

Supporting Information

Mn-Catalyzed Electrooxidative Radical Phosphorylation of 2-isocyanobiaryls

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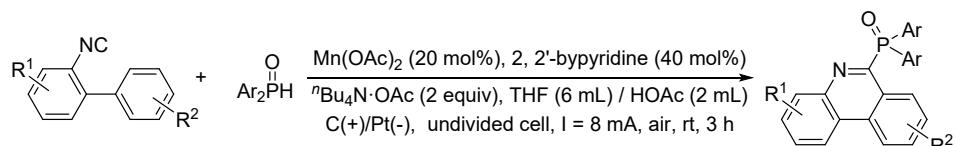
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1. General Information

Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. For chromatography, 200-300 mesh silica gel (Qingdao, China) was employed. ^1H NMR and ^{13}C NMR were recorded on a Bruker 400 MHz and 500 MHz spectrometer. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz respectively. ^1H NMR spectra were referenced to the solvent residual peak (TMS, δ 0 ppm) and ^{13}C NMR spectra were referenced to the solvent residual peak (CDCl_3 , δ 77.0 ppm). The following abbreviations are used to illuminate the diversities: δ , chemical shift; J , coupling constant; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. HRMS was obtained on Waters Xevo G2-XS QToF with ESI source. 2-isocyanobiaryls^[1] and diarylphosphine oxides^[2] were prepared according to the literature procedures. Compounds **3aa-3ca**, **3ea-3ha**, **3oa**, **3qa-3sa**, **3va**, **3ad-3af**, **3ah** are known.

2. General Procedure for Mn-Catalyzed Electrooxidative Radical Phosphorylation of 2-isocyanobiaryls



To an overdried undivided cell equipped with a stir bar, 2-isocyanobiaryl (0.4 mmol), diaryl phosphine oxide (0.8 mmol), Mn(OAc)₂ (0.08 mmol), 2, 2'-bipyridine (0.16 mmol), ⁿBu₄N•OAc (0.8 mmol), THF (6 mL) and HOAc (2 mL) were added. The flask was equipped with a graphite rod (ϕ 7 mm, about 15 mm immersion depth in solution) as the anode and a platinum plate (15 mm \times 15 mm \times 0.3 mm) as the cathode. After that, the reaction mixture was stirred and electrolyzed at a constant current of 8 mA under room temperature for 3 h (2.25 F/mol). After the reaction was finished, the reaction mixture was concentrated and purified by flash column chromatography on silica gel with petroleum and ethyl acetate as eluent to give the desired product.

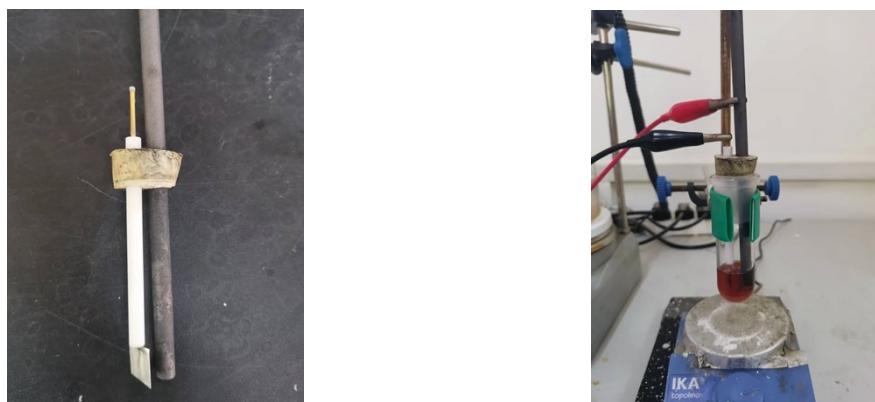
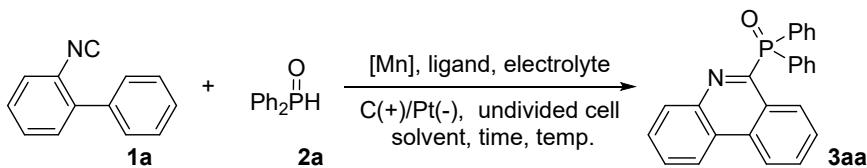


