Supporting Information

Monodentate Transient Directing Group Assisted Ruthenium(II)-Catalyzed Direct ortho C−H Imidation of Benzaldehydes for Diverse Synthesis of Quinazoline and Fused Isoindolinone

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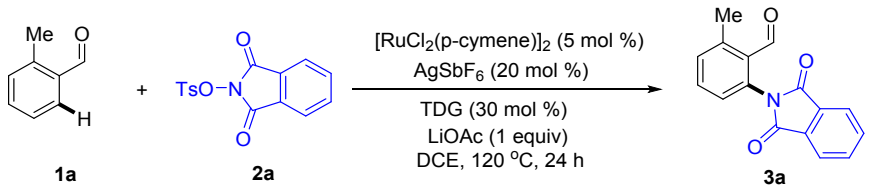
1 General Information

Aldehydes(**1**) and transient directing groups were obtained from the commercial sources. Substrate **2** is synthesized following literature procedures. $[RuCl_2(p\text{-cymene})]_2$ was obtained from Strem. Solventswere obtained from Sigma-Aldrich, Alfa-Aesar, Oakwood, and Acros and used directly without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel 60-F254. Visualization was carried out with UV light. ^1H NMR was recorded on Bruker instrument (500MHz). Chemical shifts were quoted in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q =quartet, m = multiplet, br = broad. Coupling constants, *J*, were reported in Hertz unit (Hz). ^{13}C NMR spectra were recorded on Bruker instrument (126MHz), and were fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to either the center line of a triplet at 77.0 ppm of chloroform-*d* or referenced to the center line of a septet at 39.52 ppm of DMSO-d_6. High-resolution mass spectra (HRMS) was recorded on an Agilent Mass spectrometer using ESI-TOF (electrospray ionization-time of flight).

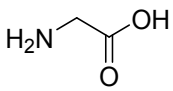
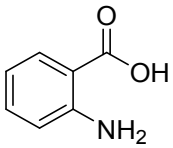
2 Experimental Section

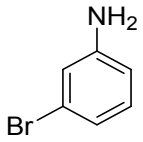
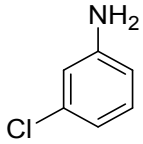
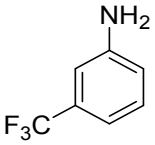
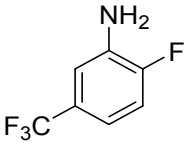
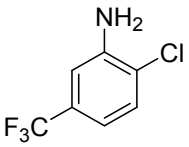
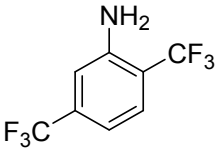
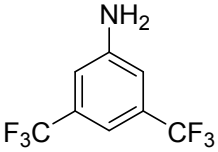
2.1 Optimization of Conditions

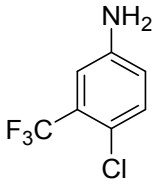
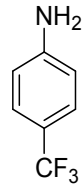
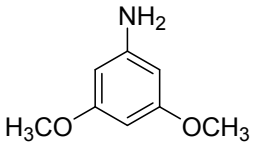
Table S1 Screening of TDG



1a + **2a** → **3a**

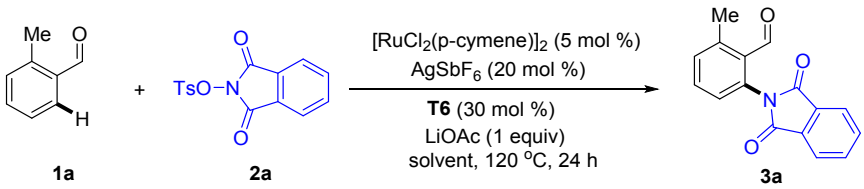
Entry	TDG	Yield **3a** (%)
1	 **T1**	N.R.
2	 **T2**	N.R.

3	 **T3**	34
4	 **T4**	36
5	 **T5**	56
6	 **T6**	81
7	 **T7**	41
8	 **T8**	5
9	 **T9**	49

10	 **T10**	65
11	 **T11**	57
12	 **T12**	22

Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.4 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5 mol %), $AgSbF_6$ (20 mol %), TDG (30 mol %), LiOAc (0.2 mmol), DCE (2.0 mL), 120 °C, 24 h. Isolated yields.

Table S2 Screening of solvent



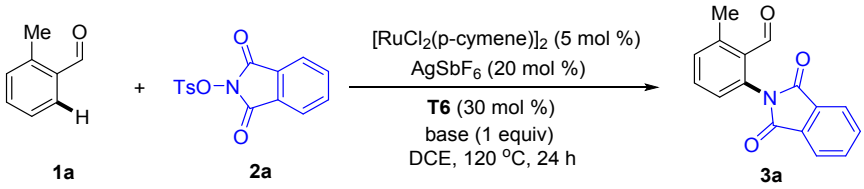
1a **2a** **3a**

Entry	Solvent	Yield **3a** (%)
1	DMSO	0
2	HFIP	0
3	MeCN	0
4	DMF	0
5	AcOH	0
6	MeOH	0
7	1,4-dioxane	11
8	$PhCH_3$	0
9	DCE	81

10	DCM	55
11	DCE:DCM = 1:1	60

Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.4 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5 mol %), $AgSbF_6$ (20 mol %), **T6** (30 mol %), LiOAc (0.2 mmol), solvent (2.0 mL), 120 °C, 24 h. Isolated yields.

Table S3 Screening of base

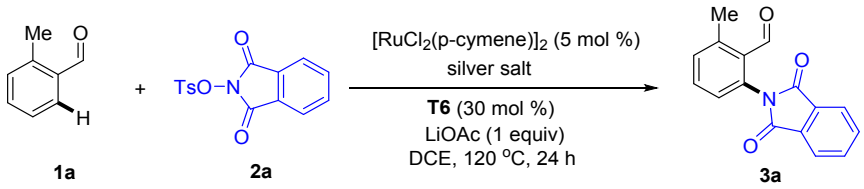


1a **2a** **3a**

Entry	Base	Yield **3a** (%)
1	NaOAc(1.0 equiv)	59
2	ZnOAc(1.0 equiv)	61
3	LiOAc(1.0 equiv)	81
4	KOAc(1.0 equiv)	57
5	CsOAc(1.0 equiv)	60
6	AgOAc(1.0 equiv)	trace
7	LiOAc(2.0 equiv)	75
8	LiOAc(0.5 equiv)	73
9	no	56

Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.4 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5 mol %), $AgSbF_6$ (20 mol %), **T6** (30 mol %), base (0.2 mmol), DCE (2.0 mL), 120 °C, 24 h. Isolated yields.

Table S4 Screening of silver salt



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

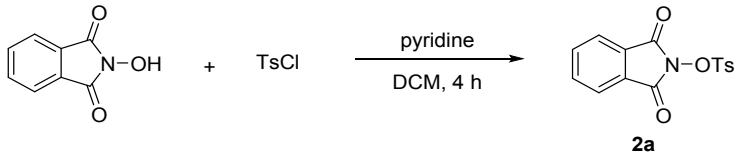
Entry	Silver salt	Yield 3a (%)
1	no	0

2	Ag_2CO_3 (0.2 eq.)	0
3	Ag_2SO_4 (0.2 eq.)	0
4	$AgNO_3$ (0.2 eq.)	0
5	AgTFA (0.2 eq.)	0
6	$AgSbF_6$ (0.2 eq.)	81
7	$AgSbF_6$ (0.3eq.)	75
8	$AgSbF_6$ (0.1 eq.)	70

Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), **2a** (0.4 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5 mol %), **T6** (30 mol %), LiOAc (0.2 mmol), silver salt, DCE (2.0 mL), 120 °C, 24 h. Isolated yields.

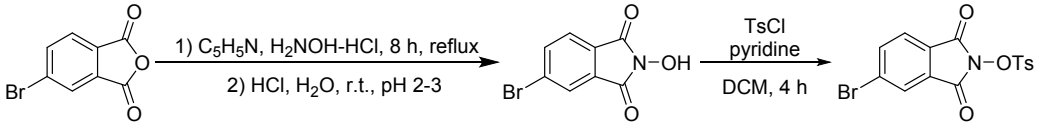
2.2 General Procedures

2.2.1 Synthesis of **2**



2a

Following a previously reported procedure[1], to a solution of 2-hydroxy-1H-isoindole-1,3(2H)-dione (1.00 g, 6.1 mmol) and TsCl (1.1 equiv) in DCM (20 mL) was added pyridine (2.0 mL) drop wise for 15 minutes. The solvent was removed in vacuo after performing the reaction for 4h. Subsequently, distilled water (30 mL) with a drop of concentrated 1N hydrochloric acid was added to the crude mixture. The solid product was filtered and washed with water (twice) to produce pure compound **2a** (90% yield).

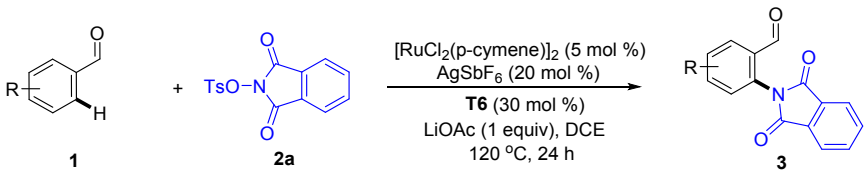


2b

According to the existing literature[2], at room temperature, hydroxylamine hydrochloride (416.9 mg, 6 mmol) was added to 50 mL of pyridine and stirred for 20 minutes until the solid was completely dissolved. Then, 1.36 g (6 mmol) of 5-bromoisobenzofuran-1,3-dione was added, and the mixture was refluxed for 8 hours. After cooling to room temperature, 20 mL of distilled water was added to the resulting yellow solution. Adjust the pH to 2-3 with 3 M HCl solution. It was washed with cold water, filtered, and evaporated under reduced pressure to obtain the target compound 5-bromo-2-hydroxyisoindoline-1,3-dione as a white powder (62% yield).
Same as the synthesis of 2a above, to a solution of 2-hydroxy-1H-isoindole-1,3(2H)-dione (798.7 mg, 3.3 mmol) and TsCl (1.1 equiv) in DCM (10 mL) was added pyridine (1.0 mL) drop wise for

15 minutes. The solvent was removed in vacuo after performing the reaction for 4h. Subsequently, distilled water (15 mL) with a drop of concentrated 1N hydrochloric acid was added to the crude mixture. The solid product was filtered and washed with water (twice) to produce pure compound **2b** 5-bromo-1,3-dioxoisoindolin-2-yl 4-methylbenzenesulfonate (80% yield).
^{1}H NMR (500 MHz, DMSO) δ 8.14 (d, *J* = 1.6 Hz, 1H), 8.10 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.2 Hz, 2H), 2.46 (s, 3H).
^{13}C NMR (126 MHz, DMSO) δ 161.4, 160.8, 147.7, 138.7, 130.9, 130.7, 130.3, 129.8, 129.6, 127.8, 127.4, 126.4, 21.8.

2.2.2 General Procedure for Ruthenium(II)-Catalyzed C-H Imidation

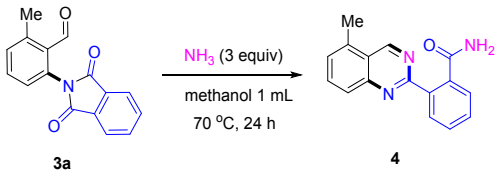


A sealed tube with magnetic stir bar was charged with benzaldehyde (**1**) (0.2 mmol), 1,3-dioxoisoindolin-2-yl 4-methylbenzenesulfonate (**2a**) (0.4 mmol, 126.9 mg), $[RuCl_2(p\text{-cymene})]_2$ (0.01 mmol, 6.1 mg), $AgSbF_6$ (0.04 mmol, 13.7mg), **T6** (0.06 mmol, 10.5 mg), LiOAc (0.2 mmol, 13.2 mg) in air, followed by DCE (2.0 mL). The reaction mixture was stirred at 120 °C for 24 hours. Upon completion, the reaction mixture was cooled to room temperature and filtered through a sand funnel. Next the reaction mixture was quenched by sat. NH_4Cl (aq) (40 mL), and extracted with CH_2Cl_2 (20 mL) for three times. The combined organic layers were washed with water (60 mL), dried over anhydrous Na_2SO_4, and concentrated in vacuo. The crude residue was purified by column chromatography on silica gel using petroleum ether/EtOAc 10:1 as eluent to afford the desired products.

2.2.3 Gram-Scale Preparation of **3a**

The 2-methylbenzaldehyde (**1a**) (10 mmol, 1.2 g), 1,3-dioxoisoindolin-2-yl 4-methylbenzenesulfonate (**2a**) (18 mmol, 5.7 g), $[RuCl_2(p\text{-cymene})]_2$ (0.5 mmol, 0.31 g), $AgSbF_6$ (2 mmol, 0.69 g), **T6** (3.0 mmol, 0.54 g) and LiOAc (10 mmol, 0.66 g) were taken in a sealed tube with magnetic stir bar. Then DCE (40 mL) was added with a syringe and the resulting mixture was heated at 100 ºC for 48 h. After completion of the reaction (TLC monitored), the reaction mixture was filtered through a sand core funnel, and then the solvent was removed by vacuum distillation. The crude product was recrystallized from ethyl acetate and washed with petroleum ether to obtain the product (**3a**) (1.99 g, 75%).

2.2.4 General Procedure to access quinazoline **4**



2-(1,3-dioxoisoindolin-2-yl)-6-methylbenzaldehyde **3a** (0.2 mmol) and ammonia in anhydrous methanol solution (0.6 mmol) were added to the glassware with 1 ml MeOH. The resulting mixture was heated at 70 ºC for 24 h monitored by TLC. The solvent was evaporated under reduced pressure,

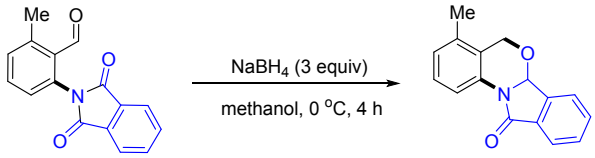
and the crude mixture was purified by silica gel column chromatography with a polarity of eluent of ether/EtOAc 1:1 to obtain the product **4** as a white solid (34.2 mg, 65%).

^{1}H NMR (500 MHz, DMSO) δ 9.74 (s, 1H), 7.98 (d, J = 7.4 Hz, 1H), 7.90 (dd, J = 8.4, 7.1 Hz, 1H), 7.83 (d, J = 8.6 Hz, 2H), 7.58 (tdd, J = 13.2, 9.4, 4.3 Hz, 4H), 7.21 (s, 1H), 2.80 (s, 3H).

^{13}C NMR (126 MHz, DMSO) δ 171.5, 162.4, 158.0, 150.6, 138.9, 138.1, 136.5, 134.8, 130.7, 129.6, 129.5, 128.6, 128.4, 126.4, 122.4, 17.7.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{14}N_3O$ 264.1132; Found 264.1128.

2.2.5 General Procedure to access polycyclic heterocyclic product **5**



3a **5**

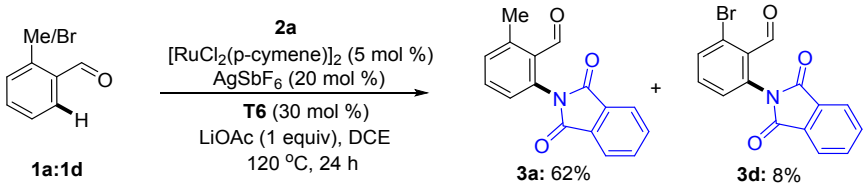
2-(1,3-dioxoisoindolin-2-yl)-6-methylbenzaldehyde **3a** (0.2 mmol) was added to the glassware with 2 ml MeOH. Then $NaBH_4$ (3 equiv) were added to the reaction mixture at 0 ºC for 4 h monitored by TLC. The solvent was evaporated under reduced pressure, and the crude mixture was purified by silica gel column chromatography with petroleum ether/EtOAc 10:1 to obtain the product **5** as a white solid (44.7 mg, 89%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (d, J = 8.2 Hz, 1H), 7.83 (d, J = 7.5 Hz, 1H), 7.60 (d, J = 7.3 Hz, 1H), 7.57 (tt, J = 8.0, 4.0 Hz, 1H), 7.51 (td, J = 7.3, 1.1 Hz, 1H), 7.23-7.16 (m, 1H), 6.90 (d, J = 7.5 Hz, 1H), 5.79 (s, 1H), 5.04 (d, J = 15.2 Hz, 1H), 4.98 (d, J = 15.2 Hz, 1H), 2.14 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.1, 140.1, 134.1, 133.7, 133.0, 132.6, 130.5, 127.6, 125.7, 124.1, 123.5, 121.4, 117.4, 84.5, 67.1, 18.3.

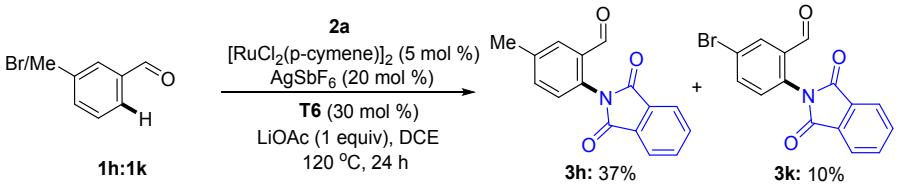
HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{14}NO_2$ 252.1019; Found 252.1017.

2.2.6 General Procedure to control experiments



1a:1d **3a:** 62% **3d:** 8%

The 2-methylbenzaldehyde (**1a**) (0.2 mmol), 2-bromobenzaldehyde (**1d**) (0.2 mmol) 1,3-dioxoisoindolin-2-yl 4-methylbenzenesulfonate (**2a**) (0.4 mmol, 126.9 mg), $[RuCl_2(p\text{-cymene})]_2$ (0.01 mmol, 6.1 mg), $AgSbF_6$ (0.04 mmol, 13.7mg), **T6** (0.06 mmol, 10.5 mg) and LiOAc (0.2 mmol, 13.2 mg) were taken in a sealed tube with magnetic stir bar. Then DCE (2.0 mL) was added with a syringe and the resulting mixture was heated at 120 ºC for 24 h. Upon completion, the reaction mixture was cooled to room temperature and filtered through a sand funnel. Next the reaction mixture was quenched by sat. NH_4Cl (aq) (40 mL), and extracted with CH_2Cl_2 (20 mL) for three times. The combined organic layers were washed with water (60 mL), dried over anhydrous Na_2SO_4, and concentrated in vacuo. The crude residue was purified by column chromatography on silica gel using petroleum ether/EtOAc 10:1 as eluent to obtain the product **3a** (32 mg, 62%) and **3d** (6 mg, 8%).



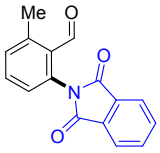
The 3-methylbenzaldehyde (**1h**) (0.2 mmol), 3-bromobenzaldehyde (**1k**) (0.2 mmol) 1,3-dioxoisoindolin-2-yl 4-methylbenzenesulfonate (**2a**) (0.4 mmol, 126.9 mg), $[RuCl_2(p\text{-cymene})]_2$ (0.01 mmol, 6.1 mg), $AgSbF_6$ (0.04 mmol, 13.7mg), **T6** (0.06 mmol, 10.5 mg) and LiOAc (0.2 mmol, 13.2 mg) were taken in a sealed tube with magnetic stir bar. Then DCE (2.0 mL) was added with a syringe and the resulting mixture was heated at 120 ºC for 24 h. Upon completion, the reaction mixture was cooled to room temperature and filtered through a sand funnel. Next the reaction mixture was quenched by sat. NH_4Cl (aq) (40 mL), and extracted with CH_2Cl_2 (20 mL) for three times. The combined organic layers were washed with water (60 mL), dried over anhydrous Na_2SO_4, and concentrated in vacuo. The crude residue was purified by column chromatography on silica gel using petroleum ether/EtOAc 10:1 as eluent to obtain the product **3h** (19 mg, 37%) and **3k** (7.5 mg, 8%).

2.2.7 The preparation of crystal **3u** (CCDC 2055086)

The pure **3u** compound (30mg) is placed in a slender glass tube, and chloroform is added to dissolve it, and slowly volatilize to obtain a single crystal.

The diffraction data of crystals were collected on a Rigaku XtaLAB Synergy CCD diffractometer with graphite monochromated Cu-Ka radiation (λ = 1.54056 Å) at 293 K. Absorption corrections were applied by SADABS. All the structures were solved by direct methods and refined by full-matrix least-squares method on Olex2 using SHELXTL-2014. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms of the ligand were generated geometrically.

2.3 Characterization Data of Products 3



3a

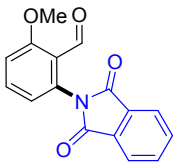
2-(1,3-dioxoisoindolin-2-yl)-6-methylbenzaldehyde

white solid, yield: 81% (43 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.98 – 7.93 (m, 2H), 7.82 – 7.78 (m, 2H), 7.60 (t, J = 7.8 Hz, 1H), 7.39 (d, J = 7.7 Hz, 1H), 7.22 (d, J = 7.8 Hz, 1H), 2.71 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 190.1, 167.5, 142.5, 134.5, 133.8, 132.9, 132.3, 132.0, 130.6, 128.0, 123.9, 19.8.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{12}NO_3$ 266.0802; Found 266.0807.



3b

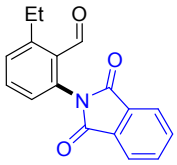
2-(1,3-dioxoisoindolin-2-yl)-6-methoxybenzaldehyde

white solid, yield: 74% (42 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.45 (s, 1H), 7.92 (dt, *J* = 5.7, 2.8 Hz, 2H), 7.78 – 7.75 (m, 2H), 7.65 (t, *J* = 8.2 Hz, 1H), 7.14 (d, *J* = 8.6 Hz, 1H), 6.95 (d, *J* = 7.8 Hz, 1H), 3.96 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 188.9, 167.4, 163.3, 135.4, 134.2, 132.3, 131.2, 123.8, 122.8, 121.2, 113.0, 56.3.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{12}NO_4$ 282.0761; Found 282.0758.



3c

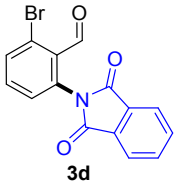
2-(1,3-dioxoisoindolin-2-yl)-6-ethylbenzaldehyde

white solid, yield: 55% (31 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.30 (s, 1H), 7.96 – 7.92 (m, 2H), 7.81 – 7.77 (m, 2H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 7.7 Hz, 1H), 7.22 (d, *J* = 7.7 Hz, 1H), 3.06 (q, *J* = 7.5 Hz, 2H), 1.31 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 190.2, 167.6, 148.7, 134.5, 134.0, 132.0, 131.4, 130.2, 128.1, 123.90, 25.9, 16.5.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{17}H_{14}NO_3$ 280.0968; Found 280.0966.



3d

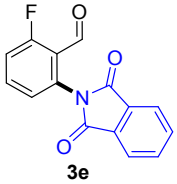
2-bromo-6-(1,3-dioxoisoindolin-2-yl)benzaldehyde

white solid, yield: 62% (41 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.33 (s, 1H), 7.95 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.81 (dd, *J* = 3.6, 1.8 Hz, 2H), 7.80 (s, 1H), 7.56 (t, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 191.1, 167.1, 134.9, 134.5, 132.0, 130.4, 130.2, 128.1, 124.0.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_9BrNO_3$ 329.9761; Found 329.9764.



