Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Palladium-Catalyzed Radical Arylation of *N*-Hydroxyphthalimides with Triphenyl Phosphites as an Unusual Arylating Agent via C(sp^2)-O Bond Cleavage

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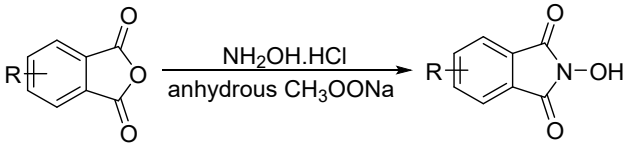
Table of Contents

1-General information

Unless otherwise noted, materials obtained from commercial suppliers are used without further purification. All reactions were performed under air atmosphere except the reaction of the synthesis of **5a** and **6a** complexes were performed under an argon atmosphere. All reactions under standard conditions were monitored by thin–layer chromatography (TLC) on gel F254 plates. Flash column chromatograph was carried out using 300–400 mesh silica gel at medium pressure. ^{1}H NMR and ^{13}C NMR spectra were obtained with an Agilent Technologies 400 or 600 spectrometer. Mass spectra were obtained on a Bruker Dalton maXis instrument. Proton spectra were recorded in $CDCl_3$ or DMSO-d_6 and ^{1}H NMR chemical shifts were referenced to the residual signals of $CHCl_3$ (at δ = 7.26 ppm) and DMSO-d_6 (at δ = 2.50 ppm) respectively. ^{13}C NMR chemical shifts were referenced against the central line of the solvent signal (for $CHCl_3$ at δ = 77.16 ppm and for DMSO-d_6 at δ = 39.52 ppm). Chemical shifts are given on δ scale (ppm). Coupling constants (J) are given in Hz. Multiplicities are indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sext (sextet) or m (multiplet).

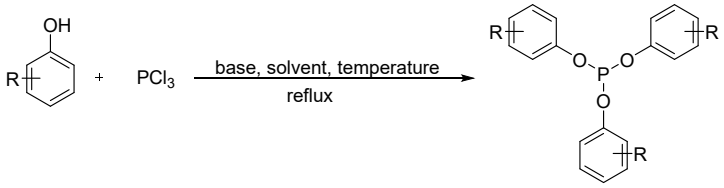
2. Preparation method of the starting material (*N*-hydroxyphthalimide) derivatives

A mixture of hydroxylamine hydrochloride (1.88g, 0.027 mol) and anhydrous sodium acetate (1.80g, 0.022 mol) in glacial acetic acid was refluxed for 5 min. The precipitated sodium chloride was filtered off and the desired 2,3-dicarboxylic acid anhydride (0.02 mol) was added to the filtrate. The mixture was refluxed for further 30 min. The crystalloid obtained on cooling was collected by filtration, dried, and then used directly.[1]



3. Preparation method of the starting material (triphenyl phosphite) derivatives

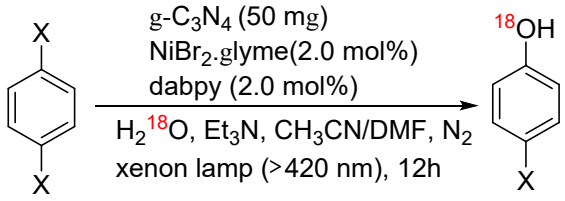
To the solution of appropriate phenol derivatives (50 mmol) and pyridine (4.84 mL, 60 mmol) in dry acetonitrile (200 mL) was added dropwise phosphorus trichloride (1.37 mL, 16 mmol) at room temperature under an inert atmosphere N_2. The obtained mixture was refluxed for 5 h. After cooling the product precipitated or deposited as an oil or solid. It was washed with acetonitrile and dried in dessicator. Phosphites were characterized by their ^{31}P NMR spectra (presence of only a single phosphorus signal) and used directly after synthesis. [2]



4. Preparation of labeled triphenyl phosphite

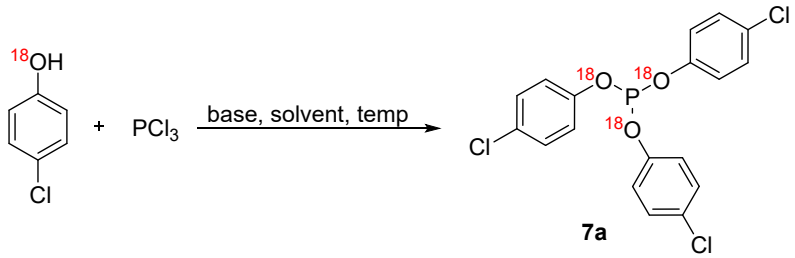
Step 1. Preparation of labeled phenol derivative

In an oven-dried quartz vial (20 × 100 mm) equipped with a stir bar was charged with 1-bromo-4-methylbenzene (85.5 mg, 0.5 mmol, 1.0 equiv.), NiBr2·glyme (2.0 mol%), 2,2′- bipyridine-5,5′-diamine (dabpy, 2.0 mol%) and g-C_3N_4 (50 mg). Then, DMF/CH_3CN (1: 1, 3.0 mL), $H_2{}^{18}O$ (360 μL, 20 equiv.) and Et_3N (0.75 mmol, 1.5 equiv.) were added and the vial was sealed with a rubber plug. The reaction mixture was stirred for 30 min.Thereafter, the mixture was degassed by bubbling nitrogen for 10 min. The reaction mixture was irradiated under a 300 W xenon lamp (>420 nm) at 25 °C with rapid stirring for 24 h. After that, the solvent was removed under vacuum directly. The residue was purified by flash column chromatography on silica gel using (petroleum ether: ethyl acetate 10: 01) as an eluent to afford the desired product.[3]



Step 2. Preparation of labeled triphenyl phosphite derivative

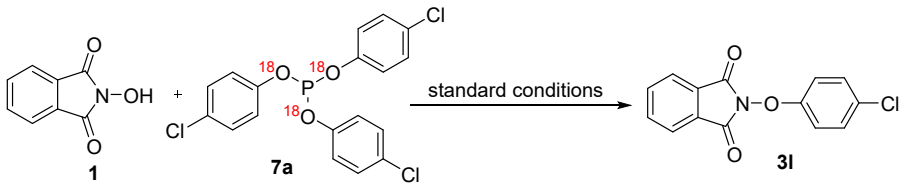
To the solution of appropriate phenol derivatives (50 mmol) and pyridine (4.84 mL, 60 mmol) in dry acetonitrile (200 mL) was added dropwise phosphorus trichloride (1.37 mL, 16 mmol) at room temperature under an inert atmosphere N_2. The obtained mixture was refluxed for 5 h. After cooling the product precipitated or deposited as an oil or solid. It was washed with acetonitrile and dried in desiccator. Phosphites were characterized by their ^{31}P NMR spectra (presence of only a single phosphorus signal) and used directly after synthesis.



7a Yellowish oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.26 – 7.11 (m, 4H), 7.01 (s, 4H), 6.82 – 6.74 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 153.7, 129.6, 125.9, 116.8. **^{31}P NMR ($CDCl_3$,162 MHz)** δ 127.6 ppm. **HRMS m/z (ESI)**: calcd for $C_{18}H_{12}Cl_3{}^{18}O_3P$, $[M+H]^+$ 440.9609; found to be, 440.9599.

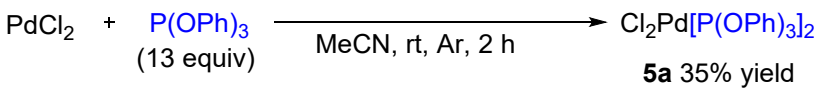
5. Distinguish of oxygen atom in the target product

When compound **7a** was used as the starting material to react with **1** under the standard conditions of the reaction system, the resulting product was then tested by HRMS, showing that no labelled oxygen atom was involved in the formed product. Indicating that the oxygen atom in the target product is coming from N-hydroxyphthalimide **1** rather than triphenyl phosphite **2**.



6. Preparation of complex 5a

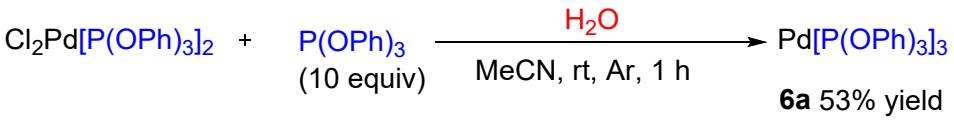
The reaction was performed under argon atmosphere by using standard Schlenck techniques. To a solution of $PdCl_2$ (53.2 mg, 0.3 mmol) in benzene (5 mL) was added $P(OPh)_3$ (1.21 g, 3.9 mmol). The resulting mixture was stirred for 3 h at rt. The reaction mixture was then settled out without stirring for 1 h. The target catalyst was gradually precipitated as a white solid in the reaction mixture. The target complex was filtered off and was washed with ethyl ether (3 mL x 2). The resulting complex was dried in vacuo affording $Cl_2Pd[P(OPh)_3]_2$ as an off-white solid (85 mg, 35% yield).[4]



5a (dichloro-bis-(triphenylphosphite) palladium (II), white solid 35% yield. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.28 (dd, *J* = 8.6, 6.8 Hz, 12H), 7.27 – 7.18 (m, 6H), 7.14 (d, *J* = 8.3 Hz, 12H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 150.5, 150.5, 150.4, 130.0, 125.9, 120.7, 120.7, 120.7. **^{31}P NMR ($CDCl_3$, 162 MHz)** δ 83.6 ppm. The spectrum data were in accordance with the literature.[4]

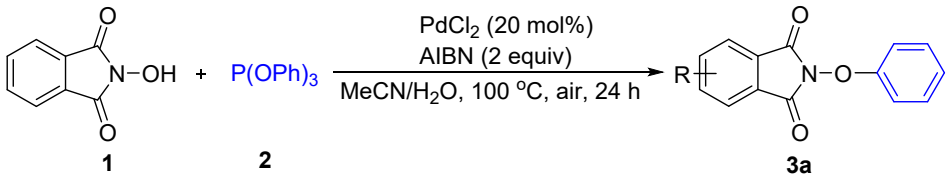
7. Preparation of complex 6a

The reaction was performed under argon atmosphere by using standard Schlenck techniques. To a solution of $Cl_2Pd[P(OPh)_3]_2$ (239.4 mg, 0.3 mmol) in benzene (5 mL) was added $P(OPh)_3$ (931 mg, 3.0 mmol). Then 100μ of distilled water was added to the formed solution. The resulting mixture was stirred for 1 h at rt. The reaction mixture was then settled out without stirring for 1 h. The target catalyst was gradually precipitated as a white solid in the reaction mixture. The target complex was filtered off and was washed with CH_3CN (3 mL x 2). The resulting complex was dried in vacuo affording $Cl_2Pd[P(OPh)_3]_2$ as an off-white solid (164 mg, 53% yield).[4]



6a (tris-(triphenylphosphite) palladium (0), white solid 53% yield. **^{1}H NMR (400 MHz, C_6D_6)** δ 7.04 (d, *J* = 7.9 Hz, 18H), 6.99 (t, *J* = 8.0 Hz, 18H), 6.83 (t, *J* = 7.3 Hz, 9H). **^{13}C NMR (101 MHz, C_6D_6) ^{13}C NMR (101 MHz, C_6D_6)** δ 152.0, 129.3, 127.9, 127.7, 127.4, 123.8, 121.2. **^{31}P NMR (C_6D_6, 202 MHz,)** δ 137.8 ppm. The spectrum data were in accordance with the literature.[5]

8. Table S1. Optimization of the reaction study[a]



1 2 3a

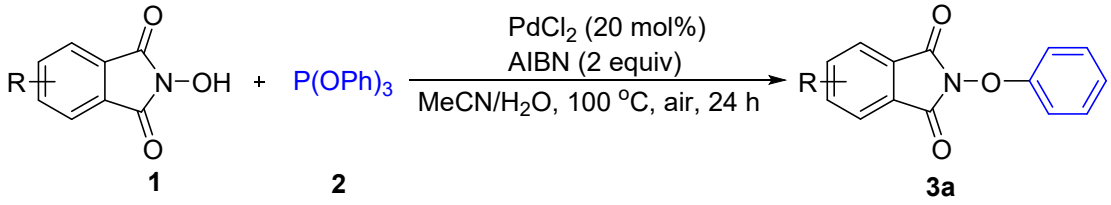
Entry	Catalyst mol%	Equiv of $P(OPh)_3$	Equiv of AIBN	Solvent	Temp °C	Yield%[b]
1	-	2.0	2.0	DCE	rt	0
2	CuCl 10	2.0	2.0	DCE	50	0
3	CuCl 10	2.0	2.0	DCE	80	0
4	$Ni(acac)_2$ 10	2.0	2.0	DCE	80	0
5	$Ni(acac)_2$ 10	3.0	2.0	DCE	80	0
6	$Ni(acac)_2$ 10	4.0	2.0	DCE	80	trace
7	$PdCl_2$ 5	4.0	-	DCE	80	0
8	$PdCl_2$ 5	4.0	2.0	MeCN	80	trace
9	$PdCl_2$ 10	5.0	2.0	MeCN	80	trace
10	$PdCl_2$ 10	6.0	2.0	MeCN	80	trace
11	$PdCl_2$ 20	6.0	2.0	MeCN	100	55
12	$PdCl_2$ 20	6.0	-	MeCN	100	35
13	$PdCl_2$ 20	6.0	2.0	MeCN/100μ H_2O	100	86
14	$PdCl_2$ 20	6.0	-	MeCN/100μ H_2O	100	77
15	$PdCl_2$ 20	5.0	2.0	MeCN/100μ H_2O	100	78
16	$PdCl_2$ 20	4.0	2.0	MeCN/100μ H_2O	100	69
18	$PdCl_2$ 20	3.0	2.0	MeCN/100μ H_2O	100	58
19	$PdCl_2$ 20	7.0	2.0	MeCN/100μ H_2O	100	87
20	$PdCl_2$ 20	6.0	1.0	MeCN/100μ H_2O	100	79
21	$Pd(dba)_3$ 10	6.0	2.0	MeCN/100μ H_2O	100	79
22	$Pd(OAc)_2$ 10	6.0	2.0	MeCN/100μ H_2O	100	67
23	$PdCl_2$ 20	6.0	2.0	DCE/100μ H_2O	100	80
24	$PdCl_2$ 20	6.0	2.0	THF/100μ H_2O	100	76
25	$PdCl_2$ 20	6.0	2.0	toluene/100μ H_2O	100	16
26[c]	$PdCl_2$ 20	6.0	2.0	MeCN/100μ H_2O	100	78
27[d]	$PdCl_2$ 20	6.0	2.0	MeCN/100μ H_2O	100	65

[a]Reaction conditions. [b]Isolated yield after chromatography. [c]Reaction under O_2. [d]Reaction under N_2

9. General experimental procedure for the Palladium-Catalyzed Radical Arylation of *N*-Hydroxyphthalimides with Triphenyl Phosphites as an Unusual Arylating Agent via C(sp^2)-O Bond Cleavage

3a as an example:

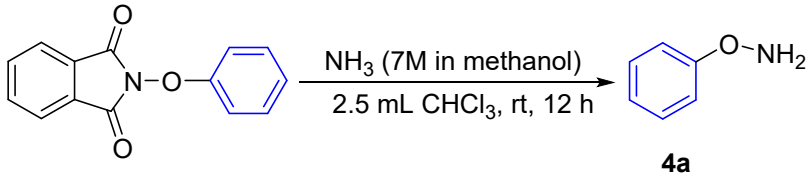
To a solution of *N*-hydroxyphthalimide (NHPI) **1** (0.3 mmol, 49 mg, 1.0 equiv), 2,2'-Azobis(2-methylpropionitrile) (AIBN) (0.6 mmol, 98.5 mg, 2.0 equiv), $PdCl_2$ (20 mol%, 10.6 mg), in acetonitrile (2.0 mL), triphenyl phosphite (TPP) **2** (1.8 mmol, 558.5 mg, 6.0 equiv), and distilled water (100 μL) were added in a sealed tube equipped with a magnetic stirrer bar. The sealed tube was then tightly sealed with a screw cap, and the reaction system was stirred for 24 h at 100 °C in an air atmosphere. The reaction mixture was then cooled to room temperature. After the solvent was removed under vacuum directly, the residue was purified by flash column chromatography on silica gel using petroleum ether: ethyl acetate (05:01) as a gradient eluent to afford the desired product **3a**.



1 **2** **3a**

10. Hydrolysis of *N*-aryloxyimide to Aryloxamine

To a solution of **3a** (0.25 mmol, 60 mg) in 2.5 mL $CHCl_3$ under stirring conditions, NH_3(1.0 mL, 7N in methanol) was added dropwise at room temperature. Stirring was continued for 12 h and then concentrated under reduced pressure. After that the crude reaction mixture was adsorbed to silica and transfer to a plug of silica gel, washing with 20% Et_2O in pentane (100 mL). Removal of solvent under reduced pressure afforded yellowish oil **4a** in 91%yield.



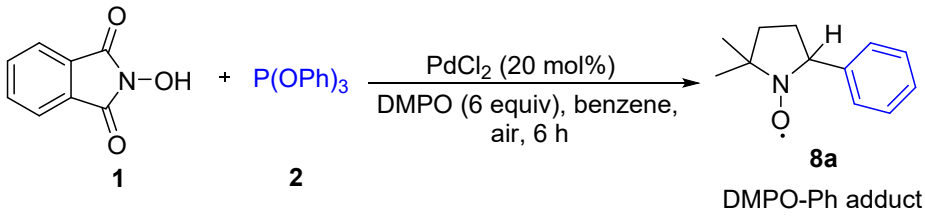
3a **4a**
90% yield

11. EPR Experiments

General

EPR experiments were carried out using (Bruker BioSpin GmbH). EPR spectrometer was used with the following instrumental parameters: microwave frequency = 9.849 GHz; incident microwave power 20 Mw; Center Field: 3508 G; scan range 200 G; modulation amplitude = 1 G; receiver gain: 1.00e+003; and time constant = 163.84 ms.

To a solution of *N*-hydroxyphthalimide **1** (0.3 mmol, 49 mg, 1.0 equiv), $PdCl_2$ (20 mol%, 10.6 mg), in benzene (2.0 mL), triphenyl phosphite **2** (1.8 mmol, 558.5 mg, 6.0 equiv), and 5,5-dimethyl-1-pyrroline N-oxide (DMPO) (1.8 mmol, 203.7 mg, 6.0 equiv) were added in a sealed tube. The sealed tube was then tightly sealed with a screw cap, and the reaction system was stirred for 6 hours at 100 °C in an air atmosphere. Then the solution was taken out into a small tube and was analyzed by EPR.



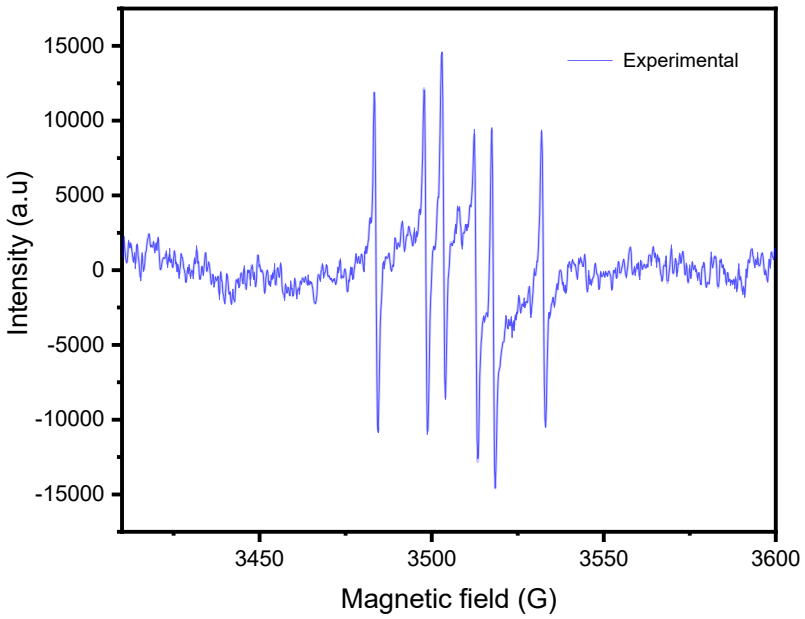
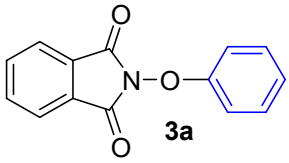


Figure S1. EPR spectrum of the spin adduct **8a** generated upon the above-mentioned reaction conditions

12. Analytical data of products

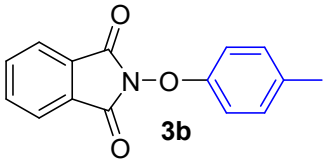


3a

2-phenoxyisoindoline-1,3-dione

3a was synthesized according to the above mentioned general experimental procedure in section 7. White solid (62 mg, 86% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.37 – 7.27 (m, 2H), 7.17 (d, *J* = 1.3 Hz, 1H), 7.18 – 7.08 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.9, 158.8, 134.9, 129.7, 128.8, 124.6, 124.0, 114.4. All spectral data correspond to those given in the literature.[6]

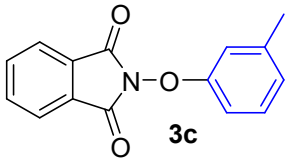


3b

2-(p-tolyloxy)isoindoline-1,3-dione

3b was synthesized according to the above mentioned general experimental procedure in section 7. White solid (68.5 mg, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.16 – 7.03 (m, 4H), 2.30 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 156.9, 134.8, 134.3, 130.1, 128.8, 123.9, 114.7, 20.6. All spectral data correspond to those given in the literature.[6]

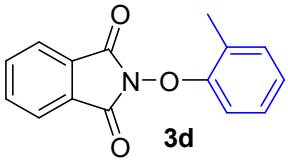


3c

2-(m-tolyloxy)isoindoline-1,3-dione

3c was synthesized according to the above mentioned general experimental procedure in section 7. White solid (64 mg, 84% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.20 (t, *J* = 7.8 Hz, 1H), 7.00 – 6.90 (m, 3H), 2.32 (d, *J* = 0.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 158.9, 140.1, 134.9, 129.4, 128.8, 125.4, 123.9, 114.9, 111.3, 21.4. All spectral data correspond to those given in the literature.[6]

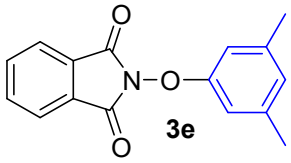


3d

2-(o-tolyloxy)isoindoline-1,3-dione

3d was synthesized according to the above mentioned general experimental procedure in section 7. White solid (56 mg, 74% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.20 (ddd, *J* = 7.3, 1.8, 0.9 Hz, 1H), 7.10 (td, *J* = 7.8, 1.8 Hz, 1H), 7.02 (td, *J* = 7.4, 1.3 Hz, 1H), 6.93 (dd, *J* = 8.2, 1.2 Hz, 1H), 2.47 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 157.0, 134.8, 131.5, 128.9, 127.0, 126.0, 124.3, 123.9, 112.7, 15.8. All spectral data correspond to those given in the literature.[6]

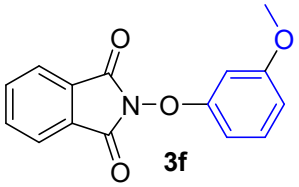


3e

2-(3,5-dimethylphenoxy)isoindoline-1,3-dione

3e was synthesized according to the above mentioned general experimental procedure in section 7. White solid (71 mg,88% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.78 – 6.73 (m, 3H), 2.27 (d, *J* = 0.8 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 158.9, 139.7, 134.8, 128.8, 126.3, 123.9, 111.8, 21.3. All spectral data correspond to those given in the literature.[6]



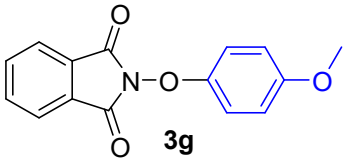
3f

2-(3-methoxyphenoxy)isoindoline-1,3-dione

3f was synthesized according to the above mentioned general experimental procedure in section 7. White solid (53.5 mg, 66% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.19 (t, *J* = 8.6 Hz, 1H), 6.73 – 6.66 (m, 2H), 6.64 (ddd, *J* = 8.2, 2.2, 0.9 Hz, 1H), 3.75 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)**

δ 162.8, 160.8, 159.9, 134.9, 130.2, 128.7, 123.9, 110.1, 106.0, 100.6, 55.5, 55.4. All spectral data correspond to those given in the literature.[6]

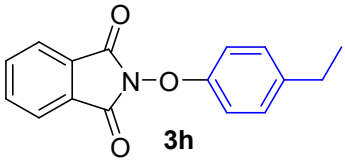


3g

2-(4-methoxyphenoxy)isoindoline-1,3-dione

3g was synthesized according to the above mentioned general experimental procedure in section 7. White solid (58 mg, 72% yield).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.96 – 7.81 (m, 4H), 7.23 – 7.14 (m, 2H), 6.98 – 6.84 (m, 2H), 3.70 (s, 3H).^{13}C NMR (101 MHz, **DMSO-d_6)** δ 169.7, 163.4, 156.4, 152.9, 135.5, 135.0, 134.7, 133.0, 132.4, 129.0, 124.1, 123.7, 123.3, 115.7, 115.1, 55.9, 55.9. All spectral data correspond to those given in the literature.[6]

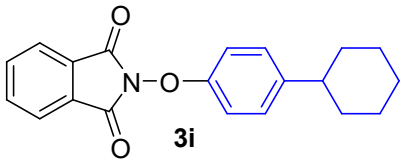


3h

2-(4-ethylphenoxy)isoindoline-1,3-dione

3h was synthesized according to the above mentioned general experimental procedure in section 7. White solid (55.5 mg, 69% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.84 (m, 2H), 7.83 – 7.75 (m, 2H), 7.18 – 7.06 (m, 4H), 2.60 (q, *J* = 7.6 Hz, 2H), 1.19 (td, *J* = 7.5, 0.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 157.0, 140.7, 134.8, 129.0, 128.8, 123.9, 114.8, 28.1, 15.7. **HRMS m/z (ESI)**: calcd for $C_{16}H_{13}NO_3$, $[M+H]^+$ 268.0968; found to be 268.0959.

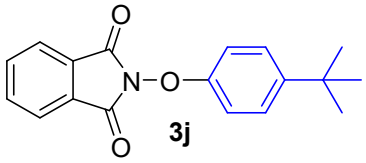


3i

2-(4-cyclohexylphenoxy)isoindoline-1,3-dione

3i was synthesized according to the above mentioned general experimental procedure in section 7. White solid (74.5 mg, 77% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (td, *J* = 7.5, 6.3, 2.9 Hz, 2H), 7.78 (td, *J* = 7.5, 6.3, 3.1 Hz, 2H), 7.18 – 7.04 (m, 4H), 1.82 (d, *J* = 9.2 Hz, 4H), 1.71 (dd, *J* = 10.5, 5.7 Hz, 1H), 1.35 (tt, *J* = 11.0, 5.6 Hz, 4H), 1.23 (s, 2H), 1.23 (d, *J* = 17.2 Hz, 0H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 157.0, 144.6, 134.8, 128.8, 127.9, 127.9, 123.9, 114.6, 114.6, 43.8, 34.5, 34.5, 26.8, 26.8, 26.1. **HRMS m/z (ESI)**: calcd for $C_{20}H_{19}NO_3$, $[M+H]^+$ 322.1435; found to be 322.1438.

