Electronic Supplementary Information

Photoredox-Catalyzed Decarboxylative Alkylation/Cyclization of Alkynylphosphine Oxides: A Metal- and Oxidant-Free Method for Accessing Benzo[*b*]phosphole Oxides

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Content

1. General Information

The reactions were carried out in 10-mL schlenk tubes under N_2 atmosphere. Reagents were used as received unless otherwise noted, and solvents were purified according to standard operation procedure. Column chromatography was performed using Silica Gel 60 (300–400 mesh). The reactions were monitored by GC and GC-MS, GC-MS results were recorded on GC-MS QP2010, and GC analysis was performed on GC 2014 plus. The ^{1}H, ^{13}C NMR, ^{31}P NMR and ^{19}F NMR spectra were recorded on a Brucker ADVANCE III spectrometer at 400 MHz, 100 MHz, 162 MHz and 376 MHz respectively, and chemical shifts were reported in parts per million (ppm). The HRMS measurements were conducted at Liaocheng University, Shandong, China. All solvents and reagents were purchased from Energy Chemical, Alfa Aesar, and Aladdin.

Fluorescence titrations of photocatalysts and Stern-Volmer luminescence quenching analyses were conducted using a HITACHI F4600 fluorescence spectrophotometer with a 10 mm standard quartz cuvette at room temperature. The following parameters were employed: EX Slit = 5 nm, EM Slit = 5.0 nm, PMT Voltage = 700 V, Scan speed = 1200 nm/min, Response time = 2.0 s. Samples were prepared in quartz cuvettes, and the solutions were irradiated at 410 nm and the luminescence was measured at 561 nm.

The fluorescence emission of products was conducted using a HITACHI F4600 fluorescence spectrophotometer with a 10 mm standard quartz cuvette at room temperature. The following parameters were employed: EX Slit = 10 nm, EM Slit = 20 nm, PMT Voltage = 500 V, Scan speed = 1200 nm/min, Response time = 2.0 s. Samples were prepared in quartz cuvettes, and the solutions were irradiated at 325nm.

Quantum yield measurements and the UV-vis absorption of products were conducted using an Aglient Cary100 UV-VIS spectrophotometer with a 10 mm standard quartz cuvette at room temperature.

2. Experimental Procedure

2.1 General experimental procedure for the synthesis of benzo[*b*]phosphole oxides

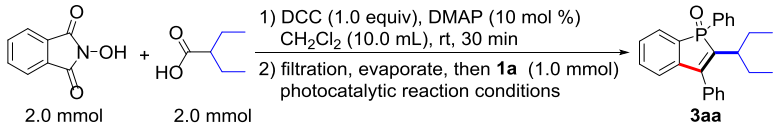
An oven-dried 10-mL Schlenk tube, equipped with a magnetic stir bar and charged with diphenyl(phenylethynyl)phosphine oxides **1** (0.2 mmol), alkyl NHPI esters **2** (0.4 mmol, 2.0 equiv), and Eosin B (0.004 mmol, 2 mol %) was evacuated and backfilled with N_2 three times. In the absence of light, DMSO (2.0 mL), H_2O (0.4 mmol, 2.0 equiv), and *i*-Pr_2NEt (0.2 mmol, 1.0 equiv) were then added and the tube was sealed. The mixture was then stirred under irradiation from Blue LED strips (2 m; 5.8 ±0.5 W/m, 120 LEDs per meter) at room temperature for 10 h and monitored by GC or GC-MS or TLC. Upon completion, the reaction mixture was cooled to room temperature, diluted with EtOAc, and washed with H_2O. The organic layer was dried with anhydrous Na_2SO_4, subject to filtration, and concentrated in vacuo. The residue was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate to afford the desired product **3**.

2.5 mmol gram-scale synthesis of 2-(pentan-3-yl)-1,3-diphenylphosphindole 1-oxide 3aa:

An oven-dried 100-mL Schlenk tube, equipped with a magnetic stir bar and charged with diphenyl(phenylethynyl)phosphine oxides **1a** (755 mg, 2.5 mmol), alkyl NHPI esters **2a** (1.3 g, 5.0 mmol, 2.0 equiv), and Eosin B (32.5 mg, 0.05 mmol, 2 mol %) was evacuated and backfilled with N_2 three times. In the absence of light, DMSO (25 mL), H_2O (90 μL, 5.0 mmol, 2.0 equiv), and *i*-Pr_2NEt (0.42 mL, 2.5 mmol, 1.0 equiv) were then added and the tube was sealed. The mixture was then stirred under irradiation from Blue LED strips at room temperature for 12 h and monitored by GC or GC-MS or TLC. Upon completion, the reaction mixture was cooled to room temperature, diluted with EtOAc, and washed with H_2O. The organic layer was dried with anhydrous Na_2SO_4, subject to filtration, and concentrated in vacuo. The residue

was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2/1) to afford the desired yellow wax product **3aa** (650 mg, 70%).

1.0 mmol gram-scale synthesis of 2-(pentan-3-yl)-1,3-diphenylphosphindole 1-oxide 3aa via one-pot:

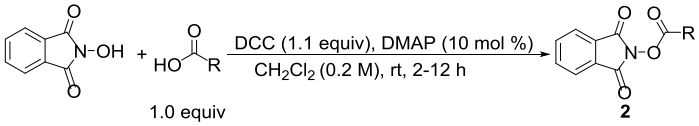


A round-bottom flask was charged with *N*-hydroxyphthalimide (325 mg, 2.0 mmol), *N*,*N*´-dicyclohexylcarbodiimide (453 mg, 2.2 mmol, 1.1 equiv), and DMAP (0.1 equiv). Dry dichloromethane was added (10 mL), and the mixture was stirred vigorously. 2-Ethylbutyric acid (255 μL) was added via syringe and the mixture was allowed to stir for 30 min (determined by TLC). The mixture was filtered over Celite and rinsed with additional CH_2Cl_2. The solvent was removed under reduced pressure to afford crude alkyl NHPI esters **2a**.

An oven-dried 25-mL Schlenk tube, equipped with a magnetic stir bar and charged with diphenyl(phenylethynyl)phosphine oxides **1a** (302 mg, 1.0 mmol), crude alkyl NHPI esters **2a**, and Eosin B (13 mg, 0.02 mmol, 2 mol %) was evacuated and backfilled with N_2 three times. In the absence of light, DMSO (25 mL), H_2O (36 μL, 2.0 mmol, 2.0 equiv), and *i*-Pr_2NEt (167 μL, 1.0 mmol, 1.0 equiv) were then added and the tube was sealed. The mixture was then stirred under irradiation from Blue LED strips at room temperature for 10 h and monitored by GC or GC-MS or TLC. Upon completion, the reaction mixture was cooled to room temperature, diluted with EtOAc, and washed with H_2O. The organic layer was dried with anhydrous Na_2SO_4, subject to filtration, and concentrated in vacuo. The residue was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2/1) to afford the desired yellow wax product **3aa** (270 mg, 73%).

2.2 General procedure for the synthesis of *N*-(Acyloxy)phthalimides

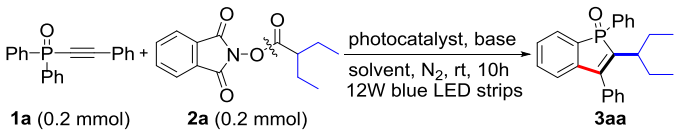
The redox-active esters *N*-(Acyloxy)phthalimides **2** were synthesized by a slightly modified procedure based on Baran[1].



A round-bottom flask was charged with *N*-hydroxyphthalimide (1.0 equiv) carboxylic acid (1.0 equiv, if solid), *N*,*N*´-dicyclohexylcarbodiimide (1.1 equiv), and DMAP (0.1 equiv). Dry dichloromethane was added (0.2 M), and the mixture was stirred vigorously. Carboxylic acid (1.0 equiv) was added via syringe (if liquid) and the mixture was allowed to stir until the carboxylic acid was consumed (determined by TLC). Typical reaction times were between 2 h and 12 h. The mixture was filtered over Celite and rinsed with additional CH_2Cl_2. The solvent was removed under reduced pressure, and purification by column chromatography afforded corresponding activated esters **2** in 60–85% yields.

2.3 Optimization of Conditions

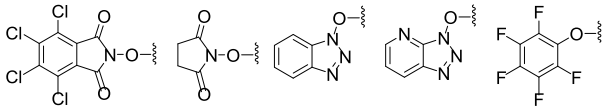
Table SI-1[a]



1a (0.2 mmol) **2a** (0.2 mmol) **3aa**

entry	photocatalyst (mol %)	base (equiv)	solvent	yield (%)[b]
1	$Ru(bpy)_3Cl_2$•$6H_2O$ (2.0)	*i*-Pr_2NEt (3.0)	DMAc	26
2	Eosin Y (2.0)	*i*-Pr_2NEt (3.0)	DMAc	53
3	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	DMAc	55
4	Rose Bengal (2.0)	*i*-Pr_2NEt (3.0)	DMAc	50
5	Methylene Blue (2.0)	*i*-Pr_2NEt (3.0)	DMAc	none
6	Eosin B (2.0)	DABCO (3.0)	DMAc	52
7	Eosin B (2.0)	Et_3N (3.0)	DMAc	51
8	Eosin B (2.0)	DBU (3.0)	DMAc	33
9	Eosin B (2.0)	TMEDA (3.0)	DMAc	47
10	Eosin B (2.0)	2,6-lutidine (3.0)	DMAc	34
11	Eosin B (2.0)	K_2CO_3 (3.0)	DMAc	30
12	Eosin B (2.0)	---	DMAc	18
13	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	DMF	50
14	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	DMSO	69
15	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	CH_3CN	7
16	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	THF	11
17	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	*i*-PrOH	20
18[c]	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	DMSO	75
19[d]	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	DMSO	84
20[d]	Eosin B (1.0)	*i*-Pr_2NEt (3.0)	DMSO	73
21[d]	Eosin B (3.0)	*i*-Pr_2NEt (3.0)	DMSO	83
22[d,e]	Eosin B (2.0)	*i*-Pr_2NEt (3.0)	DMSO	88
23[d,e]	Eosin B (2.0)	*i*-Pr_2NEt (1.0)	DMSO	87(82)[f]
24[d,e]	--	*i*-Pr_2NEt (1.0)	DMSO	none
25[d,e,g]	Eosin B (2.0)	*i*-Pr_2NEt (1.0)	DMSO	none

Other activated esters



TCNHPI 53%[b,d,e] NHS 0%[d,e] HOBt 0%[d,e] HOAt 0%[d,e] 0%[d,e]

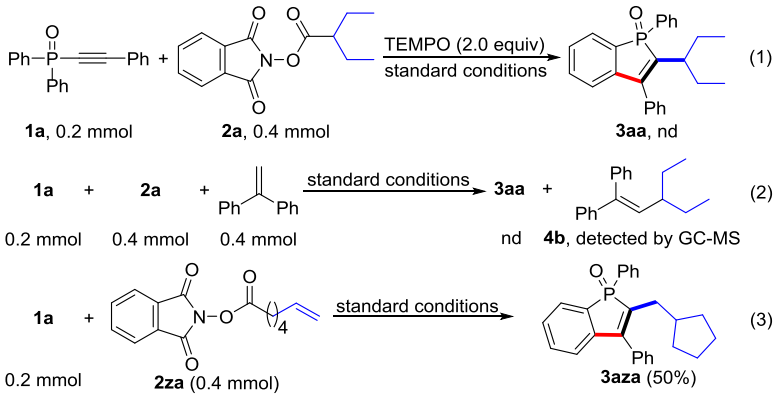
[a]Reaction conditions:**1a** (0.2 mmol), **2a** (0.2 mmol, 1.0 equiv), photocatalyst, base, solvent (2.0 mL), N_2, rt, 12 W blue LED strips, 10 h. [b]^{31}P NMR yield using tributyl phosphate as an internal standard. [c]**2a** (0.3 mmol, 1.5 equiv). [d]**2a** (0.4 mmol, 2.0 equiv). [e]H_2O (2.0 equiv) was added. [f]Isolated yield. [g]No light.

We commenced our studies with the reaction of diphenyl(phenylethynyl)phosphine oxide **1a** and alkyl NHPI ester **2a** in the presence of 2.0 mol % $Ru(bpy)_3Cl_2$•$6H_2O$, *i*-Pr_2EtN (*N*,*N*-diisopropylethylamine) in 2.0 mL of DMAc (*N*,*N*-dimethylacetamide) at room temperature under visible-light irradiation for 10 h. Gratifyingly, the desired decarboxylative Alkylation / Cyclization product **3aa** was obtained in 26% yield (Table 1, entry 1). Initially, screening of common photocatalysts found that organic dye Eosin B instead of Ru-complex as a catalyst was more efficient candidate (Table 1, entries 1–5) to promote the reaction with the desired **3aa** obtained in 55% yield (Table 1, entry 3). Next, organic or inorganic bases, such as DABCO (triethylenediamine), Et_3N, DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), TMEDA (*N*,*N*,*N*',*N*'-tetramethylethylenediamine), 2,6-lutidine and K_2CO_3 were examined as an additive (entries 6−11), and *i*-Pr_2EtN gave the best yield (55%, entry 3). Furthermore, the result proved that a base could facilitate the reaction (entry 12 *vs* entries 3 and 6−11). Further investigation of solvents (DMF, DMSO, CH_3CN, THF and *i*-PrOH) revealed that the reaction in DMSO led to the best yield (69%, entry 14). Increasing the loading of alkyl NHP ester **2a** could give the desired result (84% yield, entry 19). In addition, changing the loading of Eosin B did not increase obviously the yield (Table 1, entries 20 and 21). Satisfactorily, the loading of additional H_2O could facilitate the reaction (88%, entry 22) due to the hydrogen

bonding can increase the electron-acceptor strength of alkyl NHPI ester.[2] Furthermore, the loading of base *i*-Pr_2EtN could reduce to 1.0 equivalent and give the desired result (87%, entry 23). Notably, no desired product was observed in the absence of photocatalysts or the light (Table 1, entries 24 and 25). Finally, when the other classic NHS, HOBt, HOAt, or pentafluorophenol esters associated with 2-ethylbutyric acid coupling were employed and no product was observed by GC-MS. Replacing the NHPI ester **2a** with the more electron deficient tetrachloro derivative (TCNHPI) decrease the yield instead (53% *vs* 87%, entry 23)

3. Investigation on the reaction mechanism

3.1. The radical trapping experiments



3.2 Kinetic profiles in response to light irradiation

The time course of product formation and raw material consumption during the reaction were investigated in parallel. An oven-dried 10-mL Schlenk tube, equipped with a magnetic stir bar and charged with diphenyl(phenylethynyl)phosphine oxide **1a** (0.2 mmol), alkyl NHPI ester **2a** (0.4 mmol, 2.0 equiv), and Eosin B (0.004 mmol, 2 mol %), was evacuated and backfilled with N_2 three times. In the absence of light, DMSO (2.0 mL), H_2O (0.4 mmol, 2.0 equiv), and *i*-Pr_2NEt (0.2 mmol, 1.0 equiv) were then added and the tube was sealed. The mixture was then stirred under irradiation from Blue LED strips (2 m; 5.8 ±0.5 W/m, 120 LEDs per meter) at room temperature for specified time intervals. Upon completion, the ^{31}P NMR yield was determined every hour by Brucker ADVANCE III spectrometer using tributyl phosphate as internal standard.

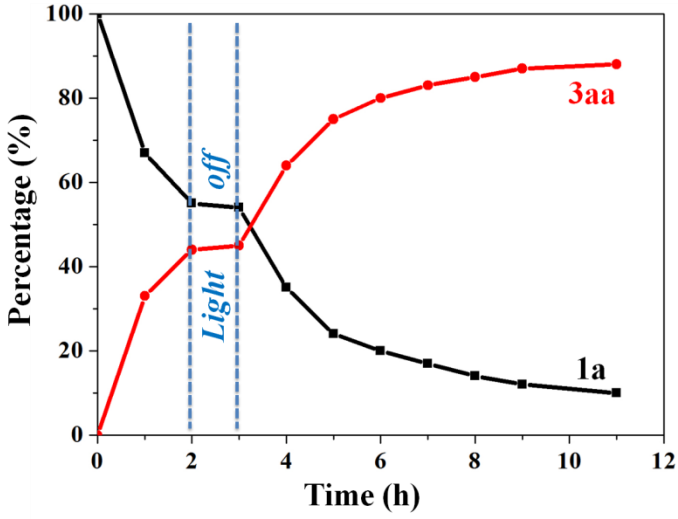


Figure S1. Kinetic profiles for the decarboxylative alkylation/cyclization of **1a** in response to light

irradiation.

3.3 Fluorescence titration of photocatalysts

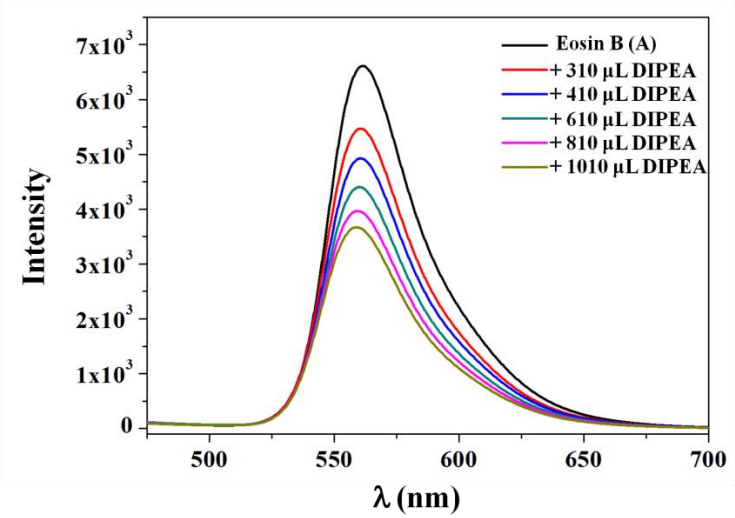


Figure S2. Fluorescence quenching of Eosin B (**A**, 1.0×10^{-5} M in DMSO, 1.5 mL) upon titration with DIPEA (*i*-Pr_2NEt)(0.1 M in DMSO).

3.4 Quantum yield measurements

The quantum yield of the reaction was measured by chemical actinometry using a set of 12W LEDs (λ= 400-410 nm) using potassium ferrioxalate following the procedure of Melchiorre[3a] and Glorius[3b]

The solutions were prepared and stored in the dark:

(1) Potassium ferrioxalate solution: 176 mg of potassium ferrioxalate trihydrate (commercially available from Alfa Aesar) and 84 µL of H_2SO_4 (95-98%) were dissolved in 20 mL of distilled water.
(2) Phenantroline solution: 50 mg of 1,10-phenanthroline was dissolved in 25 mL of distilled water.
(3) Buffer solution: 1.25 g of sodium acetate and 250 µL of H_2SO_4 (95-98%) were dissolved in 25 mL of distilled water.

The actinometry measurements were done as follows:

(1) 2.0 mL of the actinometer solution was added to a 10-mL Schlenk tube containing a stirring bar. Then, the solution was irradiated with 12W LEDs (λ= 400-410 nm) for *specified time intervals* (60, 90, 120, 150) s.
(2) After irradiation, a 30 µL aliquot was immediately taken and added to a 10 mL volumetric flask containing 0.2 mL of 1,10-phenanthroline solution and 3.0 mL of buffer solution, and the solution was then allowed to rest for 1 h in dark place to allow the ferrous ions to completely coordinate to the phenanthroline.
(3) The UV/Vis spectra of actinometry samples were recorded for each time interval. The absorbance of the actinometry solution was monitored at 510 nm. In a similar manner, this procedure is repeated with the actinometer solution stored in the dark.
(4) The moles of Fe^{2+} formed for each sample is determined according to the Beers' Law (eq 3):

$$\text{moles } Fe^{2+} = \frac{V_1 V_3 \Delta A(510\text{ nm})}{10^3 V_2 l \varepsilon(510\text{ nm})} \qquad (3)$$

Where:

V_1 = Irradiated volume (2.0 mL).

V_2 = The aliquot of the irradiated solution taken for the estimation of Fe^{+} ions (0.030 mL).

V_3 = Final volume of the solution after complexation with 1,10-phenanthroline (3.23 mL).

ε(510 nm) = Molar extinction coefficient of $[Fe(Phen)_3]^{2+}$ complex (11100 L $mol^{-1}cm^{-1}$).

l = Optical path-length of the cuvette (1 cm)

ΔA(510 nm) = absorbance difference between the irradiated solution and the solution stored in dark.

(5) The moles of Fe^{2+} formed (dx) are plotted as a function of time (dt) (Figure S3). The slope of this line was correlated to the moles of incident photons per unit time (**F = photon flux**) by the use of the following equation (4):

$$\Phi(\lambda) = \frac{\frac{dx}{dt}}{F(1\text{-}10^{-A(\lambda)})} \qquad (4)$$

Where:

Φ(λ) = The quantum yield for Fe^{2+} formation at 400 nm is 1.13[2c]

A(λ) = ferrioxalate actinometer absorbance at 400 nm, which was measured placing 3.0 mL of the solution in a cuvette of path length 1 cm by UV/Vis spectrophotometry. We obtained an absorbance value of 2.818. The determined incident photons per unit time (**F**) is **5.08×10^{-8} einsteins/s.**

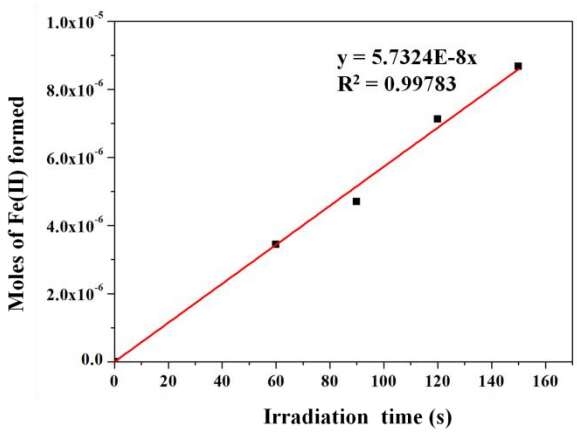


Figure S3. Plot of moles of Fe(II) formed as a function of time.

(6) To obtain the quantum yield (Φ) of the oxidative alkylation/cyclization reaction the number of moles of the product **3aa** were determined by GC analysis using tributyl phosphate as internal standard. As such, a photocatalytic reaction was performed under the set of optimized reaction conditions (Table SI-1, entry 22) under visible light irradiation of 12W LED (λ= 400–410 nm). After 30 minutes of light irradiation, **1.48×10^{-5}** moles of **3aa** (yield = 7.4%).were obtained. The quantum yield of this reaction was calculated using the following equation (5):

$$\Phi(\lambda) = \frac{\text{moles of product}}{F(1\text{-}10^{-A(400\ nm)})t} \qquad (5)$$

Where:

A(400 nm) = is the absorbance at 400 nm of the photocatalytic reaction which was measured placing 3.0 mL of the solution (30 μL reaction aliquot was taken and added to a 3.0 mL DMSO) in a cuvette of path length 1 cm by UV/Vis spectrophotometry.

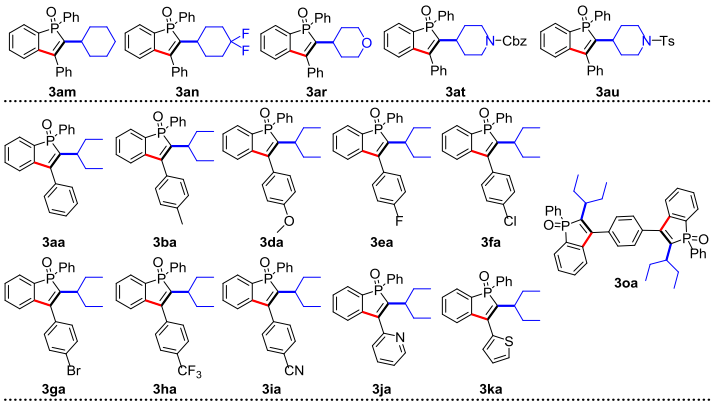
t = is the reaction time (1800s)

The quantum yield (Φ) of the reaction is 1.43 (see calculation below).

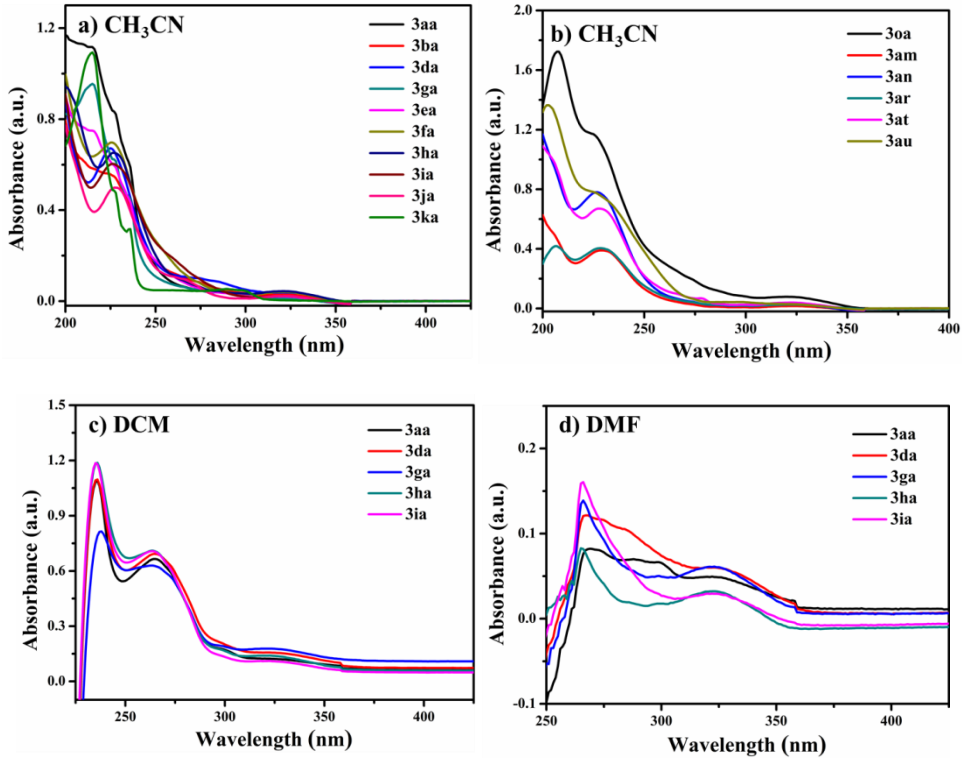
$$\Phi(400\ \text{nm}) = \frac{\text{moles of } \mathbf{3aa}}{F(1-10^{-A(400\ \text{nm})})t} = \frac{1.48*10^{-5}\ \text{moles of } \mathbf{3aa}}{5.08*10^{-8}\ \frac{\text{einsteins}}{s}(1-10^{-0.052})(1800s)} = 1.43$$

4. The photophysical data of the products

4.1 The optical representative test sample



4.2 The UV-vis absorption and fluorescence emission spectra of the products



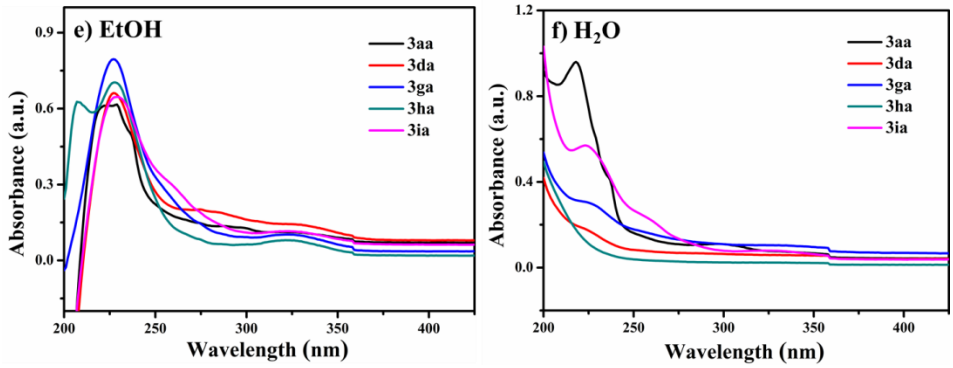


Figure S4. The UV-vis absorption spectra of the products in various solvents (CH_3CN, DCM, DMF, EtOH, and H_2O) at 2×10^{-5} mol/L at 25 °C.

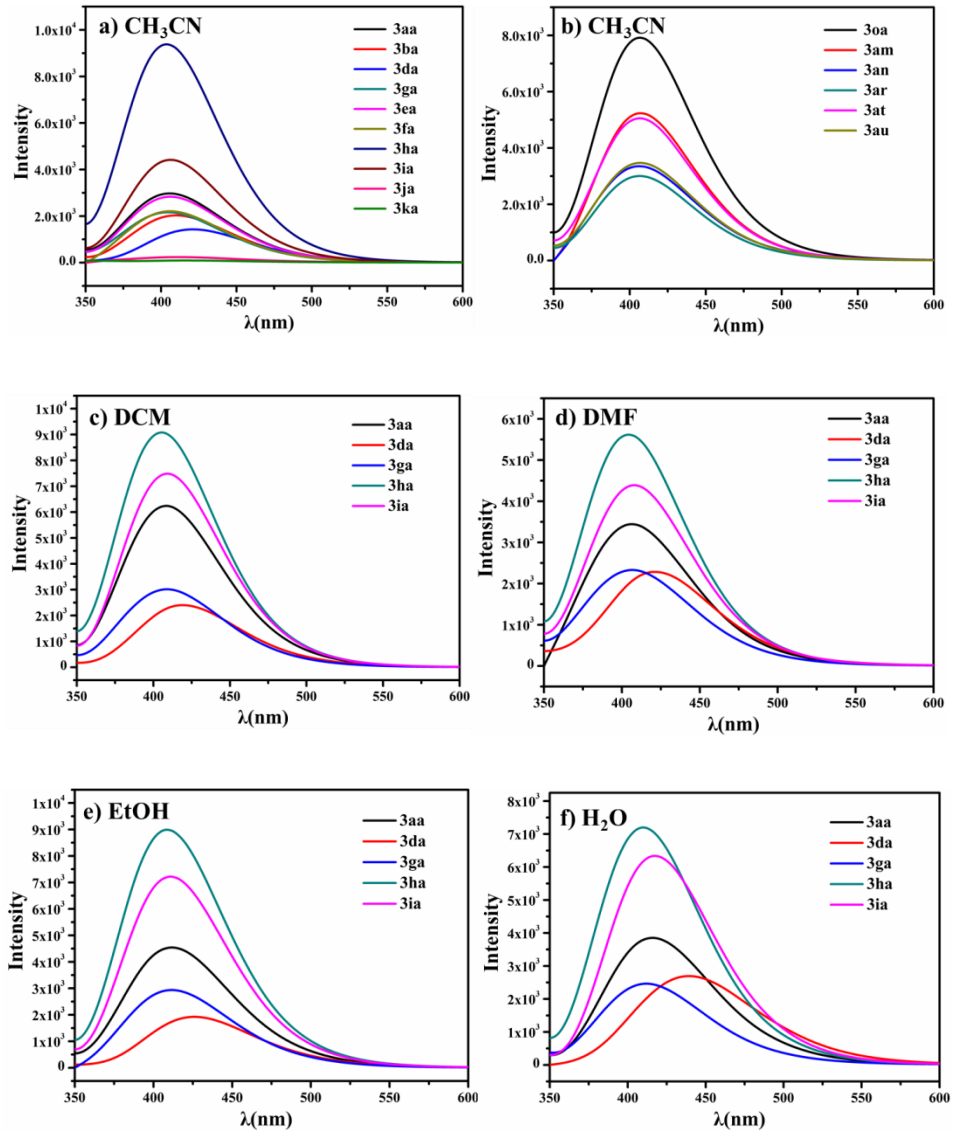


Figure S5. The fluorescence emission spectra of the products in various solvents (CH_3CN, DCM, DMF, EtOH, and H_2O) at 2×10^{-5} mol/L at 25 °C.

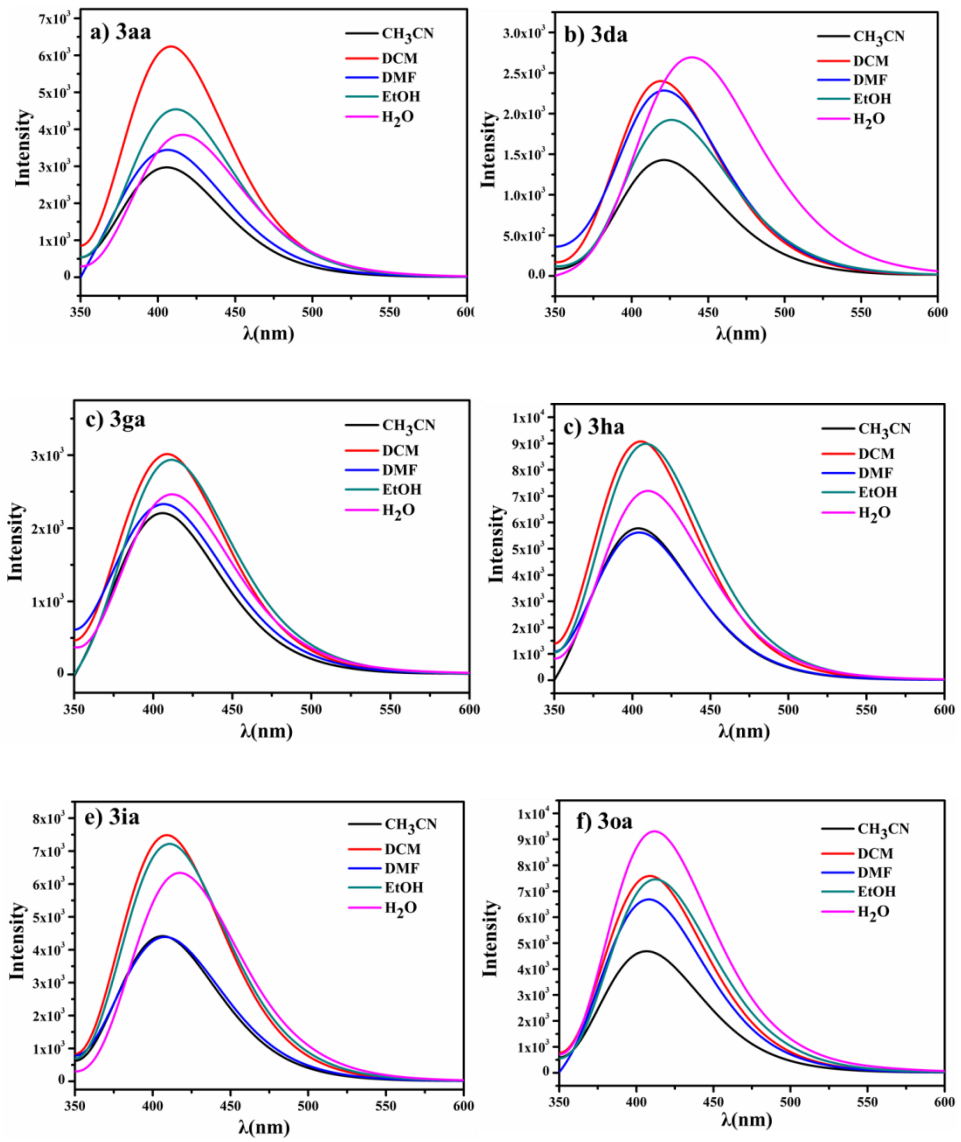


Figure S6. The solvent effect of the products on fluorescence emission at 2×10^{-5} mol/L solvents(CH_3CN, DCM, DMF, EtOH, and H_2O) at 25 °C.

Conclusion:

1) The maximum UV-vis absorption wavelengths of product are concentrated at 320–325nm, and the absorbance are weak(Figure S4);
2) The fluorescence emission of product are concentrated at 400–420nm, and the emission intensity is obviously related to the electronegativity of the group and solvent (Figure S5 and S6);
3) The product substituted with strong electron-withdrawing group have the much stronger emission intensity (**3ha**, **3ai**) than electron-donating group (**3da**) and the intensity of the order is CF_3>CN>H>Br>OMe in various solvents (CH_3CN, DCM, DMF, EtOH and H_2O) (Figure S5 (a), (c)-(f));
4) When the product substituted with OMe (3da), red-shifted fluorescence bands was observed (Figure S5 a), c)-f));
5) The solvent effect of the product is noteworthy. As the polarity of the solvent increases, red-shifted fluorescence bands was more or less observed (Figure S6), especially, the product **3da** is most obvious (Figure S6 (b)).

4.2 The UV-vis absorption and fluorescence emission data of the products

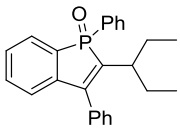
Table SI-2 Photophysical data of the representative products[a]

Compound	λ_{abs}/nm	Absorbance	ε($M^{-1}cm^{-1}$)	λ_{em}/nm
3aa	321	0.01594	797	406
3ba	321	0.03122	1561	410
3da	321	0.03833	1916	421
3ea	321	0.01317	658	405
3fa	321	0.0211	1055	406
3ga	321	0.04033	2016	406
3ha	321	0.04392	2196	403
3ia	323	0.02262	1131	406
3ja	321	0.02291	1145	411
3ka	324	0.00371	185	415
3oa	320	0.07807	3903	406
3am	324	0.01665	832	407
3an	321	0.02964	1482	406
3ar	321	0.02638	1319	406
3at	321	0.03933	1966	406
3au	321	0.03003	1501	407

[a]Emission maximum in CH_3CN at 2×10^{-5} mol/L.

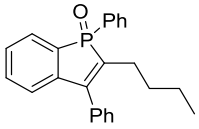
5. ^{1}H, ^{13}C and ^{31}P NMR spectra data of the products

2-(Pentan-3-yl)-1,3-diphenylphosphindole 1-oxide(3aa)



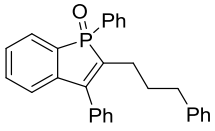
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 61.0 mg, 82%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83–7.72 (m, 2H), 7.60–7.48 (m, 4H), 7.44–7.23 (m, 7H), 6.91 (d, J = 7.2 Hz, 1H), 2.39–2.33 (m, 1H), 1.98–1.91 (m, 1H), 1.57–1.54 (m, 1H), 1.26–1.19 (m, 1H), 1.09–1.02 (m, 1H), 0.88 (t, J = 5.8 Hz, 3H), 0.43 (t, J = 5.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.6 (d, $J_{C\text{-}P}$ = 22.6 Hz), 144.3 (d, $J_{C\text{-}P}$ = 27.7 Hz), 139.9 (d, $J_{C\text{-}P}$ = 89.1 Hz), 134.4 (d, $J_{C\text{-}P}$ = 15.9 Hz), 133.9, 132.7 (d, $J_{C\text{-}P}$ = 1.6 Hz), 131.9 (d, $J_{C\text{-}P}$ = 82.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.1 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.9 (d, $J_{C\text{-}P}$ = 73.5 Hz), 128.7 (d, $J_{C\text{-}P}$ = 6.8 Hz), 128.5 (d, $J_{C\text{-}P}$ = 5.3 Hz), 128.4 (d, $J_{C\text{-}P}$ = 3.9 Hz), 128.2 (d, $J_{C\text{-}P}$ = 32.0 Hz), 123.2, 123.0 (d, $J_{C\text{-}P}$ = 10.8 Hz), 44.1 (d, $J_{C\text{-}P}$ = 9.1 Hz), 27.4 (dd, $J_{C\text{-}P}$ = 74.1, 1.6 Hz), 12.53 (d, $J_{C\text{-}P}$ = 53.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.47. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{25}OPH^+$ 373.1716; found 373.1740.

2-Butyl-1,3-diphenylphosphindole 1-oxide (3ab)[4]



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow gum; yield: 43.0 mg, 60%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 2H), 7.63–7.59 (m, 1H), 7.54–7.48 (m, 3H), 7.44–7.44 (m, 3H), 7.39–7.37 (m, 1H), 7.33–7.28 (m, 3H), 6.94 (d, J = 7.6 Hz, 1H), 2.50–2.38 (m, 1H), 2.26–2.17 (m, 1H), 1.39–1.29 (m, 2H), 1.16–1.05 (m, 2H), 0.56 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2 (d, $J_{C\text{-}P}$ = 22.3 Hz), 144.2 (d, $J_{C\text{-}P}$ = 27.9 Hz), 136.7 (d, $J_{C\text{-}P}$ = 93.7 Hz), 133.8 (d, $J_{C\text{-}P}$ = 15.9 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.7 (d, $J_{C\text{-}P}$ = 104.8 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.0 (d, $J_{C\text{-}P}$ = 97.4 Hz), 128.7, 128.6 (2C, overlap), 128.45, 128.4 (d, $J_{C\text{-}P}$ = 3.4 Hz), 128.3, 123.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 30.7, 26.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 22.5, 13.4. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.93.

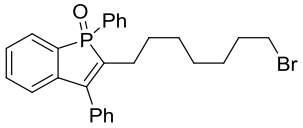
1,3-Diphenyl-2-(3-phenylpropyl)phosphindole 1-oxide (3ac)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 28.3 mg, 34%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.71 (m, 2H), 7.62 (t, J = 8.4 Hz, 1H), 7.54–7.37 (m, 7H), 7.28–7.25 (m, 3H), 7.15–7.06 (m, 3H), 7.03 (d, J = 7.6 Hz, 1H), 6.88 (d, J = 7.2 Hz, 2H), 2.52–2.34 (m, 3H), 2.30–2.18 (m, 1H), 1.80–1.63 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.6 (d, $J_{C\text{-}P}$ = 22.2 Hz), 144.1 (d, $J_{C\text{-}P}$ = 28.0 Hz), 141.4, 136.3 (d, $J_{C\text{-}P}$ = 93.7 Hz), 133.7 (d, $J_{C\text{-}P}$ = 15.8 Hz), 132.8 (d, $J_{C\text{-}P}$ = 1.7 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.0, 130.9 (d, $J_{C\text{-}P}$ = 10.7 Hz), 129.8 (d, $J_{C\text{-}P}$ = 98.1 Hz), 128.9, 128.8, 128.78, 128.73, 128.5, 128.4, 128.4 (d, $J_{C\text{-}P}$ = 5.8 Hz), 128.1 (d, $J_{C\text{-}P}$ = 15.1 Hz), 125.5, 123.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 35.4, 29.9, 26.0 (d, $J_{C\text{-}P}$ = 10.1 Hz). ^{31}P NMR (162

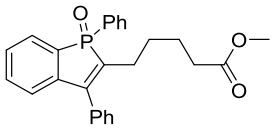
MHz, $CDCl_3$): δ 40.01. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{29}H_{25}OPH^+$ 421.1716; found 421.1710.

2-(7-Bromoheptyl)-1,3-diphenylphosphindole 1-oxide (3ad)



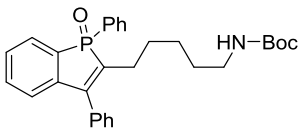
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 36.3 mg, 38%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 2H), 7.63–7.59 (m, 1H), 7.55–7.49 (m, 3H), 7.47–7.37 (m, 4H), 7.32–7.28 (m, 3H), 7.02 (d, J = 7.2 Hz, 1H), 3.31 (t, J = 6.8 Hz, 2H), 2.49–2.38 (m, 1H), 2.26–2.15 (m, 1H), 1.73–1.66 (m, 2H), 1.36–1.34 (m, 2H), 1.22–1.18 (m, 2H), 1.15–1.01 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.3 (d, $J_{C\text{-}P}$ = 22.3 Hz), 144.1 (d, $J_{C\text{-}P}$ = 28.0 Hz), 136.5 (d, $J_{C\text{-}P}$ = 93.8 Hz), 133.8 (d, $J_{C\text{-}P}$ = 15.9 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.6 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.7 (d, $J_{C\text{-}P}$ = 105.1 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.6 Hz), 129.9 (d, $J_{C\text{-}P}$ = 97.4 Hz), 128.8, 128.7, 128.6, 128.5, 128.42, 128.40, 123.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 33.8, 32.5, 29.1, 28.4, 27.9, 27.7, 26.4 (d, $J_{C\text{-}P}$ = 10.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.89. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{27}H_{28}BrOPH^+$ 479.1134; found 479.1089.

Methyl 5-(1-oxido-1,3-diphenylphosphindol-2-yl)pentanoate (3ae)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 44.1 mg, 53%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 2H), 7.62 (t, J = 8.4 Hz, 1H), 7.55–7.49 (m, 3H), 7.47–7.38 (m, 4H), 7.32–7.30 (m, 3H), 7.03 (d, J = 7.2 Hz, 1H), 3.57 (s, 3H), 2.49–2.42 (m, 1H), 2.27–2.18 (m, 1H), 2.04–1.98 (m, 2H), 1.42–1.40 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.6, 150.6 (d, $J_{C\text{-}P}$ = 22.1 Hz), 144.0 (d, $J_{C\text{-}P}$ = 27.7 Hz), 135.9 (d, $J_{C\text{-}P}$ = 93.7 Hz), 133.5 (d, $J_{C\text{-}P}$ = 15.8 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.6 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.5 (d, $J_{C\text{-}P}$ = 105.0 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.6 Hz), 129.7 (d, $J_{C\text{-}P}$ = 97.6 Hz), 128.8, 128.7, 128.6, 128.5 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.4, 128.3, 123.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 51.2, 33.2, 27.9 (d, $J_{C\text{-}P}$ = 1.3 Hz), 26.12 (d, $J_{C\text{-}P}$ = 10.2 Hz), 24.5. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.82. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{25}O_3PH^+$ 417.1614; found 416.1608.

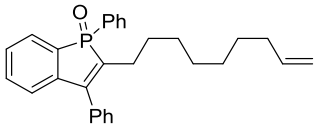
***tert*-Butyl (5-(1-oxido-1,3-diphenylphosphindol-2-yl)pentyl)carbamate (3af)**



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (1:1) to afford a yellow gum; yield: 38.9 mg, 40%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 2H), 7.61–7.54 (m, 1H), 7.56–7.39 (m, 7H), 7.31–7.28 (m, 3H), 7.03 (d, J = 7.6 Hz,

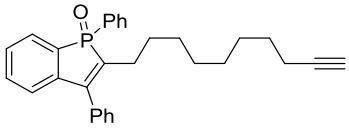
1H), 4.65 (s, 1H), 2.93–2.92 (m, 2H), 2.49–2.38 (m, 1H), 2.26–2.15 (m, 1H), 1.14 (s, 9H), 1.25–1.16 (m, 2H), 1.10–1.09 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.8, 150.6 (d, $J_{C\text{-}P}$ = 22.3 Hz), 144.1 (d, $J_{C\text{-}P}$ = 28.1 Hz), 135.8, 133.7 (d, $J_{C\text{-}P}$ = 15.8 Hz), 132.8 (d, $J_{C\text{-}P}$ = 1.7 Hz), 132.14, 132.11, 131.0, 130.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 129.8 (d, $J_{C\text{-}P}$ = 98.2 Hz), 128.8, 128.7, 128.6 (d, $J_{C\text{-}P}$ = 11.6 Hz), 128.4, 128.3, 123.2 (d, $J_{C\text{-}P}$ = 10.9 Hz), 41.6 (d, $J_{C\text{-}P}$ = 5.8 Hz), 40.4 (d, $J_{C\text{-}P}$ = 4.6 Hz), 29.2, 28.3, 28.0 (d, $J_{C\text{-}P}$ = 1.6 Hz), 26.4 (d, $J_{C\text{-}P}$ = 10.2 Hz), 23.4. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.99. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{30}H_{34}NO_3PH^+$ 488.2349; found 488.2360.

2-(Non-8-en-1-yl)-1,3-diphenylphosphindole 1-oxide (3ag)



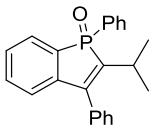
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 46.0 mg, 54%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85–7.70 (m, 3H), 7.64–7.60 (m, 1H), 7.53–7.49 (m, 3H), 7.46–7.41 (m, 3H), 7.39–7.31 (m, 3H), 7.03 (d, J = 7.2 Hz, 1H), 5.80–5.70 (m, 1H), 4.97–4.88 (m, 2H), 2.50–2.39 (m, 1H), 2.27–2.15 (m, 1H), 1.95–1.92 (m, 2H), 1.35–1.30 (m, 2H), 1.24–1.21 (m, 2H), 1.06–0.99 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.3 (d, $J_{C\text{-}P}$ = 22.4 Hz), 145.1 (d, $J_{C\text{-}P}$ = 28.0 Hz), 140.0, 137.5 (d, $J_{C\text{-}P}$ = 93.9 Hz), 134.7 (d, $J_{C\text{-}P}$ = 15.7 Hz), 134.3 (d, $J_{C\text{-}P}$ = 96.5 Hz), 133.7 (d, $J_{C\text{-}P}$ = 1.3 Hz), 133.0 (d, $J_{C\text{-}P}$ = 2.6 Hz), 132.5 (d, $J_{C\text{-}P}$ = 104.8 Hz), 131.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.8 (d, $J_{C\text{-}P}$ = 97.7 Hz), 129.7, 129.6, 129.5 (d, $J_{C\text{-}P}$ = 23.6 Hz), 129.3, 124.1, 124.0, 114.9, 34.5, 30.2, 29.67, 29.65, 29.63, 29.5 (d, $J_{C\text{-}P}$ = 0.8 Hz), 27.4 (d, $J_{C\text{-}P}$ = 10.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 40.23. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{29}H_{31}OPH^+$ 427.2185; found 427.2189.

2-(Dec-9-yn-1-yl)-1,3-diphenylphosphindole 1-oxide (3ah)



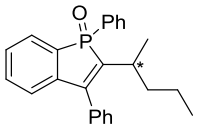
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 43.8 mg, 50%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 2H), 7.63–7.59 (m, 1H), 7.53–7.37 (m, 7H), 7.33–7.28 (m, 3H), 7.02 (d, J = 7.2 Hz, 1H), 2.49–2.38 (m, 1H), 2.26–2.16 (m, 1H), 2.12 (t, J = 7.2 Hz, 2H), 1.91 (s, 1H), 1.42 (t, J = 7.2 Hz, 2H), 1.35–1.21 (m, 5H), 1.06–0.99 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2 (d, $J_{C\text{-}P}$ = 22.2 Hz), 144.2 (d, $J_{C\text{-}P}$ = 27.9 Hz), 136.7 (d, $J_{C\text{-}P}$ = 93.6 Hz), 133.8 (d, $J_{C\text{-}P}$ = 16.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.7 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.7 (d, $J_{C\text{-}P}$ = 104.9 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.6 Hz), 130.0 (d, $J_{C\text{-}P}$ = 97.4 Hz), 128.7, 128.69, 128.67, 128.4, 128.39, 128.36, 123.1 (d, $J_{C\text{-}P}$ = 10.9 Hz), 84.6, 67.9, 29.3, 28.6 (d, $J_{C\text{-}P}$ = 3.6 Hz), 28.55, 28.54, 28.4, 28.2, 26.5 (d, $J_{C\text{-}P}$ = 10.1 Hz), 18.2. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.85. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{30}H_{31}OPH^+$ 439.2185; found 439.2187.

2-Isopropyl-1,3-diphenylphosphindole 1-oxide (3ai)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 49.6 mg, 72%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.77 (m, 2H), 7.57–7.44 (m, 7H), 7.38–7.26 (m, 4H), 6.92 (d, J = 7.6 Hz, 1H), 2.92–2.80 (m, 1H), 1.28 (d, J = 6.8 Hz, 3H), 0.79 (d, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.5 (d, $J_{C\text{-}P}$ = 22.4 Hz), 143.9 (d, $J_{C\text{-}P}$ = 27.6 Hz), 141.8 (d, $J_{C\text{-}P}$ = 90.8 Hz), 134.2 (d, $J_{C\text{-}P}$ = 15.8 Hz), 132.6 (d, $J_{C\text{-}P}$ = 1.7 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.5, 130.9 (d, $J_{C\text{-}P}$ = 96.8 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.7, 128.8, 128.5, 128.4 (d, $J_{C\text{-}P}$ = 11.1 Hz), 128.3 (d, $J_{C\text{-}P}$ = 16.1 Hz), 128.2, 123.0 (d, $J_{C\text{-}P}$ = 10.8 Hz), 28.9 (d, $J_{C\text{-}P}$ = 9.4 Hz), 22.4 (dd, $J_{C\text{-}P}$ = 24.2, 2.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.24. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{23}H_{21}OPH^+$ 345.1403; found 345.1406.

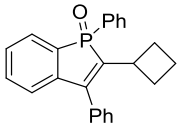
2-(Pentan-2-yl)-1,3-diphenylphosphindole 1-oxide (3aj)



One diastereoisomer 3aj': following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (3:1) to afford a pale yellow solid,; yield: 29.8 mg, 40%; mp 159–162 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80–7.74 (m, 2H), 7.58–7.43 (m, 7H), 7.39–7.27 (m, 4H), 6.93 (d, J = 7.2 Hz, 1H), 2.76–2.65 (m, 1H), 1.88–1.79 (m, 1H), 1.49–1.41 (m, 1H), 1.33–1.26 (m, 1H), 1.20–1.11 (m, 1H), 0.76–0.71 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.0 (d, $J_{C\text{-}P}$ = 22.3 Hz), 144.0 (d, $J_{C\text{-}P}$ = 27.8 Hz), 141.4 (d, $J_{C\text{-}P}$ = 90.2 Hz), 134.3 (d, $J_{C\text{-}P}$ = 16.1 Hz), 134.0, 132.6 (d, $J_{C\text{-}P}$ = 1.7 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.6 (d, $J_{C\text{-}P}$ = 3.1 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.7, 128.5, 128.4, 128.3, 128.2, 123.3, 123.1 (d, $J_{C\text{-}P}$ = 10.8 Hz), 38.0 (d, $J_{C\text{-}P}$ = 1.9 Hz), 34.1 (d, $J_{C\text{-}P}$ = 9.2 Hz), 20.9, 20.8 (d, $J_{C\text{-}P}$ = 2.4 Hz), 13.7. ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.85. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{25}OPH^+$ 373.1716; found 373.1725.

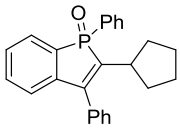
Another diastereoisomer 3aj'': following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (3:1) to afford a pale yellow solid; yield: 25.3 mg, 34%; mp 116–118 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 2H), 7.58–7.47 (m, 4H), 7.44–7.35 (m, 4H), 7.31–7.27 (m, 3H), 6.91 (d, J = 6.8 Hz, 1H), 2.74–2.59 (m, 1H), 1.30 (d, J = 6.8 Hz, 3H), 1.11–1.06 (m, 2H), 0.96–0.87 (m, 1H), 0.80–0.71 (m, 1H), 0.33 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.1 (d, $J_{C\text{-}P}$ = 22.4 Hz), 144.3 (d, $J_{C\text{-}P}$ = 27.5 Hz), 141.6 (d, $J_{C\text{-}P}$ = 90.0 Hz), 134.4 (d, $J_{C\text{-}P}$ = 15.8 Hz), 133.2 (d, $J_{C\text{-}P}$ = 136.2 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.9 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.5 (d, $J_{C\text{-}P}$ = 6.7 Hz), 131.1 (d, $J_{C\text{-}P}$ = 10.7 Hz), 129.7 (d, $J_{C\text{-}P}$ = 183.0 Hz), 128.58, 128.51, 128.46, 128.41, 128.2, 123.0 (d, $J_{C\text{-}P}$ = 10.8 Hz), 38.4 (d, $J_{C\text{-}P}$ = 2.2 Hz), 34.5 (d, $J_{C\text{-}P}$ = 9.4 Hz), 20.9, 20.5 (d, $J_{C\text{-}P}$ = 2.0 Hz), 13.3. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.54. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{25}OPH^+$ 373.1716; found 373.1730.

2-Cyclobutyl-1,3-diphenylphosphindole 1-oxide (3ak)



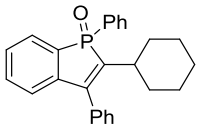
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 28.5 mg, 40%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73–7.71 (m, 2H), 7.49–7.37 (m, 7H), 7.27–7.20 (m, 4H), 6.92–6.91 (m, 1H), 3.24–3.18 (m, 1H), 2.44–2.37 (m, 1H), 1.91–1.89 (m, 2H), 1.59–1.53 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.4 (d, $J_{C\text{-}P}$ = 22.4 Hz), 143.5 (d, $J_{C\text{-}P}$ = 27.5 Hz), 138.6 (d, $J_{C\text{-}P}$ = 93.7 Hz), 134.0 (d, $J_{C\text{-}P}$ = 15.9 Hz), 132.6, 132.1 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.5 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.8 Hz), 130.5 (d, $J_{C\text{-}P}$ = 97.3 Hz), 128.8, 128.6, 128.5 (2C, overlap), 128.4 (2C, overlap), 123.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 34.6 (d, $J_{C\text{-}P}$ = 10.7 Hz), 28.8 (dd, $J_{C\text{-}P}$ = 68.9, 3.8 Hz), 19.3.^{31}P NMR (162 MHz, $CDCl_3$): δ 39.19. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{24}H_{21}OPH^+$ 357.1403; found 357.1413.

2-Cyclopentyl-1,3-diphenylphosphindole 1-oxide (3al)[5]



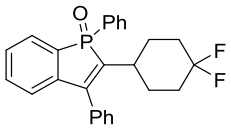
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow solid; yield: 47.4 mg, 64%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.77 (m, 2H), 7.56–7.43 (m, 7H), 7.34–7.24 (m, 4H), 6.93 (d, J = 6.8 Hz, 1H), 2.78–2.71 (m, 1H), 2.13–2.03 (m, 1H), 1.82–1.81 (m, 1H), 1.64–1.63 (m, 1H), 1.45–1.26 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.4 (d, $J_{C\text{-}P}$ = 22.5 Hz), 143.7 (d, $J_{C\text{-}P}$ = 27.3 Hz), 139.3 (d, $J_{C\text{-}P}$ = 92.1 Hz), 134.3 (d, $J_{C\text{-}P}$ = 15.9 Hz), 132.5 (d, $J_{C\text{-}P}$ = 1.4 Hz), 132.2 (d, J = 105.3 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.9 (d, $J_{C\text{-}P}$ = 96.8 Hz), 130.6 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.62 (d, $J_{C\text{-}P}$ = 12.2 Hz), 128.61 (d, $J_{C\text{-}P}$ = 21.9 Hz), 128.3 (d, $J_{C\text{-}P}$ = 5.5 Hz), 128.28, 128.23, 128.1, 122.9 (d, $J_{C\text{-}P}$ = 10.7 Hz), 40.1 (d, $J_{C\text{-}P}$ = 9.7 Hz), 32.5 (dd, $J_{C\text{-}P}$ = 24.5, 2.2 Hz), 24.8 (d, $J_{C\text{-}P}$ = 54.2). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.97.

