Synthesis of N-alkoxyphthalimide Derivatives via PIDA-promoted Dehydrogenative Coupling Reaction

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Supporting Information

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1. General information

All manipulations were carried out under air atmosphere. Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. The ^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz) and ^{19}F NMR (376 MHz) data were recorded on a Bruker DPX-400 spectrometer with $CDCl_3$ as solvent at room temperature unless specified otherwise. The chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. ^{1}H NMR spectra was recorded with tetramethylsilane (δ= 0.00 ppm) as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ = 77.00 ppm) as internal reference. HRMS were performed on Agilent ESI-quadrupole.

2. General procedures for reactions

2.1 General procedure of synthesis of α-hydroxyphthalimide ketones

A solution of aryl ketones **1** or **4** (0.3 mmol), N-hydroxyphthalimide **2a** (1.2 equiv, 0.36 mmol), iodobenzene diacetate (1.2 equiv, 0.36 mmol) and dichloromethane (2 mL) was stirred in a 10 mL sealed tube at room temperature under air for 4 h. After completion of the reaction, 5 mL of water was added and extracted by dichloromethane (3 × 5 mL). The combined organic layer was washed with brine (5 mL) and then dried over anhydrous Na_2SO_4 and evaporated in vacuum. The desired products were obtained in the corresponding yields after purification by column chromatography on silica gel eluting with petroleum ether / ethyl acetate.

2.2 General procedure of synthesis of gram scale of 3a

A solution of 1,2-diphenylethanone **1a** (5.0 mmol), N-hydroxyphthalimide **2a** (1.2 equiv, 6.0 mmol), and iodobenzene diacetate (1.2 equiv, 6.0 mmol) and dichloromethane (20 mL) was stirred in a 50 mL sealed tube at room temperature under air for 4 h. After completion of the reaction, 50 mL of water was added and extracted by dichloromethane (3 × 50 mL). The combined organic layer was washed with brine and then dried over anhydrous Na_2SO_4 and evaporated in vacuum. The desired product **3a** were prepared according to the general procedure and purified by column chromatography on silica gel eluting with petroleum ether / ethyl acetate (3:1) as white solid 1.25g (70%).

2.3 Synthetic transformations of the product 3a: oxidation to ester 6

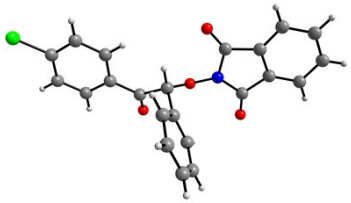
A solution of **3a** (0.3 mmol), sodium bicarbonate (0.39 mmol, 33 mg), *m*-CPBA (0.45 mmol, 78 mg) and dichloromethane (2 mL) was stirred in a 10 mL sealed tube at 0 °C, then the resultant mixture was stirred at room temperature for 16 h. The progress of the reaction was monitored by

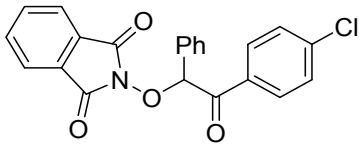
TLC. After completion, the solvent was evaporated on a rotary evaporator and the residue was treated with sodium bicarbonate solution (5 mL). The mixture was extracted with ethyl acetate (3 × 15 mL) and washed with brine (1 × 10 mL) and water (1 ×10 mL). Then dried over anhydrous Na_2SO_4 and evaporation of the solvent gave a residue that was purified on silica gel column chromatography using petroleum ether / ethyl acetate (4:1) as the eluent to obtain the desired product **6** as white solid 95.1 mg (85%).

2.4 Synthetic transformations of the product 3a: formation of 1,2-diketone 7

A solution of **3a** (0.3 mmol) and triethylamine (2 mL) was stirred in a 10 mL sealed tube equipped with a reflux condenser. The reaction mixture was heated to 90 °C for 16 h under air. The reaction mixture was cooled and washed with 1M HCl (2 × 15 mL) and extracted with CH_2Cl_2, dried over $MgSO_4$, filtered, and concentrated. The residue was purified on silica gel column chromatography using petroleum ether / ethyl acetate (20:1) as the eluent to obtain the desired product **7** as yellow solid 59.0 mg (93%).

3. X-ray crystal structure of 3f





3f (CCDC 2049393)

Table S1. Crystal data and structure refinement for **3f**.

Empirical formula	$C_{22}H_{14}ClNO_4$
Formula weight	391.79
Temperature	296(2) K
Wavelength	71.073 pm
Crystal system	Monoclinic
Space group	P 21/c
Unit cell dimensions	a = 2041.5(3) pm α= 90°
	b = 598.06(8) pm β= 105.234(2)°
	c = 1539.0(2) pm γ = 90°
Volume	1.8130(4) nm^3
Z	4
Density (calculated)	1.435 Mg/m^3
Absorption coefficient	0.240 mm^{-1}
F(000)	808
Crystal size	0.260 x 0.220 x 0.210 mm^3
Theta range for data collection	2.743 to 25.999°.
Index ranges	-18<=h<=24, -6<=k<=7, -18<=l<=18
Reflections collected	9452
Independent reflections	3540 [R(int) = 0.0243]
Completeness to theta = 25.242°	99.5 %
Data / restraints / parameters	3540 / 0 / 253
Goodness-of-fit on F^2	1.022
Final R indices [I>2sigma(I)]	R1 = 0.0385, wR2 = 0.0906

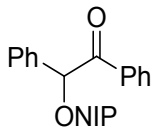
R indices (all data)	R1 = 0.0598, wR2 = 0.1031
Largest diff. peak and hole	0.148 and -0.319 e.Å^{-3}

Table S2. Bond lengths [pm] and angles [°] for **3f**.

Cl(1)-C(20)	173.43(18)	C(19)-H(19)	93.00	O(1)-C(8)-N(1)	125.16(16)
O(3)-N(1)	138.49(18)	C(13)-C(14)	93.00	O(1)-C(8)-C(6)	130.82(17)
O(3)-C(9)	144.7(2)	C(13)-H(13)	138.7(3)	N(1)-C(8)-C(6)	104.00(14)
O(4)-C(16)	120.9(2)	C(4)-C(3)	93.00	C(1)-C(6)-C(5)	121.69(17)
O(2)-C(7)	120.1(2)	C(4)-H(4)	93.00	C(1)-C(6)-C(8)	129.81(17)
O(1)-C(8)	120.2(2)	C(14)-H(14)	138.7(3)	C(5)-C(6)-C(8)	108.49(15)
N(1)-C(8)	139.6(2)	C(1)-C(2)	93.00	C(19)-C(18)-C(17)	120.90(18)
N(1)-C(7)	139.7(2)	C(1)-H(1)	137.9(3)	C(19)-C(18)-H(18)	119.5
C(10)-C(15)	138.3(2)	C(3)-C(2)	93.00	C(17)-C(18)-H(18)	119.5
C(10)-C(11)	138.8(2)	C(3)-H(3)	93.00	C(13)-C(12)-C(11)	120.26(17)
C(10)-C(9)	150.4(2)	C(2)-H(2)	138.7(3)	C(13)-C(12)-H(12)	119.9
C(17)-C(22)	138.3(3)	C(4)-C(3)	110.65(12)	C(11)-C(12)-H(12)	119.9
C(17)-C(18)	139.6(2)	N(1)-O(3)-C(9)	122.39(13)	O(2)-C(7)-N(1)	125.47(17)
C(17)-C(16)	148.5(2)	O(3)-N(1)-C(8)	120.11(14)	O(2)-C(7)-C(5)	130.61(17)
C(9)-C(16)	153.4(2)	O(3)-N(1)-C(7)	113.75(14)	N(1)-C(7)-C(5)	103.92(15)
C(9)-H(9)	98.00	C(8)-N(1)-C(7)	119.52(16)	C(17)-C(22)-C(21)	120.81(17)
C(11)-C(12)	138.5(3)	C(15)-C(10)-C(11)	118.99(15)	C(17)-C(22)-H(22)	119.6
C(11)-H(11)	93.00	C(15)-C(10)-C(9)	121.41(15)	C(21)-C(22)-H(22)	119.6
C(5)-C(6)	138.2(2)	C(11)-C(10)-C(9)	118.58(16)	C(10)-C(15)-C(14)	120.19(18)
C(5)-C(4)	138.3(2)	C(22)-C(17)-C(18)	123.08(15)	C(10)-C(15)-H(15)	119.9
C(5)-C(7)	148.3(3)	C(22)-C(17)-C(16)	118.27(16)	C(14)-C(15)-H(15)	119.9
C(8)-C(6)	148.7(2)	C(18)-C(17)-C(16)	113.94(13)	C(21)-C(20)-C(19)	121.54(17)
C(6)-C(1)	137.6(3)	O(3)-C(9)-C(10)	103.17(13)	C(21)-C(20)-Cl(1)	119.22(16)
C(18)-C(19)	137.3(3)	O(3)-C(9)-C(16)	109.72(13)	C(19)-C(20)-Cl(1)	119.23(15)
C(18)-H(18)	93.00	C(10)-C(9)-C(16)	109.9	C(18)-C(19)-C(20)	119.06(17)

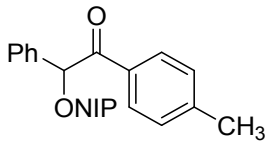
C(12)-C(13)	137.6(3)	O(3)-C(9)-H(9)	109.9	C(18)-C(19)-H(19)	120.5
C(12)-H(12)	93.00	C(10)-C(9)-H(9)	109.9	C(20)-C(19)-H(19)	120.5
C(22)-C(21)	138.5(3)	C(16)-C(9)-H(9)	121.43(15)	C(20)-C(21)-C(22)	119.10(19)
C(22)-H(22)	93.00	O(4)-C(16)-C(17)	119.25(15)	C(20)-C(21)-H(21)	120.5
C(15)-C(14)	138.7(3)	O(4)-C(16)-C(9)	121.13(18)	C(22)-C(21)-H(21)	120.5
C(15)-H(15)	93.00	C(6)-C(5)-C(4)	109.09(15)	C(14)-C(13)-C(12)	120.14(18)
C(20)-C(21)	136.8(3)	C(6)-C(5)-C(7)	129.78(18)	C(14)-C(13)-H(13)	119.9
C(20)-C(19)	137.8(3)	C(4)-C(5)-C(7)			

4. Compound characterizations



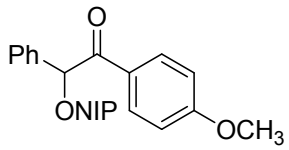
3a

2-(2-oxo-1,2-diphenylethoxy)isoindoline-1,3-dione (3a)[1]. White solid (99.6 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.7 Hz, 2H), 7.76 (dt, *J* = 7.3, 3.7 Hz, 2H), 7.73 – 7.66 (m, 2H), 7.62 (dd, *J* = 6.5, 2.8 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.38 – 7.29 (m, 3H), 6.76 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.6, 163.1, 134.6, 134.4, 133.6, 132.5, 129.9, 129.3, 128.9, 128.8, 128.6, 128.5, 123.4, 88.2.



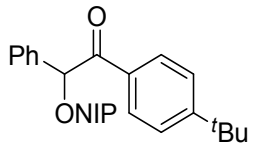
3b

2-(2-oxo-1-phenyl-2-(p-tolyl)ethoxy)isoindoline-1,3-dione (3b). White solid (95.7 mg, 86% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.1 Hz, 2H), 7.74 (dt, *J* = 7.0, 3.6 Hz, 2H), 7.71 – 7.66 (m, 2H), 7.65 – 7.57 (m, 2H), 7.38 – 7.29 (m, 3H), 7.20 (d, *J* = 8.0 Hz, 2H), 6.74 (s, 1H), 2.35 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.2, 163.1, 144.6, 134.4, 132.8, 132.1, 129.9, 129.32, 129.27, 129.1, 128.8, 128.6, 123.4, 88.1, 21.6; HRMS (ESI-TOF): Anal. Calcd. For $C_{23}H_{17}NO_4$: 394.1050, Found: 394.1051 ($M+Na^+$).



3c

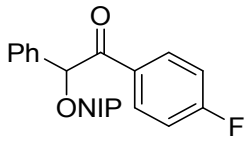
2-(2-(4-methoxyphenyl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3c)[2]. White solid (113.8 mg, 98% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.8 Hz, 2H), 7.74 (dd, *J* = 5.2, 3.2 Hz, 2H), 7.68 (dd, *J* = 5.2, 3.2 Hz, 2H), 7.65 – 7.58 (m, 2H), 7.34 (d, *J* = 3.7 Hz, 3H), 6.87 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.1, 163.8, 163.2, 134.4, 132.9, 131.4, 129.8, 129.2, 128.7, 128.5, 127.5, 123.4, 113.8, 88.0, 55.3.



3d

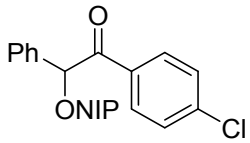
2-(2-(4-(*tert*-butyl)phenyl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3d). Yellow solid (104.1 mg, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.75 (dt, *J* = 7.3, 3.7 Hz, 2H), 7.72 – 7.67 (m, 2H), 7.66 – 7.59 (m, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.39 – 7.29 (m, 3H), 6.78

(s, 1H), 1.29 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.1, 163.2, 157.5, 134.4, 132.7, 132.0, 129.9, 129.4, 129.0, 128.8, 128.6, 125.6, 123.4, 88.1, 35.1, 30.9; HRMS (ESI-TOF): Anal. Calcd. For $C_{26}H_{23}NO_4$: 436.1519, Found: 436.1513 (M+ Na^+).



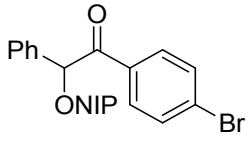
3e

2-(2-(4-fluorophenyl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3e). White solid (109.1 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 8.5, 5.5 Hz, 2H), 7.75 (dt, *J* = 7.0, 3.7 Hz, 2H), 7.72 – 7.66 (m, 2H), 7.64 – 7.57 (m, 2H), 7.40 – 7.31 (m, 3H), 7.08 (t, *J* = 8.5 Hz, 2H), 6.68 (s, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -103.44; ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.3, 165.8 (d, *J* = 247.4 Hz), 163.1, 134.5, 132.4, 131.9 (d, *J* = 10.1 Hz), 131.0 (d, *J* = 3.0 Hz), 130.0, 129.2, 128.9, 128.6, 123.5, 115.8 (d, *J* = 22.2 Hz), 88.5; HRMS (ESI-TOF): Anal. Calcd. For $C_{22}H_{14}FNO_4$: 398.0799, Found: 398.0790 (M+Na^+).



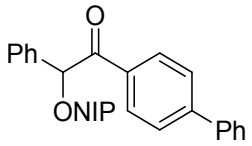
3f

2-(2-(4-chlorophenyl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3f). White solid (98.5 mg, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.6 Hz, 2H), 7.75 (dt, *J* = 7.1, 3.7 Hz, 2H), 7.72 – 7.67 (m, 2H), 7.60 (dd, *J* = 6.5, 2.7 Hz, 2H), 7.36 (dd, *J* = 8.8, 5.6 Hz, 5H), 6.67 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.7, 163.1, 140.1, 134.5, 132.9, 132.3, 130.5, 130.0, 129.1, 128.9, 128.5, 123.5, 88.6; HRMS (ESI-TOF): Anal. Calcd. For $C_{22}H_{14}ClNO_4$: 414.0504, Found: 414.0502 (M+Na^+).



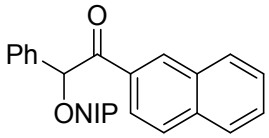
3g

2-(2-(4-bromophenyl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3g). White solid (109.2 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.5 Hz, 2H), 7.75 (dt, *J* = 7.0, 3.6 Hz, 2H), 7.72 – 7.66 (m, 2H), 7.59 (d, *J* = 3.8 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.40 – 7.31 (m, 3H), 6.66 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.9, 163.1, 134.5, 133.3, 132.3, 131.9, 130.6, 130.0, 129.1, 128.91, 128.89, 128.5, 123.5, 88.6; HRMS (ESI-TOF): Anal. Calcd. For $C_{22}H_{14}BrNO_4$: 436.0179, Found: 436.0176 (M+H^+).



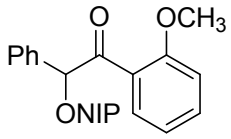
3h

2-(2-([1,1'-biphenyl]-4-yl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3h). White solid (119.5 mg, 92% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.4 Hz, 2H), 7.76 (dt, *J* = 7.2, 3.6 Hz, 2H), 7.73 – 7.68 (m, 2H), 7.64 (t, *J* = 7.7 Hz, 4H), 7.57 (d, *J* = 7.3 Hz, 2H), 7.44 (t, *J* = 7.3 Hz, 2H), 7.37 (dd, *J* = 8.0, 4.3 Hz, 4H), 6.78 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.3, 163.2, 146.3, 139.6, 134.5, 133.3, 132.7, 130.0, 129.7, 129.4, 128.9, 128.7, 128.3, 127.23, 127.21, 123.5, 88.4; HRMS (ESI-TOF): Anal. Calcd. For $C_{28}H_{19}NO_4$: 434.1387, Found: 434.1384 ($M+H^+$).



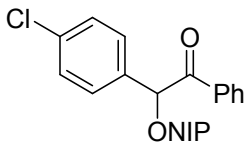
3i

2-(2-(naphthalen-2-yl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3i). Light yellow solid (112.3 mg, 92% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 8.05 (dd, *J* = 8.7, 1.4 Hz, 1H), 7.93 (d, *J* = 8.1 Hz, 1H), 7.83 (t, *J* = 8.6 Hz, 2H), 7.76 (dt, *J* = 7.1, 3.6 Hz, 2H), 7.72 – 7.64 (m, 4H), 7.60 – 7.49 (m, 2H), 7.36 (dd, *J* = 8.9, 3.1 Hz, 3H), 6.91 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.7, 163.2, 135.7, 134.5, 132.7, 132.3, 132.0, 131.3, 130.0, 129.8, 129.4, 128.9, 128.7, 128.5, 127.7, 126.8, 124.3, 123.5, 88.4; HRMS (ESI-TOF): Anal. Calcd. For $C_{26}H_{17}NO_4$: 430.1050, Found: 430.1045 ($M+Na^+$).



3j

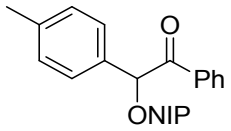
2-(2-(2-methoxyphenyl)-2-oxo-1-phenylethoxy)isoindoline-1,3-dione (3j). White solid (110.3 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 – 7.72 (m, 3H), 7.70 – 7.64 (m, 2H), 7.56 – 7.48 (m, 2H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.33 – 7.22 (m, 3H), 6.96 – 6.88 (m, 2H), 6.83 (d, *J* = 8.4 Hz, 1H), 3.79 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.7, 163.0, 158.0, 134.3, 134.2, 132.5, 131.0, 129.6, 129.5, 128.6, 128.3, 125.6, 123.3, 120.8, 111.4, 90.4, 55.2; HRMS (ESI-TOF): Anal. Calcd. For $C_{23}H_{17}NO_5$: 410.0999, Found: 410.0998 ($M+Na^+$).



3k

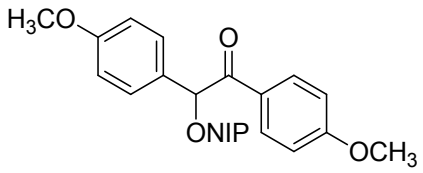
2-(1-(4-chlorophenyl)-2-oxo-2-phenylethoxy)isoindoline-1,3-dione (3k). White solid (116.1 mg, 99% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.5 Hz, 2H), 7.76 (dt, *J* = 6.9, 3.6 Hz, 2H), 7.73

– 7.66 (m, 2H), 7.56 (dd, *J* = 12.9, 7.9 Hz, 3H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 6.74 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.3, 163.1, 136.0, 134.5, 134.4, 133.8, 131.1, 130.7, 129.05, 129.03, 128.7, 128.5, 123.6, 87.3; HRMS (ESI-TOF): Anal. Calcd. For $C_{22}H_{14}ClNO_4$: 414.0504, Found: 414.0502 ($M+Na^+$).



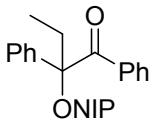
3l

2-(2-oxo-2-phenyl-1-(p-tolyl)ethoxy)isoindoline-1,3-dione (3l). Light yellow solid (96.8 mg, 87% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.7 Hz, 2H), 7.74 (dt, *J* = 7.4, 3.9 Hz, 2H), 7.71 – 7.64 (m, 2H), 7.50 (t, *J* = 7.1 Hz, 3H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 6.76 (s, 1H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.7, 163.2, 140.0, 134.6, 134.4, 133.5, 129.5, 129.4, 129.3, 128.9, 128.6, 128.5, 123.4, 87.9, 21.2; HRMS (ESI-TOF): Anal. Calcd. For $C_{23}H_{17}NO_4$: 394.1050, Found: 394.1049 ($M+Na^+$).



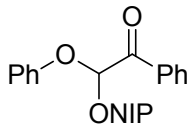
3m

2-(1,2-bis(4-methoxyphenyl)-2-oxoethoxy)isoindoline-1,3-dione (3m). White solid (123.8 mg, 99% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.8 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.71 – 7.66 (m, 2H), 7.52 (d, *J* = 8.6 Hz, 2H), 6.86 (t, *J* = 9.2 Hz, 4H), 6.69 (s, 1H), 3.82 (d, *J* = 1.0 Hz, 3H), 3.74 (d, *J* = 1.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.2, 163.8, 163.3, 160.7, 134.4, 131.4, 131.0, 128.6, 127.6, 124.8, 123.4, 114.2, 113.8, 87.4, 55.4, 55.1; HRMS (ESI-TOF): Anal. Calcd. For $C_{24}H_{19}NO_6$: 440.1105, Found: 440.1100 ($M+Na^+$).



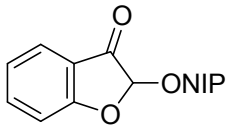
3n

2-((1-oxo-1,2-diphenylbutan-2-yl)oxy)isoindoline-1,3-dione (3n). White solid (107.4 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.4 Hz, 2H), 7.77 (dt, *J* = 7.1, 3.7 Hz, 2H), 7.74 – 7.69 (m, 2H), 7.62 (d, *J* = 7.3 Hz, 2H), 7.40 (dt, *J* = 14.6, 7.6 Hz, 3H), 7.35 – 7.25 (m, 3H), 2.56 (dq, *J* = 14.7, 7.4 Hz, 1H), 2.07 (dq, *J* = 14.5, 7.3 Hz, 1H), 0.76 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.0, 164.3, 138.3, 135.8, 134.6, 132.1, 130.7, 128.8, 128.4, 128.4, 127.7, 126.5, 123.6, 95.8, 28.1, 8.4; HRMS (ESI-TOF): Anal. Calcd. For $C_{24}H_{19}NO_4$: 408.1206, Found: 408.1208 ($M+Na^+$).



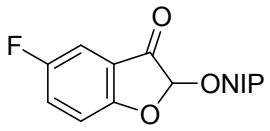
3o

2-(2-oxo-1-phenoxy-2-phenylethoxy)isoindoline-1,3-dione (3o). White solid (107.5 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 7.5 Hz, 2H), 7.68 (dt, *J* = 7.3, 3.7 Hz, 2H), 7.65 – 7.59 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.20 – 7.14 (m, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 6.95 (t, *J* = 7.2 Hz, 1H), 6.39 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.2, 162.9, 156.3, 134.7, 134.2, 133.0, 130.2, 129.6, 128.6, 128.5, 123.8, 123.7, 117.2, 104.9; HRMS (ESI-TOF): Anal. Calcd. For $C_{22}H_{15}NO_5$: 396.0842, Found: 396.0843 ($M+Na^+$).



