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Supporting Information

Radical Aminophosphinoylation of Maleimides with Anilines and Diarylphosphine Oxides

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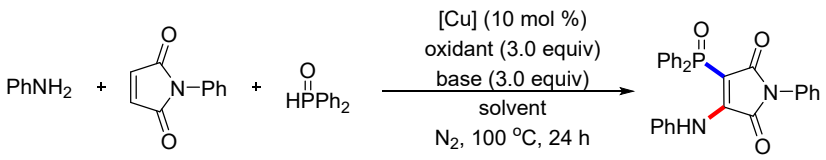
General Information

Maleimides[1] were prepared according to the reported procedures. ^{1}H and ^{13}C spectra of known compounds were in accordance with those described in the literatures. All other reagents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros, and Meryer and used without further purification. ^{1}H NMR (500 MHz), ^{13}C NMR (125 MHz) and ^{19}F NMR (470 MHz) spectra were recorded in $CDCl_3$ and DMSO-D6 solutions using a Burker AVANCE 500 spectrometer. High-resolution mass spectra were recorded on an ESI-Q-TOF mass spectrometer. Analysis of crude reaction mixture was done on the Varian 4000 GC/MS and 1200 LC. All reactions were conducted using standard Schlenk techniques. Column chromatography was performed using EM silica gel 60 (300−400 m).

Table 1. Reaction Optimization[a]



1a **2a** **3a** **4a**

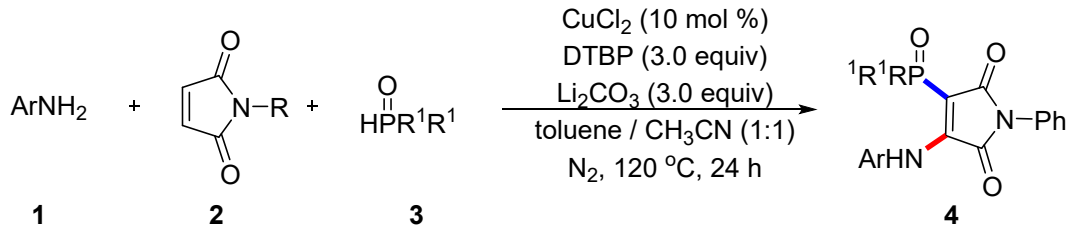
entry	catalyst	solvent	oxidant	base	yield (%)[b]
1	CuCl	MeCN	DTBP		11
2	CuI	MeCN	DTBP		7
3	$CuCl_2$	MeCN	DTBP		9
4	$Cu(OAc)_2$	MeCN	DTBP		0
5	CuCl	MeCN	70% TBHP		0
6	CuCl	MeCN	$K_2S_2O_8$		10
7	CuCl	MeCN	$Mn(OAc)_3$		5
8	CuCl	MeCN	TBPB		0
9	CuCl	MeCN	DTBP	Li_2CO_3	49
10	CuCl	MeCN	DTBP	Na_2CO_3	0
11	CuCl	MeCN	DTBP	K_2CO_3	0
12	CuCl	MeCN	DTBP	Cs_2CO_3	0
13	CuCl	toluene	DTBP	Li_2CO_3	33
14	CuCl	THF	DTBP	Li_2CO_3	0
15	CuCl	dioxane	DTBP	Li_2CO_3	0
16	CuCl	MeCN/PhMe = 1:1	DTBP	Li_2CO_3	61
17	**$CuCl_2$**	**MeCN/PhMe = 1:1**	**DTBP**	**Li_2CO_3**	**75**
18[c]	$CuCl_2$	MeCN/PhMe = 1:1	DTBP	Li_2CO_3	45
19[d]	$CuCl_2$	MeCN/PhMe = 1:1	DTBP	Li_2CO_3	70
20		MeCN/PhMe = 1:1	DTBP	Li_2CO_3	0

[a] Standard reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.4 mmol), copper salt (0.02 mmol), oxidant (0.6 mmol) and base (0.6 mmol) in solvent (2.0 mL) under N_2, heated at 100 °C for 24 h. [b] Isolated yield. [c]10% 1,10-phen as ligand. [d]Under O_2 atmosphere.

We used cheap and readily available aniline **1a**, *N*-phenyl maleimide **2a**, diphenylphosphine oxide **3a** as a model substrate, and performed the radical multi-component aminophosphinoylation to examine our assumptions (Table 1). According to our previous experience of maleimide chemistry, we focused on the cheap copper catalysts. After simple optimization of reaction conditions, we found that using DTBP as a free radical oxidant and CuCl as a catalyst, the desired product **4a** was obtained with a yield of 11% (entry 1). A variety of copper salts were further screened, and the result showed that only copper halide could promote the reaction smoothly, while other copper catalysts had no catalytic ability (entries 1-4). Further evaluation of oxidants, such as 70% TBHP, $K_2S_2O_8$, $Mn(OAc)_3$ and TBPB displayed relatively lower yields than DTBP (entries 5-8). Some *P*-Michael addition of maleimide unwished byproduct was formed during the reaction optimization. In other words, aniline did not rapidly capture the intermediate of carbon free radical, thus reducing the conversion efficiency of raw materials. Therefore, the organic and inorganic bases commonly used in the laboratory were screened (entries 9-12), and Li_2CO_3 as a base really improve the yield of the target product and the reaction became relatively clean. It was found that the choice of reaction solvent is very important for the access of the target product. Using weak coordination ethers (entries 14, 15), where there is no product, a mix of CH_3CN / toluene increased the yield to 61% (entry 16). Subsequently, we re-screened the effect of a series of useful copper salts in the early stage and found that $CuCl_2$ showed the best catalytic performance (entry 17). Next, some additives or ligands were added into the catalytic system in an attempt to improve the yield of the target product. Surprisingly, the introduction of a bidentate ligand (such as 1, 10-phen) led to a sharp drop in yield (entry 18), which may be due to the ligand occupied the coordination position of the copper ion center and reducing its Lewis acidity. In addition, the current radical cascade reaction is insensitive to the reaction atmosphere, and a considerable yield can still be obtained under oxygen conditions (entry 19). A control experiment suggested that the loss of copper catalyst led to failure of the transformation (entry 20).

General Experimental Procedures

General Procedure of Radical Aminophosphinoylation of Maleimides with Anilines and Diarylphosphine Oxides:

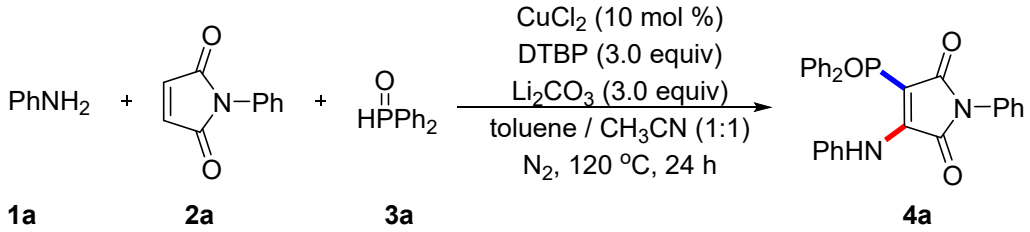


1 **2** **3** **4**

A 25 mL Schlenk tube equipped with a stir bar was charged with arylamine (0.3 mmol), maleimide (0.2 mmol), diarylphosphine oxides (0.4 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 ℃ for 24 h. After cooling down, the reaction mixture was diluted with 10 mL of ethyl ether, filtered through a pad of silica gel, followed by washing the pad of the silica gel with the same solvent (20

mL), concentrated under reduced pressure. The residue was then purified by flash chromatography on silica gel to provide the corresponding product.

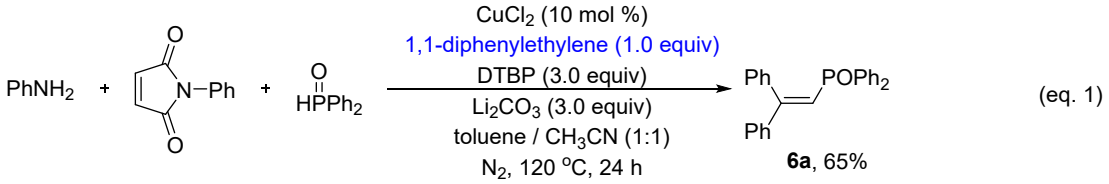
5mmol Scale Reaction Synthesis:



1a **2a** **3a** **4a**

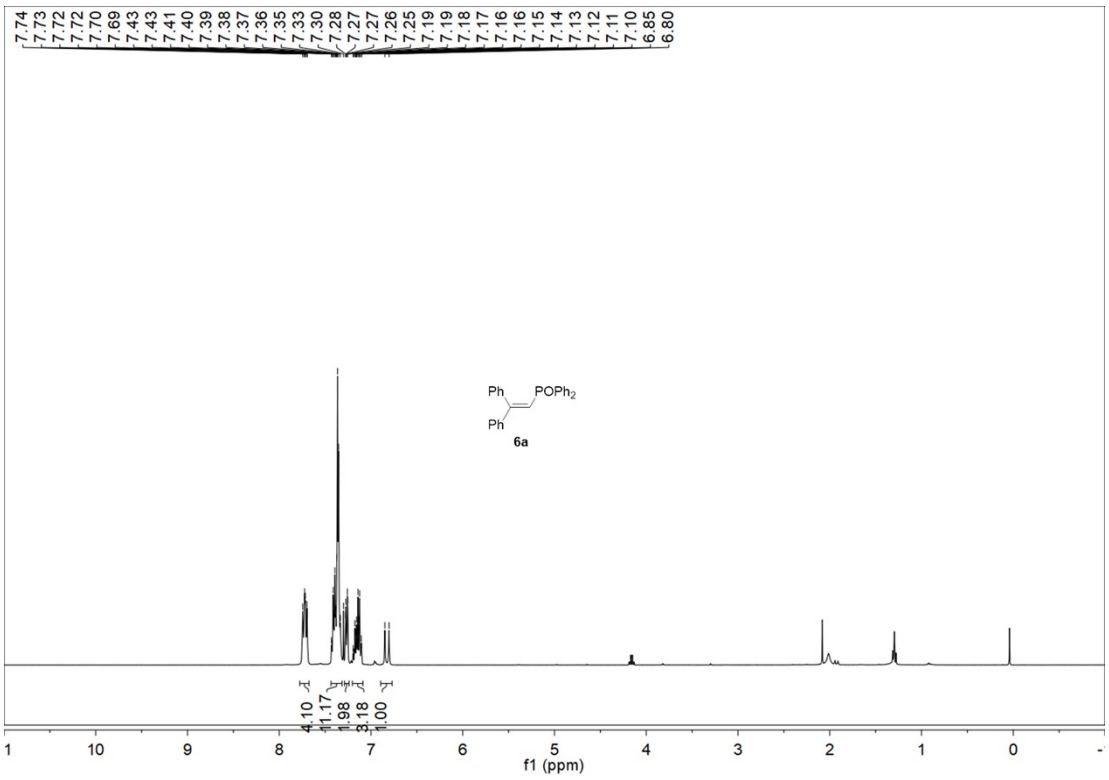
A 125 mL Schlenk tube equipped with a stir bar was charged with aniline (7.5 mmol), *N*-Phenylmaleimide (5.0 mmol), diphenylphosphine oxide (10.0 mmol), $CuCl_2$ (0.50 mmol), Li_2CO_3 (15.0 mmol), 50 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (15.0 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 ℃ for 24 h. Finally, the crude product was purified by flash chromatography on silica gel (petroleum ether : EtOAc = 9 : 1) directly to give the desired product **4a** (1624.4 mg) in 70% yield as a yellow liquid.

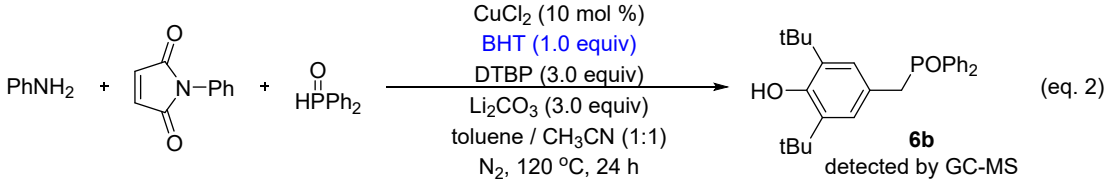
Mechanistic Studies



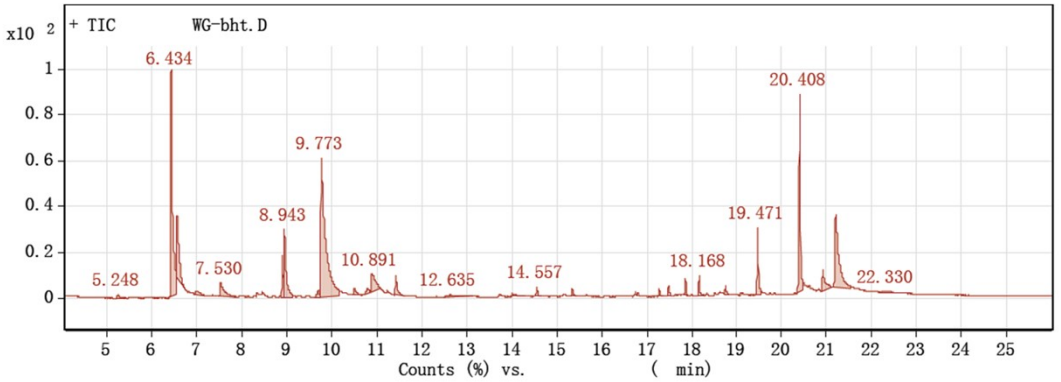
(eq. 1)

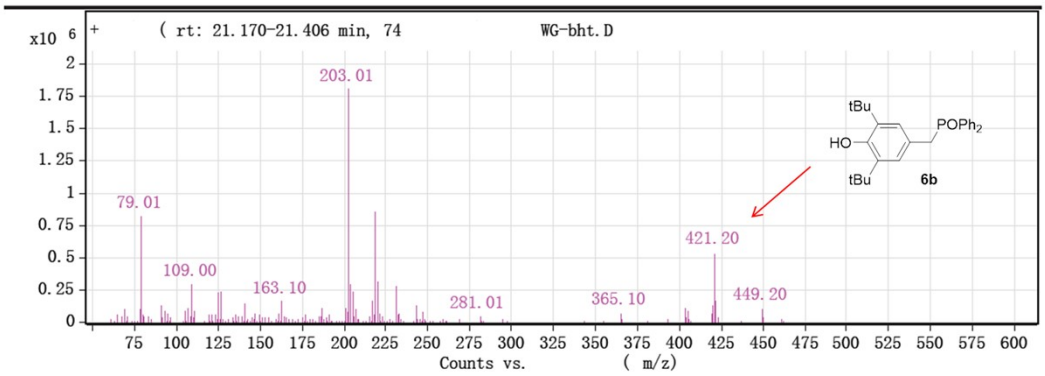
A 125 mL Schlenk tube equipped with a stir bar was charged with aniline (0.3 mmol), *N*-Phenylmaleimide (0.2 mmol), diphenylphosphine oxide (0.4 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), 1,1-diphenylethylene (0.2 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the radical phosphinoylation product **6a** was isolated in 65%.

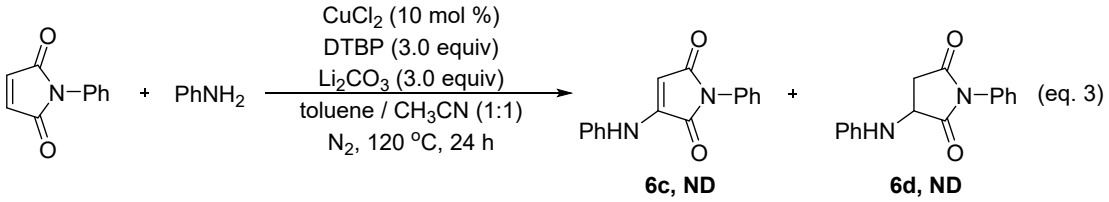




A 125 mL Schlenk tube equipped with a stir bar was charged with aniline (0.3 mmol), *N*-Phenylmaleimide (0.2 mmol), diphenylphosphine oxide (0.4 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), BHT (0.2 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 ℃. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the radical trapped product **6b** was detected by GC-MS.

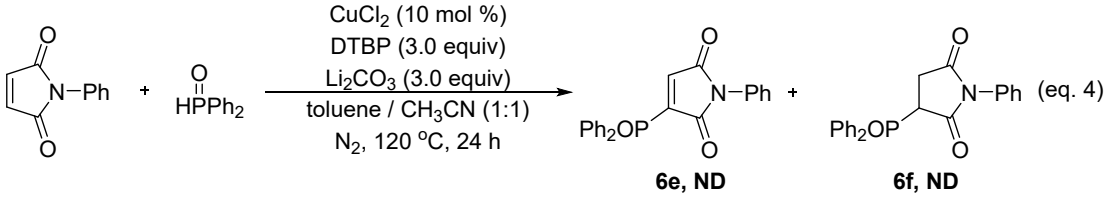






6c, ND **6d, ND**

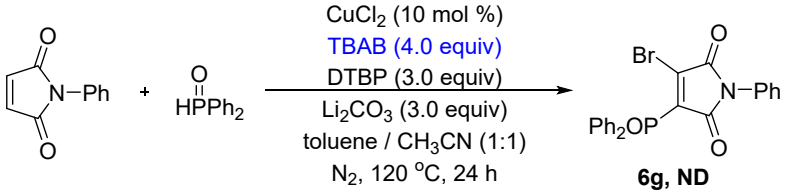
A 125 mL Schlenk tube equipped with a stir bar was charged with aniline (0.3 mmol), *N*-Phenylmaleimide (0.2 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), 1,1-diphenylethylene (0.2 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 °C. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the oxidative amination **6c** and *N*-Michael addition **6d** products did not detected by GC-MS and HRMS.



6e, ND **6f, ND**

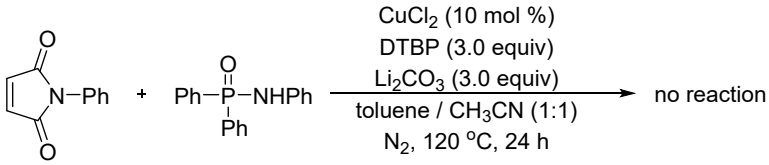
A 125 mL Schlenk tube equipped with a stir bar was charged with *N*-Phenylmaleimide (0.2 mmol), diphenylphosphine oxide (0.4 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), 1,1-diphenylethylene (0.2 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2

three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 °C. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the oxidative phosphinoylation **6e** and *P*-Michael addition **6f** products did not detected by GC-MS and HRMS.



6g, ND (eq. 5)

A 125 mL Schlenk tube equipped with a stir bar was charged with *N*-Phenylmaleimide (0.2 mmol), diphenylphosphine oxide (0.4 mmol), TBAB (0.8 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), 1,1-diphenylethylene (0.2 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 °C. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), the oxidative phosphinobromination **6g** product did not detected by GC-MS and HRMS.

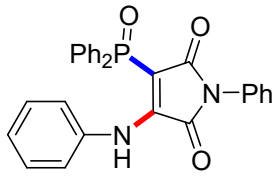


(eq. 6)

A 125 mL Schlenk tube equipped with a stir bar was charged with *N*-Phenylmaleimide (0.2 mmol), N,P,P-triphenylphosphinic amide (0.4 mmol), $CuCl_2$ (0.02 mmol), Li_2CO_3 (0.6 mmol), 1,1-diphenylethylene (0.2 mmol), 2 mL mixture solvent (toluene / CH_3CN = 1:1) and DTBP (0.6 mmol). The tube was fitted with a rubber septum, and then it was evacuated and refilled with N_2 three times, then the septum was replaced by a Teflon screwcap under N_2 flow. The reaction mixture was stirred at 120 °C. After stirring for 24 h, the reaction mixture was cooled to room temperature and the reaction was filtered through a pad of Celite and diluted with ethyl acetate (10 mL), no reaction was observed on TLC, except with a lot of raw starting materials.

Characterization of Products in Details :

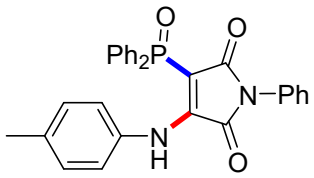
3-(diphenylphosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione



4a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (69.6 mg, 75% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.97 (s, 1H), 8.01 (t, J = 8.8 Hz, 4H), 7.63 (t, J = 7.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 4H), 7.44-7.30 (m, 10H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.54 (d, J = 12.0 Hz), 162.90 (d, J = 12.7 Hz), 155.98 (d, J = 6.1 Hz), 136.79, 132.54 (d, J = 110 Hz), 132.52 (d, J = 2.8 Hz), 131.56 (d, J = 11.0 Hz), 131.38, 128.95, 128.74 (d, J = 12.9 Hz), 127.79, 127.34, 126.19, 125.04, 113.98, 88.20 (d, J = 117.7 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.38; **HRMS** (ESI): calcd for $C_{28}H_{22}N_2O_3P$ $[M + H]^+$ 465.1368, found 465.1368.

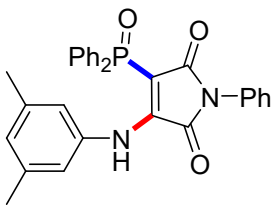
3-(diphenylphosphoryl)-1-phenyl-4-(p-tolylamino)-1H-pyrrole-2,5-dione



4b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (70.7 mg, 74% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.90 (s, 1H), 8.00 (dd, J = 13.1, 7.2 Hz, 4H), 7.64-7.60 (m, 2H), 7.58-7.53 (m, 4H), 7.44-7.40 (m, 2H), 7.36-7.32 (m, 3H), 7.26-7.19 (m, 4H), 2.39 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.52 (d, J = 12.5 Hz), 162.84 (d, *J* = 14.2 Hz), 156.08, 137.34, 134.17, 132.58 (d, J = 110 Hz), 132.42 (d, J = 2.9 Hz), 131.52 (d, J = 11.4 Hz), 131.38, 129.53, 128.87, 128.67 (d, J = 12.9 Hz), 127.68, 126.12, 124.93, 87.45 (d, J = 118.4 Hz), 21.18; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.42; **HRMS** (ESI): calcd for $C_{29}H_{24}N_2O_3P$ $[M + H]^+$ 479.1525, found 479.1523.

3-((3,5-dimethylphenyl)amino)-4-(diphenylphosphoryl)-1-phenyl-1H-pyrrole-2,5-dione

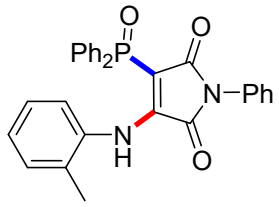


4c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow liquid (75.8 mg, 77% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.92 (s, 1H), 7.99 (dd, J = 13.1, 7.2 Hz, 4H), 7.64-7.59 (m, 2H), 7.57-7.53 (m, 4H), 7.45-7.41 (m, 2H), 7.37-7.34 (m, 3H), 7.00 (s, 2H), 6.95 (s, 1H), 2.34 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.54 (d, *J* = 12.3 Hz), 162.81 (d, *J* = 13.2 Hz), 156.20, 138.66, 136.58, 132.62 (d, J = 110 Hz), 132.41 (d, J = 2.9 Hz), 131.53 (d, J = 11.4 Hz), 131.43, 129.09, 128.92, 128.67 (d, J = 12.9 Hz), 127.72, 126.20, 122.64, 87.55 (d, J = 118.3 Hz), 21.28; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.29; **HRMS** (ESI): calcd for $C_{30}H_{26}N_2O_3P$ $[M + H]^+$ 493.1681, found 493.1680.

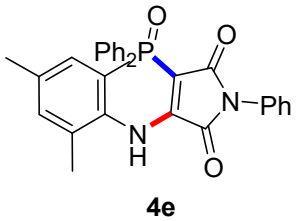
3-(diphenylphosphoryl)-1-phenyl-4-(o-tolylamino)-1H-pyrrole-2,5-dione



4d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (66.9 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.85 (s, 1H), 8.05-7.99 (m, 4H), 7.66-7.54 (m, 6H), 7.44-7.21 (m, 9H), 2.44 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.55 (d, *J* = 12.3 Hz), 162.86 (d, *J* = 12.9 Hz), 156.51 (d, *J* = 5.9 Hz), 135.76, 133.40, 132.60 (d, J = 100 Hz), 132.47 (d, *J* = 2.9 Hz), 131.51 (d, *J* = 11.3 Hz), 131.38, 130.74, 128.88, 128.72 (d, *J* = 13.0 Hz), 127.83, 127.68, 126.40, 126.35, 126.07, 87.35 (d, *J* = 118.4 Hz), 18.20; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.42; **HRMS** (ESI): calcd for $C_{29}H_{24}N_2O_3P$ $[M + H]^+$ 479.1525, found 479.1522.

3-(diphenylphosphoryl)-4-(mesitylamino)-1-phenyl-1H-pyrrole-2,5-dione

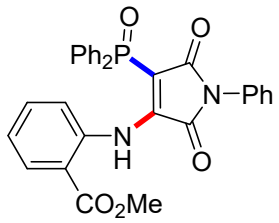


4e

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (53.6 mg, 53% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.46 (s, 1H), 8.04-7.98 (m,

4H), 7.65-7.52 (m, 7H), 7.41-7.33 (m, 3H), 7.31-7.27 (m, 1H), 6.95 (s, 2H), 2.33-2.31 (m, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.65 (d, *J* = 12.7 Hz), 162.75 (d, J = 12.9 Hz), 157.77 (d, J = 3.7 Hz), 149.27, 137.69, 134.70, 132.77 (d, J = 109.0 Hz), 132.70, 132.41 (d, J = 2.9 Hz), 131.49 (d, J = 11.3 Hz), 131.38, 130.92, 128.87 (d, J = 47.0 Hz), 128.77, 127.51, 125.87, 120.90, 85.84 (d, J = 119.2 Hz), 21.14, 18.49, 17.45; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.42; **HRMS** (ESI): calcd for $C_{31}H_{28}N_2O_3P$ $[M + H]^+$ 507.1838, found 507.1837.

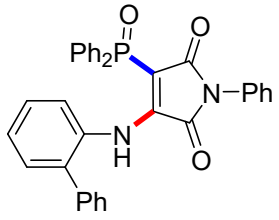
methyl 2-((4-(diphenylphosphoryl)-2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)amino)benzoate



4f

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (53.3 mg, 51% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.41 (s, 1H), 8.07-8.00 (m, 5H), 7.63-7.59 (m, 2H), 7.57-7.50 (m, 5H), 7.44-7.40 (m, 3H), 7.37-7.32 (m, 4H), 3.96 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.60 (d, *J* = 11.8 Hz), 166.32, 163.33 (d, *J* = 12.4 Hz), 155.37, 137.62, 132.68, 132.57 (d, *J* = 109.1 Hz), 132.38 (d, *J* = 2.9 Hz), 131.65 (d, *J* = 11.1 Hz), 131.33, 128.93, 128.63 (d, *J* = 13.0 Hz), 128.36, 127.75, 126.99, 126.72, 126.13, 123.79, 90.47 (d, *J* = 116.7 Hz), 52.57; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 27.42; **HRMS** (ESI): calcd for $C_{30}H_{24}N_2O_5P$ $[M + H]^+$ 523.1423, found 523.1423.

3-([1,1'-biphenyl]-2-ylamino)-4-(diphenylphosphoryl)-1-phenyl-1H-pyrrole-2,5-dione

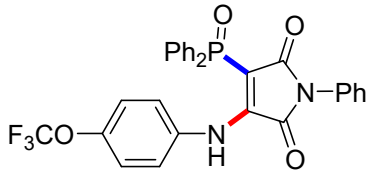


4g

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow liquid (59.4 mg, 55% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.67 (s, 1H), 7.93-7.87 (m, 4H), 7.65-7.60 (m, 2H), 7.57-7.52 (m, 4H), 7.46-7.36 (m, 8H), 7.31-7.25 (m, 2H), 7.21-7.14 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.36 (d, *J* = 12.3 Hz), 162.81 (d, *J* = 12.8 Hz), 157.21, 138.91, 138.37, 135.02, 132.51 (d, *J* = 109.1 Hz), 132.40 (d, *J* = 2.9 Hz), 131.55 (d, *J* = 11.3 Hz), 131.21, 130.77, 130.05, 128.73 (d, *J* = 13.5 Hz), 128.54, 128.50, 128.31, 128.12, 127.61, 127.33, 126.58, 126.04, 87.73 (d, *J* = 118.4 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 28.94; **HRMS** (ESI): calcd for $C_{34}H_{26}N_2O_3P$ $[M + H]^+$ 541.1681, found 541.1678.

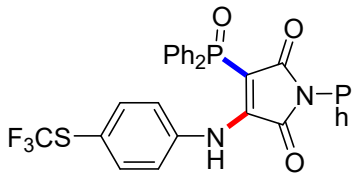
3-(diphenylphosphoryl)-1-phenyl-4-((4-(trifluoromethoxy)phenyl)amino)-1H-pyrrole-2,5-dione



4h

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (64.7 mg, 59% yield), Mp = 197-198℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.96 (s, 1H), 8.03-7.98 (m, 4H), 7.65-7.61 (m, 2H), 7.59-7.54 (m, 4H), 7.46-7.33 (m, 7H), 7.25-7.22 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.37 (d, *J* = 12.1 Hz), 162.98 (d, *J* = 13.0 Hz), 155.71, 147.82, 135.29, 132.62 (d, *J* = 2.9 Hz), 132.23 (d, *J* = 110.1 Hz), 131.53 (d, *J* = 11.2 Hz), 131.20, 129.02, 128.77 (d, *J* = 13.0 Hz), 127.94, 126.42, 126.16, 121.35, 120.48 (q, J = 257.7 Hz), 89.23 (d, *J* = 116.6 Hz); **^{19}F NMR** (375 MHz, $CDCl_3$): δ -57.86 (s, 3F); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.45; **HRMS** (ESI): calcd for $C_{29}H_{21}N_2O_4F_3P$ $[M + H]^+$ 549.1191, found 549.1188.

3-(diphenylphosphoryl)-1-phenyl-4-((4-((trifluoromethyl)thio)phenyl)amino)-1H-pyrrole-2,5-dione

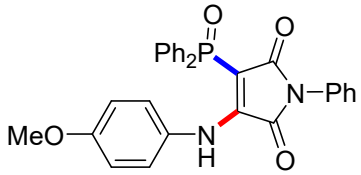


4i

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow solid (68.8 mg, 61% yield), Mp = 165-166℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.03 (s, 3H), 7.95-7.90 (m, 4H), 7.61-7.55 (m, 4H), 7.52-7.48 (m, 4H), 7.40-7.36 (m, 4H), 7.31-7.27 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.24 (d, *J* = 11.6 Hz), 162.96 (d, *J* = 12.7 Hz), 155.36, 139.30, 136.91, 132.70 (d, *J* = 2.9 Hz), 131.97 (d, *J* = 110.1 Hz), 131.55 (d, *J* = 11.4 Hz), 131.13, 131.01, 129.05, 128.79 (d, *J* = 13.2 Hz), 128.01, 126.17, 125.29, 120.79 (q, J = 324.8 Hz), 90.72 (d, *J* = 115.1 Hz); **^{19}F NMR** (375 MHz, $CDCl_3$): δ -42.68 (s, 3F); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.41; **HRMS** (ESI): calcd for $C_{29}H_{21}N_2O_3F_3PS$ $[M + H]^+$ 565.0963, found 565.0961.

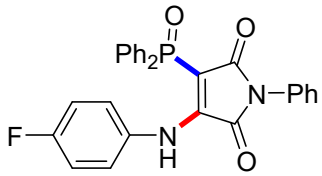
3-(diphenylphosphoryl)-4-((4-methoxyphenyl)amino)-1-phenyl-1H-pyrrole-2,5-dione



4j

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (70.2 mg, 71% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.87 (s, 1H), 8.04-7.99 (m, 4H), 7.64-7.54 (m, 6H), 7.44-7.40 (m, 2H), 7.37-7.29 (m, 5H), 6.92 (d, J = 8.5 Hz, 2H), 3.84 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.56 (d, *J* = 12.4 Hz), 162.89 (d, *J* = 12.9 Hz), 158.79, 156.16, 132.68 (d, J = 109.1 Hz), 132.48, 131.54 (d, *J* = 11.2 Hz), 131.42, 129.64, 128.92, 128.72 (d, *J* = 12.9 Hz), 127.73, 126.52, 126.18, 114.13, 86.97 (d, *J* = 118.6 Hz), 55.53; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.46; **HRMS** (ESI): calcd for $C_{29}H_{24}N_2O_4P$ $[M + H]^+$ 495.1474, found 495.1470.

3-(diphenylphosphoryl)-4-((4-fluorophenyl)amino)-1-phenyl-1H-pyrrole-2,5-dione

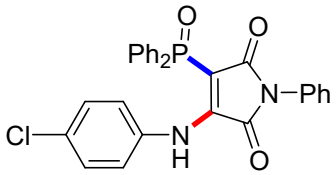


4k

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (58.8 mg, 61% yield), Mp = 170-171℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.87 (s, 1H), 8.01-7.96 (m, 4H), 7.64-7.53 (m, 6H), 7.44-7.40 (m, 2H), 7.34-7.29 (m, 5H), 7.10-7.06 (m,

2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.44 (d, *J* = 12.1 Hz), 162.90 (d, *J* = 14.7 Hz), 161.59 (d, J = 247.2 Hz), 155.99, 132.78, 132.54 (d, *J* = 3.3 Hz), 132.39 (d, *J* = 109.1 Hz), 131.51 (d, *J* = 11.3 Hz), 131.24, 128.96, 128.72 (d, *J* = 13.0 Hz), 127.83, 127.05 (d, *J* = 8.5 Hz), 126.12, 115.84 (d, *J* = 23.0 Hz), 88.20 (d, *J* = 117.2 Hz); **^{19}F NMR** (375 MHz, $CDCl_3$): δ -114.04 (s, 1F); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.44; **HRMS** (ESI): calcd for $C_{28}H_{21}N_2O_3FP$ $[M + H]^+$ 483.1274, found 483.1271.

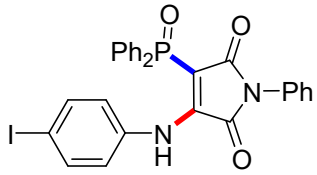
3-((4-chlorophenyl)amino)-4-(diphenylphosphoryl)-1-phenyl-1H-pyrrole-2,5-dione



4l

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (68.7 mg, 69% yield), Mp = 199-200℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.95 (s, 1H), 8.04-7.98 (m, 4H), 7.66-7.55 (m, 6H), 7.46-7.42 (m, 2H), 7.38-7.31 (m, 7H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.40 (d, *J* = 11.7 Hz), 162.94 (d, *J* = 12.9 Hz), 155.71, 135.35, 132.89, 132.61 (d, *J* = 2.9 Hz), 132.28 (d, *J* = 109.1 Hz), 131.54 (d, *J* = 11.3 Hz), 131.23, 129.07, 129.00, 128.77 (d, *J* = 13.0 Hz), 127.89, 126.35, 126.13, 88.95 (d, *J* = 116.5 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.43; **HRMS** (ESI): calcd for $C_{28}H_{21}N_2O_3PCl$ $[M + H]^+$ 499.0978, found 499.0975.

3-(diphenylphosphoryl)-4-((4-iodophenyl)amino)-1-phenyl-1H-pyrrole-2,5-dione

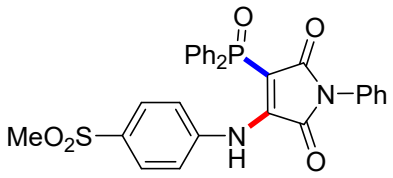


4m

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (87.3 mg, 74% yield), Mp = 192-193℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.93 (s, 1H), 8.01-7.96 (m, 4H), 7.70 (d, J = 8.5 Hz, 2H), 7.65-7.61 (m, 2H), 7.58-7.53 (m, 4H), 7.45-7.41 (m, 2H), 7.35-7.32 (m, 3H), 7.13 (d, J = 8.5 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.35 (d, *J*

= 11.9 Hz), 162.91 (d, *J* = 12.8 Hz), 155.53, 137.98, 136.55, 132.59 (d, *J* = 2.9 Hz), 132.22 (d, *J* = 109.1 Hz), 131.52 (d, *J* = 11.2 Hz), 131.21, 128.98, 128.74 (d, *J* = 13.0 Hz), 127.88, 126.73, 126.11, 91.93, 89.24 (d, *J* = 116.5 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.40; **HRMS** (ESI): calcd for $C_{28}H_{21}N_2O_3PI$ $[M + H]^+$ 591.0334, found 591.0334.

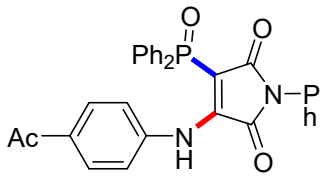
3-(diphenylphosphoryl)-4-((4-(methylsulfonyl)phenyl)amino)-1-phenyl-1H-pyrrole-2,5-dione



4n

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (78.1 mg, 72% yield), Mp = 137-138℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.18 (s, 1H), 8.00-7.86 (m, 6H), 7.65-7.51 (m, 8H), 7.46-7.40 (m, 2H), 7.37-7.29 (m, 3H), 3.05 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.11 (d, *J* = 12.2 Hz), 163.04 (d, *J* = 12.0 Hz), 155.09, 141.69, 137.99, 132.81, 131.71 (d, *J* = 110.1 Hz), 131.57 (d, *J* = 11.5 Hz), 131.04, 129.09, 128.83 (d, *J* = 12.9 Hz), 128.36, 128.10, 126.16, 124.67, 92.27 (d, *J* = 114.3 Hz), 44.65; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.39; **HRMS** (ESI): calcd for $C_{29}H_{24}N_2O_5PS$ $[M + H]^+$ 543.1144, found 543.1143.

3-((4-acetylphenyl)amino)-4-(diphenylphosphoryl)-1-phenyl-1H-pyrrole-2,5-dione

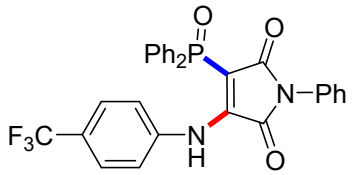


4o

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (77.9 mg, 77% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.16 (s, 1H), 8.03-7.97 (m, 6H), 7.65-7.61 (m, 2H), 7.59-7.54 (m, 4H), 7.48-7.42 (m, 4H), 7.37-7.32 (m, 3H), 2.62 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 196.87, 168.26 (d, *J* = 11.7 Hz), 162.95 (d, *J* = 12.8 Hz), 155.35, 140.96, 135.14, 132.69 (d, *J* = 2.9 Hz), 132.0 (d, *J* = 110.1 Hz), 131.56 (d, *J* = 11.3 Hz), 131.16,

129.24, 129.02, 128.79 (d, J = 13.1 Hz), 127.96, 126.14, 124.22, 90.93 (d, J = 115.2 Hz), 26.66. **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.36; **HRMS** (ESI): calcd for $C_{30}H_{24}N_2O_4P$ $[M + H]^+$ 507.1474, found 507.1475.

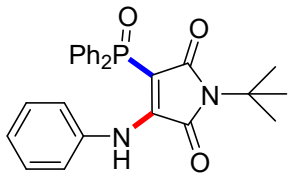
3-(diphenylphosphoryl)-1-phenyl-4-((4-(trifluoromethyl)phenyl)amino)-1H-pyrrole-2,5-dione



4p

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (74.5 mg, 70% yield), Mp = 210-211℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.11 (s, 1H), 8.02-7.97 (m, 4H), 7.66-7.61 (m, 4H), 7.59-7.54 (m, 4H), 7.50-7.48 (m, 2H), 7.46-7.43 (m, 2H), 7.37-7.34 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.26 (d, J = 12.3 Hz), 162.97 (d, J = 12.5 Hz), 155.46, 139.87, 132.69 (d, J = 2.9 Hz), 131.84 (d, J = 144.2 Hz), 131.54 (d, J = 11.4 Hz), 129.03, 128.79 (d, J = 13.0 Hz), 128.67, 127.99, 126.12, 126.08, 126.04, 124.78, 123.91 (q, J = 270.36 Hz), 90.65 (d, J = 115.3 Hz); **^{19}F NMR** (375 MHz, $CDCl_3$): δ -62.36 (s, 3F); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.35; **HRMS** (ESI): calcd for $C_{29}H_{21}N_2O_3F_3P$ $[M + H]^+$ 533.1242, found 533.1241.

1-(tert-butyl)-3-(diphenylphosphoryl)-4-(phenylamino)-1H-pyrrole-2,5-dione

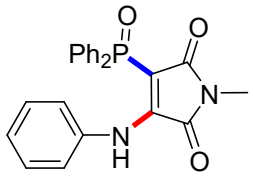


4q

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (63.9 mg, 72% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.55 (s, 1H), 7.96-7.90 (m, 4H), 7.63-7.52 (m, 6H), 7.40-7.36 (m, 2H), 7.31-7.28 (m, 3H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz,

$CDCl_3$): δ 171.03 (d, J = 12.4 Hz), 164.91 (d, J = 13.6 Hz), 155.77, 137.31, 132.85 (d, J = 109.1 Hz), 132.27 (d, J = 2.9 Hz), 131.52 (d, J = 11.2 Hz), 128.83, 128.58 (d, J = 12.9 Hz), 126.84, 124.86, 88.80 (d, J = 119.6 Hz), 57.89, 29.08; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.25; **HRMS** (ESI): calcd for $C_{26}H_{26}N_2O_3P$ $[M + H]^+$ 445.1681, found 445.1682.

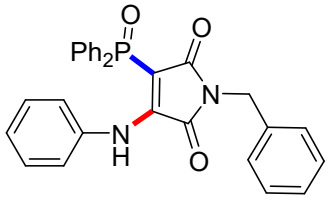
3-(diphenylphosphoryl)-1-methyl-4-(phenylamino)-1H-pyrrole-2,5-dione



4r

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (49.1 mg, 61% yield), Mp = 137-138℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.80 (s, 1H), 7.97-7.92 (m, 4H), 7.63-7.52 (m, 6H), 7.42-7.38 (m, 2H), 7.42-7.32 (m, 3H), 2.97 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.72 (d, J = 12.1 Hz), 164.14 (d, J = 13.0 Hz), 156.57, 136.88, 132.68 (d, J = 109.1 Hz), 132.41 (d, J = 3.3 Hz), 131.48 (d, J = 11.3 Hz), 128.88, 128.64 (d, J = 12.9 Hz), 127.18, 124.86, 88.01 (d, J = 119.1 Hz), 23.91. **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.25; **HRMS** (ESI): calcd for $C_{23}H_{20}N_2O_3P$ $[M + H]^+$ 403.1212, found 403.1210.

1-benzyl-3-(diphenylphosphoryl)-4-(phenylamino)-1H-pyrrole-2,5-dione

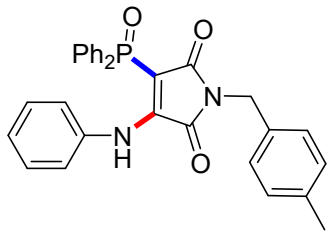


4s

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (66.9 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.84 (s, 1H), 7.96-7.90 (m, 4H), 7.63-7.51 (m, 6H), 7.41-7.26 (m, 10H), 4.63 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.32 (d, *J* = 11.8 Hz), 163.77 (d, *J* = 12.7 Hz), 156.42 (d, *J* = 4.7 Hz), 136.76, 136.33, 132.64 (d, *J* = 109.1 Hz), 132.41 (d, *J* = 2.9 Hz), 131.47 (d, *J* = 11.4 Hz), 128.73 (d, J = 12.9 Hz), 128.71, 127.85,

127.19, 124.84, 87.97 (d, J = 118.5 Hz), 41.67; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.29; **HRMS** (ESI): calcd for $C_{29}H_{24}N_2O_3P$ $[M + H]^+$ 479.1525, found 479.1524.

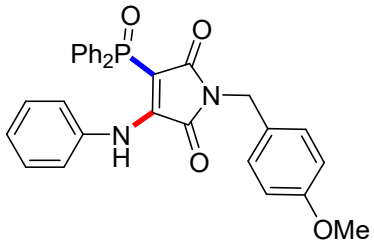
3-(diphenylphosphoryl)-1-(4-methylbenzyl)-4-(phenylamino)-1H-pyrrole-2,5-dione



4t

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (70.9 mg, 72% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.83 (s, 1H), 7.97-7.91 (m, 4H), 7.61 (td, J = 7.2, 1.6 Hz, 2H), 7.57-7.52 (m, 4H), 7.41-7.37 (m, 2H), 7.33-7.30 (m, 3H), 7.26 (d, J = 7.8 Hz, 2H), 7.14 (d, J = 7.8 Hz, 2H), 4.60 (s, 2H), 2.35 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.38 (d, J = 12.0 Hz), 163.79 (d, J = 12.9 Hz), 156.47, 137.61, 136.81, 133.41, 132.69 (d, J = 109.1 Hz), 132.41 (d, J = 2.9 Hz), 131.49 (d, J = 11.2 Hz), 129.39, 128.88, 128.73 (d, J = 2.9 Hz), 128.59, 127.17, 124.85, 87.96 (d, J = 118.6 Hz), 41.42, 21.21; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.32; **HRMS** (ESI): calcd for $C_{30}H_{26}N_2O_3P$ $[M + H]^+$ 493.1681, found 493.1682.

3-(diphenylphosphoryl)-1-(4-methoxybenzyl)-4-(phenylamino)-1H-pyrrole-2,5-dione

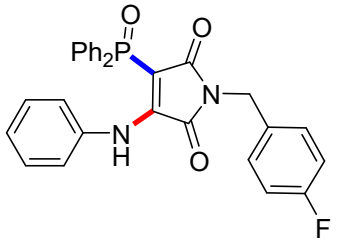


4u

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (72.1 mg, 71% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.84 (s, 1H), 7.97-7.91 (m, 4H), 7.63-7.59 (m, 2H), 7.57-7.52 (m, 4H), 7.42-7.38 (m, 2H), 7.33-7.29 (m, 5H), 6.87-6.84 (m, 2H), 4.58 (s, 2H), 3.81 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.40 (d, J = 12.0 Hz), 163.79 (d, J = 12.9 Hz), 159.26, 156.50 (d, J = 4.0 Hz), 136.81, 132.69 (d, J = 109.1 Hz), 132.41 (d, J = 2.9

Hz), 131.49 (d, J = 11.3 Hz), 130.24, 128.88, 128.65 (d, J = 13.0 Hz), 127.18, 124.85, 114.04, 87.93 (d, J = 118.6 Hz), 55.33, 41.13. **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.34; **HRMS** (ESI): calcd for $C_{30}H_{26}N_2O_4P$ $[M + H]^+$ 509.1630, found 509.1629.

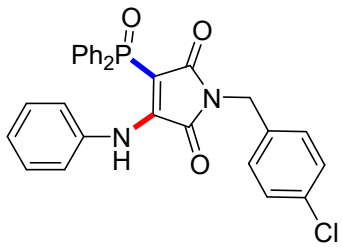
3-(diphenylphosphoryl)-1-(4-fluorobenzyl)-4-(phenylamino)-1H-pyrrole-2,5-dione



4v

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (75.4 mg, 76% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.87 (s, 1H), 7.96-7.90 (m, 4H), 7.64-7.59 (m, 2H), 7.57-7.52 (m, 4H), 7.42-7.38 (m, 2H), 7.35-7.30 (m, 5H), 7.00 (t, *J* = 8.6 Hz, 2H), 4.59 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.25 (d, *J* = 12.0 Hz), 163.73 (d, *J* = 14.3 Hz), 162.43 (d, *J* = 247.3 Hz), 156.42, 136.74, 132.62 (d, *J* = 109.1 Hz), 132.45 (d, *J* = 2.9 Hz), 132.17 (d, *J* = 3.4 Hz), 131.90 (d, *J* = 10.1 Hz), 131.47 (d, *J* = 11.3 Hz), 130.64 (d, *J* = 8.2 Hz), 128.91, 128.67 (d, *J* = 12.9 Hz), 127.26, 124.86, 87.98 (d, *J* = 118.2 Hz), 40.95; **^{19}F NMR** (375 MHz, $CDCl_3$): δ -114.19 (s, 1F); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.24; **HRMS** (ESI): calcd for $C_{29}H_{23}N_2O_3FP$ $[M + H]^+$ 497.1430, found 497.1429.

1-(4-chlorobenzyl)-3-(diphenylphosphoryl)-4-(phenylamino)-1H-pyrrole-2,5-dione

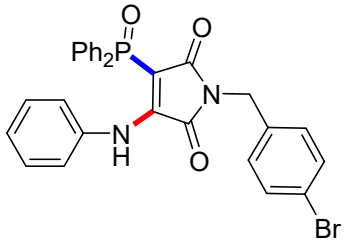


4w

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (74.7 mg, 73% yield), Mp = 128-129℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.86 (s, 1H), 7.95-7.89 (m, 4H), 7.64-7.59 (m, 2H), 7.57-7.52 (m, 4H), 7.42-7.38 (m, 2H), 7.34-7.28 (m, 7H), 4.58 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.17 (d, *J* = 12.0 Hz), 163.70 (d, *J* = 13.0

Hz), 156.42, 136.69, 134.75, 133.82, 132.59 (d, J = 109.1 Hz), 132.46 (d, J = 2.8 Hz), 131.45 (d, J = 11.3 Hz), 130.20, 128.90, 128.88, 128.67 (d, J = 13.0 Hz), 127.29, 124.86, 87.99 (d, J = 118.1 Hz), 40.98; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.22; **HRMS** (ESI): calcd for $C_{29}H_{23}N_2O_3PCl$ $[M + H]^+$ 513.1135, found 513.1135.

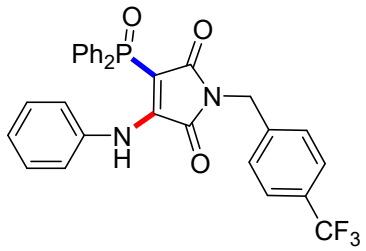
1-(4-bromobenzyl)-3-(diphenylphosphoryl)-4-(phenylamino)-1H-pyrrole-2,5-dione



4x

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (83.4 mg, 75% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.85 (s, 1H), 7.94-7.89 (m, 4H), 7.64-7.59 (m, 2H), 7.57-7.52 (m, 4H), 7.45-7.38 (m, 4H), 7.34-7.30 (m, 3H), 7.23-7.21 (m, 2H), 4.56 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.16 (d, J = 11.8 Hz), 163.69 (d, J = 12.4 Hz), 156.36, 136.68, 135.25, 132.54 (d, J = 109.1 Hz), 132.48 (d, J = 3.1 Hz), 131.85, 131.46 (d, J = 11.4 Hz), 130.54, 128.92, 128.68 (d, J = 12.9 Hz), 127.30, 124.87, 121.98, 87.96 (d, J = 118.2 Hz), 41.04; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.24; **HRMS** (ESI): calcd for $C_{29}H_{23}N_2O_3PBr$ $[M + H]^+$ 557.0630, found 557.0631.

3-(diphenylphosphoryl)-4-(phenylamino)-1-(4-(trifluoromethyl)benzyl)-1H-pyrrole-2,5-dione

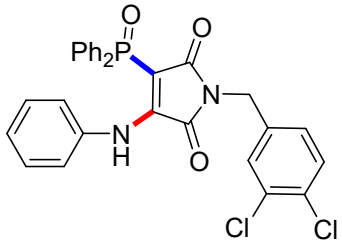


4y

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (76.4 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.88 (s, 1H), 7.95-7.90 (m, 4H), 7.64-7.53 (m, 8H), 7.46-7.38 (m, 4H), 7.35-7.30 (m, 3H), 4.67 (s, 2H); **^{13}C NMR** (100 MHz,

$CDCl_3$): δ 169.08 (d, *J* = 11.9 Hz), 163.68 (d, *J* = 13.1 Hz), 156.32, 140.08, 136.63, 132.50 (d, *J* = 3.0 Hz), 132.49 (d, *J* = 109.1 Hz), 131.44 (d, *J* = 11.3 Hz), 130.15 (d, *J* = 32.3 Hz), 128.96, 128.92, 128.68 (d, *J* = 12.9 Hz), 127.35, 125.74, 125.70, 124.87, 88.02 (d, *J* = 118.2 Hz), 41.14; **^{19}F NMR** (375 MHz, $CDCl_3$): δ -62.59 (s, 3F); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.20; **HRMS** (ESI): calcd for $C_{30}H_{23}N_2O_3F_3P$ $[M + H]^+$ 547.1398, found 547.1389.

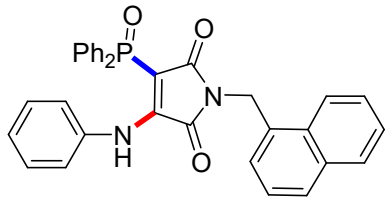
1-(3,4-dichlorobenzyl)-3-(diphenylphosphoryl)-4-(phenylamino)-1H-pyrrole-2,5-dione



4z

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (79.7 mg, 73% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.87 (s, 1H), 7.94-7.88 (m, 4H), 7.64-7.60 (m, 2H), 7.57-7.52 (m, 4H), 7.43-7.36 (m, 4H), 7.34-7.29 (m, 3H), 7.19-7.16 (m, 1H), 4.55 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.04 (d, *J* = 11.8 Hz), 163.67, 156.32, 136.61, 136.35, 132.74, 132.52 (d, *J* = 2.9 Hz), 132.45 (d, *J* = 110.3 Hz), 132.14, 131.45 (d, *J* = 11.4 Hz), 130.70, 130.66, 128.94, 128.70 (d, *J* = 13.1 Hz), 128.15, 127.38, 124.88, 88.02 (d, *J* = 117.4 Hz), 40.54; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.24; **HRMS** (ESI): calcd for $C_{29}H_{22}N_2O_3PCl_2$ $[M + H]^+$ 547.0745, found 547.0745.

3-(diphenylphosphoryl)-1-(naphthalen-1-ylmethyl)-4-(phenylamino)-1H-pyrrole-2,5-dione

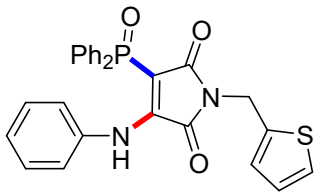


4ab

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (81.3 mg, 77% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.83 (s, 1H), 8.29-8.26 (m, 1H), 7.96-7.87 (m, 5H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.64-7.59 (m, 2H), 7.56-7.51 (m, 7H), 7.45-7.41

(m, 1H), 7.39-7.35 (m, 2H), 7.31-7.28 (m, 3H), 5.11 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.45 (d, *J* = 12.3 Hz), 163.98 (d, *J* = 13.1 Hz), 156.30, 136.74, 133.82, 132.60 (d, *J* = 109.1 Hz), 132.45 (d, *J* = 2.9 Hz), 131.52 (d, *J* = 11.5 Hz), 131.39, 131.28, 128.88, 128.80, 128.74, 128.65 (d, *J* = 13.0 Hz), 127.94, 127.17, 126.49, 125.87, 125.38, 124.78, 123.61, 88.13 (d, *J* = 118.4 Hz), 39.75; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.40; **HRMS** (ESI): calcd for $C_{33}H_{26}N_2O_3P$ $[M + H]^+$ 529.1681, found 529.1678.

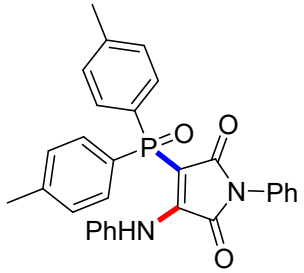
3-(diphenylphosphoryl)-4-(phenylamino)-1-(thiophen-2-ylmethyl)-1H-pyrrole-2,5-dione



4ac

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (58.1 mg, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.86 (s, 1H), 7.97-7.91 (m, 4H), 7.64-7.59 (m, 2H), 7.57-7.52 (m, 4H), 7.42-7.38 (m, 2H), 7.34-7.30 (m, 3H), 7.25-7.23 (m, 1H), 7.06-7.05 (m, 1H), 6.95-6.93 (m, 1H), 4.81 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.86 (d, *J* = 11.9 Hz), 163.32 (d, *J* = 12.8 Hz), 156.43, 138.13, 136.73, 132.61 (d, *J* = 109.1 Hz), 132.43 (d, *J* = 2.9 Hz), 131.49 (d, *J* = 11.2 Hz), 128.90, 128.65 (d, *J* = 13.0 Hz), 127.72, 127.22, 126.91, 126.01, 124.80, 88.08 (d, *J* = 117.9 Hz), 35.77; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.32; **HRMS** (ESI): calcd for $C_{27}H_{22}N_2O_3PS$ $[M + H]^+$ 485.1089, found 485.1087.

3-(di-p-tolylphosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione

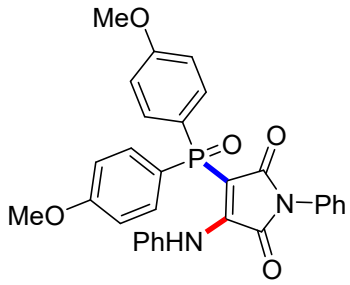


4ad

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow liquid (72.8 mg, 74% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.96 (s, 1H), 7.87 (dd, *J* = 13.0, 7.8 Hz, 4H), 7.44-7.30 (m, 14H), 2.46 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.55 (d, *J* = 11.9 Hz), 163.01 (d, *J* = 12.5 Hz), 155.68, 143.07, 143.05, 136.87, 131.60 (d, *J* = 11.6 Hz), 130.67 (d, *J* = 147.9 Hz), 129.44 (d, *J* = 13.3 Hz), 128.90, 128.82, 127.70, 127.21, 126.17, 124.98, 88.83 (d, *J* = 117.5 Hz), 21.80; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.51; **HRMS** (ESI): calcd for $C_{30}H_{26}N_2O_3P$ $[M + H]^+$ 493.1681, found 493.1683.

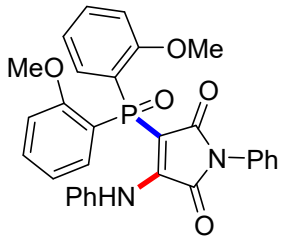
3-(bis(4-methoxyphenyl)phosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione



4ae

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (79.7 mg, 76% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.95 (s, 1H), 7.90 (t, *J* = 8.7 Hz, 4H), 7.44-7.29 (m, 10H), 7.05 (d, *J* = 8.1 Hz, 4H), 3.90 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.65 (d, *J* = 12.0 Hz), 163.07 (d, *J* = 12.4 Hz), 162.90, 162.87, 155.42, 136.92, 133.53 (d, *J* = 12.8 Hz), 131.43, 128.89, 127.41 (d, *J* = 53.5 Hz), 125.55 (d, *J* = 123.5 Hz), 124.57, 123.40, 114.31, 114.17, 89.28 (d, *J* = 117.8 Hz), 55.46; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 28.93; **HRMS** (ESI): calcd for $C_{30}H_{26}N_2O_5P$ $[M + H]^+$ 525.1579, found 525.1577.

3-(bis(2-methoxyphenyl)phosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione

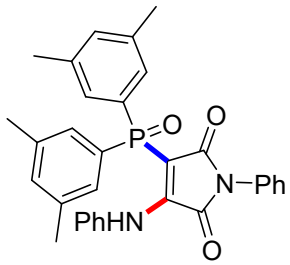


4af

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow solid (63.9 mg, 61% yield), Mp = 106-107℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.81 (s, 1H), 7.59-7.53 (m, 4H), 7.42-7.35 (m, 8H), 7.31-7.26 (m, 2H), 7.08-7.03 (m, 4H), 3.86 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.42 (d, *J* = 11.1 Hz), 163.35 (d, *J* = 13.2 Hz), 162.06 (d, *J* = 2.3 Hz), 155.26 (d, *J* = 3.9 Hz), 137.37, 134.39 (d, *J* = 2.1 Hz), 133.73 (d, *J* = 10.2 Hz), 131.73, 128.81, 127.05 (d, *J* = 77.7 Hz), 125.29 (d, *J* = 156.8 Hz), 120.79, 120.66, 120.33, 119.18, 111.43, 111.36, 90.50 (d, *J* = 123.7 Hz), 55.84; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 26.92; **HRMS** (ESI): calcd for $C_{30}H_{26}N_2O_5P$ $[M + H]^+$ 525.1579, found 525.1578.

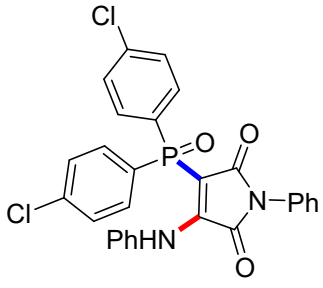
3-(bis(3,5-dimethylphenyl)phosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione



4ag

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (45.8 mg, 44% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.01 (s, 1H), 7.62 (d, *J* = 1.7 Hz, 2H), 7.58 (d, *J* = 1.6 Hz, 2H), 7.46-7.30 (m, 10H), 7.25 (s, 2H), 2.42 (s, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.31 (d, *J* = 11.9 Hz), 162.98 (d, *J* = 12.8 Hz), 155.65, 138.40, 138.26, 136.86, 134.28 (d, *J* = 109.1 Hz), 134.20 (d, *J* = 2.9 Hz), 128.99, 128.88, 128.82 (d, *J* = 4.9 Hz), 127.59, 127.07, 126.08, 124.91, 88.73 (d, *J* = 116.0 Hz), 21.43; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 30.03; **HRMS** (ESI): calcd for $C_{32}H_{30}N_2O_3P$ $[M + H]^+$ 521.1994, found 521.1993.

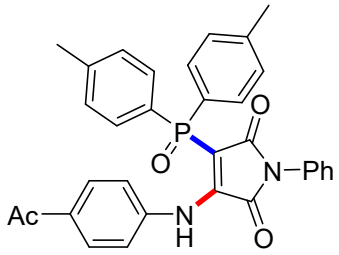
3-(bis(4-chlorophenyl)phosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione



4ah

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (77.7 mg, 73% yield), Mp = 87-88℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.85 (s, 1H), 7.95-7.90 (m, 4H), 7.56-7.53 (m, 4H), 7.46-7.32 (m, 10H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.58 (d, *J* = 13.7 Hz), 162.61 (d, *J* = 12.5 Hz), 156.04, 139.35, 136.50, 132.91 (d, *J* = 11.9 Hz), 131.27, 130.66 (d, *J* = 100.9 Hz), 129.18 (d, *J* = 13.8 Hz), 129.00, 127.95, 127.59, 126.14, 125.06, 87.19 (d, *J* = 120.5 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 27.92; **HRMS** (ESI): calcd for $C_{28}H_{20}N_2O_3PCl_2$ $[M + H]^+$ 533.0589, found 533.0585.

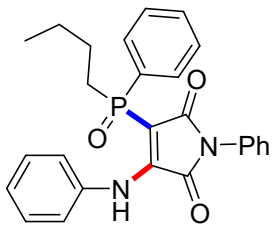
3-((4-acetylphenyl)amino)-4-(di-p-tolylphosphoryl)-1-phenyl-1H-pyrrole-2,5-dione



4ai

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (74.8 mg, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.16 (s, 1H), 7.99-7.97 (m, 2H), 7.89-7.84 (m, 4H), 7.48-7.41 (m, 4H), 7.38-7.33 (m, 7H), 2.61 (s, 3H), 2.46 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.29 (d, *J* = 11.4 Hz), 163.08 (d, *J* = 12.5 Hz), 155.03, 143.30, 143.27, 141.11, 135.00, 131.61 (d, *J* = 11.6 Hz), 131.25, 129.50 (d, *J* = 13.5 Hz), 129.22, 128.43 (d, *J* = 110.3 Hz), 128.31, 126.14, 124.09, 91.72 (d, *J* = 114.7 Hz), 26.64, 21.79; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.63; **HRMS** (ESI): calcd for $C_{32}H_{28}N_2O_4P$ $[M + H]^+$ 535.1787, found 535.1786.

3-(butyl(phenyl)phosphoryl)-1-phenyl-4-(phenylamino)-1H-pyrrole-2,5-dione

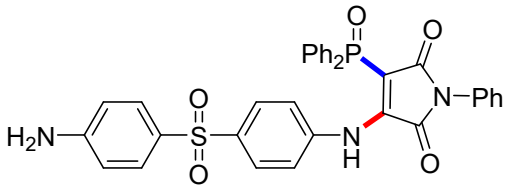


4aj

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a

yellow liquid (70.2 mg, 79% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.61 (s, 1H), 8.09-8.04 (m, 2H), 7.63-7.54 (m, 3H), 7.47-7.43 (m, 2H), 7.41-7.28 (m, 8H), 2.58-2.47 (m, 1H), 2.42-2.32 (m, 1H), 1.78-1.60 (m, 2H), 1.57-1.49 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.84 (d, *J* = 12.5 Hz), 163.02 (d, *J* = 11.9 Hz), 155.13, 136.78, 133.03 (d, *J* = 102.8 Hz), 132.10 (d, *J* = 2.9 Hz), 131.37, 130.45 (d, *J* = 10.4 Hz), 128.94 (d, *J* = 9.0 Hz), 128.78, 127.47 (d, *J* = 67.7 Hz), 126.19, 124.81, 89.36 (d, *J* = 109.1 Hz), 30.94 (d, *J* = 73.4 Hz), 23.96 (d, *J* = 15.8 Hz), 23.17 (d, *J* = 4.4 Hz), 13.74; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 36.08; **HRMS** (ESI): calcd for $C_{26}H_{26}N_2O_3P$ $[M + H]^+$ 445.1681, found 445.1683.

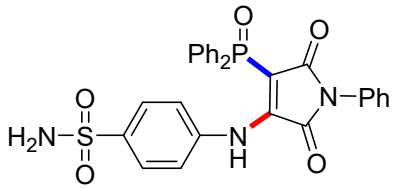
3-((4-((4-aminophenyl)sulfonyl)phenyl)amino)-4-(diphenylphosphoryl)-1-phenyl-1H-pyrrole-2,5-dione



5a

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (87.9 mg, 71% yield), Mp = 95-96℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.14 (s, 1H), 7.99-7.93 (m, 4H), 7.89-7.87 (m, 2H), 7.73-7.70 (m, 2H), 7.65-7.60 (m, 2H), 7.57-7.52 (m, 4H), 7.46-7.42 (m, 4H), 7.37-7.31 (m, 3H), 6.67 (d, *J* = 8.7 Hz, 2H), 4.20 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.14 (d, *J* = 11.6 Hz), 162.98 (d, *J* = 12.6 Hz), 155.12, 151.33, 140.63, 140.47, 132.74, 131.76 (d, *J* = 110.2 Hz), 131.54 (d, *J* = 11.3 Hz), 131.05, 129.94, 129.07, 129.01, 128.80 (d, *J* = 13.0 Hz), 128.06, 128.00, 126.19, 124.42, 114.28, 91.62 (d, *J* = 114.8 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.38; **HRMS** (ESI): calcd for $C_{34}H_{27}N_3O_5PS$ $[M + H]^+$ 620.1409, found 620.1408.

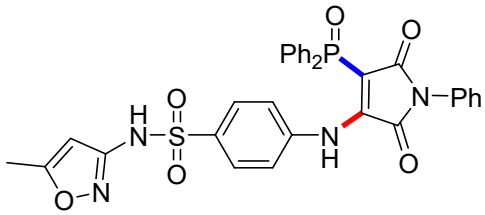
4-((4-(diphenylphosphoryl)-2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)amino)benzenesulfonamide



5b

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (79.3 mg, 73% yield), Mp = 209-210℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 11.12 (s, 1H), 8.01-7.90 (m, 6H), 7.66-7.62 (m, 2H), 7.59-7.54 (m, 4H), 7.51-7.43 (m, 4H), 7.38-7.33 (m, 3H), 4.96 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.37 (d, *J* = 11.9 Hz), 163.78 (d, *J* = 12.8 Hz), 156.46, 137.60, 136.80, 133.40, 132.67 (d, *J* = 109.3 Hz), 132.40 (d, *J* = 3.2 Hz), 131.49 (d, *J* = 11.2 Hz), 129.38, 128.81 (d, *J* = 13.7 Hz), 128.71, 128.58, 127.17, 124.85, 87.95 (d, *J* = 118.6 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.48; **HRMS** (ESI): calcd for $C_{28}H_{23}N_3O_5PS$ $[M + H]^+$ 544.1096, found 544.1094.

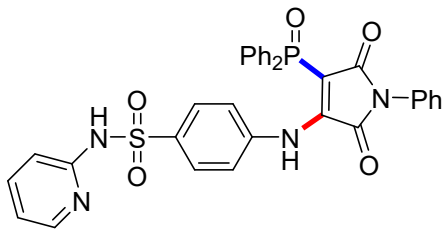
4-((4-(diphenylphosphoryl)-2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)amino)-N-(5-methylisoxazol-3-yl)benzenesulfonamide



5c

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow liquid (68.6 mg, 55% yield). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.95 (s, 1H), 9.87 (brs, 1H), 8.01-7.92 (m, 3H), 7.89-7.83 (m, 1H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.65-7.60 (m, 2H), 7.57-7.52 (m, 4H), 7.47-7.32 (m, 7H), 6.19 (s, 1H), 2.34 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.83, 168.09, 162.95 (d, *J* = 13.0 Hz), 154.90, 141.16, 136.81, 132.84, 131.82, 131.64 (d, *J* = 11.6 Hz), 131.43 (d, *J* = 111.2 Hz), 131.03, 129.08, 128.85 (d, *J* = 13.2 Hz), 128.10, 127.80, 126.18, 124.47, 95.67, 91.92 (d, *J* = 115.4 Hz), 12.73; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 29.93; **HRMS** (ESI): calcd for $C_{32}H_{26}N_4O_6PS$ $[M + H]^+$ 625.1311, found 625.1306.

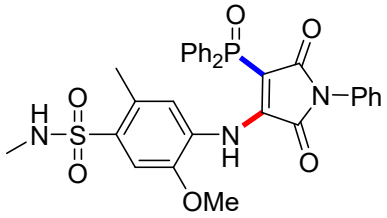
4-((4-(diphenylphosphoryl)-2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)amino)-N-(pyridin-2-yl)benzenesulfonamide



5d

Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (63.2 mg, 51% yield), Mp = 198-199℃. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 12.10 (brs, 1H), 10.76 (s, 1H), 8.05 (dd, *J* = 5.6, 1.9 Hz, 1H), 7.79-7.71 (m, 7H), 7.55-7.41 (m, 10H), 7.39-7.36 (m, 3H), 7.24 (d, *J* = 8.7 Hz, 1H), 6.90 (t, *J* = 6.4 Hz, 1H); **^{13}C NMR** (400 MHz, DMSO-d_6): δ 169.37 (d, *J* = 11.4 Hz), 164.21 (d, *J* = 12.7 Hz), 154.79, 153.78, 141.76, 141.10, 133.62 (d, *J* = 109.4 Hz), 132.29, 132.17, 131.44 (d, *J* = 10.6 Hz), 129.21, 128.92 (d, *J* = 12.5 Hz), 128.18, 127.53, 127.36, 124.07, 114.40, 91.44, 90.27, 52.24, 44.61 (d, *J* = 39.0 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$): δ 31.12; **HRMS** (ESI): calcd for $C_{33}H_{26}N_4O_5PS$ $[M + H]^+$ 621.1362, found 621.1360.

4-((4-(diphenylphosphoryl)-2,5-dioxo-1-phenyl-2,5-dihydro-1H-pyrrol-3-yl)amino)-5-methoxy-N,2-dimethylbenzenesulfonamide



5e

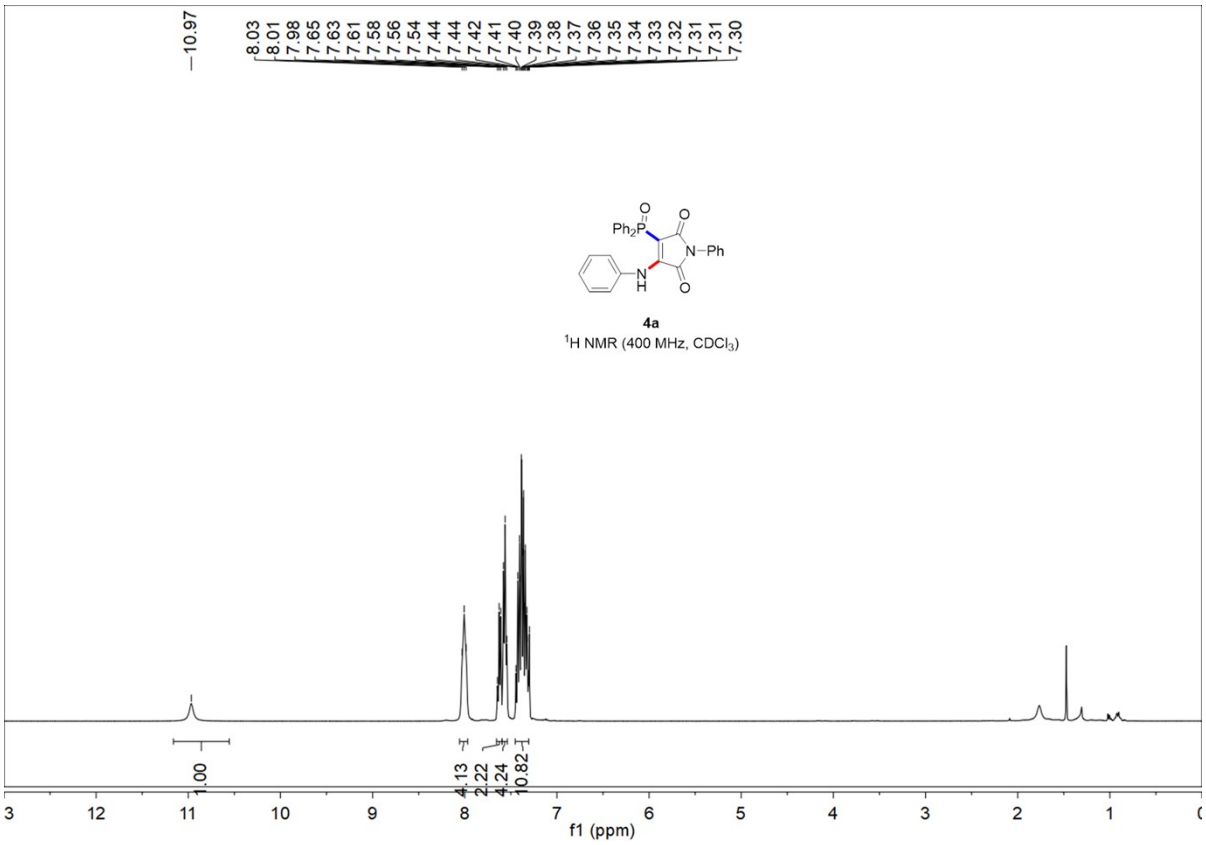
Following the general procedure, using (petroleum ether : EtOAc = 9 : 1) as the eluant afforded a yellow solid (82.9 mg, 69% yield), Mp = 128-129℃. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.67 (s, 1H), 8.00-7.94 (m, 4H), 7.64-7.59 (m, 2H), 7.57-7.52 (m, 5H), 7.45-7.41 (m, 2H), 7.35-7.32 (m, 4H), 7.26-7.22 (m, 1H), 3.91 (s, 3H), 2.62 (d, *J* = 5.2 Hz, 3H), 2.53 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.35 (d, *J* = 12.0 Hz), 163.12 (d, *J* = 12.9 Hz), 156.00, 150.61, 135.13, 132.56 (d, *J* = 2.9 Hz), 132.14 (d, *J* = 109.3 Hz), 131.57 (d, *J* = 11.2 Hz), 131.26, 129.65, 129.17, 129.04, 128.71,

128.71 (d, J = 12.9 Hz), 127.92, 126.17, 112.80, 90.36 (d, J = 116.0 Hz), 56.39, 29.05, 19.47; **^{31}P NMR** (162 MHz, $CDCl_3$): δ 28.84; **HRMS** (ESI): calcd for $C_{31}H_{29}N_3O_6PS$ $[M + H]^+$ 602.1515, found 602.1515.