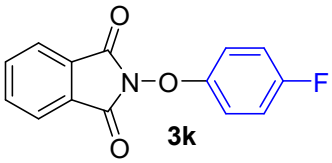


3j

2-(4-(tert-butyl)phenoxy)isoindoline-1,3-dione

3j was synthesized according to the above mentioned general experimental procedure in section 7. White solid (73 mg, 82% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.37 – 7.29 (m, 2H), 7.14 – 7.06 (m, 2H), 1.27 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.0, 156.7, 147.6, 134.8, 128.8, 126.6, 123.9, 114.2, 34.3, 31.4. All spectral data correspond to those given in the literature.[6]

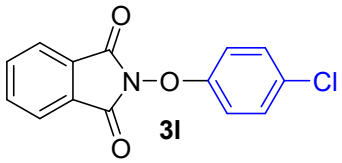


3k

2-(4-fluorophenoxy)isoindoline-1,3-dione

3k was synthesized according to the above mentioned general experimental procedure in section 7. White solid (54 mg, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.24 – 7.14 (m, 2H), 7.05 – 6.94 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.9, 160.8, 158.4, 154.9, 154.9, 134.9, 128.7, 124.0, 117.1, 117.1, 116.4, 116.1. All spectral data correspond to those given in the literature.[6]

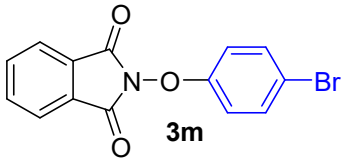


3l

2-(4-chlorophenoxy)isoindoline-1,3-dione

3l was synthesized according to the above mentioned general experimental procedure in section 7. White solid (66 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.32 – 7.24 (m, 2H), 7.16 – 7.07 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.8, 157.5, 135.0, 129.8, 129.7, 128.7, 124.1, 116.2. All spectral data correspond to those given in the literature.[7]

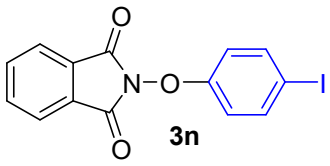


3m

2-(4-bromophenoxy)isoindoline-1,3-dione

3m was synthesized according to the above mentioned general experimental procedure in section 7. White solid (78 mg, 82% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.46 – 7.38 (m, 2H), 7.10 – 7.01 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.8, 158.0, 135.0, 132.6, 128.7, 124.1, 117.2, 116.5. All spectral data correspond to those given in the literature.[6]

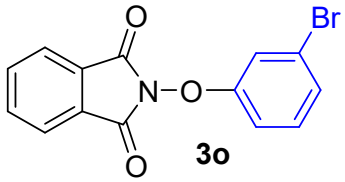


3n

2-(4-iodophenoxy)isoindoline-1,3-dione

3n was synthesized according to the above mentioned general experimental procedure in section 7. White solid (55 mg, 50% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.65 – 7.56 (m, 2H), 6.97 – 6.89 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.7, 158.9, 138.6, 135.0, 128.7, 124.1, 116.7, 87.6. All spectral data correspond to those given in the literature.[7]

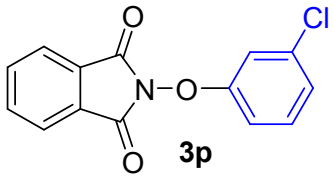


3o

2-(3-bromophenoxy)isoindoline-1,3-dione

3o was synthesized according to the above mentioned general experimental procedure in section 7. White solid (74.5 mg, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.82 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.33 – 7.21 (m, 2H), 7.20 (t, *J* = 8.1 Hz, 1H), 7.11 (ddd, *J* = 8.2, 2.5, 1.1 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.7, 159.3, 135.1, 130.9, 128.7, 127.8, 124.1, 122.9, 117.7, 113.3. All spectral data correspond to those given in the literature.[6]

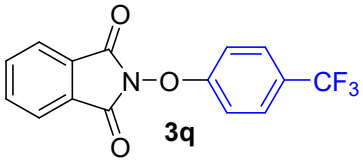


3p

2-(3-chlorophenoxy)isoindoline-1,3-dione

3p was synthesized according to the above mentioned general experimental procedure in section 7. White solid (62 mg, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.82 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.26 (t, *J* = 8.2 Hz, 1H), 7.18 – 7.03 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.7, 159.3, 135.2, 135.1, 130.6, 128.7, 124.9, 124.1, 114.9, 112.7. All spectral data correspond to those given in the literature.[7]

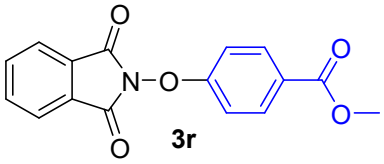


3q

2-(4-(trifluoromethyl)phenoxy)isoindoline-1,3-dione

3q was synthesized according to the above mentioned general experimental procedure in section 7. White solid (60 mg, 65% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.82 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.64 – 7.56 (m, 2H), 7.27 – 7.19 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.6, 161.0, 161.0, 135.1, 128.7, 127.3, 127.3, 127.3, 127.2, 126.9, 126.6, 126.3, 125.2, 124.2, 122.5, 114.2. All spectral data correspond to those given in the literature.[6]

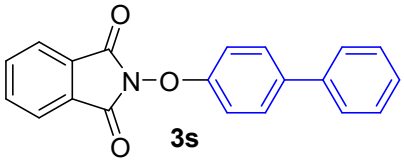


3r

methyl 4-((1,3-dioxoisoindolin-2-yl)oxy)benzoate

3r was synthesized according to the above mentioned general experimental procedure in section 7. White solid (57 mg, 64% yield).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.99 – 7.86 (m, 2H), 7.79 (s, 2H), 7.77, 7.44 – 7.36 (m, 2H), 6.86 – 6.78 (m, 2H), 3.78 (d, *J* = 22.5 Hz, 3H). **^{13}C NMR (101 MHz, DMSO-d_6)** δ 169.7, 166.5, 165.8, 163.1, 162.4, 162.4, 135.6, 134.7, 133.0, 131.9, 131.8, 129.2, 125.9, 124.2, 123.3, 120.7, 115.8, 113.7, 52.6, 52.5, 52.0, 52.0. All spectral data correspond to those given in the literature.[7]

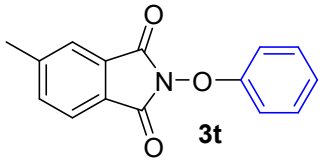


3s

2-([1,1'-biphenyl]-4-yloxy)isoindoline-1,3-dione

3s was synthesized according to the above mentioned general experimental procedure in section 7. White solid (45 mg, 47% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.87 – 7.76 (m, 2H), 7.59 – 7.48 (m, 4H), 7.46 – 7.38 (m, 2H), 7.38 – 7.29 (m, 1H), 7.33 – 7.21 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.9, 158.4, 140.2, 137.9, 134.9, 128.8, 128.5, 127.3, 127.0, 124.0, 114.8. **HRMS m/z (ESI)**: calcd for $C_{20}H_{13}NO_3$, $[M+H]^+$ 316.0968; found to be, 316.0961.

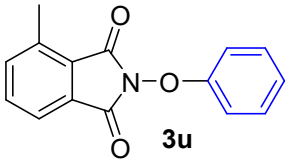


3t

5-methyl-2-phenoxyisoindoline-1,3-dione

3t was synthesized according to the above mentioned general experimental procedure in section 7. White solid (65 mg, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.7 Hz, 1H), 7.74 – 7.68 (m, 1H), 7.58 (ddd, *J* = 7.6, 1.6, 0.8 Hz, 1H), 7.37 – 7.27 (m, 2H), 7.16 (q, *J* = 1.4, 0.9 Hz, 1H), 7.18 – 7.07 (m, 2H), 2.53 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.2, 163.1, 158.9, 146.4, 135.4, 129.7, 129.1, 126.1, 124.5, 123.9, 114.3, 22.2. **HRMS m/z (ESI)**: calcd for $C_{15}H_{11}NO_3$, $[M+H]^+$ 254.0812; found to be, 254.0805.

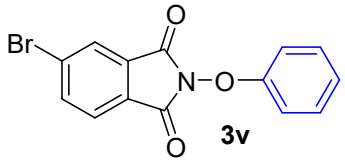


3u

4-methyl-2-phenoxyisoindoline-1,3-dione

3u was synthesized according to the above mentioned general experimental procedure in section 7. White solid (61 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 7.4, 1.0 Hz, 1H), 7.67 – 7.41 (m, 2H), 7.35 – 7.27 (m, 2H), 7.19 – 7.11 (m, 2H), 7.10 (td, *J* = 7.2, 1.1 Hz, 1H), 2.67 (d, *J* = 8.9 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.1, 168.3, 163.7, 163.0, 158.9, 138.8, 138.3, 137.3, 136.6, 134.4, 133.8, 133.1, 129.7, 129.3, 129.1, 125.4, 124.5, 121.6, 121.1, 114.4, 17.7, 17.5. **HRMS m/z (ESI)**: calcd for $C_{15}H_{11}NO_3$, $[M+H]^+$ 254.0812; found to be, 254.0805.

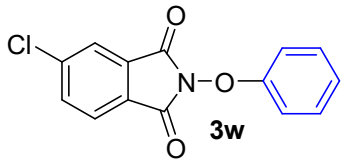


3v

5-bromo-2-phenoxyisoindoline-1,3-dione

3v was synthesized according to the above mentioned general experimental procedure in section 7. White solid (68 mg, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 1.6 Hz, 1H), 7.93 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.76 (dd, *J* = 8.0, 0.5 Hz, 1H), 7.38 – 7.28 (m, 2H), 7.19 – 7.09 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.2, 161.6, 158.7, 137.9, 130.3, 129.9, 129.8, 127.3, 127.3, 127.2, 125.4, 124.8, 114.5. **HRMS m/z (ESI)**: calcd for $C_{14}H_8BrNO_3$, $[M+H]^+$ 317.9760; found to be, 317.9752.

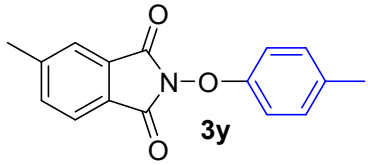


3w

5-chloro-2-phenoxyisoindoline-1,3-dione

3w was synthesized according to the above mentioned general experimental procedure in section 7. White solid (62.5 mg, 76% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.80 (m, 2H), 7.76 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.38 – 7.28 (m, 2H), 7.19 – 7.09 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.0, 161.7, 158.7, 141.7, 134.9, 130.4, 129.8, 126.8, 125.3, 124.8, 124.4, 124.4, 114.5. **HRMS m/z (ESI)**: calcd for $C_{14}H_8ClNO_3$, $[M+ H]^+$ 274.0265; found to be, 274.0258.

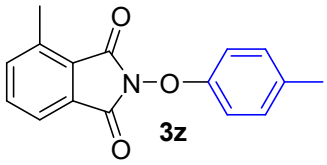


3y

5-methyl-2-(p-tolyloxy)isoindoline-1,3-dione

3y was synthesized according to the above mentioned general experimental procedure in section 7. White solid (77 mg, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.7 Hz, 1H), 7.68 (dt, *J* = 1.5, 0.7 Hz, 1H), 7.56 (ddd, *J* = 7.7, 1.6, 0.8 Hz, 1H), 7.15 – 7.02 (m, 4H), 2.51 (s, 3H), 2.29 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.2, 163.1, 157.0, 146.3, 135.3, 134.2, 130.1, 129.1, 126.1, 124.4, 123.9, 114.6, 22.1, 20.6, 20.6. **HRMS m/z (ESI)**: calcd for $C_{16}H_{13}NO_3$, $[M+H]^+$ 268.0968; found to be, 268.0961.

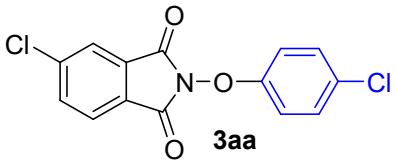


3z

4-methyl-2-(p-tolyloxy)isoindoline-1,3-dione

3z was synthesized according to the above mentioned general experimental procedure in section 7. White solid (73 mg, 91% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.56 (m, 2H), 7.52 (dt, *J* = 7.8, 0.9 Hz, 1H), 7.15 – 7.04 (m, 4H), 2.70 (s, 3H), 2.30 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.8, 163.1, 157.0, 138.8, 137.2, 134.2, 130.1, 129.2, 125.5, 121.5, 114.8, 20.6, 20.6, 17.7, 17.6. **HRMS m/z (ESI)**: calcd for $C_{16}H_{13}NO_3$, $[M+H]^+$ 268.0968; found to be, 268.0961.

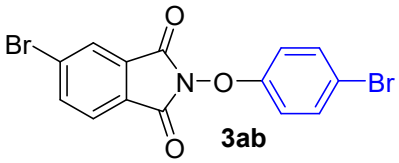


3aa

5-chloro-2-(4-chlorophenoxy)isoindoline-1,3-dione

3aa was synthesized according to the above mentioned general experimental procedure in section 7. White solid (58 mg, 63% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.82 (m, 2H), 7.77 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.37 – 7.18 (m, 2H), 7.18 – 7.08 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.9, 161.6, 157.3, 141.9, 135.1, 130.3, 130.1, 129.7, 126.7, 125.4, 124.5, 116.4. **HRMS m/z (ESI)**: calcd for $C_{14}H_7Cl_2NO_3$, $[M+H]^+$ 307.9876; found to be, 307.9870.

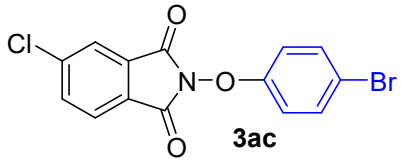


3ab

5-bromo-2-(4-bromophenoxy)isoindoline-1,3-dione

3ab was synthesized according to the above mentioned general experimental procedure in section 7. White solid (80 mg, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 1.7, 0.5 Hz, 1H), 7.95 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.77 (dd, *J* = 8.0, 0.6 Hz, 1H), 7.48 – 7.40 (m, 2H), 7.10 – 7.01 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.0, 161.5, 157.9, 138.0, 132.7, 130.2, 130.1, 127.4, 127.1, 125.4, 117.5, 116.6. **HRMS m/z (ESI)**: calcd for $C_{14}H_7Br_2NO_3$, $[M+H]^+$ 397.8845; found to be, 397.8840.

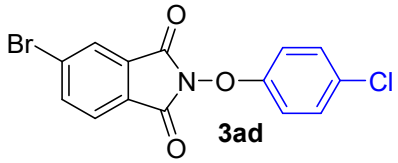


3ac

2-(4-bromophenoxy)-5-chloroisoindoline-1,3-dione

3ac was synthesized according to the above mentioned general experimental procedure in section 7. White solid (76 mg, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.78 (m, 2H), 7.77 (dd, *J* = 8.0, 1.9 Hz, 1H), 7.48 – 7.40 (m, 2H), 7.10 – 7.01 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.9, 161.6, 157.9, 154.8, 141.9, 135.1, 132.7, 132.4, 130.3, 126.6, 125.4, 124.5, 117.5, 117.2, 116.6. **HRMS m/z (ESI)**: calcd for $C_{14}H_7BrClNO_3$, $[M+H]^+$ 351.9371; found to be, 351.9365.

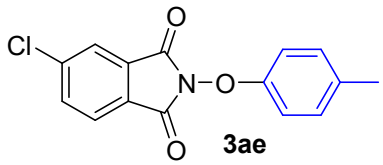


3ad

5-bromo-2-(4-chlorophenoxy)isoindoline-1,3-dione

3ad was synthesized according to the above mentioned general experimental procedure in section 7. White solid (72 mg, 68% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 1.8, 0.5 Hz, 1H), 7.95 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.77 (dd, *J* = 7.9, 0.5 Hz, 1H), 7.34 – 7.25 (m, 2H), 7.16 – 7.07 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.0, 161.5, 157.3, 138.0, 130.2, 130.1, 130.1, 129.7, 127.4, 127.1, 125.4, 116.4. **HRMS m/z (ESI)**: calcd for $C_{14}H_7BrClNO_3$, $[M+H]^+$ 351.9371; found to be, 351.9365.

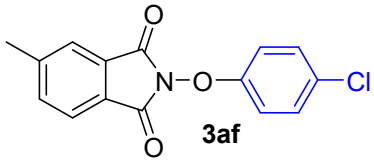


3ae

5-chloro-2-(p-tolyloxy)isoindoline-1,3-dione

3ae was synthesized according to the above mentioned general experimental procedure in section 7. White solid (66.5 mg, 77% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.80 (m, 2H), 7.75 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.16 – 7.03 (m, 4H), 2.30 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.1, 161.8, 156.8, 141.6, 134.8, 134.6, 130.4, 130.1, 126.8, 125.2, 124.4, 124.3, 114.9, 20.6, 20.6. **HRMS m/z (ESI)**: calcd for $C_{15}H_{10}ClNO_3$, $[M+H]^+$ 288.0422; found to be, 288.0415.

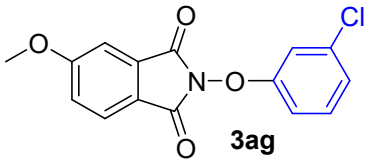


3af

2-(4-chlorophenoxy)-5-methylisoindoline-1,3-dione

3af was synthesized according to the above mentioned general experimental procedure in section 7. White solid (75 mg, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.7 Hz, 1H), 7.71 (dt, *J* = 1.5, 0.8 Hz, 1H), 7.60 (ddd, *J* = 7.6, 1.6, 0.8 Hz, 1H), 7.26 (t, *J* = 8.1 Hz, 1H), 7.17 – 7.08 (m, 2H), 7.05 (ddd, *J* = 8.4, 2.5, 1.0 Hz, 1H), 2.54 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.1, 162.9, 157.5, 146.6, 135.5, 129.7, 129.6, 129.0, 126.0, 124.6, 124.0, 116.1, 22.2. **HRMS m/z (ESI)**: calcd for $C_{15}H_{10}ClNO_3$, $[M+H]^+$ 288.0422; found to be, 288.0415.

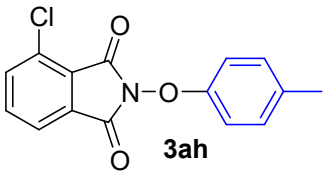


3ag

2-(3-chlorophenoxy)-5-methoxyisoindoline-1,3-dione

3ag was synthesized according to the above mentioned general experimental procedure in section 7. White solid (67.5 mg, 74% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.38 (dd, *J* = 2.4, 0.9 Hz, 1H), 7.30 – 7.19 (m, 2H), 7.17 – 7.11 (m, 1H), 7.08 (dddt, *J* = 20.8, 8.3, 2.5, 1.0 Hz, 2H), 3.94 (d, *J* = 1.0 Hz, 3H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 165.4, 162.9, 162.7, 159.5, 135.2, 131.3, 130.5, 126.1, 124.7, 120.6, 120.3, 114.8, 112.6, 109.1, 56.2, 56.2. **HRMS m/z (ESI)**: calcd for $C_{15}H_{10}ClNO_4$, $[M+H]^+$ 303.0298; found to be, 303.0295.

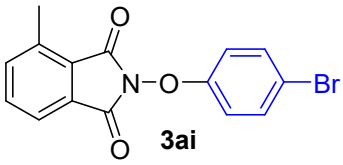


3ah

4-chloro-2-(p-tolyloxy)isoindoline-1,3-dione

3ah was synthesized according to the above mentioned general experimental procedure in section 7. White solid (60 mg, 69% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 4.6, 3.7 Hz, 1H), 7.77 – 7.64 (m, 2H), 7.16 – 7.06 (m, 4H), 2.30 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.6, 160.7, 156.8, 136.5, 135.6, 134.7, 132.1, 130.8, 130.1, 124.7, 122.3, 115.3, 20.6. **HRMS m/z (ESI)**: calcd for $C_{15}H_{10}ClNO_3$, $[M+ H]^+$ 288.0422; found to be, 288.0413.

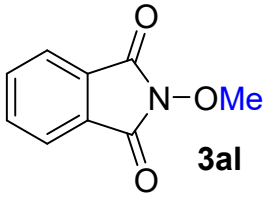


3ai

2-(4-bromophenoxy)-4-methylisoindoline-1,3-dione

3ai was synthesized according to the above mentioned general experimental procedure in section 7. White solid (77.5 mg, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.60 (m, 2H), 7.55 (m, 1H), 7.50 – 7.38 (m, 2H), 7.11 – 7.02 (m, 2H), 2.68 (d, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.6, 162.9, 158.1, 139.0, 137.4, 134.5, 132.6, 129.1, 125.4, 121.7, 121.7, 117.1, 116.5, 17.7, 17.7. **HRMS m/z (ESI)**: calcd for $C_{15}H_{10}BrNO_3$, $[M+H]^+$ 331.9917; found to be, 331.9911.

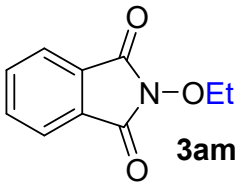


3al

2-methoxyisoindoline-1,3-dione

3al was synthesized according to the above mentioned general experimental procedure in section 7. White solid (49.5 mg, 93% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.67 (m, 4H), 4.09 – 3.99 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.3, 134.5, 128.8, 123.5, 65.8. All spectral data correspond to those given in the literature.[8]

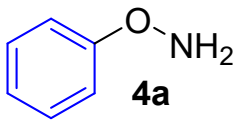


3am

2-ethoxyisoindoline-1,3-dione

3am was synthesized according to the above mentioned general experimental procedure in section 7. White solid (44 mg, 83% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (td, *J* = 5.2, 3.4 Hz, 2H), 7.73 (qd, *J* = 5.2, 2.4 Hz, 2H), 4.25 (qd, *J* = 7.0, 1.4 Hz, 2H), 1.39 (td, *J* = 7.1, 1.4 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.7, 134.4, 128.9, 123.5, 74.1, 13.6. All spectral data correspond to those given in the literature.[8]



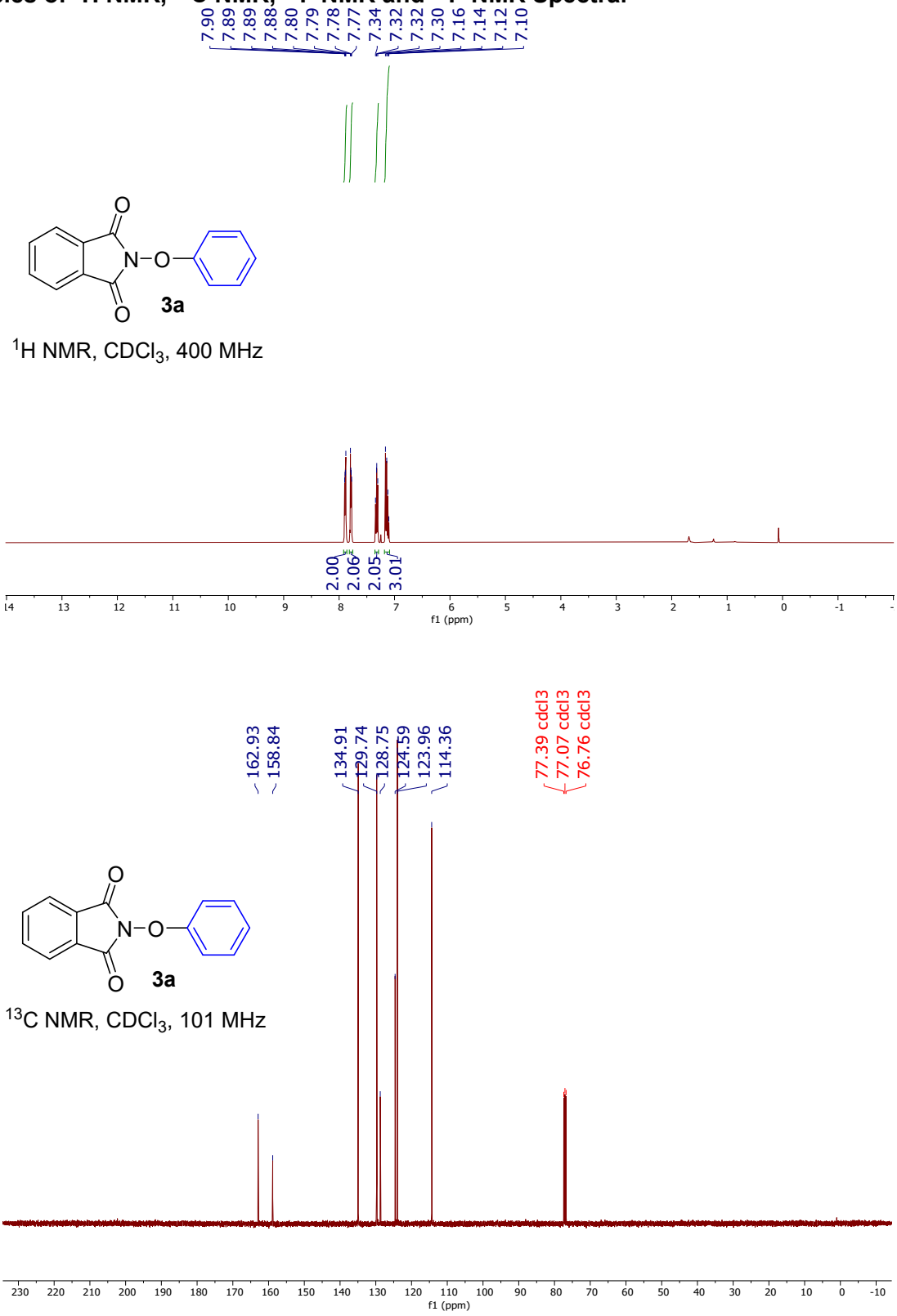
4a

O-phenylhydroxylamine

4a was synthesized according to the following general experimental procedure: To a solution of **3a** (0.25 mmol, 60 mg) in 2.5 mL $CHCl_3$ under stirring conditions, NH_3(1.0 mL, 7N in methanol) was added dropwise at room temperature. Stirring was continued for 12 h and then concentrated under reduced pressure. After that the crude reaction mixture was adsorbed to silica and transfer to a plug of silica gel, washing with 20% Et_2O in pentane (100 mL). Removal of solvent under reduced pressure afforded yellowish oil **4a** in 91%yield.

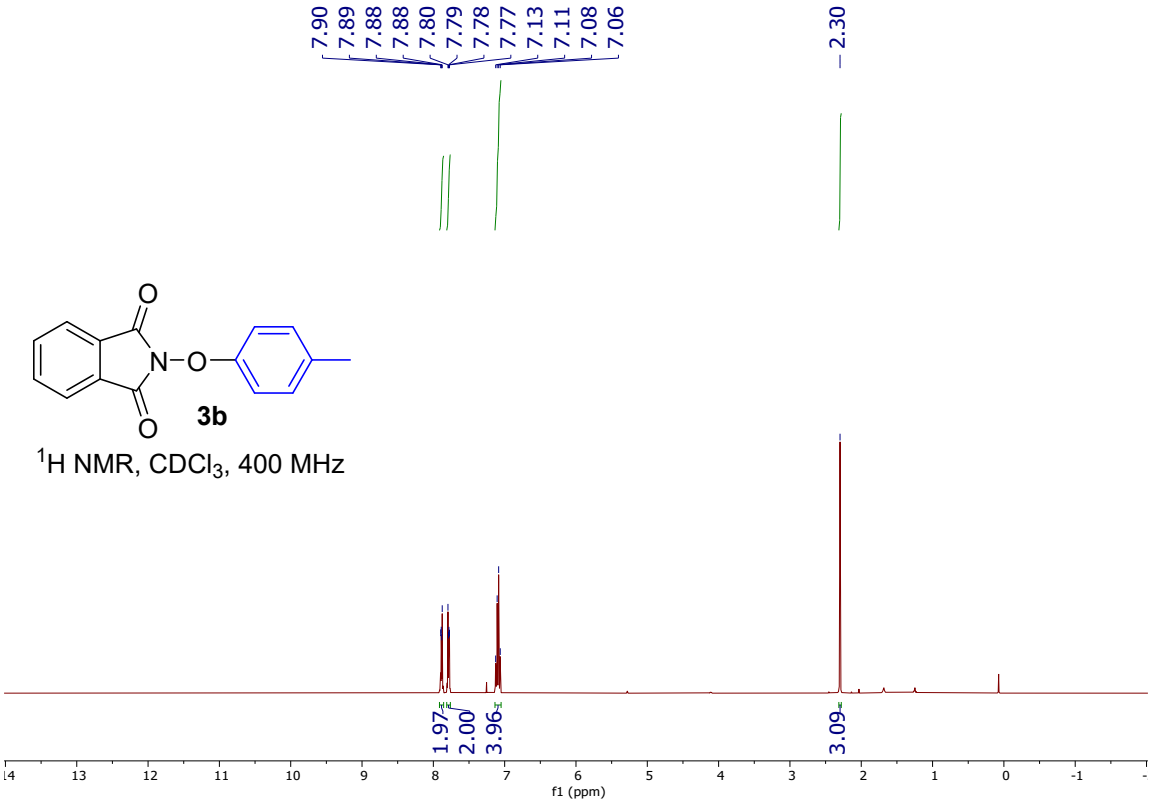
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.33 (m, 2H), 7.00 (q, *J* = 7.5 Hz, 1H), 6.87 (dd, *J* = 8.5, 4.8 Hz, 2H), 3.77 (s, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 155.2, 129.9, 121.1, 115.5. All spectral data correspond to those given in the literature.[6]

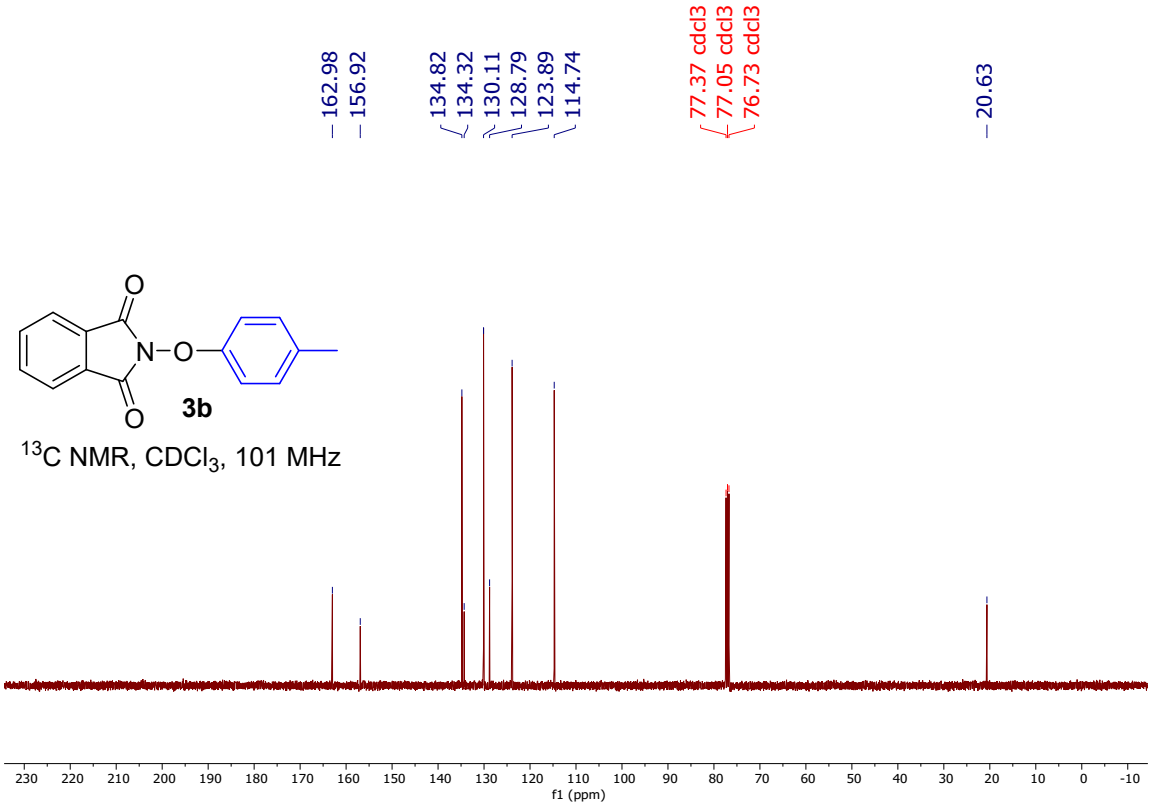
13. Copies of ^{1}H NMR, ^{13}C NMR, ^{19}F NMR and ^{31}P NMR Spectra.