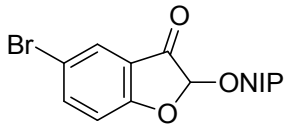
5a

2-((3-oxo-2,3-dihydrobenzofuran-2-yl)oxy)isoindoline-1,3-dione (5a). White solid (79.4 mg, 67% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.83 – 7.76 (m, 2H), 7.73 – 7.65 (m, 2H), 7.21 – 7.13 (m, 2H), 5.90 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.1, 171.6, 162.6, 139.4, 134.8, 128.7, 125.3, 123.9, 123.5, 119.2, 113.6, 101.5; HRMS (ESI-TOF): Anal. Calcd. For $C_{16}H_9NO_5$: 318.0373, Found: 318.0369 ($M+Na^+$).



5b

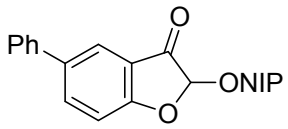
2-((5-fluoro-3-oxo-2,3-dihydrobenzofuran-2-yl)oxy)isoindoline-1,3-dione (5b). White solid (89.2 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dt, *J* = 7.2, 3.6 Hz, 2H), 7.84 – 7.77 (m, 2H), 7.46 – 7.32 (m, 2H), 7.16 (dd, *J* = 8.9, 3.5 Hz, 1H), 5.92 (s, 1H); ^{19}F NMR (376 MHz, $CDCl_3$) δ -118.48; ^{113}C NMR (101 MHz, $CDCl_3$) δ 190.6 (d, *J* = 3.0 Hz), 167.7, 162.6, 158.5 (d, *J* = 246.4 Hz), 134.9, 128.6, 126.8 (d, *J* = 25.2 Hz), 124.0, 119.8 (d, *J* = 8.1 Hz), 114.9 (d, *J* =6.1 Hz), 110.6 (d, *J* = 24.2 Hz), 102.3; HRMS (ESI-TOF): Anal. Calcd. For $C_{16}H_8FNO_5$: 336.0279, Found: 336.0273 ($M+Na^+$).



5c

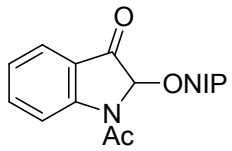
2-((5-bromo-3-oxo-2,3-dihydrobenzofuran-2-yl)oxy)isoindoline-1,3-dione (5c). White solid (35.8 mg, 32% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.83 – 7.78 (m, 3H), 7.76 (dd, *J* = 8.8, 2.1 Hz, 1H), 7.10 (d, *J* = 8.7 Hz, 1H), 5.92 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$)

δ 189.8, 170.3, 162.6, 141.9, 134.9, 128.7, 127.8, 124.0, 120.9, 116.2, 115.5, 101.9; HRMS (ESI-TOF): Anal. Calcd. For $C_{16}H_8BrNO_5$: 373.9659, Found: 373.9654 ($M+H^+$).



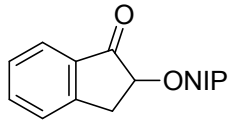
5d

2-((3-oxo-5-phenyl-2,3-dihydrobenzofuran-2-yl)oxy)isoindoline-1,3-dione (5d). White solid (60.1 mg, 54% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.85 (m, 4H), 7.82 – 7.75 (m, 2H), 7.53 (d, *J* = 7.3 Hz, 2H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.25 (d, *J* = 9.1 Hz, 1H), 5.95 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.1, 171.0, 162.7, 139.0, 138.5, 137.2, 134.8, 129.0, 128.7, 127.8, 126.9, 124.0, 123.2, 119.7, 113.9, 102.0; HRMS (ESI-TOF): Anal. Calcd. For $C_{22}H_{13}NO_5$: 394.0686, Found: 394.0694 ($M+Na^+$).



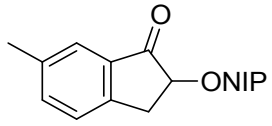
5e

2-((1-acetyl-3-oxoindolin-2-yl)oxy)isoindoline-1,3-dione (5e). White solid (95.8 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 7.6 Hz, 1H), 7.83 (dt, *J* = 7.1, 3.6 Hz, 2H), 7.80 – 7.75 (m, 2H), 7.72 (d, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 5.81 (s, 1H), 2.71 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.7, 169.8, 163.2, 153.2, 138.3, 134.8, 128.7, 125.0, 124.8, 124.0, 121.8, 118.2, 86.2, 23.8; HRMS (ESI-TOF): Anal. Calcd. For $C_{18}H_{12}N_2O_5$: 359.0638, Found: 359.0638 ($M+Na^+$).



5f

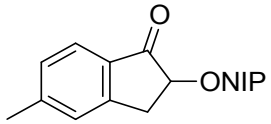
2-((1-oxo-2,3-dihydro-1*H*-inden-2-yl)oxy)isoindoline-1,3-dione (5f)[1]. White solid (81.7 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.7 Hz, 1H), 7.86 (dt, *J* = 7.1, 3.6 Hz, 2H), 7.82 – 7.76 (m, 3H), 7.74 (dd, *J* = 10.9, 4.0 Hz, 1H), 7.58 (t, *J* = 7.5 Hz, 1H), 5.92 (dd, *J* = 6.5, 1.9 Hz, 1H), 3.28 (dd, *J* = 19.2, 2.3 Hz, 1H), 3.09 (dd, *J* = 19.2, 6.6 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.3, 163.8, 148.9, 137.5, 135.1, 134.7, 130.7, 128.7, 127.6, 123.7, 123.4, 83.6, 43.4.



5g

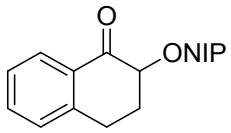
2-((6-methyl-1-oxo-2,3-dihydro-1H-inden-2-yl)oxy)isoindoline-1,3-dione (5g). White solid

(46.1 mg, 50% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.82 (m, 3H), 7.82 – 7.74 (m, 2H), 7.59 (s, 1H), 7.53 (d, *J* = 7.8 Hz, 1H), 5.88 (d, *J* = 5.3 Hz, 1H), 3.26 (dd, *J* = 19.2, 2.1 Hz, 1H), 3.07 (dd, *J* = 19.2, 6.5 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 163.9, 146.4, 141.2, 137.8, 136.3, 134.7, 128.7, 127.3, 123.7, 123.3, 83.4, 43.7, 21.4; HRMS (ESI-TOF): Anal. Calcd. For $C_{18}H_{13}NO_4$: 330.0737, Found: 330.0748 ($M+Na^+$).



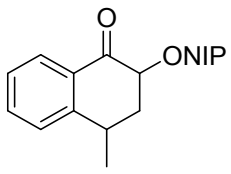
5h

2-((5-methyl-1-oxo-2,3-dihydro-1H-inden-2-yl)oxy)isoindoline-1,3-dione (5h). Yellow solid (44.2 mg, 48% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dt, *J* = 7.3, 3.7 Hz, 2H), 7.83 – 7.75 (m, 3H), 7.69 (d, *J* = 7.9 Hz, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 5.90 – 5.81 (m, 1H), 3.27 (dd, *J* = 19.1, 2.0 Hz, 1H), 3.05 (dd, *J* = 19.1, 6.6 Hz, 1H), 2.51 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.9, 163.9, 149.4, 146.6, 135.3, 134.7, 132.0, 128.8, 127.8, 123.7, 123.2, 83.7, 43.7, 22.0; HRMS (ESI-TOF): Anal. Calcd. For $C_{18}H_{13}NO_4$: 330.0737, Found: 330.0750 ($M+Na^+$).



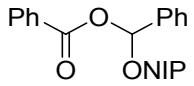
5i

2-((1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl)oxy)isoindoline-1,3-dione (5i)[3]. Yellow solid (88.4 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 7.7 Hz, 1H), 7.83 (dt, *J* = 7.4, 3.8 Hz, 2H), 7.79 – 7.74 (m, 2H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.62 – 7.55 (m, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 5.46 (s, 1H), 3.51 – 3.37 (m, 1H), 2.64 (ddt, *J* = 18.8, 13.5, 4.3 Hz, 2H), 2.47 – 2.34 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.9, 163.9, 137.4, 134.6, 133.8, 132.4, 130.3, 130.1, 128.7, 127.1, 123.6, 82.1, 33.2, 26.9.



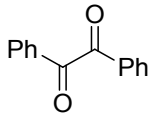
5j

2-((4-methyl-1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl)oxy)isoindoline-1,3-dione (5j). Light yellow solid (65.5 mg, 68% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, *J* = 7.8, 0.9 Hz, 1H), 7.84 – 7.78 (m, 3H), 7.78 – 7.72 (m, 2H), 7.58 (td, *J* = 7.8, 1.3 Hz, 1H), 7.48 (dd, *J* = 11.0, 4.1 Hz, 1H), 3.40 (ddd, *J* = 14.5, 10.2, 5.6 Hz, 1H), 2.76 – 2.62 (m, 2H), 2.37 (ddd, *J* = 13.2, 8.7, 5.5 Hz, 1H), 1.87 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.2, 165.2, 141.9, 134.6, 133.6, 132.0, 129.2, 129.0, 127.2, 125.9, 123.6, 85.4, 34.7, 25.3; HRMS (ESI-TOF): Anal. Calcd. For $C_{19}H_{15}NO_4$: 344.0893, Found: 344.0885 ($M+Na^+$).



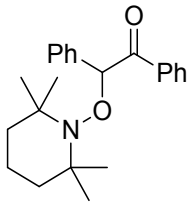
6

((1,3-dioxoisoindolin-2-yl)oxy)(phenyl)methyl benzoate (6)[3]. White solid (95.1 mg, 85% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 5.1 Hz, 2H), 7.82 (d, *J* = 13.4 Hz, 4H), 7.71 (s, 2H), 7.56 (d, *J* = 13.1 Hz, 2H), 7.47 (s, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.3, 163.0, 134.5, 133.8, 133.2, 130.3, 130.2, 128.8, 128.7, 128.6, 128.5, 127.2, 123.7, 100.4.



7

benzil (7). Yellow solid (59.0 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.92 (m, 4H), 7.65 (t, *J* = 7.4 Hz, 2H), 7.50 (t, *J* = 7.8 Hz, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.5, 134.8, 132.9, 129.8, 129.0.



8

1,2-diphenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethanone (8)[4]. Colorless liquid (21.1 mg, 20% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 8.5 Hz, 3H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.19 (dd, *J* = 12.8, 5.1 Hz, 2H), 7.11 (t, *J* = 7.3 Hz, 1H), 5.92 (s, 1H), 1.46 (d, *J* = 12.6 Hz, 1H), 1.37 (d, *J* = 4.3 Hz, 4H), 1.11 (s, 6H), 0.92 (s, 3H), 0.73 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.3, 137.8, 135.2, 132.9, 129.3, 128.3, 127.5, 127.2, 93.2, 60.0, 59.8, 40.2, 33.6, 33.3, 31.4, 20.3, 20.2, 17.0.

5.Reference

[1] A. A. Andia, M. R. Miner, K. A. Woerpel, *Org. Lett.* 2015, **17**, 2704-2707.

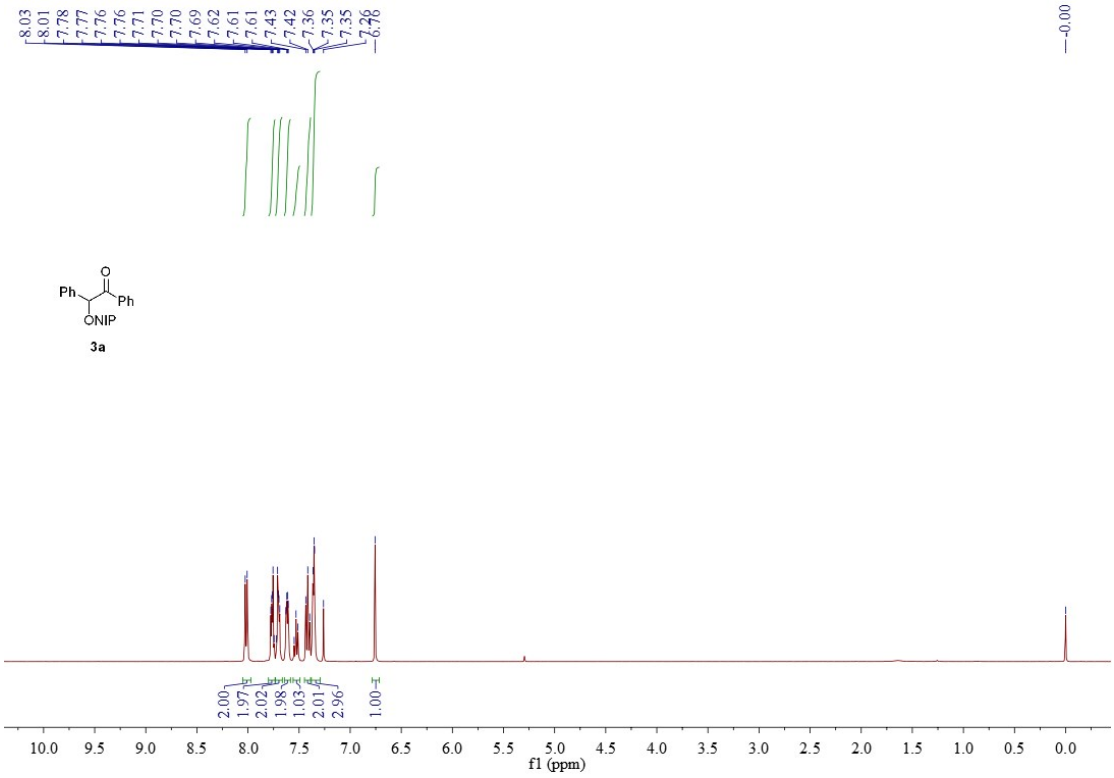
[2] J.-z. Zhang, Y. Tang, *Adv. Synth. Catal.* 2016, **358**, 752-764.

[3] R. Bag, D. Sar, T. Punniyamurthy, *Org. Lett.* 2015, **17**, 2010-2013.

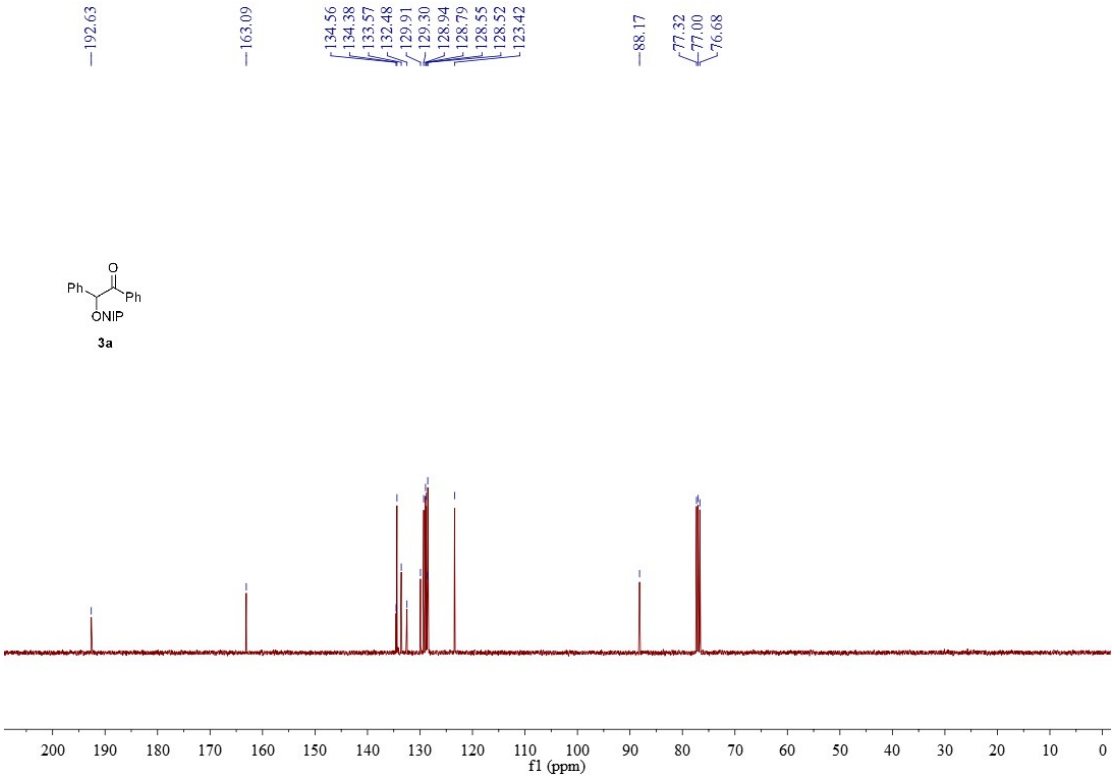
[4] J. Jayram, B. A. Xulu, V. Jeena, *Tetrahedron* 2019, **75**, 130617.

6. Spectroscopic data for products

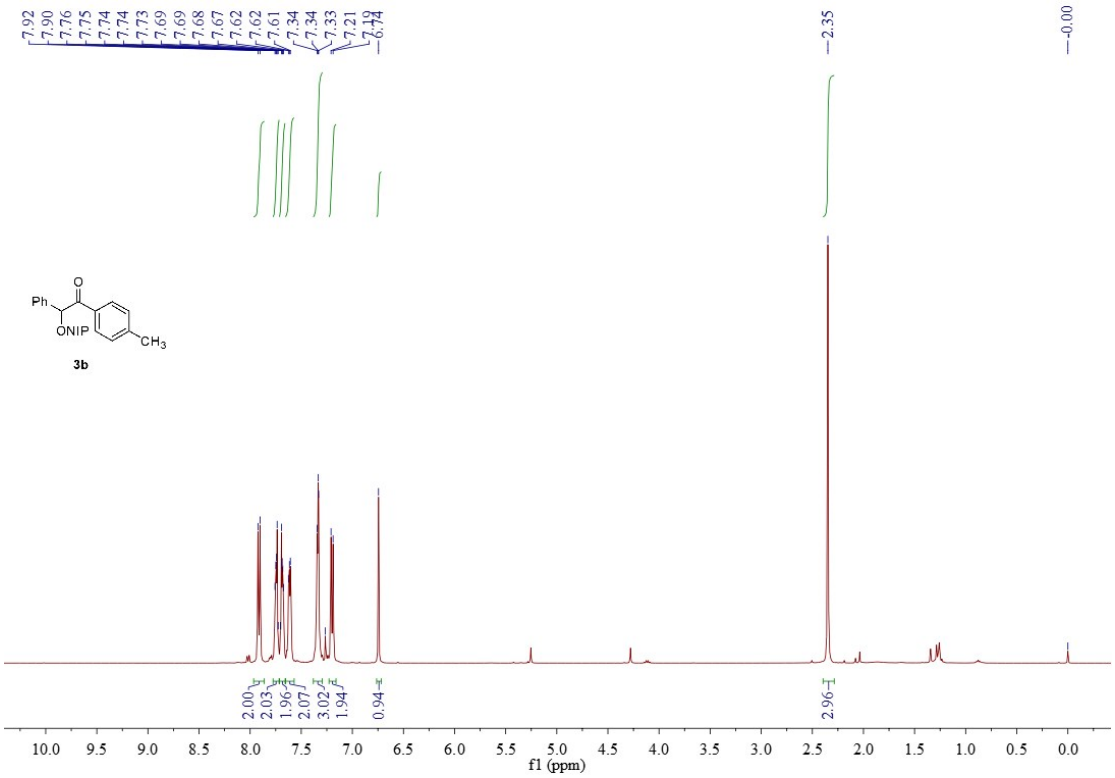
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3a**



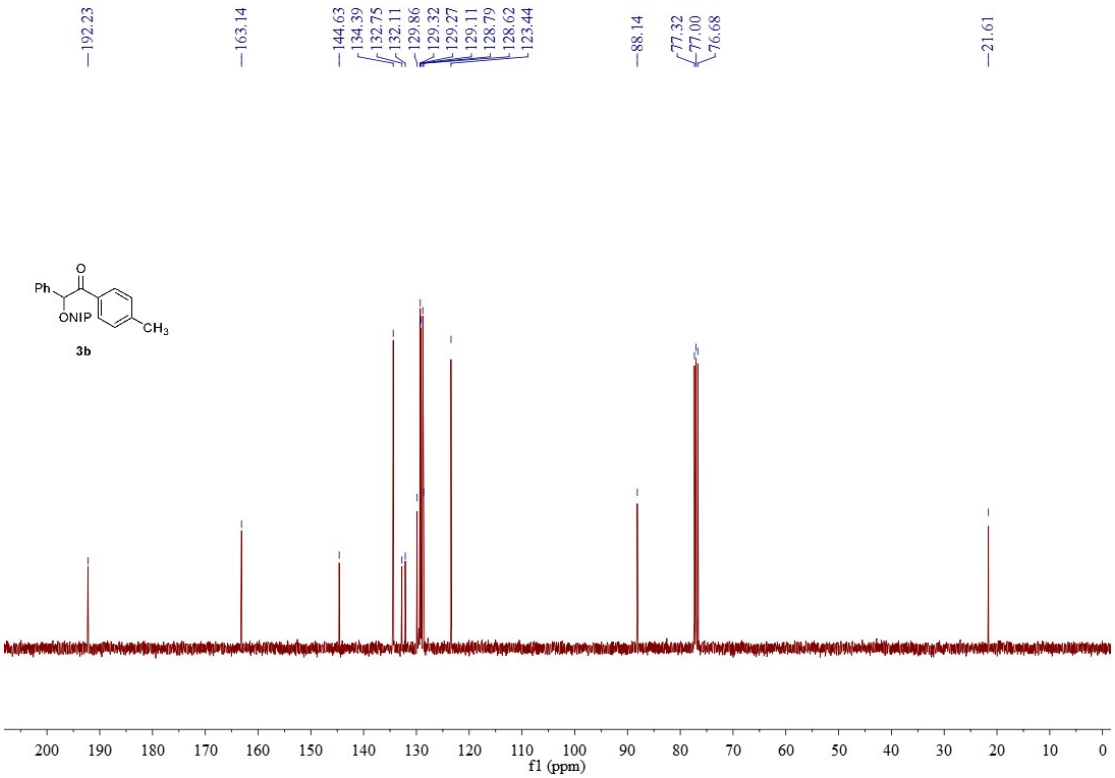
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3a**



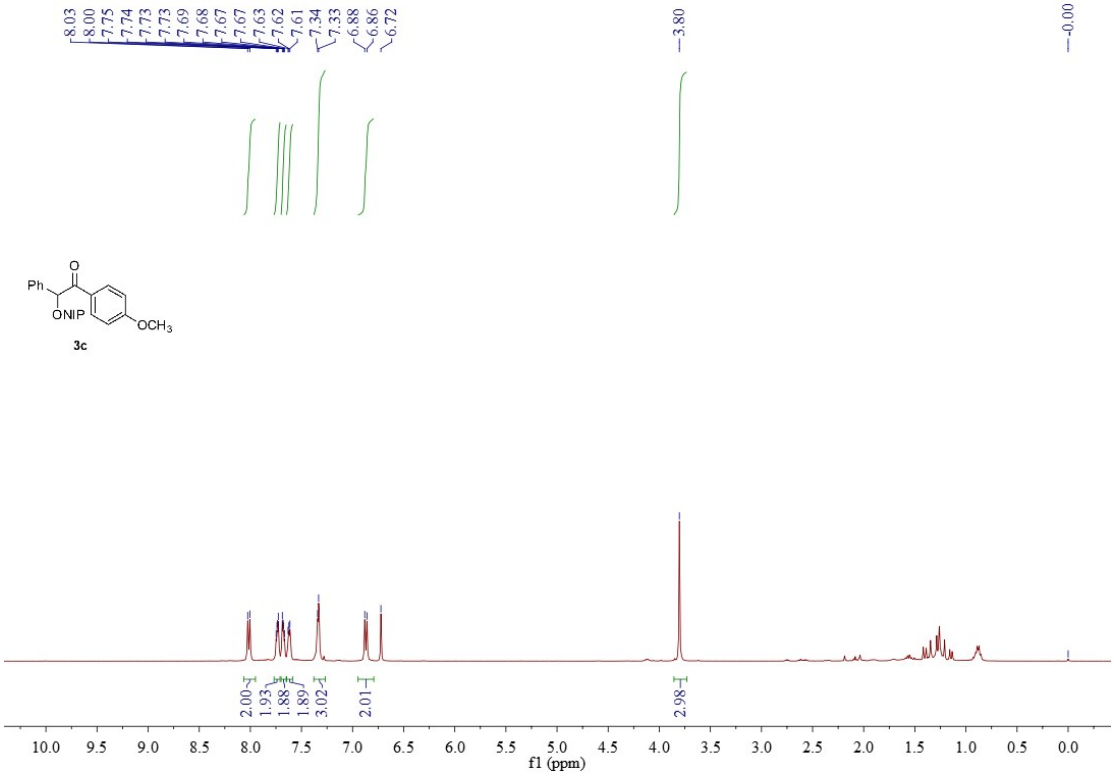
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3b**



