Supplementary Information

For

Bypassing the Limitations of Directed C–H Functionalizations of Heterocycles

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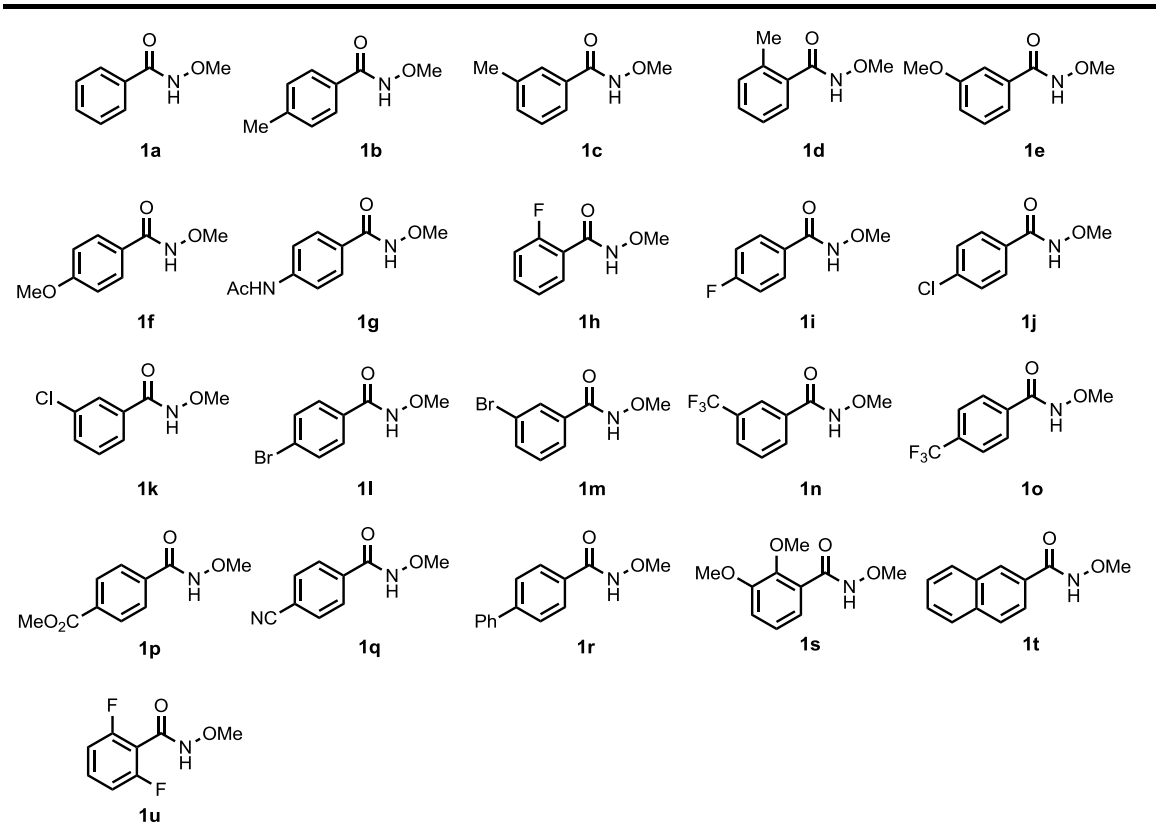
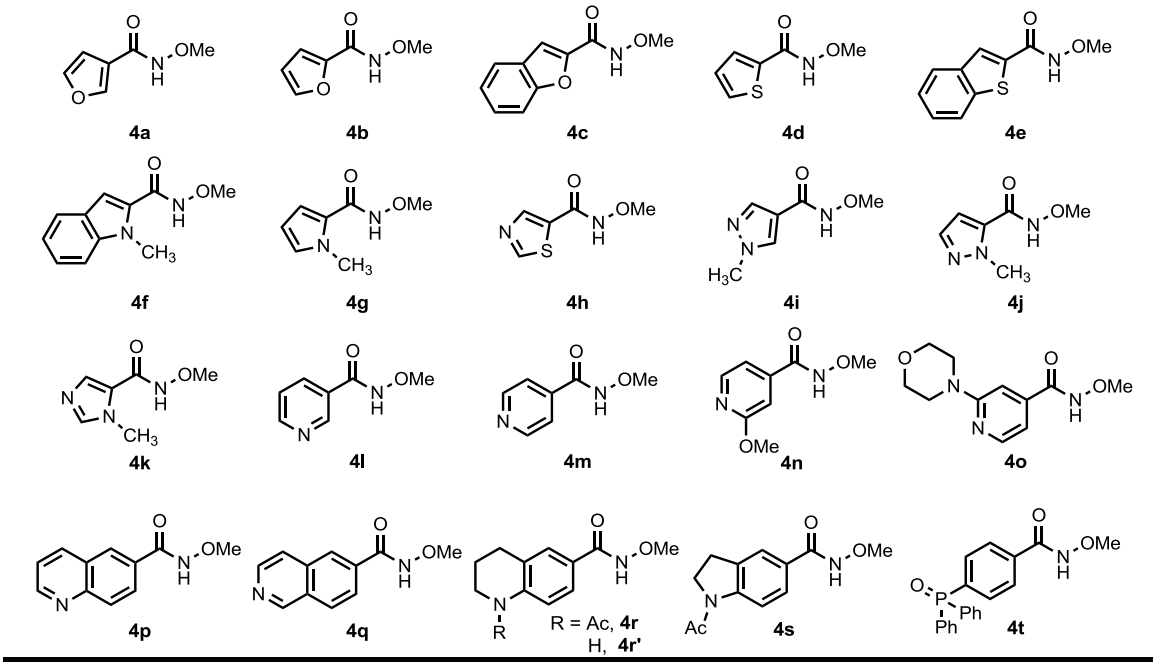
1. General Information

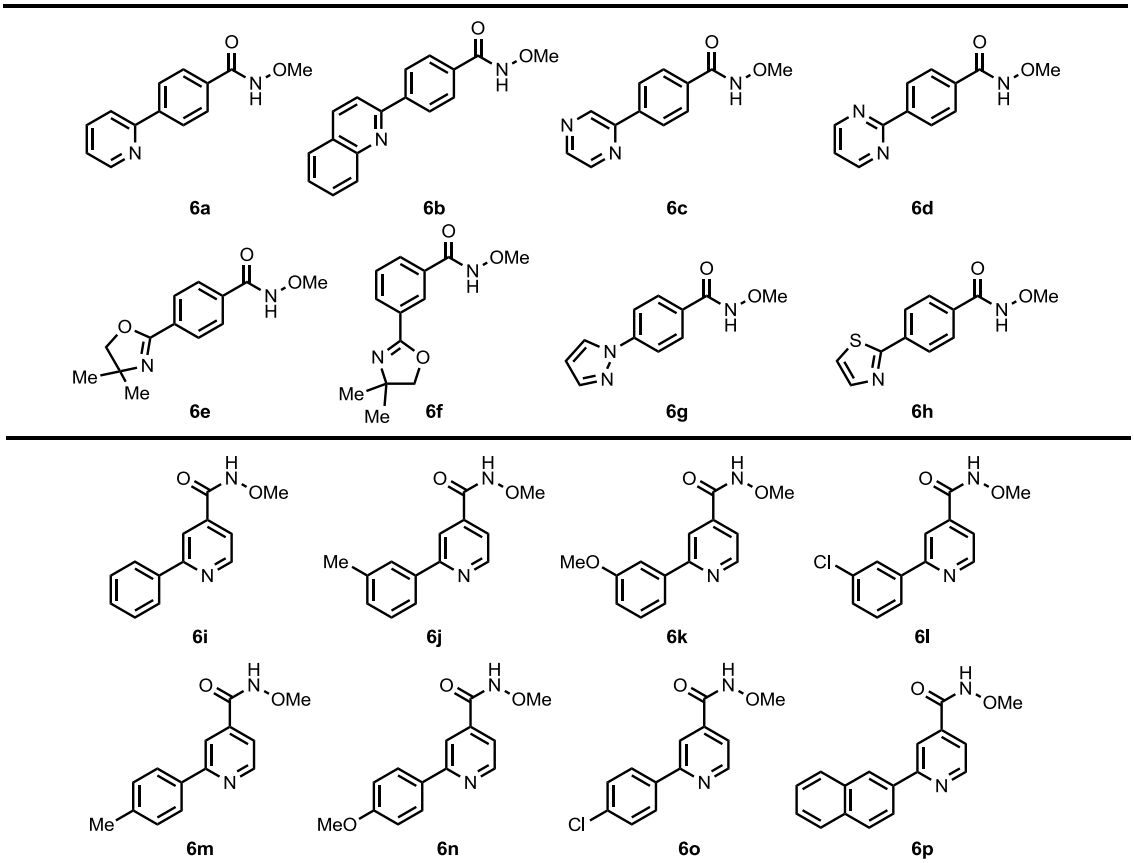
All solvents and reagents were obtained from commercial sources and were purified according to standard procedures before use. $Pd_2(dba)_3$ was obtained from Sigma-Aldrich and *t*-BuNC was obtained from TCI. Carboxylic acids or carboxylic acid chlorides were obtained from Energy Chemical, J&K, TCI, Alfa-Aesar. ^1H and ^{13}C NMR spectra were recorded on Agilent AV 400, Agilent Mercury 600 (600 MHz and 150 MHz, respectively), Varian Inova 400 (400 MHz and 100 MHz, respectively) instruments. The peaks were internally referenced to TMS (0.00 ppm) or residual undeuterated solvent signal. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. High resolution mass spectra were recorded at the Center for Mass Spectrometry, Shanghai Institute of Organic Chemistry.

2. Experimental Section

2.1 Preparation of Substrates



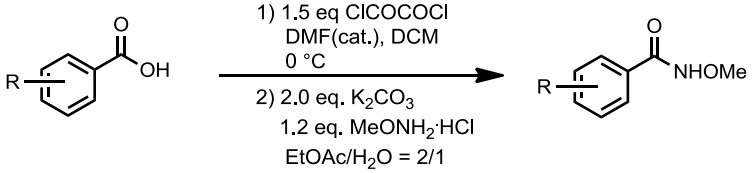




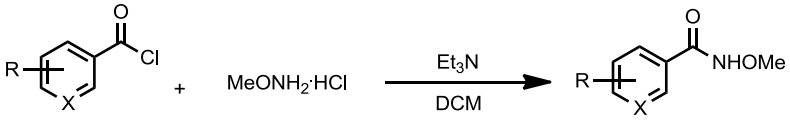
Compounds **1a-1u** and **4t** were prepared following **typical procedure 1**. Compounds **4h-4o**

were prepared following **typical procedure 2.** Compounds **1g**, **4p**-**4**t and **6a**-**6p** were prepared following typical **procedure 3**.

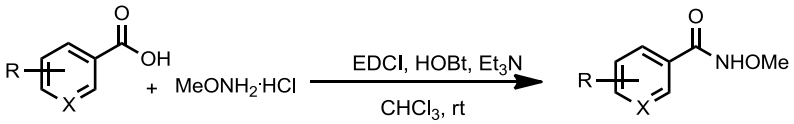
1a-1e,[1] **1f-1g,**[2] **1h-1j,**[3] **1k,**[4] **1l,**[3] **1m,**[5] **1n,**[1] **1o,**[6] **1p,**[3] **1q,**[7] **1r,**[1] **1t,** [1] **4a-4b,**[8] **4c,**[6] **4d-4g**[9] are known Compounds.



Typical Procedure 1: A dried round-bottom flask was charged with the acid (4.5 mmol), DCM (15 mL), and 2 drops of DMF. Then, oxalyl chloride (0.60 mL, d = 1.48, 0.876 g, 6.9 mmol) was added dropwise within 5 min at 0 °C. The resulting mixture was stirred at rt for 3.5 h and concentrated under reduced pressure. The residues was dissolved in EtOAc (40 mL) and K_2CO_3 (1.24 g, 9.0 mmol), $MeONH_2{\cdot}HCl$ (458 mg, 5.4 mmol), water (20 mL) were added sequentially. The resulting mixture was stirred for 20 h at rt and extracted with EtOAc (50 mL). The organic layer was washed with saturated aqueous $NaHCO_3$ (15 mL × 2), brine (15 mL × 2), and dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was recrystallized (EtOAc) to afford the product. In the case of an oil, the crude product was purified by silica gel column chromatography, using EtOAc/hexane as the eluent.



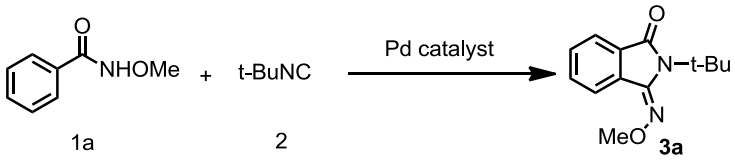
Typical Procedure 2: A mixture of methoxylamine hydrochloride (840 mg, 10 mmol) and Et_3N (3.03 g, 30 mmol) in DCM (20 mL) was stirred at rt for 3 h, and then cooled to 0 °C upon which acyl chloride (10 mmol) was added slowly. The reaction mixture was then allowed to warm to rt and stirred overnight. Upon completion, the solvent was removed under reduced pressure. The residue was diluted with acetone (20 mL), filtered and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography, using EtOAc/hexane as the eluent.



Typical Procedure 3: A mixture of acid (3 mmol), methoxylamine hydrochloride (4.5 mmol), EDCI (3.6 mmol) and HOBt (4.2 mmol), Et_3N (1.3 mL) in $CHCl_3$ (15 mL) was stirred at rt for 24 h. Upon completion, the mixture was concentrated. The residue was diluted with acetone (20 mL), filtered and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography, using ethyl acetate/hexane as the eluent.

2.2 Optimization of Reaction Conditions

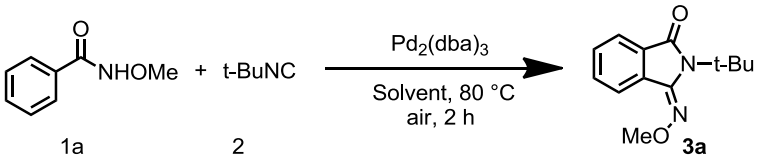
Table S1. Screening of Pd Sources[a,b]



Entry	Catalyst (mol %)	Yield (%)
1	$Pd_2(dba)_3$ (2.5)	94(93)[c]
2[d]	$Pd_2(dba)_3$ (2.5)	trace
3[e]	$Pd_2(dba)_3$ (2.5)	37
4	$Pd(dba)_2$ (5)	71
5	$Pd(OAc)_2$ (5)	49
6	$Pd(PPh_3)_4$ (5)	86
7	$PdCl_2$ (5)	0
8	$Pd(TFA)_2$ (5)	0
9	$Pd(OTf)_2(CH_3CN)_4$ (5)	0
10[f]	$Pd(TFA)_2$ (5)	0
11[f]	$Pd(OTf)_2(CH_3CN)_4$ (5)	0
12	$[Pd(allyl)Cl]_2$ (2.5)	2

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), 2.5 mol % of $Pd_2(dba)_3$, dioxane (1 mL), 80 °C, air, 30 min. [b] ^1H NMR yield using CH_2Br_2 as internal standard. [c] Isolated yield in the parenthesis. [d] Ar (1 atm). [e] O_2 (1 atm). [f] **dba** (7.5 mol %).

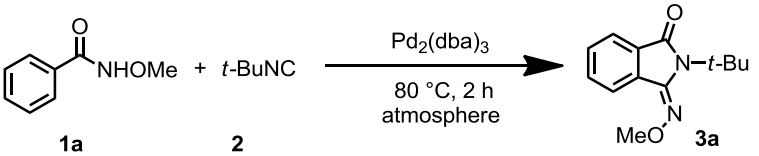
Table S2. Screening of Solvents[a,b]



Entry	Solvent	Yield (%)
1	dioxane	94
2	THF	67
3	Et_2O	35
4	DCM	8
5	CH_3CN	5
6	DMF	6
7	CH_3OH	2
8	DMSO	5
9	Acetone	8
10	Toluene	45

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), 2.5 mol % of $Pd_2(dba)_3$, solvent (1 mL), 80 °C, air, 2 h. [b] ^1H NMR yield using CH_2Br_2 as internal standard.

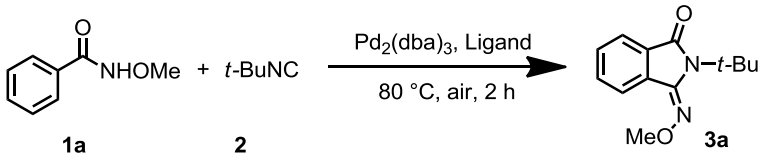
Table S3. Effect of Oxygen[a,b]



Entry	atmosphere	Yield (%)
1	air	94
2	O_2	48
3	Ar	trace

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), 2.5 mol % of $Pd_2(dba)_3$, dioxane (1 mL), 80 °C, 2 h. [b] ^1H NMR yield using CH_2Br_2 as internal standard.

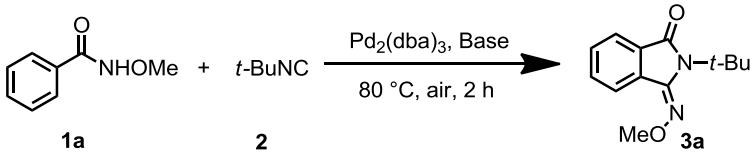
Table S4. Screening of Ligands[a,b]



Entry	Ligand	Yield (%)
1	1,10-phen (10 mol %)	96
2	bpy (10 mol %)	94
3	py (20 mol %)	93
4	Ac-Ile-OH (20 mol %)	66
5	IMes·HCl (10 mol %)	0
6	(*rac*)-BINAP (10 mol %)	97

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), 2.5 mol % of $Pd_2(dba)_3$, ligand, dioxane (1 mL), 80 °C, air, 2 h. [b] ^1H NMR yield using CH_2Br_2 as internal standard.1,10-phen = 1,10-Phenanthroline; bpy = 2,2'-Bipyridine; py = Pyridine; IMes·HCl = 1,3-Bis(2,4,6-trimethylphenyl)imidazolium chloride.

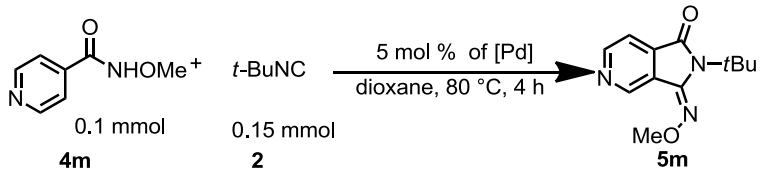
Table S5. Screening of Bases[a,b]



Entry	Base	Yield (%)	Entry	Base	Yield (%)
1	Li_2CO_3	86	9	KOAc	82
2	Na_2CO_3	94	10	$K_3PO_4·3H_2O$	34
3	K_2CO_3	92	11	$K_2HPO_4·3H_2O$	86
4	Cs_2CO_3	24	12	KH_2PO_4	94
5	$NaHCO_3$	84	13	LiCl	0
6	$KHCO_3$	82	14	NaCl	96
7	$NaOAc·H_2O$	58	15	NaTFA	0
8	NaOAc	91	16	NaOTf	0

[a] Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), 2.5 mol % of $Pd_2(dba)_3$, Base (0.2 mmol), dioxane (1 mL), 80 °C, air, 2 h. [b] ^1H NMR yield using CH_2Br_2 as internal standard.

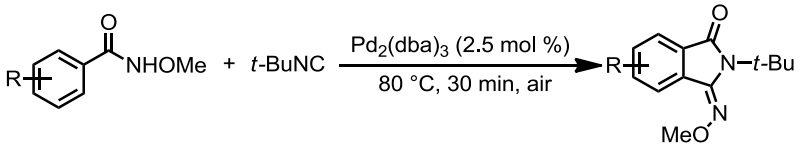
Table S6. Evaluation of Pd(II) with Heteroarene **4m**[a]



Entry	[Pd] (5 mol %)	Yield (%)[b]
1	$PdCl_2$	NR
2	$Pd(TFA)_2$	NR
3	$Pd(OTf)_2(CH_3CN)_4$	NR
4	$Pd(OAc)_2$	40

[a] Reaction conditions: **4m** (0.1 mmol), **2** (0.15 mmol), [Pd] (5 mol %), dioxane (1 mL), 80 °C, air, 4 h. [b] ^{1}H NMR yield using CH_2Br_2 as internal standard.

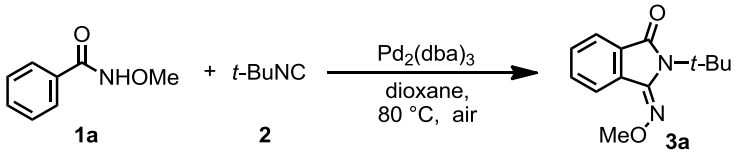
2.3 General Procedure for Directed C–H Functionalizations of Arenes



General Procedure A: A 25 mL round-bottom flask connected to a reflux condenser was charged with substrate (0.10 mmol, 1.0 equiv), *t*-BuNC **2** (0.15 mmol, 1.5 equiv), $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol, 2.5 mol%), and dioxane (1.0 mL) under air. The reaction flask was then submerged into an oil bath pre-heated to 80 °C. The reaction was stirred for 0.5–11 h and then cooled to room temperature. The solvent was removed under reduced pressure, and the resulting residue was purified by preparative TLC using hexane/EtOAc as the eluent to afford the product.

General Procedure B: A 15 mL sealed tube (with a Teflon cap) equipped with a magnetic stir bar was charged with substrate (0.10 mmol, 1.0 equiv), t-BuNC **2** (0.15 mmol, 1.5 equiv), $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol, 2.5mol %), and dioxane (1.0 mL) under air. The tube was then

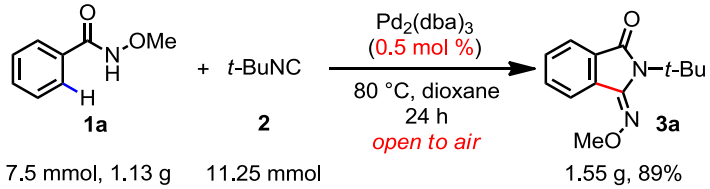
capped and submerged into a pre-heated 80 °C oil bath and stirred for 0.5 to 11 h. After cooled to rt, the solvent was removed under reduced pressure. The resulting residue was purified by preparative TLC using hexane/EtOAc as the eluent to afford the product.



Procedure	yield (%)
A	88
B	90

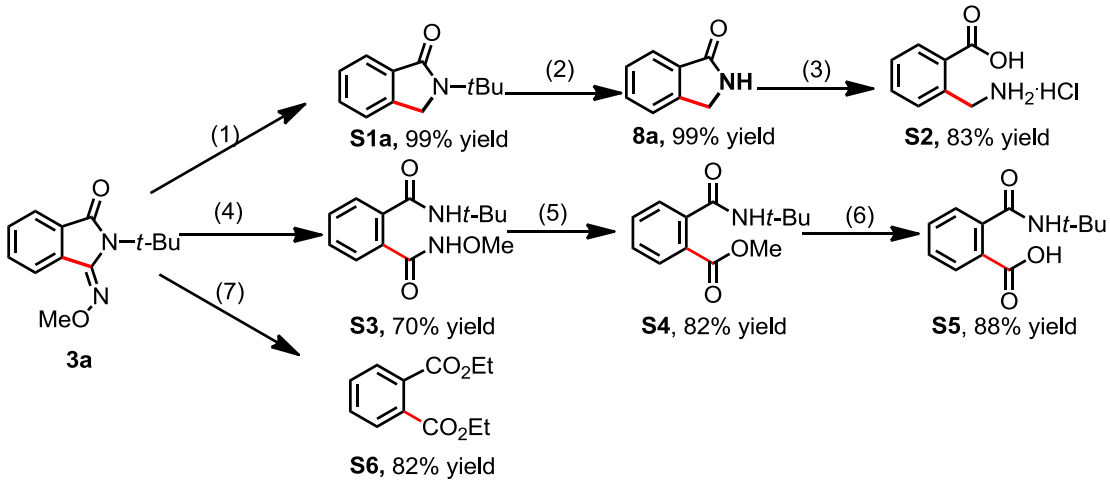
Substrate **1a** followed general procedure **A** and **B**, other substrates followed general procedure **B.**

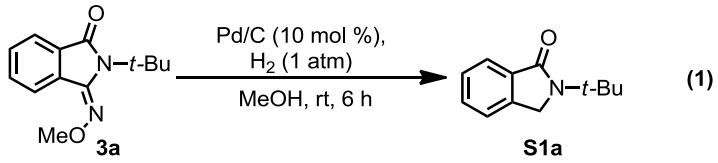
2.4 Gram-scale Reaction with 0.5 mol % $Pd_2(dba)_3$



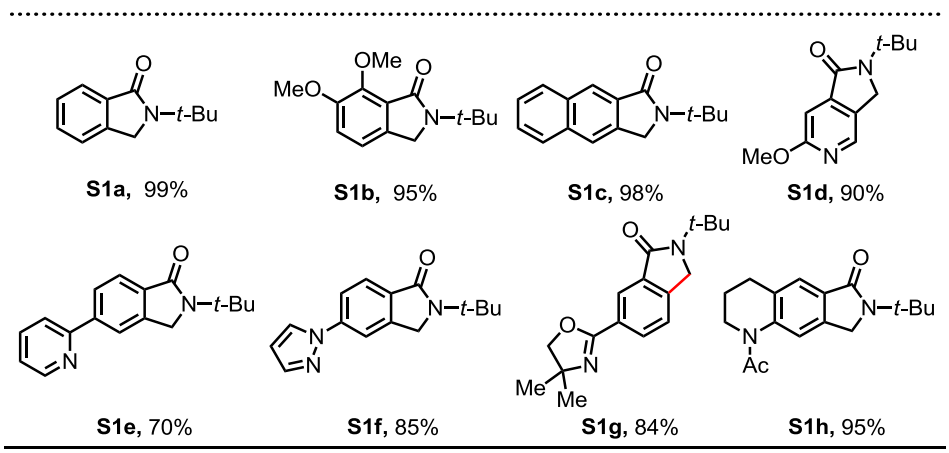
A 250 mL round flask equipped with a magnetic stir bar and condenser was charged with N-methoxybenzamide (1.13 g, 7.5 mmol, 1.0 equiv), $Pd_2(dba)_3$ (34.3 mg, 0.0375 mmol, 0.5 mmol%), *t*-BuNC **2** (11.25 mmol, 1.35 mL, 1.5 equiv), and dioxane (75 mL). The flask was then submerged into a pre-heated 80 °C oil bath and stirred for 24 h. Upon completion, the reaction vessel was cooled to rt and the solvent was removed under reduced pressure. The resulting residue was purified by chromatography on silica gel using hexane/EtOAc as the eluent to afford the product **(**1.55 g, 89% yield) as a white amorphous solid.

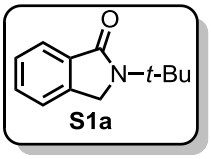
2.5 Transformation of Product





To a 25 mL reaction vessel, **3a** (46.4 mg, 0.2 mmol) and Pd/C (0.02 mmol) were dissolved into MeOH (4.0 mL). The reaction vessel was evacuated and backfilled with H_2 (×3) and the mixture was stirred at room temperature for 6 h. Upon completion, the mixture was filtered through a short silica gel-packed column and washed with MeOH. The solvents were removed under reduced pressure and **S1a** was obtained in 99% yield (38 mg).

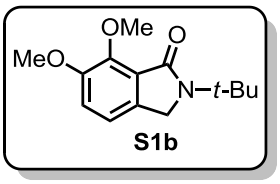




S1a

2-(tert-butyl)isoindolin-1-one(S1a)[10]

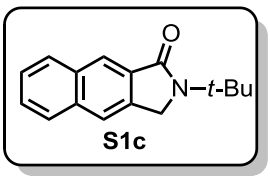
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.44-7.40 (m, 2H), 4.45 (s, 2H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.92, 140.73, 134.46, 130.92, 127.85, 123.13, 122.39, 54.38, 48.55, 28.08.



S1b

2-(tert-butyl)-6,7-dimethoxyisoindolin-1-one (S1b)

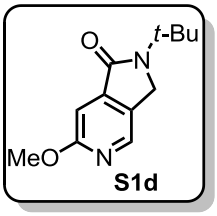
The reaction was carried out under 80 atm of H_2 pressure at 80 °C for 24 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.0 Hz, 1H), δ 7.04 (d, *J* = 8.0 Hz, 1H), 4.35 (s, 2H), 4.07 (s, 3H), 3.88 (s, 3H), 1.55 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.19, 152.31, 146.86, 134.39, 126.77, 117.57, 116.28, 62.51, 56.91, 54.46, 47.56, 28.07; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{20}NO_3$ $(M+H)^+$: 250.1438, found: 250.1439.



S1c

2-(tert-butyl)-2,3-dihydro-1H-benzo[f]isoindol-1-one (S1c)

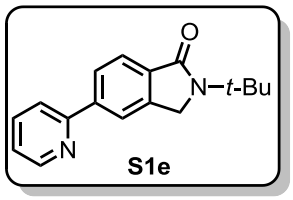
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (s, 1H), 8.00 (d, *J* = 8.0 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.83 (s, 1H), 7.57-7.50 (m, 2H), 4.62 (s, 2H), 1.62 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.77, 136.07, 135.04, 133.06, 132.62, 129.64, 128.01, 127.38, 126.15, 123.22, 121.17, 54.77, 48.42, 28.17; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{18}N_3O_2$ $(M+H)^+$: 240.1383, found: 240.1389.



S1d

2-(tert-butyl)-6-methoxy-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (S1d)

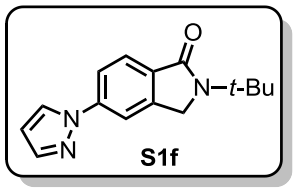
The reaction was carried out under 50 atm of H_2 pressure at rt for 24 h. 90% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.29 (s, 1H), 7.09 (s, 1H), 4.49 (s, 2H), 3.97 (s, 3H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.83, 164.43, 145.48, 141.31, 127.89, 104.01, 55.09, 54.24, 46.80, 28.02; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{17}N_2O_2$ $(M+H)^+$: 221.1285, found: 221.1292.



S1e

2-(tert-butyl)-5-(pyridin-2-yl)isoindolin-1-one (S1e)[13]

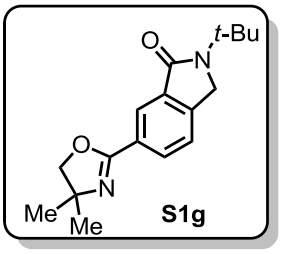
A mixture of **7a** (30.9 mg, 0.1 mmol) and HCO_2NH_4 (126 mg, 2 mmol) in 3 mL of MeOH was stirred under reflux conditions until starting material disappeared. After cooled to room temperature, the solvent was removed under reduced pressure. The resulting mixture was purified by preparative TLC using Hexane/EtOAc as the eluent to afford product **S1e** (22.6 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 4.4 Hz, 1H), 8.10 (s, 1H), 8.02 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.82-7.76 (m, 2H), 7.30-7.28 (m, 1H), 4.53 (s, 2H), 1.60 (s, 9H); ^{13}C NMR (100 MHz, CDCl3) δ 168.72, 156.88, 149.97, 142.27, 141.42, 137.10, 135.03, 126.88, 123.56, 122.81, 121.17, 54.64, 48.70, 28.20; **HRMS** (ESI-TOF) m/z Calcd for $C_{17}H_{19}N_2O$ $(M+H)^+$: 267.1492, found: 267.1503.



S1f

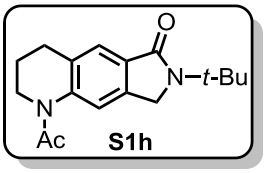
2-(tert-butyl)-5-(1H-pyrazol-1-yl)isoindolin-1-one (S1f)

The reaction was carried out under 100 atm of H_2 pressure at 80 °C for 24 h. 85% yield; **^{1}H NMR** (400 MHz, $CDCl_3$)) δ 7.99 (dd, *J* = 2.4, 0.4 Hz, 1H), 7.85-7.83 (m, 2H), 7.75 (d, *J* = 1.6 Hz, 1H), 7.73-7.70 (m, 1H), 6.51 (dd, *J* = 2.4, 1.6 Hz, 1H), 4.50 (s, 2H), 1.59 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.19, 142.37, 141.76, 132.50, 127.13, 124.41, 118.71, 113.33, 108.36, 54.65, 48.52, 28.14; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{18}N_3O$ $(M+H)^+$: 256.1444, found: 256.1449.



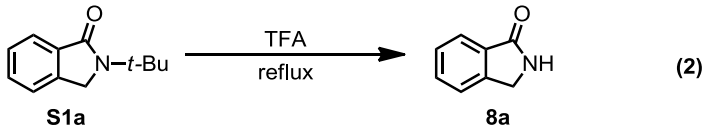
2-(tert-butyl)-6-(4,4-dimethyl-4,5-dihydrooxazol-2-yl)isoindolin-1-one (S1g)

The reaction was carried out under 50 atm of H_2 pressure at rt for 24 h. 84% yield; **^{1}H NMR** (400 MHz, $CDCl_3$)) δ 8.29 (s, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 4.49 (s, 2H), 4.13 (s, 2H), 1.57 (s, 9H), 1.39 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.13, 161.72, 143.53, 134.85, 131.05, 128.31, 123.22, 122.42, 79.35, 67.83, 54.58, 48.59, 28.52, 28.15; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{23}N_2O_2$ $(M+H)^+$: 287.1754, found: 287.1758.



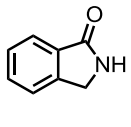
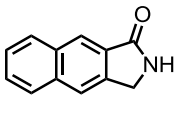
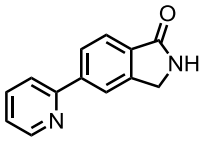
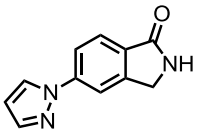
1-acetyl-7-(tert-butyl)-3,4,7,8-tetrahydro-1H-pyrrolo[3,4-g]quinolin-6(2H)-one (S1h)

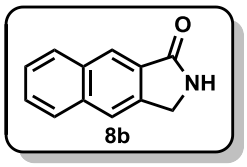
The reaction was carried out under 80 atm of H_2 pressure at 80 °C for 24 h. 90% yield; **^{1}H NMR** (400 MHz, $CDCl_3$)) δ 7.56 (s, 1H), 7.36 (s, 1H), 4.45 (s, 3H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.79 (t, *J* = 6.4 Hz, 2H), 2.27 (s, 3H), 2.02-1.95 (m, 2H), 1.57 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 170.23, 168.52, 141.69, 138.83, 131.24, 122.90, 118.72, 54.53, 48.35, 43.85, 28.15, 27.39, 24.12, 23.50; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{23}N_2O_2$ $(M+H)^+$: 287.1754, found: 287.1759.



(2)

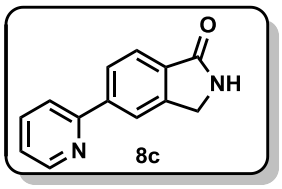
Compound **S1a** (18.9 mg, 0.1 mmol) was dissolved in trifluoroacetic acid and refluxed until the starting material was consumed indicated by TLC. The trifluoroacetic acid was then removed under reduced pressure and the residue was purified by column chromatography (hexanes/EtOAc) to afford **8a** (13.3 mg, 99%)[10]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (br, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 8.0 Hz, 1H), 7.51-7.47 (m, 2H), 4.48 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.41, 143.80, 132.24, 131.87, 128.12, 123.82, 123.29, 45.93.

			
8a, 99%	**8b**, 83%	**8c**, 91%	**8d**, 85%



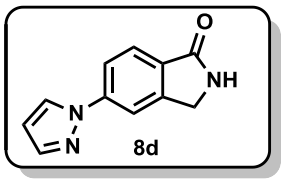
2,3-dihydro-1H-benzo[f]isoindol-1-one (8b)

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.68 (br, 1H), 8.31 (s, 1H), 8.14 (dd, *J* = 8.2 Hz, *J* = 0.4 Hz, 1H), 8.06 (s, 1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.64-7.55 (m, 2H), 4.53 (s, 2H); **^{13}C NMR** (150 MHz, DMSO-d_6) δ 169.60, 139.14, 134.67, 132.37, 130.98, 129.32, 127.94, 127.41, 126.05, 122.75, 122.14, 44.58; HRMS (ESI-TOF) m/z Calcd for $C_{12}H_{10}NO$ $(M+H)^+$: 184.0757, found: 184.0753.



5-(pyridin-2-yl)isoindolin-1-one (8c)

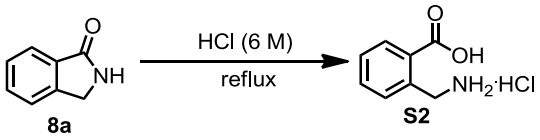
^{1}H NMR (400 MHz, $CDCl_3$)) δ 8.76 (d, *J* = 4.8 Hz, 1H), 8.17 (s, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.88-7.81 (m, 2H), 7.36-7.33 (m, 1H), 6.90 (br, 1H), 4.55 (s, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 171.47, 156.48, 149.72, 144.40, 142.80, 137.58, 132.54, 127.21, 124.35, 123.16, 122.15, 121.54, 45.86; HRMS (ESI-TOF) m/z Calcd for $C_{13}H_{11}N_2O$ $(M+H)^+$: 211.0866, found: 211.0871.



5-(1H-pyrazol-1-yl)isoindolin-1-one (8d)

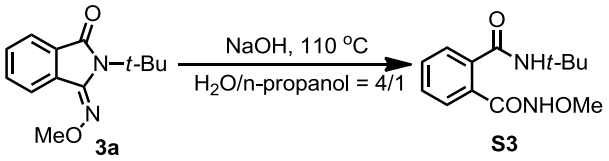
^{1}H NMR (400 MHz, DMSO-d_6)) δ 8.61 (d, *J* = 2.4 Hz, 1H), 8.06 (d, *J* = 1.2 Hz, 1H), 7.98 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.81 (d, *J* = 1.6 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 6.59 (dd, *J* = 2.4, 1.6 Hz, 1H), 4.44 (s, 2H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 169.23, 145.81, 141.99, 141.65, 130.20,

128.28, 124.06, 118.02, 113.40, 108.48, 44.89; HRMS (ESI-TOF) m/z Calcd for $C_{11}H_{10}N_3O$ $(M+H)^+$: 200.0818, found: 200.0821.



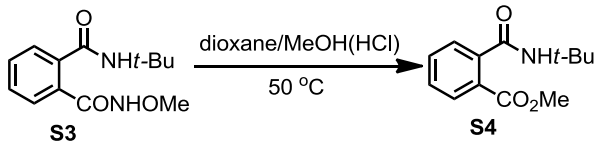
8a S2 (3)

Compound **8a** (13.3 mg, 0.1 mmol) was diluted in 6 M HCl (6 mL) and refluxed overnight. After cooled to room temperature, the mixture was washed with dichloromethane (10 mL x 3). The aqueous phase was concentrated, filtered and washed with ethanol to give **S2** as a white solid (15.5 mg, 83%).[11] **^{1}H NMR** (400 MHz, D_2O) δ 8.05 (d, *J* = 8.0 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 4.34 (s, 2H).



3a S3 (4)

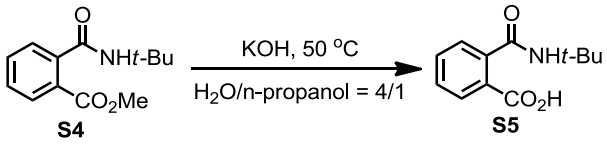
A mixture of **3a** (23.2 mg, 0.1 mmol) and NaOH (16 mg, 0.4 mmol) in 1 mL of water and 0.25 mL of n-propanol was heated to 100 °C until starting material disappeared. After cooled to room temperature, the solvents were removed under reduced pressure. The resulting mixture was purified by preparative TLC using DCM/MeOH as the eluent to afford product **S3** (17.5 mg, 70%).[12] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.2 Hz, 1H), 7.57-7.51 (m, 3H), 3.87 (s, 3H), 1.09 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 169.15, 155.02, 132.54, 131.71, 131.67, 130.27, 129.95, 128.56, 61.45, 53.07, 31.40; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{19}N_2O_3$ $(M+H)^+$: 251.1390, found: 251.1395.



S3 S4 (5)

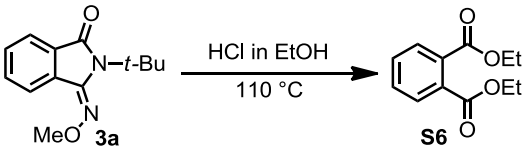
To a solution of substrate **S3** (12.5 mg , 0.05mmol) in 9 mL of dioxane was added 0.5 mL of a 10 M solution of anhydrous hydrochloric acid in methanol at room temperature. The mixture was stirred at 50 °C for 3 h. After cooling to room temperature, 5 mL of saturated $NaHCO_3$ (aq.) was added. Then, the organic phase was extracted with EtOAc and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by preparative

TLC, affording product **S4** (9.6 mg, 82%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88-7.86 (m, 1H), 7.53-7.49 (m, 1H), 7.47-7.42 (m, 2H), 5.60 (br, 1H), 3.89 (s, 3H), 1.47 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.80, 167.43, 139.47, 132.01, 130.25, 129.38, 129.00, 127.58, 52.52, 52.04, 28.85; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{18}NO_3$ $(M+H)^+$: 236.1281, found: 236.1285.



(6)

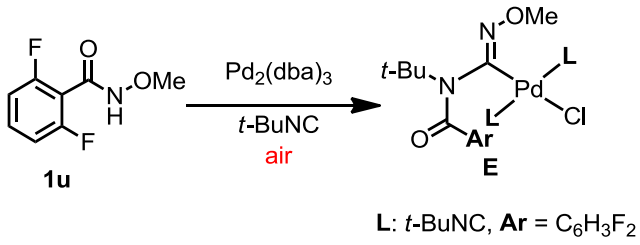
A mixture of **S4** (23.5 mg, 0.1 mmol) and KOH (82%) (40 mg, 0.6 mmol) in 2 mL of water and 0.5 mL of n-propanol was stirred at 50 °C until starting material disappeared. After cooled to room temperature, the mixture was washed with DCM (5 mL) for three times. Then the aqueous phase was acidified to pH 3 with 2 N HCl and extracted with DCM (10 mL × 3). The combined organic phases were dried over anhydrous Na_2SO_4, concentrated under reduced pressure to afford product **S5** (19.6 mg, 88%). ^{1}H NMR (400 MHz, CD_3OD) δ 7.94 (d, *J* = 8.0 Hz, 1H), 7.91 (br, 1H), 7.58 (td, *J* = 7.6, 1.2 Hz, 1H), 7.49 (td, *J* = 7.6, 1.2 Hz, 1H), 7.38 (dd, *J* = 7.6, 0.8 Hz, 1H), 1.43 (s, 9H); **^{13}C NMR** (100 MHz, CD_3OD) δ 172.39, 169.35, 141.09, 133.02, 131.25, 130.34, 130.03, 128.85, 52.59, 28.77; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{16}NO_3$ $(M+H)^+$: 222.1125, found: 222.1127.



(7)

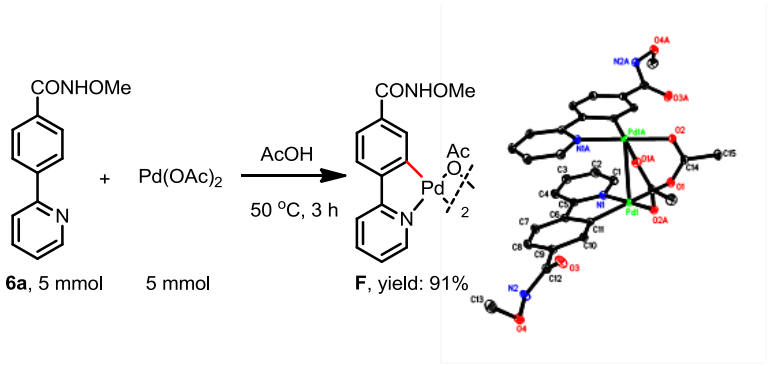
A 25 mL sealed vessel was charged with **3a** (46.4 mg, 0.2 mmol) which was dissolved in 2 mL of a saturated solution of anhydrous hydrochloric acid in ethanol at room temperature. Then, the mixture was heated to 110 °C for 12 h. After the mixture cooled to room temperature, the solution was diluted with water (10 mL) and extracted with EtOAc (10 mL × 3). The combined organic phases were washed with brine, dried over anhydrous $MgSO_4$ and evaporated. The residue was purified by silica gel column chromatography with hexane and EtOAc to afford product **S6** in 82% yield (36.4mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 6.0, 3.6 Hz, 2H), 7.53 (dd, *J* = 6.0, 3.6 Hz, 2H), 4.37 (q, *J* = 7.2 Hz, 4H), 1.37 (t, *J* = 7.2 Hz, 6H).

2.6 The Synthesis of Pd-complex E



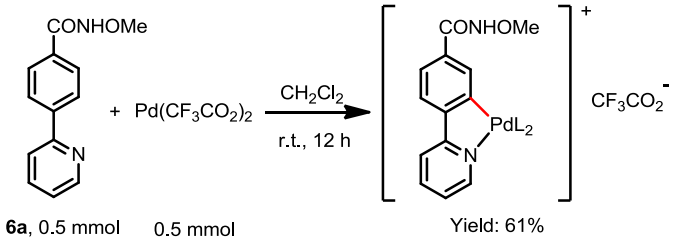
A 50 mL vessel equipped with a magnetic stir bar was charged with substrate **1u** (0.2 mmol, 37.5 mg, 1.0 equiv.), $Pd_2(dba)_3$ (46 mg, 0.05 mmol, 0.25 equiv.), *t*-Butylisocyanide (0.30 mmol, 36 μL, 1.5 equiv.), dioxane (2 mL). The mixture was then submerged into a pre-heated 80 °C oil bath for 12 h. The reaction vessel was then cooled to rt, filtrated and concentrated *in vacuo*. The resulting residue was purified by preparative TLC using hexanes/EtOAc as the eluent to afford the product **E** (15 mg, 26% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.26 (m, 1H), 6.89 (dd, *J* = 8.4, 7.2 Hz, 2H), 3.67 (s, 3H), 1.68 (s, 9H), 1.49 (s, 18H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 162.13, 158.58 (dd, *J* = 247.1, 7.5 Hz), 148.38, 129.92 (t, *J* = 9.6 Hz), 127.91 (t, *J* = 17.2 Hz), 118.77 (t, *J* = 23.1 Hz), 111.69 (dd, *J* = 20.7, 4.0 Hz), 61.76, 61.03, 58.23, 30.00, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{23}H_{33}F_2N_4O_2Pd$ $(M\text{-}Cl)^+$: 537.1622, found: 537.1642.

2.7 The Reactions of 6a with $Pd(OAc)_2$ and $Pd(TFA)_2$

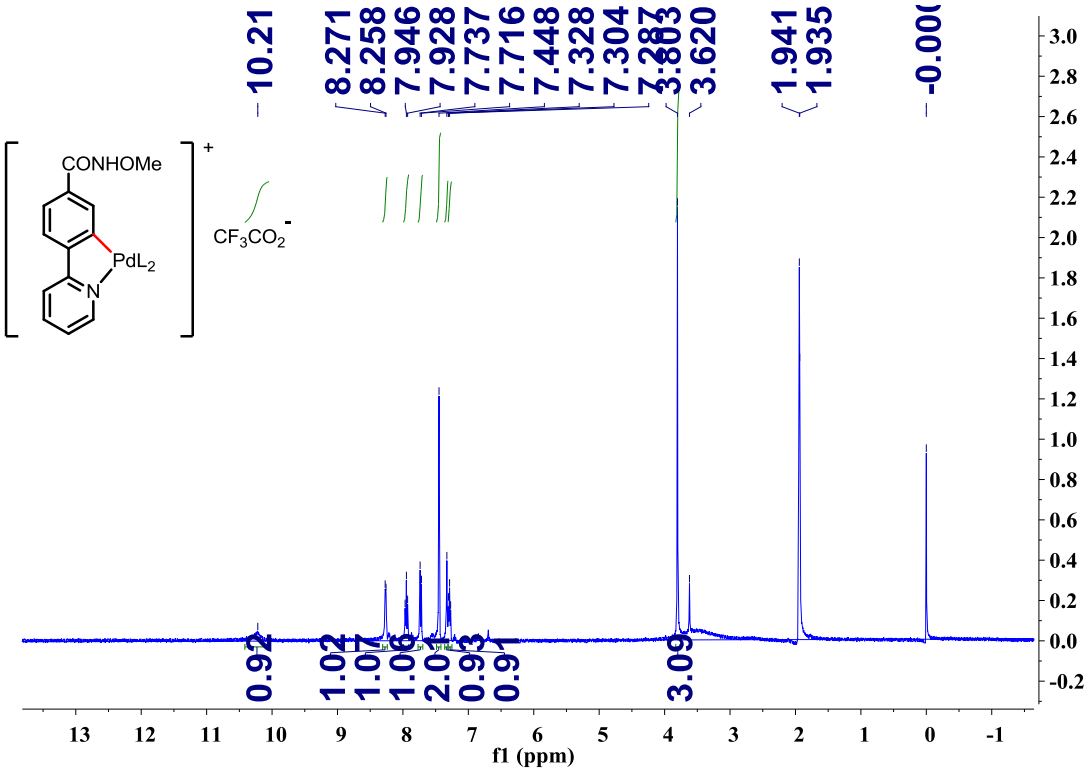


A 250 mL vessel equipped with a magnetic stir bar was charged with substrate **6a** (1.14g, 5.0 mmol, 1.0 equiv.), $Pd(OAc)_2$ (1.12 g, 5.0 mmol, 1.0 equiv.), AcOH (60 mL). The mixture was then submerged into a pre-heated 50 °C oil bath for 3 h. The reaction vessel was then cooled to rt and concentrated in vacuo. The solid residue was washed with CH_2Cl_2 (20 mL) and Et_2O (20 mL) to afford the product **F** as yellow solid (1.78 g, 91% yield). **^{1}H NMR** (400 MHz, Acetic acid-d_4)

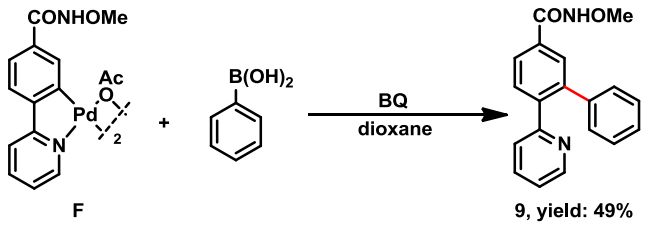
δ 7.97 (d, *J* = 5.6 Hz, 1H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 7.10 (s, 1H), 7.09 (d, *J* = 8.0 Hz, 1H), 6.87 (t, *J* = 6.6 Hz, 1H), 3.86 (s, 3H), 2.07-2.06 (m, 6H).



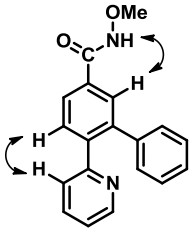
To a mixture of *N*-methoxy-4-(pyridin-2-yl)benzamide (114 mg, 0.5 mmol,) and $Pd(CF_3CO_2)_2$ (166 mg, 0.5 mmol) was added CH_2Cl_2 (25 mL). Then the mixture was stirred at room temperature overnight. After reaction, the solvent was evaporated in vacuo and the solid residue was washed with CH_2Cl_2 (10 mL) to afford 135 mg of the title compound as a yellow solid (61% yield). **^{1}H NMR** (400 MHz, CD_3CN) δ 10.22 (brs, 1H), 8.26 (d, *J* = 5.2 Hz, 1H), 7.95 (t, *J* = 7.8 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.45 (s, 2H), 7.33 (s, 1H), 7.29 (t, *J* = 6.6 Hz, 1H), 3.80 (s, 3H).



2.8 The Transformation of Pd-complex F

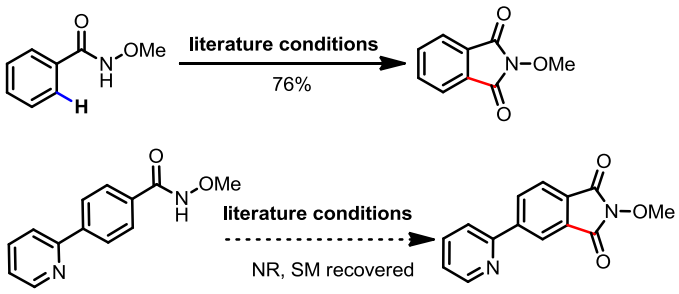


To a mixture of complex (16.6 mg, 0.021 mmol) and BQ (5.4 mg, 0.05 mmol)in dioxane (0.5 mL) in a pressure tube is added $PhB(OH)_2$ (18.3 mg, 0.15 mmol). The reaction mixture is then heated at 100 °C for 3 hours and then at 80 °C overnight. The reaction mixture is cooled to 23 °C and the solvent is removed in vacuo. The crude product is purified by chromatography on silica gel eluting with EtOAc to afford 6.2 mg of desired compound **9** (49% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.65 (br, 1H), 8.65 (d, *J* = 4.0 Hz, 1H), 7.81 (s, 1H), 7.74 (d, *J* = 7.8 Hz, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.23-7.22 (m, 3H), 7.17-7.11 (m, 3H), 6.87 (d, *J* = 7.6 Hz, 1H), 3.89 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 166.21, 158.05, 149.48, 142.60, 141.15, 140.10, 135.57, 132.02, 130.89, 129.55, 129.42, 128.26, 127.25, 125.99, 125.42, 122.01, 64.63; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{19}H_{17}N_2O_2$ $(M+H)^+$: 305.1285, found: 305.1285.
NOESY (600 MHz, $CDCl_3$).



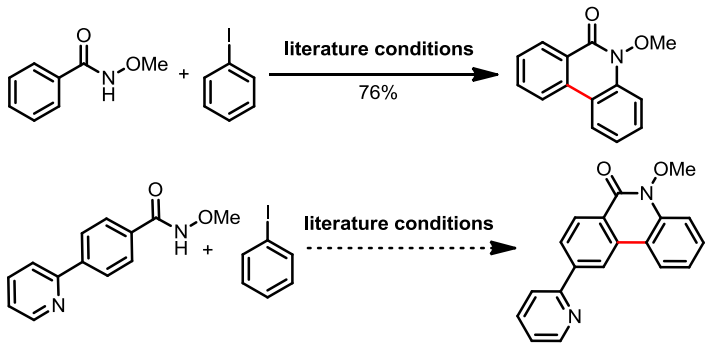
2.9 The Effect of Heterocycle in Previous Reported C–H Activation Reactions Using N-methoxyamide Directing Group

Org. Lett. **13**, 5326-5329 (2011):



conditions: $Pd(OAc)_2$ (5 mol %), BQ (2 equiv), AcOH, 110 ^oC, 8-18 h, 1 atm CO.

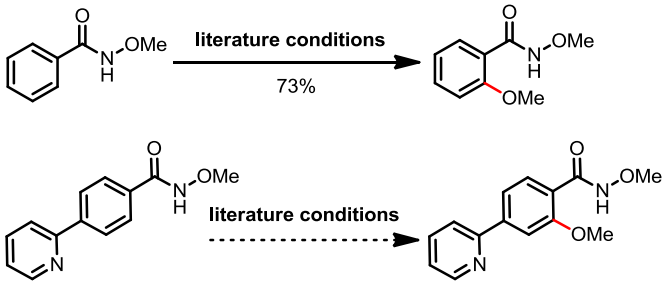
Angew. Chem. Int. Ed. **50**, 1380–1383 (2011):



No desired product.
-CONHOMe in SM was decomposed to $-CO_2CH_3$.

conditions: $Pd(OAc)_2$ (5 mol %), Ag_2O (2 equiv), AcOH, 120 ^oC, 13-36 h.

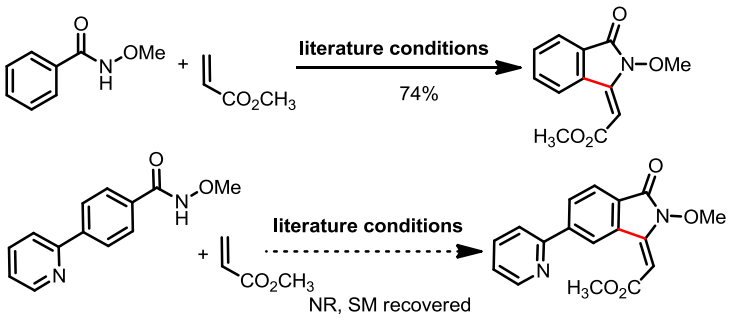
J. Org. Chem. **75**, 476–479 (2010):



No desired product.
-CONHOMe in SM was decomposed to $-CO_2CH_3$.

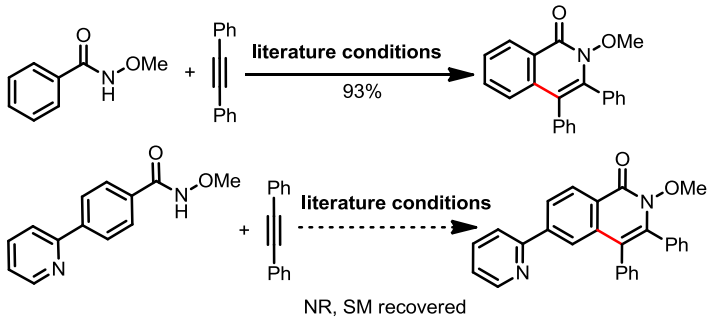
conditions: $Pd(OAc)_2$ (5 mol %), $K_2S_2O_8$, 4 A MS, MeOH/dioxane (1:1), 55 ^oC, 6.5 h.

Chem. Commun. **47**, 12789–12791 (2011):



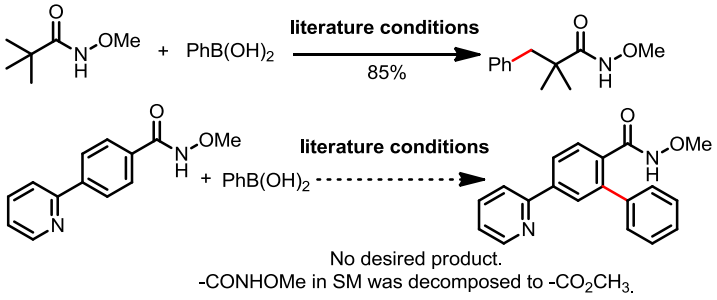
conditions: $Pd(OAc)_2$ (5 mol %), BQ (2 equiv), AcOH, 100 °C, 10 h.

Chem. Commun. **48**, 3236–3238 (2012):



conditions: $Pd(OAc)_2$ (10 mol %), NaI (1 equiv), DMF, 120 °C, 10-18 h.

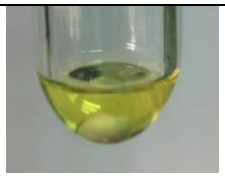
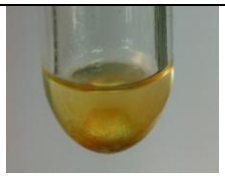
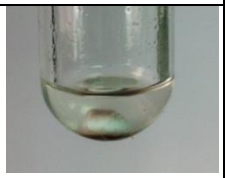
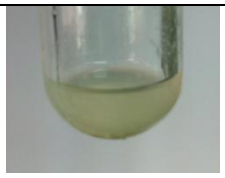
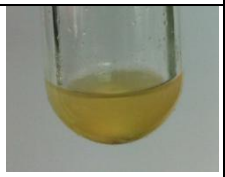
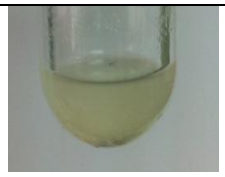
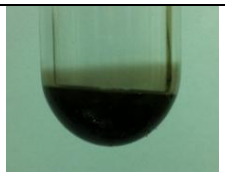
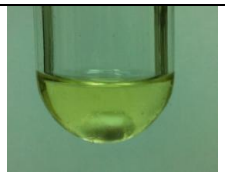
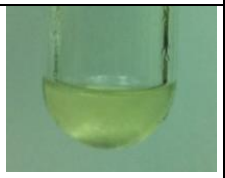
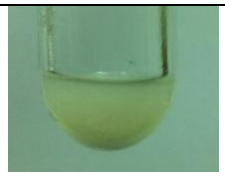
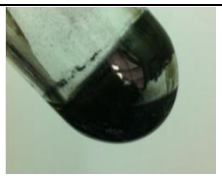
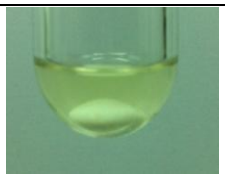
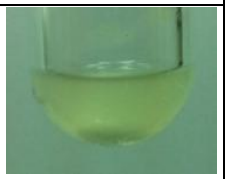
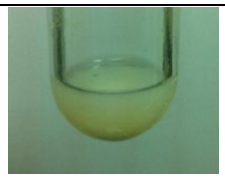
J. Am. Chem. Soc. **130**, 7190–7191 (2008):



conditions: $Pd(OAc)_2$ (10 mol %), Ag_2O (2.0 equiv), BQ (0.5 equiv), K_2CO_3 (2 equiv), *t*-BuOH/DMF, 70 °C, 18 h.

2.10 Reduction of Pd(II) Species in Isocyanides: Experimental Procedure and Photos

A 25 mL sealed tube equipped with a magnetic stir bar was charged with palladium salt (0.05 mmol), *t*-BuNC (24 μL, 0.2 mmol), dioxane (1.5 mL) under Ar atmosphere. The mixture was stirred at room temperature for 15 mins and then the tube was submerged into a pre-heated 80 °C oil bath. The pictures were taken at 0, 15 min at room temperature and 30, 180 min at 80 °C respectively. These photos show that $Pd(OAc)_2$ can be readily reduced to Pd(0) while $PdCl_2$, $Pd(TFA)_2$, $Pd(OTf)_2$ remains as homogeneous Pd(II) species under the reactions conditions.

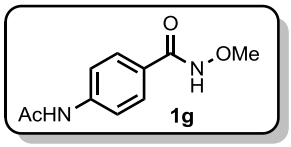
entry	$Pd(OAc)_2$	$PdCl_2$	$Pd(TFA)_2$	$Pd(OTf)_2(MeCN)_4$
1				
2				
3				
4				

Conditions: 0.05 mmol Pd, 0.2 mmol *t*-BuNC in 1.5 mL dioxane.

entry1: 0 min at r.t. entry 2: 15 min at r.t. entry 3: 30 min at 80 ^{o}C. entry 4: 180 min at 80 ^{o}C.

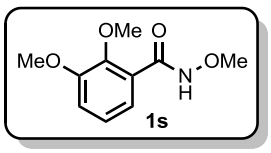
3. Analytical Data

3.1Characterization of Substrates



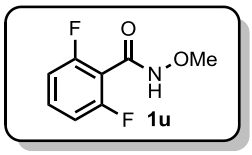
4-acetamido-N-methoxybenzamide (1g)

Following typical procedure 3: **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.62 (s, 1H), 10.18 (s, 1H), 7.70 (d, *J* = 8.8 Hz, 2H), 7.65 (d, *J* = 8.8 Hz, 2H), 3.69 (s, 3H), 2.07 (s, 3H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 168.79, 163.84, 142.30, 127.95, 126.42, 118.22, 63.27, 24.16; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{10}H_{13}N_2O_3$ $(M+H)^+$: 209.0921, found: 209.0925.



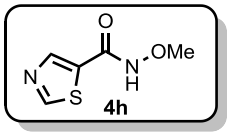
N,2,3-trimethoxybenzamide (1s)

Following typical procedure 1: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.29 (br, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.18 (t, *J* = 8.0 Hz, 1H), 7.07 (dd, *J* = 8.0, 1.6 Hz, 1H), 3.93 (s, 3H), 3.90 (s, 3H), 3.89 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.55, 152.17, 146.68, 124.63, 124.34, 121.95, 115.49, 64.00, 61.09, 55.75; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{10}H_{14}NO_4$ $(M+H)^+$: 212.0923, found: 212.0922.



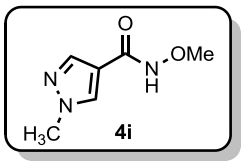
2,6-difluoro-N-methoxybenzamide (1u)

Following typical procedure 1: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.61 (br, 1H), 7.45-7.38 (m, 1H), 6.97 (t, *J* = 8.0 Hz, 2H), 3.92 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.67 (dd, *J* = 251.5, 6.7 Hz), 158.05, 132.10 (t, *J* = 10.1 Hz), 111.56-111.34 (m), 110.94 (t, *J* = 20.8 Hz), 63.76; **HRMS** (ESI-TOF) *m/z* Calcd for $C_8H_8F_2NO_2$ $(M+H)^+$: 188.0518, found: 188.0519.



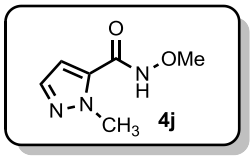
N-methoxythiazole-5-carboxamide (4h)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.92 (br, 1H), 9.07 (s, 1H), 8.52 (s, 1H), 3.88 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.10, 157.39, 143.96, 131.57, 64.34; **HRMS** (ESI-TOF) *m/z* Calcd for $C_5H_7N_2O_2S$ $(M+H)^+$: 159.0223, found: 159.0225.



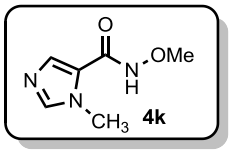
N-methoxy-1-methyl-1H-pyrazole-4-carboxamide (4i)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (br, 1H), 7.92 (s, 1H), 7.85 (s, 1H), 3.94 (s, 3H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.45, 139.16, 132.33, 115.19, 64.52, 39.27; **HRMS** (ESI-TOF) *m/z* Calcd for $C_6H_{10}N_3O_2$ $(M+H)^+$: 156.0768, found: 156.0771.



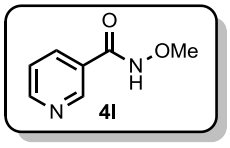
N-methoxy-1-methyl-1H-pyrazole-5-carboxamide (4j)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.65 (br, 1H), 7.48 (d, *J* = 2.0 Hz, 1H), 6.88 (d, *J* = 2.0 Hz, 1H), 4.20 (s, 3H), 3.89 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.22, 136.99, 131.90, 106.76, 63.64, 38.36; **HRMS** (ESI-TOF) *m/z* Calcd for $C_6H_{10}N_3O_2$ $(M+H)^+$: 156.0768, found: 156.0770.



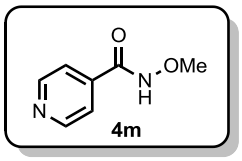
N-methoxy-1-methyl-1H-imidazole-5-carboxamide (4k)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.81 (br, 1H), 7.61 (s, 1H), 7.59 (s, 1H), 3.95 (s, 3H), 3.85 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.51, 141.51, 131.68, 123.81, 64.47, 34.18; **HRMS** (ESI-TOF) *m/z* Calcd for $C_6H_{10}N_3O_2$ $(M+H)^+$: 156.0768, found: 156.0770.



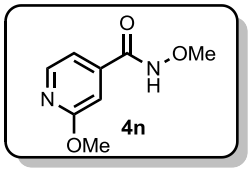
N-methoxynicotinamide (4l)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.94 (br, 1H), 9.02 (s, 1H), 8.65 (d, *J* = 4.8 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 1H), 7.40 (dd, *J* = 7.6, 4.8 Hz, 1H), 3.85 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.71, 151.79, 147.79, 135.61, 128.09, 123.64, 64.03; **HRMS** (ESI-TOF) *m/z* Calcd for $C_7H_9N_2O_2$ $(M+H)^+$: 153.0659, found: 153.0652.



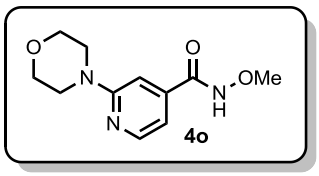
N-methoxyisonicotinamide (4m)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.41(br, 1H), 8.66 (d, *J* = 5.6 Hz, 2H), 7.72 (d, *J* = 5.6 Hz, 2H), 3.87 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.70, 149.76, 139.59, 121.35, 63.91; **HRMS** (ESI-TOF) *m/z* Calcd for $C_7H_9N_2O_2$ $(M+H)^+$: 153.0659, found: 153.0652.



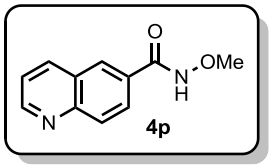
N,2-dimethoxyisonicotinamide (4n)

Following typical procedure 2:^{1}H NMR (400 MHz, $CDCl_3$) δ 10.38 (br, 1H), 8.50 (dd, *J* = 7.6, 2.0 Hz, 1H), 8.29 (dd, *J* = 4.8, 2.0 Hz, 1H), 7.08 (dd, *J* = 7.6, 5.2 Hz, 1H), 4.11 (s, 3H), 3.90 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.32, 160.08, 149.94, 141.37, 117.77, 113.97, 64.44, 54.23; **HRMS** (ESI-TOF) *m/z* Calcd for $C_8H_{11}N_2O_3$ $(M+H)^+$: 183.0764, found: 183.0771.



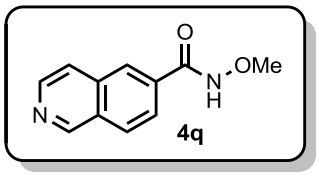
N-methoxy-2-morpholinoisonicotinamide (4o)

Following typical procedure 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.22 (br, 1H), 8.21 (d, *J* = 5.2 Hz, 1H), 7.02 (s, 1H), 6.86 (d, *J* = 3.6 Hz, 1H), 3.84 (s, 3H), 3.78 (t, *J* = 4.8, 4H), 3.52 (t, *J* = 4.8 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.04, 159.93, 148.79, 140.65, 110.06, 104.90, 66.64, 64.49, 45.37; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{16}N_3O_3$ $(M+H)^+$: 238.1186, found: 238.1177.



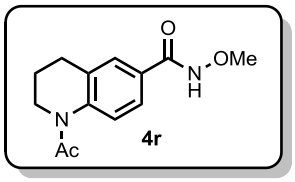
N-methoxyquinoline-6-carboxamide (4p)

Following typical procedure 3: ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.01 (br, 1H), 8.99 (s, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 8.43 (s, 1H), 8.08 (s, 2H), 7.62-7.61 (m, 1H), 3.77 (s, 3H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 163.65, 152.23, 148.73, 137.09, 130.11, 129.26, 127.99, 127.30, 127.13, 122.29, 63.34; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{11}N_2O_2$ $(M+H)^+$: 203.0815, found 203.0820.



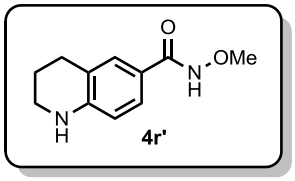
N-methoxyisoquinoline-6-carboxamide (4q)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.49 (br, 1H), 9.19 (s, 1H), 8.46 (d, *J* = 5.6 Hz, 1H), 8.30 (s, 1H), 8.00 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 5.6 Hz, 1H), 3.92 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.10, 143.26, 135.14, 133.79, 129.41, 128.14, 126.29, 125.47, 121.36, 64.24; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{11}N_2O_2$ $(M+H)^+$: 203.0815, found: 203.0814.



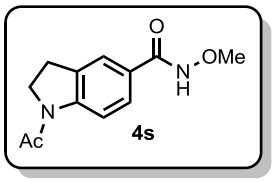
1-acetyl-N-methoxy-1,2,3,4-tetrahydroquinoline-6-carboxamide (4r)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.92 (br, 1H), 7.69 (s, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.44 (s, 1H), 3.84 (s, 3H), 3.76 (t, *J* = 5.6 Hz, 2H), 2.74 (t, *J* = 5.6 Hz, 2H), 2.26 (s, 3H), 1.95 (t, *J* = 5.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.41, 165.39, 141.56, 131.94, 128.01, 124.77, 124.23, 64.06, 44.72, 26.89, 23.58, 23.40; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{17}N_2O_3$ $(M+H)^+$: 249.1234, found: 249.1238.



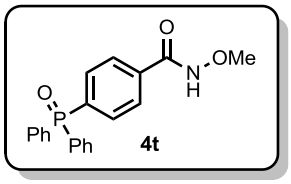
N-methoxy-1,2,3,4-tetrahydroquinoline-6-carboxamide (4r')

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 7.39 (s, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 6.40 (d, *J* = 8.4 Hz, 1H), 4.23 (brs, 1H), 3.85 (s, 3H), 3.35 (t, *J* = 5.6 Hz, 2H), 2.76 (t, *J* = 6.2 Hz, 2H), 1.95-1.89 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.24, 148.19, 128.80, 126.17, 120.42, 118.80, 112.85, 64.54, 41.67, 26.91, 21.39; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{15}N_2O_2$ $(M+H)^+$: 207.1128, found: 207.1130.



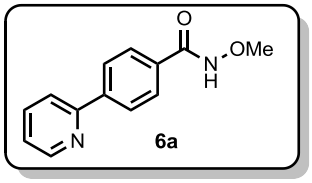
1-acetyl-N-methoxyindoline-5-carboxamide (4s)

Following typical procedure 3: ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.63 (br, 1H), 8.05 (d, *J* = 8.4 Hz, 1H), 7.61 (s, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 4.11 (t, *J* = 8.4 Hz, 2H), 3.68 (s, 3H), 3.14 (t, *J* = 8.4 Hz, 2H), 2.16 (s, 3H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 169.17, 163.91, 145.64, 132.12, 126.75, 126.70, 123.72, 114.97, 63.22, 48.49, 27.06, 24.07; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{15}N_2O_3$ $(M+H)^+$: 235.1077, found: 235.1080.



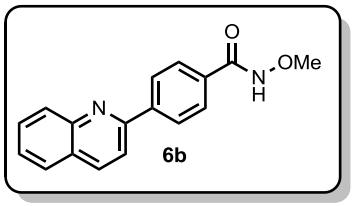
4-(diphenylphosphoryl)-N-methoxybenzamide (4t)

Following typical procedure 1: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.95 (br, 1H), 7.84 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.57-7.43 (m, 12H), 3.87 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.62, 136.23(d, *J* = 1.6 Hz, 134.85 (d, *J* = 102.1 Hz), 132.45 (d, *J* = 2.7 Hz), 131.94 (d, *J* = 11.3 Hz), 131.83 (d, *J* = 10.1 Hz), 131.09 (d, *J* = 72.6 Hz), 128.72 (d, *J* = 12.3 Hz), 127.67 (d, *J* = 12.2 Hz), 63.85; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{20}H_{19}NO_3P$ $(M+H)^+$: 352.1097, found: 352.1099.



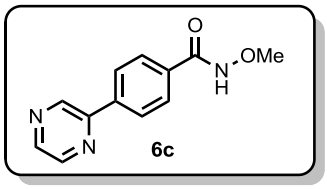
N-methoxy-4-(pyridin-2-yl)benzamide (6a)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.63 (s, 1H), 8.66 (d, *J* = 4.4 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 2H), 7.81 (d, *J* = 8.4 Hz, 2H), 7.75 (td, *J* = 7.6, 2.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.26 (dd, *J* = 6.8, 5.2 Hz, 1H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.19, 156.25, 149.67, 142.43, 137.17, 132.27, 127.76, 127.04, 122.93, 121.21, 64.34; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{13}N_2O_2$ $(M+H)^+$: 229.0972, found: 229.0979.



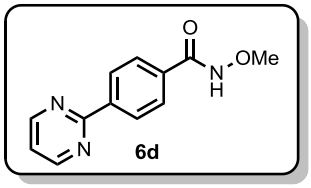
N-methoxy-4-(quinolin-2-yl)benzamide (6b)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.11 (br, 1H), 8.26-8.17 (m, 4H), 7.88-7.84 (m, 4H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 3.93 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.04, 148.37, 143.19, 137.26, 132.42, 130.14, 129.87, 127.96, 127.74, 127.68, 127.55, 126.96, 119.04, 64.87; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{15}N_2O_2$ $(M+H)^+$: 279.1128, found: 279.1130.



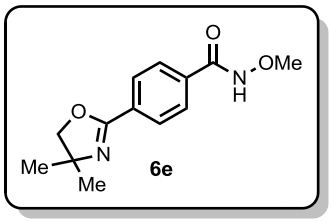
N-methoxy-4-(pyrazin-2-yl)benzamide (6c)

Following typical procedure 3: ^{1}H NMR (400 MHz, CD_3OD) δ 9.16 (d, *J* = 1.6 Hz, 1H), 8.71 (dd, *J* = 2.4, 1.6 Hz, 1H), 8.58 (d, *J* = 2.4 Hz, 1H), 8.19 (d, *J* = 8.4 Hz, 2H), 7.91 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, CD_3OD) δ 153.01, 145.84, 144.72, 143.35, 140.82, 134.36, 128.93, 128.24, 64.46; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{12}N_3O_2$ $(M+H)^+$: 230.0924, found: 230.0929.



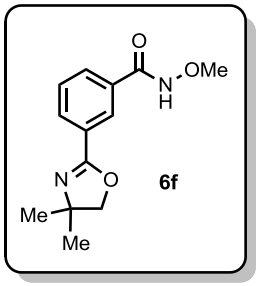
N-methoxy-4-(pyrimidin-2-yl)benzamide (6d)

Following typical procedure 3: ^{1}H NMR (400 MHz, CD_3OD) δ 8.89 (d, *J* = 4.8 Hz, 2H), 8.51 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.8 Hz, 2H), 7.40 (t, *J* = 4.8 Hz, 1H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, CD_3OD) δ 167.04, 164.33, 158.59, 141.84, 134.76, 129.18, 128.34, 121.09, 64.43; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{12}N_3O_2$ $(M+H)^+$: 230.0924, found: 230.0933.



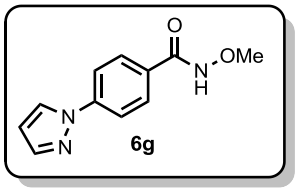
4-(4,4-dimethyl-4,5-dihydrooxazol-2-yl)-N-methoxybenzamide (6e)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.03 (br, 1H), 7.90 (d, *J* = 8.0 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 2H), 4.13 (s, 2H), 3.86 (s, 3H), 1.38 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.94, 161.60, 134.41, 131.20, 128.50, 127.29, 79.44, 67.93, 64.58, 28.44; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{17}N_2O_3$ $(M+H)^+$: 249.1234, found: 249.1238.



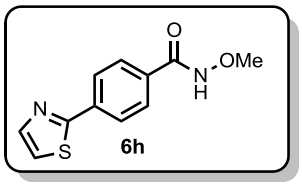
3-(4,4-dimethyl-4,5-dihydrooxazol-2-yl)-N-methoxybenzamide (6f)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.68 (br, 1H), 8.30 (s, 1H), 7.93 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.36 (t, *J* = 7.6 Hz, 1H), 4.05 (s, 2H), 3.84 (s, 3H), 1.31 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.25, 161.37, 132.04, 130.91, 130.00, 128.26, 127.72, 126.70, 78.93, 67.36, 63.76, 27.95; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{17}N_2O_3$ $(M+H)^+$: 249.1234, found: 249.1241.



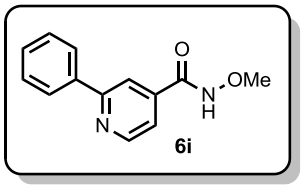
N-methoxy-4-(1H-pyrazol-1-yl)benzamide (6g)

Following typical procedure 3: ^{1}H NMR (400 MHz, acetone-d_6) δ 8.43 (dd, J = 2.4, 0.4 Hz, 1H), 7.96 (s, 4H), 7.75 (d, J = 1.2 Hz, 1H), 6.55 (dd, J = 2.4, 1.6 Hz, 1H), 3.81 (s, 3H); **^{13}C NMR** (100 MHz, acetone-d_6) δ 164.97, 143.33, 142.31, 130.90, 129.41, 128.12, 118.88, 109.03, 64.03; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{12}N_3O_2$ $(M+H)^+$: 218.0924, found: 218.0930.



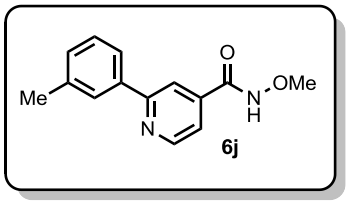
N-methoxy-4-(thiazol-2-yl)benzamide (6h)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.45 (br, 1H), 7.91 (d, J = 8.0 Hz, 2H), 7.87 (d, J = 3.2 Hz, 1H), 7.82 (d, J = 8.0 Hz, 2H), 7.38 (d, J = 3.2 Hz, 1H), 3.86 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.11, 165.84, 144.02, 136.47, 133.04, 128.05, 126.66, 120.08, 64.44; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{11}N_2O_2S$ $(M+H)^+$: 235.0536, found: 235.0540.



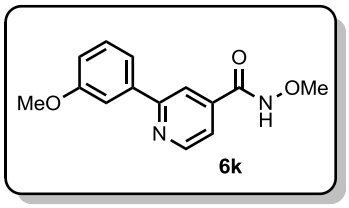
N-methoxy-5-phenylnicotinamide (6i)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.94 (br, 1H), 8.59 (d, J = 5.2 Hz, 1H), 8.05 (s, 1H), 7.85-7.83 (m, 2H), 7.51 (dd, J = 5.2, 1.6 Hz, 1H), 7.33-7.31 (m, 3H), 3.78 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.04, 158.08, 149.77, 139.88, 137.97, 129.38, 128.63, 126.75, 119.43, 118.16, 63.90; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{13}N_2O_2$ $(M+H)^+$: 229.0972, found: 229.0979.



N-methoxy-2-(m-tolyl)isonicotinamide (6j)

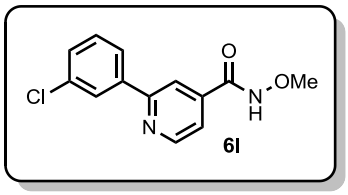
Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.48 (br, 1H), 8.59 (d, *J* = 5.2 Hz, 1H), 8.01 (s, 1H), 7.68 (s, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.46 (d, *J* = 4.4 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 7.6 Hz, 1H), 3.79 (s, 3H), 2.31 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.42, 158.52, 149.92, 140.01, 138.54, 138.12, 130.35, 128.72, 127.63, 124.04, 119.40, 118.37, 64.25, 21.42; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{15}N_2O_2$ $(M+H)^+$: 243.1128, found: 243.1134.



6k

N-methoxy-2-(3-methoxyphenyl)isonicotinamide (6k)

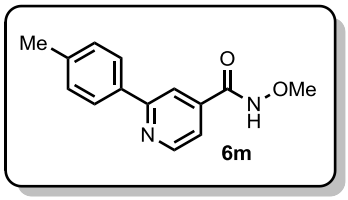
Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.30 (br, 1H), 8.60 (d, *J* = 4.8 Hz, 1H), 7.99 (s, 1H), 7.48 (d, *J* = 4.0 Hz, 1H), 7.44 (s, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 6.89 (dd, *J* = 8.0, 2.4 Hz, 1H), 3.80 (s, 3H), 3.78 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.41, 160.03, 158.07, 149.93, 140.11, 139.65, 129.87, 119.66, 119.34, 118.41, 115.36, 112.36, 64.30, 55.35; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{15}N_2O_3$ $(M+H)^+$: 259.1077, found: 259.1080.



6l

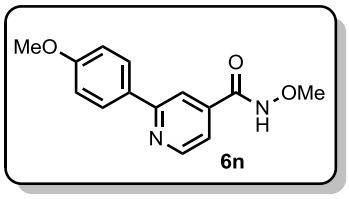
2-(3-chlorophenyl)-N-methoxyisonicotinamide (6l)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.52 (br, 1H), 8.70 (d, *J* = 5.2 Hz, 1H), 8.00 (s, 1H), 7.93 (s, 1H), 7.80 (d, *J* = 6.8 Hz, 1H), 7.52 (d, *J* = 4.4 Hz, 1H), 7.37-7.31 (m, 2H), 3.86 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.27, 156.77, 150.28, 140.13, 139.93, 134.92, 130.12, 129.55, 127.05, 125.03, 120.10, 118.25, 64.39; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{12}ClN_2O_2$ $(M+H)^+$: 263.0582, found: 263.0592.



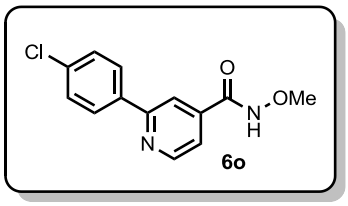
N-methoxy-2-(p-tolyl)isonicotinamide (6m)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.84 (br, 1H), 8.56 (d, *J* = 4.8 Hz, 1H), 8.01 (s, 1H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 4.8 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 2H), 3.79 (s, 3H), 2.29 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.35, 158.19, 149.71, 139.88, 139.57, 135.26, 129.43, 126.69, 119.13, 117.90, 64.00, 21.11; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{15}N_2O_2$ $(M+H)^+$: 243.1128, found: 243.1118.



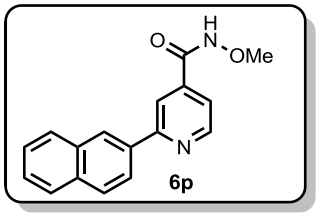
N-methoxy-2-(4-methoxyphenyl)isonicotinamide (6n)

Following typical procedure 3: ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.83 (br, 1H), 8.55 (d, *J* = 5.2 Hz, 1H), 7.98 (s, 1H), 7.80 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 5.2 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 2H), 3.81 (s, 3H), 3.72 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.23, 160.66, 157.75, 149.66, 139.79, 130.52, 128.11, 118.66, 117.34, 114.00, 63.97, 55.11; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{15}N_2O_3$ $(M+H)^+$: 259.1077, found: 259.1080.



2-(4-chlorophenyl)-N-methoxyisonicotinamide (6o)

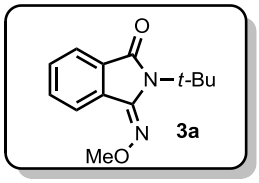
Following typical procedure 3: ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.16 (br, 1H), 8.80 (d, *J* = 4.8 Hz, 1H), 8.21 (s, 1H), 8.14 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 4.4 Hz, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 3.78 (s, 3H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 162.04, 155.51, 150.43, 140.60, 136.82, 134.44, 128.90, 128.42, 120.22, 117.35, 63.48; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{12}ClN_2O_2$ $(M+H)^+$: 263.0582, found: 263.0592.



N-methoxy-2-(naphthalen-2-yl)isonicotinamide (6p)

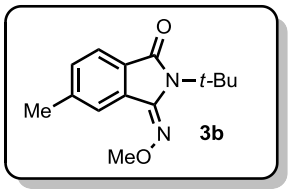
Following typical procedure 2: ^{1}H NMR (400 MHz, acetone-d_6) δ 11.33 (br, 1H), 8.84 (d, J = 4.8 Hz, 1H), 8.69 (s, 1H), 8.41 (s, 1H), 8.32 (d, J = 8.4 Hz, 1H), 8.01 (d, J = 8.4 Hz, 2H), 7.94 (dd, J = 5.2, 3.2 Hz, 1H), 7.70 (d, J = 4.4 Hz, 1H), 7.55 (dd, J = 6.0, 2.8 Hz, 2H), 3.88 (s, 3H); **^{13}C NMR** (100 MHz, acetone-d_6) δ 164.59, 159.04, 152.04, 142.52, 137.52, 135.54, 135.14, 130.33, 129.98, 129.23, 128.42, 128.06, 127.93, 125.94, 121.32, 119.22, 65.00; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{15}N_2O_2$ $(M+H)^+$: 279.1128, found: 279.1128.

3.2 Characterization of Products



(E)-2-(tert-butyl)-3-(methoxyimino)isoindolin-1-one (3a)

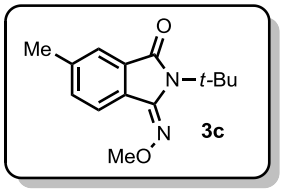
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26-8.24 (m, 1H), 7.77-7.75 (m, 1H), 7.58-7.50 (m, 2H), 4.06 (s, 3H), 1.76 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.33, 149.98, 132.78, 131.74, 131.10, 128.53, 127.73, 122.65, 63.24, 58.65, 29.60; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{17}N_2O_2$ $(M+H)^+$: 233.1285, found: 233.1276.



(E)-2-(tert-butyl)-3-(methoxyimino)-5-methylisoindolin-1-one (3b)

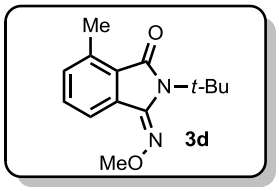
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.64 (d, J = 7.6 Hz, 1H), 7.34 (d, J = 7.6 Hz, 1H), 4.07 (s, 3H), 2.46 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.58, 150.14, 143.63,

132.01, 129.43, 128.93, 128.23, 122.61, 63.33, 58.64, 29.68, 22.17;**HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{19}N_2O_2$ $(M+H)^+$: 247.1441, found: 247.1452.



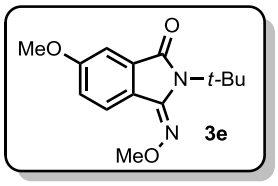
(E)-2-(tert-butyl)-3-(methoxyimino)-6-methylisoindolin-1-one (3c)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 7.6 Hz, 1H), 7.55 (s, 1H), 7.36 (dd, *J* = 8.0, 0.8 Hz, 1H), 4.05 (s, 3H), 2.44 (s, 3H), 1.75 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.63, 150.25, 141.86, 133.63, 132.12, 127.58, 126.18, 123.09, 63.23, 58.66, 29.70, 21.81; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{19}N_2O_2$ $(M+H)^+$: 247.1441, found: 247.1452.



(E)-2-(tert-butyl)-3-(methoxyimino)-7-methylisoindolin-1-one (3d)

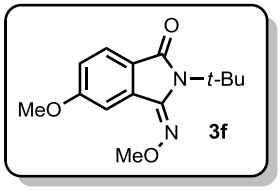
10 mol % of $Pd_2(dba)_3$ was used at 100 °C for 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 7.6 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 4.05 (s, 3H), 2.67 (s, 3H), 1.75 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.37, 149.80, 137.14, 133.71, 132.35, 129.10, 128.40, 125.51, 63.30, 58.58, 29.73, 17.90; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{19}N_2O_2$ $(M+H)^+$: 247.1441, found: 247.1451.



(E)-2-(tert-butyl)-6-methoxy-3-(methoxyimino)isoindolin-1-one (3e)

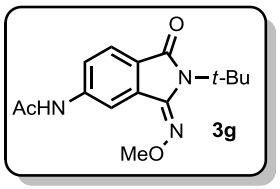
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.4 Hz, 1H), 7.24 (d, *J* = 2.8 Hz, 1H), 7.07 (dd, *J* = 8.8, 2.8 Hz, 1H), 4.04 (s, 3H), 3.88 (s, 3H), 1.75 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.38, 162.17, 150.03, 134.01, 129.18, 121.39, 119.99, 106.15, 63.22,

58.80, 55.85, 29.76; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{19}N_2O_3$ $(M+H)^+$: 263.1390, found: 263.1391



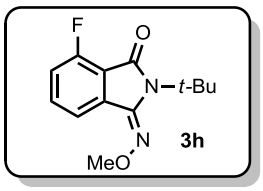
(E)-2-(tert-butyl)-5-methoxy-3-(methoxyimino)isoindolin-1-one (3f)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 2.4 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 7.04 (dd, *J* = 8.4, 2.4 Hz, 1H), 4.06 (s, 3H), 3.88 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.39, 163.61, 149.87, 130.41, 124.19, 117.20, 112.93, 63.41, 58.63, 55.93, 29.66; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{19}N_2O_3$ $(M+H)^+$: 263.1390, found: 263.1398.



(E)-N-(2-(tert-butyl)-3-(methoxyimino)-1-oxoisoindolin-5-yl)acetamide (3g)

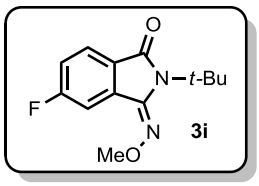
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 1.2 Hz, 1H), 8.18 (br, 1H), 7.80 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 4.02 (s, 3H), 2.22 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.97, 167.17, 149.57, 142.47, 129.45, 127.15, 123.60, 122.23, 118.73, 63.43, 58.84, 29.64, 24.78; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{20}N_3O_3$ $(M+H)^+$: 290.1499, found: 290.1503.



(E)-2-(tert-butyl)-7-fluoro-3-(methoxyimino)isoindolin-1-one (3h)

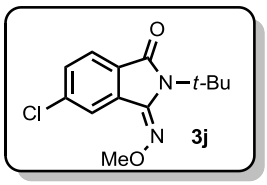
10 mol % of $Pd_2(dba)_3$ was used at 100 °C for 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 7.6 Hz, 1H), 7.58-7.30 (m, 1H), 7.21-7.17 (m, 1H), 4.07 (s, 3H), 1.75 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.32 (d, *J* = 2.3 Hz), 158.06 (d, *J* = 260.5 Hz), 149.03 (d, *J* = 1.9 Hz), 134.91 (d, *J* = 7.4 Hz), 130.67 (d, *J* = 2.4 Hz), 124.05 (d, *J* = 4.1 Hz), 119.01 (d, *J* = 19.2 Hz), 118.10 (d,

J = 11.4 Hz), 63.52, 59.13, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{16}FN_2O_2$ $(M+H)^+$: 251.1190, found: 251.1199.



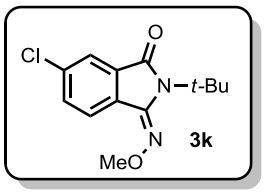
(E)-2-(tert-butyl)-5-fluoro-3-(methoxyimino)isoindolin-1-one (3i)

The reaction time was 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.74 (dd, *J* = 8.4, 4.8 Hz, 1H), 7.23 (td, *J* = 8.4, 2.4 Hz, 1H), 4.07 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.88, 165.42 (d, *J* = 207.2 Hz), 149.08 (d, *J* = 3.0 Hz), 130.24 (d, *J* = 11.3 Hz), 127.72 (d, *J* = 2.2 Hz), 124.74 (d, *J* = 9.6 Hz), 118.59 (d, *J* = 23.5 Hz), 115.24 (d, *J* = 26.3 Hz), 63.51, 59.01, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{16}FN_2O_2$ $(M+H)^+$: 251.1190, found: 251.1196.



(E)-2-(tert-butyl)-5-chloro-3-(methoxyimino)isoindolin-1-one (3j)

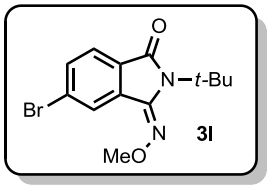
The reaction time was 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.26 (dd, *J* = 1.6, 0.4 Hz, 1H), 7.69 (dd, *J* = 8.0, 0.4 Hz, 1H), 7.51 (dd, *J* = 8.0, 2.0 Hz, 1H), 4.08 (s, 3H), 1.74 (s, 9 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.44, 148.93, 139.01, 131.45, 130.05, 129.69, 127.96, 123.94, 63.56, 59.08, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{16}ClN_2O_2$ $(M+H)^+$: 267.0895, found: 267.0894.



(E)-2-(tert-butyl)-6-chloro-3-(methoxyimino)isoindolin-1-one (3k)

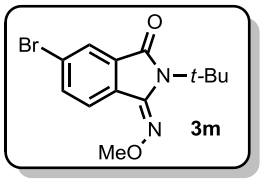
The reaction time was 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 2.0 Hz, 1H), 7.52 (dd, *J* = 8.4, 2.0 Hz, 1H), 4.07 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz,

$CDCl_3$) δ 166.05, 149.24, 137.45, 133.47, 132.90, 128.99, 126.66, 123.03, 63.48, 59.12, 29.61; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{16}ClN_2O_2$ $(M+H)^+$: 267.0895, found:267.0894.



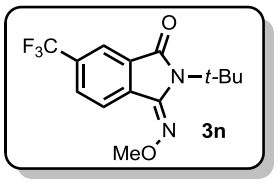
(E)-5-bromo-2-(tert-butyl)-3-(methoxyimino)isoindolin-1-one (3l)

5 mol % of $Pd_2(dba)_3$ was used at 80 °C for 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 1.2 Hz, 1H), 7.68 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 4.08 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.54, 148.83, 134.34, 130.79, 130.50, 129.87, 127.40, 124.15, 63.58, 59.09, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{16}BrN_2O_2$ $(M+H)^+$: 311.0390, found: 311.0401.



(E)-6-bromo-2-(tert-butyl)-3-(methoxyimino)isoindolin-1-one (3m)

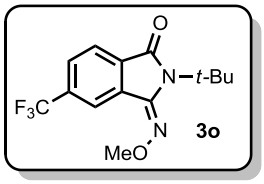
10 mol % of $Pd_2(dba)_3$ was used at 100 °C for 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.4 Hz, 1H), 7.88 (d, *J* = 2.0 Hz, 1H), 7.69 (dd, *J* = 8.0, 1.6 Hz, 1H), 4.07 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.97, 149.35, 135.82, 133.56, 129.19, 127.11, 126.08, 125.62, 63.52, 59.14, 29.63; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{16}BrN_2O_2$ $(M+H)^+$: 311.0390, found: 311.0399.



(E)-2-(tert-butyl)-3-(methoxyimino)-6-(trifluoromethyl)isoindolin-1-one (3n)

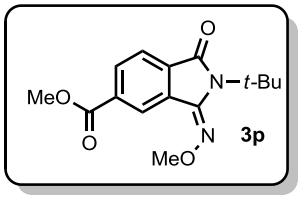
10 mol % of $Pd_2(dba)_3$ was used at 80 °C for 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.0 Hz, 1H), 8.03 (s, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 4.10 (s, 3H), 1.76 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.99, 149.01, 133.22 (q, *J* = 32.9 Hz), 132.40, 131.09, 129.74 (q, *J* = 3.7 Hz),

128.35, 123.57 (q, J = 271.2 Hz), 120.05 (q, J = 3.9 Hz), 63.69, 59.34, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{16}F_3N_2O_2$ $(M+H)^+$: 301.1158, found: 301.1159.



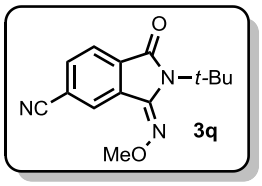
(E)-2-(tert-butyl)-3-(methoxyimino)-5-(trifluoromethyl)isoindolin-1-one (3o)

The reaction time was 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.52 (s, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 4.13 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.81, 148.72, 134.61 (q, J = 32.2 Hz, 134.46, 128.62, 128.12 (q, J = 3.5 Hz), 124.93 (q, J = 3.9 Hz), 123.65 (q, J = 271.5 Hz), 123.19, 63.55, 59.24, 29.43; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{16}F_3N_2O_2$ $(M+H)^+$: 301.1158, found: 301.1168.



(E)-methyl 2-(tert-butyl)-3-(methoxyimino)-1-oxoisoindoline-5-carboxylate (3p)

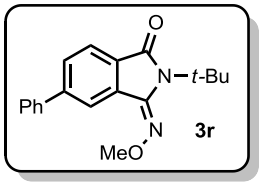
The reaction time was 3 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.87 (t, J = 0.8 Hz, 1H), 8.23 (dd, J = 8.0, 0.8 Hz, 1H), 7.83 (d, J = 8.0 Hz, 1H), 4.11 (s, 3H), 3.97 (s, 3H), 1.76 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.41, 166.30, 149.17, 135.24, 134.37, 132.58, 129.09, 128.50, 122.77, 63.65, 59.26, 52.73, 29.63; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{19}N_2O_4$ $(M+H)^+$: 291.1339, found:291.1337.



(E)-2-(tert-butyl)-3-(methoxyimino)-1-oxoisoindoline-5-carbonitrile (3q)

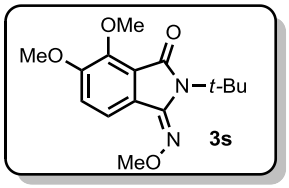
5 mol % of $Pd_2(dba)_3$ was used at 80 °C for 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 4.12 (s, 3H), 1.76 (s, 9H); **^{13}C NMR** (100

MHz, $CDCl_3$) δ 165.47, 148.30, 134.78, 134.76, 131.69, 128.61, 123.55, 118.08, 116.40, 63.76, 59.55, 29.48; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{14}H_{16}N_3O_2$ $(M+H)^+$: 258.1237, found: 258.1240.



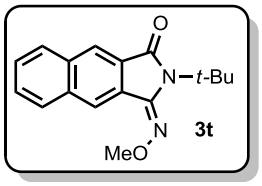
(E)-2-(tert-butyl)-3-(methoxyimino)-5-phenylisoindolin-1-one (3r)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (dd, *J* = 1.6, 0.8 Hz, 1H), 7.82 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.74 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.62-7.60 (m, 2H), 7.50-7.46 (m, 2H), 7.43-7.39 (m, 1H), 4.08 (s, 3H), 1.77 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.37, 149.95, 146.37, 140.51, 130.59, 130.33, 129.30, 129.07, 128.26, 127.68, 126.68, 123.14, 63.49, 58.87, 29.69; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{19}H_{21}N_2O_2$ $(M+H)^+$: 309.1598, found: 309.1611.



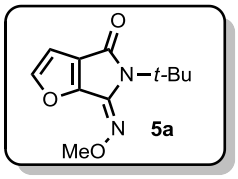
(E)-2-(tert-butyl)-6,7-dimethoxy-3-(methoxyimino)isoindolin-1-one (3s)

5 mol % of $Pd_2(dba)_3$ was used at 80 °C for 12 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.4 Hz, 1H), 7.05 (d, *J* = 8.4 Hz, 1H), 4.03 (s, 3H), 4.02 (s, 3H), 3.92 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.51, 155.36, 149.31, 146.43, 124.25, 123.54, 122.17, 116.03, 63.19, 62.24, 58.77, 56.53, 29.71; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{21}N_2O_4$ $(M+H)^+$: 293.1496, found: 293.1484.



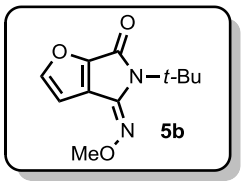
(E)-2-(tert-butyl)-3-(methoxyimino)-2,3-dihydro-1H-benzo[f]isoindol-1-one (3t)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.76 (s, 1H), 8.24 (s, 1H), 7.99-7.96 (m, 2H), 7.62-7.56 (m, 2H), 4.13 (s, 3H), 1.81 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.39, 149.95, 135.89, 134.14, 129.81, 129.67, 128.86, 128.64, 128.07, 127.97, 124.43, 123.31, 63.41, 59.09, 29.61; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{19}N_2O_2$ $(M+H)^+$: 283.1441, found: 283.1442.



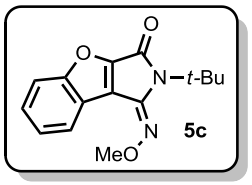
(E)-5-(tert-butyl)-6-(methoxyimino)-5,6-dihydro-4H-furo[2,3-c]pyrrol-4-one (5a)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 1.6 Hz, 1H), 6.59 (d, *J* = 2.0 Hz, 1H), 4.06 (s, 3H), 1.68 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.24, 153.64, 150.17, 143.60, 125.37, 106.00, 63.63, 59.11, 29.94; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{15}N_2O_3$ $(M+H)^+$: 223.1077, found: 223.1076.



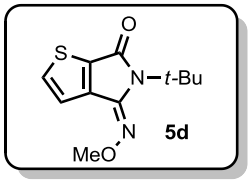
(E)-5-(tert-butyl)-4-(methoxyimino)-4H-furo[2,3-c]pyrrol-6(5H)-one (5b)

The reaction time was 1 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 2.0 Hz, 1H), 6.70 (d, *J* = 1.6 Hz, 1H), 4.01 (s, 3H), 1.69 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.64, 152.09, 150.30, 146.32, 124.00, 109.02, 63.15, 59.29, 29.95; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{15}N_2O_3$ $(M+H)^+$: 223.1077, found: 223.1079.



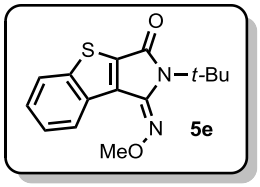
(E)-2-(tert-butyl)-1-(methoxyimino)-1H-benzofuro[2,3-c]pyrrol-3(2H)-one (5c)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82-7.80 (m, 1H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.47-7.42 (m, 1H), 7.39-7.35 (m, 1H), 4.17 (s, 3H), 1.75 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 160.94, 158.47, 153.57, 146.84, 127.58, 124.62, 123.88, 121.92, 119.88, 113.16, 63.22, 59.74, 30.06; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{17}N_2O_3$ $(M+H)^+$: 273.1234, found: 273.1235.



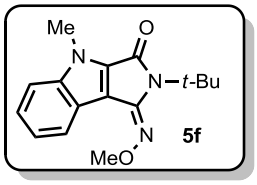
(E)-5-(tert-butyl)-4-(methoxyimino)-4H-thieno[2,3-c]pyrrol-6(5H)-one (5d)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 4.8 Hz, 1H), 7.43 (d, *J* = 4.8 Hz, 1H), 4.03 (s, 3H), 1.72 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.67, 148.29, 138.62, 138.26, 134.83, 124.74, 63.17, 59.22, 29.84; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{15}N_2O_2S$ $(M+H)^+$: 239.0849, found: 239.0860.



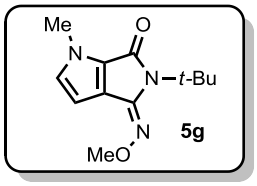
(E)-2-(tert-butyl)-1-(methoxyimino)-1H-benzo[4,5]thieno[2,3-c]pyrrol-3(2H)-one (5e)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43-8.41 (m, 1H), 7.88-7.86 (m, 1H), 7.48-7.42 (m, 2H), 4.18 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.07, 147.97, 145.91, 140.78, 133.88, 132.17, 127.04, 126.67, 125.58, 123.86, 63.08, 59.69, 30.04; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{15}H_{17}N_2O_2S$ $(M+H)^+$: 289.1005, found: 289.1003.



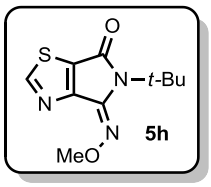
(E)-2-(tert-butyl)-1-(methoxyimino)-4-methyl-1,2-dihydropyrrolo[3,4-b]indol-3(4H)-one (5f)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.0 Hz, 1H), 7.48-7.33 (m, 3H), 4.05 (s, 3H), 4.03 (s, 3H), 1.65 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.98, 143.16, 142.34, 134.95, 124.72, 122.85, 122.78, 121.75, 111.54, 107.97, 64.99, 54.34, 31.13, 30.87; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{16}H_{20}N_3O_2$ $(M+H)^+$: 286.1550, found: 286.1538.



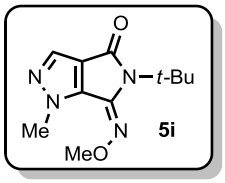
(E)-5-(tert-butyl)-4-(methoxyimino)-1-methyl-4,5-dihydropyrrolo[3,4-b]pyrrol-6(1H)-one (5g)

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.87 (d, *J* = 2.4 Hz, 1H), 6.32 (d, *J* = 2.4 Hz, 1H), 3.96 (s, 3H), 3.87 (s, 3H), 1.47 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.72, 143.73, 130.35, 127.89, 118.45, 108.02, 64.77, 54.50, 34.69, 30.17; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{18}N_3O_2$ $(M+H)^+$: 236.1394, found: 236.1396.



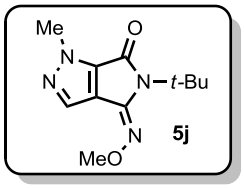
(E)-5-(tert-butyl)-4-(methoxyimino)-4H-pyrrolo[3,4-d]thiazol-6(5H)-one (5h)

5 mol % of $Pd_2(dba)_3$ was used at 80 °C for 8 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.12 (s, 1H), 4.15 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.01, 160.30, 154.59, 145.26, 133.06, 64.22, 60.09, 29.90; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{10}H_{14}N_3O_2S$ $(M+H)^+$: 240.0801, found: 240.0808.



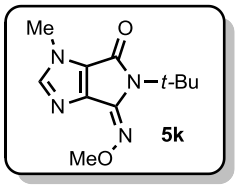
(E)-5-(tert-butyl)-6-(methoxyimino)-1-methyl-5,6-dihydropyrrolo[3,4-c]pyrazol-4(1H)-one (5i)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (s, 1H), 4.16 (s, 3H), 4.02 (s, 3H), 1.68 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.70, 143.03, 140.30, 132.27, 122.79, 62.83, 59.10, 41.73, 29.75; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{17}N_4O_2$ $(M+H)^+$: 237.1346, found: 237.1351.