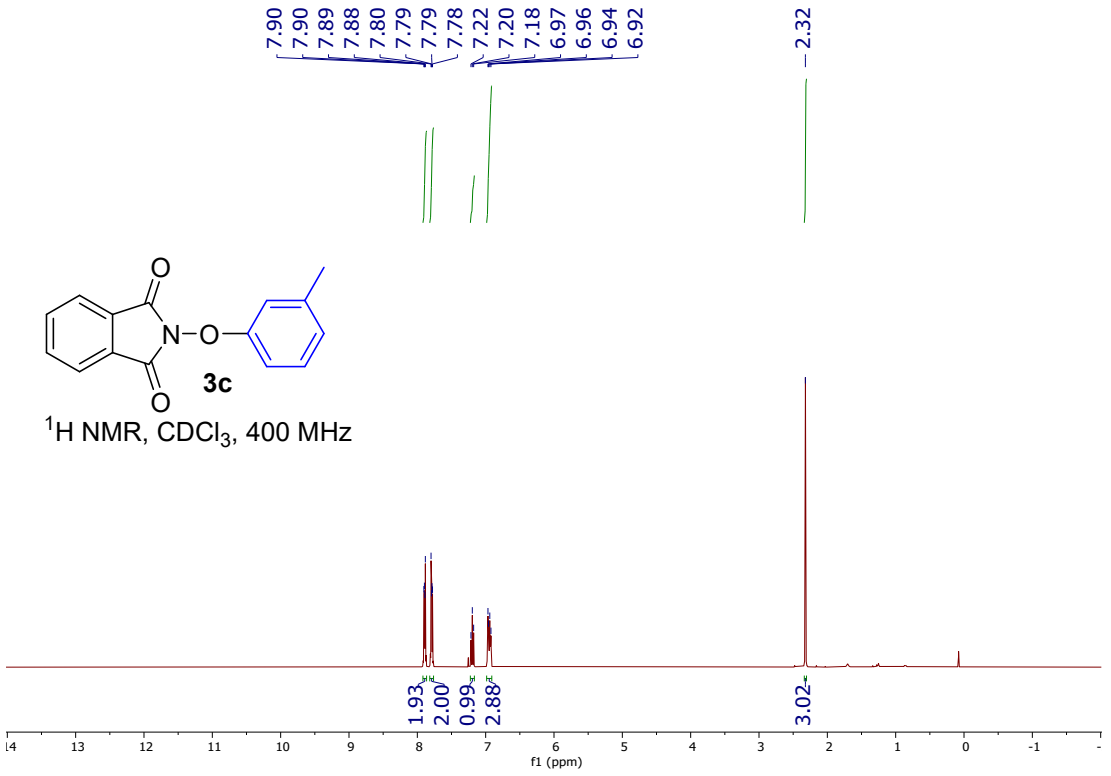


^{1}H NMR, $CDCl_3$, 400 MHz

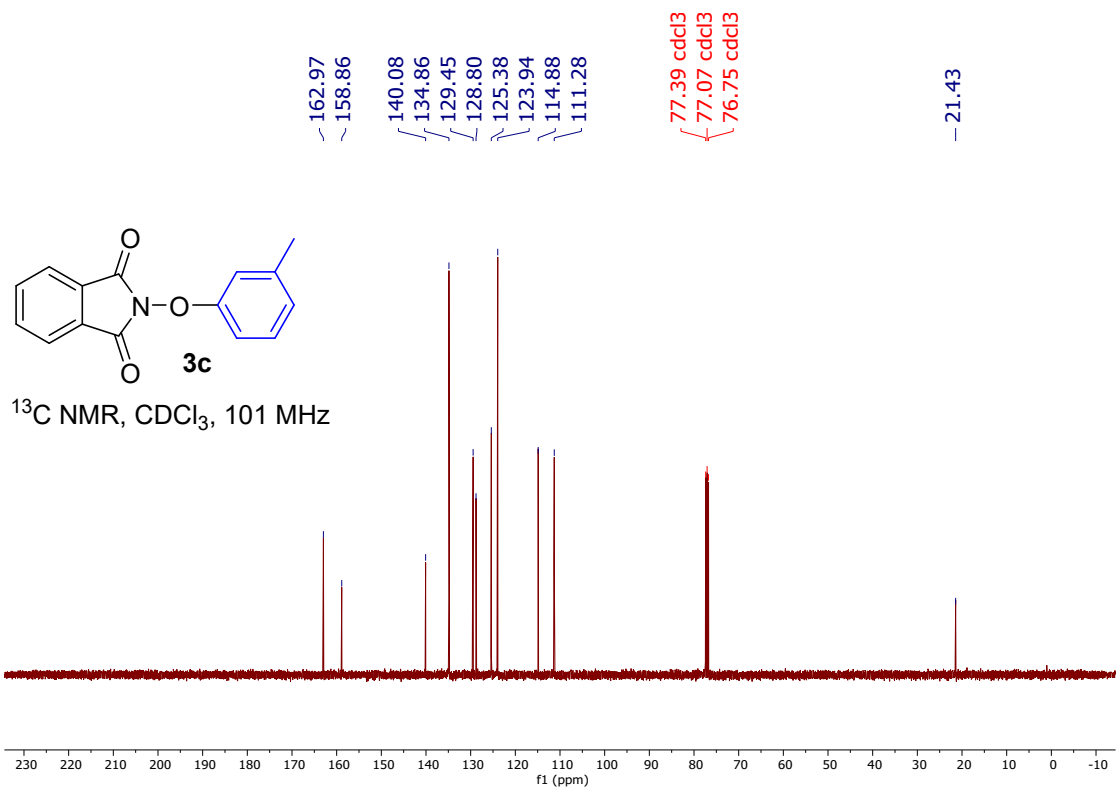
^{13}C NMR, $CDCl_3$, 101 MHz


7.90
7.89
7.88
7.88
7.80
7.79
7.78
7.77
7.13
7.11
7.08
7.06
2.30
O
N
O
O
O
3b
1H NMR, CDCl3, 400 MHz
1.97
2.00
3.96
3.09
f1 (ppm)

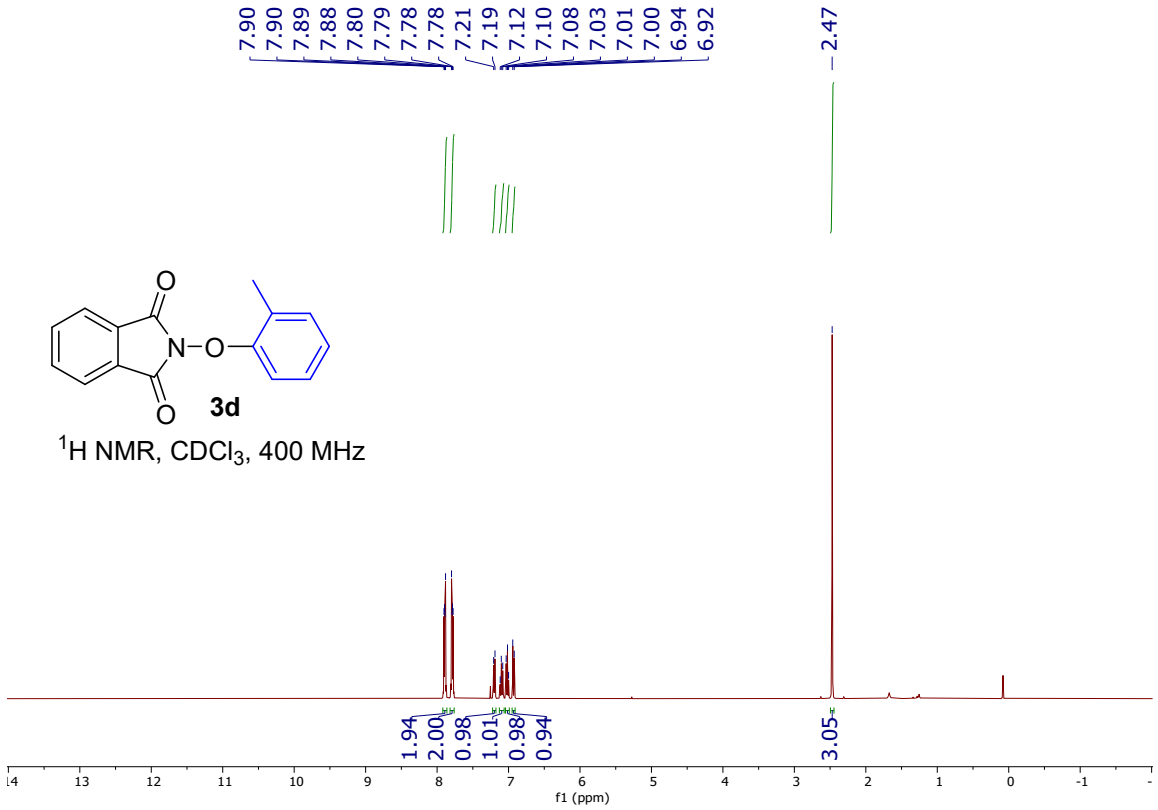

162.98
156.92
134.82
134.32
130.11
128.79
123.89
114.74
77.37 cdcl3
77.05 cdcl3
76.73 cdcl3
20.63
O
N
O
O
O
3b
13C NMR, CDCl3, 101 MHz
f1 (ppm)



