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Supporting Information

for

Solvent-dependent chemoselective synthesis of different isoquinolinones mediated by the hypervalent iodine(III) reagent PISA

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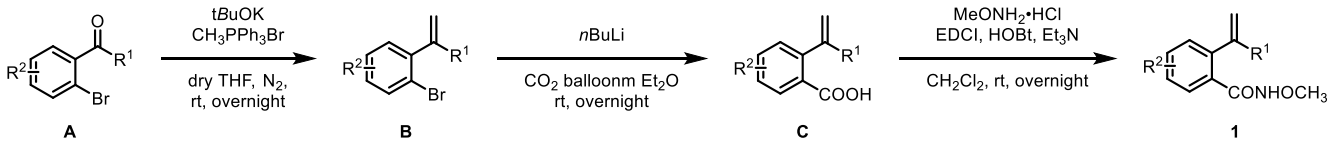
Experimental details, optimization studies, compound characterization data, and spectra

I. General experimental

Some chemicals were used as received from commercial suppliers without further purification. Other chemicals were prepared by the reported procedures. All solvents before use were dried and purified according to the standard procedure. NMR spectra were recorded for ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) using TMS as an internal standard and Bruker AV 400 as an instrument. The following abbreviations were used to describe peak patterns where appropriate: singlet (s), doublet (d), triplet (t), multiplet (m). High-resolution mass spectroscopy (HRMS) was recorded on a high-resolution ESI–FTICR mass spectrometer (Varian 7.0 T). IR spectra were recorded with a FT-IR Bruker EQUINOX55 spectrometer in KBr pellets. Melting points were determined on a RY-1 electrothermal micromelting point apparatus.

II. Experimental procedures

General procedure (I) for the synthesis of benzamide 1a and 1f–o



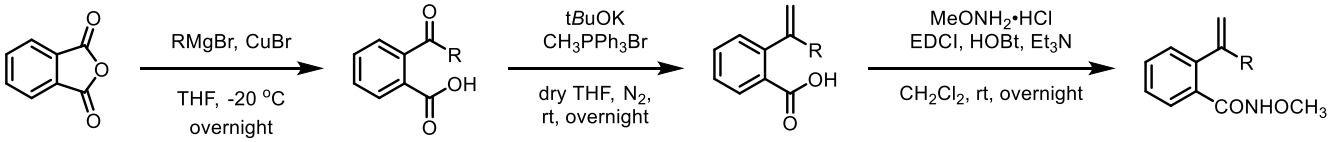
Benzamide **1a** and **1f**–**o** were synthesized according to literature procedure.[1] For example, to the mixture of methyl triphenylphosphonium bromide (3 equiv, 15 mmol) and *t*-BuOK (3 equiv, 15 mmol) was added anhydrous THF (20.0 mL) and stirred at rt for 0.5 hour under argon, then a solution of **A** (1 equiv, 5 mmol) in THF (5.0 mL) was added dropwise. The resulting reaction mixture was stirred overnight at room temperature and quenched with saturated NH_4Cl solution. The organic layer was separated and the aqueous layer was extracted with EtOAc (2 × 50 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by flash chromatography on silica gel using petroleum/EtOAc as eluent to yield **B** as a colorless oil.

To the solution of **B** (1.0 equiv, 5 mmol) in Et_2O was added dropwise *n*-BuLi (1.2 equiv, 6 mmol) at 0 °C, after 30 min, to the lithiated mixture was insert a CO_2 balloon. The mixture was allowed to warm to room temperature and stirred for overnight. The reaction was quenched with

saturated $NaHCO_3$ (15 mL) solution and washed with Et_2O (2 × 10 mL). The aqueous layer was then acidified with 2 N HCl to pH 1 and extracted with Et_2O (3 × 20 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated under reduced pressure to give **C** as a colorless solid.

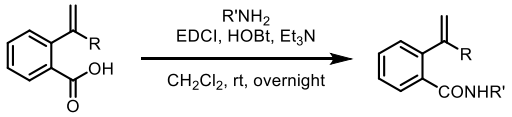
To a solution of **C** (1 equiv, 2 mmol) in DCM (5 mL), were added NH_2OMe•HCl (1.5 equiv, 3 mmol), triethylamine (5 equiv, 10 mmol). After stirring for 5 min, 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI, 1.5 equiv, 3 mmol), 1-hydroxybenzotriazole (HOBt, 1.5 equiv, 3 mmol) were added. The reaction mixture was stirred overnight at room temperature. After completion of the reaction (monitored by TLC), water and DCM were added to the reaction mixture. The organic phase was washed with aqueous HCl (1.0 M), saturated $NaHCO_3$ solution, brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to afford substrate **1** as a white solid.

General procedure (II) for the synthesis of benzamide 1b–e



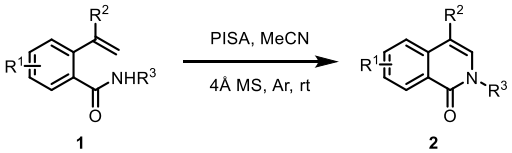
Phthalic anhydride (1.0 equiv, 10 mmol), copper bromide (0.07 equiv, 0.7 mmol) and anhydrous THF (15 mL) were added to a flame-dried flask and cooled to −20 °C under N_2 atmosphere. RMgBr (1.1 equiv, 11 mmol) was added dropwise to the mixture over 1 h. The reaction mixture was stirred overnight at −20 °C, then allowed to warm to room temperature, quenched with water, basified with aqueous NaOH (3.0 M) until pH 13–14 and washed with diethyl ether. The resulting aqueous phase was acidified with aqueous HCl (2.0 M) until pH 1–2 and extracted twice with ethyl acetate. The combined organic layers were washed with water, brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The crude product was dissolved in DCM and the solid was filtered off. The crude benzoic acid derivative was used for the next step without further purification. From benzoic acid derivative, the corresponding benzamide derivate was synthesized by the same method **1** (**method A**).

General procedure (III) for the synthesis of benzamide 1p–r



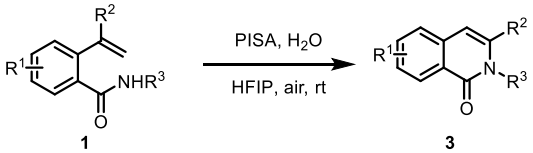
To a solution of **C** (1 equiv, 2 mmol) in DCM (5 mL), were added R'NH_2 (1.5 equiv, 3 mmol), triethylamine (5 equiv, 10 mmol). After stirring for 5 min, 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI, 1.5 equiv, 3 mmol), 1-hydroxybenzotriazole (HOBt, 1.5 equiv, 3 mmol) were added. The reaction mixture was stirred overnight at room temperature. After completion of the reaction (monitored by TLC), water and DCM were added to the reaction mixture. The organic phase was washed with aqueous HCl (1.0 M), saturated $NaHCO_3$ solution, brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to afford substrate **1p**–**r** as a white solid.

General procedure (IV) for the synthesis of 4-substituted isoquinolin-1(2*H*)-one



To a solution of PISA (0.45 mmol, 1.5 equiv), 4 Å MS (7.6 mg) in dry MeCN (3 mL, 0.1 M), were added **1** (1 equiv, 0.3 mmol) in dry MeCN under N_2 atmosphere. The reaction mixture was stirred at room temperature. After completion of the reaction (monitored by TLC), $NaHCO_3$ solution, saturated $Na_2S_2O_3$ solution and EtOAc were added to the reaction mixture. The organic phase was washed with brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to afford product **2**.

General procedure (V) for the synthesis of 3-substituted isoquinolin-1(2*H*)-one

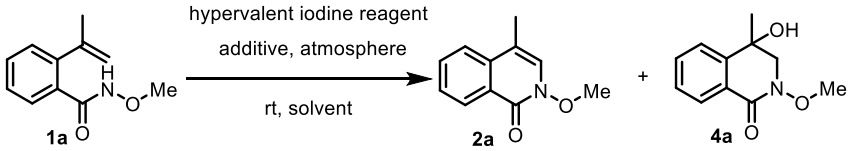


To a solution of **1** (0.3 mmol), H_2O (0.75 mmol, 2.5 equiv) in HFIP (3 mL, 0.1 M), were added PISA (0.33 mmol, 1.1 equiv). The reaction mixture was stirred at room temperature. After

completion of the reaction (monitored by TLC), $NaHCO_3$ solution, saturated $Na_2S_2O_3$ solution and EtOAc were added to the reaction mixture. The organic phase was washed with brine, dried over Na_2SO_4, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to afford product **3**.

III. Optimization studies

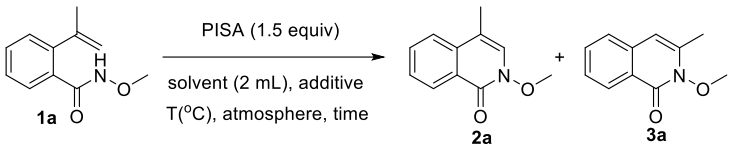
Table S1: Optimization of the reaction conditions for the synthesis of 4-methylisoquinolinone.[a]



entry	hypervalent iodine reagent	additive	atmosphere	solvent	time	yield (%)[b] 2a	4a
1	PISA	H_2O (1.5 equiv)	air	MeCN	50 min	79	13
2	PISA	H_2O (1.5 equiv)	Ar	MeCN	5 min	80	6
3	PISA	-	Ar	MeCN	20 min	86	8
4	uncrystalized PISA	4Å MS	Ar	MeCN	1 h	88	11
5	uncrystalized PISA	Na_2SO_4 (7.6 mg)	Ar	MeCN	1 h	81	14
6	PhIO	4Å MS	Ar	MeCN	1.5 h	-	-
7	PIFA	4Å MS	Ar	MeCN	20 min	77	-
8	PIDA	4Å MS	Ar	MeCN	20 min	52	28
9	HTIB	4Å MS	Ar	MeCN	20 min	52	-
10	PhINTs	4Å MS	Ar	MeCN	20 min	61	-
11	*p*-TolICl_2	4Å MS	Ar	MeCN	20 min	-	-
12	*p*-TolIF_2	4Å MS	Ar	MeCN	30 min	79	-
13[c]	IBX	4Å MS	Ar	MeCN	overnight	-	-
14[d]	IBX-KSO_3	4Å MS	Ar	MeCN	overnight	-	-
15[e]	uncrystalized PISA	4Å MS	Ar	$CHCl_3$	1 h	67	5
16	uncrystalized PISA	4Å MS	Ar	DCM	1 h	80	-
17	uncrystalized PISA	4Å MS	Ar	DCE	3 h	78	-
18	uncrystalized PISA	4Å MS	Ar	toluene	1.5 h	74	12
19	uncrystalized PISA	4Å MS	Ar	nitrobenzene	50 min	74	-
20	uncrystalized PISA	4Å MS	Ar	Et_2O	1.67 h	76	12
21	uncrystalized PISA	4Å MS	Ar	THF	1.67 h	76	-
22	uncrystalized PISA	4Å MS	Ar	EA	1.67 h	84	-
23[f]	uncrystalized PISA	4Å MS	Ar	DMF	25 min	59	-
24[g]	uncrystalized PISA	4Å MS	Ar	HFIP	1.67 h	0	-
25	uncrystalized PISA	4Å MS	Ar	H_2O	35 min	51	41

[a]Reaction conditions: **1a** (0.2 mmol), hypervalent iodine reagent (1.5 equiv), 4 Å MS (7.6 mg) in solvent (2.0 mL) at rt. [b]Yield of isolated product after column chromatography. [c]The recovery of 1a was 77%. [d]The recovery of 1a was 96%. [e]The recovery of 1a was 21%. [f]The recovery of 1a was 30%. [g]The yield of 3a was 51%.

Table S2: Optimization of the reaction conditions for the synthesis 3-methylisoquinolinone.[a]

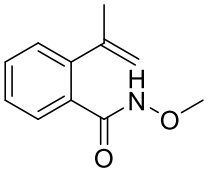


entry	solvent	additive	T(°C)	atmosphere	time	yield (%)[b] 2a	3a
1	HFIP	4Å MS	rt	Ar	1 h	0	51
2	HFIP	-	rt	Ar	20 min	0	55
3	HFIP	-	rt	air	20 min	0	57
4	HFIP	H_2O(1.5 equiv)	rt	air	20 min	0	64
5[c]	HFIP	H_2O(1.5 equiv)	rt	air	13 min	0	62
6[d]	HFIP	H_2O(1.5 equiv)	rt	air	12 min	0	38
7[e]	HFIP	H_2O(1.5 equiv)	rt	air	13 min	0	62
8	HFIP	H_2O(0.5 equiv)	rt	air	12 min	0	61
9	HFIP	H_2O(2.5 equiv)	rt	air	9 min	0	69
10	HFIP	H_2O(4.5 equiv)	rt	air	18 min	0	69
11	HFIP : H_2O = 3 : 1	-	rt	air	30 min	24	37
12	Trifluoroethanol	-	rt	air	20 min	11	54
13	Perfluoro-tert-butanol	-	rt	air	20 min	24	37
14	HFIP	-	0	air	21 min	0	54
15	HFIP	-	40	air	23 min	3	48
16[f]	HFIP	H_2O(2.5 equiv)	rt	air	13 min	0	84

[a]Reaction conditions: 1a (0.2 mmol), PISA (1.5 equiv) in solvent (2.0 mL) at rt. [b]Yield of isolated product after column chromatography. [c]Using PISA recrystallized in water. [d]HFIP was 1 mL, concentration of 1 in the solvent was 0.2 mol/L. [e]HFIP was 4 mL, concentration of 1 in the solvent was 0.05 mol/L. [f]Using 1.1 equiv PISA.

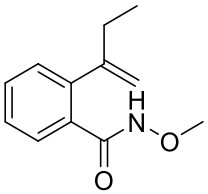
IV. Characterization data

_N_-Methoxy-2-(prop-1-en-2-yl)benzamide (1a) [1]



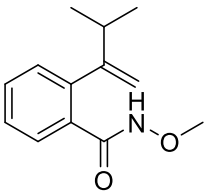
Colorless solid; m.p. 77-79 °C; yield: 68% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.91 (s, 1H), 7.36-7.28 (m, 2H), 7.22-7.15 (m, 2H), 5.10 (s, 1H), 4.95 (s, 1H), 3.66 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.1, 144.3, 141.9, 131.0, 130.0, 128.1, 127.9, 126.7, 115.6, 63.2, 23.5.

2-(But-1-en-2-yl)-_N_-methoxybenzamide (1b) [1]



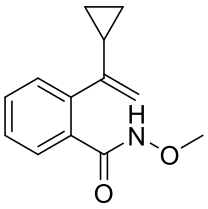
Yellowish oil; yield: 73% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.77 (s, 1H), 7.62 (d, J = 6.9 Hz, 1H), 7.46-7.28 (m, 2H), 7.18 (d, J = 7.3 Hz, 1H), 5.23 (s, 1H), 5.09 (s, 1H), 3.81 (s, 3H), 2.38 (d, J = 7.1 Hz, 2H), 1.03 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.3, 151.9, 141.5, 130.9, 130.7, 129.2, 128.9, 127.5, 114.3, 64.2, 30.6, 12.4.

_N_-Methoxy-2-(3-methylbut-1-en-2-yl)benzamide (1c) [2]



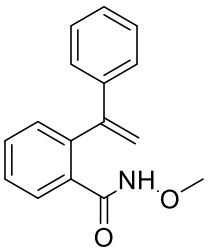
Colorless oil; yield: 56% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.74 (s, 1H), 7.69 (d, J = 7.5 Hz, 1H), 7.38 (dtd, J = 24.5, 7.5, 1.4 Hz, 2H), 7.16 (dd, J = 7.5, 1.1 Hz, 1H), 5.26 (s, 1H), 5.10 (s, 1H), 3.83 (s, 3H), 2.62-2.46 (m, 1H), 1.05 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.3, 157.3, 141.7, 130.9, 130.8, 129.8, 129.2, 127.6, 113.0, 64.4, 34.9, 21.5.

2-(1-Cyclopropylvinyl)-*N*-methoxybenzamide (1d) [2]



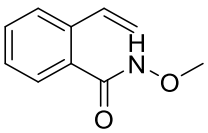
Colorless oil; yield: 70% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.88 (s, 1H), 7.73 (d, *J* = 7.5 Hz, 1H), 7.39 (dtd, *J* = 22.0, 7.4, 1.2 Hz, 2H), 7.19 (d, *J* = 7.3 Hz, 1H), 5.18 (s, 1H), 5.03 (s, 1H), 3.86 (s, 3H), 1.65 (ddd, *J* = 13.4, 8.3, 5.2 Hz, 2H), 0.86-0.72 (m, 2H), 0.50 (d, *J* = 5.3 Hz, 2H); ^{13}C NMR (100 MHz, CDCl3) δ: 151.9, 139.8, 131.1, 130.7, 129.5, 129.2, 127.8, 112.5, 110.0, 64.3, 17.4, 7.4.

***N*-Methoxy-2-(1-phenylvinyl)benzamide (1e)** [2]



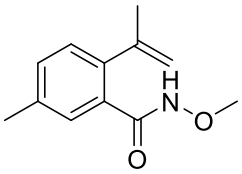
Yellow oil; yield: 20% (EA/PE = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 9.71 (s, 1H), 7.36 (d, *J* = 7.2 Hz, 2H), 7.31-7.09 (m, 7H), 5.62 (s, 1H), 5.26 (s, 1H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 165.9, 147.9, 140.0, 139.8, 132.4, 130.3, 130.0, 127.9, 127.7, 127.3, 127.2, 126.7, 115.5, 62.7.

***N*-Methoxy-2-vinylbenzamide (1f)** [3]



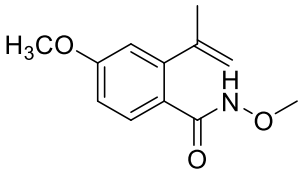
Colorless oil; yield: 83% (EA/PE = 1/8); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.35 (s, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.44 (s, 2H), 7.10-6.97 (m, 1H), 5.75 (d, *J* = 17.2 Hz, 1H), 5.39 (d, *J* = 10.8 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 167.2, 136.4, 133.8, 131.6, 130.7, 127.7, 127.6, 126.2, 117.2, 64.5.

***N*-Methoxy-5-methyl-2-(prop-1-en-2-yl)benzamide (1g)** [2]



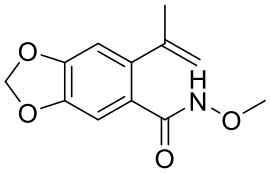
Colorless solid; m.p. 84-86 °C; yield: 80% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.65 (s, 1H), 7.43 (s, 1H), 7.22 (d, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 5.23 (s, 1H), 5.07 (s, 1H), 3.84 (s, 3H), 2.35 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 167.6, 145.4, 139.0, 137.4, 131.5, 130.6, 129.3, 128.5, 116.3, 64.1, 24.2, 20.8; IR (KBr); 3126, 2935, 1657, 1507, 1444, 1310, 1042, 968, 892, 828 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{15}NO_2 \cdot H^+$ $[M+H]^+$: 206.1176, found: 206.1175.

***N*,4-Dimethoxy-2-(prop-1-en-2-yl)benzamide (1h)** [1]



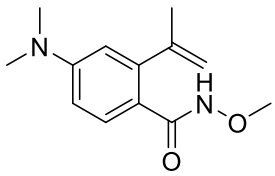
Colorless solid; m.p. 78-82 °C; yield: 80% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.85 (s, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 6.81 (dd, *J* = 8.6, 1.8 Hz, 1H), 6.69 (d, *J* = 2.0 Hz, 1H), 5.23 (s, 1H), 5.08 (s, 1H), 3.81 (s, 6H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 167.1, 161.3, 146.0, 143.9, 130.9, 123.1, 116.4, 114.2, 112.6, 64.1, 55.3, 24.1.

***N*-Methoxy-6-(prop-1-en-2-yl)benzo[d][1,3]dioxole-5-carboxamide (1i)** [1]



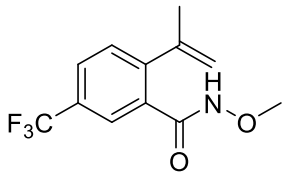
Colorless solid; m.p. 121-123 °C; yield: 79% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.67 (s, 1H), 7.11 (s, 1H), 6.66 (s, 1H), 5.99 (s, 2H), 5.23 (s, 1H), 5.07 (s, 1H), 3.83 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 166.8, 149.5, 146.9, 145.6, 137.1, 124.4, 116.6, 108.9, 108.8, 101.7, 64.1, 24.4.

4-(Dimethylamino)-*N*-methoxy-2-(prop-1-en-2-yl)benzamide (1j)



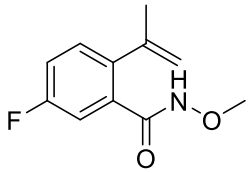
Colorless oil; yield 60% (EA/PE = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.84 (s, 1H), 7.65 (d, *J* = 8.7 Hz, 1H), 6.61 (dd, *J* = 8.8, 2.6 Hz, 1H), 6.40 (d, *J* = 2.7 Hz, 1H), 5.27-5.20 (m, 1H), 5.12 (s, 1H), 3.83 (s, 3H), 3.00 (s, 6H), 2.08 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 151.9, 148.0, 143.5, 130.9, 117.2, 115.8, 111.3, 110. 5, 64.2, 40.0, 24.5.

***N*-Methoxy-2-(prop-1-en-2-yl)-5-(trifluoromethyl)benzamide (1k)** [1]



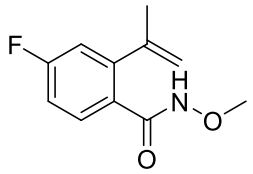
Colorless solid; m.p. 80-84 °C; yield: 59% (EA/PE = 1/7); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.80 (s, 1H), 7.86 (s, 1H), 7.66 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 5.32 (s, 1H), 5.13 (s, 1H), 3.86 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 165.9, 145.7, 143.8, 131.8, 129.5 (q, *J* = 32.8 Hz), 129.2, 127.2, 125.5, 123.5 (q, *J* = 270.6 Hz), 117.4, 63.9, 23.6; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -62.70; IR (KBr); 3215, 2983, 1565, 1509, 1340, 1294, 1167, 1109, 940, 910, 850, 780 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{12}F_3NO_2 \cdot H^+$ $[M+H]^+$: 260.0893, found: 260.0892.

5-Fluoro-*N*-methoxy-2-(prop-1-en-2-yl)benzamide (1l) [1]



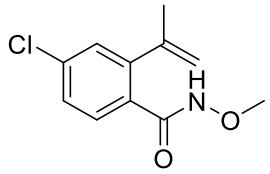
Colorless solid; m.p. 64-66 °C; yield: 13% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.86 (s, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 7.22 (dd, *J* = 8.4, 5.6 Hz, 1H), 7.10 (dd, *J* = 8.4, 2.8 Hz, 1H), 5.26 (s, 1H), 5.08 (s, 1H), 3.84 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 165.9, 161.6 (d, *J* = 246.7 Hz), 144.7, 138.0 (d, *J* = 3.5 Hz), 132.7 (d, *J* = 2.4 Hz), 130.6 (d, *J* = 7.5 Hz), 117.8 (d, *J* = 19.6 Hz), 117.0, 115.8 (d, *J* = 22.4 Hz), 64.2, 24.2; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -118.48.

4-Fluoro-*N*-methoxy-2-(prop-1-en-2-yl)benzamide (1m) [1]



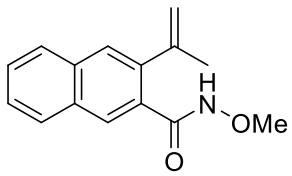
Colorless solid; m.p. 113-115 °C; yield: 29% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 9.10 (s, 1H), 7.49 (s, 1H), 7.04-6.80 (m, 2H), 5.21 (s, 1H), 5.05 (s, 1H), 3.77 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 166.4, 163.6 (d, *J* = 250.1 Hz), 144.8 (d, *J* = 8.1 Hz), 144.2, 131.0 (d, *J* = 8.6 Hz), 127.2, 117., 115.5 (d, *J* = 22.1 Hz), 114.3 (d, *J* = 21.3 Hz), 64.0, 23.7; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -108.89.

4-Chloro-*N*-methoxy-2-(prop-1-en-2-yl)benzamide (1n) [1]



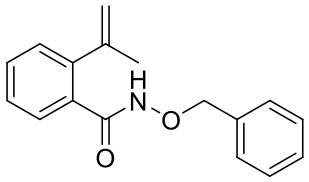
Colorless solid; m.p. 107-109 °C; yield: 54% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.70 (s, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 11.2 Hz, 1H), 5.29 (s, 1H), 5.12 (s, 1H), 3.85 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 166.3, 144.0, 143.8, 136.5, 130.0, 129.4, 128.6, 127.4, 117.1, 64.0, 23.7.

***N*-Methoxy-3-(prop-1-en-2-yl)-2-naphthamide (1o)**



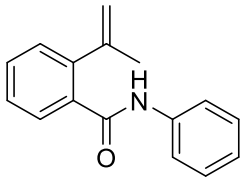
Colorless solid; m.p. 122-124 °C; yield: 46% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.90 (s, 1H), 8.13 (s, 1H), 7.82 (dd, *J* = 12.8, 8.1 Hz, 2H), 7.69 (s, 1H), 7.52 (dtd, *J* = 14.7, 7.0, 1.2 Hz, 2H), 5.30 (s, 1H), 5.20 (s, 1H), 3.89 (s, 3H), 2.16 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 167.36, 145.95, 138.81, 134.15, 131.82, 129.64, 128.38, 127.92, 127.68, 126.77, 116.61, 64.39, 24.33. HRMS (ESI): calcd for $C_{15}H_{15}NO_2 \cdot Na^+$ $[M+Na]^+$: 264.0995, found: 264.1000.

***N*-(Benzyloxy)-2-(prop-1-en-2-yl)benzamide (1p)** [2]



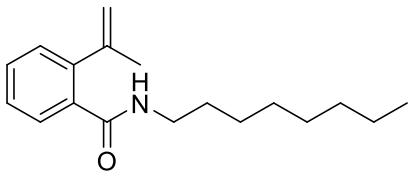
Colorless solid; m.p. 84-86 °C; yield: 65% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.72 (s, 1H), 7.53 (d, *J* = 7.4 Hz, 1H), 7.46-7.24 (m, 9H), 7.19 (d, *J* = 7.5 Hz, 1H), 5.15-5.06 (m, 1H), 5.00 (d, *J* = 10.0 Hz, 3H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.28, 145.47, 142.14, 137.34, 135.46, 130.76, 129.05, 128.73, 128.63, 128.49, 128.39, 128.02, 127.43, 116.42, 78.09, 77.95, 77.45, 77.13, 76.81, 24.25. RMS (ESI): calcd for $C_{17}H_{17}NO_2 \cdot Na^+$ $[M+Na]^+$: 290.1151, found: 290.1155.

***N*-Phenyl-2-(prop-1-en-2-yl)benzamide (1q)** [4]



Colorless solid; m.p. 104-105 °C; yield: 74% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.99 (s, 1H), 7.78 (d, *J* = 7.5 Hz, 1H), 7.58 (d, *J* = 7.8 Hz, 2H), 7.45 (td, *J* = 7.5, 1.4 Hz, 1H), 7.42 – 7.31 (m, 4H), 7.30 – 7.26 (m, 1H), 7.14 (t, *J* = 7.4 Hz, 1H), 5.33 (s, 1H), 5.21 (s, 1H), 2.10 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 167.0, 146.8, 141.8, 138.1, 133.9, 130.9, 129.1, 129.1, 129.0, 127.9, 124.5, 119.7, 116.3, 24.3. HRMS (ESI): calcd for $C_{16}H_{15}NO \cdot H^+$ $[M+H]^+$: 238.1226, found: 238.1229.

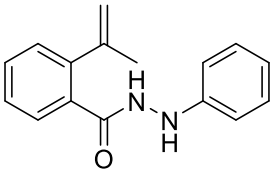
***N*-Octyl-2-(prop-1-en-2-yl)benzamide (1r)**



Colorless solid; m.p. 107-108 °C; yield: 69% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.65 (d, J = 7.4 Hz, 1H), 7.35 (dt, J = 22.4, 7.4 Hz, 2H), 7.21 (d, J = 7.5 Hz, 1H), 6.15 (s, 1H), 5.22 (s, 1H), 5.08 (s, 1H), 3.39 (dt, J = 7.1, 3.6 Hz, 2H), 2.07 (s, 3H), 1.59 – 1.49 (m, 2H), 1.30 (d, J

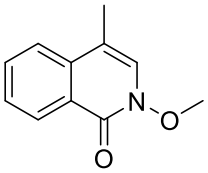
= 16.4 Hz, 10H), 0.88 (dd, J = 6.7, 5.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 169.0, 146.7, 141.6, 130.2, 128.8, 128.6, 127.5, 115.76, 40.1, 31.8, 29.3, 27.1, 24.3, 22.6, 14.1. HRMS (ESI): calcd for $C_{18}H_{27}NO{\cdot}H^+$ $[M+H]^+$: 274.2165, found: 274.2168.

***N'*-Phenyl-2-(prop-1-en-2-yl)benzohydrazide (1s)**



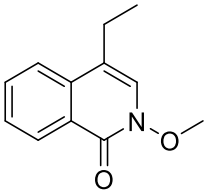
Colorless solid; m.p. 97-98 °C; yield: 27% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 7.88 (s, 1H), 7.67 (d, J = 7.5 Hz, 1H), 7.45 (t, J = 7.3 Hz, 1H), 7.36 (t, J = 7.4 Hz, 1H), 7.31 – 7.18 (m, 1H), 6.92 (t, J = 9.4 Hz, 1H), 5.28 (s, 1H), 5.12 (s, 1H), 2.12 (s, 1H); ^{13}C NMR (100 MHz, CDCl3) *δ:* 169.2, 148.0, 145.9, 142.2, 131.7, 131.0, 129.2, 129.0, 128.8, 127.6, 121.5, 116.5, 113.9, 24.7.

2-Methoxy-4-methylisoquinolin-1(2*H*)-one (2a) [5]



Colorless solid; m.p. 67-69 °C; yield: 88% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.50 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.16 (s, 1H), 4.08 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.7, 136.4, 132.1, 128.0, 127.3, 126.9, 126.8, 123.3, 112.1, 64.2, 15.2.

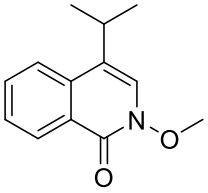
4-Ethyl-2-methoxyisoquinolin-1(2*H*)-one (2b) [6]



Colorless solid; m.p. 86-89 °C; yield: 94% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.51 (d, *J* = 8.0 Hz, 1H), 7.74-7.62 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.14 (s, 1H), 4.09 (s, 3H), 2.74 (q,

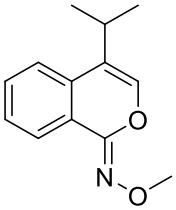
J = 7.2 Hz, 2H), 1.31 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.6, 135.8, 132.0, 128.2, 127.5, 126.7, 126.0, 122.9, 118.0, 64.2, 22.1, 13.4.

4-Isopropyl-2-methoxyisoquinolin-1(2*H*)-one (2c)



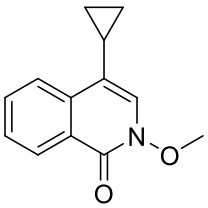
Colorless oil; yield: 51% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.53 (ddd, *J* = 8.1, 1.4, 0.5 Hz, 1H), 7.85-7.61 (m, 2H), 7.52 (ddd, *J* = 8.1, 6.9, 1.3 Hz, 1H), 7.16 (d, *J* = 0.8 Hz, 1H), 4.11 (s, 3H), 3.29 (dt, *J* = 13.4, 6.8 Hz, 1H), 1.33 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.5, 135.5, 132.1, 128.4, 127.6, 126.7, 124.9, 122.8, 122.7, 64.2, 26.6, 22.6.

(Z)-4-Isopropyl-1H-isochromen-1-one O-methyl oxime (2c')



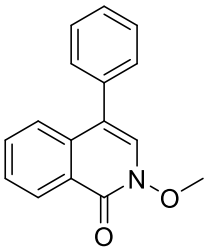
Colorless oil; yield: 31% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.90 (d, *J* = 7.9 Hz, 1H), 7.40 – 7.31 (m, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 5.82 (s, 1H), 3.96 (s, 3H), 2.69 (p, *J* = 6.9 Hz, 1H), 1.25 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 153.8, 146.8, 131.6, 130.9, 127.3, 125.9, 124.8, 123.9, 98.9, 62.7, 31.8, 20.2.

4-Cyclopropyl-2-methoxyisoquinolin-1(2*H*)-one (2d)



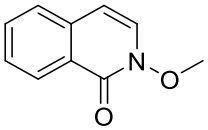
Colorless oil; yield: 54% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.49 (d, *J* = 8.1 Hz, 1H), 8.05 (d, *J* = 8.1 Hz, 1H), 7.78-7.67 (m, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 1.0 Hz, 1H), 4.08 (s, 3H), 1.94-1.86 (m, 1H), 1.01-0.94 (m, 2H), 0.60 (q, *J* = 5.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.8, 137.1, 132.1, 128.0, 127.3, 127.0, 126.9, 123.7, 118.0, 64.3, 10.1, 5.3.

2-Methoxy-4-phenylisoquinolin-1(2*H*)-one (2e) [7]



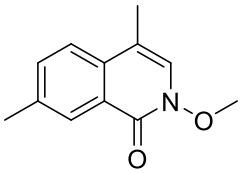
Colorless solid; m.p. 68-70 °C; yield: 68% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.49 (d, *J* = 7.6 Hz, 1H), 7.62-7.30 (m, 8H), 7.25 (s, 1H), 4.07 (s, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.6, 135.5, 135.4, 132.2, 129.9, 128.7, 128.0, 128.0, 128.0, 127.2, 127.0, 125.0, 119.4, 64.4.

2-Methoxyisoquinolin-1(2*H*)-one (2f) [6]



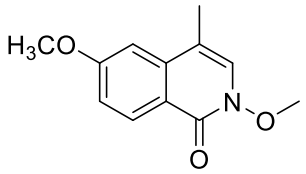
Colorless oil; yield: 87% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.47 (d, *J* = 8.0 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.57-7.47 (m, 2H), 7.33 (d, *J* = 7.6 Hz, 1H), 6.47 (d, *J* = 7.6 Hz, 1H), 4.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 158.2, 136.1, 132.3, 129.4, 127.7, 127.5, 126.9, 126.2, 105.6, 64.4.

2-Methoxy-4,7-dimethylisoquinolin-1(2*H*)-one (2g)



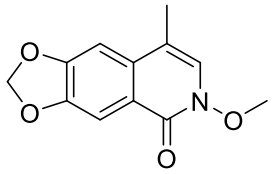
Colorless solid; m.p. 68-70 °C; yield: 95% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.31 (s, 1H), 7.26 (s, 1H), 7.11 (s, 1H), 4.08 (s, 3H), 2.50 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.7, 137.0, 134.2, 133.6, 127.7, 127.2, 126.0, 123.3, 112.2, 64.2, 21.3, 15.2; IR (KBr); 3722, 3657, 3292, 3174, 3072, 2937, 1656, 1612, 1502, 1337, 1262, 1098, 1027, 852, 811 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{13}NO_2 \cdot H^+$ $[M+H]^+$: 204.1019, found: 204.1019.

2,6-Dimethoxy-4-methylisoquinolin-1(2*H*)-one (2h)



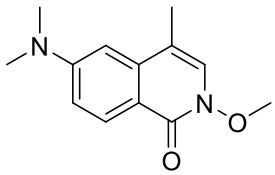
Colorless solid; m.p. 92-94 °C; yield: 68% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.38 (d, J = 8.8 Hz, 1H), 7.15-6.99 (m, 2H), 6.89 (s, 1H), 4.05 (s, 3H), 3.91 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.6, 157.5, 138.5, 130.0, 127.5, 120.8, 115.4, 111.5, 105.2, 64.2, 55.4, 15.3; IR (KBr); 3657, 3006, 1663, 1607, 1486, 1260, 1196, 924, 854 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{13}NO_3 \cdot H^+$ $[M+H]^+$: 220.0968, found: 220.0968.

6-Methoxy-8-methyl-[1,3]dioxolo[4,5-*g*]isoquinolin-5(6*H*)-one (2i)



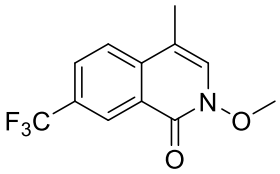
Colorless solid; m.p. 138-139 °C; yield: 76% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (s, 1H), 7.07 (s, 1H), 6.92 (s, 1H), 6.08 (s, 2H), 4.06 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.0, 151.9, 147.6, 133.8, 125.7, 122.8, 111.6, 105.9, 101.9, 101.5, 64.2, 15.6; IR (KBr); 3656, 3060, 1659, 1617, 1582, 1487, 1248, 1036, 933, 873 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{11}NO_4 \cdot H^+$ $[M+H]^+$: 234.0761, found: 234.0760.

6-(Dimethylamino)-2-methoxy-4-methylisoquinolin-1(*2H*)-one (2j)



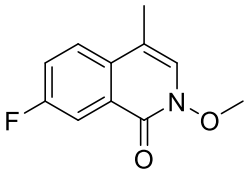
Colorless oil; yield: 85% (EA/PE = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.32 (d, J = 9.1 Hz, 1H), 7.08 (d, J = 1.0 Hz, 1H), 6.93 (dd, J = 9.1, 2.5 Hz, 1H), 6.54 (d, J = 2.5 Hz, 1H), 4.06 (s, 3H), 3.10 (s, 6H), 2.24 (d, J = 1.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.0, 152.6, 138.1, 129.4, 127.1, 112.7, 111.5, 110.0, 102.5, 64.2, 40.2, 15.4.

2-Methoxy-4-methyl-7-(trifluoromethyl)isoquinolin-1(2*H*)-one (2k)



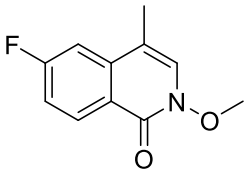
Colorless solid; m.p. 230-232 °C; yield: 84% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.77 (s, 1H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 8.8 Hz, 1H), 7.27 (s, 1H), 4.09 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.1, 138.8, 129.2, 128.7 (q, *J* = 33.2 Hz), 128.2 (q, *J* = 3.2 Hz), 127.2, 125.6 (q, *J* = 3.7 Hz), 124.3, 123.9 (q, *J* = 270.7 Hz), 111.5, 64.4, 15.1; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -62.24; IR (KBr); 3655, 2991, 1666, 1516, 1330, 1260, 1093, 804, 622 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{10}F_3NO_2 \cdot H^+$ $[M+H]^+$: 258.0736, found: 258.0738.

7-Fluoro-2-methoxy-4-methylisoquinolin-1(2*H*)-one (2l)



Colorless solid; m.p. 121-123 °C; yield: 84% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.14 (d, *J* = 8.8 Hz, 1H), 7.67-7.54 (m, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.13 (s, 1H), 4.08 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 161.4 (d, *J* = 246.9 Hz), 156.9 (d, *J* = 3.2 Hz), 133.1 (d, *J* = 1.7 Hz), 129.0 (d, *J* = 8.1 Hz), 126.3 (d, *J* = 1.7 Hz), 125.8 (d, *J* = 7.9 Hz), 120.8 (d, *J* = 23.4 Hz), 113.3 (d, *J* = 22.9 Hz), 111.7, 64.2, 15.3; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -108.61; IR (KBr); 3070, 2936, 1656, 1259, 1182, 1020, 952, 871, 816, 778, 705 cm^{-1}; HRMS (ESI): calcd for $C_{11}H_{10}FNO_2 \cdot H^+$ $[M+H]^+$: 208.0768, found: 208.0771.

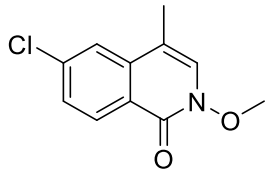
6-Fluoro-2-methoxy-4-methylisoquinolin-1(2*H*)-one (2m)



Colorless solid; m.p. 99-101 °C; yield: 81% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.55-8.42 (m, 1H), 7.30-7.14 (m, 3H), 4.08 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 165.2

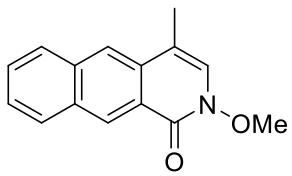
(d, *J* = 252.6 Hz), 157.1, 139.0 (d, *J* = 10.0 Hz), 131.2 (d, *J* = 9.9 Hz), 128.3, 123.8, 115.3 (d, *J* = 23.5 Hz), 111.4 (d, *J* = 3.3 Hz), 108.8 (d, *J* = 22.6 Hz), 64.3, 15.2; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -106.58; IR (KBr); 2922, 1662, 1621, 1476, 1452, 1326, 1017, 988, 948, 857, 821, 770, 693 cm^{-1}; HRMS (ESI): calcd for $C_{11}H_{10}FNO_2 \cdot H^+$ [M+H]+: 208.0768, found: 208.0772.

6-Chloro-2-methoxy-4-methylisoquinolin-1(2*H*)-one (2n)



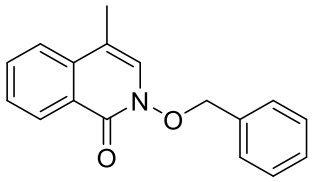
Colorless solid; m.p. 107-109 °C; yield: 93% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.40 (d, *J* = 8.4 Hz, 1H), 7.55 (s, 1H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.18 (s, 1H), 4.07 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 157.2, 138.8, 137.8, 129.8, 128.3, 127.2, 125.5, 122.9, 111.1, 64.3, 15.1; IR (KBr); 3066, 2939, 1662, 1614, 1470, 1429, 1323, 1094, 1017, 920, 822, 775, 687, 616 cm^{-1}; HRMS (ESI): calcd for $C_{11}H_{10}ClNO_2 \cdot H^+$ [M+H]+: 224.0473, found: 224.0469.

2-Methoxy-4-methylbenzo[*g*]isoquinolin-1(*2H*)-one (2o)



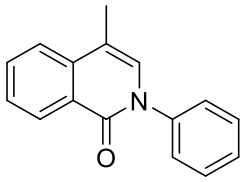
Colorless oil; yield: 40% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 9.10 (s, 1H), 8.06 (d, *J* = 8.2 Hz, 1H), 8.04 (s, 1H), 7.98 (d, *J* = 8.3 Hz, 1H), 7.61 (dd, *J* = 11.1, 4.0 Hz, 1H), 7.55 (dd, *J* = 11.0, 3.9 Hz, 1H), 7.11 (s, 1H), 4.11 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 158.4, 135.0, 132.7, 131.5, 129.4, 129.3, 128.3, 127.9, 126.4, 126.1, 125.4, 122.2, 112.3, 64.1, 15.5. HRMS (ESI): calcd for $C_{15}H_{13}NO_2 \cdot H^+$ [M+H]+: 240.1019, found: 240.1025.

2-(Benzyloxy)-4-methylisoquinolin-1(*2H*)-one (2p)



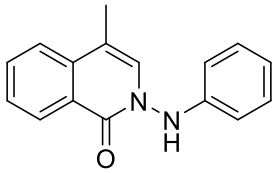
Colorless oil; yield: 69% (EA/PE = 1/10); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 8.54 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.71 (ddd, *J* = 8.3, 7.1, 1.4 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.56 – 7.51 (m, 1H), 7.49 (dt, *J* = 4.8, 3.0 Hz, 2H), 7.41 – 7.35 (m, 3H), 6.94 (d, *J* = 1.1 Hz, 1H), 5.29 (s, 2H), 2.20 (d, *J* = 1.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 158.2, 136.5, 134.2, 132.2, 129.9, 129.2, 128.7, 128.3, 128.1, 127.3, 126.7, 123.3, 111.4, 78.4, 15.1; HRMS (ESI): calcd for $C_{17}H_{15}NO_2 \cdot H^+$ [M+H]+: 288.0995, found: 288.1001.