3e

2-(1,3-dioxoisoindolin-2-yl)-6-fluorobenzaldehyde

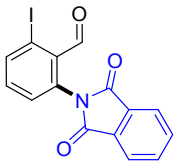
white solid, yield: 89% (48 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.34 (s, 1H), 7.97 – 7.94 (m, 2H), 7.83 – 7.79 (m, 2H), 7.73 (td, *J*

= 8.2, 6.0 Hz, 1H), 7.38 – 7.31 (m, 1H), 7.23 (d, *J* = 7.9 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 185.8 (d, *J* = 10.0 Hz), 167.0, 165.8 (d, *J* = 259.7 Hz), 135.7 (d, *J* = 10.8 Hz), 134.5, 132.1, 131.4 (d, *J* = 3.5 Hz), 126.7 (d, *J* = 3.6 Hz), 124.0, 120.9 (d, *J* = 8.2 Hz), 117.6 (d, *J* = 21.5 Hz).

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_9FNO_3$ 270.0561; Found 270.0562



3f

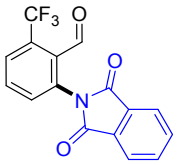
2-(1,3-dioxoisoindolin-2-yl)-6-iodobenzaldehyde

white solid, yield: 65% (49 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.00 (s, 1H), 8.09 (d, *J* = 7.6 Hz, 1H), 7.97 – 7.91 (m, 2H), 7.84 – 7.76 (m, 2H), 7.40 (d, *J* = 7.2 Hz, 1H), 7.36 (t, *J* = 7.8 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 194.8, 167.1, 141.6, 134.6, 131.9, 131.6, 131.2, 124.0, 101.4.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_9INO_3$ 377.9622; Found 377.9619.



3g

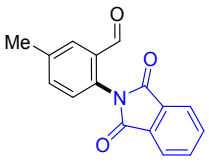
2-(1,3-dioxoisoindolin-2-yl)-6-(trifluoromethyl)benzaldehyde

white solid, yield: 47% (30 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.36-10.33 (m, 1H), 7.97-7.93 (m, 2H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.83-7.81 (m, 2H), 7.80 (d, *J* = 4.7 Hz, 1H), 7.64 (d, *J* = 7.9 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 188.6, 166.9, 134.7, 133.9, 133.0, 131.9, 131.8, 131.8 (q, *J* = 32.7 Hz), 131.1, 126.7 (q, *J* = 5.6 Hz), 124.1, 123.3 (q, *J* = 275.1 Hz).

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_9F_3NO_3$ 320.0529; Found 320.0529.



3h

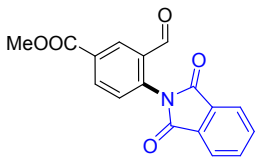
2-(1,3-dioxoisoindolin-2-yl)-5-methylbenzaldehyde

white solid, yield: 65% (34 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.97 (d, *J* = 3.1 Hz, 1H), 7.96 (d, *J* = 3.1 Hz, 1H), 7.81 (d, *J* = 3.0 Hz, 1H), 7.80 (s, 1H), 7.80 (s, 1H), 7.57 – 7.55 (m, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 2.50 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 189.5, 167.5, 140.0, 135.3, 134.5, 133.2, 132.0, 129.9, 129.1, 124.0, 21.1.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{12}NO_3$ 266.0802; Found 266.0807.



3i

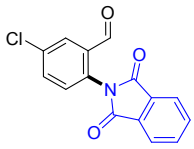
methyl 4-(1,3-dioxoisoindolin-2-yl)-3-formylbenzoate

white solid, yield: 71% (44 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.97 (s, 1H), 8.65 (d, *J* = 1.7 Hz, 1H), 8.39 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.97 (dt, *J* = 6.9, 3.5 Hz, 2H), 7.86 – 7.81 (m, 2H), 7.54 (d, *J* = 8.2 Hz, 1H), 3.99 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 188.5, 166.8, 165.2, 135.2, 134.8, 133.7, 132.2, 131.8, 131.3, 130.1, 124.2, 52.8.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{17}H_{12}NO_5$ 310.0710; Found 310.0712.



3j

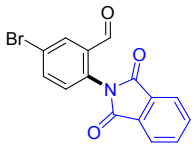
5-chloro-2-(1,3-dioxoisoindolin-2-yl)benzaldehyde

white solid, yield: 40% (25 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.88 (s, 1H), 8.00 – 7.98 (m, 2H), 7.97 (d, *J* = 3.9 Hz, 1H), 7.87 – 7.81 (m, 2H), 7.72 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 187.7, 167.1, 135.9, 134.8, 134.5, 133.4, 131.8, 131.1, 130.3, 124.2.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_9ClNO_3$ 286.0266; Found 286.0264.



3k

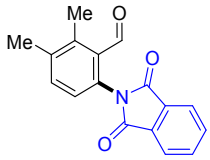
5-bromo-2-(1,3-dioxoisoindolin-2-yl)benzaldehyde

white solid, yield: 55% (36 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.87 (d, *J* = 0.5 Hz, 1H), 8.14 – 8.12 (m, 1H), 7.99 – 7.98 (m, 1H), 7.97 – 7.96 (m, 1H), 7.88 – 7.85 (m, 1H), 7.85-7.83 (m, 1H), 7.83 – 7.82 (m, 1H), 7.31 (d, *J* = 8.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 187.6, 167.0, 137.5, 134.8, 133.5, 131.7, 131.3, 130.8, 124.2, 123.6.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_9BrNO_3$ 329.9761; Found 329.9764.



3l

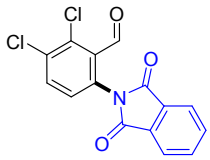
6-(1,3-dioxoisoindolin-2-yl)-2,3-dimethylbenzaldehyde

white solid, yield: 54% (30 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.35 (s, 1H), 7.96 – 7.92 (m, 2H), 7.81 – 7.77 (m, 2H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 2.56 (s, 3H), 2.39 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 191.2, 167.7, 140.5, 139.8, 135.1, 134.4, 132.0, 131.1, 129.6, 127.3, 123.9, 20.3, 15.1.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{17}H_{14}NO_3$ 280.0968; Found 280.0966.



3m

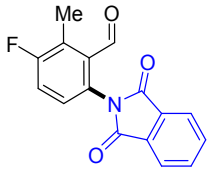
2,3-dichloro-6-(1,3-dioxoisoindolin-2-yl)benzaldehyde

white solid, yield: 45% (29 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.41 (s, 1H), 7.96 – 7.92 (m, 2H), 7.83 – 7.80 (m, 2H), 7.79 (d, *J* = 3.8 Hz, 1H), 7.30 (d, *J* = 8.5 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 188.7, 166.8, 136.9, 135.3, 134.6, 131.9, 131.2, 129.9, 129.6, 124.1.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_8Cl_2NO_3$ 319.9876; Found 319.9873.



3n

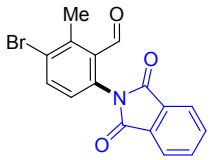
6-(1,3-dioxoisoindolin-2-yl)-3-fluoro-2-methylbenzaldehyde

white solid, yield: 48% (27 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.23 (s, 1H), 7.96 (td, *J* = 5.3, 2.2 Hz, 2H), 7.84 – 7.80 (m, 2H), 7.38 (t, *J* = 8.7 Hz, 1H), 7.21 (dd, *J* = 8.6, 4.7 Hz, 1H), 2.59 (d, *J* = 2.1 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 189.4, 167.5, 164.6 (d, *J* = 257.5 Hz), 145.5 (d, *J* = 9.8 Hz), 134.8 (d, *J* = 11.7 Hz), 134.7, 131.8, 127.3 (d, *J* = 3.4 Hz), 124.1, 119.6 (d, *J* = 20.8 Hz), 115.7 (d, *J* = 23.4 Hz), 20.2.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}FNO_3$ 284.0718; Found 284.0713.



3o

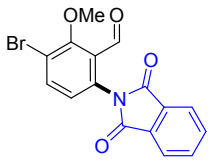
3-bromo-6-(1,3-dioxoisoindolin-2-yl)-2-methylbenzaldehyde

white solid, yield: 47% (32 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.25 (s, 1H), 7.97 – 7.92 (m, 2H), 7.89 (d, *J* = 8.4 Hz, 1H), 7.82 – 7.78 (m, 2H), 7.12 (d, *J* = 8.4 Hz, 1H), 2.73 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 190.0, 167.2, 141.0, 137.5, 134.6, 132.9, 131.8, 131.0, 128.7, 128.1, 124.1, 19.0.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}BrNO_3$ 343.9917; Found 343.9913.



3p

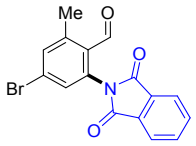
3-bromo-6-(1,3-dioxoisoindolin-2-yl)-2-methoxybenzaldehyde

white solid, yield: 49% (35 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.32 (d, *J* = 0.5 Hz, 1H), 7.96 – 7.93 (m, 2H), 7.93 – 7.90 (m, 1H), 7.80 – 7.77 (m, 2H), 7.08 (dd, *J* = 8.4, 0.5 Hz, 1H), 4.02 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 188.4, 167.0, 161.3, 138.9, 134.5, 132.0, 130.3, 127.5, 124.0, 119.4, 63.7.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}BrNO_4$ 359.9866; Found 359.9862.



3q

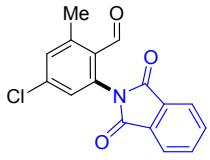
4-bromo-2-(1,3-dioxoisoindolin-2-yl)-6-methylbenzaldehyde

white solid, yield: 60% (41 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.20 (s, 1H), 7.97 – 7.93 (m, 2H), 7.82 (td, *J* = 5.2, 2.1 Hz, 2H), 7.56 (d, *J* = 1.1 Hz, 1H), 7.40 (d, *J* = 1.7 Hz, 1H), 2.68 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 189.2, 167.1, 143.8, 135.7, 134.7, 133.4, 131.8, 131.0, 129.4, 127.9, 124.1, 19.7.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}BrNO_3$ 343.9917; Found 343.9913.



3r

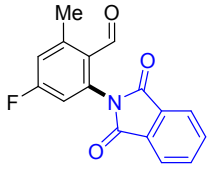
4-chloro-2-(1,3-dioxoisoindolin-2-yl)-6-methylbenzaldehyde

white solid, yield: 53% (32 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.20 (s, 1H), 7.99 – 7.94 (m, 2H), 7.85 – 7.79 (m, 2H), 7.39 (d, *J* = 1.4 Hz, 1H), 7.25 (d, *J* = 1.9 Hz, 1H), 2.68 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 189.0, 167.1, 143.8, 139.5, 134.7, 133.6, 132.7, 131.8, 129.0, 128.2, 124.1, 19.9.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}ClNO_3$ 300.0422; Found 300.0419.

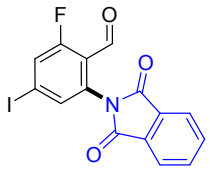


3s

2-(1,3-dioxoisoindolin-2-yl)-4-fluoro-6-methylbenzaldehyde

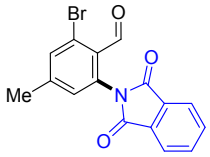
white solid, yield: 66% (37 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.18 (s, 1H), 7.98 – 7.94 (m, 2H), 7.84 – 7.80 (m, 2H), 7.10 (dd, *J* = 9.0, 2.3 Hz, 1H), 6.97 (dd, *J* = 8.4, 2.5 Hz, 1H), 2.71 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 188.6, 167.1, 164.6 (d, *J* = 257.5 Hz), 145.5 (d, *J* = 9.8 Hz), 134.8 (d, *J* = 11.7 Hz), 134.7, 131.8, 127.3 (d, *J* = 3.4 Hz), 124.1, 119.6 (d, *J* = 20.8 Hz), 115.7 (d, *J* = 23.4 Hz), 20.2.
HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}FNO_3$ 284.0718; Found 284.0713.



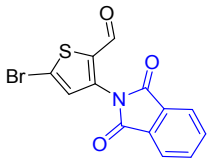
3t

2-(1,3-dioxoisoindolin-2-yl)-6-fluoro-4-iodobenzaldehyde
white solid, yield: 50% (40 mg).
^{1}H NMR (500 MHz, $CDCl_3$) δ 10.26 (s, 1H), 7.95 (dt, *J* = 7.3, 3.7 Hz, 2H), 7.83 – 7.79 (m, 2H), 7.74 (dd, *J* = 9.4, 1.4 Hz, 1H), 7.60 (s, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 185.2, 166.6, 164.5 (d, *J* = 264.4 Hz), 135.9 (d, *J* = 3.7 Hz), 134.7, 131.9, 131.7 (d, *J* = 3.9 Hz), 127.0 (d, *J* = 23.9 Hz), 124.1, 120.4 (d, *J* = 8.3 Hz), 100.8 (d, *J* = 10.5 Hz).
HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{15}H_8FINO_3$ 395.9528; Found 395.9527.



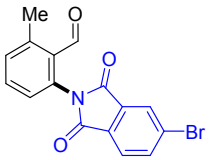
3u

2-bromo-6-(1,3-dioxoisoindolin-2-yl)-4-methylbenzaldehyde
white solid, yield: 72% (49 mg).
^{1}H NMR (500 MHz, $CDCl_3$) δ 10.28 (s, 1H), 7.96 – 7.94 (m, 1H), 7.93 (t, *J* = 3.4 Hz, 1H), 7.80 (dd, *J* = 6.0, 2.1 Hz, 1H), 7.79 (d, *J* = 3.1 Hz, 1H), 7.62 (s, 1H), 7.16 (s, 1H), 2.45 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 190.8, 167.2, 146.4, 135.5, 134.5, 132.1, 131.9, 131.2, 128.7, 127.5, 123.9, 21.3.
HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}BrNO_3$ 343.9917; Found 343.9913.



3v

5-bromo-3-(1,3-dioxoisoindolin-2-yl)thiophene-2-carbaldehyde
white solid, yield: 61% (41 mg).
^{1}H NMR (500 MHz, $CDCl_3$) δ 9.65 (s, 1H), 8.03 – 7.99 (m, 2H), 7.89 – 7.85 (m, 2H), 7.22 (s, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 180.4, 166.1, 137.2, 135.2, 133.3, 131.3, 129.7, 124.5, 123.2.
HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{13}H_7BrNO_3S$ 335.9325; Found 335.9322.



3w

2-(5-bromo-1,3-dioxoisoindolin-2-yl)-6-methylbenzaldehyde

white solid, yield: 59% (40 mg).

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.31 (s, 1H), 8.10 (d, *J* = 1.4 Hz, 1H), 7.94 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.82 (d, *J* = 7.9 Hz, 1H), 7.61 (t, *J* = 7.8 Hz, 1H), 7.41 (d, *J* = 7.7 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 1H), 2.72 (s, 3H).

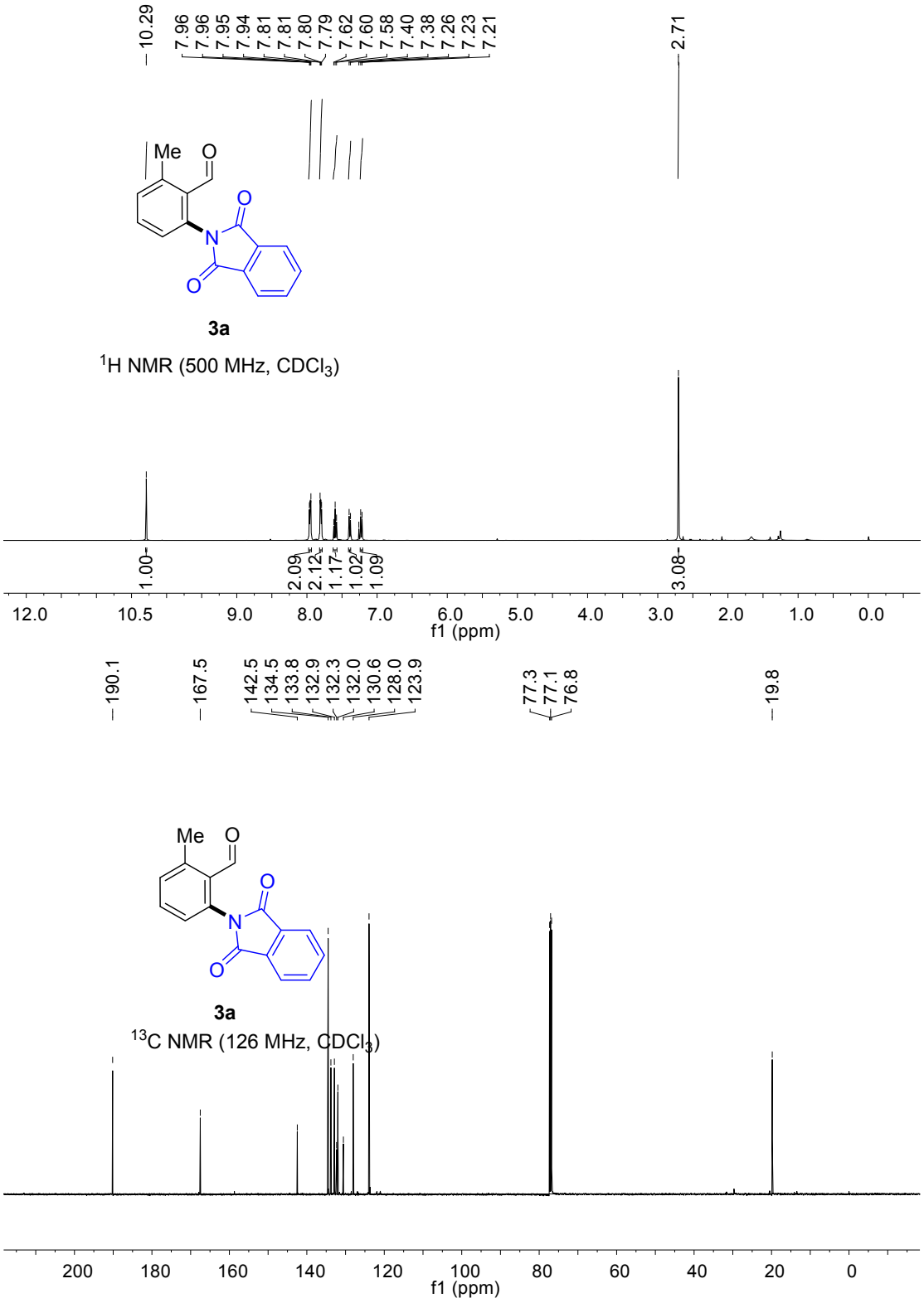
^{13}C NMR (126 MHz, $CDCl_3$) δ 190.1, 166.7, 166.2, 142.8, 137.5, 133.9, 133.7, 133.1, 131.5, 130.6, 130.3, 129.4, 128.2, 127.3, 125.3, 19.6.

HRMS (ESI) *m/z*: $[M + H]^+$ Calcd for $C_{16}H_{11}BrNO_3$ 343.9917; Found 343.9913.

3.References

1. Yadav, M. R.; Shankar, M.; Ramesh, E.; Ghosh, K.; Sahoo, A. K. *Org. Lett* **2015**, 17, 1886-9.
2. Olga K.; Iryna H.; Katerina N.; Yurii L.; Mykhailo K.; Alexander S.; Iosip O. J. Org. Chem. **2020**, 85, 7112-7124.