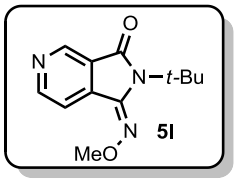
(E)-5-(tert-butyl)-4-(methoxyimino)-1-methyl-4,5-dihydropyrrolo[3,4-c]pyrazol-6(1H)-one (5j)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (s, 1H), 4.02 (s, 3H), 4.01 (s, 3H), 1.70 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 157.89, 146.29, 139.44, 134.44, 118.43, 63.05, 59.46, 37.24, 29.78; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{17}N_4O_2$ $(M+H)^+$: 237.1346, found: 237.1349.



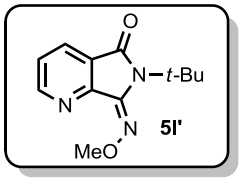
(E)-5-(tert-butyl)-6-(methoxyimino)-3-methyl-5,6-dihydropyrrolo[3,4-d]imidazol-4(3H)-one (5k)

10 mol % of $Pd_2(dba)_3$ was used at 100 °C for 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.62 (s, 1H), 4.09 (s, 3H), 3.83 (s, 3H), 1.70 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.90, 145.60, 145.50, 142.38, 130.01, 63.76, 59.24, 32.55, 30.04; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{11}H_{17}N_4O_2$ $(M+H)^+$: 237.1346, found: 237.1348.



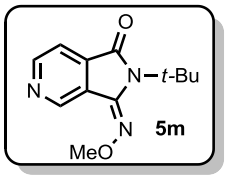
(E)-2-(tert-butyl)-1-(methoxyimino)-1H-pyrrolo[3,4-c]pyridin-3(2H)-one (5l)

10 mol % of $Pd_2(dba)_3$ was used at 100 °C for 11 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.05 (d, *J* = 1.2 Hz, 1H), 8.86 (d, *J* = 5.2 Hz, 1H), 8.09 (dd, *J* = 5.2, 1.2 Hz, 1H), 4.10 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.10, 154.00, 148.86, 145.15, 134.92, 125.89, 121.33, 63.80, 59.34, 29.44; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{16}N_3O_2$ $(M+H)^+$: 234.1237, found: 234.1233.



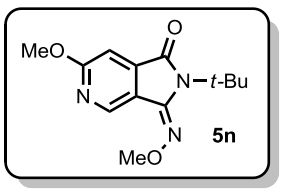
(E)-6-(tert-butyl)-7-(methoxyimino)-6,7-dihydro-5H-pyrrolo[3,4-b]pyridin-5-one (5l')

10 mol % of $Pd_2(dba)_3$ was used at 100 °C for 11 h. This byproduct was isolated as a mixture of inseparable E:Z isomers of ratio 12:1. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.95 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.08 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.46 (dd, *J* = 7.6, 4.8 Hz, 1H), 4.19 (s, 3H), 1.78 (s, 9H; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.57, 164.92, 158.89, 154.34, 151.24, 150.87, 148.69, 147.35, 136.25, 131.04, 126.62, 125.22, 122.99, 64.74, 59.55, 30.04, 29.72; HRMS (ESI-TOF) m/z Calcd for HRMS (ESI-TOF) m/z Calcd for $C_{12}H_{16}N_3O_2$ $(M+H)^+$: 234.1237, found: 234.1233.



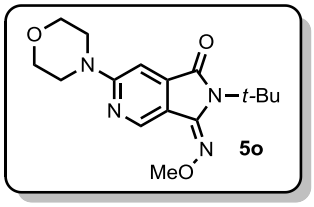
(E)-2-(tert-butyl)-3-(methoxyimino)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (5m)

The reaction time was 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.50 (s, 1H), 8.85 (d, *J* = 4.8 Hz, 1H), 7.68-7.67 (m, 1H), 4.13 (s, 3H), 1.76 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.77, 152.02, 148.95, 148.79, 138.50, 123.89, 116.54, 63.66, 59.52, 29.52; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{12}H_{16}N_3O_2$ $(M+H)^+$: 234.1237, found: 234.1233.



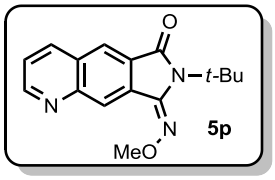
(E)-2-(tert-butyl)-6-methoxy-3-(methoxyimino)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (5n)

5 mol % of $Pd_2(dba)_3$ was used at 80 °C for 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.03 (s, 1H), 7.05 (s, 1H), 4.07 (s, 3H), 4.01 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.57, 165.48, 149.03, 146.95, 141.95, 117.77, 104.18, 63.34, 59.45, 54.56, 29.53; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{13}H_{18}N_3O_3$ $(M+H)^+$: 264.1343, found: 264.1345.



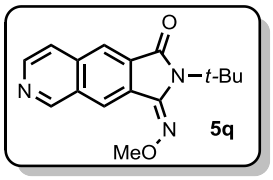
(E)-2-(tert-butyl)-3-(methoxyimino)-6-morpholino-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (5o)

5 mol % of $Pd_2(dba)_3$ was used at 80 °C for 6 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 6.90 (s, 1H), 4.04 (s, 3H), 3.82 (t, *J* = 4.0 Hz, 4H), 3.66 (t, *J* = 4.0 Hz, 4H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.40, 160.08, 149.65, 147.82, 140.96, 113.79, 98.66, 66.64, 63.15, 59.17, 45.39, 29.59; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{16}H_{23}N_4O_3$ $(M+H)^+$:319.1765, found: 319.1760.



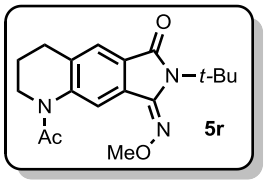
(E)-7-(tert-butyl)-8-(methoxyimino)-7,8-dihydro-6H-pyrrolo[3,4-g]quinolin-6-one (5p)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.07 (s, 1H), 9.03 (dd, *J* = 4.0, 1.6 Hz, 1H), 8.32 (d, *J* = 7.6 Hz, 1H), 8.25 (s, 1H), 7.52 (dd, *J* = 8.4, 4.0 Hz, 1H), 4.14 (s, 3H), 1.81 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.69, 152.35, 150.62, 149.40, 137.61, 130.07, 129.42, 129.37, 127.54, 123.18, 122.65, 63.52, 59.40, 29.55; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{16}H_{18}N_3O_2$ $(M+H)^+$: 284.1394, found: 284.1397.



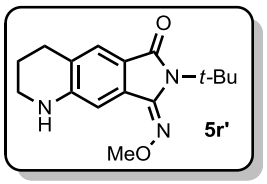
(E)-2-(tert-butyl)-3-(methoxyimino)-2,3-dihydro-1H-pyrrolo[3,4-g]isoquinolin-1-one (5q)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.92 (s, 1H), 8.65 (d, *J* = 5.6 Hz, 1H), 8.24 (s, 1H), 7.81 (d, *J* = 6.0 Hz, 1H), 4.17 (s, 3H), 1.81 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.37, 154.16, 149.16, 144.92, 136.79, 133.49, 132.63, 130.64, 128.56, 125.46, 121.86, 63.66, 59.57, 29.54; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{16}H_{18}N_3O_2$ $(M+H)^+$: 284.1394, found: 284.1396.



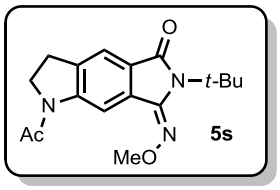
(E)-1-acetyl-7-(tert-butyl)-8-(methoxyimino)-3,4,7,8-tetrahydro-1H-pyrrolo[3,4-g]quinolin-6(2H)-one (5r)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.54 (s, 1H), 4.05 (s, 3H), 3.80 (t, *J* = 6.8 Hz, 2H), 2.80 (t, *J* = 6.8 Hz, 2H), 2.28 (s, 3H), 1.98 (m, 2H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.30, 167.01, 149.72, 143.10, 128.27, 126.89, 123.98, 122.54, 63.44, 58.82, 43.85, 29.61, 27.71, 23.95, 23.63; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{24}N_3O_3(M+H)^+$: 330.1812, found: 330.1814.



(E)-7-(tert-butyl)-8-(methoxyimino)-3,4,7,8-tetrahydro-1H-pyrrolo[3,4-g]quinolin-6(2H)-one (5r')

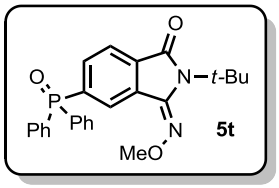
The reaction time was 4 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 (s, 1H), 7.29 (s, 1H), 4.43 (brs, 1H), 3.99 (s, 3H), 3.35 (t, *J* = 4.6 Hz, 2H), 2.79 (t, *J* = 6.2 Hz, 2H), 1.94-1.88 (m, 2H), 1.71 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.23, 150.54, 148.98, 128.35, 123.75, 123.29, 119.63, 111.90, 62.89, 57.91, 41.75, 29.53, 27.41, 21.16; **HRMS** (ESI-TOF) m/z Calcd for $C_{16}H_{22}N_3O_2$ $(M+H)^+$: 288.1707, found: 288.1703.



(E)-1-acetyl-6-(tert-butyl)-7-(methoxyimino)-2,3,6,7-tetrahydropyrrolo[3,4-f]indol-5(1H)-one (5s)

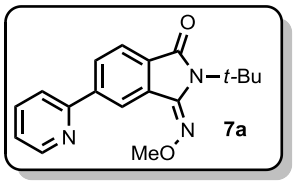
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.02 (s, 1H), 7.50 (s, 1H), 4.15 (t, *J* = 8.4 Hz, 2H), 4.07 (s, 3H), 3.25 (t, *J* = 8.4 Hz, 2H), 2.26 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.23, 167.15, 149.74, 147.03, 134.85, 128.86, 127.57, 118.82, 115.81, 63.44,

58.68, 49.42, 29.69, 27.88, 24.51; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{22}N_3O_3(M+H)^+$: 316.1656, found: 316.1664.



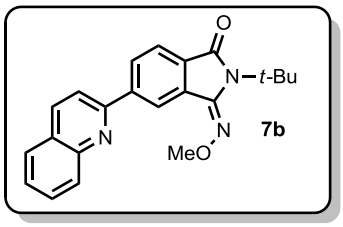
(E)-2-(tert-butyl)-5-(diphenylphosphoryl)-3-(methoxyimino)isoindolin-1-one (5t)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 12.0 Hz, 1H), 7.86-7.47 (m, 12H), 3.96 (s, 3H), 1.74 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.32, 149.11 (d, *J* = 1.7 Hz), 138.04 (d, *J* = 99.9 Hz), 134.75 (d, *J* = 10.9 Hz, 6H), 134.47 (d, *J* = 2.5 Hz), 132.40 (d, *J* = 2.8 Hz), 132.15 (d, *J* = 9.9 Hz), 131.80 (d, *J* = 104.6 Hz), 131.55 (d, *J* = 11.6 Hz), 128.76 (d, *J* = 12.3 Hz), 128.38 (d, *J* = 14.5 Hz), 122.72 (d, *J* = 12.6 Hz), 63.44, 59.20, 29.55; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{25}H_{26}N_2O_3P$ $(M+H)^+$: 433.1676, found: 433.1677.



(E)-2-(tert-butyl)-3-(methoxyimino)-5-(pyridin-2-yl)isoindolin-1-one (7a)

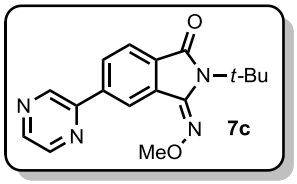
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.83-8.82 (m, 1H), 8.73 (d, *J* = 4.8 Hz, 1H), 8.22-8.17 (m, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.82-7.76 (m, 2H), 7.31-7.28 (m, 1H), 4.11 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.18, 156.49, 149.98, 149.74, 144.23, 137.05, 131.91, 130.16, 129.14, 126.33, 123.09, 122.97, 121.32, 63.53, 58.90, 29.66; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{20}N_3O_2$ $(M+H)^+$: 310.1550, found: 310.1540.



(E)-2-(tert-butyl)-3-(methoxyimino)-5-(quinolin-2-yl)isoindolin-1-one (7b)

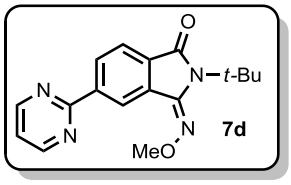
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.97 (d, *J* = 0.8 Hz, 1H), 8.40 (d, *J* = 8.0 Hz, 1H), 8.26 (d, *J* = 8.8 Hz, 1H), 8.20 (d, *J* = 8.6 Hz, 1H), 7.91 (d, *J* = 4.4 Hz, 1H), 7.89 (d,

J = 5.2 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.6 Hz, 1H), 4.13 (s, 3H), 1.79 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.21, 156.29, 149.82, 148.35, 144.47, 137.21, 132.20, 130.86, 130.08, 129.98, 129.21, 127.62, 127.50, 126.95, 126.92, 123.21, 119.27, 63.58, 58.99, 29.70; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{22}H_{22}N_3O_2(M+H)^+$: 360.1707, found: 360.1715.



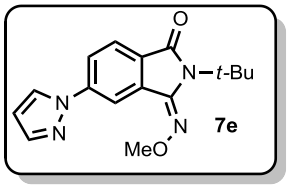
(E)-2-(tert-butyl)-3-(methoxyimino)-5-(pyrazin-2-yl)isoindolin-1-one (7c)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.08 (d, *J* = 0.8 Hz, 1H), 8.88 (s, 1H), 8.69 (d, *J* = 1.6 Hz, 1H), 8.59 (d, *J* = 2.4 Hz, 1H), 8.22 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 1H), 4.13 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.84, 151.96, 149.50, 144.46, 143.83, 142.68, 141.00, 132.70, 130.08, 129.29, 126.33, 123.42, 63.62, 59.07, 29.63; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{19}N_4O_2$ $(M+H)^+$: 311.1503, found: 311.1513.



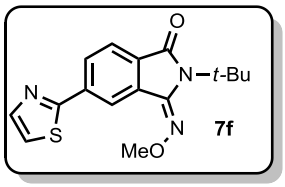
(E)-2-(tert-butyl)-3-(methoxyimino)-5-(pyrimidin-2-yl)isoindolin-1-one (7d)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.27 (s, 1H), 8.85 (d, *J* = 4.8 Hz, 2H), 8.66 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.25 (t, *J* = 4.8 Hz, 1H), 4.14 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.06, 163.82, 157.44, 149.63, 142.20, 133.39, 131.23, 128.88, 127.60, 122.85, 119.82, 63.58, 58.95, 29.66; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{17}H_{19}N_4O_2$ $(M+H)^+$: 311.1503, found: 311.1509.



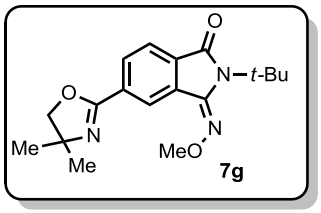
(E)-2-(tert-butyl)-3-(methoxyimino)-5-(1H-pyrazol-1-yl)isoindolin-1-one (7e)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.58 (dd, *J* = 2.0, 0.4 Hz, 1H), 8.00 (dd, *J* = 2.0, 0.4 Hz, 1H), 7.91 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.83 (dd, *J* = 8.4, 0.4 Hz, 1H), 7.77 (d, *J* = 1.6, 1H), 6.52 (dd, *J* = 2.8, 2.0 Hz, 1H), 4.10 (s, 3H), 1.76 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.60, 149.20, 143.67, 142.00, 129.74, 129.31, 127.26, 123.96, 121.81, 118.09, 108.55, 63.58, 58.95, 29.57; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{16}H_{19}N_4O_2$ $(M+H)^+$: 299.1503, found: 299.1507.



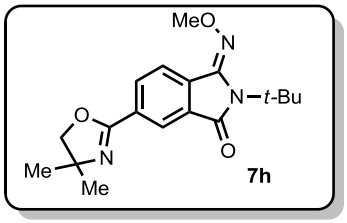
(E)-2-(tert-butyl)-3-(methoxyimino)-5-(thiazol-2-yl)isoindolin-1-one (7f)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.84 (s, 1H), 8.15 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.94 (d, *J* = 3.2 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 3.2 Hz, 1H), 4.12 (s, 3H), 1.77 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.16, 166.79, 149.38, 144.33, 137.92, 132.67, 129.54, 129.29, 125.88, 123.43, 120.14, 63.67, 59.10, 29.65; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{16}H_{18}N_3O_2S$ $(M+H)^+$: 316.1114, found: 316.1122.



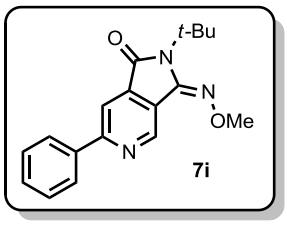
(E)-2-(tert-butyl)-5-(4,4-dimethyl-4,5-dihydrooxazol-2-yl)-3-(methoxyimino)isoindolin-1-one (7g)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.74-8.73 (m, 1H), 8.16 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.79 (dd, *J* = 8.0, 0.8 Hz, 1H), 4.15 (s, 2H), 4.10 (s, 3H), 1.76 (s, 9H), 1.41 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.74, 161.39, 149.30, 133.81, 132.61, 131.40, 128.47, 127.48, 122.67, 79.49, 68.11, 63.65, 59.10, 29.65, 28.51; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{24}N_3O_3$$(M+H)^+$: 330.1812, found: 330.1818.



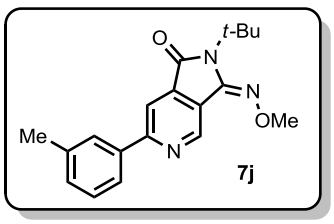
(E)-2-(tert-butyl)-6-(4,4-dimethyl-4,5-dihydrooxazol-2-yl)-3-(methoxyimino)isoindolin-1-one (7h)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.29 (dd, *J* = 1.6, 0.8 Hz, 1H), 8.26 (dd, *J* = 8.0, 0.8 Hz, 1H), 8.19 (dd, *J* = 8.0, 1.6 Hz, 1H), 4.15 (s, 2H), 4.09 (s, 3H), 1.75 (s, 9H), 1.40 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.58, 161.14, 149.57, 132.77, 132.00, 130.91, 130.34, 127.59, 122.67, 79.47, 68.06, 63.49, 58.96, 29.61, 28.47; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{24}N_3O_3(M+H)^+$: 330.1812, found: 330.1820.



(E)-2-(tert-butyl)-3-(methoxyimino)-6-phenyl-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7i)

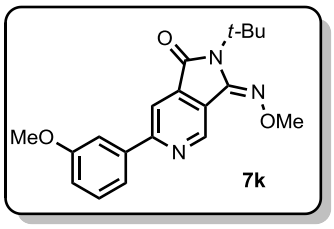
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.52 (d, *J* = 1.2 Hz, 1H), 8.10-8.09 (m, 3H), 7.53-7.47 (m, 3H), 4.13 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.00, 159.82, 149.17, 148.53, 139.77, 138.37, 130.13, 129.10, 127.42, 122.24, 112.99, 63.62, 59.55, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{20}N_3O_2(M+H)^+$: 310.1550, found: 310.1541.



(E)-2-(tert-butyl)-3-(methoxyimino)-6-(m-tolyl)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7j)

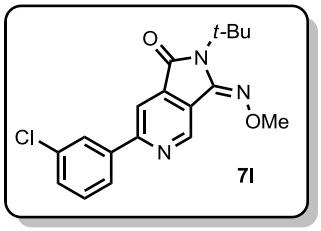
The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.51 (d, *J* = 1.2 Hz, 1H), 8.07 (d, *J* = 1.2 Hz, 1H), 7.92 (s, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 7.6 Hz,

1H), 4.13 (s, 3H), 2.45 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.00, 159.97, 149.17, 148.43, 139.69, 138.80, 138.29, 130.89, 128.99, 128.10, 124.51, 122.13, 113.02, 63.59, 59.51, 29.56, 21.65; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{19}H_{22}N_3O_2(M+H)^+$: 324.1707, found: 324.1703.



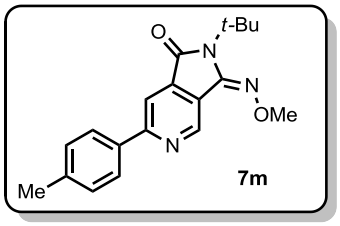
(E)-2-(tert-butyl)-3-(methoxyimino)-6-(3-methoxyphenyl)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7k)

The reaction time was 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.51 (d, *J* = 1.2 Hz, 1H), 8.08 (d, *J* = 1.2 Hz, 1H), 7.68-7.63 (m, 2H), 7.41 (t, *J* = 8.0 Hz, 1H), 7.02 (ddd, *J* = 8.0, 2.4, 0.8 Hz, 1H), 4.13 (s, 3H), 3.90 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.97, 160.29, 159.54, 149.14, 148.43, 139.75, 139.72, 130.09, 122.32, 119.78, 116.41, 113.11, 112.28, 63.61, 59.55, 55.53, 29.57; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{19}H_{22}N_3O_3(M+H)^+$: 340.1656, found: 340.1663.



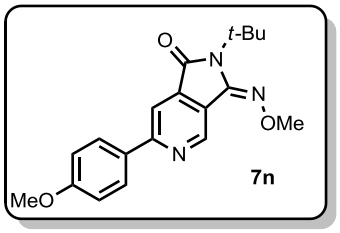
(E)-2-(tert-butyl)-6-(3-chlorophenyl)-3-(methoxyimino)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7l)

The reaction time was 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.51 (d, *J* = 1.2 Hz, 1H), 8.12 (dd, *J* = 2.4, 1.2 Hz, 1H), 8.06 (d, *J* = 1.2 Hz, 1H), 7.95-7.92 (m, 1H), 7.44-7.43 (m, 2H), 4.14 (s, 3H), 1.78 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.74, 158.20, 148.98, 148.60, 140.08, 139.90, 135.29, 130.31, 130.07, 127.65, 125.38, 122.71, 113.09, 63.70, 59.65, 29.57; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{19}ClN_3O_2(M+H)^+$: 344.1160, found: 344.1165.



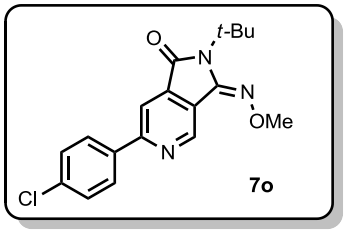
(E)-2-(tert-butyl)-3-(methoxyimino)-6-(p-tolyl)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7m)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.48 (d, *J* = 1.2 Hz, 1H), 8.05 (d, *J* = 1.2 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 4.11 (s, 3H), 2.41 (s, 3H), 1.77 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.02, 159.75, 149.18, 148.40, 140.33, 139.63, 135.54, 129.78, 127.25, 121.86, 112.54, 63.53, 59.44, 29.54, 21.45; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{19}H_{22}N_3O_2(M+H)^+$: 324.1707, found: 324.1712.



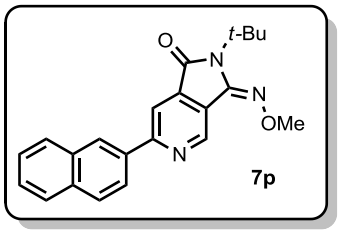
(E)-2-(tert-butyl)-3-(methoxyimino)-6-(4-methoxyphenyl)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7n)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.46 (d, *J* = 1.2 Hz, 1H), 8.06 (d, *J* = 8.8 Hz, 2H), 8.01 (d, *J* = 1.2 Hz, 1H), 7.02 (d, *J* = 9.2 Hz, 2H), 4.12 (s, 3H), 3.88 (s, 3H), 1.77 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.13, 161.41, 159.43, 149.27, 148.42, 139.67, 130.97, 128.84, 121.47, 114.46, 112.05, 63.56, 59.48, 55.54, 29.58; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{19}H_{22}N_3O_3(M+H)^+$: 340.1656, found: 340.1663.



(E)-2-(tert-butyl)-6-(4-chlorophenyl)-3-(methoxyimino)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7o)

The reaction time was 2 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.49 (s, 1H), 8.04 (s, 1H), 8.03 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 4.13 (s, 3H), 1.77 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.59, 158.23, 148.81, 148.35, 139.63, 136.52, 136.16, 129.08, 128.43, 122.18, 112.52, 63.45, 59.39, 29.35; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{18}H_{19}ClN_3O_2(M+H)^+$: 344.1160, found: 344.1167.



(E)-2-(tert-butyl)-3-(methoxyimino)-6-(naphthalen-2-yl)-2,3-dihydro-1H-pyrrolo[3,4-c]pyridin-1-one (7p)

The reaction time was 10 h. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.56 (d, *J* = 1.2 Hz, 1H), 8.59 (d, *J* = 1.2 Hz, 1H), 8.23-8.20 (m, 2H), 7.98-7.96 (m, 2H), 7.89-7.87 (m, 1H), 7.55-7.52 (m, 2H), 4.14 (s, 3H), 1.79 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.01, 159.69, 149.19, 148.61, 139.81, 135.61, 134.21, 133.51, 129.11, 128.88, 127.83, 127.41, 127.27, 126.70, 124.51, 122.21, 113.22, 63.66, 59.58, 29.60; **HRMS** (ESI-TOF) *m/z* Calcd for $C_{22}H_{22}N_3O_2(M+H)^+$: 360.1707, found: 360.1718.

4. References

(S1) Wrigglesworth, J. W., Cox, B., Lloyd-Jones, G. C. & Booker-Milburn, K. I. New Heteroannulation Reactions of N-Alkoxybenzamides by Pd(II) Catalyzed C−H Activation. *Org. Lett.* **13**, 5326–5329 (2011).

(S2) Guimond, N., Gouliaras, C. & Fagnou, K. Rhodium(III)-Catalyzed Isoquinolone Synthesis: The N−O Bond as a Handle for C−N Bond Formation and Catalyst Turnover. *J. Am. Chem. Soc.* ***132***, 6908-6909 (2010**)**.

(S3) Rakshit, S., Grohmann, C., Besset, T. & Glorius, F. Rh(III)-Catalyzed Directed C-H Olefination Using an Oxidizing Directing Group: Mild, Efficient, and Versatile. *J. Am. Chem. Soc.* **133**, 2350-2353 (2011).

(S4) Zhong, H. B., Yang, D., Wang, S. Q. & Hang, J. H. Pd-catalyzed synthesis of isoquinolinones and analogues *via* C–H and N–H bonds double activation. *Chem. Commun.* **48**, 3236-3238 (2012).

(S5) Li, B. *et al*. Ruthenium-Catalyzed Oxidative C–H bonds Olefination of N-Methoxybenzamide Using an Oxidizing Directing Group. *Org. Lett.* **14**, 736-739 (2012).

(S6) Kukosha, T., Trufilkina, N., Belyakov, S. & Katkevics, M. Cooper-Catalyzed Cross-Coupling of *O*-Alkyl Hydroxamates with Aryl Iodides. *Synthesis* **44**, 2413-2423 (2012).

(S7) Li, C. L., Li, P. H., Yang, J. & Wang, L. Palladium-catalyzed deamiedative arylation of azoles with arylamides though a tandem decarbonylation-C–H functionalization. *Chem. Commun.* **48**, 4214-4216 (2012).

(S8) Zeng, R., Fu, C. L. & Ma, S. M. Highly Selective Mild Stepwise Allylation of N-Methoxybenzamides with Allenes. *J. Am. Chem. Soc.* **134**, 9597-9600 (2012).

(S9) Grohmann, C., Wang, H. G. & Glorius, F. Rh[III]-Catalyzed C–H Amidation Using Aroylxycarbamates To Give N-Boc Protected Arylamines. *Org. Lett.* **15**, 3014-3017 (2013).

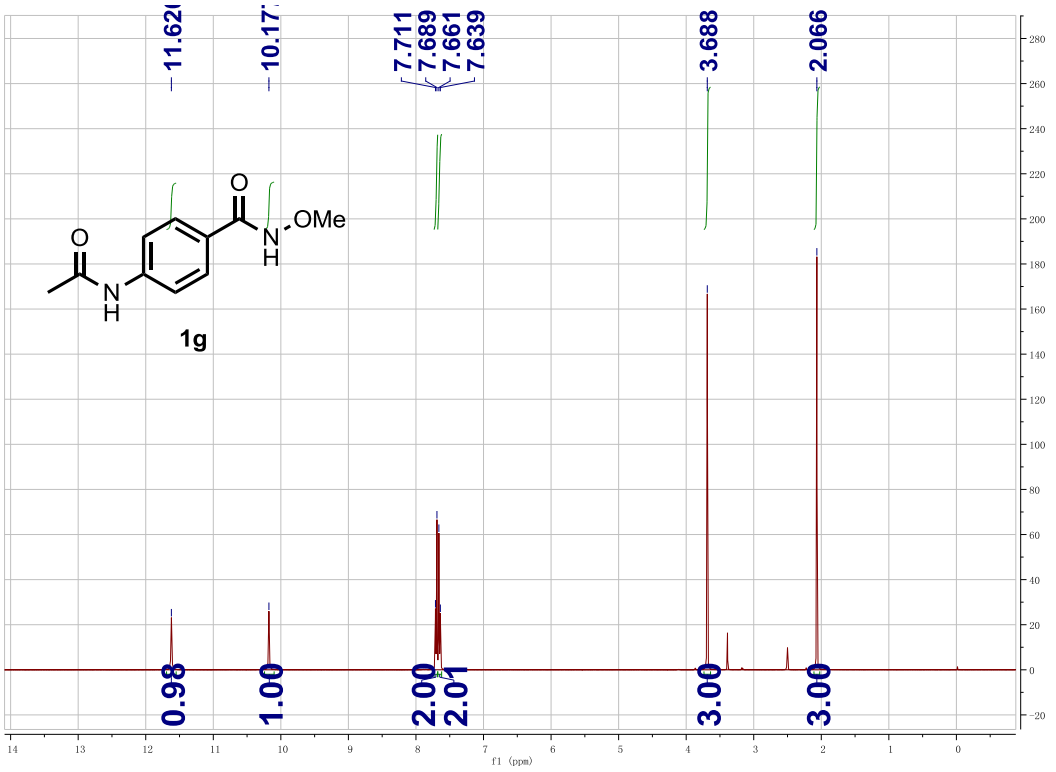
(S10) German, L. V., Simon, O. U., Alejandra, M. O., Rocio, G. M. & Luis, D. M. Convenient access to isoindolinones via carbamoyl radical cyclization. Synthesis of cichorine and 4-hydroxyisoindolin-1-one natural products. *Tetrahedron* **67**, 2693-2701 (2011).

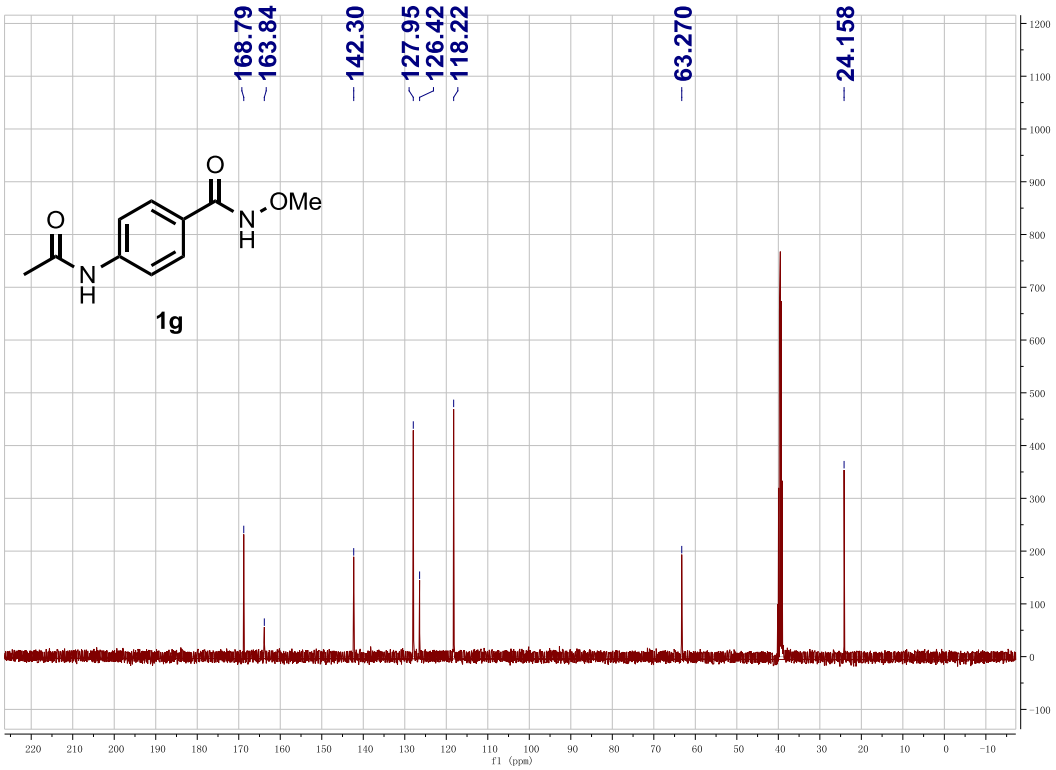
(S11) Clift, M. D. & Silverman, R. B. Synthesis and evaluation of novel aromatic substrates and competitive inhibitors of GABA aminotransferase. *Bioorg. Med. Chem. Lett.* **18**, 3122-3125 (2008).

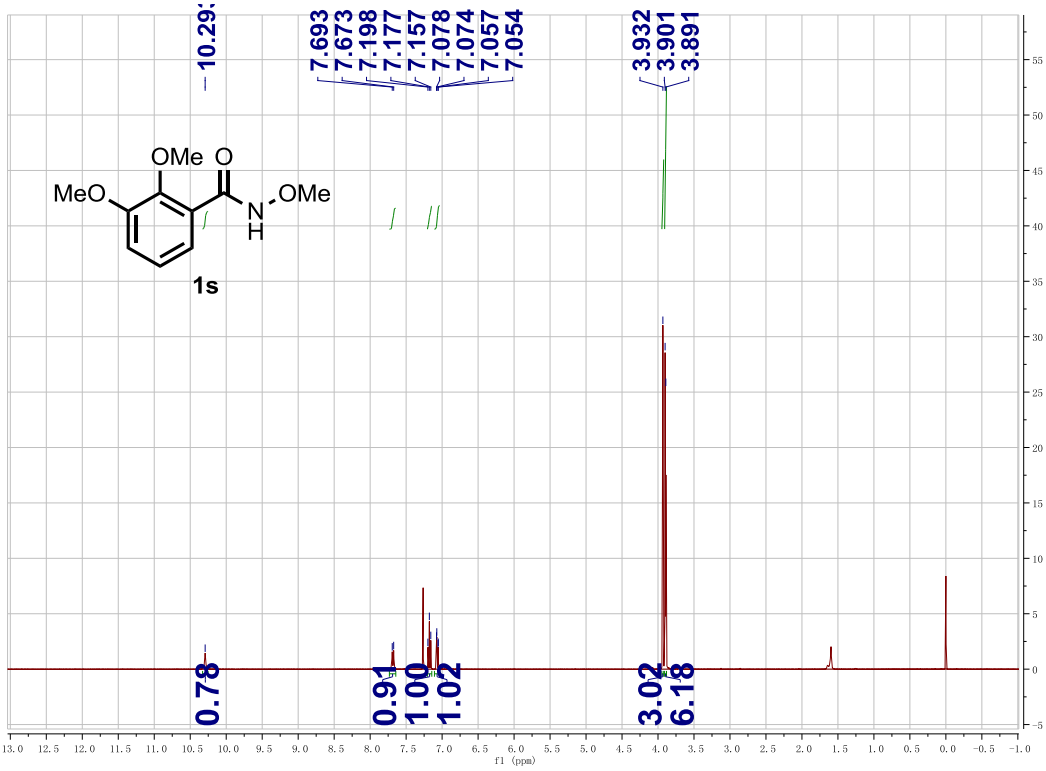
(S12) Kauffman, J. M. & Moyna, G. Diarylamino Groups as Photostable Auxofluors in 2-Benzoxazolylfluorene, 2,5-Diphenyloxazoles, 1,3,5-Hexatrienes, 1,4-Distyrylbenzenes, and 2,7-Distyrylfluorenes. *J. Org. Chem.* **68**, 839-853 (2003).

(S13) Zhang, C. Y. *et al*. Tetrahydroquinoline derivatives as opioid receptor antagonists. *Bioorg. Med. Chem. Lett.* **21**, 670-676 (2011).

5. NMR Spectra for New Compounds



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7.074
7.057
7.054
3.932
3.901
3.891
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MeO
N
H
OMe
1s
0.78
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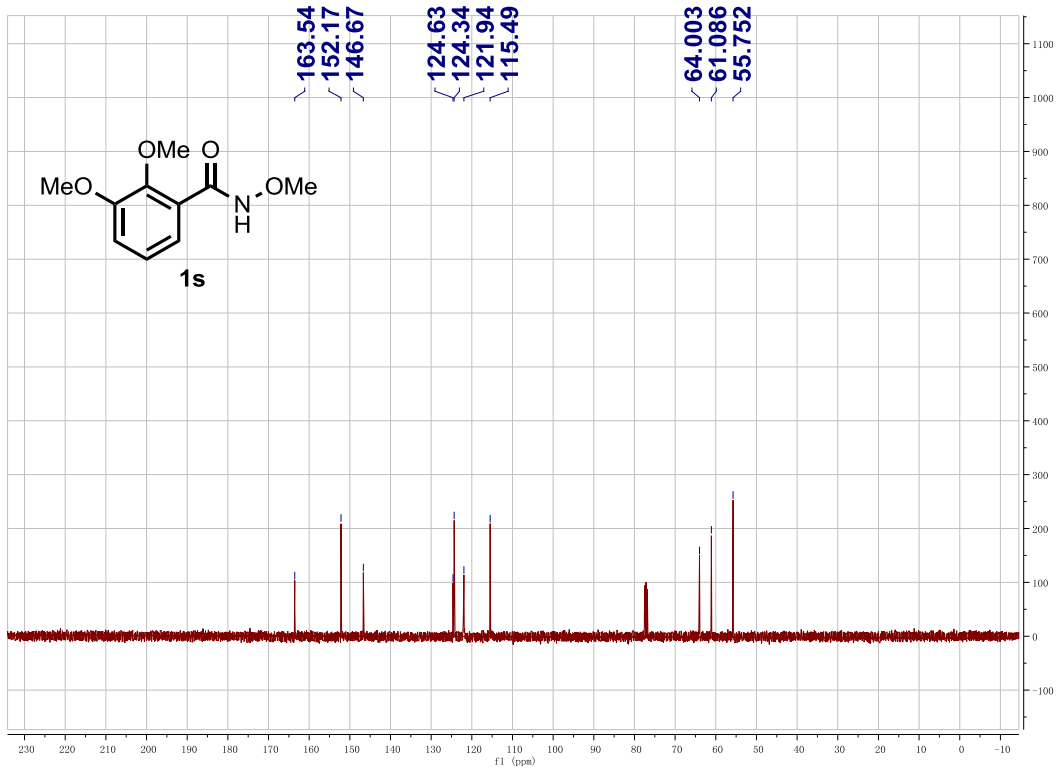
1.00
1.02
3.02
6.18
f1 (ppm)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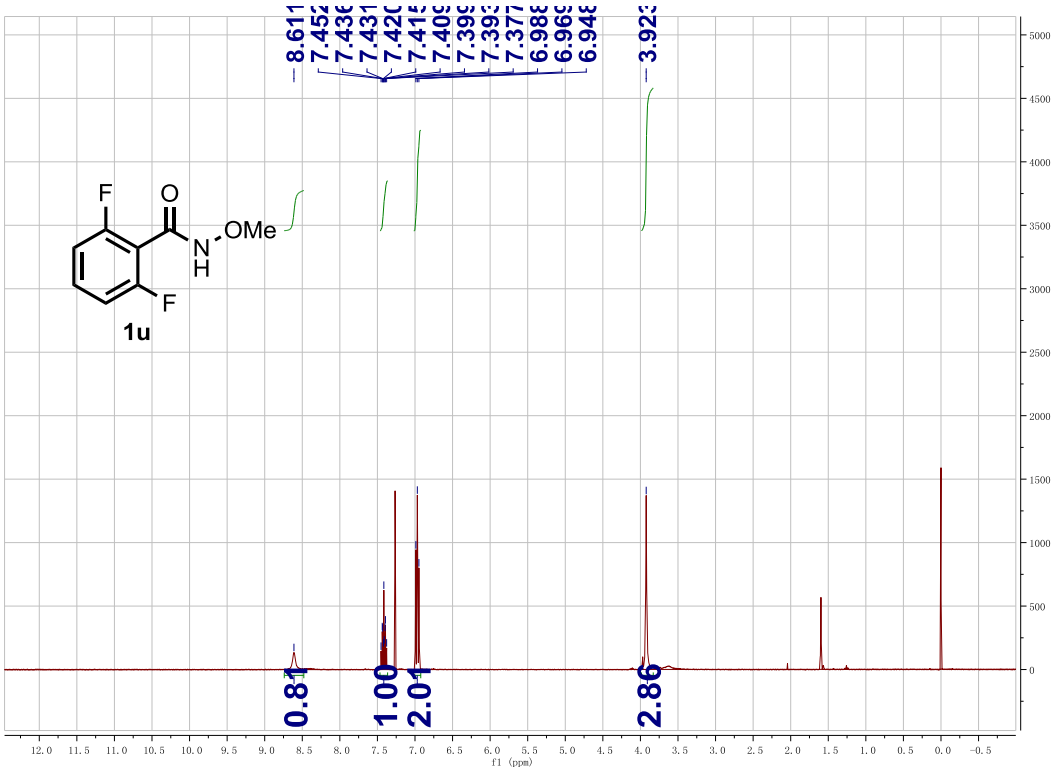
N
H
OMe
1s
f1 (ppm)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F
O
N
H
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F
1u
0.81
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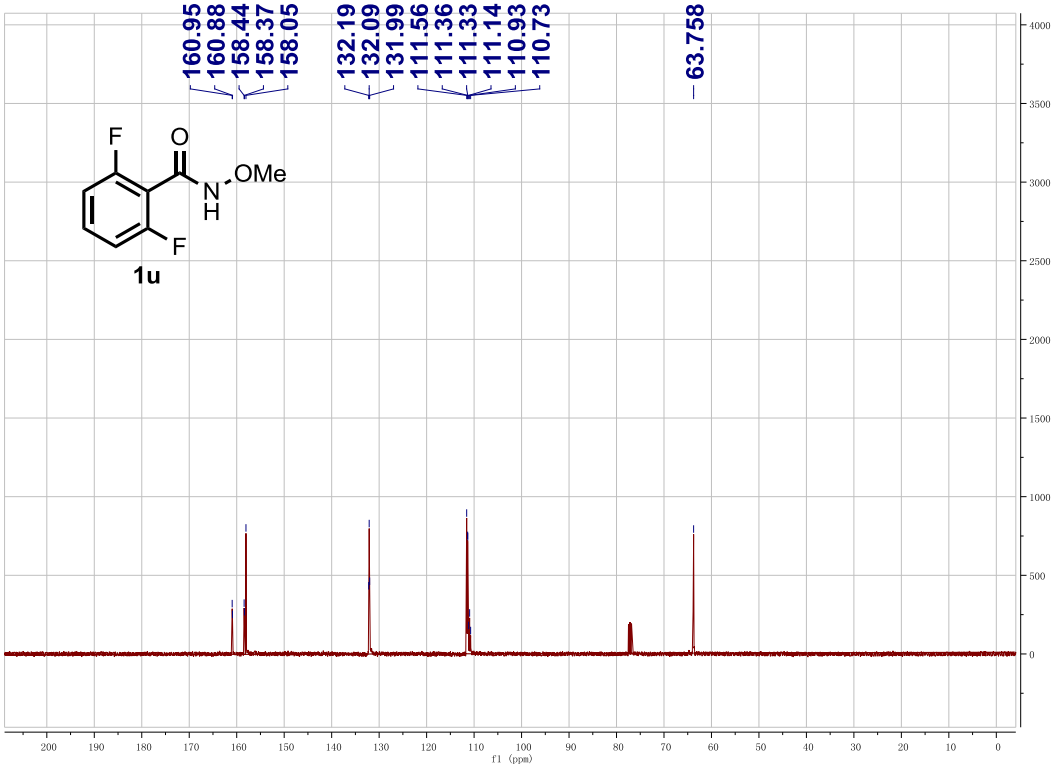
2.01
2.86
f1 (ppm)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N
H
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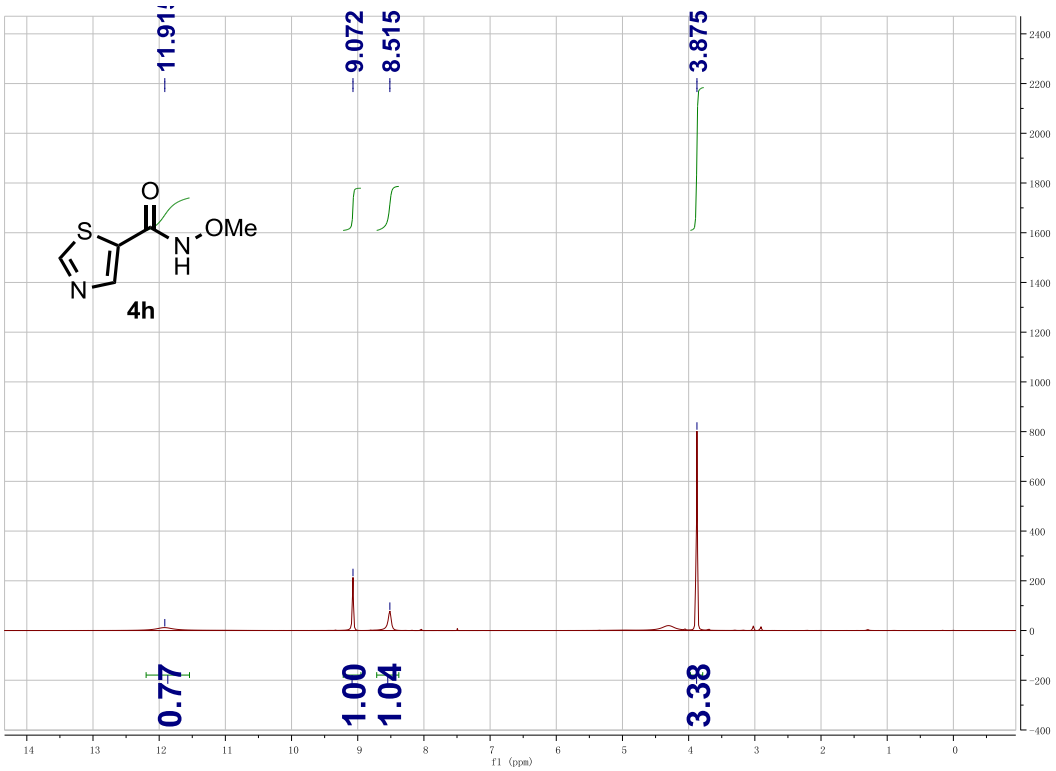
F
1u
f1 (ppm)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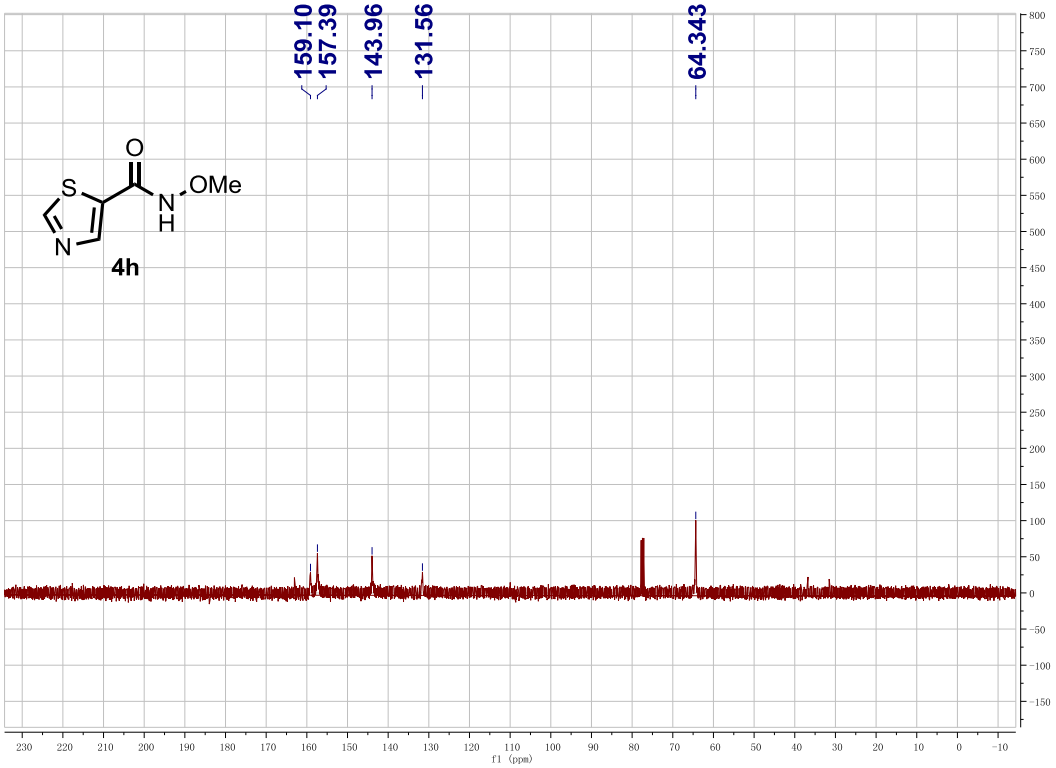
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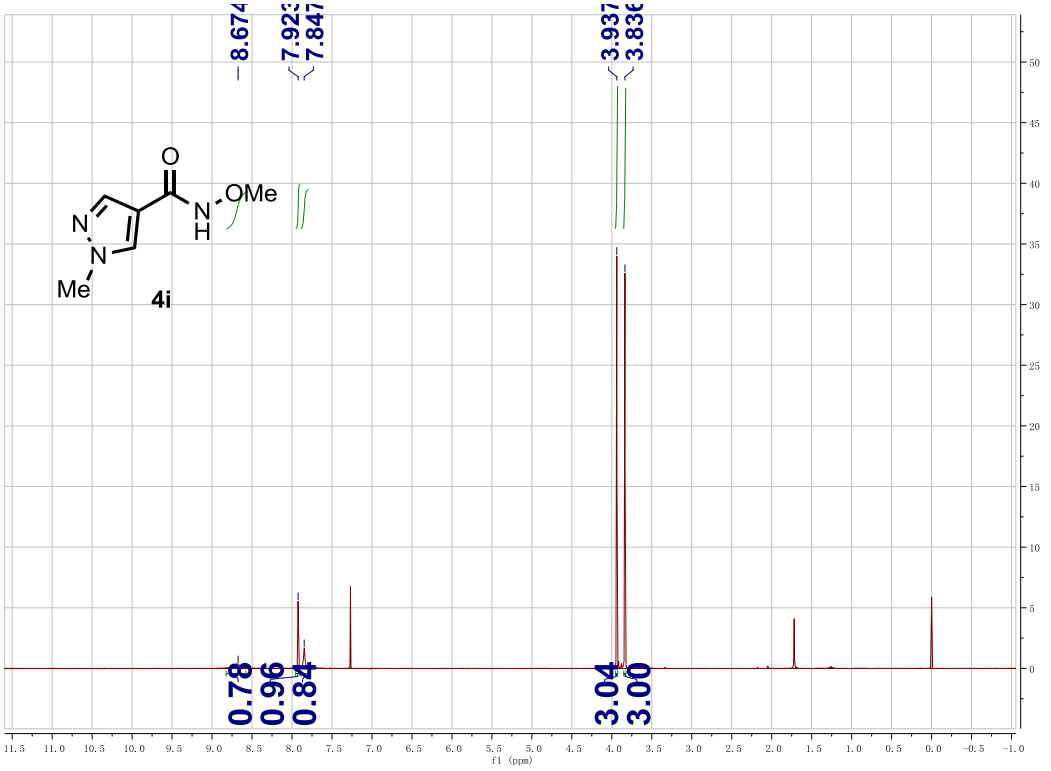
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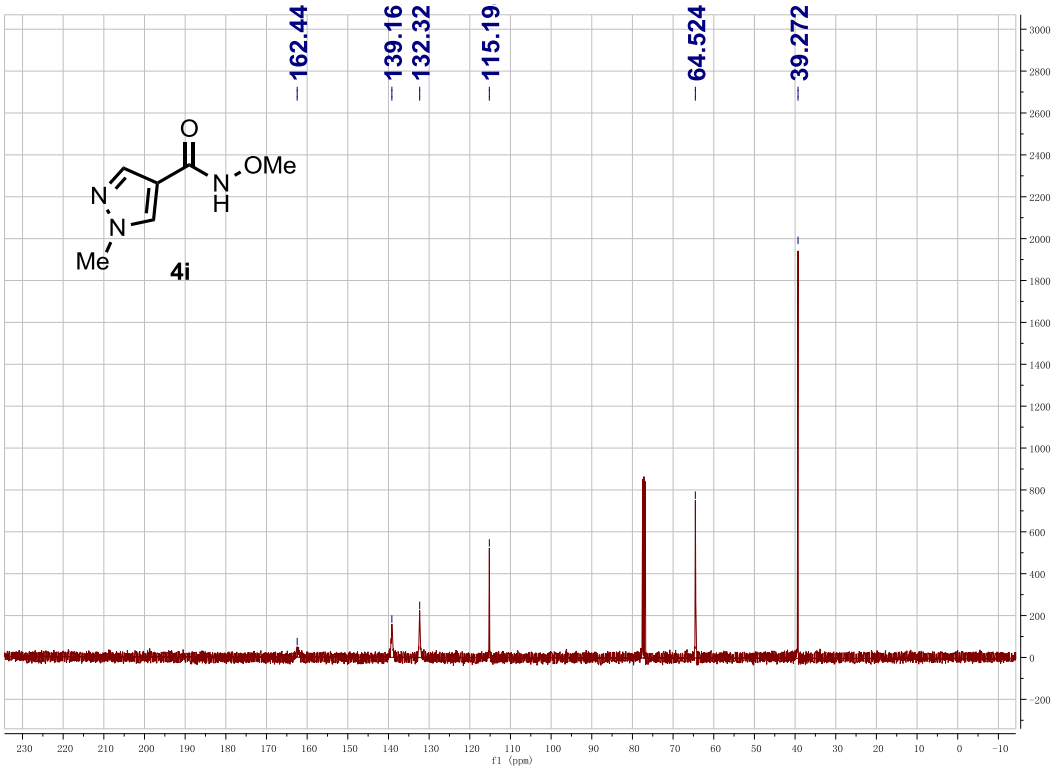
f1 (ppm)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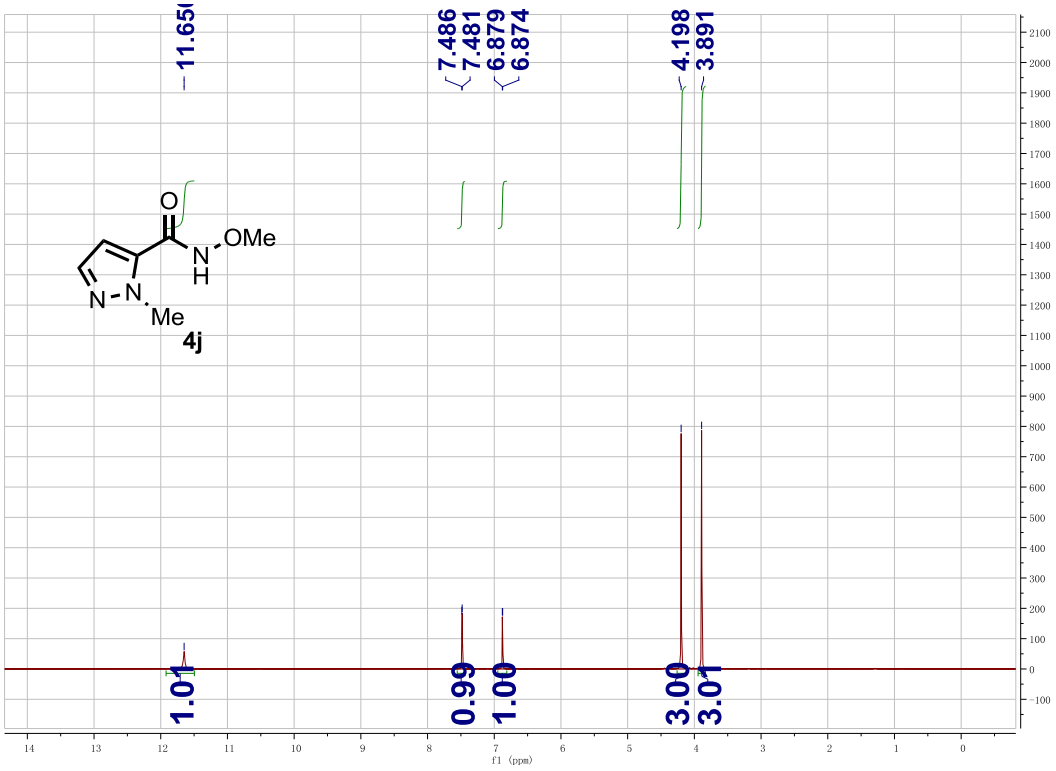
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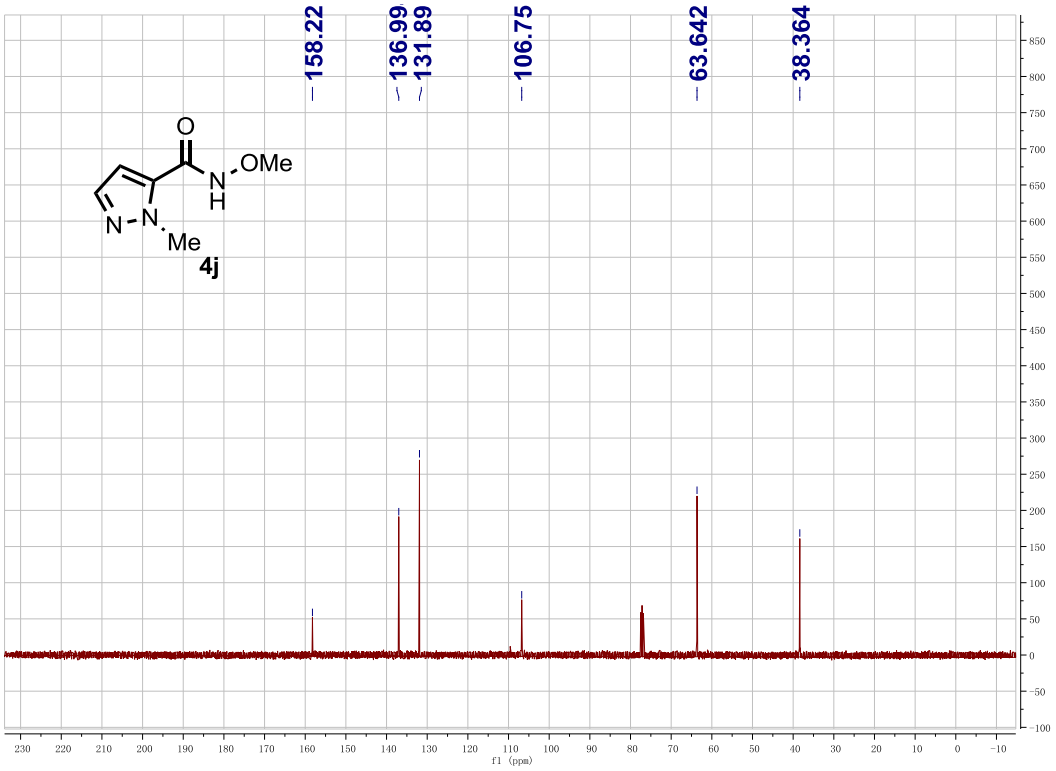
3.01
f1 (ppm)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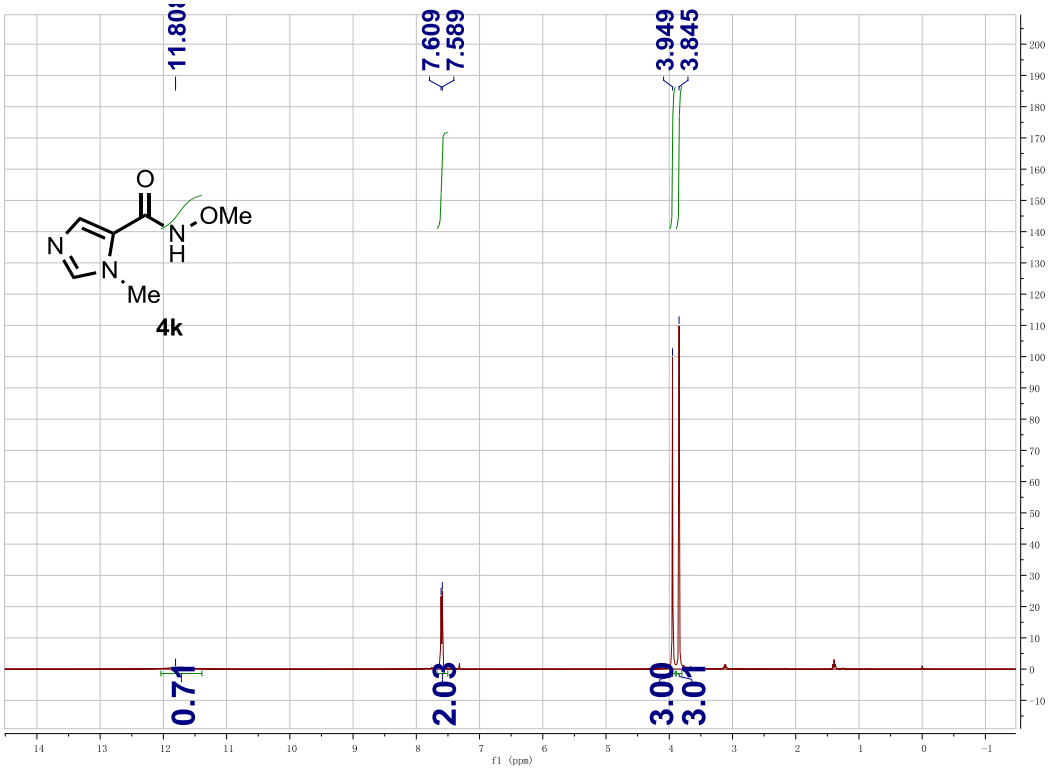
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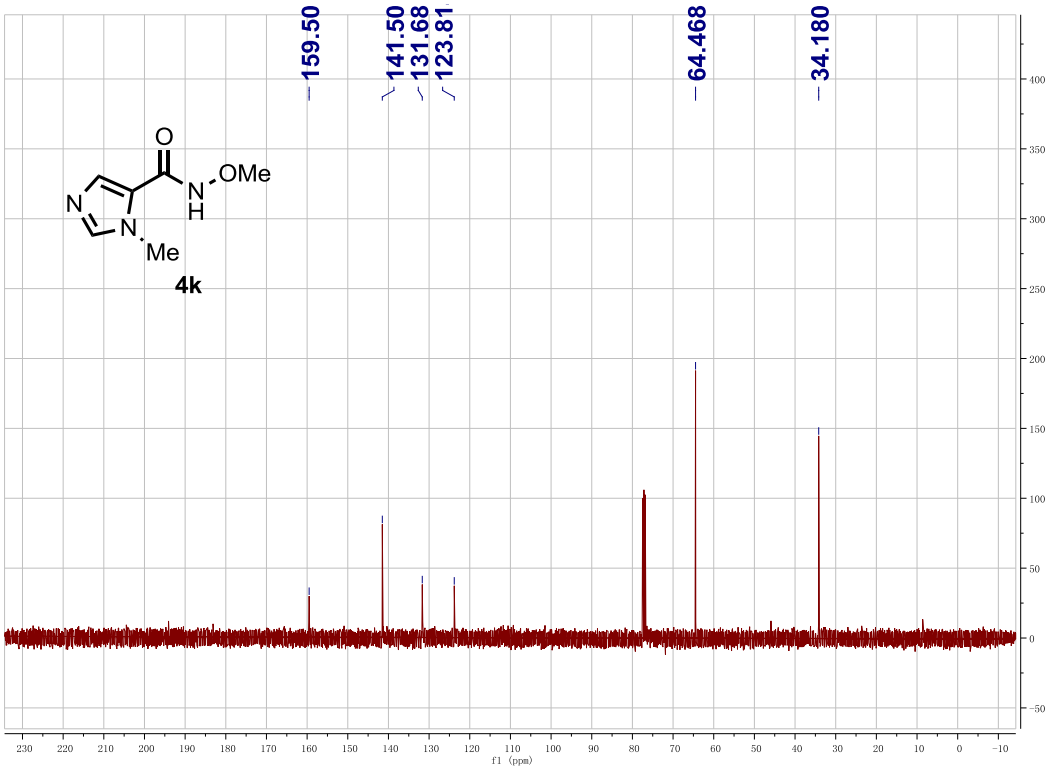
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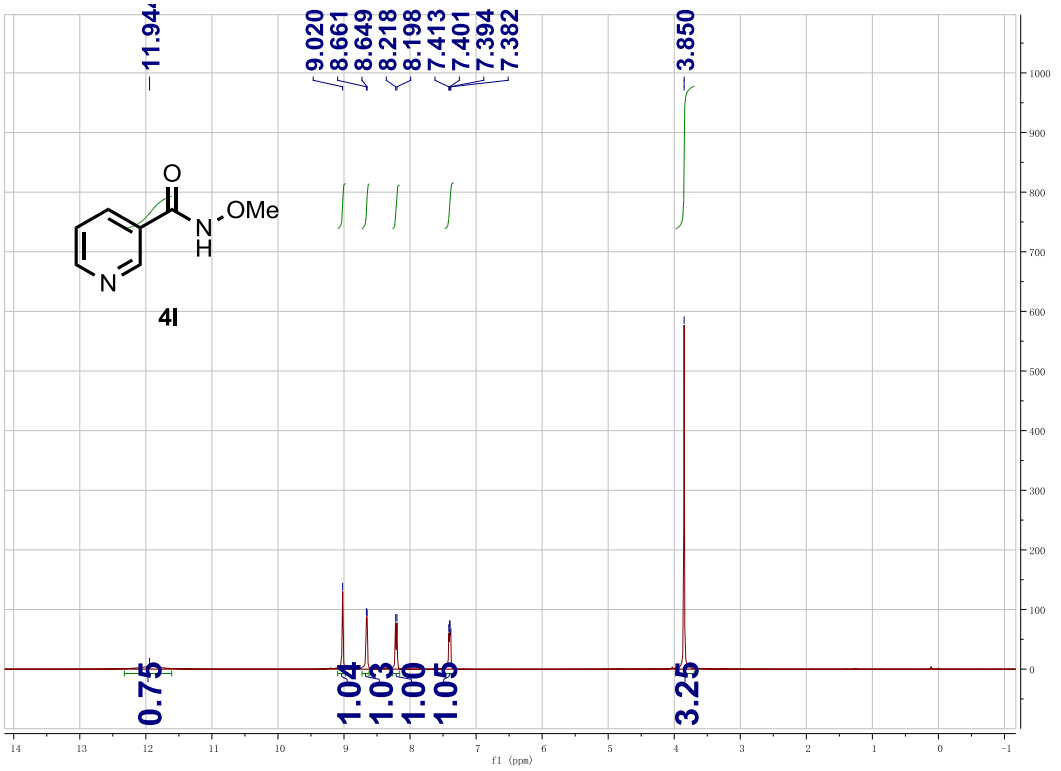
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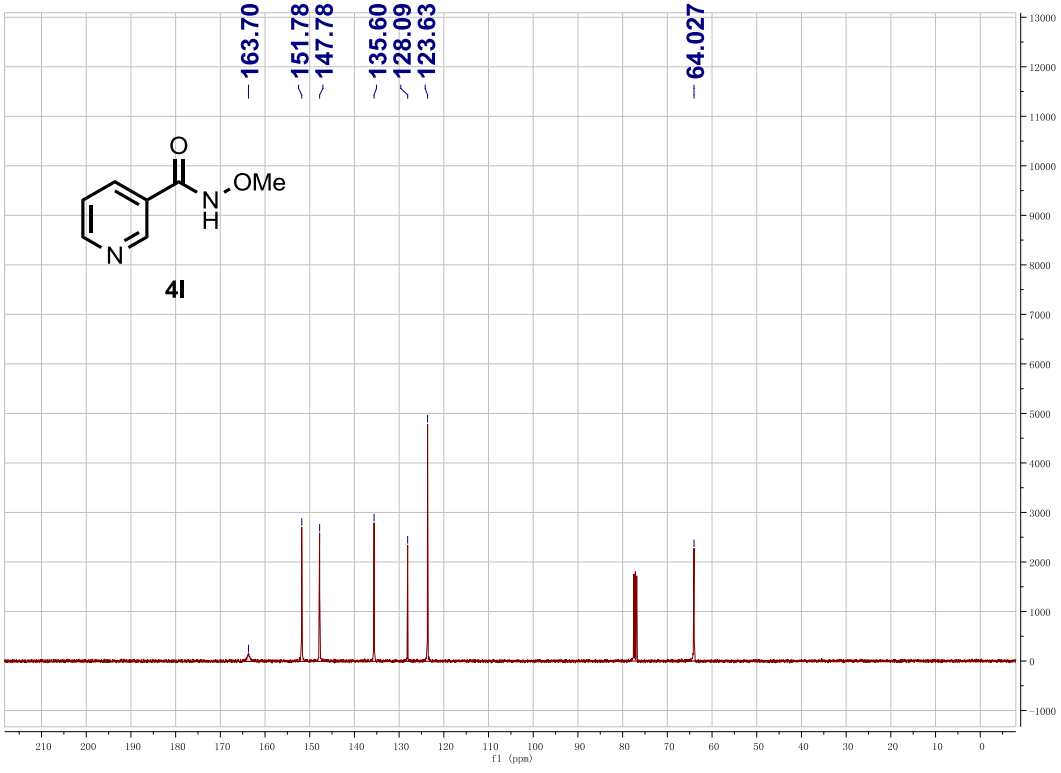
f1 (ppm)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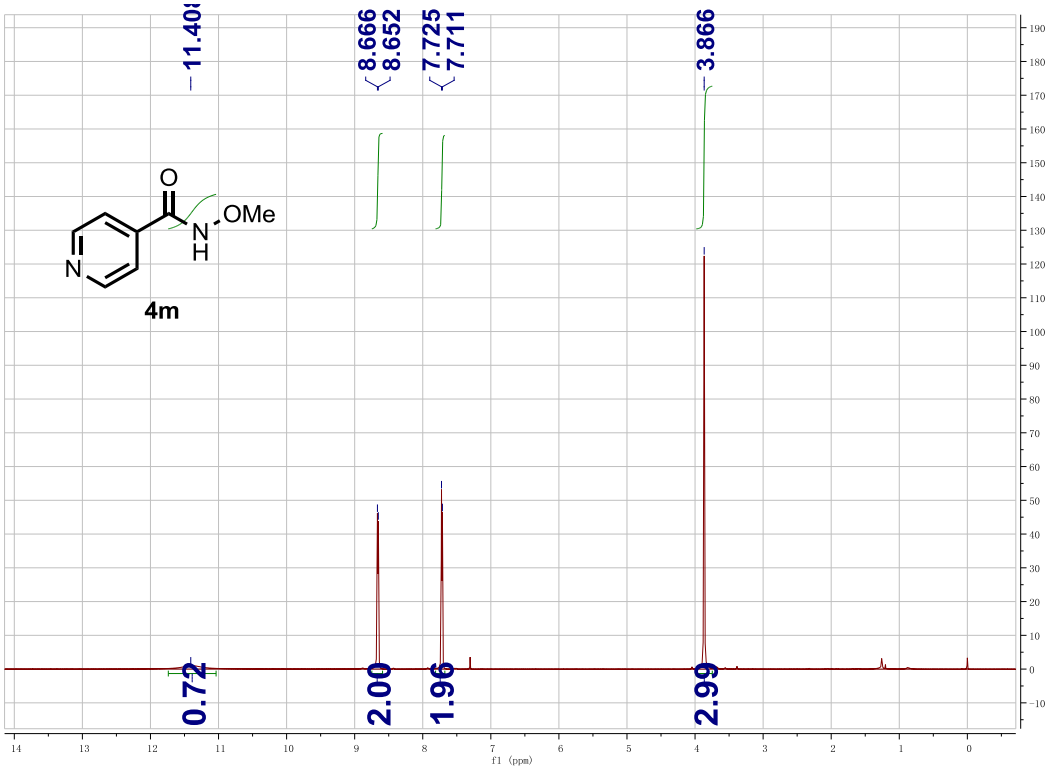
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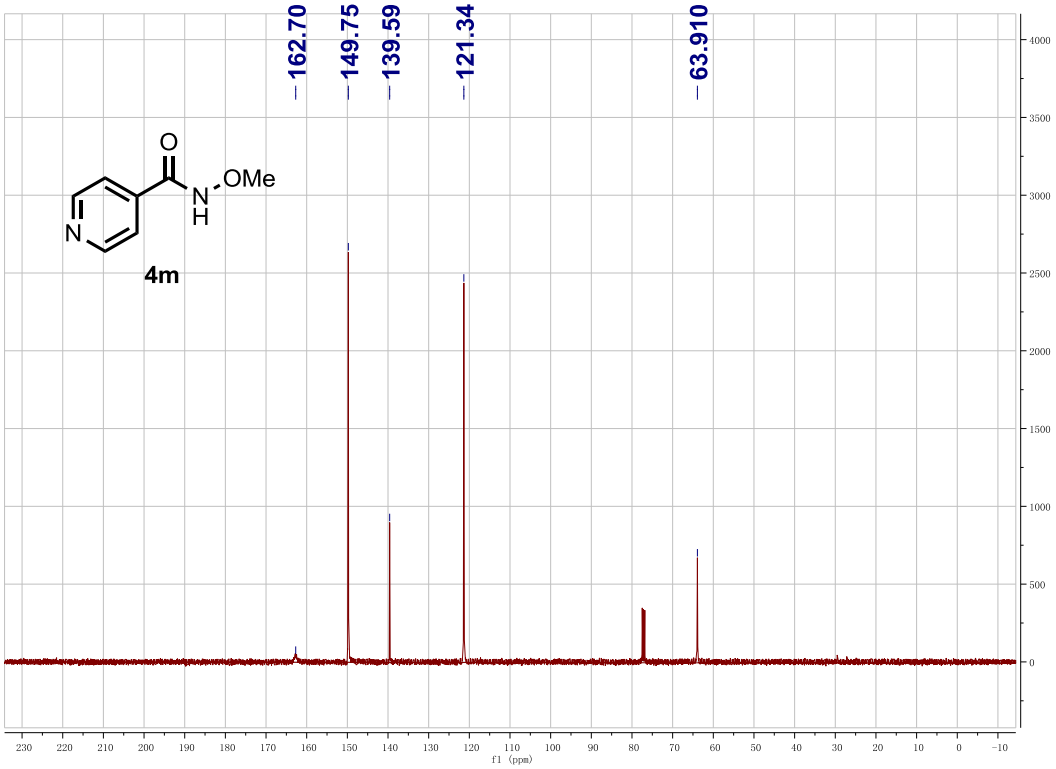
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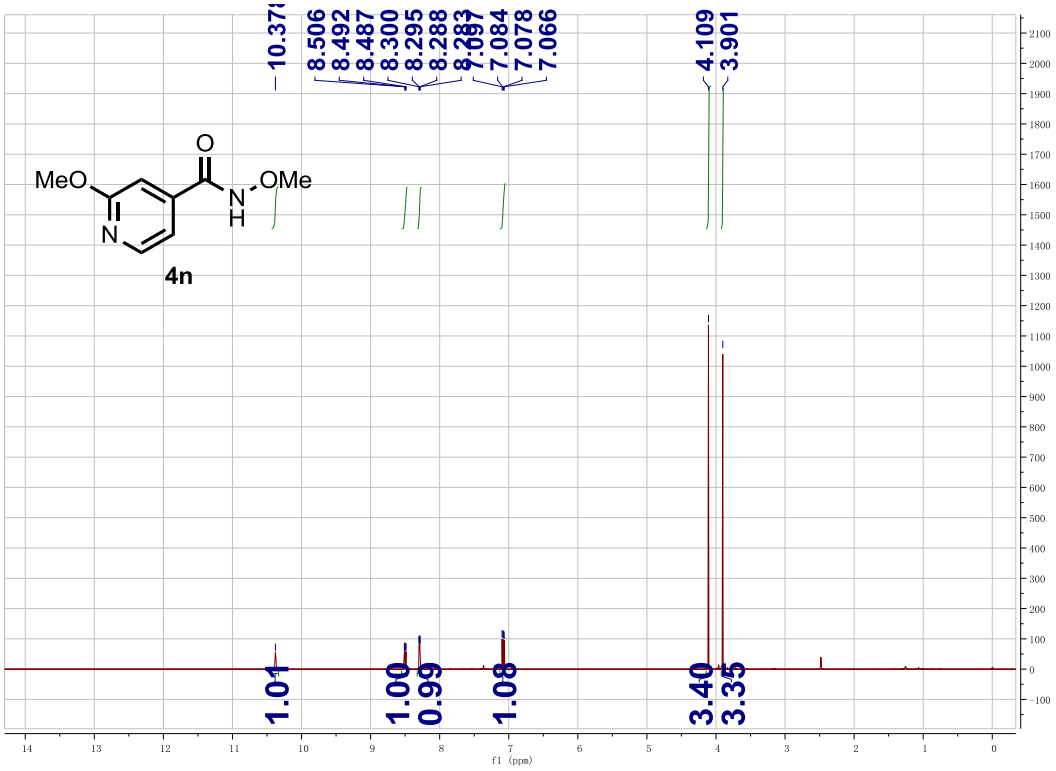
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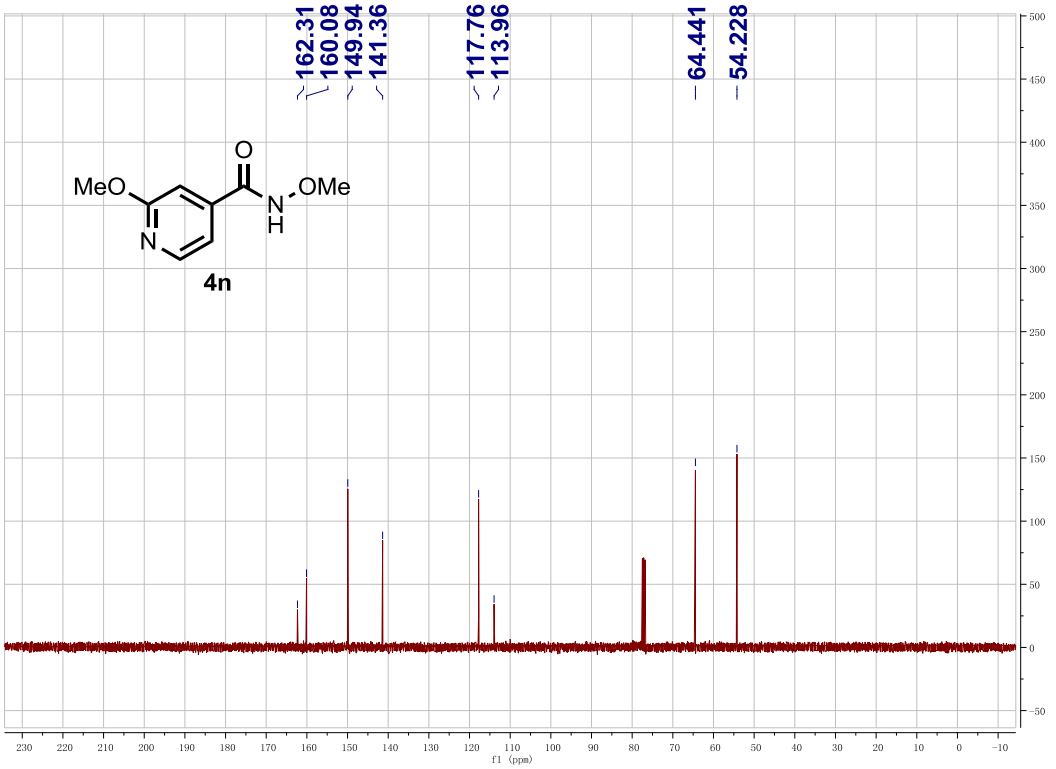
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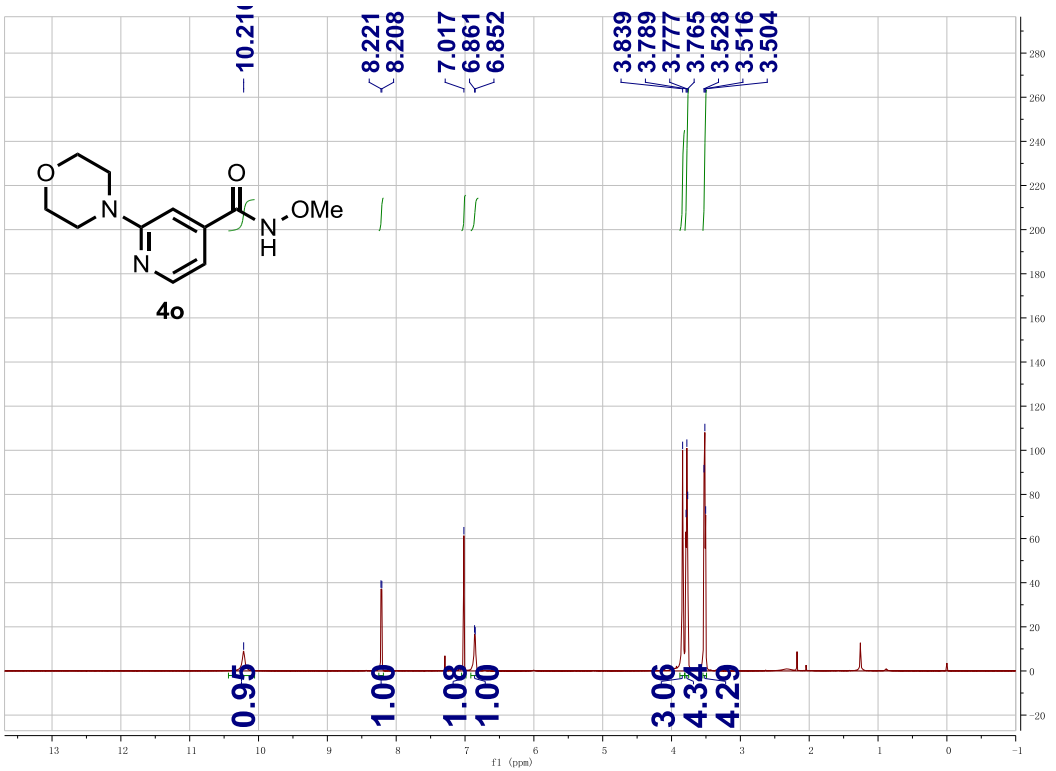
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O
N
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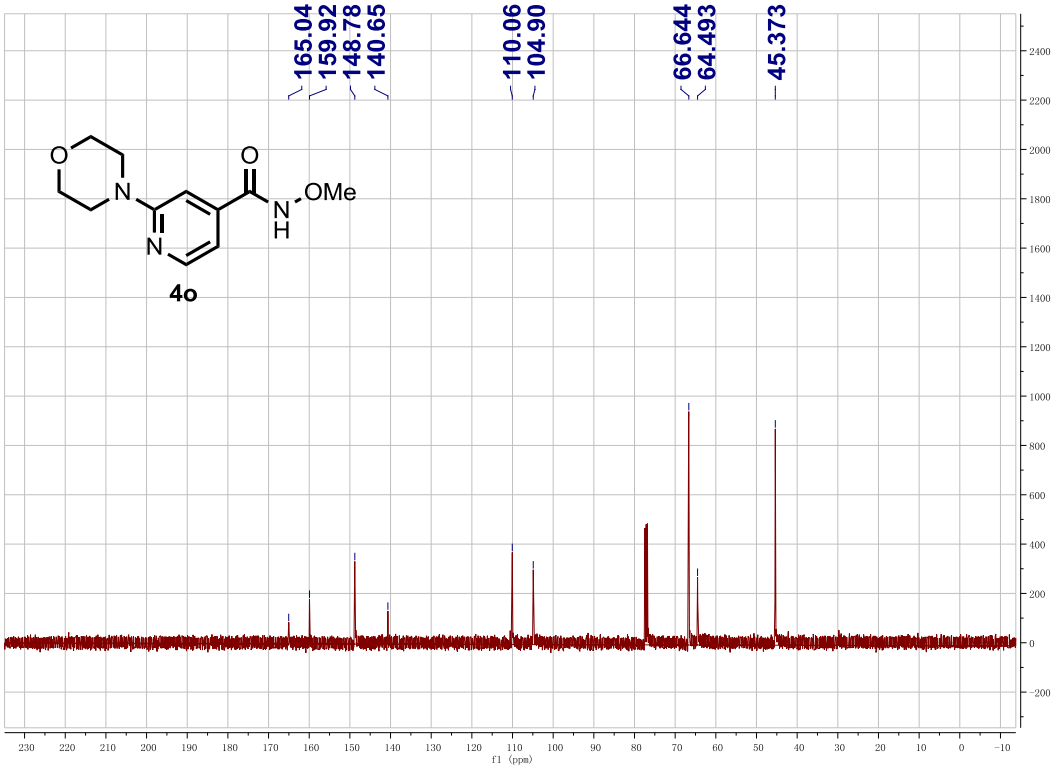
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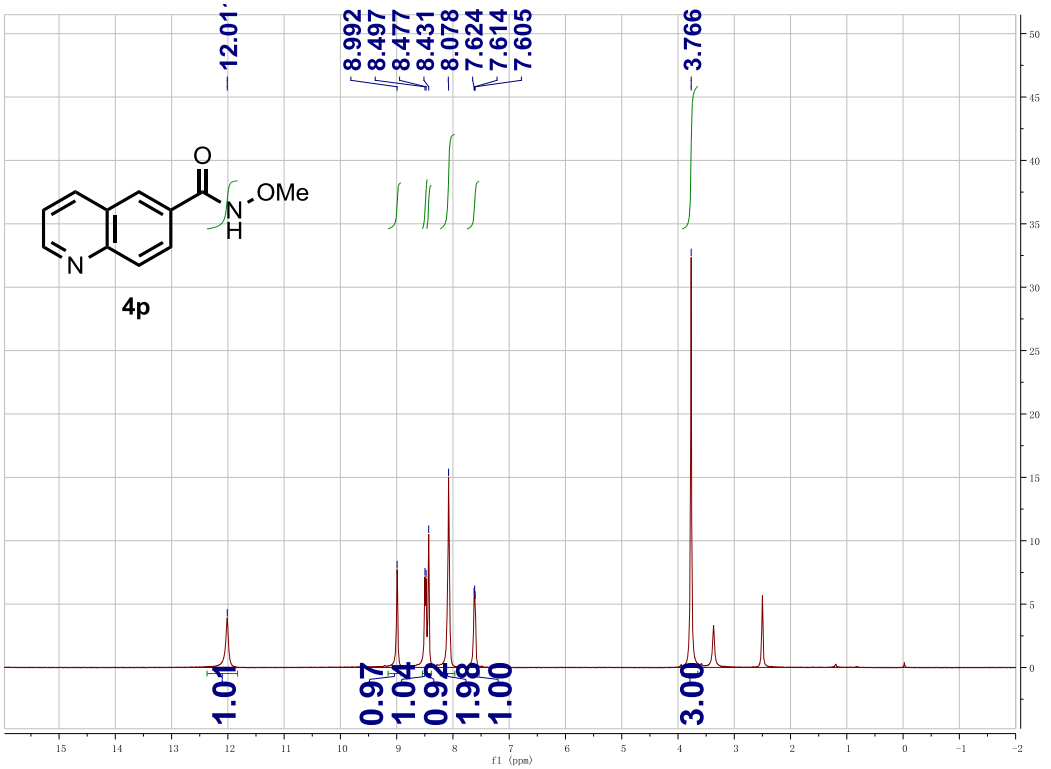
f1 (ppm)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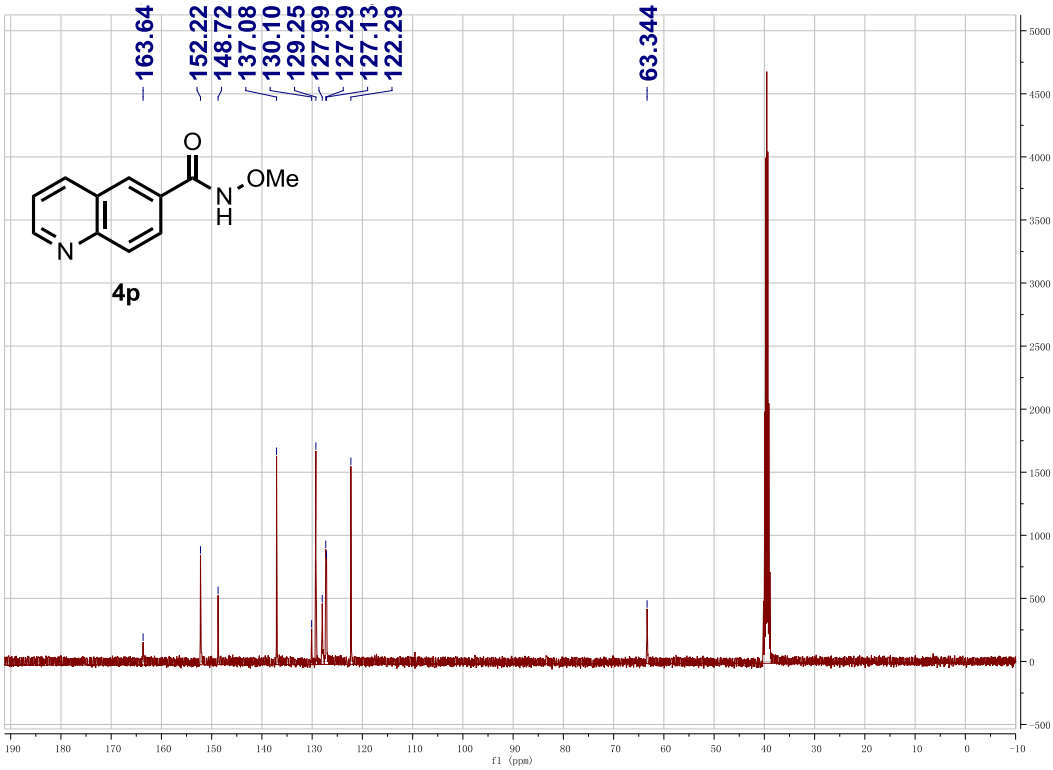
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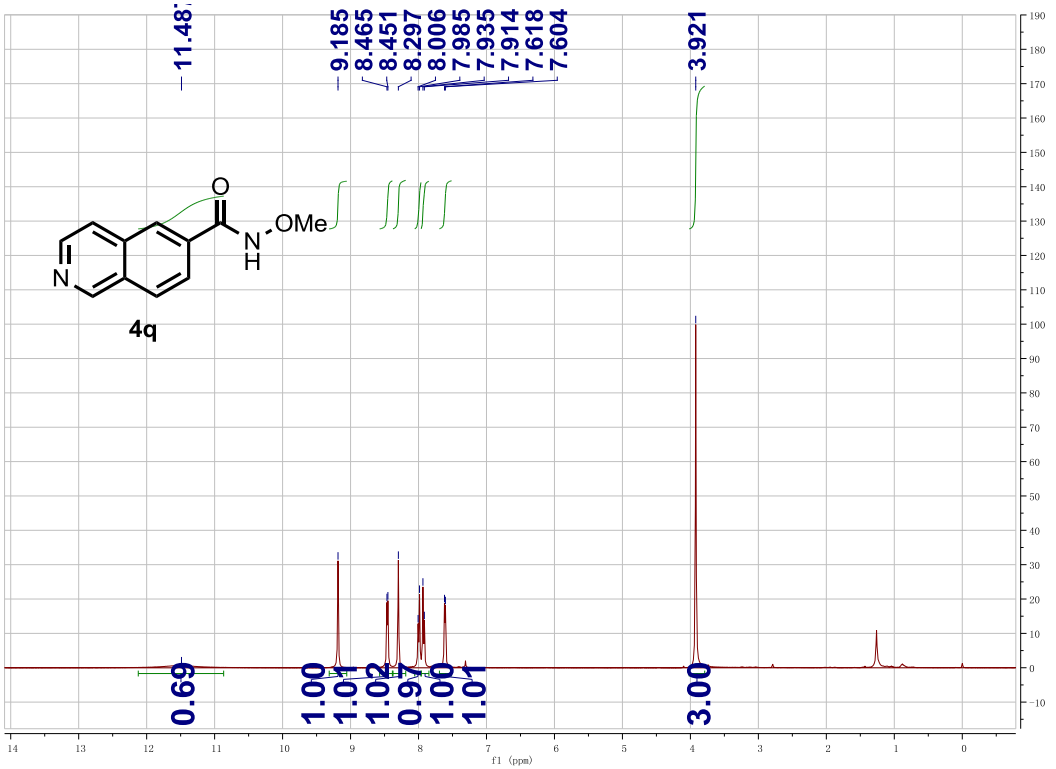
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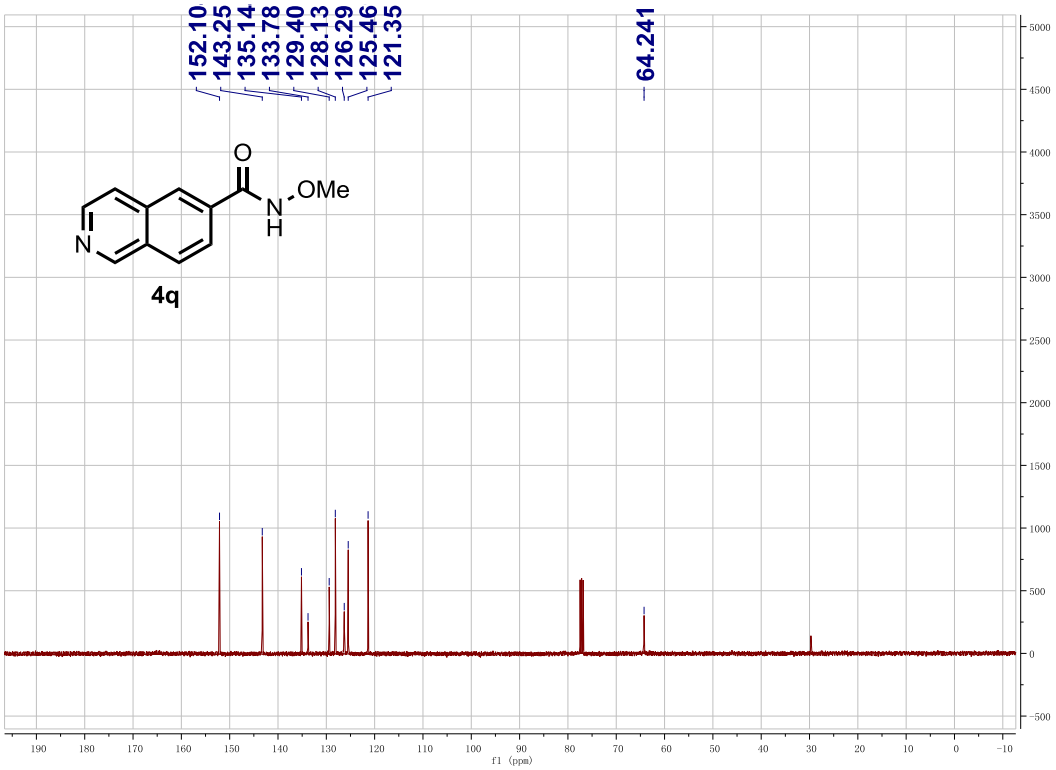
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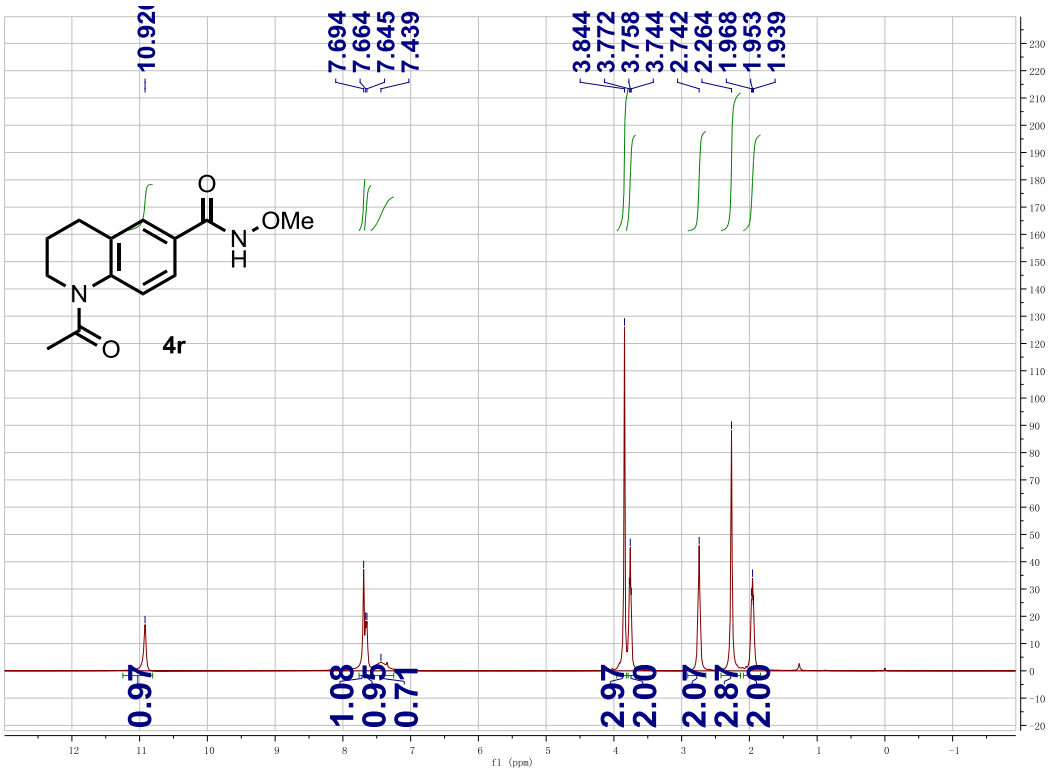
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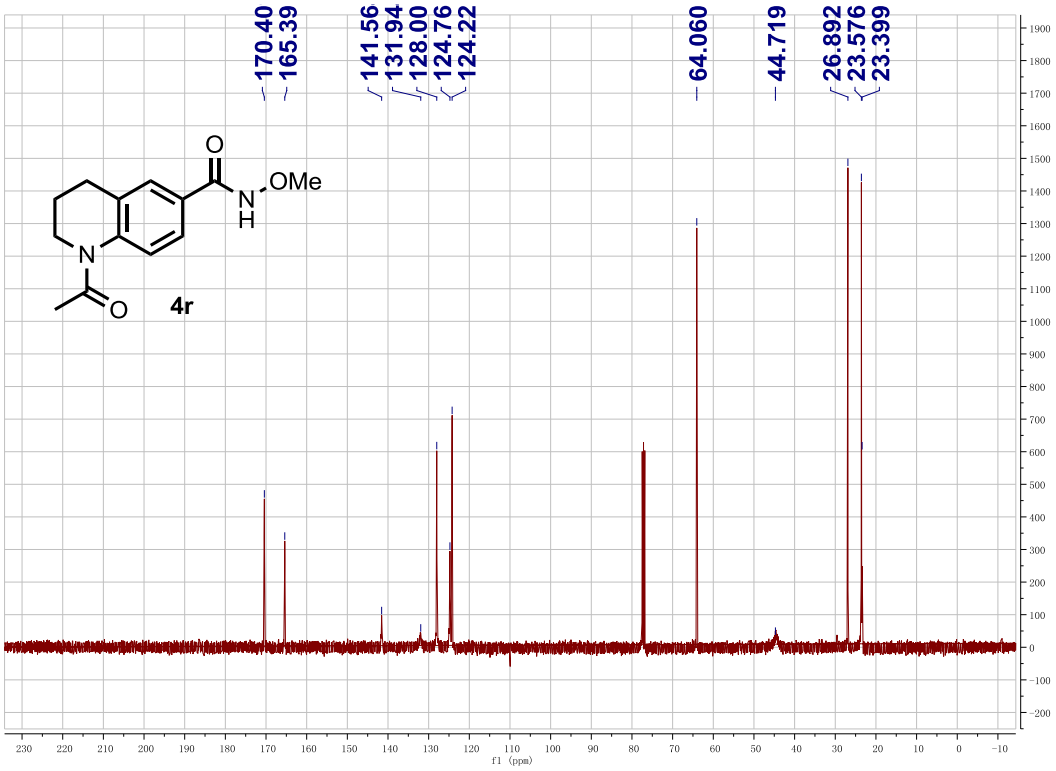
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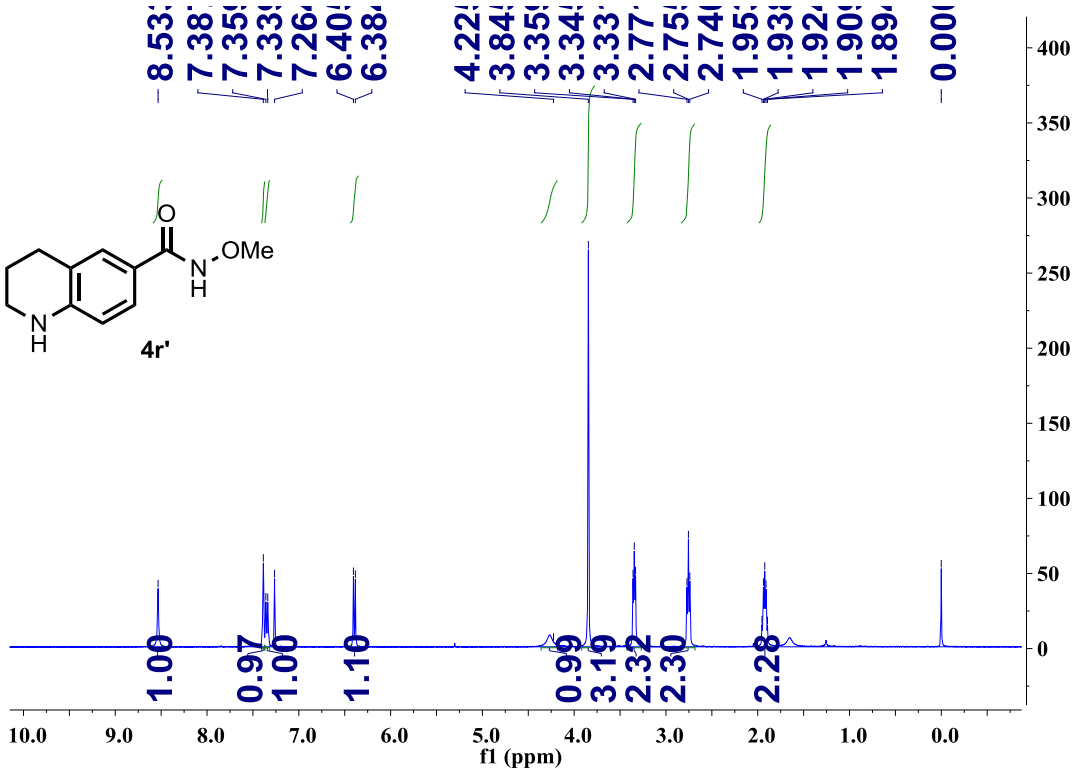
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1.89
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N
OMe
H
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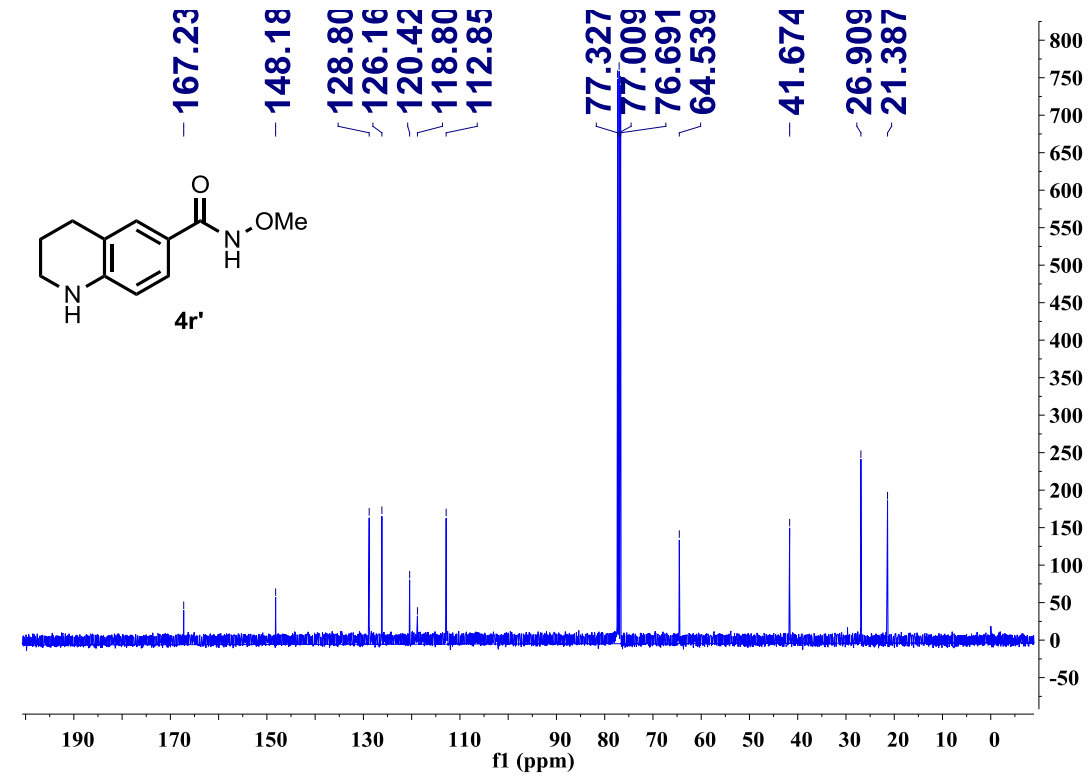
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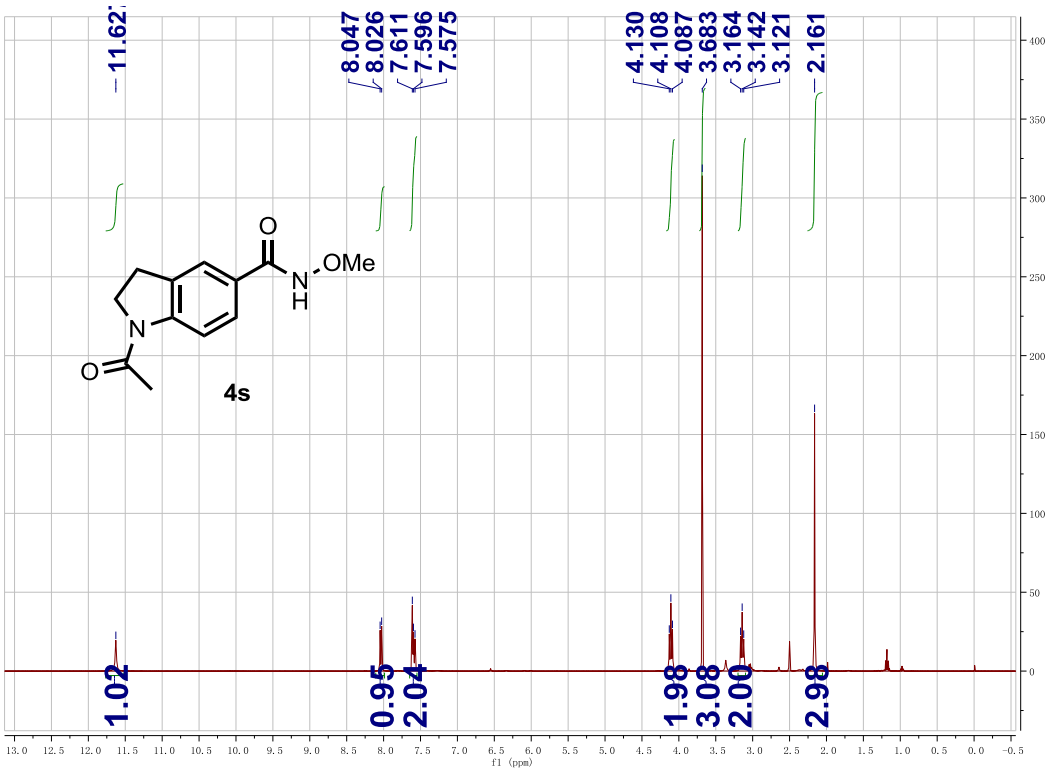
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2.161
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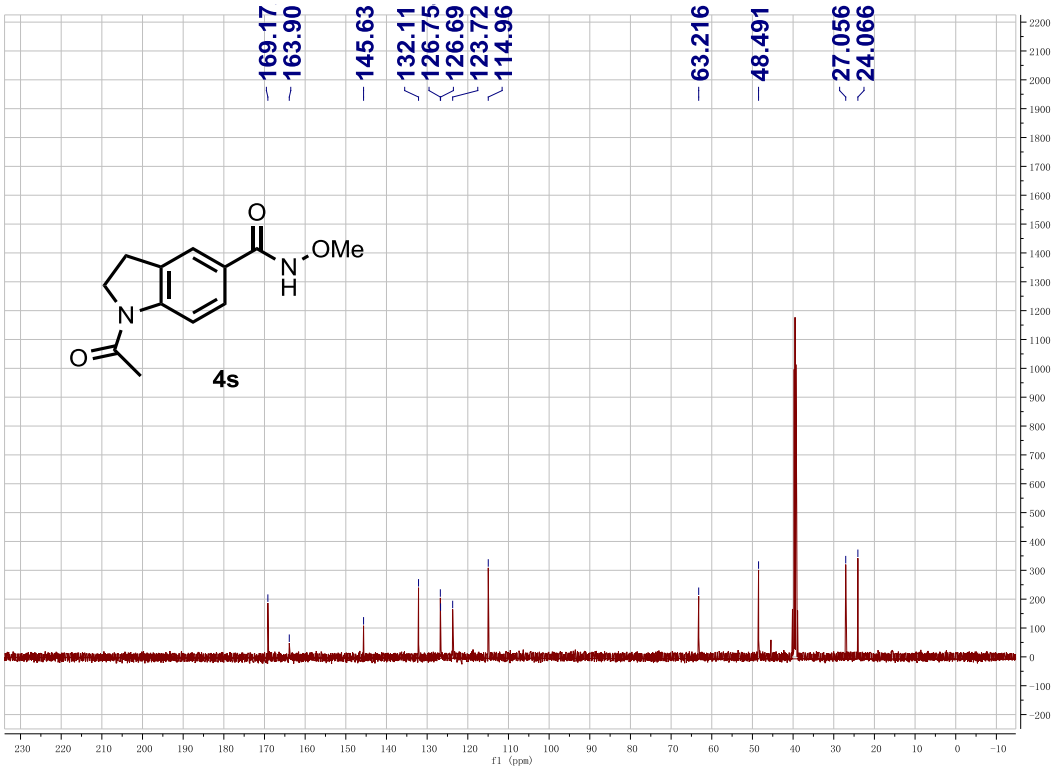
f1 (ppm)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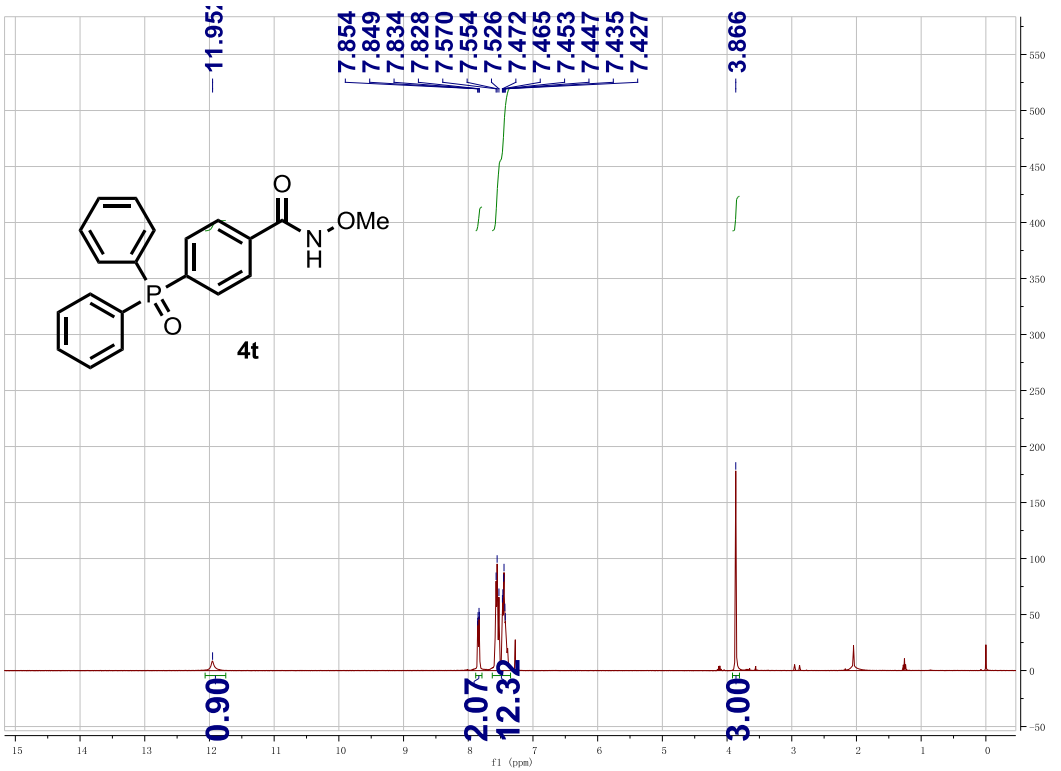
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7.435
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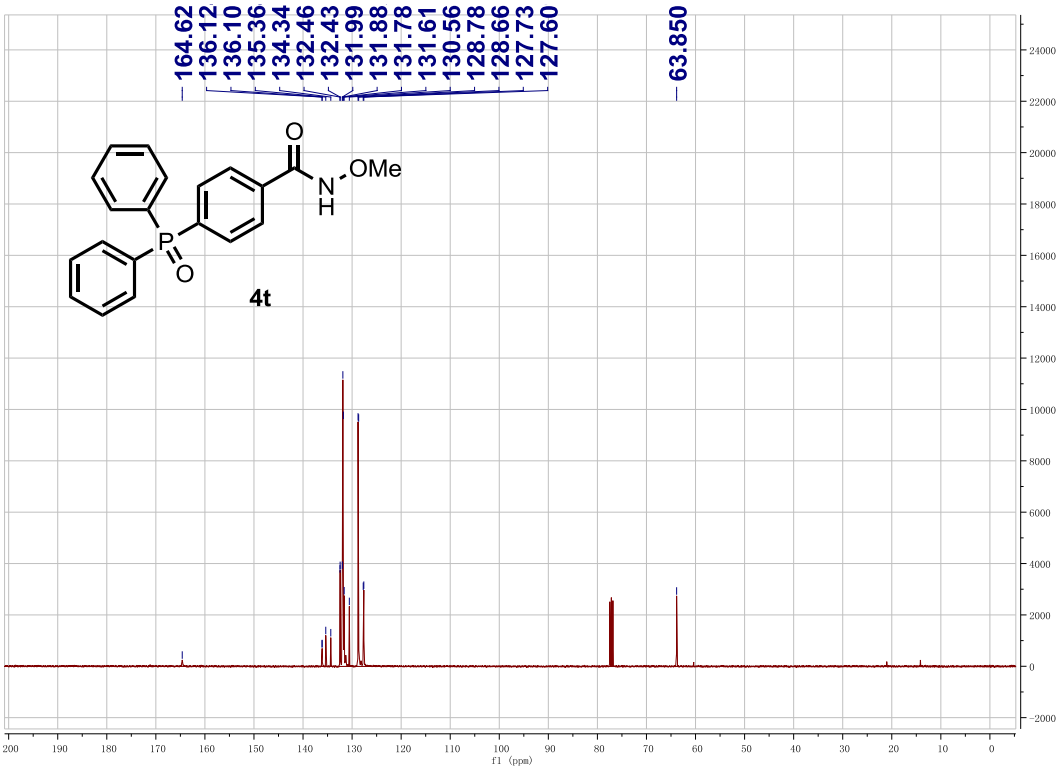
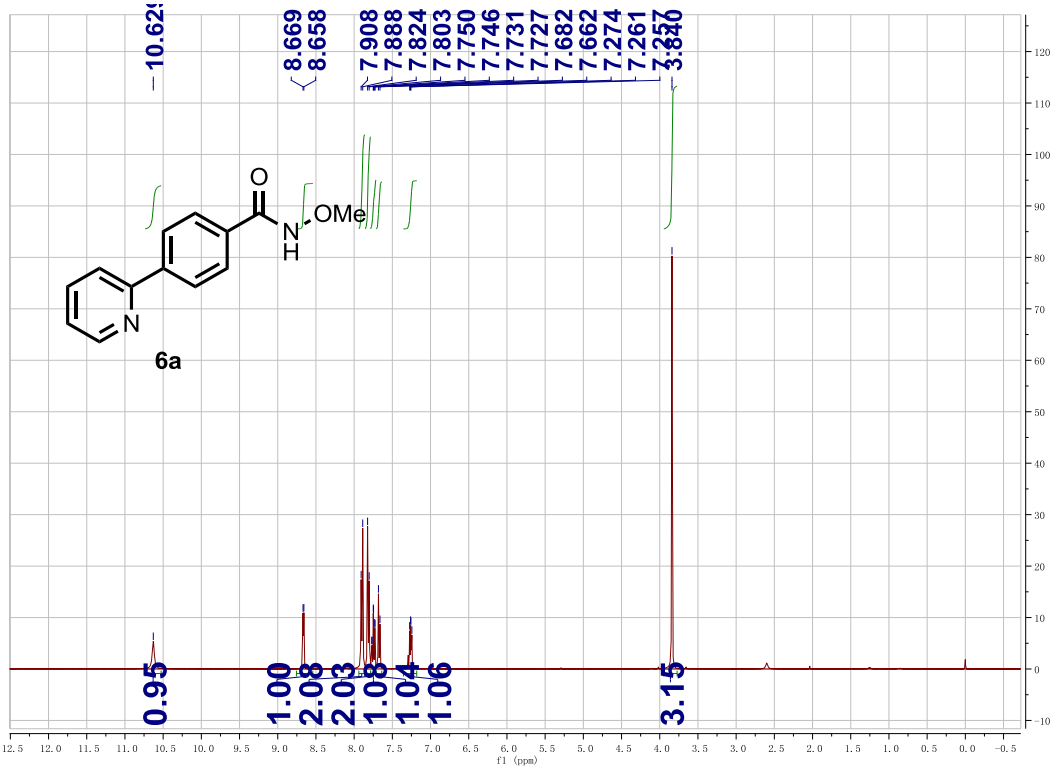
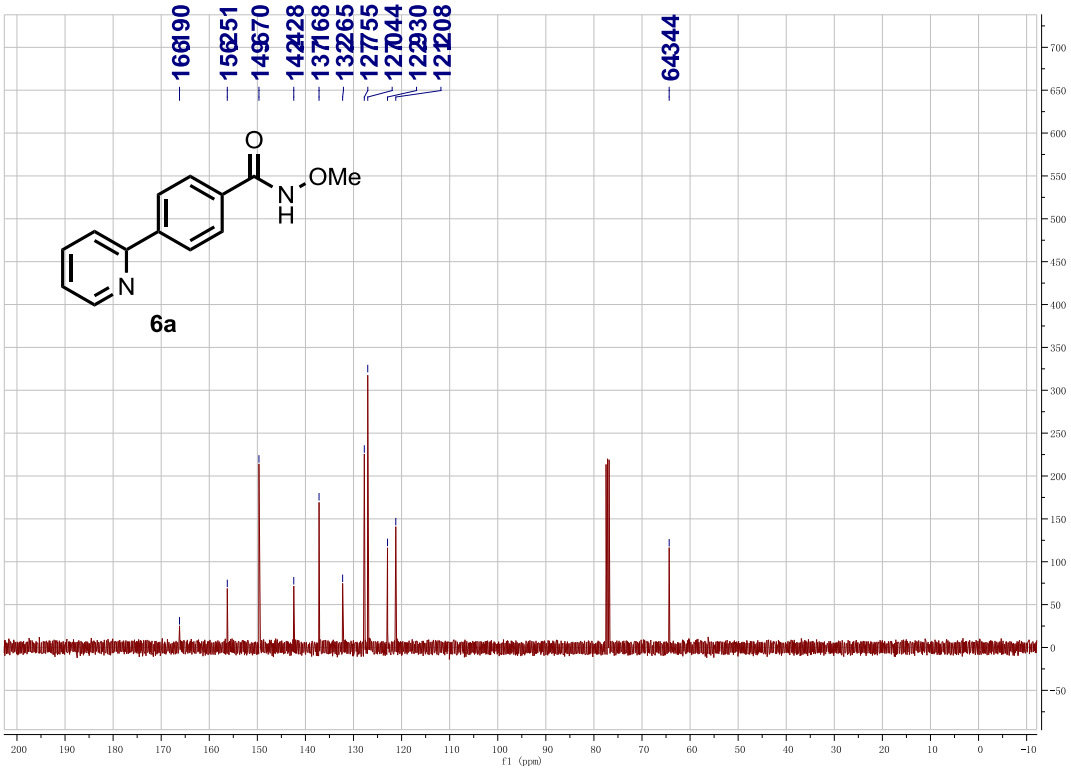
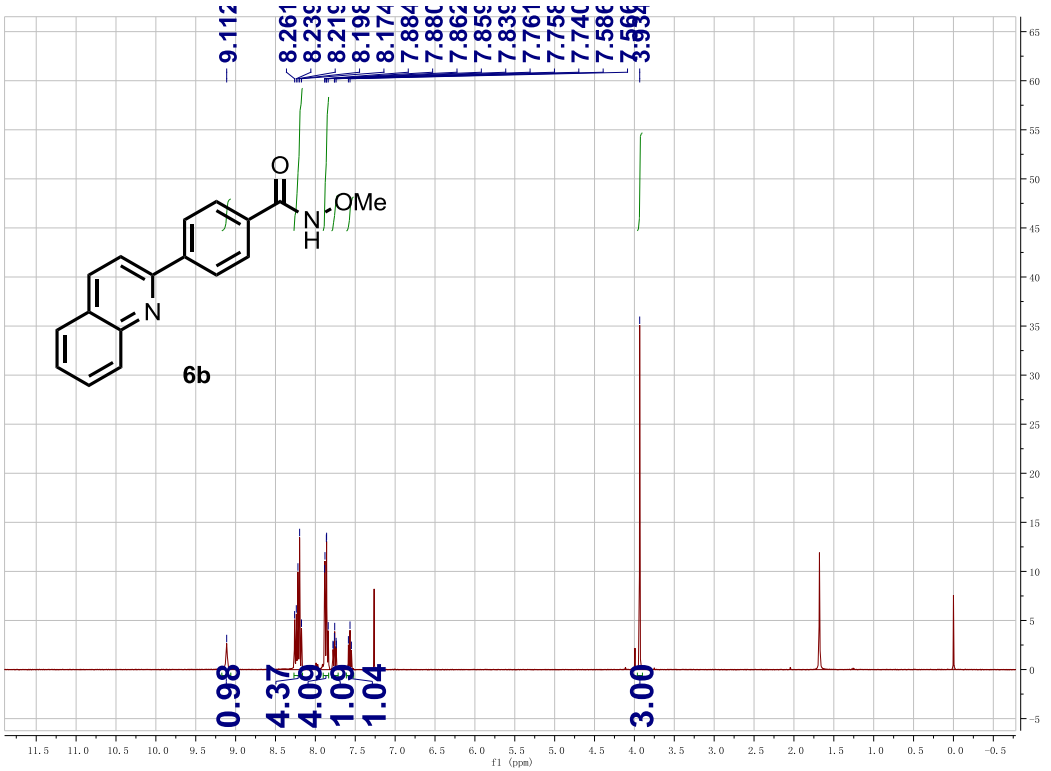
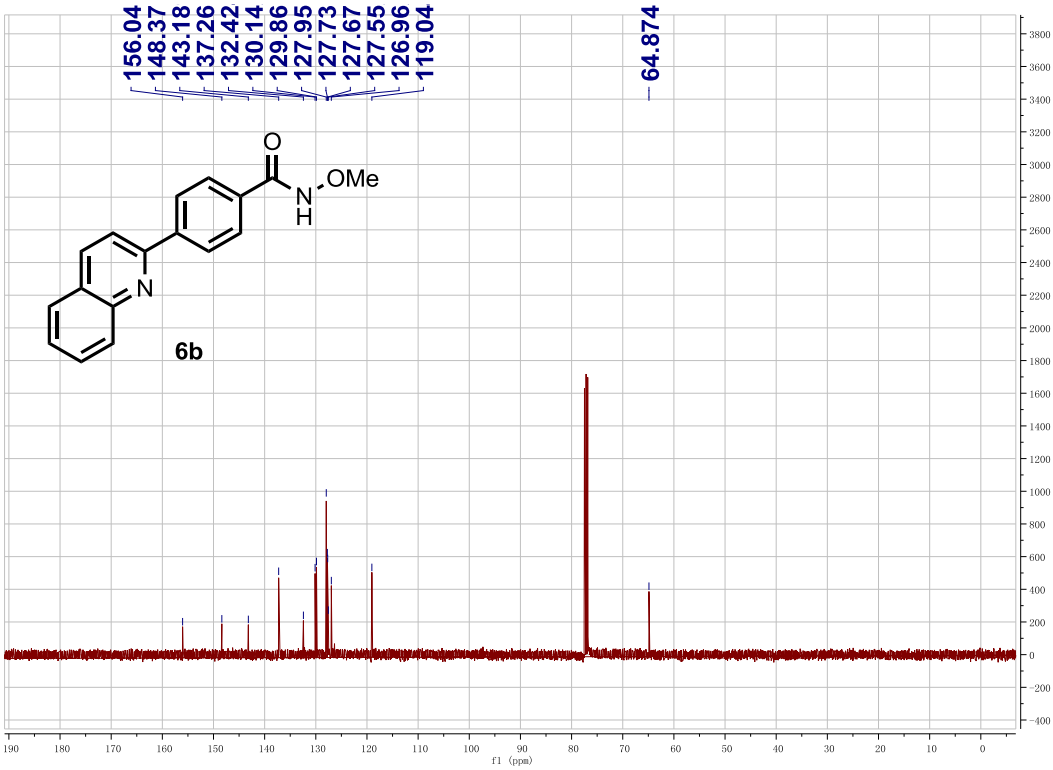
3.866
O
N
H
OMe
P
O
4t
0.90
2.07
12.32
3.00
f1 (ppm)