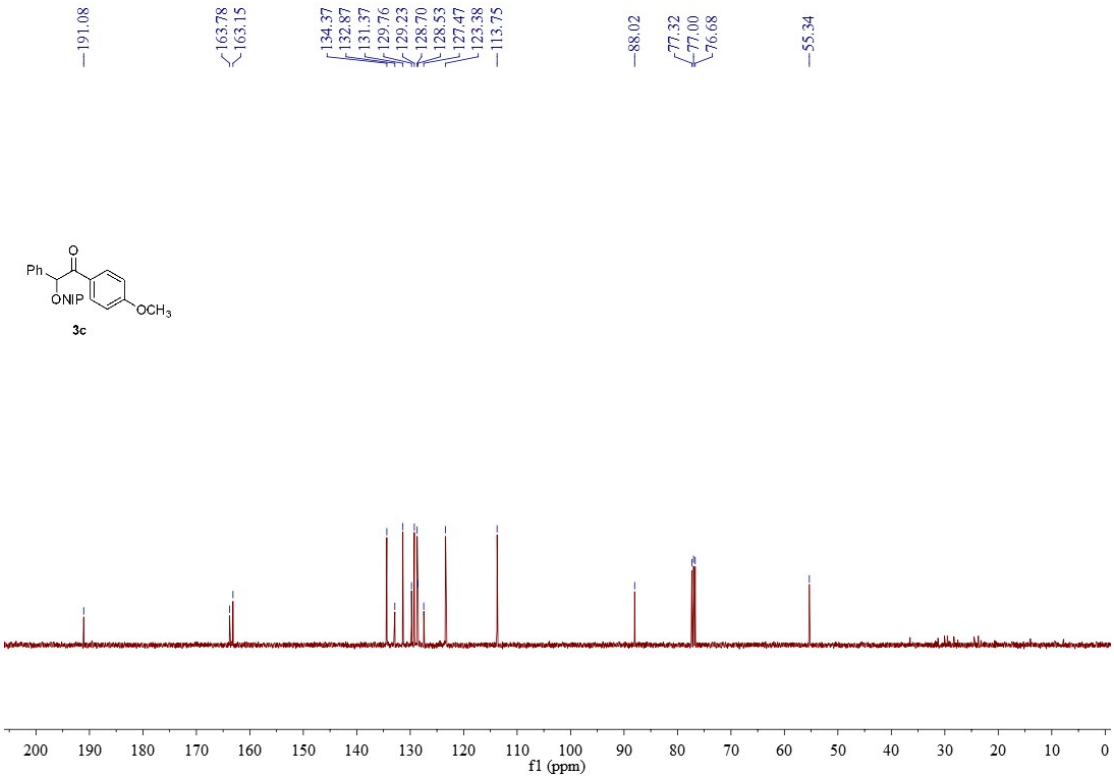
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3b**



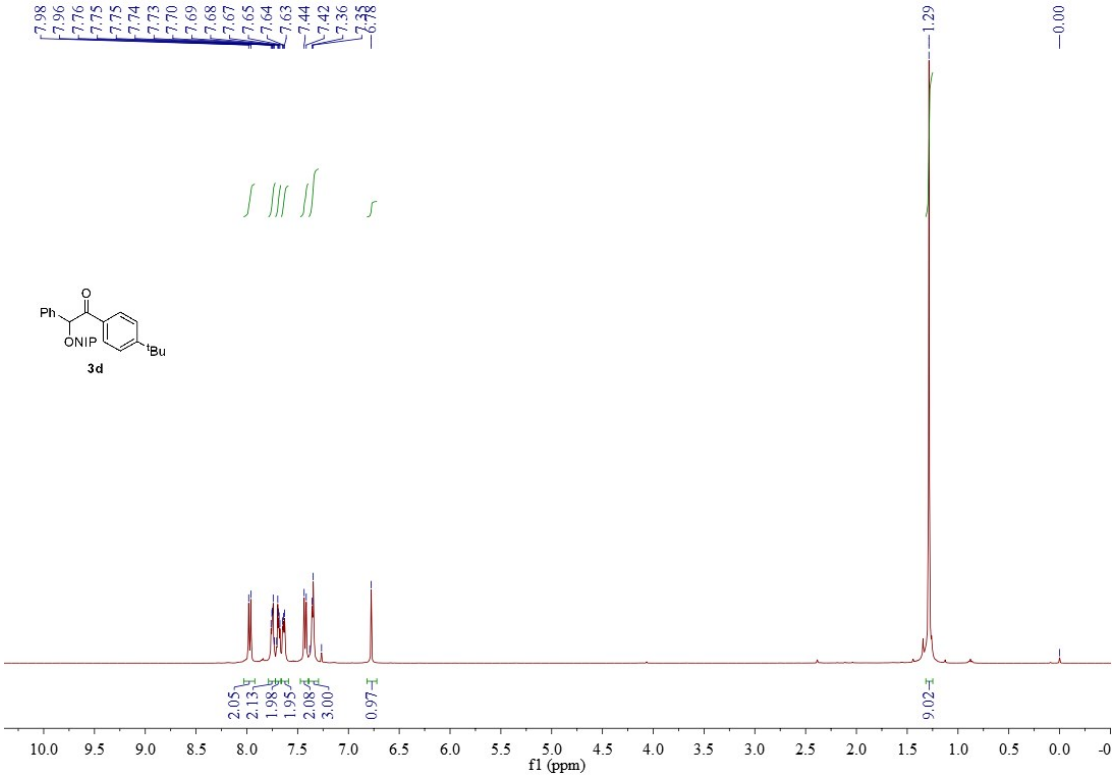
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3c**



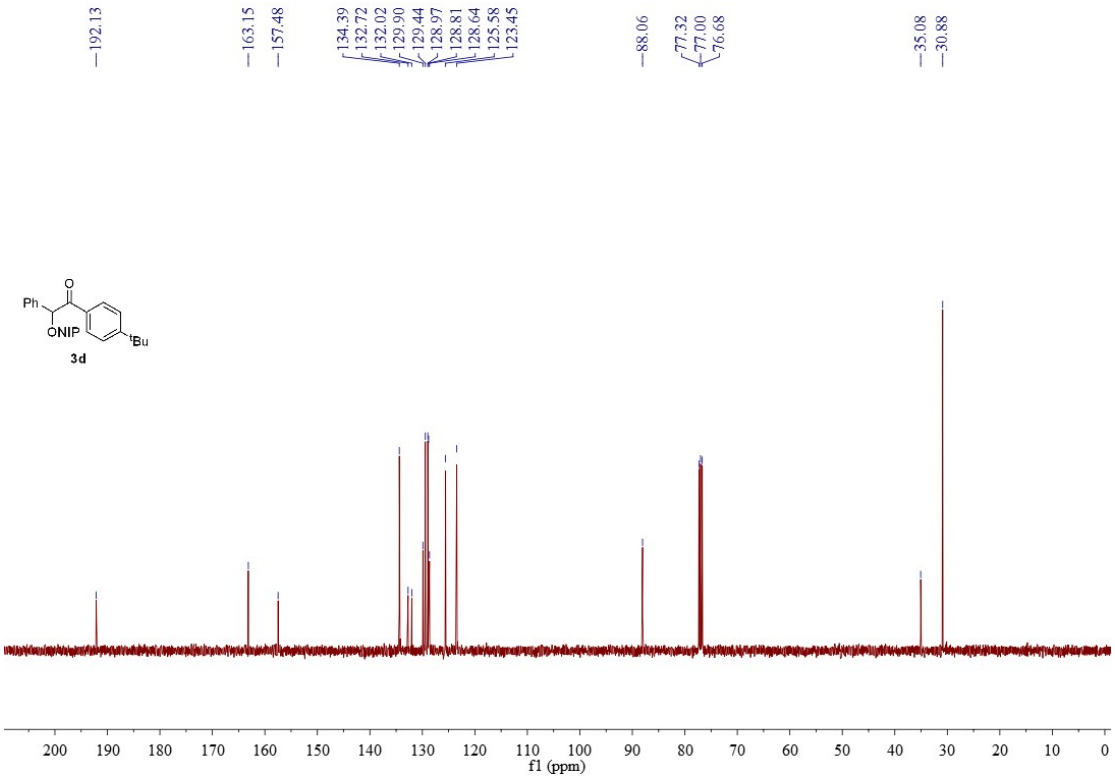
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3c**



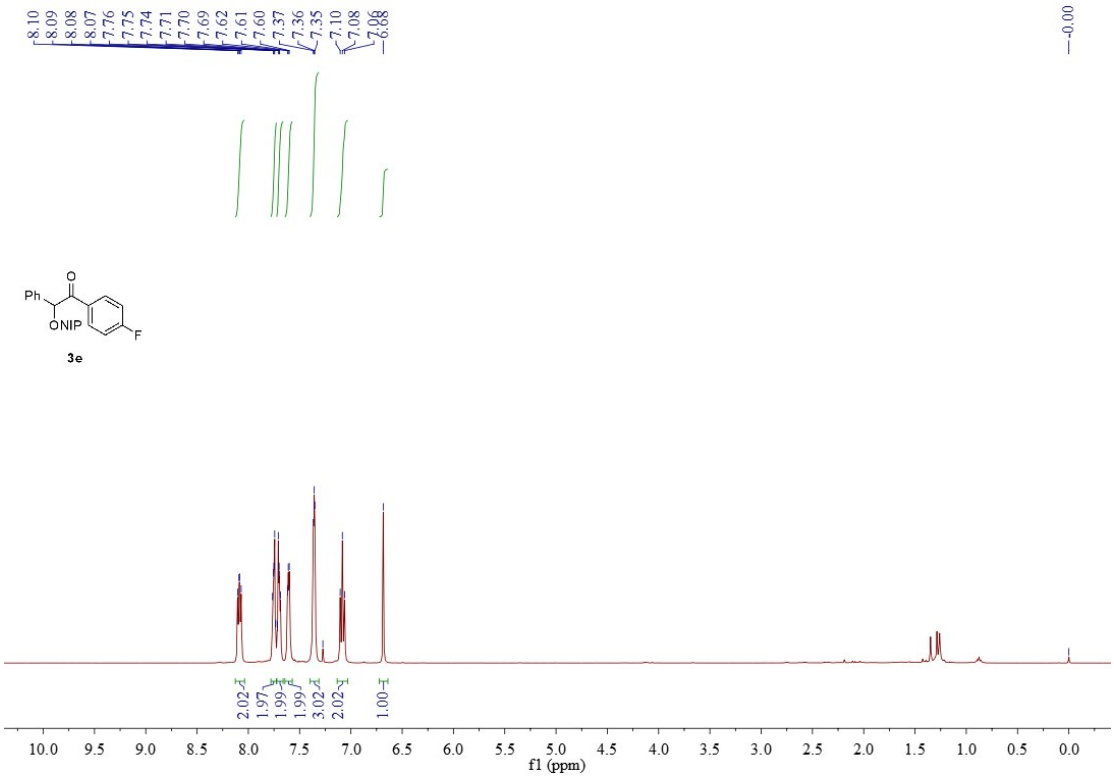
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3d**



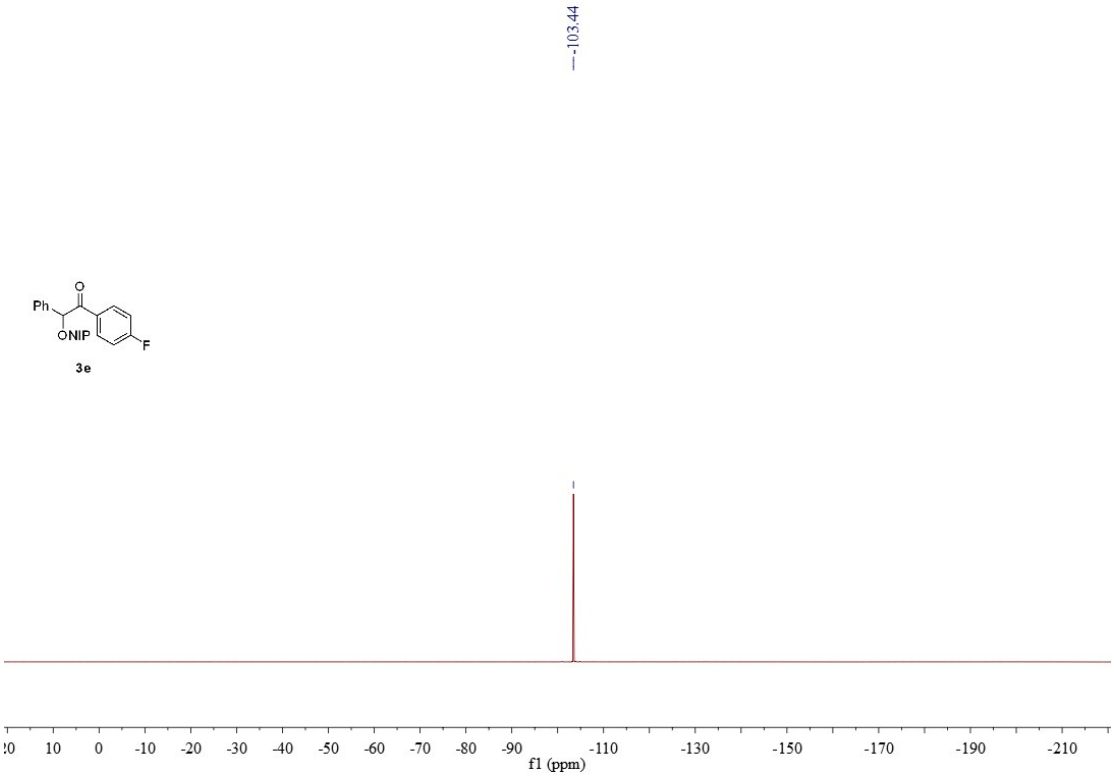
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3d**



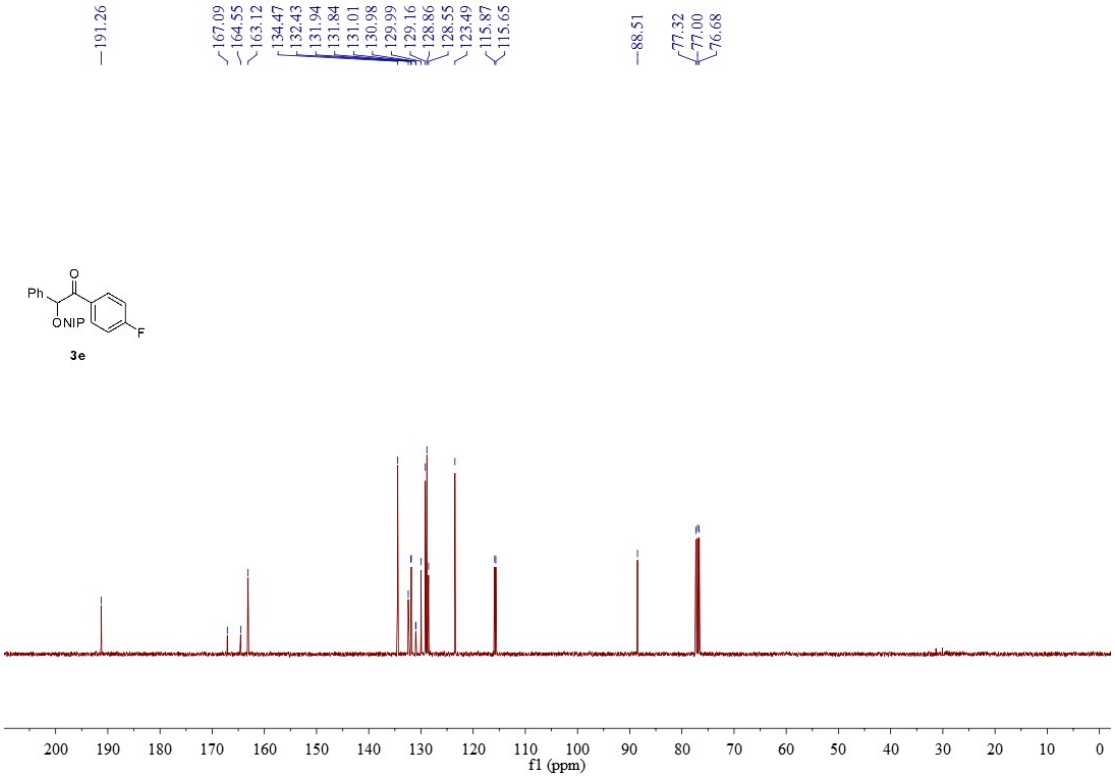
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3e**



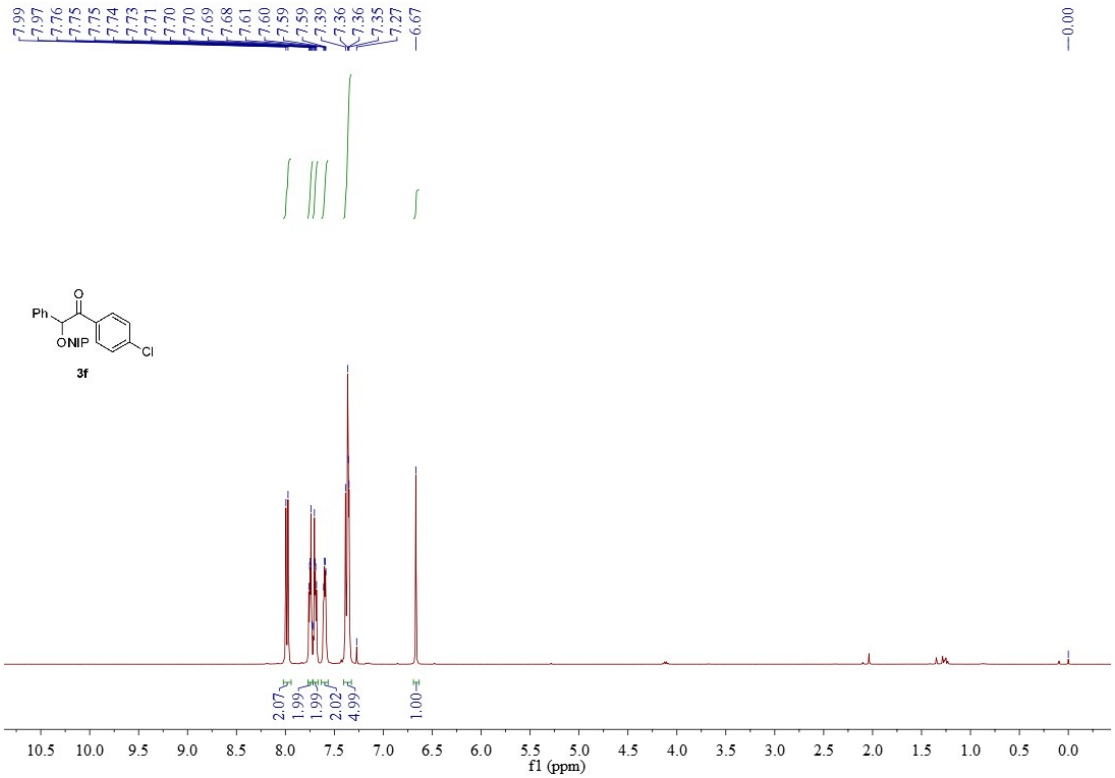
^{19}F NMR (376 MHz, $CDCl_3$) spectra of **3e**



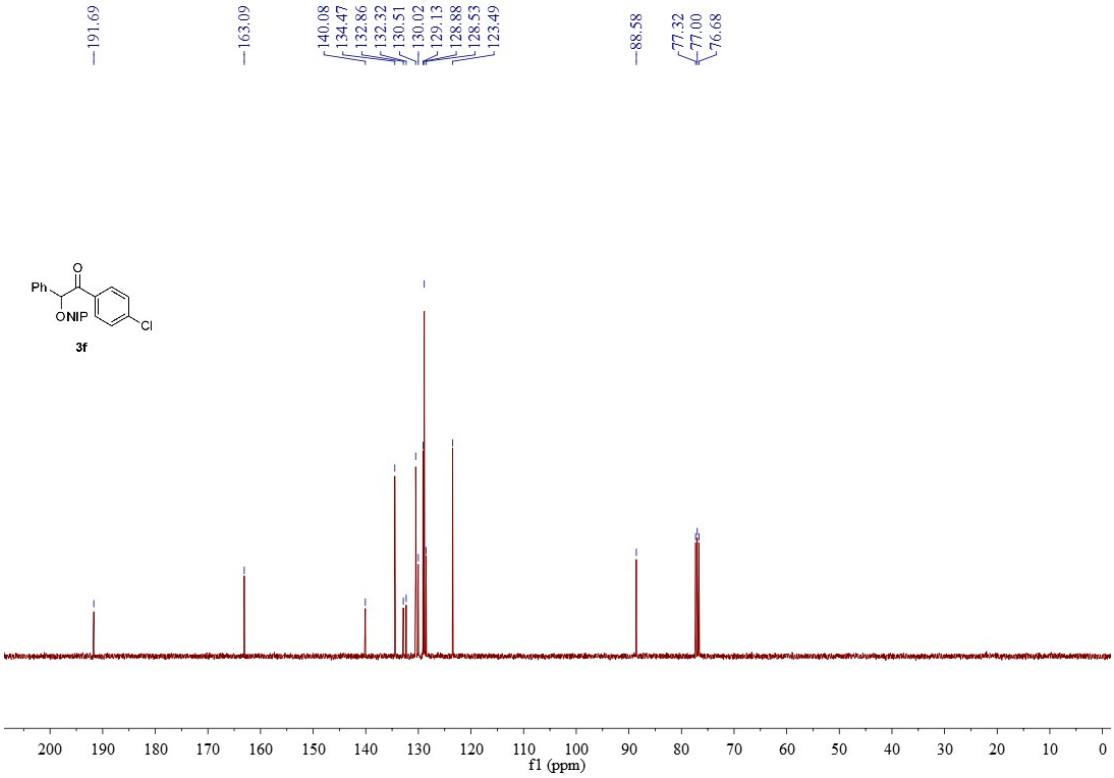
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3e**