4. ^{1}H & ^{13}C NMR Spectra

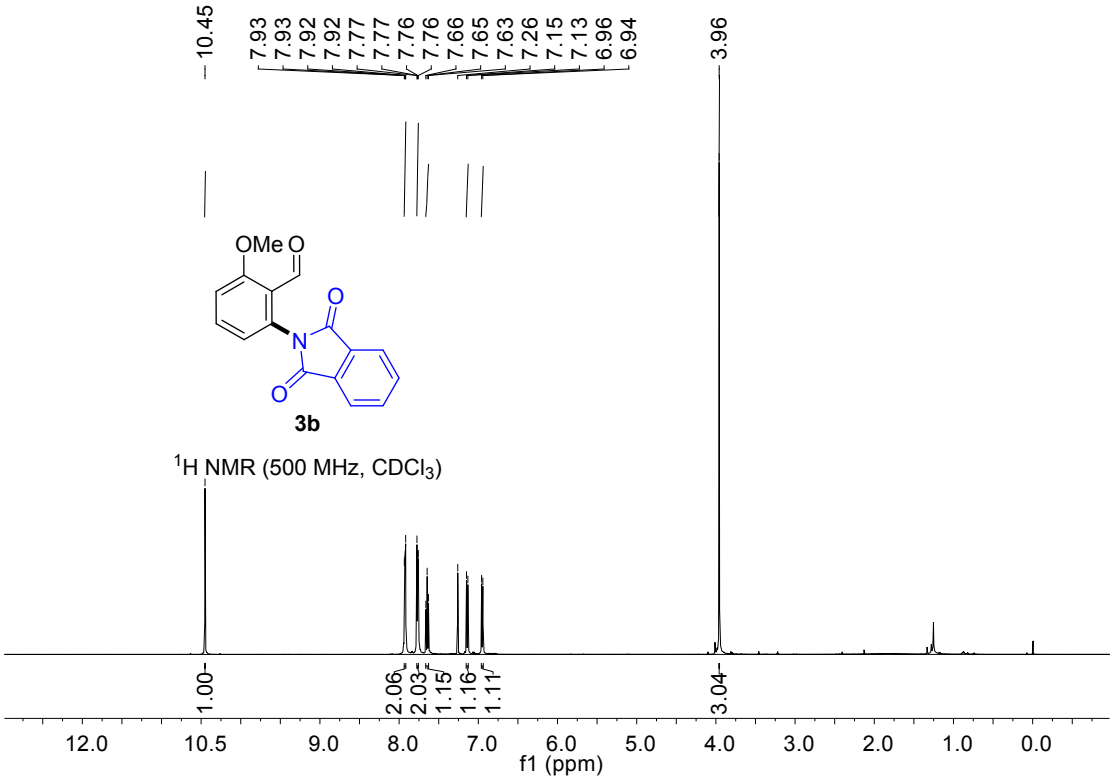


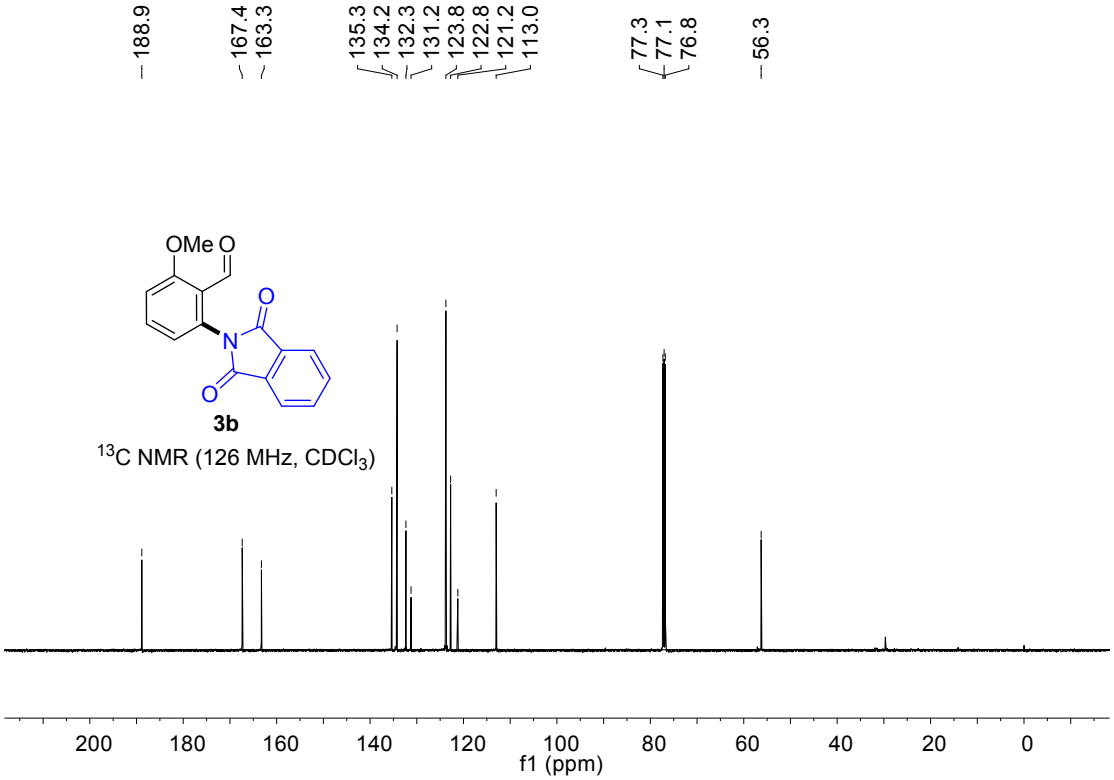
3a

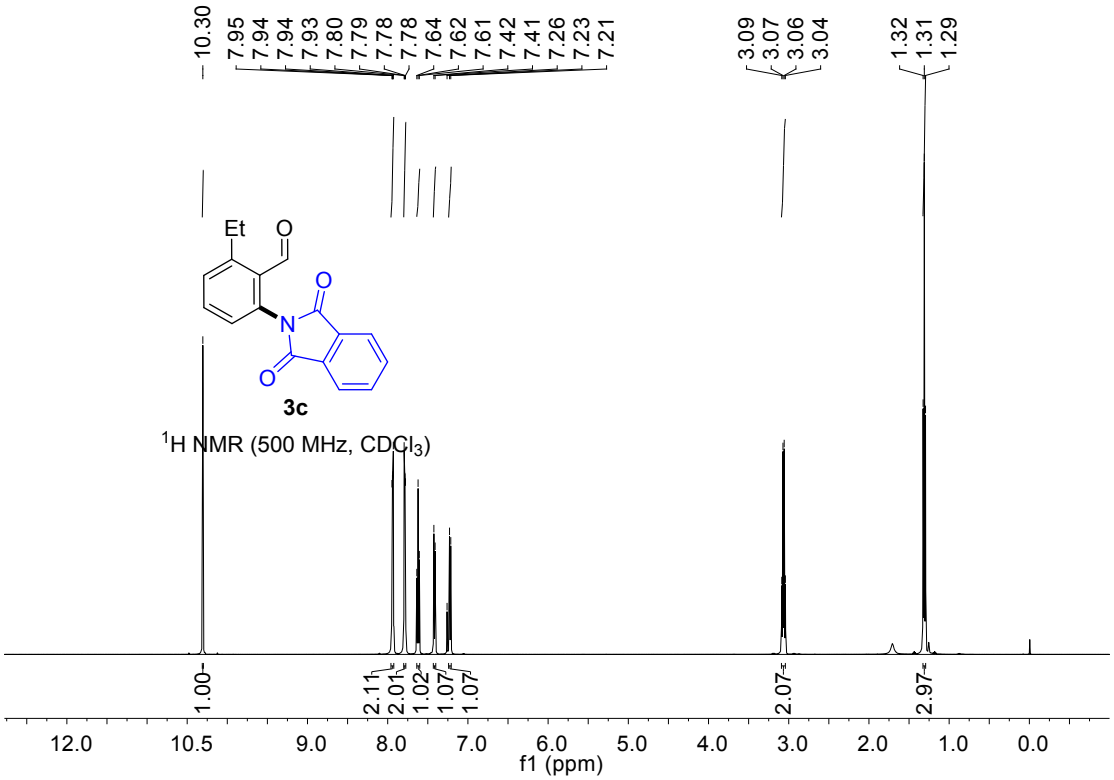
^{1}H NMR (500 MHz, $CDCl_3$)

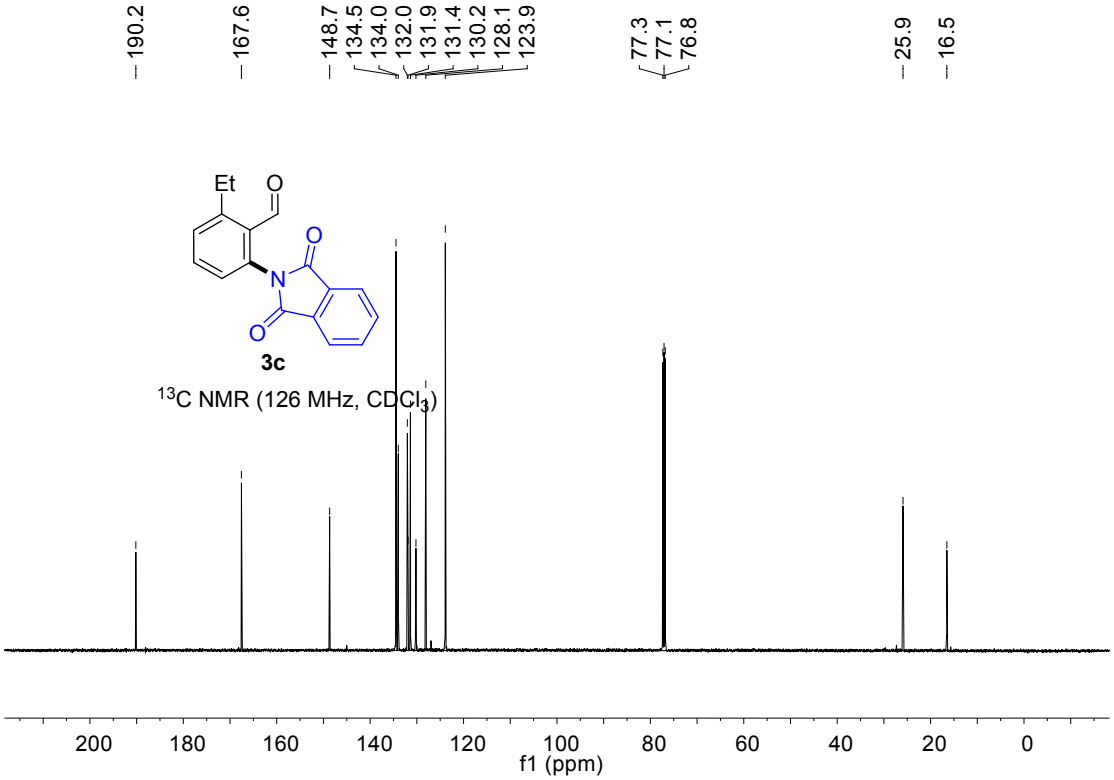
3a

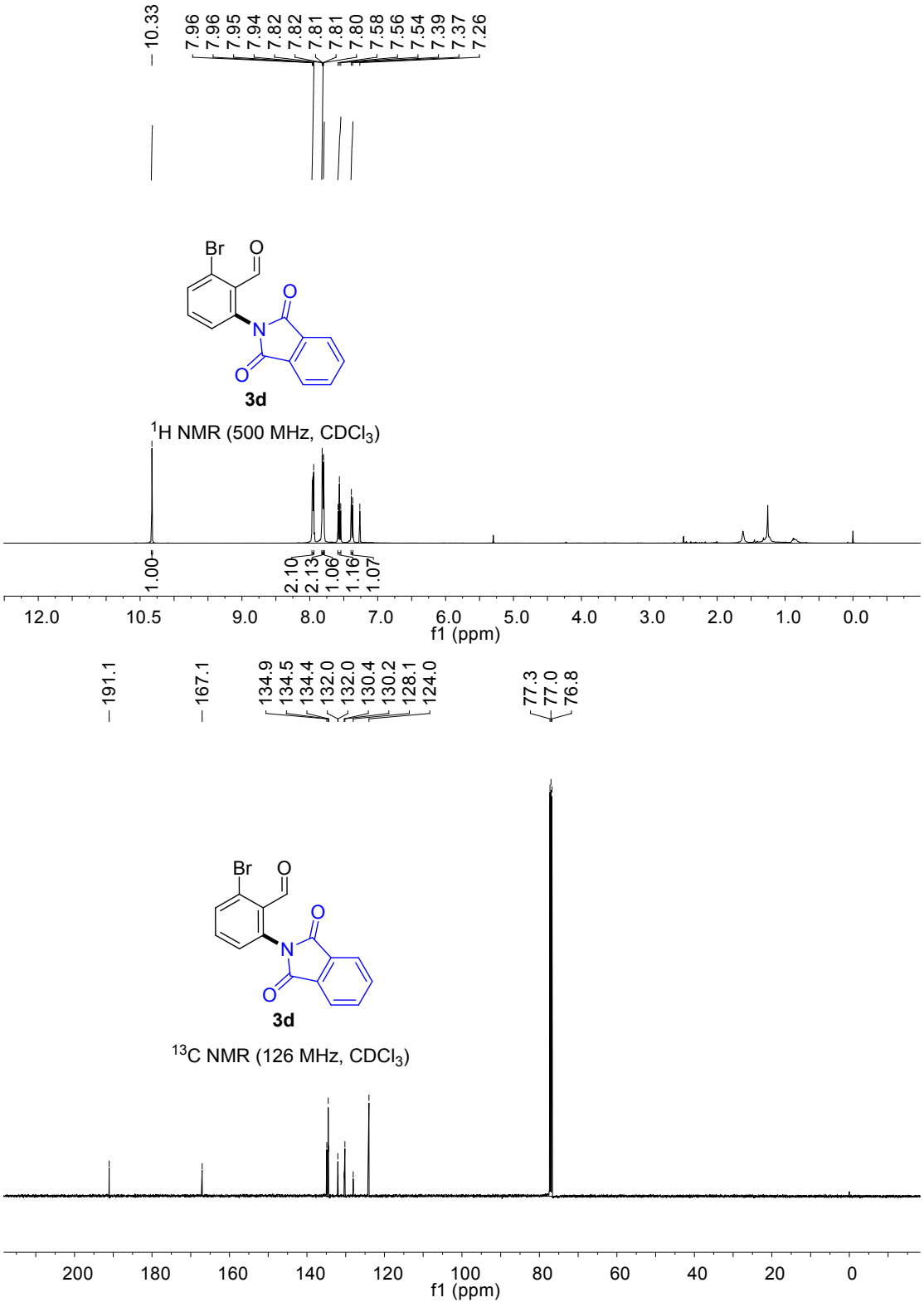
^{13}C NMR (126 MHz, $CDCl_3$)

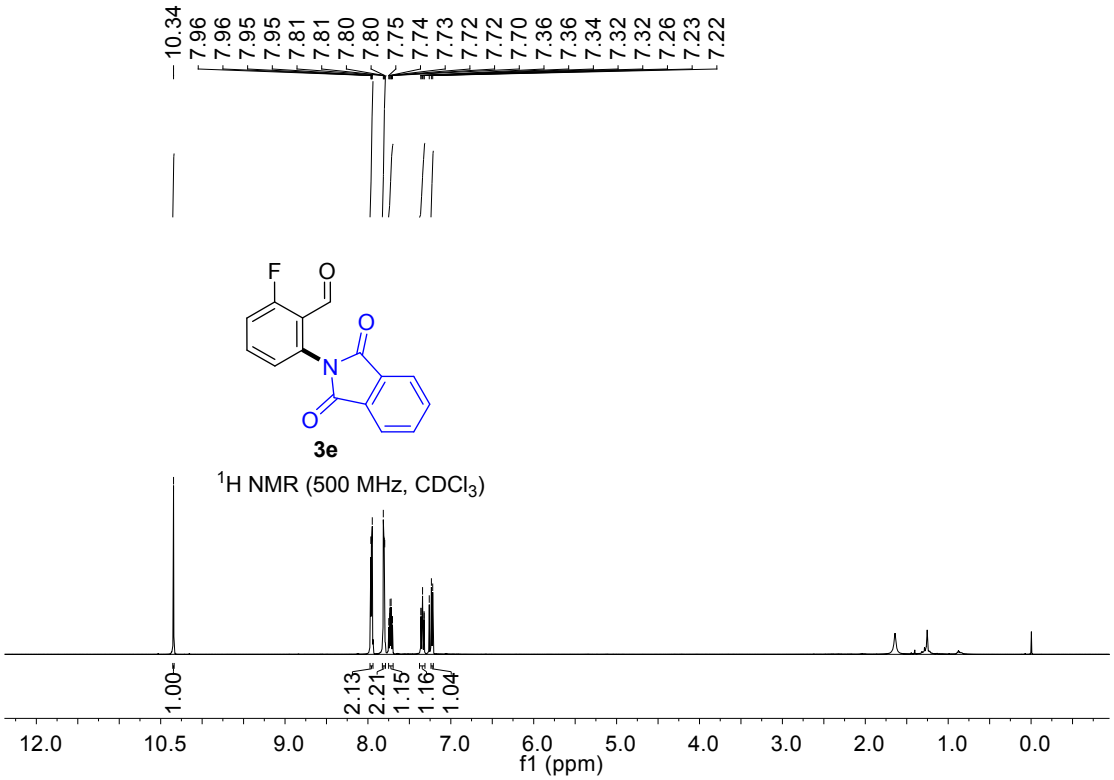

10.45
7.93
7.93
7.92
7.92
7.77
7.77
7.76
7.76
7.66
7.65
7.63
7.26
7.15
7.13
6.96
6.94
3.96
OMe O
O
N
O
3b
1H NMR (500 MHz, CDCl3)
1.00
2.06
2.03
1.15
1.16
1.11
3.04
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


188.9
167.4
163.3
135.3
134.2
132.3
131.2
123.8
122.8
121.2
113.0
77.3
77.1
76.8
56.3
OMe O
O
N
O
3b
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


10.30
7.95
7.94
7.94
7.93
7.80
7.79
7.78
7.78
7.64
7.62
7.61
7.42
7.41
7.26
7.23
7.21
3.09
3.07
3.06
3.04
1.32
1.31
1.29
Et
O
O
N
O
3c
1H NMR (500 MHz, CDCl3)
1.00
2.11
2.01
1.02
1.07
1.07
2.07
2.97
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

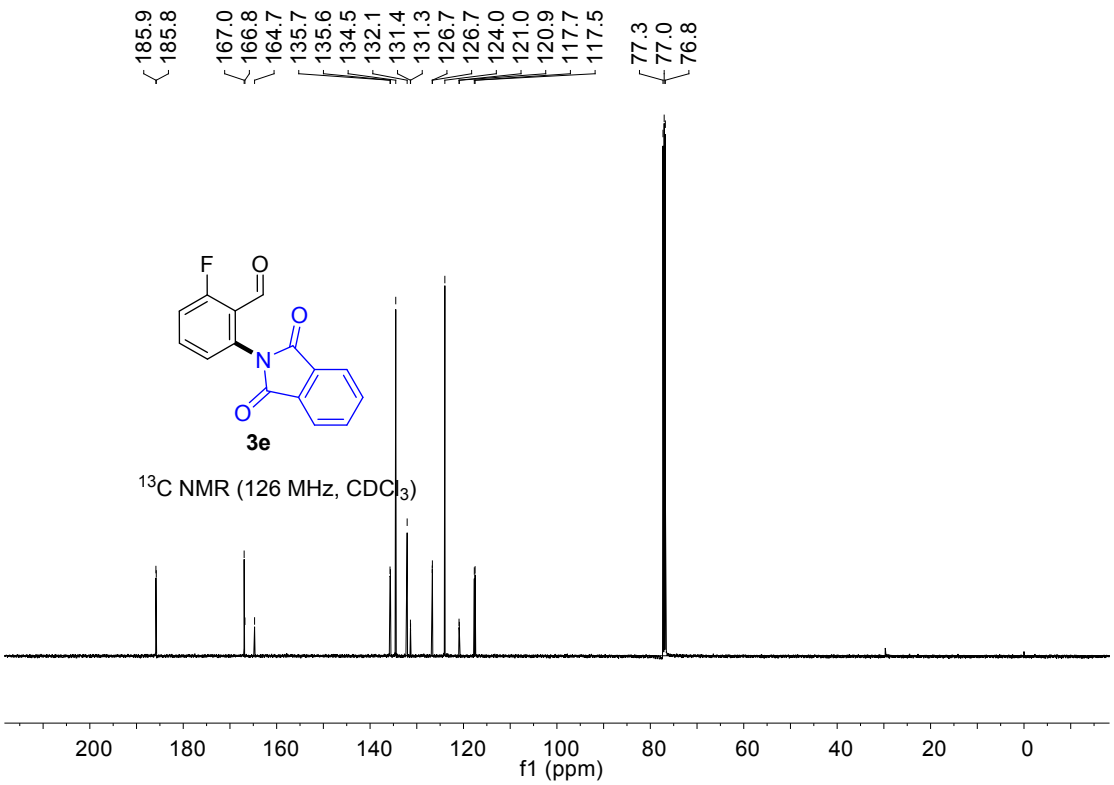

190.2
167.6
148.7
134.5
134.0
132.0
131.9
131.4
130.2
128.1
123.9
77.3
77.1
76.8
25.9
16.5
Et
O
O
N
O
3c
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


10.33
7.96
7.96
7.95
7.94
7.82
7.82
7.81
7.81
7.80
7.58
7.56
7.54
7.39
7.37
7.26
Br
O
O
N
O
3d
1H NMR (500 MHz, CDCl3)
1.00
2.10
2.13
1.06
1.16
1.07
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)
191.1
167.1
134.9
134.5
134.4
132.0
132.0
130.4
130.2
128.1
124.0
77.3
77.0
76.8
Br
O
O
N
O
3d
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



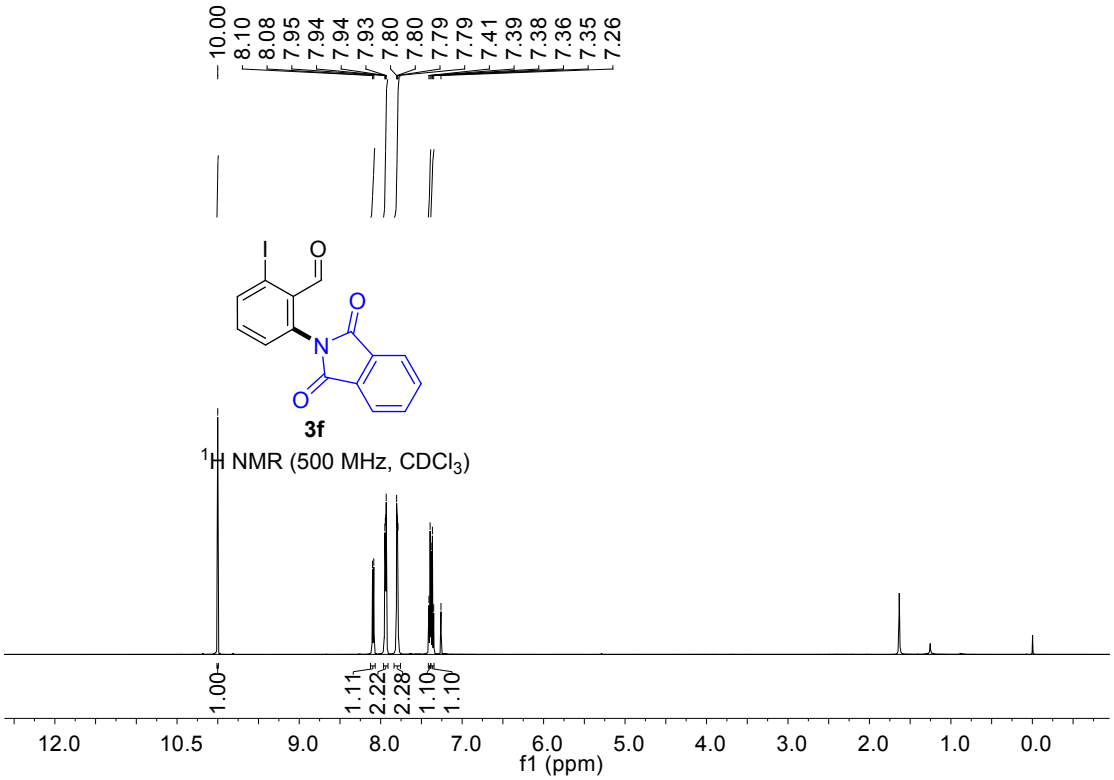
3e

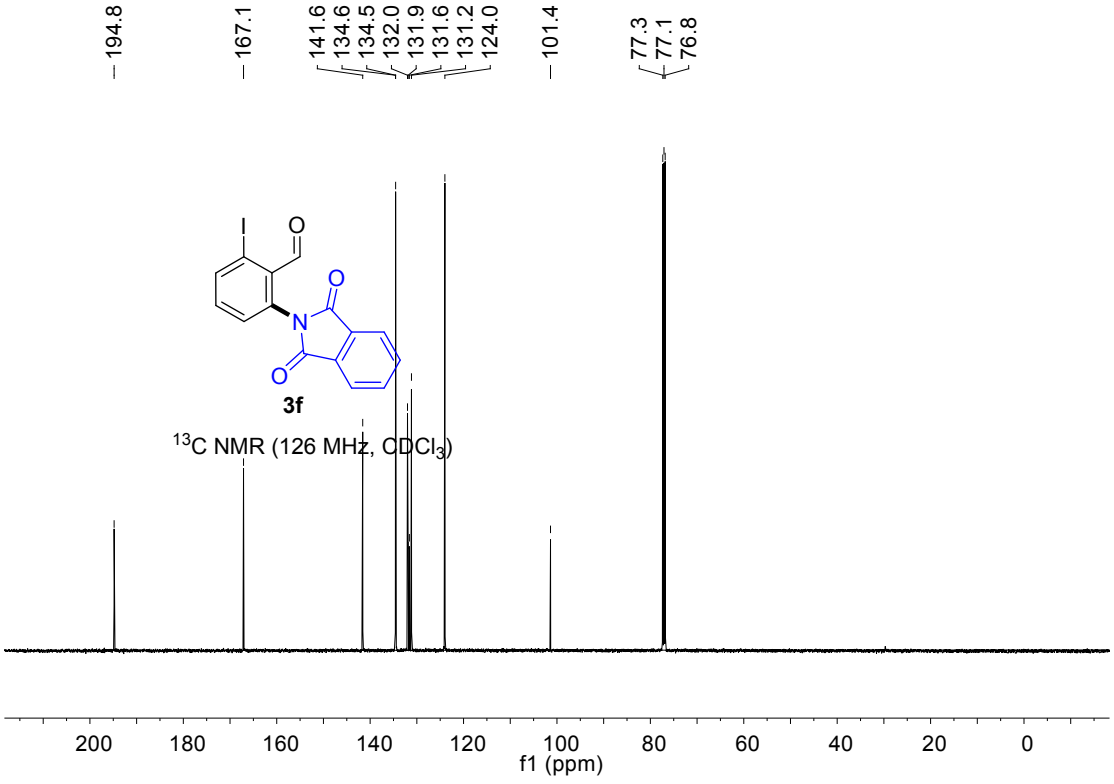
^{1}H NMR (500 MHz, $CDCl_3$)