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

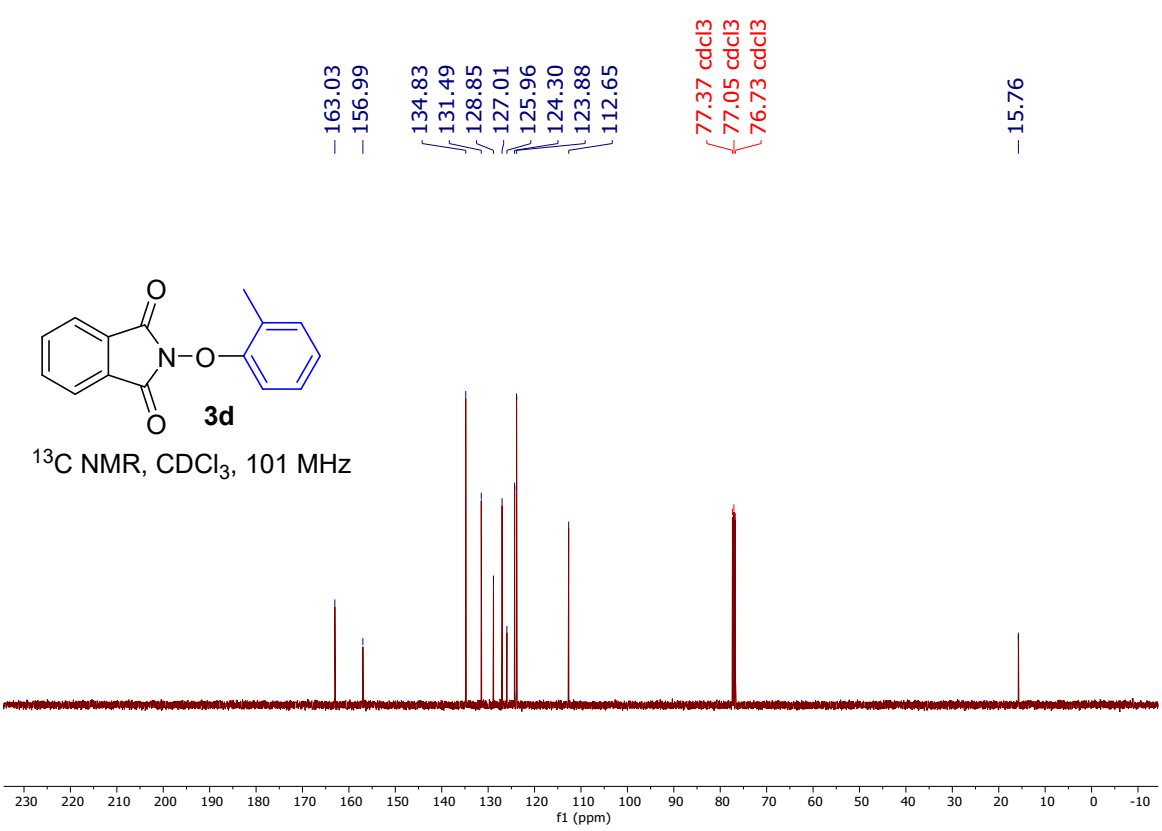


^{13}C NMR, $CDCl_3$, 101 MHz



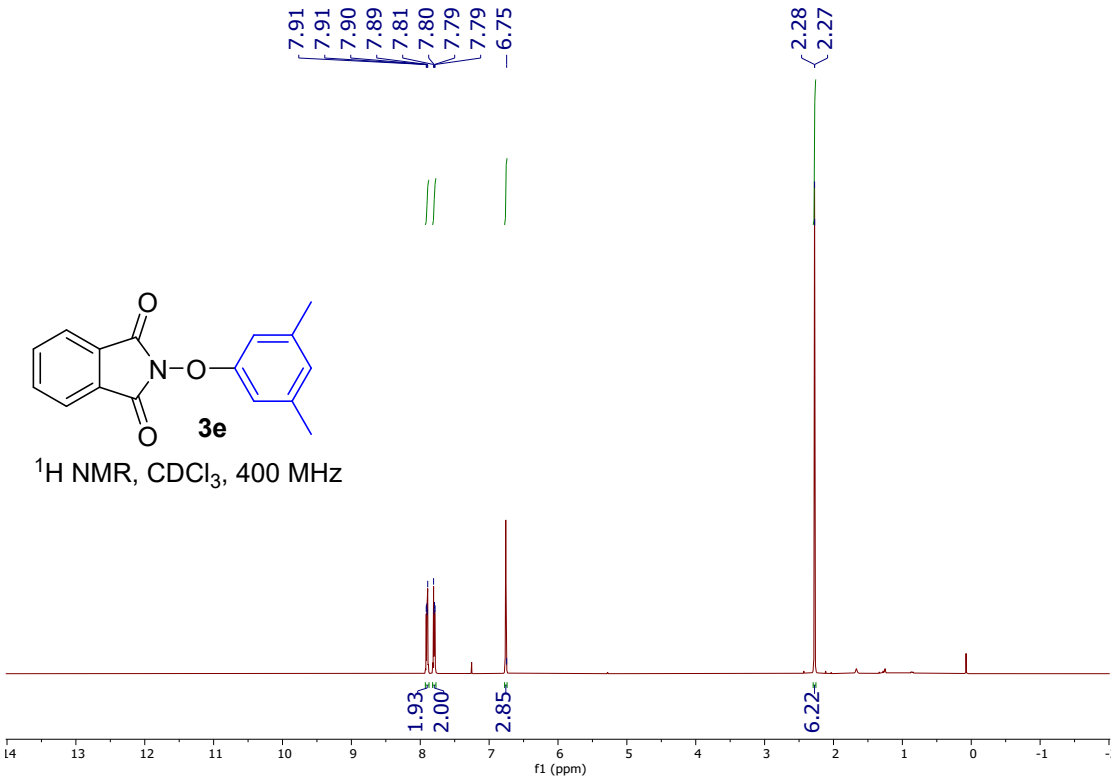
3d

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



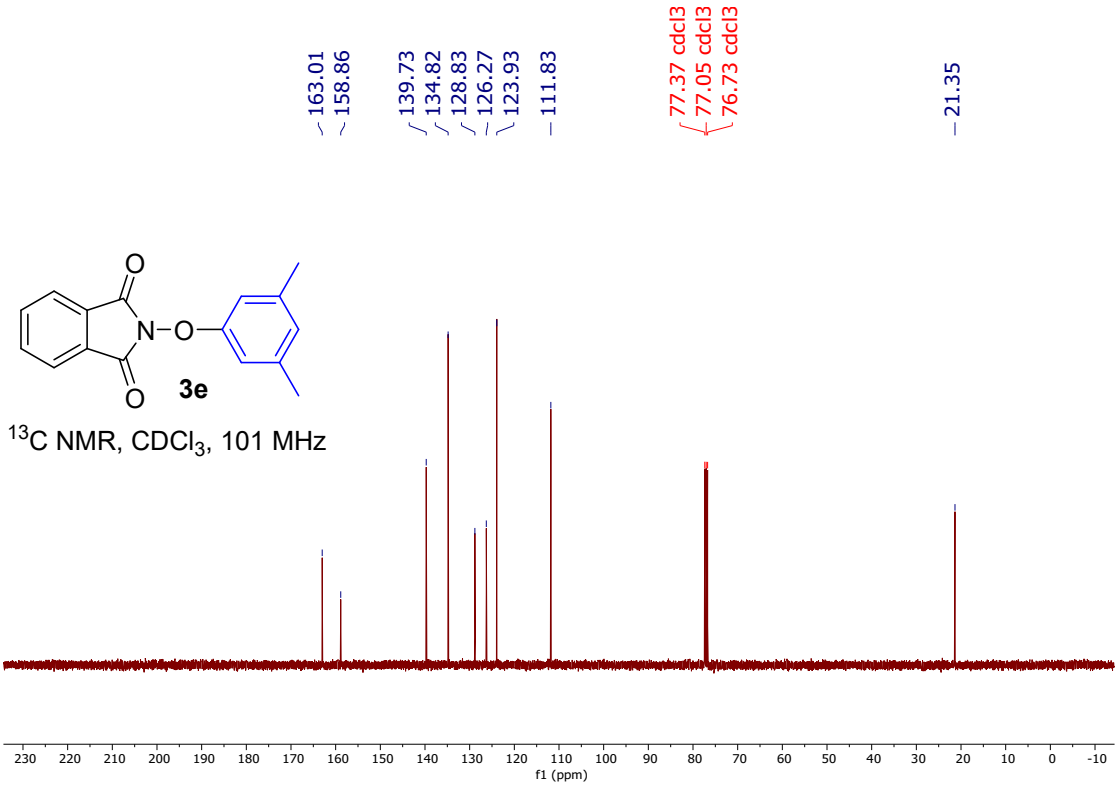
3d

^{13}C NMR, $CDCl_3$, 101 MHz



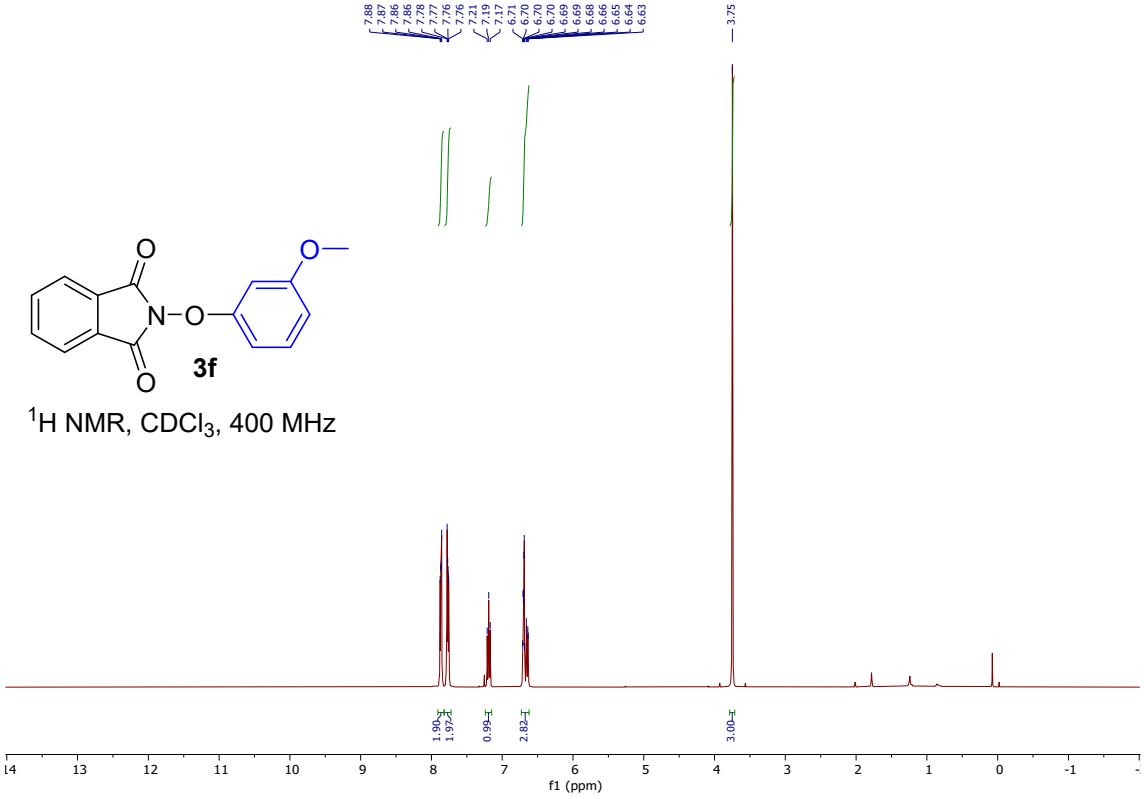
3e

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



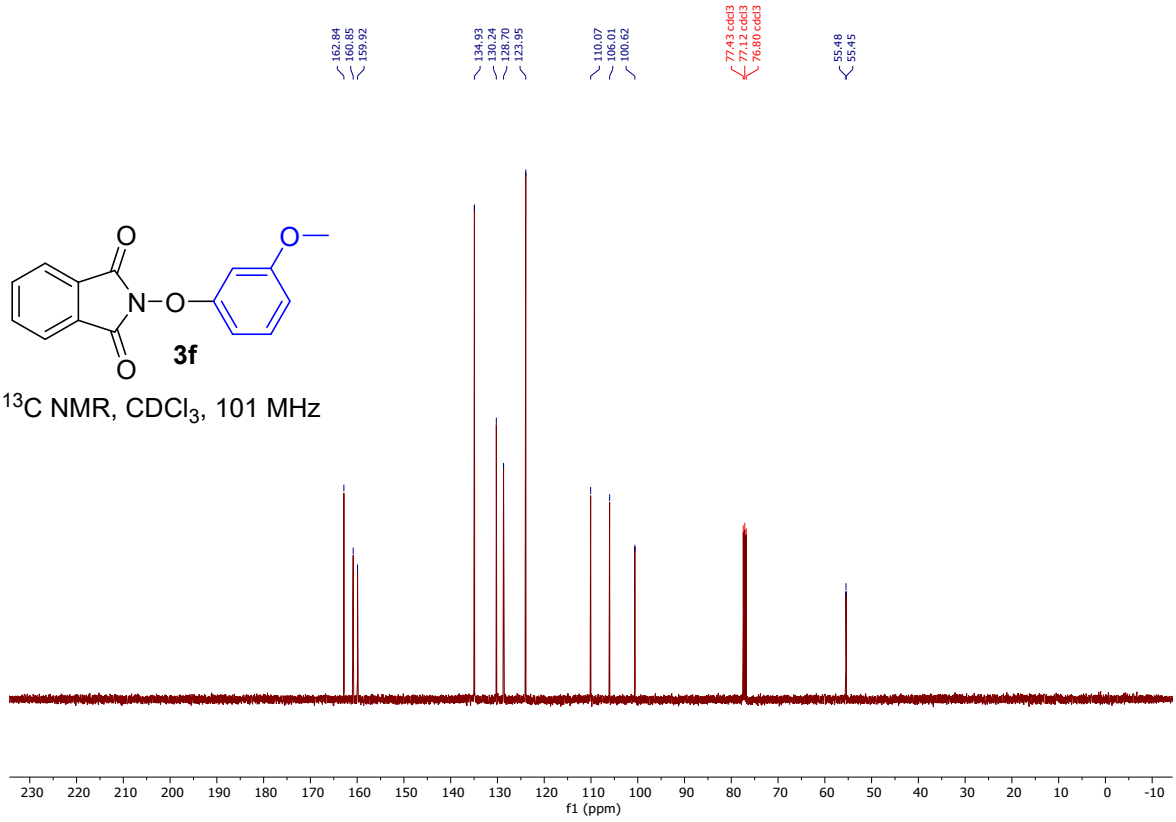
3e

^{13}C NMR, $CDCl_3$, 101 MHz



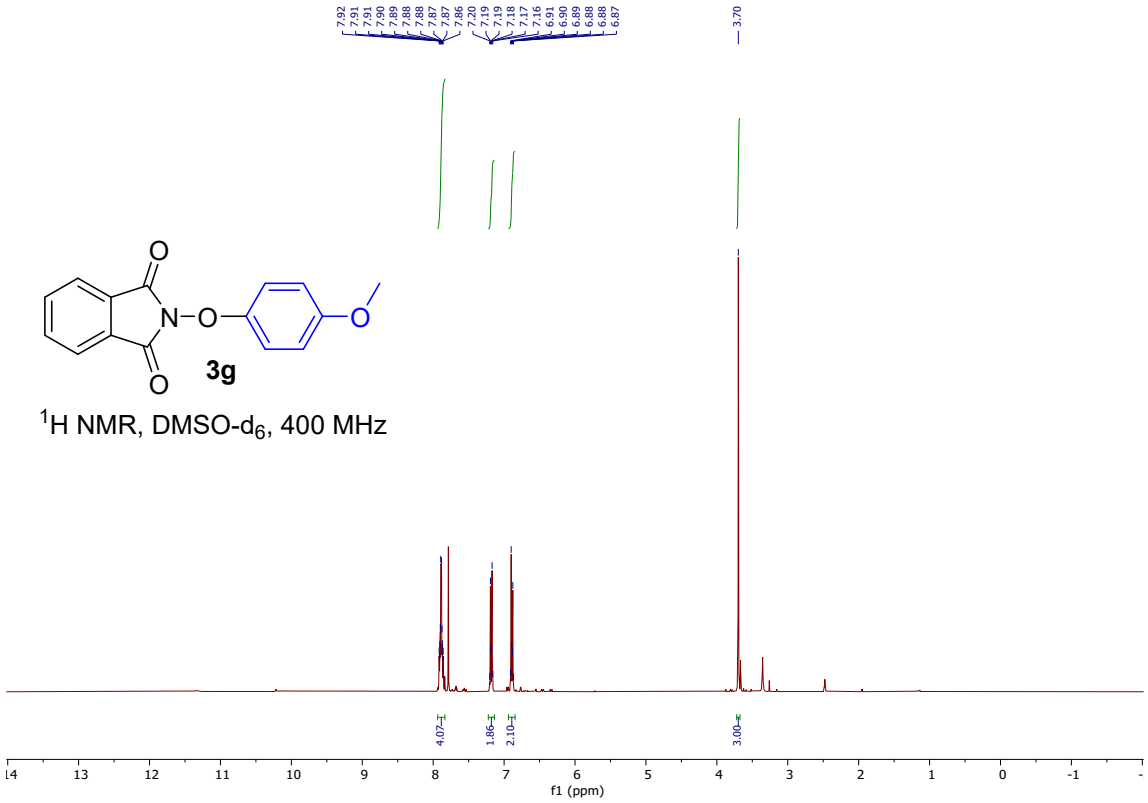
3f

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



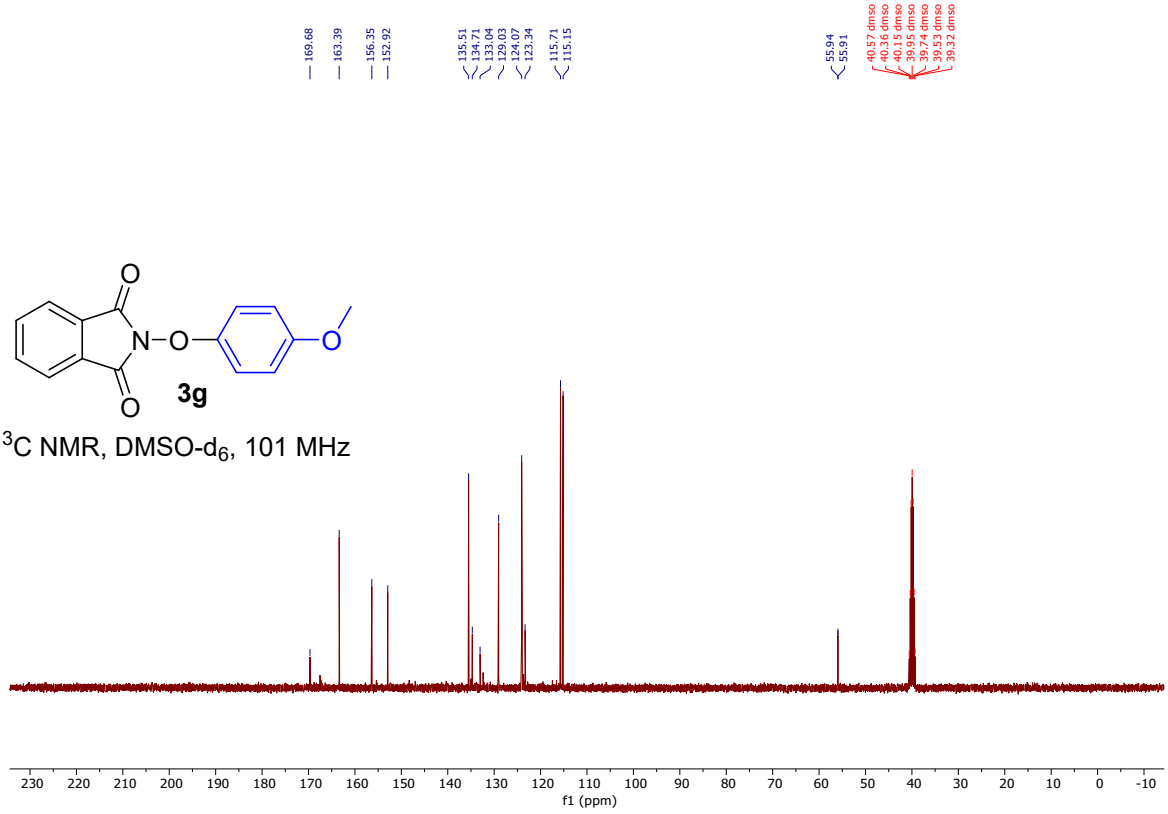
3f

^{13}C NMR, $CDCl_3$, 101 MHz



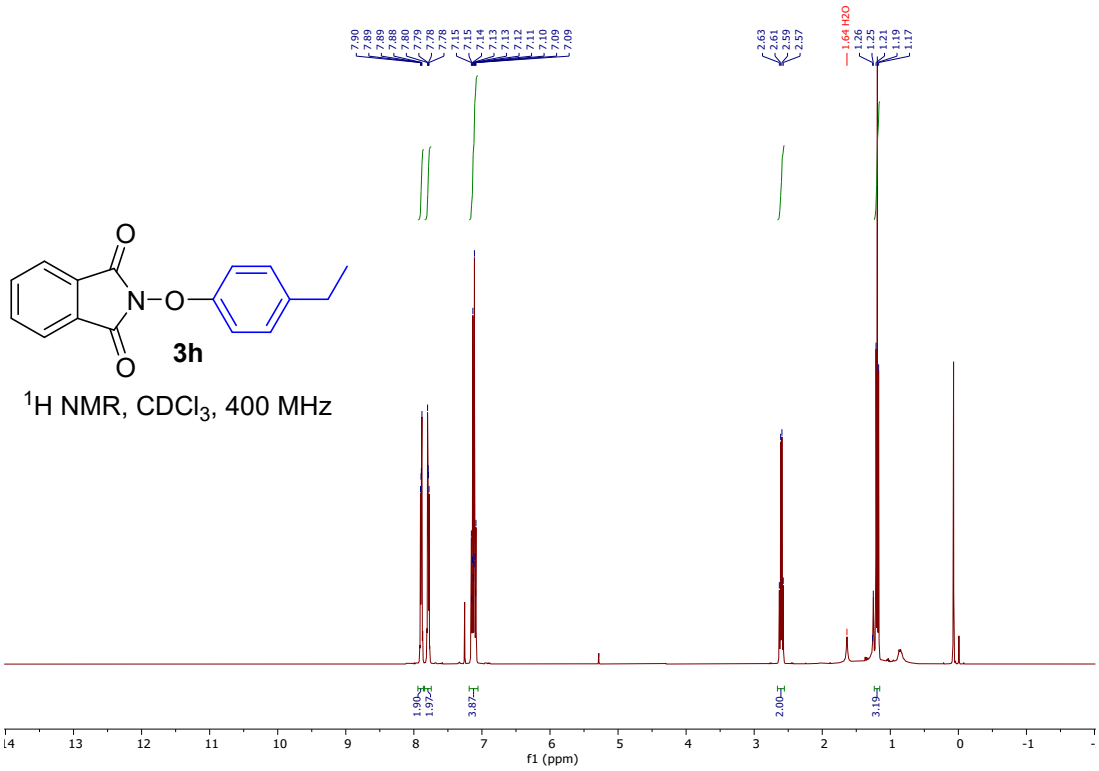
3g

^{1}H NMR, DMSO-d_6, 400 MHz



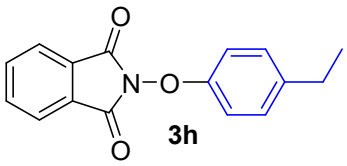
3g

^{13}C NMR, DMSO-d_6, 101 MHz



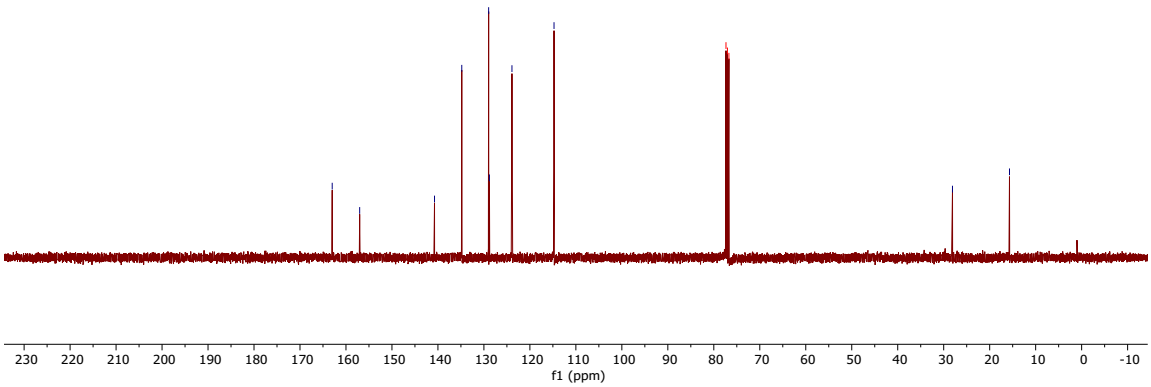
3h

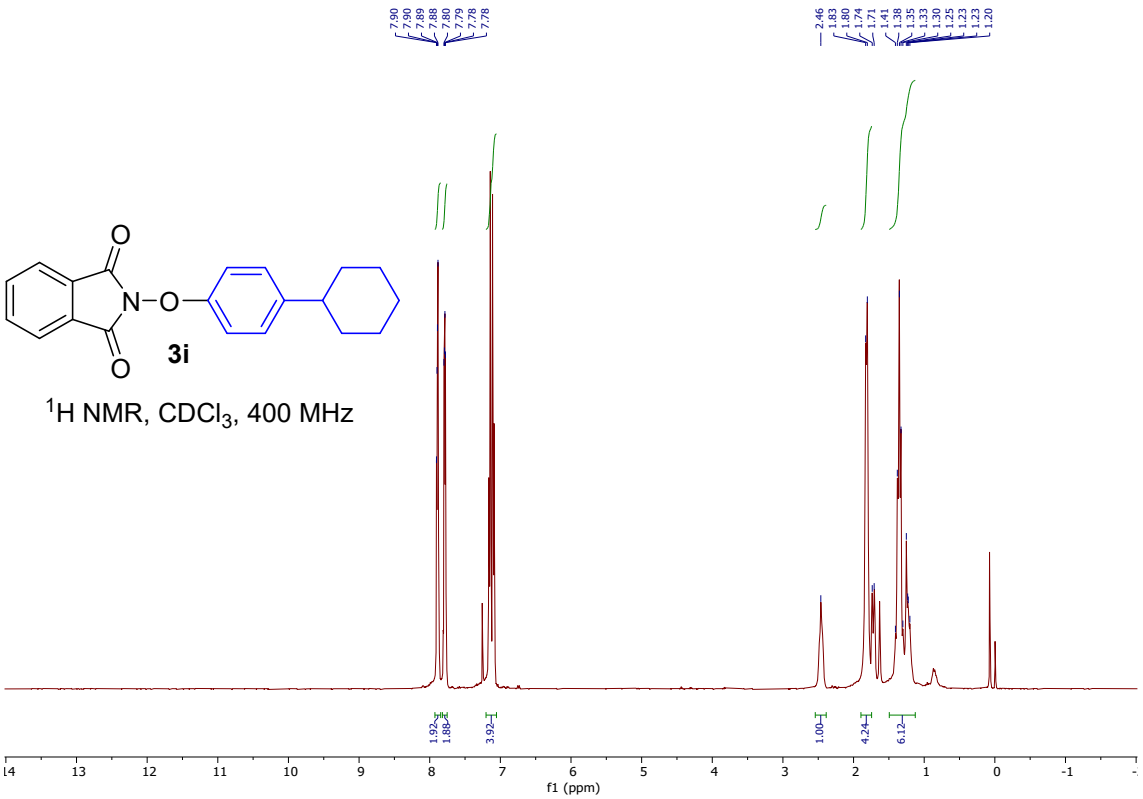
^{1}H NMR, $CDCl_3$, 400 MHz



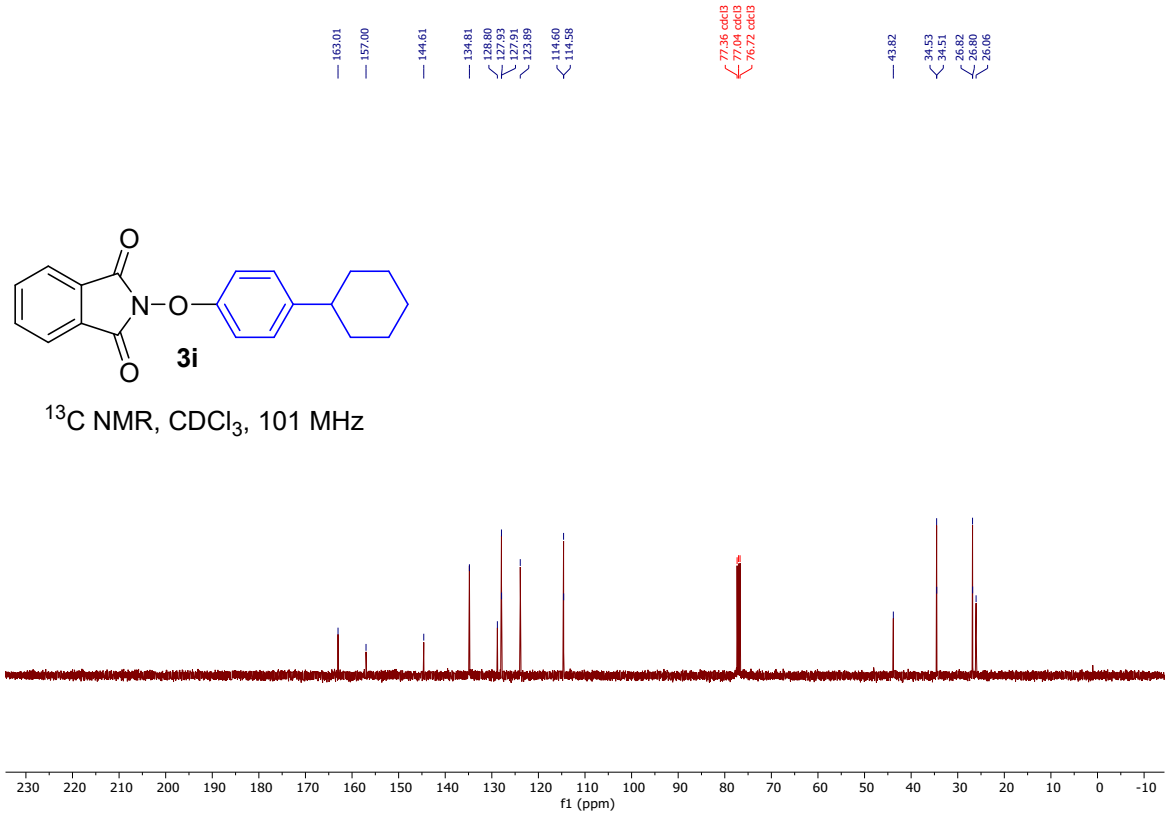
3h

^{13}C NMR, $CDCl_3$, 101 MHz

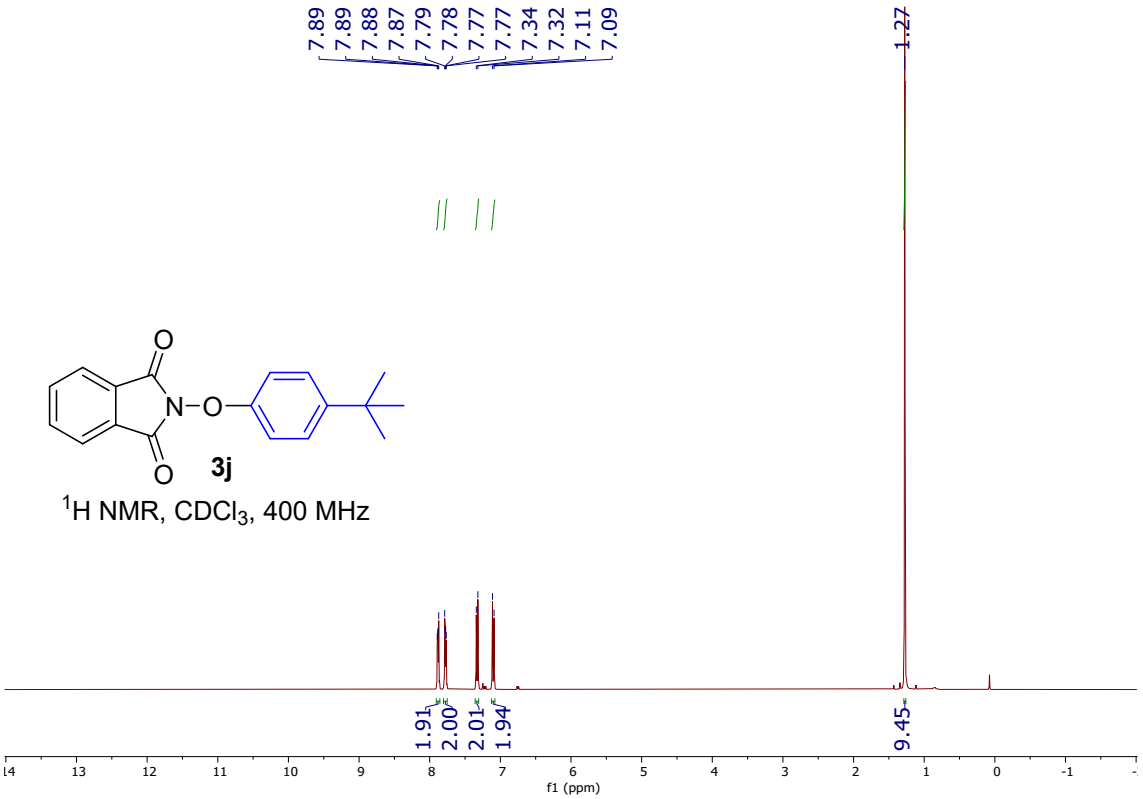




^{1}H NMR, $CDCl_3$, 400 MHz

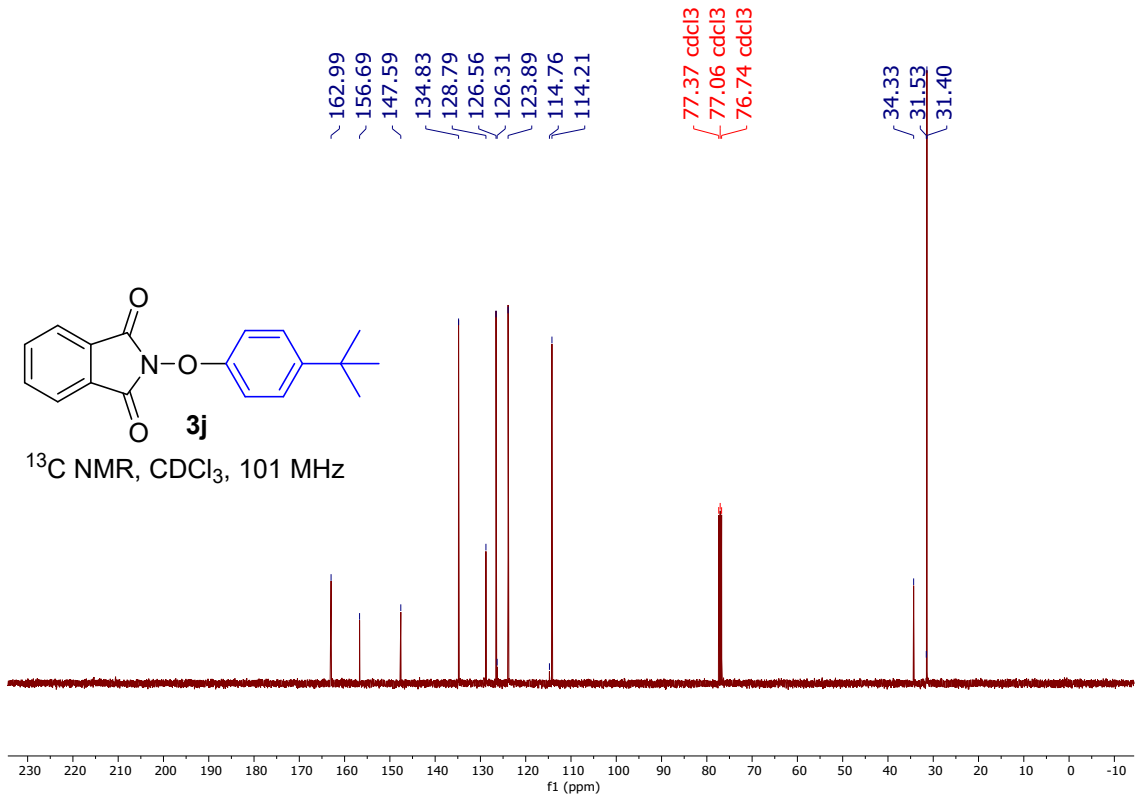


^{13}C NMR, $CDCl_3$, 101 MHz



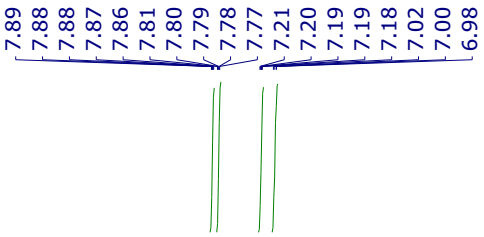
3j

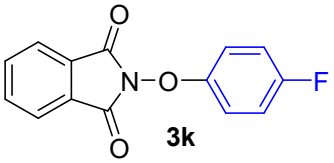
^{1}H NMR, $CDCl_3$, 400 MHz



3j

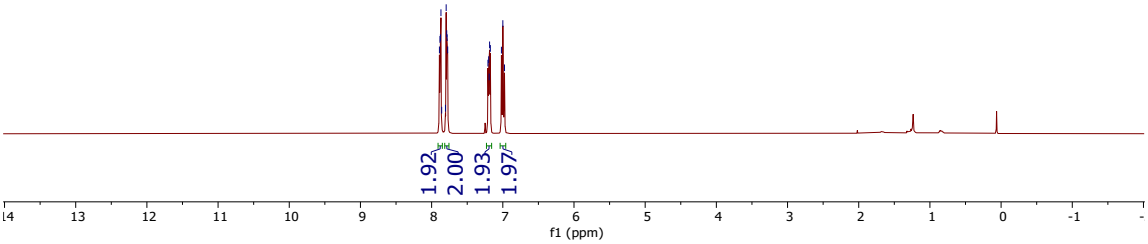
^{13}C NMR, $CDCl_3$, 101 MHz

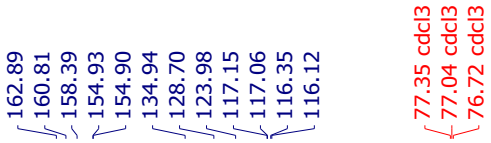


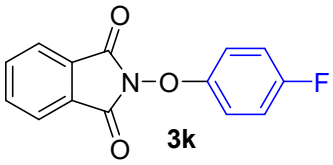


3k

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

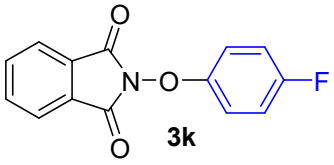






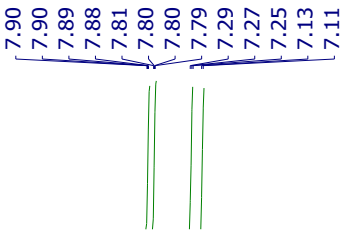
3k

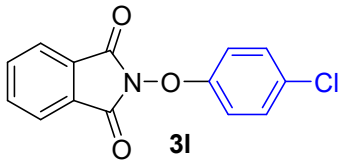
^{13}C NMR, $CDCl_3$, 101 MHz



3k

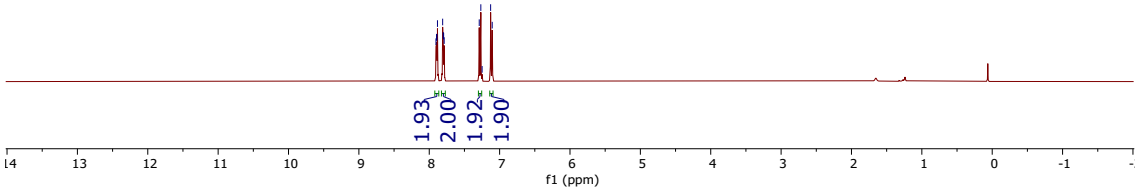
^{19}F NMR, $CDCl_3$, 471 MHz

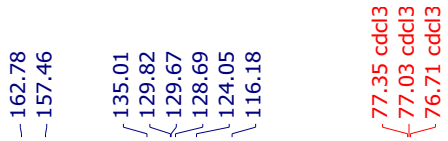


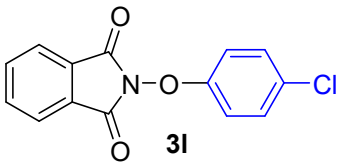


3l

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