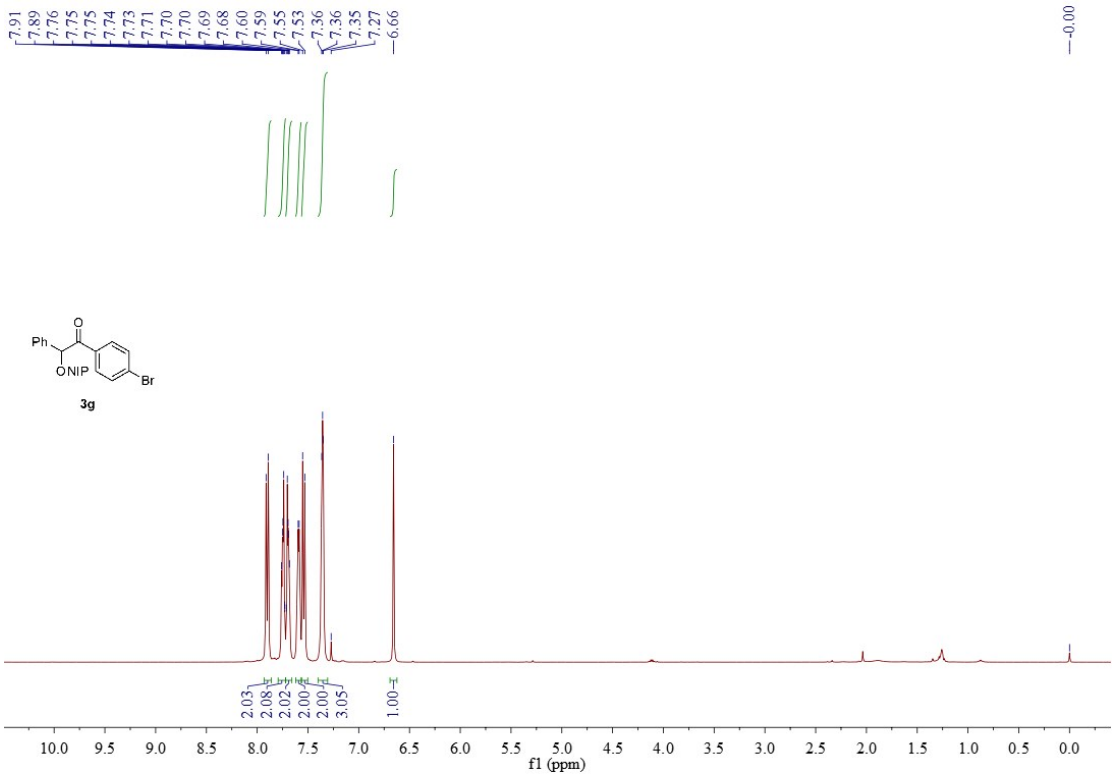
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3f**



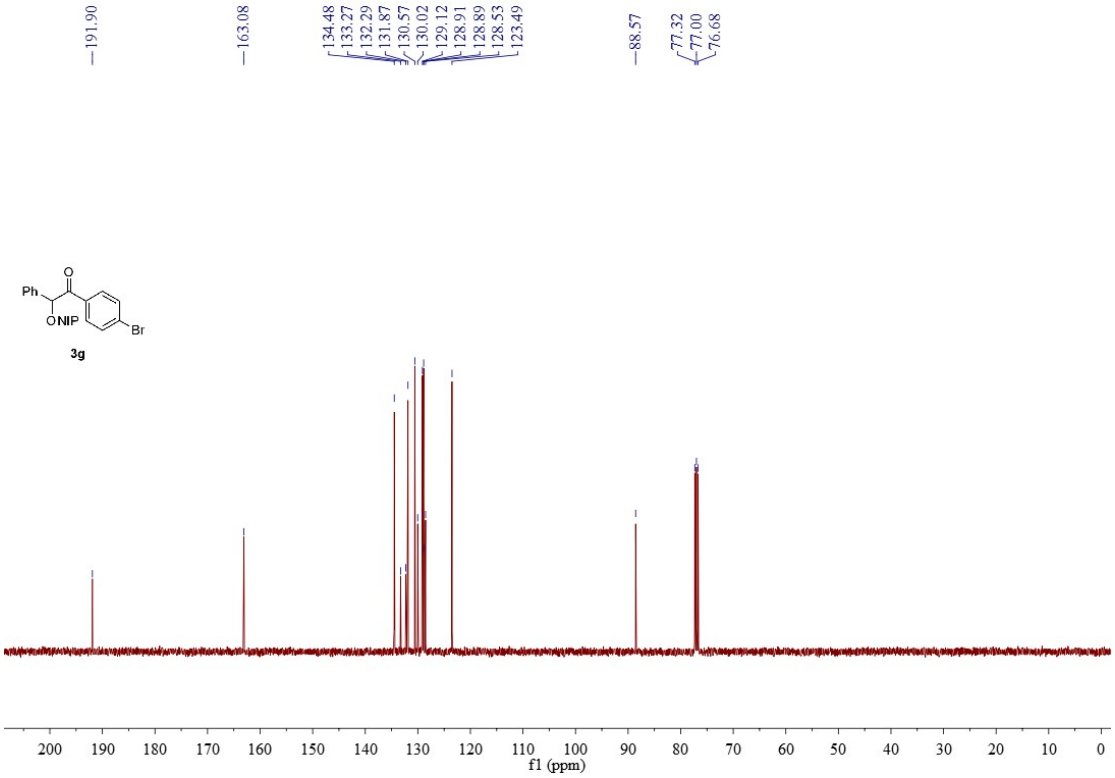
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3f**



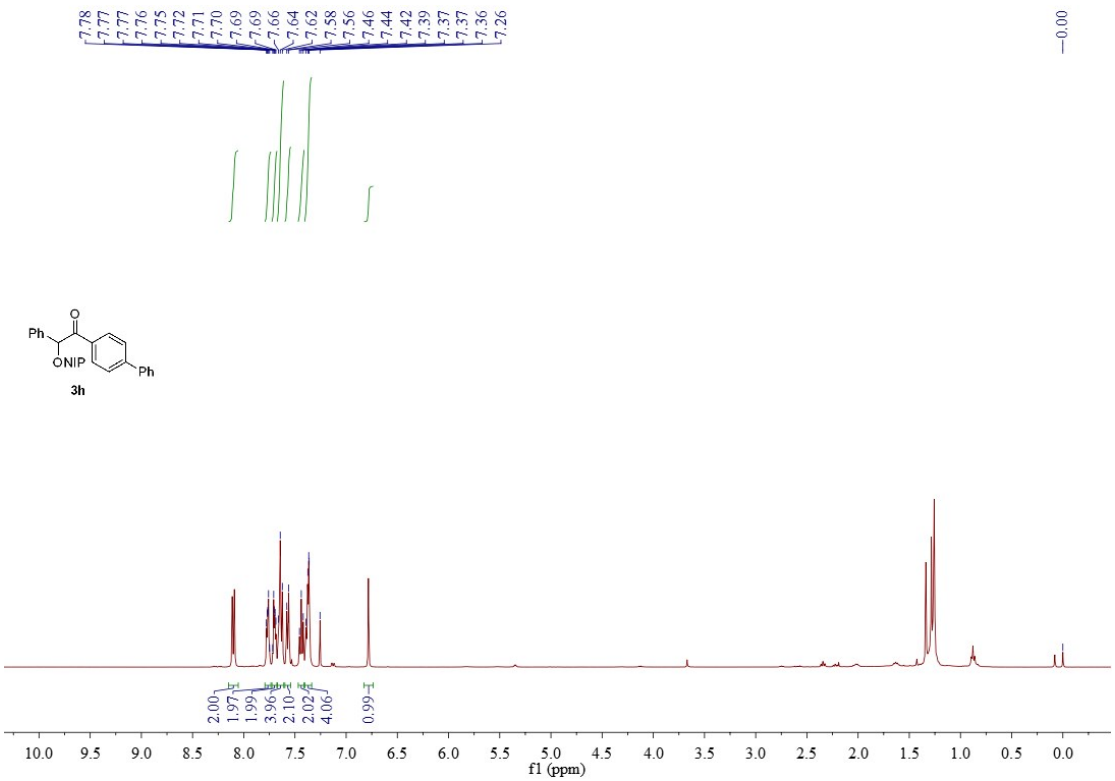
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3g**



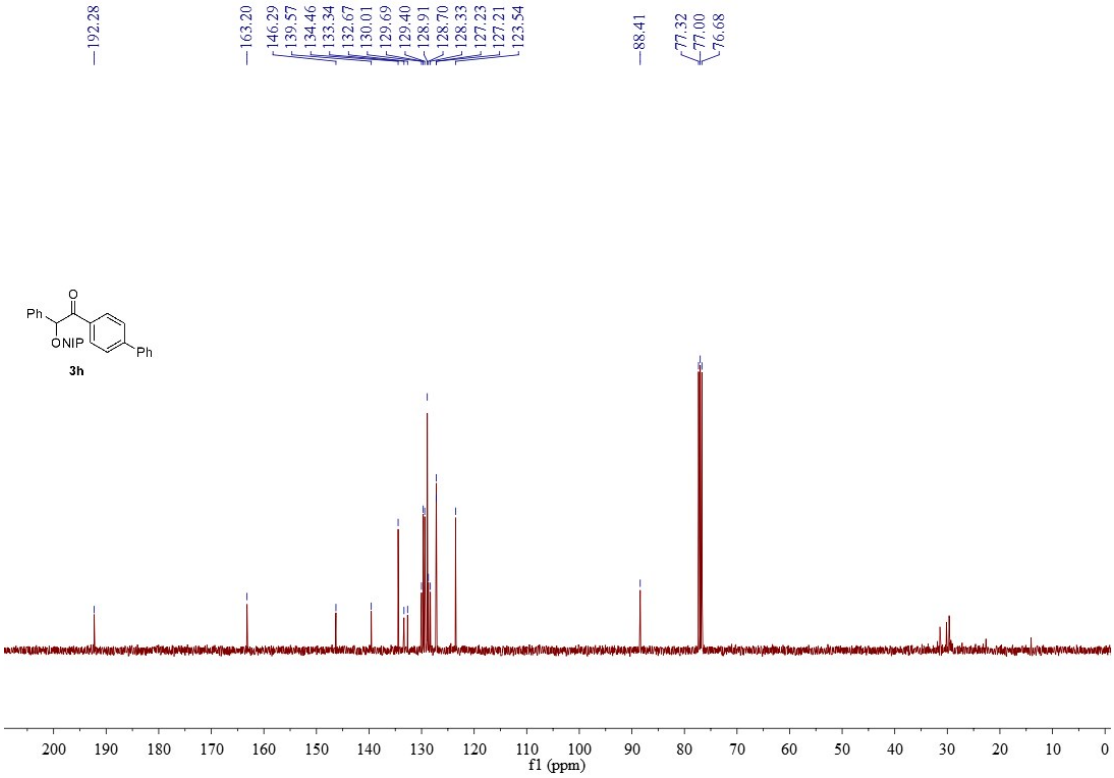
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3g**



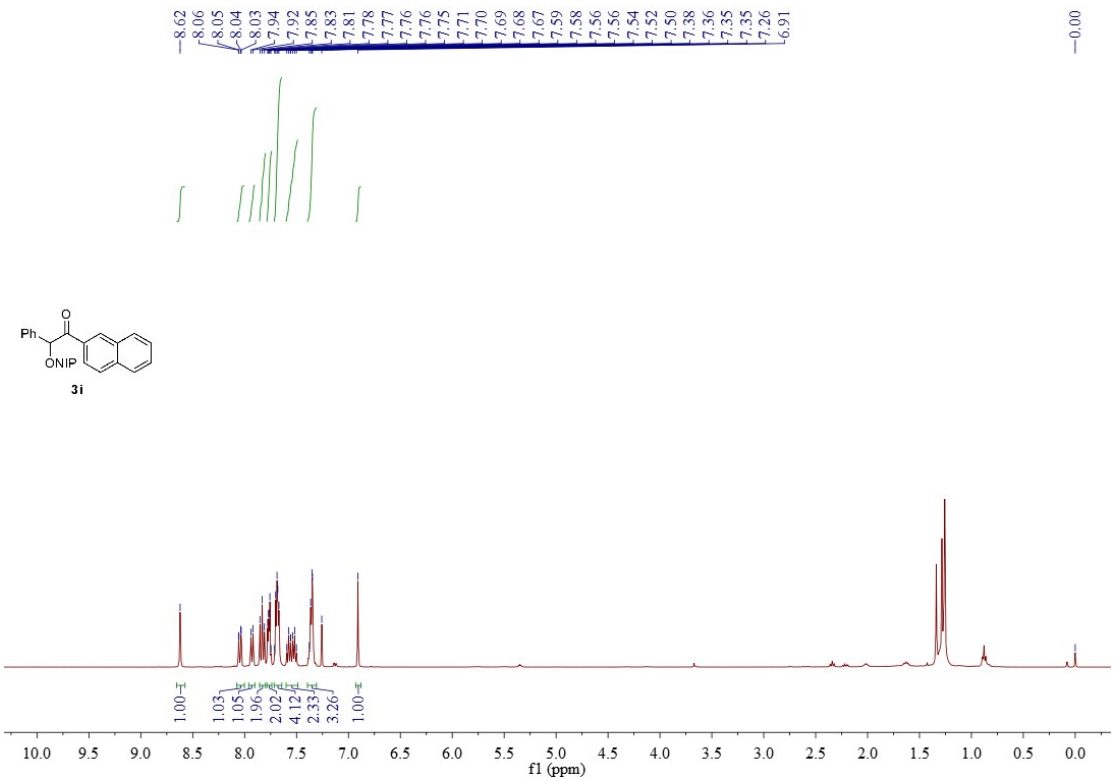
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3h**



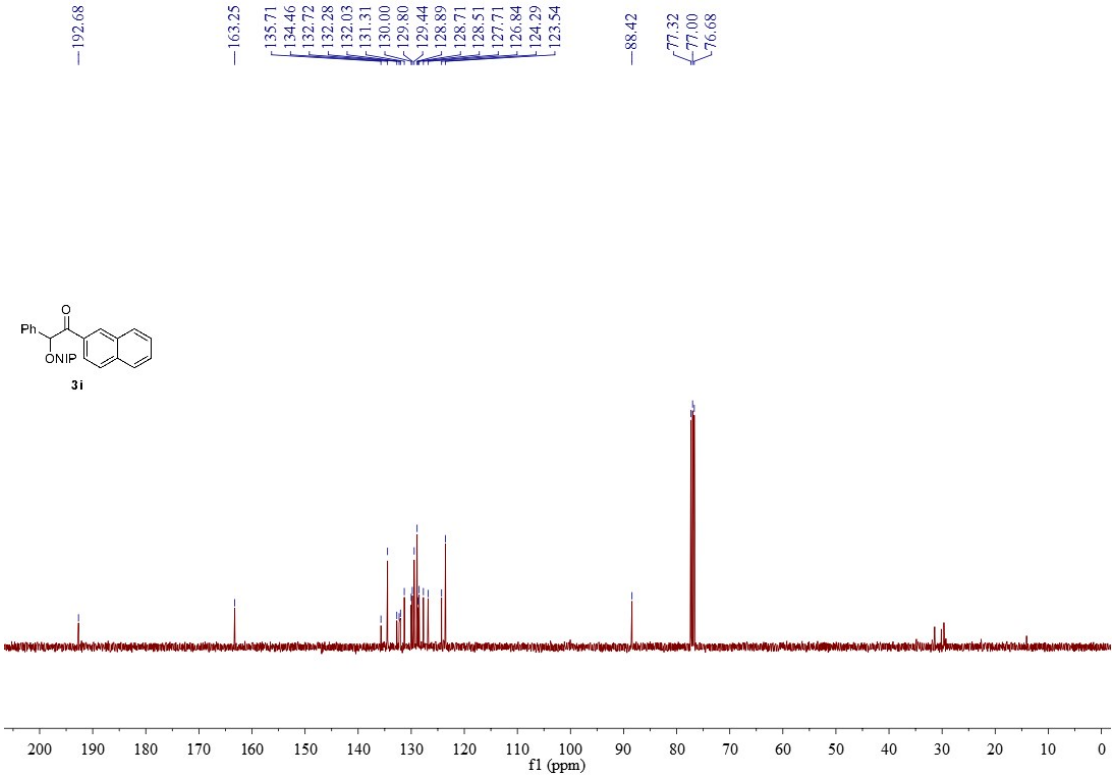
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3h**



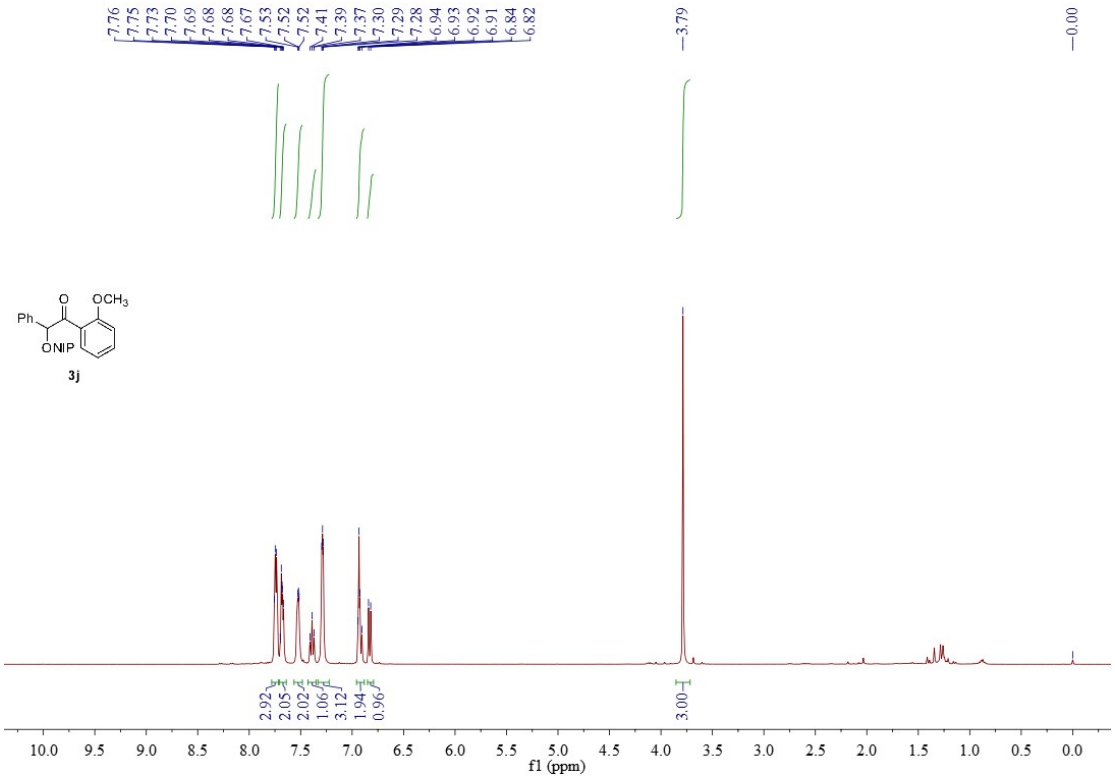
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3i**



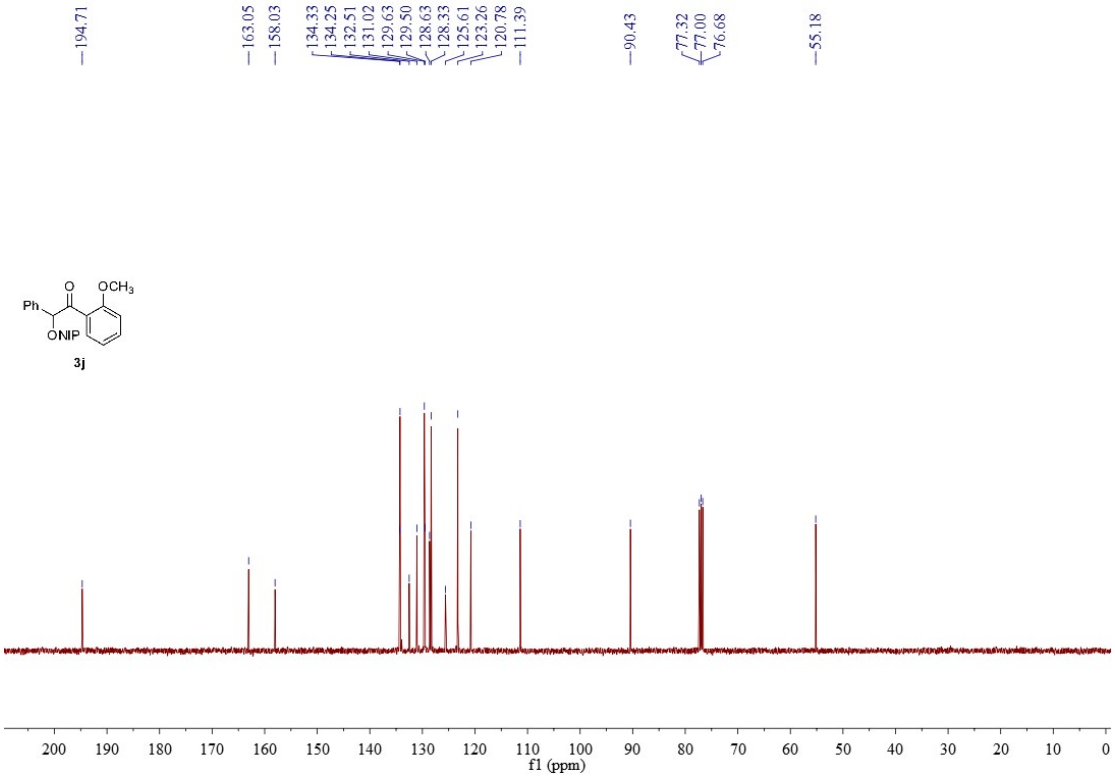
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3i**



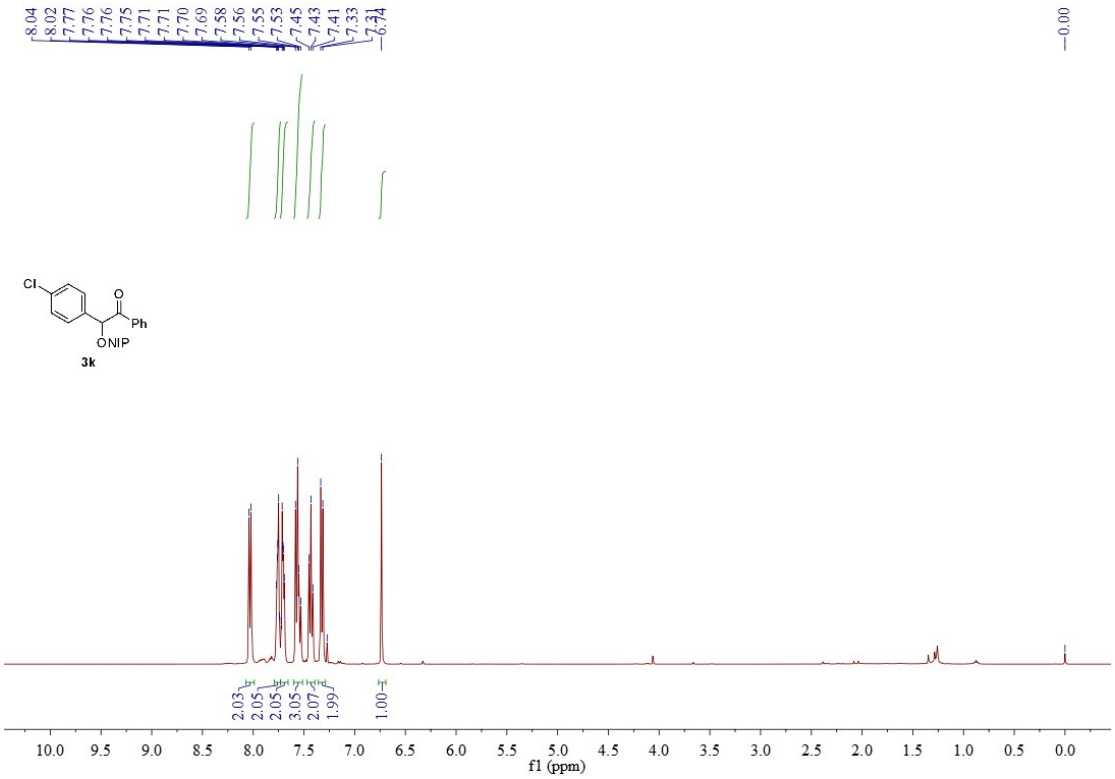
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3j**