References:

(1) (a) Ding, G.; Li, C.; Shen, Y.; Lu, B.; Zhang, Z.; Xie, X. *Adv. Synth. Catal.* **2016**, *358*, 1241-1250. (b) Matuszak, N.; Muccioli, G. G.; Labar, G.; Lambert, D. M. *J. Med Chem*. **2009**, *52*, 7410-7420.

^{1}H, ^{13}C and ^{19}F NMR spectra of products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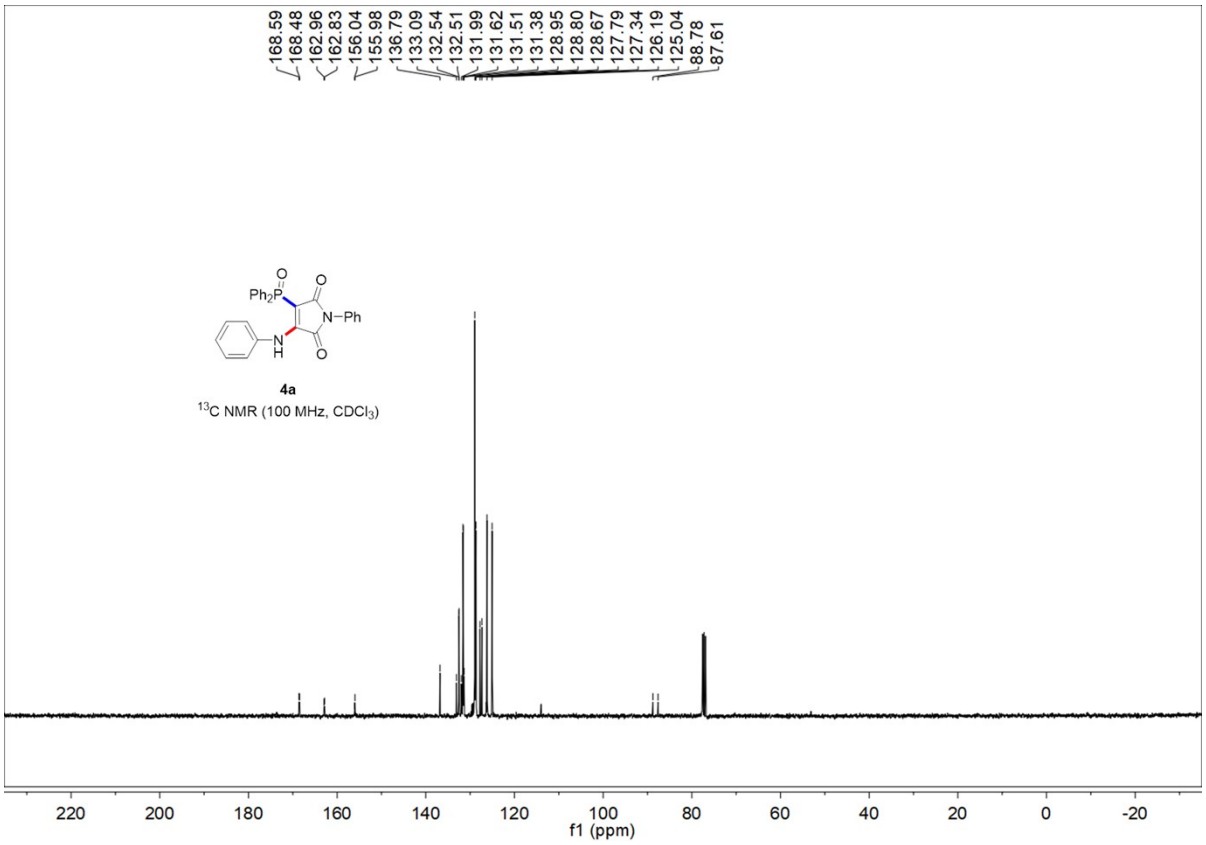
8.01
7.98
7.65
7.63
7.61
7.58
7.56
7.54
7.44
7.44
7.42
7.41
7.40
7.39
7.38
7.37
7.36
7.35
7.34
7.33
7.32
7.31
7.31
7.30
Ph2P
O
O
N–Ph
N
H
O
4a
1H NMR (400 MHz, CDCl3)
1.00
4.13
2.22
4.24
10.82
f1 (ppm)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168.48
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162.83
156.04
155.98
136.79
133.09
132.54
132.51
131.99
131.62
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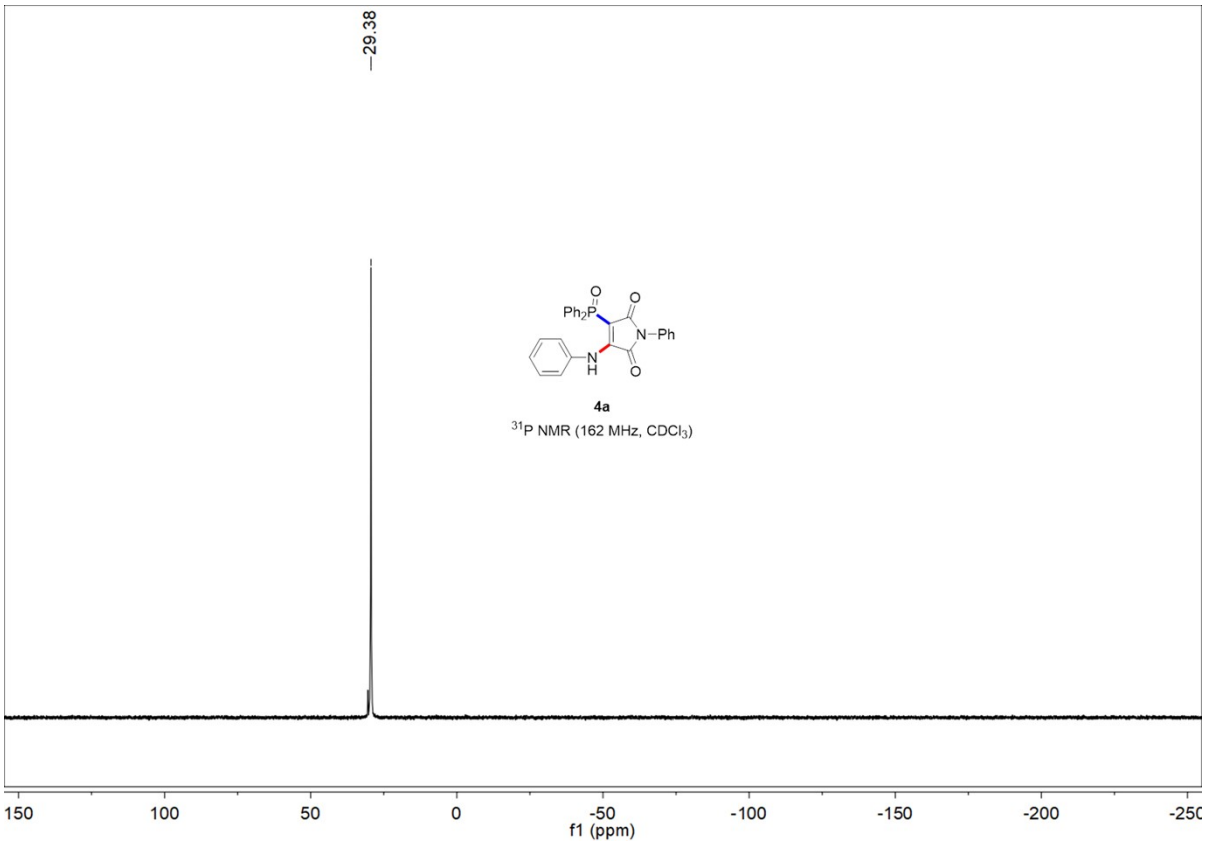
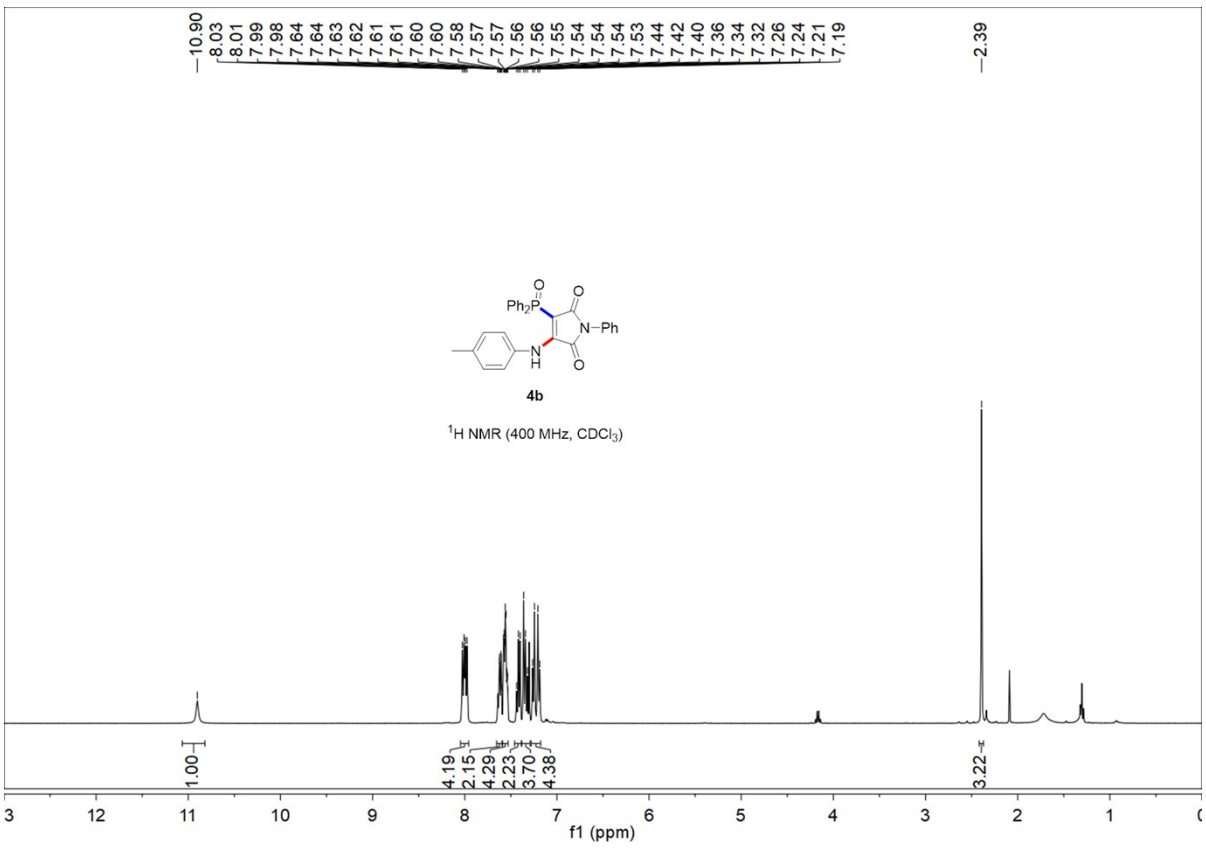
131.38
128.95
128.80
128.67
127.79
127.34
126.19
125.04
88.78
87.61
Ph2P
O
O
N–Ph
N
H
O
4a
13C NMR (100 MHz, CDCl3)
f1 (ppm)



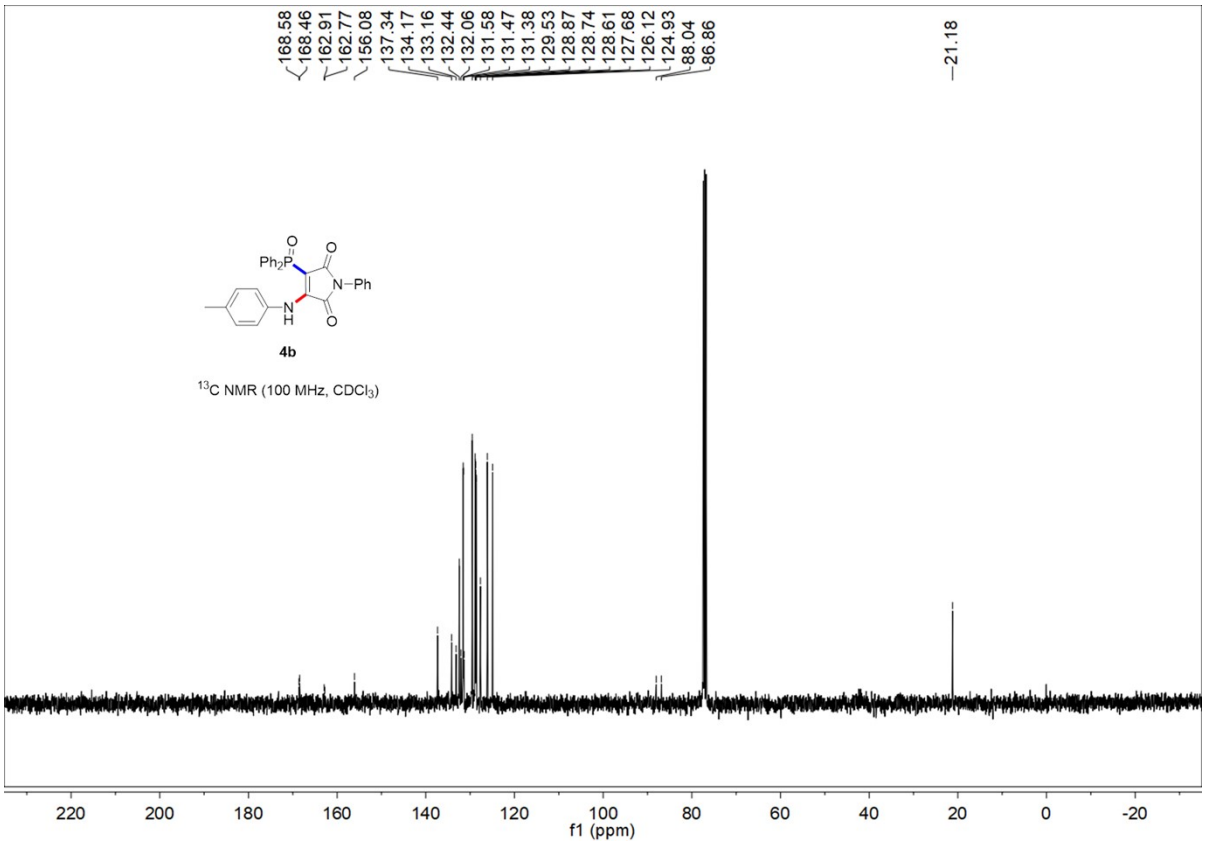
4a

^{31}P NMR (162 MHz, $CDCl_3$)



4b

^{1}H NMR (400 MHz, $CDCl_3$)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137.34
134.17
133.16
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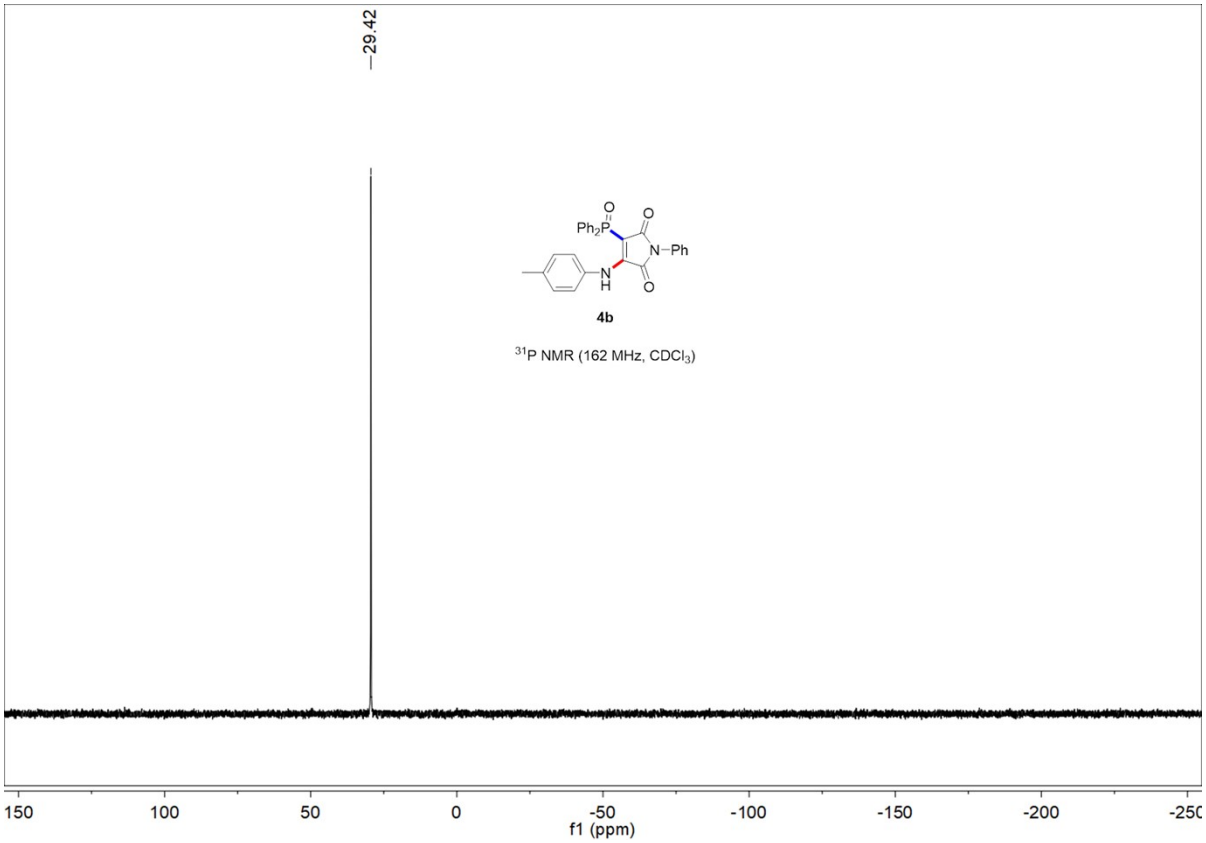
132.06
131.58
131.47
131.38
129.53
128.87
128.74
128.61
127.68
126.12
124.93
88.04
86.86
21.18
Ph2P
O
O
N–Ph
N
H
O
4b
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)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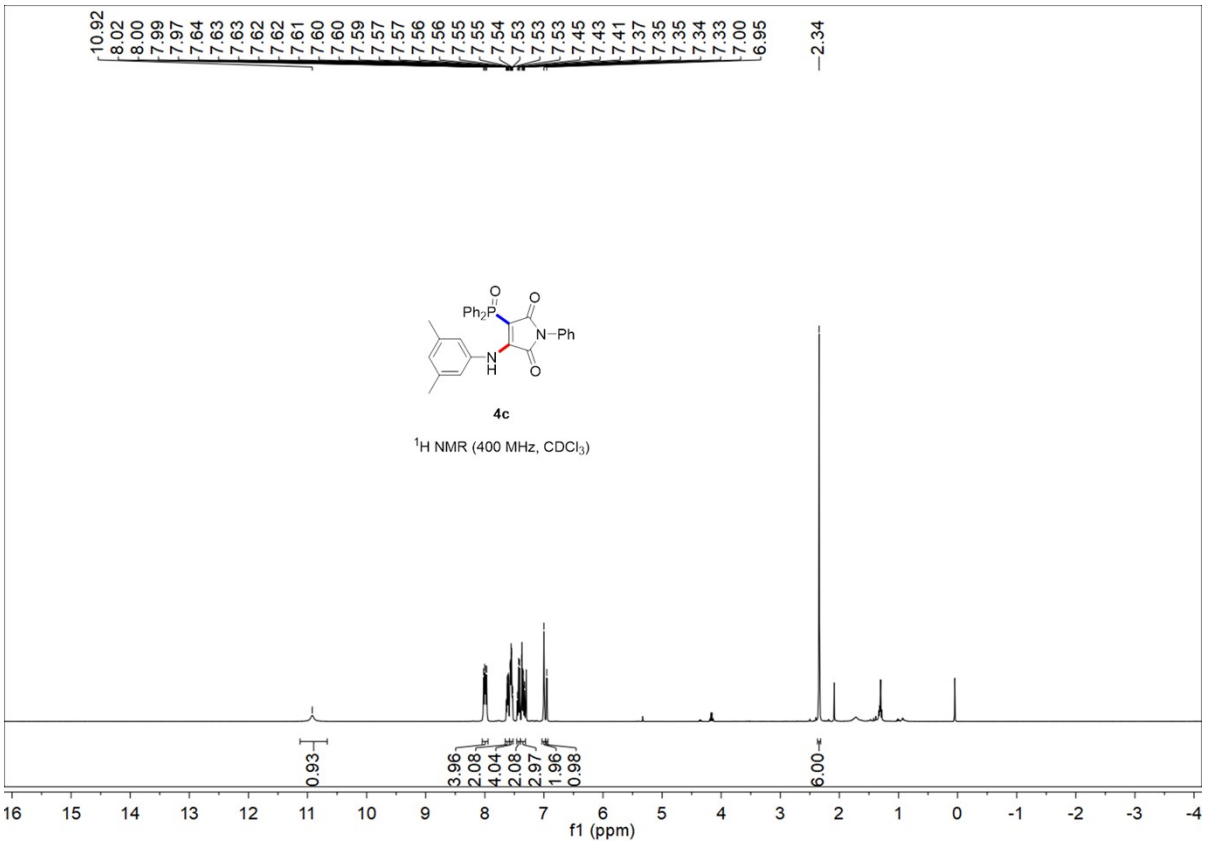
Ph2P
O
O
N–Ph
N
H
O
4b
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)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7.33
7.00
6.95
2.34
Ph2P
O
O
N–Ph
N
H
O
4c
1H NMR (400 MHz, CDCl3)
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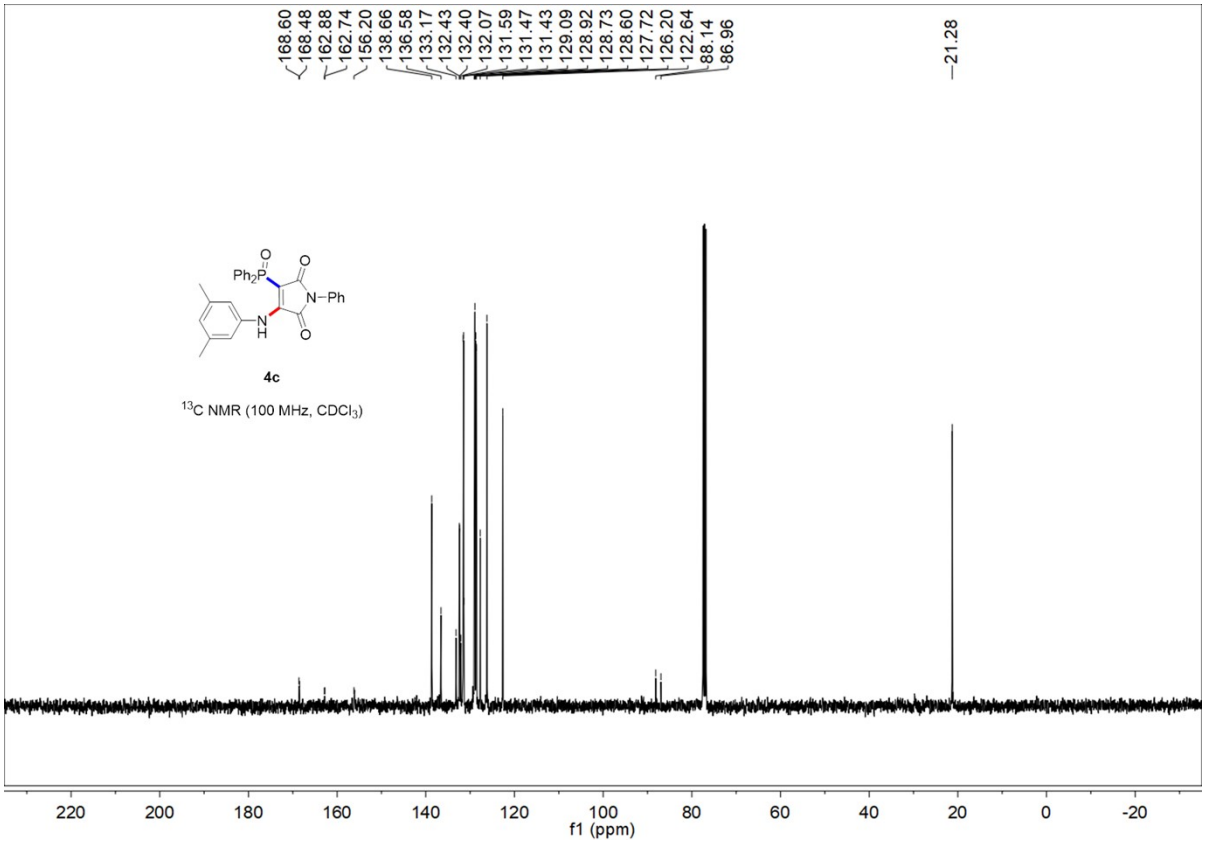
3.96
2.08
4.04
2.08
2.97
1.96
0.98
6.00
f1 (ppm)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133.17
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132.07
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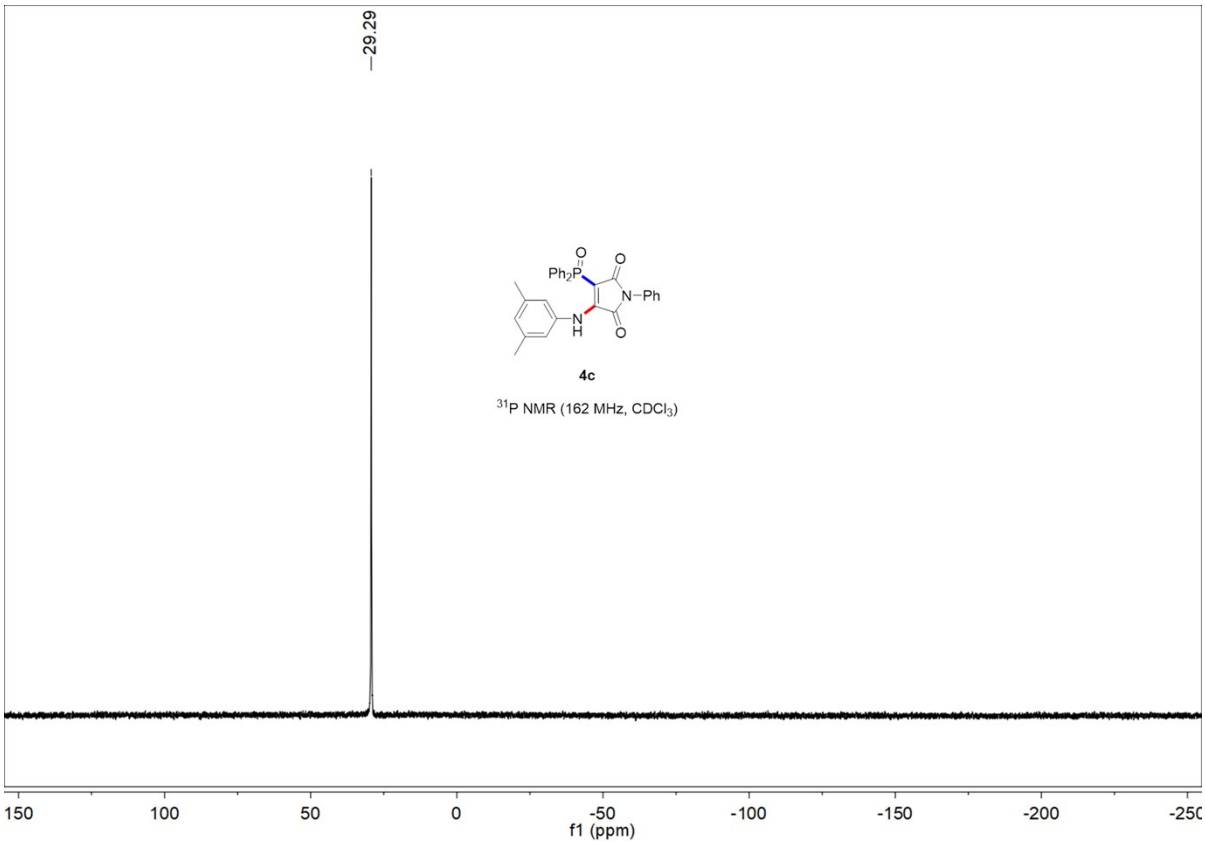
131.47
131.43
129.09
128.92
128.73
128.60
127.72
126.20
122.64
88.14
86.96
21.28
Ph2P
O
O
N–Ph
N
H
O
4c
13C NMR (100 MHz, CDCl3)
f1 (ppm)


29.29
Ph2P
O
O
N–Ph
N
H
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4c
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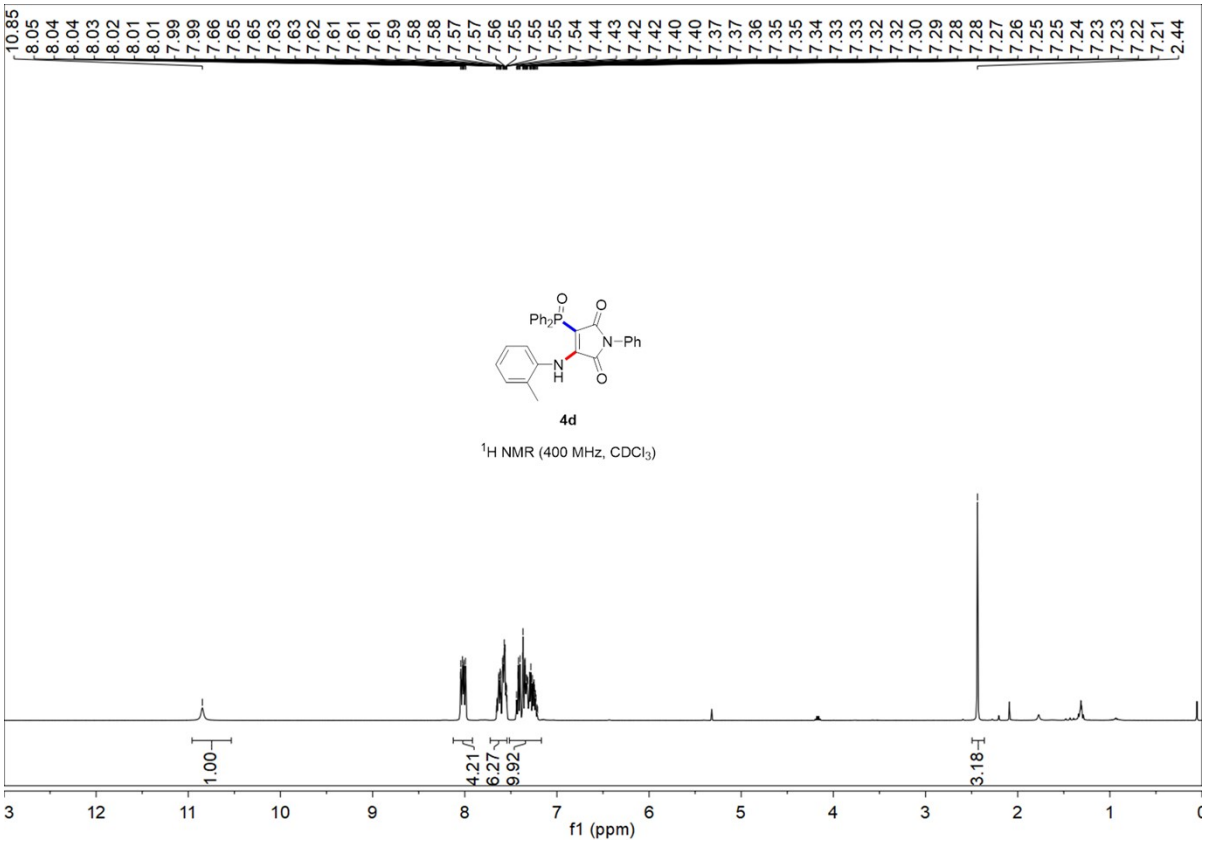
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)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7.27
7.26
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7.25
7.24
7.23
7.23
7.22
7.21
2.44
Ph2P
O
O
N–Ph
N
H
O
4d
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1.00
4.21
6.27
9.92
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3
12
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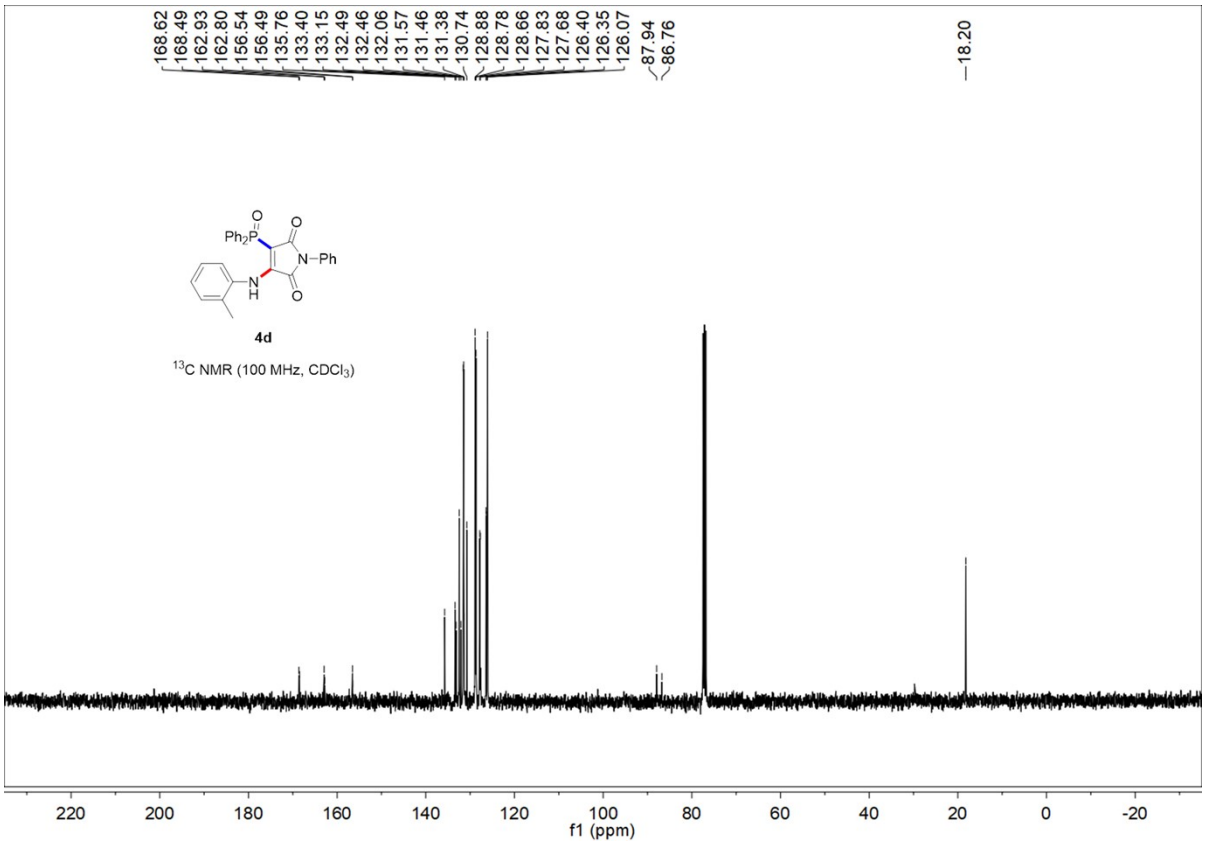
8
7
6
5
4
3
2
1
f1 (ppm)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131.46
131.38
130.74
128.88
128.78
128.66
127.83
127.68
126.40
126.35
126.07
87.94
86.76
18.20
Ph2P
O
O
N–Ph
N
H
O
4d
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220
200
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160
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120
100
80
60
40
20
0
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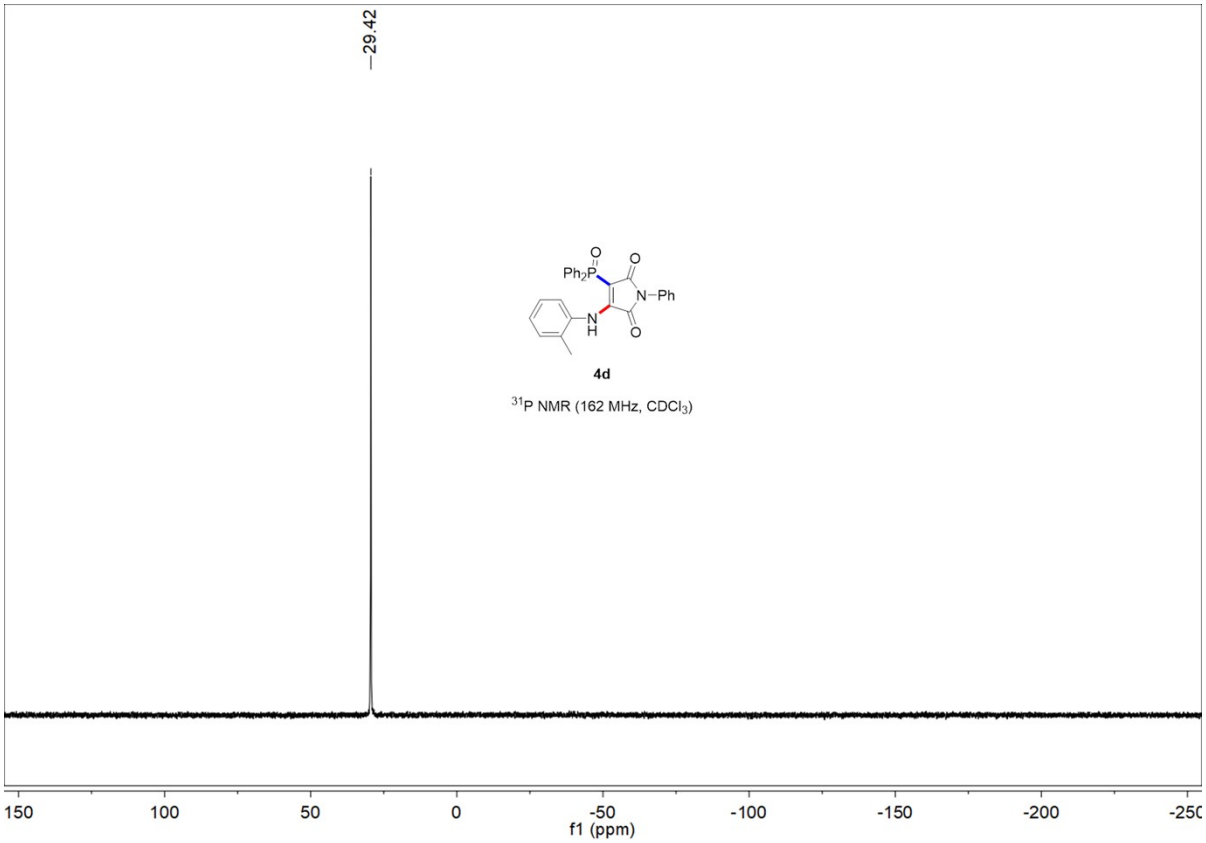
f1 (ppm)


29.42
Ph2P
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N–Ph
N
H
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4d
31P NMR (162 MHz, CDCl3)
150
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50
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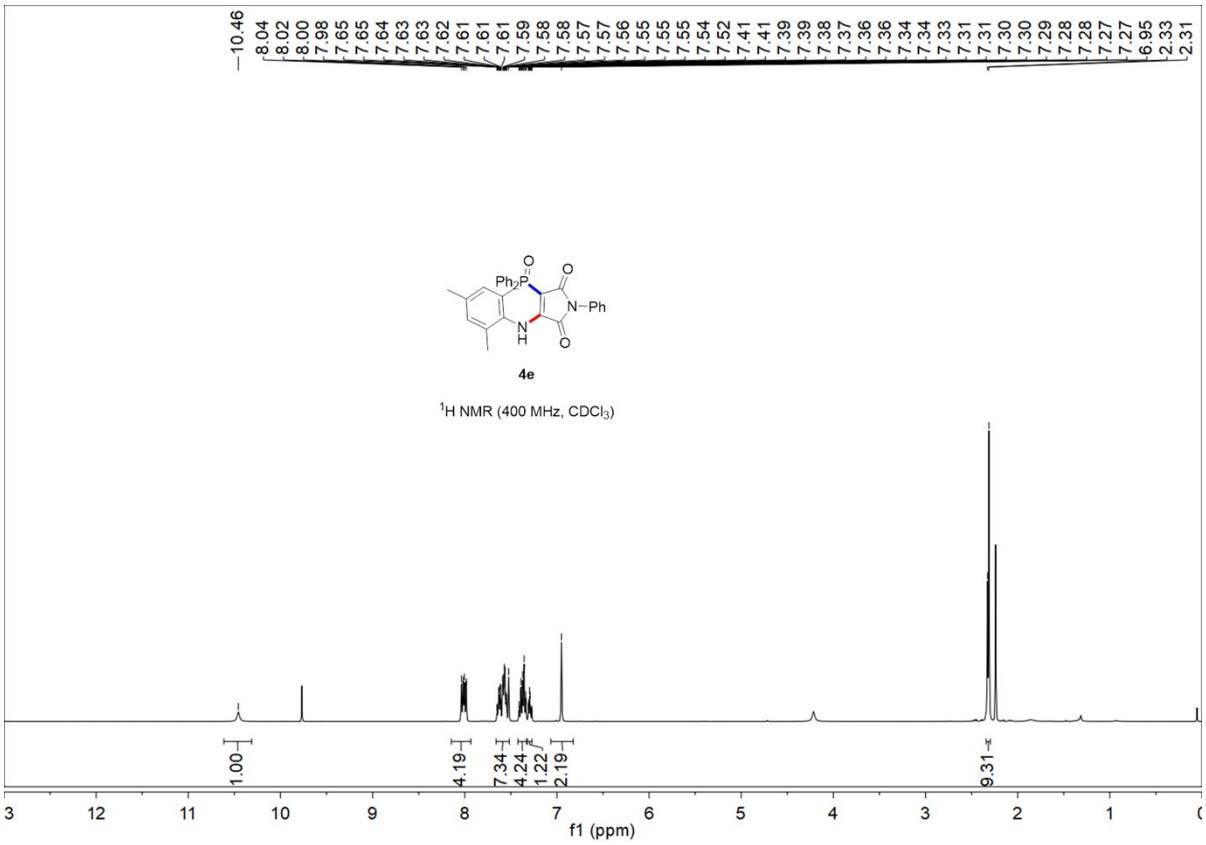
-50
-100
-150
-200
-250
f1 (ppm)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7.34
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N
4e
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7.34
4.24
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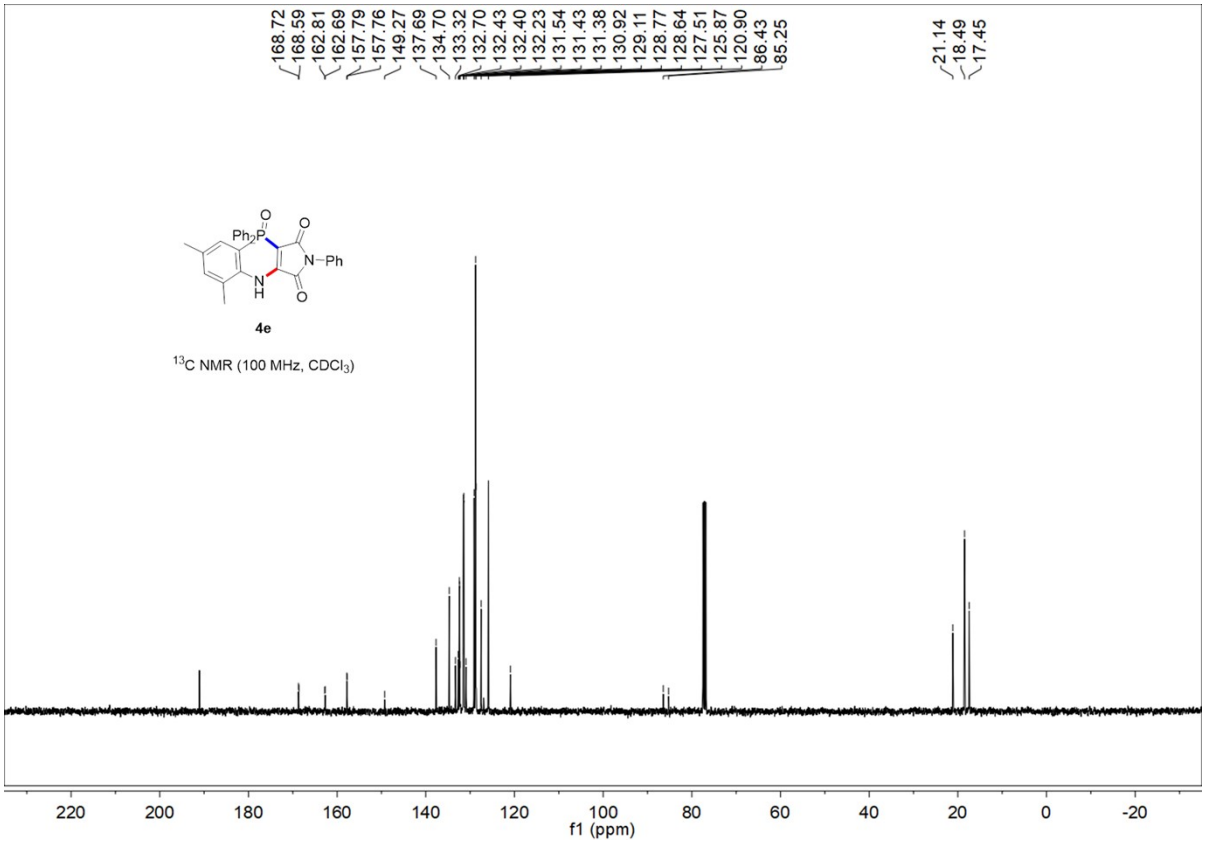
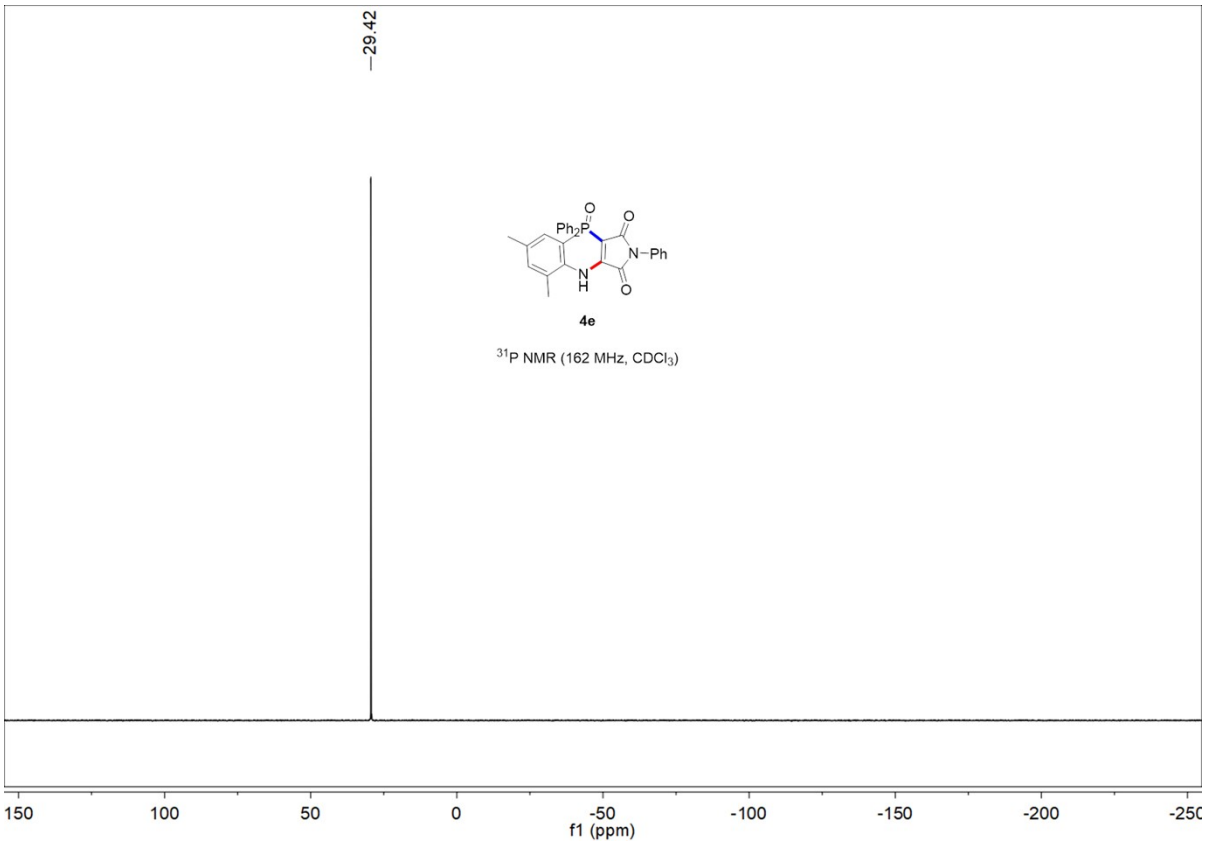
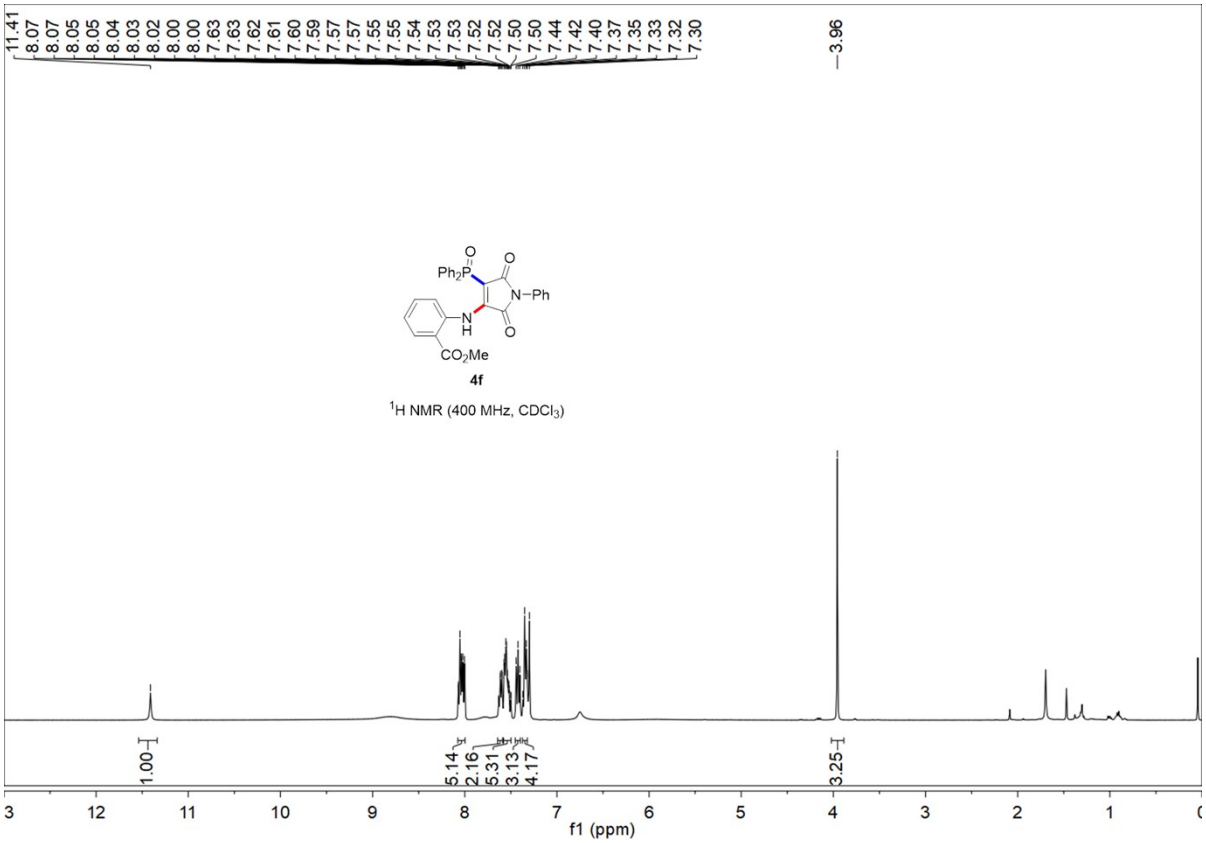
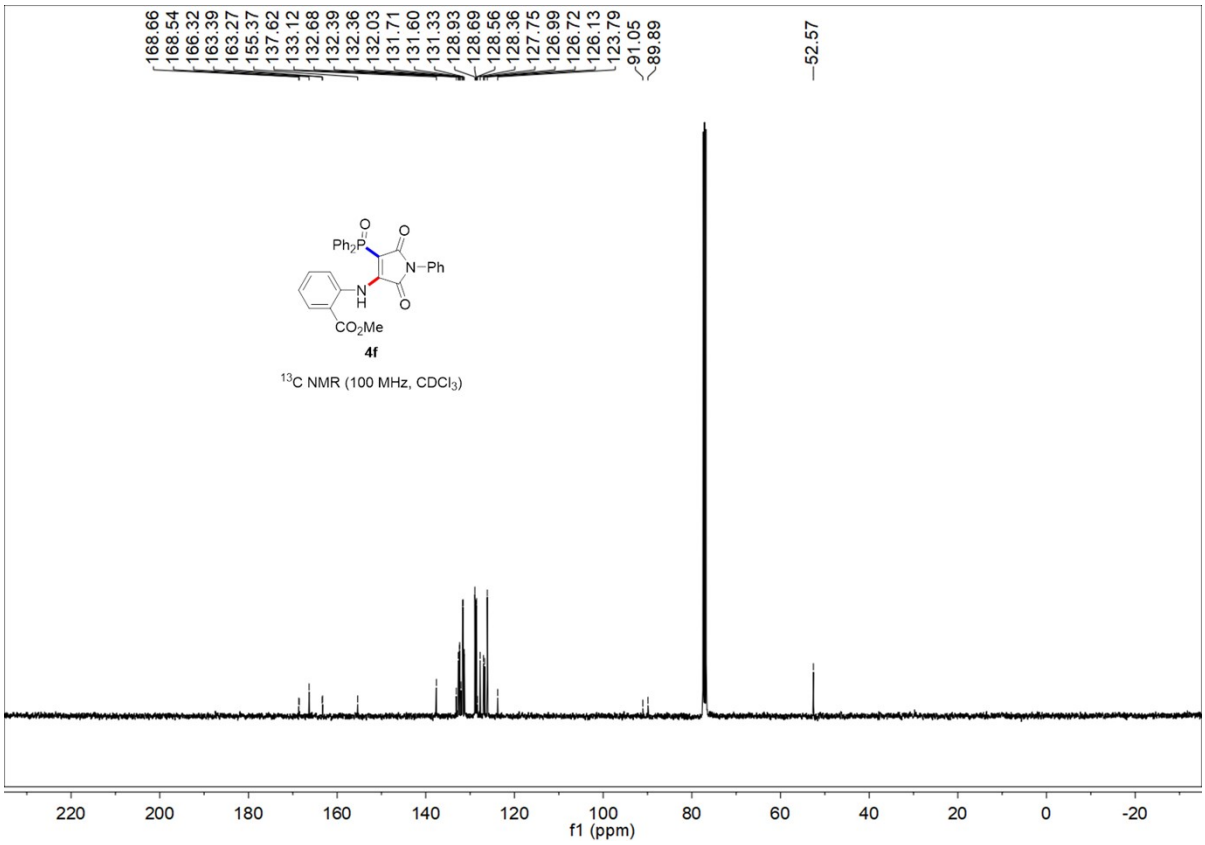
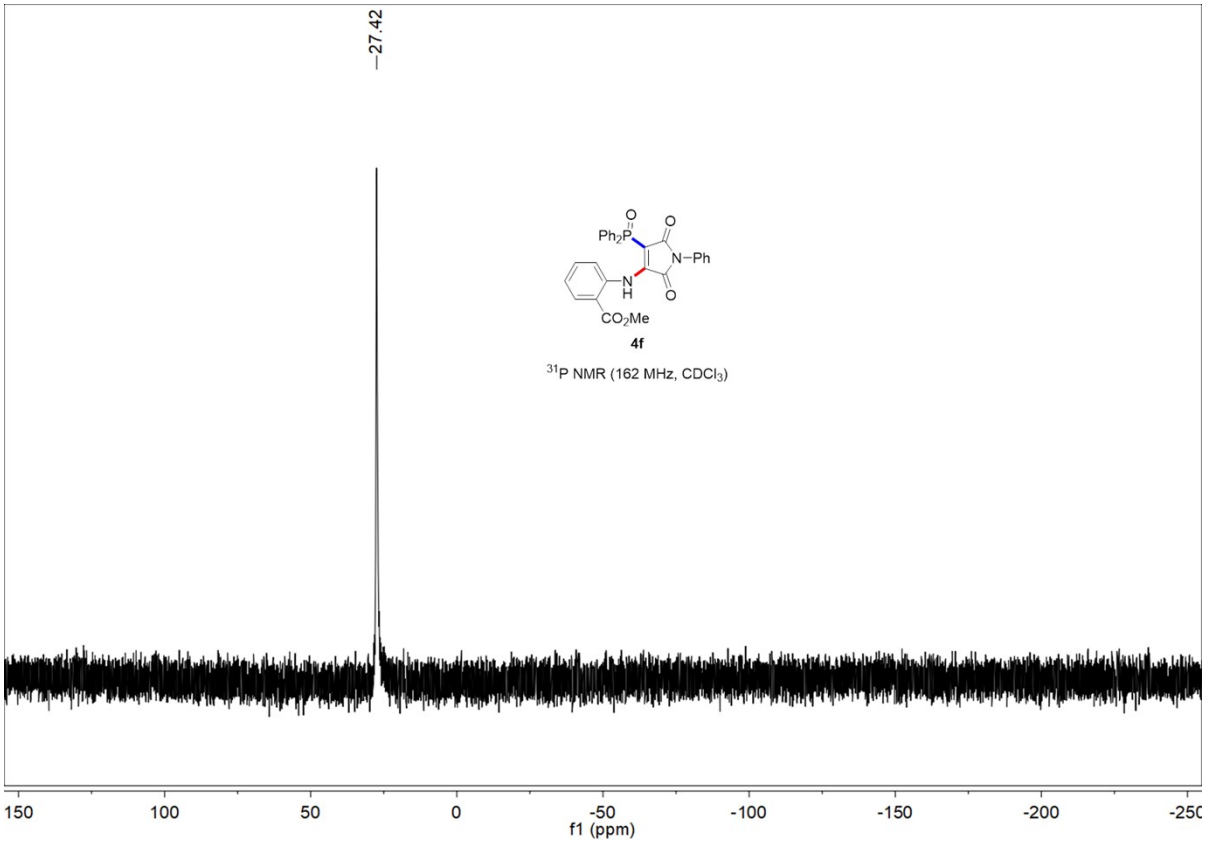
9.31
f1 (ppm)

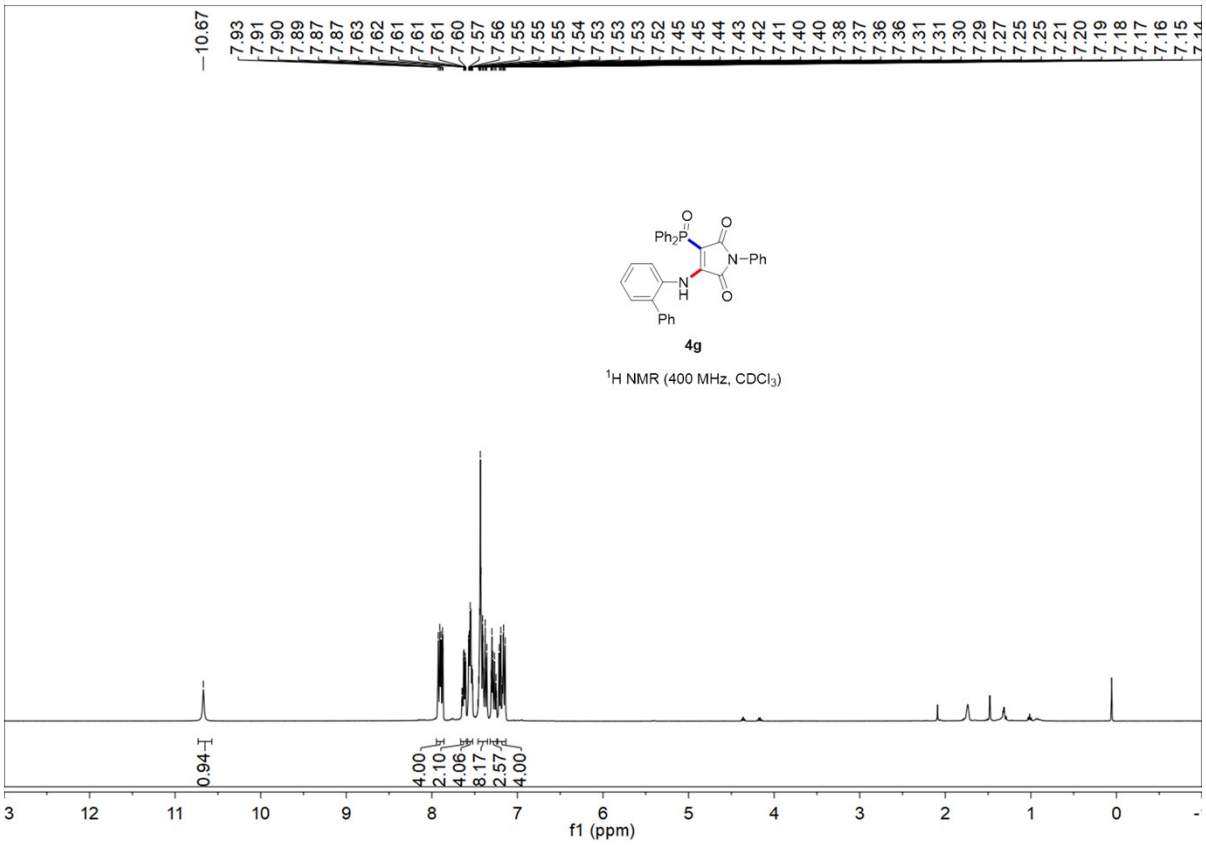

168.72
168.59
162.81
162.69
157.79
157.76
149.27
137.69
134.70
133.32
132.70
132.43
132.40
132.23
131.54
131.43
131.38
130.92
129.11
128.77
128.64
127.51
125.87
120.90
86.43
85.25
21.14
18.49
17.45
Ph2P
O
O
N–Ph
O
H
N
4e
13C NMR (100 MHz, CDCl3)
f1 (ppm)

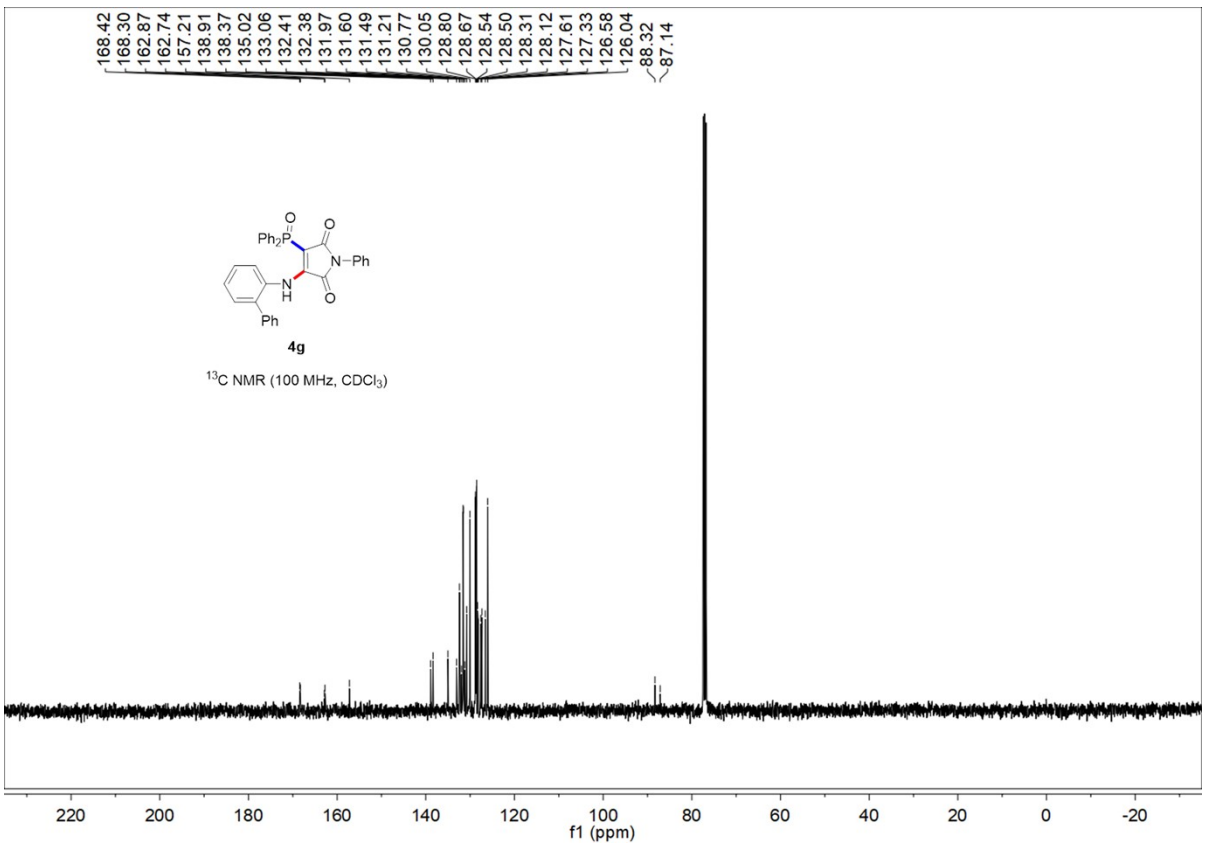

29.42
Ph2P
O
O
N–Ph
N
H
O
4e
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

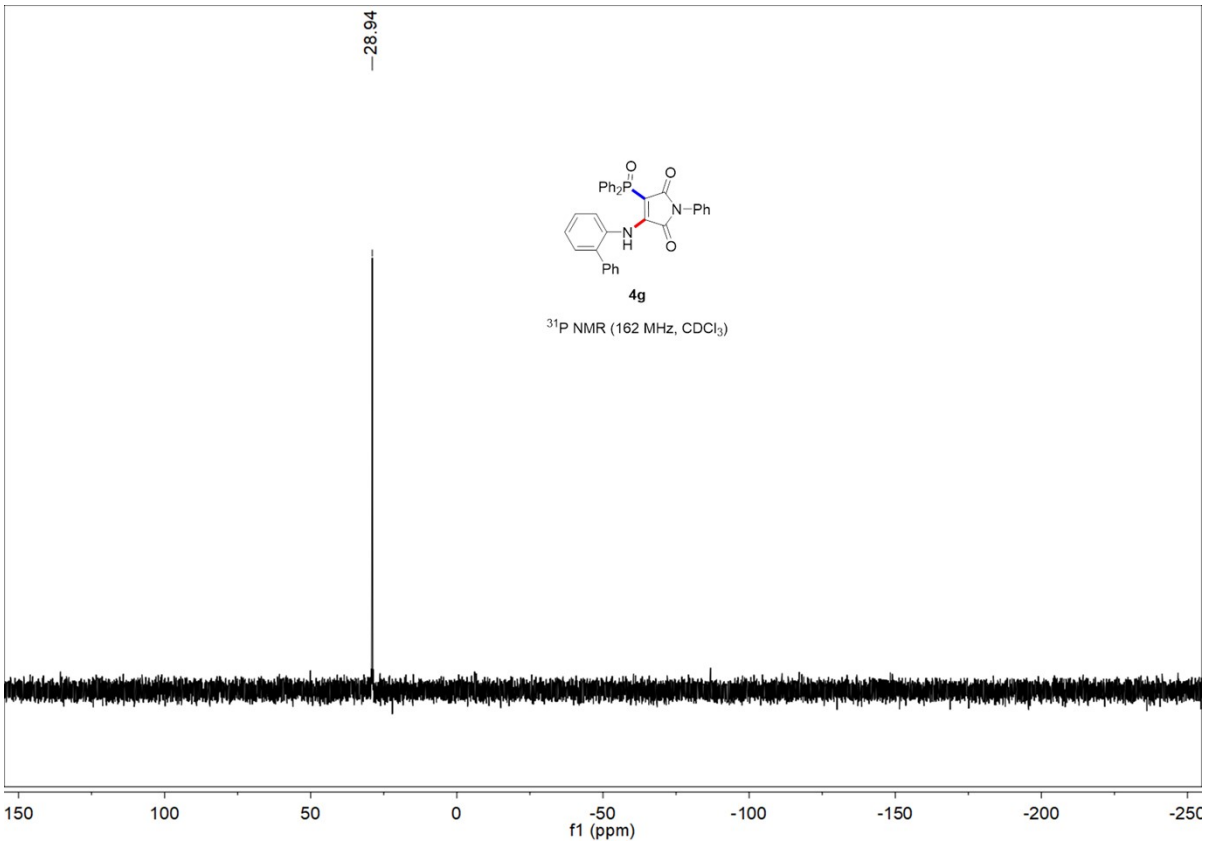

11.41
8.07
8.07
8.05
8.05
8.04
8.03
8.02
8.00
8.00
7.63
7.63
7.62
7.61
7.60
7.59
7.57
7.57
7.55
7.55
7.54
7.53
7.53
7.52
7.52
7.50
7.50
7.44
7.42
7.40
7.37
7.35
7.33
7.32
7.30
3.96
Ph2P
O
O
N–Ph
N
H
O
CO2Me
4f
1H NMR (400 MHz, CDCl3)
1.00
5.14
2.16
5.31
3.13
4.17
3.25
3
12
11
10
9
8
7
6
5
4
3
2
1
f1 (ppm)

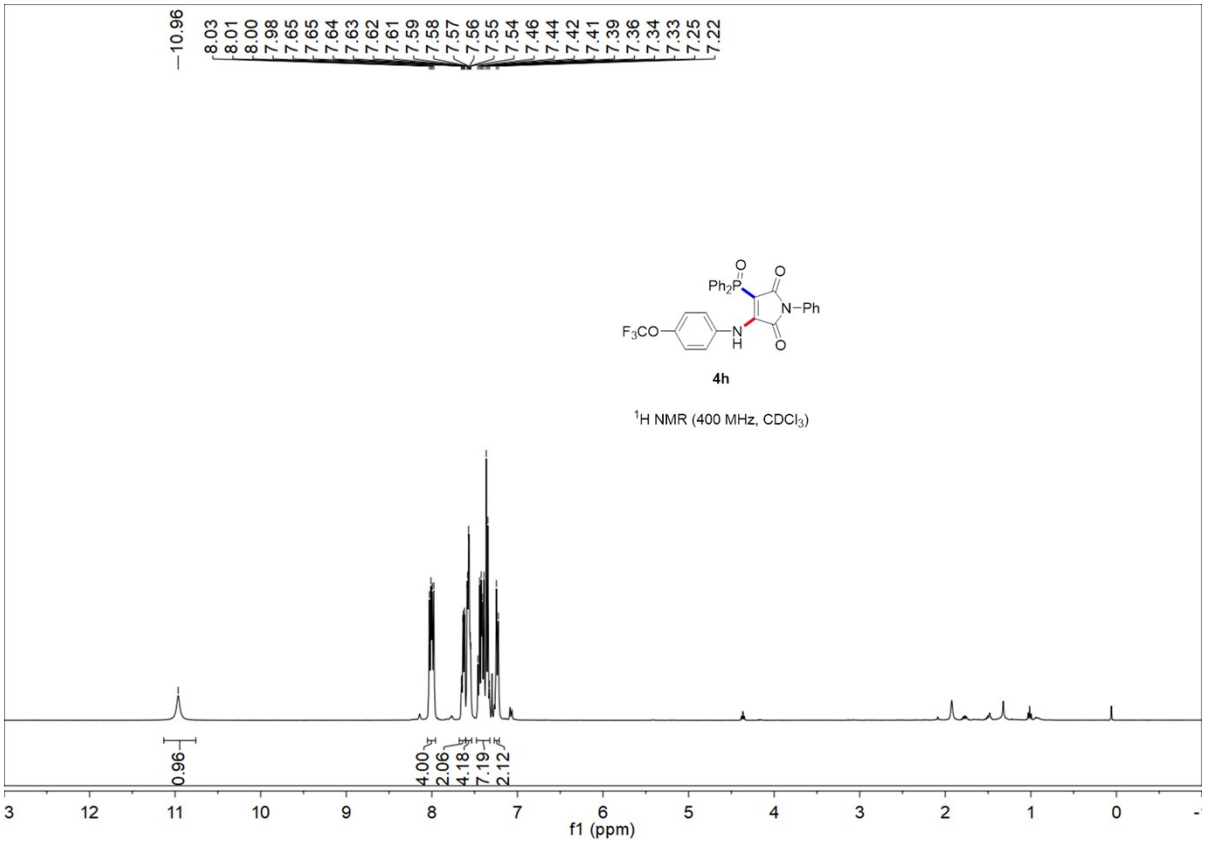

168.66
168.54
166.32
163.39
163.27
155.37
137.62
133.12
132.68
132.39
132.36
132.03
131.71
131.60
131.33
128.93
128.69
128.56
128.36
127.75
126.99
126.72
126.13
123.79
91.05
89.89
52.57
Ph2P
O
O
N–Ph
N
H
O
CO2Me
4f
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