2-Cyclohexyl-1,3-diphenylphosphindole 1-oxide (3am)[5]



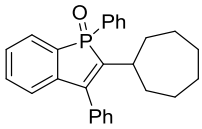
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow solid; yield: 54.5 mg, 71%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81–7.76 (m, 2H), 7.55–7.43 (m, 7H), 7.36–7.24 (m, 4H), 6.91 (d, J = 7.2 Hz, 1H), 2.55–2.40 (m, 1H), 1.91–1.77 (m, 2H), 1.65–1.25 (m, 4H), 1.11–0.85 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7 (d, $J_{C\text{-}P}$ = 22.3 Hz), 143.9 (d, $J_{C\text{-}P}$ = 27.6 Hz), 141.0 (d, $J_{C\text{-}P}$ = 90.7 Hz), 134.3 (d, $J_{C\text{-}P}$ = 15.9 Hz), 132.5 (d, $J_{C\text{-}P}$ = 1.5 Hz), 132.2 (d, $J_{C\text{-}P}$ = 105.1 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.8 Hz), 130.9 (d, $J_{C\text{-}P}$ = 96.7 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.7, 128.59 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.56, 128.4 (d, $J_{C\text{-}P}$ = 10.6 Hz), 128.25 (d, $J_{C\text{-}P}$ = 9.5 Hz), 128.24, 123.0 (d, $J_{C\text{-}P}$ = 10.8 Hz), 39.3 (d, $J_{C\text{-}P}$ = 9.2 Hz), 32.4 (dd, $J_{C\text{-}P}$ = 42.1, 2.2 Hz), 26.1, 25.2. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.28.

2-(4,4-Difluorocyclohexyl)-1,3-diphenylphosphindole 1-oxide (3an)



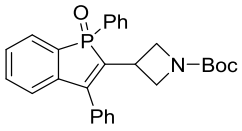
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow solid; yield: 58.8 mg, 70%; mp 176–178 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79–7.75 (m, 2H), 7.57–7.53 (m, 4H), 7.49–7.44 (m, 3H), 7.39–7.29 (m, 4H), 6.93 (d, J = 7.6 Hz, 1H), 2.56–2.54 (m, 1H), 2.20 (dd, J = 26.4, 13.2 Hz, 1H), 2.03 (m, 1H), 1.89–1.80 (m, 2H), 1.52–1.27 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.9 (d, J = 21.8 Hz), 143.6 (d, J = 27.0 Hz), 138.7 (d, J = 91.3 Hz), 133.8 (d, J = 15.4 Hz), 132.7 (d, J = 1.3 Hz), 132.2 (d, J = 94.3 Hz), 132.0 (d, J = 2.7 Hz), 131.7 (d, J = 106.0 Hz), 130.7 (d, J = 10.8 Hz), 128.84 (d, J = 22.4 Hz), 128.83, 128.6 (d, J = 16.5 Hz), 128.3 (d, J = 9.9 Hz), 127.9 (d, J = 10.4 Hz), 123.8 (d, J = 162.0 Hz), 123.3 (d, J = 10.8 Hz), 121.0 (d, J = 241.8 Hz), 37.2 (d, J = 9.4 Hz), 33.7–33.2 (m), 28.6–27.9 (m). ^{19}F NMR (376 MHz, $CDCl_3$): δ -91.05 (d, J = 235.5 Hz), -102.28 (d, J = 235.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.94. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{23}F_2OPH^+$ 421.1527; found 421.1535.

2-Cycloheptyl-1,3-diphenylphosphindole 1-oxide (3ao)[5]



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow solid; yield: 46.2 mg, 58%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79–7.75 (m, 2H), 7.54–7.43 (m, 7H), 7.35–7.28 (m, 4H), 6.92 (d, J = 7.2 Hz, 1H), 2.66–2.60 (m, 1H), 2.01–1.95 (m, 1H), 1.66–1.65 (m, 1H), 1.40–1.20 (m, 8H), 1.18–1.10 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3 (d, $J_{C\text{-}P}$ = 22.5 Hz), 144.1 (d, $J_{C\text{-}P}$ = 27.6 Hz), 142.3 (d, $J_{C\text{-}P}$ = 90.1 Hz), 134.3 (d, $J_{C\text{-}P}$ = 15.8 Hz), 132.5 (d, $J_{C\text{-}P}$ = 1.2 Hz), 132.0 (d, $J_{C\text{-}P}$ = 105.7 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.89 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.88 (d, $J_{C\text{-}P}$ = 96.9 Hz), 128.7, 128.6, 128.5, 128.4, 128.3 (d, $J_{C\text{-}P}$ = 4.7 Hz), 128.2 (d, $J_{C\text{-}P}$ = 7.4 Hz), 123.1 (d, $J_{C\text{-}P}$ = 10.8 Hz), 41.0 (d, $J_{C\text{-}P}$ = 8.8 Hz), 34.2 (dd, $J_{C\text{-}P}$ = 51.4, 1.9 Hz), 27.3 (d, $J_{C\text{-}P}$ = 20.9 Hz), 26.9 (d, $J_{C\text{-}P}$ = 3.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.36.

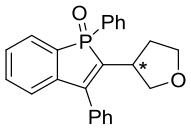
(4-(1-Oxido-1,3-diphenylphosphindol-2-yl)piperidin-1-yl)(phenyl)methanone (3ap)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 26.5 mg, 29%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80–7.75 (m, 2H), 7.65–7.61 (m, 1H), 7.58–7.41 (m, 7H), 7.37–7.28 (m, 3H), 7.07 (d, J = 7.6 Hz, 1H), 4.39–4.01 (m, 1H), 3.90 (m, 1H), 3.72–3.57 (m, 3H), 1.36 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 143.2 (d, $J_{C\text{-}P}$ = 26.2 Hz), 134.7 (d, $J_{C\text{-}P}$ = 94.7 Hz), 133.2 (d, $J_{C\text{-}P}$ = 14.9 Hz), 132.9 (d, $J_{C\text{-}P}$ = 1.0 Hz), 132.4 (d, $J_{C\text{-}P}$ = 1.9 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.9 Hz), 130.0, 129.1, 129.1, 129.06, 129.01, 128.9,

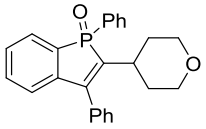
128.8, 128.7, 128.1, 123.5 (d, $J_{C\text{-}P}$ = 10.6 Hz), 79.2, 53.7, 28.2, 28.0 (d, $J_{C\text{-}P}$ = 10.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.29. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{28}H_{28}NO_3PH^+$ 458.1880; found 458.1872.

1,3-Diphenyl-2-(tetrahydrofuran-3-yl)phosphindole 1-oxide (3aq)



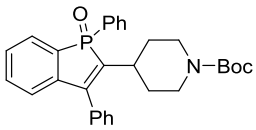
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow viscous liquid; yield: 45.4 mg, 61%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83–7.73 (m, 2H), 7.59–7.45 (m, 7H), 7.41–7.29 (m, 4H), 6.98–6.95 (m, 1H), 4.17–3.80 (m, 2H), 3.68–3.44 (m, 2H), 3.25–3.14 (m, 1H), 2.59–2.50 (m, 0.5H), 2.09–2.04 (m, 0.5H), 1.72–1.66 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.5 (d, $J_{C\text{-}P}$ = 21.6 Hz) [152.2 (d, $J_{C\text{-}P}$ = 21.7 Hz)], 143.4 (d, $J_{C\text{-}P}$ = 5.4 Hz) [143.2 (d, $J_{C\text{-}P}$ = 5.2 Hz)], 135.0 (d, $J_{C\text{-}P}$ = 92.6 Hz) [134.9 (d, $J_{C\text{-}P}$ = 92.8 Hz)], 133.8 (d, $J_{C\text{-}P}$ = 1.3 Hz) [133.6 (d, $J_{C\text{-}P}$ = 1.4 Hz)], 132.76 (132.78), 132.6 (132.4), 132.1 (d, $J_{C\text{-}P}$ = 2.8 Hz) [132.0 (d, $J_{C\text{-}P}$ = 2.9 Hz)], 131.2 (d, $J_{C\text{-}P}$ = 52.3 Hz) [131.1 (d, $J_{C\text{-}P}$ = 49.9 Hz)], 130.7 (d, $J_{C\text{-}P}$ = 10.8 Hz) [130.6 (d, $J_{C\text{-}P}$ = 10.7 Hz)], 130.0 (d, $J_{C\text{-}P}$ = 97.6 Hz) [129.4 (d, $J_{C\text{-}P}$ = 79.2 Hz)], 128.9, 128.8 (d, $J_{C\text{-}P}$ = 7.5 Hz) [128.7 (d, $J_{C\text{-}P}$ = 2.5 Hz)], 128.6, 128.5(128.4), 128.1 123.3 (d, $J_{C\text{-}P}$ = 1.6 Hz) [123.25 (d, $J_{C\text{-}P}$ = 1.7 Hz)], 71.1 (d, $J_{C\text{-}P}$ = 2.3 Hz) [71.0 (d, $J_{C\text{-}P}$ = 2.2 Hz)], 68.0 (67.8), 39.5 (d, $J_{C\text{-}P}$ = 9.2 Hz) [39.2 (d, $J_{C\text{-}P}$ = 9.3 Hz)], 31.8 (d, $J_{C\text{-}P}$ = 2.5 Hz) [31.2 (d, $J_{C\text{-}P}$ = 2.1 Hz)]. ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.44, 38.42. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{24}H_{21}O_2PH^+$ 373.1352; found 373.1355.

1,3-Diphenyl-2-(tetrahydro-2*H*-pyran-4-yl)phosphindole 1-oxide (3ar)



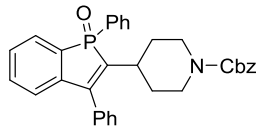
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow wax; yield: 49.5 mg, 64%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81–7.76 (m, 2H), 7.53–7.45 (m, 7H), 7.37–7.29 (m, 4H), 6.93 (d, J = 6.8 Hz, 1H), 3.88 (d, J = 11.2 Hz, 1H), 3.67 (d, J = 11.2 Hz, 1H), 3.17 (t, J = 12.0 Hz, 1H), 3.08 (t, J = 12.0 Hz, 1H), 2.80–2.70 (m, 1H), 2.23 (q, J = 12.4 Hz, 1H), 1.66 (d, J = 13.2 Hz, 1H), 1.41 (q, J = 12.4 Hz, 1H), 1.12 (d, J = 13.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.6 (d, $J_{C\text{-}P}$ = 22.0 Hz), 143.6 (d, $J_{C\text{-}P}$ = 27.1 Hz), 138.7 (d, $J_{C\text{-}P}$ = 91.7 Hz), 133.7 (d, $J_{C\text{-}P}$ = 15.5 Hz), 132.6 (d, $J_{C\text{-}P}$ = 1.2 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.7 (d, $J_{C\text{-}P}$ = 106.0 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.8 Hz), 130.1 (d, $J_{C\text{-}P}$ = 97.5 Hz), 128.8 (d, $J_{C\text{-}P}$ = 13.2 Hz), 128.7, 128.6, 128.4, 128.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.0 (d, $J_{C\text{-}P}$ = 7.9 Hz), 123.2 (d, $J_{C\text{-}P}$ = 10.8 Hz), 67.5 (d, $J_{C\text{-}P}$ = 21.8 Hz), 36.3 (d, $J_{C\text{-}P}$ = 9.5 Hz), 31.7 (dd, $J_{C\text{-}P}$ = 69.4, 2.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.10. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{23}O_2PH^+$ 387.1508; found 387.1523.

***tert*-Butyl 4-(1-oxido-1,3-diphenylphosphindol-2-yl)piperidine-1-carboxylate (3as)**



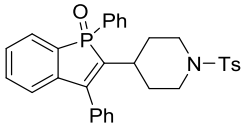
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow solid; yield: 58.2 mg, 60%; mp 150–152 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.74 (m, 2H), 7.53–7.37 (m, 7H), 7.35–7.29 (m, 4H), 6.93 (d, J = 6.8 Hz, 1H), 4.05–3.83 (m, 2H), 2.66–2.56 (m, 1H), 2.45–2.32 (m, 2H), 2.04–1.69 (m, 2H), 1.49–1.45 (m, 2H), 1.40 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.4, 150.8 (d, $J_{C\text{-}P}$ = 22.2 Hz), 143.6 (d, $J_{C\text{-}P}$ = 27.0 Hz), 139.0 (d, $J_{C\text{-}P}$ = 91.2 Hz), 133.8 (d, $J_{C\text{-}P}$ = 15.1 Hz), 132.7, 132.0 (d, $J_{C\text{-}P}$ = 1.2 Hz), 131.9 (d, $J_{C\text{-}P}$ = 105.6 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.8 Hz), 128.89 (d, $J_{C\text{-}P}$ = 13.3 Hz), 128.87, 128.7 (2C, overlap), 128.5, 128.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.1 (d, $J_{C\text{-}P}$ = 6.5 Hz), 123.3 (d, $J_{C\text{-}P}$ = 10.9 Hz), 79.2, 37.5, 31.3, 30.9, 28.2. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.11. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{30}H_{32}NO_3PH^+$ 486.2193; found 486.2195.

Benzyl 4-(1-oxido-1,3-diphenylphosphindol-2-yl)piperidine-1-carboxylate (3at)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow solid; yield: 61.3 mg, 59%; mp 141–143 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.73 (m, 2H), 7.56–7.43 (m, 7H), 7.38–7.27 (m, 9H), 6.92 (d, J = 7.2 Hz, 1H), 5.05 (s, 2H), 4.15–3.98 (m, 2H), 2.66–2.42 (m, 3H), 2.08–2.03 (m, 1H), 1.73–1.70 (m, 1H), 1.34–1.20 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.8, 150.9 (d, $J_{C\text{-}P}$ = 22.1 Hz), 143.6 (d, $J_{C\text{-}P}$ = 26.9 Hz), 138.8 (d, $J_{C\text{-}P}$ = 91.8 Hz), 136.6, 133.8 (d, $J_{C\text{-}P}$ = 15.2 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.5 Hz), 132.03, 132.02 (d, $J_{C\text{-}P}$ = 106.1 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.7 Hz), 128.9, 128.8, 128.7, 128.5, 128.38 (d, $J_{C\text{-}P}$ = 10.1 Hz), 128.30, 128.1, 128.0, 127.77, 127.70, 123.3 (d, $J_{C\text{-}P}$ = 10.8 Hz), 66.8, 43.7 (d, $J_{C\text{-}P}$ = 14.6 Hz), 31.2, 30.9 (d, $J_{C\text{-}P}$ = 1.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.08. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{33}H_{30}NO_3PH^+$ 520.2036; found 520.2034.

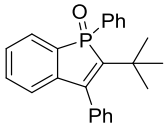
1,3-Diphenyl-2-(1-tosylpiperidin-4-yl)phosphindole 1-oxide (3au)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a white solid; yield: 53.9 mg, 50%; mp 100–103 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.71 (m, 2H), 7.56–7.43 (m, 9H), 7.38–7.34 (m, 1H), 7.28–7.21 (m, 5H), 6.87 (d, J = 7.2 Hz, 1H), 3.74 (d, J = 11.2 Hz, 1H), 3.54 (d, J = 11.2 Hz, 1H), 2.39 (s, 3H), 2.31–2.17 (m, 2H), 2.00–1.77 (m, 3H), 1.44–1.35 (m, 1H), 1.29–1.25 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.1 (d, $J_{C\text{-}P}$ = 21.7 Hz), 143.6 (d, $J_{C\text{-}P}$ = 27.0 Hz), 143.2, 138.3 (d, $J_{C\text{-}P}$ = 91.8 Hz), 133.8 (d, $J_{C\text{-}P}$ = 15.5 Hz), 132.80 (d, $J_{C\text{-}P}$ = 4.6 Hz), 132.80, 132.1 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.8 (d, $J_{C\text{-}P}$ = 105.9 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.8 Hz), 130.06, 130.02 (d, $J_{C\text{-}P}$ = 97.3 Hz), 129.4, 128.9 (d, $J_{C\text{-}P}$ = 12.2 Hz), 128.8 (d, $J_{C\text{-}P}$ = 22.0 Hz), 128.4 (d, $J_{C\text{-}P}$ =

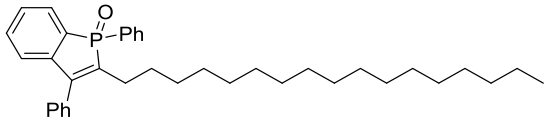
9.9 Hz), 128.0 (d, $J_{C\text{-}P}$ = 7.3 Hz), 127.5, 123.9, 123.3 (d, $J_{C\text{-}P}$ = 10.9 Hz), 46.1 (d, $J_{C\text{-}P}$ = 12.9 Hz), 36.6 (d, $J_{C\text{-}P}$ = 9.5 Hz), 30.5 (dd, $J_{C\text{-}P}$ = 79.0, 2.0 Hz), 21.3. ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.92. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{32}H_{30}NO_3PSH^+$ 540.1757; found 540.1754.

2-(*tert*-Butyl)-1,3-diphenylphosphindole 1-oxide (3av)



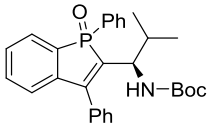
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow gum; yield: 28.7 mg, 40%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83–7.79 (m, 2H), 7.49–7.43 (m, 7H), 7.29–7.22 (m, 4H), 6.59 (d, J = 7.2 Hz, 1H), 1.07 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2 (d, $J_{C\text{-}P}$ = 22.5 Hz), 145.2 (d, $J_{C\text{-}P}$ = 28.5 Hz), 143.8 (d, $J_{C\text{-}P}$ = 87.6 Hz), 136.4 (d, $J_{C\text{-}P}$ = 17.2 Hz), 132.5 (d, $J_{C\text{-}P}$ = 1.7 Hz), 131.9, 131.6 (d, $J_{C\text{-}P}$ = 2.8 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.8 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.6 Hz), 128.65 (d, $J_{C\text{-}P}$ = 12.1 Hz), 128.62 (d, $J_{C\text{-}P}$ = 11.9 Hz), 128.48 (d, $J_{C\text{-}P}$ = 10.4 Hz), 128.46, 128.0 (d, $J_{C\text{-}P}$ = 40.3 Hz), 127.7 (d, $J_{C\text{-}P}$ = 11.4 Hz), 123.0 (d, $J_{C\text{-}P}$ = 11.2 Hz), 35.6 (d, $J_{C\text{-}P}$ = 9.6 Hz), 31.4 (d, $J_{C\text{-}P}$ = 4.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 41.22. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{24}H_{23}OP$ 359.1559; found 359.1554.

2-Heptadecyl-1,3-diphenylphosphindole 1-oxide (3ax)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 62.7 mg, 58%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79–7.74 (m, 2H), 7.64–7.59 (m, 1H), 7.54–7.48 (m, 3H), 7.45–7.36 (m, 4H), 7.33–7.29 (m, 3H), 7.02 (d, J = 7.2 Hz, 1H), 2.50–2.39 (m, 1H), 2.27–2.15 (m, 1H), 1.42–0.93 (m, 30H), 0.87 (t, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2 (d, $J_{C\text{-}P}$ = 22.2 Hz), 144.2 (d, $J_{C\text{-}P}$ = 28.1 Hz), 136.7 (d, $J_{C\text{-}P}$ = 93.5 Hz), 133.8, 133.7, 132.6 (d, $J_{C\text{-}P}$ = 1.5 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.6 (d, $J_{C\text{-}P}$ = 105.0 Hz), 130.8 (d, $J_{C\text{-}P}$ = 10.7 Hz), 129.9(d, $J_{C\text{-}P}$ = 97.6 Hz), 128.7, 128.6, 128.5 (d, $J_{C\text{-}P}$ = 24.6 Hz), 128.3, 123.1 (d, $J_{C\text{-}P}$ = 2.1 Hz), 123.0, 31.7, 29.55 (2C, overlap), 29.51, 29.46, 29.40, 29.3, 29.2, 28.8, 28.5 (2C, overlap), 26.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 22.5, 13.9. ^{31}P NMR (162 MHz, $CDCl_3$): δ 40.06. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{37}H_{49}OPH^+$ 541.3594; found 541.3592.

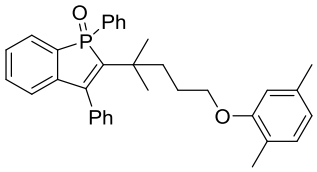
***tert*-Butyl ((1*R*)-2-methyl-1-(1-oxido-1,3-diphenylphosphindol-2-yl)propyl)carbamate (3ay)**



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a colorless viscous liquid; yield: 30.3 mg, 32%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 2H), 7.69 (br, 1H, -NH), 7.54–7.49 (m, 4H), 7.47–7.37 (m, 5H), 7.31–7.27 (m, 2H), 7.04 (d, J = 7.6 Hz, 1H), 4.83 (d, J = 9.4 Hz, 1H), 4.57 (dt, J = 17.4, 8.6 Hz, 1H), 1.15 (s, 9H),

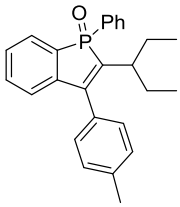
0.88 (d, J = 6.4 Hz, 3H), 0.74 (d, J = 6.4 Hz, 3H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.7, 151.2 (d, $J_{C\text{-}P}$ = 21.0 Hz), 143.2 (d, $J_{C\text{-}P}$ = 27.6 Hz), 137.5, 136.6, 133.3 (d, $J_{C\text{-}P}$ = 15.5 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.2 Hz), 132.2 (d, $J_{C\text{-}P}$ = 105.6 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.7 (d, $J_{C\text{-}P}$ = 99.9 Hz), 130.5 (d, $J_{C\text{-}P}$ = 10.8 Hz), 128.9, 128.8, 128.7, 128.6, 128.4 (d, $J_{C\text{-}P}$ = 9.9 Hz), 123.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 78.6, 55.4 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.4, 28.0, 19.0 (d, $J_{C\text{-}P}$ = 162.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.29. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{29}H_{32}NO_3PH^+$ 474.2193; found 474.2180.

2-(5-(2,5-Dimethylphenoxy)-2-methylpentan-2-yl)-1,3-diphenylphosphindole 1-oxide (3az)



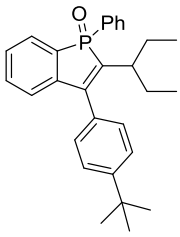
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a colorless viscous liquid; yield: 40.5 mg, 40%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.77 (m, 2H), 7.52–7.44 (m, 7H), 7.32–7.25 (m, 4H), 6.97 (d, J = 7.6 Hz, 1H), 6.63 (d, J = 7.2 Hz, 1H), 6.58 (d, J = 7.6 Hz, 1H), 6.53 (s, 1H), 3.72–3.63 (m, 2H), 2.28 (s, 3H), 2.11 (s, 3H), 1.81–1.70 (m, 1H), 1.66–1.58 (m, 1H), 1.50–1.46 (m, 2H), 1.17 (s, 3H), 1.05 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 150.6 (d, $J_{C\text{-}P}$ = 22.8 Hz), 145.3 (d, $J_{C\text{-}P}$ = 28.4 Hz), 143.0 (d, $J_{C\text{-}P}$ = 87.2 Hz), 136.4, 136.3, 132.7, 131.9 (d, $J_{C\text{-}P}$ = 5.8 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.0, 130.5 (d, $J_{C\text{-}P}$ = 80.4 Hz), 128.7, 128.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.59, 128.56, 128.2, 128.0, 127.9 (d, $J_{C\text{-}P}$ = 9.8 Hz), 123.4, 123.1 (d, $J_{C\text{-}P}$ = 11.1 Hz), 120.4, 111.9, 68.0, 39.5 (d, $J_{C\text{-}P}$ = 3.5 Hz), 39.0 (d, $J_{C\text{-}P}$ = 9.3 Hz), 29.4 (dd, $J_{C\text{-}P}$ = 22.6, 4.9 Hz), 25.1, 21.3, 15.7. ^{31}P NMR (162 MHz, $CDCl_3$): δ 41.27. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{34}H_{35}O_2P$ 507.2447; found 507.2449.

2-(Pentan-3-yl)-1-phenyl-3-(p-tolyl)phosphindole 1-oxide (3ba)



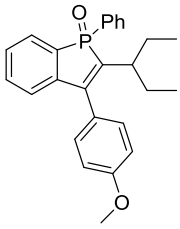
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow solid; yield: 54.8 mg, 71%; mp 151–154 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.71 (m, 2H), 7.58–7.48 (m, 2H), 7.42–7.36 (m, 3H), 7.28 (m, 3H), 7.20–7.12 (m, 2H), 6.93 (d, J = 7.6 Hz, 1H), 2.42 (s, 3H), 2.36–2.34 (m, 1H), 1.99–1.88 (m, 1H), 1.58–1.52 (m, 1H), 1.26–1.17 (m, 1H), 1.11–1.00 (m, 1H), 0.88 (t, J = 7.2 Hz, 3H), 0.44 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.6 (d, $J_{C\text{-}P}$ = 22.4 Hz), 144.4 (d, $J_{C\text{-}P}$ = 27.6 Hz), 139.7 (d, $J_{C\text{-}P}$ = 89.4 Hz), 137.8, 132.6 (d, $J_{C\text{-}P}$ = 1.6 Hz), 132.1 (d, $J_{C\text{-}P}$ = 73.5 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.4, 131.0 (d, $J_{C\text{-}P}$ = 48.6 Hz), 131.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 129.2 (d, $J_{C\text{-}P}$ = 20.6 Hz), 128.5 (d, $J_{C\text{-}P}$ = 17.2 Hz), 128.4, 128.36, 128.33, 123.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 44.1 (d, $J_{C\text{-}P}$ = 9.2 Hz), 27.4 (dd, $J_{C\text{-}P}$ = 72.5, 1.8 Hz), 21.2, 12.5 (d, $J_{C\text{-}P}$ = 52.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.12. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{27}OPH^+$ 387.1872; found 387.1856.

3-(4-(*tert*-Butyl)phenyl)-2-(pentan-3-yl)-1-phenylphosphindole 1-oxide (3ca)



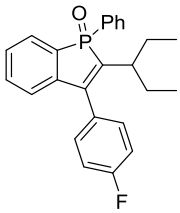
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow solid; yield: 63.4 mg, 74%; mp 153–155 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67–7.62 (m, 2H), 7.48–7.37 (m, 4H), 7.32–7.26 (m, 3H), 7.19–7.13 (m, 2H), 7.08–7.06 (m, 1H), 6.86 (d, J = 7.6 Hz, 1H), 2.34–2.28 (m, 1H), 1.93–1.80 (m, 1H), 1.47–1.44 (m, 1H), 1.28 (s, 9H), 1.16–1.08 (m, 1H), 1.00–0.92 (m, 1H), 0.79 (t, J = 7.2 Hz, 3H), 0.34 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.6 (d, $J_{C\text{-}P}$ = 22.4 Hz), 150.9, 144.4 (d, $J_{C\text{-}P}$ = 27.7 Hz), 139.6 (d, $J_{C\text{-}P}$ = 89.5 Hz), 133.7, 132.6 (d, $J_{C\text{-}P}$ = 1.4 Hz), 132.1 (d, $J_{C\text{-}P}$ = 69.2 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.3 (d, $J_{C\text{-}P}$ = 9.1 Hz), 131.04 (d, $J_{C\text{-}P}$ = 10.7 Hz), 131.01 (d, $J_{C\text{-}P}$ = 35.7 Hz), 128.4 (d, $J_{C\text{-}P}$ = 7.0 Hz), 128.3 ($J_{C\text{-}P}$ = 9.3 Hz), 128.2 (d, $J_{C\text{-}P}$ = 7.2 Hz), 125.3 (d, $J_{C\text{-}P}$ = 23.3 Hz), 123.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 44.0 (d, $J_{C\text{-}P}$ = 9.3 Hz), 34.5, 31.2, 27.4 (dd, $J_{C\text{-}P}$ = 70.9, 1.3 Hz), 12.4 (d, $J_{C\text{-}P}$ = 54.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.21. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{29}H_{33}OPH^+$ 429.2342; found 429.2348.

3-(4-Methoxyphenyl)-2-(pentan-3-yl)-1-phenylphosphindole 1-oxide (3da)



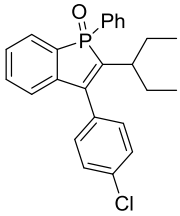
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 53.9 mg, 67%; mp 145–148 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.71 (m, 2H), 7.57–7.47 (m, 2H), 7.42–7.39 (m, 3H), 7.30–7.16 (m, 3H), 7.04–7.02 (m, 2H), 6.96 (d, J = 7.6 Hz, 1H), 3.86 (s, 3H), 2.43–2.37 (m, 1H), 2.00–1.89 (m, 1H), 1.59–1.52 (m, 1H), 1.25–1.18 (m, 1H), 1.10–1.02 (m, 1H), 0.87 (t, J = 6.8Hz, 3H), 0.44 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ159.2, 151.2 (d, $J_{C\text{-}P}$ = 22.6 Hz), 144.4 (d, $J_{C\text{-}P}$ = 27.6 Hz), 139.7 (d, $J_{C\text{-}P}$ = 89.4 Hz), 132.6 (d, $J_{C\text{-}P}$ = 1.2 Hz), 132.1 (d, $J_{C\text{-}P}$ = 72.9 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.1 (d, $J_{C\text{-}P}$ = 64.5 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.6 Hz), 129.9, 129.7, 128.37 (d, $J_{C\text{-}P}$ = 11.7 Hz), 128.31 (d, $J_{C\text{-}P}$ = 9.7 Hz), 126.4 (d, $J_{C\text{-}P}$ = 16.0 Hz), 122.9 (d, $J_{C\text{-}P}$ = 11.0 Hz), 113.9, 55.1, 44.0 (d, J $J_{C\text{-}P}$ = 9.3 Hz), 27.3 (dd, $J_{C\text{-}P}$ = 71.4, 1.4 Hz), 12.4 (d, $J_{C\text{-}P}$ = 51.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.96. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{27}O_2PH^+$ 403.1821; found 403.1823.

3-(4-Fluorophenyl)-2-(pentan-3-yl)-1-phenylphosphindole 1-oxide (3ea)



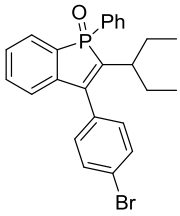
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a white solid; yield: 56.2 mg, 72%; mp 154–157 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.71 (m, 2H), 7.60–7.56 (m, 1H), 7.53–7.49 (m, 1H), 7.43–7.40 (m, 3H), 7.30–7.18 (m, 5H), 6.90 (d, J = 7.2 Hz, 1H), 2.36–2.29 (m, 1H), 2.00–2.89 (m, 1H), 1.60–1.51 (m, 1H), 1.26–1.19 (m, 1H), 1.11–1.00 (m, 1H), 0.87 (t, J = 7.2 Hz, 3H), 0.43 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.4 (d, J_{F-P} = 247.9 Hz), 150.4 (d, J_{C-P} = 22.9 Hz), 144.1 (d, J_{C-P} = 27.4 Hz), 140.6 (d, J_{C-P} = 88.8 Hz), 133.0 (d, J_{C-P} = 138.8 Hz), 132.8 (d, J_{C-P} = 1.6 Hz), 131.9 (d, J_{C-P} = 2.8 Hz), 131.4 (d, J_{C-P} = 15.3 Hz), 131.0 (d, J_{C-P} = 10.7 Hz), 130.5, 130.5 (d, J_{C-P} = 21.1 Hz), 130.2 (dd, $J_{C-P/C-F}$ = 12.8, 3.1 Hz), 128.6 (dd, $J_{C-P/C-F}$ = 10.0, 7.8 Hz), 128.4 (J_{C-P} = 12.1 Hz), 122.8 (d, J_{C-P} = 10.8 Hz), 115.7 (t, J_{C-F} = 22.6 Hz), 44.2 (d, J_{C-P} = 9.1 Hz), 27.3 (d, J_{C-P} = 72.4, 1.6 Hz), 12.5 (d, J_{C-P} = 53.7 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ -113.05. ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.99. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{24}FOPH^+$ 391.1622; found 391.1629.

3-(4-Chlorophenyl)-2-(pentan-3-yl)-1-phenylphosphindole 1-oxide (3fa)



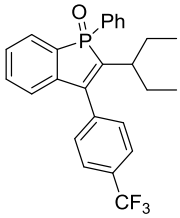
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a pale yellow solid; yield: 61.7 mg, 76%; mp 156–158 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75–7.71 (m, 2H), 7.76–7.57 (m, 1H), 7.50–7.42 (m, 6H), 7.31–7.25 (m, 2H), 7.19 (d, J = 7.6 Hz, 1H), 6.89 (d, J = 7.2 Hz, 1H), 2.36–2.27 (m, 1H), 1.99–1.88 (m, 1H), 1.60–1.53 (m, 1H), 1.28–1.19 (m, 1H), 1.10–1.01 (m, 1H), 0.87 (t, J = 6.4 Hz, 3H), 0.43 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2 (d, J_{C-P} = 22.8 Hz), 143.9 (d, J_{C-P} = 27.4 Hz), 140.8 (d, J_{C-P} = 88.9 Hz), 134.0 (d, J_{C-P} = 22.9 Hz), 132.8 (d, J_{C-P} = 1.8 Hz), 132.6 (d, J_{C-P} = 66.7 Hz), 131.9 (d, J_{C-P} = 2.8 Hz), 131.0 (d, J_{C-P} = 10.8 Hz), 130.9 (d, J_{C-P} = 97.2 Hz), 130.1 (d, J_{C-P} = 20.6 Hz), 128.9 (d, J_{C-P} = 22.7 Hz), 128.7 (d, J_{C-P} = 10.7 Hz), 128.6 (d, J_{C-P} = 10.9 Hz), 128.5 (d, J_{C-P} = 12.3 Hz), 123.2, 122.8 (d, J_{C-P} = 10.9 Hz), 44.3 (d, J_{C-P} = 9.0 Hz), 27.4 (dd, J_{C-P} = 72.5, 1.5 Hz), 12.5 (d, J_{C-P} = 54.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.16. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{24}ClOPH^+$ 407.1326; found 407.1324.

3-(4-Bromophenyl)-2-(pentan-3-yl)-1-phenylphosphindole 1-oxide (3ga)



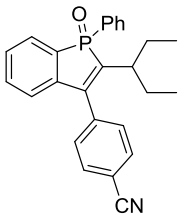
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 63.0 mg, 70%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65–7.60 (m, 2H), 7.56–7.39 (m, 4H), 7.31–7.29 (m, 3H), 7.21 (m, 1H), 7.10 (d, J = 7.6 Hz, 1H), 7.04 (d, J = 7.6 Hz, 1H), 6.79 (d, J = 7.6 Hz, 1H), 2.26–2.21 (m, 1H), 1.90–1.79 (m, 1H), 1.51–1.41 (m, 1H), 1.16–1.09 (m, 1H), 1.01–0.90 (m, 1H), 0.77 (t, J = 7.2 Hz, 3H), 0.33 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.0 (d, $J_{C\text{-}P}$ = 22.8 Hz), 143.7 (d, $J_{C\text{-}P}$ = 27.3 Hz), 140.7 (d, $J_{C\text{-}P}$ = 88.6 Hz), 133.3 (d, $J_{C\text{-}P}$ = 16.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.6 Hz), 132.3, 131.8, 131.7 (d, $J_{C\text{-}P}$ = 15.1 Hz), 131.3 (d, $J_{C\text{-}P}$ = 8.0 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.3 (d, $J_{C\text{-}P}$ = 15.9 Hz), 128.6, 128.48, 128.47 (d, $J_{C\text{-}P}$ = 21.0 Hz), 122.7 (d, $J_{C\text{-}P}$ = 10.7 Hz), 122.2, 44.2 (d, $J_{C\text{-}P}$ = 9.1 Hz), 27.3 (dd, $J_{C\text{-}P}$ = 73.2, 1.6 Hz), 12.4 (d, $J_{C\text{-}P}$ = 54.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.86. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{25}H_{24}BrOPH^+$ 451.0821; found 451.0810.

2-(Pentan-3-yl)-1-phenyl-3-(4-(trifluoromethyl)phenyl)phosphindole 1-oxide (3ha)



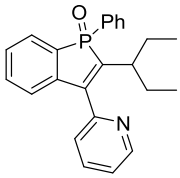
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow solid; yield: 60.7 mg, 69%; mp 167–169 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80–7.72 (m, 4H), 7.62–7.57 (m, 1H), 7.54–7.38 (m, 6H), 7.35–7.29 (m, 1H), 6.85 (d, J = 7.2 Hz, 1H), 2.36–2.22 (m, 1H), 2.03–1.91 (m, 1H), 1.63–1.53 (m, 1H), 1.27–1.20 (m, 1H), 1.12–1.00 (m, 1H), 0.89 (t, J = 7.2Hz, 3H), 0.44 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9 (d, $J_{C\text{-}P}$ = 23.0 Hz), 143.6 (d, $J_{C\text{-}P}$ = 27.2 Hz), 141.3 (d, $J_{C\text{-}P}$ = 88.4 Hz), 138.4 (d, $J_{C\text{-}P}$ = 16.1 Hz), 132.9 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.40, 132.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.3, 131.0 (d, $J_{C\text{-}P}$ = 10.8 Hz), 130.5–130.2 (m, 1C), 129.2 (d, $J_{C\text{-}P}$ = 19.6 Hz), 128.79 (d, $J_{C\text{-}P}$ = 20.4 Hz), 128.79, 128.5 (d, $J_{C\text{-}P}$ = 12.2 Hz), 125.8–125.6 (m, 1C), 125.5 (d, $J_{C\text{-}P}$ = 3.6 Hz), 122.7 (d, $J_{C\text{-}P}$ = 10.8 Hz), 44.4 (d, $J_{C\text{-}P}$ = 9.0 Hz), 27.4 (dd, $J_{C\text{-}P}$ = 72.5, 1.7 Hz), 12.5 (d, $J_{C\text{-}P}$ = 56.6 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.61. ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.90. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{24}F_3OPH^+$ 441.1590; found 441.1584.

4-(1-Oxido-2-(pentan-3-yl)-1-phenylphosphindol-3-yl)benzonitrile (3ia)



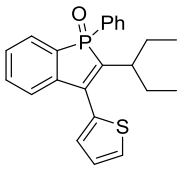
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 51.6 mg, 65%; mp 158–160 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85–7.80 (m, 2H), 7.76–7.71 (m, 2H), 7.65–7.52 (m, 3H), 7.45–7.35 (m, 5H), 6.84 (d, J = 7.6 Hz, 1H), 2.28–2.21 (m, 1H), 2.01–1.90 (m, 1H), 1.63–1.54 (m, 1H), 1.25–1.20 (m, 1H), 1.11–1.00 (m, 1H), 0.87 (t, J = 7.2Hz, 3H), 0.43 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.3 (d, $J_{C\text{-}P}$ = 23.1 Hz), 143.2 (d, $J_{C\text{-}P}$ = 27.1 Hz), 141.7 (d, $J_{C\text{-}P}$ = 87.9 Hz), 139.5 (d, $J_{C\text{-}P}$ = 16.1 Hz), 132.9 (d, $J_{C\text{-}P}$ = 1.7 Hz), 132.4 (d, $J_{C\text{-}P}$ = 16.0 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.7 (d, $J_{C\text{-}P}$ = 9.4 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.6 (d, $J_{C\text{-}P}$ = 114.0 Hz), 129.6 (d, $J_{C\text{-}P}$ = 16.4 Hz), 128.9 (d, $J_{C\text{-}P}$ = 10.4 Hz), 128.8 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.4 Hz), 122.5 (d, $J_{C\text{-}P}$ = 10.7 Hz), 118.2, 112.1, 44.50 (d, $J_{C\text{-}P}$ = 8.9 Hz), 27.3 (dd, $J_{C\text{-}P}$ = 73.5, 1.5 Hz), 12.50 (d, $J_{C\text{-}P}$ = 57.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.89. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{26}H_{24}NOPH^+$ 398.1668; found 398.1647.

2-(Pentan-3-yl)-1-phenyl-3-(pyridin-2-yl)phosphindole 1-oxide (3ja)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 47.7 mg, 64%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.79 (d, J = 4.8 Hz, 1H), 7.83–7.73 (m, 3H), 7.57–7.53 (m, 1H), 7.50–7.46 (m, 1H), 7.40–7.32 (m, 5H), 7.28–7.27 (m, 1H), 6.97 (d, J = 7.2 Hz, 1H), 2.43–2.30 (m, 1H), 1.99–1.87 (m, 1H), 1.61–1.51 (m, 1H), 1.23–1.14 (m, 1H), 1.07–0.99 (m, 1H), 0.89 (t, J = 7.2Hz, 3H), 0.41 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.0 (d, $J_{C\text{-}P}$ = 17.9 Hz), 149.9, 149.5 (d, $J_{C\text{-}P}$ = 22.1 Hz), 143.3 (d, $J_{C\text{-}P}$ = 27.4 Hz), 141.5 (d, $J_{C\text{-}P}$ = 88.0 Hz), 136.5, 132.7 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.4, 131.8 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.3, 131.1 (d, $J_{C\text{-}P}$ = 10.8 Hz), 130.3, 128.5 (d, $J_{C\text{-}P}$ = 10.2 Hz), 128.4 (d, $J_{C\text{-}P}$ = 12.3 Hz), 124.3, 123.0 (d, $J_{C\text{-}P}$ = 10.8 Hz), 122.8, 44.2 (d, $J_{C\text{-}P}$ = 8.9 Hz), 27.2 (dd, $J_{C\text{-}P}$ = 86.1, 1.9 Hz), 12.5 (d, $J_{C\text{-}P}$ = 66.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.61. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{24}H_{24}NOPH^+$ 374.1668; found 374.1665.

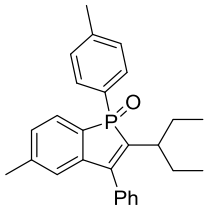
2-(Pentan-3-yl)-1-phenyl-3-(thiophen-2-yl)phosphindole 1-oxide (3ka)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 30.2 mg, 40%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47–7.69 (m, 2H), 7.58–7.54 (m, 1H), 7.52–7.48 (m, 2H), 7.44–7.39 (m, 3H), 7.32–7.27 (m, 2H), 7.07–7.04 (m, 2H), 2.52–2.39 (m, 1H), 1.99–1.88 (m, 1H), 1.62–1.52 (m, 1H), 1.27–1.20 (m, 1H), 1.09–1.01 (m, 1H), 0.87 (t, J = 7.2Hz, 3H), 0.45 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8 (d, $J_{C\text{-}P}$ = 23.7 Hz), 144.1 (d, $J_{C\text{-}P}$ = 27.2 Hz), 141.0 (d, $J_{C\text{-}P}$ = 89.6 Hz), 134.3 (d, $J_{C\text{-}P}$ = 16.7 Hz), 132.8 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.0 (d, $J_{C\text{-}P}$ = 80.2 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.1 (d, $J_{C\text{-}P}$ = 10.7 Hz), 131.0 (d, $J_{C\text{-}P}$ = 72.1 Hz), 128.5 (d, $J_{C\text{-}P}$ = 18.4 Hz), 128.5 (2C, overlap), 128.2 (d, $J_{C\text{-}P}$ = 27.8 Hz), 126.1, 124.2, 122.8 (d, $J_{C\text{-}P}$ = 10.8

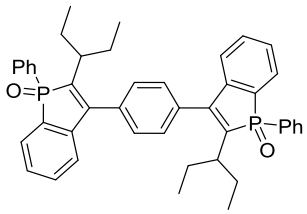
Hz), 44.4 (d, $J_{C\text{-}P}$ = 9.0 Hz), 27.4 (dd, $J_{C\text{-}P}$ = 70.6, 1.8 Hz), 12.5 (d, $J_{C\text{-}P}$ = 53.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 38.74. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{23}H_{23}OPSH^+$ 379.1280; found 379.1251.

5-Methyl-2-(pentan-3-yl)-3-phenyl-1-(*p*-tolyl)phosphindole 1-oxide (3na)



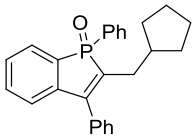
Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (2:1) to afford a yellow gum; yield: 39.2 mg, 49%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63–7.58 (m, 2H), 7.49–7.39 (m, 4H), 7.29–7.28 (m, 1H), 7.21–7.19 (m, 3H), 7.08–7.07 (m, 1H), 6.66 (m, 1H), 2.42–2.21 [m, 7H, including 2.36 (s, 3H) and 2.27 (s, 3H)], 1.94–1.87 (m, 1H), 1.55–1.49 (m, 1H), 1.20–1.15 (m, 1H), 1.06–1.01 (m, 1H), 0.85 (t, J = 7.2Hz, 3H), 0.42 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.3 (d, $J_{C\text{-}P}$ = 22.6 Hz), 144.7 (d, $J_{C\text{-}P}$ = 27.9 Hz), 143.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 142.2 (d, $J_{C\text{-}P}$ = 2.8 Hz), 140.4 (d, $J_{C\text{-}P}$ = 89.0 Hz), 134.7 (d, $J_{C\text{-}P}$ = 15.8 Hz), 131.1 (d, $J_{C\text{-}P}$ = 11.1 Hz), 129.2 (d, $J_{C\text{-}P}$ = 12.6 Hz), 129.09 (d, $J_{C\text{-}P}$ = 71.2 Hz), 129.07 (d, $J_{C\text{-}P}$ = 10.9 Hz), 128.8, 128.6, 128.45 (d, $J_{C\text{-}P}$ = 10.2 Hz), 128.45, 128.0, 123.9 (d, $J_{C\text{-}P}$ = 11.2 Hz), 44.2 (d, $J_{C\text{-}P}$ = 9.2 Hz), 27.4 (d, $J_{C\text{-}P}$ = 74.1, 1.6 Hz), 21.8, 21.5, 12.6 (d, $J_{C\text{-}P}$ = 50.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.38. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{27}H_{29}OPH^+$ 401.2029; found 401.2033.

3,3'-(1,4-Phenylene)bis(2-(pentan-3-yl)-1-phenylphosphindole 1-oxide) (3oa)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (1:1) to afford a yellow solid; yield: 66.3 mg, 50%; mp 133–136 ℃. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74–7.70 (m, 4H), 7.57–7.55 (m, 2H), 7.49–7.31 (m, 14H), 7.02–6.92 (m, 2H), 2.45–2.30 (m, 2H), 2.00–1.95 (m, 2H), 1.66–1.57 (m, 2H), 1.22–1.07 (m, 4H), 0.90–0.85 (m, 6H), 0.45–0.40 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.7 (d, $J_{C\text{-}P}$ = 22.2 Hz), 144.0 (d, $J_{C\text{-}P}$ = 27.8 Hz), 134.5 (d, $J_{C\text{-}P}$ = 16.6 Hz), 132.8, 132.0 (d, $J_{C\text{-}P}$ = 104.8 Hz), 131.9, 131.0 (d, $J_{C\text{-}P}$ = 10.7 Hz), 130.5, 129.2–128.9 (m, 1C), 128.76 (d, $J_{C\text{-}P}$ = 7.9 Hz), 128.70, 128.6, 128.4, 122.8 (d, $J_{C\text{-}P}$ = 11.3 Hz), 44.4 (d, $J_{C\text{-}P}$ = 9.0 Hz), 27.5 (d, $J_{C\text{-}P}$ = 68.3 Hz), 12.6 (d, $J_{C\text{-}P}$ = 55.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.13. HRMS (EI) m/z: $[M+H]^+$ calcd. for $C_{44}H_{44}O_2P_2H^+$ 666.2889; found 666.2834.

2-(Cyclopentylmethyl)-1,3-diphenylphosphindole 1-oxide (3aza)



Following general procedure, the crude product was purified by column chromatography on silica gel and eluted with petroleum/ethyl acetate (1:1) to afford a yellow gum; yield: 38.4 mg, 50%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85–7.73 (m, 2H), 7.63–7.59 (m, 1H), 7.54–7.48 (m, 3H), 7.45–7.36 (m, 4H), 7.30–7.28 (m, 3H), 6.98 (d, J = 7.6 Hz, 1H), 2.50–2.41 (m, 1H), 2.27–2.18 (m, 1H), 2.06–1.98 (m, 1H), 1.65–1.64 (m, 1H), 1.33–1.23 (m, 5H), 0.95–0.85 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.4 (d, $J_{C\text{-}P}$ = 22.2 Hz), 144.3 (d, $J_{C\text{-}P}$ = 28.0 Hz), 136.5 (d, $J_{C\text{-}P}$ = 92.9 Hz), 133.9 (d, $J_{C\text{-}P}$ = 15.8 Hz), 132.7 (d, $J_{C\text{-}P}$ = 1.5 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.6 (d, $J_{C\text{-}P}$ = 105.0 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10.6 Hz), 130.0 (d, $J_{C\text{-}P}$ = 97.4 Hz), 128.7, 128.6, 128.59, 128.54, 128.3 (d, $J_{C\text{-}P}$ = 10.5 Hz), 128.2, 123.1 (d, $J_{C\text{-}P}$ = 10.8 Hz), 38.6 (d, $J_{C\text{-}P}$ = 1.3 Hz), 32.4, 32.3, 24.5 (d, $J_{C\text{-}P}$ = 5.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 40.00. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{26}H_{25}OP$ 385.1716; found 385.1724.

6. References

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3. (a) Wozniak, Ł.; Murphy, J. J.; Melchiorre, P. *J. Am. Chem. Soc.* **2015**, *137*, 5678. (b) Tlahuext-Aca, A.; Candish, L.; Garza-Sanchez, R. A.; Glorius, F. *ACS Catal.* **2018**, *8*, 1715. (c) Wegner, E. E.; Adamson, A. W. *J. Am. Chem. Soc.* **1966**, *88*, 394.
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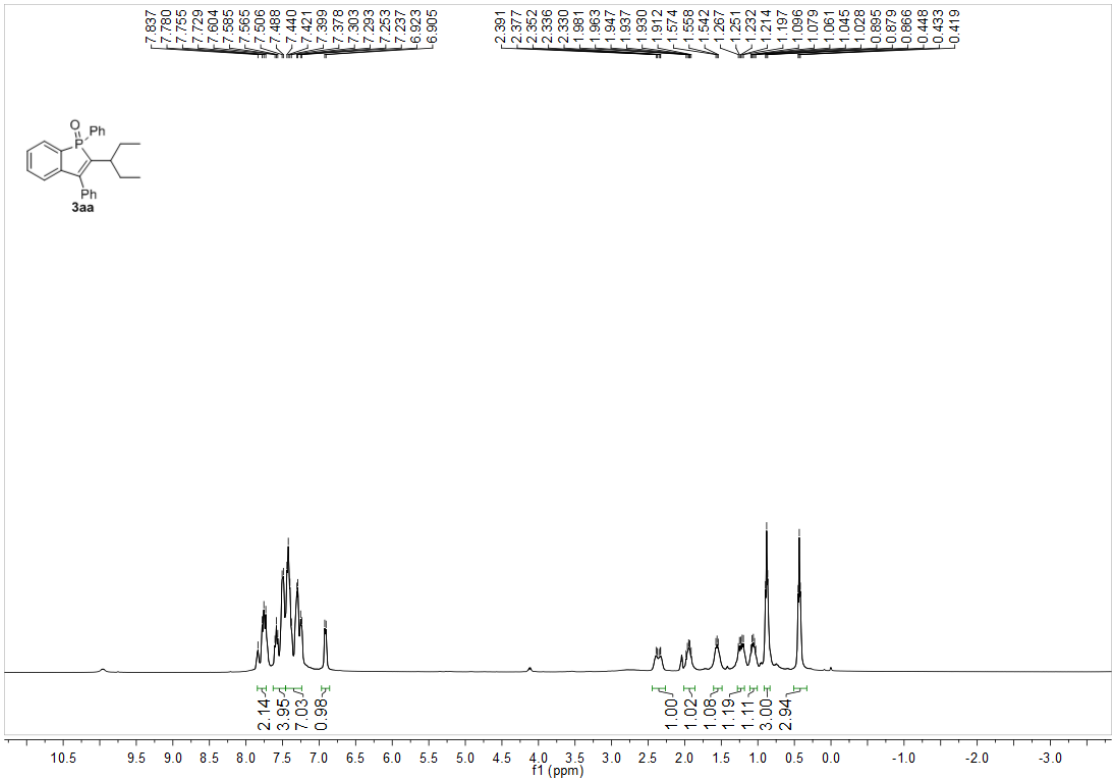
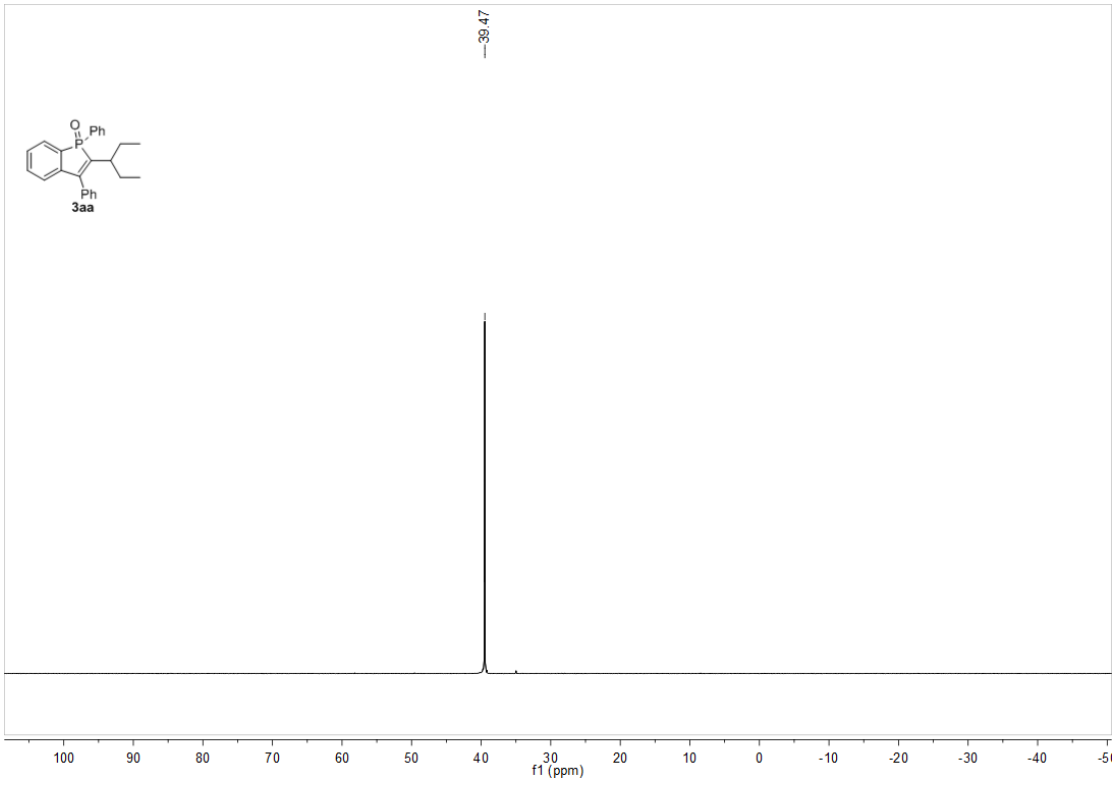
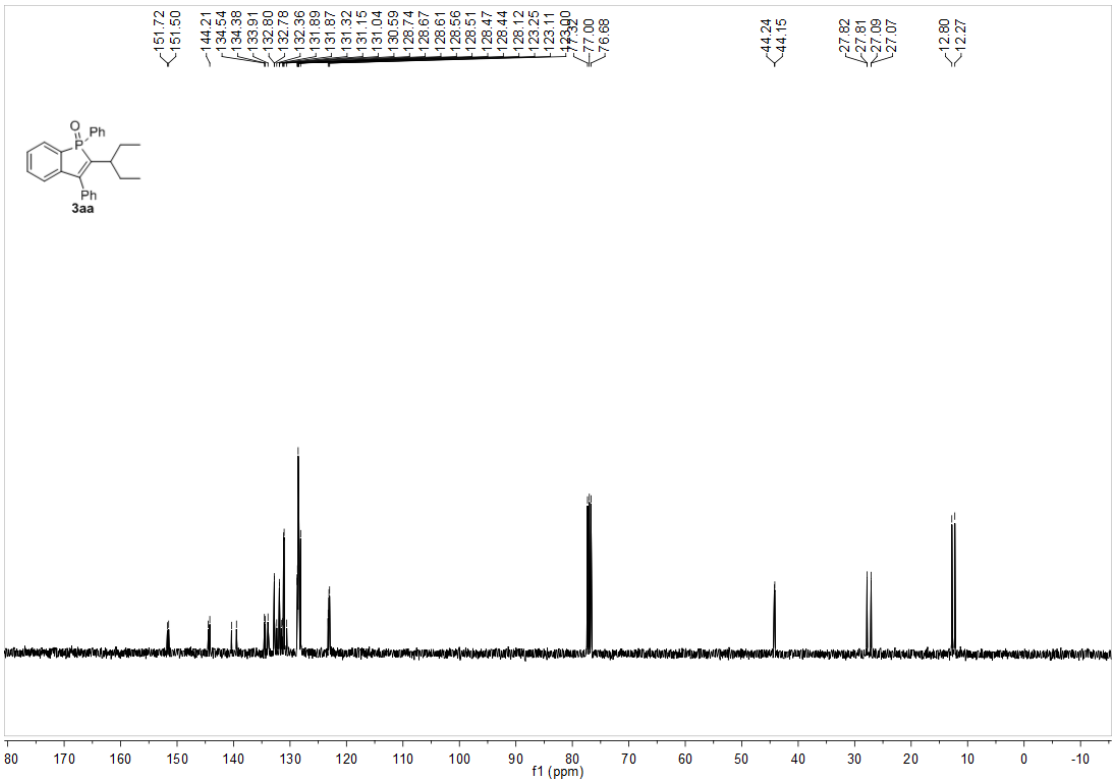
5. Ma, D.; Pan, J.; Yin, L.; Xu, P.; Gao, Y.; Yin, Y.; Zhao, Y. *Org. Lett.* **2018**, *20*, 3455.