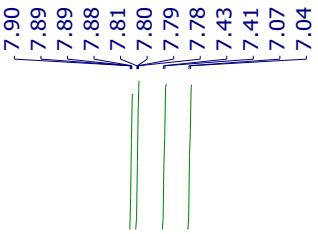


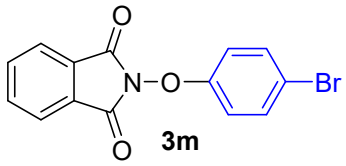




3l

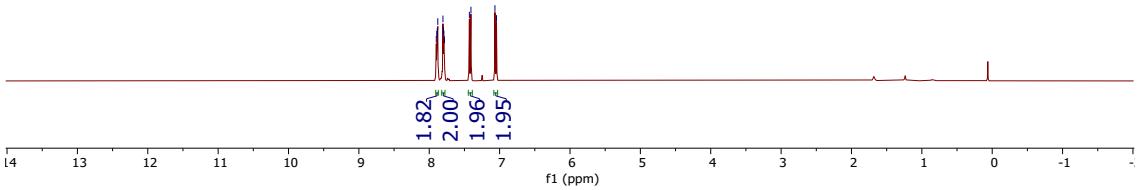
^{13}C NMR, $CDCl_3$, 101 MHz

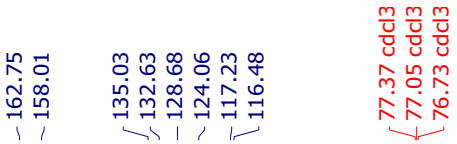


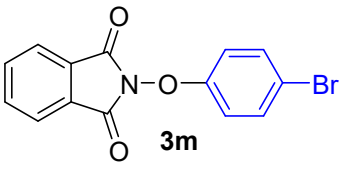


3m

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

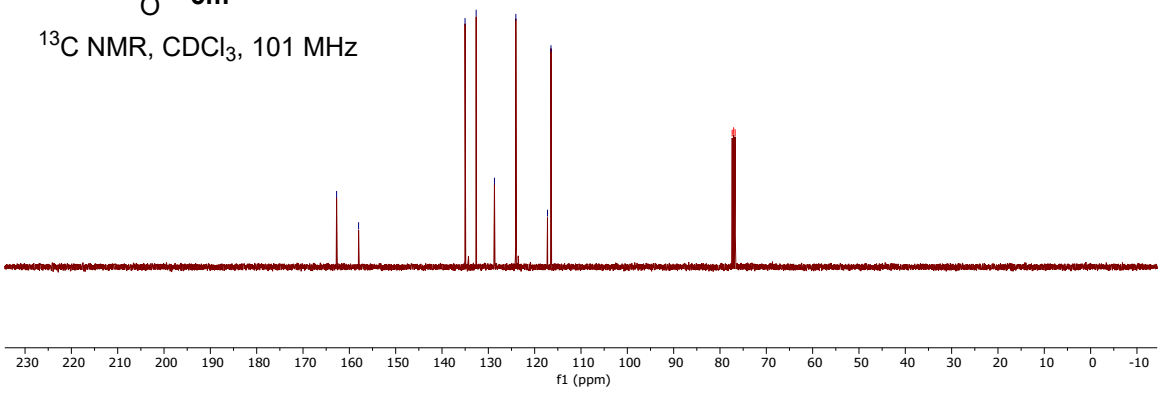


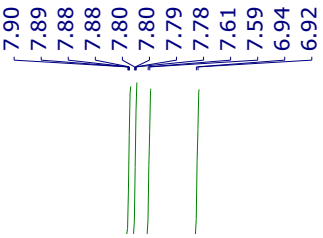


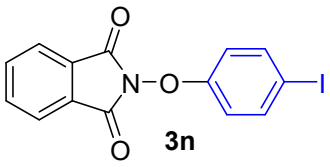


3m

^{13}C NMR, $CDCl_3$, 101 MHz

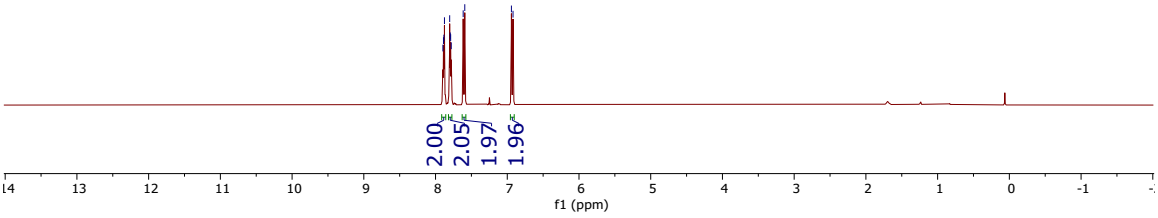


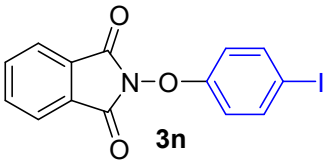




3n

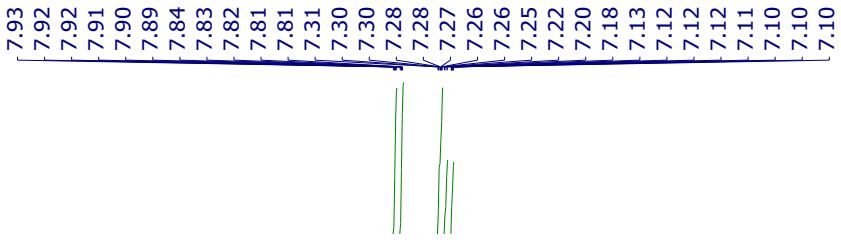
^{1}H NMR, $CDCl_3$, 400 MHz

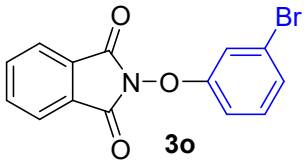




3n

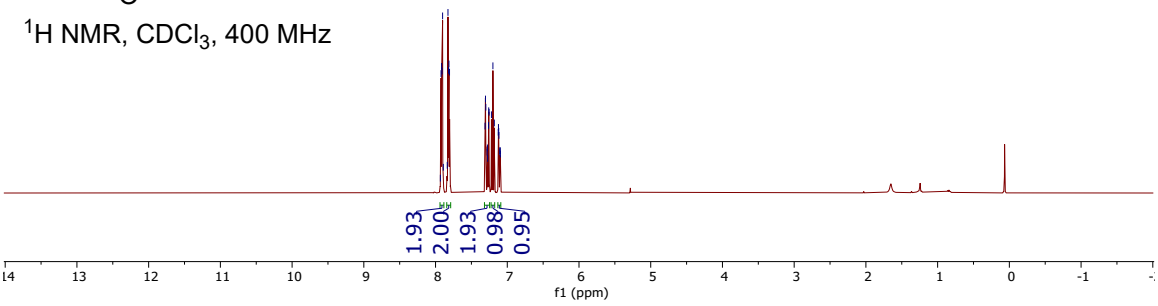
^{13}C NMR, $CDCl_3$, 101 MHz

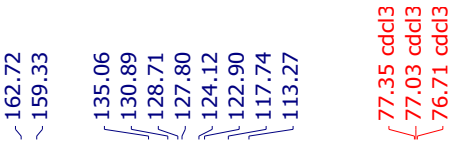


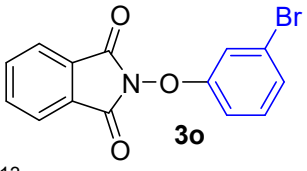


3o

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

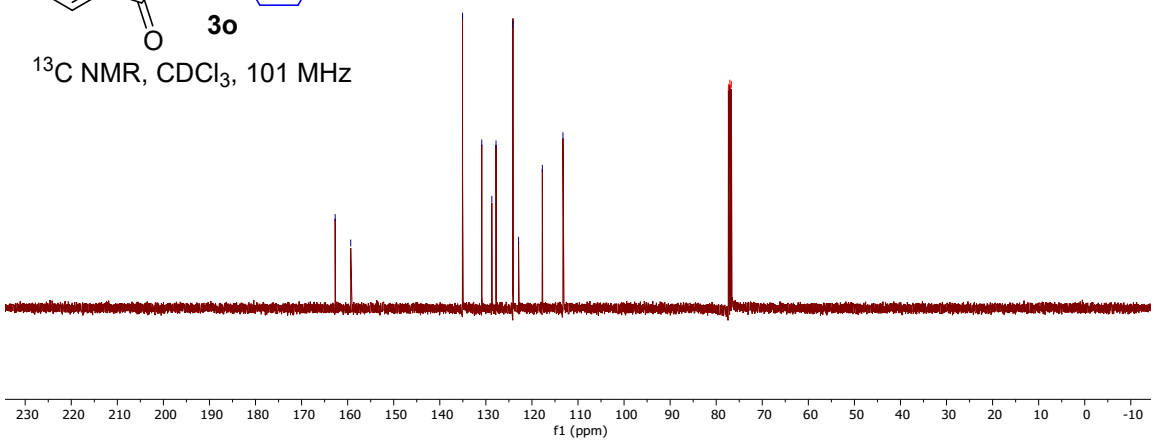


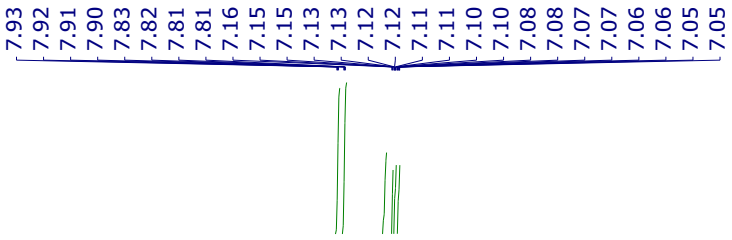


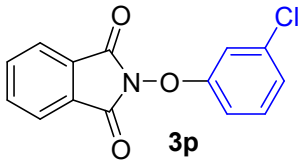


3o

^{13}C NMR, $CDCl_3$, 101 MHz

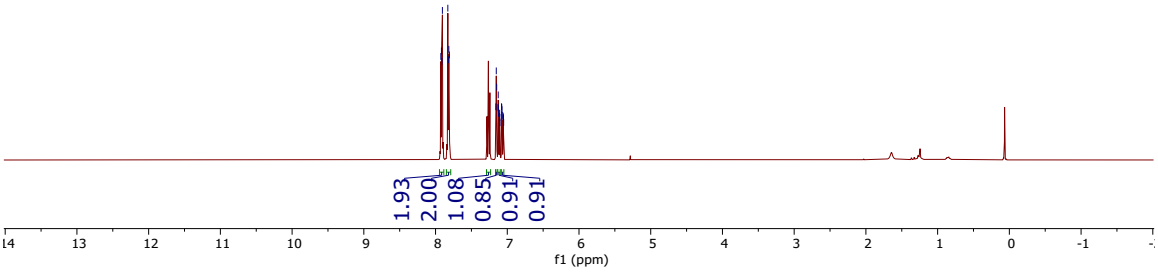


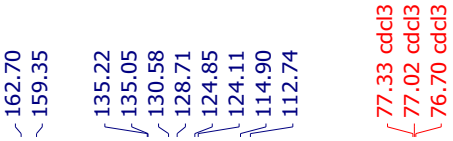


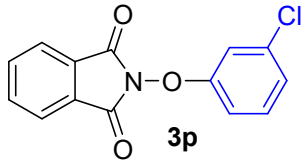


3p

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

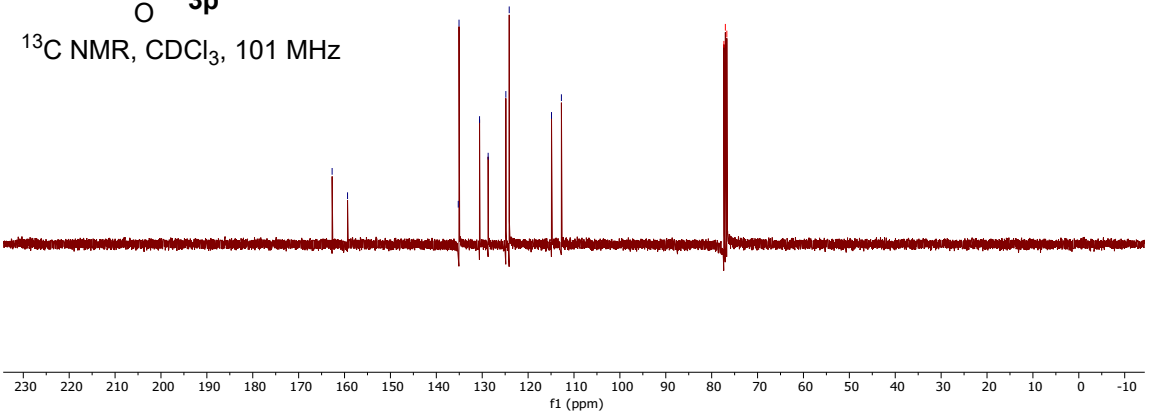


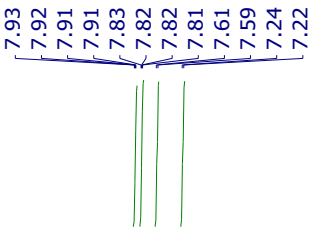


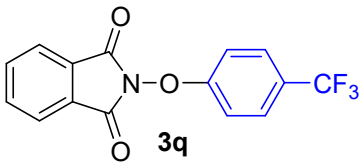


3p

^{13}C NMR, $CDCl_3$, 101 MHz

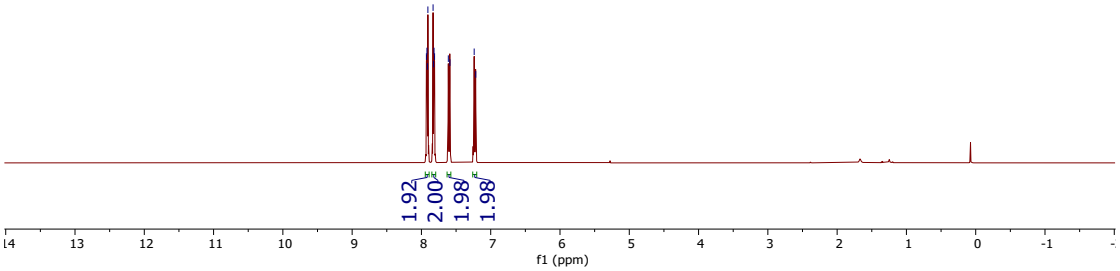


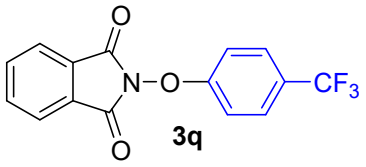




3q

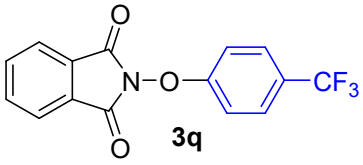
^{1}H NMR, $CDCl_3$, 400 MHz





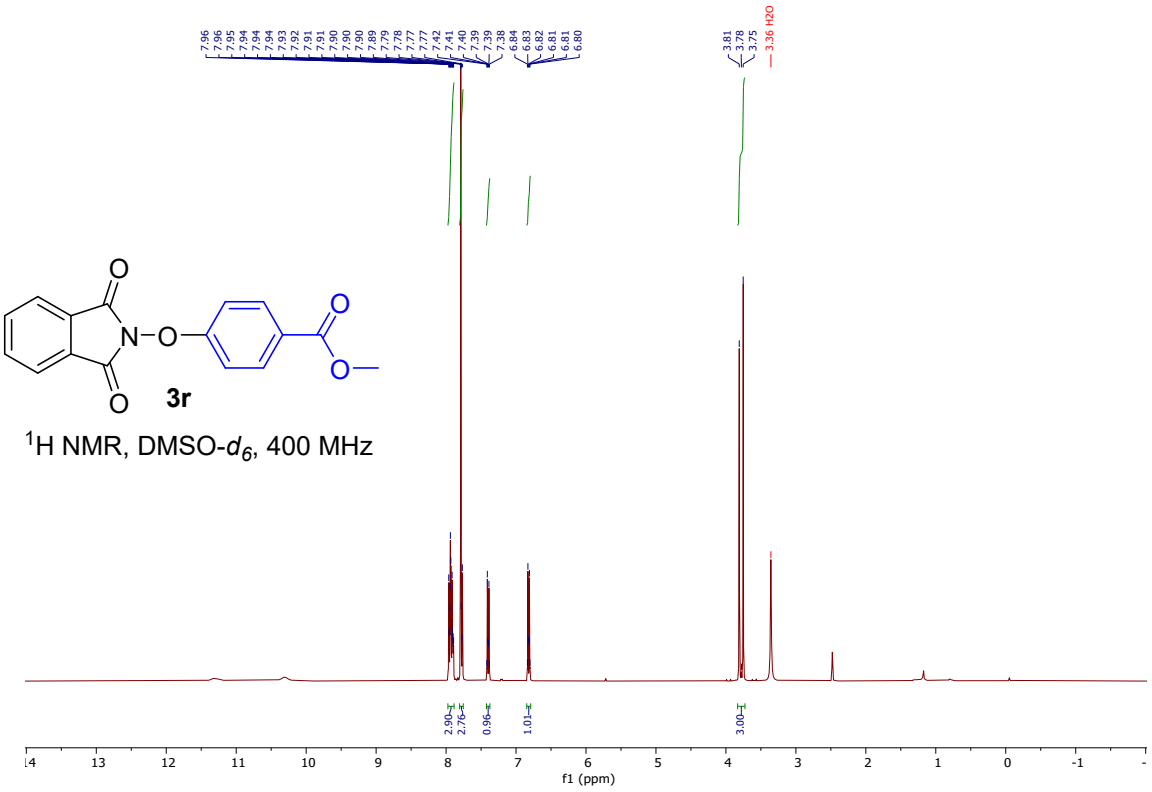
3q

^{13}C NMR, $CDCl_3$, 101 MHz



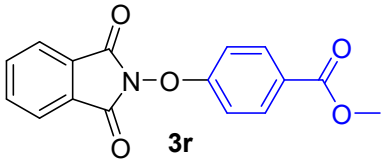
3q

^{19}F NMR, $CDCl_3$, 471 MHz



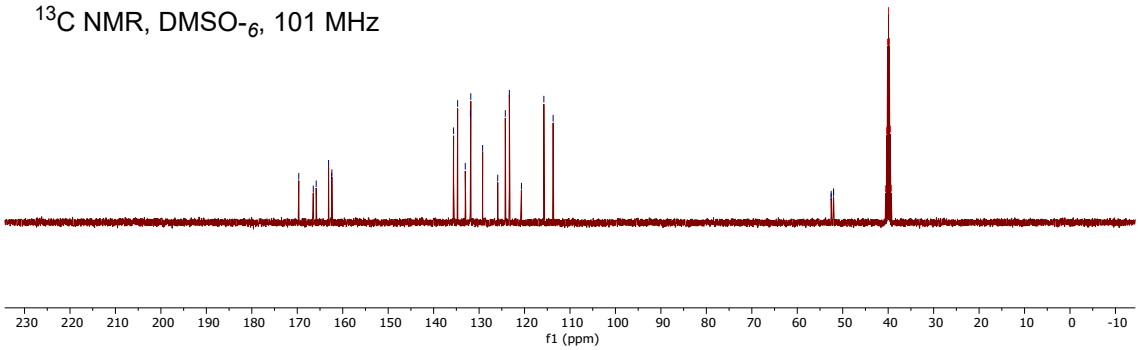
3r

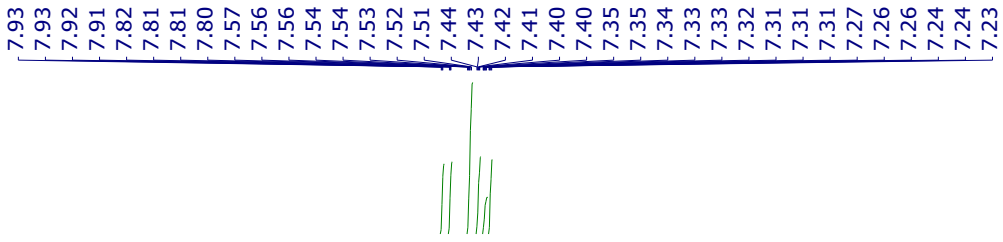
^{1}H NMR, DMSO-d_6, 400 MHz

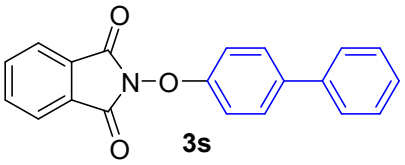