4-Methyl-2-phenylisoquinolin-1(*2H*)-one (2q) [8]



Colorless solid; m.p. 107-108 °C; yield: 41% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 8.37 (d, *J* = 8.0 Hz, 1H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.53 (t, *J* = 7.4 Hz, 2H), 7.49 – 7.40 (m, 3H), 7.35 (d, *J* = 6.4 Hz, 1H), 7.24 (s, 1H), 6.44 (s, 1H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ:* 163.5, 139.5, 139.2, 137.2, 132.6, 129.6, 128.6, 128.5, 128.2, 126.1, 125.2, 122.8, 105.5, 21.7.

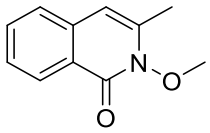
4-Methyl-2-(phenylamino)isoquinolin-1(*2H*)-one (2r)



Colorless oil; yield: 40% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 8.46 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.75 (ddd, *J* = 8.3, 7.1, 1.4 Hz, 1H), 7.68 (d, *J* = 7.7 Hz, 1H), 7.58 – 7.49 (m, 1H), 7.45 (s, 1H), 7.25 – 7.19 (m, 3H), 6.96 (t, *J* = 7.4 Hz, 1H), 6.78 (dd, *J* = 8.6, 0.9 Hz, 2H), 2.35 (d, *J* = 1.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ:* 161.2, 147.3, 137.3, 132.5, 131.2, 129.4, 128.4, 126.9,

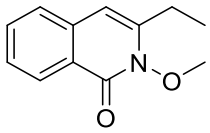
126.2, 123.4, 122.5, 114.6, 112.1, 15.4; HRMS (ESI): calcd for $C_{16}H_{14}N_2O \cdot Na^+$ $[M+Na]^+$: 273.0998, found: 273.1002.

2-Methoxy-3-methylisoquinolin-1(2*H*)-one (3a) [9]



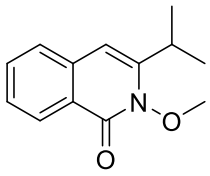
Colorless solid; m.p. 81-87 °C; yield: 84% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.36 (d, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.48-7.33 (m, 2H), 6.25 (s, 1H), 4.06 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 158.8, 139.1, 136.0, 132.2, 127.4, 125.7, 125.2, 104.2, 63.6, 17.1.

3-Ethyl-2-methoxyisoquinolin-1(2*H*)-one (3b) [9]



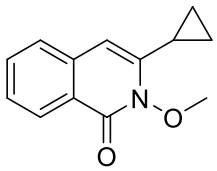
Colorless solid; m.p. 98-100 °C; yield: 74% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.39 (d, *J* = 8.0 Hz, 1H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.50-7.36 (m, 2H), 6.26 (s, 1H), 4.09 (s, 3H), 2.78 (q, *J* = 7.3 Hz, 2H), 1.34 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 159.0, 144.4, 136.0, 132.2, 127.5, 125.8, 125.8, 125.5, 102.5, 63.8, 23.6, 12.4.

3-Isopropyl-2-methoxyisoquinolin-1(2*H*)-one (3c) [9]



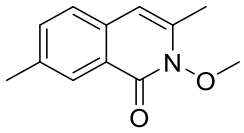
Colorless oil; yield: 72% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.43-8.36 (m, 1H), 7.66-7.57 (m, 1H), 7.44 (ddd, *J* = 12.1, 8.1, 6.9 Hz, 2H), 6.30 (s, 1H), 4.09 (s, 3H), 3.20 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.34 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 159.0, 148.9, 136.0, 132.2, 127.5, 125.9, 125.7, 125.6, 100.8, 64.0, 28.3, 22.1.

3-Cyclopropyl-2-methoxyisoquinolin-1(*2H*)-one (3d) [10]



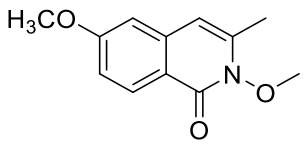
Colorless oil; yield: 52% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.1 Hz, 1H), 7.59 (t, *J* = 7.0 Hz, 1H), 7.46-7.37 (m, 2H), 6.08 (s, 1H), 4.15 (s, 3H), 2.28-2.16 (m, 1H), 1.11-1.01 (m, 2H), 0.83 (q, *J* = 5.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 144.6, 136.0, 132.2, 127.6, 125.9, 125.7, 125.6, 100.6, 63.8, 10.6, 7.0.

2-Methoxy-3,7-dimethylisoquinolin-1(2*H*)-one (3e)



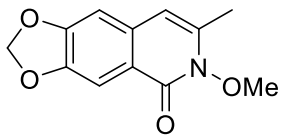
Colorless solid; m.p. 109-111 °C; yield: 69% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.18 (s, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.1 Hz, 1H), 6.23 (s, 1H), 4.06 (s, 3H), 2.45 (s, 1H), 2.41 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 158.8, 138.0, 135.8, 133.8, 133.7, 127.0, 125.8, 125.2, 104.1, 63.5, 21.3, 17.1; IR (KBr); 2923, 1664, 1612, 1496, 1449, 1380, 1335, 1256, 977, 825, 784, 690, 592, 544 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{13}NO_2 \cdot H^+$ $[M+H]^+$: 204.1019, found: 204.1020.

2,6-Dimethoxy-3-methylisoquinolin-1(2*H*)-one (3f)



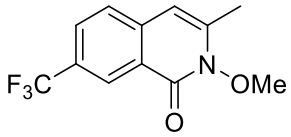
Colorless solid; m.p. 126-128 °C; yield: 65% (EA/PE = 1/4); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.27 (d, *J* = 8.8 Hz, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 6.76 (s, 1H), 6.17 (s, 1H), 4.06 (s, 3H), 3.87 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 162.7, 158.6, 139.7, 138.0, 129.6, 119.6, 115.5, 106.0, 103.8, 63.6, 55.34, 17.2; IR (KBr); 3658, 2994, 1665, 1604, 1487, 1257, 1225, 1170, 978, 855, 829, 686 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{13}NO_3 \cdot H^+$ $[M+H]^+$: 220.0968, found: 220.0965.

6-Methoxy-7-methyl-[1,3]dioxolo[4,5-*g*]isoquinolin-5(6*H*)-one (3g)



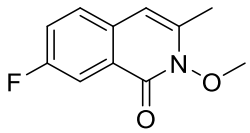
Colorless solid; m.p. 124-126 °C; yield: 63% (EA/PE = 1/2); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.70 (s, 1H), 6.74 (s, 1H), 6.13 (s, 1H), 6.04 (s, 2H), 4.06 (s, 3H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 158.2, 151.9, 147.1, 137.6, 133.3, 121.2, 105.2, 104.0, 103.0, 101.6, 63.6, 17.0; IR (KBr); 3660, 2995, 1662, 1621, 1412, 1240, 1037, 931, 868 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{11}NO_4 \cdot H^+$ $[M+H]^+$: 234.0761, found: 234.0762.

2-Methoxy-3-methyl-7-(trifluoromethyl)isoquinolin-1(2*H*)-one (3h)



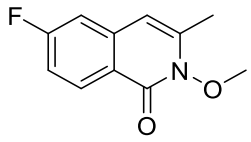
Colorless solid; m.p. 158-160 °C; yield: 44% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.67 (s, 1H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 6.32 (s, 1H), 4.10 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 158.3, 141.9, 138.4, 128.3 (q, *J* = 3.2 Hz), 127.8 (q, *J* = 33.4 Hz), 126.2, 125.5, 125.4 (d, *J* = 4.1 Hz), 123.9 (d, *J* = 285.6 Hz), 103.6, 63.8, 17.4; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -62.15.

7-Fluoro-2-methoxy-3-methylisoquinolin-1(2*H*)-one (3i)



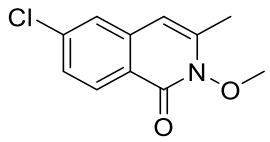
Colorless solid; m.p. 116-118 °C; yield: 90% (EA/PE = 1/3); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.01 (dd, *J* = 9.2, 2.4 Hz, 1H), 7.41 (dd, *J* = 8.8, 5.2 Hz, 1H), 7.32 (td, *J* = 8.4, 2.7 Hz, 1H), 6.25 (s, 1H), 4.07 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 160.7 (d, *J* = 245.5 Hz), 158.1 (d, *J* = 3.6 Hz), 138.4 (d, *J* = 2.9 Hz), 132.67, 127.67 (d, *J* = 7.8 Hz), 127.3 (d, *J* = 7.7 Hz), 121.2 (d, *J* = 23.7 Hz), 112.5 (d, *J* = 22.7 Hz), 103.6, 63.62 (d, *J* = 3.3 Hz), 17.10 (d, *J* = 1.9 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -113.99; IR (KBr); 2930, 1660, 1620, 1497, 1346, 1256, 1090, 954, 843, 798 cm^{-1}; HRMS (ESI): calcd for $C_{11}H_{10}FNO_2 \cdot H^+$ $[M+H]^+$: 208.0768, found: 208.0772.

6-Fluoro-2-methoxy-3-methylisoquinolin-1(2*H*)-one (3j)



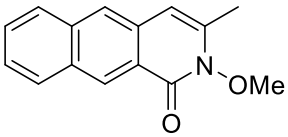
Colorless solid; m.p. 120-122 °C; yield: 53% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.39 (dd, *J* = 8.8, 6.0 Hz, 1H), 7.11 (td, *J* = 8.8, 2.3 Hz, 1H), 7.04 (dd, *J* = 9.4, 2.2 Hz, 1H), 6.21 (s, 1H), 4.08 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 165.2 (d, *J* = 250.8 Hz), 158.3, 140.7, 138.2 (d, *J* = 10.5 Hz), 130.8 (d, *J* = 10.1 Hz), 122.5, 114.6 (d, *J* = 23.5 Hz), 110.0 (d, *J* = 21.8 Hz), 103.5 (d, *J* = 3.2 Hz), 63.7, 17.3; ^{19}F NMR (376 MHz, $CDCl_3$) *δ*: -109.15.

6-Chloro-2-methoxy-3-methylisoquinolin-1(2*H*)-one (3k)



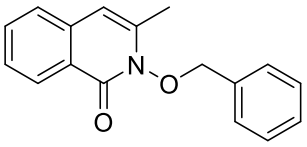
Colorless solid; m.p. 105-107 °C; yield: 76% (EA/PE = 1/8); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.31 (d, *J* = 8.8 Hz, 1H), 7.41 (d, *J* = 1.6 Hz, 1H), 7.35 (dd, *J* = 8.6, 1.8 Hz, 1H), 6.19 (s, 1H), 4.08 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ*: 158.4, 140.8, 138.7, 137.2, 129.4, 126.4, 124.5, 124.1, 103.2, 63.7, 17.3; IR (KBr); 1659, 1380, 1257, 1078, 977, 908, 825, 680 cm^{-1}; HRMS (ESI): calcd for $C_{11}H_{10}ClNO_2 \cdot H^+$ $[M+H]^+$: 224.0473, found: 224.0469.

2-Methoxy-3-methylbenzo[*g*]isoquinolin-1(*2H*)-one (3l)



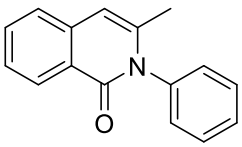
Colorless solid; m.p. 123-125 °C; yield: 54% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 9.00 (s, 1H), 8.03 (d, *J* = 8.3 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 2H), 7.53 (dt, *J* = 14.9, 7.2 Hz, 2H), 6.38 (s, 1H), 4.11 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 159.4, 138.1, 135.4, 132.0, 131.1, 129.4, 128.9, 128.1, 127.5, 125.8, 124.5, 123.3, 104. 63.8, 17.4; HRMS (ESI): calcd for $C_{15}H_{13}NO_2 \cdot H^+$ $[M+H]^+$: 240.1019, found: 240.1021.

2-(Benzyloxy)-3-methylisoquinolin-1(*2H*)-one (3m)



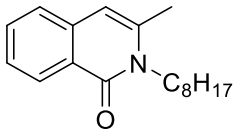
Colorless oil; yield: 64% (EA/PE = 1/10); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 8.43 (d, *J* = 7.4 Hz, 1H), 7.66 – 7.58 (m, 1H), 7.54 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.42 (ddd, *J* = 9.0, 8.5, 5.0 Hz, 5H), 6.25 (s, 1H), 5.29 (s, 2H), 2.33 (d, *J* = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 159.2, 139.7, 136.2, 134.2, 132.3, 129.9, 129.2, 128.7, 127.6, 125.9, 125.9, 125.3, 104.1, 77.7, 17.7; HRMS (ESI): calcd for $C_{17}H_{15}NO_2 \cdot H^+$ $[M+H]^+$: 288.0995, found: 288.0998.

3-Methyl-2-phenylisoquinolin-1(*2H*)-one (3n) [11]



Colorless solid; m.p. 113-114 °C; yield: 51% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 8.38 (d, *J* = 8.0 Hz, 1H), 7.68 – 7.59 (m, 1H), 7.53 (dd, *J* = 10.1, 4.7 Hz, 2H), 7.49 – 7.38 (m, 3H), 7.25 (d, *J* = 7.3 Hz, 2H), 6.44 (s, 1H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 163.5, 139.5, 139.2, 137.2, 132.6, 129.6, 128.6, 128.5, 128.2, 126.1, 125.2, 124.9, 105.5, 21.6; HRMS (ESI): calcd for $C_{16}H_{13}NO \cdot H^+$ $[M+H]^+$: 236.1070, found: 236.1071.

3-Methyl-2-octylisoquinolin-1(*2H*)-one (3o)



Colorless oil; yield: 32% (EA/PE = 1/5); ^{1}H NMR (400 MHz, $CDCl_3$) *δ:* 8.36 (d, *J* = 8.1 Hz, 1H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 6.9 Hz, 2H), 6.34 (s, 1H), 4.07 (t, 2H), 1.70 (dd, *J* = 16.7, 9.2 Hz, 2H), 1.35 (dd, *J* = 47.8, 9.4 Hz, 10H), 0.87 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ:* 163.0, 139.0, 136.7, 132.0, 127.9, 125.8, 124.9, 124.6, 105.9, 44.5, 31.8, 29.3, 29.3, 28.9, 27.1, 22.6, 20.6, 14.1.

V. References

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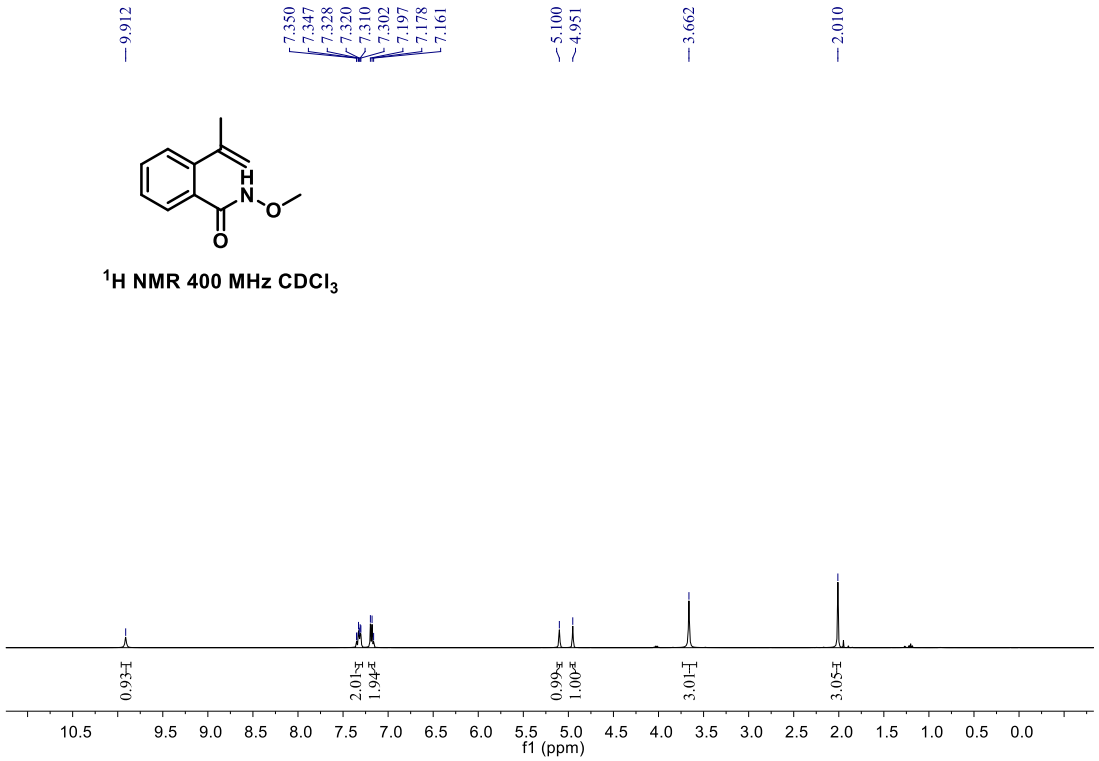
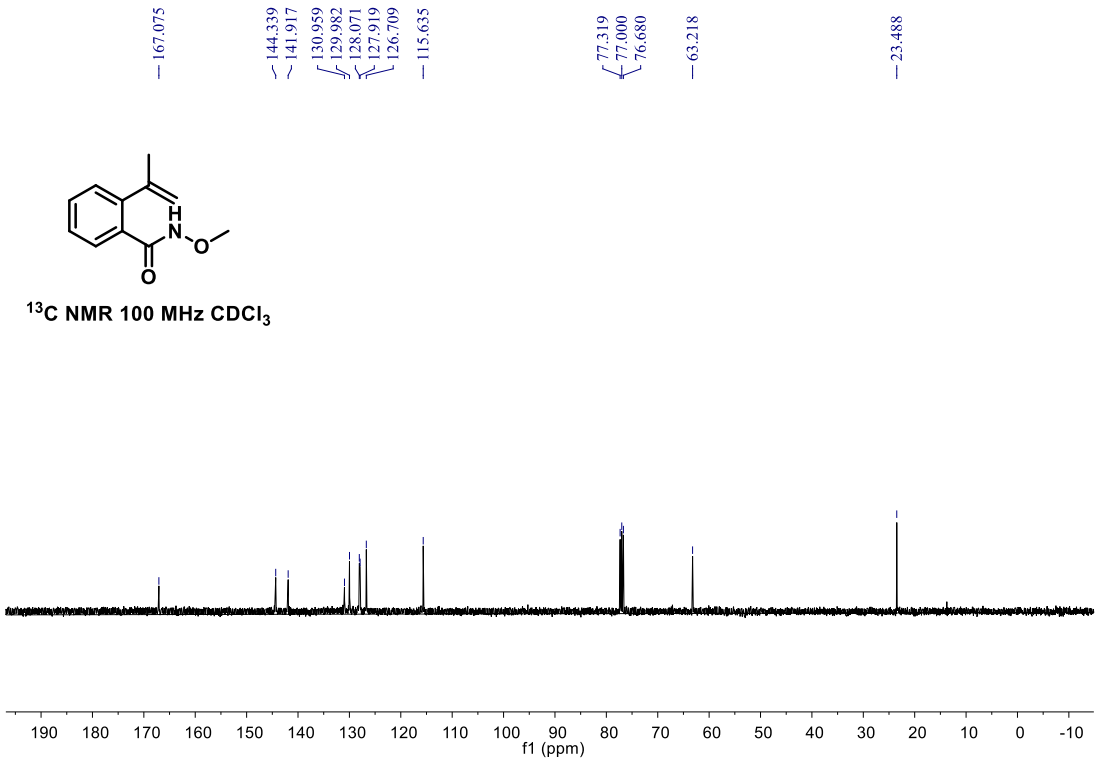
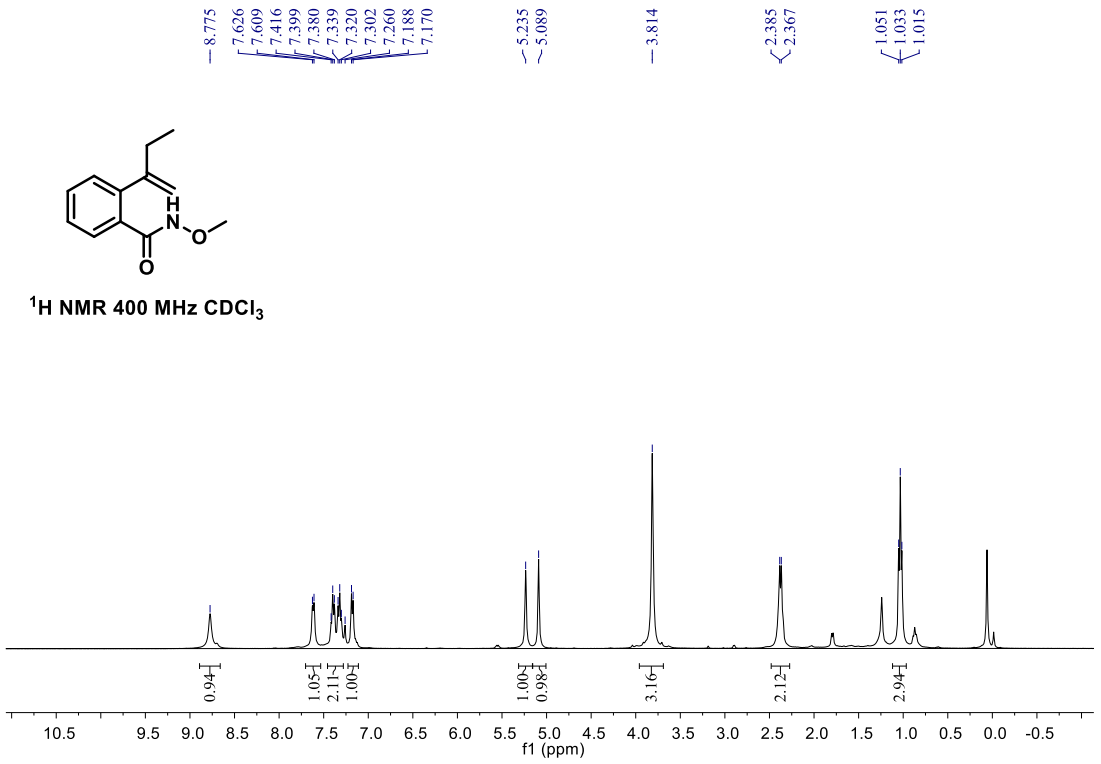
VI. Spectra



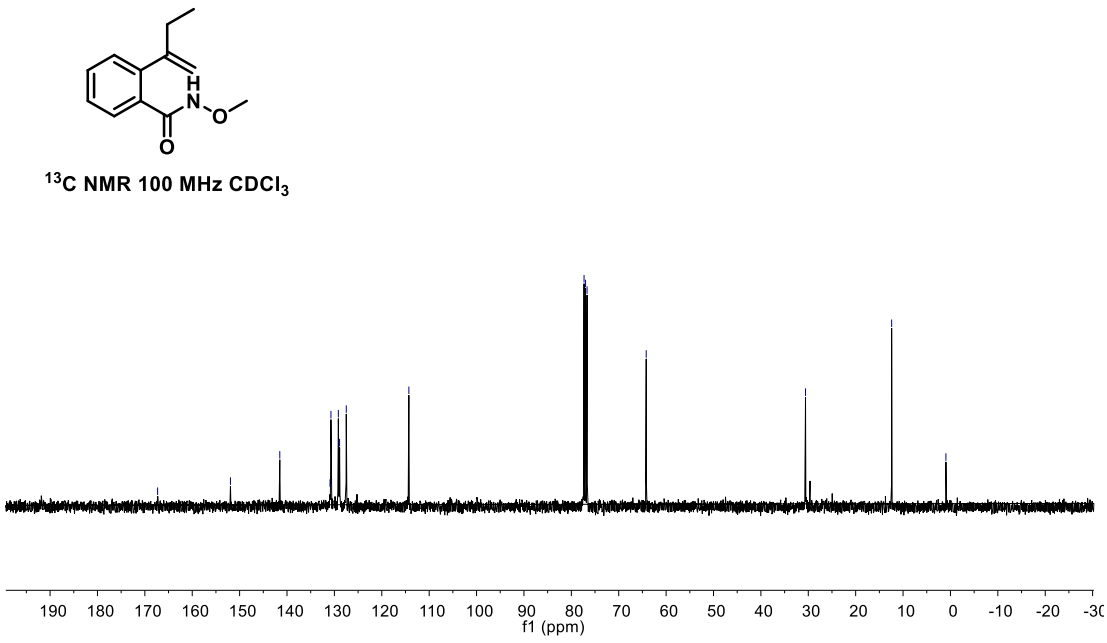
^{1}H NMR 400 MHz $CDCl_3$



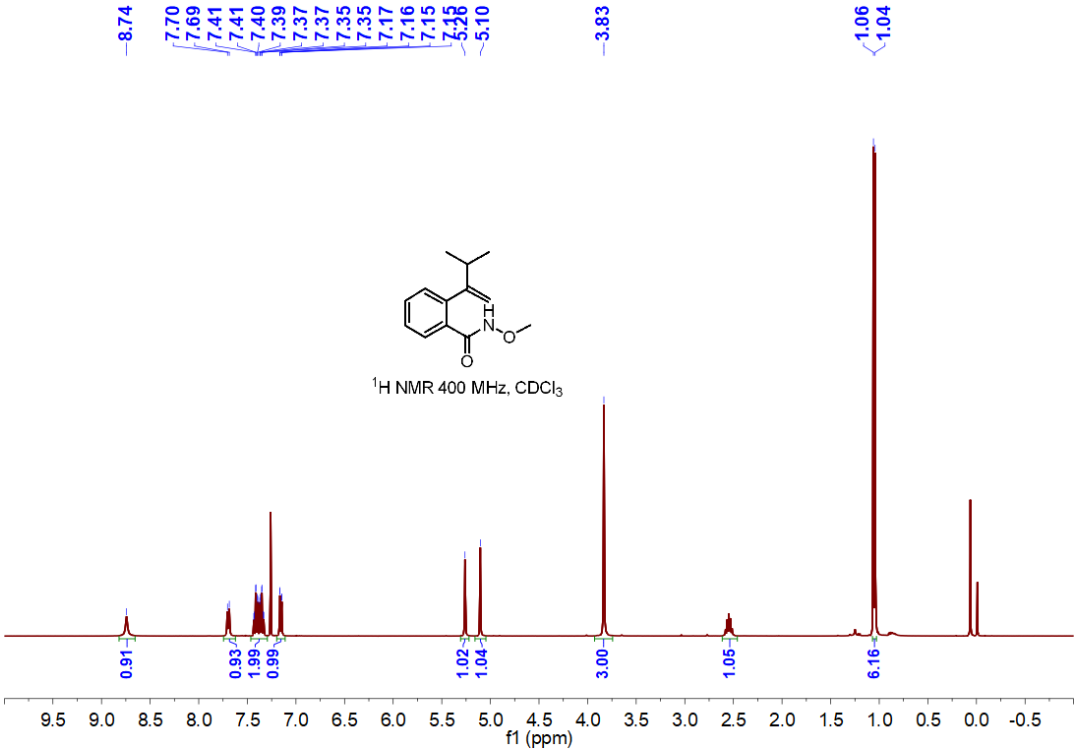
^{13}C NMR 100 MHz $CDCl_3$

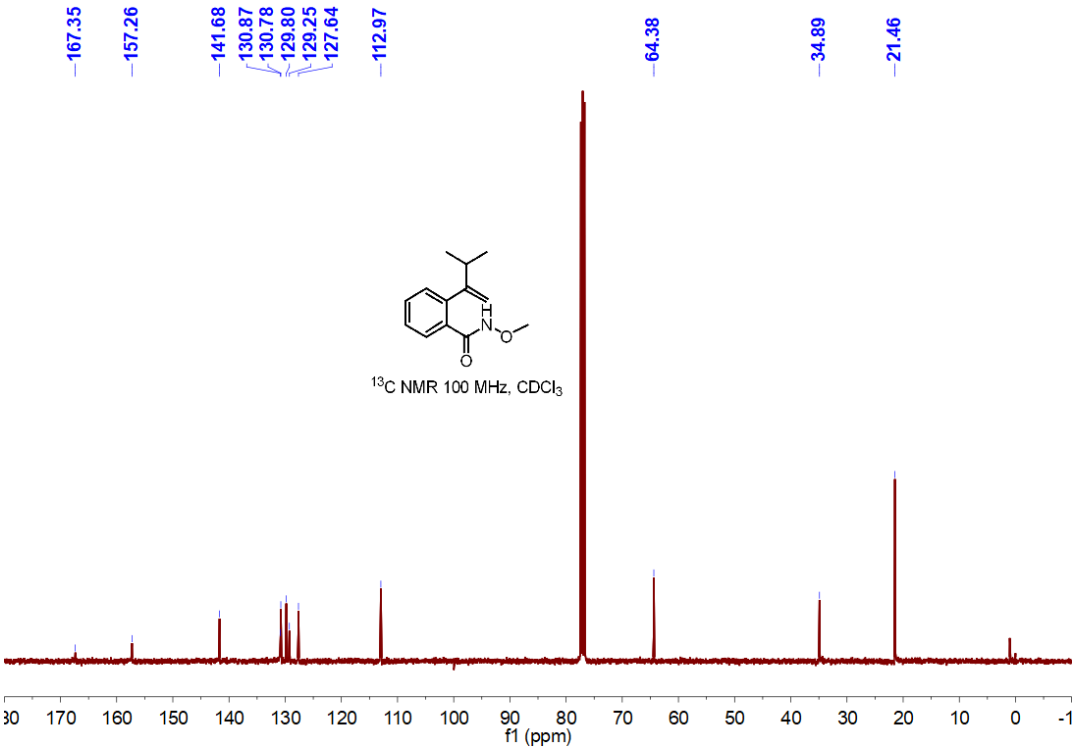


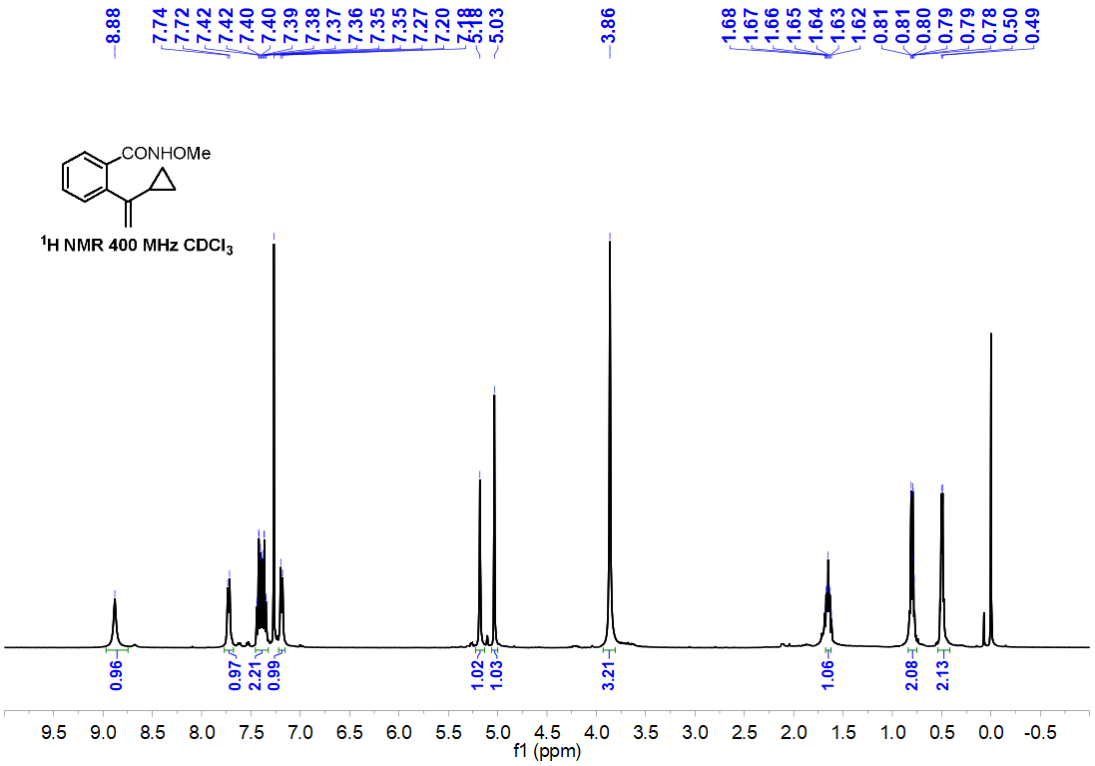
^{1}H NMR 400 MHz $CDCl_3$

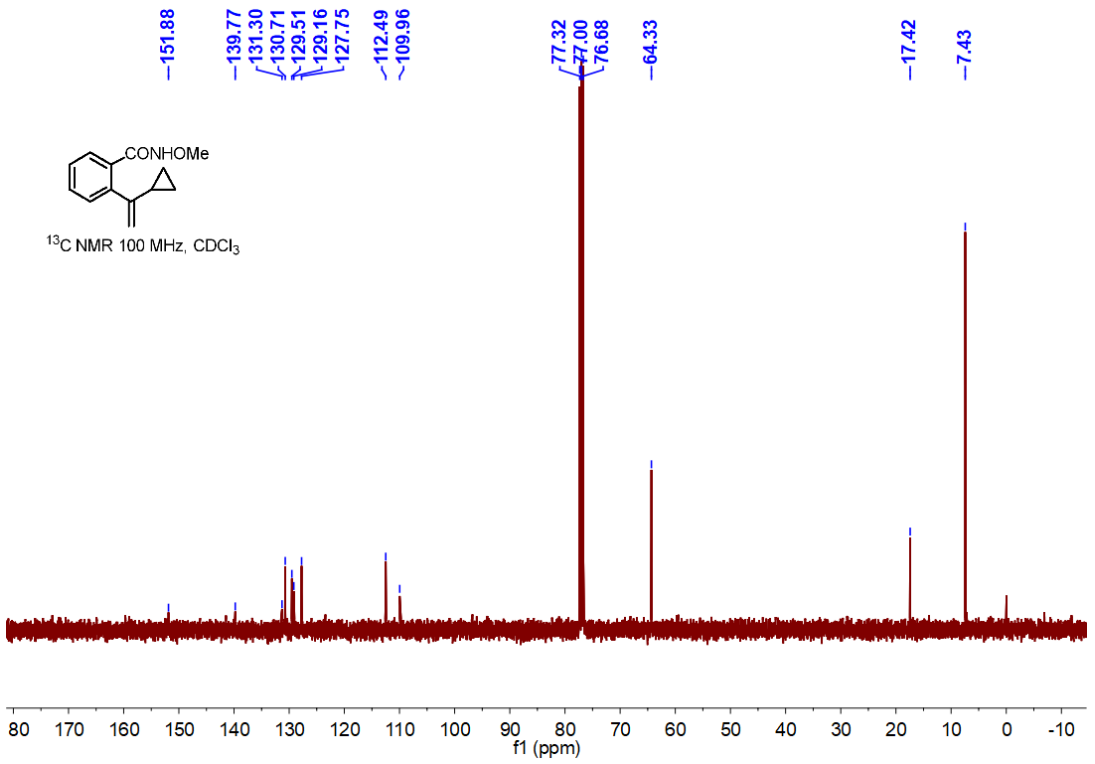


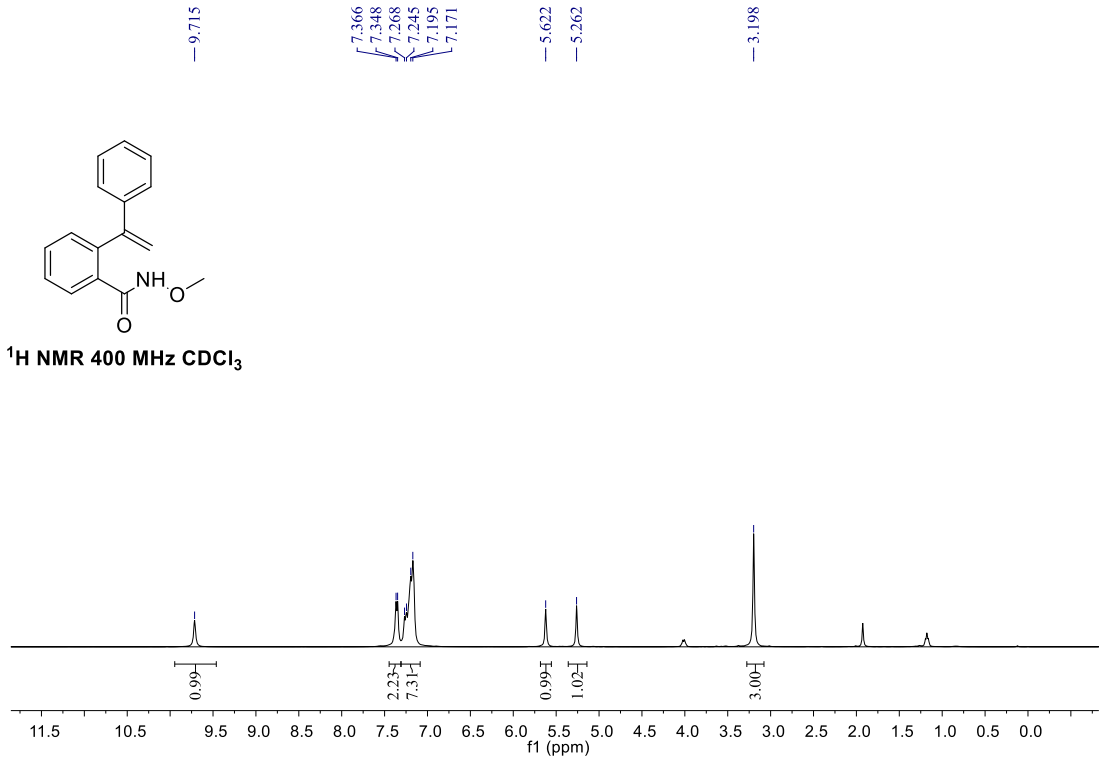
^{13}C NMR 100 MHz $CDCl_3$


8.74
7.70
7.69
7.41
7.41
7.40
7.39
7.37
7.37
7.35
7.35
7.17
7.16
7.15
5.26
5.10
3.83
1.06
1.04
0.91
0.93
1.99
0.99
1.02
1.04
3.00
1.05
6.16
^{1}H NMR 400 MHz, $CDCl_3$
f1 (ppm)

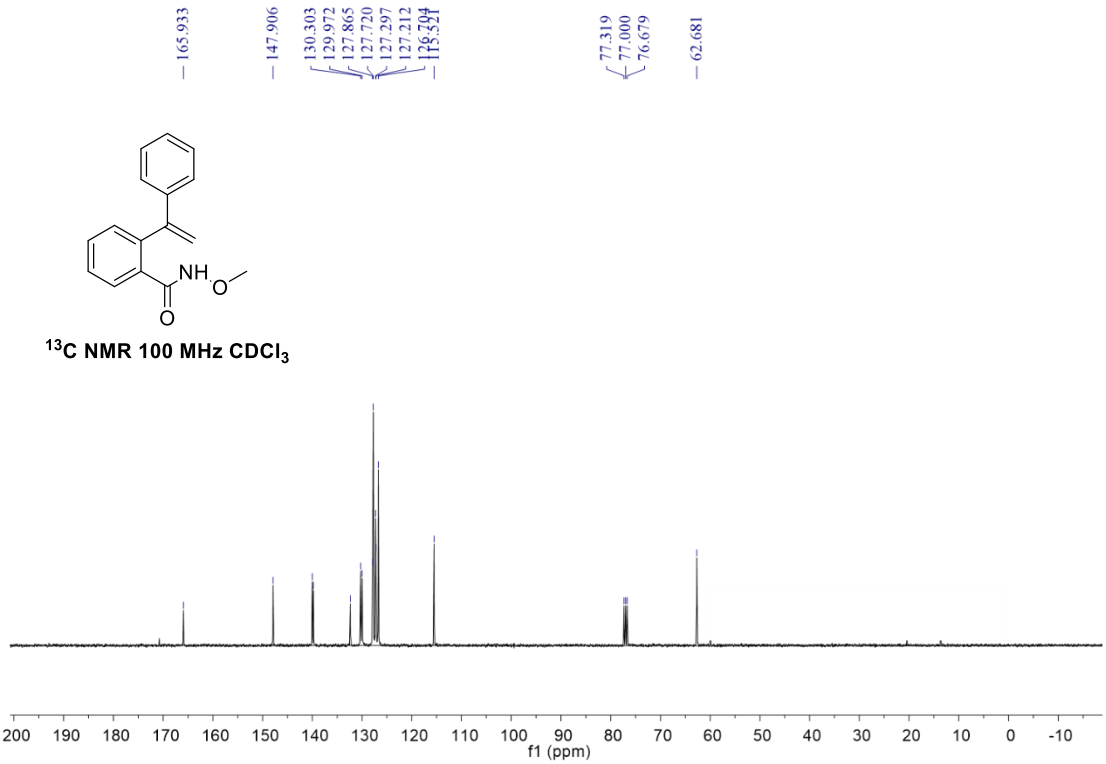

167.35
157.26
141.68
130.87
130.78
129.80
129.25
127.64
112.97
64.38
34.89
21.46
^{13}C NMR 100 MHz, $CDCl_3$
f1 (ppm)


8.88
7.74
7.72
7.42
7.42
7.40
7.40
7.39
7.38
7.37
7.36
7.35
7.35
7.27
7.20
5.18
5.03
3.86
1.68
1.67
1.66
1.65
1.64
1.63
1.62
0.81
0.81
0.80
0.79
0.79
0.78
0.50
0.49
CONHOMe
1H NMR 400 MHz CDCl3
0.96
0.97
2.21
0.99
1.02
1.03
3.21
1.06
2.08
2.13
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

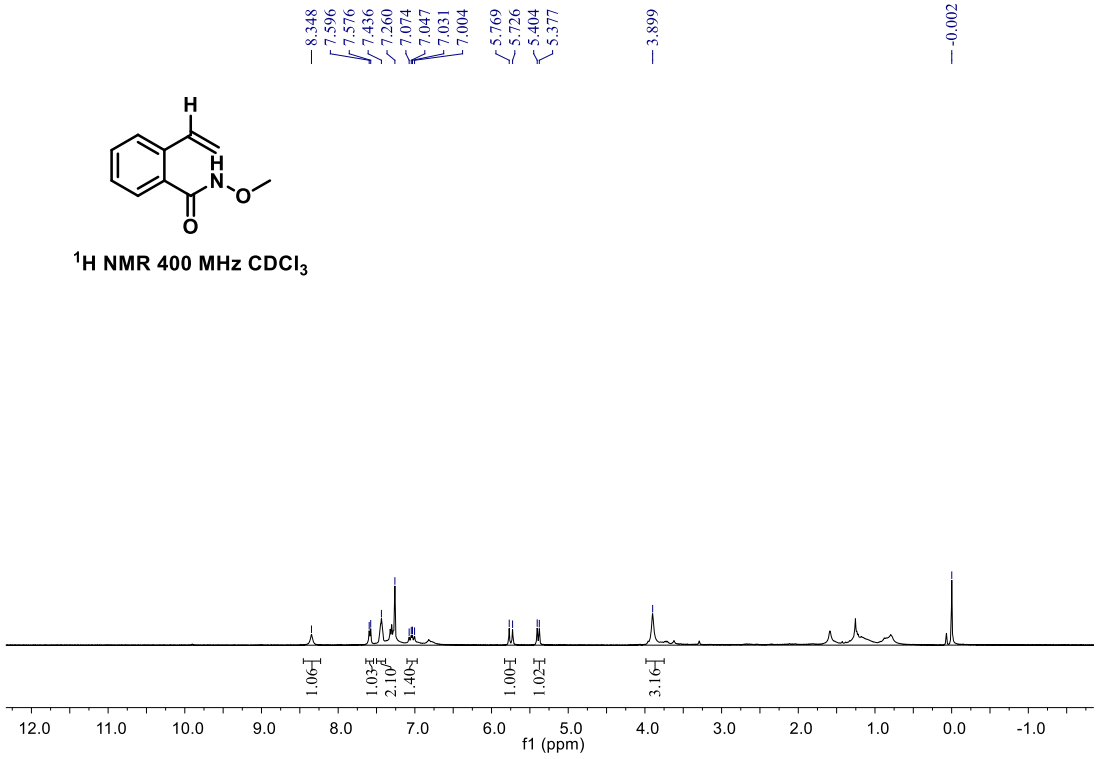

151.88
139.77
131.30
130.71
129.51
129.16
127.75
112.49
109.96
77.32
77.00
76.68
64.33
17.42
7.43
CONHOMe
13C NMR 100 MHz, CDCl3
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