7. Copies of ^{1}H, ^{31}P and ^{13}C NMR charts of the products



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131.87
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131.15
131.04
130.59
128.74
128.67
128.61
128.56
128.51
128.47
128.44
128.12
123.25
123.11
77.32
77.00
76.68
44.24
44.15
27.82
27.81
27.09
27.07
12.80
12.27
O
P
Ph
Ph
3aa
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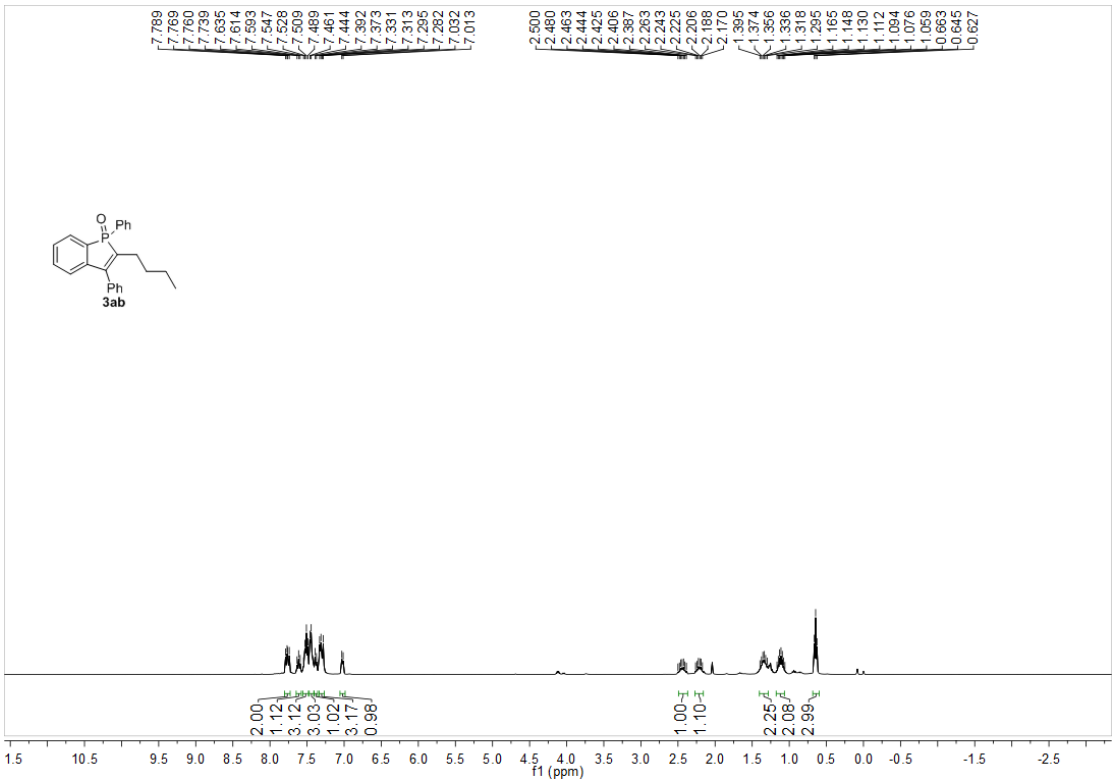
f1 (ppm)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7.444
7.392
7.373
7.331
7.313
7.295
7.282
7.032
7.013
2.500
2.480
2.463
2.444
2.425
2.406
2.387
2.263
2.243
2.225
2.206
2.188
2.170
1.395
1.374
1.356
1.336
1.318
1.295
1.165
1.148
1.130
1.112
1.094
1.076
1.059
0.663
0.645
0.627
O
P
Ph
Ph
3ab
2.00
1.12
3.12
3.03
1.02
3.17
0.98
1.00
1.10
2.25
2.08
2.99
1.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.5 -2.5
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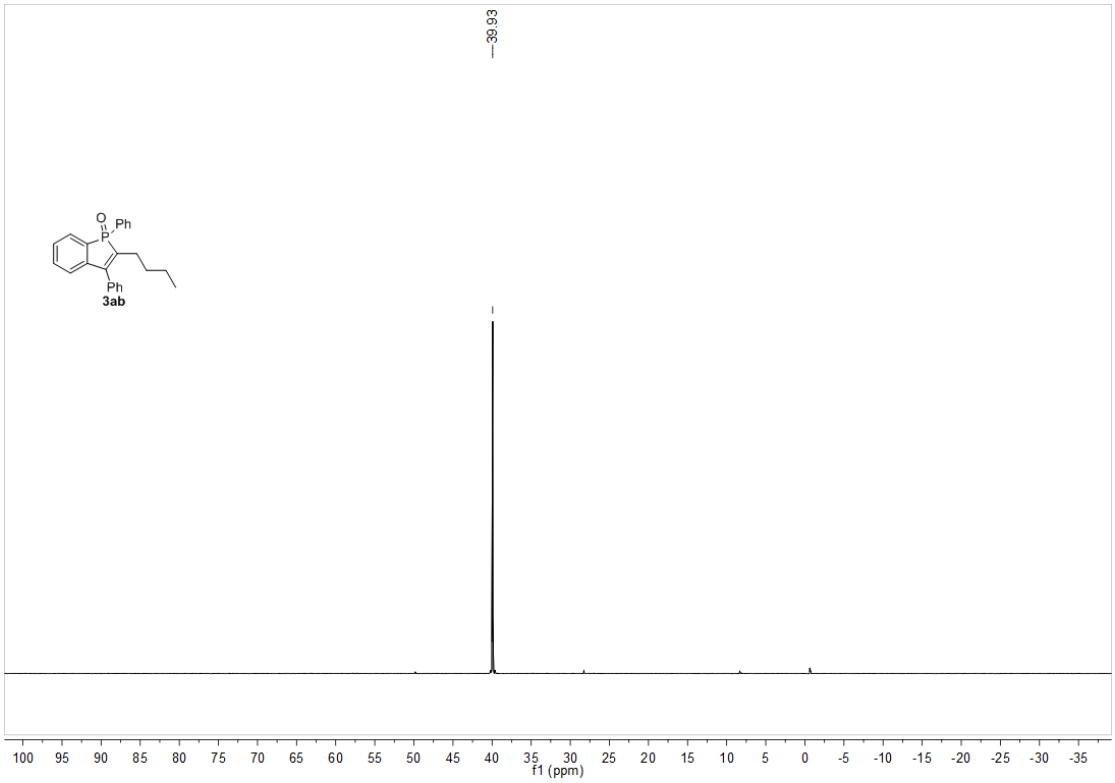
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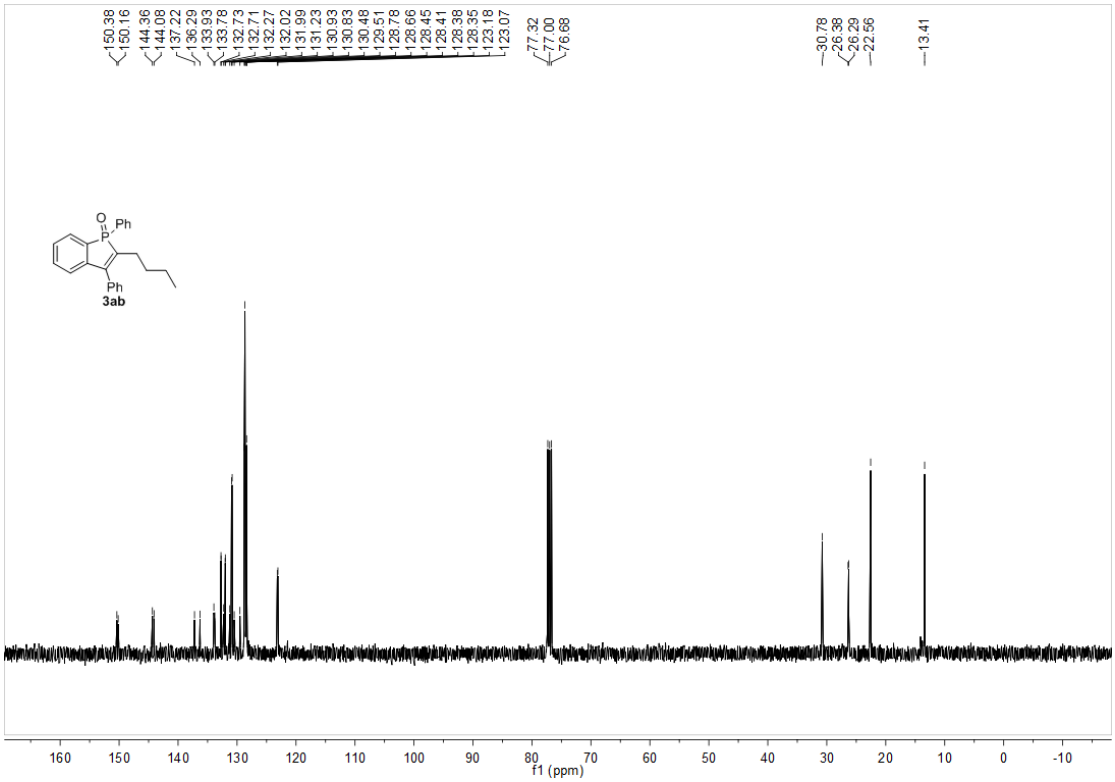
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131.23
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130.83
130.48
129.51
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128.41
128.38
128.35
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Ph
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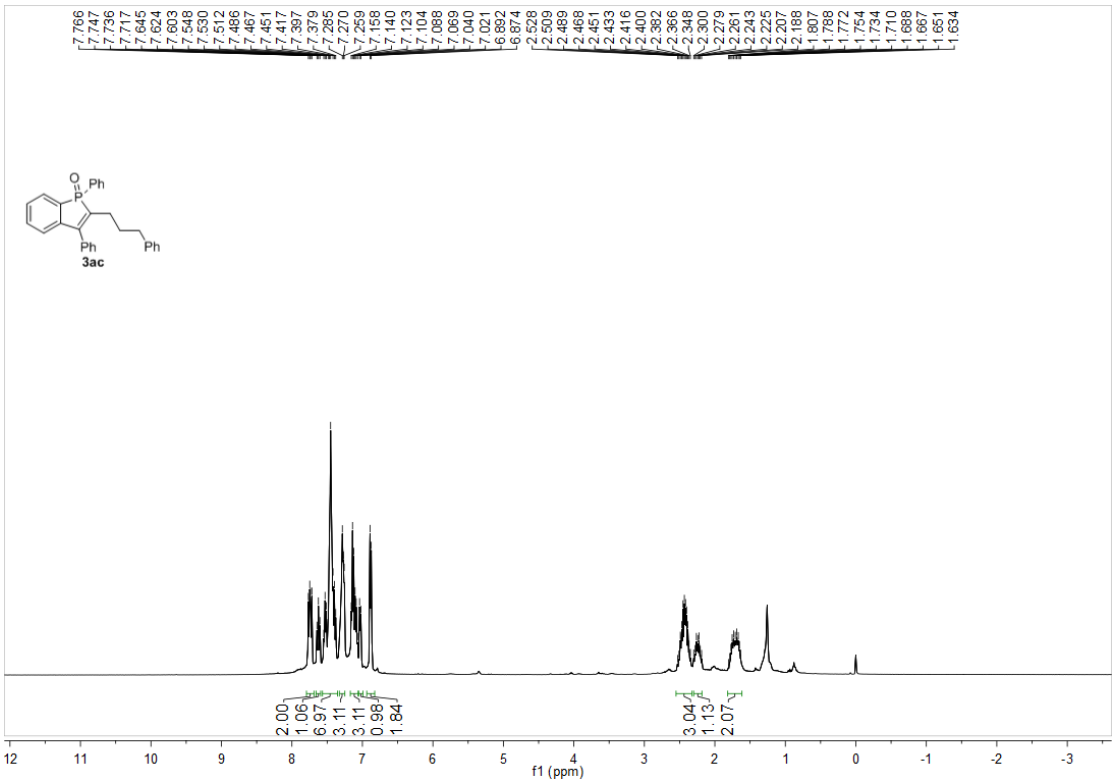
f1 (ppm)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Ph
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0.98
1.84
3.04
1.13
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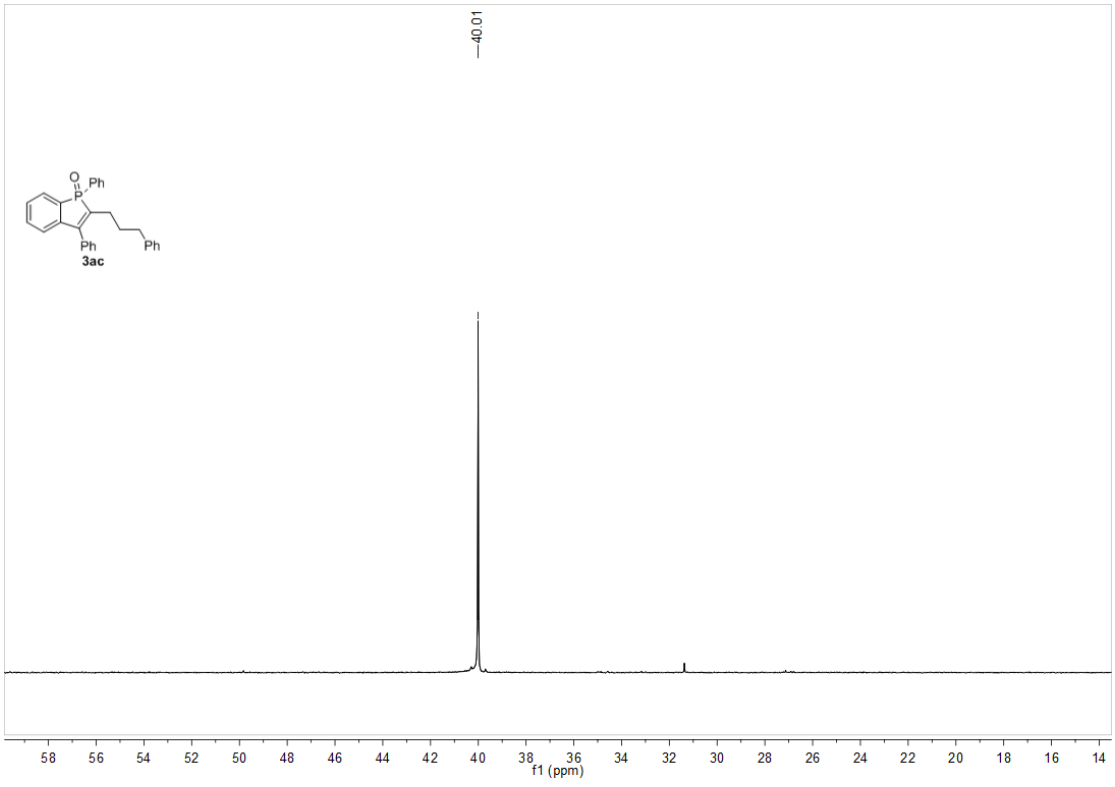
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)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24
22
20
18
16
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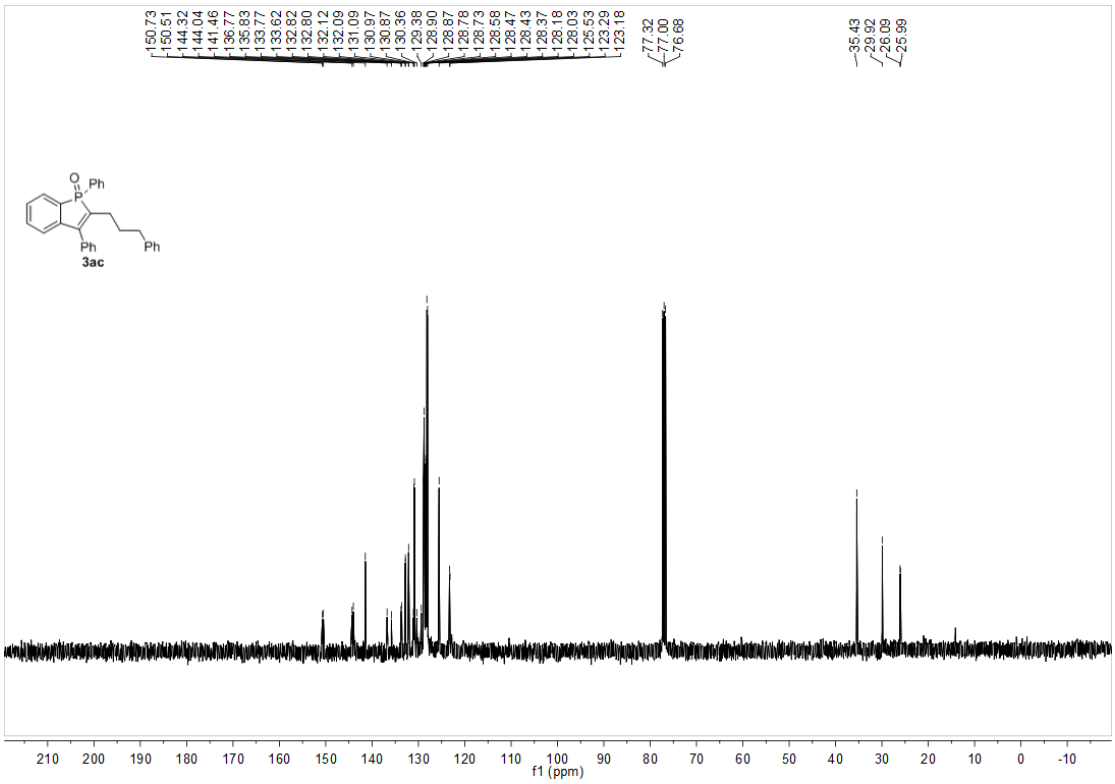
f1 (ppm)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132.09
131.09
130.97
130.87
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Ph
Ph
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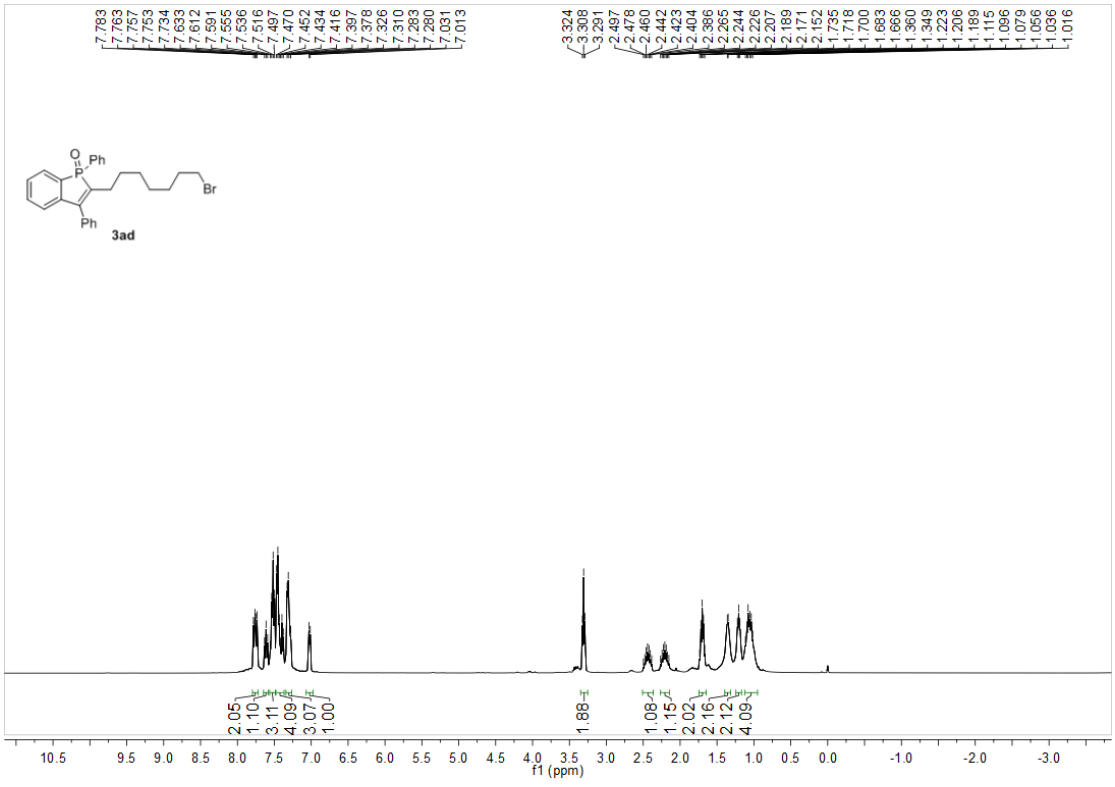
f1 (ppm)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3.07
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1.15
2.02
2.16
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4.09
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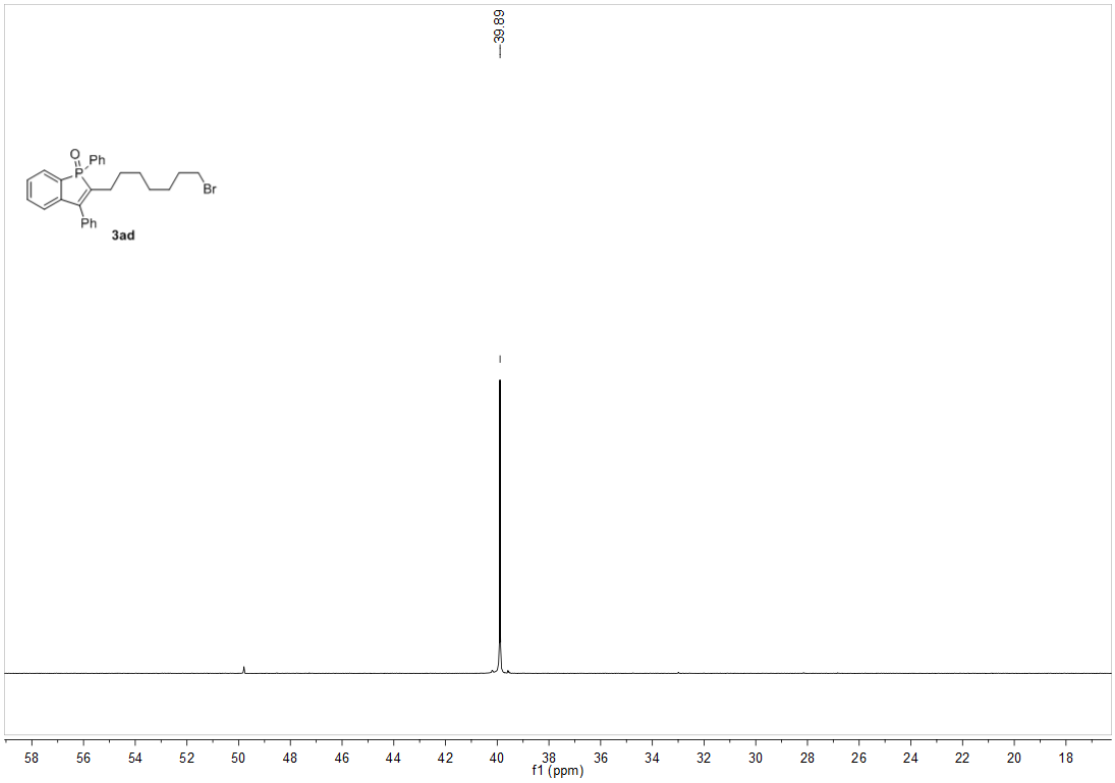
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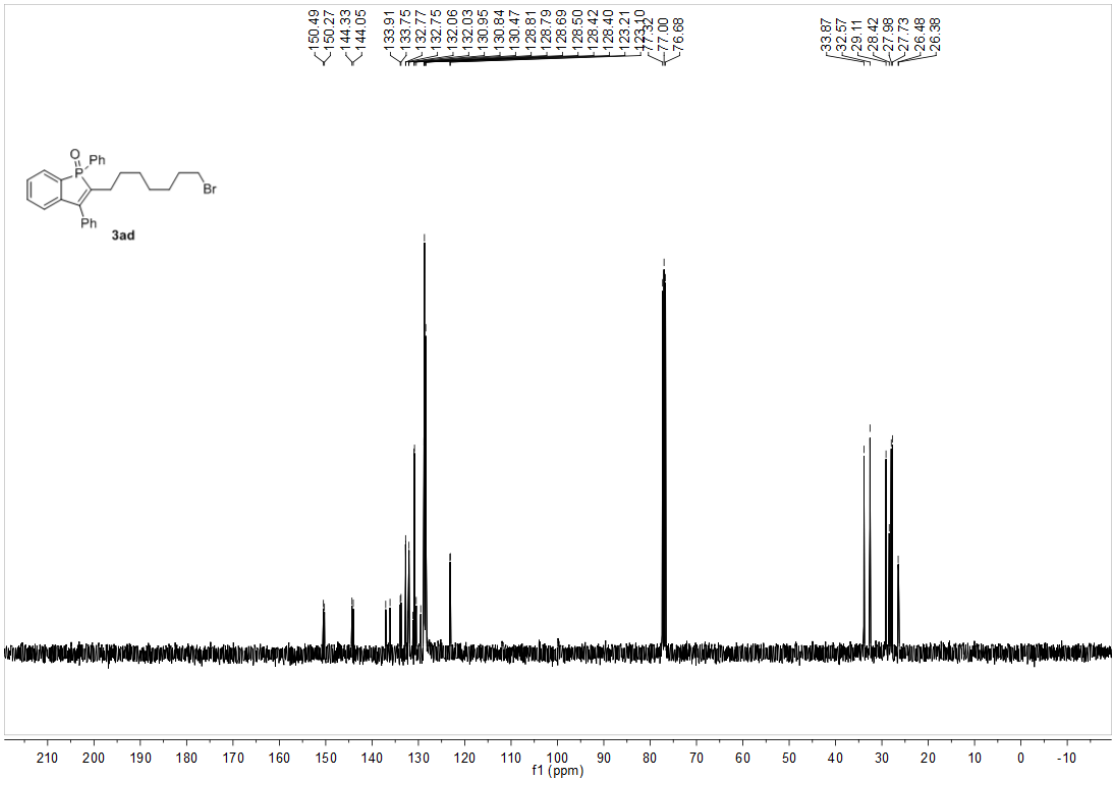
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32.57
29.11
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P
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Ph
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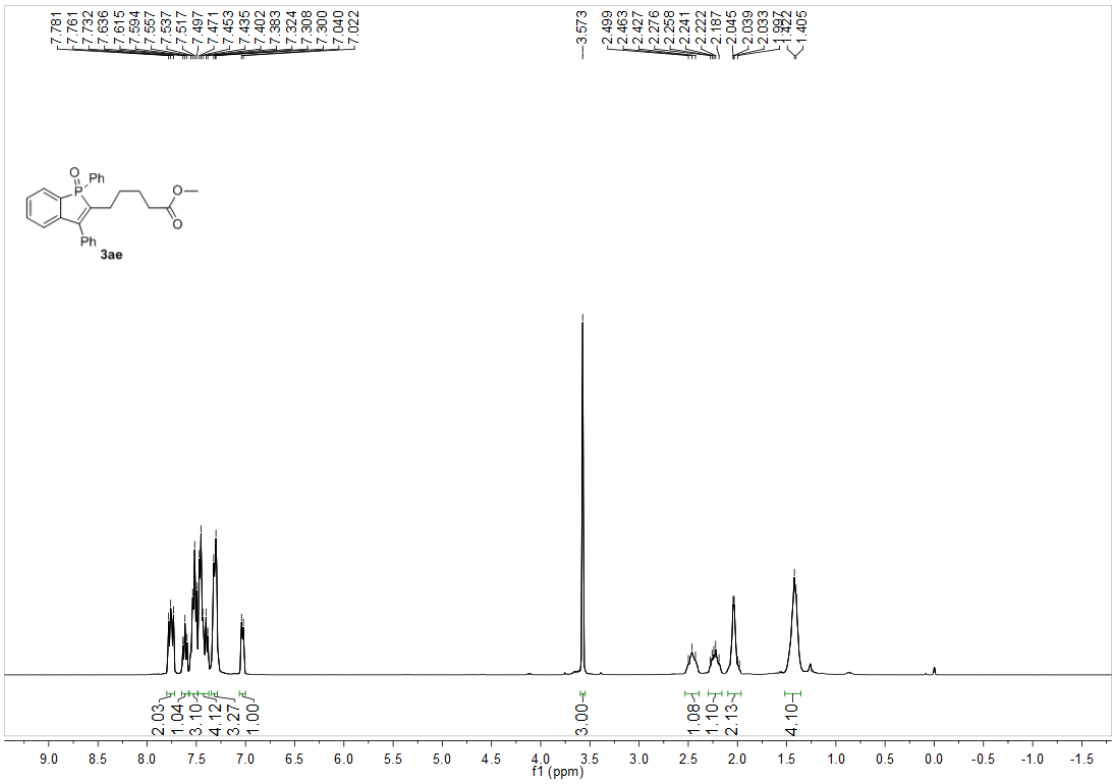
f1 (ppm)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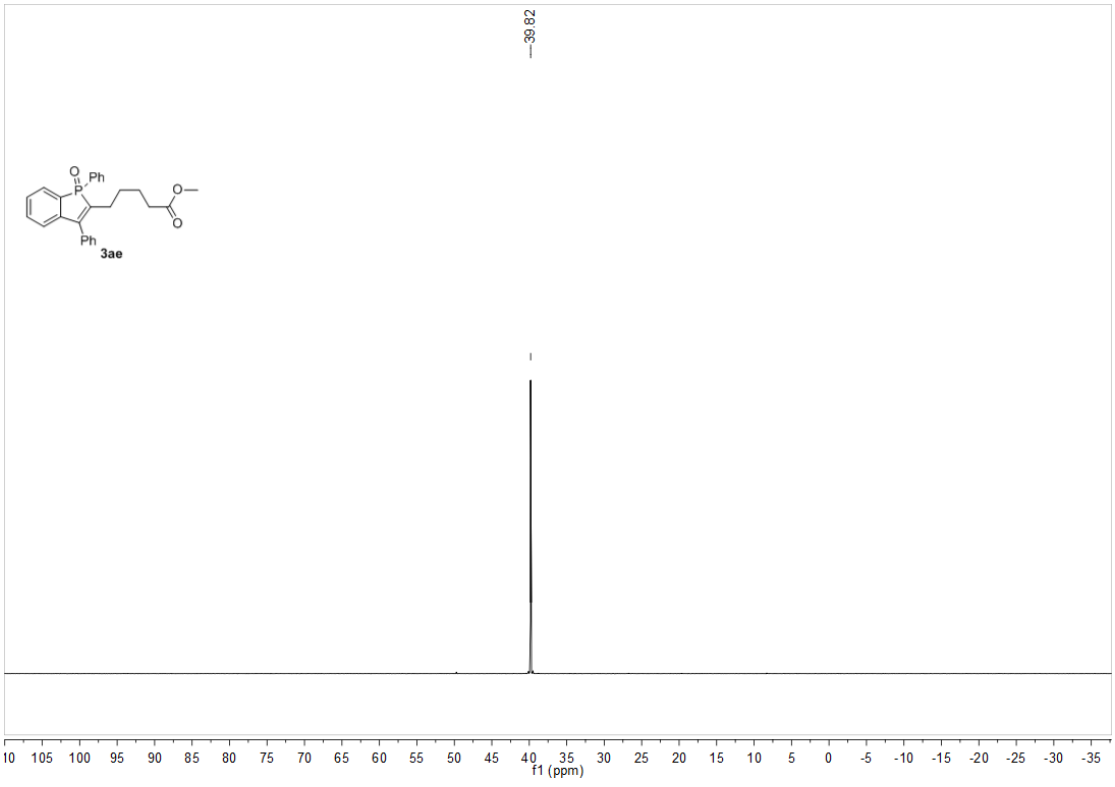
f1 (ppm)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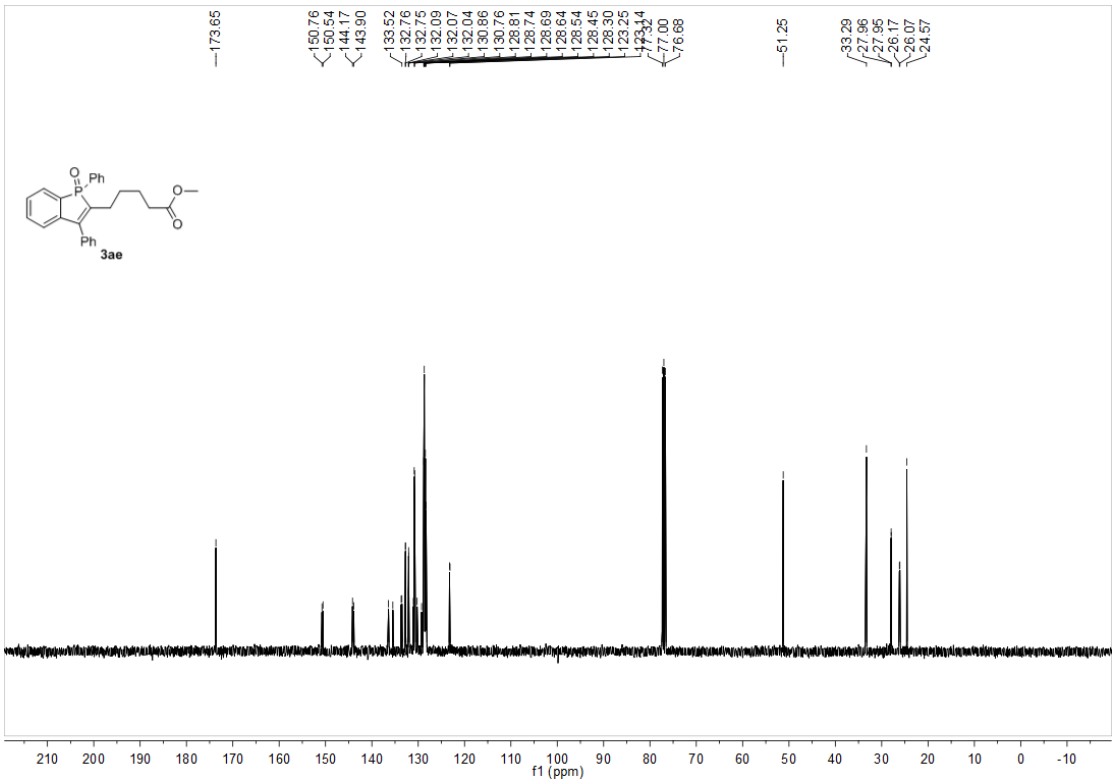
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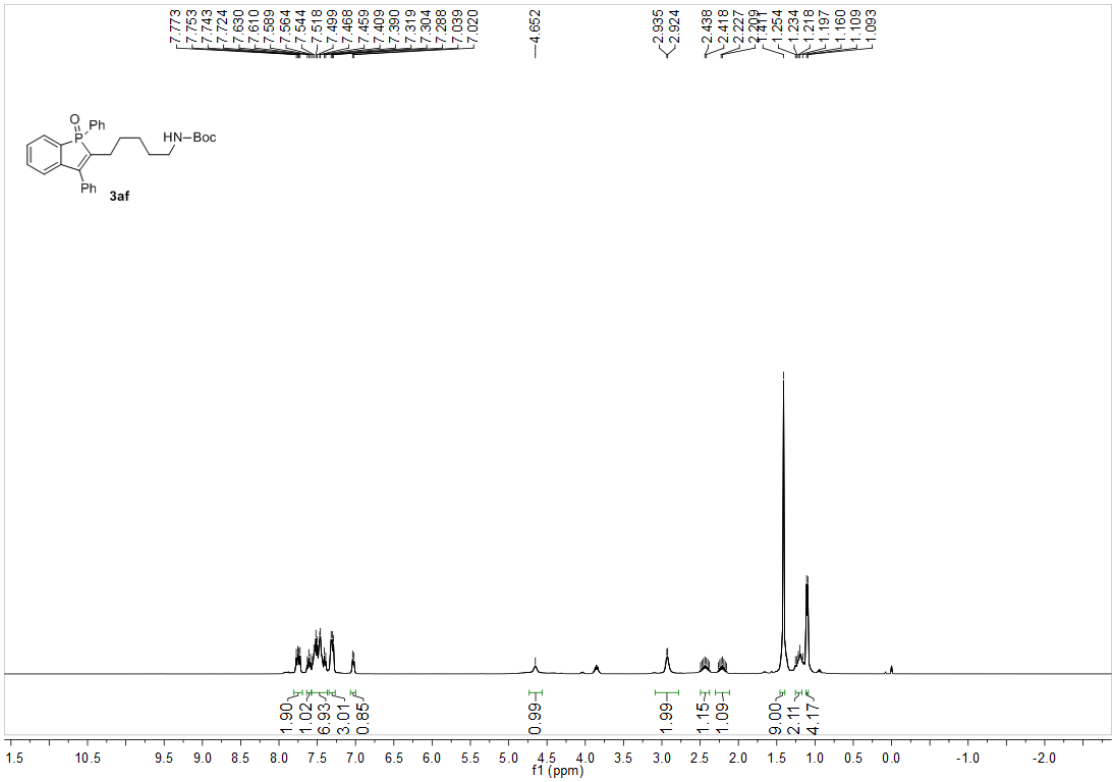
f1 (ppm)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HN–Boc
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1.09
9.00
2.11
4.17
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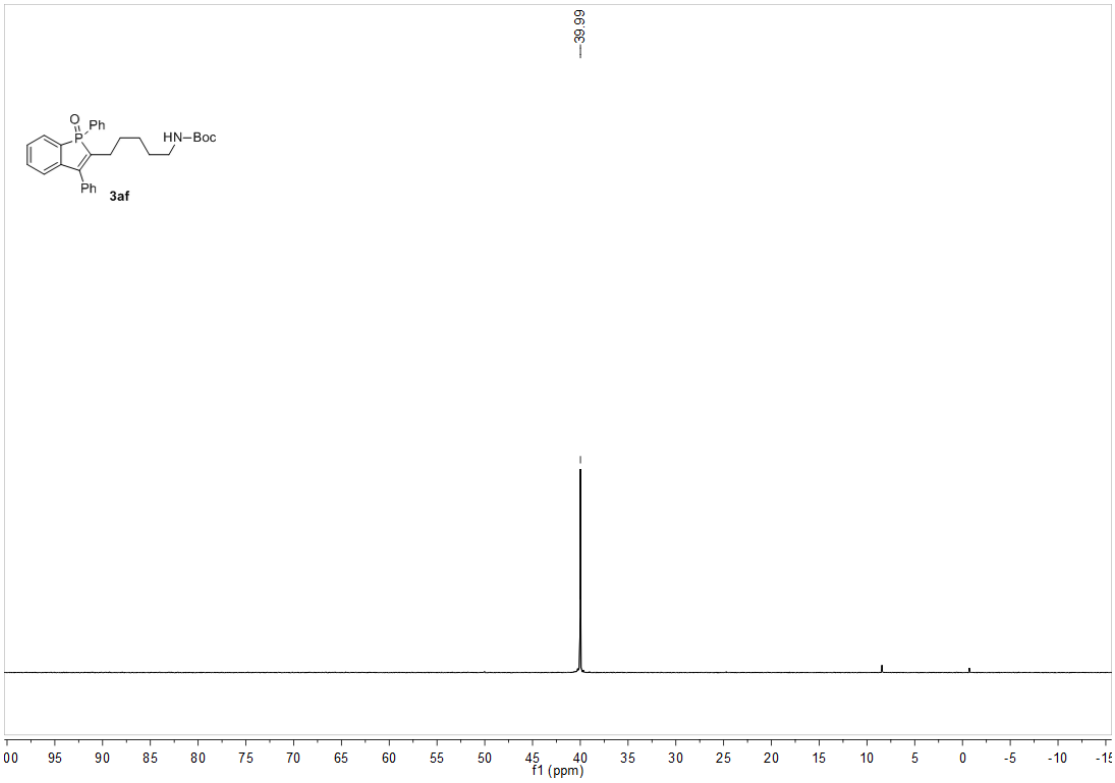
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HN–Boc
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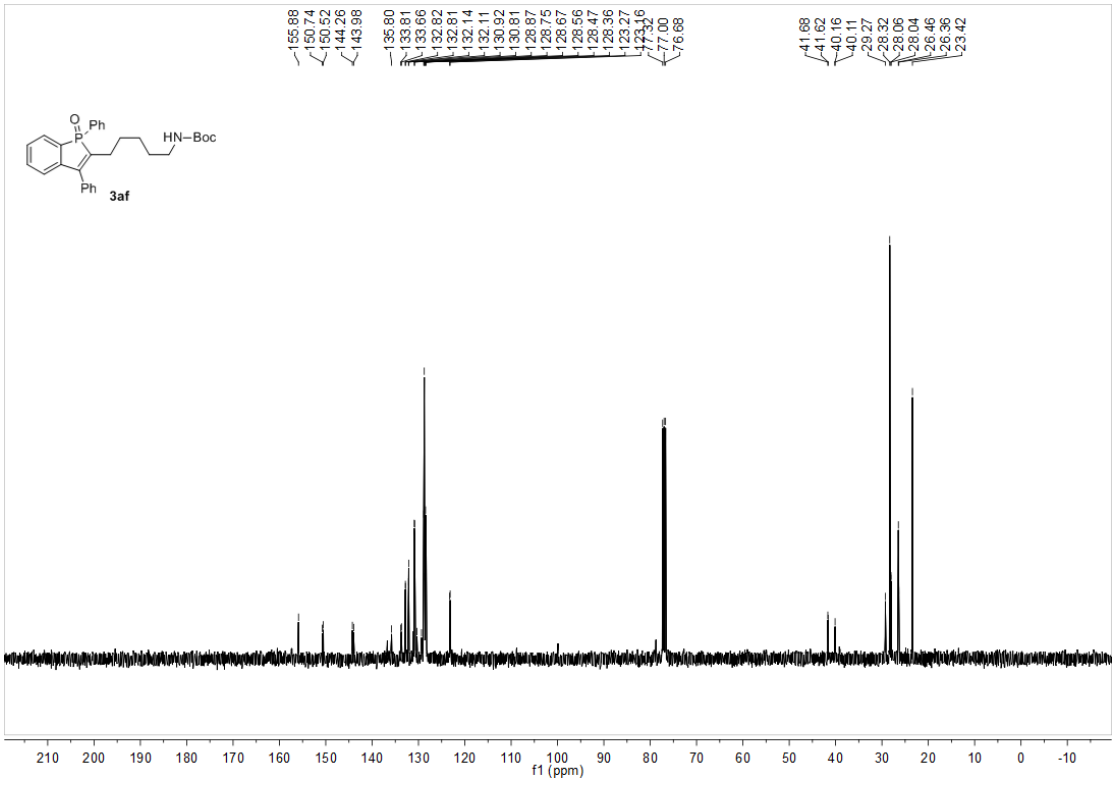
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Ph
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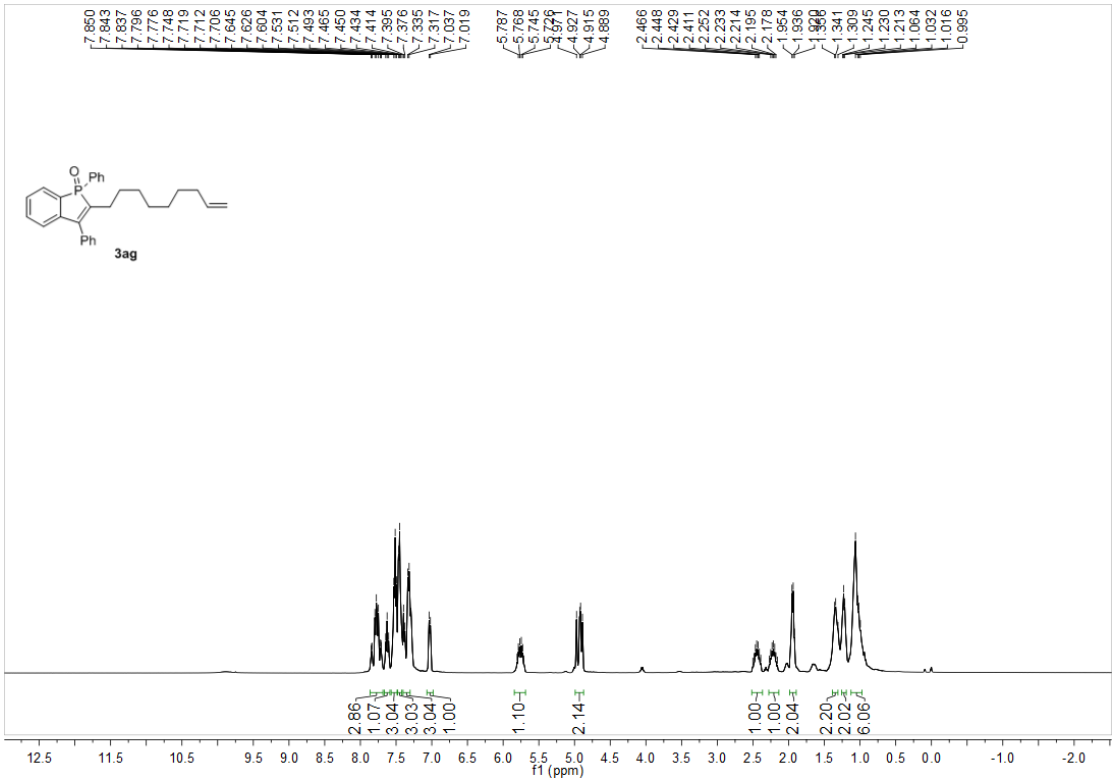
f1 (ppm)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1.309
1.245
1.230
1.213
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1.032
1.016
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O
P
Ph
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3.03
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1.10
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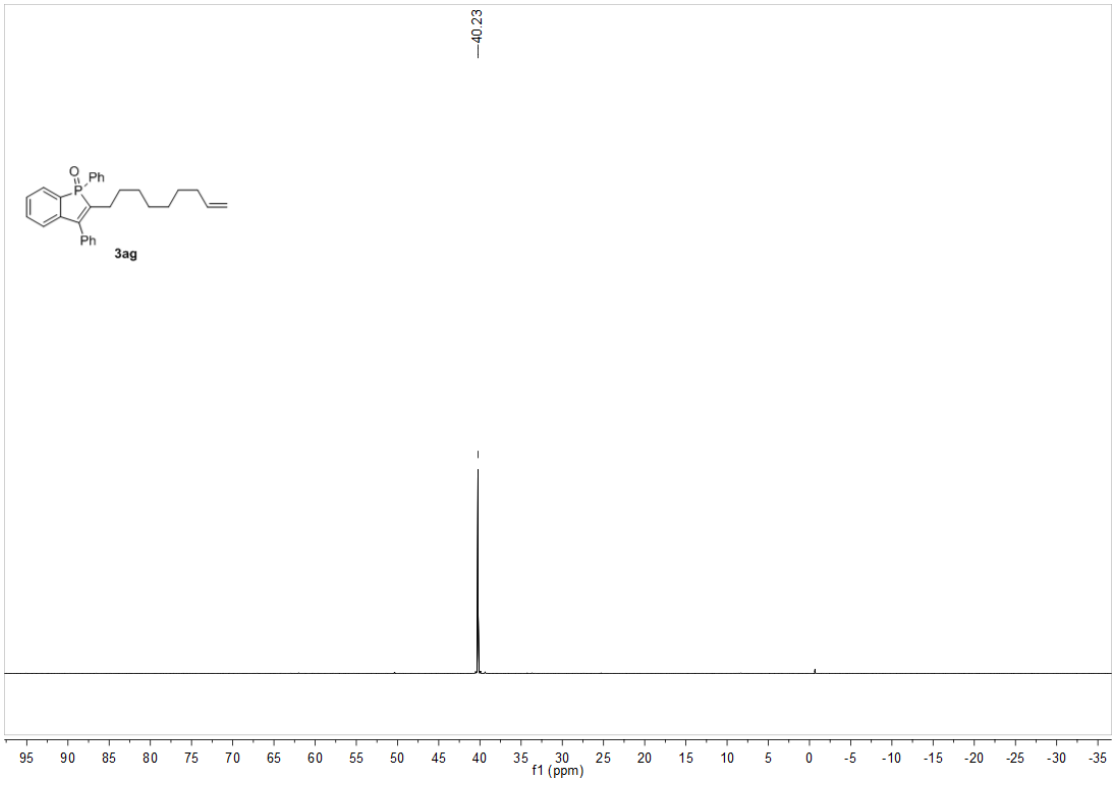
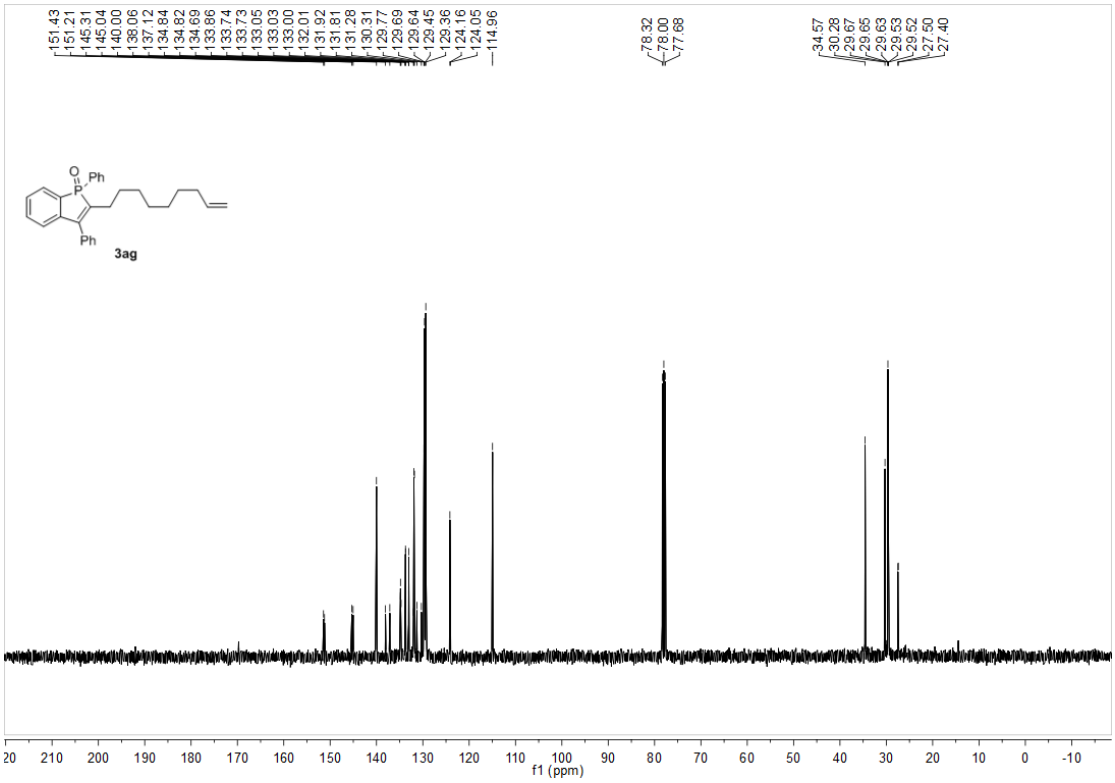
1.00
2.04
2.20
2.02
6.06
f1 (ppm)