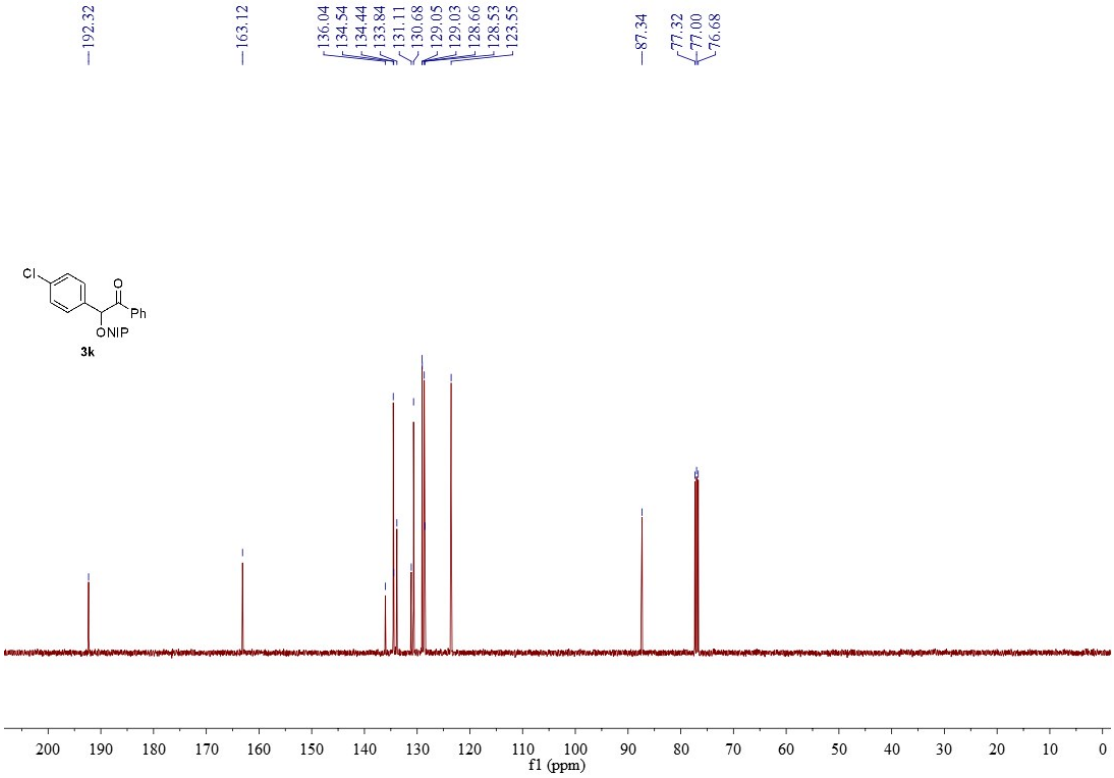
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3j**



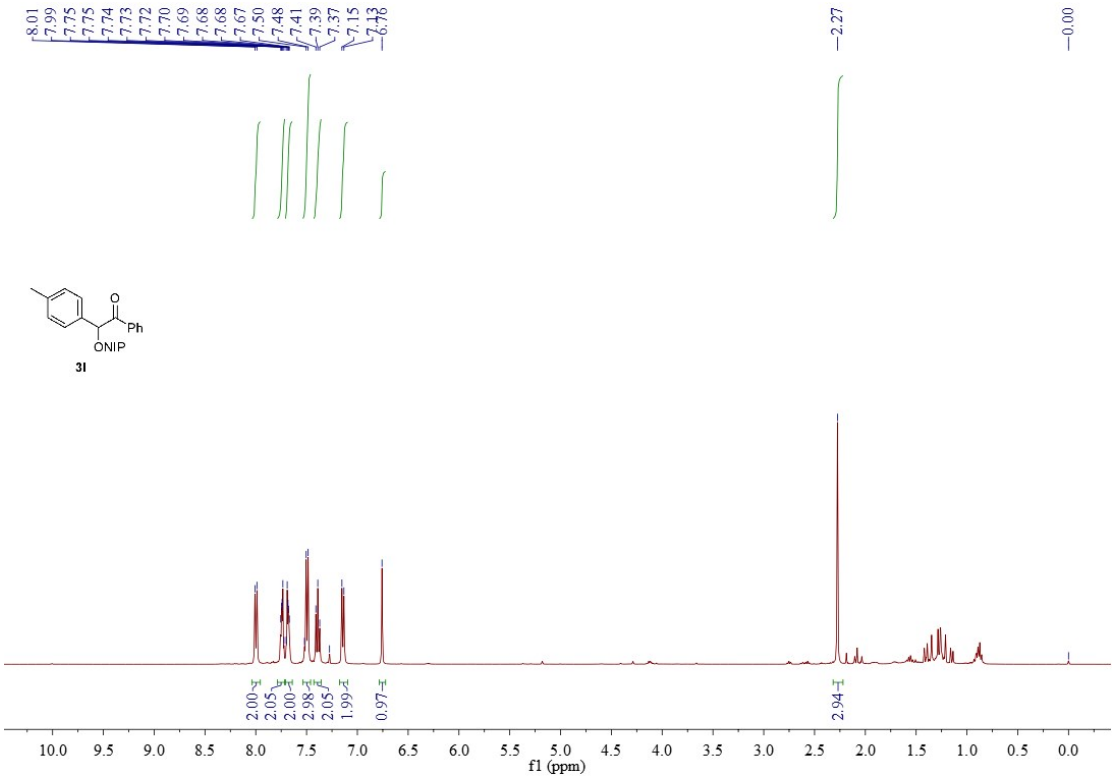
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3k**



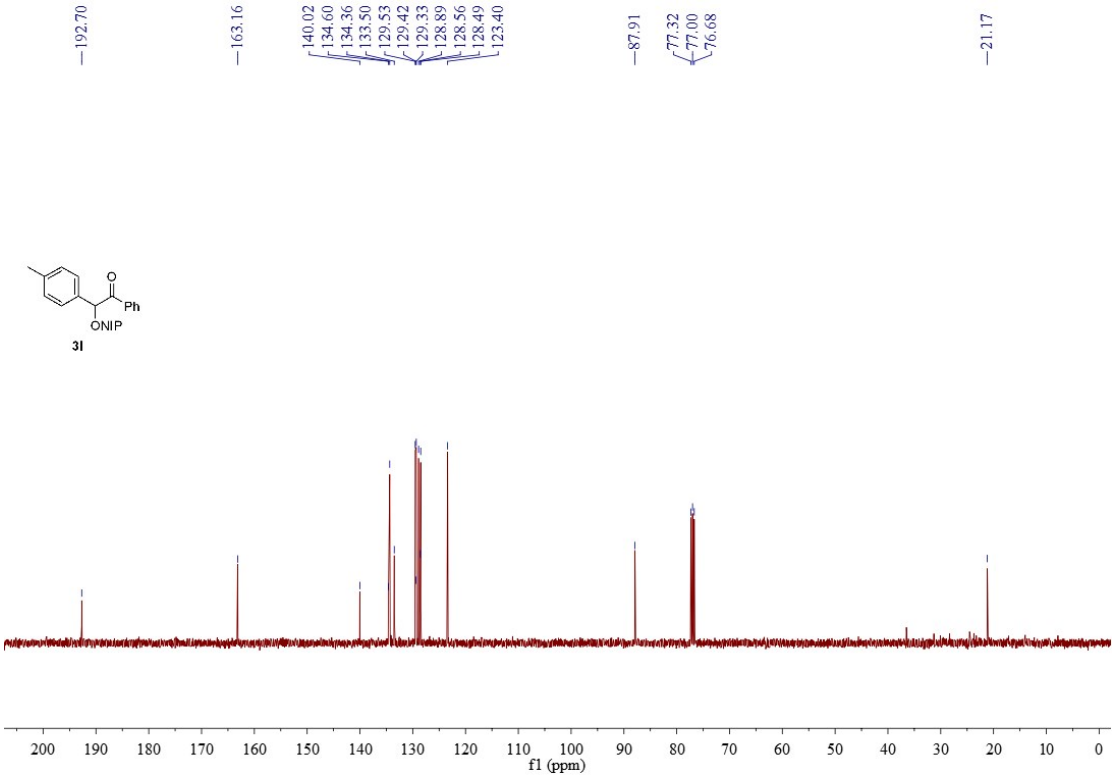
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3k**



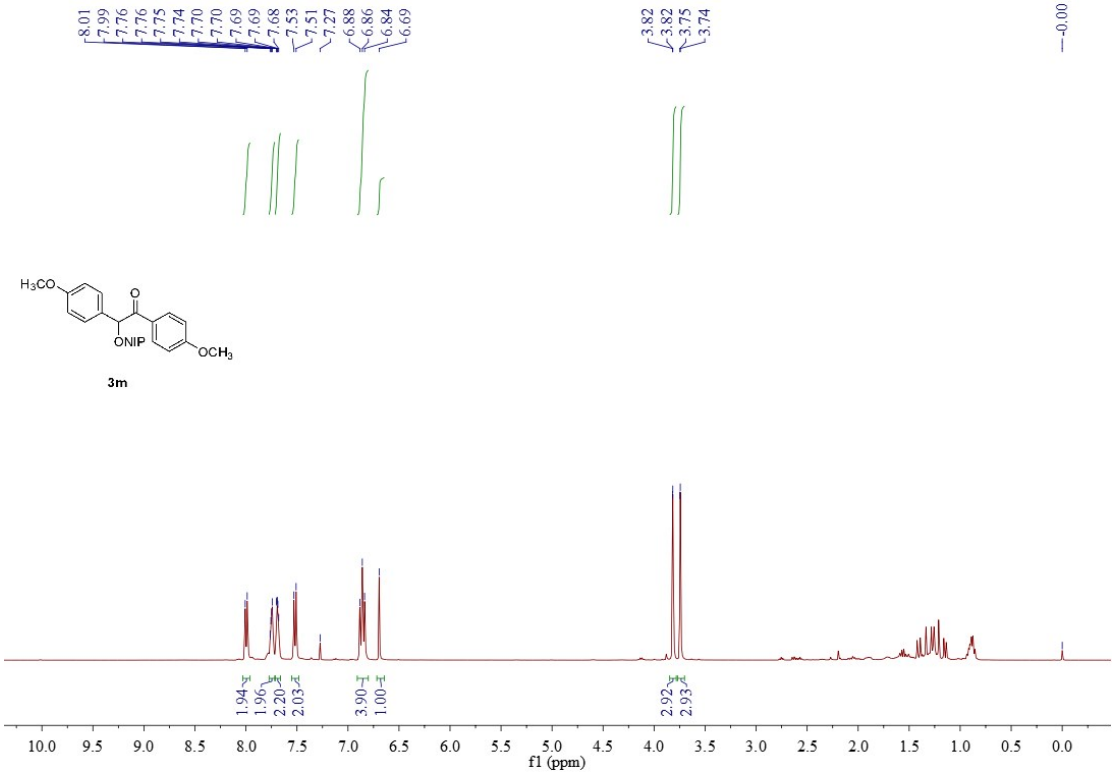
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3l**



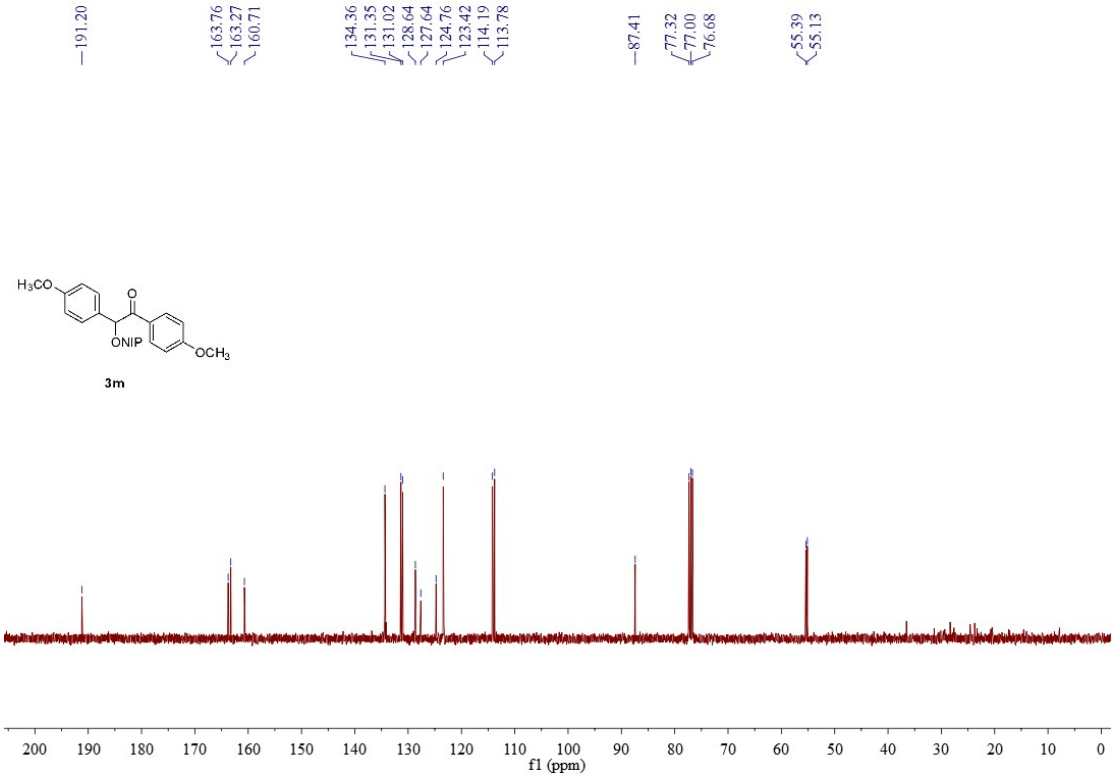
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3l**



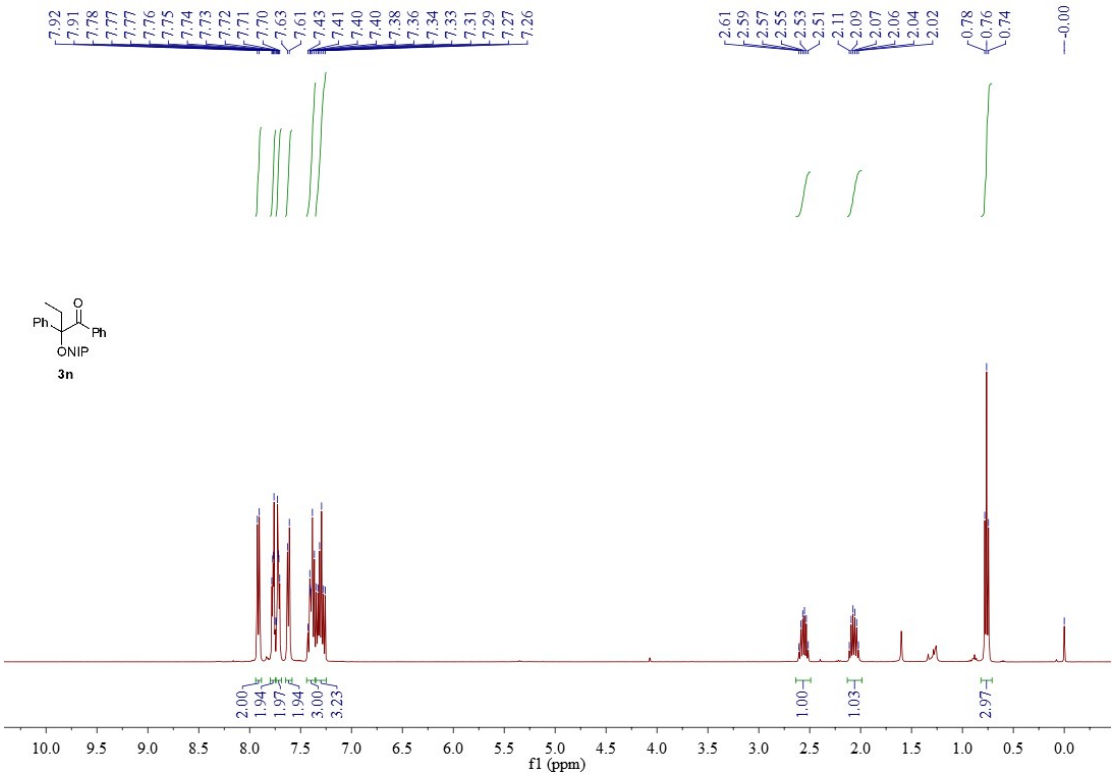
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3m**



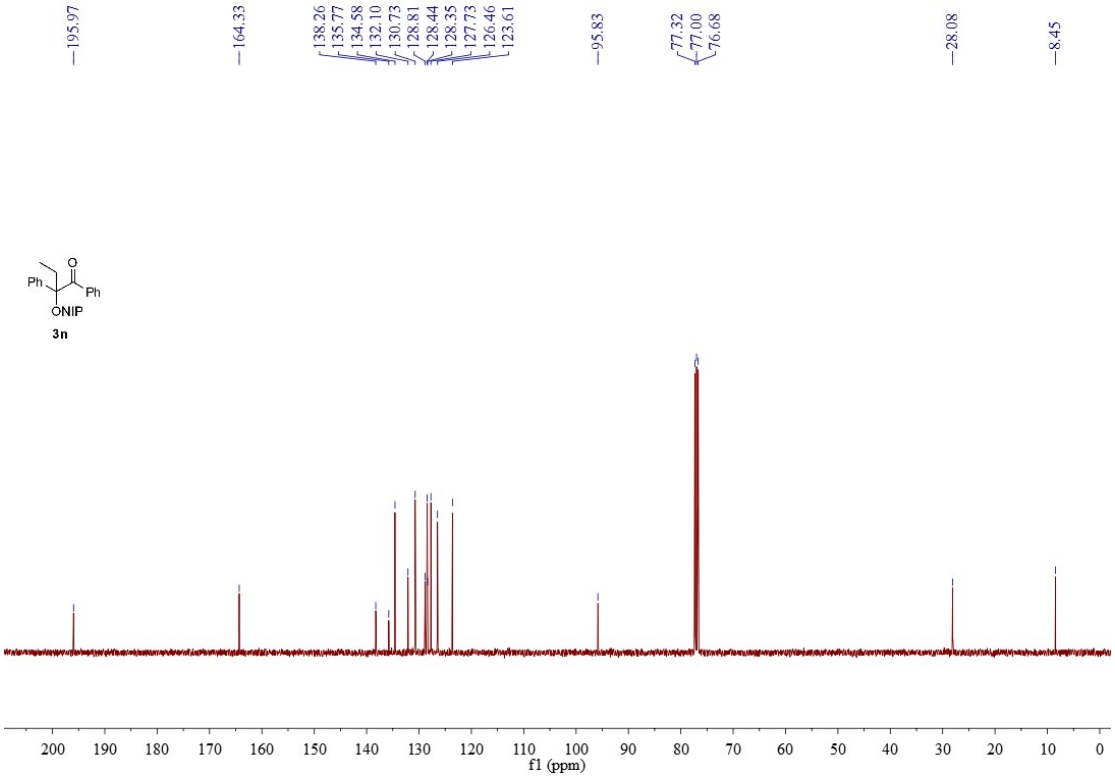
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3m**



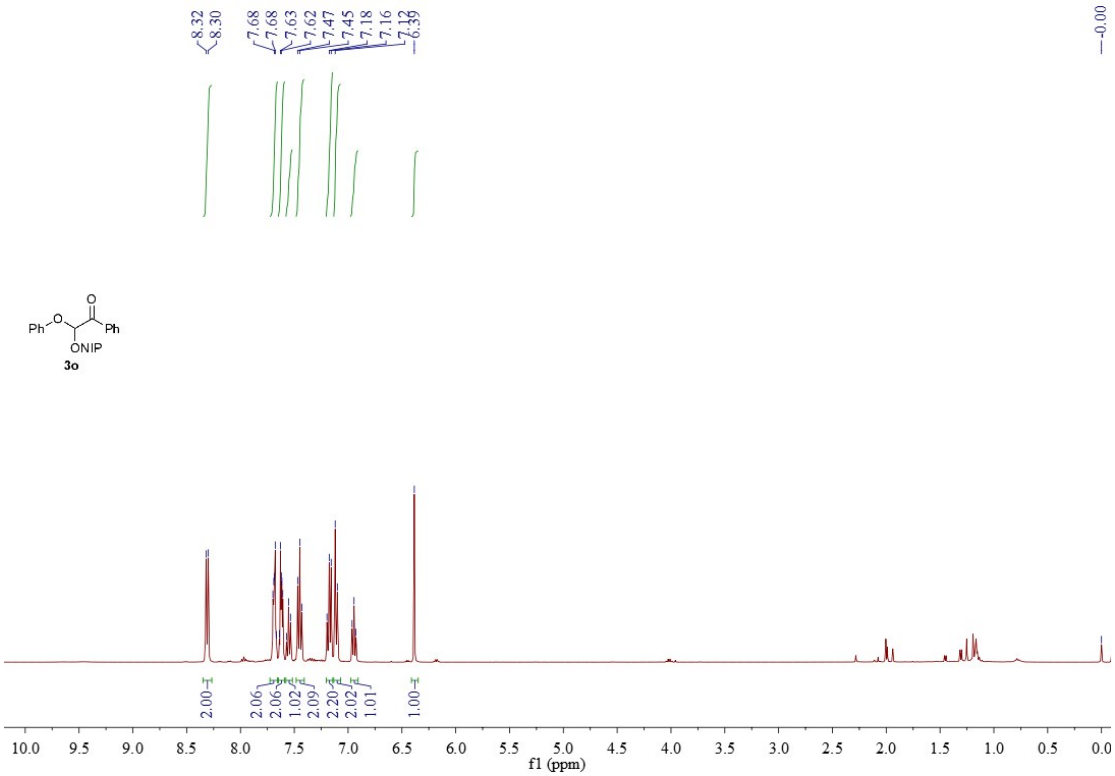
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3n**



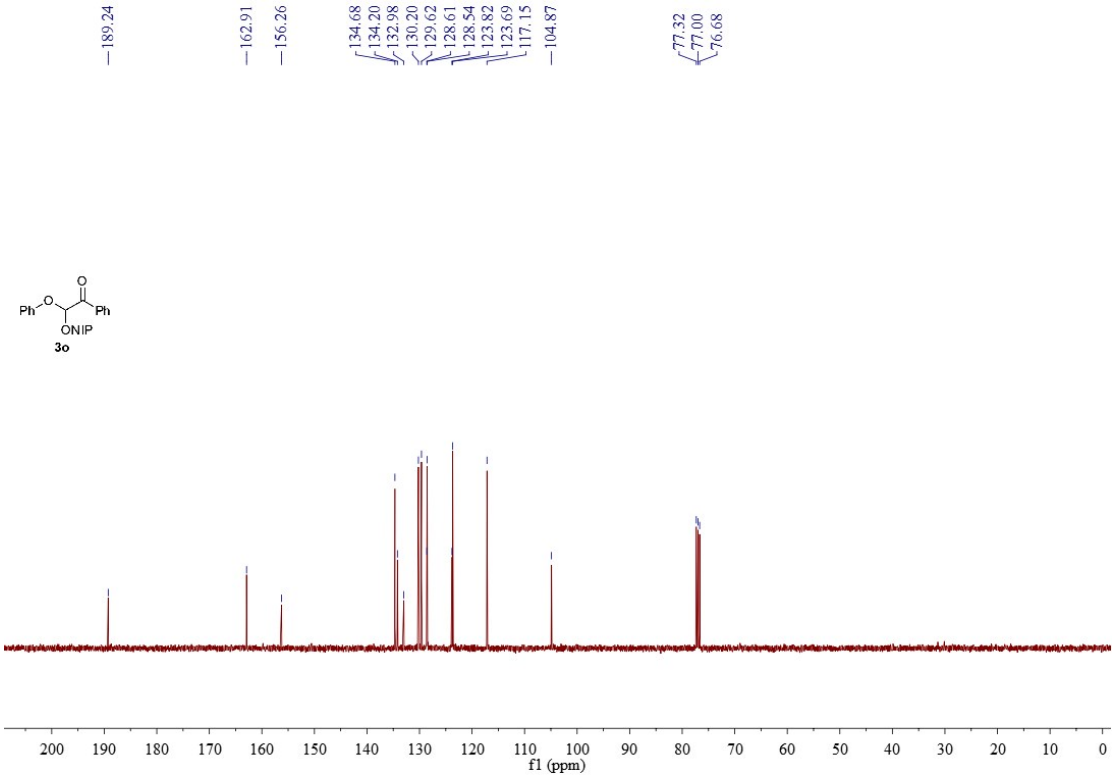
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3n**