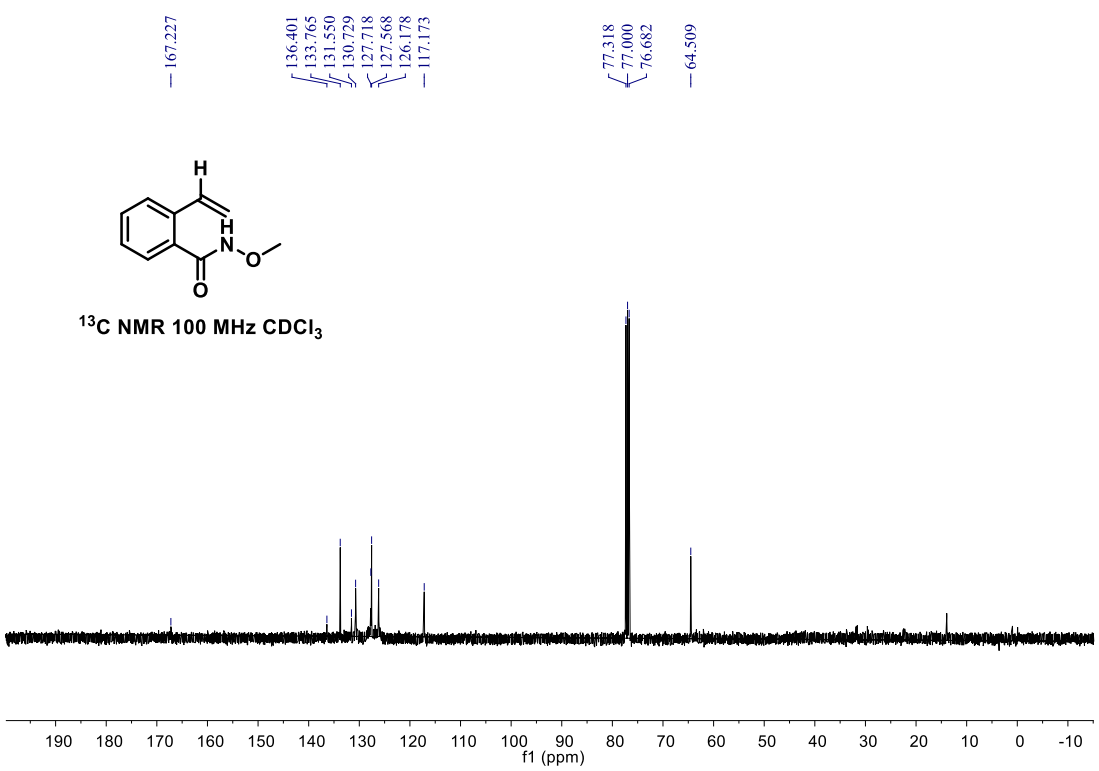
^{1}H NMR 400 MHz $CDCl_3$



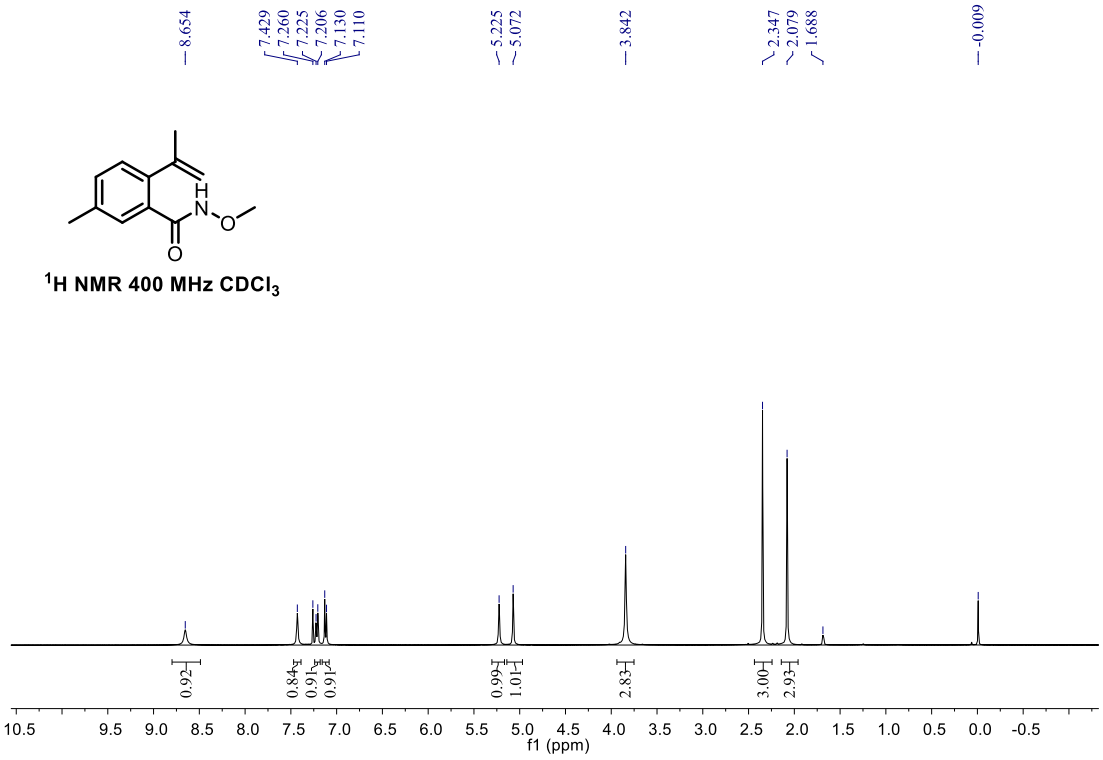
^{13}C NMR 100 MHz $CDCl_3$



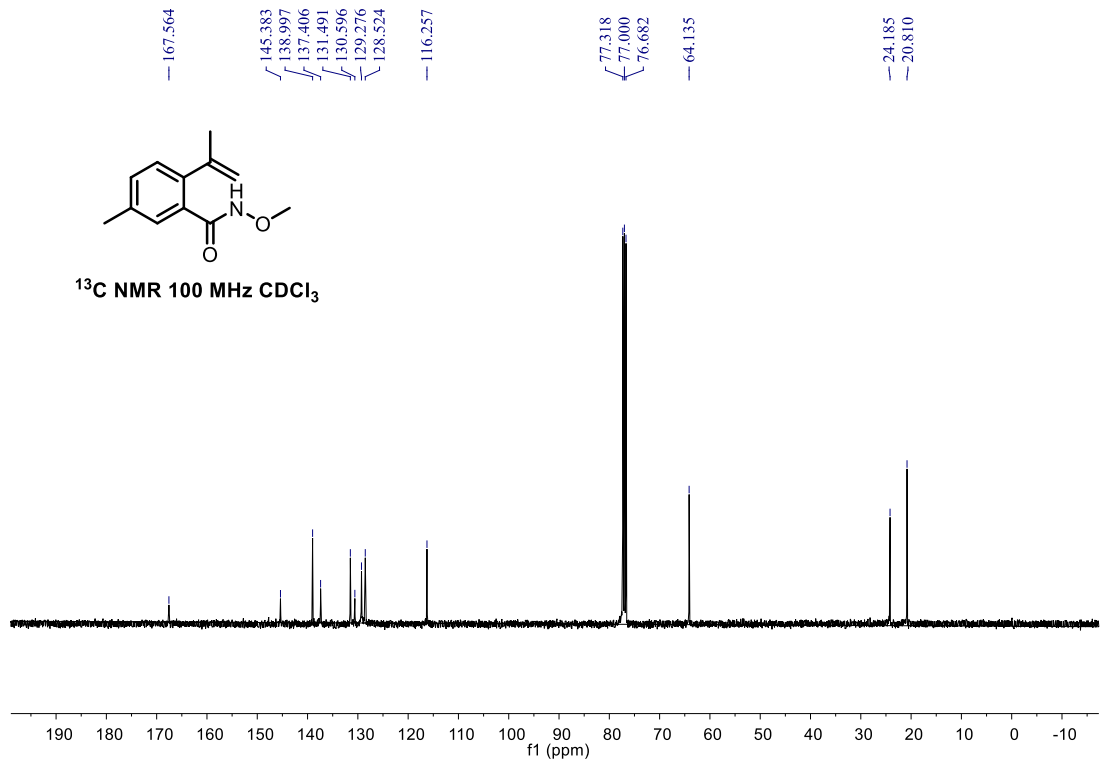
^{1}H NMR 400 MHz $CDCl_3$



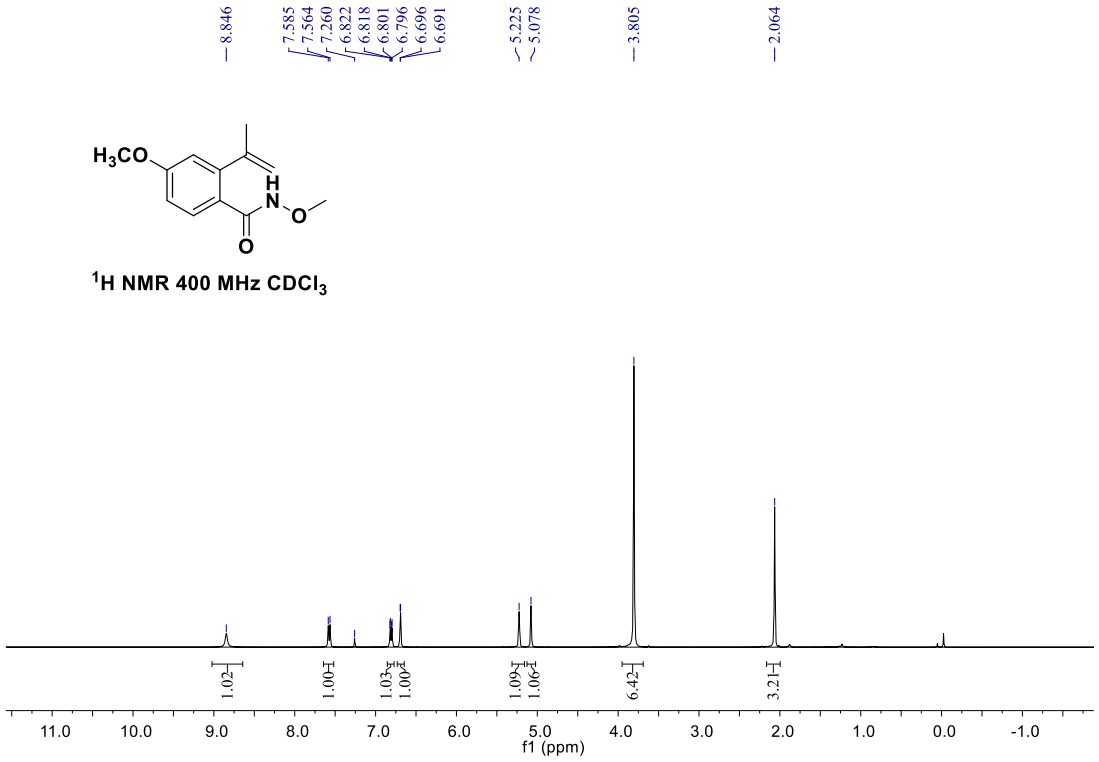
^{13}C NMR 100 MHz $CDCl_3$



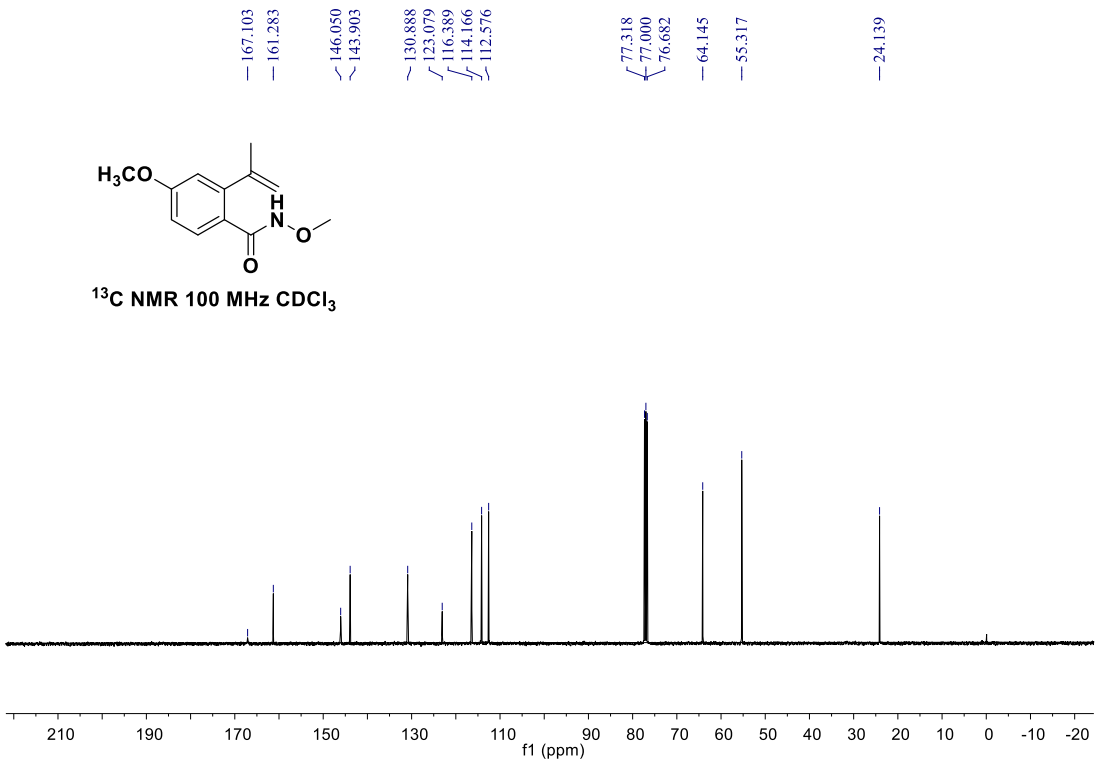
^{1}H NMR 400 MHz $CDCl_3$



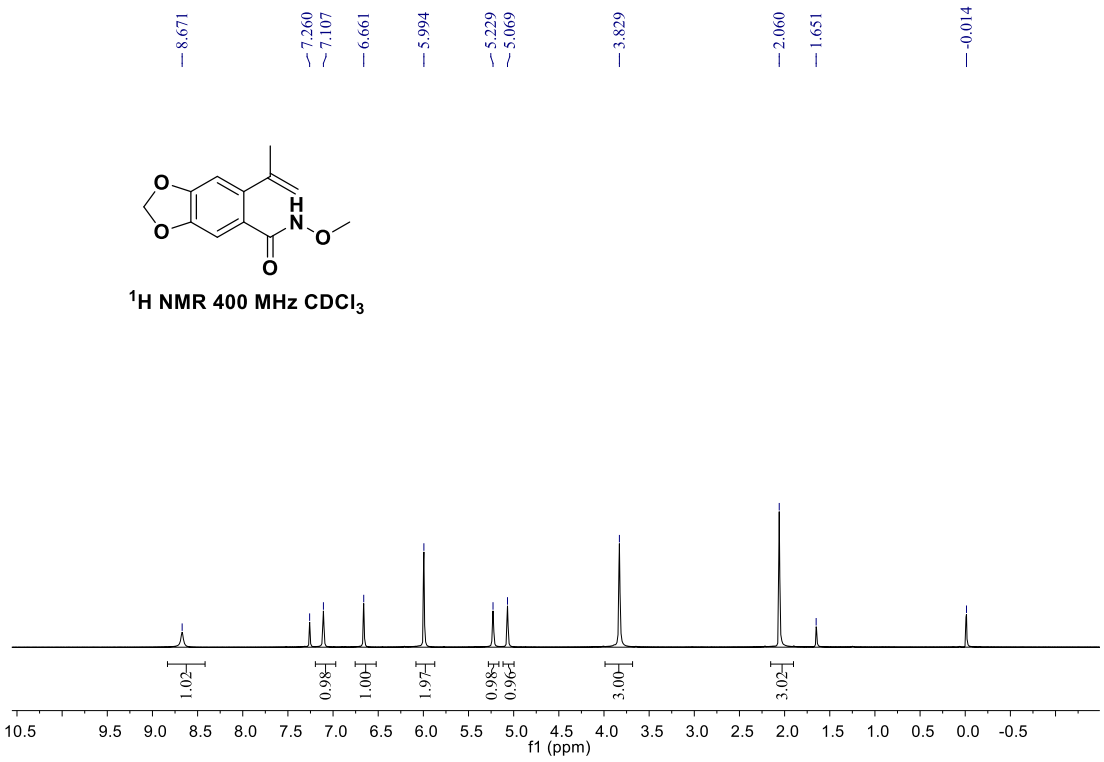
^{13}C NMR 100 MHz $CDCl_3$



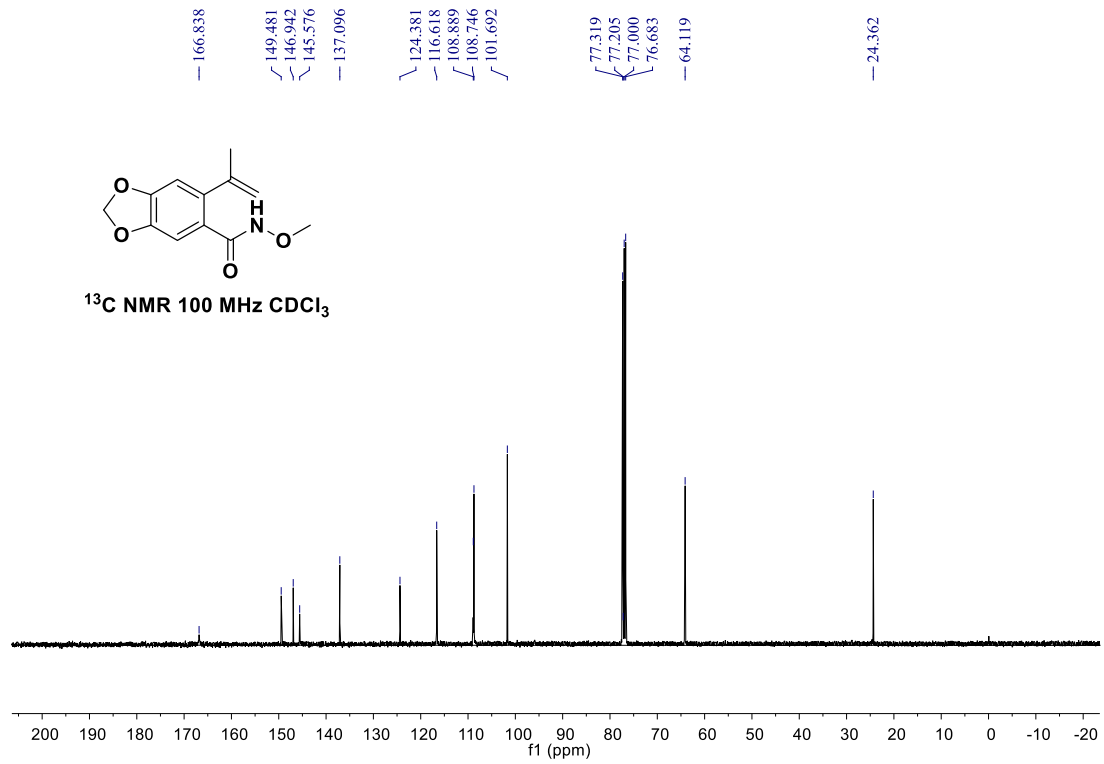
^{1}H NMR 400 MHz $CDCl_3$



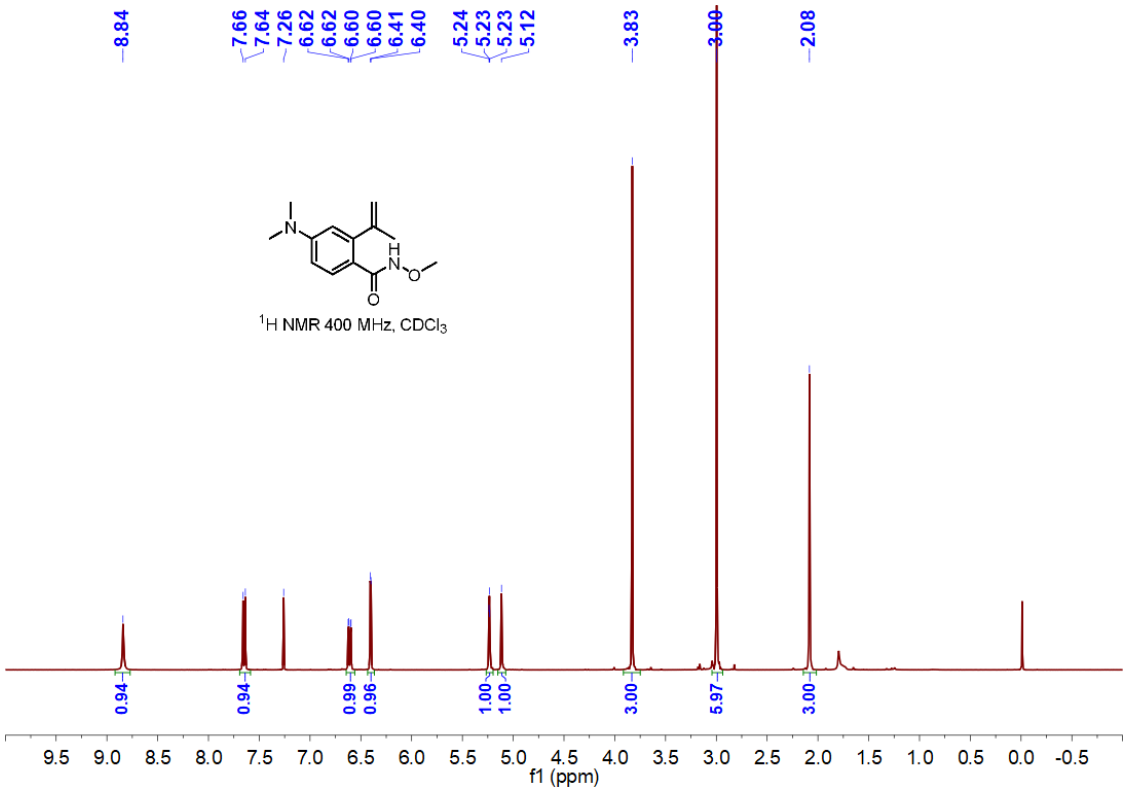
^{13}C NMR 100 MHz $CDCl_3$



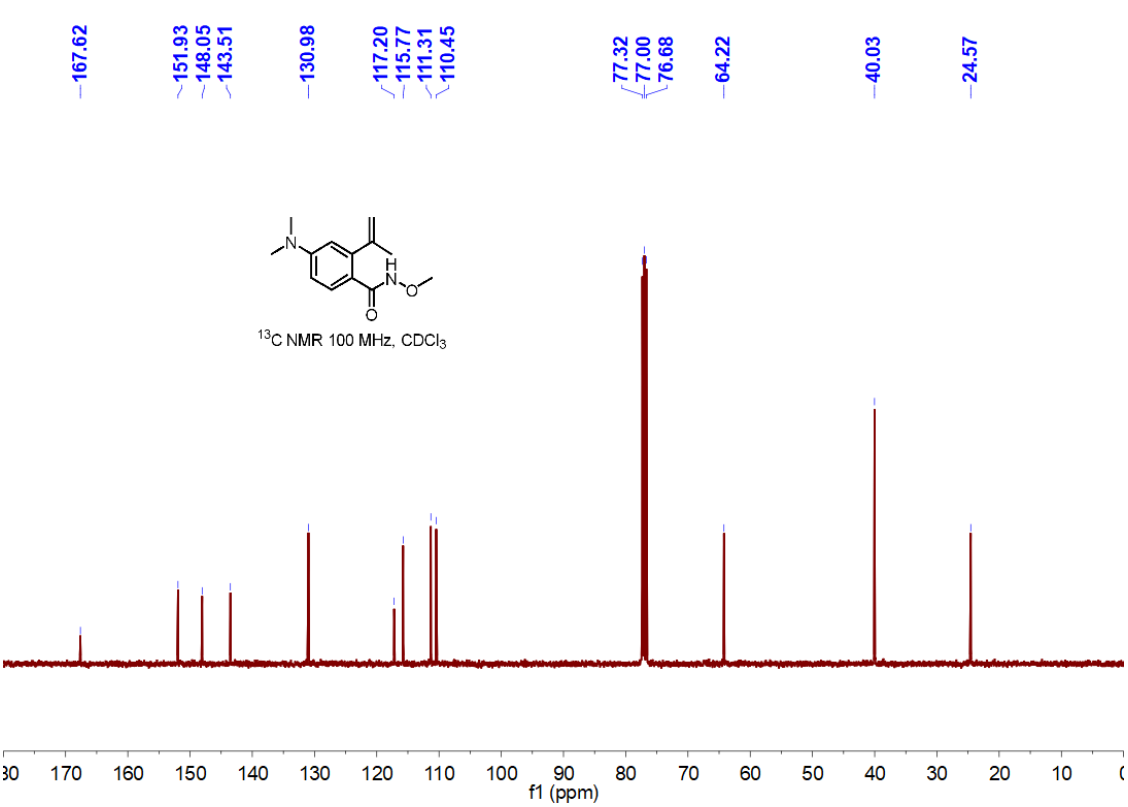
^{1}H NMR 400 MHz $CDCl_3$



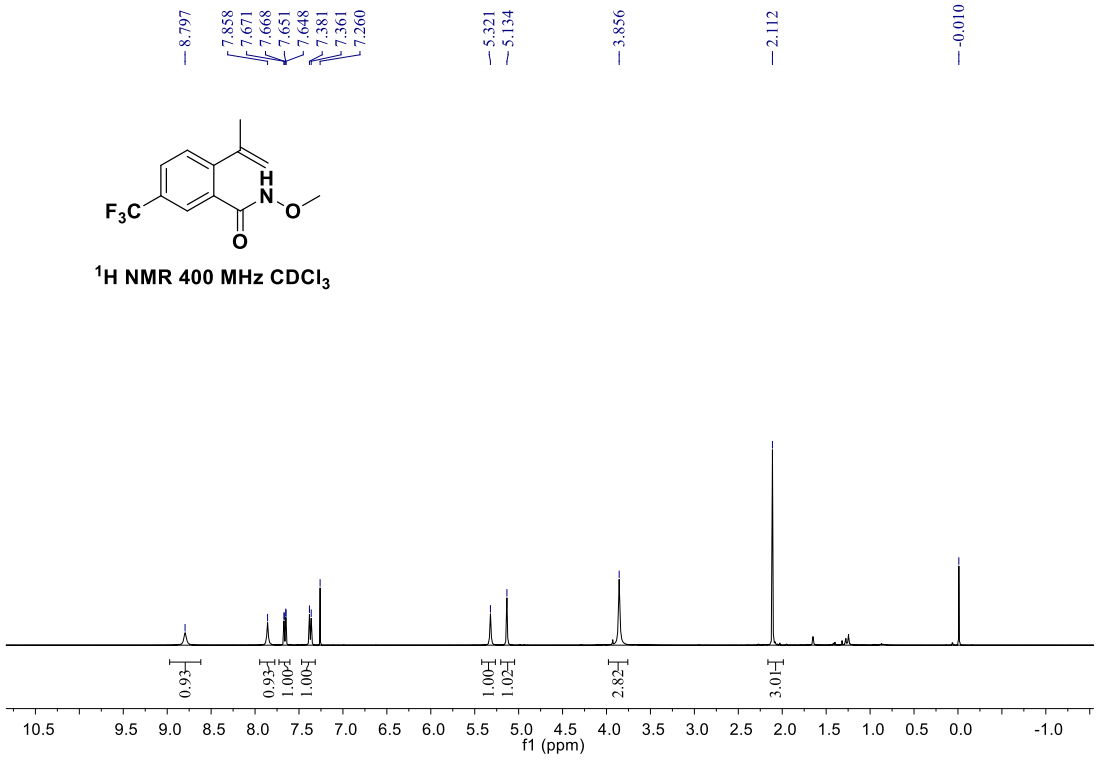
^{13}C NMR 100 MHz $CDCl_3$



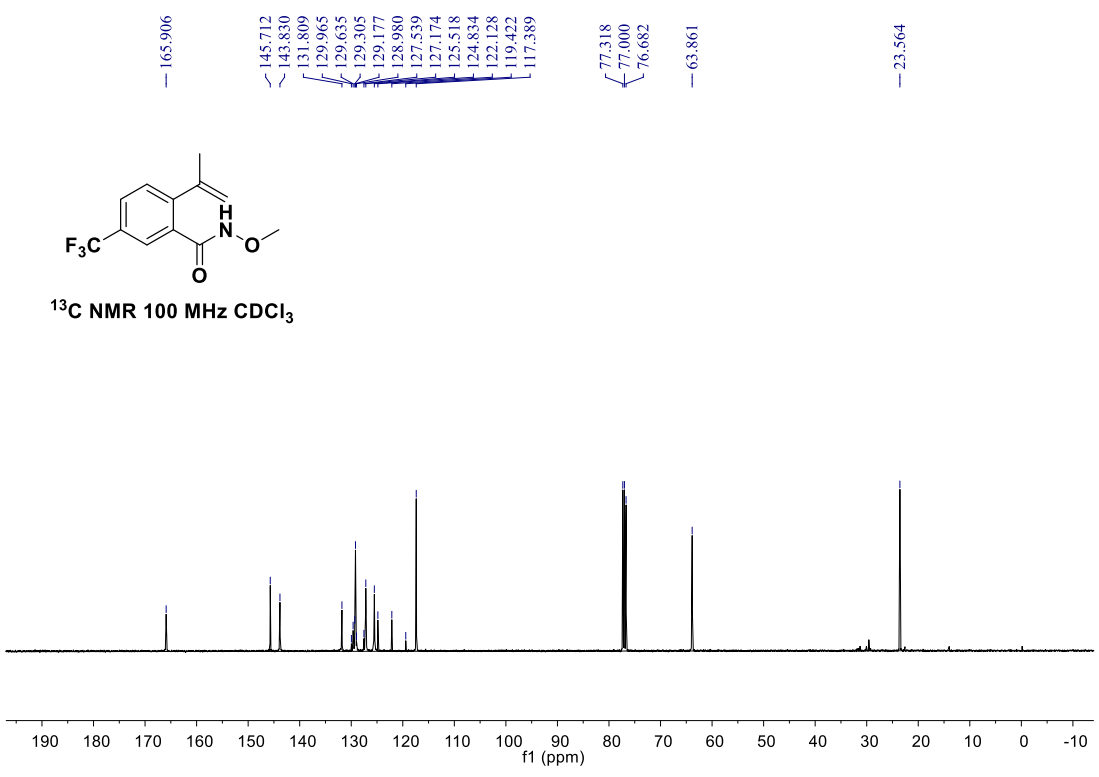
^{1}H NMR 400 MHz, $CDCl_3$



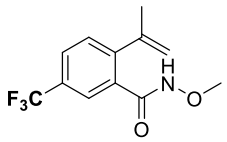
^{13}C NMR 100 MHz, $CDCl_3$



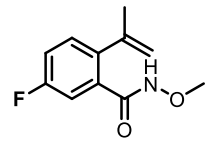
^{1}H NMR 400 MHz $CDCl_3$



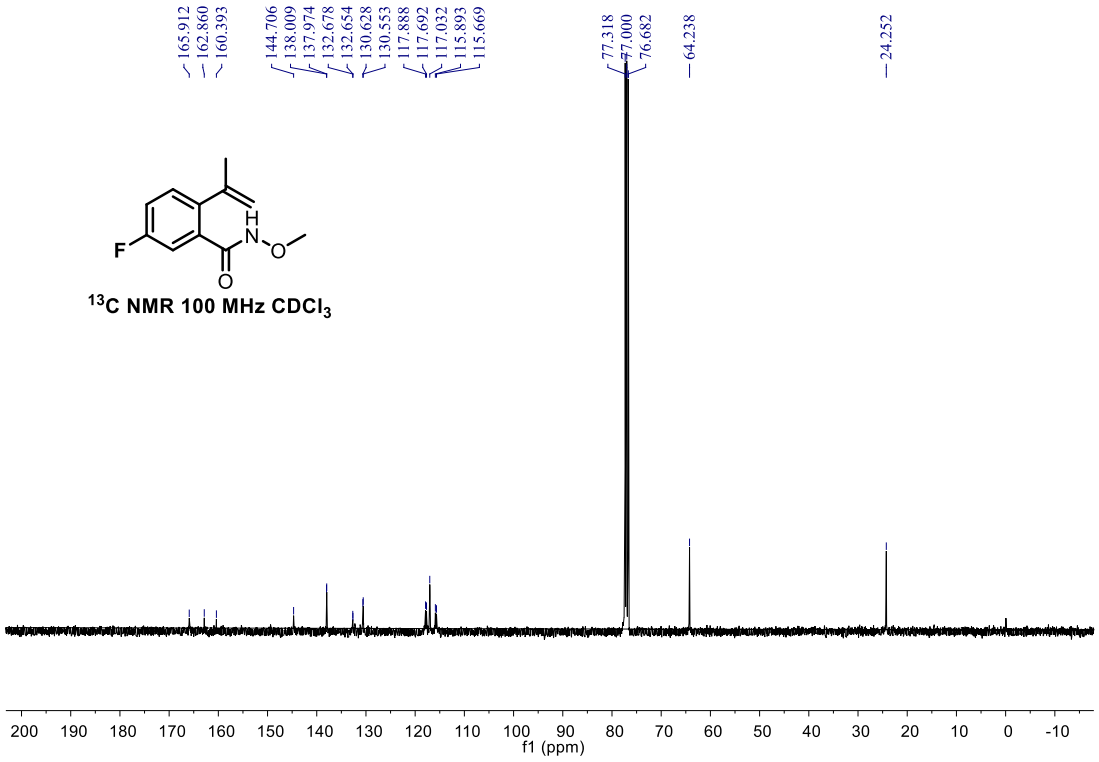
^{13}C NMR 100 MHz $CDCl_3$



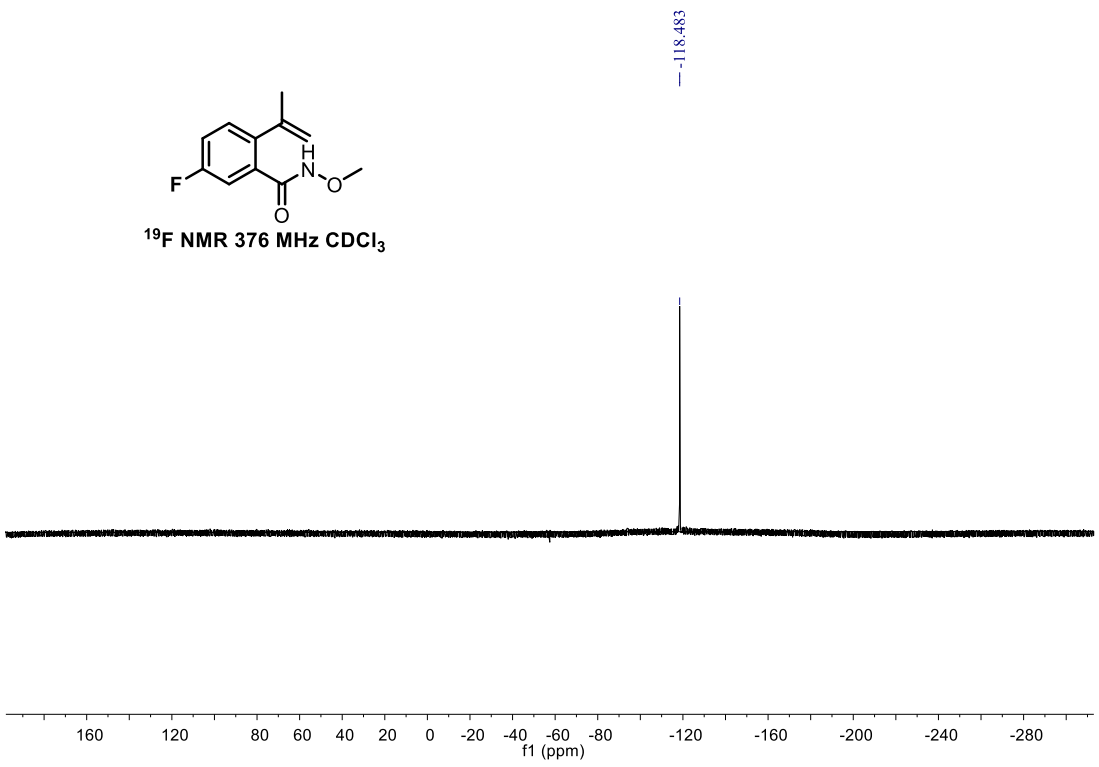
^{19}F NMR 376 MHz $CDCl_3$



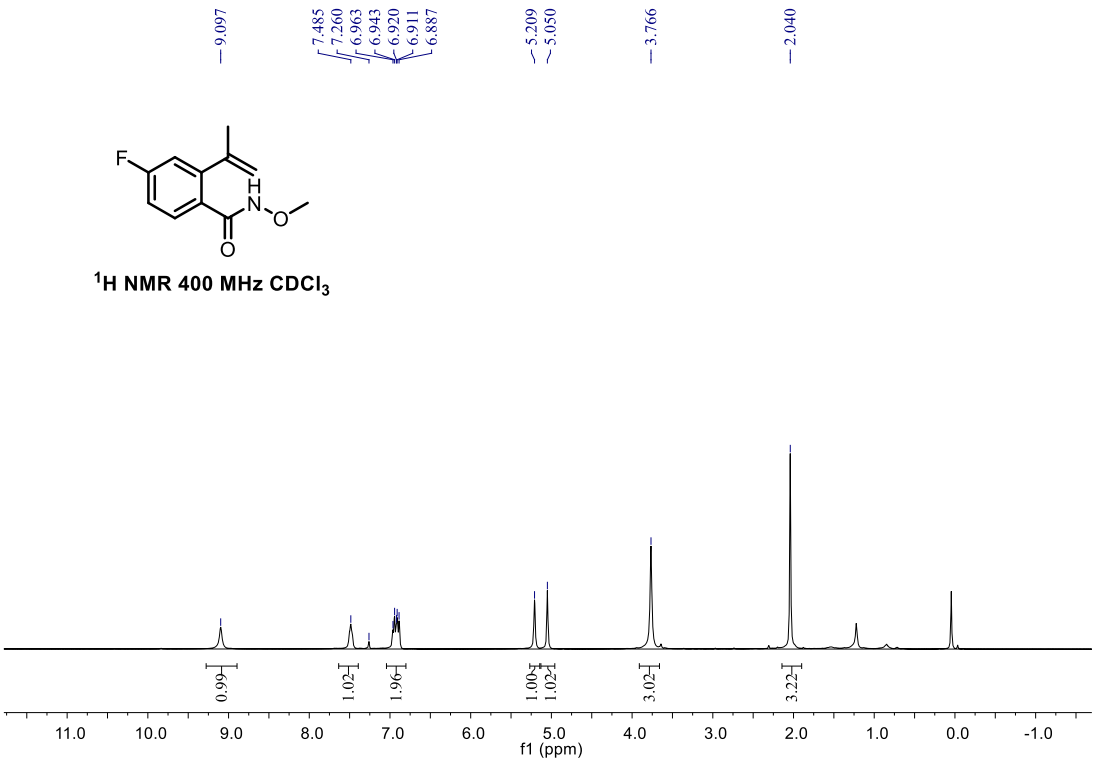
^{1}H NMR 400 MHz $CDCl_3$



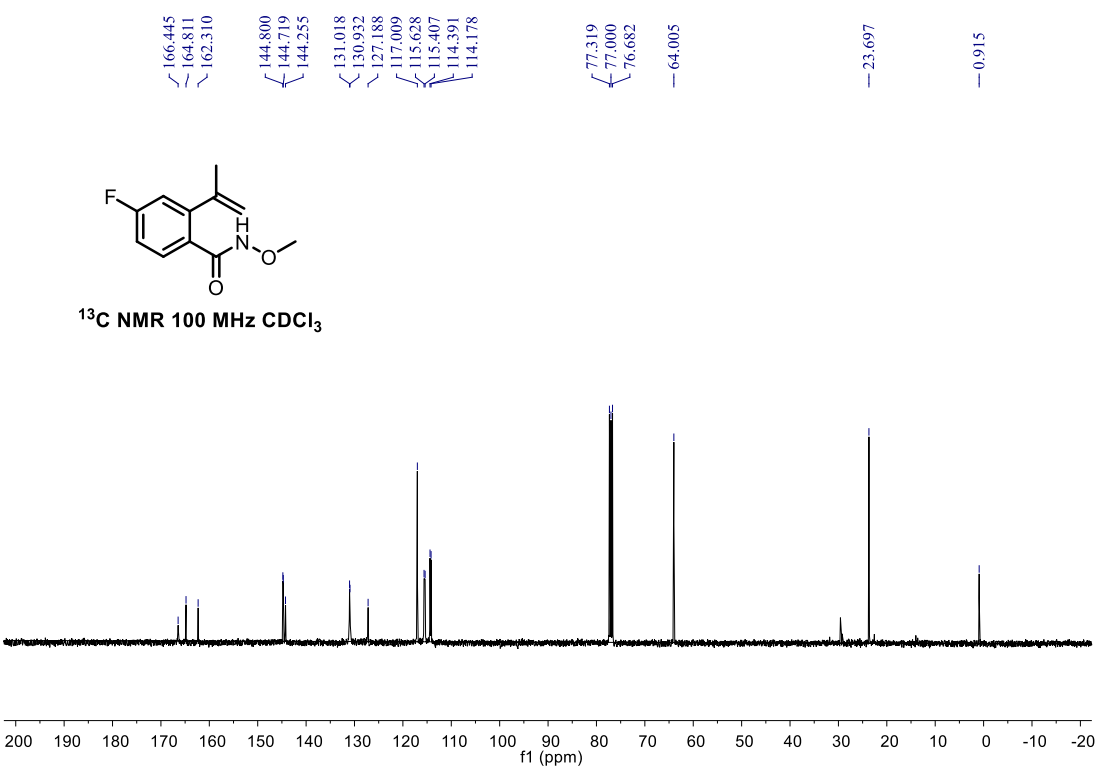
^{13}C NMR 100 MHz $CDCl_3$



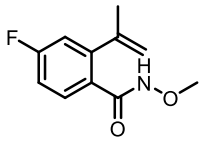
^{19}F NMR 376 MHz $CDCl_3$



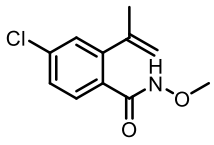
^{1}H NMR 400 MHz $CDCl_3$



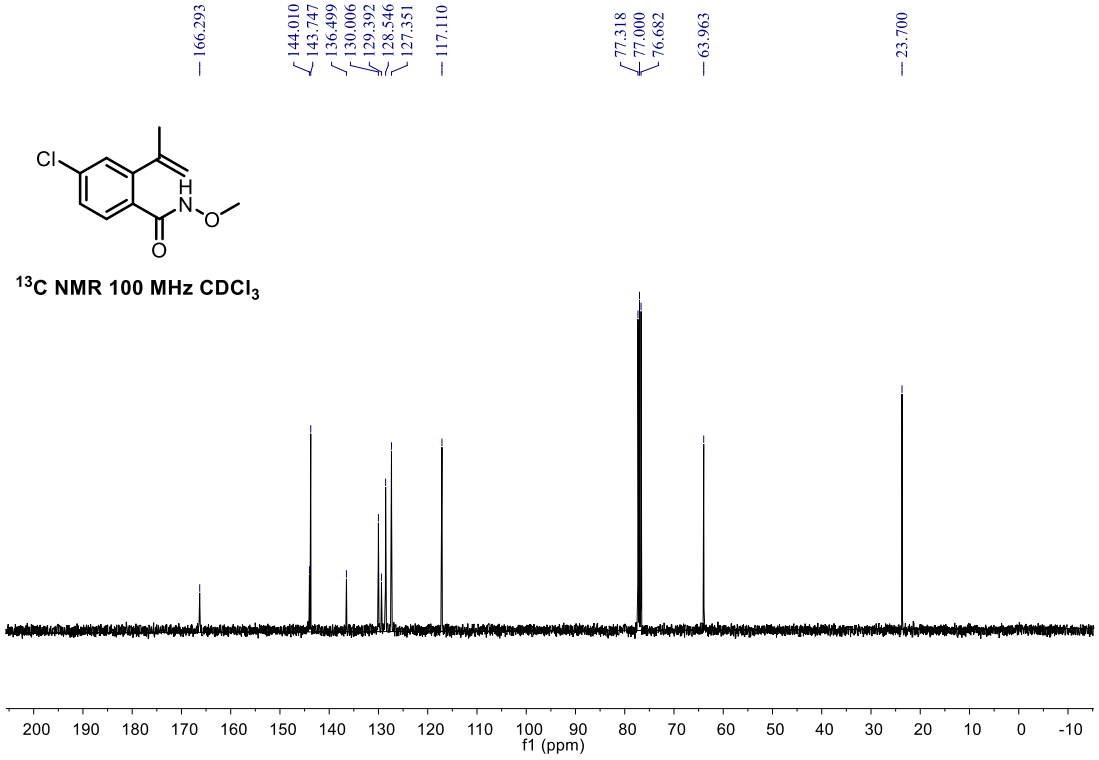
^{13}C NMR 100 MHz $CDCl_3$

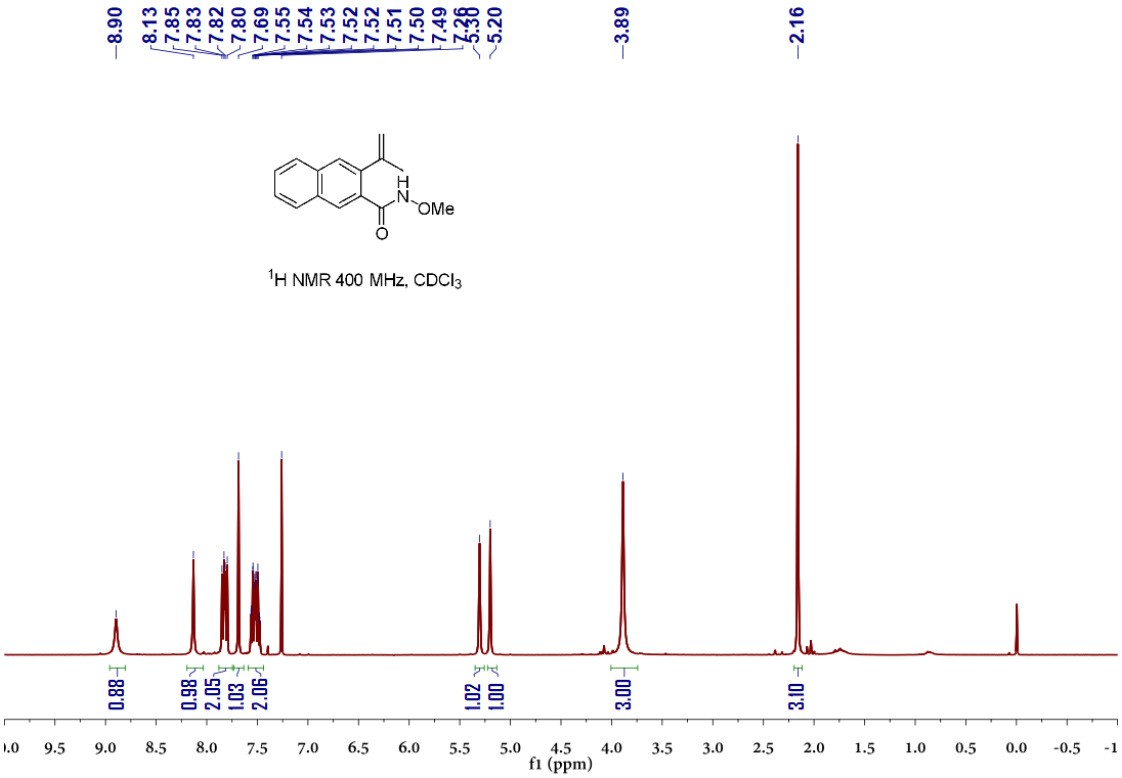


^{19}F NMR 376 MHz $CDCl_3$



^{1}H NMR 400 MHz $CDCl_3$


166.293
144.010
143.747
136.499
130.006
129.392
128.546
127.351
117.110
77.318
77.000
76.682
63.963
23.700
Cl
H
N
O
O
13C NMR 100 MHz CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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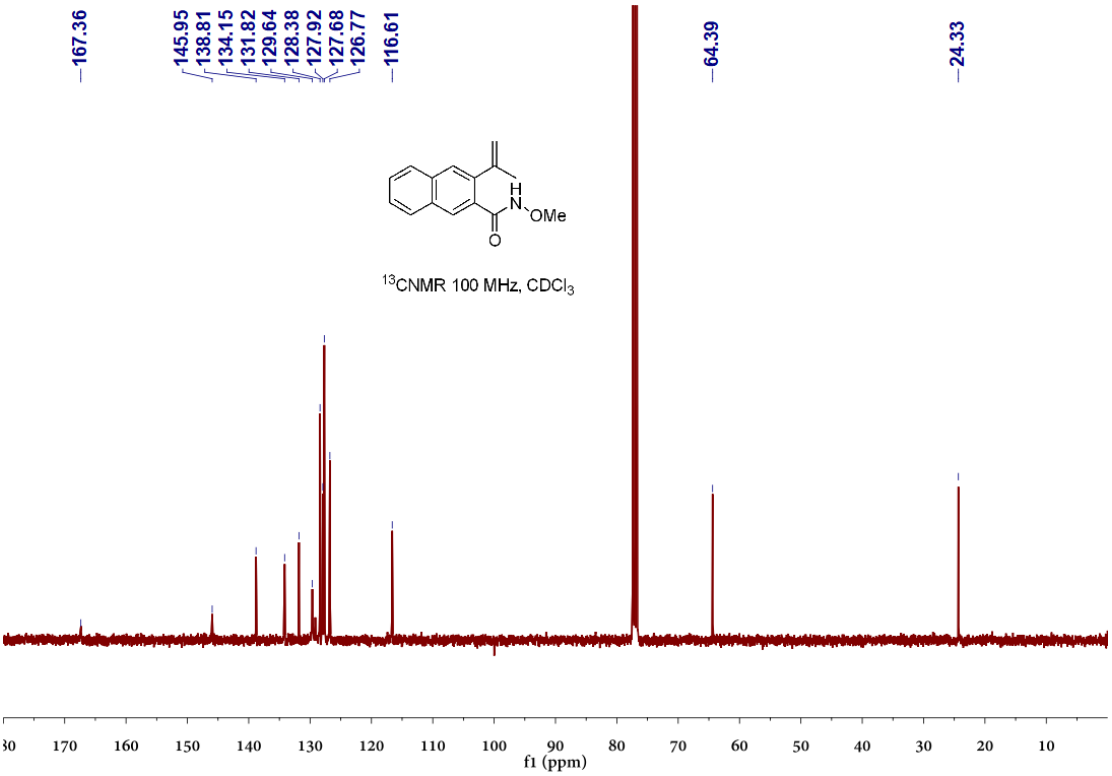
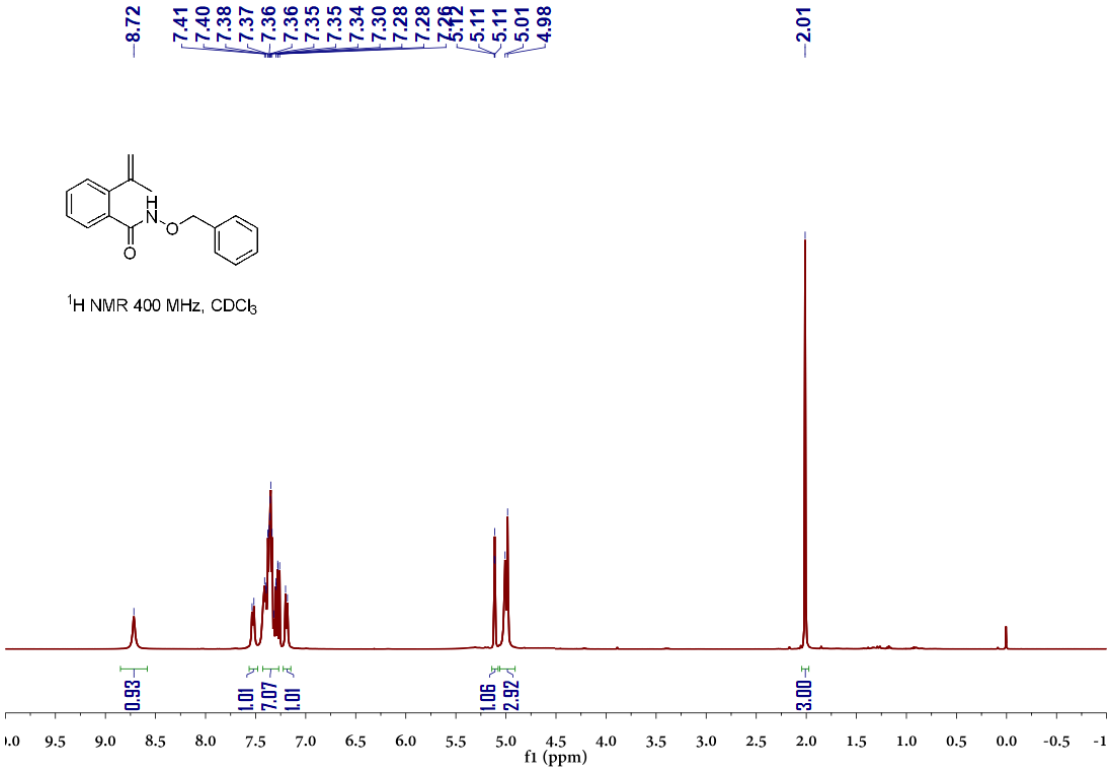
8.13
7.85
7.83
7.82
7.80
7.69
7.55
7.54
7.53
7.52
7.52
7.51
7.50
7.49
7.26
5.30
5.20
3.89
2.16
H
N
OMe
O
1H NMR 400 MHz, CDCl3
0.88
0.98
2.05
1.03
2.06
1.02
1.00
3.00
3.10
1.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


167.36
145.95
138.81
134.15
131.82
129.64
128.38
127.92
127.68
126.77
116.61
64.39
24.33
OMe
13CNMR 100 MHz, CDCl3