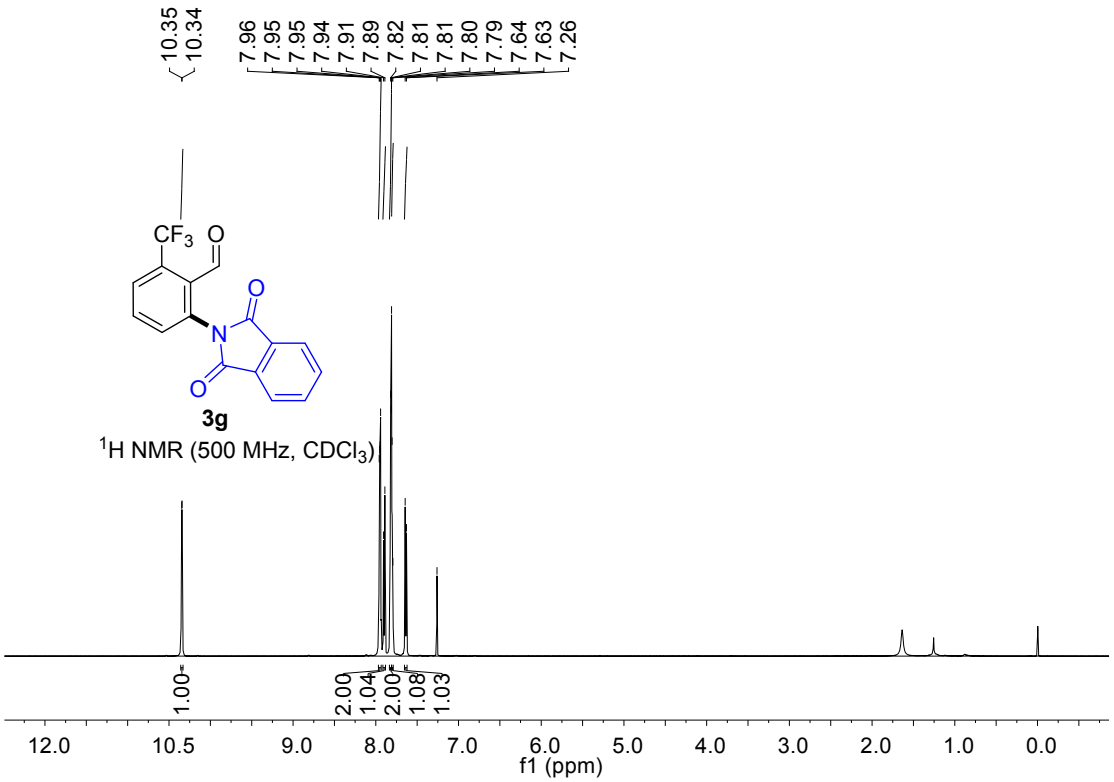


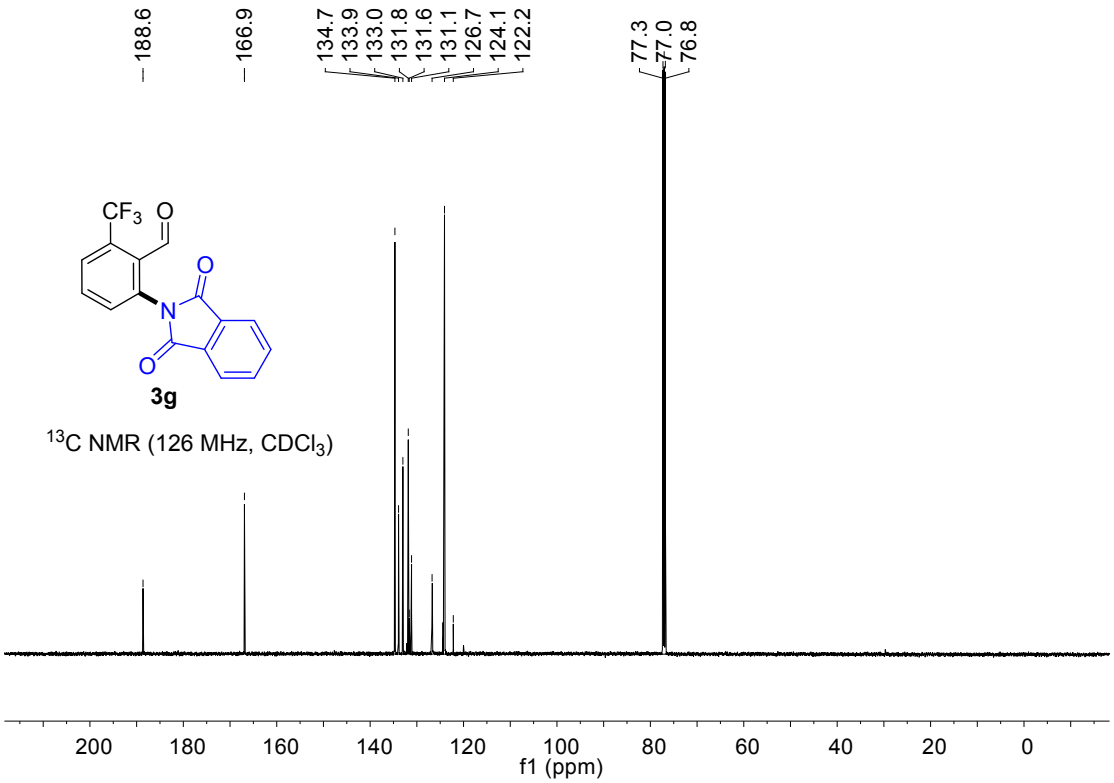
3e

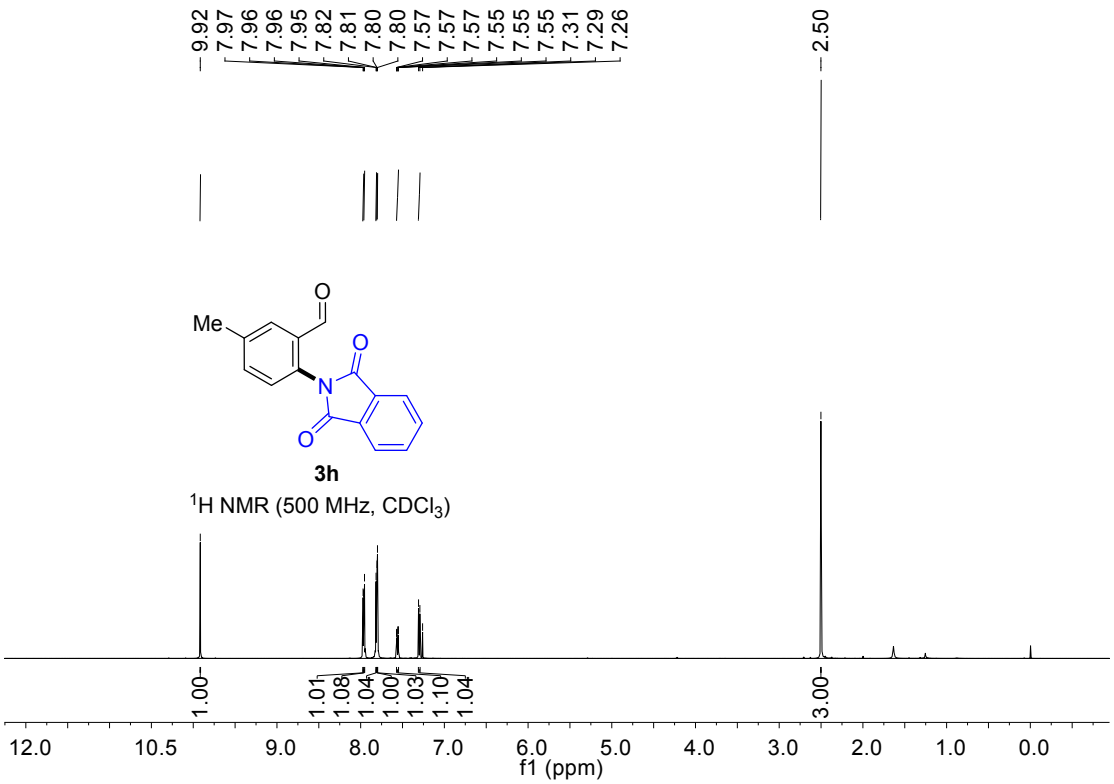
^{13}C NMR (126 MHz, $CDCl_3$)


10.00
8.10
8.08
7.95
7.94
7.94
7.93
7.80
7.80
7.79
7.79
7.41
7.39
7.38
7.36
7.35
7.26
I
O
O
N
O
3f
1H NMR (500 MHz, CDCl3)
1.00
1.11
2.22
2.28
1.10
1.10
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


194.8
167.1
141.6
134.6
134.5
132.0
131.9
131.6
131.2
124.0
101.4
77.3
77.1
76.8
I
O
O
N
O
3f
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


10.35
10.34
7.96
7.95
7.95
7.94
7.91
7.89
7.82
7.81
7.81
7.80
7.79
7.64
7.63
7.26
CF3 O
O
N
O
3g
1H NMR (500 MHz, CDCl3)
1.00
2.00
1.04
2.00
1.08
1.03
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


188.6
166.9
134.7
133.9
133.0
131.8
131.6
131.1
126.7
124.1
122.2
77.3
77.0
76.8
CF3 O
O
N
O
3g
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


9.92
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7.96
7.95
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7.81
7.80
7.80
7.57
7.57
7.55
7.55
7.55
7.31
7.29
7.26
2.50
Me
O
N
O
O
3h
1H NMR (500 MHz, CDCl3)
1.00
1.01
1.08
1.04
1.00
1.03
1.10
1.04
3.00
12.0
10.5
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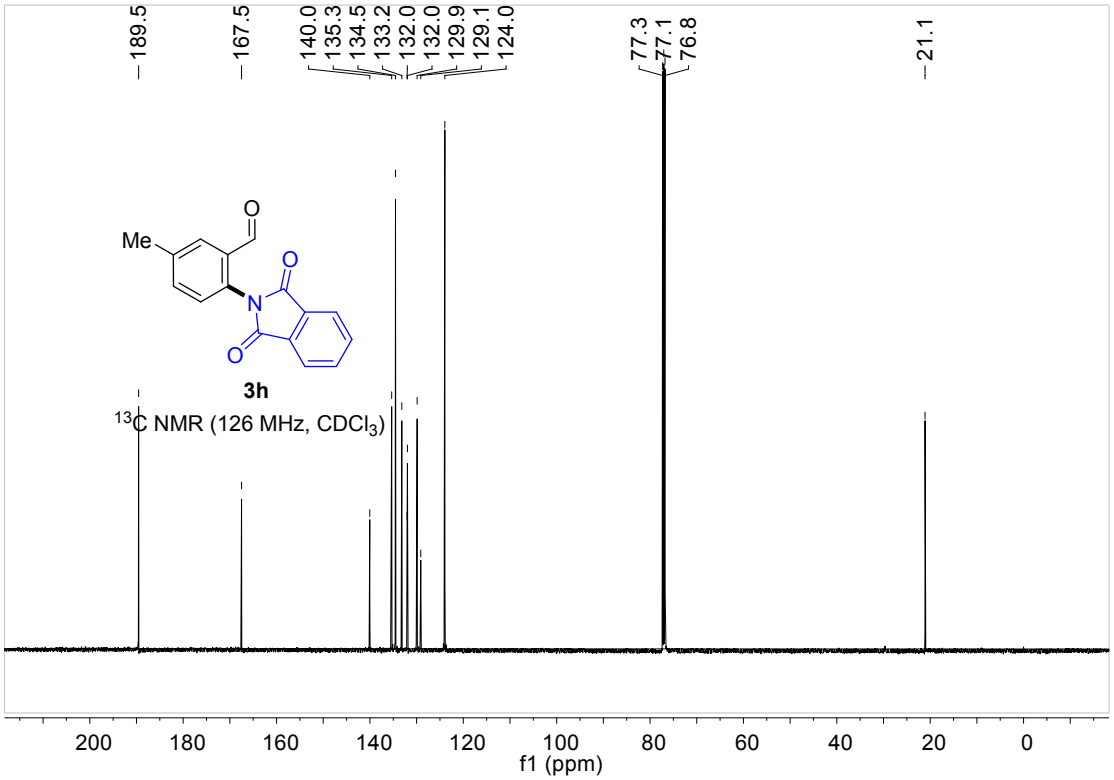
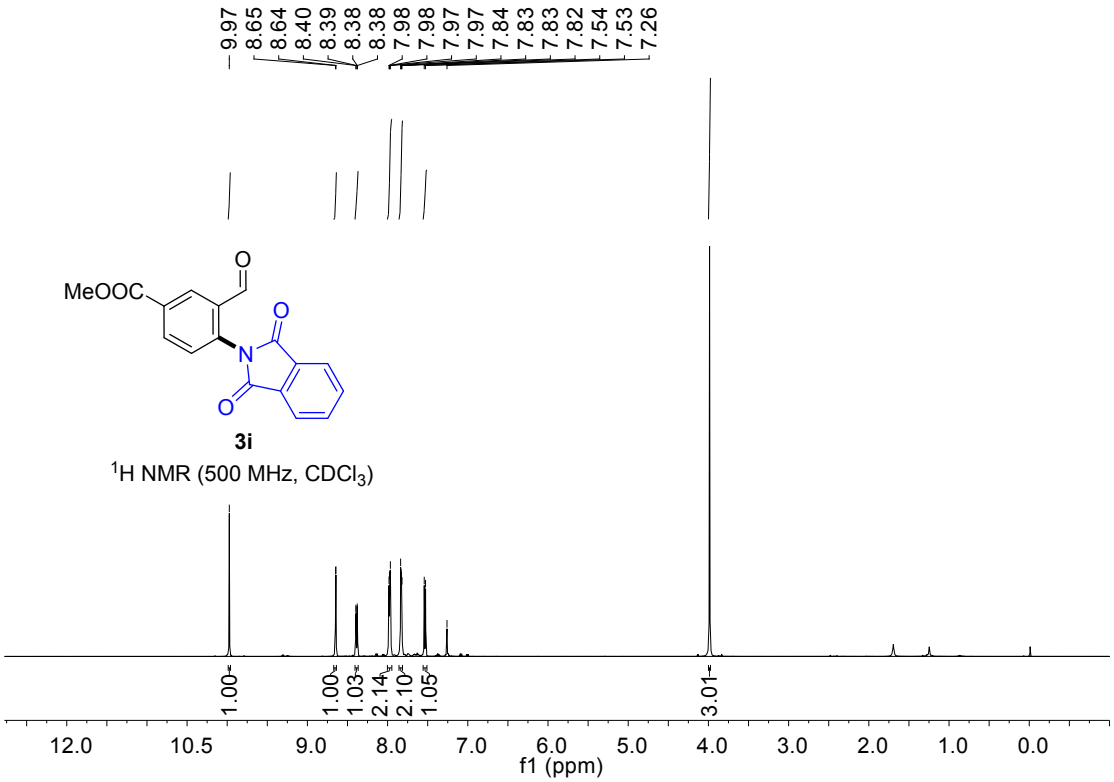
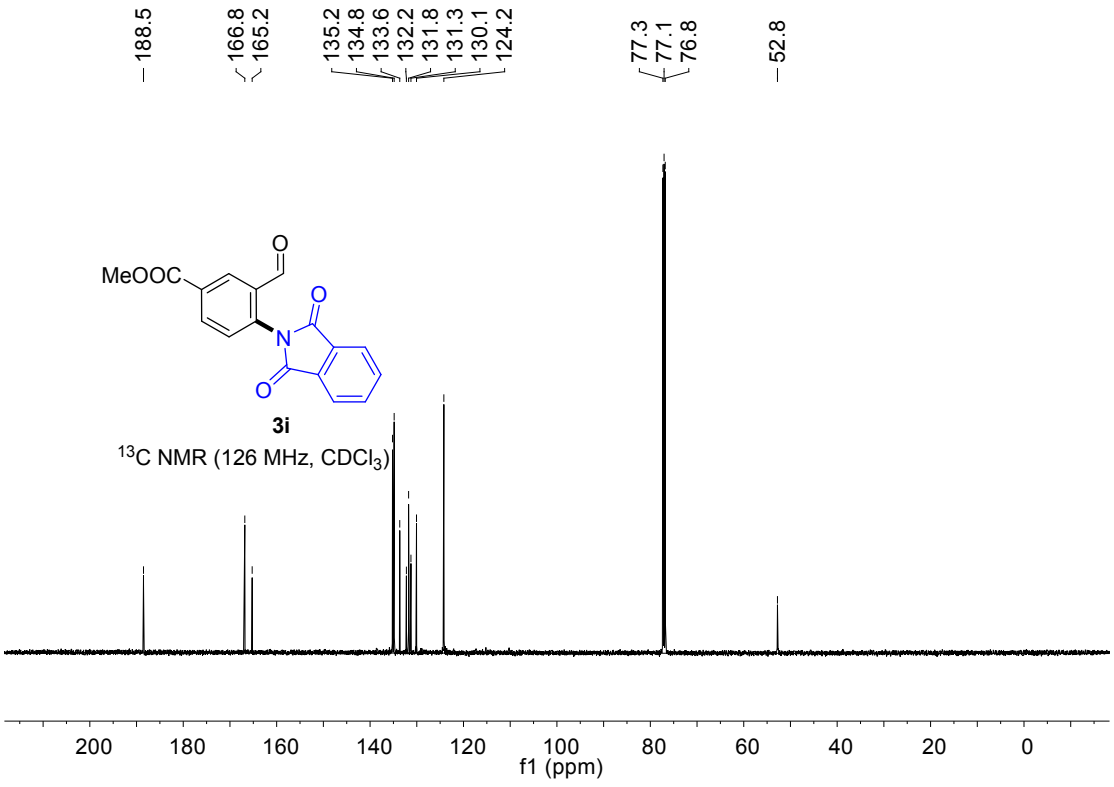
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


189.5
167.5
140.0
135.3
134.5
133.2
132.0
132.0
129.9
129.1
124.0
77.3
77.1
76.8
21.1
Me
O
N
O
O
3h
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



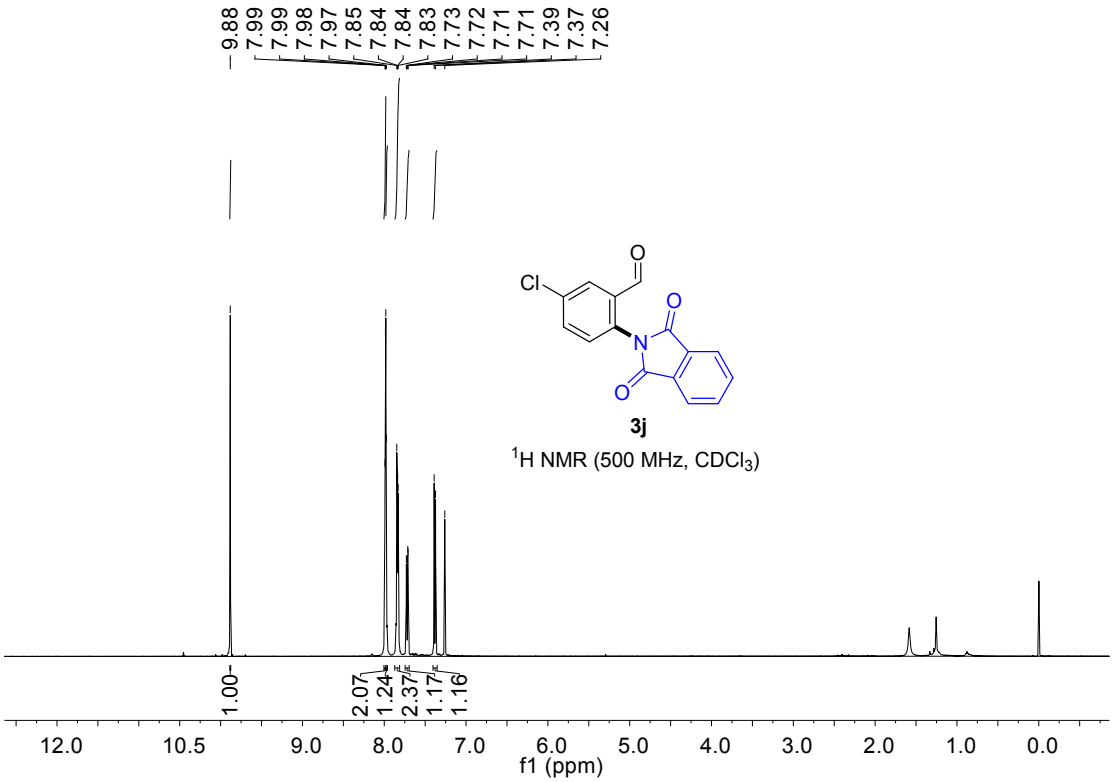
3i

^{1}H NMR (500 MHz, $CDCl_3$)



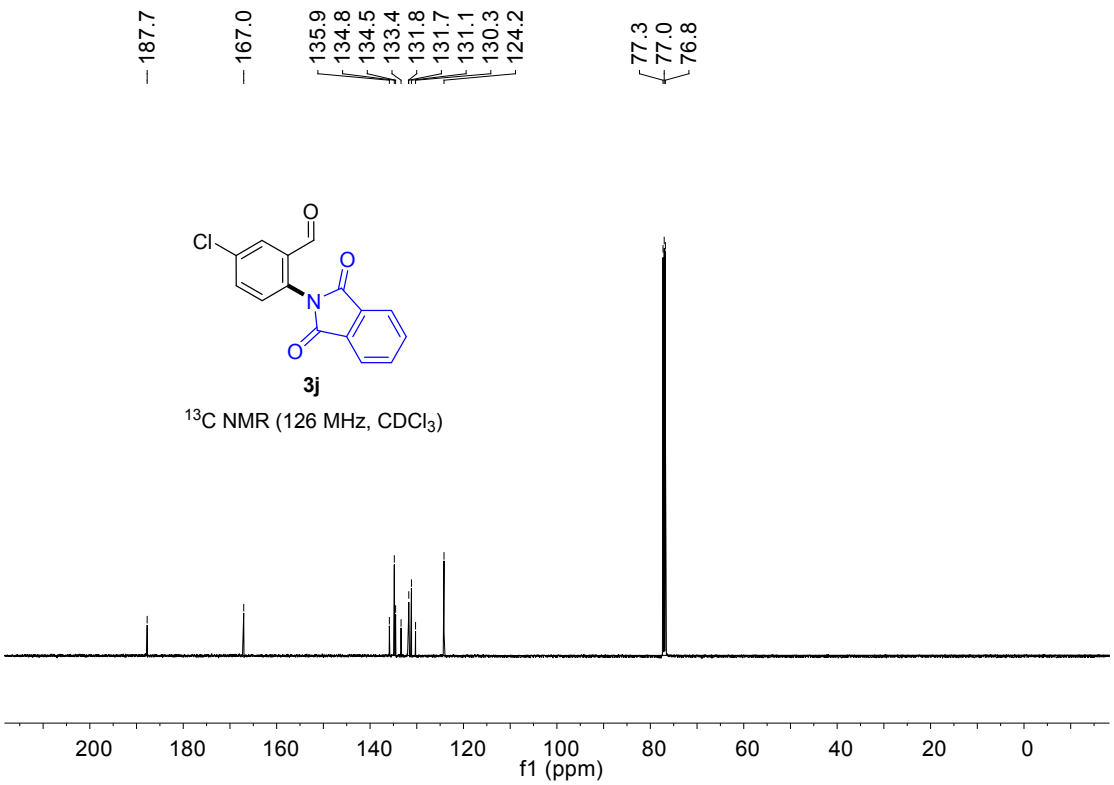
3i

^{13}C NMR (126 MHz, $CDCl_3$)