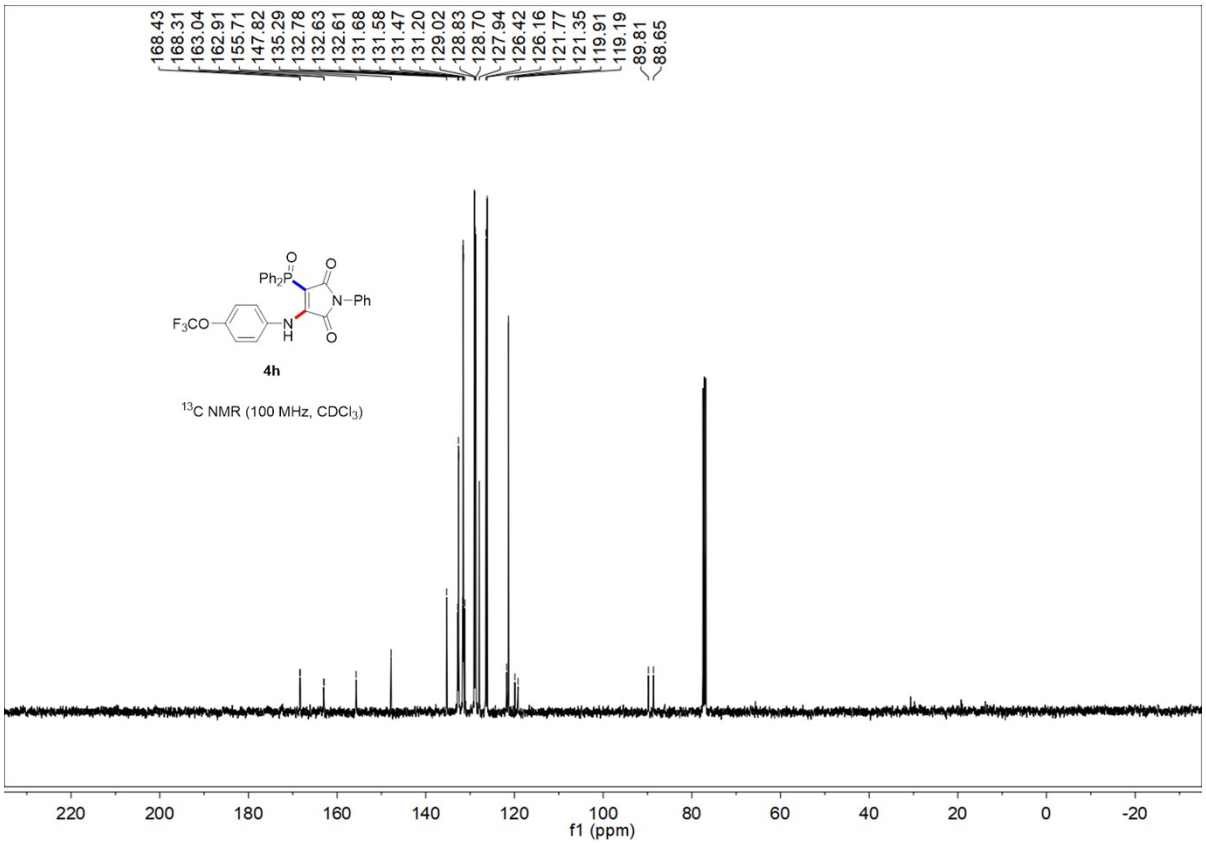

27.42
Ph2P
O
O
N–Ph
N
H
O
CO2Me
4f
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

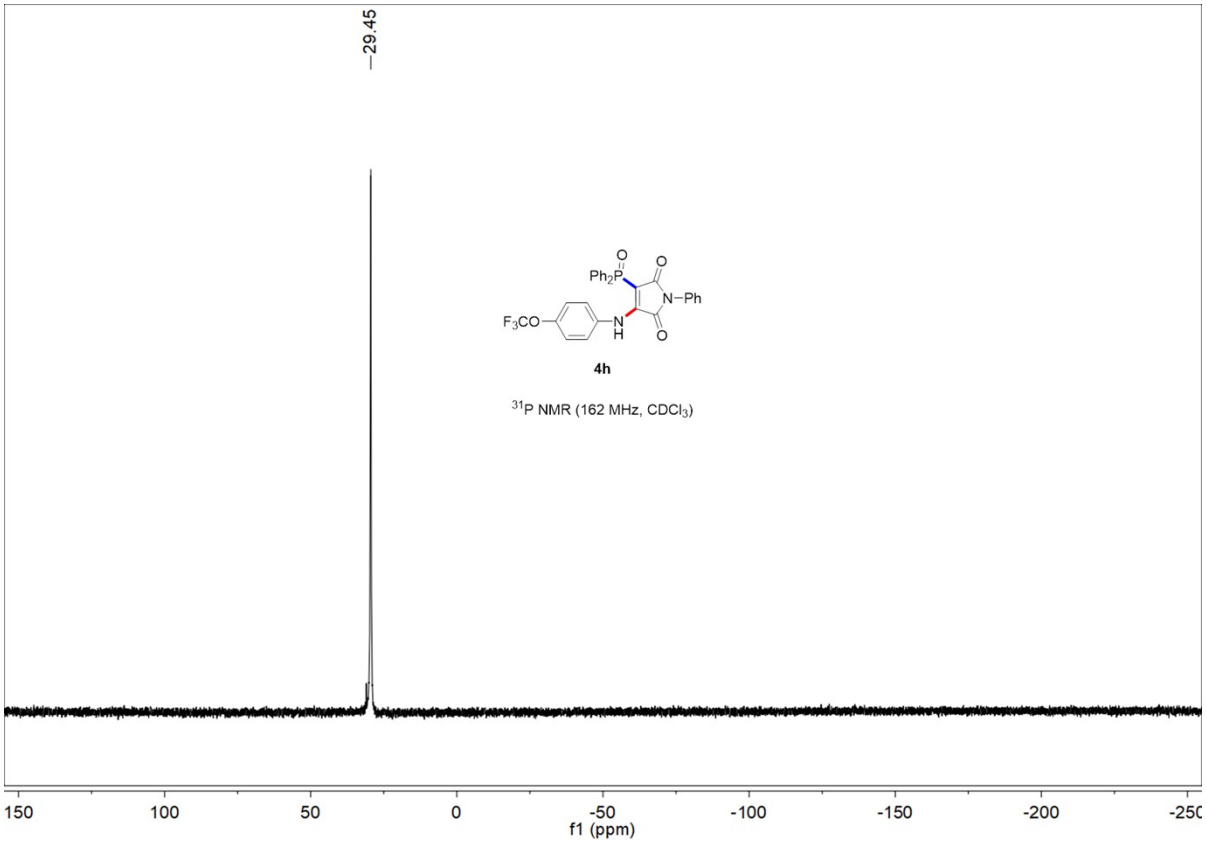

10.67
7.93
7.91
7.90
7.89
7.87
7.87
7.63
7.62
7.61
7.61
7.61
7.60
7.57
7.56
7.55
7.55
7.55
7.54
7.53
7.53
7.53
7.52
7.45
7.45
7.44
7.43
7.42
7.41
7.40
7.40
7.38
7.37
7.36
7.36
7.31
7.31
7.30
7.29
7.27
7.25
7.25
7.21
7.20
7.19
7.18
7.17
7.16
7.15
Ph2P
O
O
N–Ph
N
H
O
Ph
4g
1H NMR (400 MHz, CDCl3)
0.94
4.00
2.10
4.06
8.17
2.57
4.00
f1 (ppm)

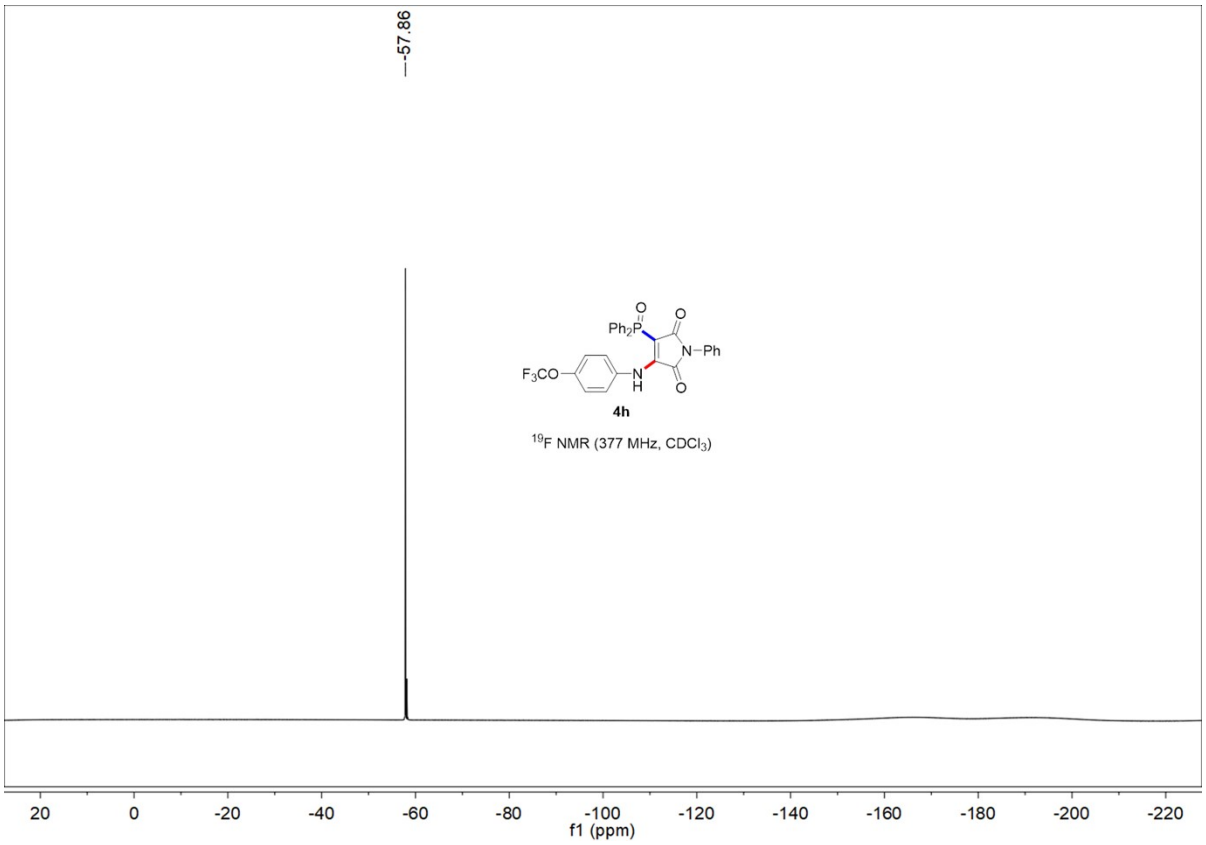

168.42
168.30
162.87
162.74
157.21
138.91
138.37
135.02
133.06
132.41
132.38
131.97
131.60
131.49
131.21
130.77
130.05
128.80
128.67
128.54
128.50
128.31
128.12
127.61
127.33
126.58
126.04
88.32
87.14
Ph2P
O
O
N–Ph
N
H
O
Ph
4g
13C NMR (100 MHz, CDCl3)
f1 (ppm)

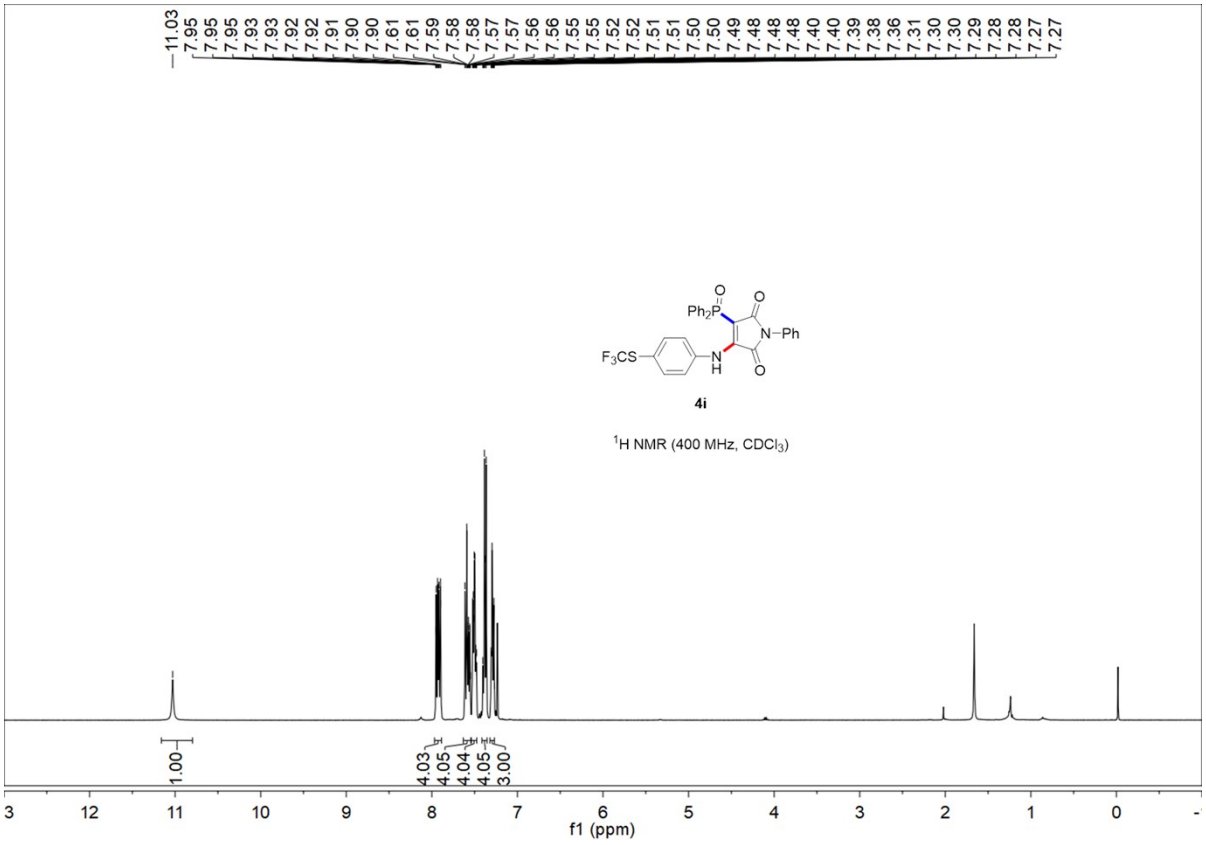

28.94
Ph2P
O
O
N–Ph
N
H
O
Ph
4g
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

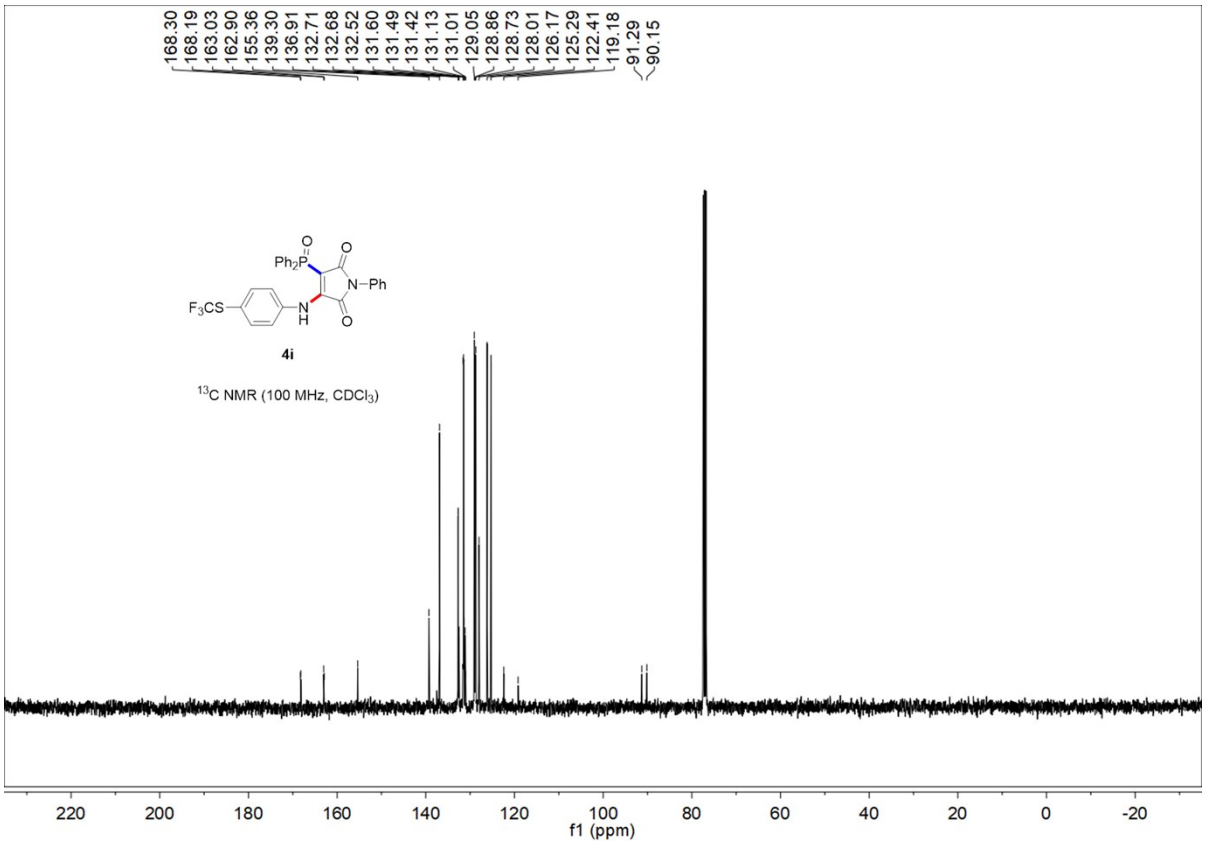

10.96
8.03
8.01
8.00
7.98
7.65
7.65
7.64
7.63
7.62
7.61
7.59
7.58
7.57
7.56
7.55
7.54
7.46
7.44
7.42
7.41
7.39
7.36
7.34
7.33
7.25
7.22
Ph2P
O
O
N–Ph
F3CO
N
H
O
4h
1H NMR (400 MHz, CDCl3)
0.96
4.00
2.06
4.18
7.19
2.12
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

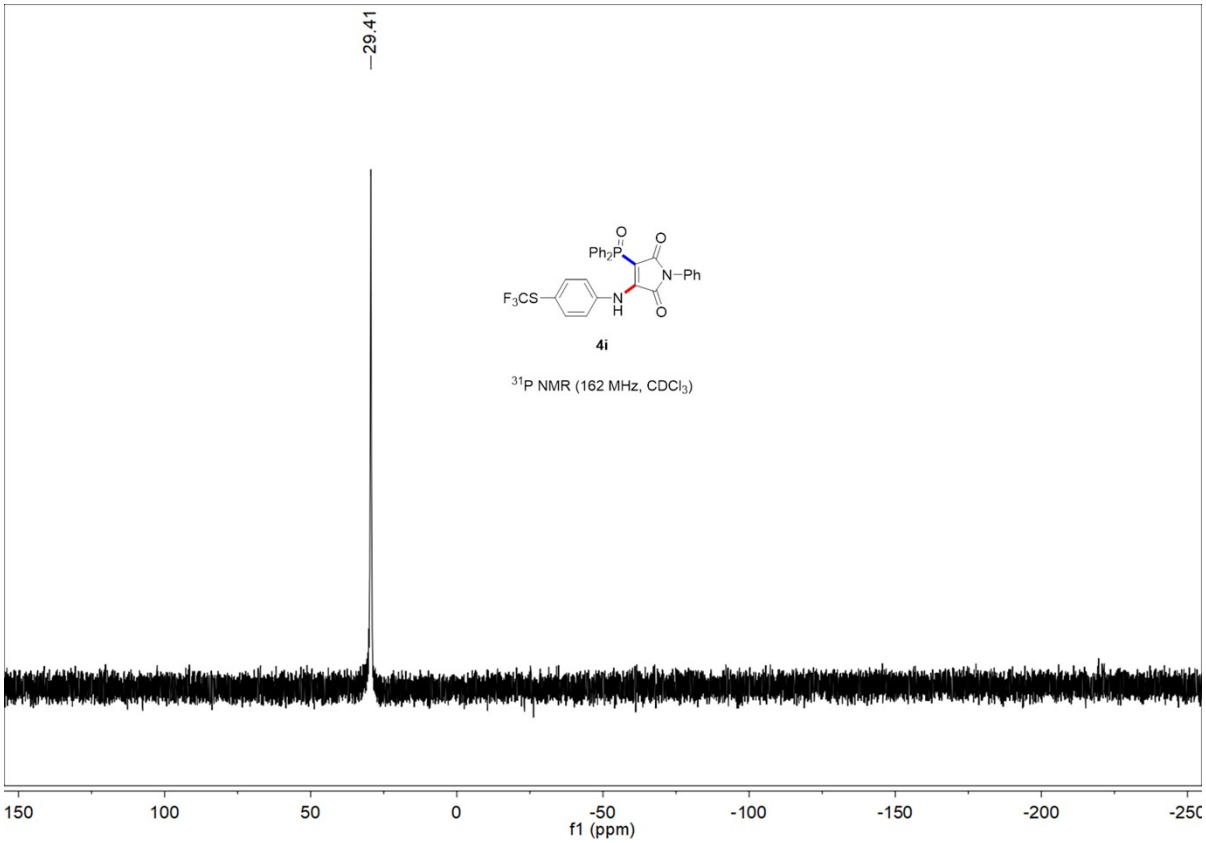

168.43
168.31
163.04
162.91
155.71
147.82
135.29
132.78
132.63
132.61
131.68
131.58
131.47
131.20
129.02
128.83
128.70
127.94
126.42
126.16
121.77
121.35
119.91
119.19
89.81
88.65
Ph2P
O
O
N–Ph
F3CO
N
H
O
4h
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

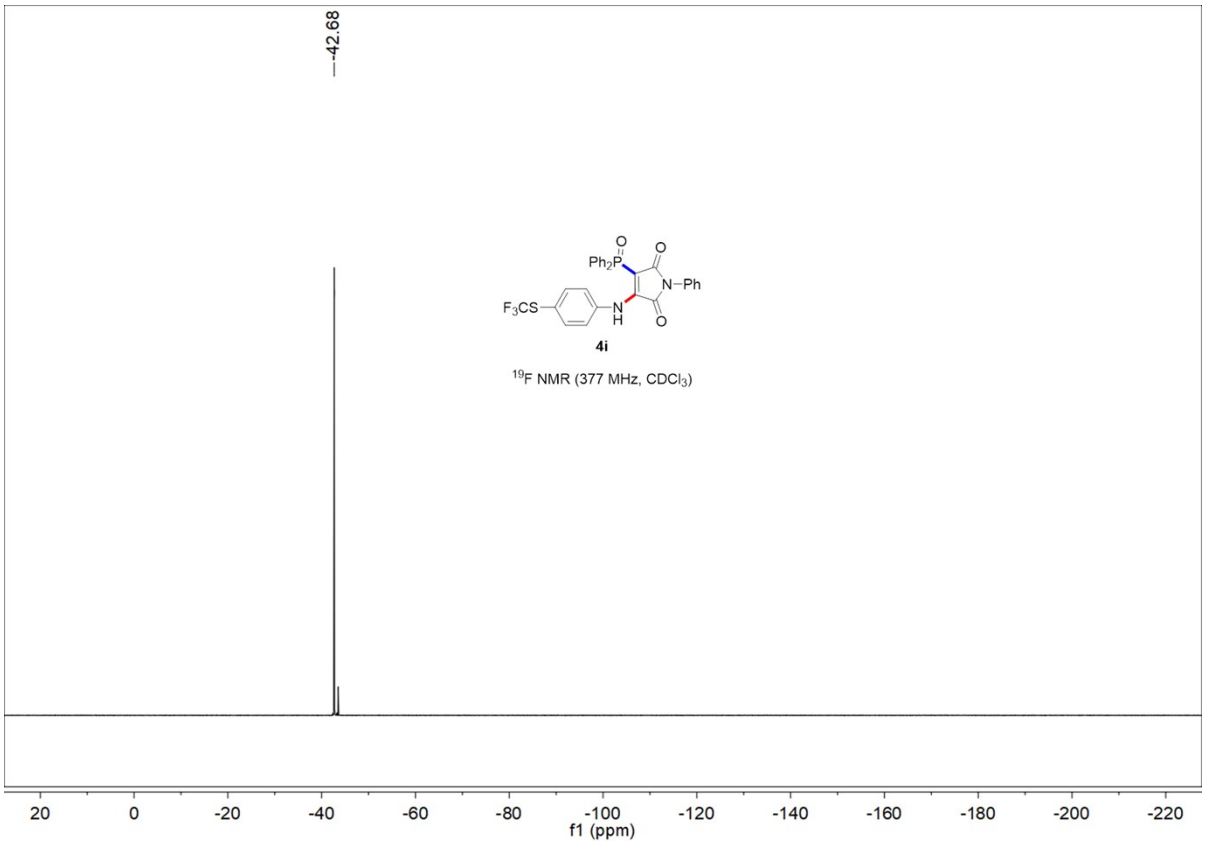

29.45
Ph2P
O
O
N–Ph
F3CO
N
H
O
4h
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

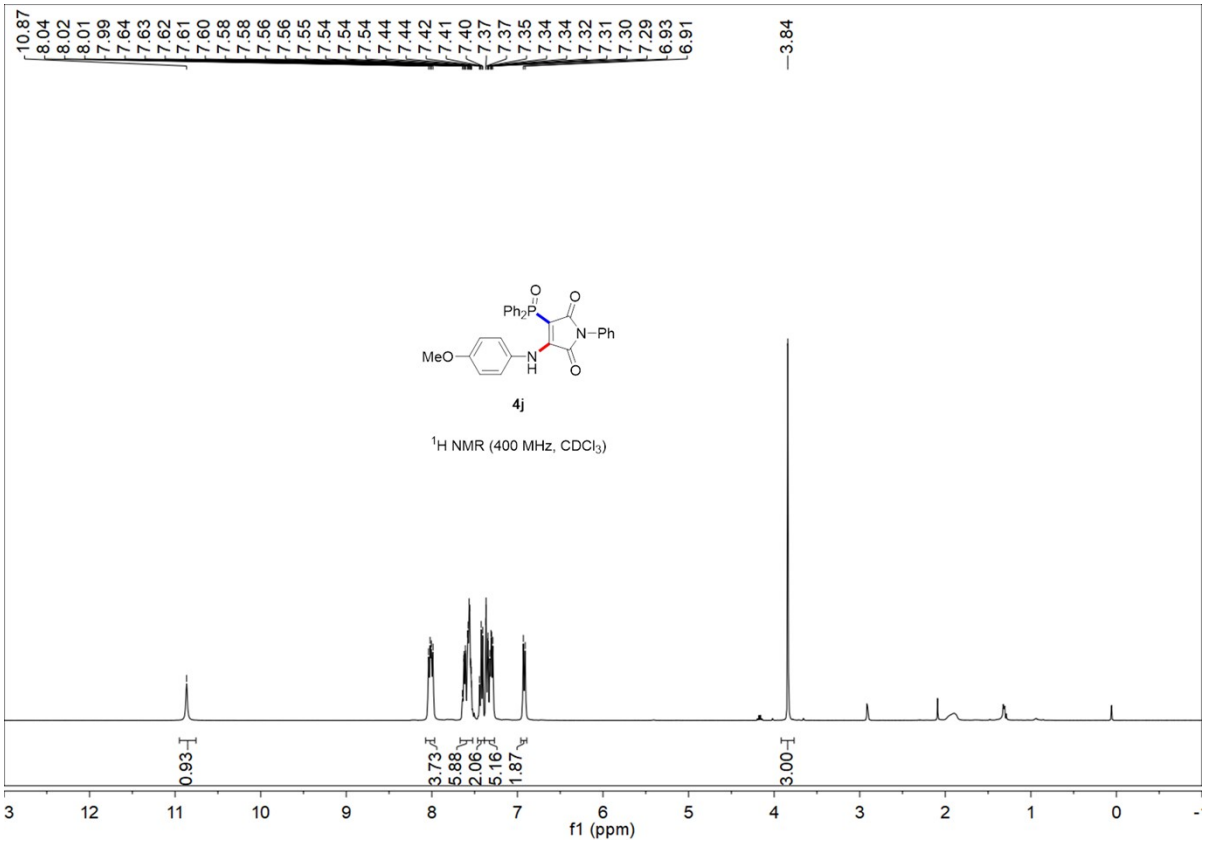

-57.86
Ph2P
O
O
N–Ph
F3CO
N
H
O
4h
19F NMR (377 MHz, CDCl3)
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

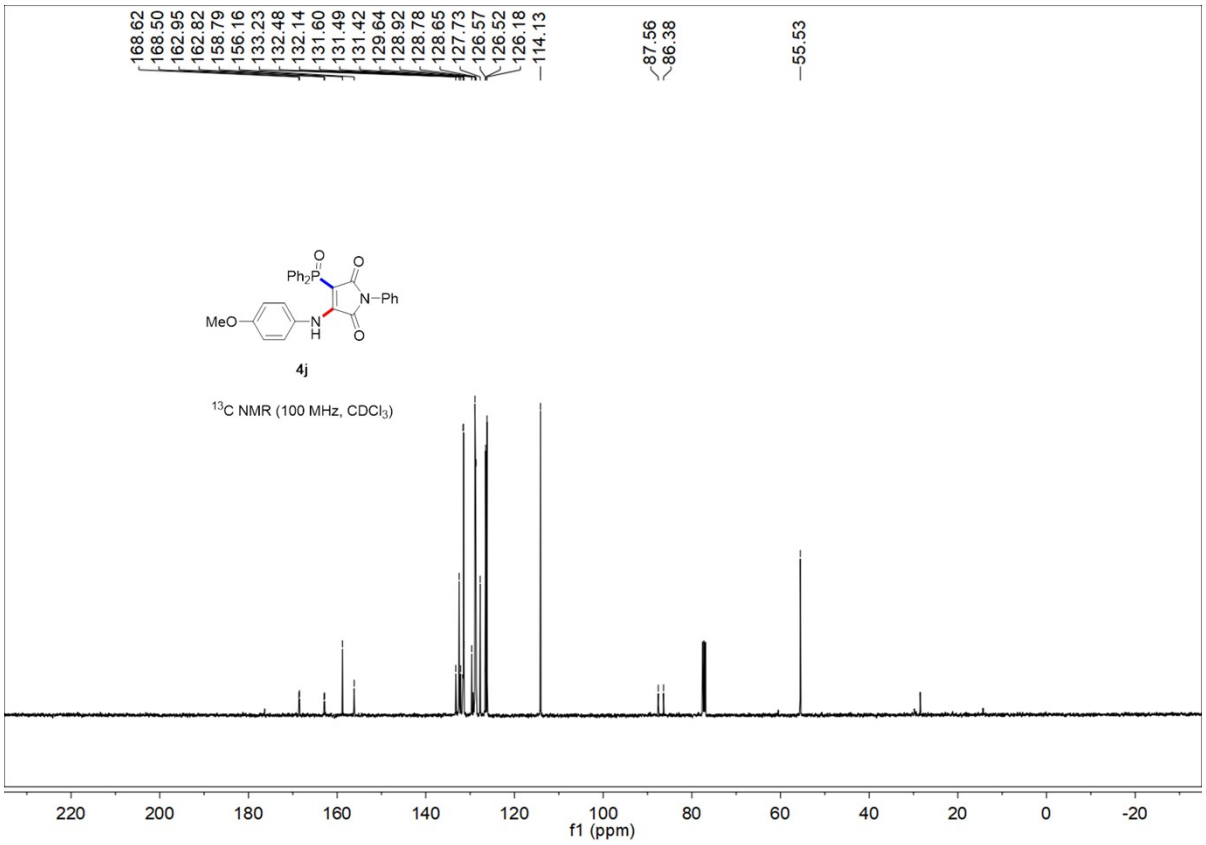

11.03
7.95
7.95
7.93
7.93
7.92
7.92
7.91
7.90
7.90
7.61
7.59
7.58
7.58
7.57
7.57
7.56
7.56
7.55
7.55
7.52
7.52
7.51
7.51
7.50
7.50
7.49
7.48
7.48
7.48
7.40
7.39
7.38
7.36
7.31
7.30
7.30
7.29
7.28
7.28
7.27
7.27
Ph2P
O
O
N–Ph
F3CS
N
H
O
4i
1H NMR (400 MHz, CDCl3)
1.00
4.03
4.05
4.04
4.05
3.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

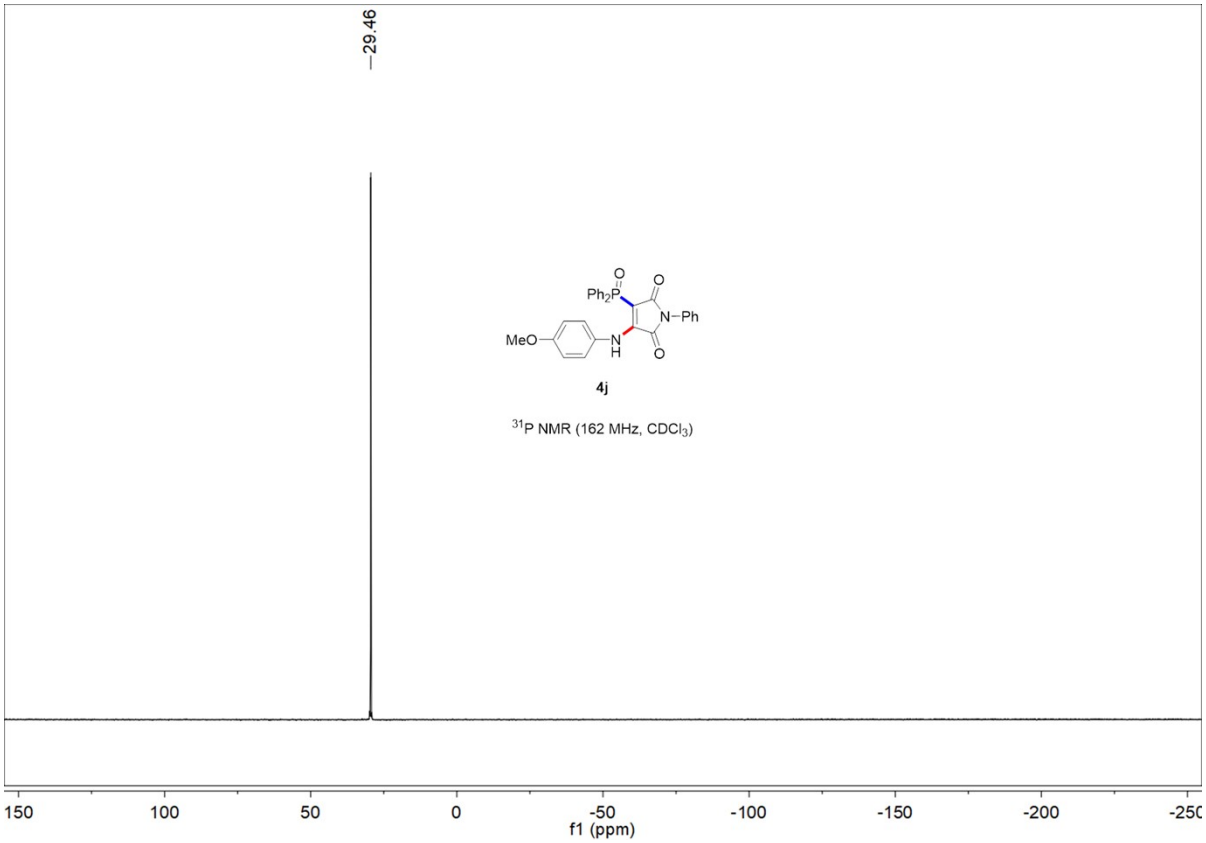

168.30
168.19
163.03
162.90
155.36
139.30
136.91
132.71
132.68
132.52
131.60
131.49
131.42
131.13
131.01
129.05
128.86
128.73
128.01
126.17
125.29
122.41
119.18
91.29
90.15
Ph2P
O
O
N–Ph
F3CS
N
H
O
4i
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

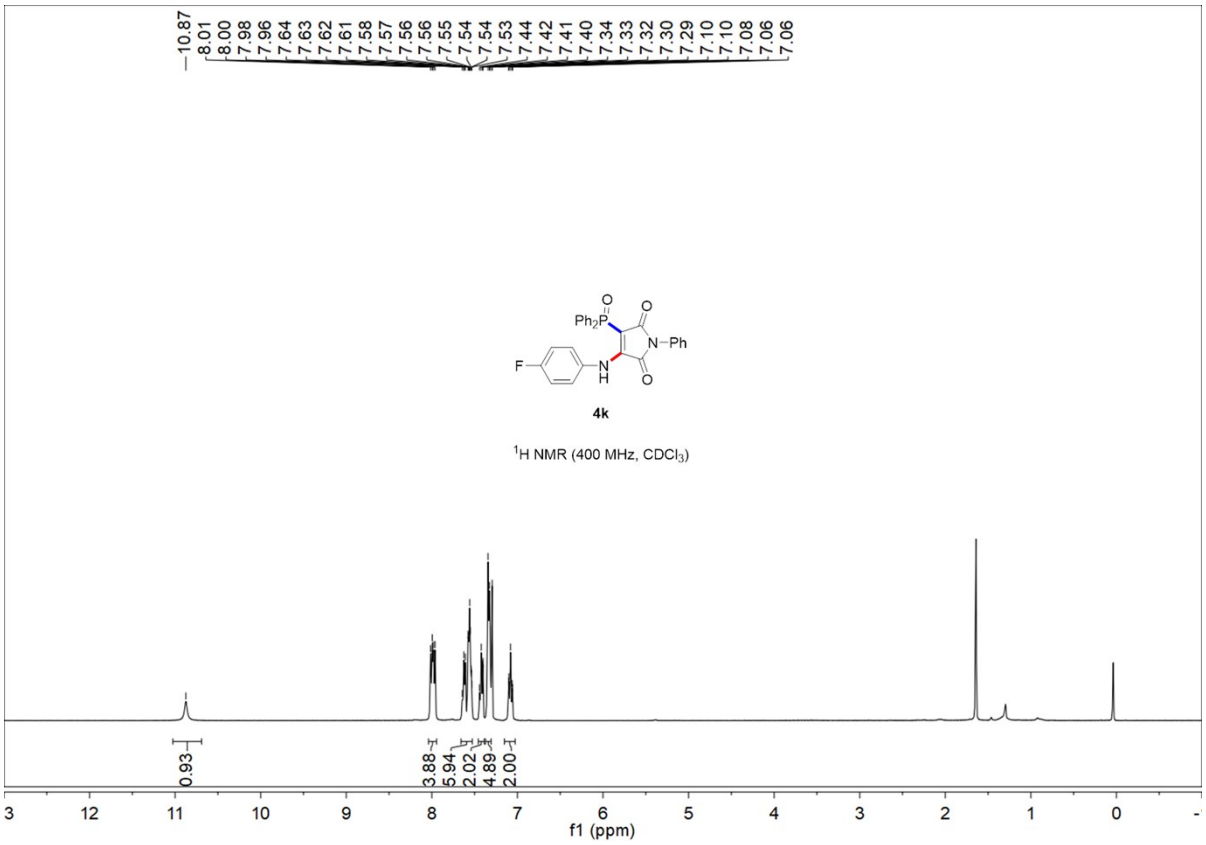

29.41
Ph2P
O
O
N–Ph
F3CS
N
H
O
4i
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

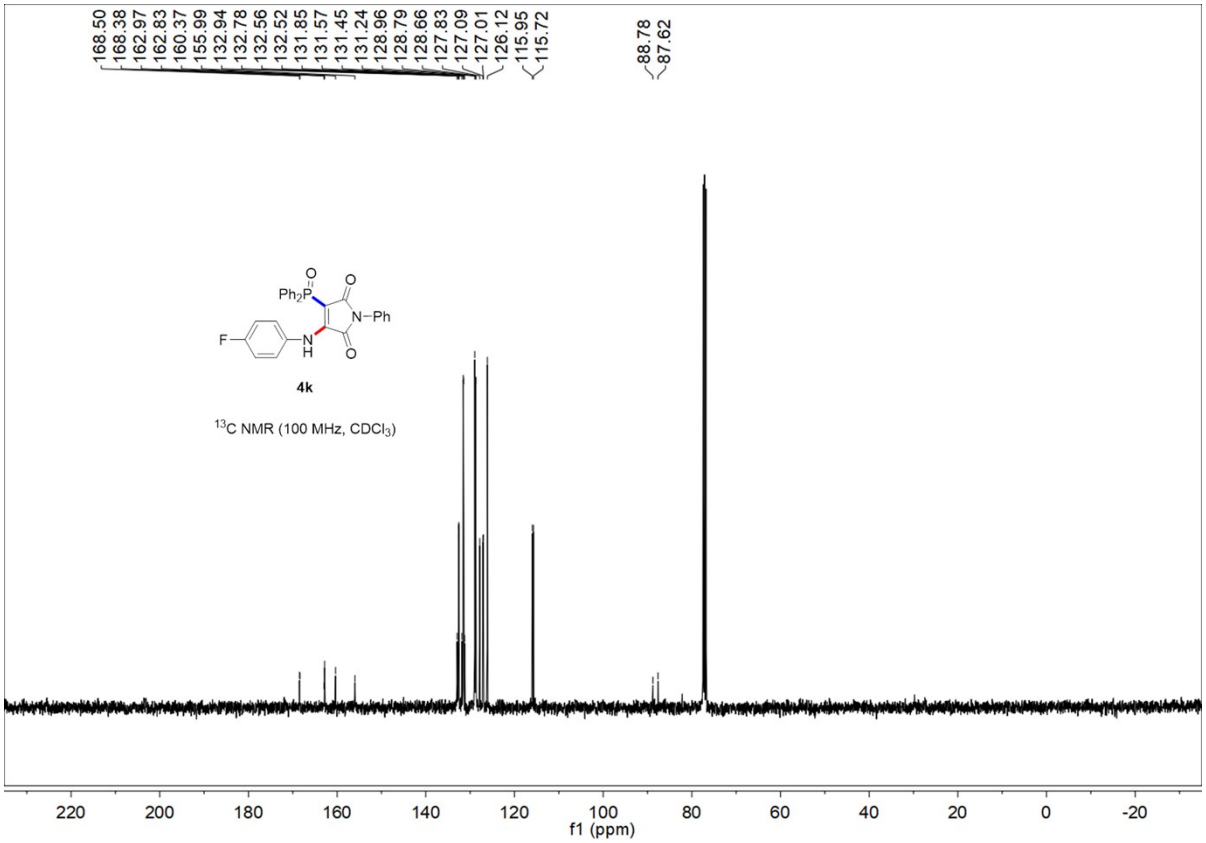

-42.68
Ph2P
O
O
N–Ph
F3CS
N
H
O
4i
19F NMR (377 MHz, CDCl3)
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

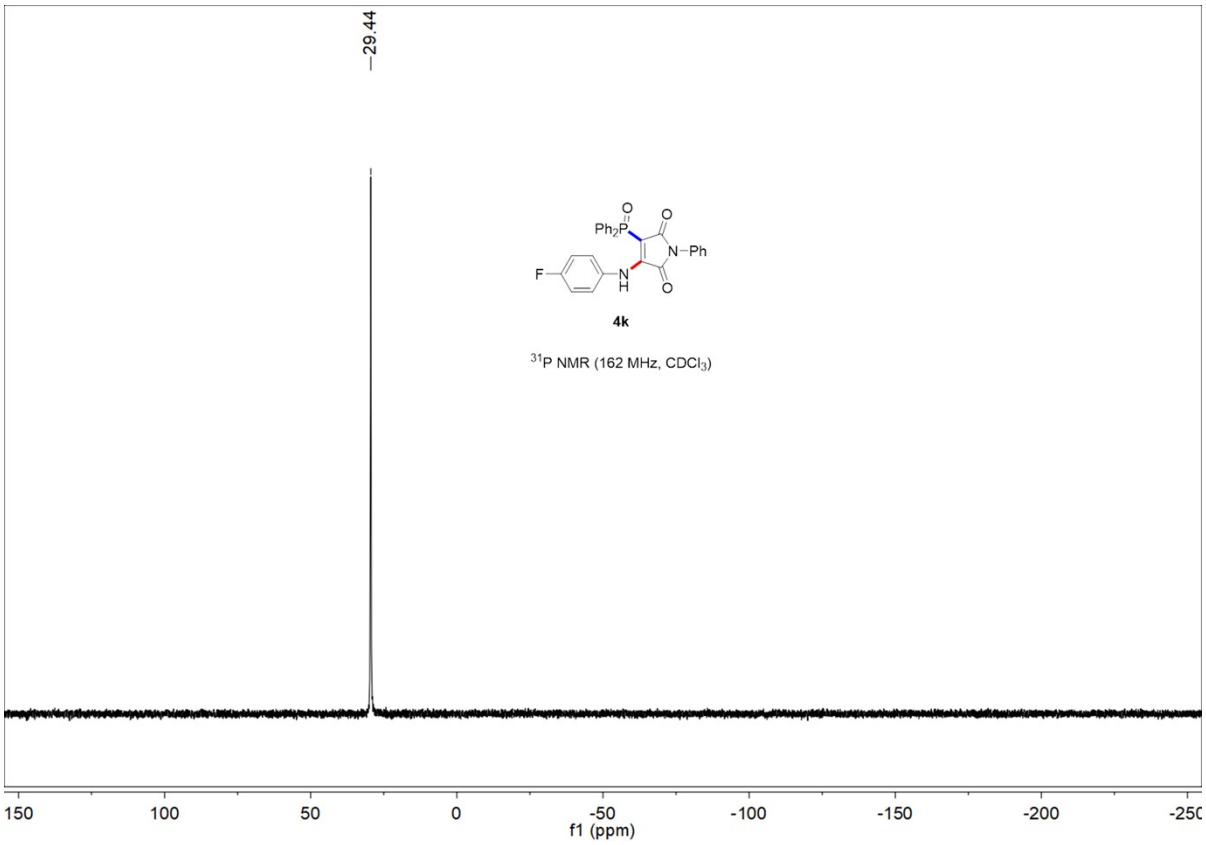

10.87
8.04
8.02
8.01
7.99
7.64
7.63
7.62
7.61
7.60
7.58
7.58
7.56
7.56
7.55
7.54
7.54
7.54
7.44
7.44
7.42
7.41
7.40
7.37
7.37
7.35
7.34
7.34
7.32
7.31
7.30
7.29
6.93
6.91
3.84
Ph2P
O
O
N–Ph
MeO
N
H
O
4j
1H NMR (400 MHz, CDCl3)
0.93
3.73
5.88
2.06
5.16
1.87
3.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

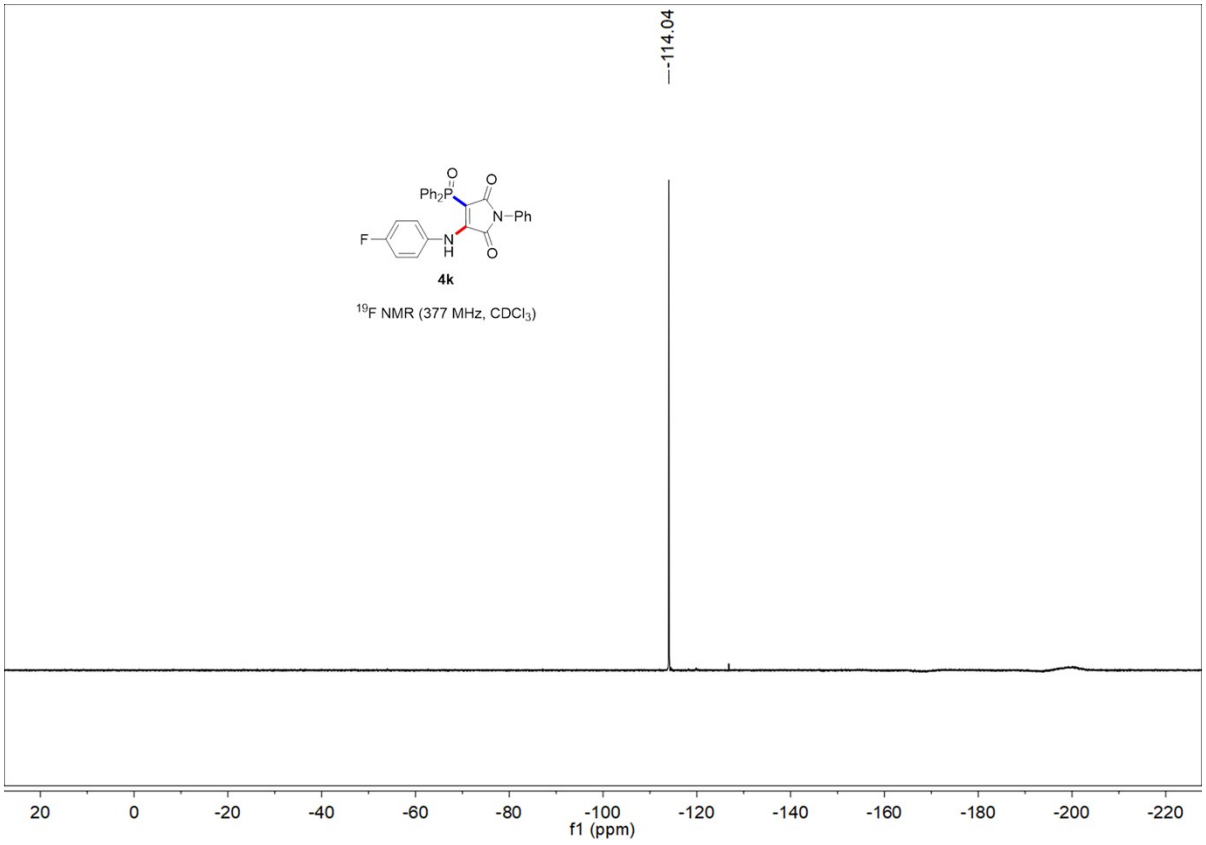

168.62
168.50
162.95
162.82
158.79
156.16
133.23
132.48
132.14
131.60
131.49
131.42
129.64
128.92
128.78
128.65
127.73
126.57
126.52
126.18
114.13
87.56
86.38
55.53
Ph2P
O
O
N–Ph
MeO
N
H
O
4j
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

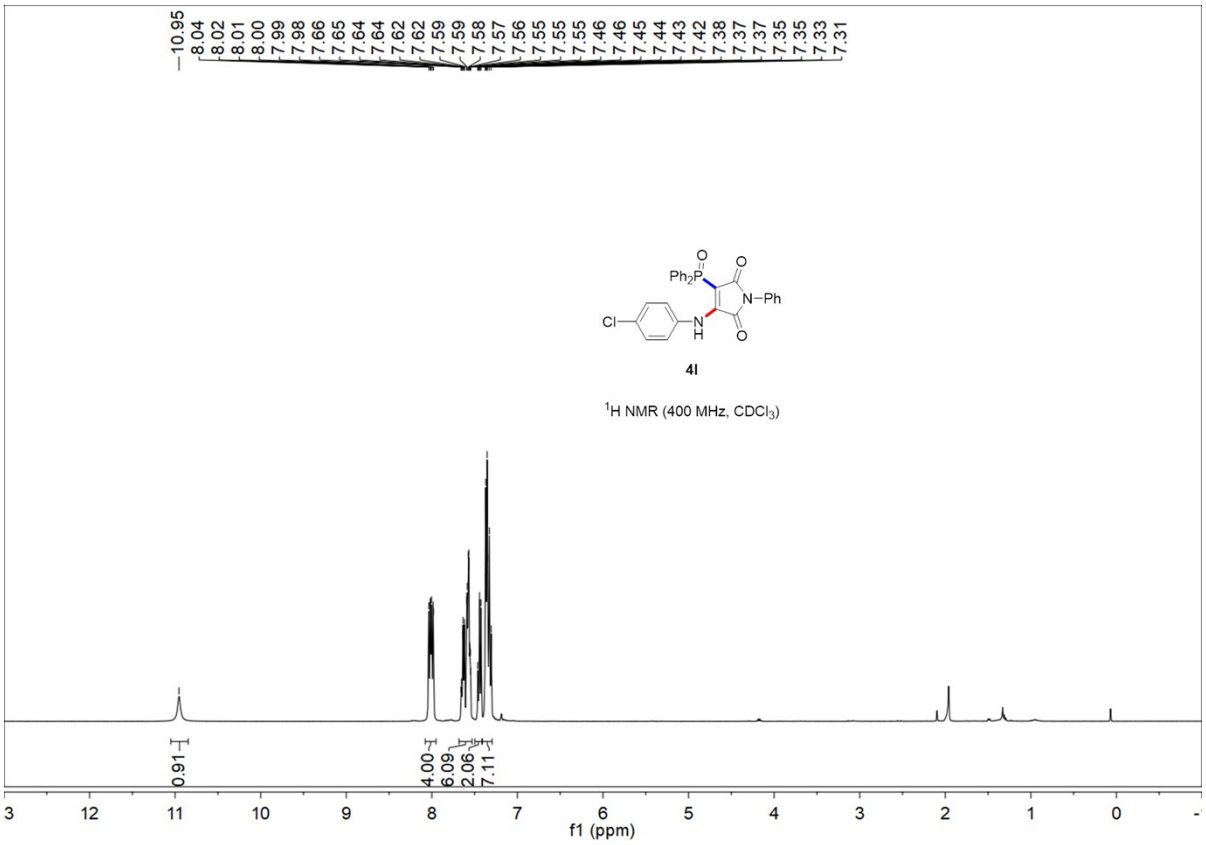

29.46
Ph2P
O
O
N–Ph
MeO
N
H
O
4j
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

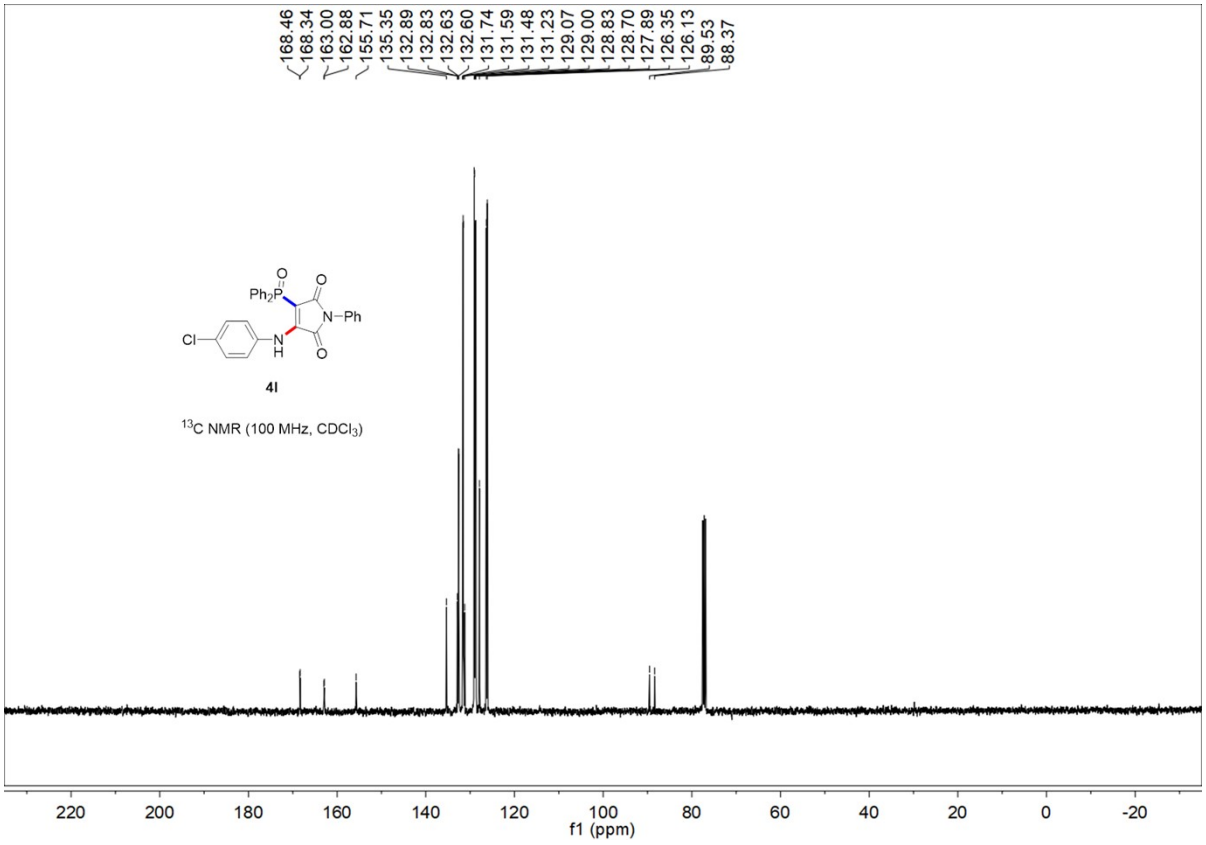

10.87
8.01
8.00
7.98
7.96
7.64
7.63
7.62
7.61
7.58
7.57
7.56
7.56
7.55
7.54
7.54
7.53
7.44
7.42
7.41
7.40
7.34
7.33
7.32
7.30
7.29
7.10
7.10
7.08
7.06
7.06
Ph2P
O
O
N–Ph
F
N
H
O
4k
1H NMR (400 MHz, CDCl3)
0.93
3.88
5.94
2.02
4.89
2.00
f1 (ppm)

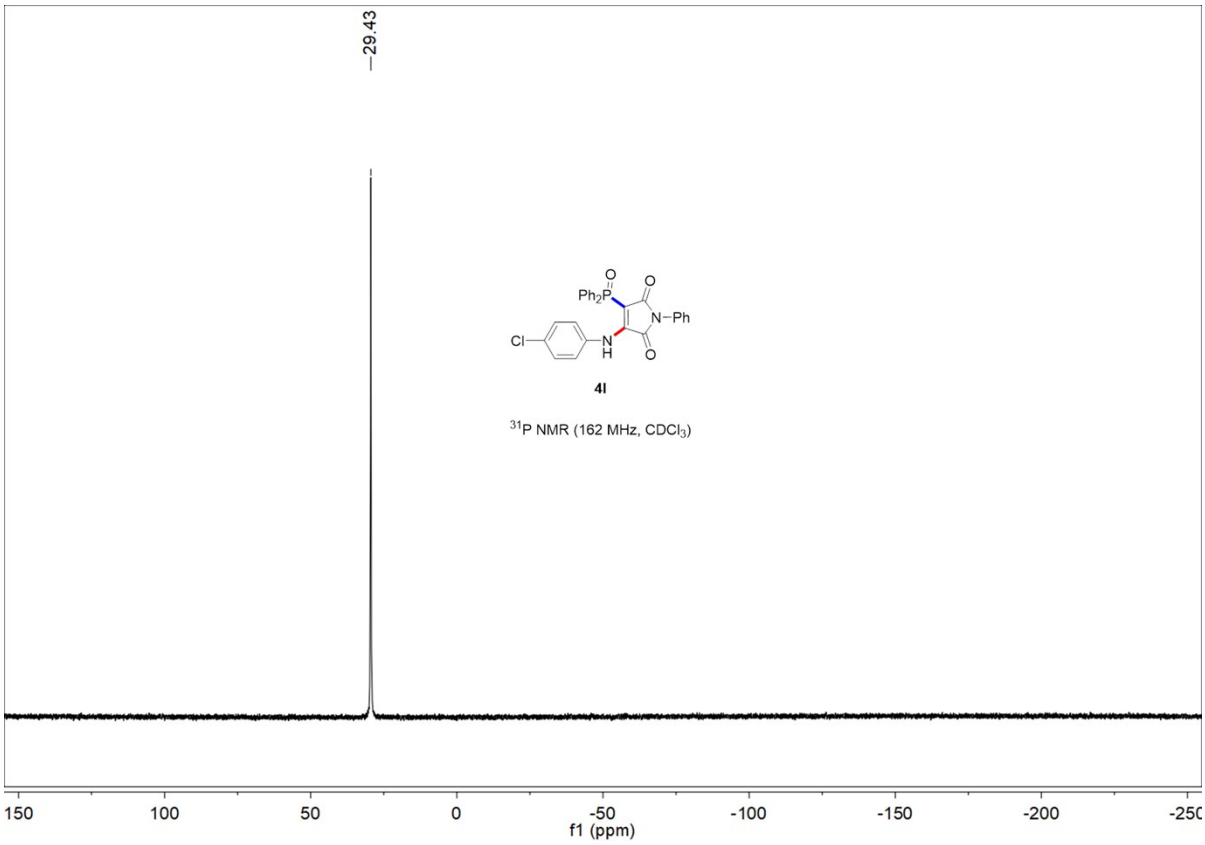

168.50
168.38
162.97
162.83
160.37
155.99
132.94
132.78
132.56
132.52
131.85
131.57
131.45
131.24
128.96
128.79
128.66
127.83
127.09
127.01
126.12
115.95
115.72
88.78
87.62
Ph2P
O
O
N–Ph
F
N
H
O
4k
13C NMR (100 MHz, CDCl3)
f1 (ppm)


29.44
Ph2P
O
N–Ph
F
N
H
O
O
4k
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)


-114.04
Ph2P
O
N–Ph
F
N
H
O
O
4k
19F NMR (377 MHz, CDCl3)
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

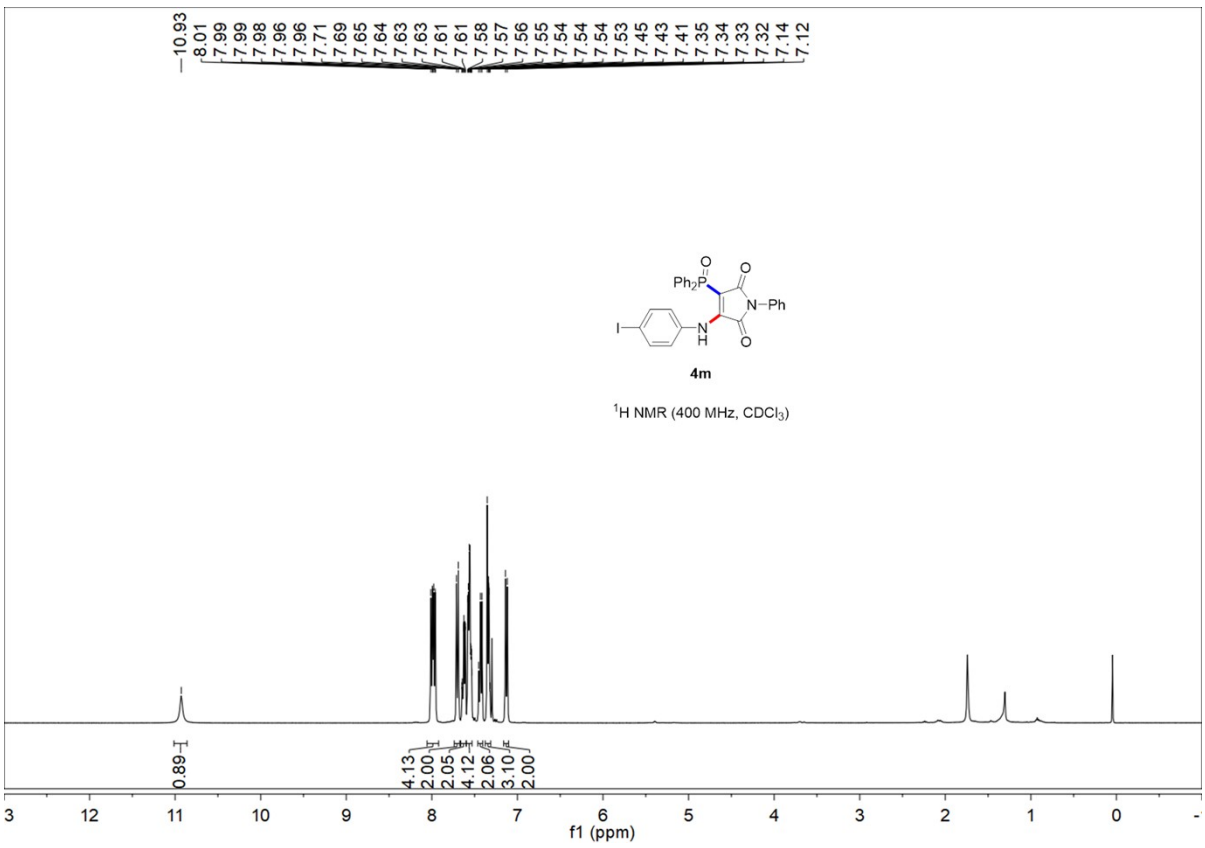

10.95
8.04
8.02
8.01
8.00
7.99
7.98
7.66
7.65
7.64
7.64
7.62
7.62
7.59
7.59
7.58
7.57
7.56
7.55
7.55
7.55
7.46
7.46
7.45
7.44
7.43
7.42
7.38
7.37
7.37
7.35
7.35
7.33
7.31
Ph2P
O
O
N–Ph
Cl
N
H
O
O
4l
1H NMR (400 MHz, CDCl3)
0.91
4.00
6.09
2.06
7.11
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


168.46
168.34
163.00
162.88
155.71
135.35
132.89
132.83
132.63
132.60
131.74
131.59
131.48
131.23
129.07
129.00
128.83
128.70
127.89
126.35
126.13
89.53
88.37
Ph2P
O
O
N–Ph
Cl
N
H
O
4l
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)



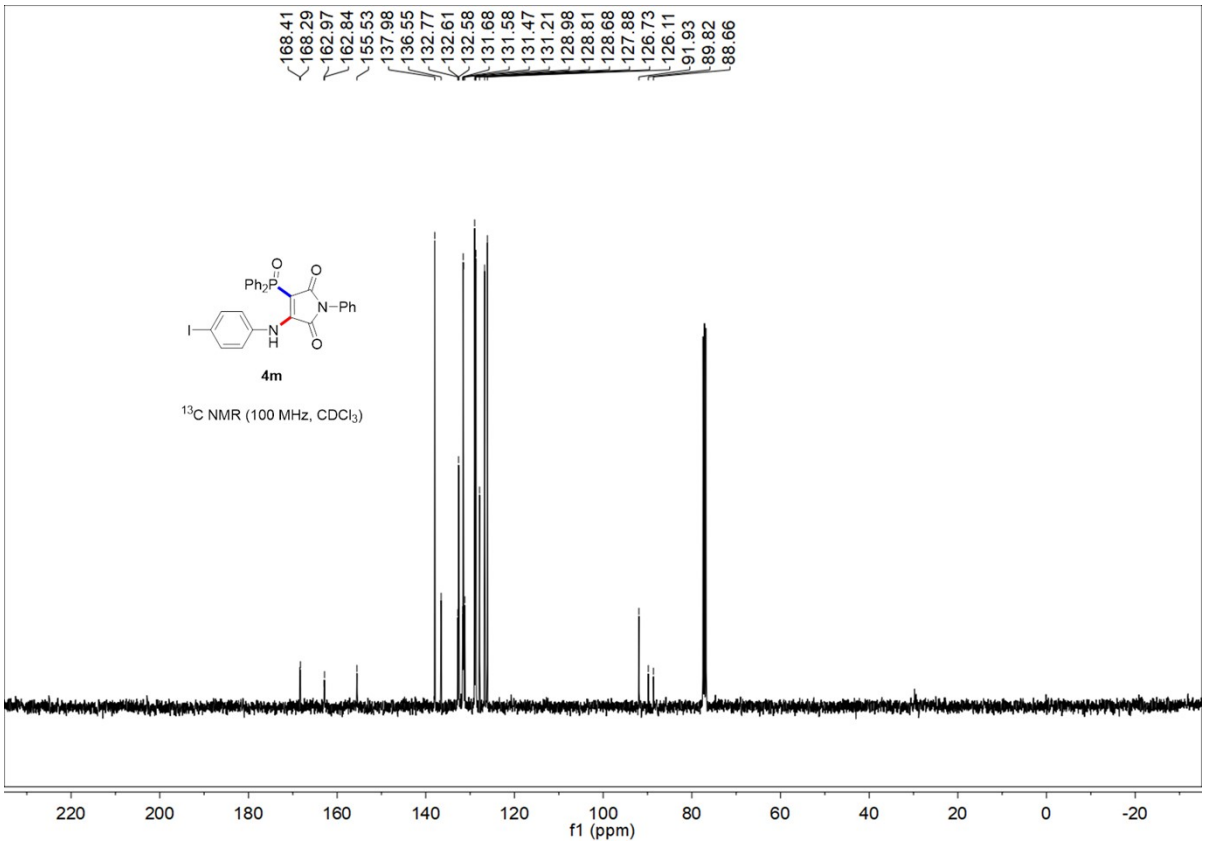
4l

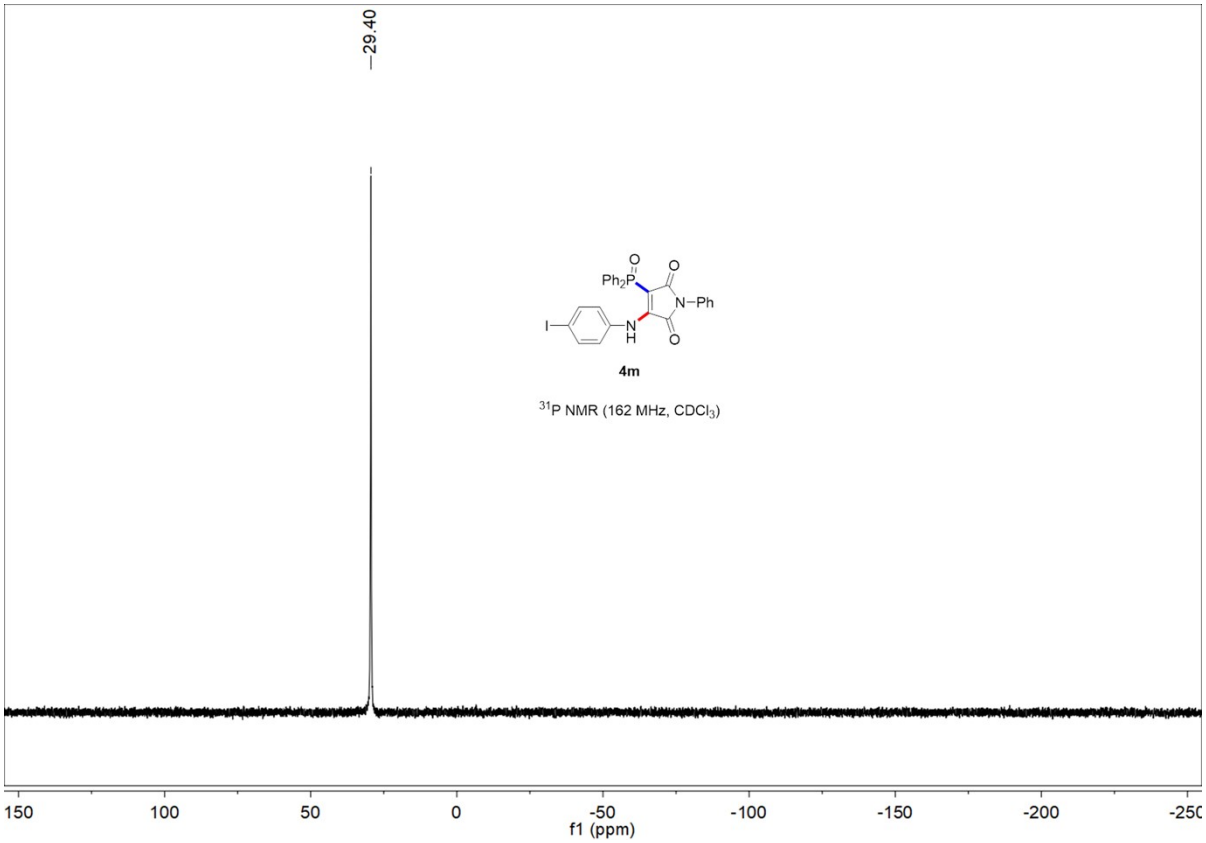
^{31}P NMR (162 MHz, $CDCl_3$)