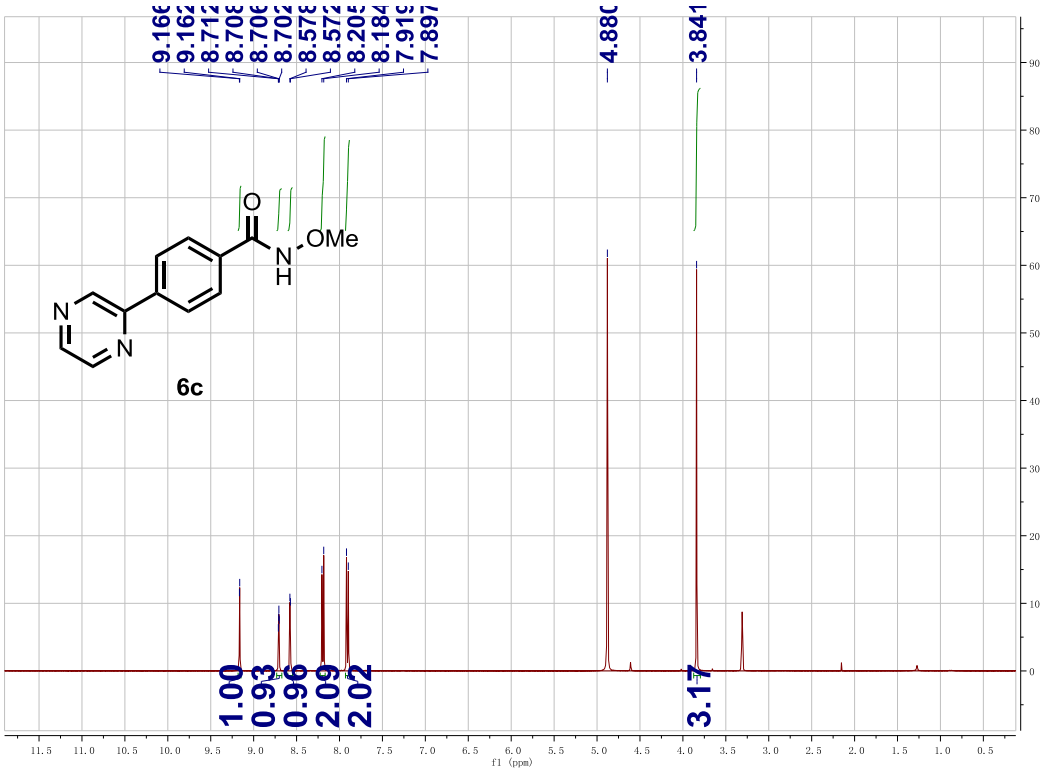

164.62
136.12
136.10
135.36
134.34
132.46
132.43
131.99
131.88
131.78
131.61
130.56
128.78
128.66
127.73
127.60
63.850
O
N
H
OMe
P
O
4t
f1 (ppm)

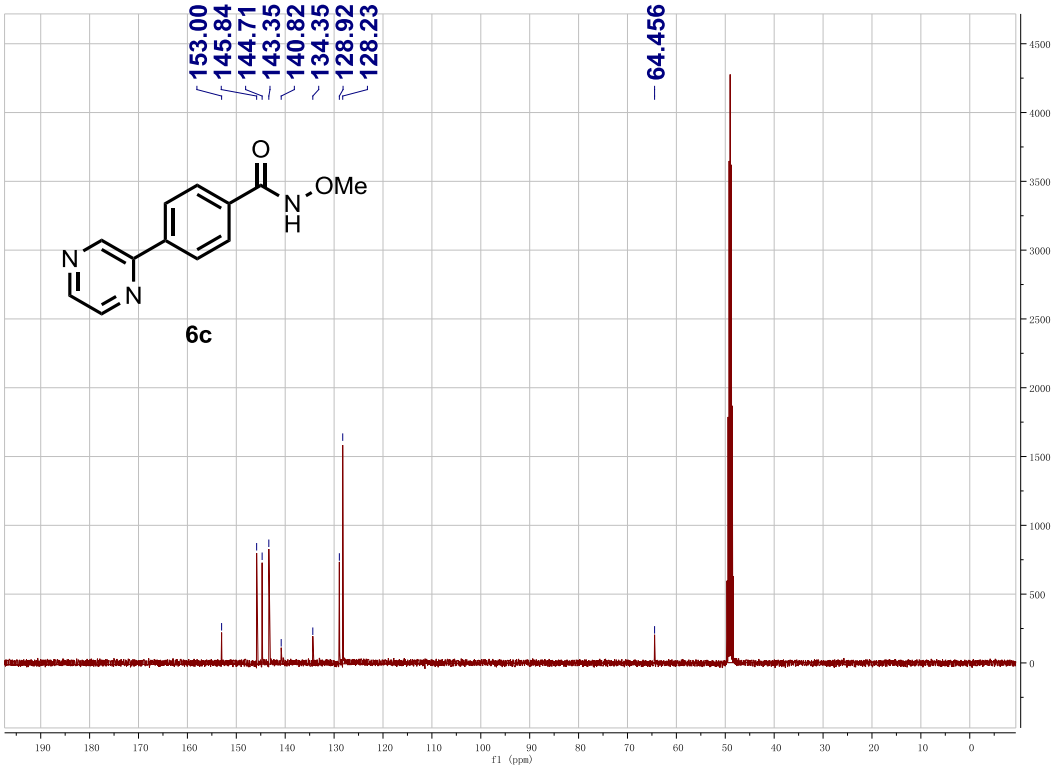

10.621
8.669
8.658
7.908
7.888
7.824
7.803
7.750
7.746
7.731
7.727
7.682
7.662
7.274
7.261
3.840
O
N
OMe
H
N
6a
0.95
1.00
2.08
2.03
1.08
1.04
1.06
3.15
f1 (ppm)

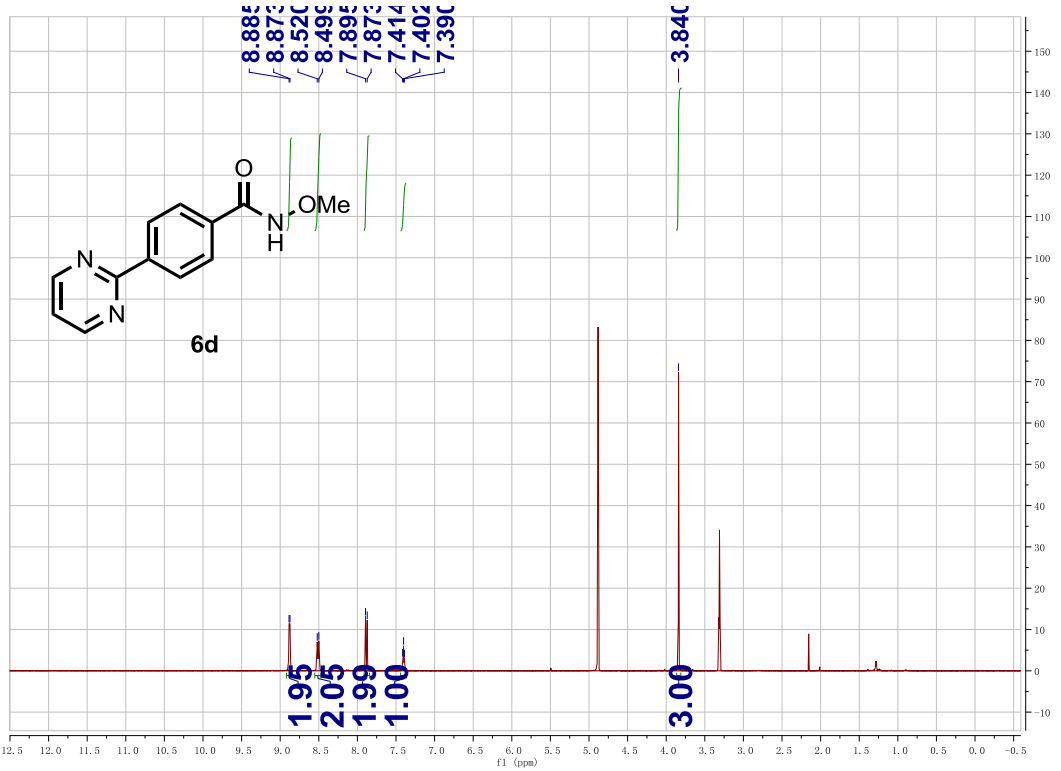

166.190
152.251
149.670
142.428
137.168
132.265
127.755
127.044
122.930
122.208
64.344
O
N
OMe
H
N
6a
f1 (ppm)


9.112
8.261
8.235
8.219
8.198
8.174
7.884
7.880
7.862
7.859
7.839
7.761
7.758
7.740
7.586
7.566
3.932
O
N
H
OMe
N
6b
0.98
4.37
4.09
1.09
1.04
3.00
f1 (ppm)


156.04
148.37
143.18
137.26
132.42
130.14
129.86
127.95
127.73
127.67
127.55
126.96
119.04
64.874
O
N
H
OMe
N
6b
f1 (ppm)


9.166
9.162
8.712
8.708
8.706
8.702
8.578
8.572
8.205
8.184
7.919
7.897
4.880
3.841
O
N
OMe
H
N
N
6c
1.00
0.93
0.96
2.09
2.02
3.17
f1 (ppm)


153.00
145.84
144.71
143.35
140.82
134.35
128.92
128.23
64.456
O
N
OMe
H
N
N
6c
f1 (ppm)


8.885
8.873
8.520
8.495
7.895
7.873
7.414
7.402
7.390
3.840
O
N
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OMe
N
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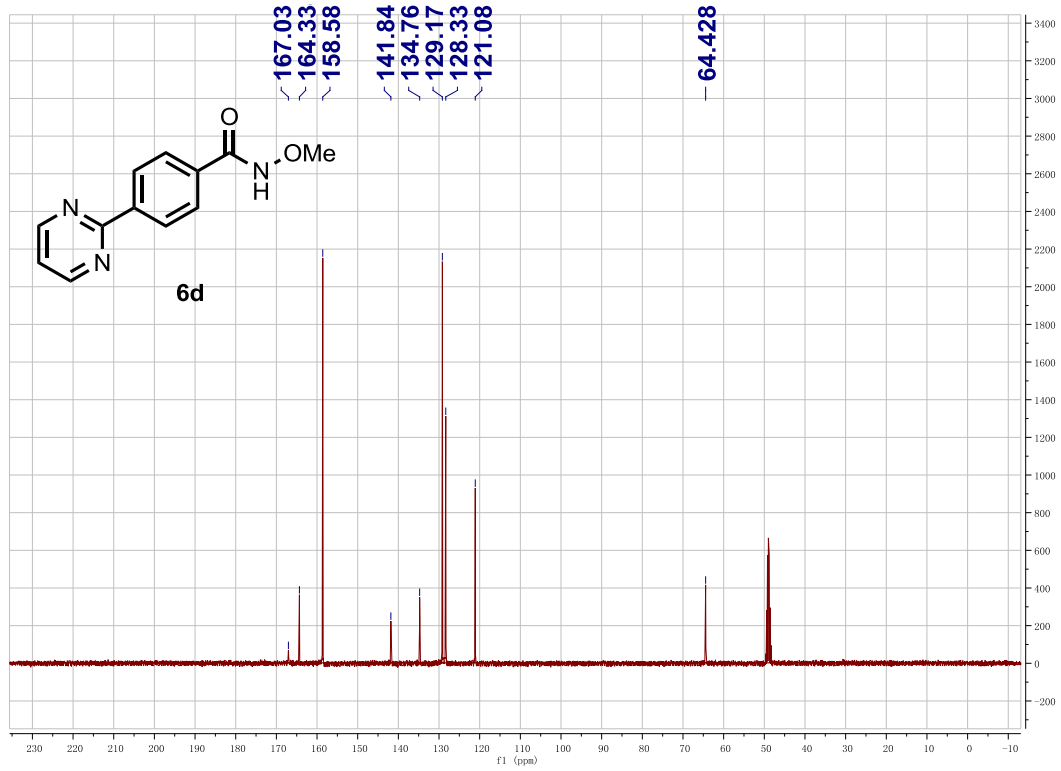
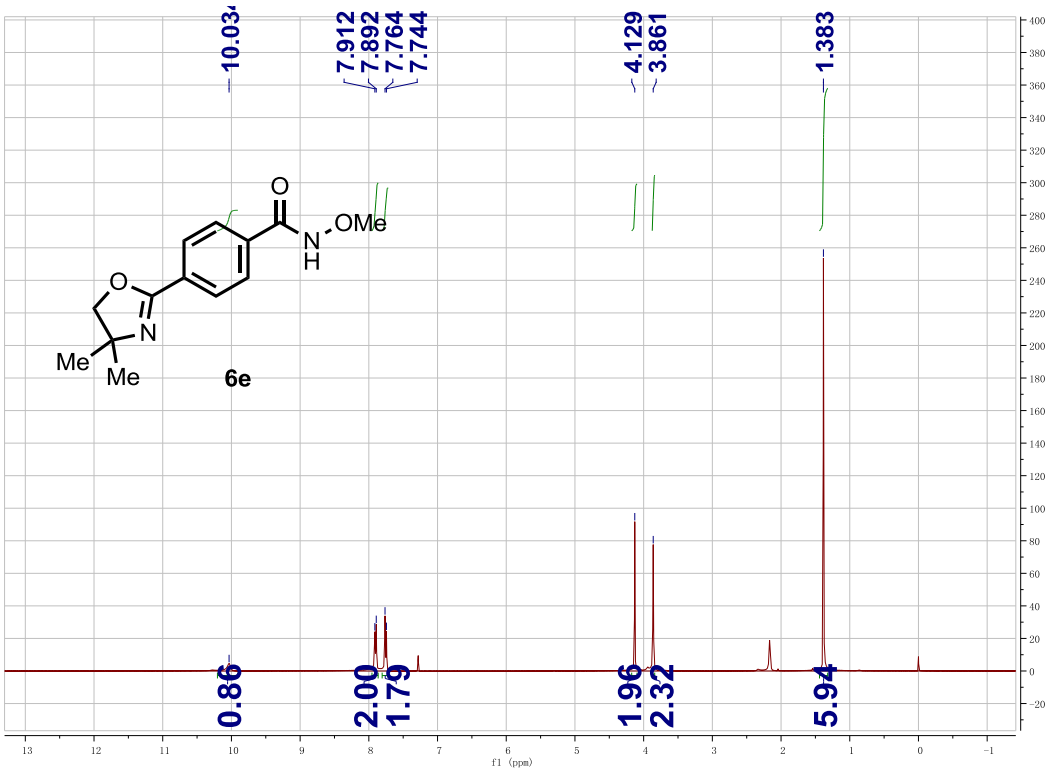
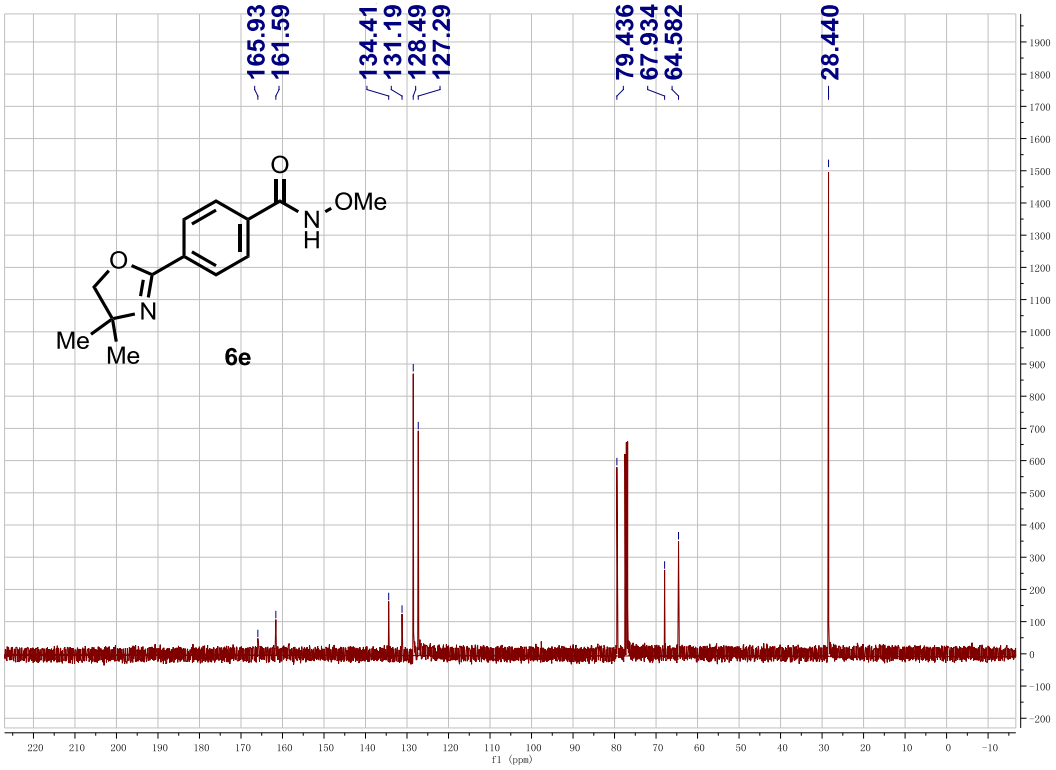
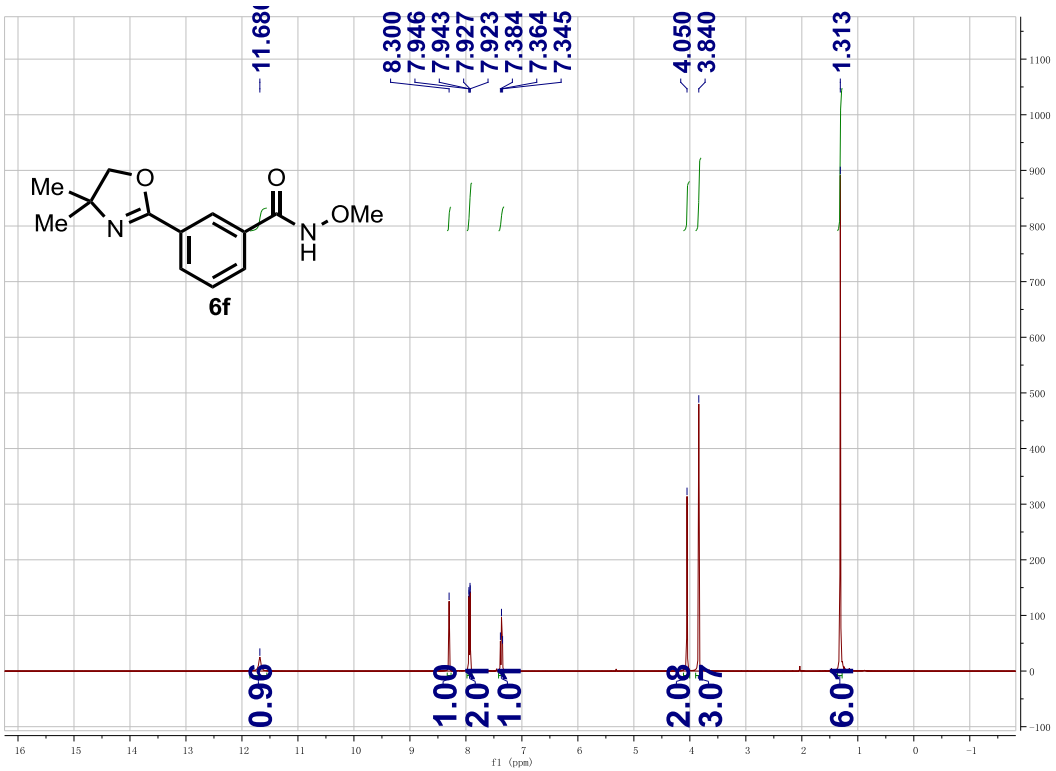
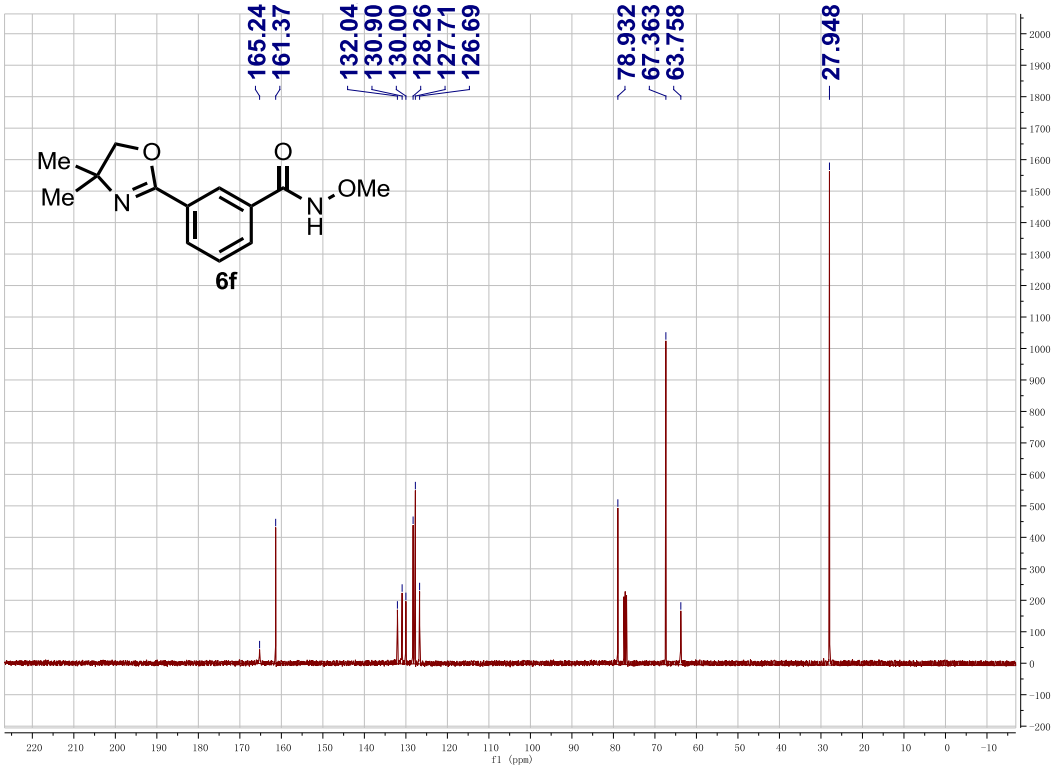
2.05
1.99
1.00
3.00
f1 (ppm)

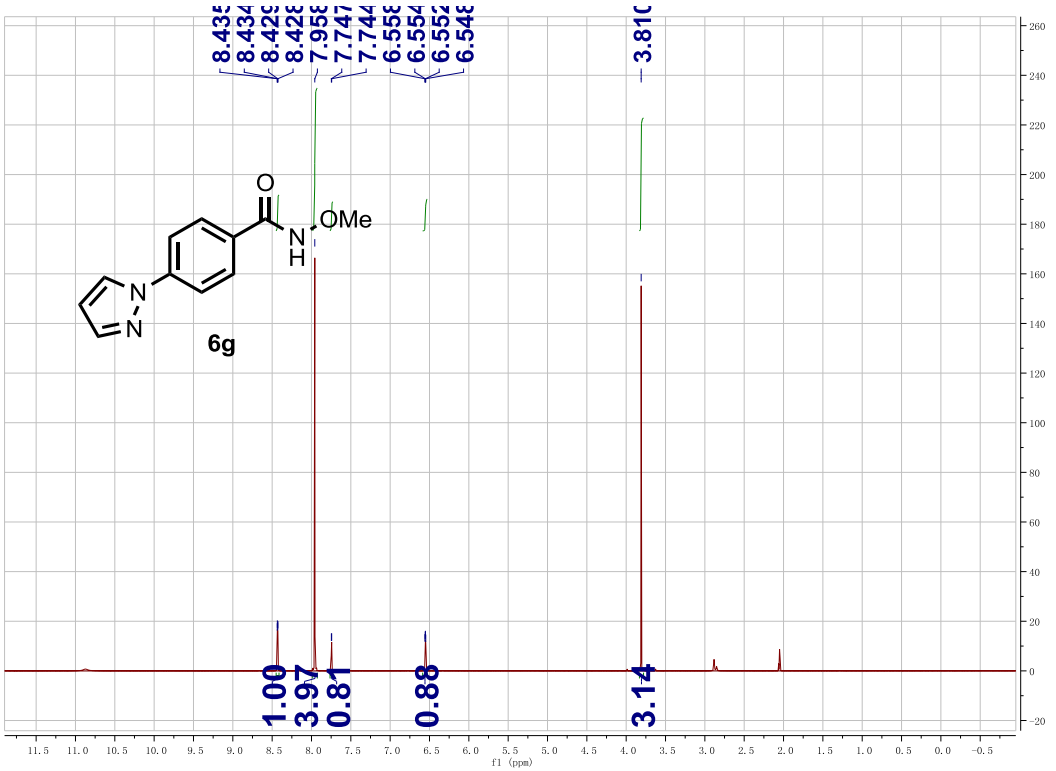

167.03
164.33
158.58
141.84
134.76
129.17
128.33
121.08
64.428
O
N
H
OMe
N
N
6d
f1 (ppm)

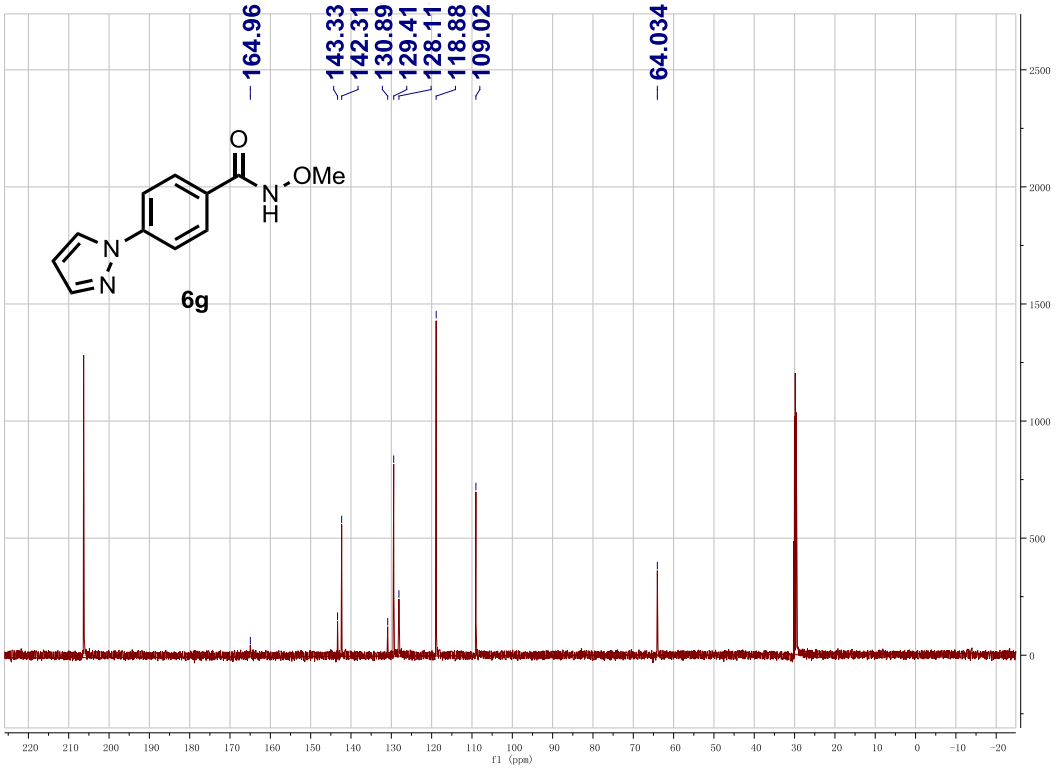

10.03
7.912
7.892
7.764
7.744
4.129
3.861
1.383
0.86
2.00
1.79
1.96
2.32
5.94
O
N
OMe
H
O
N
Me
Me
6e
f1 (ppm)

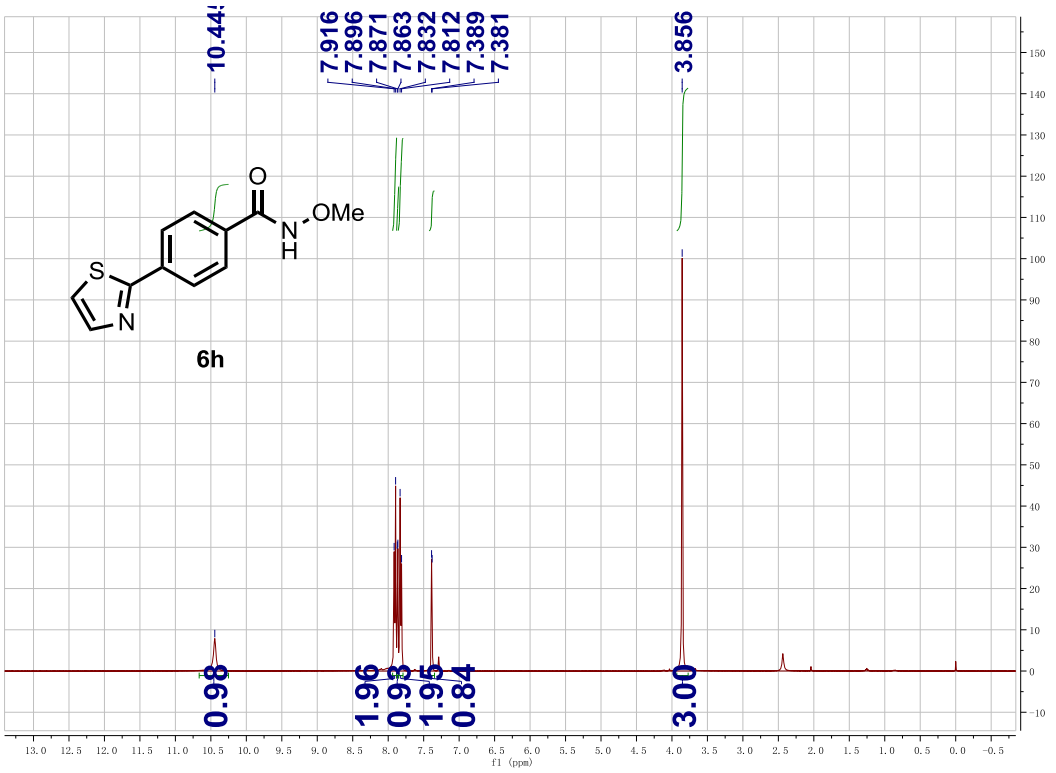

165.93
161.59
134.41
131.19
128.49
127.29
79.436
67.934
64.582
28.440
O
N
OMe
H
O
N
Me
Me
6e
f1 (ppm)

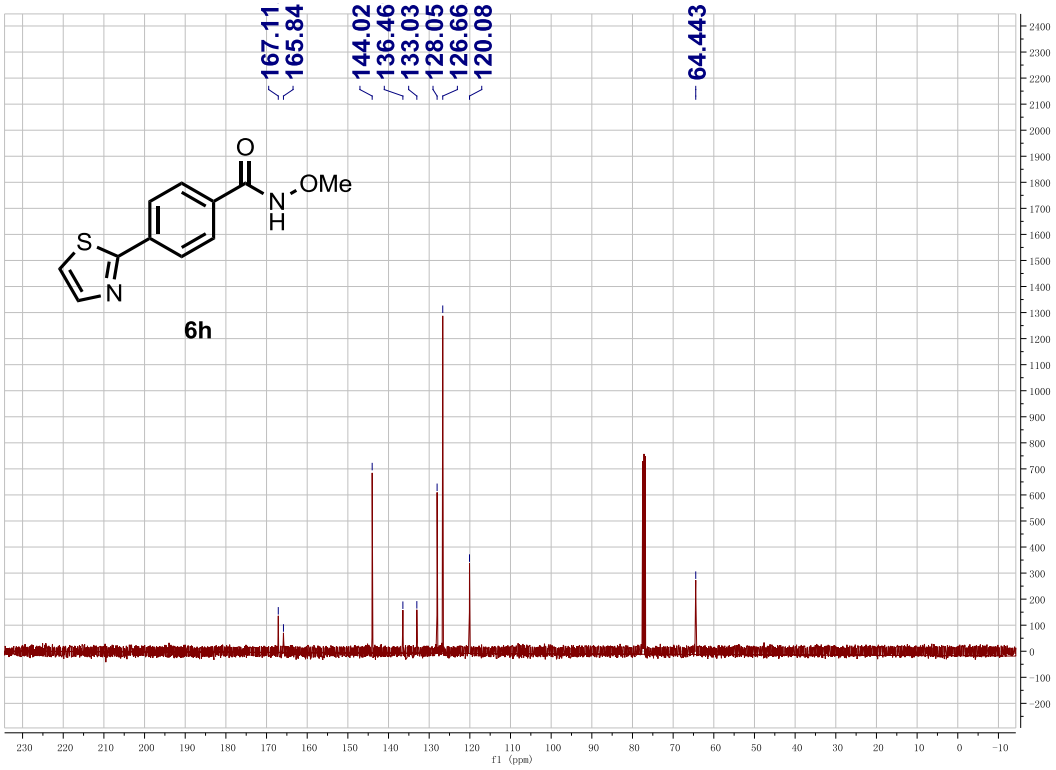

11.681
8.300
7.946
7.943
7.927
7.923
7.384
7.364
7.345
4.050
3.840
1.313
Me
Me
O
N
O
N
H
OMe
6f
0.96
1.00
2.01
1.01
2.08
3.07
6.01
f1 (ppm)

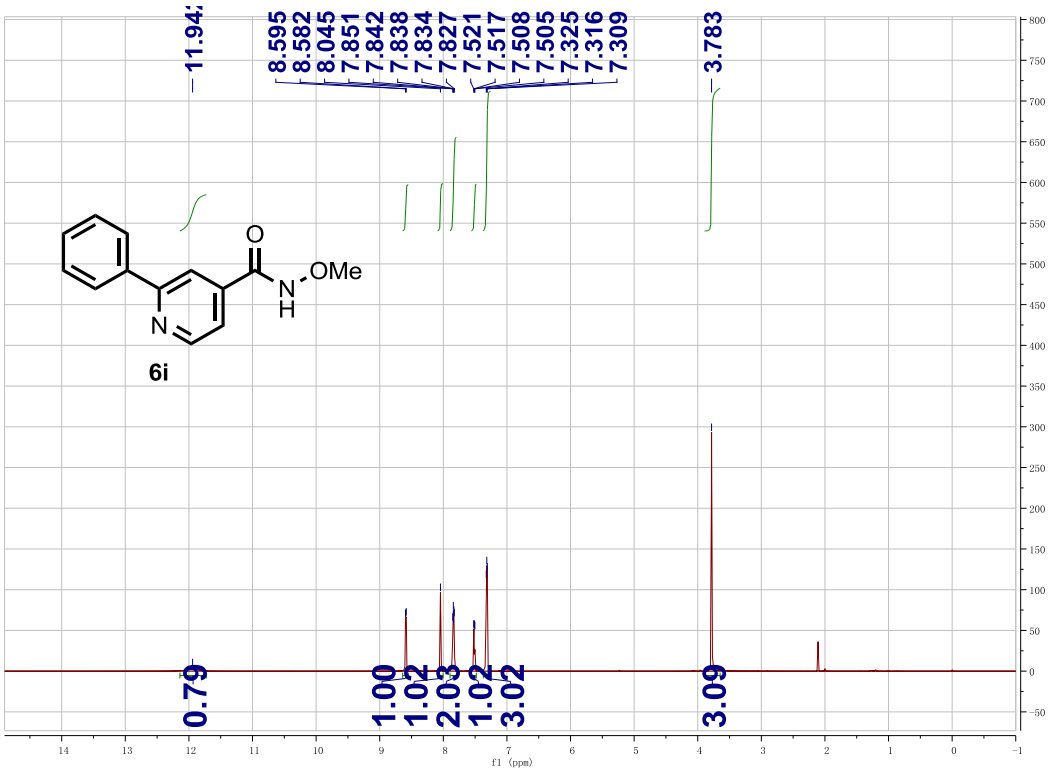

165.24
161.37
132.04
130.90
130.00
128.26
127.71
126.69
78.932
67.363
63.758
27.948
Me
Me
O
N
O
N
H
OMe
6f
f1 (ppm)

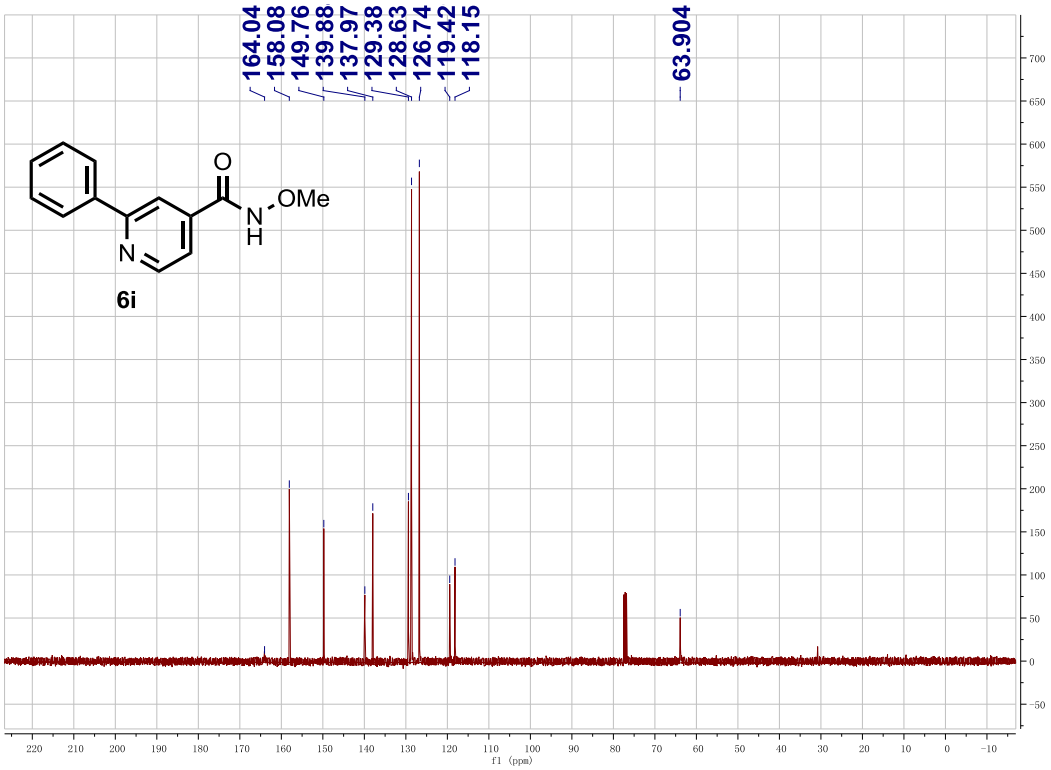

8.435
8.434
8.429
8.428
7.958
7.747
7.744
6.558
6.554
6.552
6.548
3.810
O
N
H
OMe
N
N
6g
1.00
3.97
0.81
0.88
3.14
f1 (ppm)

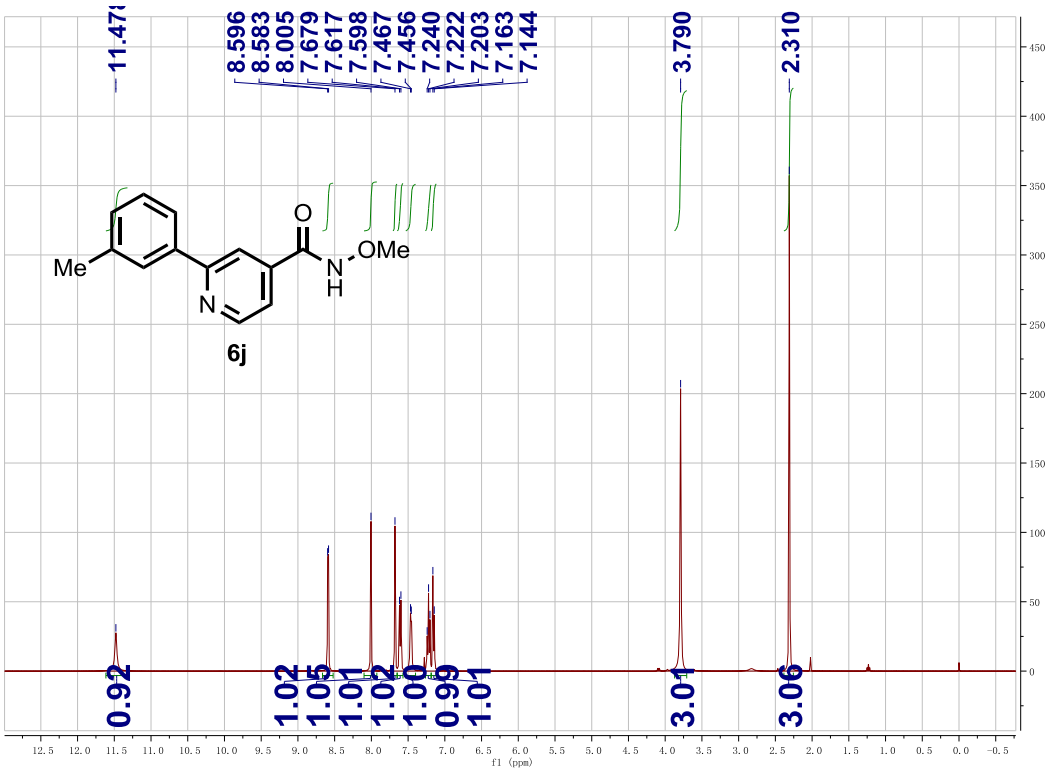

164.96
143.33
142.31
130.89
129.41
128.11
118.88
109.02
64.034
O
N
H
OMe
N
N
6g
f1 (ppm)


10.44
7.916
7.896
7.871
7.863
7.832
7.812
7.389
7.381
3.856
O
N
H
OMe
S
N
6h
0.98
1.96
0.93
1.95
0.84
3.00
f1 (ppm)


167.11
165.84
144.02
136.46
133.03
128.05
126.66
120.08
64.443
O
N
H
OMe
S
N
6h
f1 (ppm)


11.94
8.595
8.582
8.045
7.851
7.842
7.838
7.834
7.827
7.521
7.517
7.508
7.505
7.325
7.316
7.309
3.783
O
N
OMe
H
N
6i
0.79
1.00
1.02
2.03
1.02
3.02
3.09
f1 (ppm)


164.04
158.08
149.76
139.88
137.97
129.38
128.63
126.74
119.42
118.15
63.904
O
N
OMe
H
N
6i
f1 (ppm)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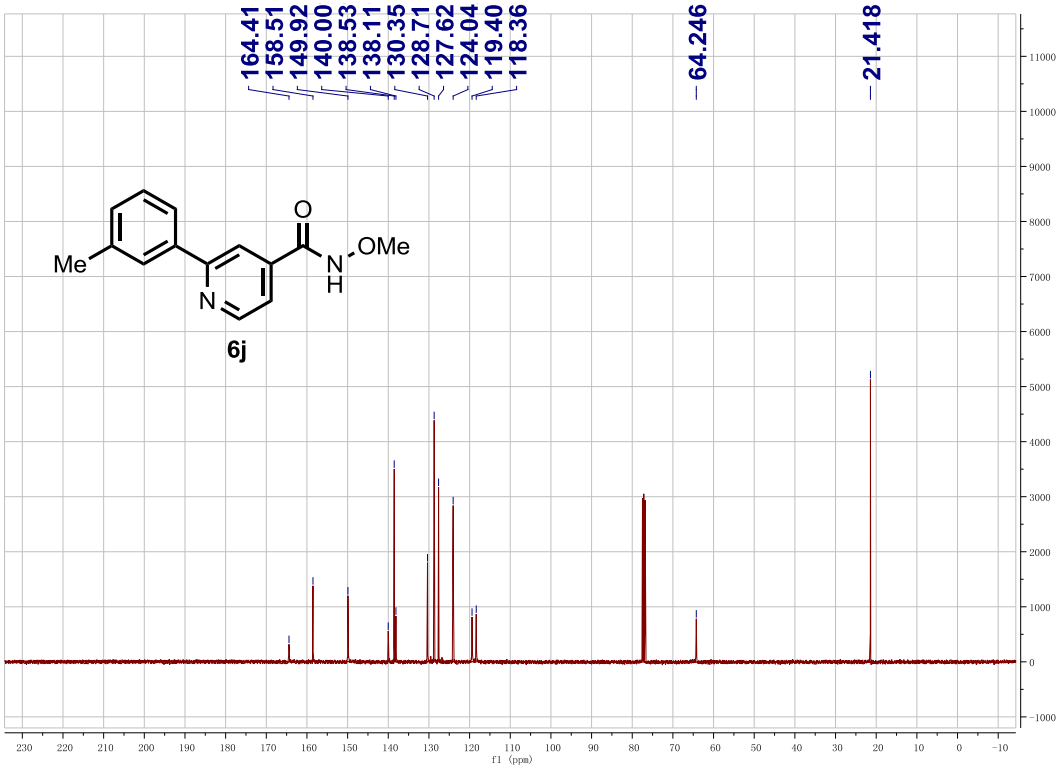
8.583
8.005
7.679
7.617
7.598
7.467
7.456
7.240
7.222
7.203
7.163
7.144
3.790
2.310
Me
N
O
N
H
OMe
6j
0.92
1.02
1.05
1.01
1.02
1.00
0.99
1.01
3.01
3.06
f1 (ppm)


164.41
158.51
149.92
140.00
138.53
138.11
130.35
128.71
127.62
124.04
119.40
118.36
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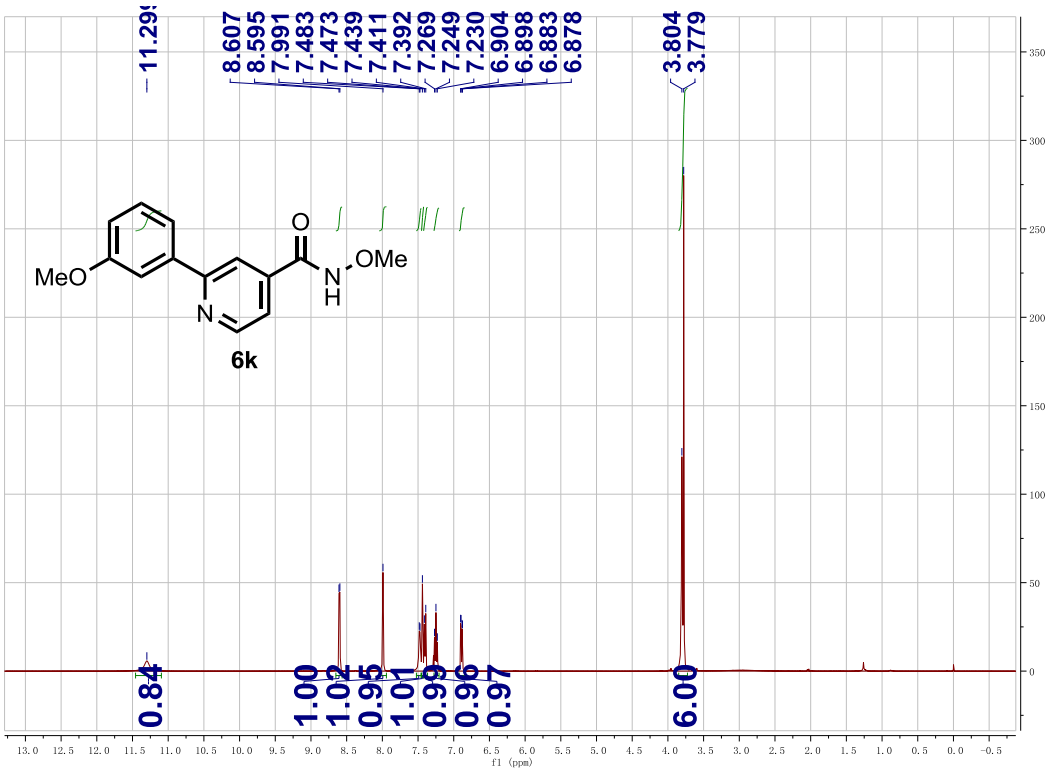
21.418
Me
N
O
N
H
OMe
6j
f1 (ppm)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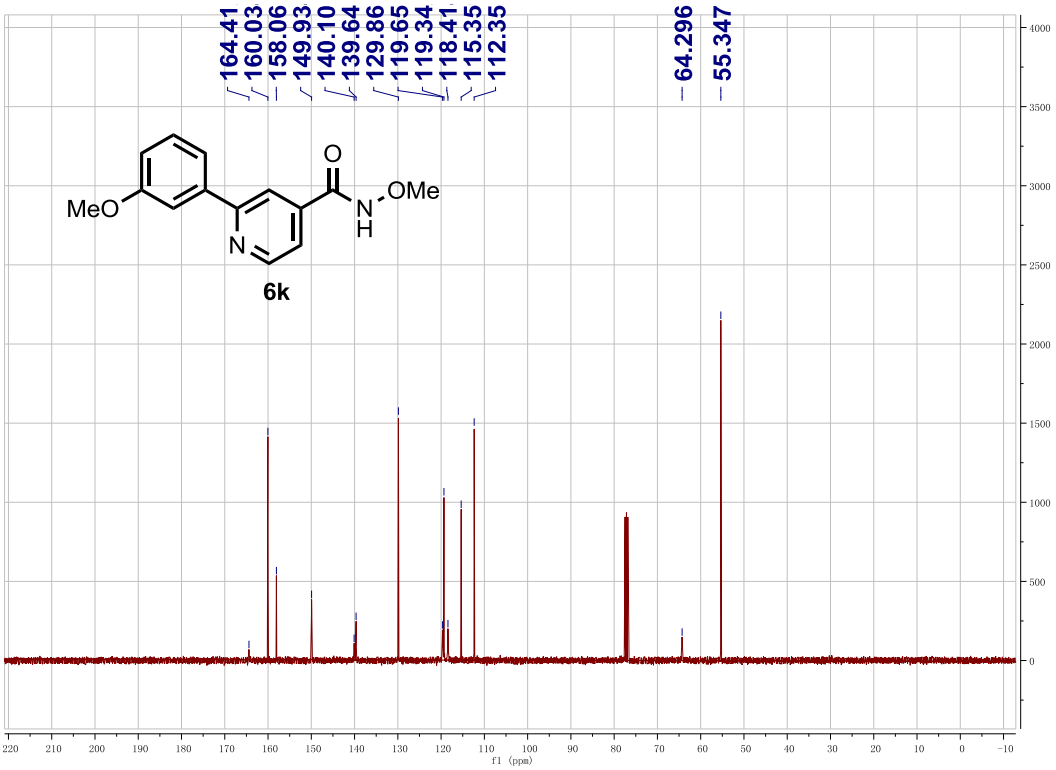
8.595
7.991
7.483
7.473
7.439
7.411
7.392
7.269
7.249
7.230
6.904
6.898
6.883
6.878
3.804
3.779
MeO
N
O
N
H
OMe
6k
0.84
1.00
1.02
0.95
1.01
0.99
0.96
0.97
6.00
f1 (ppm)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160.03
158.06
149.93
140.10
139.64
129.86
119.65
119.34
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112.35
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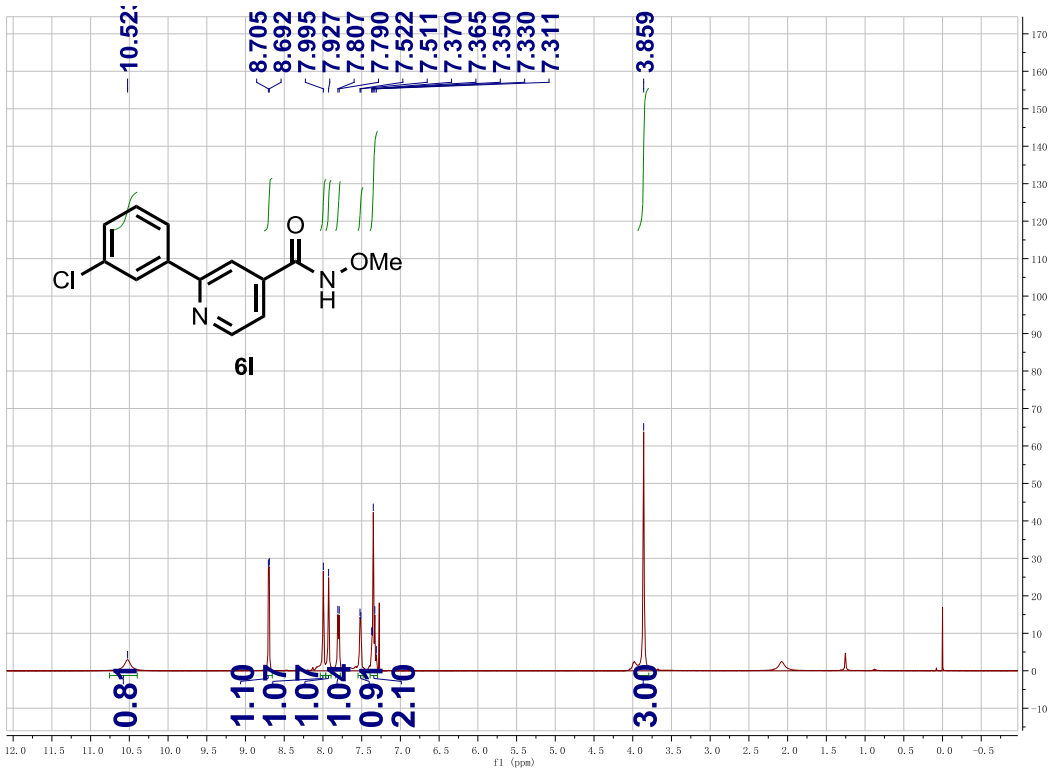
55.347
MeO
N
O
N
H
OMe
6k
f1 (ppm)


10.52
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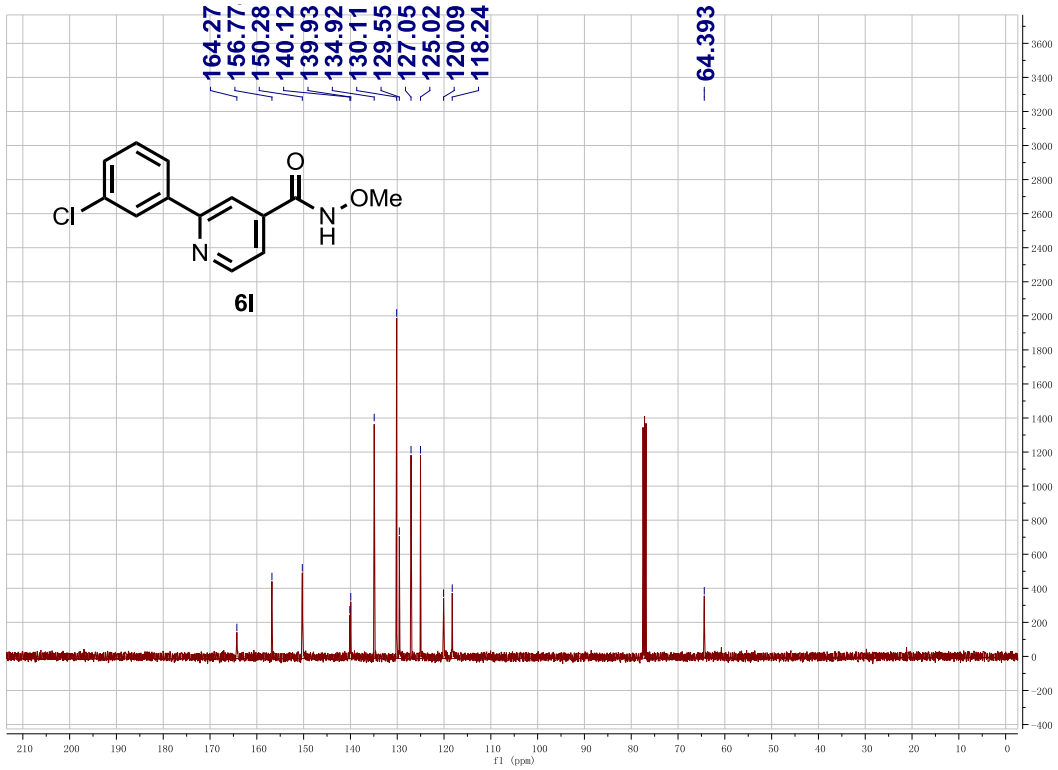
8.692
7.995
7.927
7.807
7.790
7.522
7.511
7.370
7.365
7.350
7.330
7.311
3.859
Cl
N
O
N
H
OMe
6l
0.81
1.10
1.07
1.07
1.04
0.91
2.10
3.00
f1 (ppm)


164.27
156.77
150.28
140.12
139.93
134.92
130.11
129.55
127.05
125.02
120.09
118.24
64.393
Cl
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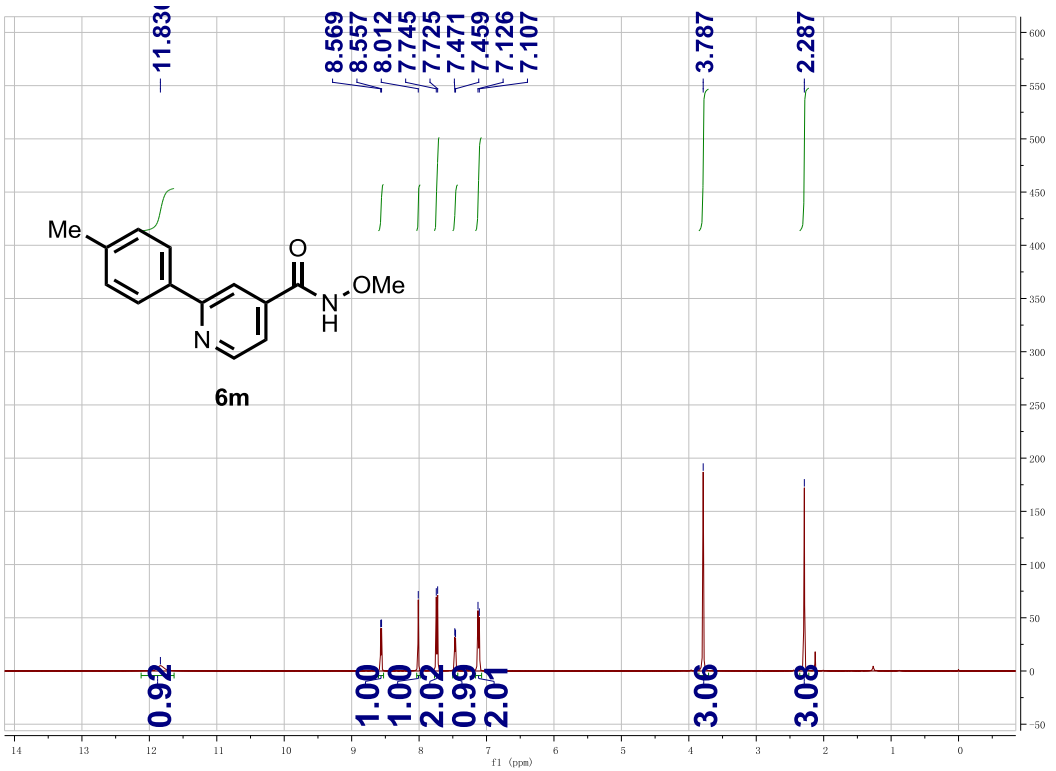
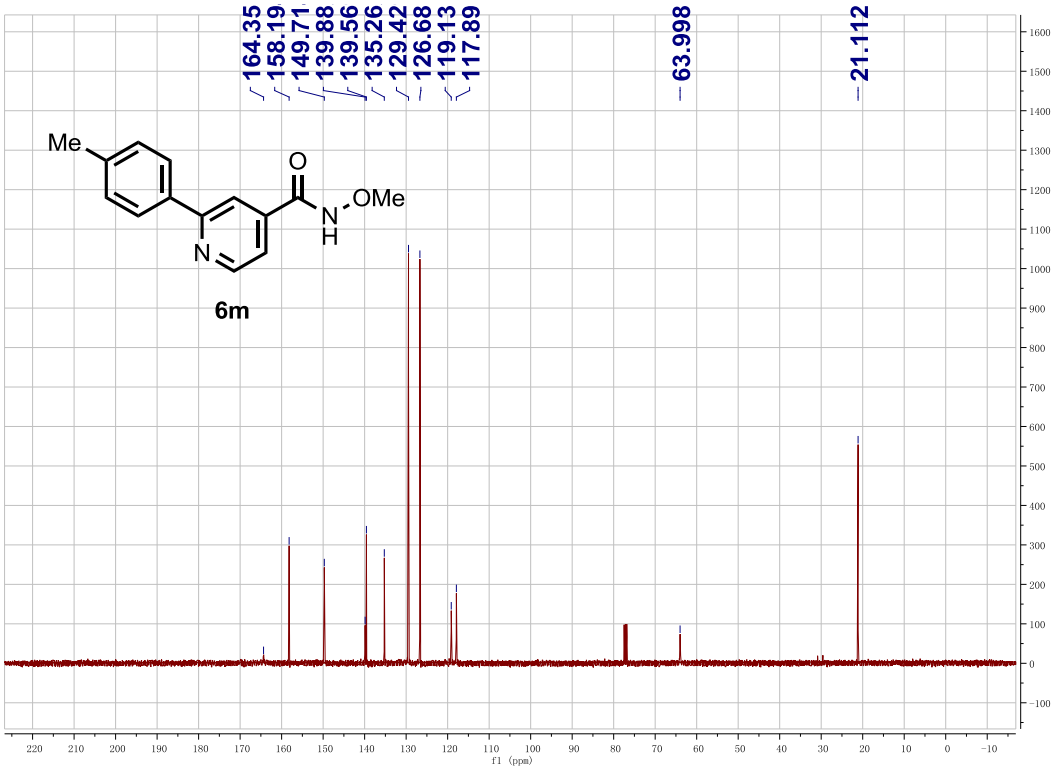
O
N
H
OMe
6l
f1 (ppm)


11.831
8.569
8.557
8.012
7.745
7.725
7.471
7.459
7.126
7.107
3.787
2.287
Me
O
N
H
OMe
N
6m
0.92
1.00
1.00
2.02
0.99
2.01
3.06
3.08
f1 (ppm)


164.35
158.19
149.71
139.88
139.56
135.26
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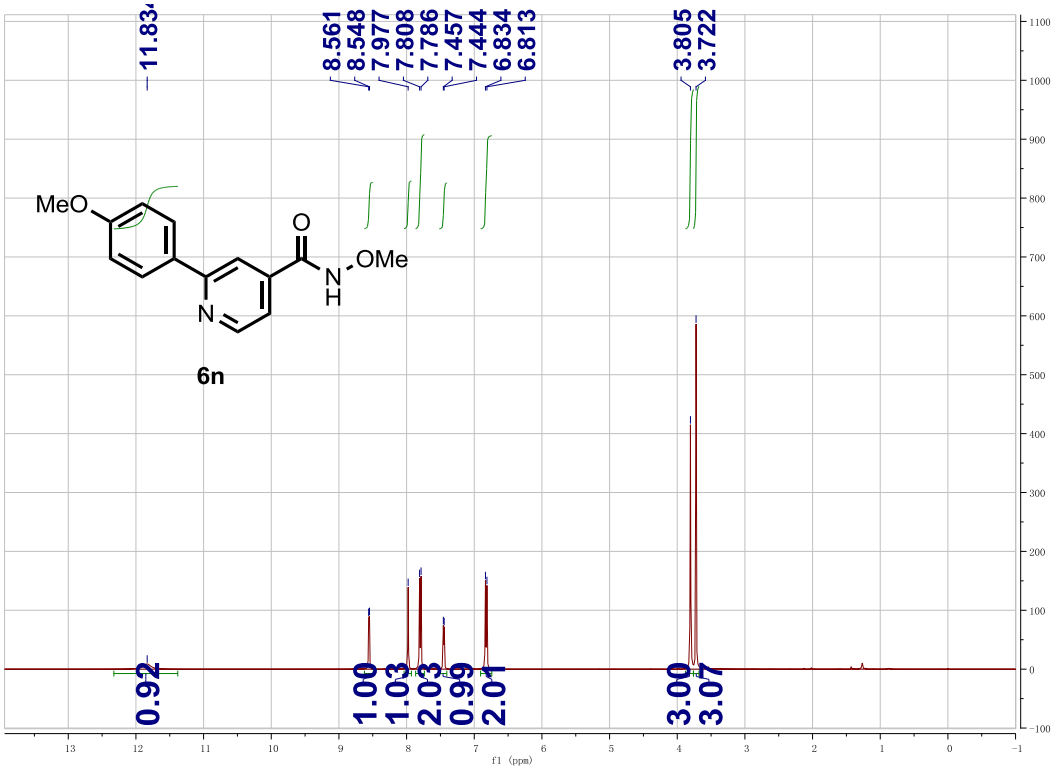
126.68
119.13
117.89
63.998
21.112
Me
O
N
H
OMe
N
6m
f1 (ppm)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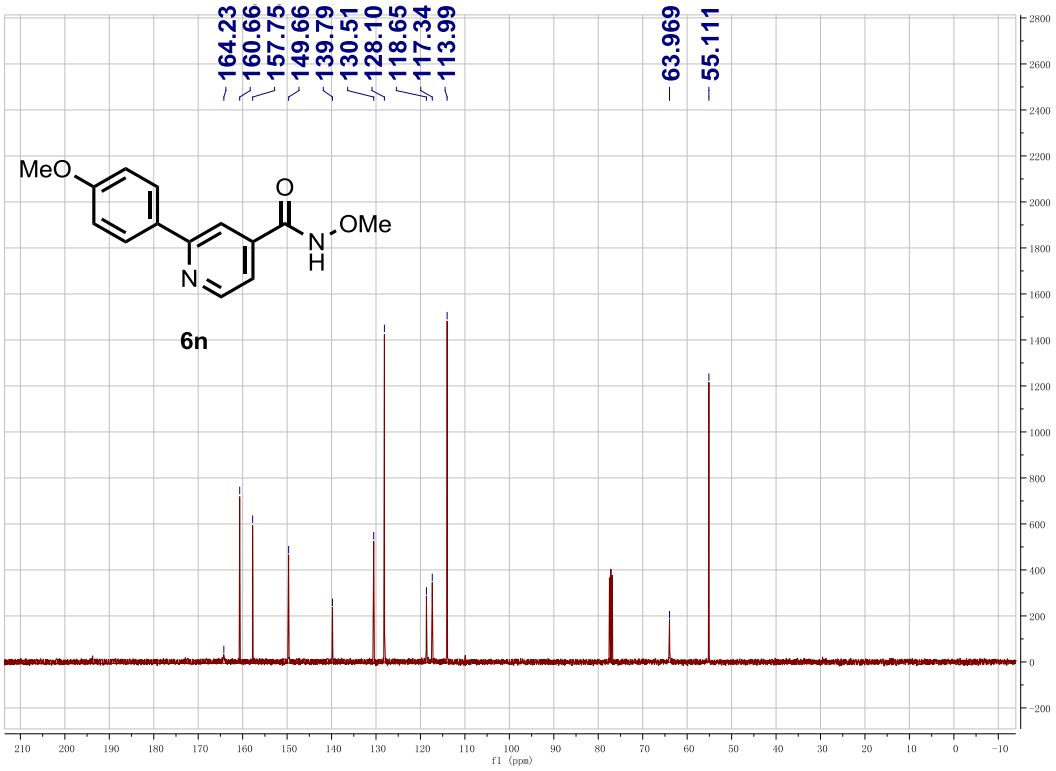
7.808
7.786
7.457
7.444
6.834
6.813
3.805
3.722
MeO
O
N
OMe
H
N
6n
0.92
1.00
1.03
2.03
0.99
2.01
3.00
3.07
f1 (ppm)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160.66
157.75
149.66
139.79
130.51
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118.65
117.34
113.99
63.969
55.111
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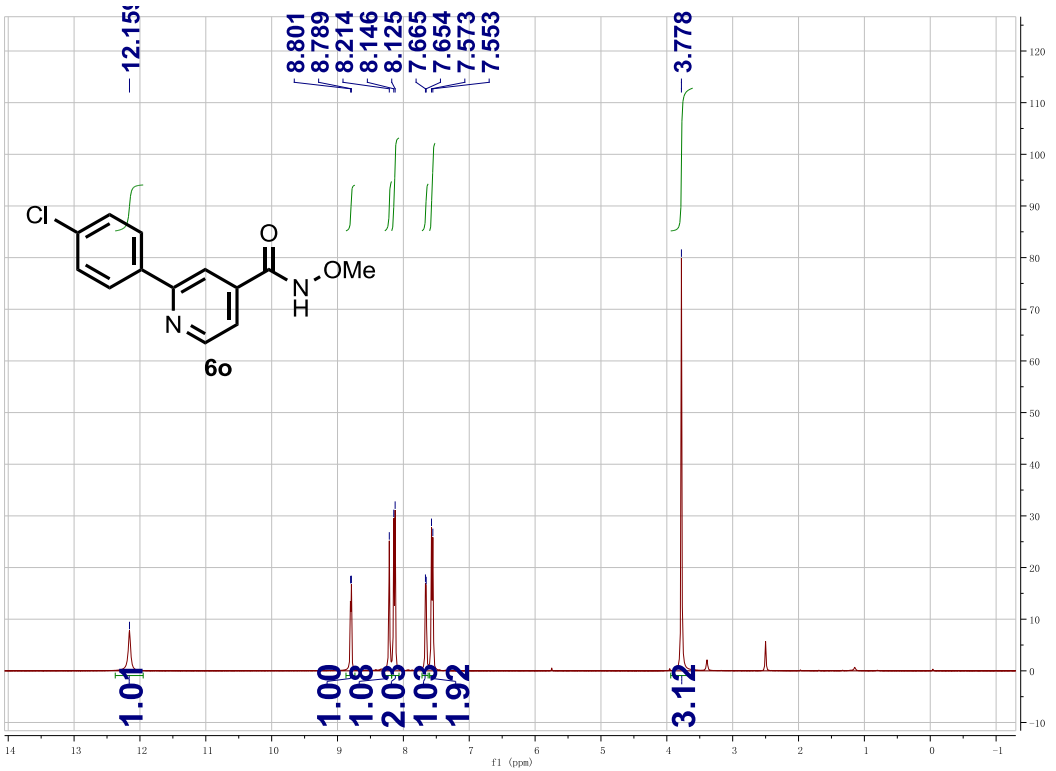
O
N
OMe
H
N
6n
f1 (ppm)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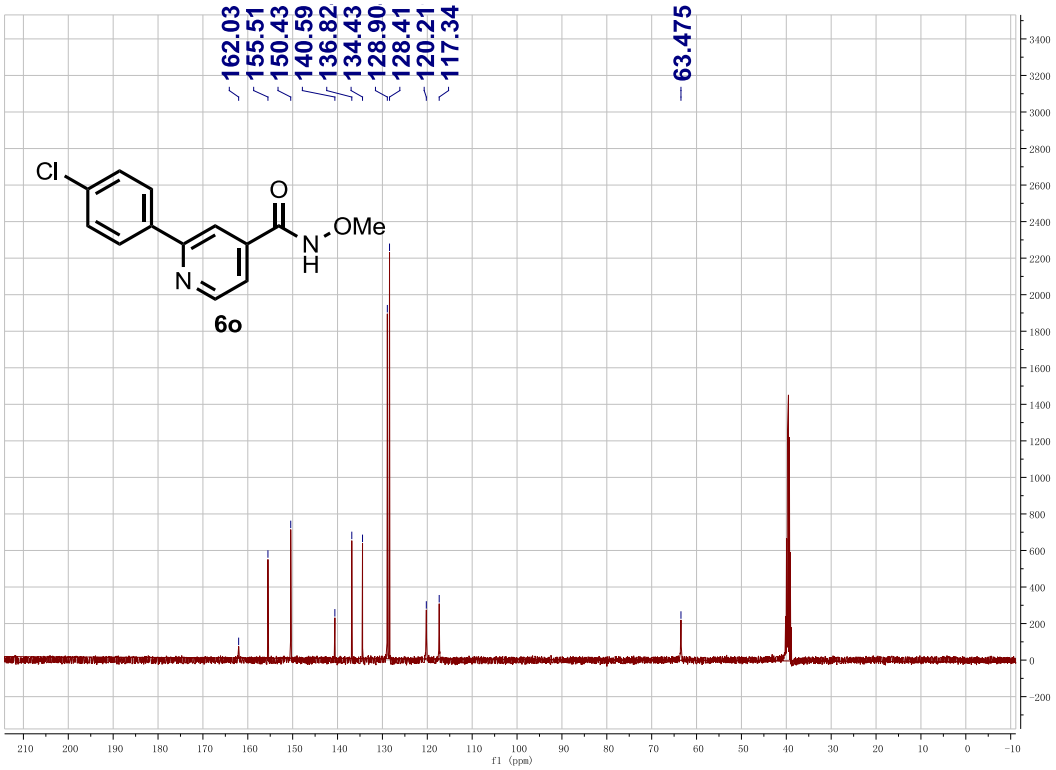
8.801
8.789
8.214
8.146
8.125
7.665
7.654
7.573
7.553
3.778
Cl
O
N
OMe
H
N
6o
1.01
1.00
1.08
2.03
1.03
1.92
3.12
f1 (ppm)


162.03
155.51
150.43
140.59
136.82
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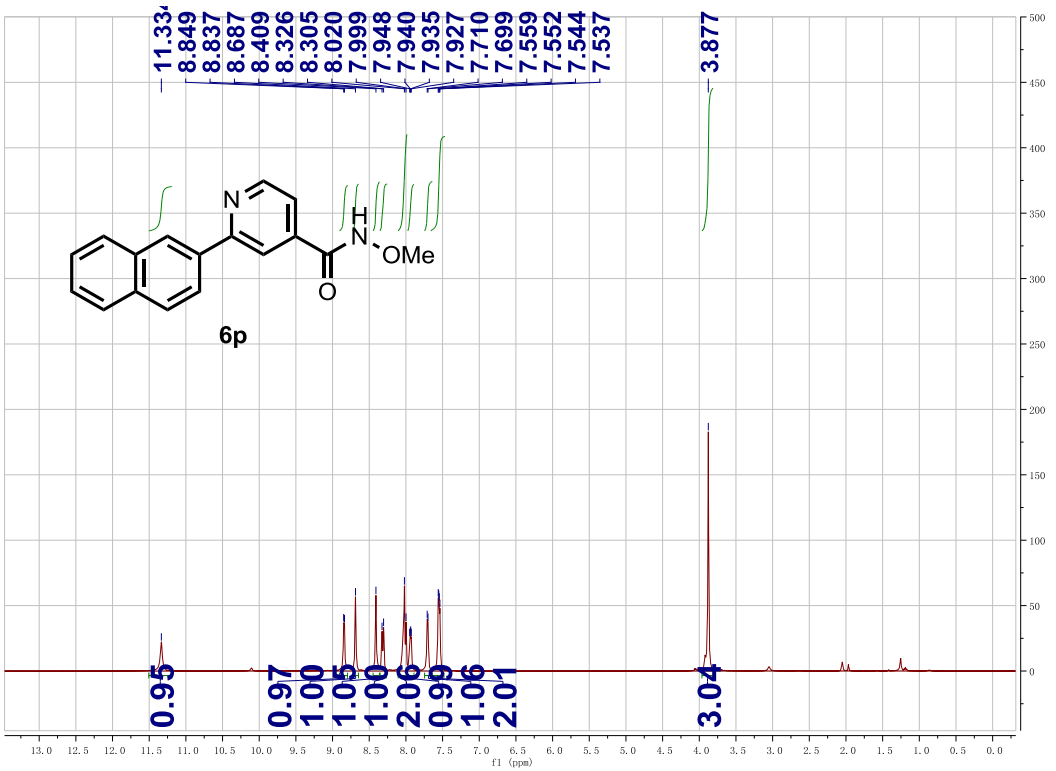
128.41
120.21
117.34
63.475
Cl
O
N
OMe
H
N
6o
f1 (ppm)


11.33
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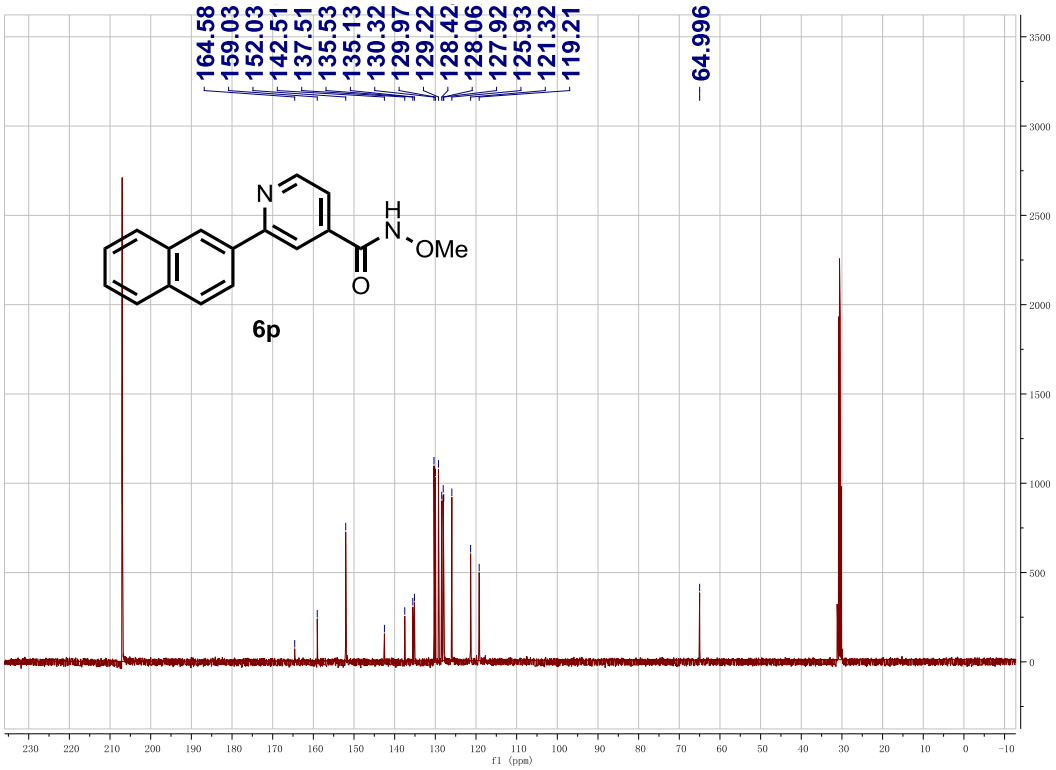
8.837
8.687
8.409
8.326
8.305
8.020
7.999
7.948
7.940
7.935
7.927
7.710
7.699
7.559
7.552
7.544
7.537
3.877
N
H
N
OMe
O
6p
0.95
0.97
1.00
1.02
1.00
2.06
0.99
1.06
2.01
3.04
f1 (ppm)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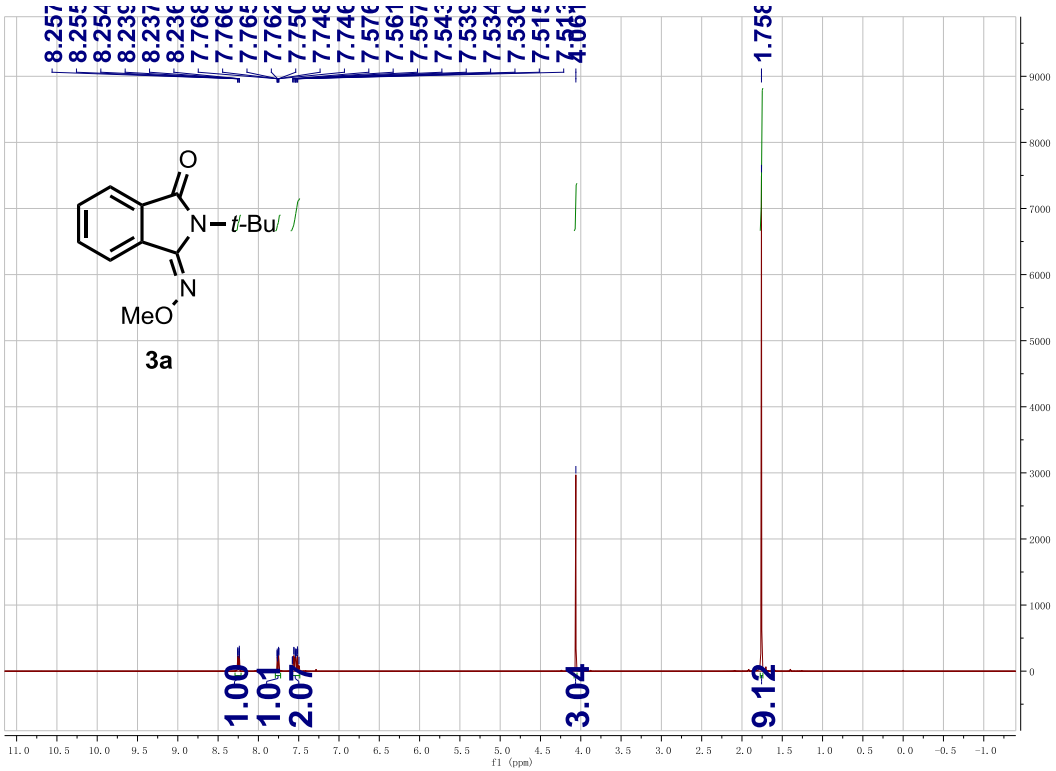
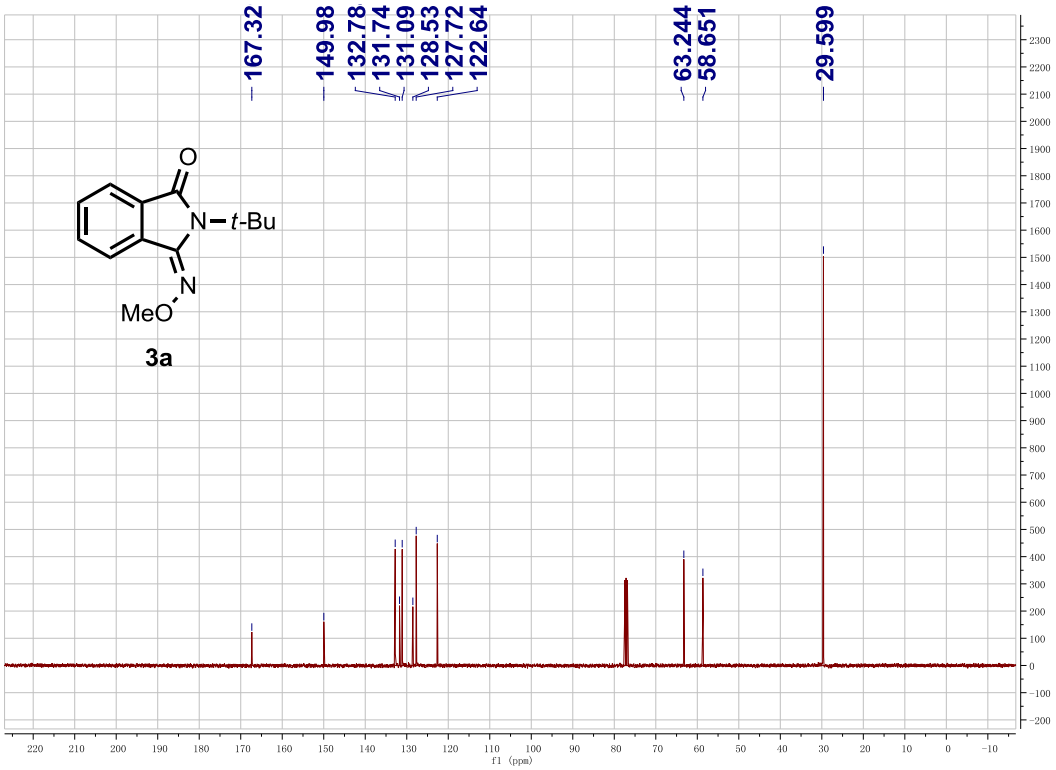
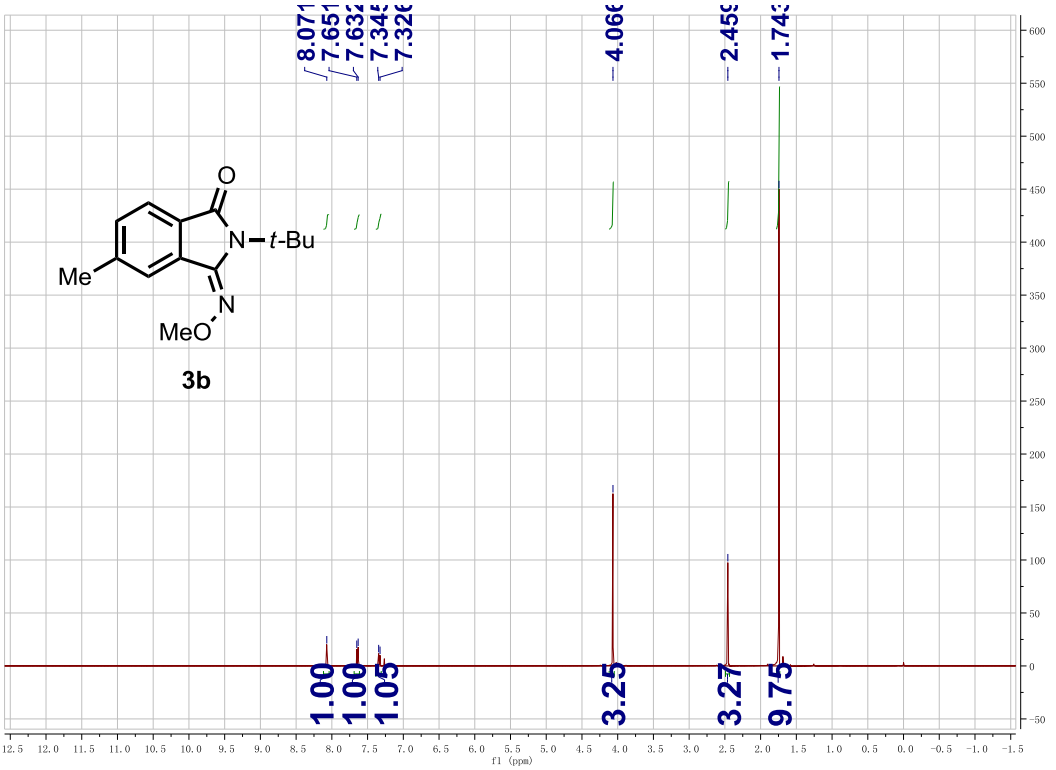
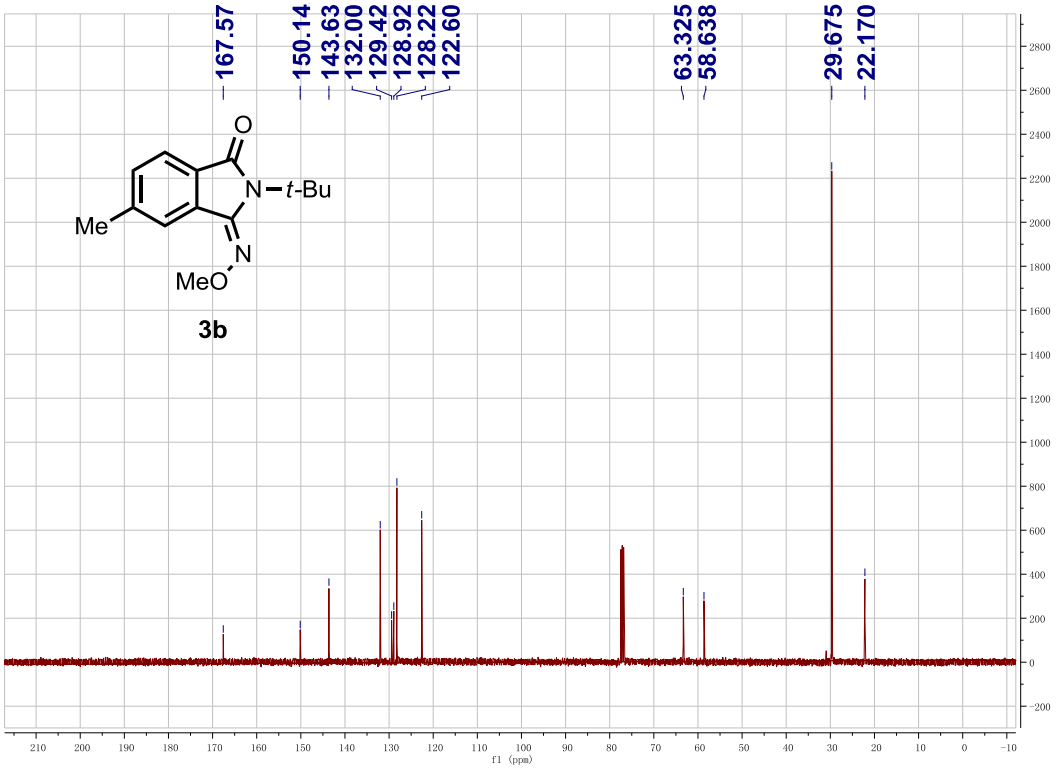
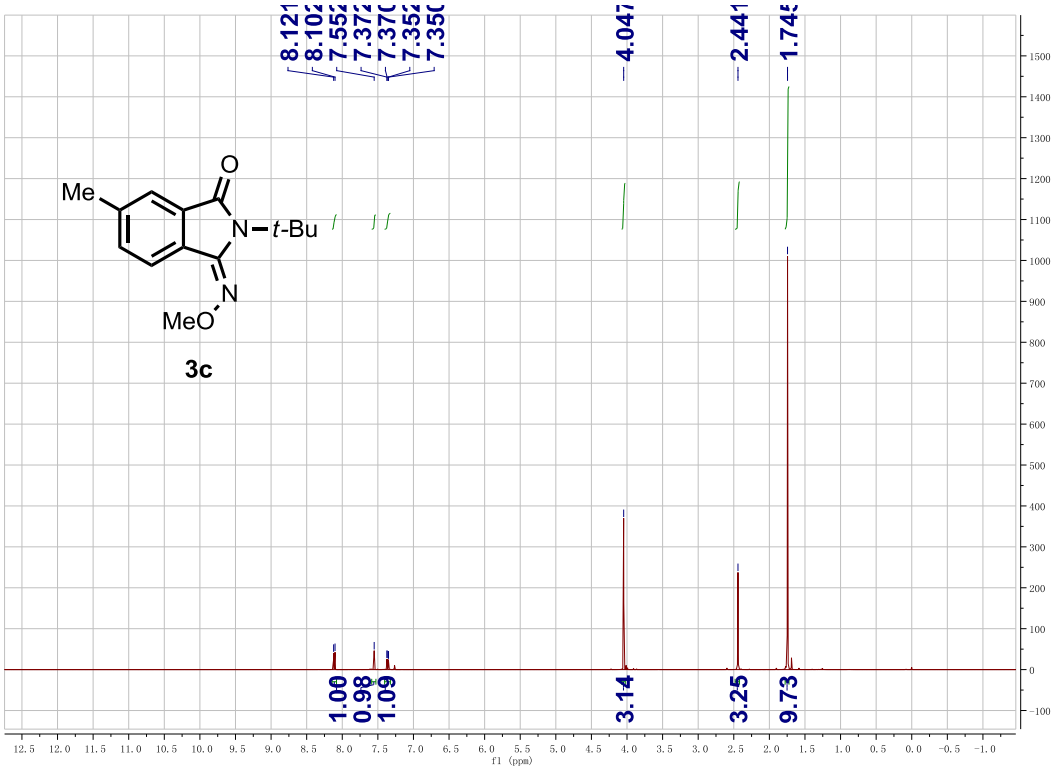
152.03
142.51
137.51
135.53
135.13
130.32
129.97
129.22
128.42
128.06
127.92
125.93
121.32
119.21
64.996
N
H
N
OMe
O
6p
f1 (ppm)