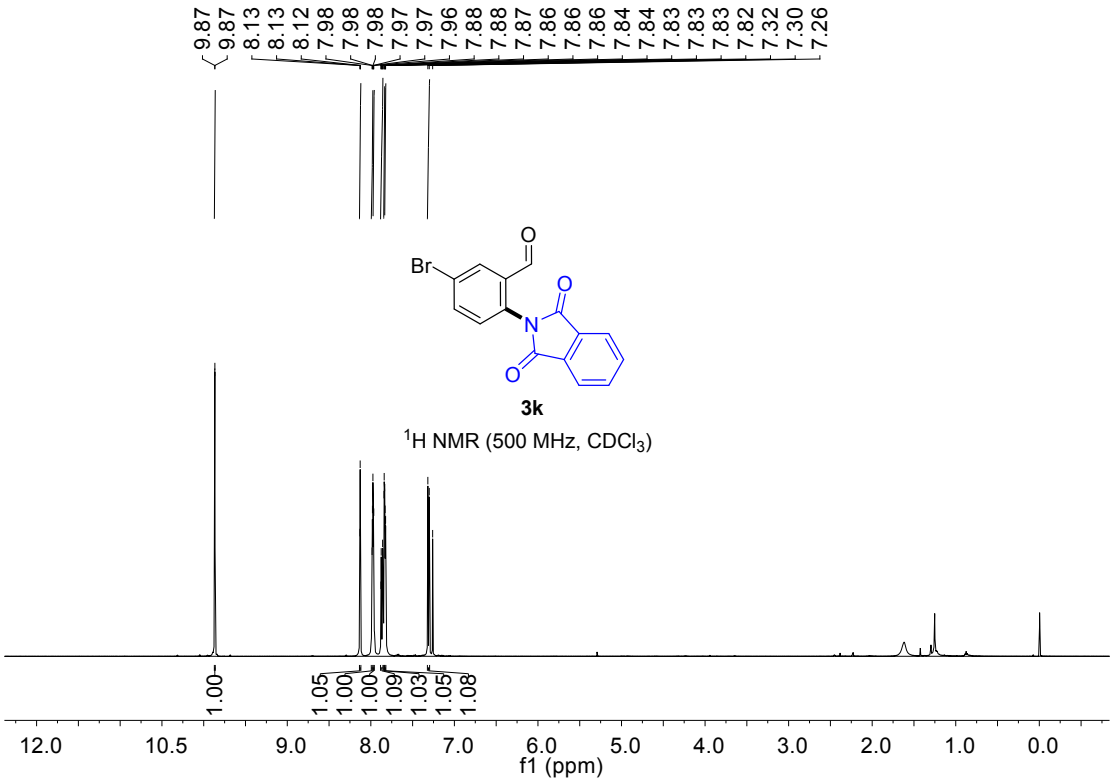
3j

^{1}H NMR (500 MHz, $CDCl_3$)



3j

^{13}C NMR (126 MHz, $CDCl_3$)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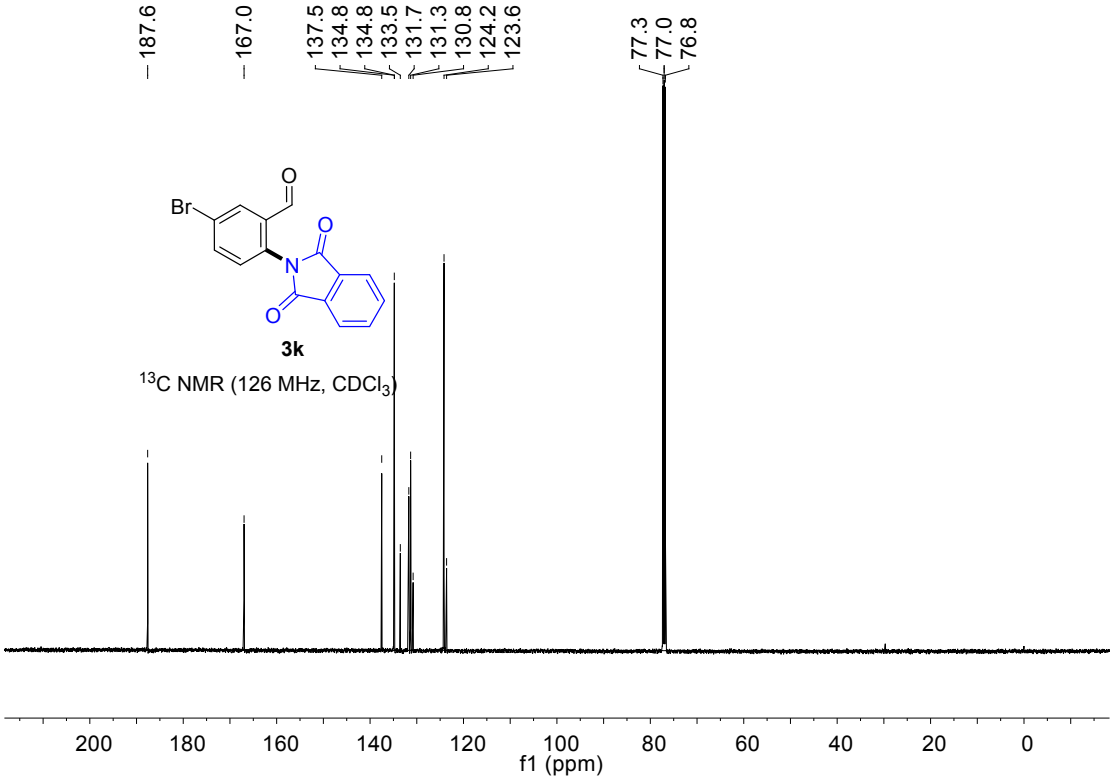
8.12
7.98
7.98
7.98
7.97
7.97
7.96
7.88
7.88
7.87
7.86
7.86
7.86
7.84
7.84
7.83
7.83
7.83
7.82
7.32
7.30
7.26
Br
O
O
N
O
3k
1H NMR (500 MHz, CDCl3)
1.00
1.05
1.00
1.00
1.09
1.03
1.05
1.08
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


187.6
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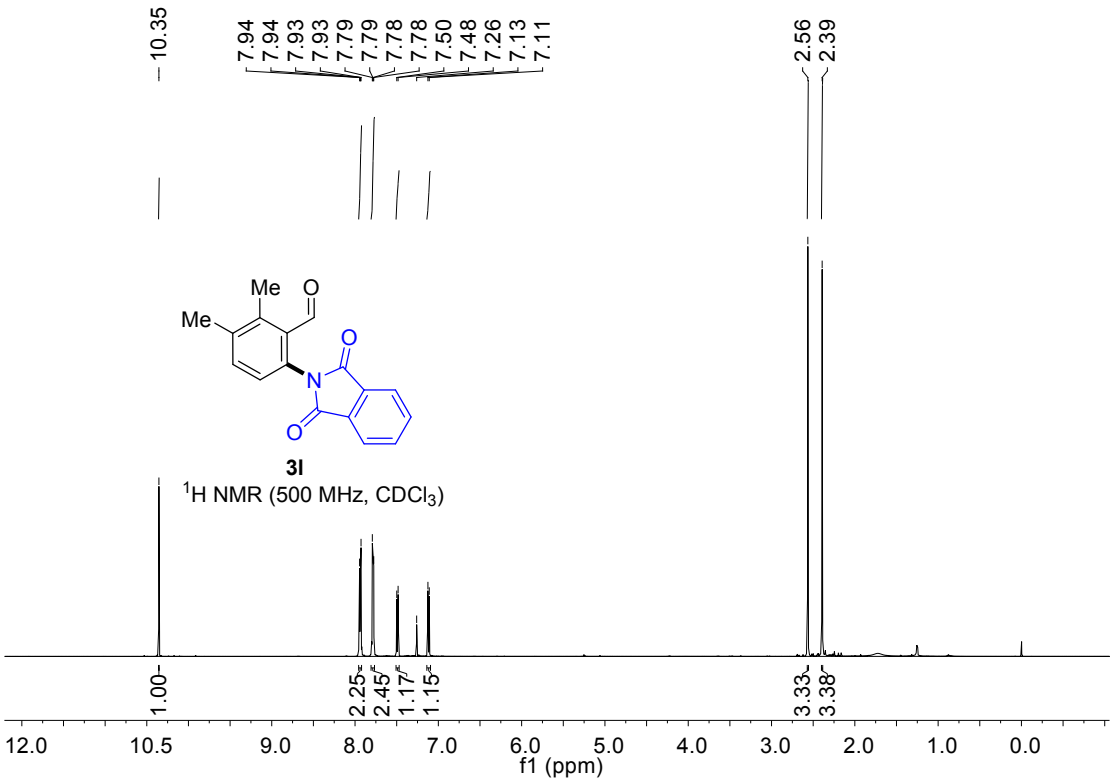
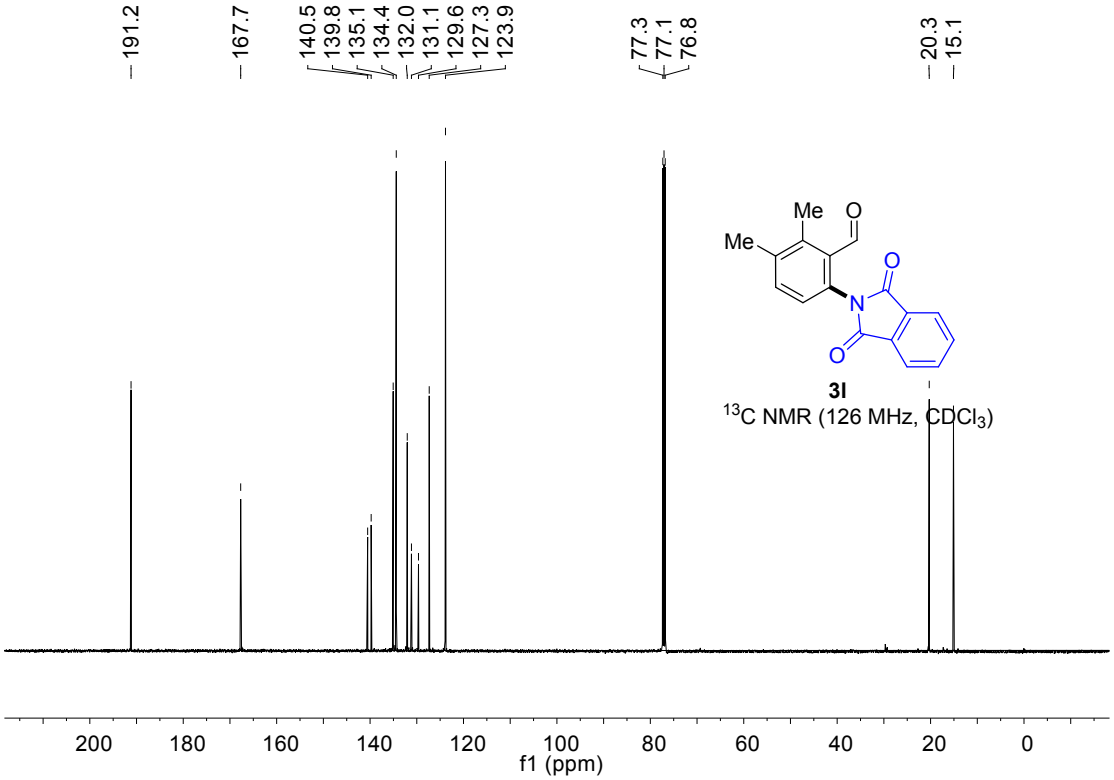
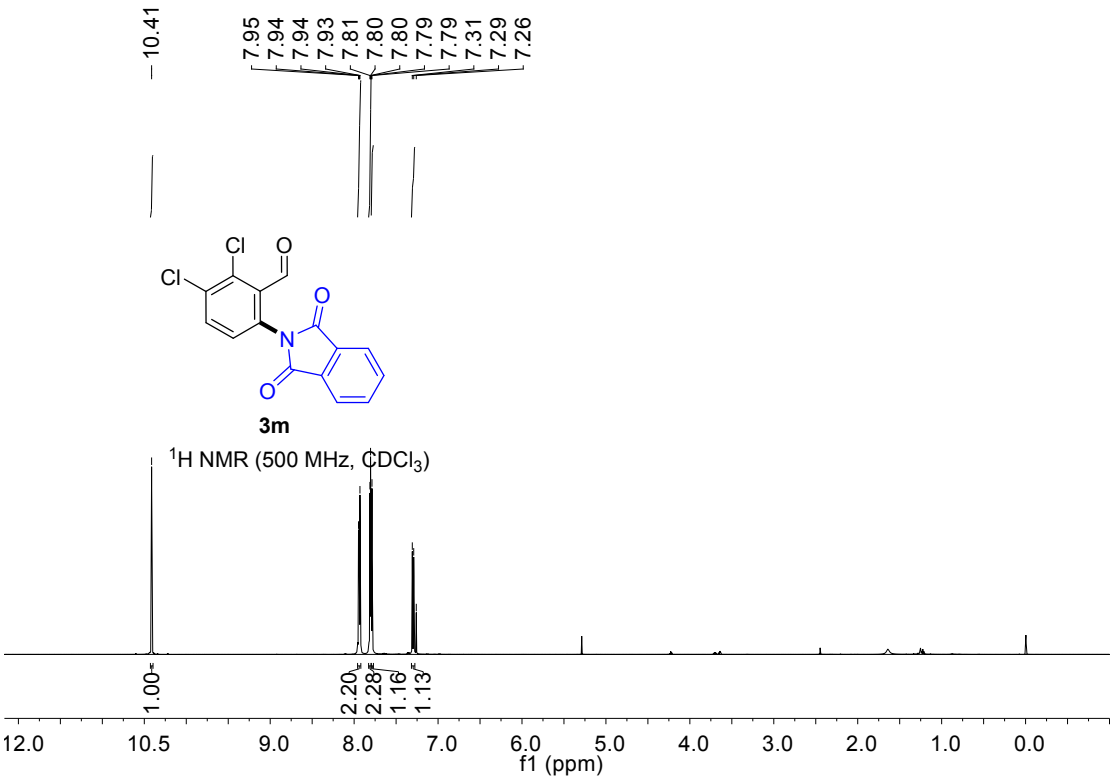
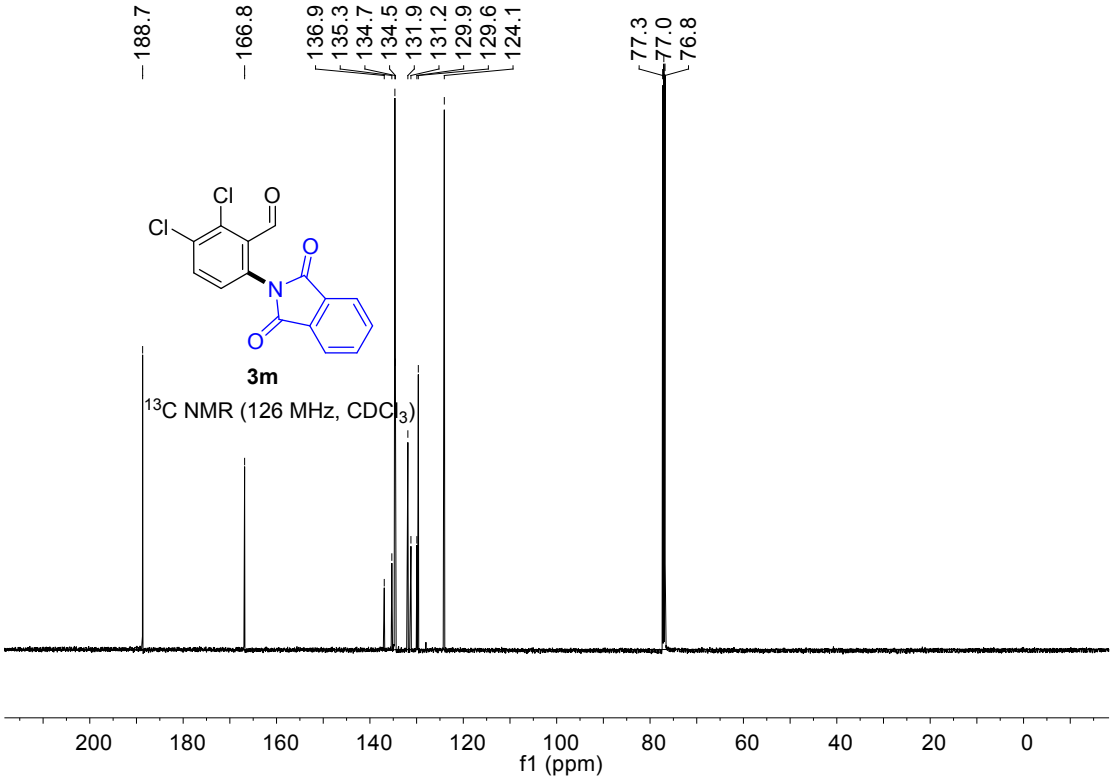
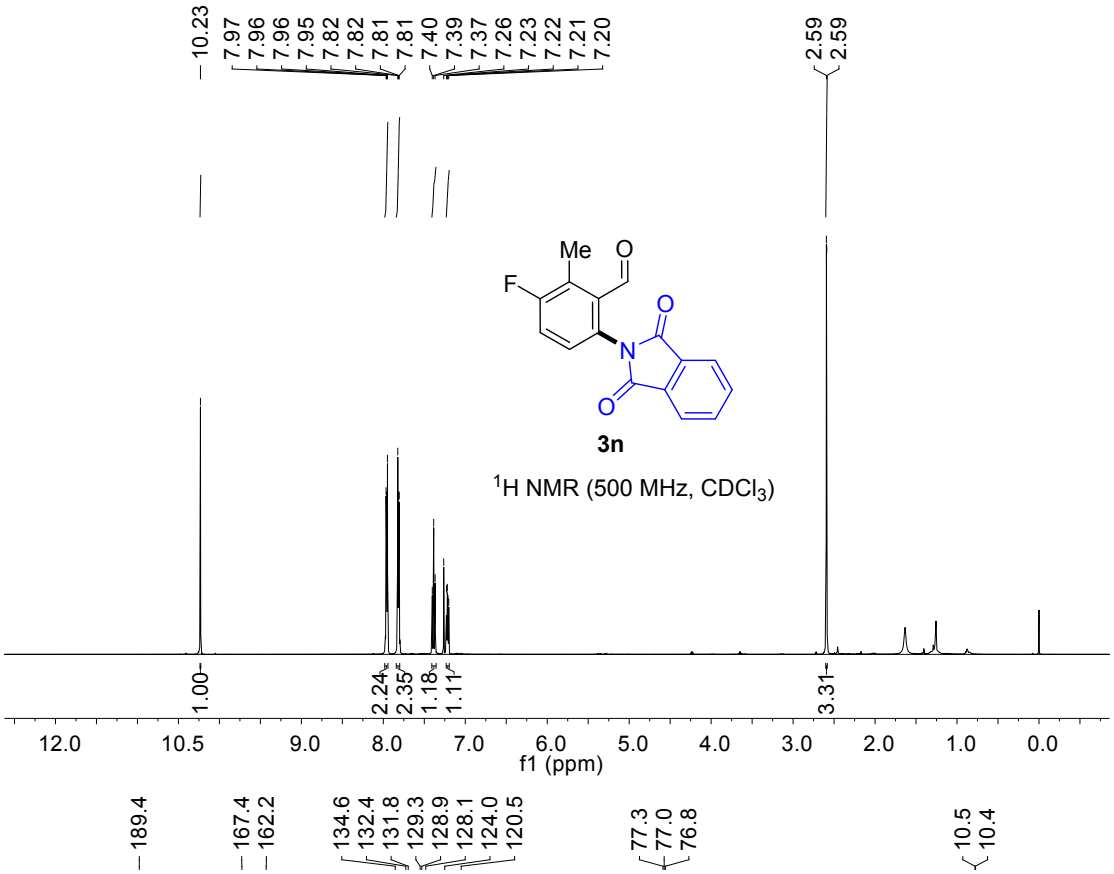
137.5
134.8
134.8
133.5
131.7
131.3
130.8
124.2
123.6
77.3
77.0
76.8
Br
O
O
N
O
3k
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


10.35
7.94
7.94
7.93
7.93
7.79
7.79
7.78
7.78
7.50
7.48
7.26
7.13
7.11
2.56
2.39
Me
O
Me
O
N
O
3l
1H NMR (500 MHz, CDCl3)
1.00
2.25
2.45
1.17
1.15
3.33
3.38
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


191.2
167.7
140.5
139.8
135.1
134.4
132.0
131.1
129.6
127.3
123.9
77.3
77.1
76.8
20.3
15.1
Me
O
Me
O
N
O
3l
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

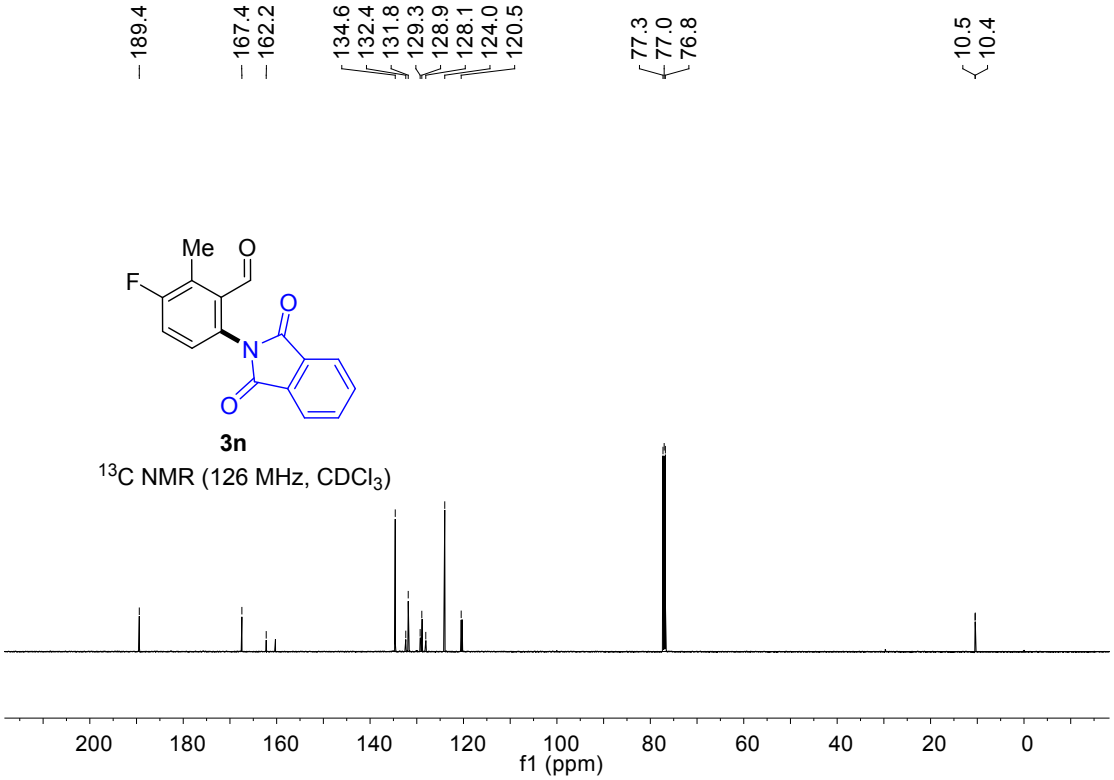

10.41
7.95
7.94
7.94
7.93
7.81
7.80
7.80
7.79
7.79
7.31
7.29
7.26
Cl
Cl
O
O
N
O
3m
1H NMR (500 MHz, CDCl3)
1.00
2.20
2.28
1.16
1.13
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