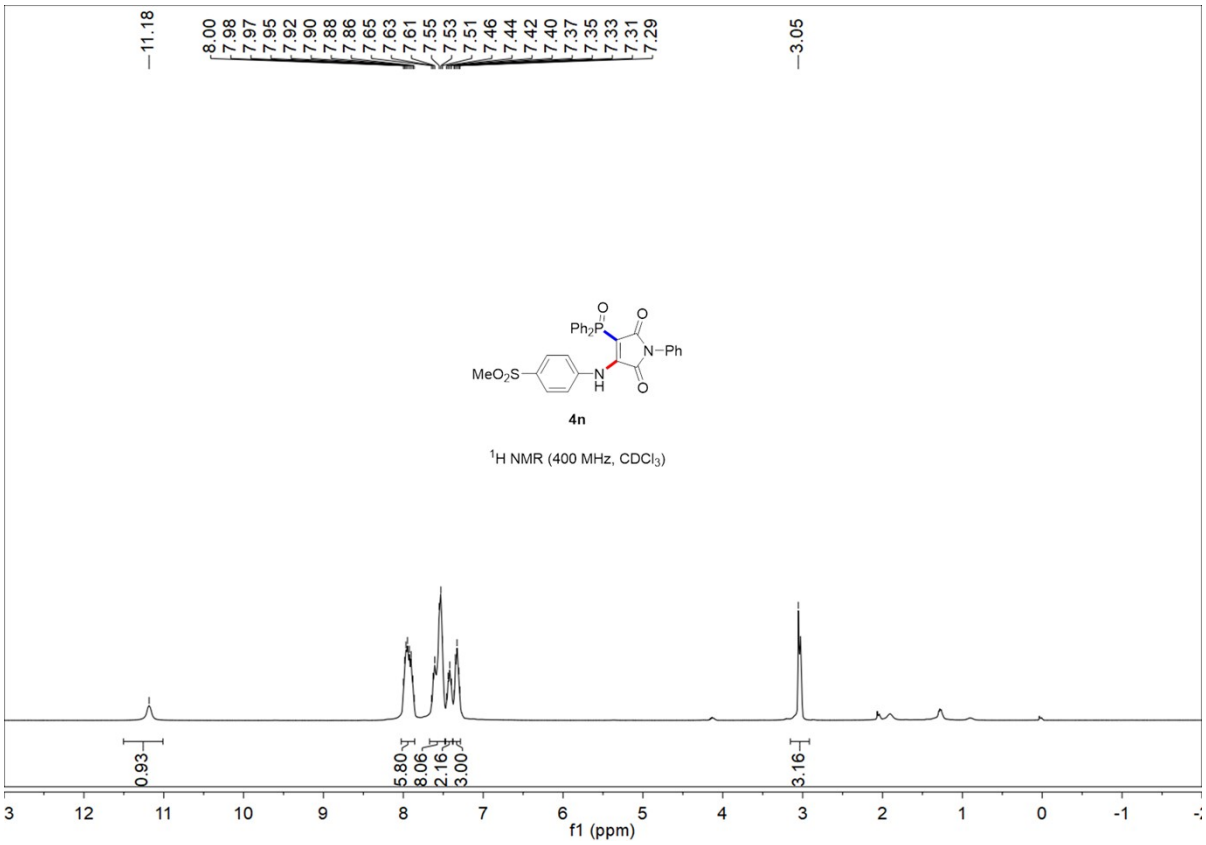


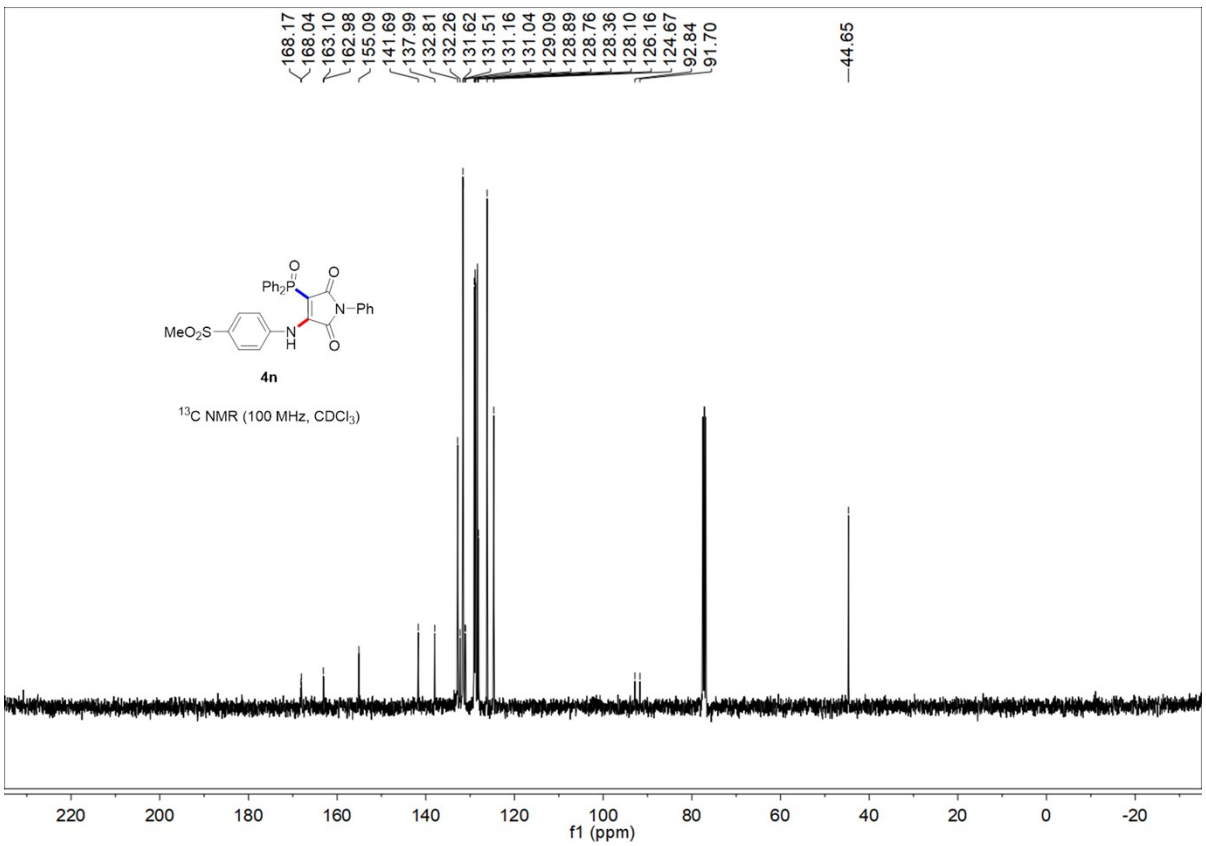
4m

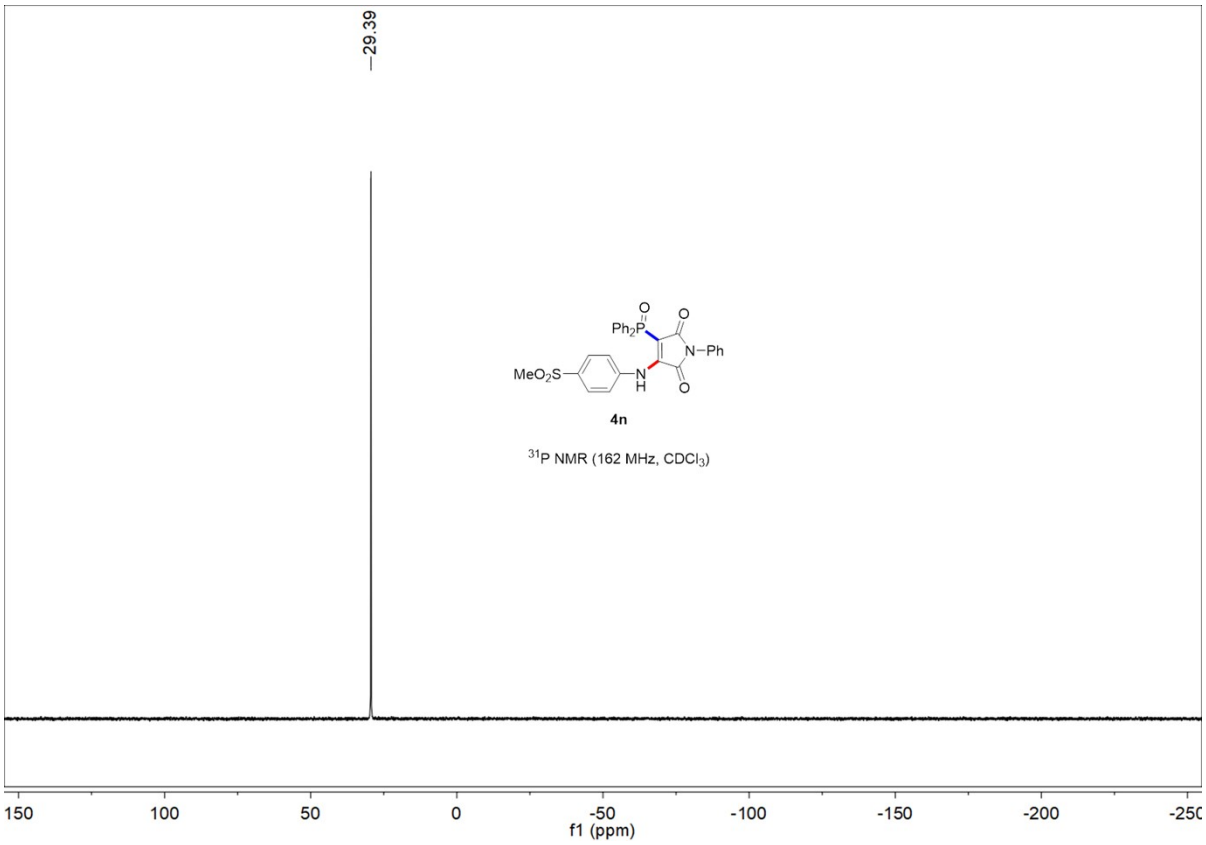
^{1}H NMR (400 MHz, $CDCl_3$)


168.41
168.29
162.97
162.84
155.53
137.98
136.55
132.77
132.61
132.58
131.68
131.58
131.47
131.21
128.98
128.81
128.68
127.88
126.73
126.11
91.93
89.82
88.66
Ph2P
O
O
N–Ph
I
N
H
O
4m
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)


29.40
Ph2P
O
O
N–Ph
I
N
H
O
4m
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)

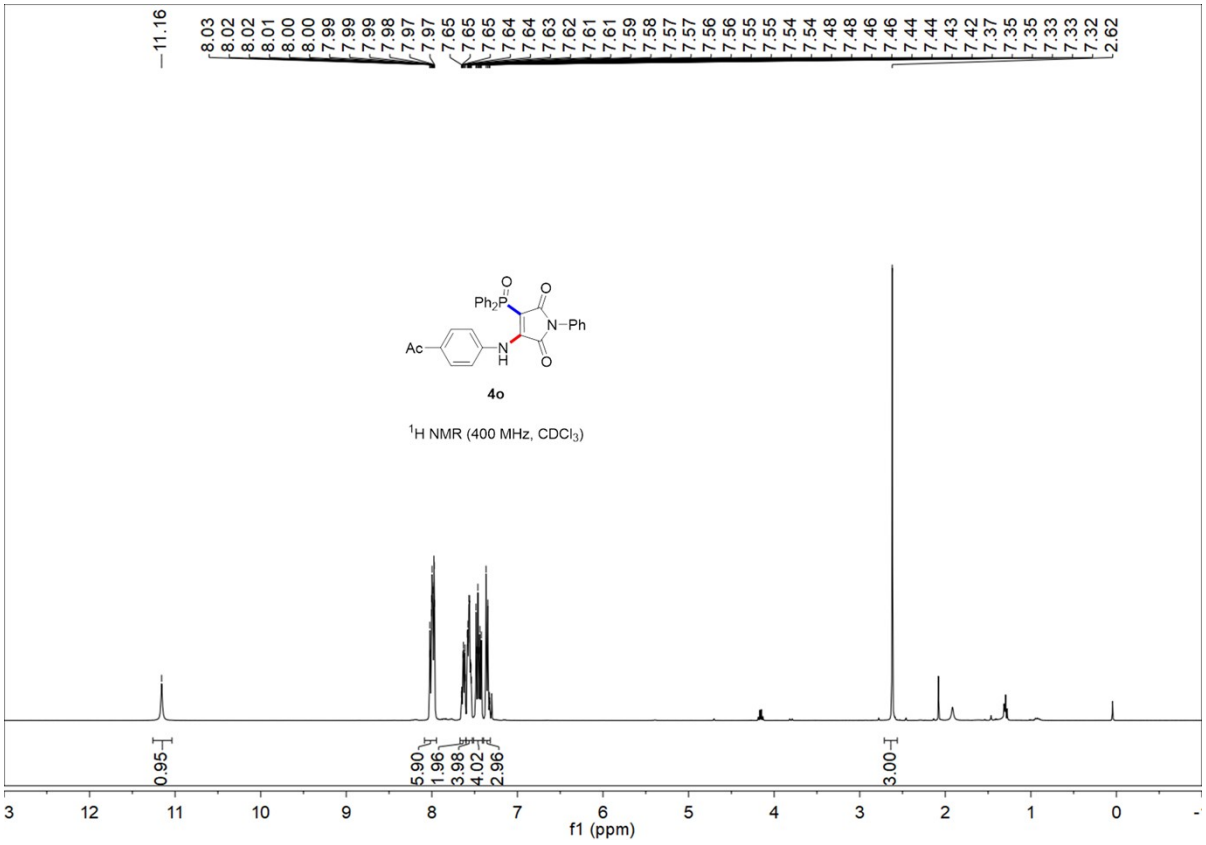

11.18
8.00
7.98
7.97
7.95
7.92
7.90
7.88
7.86
7.65
7.63
7.61
7.55
7.53
7.51
7.46
7.44
7.42
7.40
7.37
7.35
7.33
7.31
7.29
3.05
Ph2P
O
O
N–Ph
MeO2S
N
H
O
4n
1H NMR (400 MHz, CDCl3)
0.93
5.80
8.06
2.16
3.00
3.16
f1 (ppm)


168.17
168.04
163.10
162.98
155.09
141.69
137.99
132.81
132.26
131.62
131.51
131.16
131.04
129.09
128.89
128.76
128.36
128.10
126.16
124.67
92.84
91.70
44.65
Ph2P
O
O
N–Ph
MeO2S
N
H
O
4n
13C NMR (100 MHz, CDCl3)
f1 (ppm)



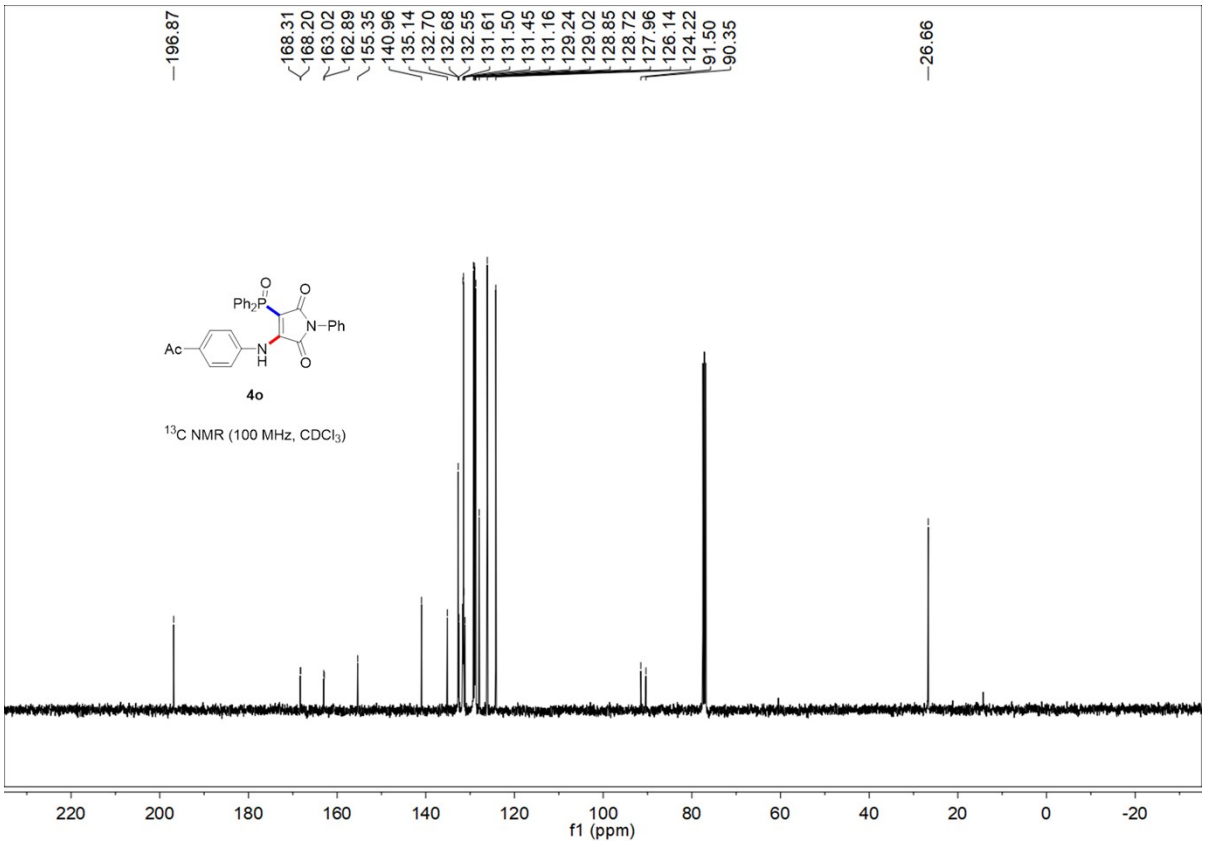
4n

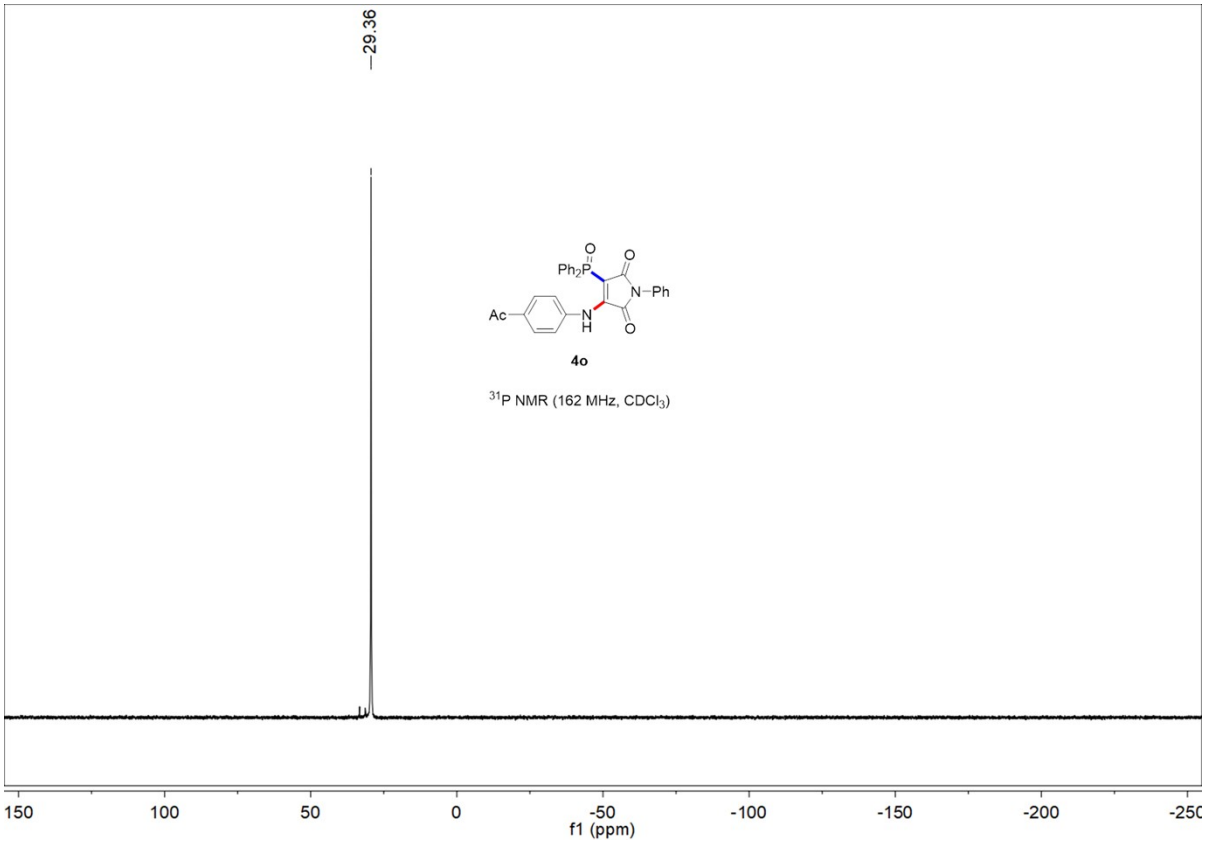
^{31}P NMR (162 MHz, $CDCl_3$)

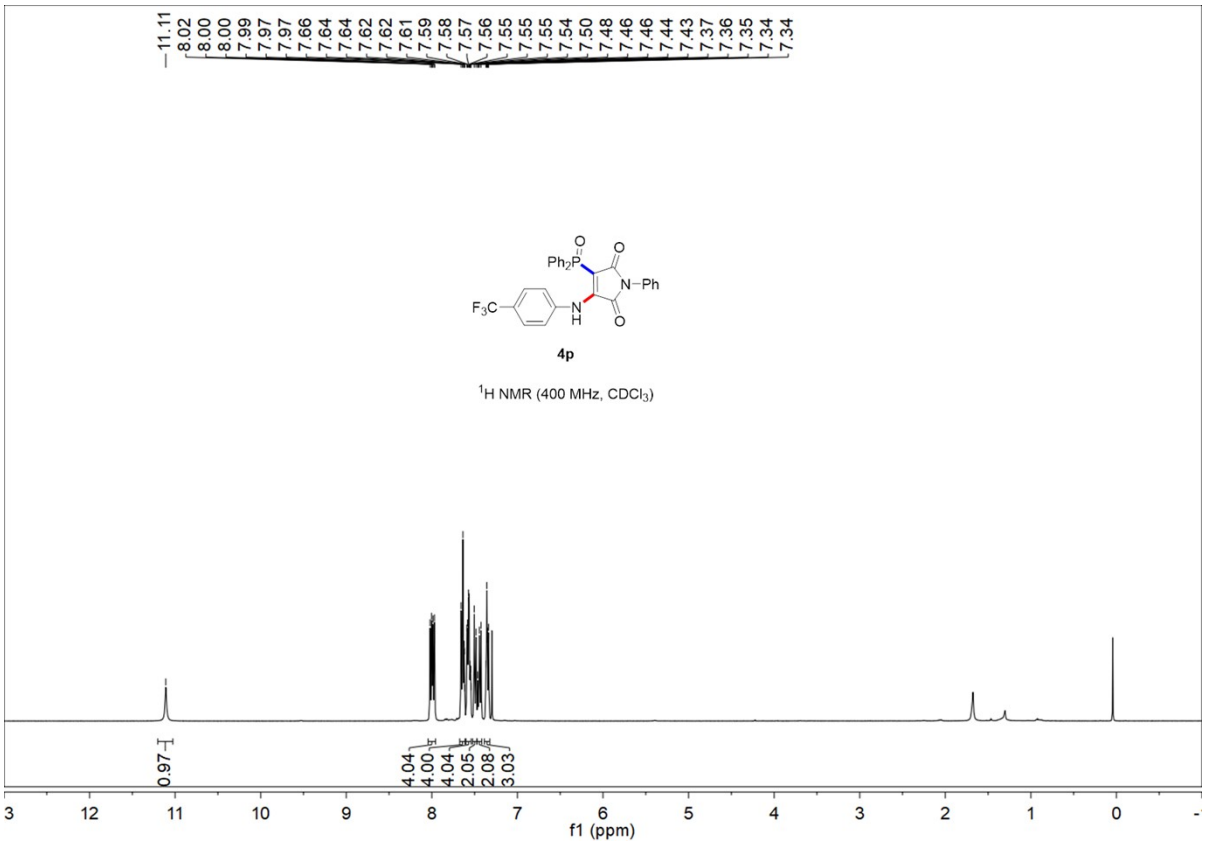


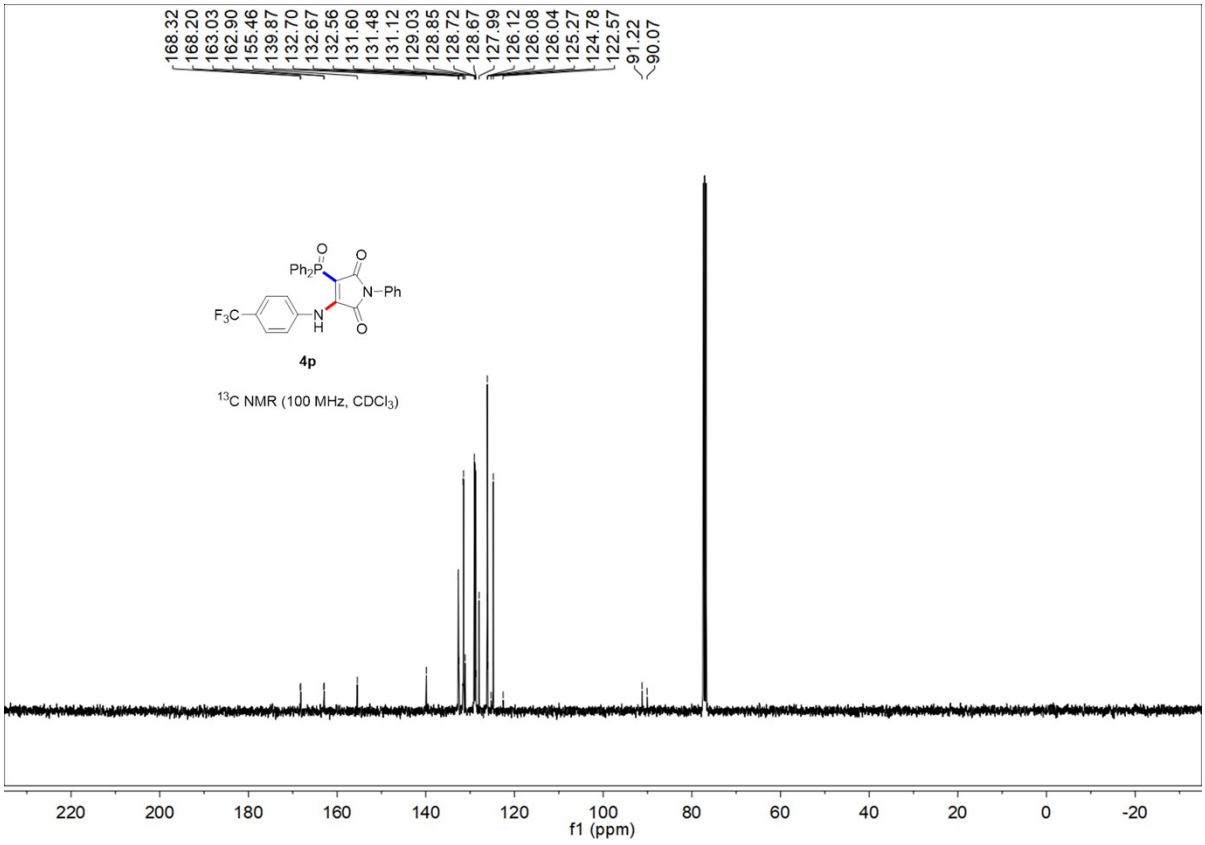
4o

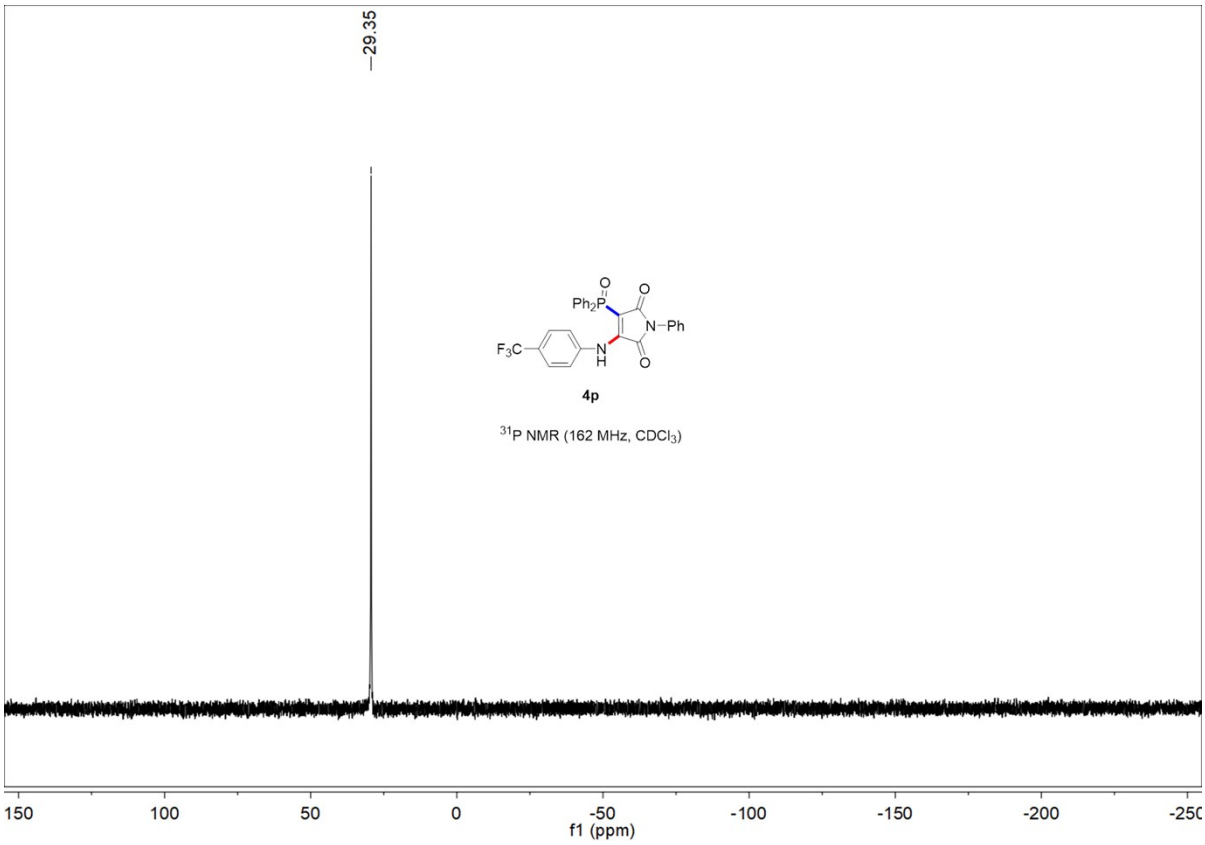
^{1}H NMR (400 MHz, $CDCl_3$)

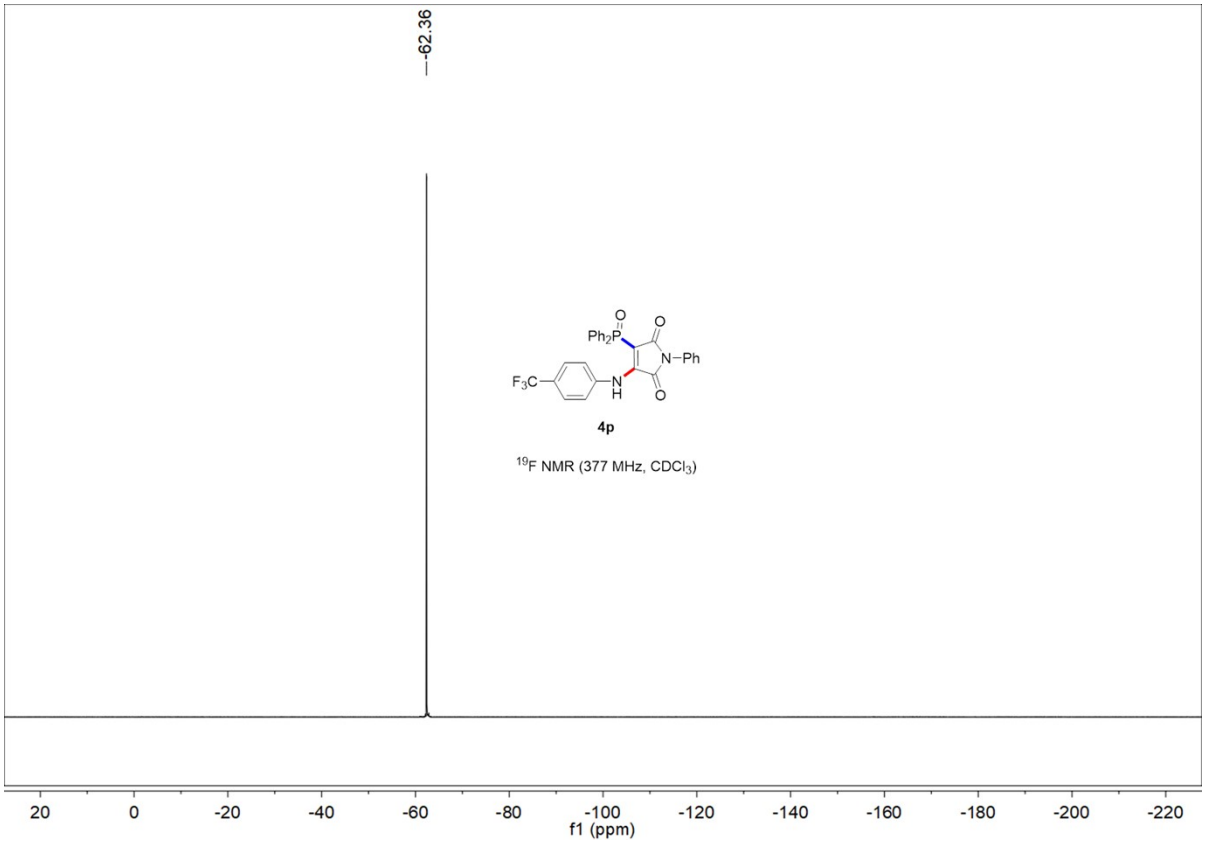

196.87
168.31
168.20
163.02
162.89
155.35
140.96
135.14
132.70
132.68
132.55
131.61
131.50
131.45
131.16
129.24
129.02
128.85
128.72
127.96
126.14
124.22
91.50
90.35
26.66
Ac
N
H
Ph2P
O
N–Ph
4o
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

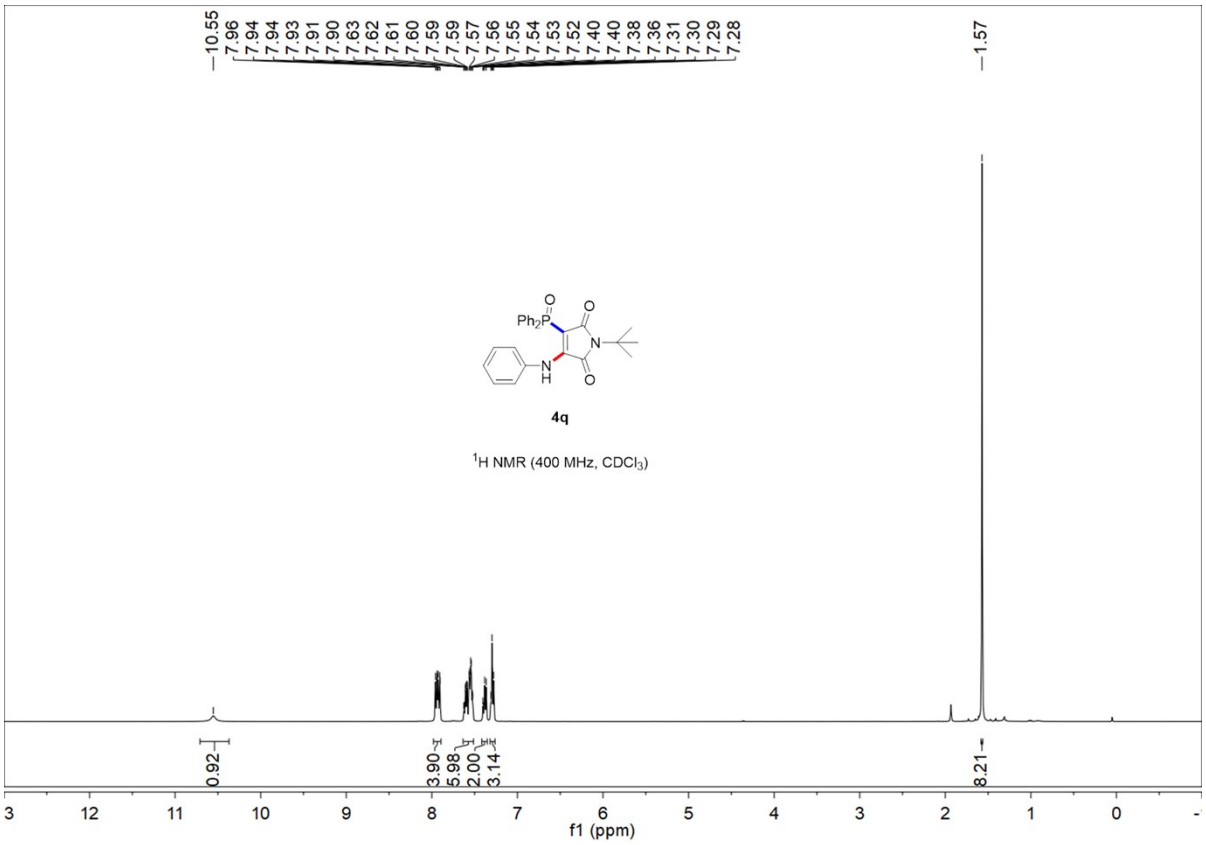

29.36
Ac
N
H
Ph2P
O
N–Ph
4o
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)


11.11
8.02
8.00
8.00
7.99
7.97
7.97
7.66
7.64
7.64
7.62
7.62
7.61
7.59
7.58
7.57
7.56
7.55
7.55
7.55
7.54
7.50
7.48
7.46
7.46
7.44
7.43
7.37
7.36
7.35
7.34
7.34
Ph2P
O
O
N–Ph
F3C
N
H
O
4p
1H NMR (400 MHz, CDCl3)
0.97
4.04
4.00
4.04
2.05
2.08
3.03
f1 (ppm)


168.32
168.20
163.03
162.90
155.46
139.87
132.70
132.67
132.56
131.60
131.48
131.12
129.03
128.85
128.72
128.67
127.99
126.12
126.08
126.04
125.27
124.78
122.57
91.22
90.07
Ph2P
O
O
N–Ph
F3C
N
H
O
4p
13C NMR (100 MHz, CDCl3)
f1 (ppm)

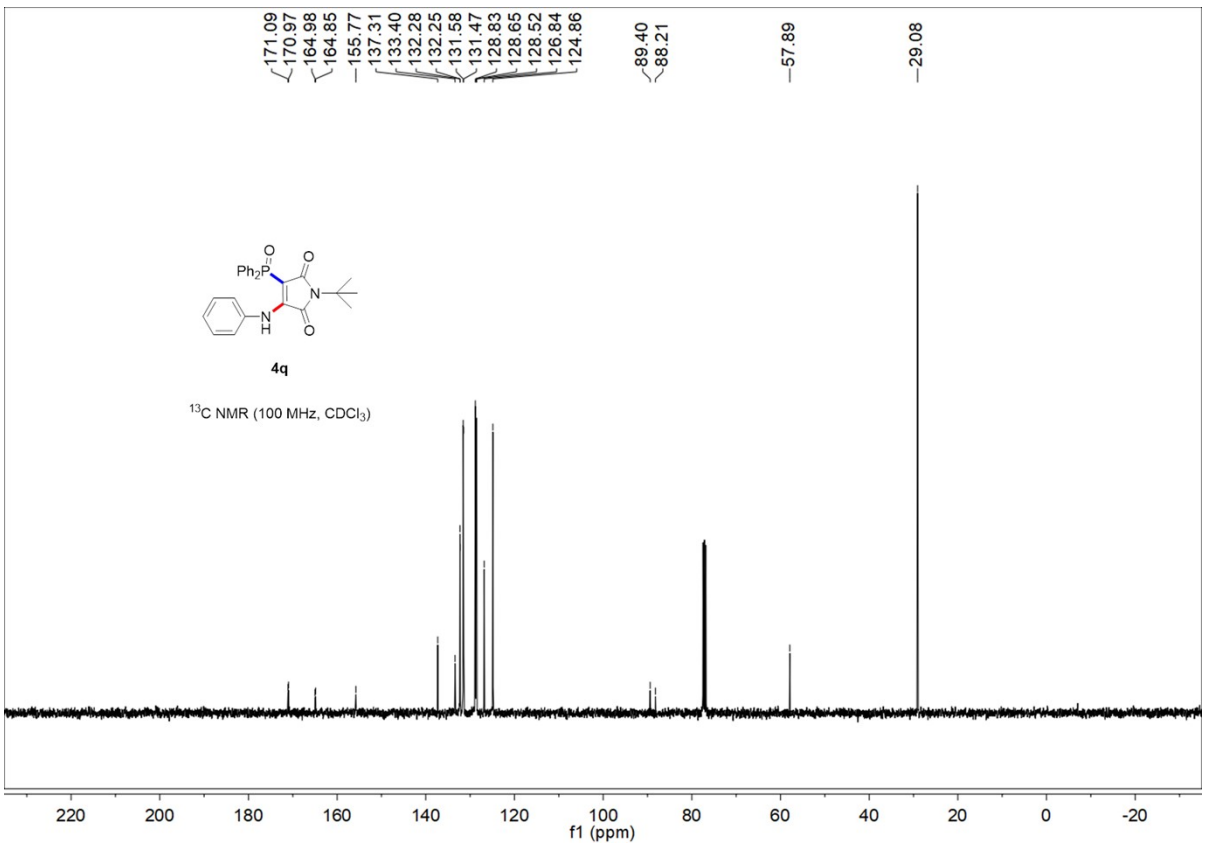

29.35
Ph2P
O
O
N–Ph
F3C
N
H
O
4p
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)


62.36
Ph2P
O
O
N–Ph
F3C
N
H
O
4p
19F NMR (377 MHz, CDCl3)
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)



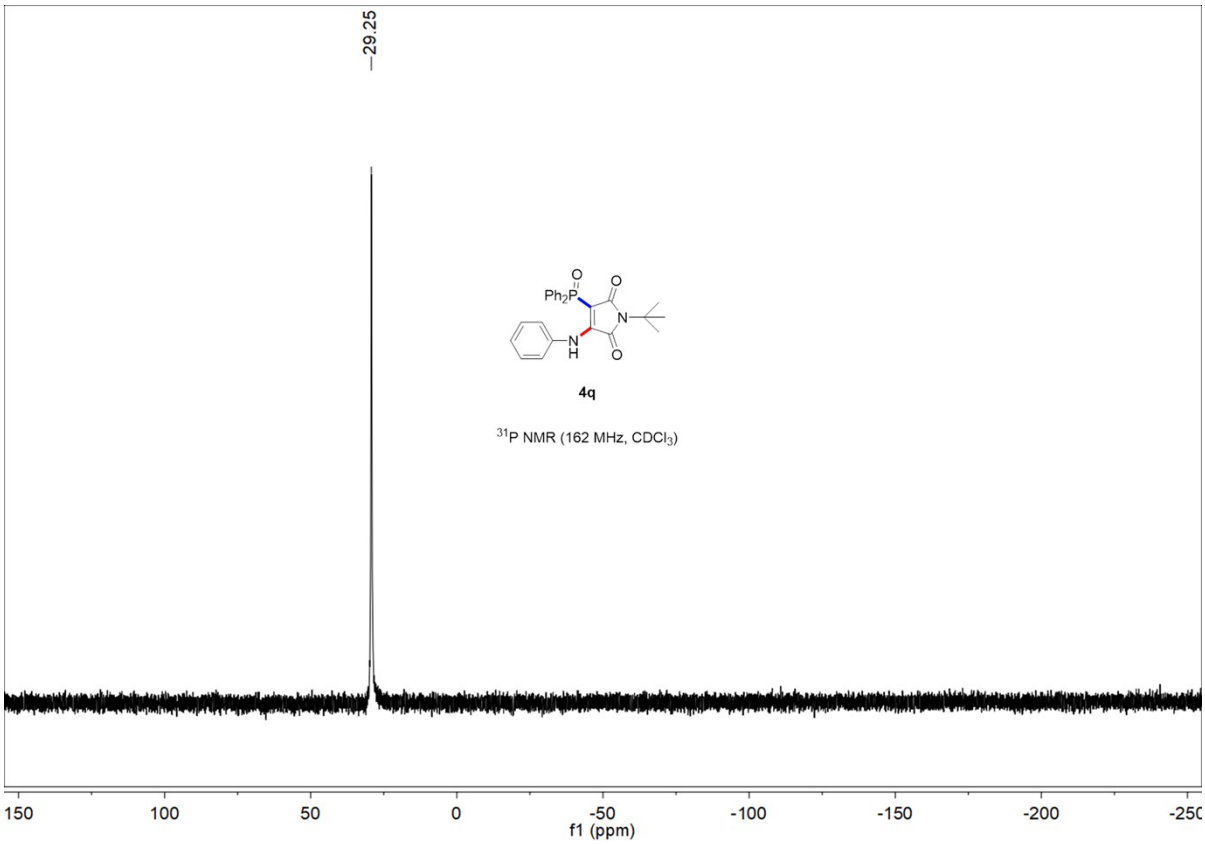
4q

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



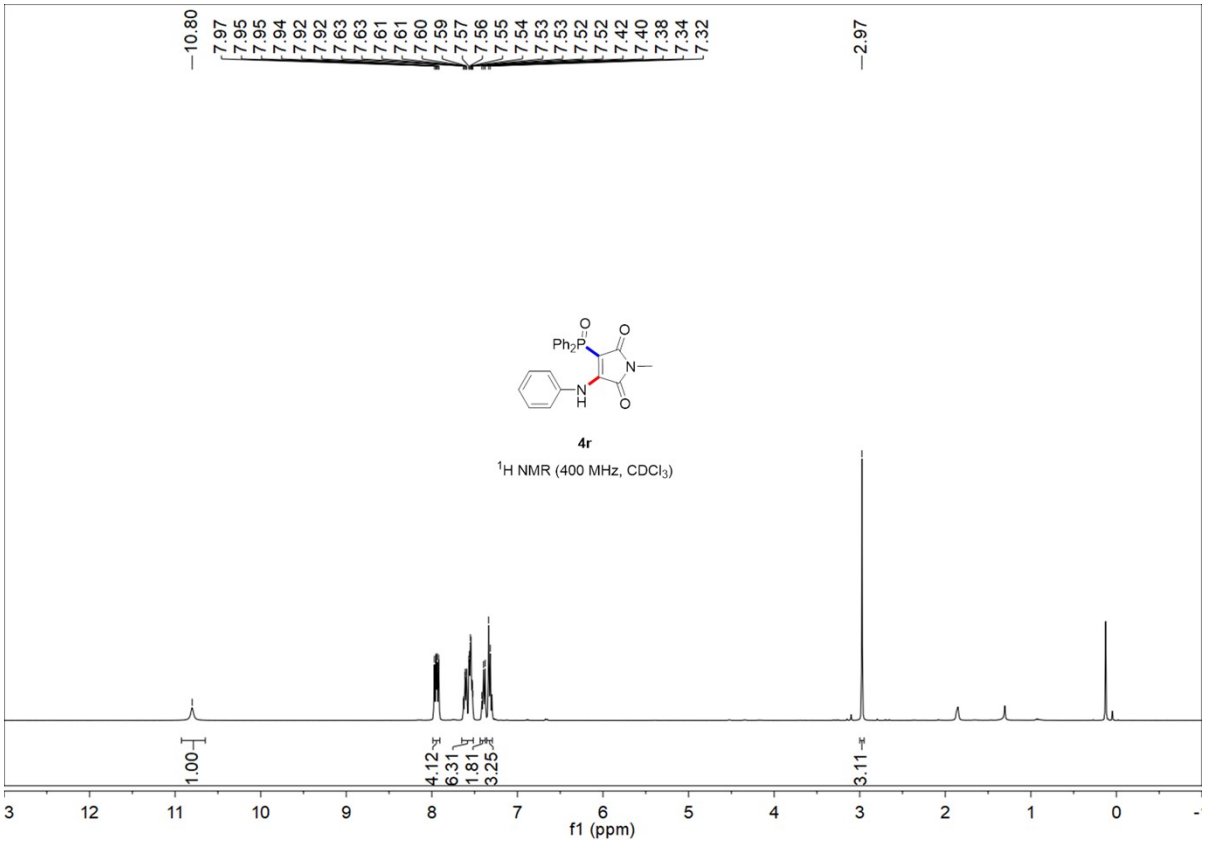
4q

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



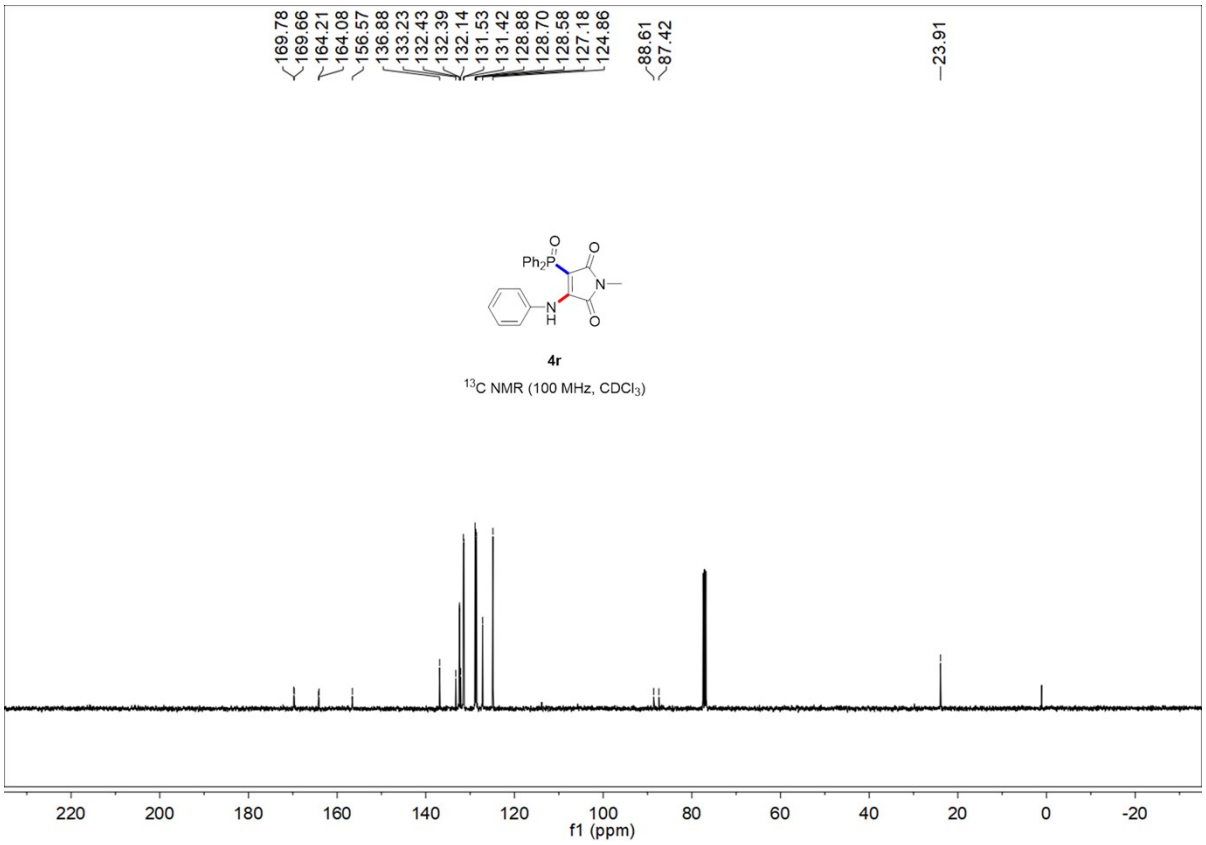
4q

^{31}P NMR (162 MHz, $CDCl_3$)



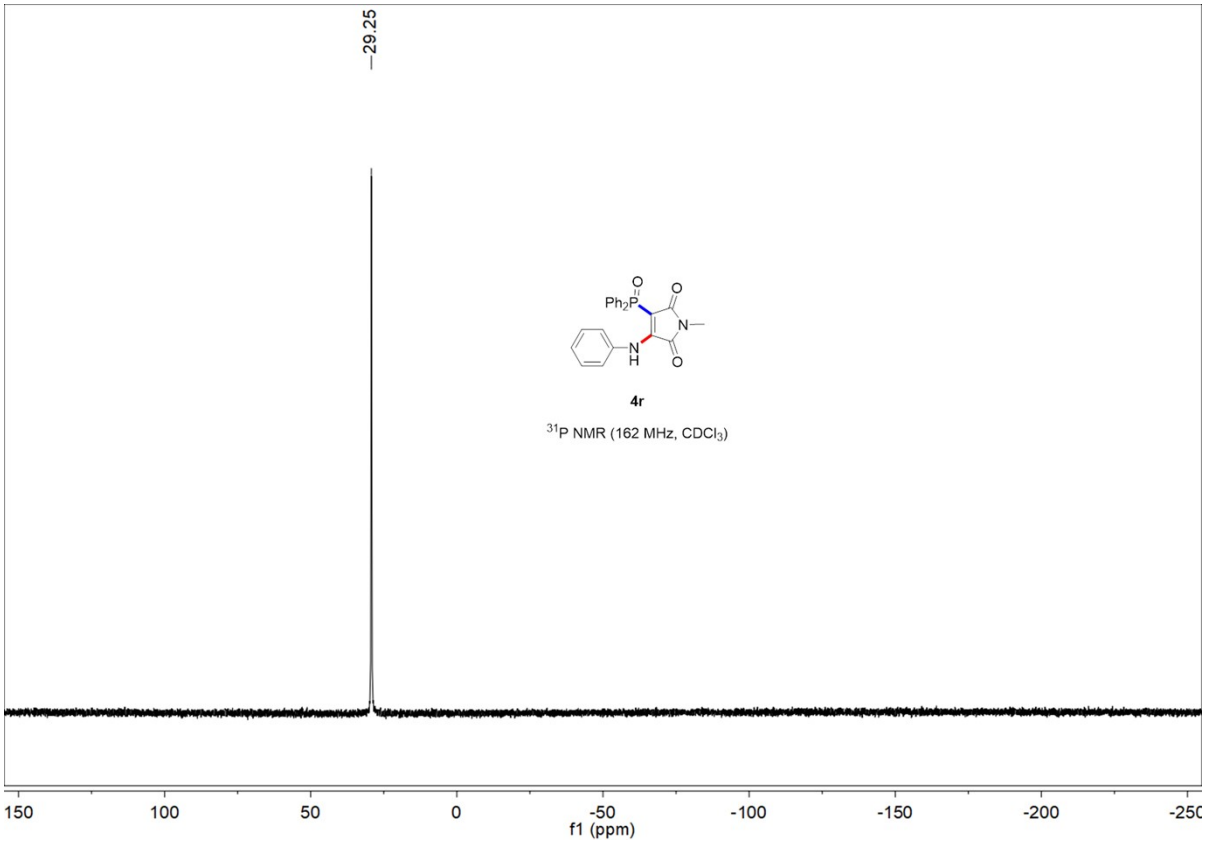
4r

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



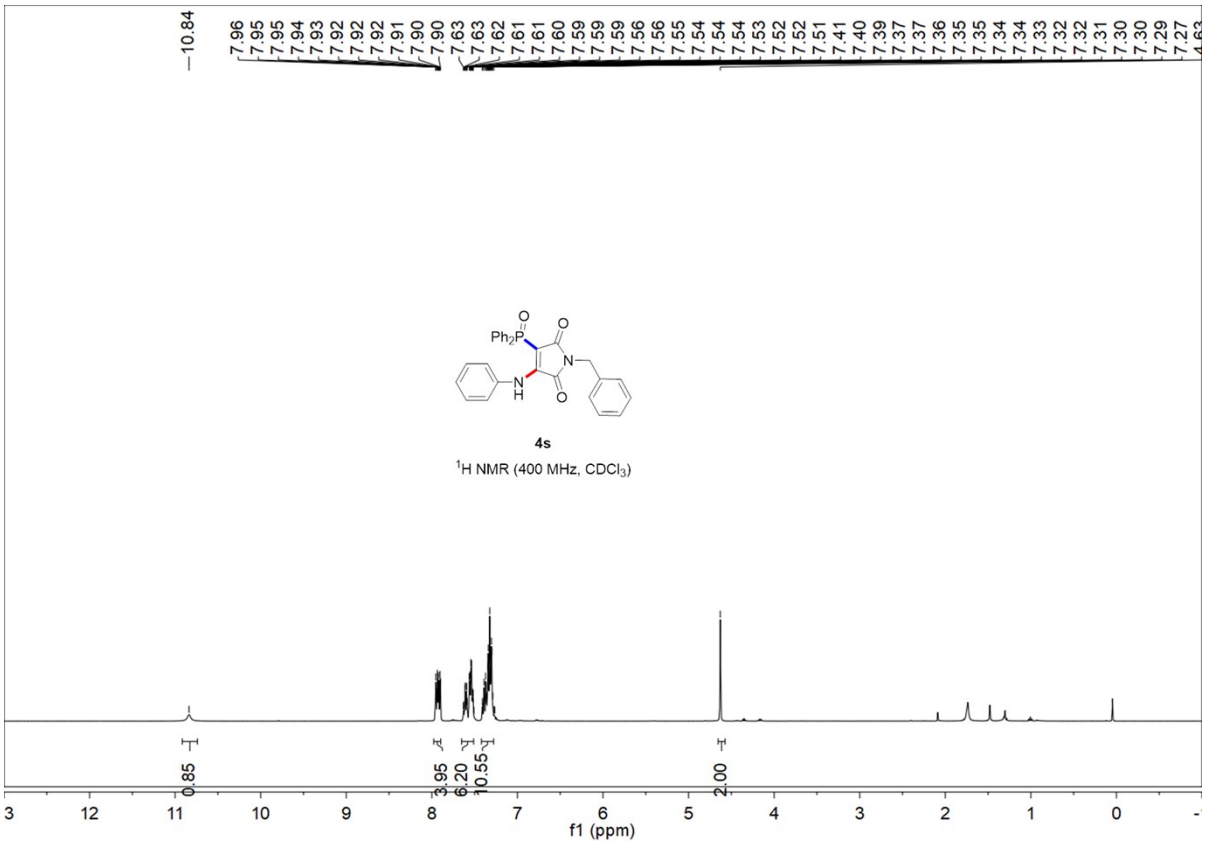
4r

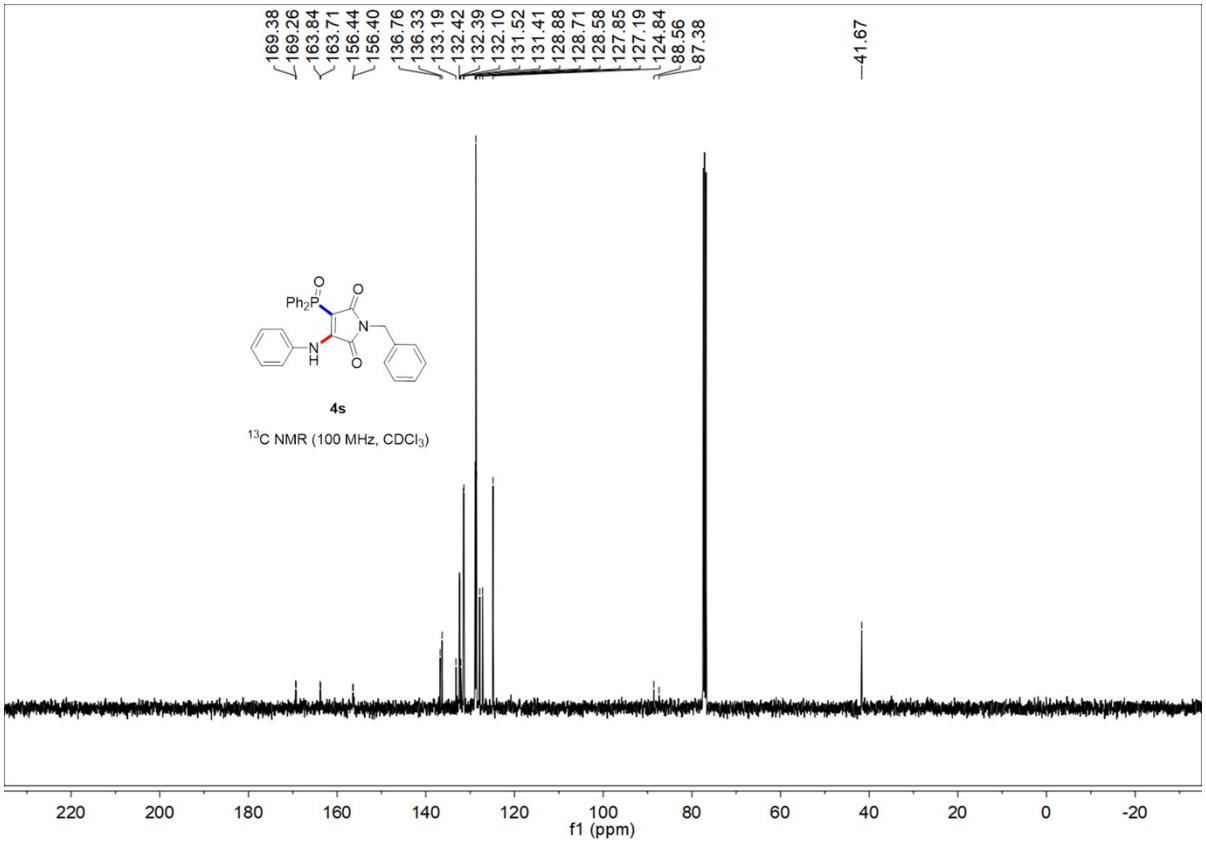
^{13}C NMR (100 MHz, $CDCl_3$)

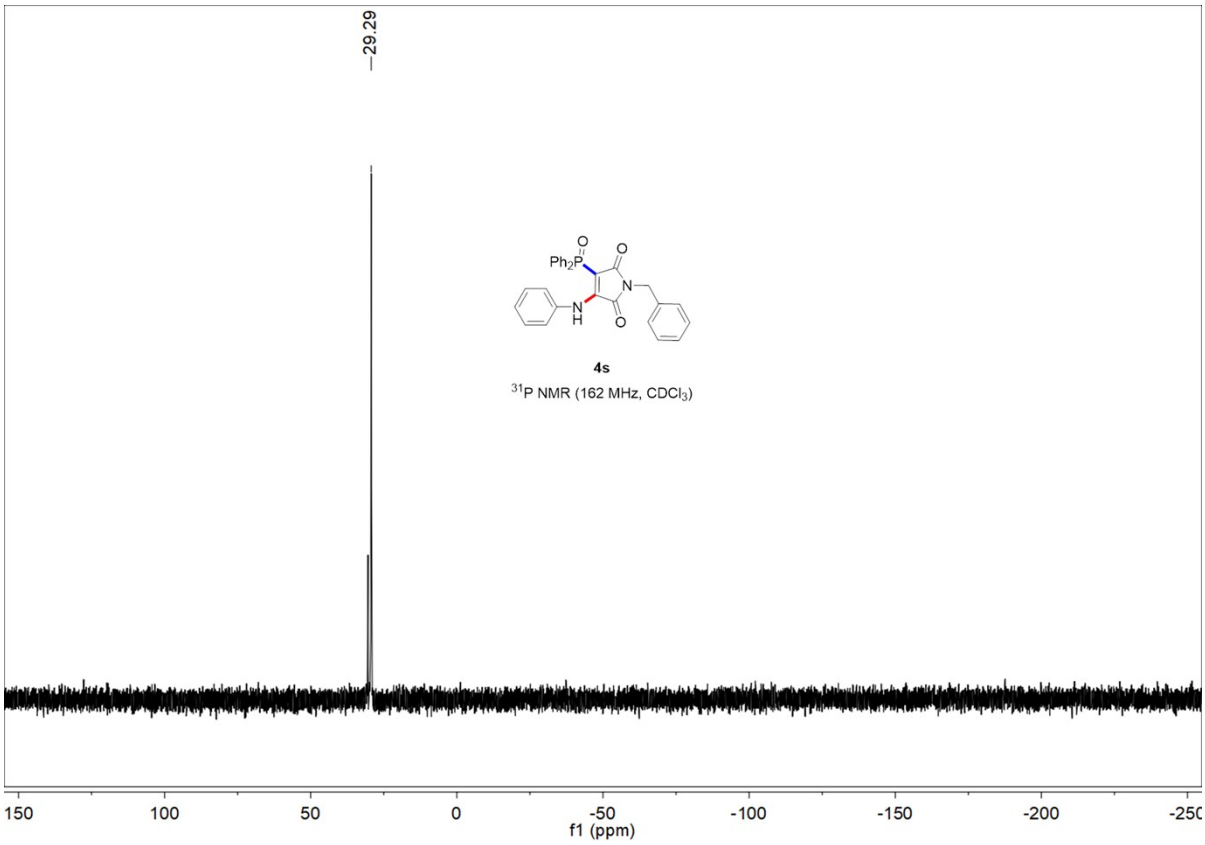


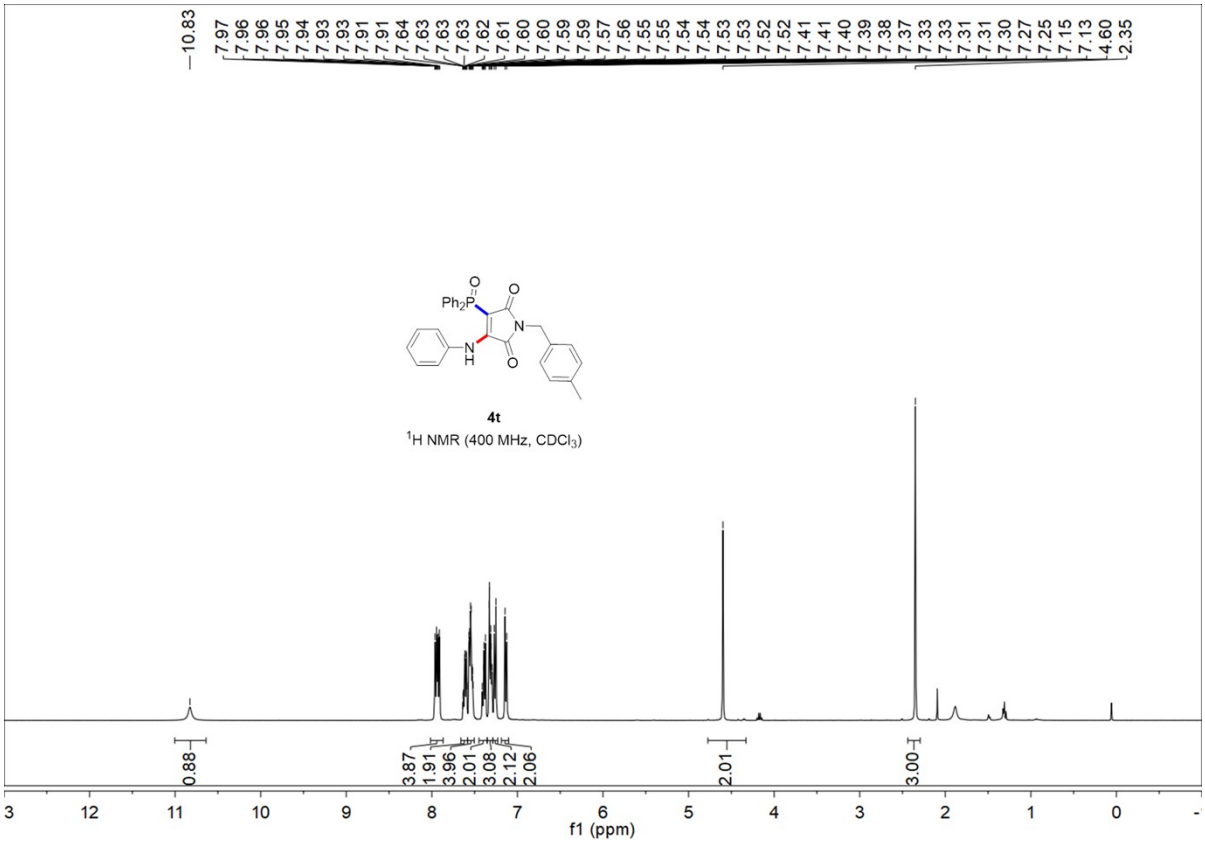
4r

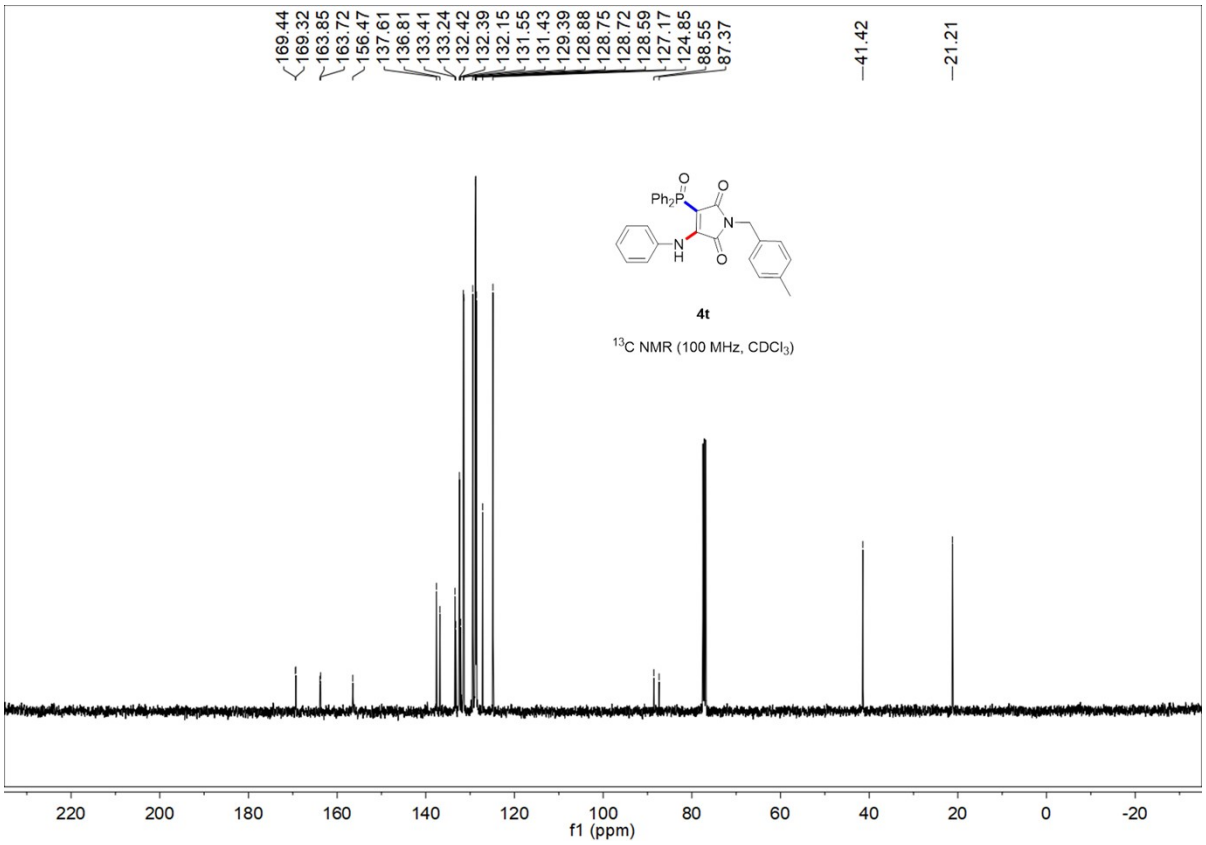
^{31}P NMR (162 MHz, $CDCl_3$)

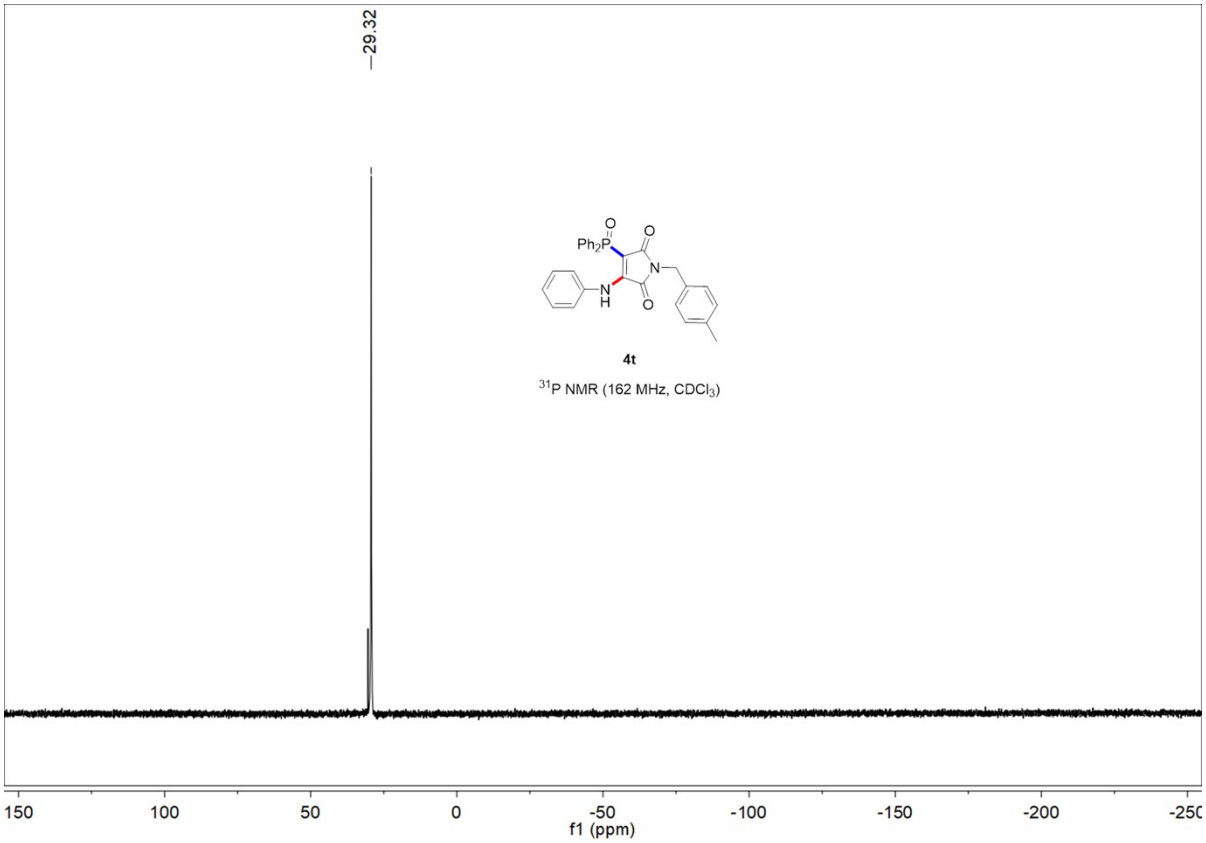

10.84
7.96
7.95
7.95
7.94
7.93
7.92
7.92
7.92
7.91
7.90
7.90
7.63
7.63
7.62
7.61
7.61
7.60
7.59
7.59
7.59
7.56
7.56
7.55
7.54
7.54
7.54
7.53
7.52
7.52
7.51
7.41
7.40
7.39
7.37
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.32
7.32
7.31
7.30
7.30
7.29
7.27
Ph2P
O
O
N
H
O
4s
1H NMR (400 MHz, CDCl3)
0.85
3.95
6.20
10.55
2.00
f1 (ppm)