3r

^{13}C NMR, DMSO-$_6$, 101 MHz

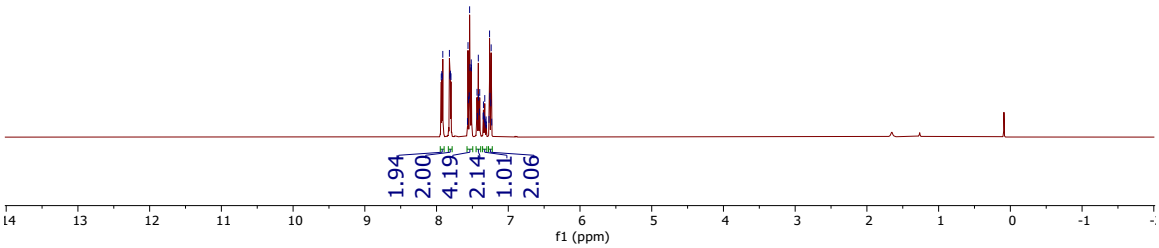


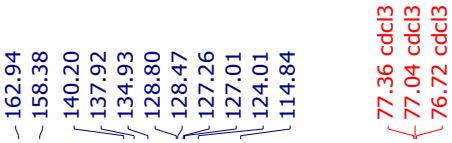


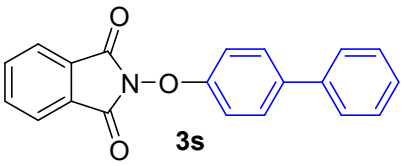


3s

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

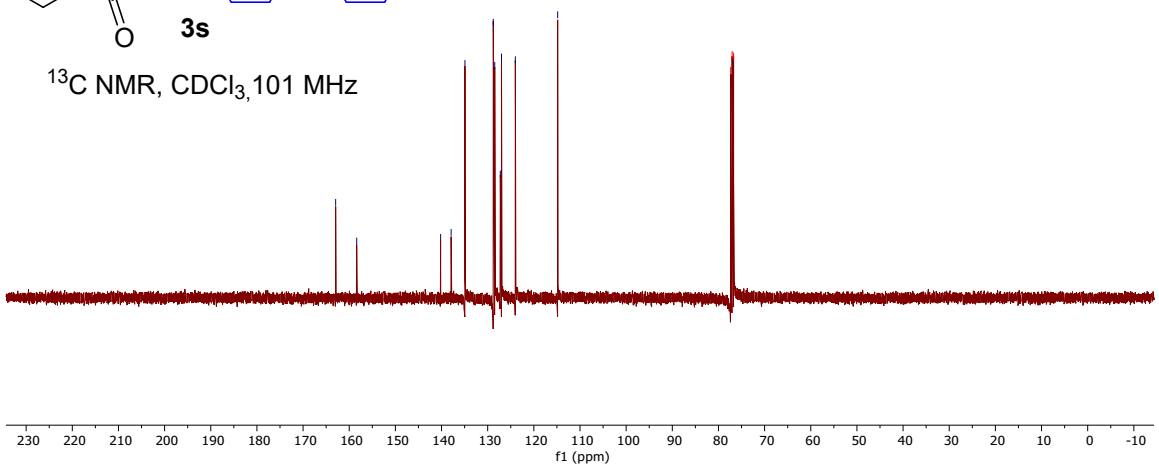


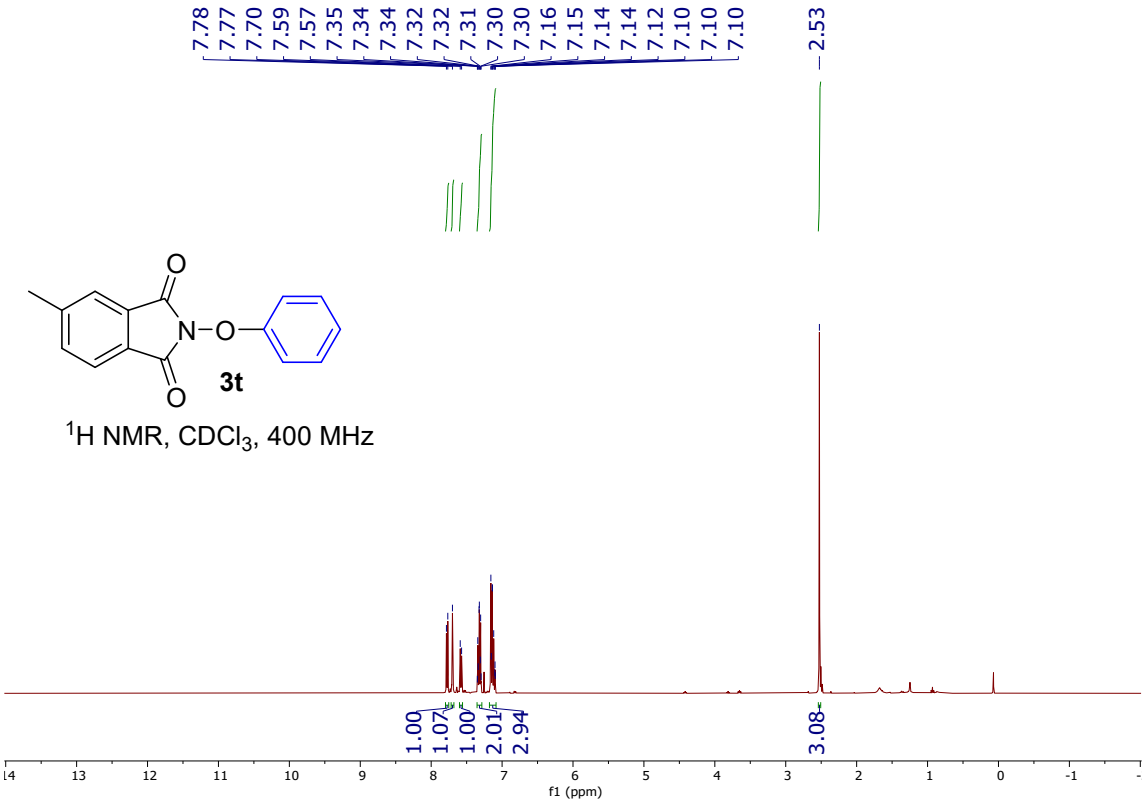




3s

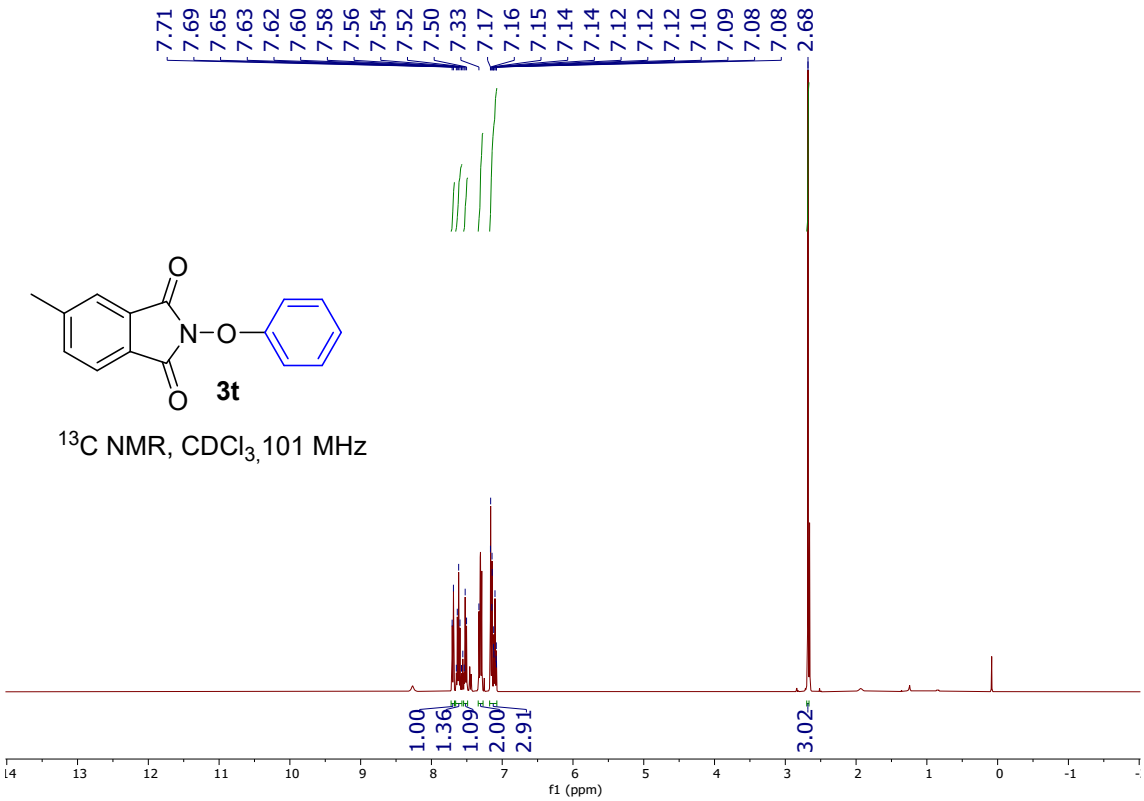
^{13}C NMR, $CDCl_3$, 101 MHz





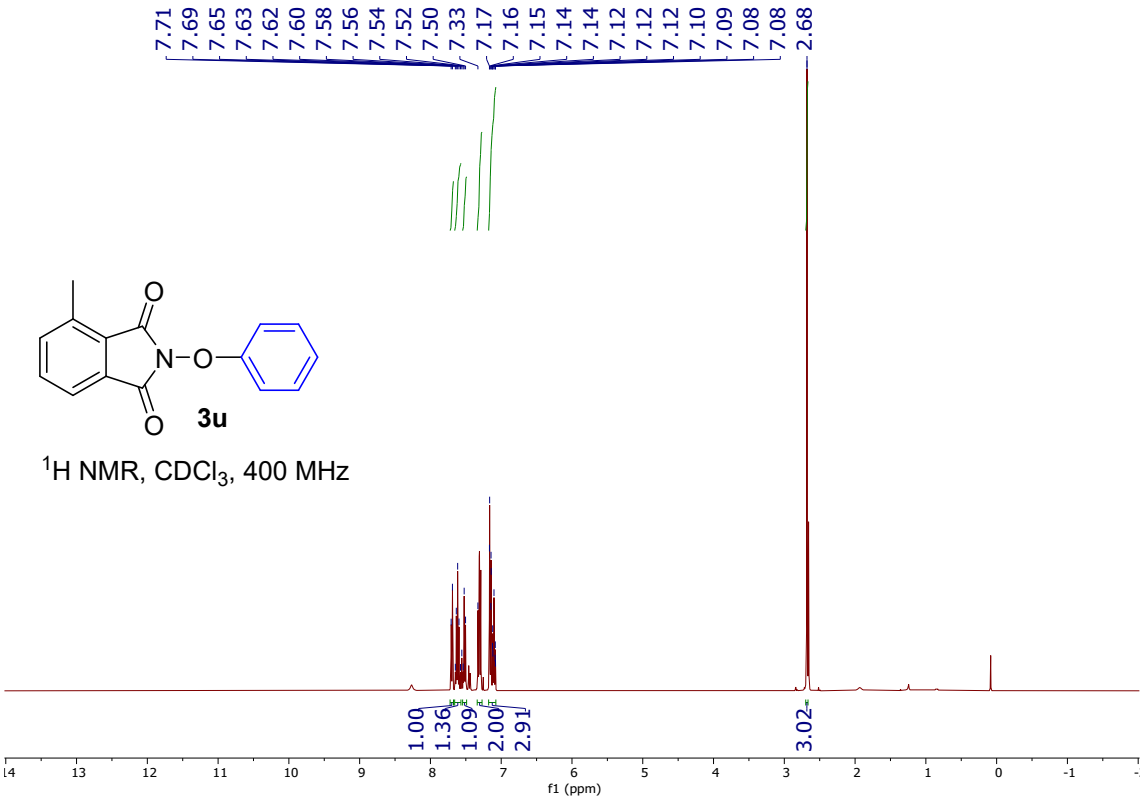
3t

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



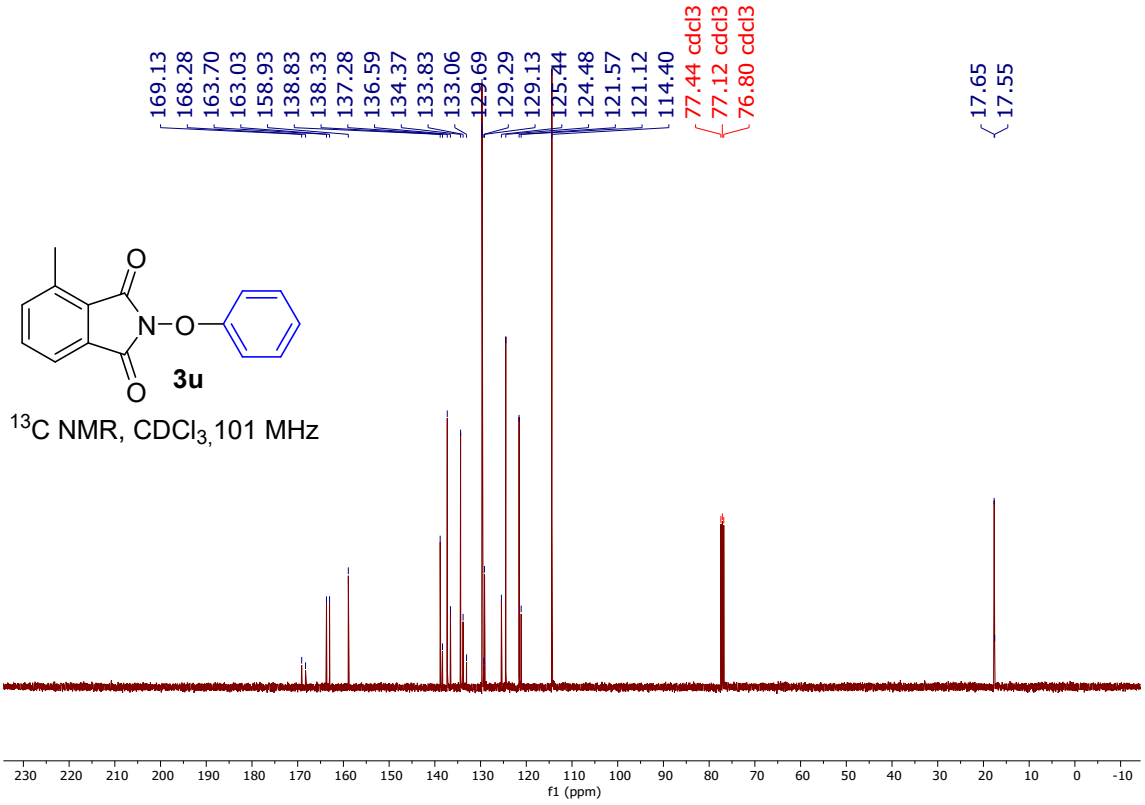
3t

^{13}C NMR, $CDCl_3$, 101 MHz



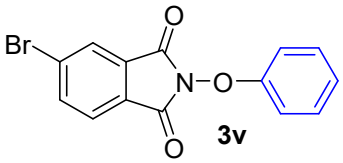
3u

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



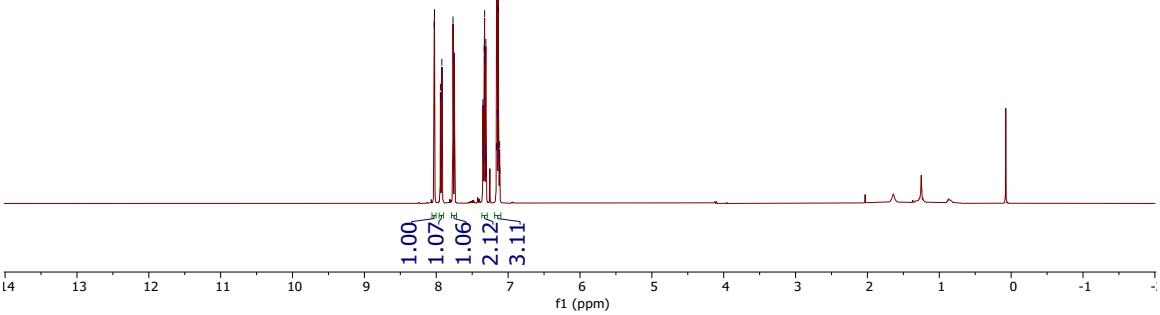
3u

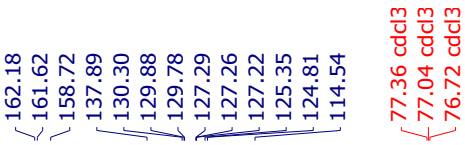
^{13}C NMR, $CDCl_3$, 101 MHz

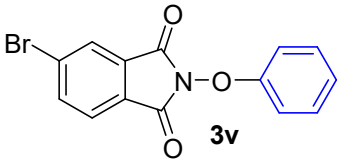


3v

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

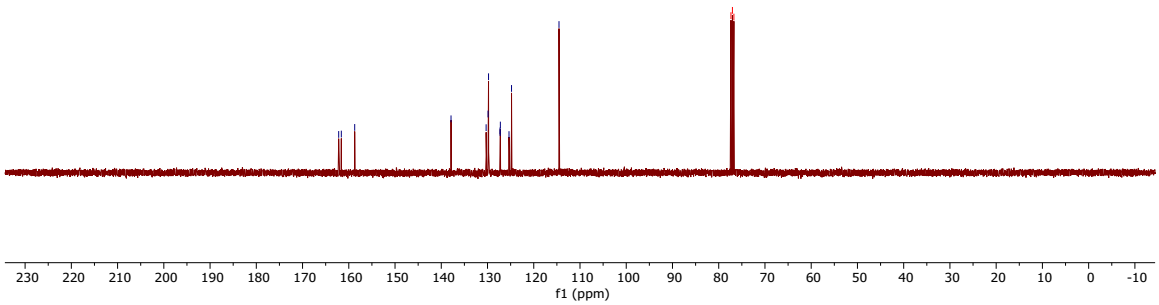


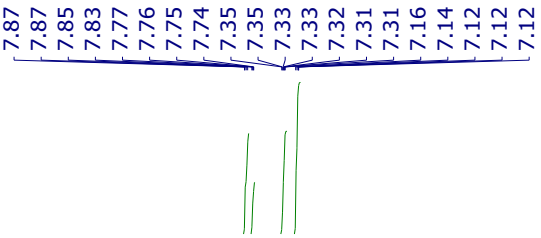


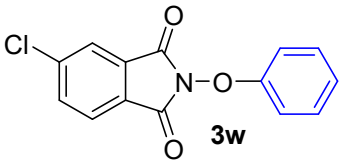


3v

^{13}C NMR, $CDCl_3$,101 MHz

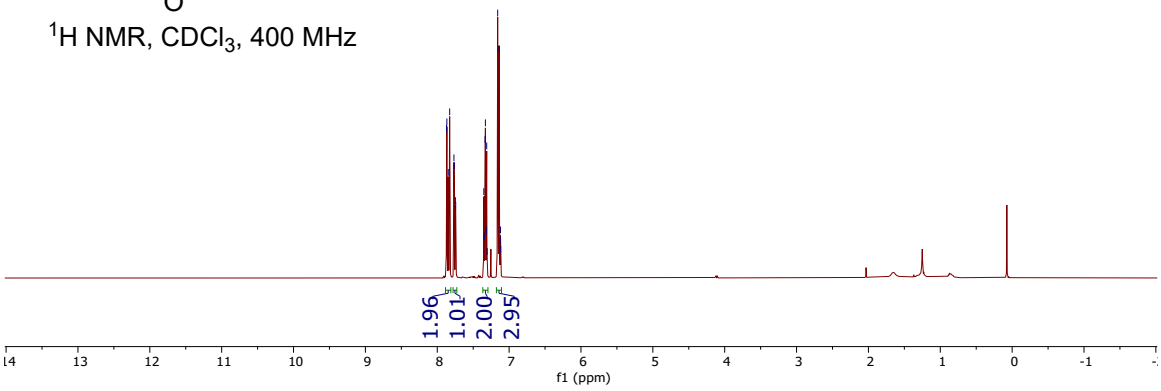


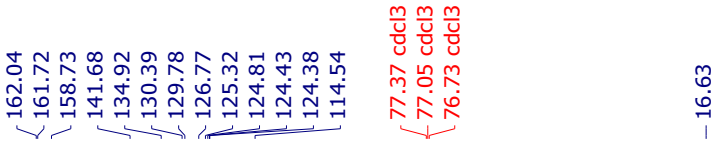


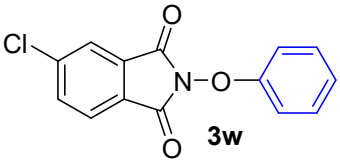


3w

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

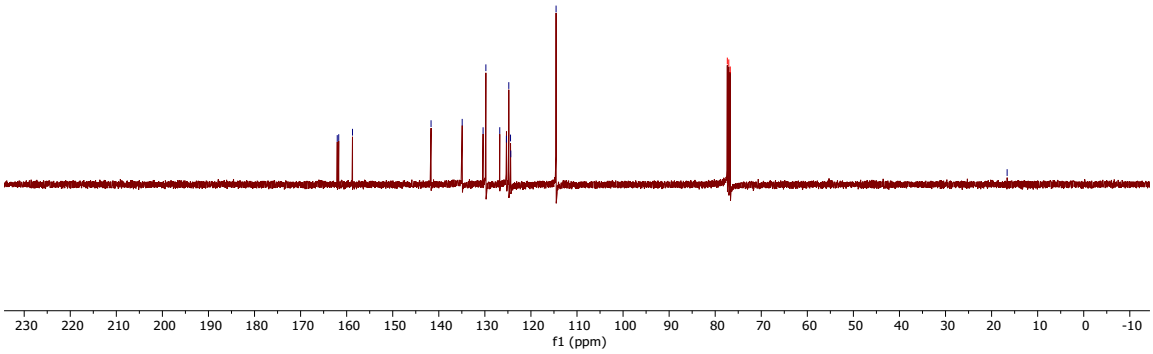


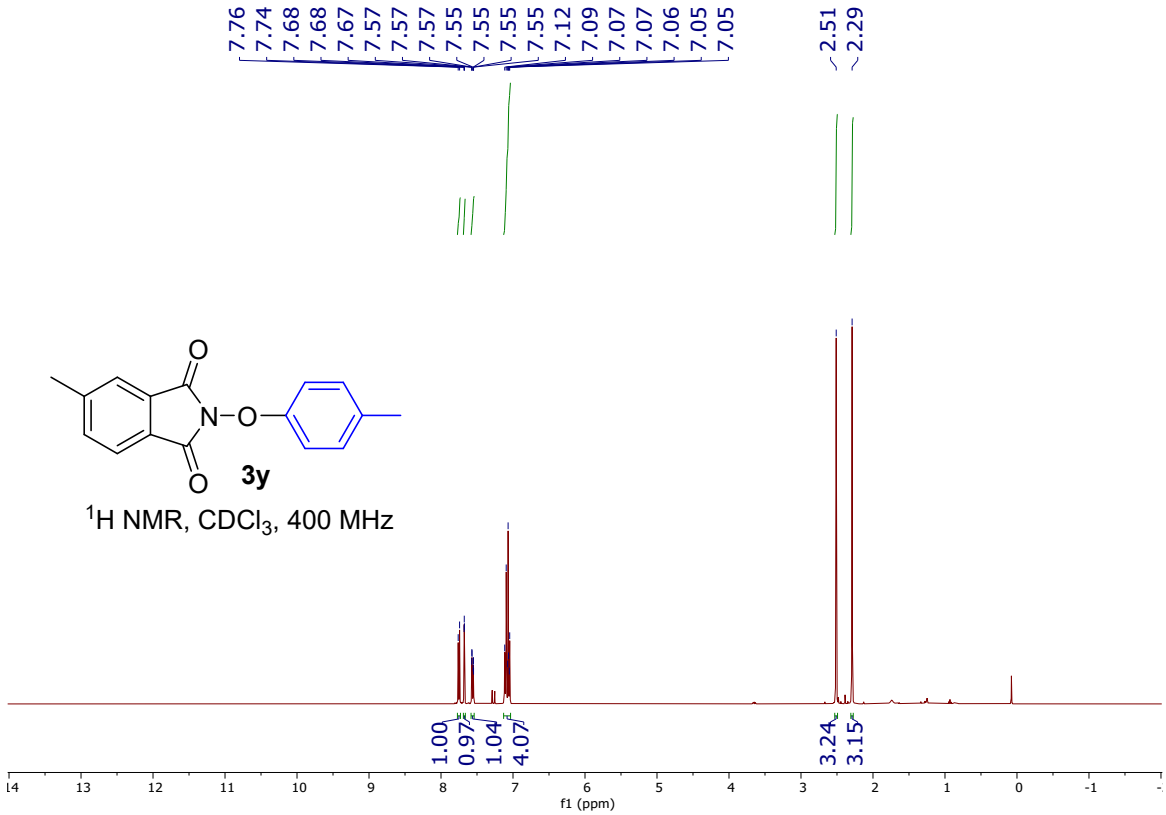


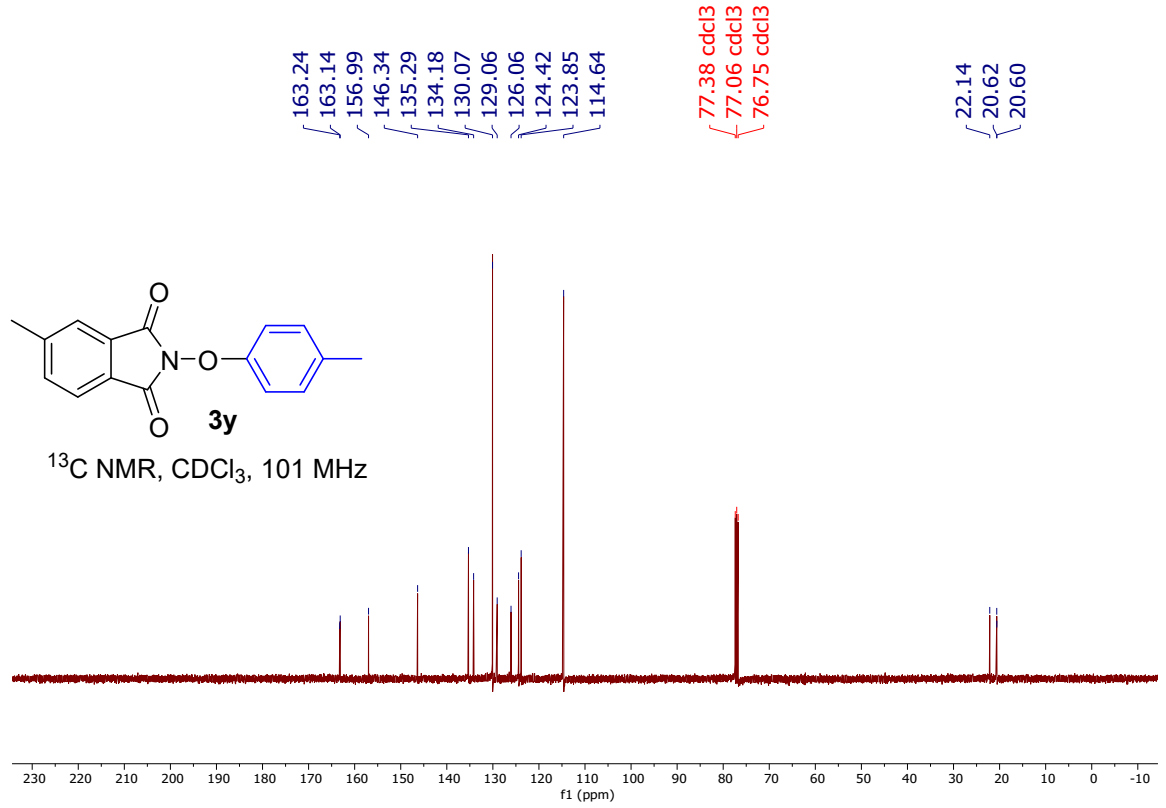


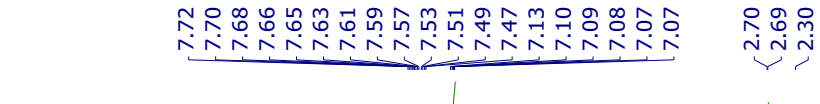
3w

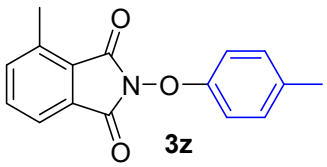
^{13}C NMR, $CDCl_3$,101 MHz




7.76
7.74
7.68
7.68
7.67
7.57
7.57
7.57
7.55
7.55
7.55
7.55
7.12
7.09
7.07
7.07
7.06
7.05
7.05
2.51
2.29
3y
1H NMR, CDCl3, 400 MHz
1.00
0.97
1.04
4.07
3.24
3.15
f1 (ppm)

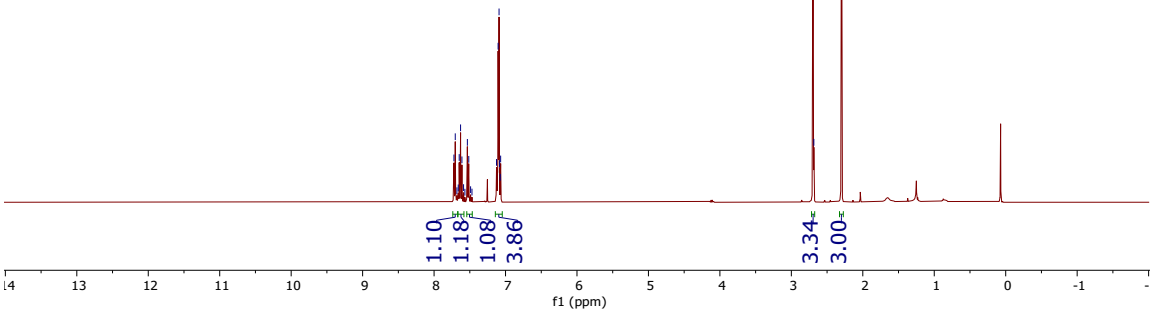

163.24
163.14
156.99
146.34
135.29
134.18
130.07
129.06
126.06
124.42
123.85
114.64
77.38 cdcl3
77.06 cdcl3
76.75 cdcl3
22.14
20.62
20.60
3y
13C NMR, CDCl3, 101 MHz
f1 (ppm)