188.7
166.8
136.9
135.3
134.7
134.5
131.9
131.2
129.9
129.6
124.1
77.3
77.0
76.8
Cl
Cl
O
O
N
O
3m
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



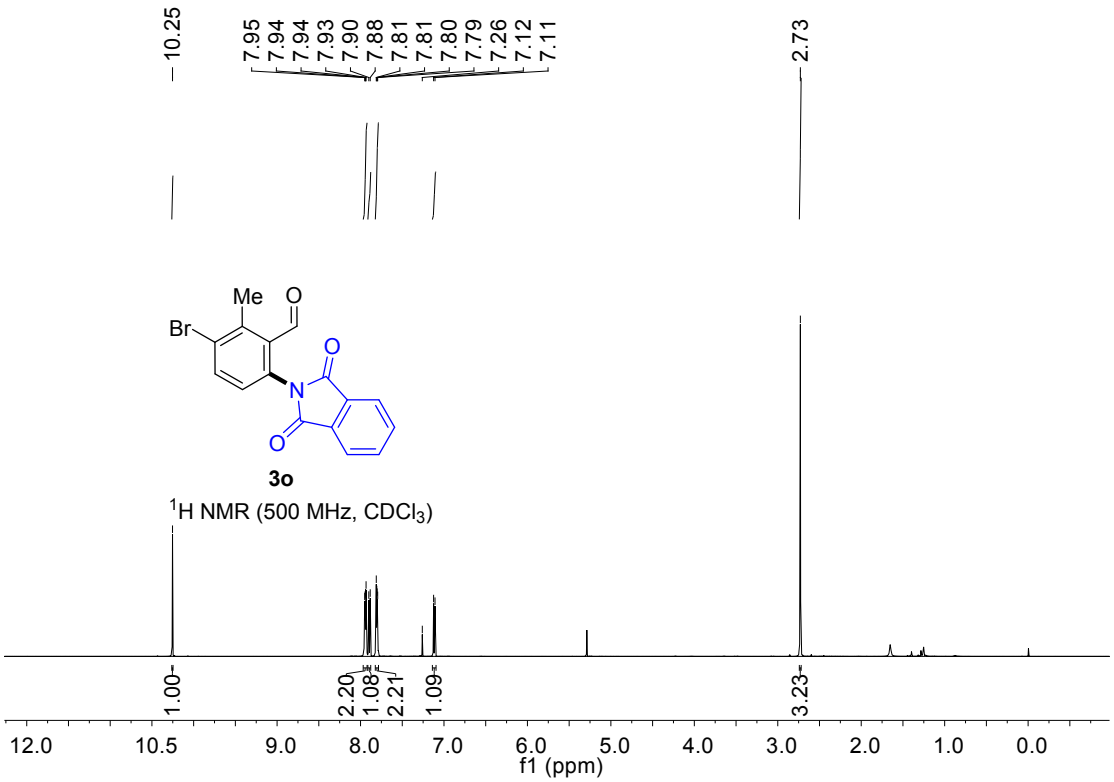
3n

^{1}H NMR (500 MHz, $CDCl_3$)



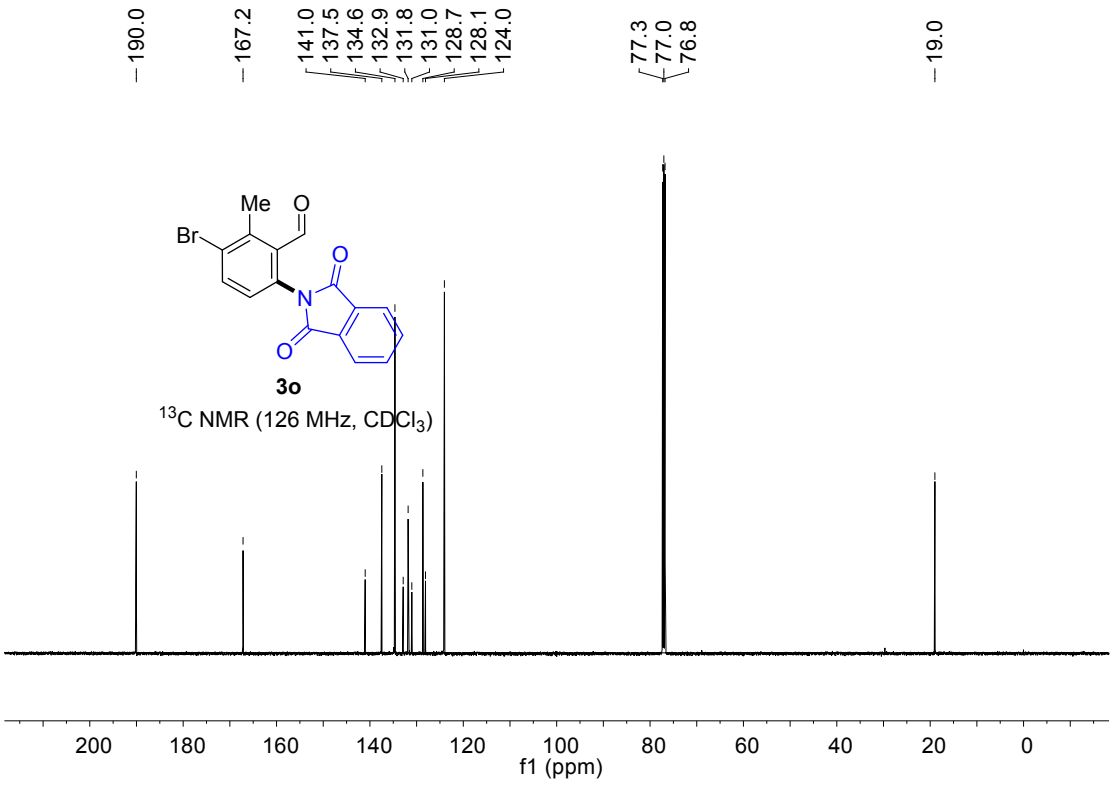
3n

^{13}C NMR (126 MHz, $CDCl_3$)



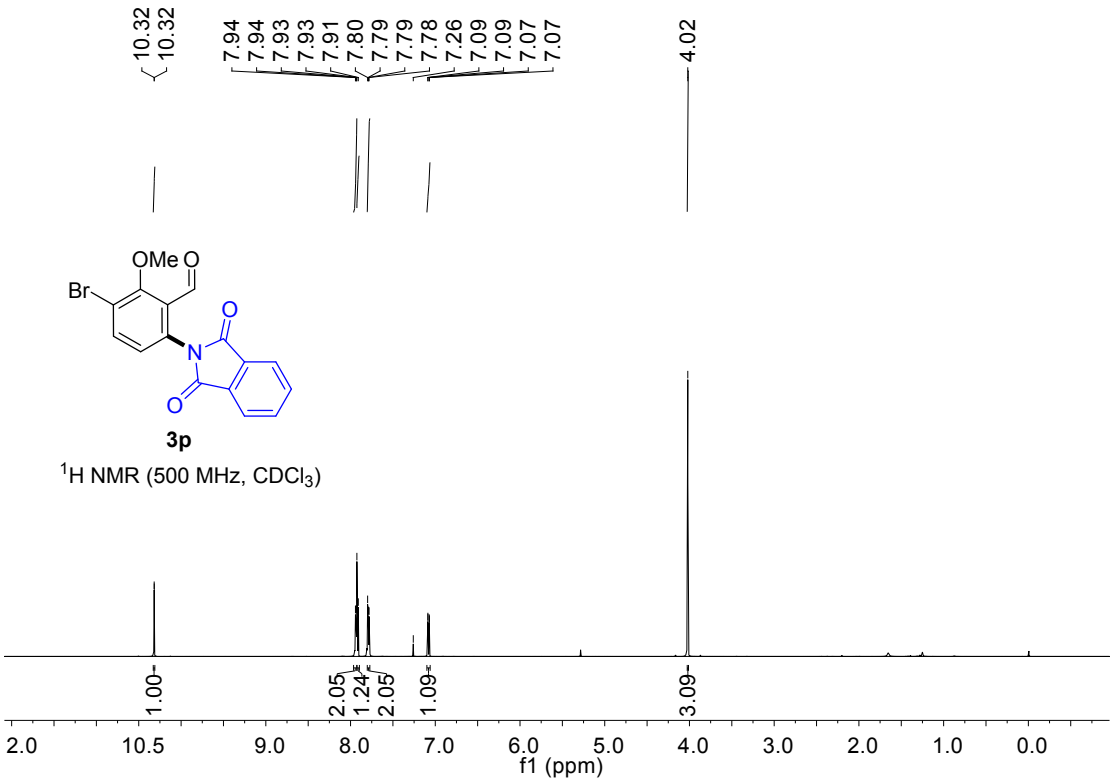
3o

^{1}H NMR (500 MHz, $CDCl_3$)



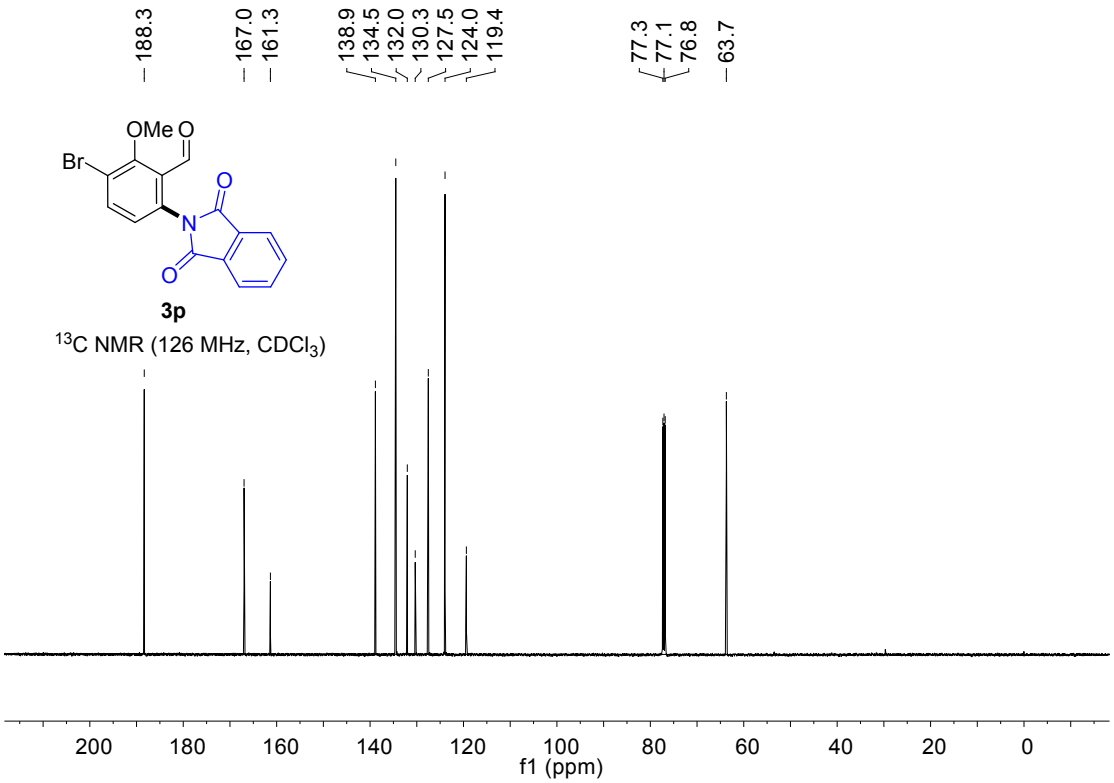
3o

^{13}C NMR (126 MHz, $CDCl_3$)



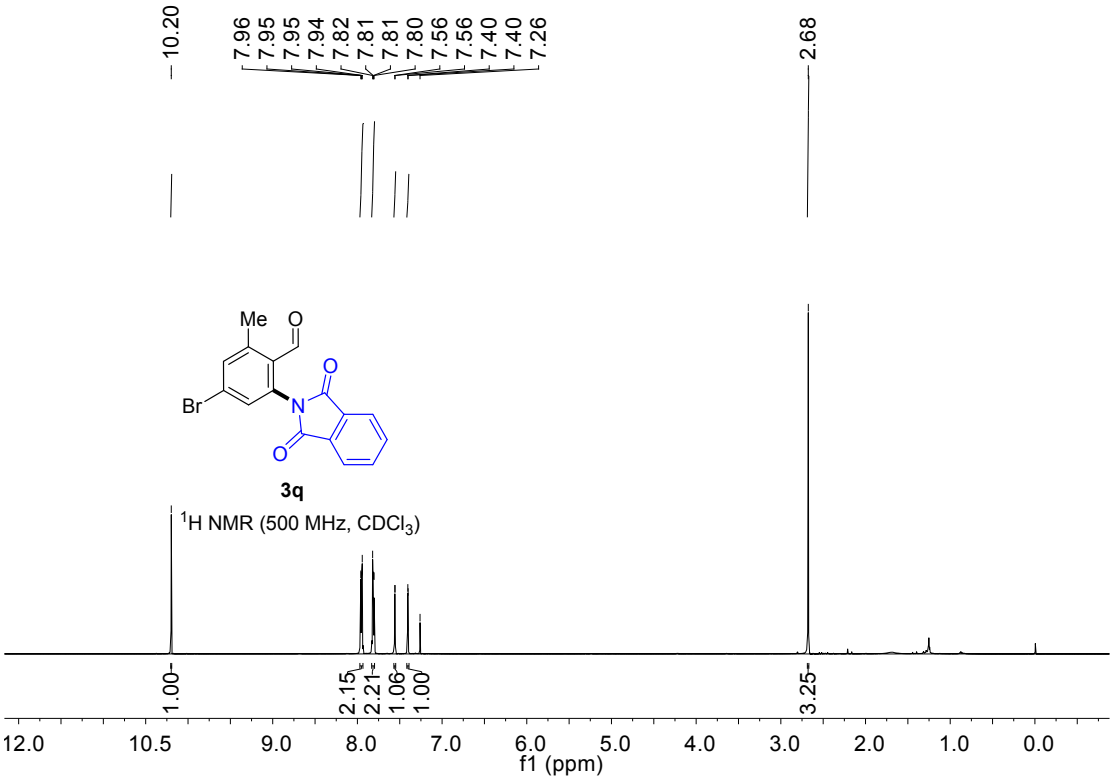
3p

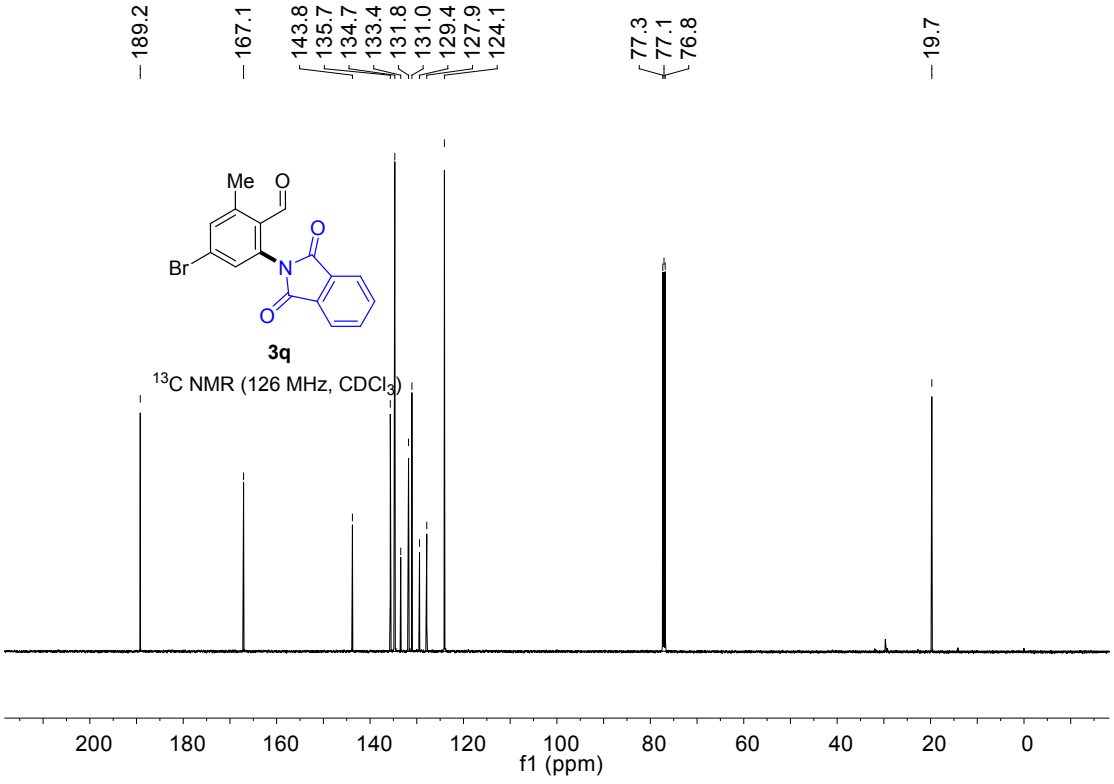
^{1}H NMR (500 MHz, $CDCl_3$)

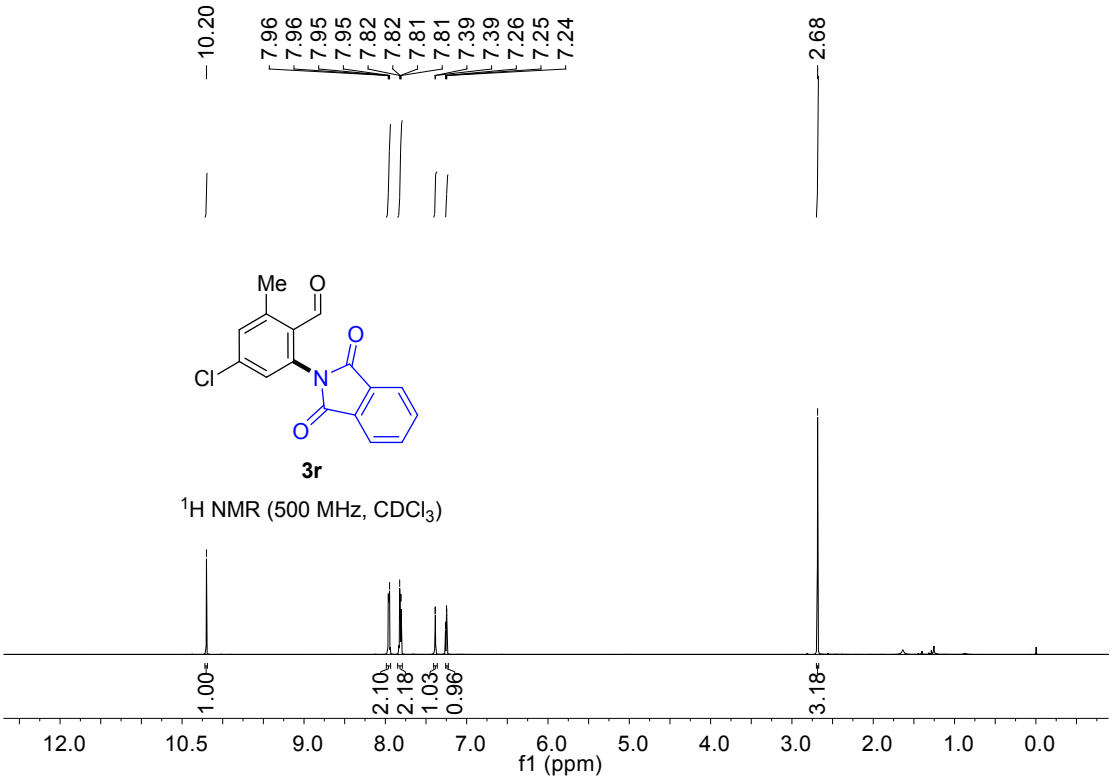


3p

^{13}C NMR (126 MHz, $CDCl_3$)

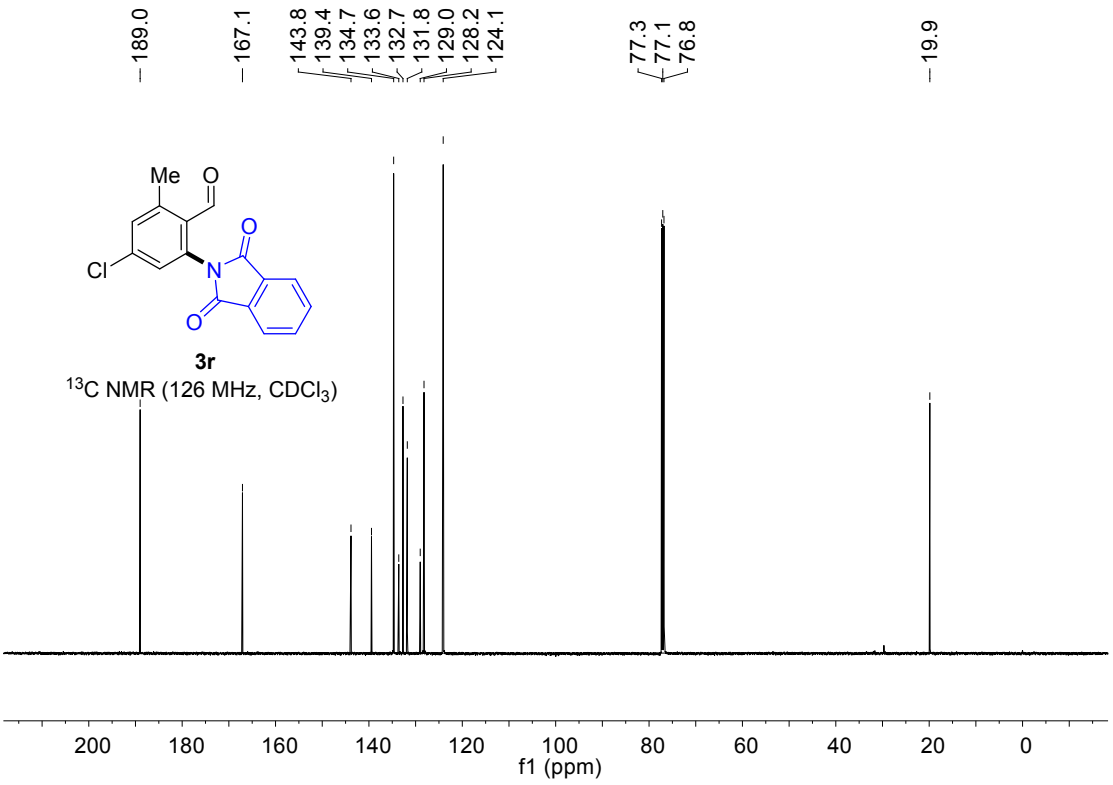

10.20
7.96
7.95
7.95
7.94
7.82
7.81
7.81
7.80
7.56
7.56
7.40
7.40
7.26
2.68
Me
O
Br
N
O
O
3q
1H NMR (500 MHz, CDCl3)
1.00
2.15
2.21
1.06
1.00
3.25
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


189.2
167.1
143.8
135.7
134.7
133.4
131.8
131.0
129.4
127.9
124.1
77.3
77.1
76.8
19.7
Me
O
Br
N
O
O
3q
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