40.23
Ph
O
P
Ph
3ag
f1 (ppm)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134.69
133.86
133.74
133.73
133.05
133.03
133.00
132.01
131.92
131.81
131.28
130.31
129.77
129.69
129.64
129.45
129.36
124.16
124.05
114.96
78.32
78.00
77.68
34.57
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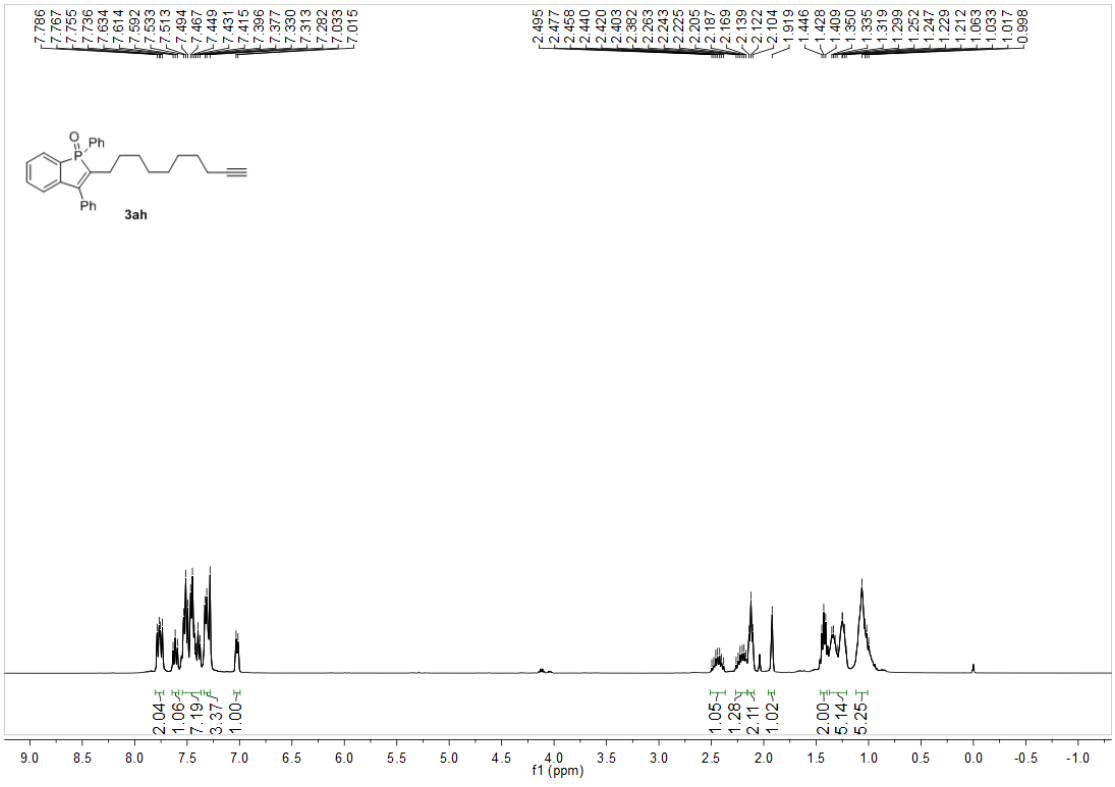
29.63
29.53
29.52
27.50
27.40
O
P
Ph
Ph
3ag
f1 (ppm)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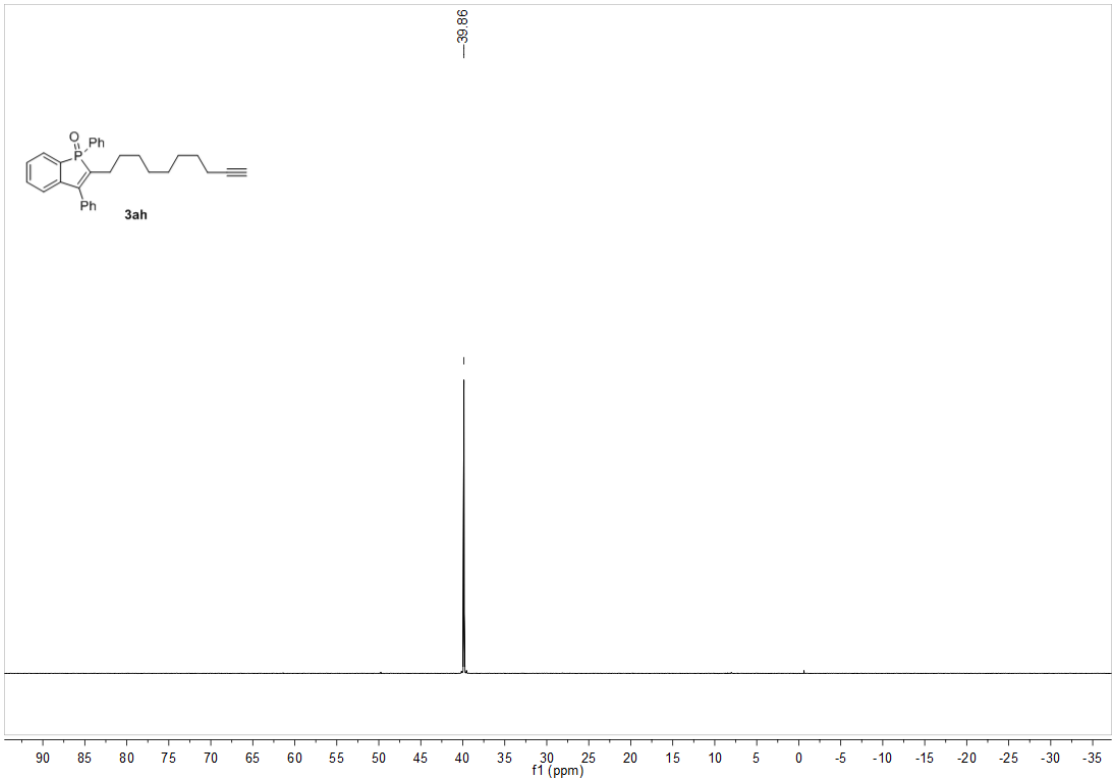
7.533
7.513
7.494
7.467
7.449
7.431
7.415
7.396
7.377
7.330
7.313
7.282
7.033
7.015
2.495
2.477
2.458
2.440
2.420
2.403
2.382
2.263
2.243
2.225
2.205
2.187
2.169
2.139
2.122
2.104
1.919
1.446
1.428
1.409
1.350
1.335
1.319
1.299
1.252
1.247
1.229
1.212
1.063
1.033
1.017
0.998
O
P
Ph
Ph
3ah
2.04
1.06
7.19
3.37
1.00
1.05
1.28
2.11
1.02
2.00
5.14
5.25
f1 (ppm)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Ph
Ph
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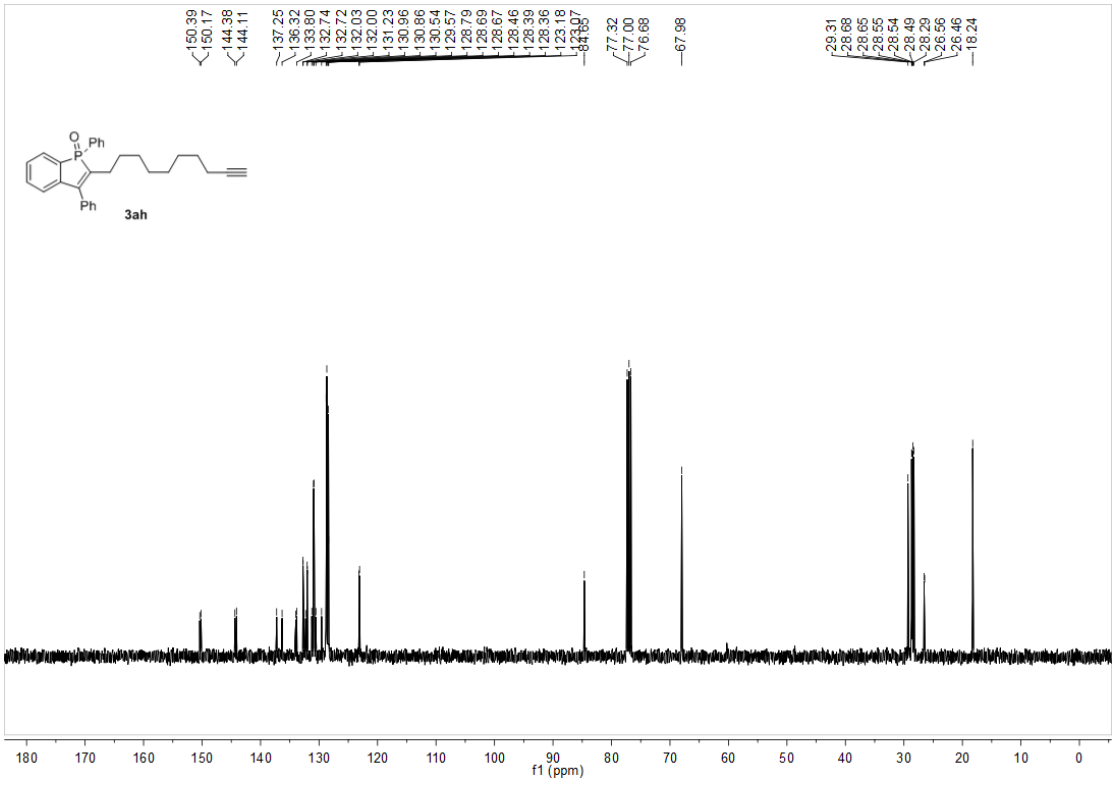
f1 (ppm)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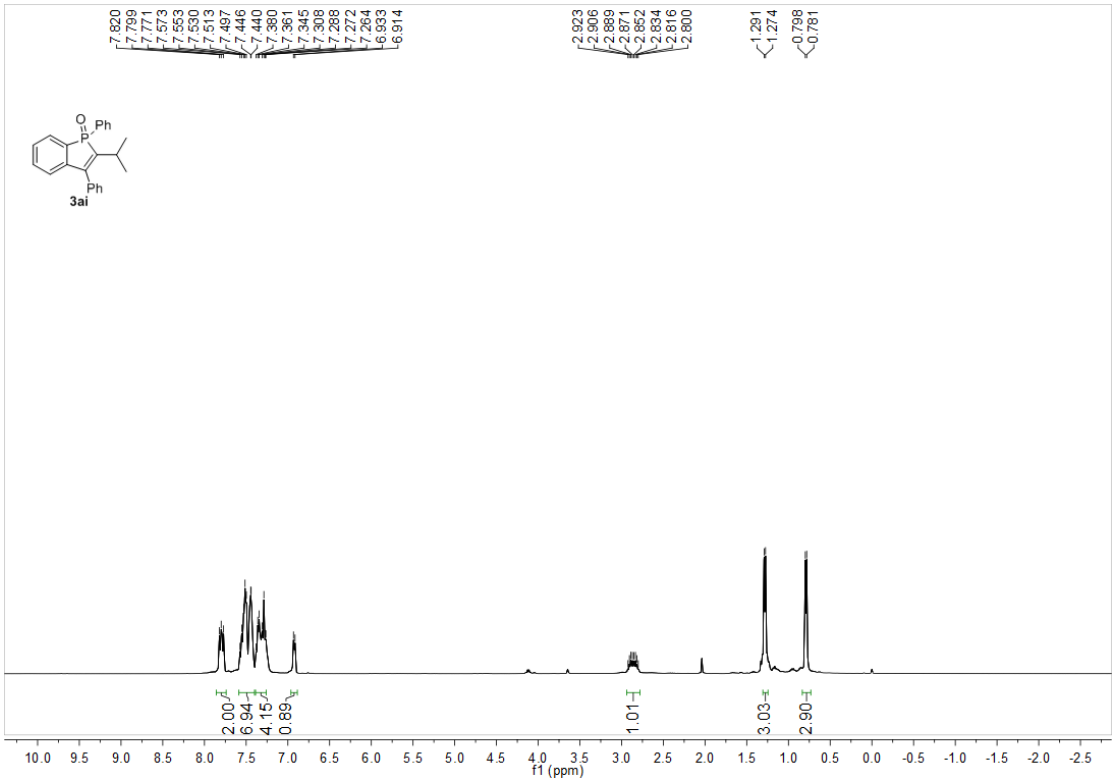
131.23
130.96
130.86
130.54
129.57
128.79
128.69
128.67
128.46
128.39
128.36
123.18
123.07
84.65
77.32
77.00
76.68
67.98
29.31
28.68
28.65
28.55
28.54
28.49
28.29
26.56
26.46
18.24
Ph
Ph
3ah
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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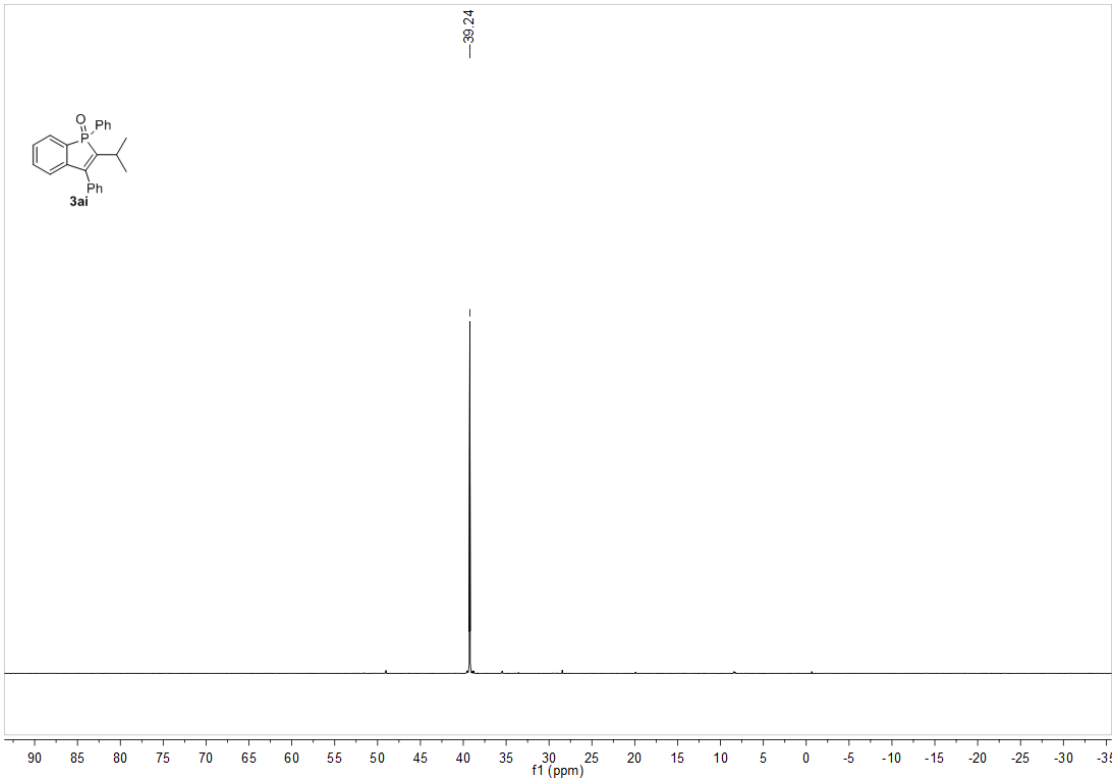
7.513
7.497
7.446
7.440
7.380
7.361
7.345
7.308
7.288
7.272
7.264
6.933
6.914
2.923
2.906
2.889
2.871
2.852
2.834
2.816
2.800
1.291
1.274
0.798
0.781
O
P
Ph
Ph
3ai
2.00
6.94
4.15
0.89
1.01
3.03
2.90
f1 (ppm)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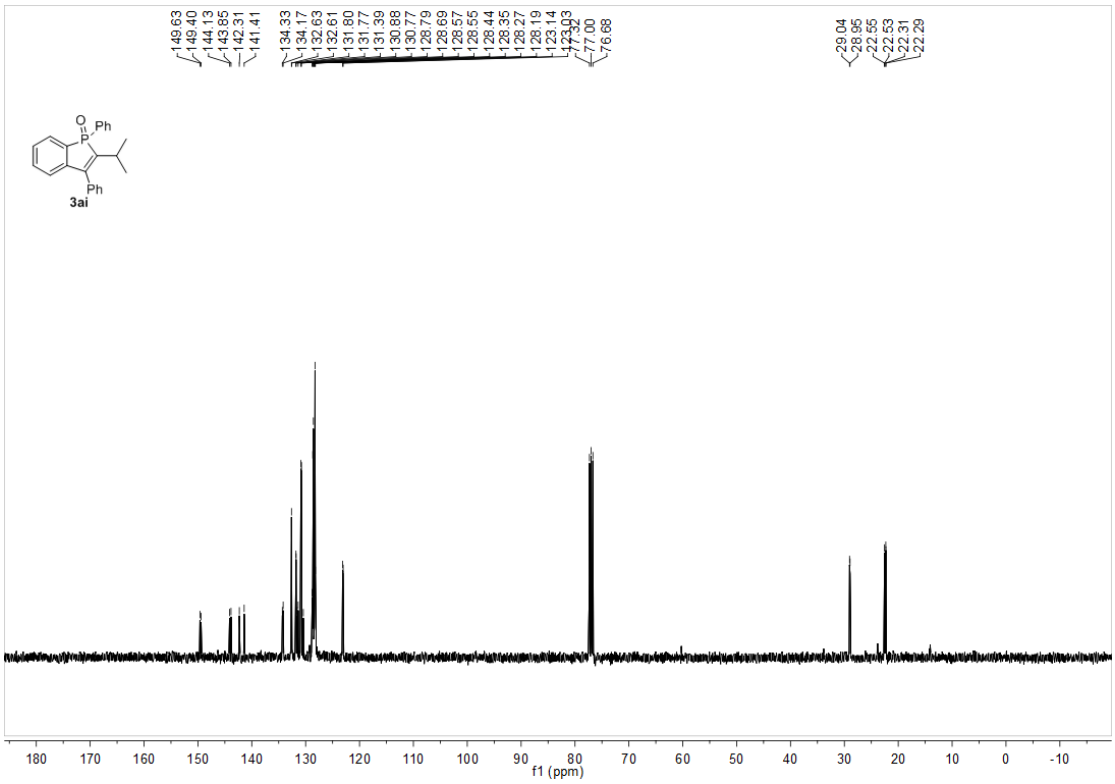
P
Ph
Ph
3ai
f1 (ppm)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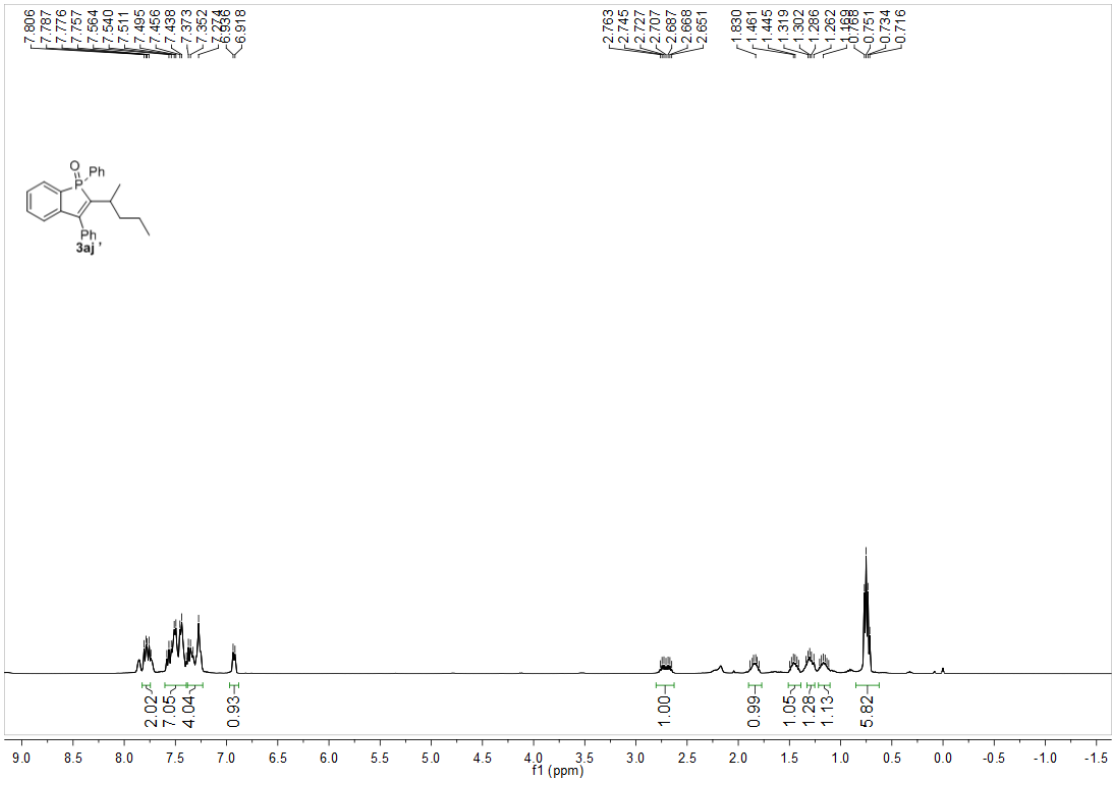
141.41
134.33
134.17
132.63
132.61
131.80
131.77
131.39
130.88
130.77
128.79
128.69
128.57
128.55
128.44
128.35
128.27
128.19
123.14
77.32
77.00
76.68
29.04
28.95
22.55
22.53
22.31
22.29
O
P
Ph
Ph
3ai
f1 (ppm)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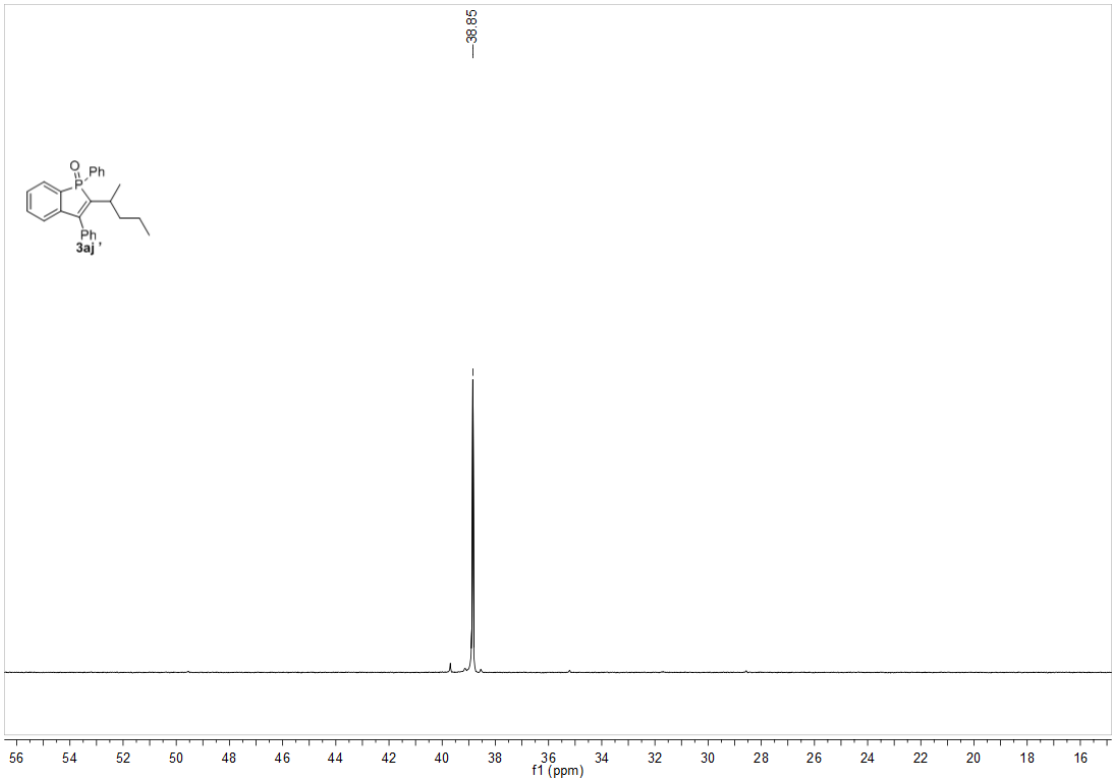
7.776
7.757
7.564
7.540
7.511
7.495
7.456
7.438
7.373
7.362
6.936
6.918
2.763
2.745
2.727
2.707
2.687
2.668
2.651
1.830
1.461
1.445
1.319
1.302
1.286
1.262
0.751
0.734
0.716
2.02
7.05
4.04
0.93
1.00
0.99
1.05
1.28
1.13
5.82
O
P
Ph
Ph
3aj '
f1 (ppm)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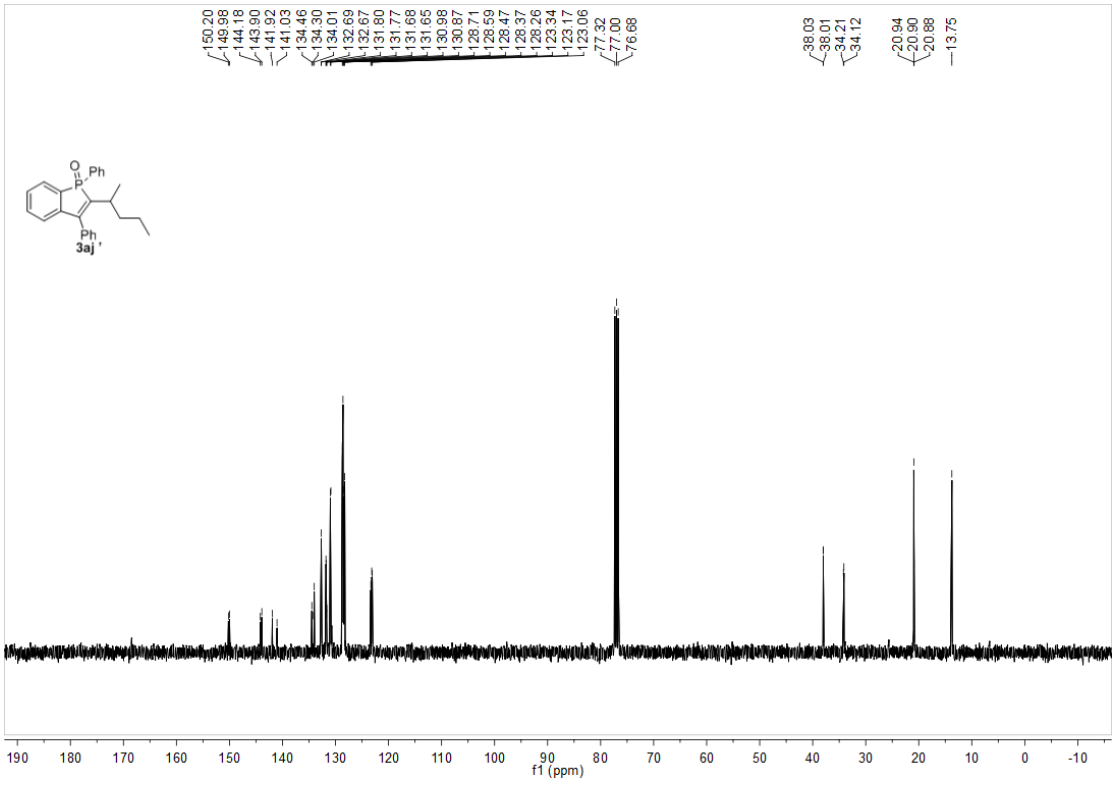
O
Ph
P
Ph
3aj'
56 54 52 50 48 46 44 42 40 38 36 34 32 30 28 26 24 22 20 18 16
f1 (ppm)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131.77
131.68
131.65
130.98
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128.71
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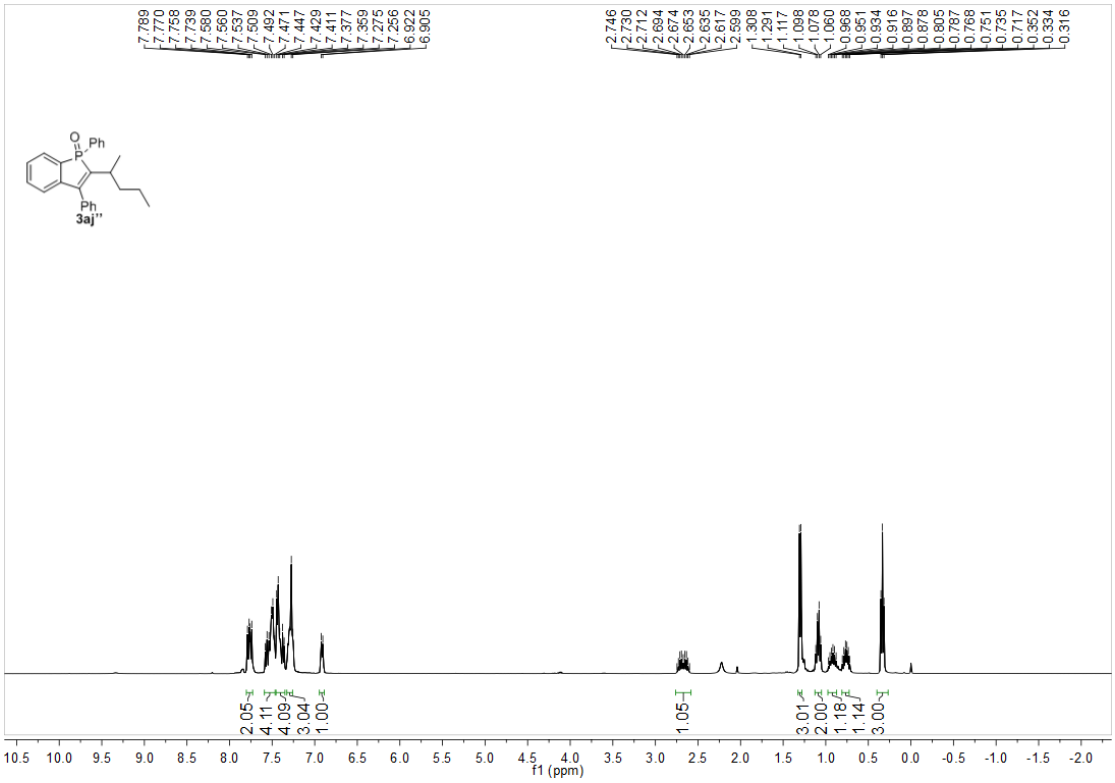
128.47
128.37
128.26
123.34
123.17
123.06
77.32
77.00
76.68
38.03
38.01
34.21
34.12
20.94
20.90
20.88
13.75
O
Ph
P
Ph
3aj'
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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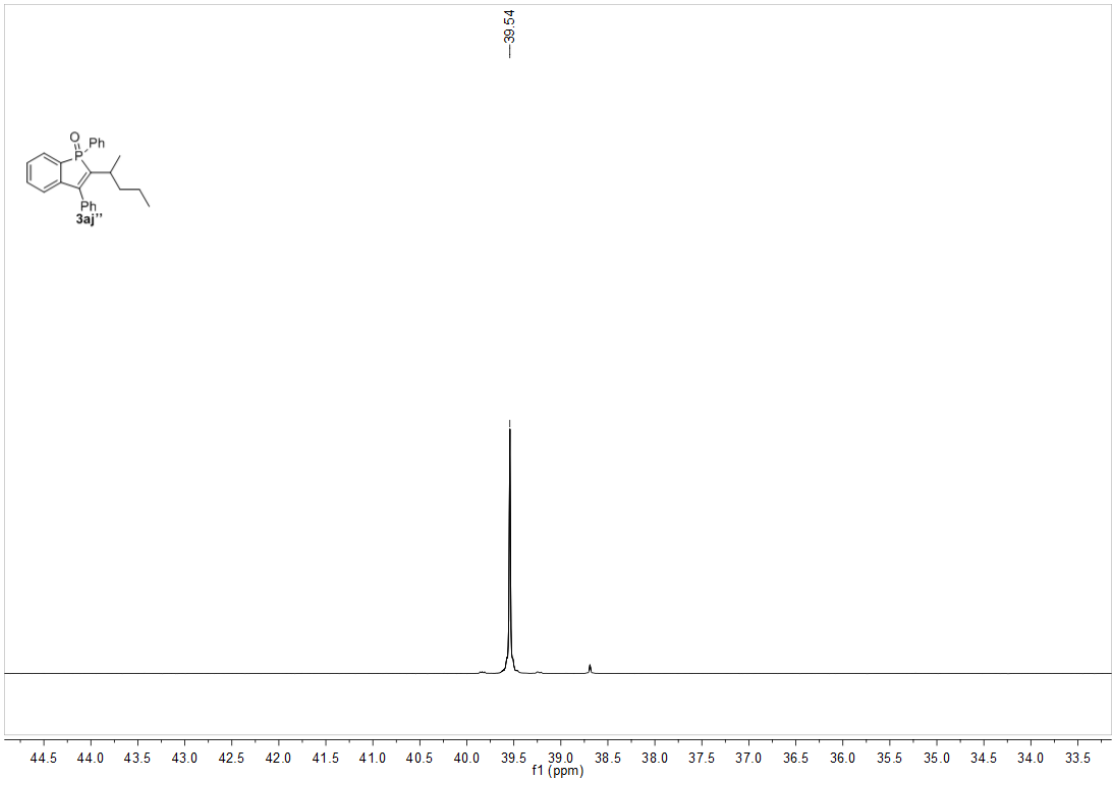
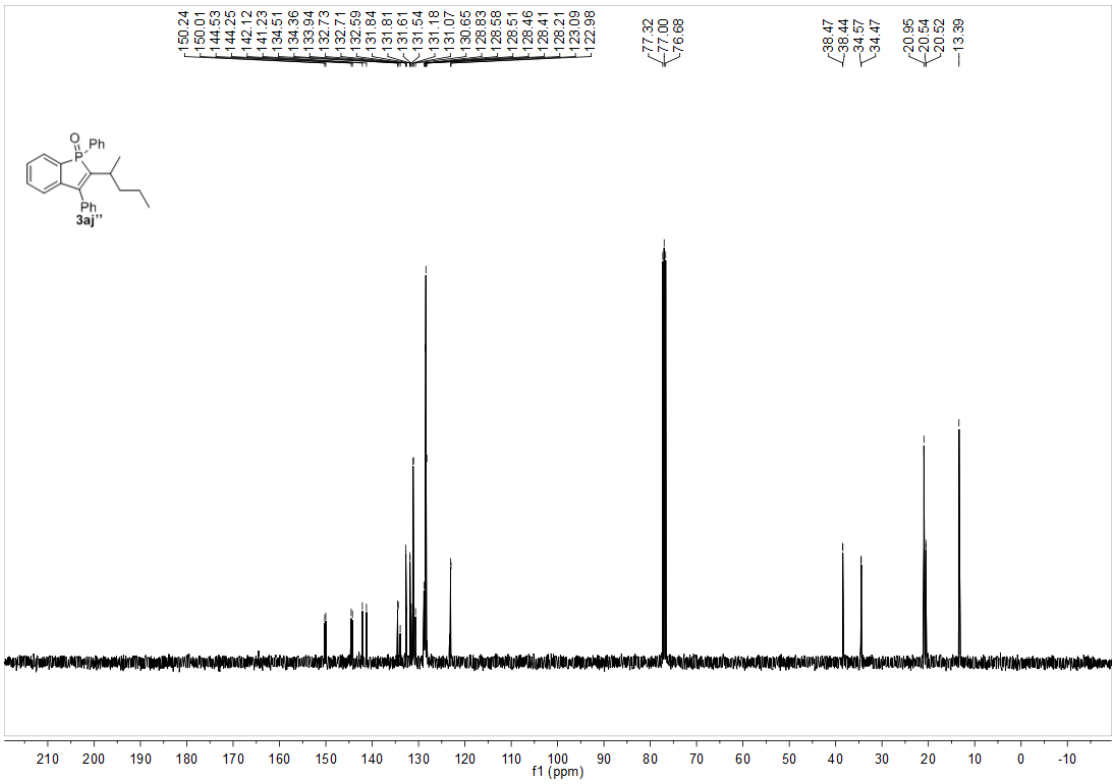
7.537
7.509
7.492
7.471
7.447
7.429
7.411
7.377
7.359
7.275
7.256
6.922
6.905
2.746
2.730
2.712
2.694
2.674
2.653
2.635
2.617
2.599
1.308
1.291
1.117
1.098
1.078
1.060
0.968
0.951
0.934
0.916
0.897
0.878
0.805
0.787
0.768
0.751
0.735
0.717
0.352
0.334
0.316
Ph
Ph
3aj''
2.05
4.11
4.09
3.04
1.00
1.05
3.01
2.00
1.18
1.14
3.00
f1 (ppm)


39.54
Ph
Ph
3aj''
f1 (ppm)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141.23
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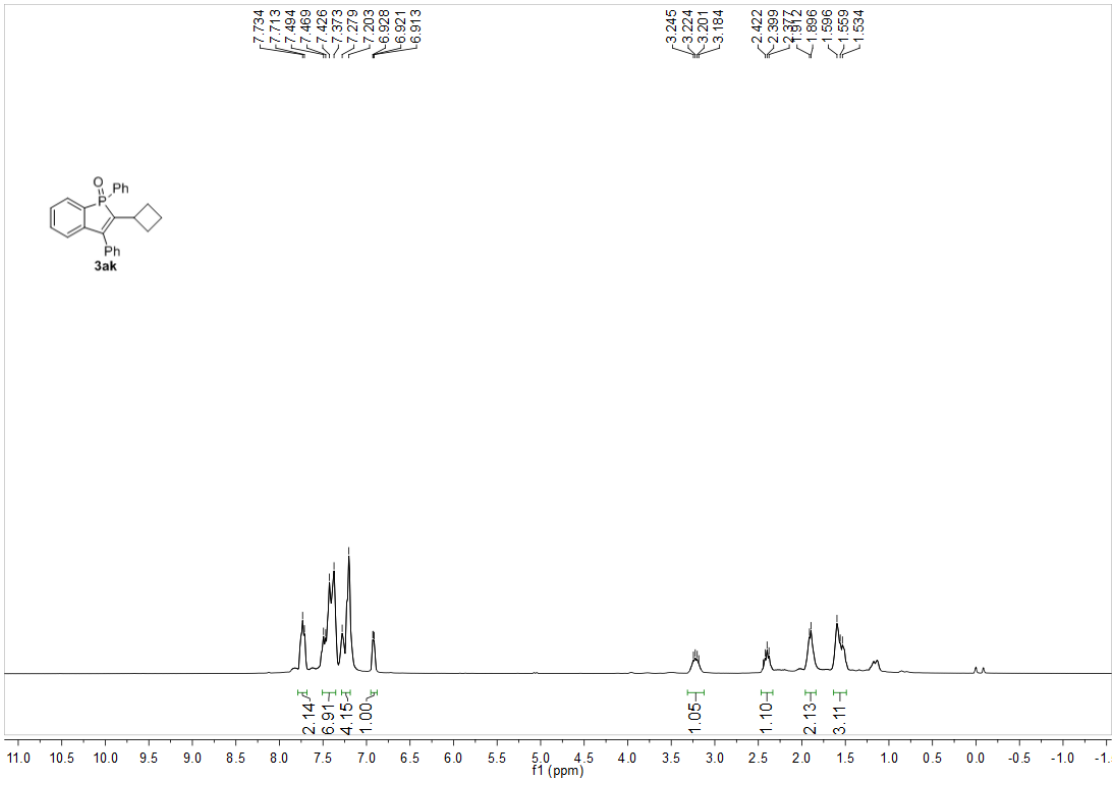
134.36
133.94
132.73
132.71
132.59
131.84
131.81
131.61
131.54
131.18
131.07
130.65
128.83
128.58
128.51
128.46
128.41
128.21
123.09
122.98
77.32
77.00
76.68
38.47
38.44
34.57
34.47
20.95
20.54
20.52
13.39
Ph
O
P
Ph
3aj''
f1 (ppm)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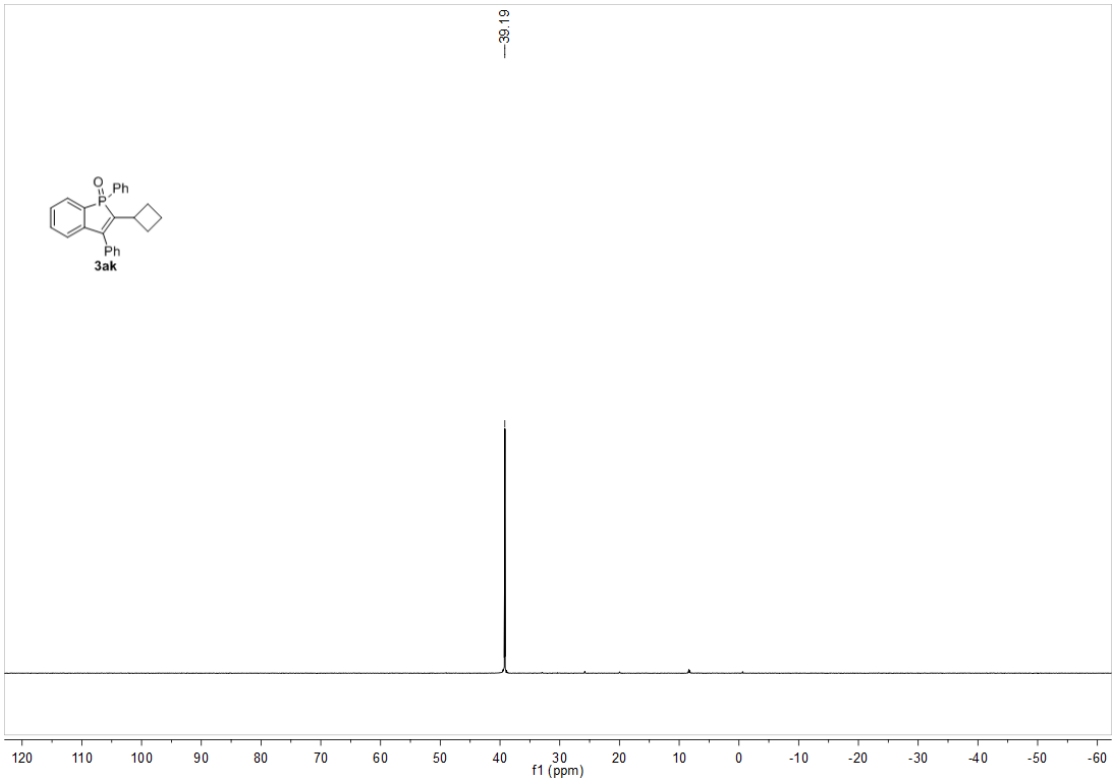
7.426
7.373
7.279
7.203
6.928
6.921
6.913
3.245
3.224
3.201
3.184
2.422
2.399
1.886
1.596
1.559
1.534
Ph
O
P
Ph
3ak
2.14
6.91
4.15
1.00
1.05
1.10
2.13
3.11
f1 (ppm)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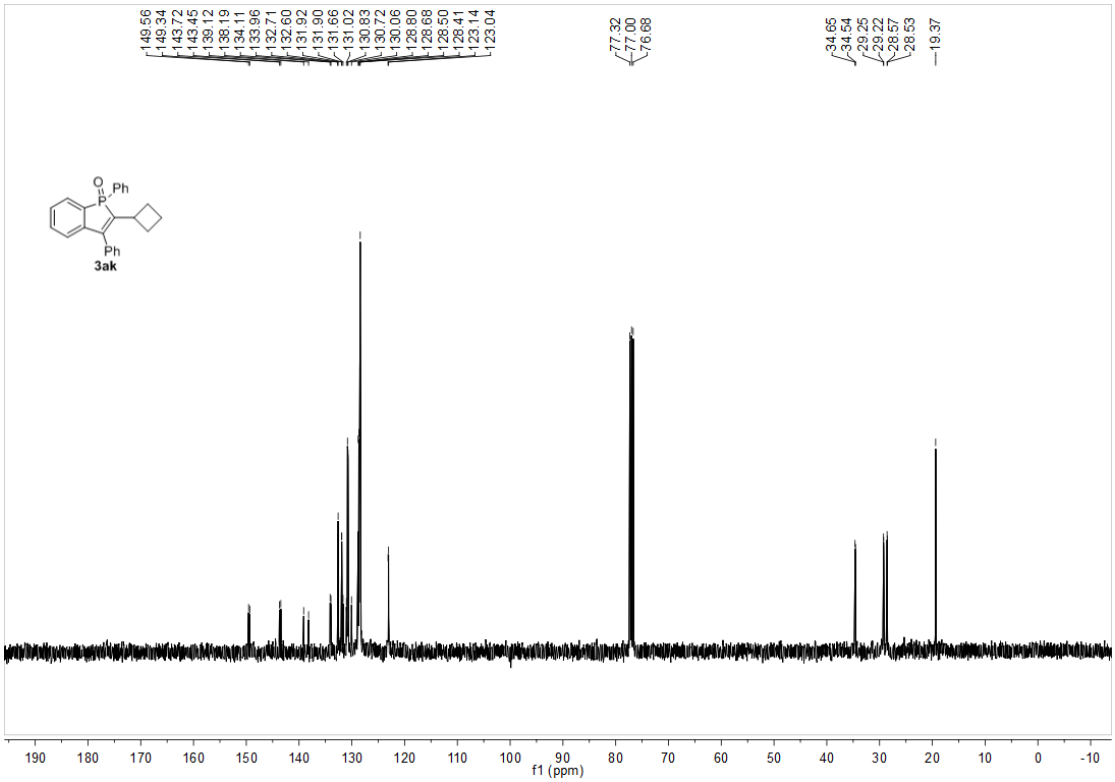
Ph
Ph
3ak
120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60
f1 (ppm)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138.19
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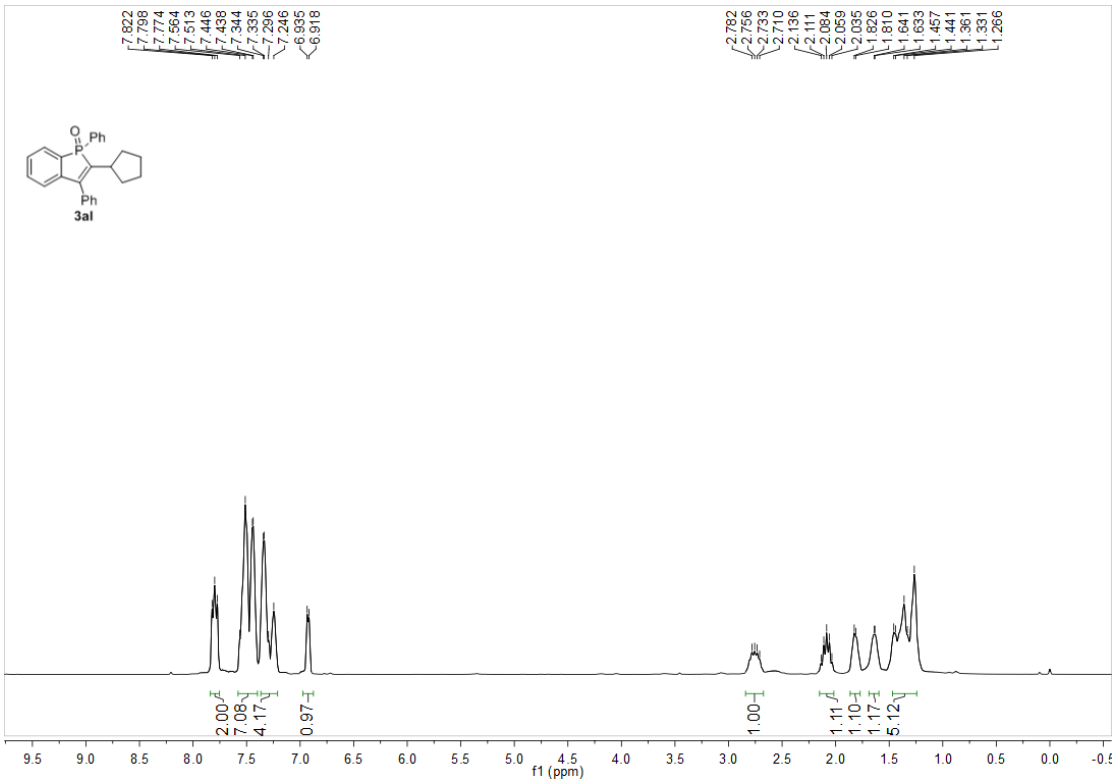
132.60
131.92
131.90
131.66
131.02
130.83
130.72
130.06
128.80
128.68
128.50
128.41
123.14
123.04
77.32
77.00
76.68
34.65
34.54
29.25
29.22
28.57
28.53
19.37
Ph
Ph
3ak
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.446
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7.344
7.335
7.296
7.246
6.935
6.918
2.782
2.756
2.733
2.710
2.136
2.111
2.084
2.059
2.035
1.826
1.810
1.641
1.633
1.457
1.441
1.361
1.331
1.266
Ph
Ph
3al
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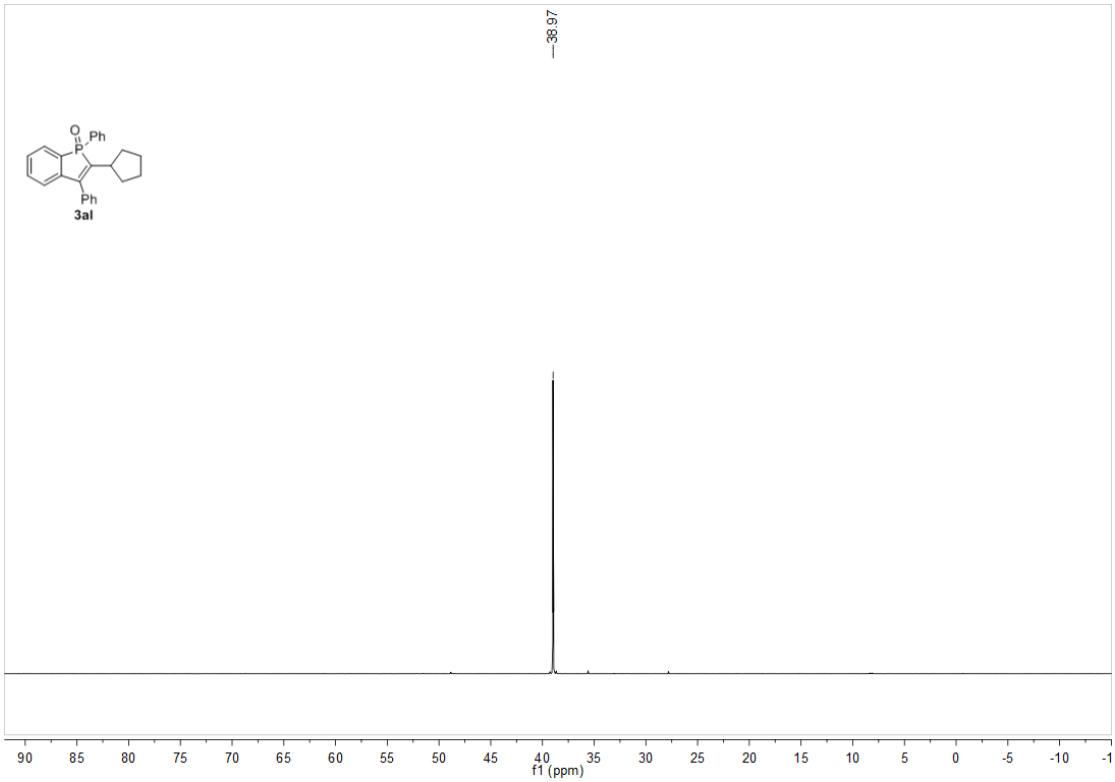
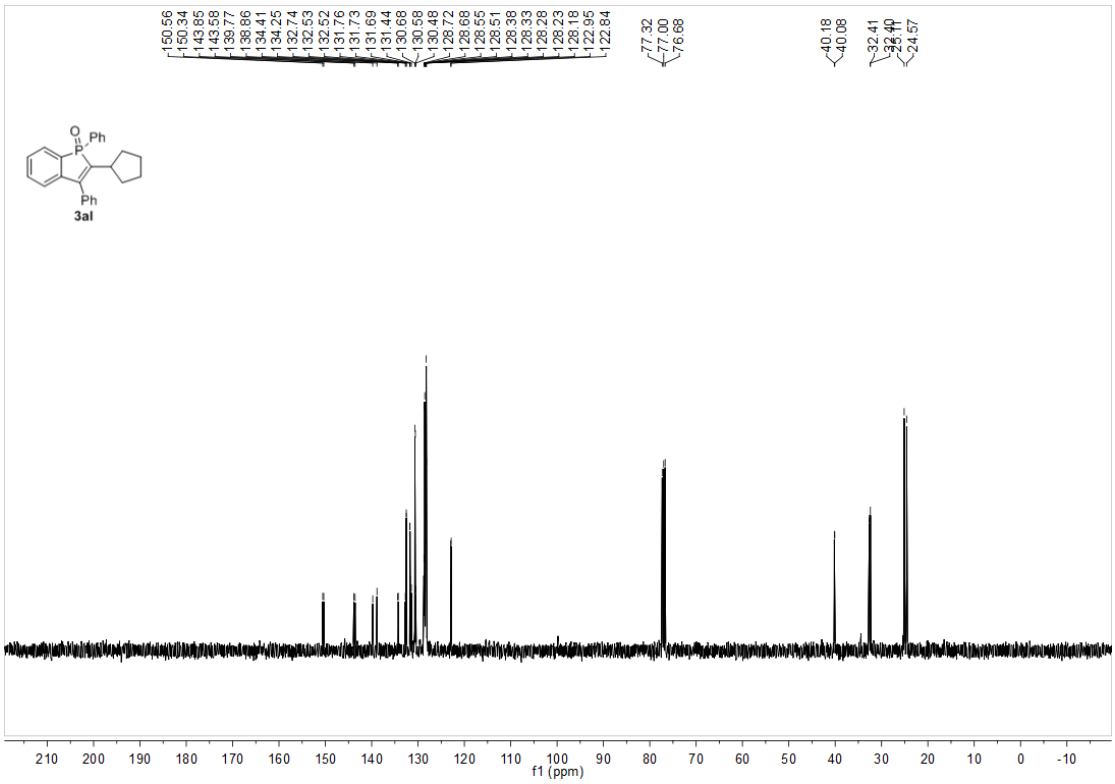
4.17
0.97
1.00
1.11
1.10
1.17
5.12
f1 (ppm)


