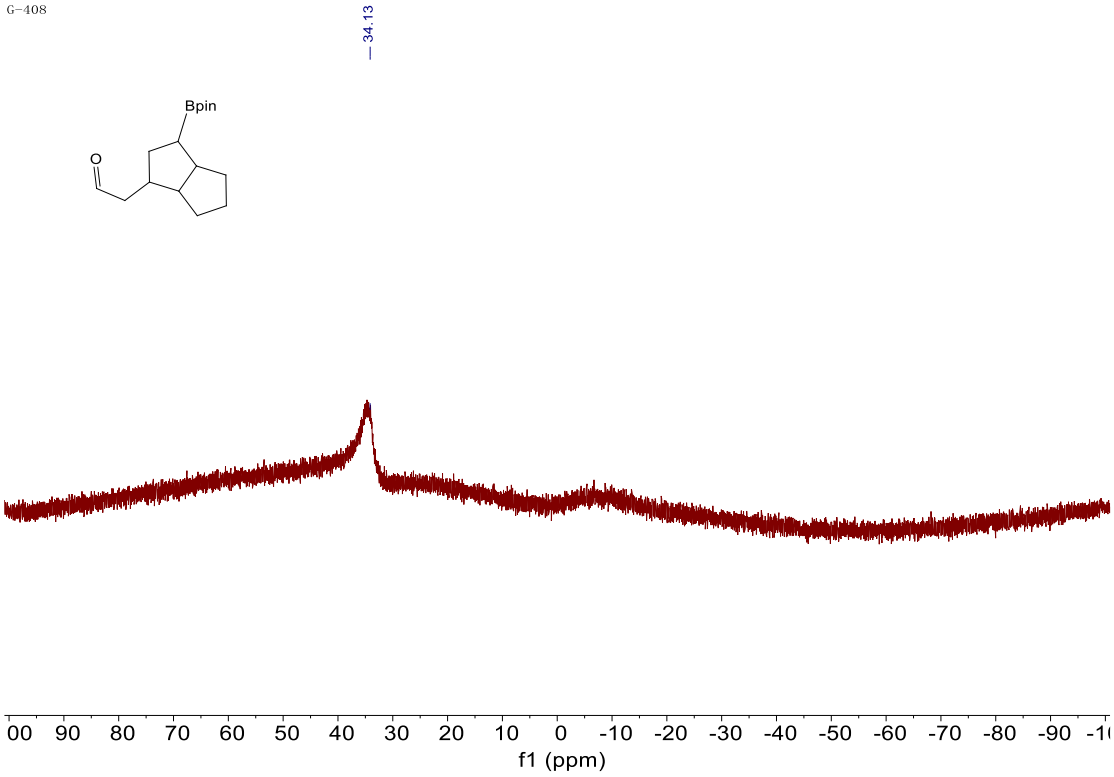
^{1}H NMR of compound **75**



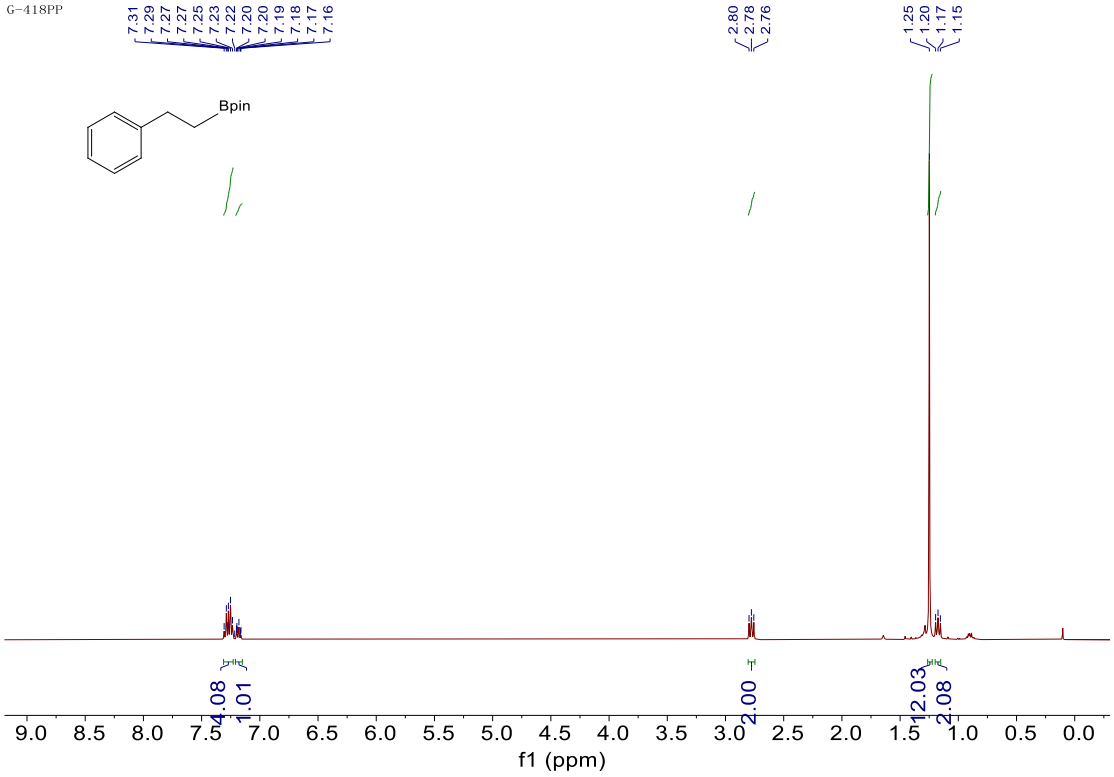
^{13}C NMR of compound **75**