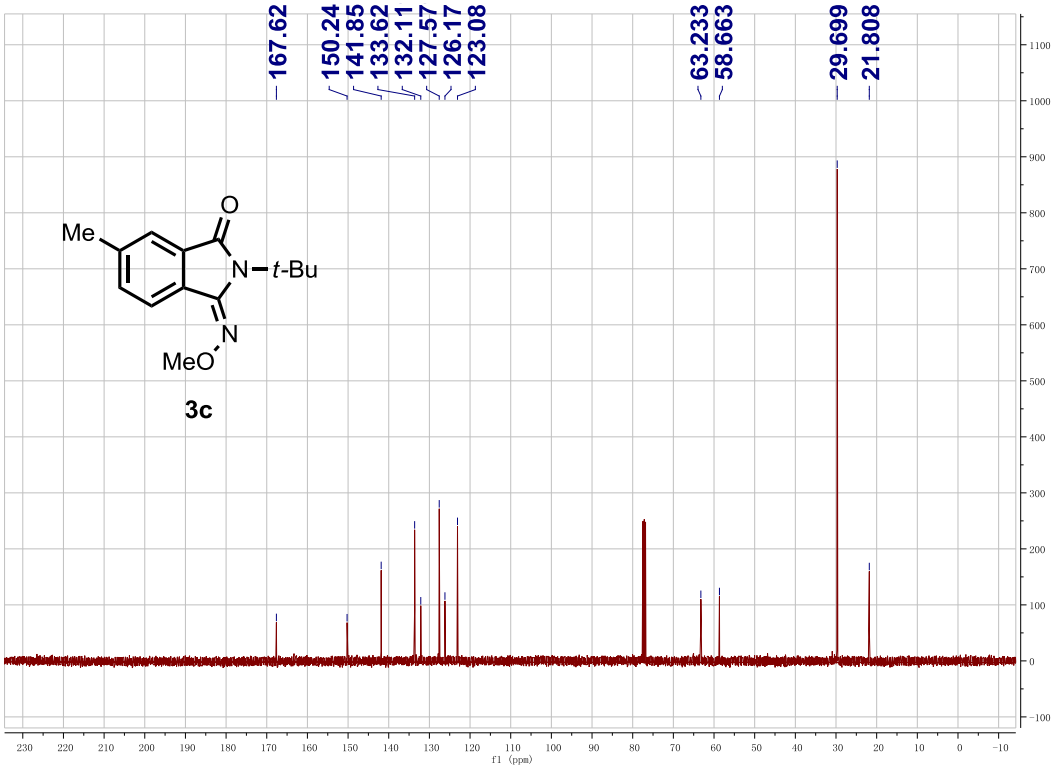

8.257
8.255
8.254
8.239
8.237
8.236
7.768
7.766
7.765
7.762
7.750
7.748
7.746
7.576
7.561
7.557
7.543
7.539
7.534
7.530
7.515
7.513
4.061
1.758
O
N–t-Bu
N
MeO
3a
1.00
1.01
2.07
3.04
9.12
f1 (ppm)

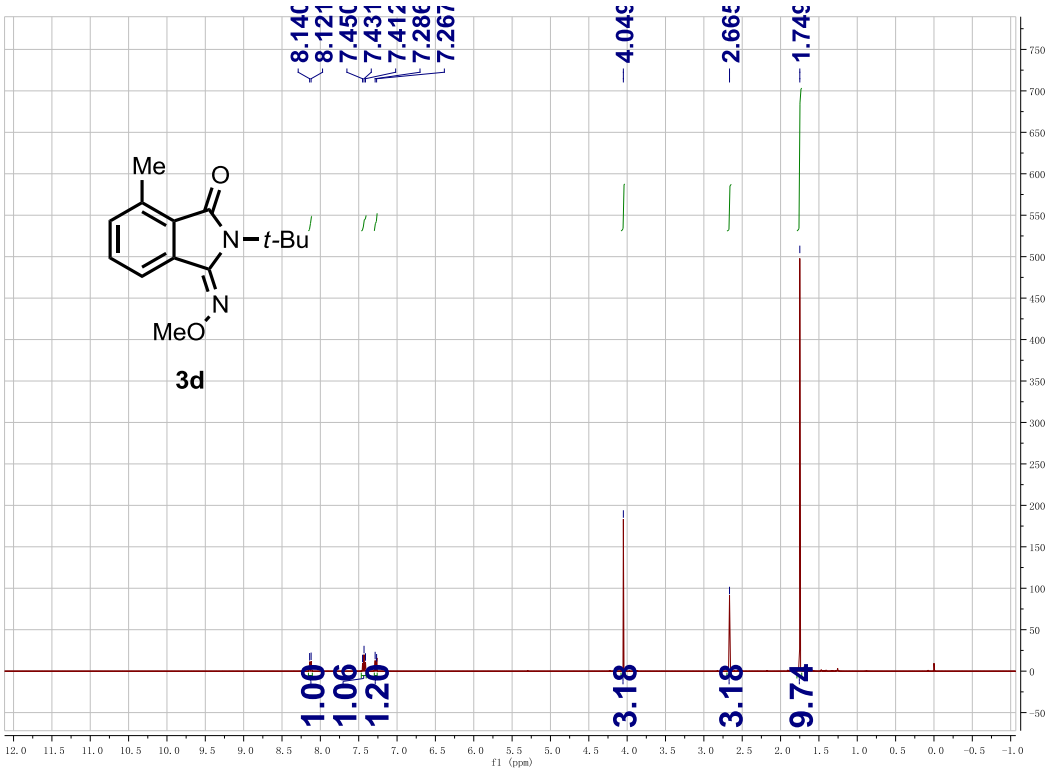

167.32
149.98
132.78
131.74
131.09
128.53
127.72
122.64
63.244
58.651
29.599
O
N–t-Bu
N
MeO
3a
f1 (ppm)

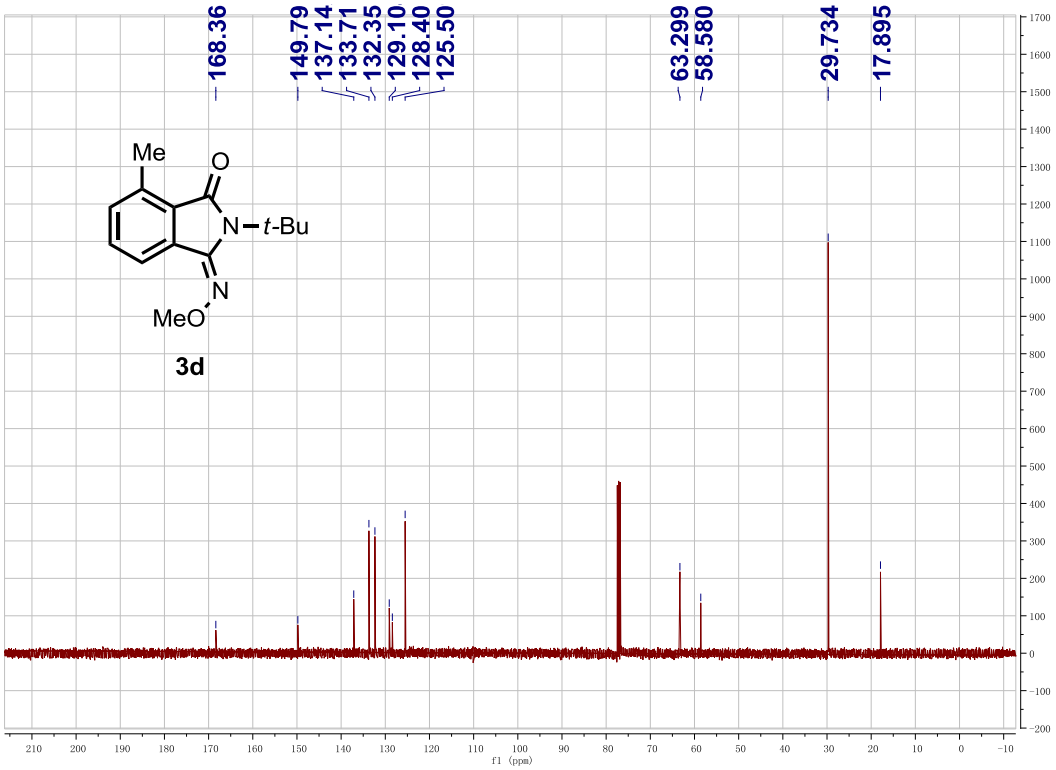

8.071
7.651
7.634
7.345
7.326
4.066
2.455
1.743
1.00
1.00
1.05
3.25
3.27
9.75
f1 (ppm)
O
N–t-Bu
Me
N
MeO
3b

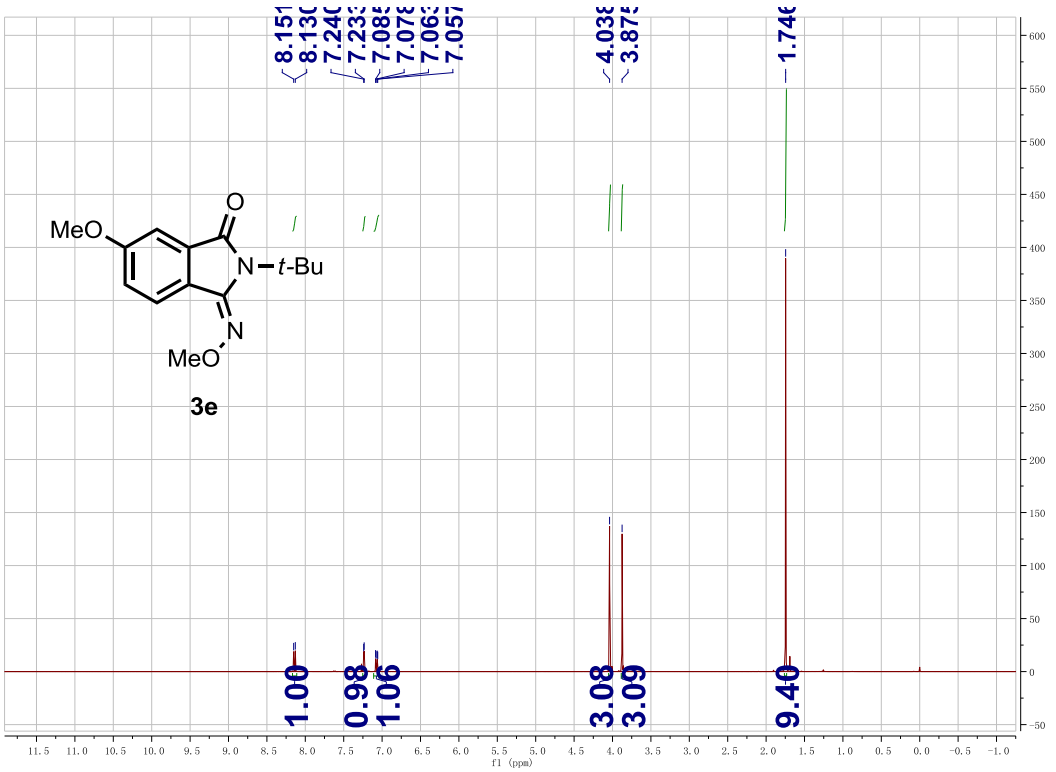

167.57
150.14
143.63
132.00
129.42
128.92
128.22
122.60
63.325
58.638
29.675
22.170
f1 (ppm)
O
N–t-Bu
Me
N
MeO
3b

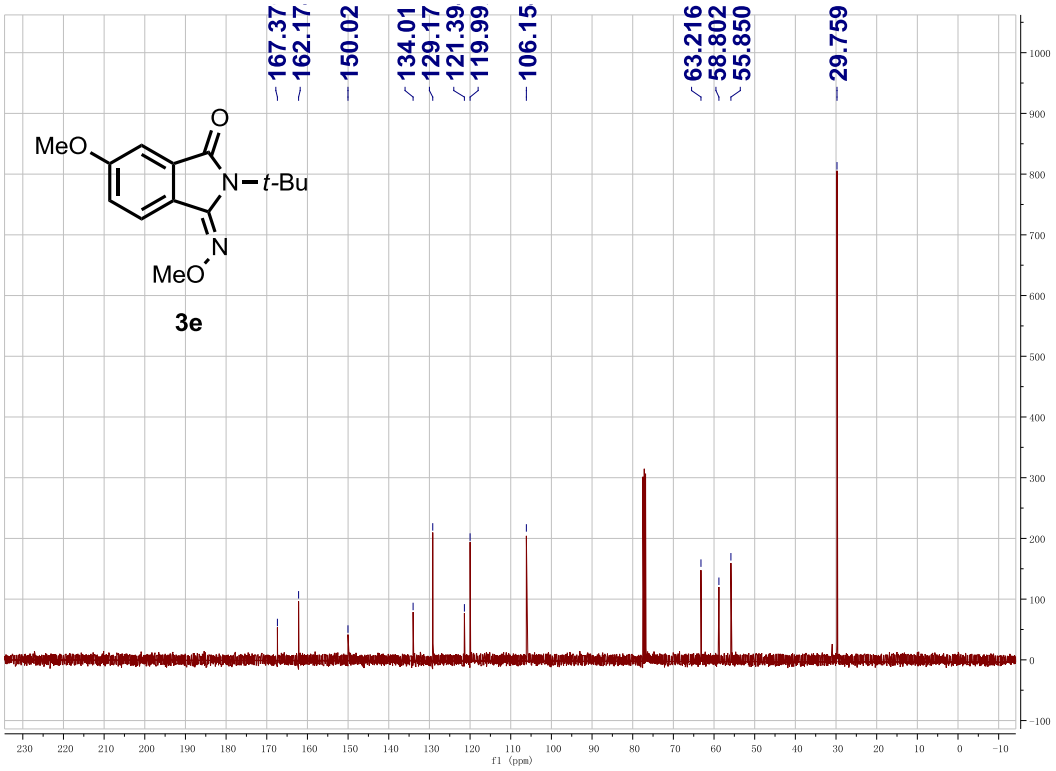

8.121
8.102
7.552
7.372
7.370
7.352
7.350
4.047
2.441
1.745
Me
O
N–t-Bu
N
MeO
3c
1.00
0.98
1.09
3.14
3.25
9.73
f1 (ppm)

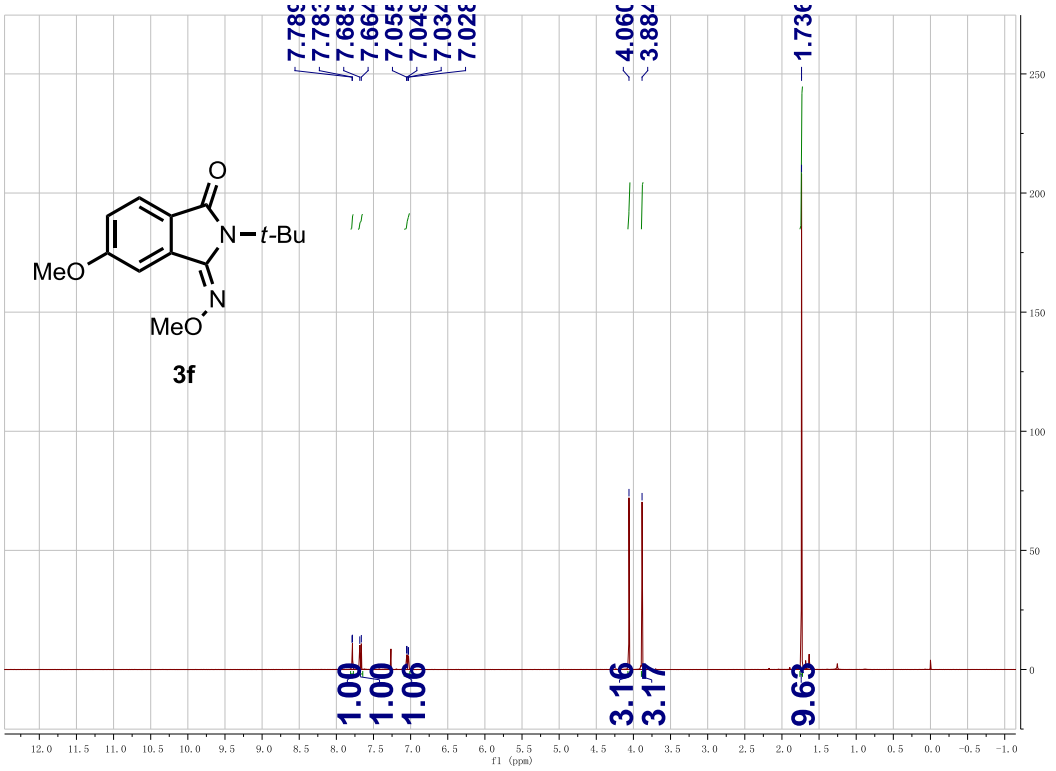

167.62
150.24
141.85
133.62
132.11
127.57
126.17
123.08
63.233
58.663
29.699
21.808
Me
O
N–t-Bu
N
MeO
3c
f1 (ppm)

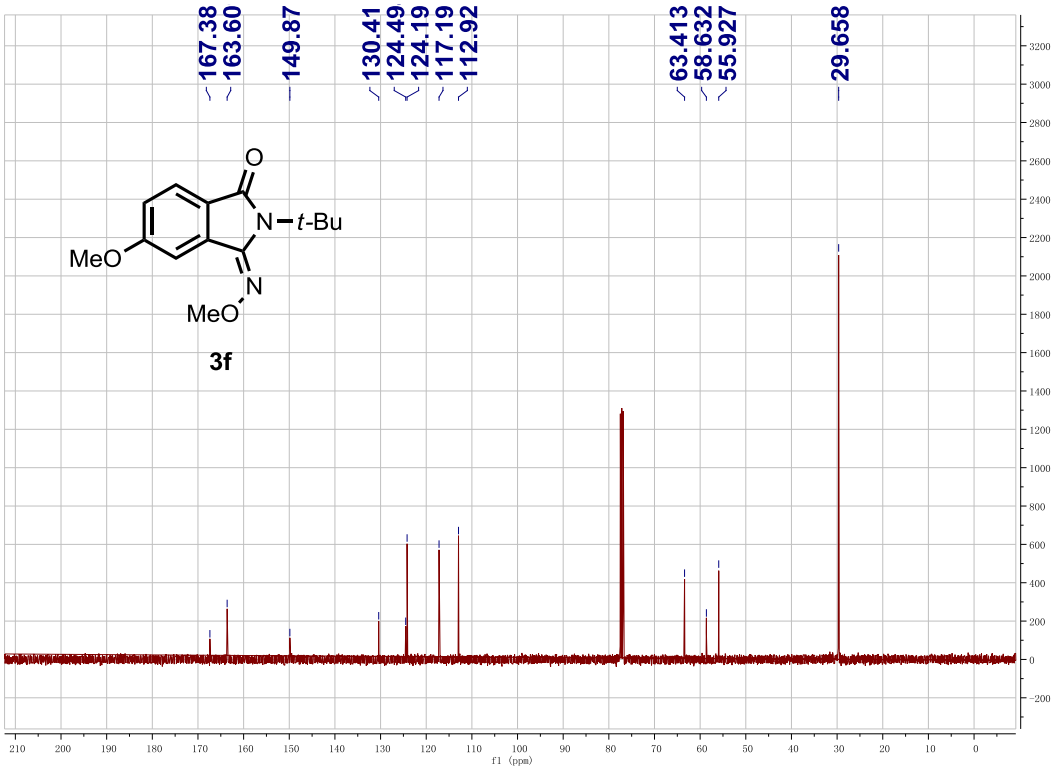

8.140
8.121
7.450
7.431
7.412
7.288
7.267
4.045
2.665
1.745
Me
O
N–t-Bu
N
MeO
3d
1.00
1.06
1.20
3.18
3.18
9.74
f1 (ppm)

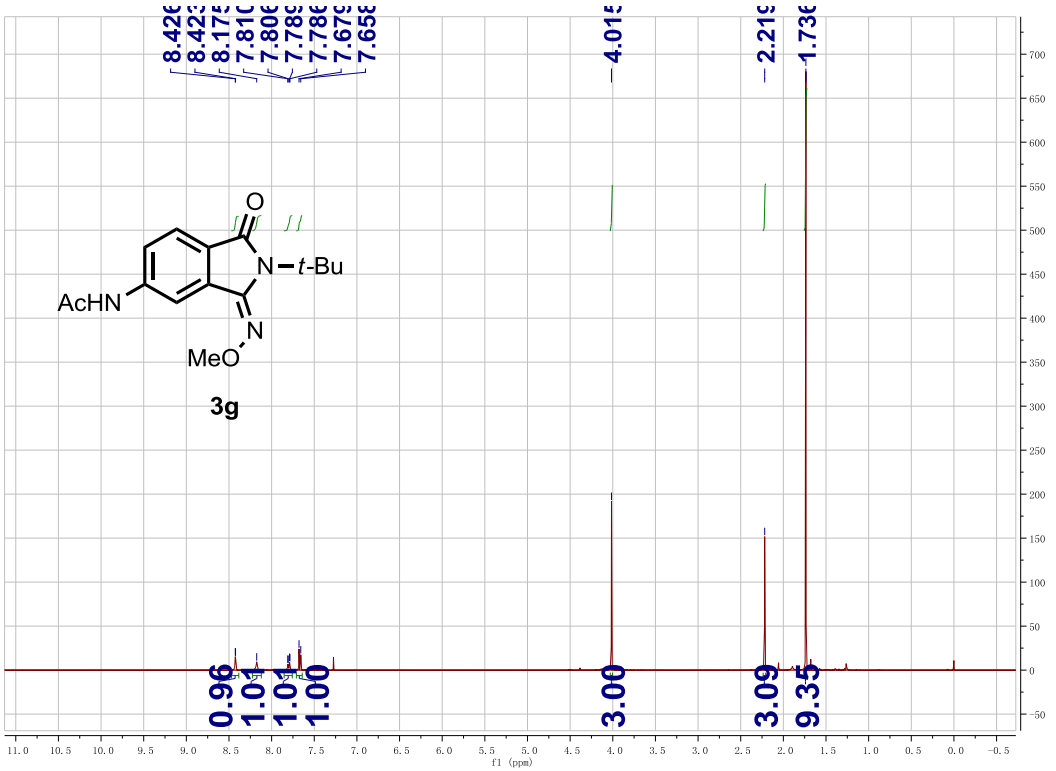

168.36
149.79
137.14
133.71
132.35
129.10
128.40
125.50
63.299
58.580
29.734
17.895
Me
O
N–t-Bu
N
MeO
3d
f1 (ppm)

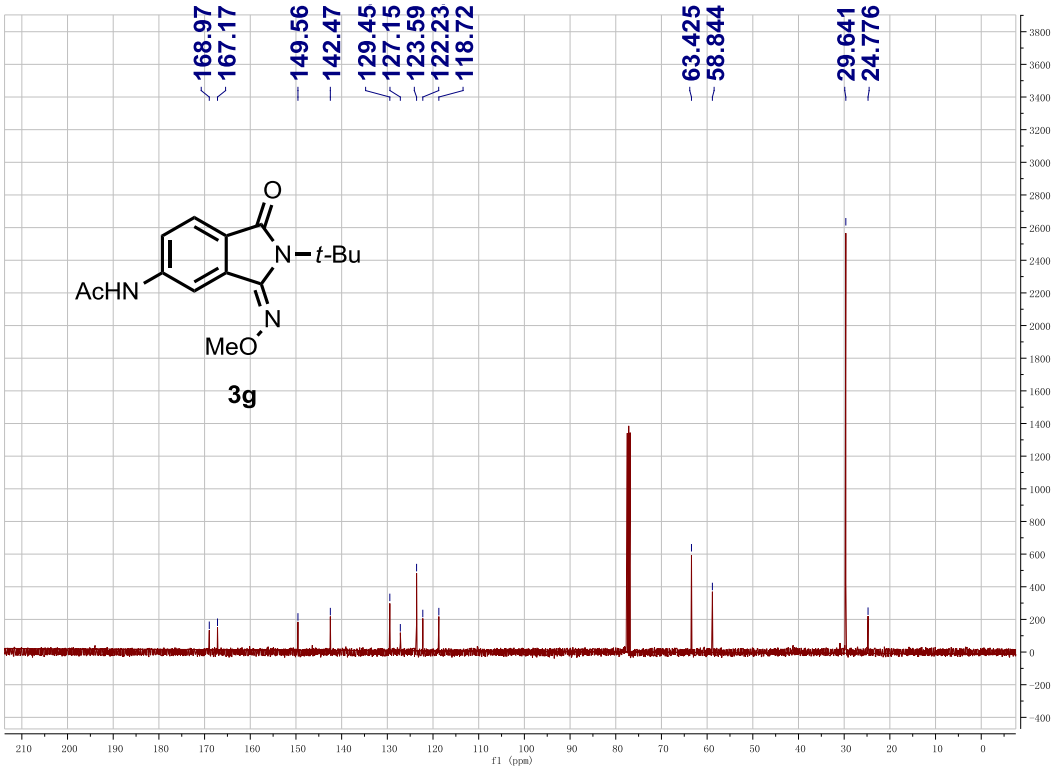

MeO
O
N–t-Bu
N
MeO
3e
1.00
0.98
1.06
3.08
3.09
9.40
f1 (ppm)

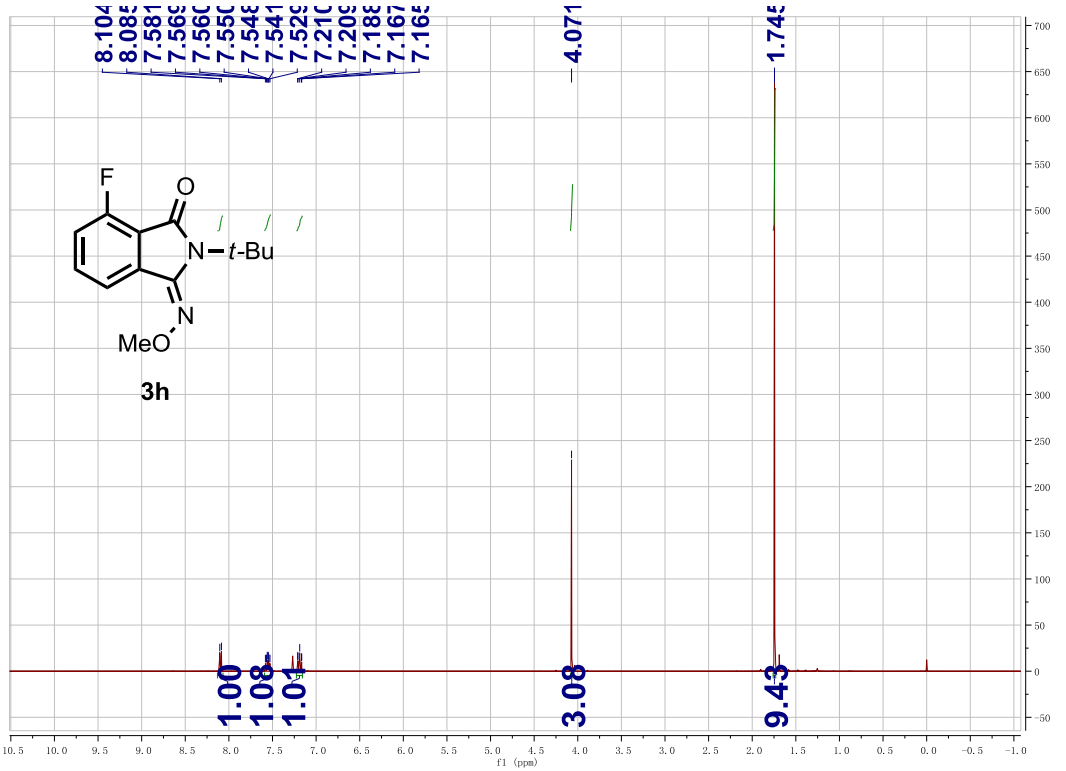

MeO
O
N–t-Bu
N
MeO
3e
167.37
162.17
150.02
134.01
129.17
121.39
119.99
106.15
63.216
58.802
55.850
29.759
f1 (ppm)

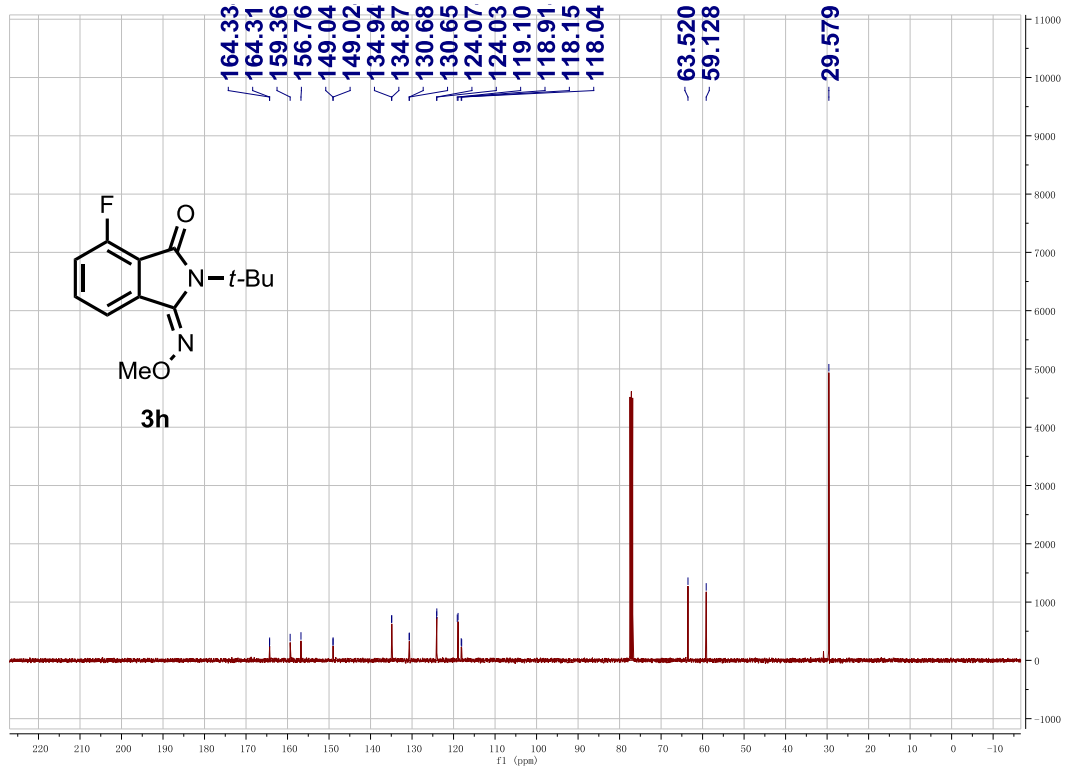

7.785
7.783
7.685
7.662
7.052
7.045
7.034
7.028
4.060
3.882
1.736
O
N–t-Bu
MeO
N
MeO
3f
1.00
1.00
1.06
3.16
3.17
9.63
f1 (ppm)

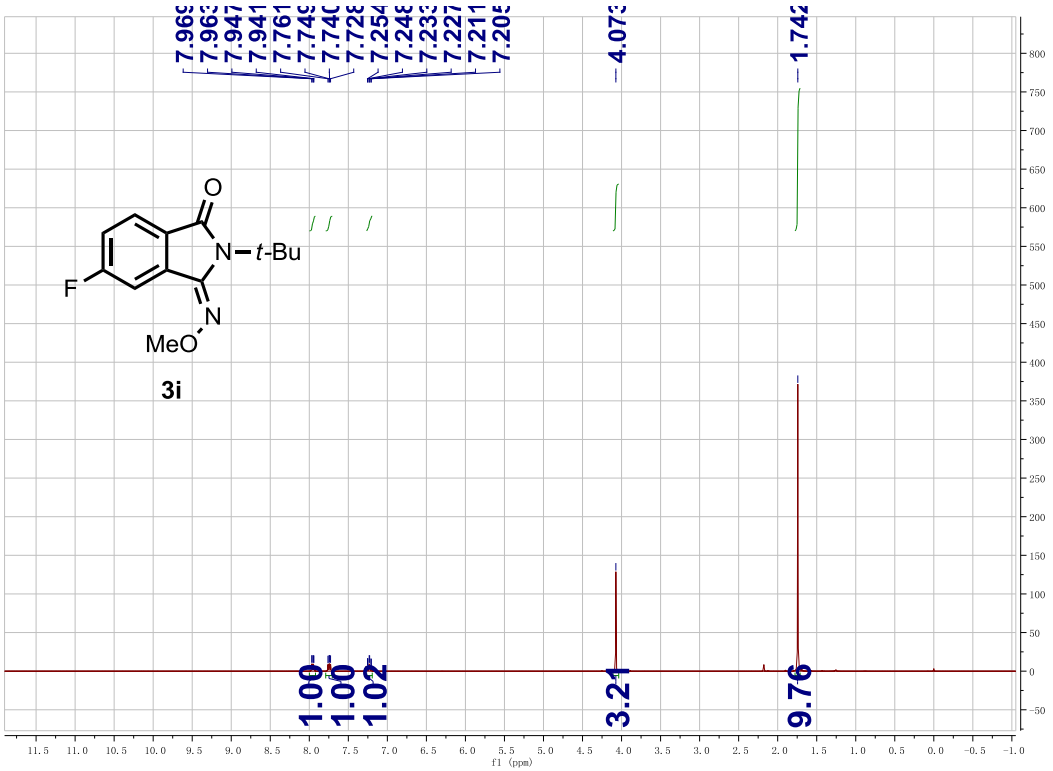

167.38
163.60
149.87
130.41
124.49
124.19
117.19
112.92
63.413
58.632
55.927
29.658
O
N–t-Bu
MeO
N
MeO
3f
f1 (ppm)

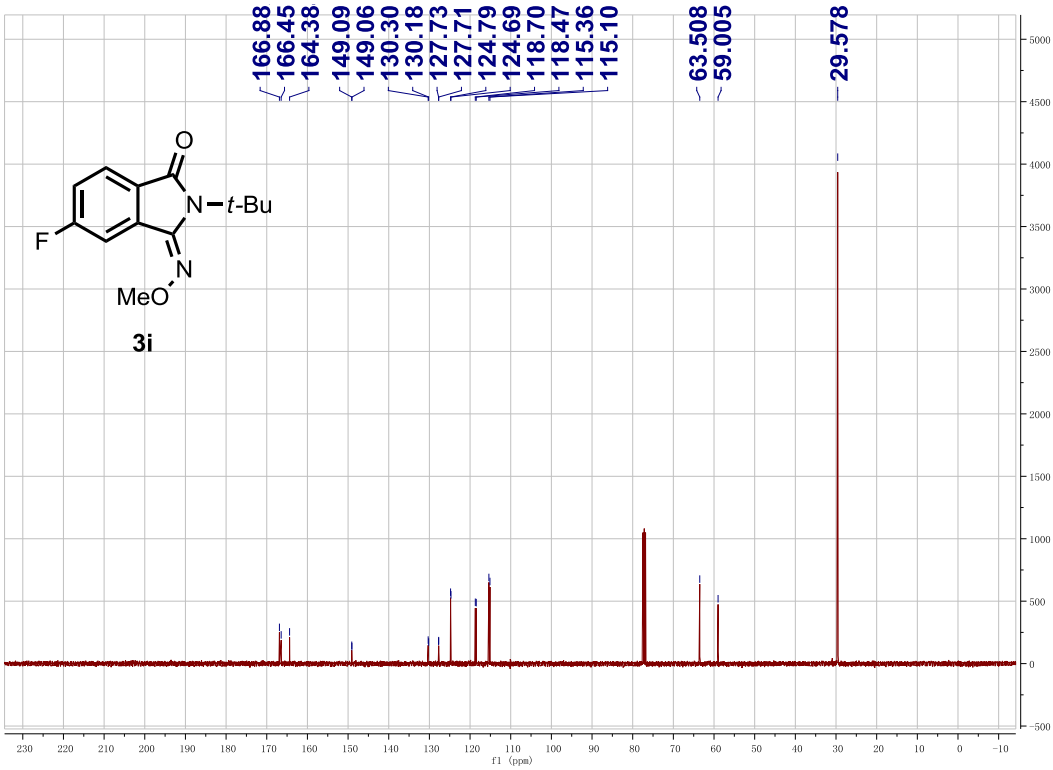

8.426
8.423
8.175
7.810
7.806
7.785
7.780
7.675
7.658
4.015
2.215
1.736
O
N–t-Bu
AcHN
N
MeO
3g
0.96
1.02
1.02
1.00
3.00
3.09
9.35
f1 (ppm)

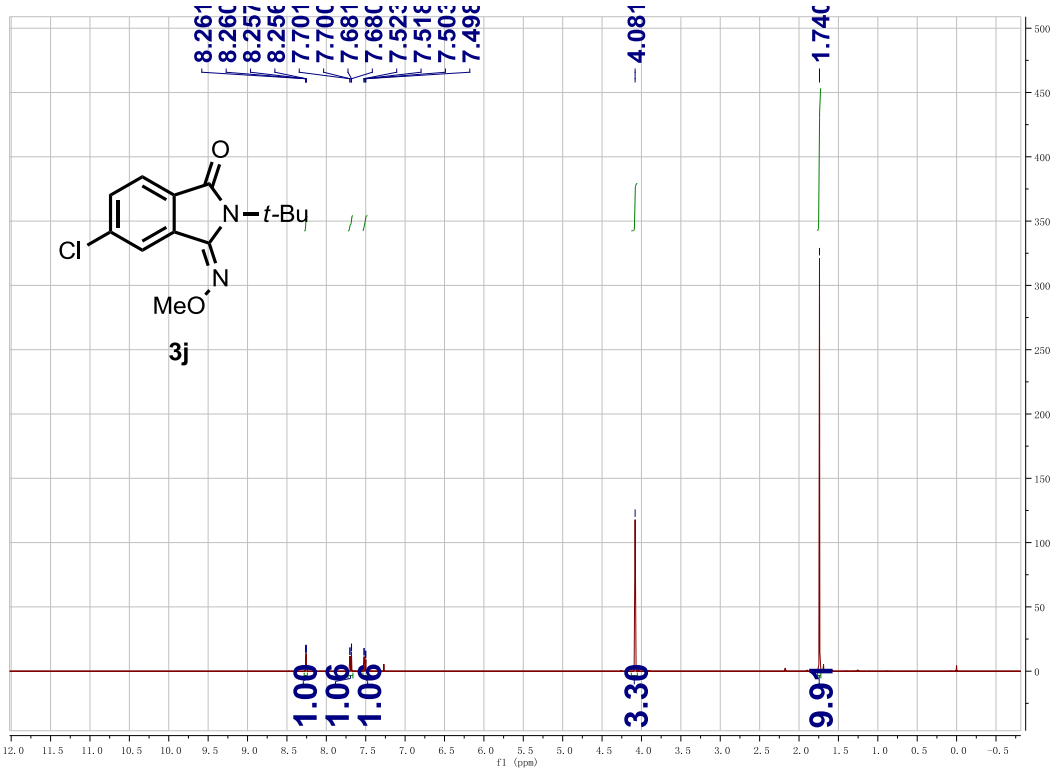

168.97
167.17
149.56
142.47
129.45
127.15
123.59
122.23
118.72
63.425
58.844
29.641
24.776
O
N–t-Bu
AcHN
N
MeO
3g
f1 (ppm)

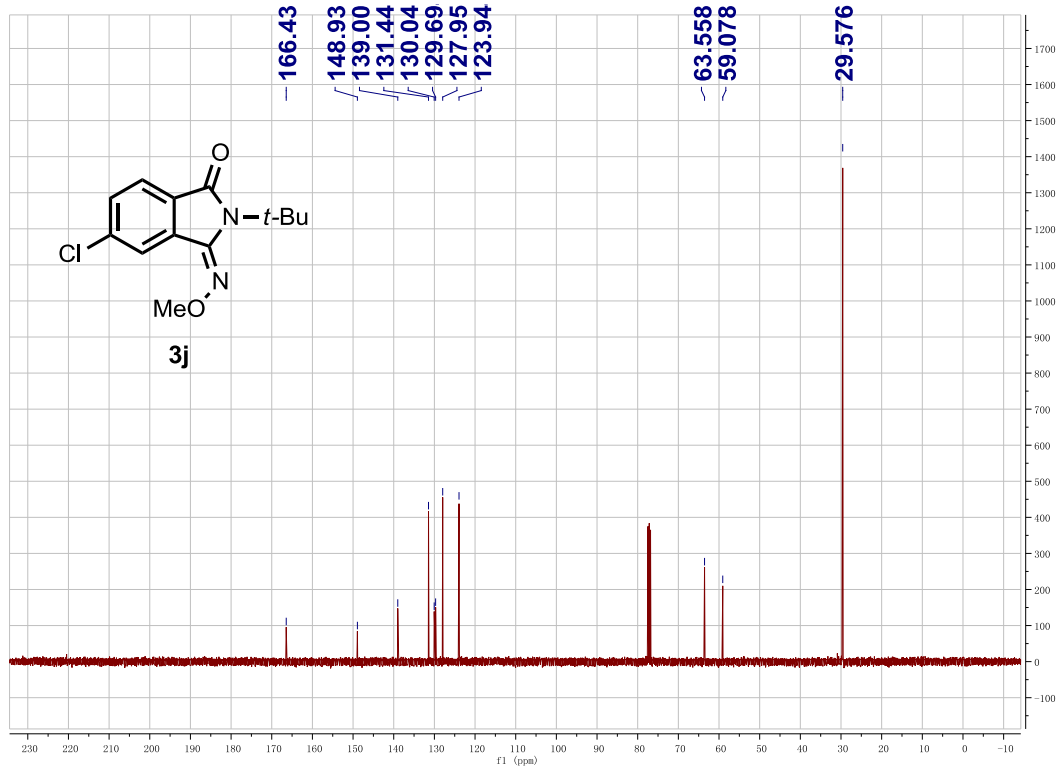

F
O
N–t-Bu
N
MeO
3h
1.00
1.08
1.01
3.08
9.43
f1 (ppm)

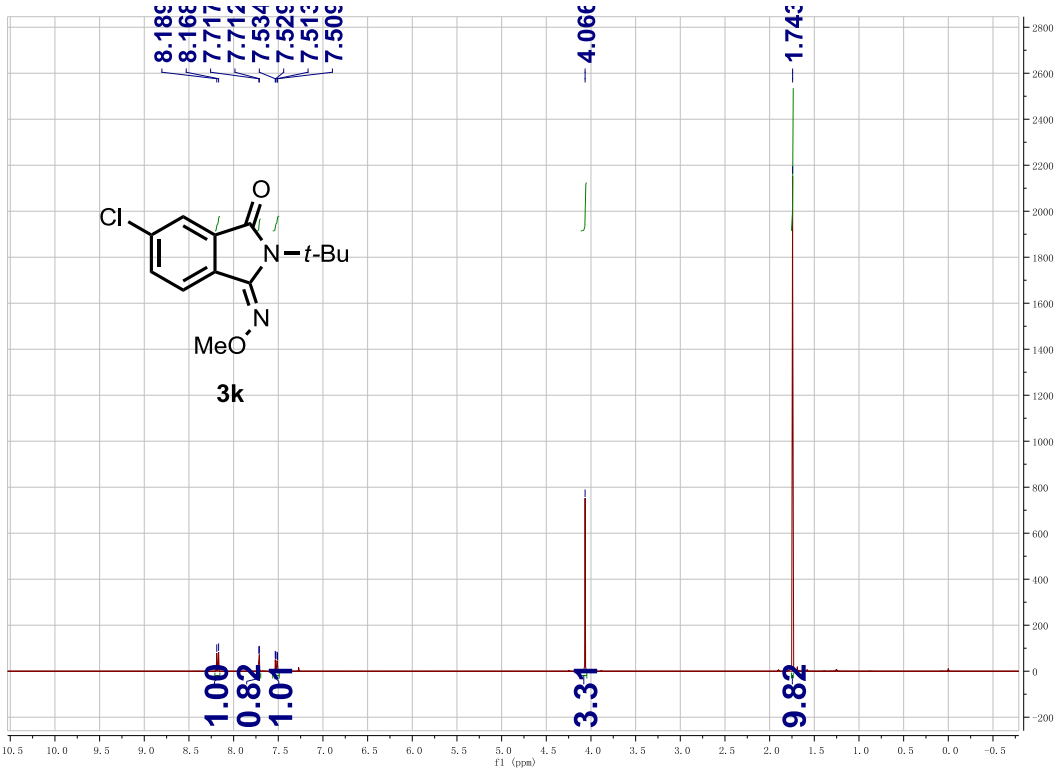

164.33
164.31
159.36
156.76
149.04
149.02
134.94
134.87
130.68
130.65
124.07
124.03
119.10
118.91
118.15
118.04
63.520
59.128
29.579
F
O
N–t-Bu
N
MeO
3h
f1 (ppm)

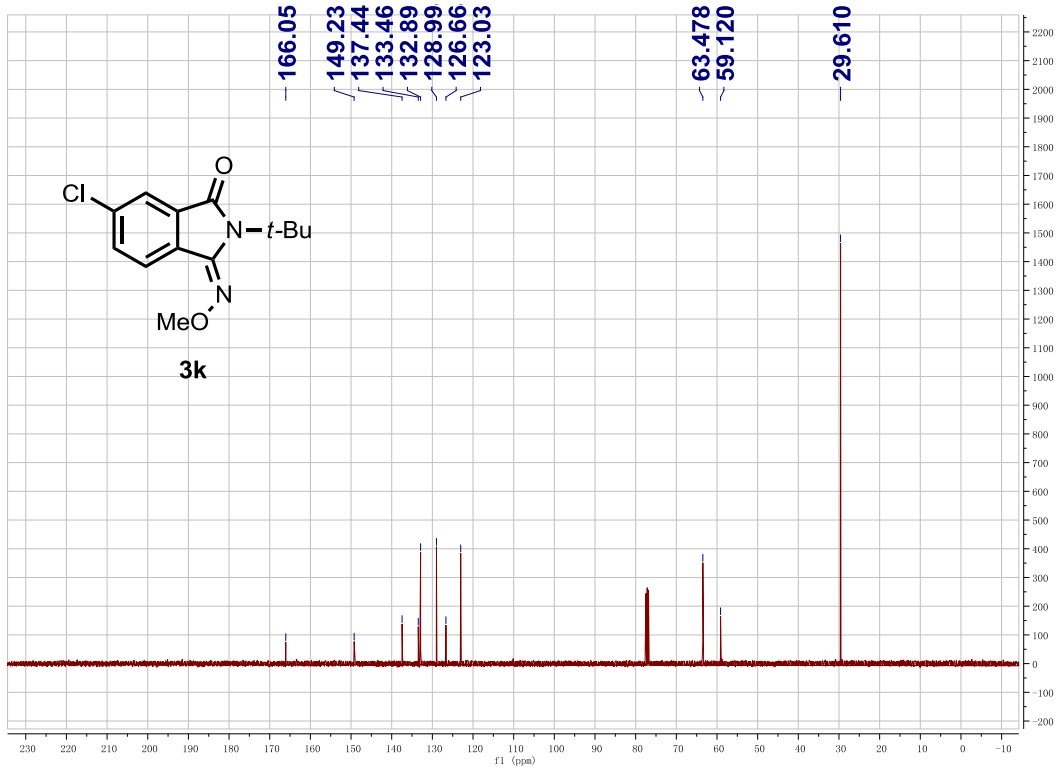

7.965
7.963
7.947
7.941
7.761
7.745
7.740
7.728
7.254
7.248
7.233
7.227
7.211
7.205
4.073
1.742
1.00
1.00
1.02
3.21
9.76
f1 (ppm)

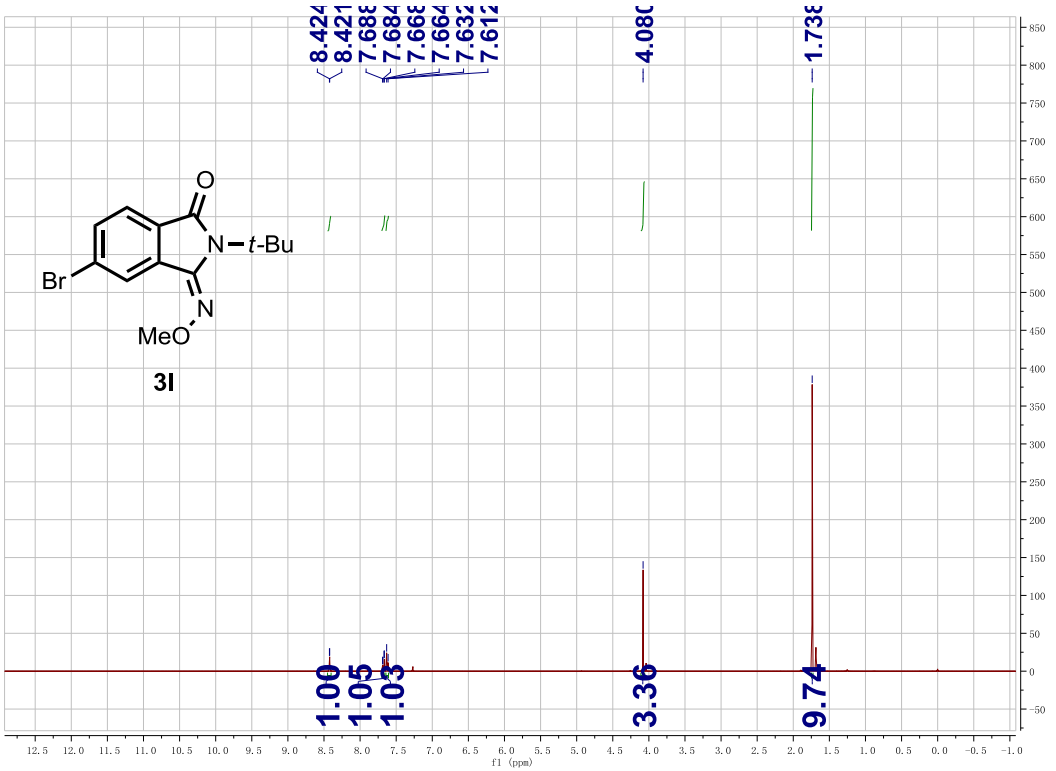

166.88
166.45
164.38
149.09
149.06
130.30
130.18
127.73
127.71
124.79
124.69
118.70
118.47
115.36
115.10
63.508
59.005
29.578
f1 (ppm)

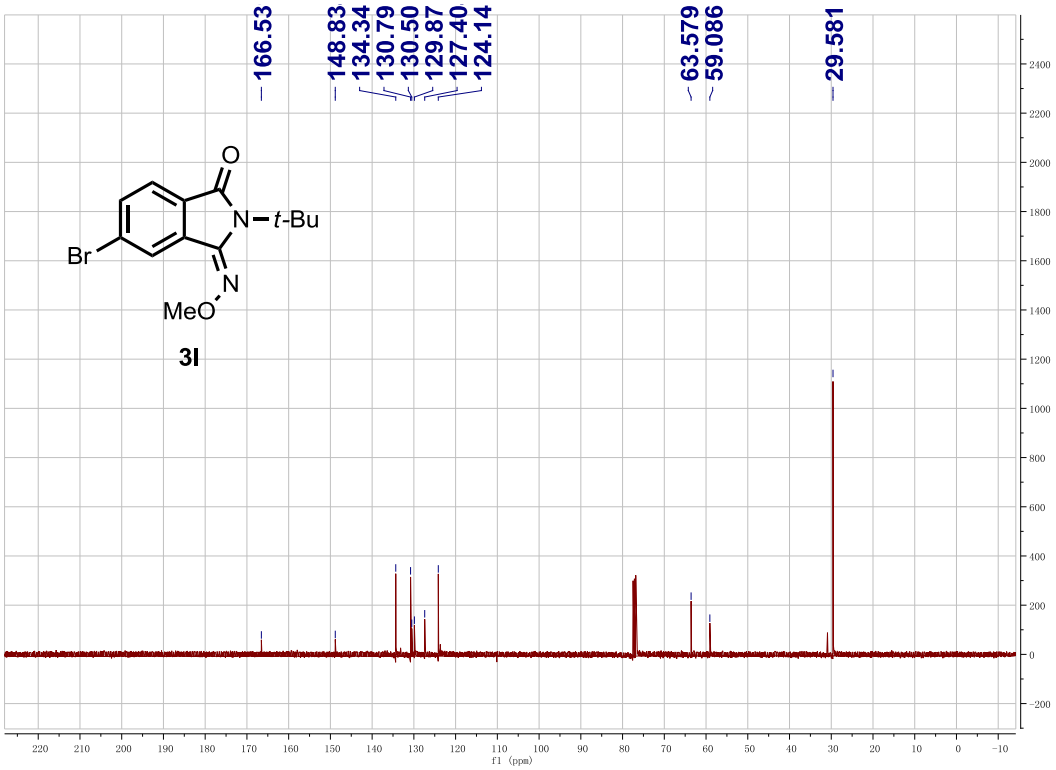

8.261
8.260
8.257
8.256
7.701
7.700
7.681
7.680
7.523
7.518
7.503
7.498
4.081
1.740
O
N–t-Bu
Cl
N
MeO
3j
1.00
1.06
1.06
3.30
9.94
f1 (ppm)

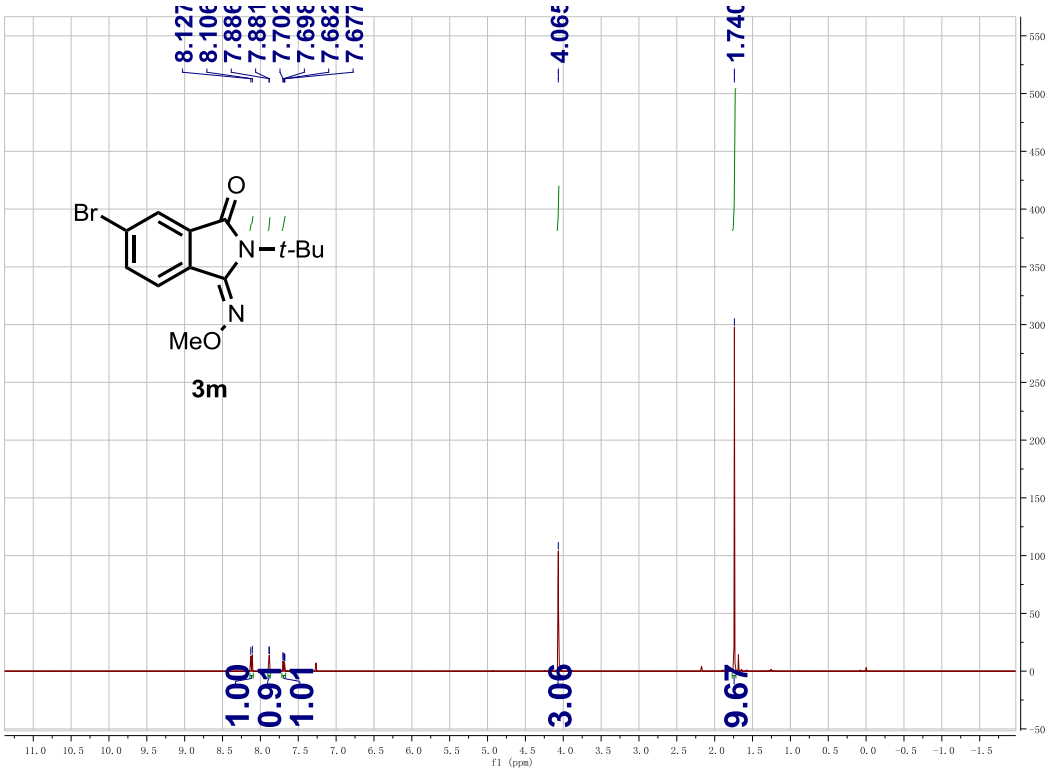

166.43
148.93
139.00
131.44
130.04
129.69
127.95
123.94
63.558
59.078
29.576
O
N–t-Bu
Cl
N
MeO
3j
f1 (ppm)

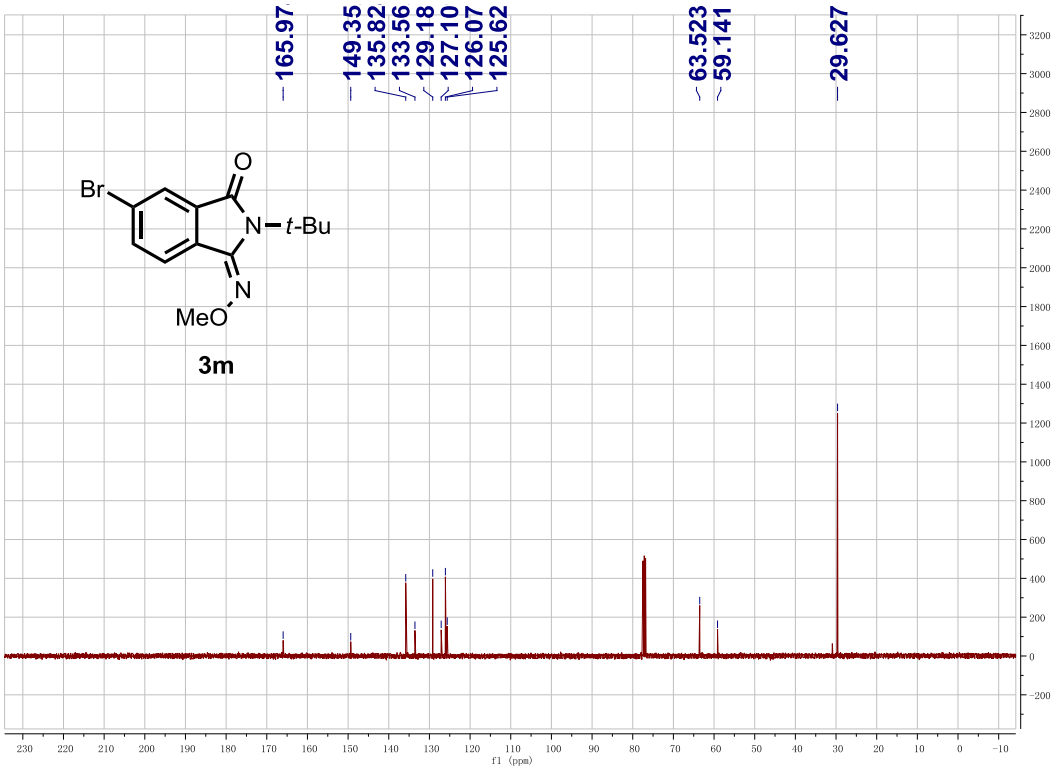

8.185
8.168
7.717
7.712
7.534
7.529
7.513
7.505
4.066
1.743
Cl
O
N–t-Bu
N
MeO
3k
1.00
0.82
1.01
3.31
9.82
f1 (ppm)

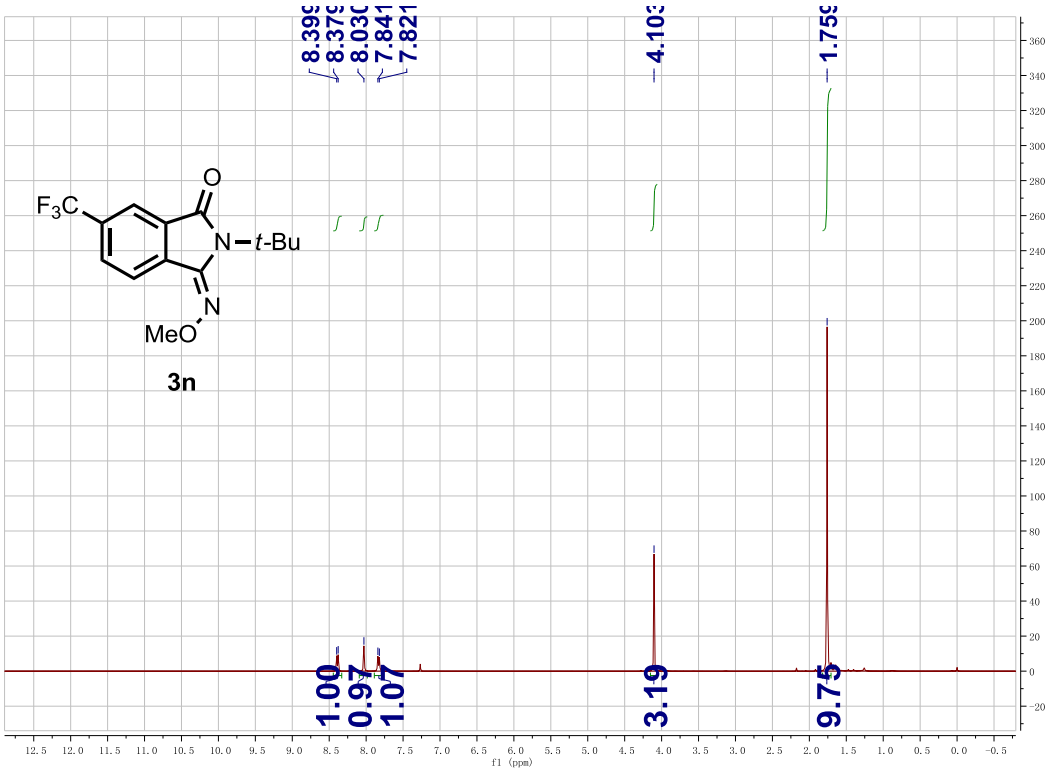

166.05
149.23
137.44
133.46
132.89
128.99
126.66
123.03
63.478
59.120
29.610
Cl
O
N–t-Bu
N
MeO
3k
f1 (ppm)

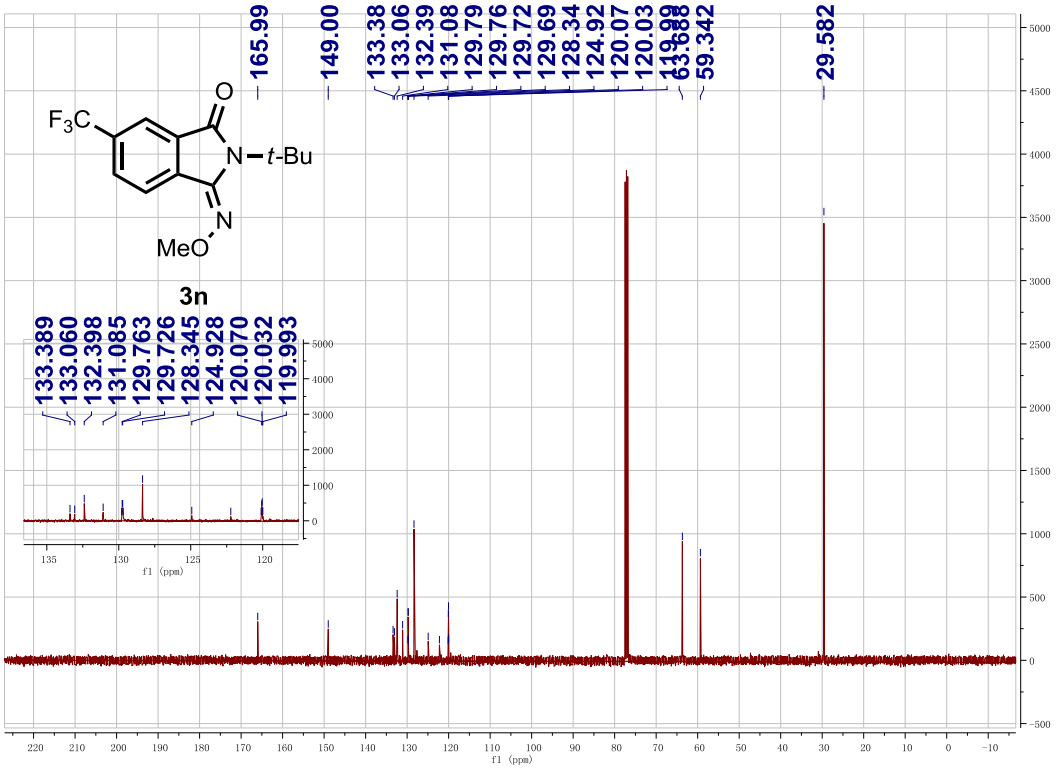

8.424
8.421
7.688
7.684
7.666
7.664
7.632
7.612
4.080
1.738
O
N–t-Bu
Br
N
MeO
3l
1.00
1.05
1.03
3.36
9.74
f1 (ppm)

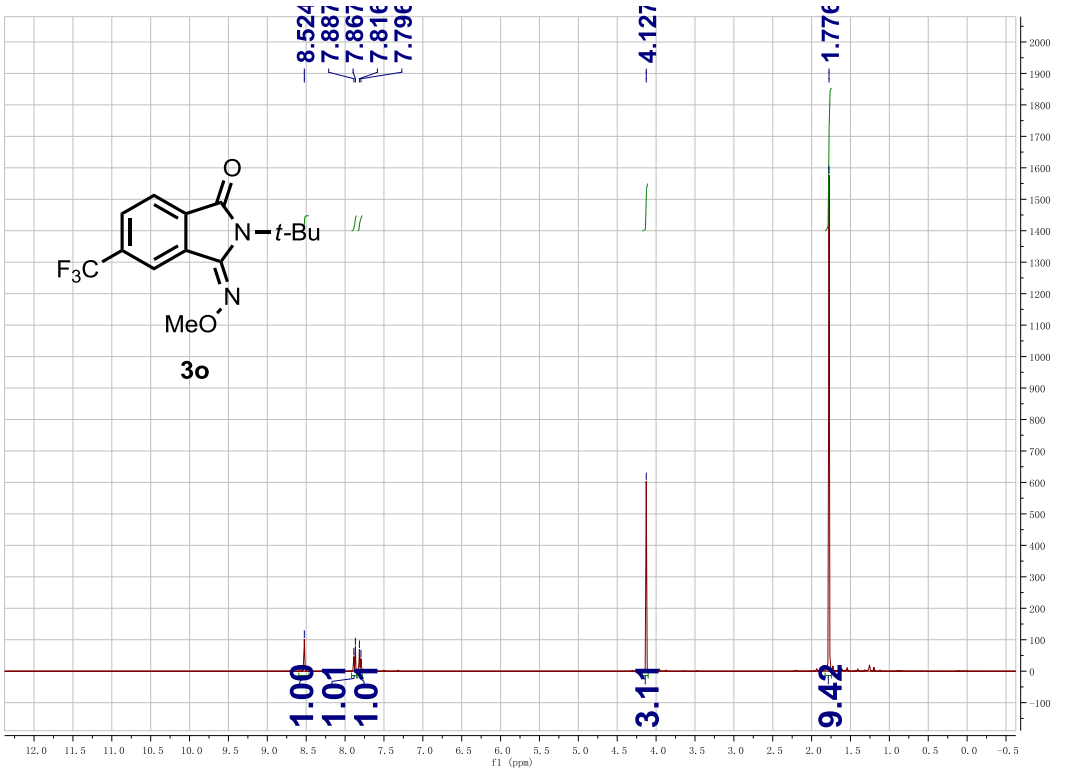

166.53
148.83
134.34
130.79
130.50
129.87
127.40
124.14
63.579
59.086
29.581
O
N–t-Bu
Br
N
MeO
3l
f1 (ppm)

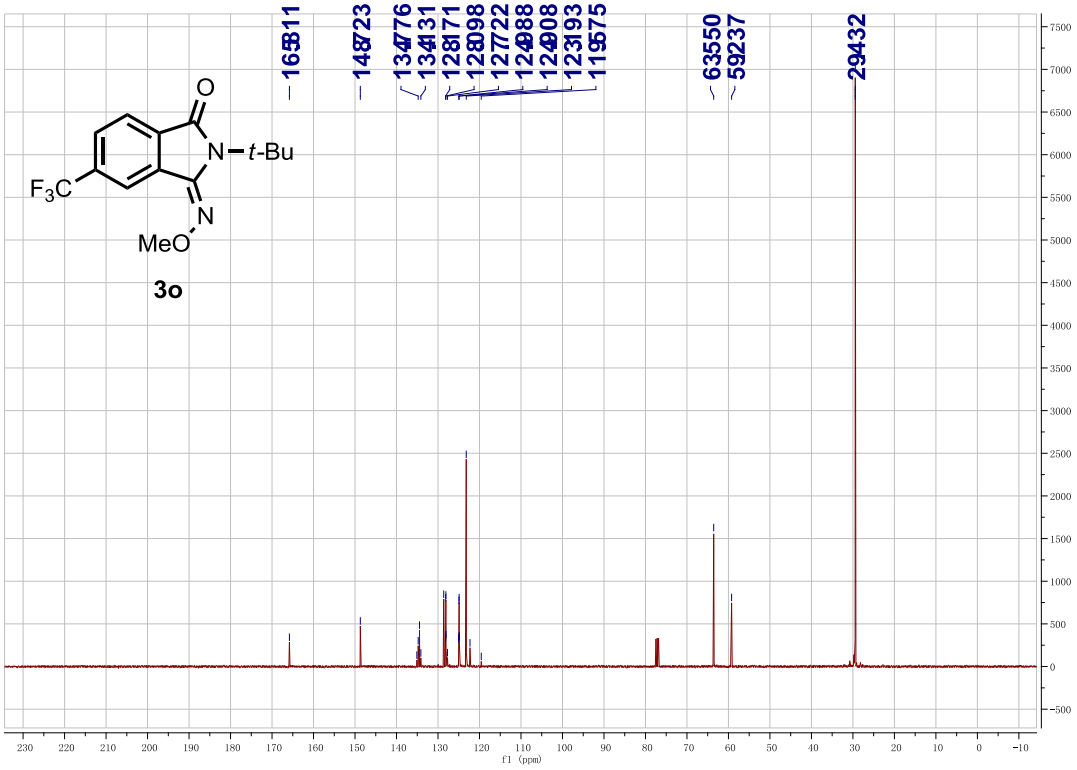

8.127
8.106
7.886
7.881
7.702
7.698
7.682
7.677
4.065
1.740
Br
O
N–t-Bu
N
MeO
3m
1.00
0.97
1.01
3.06
9.67
f1 (ppm)

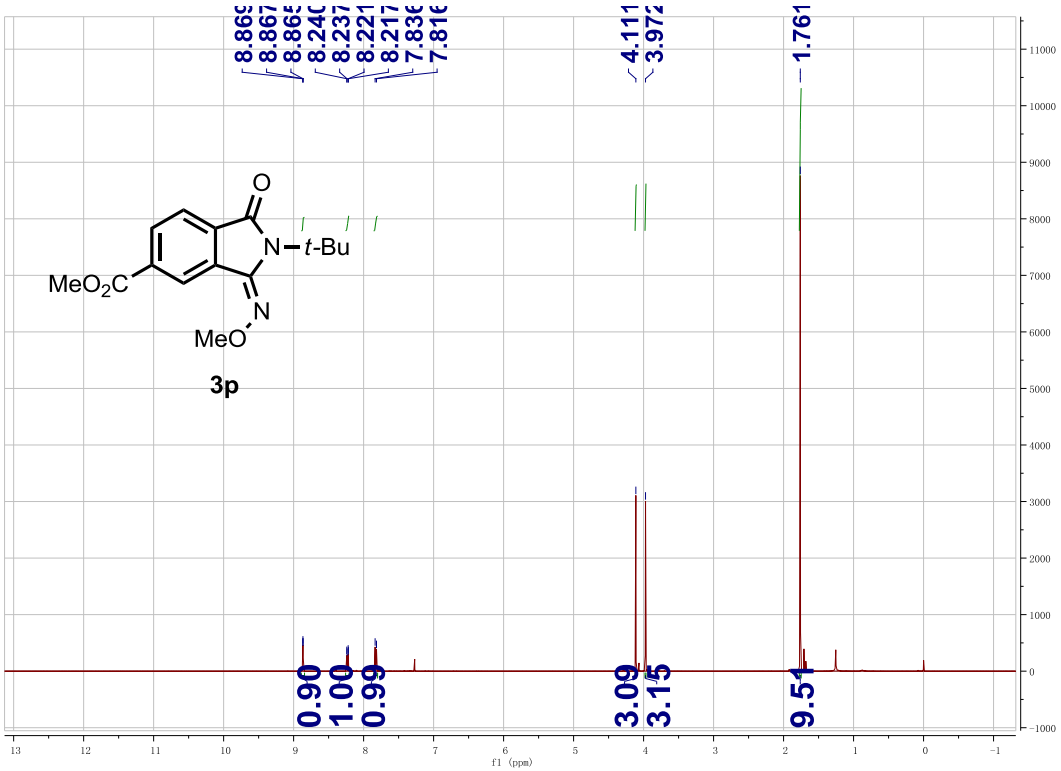

165.97
149.35
135.82
133.56
129.18
127.10
126.07
125.62
63.523
59.141
29.627
Br
O
N–t-Bu
N
MeO
3m
f1 (ppm)

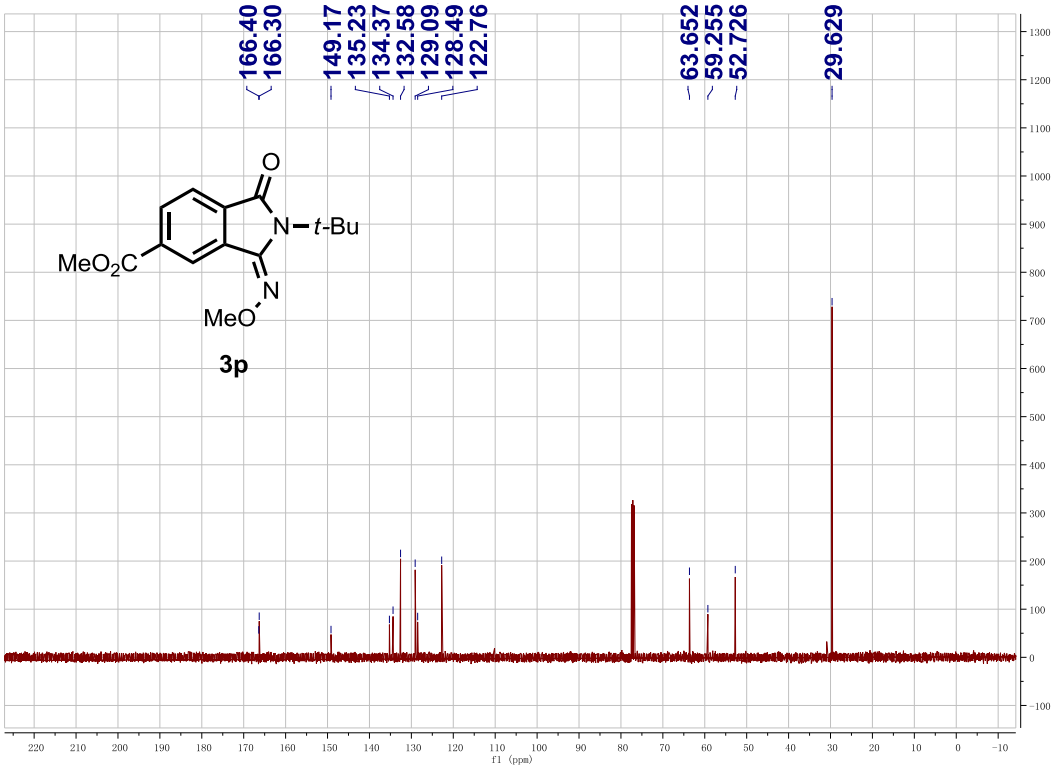

8.399
8.375
8.030
7.841
7.821
4.103
1.759
F3C
O
N–t-Bu
N
MeO
3n
1.00
0.97
1.07
3.19
9.75
f1 (ppm)

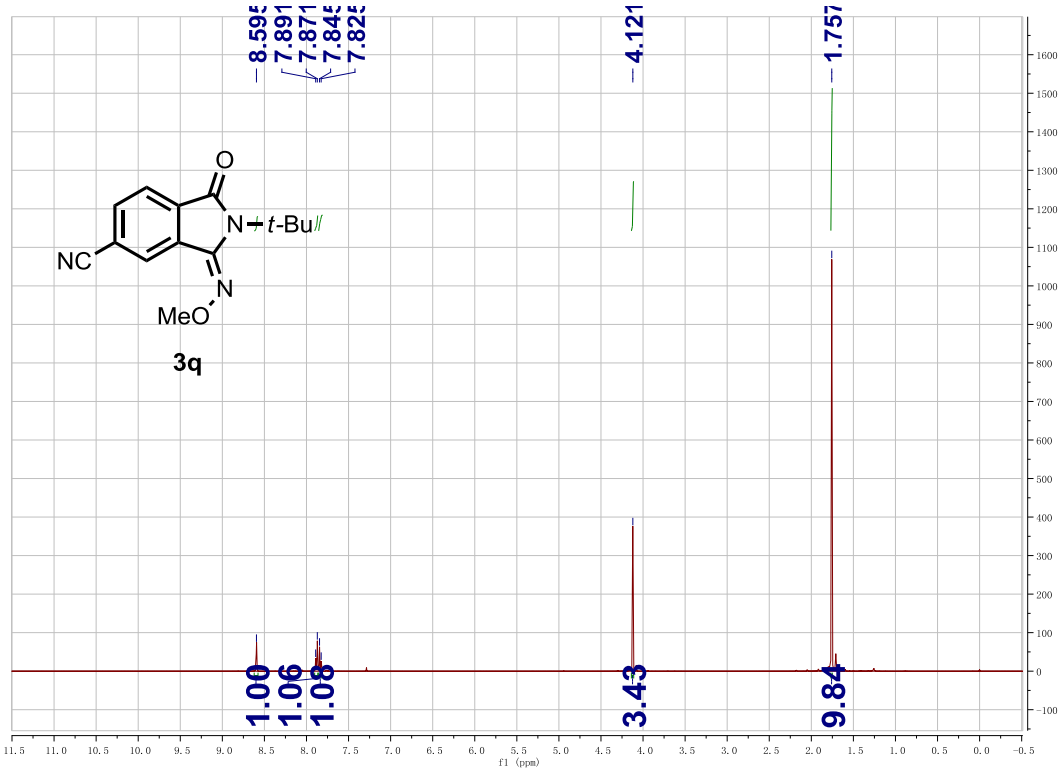

165.99
149.00
133.38
133.06
132.39
131.08
129.79
129.76
129.72
129.69
128.34
124.92
120.07
120.03
119.99
63.688
59.342
29.582
F3C
O
N–t-Bu
N
MeO
3n
133.389
133.060
132.398
131.085
129.763
129.726
128.345
124.928
120.070
120.032
119.993
f1 (ppm)

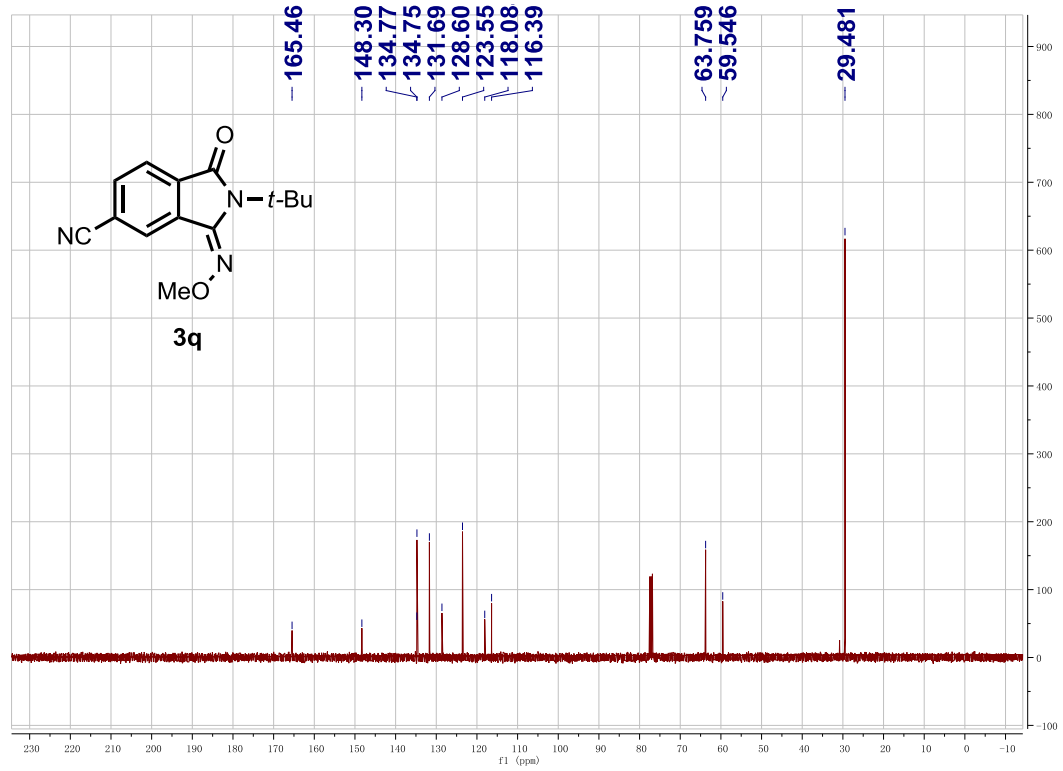

8.524
7.887
7.867
7.816
7.796
4.127
1.776
1.00
1.01
1.04
3.11
9.42
O
N–t-Bu
F3C
N
MeO
3o
f1 (ppm)

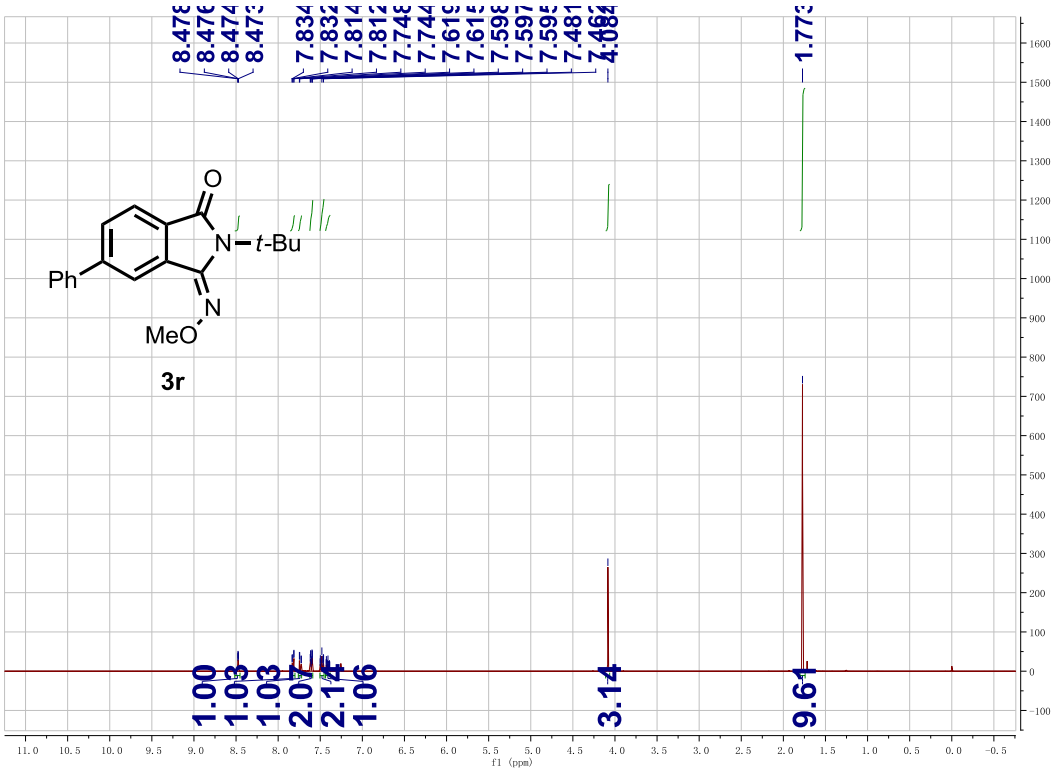

168.311
147.723
134.776
134.431
128.171
128.098
127.722
124.888
124.408
123.193
119.575
63.550
59.237
29.432
O
N–t-Bu
F3C
N
MeO
3o
f1 (ppm)

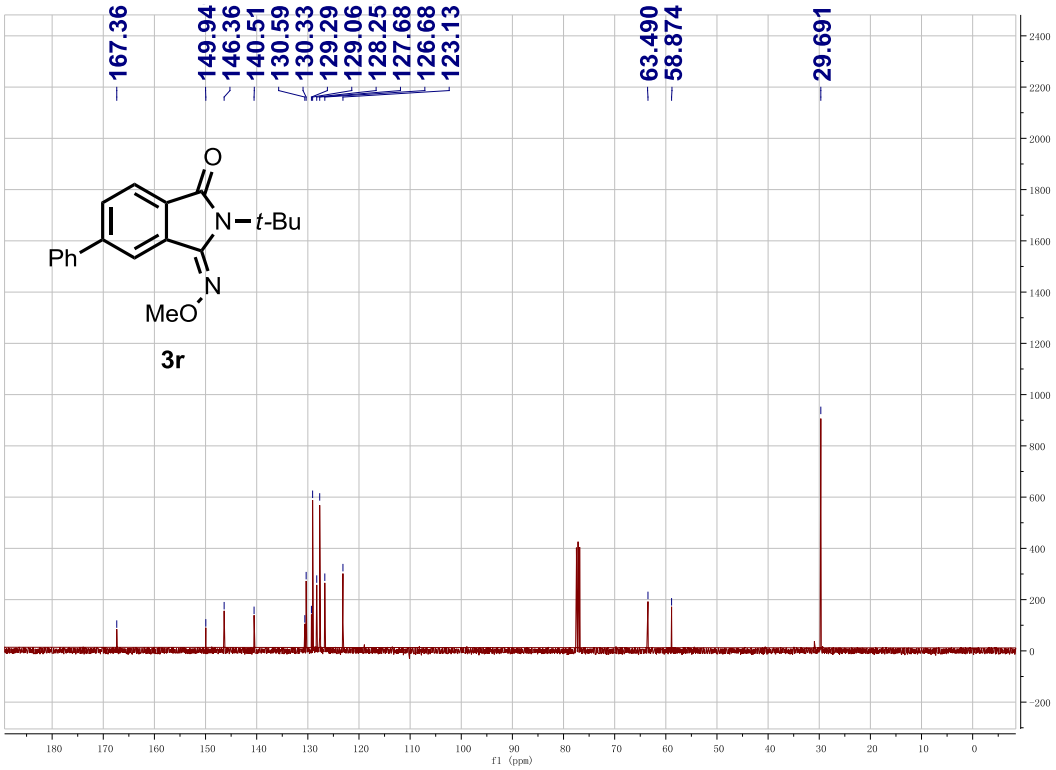

8.865
8.867
8.865
8.240
8.237
8.221
8.217
7.836
7.816
4.111
3.972
1.761
O
N–t-Bu
MeO2C
N
MeO
3p
0.90
1.00
0.99
3.09
3.15
9.54
f1 (ppm)

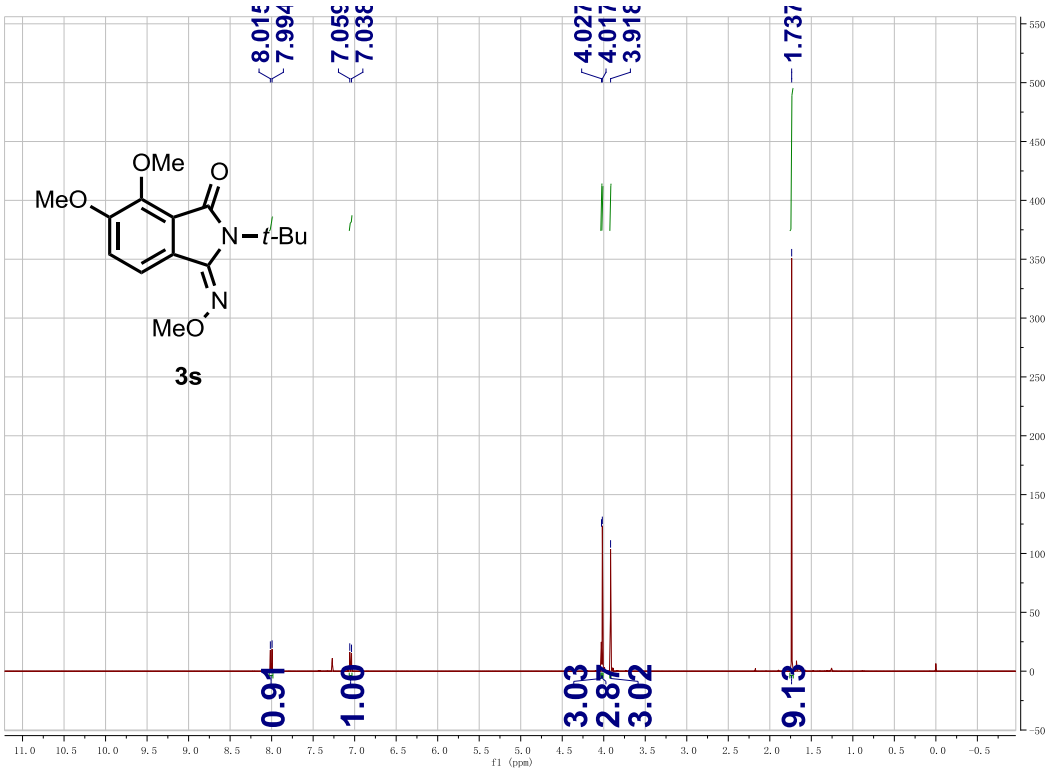

166.40
166.30
149.17
135.23
134.37
132.58
129.09
128.49
122.76
63.652
59.255
52.726
29.629
O
N–t-Bu
MeO2C
N
MeO
3p
f1 (ppm)

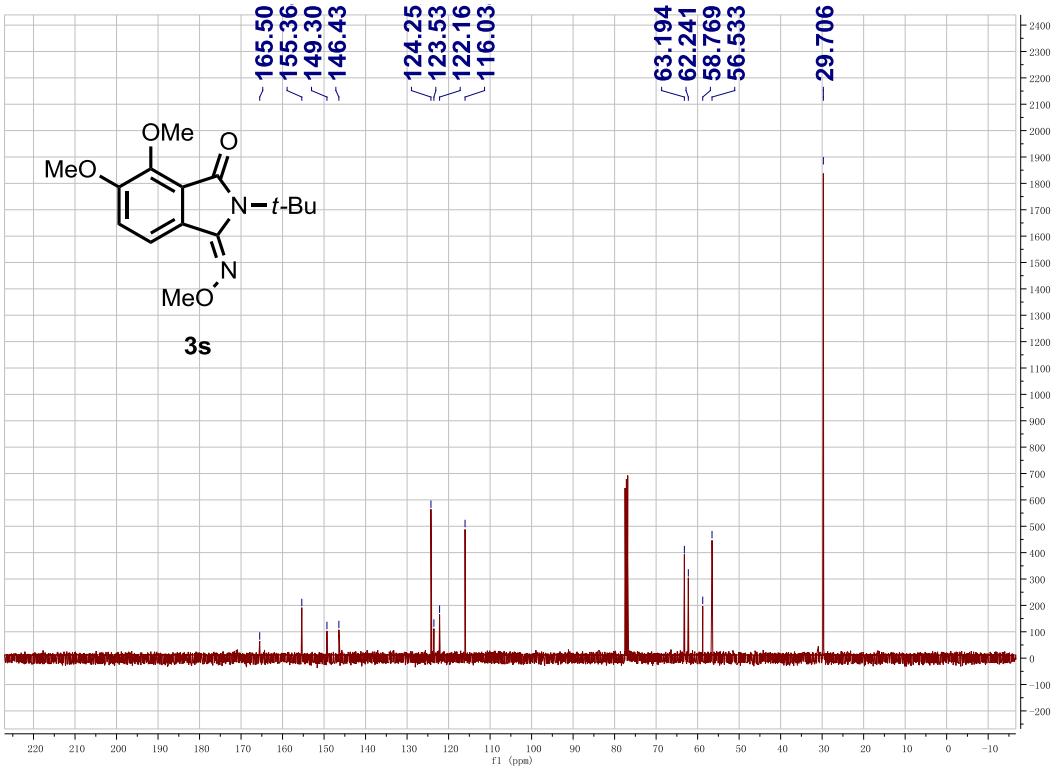

8.595
7.891
7.871
7.845
7.825
4.121
1.757
O
N
t-Bu
NC
N
MeO
3q
1.00
1.06
1.08
3.43
9.84
f1 (ppm)

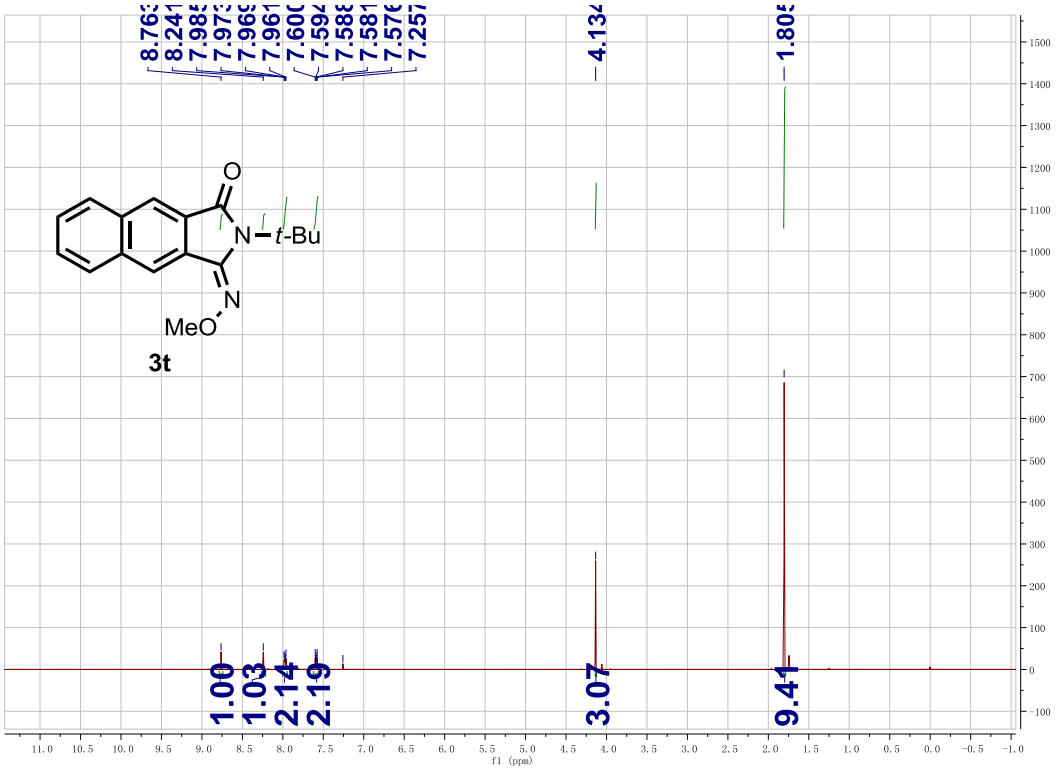

165.46
148.30
134.77
134.75
131.69
128.60
123.55
118.08
116.39
63.759
59.546
29.481
O
N
t-Bu
NC
N
MeO
3q
f1 (ppm)

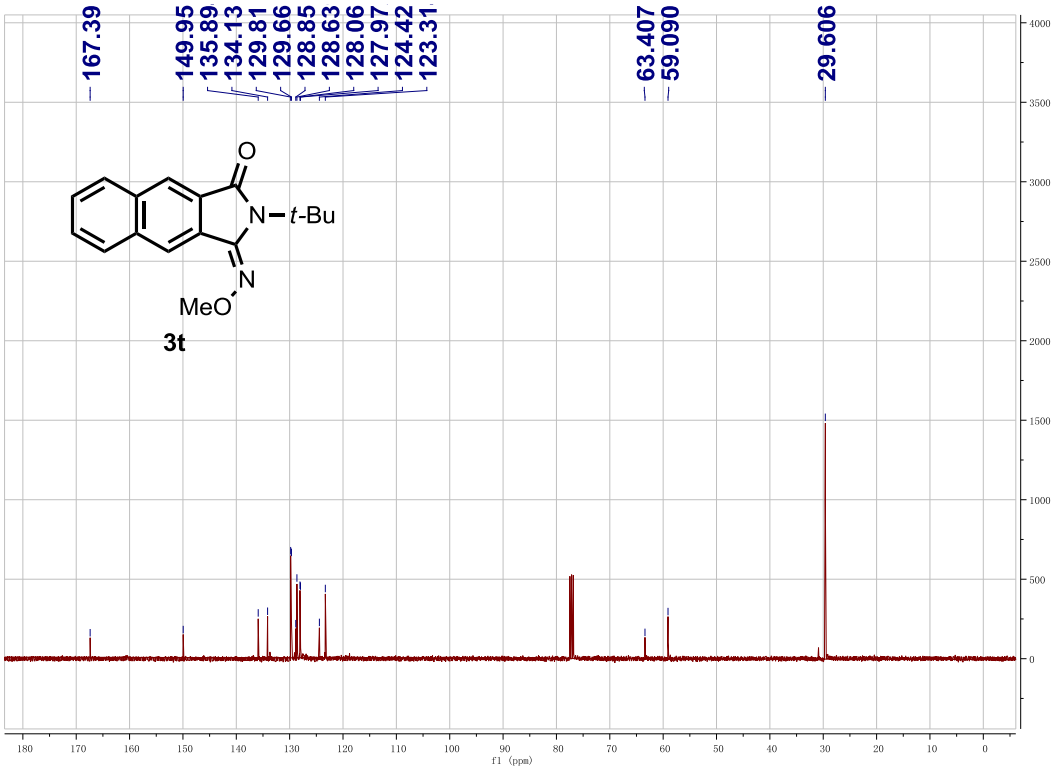

8.478
8.476
8.474
8.473
7.834
7.832
7.814
7.812
7.748
7.744
7.619
7.615
7.598
7.597
7.595
7.481
7.463
4.084
1.773
O
N–t-Bu
Ph
N
MeO
3r
1.00
1.03
1.03
2.01
2.14
1.06
3.14
9.64
f1 (ppm)

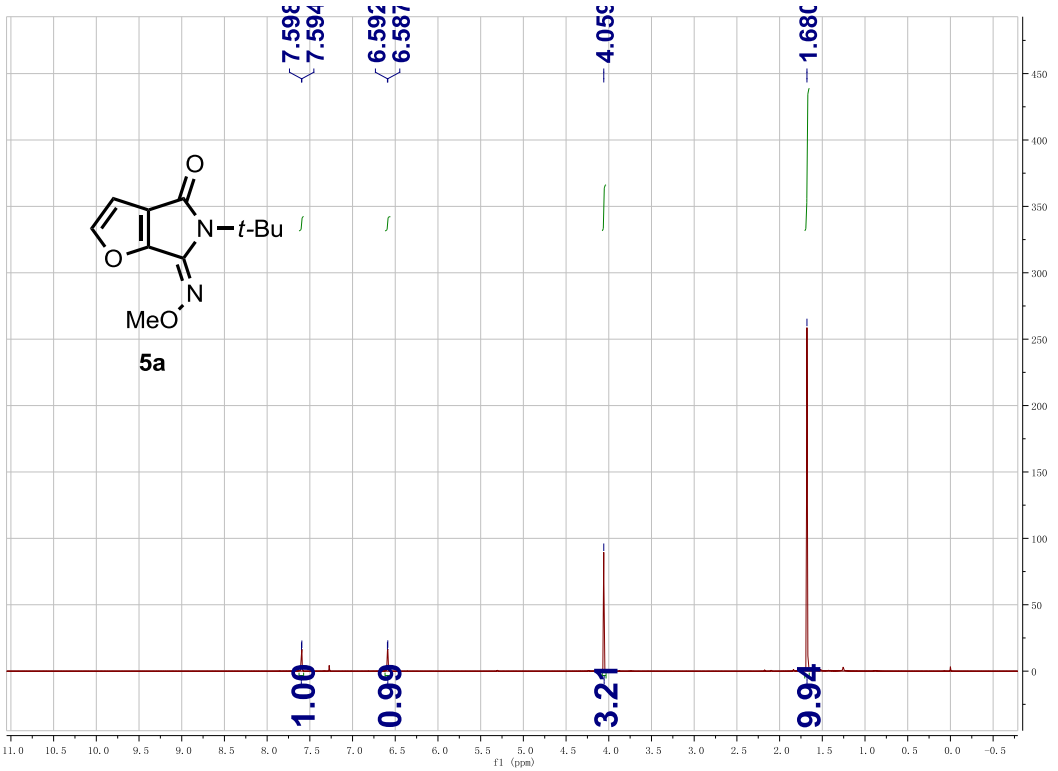

167.36
149.94
146.36
140.51
130.59
130.33
129.29
129.06
128.25
127.68
126.68
123.13
63.490
58.874
29.691
O
N–t-Bu
Ph
N
MeO
3r
f1 (ppm)

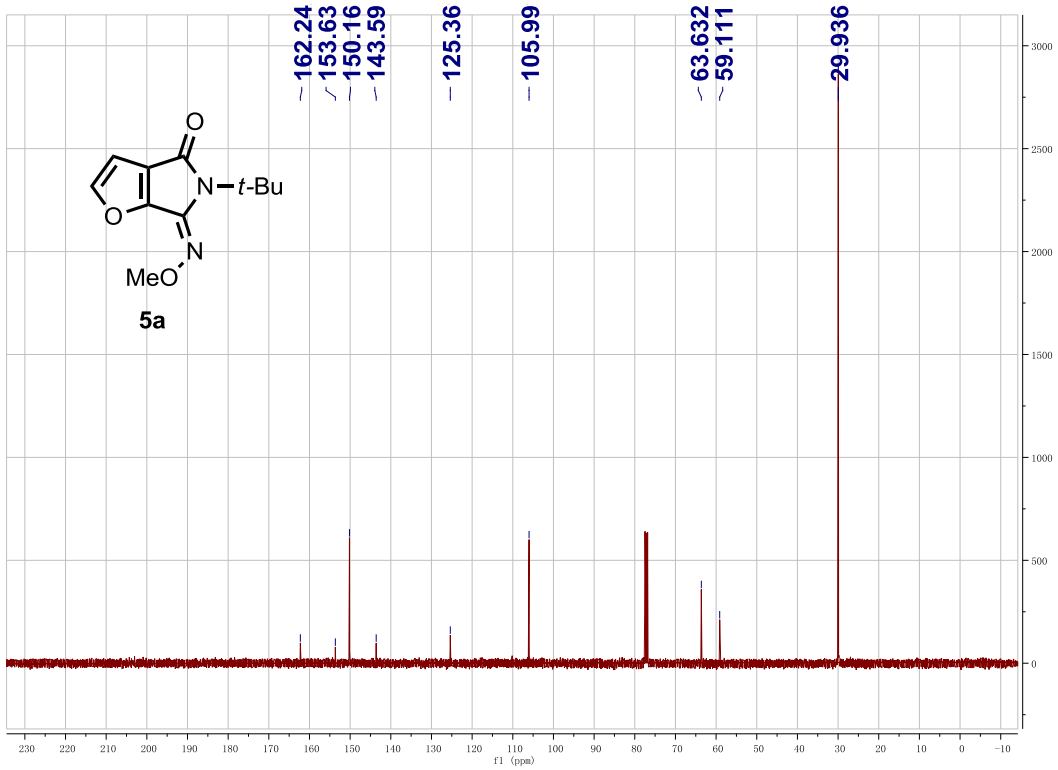

8.015
7.994
7.055
7.038
4.027
4.017
3.918
1.737
OMe
O
MeO
N–t-Bu
N
MeO
3s
0.91
1.00
3.03
2.87
3.02
9.13
f1 (ppm)

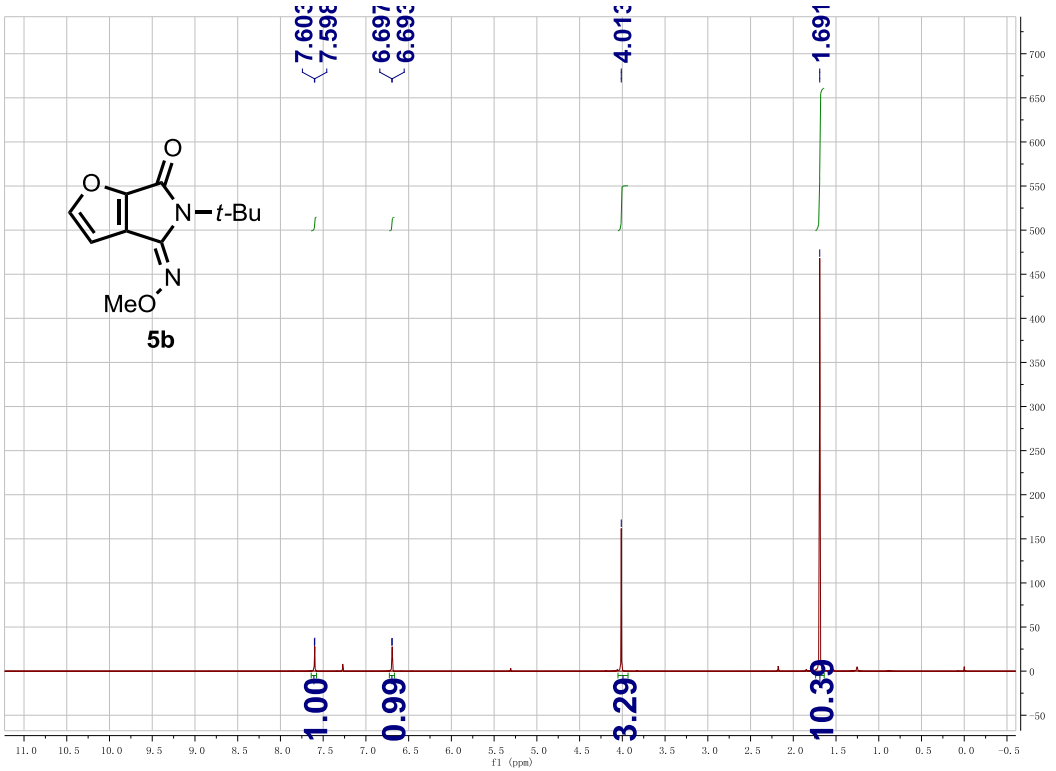

165.50
155.36
149.30
146.43
124.25
123.53
122.16
116.03
63.194
62.241
58.769
56.533
29.706
OMe
O
MeO
N–t-Bu
N
MeO
3s
f1 (ppm)