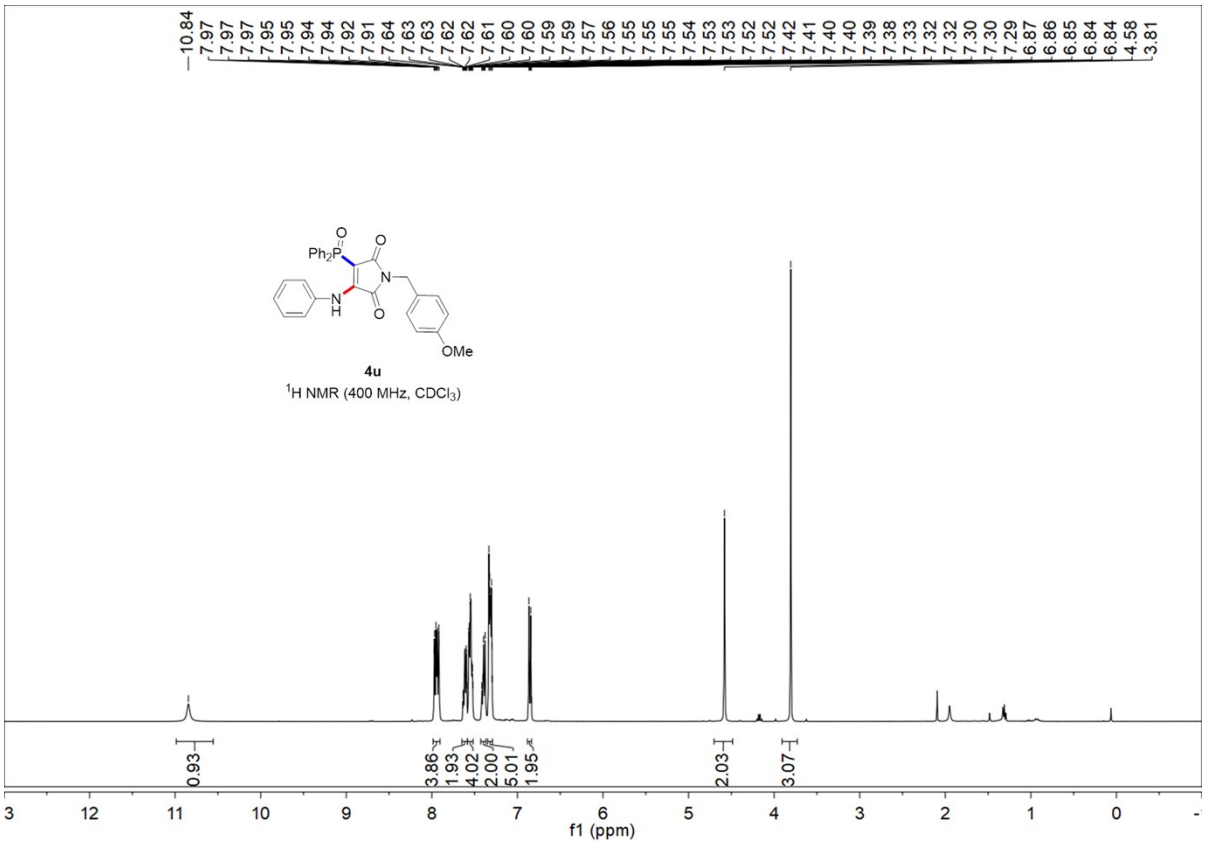

169.38
169.26
163.84
163.71
156.44
156.40
136.76
136.33
133.19
132.42
132.39
132.10
131.52
131.41
128.88
128.71
128.58
127.85
127.19
124.84
88.56
87.38
41.67
Ph2P
O
O
N
H
O
4s
13C NMR (100 MHz, CDCl3)
f1 (ppm)

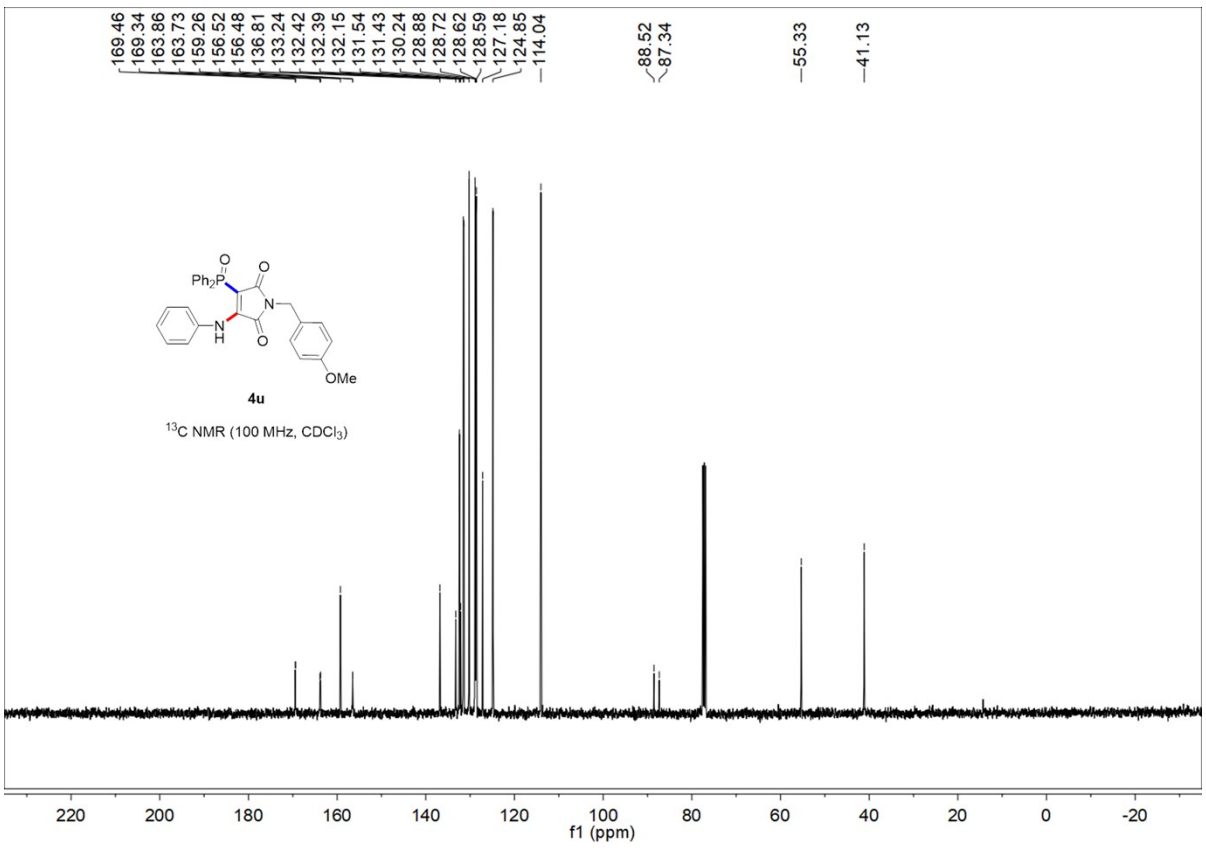

29.29
Ph2P
O
O
N
H
O
4s
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

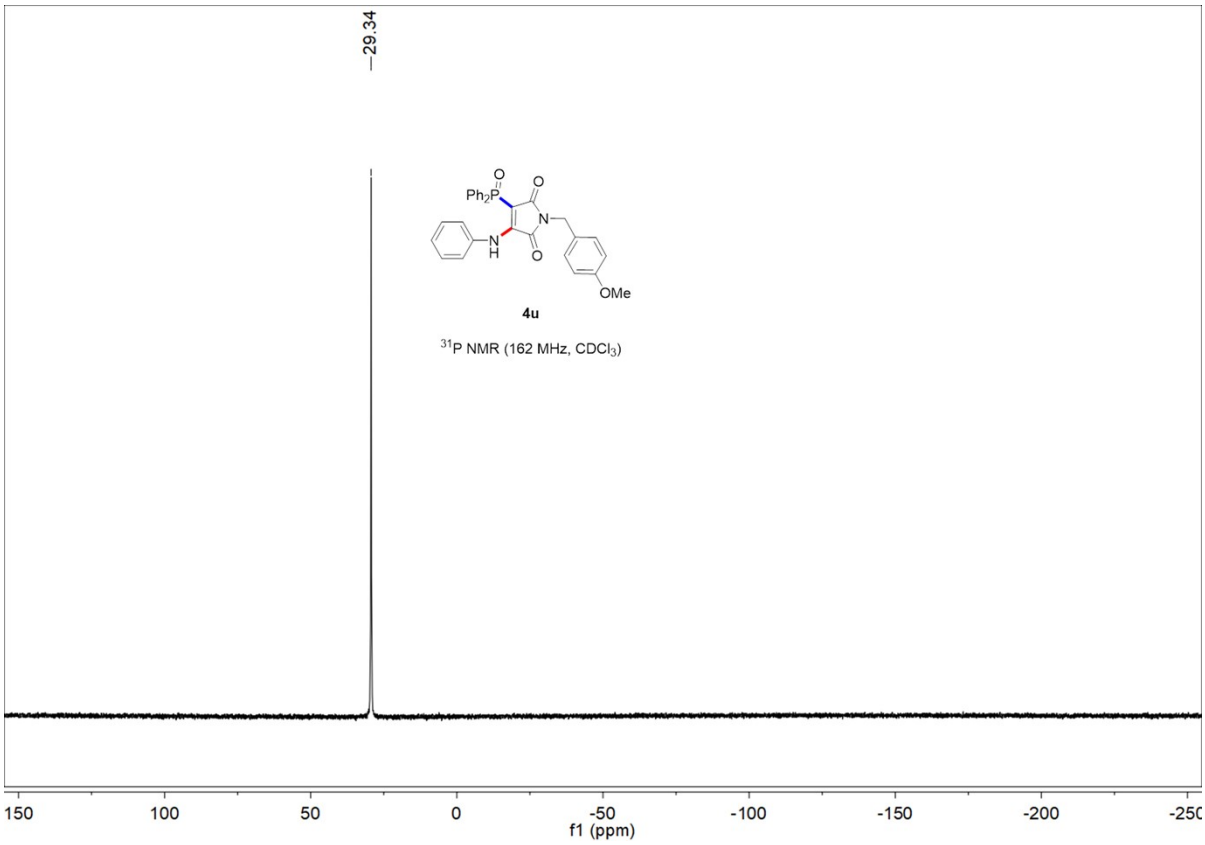

10.83
7.97
7.96
7.96
7.95
7.94
7.93
7.93
7.91
7.91
7.64
7.63
7.63
7.63
7.62
7.61
7.60
7.60
7.59
7.59
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.53
7.52
7.52
7.41
7.41
7.40
7.39
7.38
7.37
7.33
7.33
7.31
7.31
7.30
7.27
7.25
7.15
7.13
4.60
2.35
Ph2P
O
O
N
H
O
4t
1H NMR (400 MHz, CDCl3)
0.88
3.87
1.91
3.96
2.01
3.08
2.12
2.06
2.01
3.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

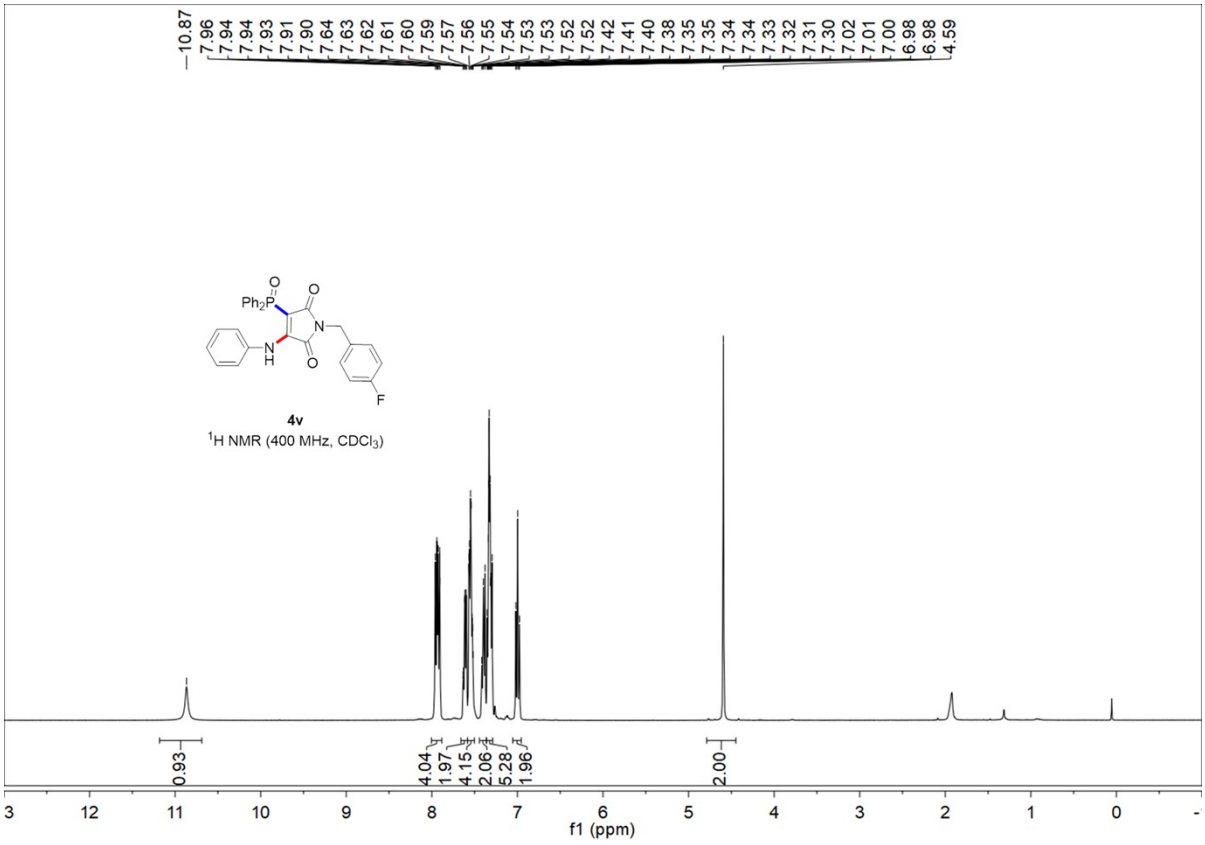

169.44
169.32
163.85
163.72
156.47
137.61
136.81
133.41
133.24
132.42
132.39
132.15
131.55
131.43
129.39
128.88
128.75
128.72
128.59
127.17
124.85
88.55
87.37
41.42
21.21
4t
13C NMR (100 MHz, CDCl3)
f1 (ppm)

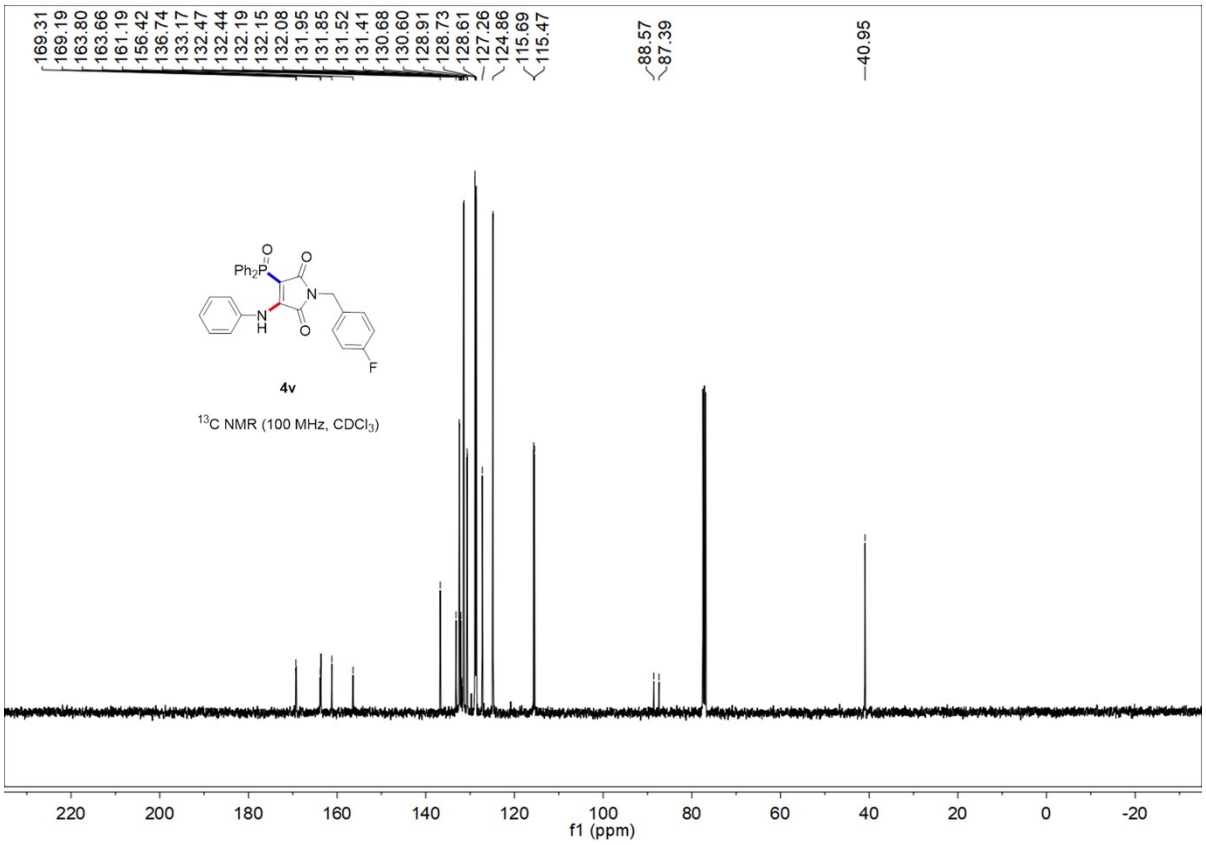

29.32
4t
31P NMR (162 MHz, CDCl3)
f1 (ppm)

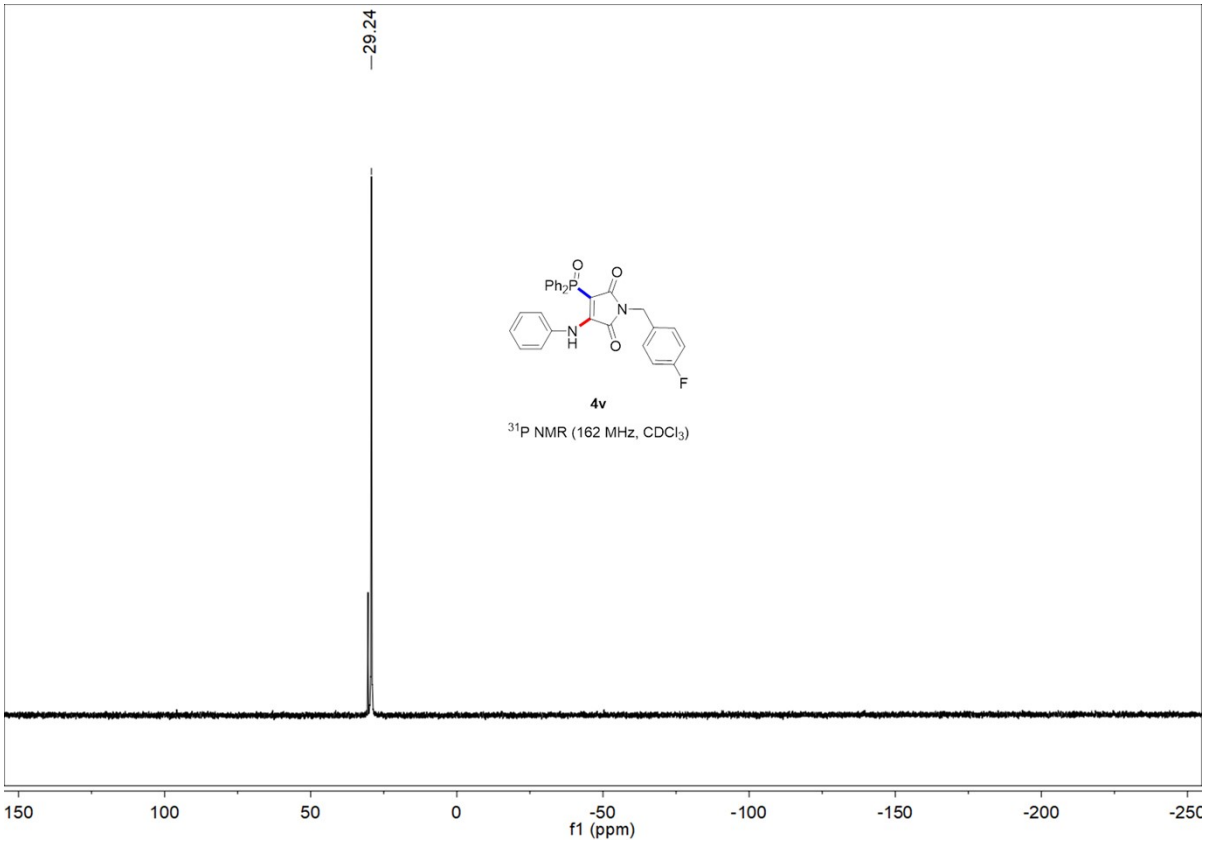

10.84
7.97
7.97
7.97
7.95
7.95
7.94
7.94
7.92
7.91
7.64
7.63
7.63
7.62
7.62
7.61
7.60
7.60
7.59
7.59
7.57
7.56
7.55
7.55
7.55
7.54
7.53
7.53
7.52
7.52
7.42
7.41
7.40
7.40
7.39
7.38
7.33
7.32
7.32
7.30
7.30
7.29
6.87
6.86
6.85
6.84
6.84
4.58
3.81
Ph2P
O
O
N
H
O
OMe
4u
1H NMR (400 MHz, CDCl3)
0.93
3.86
1.93
4.02
2.00
5.01
1.95
2.03
3.07
f1 (ppm)

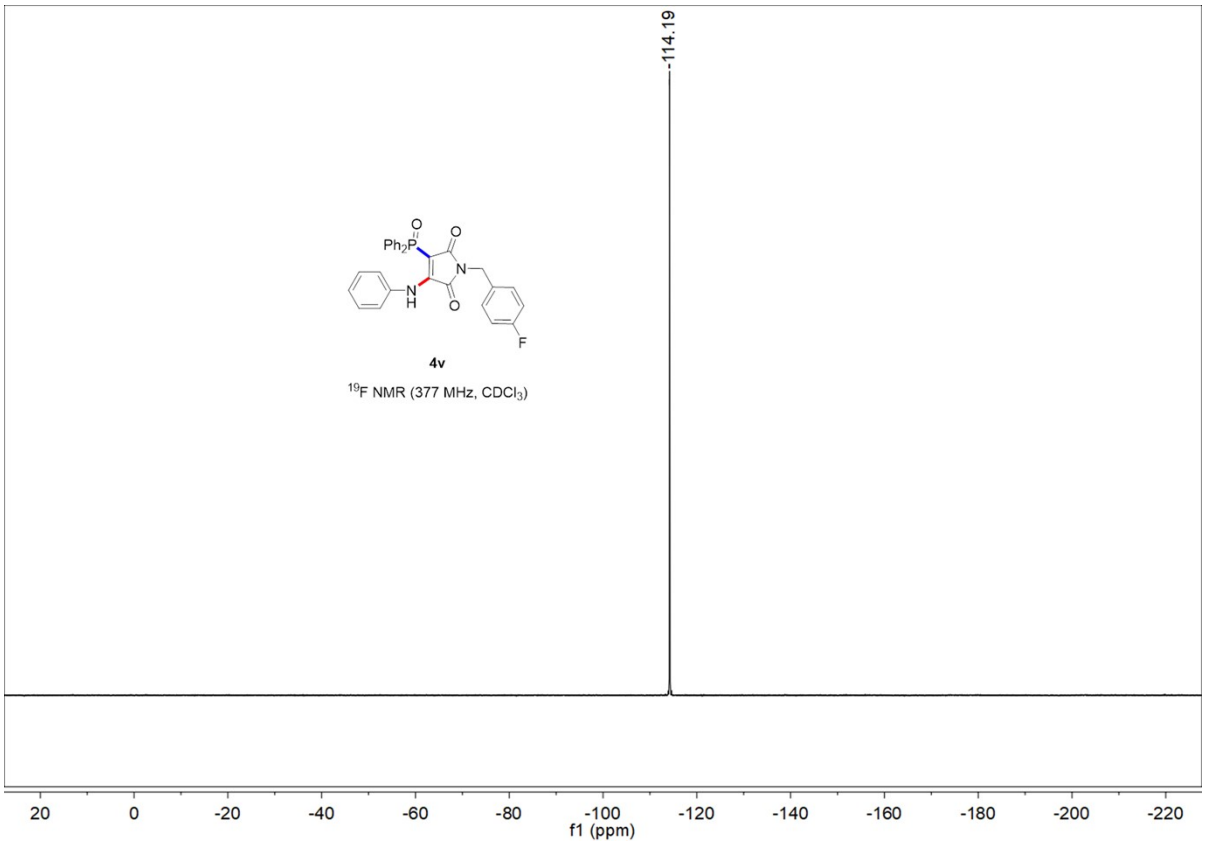

169.46
169.34
163.86
163.73
159.26
156.52
156.48
136.81
133.24
132.42
132.39
132.15
131.54
131.43
130.24
128.88
128.72
128.62
128.59
127.18
124.85
114.04
88.52
87.34
55.33
41.13
Ph2P
O
O
N
H
O
OMe
4u
13C NMR (100 MHz, CDCl3)
f1 (ppm)

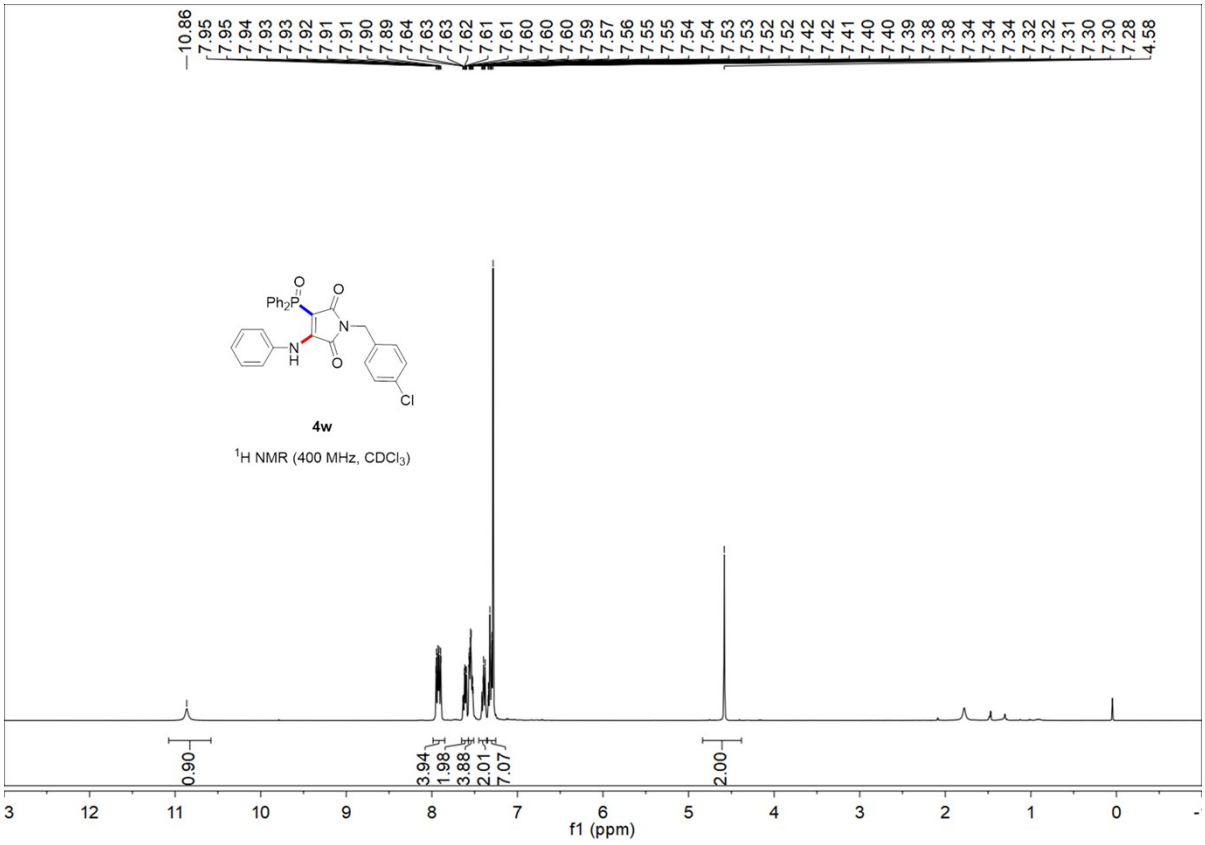

29.34
Ph2P
O
O
N
H
O
OMe
4u
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

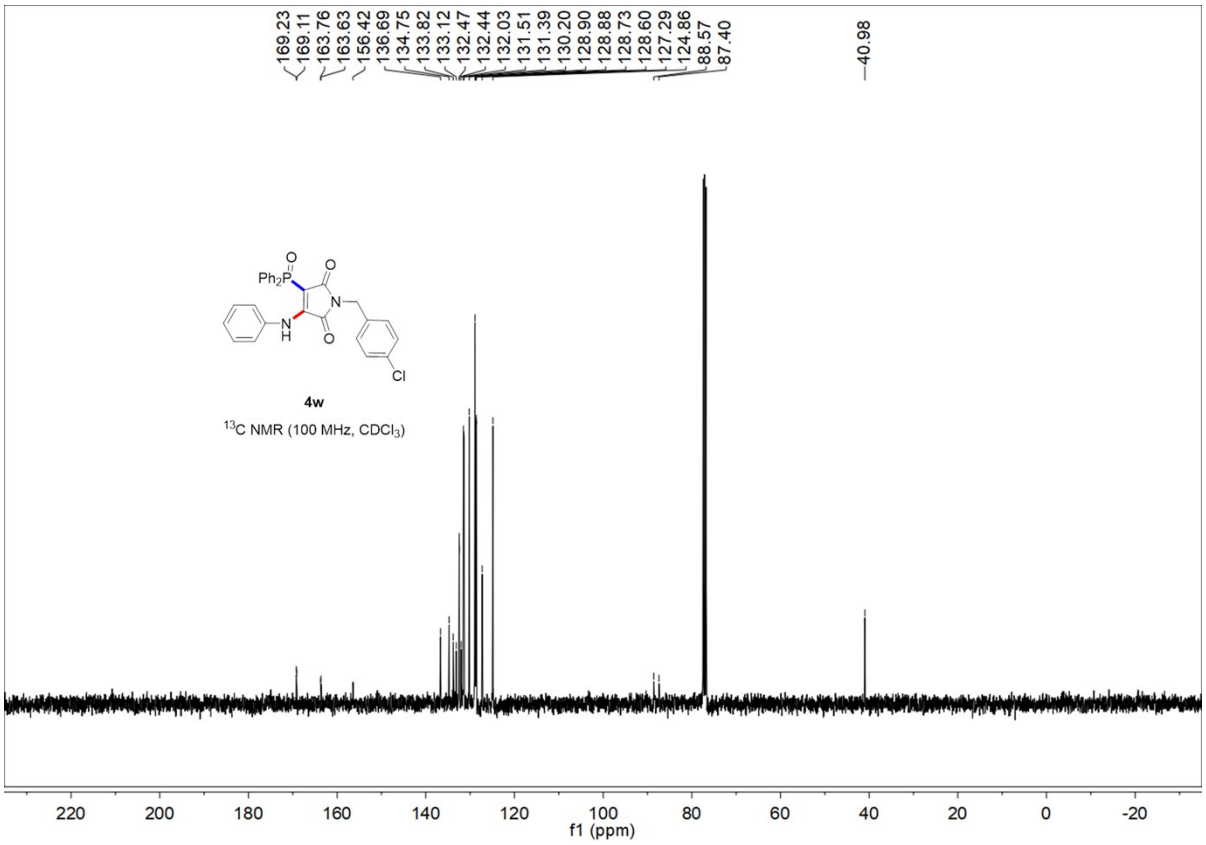

10.87
7.96
7.94
7.94
7.93
7.91
7.90
7.64
7.63
7.62
7.61
7.60
7.59
7.57
7.56
7.55
7.54
7.53
7.53
7.52
7.52
7.42
7.41
7.40
7.38
7.35
7.35
7.34
7.34
7.33
7.32
7.31
7.30
7.02
7.01
7.00
6.98
6.98
4.59
Ph2P
O
O
N
H
O
F
4v
1H NMR (400 MHz, CDCl3)
0.93
4.04
1.97
4.15
2.06
5.28
1.96
2.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

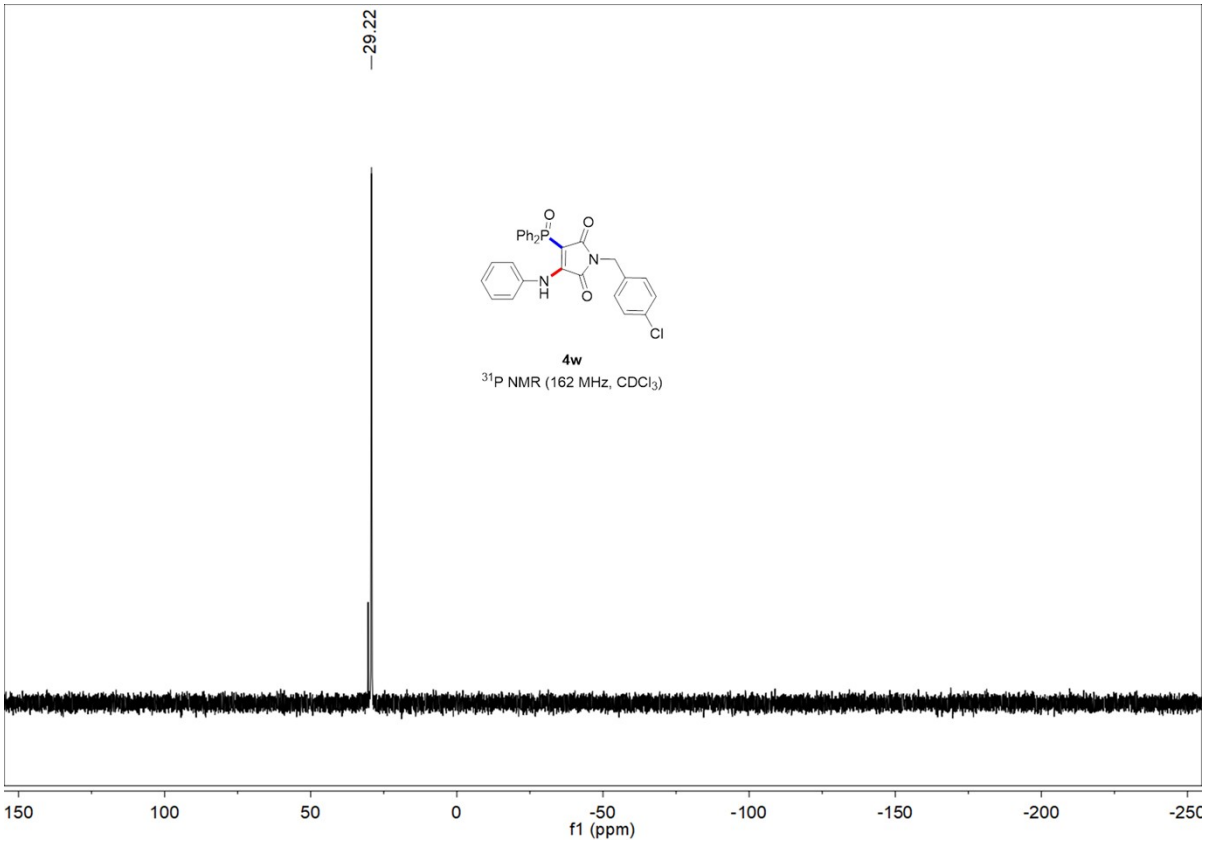

169.31
169.19
163.80
163.66
161.19
156.42
136.74
133.17
132.47
132.44
132.19
132.15
132.08
131.95
131.85
131.52
131.41
130.68
130.60
128.91
128.73
128.61
127.26
124.86
115.69
115.47
88.57
87.39
40.95
Ph2P
O
O
N
H
O
F
4v
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

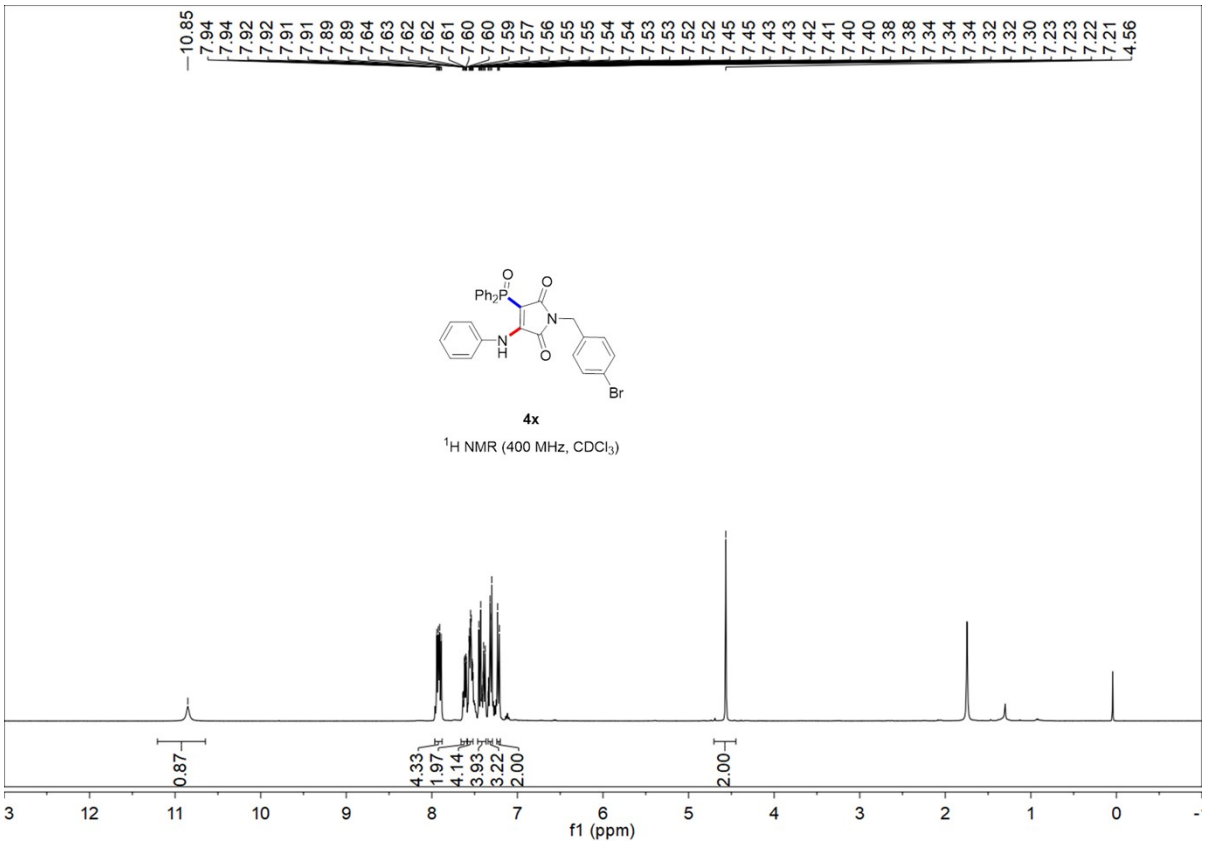

29.24
Ph2P
O
O
N
H
O
F
4v
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

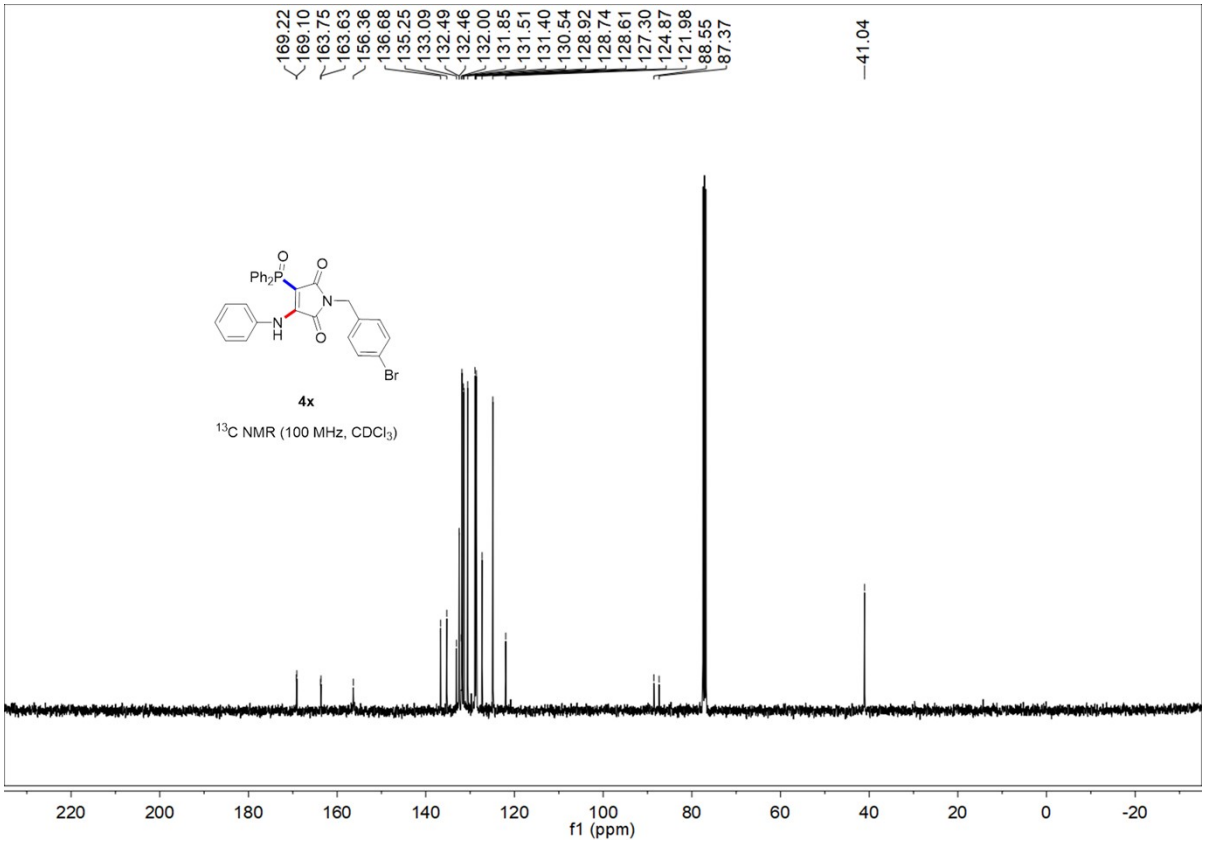

-114.19
Ph2P
O
O
N
H
O
F
4v
19F NMR (377 MHz, CDCl3)
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

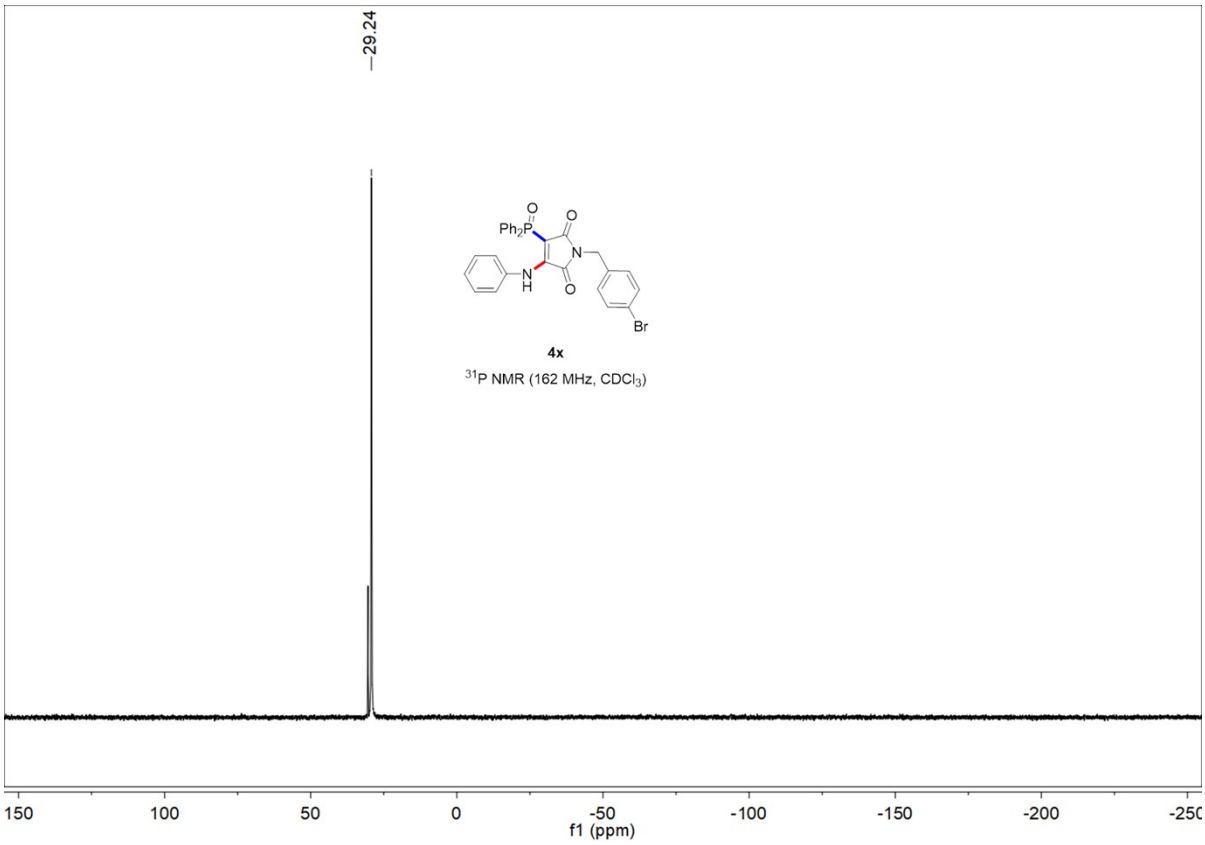

10.86
7.95
7.95
7.94
7.93
7.93
7.92
7.91
7.91
7.90
7.89
7.64
7.63
7.63
7.62
7.61
7.61
7.60
7.60
7.60
7.59
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.53
7.52
7.52
7.42
7.42
7.41
7.40
7.40
7.39
7.38
7.38
7.34
7.34
7.34
7.32
7.32
7.31
7.30
7.30
7.28
4.58
Ph2P
O
O
N
H
O
Cl
4w
1H NMR (400 MHz, CDCl3)
0.90
3.94
1.98
3.88
2.01
7.07
2.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


169.23
169.11
163.76
163.63
156.42
136.69
134.75
133.82
133.12
132.47
132.44
132.03
131.51
131.39
130.20
128.90
128.88
128.73
128.60
127.29
124.86
88.57
87.40
40.98
4w
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)


29.22
4w
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

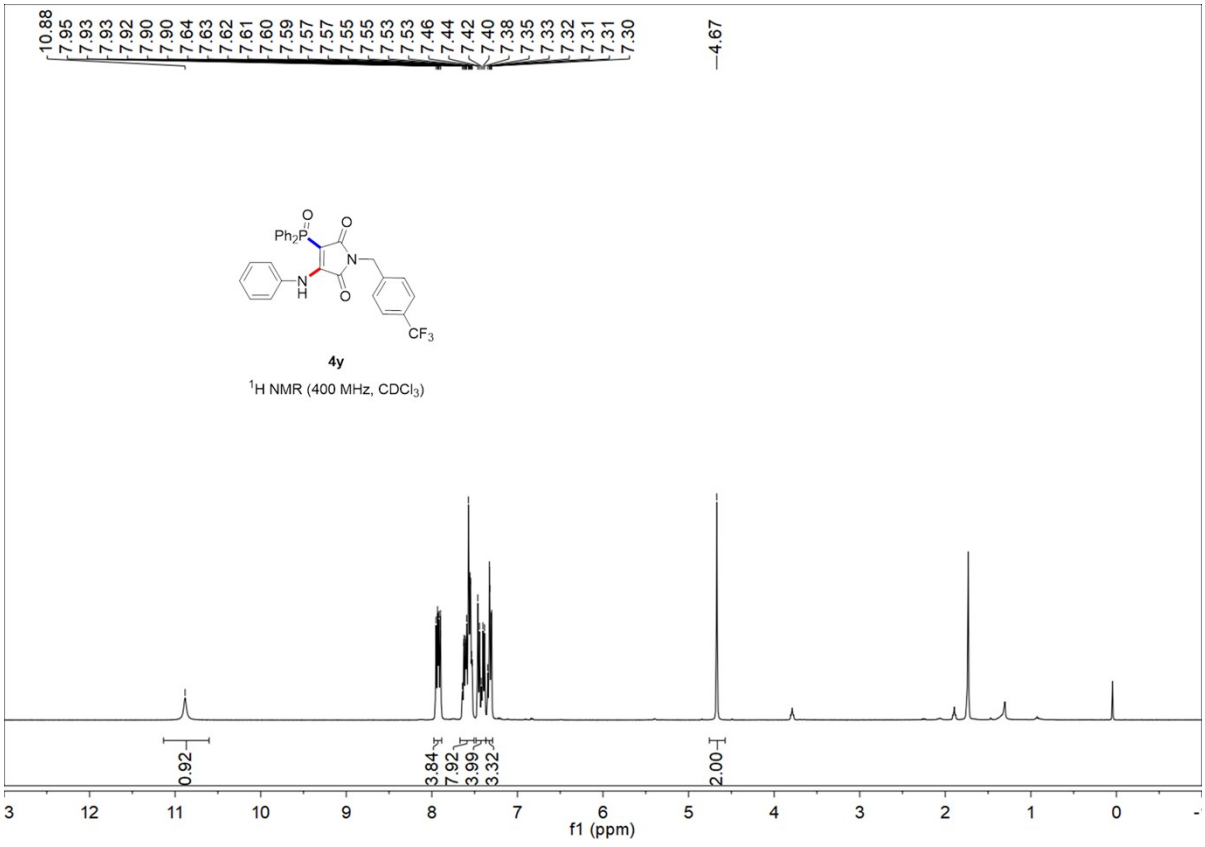

10.85
7.94
7.94
7.92
7.92
7.91
7.91
7.89
7.89
7.64
7.63
7.62
7.62
7.61
7.60
7.60
7.59
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.53
7.52
7.52
7.45
7.45
7.43
7.43
7.42
7.41
7.40
7.40
7.38
7.34
7.34
7.32
7.32
7.30
7.23
7.23
7.22
7.21
4.56
Ph2P
O
O
N
H
O
Br
4x
1H NMR (400 MHz, CDCl3)
0.87
4.33
1.97
4.14
3.93
3.22
2.00
2.00
f1 (ppm)


169.22
169.10
163.75
163.63
156.36
136.68
135.25
133.09
132.49
132.46
132.00
131.85
131.51
131.40
130.54
128.92
128.74
128.61
127.30
124.87
121.98
88.55
87.37
41.04
Ph2P
O
O
N
H
O
Br
4x
13C NMR (100 MHz, CDCl3)
f1 (ppm)



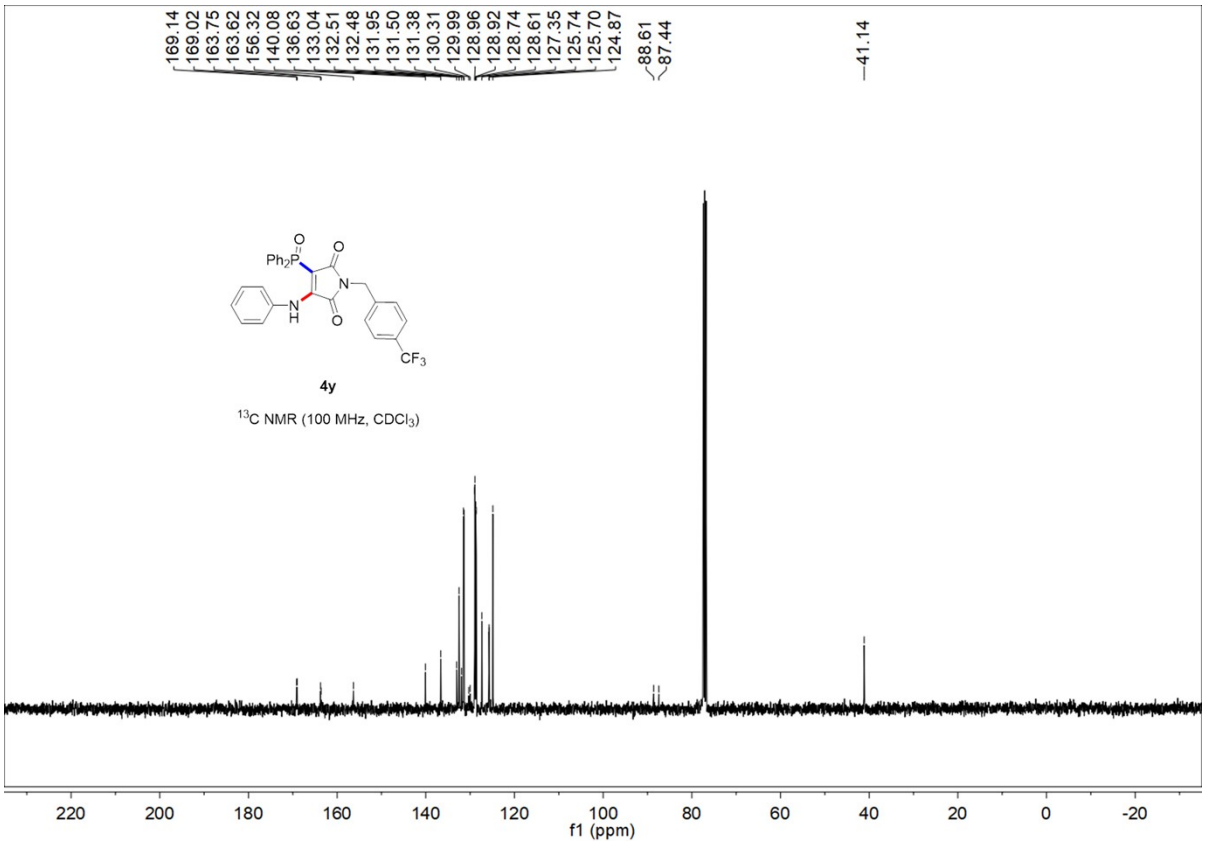
4x

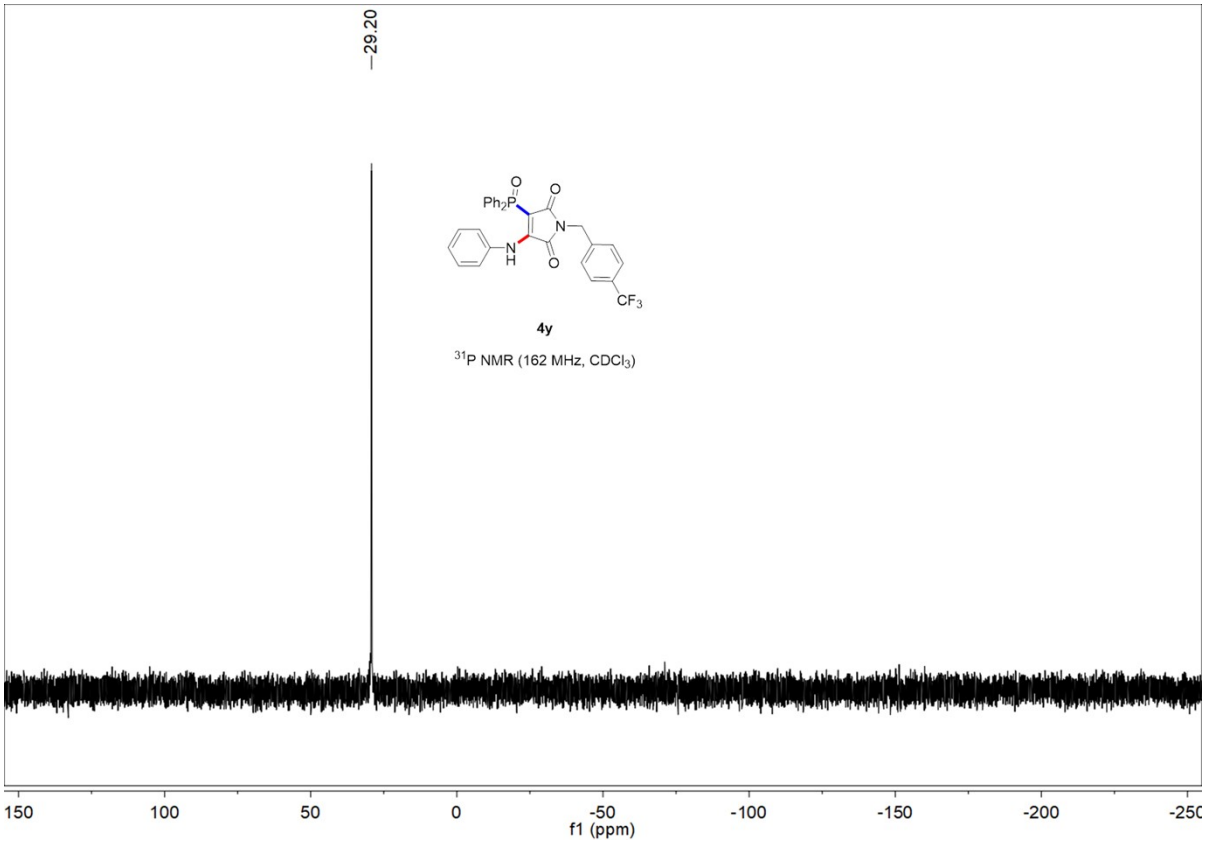
^{31}P NMR (162 MHz, $CDCl_3$)

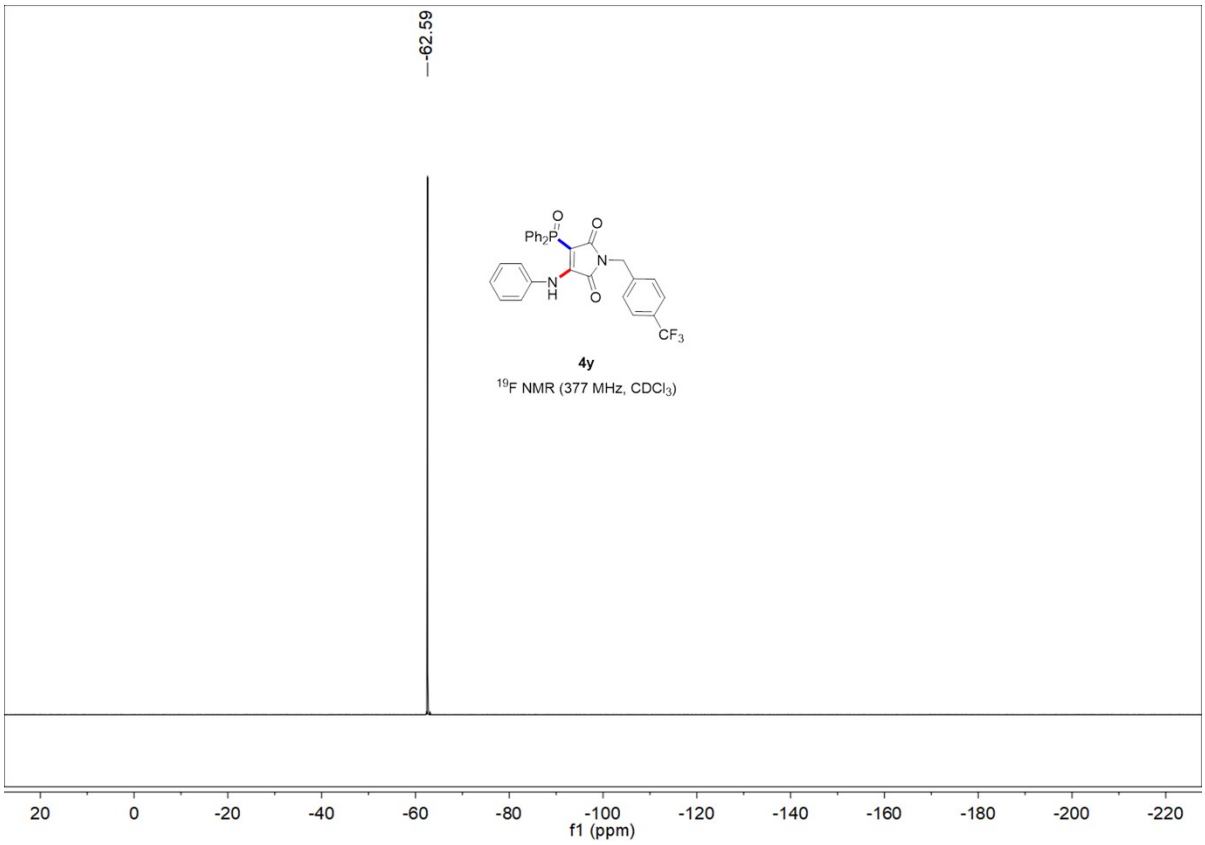


4y

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

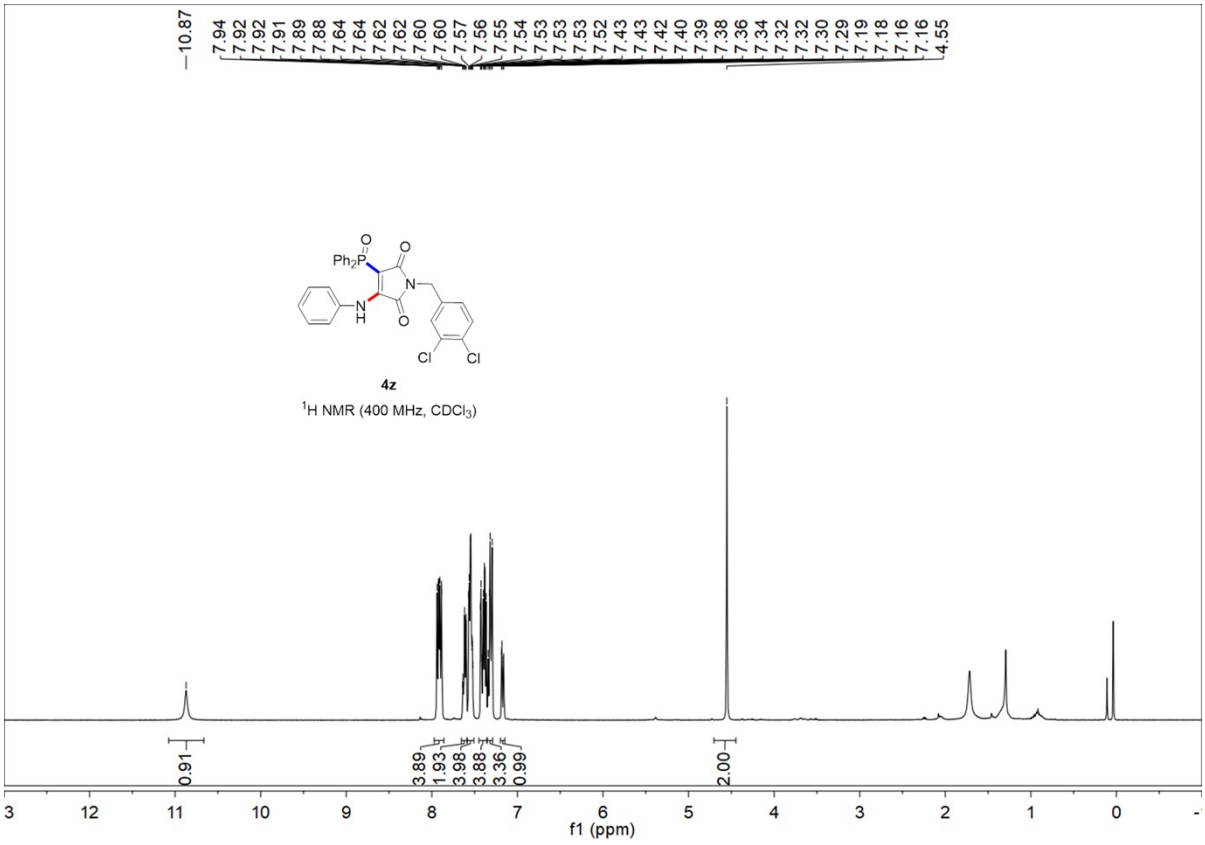

169.14
169.02
163.75
163.62
156.32
140.08
136.63
133.04
132.51
132.48
131.95
131.50
131.38
130.31
129.99
128.96
128.92
128.74
128.61
127.35
125.74
125.70
124.87
88.61
87.44
41.14
4y
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)


29.20
4y
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)



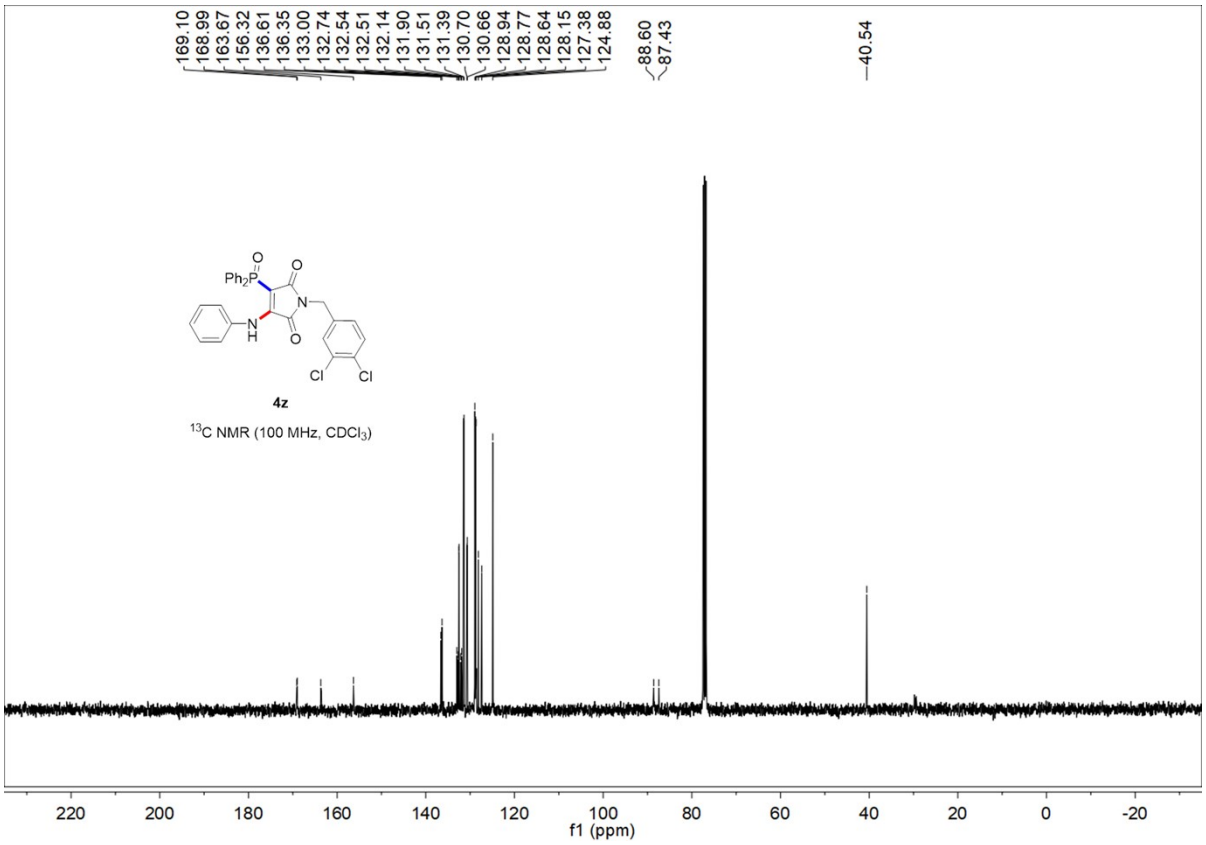
4y

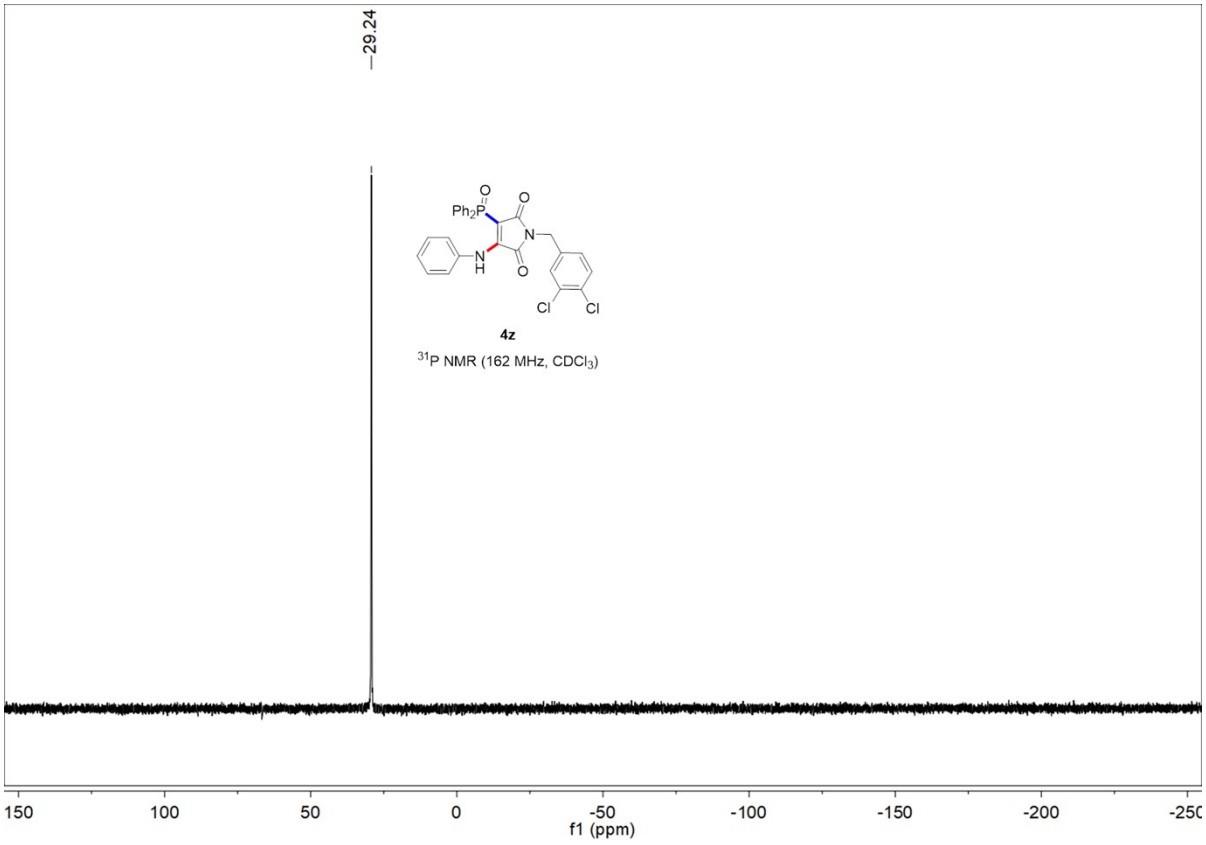
^{19}F NMR (377 MHz, $CDCl_3$)