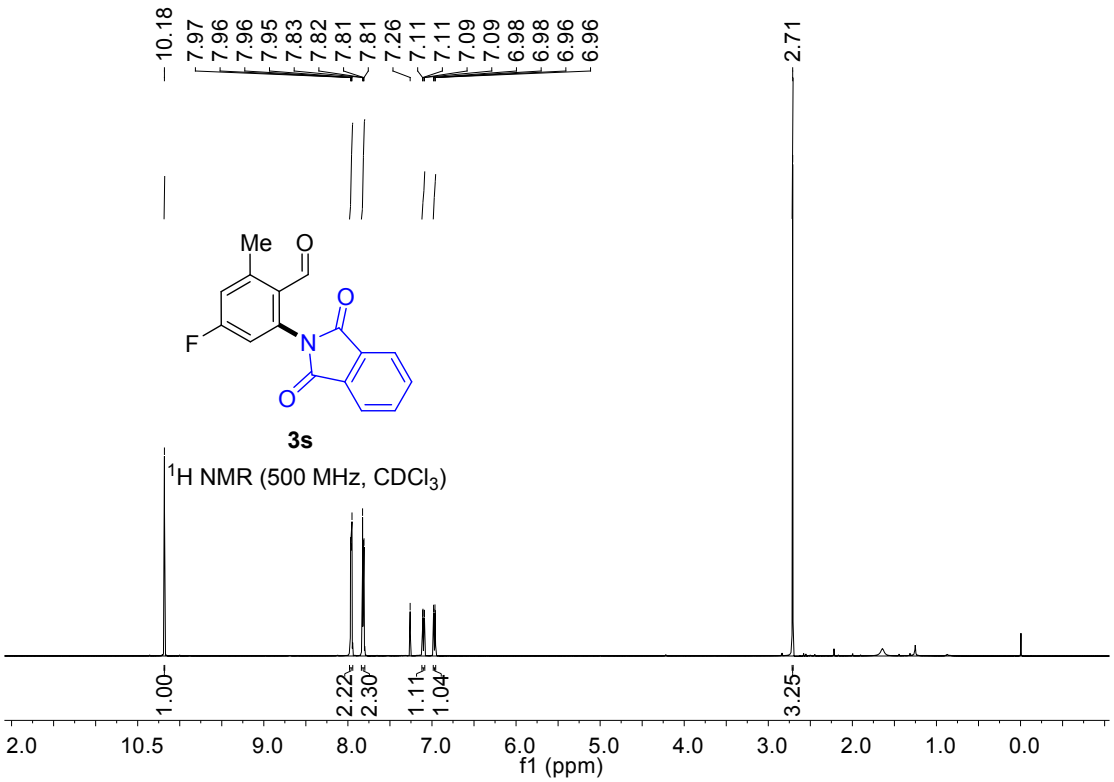
3r

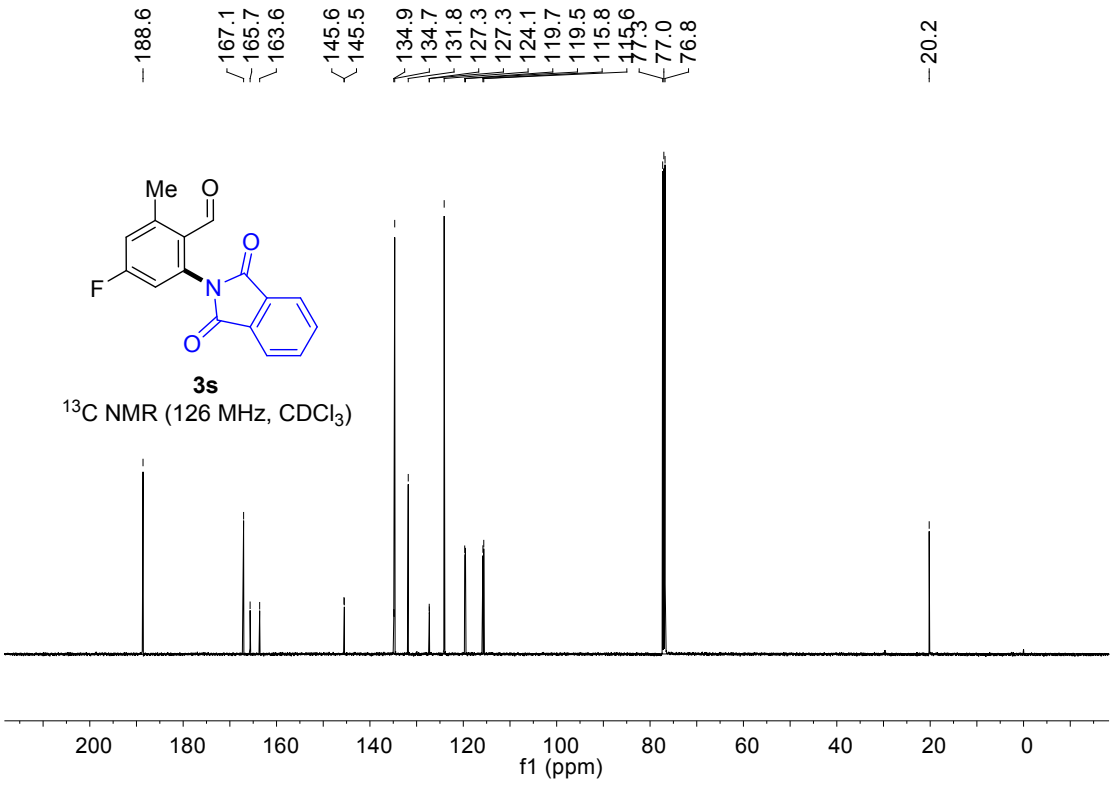
^{1}H NMR (500 MHz, $CDCl_3$)

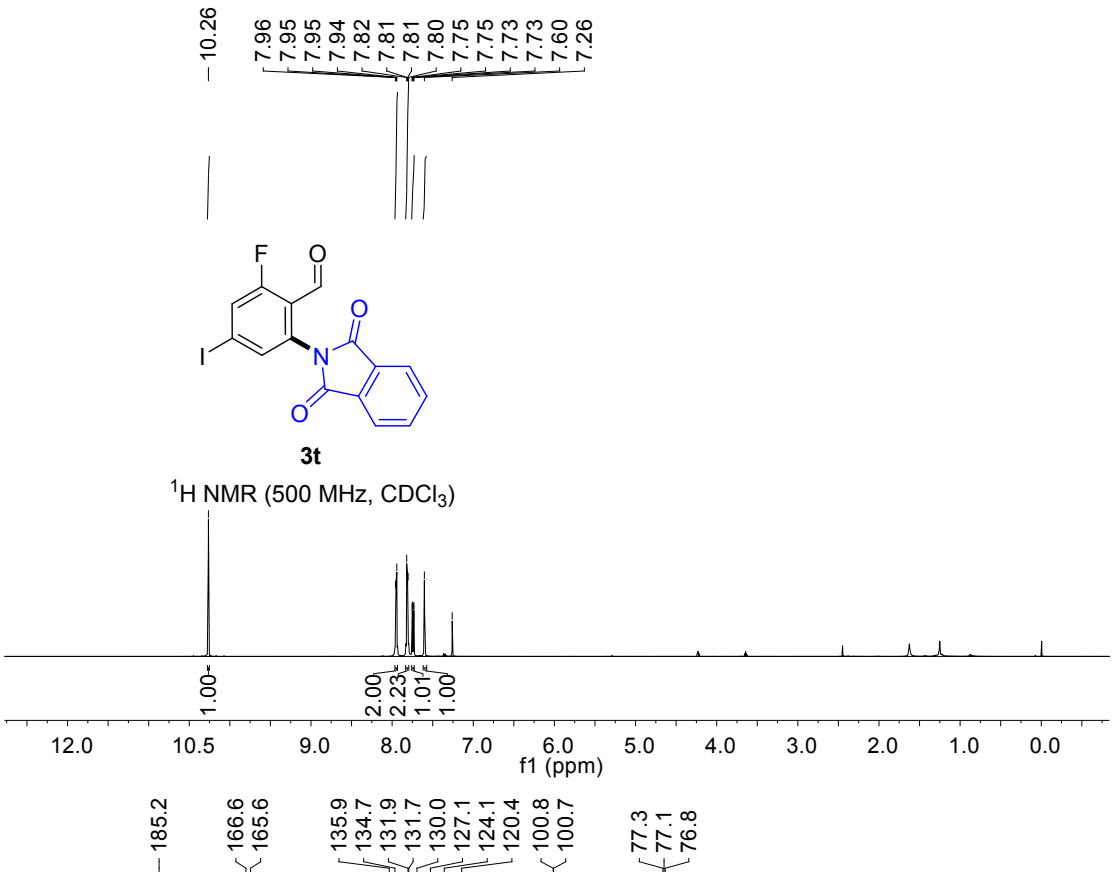


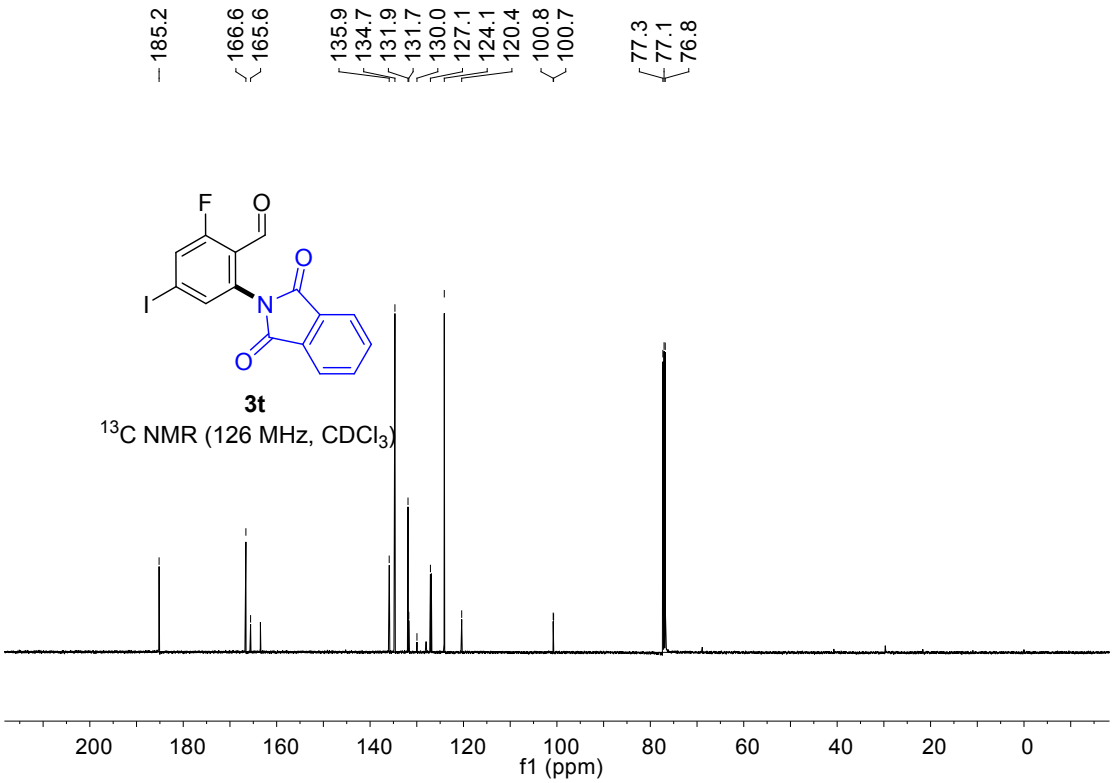
3r

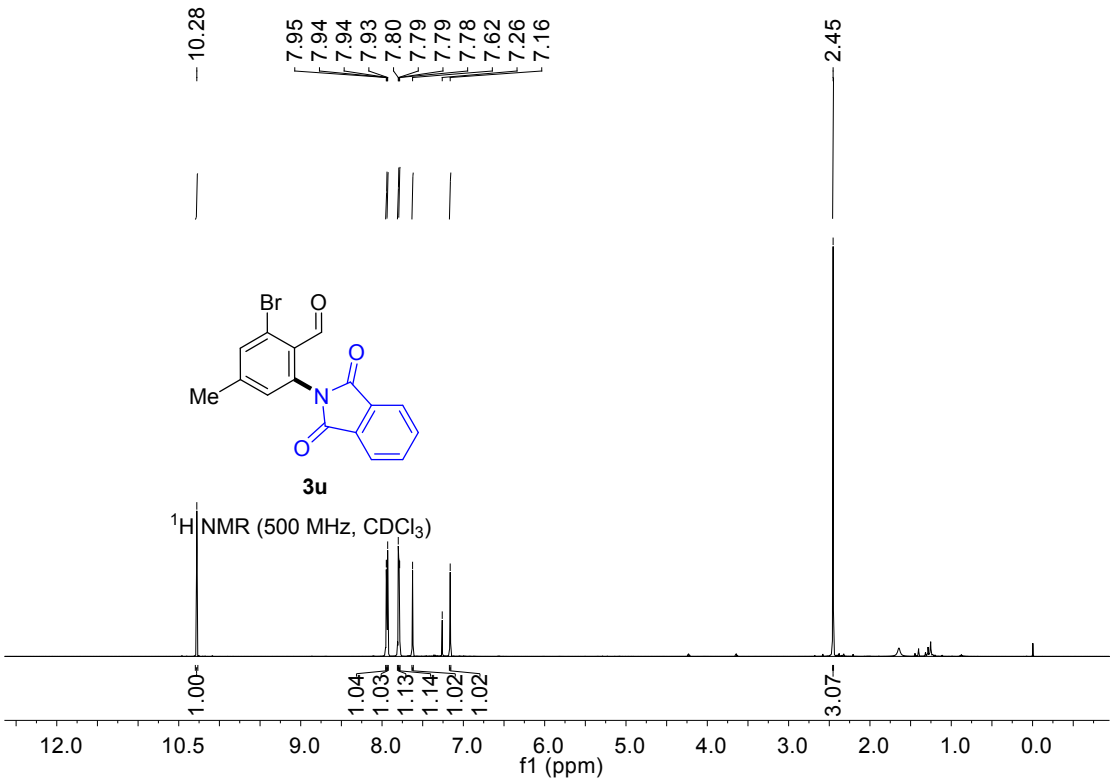
^{13}C NMR (126 MHz, $CDCl_3$)


10.18
7.97
7.96
7.96
7.95
7.83
7.82
7.81
7.81
7.26
7.11
7.11
7.09
7.09
6.98
6.98
6.96
6.96
2.71
Me
O
O
N
F
O
3s
1H NMR (500 MHz, CDCl3)
1.00
2.22
2.30
1.11
1.04
3.25
2.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


188.6
167.1
165.7
163.6
145.6
145.5
134.9
134.7
131.8
127.3
127.3
124.1
119.7
119.5
115.8
115.6
77.3
77.0
76.8
20.2
Me
O
O
N
F
O
3s
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

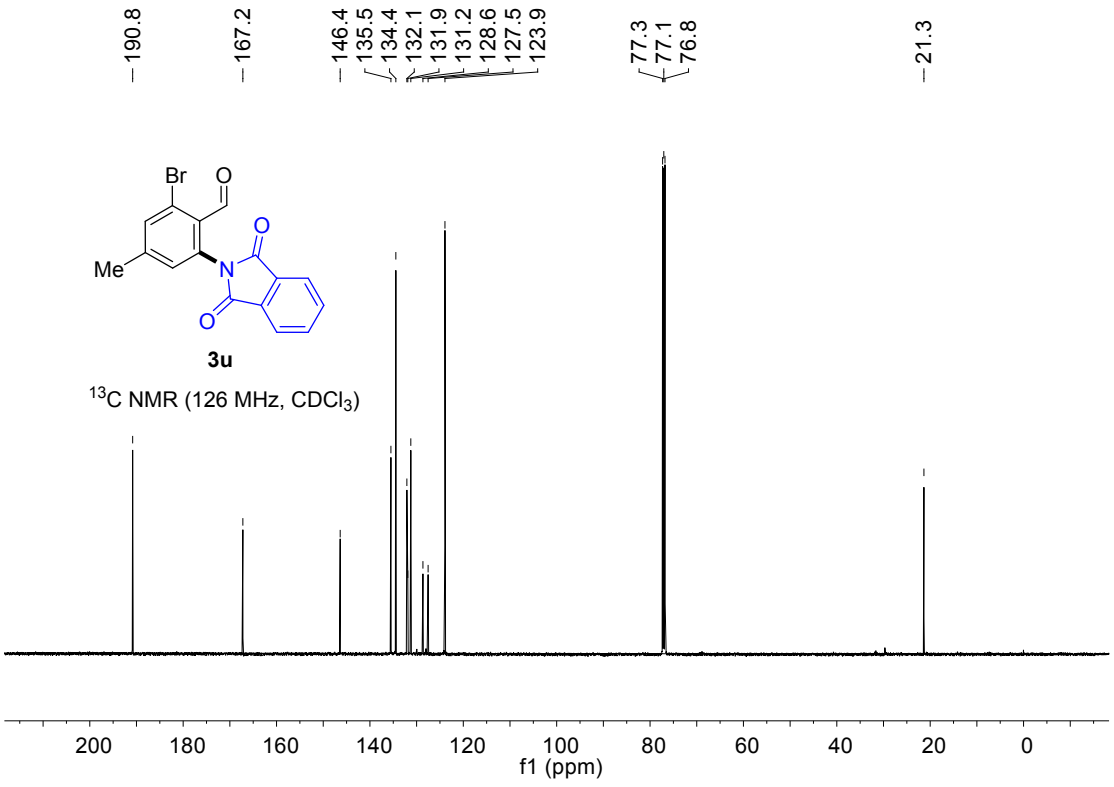

10.26
7.96
7.95
7.95
7.94
7.82
7.81
7.81
7.80
7.75
7.75
7.73
7.73
7.60
7.26
F
O
O
I
N
O
3t
1H NMR (500 MHz, CDCl3)
1.00
2.00
2.23
1.01
1.00
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


185.2
166.6
165.6
135.9
134.7
131.9
131.7
130.0
127.1
124.1
120.4
100.8
100.7
77.3
77.1
76.8
F
O
O
I
N
O
3t
13C NMR (126 MHz, CDCl3)
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



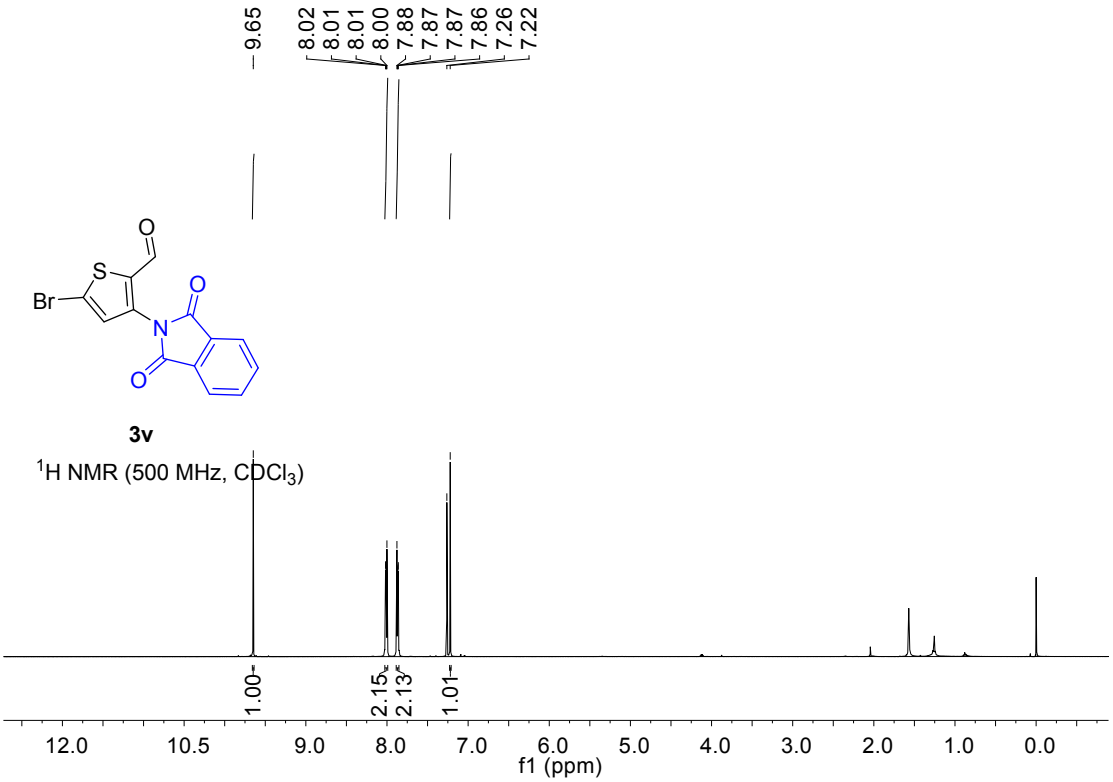
3u

^{1}H NMR (500 MHz, $CDCl_3$)



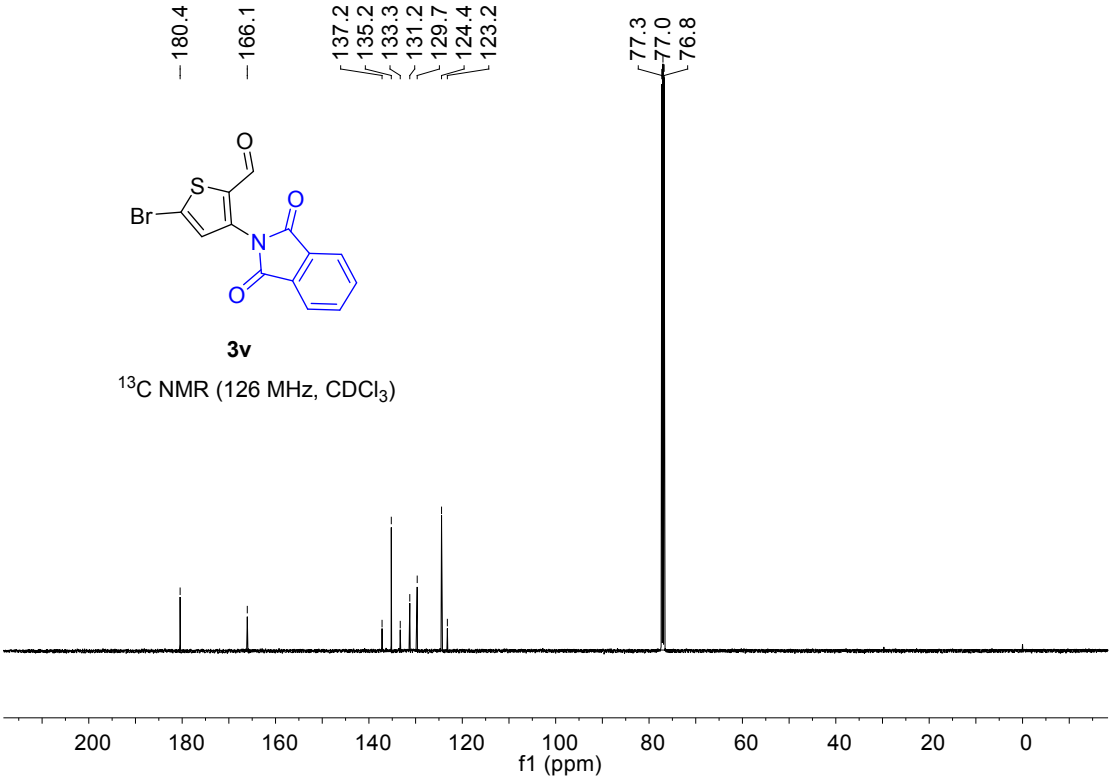
3u

^{13}C NMR (126 MHz, $CDCl_3$)