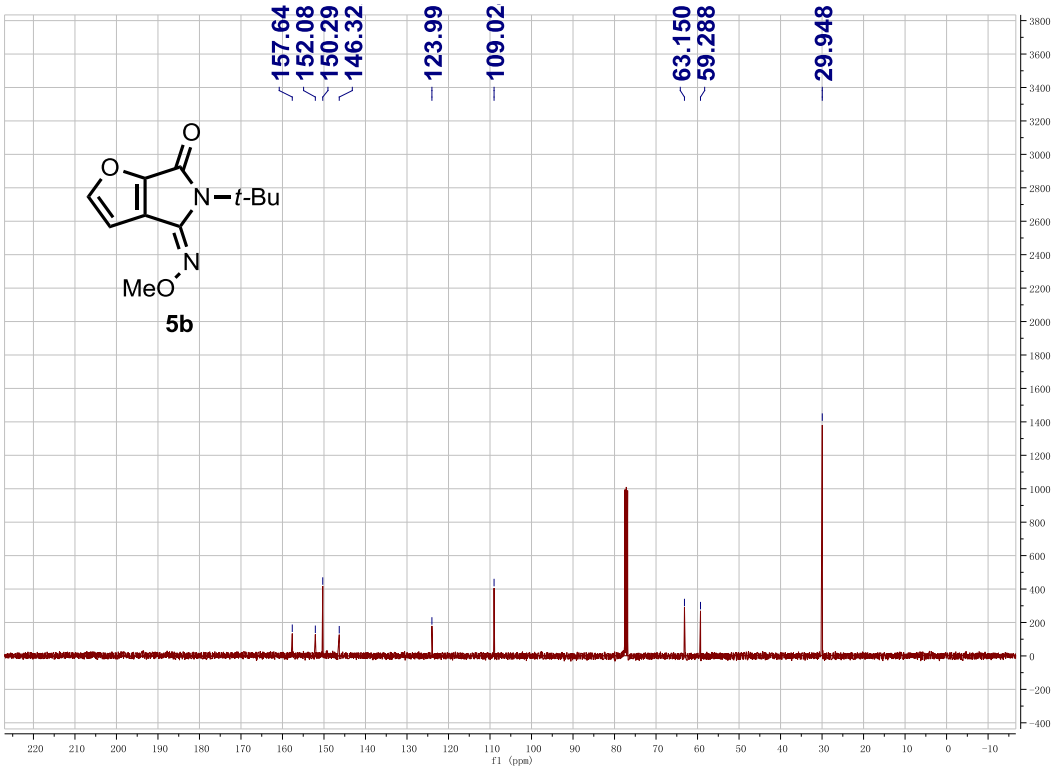

8.763
8.241
7.982
7.973
7.965
7.961
7.600
7.594
7.588
7.581
7.576
7.257
4.134
1.805
O
N–t-Bu
N
MeO
3t
1.00
1.03
2.14
2.19
3.07
9.41
f1 (ppm)

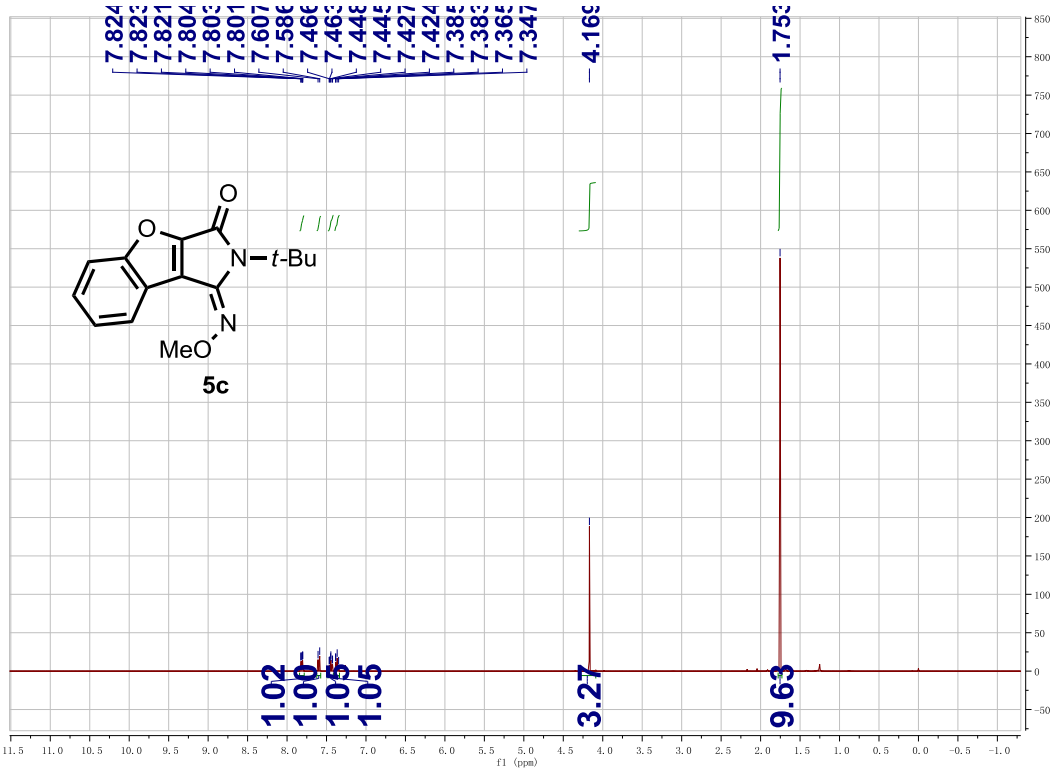

167.39
149.95
135.89
134.13
129.81
129.66
128.85
128.63
128.06
127.97
124.42
123.31
63.407
59.090
29.606
O
N–t-Bu
N
MeO
3t
f1 (ppm)

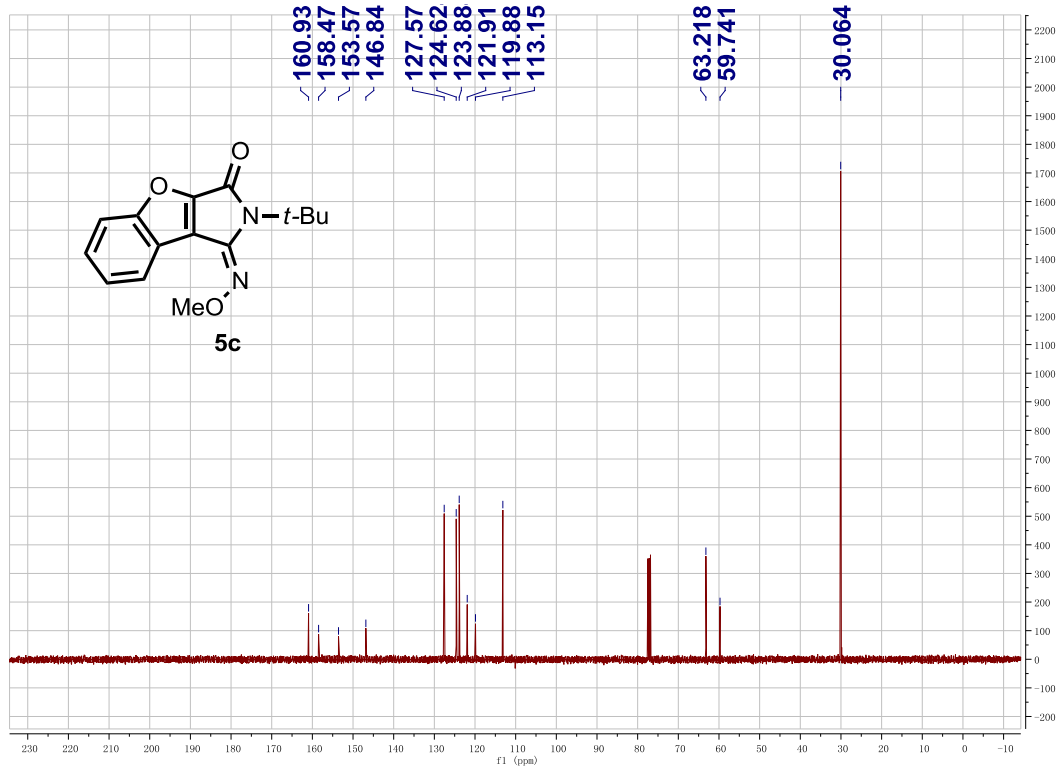

7.598
7.594
6.592
6.587
4.059
1.680
1.00
0.99
3.21
9.94
f1 (ppm)
O
N–t-Bu
O
N
MeO
5a

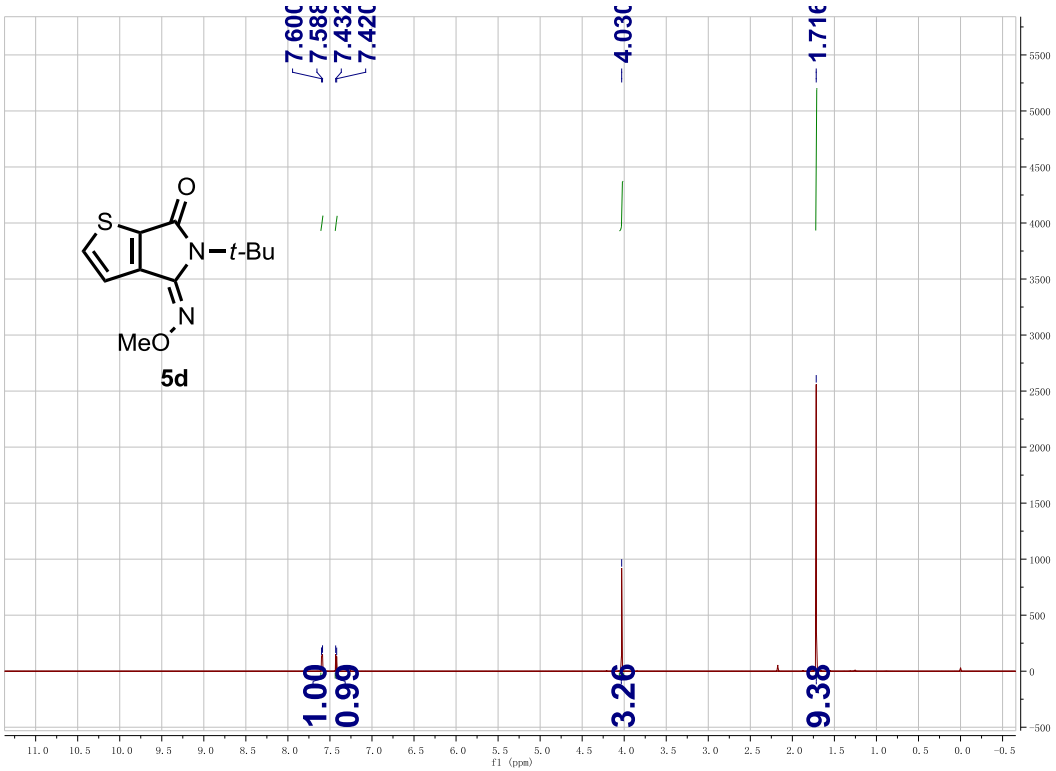

162.24
153.63
150.16
143.59
125.36
105.99
63.632
59.111
29.936
f1 (ppm)
O
N–t-Bu
O
N
MeO
5a

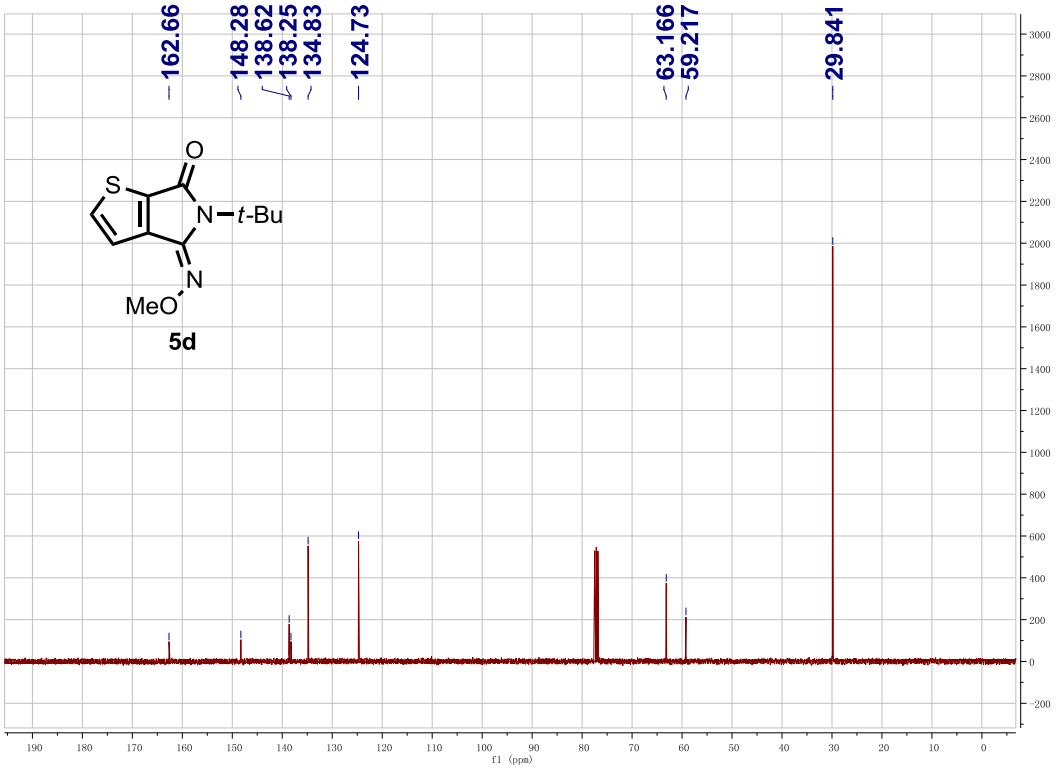

7.603
7.598
6.697
6.693
4.013
1.691
1.00
0.99
3.29
10.39
f1 (ppm)
5b

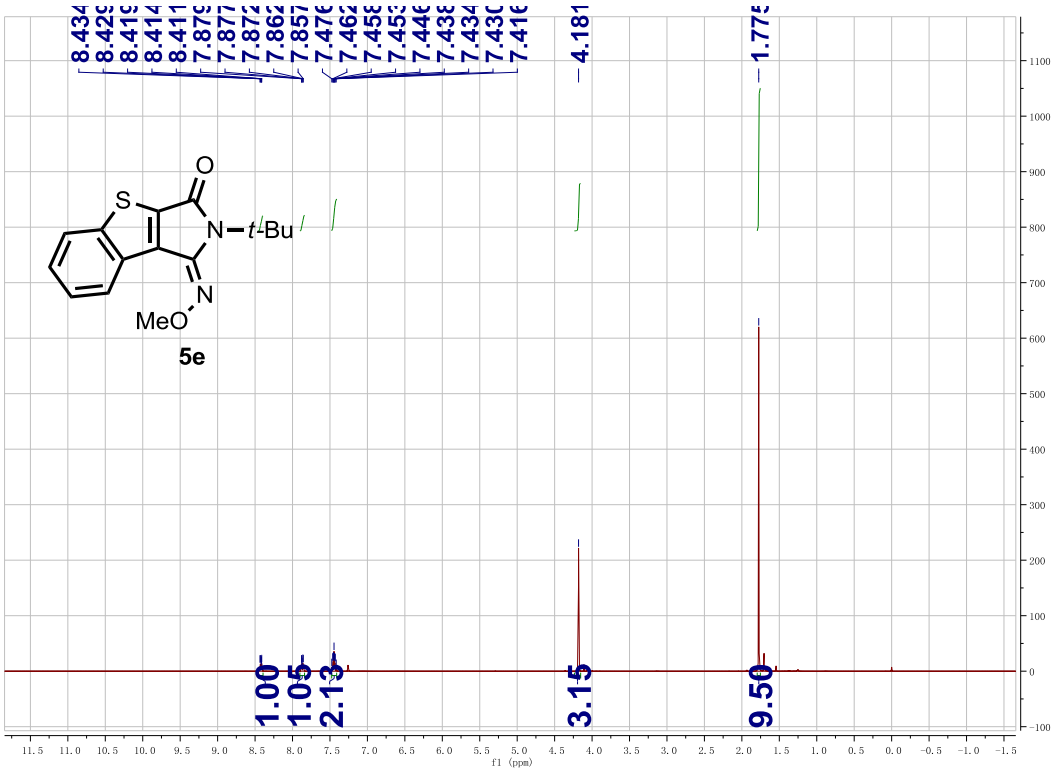

157.64
152.08
150.29
146.32
123.99
109.02
63.150
59.288
29.948
f1 (ppm)
5b

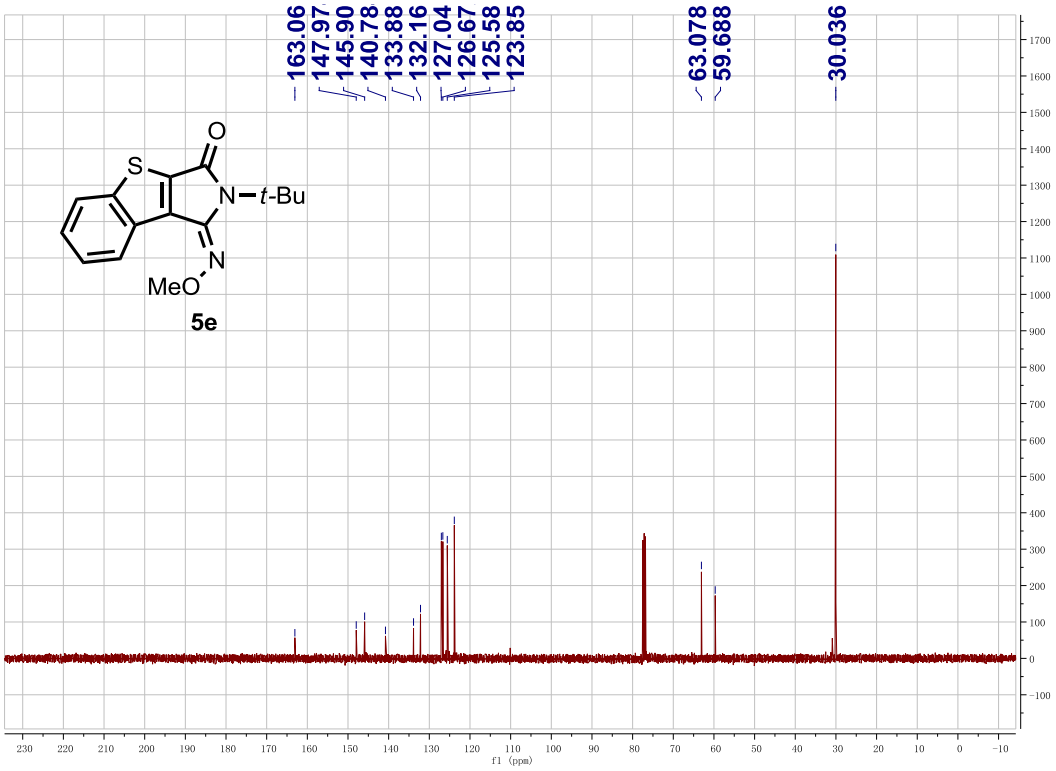

7.824
7.823
7.821
7.804
7.803
7.801
7.607
7.586
7.466
7.463
7.448
7.445
7.427
7.424
7.385
7.383
7.365
7.347
4.165
1.753
O
O
N–t-Bu
N
MeO
5c
1.02
1.00
1.05
1.05
3.27
9.63
f1 (ppm)

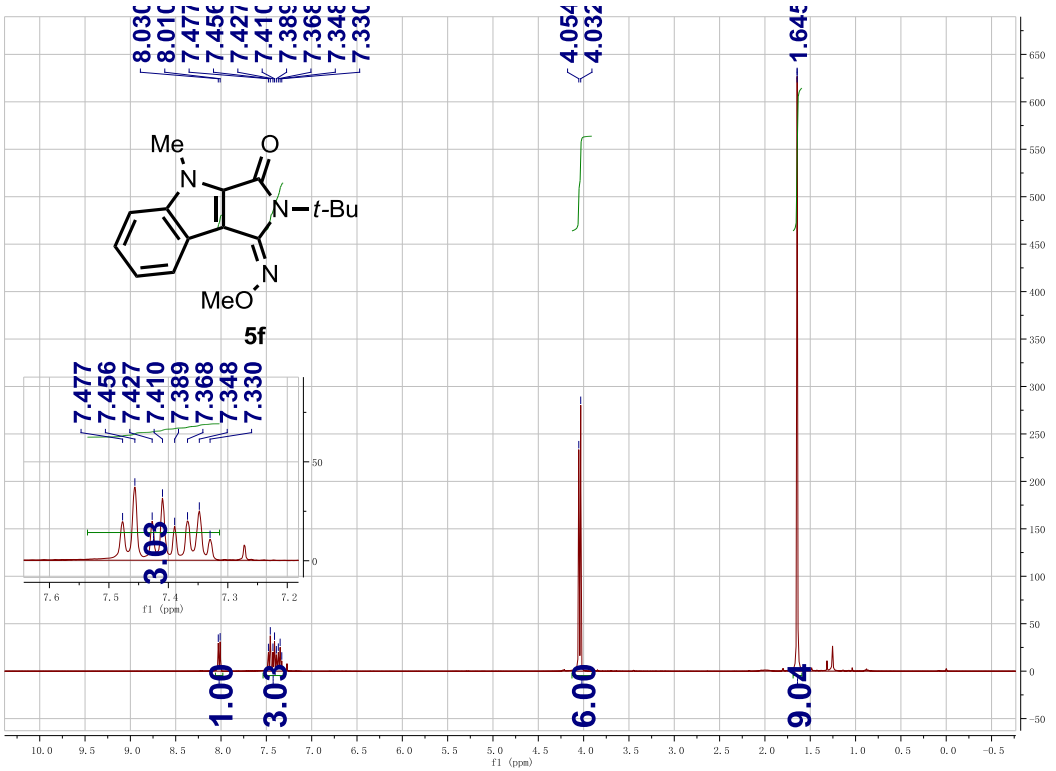

160.93
158.47
153.57
146.84
127.57
124.62
123.88
121.91
119.88
113.15
63.218
59.741
30.064
O
O
N–t-Bu
N
MeO
5c
f1 (ppm)

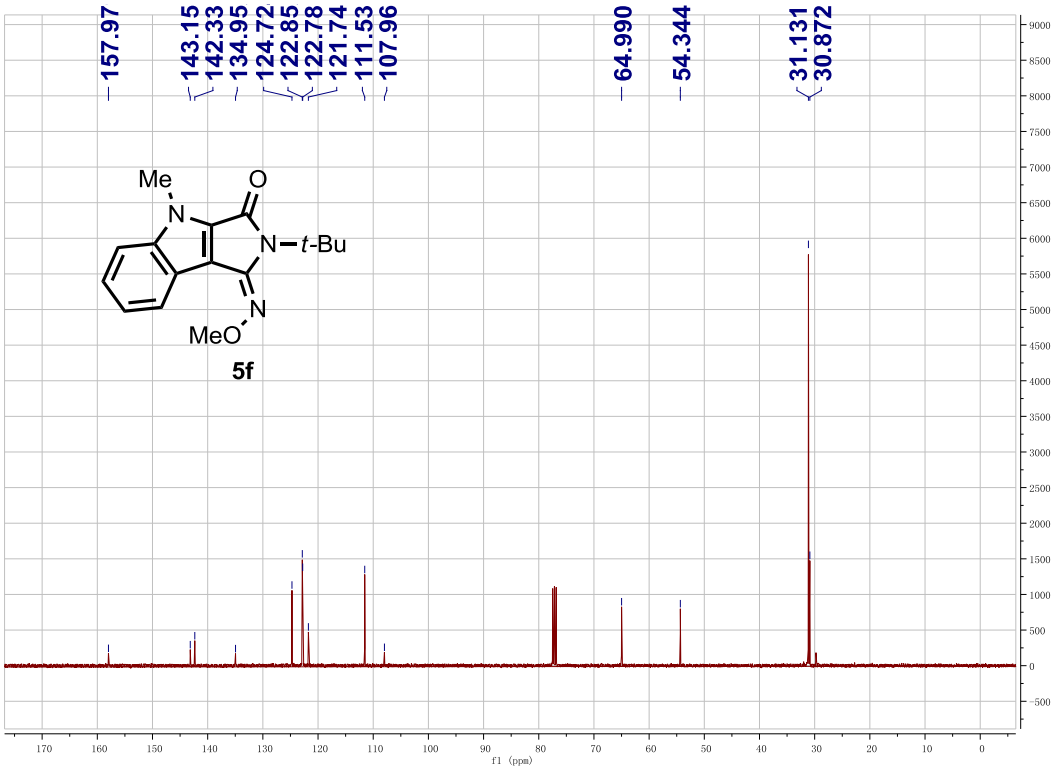

7.600
7.588
7.432
7.420
4.030
1.716
O
S
N–t-Bu
N
MeO
5d
1.00
0.99
3.26
9.38
f1 (ppm)

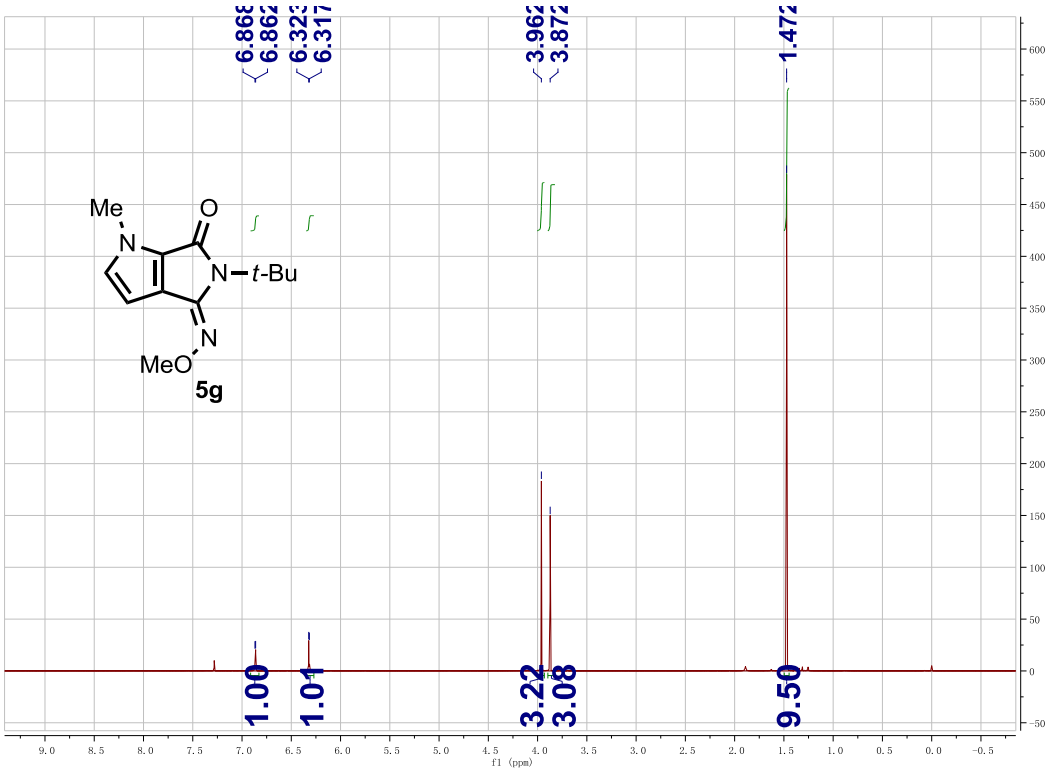

162.66
148.28
138.62
138.25
134.83
124.73
63.166
59.217
29.841
O
S
N–t-Bu
N
MeO
5d
f1 (ppm)

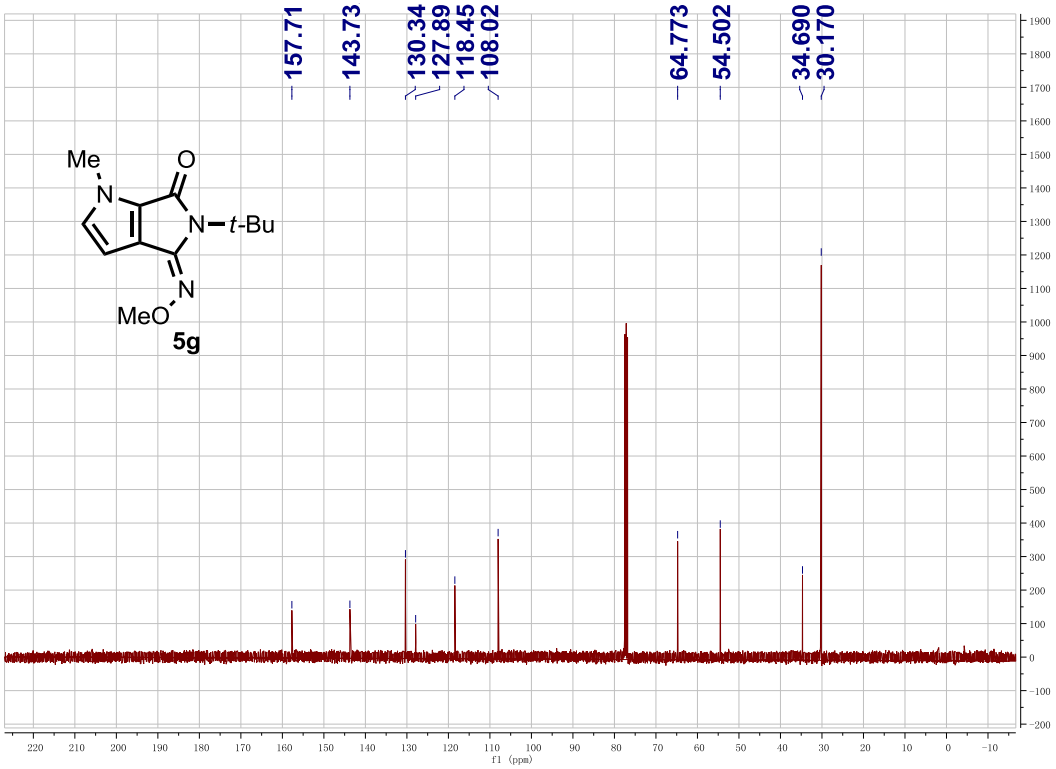

8.434
8.423
8.419
8.414
8.411
7.879
7.877
7.872
7.862
7.857
7.476
7.462
7.458
7.453
7.446
7.438
7.434
7.430
7.416
4.181
1.775
O
S
N–t-Bu
N
MeO
5e
1.00
1.05
2.13
3.15
9.50
f1 (ppm)

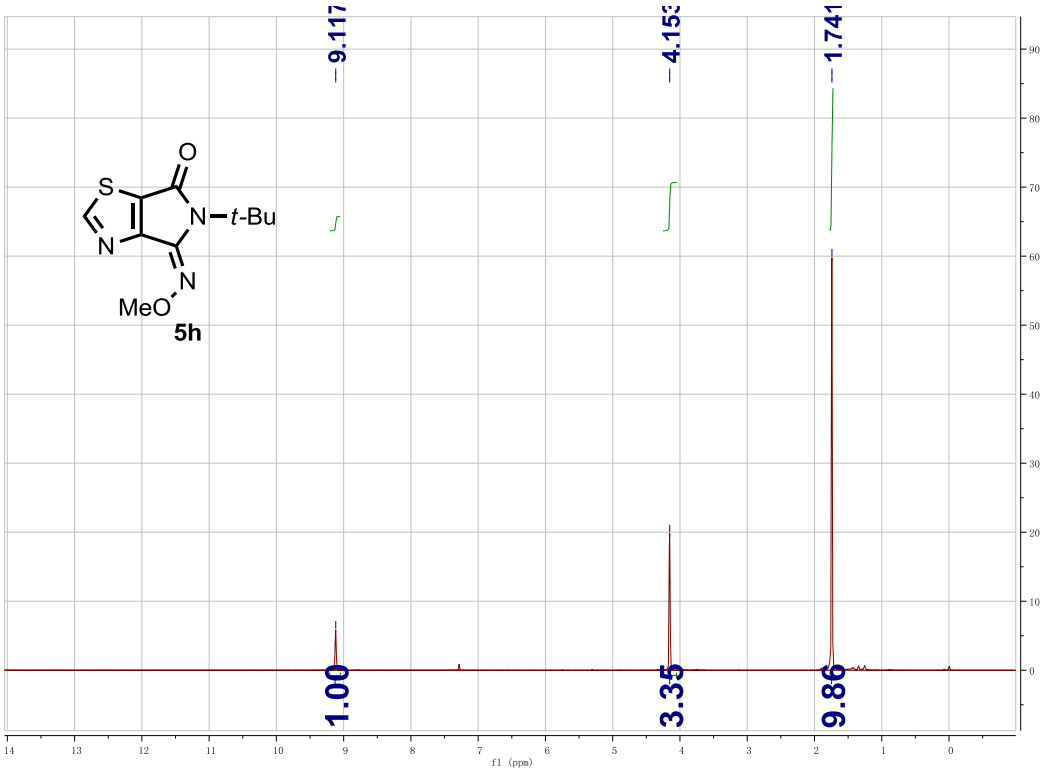

163.06
147.97
145.90
140.78
133.88
132.16
127.04
126.67
125.58
123.85
63.078
59.688
30.036
O
S
N–t-Bu
N
MeO
5e
f1 (ppm)

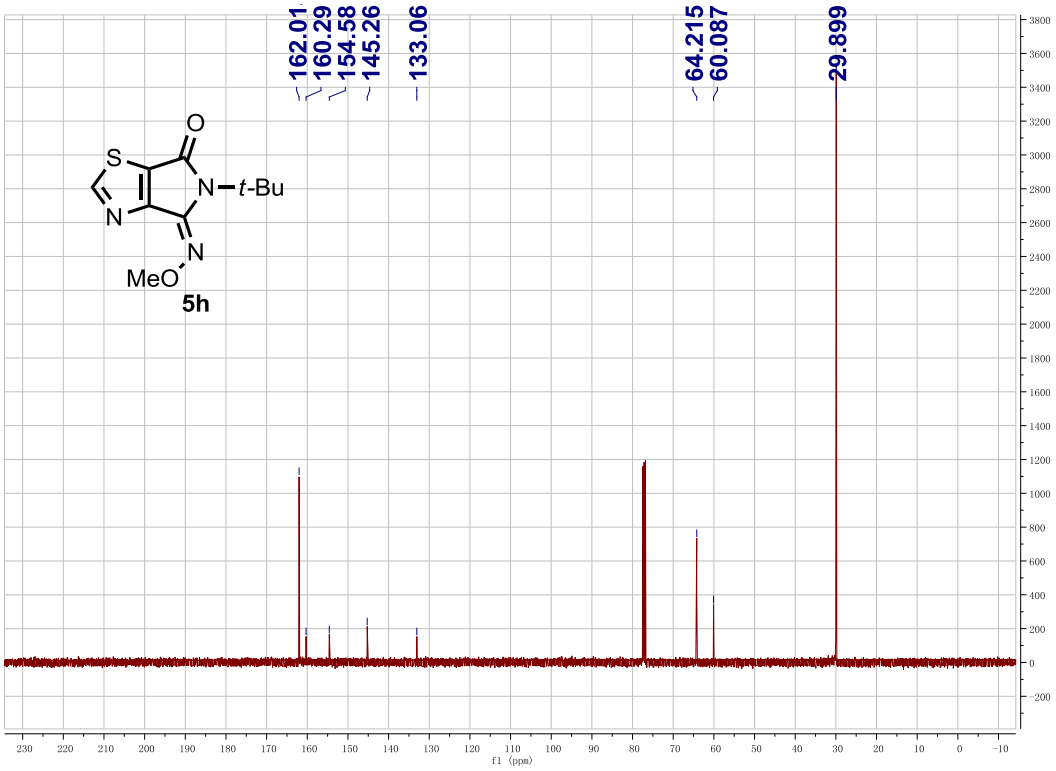

8.030
8.010
7.477
7.456
7.427
7.410
7.389
7.368
7.348
7.330
4.054
4.032
1.645
Me
O
N
N−t-Bu
N
MeO
5f
3.03
1.00
3.03
6.00
9.04
f1 (ppm)

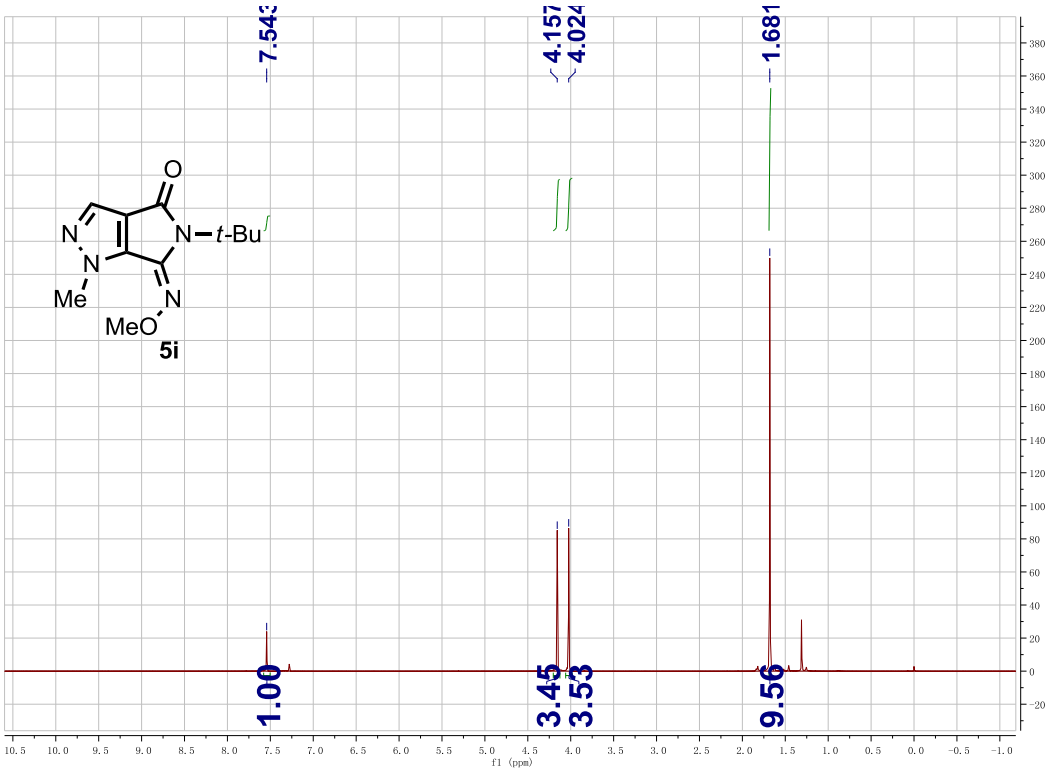

157.97
143.15
142.33
134.95
124.72
122.85
122.78
121.74
111.53
107.96
64.990
54.344
31.131
30.872
Me
O
N
N−t-Bu
N
MeO
5f
f1 (ppm)

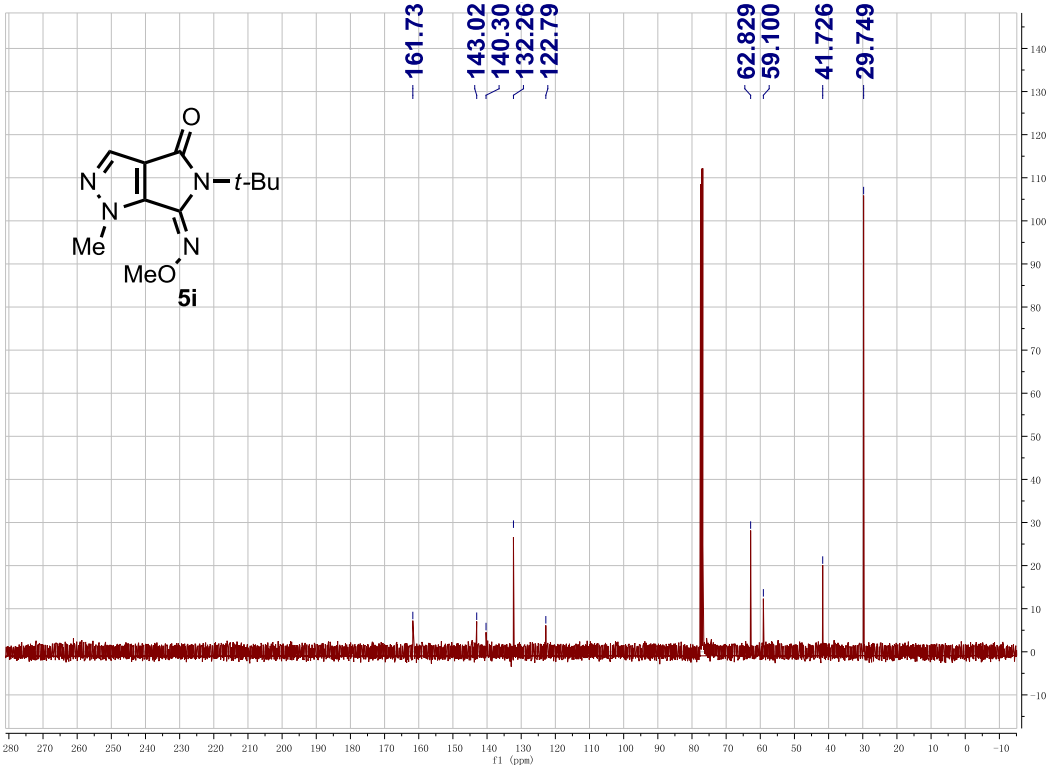

6.868
6.862
6.323
6.317
3.962
3.872
1.472
Me
O
N
N–t-Bu
N
MeO
5g
1.00
1.01
3.22
3.08
9.50
f1 (ppm)

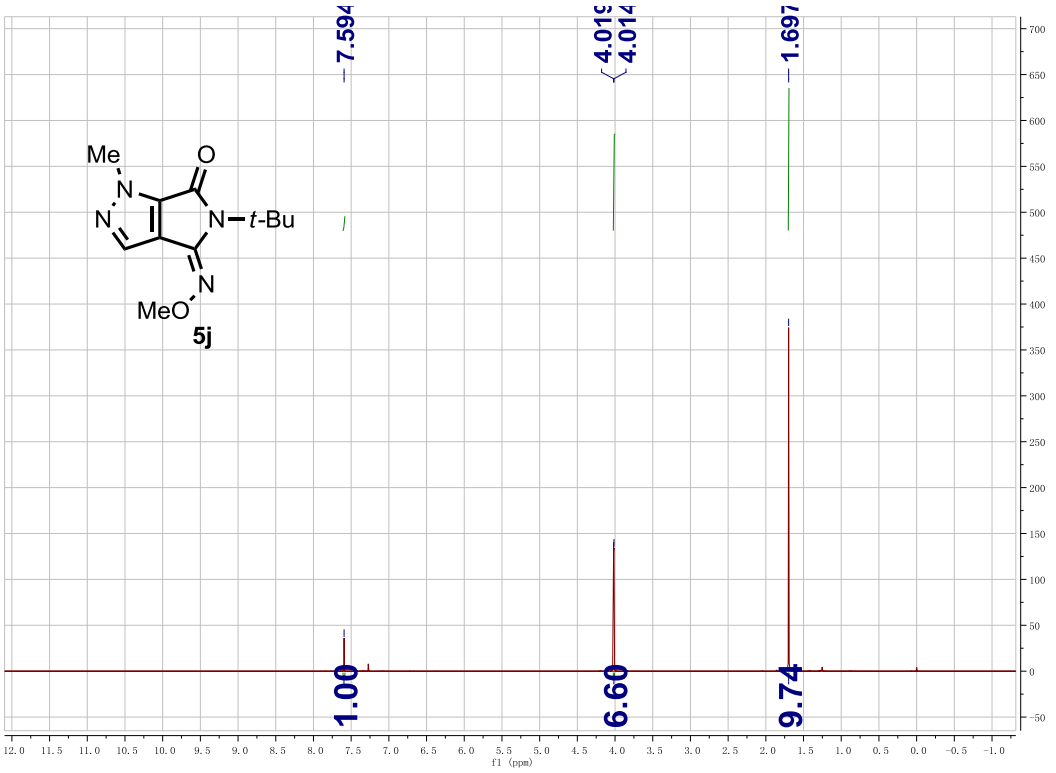

157.71
143.73
130.34
127.89
118.45
108.02
64.773
54.502
34.690
30.170
Me
O
N
N–t-Bu
N
MeO
5g
f1 (ppm)

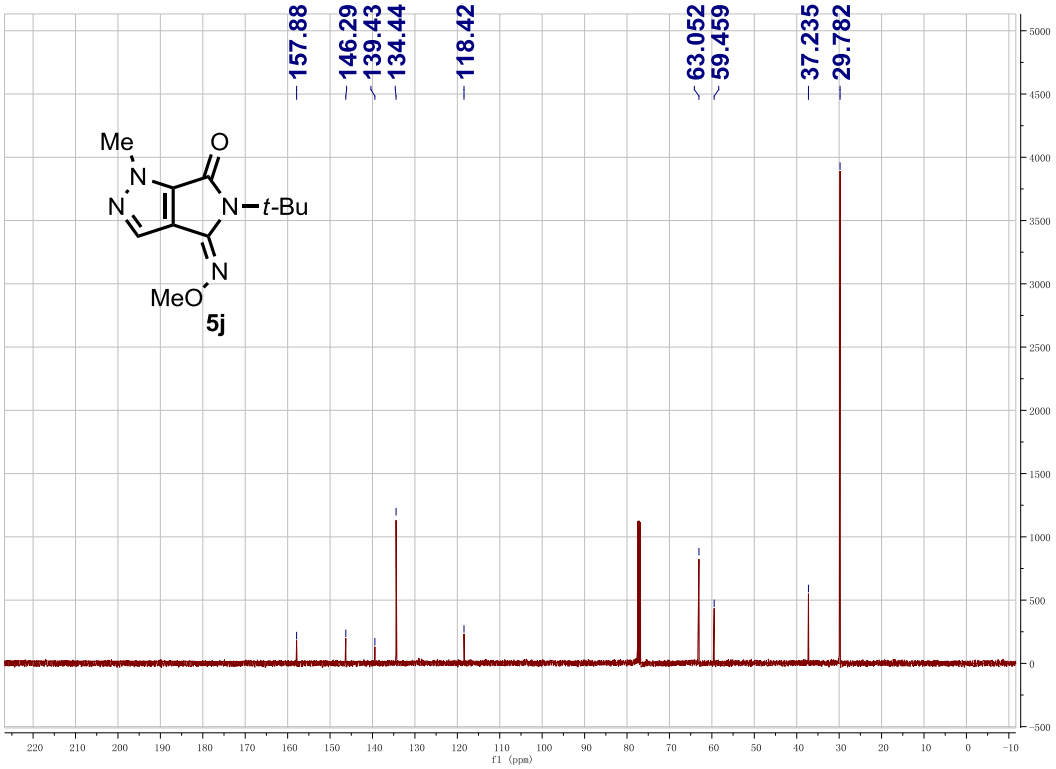

9.117
4.153
1.741
1.00
3.35
9.86
f1 (ppm)
5h

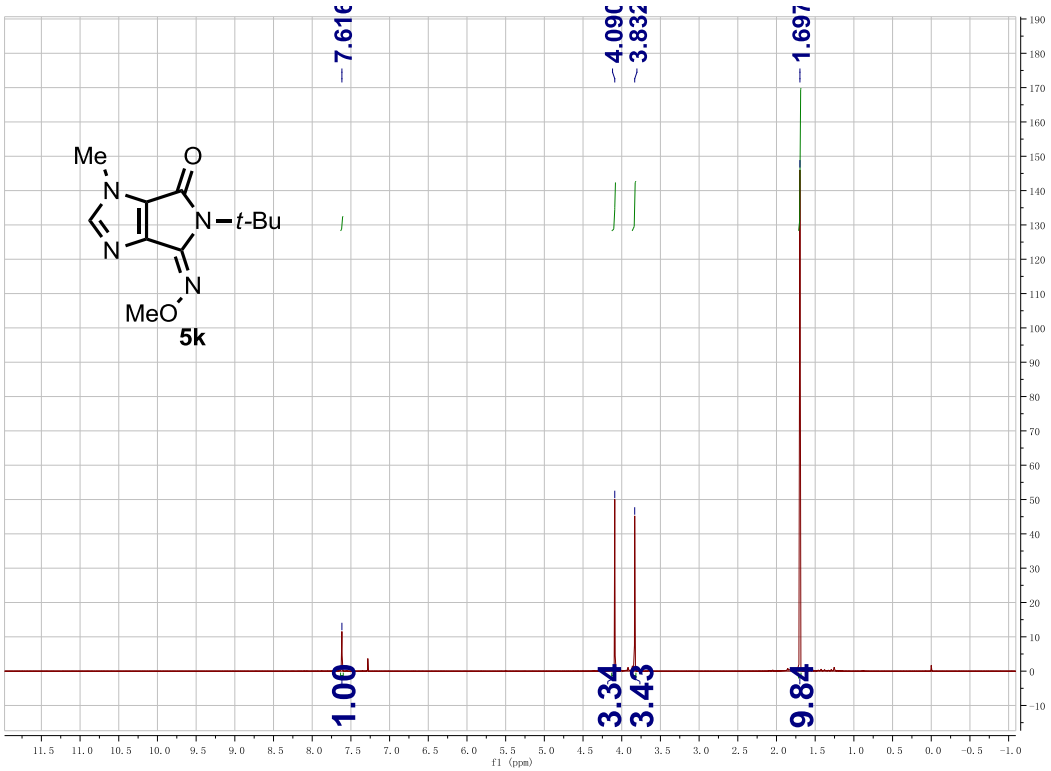

162.01
160.29
154.58
145.26
133.06
64.215
60.087
29.899
f1 (ppm)
5h

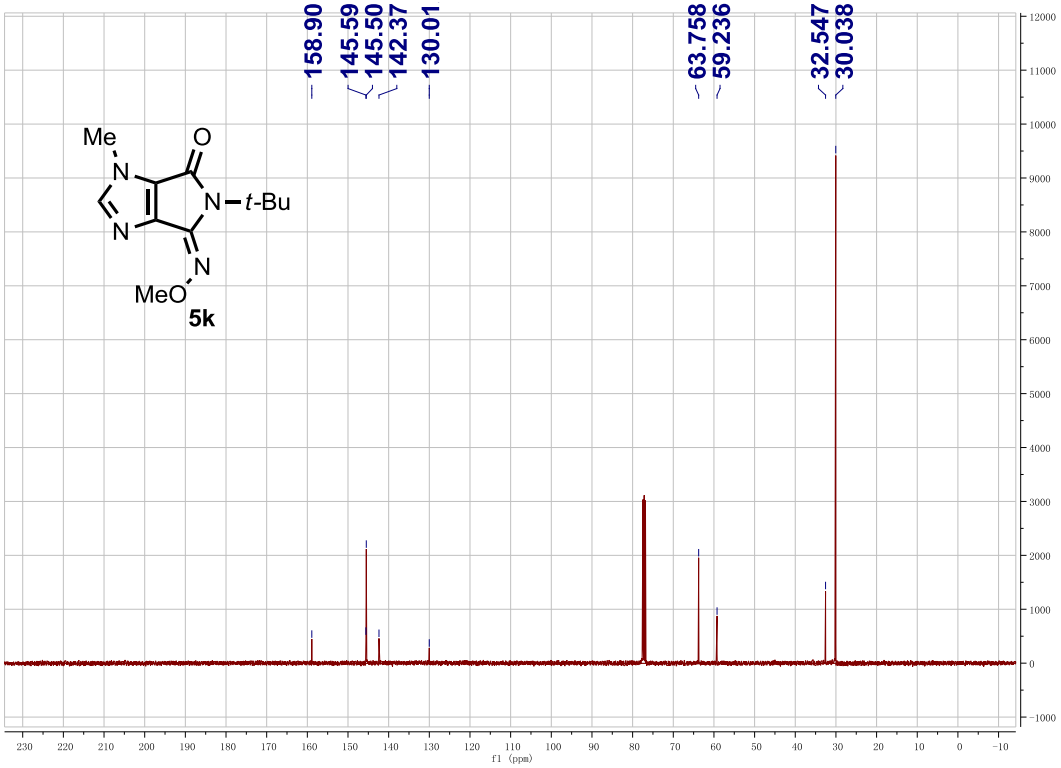

7.543
4.157
4.024
1.681
1.00
3.45
3.53
9.56
f1 (ppm)
5i

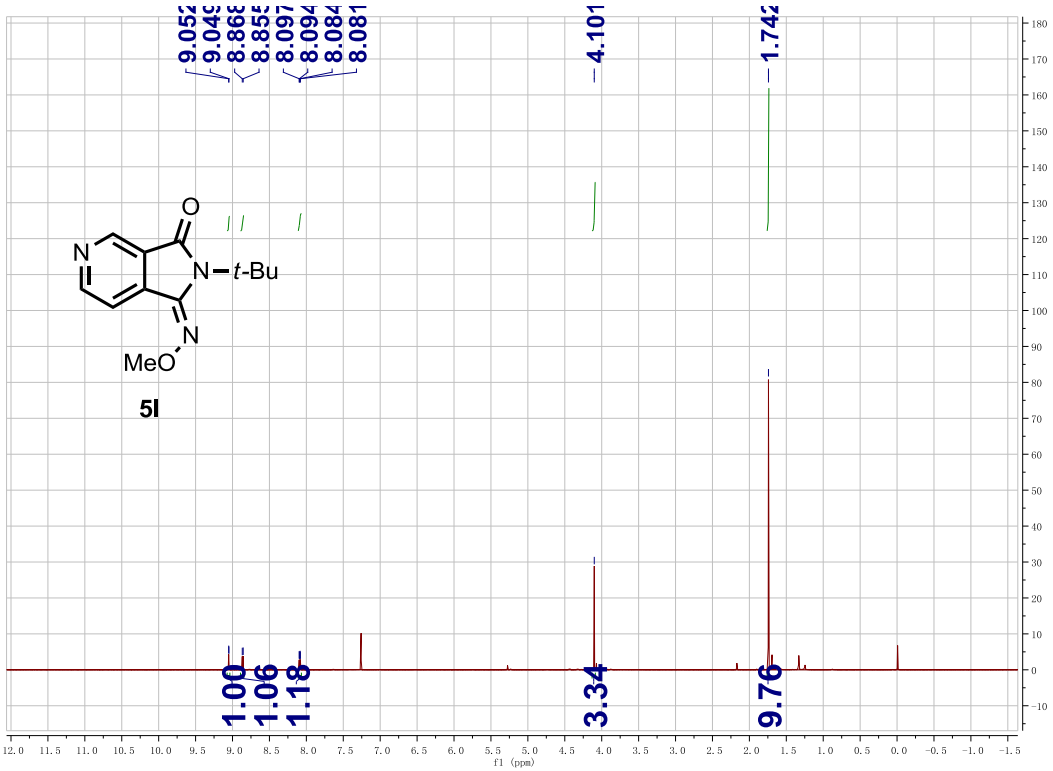

161.73
143.02
140.30
132.26
122.79
62.829
59.100
41.726
29.749
f1 (ppm)
5i

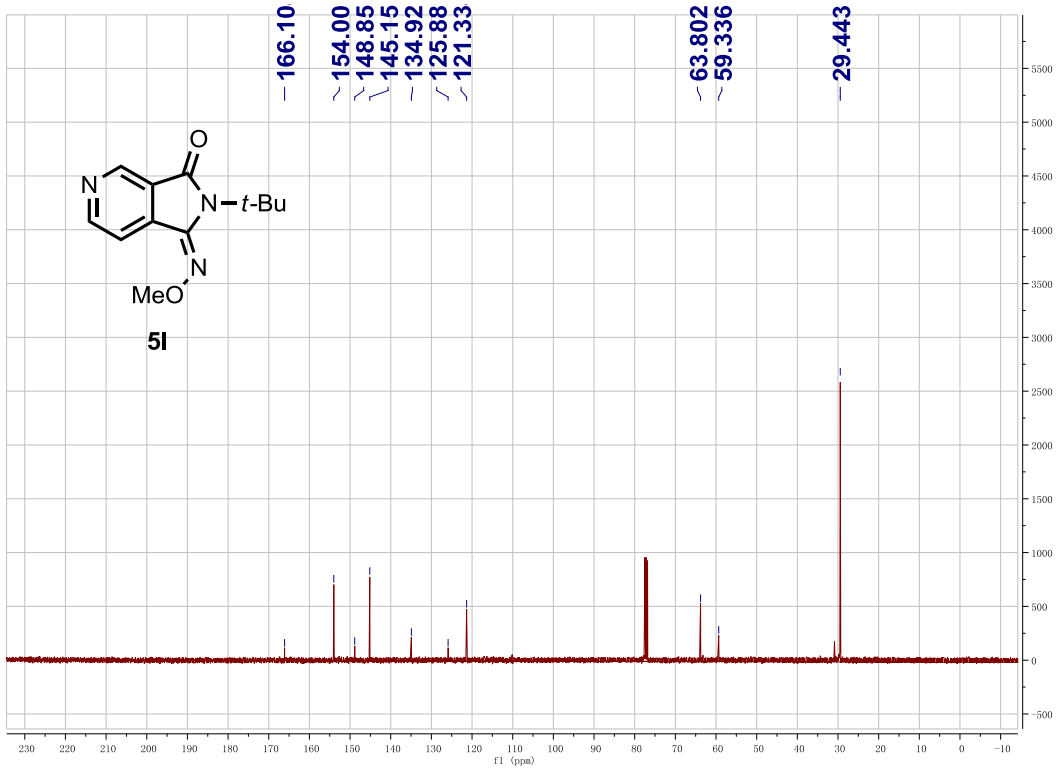

7.594
4.019
4.014
1.697
Me
O
N
N
N
t-Bu
N
MeO
5j
1.00
6.60
9.74
f1 (ppm)

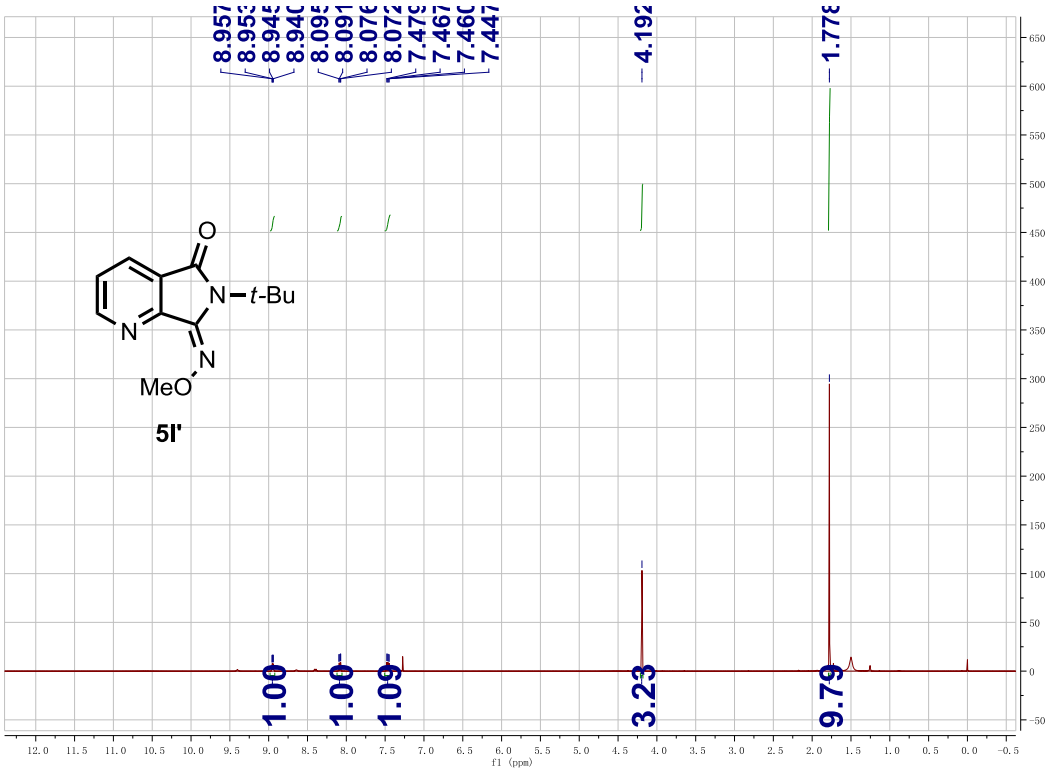

157.88
146.29
139.43
134.44
118.42
63.052
59.459
37.235
29.782
Me
O
N
N
N
t-Bu
N
MeO
5j
f1 (ppm)

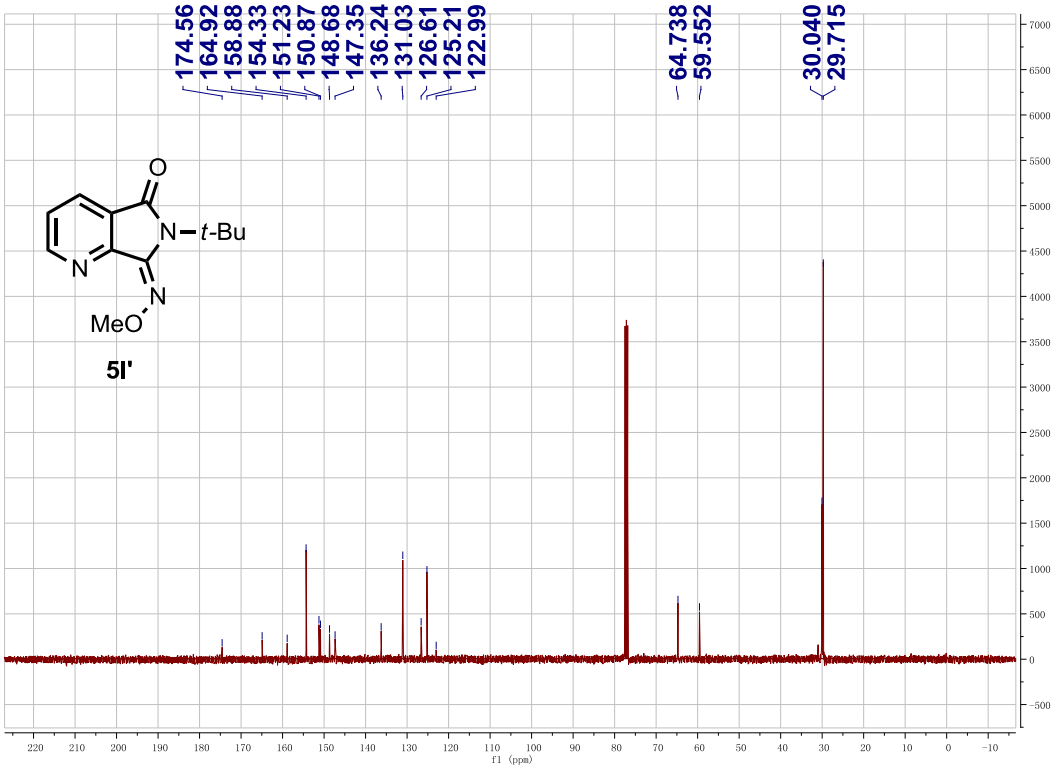

7.61
4.09
3.83
1.69
Me
O
N
N
N
N
t-Bu
MeO
5k
1.00
3.34
3.43
9.84
f1 (ppm)

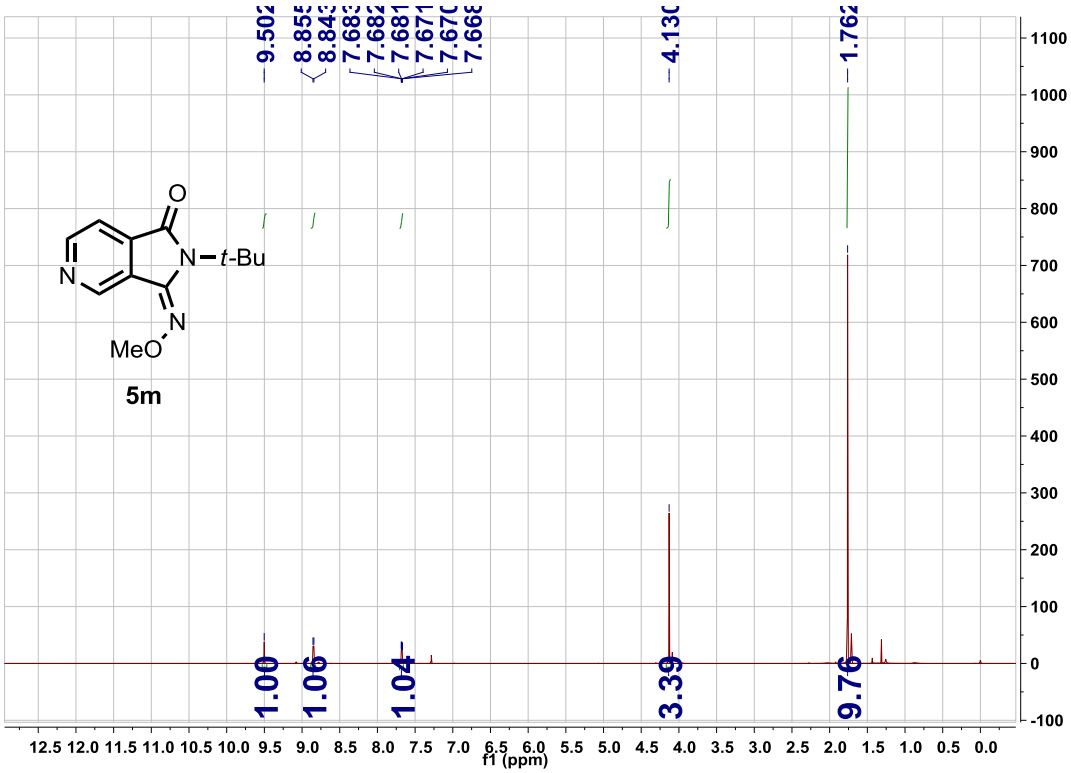

158.90
145.59
145.50
142.37
130.01
63.758
59.236
32.547
30.038
Me
O
N
N
N
N
t-Bu
MeO
5k
f1 (ppm)

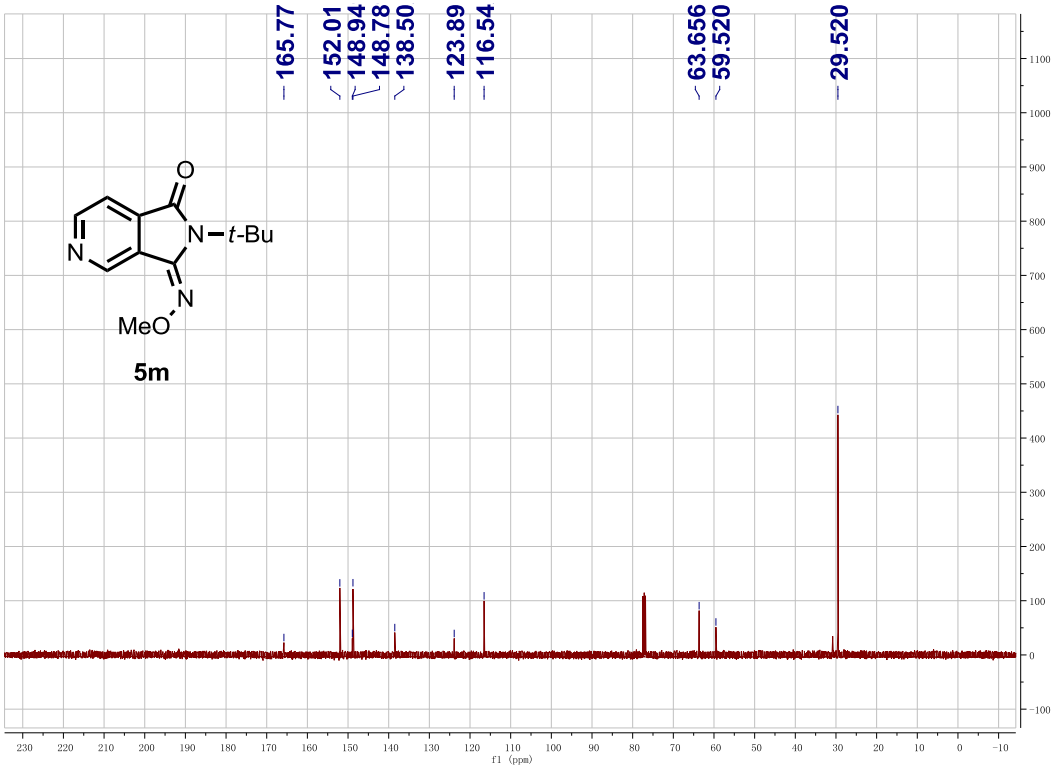

9.052
9.045
8.868
8.855
8.097
8.094
8.084
8.081
4.101
1.742
1.00
1.06
1.18
3.34
9.76
N
O
N–t-Bu
N
MeO
5l
f1 (ppm)

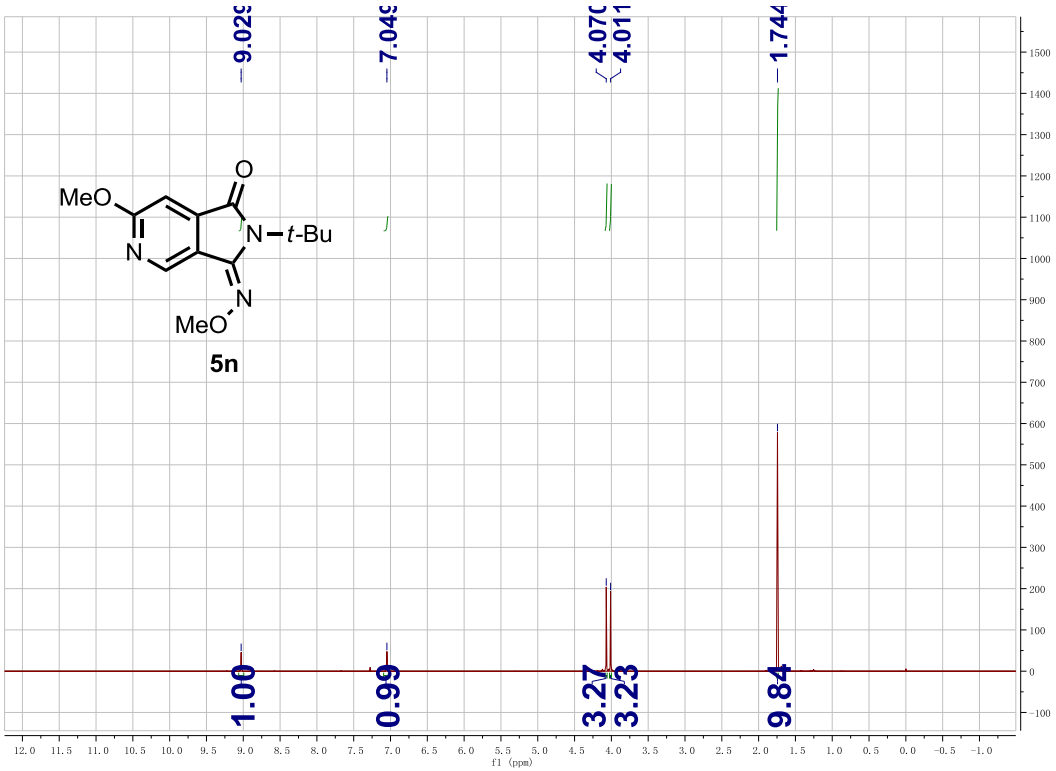

166.10
154.00
148.85
145.15
134.92
125.88
121.33
63.802
59.336
29.443
N
O
N–t-Bu
N
MeO
5l
f1 (ppm)

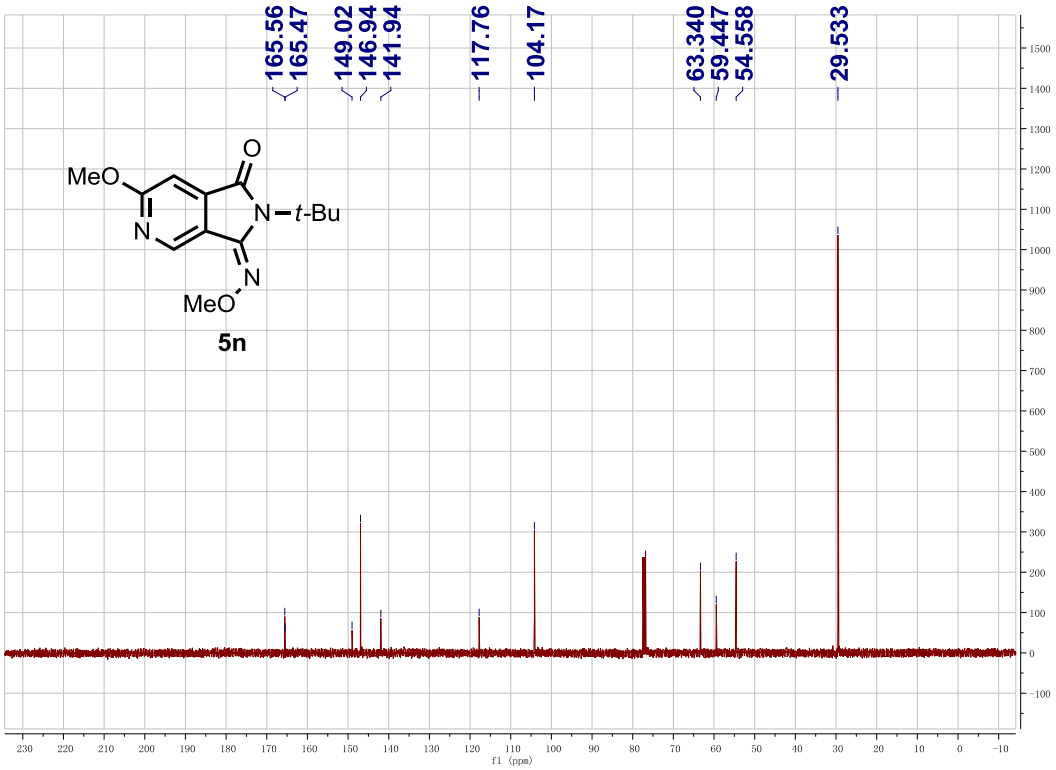

8.957
8.953
8.945
8.940
8.095
8.091
8.076
8.072
7.475
7.467
7.460
7.447
4.192
1.778
O
N–t-Bu
N
N
MeO
5l'
1.00
1.00
1.09
3.23
9.79
f1 (ppm)

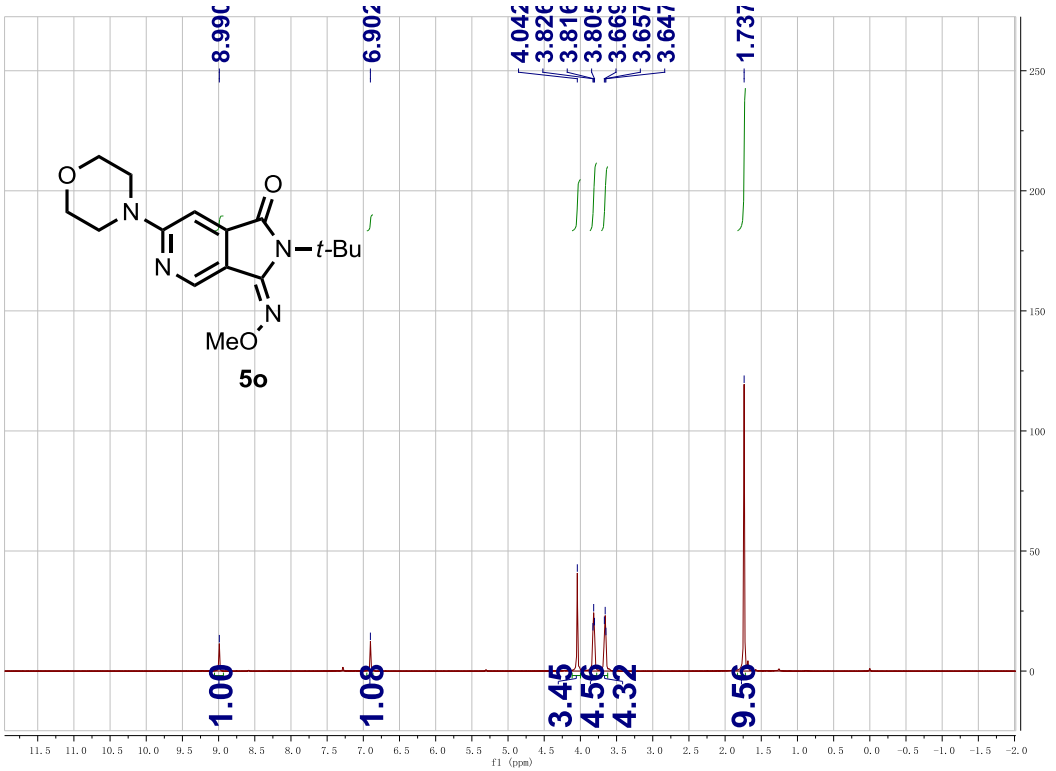

174.56
164.92
158.88
154.33
151.23
150.87
148.68
147.35
136.24
131.03
126.61
125.21
122.99
64.738
59.552
30.040
29.715
O
N–t-Bu
N
N
MeO
5l'
f1 (ppm)

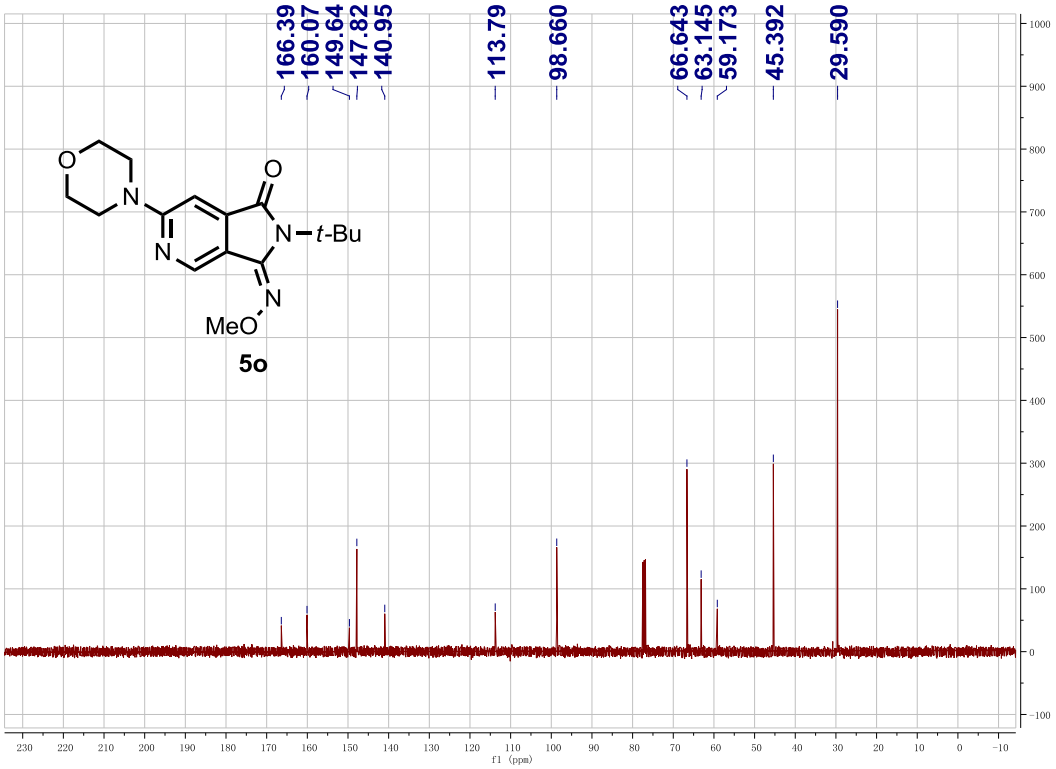

9.502
8.855
8.843
7.683
7.682
7.681
7.671
7.670
7.668
4.130
1.762
1.00
1.06
1.04
3.39
9.76
f1 (ppm)
N
O
N–t-Bu
N
MeO
5m

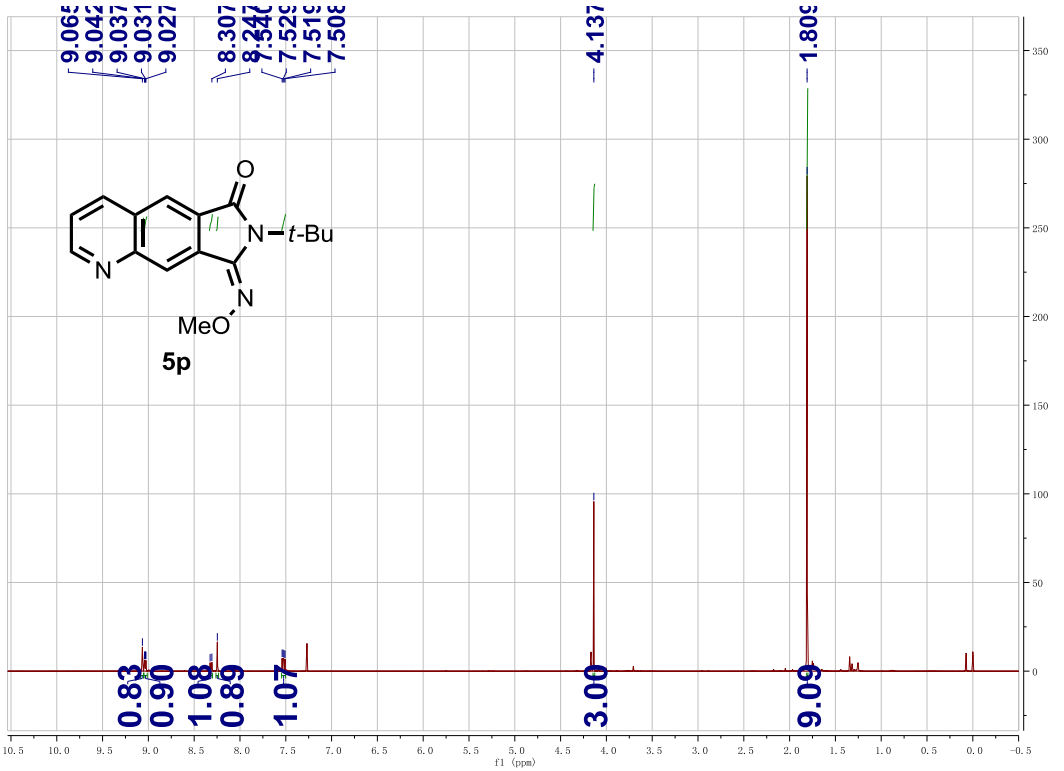

165.77
152.01
148.94
148.78
138.50
123.89
116.54
63.656
59.520
29.520
f1 (ppm)
N
O
N–t-Bu
N
MeO
5m


9.025
7.045
4.070
4.011
1.744
MeO
O
N
N–t-Bu
N
MeO
5n
1.00
0.99
3.27
3.23
9.84
f1 (ppm)


165.56
165.47
149.02
146.94
141.94
117.76
104.17
63.340
59.447
54.558
29.533
MeO
O
N
N–t-Bu
N
MeO
5n
f1 (ppm)


8.990
6.902
4.042
3.826
3.816
3.805
3.669
3.657
3.647
1.737
O
N
O
N
t-Bu
N
MeO
5o
1.00
1.08
3.45
4.56
4.32
9.56
f1 (ppm)


166.39
160.07
149.64
147.82
140.95
113.79
98.660
66.643
63.145
59.173
45.392
29.590
O
N
O
N
t-Bu
N
MeO
5o
f1 (ppm)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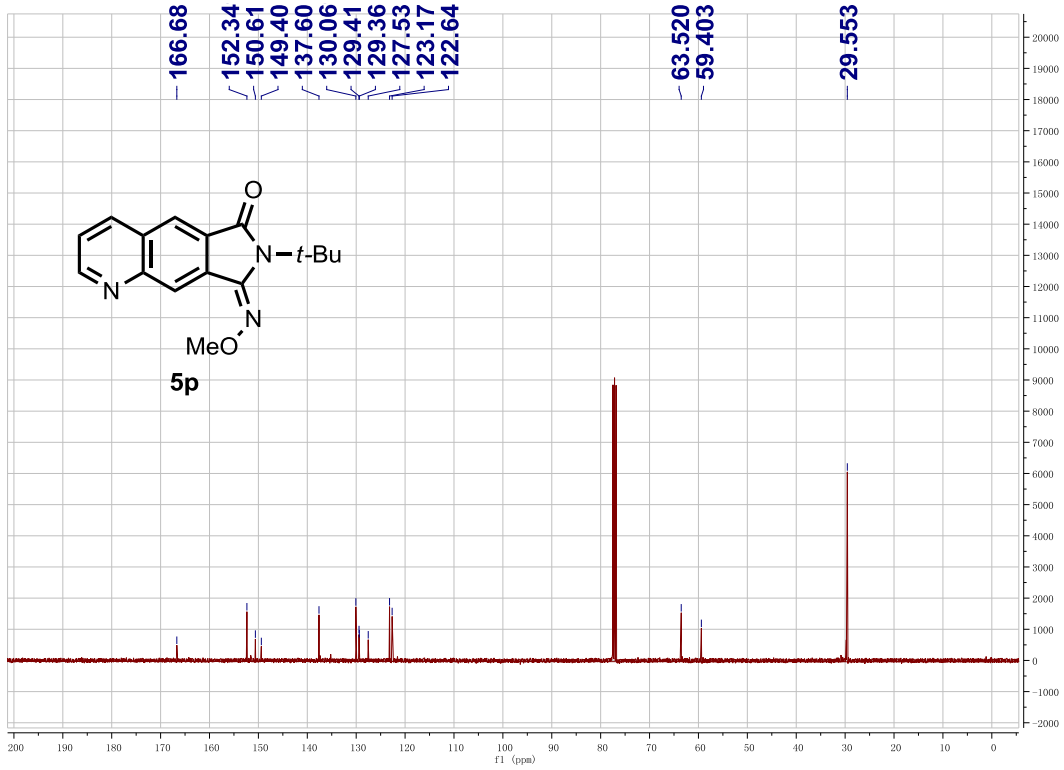
9.042
9.037
9.031
9.027
8.307
7.525
7.519
7.508
4.137
1.809
O
N
N
N–t-Bu
MeO
5p
0.83
0.90
1.08
0.89
1.07
3.00
9.09
f1 (ppm)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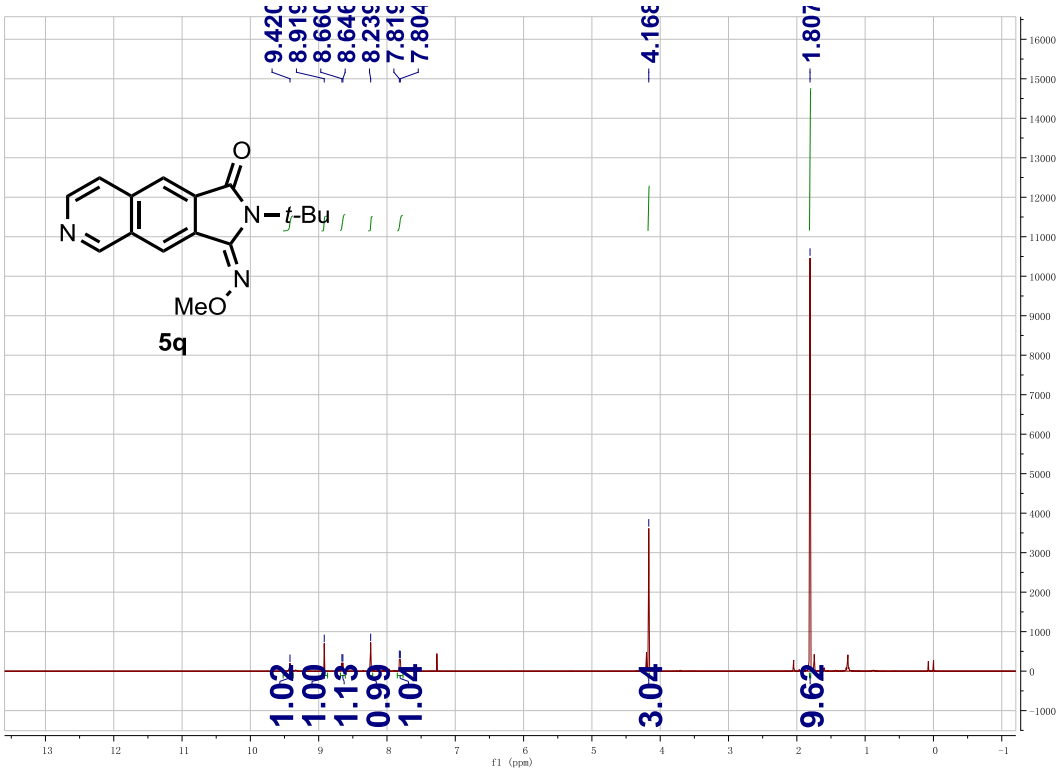
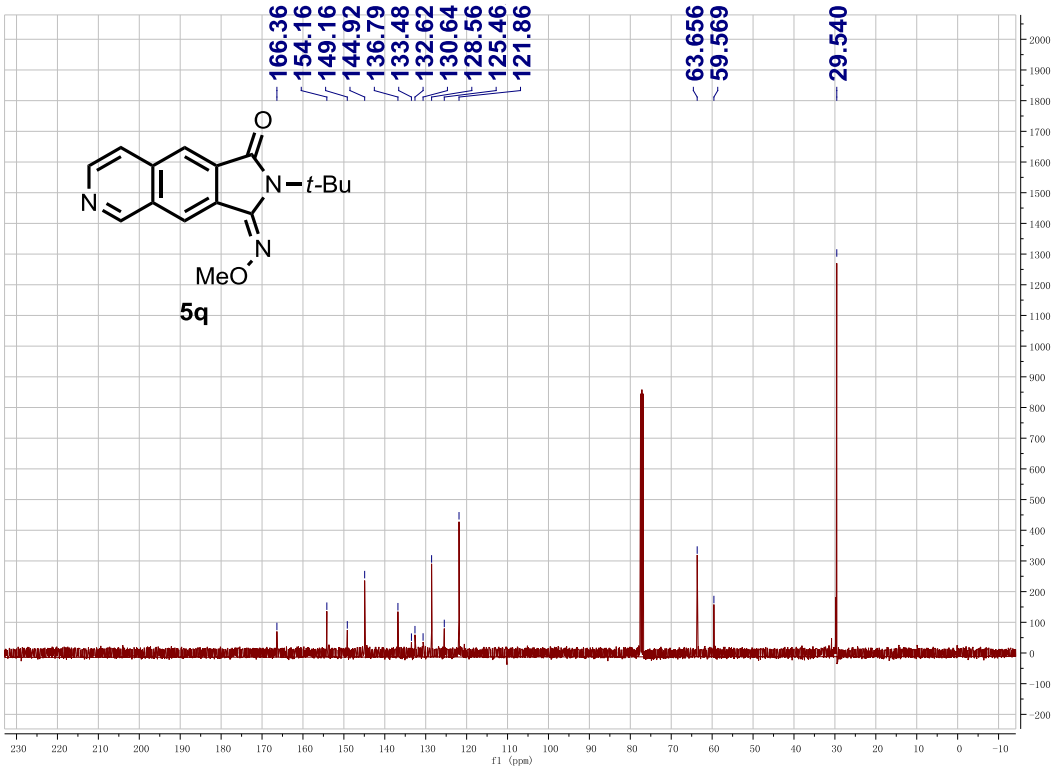
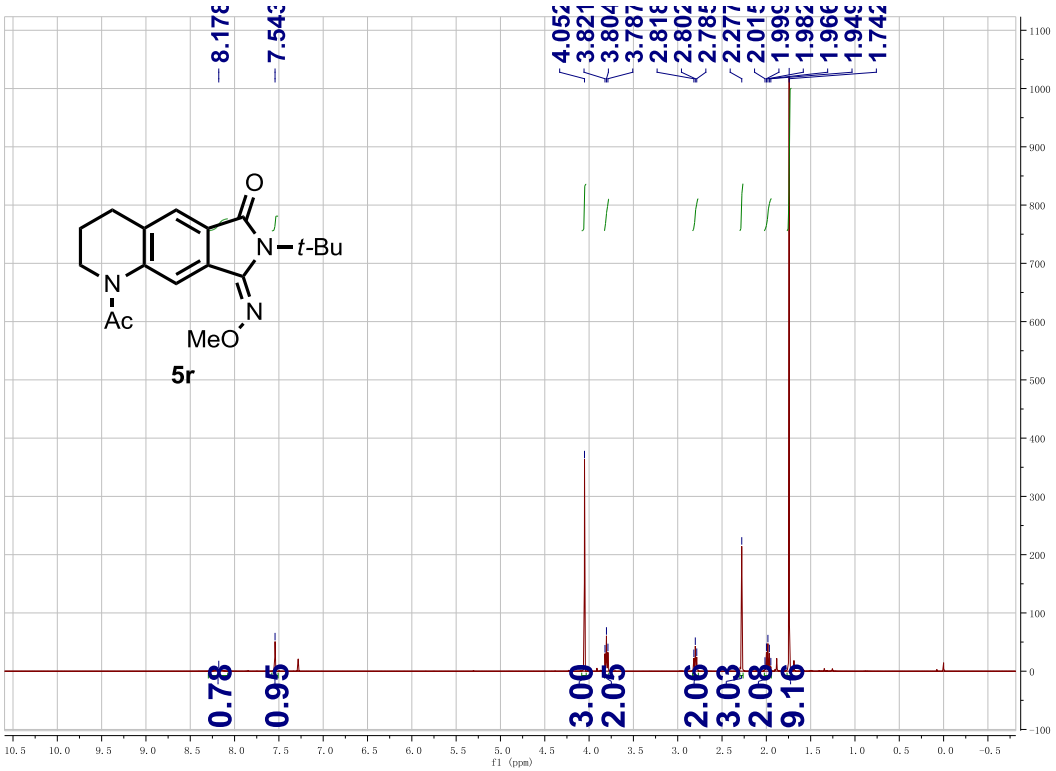
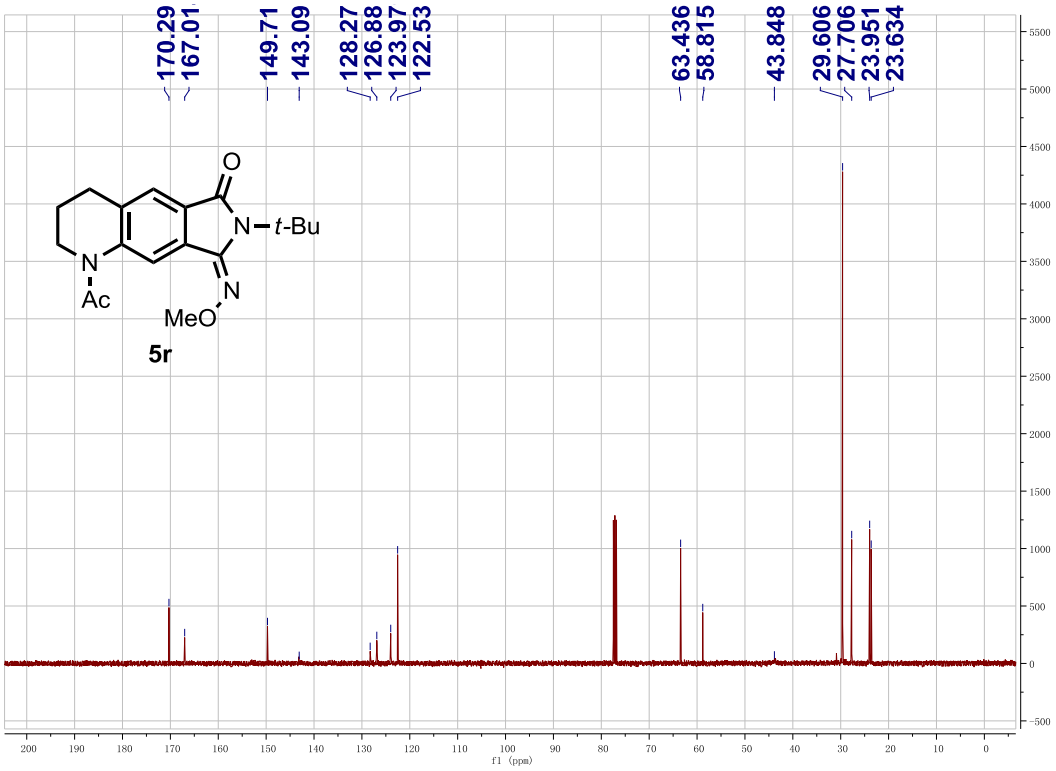
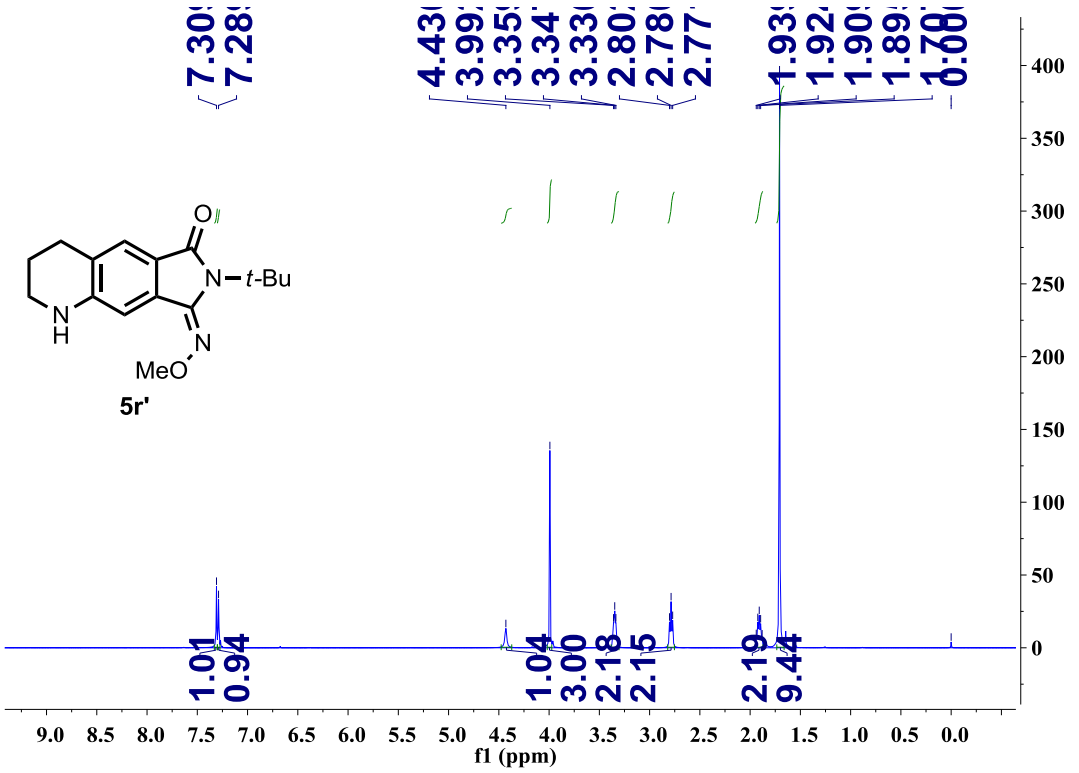
152.34
150.61
149.40
137.60
130.06
129.41
129.36
127.53
123.17
122.64
63.520
59.403
29.553
O
N
N
N–t-Bu
MeO
5p
f1 (ppm)

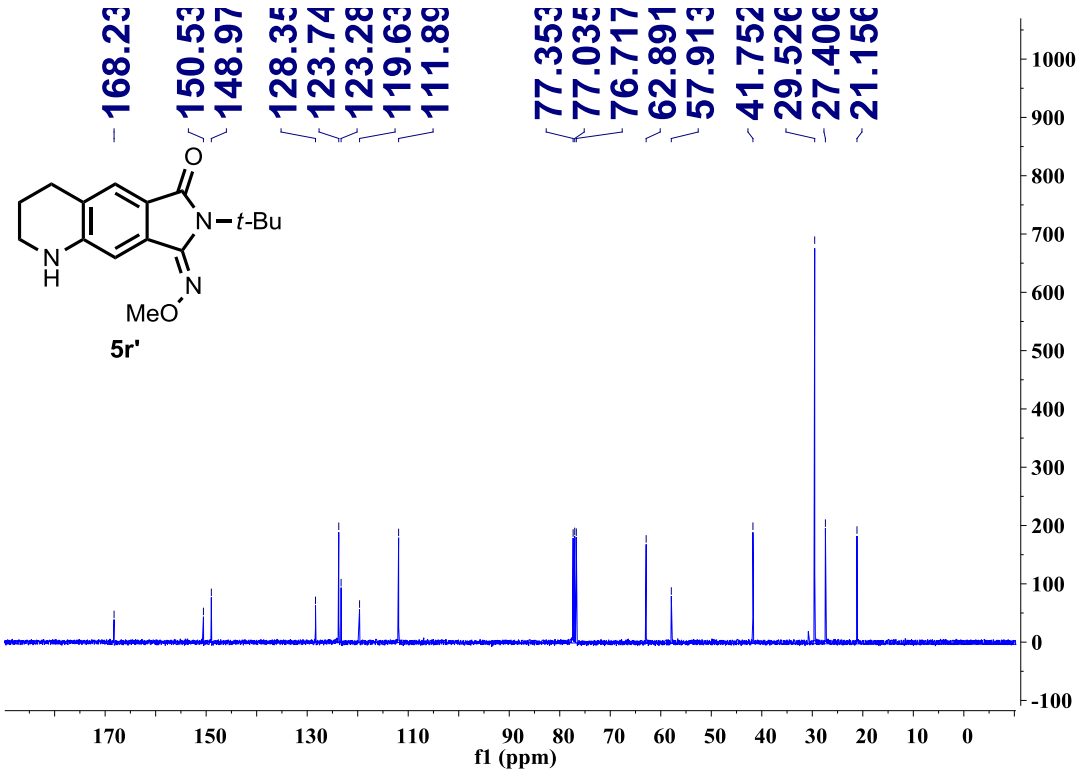

9.420
8.915
8.660
8.646
8.239
7.815
7.804
4.168
1.807
1.02
1.00
1.13
0.99
1.04
3.04
9.62
N–t-Bu
MeO
5q
f1 (ppm)

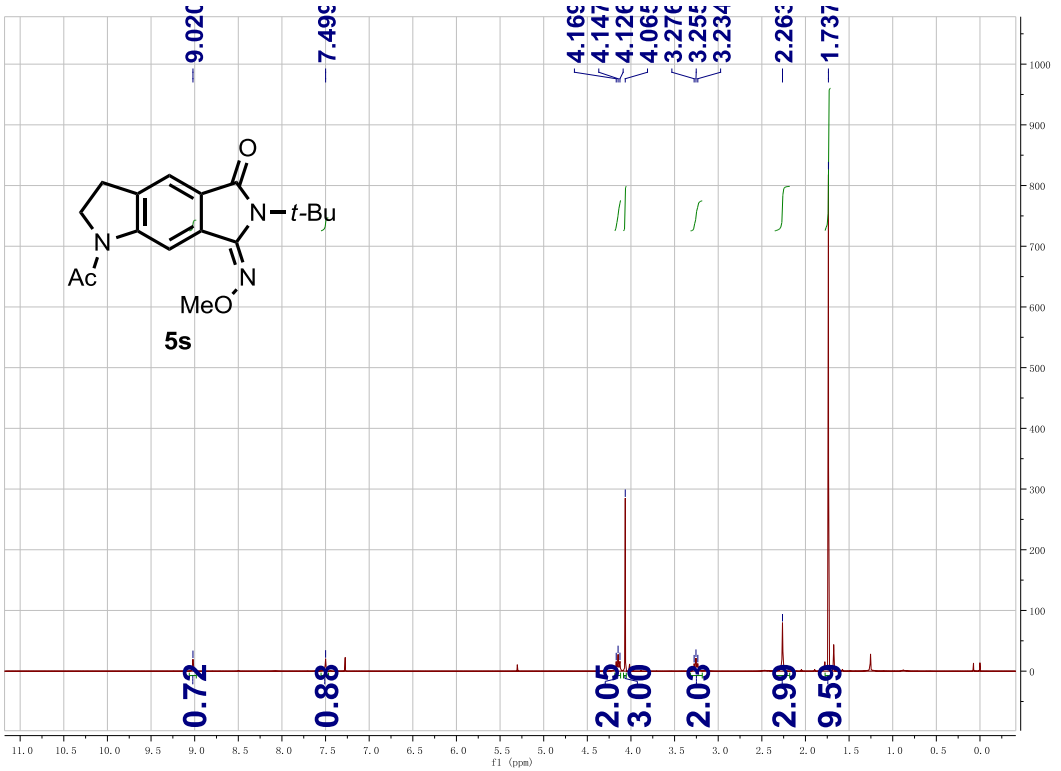

166.36
154.16
149.16
144.92
136.79
133.48
132.62
130.64
128.56
125.46
121.86
63.656
59.569
29.540
N–t-Bu
MeO
5q
f1 (ppm)

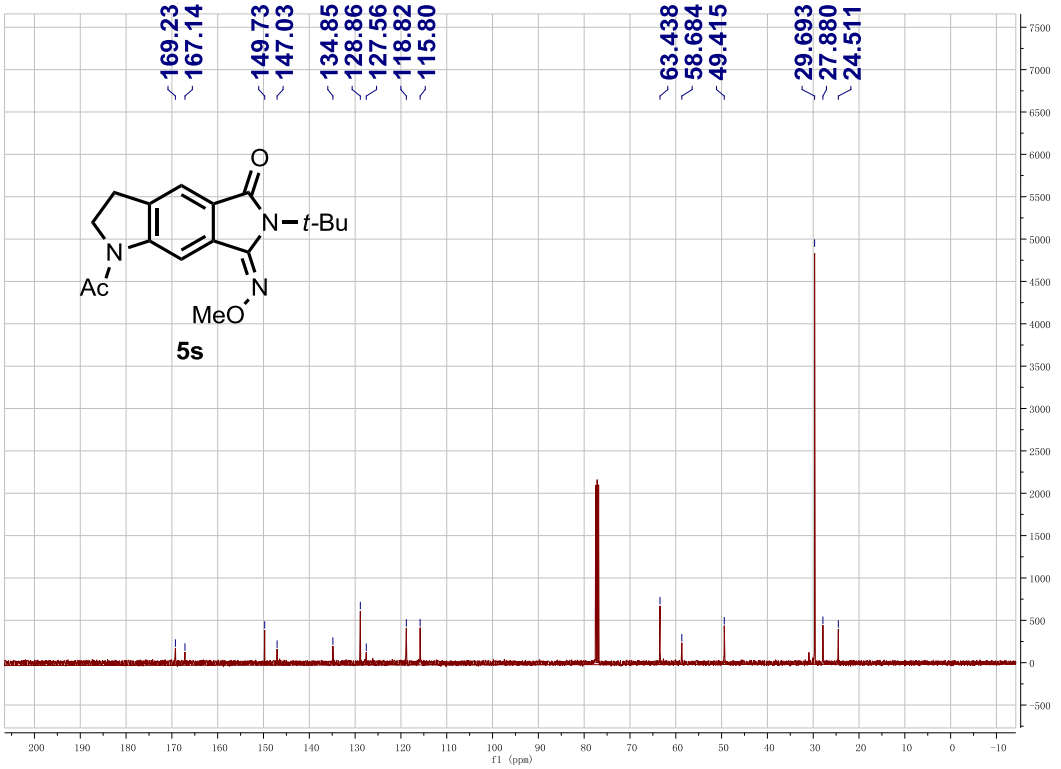

8.178
7.543
4.052
3.821
3.804
3.787
2.818
2.802
2.785
2.277
2.015
1.995
1.982
1.966
1.945
1.742
O
N–t-Bu
N
Ac
N
MeO
5r
0.78
0.95
3.00
2.05
2.06
3.03
2.08
9.16
f1 (ppm)