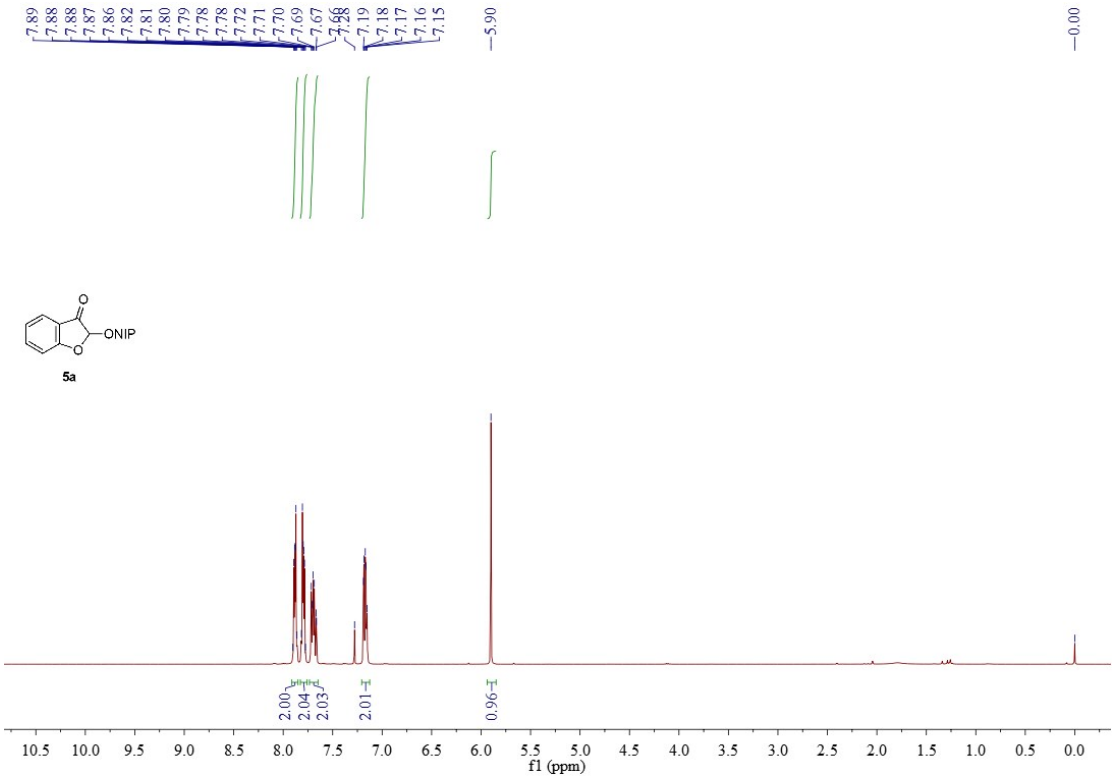
^{1}H NMR (400MHz, $CDCl_3$) spectra of **3o**



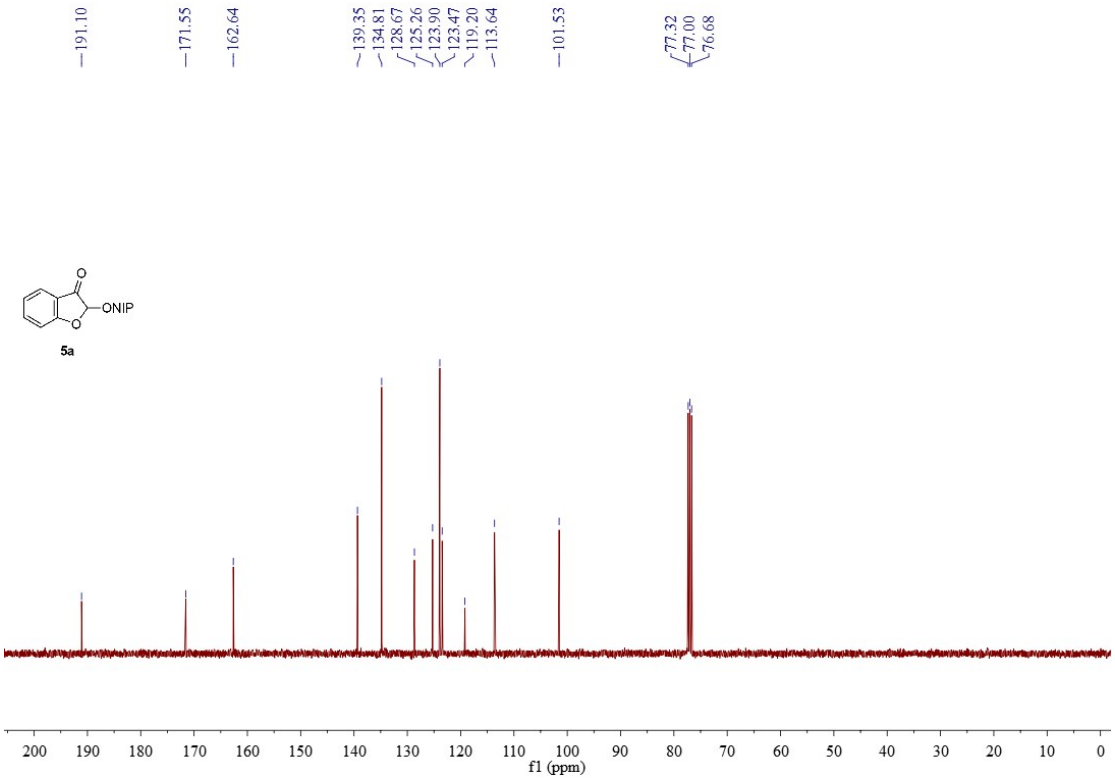
^{13}C NMR (101MHz, $CDCl_3$) spectra of **3o**



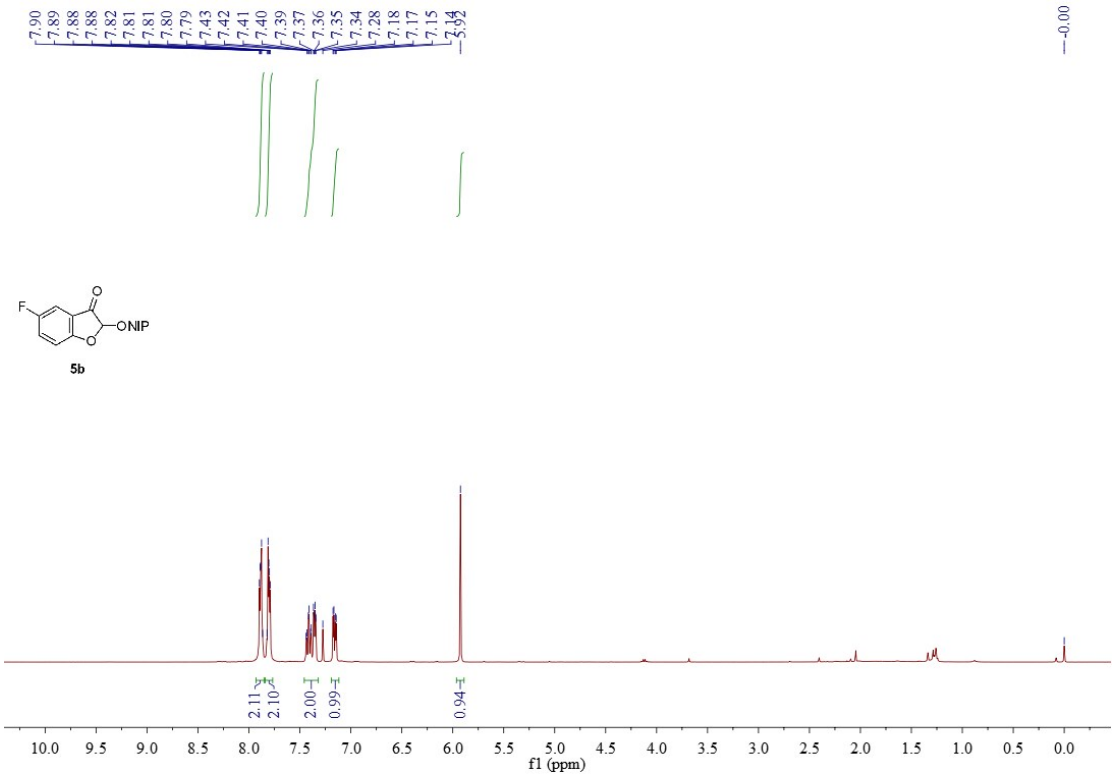
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5a**



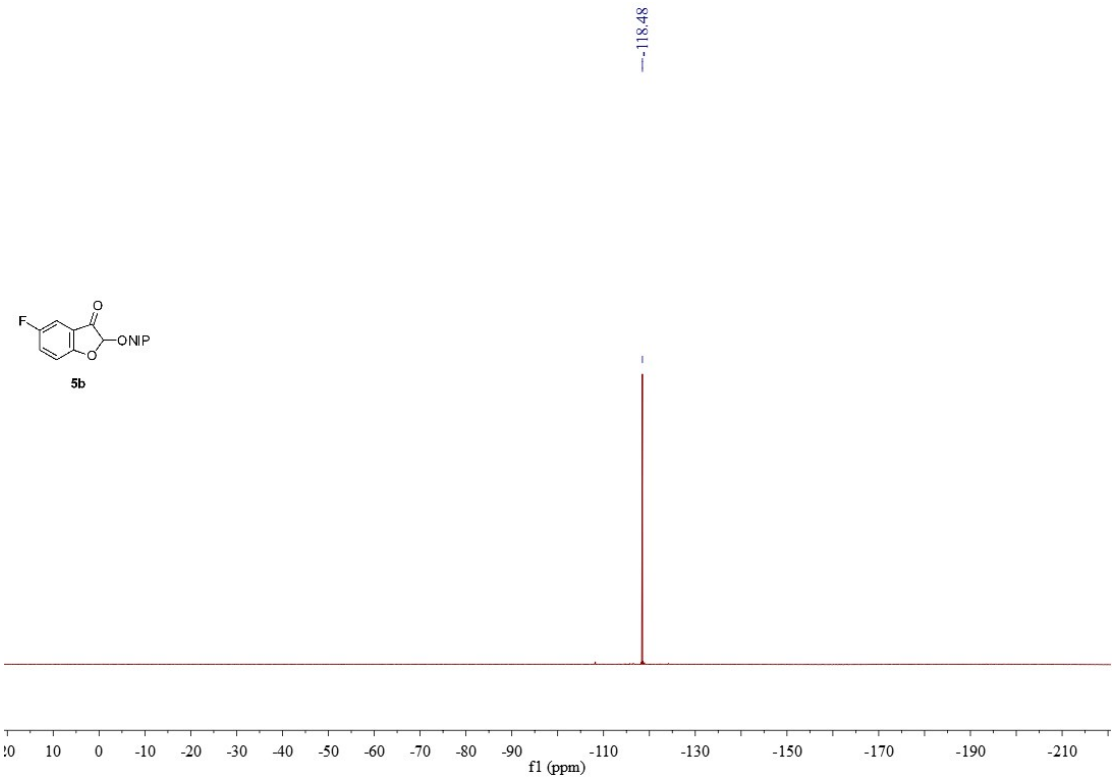
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5a**



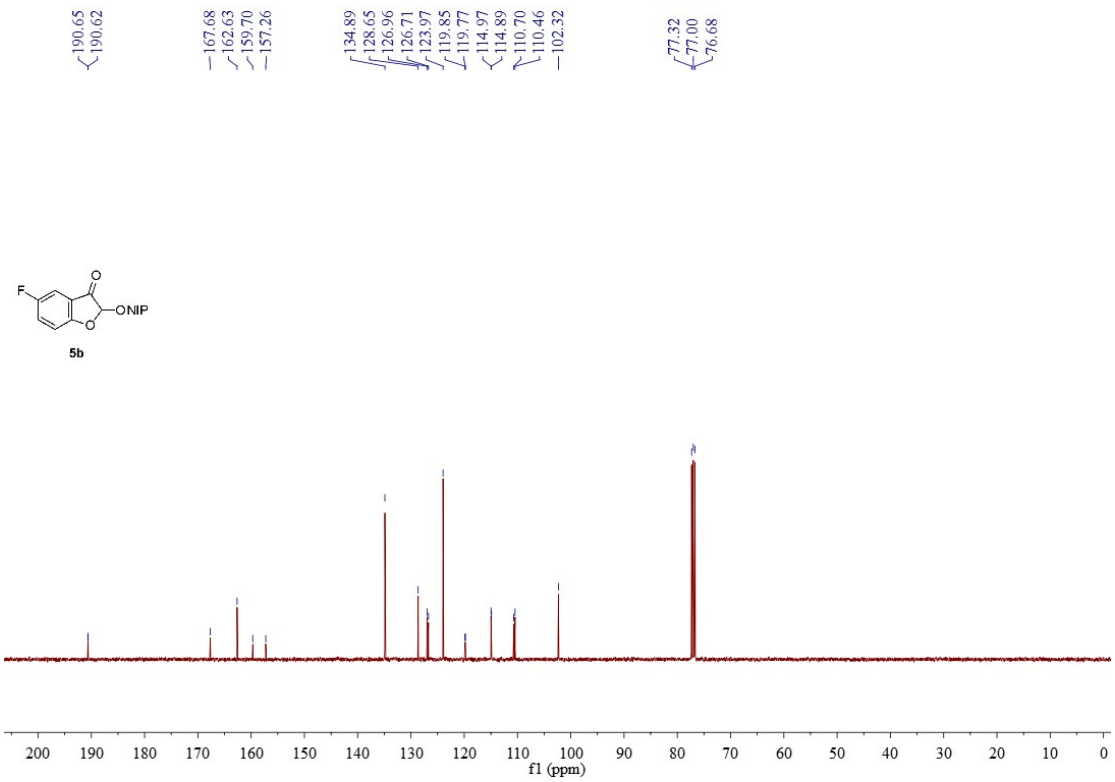
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5b**



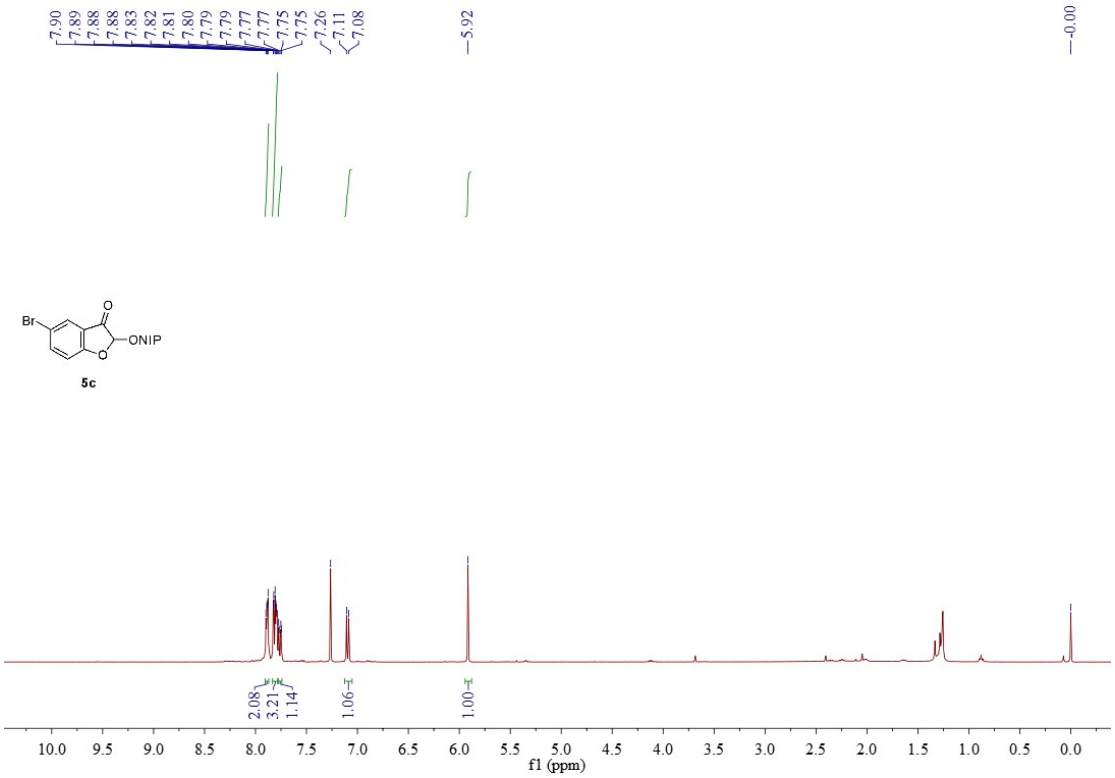
^{19}F NMR (376 MHz, $CDCl_3$) spectra of **5b**



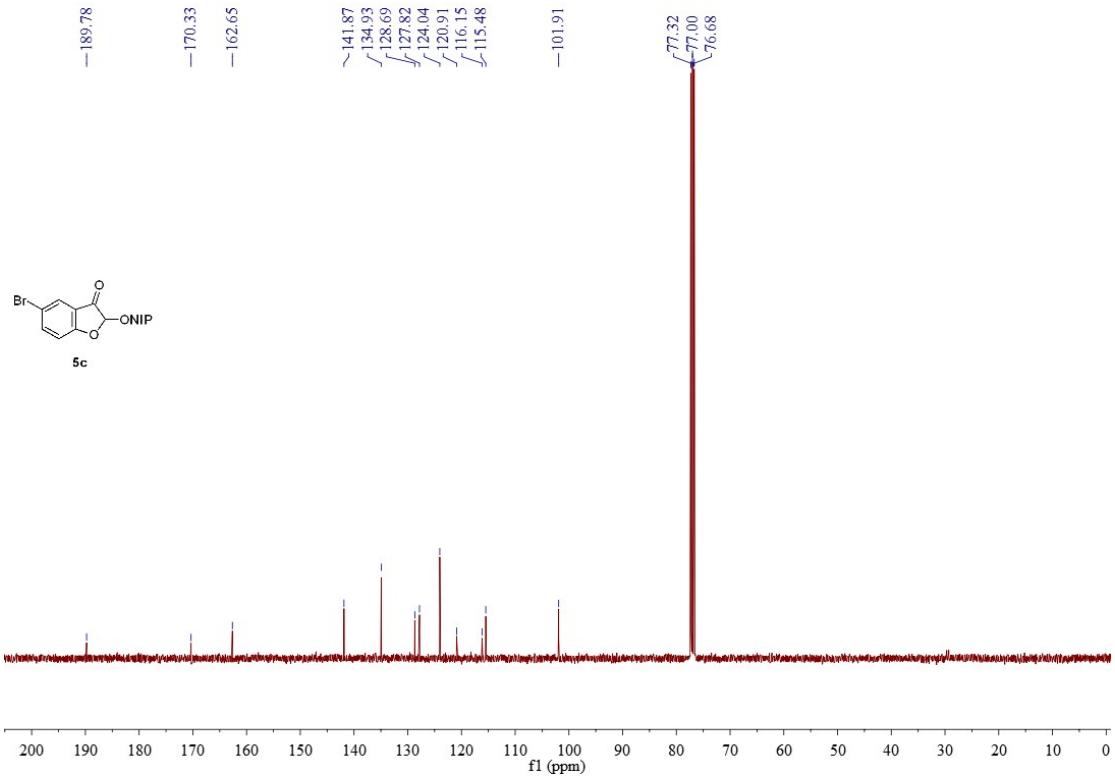
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5b**



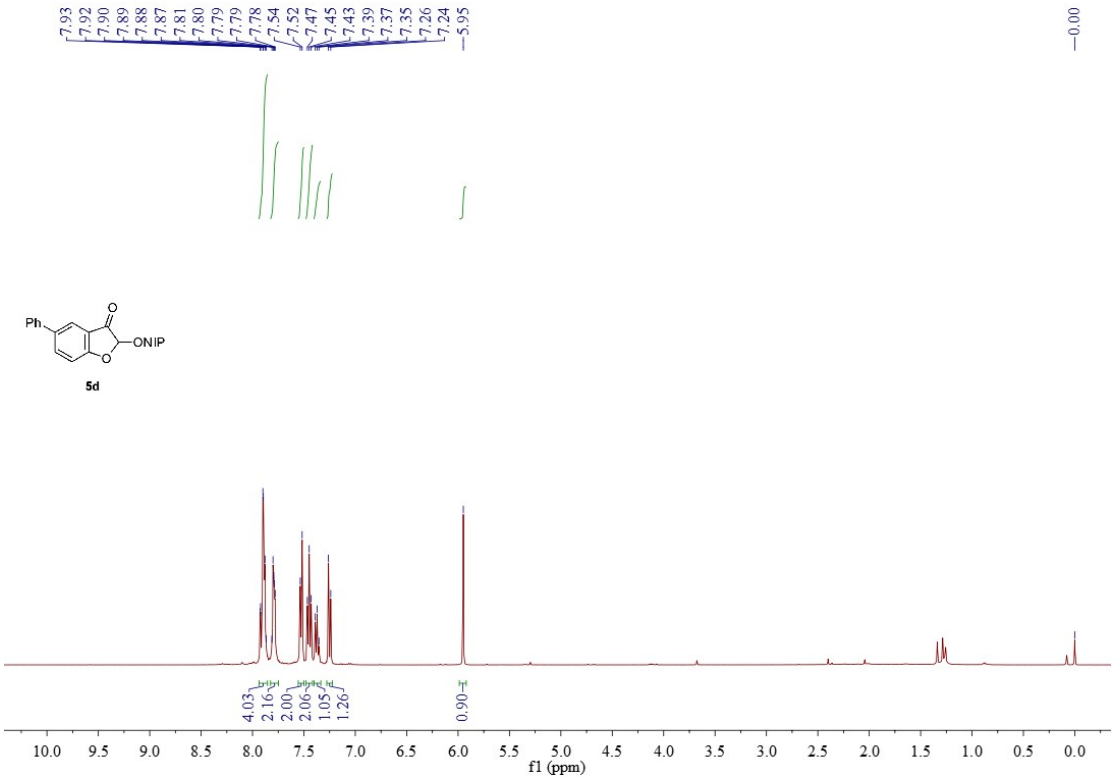
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5c**



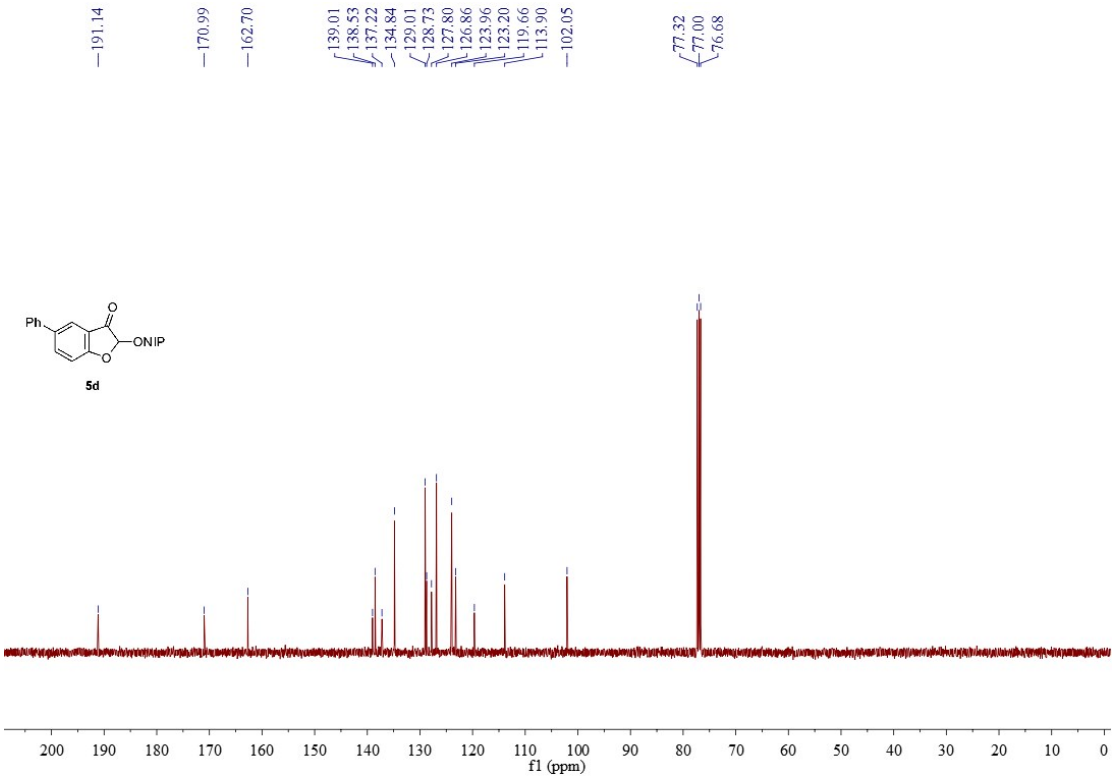
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5c**