f1 (ppm)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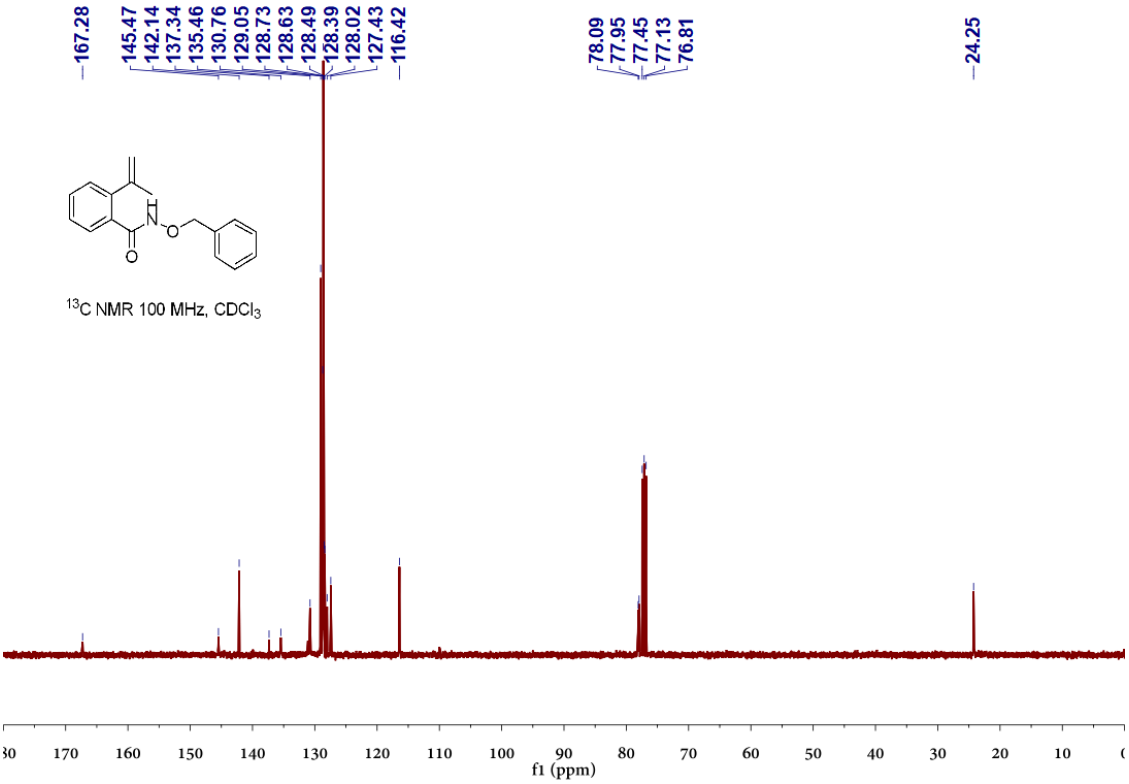
7.36
7.36
7.35
7.35
7.34
7.30
7.28
7.28
5.11
5.11
5.01
4.98
2.01
0.93
1.01
7.07
1.01
1.06
2.92
3.00
1H NMR 400 MHz, CDCl3
f1 (ppm)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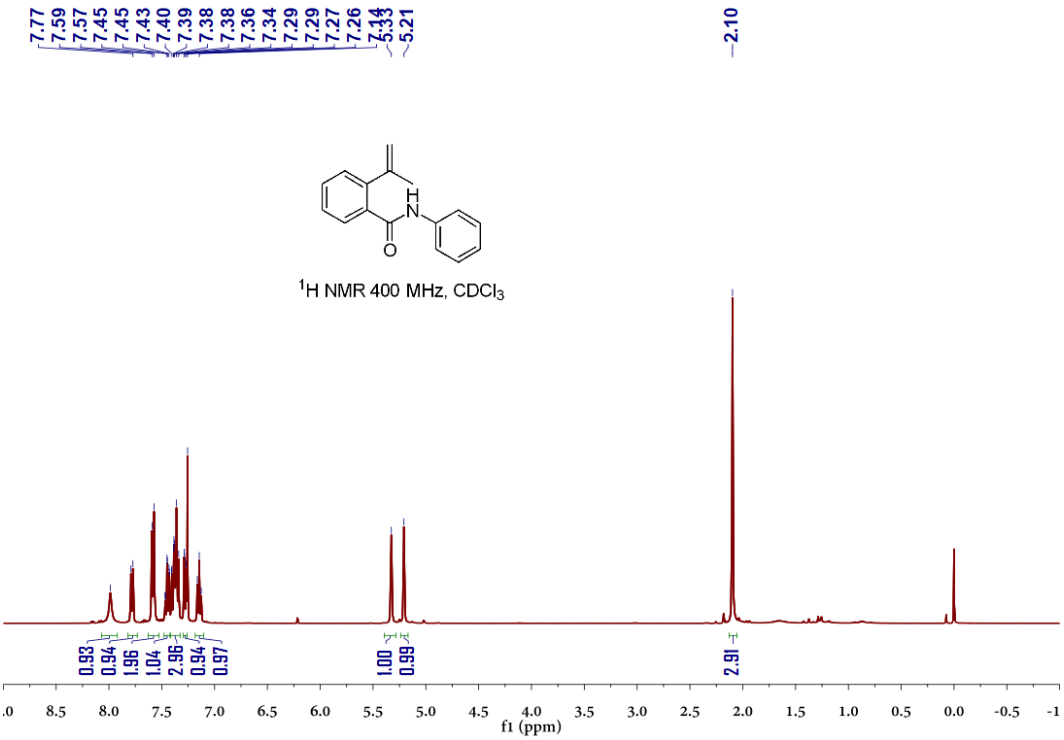
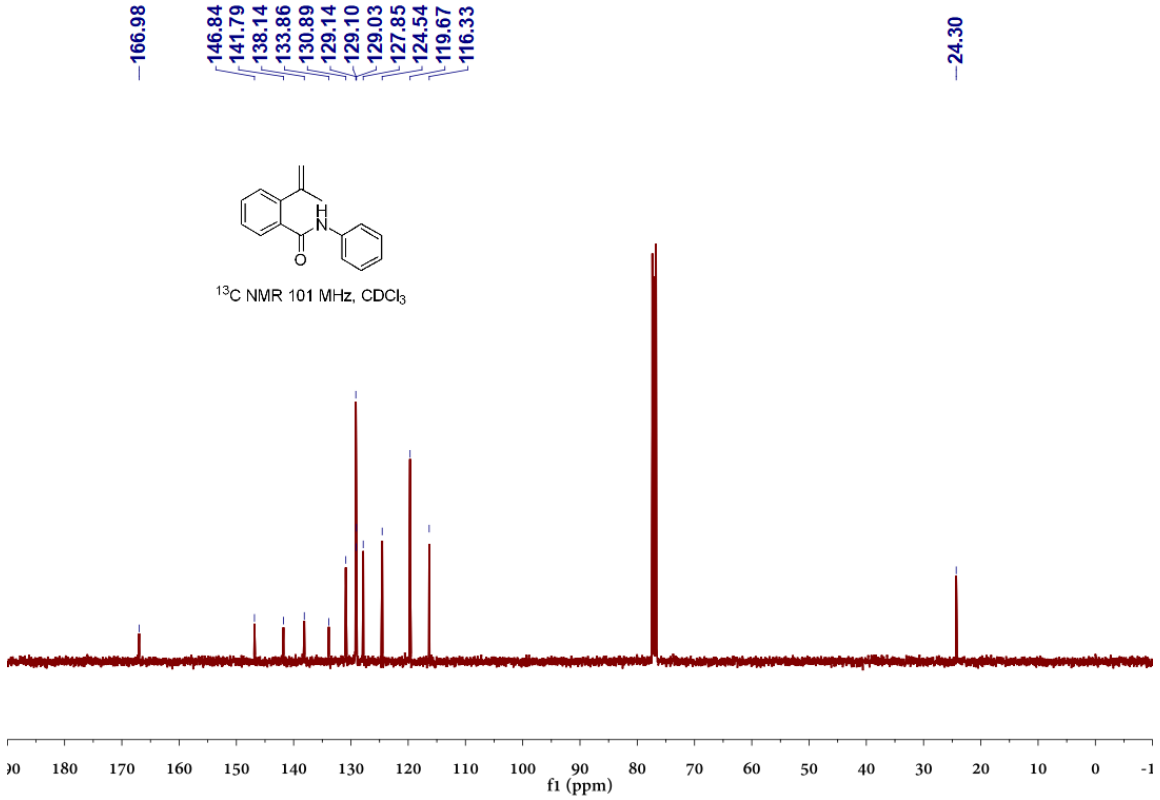
145.47
142.14
137.34
135.46
130.76
129.05
128.73
128.63
128.49
128.39
128.02
127.43
116.42
78.09
77.95
77.45
77.13
76.81
24.25
13C NMR 100 MHz, CDCl3
f1 (ppm)


7.77
7.59
7.57
7.45
7.45
7.43
7.40
7.39
7.38
7.38
7.36
7.34
7.29
7.29
7.27
7.26
5.33
5.21
2.10
1H NMR 400 MHz, CDCl3
0.93
0.94
1.96
1.04
2.96
0.94
0.97
1.00
0.99
2.91
f1 (ppm)


166.98
146.84
141.79
138.14
133.86
130.89
129.14
129.10
129.03
127.85
124.54
119.67
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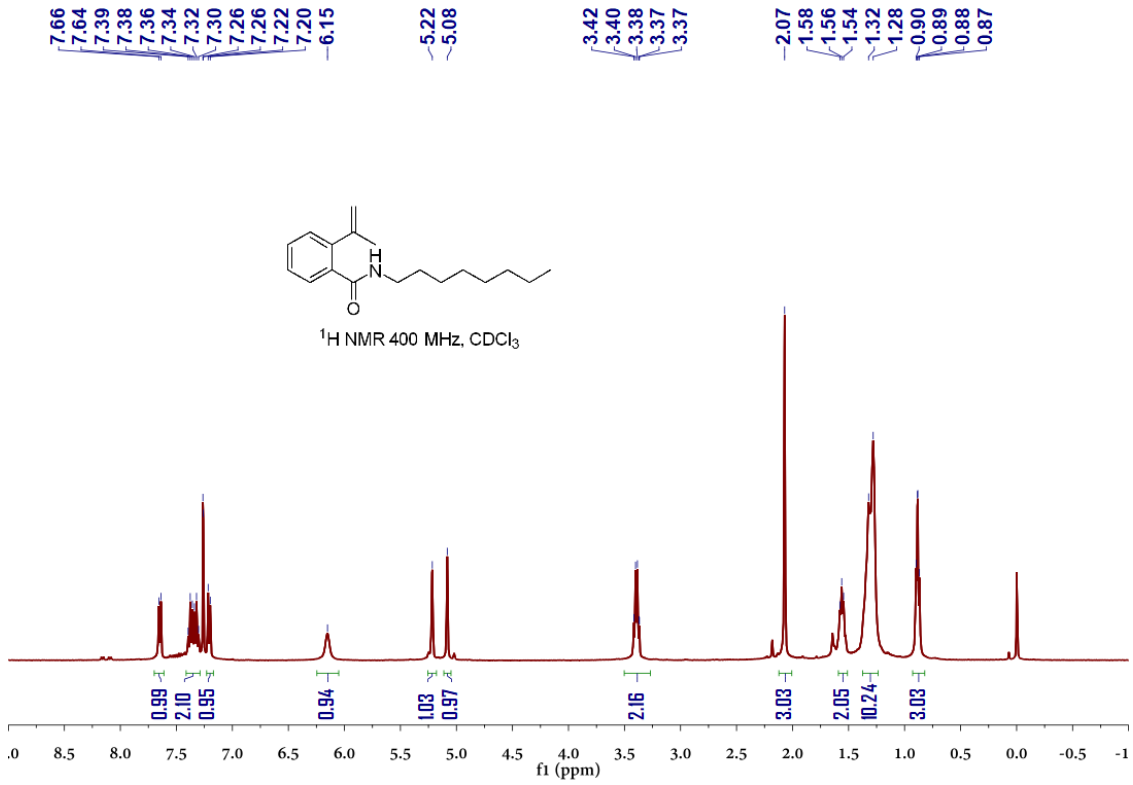
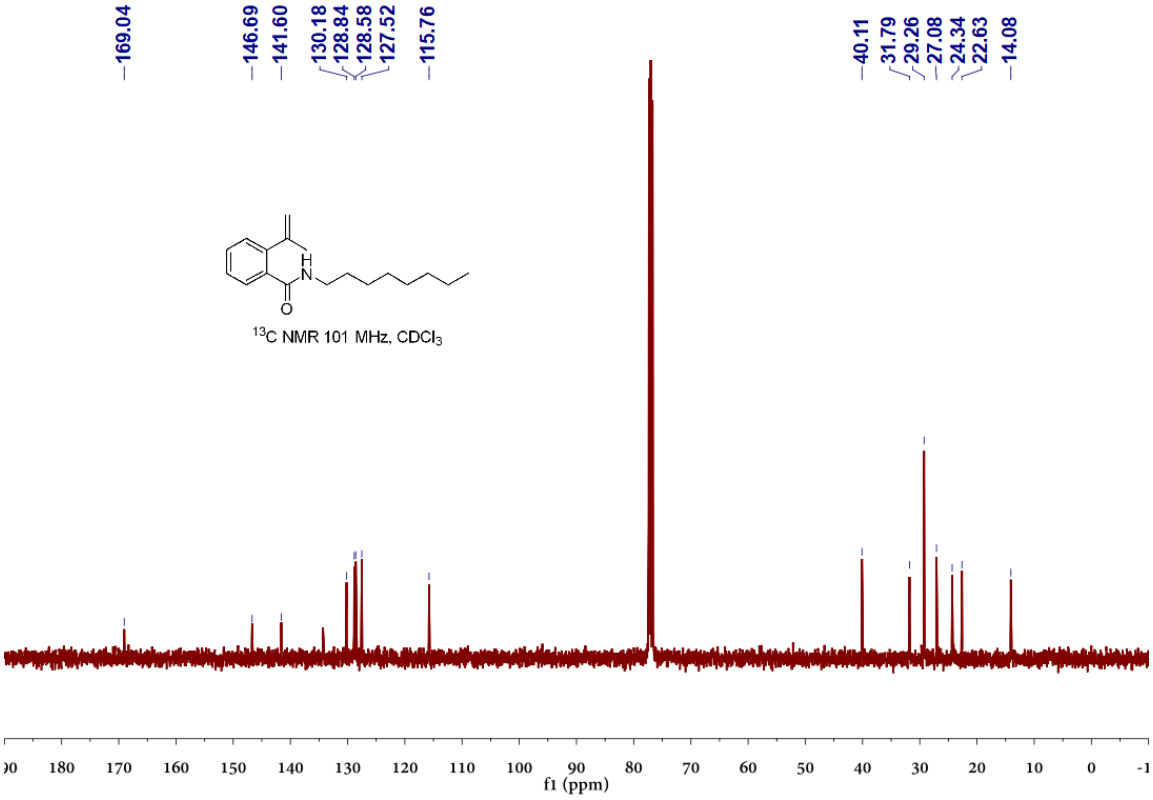
24.30
^{13}C NMR 101 MHz, $CDCl_3$
f1 (ppm)


7.66
7.64
7.39
7.38
7.36
7.34
7.32
7.30
7.26
7.26
7.22
7.20
6.15
5.22
5.08
3.42
3.40
3.38
3.37
3.37
2.07
1.58
1.56
1.54
1.32
1.28
0.90
0.89
0.88
0.87
^{1}H NMR 400 MHz, $CDCl_3$
0.99
2.10
0.95
0.94
1.03
0.97
2.16
3.03
2.05
10.24
3.03
f1 (ppm)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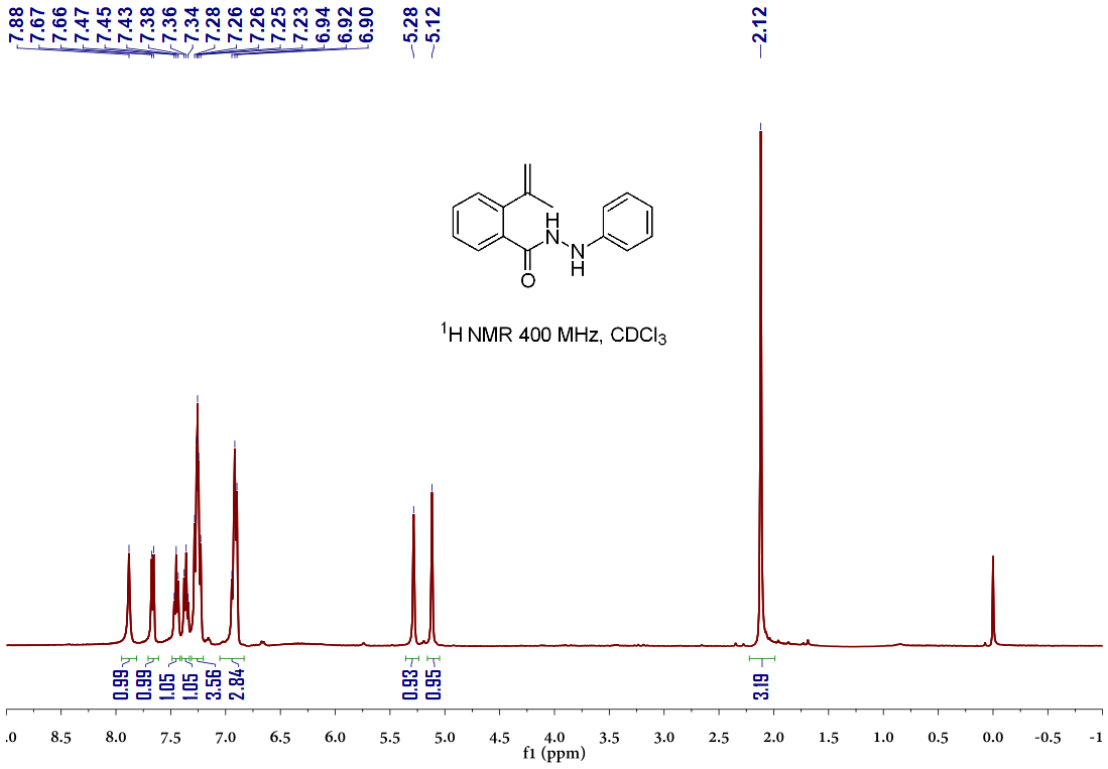
146.69
141.60
130.18
128.84
128.58
127.52
115.76
40.11
31.79
29.26
27.08
24.34
22.63
14.08
H
N
O
13C NMR 101 MHz, CDCl3
f1 (ppm)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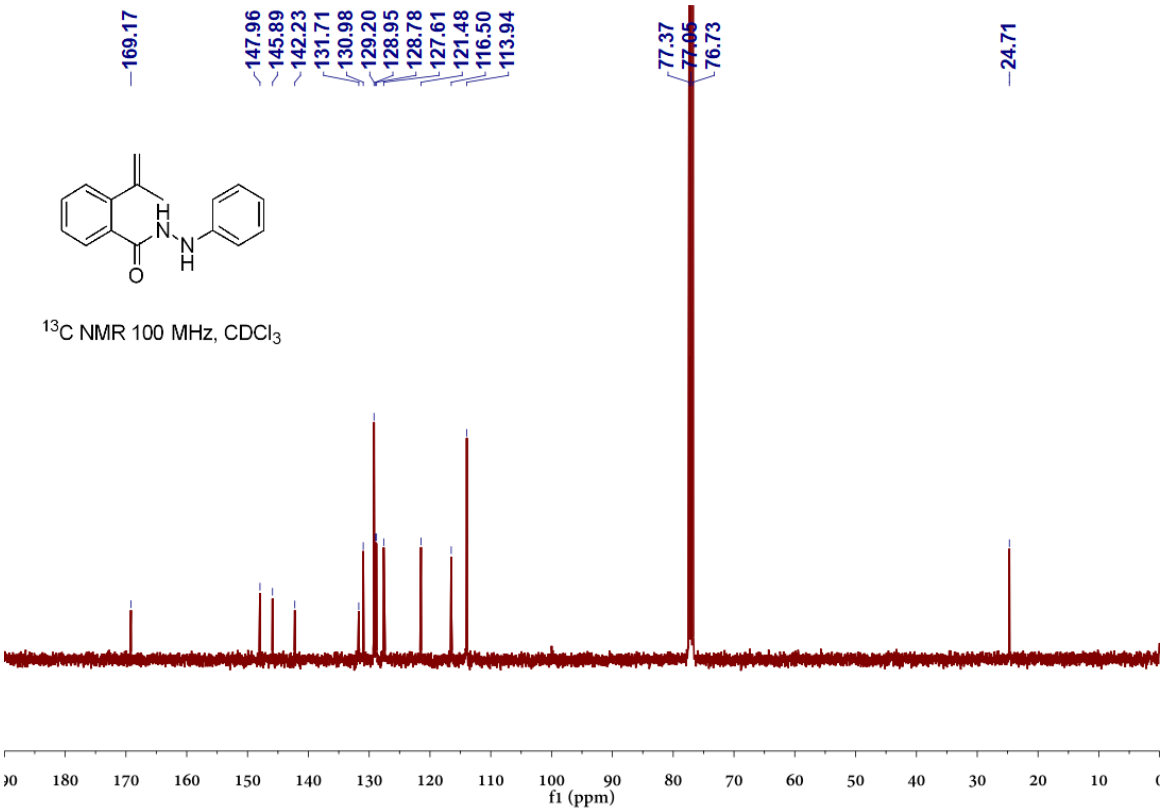
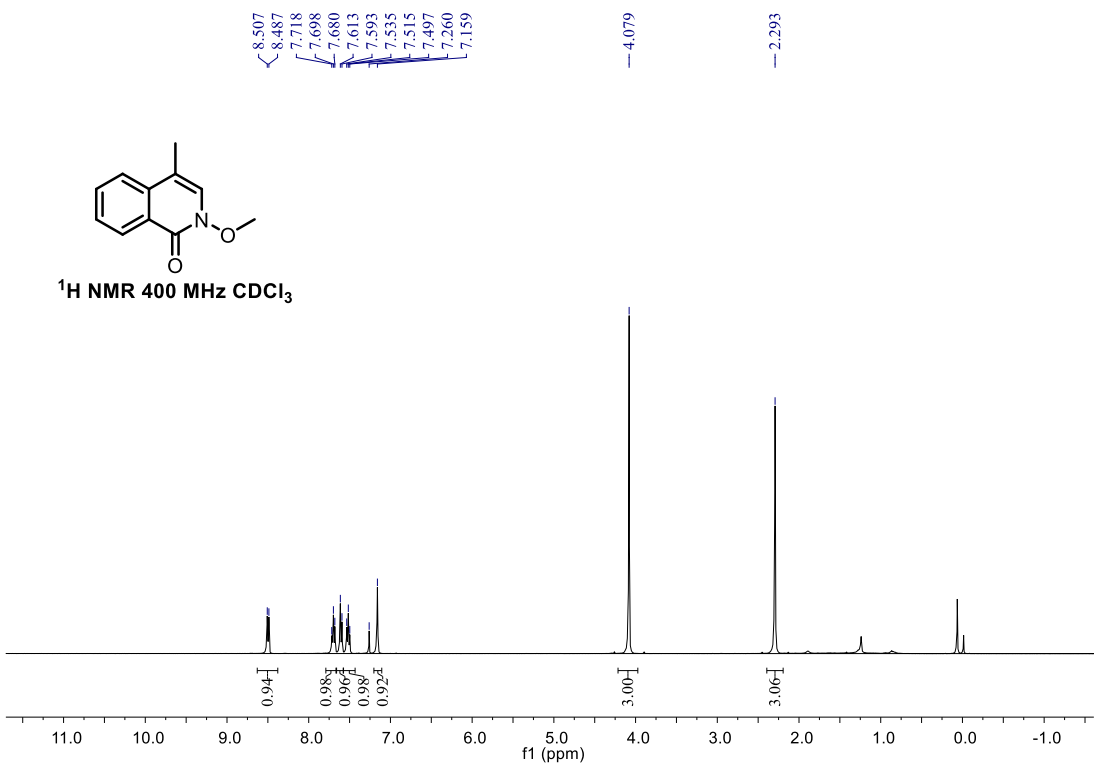
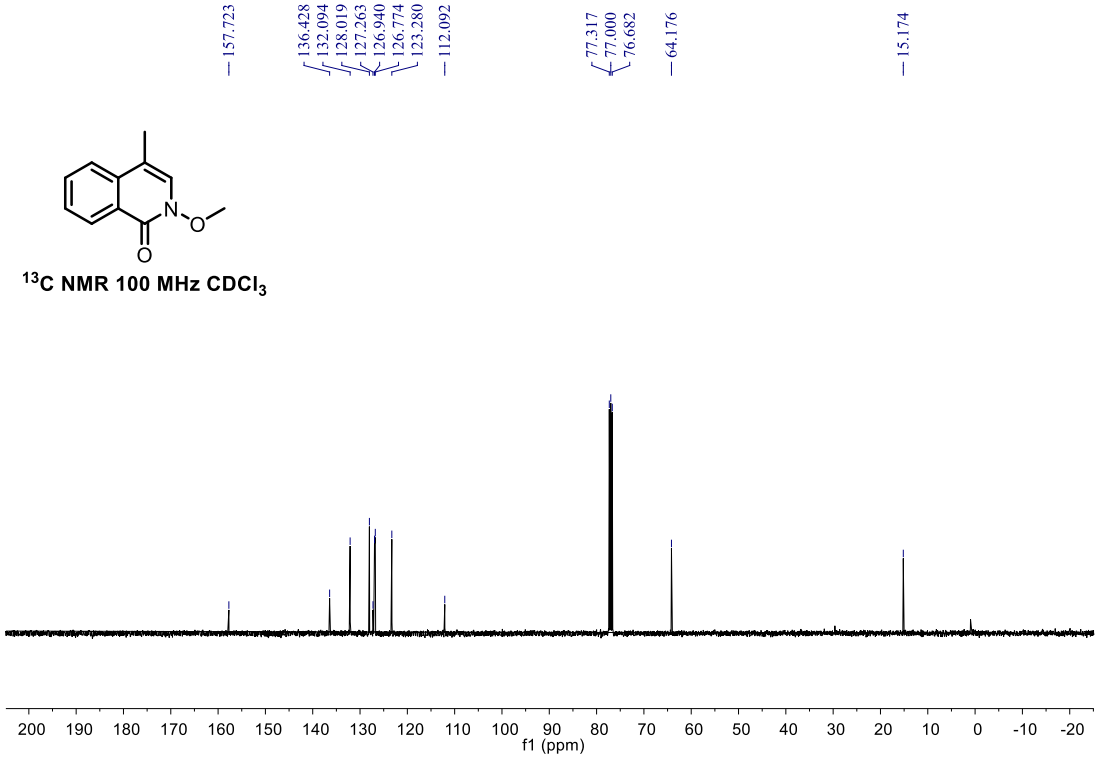
7.67
7.66
7.47
7.45
7.43
7.38
7.36
7.34
7.28
7.26
7.26
7.25
7.23
6.94
6.92
6.90
5.28
5.12
2.12
H
N
N
H
O
1H NMR 400 MHz, CDCl3
0.99
0.99
1.05
1.05
3.56
2.84
0.93
0.95
3.19
f1 (ppm)



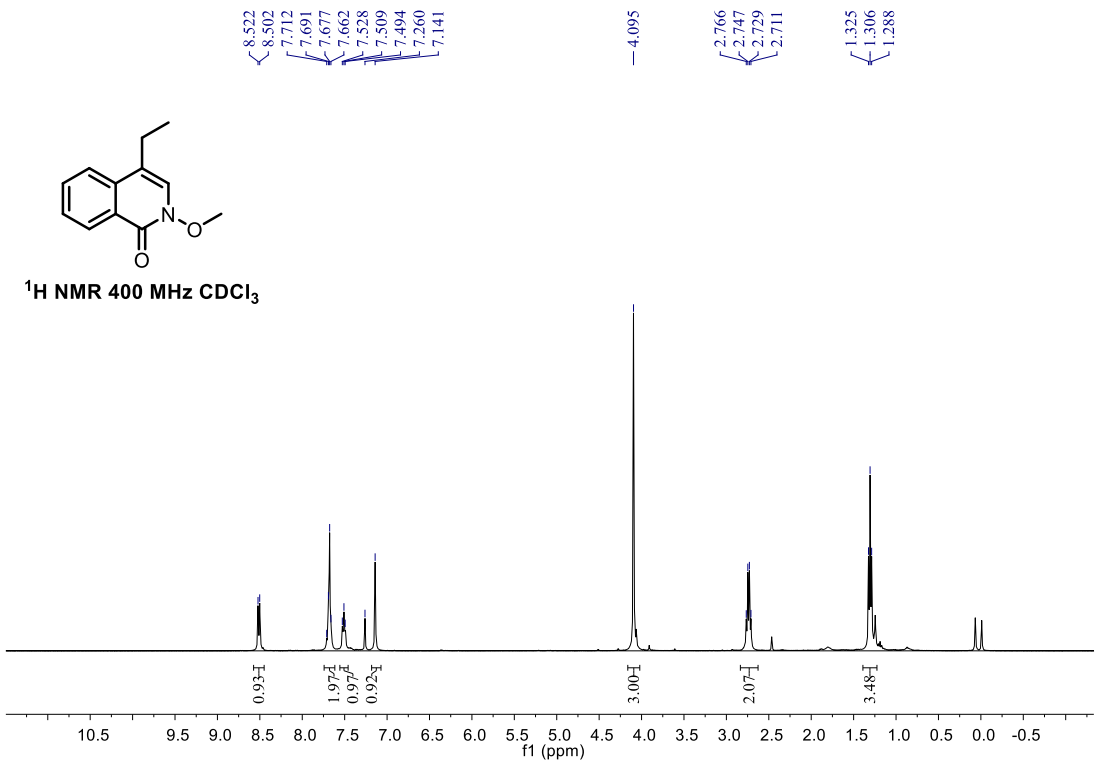
^{13}C NMR 100 MHz, $CDCl_3$



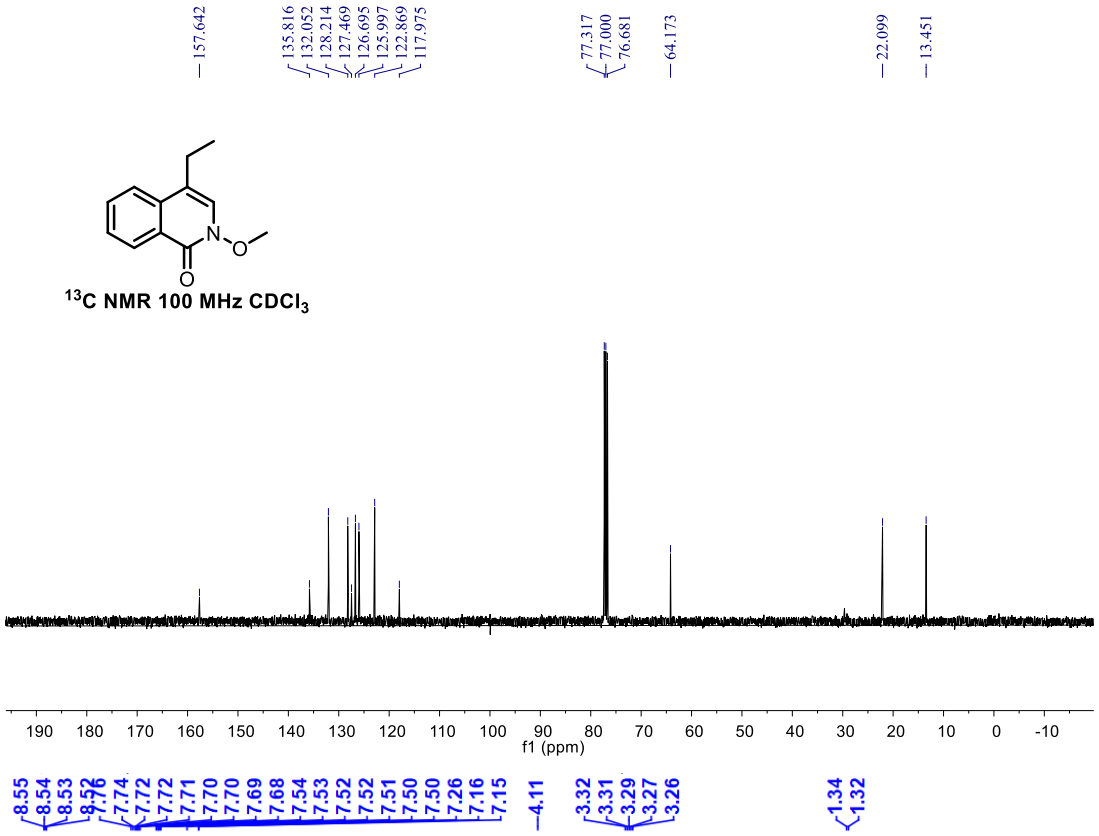
^{1}H NMR 400 MHz $CDCl_3$



^{13}C NMR 100 MHz $CDCl_3$



^{1}H NMR 400 MHz $CDCl_3$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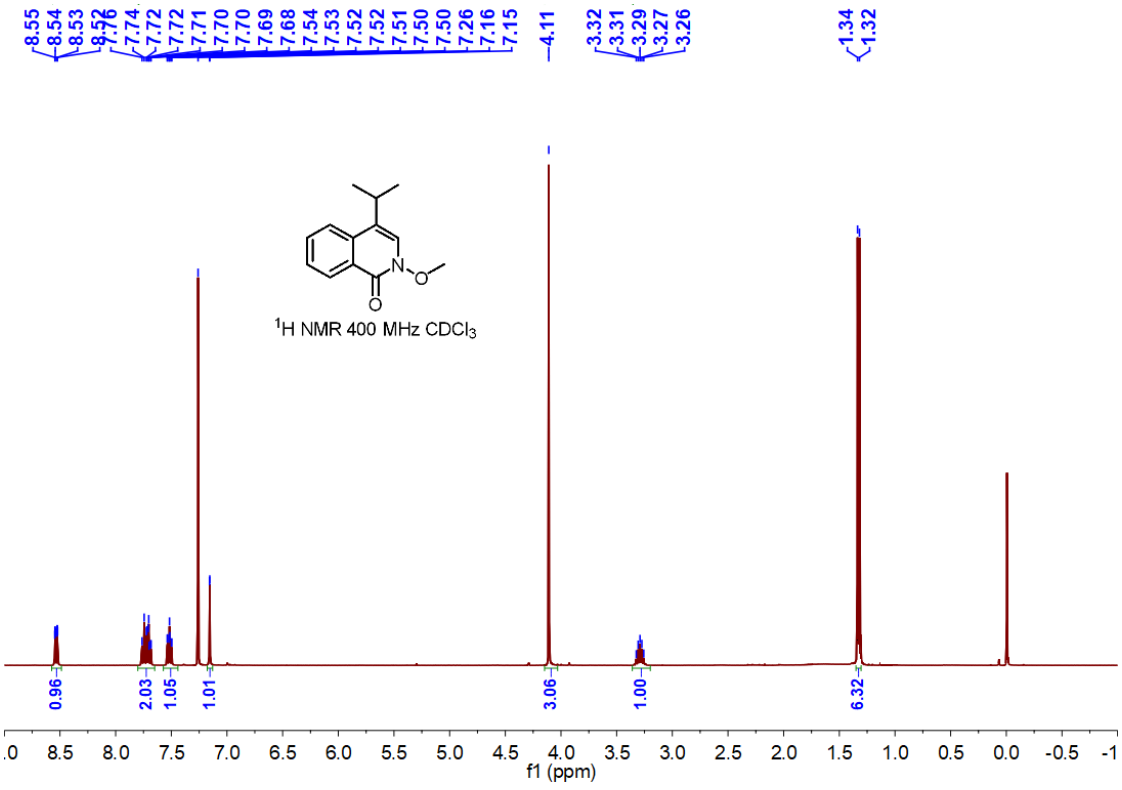
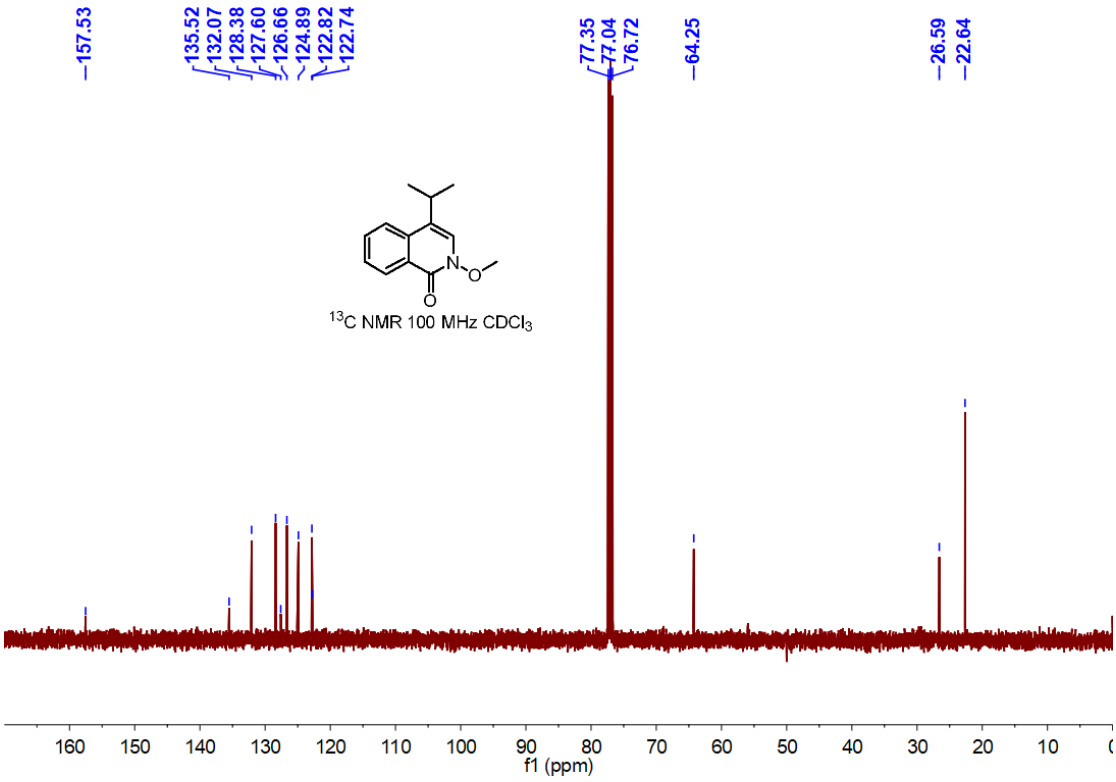
132.052
128.214
127.469
126.695
125.997
122.869
117.975
77.317
77.000
76.681
64.173
22.099
13.451
13C NMR 100 MHz CDCl3
f1 (ppm)