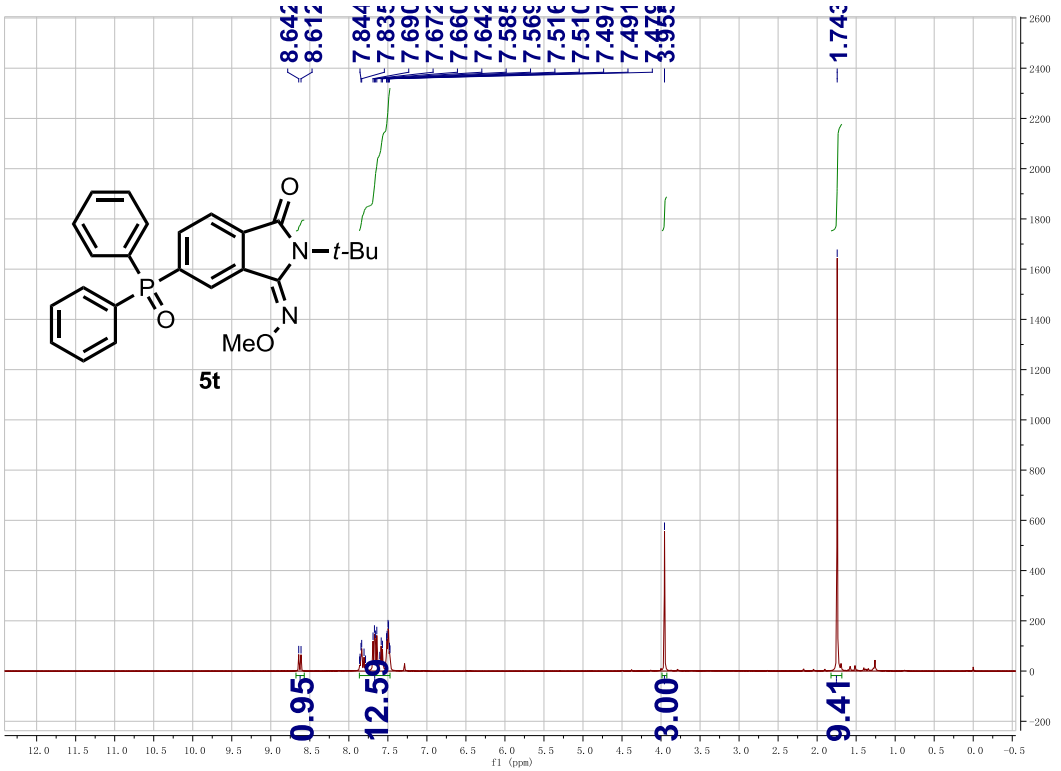

170.29
167.01
149.71
143.09
128.27
126.88
123.97
122.53
63.436
58.815
43.848
29.606
27.706
23.951
23.634
O
N–t-Bu
N
Ac
N
MeO
5r
f1 (ppm)

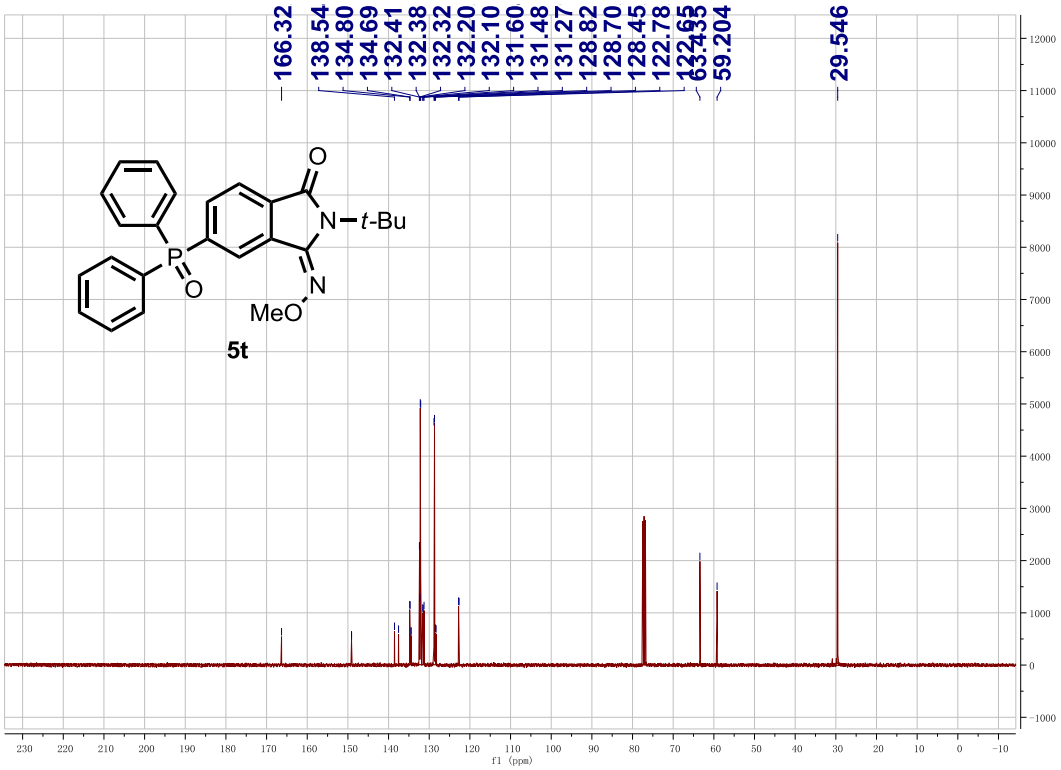

7.30
7.28
4.43
3.99
3.35
3.34
3.33
2.80
2.78
2.77
1.93
1.92
1.90
1.89
1.70
0.00
1.01
0.94
1.04
3.00
2.18
2.15
2.19
9.44
O
N–t-Bu
N
H
N
MeO
5r'
f1 (ppm)

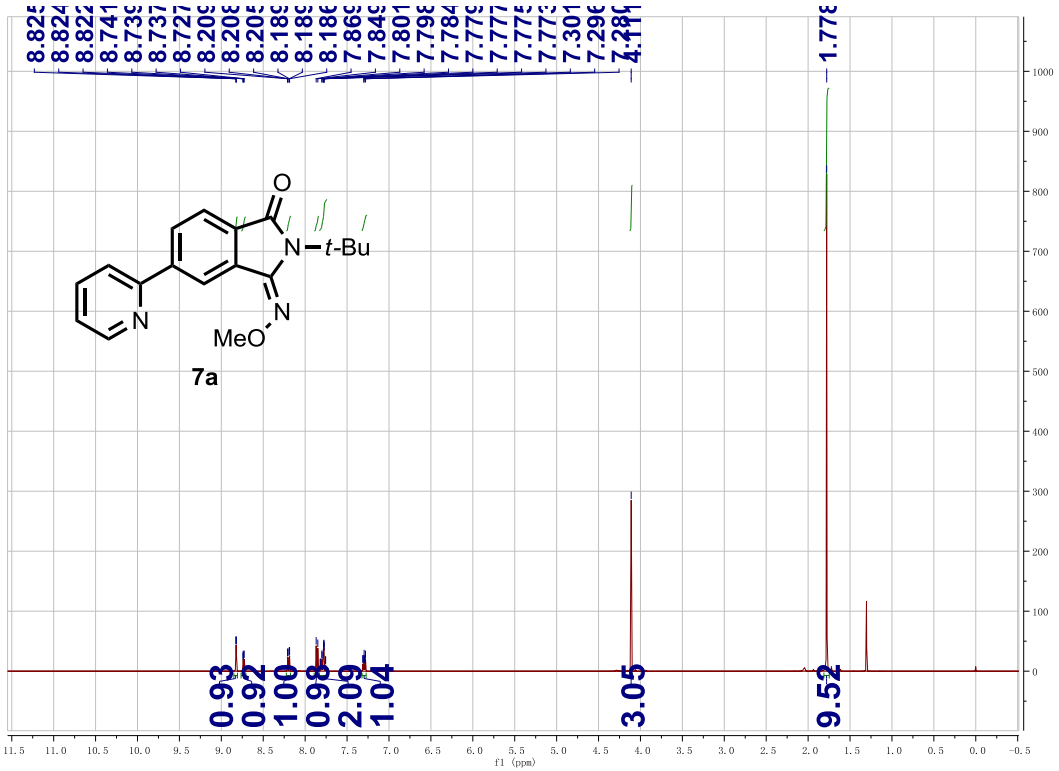

168.23
150.53
148.97
128.35
123.74
123.28
119.63
111.89
77.353
77.035
76.717
62.891
57.913
41.752
29.526
27.406
21.156
O
N–t-Bu
N
H
N
MeO
5r'
f1 (ppm)

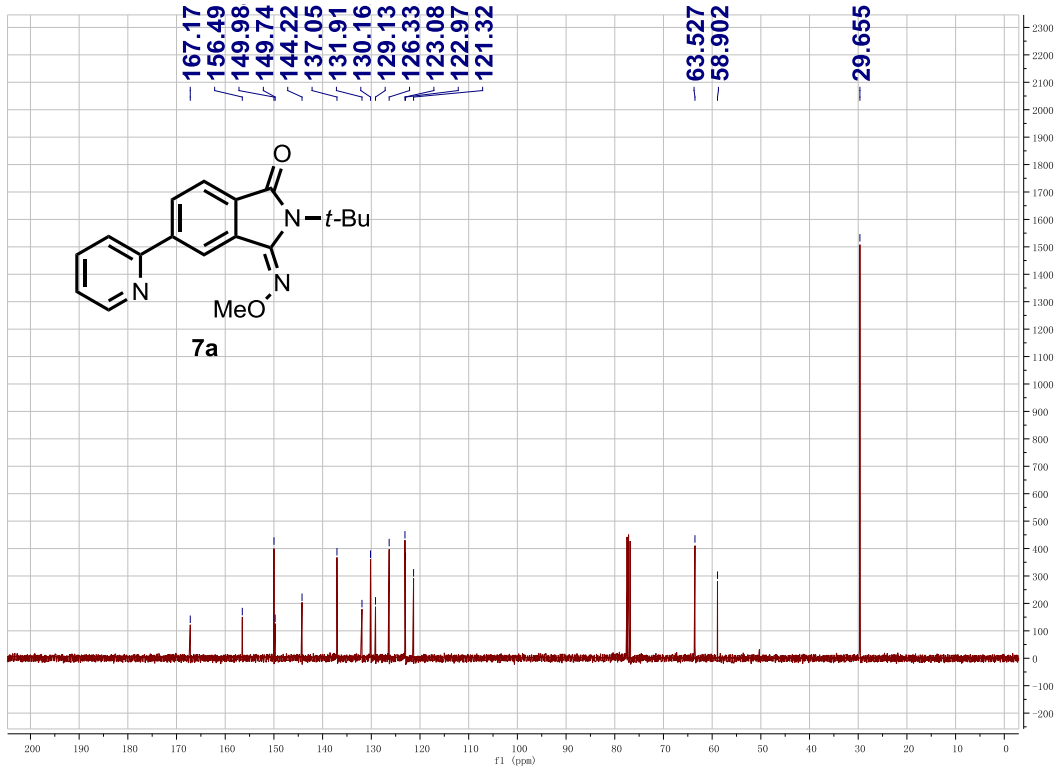

9.020
7.495
4.165
4.147
4.126
4.065
3.276
3.255
3.234
2.263
1.737
O
N–t-Bu
N
Ac
N
MeO
5s
0.72
0.88
2.05
3.00
2.03
2.99
9.59
f1 (ppm)

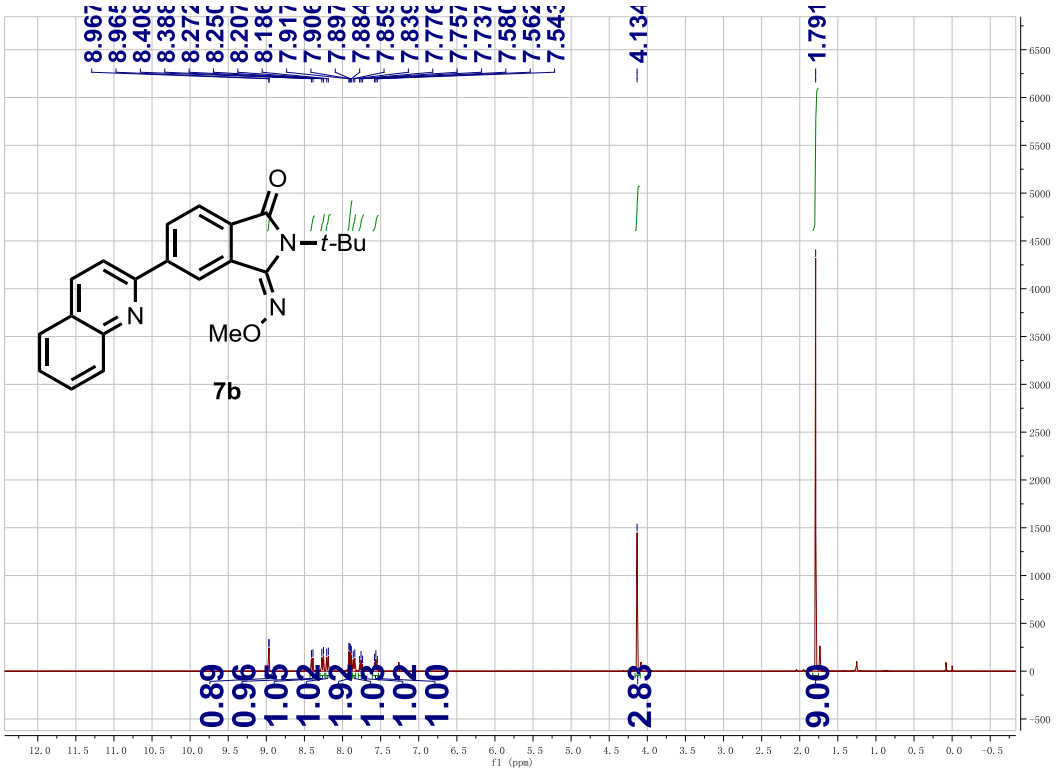

169.23
167.14
149.73
147.03
134.85
128.86
127.56
118.82
115.80
63.438
58.684
49.415
29.693
27.880
24.511
O
N–t-Bu
N
Ac
N
MeO
5s
f1 (ppm)

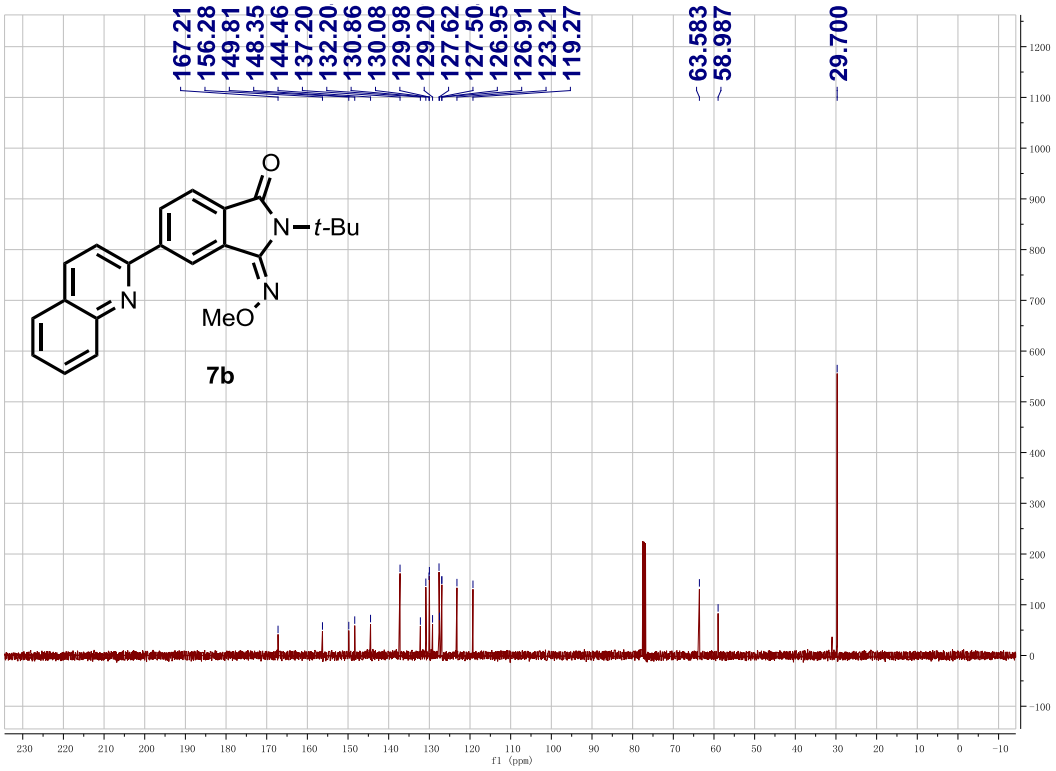

8.642
8.612
7.844
7.835
7.690
7.672
7.660
7.642
7.585
7.563
7.516
7.510
7.497
7.491
7.475
3.953
1.743
N–t-Bu
P
O
N
MeO
5t
0.95
12.59
3.00
9.41
f1 (ppm)

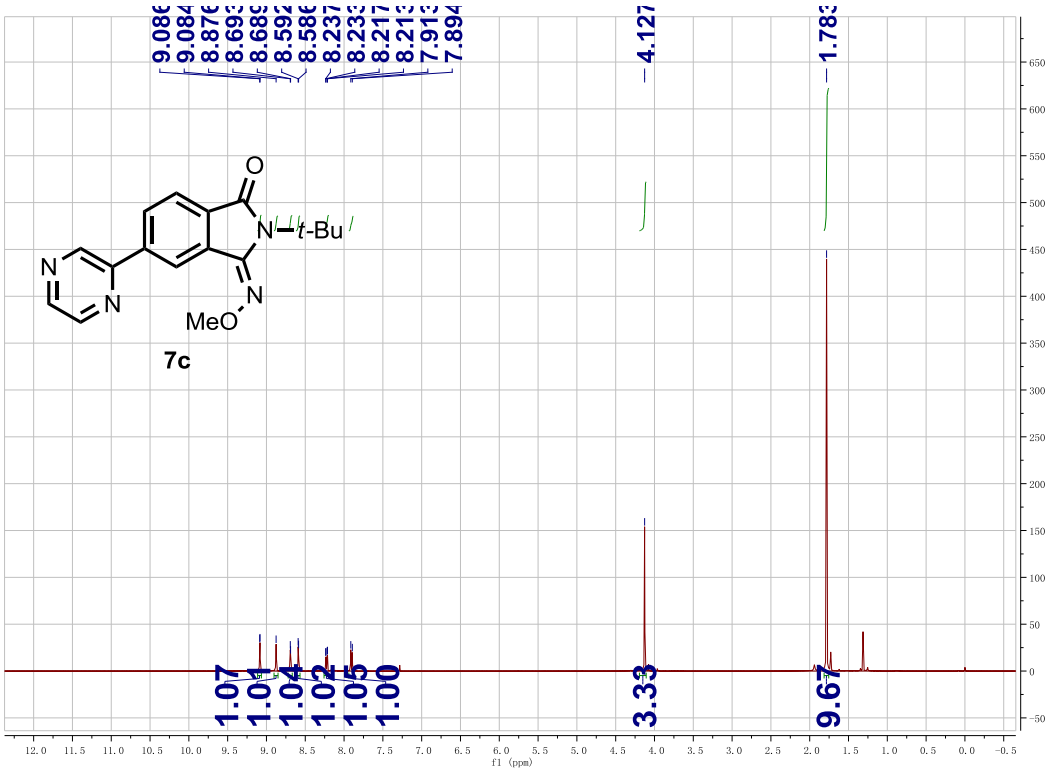

166.32
138.54
134.80
134.69
132.41
132.38
132.32
132.20
132.10
131.60
131.48
131.27
128.82
128.70
128.45
122.78
63.435
59.204
29.546
N–t-Bu
P
O
N
MeO
5t
f1 (ppm)

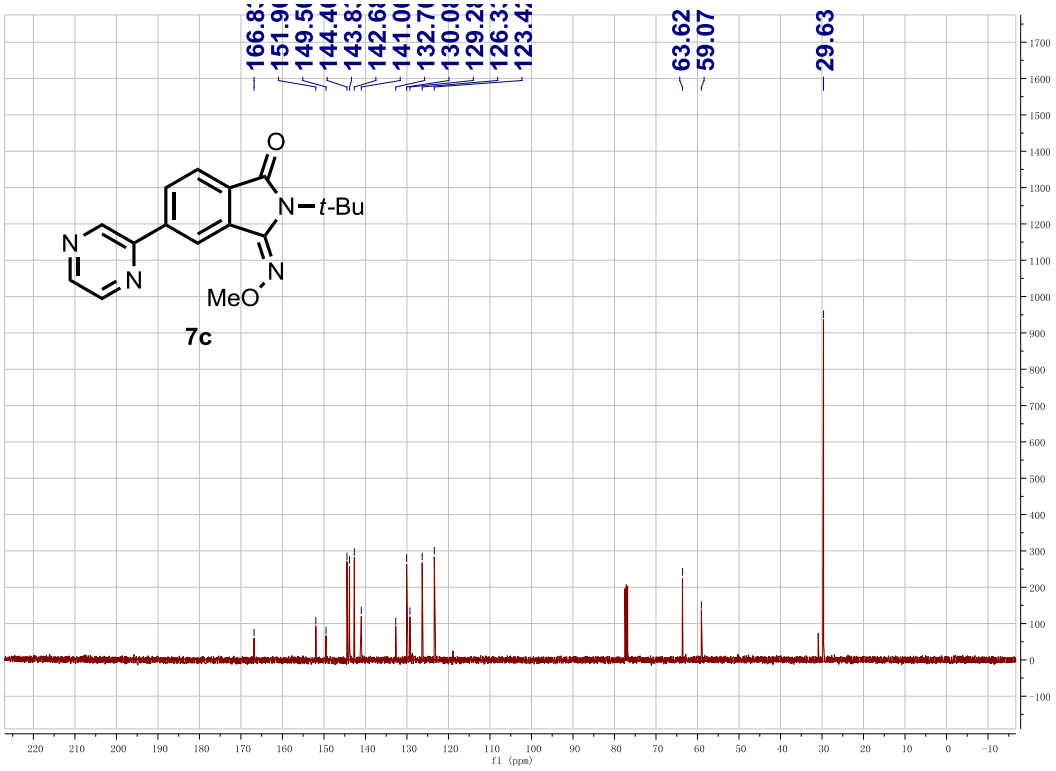

8.825
8.824
8.822
8.741
8.735
8.737
8.727
8.205
8.203
8.202
8.185
8.183
8.181
7.865
7.845
7.801
7.798
7.784
7.775
7.771
7.772
7.773
7.301
7.296
4.111
1.778
O
N−t-Bu
N
N
MeO
7a
0.93
0.92
1.00
0.98
2.09
1.04
3.05
9.52
f1 (ppm)


167.17
156.49
149.98
149.74
144.22
137.05
131.91
130.16
129.13
126.33
123.08
122.97
121.32
63.527
58.902
29.655
O
N−t-Bu
N
N
MeO
7a
f1 (ppm)


8.967
8.965
8.408
8.388
8.272
8.250
8.207
8.186
7.917
7.906
7.897
7.884
7.859
7.839
7.776
7.757
7.737
7.580
7.562
7.543
4.134
1.791
O
N–t-Bu
N
N
MeO
7b
0.89
0.96
1.05
1.03
1.92
1.03
1.02
1.00
2.83
9.00
f1 (ppm)


167.21
156.28
149.81
148.35
144.46
137.20
132.20
130.86
130.08
129.98
129.20
127.62
127.50
126.95
126.91
123.21
119.27
63.583
58.987
29.700
O
N–t-Bu
N
N
MeO
7b
f1 (ppm)


9.086
9.084
8.876
8.693
8.685
8.592
8.586
8.237
8.233
8.217
8.213
7.913
7.894
4.127
1.783
O
N–t-Bu
N
N
N
MeO
7c
1.07
1.01
1.04
1.02
1.05
1.00
3.33
9.67
f1 (ppm)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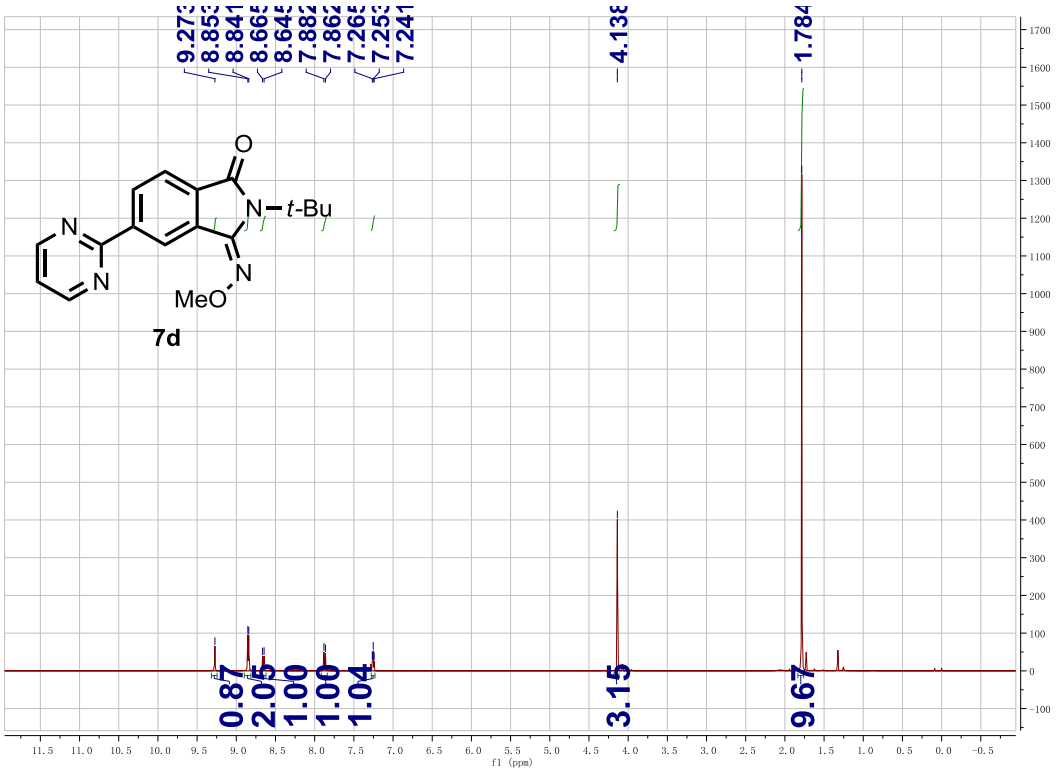
151.9
149.5
144.4
143.8
142.6
141.0
132.7
130.0
129.2
126.3
123.4
63.62
59.07
29.63
O
N–t-Bu
N
N
N
MeO
7c
f1 (ppm)


9.273
8.853
8.841
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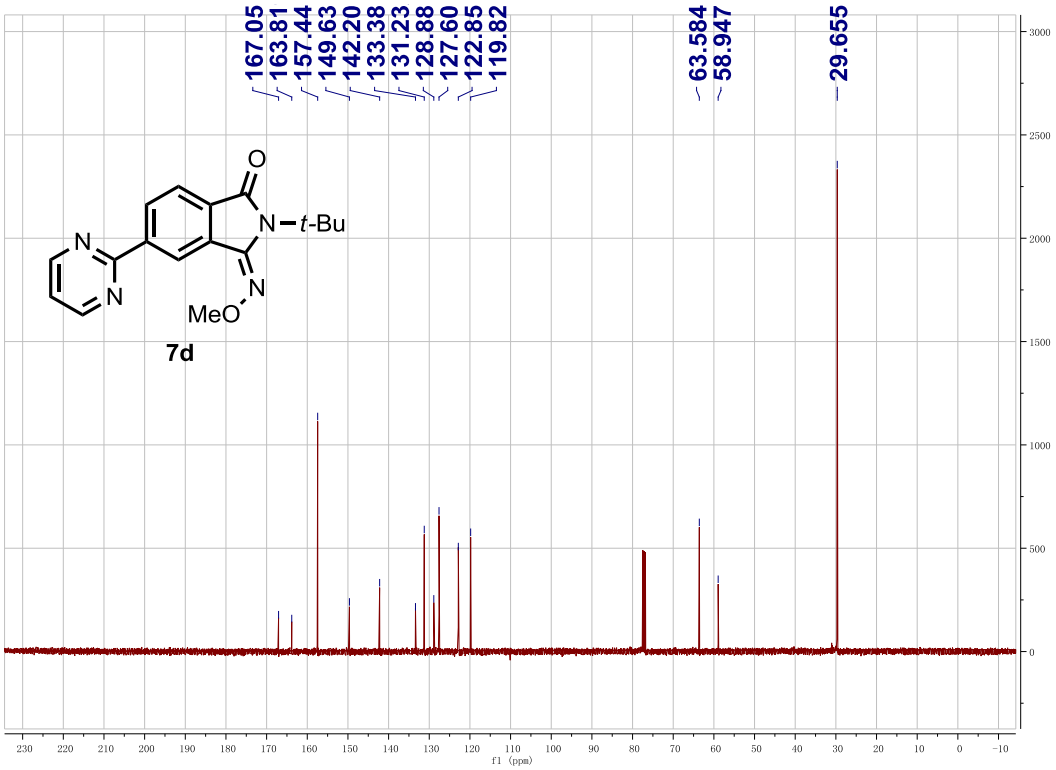
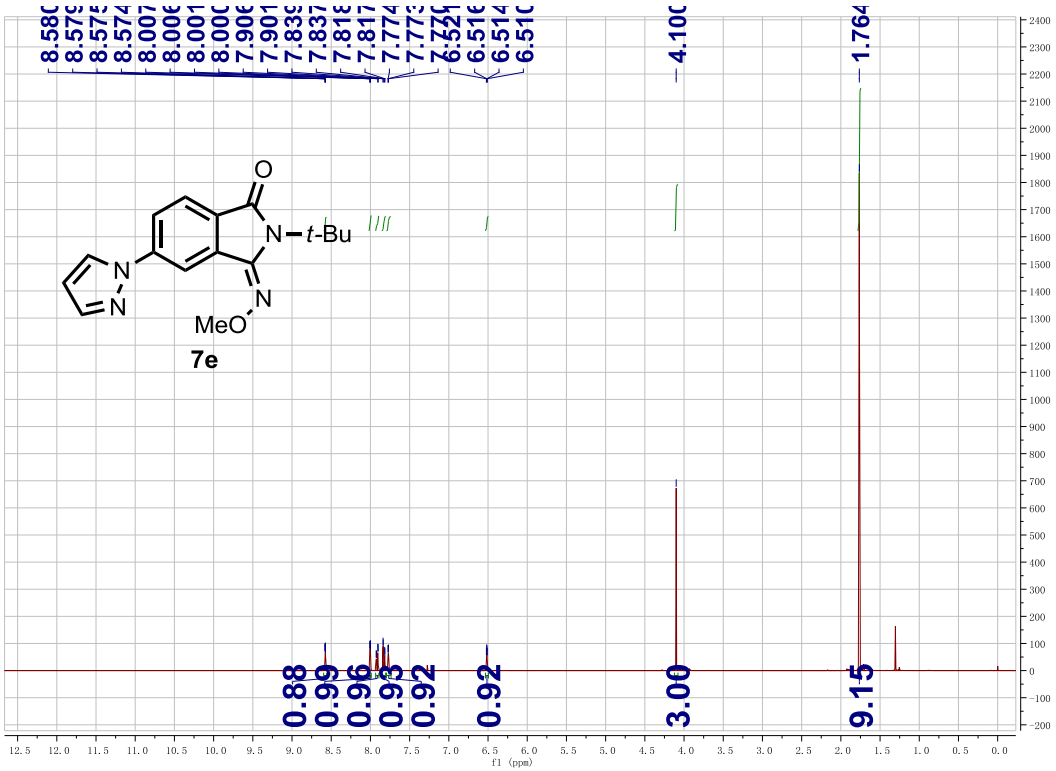
8.645
7.882
7.862
7.265
7.253
7.241
4.138
1.784
O
N
t-Bu
N
N
MeO
7d
0.87
2.05
1.00
1.00
1.04
3.15
9.67
f1 (ppm)


167.05
163.81
157.44
149.63
142.20
133.38
131.23
128.88
127.60
122.85
119.82
63.584
58.947
29.655
O
N
t-Bu
N
N
MeO
7d
f1 (ppm)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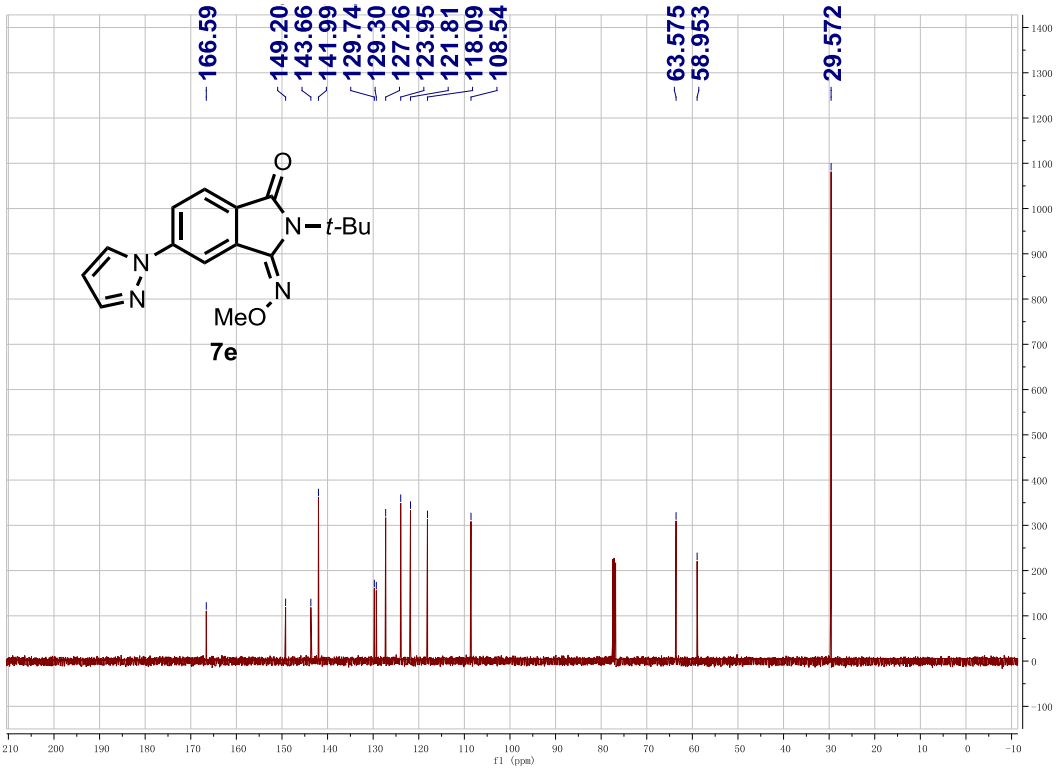
8.001
8.000
7.906
7.901
7.839
7.837
7.818
7.817
7.774
7.773
6.521
6.516
6.514
6.510
4.100
1.764
O
N–t-Bu
N
N
N
MeO
7e
0.88
0.99
0.96
0.93
0.92
0.92
3.00
9.15
f1 (ppm)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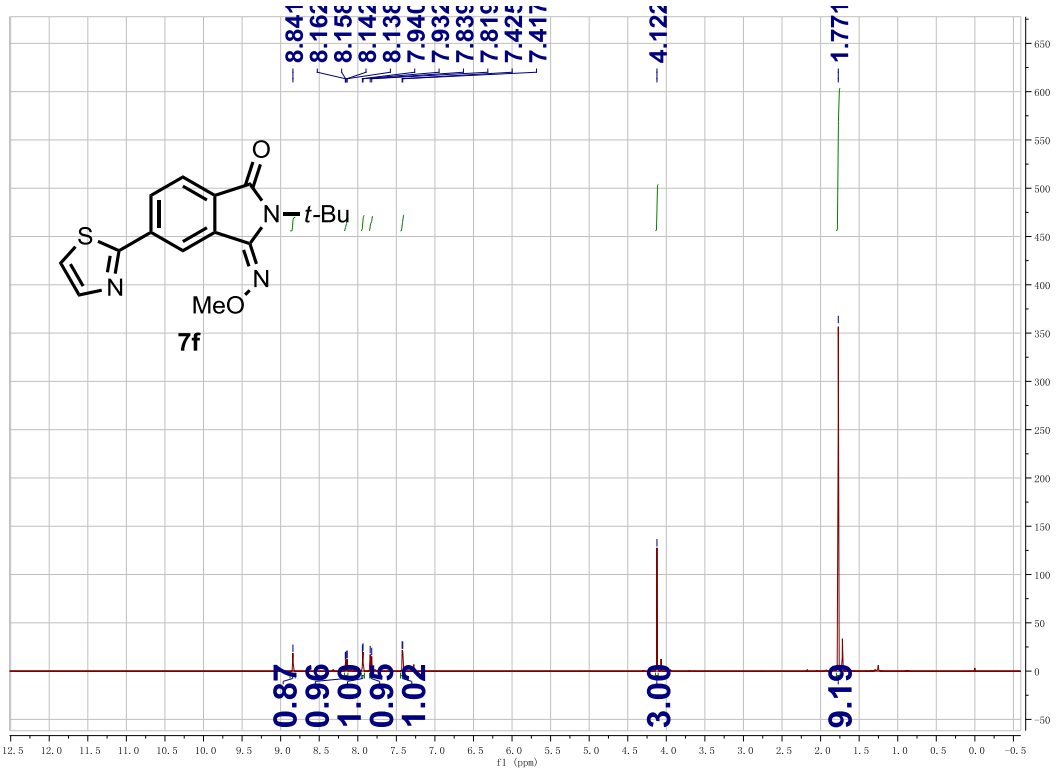
149.20
143.66
141.99
129.74
129.30
127.26
123.95
121.81
118.09
108.54
63.575
58.953
29.572
O
N–t-Bu
N
N
N
MeO
7e
f1 (ppm)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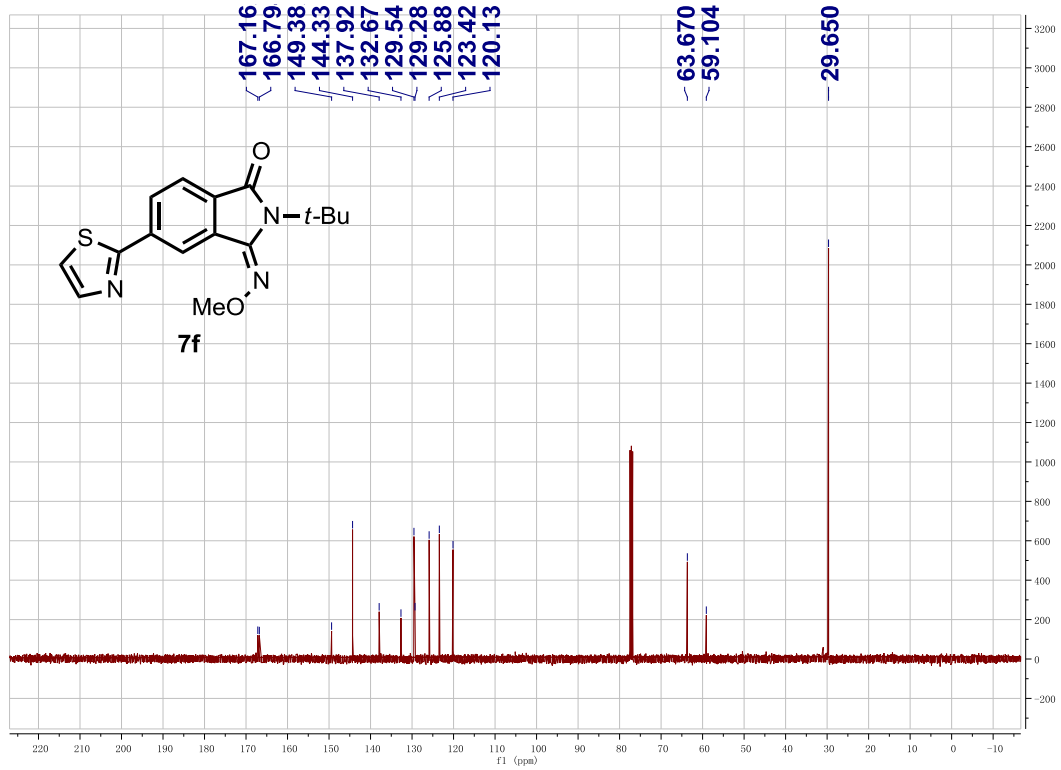
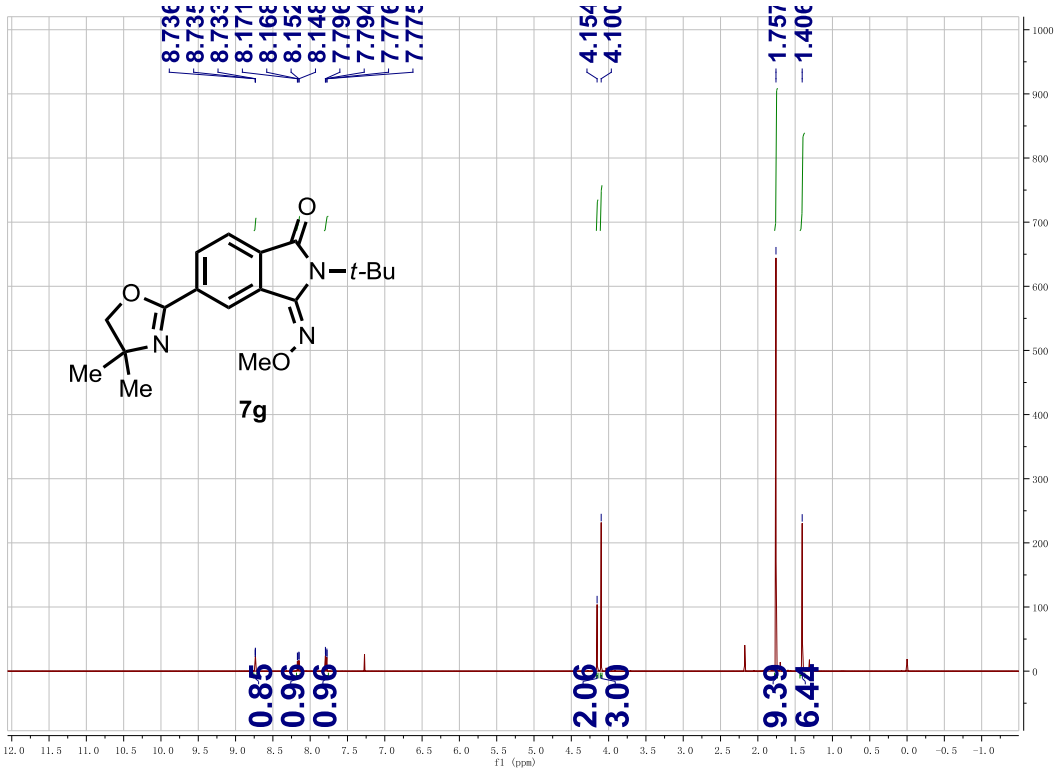
8.162
8.158
8.142
8.138
7.940
7.932
7.835
7.815
7.425
7.417
4.122
1.771
O
N–t-Bu
S
N
N
MeO
7f
0.87
0.96
1.00
0.95
1.02
3.00
9.19
f1 (ppm)


167.16
166.79
149.38
144.33
137.92
132.67
129.54
129.28
125.88
123.42
120.13
63.670
59.104
29.650
O
N–t-Bu
S
N
N
MeO
7f
f1 (ppm)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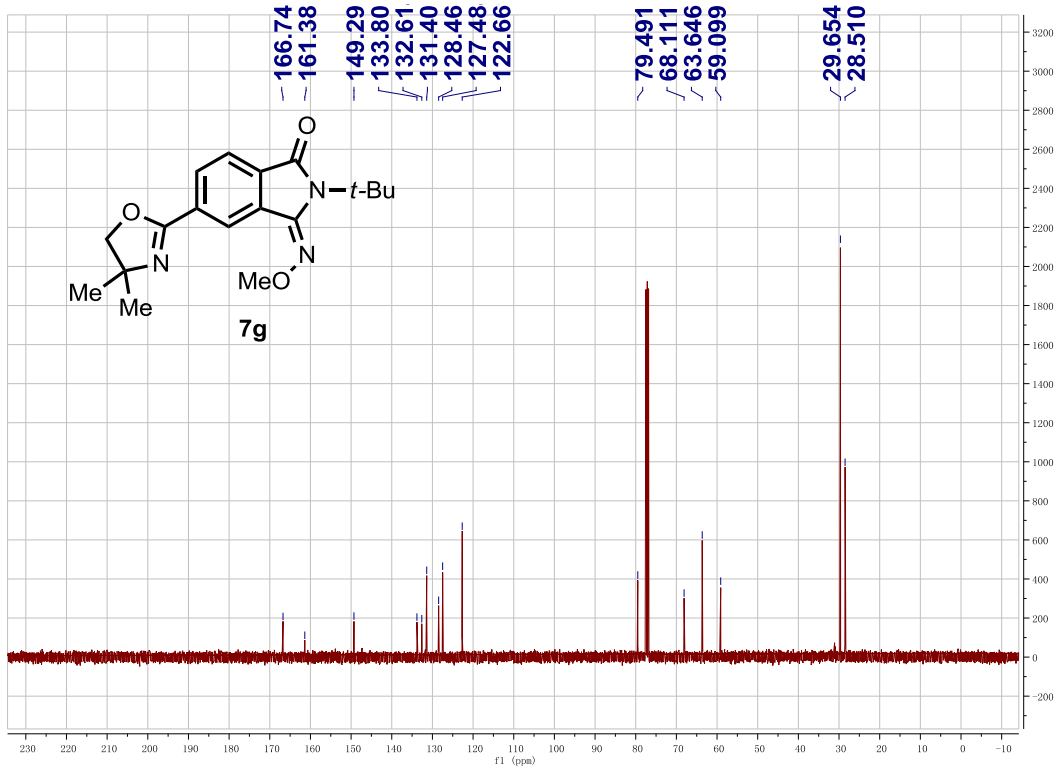
8.735
8.733
8.171
8.168
8.152
8.148
7.796
7.794
7.776
7.775
4.154
4.100
1.757
1.406
O
N–t-Bu
O
N
N
Me
Me
MeO
7g
0.85
0.96
0.96
2.06
3.00
9.39
6.44
f1 (ppm)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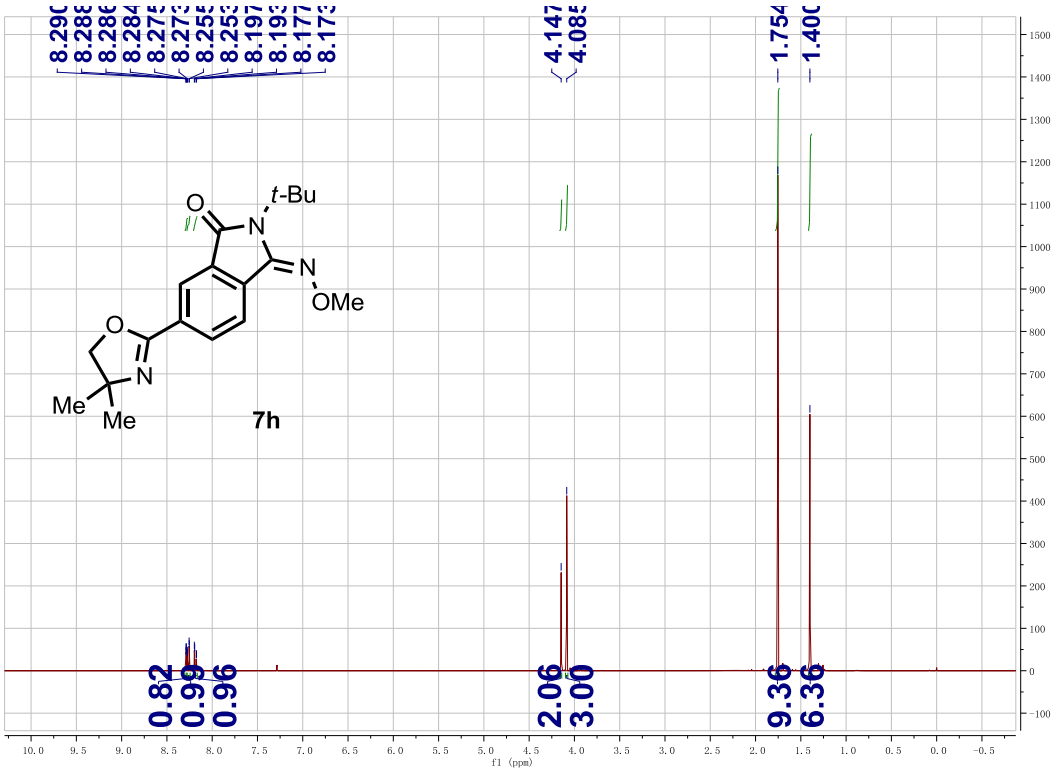
149.29
133.80
132.61
131.40
128.46
127.48
122.66
79.491
68.111
63.646
59.099
29.654
28.510
O
N–t-Bu
O
N
N
Me
Me
MeO
7g
f1 (ppm)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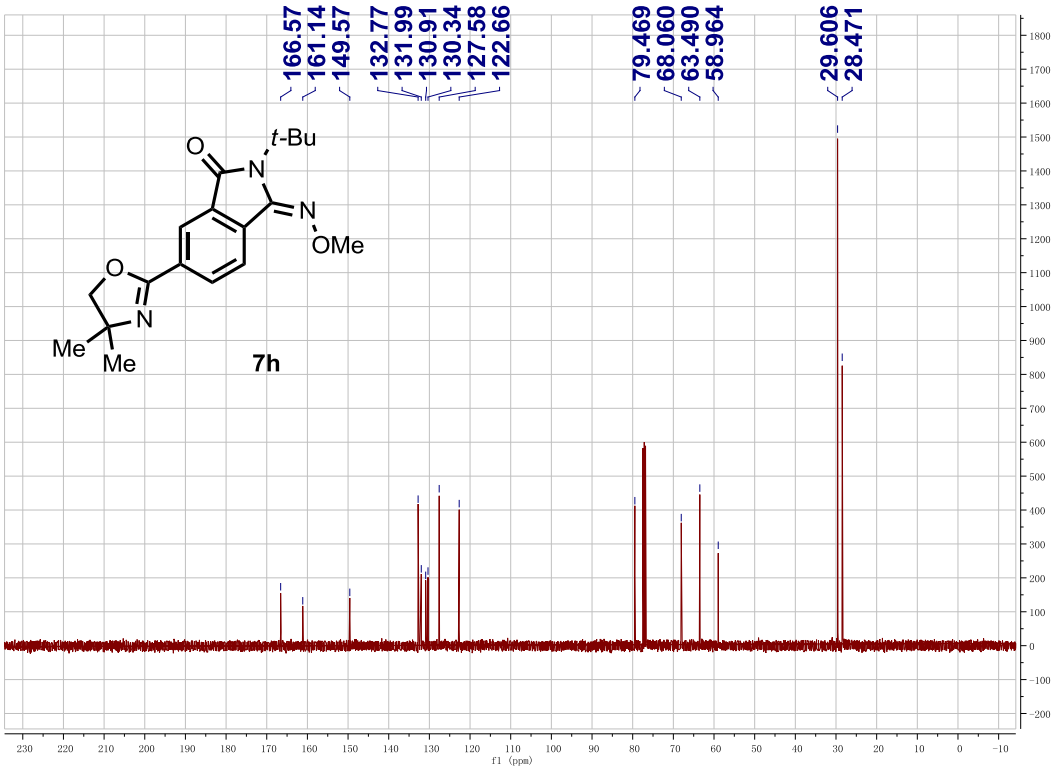
8.288
8.286
8.284
8.275
8.273
8.255
8.253
8.197
8.193
8.177
8.173
4.147
4.085
1.754
1.400
t-Bu
O
N
N
OMe
O
N
Me
Me
7h
0.82
0.99
0.96
2.06
3.00
9.36
6.36
f1 (ppm)


166.57
161.14
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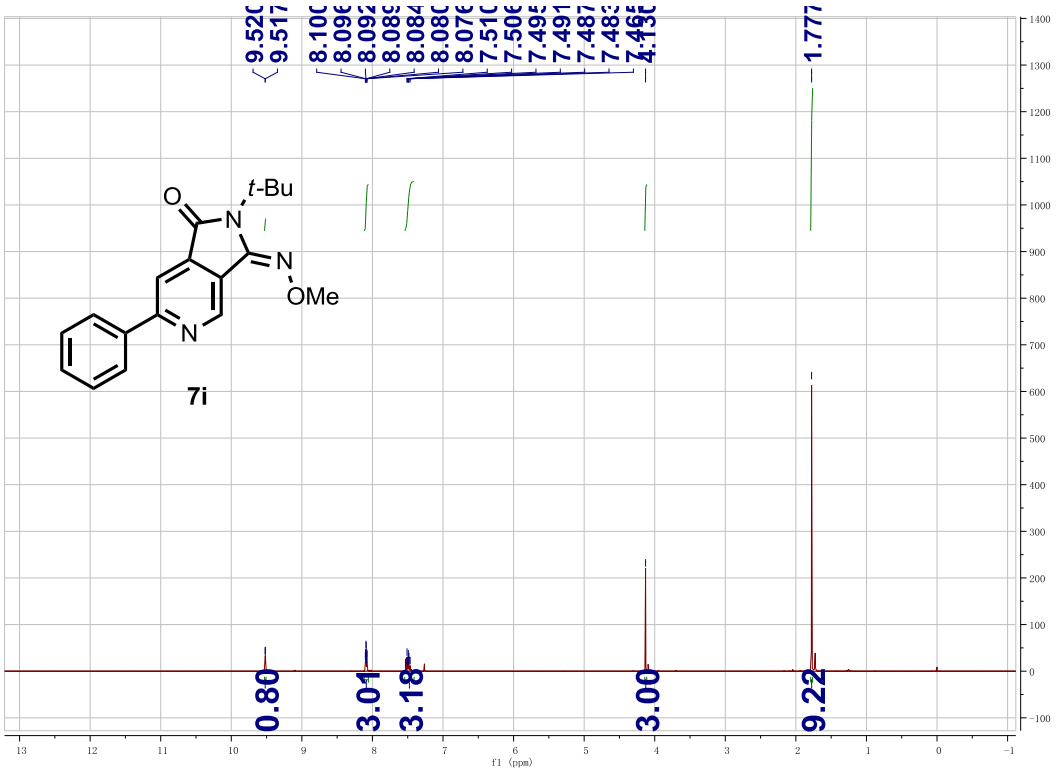
131.99
130.91
130.34
127.58
122.66
79.469
68.060
63.490
58.964
29.606
28.471
t-Bu
O
N
N
OMe
O
N
Me
Me
7h
f1 (ppm)


9.520
9.517
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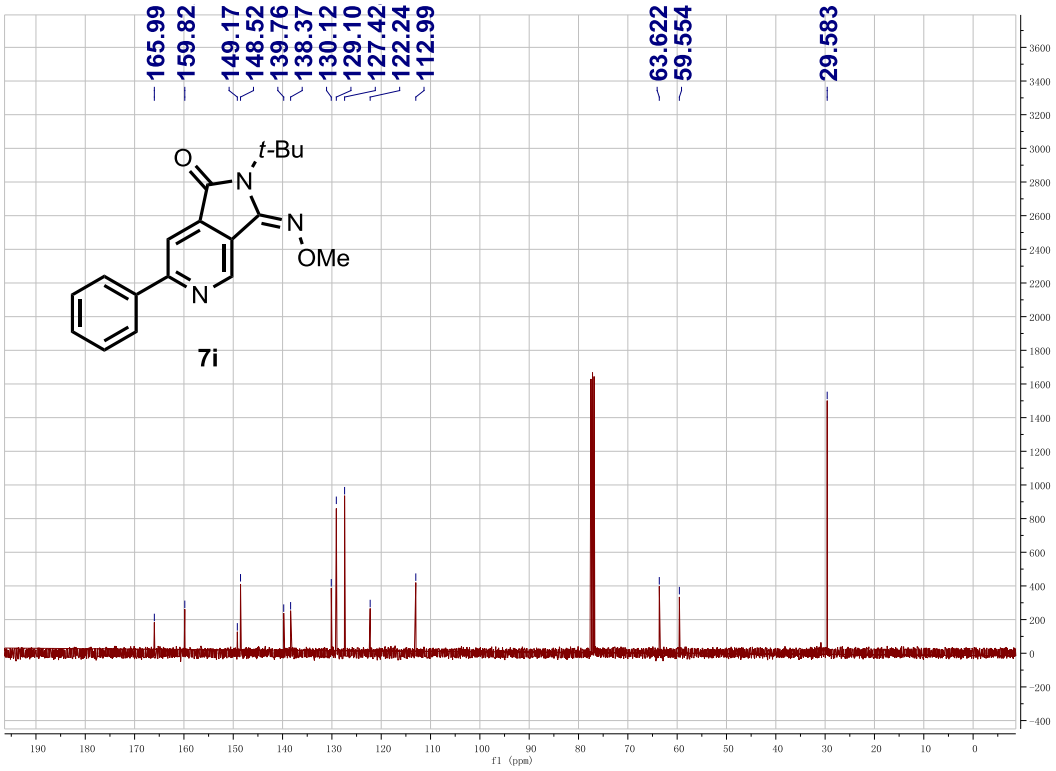
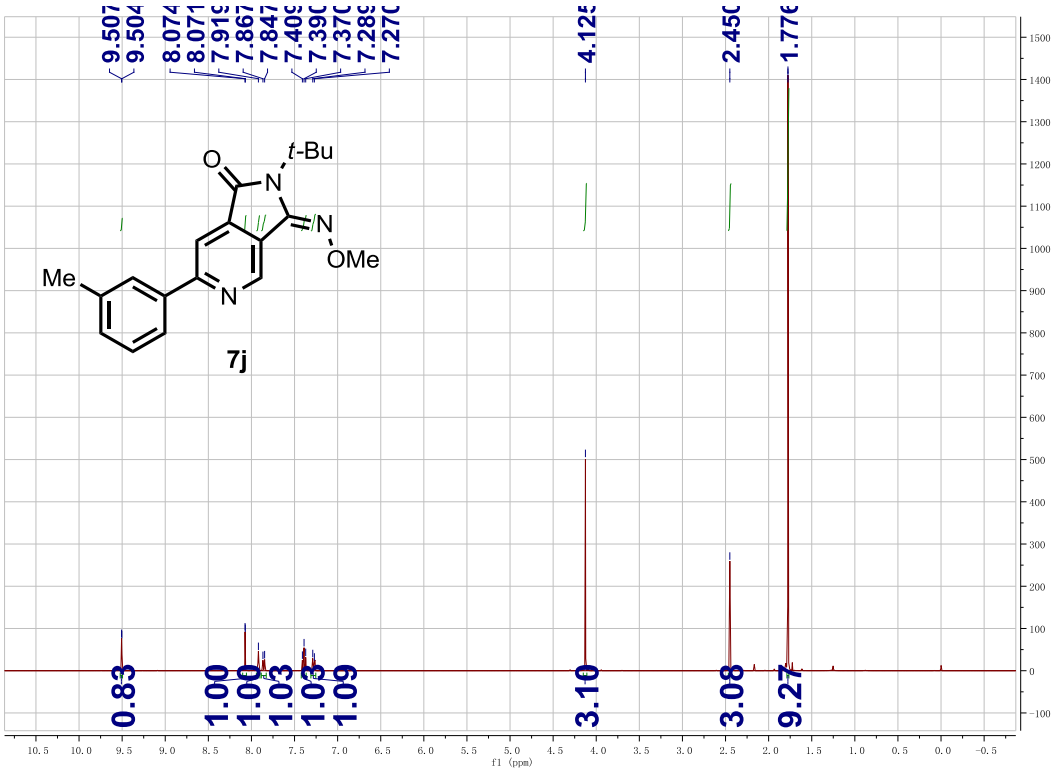
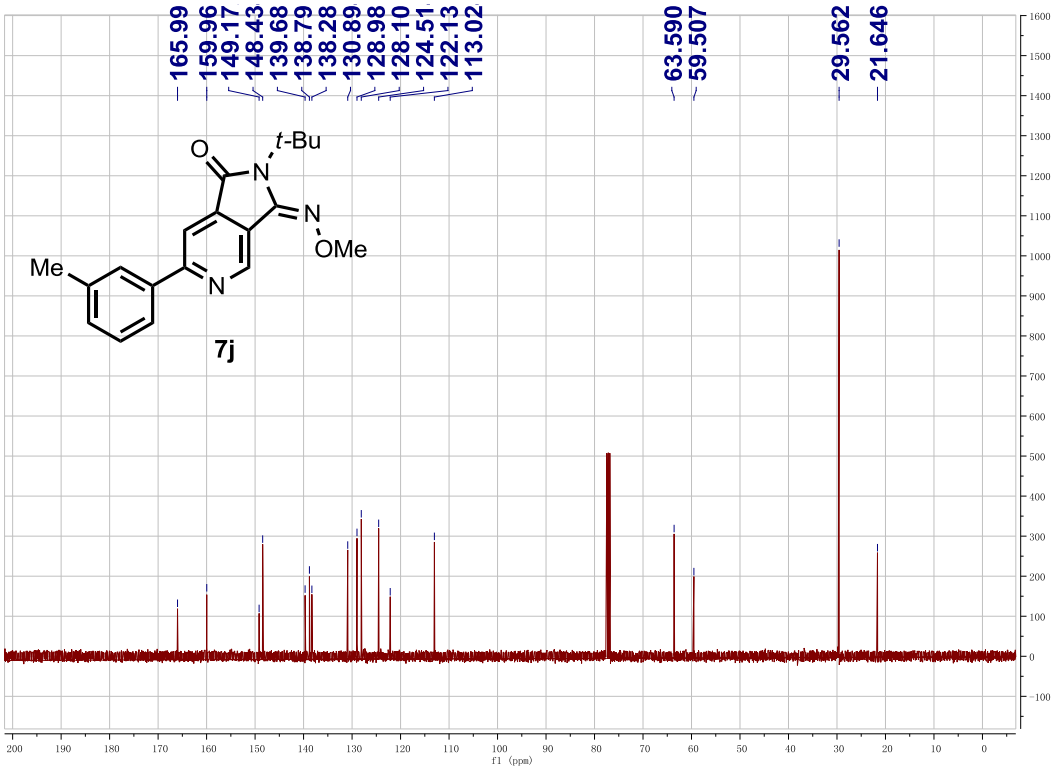
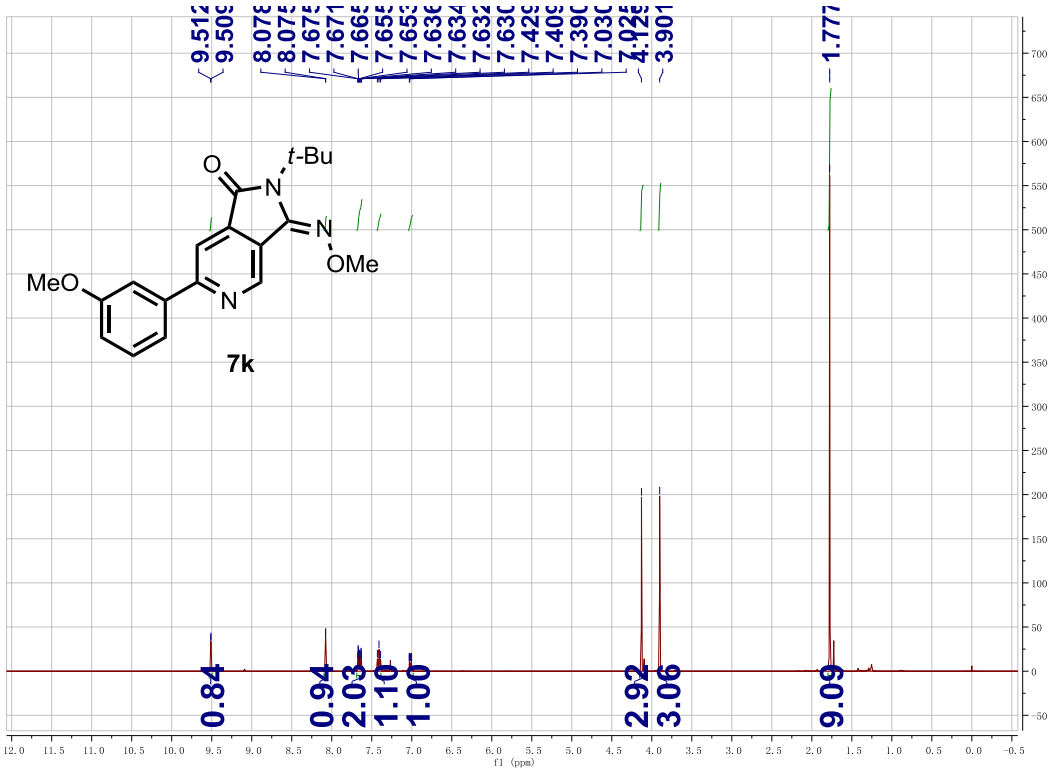
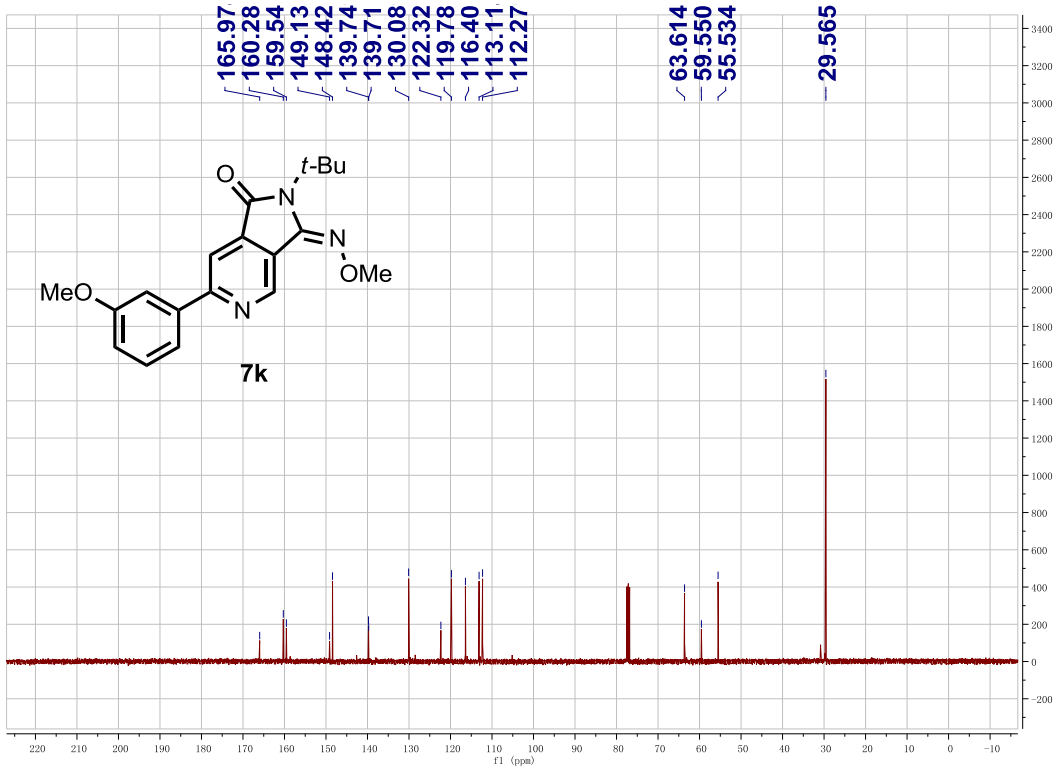
8.096
8.092
8.089
8.084
8.080
8.076
7.510
7.506
7.495
7.491
7.487
7.483
7.465
7.430
1.777
0.80
3.01
3.18
3.00
9.22
t-Bu
O
N
N
OMe
N
7i
f1 (ppm)

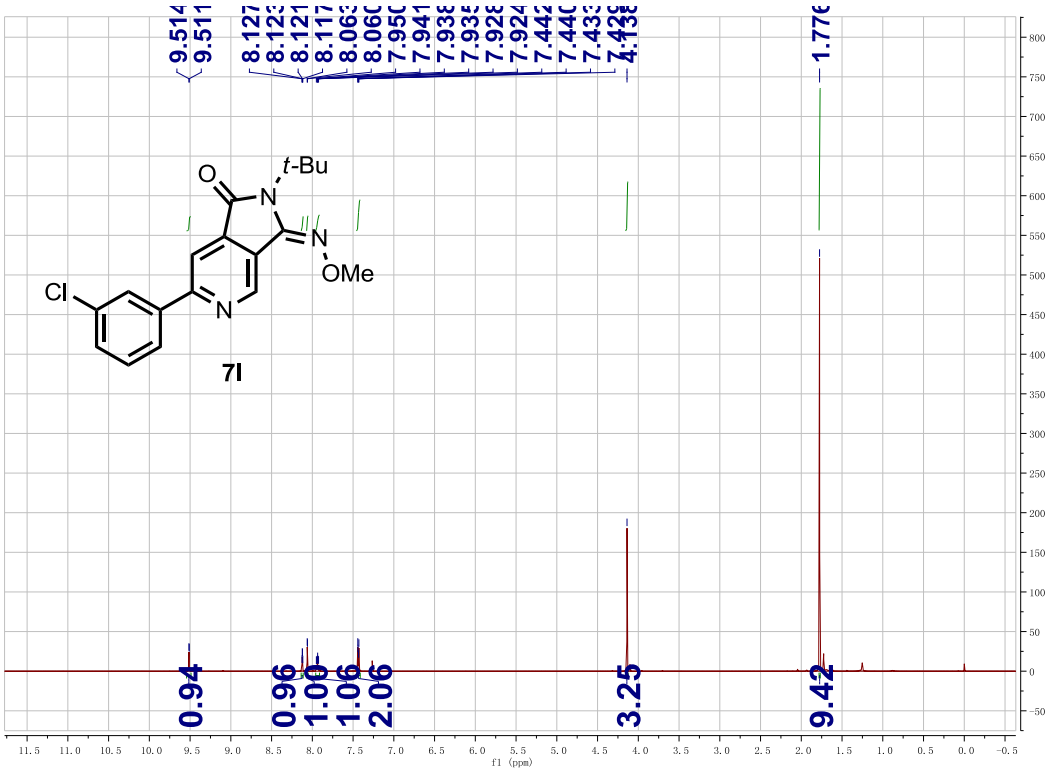

165.99
159.82
149.17
148.52
139.76
138.37
130.12
129.10
127.42
122.24
112.99
63.622
59.554
29.583
t-Bu
O
N
N
OMe
N
7i
f1 (ppm)

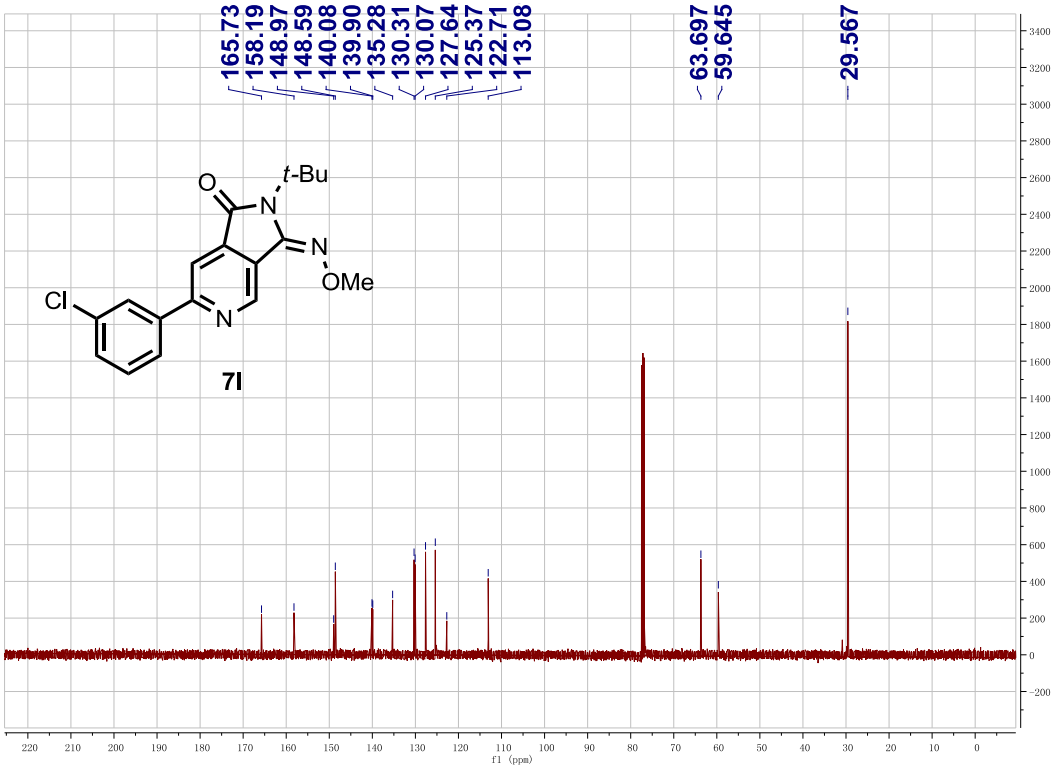

9.507
9.504
8.074
8.071
7.919
7.867
7.847
7.405
7.390
7.370
7.289
7.270
4.125
2.450
1.776
t-Bu
O
N
N
OMe
Me
N
7j
0.83
1.00
1.00
1.03
1.03
1.09
3.10
3.08
9.27
f1 (ppm)

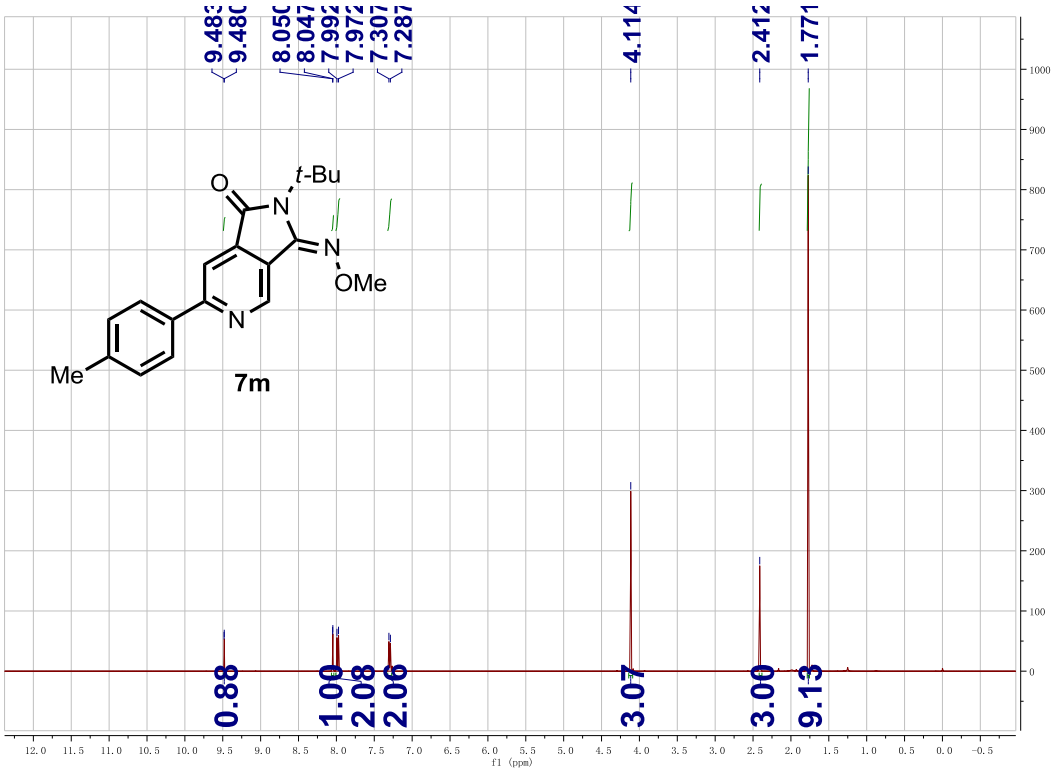

165.99
159.96
149.17
148.43
139.68
138.79
138.28
130.89
128.98
128.10
124.51
122.13
113.02
63.590
59.507
29.562
21.646
t-Bu
O
N
N
OMe
Me
N
7j
f1 (ppm)

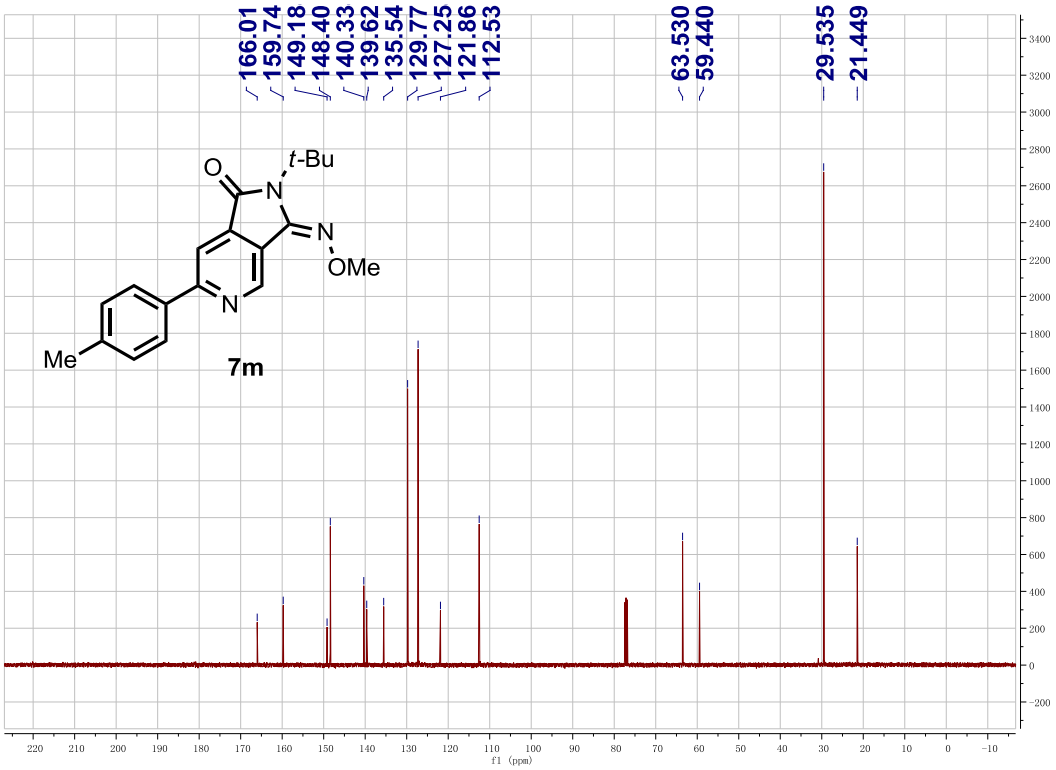

t-Bu
O
N
N
OMe
MeO
N
7k
1.777
3.901
4.125
7.025
7.030
7.390
7.405
7.425
7.630
7.632
7.634
7.636
7.653
7.655
7.665
7.671
7.675
8.075
8.078
9.505
9.512
0.84
0.94
2.03
1.10
1.00
2.92
3.06
9.09
f1 (ppm)

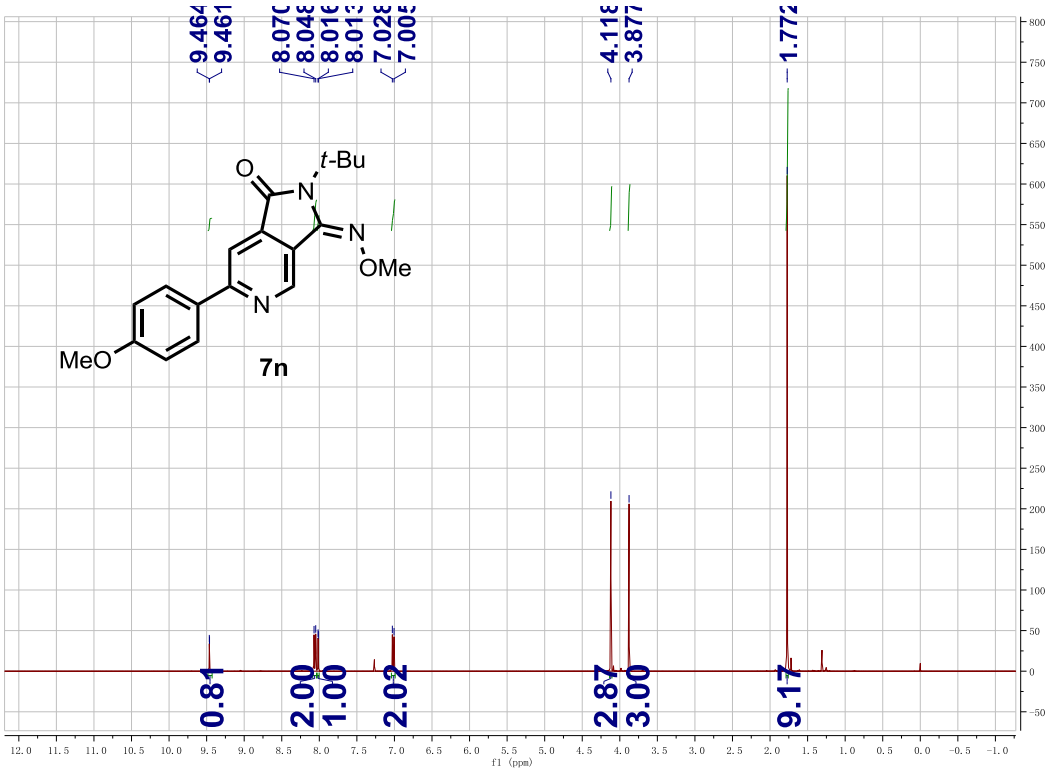

t-Bu
O
N
N
OMe
MeO
N
7k
165.97
160.28
159.54
149.13
148.42
139.74
139.71
130.08
122.32
119.78
116.40
113.11
112.27
63.614
59.550
55.534
29.565
f1 (ppm)

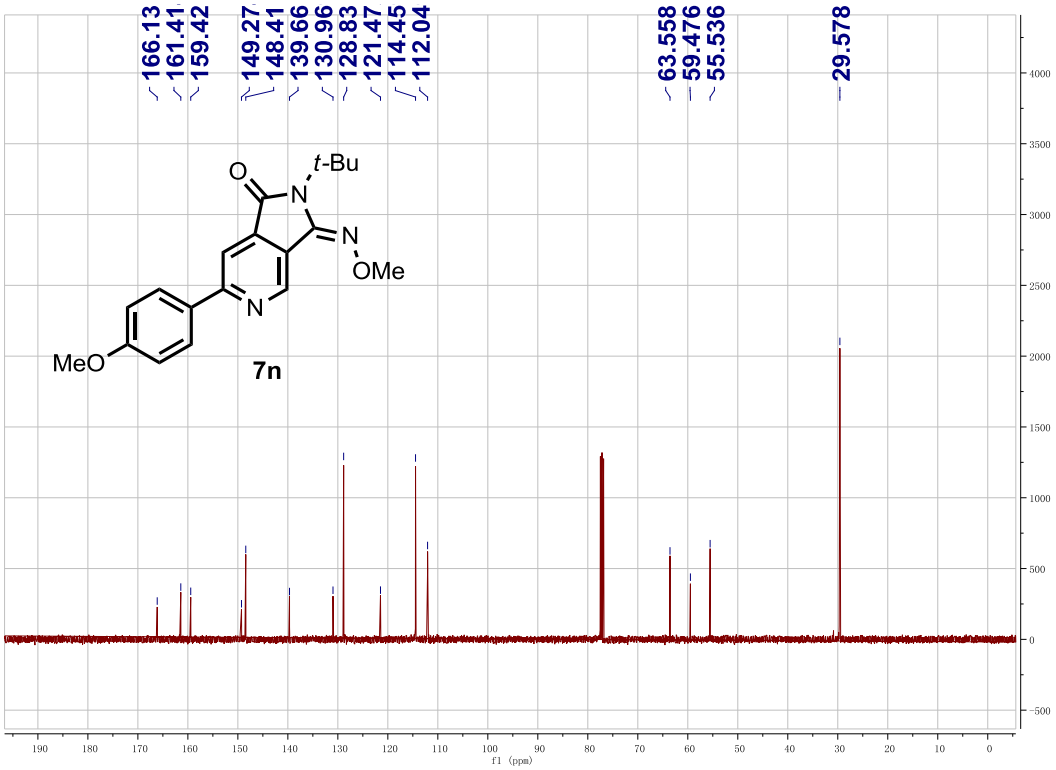

9.514
9.511
8.127
8.123
8.121
8.117
8.063
8.060
7.950
7.941
7.938
7.935
7.928
7.924
7.442
7.440
7.433
4.438
1.776
t-Bu
O
N
N
OMe
Cl
N
7l
0.94
0.96
1.00
1.06
2.06
3.25
9.42
f1 (ppm)

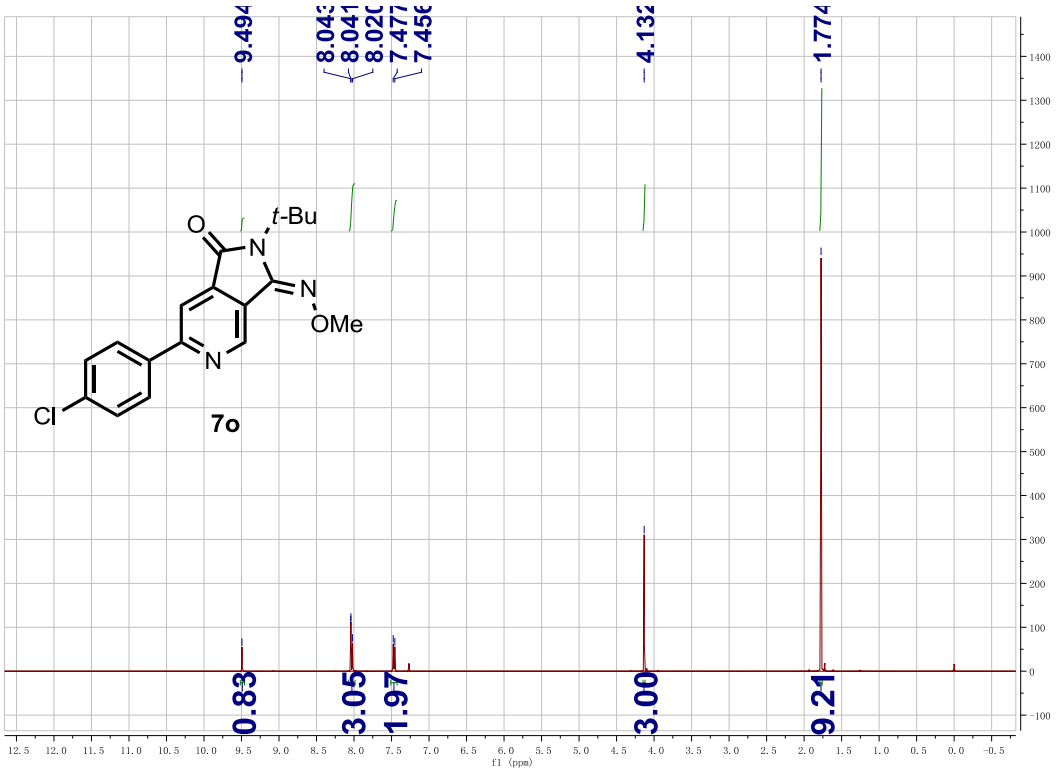

165.73
158.19
148.97
148.59
140.08
139.90
135.28
130.31
130.07
127.64
125.37
122.71
113.08
63.697
59.645
29.567
t-Bu
O
N
N
OMe
Cl
N
7l
f1 (ppm)

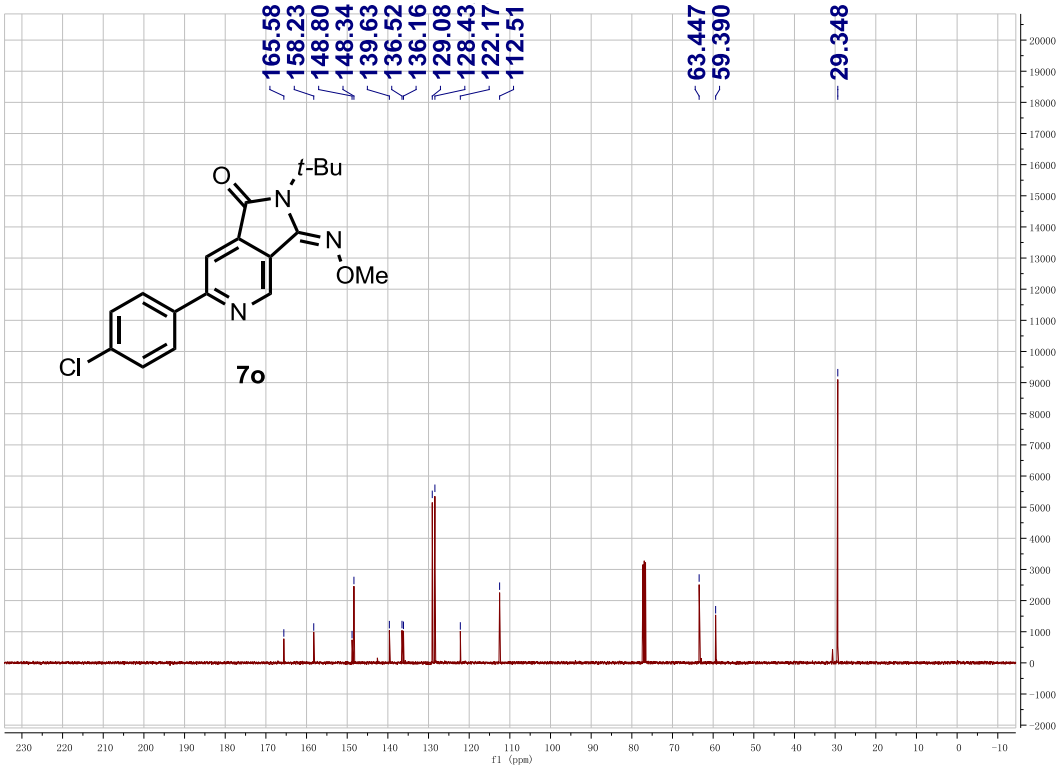

9.483
9.480
8.050
8.047
7.992
7.972
7.307
7.287
4.114
2.412
1.771
t-Bu
O
N
N
OMe
N
Me
7m
0.88
1.00
2.08
2.06
3.07
3.00
9.13
f1 (ppm)

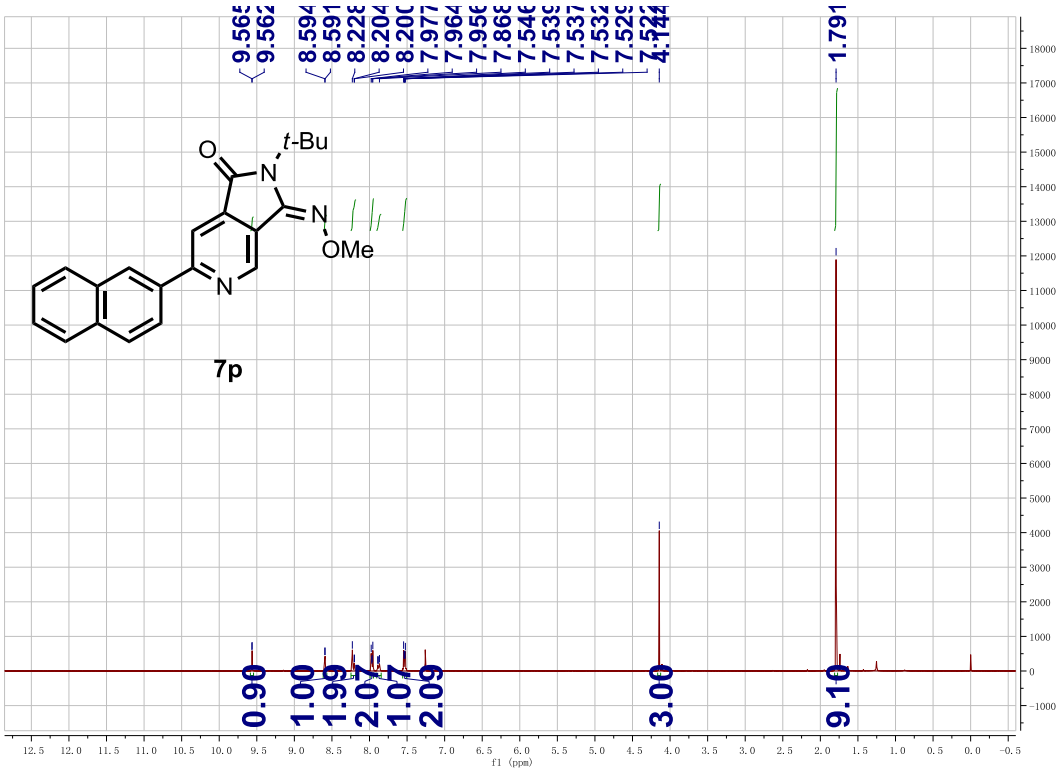

166.01
159.74
149.18
148.40
140.33
139.62
135.54
129.77
127.25
121.86
112.53
63.530
59.440
29.535
21.449
t-Bu
O
N
N
OMe
N
Me
7m
f1 (ppm)

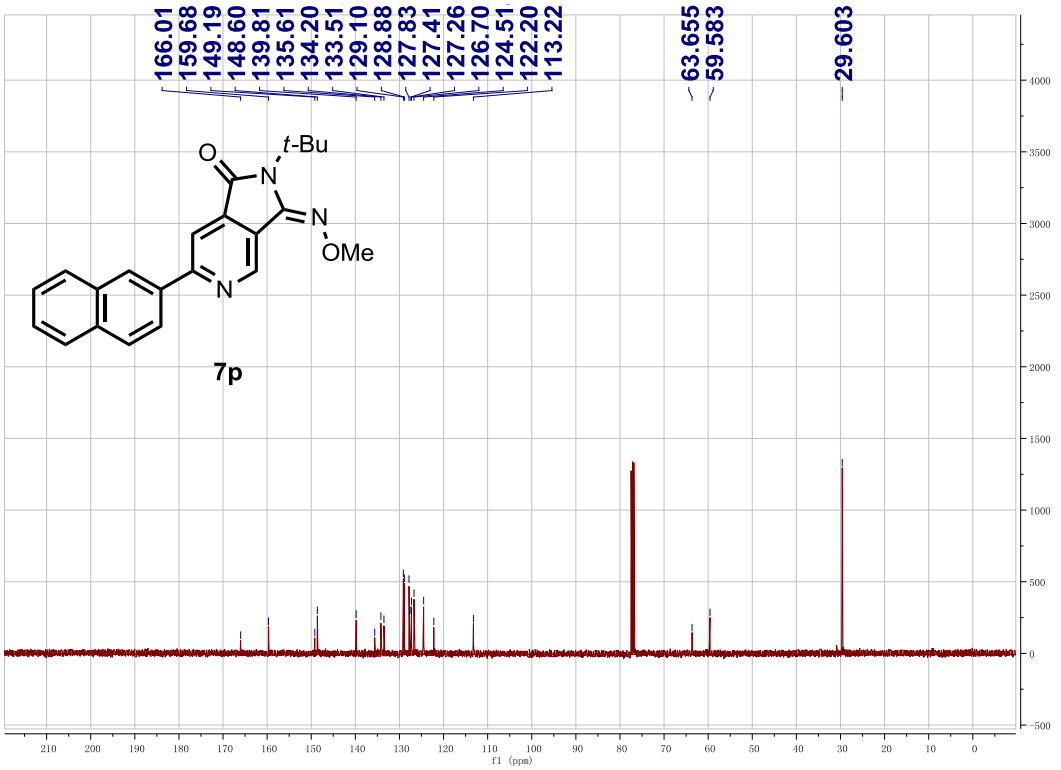

9.464
9.461
8.07
8.04
8.01
8.01
7.02
7.00
4.11
3.87
1.77
t-Bu
O
N
N
OMe
N
MeO
7n
0.81
2.00
1.00
2.02
2.87
3.00
9.17
f1 (ppm)

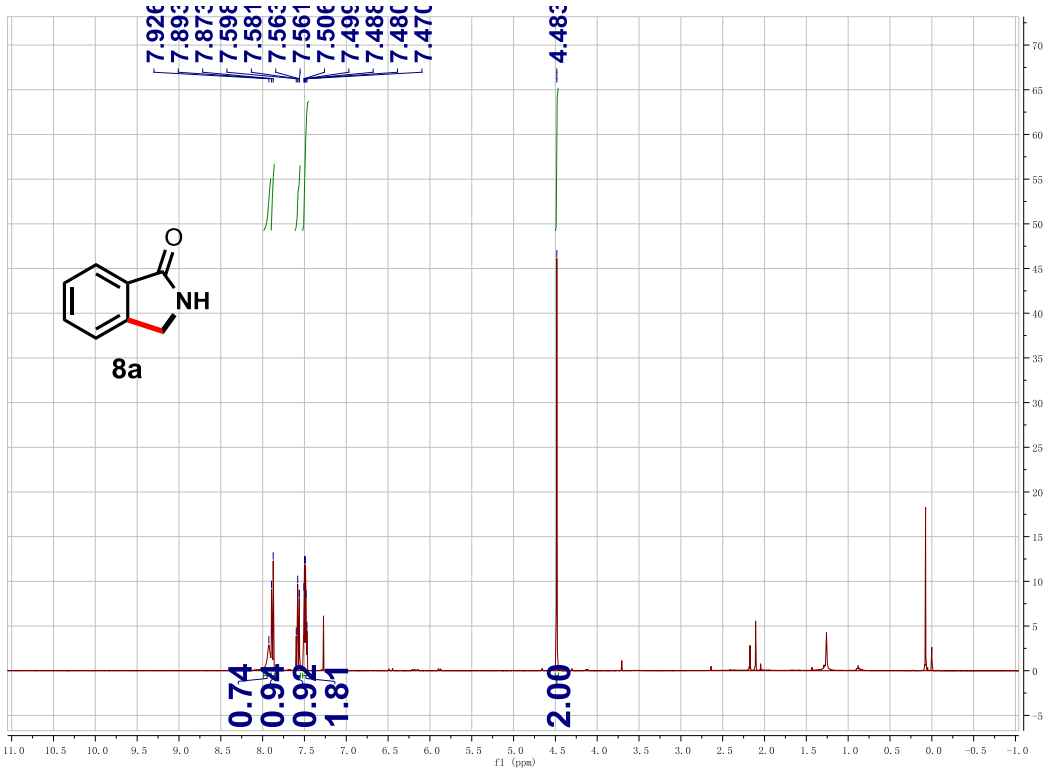

166.13
161.41
159.42
149.27
148.41
139.66
130.96
128.83
121.47
114.45
112.04
63.558
59.476
55.536
29.578
t-Bu
O
N
N
OMe
N
MeO
7n
f1 (ppm)

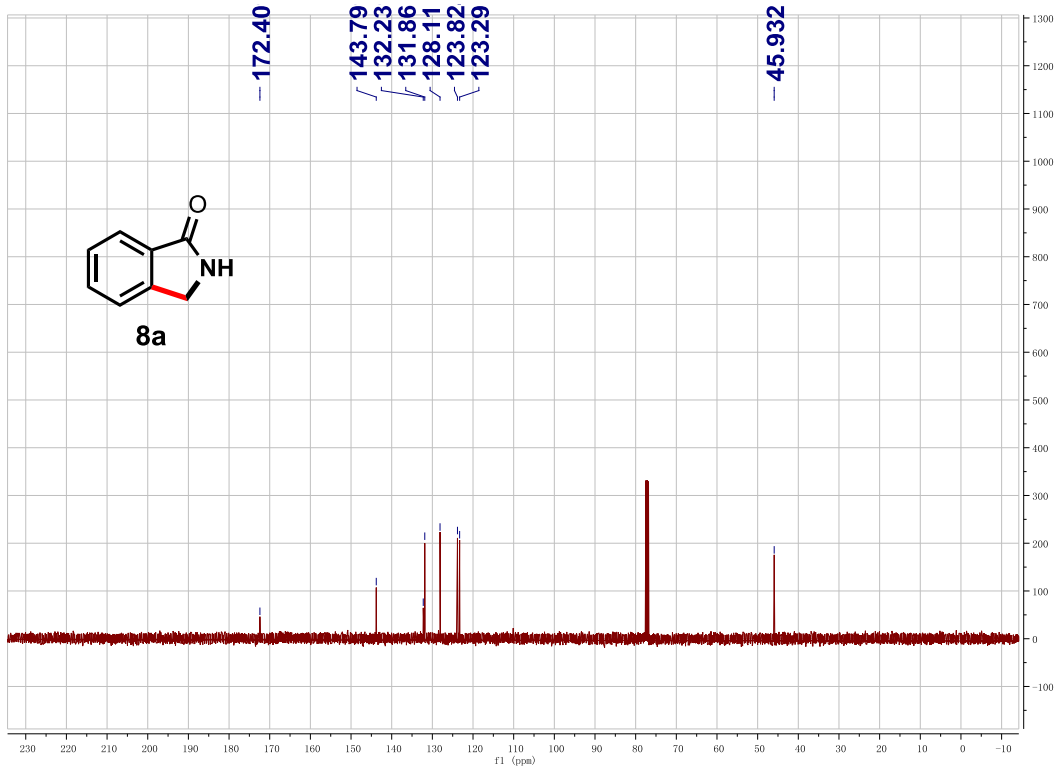

9.494
8.043
8.041
8.020
7.477
7.456
4.132
1.774
0.83
3.05
1.97
3.00
9.21
f1 (ppm)
7o

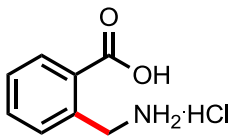

165.58
158.23
148.80
148.34
139.63
136.52
136.16
129.08
128.43
122.17
112.51
63.447
59.390
29.348
f1 (ppm)
7o


9.565
9.562
8.594
8.591
8.228
8.204
8.200
7.977
7.964
7.958
7.868
7.546
7.539
7.537
7.532
7.525
7.521
4.144
1.791
t-Bu
O
N
N
OMe
N
7p
0.90
1.00
1.99
2.07
1.07
2.09
3.00
9.10
f1 (ppm)

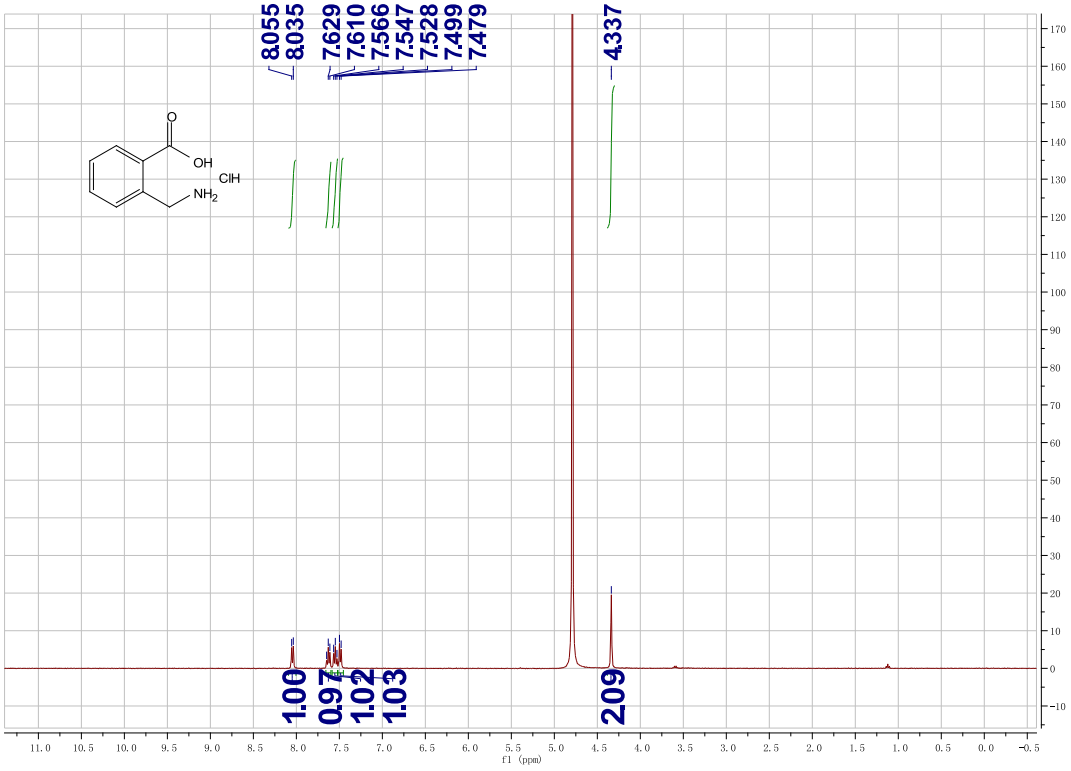

166.01
159.68
149.19
148.60
139.81
135.61
134.20
133.51
129.10
128.88
127.83
127.41
127.26
126.70
124.51
122.20
113.22
63.655
59.583
29.603
t-Bu
O
N
N
OMe
N
7p
f1 (ppm)

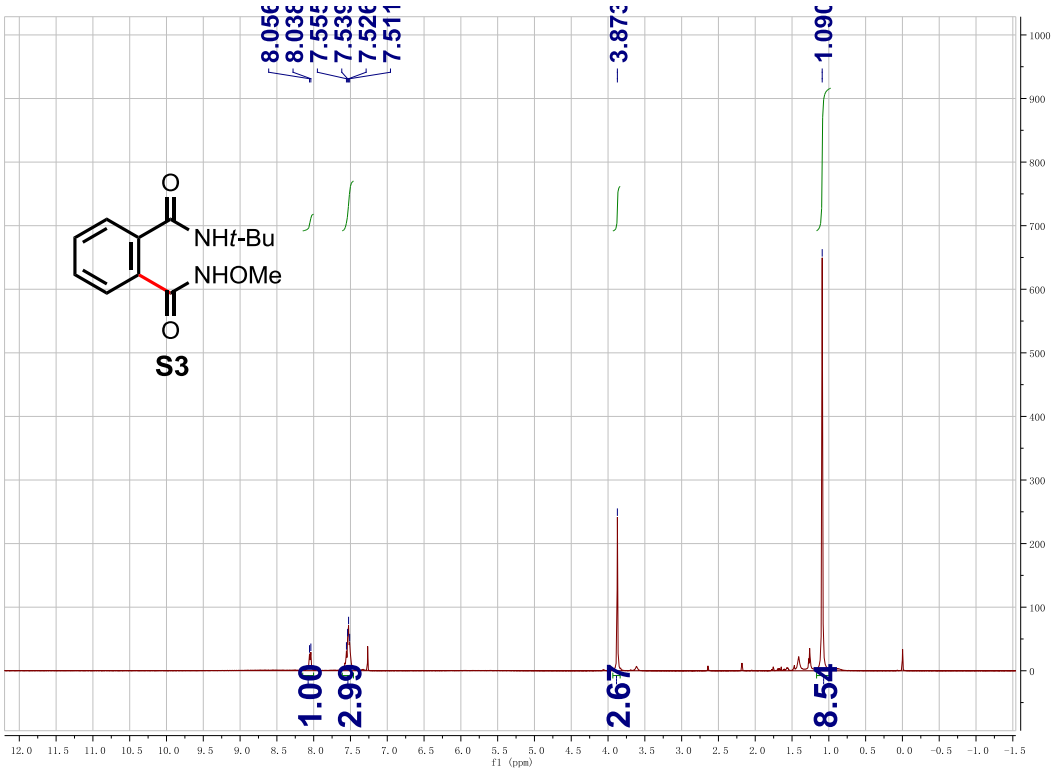

7.926
7.893
7.873
7.598
7.581
7.563
7.561
7.506
7.495
7.488
7.480
7.470
4.483
O
NH
8a
0.74
0.94
0.92
1.81
2.00
f1 (ppm)