38.97
Ph
Ph
3al
f1 (ppm)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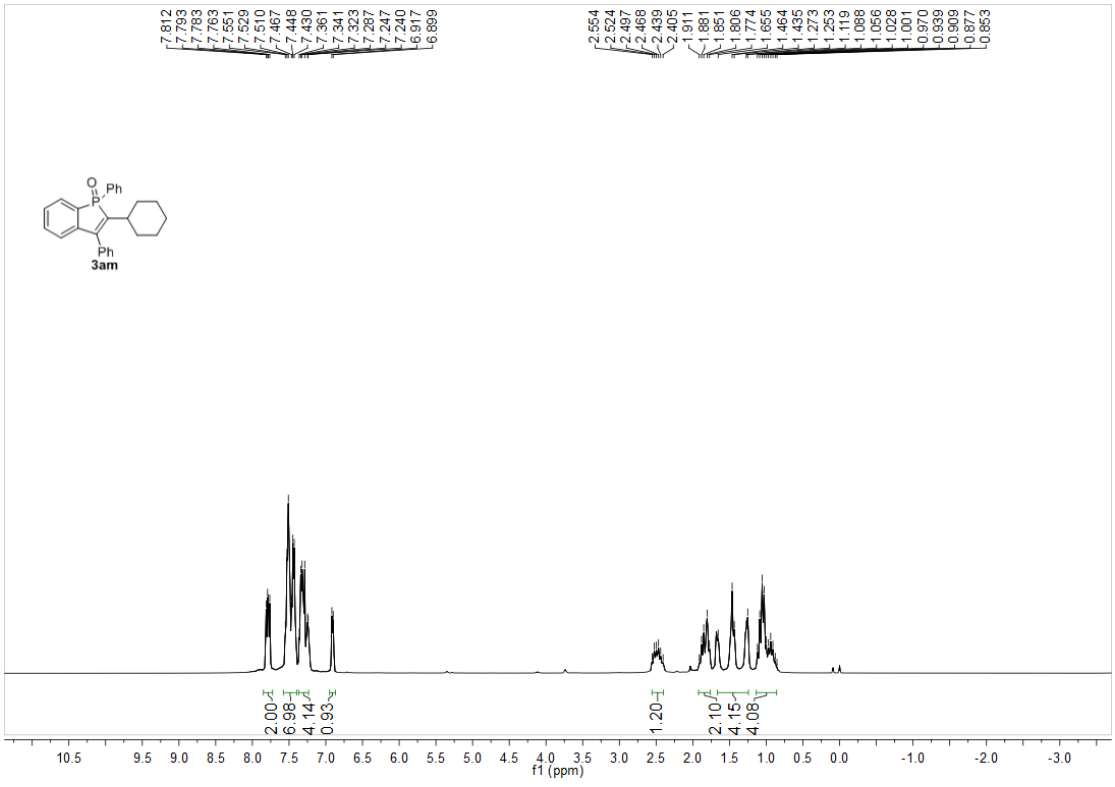
131.69
131.44
130.68
130.58
130.48
128.72
128.68
128.55
128.51
128.38
128.33
128.28
128.23
128.18
122.95
122.84
77.32
77.00
76.68
40.18
40.08
32.41
32.40
24.57
O
P
Ph
Ph
3al
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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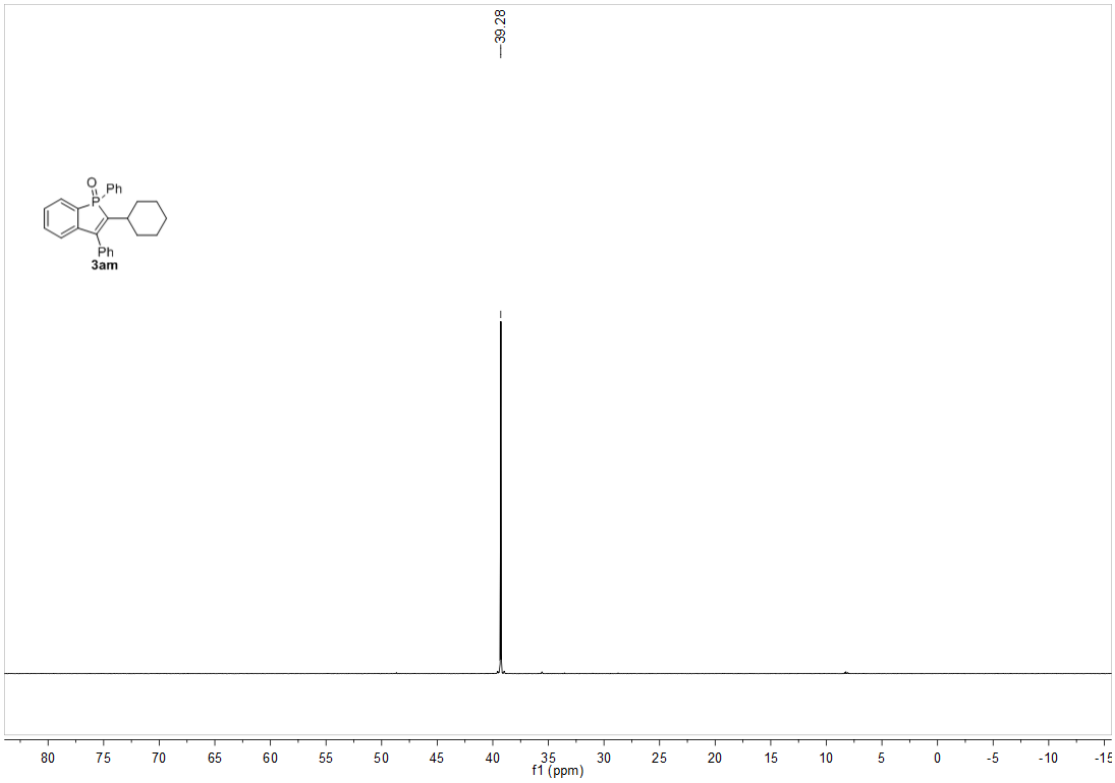
7.361
7.341
7.323
7.287
7.247
7.240
6.917
6.899
2.554
2.524
2.497
2.468
2.439
2.405
1.911
1.881
1.851
1.806
1.774
1.655
1.464
1.435
1.273
1.253
1.119
1.088
1.056
1.028
1.001
0.970
0.939
0.909
0.877
0.853
O
P
Ph
Ph
3am
2.00
6.98
4.14
0.93
1.20
2.10
4.15
4.08
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0 -2.0 -3.0
f1 (ppm)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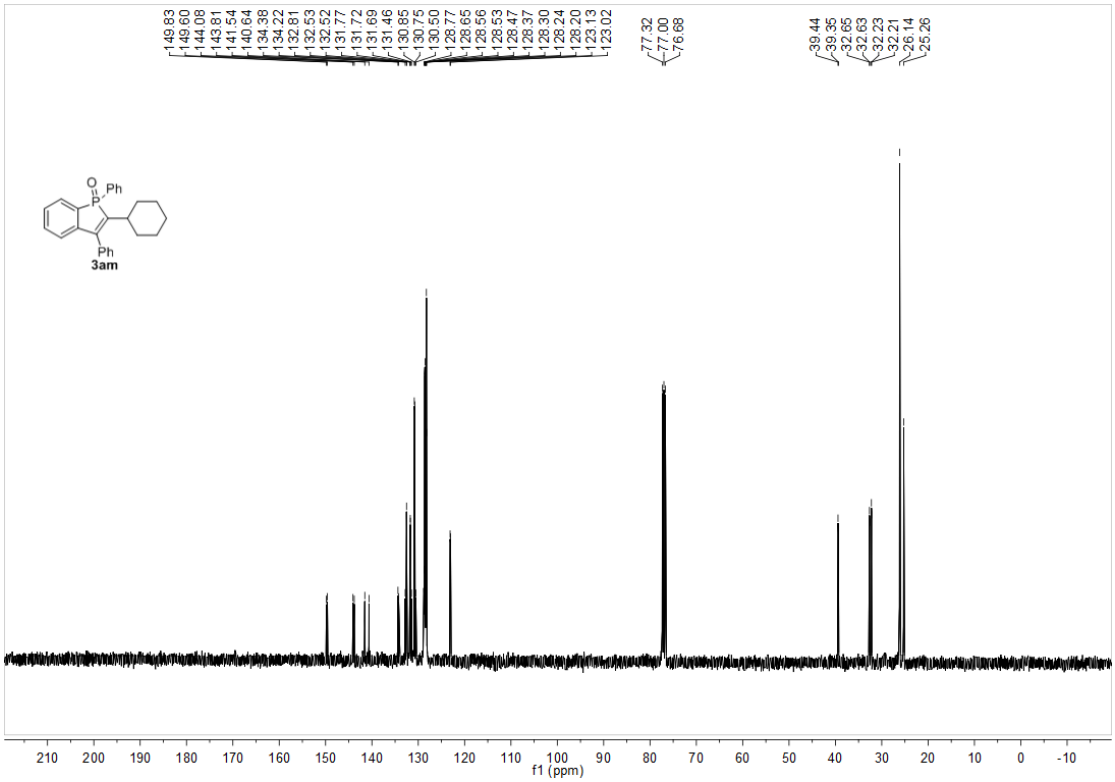
Ph
Ph
3am
f1 (ppm)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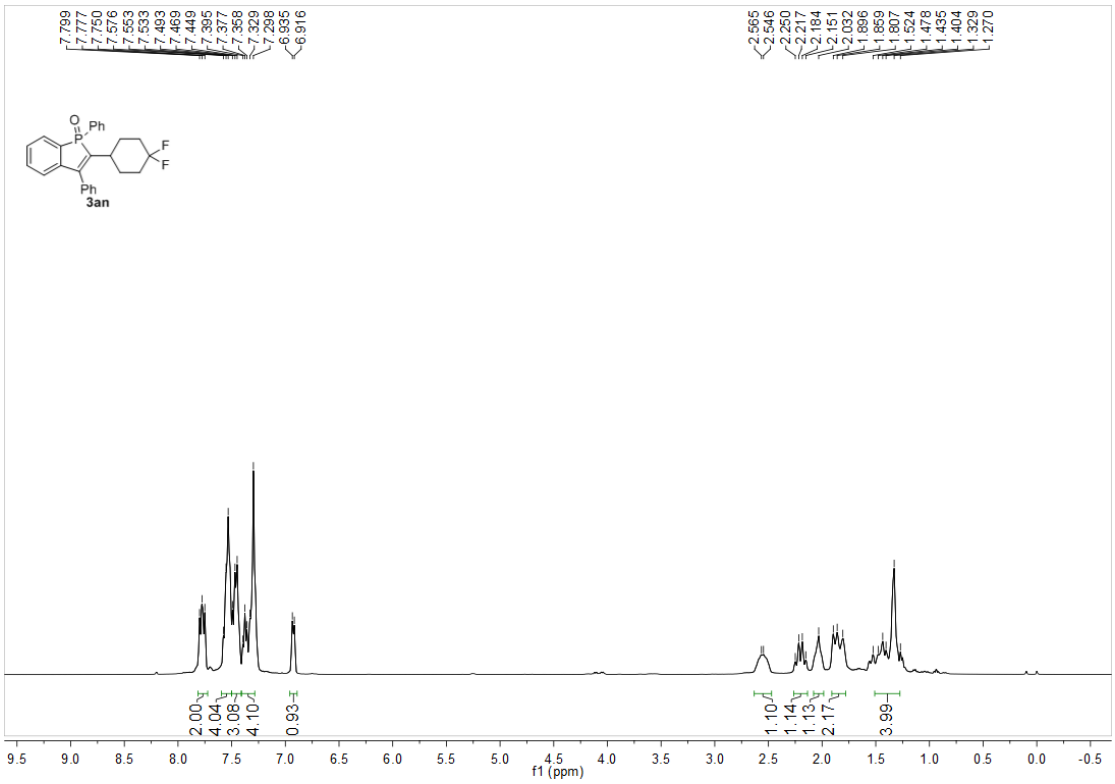
131.72
131.69
131.46
130.85
130.75
130.50
128.77
128.65
128.56
128.53
128.47
128.37
128.30
128.24
128.20
123.13
123.02
77.32
77.00
76.68
39.44
39.35
32.65
32.63
32.23
32.21
26.14
25.26
Ph
Ph
3am
f1 (ppm)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7.329
7.298
6.935
6.916
2.565
2.546
2.250
2.217
2.184
2.151
2.032
1.896
1.859
1.807
1.524
1.478
1.435
1.404
1.329
1.270
Ph
O
P
F
F
Ph
3an
2.00
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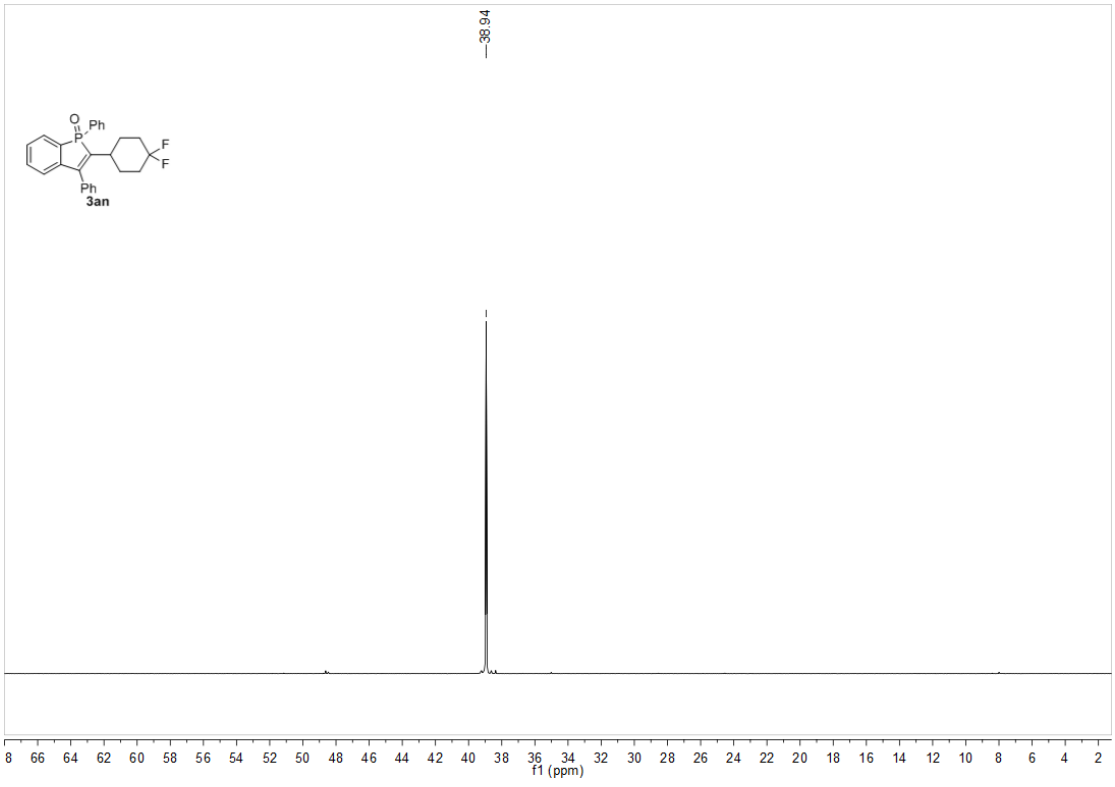
3.08
4.10
0.93
1.10
1.14
1.13
2.17
3.99
f1 (ppm)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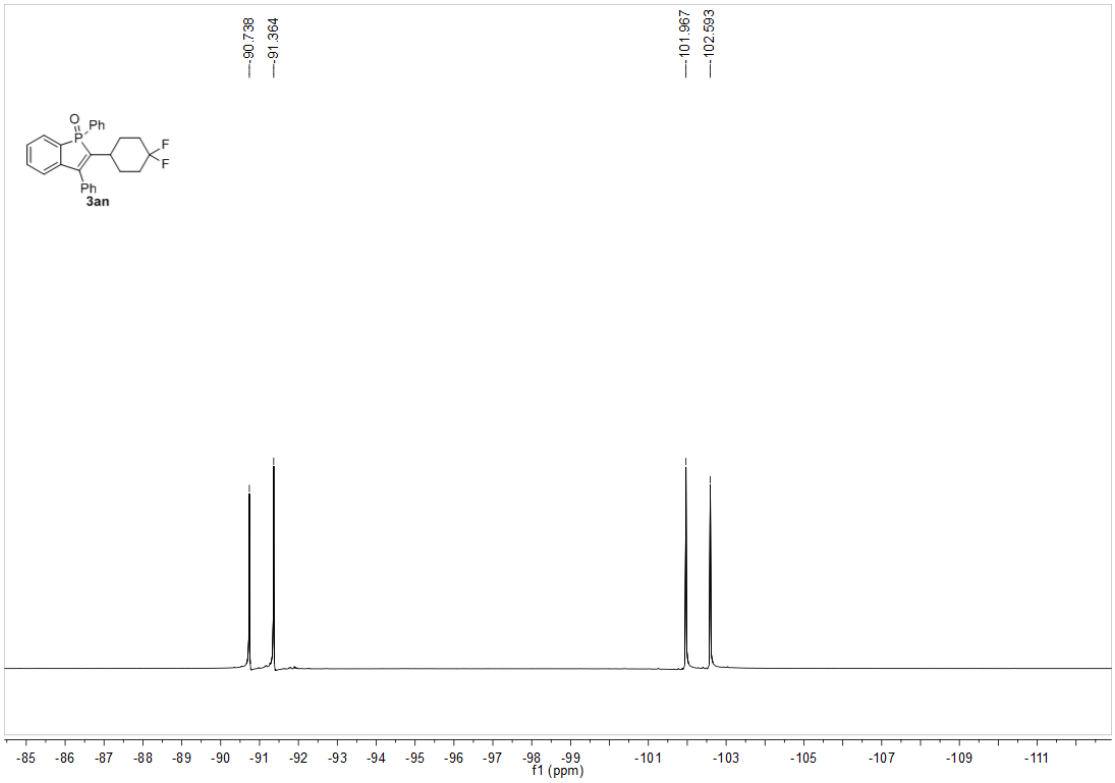
Ph
O
P
F
F
Ph
3an
f1 (ppm)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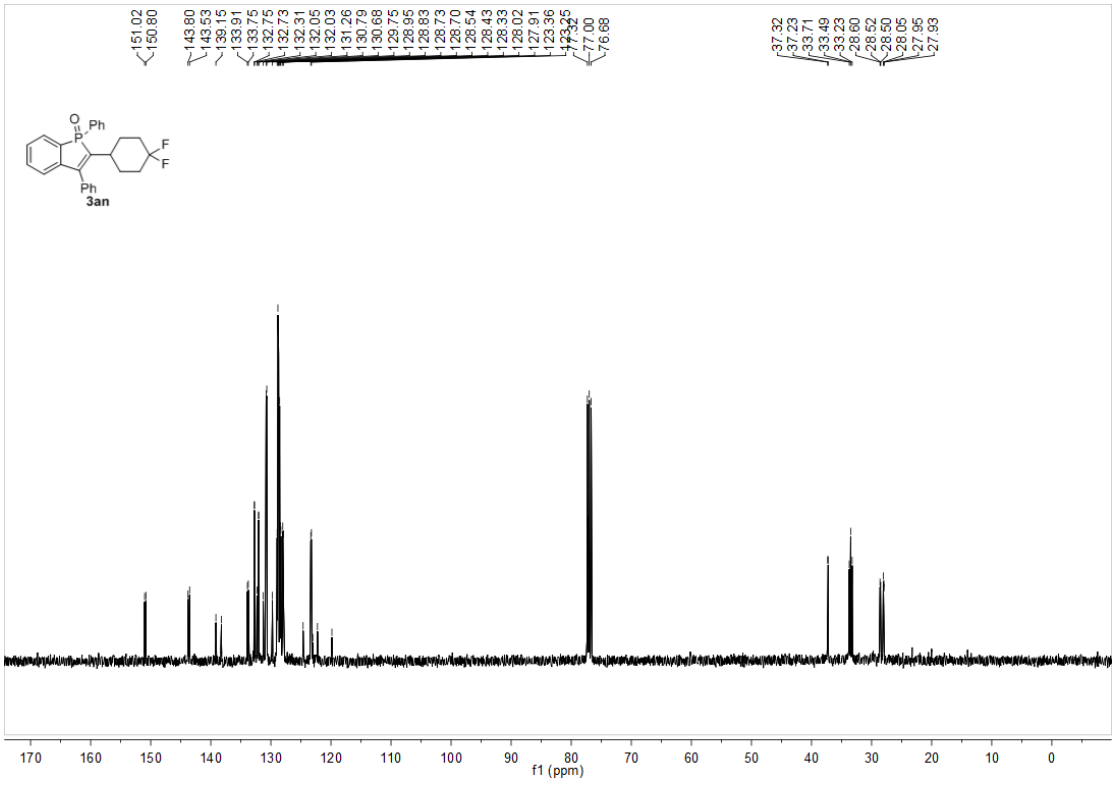
91.364
101.967
102.593
O
P
Ph
F
F
Ph
3an
-85 -86 -87 -88 -89 -90 -91 -92 -93 -94 -95 -96 -97 -98 -99 -101 -103 -105 -107 -109 -111
f1 (ppm)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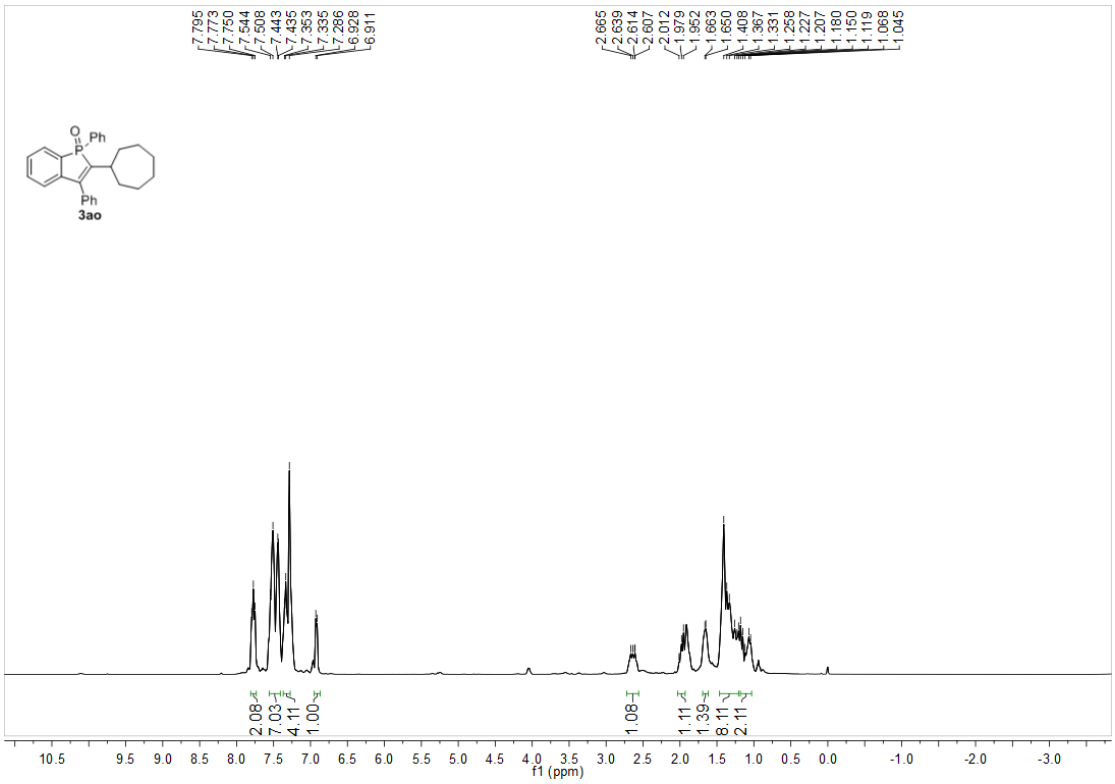
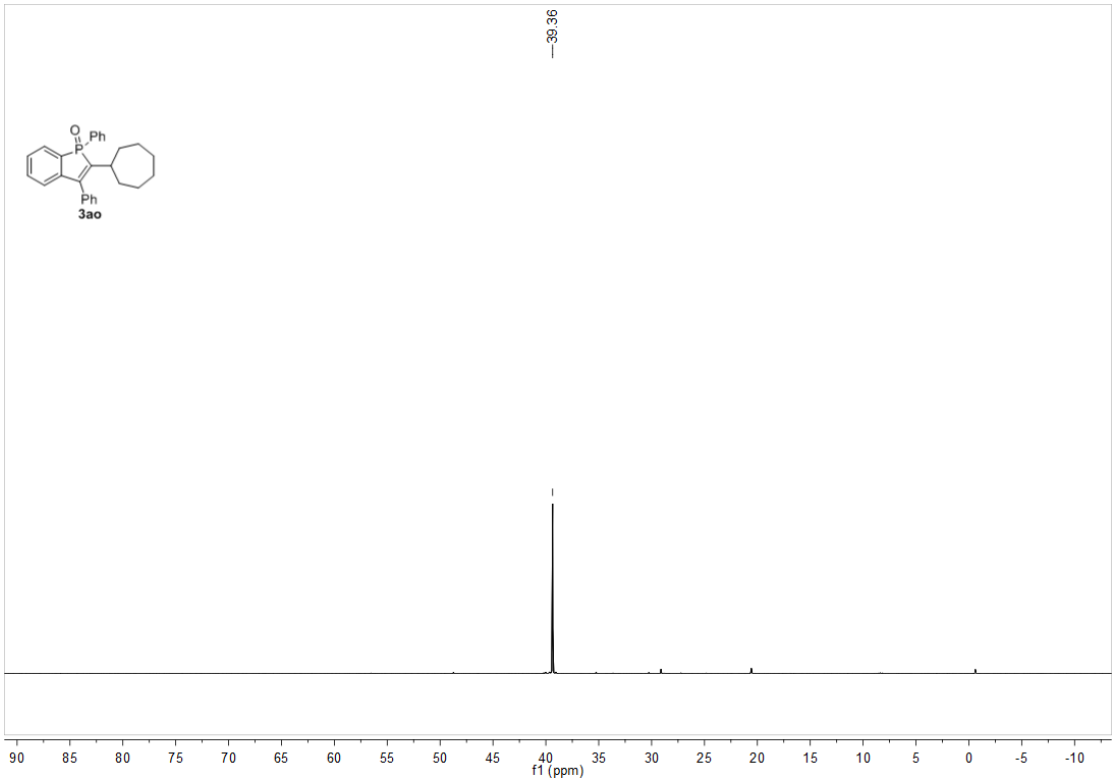
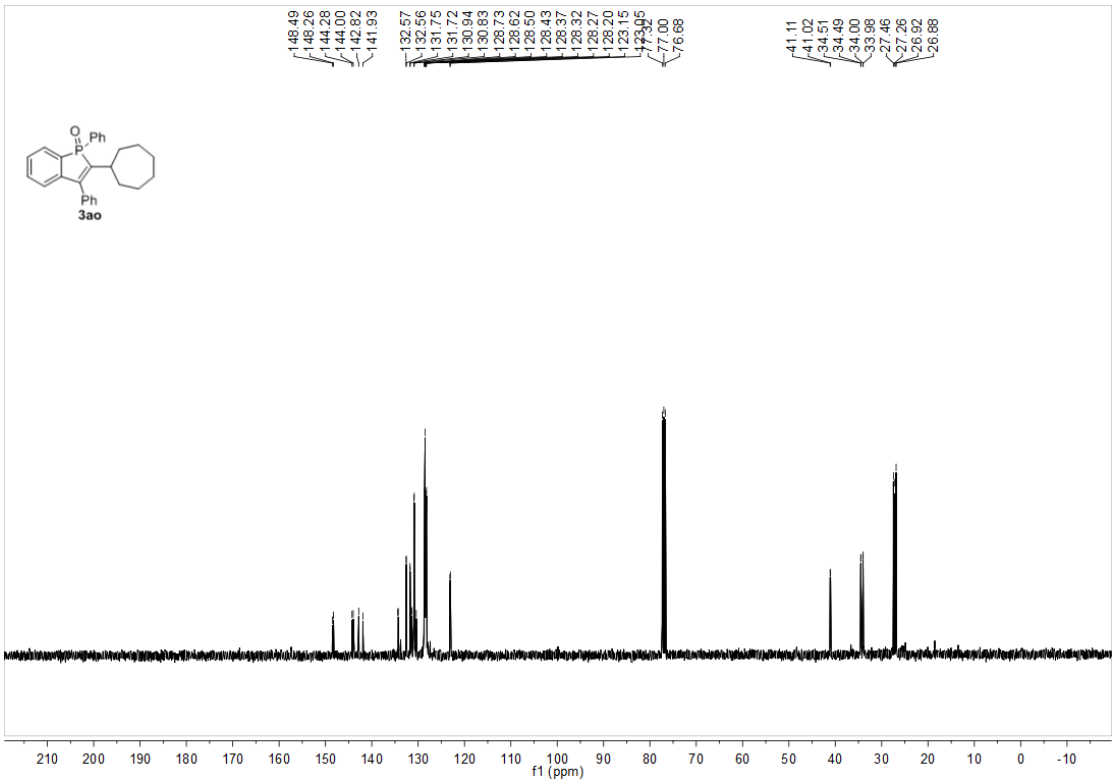
33.49
33.23
28.60
28.52
28.50
28.05
27.95
27.93
O
P
Ph
F
F
Ph
3an
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.795
7.773
7.750
7.544
7.508
7.443
7.435
7.353
7.335
7.286
6.928
6.911
2.665
2.639
2.614
2.607
2.012
1.979
1.952
1.663
1.650
1.408
1.367
1.331
1.258
1.227
1.207
1.180
1.150
1.119
1.068
1.045
O
P
Ph
Ph
3ao
2.08
7.03
4.11
1.00
1.08
1.11
1.39
8.11
2.11
f1 (ppm)


39.36
O
P
Ph
Ph
3ao
f1 (ppm)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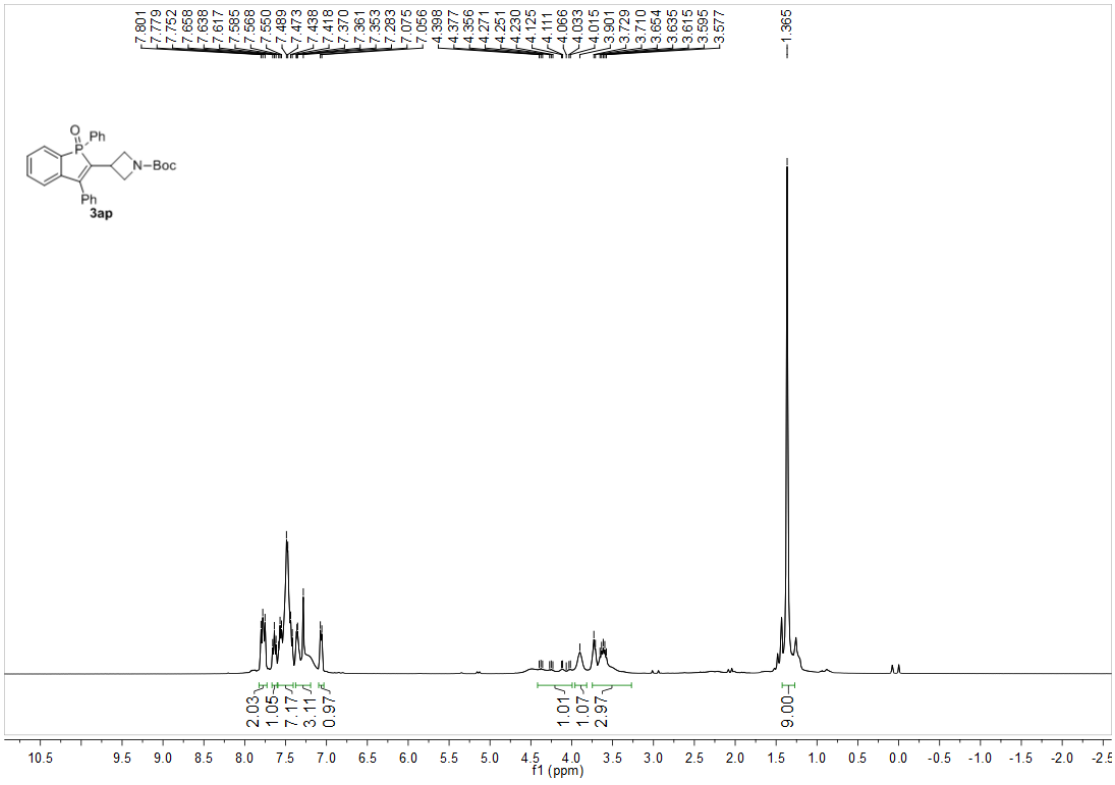
148.26
144.28
144.00
142.82
141.93
132.57
132.56
131.75
131.72
130.94
130.83
128.73
128.62
128.50
128.43
128.37
128.32
128.27
128.20
123.15
77.32
77.00
76.68
41.11
41.02
34.51
34.49
34.00
33.98
27.46
27.26
26.92
26.88
Ph
Ph
3ao
f1 (ppm)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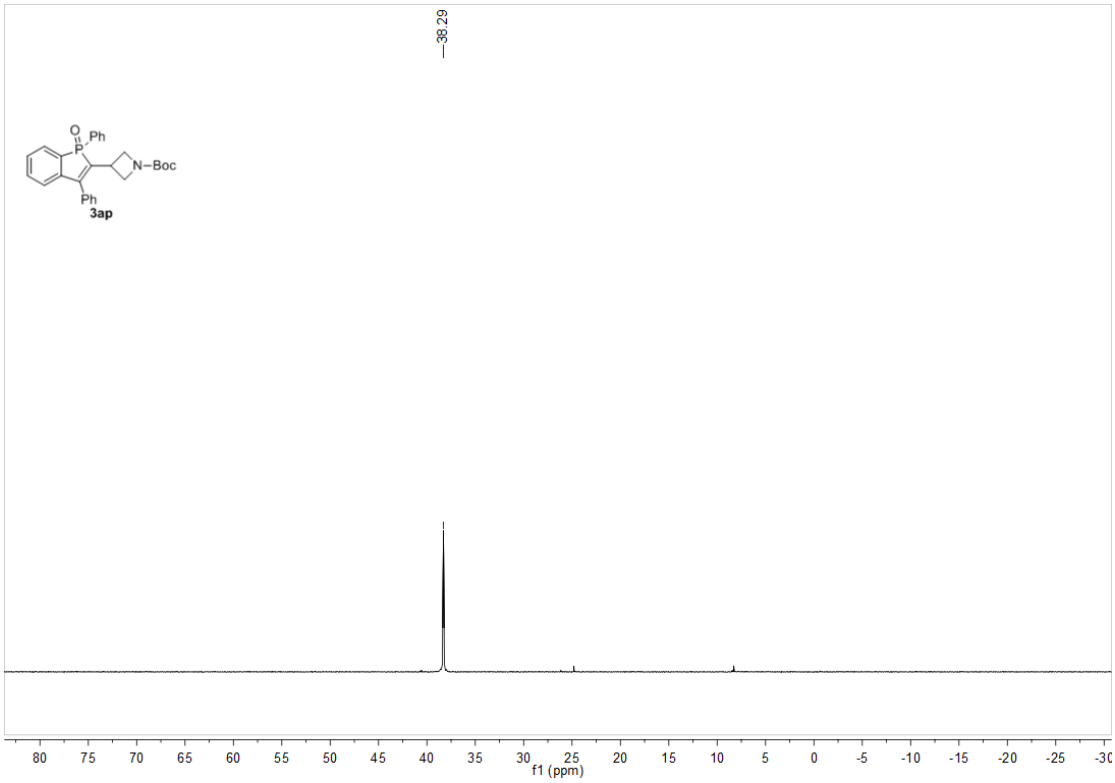
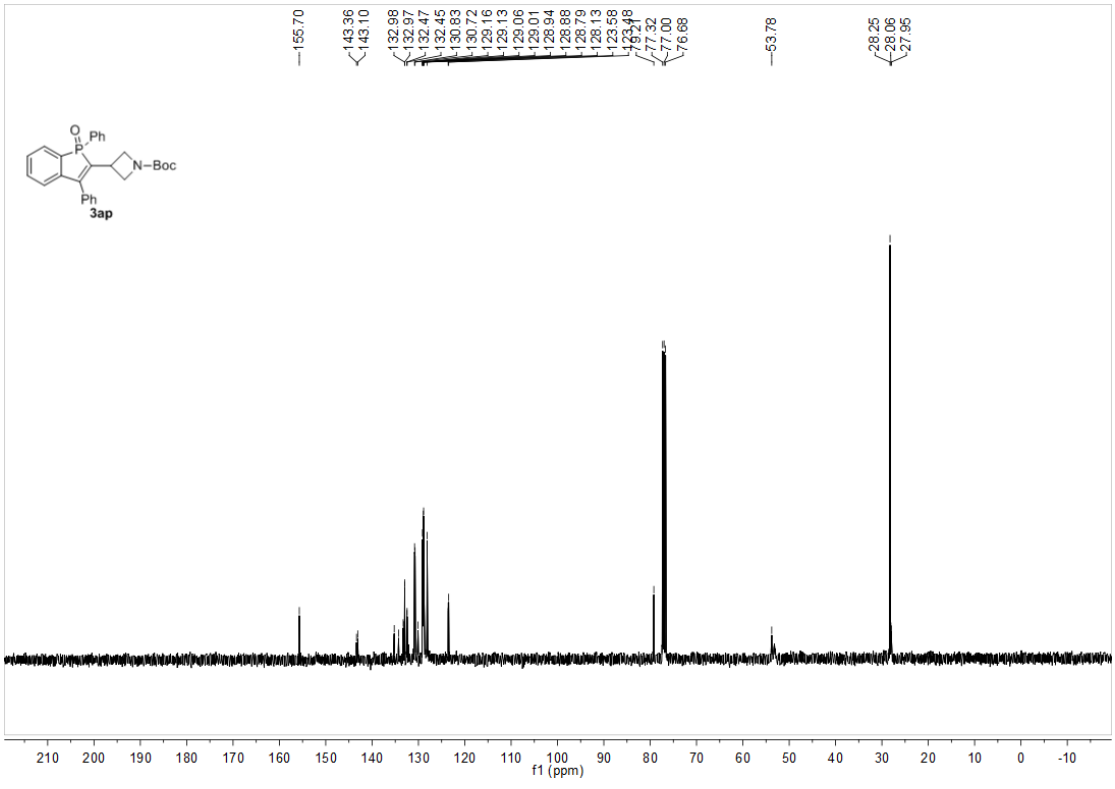
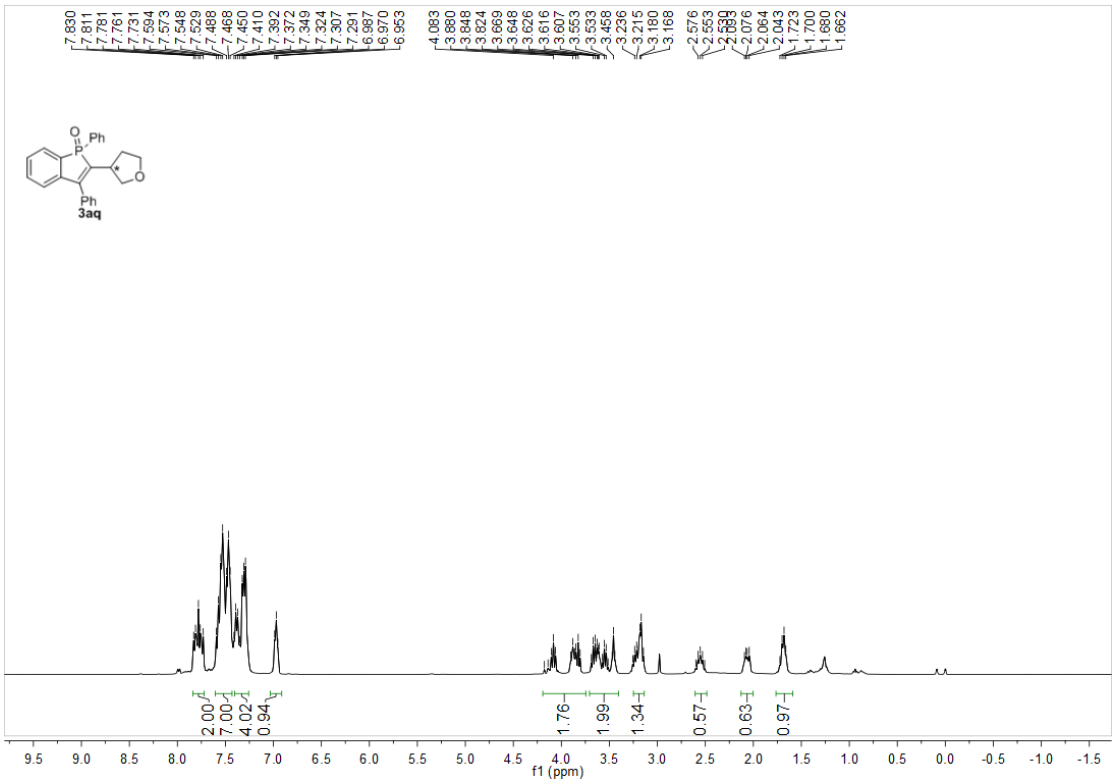
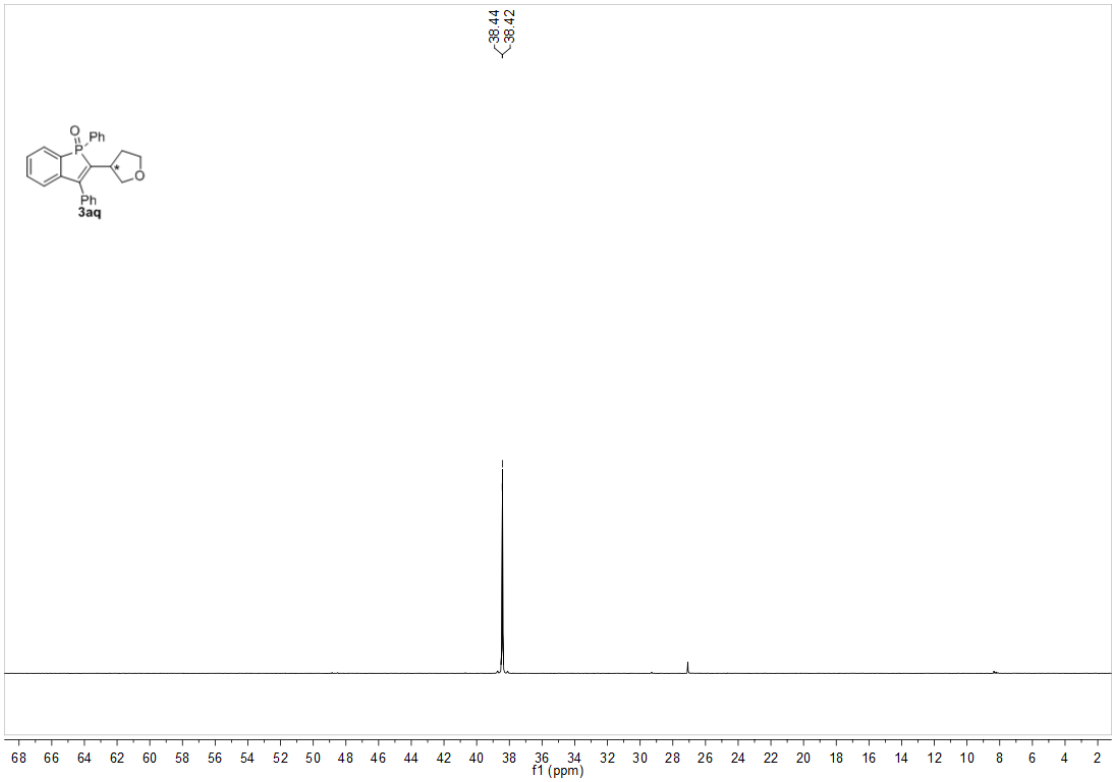
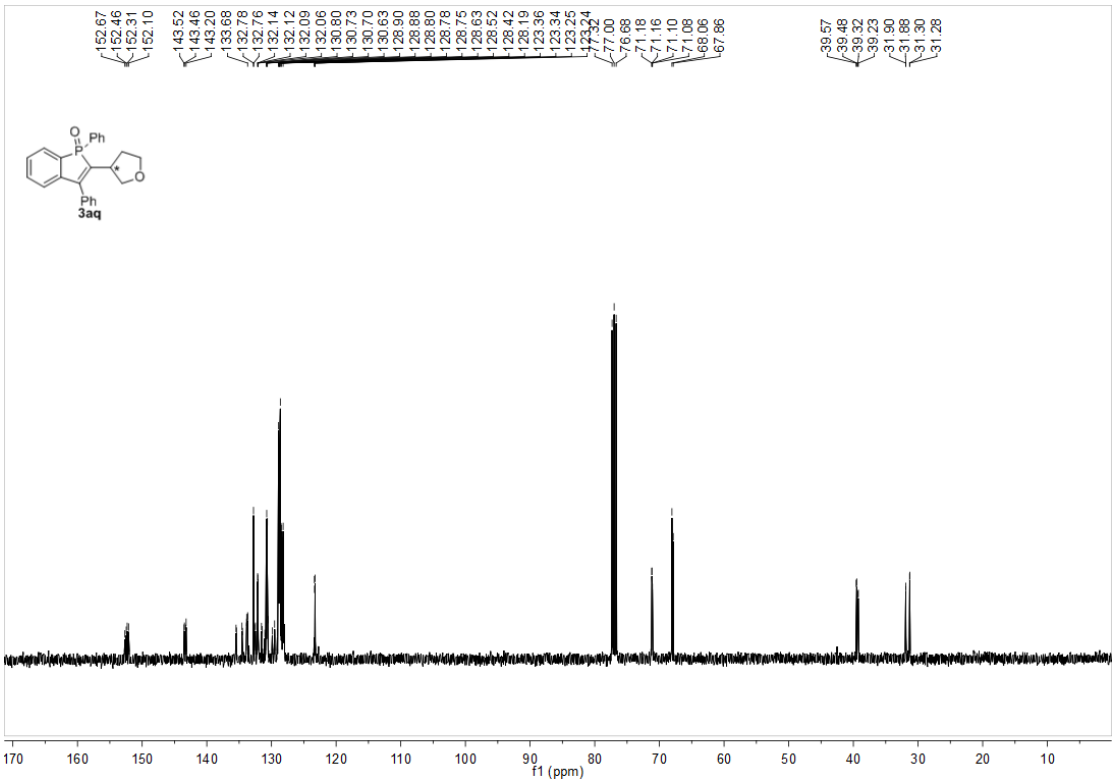
7.752
7.658
7.638
7.617
7.585
7.568
7.550
7.489
7.473
7.438
7.418
7.370
7.361
7.353
7.283
7.075
7.056
4.398
4.377
4.356
4.271
4.251
4.230
4.125
4.111
4.066
4.033
4.015
3.901
3.729
3.710
3.654
3.635
3.615
3.595
3.577
1.365
Ph
N–Boc
Ph
3ap
2.03
1.05
7.17
3.11
0.97
1.01
1.07
2.97
9.00
f1 (ppm)

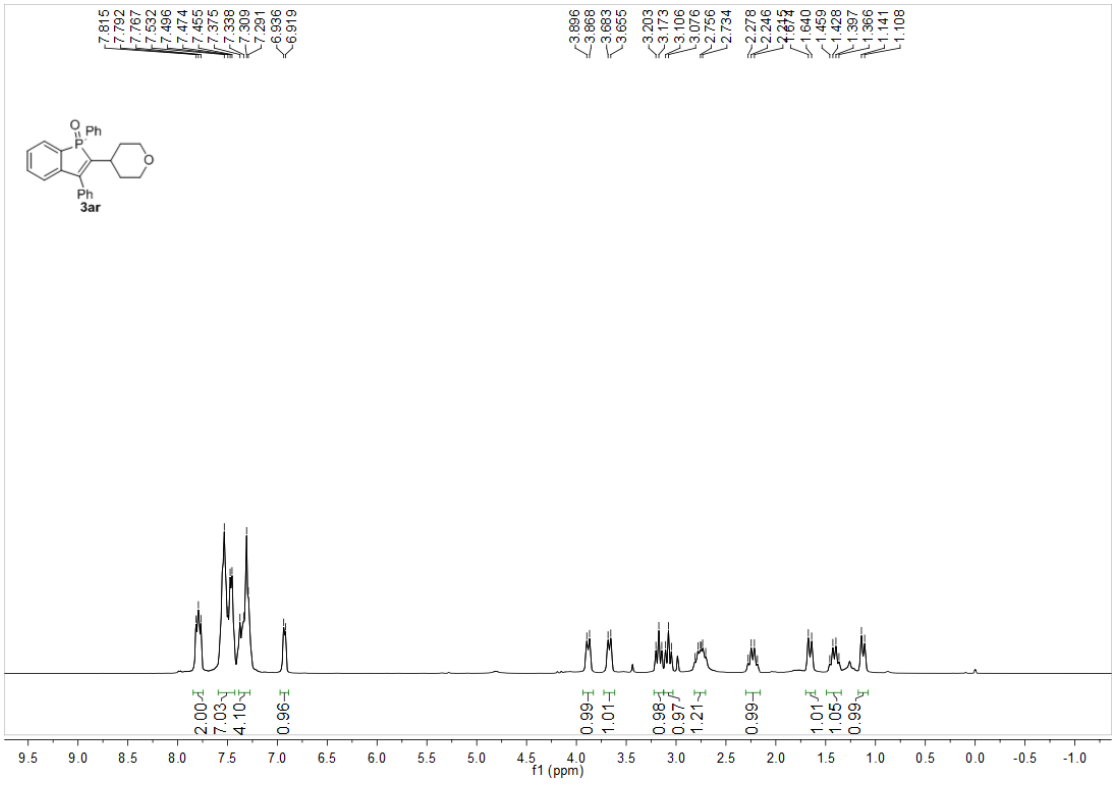

38.29
O
Ph
P
N–Boc
Ph
3ap
80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30
f1 (ppm)

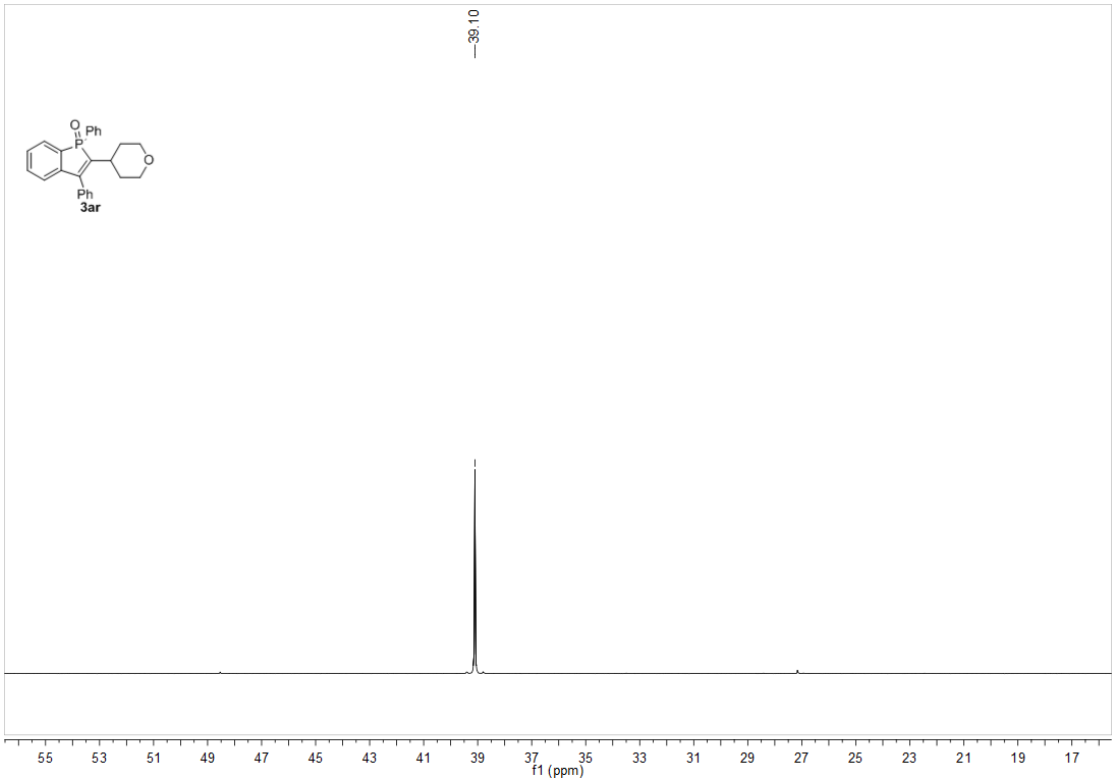

155.70
143.36
143.10
132.98
132.97
132.47
132.45
130.83
130.72
129.16
129.13
129.06
129.01
128.94
128.88
128.79
128.13
123.58
123.48
79.21
77.32
77.00
76.68
53.78
28.25
28.06
27.95
O
Ph
P
N–Boc
Ph
3ap
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

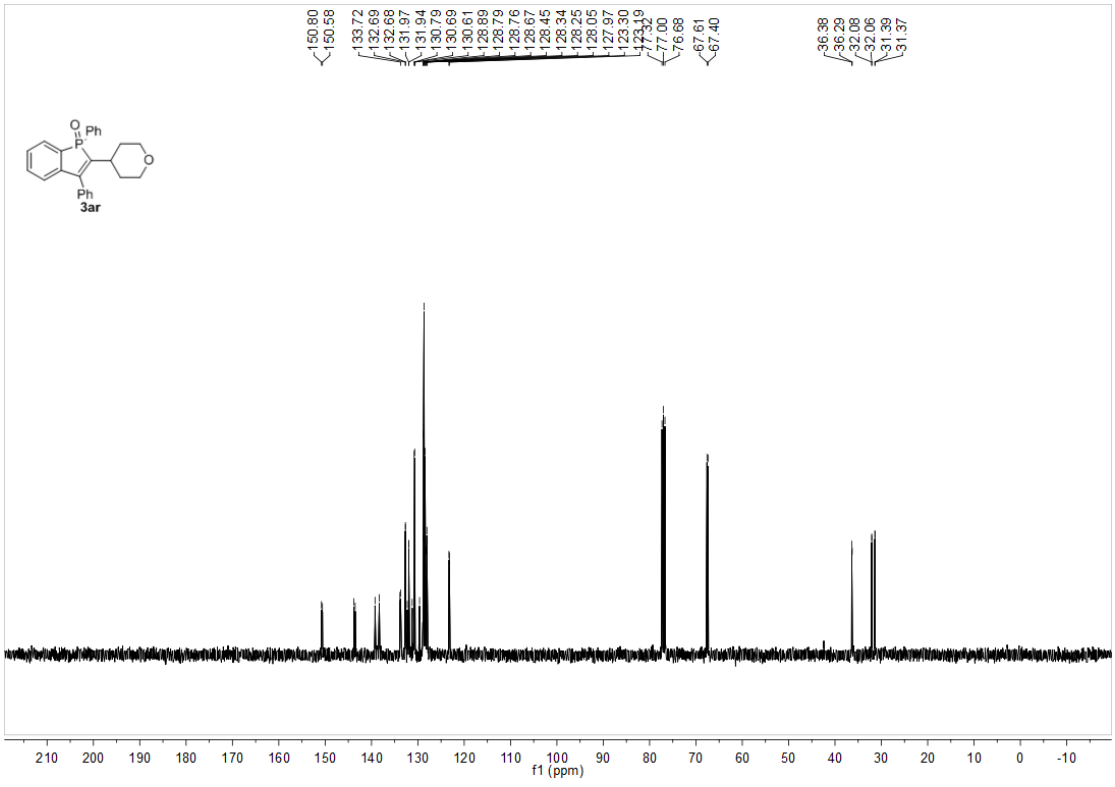

7.830
7.811
7.781
7.761
7.731
7.594
7.573
7.548
7.529
7.488
7.468
7.450
7.410
7.392
7.372
7.349
7.324
7.307
7.291
6.987
6.970
6.953
4.083
3.880
3.848
3.824
3.669
3.648
3.626
3.616
3.607
3.553
3.533
3.458
3.236
3.215
3.180
3.168
2.576
2.553
2.076
2.064
2.043
1.723
1.700
1.680
1.662
Ph
O
P
Ph
3aq
2.00
7.00
4.02
0.94
1.76
1.99
1.34
0.57
0.63
0.97
f1 (ppm)

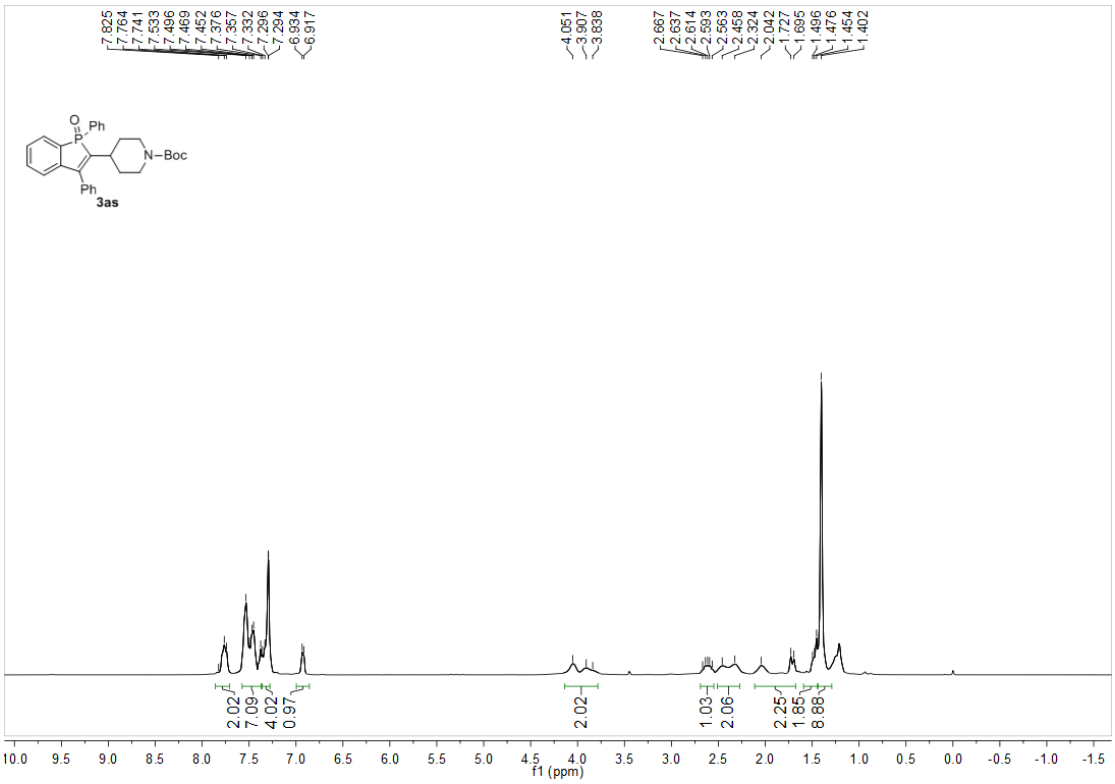

38.44
38.42
Ph
O
P
Ph
3aq
f1 (ppm)

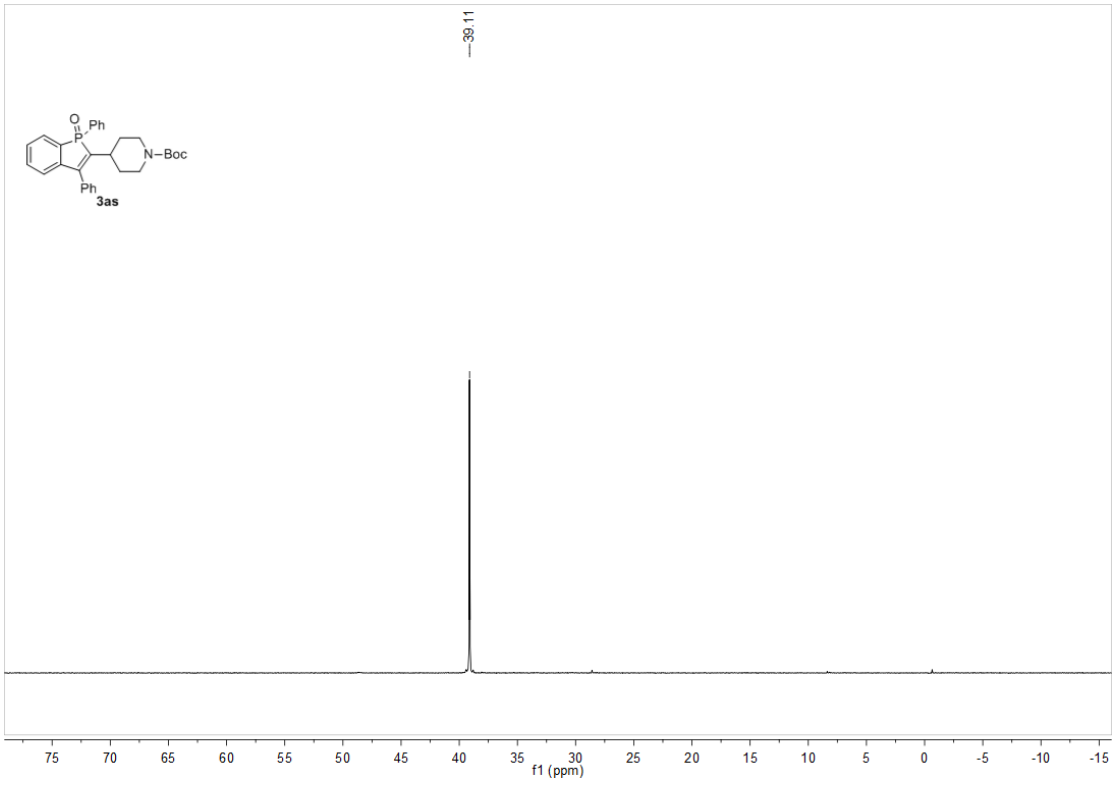

152.67
152.46
152.31
152.10
143.52
143.46
143.20
133.68
132.78
132.76
132.14
132.12
132.09
132.06
130.80
130.73
130.70
130.63
128.90
128.88
128.80
128.78
128.75
128.63
128.52
128.42
128.19
123.36
123.34
123.25
123.24
77.32
77.00
76.68
71.18
71.16
71.10
71.08
68.06
67.86
39.57
39.48
39.32
39.23
31.90
31.88
31.30
31.28
O
P
Ph
Ph
O
3aq
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

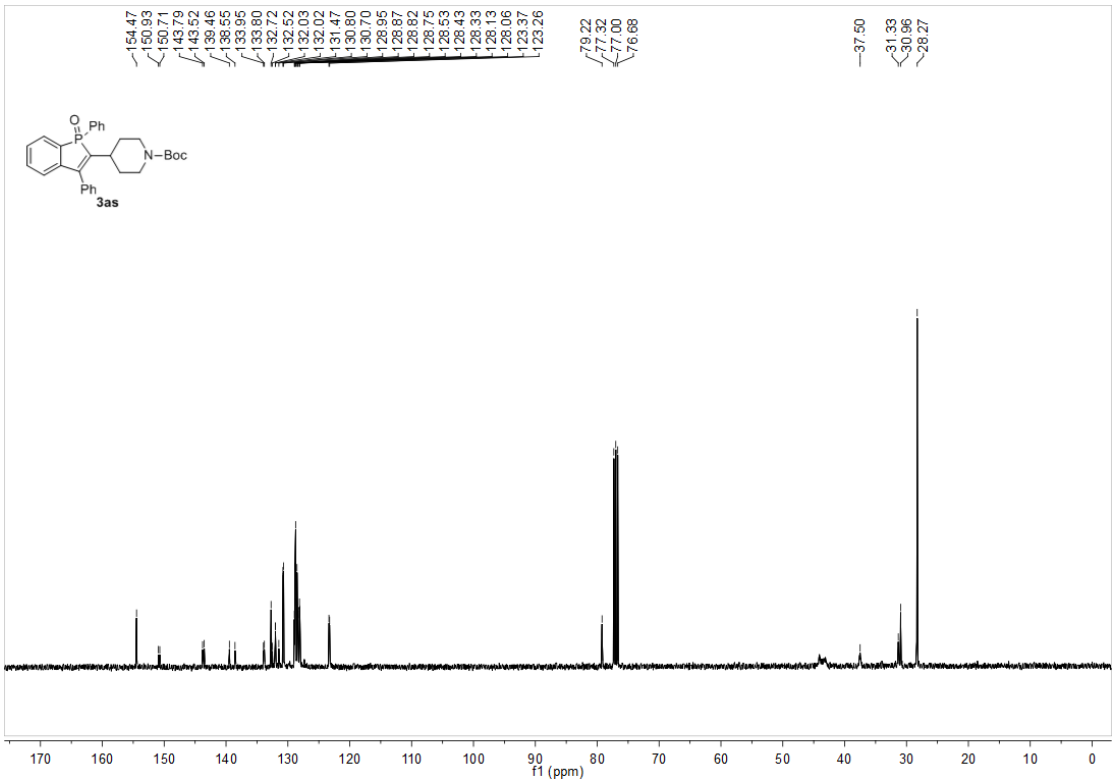

7.815
7.792
7.767
7.532
7.496
7.474
7.455
7.375
7.338
7.309
7.291
6.936
6.919
3.896
3.868
3.683
3.655
3.203
3.173
3.106
3.076
2.756
2.734
2.278
2.246
2.215
1.674
1.640
1.459
1.428
1.397
1.366
1.141
1.108
O
P
Ph
Ph
O
3ar
2.00
7.03
4.10
0.96
0.99
1.01
0.98
0.97
1.21
0.99
1.01
1.05
0.99
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


39.10
Ph
O
P
Ph
O
3ar
55 53 51 49 47 45 43 41 39 37 35 33 31 29 27 25 23 21 19 17
f1 (ppm)


150.80
150.58
133.72
132.69
132.68
131.97
131.94
130.79
130.69
130.61
128.89
128.79
128.76
128.67
128.45
128.34
128.25
128.05
127.97
123.30
123.19
77.32
77.00
76.68
67.61
67.40
36.38
36.29
32.08
32.06
31.39
31.37
Ph
O
P
Ph
O
3ar
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.825
7.764
7.741
7.533
7.496
7.469
7.452
7.376
7.357
7.332
7.296
7.294
6.934
6.917
4.051
3.907
3.838
2.667
2.637
2.614
2.593
2.563
2.458
2.324
2.042
1.727
1.695
1.496
1.476
1.454
1.402
O
P
Ph
N–Boc
Ph
3as
2.02
7.09
4.02
0.97
2.02
1.03
2.06
2.25
1.85
8.88
f1 (ppm)


39.11
O
P
Ph
N–Boc
Ph
3as
f1 (ppm)


154.47
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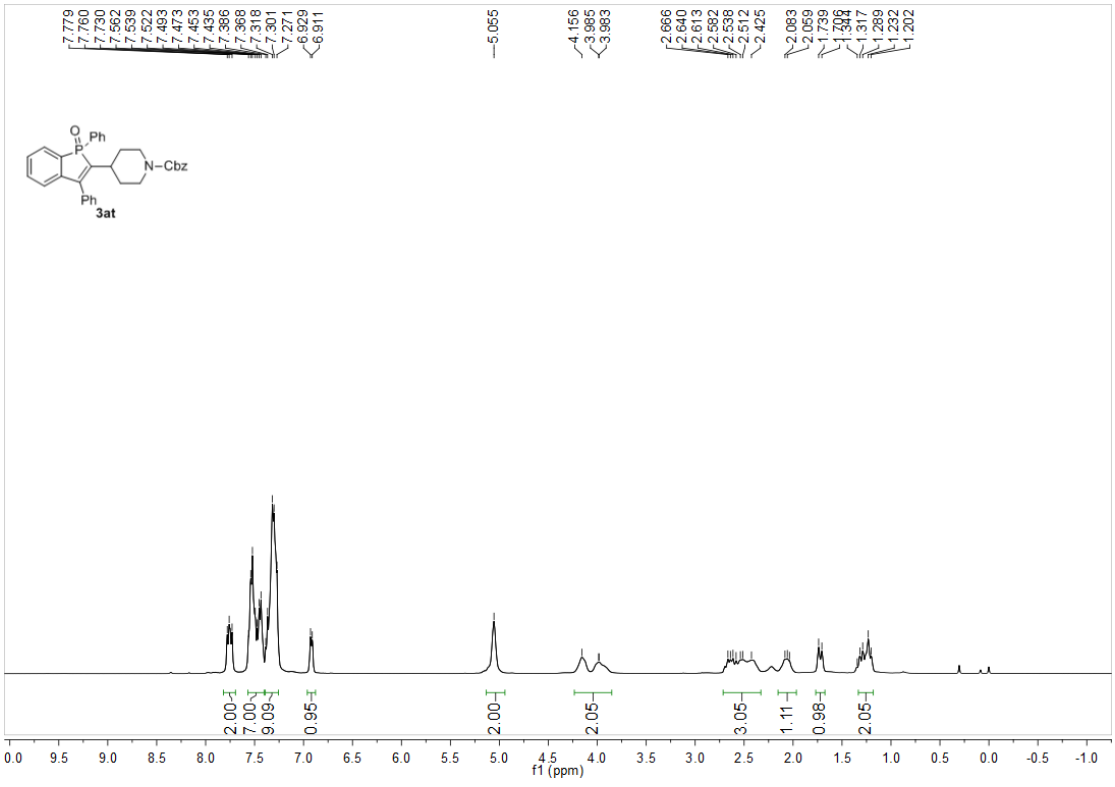
150.71
143.79
143.52
139.46
138.55
133.95
133.80
132.72
132.52
132.03
132.02
131.47
130.80
130.70
128.95
128.87
128.82
128.75
128.53
128.43
128.33
128.13
128.06
123.37
123.26
79.22
77.32
77.00
76.68
37.50
31.33
30.96
28.27
O
P
Ph
N–Boc
Ph
3as
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.779
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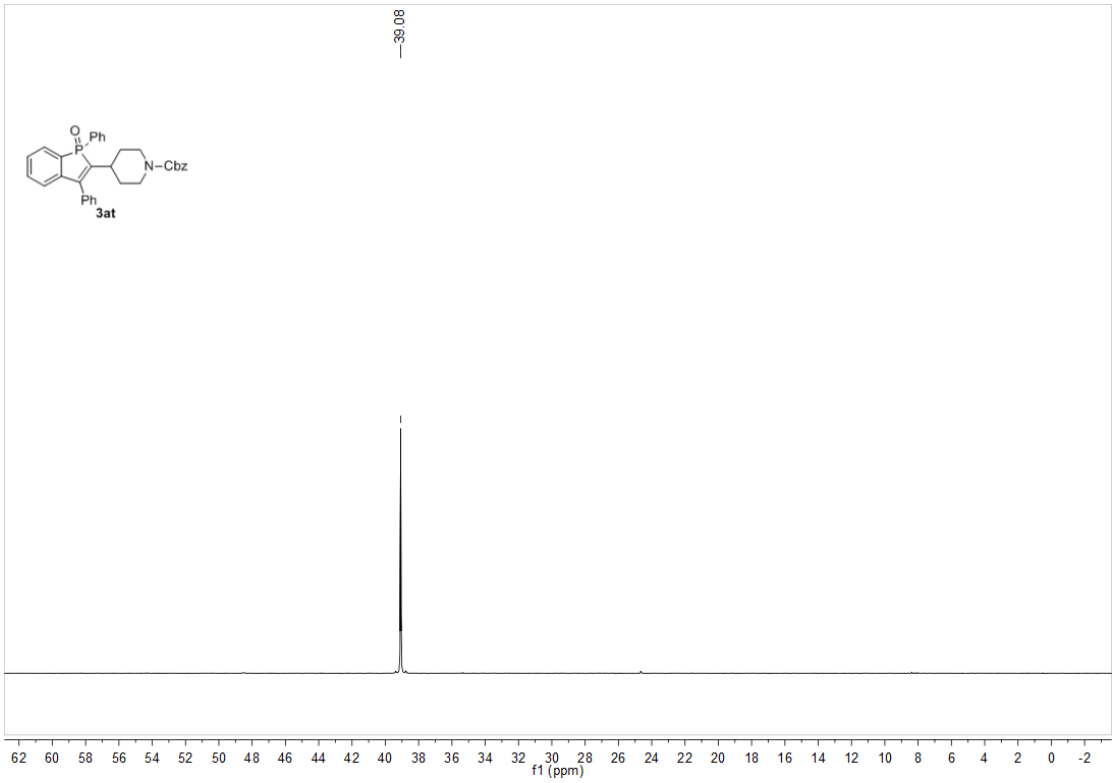
7.730
7.562
7.539
7.522
7.493
7.473
7.453
7.435
7.386
7.368
7.318
7.301
7.271
6.929
6.911
5.055
4.156
3.985
3.983
2.666
2.640
2.613
2.582
2.538
2.512
2.425
2.083
2.059
1.739
1.706
1.344
1.317
1.289
1.232
1.202
O
P
Ph
N–Cbz
Ph
3at
2.00
7.00
9.09
0.95
2.00
2.05
3.05
1.11
0.98
2.05
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