Fig. S1: General electrolysis setup

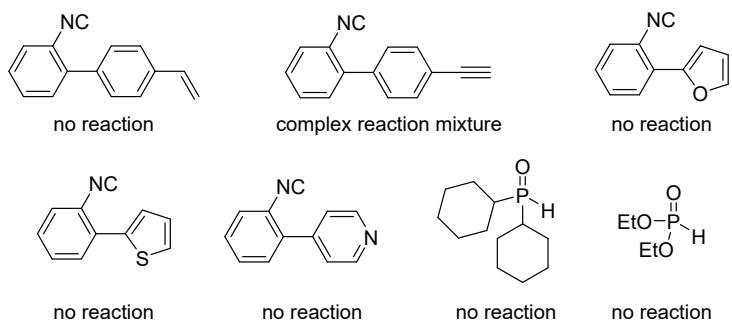
3. Optimization of Reaction Conditions



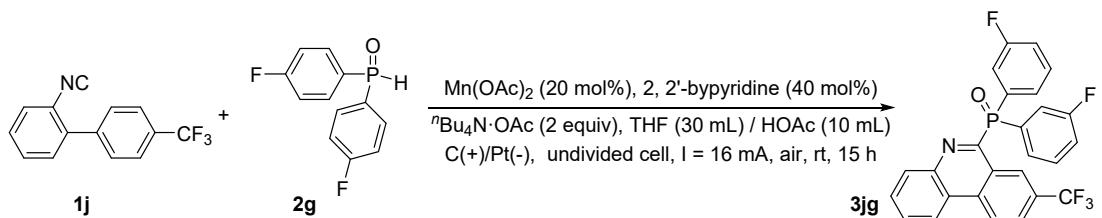
entry	2a	catalyst	ligand	electrolyte	solvent	yield (%)
1	1.2 equiv.	Mn(OAc) ₂	1,10-phen	Me ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	38
2	1.2 equiv.	Mn(OAc) ₂	1,10-phen	Me ₄ N·OAc (1 equiv)	CH ₃ CN (4 mL) / HOAc (4 mL)	20
3	1.2 equiv.	Mn(OAc) ₂	1,10-phen	Me ₄ N·OAc (1 equiv)	DMF (4 mL) / HOAc (4 mL)	35
4	1.2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	41
5	1.2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·PF ₆ (1 equiv)	THF (4 mL) / HOAc (4 mL)	trace
6	1.2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	trace
7	1.2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HFIP (4 mL)	24
8	1.2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / TFA (4 mL)	33
9	2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	44
10	1.2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	44
11	2 equiv.	Mn(OAc) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	48
12	2 equiv.	Mn(OTf) ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	40
13	2 equiv.	MnCl ₂	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	38
14	2 equiv.	Mn(acac) ₃	1,10-phen	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	45
15	2 equiv.	Mn(OAc) ₂	—	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	30
16	2 equiv.	—	—	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	0
17	2 equiv.	Mn(OAc) ₂	2, 2'-bipyridine	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	58
18	2 equiv.	Mn(OAc) ₂	4,4'-DTBDP _y	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	44
19	2 equiv.	Mn(OAc) ₂	Bipicoline	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	33
20 ^b	2 equiv.	Mn(OAc) ₂	2, 2'-bipyridine	ⁿ Bu ₄ N·OAc (1 equiv)	THF (4 mL) / HOAc (4 mL)	54
21	2 equiv.	Mn(OAc) ₂	2, 2'-bipyridine	ⁿ Bu ₄ N·OAc (1 equiv)	THF (6 mL) / HOAc (2 mL)	68
22 ^c	2 equiv.	Mn(OAc) ₂	2, 2'-bipyridine	ⁿ Bu ₄ N·OAc (1 equiv)	THF (6 mL) / HOAc (2 mL)	59
23 ^d	2 equiv.	Mn(OAc) ₂	2, 2'-bipyridine	ⁿ Bu ₄ N·OAc (1 equiv)	THF (6 mL) / HOAc (2 mL)	54

^a Reaction conditions: **1a** (0.4 mmol), **2a**, [Mn] (20 mol%), ligand (40 mol%), electrolyte, solvent/HOAc, undivided cell, constant current = 8 mA, 3 h, graphite rod(+) / Pt plate(-), rt, air. ^b constant current = 5 mA, 5 h. ^c 60 °C, ^d [Mn] (10 mol%), 2, 2'-bipyridine (20 mol%).

4. Unsuccessful substrates



5. Procedure for Scale-up Experiment



To an overdried 100 mL beaker equipped with a stir bar, **1j** (2 mmol), **2g** (4 mmol), Mn(OAc)₂ (0.4 mmol), 2, 2'-bipyridine (0.8 mmol), *n*Bu₄N•OAc (4 mmol), THF (30 mL) and HOAc (10 mL) were added. A graphite felt, which is about 60 mm wide and 20 mm high and thread it with copper wire, was connected with two electrode holders as anode, the length of the graphite felt in the solution is about 10mm. A platinum plate (15 mm × 15 mm × 0.3 mm) was equipped as cathode. After the electrodes are fixed, the reaction mixture was stirred and electrolyzed at a constant current of 16 mA under room temperature for 15 h. After the reaction was finished, the reaction mixture was concentrated and purified by flash column chromatography on silica gel with petroleum and ethyl acetate as eluent to give the desired product **3jg** as white solid.