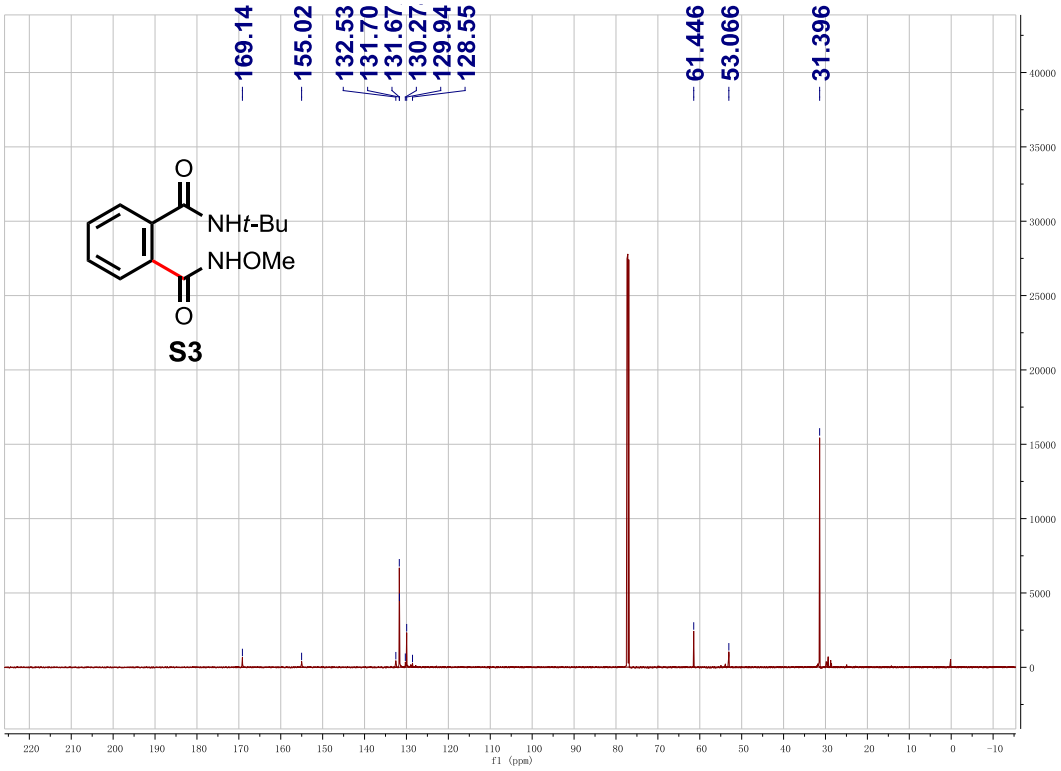

172.40
143.79
132.23
131.86
128.11
123.82
123.29
45.932
O
NH
8a
f1 (ppm)

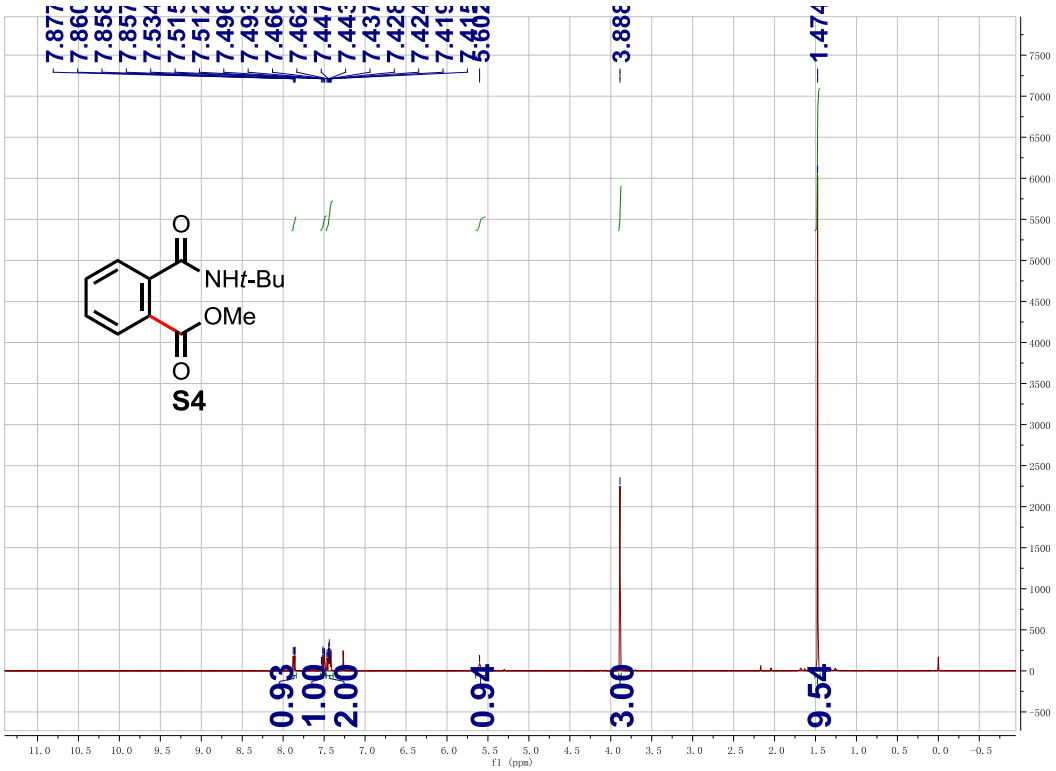

O
OH
NH2·HCl

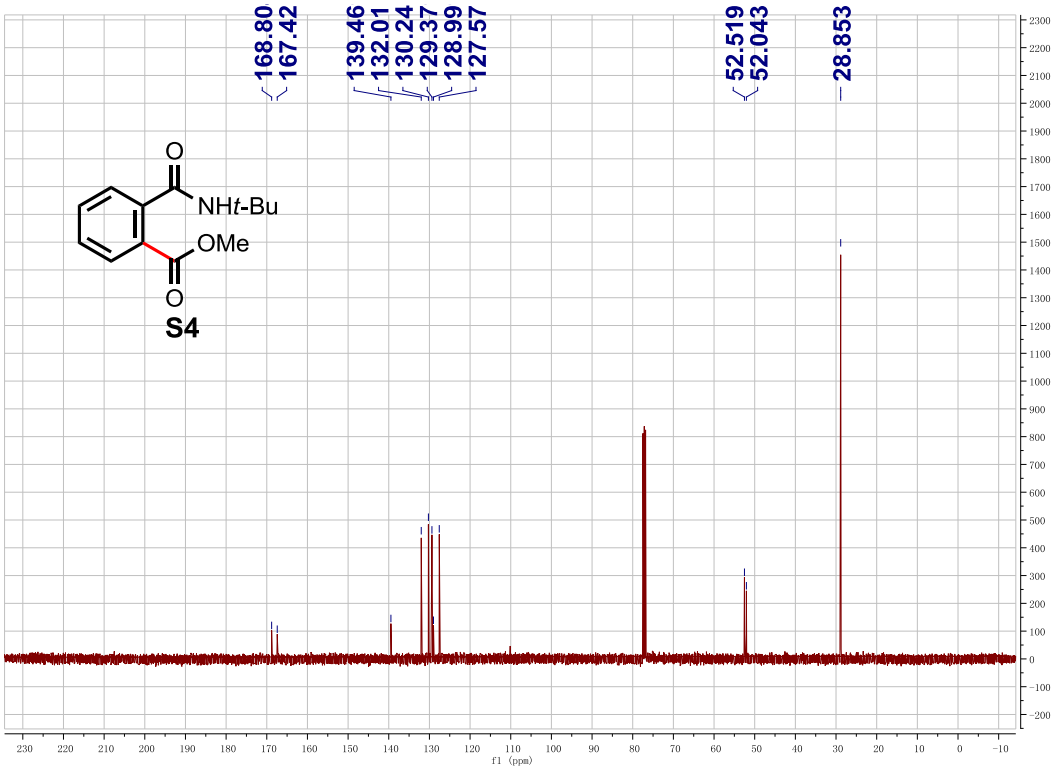
S2

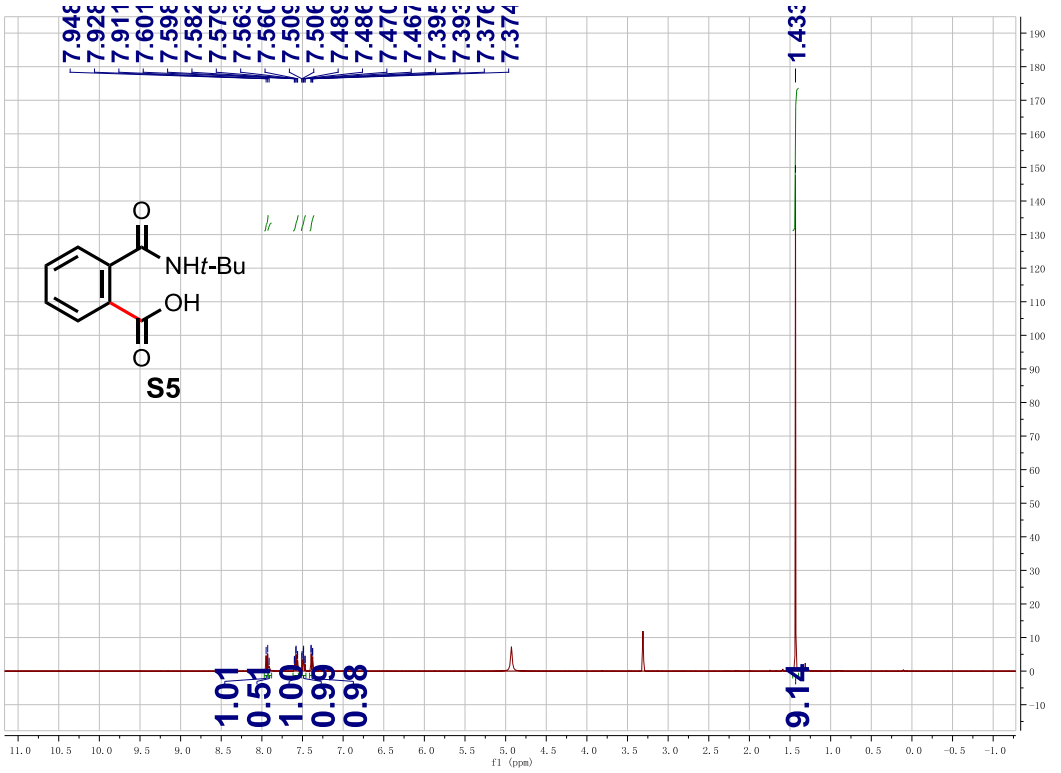

8.055
8.035
7.629
7.610
7.566
7.547
7.528
7.499
7.479
4.337
1.00
0.97
1.02
1.03
2.09
f1 (ppm)

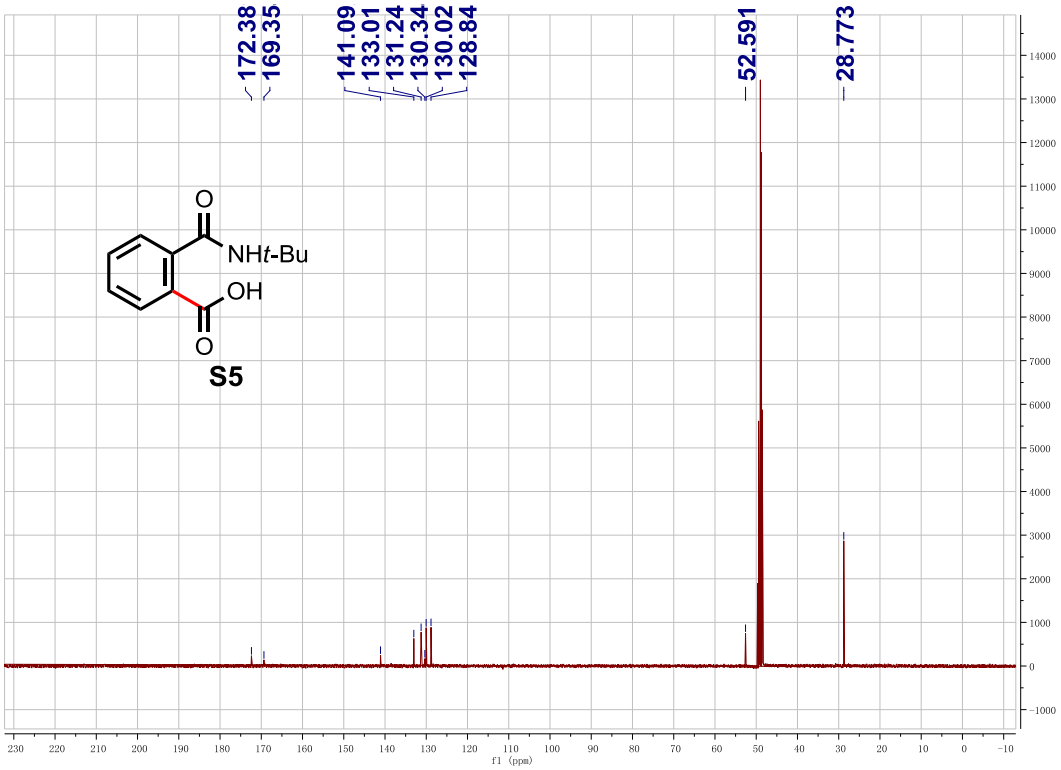

8.05
8.03
7.55
7.53
7.52
7.51
3.87
1.09
O
NHt-Bu
NHOMe
O
S3
1.00
2.99
2.67
8.54
f1 (ppm)

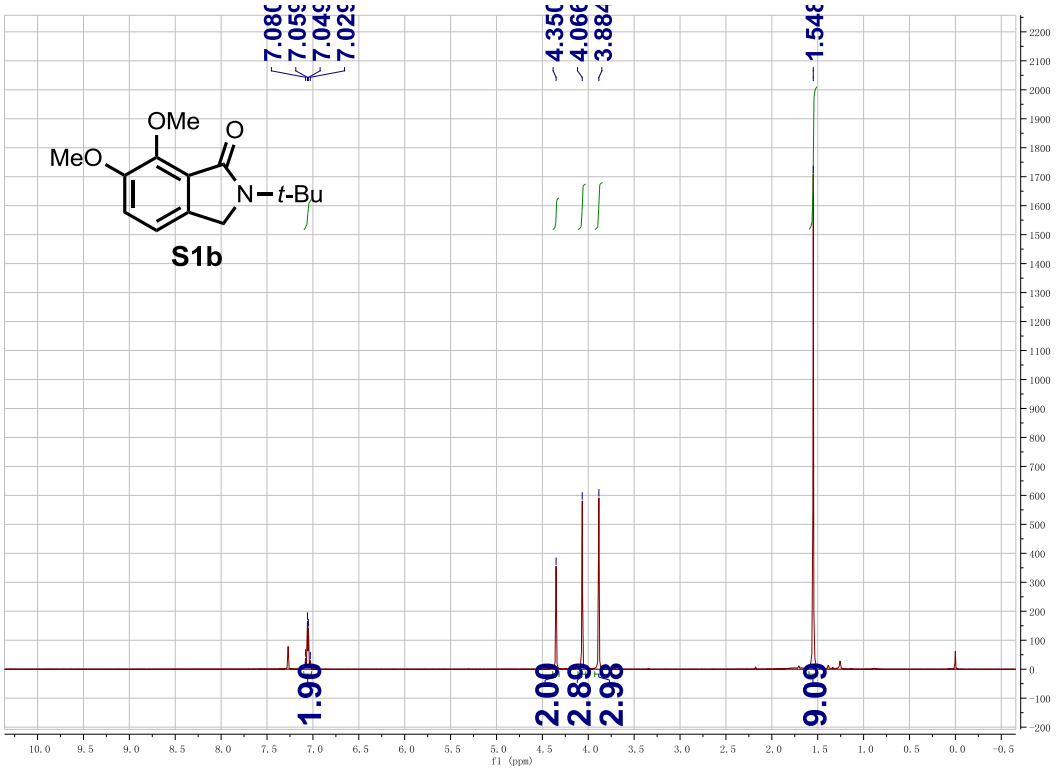

169.14
155.02
132.53
131.70
131.67
130.27
129.94
128.55
61.446
53.066
31.396
O
NHt-Bu
NHOMe
O
S3
f1 (ppm)

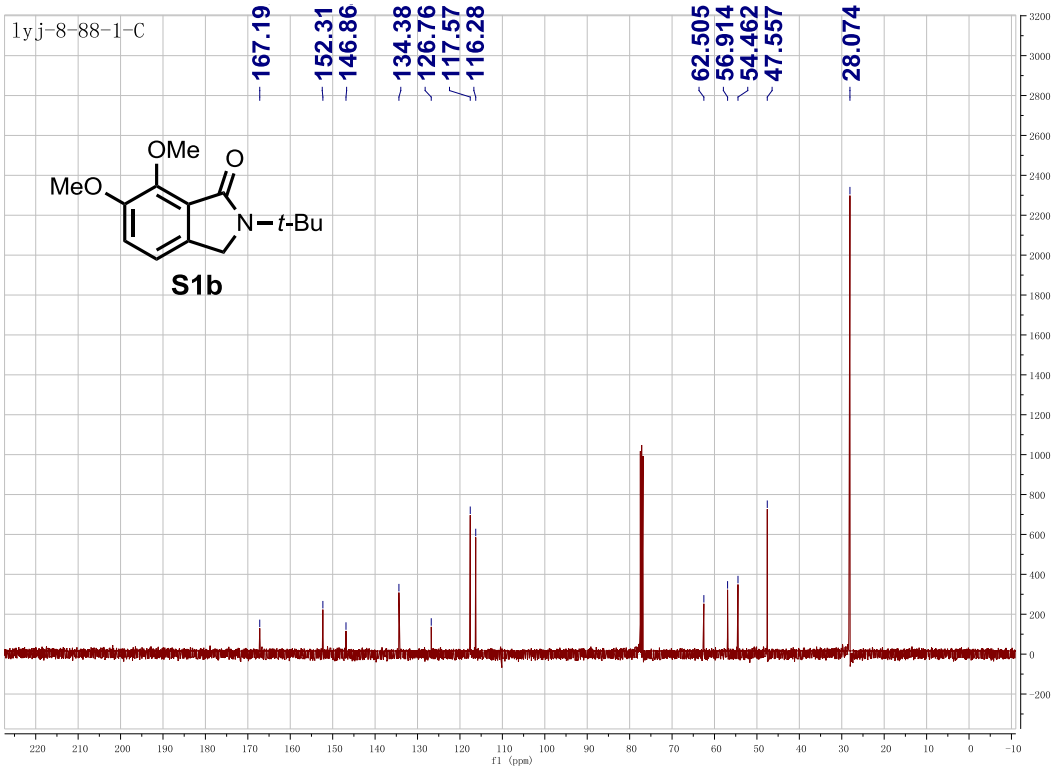

7.877
7.860
7.858
7.857
7.534
7.515
7.512
7.496
7.493
7.466
7.462
7.447
7.443
7.437
7.428
7.424
7.415
7.402
3.888
1.474
NHt-Bu
OMe
O
O
S4
0.93
1.00
2.00
0.94
3.00
9.54
f1 (ppm)

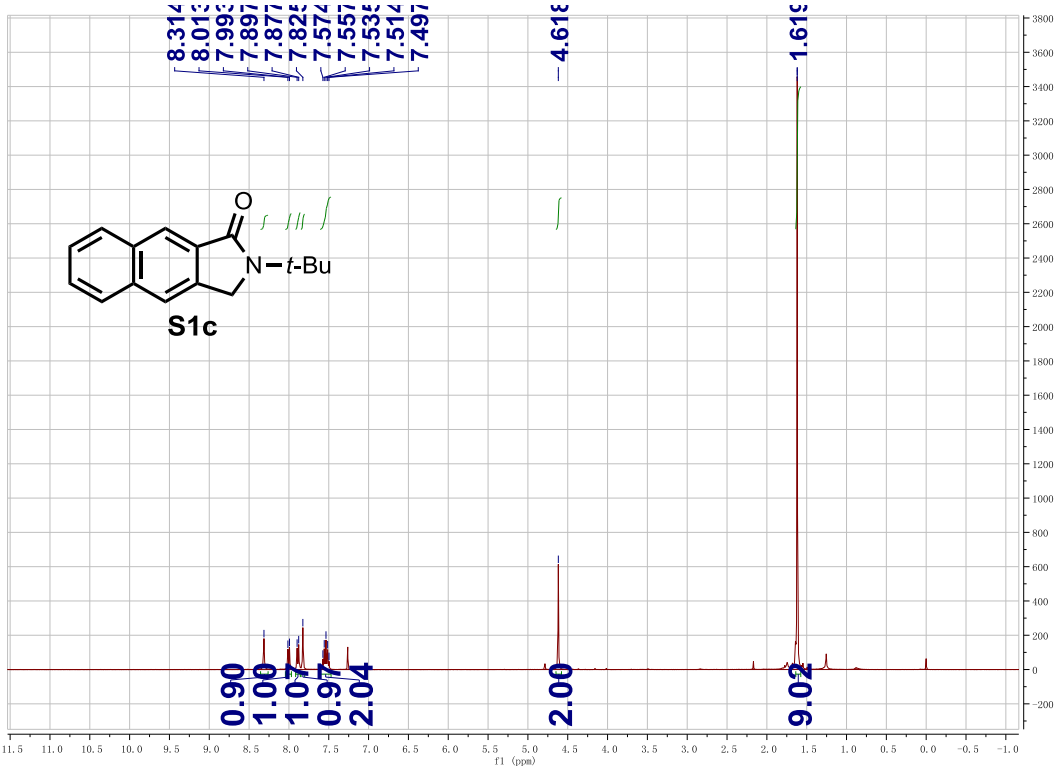

168.80
167.42
139.46
132.01
130.24
129.37
128.99
127.57
52.519
52.043
28.853
NHt-Bu
OMe
O
O
S4
f1 (ppm)

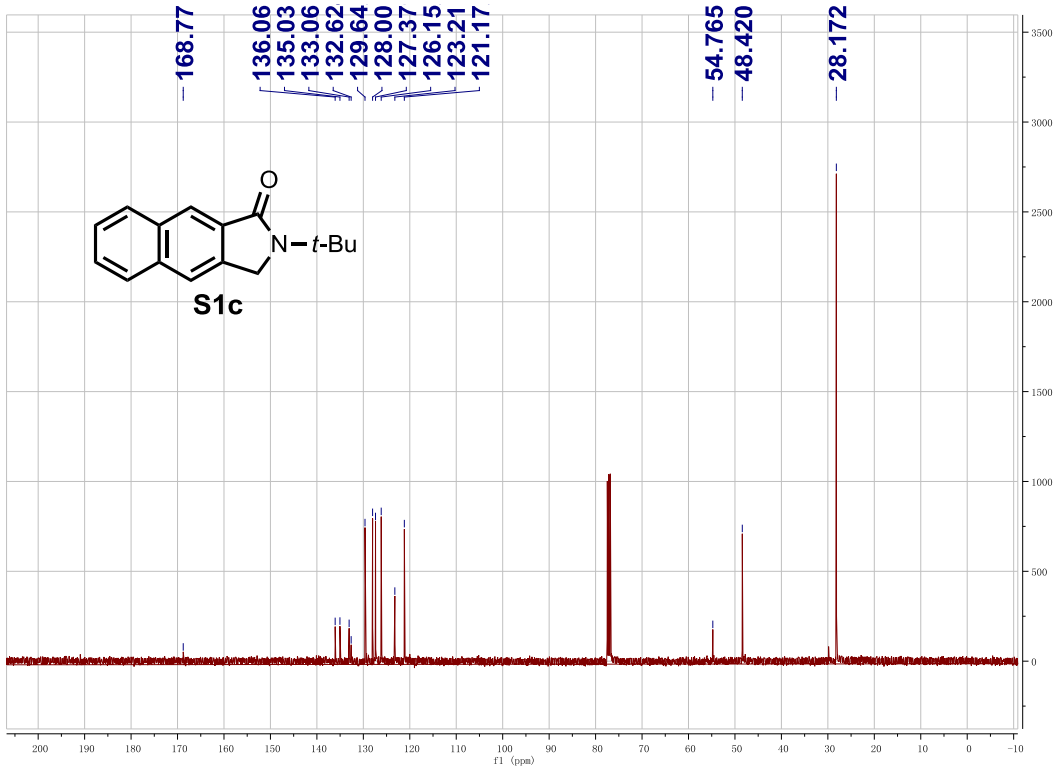

7.946
7.928
7.911
7.601
7.598
7.582
7.575
7.563
7.560
7.505
7.506
7.485
7.486
7.470
7.467
7.395
7.393
7.376
7.374
1.433
O
NHt-Bu
OH
O
S5
1.01
0.51
1.00
0.99
0.98
9.14
f1 (ppm)

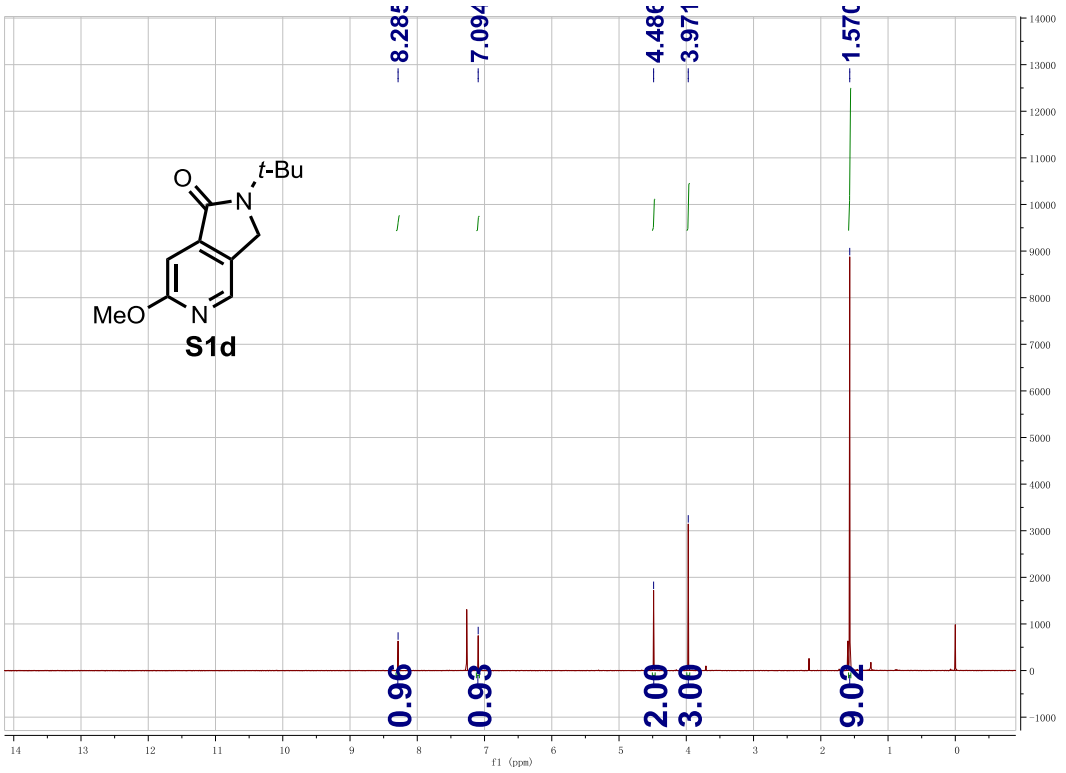

172.38
169.35
141.09
133.01
131.24
130.34
130.02
128.84
52.591
28.773
O
NHt-Bu
OH
O
S5
f1 (ppm)

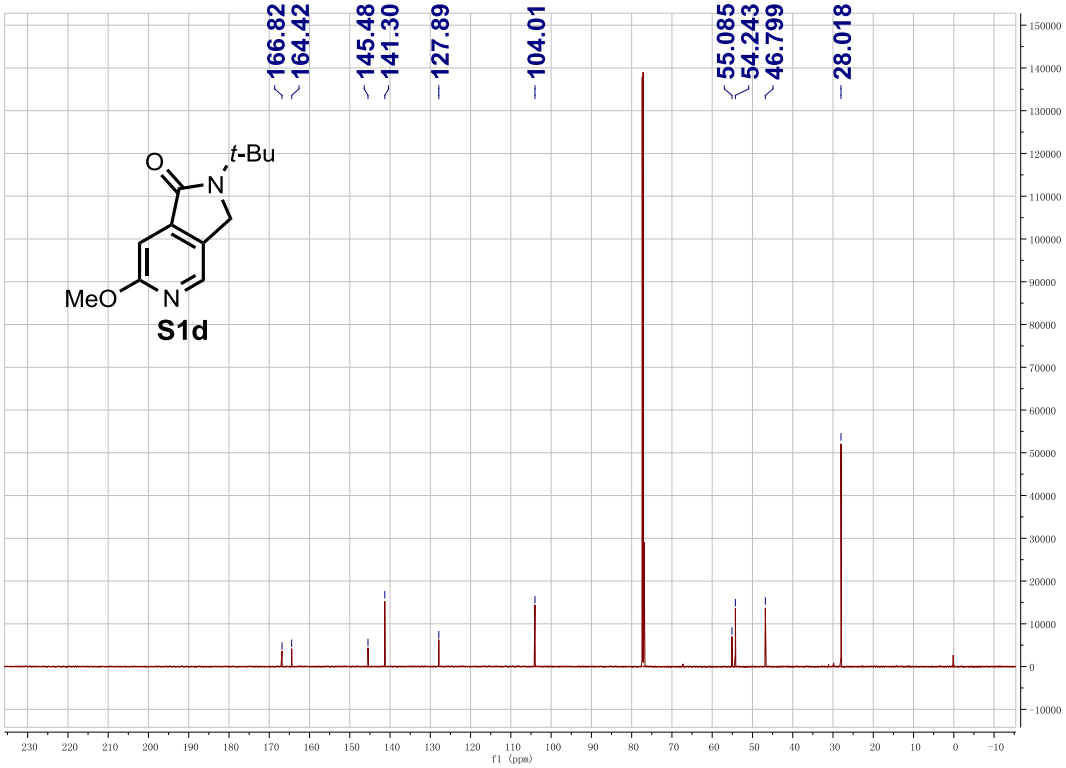

7.08
7.05
7.04
7.02
4.35
4.06
3.88
1.54
OMe
O
MeO
N–t-Bu
S1b
1.90
2.00
2.89
2.98
9.09
f1 (ppm)

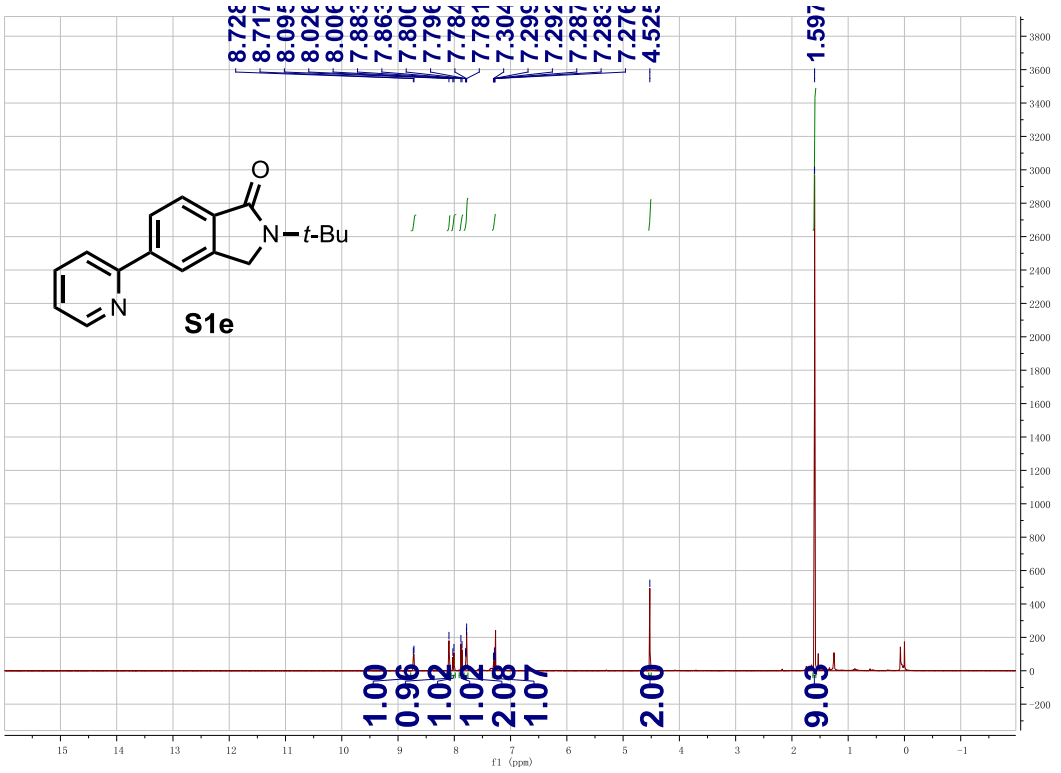

lyj-8-88-1-C
167.19
152.31
146.86
134.38
126.76
117.57
116.28
62.505
56.914
54.462
47.557
28.074
OMe
O
MeO
N–t-Bu
S1b
f1 (ppm)

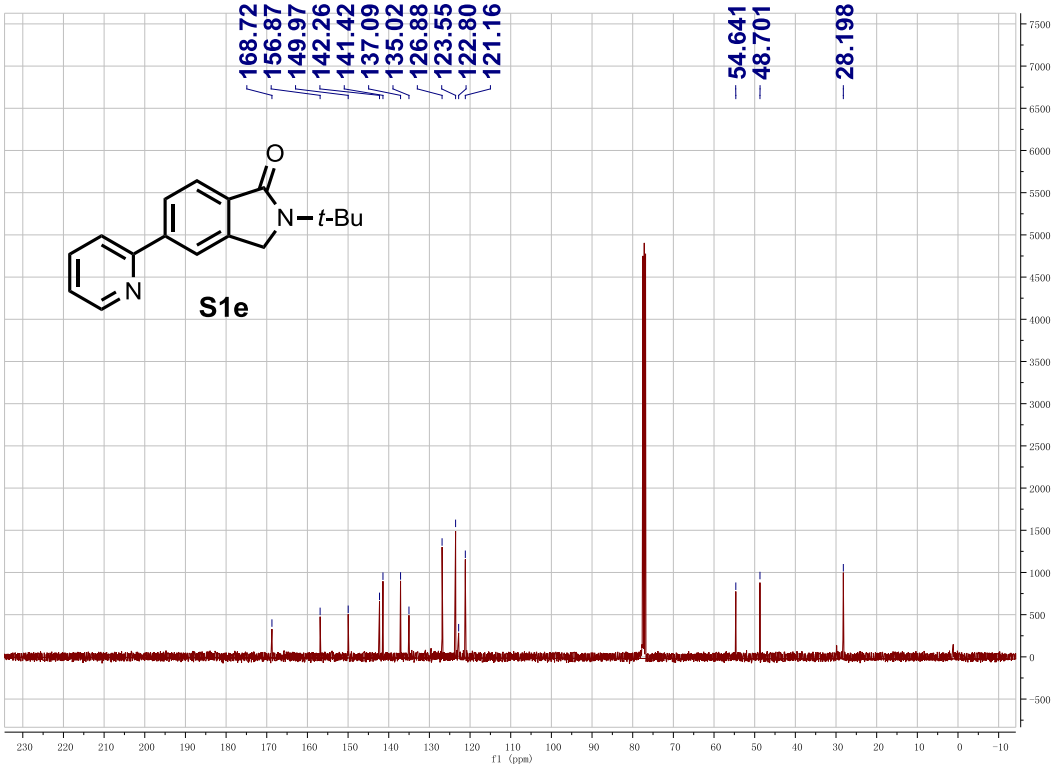

8.314
8.013
7.995
7.891
7.877
7.825
7.574
7.551
7.535
7.514
7.497
4.615
1.615
O
N−t-Bu
S1c
0.90
1.00
1.07
0.97
2.04
2.00
9.02
f1 (ppm)

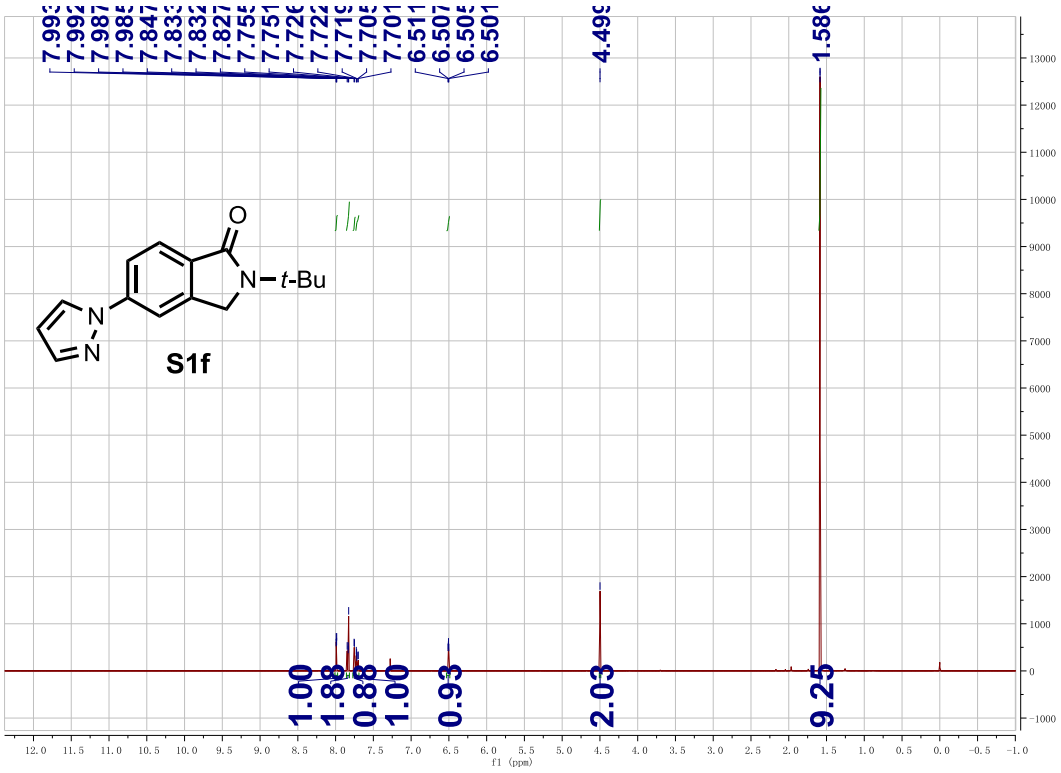

168.77
136.06
135.03
133.06
132.62
129.64
128.00
127.37
126.15
123.21
121.17
54.765
48.420
28.172
O
N−t-Bu
S1c
f1 (ppm)

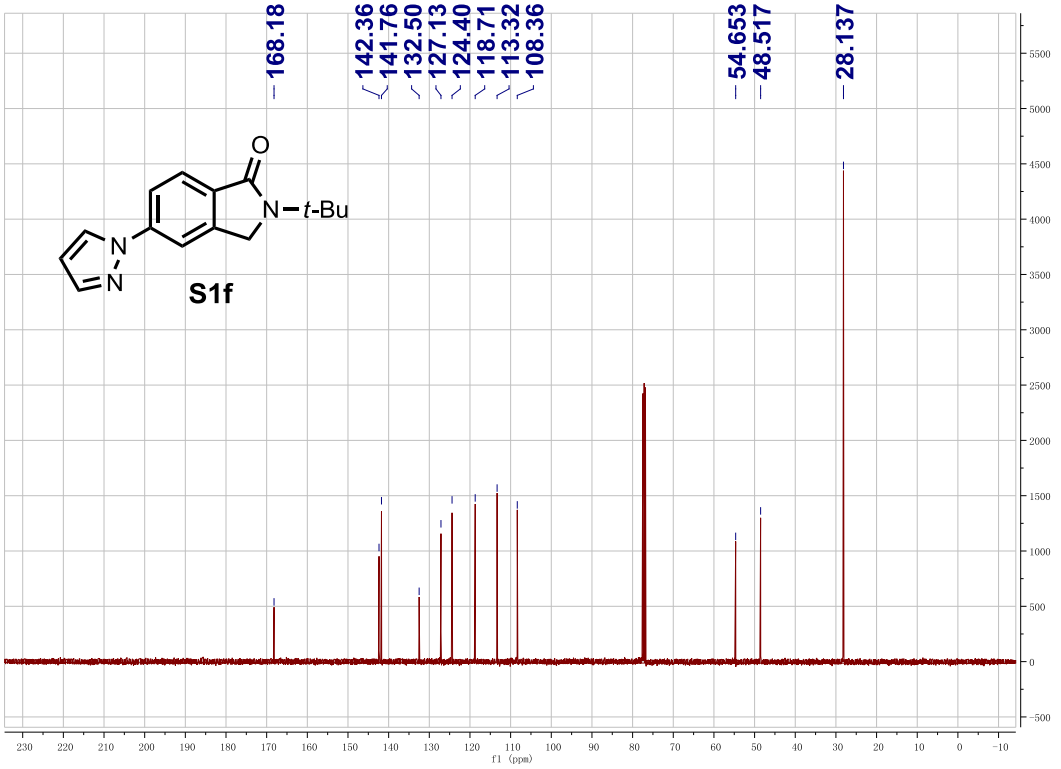

8.28
7.09
4.48
3.97
1.57
0.96
0.93
2.00
3.00
9.02
O
N
t-Bu
MeO
N
S1d
f1 (ppm)

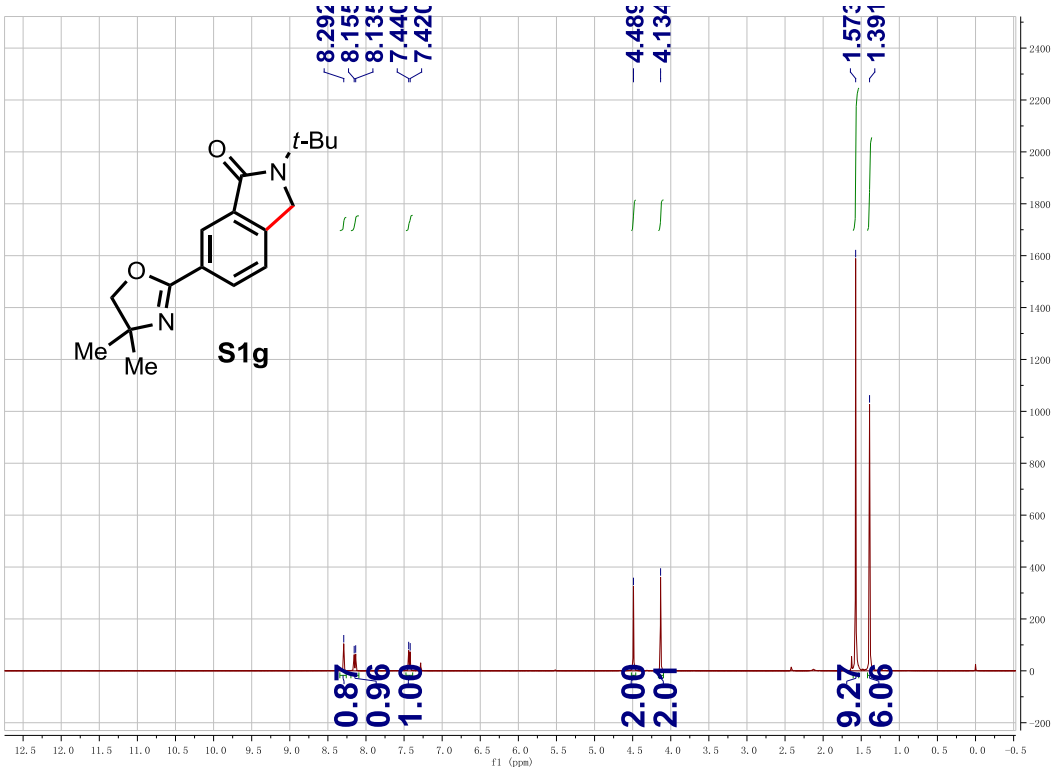

166.82
164.42
145.48
141.30
127.89
104.01
55.085
54.243
46.799
28.018
O
N
t-Bu
MeO
N
S1d
f1 (ppm)

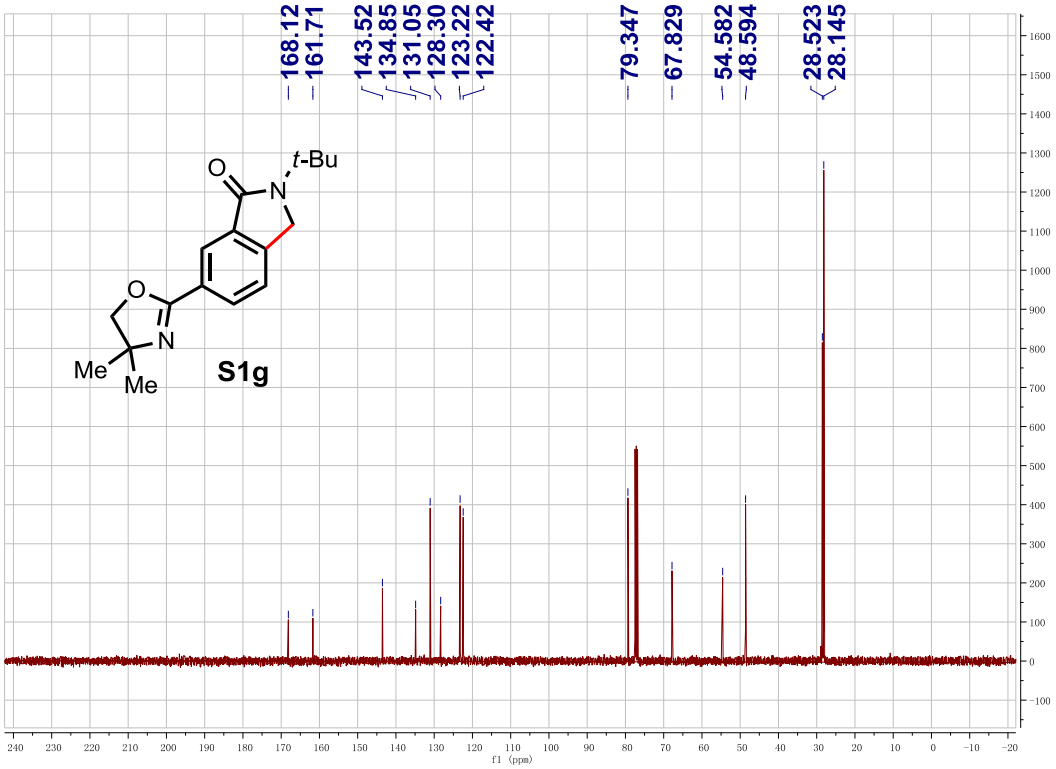

8.728
8.717
8.095
8.026
8.006
7.883
7.863
7.800
7.796
7.784
7.781
7.304
7.295
7.292
7.287
7.283
7.276
4.525
1.597
O
N–t-Bu
N
S1e
1.00
0.96
1.02
1.02
2.08
1.07
2.00
9.03
f1 (ppm)

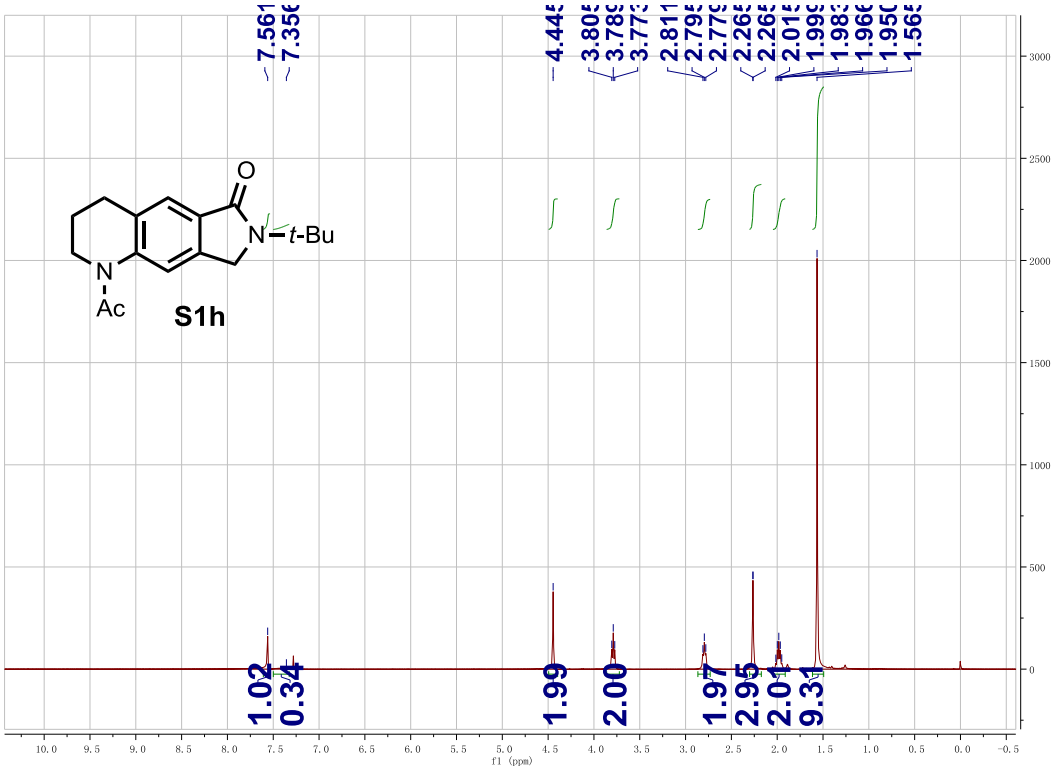

168.72
156.87
149.97
142.26
141.42
137.09
135.02
126.88
123.55
122.80
121.16
54.641
48.701
28.198
O
N–t-Bu
N
S1e
f1 (ppm)

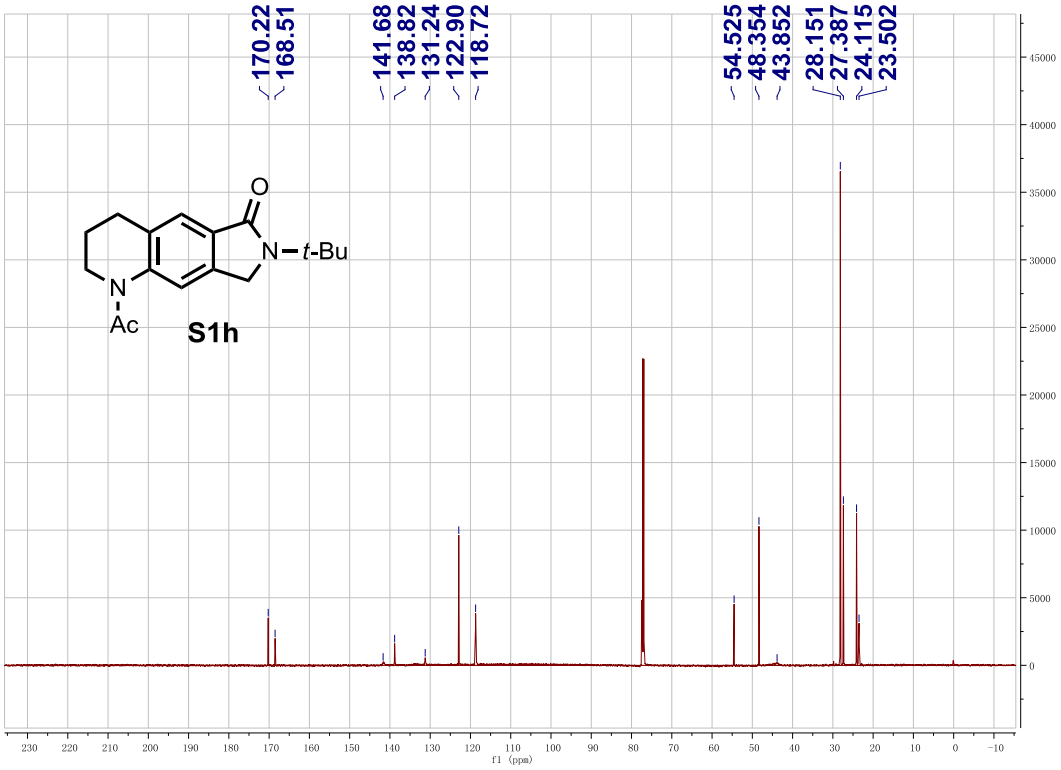

7.995
7.992
7.987
7.985
7.847
7.833
7.832
7.827
7.755
7.751
7.726
7.722
7.719
7.705
7.701
6.511
6.507
6.505
6.501
4.495
1.586
O
N−t-Bu
N
N
S1f
1.00
1.88
0.88
1.00
0.93
2.03
9.25
f1 (ppm)

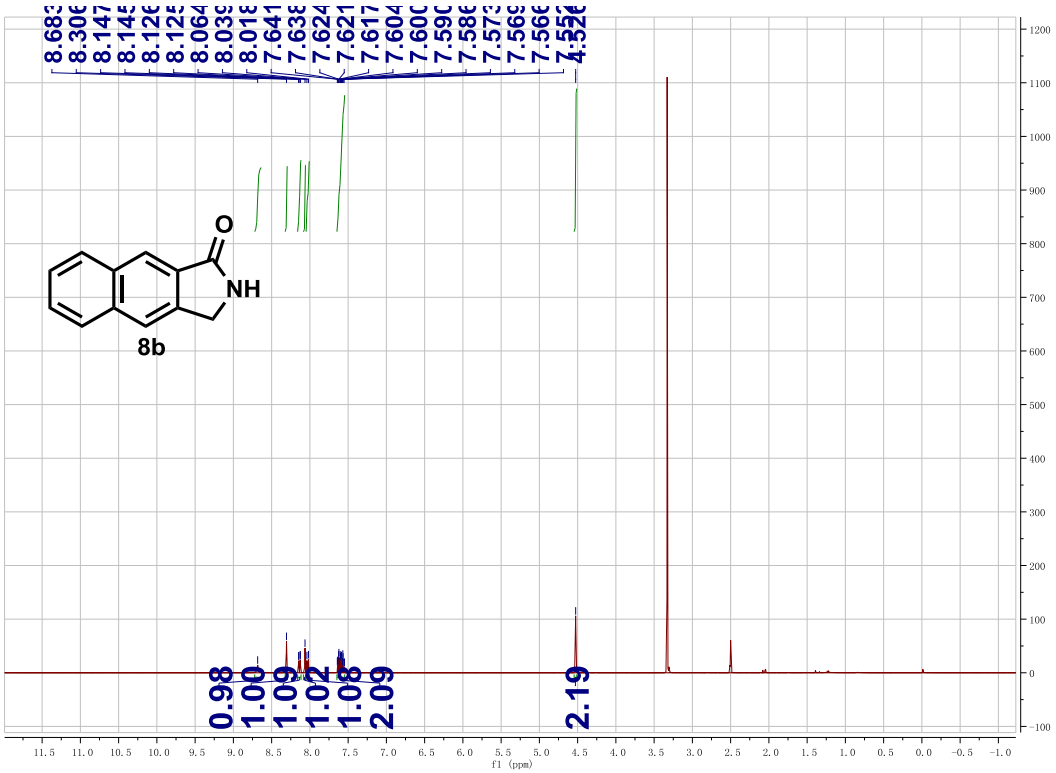

168.18
142.36
141.76
132.50
127.13
124.40
118.71
113.32
108.36
54.653
48.517
28.137
O
N−t-Bu
N
N
S1f
f1 (ppm)

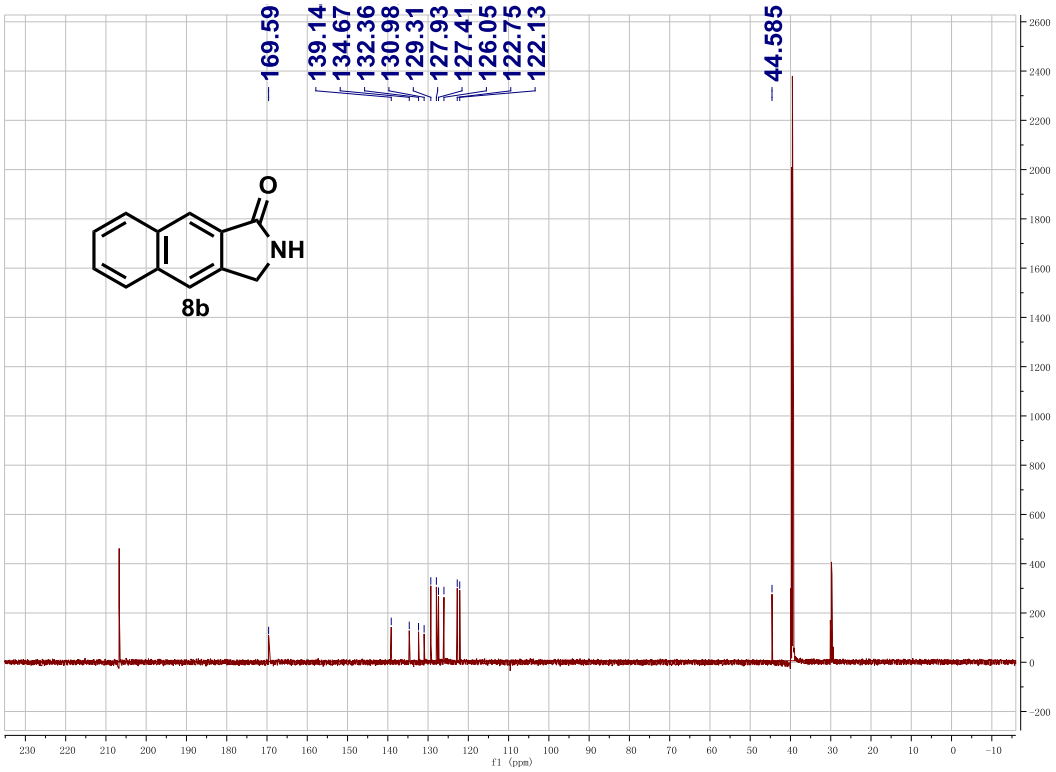

8.292
8.155
8.135
7.440
7.420
4.485
4.134
1.573
1.391
t-Bu
O
N
O
N
Me
Me
S1g
0.87
0.96
1.00
2.00
2.01
9.27
6.06
f1 (ppm)

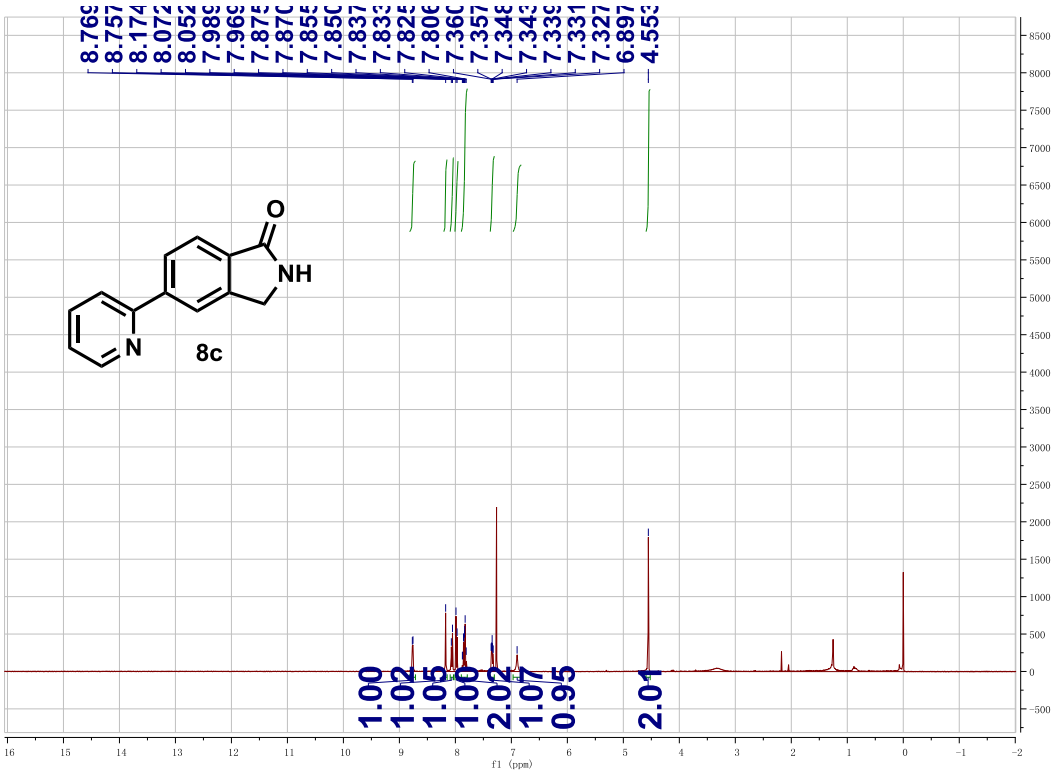

168.12
161.71
143.52
134.85
131.05
128.30
123.22
122.42
79.347
67.829
54.582
48.594
28.523
28.145
t-Bu
O
N
O
N
Me
Me
S1g
f1 (ppm)

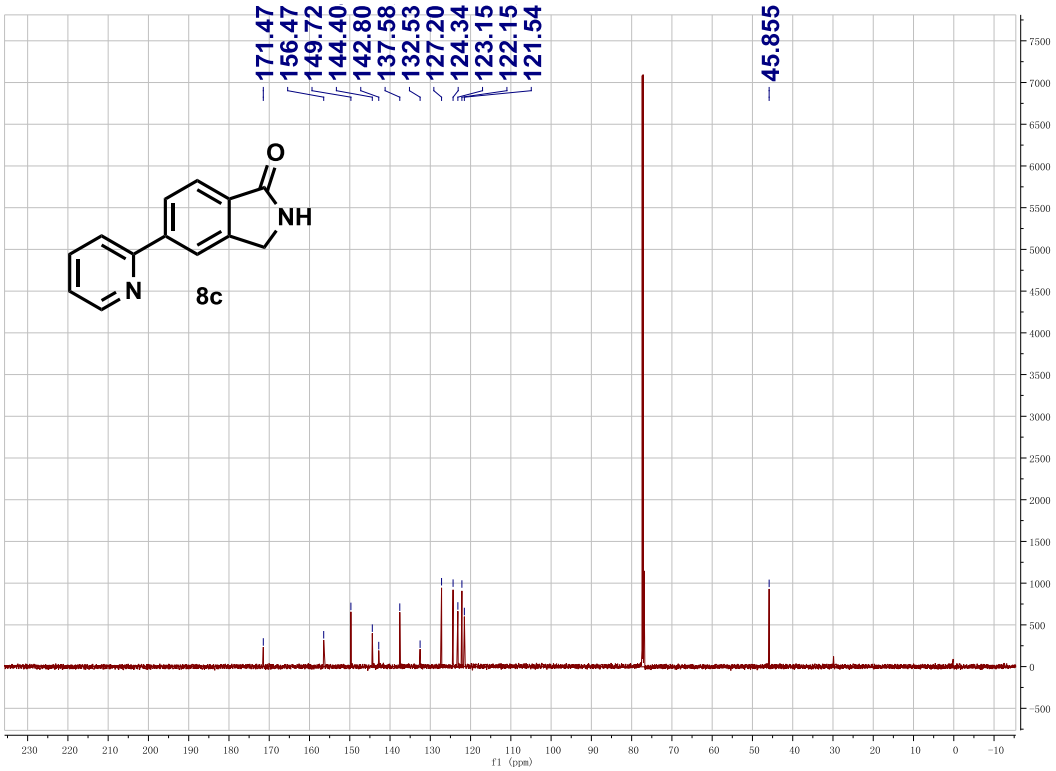

7.561
7.356
4.445
3.805
3.785
3.772
2.811
2.795
2.775
2.265
2.262
2.015
1.999
1.983
1.966
1.950
1.562
O
N–t-Bu
N
Ac
S1h
1.02
0.34
1.99
2.00
1.97
2.95
2.01
9.31
f1 (ppm)

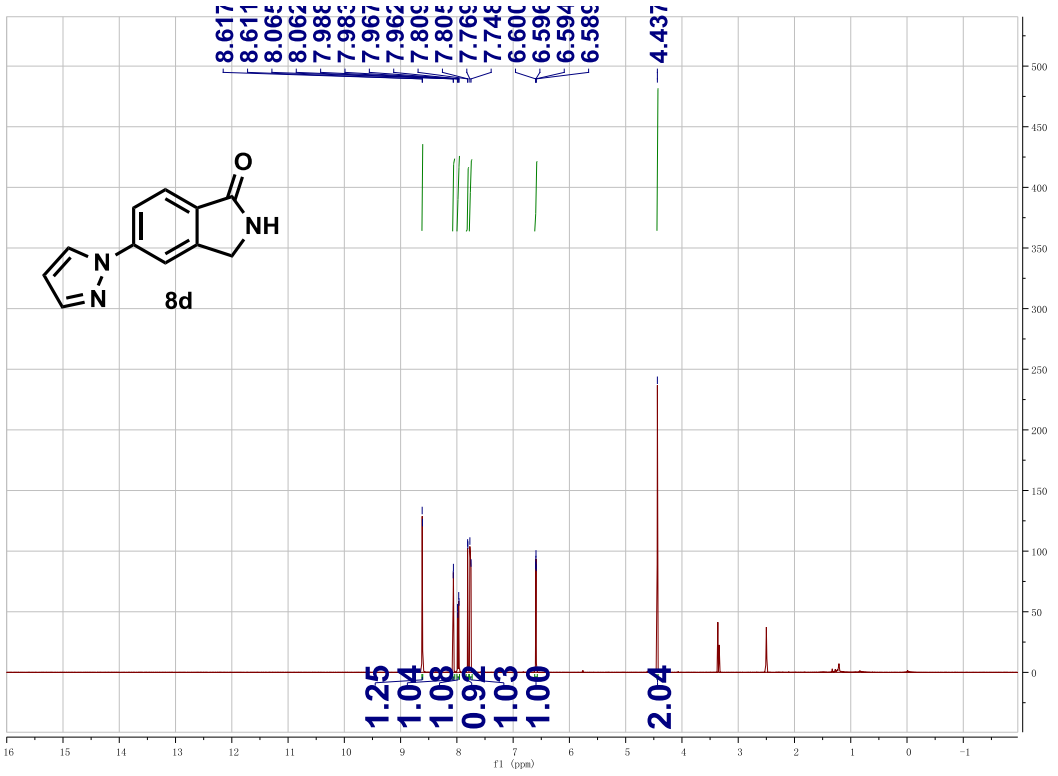

170.22
168.51
141.68
138.82
131.24
122.90
118.72
54.525
48.354
43.852
28.151
27.387
24.115
23.502
O
N–t-Bu
N
Ac
S1h
f1 (ppm)

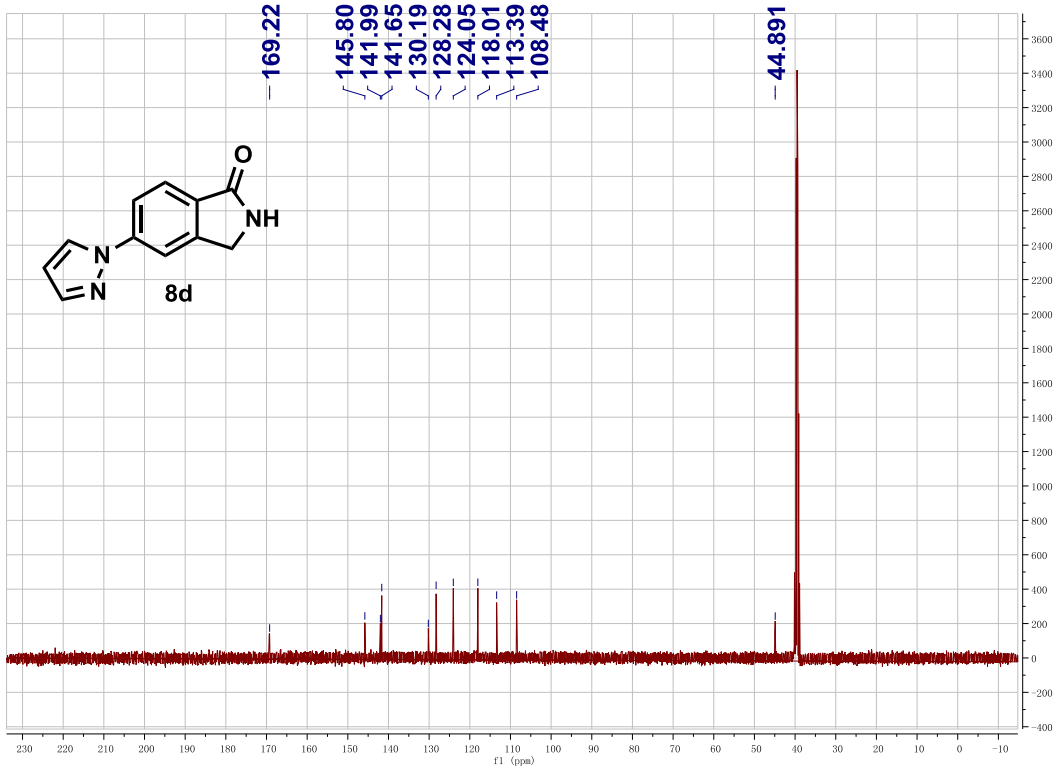

8.683
8.308
8.147
8.145
8.126
8.125
8.064
8.039
8.015
7.641
7.638
7.624
7.621
7.617
7.604
7.600
7.590
7.588
7.573
7.569
7.566
7.552
4.524
O
NH
8b
0.98
1.00
1.09
1.02
1.08
2.09
2.19
f1 (ppm)

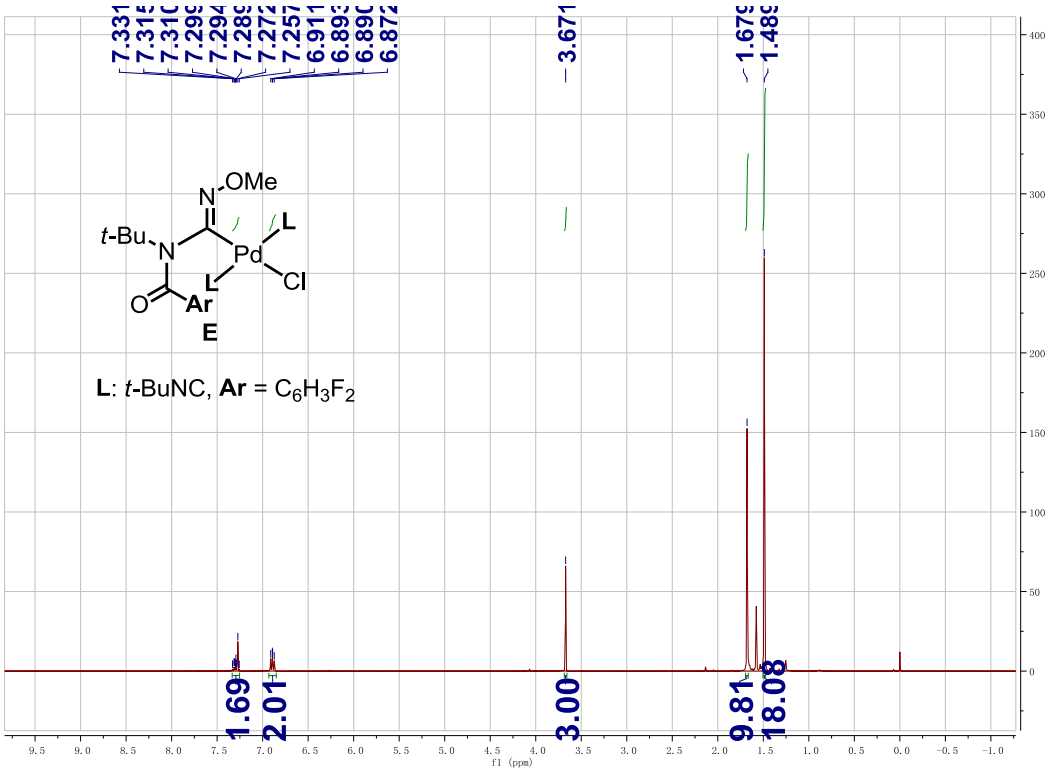

169.59
139.14
134.67
132.36
130.98
129.31
127.93
127.41
126.05
122.75
122.13
44.585
O
NH
8b
f1 (ppm)

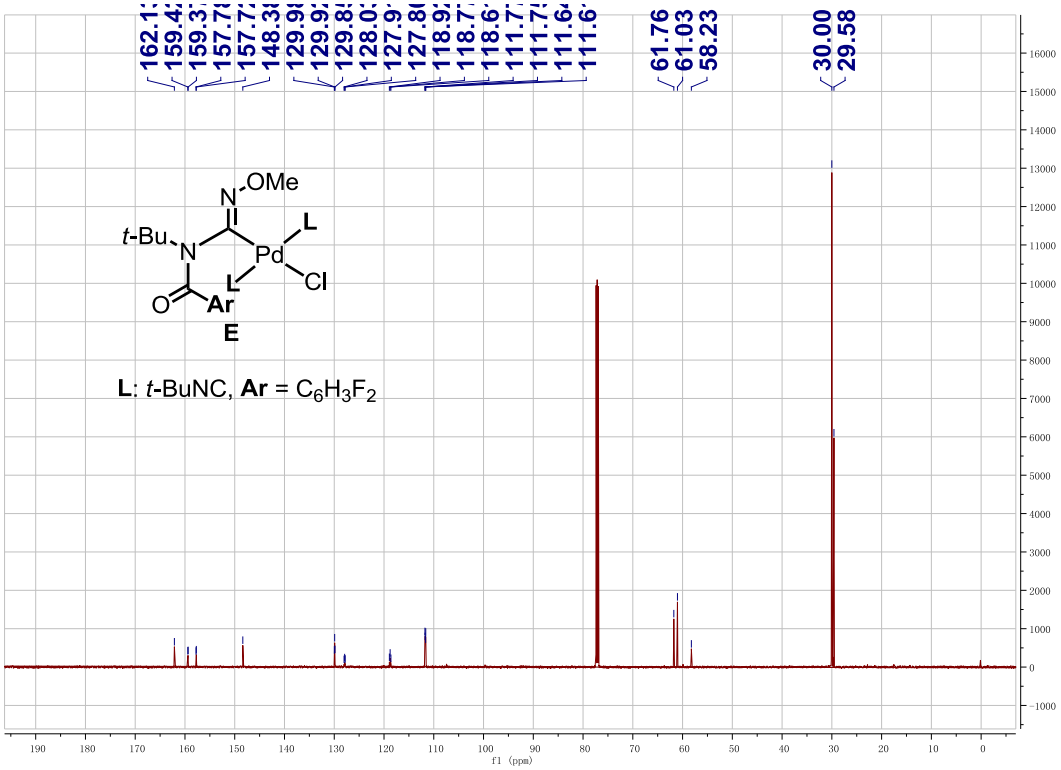

8.769
8.757
8.174
8.072
8.052
7.983
7.965
7.875
7.870
7.855
7.850
7.837
7.833
7.825
7.806
7.360
7.357
7.348
7.343
7.339
7.331
7.327
6.897
4.553
O
NH
N
8c
1.00
1.02
1.05
1.00
2.02
1.07
0.95
2.01
f1 (ppm)

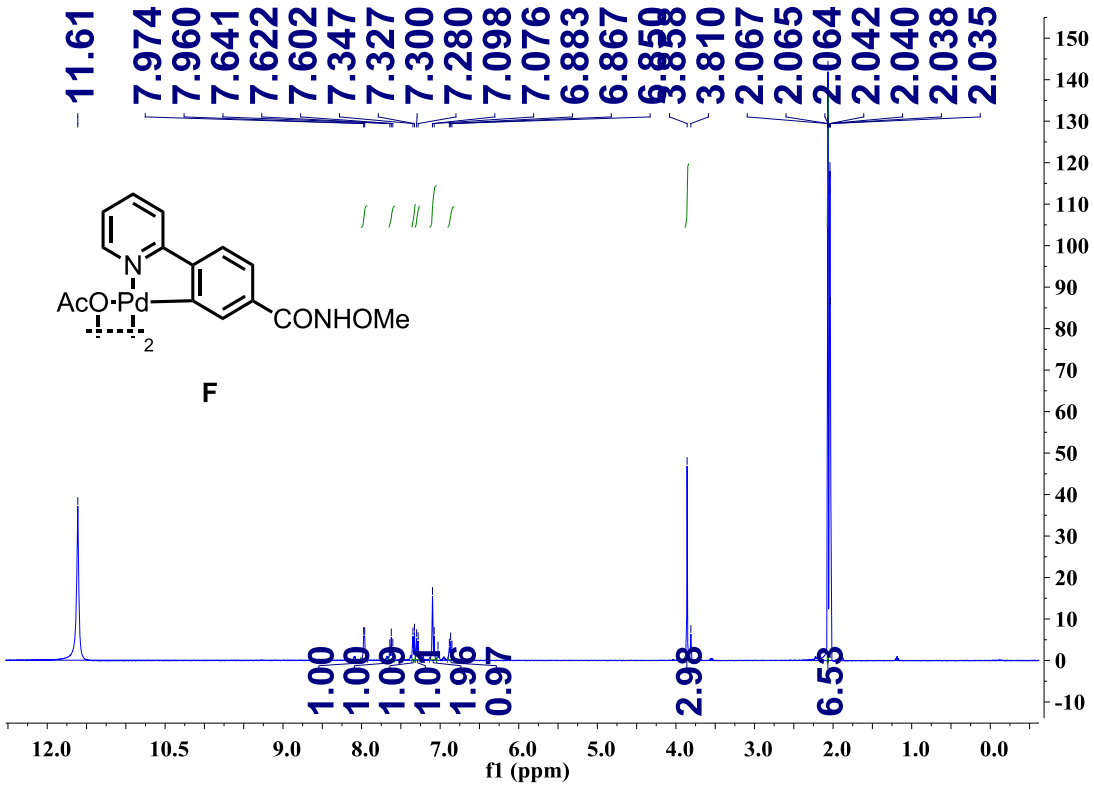

171.47
156.47
149.72
144.40
142.80
137.58
132.53
127.20
124.34
123.15
122.15
121.54
45.855
O
NH
N
8c
f1 (ppm)

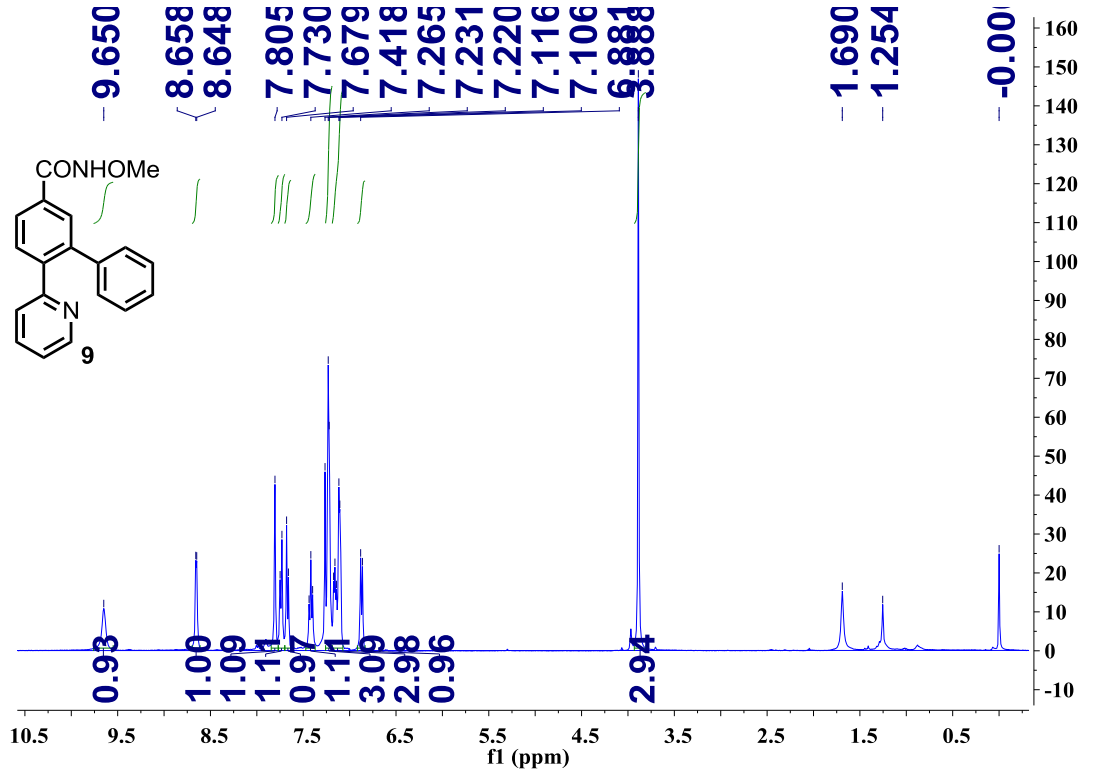

8.617
8.611
4.437
O
NH
N
N
8d
1.25
1.04
1.08
0.92
1.03
1.00
2.04
f1 (ppm)

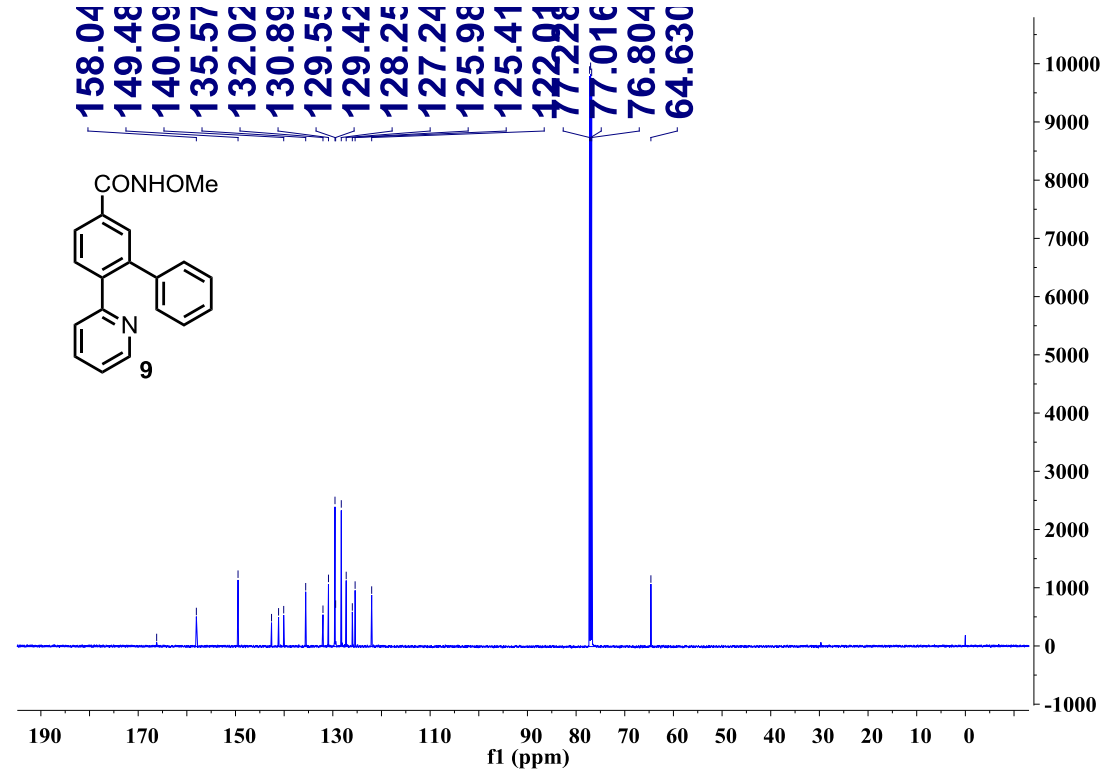

169.22
145.80
141.99
141.65
130.19
128.28
124.05
118.01
113.39
108.48
44.891
O
NH
N
N
8d
f1 (ppm)

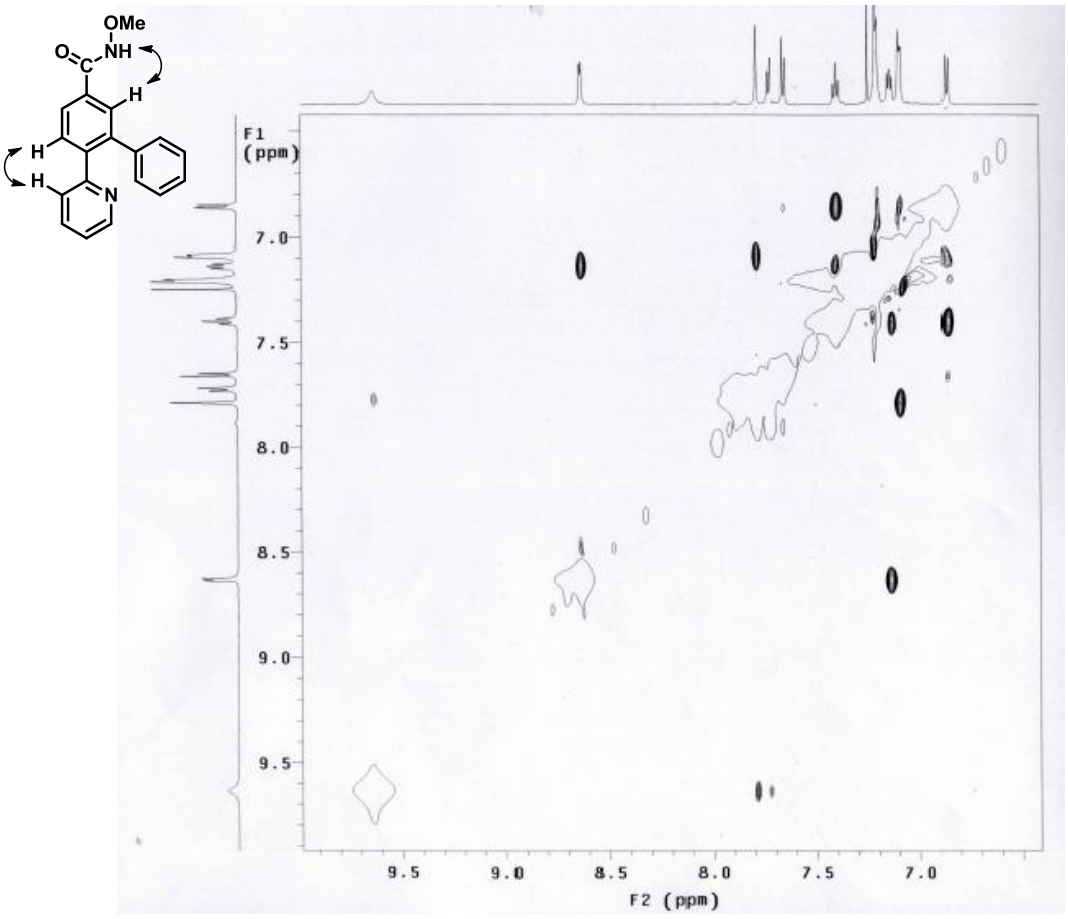

7.331
7.315
7.310
7.295
7.294
7.285
7.272
7.257
6.911
6.893
6.890
6.872
3.671
1.675
1.485
OMe
N
t-Bu
N
Pd
L
L
Cl
O
Ar
E
L: t-BuNC, Ar = C6H3F2
1.69
2.01
3.00
9.81
18.08
f1 (ppm)


162.1
159.4
159.3
157.7
157.7
148.3
129.9
129.9
129.8
128.0
127.9
127.8
118.9
118.7
118.6
111.7
111.7
111.6
111.6
61.76
61.03
58.23
30.00
29.58
OMe
N
t-Bu
N
Pd
L
L
Cl
O
Ar
E
L: t-BuNC, Ar = C6H3F2
f1 (ppm)


11.61
7.974
7.960
7.641
7.622
7.602
7.347
7.327
7.300
7.280
7.098
7.076
6.883
6.867
3.858
3.810
2.067
2.065
2.064
2.042
2.040
2.038
2.035
AcO-Pd
2
CONHOMe
F
1.00
1.00
1.09
1.04
1.96
0.97
2.98
6.53
f1 (ppm)

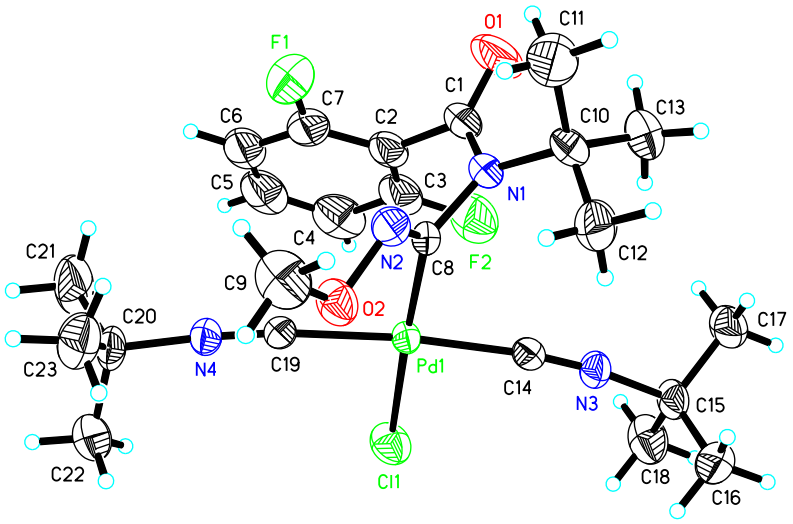

9.650
8.658
8.648
7.805
7.730
7.679
7.418
7.265
7.231
7.220
7.116
7.106
1.690
1.254
-0.00
CONHOMe
N
9
0.93
1.00
1.09
1.11
0.97
1.11
3.09
2.98
0.96
2.94
f1 (ppm)


158.04
149.48
140.09
135.57
132.02
130.89
129.55
129.42
128.25
127.24
125.98
125.41
77.016
76.804
64.630
CONHOMe
N
9
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OMe
NH
O
C
H
H
H
N
F1
(ppm)
7.0
7.5
8.0
8.5
9.0
9.5
9.5
9.0
8.5
8.0
7.5
7.0
F2 (ppm)

6. X-ray Crystallographic Data of Compounds E, F, 3a, 5i

6.1 X-ray Crystallographic Data of Compound E



E

Table 1. Crystal data and structure refinement for cd213256.

Identification code	cd213256
Empirical formula	C23 H33 Cl F2 N4 O2 Pd
Formula weight	577.38
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, Cc
Unit cell dimensions	a = 18.6367(11) A alpha = 90 deg. b = 18.7315(11) A beta = 90.5090(10) deg. c = 32.542(2) A gamma = 90 deg.
Volume	11359.6(12) A^3
Z, Calculated density	16, 1.350 Mg/m^3

Absorption coefficient	0.784 mm^-1
F(000)	4736
Crystal size	0.213 x 0.178 x 0.121 mm
Theta range for data collection	1.66 to 26.00 deg.
Limiting indices	-22<=h<=22, -23<=k<=19, -38<=l<=40
Reflections collected / unique	34070 / 19614 [R(int) = 0.0245]
Completeness to theta = 26.00	100.0 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.63145
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	19614 / 38 / 1267
Goodness-of-fit on F^2	1.016
Final R indices [I>2sigma(I)]	R1 = 0.0346, wR2 = 0.0842
R indices (all data)	R1 = 0.0415, wR2 = 0.0885
Absolute structure parameter	0.00(4)
Largest diff. peak and hole	0.562 and -0.317 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd213256.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Pd(1)	1424(1)	4677(1)	4990(1)	46(1)
PdA	2813(1)	2130(1)	7891(1)	48(1)

Pd(1B)	8657(1)	3025(1)	7903(1)	49(1)
Pd(1C)	540(1)	543(1)	4990(1)	46(1)
Cl(1)	1829(1)	4513(1)	4309(1)	79(1)
Cl(1A)	2358(1)	2017(1)	8567(1)	85(1)
Cl(1B)	8825(1)	3486(1)	8575(1)	84(1)
Cl(1C)	421(1)	1043(1)	4325(1)	83(1)
F(1)	200(3)	3357(2)	5870(2)	130(2)
F(2)	-271(2)	4562(3)	4656(2)	118(1)
F(1A)	4506(2)	2088(3)	8287(1)	108(1)
F(2A)	4036(3)	704(3)	7127(2)	126(2)
F(1B)	8688(2)	1346(2)	8323(1)	98(1)
F(2B)	10020(2)	1749(3)	7142(1)	113(1)
F(1C)	440(2)	-1119(2)	4549(1)	94(1)
F(2C)	-811(2)	-773(3)	5764(1)	104(1)
N(1)	325(2)	5099(2)	5612(1)	59(1)
N(2)	1379(3)	4767(2)	5898(1)	63(1)
N(3)	896(2)	6125(2)	4636(1)	56(1)
N(4)	2128(2)	3202(2)	5201(1)	60(1)
N(1A)	3972(2)	2469(2)	7286(1)	61(1)
N(2A)	2912(3)	2170(3)	6994(2)	79(2)
N(3A)	3330(2)	3600(2)	8204(1)	63(1)
N(4A)	2132(2)	651(2)	7680(2)	66(1)
N(1B)	8237(2)	1850(2)	7323(1)	59(1)
N(2B)	8552(3)	2871(3)	7004(1)	73(1)
N(3B)	10139(2)	3657(3)	7648(2)	78(1)
N(4B)	7194(2)	2529(2)	8250(1)	62(1)
N(1C)	907(2)	-650(2)	5561(1)	59(1)
N(2C)	524(3)	337(3)	5891(1)	72(1)
N(3C)	-941(2)	1235(2)	5177(1)	61(1)
N(4C)	2010(2)	33(2)	4658(1)	63(1)
O(1)	-847(3)	4744(3)	5601(2)	134(2)
O(2)	2111(2)	4597(2)	5836(1)	71(1)
O(1A)	5139(2)	2112(3)	7370(2)	115(2)
O(2A)	2168(2)	2020(3)	7048(1)	90(2)
O(1B)	8590(3)	693(2)	7407(2)	114(2)
O(2B)	8728(3)	3603(2)	7029(1)	83(1)
O(1C)	598(3)	-1818(2)	5444(2)	102(2)
O(2C)	336(3)	1068(2)	5868(1)	88(1)
C(1)	-231(3)	4658(3)	5510(2)	83(2)
C(2)	-37(3)	3986(4)	5284(2)	74(2)
C(3)	-94(3)	3966(5)	4869(3)	97(3)
C(4)	36(4)	3346(5)	4632(3)	107(3)
C(5)	218(4)	2743(5)	4843(4)	117(3)
C(6)	277(4)	2731(4)	5259(3)	104(3)
C(7)	144(4)	3357(4)	5458(3)	86(2)
C(8)	1038(3)	4843(3)	5559(1)	48(1)
C(9)	2423(4)	4406(4)	6220(2)	99(2)

C(10)	171(4)	5803(3)	5829(2)	76(2)
C(11)	-55(6)	5642(4)	6270(2)	135(4)
C(12)	833(4)	6263(3)	5833(2)	89(2)
C(13)	-408(4)	6209(4)	5604(2)	99(2)
C(14)	1079(3)	5614(3)	4797(2)	53(1)
C(15)	719(3)	6739(3)	4376(2)	63(1)
C(16)	1288(4)	7309(3)	4458(2)	92(2)
C(17)	-15(4)	7007(4)	4481(2)	85(2)
C(18)	757(4)	6483(4)	3937(2)	94(2)
C(19)	1843(2)	3737(2)	5143(2)	52(1)
C(20)	2535(3)	2540(2)	5279(2)	62(1)
C(21)	2015(5)	1925(4)	5282(5)	206(7)
C(22)	3084(4)	2477(4)	4941(2)	101(2)
C(23)	2898(5)	2625(4)	5688(2)	110(2)
C(1A)	4494(3)	2035(4)	7433(2)	79(2)
C(2A)	4284(3)	1425(4)	7706(2)	66(2)
C(3A)	4100(4)	758(4)	7544(3)	88(2)
C(4A)	3979(4)	170(4)	7763(3)	109(3)
C(5A)	4025(4)	231(5)	8170(4)	127(4)
C(6A)	4204(4)	871(5)	8377(2)	103(3)
C(7A)	4329(3)	1456(4)	8111(2)	78(2)
C(8A)	3236(3)	2241(3)	7332(2)	56(1)
C(9A)	1864(5)	1888(8)	6661(3)	186(6)
C(10A)	4164(4)	3136(4)	7031(2)	81(2)
C(11A)	4750(4)	3541(4)	7260(3)	104(2)
C(12A)	3504(4)	3616(4)	6997(3)	121(3)
C(13A)	4429(6)	2880(5)	6606(2)	134(3)
C(14A)	3156(3)	3084(3)	8062(2)	54(1)
C(15A)	3484(3)	4234(3)	8461(2)	74(2)
C(16A)	3464(6)	3988(5)	8905(2)	129(3)
C(17A)	4217(4)	4508(4)	8331(3)	99(2)
C(18A)	2900(5)	4772(4)	8378(3)	113(3)
C(19A)	2401(3)	1183(3)	7739(2)	56(1)
C(20A)	1747(5)	-5(4)	7583(3)	119(2)
C(21A)	1265(5)	125(5)	7227(3)	145(3)
C(22A)	1122(6)	-76(5)	7919(4)	150(3)
C(23A)	2181(6)	-597(4)	7670(4)	157(3)
C(1B)	8677(3)	1324(3)	7463(2)	71(2)
C(2B)	9319(3)	1545(3)	7726(2)	61(1)
C(3B)	9311(4)	1504(3)	8139(2)	72(2)
C(4B)	9914(5)	1644(3)	8382(2)	93(2)
C(5B)	10545(5)	1797(4)	8194(3)	103(2)
C(6B)	10602(4)	1830(3)	7772(3)	88(2)
C(7B)	9984(3)	1700(3)	7553(2)	74(2)
C(8B)	8493(3)	2582(3)	7349(2)	51(1)
C(9B)	8830(5)	3868(4)	6620(2)	119(3)
C(10B)	7561(4)	1675(4)	7085(2)	80(2)

C(11B)	7767(5)	1398(5)	6671(2)	125(3)
C(12B)	7076(4)	2333(5)	7067(3)	102(2)
C(13B)	7145(4)	1102(4)	7322(2)	100(2)
C(14B)	9595(2)	3410(3)	7728(2)	60(1)
C(15B)	10800(3)	4035(4)	7537(3)	115(3)
C(16B)	11449(9)	3542(10)	7625(10)	138(8)
C(17B)	10868(5)	4696(6)	7874(5)	103(4)
C(18B)	10693(11)	4392(13)	7127(5)	132(7)
C(16D)	11309(15)	3821(17)	7743(16)	149(9)
C(18D)	11035(15)	3479(19)	7091(9)	193(15)
C(17D)	10672(17)	4600(20)	7361(9)	141(8)
C(19B)	7714(3)	2699(2)	8095(2)	53(1)
C(20B)	6562(3)	2378(3)	8508(2)	65(1)
C(21B)	6003(4)	2941(4)	8412(2)	91(2)
C(22B)	6304(3)	1636(3)	8394(2)	83(2)
C(23B)	6815(4)	2419(5)	8944(2)	107(2)
C(1C)	492(3)	-1180(3)	5411(2)	66(1)
C(2C)	-159(3)	-949(3)	5170(2)	62(1)
C(3C)	-180(4)	-949(3)	4739(2)	72(2)
C(4C)	-791(5)	-785(4)	4524(2)	90(2)
C(5C)	-1394(4)	-626(3)	4724(3)	92(2)
C(6C)	-1401(4)	-602(4)	5139(3)	94(2)
C(7C)	-798(3)	-773(3)	5353(2)	72(2)
C(8C)	648(3)	67(3)	5544(2)	54(1)
C(9C)	24(5)	1245(5)	6248(2)	126(3)
C(10C)	1589(4)	-825(3)	5811(2)	75(2)
C(11C)	1345(5)	-1158(5)	6224(2)	118(3)
C(12C)	2052(4)	-1349(5)	5565(3)	112(3)
C(13C)	2016(4)	-164(5)	5895(3)	104(2)
C(14C)	-410(2)	960(2)	5140(2)	53(1)
C(15C)	-1616(3)	1621(3)	5240(2)	69(1)
C(16C)	-2242(5)	1128(6)	5120(5)	96(4)
C(16H)	-2103(9)	1120(10)	5502(8)	111(5)
C(17C)	-1528(5)	2019(7)	5640(3)	156(5)
C(18C)	-1681(5)	2190(6)	4916(3)	135(4)
C(19C)	1496(3)	207(3)	4809(2)	53(1)
C(20C)	2629(3)	-126(4)	4404(2)	74(2)
C(21C)	2886(4)	-864(4)	4502(2)	98(2)
C(22C)	3191(4)	432(4)	4497(3)	107(3)
C(23C)	2366(5)	-57(5)	3955(2)	113(3)

Table 3. Bond lengths [A] and angles [deg] for cd213256.