39.08
O
Ph
P
N–Cbz
Ph
3at
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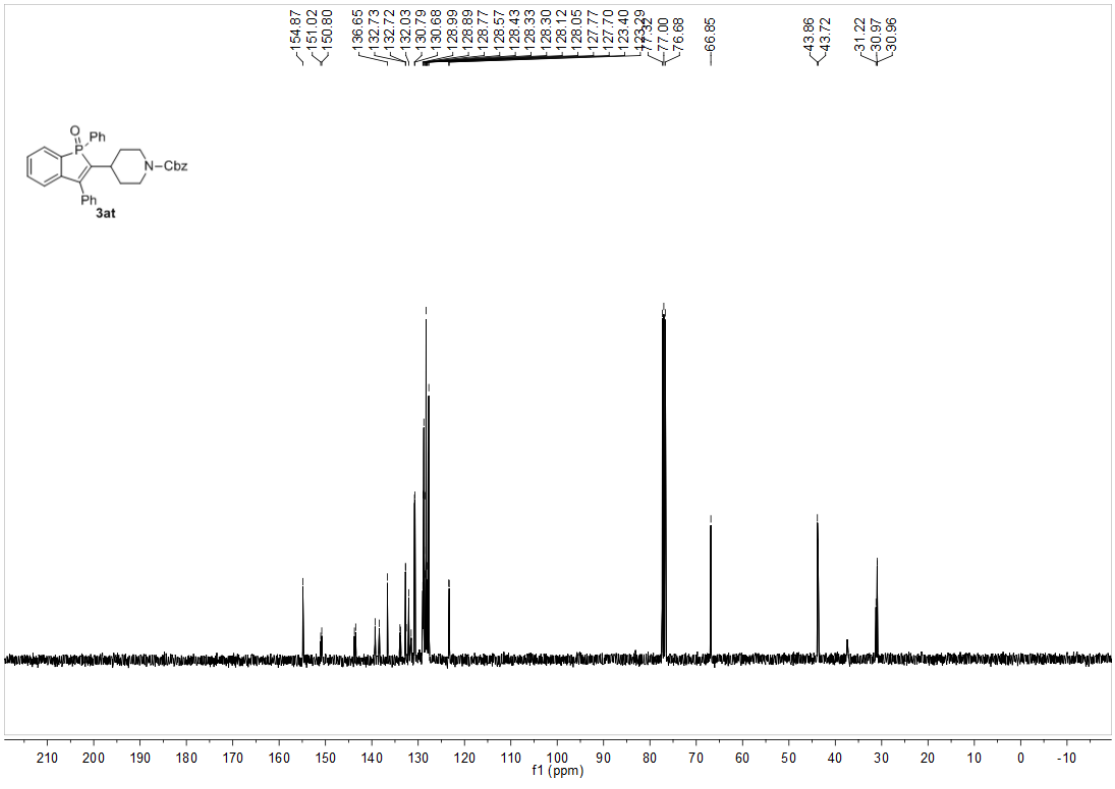
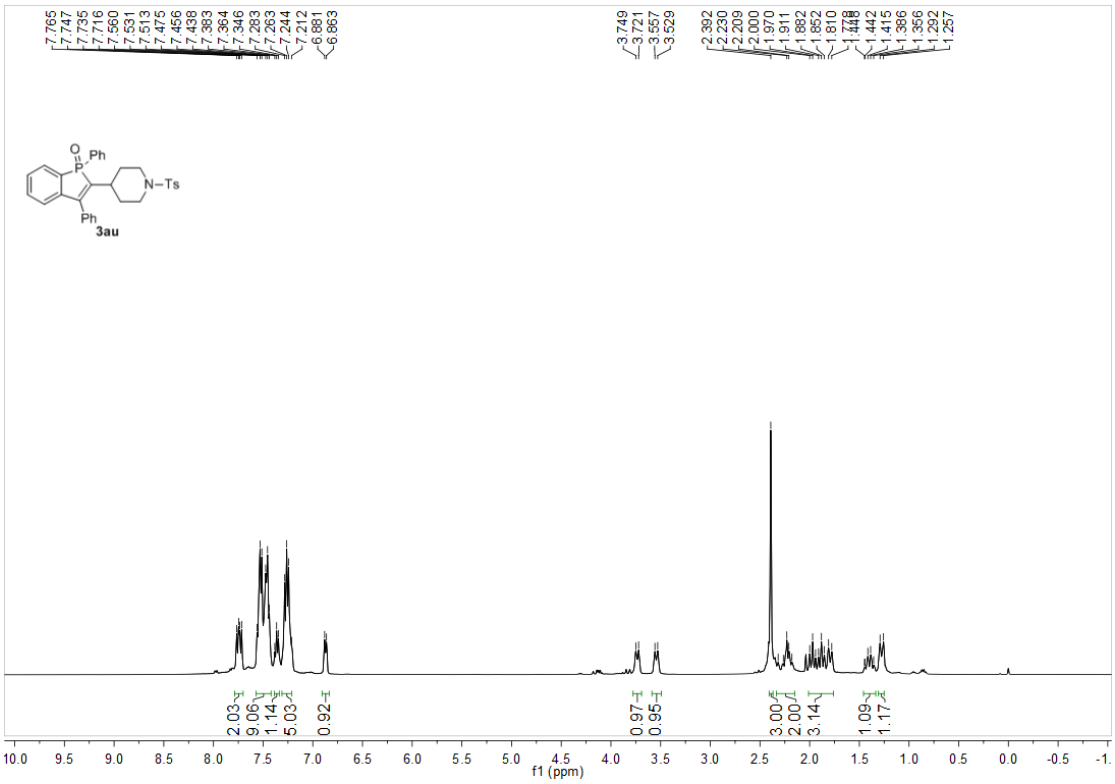
f1 (ppm)


154.87
151.02
150.80
136.65
132.73
132.72
132.03
130.79
130.68
128.99
128.89
128.77
128.57
128.43
128.33
128.30
128.12
128.05
127.77
127.70
123.40
123.29
77.32
77.00
76.68
66.85
43.86
43.72
31.22
30.97
30.96
O
Ph
P
N–Cbz
Ph
3at
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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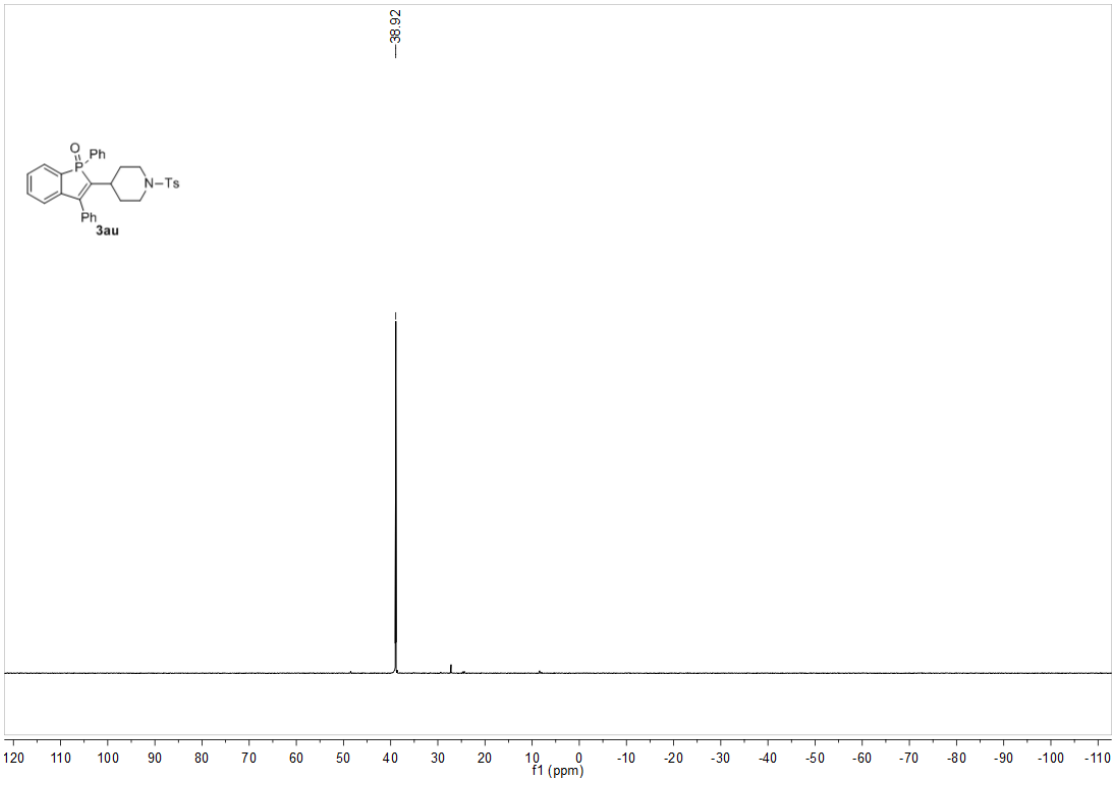
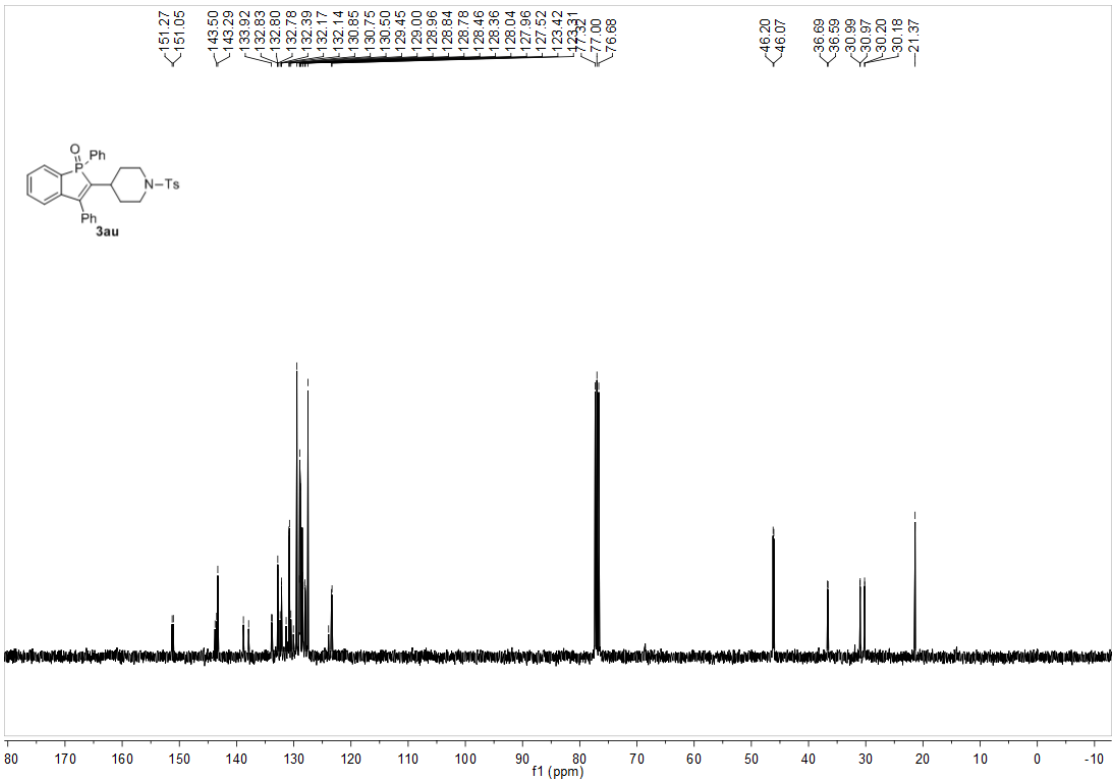
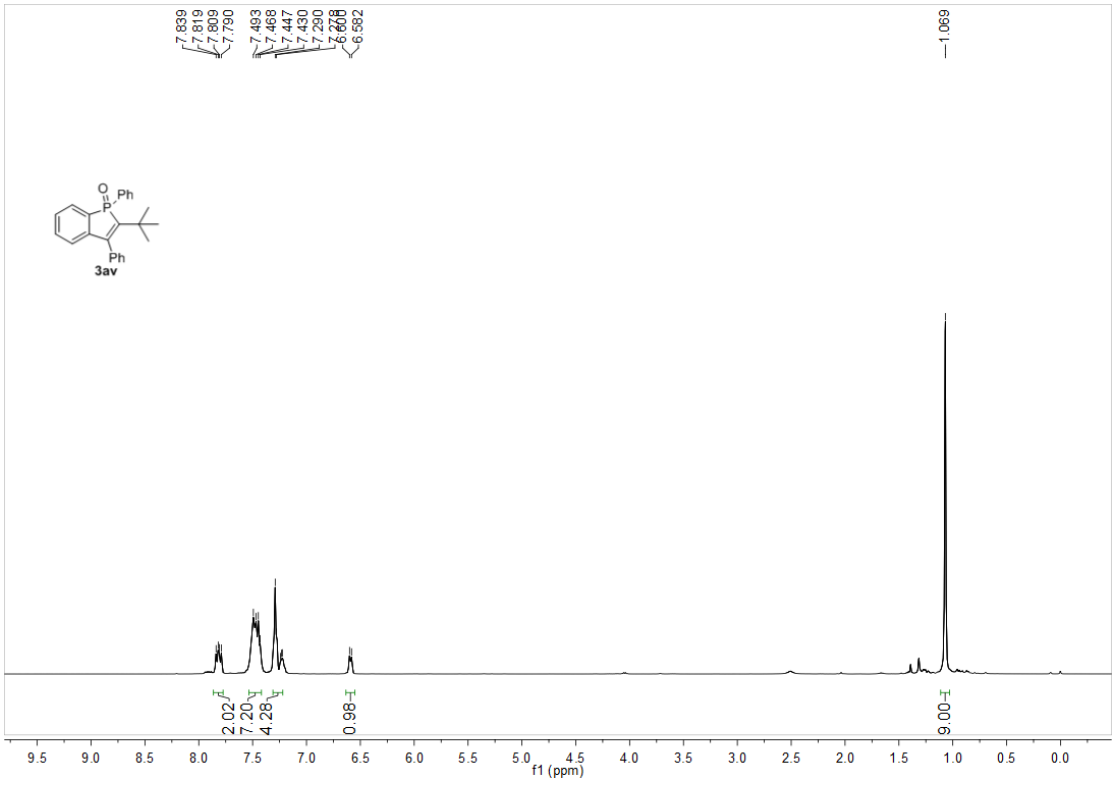
7.735
7.716
7.560
7.531
7.513
7.475
7.456
7.438
7.383
7.364
7.346
7.283
7.263
7.244
7.212
6.881
6.863
3.749
3.721
3.557
3.529
2.392
2.230
2.209
2.000
1.970
1.911
1.882
1.852
1.810
1.442
1.415
1.386
1.356
1.292
1.257
O
P
Ph
N–Ts
Ph
3au
2.03
9.06
1.14
5.03
0.92
0.97
0.95
3.00
2.00
3.14
1.09
1.17
f1 (ppm)


38.92
O
P
Ph
N–Ts
Ph
3au
f1 (ppm)


151.27
151.05
143.50
143.29
133.92
132.83
132.80
132.78
132.39
132.17
132.14
130.85
130.75
130.50
129.45
129.00
128.96
128.84
128.78
128.46
128.36
128.04
127.96
127.52
123.42
123.31
77.32
77.00
76.68
46.20
46.07
36.69
36.59
30.99
30.97
30.20
30.18
21.37
O
P
Ph
N–Ts
Ph
3au
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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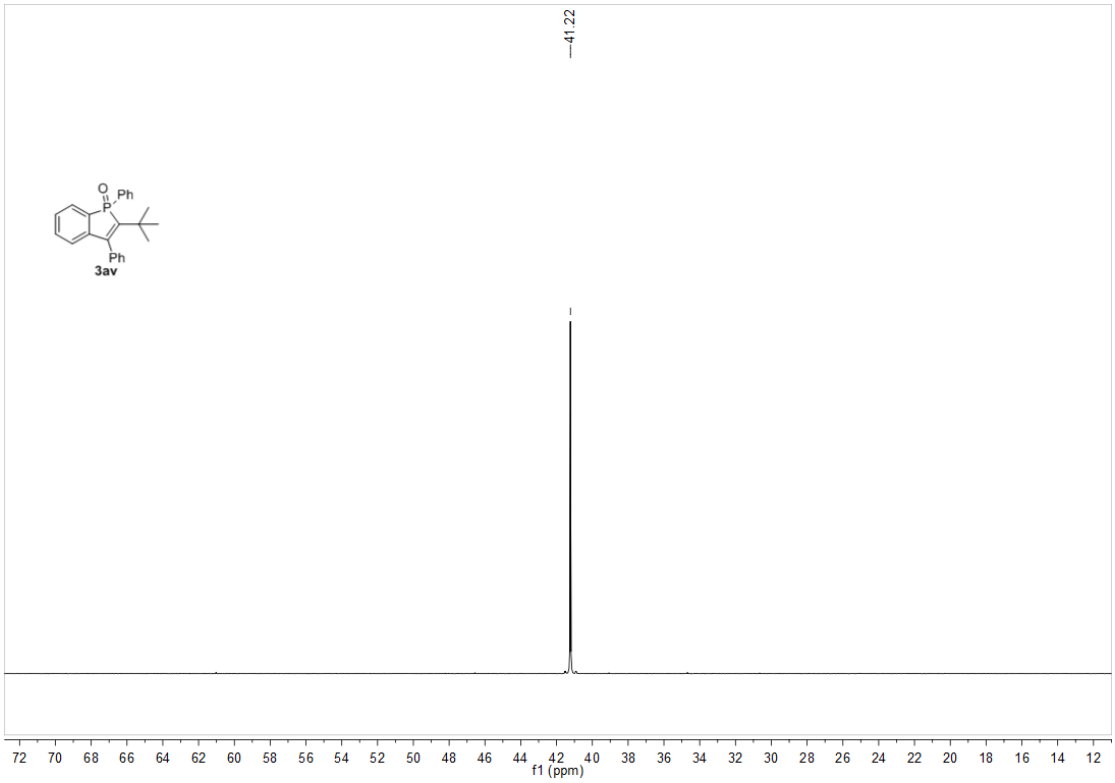
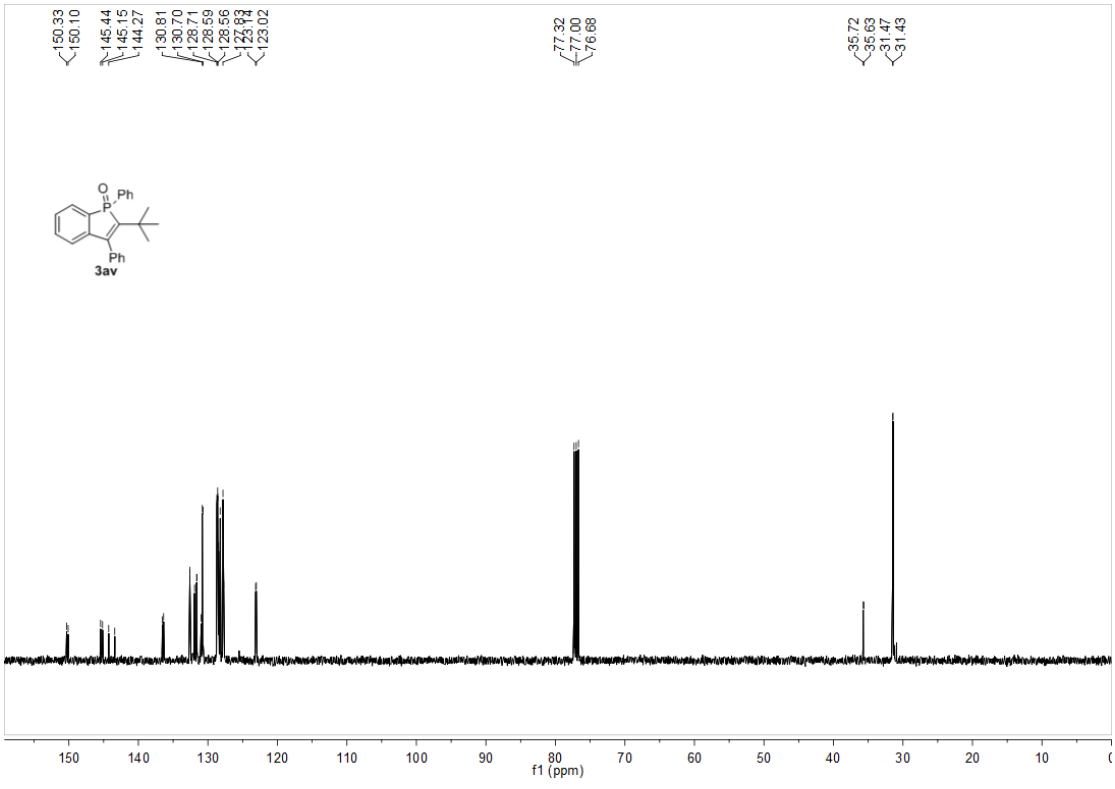
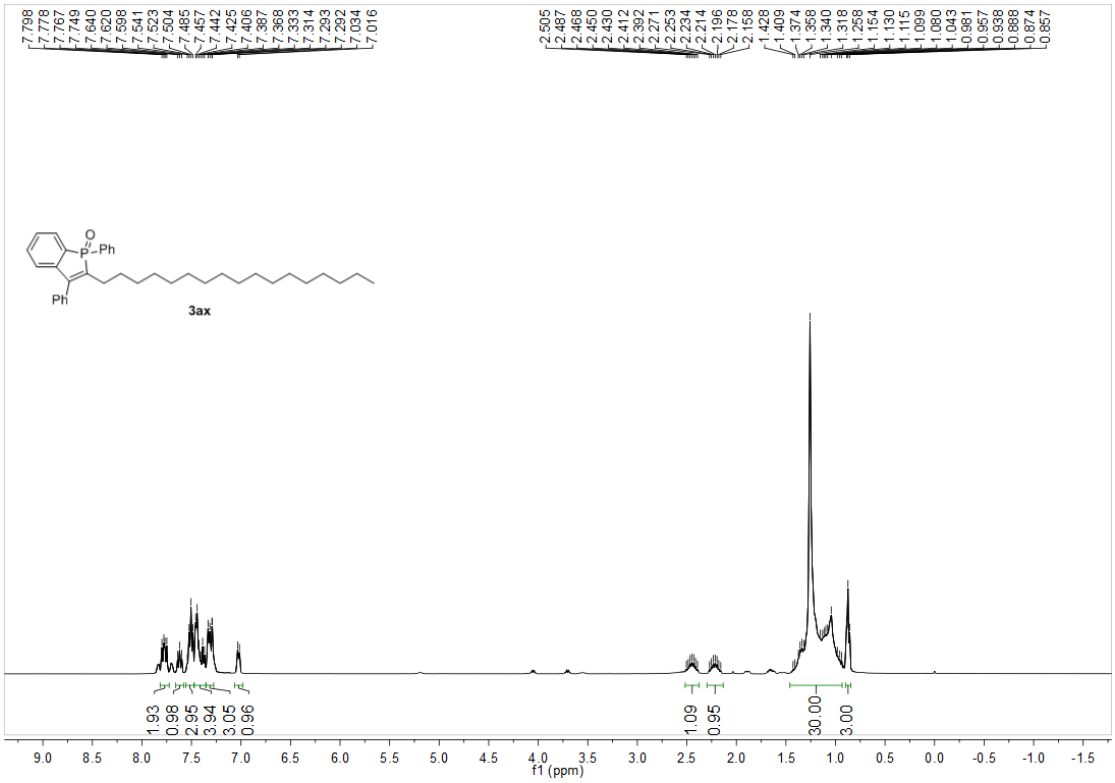
7.809
7.790
7.493
7.468
7.447
7.430
7.290
6.600
6.582
1.069
O
P
Ph
Ph
3av
2.02
7.20
4.28
0.98
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


41.22
Ph
Ph
3av
72 70 68 66 64 62 60 58 56 54 52 50 48 46 44 42 40 38 36 34 32 30 28 26 24 22 20 18 16 14 12
f1 (ppm)


150.33
150.10
145.44
145.15
144.27
130.81
130.70
128.71
128.59
128.56
127.83
123.14
123.02
77.32
77.00
76.68
35.72
35.63
31.47
31.43
Ph
Ph
3av
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.798
7.778
7.767
7.749
7.640
7.620
7.598
7.541
7.523
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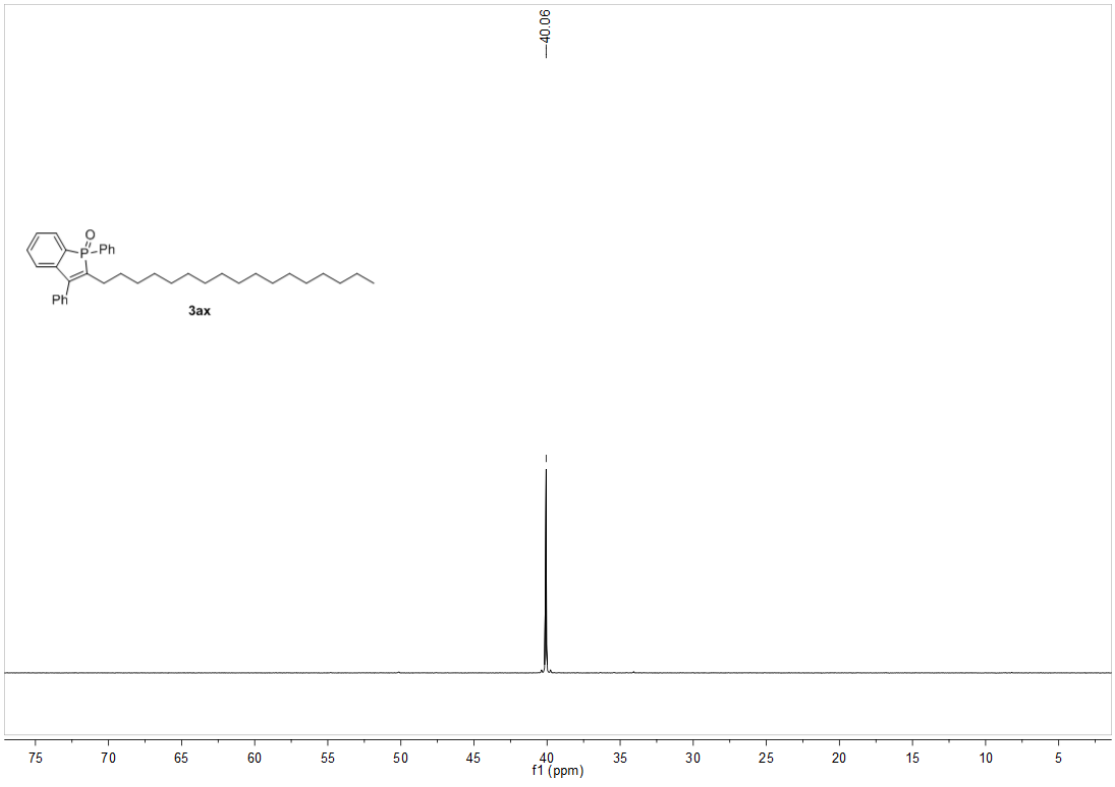
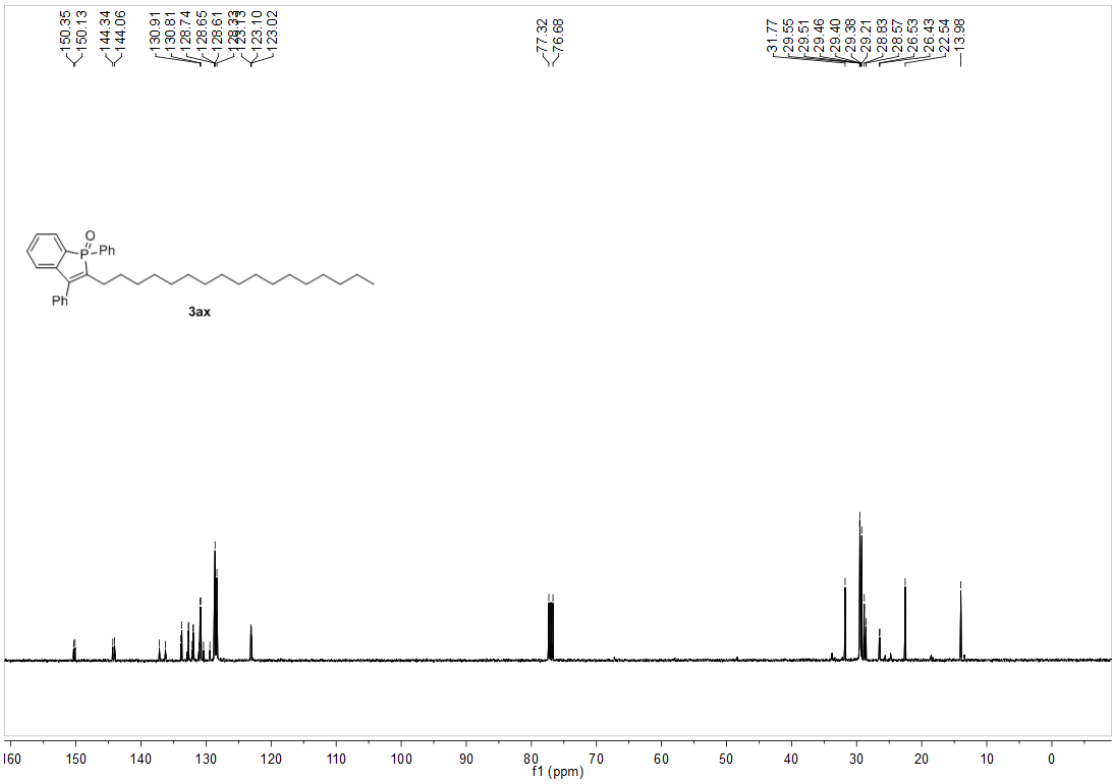
7.485
7.457
7.442
7.425
7.406
7.387
7.368
7.333
7.314
7.293
7.292
7.034
7.016
2.505
2.487
2.468
2.450
2.430
2.412
2.392
2.271
2.253
2.234
2.214
2.196
2.178
2.158
1.428
1.409
1.374
1.358
1.340
1.318
1.258
1.154
1.130
1.115
1.099
1.080
1.043
0.981
0.957
0.938
0.888
0.874
0.857
Ph
O
P
Ph
3ax
1.93
0.98
2.95
3.94
3.05
0.96
1.09
0.95
30.00
3.00
f1 (ppm)


40.06
Ph
O
P
Ph
3ax
f1 (ppm)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150.13
144.34
144.06
130.91
130.81
128.74
128.65
128.61
123.10
123.02
77.32
76.68
31.77
29.55
29.51
29.46
29.40
29.38
29.21
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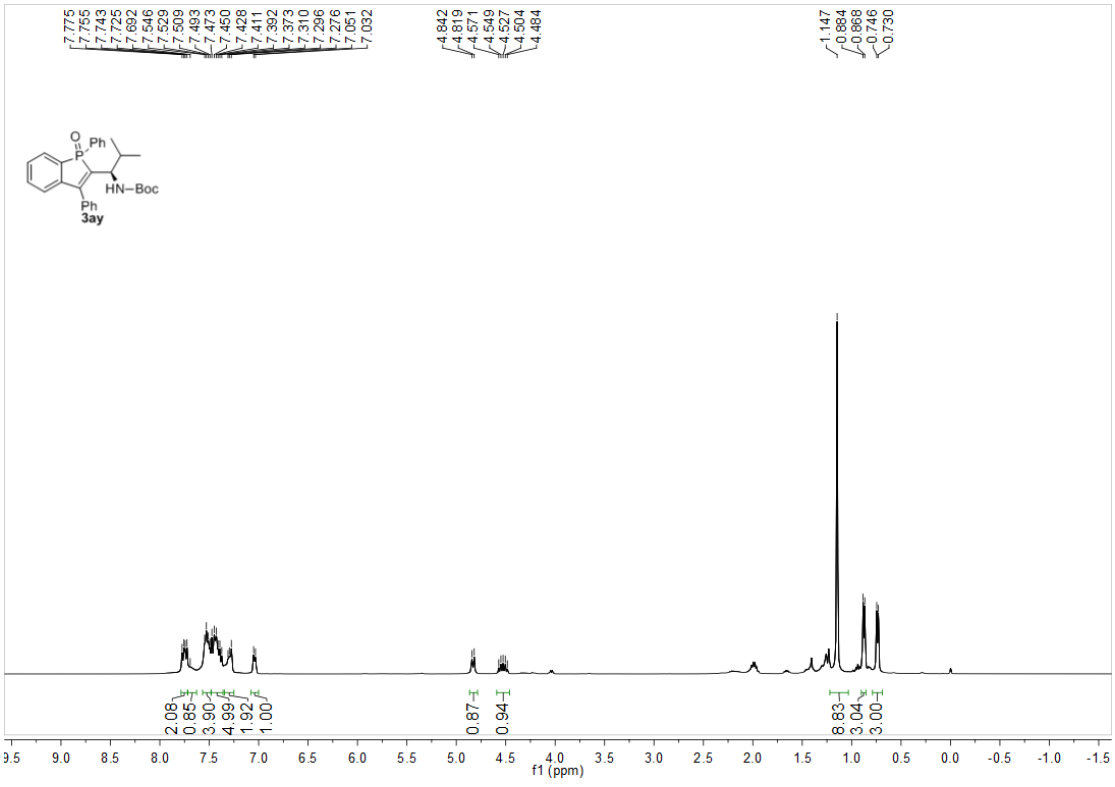
28.57
26.53
26.43
22.54
13.98
O
P
Ph
Ph
3ax
f1 (ppm)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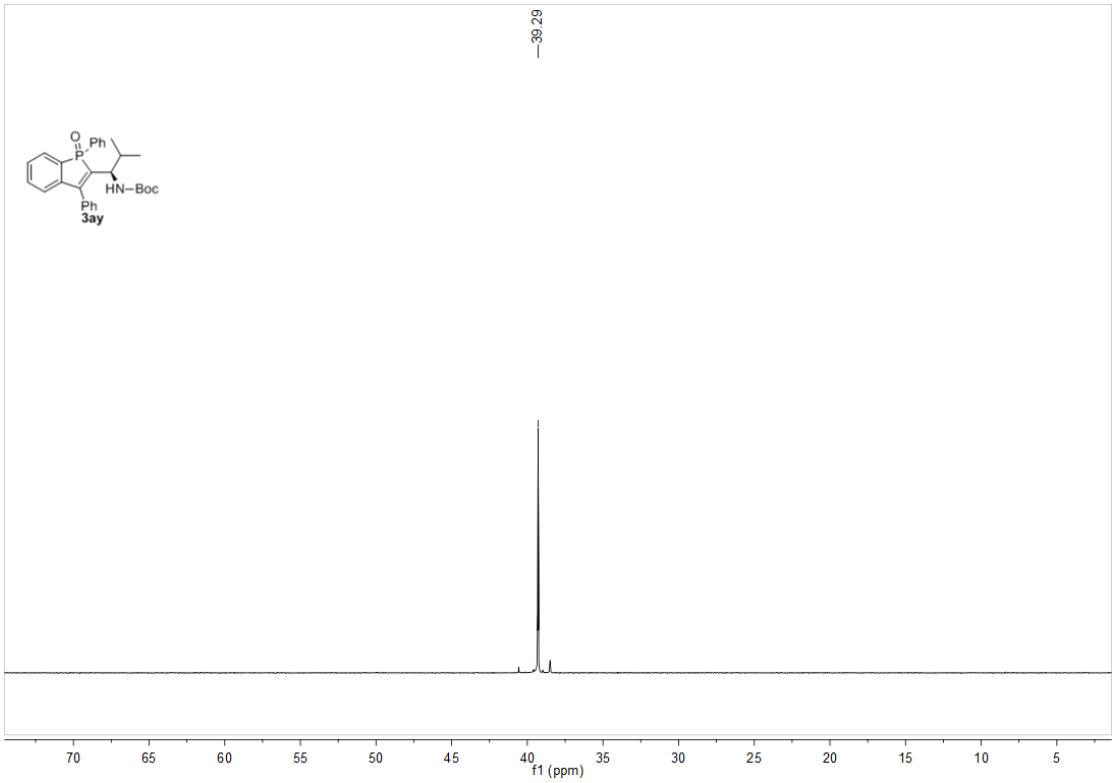
7.509
7.493
7.473
7.450
7.428
7.411
7.392
7.373
7.310
7.296
7.276
7.051
7.032
4.842
4.819
4.571
4.549
4.527
4.504
4.484
1.147
0.884
0.868
0.746
0.730
O
P
Ph
HN–Boc
Ph
3ay
2.08
0.85
3.90
4.99
1.92
1.00
0.87
0.94
8.83
3.04
3.00
f1 (ppm)


39.29
Ph
O
P
HN–Boc
Ph
3ay
70
65
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55
50
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40
35
30
25
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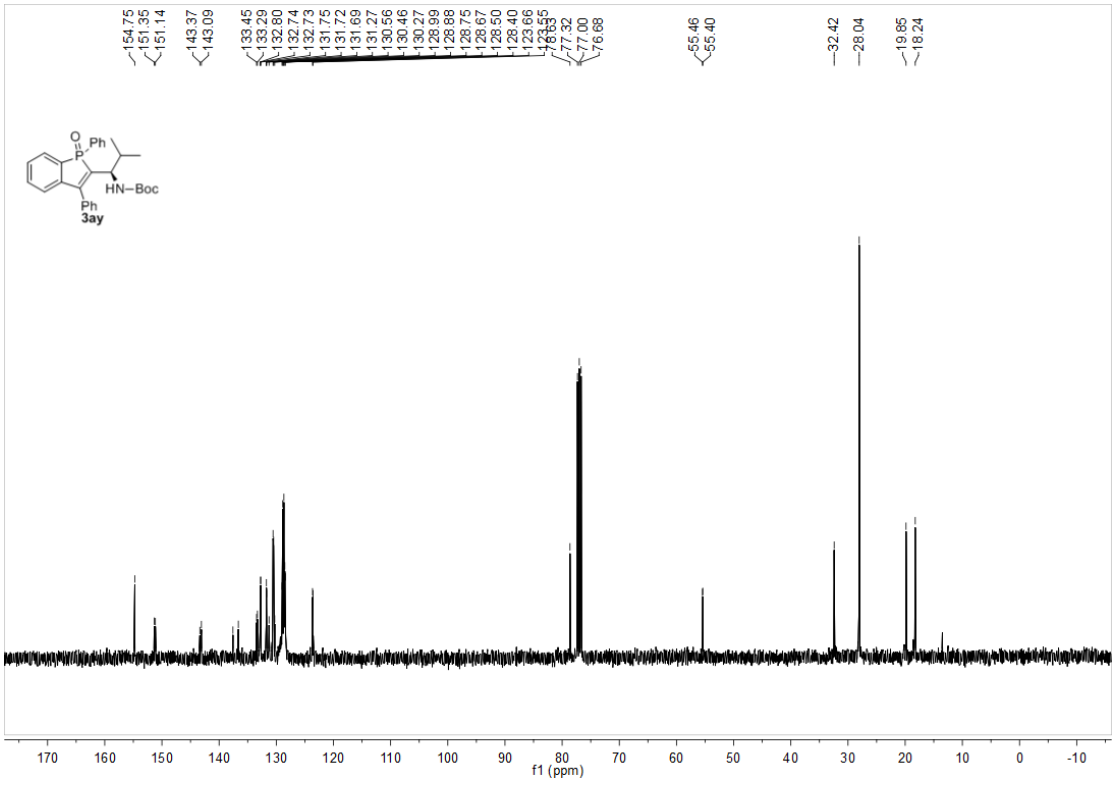
15
10
5
f1 (ppm)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133.29
132.80
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132.73
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131.72
131.69
131.27
130.56
130.46
130.27
128.99
128.88
128.75
128.67
128.50
128.40
123.66
123.55
78.63
77.32
77.00
76.68
55.46
55.40
32.42
28.04
19.85
18.24
Ph
O
P
HN–Boc
Ph
3ay
170
160
150
140
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120
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100
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80
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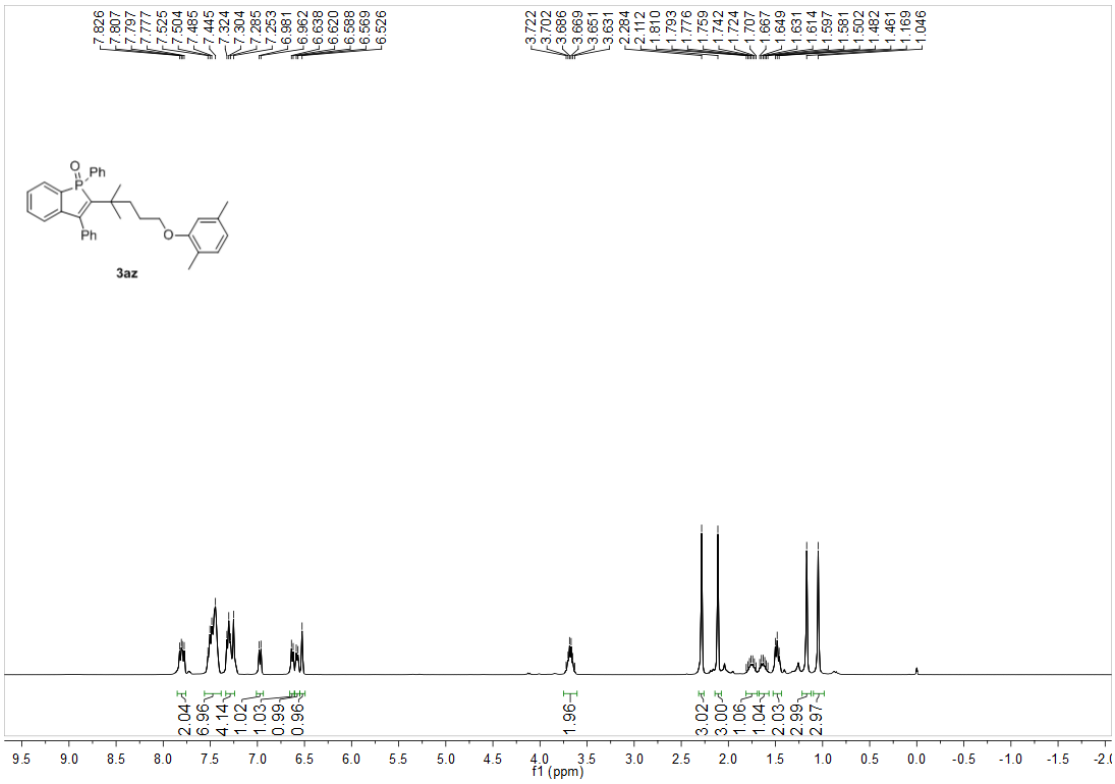
40
30
20
10
0
-10
f1 (ppm)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7.797
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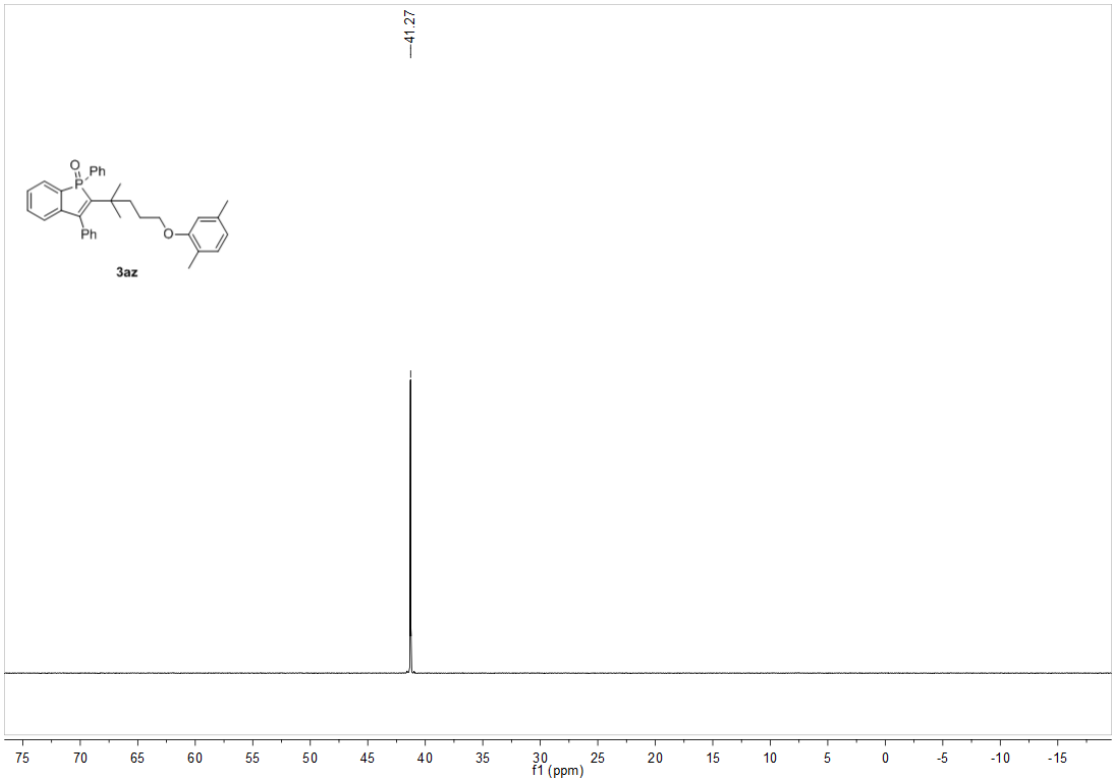
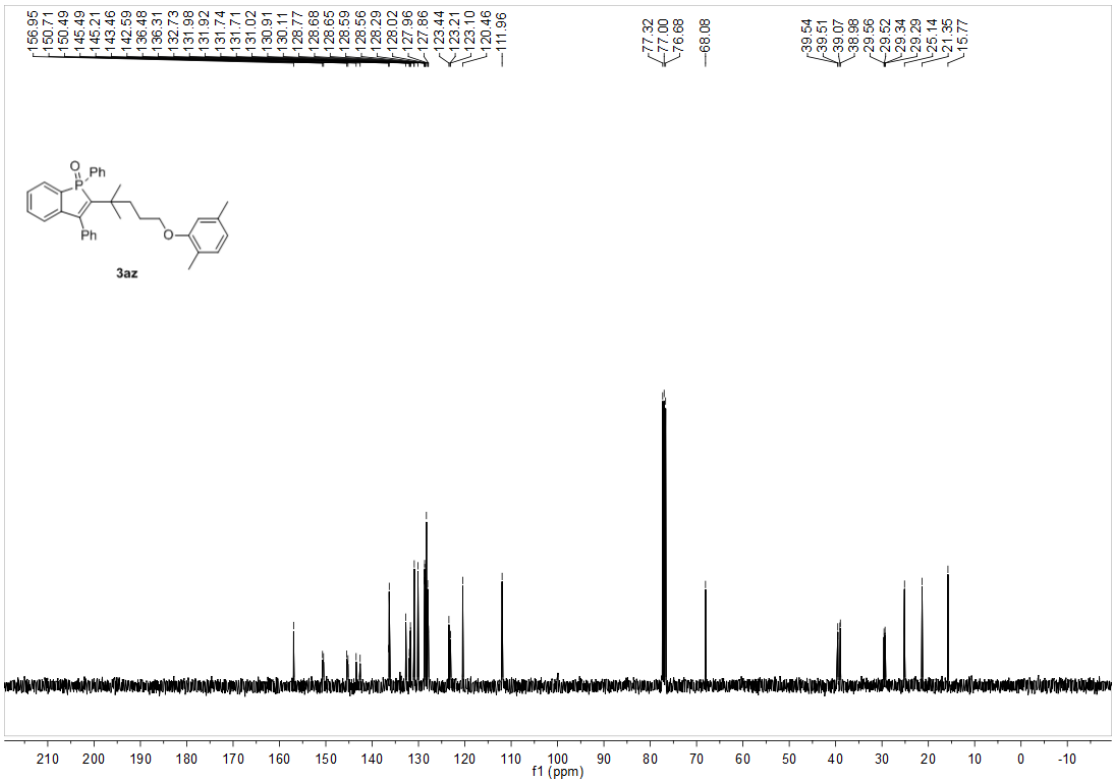
7.504
7.485
7.445
7.324
7.304
7.285
7.253
6.981
6.962
6.638
6.620
6.588
6.569
6.526
3.722
3.702
3.686
3.669
3.651
3.631
2.284
2.112
1.810
1.793
1.776
1.759
1.742
1.724
1.707
1.667
1.649
1.631
1.614
1.597
1.581
1.502
1.482
1.461
1.169
1.046
O
Ph
P
Ph
O
3az
2.04
6.96
4.14
1.02
1.03
0.99
0.96
1.96
3.02
3.00
1.06
1.04
2.03
2.99
2.97
f1 (ppm)


41.27
O
Ph
P
Ph
O
3az
f1 (ppm)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136.48
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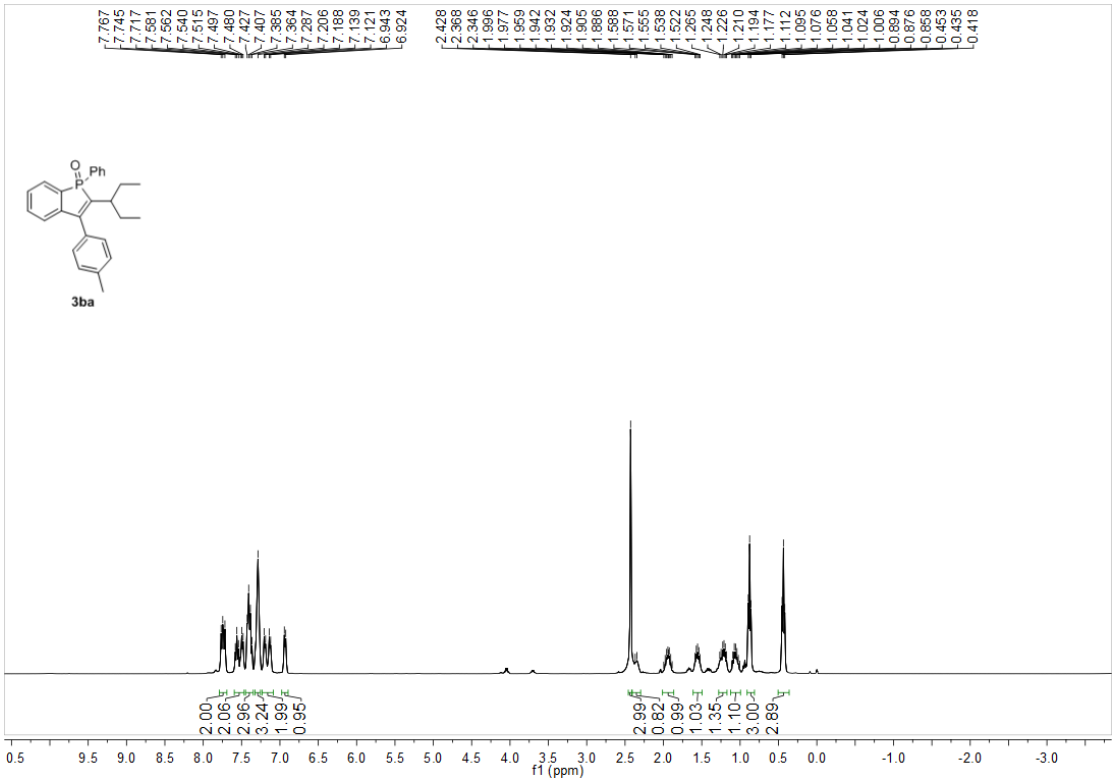
132.73
131.98
131.92
131.74
131.71
131.02
130.91
130.11
128.77
128.68
128.65
128.59
128.56
128.29
128.02
127.96
127.86
123.44
123.21
123.10
120.46
111.96
77.32
77.00
76.68
68.08
39.54
39.51
39.07
38.98
29.56
29.52
29.34
29.29
25.14
21.35
15.77
Ph
Ph
O
P
O
3az
f1 (ppm)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7.540
7.515
7.497
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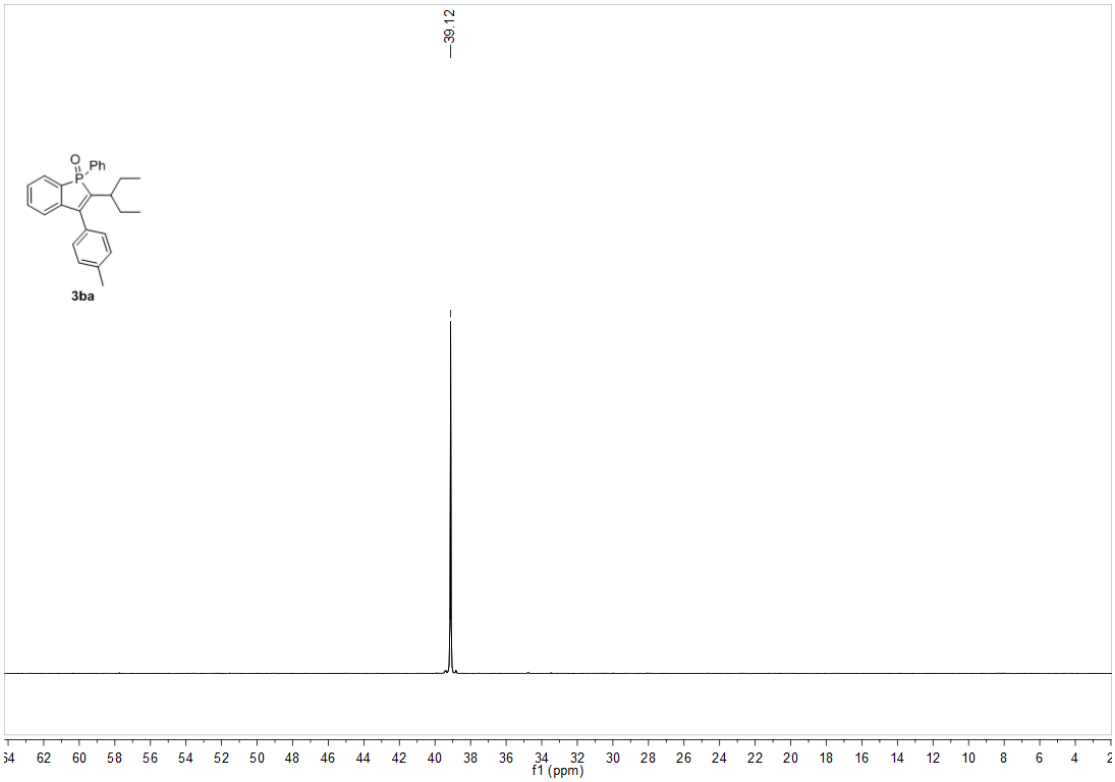
7.407
7.385
7.364
7.287
7.206
7.188
7.139
7.121
6.943
6.924
2.428
2.368
2.346
1.996
1.977
1.959
1.942
1.932
1.924
1.905
1.886
1.588
1.571
1.555
1.538
1.522
1.265
1.248
1.226
1.210
1.194
1.177
1.112
1.095
1.076
1.058
1.041
1.024
1.006
0.894
0.876
0.858
0.453
0.435
0.418
Ph
O
P
3ba
2.00
2.06
2.96
3.24
1.99
0.95
2.99
0.82
0.99
1.03
1.35
1.10
3.00
2.89
f1 (ppm)