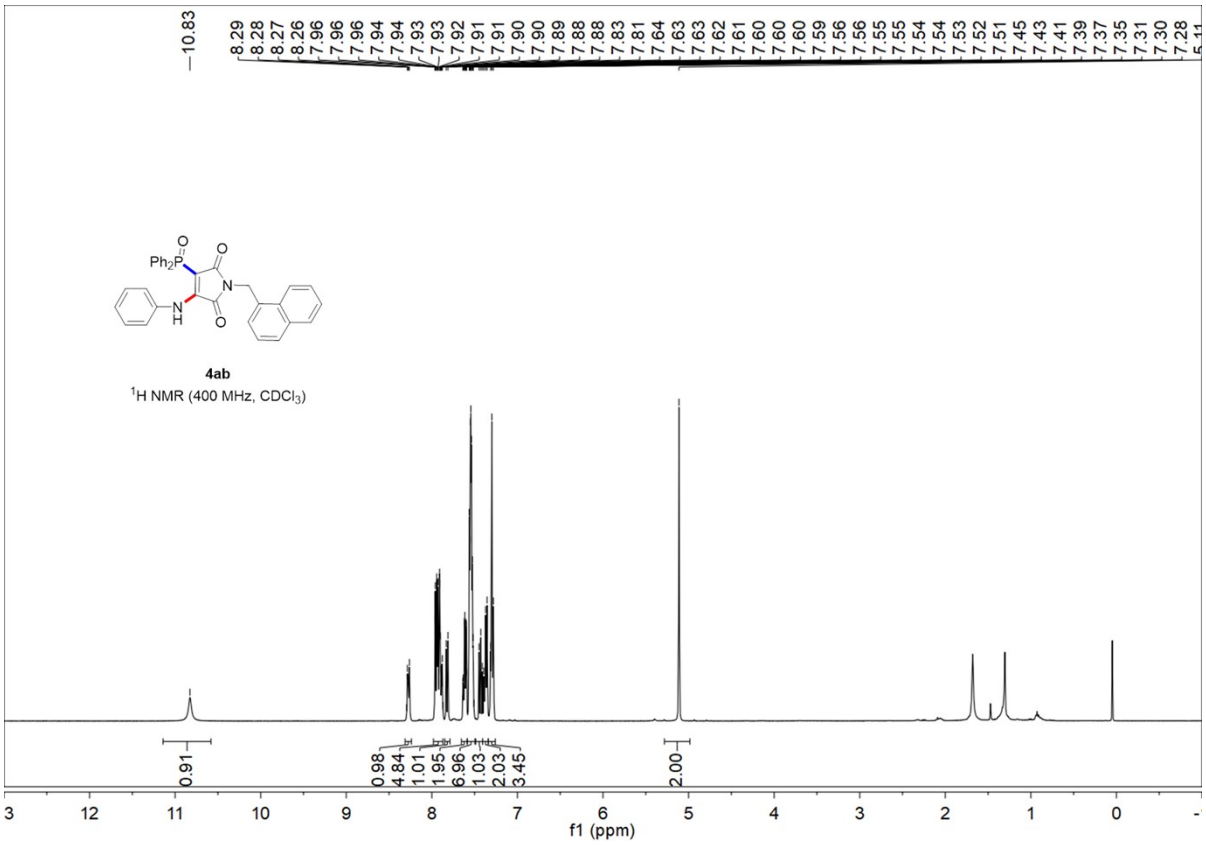


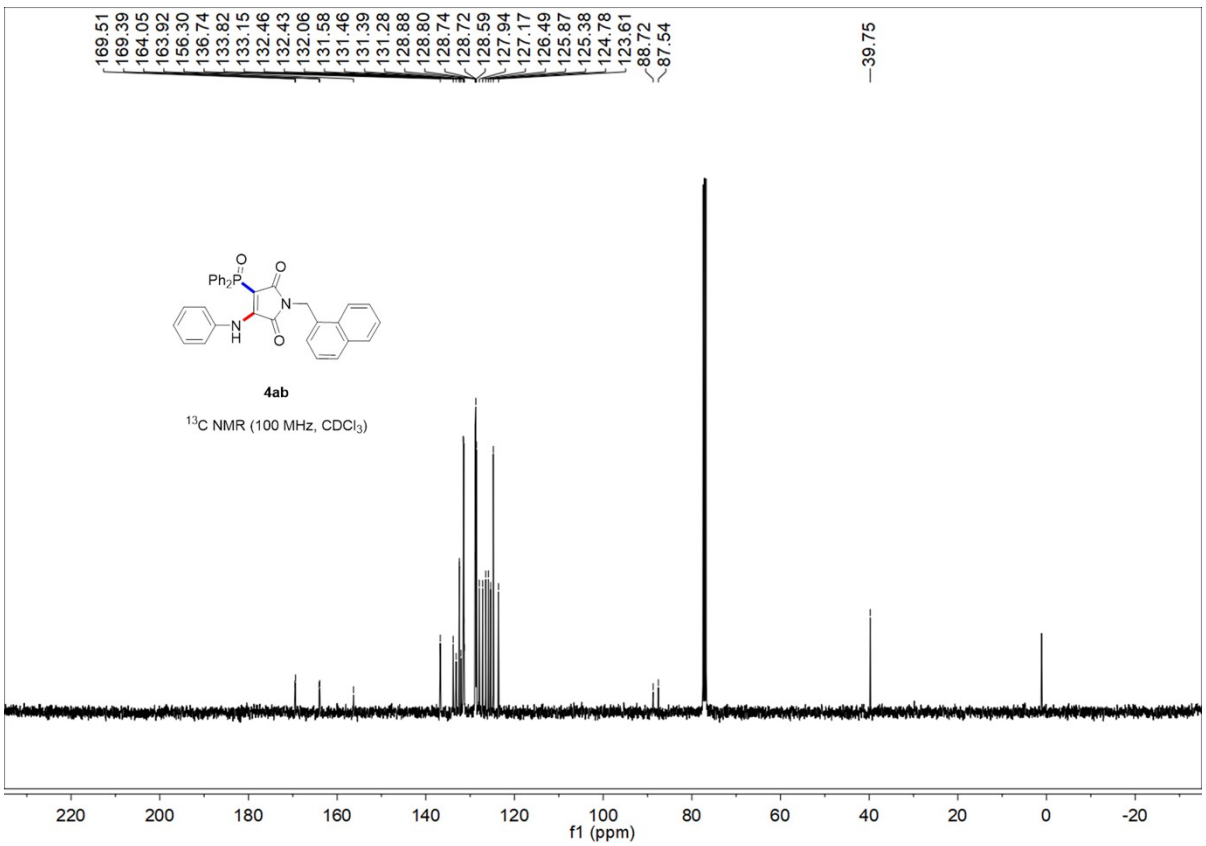
4z

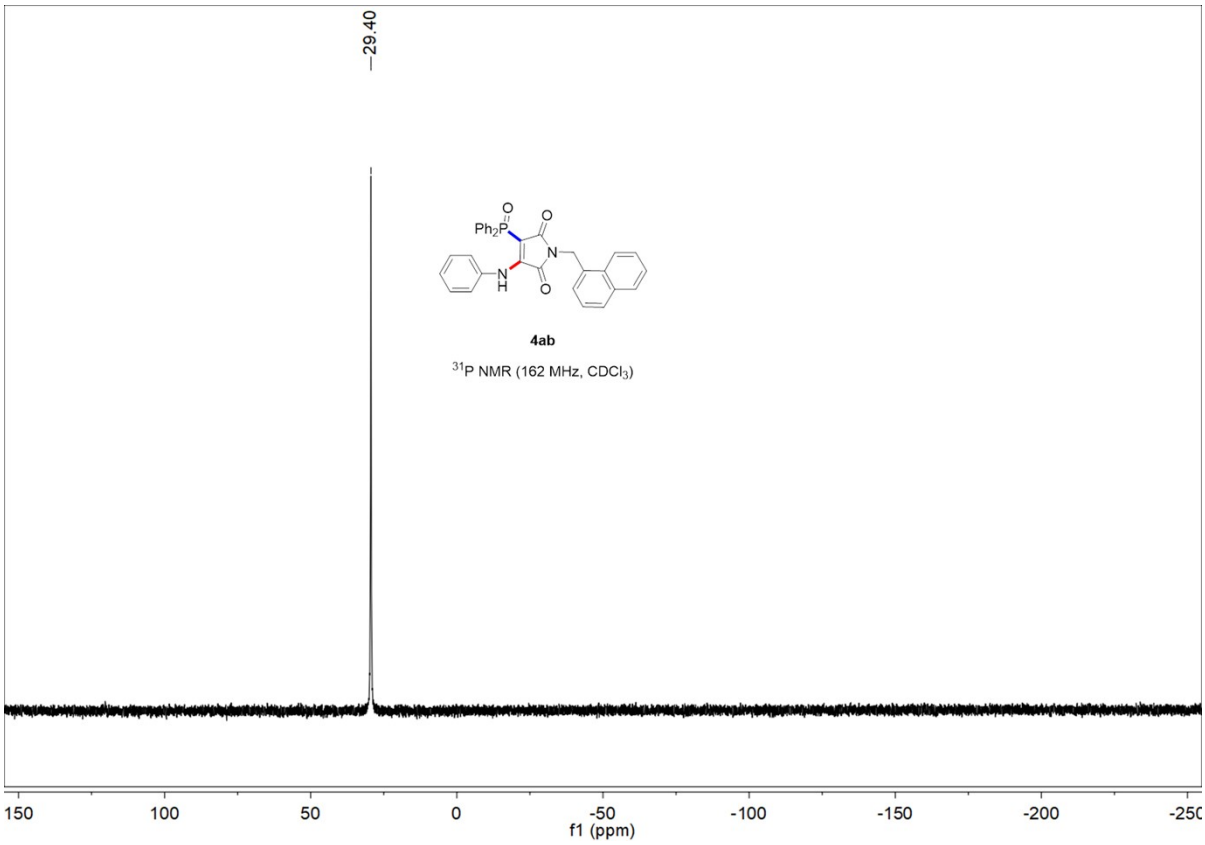
^{1}H NMR (400 MHz, $CDCl_3$)

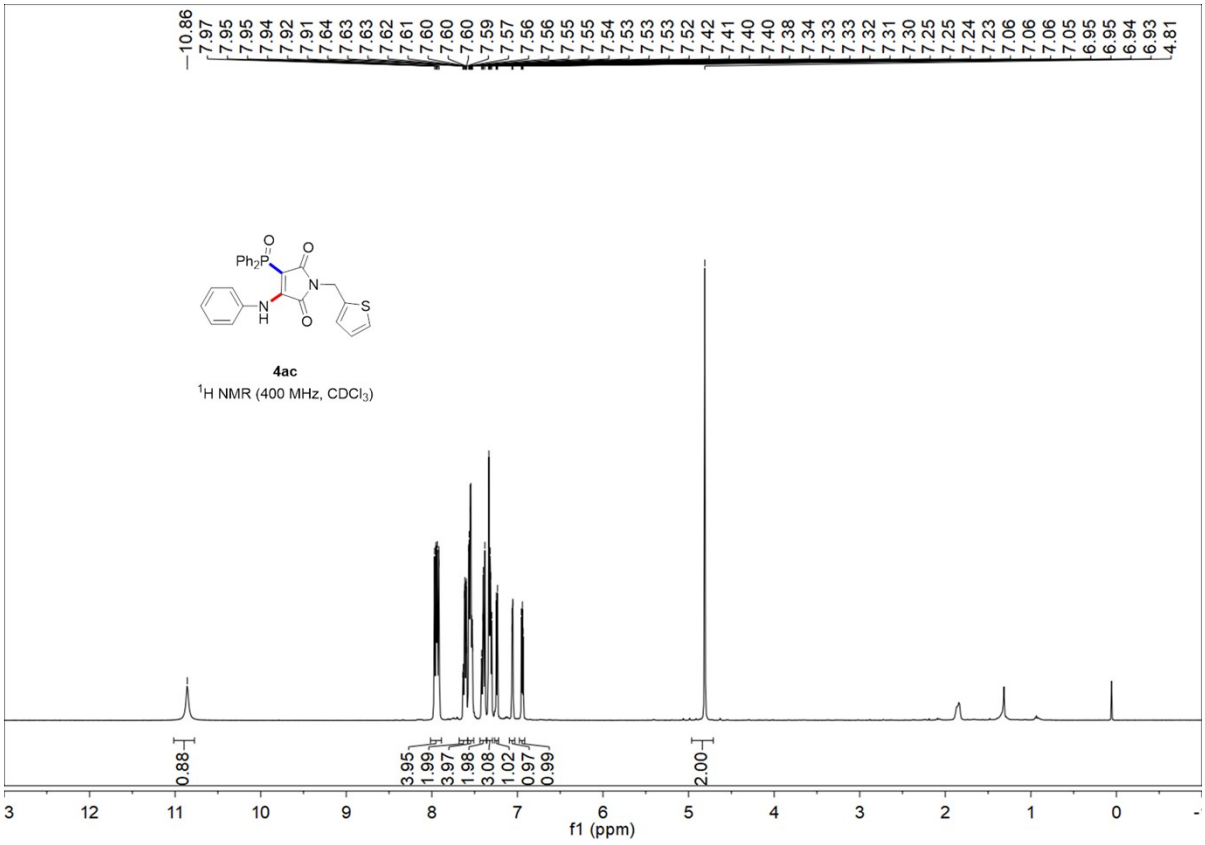

169.10
168.99
163.67
156.32
136.61
136.35
133.00
132.74
132.54
132.51
132.14
131.90
131.51
131.39
130.70
130.66
128.94
128.77
128.64
128.15
127.38
124.88
88.60
87.43
40.54
4z
13C NMR (100 MHz, CDCl3)
f1 (ppm)

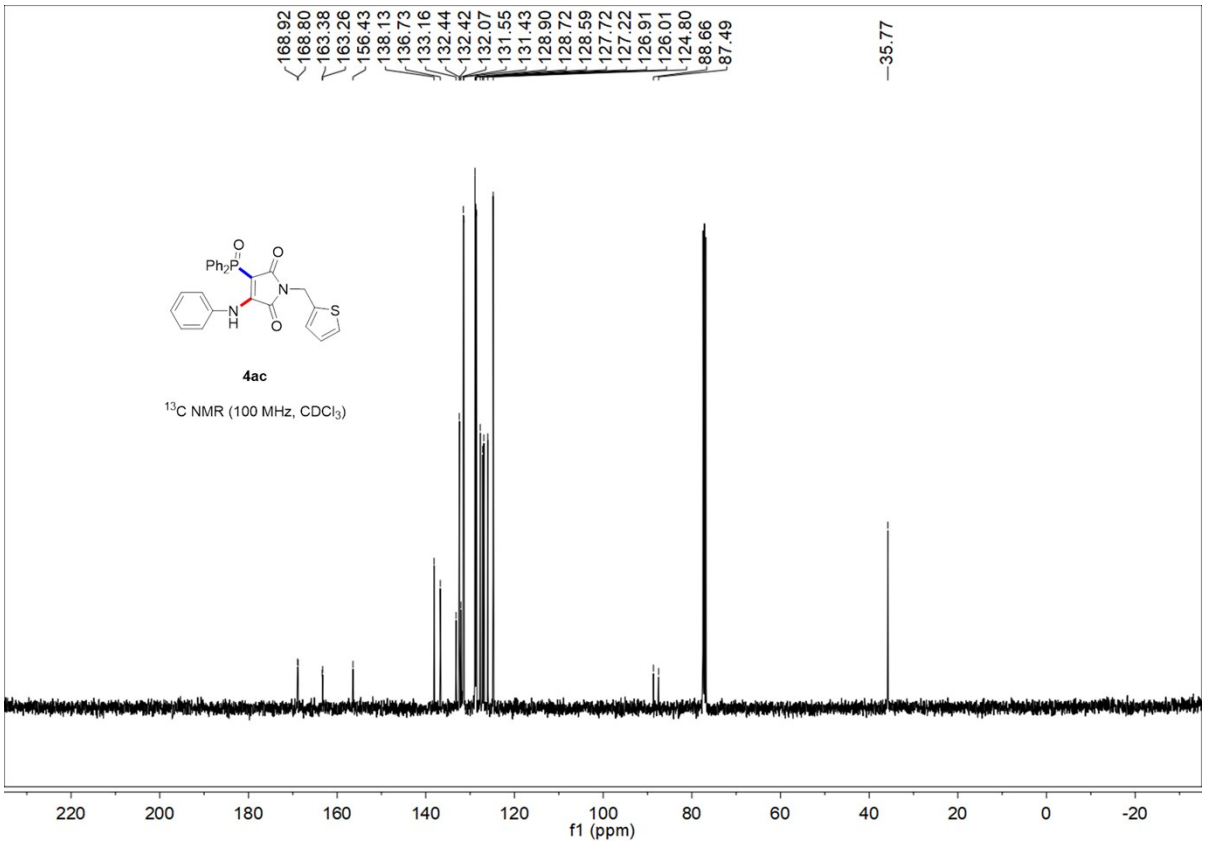

29.24
4z
31P NMR (162 MHz, CDCl3)
f1 (ppm)

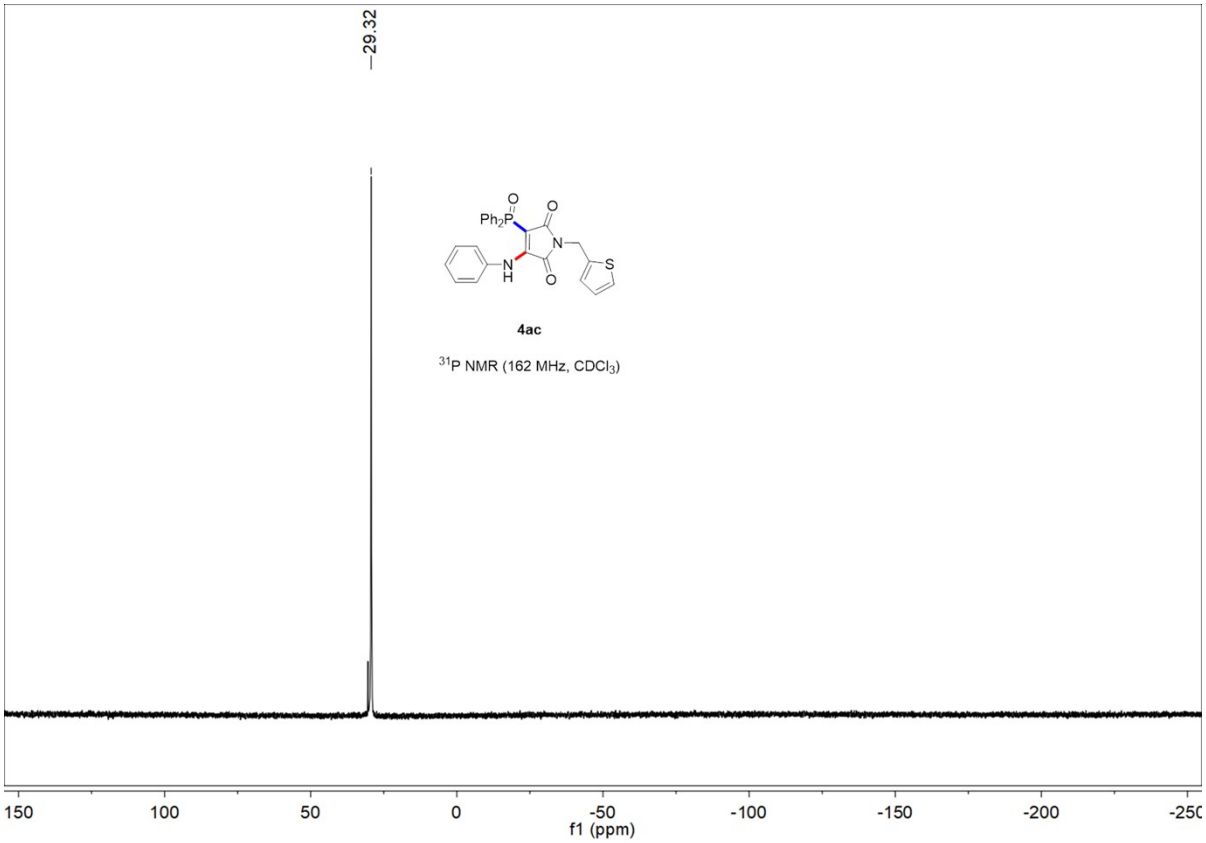

10.83
8.29
8.28
8.27
8.26
7.96
7.96
7.96
7.94
7.94
7.93
7.93
7.92
7.91
7.91
7.90
7.90
7.89
7.88
7.88
7.83
7.81
7.64
7.63
7.63
7.62
7.61
7.60
7.60
7.60
7.59
7.56
7.56
7.55
7.55
7.54
7.54
7.53
7.52
7.51
7.45
7.43
7.41
7.39
7.37
7.35
7.31
7.30
7.28
Ph2P
O
O
N
N
H
O
4ab
1H NMR (400 MHz, CDCl3)
0.91
0.98
4.84
1.01
1.95
6.96
1.03
2.03
3.45
2.00
f1 (ppm)

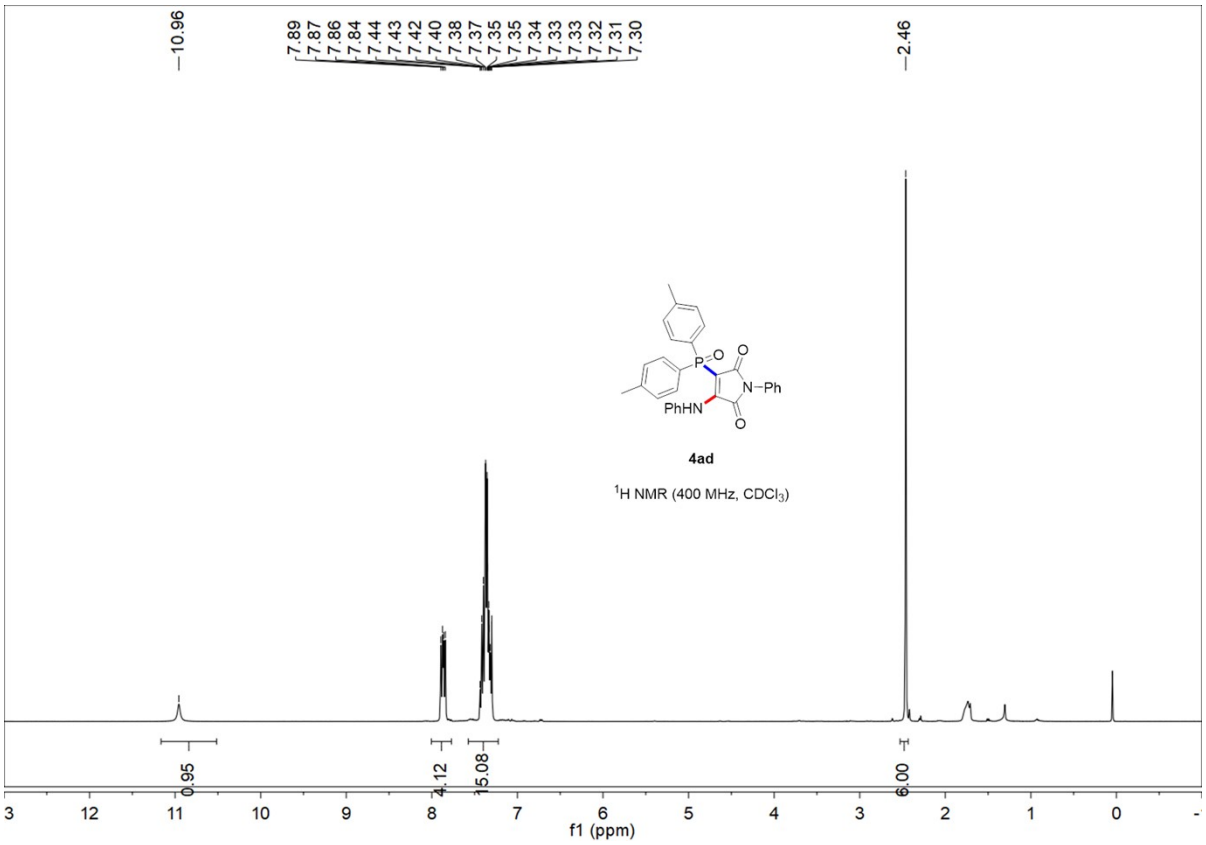

169.51
169.39
164.05
163.92
156.30
136.74
133.82
133.15
132.46
132.43
132.06
131.58
131.46
131.39
131.28
128.88
128.80
128.74
128.72
128.59
127.94
127.17
126.49
125.87
125.38
124.78
123.61
88.72
87.54
39.75
Ph2P
O
O
N
N
H
O
4ab
13C NMR (100 MHz, CDCl3)
f1 (ppm)

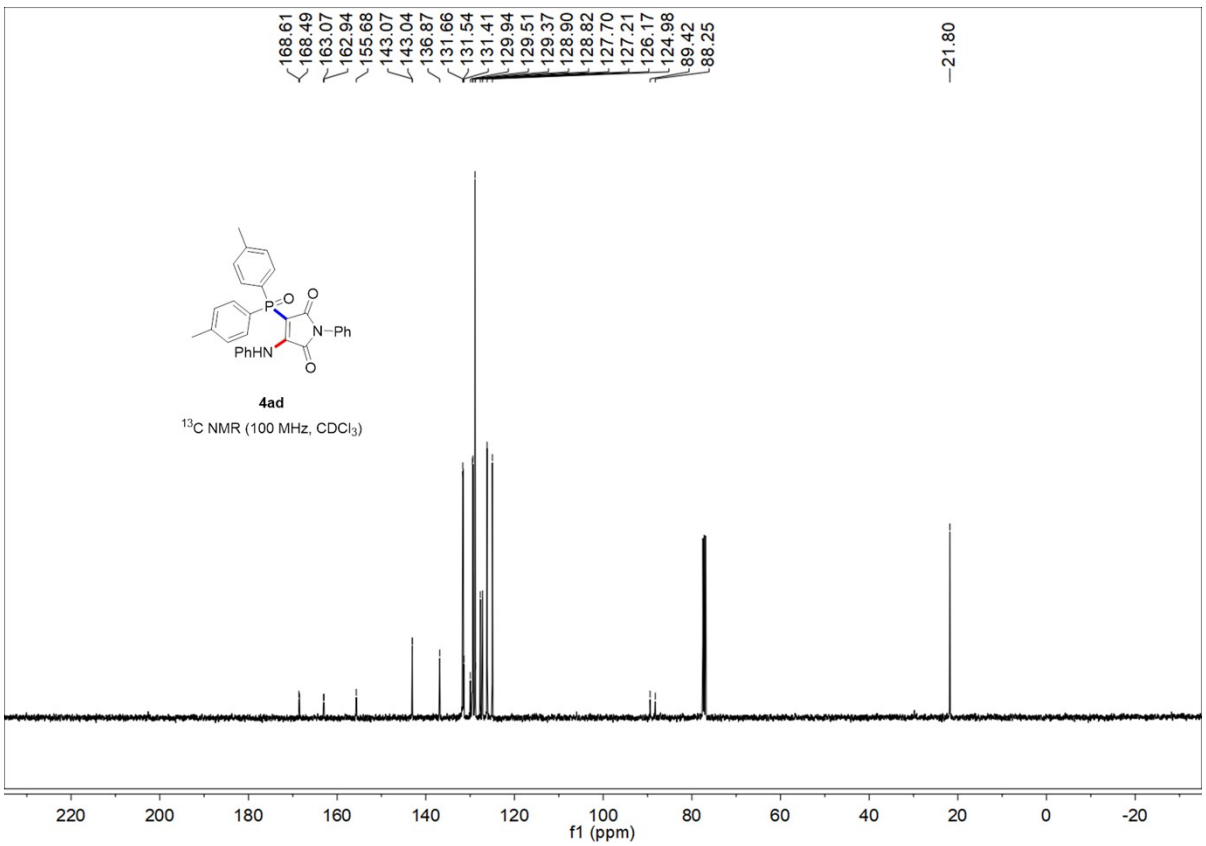

29.40
Ph2P
O
O
N
H
O
O
4ab
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

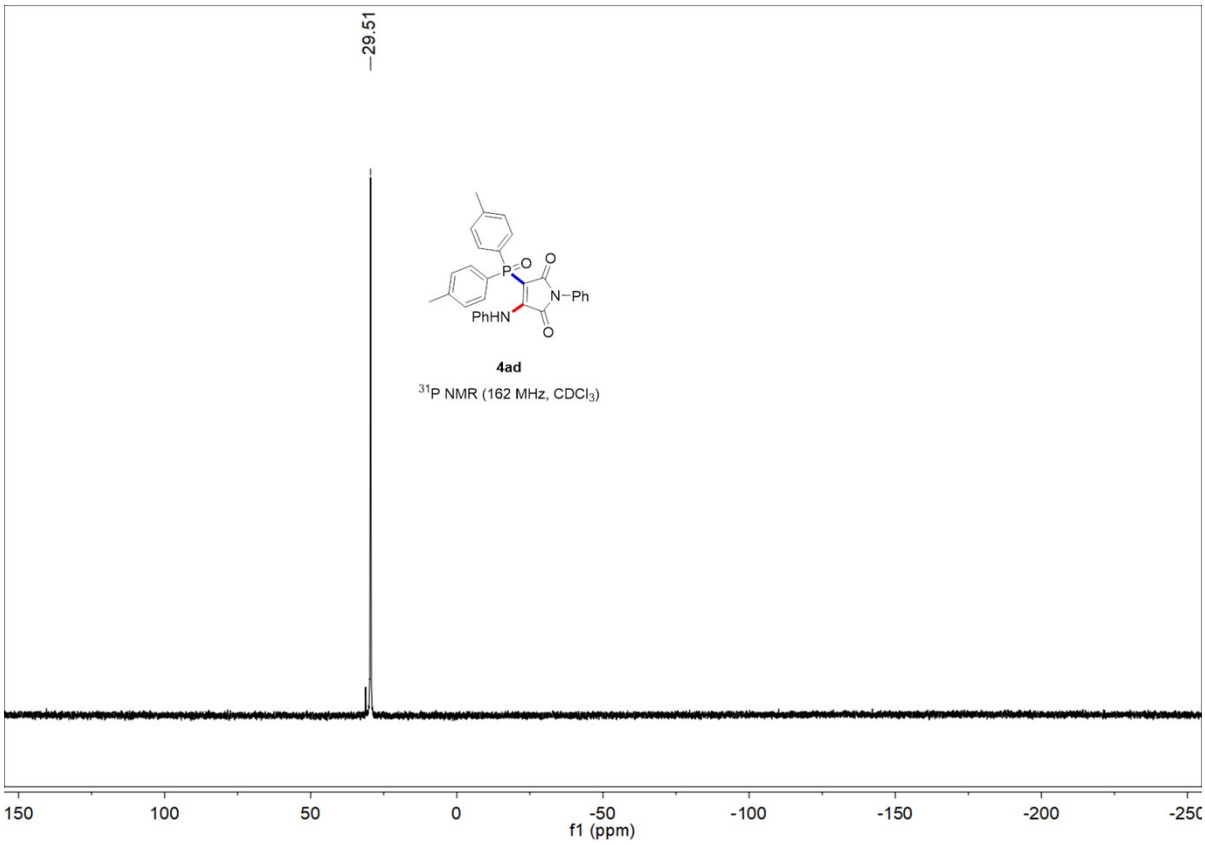

10.86
7.97
7.95
7.95
7.94
7.92
7.91
7.64
7.63
7.63
7.62
7.61
7.60
7.60
7.60
7.59
7.57
7.56
7.56
7.55
7.55
7.54
7.53
7.53
7.53
7.52
7.42
7.41
7.40
7.40
7.38
7.34
7.33
7.33
7.32
7.31
7.30
7.25
7.25
7.24
7.23
7.06
7.06
7.05
6.95
6.95
6.94
6.93
4.81
Ph2P
O
O
N
H
N
O
S
4ac
1H NMR (400 MHz, CDCl3)
0.88
3.95
1.99
3.97
1.98
3.08
1.02
0.97
0.99
2.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

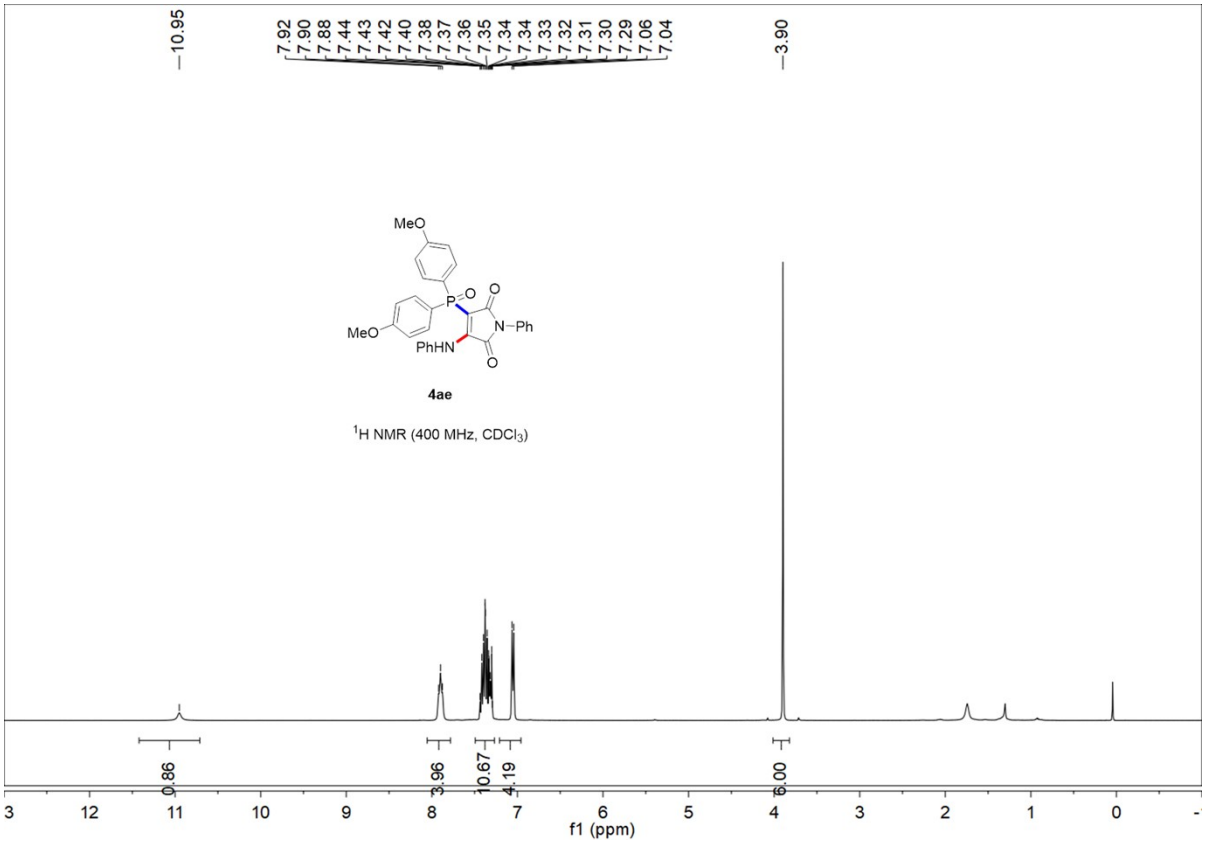

168.92
168.80
163.38
163.26
156.43
138.13
136.73
133.16
132.44
132.42
132.07
131.55
131.43
128.90
128.72
128.59
127.72
127.22
126.91
126.01
124.80
88.66
87.49
35.77
4ac
13C NMR (100 MHz, CDCl3)
f1 (ppm)

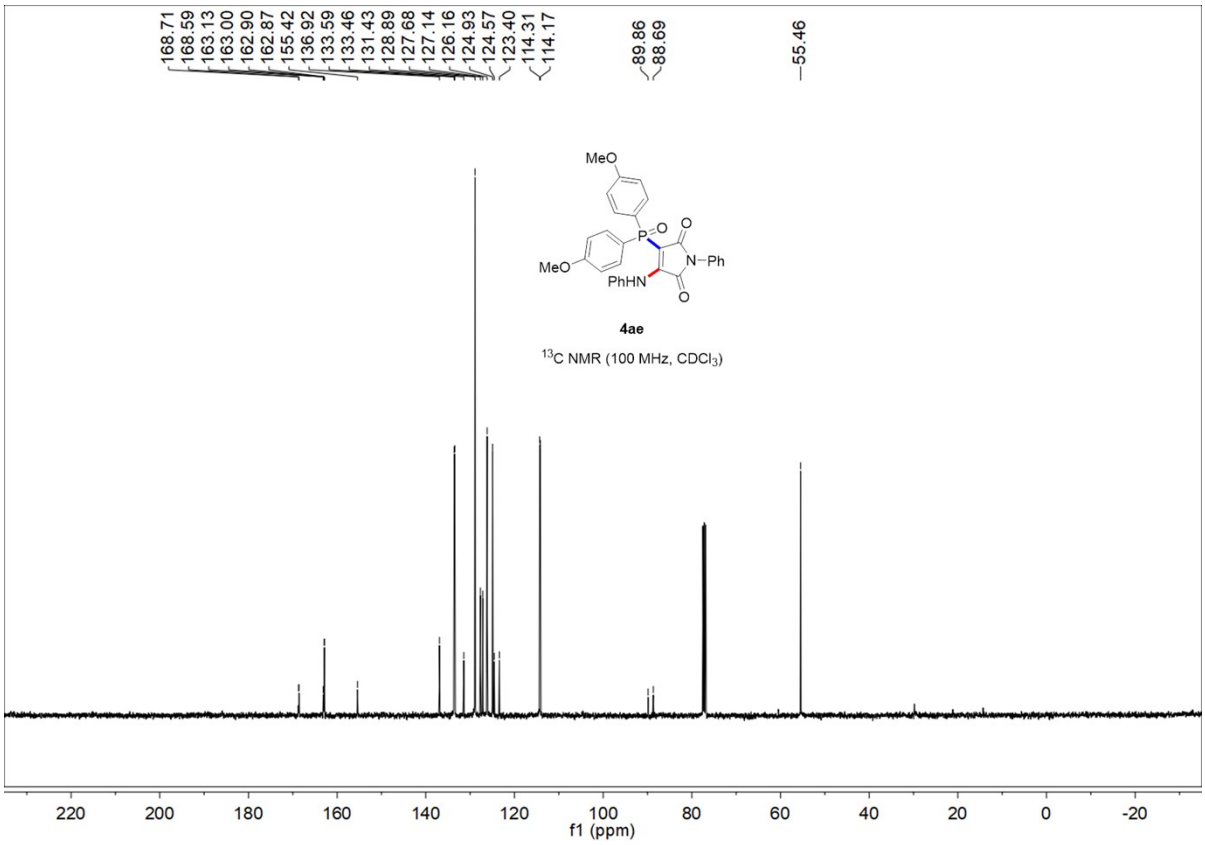

29.32
4ac
31P NMR (162 MHz, CDCl3)
f1 (ppm)

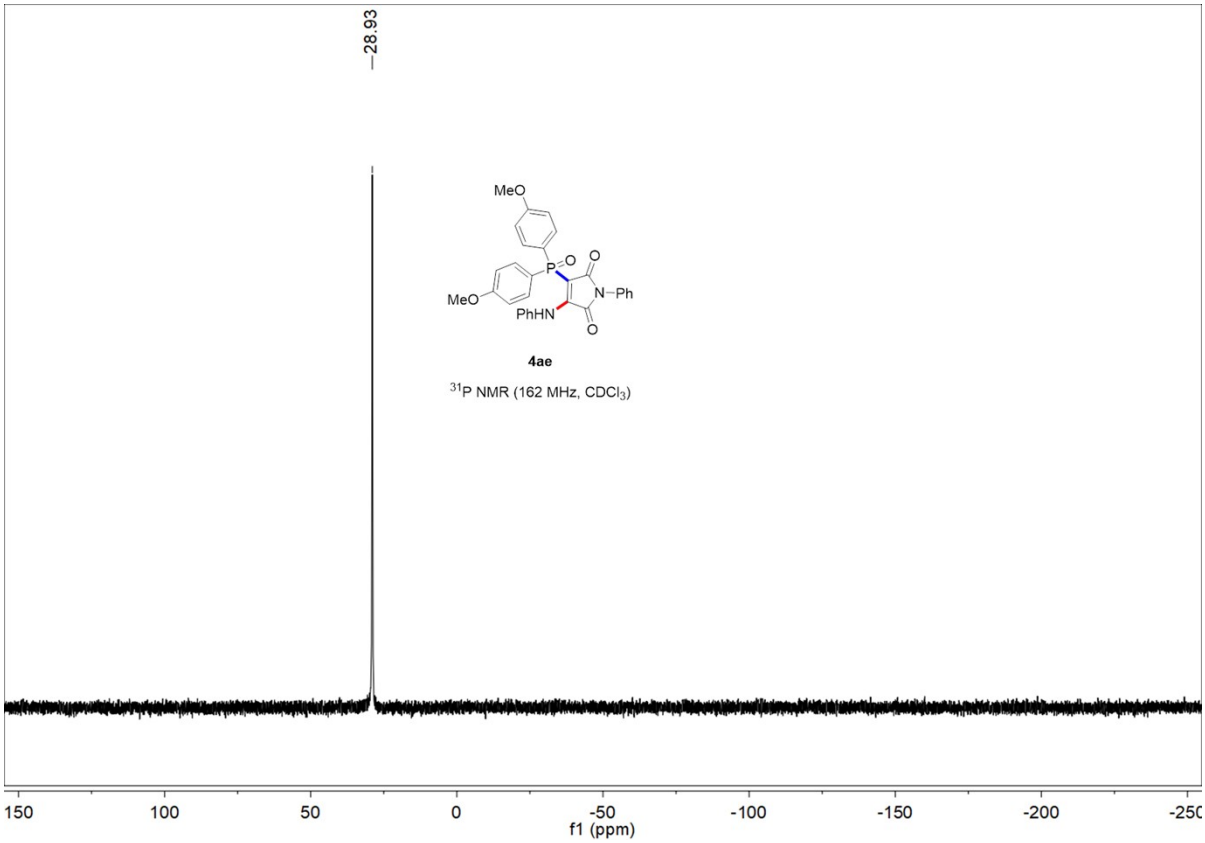

10.96
7.89
7.87
7.86
7.84
7.44
7.43
7.42
7.40
7.38
7.37
7.35
7.35
7.34
7.33
7.33
7.32
7.31
7.30
2.46
P
O
O
N–Ph
PhHN
O
4ad
^{1}H NMR (400 MHz, $CDCl_3$)
0.95
4.12
15.08
6.00
f1 (ppm)

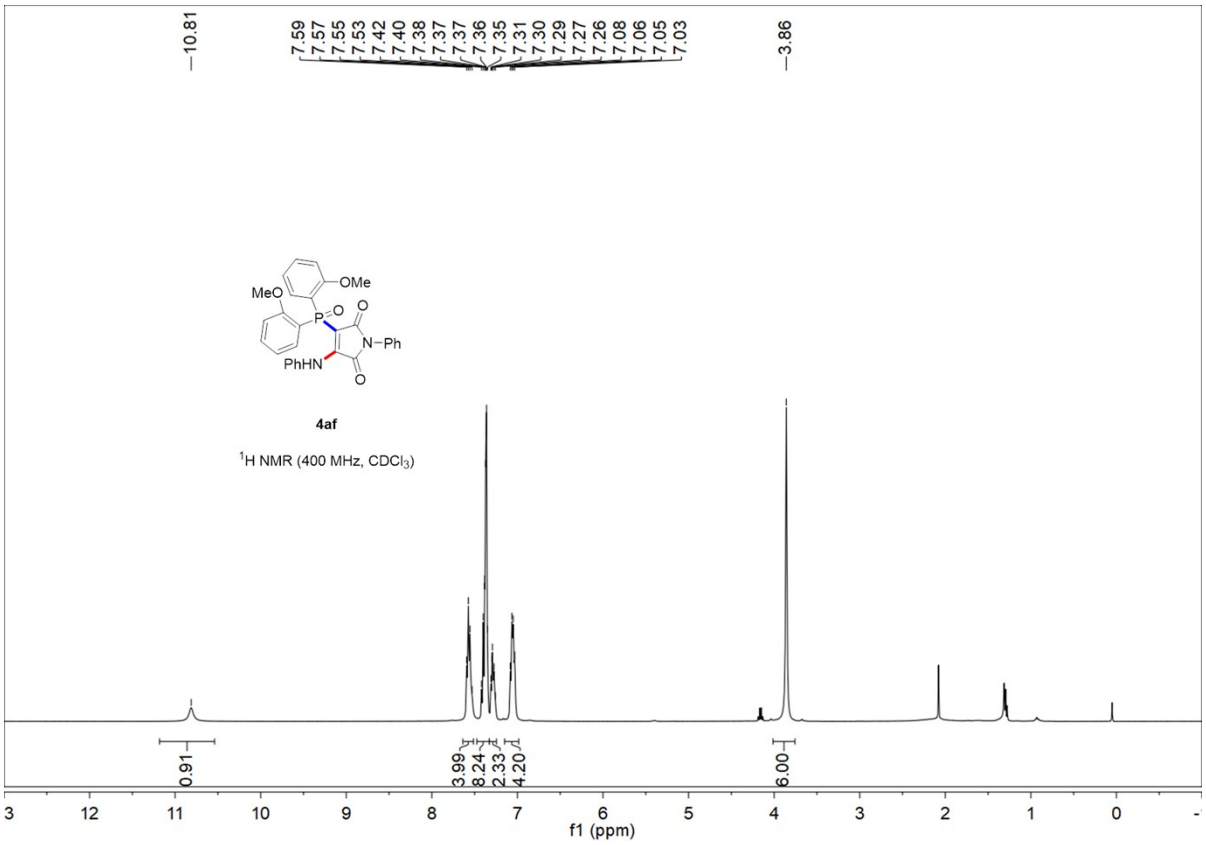

168.61
168.49
163.07
162.94
155.68
143.07
143.04
136.87
131.66
131.54
131.41
129.94
129.51
129.37
128.90
128.82
127.70
127.21
126.17
124.98
89.42
88.25
21.80
P
O
O
N–Ph
PhHN
O
4ad
^{13}C NMR (100 MHz, $CDCl_3$)
f1 (ppm)

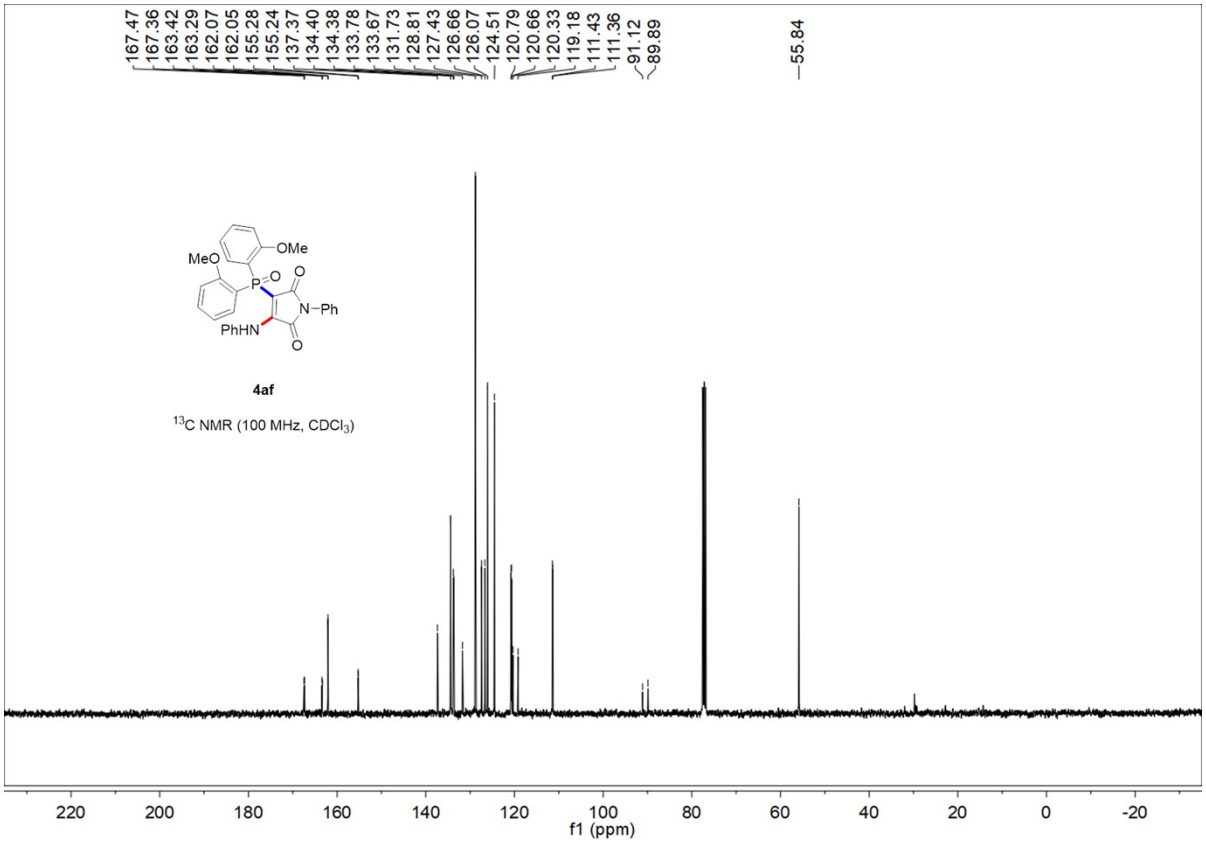

29.51
PhHN
N–Ph
4ad
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

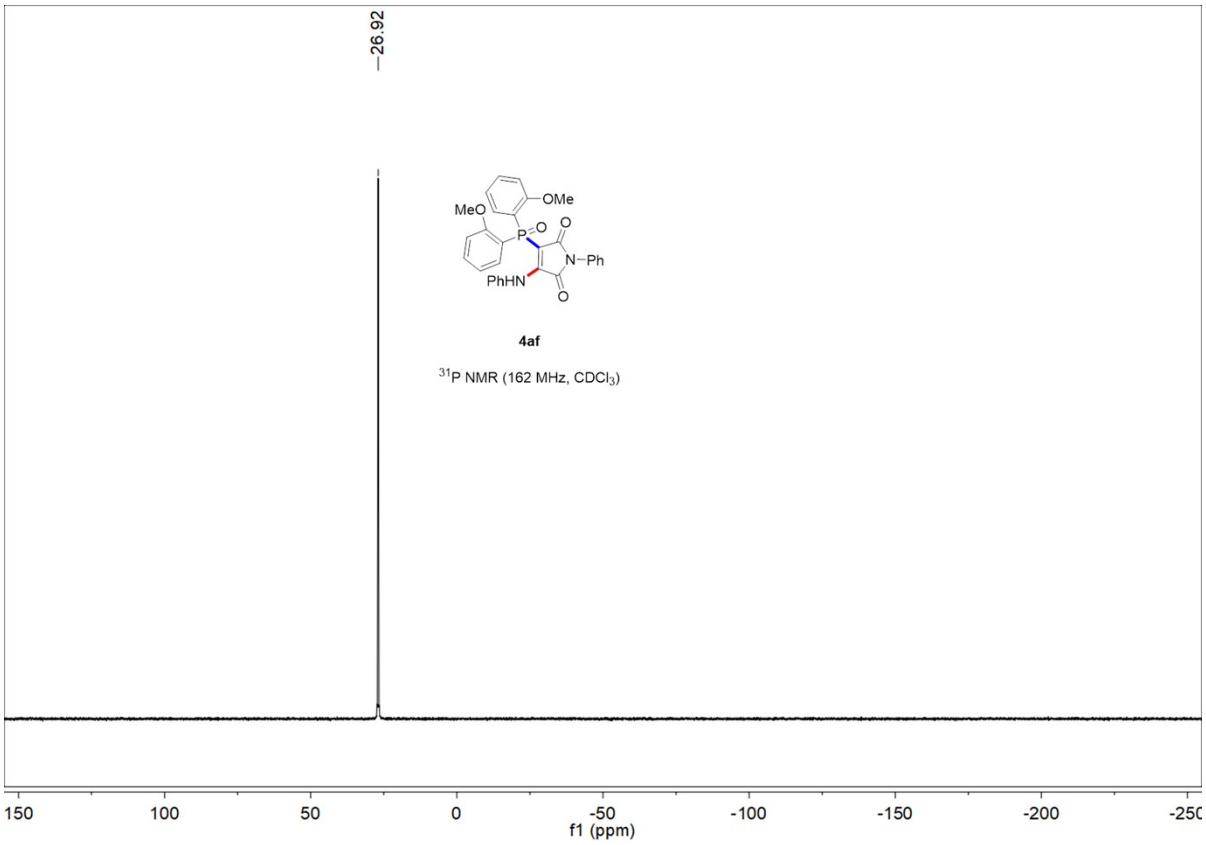

10.95
7.92
7.90
7.88
7.44
7.43
7.42
7.40
7.38
7.37
7.36
7.35
7.34
7.34
7.33
7.32
7.31
7.30
7.29
7.06
7.04
3.90
MeO
MeO
PhHN
N–Ph
4ae
1H NMR (400 MHz, CDCl3)
0.86
3.96
10.67
4.19
6.00
f1 (ppm)


168.71
168.59
163.13
163.00
162.90
162.87
155.42
136.92
133.59
133.46
131.43
128.89
127.68
127.14
126.16
124.93
124.57
123.40
114.31
114.17
89.86
88.69
55.46
MeO
MeO
PhHN
N–Ph
4ae
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)


28.93
MeO
MeO
PhHN
N–Ph
4ae
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)

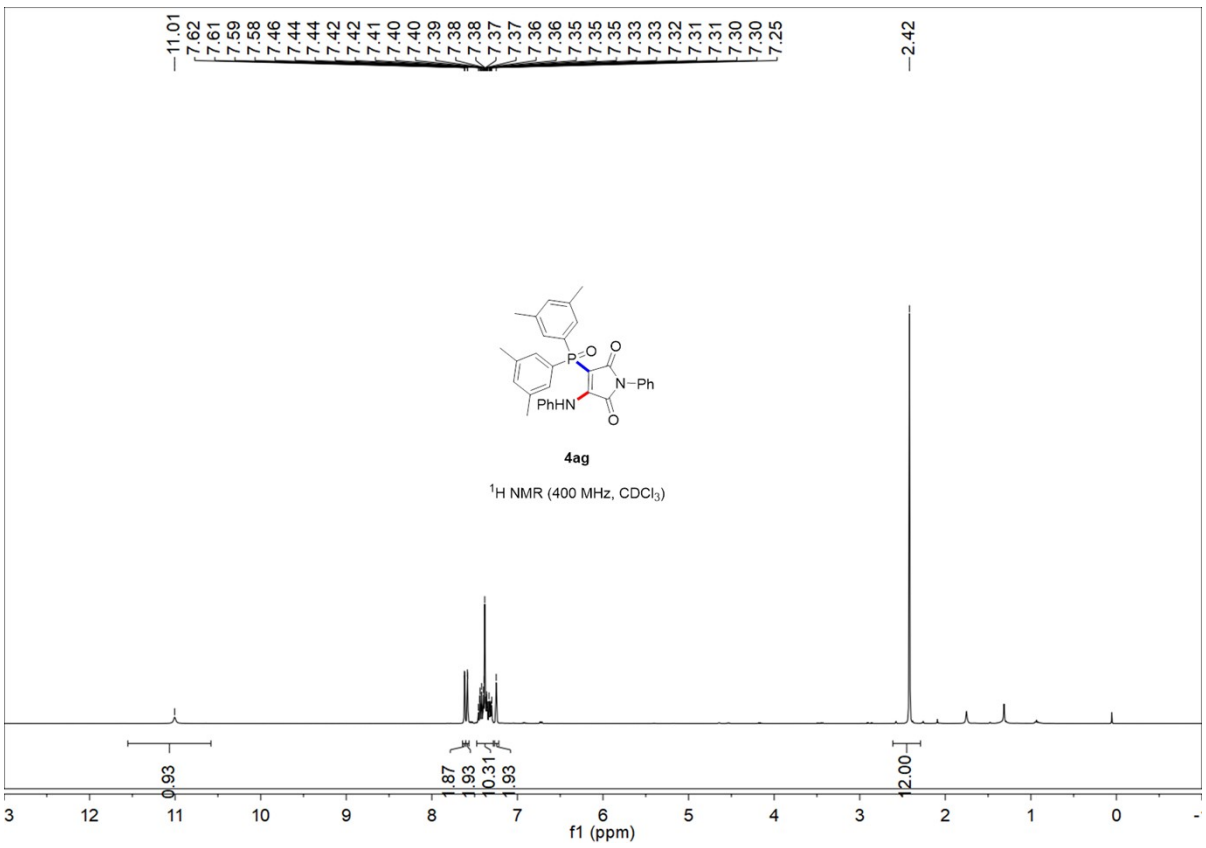

10.81
7.59
7.57
7.55
7.53
7.42
7.40
7.38
7.37
7.37
7.36
7.35
7.31
7.30
7.29
7.27
7.26
7.08
7.06
7.05
7.03
3.86
MeO
OMe
P
O
O
N−Ph
PhHN
O
4af
1H NMR (400 MHz, CDCl3)
0.91
3.99
8.24
2.33
4.20
6.00
f1 (ppm)


167.47
167.36
163.42
163.29
162.07
162.05
155.28
155.24
137.37
134.40
134.38
133.78
133.67
131.73
128.81
127.43
126.66
126.07
124.51
120.79
120.66
120.33
119.18
111.43
111.36
91.12
89.89
55.84
MeO
OMe
P
O
O
N−Ph
PhHN
O
4af
13C NMR (100 MHz, CDCl3)
f1 (ppm)



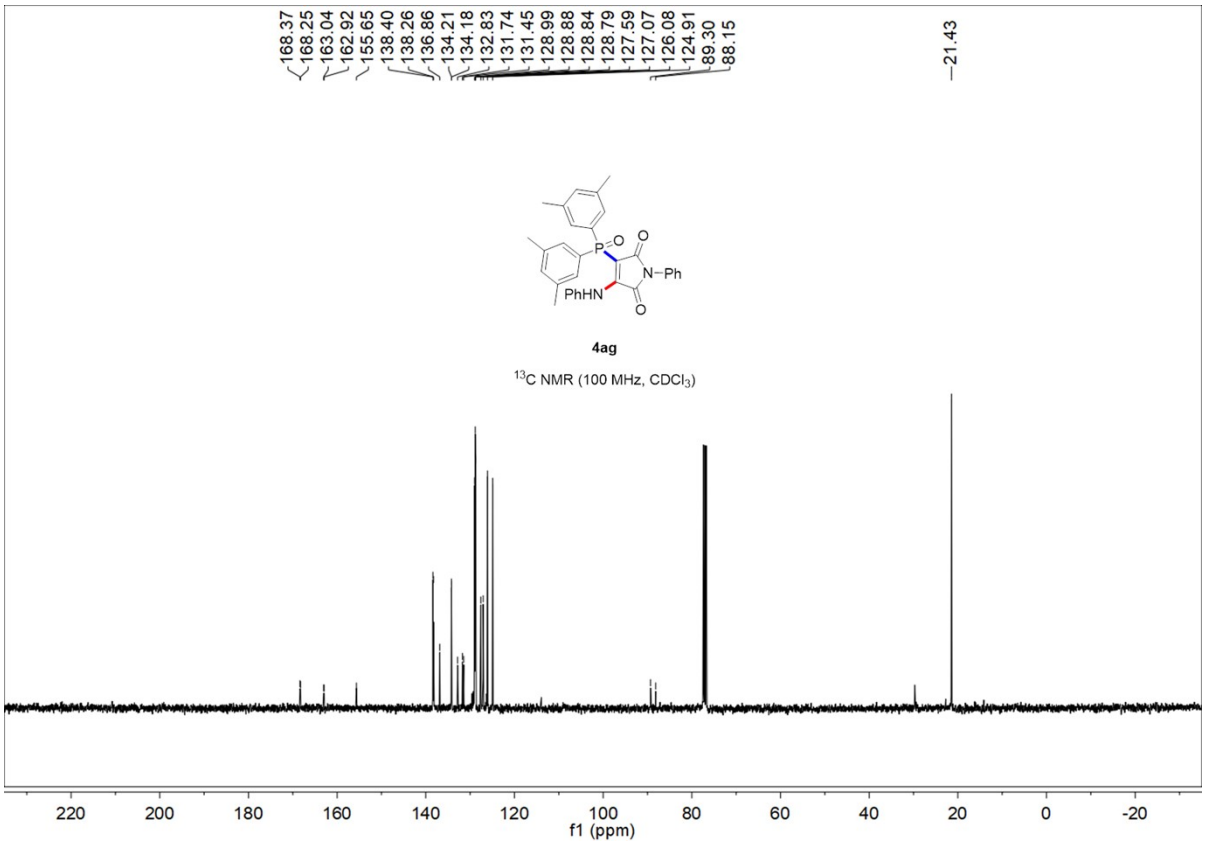
4af

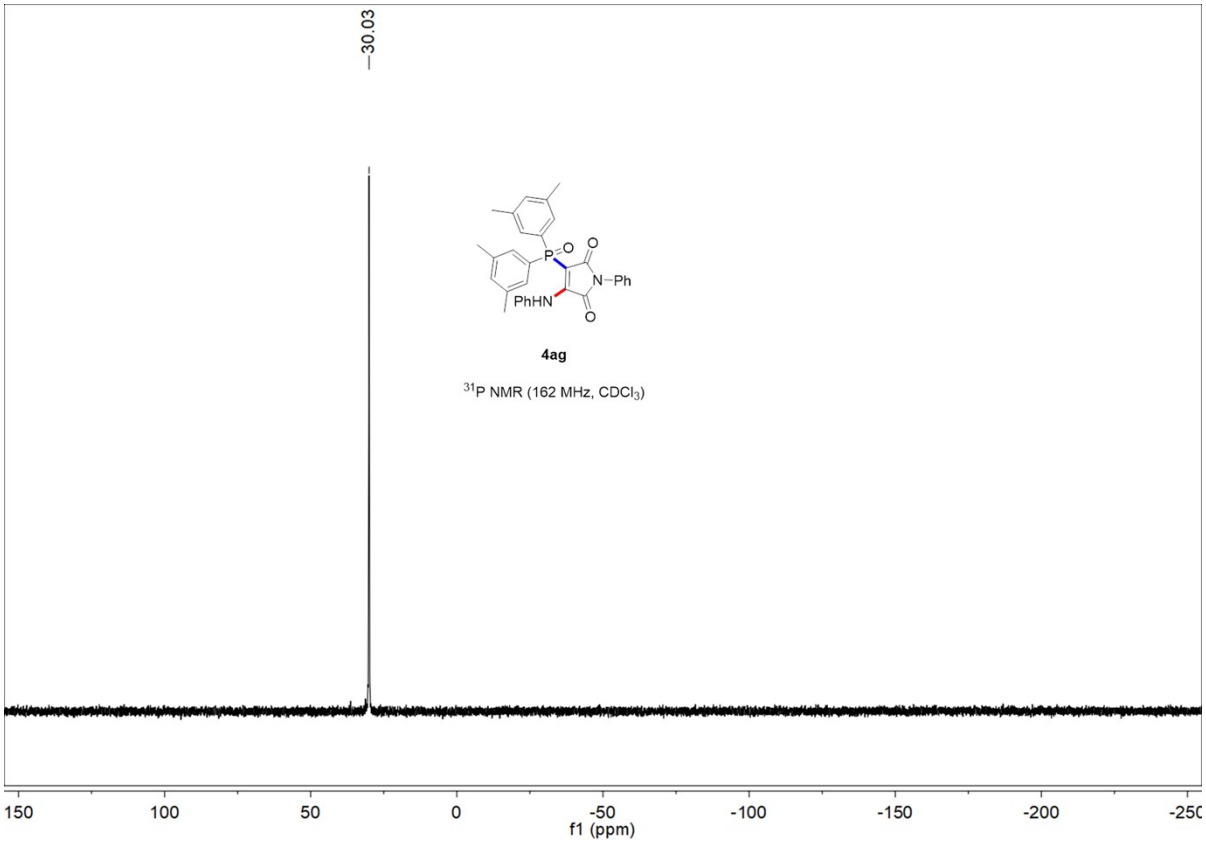
^{31}P NMR (162 MHz, $CDCl_3$)

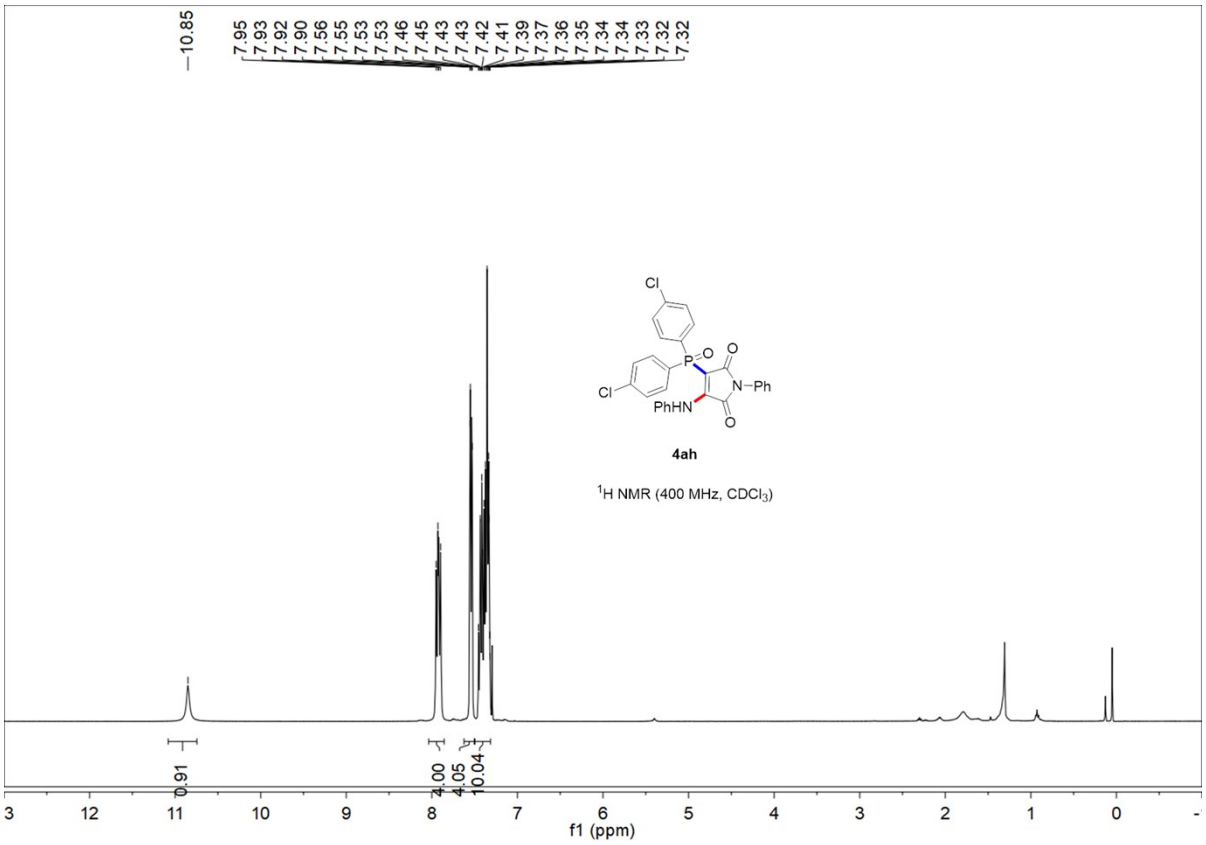


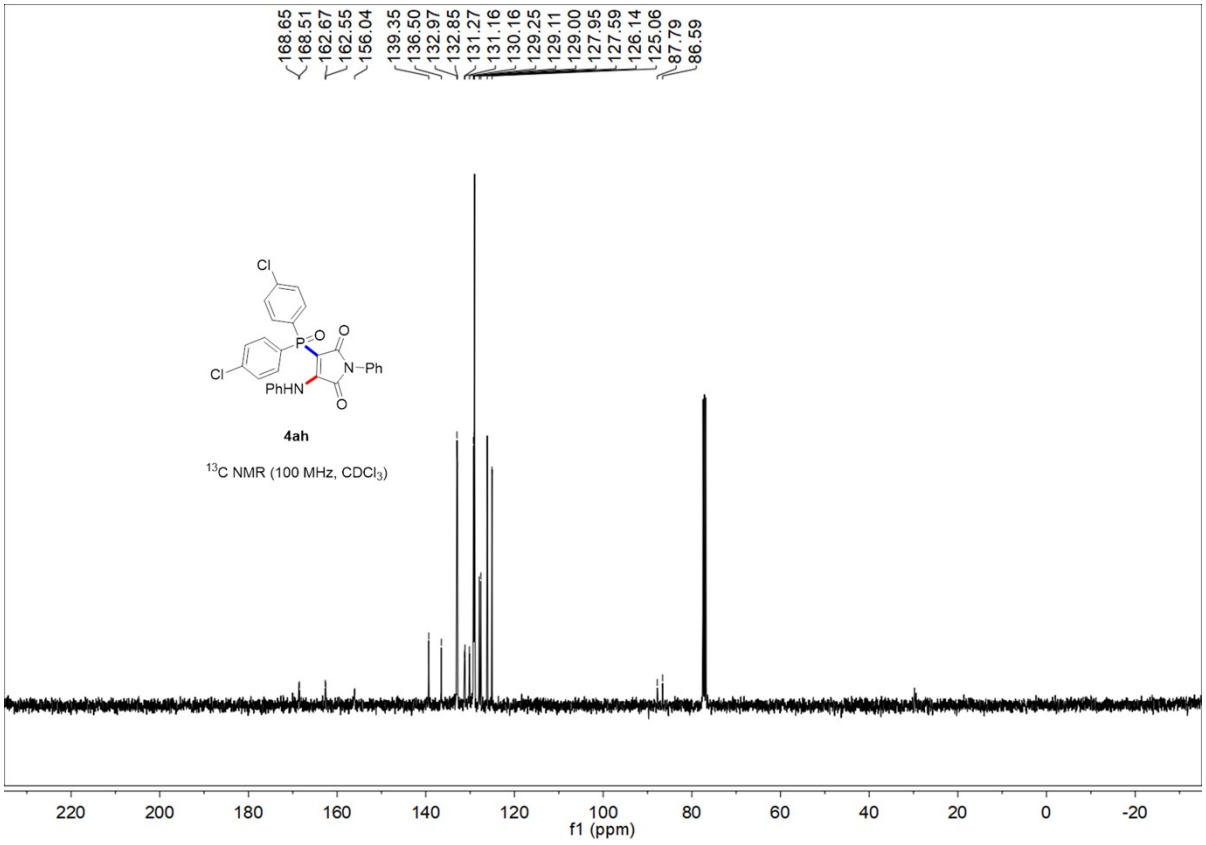
4ag

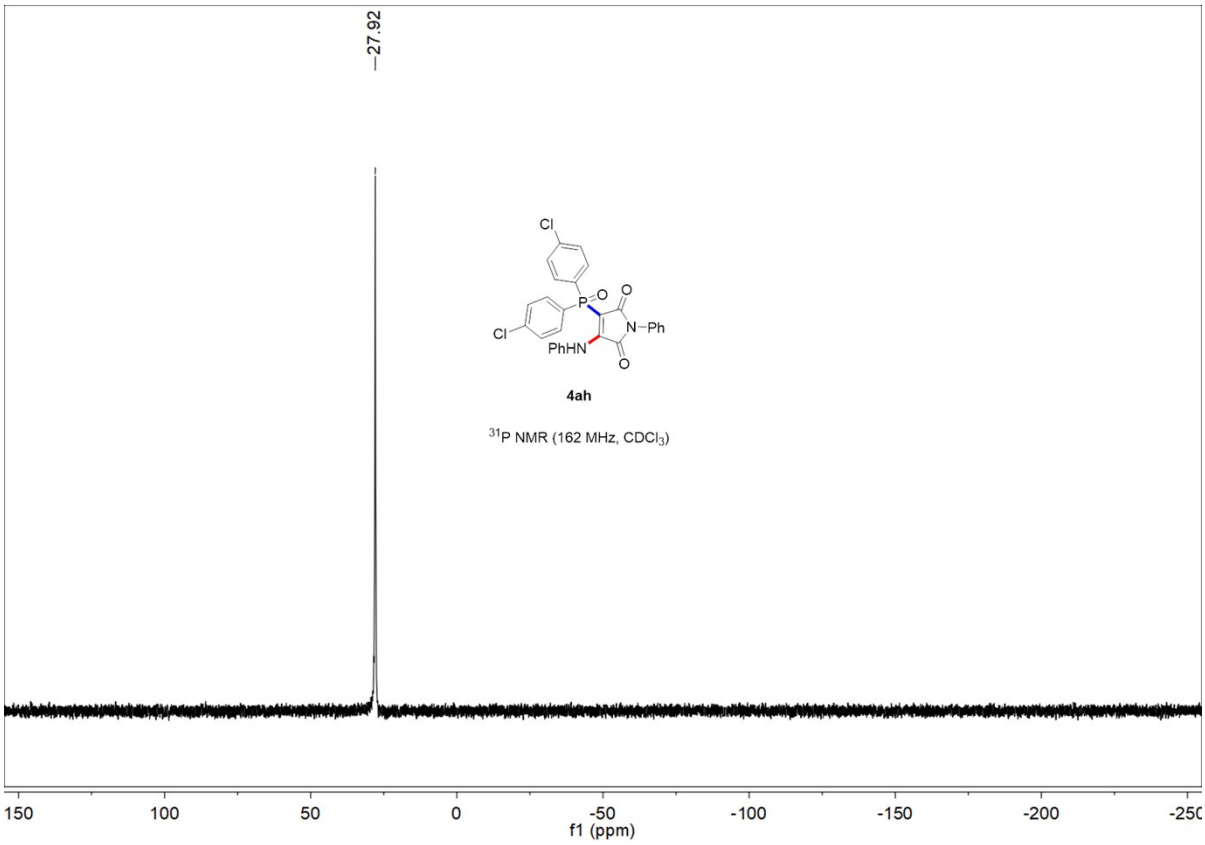
^{1}H NMR (400 MHz, $CDCl_3$)

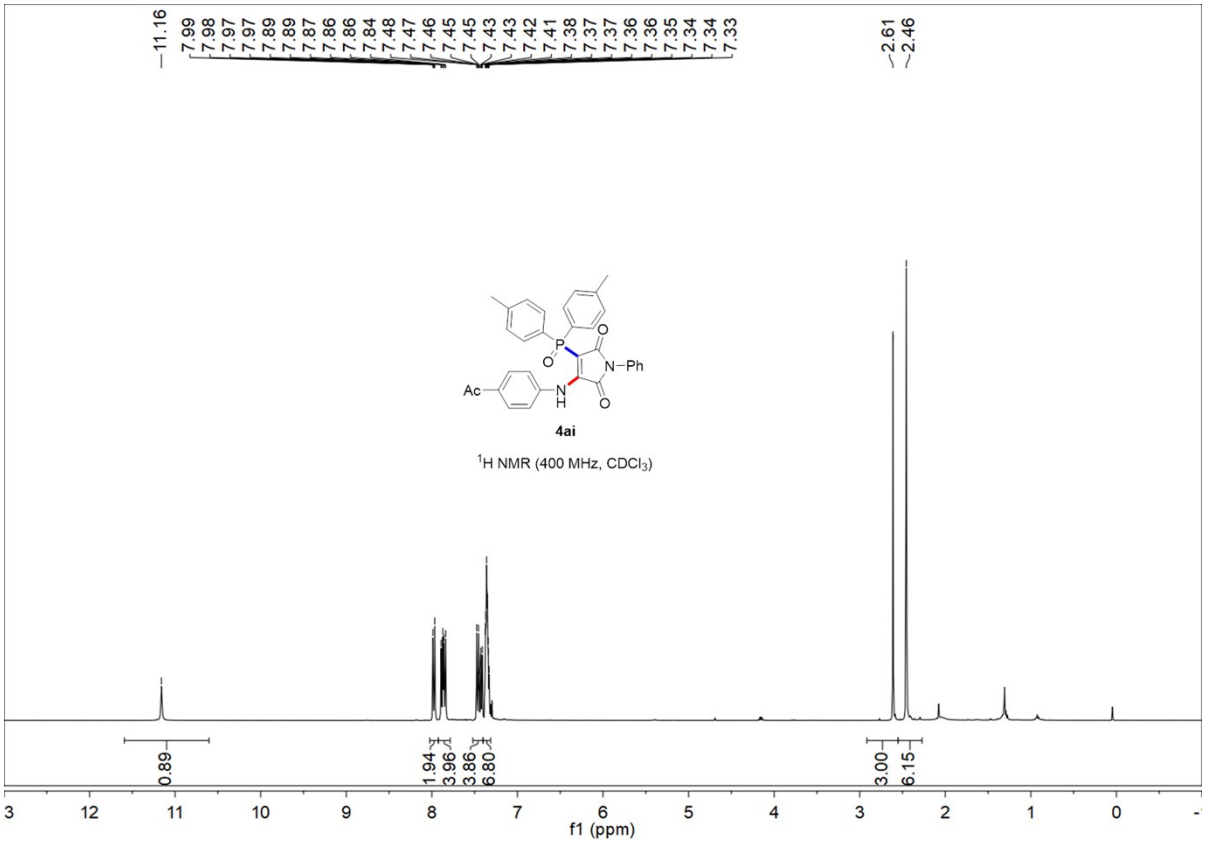

168.37
168.25
163.04
162.92
155.65
138.40
138.26
136.86
134.21
134.18
132.83
131.74
131.45
128.99
128.88
128.84
128.79
127.59
127.07
126.08
124.91
89.30
88.15
21.43
O
P
O
N–Ph
PhHN
O
4ag
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