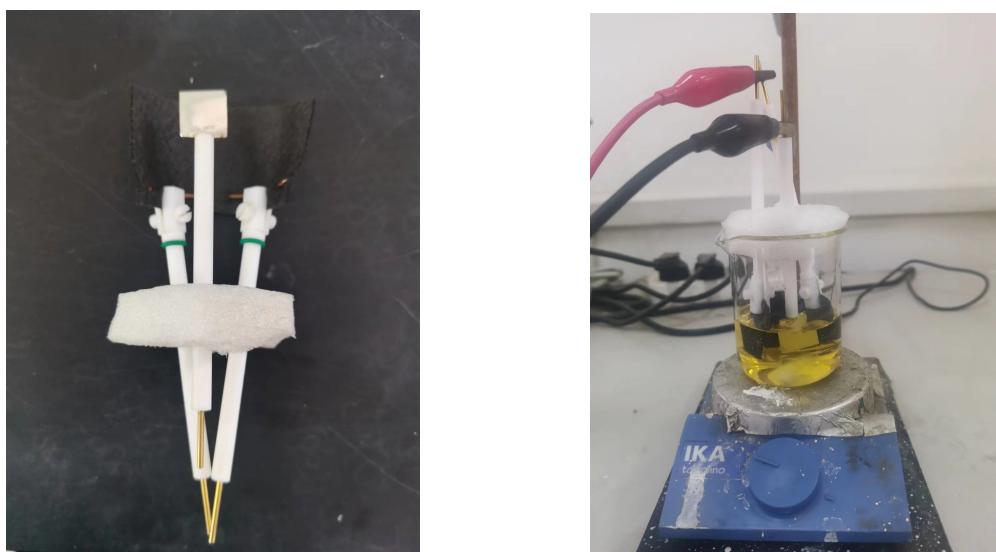
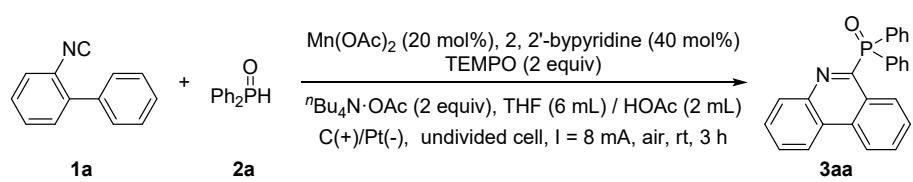