^{1}H NMR (400MHz, $CDCl_3$) spectra of **5d**



^{13}C NMR (101MHz, $CDCl_3$) spectra of **5d**



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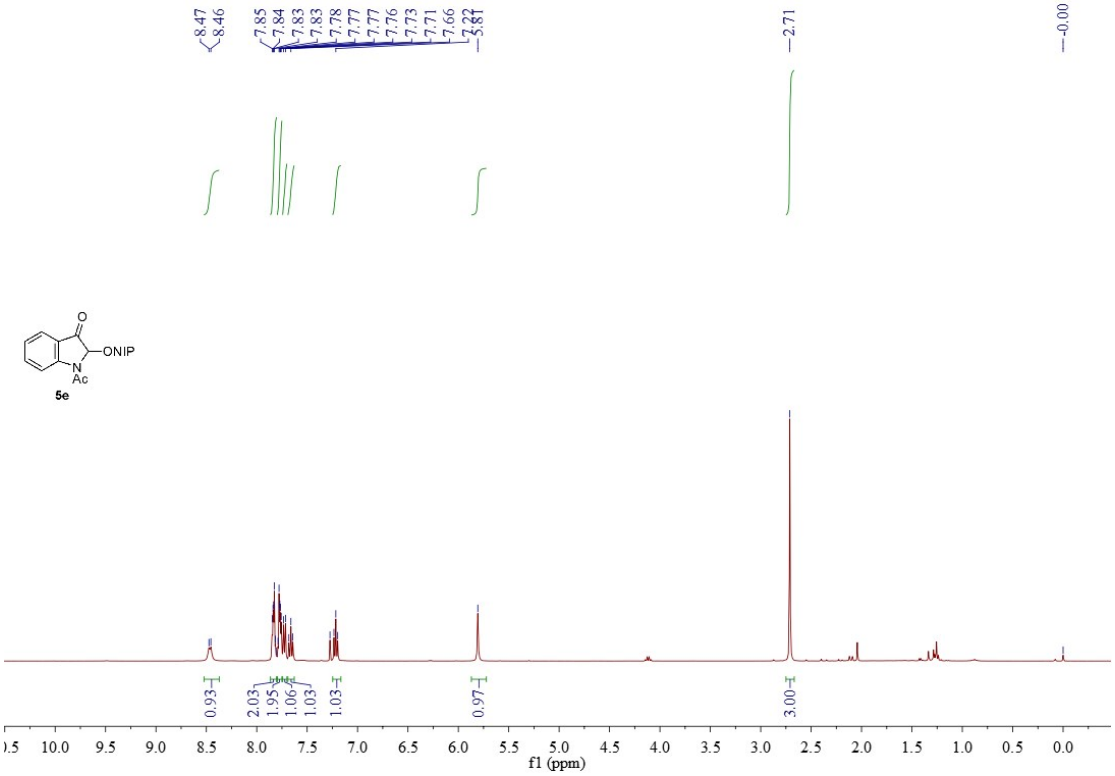
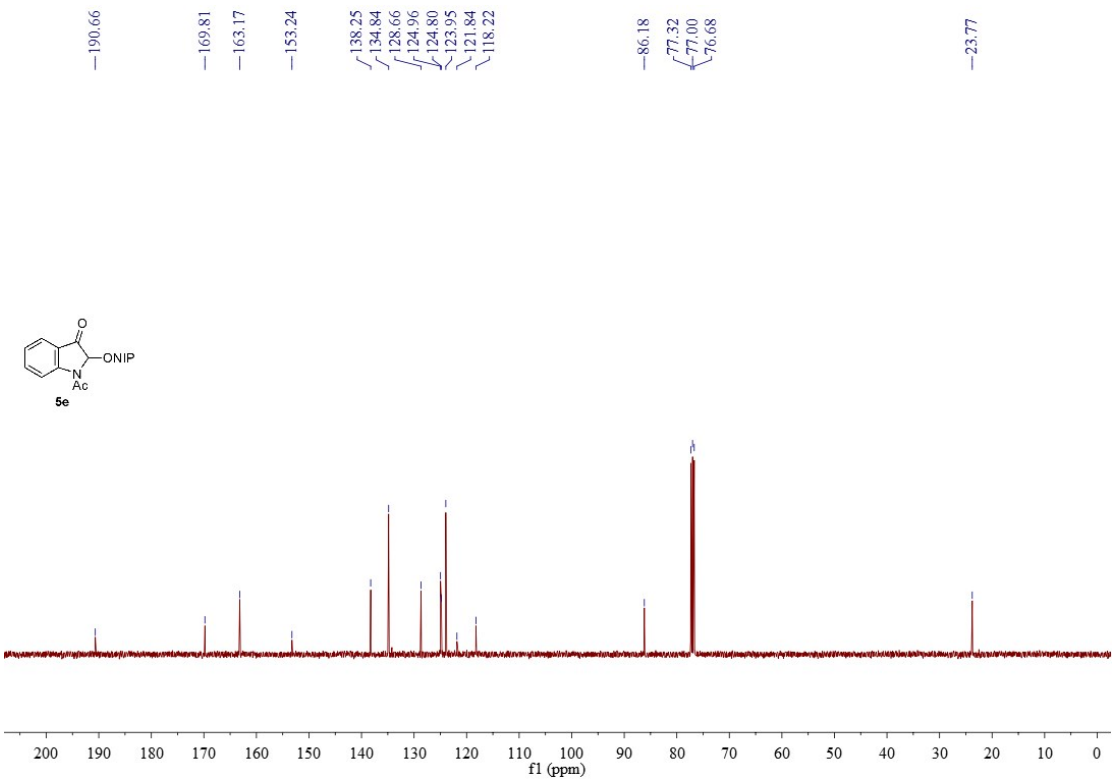
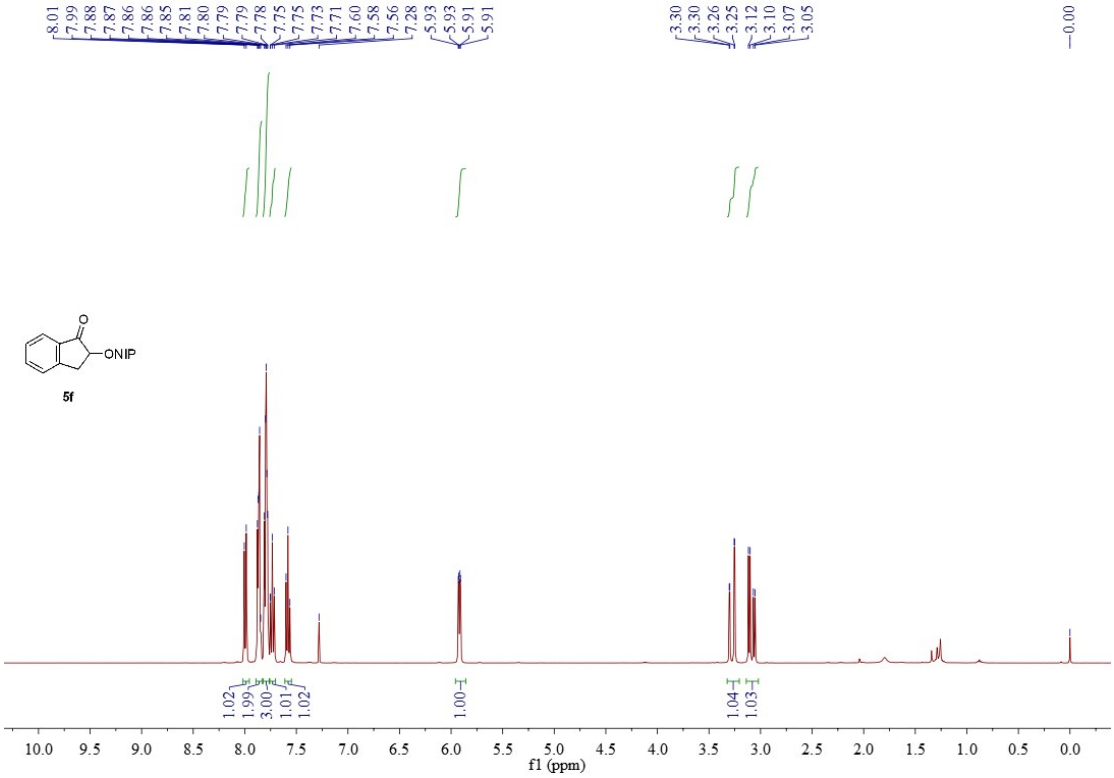


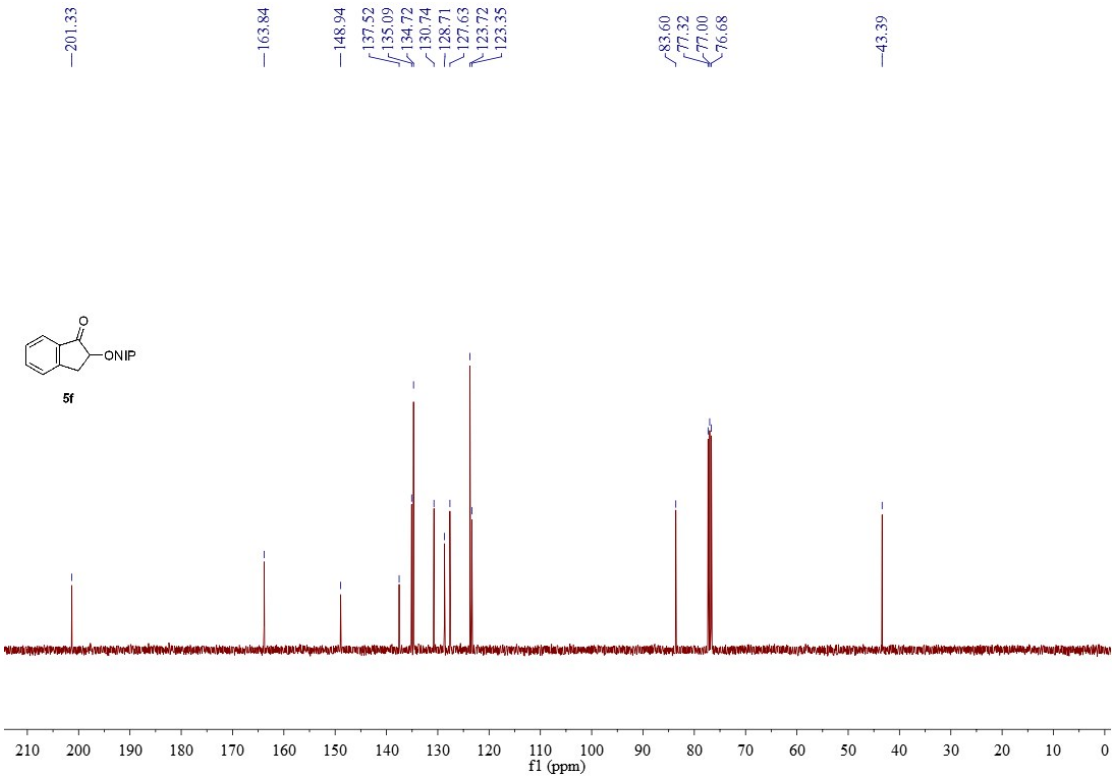
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5e**



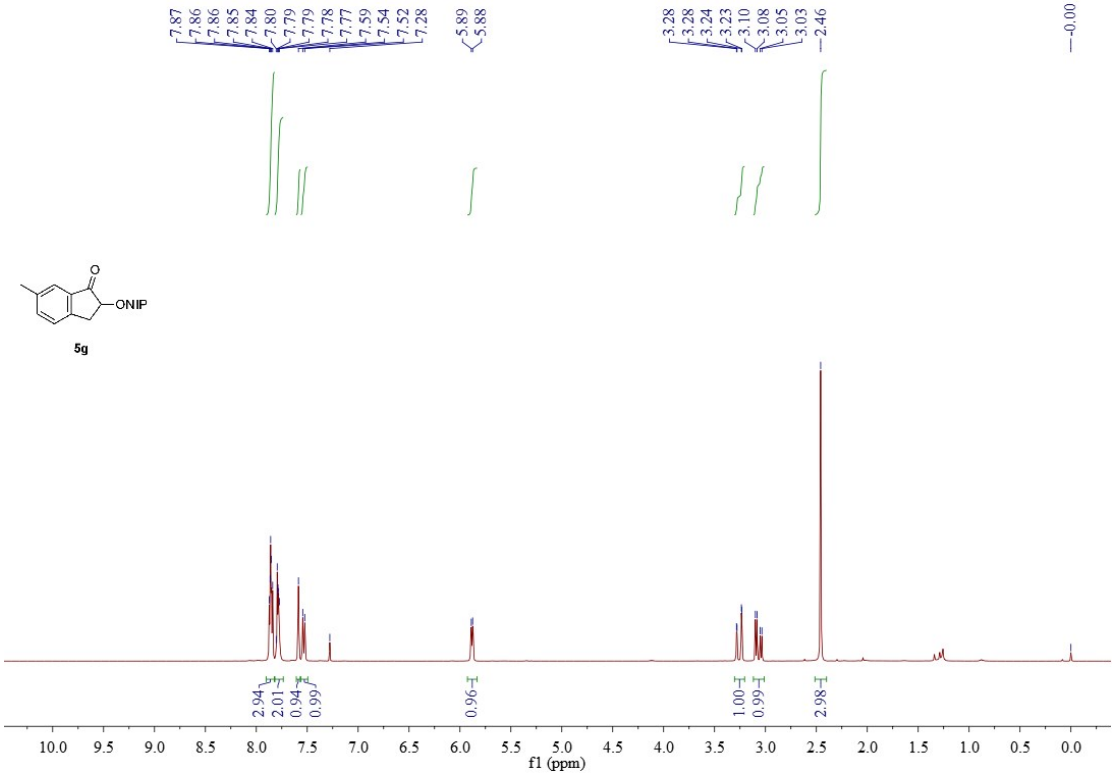
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5f**



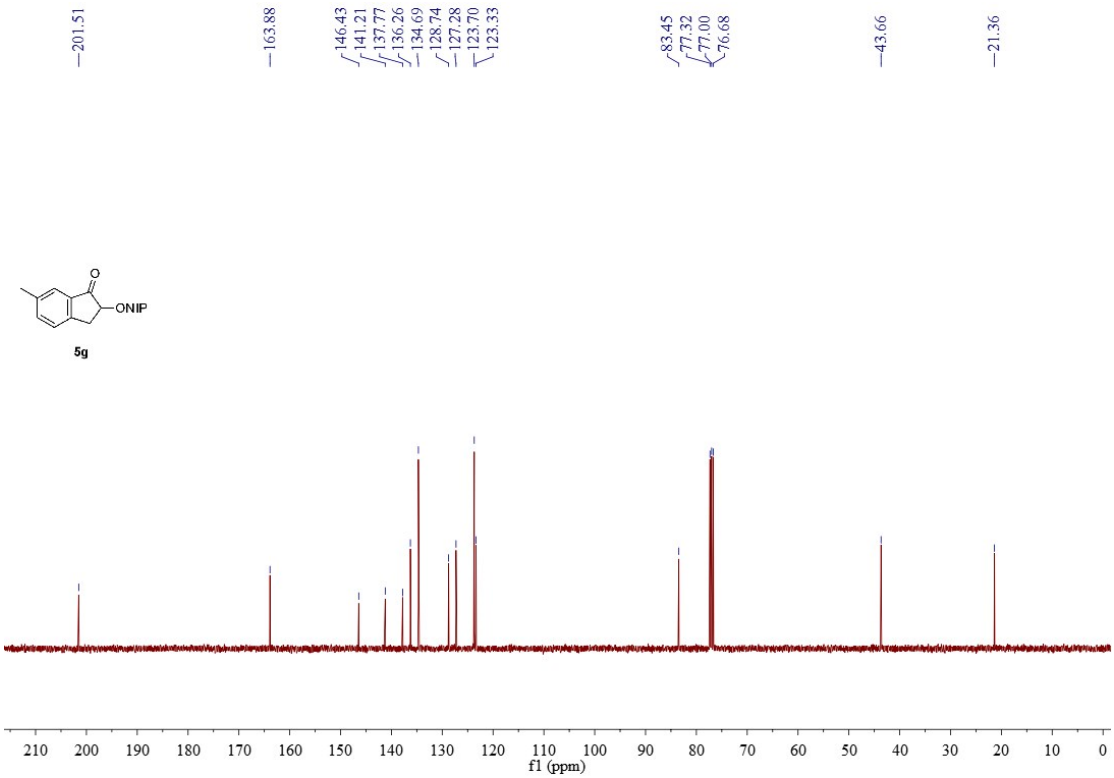
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5f**



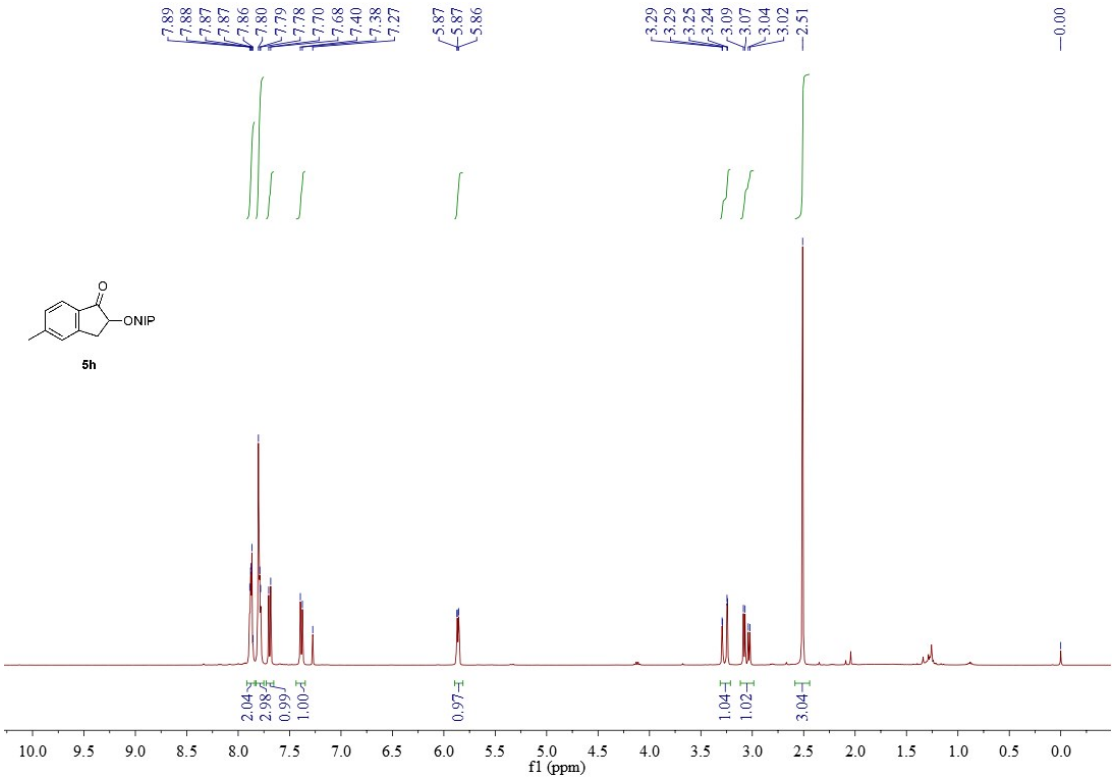
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5g**



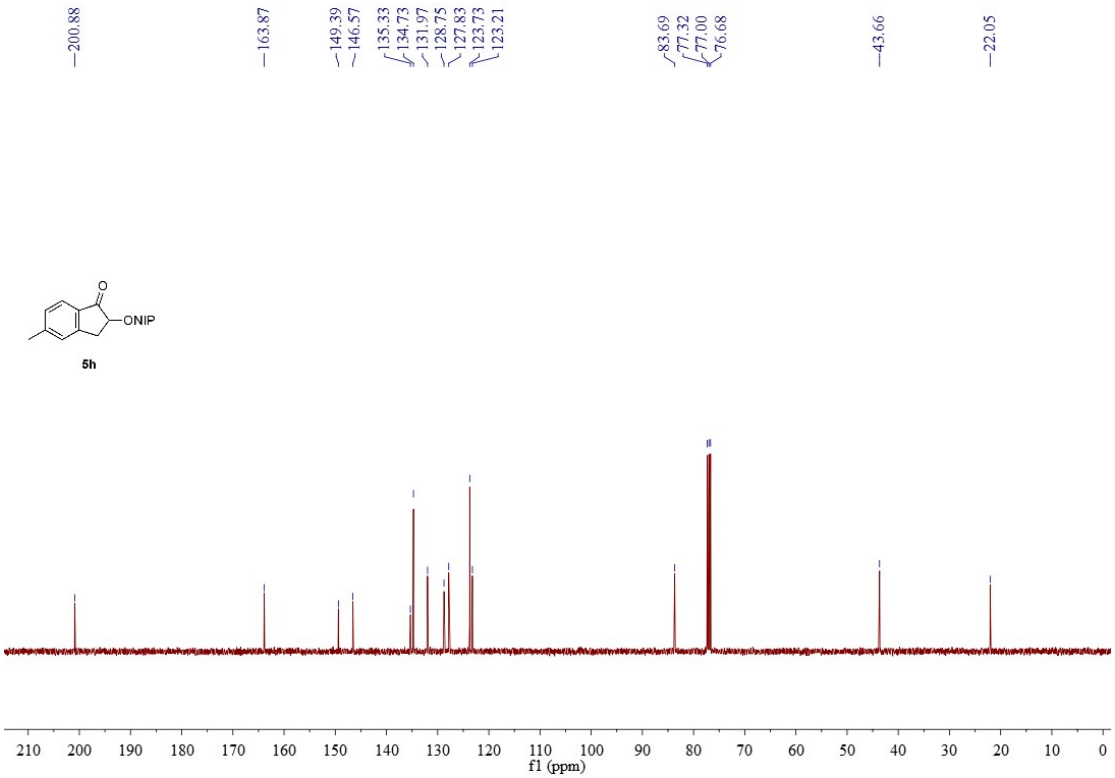
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5g**