8.55
8.54
8.53
7.74
7.72
7.72
7.71
7.70
7.70
7.69
7.68
7.54
7.53
7.52
7.52
7.51
7.50
7.50
7.26
7.16
7.15
4.11
3.32
3.31
3.29
3.27
3.26
1.34
1.32
1H NMR 400 MHz CDCl3
0.96
2.03
1.05
1.01
3.06
1.00
6.32
f1 (ppm)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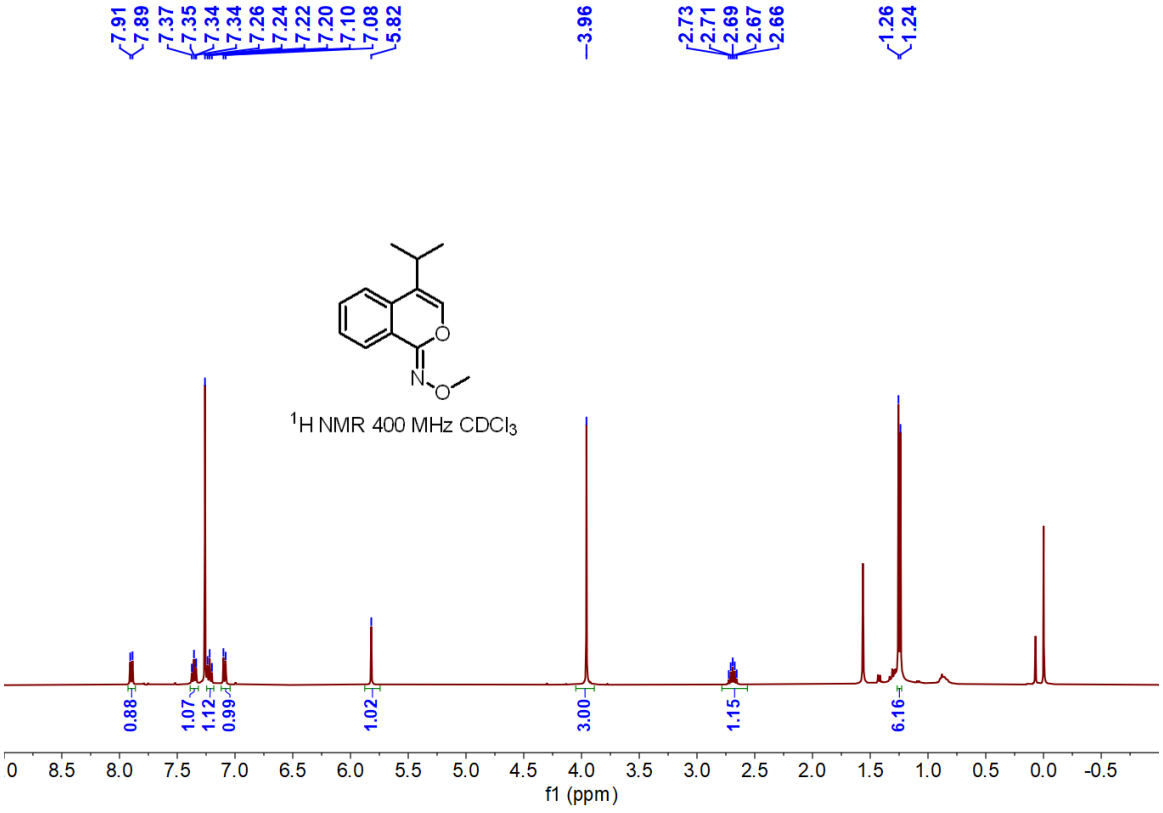
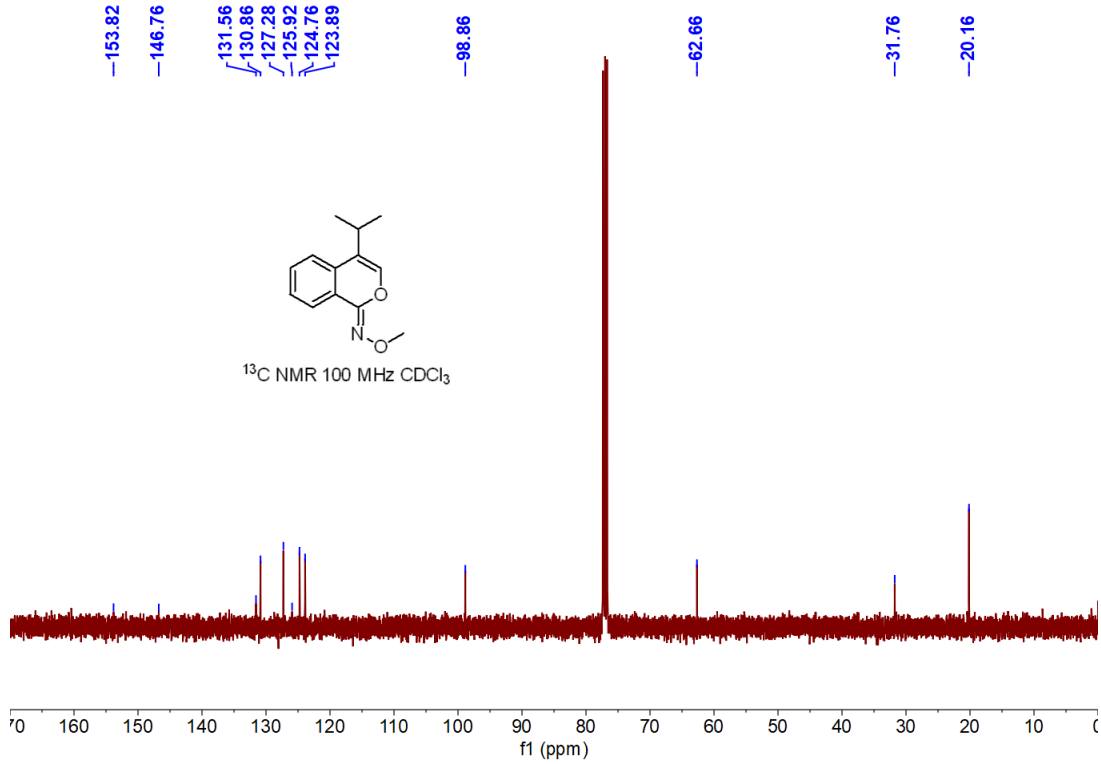
135.52
132.07
128.38
127.60
126.66
124.89
122.82
122.74
77.35
77.04
76.72
64.25
26.59
22.64
13C NMR 100 MHz CDCl3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.91
7.89
7.37
7.35
7.34
7.34
7.26
7.24
7.22
7.20
7.10
7.08
5.82
3.96
2.73
2.71
2.69
2.67
2.66
1.26
1.24
1H NMR 400 MHz CDCl3
0.88
1.07
1.12
0.99
1.02
3.00
1.15
6.16
0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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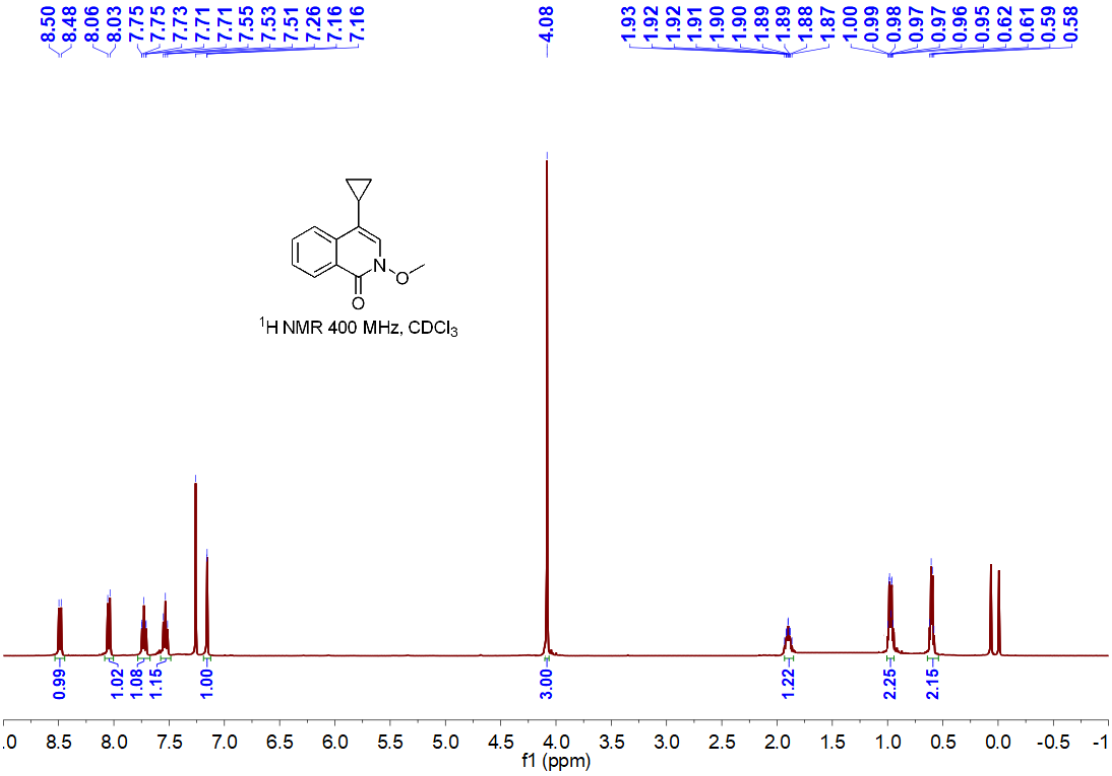
131.56
130.86
127.28
125.92
124.76
123.89
98.86
62.66
31.76
20.16
13C NMR 100 MHz CDCl3
f1 (ppm)


8.50
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8.03
7.75
7.75
7.73
7.71
7.71
7.55
7.53
7.51
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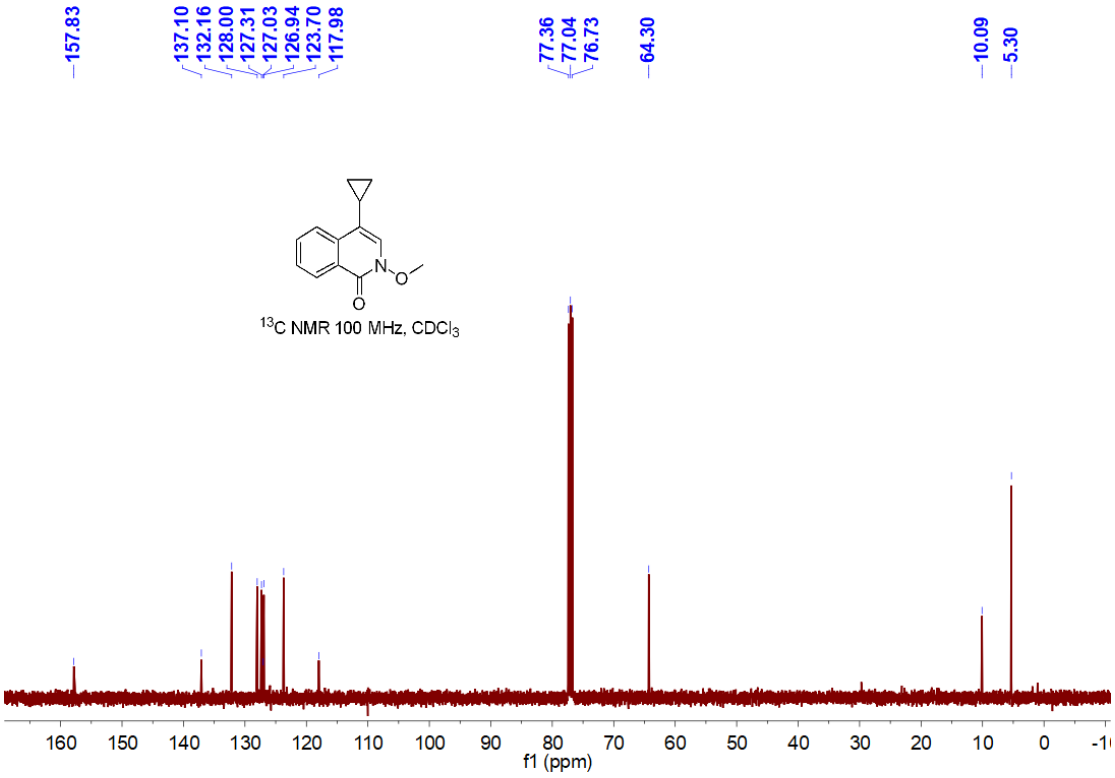
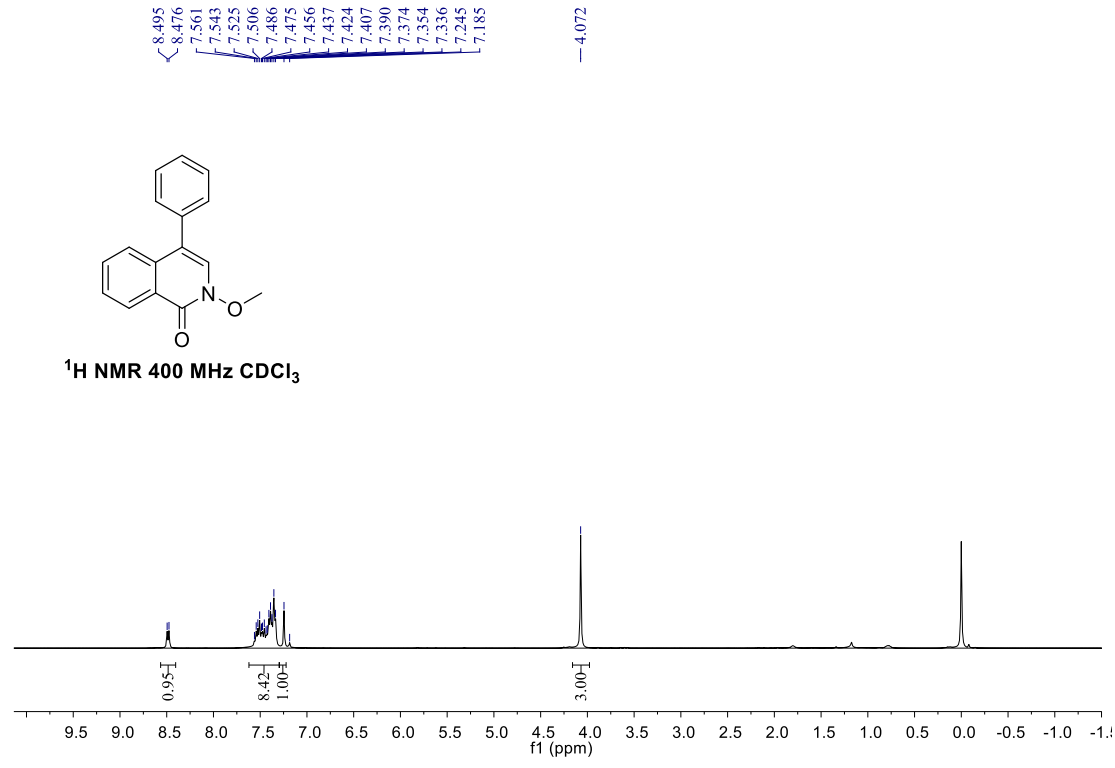
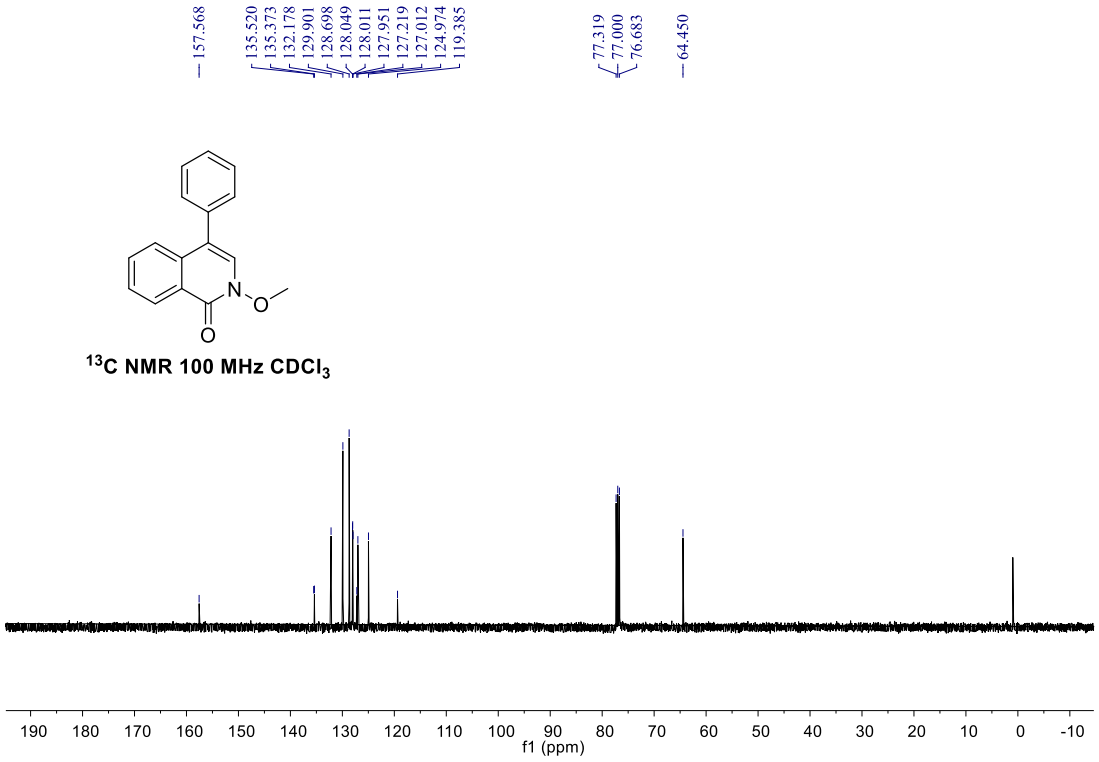
7.16
7.16
4.08
1.93
1.92
1.92
1.91
1.90
1.90
1.89
1.89
1.88
1.87
1.00
0.99
0.98
0.97
0.97
0.96
0.95
0.62
0.61
0.59
0.58
1H NMR 400 MHz, CDCl3
0.99
1.02
1.08
1.15
1.00
3.00
1.22
2.25
2.15
f1 (ppm)