39.12
O
P
Ph
3ba
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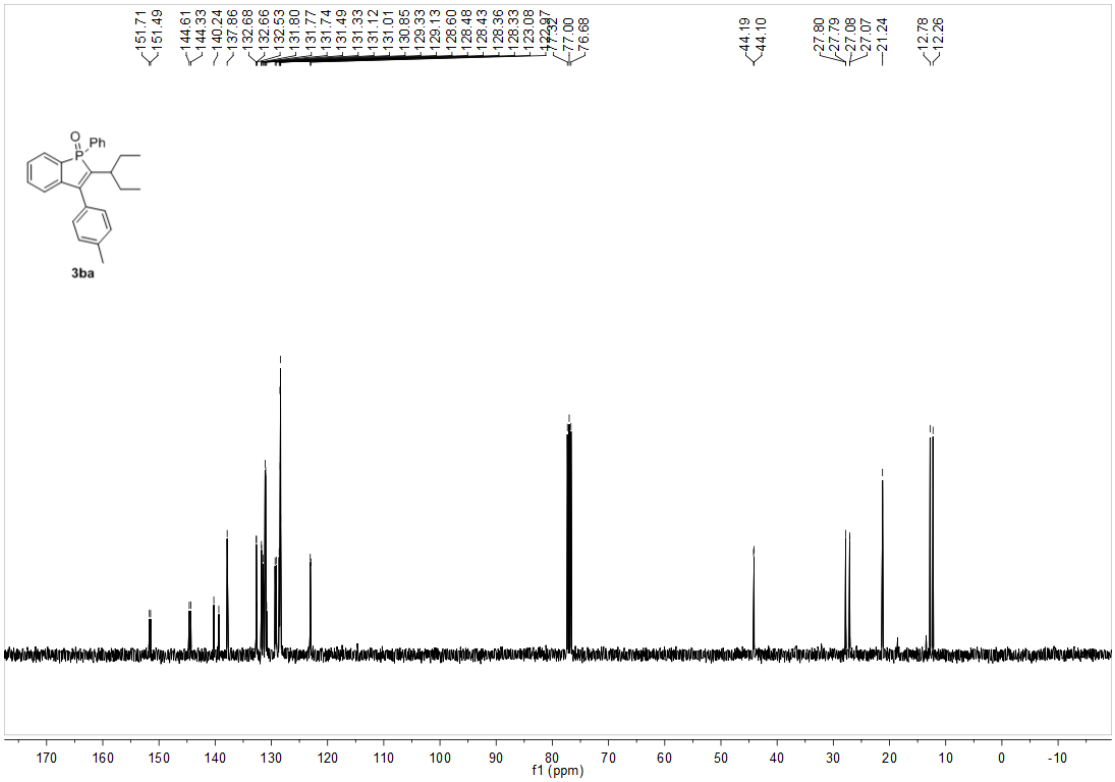
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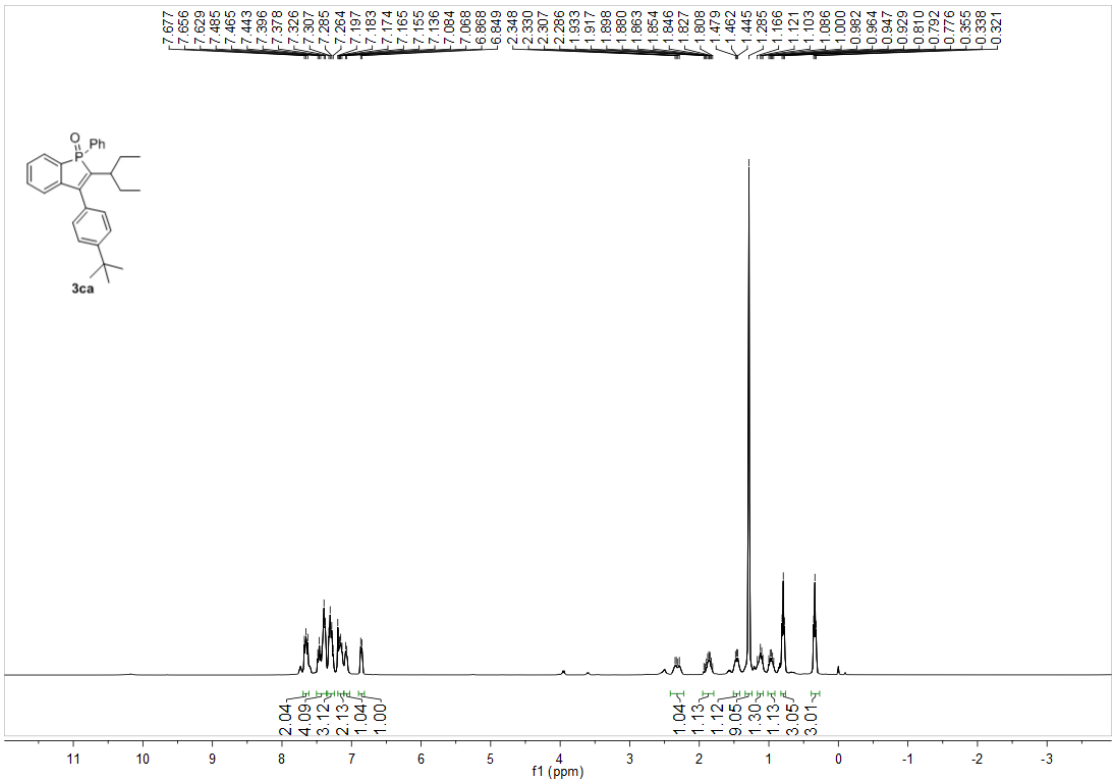
131.80
131.77
131.74
131.49
131.33
131.12
131.01
130.85
129.33
129.13
128.60
128.48
128.43
128.36
128.33
123.08
77.32
77.00
76.68
44.19
44.10
27.80
27.79
27.08
27.07
21.24
12.78
12.26
O
P
Ph
3ba
f1 (ppm)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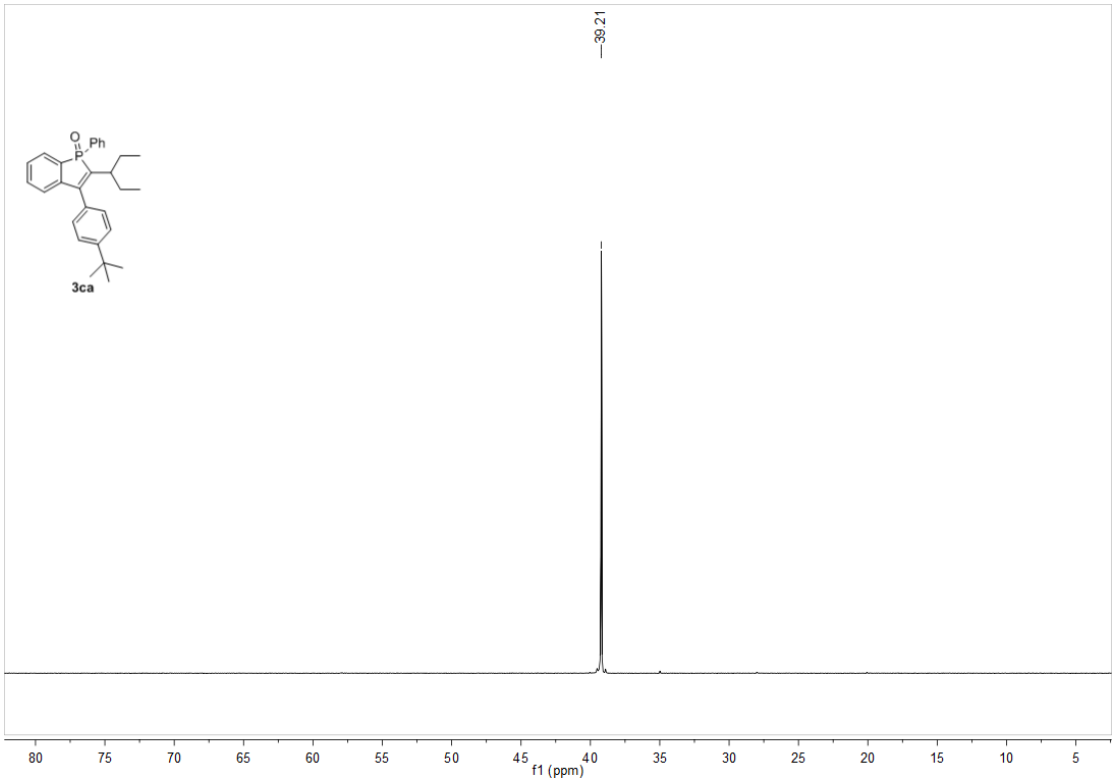
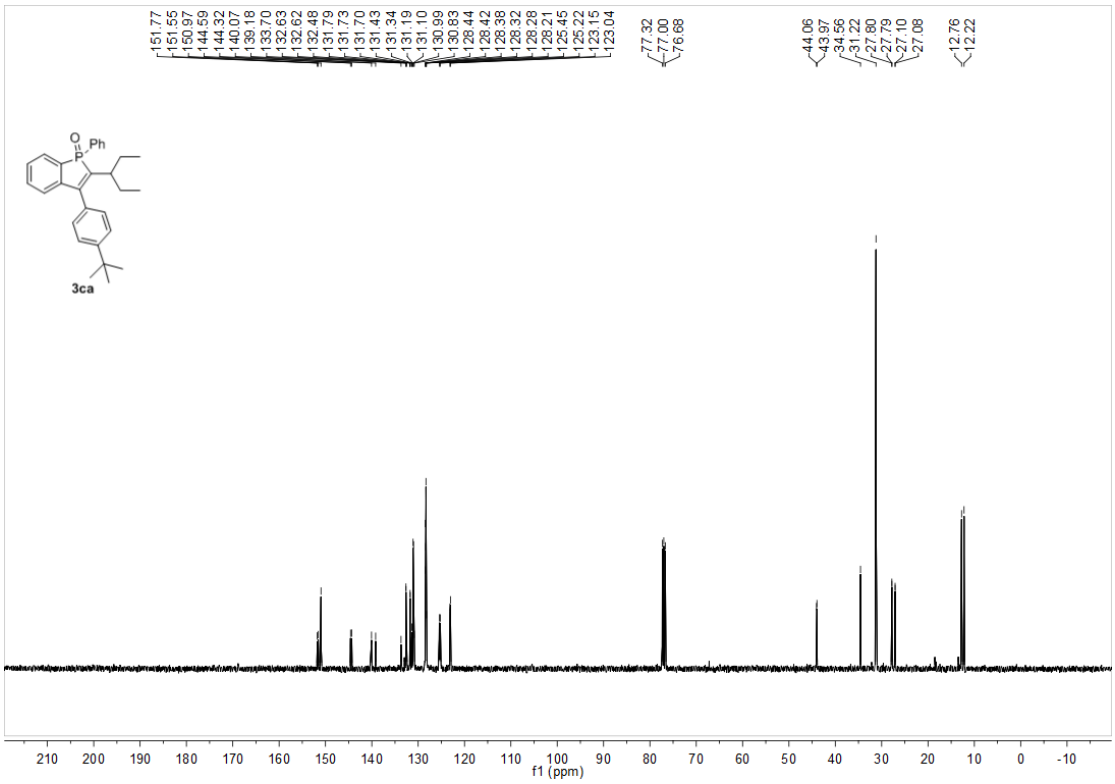
7.183
7.174
7.165
7.155
7.136
7.084
7.068
6.868
6.849
2.348
2.330
2.307
2.286
1.933
1.917
1.898
1.880
1.863
1.854
1.846
1.827
1.808
1.479
1.462
1.445
1.285
1.166
1.121
1.103
1.086
1.000
0.982
0.964
0.947
0.929
0.810
0.792
0.776
0.355
0.338
0.321
O
Ph
P
3ca
2.04
4.09
3.12
2.13
1.04
1.00
1.04
1.13
1.12
9.05
1.30
1.13
3.05
3.01
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


39.21
O
Ph
P
3ca
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
f1 (ppm)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131.79
131.73
131.70
131.43
131.34
131.19
131.10
130.99
130.83
128.44
128.42
128.38
128.32
128.28
128.21
125.45
125.22
123.15
123.04
77.32
77.00
76.68
44.06
43.97
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31.22
27.80
27.79
27.10
27.08
12.76
12.22
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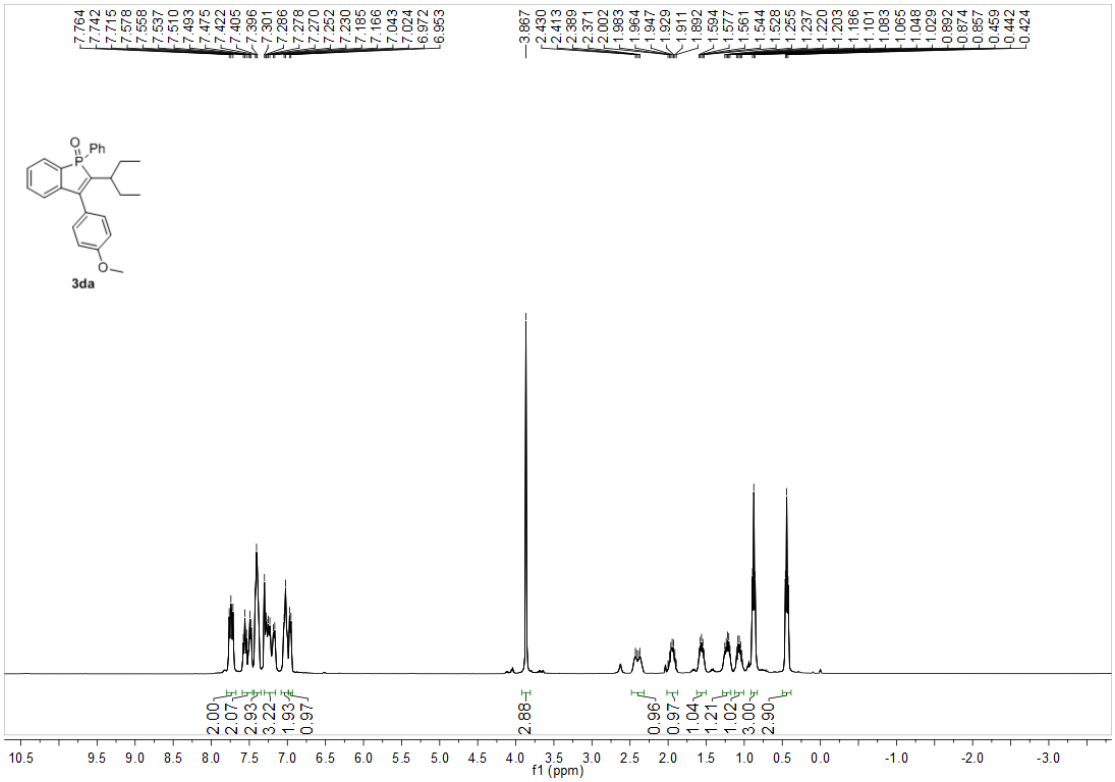
P
Ph
3ca
f1 (ppm)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7.396
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7.286
7.278
7.270
7.252
7.230
7.185
7.166
7.043
7.024
6.972
6.953
3.867
2.430
2.413
2.389
2.371
2.002
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1.964
1.947
1.929
1.911
1.892
1.594
1.577
1.561
1.544
1.528
1.255
1.237
1.220
1.203
1.186
1.101
1.083
1.065
1.048
1.029
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0.874
0.857
0.459
0.442
0.424
O
P
Ph
O
3da
2.00
2.07
2.93
3.22
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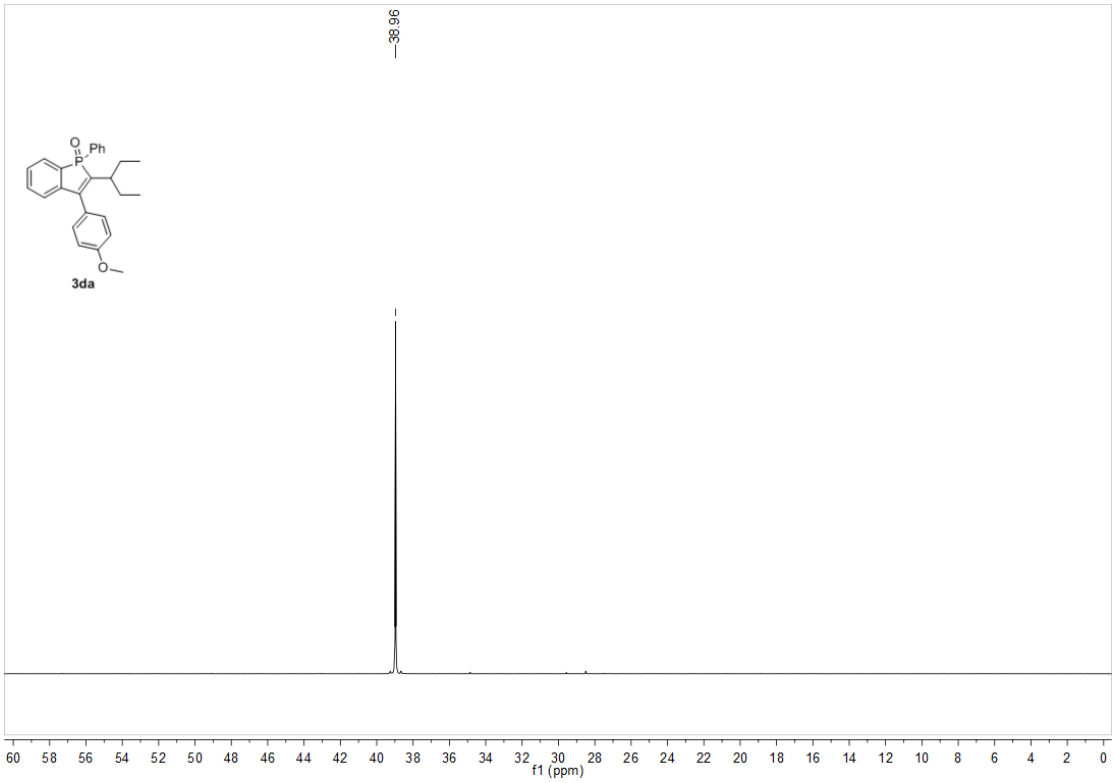
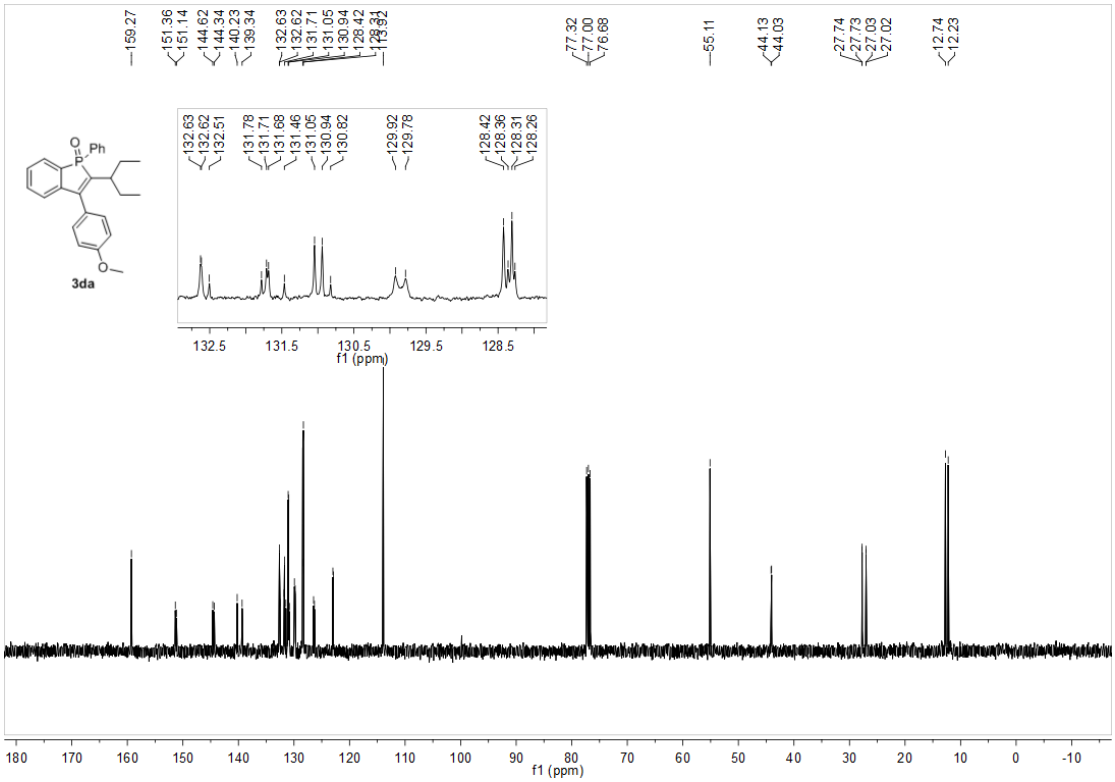
2.88
0.96
0.97
1.04
1.21
1.02
3.00
2.90
f1 (ppm)


38.96
3da
f1 (ppm)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130.94
128.42
128.31
113.92
77.32
77.00
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44.03
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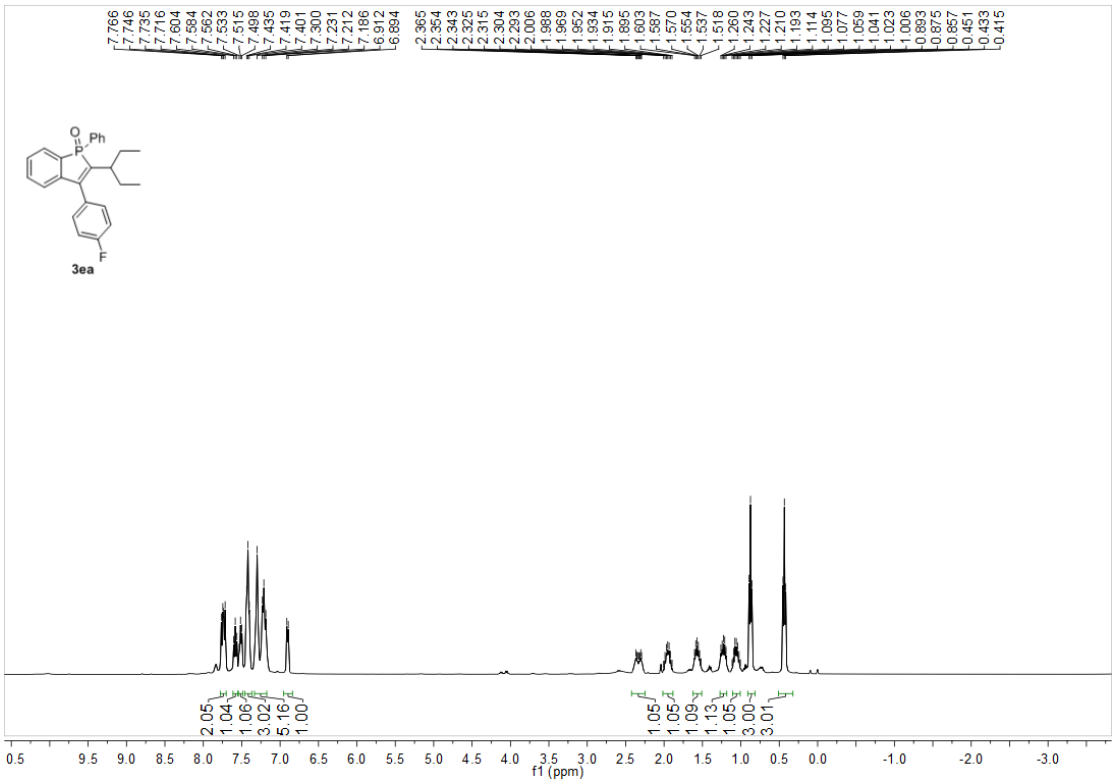
27.73
27.03
27.02
12.74
12.23
3da
f1 (ppm)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7.419
7.401
7.300
7.231
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2.343
2.325
2.315
2.304
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2.006
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1.952
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1.915
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0.875
0.857
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0.415
O
Ph
P
F
3ea
2.05
1.04
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1.05
1.09
1.13
1.05
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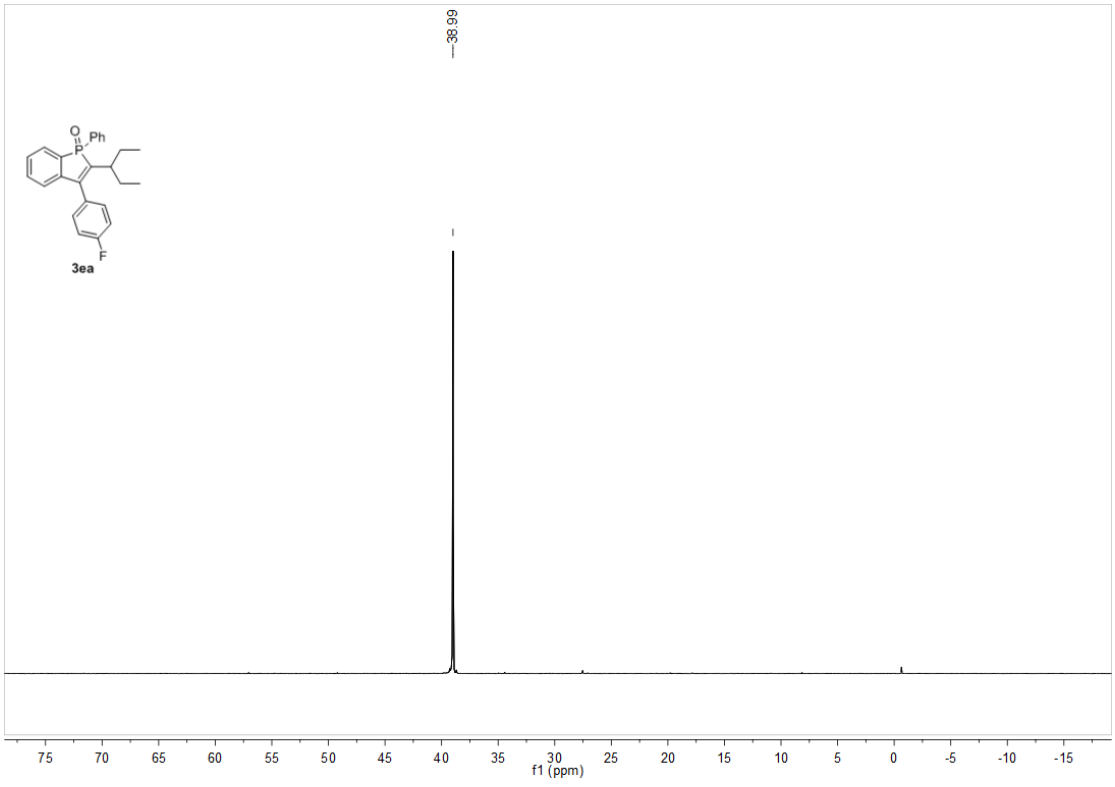
3.01
f1 (ppm)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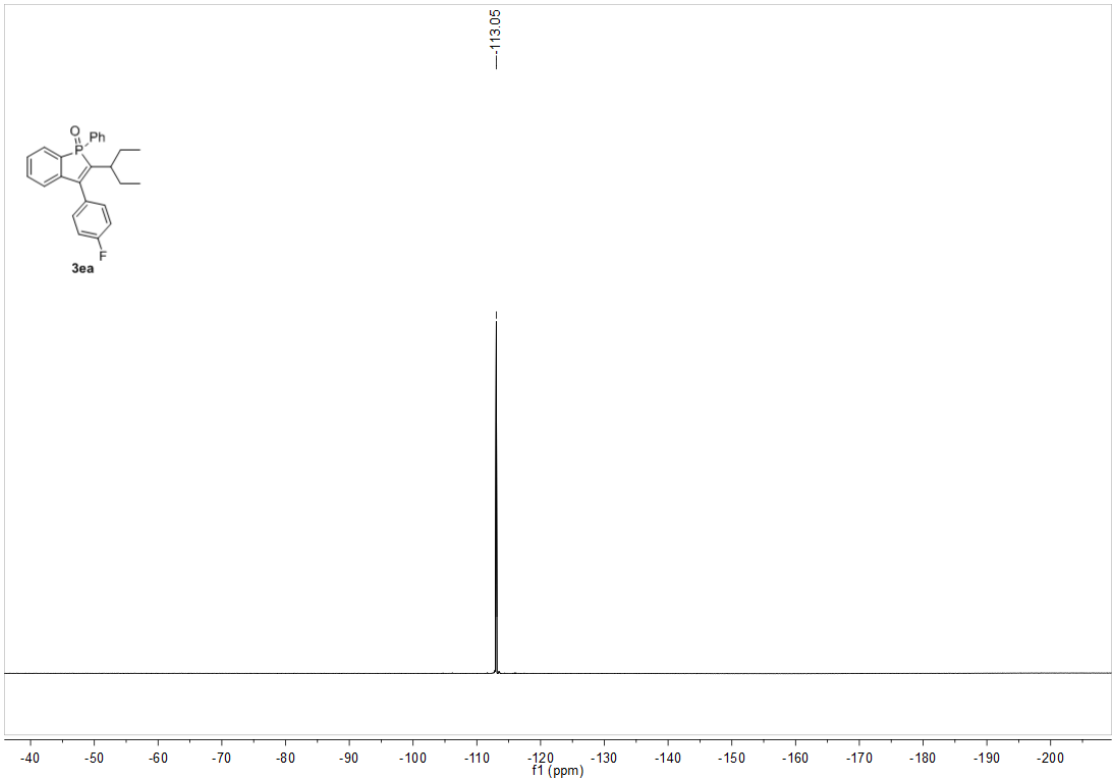
Ph
P
F
3ea
f1 (ppm)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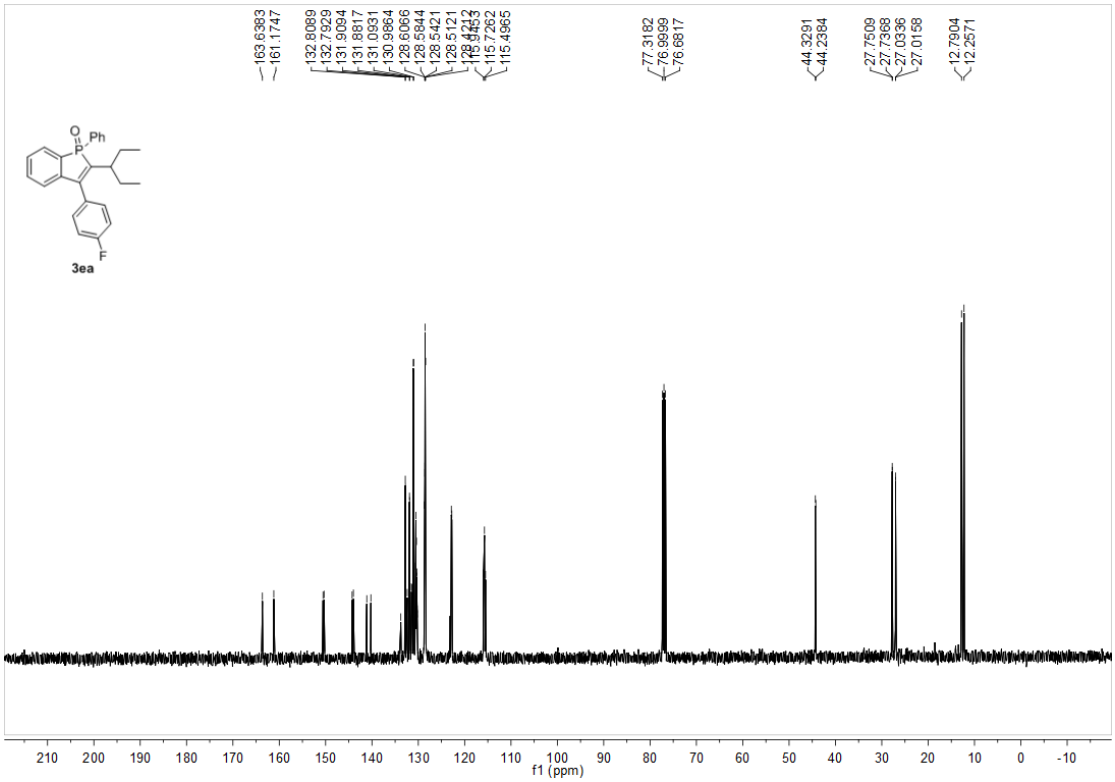
3ea
-40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)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76.6817
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27.7368
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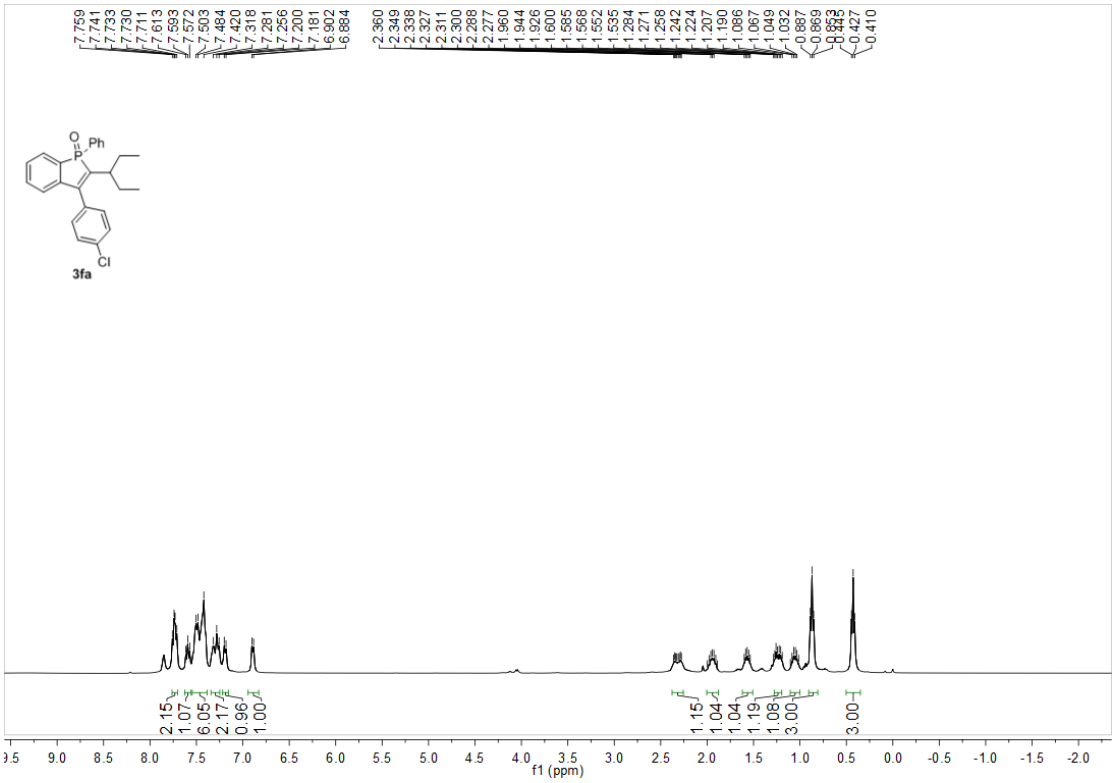
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.200
7.181
6.902
6.884
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2.327
2.311
2.300
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2.277
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1.944
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1.600
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1.552
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1.067
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0.410
O
P
Ph
Cl
3fa
2.15
1.07
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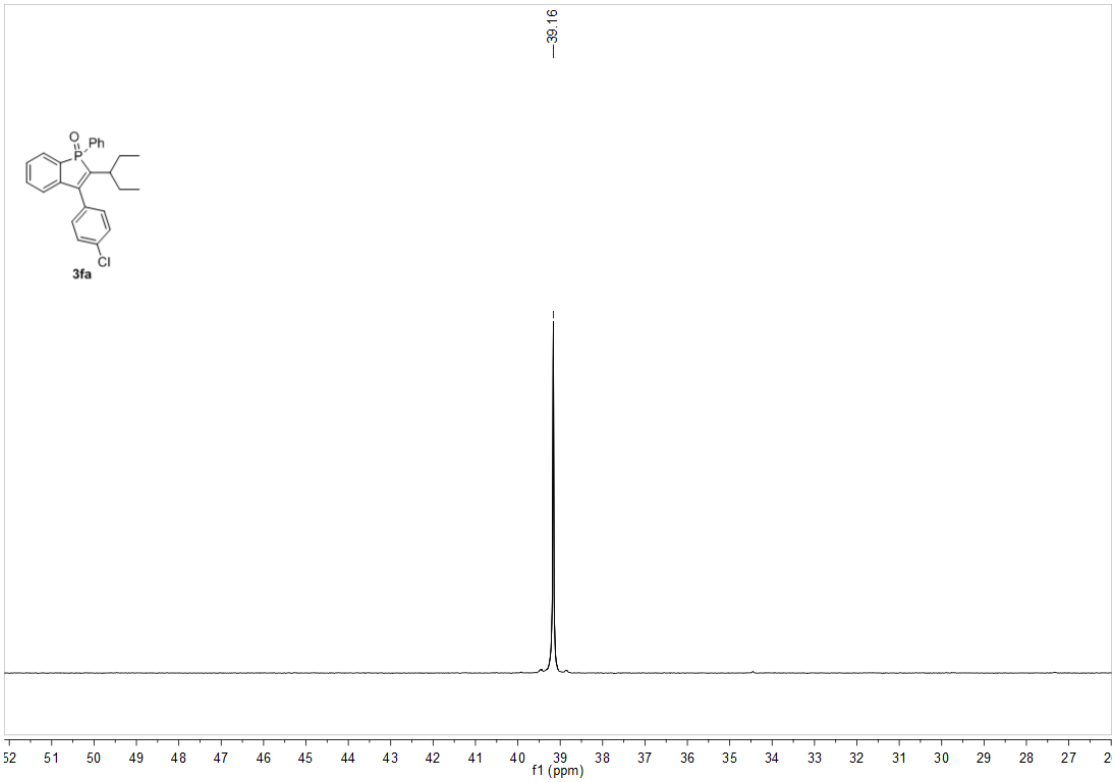
1.04
1.04
1.19
1.08
3.00
3.00
f1 (ppm)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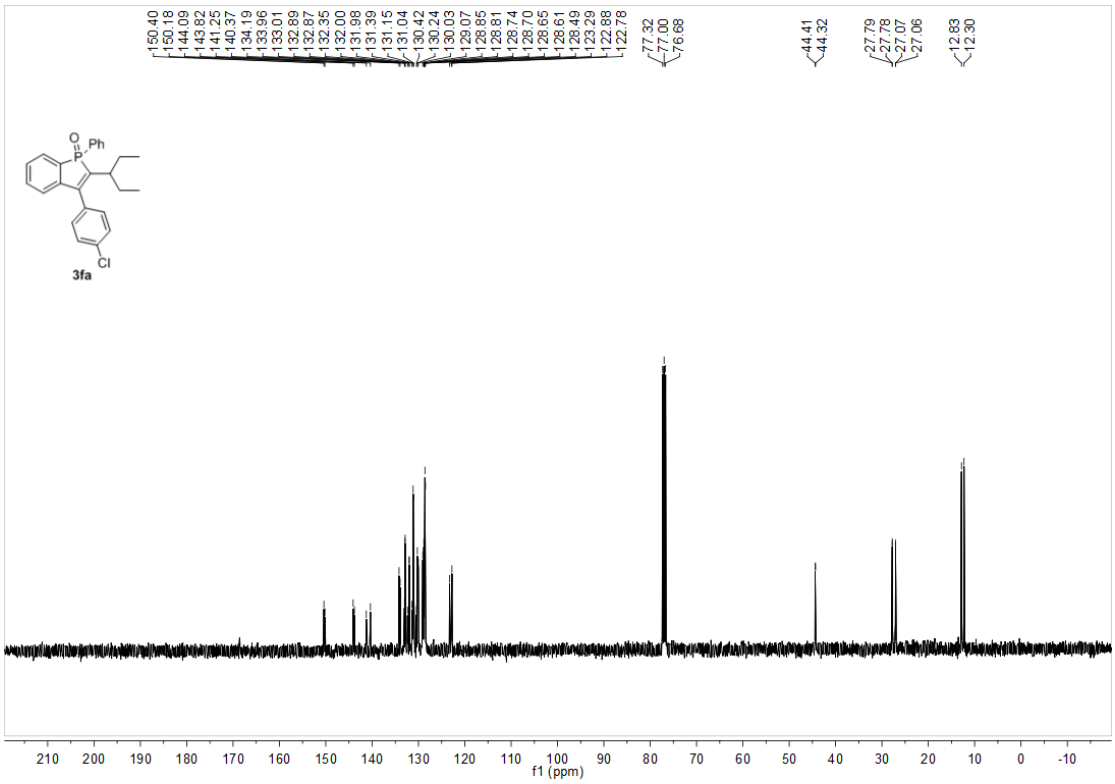
O
P
Ph
Cl
3fa
f1 (ppm)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141.25
140.37
134.19
133.96
133.01
132.89
132.87
132.35
132.00
131.98
131.39
131.15
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130.24
130.03
129.07
128.85
128.81
128.74
128.70
128.65
128.61
128.49
123.29
122.88
122.78
77.32
77.00
76.68
44.41
44.32
27.79
27.78
27.07
27.06
12.83
12.30
O
P
Ph
Cl
3fa
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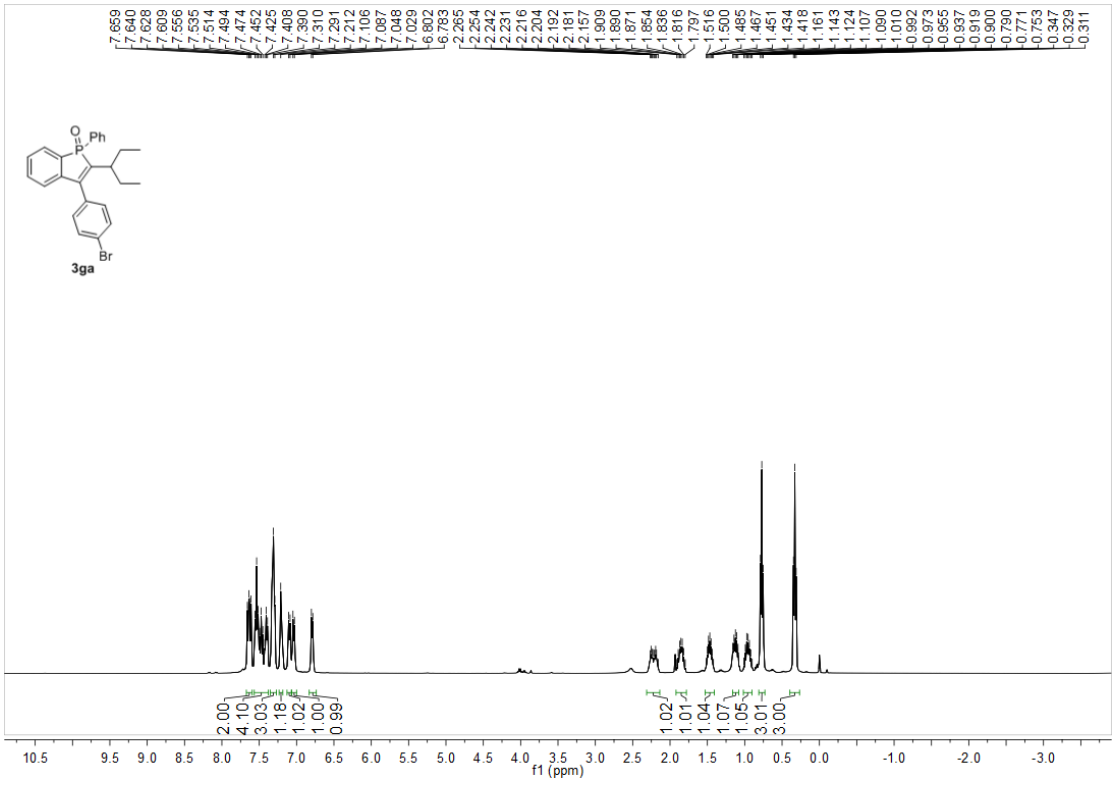
f1 (ppm)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0.311
O
P
Ph
Br
3ga
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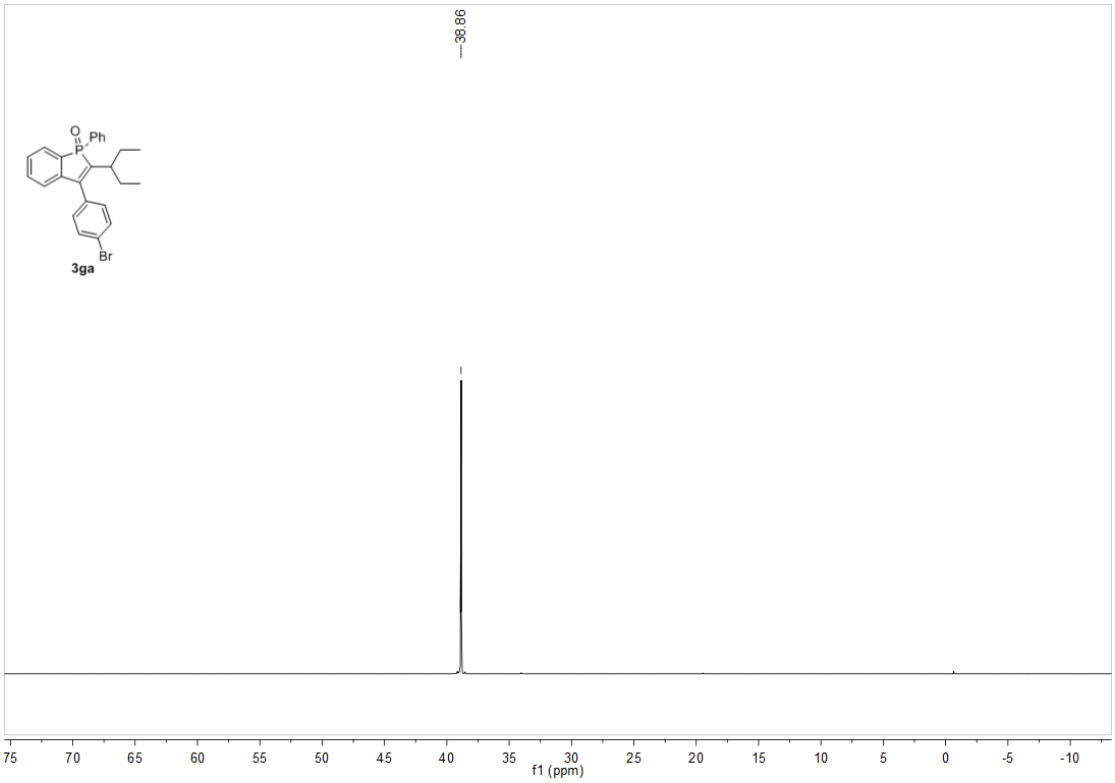
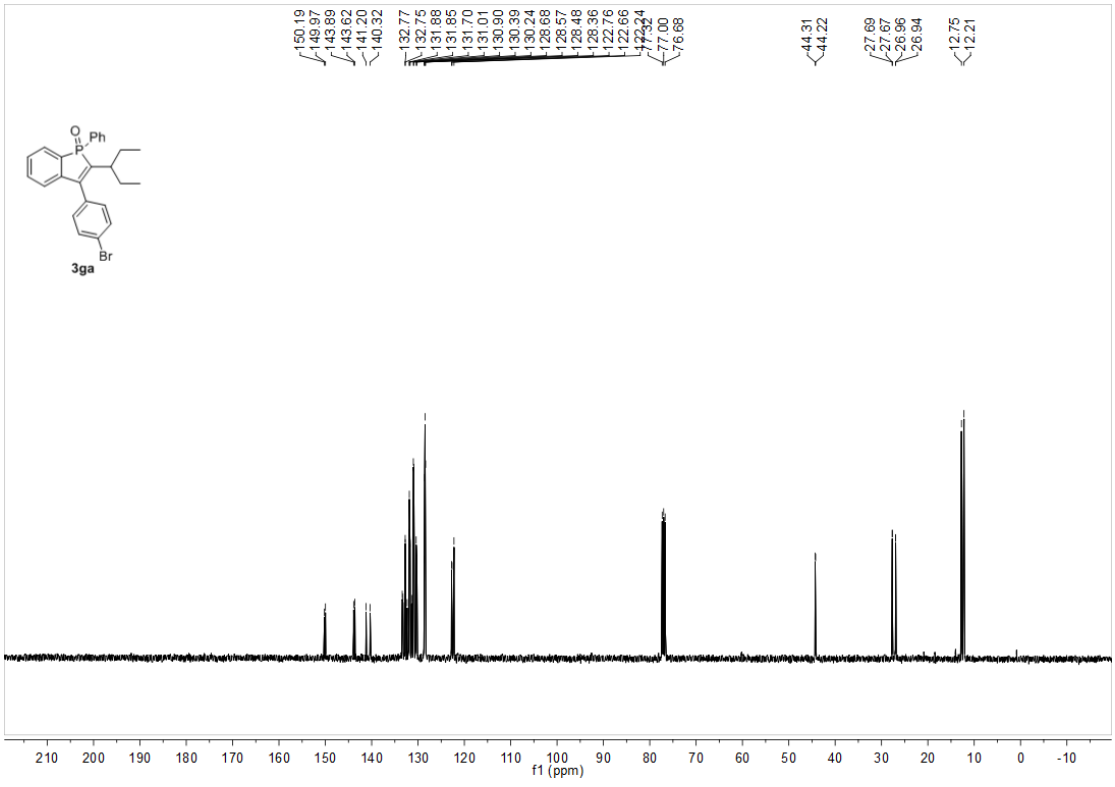
4.10
3.03
1.18
1.02
1.00
0.99
1.02
1.01
1.04
1.07
1.05
3.01
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0 -2.0 -3.0
f1 (ppm)


38.86
Ph
Br
3ga
75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5 -10
f1 (ppm)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149.97
143.89
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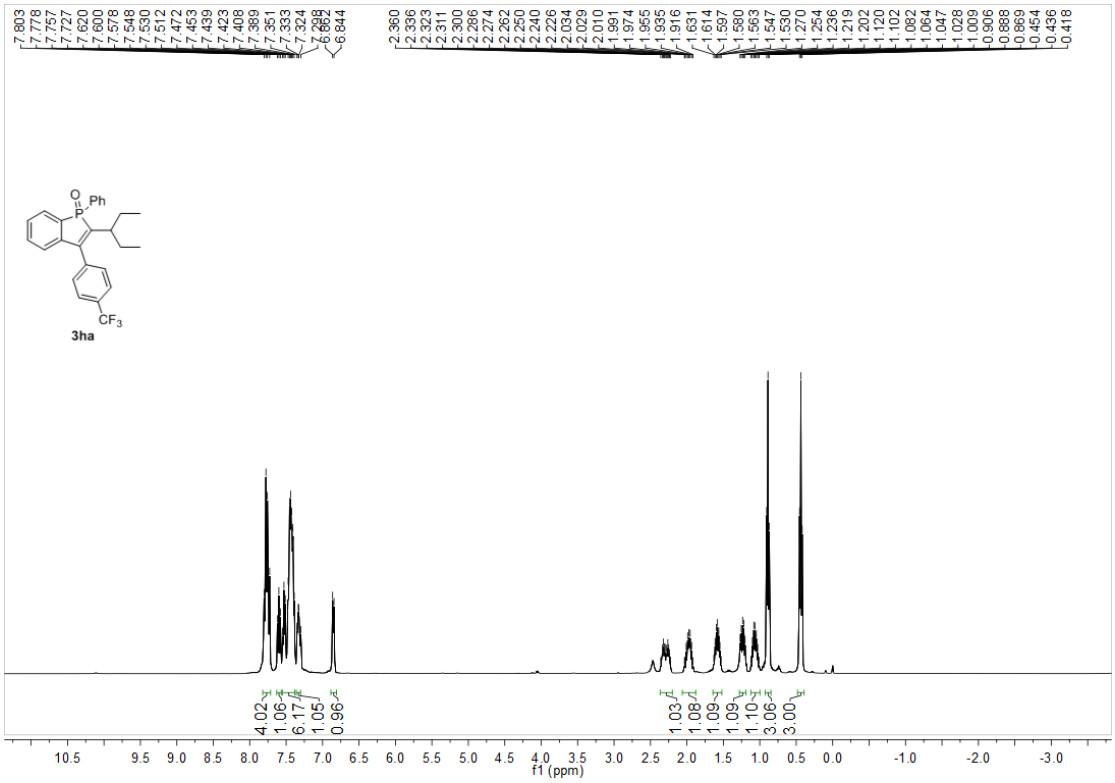
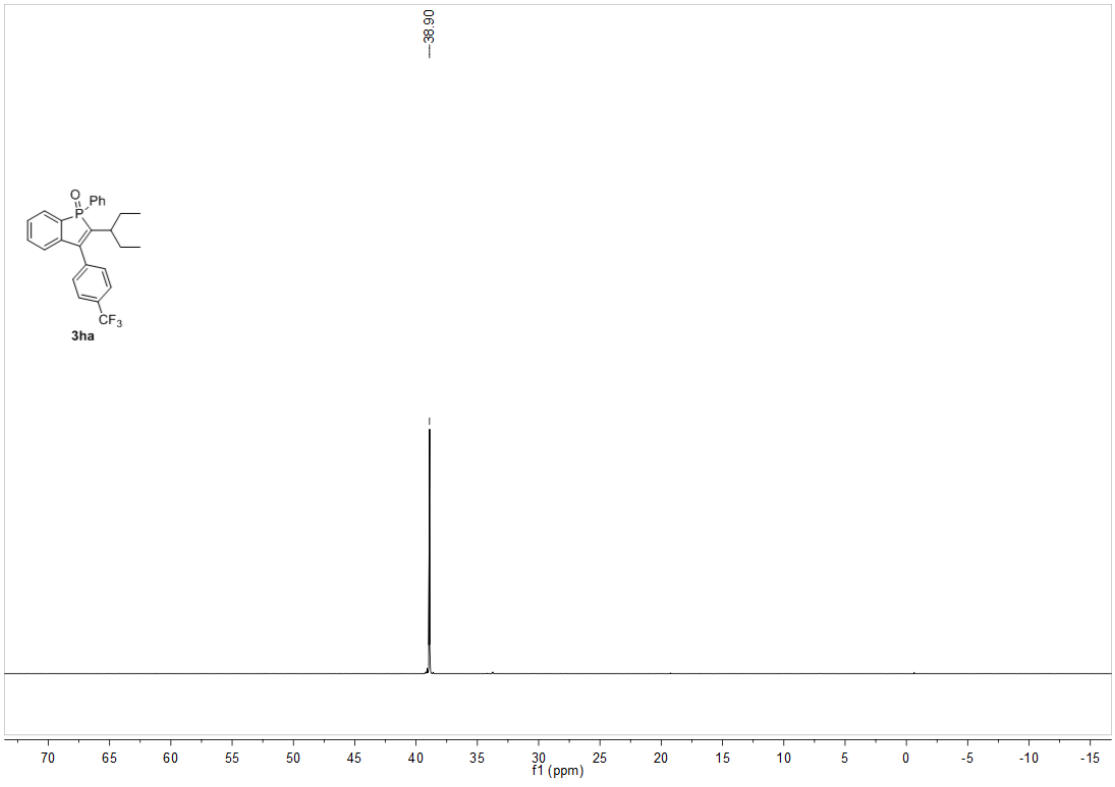
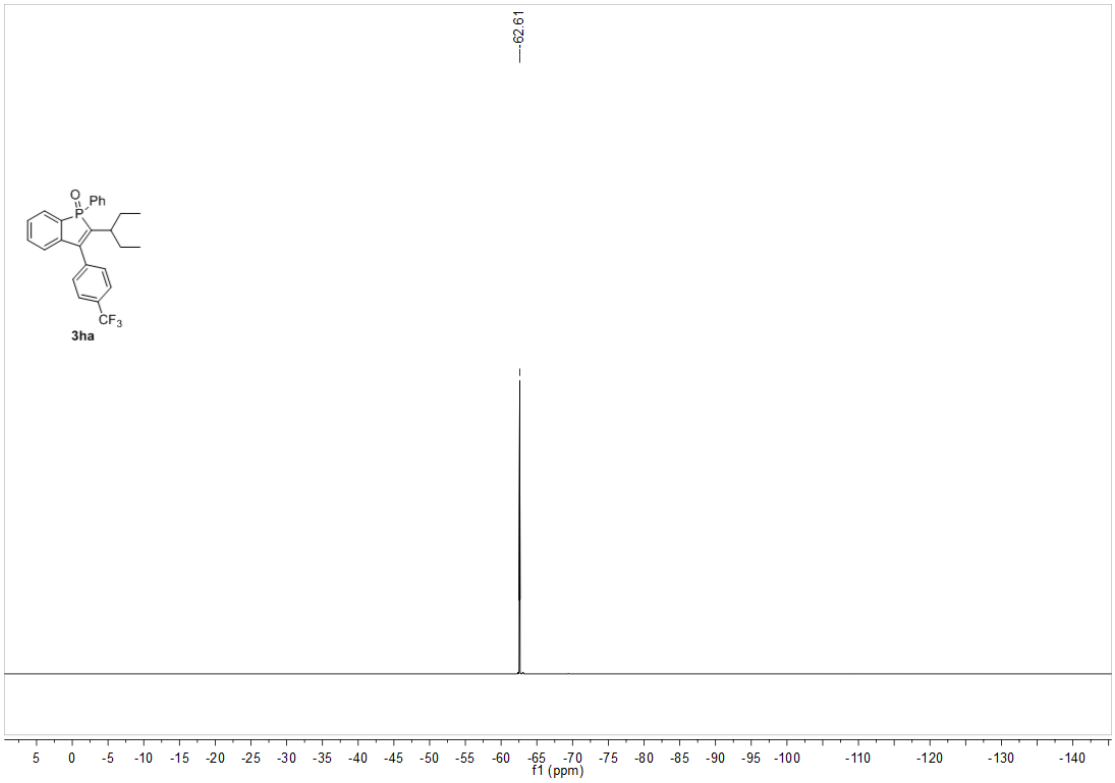
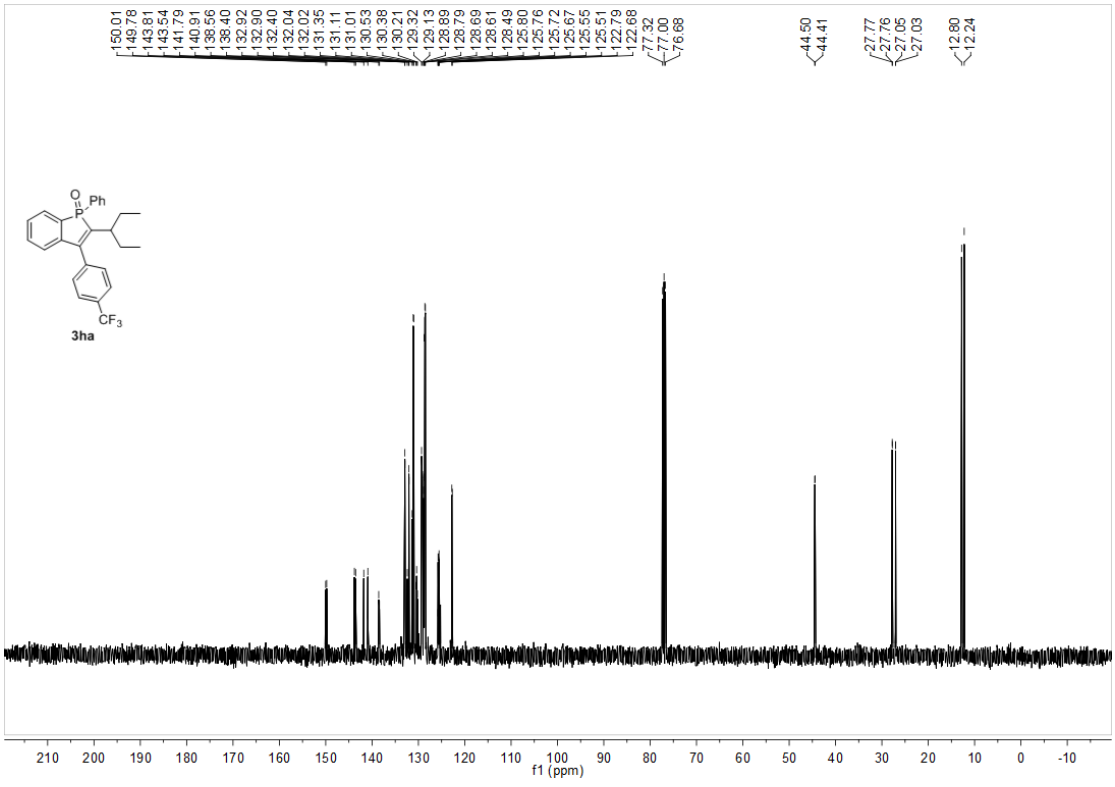
141.20
140.32
132.77
132.75
131.88
131.85
131.70
131.01
130.90
130.39
130.24
128.68
128.57
128.48
128.36
122.76
122.66
77.32
77.00
76.68
44.31
44.22
27.69
27.67
26.96
26.94
12.75
12.21
Ph
Br
3ga
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.803
7.778
7.757
7.727
7.620
7.600
7.578
7.548
7.530
7.512
7.472
7.453
7.439
7.423
7.408
7.389
7.351
7.333
7.324
6.862
6.844
2.360
2.336
2.323
2.311
2.300
2.286
2.274
2.262
2.250
2.240
2.226
2.034
2.029
2.010
1.991
1.974
1.955
1.935
1.916
1.631
1.614
1.597
1.580
1.563
1.547
1.530
1.270
1.254
1.236
1.219
1.202
1.120
1.102
1.082
1.064
1.047
1.028
1.009
0.906
0.888
0.869
0.454
0.436
0.418
O
Ph
P
CF3
3ha
4.02
1.06
6.17
1.05
0.96
1.03
1.08
1.09
1.09
1.10
3.06
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-1.0
-2.0
-3.0
f1 (ppm)