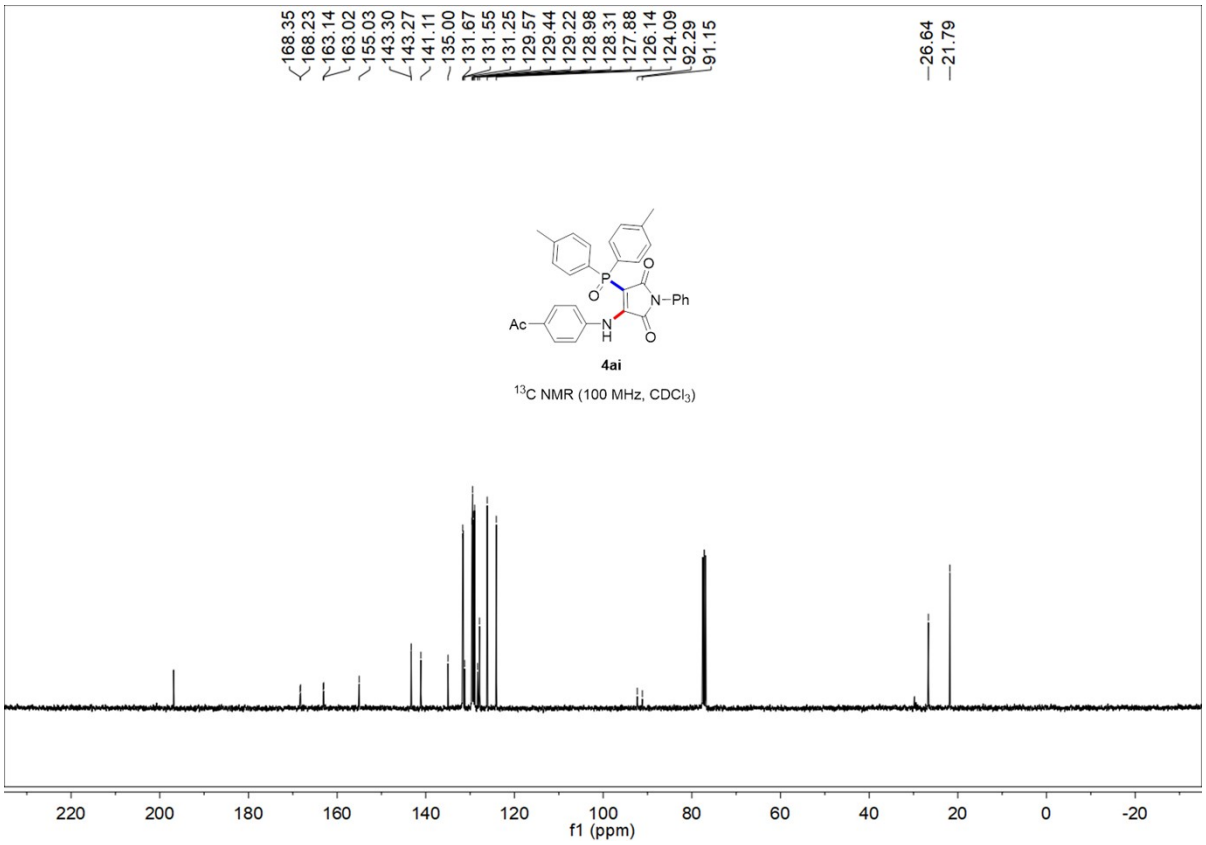

30.03
O
P
O
N–Ph
PhHN
O
4ag
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

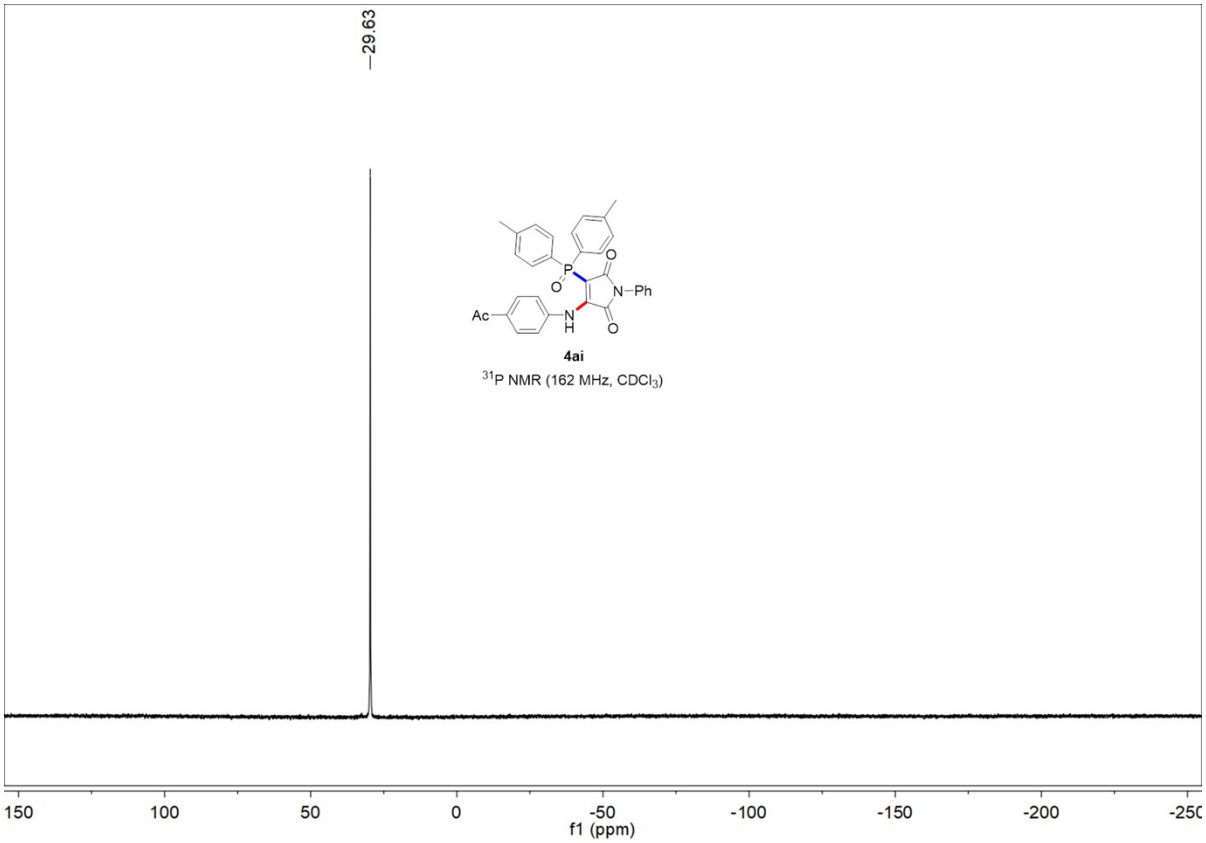

10.85
7.95
7.93
7.92
7.90
7.56
7.55
7.53
7.53
7.46
7.45
7.43
7.43
7.42
7.41
7.39
7.37
7.36
7.35
7.34
7.34
7.33
7.32
7.32
Cl
P
O
O
N–Ph
Cl
PhHN
O
4ah
^{1}H NMR (400 MHz, CDCl$_3$)
0.91
4.00
4.05
10.04
3
12
11
10
9
8
7
6
5
4
3
2
1
0
-
f1 (ppm)

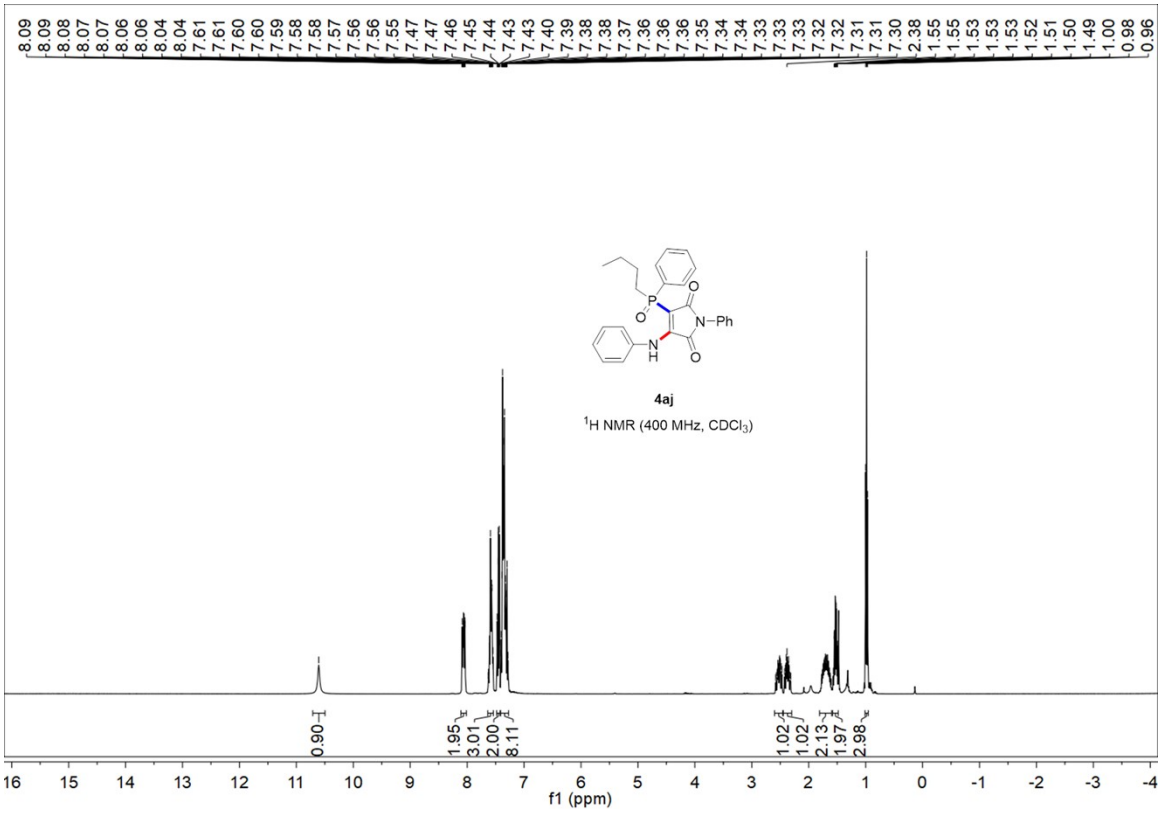

168.65
168.51
162.67
162.55
156.04
139.35
136.50
132.97
132.85
131.27
131.16
130.16
129.25
129.11
129.00
127.95
127.59
126.14
125.06
87.79
86.59
Cl
P
O
O
N–Ph
Cl
PhHN
O
4ah
^{13}C NMR (100 MHz, CDCl$_3$)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

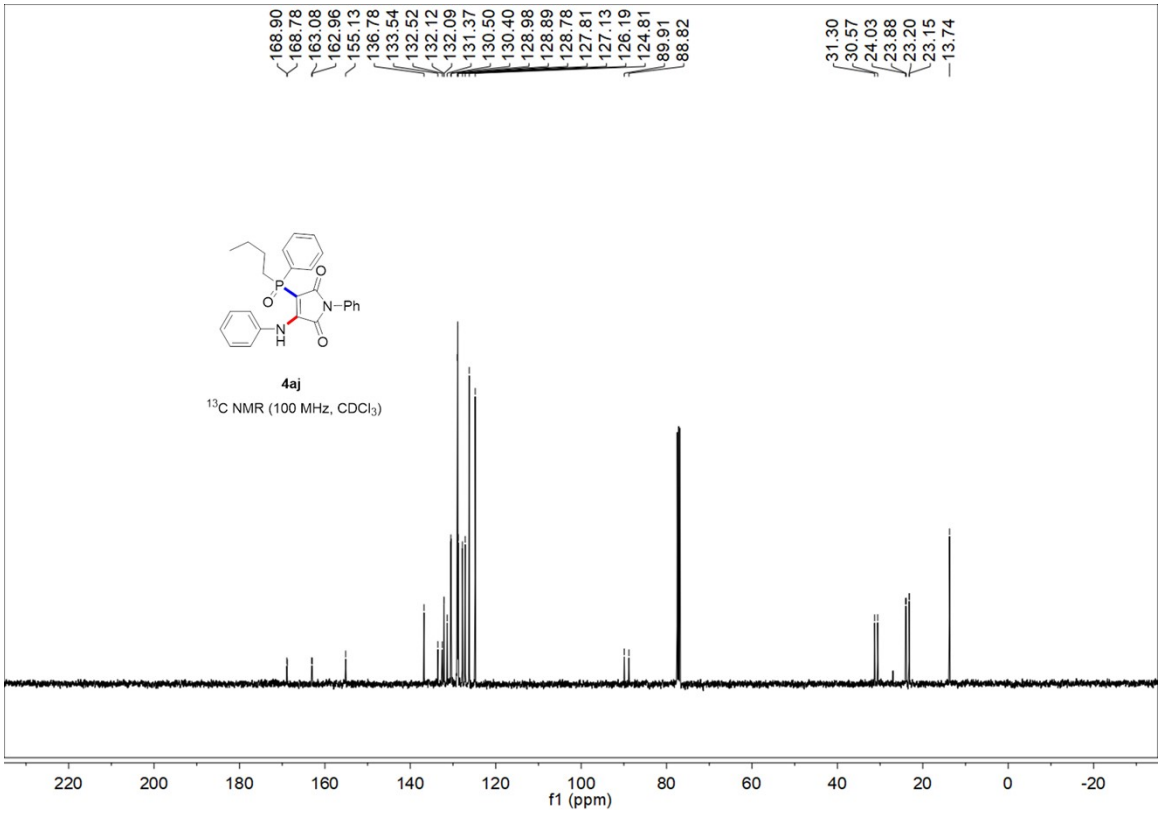

27.92
Cl
Cl
PhHN
N–Ph
4ah
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)

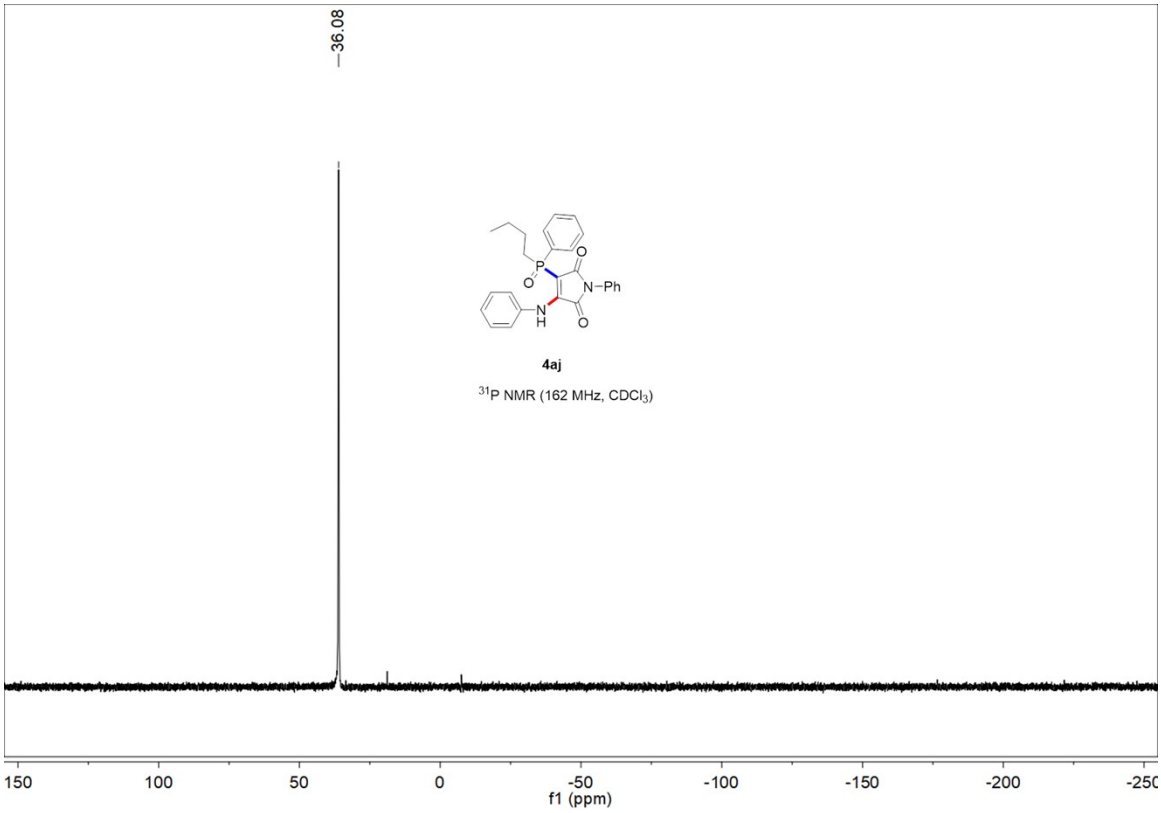

11.16
7.99
7.98
7.97
7.97
7.89
7.89
7.87
7.86
7.86
7.84
7.48
7.47
7.46
7.45
7.45
7.43
7.43
7.42
7.41
7.38
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.33
2.61
2.46
Ac
N–Ph
4ai
1H NMR (400 MHz, CDCl3)
0.89
1.94
3.96
3.86
6.80
3.00
6.15
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

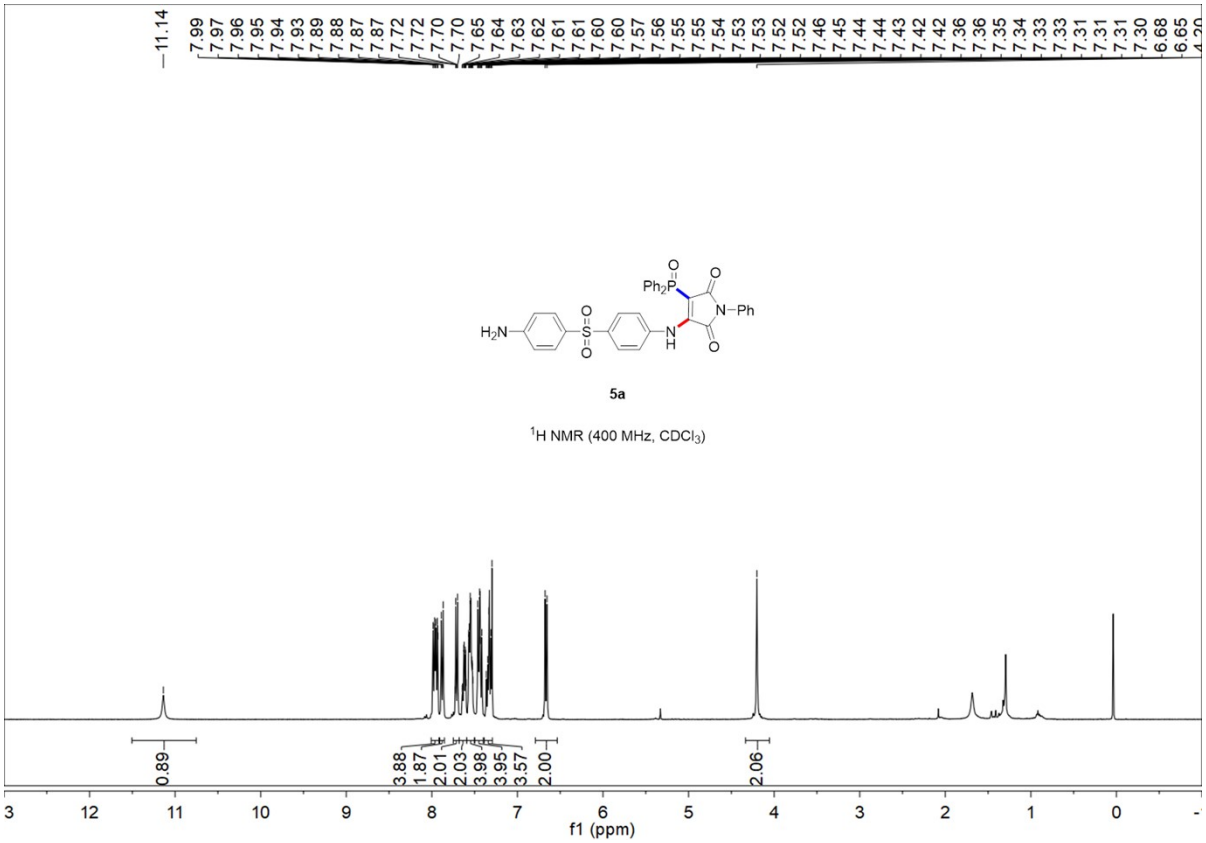

168.35
168.23
163.14
163.02
155.03
143.30
143.27
141.11
135.00
131.67
131.55
131.25
129.57
129.44
129.22
128.98
128.31
127.88
126.14
124.09
92.29
91.15
26.64
21.79
Ac
N
H
N–Ph
O
P
O
O
O
4ai
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

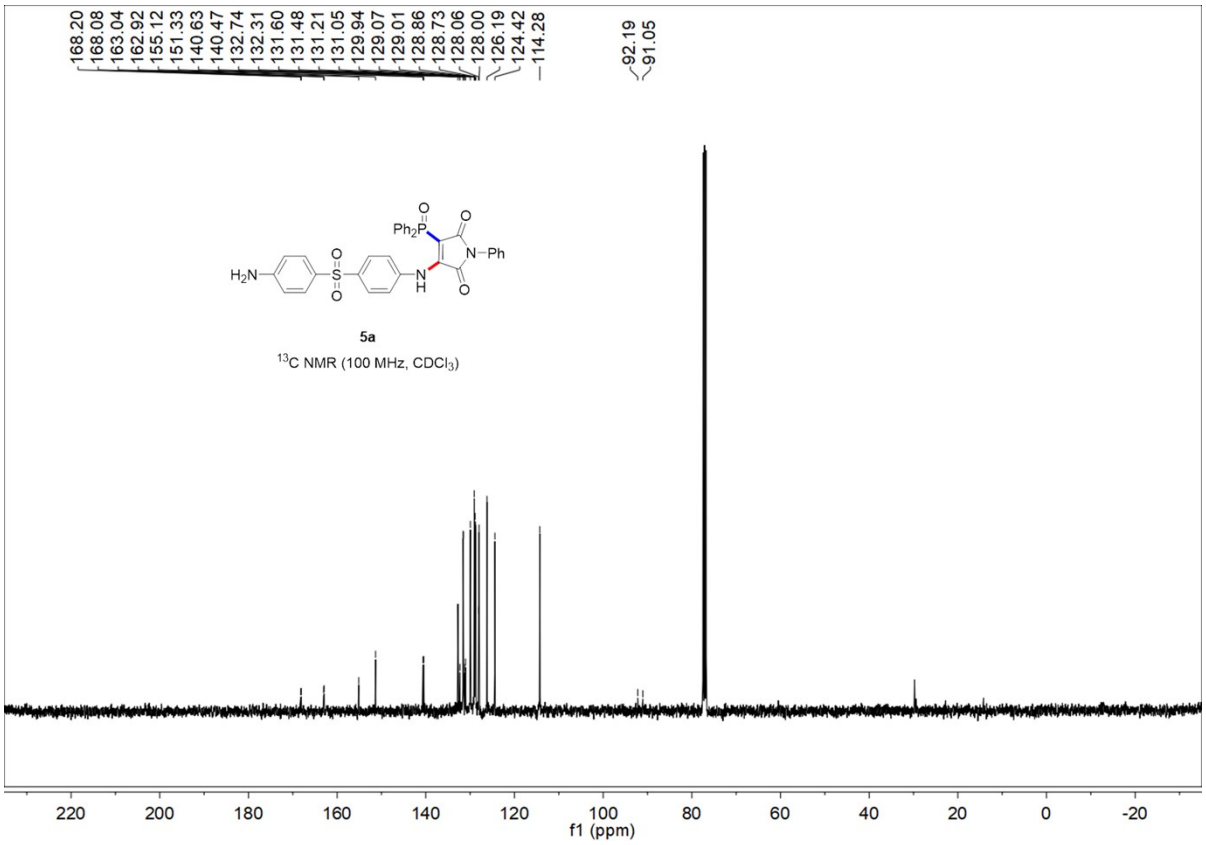

29.63
Ac
N
H
N–Ph
O
P
O
O
O
4ai
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)


4aj
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)


4aj
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)

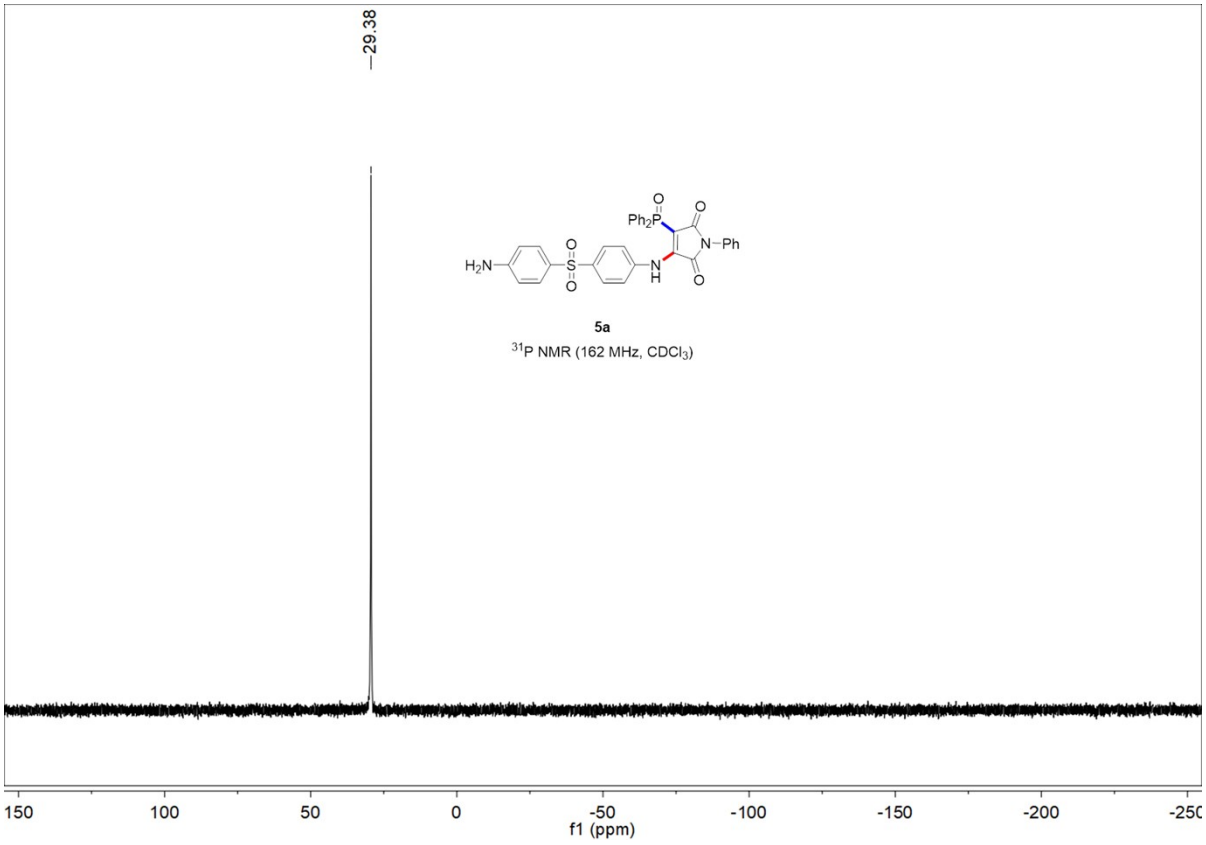

36.08
4aj
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)


11.14
5a
1H NMR (400 MHz, CDCl3)
0.89
3.88
1.87
2.01
2.03
3.98
3.95
3.57
2.00
2.06
f1 (ppm)



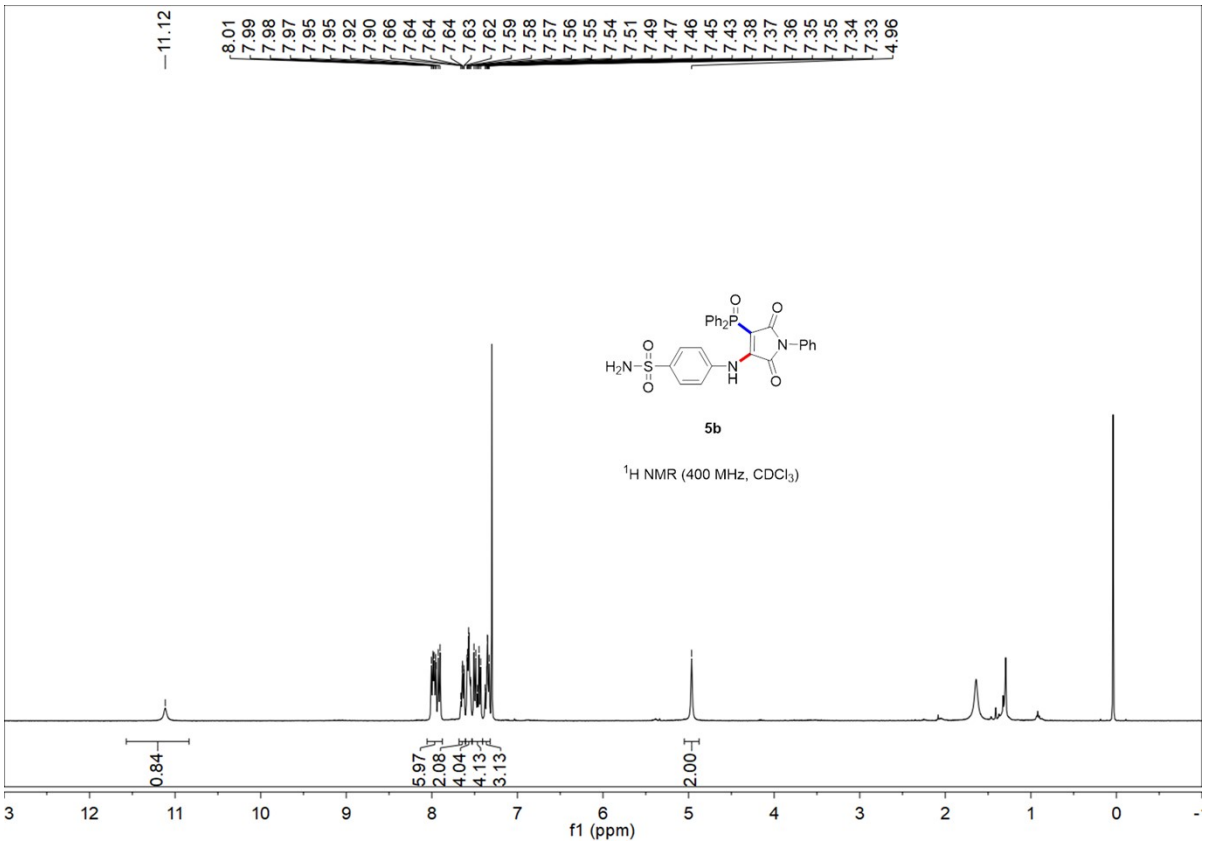
5a

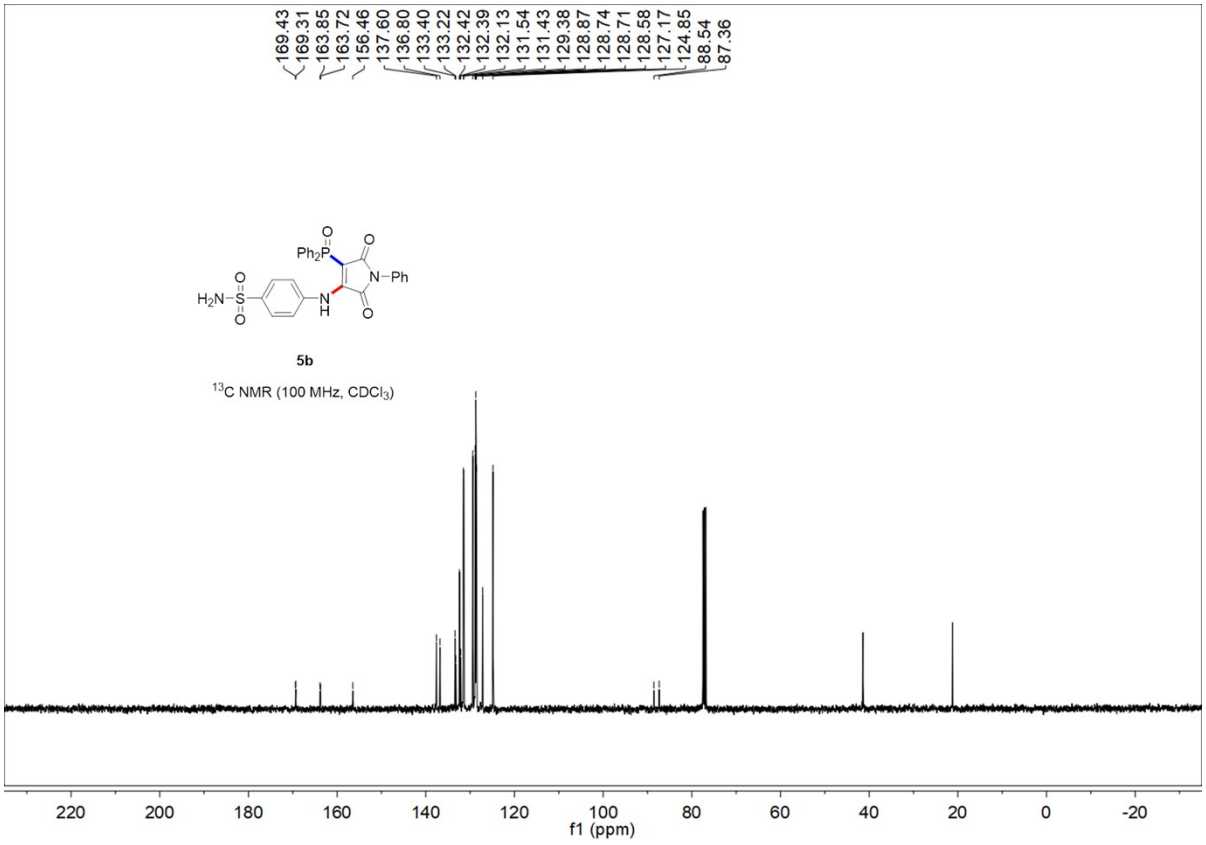
^{13}C NMR (100 MHz, $CDCl_3$)

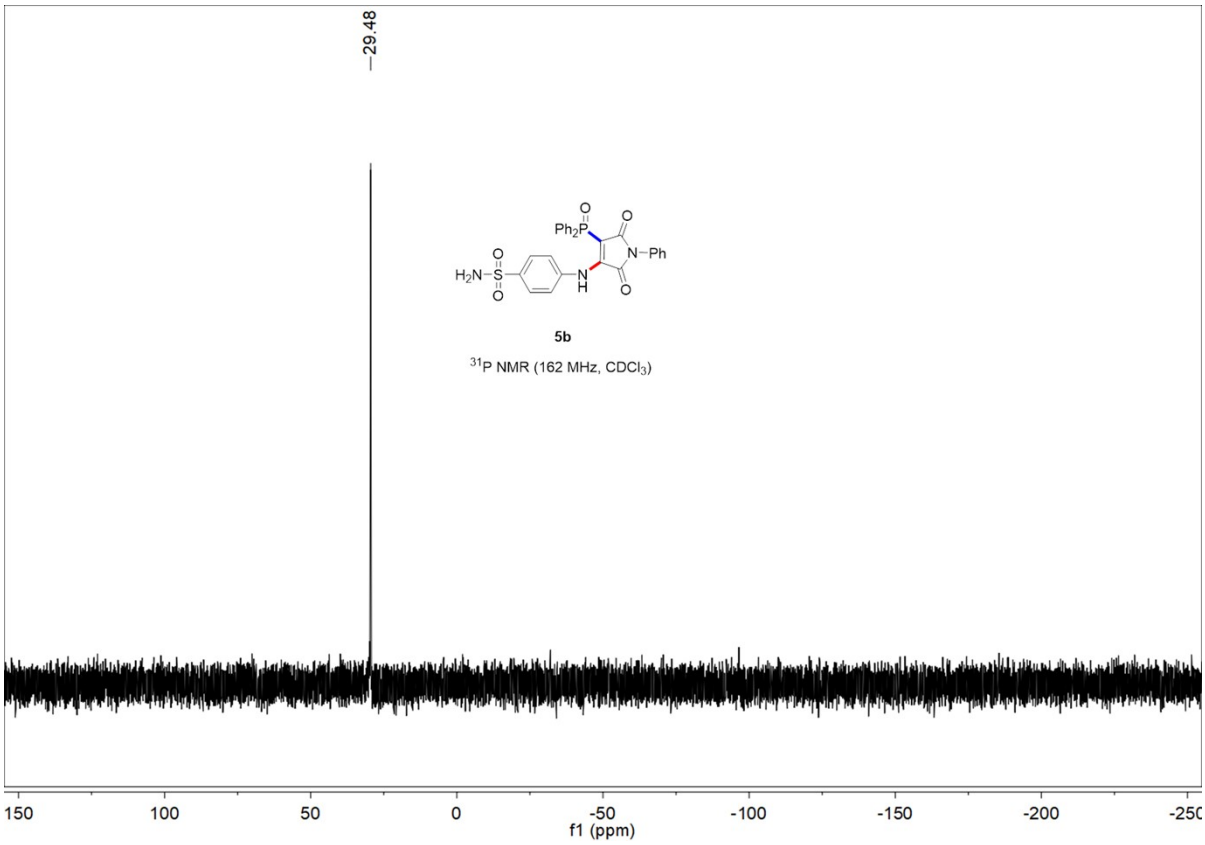


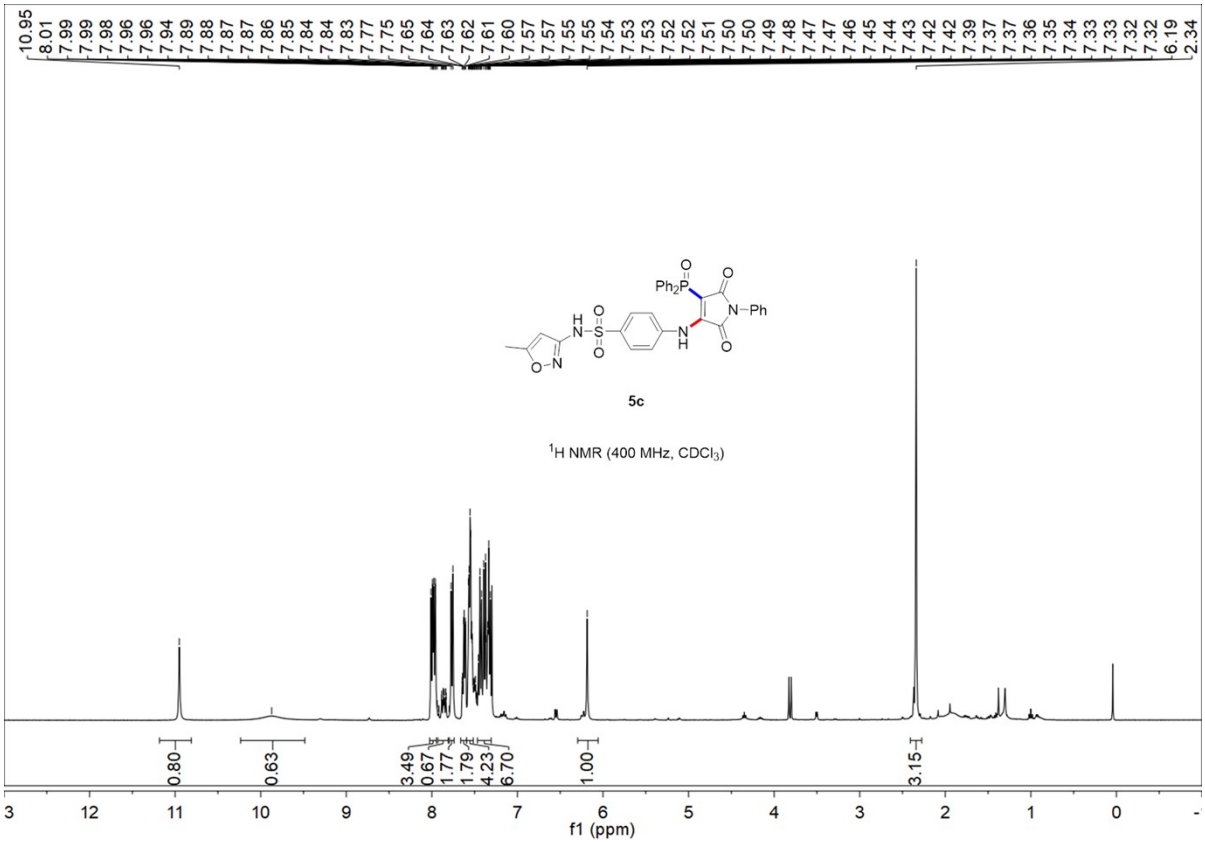
5a

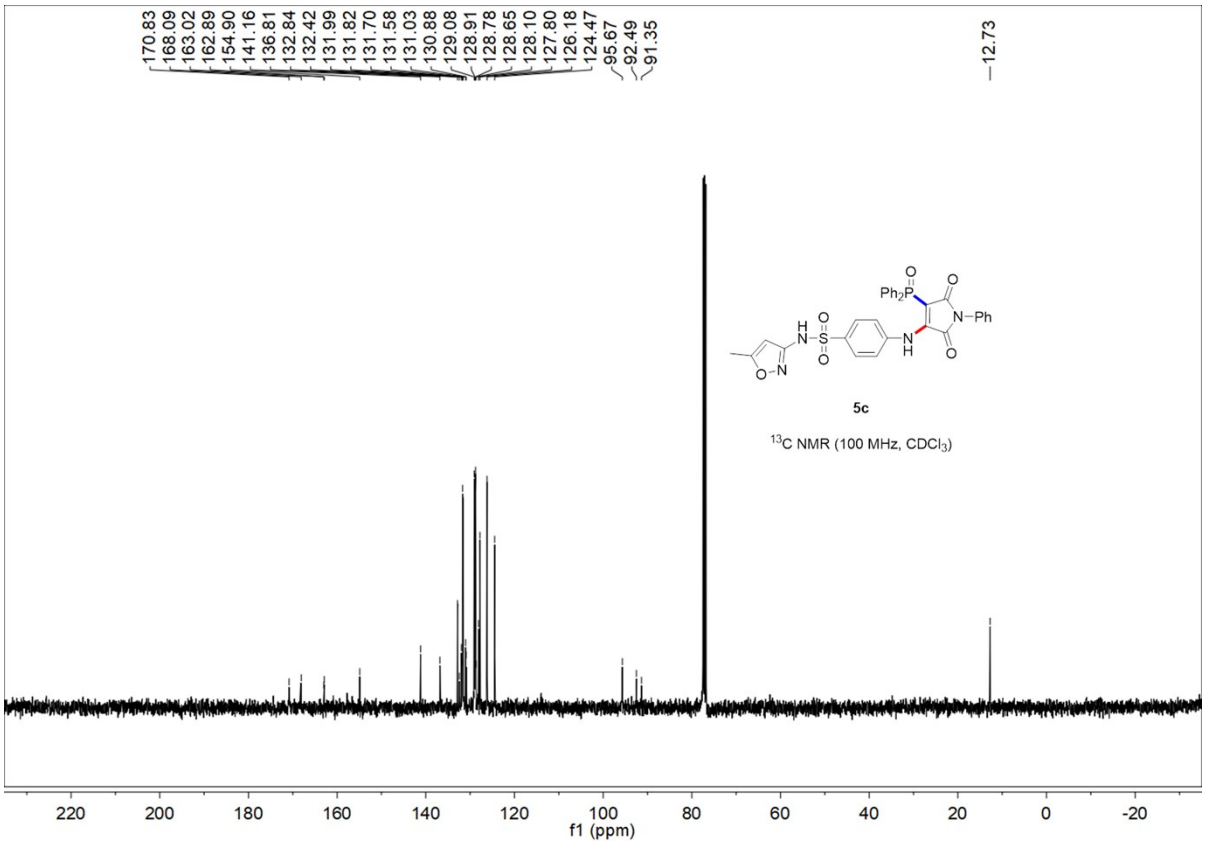
^{31}P NMR (162 MHz, $CDCl_3$)

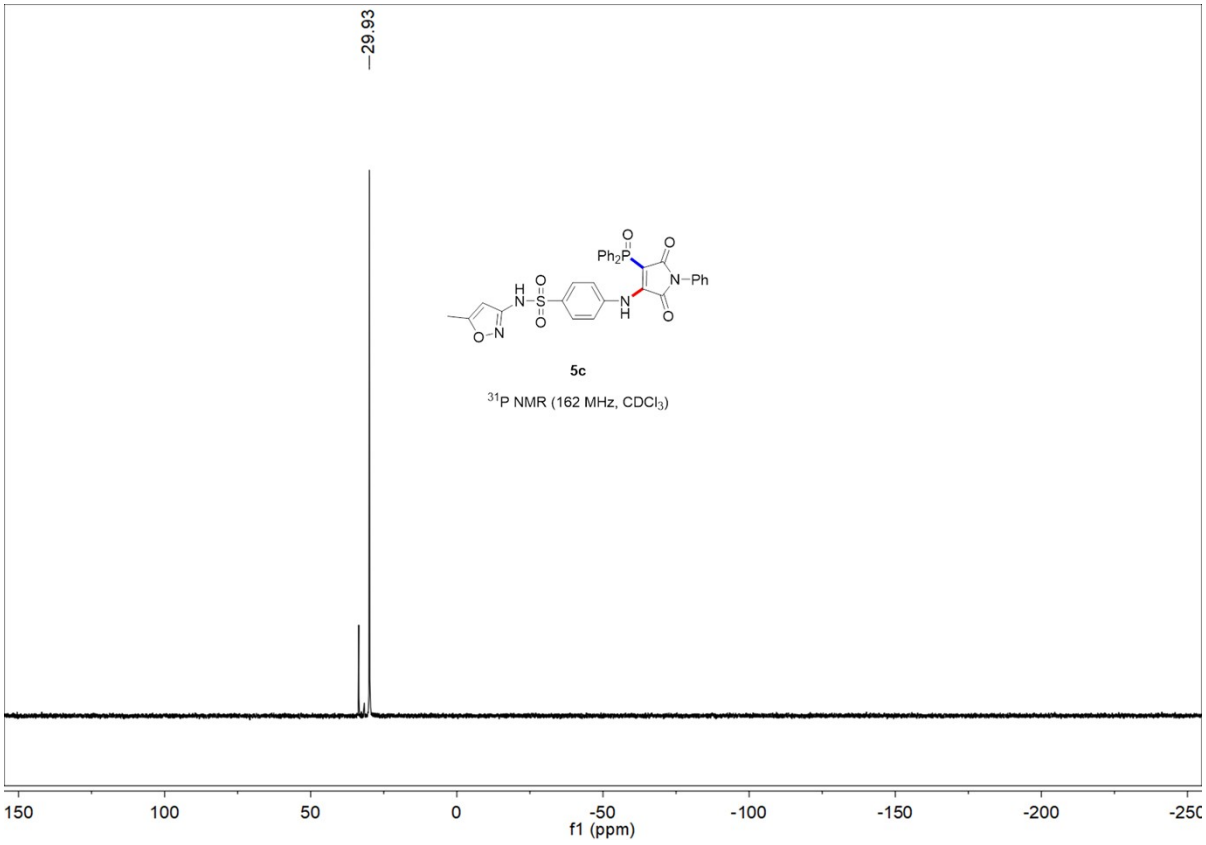

11.12
8.01
7.99
7.98
7.97
7.95
7.95
7.92
7.90
7.66
7.64
7.64
7.64
7.63
7.62
7.59
7.58
7.57
7.56
7.55
7.54
7.51
7.49
7.47
7.46
7.45
7.43
7.38
7.37
7.36
7.35
7.35
7.34
7.33
4.96
Ph2P
O
O
N–Ph
H2N–S
O=O
N
H
O
5b
1H NMR (400 MHz, CDCl3)
0.84
5.97
2.08
4.04
4.13
3.13
2.00
3
12
11
10
9
8
7
6
5
4
3
2
1
0
-
f1 (ppm)

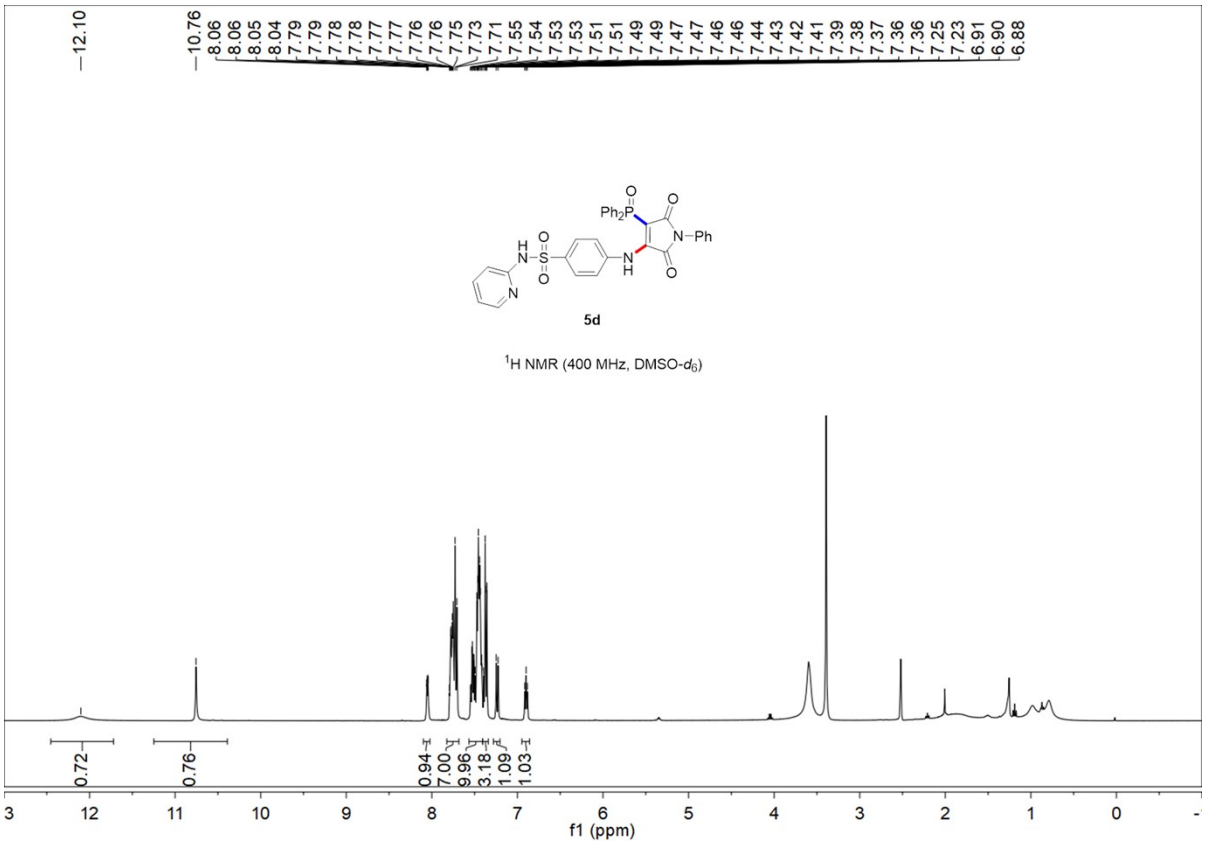

169.43
169.31
163.85
163.72
156.46
137.60
136.80
133.40
133.22
132.42
132.39
132.13
131.54
131.43
129.38
128.87
128.74
128.71
128.58
127.17
124.85
88.54
87.36
Ph2P
O
O
N–Ph
H2N–S
O=O
N
H
O
5b
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

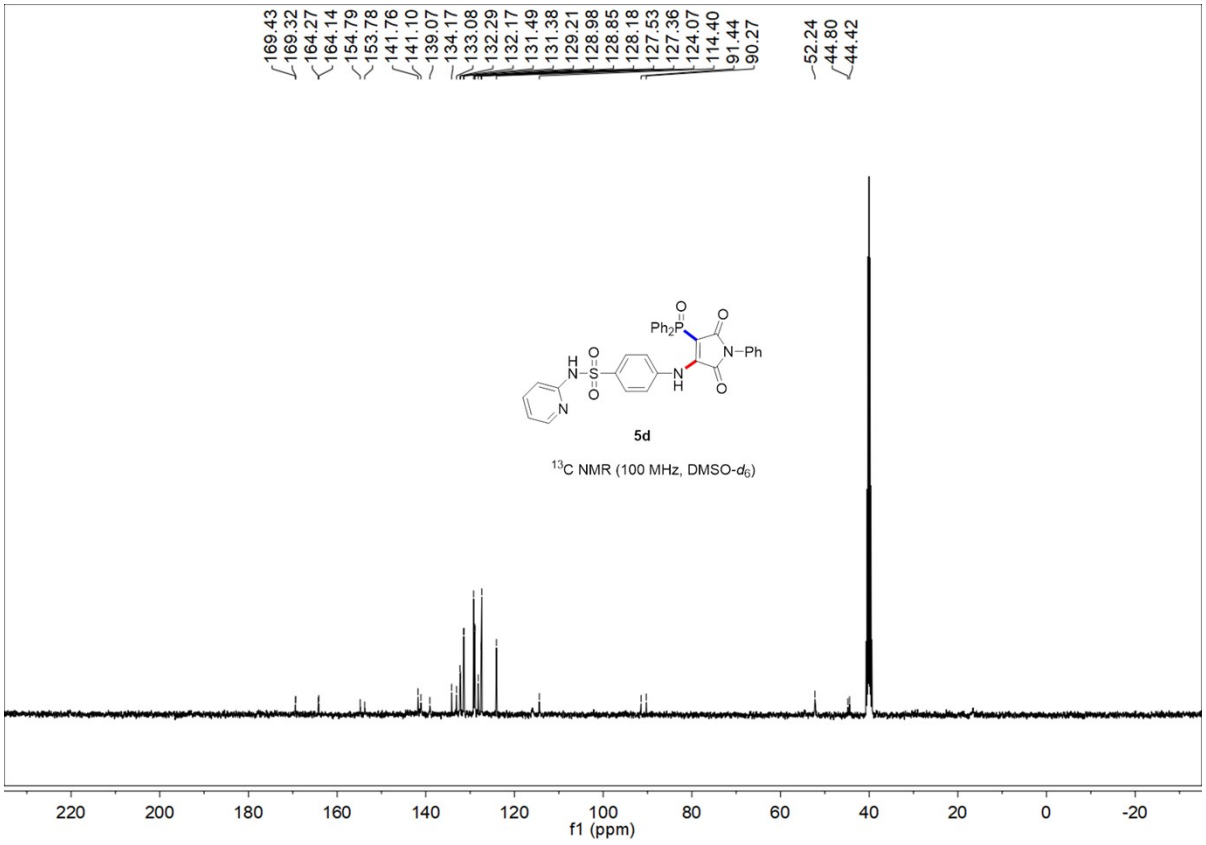

29.48
Ph2P
O
N–Ph
H2N–S
N
H
5b
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
f1 (ppm)

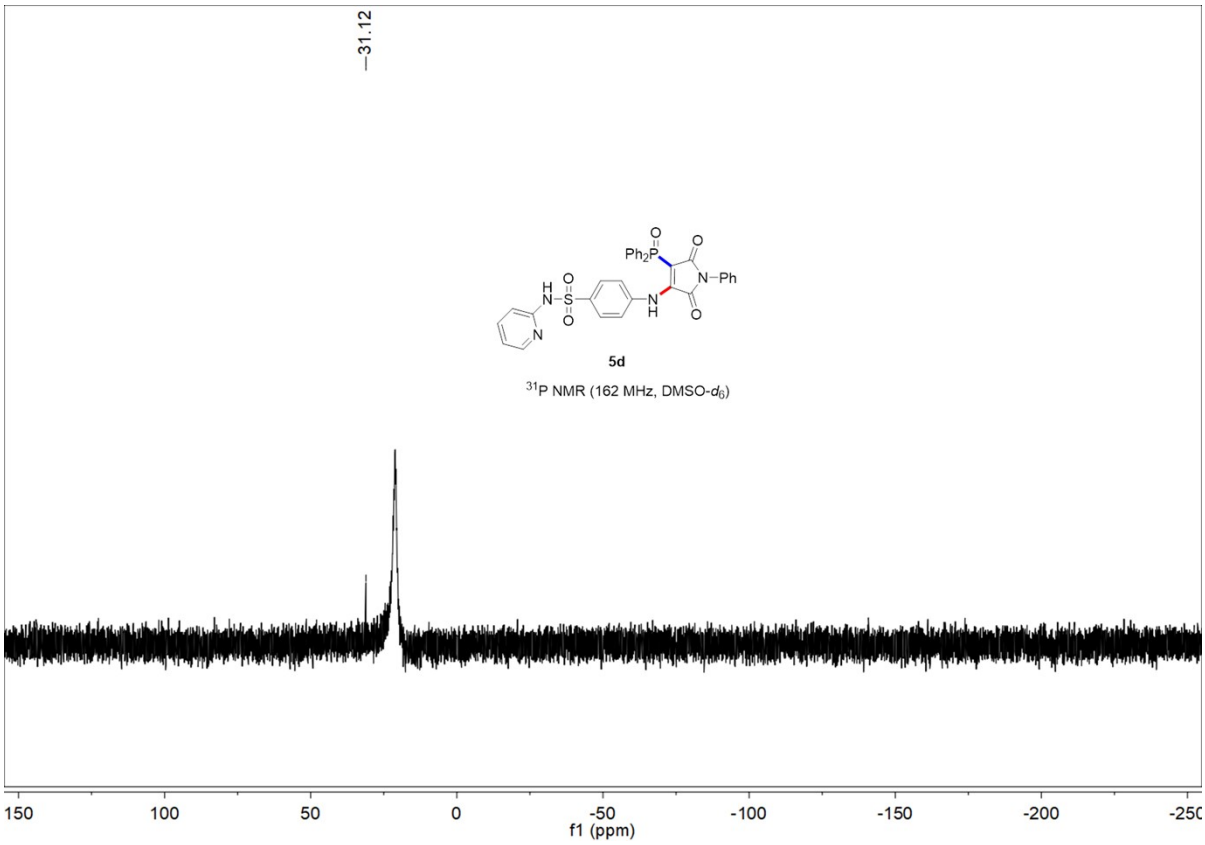

10.95
8.01
7.99
7.99
7.98
7.96
7.96
7.94
7.89
7.88
7.87
7.87
7.86
7.85
7.84
7.84
7.83
7.77
7.75
7.65
7.64
7.63
7.62
7.61
7.60
7.57
7.57
7.55
7.55
7.54
7.53
7.53
7.52
7.52
7.51
7.50
7.50
7.49
7.48
7.47
7.47
7.46
7.45
7.44
7.43
7.42
7.42
7.39
7.37
7.37
7.36
7.35
7.34
7.33
7.32
7.32
6.19
2.34
Ph2P
O
N–Ph
H
N–S
N
H
5c
1H NMR (400 MHz, CDCl3)
0.80
0.63
3.49
0.67
1.77
1.79
4.23
6.70
1.00
3.15
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

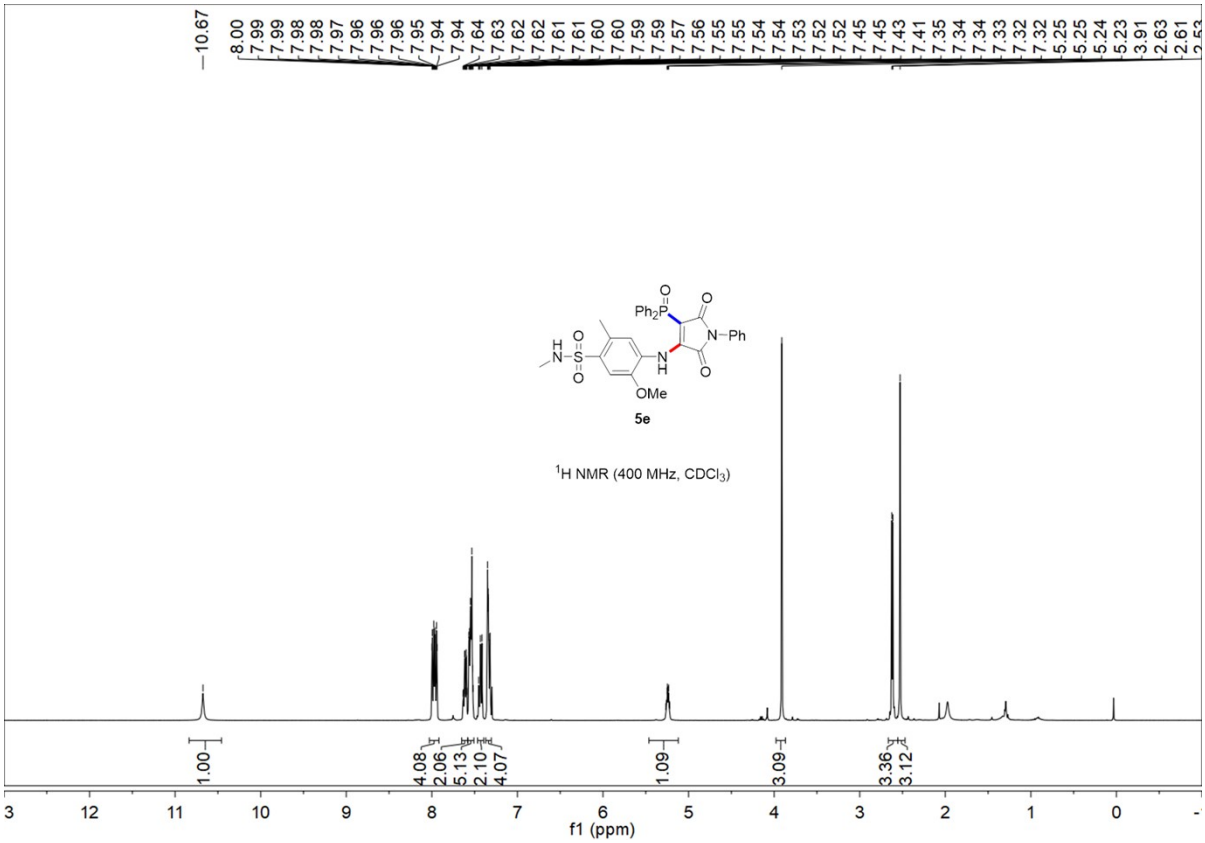

170.83
168.09
163.02
162.89
154.90
141.16
136.81
132.84
132.42
131.99
131.82
131.70
131.58
131.03
130.88
129.08
128.91
128.78
128.65
128.10
127.80
126.18
124.47
95.67
92.49
91.35
12.73
5c
13C NMR (100 MHz, CDCl3)
f1 (ppm)

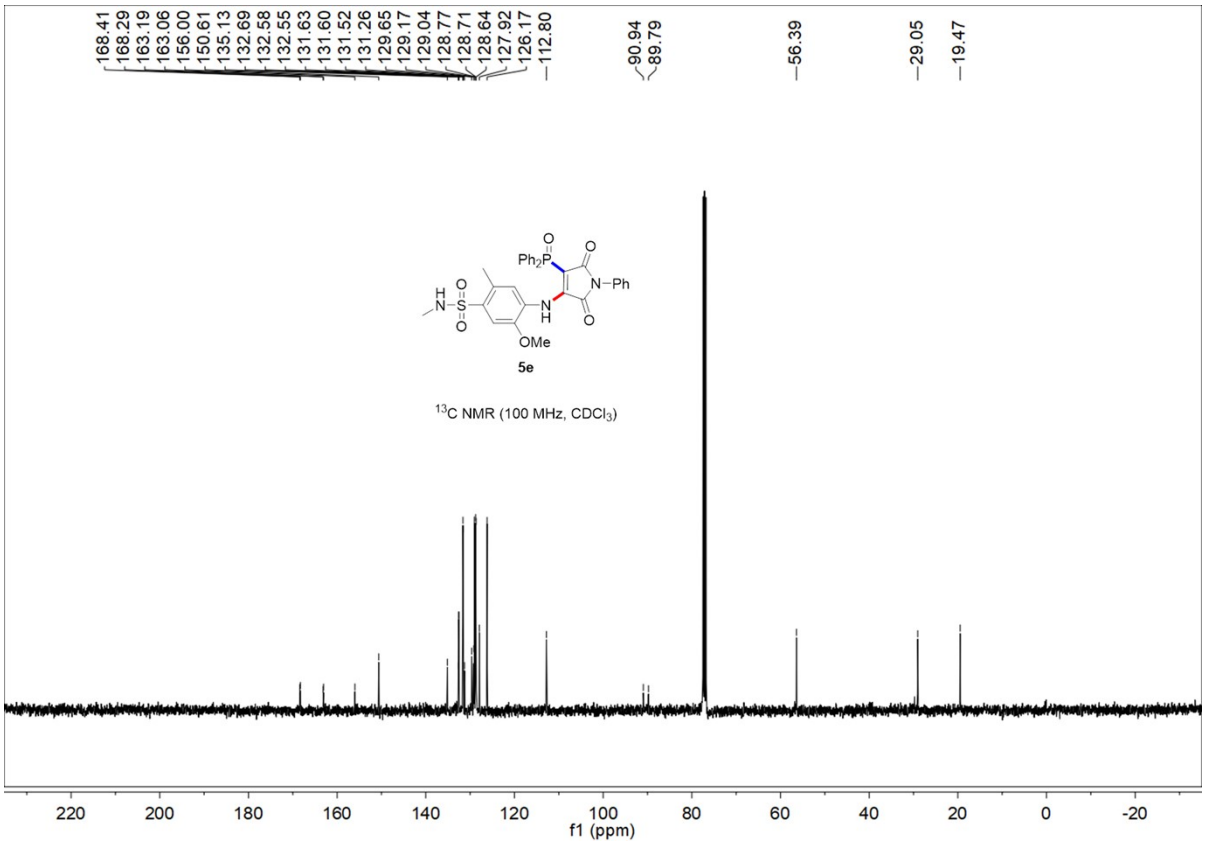

29.93
5c
31P NMR (162 MHz, CDCl3)
f1 (ppm)

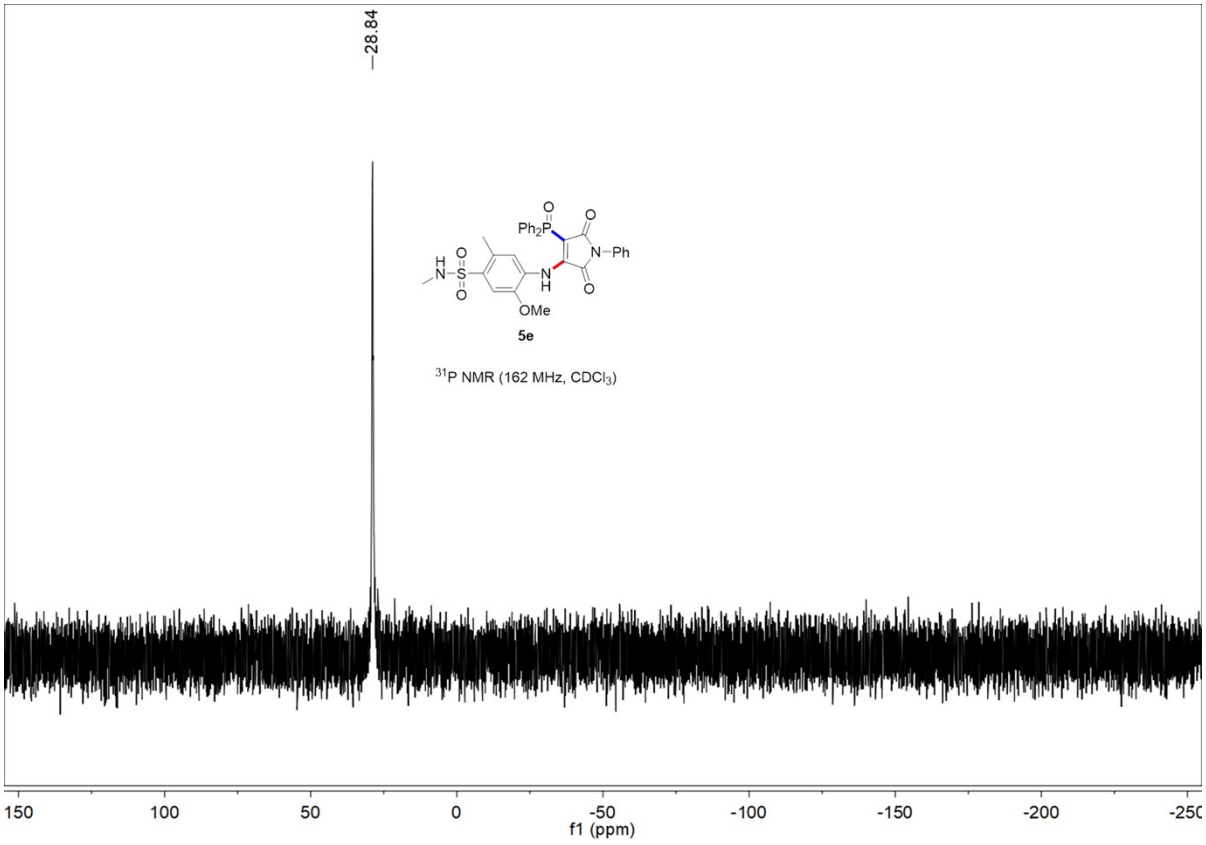

12.10
10.76
8.06
8.06
8.05
8.04
7.79
7.79
7.78
7.78
7.77
7.77
7.76
7.76
7.75
7.73
7.71
7.55
7.54
7.53
7.53
7.51
7.51
7.49
7.49
7.47
7.47
7.46
7.46
7.44
7.43
7.42
7.41
7.39
7.38
7.37
7.36
7.36
7.25
7.23
6.91
6.90
6.88
Ph2P
O
O
N–Ph
H
N–S
O
O
N
H
N
O
O
5d
1H NMR (400 MHz, DMSO-d6)
0.72
0.76
0.94
7.00
9.96
3.18
1.09
1.03
3
12
11
10
9
8
7
6
5
4
3
2
1
0
-
f1 (ppm)

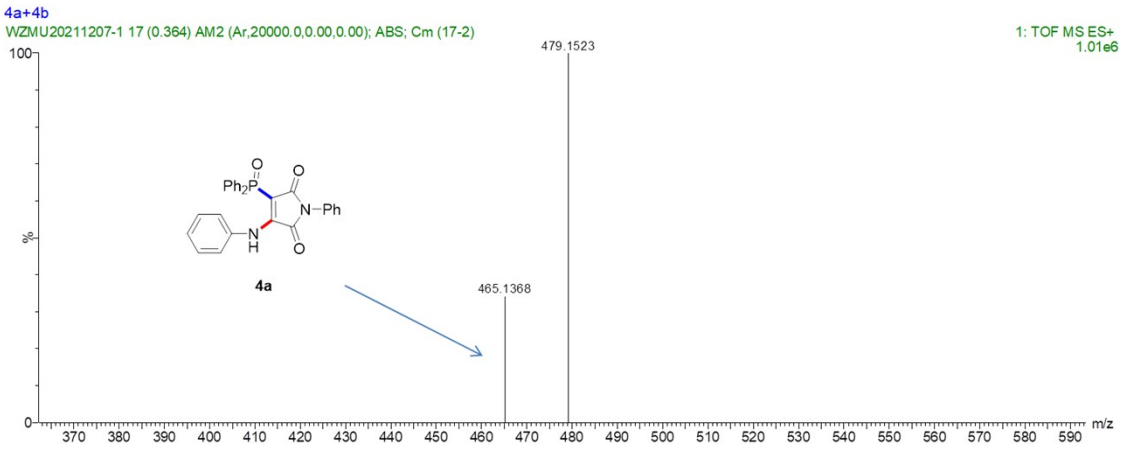

169.43
169.32
164.27
164.14
154.79
153.78
141.76
141.10
139.07
134.17
133.08
132.29
132.17
131.49
131.38
129.21
128.98
128.85
128.18
127.53
127.36
124.07
114.40
91.44
90.27
52.24
44.80
44.42
Ph2P
O
O
N–Ph
H
N–S
O
O
N
H
N
O
O
5d
13C NMR (100 MHz, DMSO-d6)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