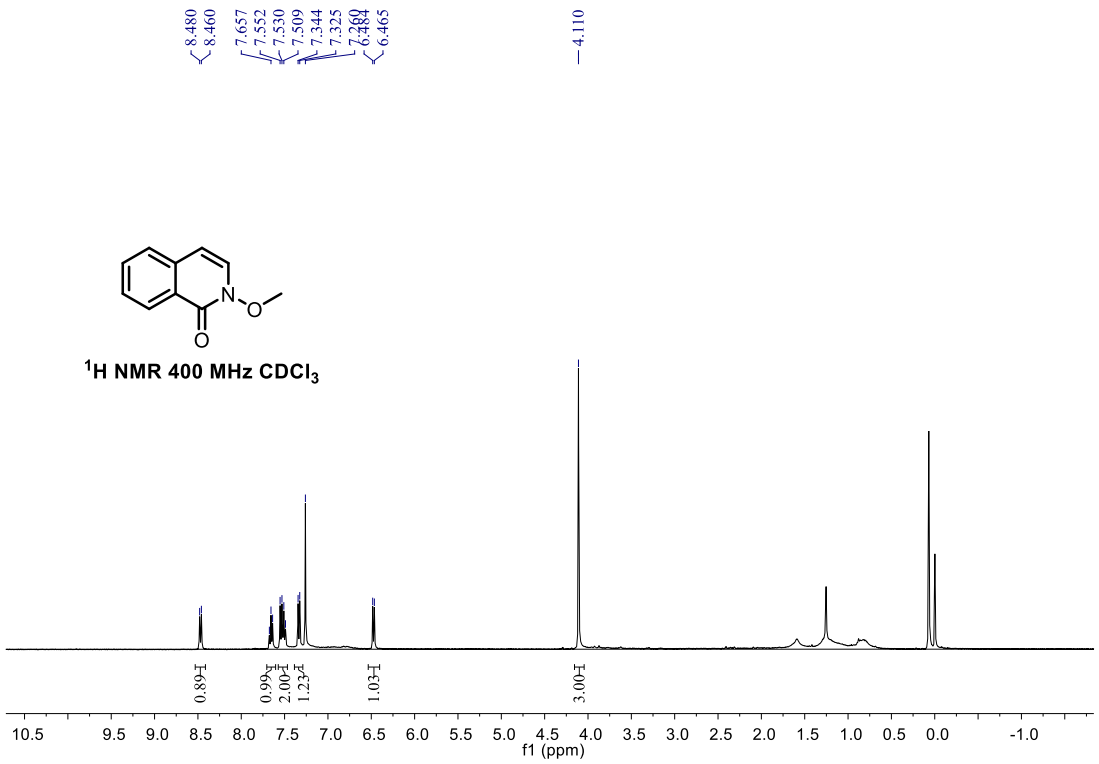




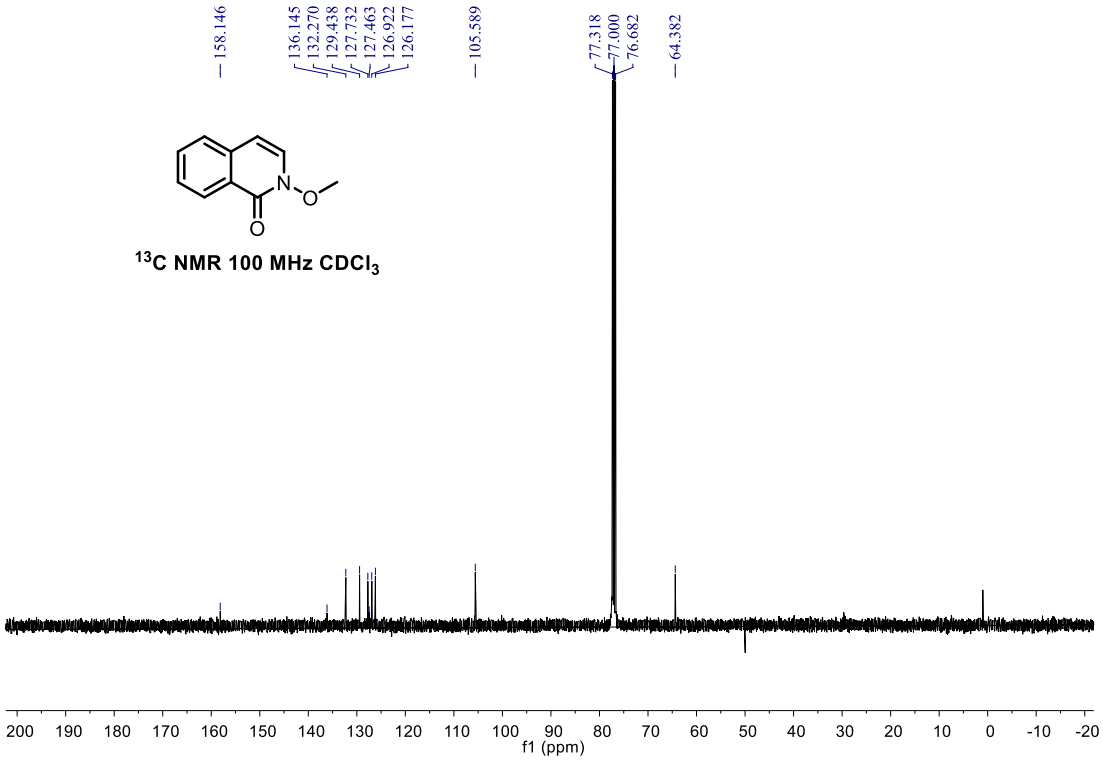
^{1}H NMR 400 MHz $CDCl_3$



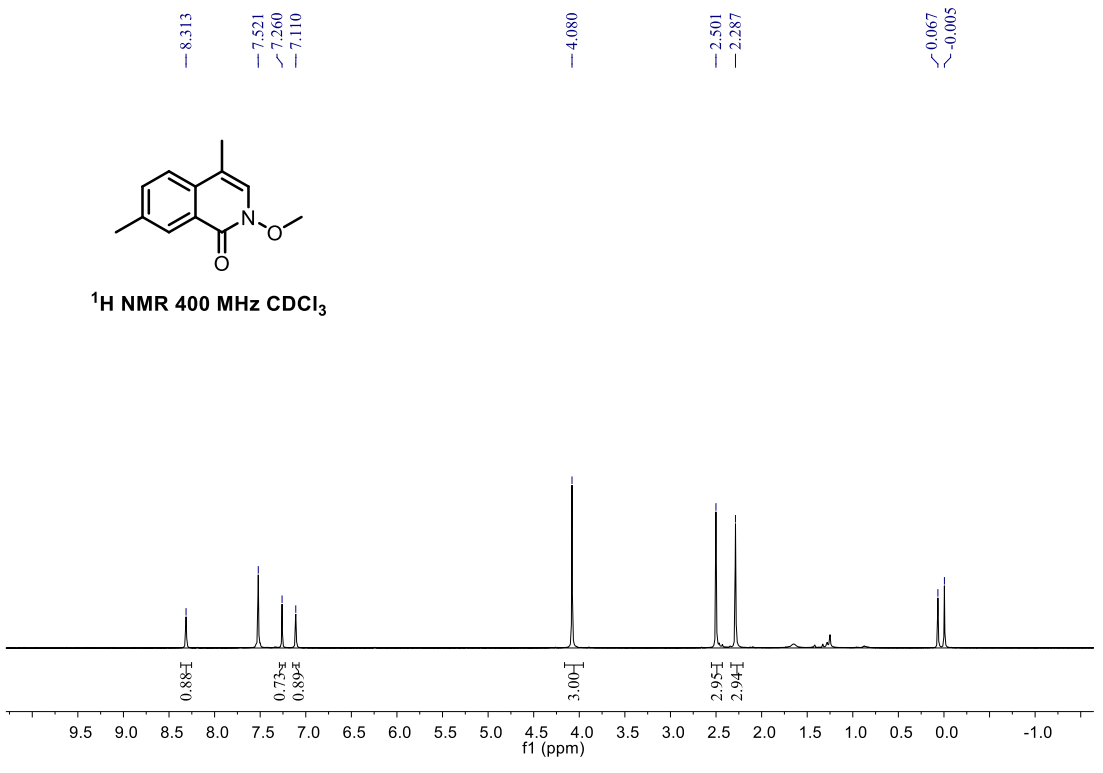
^{13}C NMR 100 MHz $CDCl_3$



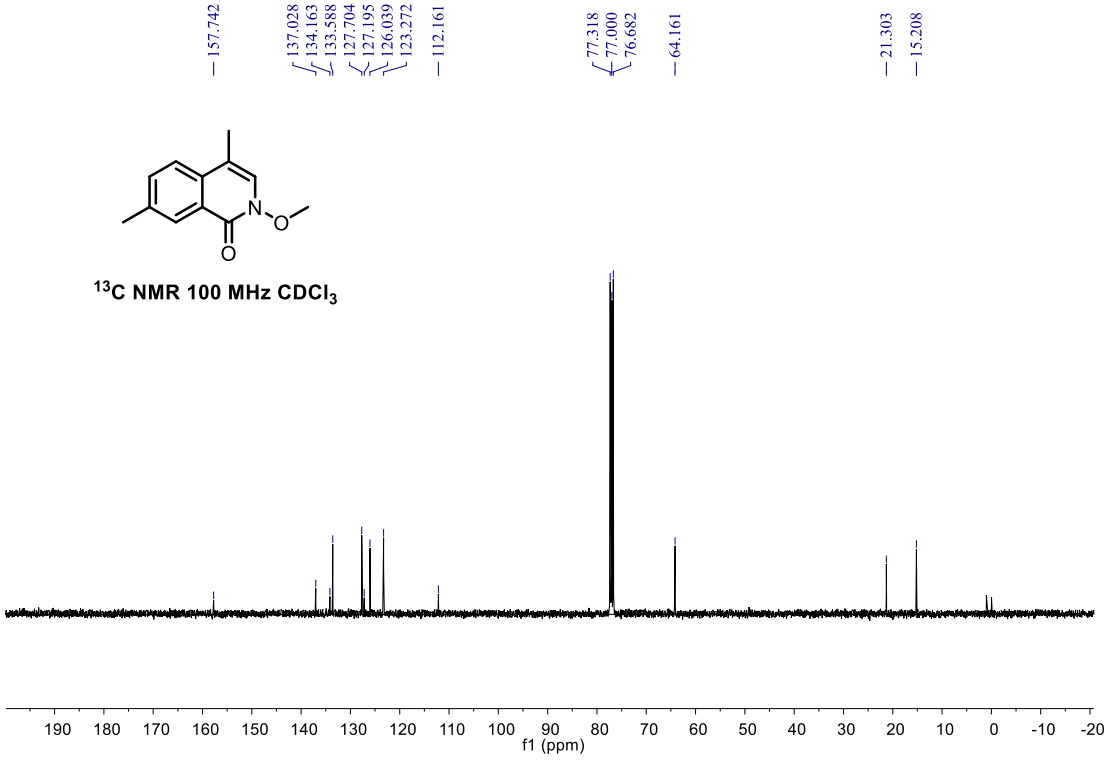
^{1}H NMR 400 MHz $CDCl_3$



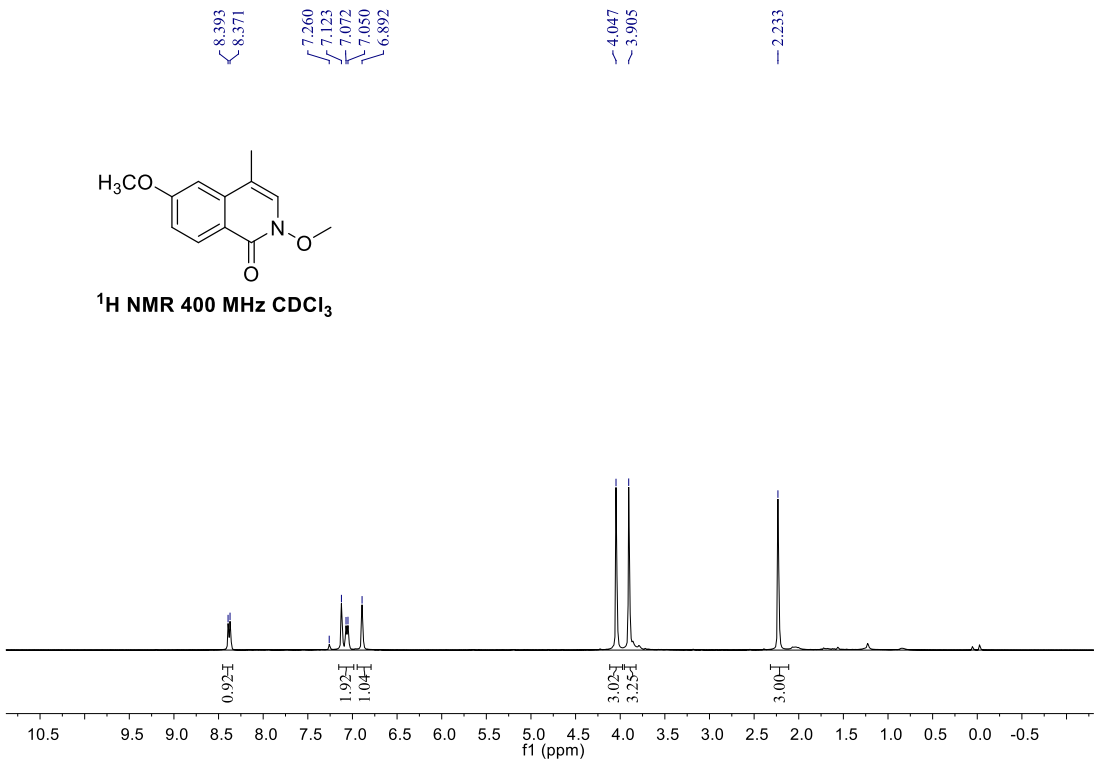
^{13}C NMR 100 MHz $CDCl_3$



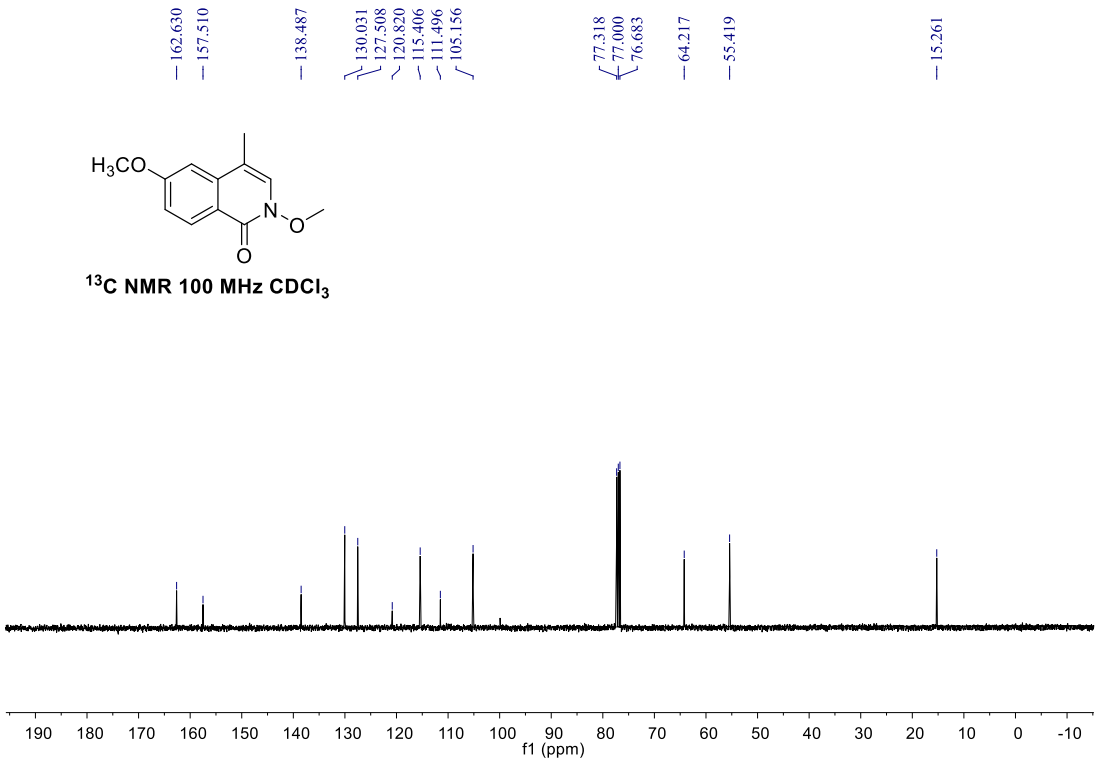
^{1}H NMR 400 MHz $CDCl_3$



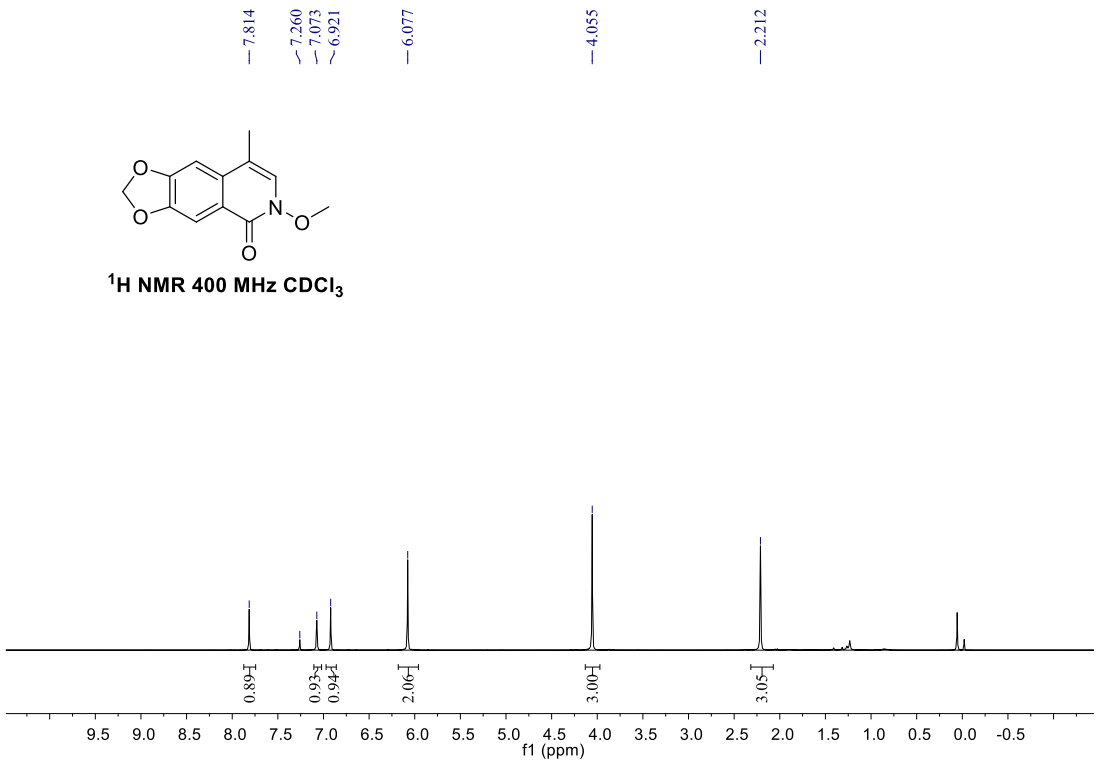
^{13}C NMR 100 MHz $CDCl_3$



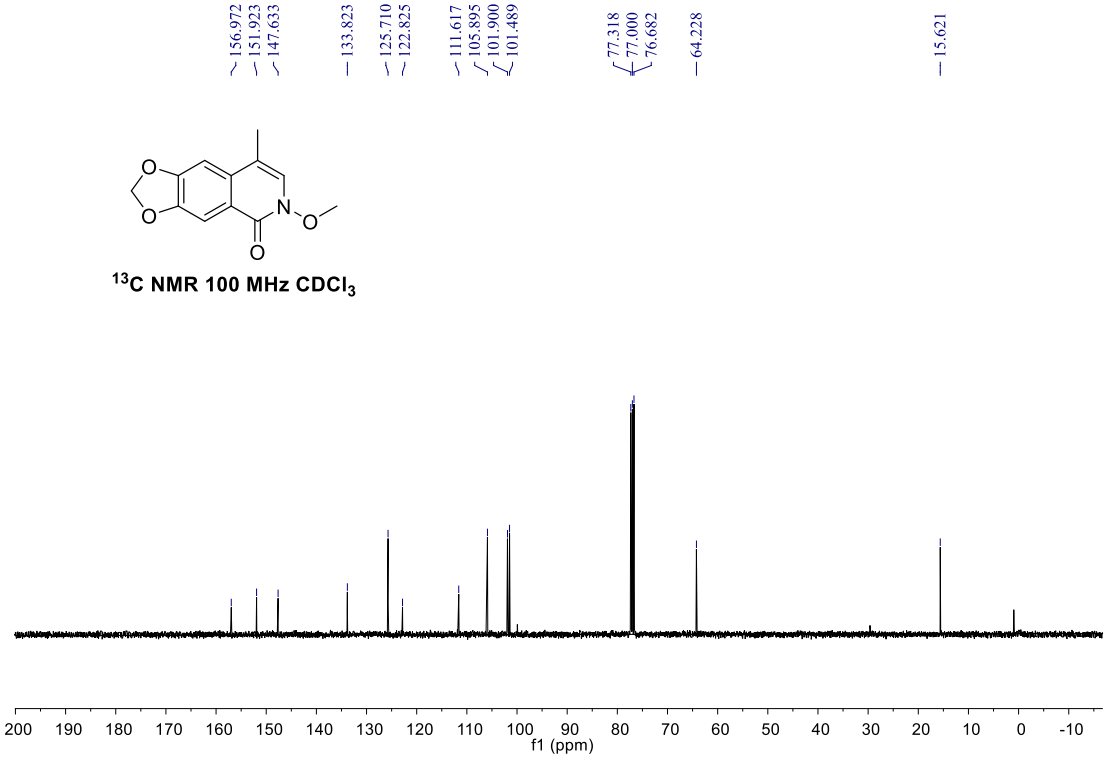
^{1}H NMR 400 MHz $CDCl_3$



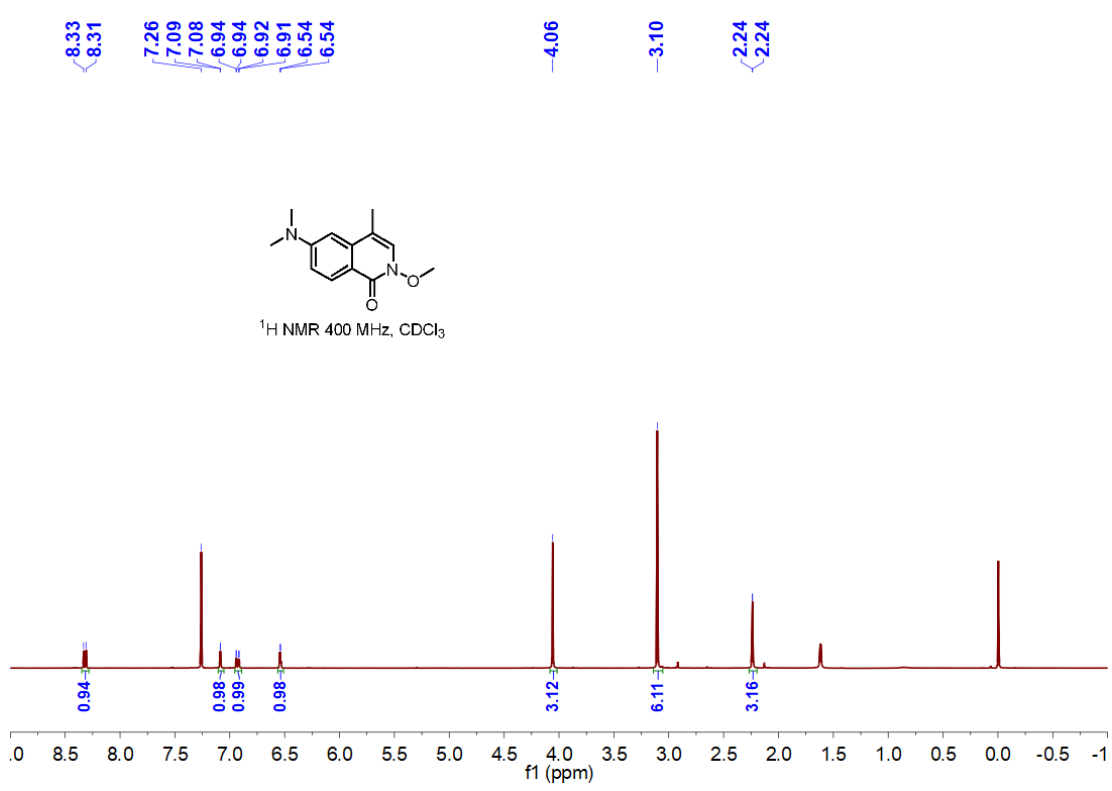
^{13}C NMR 100 MHz $CDCl_3$



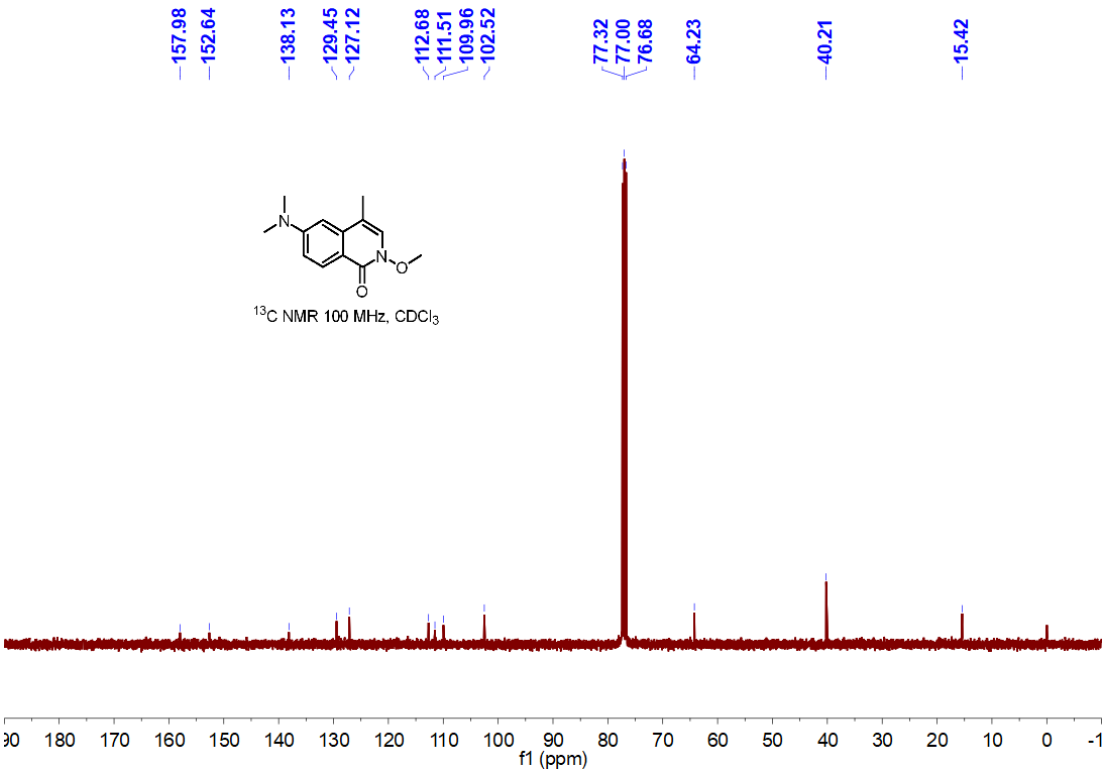
^{1}H NMR 400 MHz $CDCl_3$

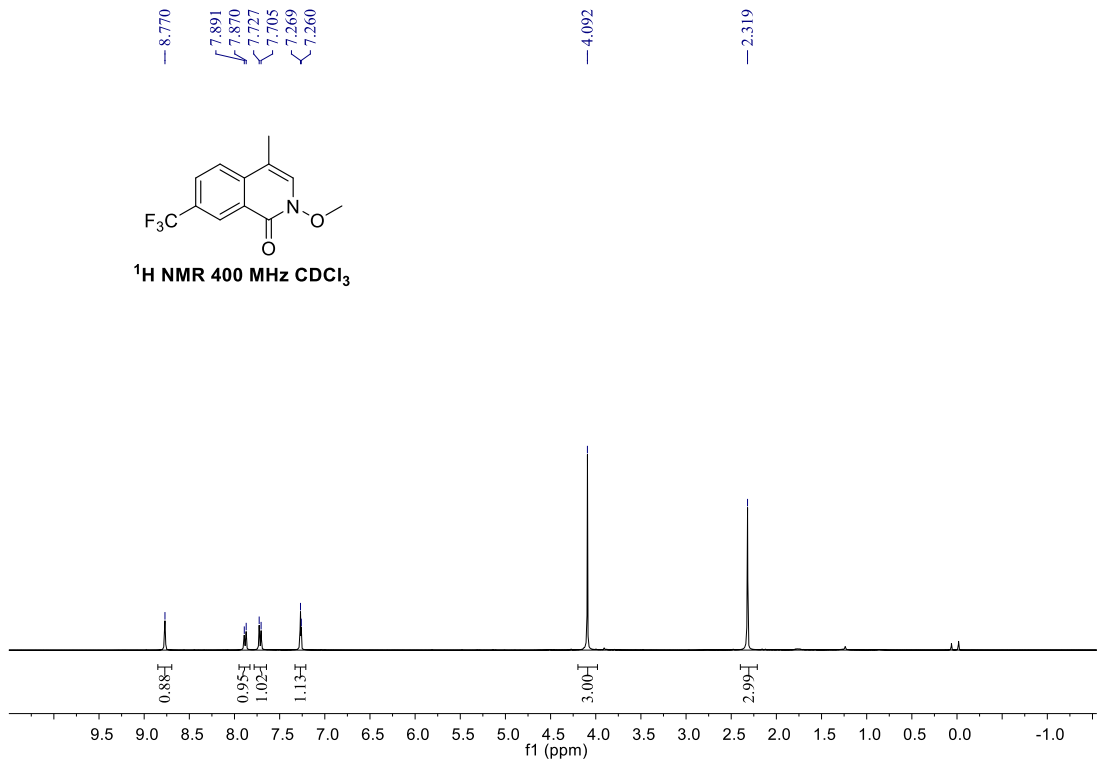


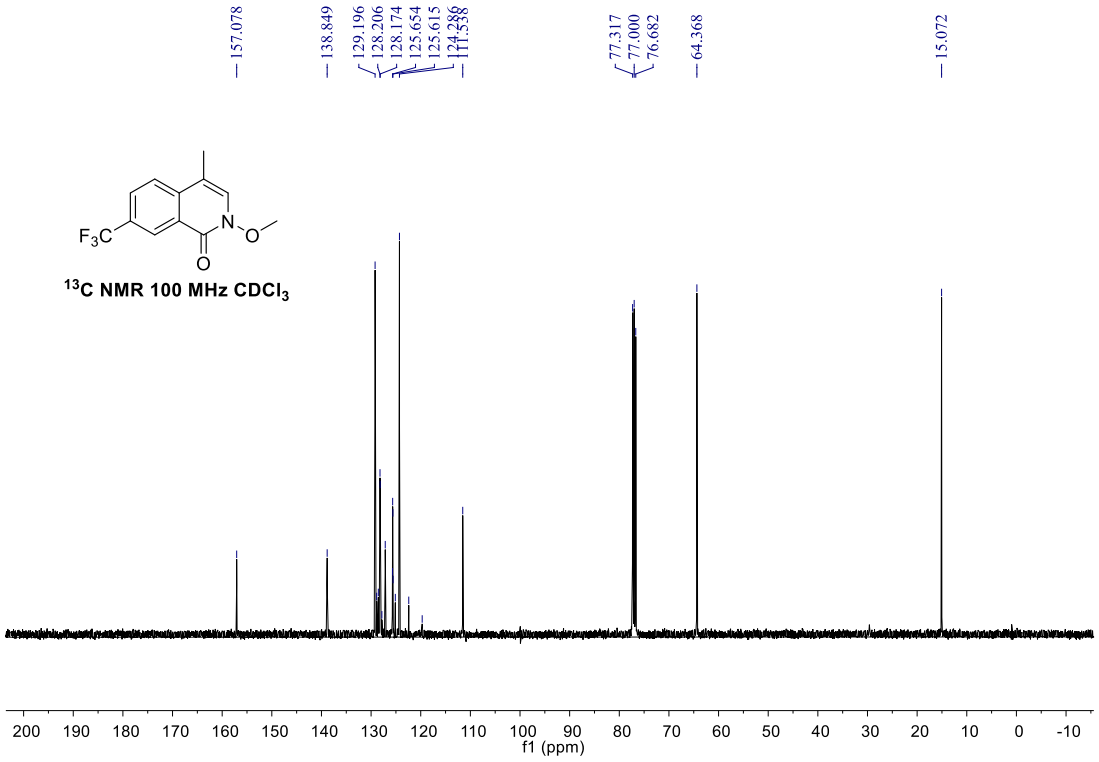
^{13}C NMR 100 MHz $CDCl_3$



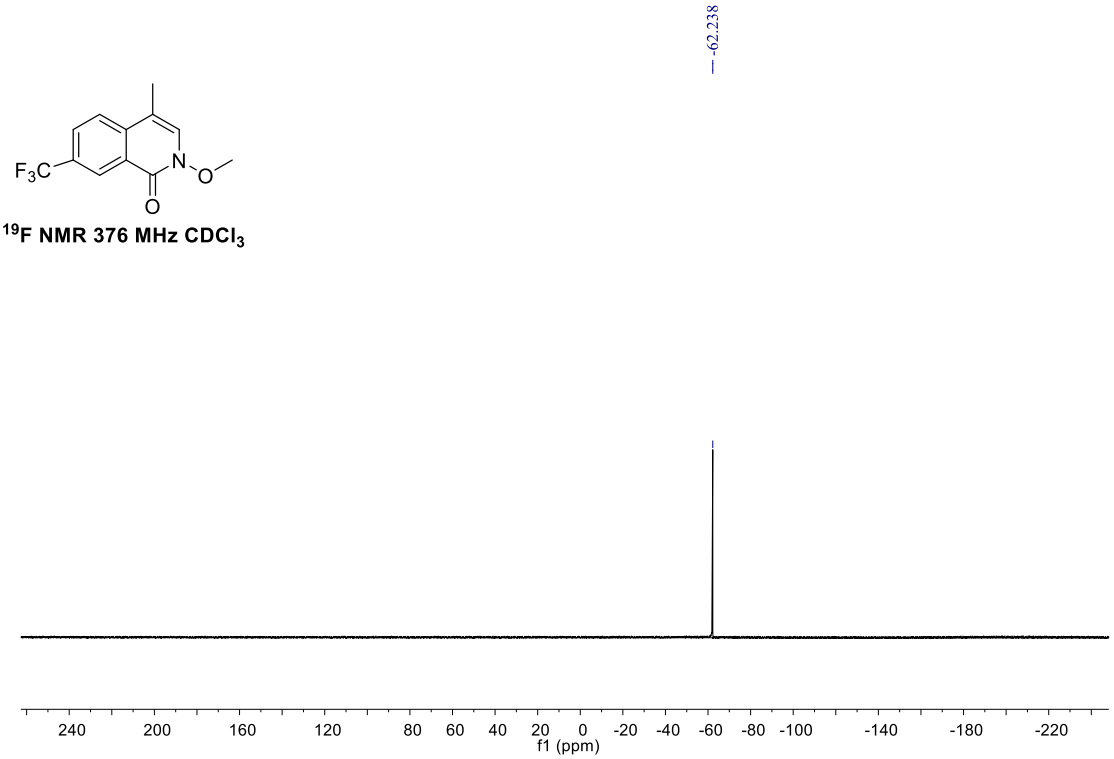
^{1}H NMR 400 MHz, $CDCl_3$


157.98
152.64
138.13
129.45
127.12
112.68
111.51
109.96
102.52
77.32
77.00
76.68
64.23
40.21
15.42
13C NMR 100 MHz, CDCl3
f1 (ppm)

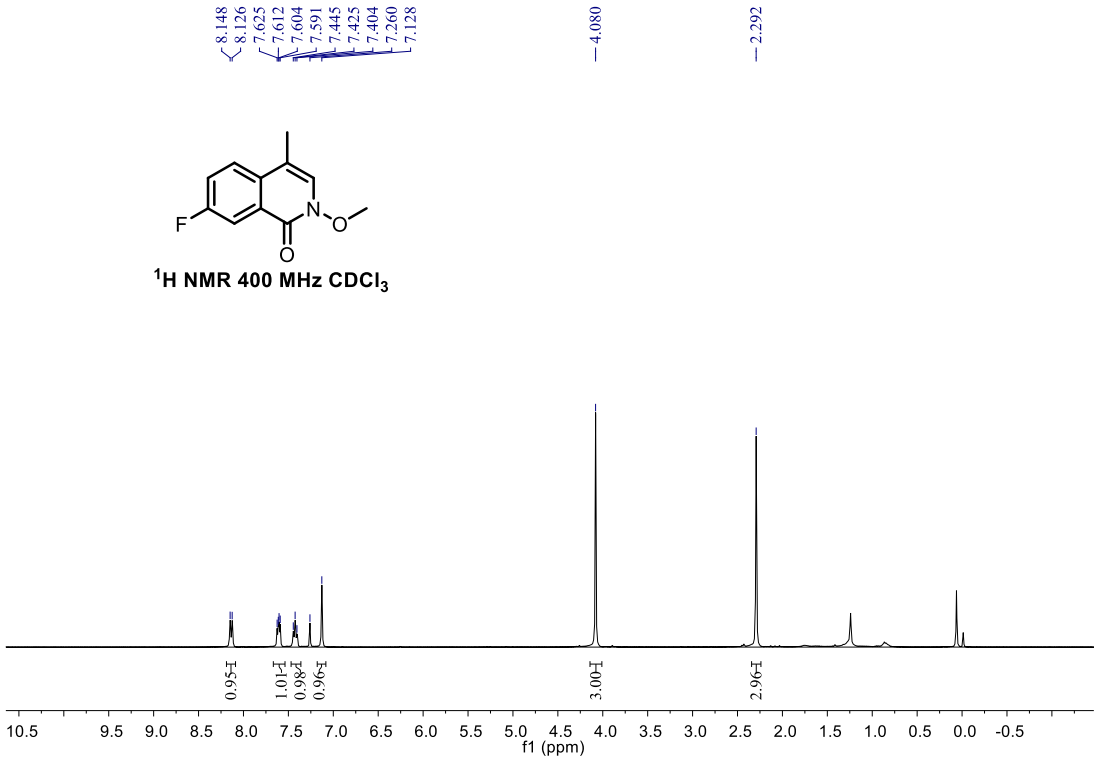

8.770
7.891
7.870
7.727
7.705
7.269
7.260
4.092
2.319
1H NMR 400 MHz CDCl3
0.88
0.95
1.02
1.13
3.00
2.99
f1 (ppm)