38.90
O
Ph
P
CF3
3ha
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
f1 (ppm)


62.61
Ph
O
P
CF3
3ha
5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -110 -120 -130 -140
f1 (ppm)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143.81
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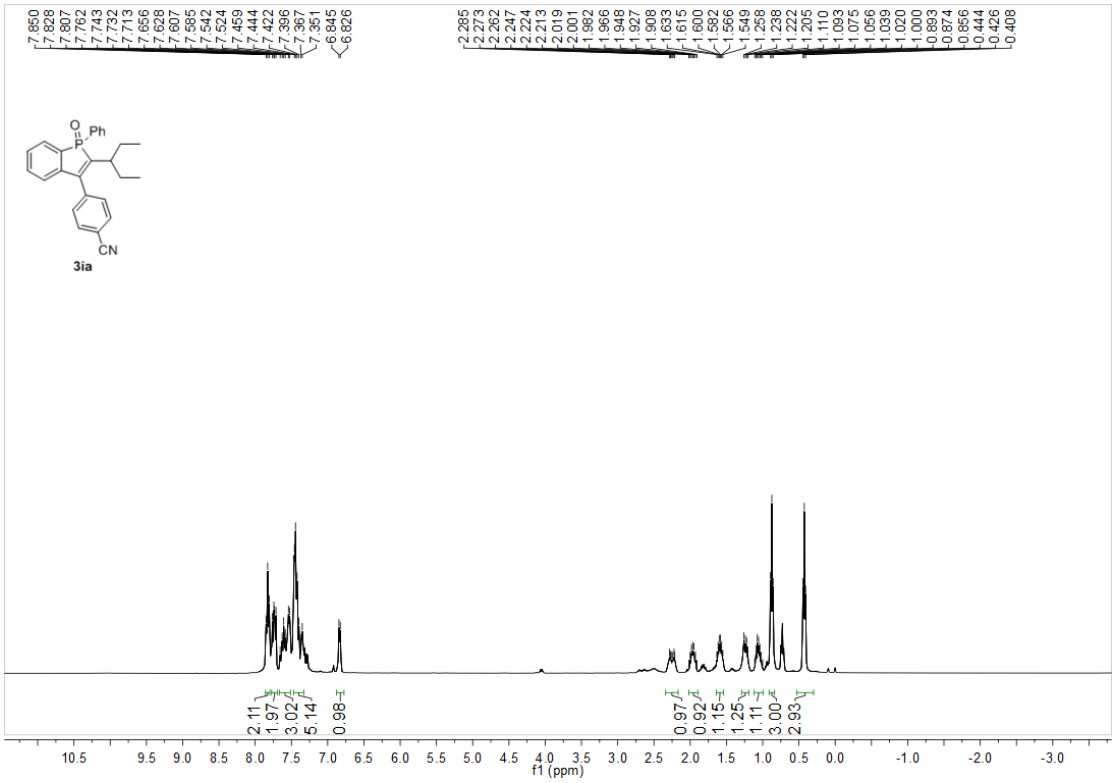
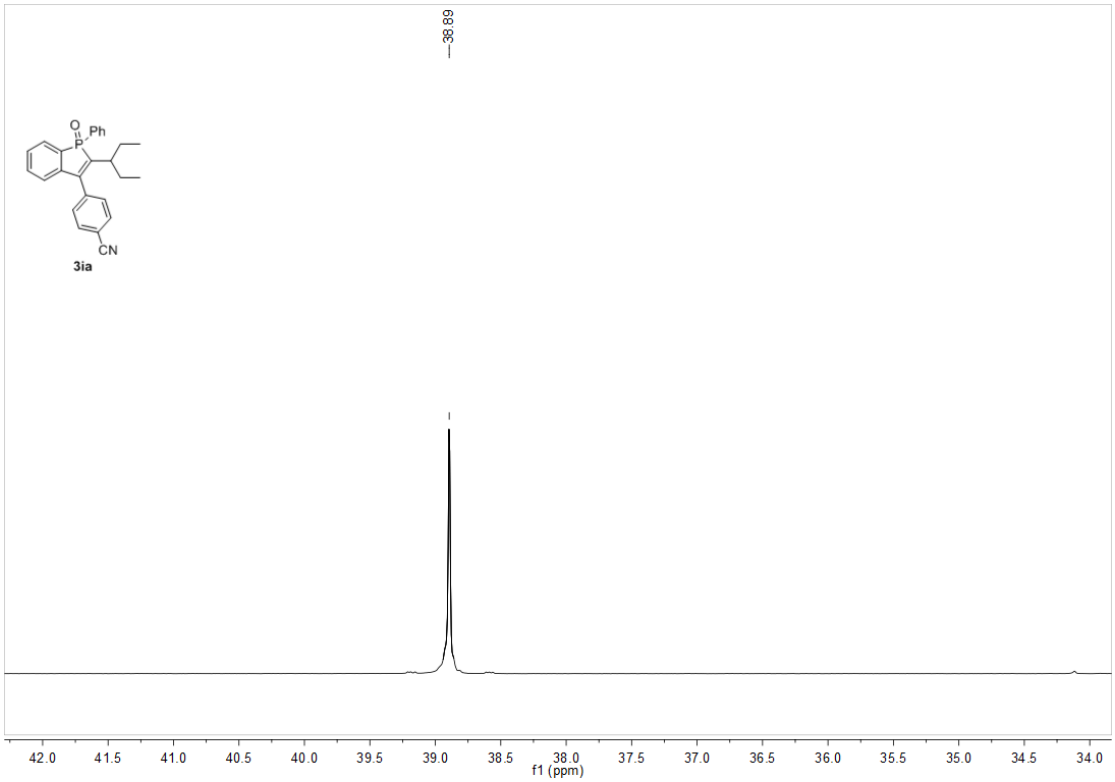
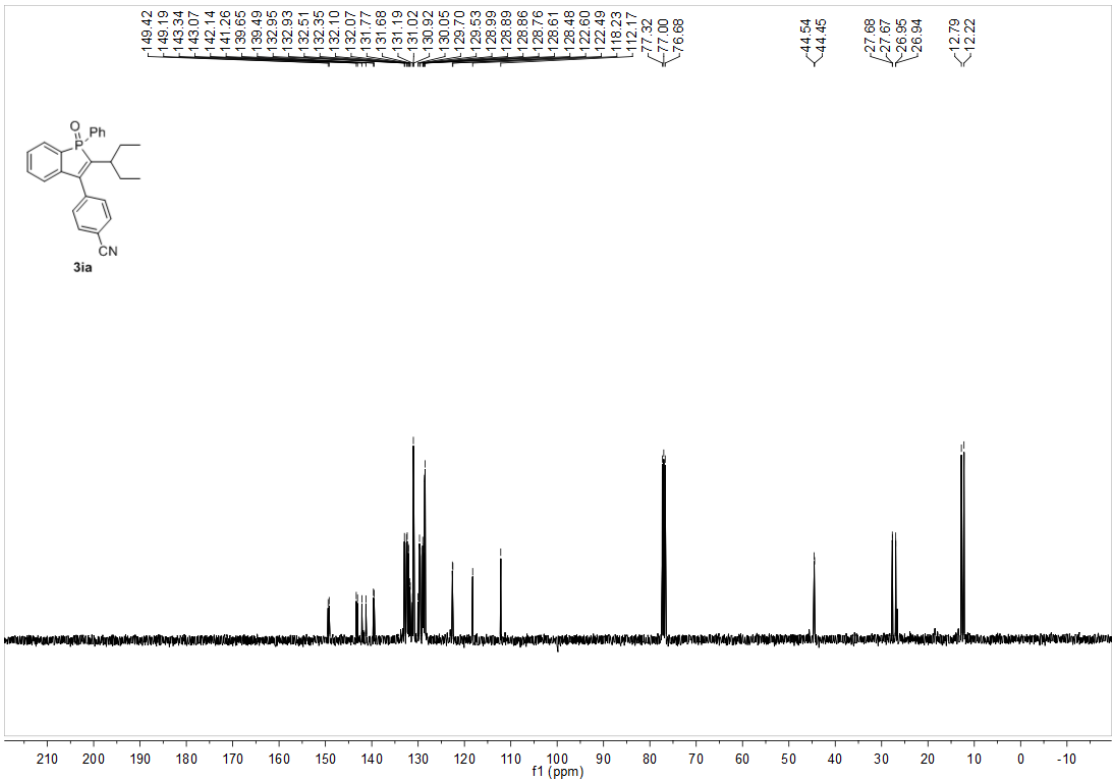
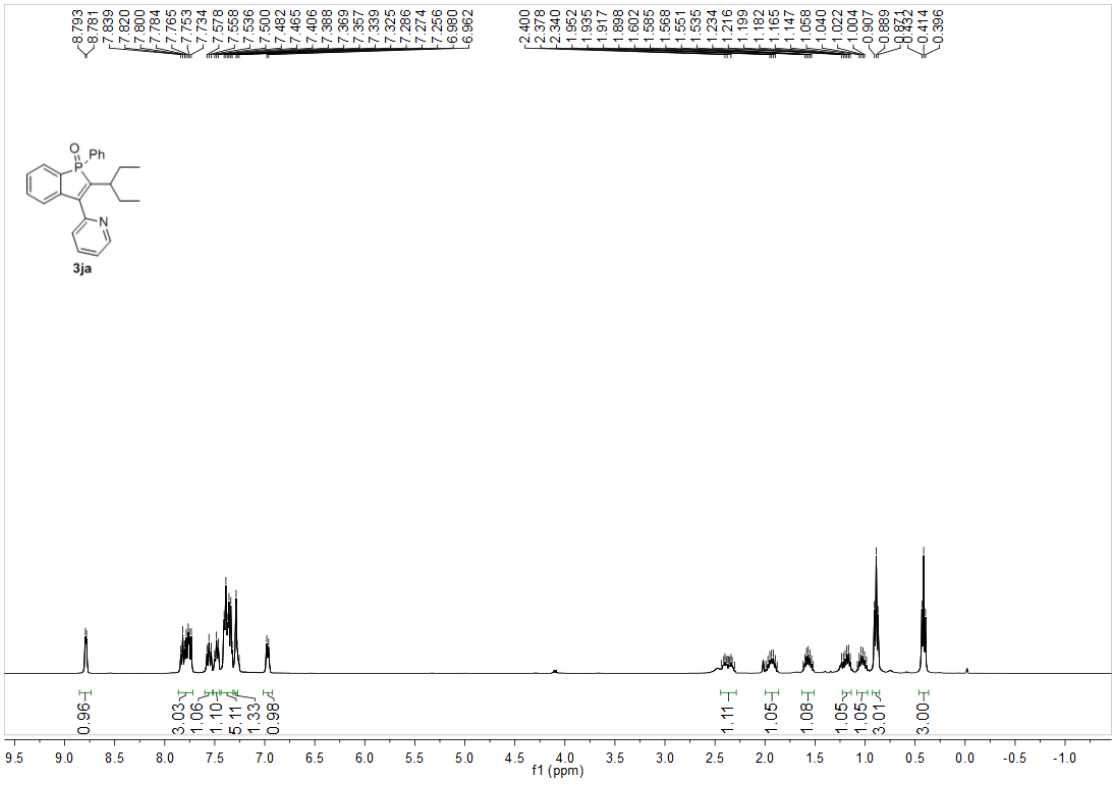
140.91
138.56
138.40
132.92
132.90
132.40
132.04
132.02
131.35
131.11
131.01
130.53
130.38
130.21
129.32
129.13
128.89
128.79
128.69
128.61
128.49
125.80
125.76
125.72
125.67
125.55
125.51
122.79
122.68
77.32
77.00
76.68
44.50
44.41
27.77
27.76
27.05
27.03
12.80
12.24
Ph
O
P
CF3
3ha
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.850
7.828
7.807
7.762
7.743
7.732
7.713
7.656
7.628
7.607
7.585
7.542
7.524
7.459
7.444
7.422
7.396
7.367
7.351
6.845
6.826
2.285
2.273
2.262
2.247
2.224
2.213
2.019
2.001
1.982
1.966
1.948
1.927
1.908
1.633
1.615
1.600
1.582
1.566
1.549
1.258
1.238
1.222
1.205
1.110
1.093
1.075
1.056
1.039
1.020
1.000
0.893
0.874
0.856
0.444
0.426
0.408
O
Ph
P
CN
3ia
2.11
1.97
3.02
5.14
0.98
0.97
0.92
1.15
1.25
1.11
3.00
2.93
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-1.0
-2.0
-3.0
f1 (ppm)


38.89
O
Ph
P
CN
3ia
42.0
41.5
41.0
40.5
40.0
39.5
39.0
38.5
38.0
37.5
37.0
36.5
36.0
35.5
35.0
34.5
34.0
f1 (ppm)


149.42
149.19
143.34
143.07
142.14
141.26
139.65
139.49
132.95
132.93
132.51
132.35
132.10
132.07
131.77
131.68
131.19
131.02
130.92
130.05
129.70
129.53
128.99
128.89
128.86
128.76
128.61
128.48
122.60
122.49
118.23
112.17
77.32
77.00
76.68
44.54
44.45
27.68
27.67
26.95
26.94
12.79
12.22
O
Ph
P
CN
3ia
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.839
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7.784
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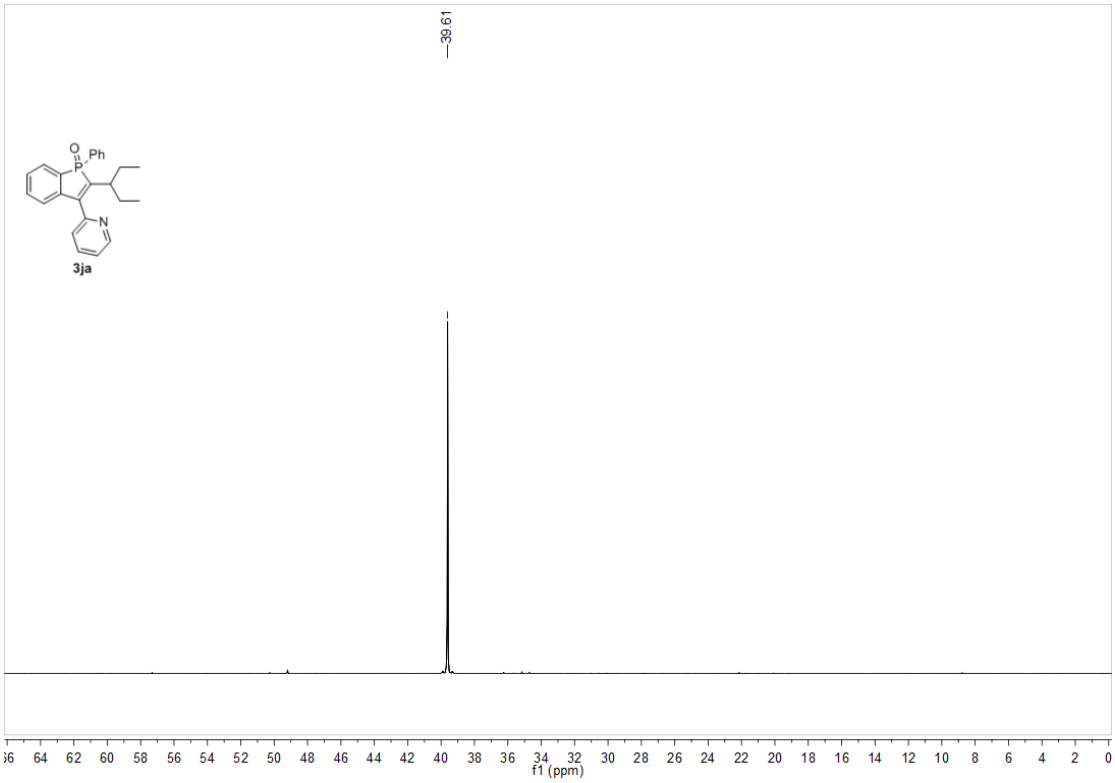
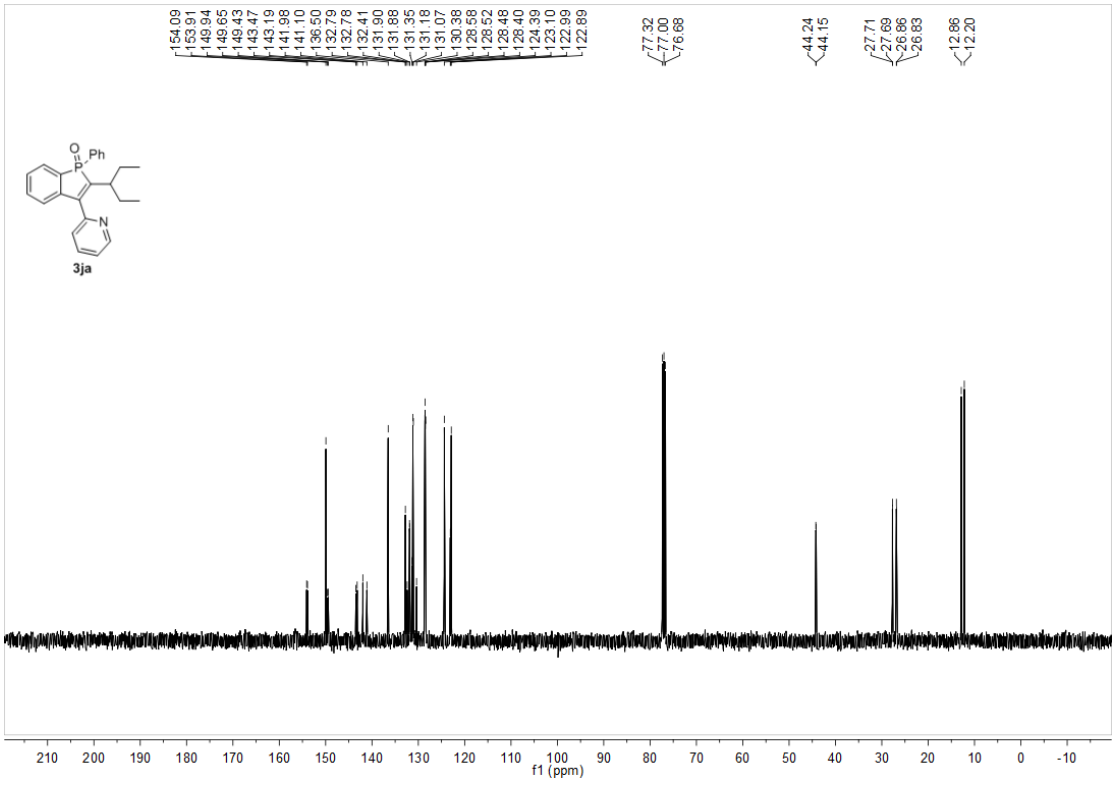
7.753
7.734
7.578
7.558
7.536
7.500
7.482
7.465
7.406
7.388
7.369
7.357
7.339
7.325
7.286
7.274
7.256
6.980
6.962
2.400
2.378
2.340
1.952
1.935
1.917
1.898
1.602
1.585
1.568
1.551
1.535
1.234
1.216
1.199
1.182
1.165
1.147
1.058
1.040
1.022
1.004
0.907
0.889
0.414
0.396
O
Ph
P
N
3ja
0.96
3.03
1.06
1.10
5.11
1.33
0.98
1.11
1.05
1.08
1.05
1.05
3.01
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


39.61
O
P
Ph
N
3ja
f1 (ppm)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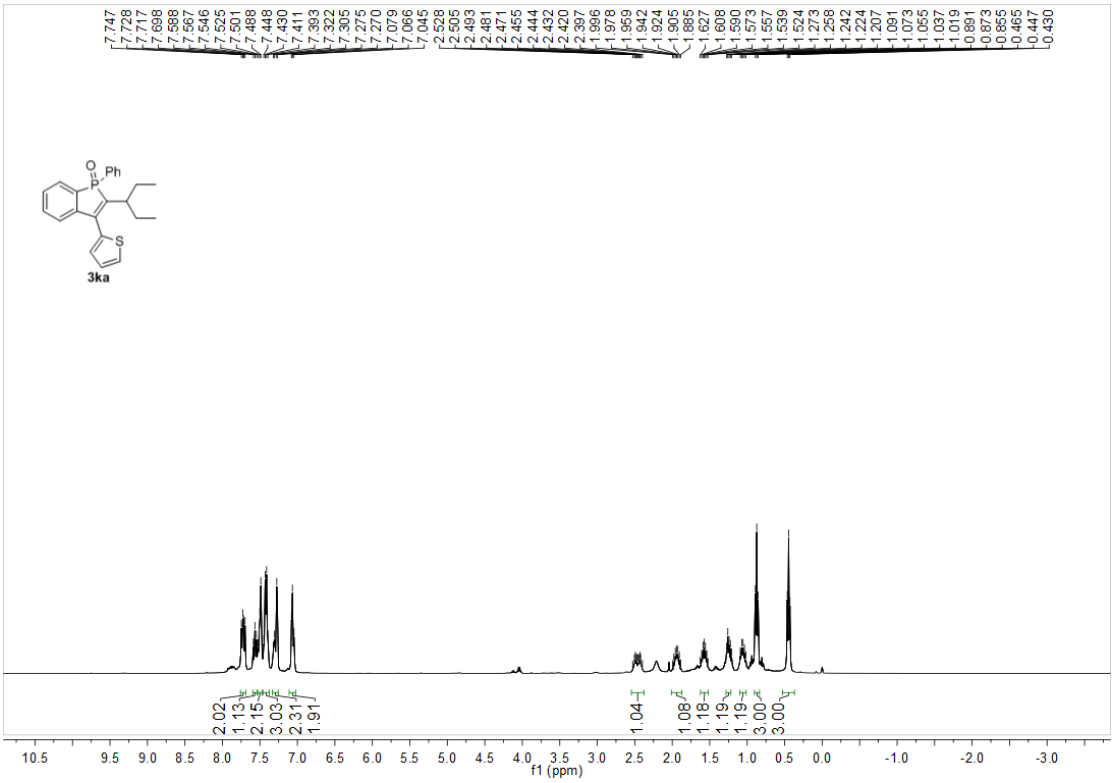
153.91
149.94
149.65
149.43
143.47
143.19
141.98
141.10
136.50
132.79
132.78
132.41
131.90
131.88
131.35
131.18
131.07
130.38
128.58
128.52
128.48
128.40
124.39
123.10
122.99
122.89
77.32
77.00
76.68
44.24
44.15
27.71
27.69
26.86
26.83
12.86
12.20
O
P
Ph
N
3ja
f1 (ppm)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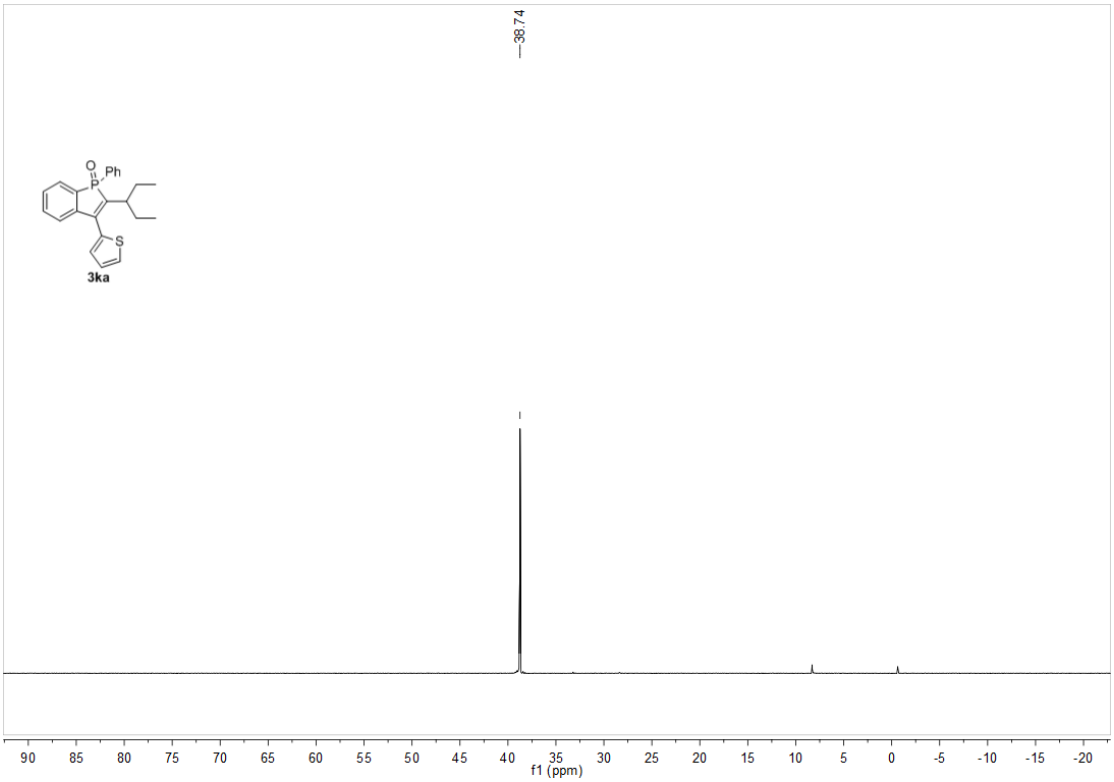
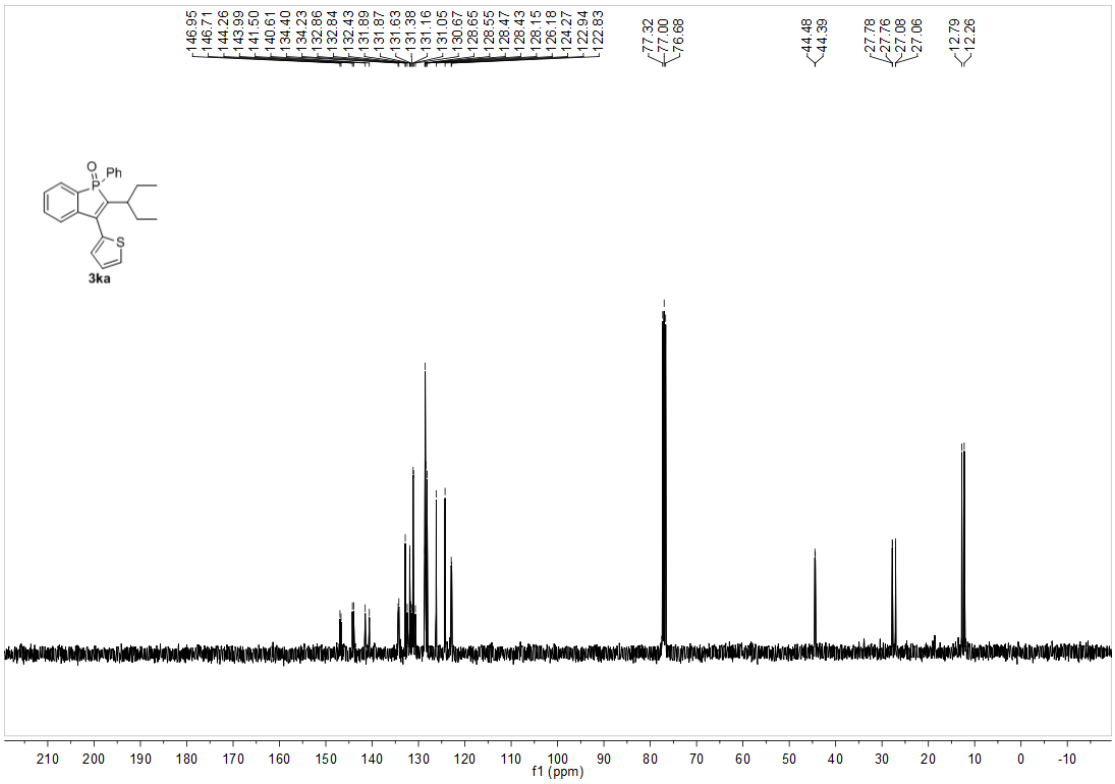
7.546
7.525
7.501
7.488
7.448
7.430
7.411
7.393
7.322
7.305
7.275
7.270
7.079
7.066
7.045
2.528
2.505
2.493
2.481
2.471
2.455
2.444
2.432
2.420
2.397
1.996
1.978
1.959
1.942
1.924
1.905
1.885
1.627
1.608
1.590
1.573
1.557
1.539
1.524
1.273
1.258
1.242
1.224
1.207
1.091
1.073
1.055
1.037
1.019
0.891
0.873
0.855
0.465
0.447
0.430
O
Ph
P
S
3ka
2.02
1.13
2.15
3.03
2.31
1.91
1.04
1.08
1.18
1.19
1.19
3.00
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-1.0
-2.0
-3.0
f1 (ppm)


38.74
O
Ph
P
S
3ka
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
f1 (ppm)


146.95
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141.50
140.61
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134.23
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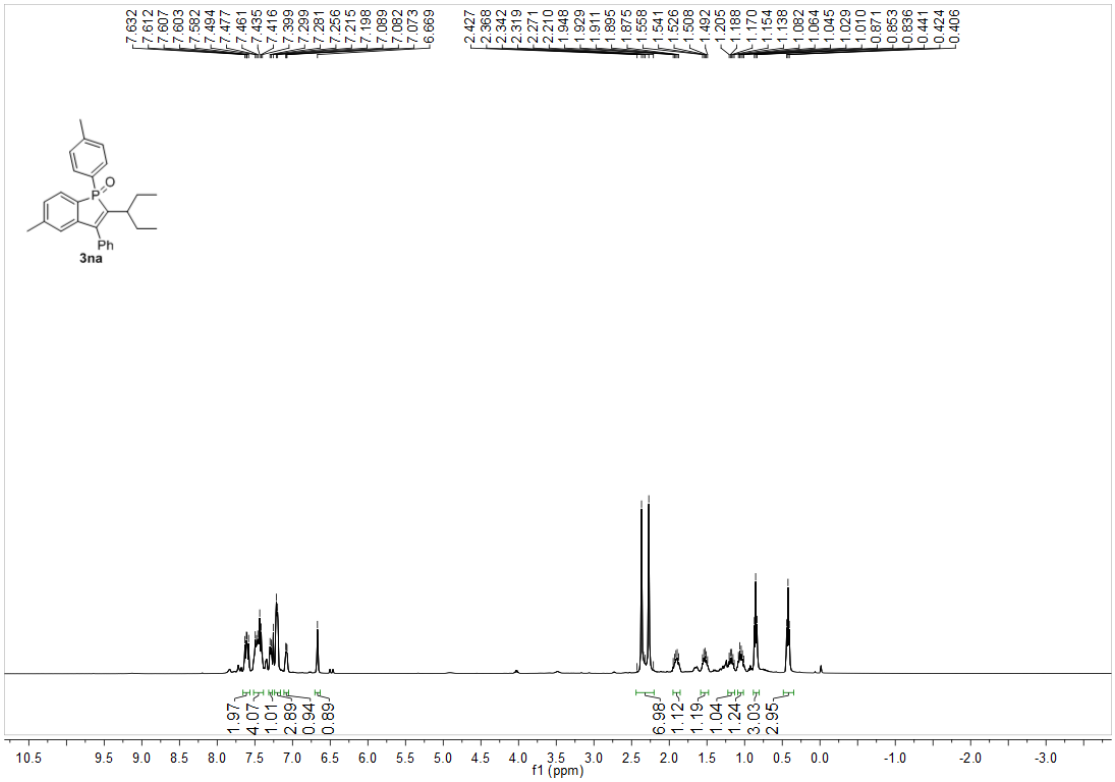
132.84
132.43
131.89
131.87
131.63
131.38
131.16
131.05
130.67
128.65
128.55
128.47
128.43
128.15
126.18
124.27
122.94
122.83
77.32
77.00
76.68
44.48
44.39
27.78
27.76
27.08
27.06
12.79
12.26
O
P
Ph
S
3ka
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.461
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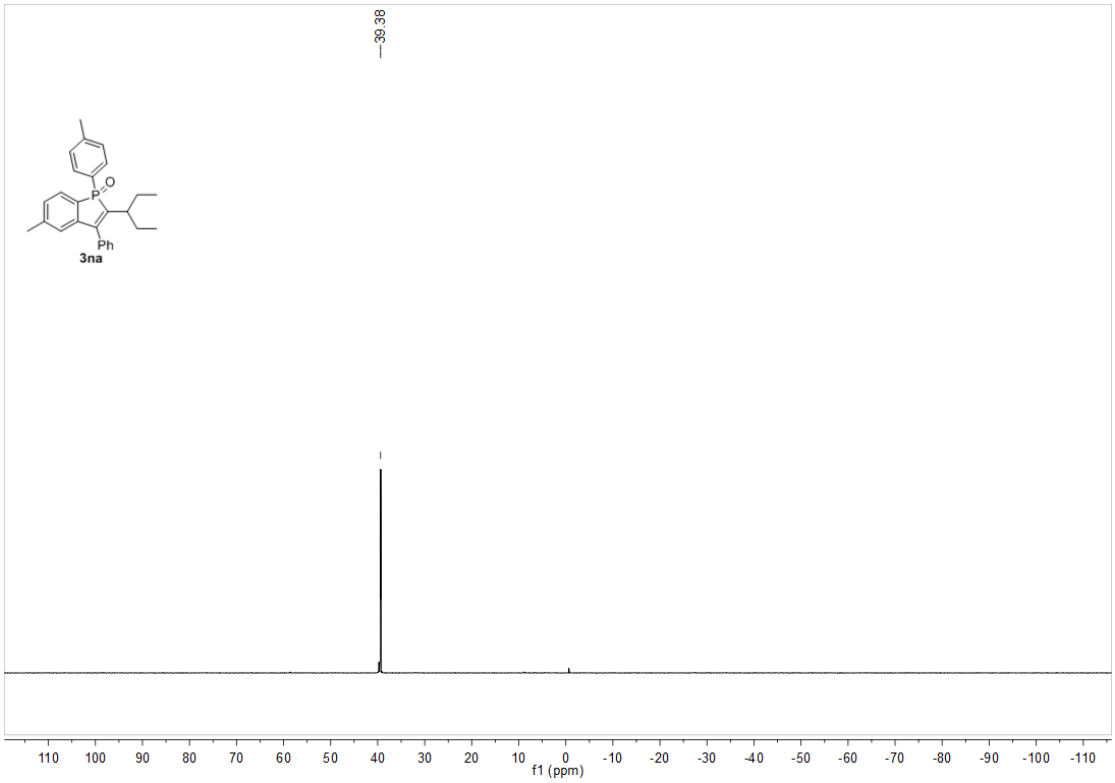
7.416
7.399
7.299
7.281
7.256
7.215
7.198
7.089
7.082
7.073
6.669
2.427
2.368
2.342
2.319
2.271
2.210
1.948
1.929
1.911
1.895
1.875
1.558
1.541
1.526
1.508
1.492
1.205
1.188
1.170
1.154
1.138
1.082
1.064
1.045
1.029
1.010
0.871
0.853
0.836
0.441
0.424
0.406
O
P
Ph
3na
1.97
4.07
1.01
2.89
0.94
0.89
6.98
1.12
1.19
1.04
1.24
3.03
2.95
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0 -2.0 -3.0
f1 (ppm)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Ph
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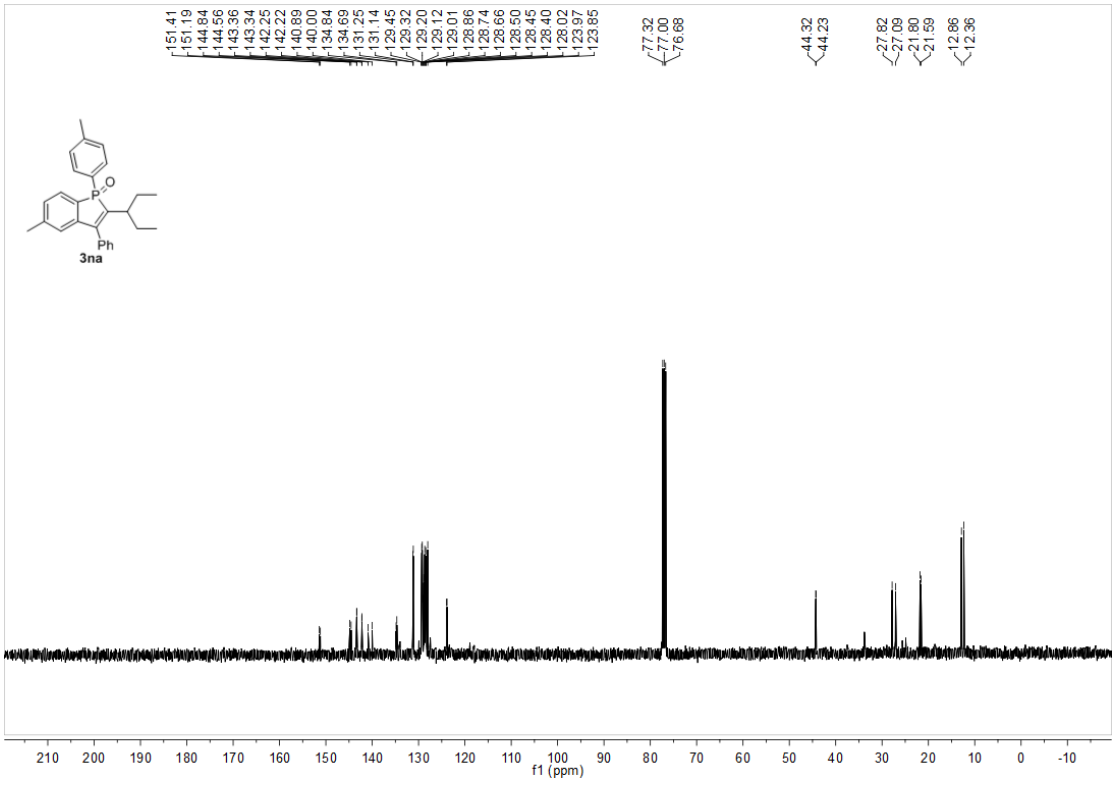
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110
f1 (ppm)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142.22
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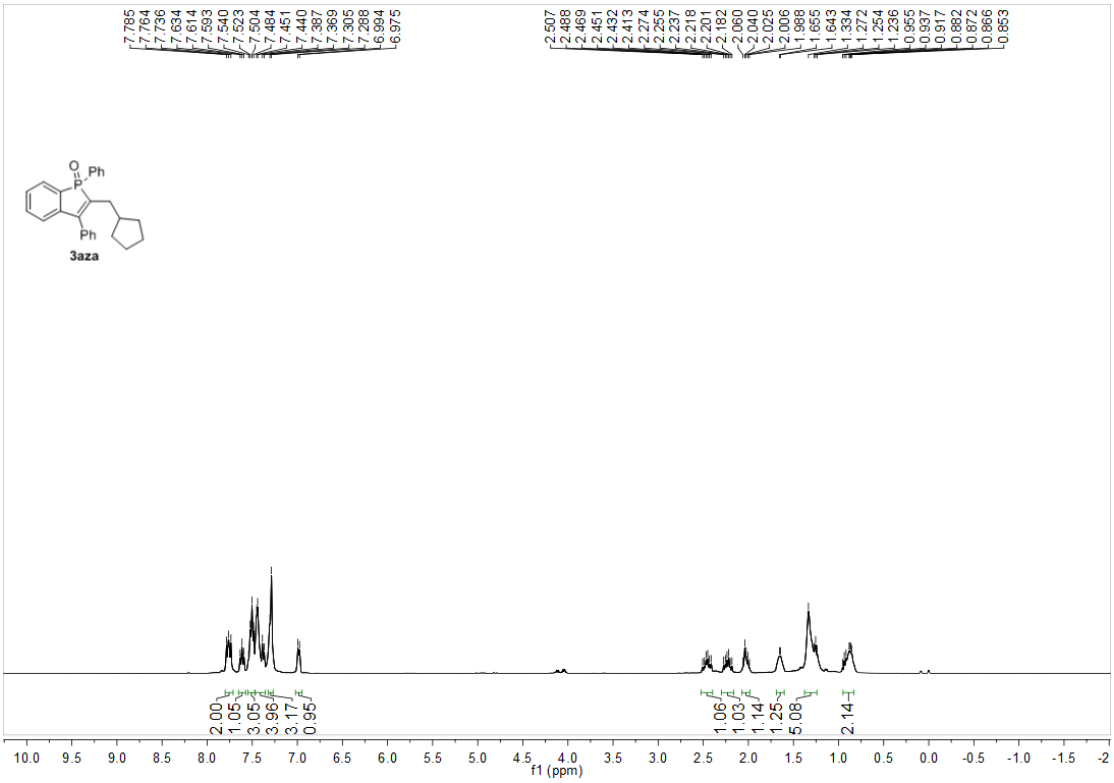
140.00
134.84
134.69
131.25
131.14
129.45
129.32
129.20
129.12
129.01
128.86
128.74
128.66
128.50
128.45
128.40
128.02
123.97
123.85
77.32
77.00
76.68
44.32
44.23
27.82
27.09
21.80
21.59
12.86
12.36
Ph
3na
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.523
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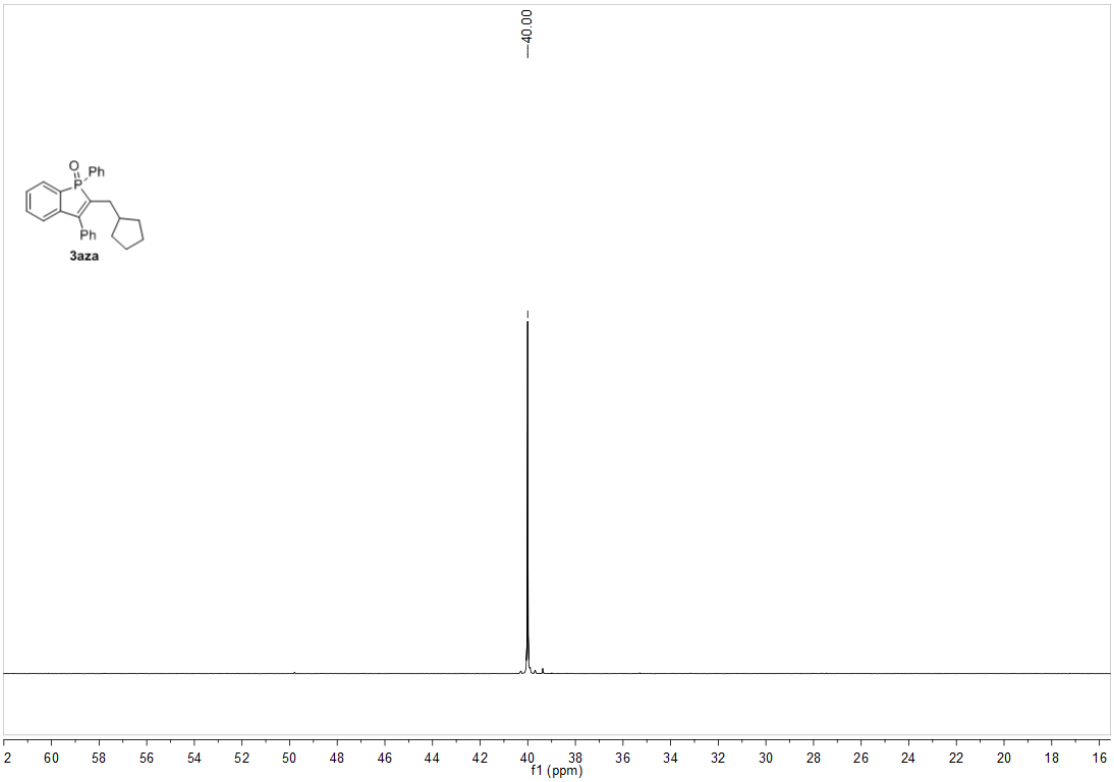
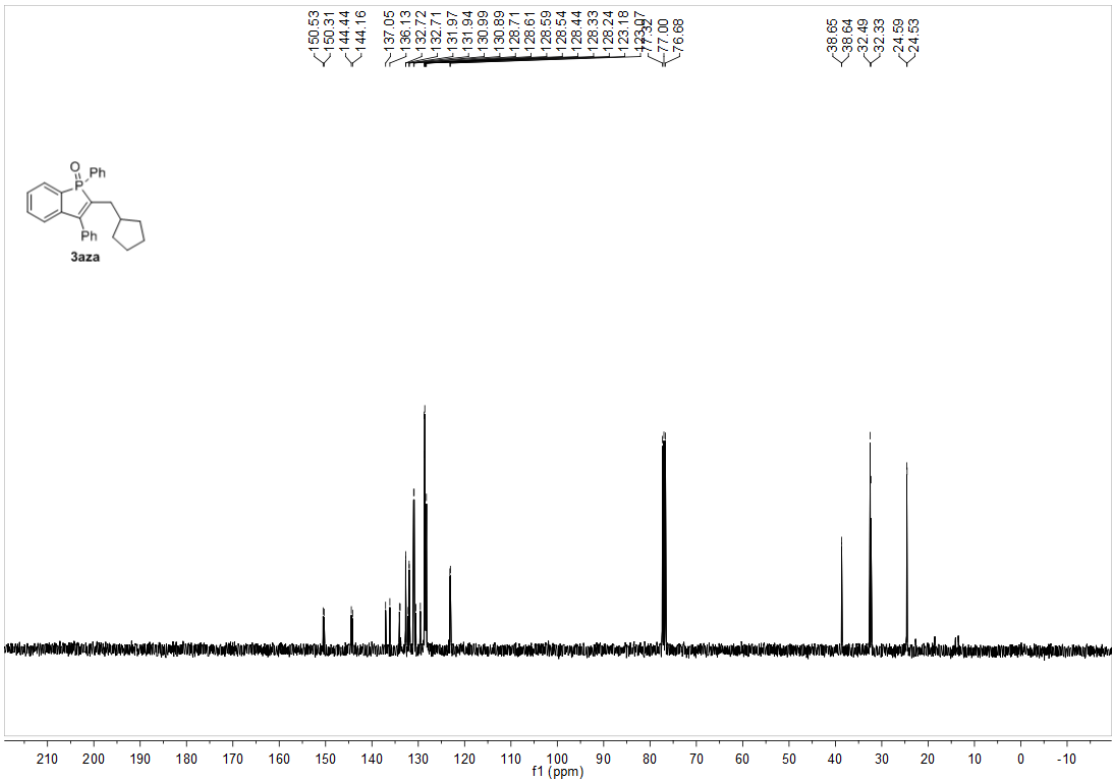
7.484
7.451
7.440
7.387
7.369
7.305
7.288
6.994
6.975
2.507
2.488
2.469
2.451
2.432
2.413
2.274
2.255
2.237
2.218
2.201
2.182
2.060
2.040
2.025
2.006
1.988
1.655
1.643
1.334
1.272
1.254
1.236
0.955
0.937
0.917
0.882
0.872
0.866
0.853
O
P
Ph
Ph
3aza
2.00
1.05
3.05
3.96
3.17
0.95
1.06
1.03
1.14
1.25
5.08
2.14
f1 (ppm)


40.00
O
P
Ph
Ph
3aza
f1 (ppm)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136.13
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131.94
130.99
130.89
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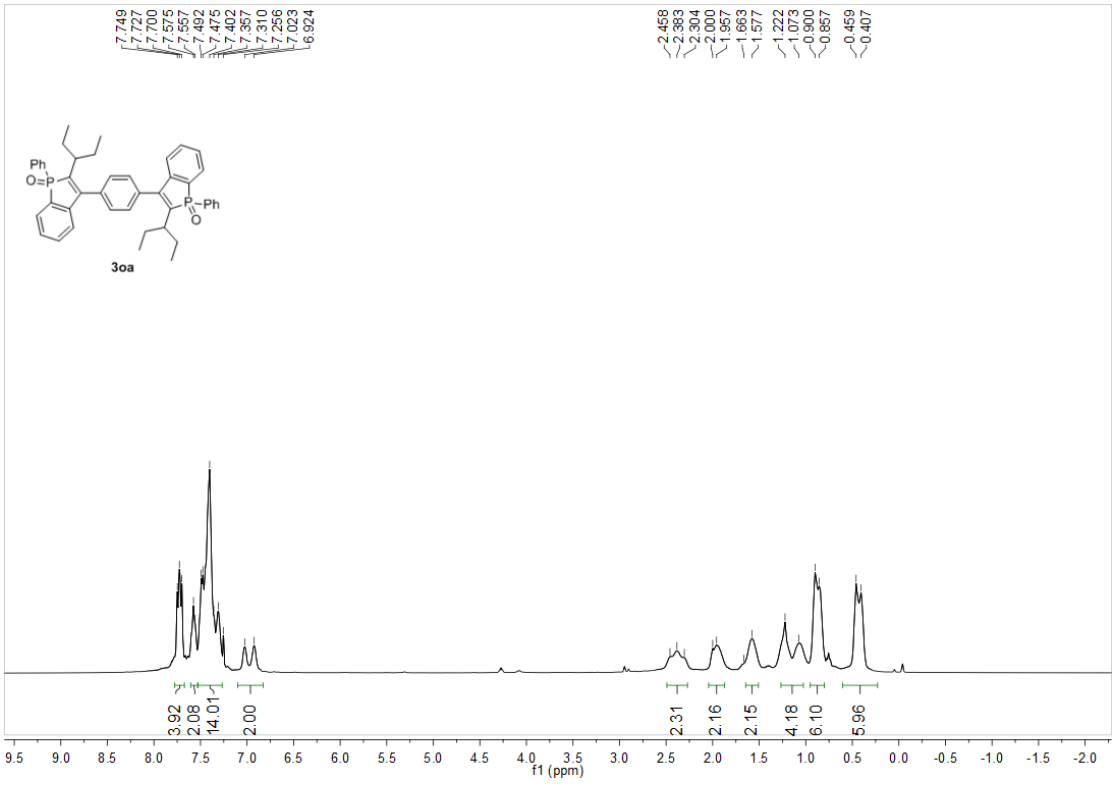
128.61
128.59
128.54
128.44
128.33
128.24
123.18
77.32
77.00
76.68
38.65
38.64
32.49
32.33
24.59
24.53
Ph
O
P
Ph
3aza
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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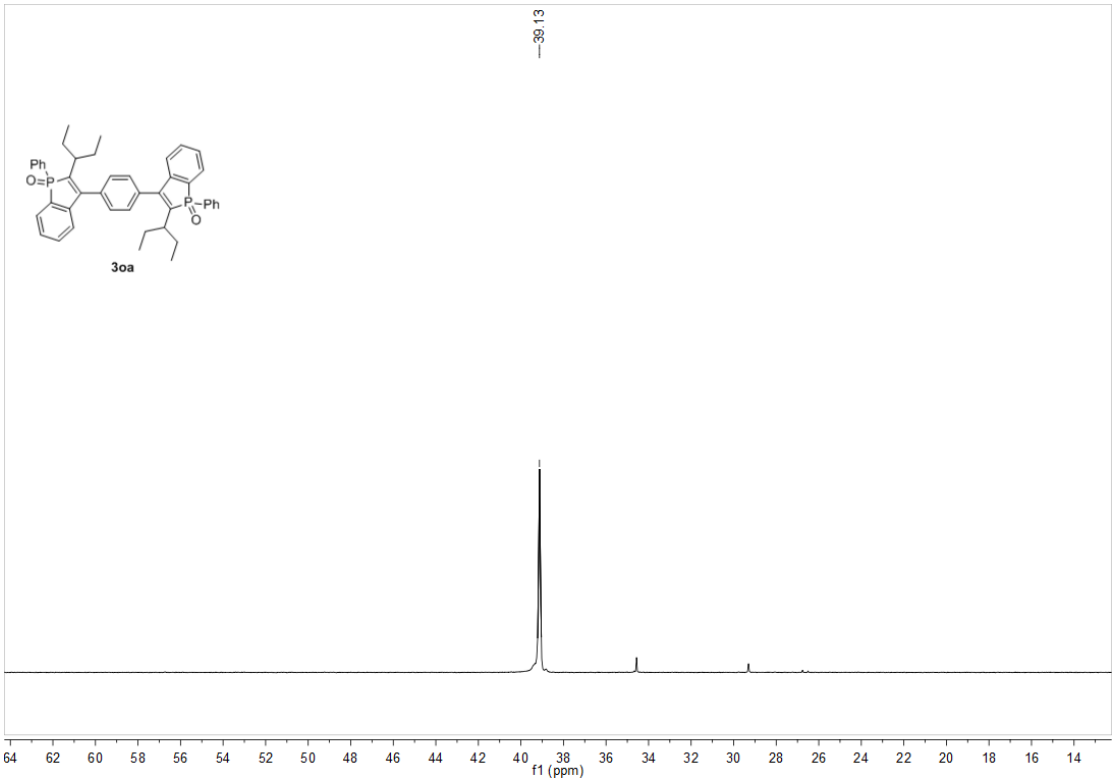
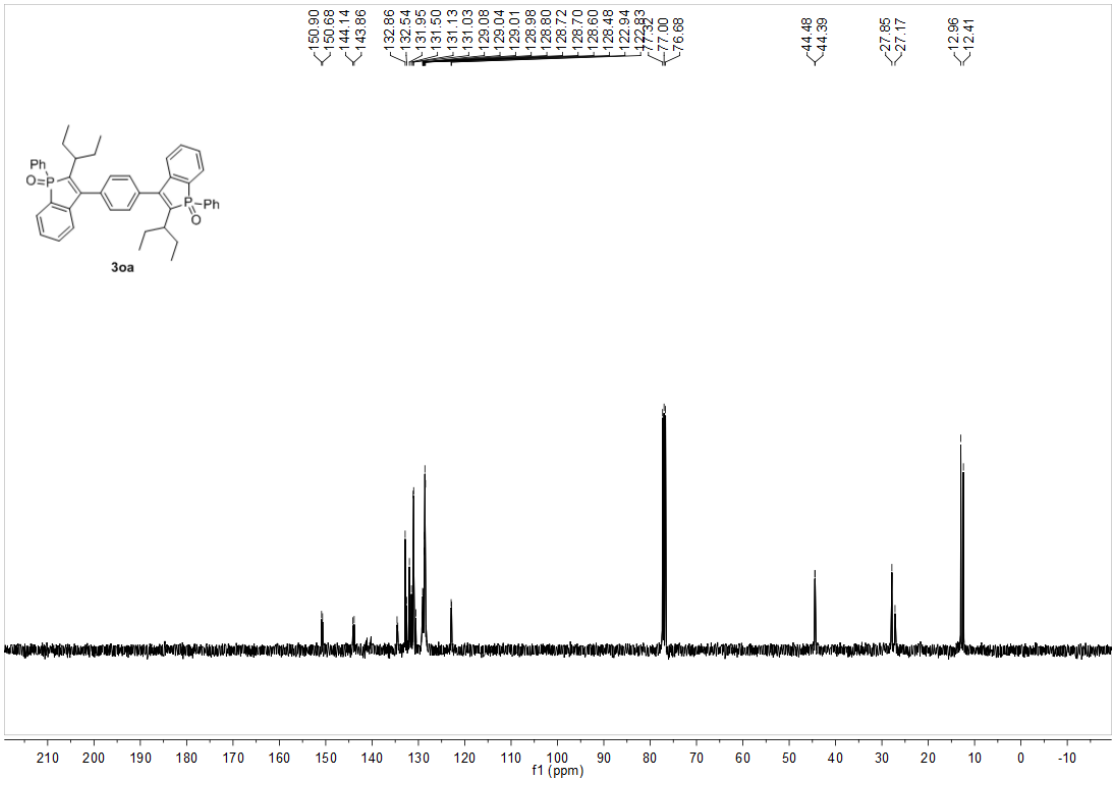
7.492
7.475
7.402
7.357
7.310
7.256
7.023
6.924
2.458
2.383
2.304
2.000
1.957
1.663
1.577
1.222
1.073
0.900
0.857
0.459
0.407
Ph
O
P
P
Ph
O
3oa
3.92
2.08
14.01
2.00
2.31
2.16
2.15
4.18
6.10
5.96
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)


39.13
Ph
O=P
P-Ph
O
3oa
64 62 60 58 56 54 52 50 48 46 44 42 40 38 36 34 32 30 28 26 24 22 20 18 16 14
f1 (ppm)


150.90
150.68
144.14
143.86
132.86
132.54
131.95
131.50
131.13
131.03
129.08
129.04
129.01
128.98
128.80
128.72
128.70
128.60
128.48
122.94
122.83
77.32
77.00
76.68
44.48
44.39
27.85
27.17
12.96
12.41
Ph
O=P
P-Ph
O
3oa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)