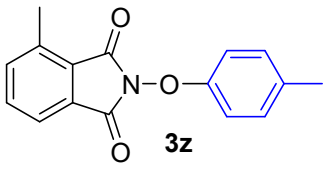




3z

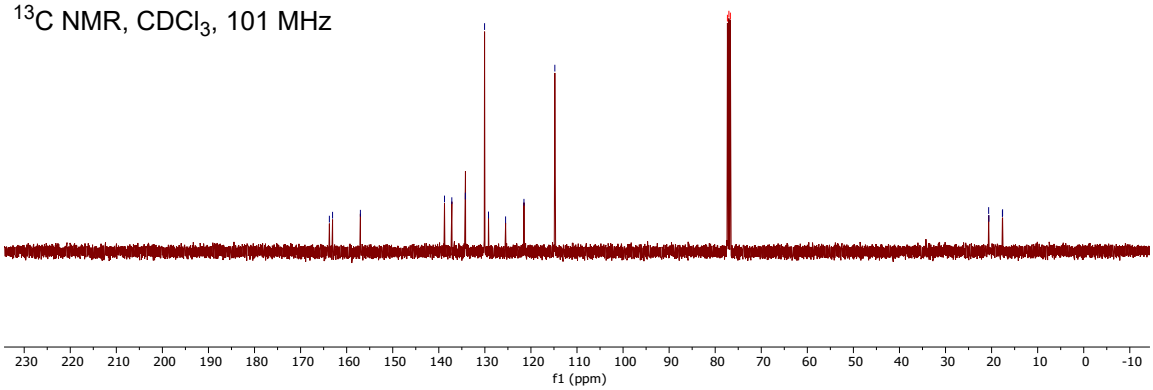
^{1}H NMR, $CDCl_3$, 400 MHz

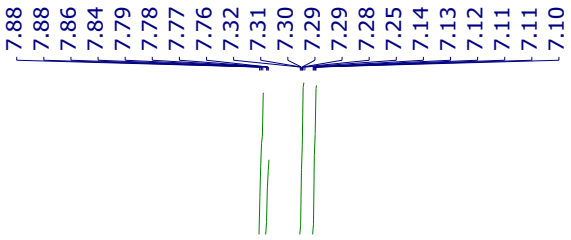


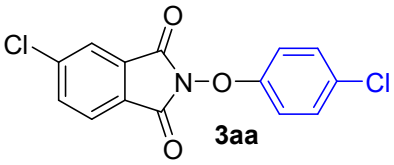


3z

^{13}C NMR, $CDCl_3$, 101 MHz

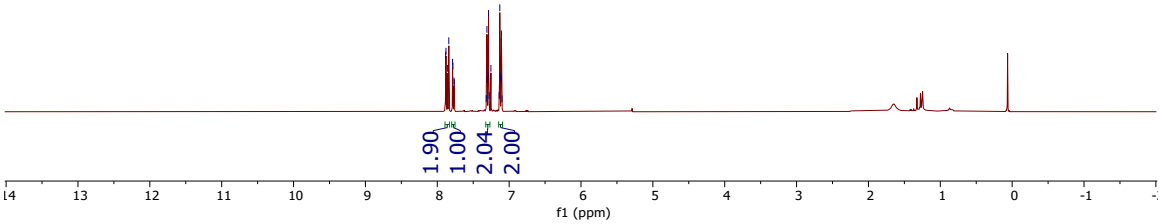


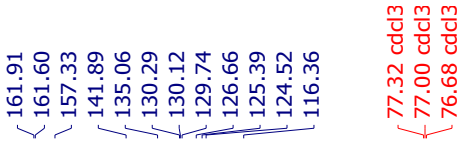


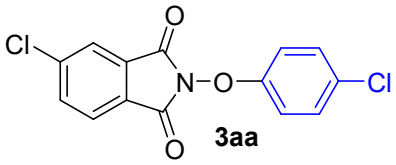


3aa

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

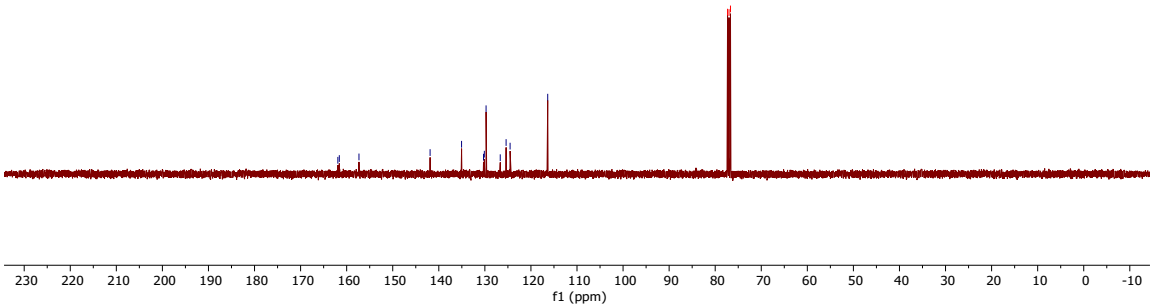


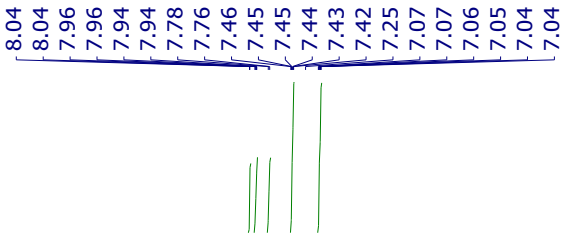


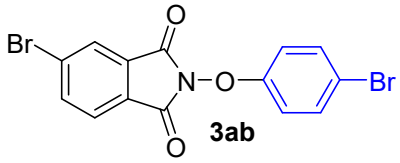


3aa

^{13}C NMR, $CDCl_3$,101 MHz

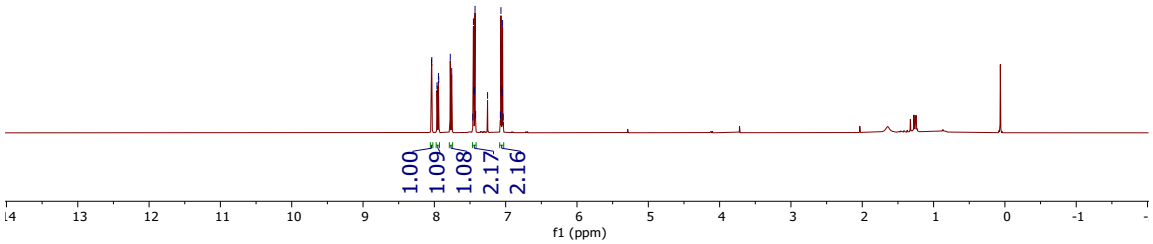


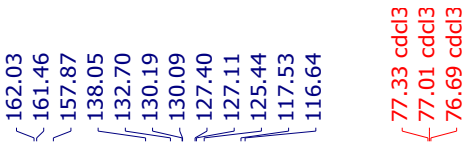


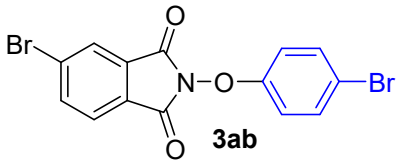


3ab

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

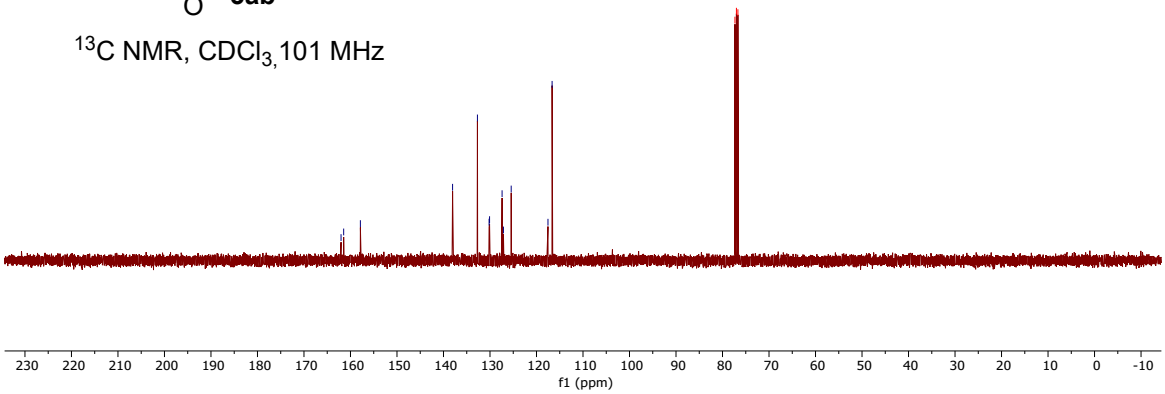


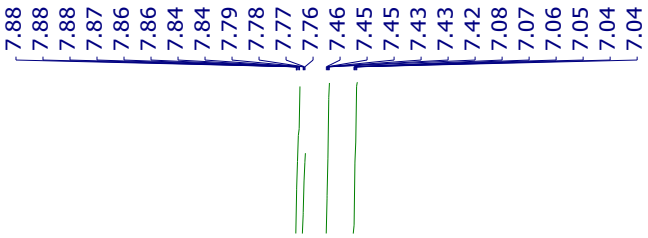


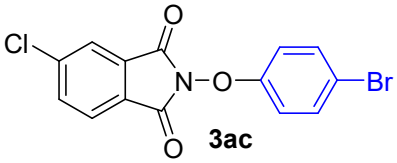


3ab

^{13}C NMR, $CDCl_3$, 101 MHz

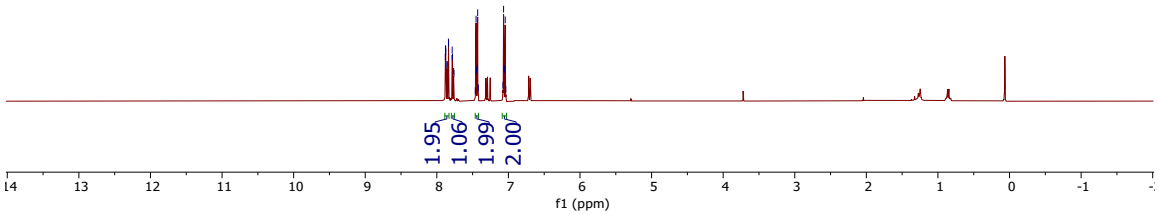


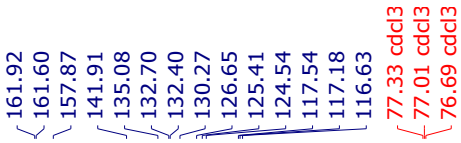


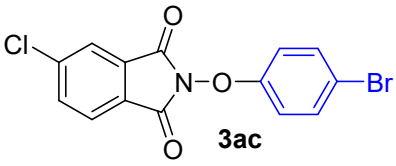


3ac

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

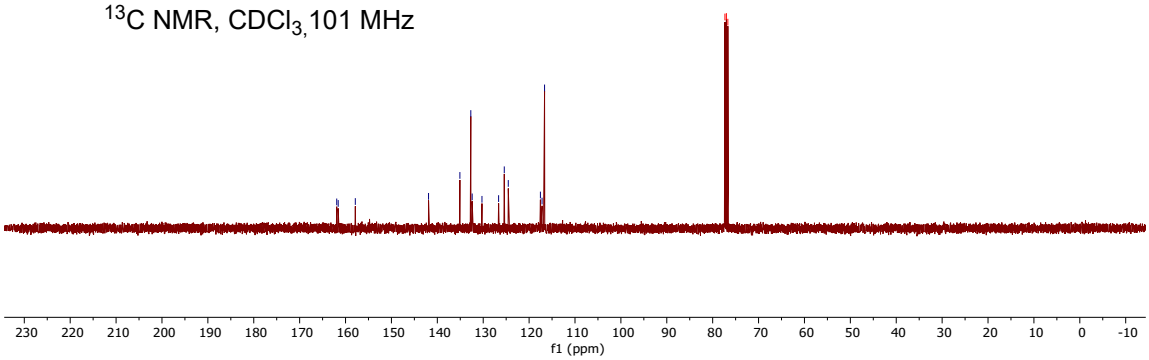


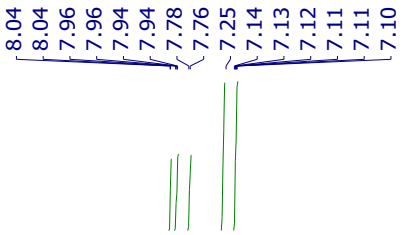


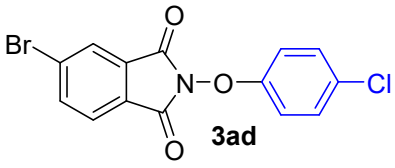


3ac

^{13}C NMR, $CDCl_3$, 101 MHz

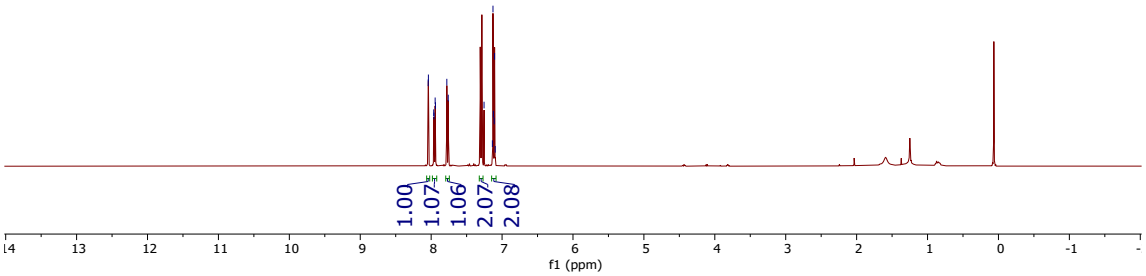


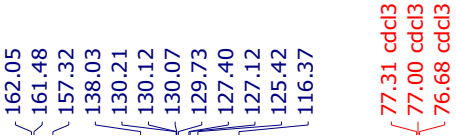


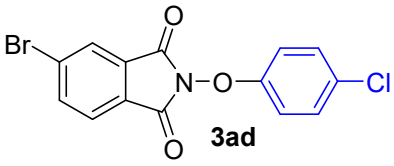


3ad

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

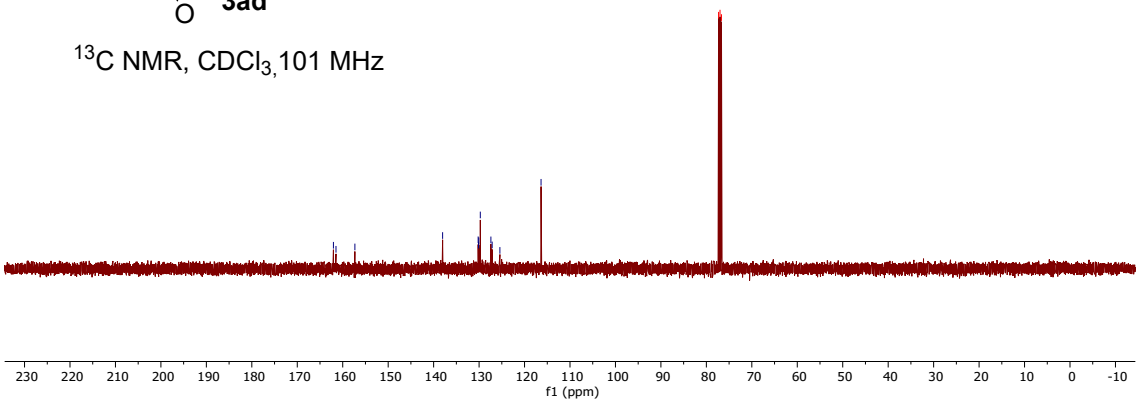


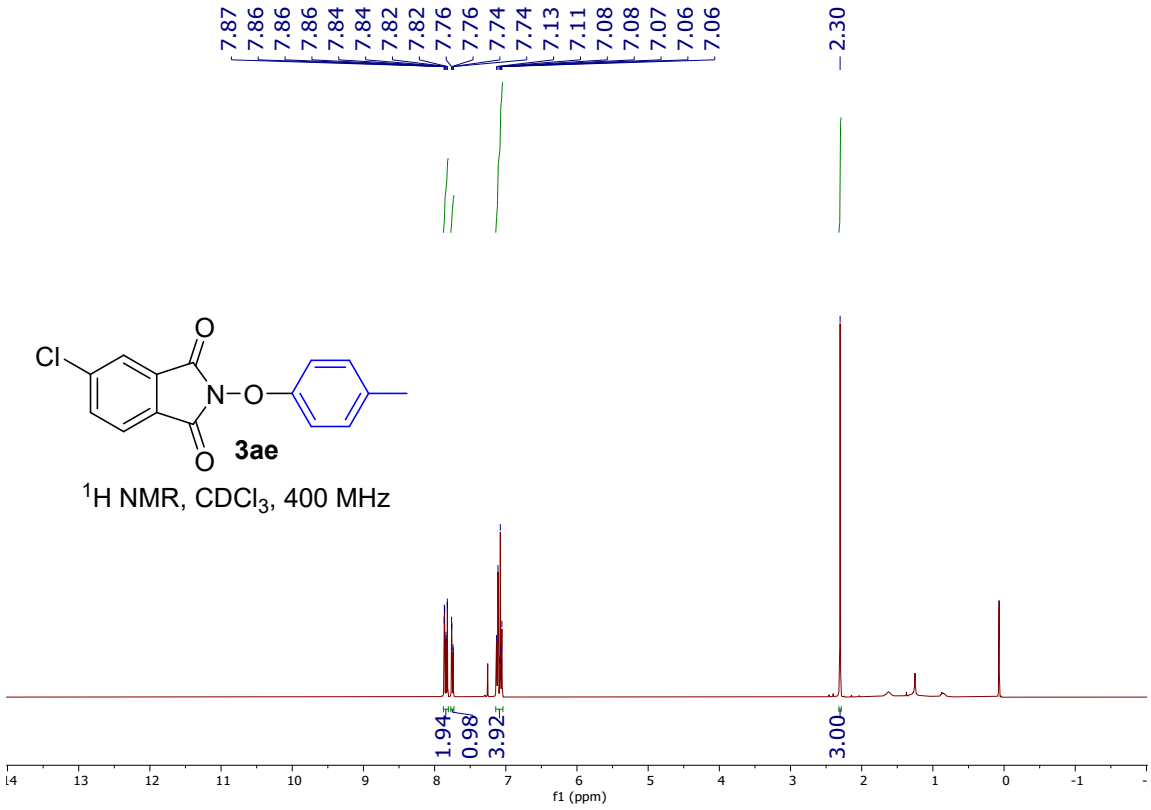




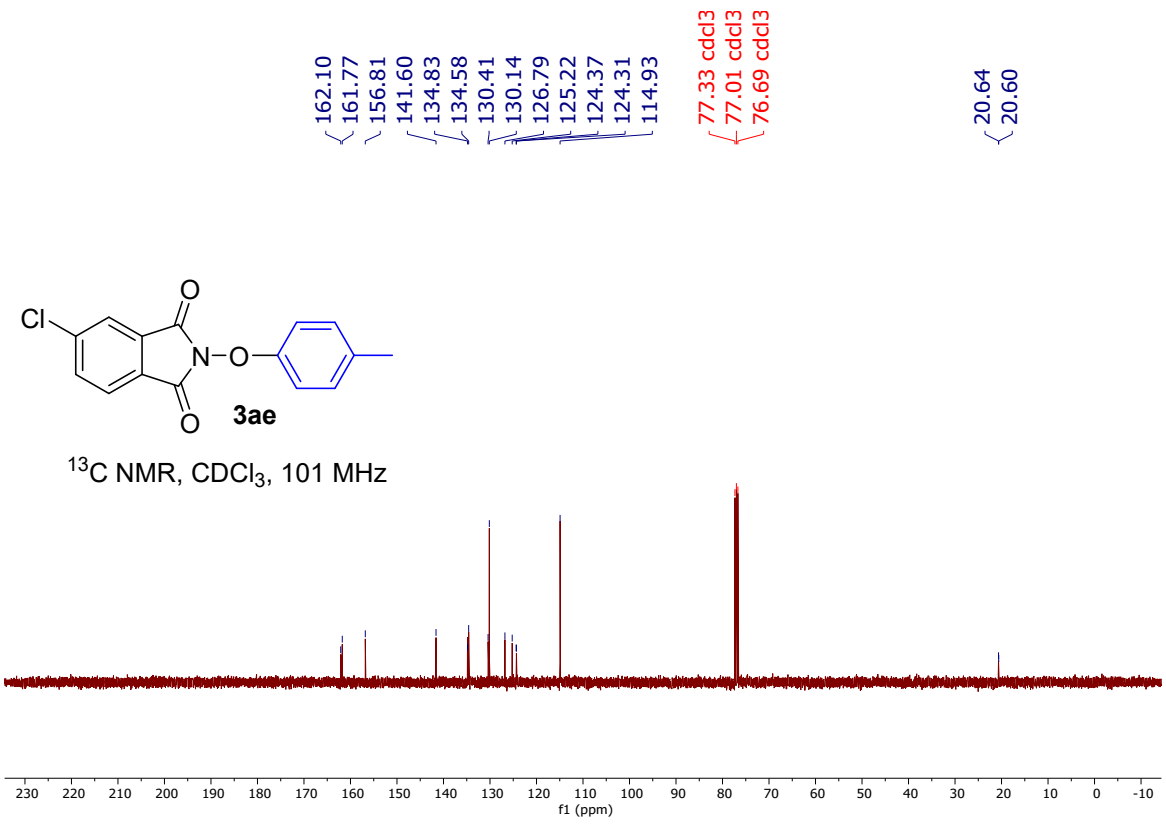
3ad

^{13}C NMR, $CDCl_3$, 101 MHz

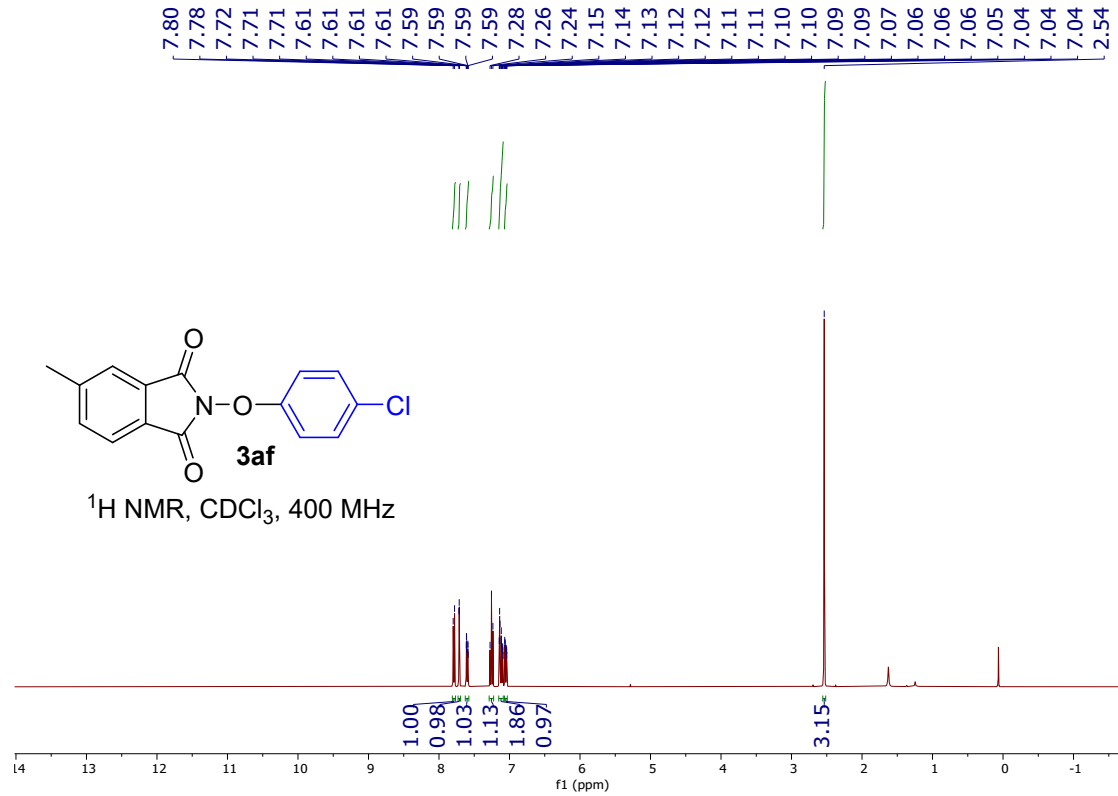




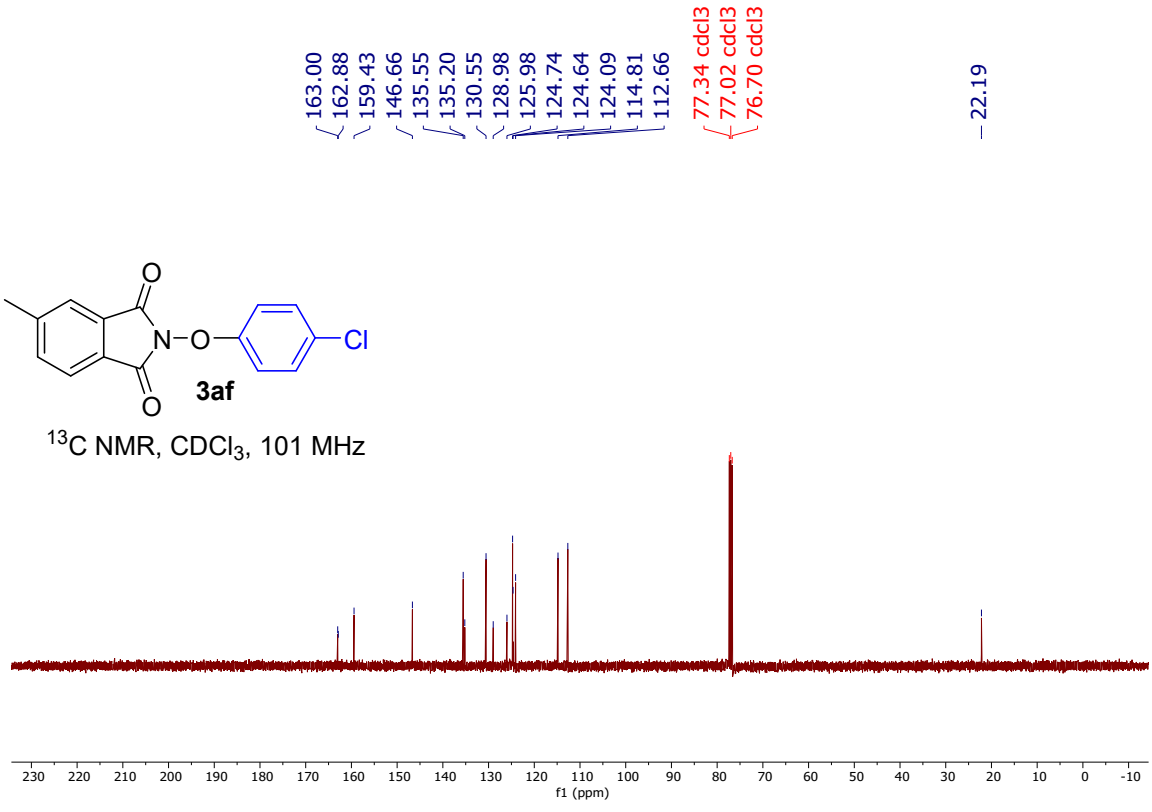
^{1}H NMR, $CDCl_3$, 400 MHz



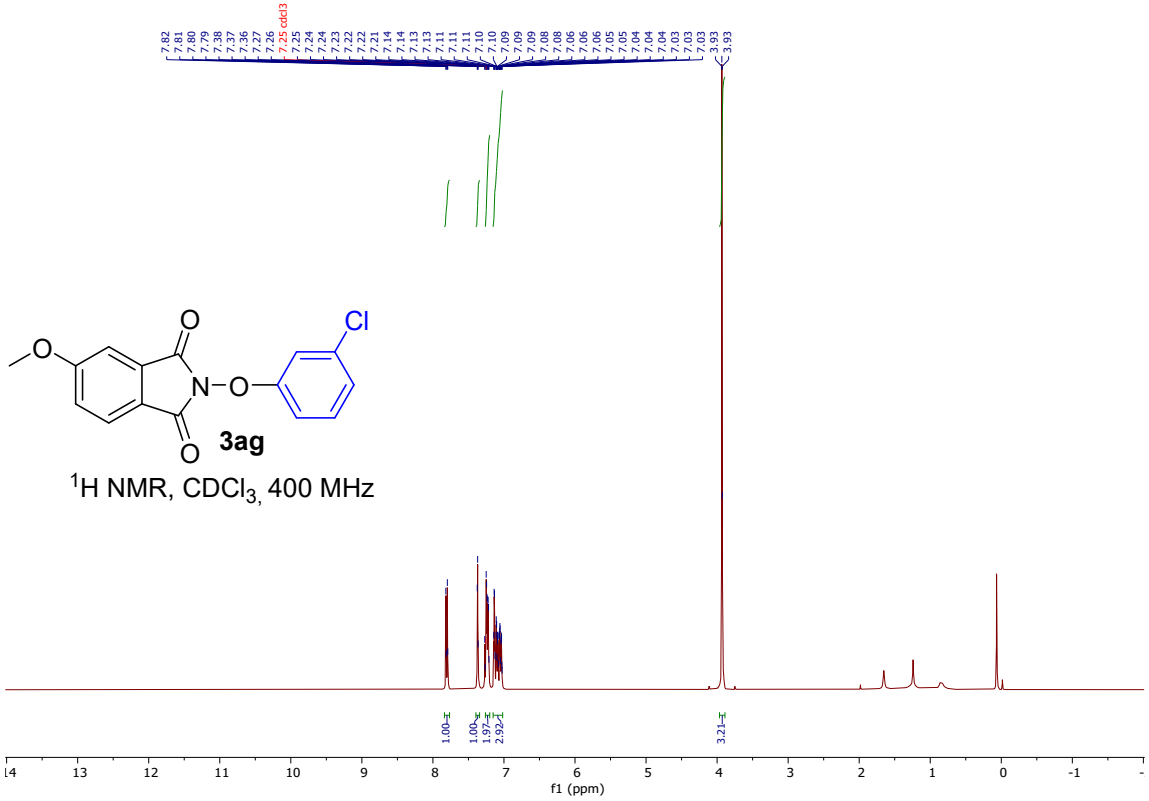
^{13}C NMR, $CDCl_3$, 101 MHz



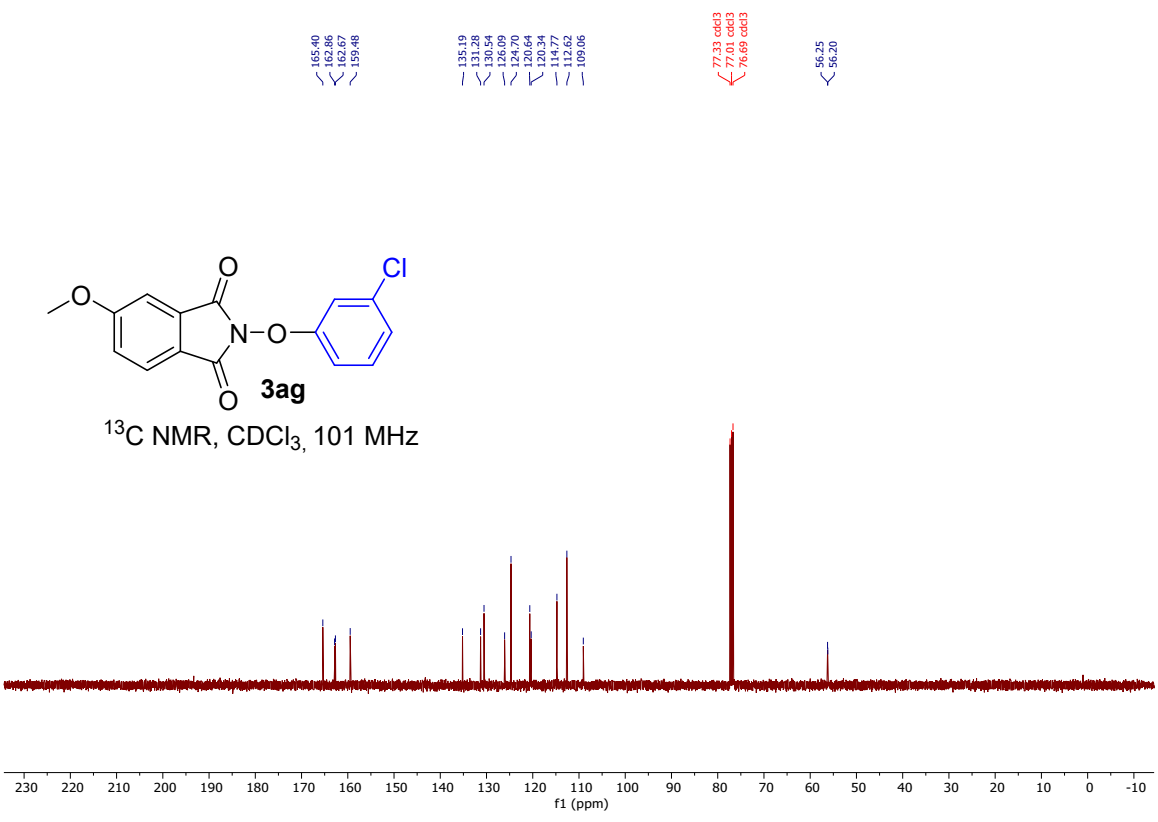
^{1}H NMR, $CDCl_3$, 400 MHz



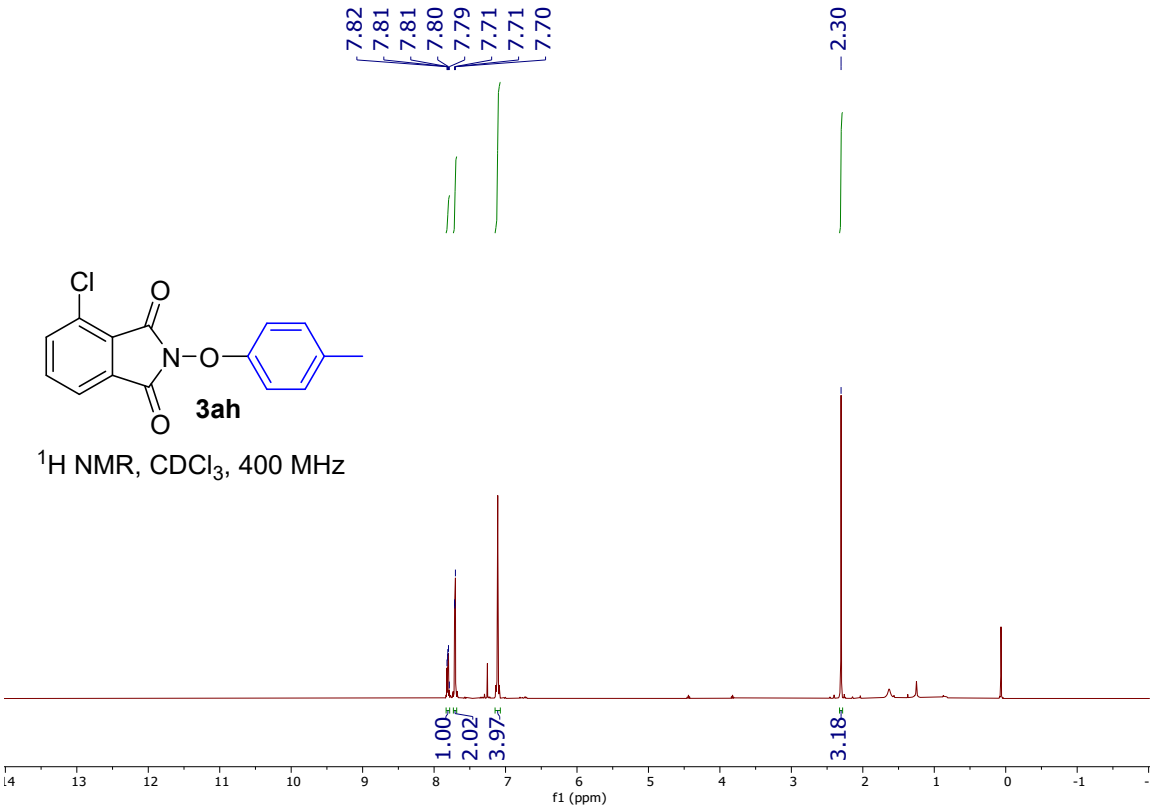
^{13}C NMR, $CDCl_3$, 101 MHz



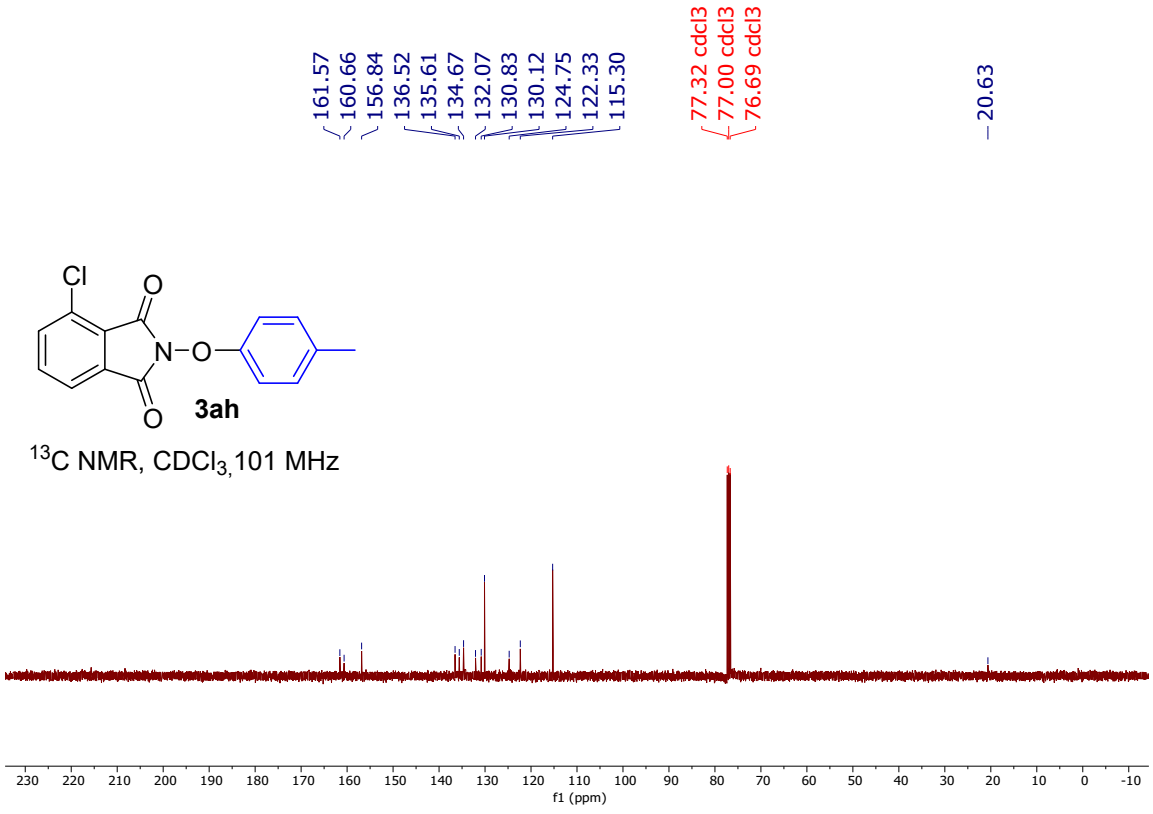
^{1}H NMR, $CDCl_3$, 400 MHz



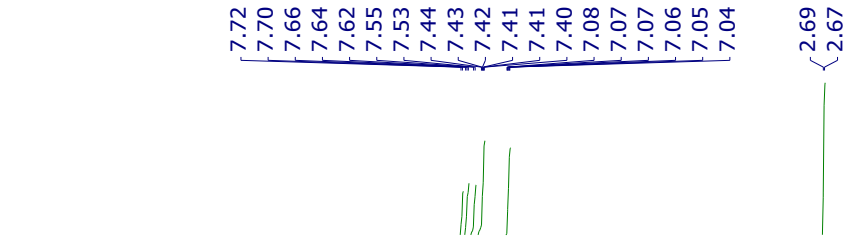
^{13}C NMR, $CDCl_3$, 101 MHz

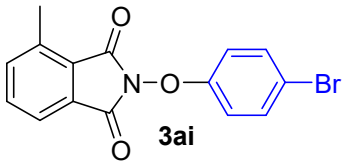


^{1}H NMR, $CDCl_3$, 400 MHz



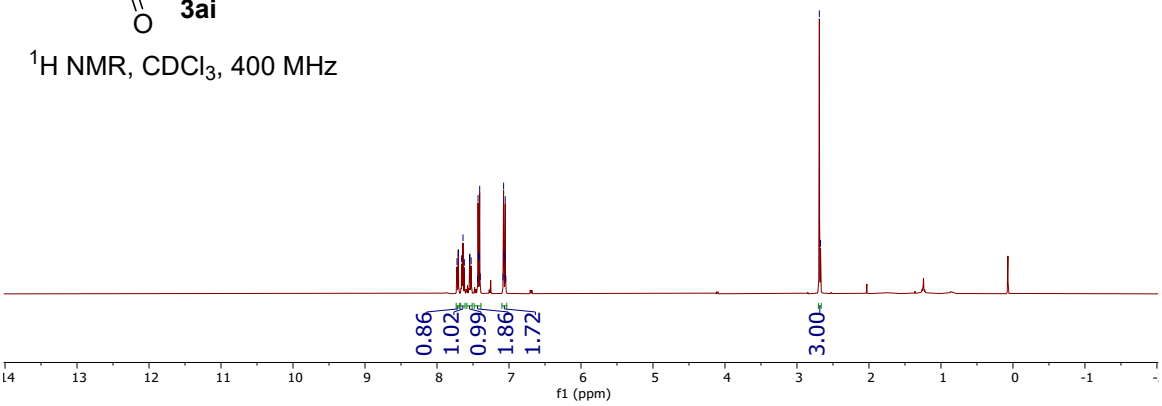
^{13}C NMR, $CDCl_3$, 101 MHz

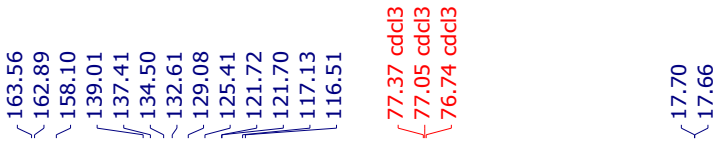


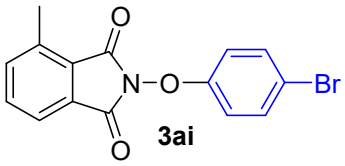


3ai

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

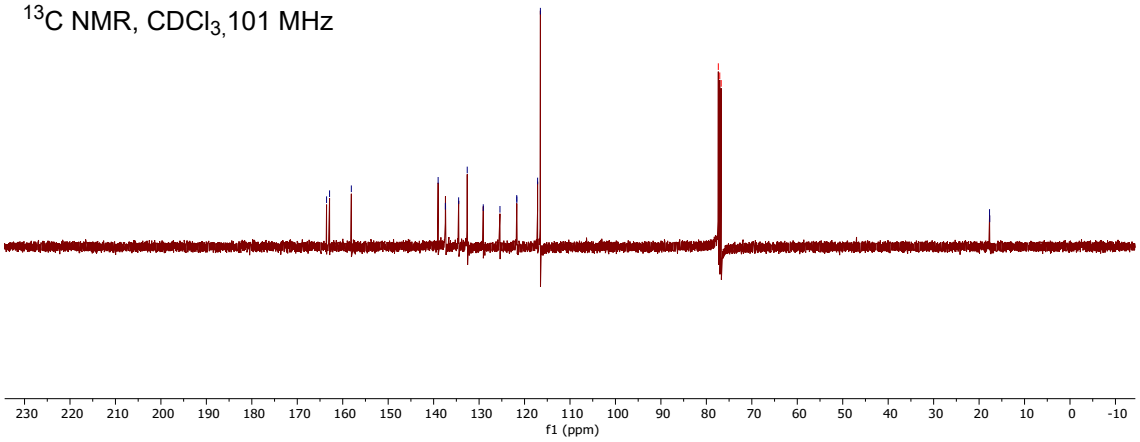