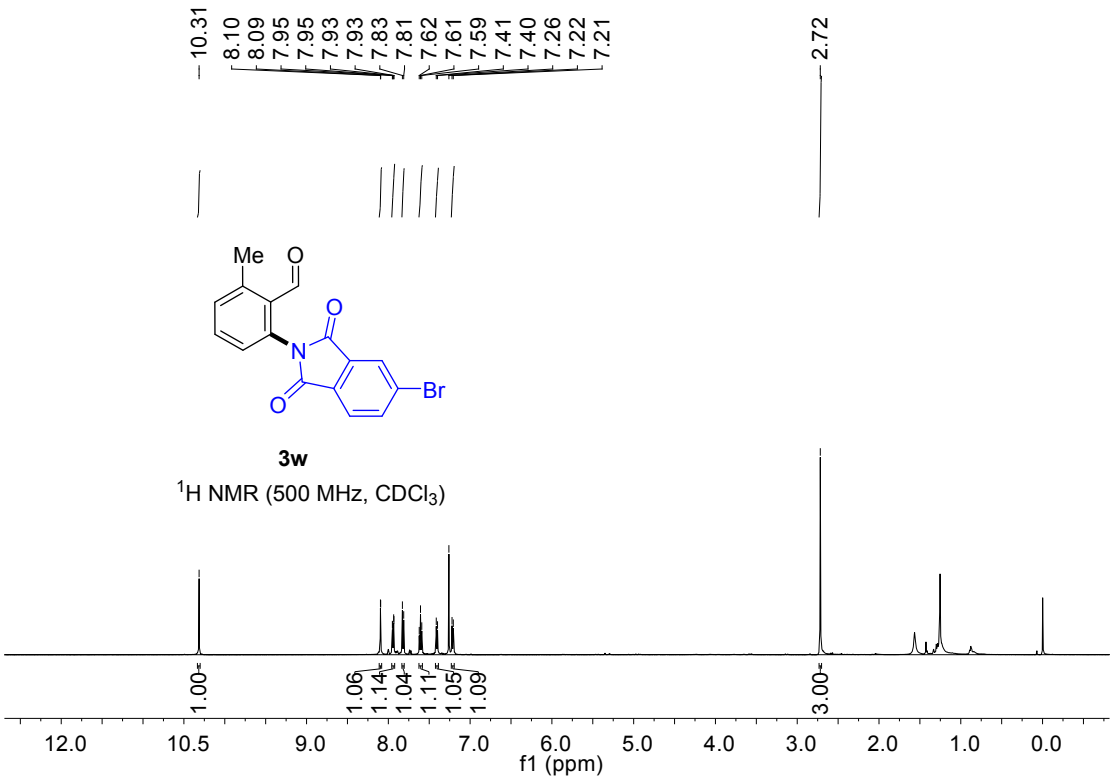
3v

^{1}H NMR (500 MHz, $CDCl_3$)



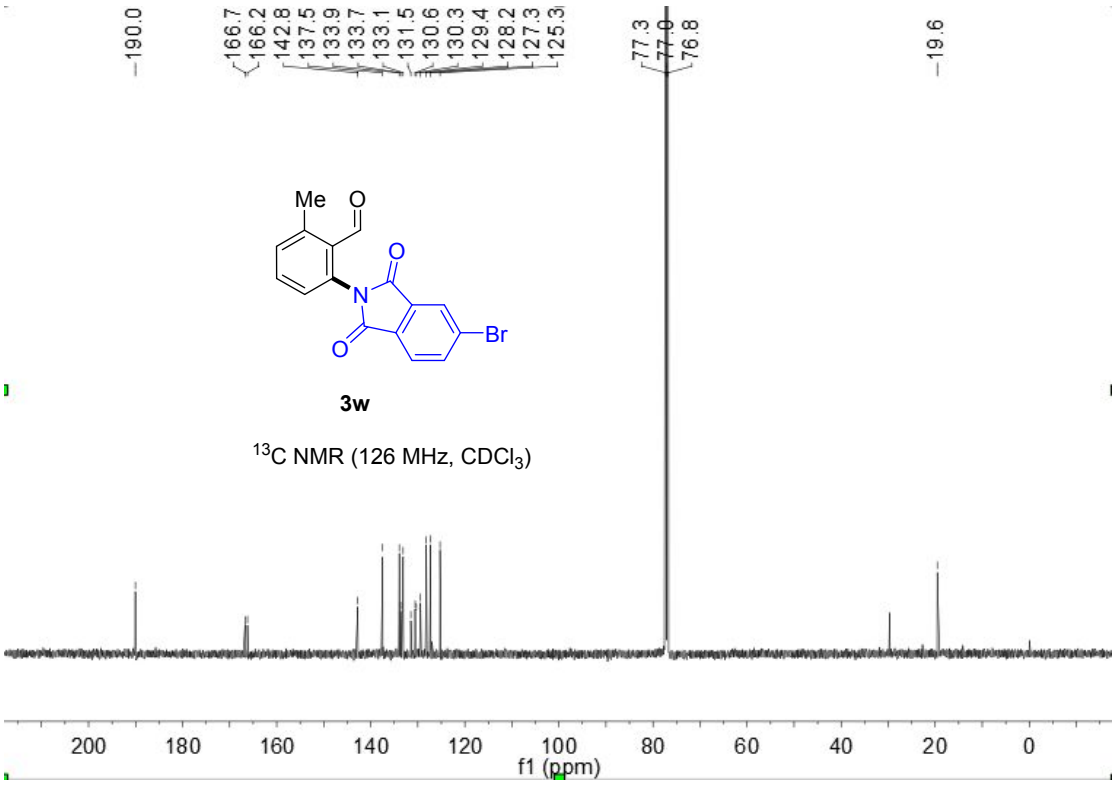
3v

^{13}C NMR (126 MHz, $CDCl_3$)



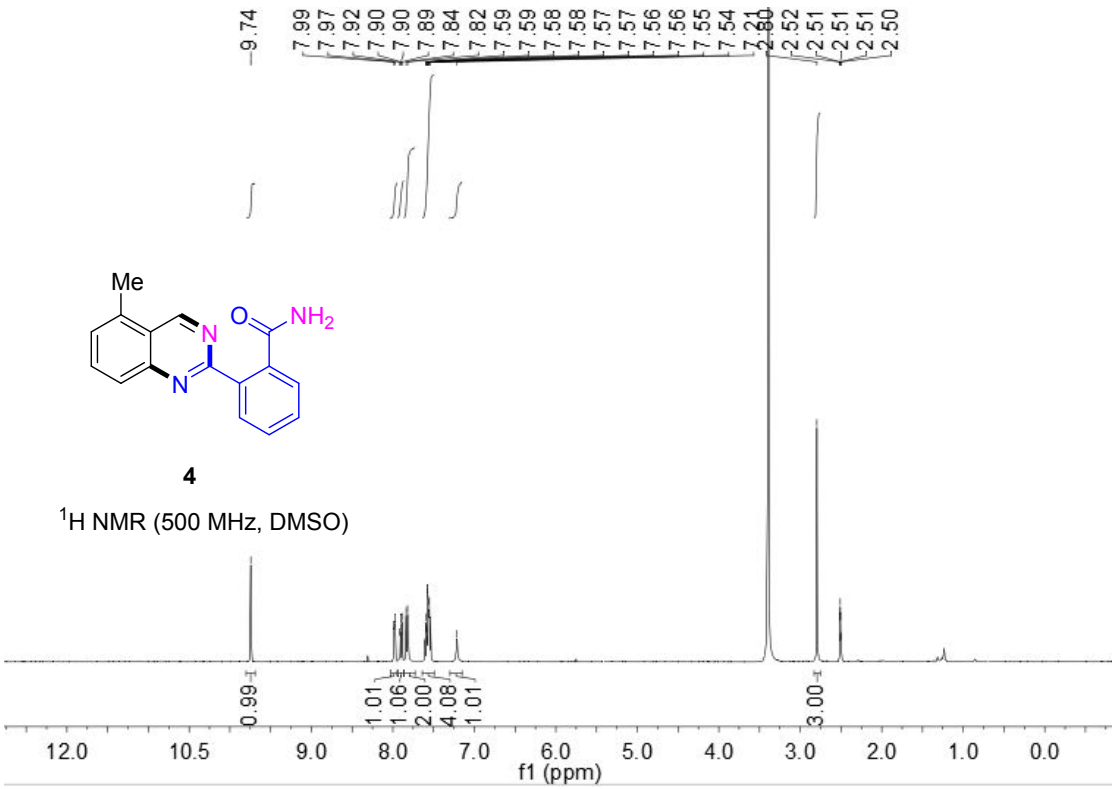
3w

^{1}H NMR (500 MHz, $CDCl_3$)



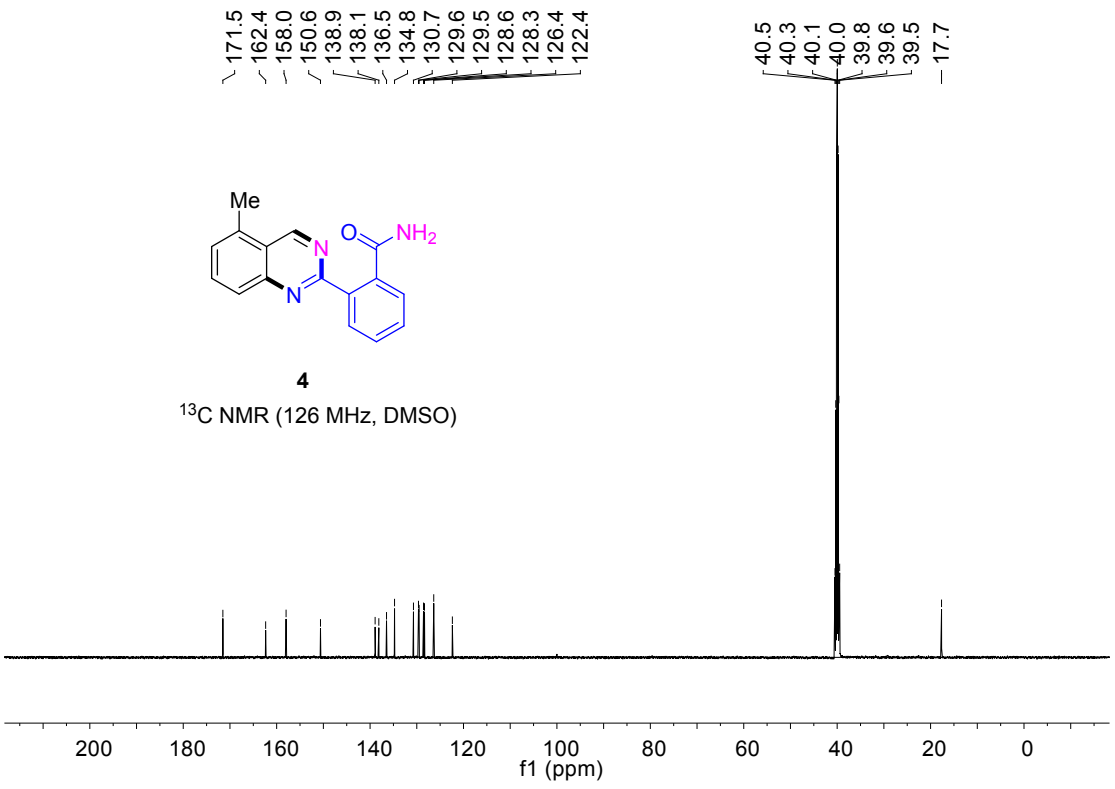
3w

^{13}C NMR (126 MHz, $CDCl_3$)



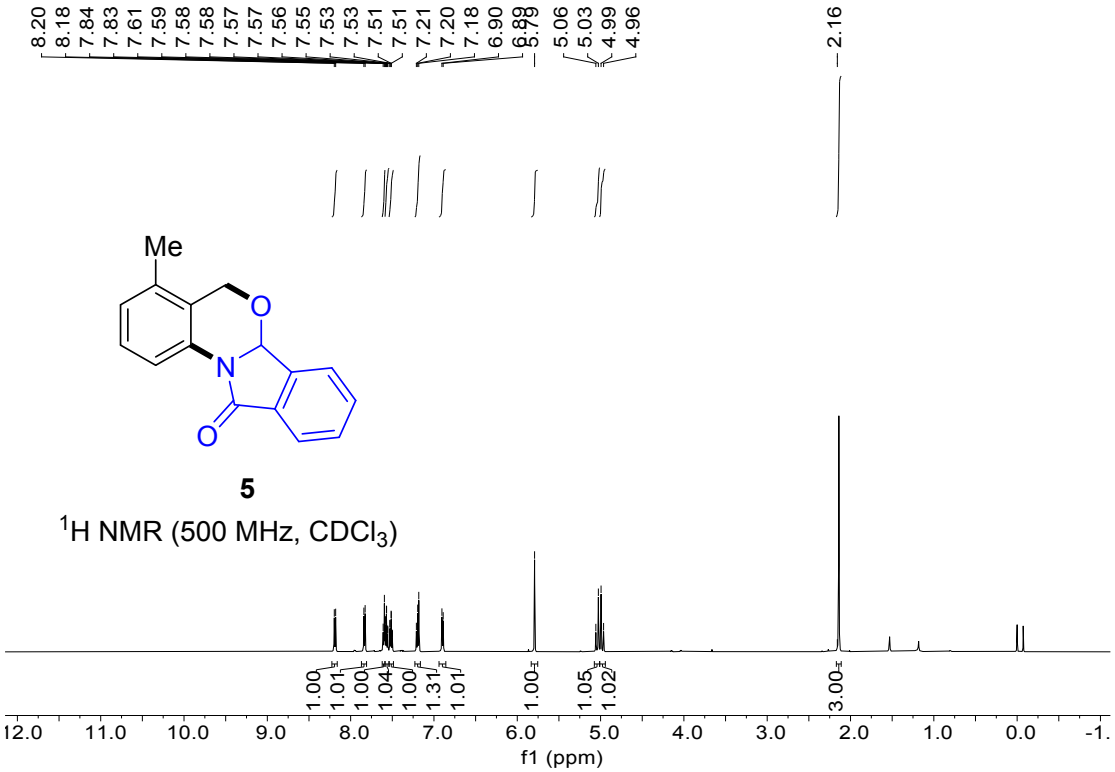
4

^{1}H NMR (500 MHz, DMSO)



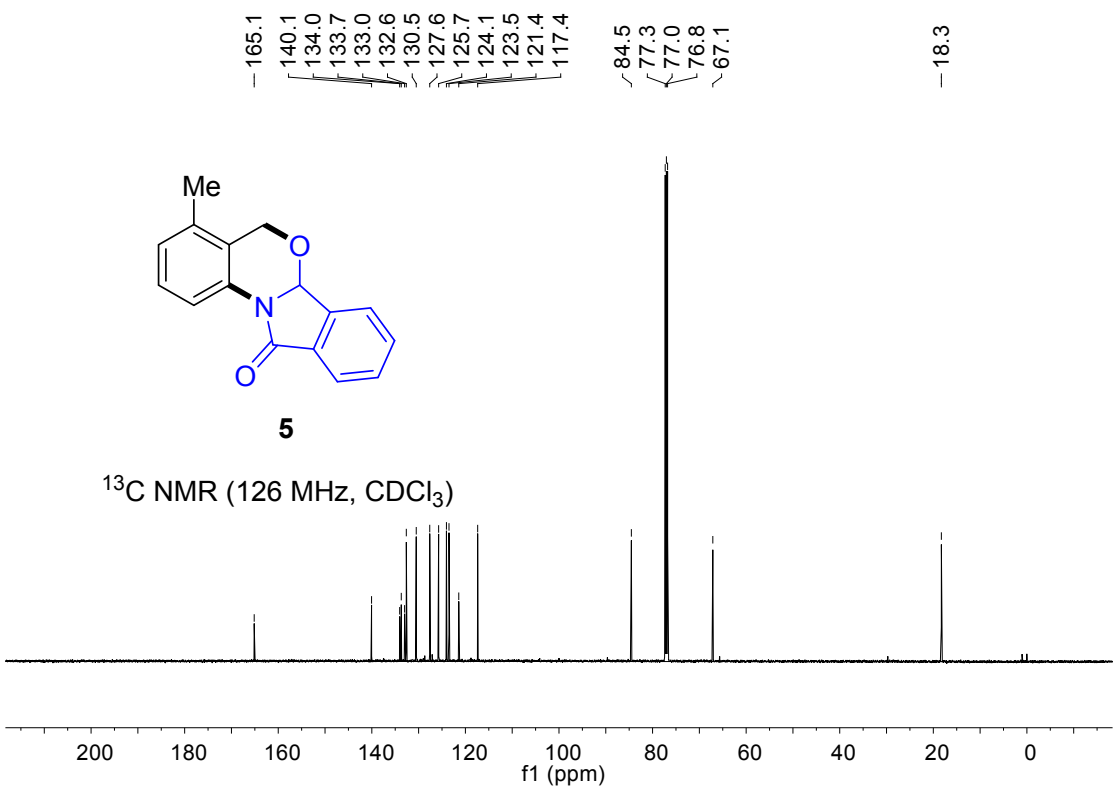
4

^{13}C NMR (126 MHz, DMSO)



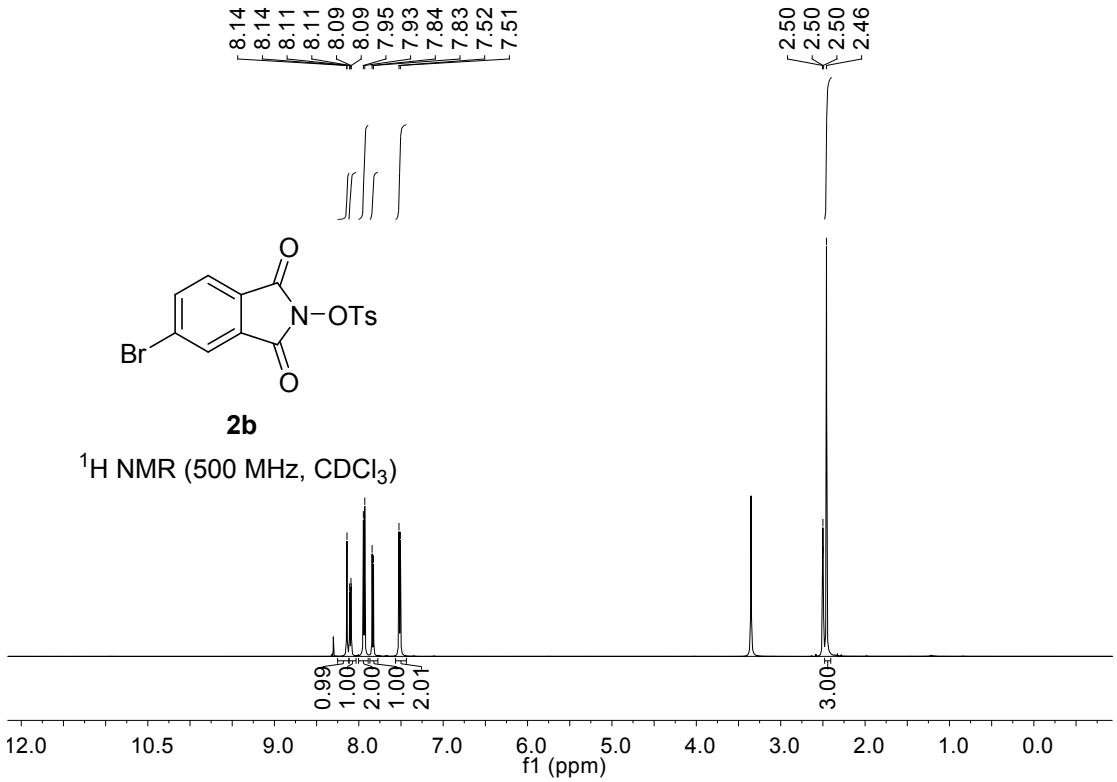
5

^{1}H NMR (500 MHz, $CDCl_3$)



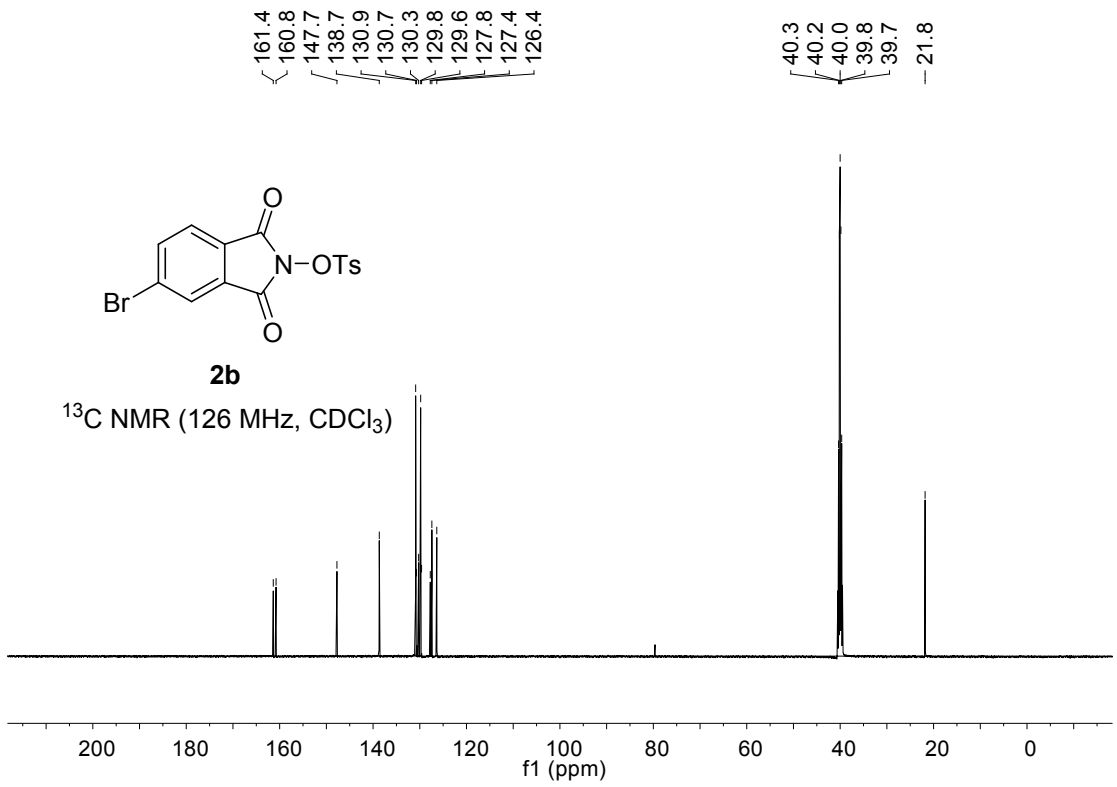
5

^{13}C NMR (126 MHz, $CDCl_3$)



2b

^{1}H NMR (500 MHz, $CDCl_3$)



2b

^{13}C NMR (126 MHz, $CDCl_3$)

5. X-ray Structure of **3u** with 50% ellipsoid probability