Pd(1)-C(14)	1.971(5)
Pd(1)-C(19)	1.988(5)

Pd(1)-C(8)	2.017(5)
Pd(1)-Cl(1)	2.3667(14)
PdA-C(14A)	1.976(5)
PdA-C(19A)	1.993(5)
PdA-C(8A)	2.002(5)
PdA-Cl(1A)	2.3727(15)
Pd(1B)-C(19B)	1.968(5)
Pd(1B)-C(14B)	1.979(5)
Pd(1B)-C(8B)	2.006(5)
Pd(1B)-Cl(1B)	2.3688(15)
Pd(1C)-C(19C)	1.983(5)
Pd(1C)-C(14C)	1.999(5)
Pd(1C)-C(8C)	2.019(5)
Pd(1C)-Cl(1C)	2.3673(15)
F(1)-C(7)	1.344(9)
F(2)-C(3)	1.352(10)
F(1A)-C(7A)	1.355(9)
F(2A)-C(3A)	1.365(9)
F(1B)-C(3B)	1.343(8)
F(2B)-C(7B)	1.343(7)
F(1C)-C(3C)	1.352(8)
F(2C)-C(7C)	1.335(8)
N(1)-C(1)	1.364(8)
N(1)-C(8)	1.425(6)
N(1)-C(10)	1.525(7)
N(2)-C(8)	1.277(6)
N(2)-O(2)	1.417(6)
N(3)-C(14)	1.142(6)
N(3)-C(15)	1.462(6)
N(4)-C(19)	1.148(6)
N(4)-C(20)	1.475(6)
N(1A)-C(1A)	1.353(8)
N(1A)-C(8A)	1.445(6)
N(1A)-C(10A)	1.543(7)
N(2A)-C(8A)	1.257(7)
N(2A)-O(2A)	1.428(6)
N(3A)-C(14A)	1.118(6)
N(3A)-C(15A)	1.479(7)
N(4A)-C(19A)	1.131(6)
N(4A)-C(20A)	1.457(8)
N(1B)-C(1B)	1.357(8)
N(1B)-C(8B)	1.454(6)
N(1B)-C(10B)	1.510(8)
N(2B)-C(8B)	1.253(6)
N(2B)-O(2B)	1.413(6)
N(3B)-C(14B)	1.147(6)
N(3B)-C(15B)	1.469(7)

N(4B)-C(19B)	1.142(6)
N(4B)-C(20B)	1.479(6)
N(1C)-C(1C)	1.348(8)
N(1C)-C(8C)	1.429(7)
N(1C)-C(10C)	1.538(7)
N(2C)-C(8C)	1.262(7)
N(2C)-O(2C)	1.416(6)
N(3C)-C(14C)	1.123(6)
N(3C)-C(15C)	1.468(6)
N(4C)-C(19C)	1.128(6)
N(4C)-C(20C)	1.456(6)
O(1)-C(1)	1.199(7)
O(2)-C(9)	1.420(7)
O(1A)-C(1A)	1.229(7)
O(2A)-C(9A)	1.400(9)
O(1B)-C(1B)	1.207(7)
O(2B)-C(9B)	1.434(8)
O(1C)-C(1C)	1.216(7)
O(2C)-C(9C)	1.409(8)
C(1)-C(2)	1.505(9)
C(2)-C(7)	1.349(10)
C(2)-C(3)	1.354(11)
C(3)-C(4)	1.416(11)
C(4)-C(5)	1.364(13)
C(4)-H(4)	0.9300
C(5)-C(6)	1.356(12)
C(5)-H(5)	0.9300
C(6)-C(7)	1.365(10)
C(6)-H(6)	0.9300
C(9)-H(9A)	0.9600
C(9)-H(9B)	0.9600
C(9)-H(9C)	0.9600
C(10)-C(12)	1.505(9)
C(10)-C(13)	1.505(9)
C(10)-C(11)	1.529(9)
C(11)-H(11A)	0.9600
C(11)-H(11B)	0.9600
C(11)-H(11C)	0.9600
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(15)-C(17)	1.500(8)
C(15)-C(18)	1.512(8)
C(15)-C(16)	1.527(9)

C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(20)-C(23)	1.498(9)
C(20)-C(21)	1.505(9)
C(20)-C(22)	1.514(9)
C(21)-H(21A)	0.9600
C(21)-H(21B)	0.9600
C(21)-H(21C)	0.9600
C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600
C(22)-H(22C)	0.9600
C(23)-H(23A)	0.9600
C(23)-H(23B)	0.9600
C(23)-H(23C)	0.9600
C(1A)-C(2A)	1.500(8)
C(2A)-C(7A)	1.321(9)
C(2A)-C(3A)	1.397(10)
C(3A)-C(4A)	1.332(10)
C(4A)-C(5A)	1.332(14)
C(4A)-H(4A)	0.9300
C(5A)-C(6A)	1.415(14)
C(5A)-H(5A)	0.9300
C(6A)-C(7A)	1.419(10)
C(6A)-H(6A)	0.9300
C(9A)-H(9A1)	0.9600
C(9A)-H(9A2)	0.9600
C(9A)-H(9A3)	0.9600
C(10A)-C(11A)	1.519(9)
C(10A)-C(12A)	1.527(10)
C(10A)-C(13A)	1.549(11)
C(11A)-H(11D)	0.9600
C(11A)-H(11E)	0.9600
C(11A)-H(11F)	0.9600
C(12A)-H(12D)	0.9600
C(12A)-H(12E)	0.9600
C(12A)-H(12F)	0.9600
C(13A)-H(13D)	0.9600
C(13A)-H(13E)	0.9600
C(13A)-H(13F)	0.9600
C(15A)-C(18A)	1.506(10)

C(15A)-C(16A)	1.515(10)
C(15A)-C(17A)	1.524(9)
C(16A)-H(16D)	0.9600
C(16A)-H(16E)	0.9600
C(16A)-H(16F)	0.9600
C(17A)-H(17D)	0.9600
C(17A)-H(17E)	0.9600
C(17A)-H(17F)	0.9600
C(18A)-H(18D)	0.9600
C(18A)-H(18E)	0.9600
C(18A)-H(18F)	0.9600
C(20A)-C(23A)	1.400(12)
C(20A)-C(21A)	1.481(13)
C(20A)-C(22A)	1.609(14)
C(21A)-H(21D)	0.9600
C(21A)-H(21E)	0.9600
C(21A)-H(21F)	0.9600
C(22A)-H(22D)	0.9600
C(22A)-H(22E)	0.9600
C(22A)-H(22F)	0.9600
C(23A)-H(23D)	0.9600
C(23A)-H(23E)	0.9600
C(23A)-H(23F)	0.9600
C(1B)-C(2B)	1.522(8)
C(2B)-C(3B)	1.346(9)
C(2B)-C(7B)	1.394(9)
C(3B)-C(4B)	1.393(10)
C(4B)-C(5B)	1.361(12)
C(4B)-H(4B)	0.9300
C(5B)-C(6B)	1.379(11)
C(5B)-H(5B)	0.9300
C(6B)-C(7B)	1.371(9)
C(6B)-H(6B)	0.9300
C(9B)-H(9B1)	0.9600
C(9B)-H(9B2)	0.9600
C(9B)-H(9B3)	0.9600
C(10B)-C(11B)	1.497(10)
C(10B)-C(12B)	1.530(11)
C(10B)-C(13B)	1.535(9)
C(11B)-H(11G)	0.9600
C(11B)-H(11H)	0.9600
C(11B)-H(11I)	0.9600
C(12B)-H(12G)	0.9600
C(12B)-H(12H)	0.9600
C(12B)-H(12I)	0.9600
C(13B)-H(13G)	0.9600
C(13B)-H(13H)	0.9600

C(13B)-H(13I)	0.9600
C(15B)-C(16D)	1.22(4)
C(15B)-C(17D)	1.23(4)
C(15B)-C(18B)	1.505(19)
C(15B)-C(16B)	1.546(16)
C(15B)-C(17B)	1.658(16)
C(15B)-C(18D)	1.84(3)
C(16B)-H(16G)	0.9600
C(16B)-H(16H)	0.9600
C(16B)-H(16I)	0.9600
C(17B)-H(17Y)	0.9600
C(17B)-H(17X)	0.9600
C(17B)-H(17Z)	0.9600
C(18B)-H(18G)	0.9600
C(18B)-H(18H)	0.9600
C(18B)-H(18I)	0.9600
C(16D)-H(16J)	0.9600
C(16D)-H(16K)	0.9600
C(16D)-H(16L)	0.9600
C(18D)-H(18J)	0.9600
C(18D)-H(18K)	0.9600
C(18D)-H(18L)	0.9600
C(17D)-H(17J)	0.9600
C(17D)-H(17K)	0.9600
C(17D)-H(17L)	0.9600
C(20B)-C(23B)	1.491(9)
C(20B)-C(21B)	1.514(9)
C(20B)-C(22B)	1.516(8)
C(21B)-H(21G)	0.9600
C(21B)-H(21H)	0.9600
C(21B)-H(21I)	0.9600
C(22B)-H(22G)	0.9600
C(22B)-H(22H)	0.9600
C(22B)-H(22I)	0.9600
C(23B)-H(23G)	0.9600
C(23B)-H(23H)	0.9600
C(23B)-H(23I)	0.9600
C(1C)-C(2C)	1.501(9)
C(2C)-C(7C)	1.378(9)
C(2C)-C(3C)	1.404(9)
C(3C)-C(4C)	1.366(10)
C(4C)-C(5C)	1.336(11)
C(4C)-H(4C)	0.9300
C(5C)-C(6C)	1.354(10)
C(5C)-H(5C)	0.9300
C(6C)-C(7C)	1.355(10)
C(6C)-H(6C)	0.9300

C(9C)-H(9C1)	0.9600
C(9C)-H(9C2)	0.9600
C(9C)-H(9C3)	0.9600
C(10C)-C(13C)	1.496(10)
C(10C)-C(12C)	1.538(10)
C(10C)-C(11C)	1.551(10)
C(11C)-H(11J)	0.9600
C(11C)-H(11K)	0.9600
C(11C)-H(11L)	0.9600
C(12C)-H(12J)	0.9600
C(12C)-H(12K)	0.9600
C(12C)-H(12L)	0.9600
C(13C)-H(13J)	0.9600
C(13C)-H(13K)	0.9600
C(13C)-H(13L)	0.9600
C(15C)-C(18C)	1.502(11)
C(15C)-C(17C)	1.509(11)
C(15C)-C(16C)	1.536(12)
C(15C)-C(16H)	1.56(2)
C(16C)-H(16M)	0.9600
C(16C)-H(16N)	0.9600
C(16C)-H(16O)	0.9600
C(16H)-H(16P)	0.9600
C(16H)-H(16Q)	0.9600
C(16H)-H(16R)	0.9600
C(17C)-H(17G)	0.9600
C(17C)-H(17H)	0.9600
C(17C)-H(17I)	0.9600
C(18C)-H(18X)	0.9600
C(18C)-H(18Y)	0.9600
C(18C)-H(18Z)	0.9600
C(20C)-C(21C)	1.496(9)
C(20C)-C(22C)	1.509(10)
C(20C)-C(23C)	1.542(10)
C(21C)-H(21J)	0.9600
C(21C)-H(21K)	0.9600
C(21C)-H(21L)	0.9600
C(22C)-H(22J)	0.9600
C(22C)-H(22K)	0.9600
C(22C)-H(22L)	0.9600
C(23C)-H(23J)	0.9600
C(23C)-H(23K)	0.9600
C(23C)-H(23L)	0.9600

C(14)-Pd(1)-C(19)	174.59(19)
C(14)-Pd(1)-C(8)	92.10(19)
C(19)-Pd(1)-C(8)	92.75(19)

C(14)-Pd(1)-Cl(1)	85.61(14)
C(19)-Pd(1)-Cl(1)	89.61(14)
C(8)-Pd(1)-Cl(1)	177.19(14)
C(14A)-PdA-C(19A)	175.9(2)
C(14A)-PdA-C(8A)	91.9(2)
C(19A)-PdA-C(8A)	91.2(2)
C(14A)-PdA-Cl(1A)	86.46(15)
C(19A)-PdA-Cl(1A)	90.61(15)
C(8A)-PdA-Cl(1A)	177.51(16)
C(19B)-Pd(1B)-C(14B)	176.4(2)
C(19B)-Pd(1B)-C(8B)	91.6(2)
C(14B)-Pd(1B)-C(8B)	91.1(2)
C(19B)-Pd(1B)-Cl(1B)	86.10(15)
C(14B)-Pd(1B)-Cl(1B)	91.27(17)
C(8B)-Pd(1B)-Cl(1B)	176.66(14)
C(19C)-Pd(1C)-C(14C)	174.7(2)
C(19C)-Pd(1C)-C(8C)	92.5(2)
C(14C)-Pd(1C)-C(8C)	92.1(2)
C(19C)-Pd(1C)-Cl(1C)	86.04(15)
C(14C)-Pd(1C)-Cl(1C)	89.53(15)
C(8C)-Pd(1C)-Cl(1C)	177.05(15)
C(1)-N(1)-C(8)	118.3(4)
C(1)-N(1)-C(10)	119.3(5)
C(8)-N(1)-C(10)	121.9(5)
C(8)-N(2)-O(2)	112.0(4)
C(14)-N(3)-C(15)	171.4(5)
C(19)-N(4)-C(20)	176.6(5)
C(1A)-N(1A)-C(8A)	117.8(4)
C(1A)-N(1A)-C(10A)	120.5(5)
C(8A)-N(1A)-C(10A)	121.4(4)
C(8A)-N(2A)-O(2A)	111.8(5)
C(14A)-N(3A)-C(15A)	169.1(5)
C(19A)-N(4A)-C(20A)	175.5(6)
C(1B)-N(1B)-C(8B)	117.9(4)
C(1B)-N(1B)-C(10B)	120.9(5)
C(8B)-N(1B)-C(10B)	120.4(5)
C(8B)-N(2B)-O(2B)	113.0(5)
C(14B)-N(3B)-C(15B)	174.7(6)
C(19B)-N(4B)-C(20B)	170.8(5)
C(1C)-N(1C)-C(8C)	119.0(4)
C(1C)-N(1C)-C(10C)	120.3(5)
C(8C)-N(1C)-C(10C)	119.9(5)
C(8C)-N(2C)-O(2C)	112.8(5)
C(14C)-N(3C)-C(15C)	177.1(5)
C(19C)-N(4C)-C(20C)	170.3(6)
N(2)-O(2)-C(9)	108.7(5)
C(9A)-O(2A)-N(2A)	108.0(5)

N(2B)-O(2B)-C(9B)	108.4(5)
C(9C)-O(2C)-N(2C)	106.6(5)
O(1)-C(1)-N(1)	125.9(6)
O(1)-C(1)-C(2)	118.0(6)
N(1)-C(1)-C(2)	116.1(5)
C(7)-C(2)-C(3)	114.4(7)
C(7)-C(2)-C(1)	125.7(7)
C(3)-C(2)-C(1)	119.6(7)
F(2)-C(3)-C(2)	120.3(7)
F(2)-C(3)-C(4)	116.3(10)
C(2)-C(3)-C(4)	123.5(9)
C(5)-C(4)-C(3)	116.7(9)
C(5)-C(4)-H(4)	121.6
C(3)-C(4)-H(4)	121.6
C(6)-C(5)-C(4)	122.3(9)
C(6)-C(5)-H(5)	118.9
C(4)-C(5)-H(5)	118.9
C(5)-C(6)-C(7)	116.5(8)
C(5)-C(6)-H(6)	121.8
C(7)-C(6)-H(6)	121.8
F(1)-C(7)-C(2)	116.0(7)
F(1)-C(7)-C(6)	117.4(8)
C(2)-C(7)-C(6)	126.6(8)
N(2)-C(8)-N(1)	112.9(4)
N(2)-C(8)-Pd(1)	126.8(4)
N(1)-C(8)-Pd(1)	120.3(3)
O(2)-C(9)-H(9A)	109.5
O(2)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
O(2)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(12)-C(10)-C(13)	107.4(6)
C(12)-C(10)-N(1)	109.9(5)
C(13)-C(10)-N(1)	110.3(6)
C(12)-C(10)-C(11)	109.8(7)
C(13)-C(10)-C(11)	110.8(6)
N(1)-C(10)-C(11)	108.7(5)
C(10)-C(11)-H(11A)	109.5
C(10)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(10)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(10)-C(12)-H(12A)	109.5
C(10)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5

C(10)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(10)-C(13)-H(13A)	109.5
C(10)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(10)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
N(3)-C(14)-Pd(1)	171.3(4)
N(3)-C(15)-C(17)	109.5(4)
N(3)-C(15)-C(18)	106.6(4)
C(17)-C(15)-C(18)	111.8(5)
N(3)-C(15)-C(16)	107.3(5)
C(17)-C(15)-C(16)	111.1(5)
C(18)-C(15)-C(16)	110.4(5)
C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-H(17A)	109.5
C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(15)-C(18)-H(18A)	109.5
C(15)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(15)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
N(4)-C(19)-Pd(1)	173.5(4)
N(4)-C(20)-C(23)	106.8(5)
N(4)-C(20)-C(21)	108.4(5)
C(23)-C(20)-C(21)	111.2(7)
N(4)-C(20)-C(22)	106.9(4)
C(23)-C(20)-C(22)	110.5(6)
C(21)-C(20)-C(22)	112.7(8)
C(20)-C(21)-H(21A)	109.5
C(20)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5
C(20)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5

C(20)-C(22)-H(22A)	109.5
C(20)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(20)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(20)-C(23)-H(23A)	109.5
C(20)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5
C(20)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5
O(1A)-C(1A)-N(1A)	124.9(6)
O(1A)-C(1A)-C(2A)	116.7(6)
N(1A)-C(1A)-C(2A)	118.4(5)
C(7A)-C(2A)-C(3A)	115.3(6)
C(7A)-C(2A)-C(1A)	122.8(7)
C(3A)-C(2A)-C(1A)	121.6(6)
C(4A)-C(3A)-F(2A)	117.2(8)
C(4A)-C(3A)-C(2A)	125.5(9)
F(2A)-C(3A)-C(2A)	117.3(6)
C(5A)-C(4A)-C(3A)	116.9(9)
C(5A)-C(4A)-H(4A)	121.6
C(3A)-C(4A)-H(4A)	121.6
C(4A)-C(5A)-C(6A)	124.0(8)
C(4A)-C(5A)-H(5A)	118.0
C(6A)-C(5A)-H(5A)	118.0
C(5A)-C(6A)-C(7A)	113.7(8)
C(5A)-C(6A)-H(6A)	123.1
C(7A)-C(6A)-H(6A)	123.1
C(2A)-C(7A)-F(1A)	118.3(6)
C(2A)-C(7A)-C(6A)	124.6(8)
F(1A)-C(7A)-C(6A)	117.1(8)
N(2A)-C(8A)-N(1A)	112.9(5)
N(2A)-C(8A)-PdA	126.5(4)
N(1A)-C(8A)-PdA	120.5(4)
O(2A)-C(9A)-H(9A1)	109.5
O(2A)-C(9A)-H(9A2)	109.5
H(9A1)-C(9A)-H(9A2)	109.5
O(2A)-C(9A)-H(9A3)	109.5
H(9A1)-C(9A)-H(9A3)	109.5
H(9A2)-C(9A)-H(9A3)	109.5
C(11A)-C(10A)-C(12A)	108.4(6)
C(11A)-C(10A)-N(1A)	108.2(5)
C(12A)-C(10A)-N(1A)	109.0(5)
C(11A)-C(10A)-C(13A)	110.9(6)
C(12A)-C(10A)-C(13A)	112.4(7)

N(1A)-C(10A)-C(13A)	107.8(6)
C(10A)-C(11A)-H(11D)	109.5
C(10A)-C(11A)-H(11E)	109.5
H(11D)-C(11A)-H(11E)	109.5
C(10A)-C(11A)-H(11F)	109.5
H(11D)-C(11A)-H(11F)	109.5
H(11E)-C(11A)-H(11F)	109.5
C(10A)-C(12A)-H(12D)	109.5
C(10A)-C(12A)-H(12E)	109.5
H(12D)-C(12A)-H(12E)	109.5
C(10A)-C(12A)-H(12F)	109.5
H(12D)-C(12A)-H(12F)	109.5
H(12E)-C(12A)-H(12F)	109.5
C(10A)-C(13A)-H(13D)	109.5
C(10A)-C(13A)-H(13E)	109.5
H(13D)-C(13A)-H(13E)	109.5
C(10A)-C(13A)-H(13F)	109.5
H(13D)-C(13A)-H(13F)	109.5
H(13E)-C(13A)-H(13F)	109.5
N(3A)-C(14A)-PdA	171.8(5)
N(3A)-C(15A)-C(18A)	107.4(5)
N(3A)-C(15A)-C(16A)	106.8(5)
C(18A)-C(15A)-C(16A)	110.5(7)
N(3A)-C(15A)-C(17A)	106.4(5)
C(18A)-C(15A)-C(17A)	111.9(6)
C(16A)-C(15A)-C(17A)	113.4(6)
C(15A)-C(16A)-H(16D)	109.5
C(15A)-C(16A)-H(16E)	109.5
H(16D)-C(16A)-H(16E)	109.5
C(15A)-C(16A)-H(16F)	109.5
H(16D)-C(16A)-H(16F)	109.5
H(16E)-C(16A)-H(16F)	109.5
C(15A)-C(17A)-H(17D)	109.5
C(15A)-C(17A)-H(17E)	109.5
H(17D)-C(17A)-H(17E)	109.5
C(15A)-C(17A)-H(17F)	109.5
H(17D)-C(17A)-H(17F)	109.5
H(17E)-C(17A)-H(17F)	109.5
C(15A)-C(18A)-H(18D)	109.5
C(15A)-C(18A)-H(18E)	109.5
H(18D)-C(18A)-H(18E)	109.5
C(15A)-C(18A)-H(18F)	109.5
H(18D)-C(18A)-H(18F)	109.5
H(18E)-C(18A)-H(18F)	109.5
N(4A)-C(19A)-PdA	174.4(5)
C(23A)-C(20A)-N(4A)	110.0(7)
C(23A)-C(20A)-C(21A)	129.3(9)

N(4A)-C(20A)-C(21A)	109.0(7)
C(23A)-C(20A)-C(22A)	102.5(9)
N(4A)-C(20A)-C(22A)	106.4(7)
C(21A)-C(20A)-C(22A)	96.2(8)
C(20A)-C(21A)-H(21D)	109.5
C(20A)-C(21A)-H(21E)	109.5
H(21D)-C(21A)-H(21E)	109.5
C(20A)-C(21A)-H(21F)	109.5
H(21D)-C(21A)-H(21F)	109.5
H(21E)-C(21A)-H(21F)	109.5
C(20A)-C(22A)-H(22D)	108.1
C(20A)-C(22A)-H(22E)	112.8
H(22D)-C(22A)-H(22E)	109.5
C(20A)-C(22A)-H(22F)	107.4
H(22D)-C(22A)-H(22F)	109.5
H(22E)-C(22A)-H(22F)	109.5
C(20A)-C(23A)-H(23D)	114.3
C(20A)-C(23A)-H(23E)	112.7
H(23D)-C(23A)-H(23E)	109.5
C(20A)-C(23A)-H(23F)	101.0
H(23D)-C(23A)-H(23F)	109.5
H(23E)-C(23A)-H(23F)	109.5
O(1B)-C(1B)-N(1B)	125.4(6)
O(1B)-C(1B)-C(2B)	117.1(6)
N(1B)-C(1B)-C(2B)	117.4(5)
C(3B)-C(2B)-C(7B)	115.6(6)
C(3B)-C(2B)-C(1B)	121.9(6)
C(7B)-C(2B)-C(1B)	121.9(5)
F(1B)-C(3B)-C(2B)	118.4(6)
F(1B)-C(3B)-C(4B)	118.9(6)
C(2B)-C(3B)-C(4B)	122.7(7)
C(5B)-C(4B)-C(3B)	118.7(7)
C(5B)-C(4B)-H(4B)	120.7
C(3B)-C(4B)-H(4B)	120.7
C(4B)-C(5B)-C(6B)	122.2(7)
C(4B)-C(5B)-H(5B)	118.9
C(6B)-C(5B)-H(5B)	118.9
C(7B)-C(6B)-C(5B)	115.8(7)
C(7B)-C(6B)-H(6B)	122.1
C(5B)-C(6B)-H(6B)	122.1
F(2B)-C(7B)-C(6B)	117.0(6)
F(2B)-C(7B)-C(2B)	117.9(6)
C(6B)-C(7B)-C(2B)	125.0(6)
N(2B)-C(8B)-N(1B)	112.7(5)
N(2B)-C(8B)-Pd(1B)	127.8(4)
N(1B)-C(8B)-Pd(1B)	119.4(3)
O(2B)-C(9B)-H(9B1)	109.5

O(2B)-C(9B)-H(9B2)	109.5
H(9B1)-C(9B)-H(9B2)	109.5
O(2B)-C(9B)-H(9B3)	109.5
H(9B1)-C(9B)-H(9B3)	109.5
H(9B2)-C(9B)-H(9B3)	109.5
C(11B)-C(10B)-N(1B)	108.5(6)
C(11B)-C(10B)-C(12B)	113.6(7)
N(1B)-C(10B)-C(12B)	109.5(5)
C(11B)-C(10B)-C(13B)	110.2(6)
N(1B)-C(10B)-C(13B)	108.4(5)
C(12B)-C(10B)-C(13B)	106.4(6)
C(10B)-C(11B)-H(11G)	109.5
C(10B)-C(11B)-H(11H)	109.5
H(11G)-C(11B)-H(11H)	109.5
C(10B)-C(11B)-H(11I)	109.5
H(11G)-C(11B)-H(11I)	109.5
H(11H)-C(11B)-H(11I)	109.5
C(10B)-C(12B)-H(12G)	109.5
C(10B)-C(12B)-H(12H)	109.5
H(12G)-C(12B)-H(12H)	109.5
C(10B)-C(12B)-H(12I)	109.5
H(12G)-C(12B)-H(12I)	109.5
H(12H)-C(12B)-H(12I)	109.5
C(10B)-C(13B)-H(13G)	109.5
C(10B)-C(13B)-H(13H)	109.5
H(13G)-C(13B)-H(13H)	109.5
C(10B)-C(13B)-H(13I)	109.5
H(13G)-C(13B)-H(13I)	109.5
H(13H)-C(13B)-H(13I)	109.5
N(3B)-C(14B)-Pd(1B)	176.0(5)
C(16D)-C(15B)-C(17D)	133(2)
C(16D)-C(15B)-N(3B)	110.9(16)
C(17D)-C(15B)-N(3B)	111.8(15)
C(16D)-C(15B)-C(18B)	136.6(19)
C(17D)-C(15B)-C(18B)	34.8(19)
N(3B)-C(15B)-C(18B)	109.0(9)
C(16D)-C(15B)-C(16B)	26(3)
C(17D)-C(15B)-C(16B)	138.6(16)
N(3B)-C(15B)-C(16B)	108.9(9)
C(18B)-C(15B)-C(16B)	121.8(15)
C(16D)-C(15B)-C(17B)	80(2)
C(17D)-C(15B)-C(17B)	71.0(18)
N(3B)-C(15B)-C(17B)	105.0(7)
C(18B)-C(15B)-C(17B)	105.2(11)
C(16B)-C(15B)-C(17B)	105.6(14)
C(16D)-C(15B)-C(18D)	93(2)
C(17D)-C(15B)-C(18D)	100(2)

N(3B)-C(15B)-C(18D)	97.2(9)
C(18B)-C(15B)-C(18D)	65.3(15)
C(16B)-C(15B)-C(18D)	67.6(18)
C(17B)-C(15B)-C(18D)	157.8(8)
C(15B)-C(16B)-H(16G)	109.5
C(15B)-C(16B)-H(16H)	109.5
C(15B)-C(16B)-H(16I)	109.5
C(15B)-C(17B)-H(17Y)	109.5
C(15B)-C(17B)-H(17X)	109.5
C(15B)-C(17B)-H(17Z)	109.5
C(15B)-C(18B)-H(18G)	109.5
C(15B)-C(18B)-H(18H)	109.5
C(15B)-C(18B)-H(18I)	109.5
C(15B)-C(16D)-H(16J)	109.5
C(15B)-C(16D)-H(16K)	109.5
H(16J)-C(16D)-H(16K)	109.5
C(15B)-C(16D)-H(16L)	109.5
H(16J)-C(16D)-H(16L)	109.5
H(16K)-C(16D)-H(16L)	109.5
C(15B)-C(18D)-H(18J)	109.1
C(15B)-C(18D)-H(18K)	109.4
H(18J)-C(18D)-H(18K)	109.5
C(15B)-C(18D)-H(18L)	110.0
H(18J)-C(18D)-H(18L)	109.5
H(18K)-C(18D)-H(18L)	109.5
C(15B)-C(17D)-H(17J)	109.5
C(15B)-C(17D)-H(17K)	109.5
H(17J)-C(17D)-H(17K)	109.5
C(15B)-C(17D)-H(17L)	109.5
H(17J)-C(17D)-H(17L)	109.5
H(17K)-C(17D)-H(17L)	109.5
N(4B)-C(19B)-Pd(1B)	172.2(5)
N(4B)-C(20B)-C(23B)	106.4(5)
N(4B)-C(20B)-C(21B)	107.4(5)
C(23B)-C(20B)-C(21B)	111.7(6)
N(4B)-C(20B)-C(22B)	106.7(5)
C(23B)-C(20B)-C(22B)	112.2(6)
C(21B)-C(20B)-C(22B)	111.9(5)
C(20B)-C(21B)-H(21G)	109.5
C(20B)-C(21B)-H(21H)	109.5
H(21G)-C(21B)-H(21H)	109.5
C(20B)-C(21B)-H(21I)	109.5
H(21G)-C(21B)-H(21I)	109.5
H(21H)-C(21B)-H(21I)	109.5
C(20B)-C(22B)-H(22G)	109.5
C(20B)-C(22B)-H(22H)	109.5
H(22G)-C(22B)-H(22H)	109.5

C(20B)-C(22B)-H(22I)	109.5
H(22G)-C(22B)-H(22I)	109.5
H(22H)-C(22B)-H(22I)	109.5
C(20B)-C(23B)-H(23G)	109.5
C(20B)-C(23B)-H(23H)	109.5
H(23G)-C(23B)-H(23H)	109.5
C(20B)-C(23B)-H(23I)	109.5
H(23G)-C(23B)-H(23I)	109.5
H(23H)-C(23B)-H(23I)	109.5
O(1C)-C(1C)-N(1C)	126.8(6)
O(1C)-C(1C)-C(2C)	117.4(6)
N(1C)-C(1C)-C(2C)	115.8(5)
C(7C)-C(2C)-C(3C)	114.6(6)
C(7C)-C(2C)-C(1C)	122.7(6)
C(3C)-C(2C)-C(1C)	122.4(6)
F(1C)-C(3C)-C(4C)	122.1(7)
F(1C)-C(3C)-C(2C)	116.1(6)
C(4C)-C(3C)-C(2C)	121.8(7)
C(5C)-C(4C)-C(3C)	120.2(7)
C(5C)-C(4C)-H(4C)	119.9
C(3C)-C(4C)-H(4C)	119.9
C(4C)-C(5C)-C(6C)	120.5(7)
C(4C)-C(5C)-H(5C)	119.7
C(6C)-C(5C)-H(5C)	119.7
C(5C)-C(6C)-C(7C)	119.4(7)
C(5C)-C(6C)-H(6C)	120.3
C(7C)-C(6C)-H(6C)	120.3
F(2C)-C(7C)-C(6C)	119.5(7)
F(2C)-C(7C)-C(2C)	117.1(6)
C(6C)-C(7C)-C(2C)	123.4(7)
N(2C)-C(8C)-N(1C)	113.9(5)
N(2C)-C(8C)-Pd(1C)	127.3(4)
N(1C)-C(8C)-Pd(1C)	118.7(3)
O(2C)-C(9C)-H(9C1)	109.5
O(2C)-C(9C)-H(9C2)	109.5
H(9C1)-C(9C)-H(9C2)	109.5
O(2C)-C(9C)-H(9C3)	109.5
H(9C1)-C(9C)-H(9C3)	109.5
H(9C2)-C(9C)-H(9C3)	109.5
C(13C)-C(10C)-N(1C)	110.8(5)
C(13C)-C(10C)-C(12C)	108.8(7)
N(1C)-C(10C)-C(12C)	108.9(5)
C(13C)-C(10C)-C(11C)	109.6(6)
N(1C)-C(10C)-C(11C)	107.3(6)
C(12C)-C(10C)-C(11C)	111.4(6)
C(10C)-C(11C)-H(11J)	109.5
C(10C)-C(11C)-H(11K)	109.5

H(11J)-C(11C)-H(11K)	109.5
C(10C)-C(11C)-H(11L)	109.5
H(11J)-C(11C)-H(11L)	109.5
H(11K)-C(11C)-H(11L)	109.5
C(10C)-C(12C)-H(12J)	109.5
C(10C)-C(12C)-H(12K)	109.5
H(12J)-C(12C)-H(12K)	109.5
C(10C)-C(12C)-H(12L)	109.5
H(12J)-C(12C)-H(12L)	109.5
H(12K)-C(12C)-H(12L)	109.5
C(10C)-C(13C)-H(13J)	109.5
C(10C)-C(13C)-H(13K)	109.5
H(13J)-C(13C)-H(13K)	109.5
C(10C)-C(13C)-H(13L)	109.5
H(13J)-C(13C)-H(13L)	109.5
H(13K)-C(13C)-H(13L)	109.5
N(3C)-C(14C)-Pd(1C)	171.4(5)
N(3C)-C(15C)-C(18C)	108.4(5)
N(3C)-C(15C)-C(17C)	106.0(5)
C(18C)-C(15C)-C(17C)	105.3(8)
N(3C)-C(15C)-C(16C)	108.6(6)
C(18C)-C(15C)-C(16C)	101.1(8)
C(17C)-C(15C)-C(16C)	126.3(8)
N(3C)-C(15C)-C(16H)	106.4(8)
C(18C)-C(15C)-C(16H)	139.9(10)
C(17C)-C(15C)-C(16H)	83.4(10)
C(16C)-C(15C)-C(16H)	48.2(8)
C(15C)-C(16C)-H(16M)	109.5
C(15C)-C(16C)-H(16N)	109.5
C(15C)-C(16C)-H(16O)	109.5
C(15C)-C(16H)-H(16P)	109.5
C(15C)-C(16H)-H(16Q)	109.5
H(16P)-C(16H)-H(16Q)	109.5
C(15C)-C(16H)-H(16R)	109.5
H(16P)-C(16H)-H(16R)	109.5
H(16Q)-C(16H)-H(16R)	109.5
C(15C)-C(17C)-H(17G)	109.5
C(15C)-C(17C)-H(17H)	109.5
H(17G)-C(17C)-H(17H)	109.5
C(15C)-C(17C)-H(17I)	109.5
H(17G)-C(17C)-H(17I)	109.5
H(17H)-C(17C)-H(17I)	109.5
C(15C)-C(18C)-H(18X)	109.5
C(15C)-C(18C)-H(18Y)	109.5
H(18X)-C(18C)-H(18Y)	109.5
C(15C)-C(18C)-H(18Z)	109.5
H(18X)-C(18C)-H(18Z)	109.5

H(18Y)-C(18C)-H(18Z)	109.5
N(4C)-C(19C)-Pd(1C)	171.6(5)
N(4C)-C(20C)-C(21C)	108.7(5)
N(4C)-C(20C)-C(22C)	107.2(5)
C(21C)-C(20C)-C(22C)	112.1(6)
N(4C)-C(20C)-C(23C)	105.9(5)
C(21C)-C(20C)-C(23C)	112.2(6)
C(22C)-C(20C)-C(23C)	110.3(6)
C(20C)-C(21C)-H(21J)	109.5
C(20C)-C(21C)-H(21K)	109.5
H(21J)-C(21C)-H(21K)	109.5
C(20C)-C(21C)-H(21L)	109.5
H(21J)-C(21C)-H(21L)	109.5
H(21K)-C(21C)-H(21L)	109.5
C(20C)-C(22C)-H(22J)	109.5
C(20C)-C(22C)-H(22K)	109.5
H(22J)-C(22C)-H(22K)	109.5
C(20C)-C(22C)-H(22L)	109.5
H(22J)-C(22C)-H(22L)	109.5
H(22K)-C(22C)-H(22L)	109.5
C(20C)-C(23C)-H(23J)	109.5
C(20C)-C(23C)-H(23K)	109.5
H(23J)-C(23C)-H(23K)	109.5
C(20C)-C(23C)-H(23L)	109.5
H(23J)-C(23C)-H(23L)	109.5
H(23K)-C(23C)-H(23L)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Aˆ2 x 10ˆ3) for cd213256. The anisotropic displacement factor exponent takes the form: -2 piˆ2 [hˆ2 a*ˆ2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Pd(1)	40(1)	39(1)	58(1)	3(1)	1(1)	4(1)
PdA	39(1)	40(1)	66(1)	0(1)	1(1)	-5(1)
Pd(1B)	40(1)	41(1)	66(1)	3(1)	7(1)	-5(1)
Pd(1C)	39(1)	39(1)	61(1)	-1(1)	7(1)	5(1)
Cl(1)	88(1)	87(1)	63(1)	-9(1)	10(1)	16(1)
Cl(1A)	87(1)	91(1)	79(1)	1(1)	23(1)	-13(1)
Cl(1B)	89(1)	82(1)	82(1)	-19(1)	-2(1)	-13(1)

Cl(1C)	90(1)	82(1)	76(1)	22(1)	11(1)	18(1)
F(1)	126(4)	94(3)	169(5)	30(3)	32(3)	-3(3)
F(2)	75(2)	133(4)	146(4)	2(3)	-19(2)	15(2)
F(1A)	74(2)	137(4)	112(3)	-2(3)	-20(2)	-12(2)
F(2A)	129(4)	120(4)	129(4)	-32(3)	27(3)	11(3)
F(1B)	130(3)	79(2)	85(2)	16(2)	22(2)	-18(2)
F(2B)	104(3)	142(4)	94(3)	-15(3)	29(2)	12(3)
F(1C)	118(3)	78(2)	86(2)	-10(2)	10(2)	20(2)
F(2C)	100(3)	124(3)	88(3)	2(2)	26(2)	-19(2)
N(1)	53(2)	52(2)	73(3)	0(2)	17(2)	9(2)
N(2)	67(3)	65(3)	58(3)	9(2)	7(2)	11(2)
N(3)	61(2)	50(2)	57(2)	11(2)	-1(2)	10(2)
N(4)	54(2)	42(2)	84(3)	7(2)	2(2)	1(2)
N(1A)	48(2)	62(3)	71(3)	17(2)	-2(2)	-13(2)
N(2A)	72(3)	98(4)	66(3)	9(2)	-5(2)	-28(3)
N(3A)	65(3)	47(2)	78(3)	-2(2)	-2(2)	-11(2)
N(4A)	60(2)	44(2)	92(3)	-4(2)	-6(2)	-6(2)
N(1B)	64(3)	56(3)	58(2)	0(2)	2(2)	-13(2)
N(2B)	72(3)	82(3)	65(3)	16(2)	6(2)	-20(3)
N(3B)	44(2)	67(3)	124(4)	25(3)	11(2)	-1(2)
N(4B)	53(2)	65(3)	67(3)	5(2)	7(2)	-10(2)
N(1C)	62(3)	52(3)	61(2)	4(2)	-5(2)	15(2)
N(2C)	80(3)	74(3)	61(3)	-8(2)	4(2)	16(3)
N(3C)	41(2)	56(2)	85(3)	-7(2)	9(2)	5(2)
N(4C)	45(2)	62(3)	80(3)	-3(2)	15(2)	10(2)
O(1)	60(3)	109(4)	235(7)	-39(4)	58(4)	-7(3)
O(2)	67(2)	82(3)	64(2)	6(2)	-9(2)	18(2)
O(1A)	47(2)	141(5)	158(5)	58(4)	24(3)	-1(2)
O(2A)	60(2)	132(4)	77(3)	20(2)	-21(2)	-34(3)
O(1B)	131(4)	56(3)	153(5)	-23(3)	-40(4)	-9(3)
O(2B)	89(3)	76(3)	84(3)	28(2)	-3(2)	-16(2)
O(1C)	139(4)	49(2)	117(4)	11(2)	-24(3)	8(3)
O(2C)	103(3)	76(3)	86(3)	-38(2)	1(2)	22(2)
C(1)	67(4)	65(3)	117(5)	-3(3)	37(4)	11(3)
C(2)	50(3)	63(4)	110(5)	-13(3)	16(3)	-9(3)
C(3)	42(3)	89(5)	161(8)	-17(5)	6(4)	-8(3)
C(4)	67(4)	122(7)	132(6)	-40(5)	-10(4)	-27(4)
C(5)	61(4)	81(5)	208(11)	-31(6)	20(6)	-20(4)
C(6)	61(4)	61(4)	192(9)	3(5)	26(5)	-13(3)
C(7)	73(4)	65(4)	119(6)	4(4)	22(4)	-14(3)
C(8)	47(2)	37(2)	59(3)	10(2)	7(2)	9(2)
C(9)	99(5)	126(6)	72(4)	-5(4)	-24(3)	17(4)
C(10)	85(4)	69(3)	75(4)	-6(3)	33(3)	21(3)
C(11)	213(10)	107(6)	87(5)	5(4)	63(6)	34(6)
C(12)	107(5)	63(4)	98(5)	-13(3)	13(4)	5(4)
C(13)	89(4)	83(4)	125(6)	9(4)	8(4)	33(4)
C(14)	46(3)	54(3)	60(3)	3(2)	7(2)	5(2)

C(15)	72(3)	59(3)	56(3)	16(2)	−1(2)	23(3)
C(16)	77(4)	69(4)	130(6)	23(4)	13(4)	5(3)
C(17)	78(4)	88(5)	89(4)	21(3)	7(3)	31(3)
C(18)	116(6)	97(5)	69(4)	5(3)	−9(4)	33(4)
C(19)	48(2)	41(2)	67(3)	0(2)	8(2)	−3(2)
C(20)	65(3)	36(2)	86(3)	10(2)	−8(3)	6(2)
C(21)	92(6)	48(4)	480(20)	69(8)	−54(9)	−20(4)
C(22)	113(6)	71(4)	118(5)	−7(4)	24(4)	31(4)
C(23)	134(7)	98(5)	97(5)	24(4)	−21(4)	25(5)
C(1A)	55(3)	90(4)	92(4)	16(3)	13(3)	4(3)
C(2A)	43(3)	73(4)	83(4)	21(3)	12(3)	4(3)
C(3A)	67(4)	78(5)	122(6)	12(4)	27(4)	18(3)
C(4A)	78(4)	75(4)	174(8)	30(5)	35(5)	25(4)
C(5A)	72(5)	94(6)	217(11)	74(7)	37(6)	23(4)
C(6A)	67(4)	140(7)	102(5)	55(5)	11(3)	15(4)
C(7A)	50(3)	90(5)	93(5)	14(4)	−4(3)	−2(3)
C(8A)	51(3)	53(3)	63(3)	7(2)	−7(2)	−8(2)
C(9A)	130(7)	338(17)	89(6)	31(7)	−40(5)	−134(9)
C(10A)	80(4)	86(4)	76(4)	26(3)	−11(3)	−34(3)
C(11A)	83(4)	88(5)	141(6)	38(4)	−16(4)	−44(4)
C(12A)	104(6)	91(5)	166(8)	78(5)	−37(5)	−22(4)
C(13A)	164(8)	167(8)	71(4)	33(5)	18(5)	−50(7)
C(14A)	48(3)	48(3)	67(3)	2(2)	0(2)	−11(2)
C(15A)	86(4)	61(3)	74(4)	−5(3)	−4(3)	−14(3)
C(16A)	171(9)	132(7)	83(5)	−17(4)	−8(5)	−57(6)
C(17A)	79(4)	79(4)	137(6)	−20(4)	−14(4)	−29(3)
C(18A)	115(6)	73(4)	153(7)	−33(4)	10(5)	−3(4)
C(19A)	43(2)	46(3)	80(3)	−2(2)	1(2)	−5(2)
C(20A)	101(4)	47(3)	208(6)	−10(4)	−31(4)	−25(3)
C(21A)	130(6)	93(5)	211(8)	−28(5)	−46(6)	−37(5)
C(22A)	120(6)	91(5)	239(8)	−7(5)	−3(6)	−44(5)
C(23A)	138(6)	58(4)	274(8)	3(5)	−34(6)	−15(4)
C(1B)	80(4)	61(4)	73(3)	−14(3)	1(3)	−4(3)
C(2B)	75(4)	42(3)	66(4)	−7(2)	−8(3)	4(3)
C(3B)	96(5)	46(3)	74(4)	7(3)	−4(3)	−7(3)
C(4B)	146(7)	60(4)	73(4)	−2(3)	−33(4)	5(4)
C(5B)	92(5)	70(4)	146(8)	−12(4)	−42(5)	17(4)
C(6B)	68(4)	69(4)	127(6)	−15(4)	−4(4)	16(3)
C(7B)	70(4)	71(4)	79(4)	−10(3)	5(3)	17(3)
C(8B)	46(3)	46(3)	62(3)	5(2)	4(2)	−8(2)
C(9B)	130(6)	122(6)	107(5)	63(5)	9(5)	−30(5)
C(10B)	83(4)	89(4)	66(4)	4(3)	−6(3)	−37(4)
C(11B)	143(7)	158(8)	73(5)	−13(4)	−17(5)	−54(6)
C(12B)	75(4)	121(6)	110(5)	31(5)	−27(4)	−26(4)
C(13B)	95(5)	96(5)	109(5)	8(4)	7(4)	−40(4)
C(14B)	37(2)	53(3)	91(4)	7(2)	8(2)	1(2)
C(15B)	38(3)	119(6)	189(8)	87(6)	26(4)	−4(3)

C(16B)	33(6)	104(14)	280(20)	59(15)	34(8)	26(6)
C(17B)	48(5)	51(5)	211(15)	5(7)	13(7)	-18(4)
C(18B)	104(8)	195(19)	98(12)	48(13)	37(11)	-44(10)
C(16D)	38(10)	125(17)	280(20)	74(17)	20(11)	-1(10)
C(18D)	130(20)	240(30)	210(30)	-80(30)	100(20)	-100(20)
C(17D)	99(9)	200(20)	128(16)	54(16)	40(14)	-39(11)
C(19B)	52(3)	46(3)	61(3)	-1(2)	6(2)	-8(2)
C(20B)	53(3)	78(3)	63(3)	9(3)	15(2)	-22(3)
C(21B)	60(4)	93(5)	119(5)	-8(4)	19(4)	6(3)
C(22B)	68(3)	77(4)	104(5)	12(3)	15(3)	-22(3)
C(23B)	102(5)	150(7)	68(4)	0(4)	13(4)	-30(5)
C(1C)	85(4)	54(3)	60(3)	0(2)	-2(3)	13(3)
C(2C)	69(4)	38(3)	78(4)	3(2)	-5(3)	-9(2)
C(3C)	87(4)	48(3)	80(4)	-1(3)	-4(3)	-2(3)
C(4C)	106(5)	71(4)	91(4)	4(3)	-33(4)	-13(4)
C(5C)	72(4)	65(4)	138(7)	12(4)	-31(4)	-4(3)
C(6C)	64(4)	63(4)	156(7)	10(4)	2(4)	-16(3)
C(7C)	69(4)	50(3)	97(5)	5(3)	5(3)	-14(3)
C(8C)	47(3)	55(3)	59(3)	-2(2)	2(2)	1(2)
C(9C)	146(7)	149(7)	83(4)	-50(4)	-13(4)	65(6)
C(10C)	73(4)	79(4)	73(4)	10(3)	-15(3)	16(3)
C(11C)	152(8)	132(7)	69(4)	25(4)	-13(4)	20(6)
C(12C)	93(5)	131(7)	113(6)	4(5)	-7(4)	61(5)
C(13C)	71(4)	109(6)	130(6)	4(5)	-40(4)	17(4)
C(14C)	43(2)	44(2)	72(3)	-2(2)	7(2)	-1(2)
C(15C)	39(2)	75(4)	92(4)	-2(3)	14(2)	12(2)
C(16C)	28(4)	80(6)	179(11)	-7(8)	-4(6)	-9(4)
C(16H)	53(7)	88(8)	193(13)	-38(10)	19(9)	-18(6)
C(17C)	120(7)	220(12)	127(8)	-48(7)	17(6)	89(8)
C(18C)	98(6)	170(9)	136(7)	45(6)	8(5)	65(6)
C(19C)	51(3)	48(3)	61(3)	-6(2)	-1(2)	4(2)
C(20C)	55(3)	86(4)	82(4)	-6(3)	18(3)	16(3)
C(21C)	86(4)	84(4)	125(6)	2(4)	39(4)	25(4)
C(22C)	67(4)	100(5)	154(7)	-7(5)	34(4)	0(4)
C(23C)	120(6)	142(7)	76(4)	-8(4)	13(4)	40(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd213256.

	x	y	z	U(eq)
H(4)	0	3350	4347	128
H(5)	305	2325	4697	140

H(6)	402	2317	5401	125
H(9A)	2389	4801	6407	149
H(9B)	2919	4285	6182	149
H(9C)	2173	4002	6332	149
H(11A)	-202	6077	6402	203
H(11B)	343	5439	6419	203
H(11C)	-448	5310	6267	203
H(12A)	737	6698	5978	134
H(12B)	967	6371	5555	134
H(12C)	1218	6012	5967	134
H(13A)	-868	6009	5668	149
H(13B)	-331	6175	5313	149
H(13C)	-396	6701	5686	149
H(16A)	1276	7446	4742	138
H(16B)	1191	7719	4289	138
H(16C)	1753	7122	4394	138
H(17A)	-353	6621	4464	128
H(17B)	-152	7375	4291	128
H(17C)	-10	7196	4755	128
H(18A)	1240	6343	3876	141
H(18B)	613	6862	3755	141
H(18C)	443	6082	3899	141
H(21A)	1649	2014	5481	309
H(21B)	2266	1494	5352	309
H(21C)	1798	1875	5015	309
H(22A)	2853	2547	4679	151
H(22B)	3299	2012	4949	151
H(22C)	3449	2834	4980	151
H(23A)	3230	3017	5677	165
H(23B)	3153	2195	5756	165
H(23C)	2545	2718	5894	165
H(4A)	3867	-262	7638	131
H(5A)	3934	-173	8328	153
H(6A)	4238	906	8662	124
H(9A1)	1821	2329	6512	278
H(9A2)	1397	1680	6693	278
H(9A3)	2165	1565	6512	278
H(11D)	4548	3780	7492	156
H(11E)	4960	3887	7079	156
H(11F)	5113	3213	7352	156
H(12D)	3405	3822	7261	181
H(12E)	3099	3338	6908	181
H(12F)	3593	3989	6802	181
H(13D)	4498	3286	6430	201
H(13E)	4078	2567	6485	201
H(13F)	4875	2630	6639	201
H(16D)	2996	3804	8965	193

H(16E)	3568	4384	9083	193
H(16F)	3816	3620	8947	193
H(17D)	4561	4128	8348	148
H(17E)	4363	4891	8509	148
H(17F)	4189	4678	8053	148
H(18D)	2896	4895	8092	170
H(18E)	2987	5193	8540	170
H(18F)	2444	4571	8451	170
H(21D)	1548	210	6986	218
H(21E)	971	535	7280	218
H(21F)	965	-285	7183	218
H(22D)	970	-566	7925	225
H(22E)	717	225	7860	225
H(22F)	1325	52	8181	225
H(23D)	1921	-1015	7753	235
H(23E)	2549	-494	7869	235
H(23F)	2395	-679	7407	235
H(4B)	9886	1633	8667	112
H(5B)	10950	1882	8355	124
H(6B)	11034	1935	7643	106
H(9B1)	9207	3604	6490	179
H(9B2)	8960	4364	6631	179
H(9B3)	8393	3815	6464	179
H(11G)	7966	1780	6511	187
H(11H)	7350	1211	6533	187
H(11I)	8118	1026	6703	187
H(12G)	6905	2440	7338	153
H(12H)	6676	2241	6887	153
H(12I)	7344	2733	6965	153
H(13G)	7436	682	7350	150
H(13H)	6712	986	7175	150
H(13I)	7028	1281	7590	150
H(16G)	11886	3798	7575	208
H(16H)	11425	3133	7448	208
H(16I)	11439	3390	7907	208
H(17Y)	10951	4504	8143	155
H(17X)	10431	4968	7872	155
H(17Z)	11260	5001	7800	155
H(18G)	11106	4678	7065	198
H(18H)	10275	4691	7136	198
H(18I)	10630	4035	6918	198
H(16J)	11748	3931	7605	223
H(16K)	11271	3313	7779	223
H(16L)	11307	4050	8007	223
H(18J)	11161	3784	6866	289
H(18K)	10629	3191	7014	289
H(18L)	11433	3175	7159	289

H(17J)	10250	4815	7475	211
H(17K)	10596	4516	7073	211
H(17L)	11072	4921	7397	211
H(21G)	6183	3402	8493	136
H(21H)	5573	2839	8561	136
H(21I)	5900	2941	8123	136
H(22G)	6163	1629	8109	124
H(22H)	5902	1510	8561	124
H(22I)	6685	1299	8438	124
H(23G)	7210	2098	8984	160
H(23H)	6430	2289	9123	160
H(23I)	6966	2898	9004	160
H(4C)	-787	-784	4238	107
H(5C)	-1812	-531	4575	110
H(6C)	-1816	-470	5277	113
H(9C1)	-443	1032	6265	189
H(9C2)	-18	1754	6270	189
H(9C3)	322	1069	6468	189
H(11J)	1758	-1322	6375	177
H(11K)	1030	-1552	6169	177
H(11L)	1098	-804	6382	177
H(12J)	2036	-1221	5279	169
H(12K)	1870	-1825	5598	169
H(12L)	2539	-1329	5663	169
H(13J)	2113	75	5640	155
H(13K)	2461	-291	6027	155
H(13L)	1749	148	6071	155
H(16M)	-2276	1096	4826	143
H(16N)	-2682	1319	5226	143
H(16O)	-2161	661	5233	143
H(16P)	-2223	702	5345	167
H(16Q)	-2535	1368	5574	167
H(16R)	-1850	981	5747	167
H(17G)	-1169	1787	5806	234
H(17H)	-1976	2022	5783	234
H(17I)	-1382	2501	5585	234
H(18X)	-1265	2491	4926	202
H(18Y)	-2103	2471	4965	202
H(18Z)	-1717	1970	4650	202
H(21J)	3009	-893	4789	147
H(21K)	3300	-971	4341	147
H(21L)	2512	-1201	4441	147
H(22J)	2998	898	4442	160
H(22K)	3603	352	4328	160
H(22L)	3330	401	4782	160
H(23J)	1966	-371	3910	169
H(23K)	2748	-184	3773	169

H(23L)	2221	426	3903	169

Table 6. Torsion angles [deg] for cd213256.

C(8)-N(2)-O(2)-C(9)	-170.4(5)
C(8A)-N(2A)-O(2A)-C(9A)	-174.9(8)
C(8B)-N(2B)-O(2B)-C(9B)	176.1(6)
C(8C)-N(2C)-O(2C)-C(9C)	164.0(6)
C(8)-N(1)-C(1)-O(1)	166.4(7)
C(10)-N(1)-C(1)-O(1)	-5.4(11)
C(8)-N(1)-C(1)-C(2)	-10.2(8)
C(10)-N(1)-C(1)-C(2)	178.0(6)
O(1)-C(1)-C(2)-C(7)	-88.1(10)
N(1)-C(1)-C(2)-C(7)	88.8(8)
O(1)-C(1)-C(2)-C(3)	86.3(9)
N(1)-C(1)-C(2)-C(3)	-96.8(8)
C(7)-C(2)-C(3)-F(2)	-179.8(5)
C(1)-C(2)-C(3)-F(2)	5.2(9)
C(7)-C(2)-C(3)-C(4)	-1.2(10)
C(1)-C(2)-C(3)-C(4)	-176.2(6)
F(2)-C(3)-C(4)-C(5)	179.7(6)
C(2)-C(3)-C(4)-C(5)	1.0(10)
C(3)-C(4)-C(5)-C(6)	-0.4(11)
C(4)-C(5)-C(6)-C(7)	0.0(11)
C(3)-C(2)-C(7)-F(1)	-179.5(6)
C(1)-C(2)-C(7)-F(1)	-4.9(10)
C(3)-C(2)-C(7)-C(6)	0.9(10)
C(1)-C(2)-C(7)-C(6)	175.5(6)
C(5)-C(6)-C(7)-F(1)	-179.9(6)
C(5)-C(6)-C(7)-C(2)	-0.3(11)
O(2)-N(2)-C(8)-N(1)	-172.8(4)
O(2)-N(2)-C(8)-Pd(1)	4.5(6)
C(1)-N(1)-C(8)-N(2)	-113.9(6)
C(10)-N(1)-C(8)-N(2)	57.7(6)
C(1)-N(1)-C(8)-Pd(1)	68.7(6)
C(10)-N(1)-C(8)-Pd(1)	-119.7(5)
C(14)-Pd(1)-C(8)-N(2)	-129.6(5)
C(19)-Pd(1)-C(8)-N(2)	48.0(5)
Cl(1)-Pd(1)-C(8)-N(2)	-165(3)
C(14)-Pd(1)-C(8)-N(1)	47.5(4)
C(19)-Pd(1)-C(8)-N(1)	-134.9(4)
Cl(1)-Pd(1)-C(8)-N(1)	12(3)
C(1)-N(1)-C(10)-C(12)	-168.6(6)
C(8)-N(1)-C(10)-C(12)	19.9(7)
C(1)-N(1)-C(10)-C(13)	-50.3(7)

C(8)-N(1)-C(10)-C(13)	138.1(5)
C(1)-N(1)-C(10)-C(11)	71.3(8)
C(8)-N(1)-C(10)-C(11)	-100.2(7)
C(15)-N(3)-C(14)-Pd(1)	-17(6)
C(19)-Pd(1)-C(14)-N(3)	40(4)
C(8)-Pd(1)-C(14)-N(3)	-167(3)
Cl(1)-Pd(1)-C(14)-N(3)	12(3)
C(14)-N(3)-C(15)-C(17)	146(3)
C(14)-N(3)-C(15)-C(18)	24(4)
C(14)-N(3)-C(15)-C(16)	-94(4)
C(20)-N(4)-C(19)-Pd(1)	59(11)
C(14)-Pd(1)-C(19)-N(4)	-4(6)
C(8)-Pd(1)-C(19)-N(4)	-157(4)
Cl(1)-Pd(1)-C(19)-N(4)	24(4)
C(19)-N(4)-C(20)-C(23)	57(9)
C(19)-N(4)-C(20)-C(21)	177(100)
C(19)-N(4)-C(20)-C(22)	-61(9)
C(8A)-N(1A)-C(1A)-O(1A)	170.2(7)
C(10A)-N(1A)-C(1A)-O(1A)	-2.5(11)
C(8A)-N(1A)-C(1A)-C(2A)	-11.9(9)
C(10A)-N(1A)-C(1A)-C(2A)	175.4(6)
O(1A)-C(1A)-C(2A)-C(7A)	79.6(9)
N(1A)-C(1A)-C(2A)-C(7A)	-98.5(8)
O(1A)-C(1A)-C(2A)-C(3A)	-93.3(8)
N(1A)-C(1A)-C(2A)-C(3A)	88.6(8)
C(7A)-C(2A)-C(3A)-C(4A)	-0.8(10)
C(1A)-C(2A)-C(3A)-C(4A)	172.6(6)
C(7A)-C(2A)-C(3A)-F(2A)	179.0(5)
C(1A)-C(2A)-C(3A)-F(2A)	-7.6(9)
F(2A)-C(3A)-C(4A)-C(5A)	-178.5(6)
C(2A)-C(3A)-C(4A)-C(5A)	1.3(11)
C(3A)-C(4A)-C(5A)-C(6A)	-1.1(12)
C(4A)-C(5A)-C(6A)-C(7A)	0.4(11)
C(3A)-C(2A)-C(7A)-F(1A)	179.9(5)
C(1A)-C(2A)-C(7A)-F(1A)	6.6(9)
C(3A)-C(2A)-C(7A)-C(6A)	0.0(10)
C(1A)-C(2A)-C(7A)-C(6A)	-173.4(6)
C(5A)-C(6A)-C(7A)-C(2A)	0.2(10)
C(5A)-C(6A)-C(7A)-F(1A)	-179.7(6)
O(2A)-N(2A)-C(8A)-N(1A)	-173.7(5)
O(2A)-N(2A)-C(8A)-PdA	2.1(8)
C(1A)-N(1A)-C(8A)-N(2A)	-119.7(6)
C(10A)-N(1A)-C(8A)-N(2A)	52.9(7)
C(1A)-N(1A)-C(8A)-PdA	64.2(6)
C(10A)-N(1A)-C(8A)-PdA	-123.2(5)
C(14A)-PdA-C(8A)-N(2A)	-126.1(6)
C(19A)-PdA-C(8A)-N(2A)	51.2(6)

Cl(1A)-PdA-C(8A)-N(2A)	-174(3)
C(14A)-PdA-C(8A)-N(1A)	49.5(4)
C(19A)-PdA-C(8A)-N(1A)	-133.3(4)
Cl(1A)-PdA-C(8A)-N(1A)	2(4)
C(1A)-N(1A)-C(10A)-C(11A)	-48.4(8)
C(8A)-N(1A)-C(10A)-C(11A)	139.2(6)
C(1A)-N(1A)-C(10A)-C(12A)	-166.1(6)
C(8A)-N(1A)-C(10A)-C(12A)	21.4(8)
C(1A)-N(1A)-C(10A)-C(13A)	71.6(8)
C(8A)-N(1A)-C(10A)-C(13A)	-100.8(7)
C(15A)-N(3A)-C(14A)-PdA	-15(6)
C(19A)-PdA-C(14A)-N(3A)	55(5)
C(8A)-PdA-C(14A)-N(3A)	-167(3)
Cl(1A)-PdA-C(14A)-N(3A)	11(3)
C(14A)-N(3A)-C(15A)-C(18A)	-91(3)
C(14A)-N(3A)-C(15A)-C(16A)	28(3)
C(14A)-N(3A)-C(15A)-C(17A)	149(3)
C(20A)-N(4A)-C(19A)-PdA	93(10)
C(14A)-PdA-C(19A)-N(4A)	-26(7)
C(8A)-PdA-C(19A)-N(4A)	-164(5)
Cl(1A)-PdA-C(19A)-N(4A)	18(5)
C(19A)-N(4A)-C(20A)-C(23A)	160(8)
C(19A)-N(4A)-C(20A)-C(21A)	13(9)
C(19A)-N(4A)-C(20A)-C(22A)	-89(9)
C(8B)-N(1B)-C(1B)-O(1B)	-168.1(6)
C(10B)-N(1B)-C(1B)-O(1B)	2.1(9)
C(8B)-N(1B)-C(1B)-C(2B)	13.9(7)
C(10B)-N(1B)-C(1B)-C(2B)	-175.9(5)
O(1B)-C(1B)-C(2B)-C(3B)	-78.8(8)
N(1B)-C(1B)-C(2B)-C(3B)	99.4(7)
O(1B)-C(1B)-C(2B)-C(7B)	91.4(8)
N(1B)-C(1B)-C(2B)-C(7B)	-90.4(7)
C(7B)-C(2B)-C(3B)-F(1B)	-179.1(5)
C(1B)-C(2B)-C(3B)-F(1B)	-8.3(9)
C(7B)-C(2B)-C(3B)-C(4B)	3.4(9)
C(1B)-C(2B)-C(3B)-C(4B)	174.2(6)
F(1B)-C(3B)-C(4B)-C(5B)	179.4(6)
C(2B)-C(3B)-C(4B)-C(5B)	-3.1(10)
C(3B)-C(4B)-C(5B)-C(6B)	1.2(11)
C(4B)-C(5B)-C(6B)-C(7B)	0.0(10)
C(5B)-C(6B)-C(7B)-F(2B)	178.2(6)
C(5B)-C(6B)-C(7B)-C(2B)	0.5(9)
C(3B)-C(2B)-C(7B)-F(2B)	-179.8(5)
C(1B)-C(2B)-C(7B)-F(2B)	9.4(9)
C(3B)-C(2B)-C(7B)-C(6B)	-2.2(9)
C(1B)-C(2B)-C(7B)-C(6B)	-173.0(6)
O(2B)-N(2B)-C(8B)-N(1B)	173.8(4)

O(2B)-N(2B)-C(8B)-Pd(1B)	-2.7(7)
C(1B)-N(1B)-C(8B)-N(2B)	117.6(6)
C(10B)-N(1B)-C(8B)-N(2B)	-52.7(7)
C(1B)-N(1B)-C(8B)-Pd(1B)	-65.6(6)
C(10B)-N(1B)-C(8B)-Pd(1B)	124.2(4)
C(19B)-Pd(1B)-C(8B)-N(2B)	128.2(5)
C(14B)-Pd(1B)-C(8B)-N(2B)	-49.6(5)
Cl(1B)-Pd(1B)-C(8B)-N(2B)	174(2)
C(19B)-Pd(1B)-C(8B)-N(1B)	-48.2(4)
C(14B)-Pd(1B)-C(8B)-N(1B)	134.1(4)
Cl(1B)-Pd(1B)-C(8B)-N(1B)	-2(3)
C(1B)-N(1B)-C(10B)-C(11B)	-70.7(7)
C(8B)-N(1B)-C(10B)-C(11B)	99.3(7)
C(1B)-N(1B)-C(10B)-C(12B)	164.8(5)
C(8B)-N(1B)-C(10B)-C(12B)	-25.3(7)
C(1B)-N(1B)-C(10B)-C(13B)	49.1(8)
C(8B)-N(1B)-C(10B)-C(13B)	-141.0(6)
C(15B)-N(3B)-C(14B)-Pd(1B)	-70(12)
C(19B)-Pd(1B)-C(14B)-N(3B)	28(9)
C(8B)-Pd(1B)-C(14B)-N(3B)	168(7)
Cl(1B)-Pd(1B)-C(14B)-N(3B)	-14(7)
C(14B)-N(3B)-C(15B)-C(16D)	145(8)
C(14B)-N(3B)-C(15B)-C(17D)	-16(9)
C(14B)-N(3B)-C(15B)-C(18B)	-53(8)
C(14B)-N(3B)-C(15B)-C(16B)	172(8)
C(14B)-N(3B)-C(15B)-C(17B)	60(8)
C(14B)-N(3B)-C(15B)-C(18D)	-119(8)
C(20B)-N(4B)-C(19B)-Pd(1B)	18(6)
C(14B)-Pd(1B)-C(19B)-N(4B)	-53(6)
C(8B)-Pd(1B)-C(19B)-N(4B)	167(3)
Cl(1B)-Pd(1B)-C(19B)-N(4B)	-11(3)
C(19B)-N(4B)-C(20B)-C(23B)	-28(4)
C(19B)-N(4B)-C(20B)-C(21B)	91(3)
C(19B)-N(4B)-C(20B)-C(22B)	-149(3)
C(8C)-N(1C)-C(1C)-O(1C)	-171.1(6)
C(10C)-N(1C)-C(1C)-O(1C)	-1.4(9)
C(8C)-N(1C)-C(1C)-C(2C)	10.4(7)
C(10C)-N(1C)-C(1C)-C(2C)	-179.9(5)
O(1C)-C(1C)-C(2C)-C(7C)	96.8(7)
N(1C)-C(1C)-C(2C)-C(7C)	-84.6(7)
O(1C)-C(1C)-C(2C)-C(3C)	-78.0(8)
N(1C)-C(1C)-C(2C)-C(3C)	100.6(6)
C(7C)-C(2C)-C(3C)-F(1C)	179.9(5)
C(1C)-C(2C)-C(3C)-F(1C)	-4.9(8)
C(7C)-C(2C)-C(3C)-C(4C)	0.2(9)
C(1C)-C(2C)-C(3C)-C(4C)	175.4(6)
F(1C)-C(3C)-C(4C)-C(5C)	179.5(6)

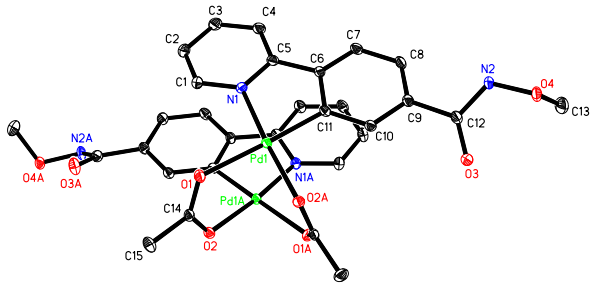
C(2C)-C(3C)-C(4C)-C(5C)	-0.8(10)
C(3C)-C(4C)-C(5C)-C(6C)	2.2(10)
C(4C)-C(5C)-C(6C)-C(7C)	-3.0(10)
C(5C)-C(6C)-C(7C)-F(2C)	-177.8(6)
C(5C)-C(6C)-C(7C)-C(2C)	2.4(9)
C(3C)-C(2C)-C(7C)-F(2C)	179.2(5)
C(1C)-C(2C)-C(7C)-F(2C)	4.0(8)
C(3C)-C(2C)-C(7C)-C(6C)	-1.0(8)
C(1C)-C(2C)-C(7C)-C(6C)	-176.2(5)
O(2C)-N(2C)-C(8C)-N(1C)	174.2(4)
O(2C)-N(2C)-C(8C)-Pd(1C)	-4.2(7)
C(1C)-N(1C)-C(8C)-N(2C)	113.2(6)
C(10C)-N(1C)-C(8C)-N(2C)	-56.6(7)
C(1C)-N(1C)-C(8C)-Pd(1C)	-68.3(5)
C(10C)-N(1C)-C(8C)-Pd(1C)	121.9(5)
C(19C)-Pd(1C)-C(8C)-N(2C)	132.7(5)
C(14C)-Pd(1C)-C(8C)-N(2C)	-44.7(5)
Cl(1C)-Pd(1C)-C(8C)-N(2C)	-167(3)
C(19C)-Pd(1C)-C(8C)-N(1C)	-45.6(4)
C(14C)-Pd(1C)-C(8C)-N(1C)	137.0(4)
Cl(1C)-Pd(1C)-C(8C)-N(1C)	14(3)
C(1C)-N(1C)-C(10C)-C(13C)	172.0(6)
C(8C)-N(1C)-C(10C)-C(13C)	-18.4(8)
C(1C)-N(1C)-C(10C)-C(12C)	52.4(7)
C(8C)-N(1C)-C(10C)-C(12C)	-138.0(6)
C(1C)-N(1C)-C(10C)-C(11C)	-68.3(7)
C(8C)-N(1C)-C(10C)-C(11C)	101.3(6)
C(15C)-N(3C)-C(14C)-Pd(1C)	-101(12)
C(19C)-Pd(1C)-C(14C)-N(3C)	26(4)
C(8C)-Pd(1C)-C(14C)-N(3C)	176(3)
Cl(1C)-Pd(1C)-C(14C)-N(3C)	-6(3)
C(14C)-N(3C)-C(15C)-C(18C)	88(12)
C(14C)-N(3C)-C(15C)-C(17C)	-25(12)
C(14C)-N(3C)-C(15C)-C(16C)	-163(12)
C(14C)-N(3C)-C(15C)-C(16H)	-113(12)
C(20C)-N(4C)-C(19C)-Pd(1C)	17(6)
C(14C)-Pd(1C)-C(19C)-N(4C)	-48(4)
C(8C)-Pd(1C)-C(19C)-N(4C)	162(3)
Cl(1C)-Pd(1C)-C(19C)-N(4C)	-16(3)
C(19C)-N(4C)-C(20C)-C(21C)	-144(3)
C(19C)-N(4C)-C(20C)-C(22C)	94(3)
C(19C)-N(4C)-C(20C)-C(23C)	-23(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd213256 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

6.2 X-ray Crystallographic Data of Compound F



F

Table 1. Crystal data and structure refinement for mo_dm14259_0m.

Identification code	mo_dm14259_0m	
Empirical formula	C34 H36 N4 O12 Pd2	
Formula weight	905.47	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 17.685(2) Å	α= 90°.
b = 11.2677(13) Å	β= 106.035(2)°.	
c = 18.973(2) Å	γ = 90°.	
Volume	3633.8(7) Å^3	

Z	4
Density (calculated)	1.655 Mg/m^3
Absorption coefficient	1.057 mm^{-1}
F(000)	1824
Crystal size	0.25 x 0.20 x 0.11 mm^3
Theta range for data collection	2.17 to 30.70°.
Index ranges	-25<=h<=25, -16<=k<=16, -27<=l<=26
Reflections collected	18036
Independent reflections	5571 [R(int) = 0.0453]
Completeness to theta = 30.70°	99.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8926 and 0.7780
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5571 / 0 / 238
Goodness-of-fit on F^2	0.985
Final R indices [I>2sigma(I)]	R1 = 0.0318, wR2 = 0.0680
R indices (all data)	R1 = 0.0451, wR2 = 0.0733
Largest diff. peak and hole	1.118 and -0.819 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for mo_dm14259_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(2)	-948(1)	11803(1)	7486(1)	19(1)
Pd(1)	416(1)	10331(1)	6963(1)	16(1)
O(1)	-575(1)	11446(1)	6468(1)	21(1)
O(3)	3456(1)	10043(2)	9014(1)	30(1)
O(4)	4759(1)	8961(2)	8866(1)	24(1)
O(5)	2524(1)	10549(2)	5369(1)	53(1)
O(6)	2407(1)	9019(2)	4613(1)	38(1)
N(1)	-56(1)	8804(2)	6504(1)	19(1)
N(2)	4002(1)	8581(2)	8504(1)	22(1)
C(1)	-805(1)	8712(2)	6082(1)	22(1)
C(2)	-1148(1)	7626(2)	5873(1)	26(1)
C(3)	-718(1)	6609(2)	6107(1)	26(1)
C(4)	49(1)	6700(2)	6535(1)	24(1)
C(5)	376(1)	7811(2)	6734(1)	20(1)
C(6)	1163(1)	8074(2)	7213(1)	18(1)
C(7)	1727(1)	7223(2)	7510(1)	23(1)
C(8)	2453(1)	7560(2)	7970(1)	23(1)
C(9)	2606(1)	8759(2)	8125(1)	20(1)

C(10)	2036(1)	9614(2)	7842(1)	19(1)
C(11)	1309(1)	9284(2)	7380(1)	17(1)
C(12)	3383(1)	9184(2)	8595(1)	22(1)
C(13)	4996(2)	8474(3)	9586(1)	35(1)
C(14)	-944(1)	12028(2)	6826(1)	19(1)
C(15)	-1415(1)	13088(2)	6476(1)	28(1)
C(16)	2233(2)	9615(2)	5147(2)	32(1)
C(17)	1624(2)	8995(3)	5428(2)	41(1)

Table 3. Bond lengths [Å] and angles [°] for mo_dm14259_0m.

O(2)-C(14)	1.280(3)
O(2)-Pd(1)#1	2.0466(15)
Pd(1)-C(11)	1.956(2)
Pd(1)-N(1)	2.0035(19)
Pd(1)-O(2)#1	2.0465(15)
Pd(1)-O(1)	2.1493(15)
Pd(1)-Pd(1)#1	2.8232(4)
O(1)-C(14)	1.248(3)
O(3)-C(12)	1.237(3)
O(4)-N(2)	1.392(2)
O(4)-C(13)	1.423(3)

O(5)-C(16)	1.195(3)
O(6)-C(16)	1.321(3)
O(6)-H(6A)	0.8400
N(1)-C(1)	1.349(3)
N(1)-C(5)	1.356(3)
N(2)-C(12)	1.340(3)
N(2)-H(2)	0.8800
C(1)-C(2)	1.376(3)
C(1)-H(1)	0.9500
C(2)-C(3)	1.379(3)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.379(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.387(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.467(3)
C(6)-C(7)	1.387(3)
C(6)-C(11)	1.407(3)
C(7)-C(8)	1.391(3)
C(7)-H(7)	0.9500
C(8)-C(9)	1.393(3)
C(8)-H(8)	0.9500
C(9)-C(10)	1.390(3)

C(9)-C(12)	1.495(3)
C(10)-C(11)	1.392(3)
C(10)-H(10)	0.9500
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(15)	1.501(3)
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-C(17)	1.501(4)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800

C(14)-O(2)-Pd(1)#1	122.29(14)
C(11)-Pd(1)-N(1)	81.40(8)
C(11)-Pd(1)-O(2)#1	94.37(8)
N(1)-Pd(1)-O(2)#1	174.81(7)
C(11)-Pd(1)-O(1)	177.92(8)
N(1)-Pd(1)-O(1)	96.95(7)
O(2)#1-Pd(1)-O(1)	87.34(6)
C(11)-Pd(1)-Pd(1)#1	103.41(7)

N(1)-Pd(1)-Pd(1)#1	94.40(5)
O(2)#1-Pd(1)-Pd(1)#1	83.61(4)
O(1)-Pd(1)-Pd(1)#1	77.93(4)
C(14)-O(1)-Pd(1)	123.72(14)
N(2)-O(4)-C(13)	110.11(18)
C(16)-O(6)-H(6A)	109.5
C(1)-N(1)-C(5)	120.1(2)
C(1)-N(1)-Pd(1)	123.08(16)
C(5)-N(1)-Pd(1)	116.01(14)
C(12)-N(2)-O(4)	119.24(19)
C(12)-N(2)-H(2)	120.4
O(4)-N(2)-H(2)	120.4
N(1)-C(1)-C(2)	121.4(2)
N(1)-C(1)-H(1)	119.3
C(2)-C(1)-H(1)	119.3
C(1)-C(2)-C(3)	119.1(2)
C(1)-C(2)-H(2A)	120.5
C(3)-C(2)-H(2A)	120.5
C(4)-C(3)-C(2)	119.6(2)
C(4)-C(3)-H(3)	120.2
C(2)-C(3)-H(3)	120.2
C(3)-C(4)-C(5)	119.7(2)
C(3)-C(4)-H(4)	120.2

C(5)-C(4)-H(4)	120.2
N(1)-C(5)-C(4)	120.1(2)
N(1)-C(5)-C(6)	112.7(2)
C(4)-C(5)-C(6)	127.1(2)
C(7)-C(6)-C(11)	120.8(2)
C(7)-C(6)-C(5)	124.4(2)
C(11)-C(6)-C(5)	114.8(2)
C(6)-C(7)-C(8)	120.1(2)
C(6)-C(7)-H(7)	119.9
C(8)-C(7)-H(7)	119.9
C(7)-C(8)-C(9)	119.3(2)
C(7)-C(8)-H(8)	120.3
C(9)-C(8)-H(8)	120.3
C(10)-C(9)-C(8)	120.8(2)
C(10)-C(9)-C(12)	117.3(2)
C(8)-C(9)-C(12)	121.9(2)
C(9)-C(10)-C(11)	120.2(2)
C(9)-C(10)-H(10)	119.9
C(11)-C(10)-H(10)	119.9
C(10)-C(11)-C(6)	118.8(2)
C(10)-C(11)-Pd(1)	126.59(17)
C(6)-C(11)-Pd(1)	114.54(15)
O(3)-C(12)-N(2)	122.4(2)

O(3)-C(12)-C(9)	123.3(2)
N(2)-C(12)-C(9)	114.2(2)
O(4)-C(13)-H(13A)	109.5
O(4)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
O(4)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
O(1)-C(14)-O(2)	124.9(2)
O(1)-C(14)-C(15)	119.2(2)
O(2)-C(14)-C(15)	115.9(2)
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
O(5)-C(16)-O(6)	123.3(3)
O(5)-C(16)-C(17)	124.4(3)
O(6)-C(16)-C(17)	112.2(2)
C(16)-C(17)-H(17A)	109.5
C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5

C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for mo_dm14259_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(2)	18(1)	15(1)	24(1)	1(1)	4(1)	1(1)
Pd(1)	13(1)	14(1)	19(1)	-1(1)	4(1)	0(1)
O(1)	18(1)	20(1)	22(1)	2(1)	2(1)	3(1)
O(3)	19(1)	27(1)	40(1)	-13(1)	3(1)	2(1)
O(4)	14(1)	25(1)	29(1)	1(1)	1(1)	0(1)
O(5)	47(1)	40(1)	68(2)	-15(1)	11(1)	-15(1)
O(6)	38(1)	35(1)	40(1)	2(1)	12(1)	-14(1)
N(1)	18(1)	19(1)	18(1)	-3(1)	4(1)	-1(1)
N(2)	12(1)	24(1)	29(1)	-6(1)	1(1)	1(1)
C(1)	19(1)	24(1)	22(1)	-2(1)	2(1)	1(1)
C(2)	20(1)	29(1)	28(1)	-5(1)	3(1)	-4(1)

C(3)	24(1)	21(1)	33(1)	-6(1)	7(1)	-6(1)
C(4)	23(1)	18(1)	32(1)	-5(1)	7(1)	0(1)
C(5)	19(1)	20(1)	21(1)	-3(1)	8(1)	1(1)
C(6)	15(1)	19(1)	22(1)	-3(1)	6(1)	0(1)
C(7)	20(1)	16(1)	34(1)	-3(1)	9(1)	0(1)
C(8)	16(1)	20(1)	31(1)	1(1)	4(1)	5(1)
C(9)	15(1)	21(1)	25(1)	-2(1)	6(1)	0(1)
C(10)	18(1)	16(1)	24(1)	-2(1)	6(1)	0(1)
C(11)	15(1)	17(1)	21(1)	1(1)	7(1)	1(1)
C(12)	17(1)	21(1)	26(1)	2(1)	5(1)	3(1)
C(13)	29(1)	43(2)	29(1)	2(1)	0(1)	7(1)
C(14)	13(1)	15(1)	25(1)	0(1)	1(1)	-2(1)
C(15)	26(1)	24(1)	31(1)	8(1)	6(1)	8(1)
C(16)	23(1)	32(1)	32(1)	7(1)	-5(1)	-2(1)
C(17)	34(2)	51(2)	35(2)	6(1)	5(1)	-5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mo_dm14259_0m.

	x	y	z	U(eq)

H(6A)	2743	9398	4465	56
H(2)	3932	7952	8218	27
H(1)	-1102	9414	5927	27
H(2A)	-1675	7576	5572	32
H(3)	-948	5851	5973	31
H(4)	352	6004	6694	29
H(7)	1618	6408	7399	27
H(8)	2840	6980	8176	27
H(10)	2144	10426	7964	23
H(13A)	4978	7606	9556	53
H(13B)	4639	8751	9865	53
H(13C)	5533	8731	9832	53
H(15A)	-1550	13011	5942	41
H(15B)	-1103	13810	6628	41
H(15C)	-1898	13134	6632	41
H(17A)	1554	9421	5856	62
H(17B)	1796	8181	5569	62
H(17C)	1123	8978	5043	62

Table 6. Torsion angles [°] for mo_dm14259_0m.

C(11)-Pd(1)-O(1)-C(14)	157(2)
N(1)-Pd(1)-O(1)-C(14)	119.22(18)

O(2)#1-Pd(1)-O(1)-C(14)	-57.85(17)
Pd(1)#1-Pd(1)-O(1)-C(14)	26.18(16)
C(11)-Pd(1)-N(1)-C(1)	176.5(2)
O(2)#1-Pd(1)-N(1)-C(1)	140.8(7)
O(1)-Pd(1)-N(1)-C(1)	-4.77(19)
Pd(1)#1-Pd(1)-N(1)-C(1)	73.59(18)
C(11)-Pd(1)-N(1)-C(5)	7.00(16)
O(2)#1-Pd(1)-N(1)-C(5)	-28.7(8)
O(1)-Pd(1)-N(1)-C(5)	-174.27(16)
Pd(1)#1-Pd(1)-N(1)-C(5)	-95.92(16)
C(13)-O(4)-N(2)-C(12)	85.6(3)
C(5)-N(1)-C(1)-C(2)	-0.3(3)
Pd(1)-N(1)-C(1)-C(2)	-169.42(18)
N(1)-C(1)-C(2)-C(3)	0.7(4)
C(1)-C(2)-C(3)-C(4)	-0.9(4)
C(2)-C(3)-C(4)-C(5)	0.8(4)
C(1)-N(1)-C(5)-C(4)	0.2(3)
Pd(1)-N(1)-C(5)-C(4)	170.05(18)
C(1)-N(1)-C(5)-C(6)	-177.4(2)
Pd(1)-N(1)-C(5)-C(6)	-7.6(2)
C(3)-C(4)-C(5)-N(1)	-0.4(4)
C(3)-C(4)-C(5)-C(6)	176.8(2)
N(1)-C(5)-C(6)-C(7)	-178.5(2)

C(4)-C(5)-C(6)-C(7)	4.1(4)
N(1)-C(5)-C(6)-C(11)	3.6(3)
C(4)-C(5)-C(6)-C(11)	-173.8(2)
C(11)-C(6)-C(7)-C(8)	-1.0(3)
C(5)-C(6)-C(7)-C(8)	-178.8(2)
C(6)-C(7)-C(8)-C(9)	-0.3(4)
C(7)-C(8)-C(9)-C(10)	1.7(4)
C(7)-C(8)-C(9)-C(12)	-178.0(2)
C(8)-C(9)-C(10)-C(11)	-1.8(3)
C(12)-C(9)-C(10)-C(11)	177.8(2)
C(9)-C(10)-C(11)-C(6)	0.6(3)
C(9)-C(10)-C(11)-Pd(1)	176.88(17)
C(7)-C(6)-C(11)-C(10)	0.8(3)
C(5)-C(6)-C(11)-C(10)	178.8(2)
C(7)-C(6)-C(11)-Pd(1)	-175.92(18)
C(5)-C(6)-C(11)-Pd(1)	2.1(3)
N(1)-Pd(1)-C(11)-C(10)	178.8(2)
O(2)#1-Pd(1)-C(11)-C(10)	-4.2(2)
O(1)-Pd(1)-C(11)-C(10)	141(2)
Pd(1)#1-Pd(1)-C(11)-C(10)	-88.7(2)
N(1)-Pd(1)-C(11)-C(6)	-4.73(16)
O(2)#1-Pd(1)-C(11)-C(6)	172.24(16)
O(1)-Pd(1)-C(11)-C(6)	-42(2)

Pd(1)#1-Pd(1)-C(11)-C(6)	87.78(16)
O(4)-N(2)-C(12)-O(3)	-2.9(4)
O(4)-N(2)-C(12)-C(9)	174.58(19)
C(10)-C(9)-C(12)-O(3)	34.9(3)
C(8)-C(9)-C(12)-O(3)	-145.5(3)
C(10)-C(9)-C(12)-N(2)	-142.6(2)
C(8)-C(9)-C(12)-N(2)	37.0(3)
Pd(1)-O(1)-C(14)-O(2)	-21.1(3)
Pd(1)-O(1)-C(14)-C(15)	157.88(16)
Pd(1)#1-O(2)-C(14)-O(1)	-5.7(3)
Pd(1)#1-O(2)-C(14)-C(15)	175.23(14)

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2

Table 7. Hydrogen bonds for mo_dm14259_0m [Å and °].

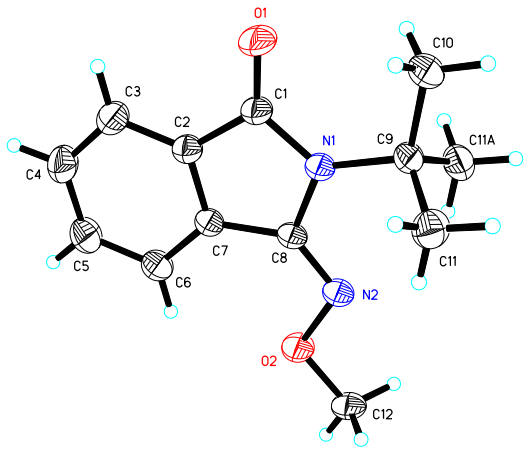
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2)-H(2)...O(2)#2	0.88	1.95	2.801(3)	161.3
C(1)-H(1)...O(1)	0.95	2.58	3.166(3)	120.5
C(2)-H(2A)...O(5)#3	0.95	2.66	3.265(3)	122.2
C(4)-H(4)...O(4)#4	0.95	2.52	3.219(3)	130.5

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2 #2 x+1/2,y-1/2,z #3 x-1/2,y-1/2,z

#4 -x+1/2,y-1/2,-z+3/2

6.3 X-ray Crystallographic Data of Compound 3a



3a

Table 1. Crystal data and structure refinement for cd213442.

Identification code	cd213442
Empirical formula	C13 H16 N2 O2
Formula weight	232.28
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/m
Unit cell dimensions	a = 9.306(2) A alpha = 90 deg. b = 7.1675(17) A beta = 110.334(4) deg. c = 9.855(3) A gamma = 90 deg.

Volume	616.4(3) A^3
Z, Calculated density	2, 1.252 Mg/m^3
Absorption coefficient	0.086 mm^-1
F(000)	248
Crystal size	0.25 x 0.20 x 0.16 mm
Theta range for data collection	2.59 to 28.31 deg.
Limiting indices	-12<=h<=10, -8<=k<=9, -7<=l<=13
Reflections collected / unique	4212 / 1642 [R(int) = 0.0290]
Completeness to theta = 28.31	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9864 and 0.9789
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1642 / 0 / 103
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0511, wR2 = 0.1463
R indices (all data)	R1 = 0.0666, wR2 = 0.1575
Largest diff. peak and hole	0.213 and -0.228 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd213442.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	5628(2)	2500	123(2)	75(1)

O(2)	5149(2)	2500	5700(1)	59(1)
N(1)	4542(2)	2500	1935(2)	43(1)
N(2)	4274(2)	2500	4217(2)	47(1)
C(1)	5725(2)	2500	1376(2)	47(1)
C(2)	7166(2)	2500	2636(2)	45(1)
C(3)	8646(2)	2500	2634(2)	60(1)
C(4)	9820(2)	2500	3959(3)	68(1)
C(5)	9501(3)	2500	5232(3)	66(1)
C(6)	8018(2)	2500	5231(2)	54(1)
C(7)	6835(2)	2500	3894(2)	41(1)
C(8)	5155(2)	2500	3469(2)	40(1)
C(9)	2848(2)	2500	1105(2)	49(1)
C(10)	2513(3)	2500	-525(2)	70(1)
C(11)	2166(2)	730(2)	1492(2)	64(1)
C(12)	4136(3)	2500	6489(2)	54(1)

Table 3. Bond lengths [A] and angles [deg] for cd213442.

O(1)-C(1)	1.208(2)
O(2)-N(2)	1.4044(18)
O(2)-C(12)	1.415(2)
N(1)-C(1)	1.391(2)
N(1)-C(8)	1.418(2)
N(1)-C(9)	1.503(2)
N(2)-C(8)	1.279(2)
C(1)-C(2)	1.478(3)
C(2)-C(7)	1.377(2)
C(2)-C(3)	1.377(3)
C(3)-C(4)	1.381(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.386(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.380(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.393(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.472(3)
C(9)-C(10)	1.526(3)
C(9)-C(11)	1.5254(18)
C(9)-C(11)#1	1.5254(18)
C(10)-H(10A)	0.9600
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-H(11A)	0.9600

C(11)-H(11B)	0.9600
C(11)-H(11C)	0.9600
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600

N(2)-O(2)-C(12)	108.43(14)
C(1)-N(1)-C(8)	109.96(14)
C(1)-N(1)-C(9)	127.51(15)
C(8)-N(1)-C(9)	122.53(14)
C(8)-N(2)-O(2)	110.11(14)
O(1)-C(1)-N(1)	128.13(18)
O(1)-C(1)-C(2)	125.63(18)
N(1)-C(1)-C(2)	106.25(15)
C(7)-C(2)-C(3)	122.53(18)
C(7)-C(2)-C(1)	109.52(16)
C(3)-C(2)-C(1)	127.95(18)
C(2)-C(3)-C(4)	117.5(2)
C(2)-C(3)-H(3)	121.2
C(4)-C(3)-H(3)	121.3
C(3)-C(4)-C(5)	120.5(2)
C(3)-C(4)-H(4)	119.8
C(5)-C(4)-H(4)	119.8
C(6)-C(5)-C(4)	121.9(2)
C(6)-C(5)-H(5)	119.0
C(4)-C(5)-H(5)	119.0
C(5)-C(6)-C(7)	117.5(2)
C(5)-C(6)-H(6)	121.3
C(7)-C(6)-H(6)	121.3
C(2)-C(7)-C(6)	120.09(18)
C(2)-C(7)-C(8)	106.97(15)
C(6)-C(7)-C(8)	132.94(17)
N(2)-C(8)-N(1)	120.88(15)
N(2)-C(8)-C(7)	131.82(16)
N(1)-C(8)-C(7)	107.30(14)
N(1)-C(9)-C(10)	111.42(16)
N(1)-C(9)-C(11)	108.22(10)
C(10)-C(9)-C(11)	108.23(11)
N(1)-C(9)-C(11)#1	108.22(10)
C(10)-C(9)-C(11)#1	108.23(11)
C(11)-C(9)-C(11)#1	112.56(17)
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5

C(9)-C(11)-H(11A)	109.5
C(9)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(9)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
O(2)-C(12)-H(12A)	109.5
O(2)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
O(2)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd213442. The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	60(1)	132(1)	35(1)	0	21(1)	0
O(2)	54(1)	92(1)	29(1)	0	14(1)	0
N(1)	41(1)	58(1)	30(1)	0	11(1)	0
N(2)	49(1)	60(1)	30(1)	0	12(1)	0
C(1)	47(1)	60(1)	36(1)	0	17(1)	0
C(2)	45(1)	49(1)	40(1)	0	16(1)	0
C(3)	50(1)	78(1)	57(1)	0	23(1)	0
C(4)	42(1)	90(2)	69(2)	0	15(1)	0
C(5)	49(1)	84(2)	54(1)	0	3(1)	0
C(6)	52(1)	63(1)	42(1)	0	10(1)	0
C(7)	43(1)	40(1)	37(1)	0	12(1)	0
C(8)	45(1)	42(1)	30(1)	0	11(1)	0
C(9)	39(1)	68(1)	35(1)	0	9(1)	0
C(10)	54(1)	115(2)	34(1)	0	6(1)	0
C(11)	54(1)	77(1)	57(1)	-7(1)	14(1)	-15(1)
C(12)	67(1)	63(1)	36(1)	0	24(1)	0

Table 5. Hydrogen coordinates (x 10^4) and isotropic

displacement parameters (A^2 x 10^3) for cd213442.

	x	y	z	U(eq)
H(3)	8846	2500	1773	72
H(4)	10833	2500	3997	82
H(5)	10310	2500	6112	79
H(6)	7815	2500	6092	65
H(10A)	2865	1351	-801	106
H(10B)	3033	3529	-775	106
H(10C)	1429	2620	-1024	106
H(11A)	2612	-342	1208	96
H(11B)	1077	723	994	96
H(11C)	2379	695	2517	96
H(12A)	3657	1299	6408	81
H(12B)	3365	3438	6106	81
H(12C)	4700	2763	7489	81

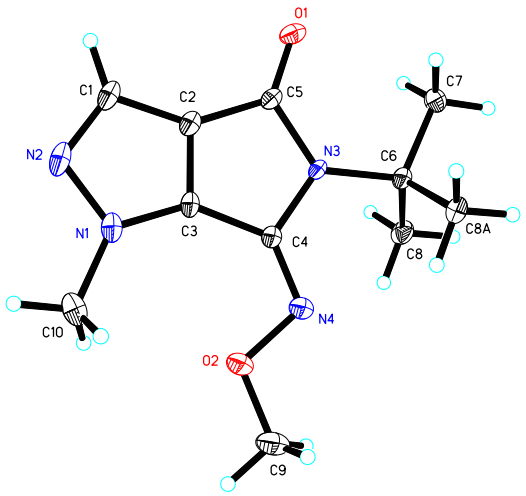
Table 6. Torsion angles [deg] for cd213442.

C(12)-O(2)-N(2)-C(8)	180.0
C(8)-N(1)-C(1)-O(1)	180.0
C(9)-N(1)-C(1)-O(1)	0.0
C(8)-N(1)-C(1)-C(2)	0.0
C(9)-N(1)-C(1)-C(2)	180.0
O(1)-C(1)-C(2)-C(7)	180.0
N(1)-C(1)-C(2)-C(7)	0.0
O(1)-C(1)-C(2)-C(3)	0.0
N(1)-C(1)-C(2)-C(3)	180.0
C(7)-C(2)-C(3)-C(4)	0.0
C(1)-C(2)-C(3)-C(4)	180.0
C(2)-C(3)-C(4)-C(5)	0.0
C(3)-C(4)-C(5)-C(6)	0.0
C(4)-C(5)-C(6)-C(7)	0.0
C(3)-C(2)-C(7)-C(6)	0.0
C(1)-C(2)-C(7)-C(6)	180.0
C(3)-C(2)-C(7)-C(8)	180.0
C(1)-C(2)-C(7)-C(8)	0.0
C(5)-C(6)-C(7)-C(2)	0.0
C(5)-C(6)-C(7)-C(8)	180.0
O(2)-N(2)-C(8)-N(1)	180.0
O(2)-N(2)-C(8)-C(7)	0.0

C(1)-N(1)-C(8)-N(2)	180.0
C(9)-N(1)-C(8)-N(2)	0.0
C(1)-N(1)-C(8)-C(7)	0.0
C(9)-N(1)-C(8)-C(7)	180.0
C(2)-C(7)-C(8)-N(2)	180.0
C(6)-C(7)-C(8)-N(2)	0.0
C(2)-C(7)-C(8)-N(1)	0.0
C(6)-C(7)-C(8)-N(1)	180.0
C(1)-N(1)-C(9)-C(10)	0.0
C(8)-N(1)-C(9)-C(10)	180.0
C(1)-N(1)-C(9)-C(11)	118.87(11)
C(8)-N(1)-C(9)-C(11)	-61.13(11)
C(1)-N(1)-C(9)-C(11)#1	-118.88(11)
C(8)-N(1)-C(9)-C(11)#1	61.12(11)

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z

6.4 X-ray Crystallographic Data of Compound 5i



5i

Table 1. Crystal data and structure refinement for mo_dm13420_0m.

Identification code	mo_dm13420_0m
Empirical formula	C11 H16 N4 O2

Formula weight	236.28	
Temperature	140(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P m c 21	
Unit cell dimensions	a = 6.7109(16) Å	α= 90°.
	b = 10.133(3) Å	β= 90°.
	c = 18.129(4) Å	γ = 90°.
Volume	1232.8(5) $Å^3$	
Z	4	
Density (calculated)	1.273 Mg/m^3	
Absorption coefficient	0.091 mm^{-1}	
F(000)	504	
Crystal size	0.200 x 0.150 x 0.100 mm^3	
Theta range for data collection	2.010 to 30.563°.	
Index ranges	-9<=h<=9, -14<=k<=14, -25<=l<=24	
Reflections collected	11716	
Independent reflections	3642 [R(int) = 0.0538]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7461 and 0.5572	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3642 / 1 / 202	

Goodness-of-fit on F^2	1.011
Final R indices [I>2sigma(I)]	R1 = 0.0459, wR2 = 0.1035
R indices (all data)	R1 = 0.0689, wR2 = 0.1179
Absolute structure parameter	-0.6(10)
Extinction coefficient	n/a
Largest diff. peak and hole	0.183 and -0.335 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for mo_dm13420_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	0	699(3)	1812(2)	30(1)
N(2)	0	-90(3)	1197(2)	36(1)
N(3)	0	4171(2)	1277(1)	20(1)
N(4)	0	3778(3)	2561(2)	28(1)
N(5)	5000	799(3)	1654(2)	31(1)
N(6)	5000	1654(3)	2237(2)	34(1)
N(7)	5000	2591(2)	-91(1)	22(1)
N(8)	5000	267(3)	-202(2)	34(1)
O(1)	0	3846(3)	-8(1)	32(1)
O(2)	0	2706(3)	3054(1)	41(1)
O(3)	5000	4735(2)	408(1)	28(1)

O(4)	5000	-868(3)	240(2)	54(1)
C(1)	0	719(3)	614(2)	30(1)
C(2)	0	2039(3)	859(2)	24(1)
C(3)	0	1984(3)	1604(2)	23(1)
C(4)	0	3326(3)	1903(2)	22(1)
C(5)	0	3426(3)	615(2)	22(1)
C(6)	0	5650(3)	1354(2)	24(1)
C(7)	0	6329(3)	599(2)	34(1)
C(8)	1885(4)	6076(2)	1767(1)	31(1)
C(9)	0	3197(5)	3773(2)	60(1)
C(10)	0	69(4)	2534(2)	44(1)
C(11)	5000	2857(4)	1946(2)	29(1)
C(12)	5000	2780(3)	1169(2)	22(1)
C(13)	5000	1473(3)	1010(2)	24(1)
C(14)	5000	1293(3)	210(2)	24(1)
C(15)	5000	3547(3)	480(2)	21(1)
C(16)	5000	2833(3)	-910(2)	28(1)
C(17)	3105(4)	2228(3)	-1239(2)	40(1)
C(18)	5000	4315(4)	-1075(2)	52(1)
C(19)	5000	-1985(4)	-220(4)	72(2)
C(20)	5000	-613(4)	1814(3)	48(1)

Table 3. Bond lengths [Å] and angles [°] for mo_dm13420_0m.

N(1)-C(3)	1.355(4)
N(1)-N(2)	1.371(4)
N(1)-C(10)	1.458(5)
N(2)-C(1)	1.338(5)
N(3)-C(5)	1.418(4)
N(3)-C(4)	1.421(4)
N(3)-C(6)	1.505(4)
N(4)-C(4)	1.278(4)
N(4)-O(2)	1.407(3)
N(5)-C(13)	1.351(4)
N(5)-N(6)	1.368(4)
N(5)-C(20)	1.459(4)
N(6)-C(11)	1.328(5)
N(7)-C(15)	1.418(4)
N(7)-C(14)	1.423(4)
N(7)-C(16)	1.504(4)
N(8)-C(14)	1.280(5)
N(8)-O(4)	1.401(4)
O(1)-C(5)	1.206(4)
O(2)-C(9)	1.395(5)
O(3)-C(15)	1.211(4)

O(4)-C(19)	1.406(6)
C(1)-C(2)	1.409(4)
C(1)-H(1)	0.9500
C(2)-C(3)	1.352(4)
C(2)-C(5)	1.473(5)
C(3)-C(4)	1.464(4)
C(6)-C(8)#1	1.532(3)
C(6)-C(8)	1.532(3)
C(6)-C(7)	1.532(4)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-H(10A)	0.9598
C(10)-H(10B)	0.9600
C(10)-H(10C)	0.9600
C(11)-C(12)	1.411(5)
C(11)-H(11)	0.9500

C(12)-C(13)	1.355(4)
C(12)-C(15)	1.470(4)
C(13)-C(14)	1.463(5)
C(16)-C(18)	1.532(5)
C(16)-C(17)#2	1.533(3)
C(16)-C(17)	1.533(3)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-H(19A)	0.9599
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(20)-H(20A)	0.9800
C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800

C(3)-N(1)-N(2)	109.6(3)
C(3)-N(1)-C(10)	132.1(3)
N(2)-N(1)-C(10)	118.3(3)
C(1)-N(2)-N(1)	106.5(3)

C(5)-N(3)-C(4)	110.8(2)
C(5)-N(3)-C(6)	127.5(2)
C(4)-N(3)-C(6)	121.7(2)
C(4)-N(4)-O(2)	108.5(3)
C(13)-N(5)-N(6)	110.3(3)
C(13)-N(5)-C(20)	131.8(3)
N(6)-N(5)-C(20)	117.9(3)
C(11)-N(6)-N(5)	105.9(3)
C(15)-N(7)-C(14)	110.6(3)
C(15)-N(7)-C(16)	127.5(2)
C(14)-N(7)-C(16)	121.9(3)
C(14)-N(8)-O(4)	109.4(3)
C(9)-O(2)-N(4)	108.6(3)
N(8)-O(4)-C(19)	108.8(4)
N(2)-C(1)-C(2)	109.4(3)
N(2)-C(1)-H(1)	125.3
C(2)-C(1)-H(1)	125.3
C(3)-C(2)-C(1)	106.0(3)
C(3)-C(2)-C(5)	109.8(3)
C(1)-C(2)-C(5)	144.2(3)
C(2)-C(3)-N(1)	108.5(3)
C(2)-C(3)-C(4)	109.3(3)
N(1)-C(3)-C(4)	142.2(3)

N(4)-C(4)-N(3)	122.0(3)
N(4)-C(4)-C(3)	132.7(3)
N(3)-C(4)-C(3)	105.3(3)
O(1)-C(5)-N(3)	127.1(3)
O(1)-C(5)-C(2)	128.2(3)
N(3)-C(5)-C(2)	104.7(3)
N(3)-C(6)-C(8)#1	109.04(16)
N(3)-C(6)-C(8)	109.04(16)
C(8)#1-C(6)-C(8)	111.3(3)
N(3)-C(6)-C(7)	111.4(2)
C(8)#1-C(6)-C(7)	108.06(17)
C(8)-C(6)-C(7)	108.07(17)
C(6)-C(7)-H(7A)	109.5
C(6)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(6)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(6)-C(8)-H(8A)	109.5
C(6)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(6)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5

H(8B)-C(8)-H(8C)	109.5
O(2)-C(9)-H(9A)	109.5
O(2)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
O(2)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
N(1)-C(10)-H(10A)	109.5
N(1)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	110.4
N(1)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	108.5
H(10B)-C(10)-H(10C)	109.5
N(6)-C(11)-C(12)	110.3(3)
N(6)-C(11)-H(11)	124.9
C(12)-C(11)-H(11)	124.9
C(13)-C(12)-C(11)	105.4(3)
C(13)-C(12)-C(15)	109.7(3)
C(11)-C(12)-C(15)	144.9(3)
N(5)-C(13)-C(12)	108.2(3)
N(5)-C(13)-C(14)	142.4(3)
C(12)-C(13)-C(14)	109.4(3)
N(8)-C(14)-N(7)	121.8(3)

N(8)-C(14)-C(13)	132.8(3)
N(7)-C(14)-C(13)	105.3(3)
O(3)-C(15)-N(7)	126.9(3)
O(3)-C(15)-C(12)	128.1(3)
N(7)-C(15)-C(12)	105.0(3)
N(7)-C(16)-C(18)	110.7(3)
N(7)-C(16)-C(17)#2	108.62(18)
C(18)-C(16)-C(17)#2	108.4(2)
N(7)-C(16)-C(17)	108.62(18)
C(18)-C(16)-C(17)	108.4(2)
C(17)#2-C(16)-C(17)	112.1(3)
C(16)-C(17)-H(17A)	109.5
C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(16)-C(18)-H(18A)	109.5
C(16)-C(18)-H(18B)	109.4
H(18A)-C(18)-H(18B)	109.5
C(16)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5

O(4)-C(19)-H(19A) 109.4

O(4)-C(19)-H(19B) 109.5

H(19A)-C(19)-H(19B) 110.2

O(4)-C(19)-H(19C) 109.5

H(19A)-C(19)-H(19C) 108.8

H(19B)-C(19)-H(19C) 109.5

N(5)-C(20)-H(20A) 109.5

N(5)-C(20)-H(20B) 109.5

H(20A)-C(20)-H(20B) 109.5

N(5)-C(20)-H(20C) 109.5

H(20A)-C(20)-H(20C) 109.5

H(20B)-C(20)-H(20C) 109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,z #2 -x+1,y,z

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for mo_dm13420_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)29(1)	20(1)	40(2)	-1(1)	0	0
N(2)33(1)	21(1)	53(2)	-12(1)	0	0

N(3)26(1)	16(1)	18(1)	-5(1)	0	0
N(4)34(2)	28(1)	22(1)	2(1)	0	0
N(5)27(1)	30(2)	37(2)	12(1)	0	0
N(6)29(2)	45(2)	29(2)	10(1)	0	0
N(7)26(1)	17(1)	23(1)	-1(1)	0	0
N(8)36(2)	20(1)	48(2)	-6(1)	0	0
O(1)42(1)	35(1)	20(1)	-6(1)	0	0
O(2)69(2)	31(1)	23(1)	6(1)	0	0
O(3)38(1)	17(1)	30(1)	-1(1)	0	0
O(4)75(2)	17(1)	72(2)	-1(1)	0	0
C(1)26(2)	24(2)	41(2)	-13(1)	0	0
C(2)20(2)	23(2)	30(2)	-6(1)	0	0
C(3)22(2)	16(1)	31(2)	-2(1)	0	0
C(4)22(1)	20(1)	23(1)	-1(1)	0	0
C(5)21(1)	23(2)	22(1)	-6(1)	0	0
C(6)31(2)	18(1)	24(2)	-3(1)	0	0
C(7)52(2)	21(2)	28(2)	5(1)	0	0
C(8)36(1)	24(1)	32(1)	-6(1)	-2(1)	-5(1)
C(9)105(4)	49(3)	25(2)	7(2)	0	0
C(10)55(2)	27(2)	49(2)	10(2)	0	0
C(11)24(2)	38(2)	26(2)	0(1)	0	0
C(12)17(1)	25(2)	24(2)	2(1)	0	0
C(13)21(2)	20(2)	31(2)	5(1)	0	0

C(14)21(2)	19(1)	32(2)	0(1)	0	0
C(15)19(1)	22(1)	23(2)	-3(1)	0	0
C(16)35(2)	27(2)	22(1)	-1(1)	0	0
C(17)36(1)	57(2)	28(1)	-9(1)	-8(1)	3(1)
C(18)95(3)	36(2)	24(2)	6(2)	0	0
C(19)75(4)	19(2)	121(5)	-14(2)	0	0
C(20)44(2)	32(2)	68(3)	29(2)	0	0

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mo_dm13420_0m.

	x	y	z	U(eq)
H(1)	0	446	113	36
H(7A)	-16	7289	667	51
H(7B)	-1184	6059	321	51
H(7C)	1200	6075	326	51
H(8A)	1888	5681	2261	46
H(8B)	1908	7040	1811	46
H(8C)	3063	5779	1495	46
H(9A)	935	3937	3808	89

H(9B)	407	2498	4115	89
H(9C)	-1343	3501	3901	89
H(10A)	0	-872	2474	66
H(10B)	-1154	346	2806	66
H(10C)	1181	318	2798	66
H(11)	5000	3655	2221	35
H(17A)	3122	1270	-1166	61
H(17B)	3048	2423	-1768	61
H(17C)	1933	2604	-994	61
H(18A)	4079	4753	-752	78
H(18B)	4608	4456	-1578	78
H(18C)	6314	4664	-999	78
H(19A)	5000	-2768	78	108
H(19B)	6158	-1968	-531	108
H(19C)	3822	-1982	-520	108
H(20A)	6059	-1044	1530	72
H(20B)	3709	-991	1676	72
H(20C)	5232	-750	2342	72

Table 6. Torsion angles [°] for mo_dm13420_0m.

C(3)-N(1)-N(2)-C(1)	0.000(1)

C(10)-N(1)-N(2)-C(1)	180.000(1)
C(13)-N(5)-N(6)-C(11)	0.000(1)
C(20)-N(5)-N(6)-C(11)	180.000(1)
C(4)-N(4)-O(2)-C(9)	180.000(1)
C(14)-N(8)-O(4)-C(19)	180.000(1)
N(1)-N(2)-C(1)-C(2)	0.000(1)
N(2)-C(1)-C(2)-C(3)	0.000(1)
N(2)-C(1)-C(2)-C(5)	180.000(1)
C(1)-C(2)-C(3)-N(1)	0.000(1)
C(5)-C(2)-C(3)-N(1)	180.000(1)
C(1)-C(2)-C(3)-C(4)	180.000(1)
C(5)-C(2)-C(3)-C(4)	0.000(1)
N(2)-N(1)-C(3)-C(2)	0.000(1)
C(10)-N(1)-C(3)-C(2)	180.000(1)
N(2)-N(1)-C(3)-C(4)	180.000(1)
C(10)-N(1)-C(3)-C(4)	0.000(1)
O(2)-N(4)-C(4)-N(3)	180.000(1)
O(2)-N(4)-C(4)-C(3)	0.000(1)
C(5)-N(3)-C(4)-N(4)	180.000(1)
C(6)-N(3)-C(4)-N(4)	0.000(1)
C(5)-N(3)-C(4)-C(3)	0.000(1)
C(6)-N(3)-C(4)-C(3)	180.000(1)
C(2)-C(3)-C(4)-N(4)	180.000(1)

N(1)-C(3)-C(4)-N(4)	0.000(1)
C(2)-C(3)-C(4)-N(3)	0.000(1)
N(1)-C(3)-C(4)-N(3)	180.000(1)
C(4)-N(3)-C(5)-O(1)	180.000(1)
C(6)-N(3)-C(5)-O(1)	0.000(1)
C(4)-N(3)-C(5)-C(2)	0.000(1)
C(6)-N(3)-C(5)-C(2)	180.000(1)
C(3)-C(2)-C(5)-O(1)	180.000(1)
C(1)-C(2)-C(5)-O(1)	0.000(1)
C(3)-C(2)-C(5)-N(3)	0.000(1)
C(1)-C(2)-C(5)-N(3)	180.000(1)
C(5)-N(3)-C(6)-C(8)#1	119.16(18)
C(4)-N(3)-C(6)-C(8)#1	-60.84(18)
C(5)-N(3)-C(6)-C(8)	-119.17(18)
C(4)-N(3)-C(6)-C(8)	60.83(18)
C(5)-N(3)-C(6)-C(7)	0.000(1)
C(4)-N(3)-C(6)-C(7)	180.000(1)
N(5)-N(6)-C(11)-C(12)	0.000(1)
N(6)-C(11)-C(12)-C(13)	0.000(1)
N(6)-C(11)-C(12)-C(15)	180.000(1)
N(6)-N(5)-C(13)-C(12)	0.000(1)
C(20)-N(5)-C(13)-C(12)	180.000(1)
N(6)-N(5)-C(13)-C(14)	180.000(1)

C(20)-N(5)-C(13)-C(14)	0.000(1)
C(11)-C(12)-C(13)-N(5)	0.000(1)
C(15)-C(12)-C(13)-N(5)	180.000(1)
C(11)-C(12)-C(13)-C(14)	180.000(1)
C(15)-C(12)-C(13)-C(14)	0.000(1)
O(4)-N(8)-C(14)-N(7)	180.000(1)
O(4)-N(8)-C(14)-C(13)	0.000(1)
C(15)-N(7)-C(14)-N(8)	180.000(1)
C(16)-N(7)-C(14)-N(8)	0.000(1)
C(15)-N(7)-C(14)-C(13)	0.000(1)
C(16)-N(7)-C(14)-C(13)	180.000(1)
N(5)-C(13)-C(14)-N(8)	0.000(1)
C(12)-C(13)-C(14)-N(8)	180.000(1)
N(5)-C(13)-C(14)-N(7)	180.000(1)
C(12)-C(13)-C(14)-N(7)	0.000(1)
C(14)-N(7)-C(15)-O(3)	180.000(1)
C(16)-N(7)-C(15)-O(3)	0.000(1)
C(14)-N(7)-C(15)-C(12)	0.000(1)
C(16)-N(7)-C(15)-C(12)	180.000(1)
C(13)-C(12)-C(15)-O(3)	180.000(1)
C(11)-C(12)-C(15)-O(3)	0.000(1)
C(13)-C(12)-C(15)-N(7)	0.000(1)
C(11)-C(12)-C(15)-N(7)	180.000(1)

C(15)-N(7)-C(16)-C(18)	0.000(1)
C(14)-N(7)-C(16)-C(18)	180.000(1)
C(15)-N(7)-C(16)-C(17)#2	118.91(19)
C(14)-N(7)-C(16)-C(17)#2	-61.09(19)
C(15)-N(7)-C(16)-C(17)	-118.91(19)
C(14)-N(7)-C(16)-C(17)	61.09(19)

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,z #2 -x+1,y,z

Table 7. Hydrogen bonds for mo_dm13420_0m [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(8)-H(8A)...N(4)	0.98	2.37	3.015(4)	122.7
C(8)-H(8C)...O(3)	0.98	2.59	3.505(3)	156.2
C(17)-H(17A)...N(8)	0.98	2.38	3.017(4)	121.9
C(17)-H(17C)...O(1)	0.98	2.54	3.466(3)	157.0
C(18)-H(18A)...O(3)	0.96	2.19	2.723(5)	113.5
C(19)-H(19A)...O(3)#3	0.96	2.60	3.513(6)	159.1

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,z #2 -x+1,y,z #3 x,y-1,z