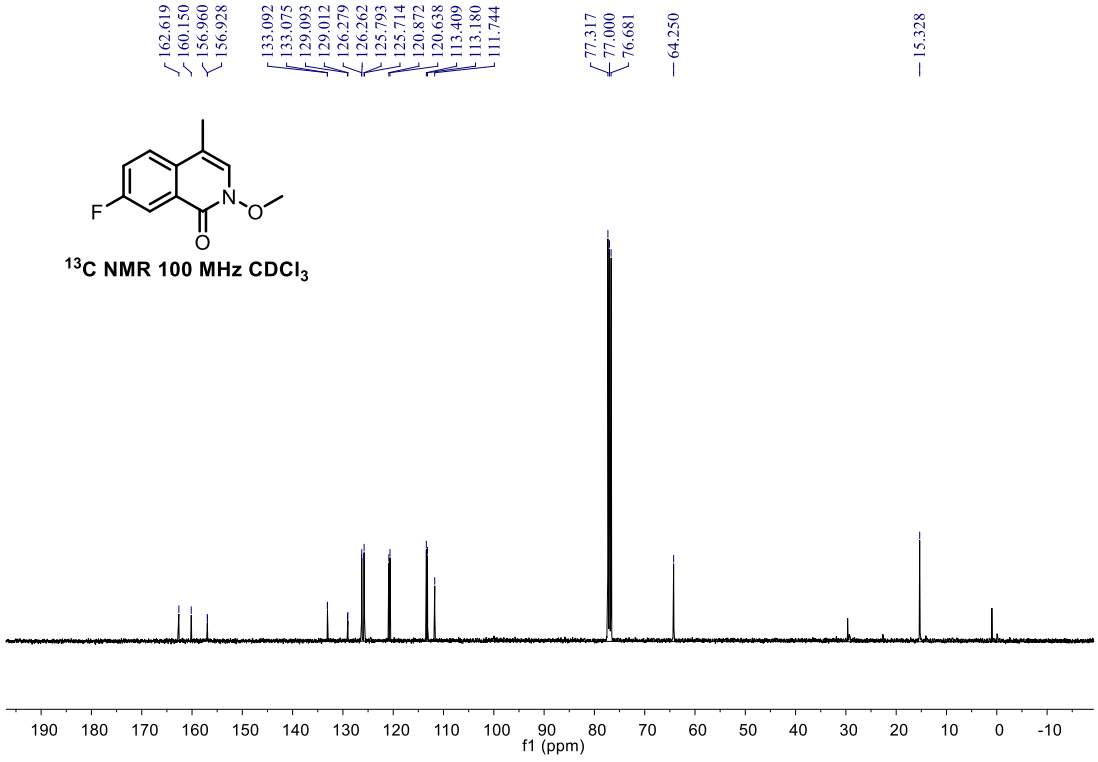
^{13}C NMR 100 MHz $CDCl_3$



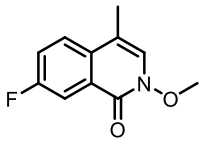
^{19}F NMR 376 MHz $CDCl_3$



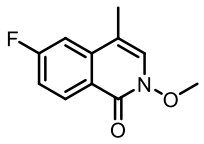
^{1}H NMR 400 MHz $CDCl_3$



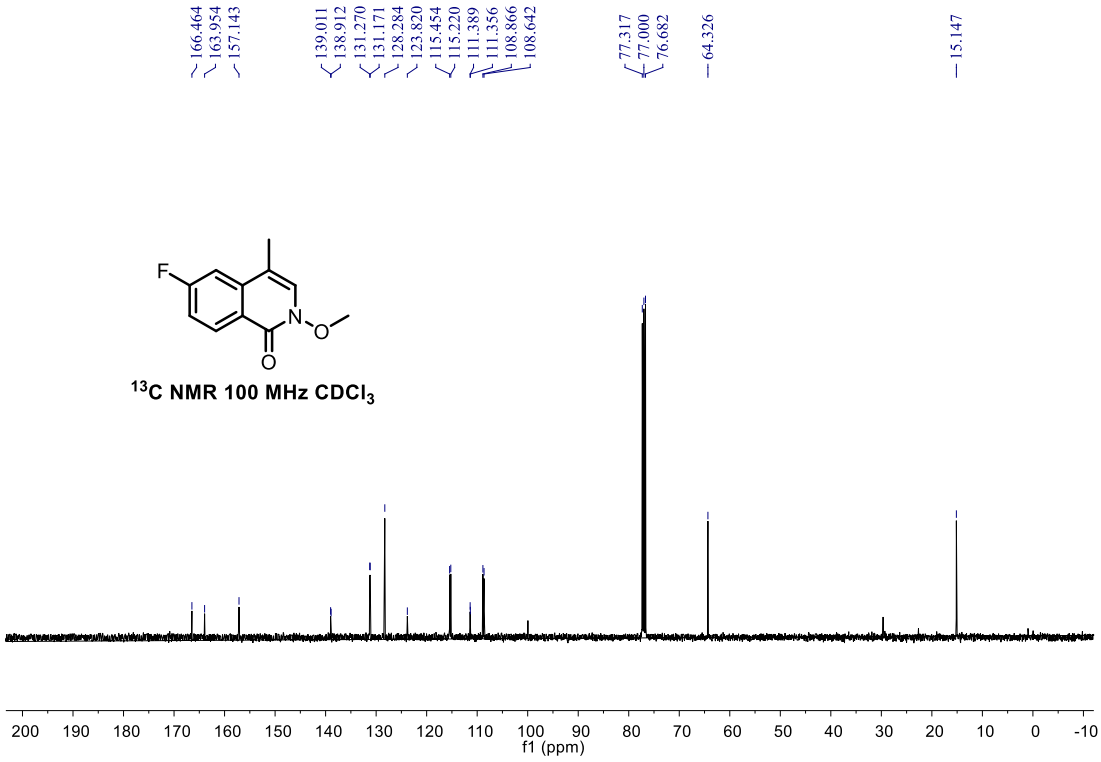
^{13}C NMR 100 MHz $CDCl_3$



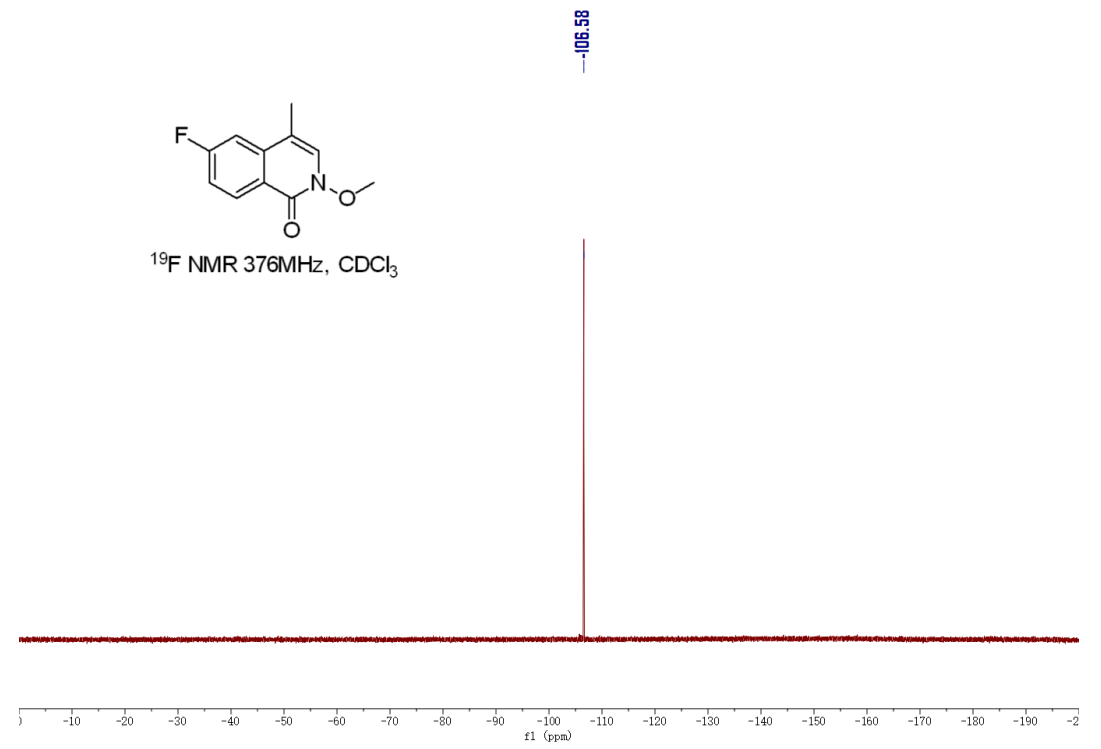
^{19}F NMR 376 MHz $CDCl_3$



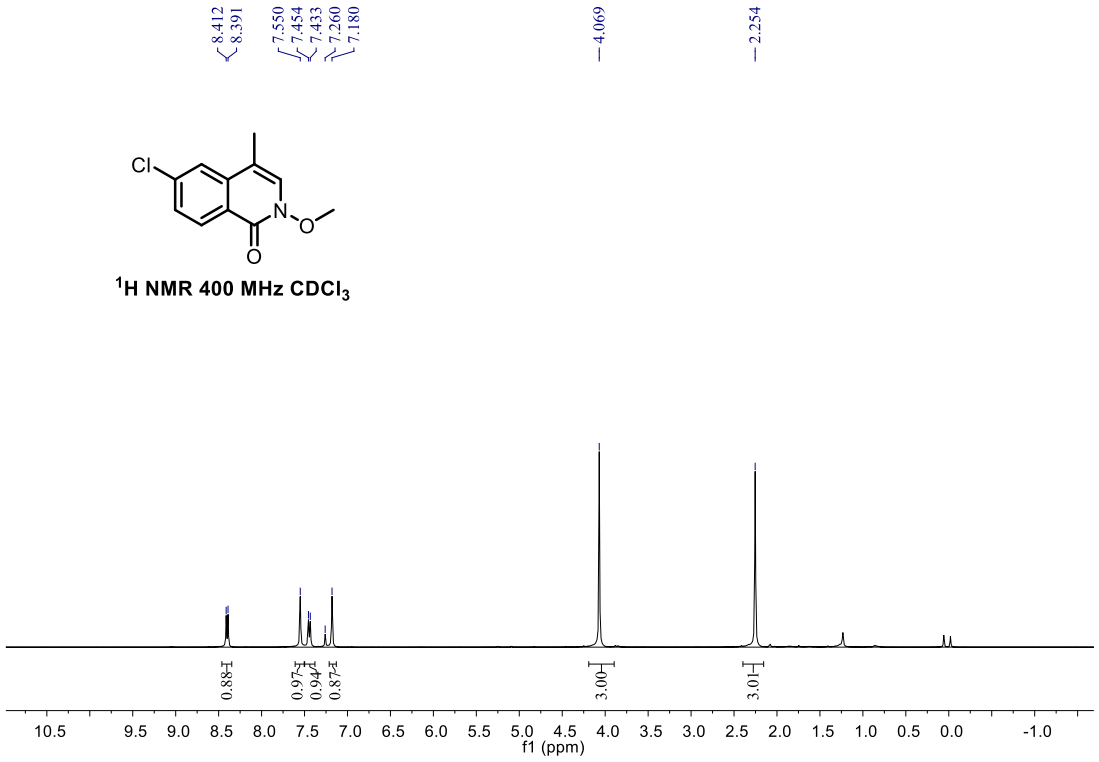
^{1}H NMR 400 MHz $CDCl_3$



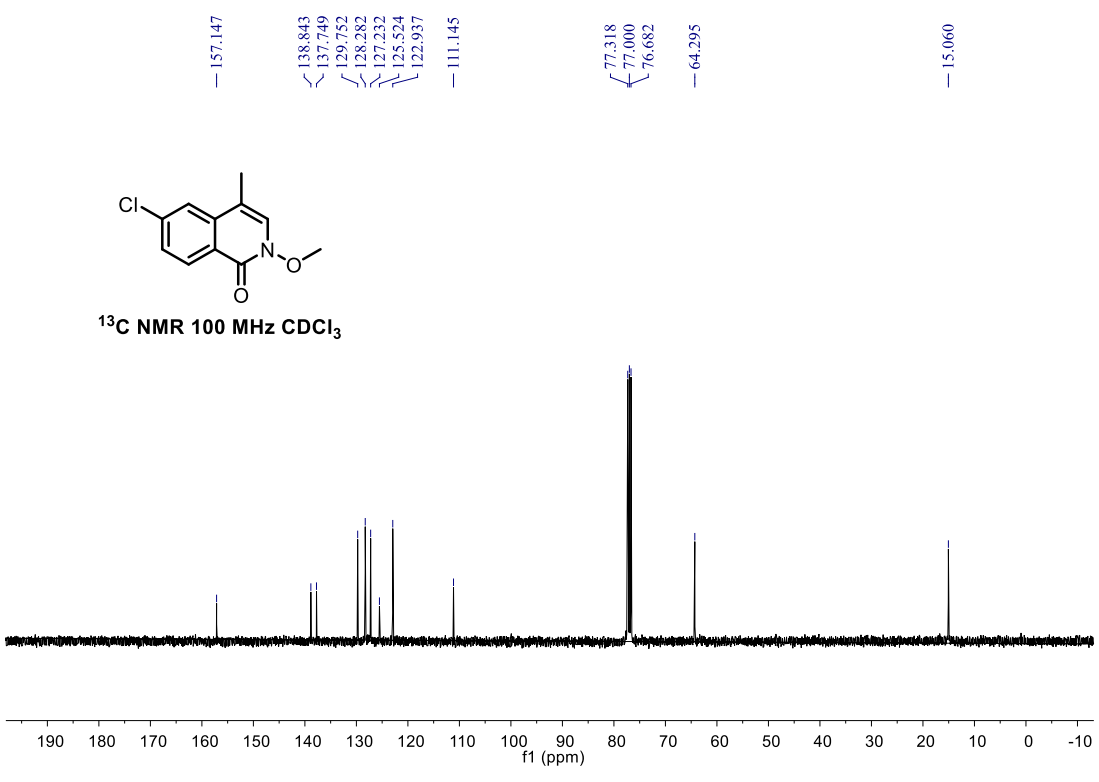
^{13}C NMR 100 MHz $CDCl_3$



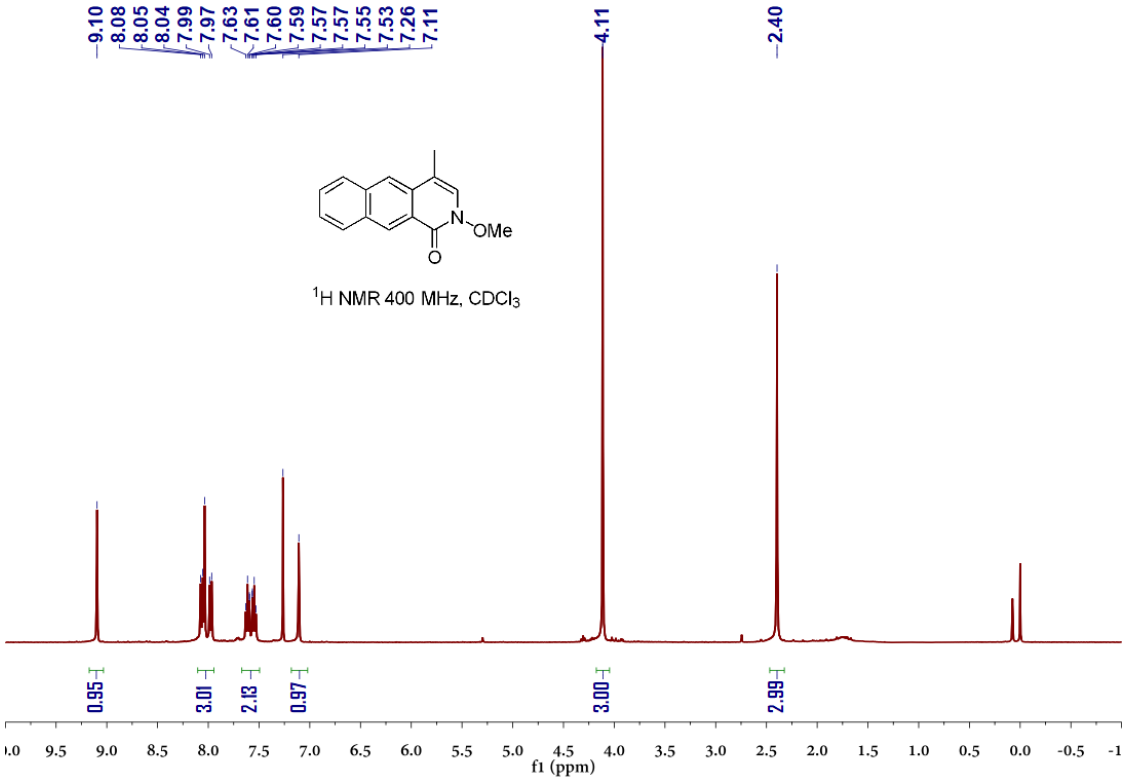
^{19}F NMR 376MHz, $CDCl_3$



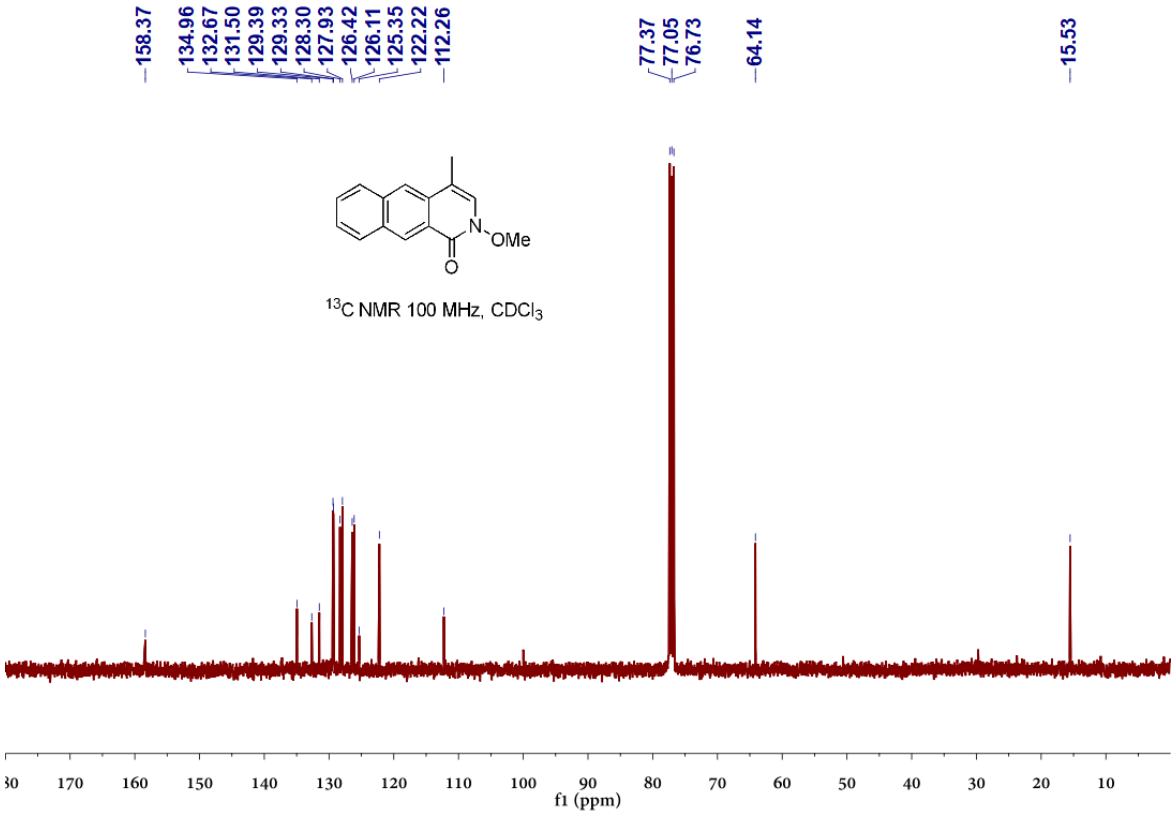
^{1}H NMR 400 MHz $CDCl_3$



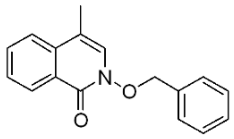
^{13}C NMR 100 MHz $CDCl_3$



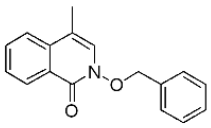
^{1}H NMR 400 MHz, $CDCl_3$



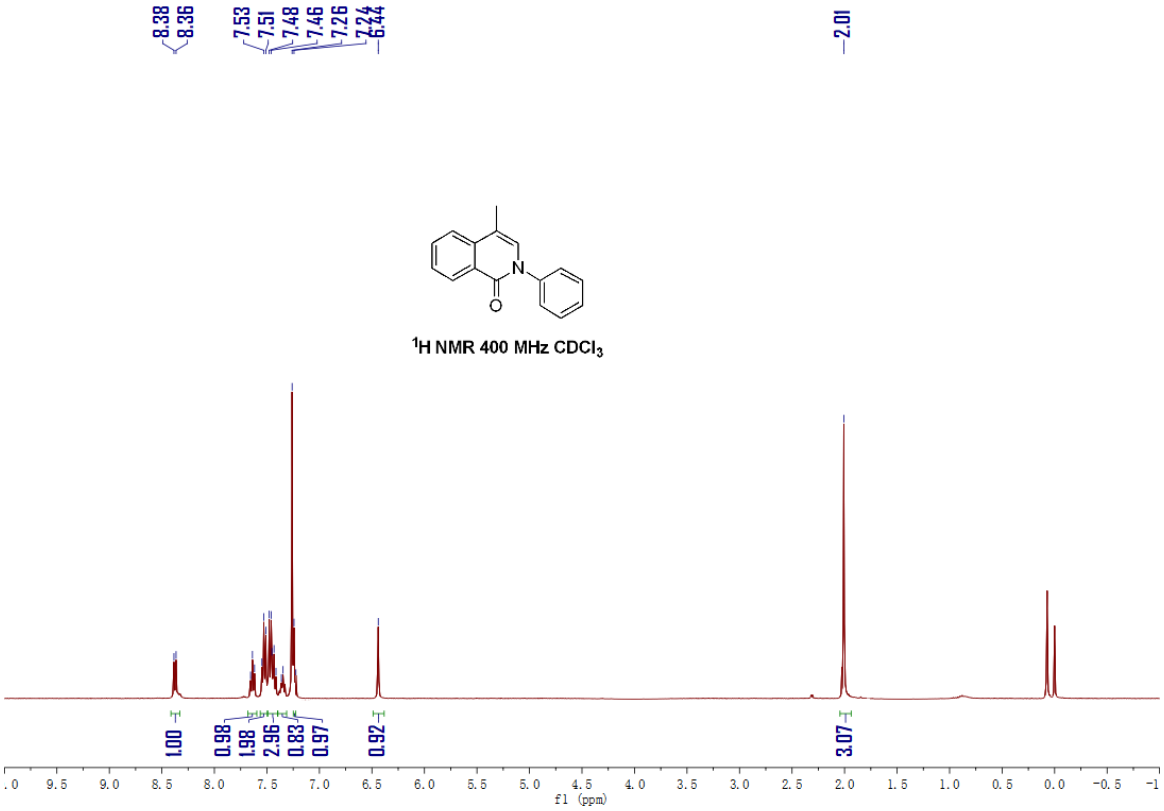
^{13}C NMR 100 MHz, $CDCl_3$



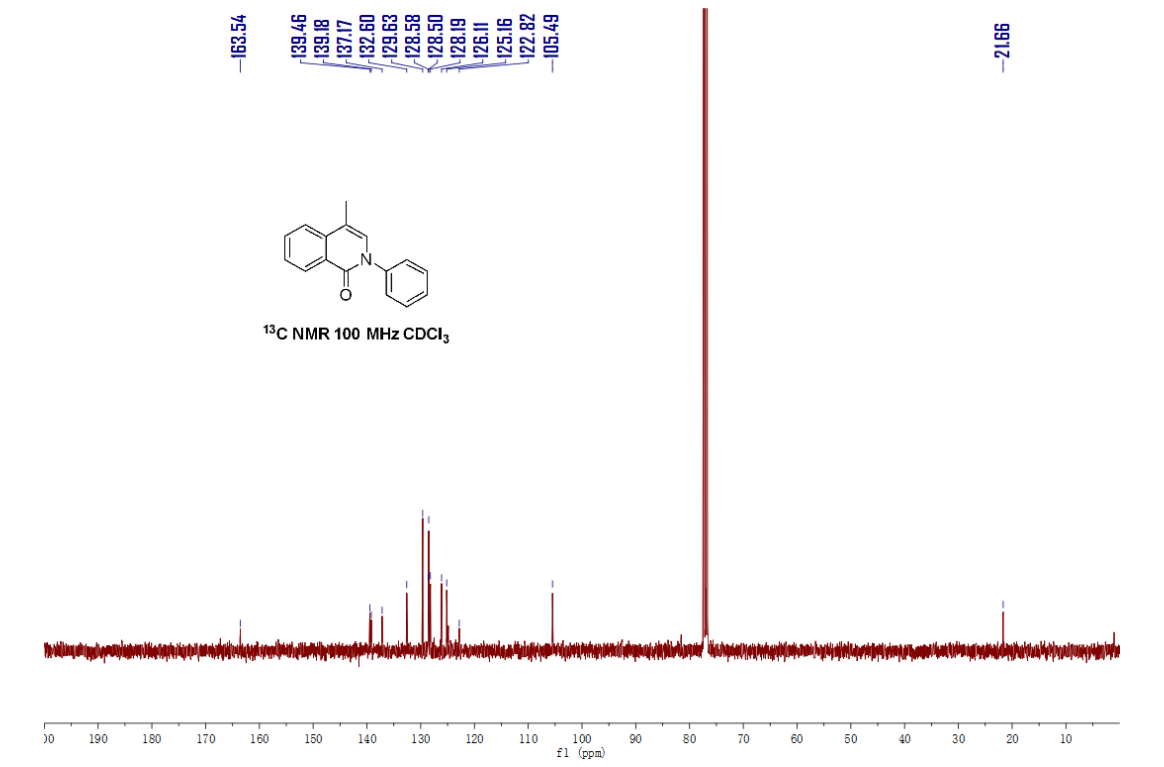
^{1}H NMR 400 MHz $CDCl_3$



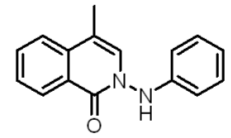
^{13}C NMR 100 MHz $CDCl_3$



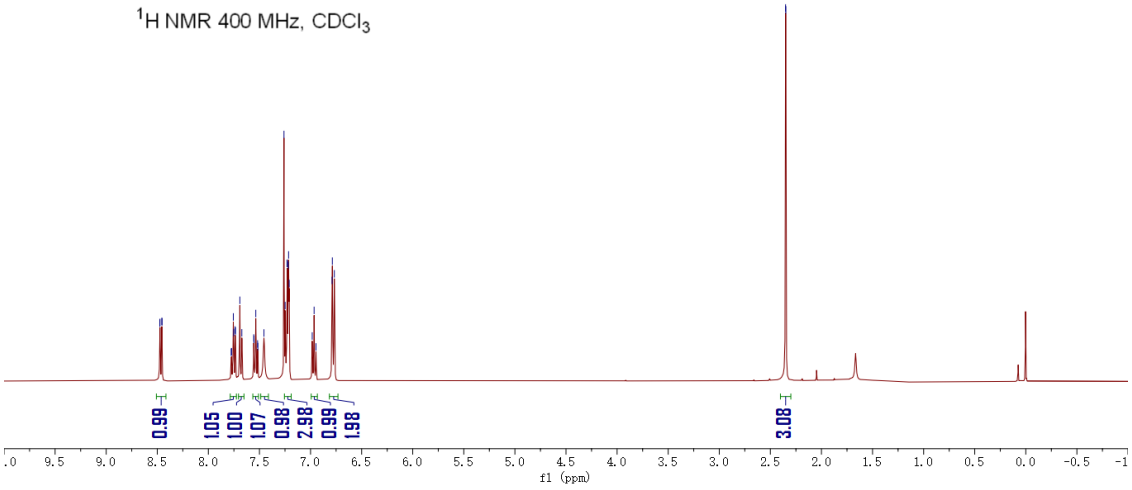
^{1}H NMR 400 MHz $CDCl_3$

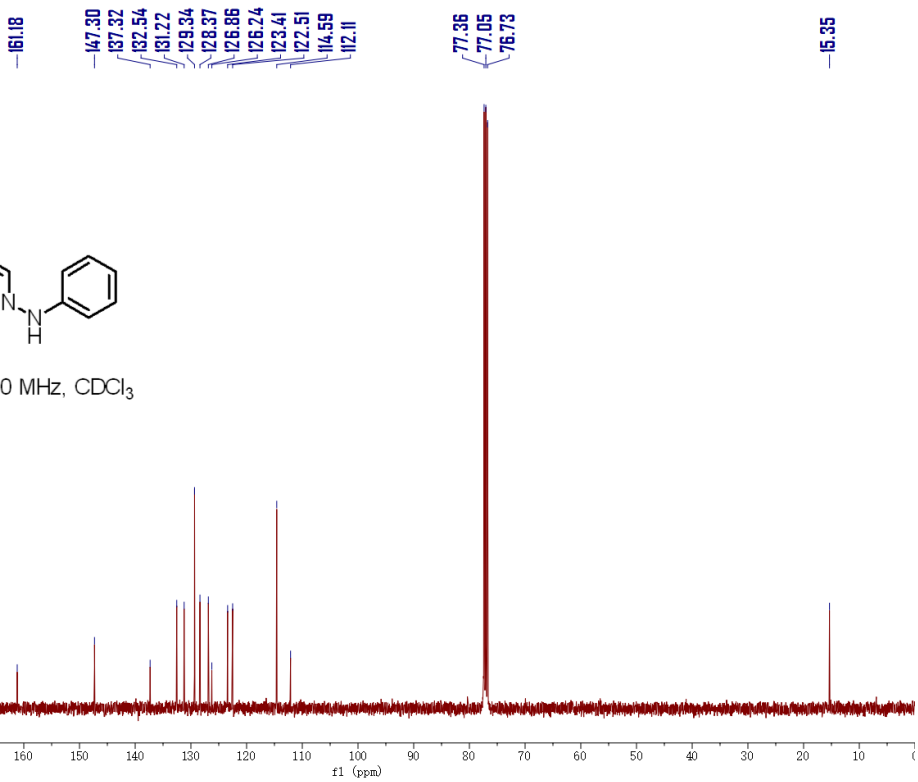


^{13}C NMR 100 MHz $CDCl_3$

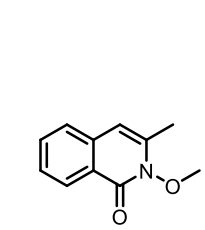


^{1}H NMR 400 MHz, $CDCl_3$

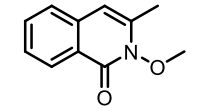




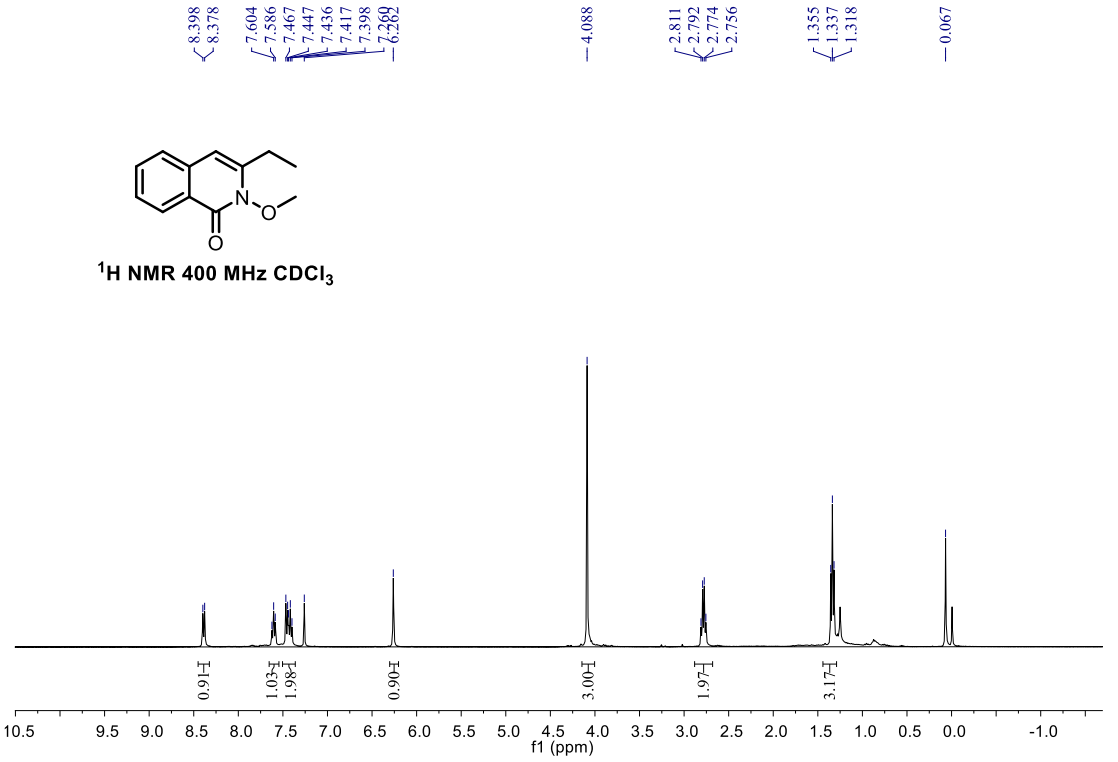
^{13}C NMR 100 MHz, $CDCl_3$



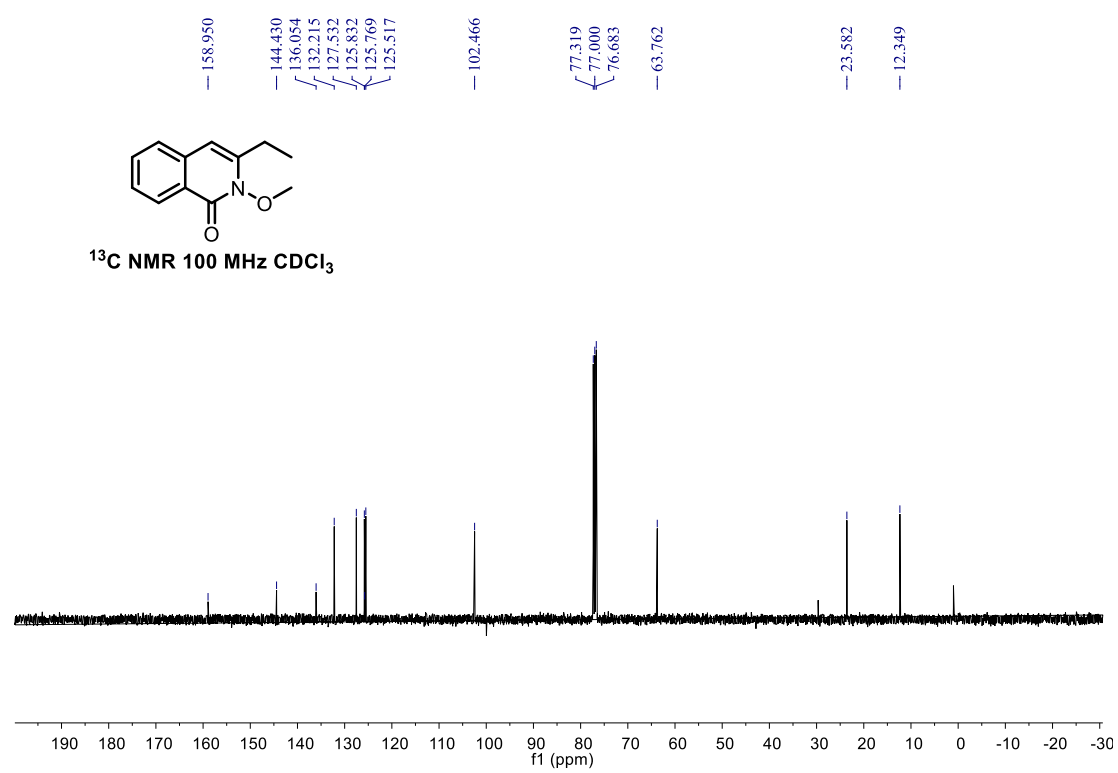
^{1}H NMR 400 MHz $CDCl_3$



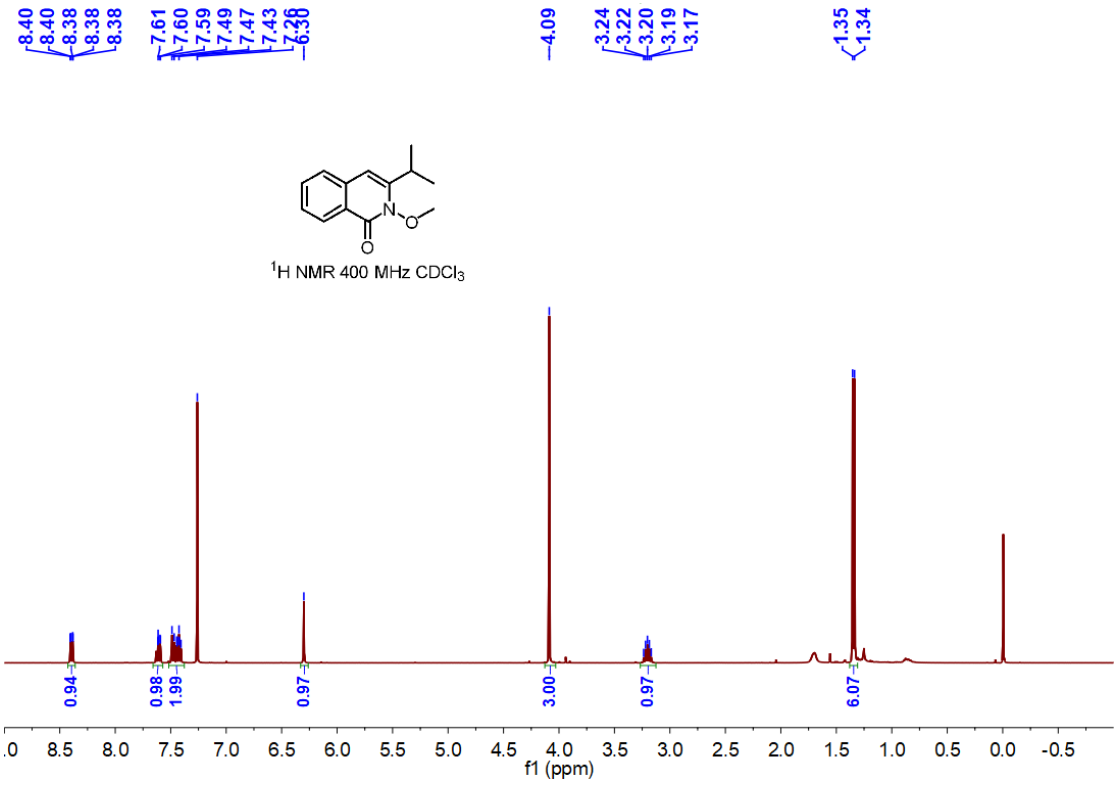
^{13}C NMR 100 MHz $CDCl_3$



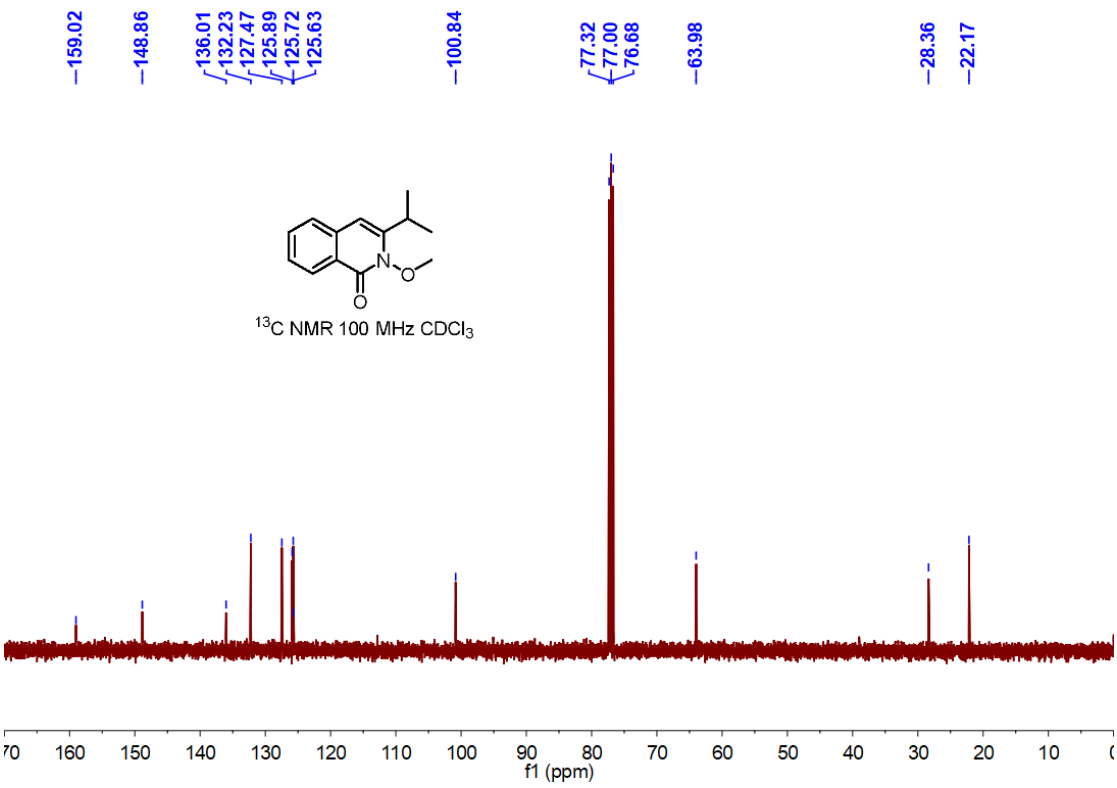
^{1}H NMR 400 MHz $CDCl_3$



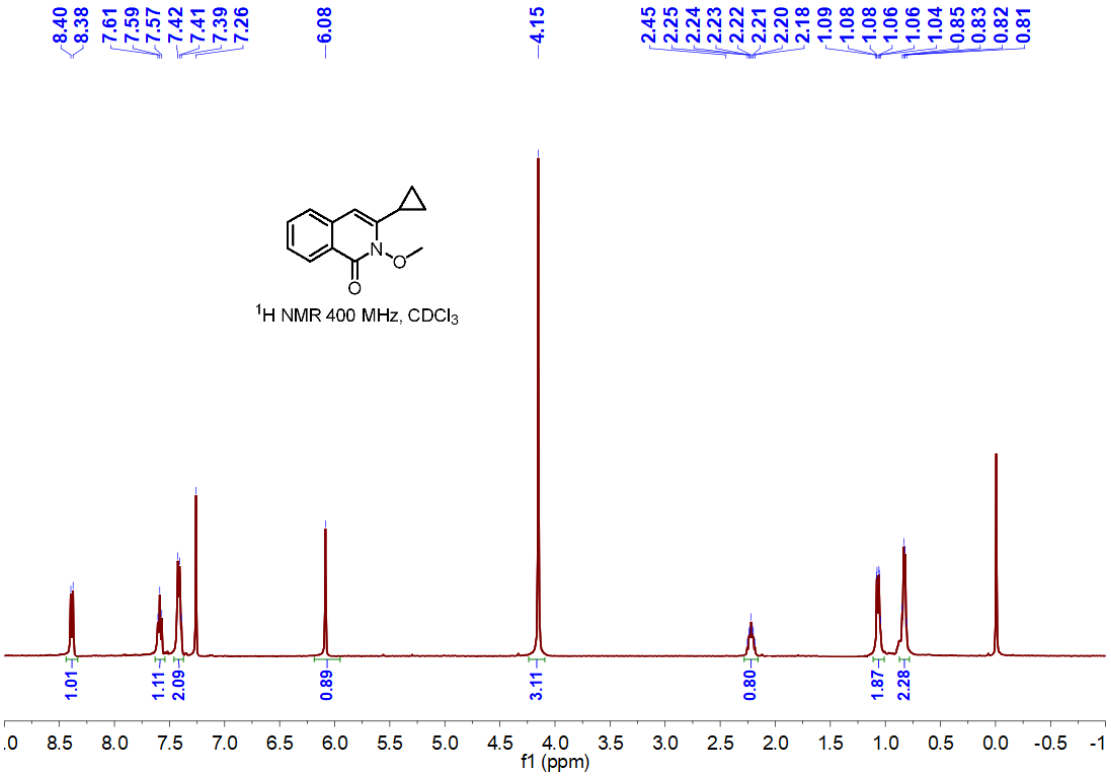
^{13}C NMR 100 MHz $CDCl_3$

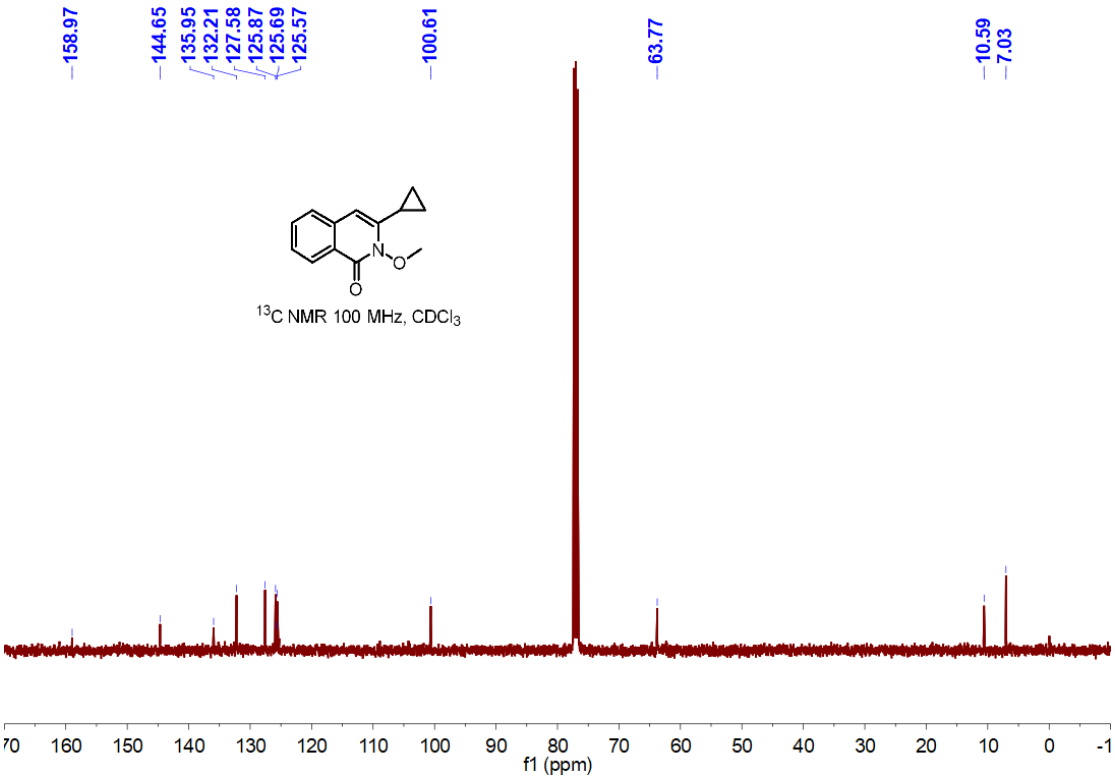


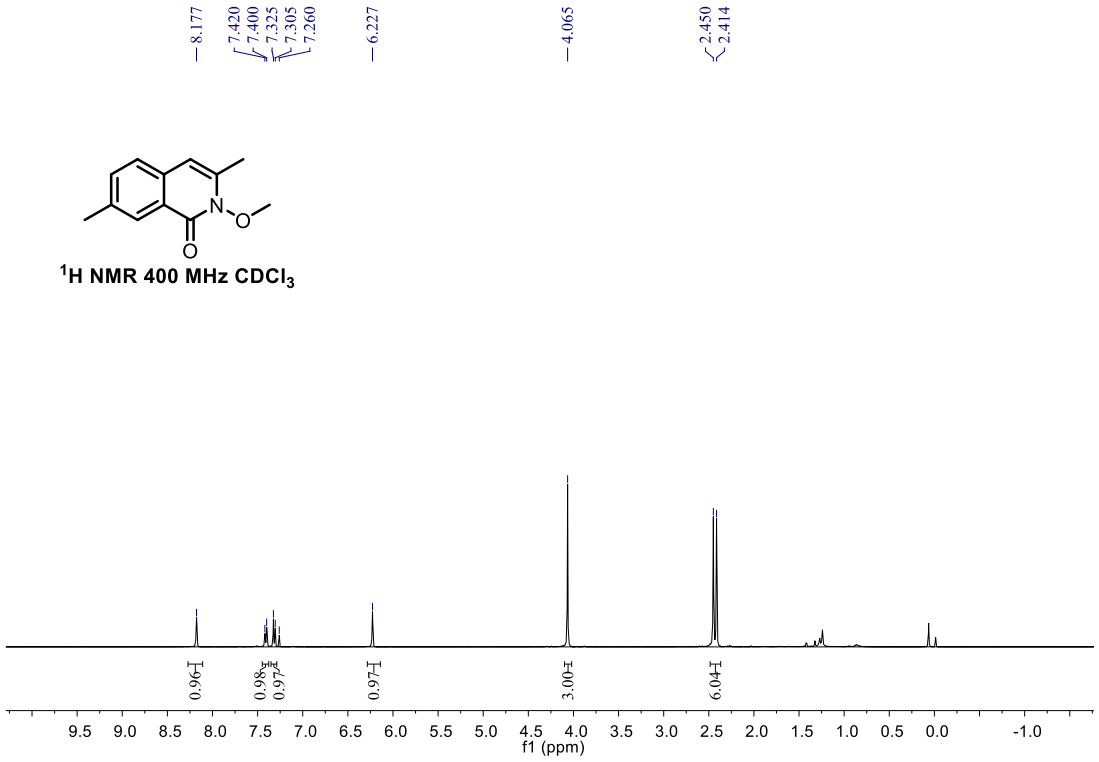
^{1}H NMR 400 MHz $CDCl_3$



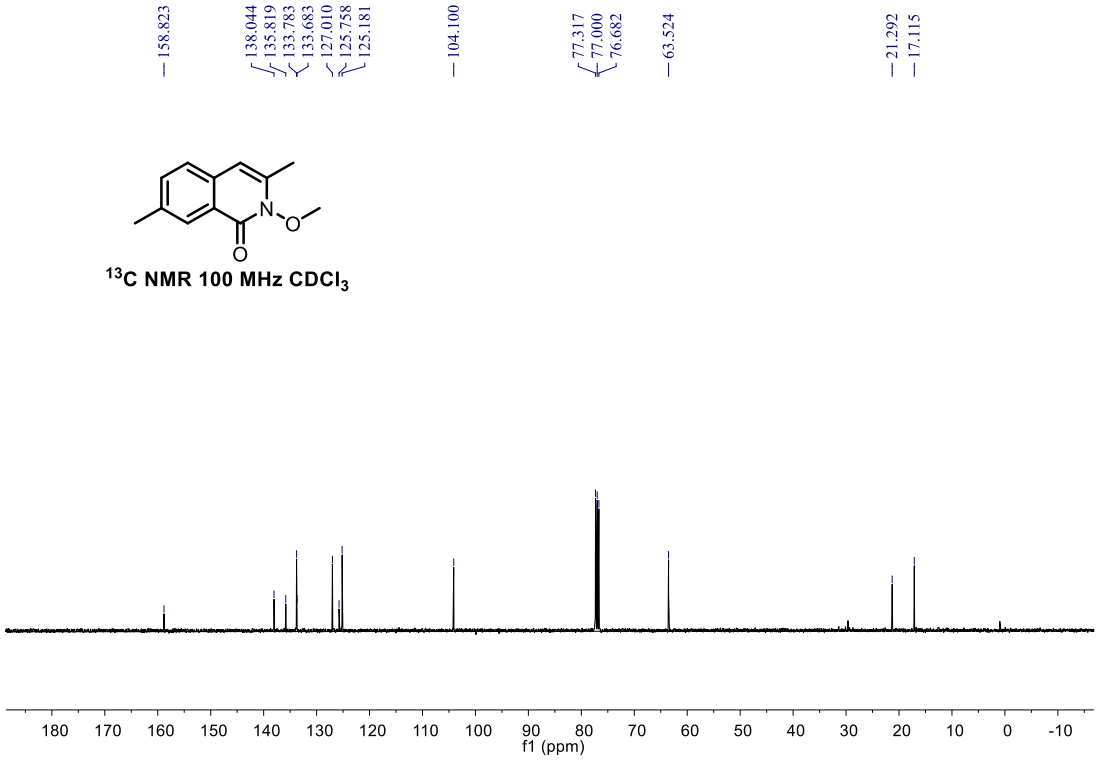
^{13}C NMR 100 MHz $CDCl_3$


8.40
8.38
7.61
7.59
7.57
7.42
7.41
7.39
7.26
6.08
4.15
2.45
2.25
2.24
2.23
2.22
2.21
2.20
2.18
1.09
1.08
1.08
1.06
1.06
1.04
0.85
0.83
0.82
0.81
1H NMR 400 MHz, CDCl3
1.01
1.11
2.09
0.89
3.11
0.80
1.87
2.28
f1 (ppm)

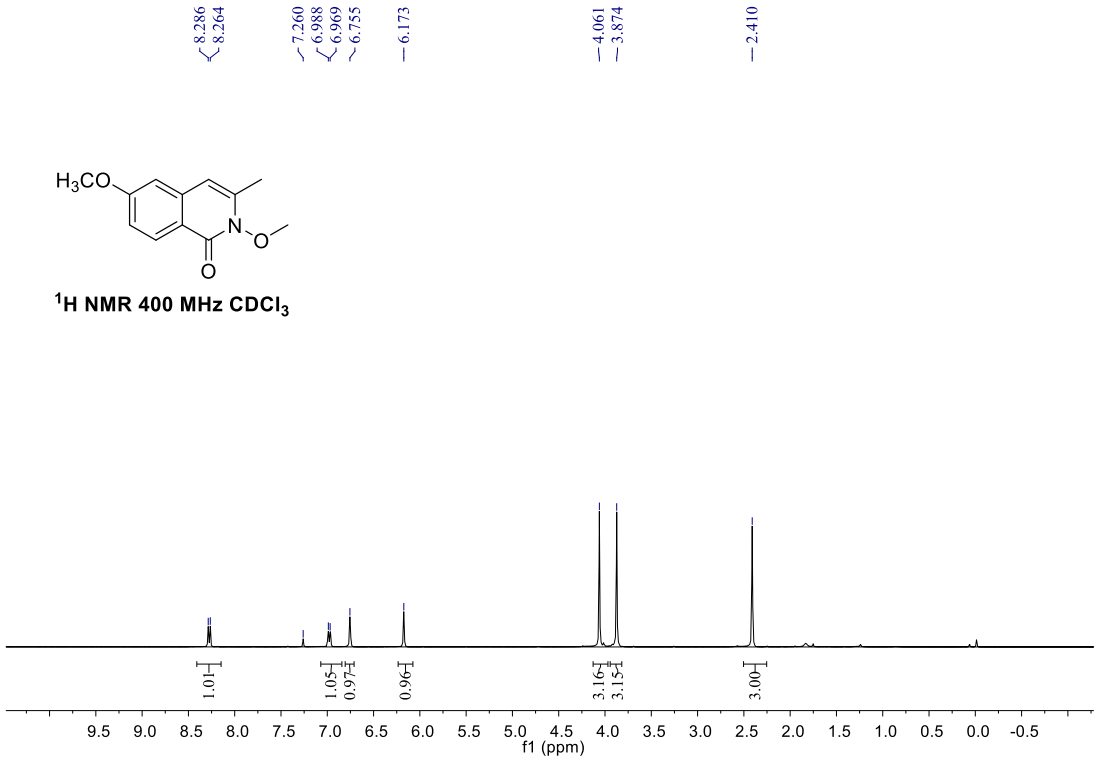

158.97
144.65
135.95
132.21
127.58
125.87
125.69
125.57
100.61
63.77
10.59
7.03
13C NMR 100 MHz, CDCl3
f1 (ppm)

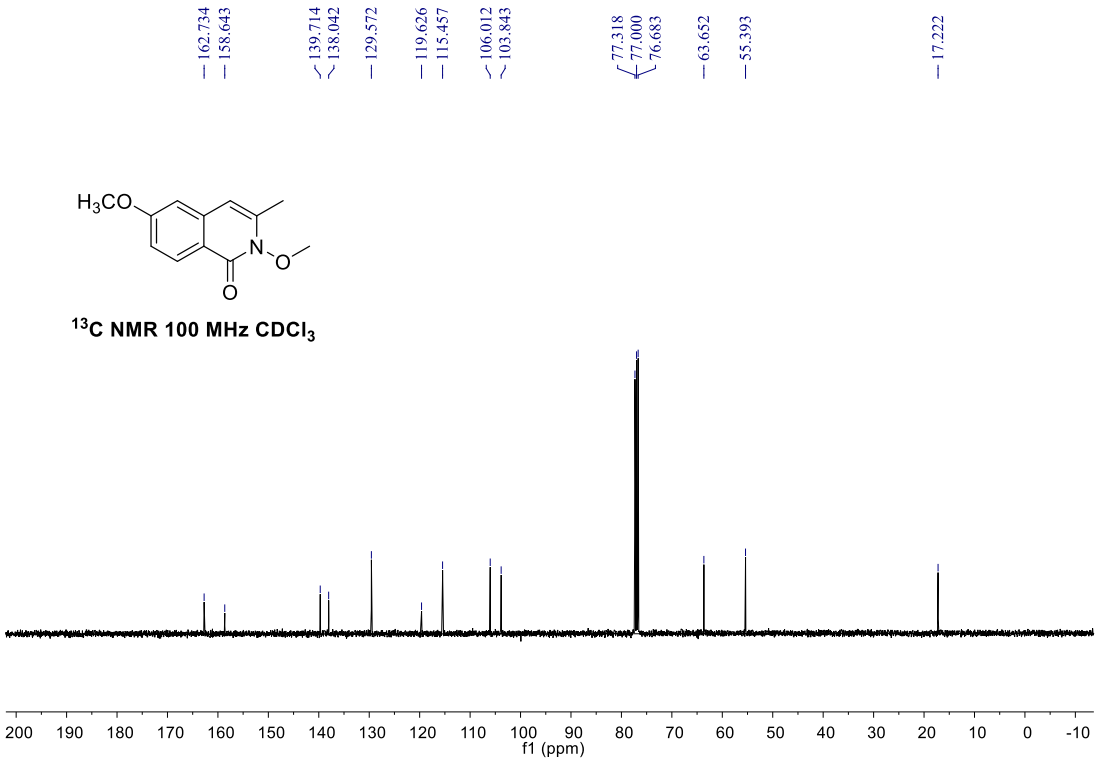


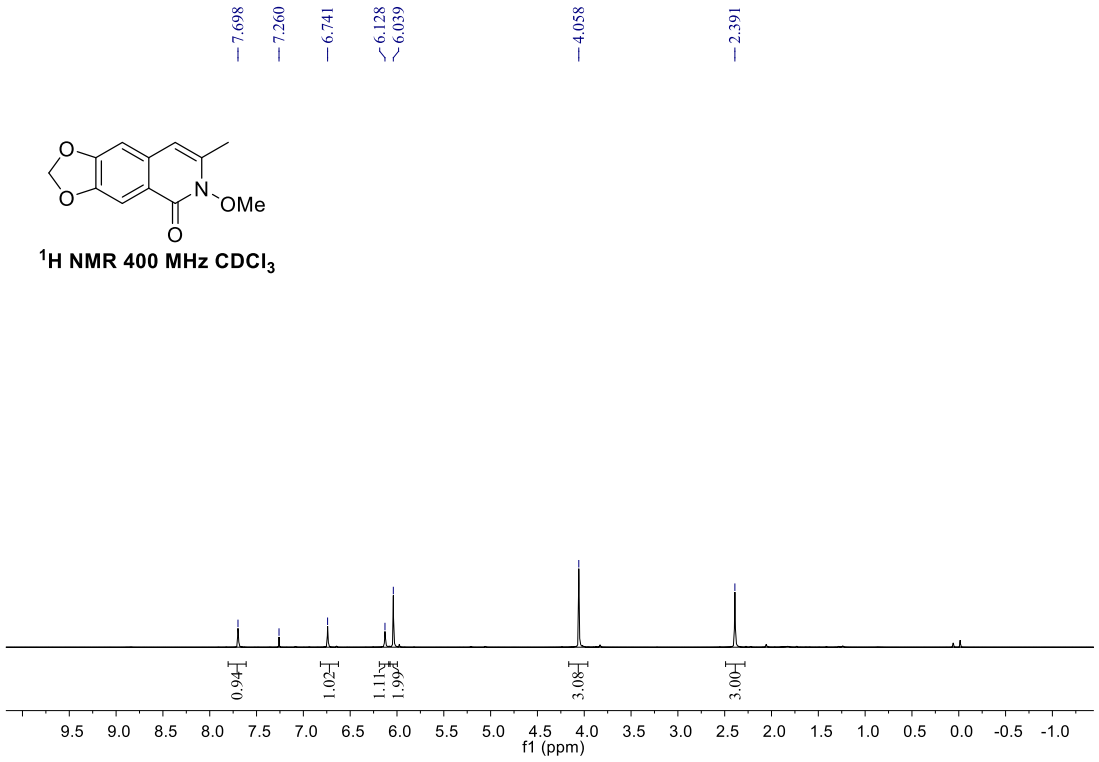
^{1}H NMR 400 MHz $CDCl_3$



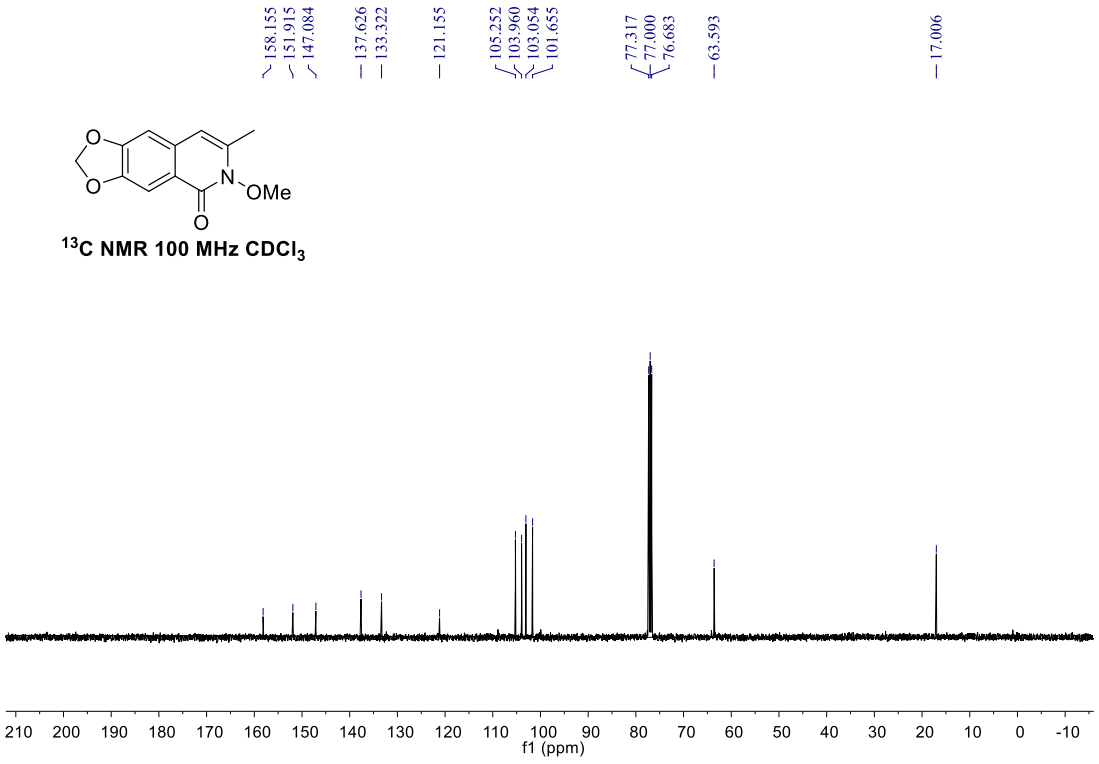
^{13}C NMR 100 MHz $CDCl_3$


8.286
8.264
7.260
6.988
6.969
6.755
6.173
4.061
3.874
2.410
H3CO
N
O
O
1H NMR 400 MHz CDCl3
1.01
1.05
0.97
0.96
3.16
3.15
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

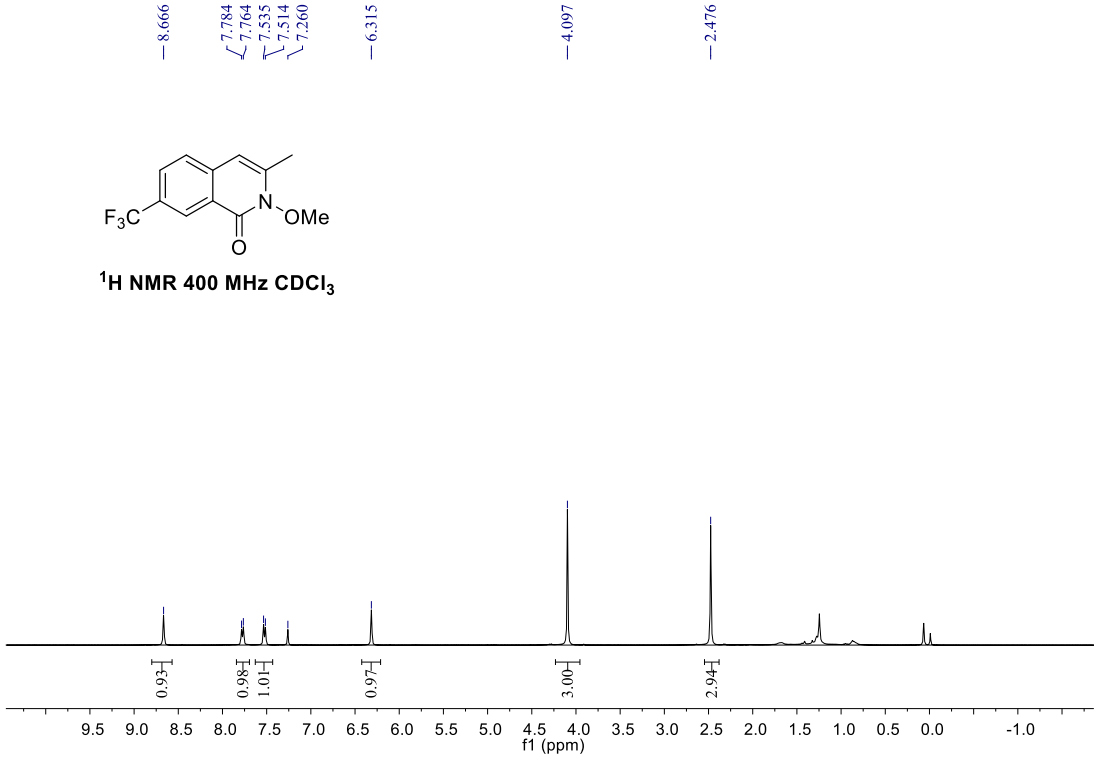

162.734
158.643
139.714
138.042
129.572
119.626
115.457
106.012
103.843
77.318
77.000
76.683
63.652
55.393
17.222
H3CO
N
O
O
13C NMR 100 MHz CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