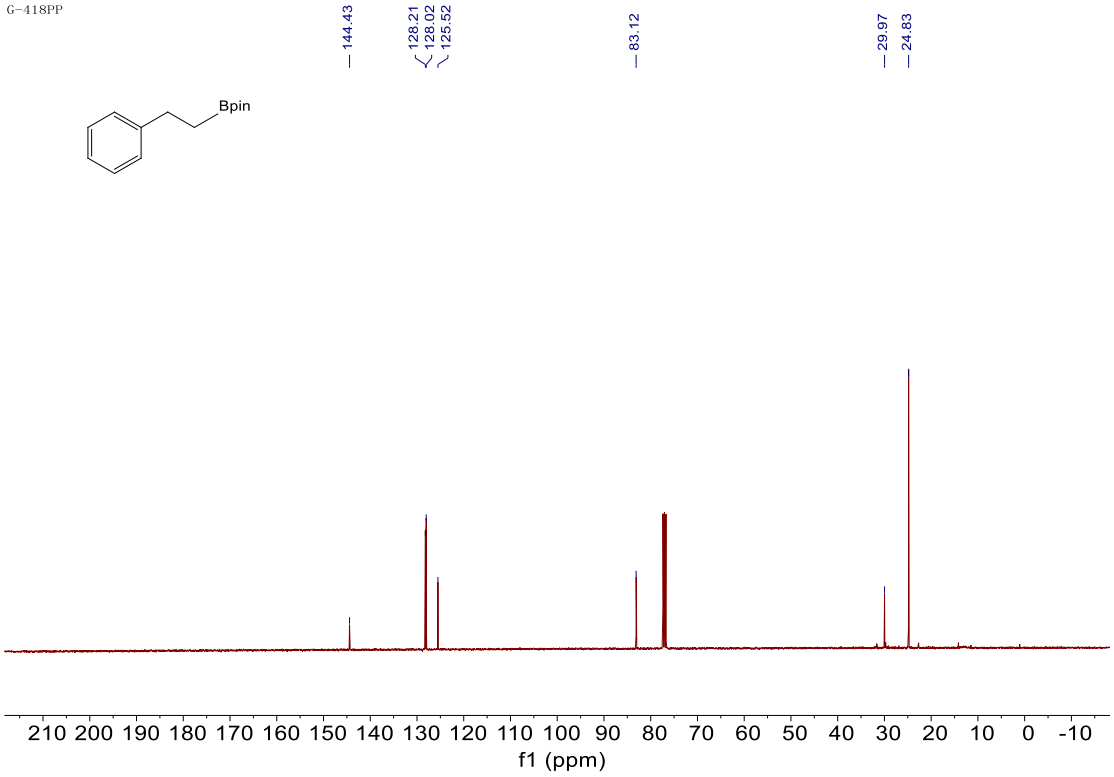
^{11}B NMR of compound **75**



^{1}H NMR of compound **76**



^{13}C NMR of compound **76**



^{11}B NMR of compound **76**