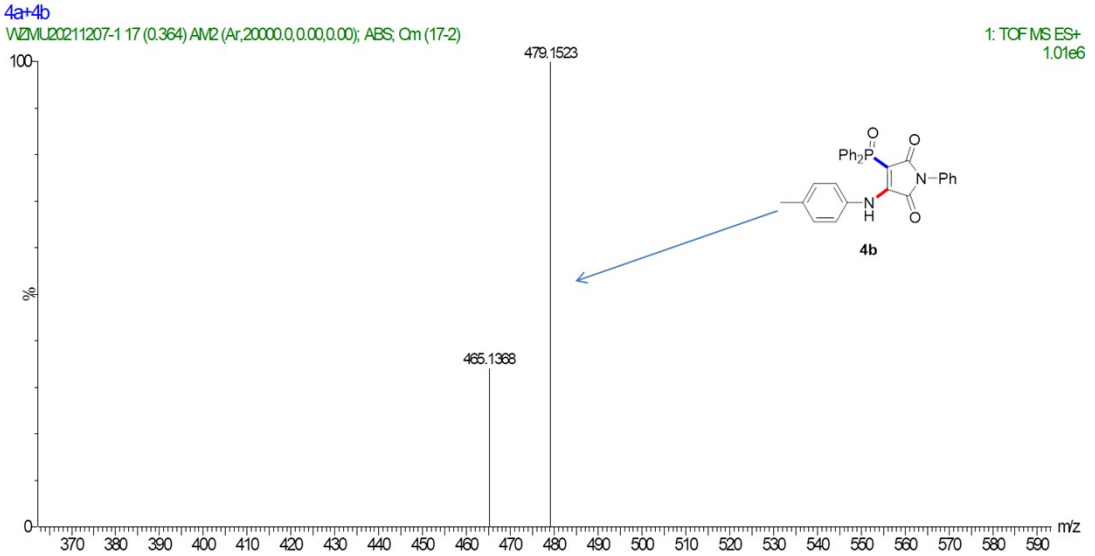

31.12
Ph2P
O
N–Ph
H
N
S
O
N
5d
31P NMR (162 MHz, DMSO-d6)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)

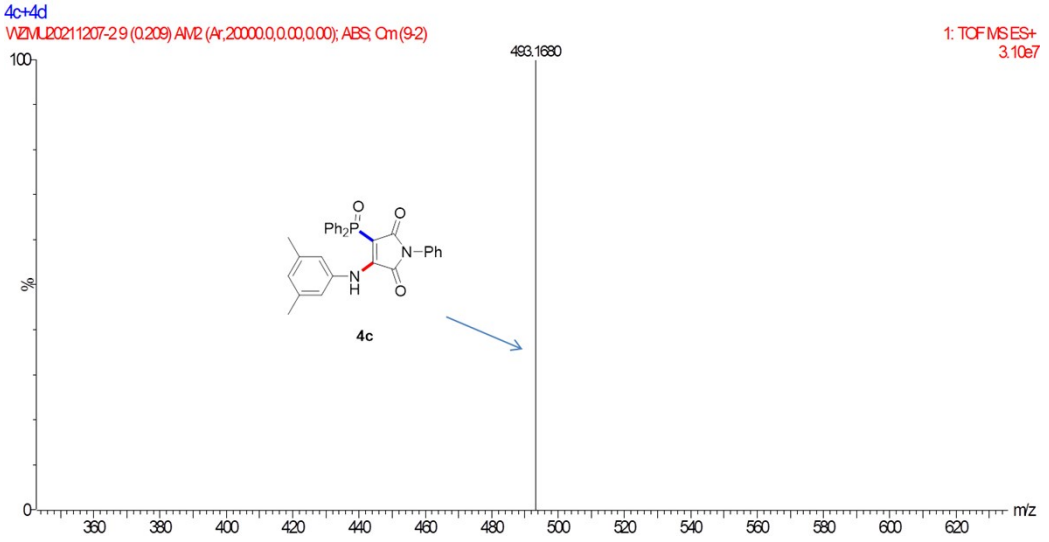

10.67
8.00
7.99
7.99
7.98
7.98
7.97
7.96
7.96
7.96
7.95
7.94
7.94
7.64
7.63
7.62
7.62
7.61
7.61
7.60
7.60
7.59
7.59
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.52
7.52
7.45
7.45
7.43
7.41
7.35
7.34
7.34
7.33
7.32
7.32
5.25
5.25
5.24
5.23
3.91
2.63
2.61
Ph2P
O
N–Ph
H
N
S
O
OMe
5e
1H NMR (400 MHz, CDCl3)
1.00
4.08
2.06
5.13
2.10
4.07
1.09
3.09
3.36
3.12
3
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


168.41
168.29
163.19
163.06
156.00
150.61
135.13
132.69
132.58
132.55
131.63
131.60
131.52
131.26
129.65
129.17
129.04
128.77
128.71
128.64
127.92
126.17
112.80
90.94
89.79
56.39
29.05
19.47
Ph2P
O
N–Ph
H
N–S
O
OMe
5e
13C NMR (100 MHz, CDCl3)
220
200
180
160
140
120
100
80
60
40
20
0
-20
f1 (ppm)

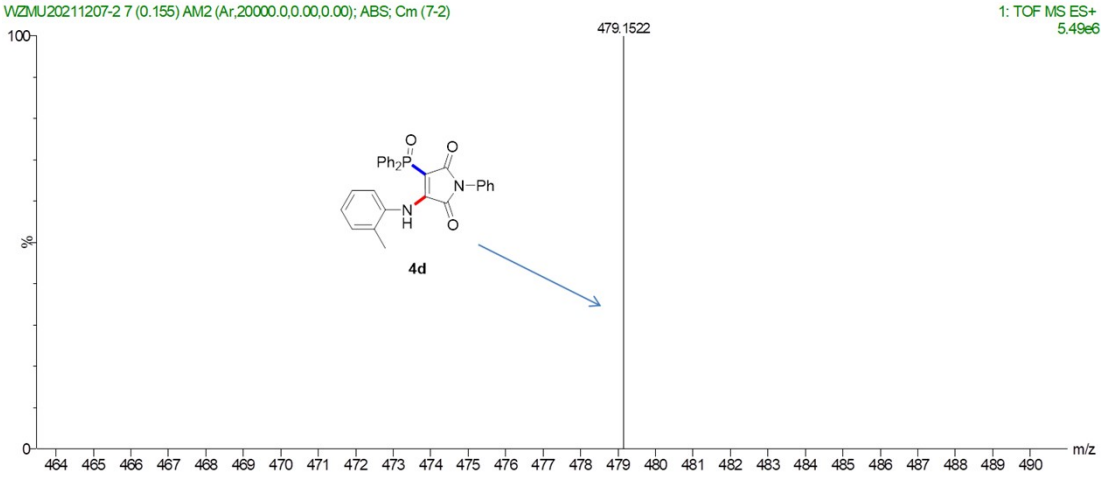

28.84
Ph2P
O
N–Ph
H
N–S
O
OMe
5e
31P NMR (162 MHz, CDCl3)
150
100
50
0
-50
-100
-150
-200
-250
f1 (ppm)


4a+4b
WZMU20211207-1 17 (0.364) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (17-2)
1: TOF MS ES+
1.01e6
479.1523
465.1368
4a
m/z

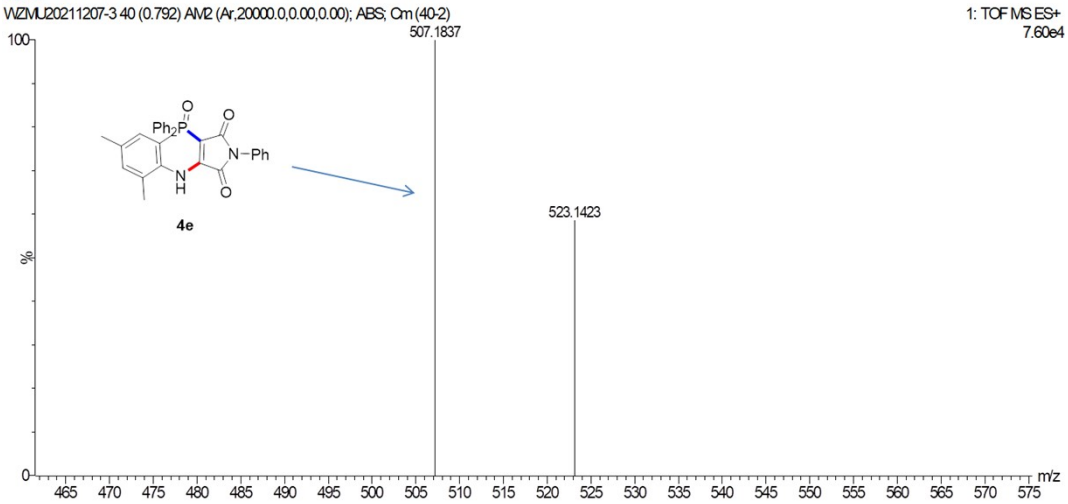

4a+4b
WZMU20211207-1 17 (0.364) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (17-2)
1: TOF MS ES+
1.01e6
479.1523
465.1368
4b
m/z


4c+4d
WZMU20211207-2 9 (0.209) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (9-2)
1: TOF MS ES+
3.10e7
493.1680
4c
m/z

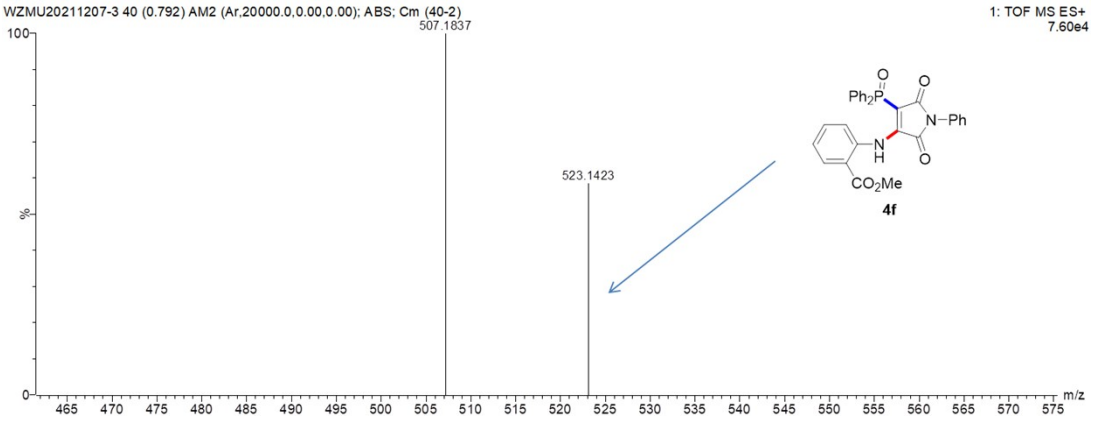
4c+4d

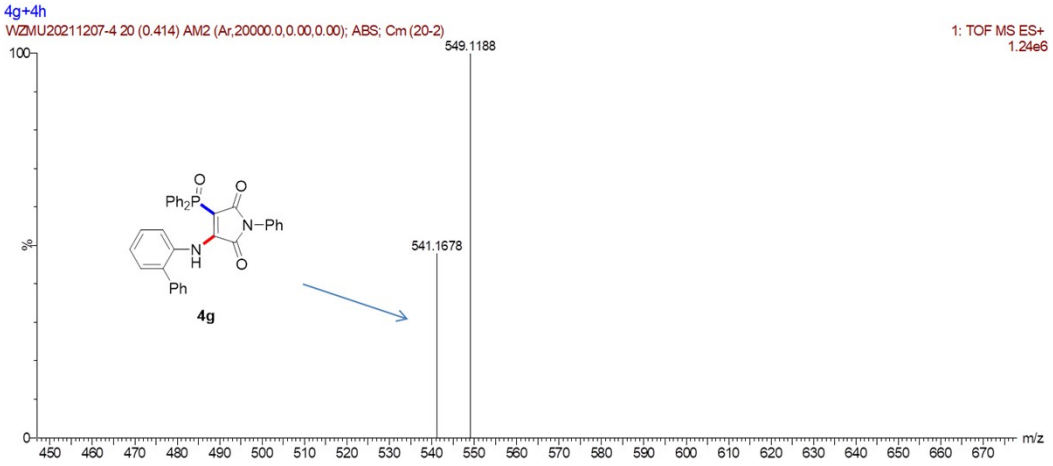


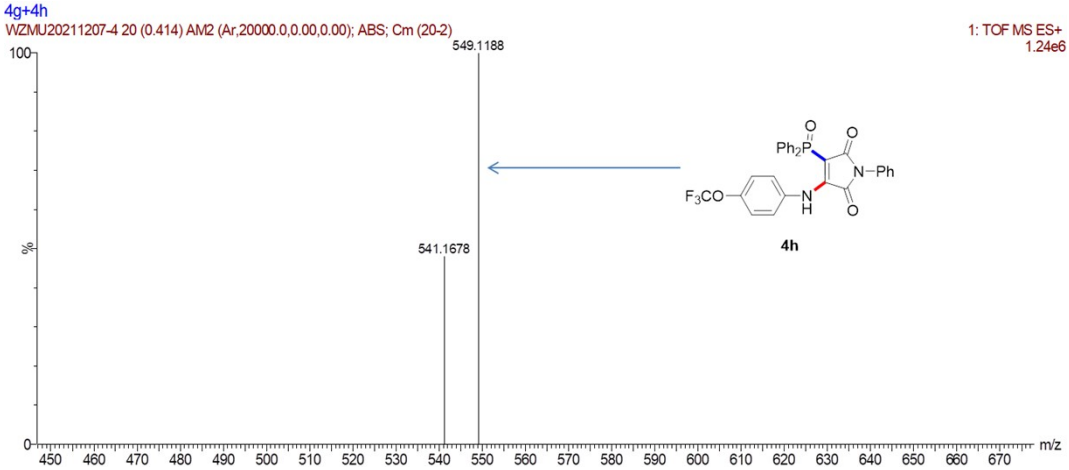
4e+4f

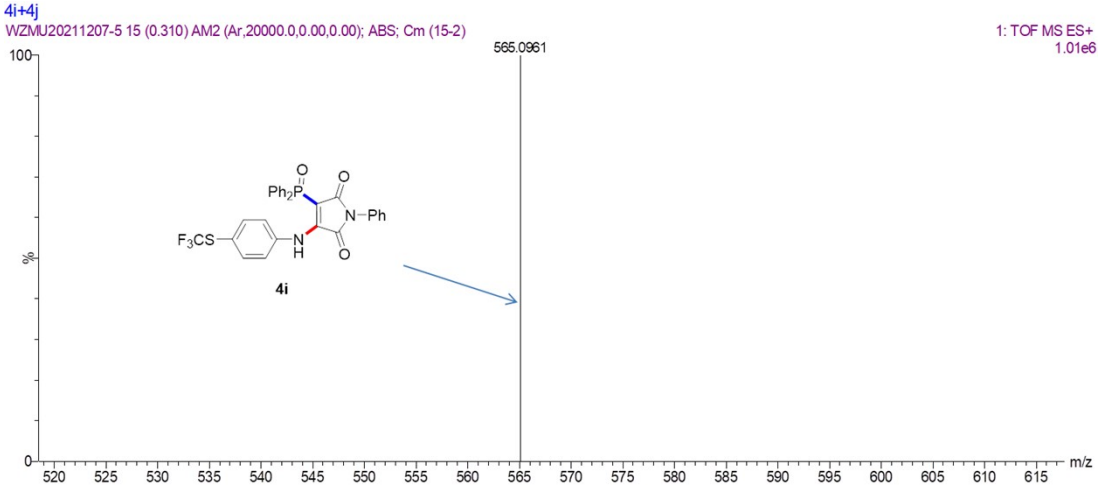


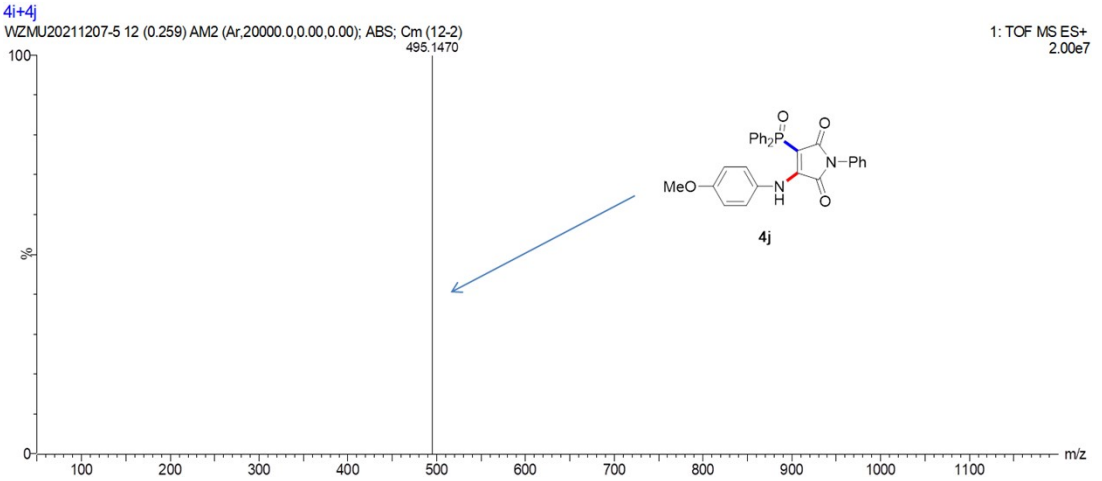
4e+4f

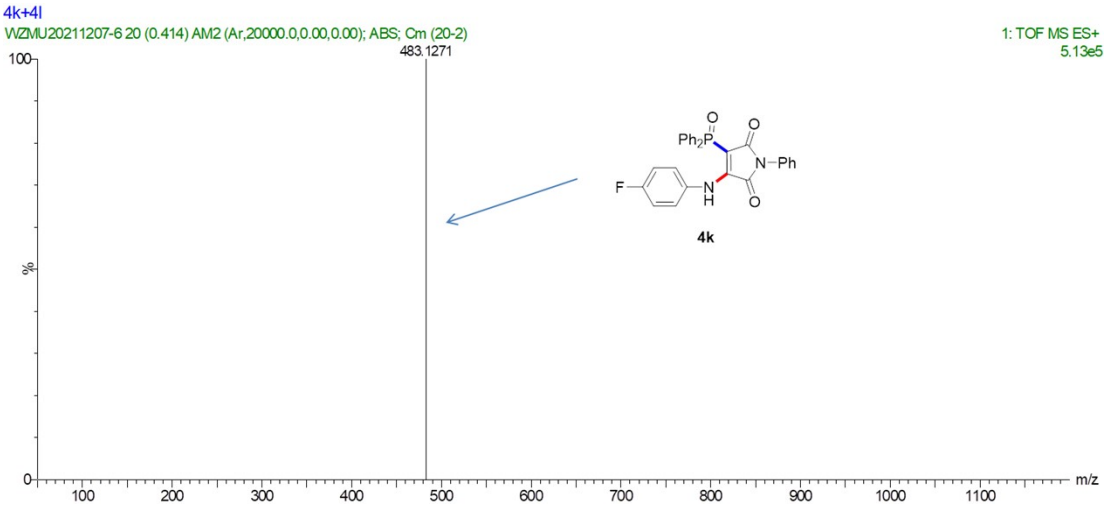


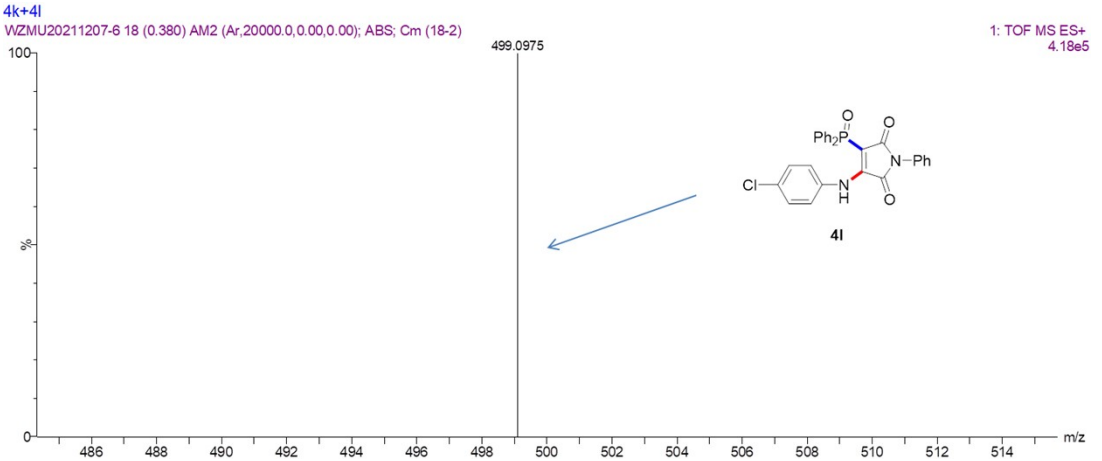

4g+4h
WZMU20211207-4 20 (0.414) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (20-2)
1: TOF MS ES+
1.24e6
549.1188
541.1678
4g
m/z

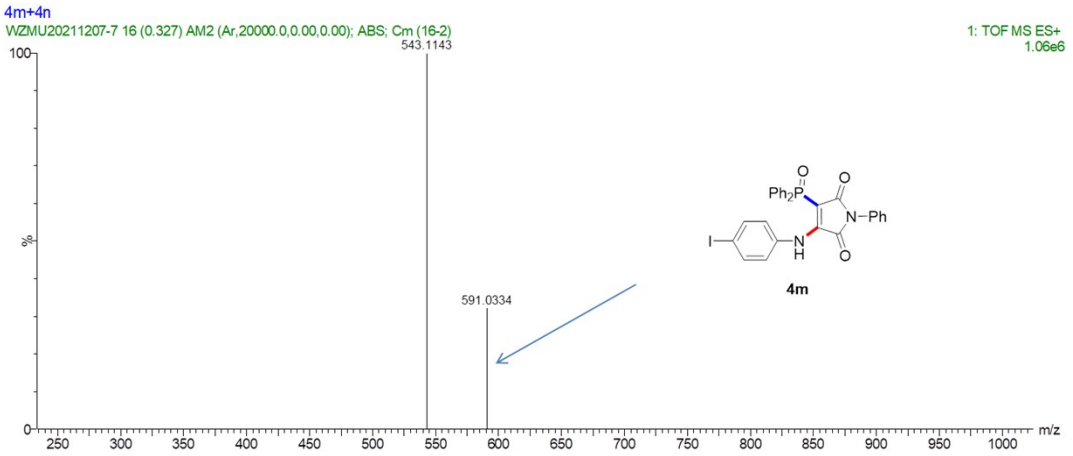

4g+4h
WZMU20211207-4 20 (0.414) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (20-2)
1: TOF MS ES+
1.24e6
549.1188
541.1678
4h
m/z

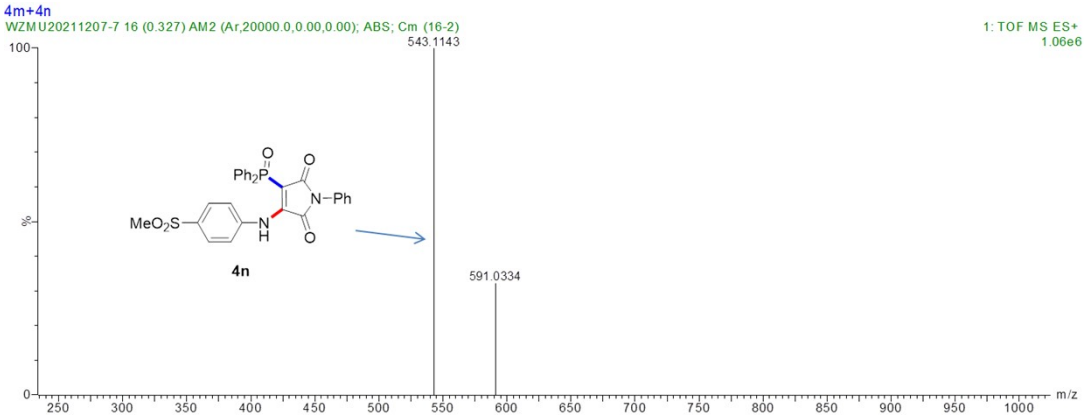

4i+4j
WZMU20211207-5 15 (0.310) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (15-2)
1: TOF MS ES+
1.01e6
565.0961
4i
m/z

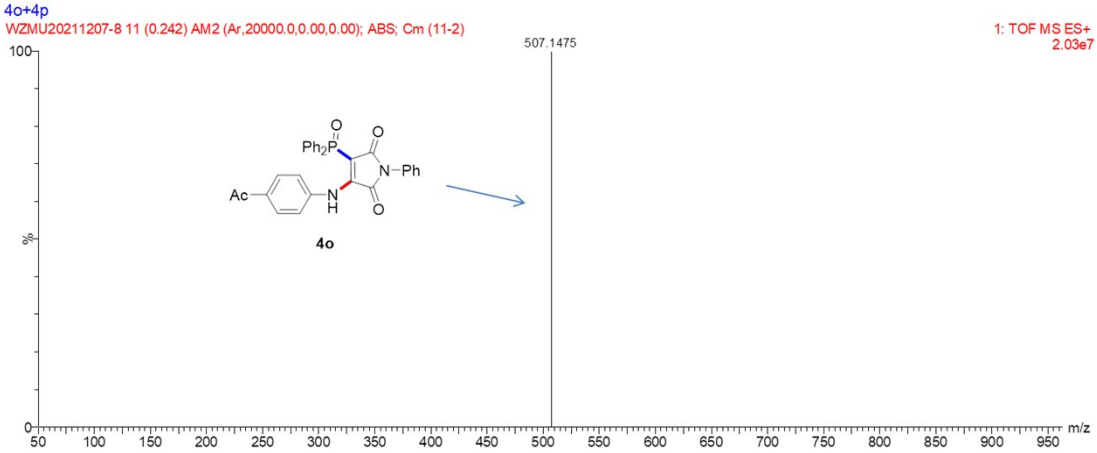

4i+4j
WZMU20211207-5 12 (0.259) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (12-2)
1: TOF MS ES+
2.00e7
495.1470
100
%
0
100
200
300
400
500
600
700
800
900
1000
1100
m/z
Ph2P
O
O
N–Ph
MeO
N
H
O
4j


4k+4l
WZMU20211207-6 20 (0.414) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (20-2)
1: TOF MS ES+
5.13e5
483.1271
100
%
0
100
200
300
400
500
600
700
800
900
1000
1100
m/z
Ph2P
O
O
N–Ph
F
N
H
O
4k

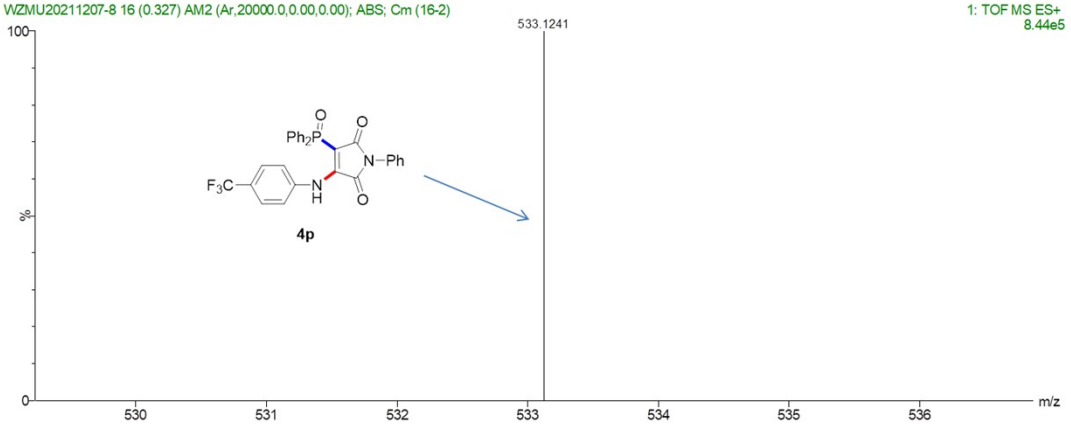

4k+4l
WZMU20211207-6 18 (0.380) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (18-2)
1: TOF MS ES+
4.18e5
499.0975
100
%
0
486
488
490
492
494
496
498
500
502
504
506
508
510
512
514
m/z
Ph2P
O
O
N–Ph
Cl
N
H
O
4l


4m+4n
WZMU20211207-7 16 (0.327) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (16-2)
1: TOF MS ES+
1.06e6
543.1143
591.0334
4m
m/z

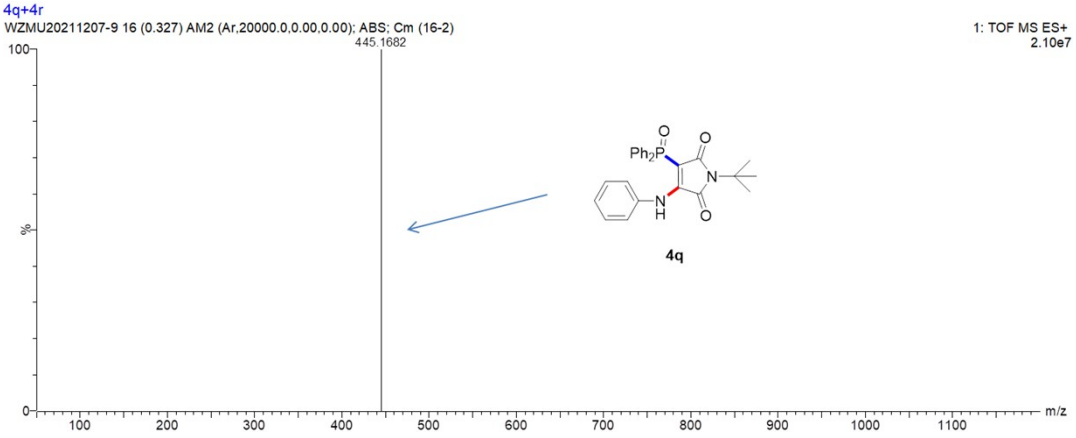

4m+4n
WZMU20211207-7 16 (0.327) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (16-2)
1: TOF MS ES+
1.06e6
543.1143
591.0334
4n
m/z


4o+4p
WZMU20211207-8 11 (0.242) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (11-2)
1: TOF MS ES+
2.03e7
507.1475
4o
m/z

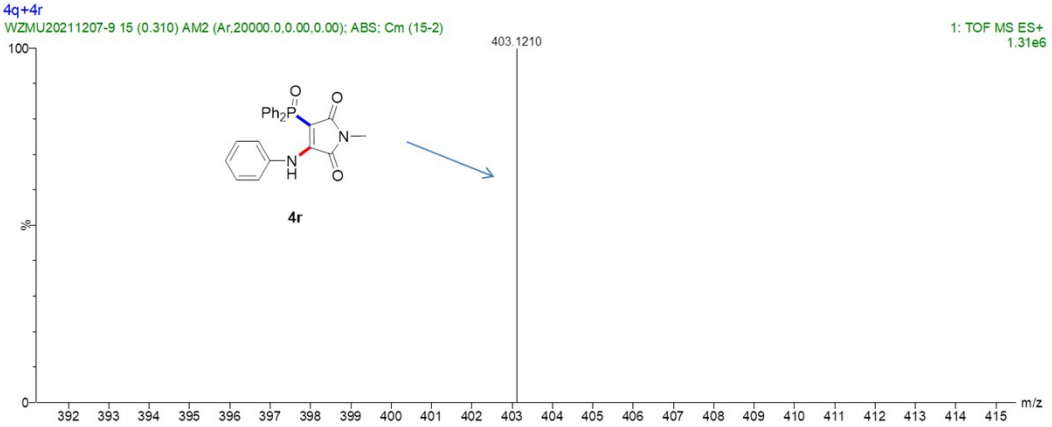
4o+4p

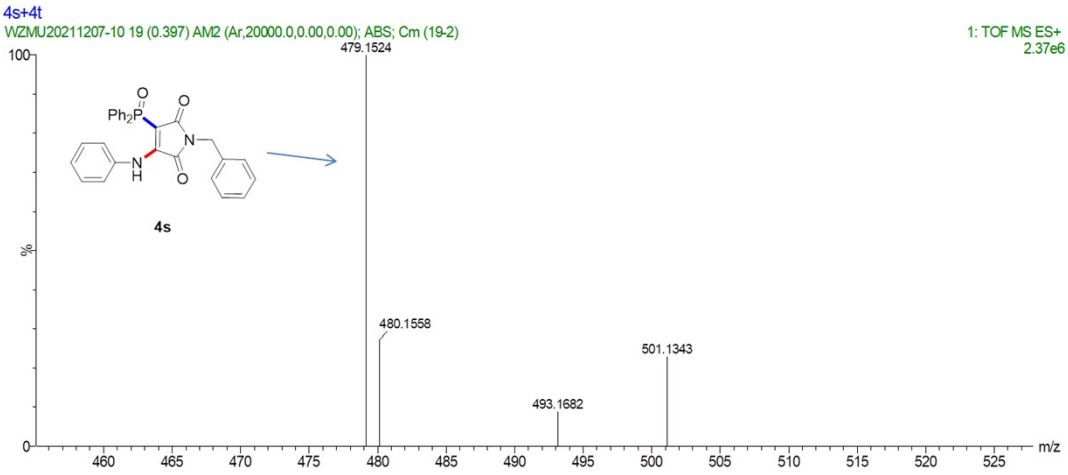


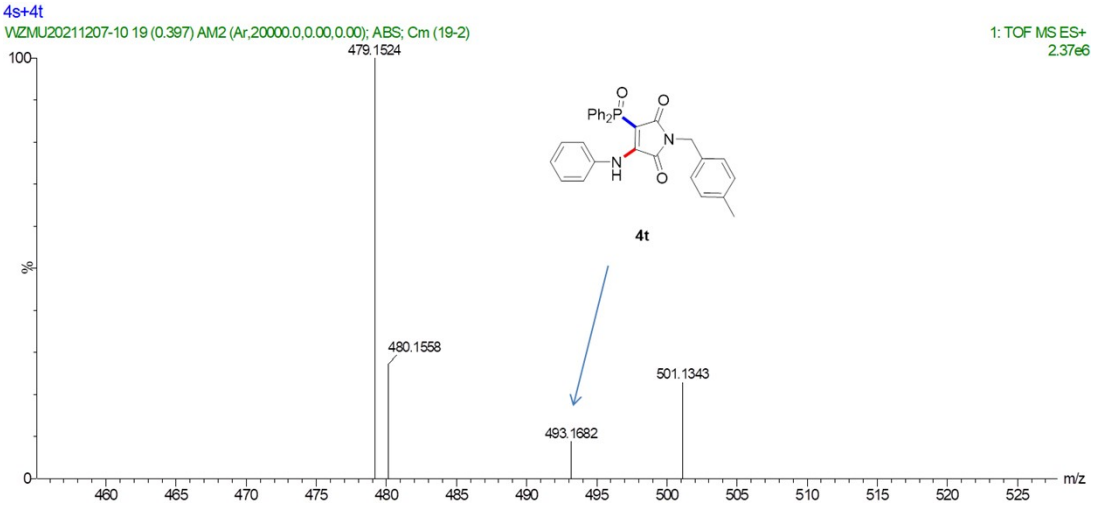
4q+4r

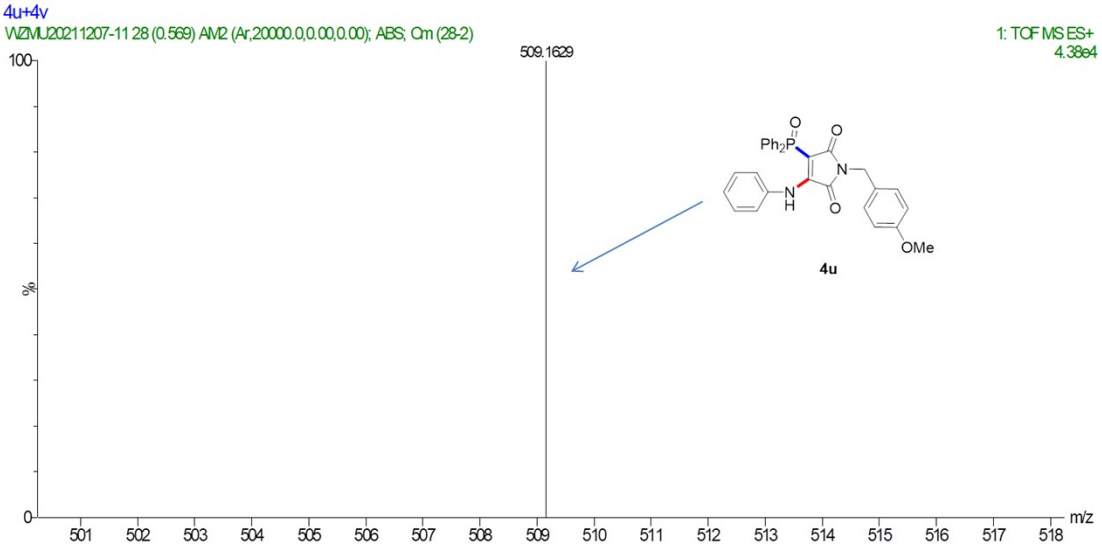


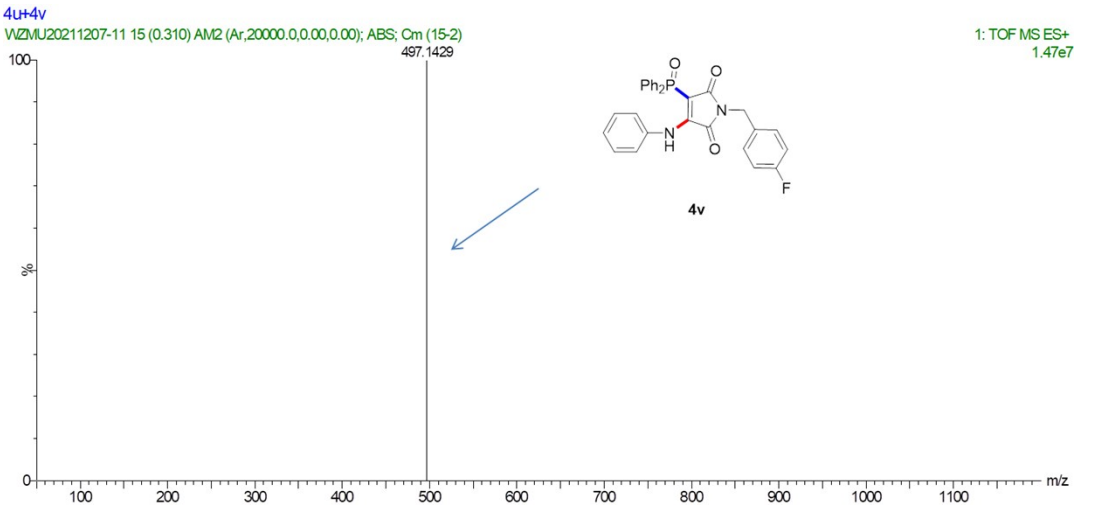
4q+4r

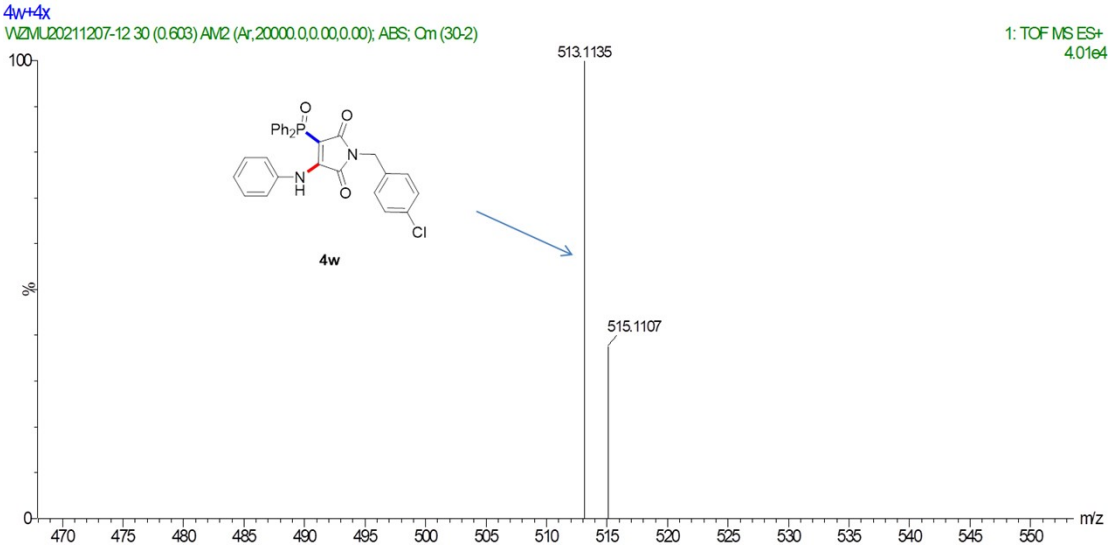


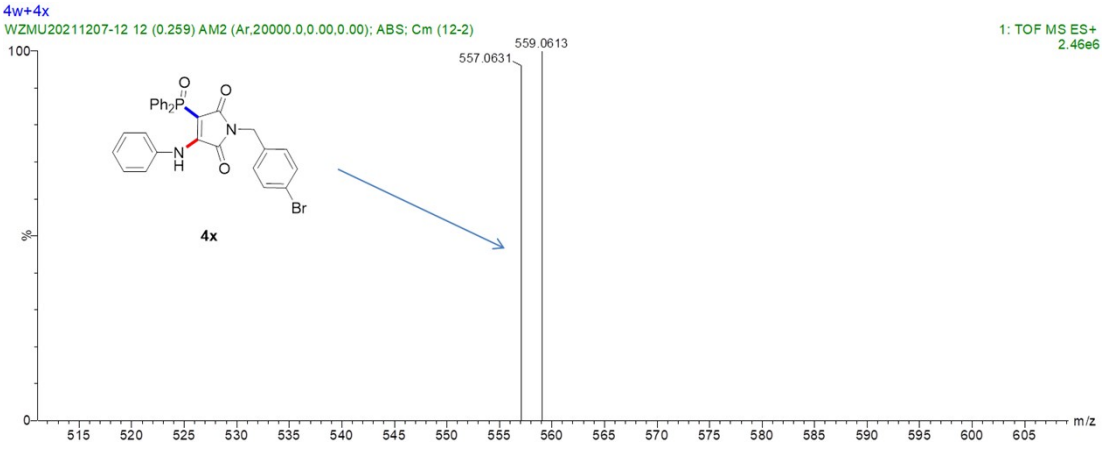

4s+4t
WZMU20211207-10 19 (0.397) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (19-2)
1: TOF MS ES+
2.37e6
479.1524
480.1558
501.1343
493.1682
4s
%
m/z


4s+4t
WZMU20211207-10 19 (0.397) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (19-2)
1: TOF MS ES+
2.37e6
479.1524
480.1558
501.1343
493.1682
4t
%
m/z


4u+4v
WZMU20211207-11 28 (0.569) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (28-2)
1: TOF MS ES+
4.38e4
509.1629
4u
%
m/z


4u+4v
WZMU20211207-11 15 (0.310) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (15-2)
1: TOF MS ES+
1.47e7
497.1429
100
%
0
100
200
300
400
500
600
700
800
900
1000
1100
m/z
4v

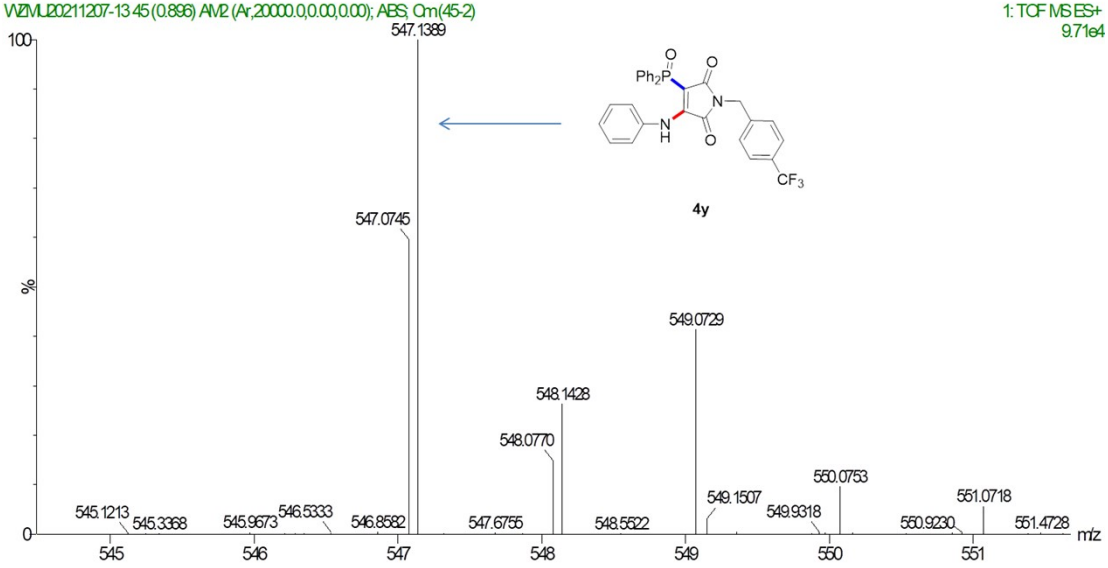

4w+4x
WZMU20211207-12 30 (0.603) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (30-2)
1: TOF MS ES+
4.01e4
513.1135
515.1107
100
%
0
470
475
480
485
490
495
500
505
510
515
520
525
530
535
540
545
550
m/z
4w


4w+4x
WZMU20211207-12 12 (0.259) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (12-2)
1: TOF MS ES+
2.46e6
559.0613
557.0631
100
%
0
515
520
525
530
535
540
545
550
555
560
565
570
575
580
585
590
595
600
605
m/z
4x

4y+4z

WZMU20211207-13 45 (0.896) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm(45-2)

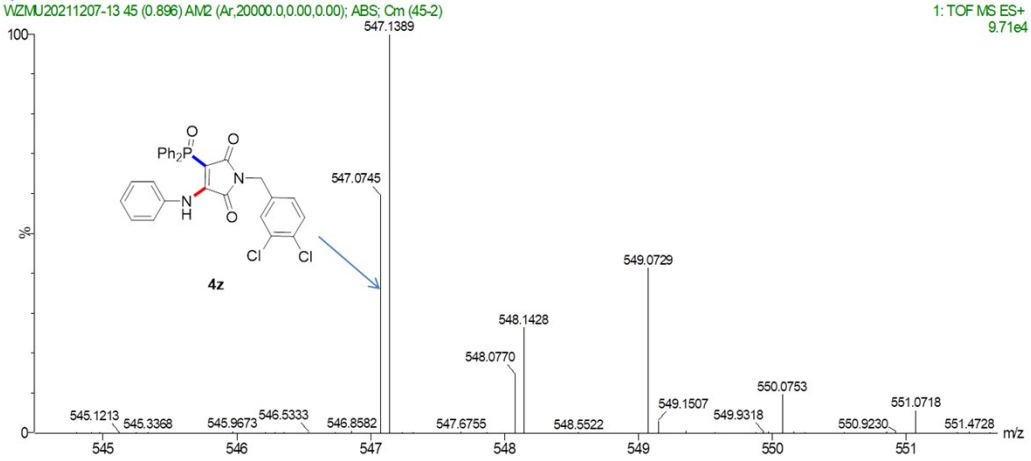
1: TOF MS ES+
9.71e4



4y+4z

WZMU20211207-13 45 (0.896) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (45-2)

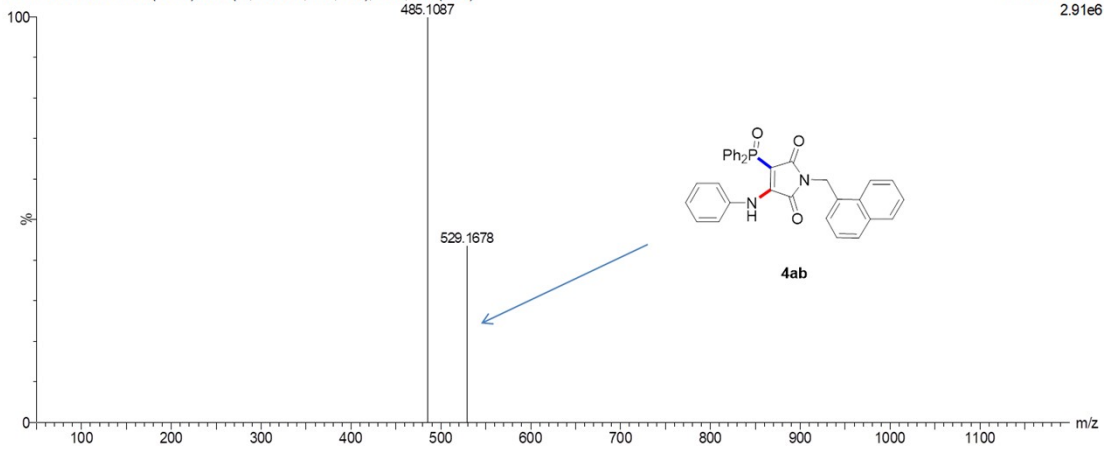
1: TOF MS ES+
9.71e4



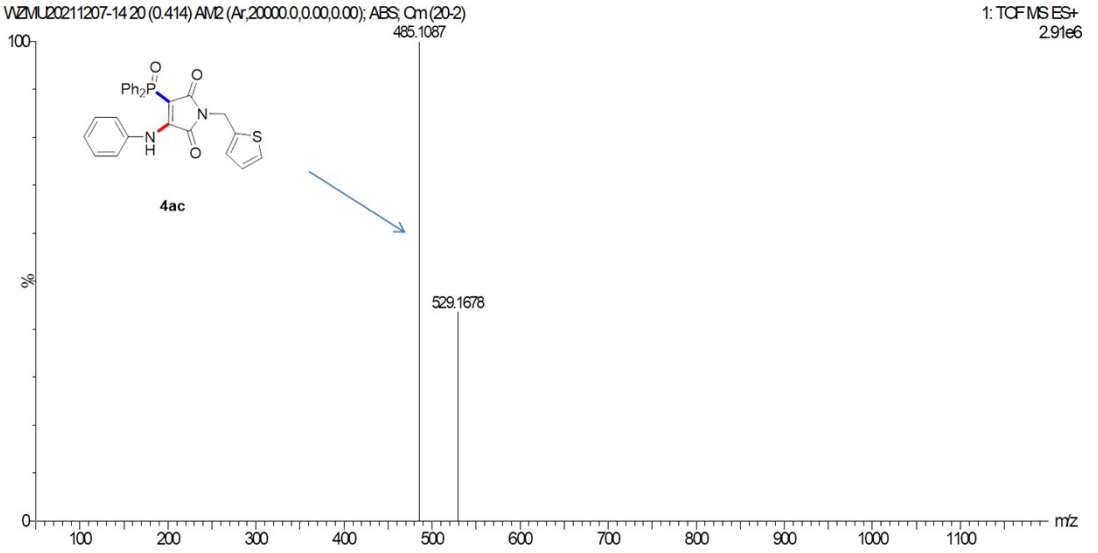
4ab+4ac

WZMU20211207-14 20 (0.414) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (20-2)

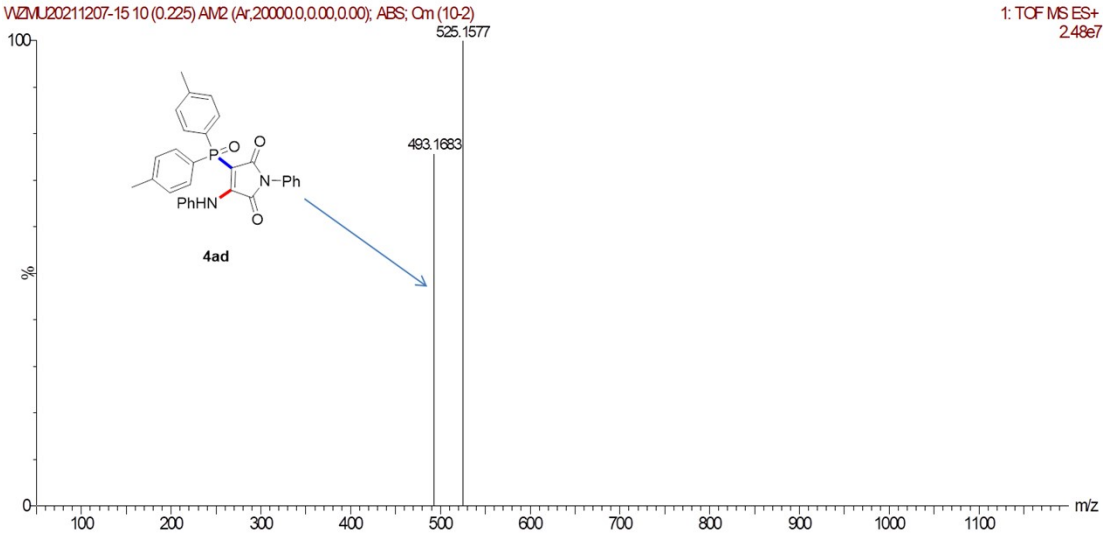
1: TOF MS ES+
2.91e6



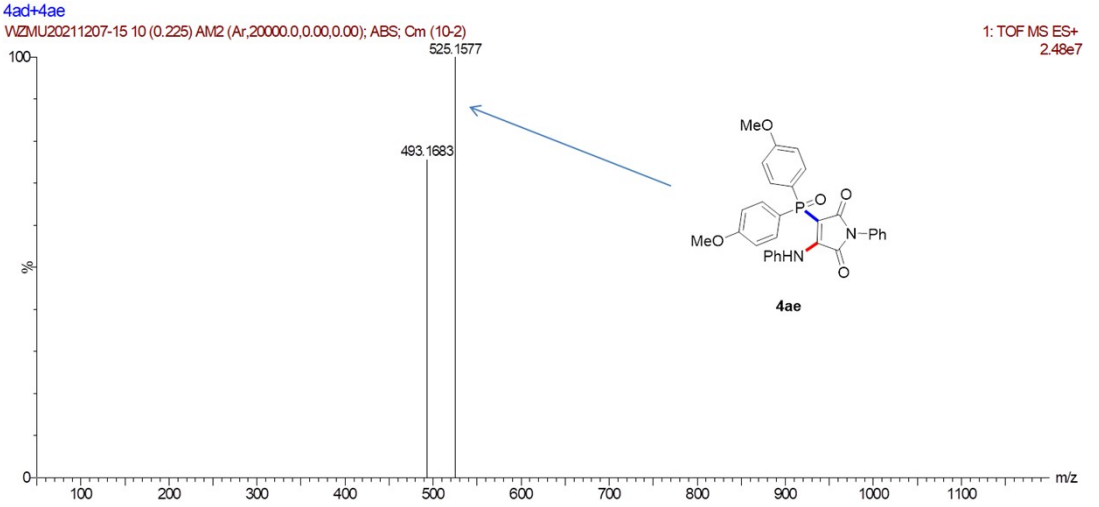
4ab+4ac

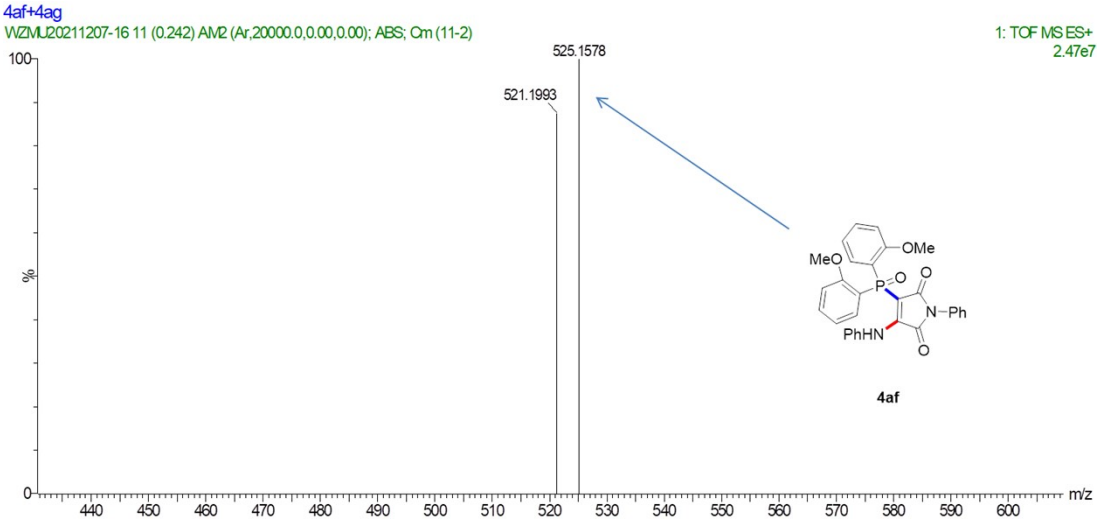


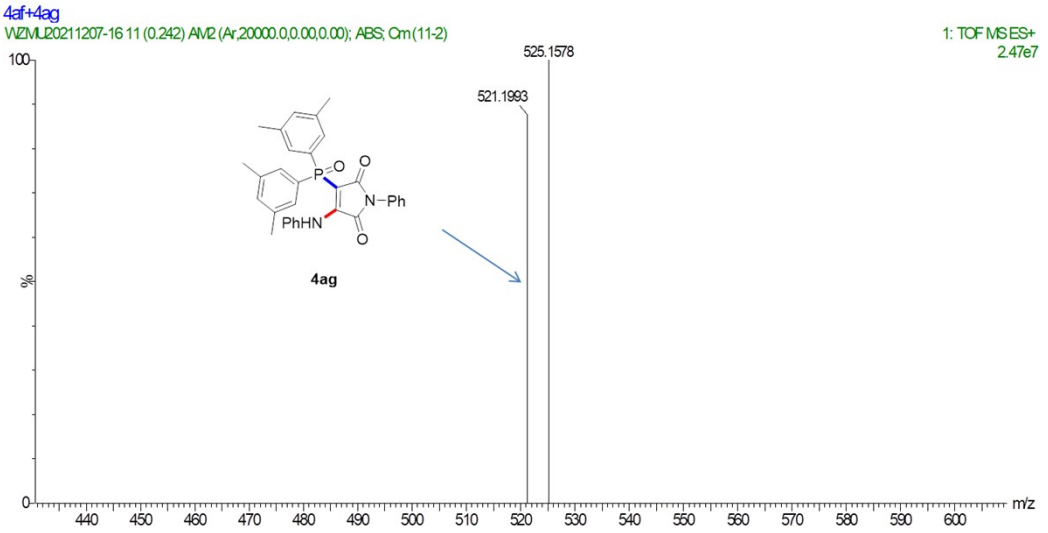
4ad+4ae

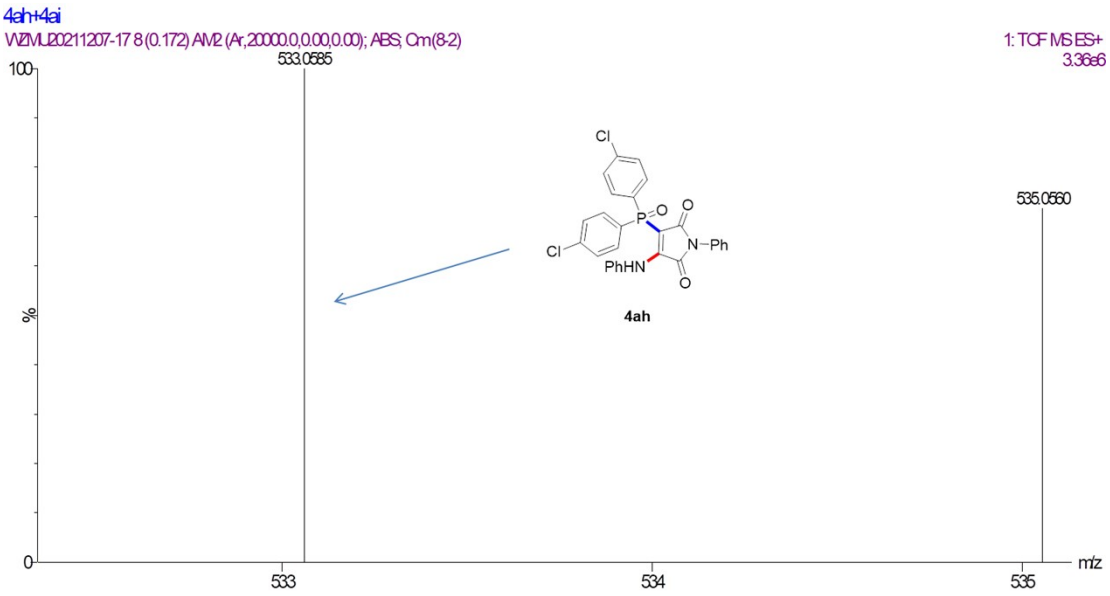


4ad+4ae

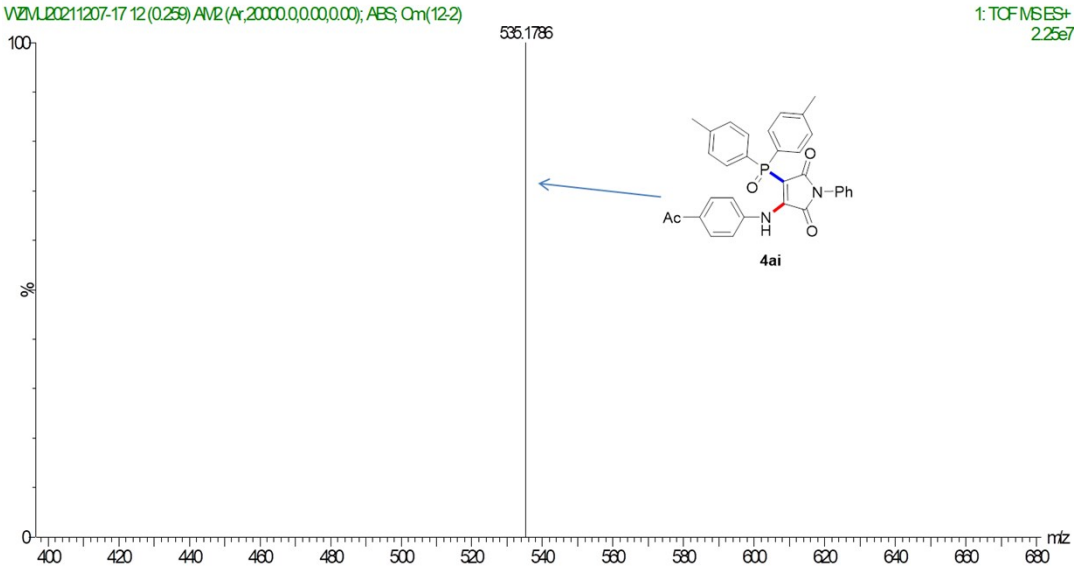



4af+4ag
WZMU20211207-16 11 (0.242) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (11-2)
1: TOF MS ES+
2.47e7
525.1578
521.1993
MeO
OMe
P
O
N–Ph
PhHN
4af
%
m/z

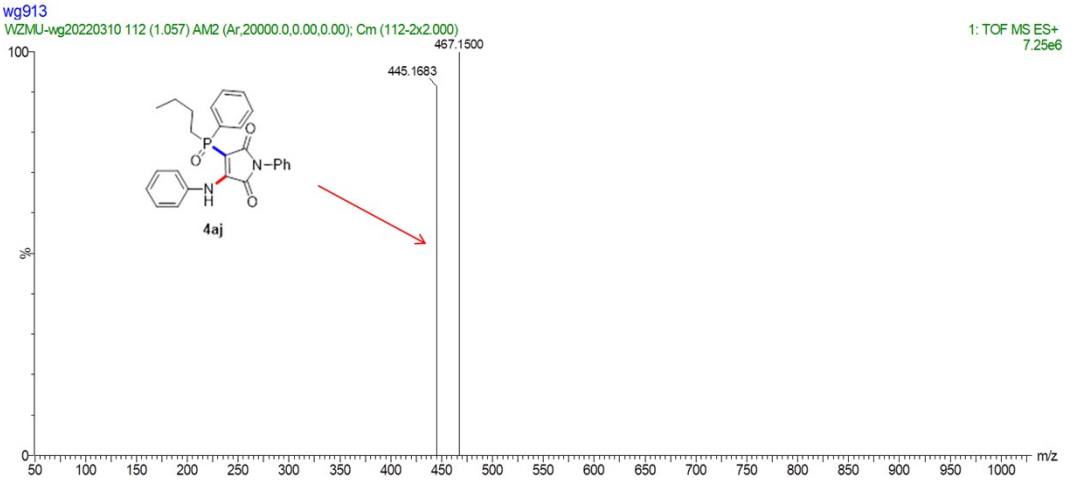

4af+4ag
WZMU20211207-16 11 (0.242) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm (11-2)
1: TOF MS ES+
2.47e7
525.1578
521.1993
P
O
N–Ph
PhHN
4ag
%
m/z


4ah+4ai
WZMU20211207-17 8 (0.172) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm(8-2)
1: TOF MS ES+
3.36e6
533.0585
535.0560
Cl
Cl
P
O
N–Ph
PhHN
4ah
%
m/z

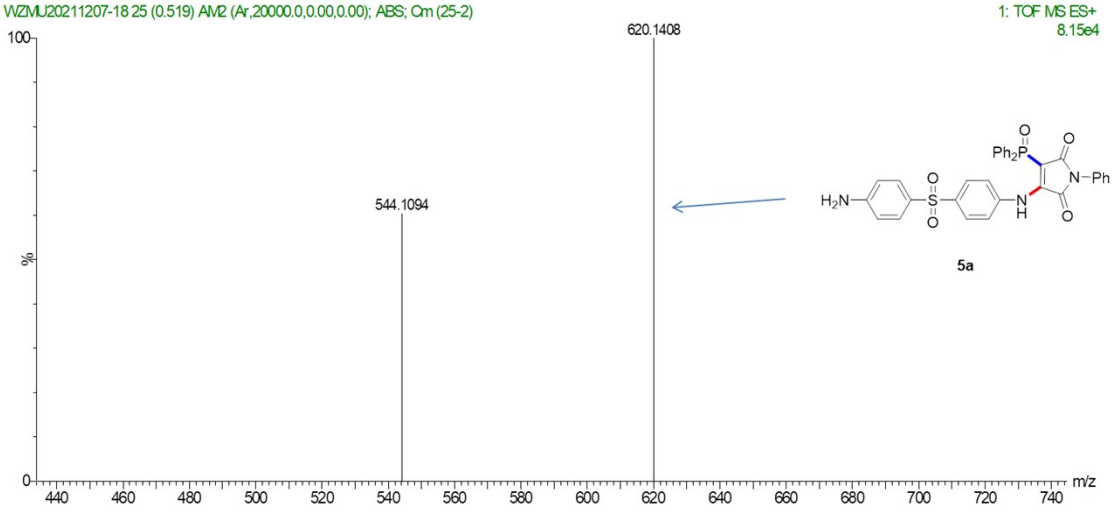
4ah+4ai



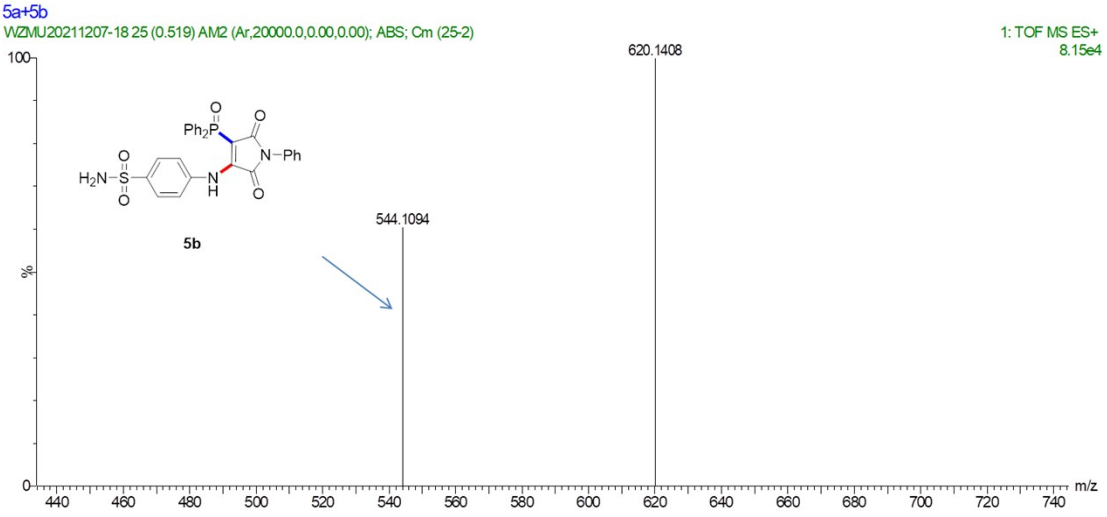
wg913



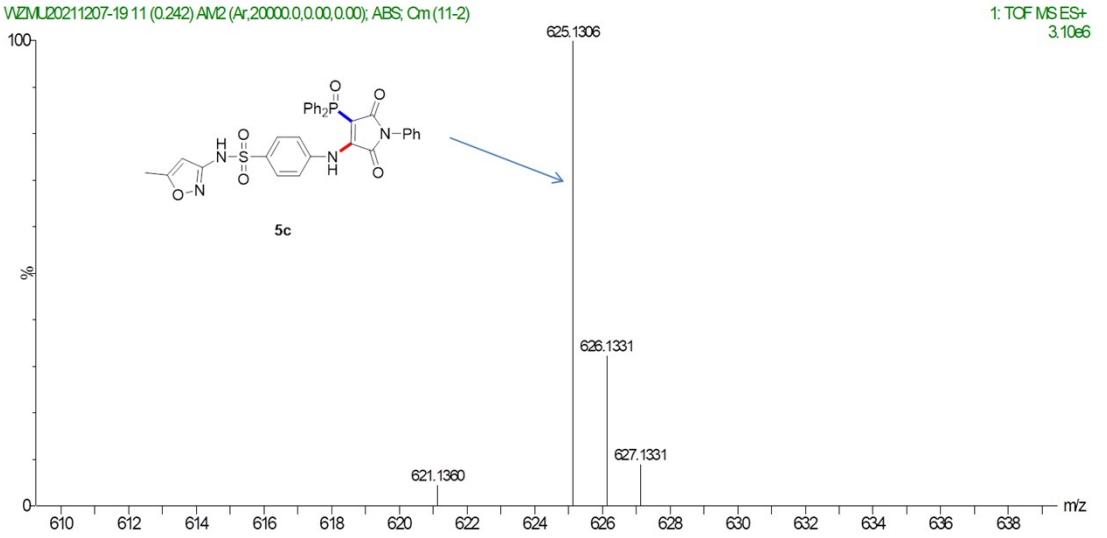
5a+5b



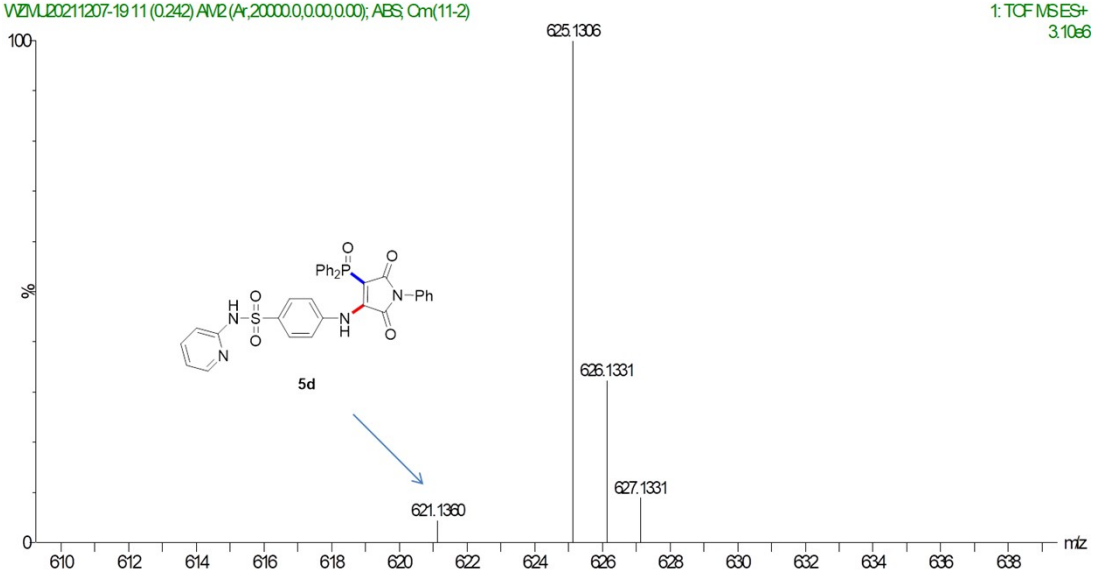
5a+5b

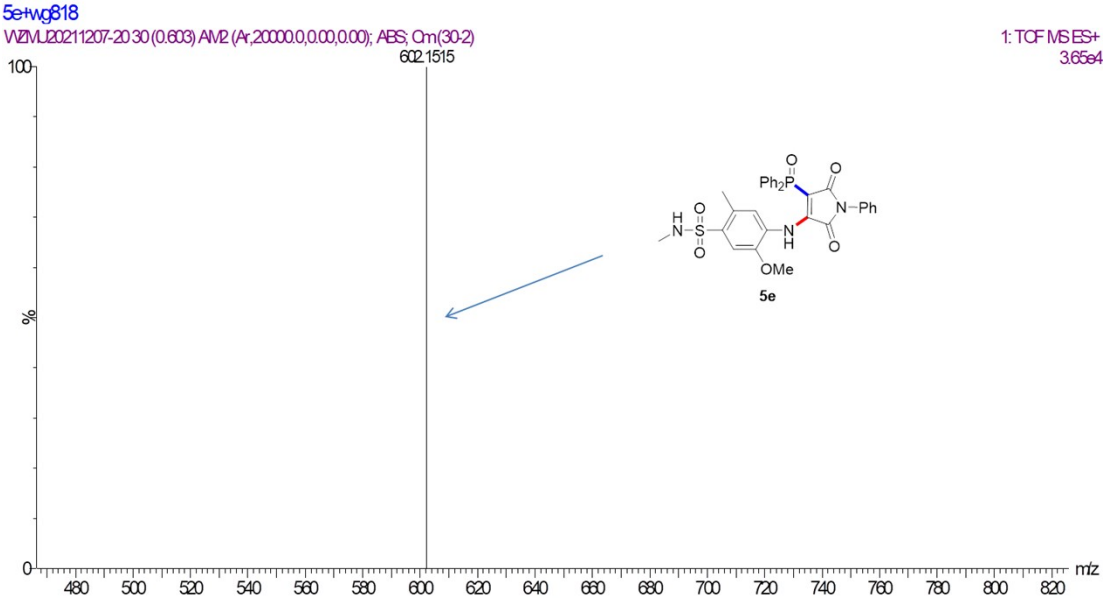


5c+5d



5c+5d




5e+wg818
VWMU20211207-2030 (0.603) AM2 (Ar,20000.0,0.00,0.00); ABS; Cm(30-2)
1: TOF MS ES+
3.65e4
602.1515
100
%
0
480
500
520
540
560
580
600
620
640
660
680
700
720
740
760
780
800
820
m/z
Ph2P
O
N–Ph
H
N
S
OMe
5e