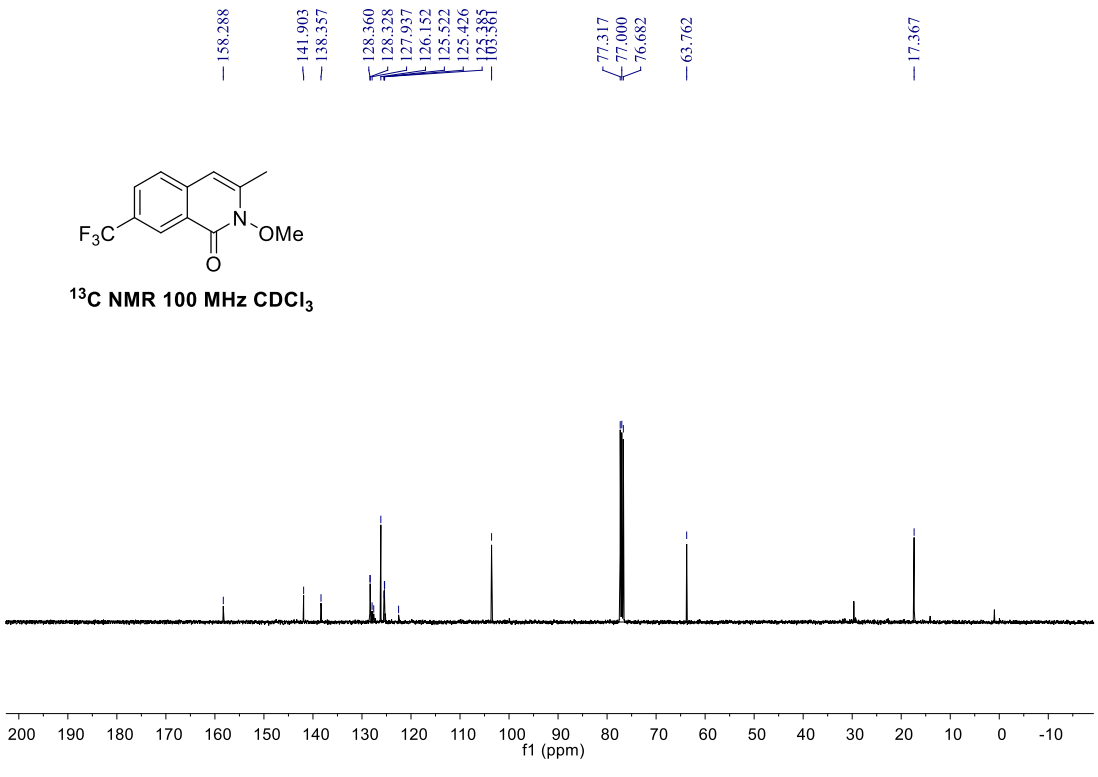
^{1}H NMR 400 MHz $CDCl_3$



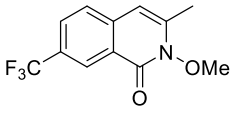
^{13}C NMR 100 MHz $CDCl_3$



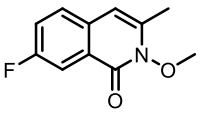
^{1}H NMR 400 MHz $CDCl_3$



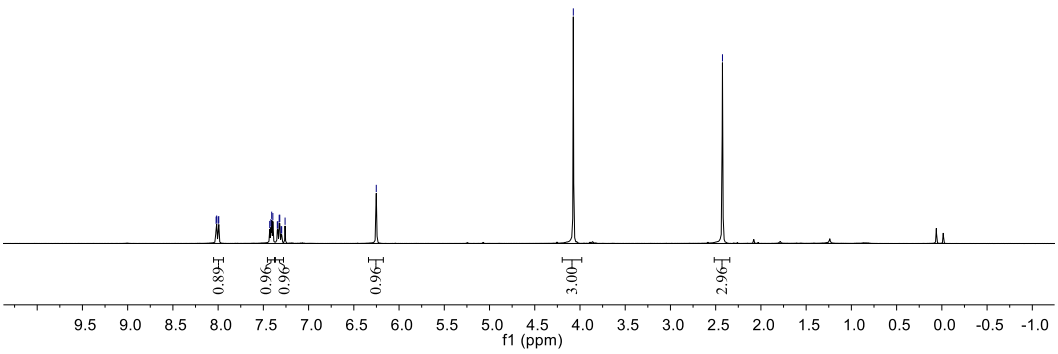
^{13}C NMR 100 MHz $CDCl_3$

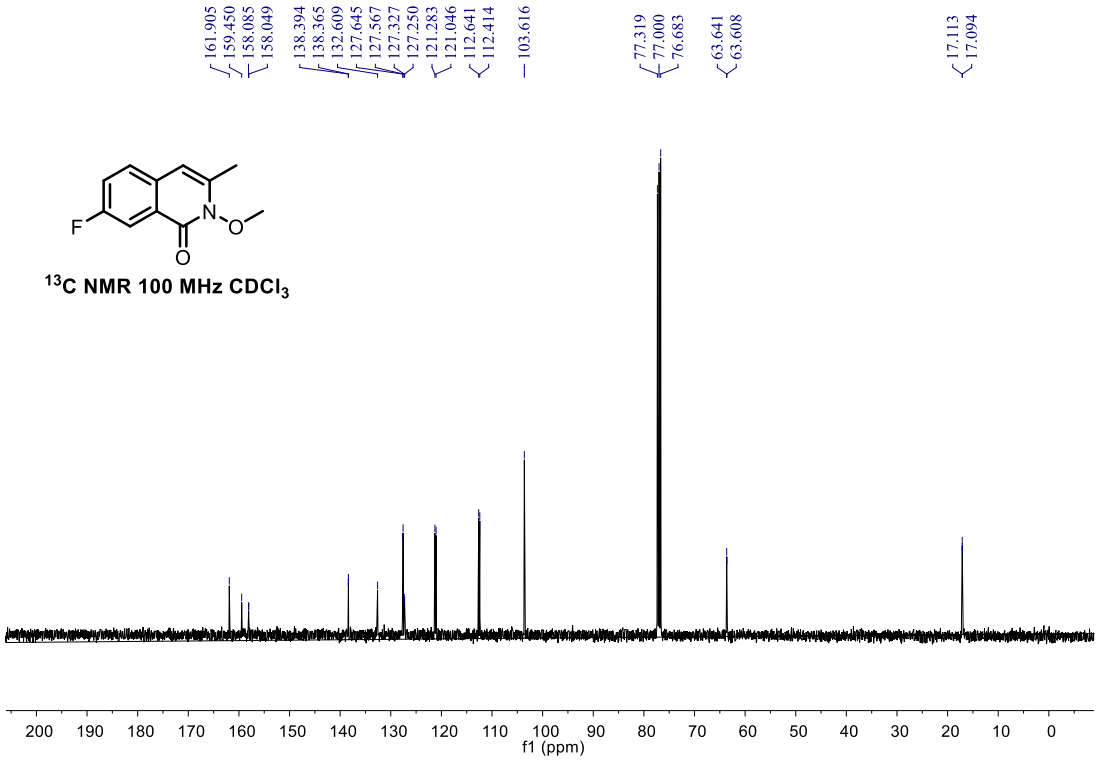


^{19}F NMR 376 MHz $CDCl_3$

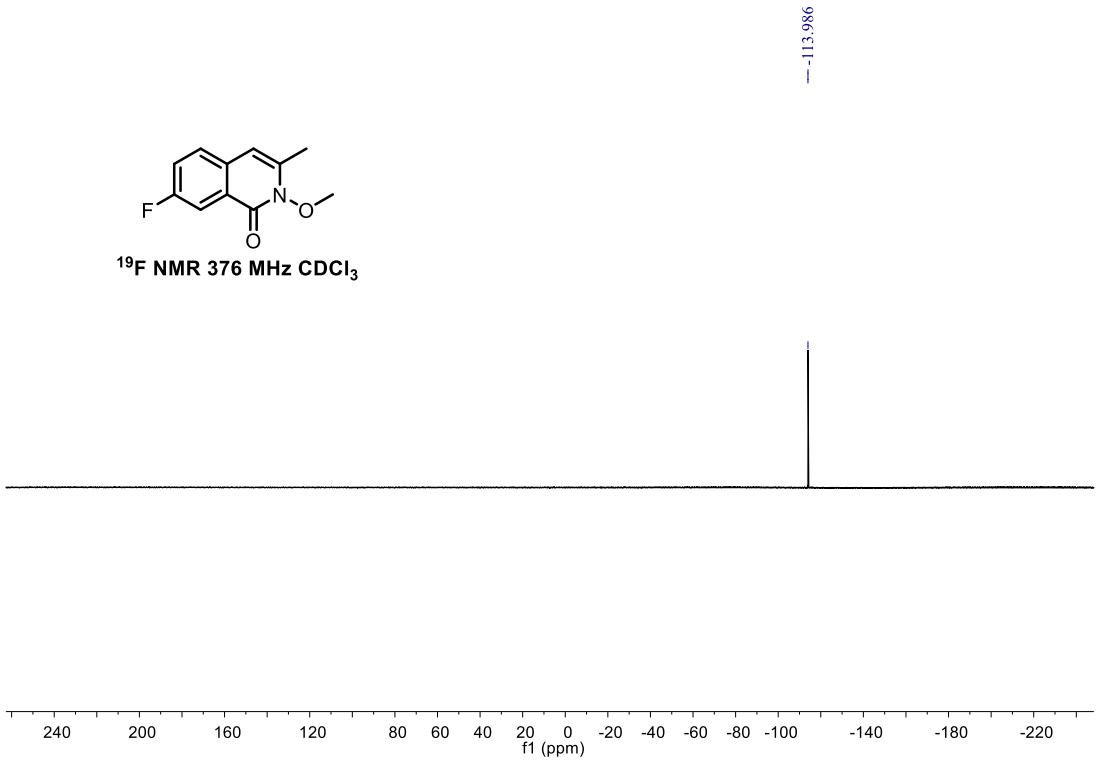


^{1}H NMR 400 MHz $CDCl_3$

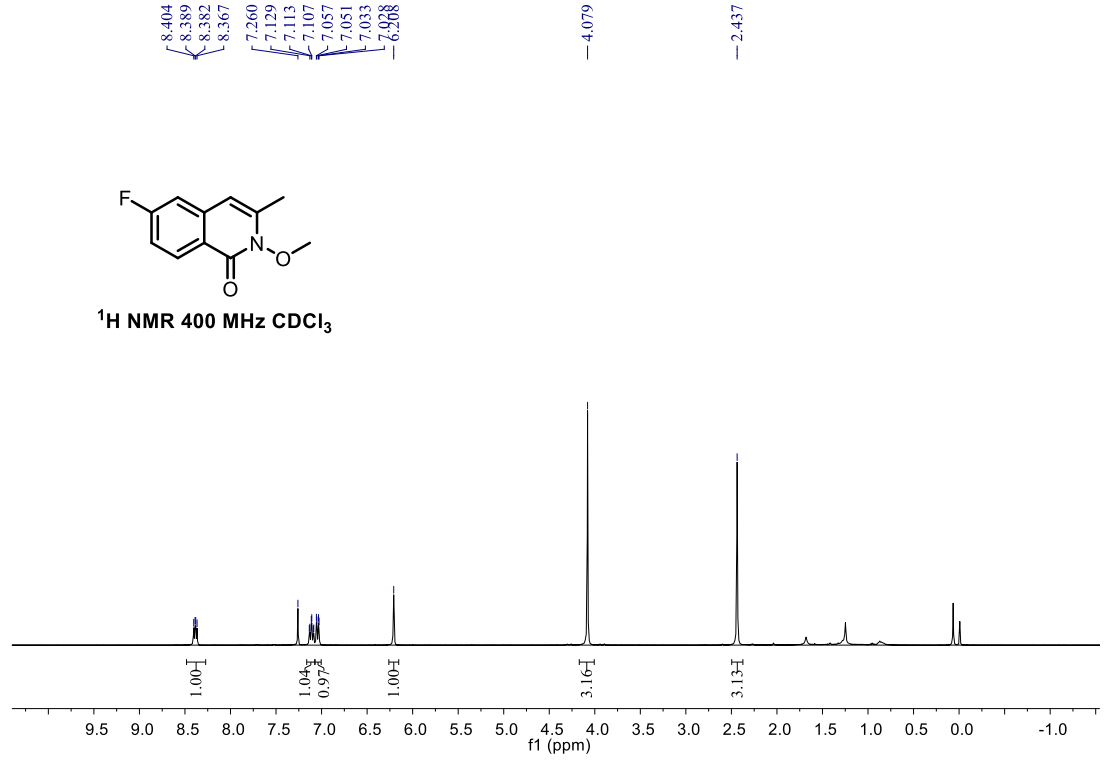


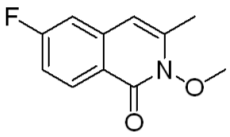


^{13}C NMR 100 MHz $CDCl_3$

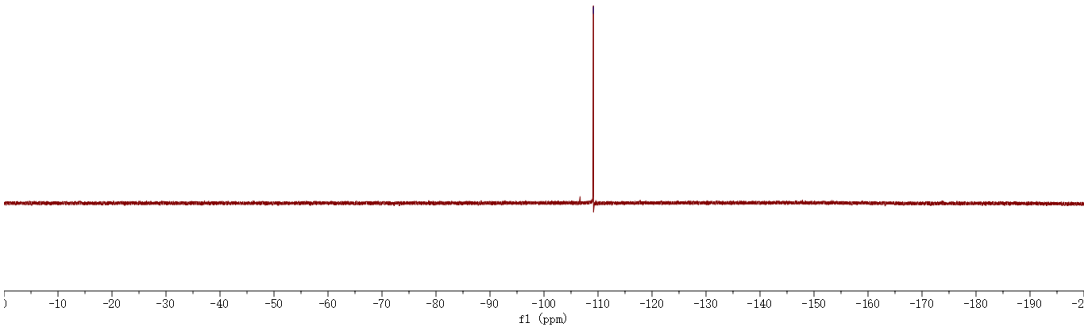


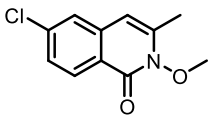
^{19}F NMR 376 MHz $CDCl_3$


8.404
8.389
8.382
8.367
7.260
7.129
7.113
7.107
7.057
7.051
7.033
6.208
4.079
2.437
F
N
O
O
1H NMR 400 MHz CDCl3
1.00
1.04
0.97
1.00
3.16
3.13
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0
f1 (ppm)
166.446
163.938
158.304
140.671
138.238
138.133
130.857
130.756
122.460
114.737
114.502
110.130
109.912
103.516
103.484
77.317
77.000
76.682
63.717
17.267
F
N
O
O
13C NMR 100 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

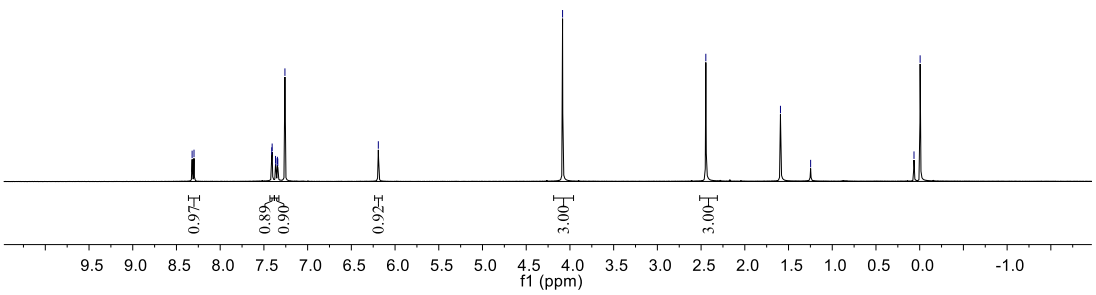


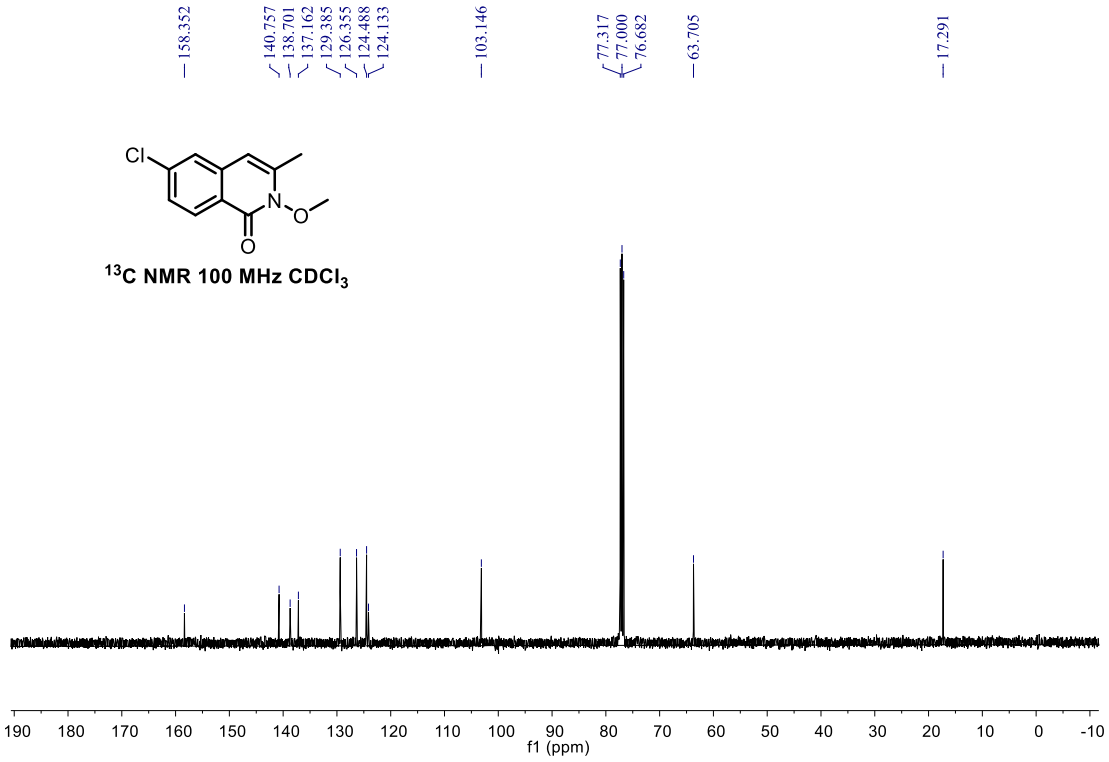
^{19}F NMR 376MHz, $CDCl_3$

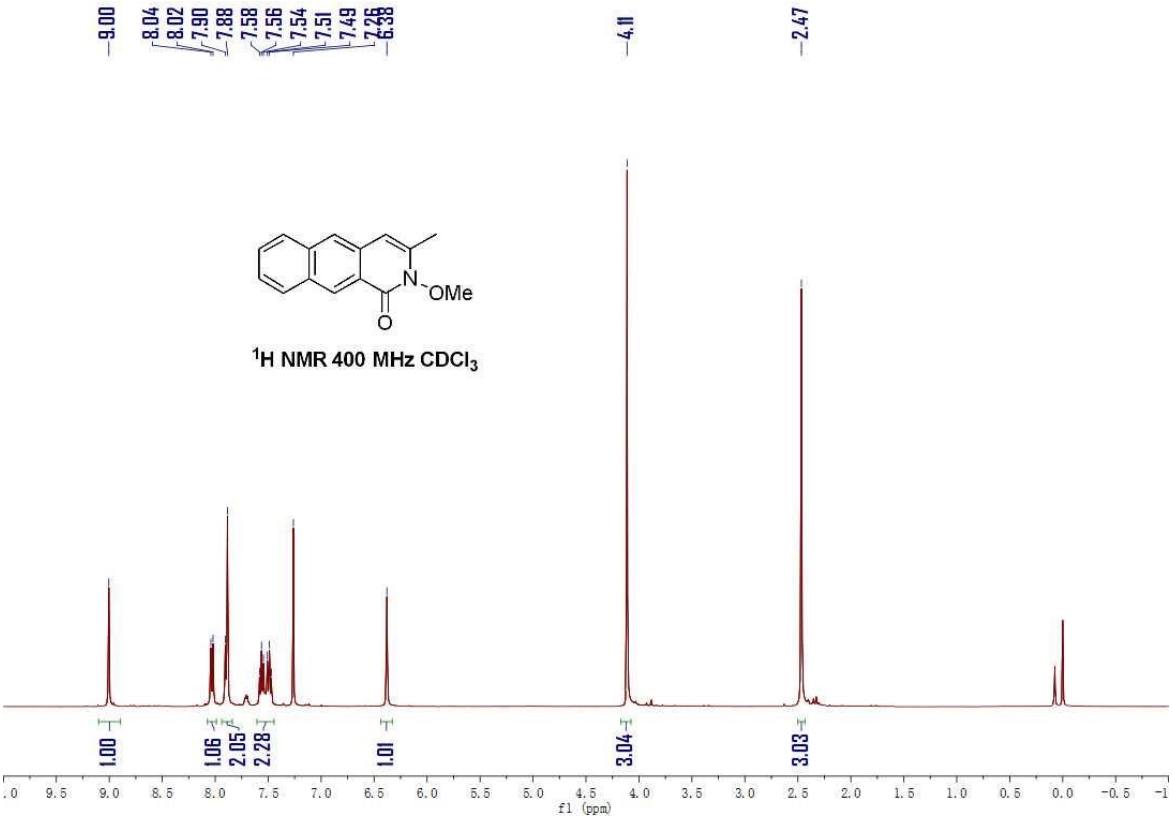


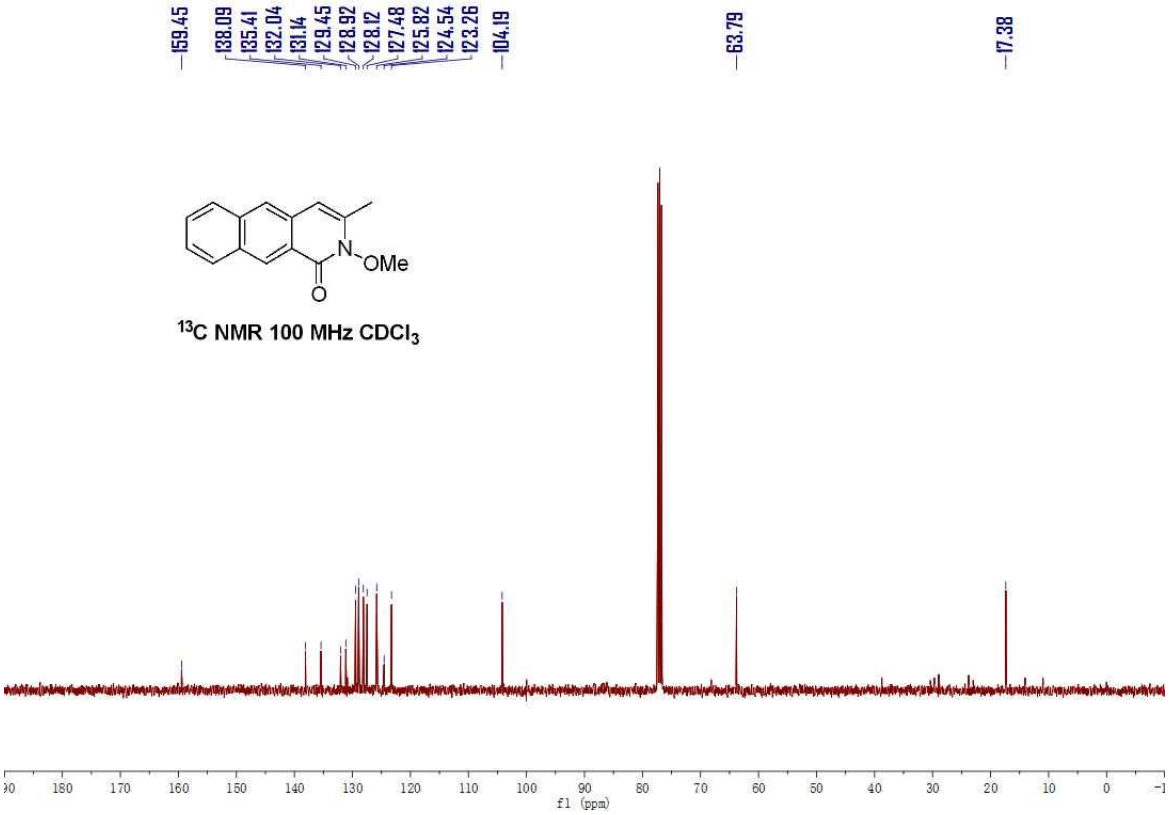


^{1}H NMR 400 MHz $CDCl_3$

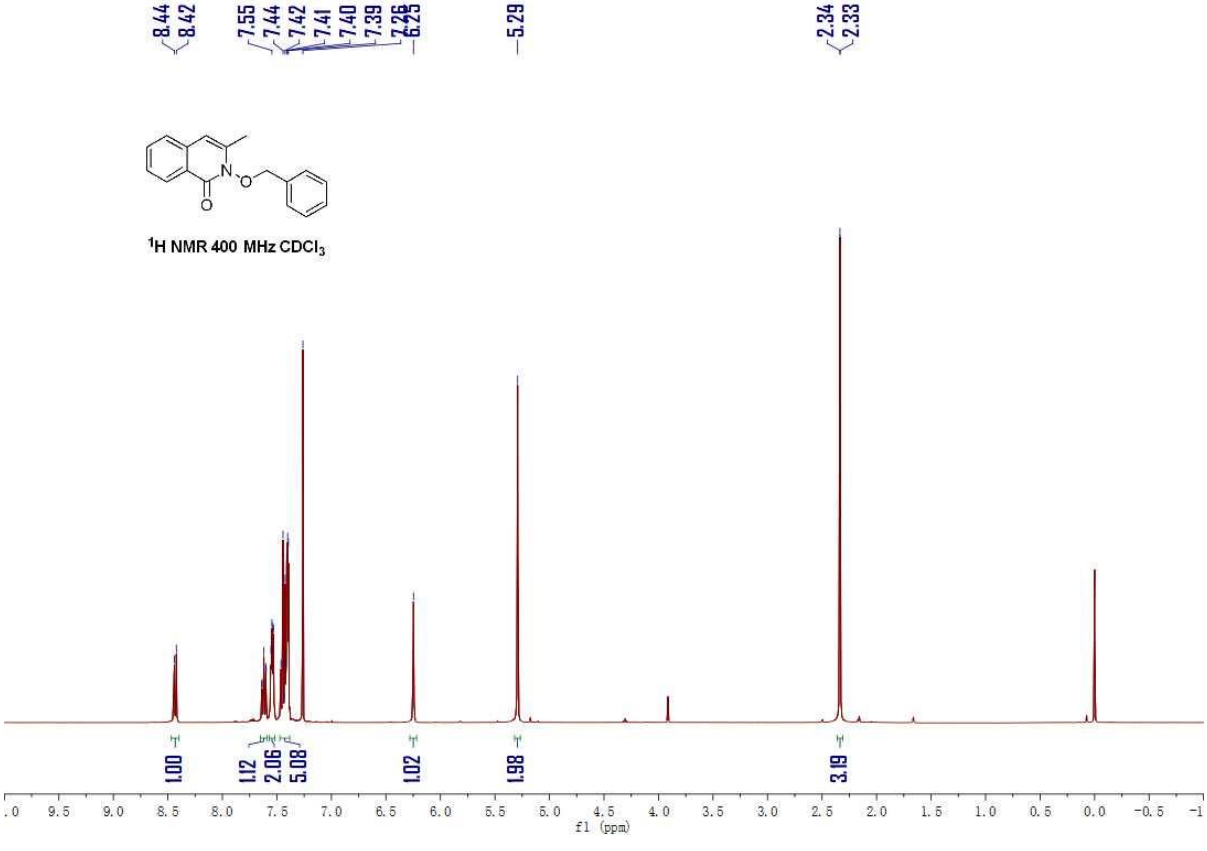



158.352
140.757
138.701
137.162
129.385
126.355
124.488
124.133
103.146
77.317
77.000
76.682
63.705
17.291
Cl
N
O
O
13C NMR 100 MHz CDCl3
f1 (ppm)

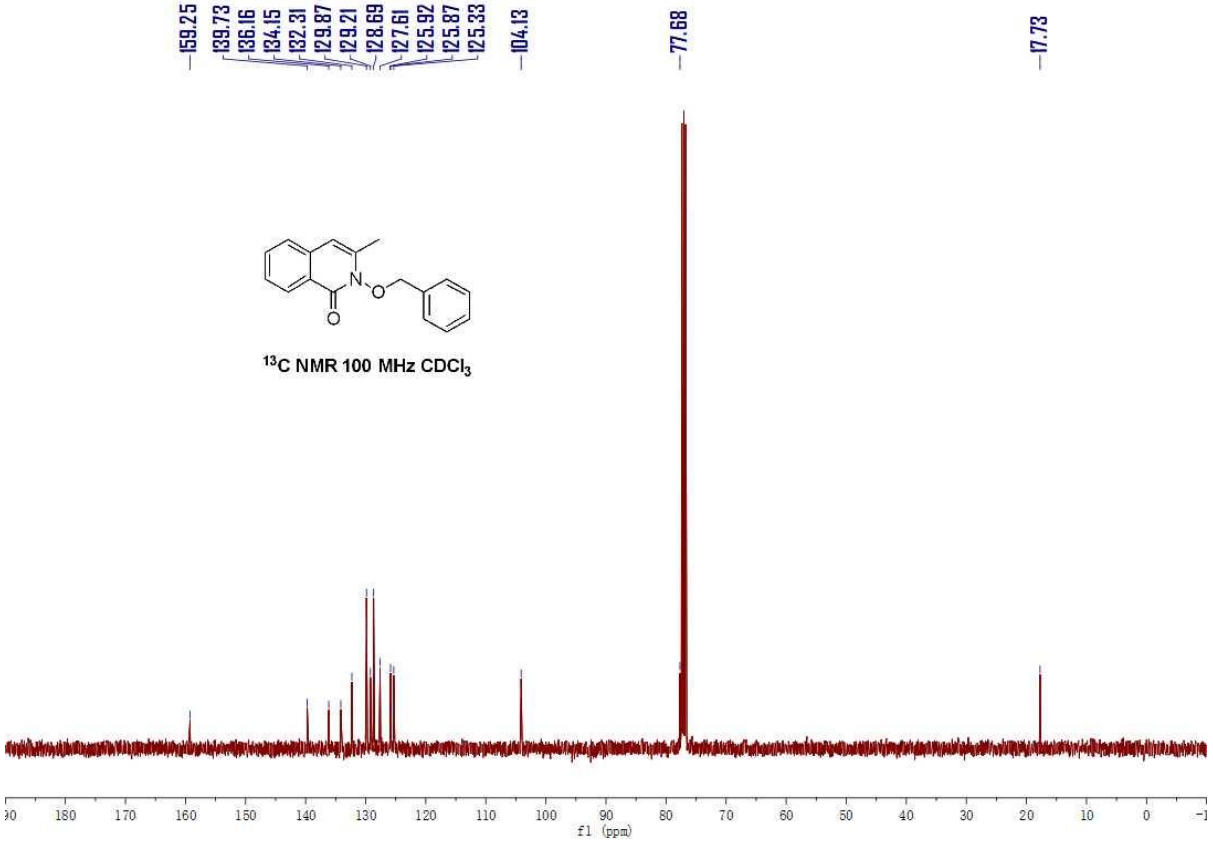

9.00
8.04
8.02
7.90
7.88
7.58
7.56
7.54
7.51
7.49
4.11
2.47
N
OMe
O
1H NMR 400 MHz CDCl3
1.00
1.06
2.05
2.28
1.01
3.04
3.03
f1 (ppm)



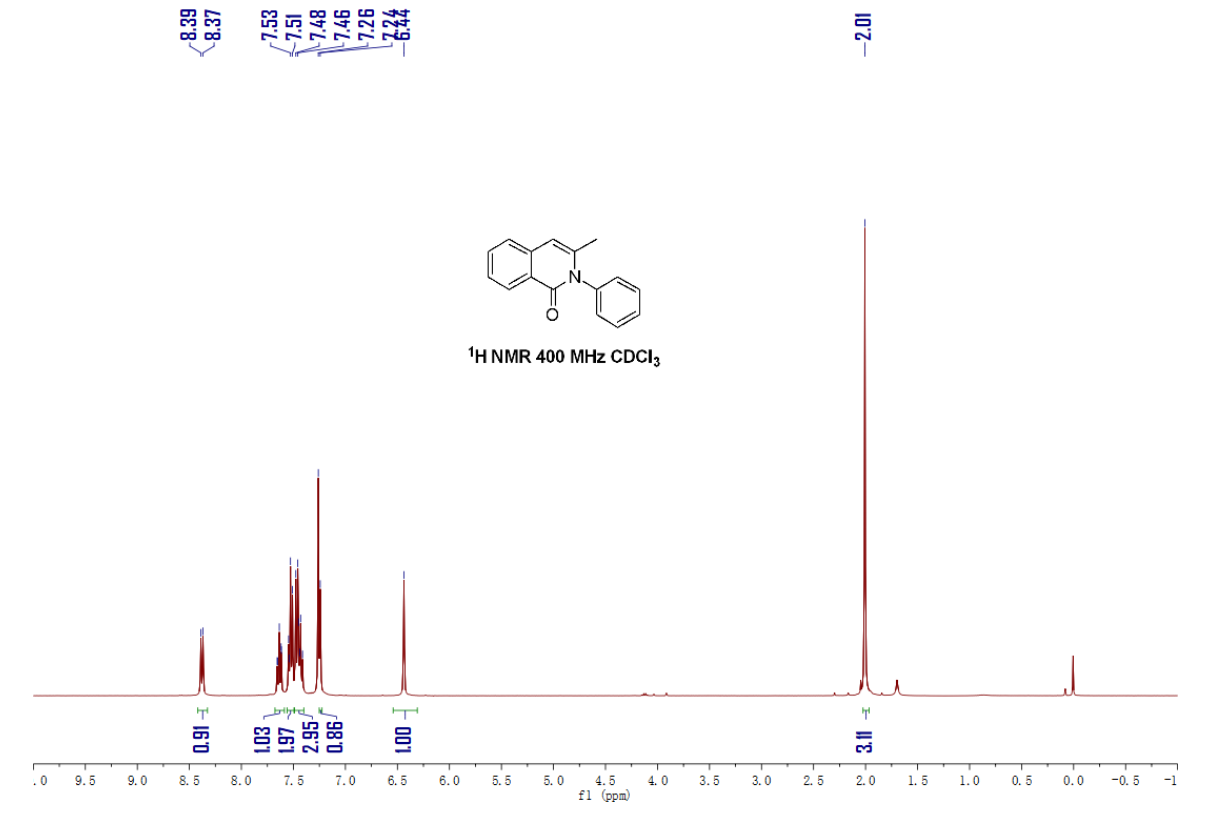
^{13}C NMR 100 MHz $CDCl_3$



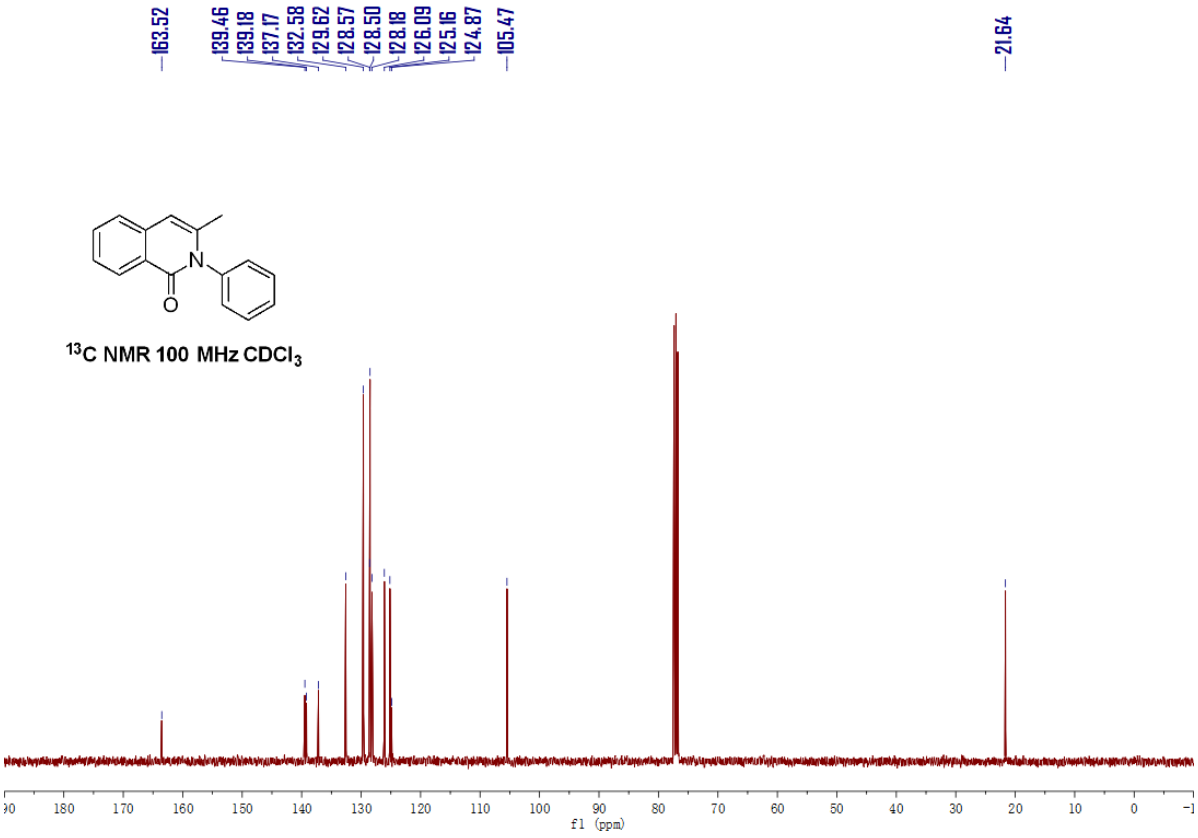
^{1}H NMR 400 MHz $CDCl_3$

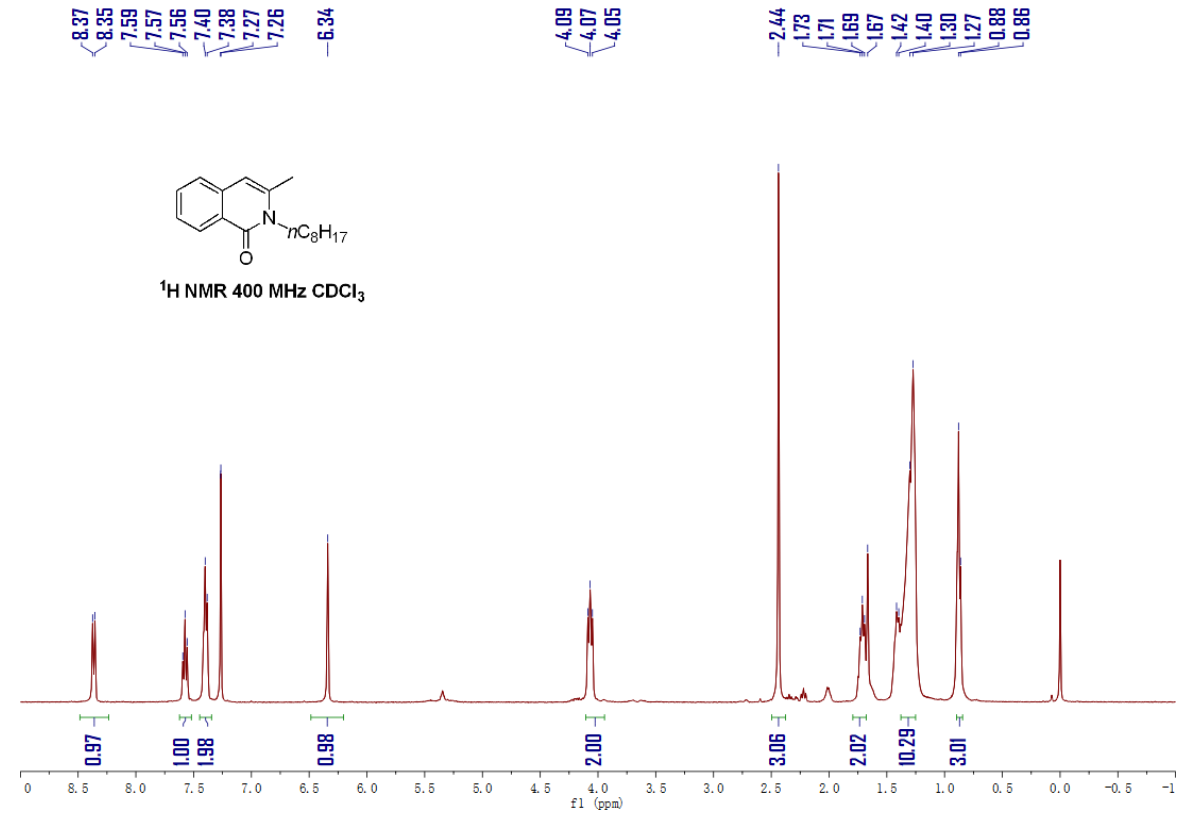


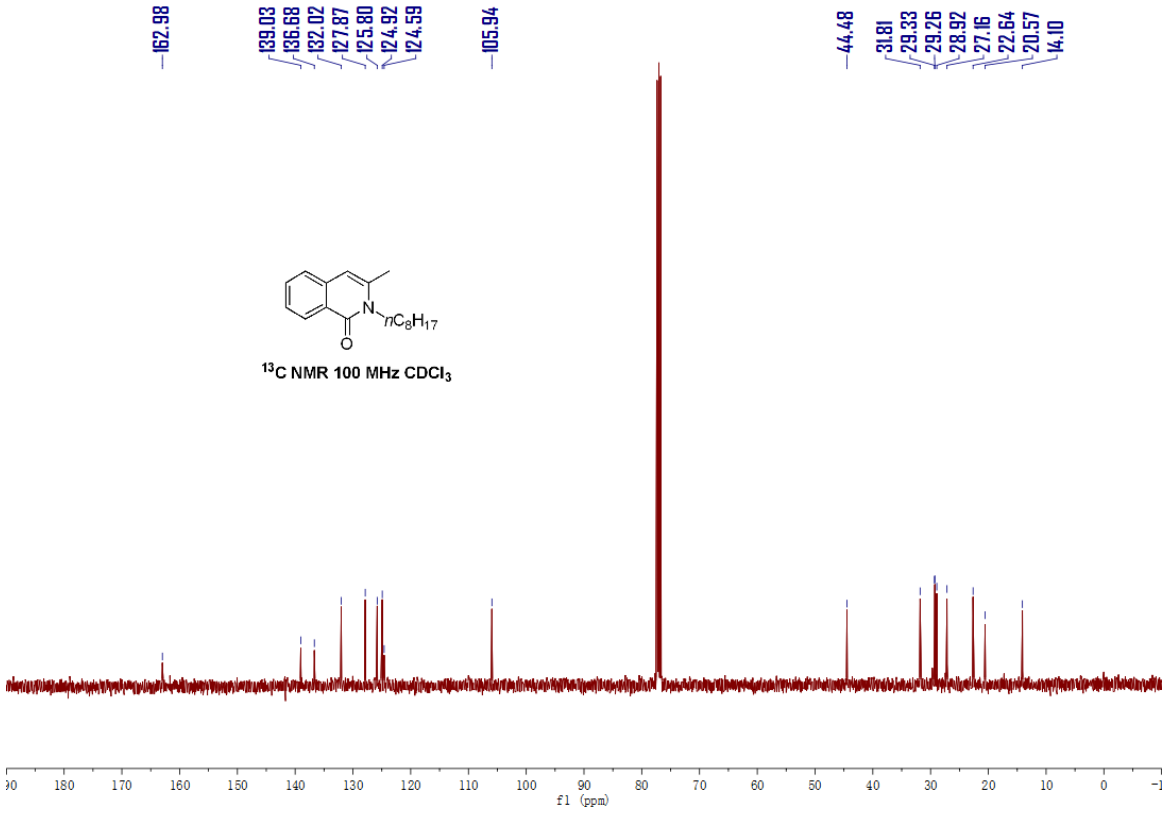
^{13}C NMR 100 MHz $CDCl_3$



^{1}H NMR 400 MHz $CDCl_3$


163.52
139.46
139.18
137.17
132.58
129.62
128.57
128.50
128.18
126.09
125.16
124.87
105.47
21.64
13C NMR 100 MHz CDCl3
f1 (ppm)


8.37
8.35
7.59
7.57
7.56
7.40
7.38
7.27
7.26
6.34
4.09
4.07
4.05
2.44
1.73
1.71
1.69
1.67
1.42
1.40
1.30
1.27
0.88
0.86
nC8H17
1H NMR 400 MHz CDCl3
0.97
1.00
1.98
0.98
2.00
3.06
2.02
10.29
3.01
f1 (ppm)


162.98
139.03
136.68
132.02
127.87
125.80
124.92
124.59
105.94
44.48
31.81
29.33
29.26
28.92
27.16
22.64
20.57
14.10
N
nC6H17
O
13C NMR 100 MHz CDCl3
f1 (ppm)