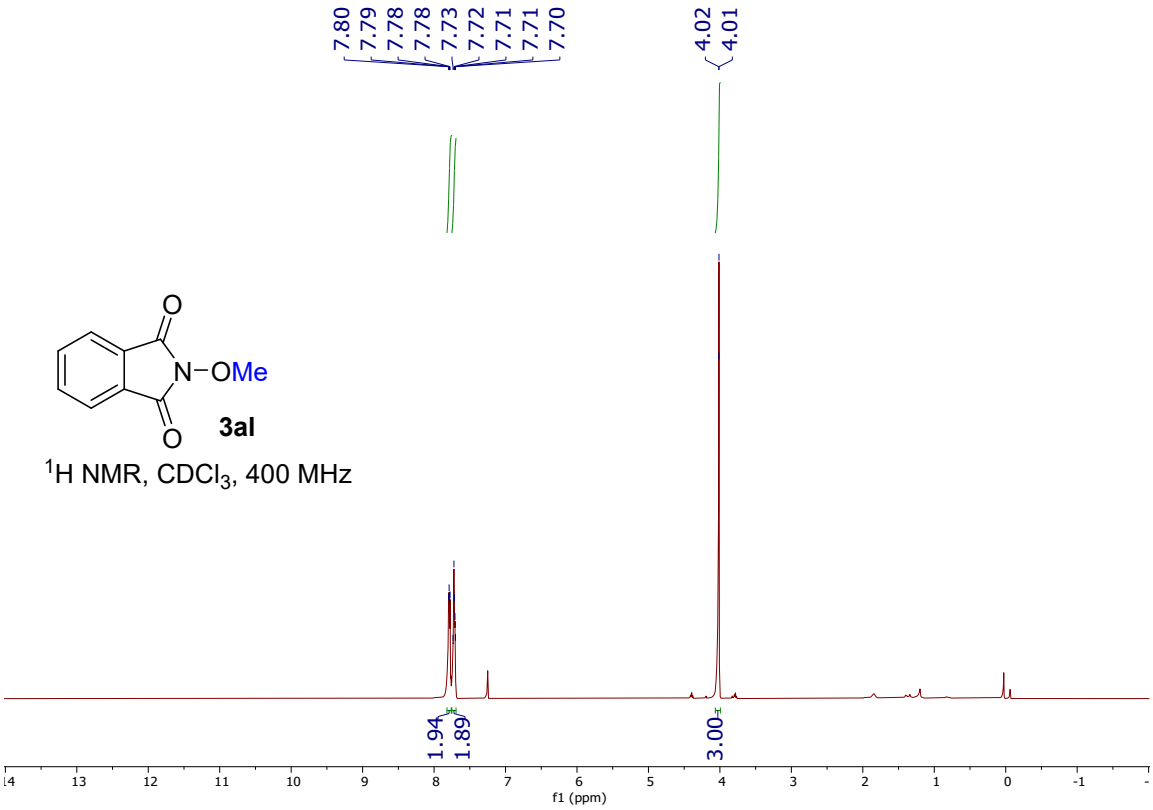




3ai

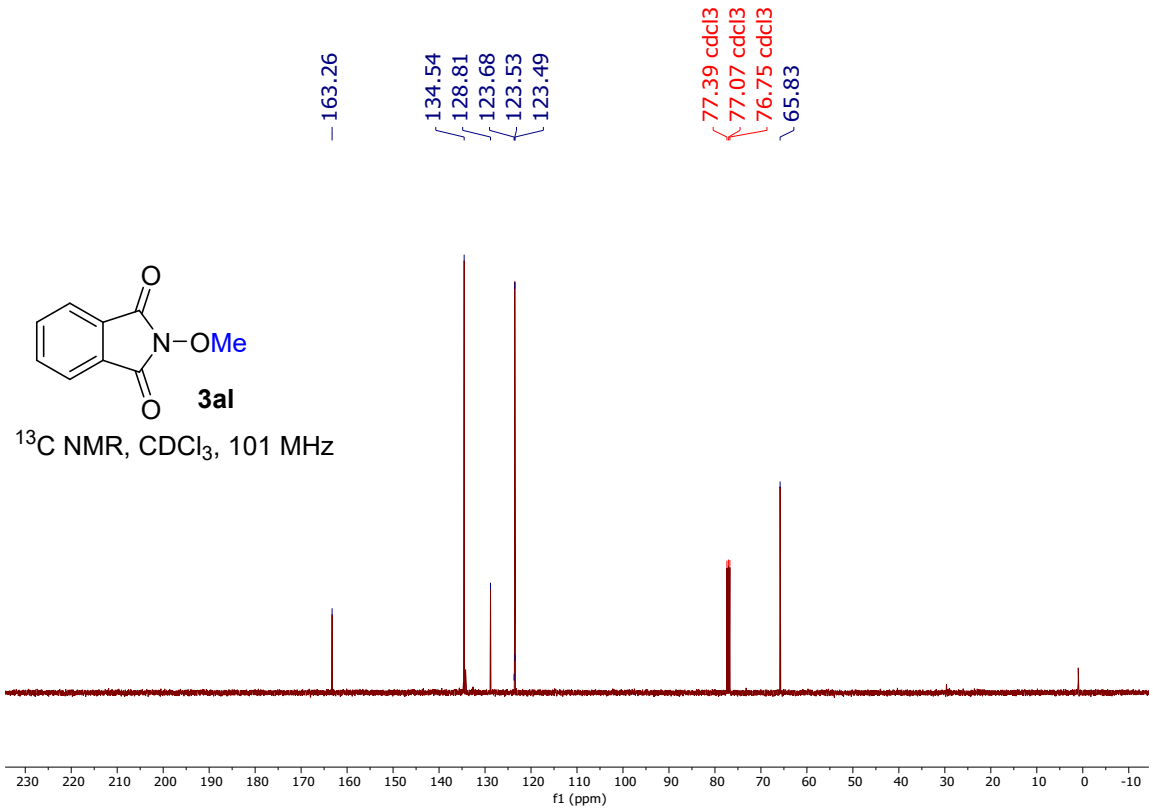
^{13}C NMR, $CDCl_3$, 101 MHz





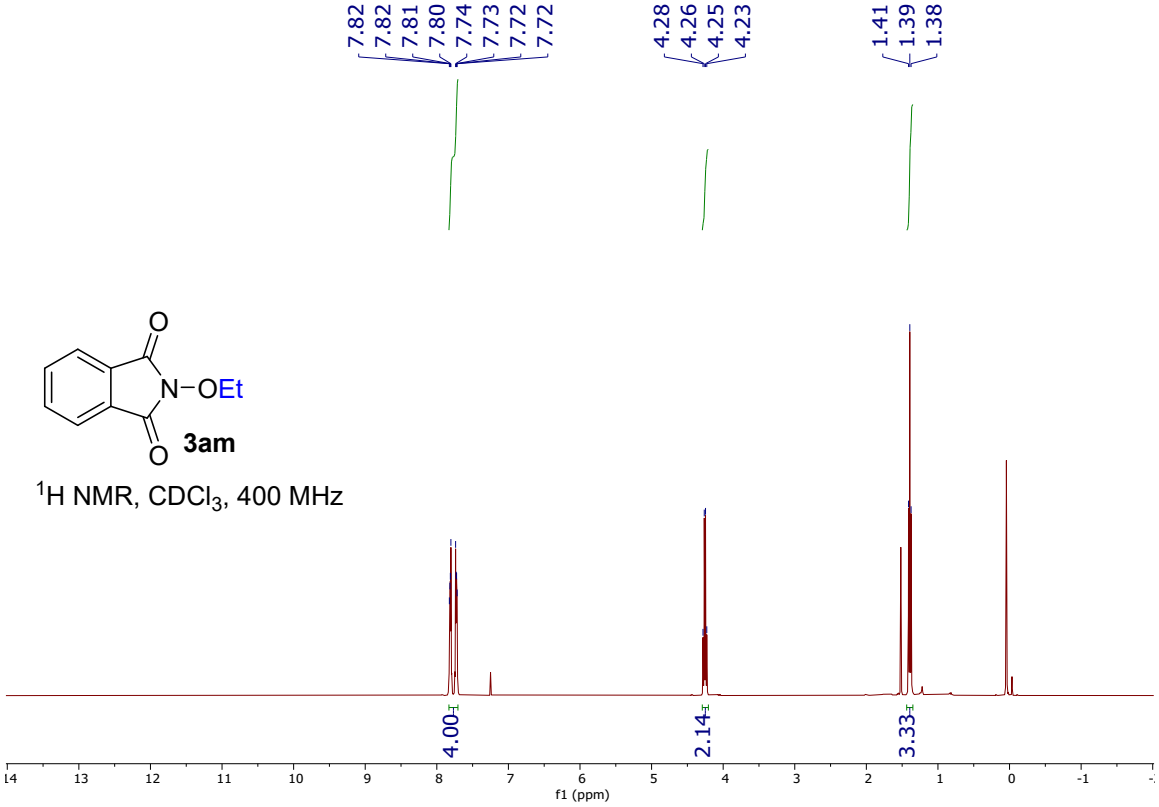
3al

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



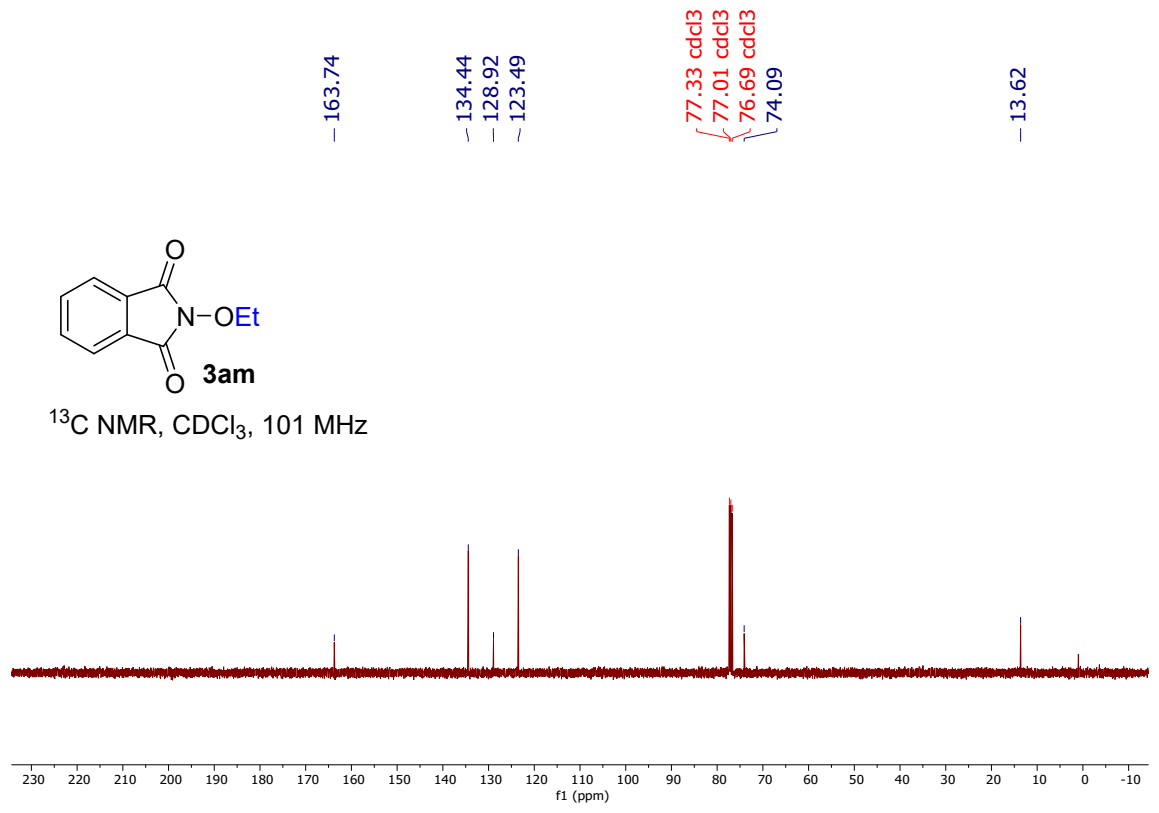
3al

^{13}C NMR, $CDCl_3$, 101 MHz



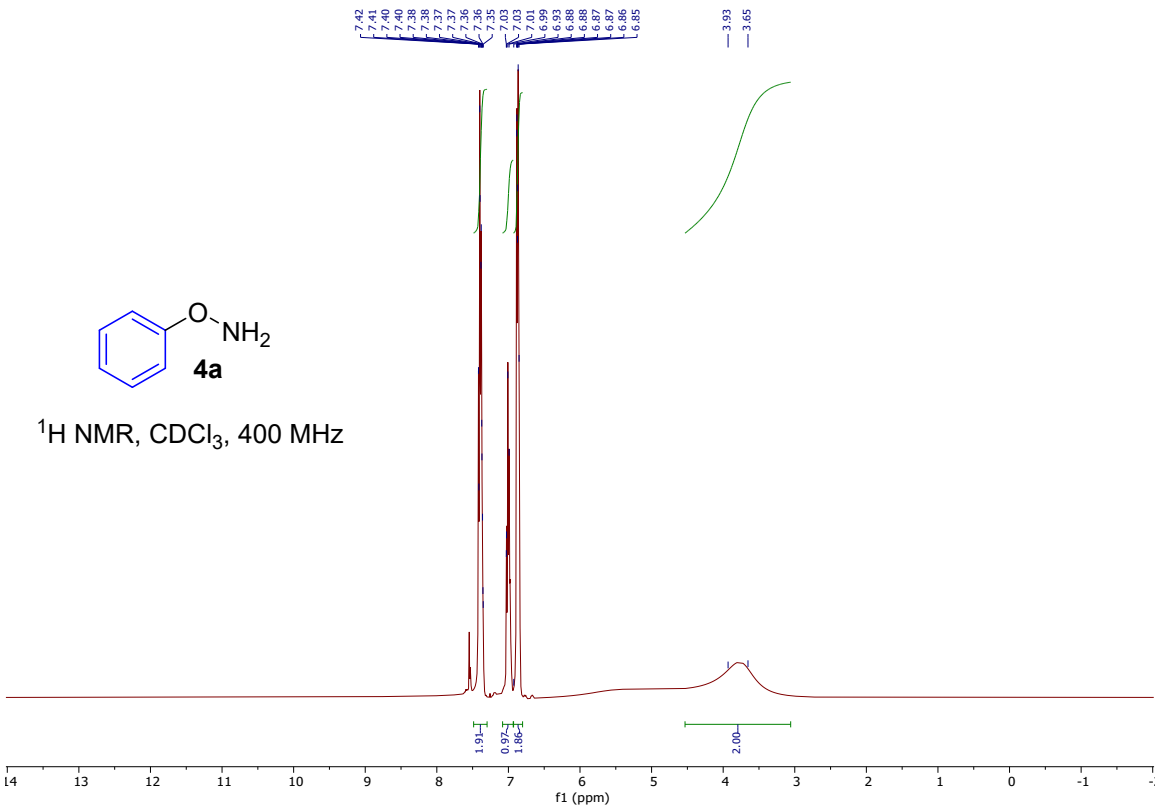
3am

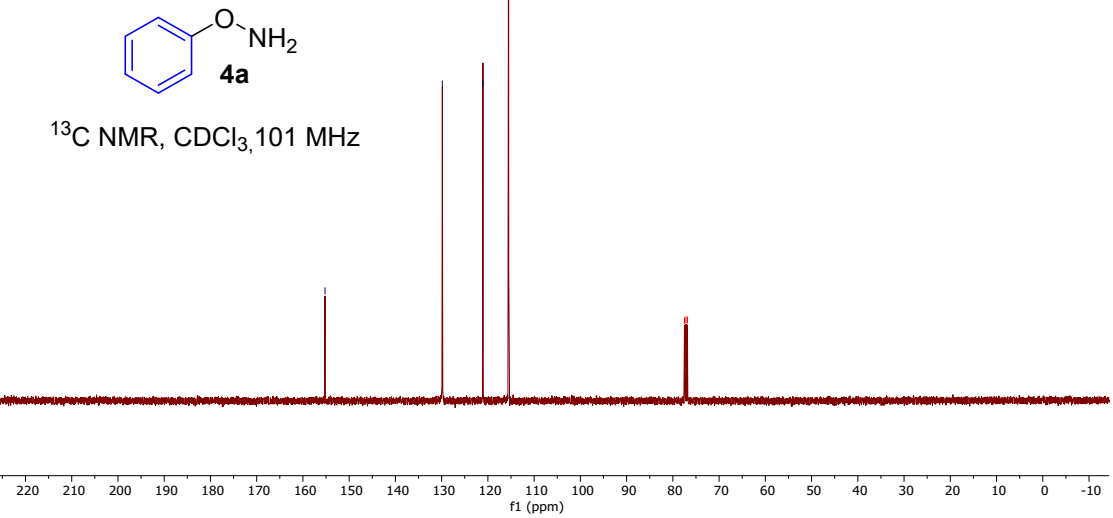
^{1}H NMR, $CDCl_3$, 400 MHz



3am

^{13}C NMR, $CDCl_3$, 101 MHz





4a

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

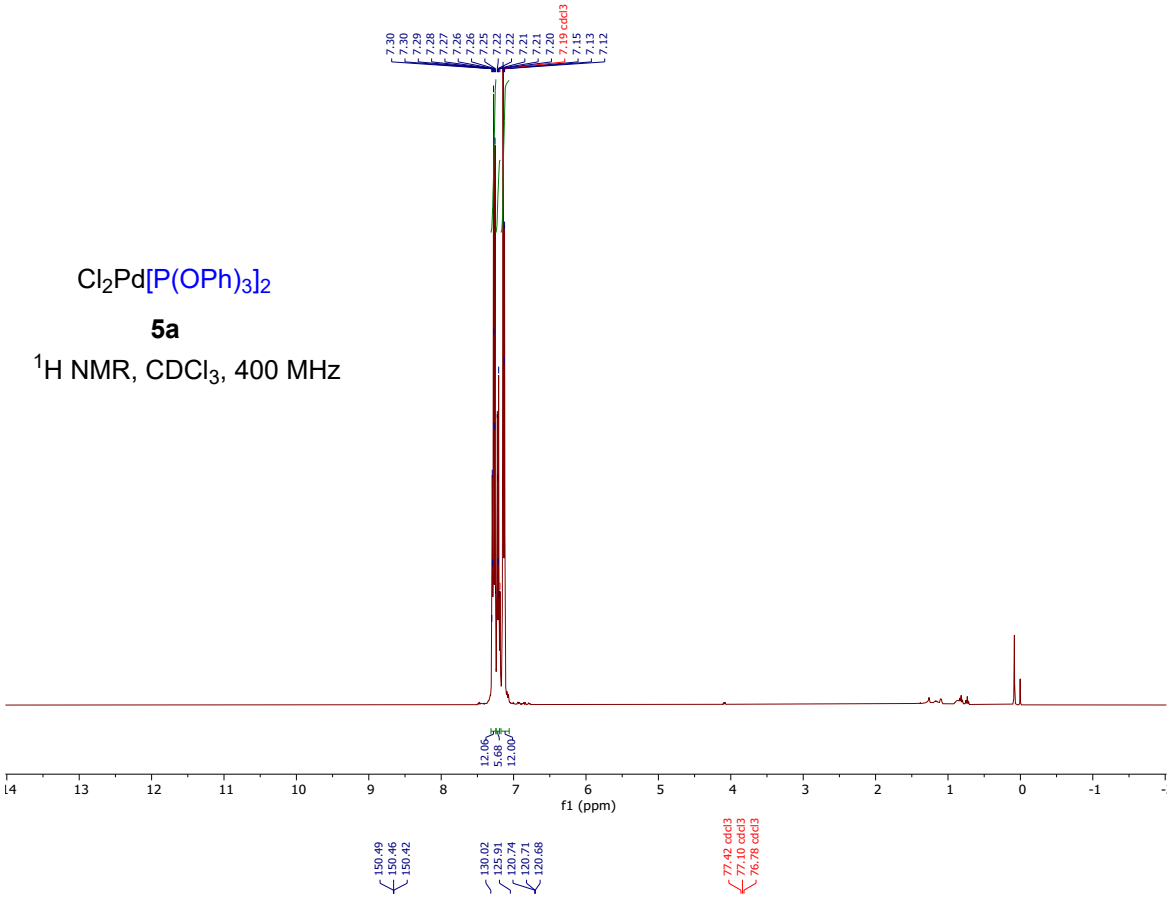
4a

^{13}C NMR, $CDCl_3$, 101 MHz

$Cl_2Pd[P(OPh)_3]_2$

5a

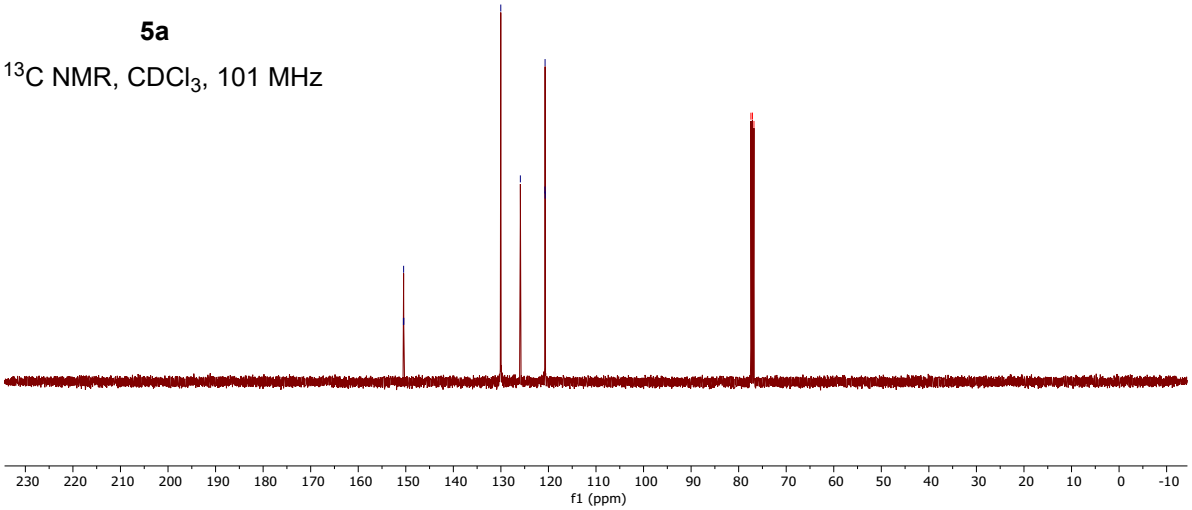
^1H NMR, $CDCl_3$, 400 MHz

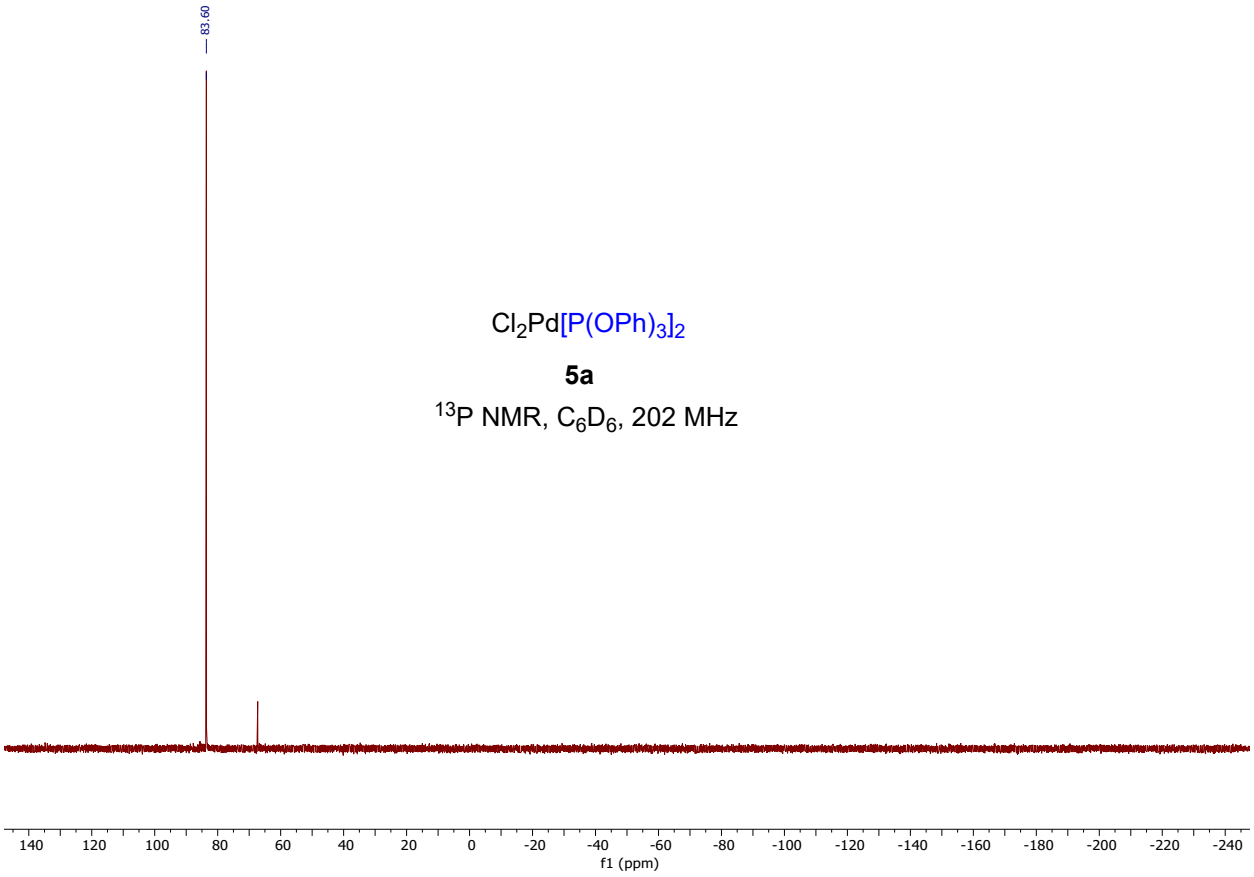


$Cl_2Pd[P(OPh)_3]_2$

5a

^{13}C NMR, $CDCl_3$, 101 MHz





$Cl_2Pd[P(OPh)_3]_2$

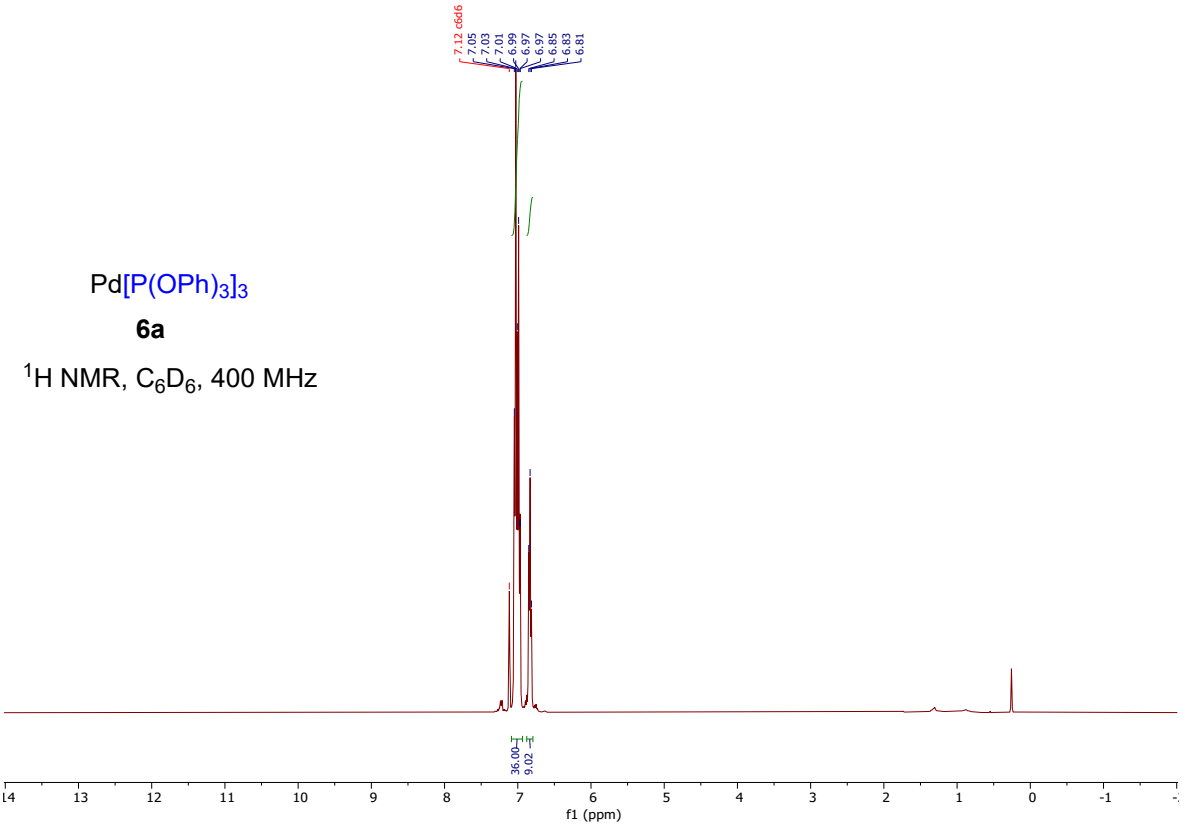
5a

^{13}P NMR, C_6D_6, 202 MHz

Pd[P(OPh)$_3$]$_3$

6a

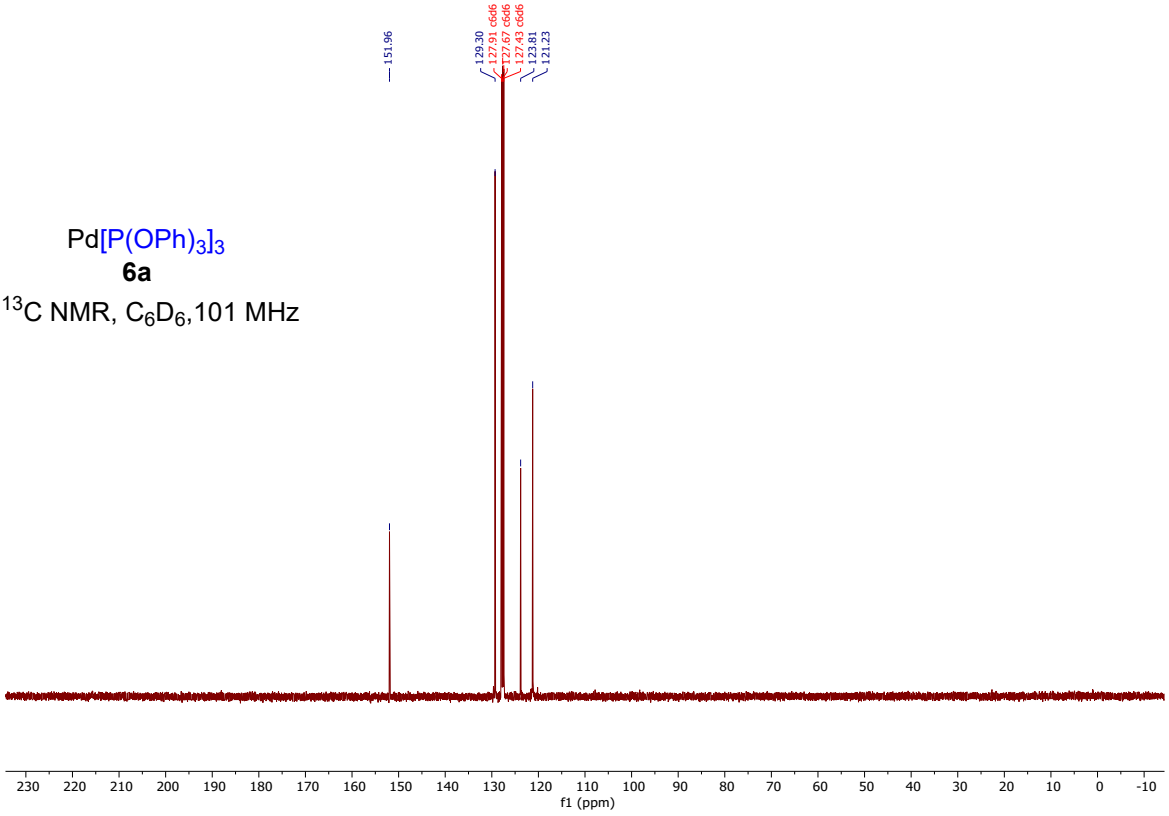
^{1}H NMR, C_6D_6, 400 MHz

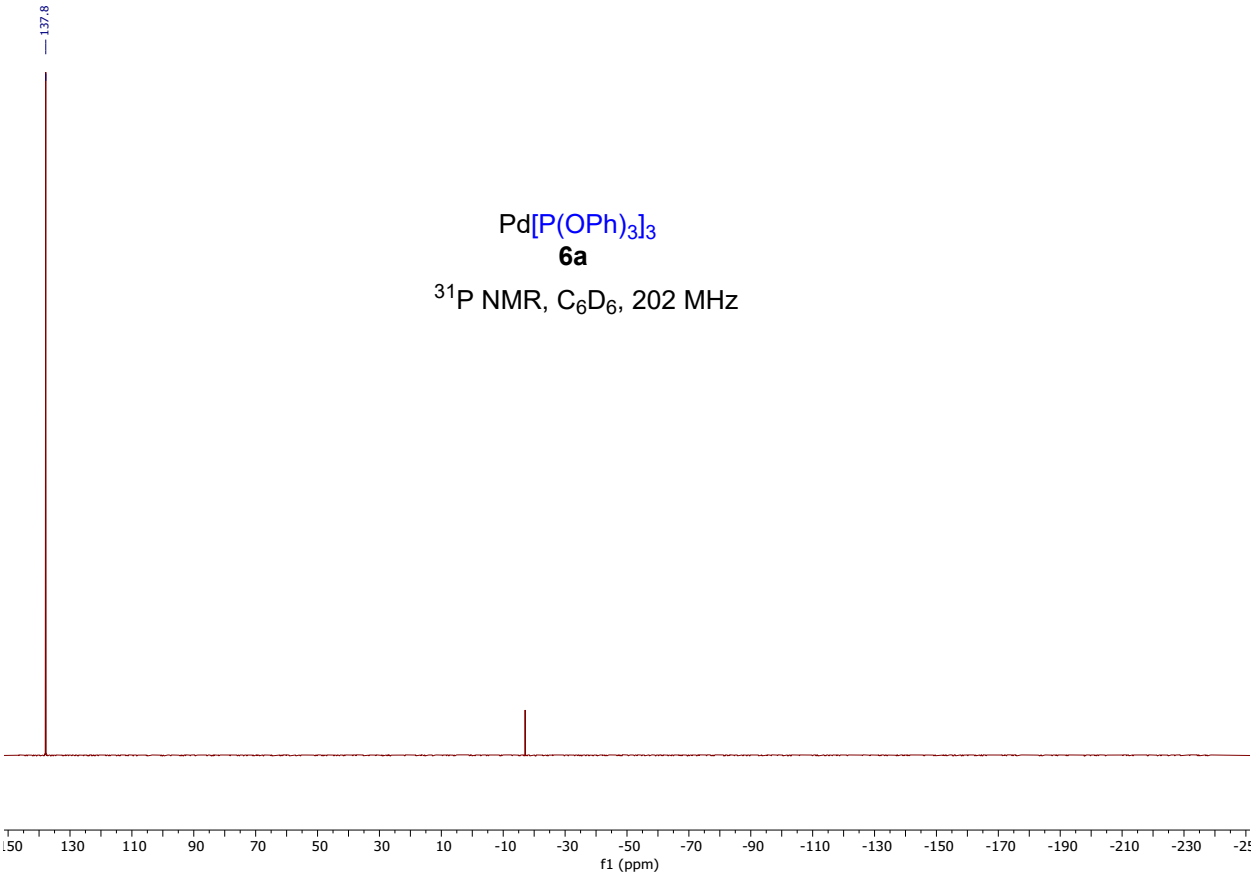


Pd[P(OPh)$_3$]$_3$

6a

^{13}C NMR, C_6D_6,101 MHz





$Pd[P(OPh)_3]_3$

6a

^{31}P NMR, C_6D_6, 202 MHz

14. References

[1] B. Lv, P. Gao, S. Zhang, X. Jia, M. Wang, Y. Yuan, *Org. Chem. Front.* **2021**, *8*, 5440-5445.

[2] a) M. G. vel Górniak, A. Czernicka, P. Młynarz, W. Balcerzak, P. Kafarski, *Beilstein J. Org. Chem.* **2014**, *10*, 741-745; b) M. Mao, L. Zhang, H. Yao, L. Wan, Z. Xin, *ACS omega* **2020**, *5*, 9503-9509; c) M. Oba, Y. Okada, K. Nishiyama, W. Ando, *Org. Lett.* **2009**, *11*, 1879-1881; d) C. T. Estorach, A. Orejón, A. M. Masdeu-Bultó, *Green Chem.* **2008**, *10*, 545-552; eY. Zhang, Z. Cai, Y. Chi, X. Zeng, S. Chen, Y. Liu, G. Tang, Y. Zhao, *Org. Lett.* **2021**, *23*, 5158-5163.

[3] K. Wang, M. Tong, Y. Yang, B. Zhang, H. Liu, H. Li, F. Zhang, *Green Chem.* **2020**, *22*, 7417-7423.

[4] a) A. M. Trzeciak, J. J. Ziółkowski, *Monatsh. Chem.* **2000**, *131*, 1281-1291; b) A. M. Trzeciak, Z. Ciunik, J. J. Ziółkowski, *Organometallics* **2002**, *21*, 132-137.

[5] T. Yurino, S. Saito, M. Ichihashi, T. Ohkuma, *J. Org. Chem.* **2022**, *87*, 2864-2872.

[6] R. Ghosh, B. Olofsson, *Org. lett.* **2014**, *16*, 1830-1832.

[7] L. C. M. Castro, N. Chatani, *Synthesis* **2014**, *46*, 2312-2316.

[8] a) J. N. Kim, K. M. Kim, E. K. Ryu, *Synth. commun.* **1992**, *22*, 1427-1432; b) N. Pawar, U. Kapadi, D. Hundiwale, P. Kumbhar, *J. Sci. Ind. Res.* **2002,** 61, 454-456**.**