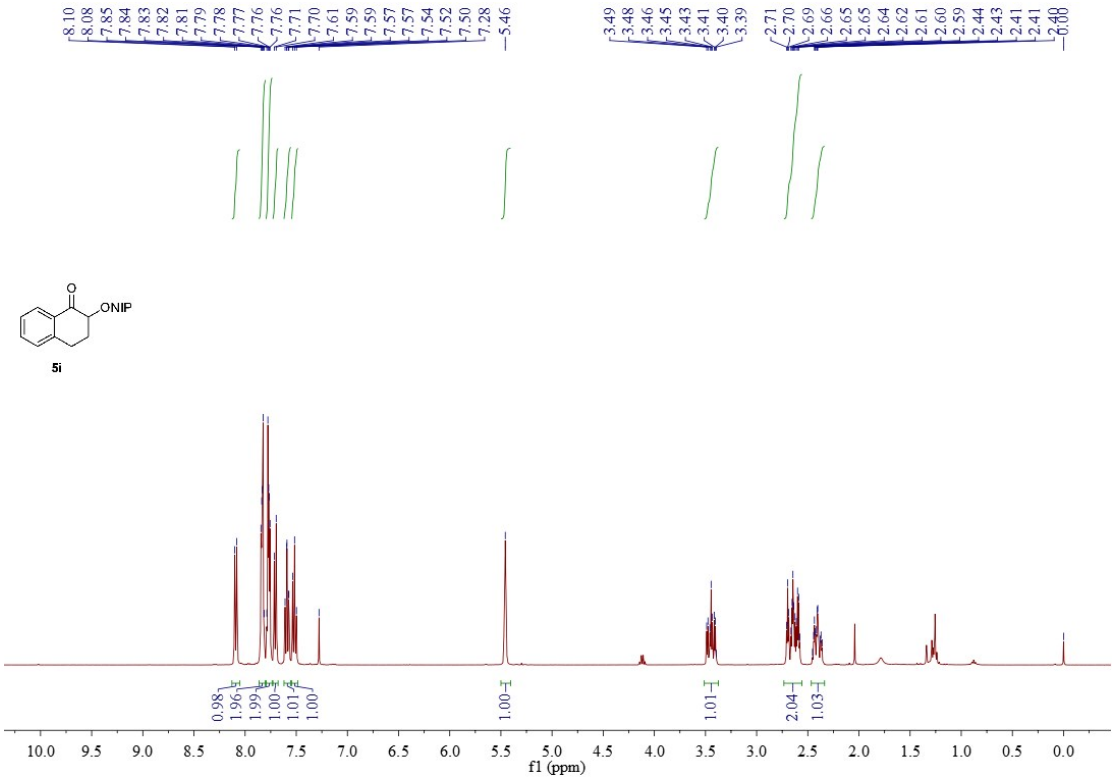
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5h**



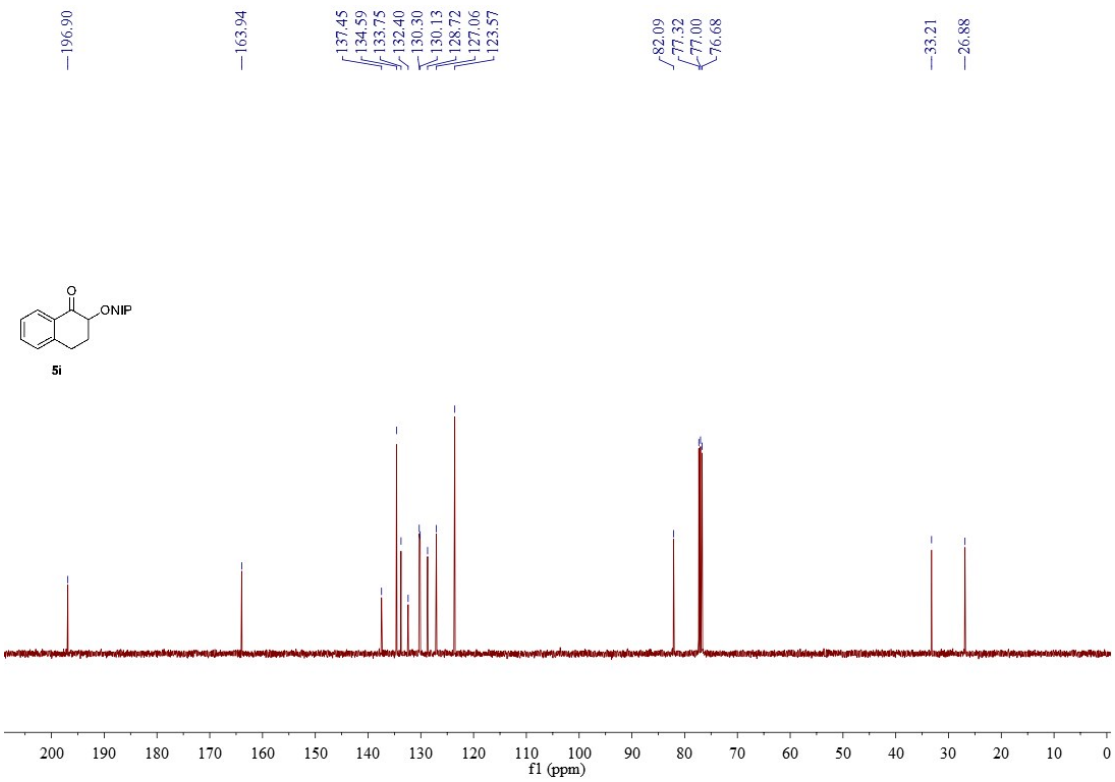
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5h**



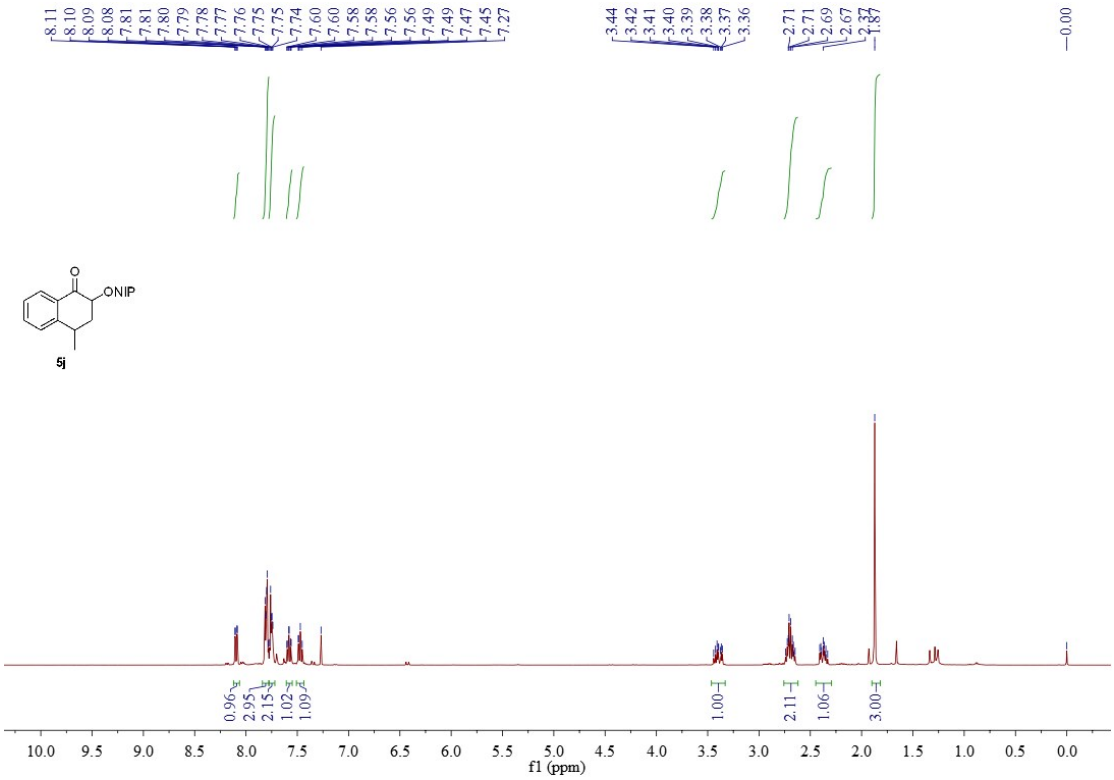
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5i**



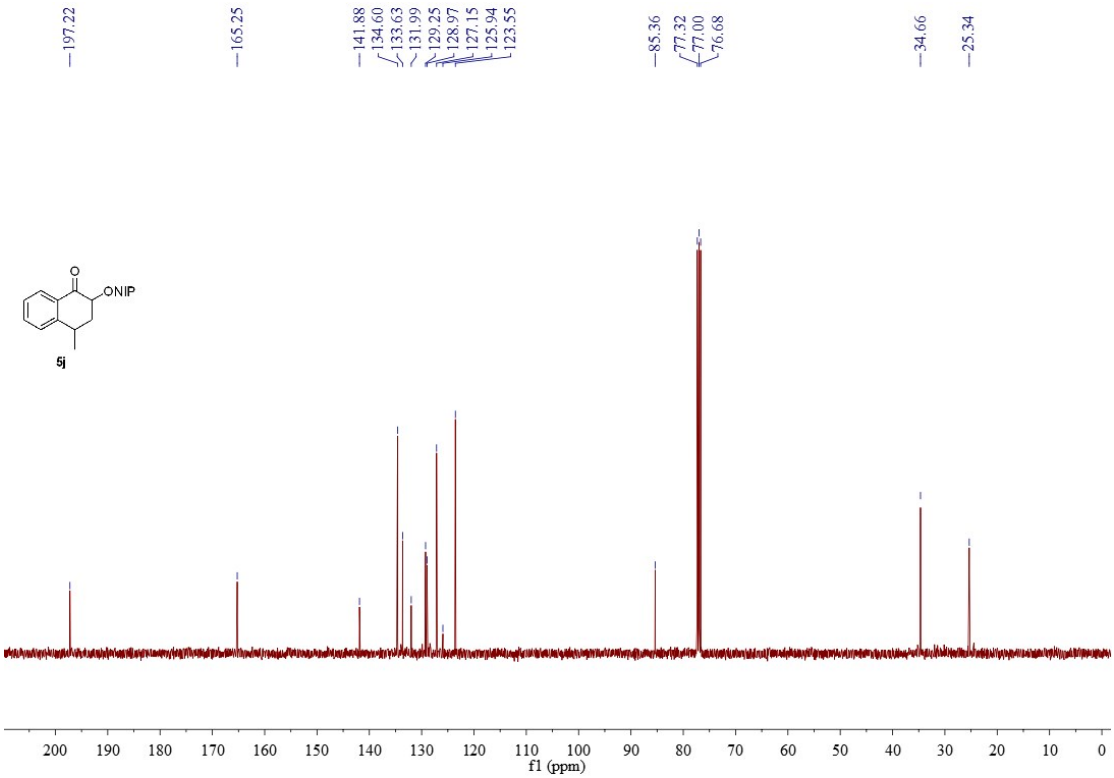
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5i**



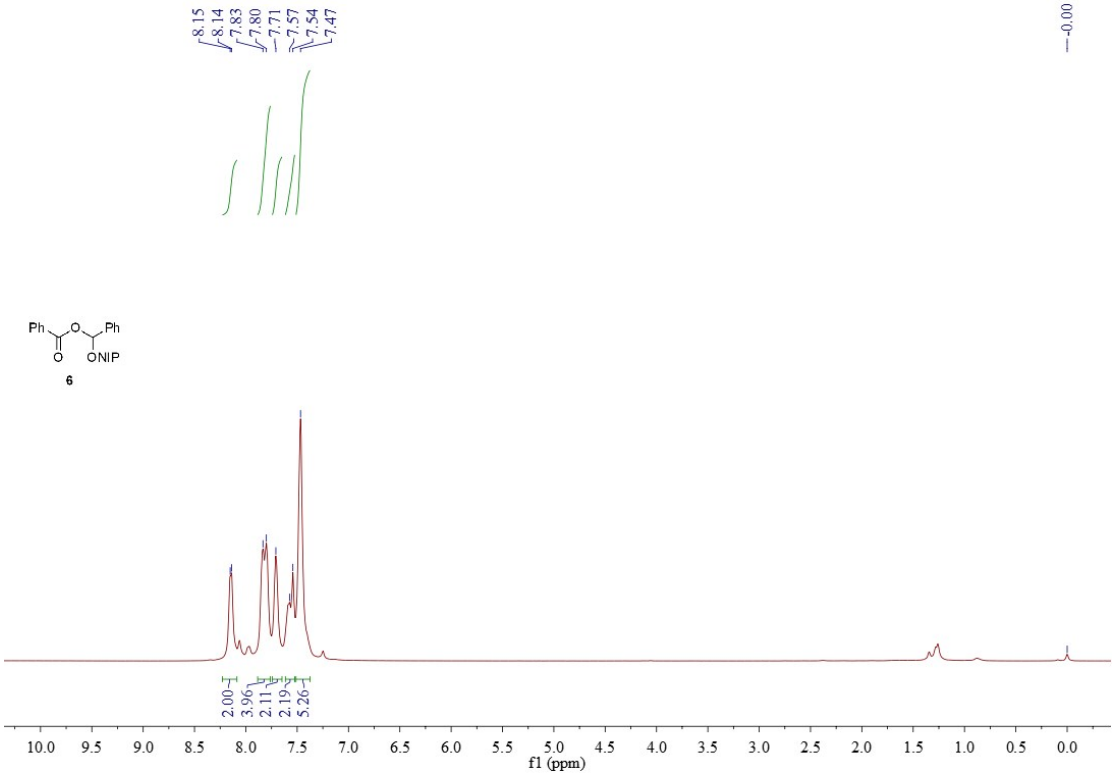
^{1}H NMR (400MHz, $CDCl_3$) spectra of **5j**



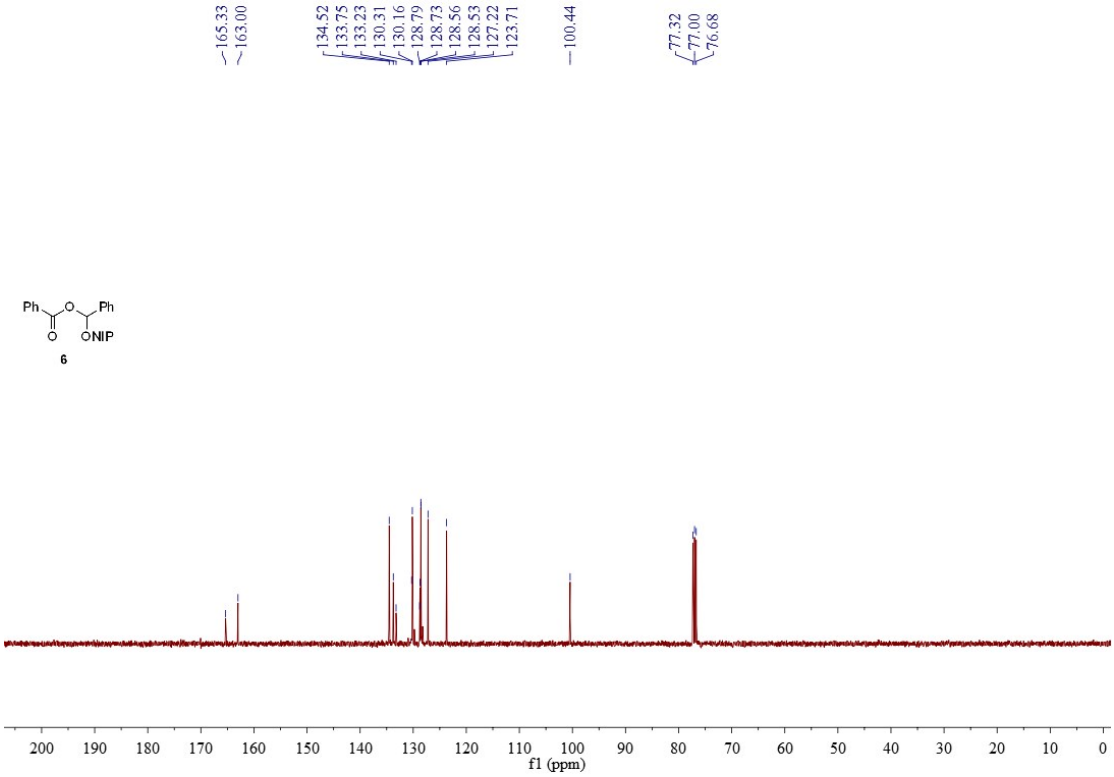
^{13}C NMR (101MHz, $CDCl_3$) spectra of **5j**



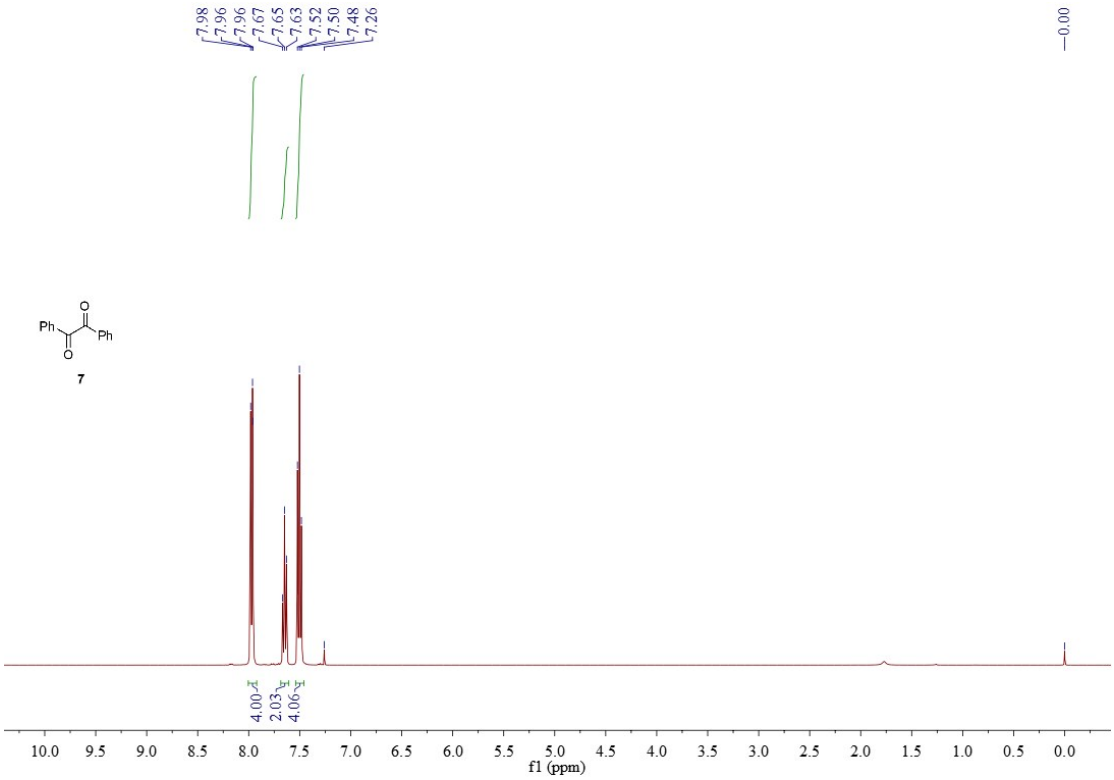
^{1}H NMR 400MHz, $CDCl_3$) spectra of **6**



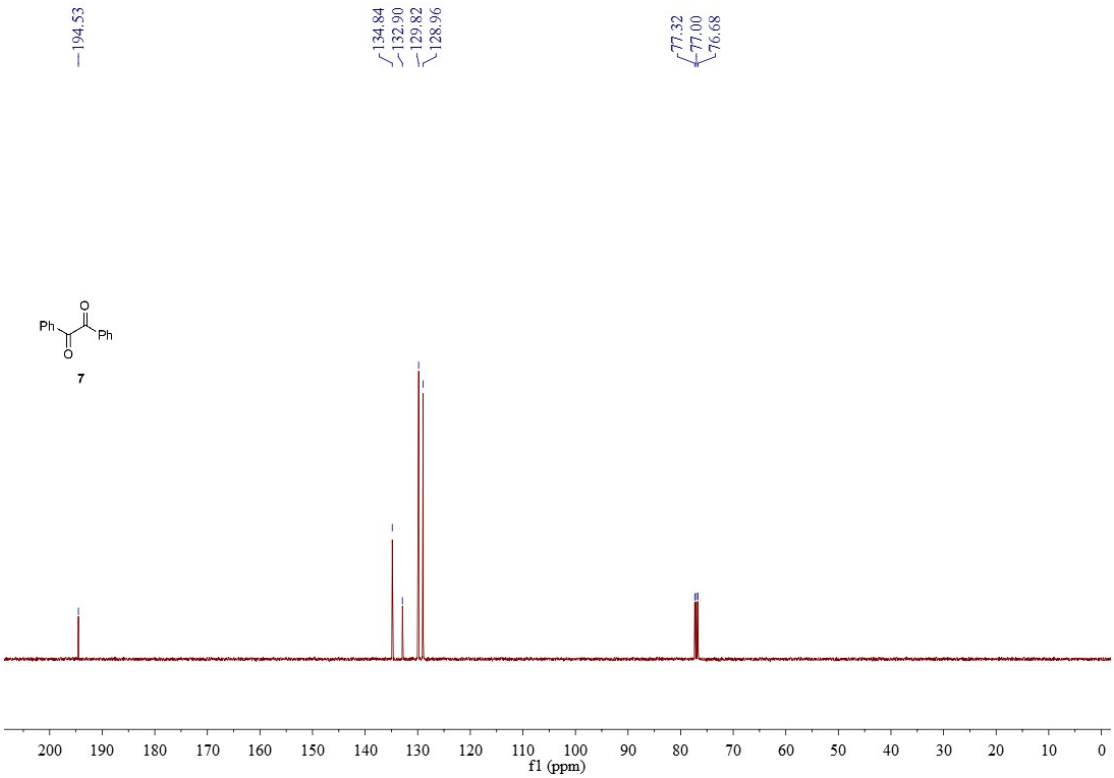
^{13}C NMR (101MHz, $CDCl_3$) spectra of **6**



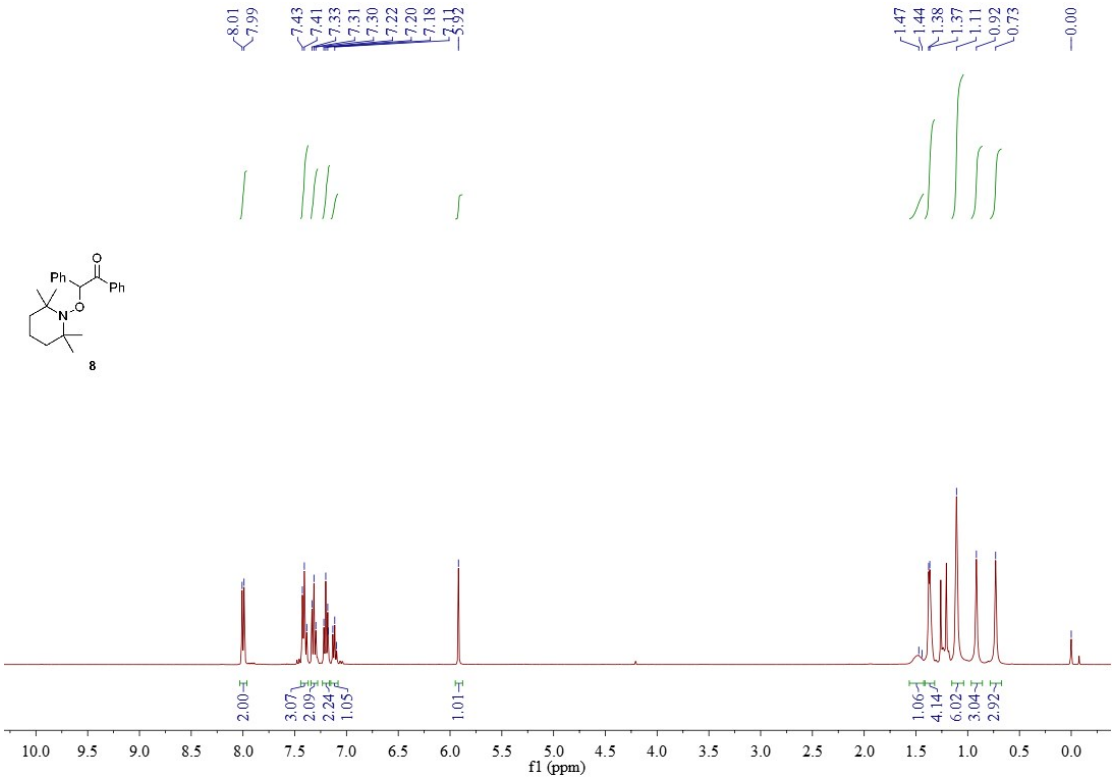
^{1}H NMR (400MHz, $CDCl_3$) spectra of **7**



^{13}C NMR (101MHz, $CDCl_3$) spectra of **7**



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



^{13}C NMR (101MHz, $CDCl_3$) spectra of **8**