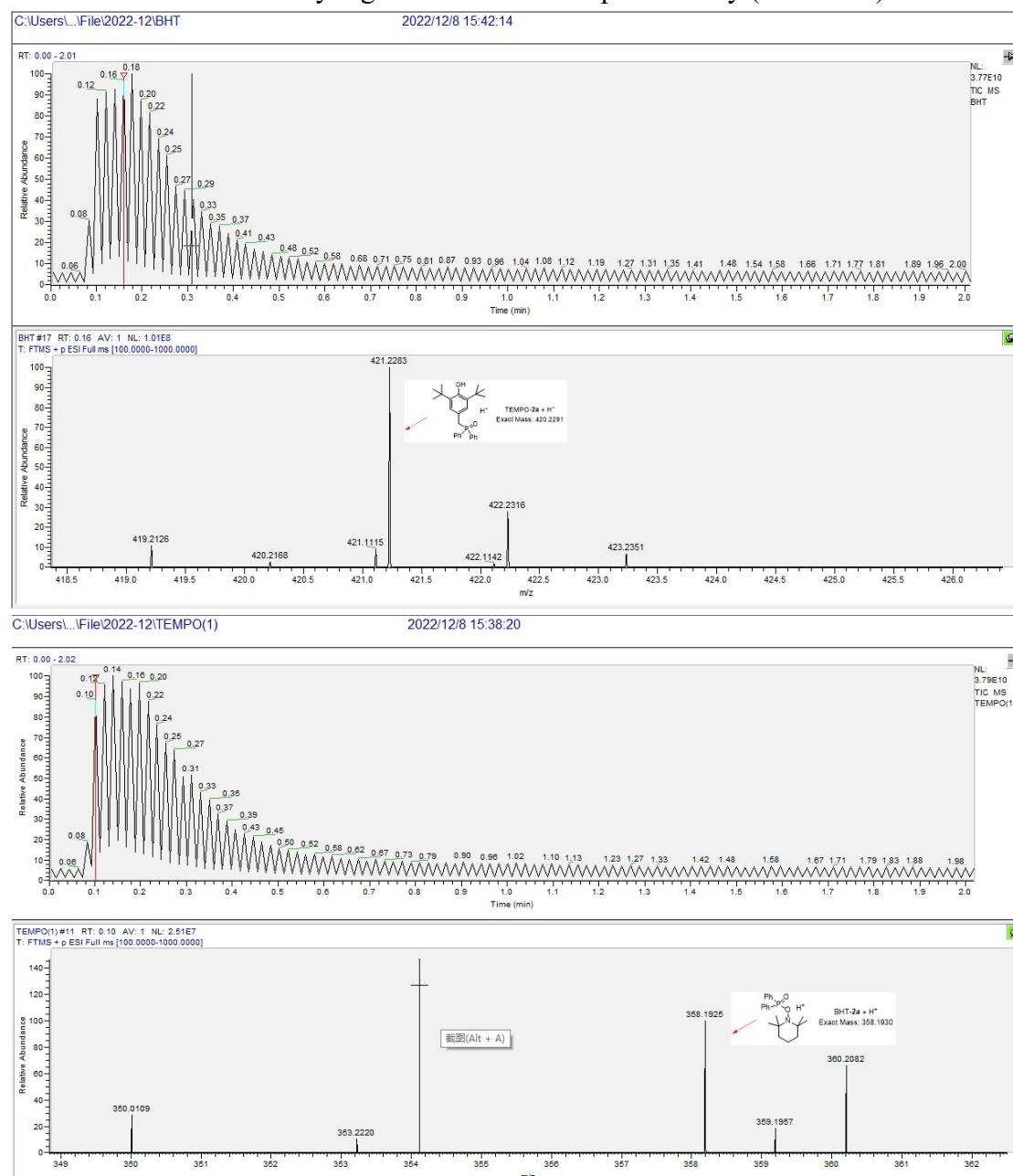
Fig. S2: Scale-up electrolysis setup

6. Procedure for Experimental Probe Reaction



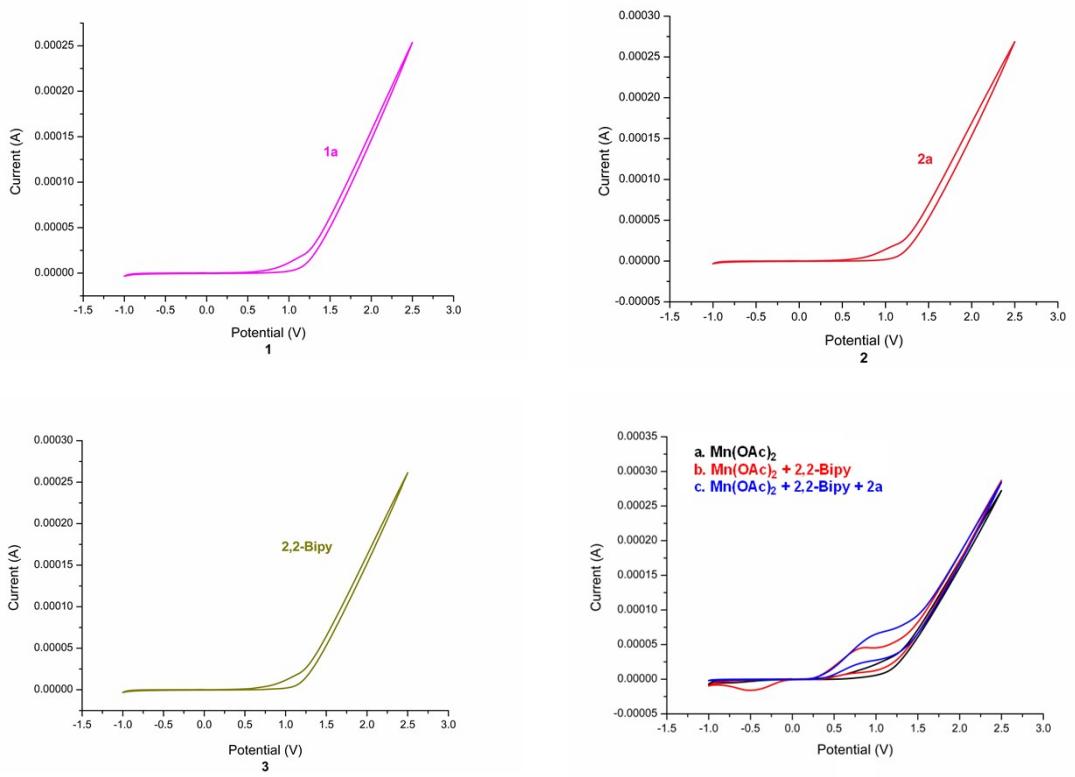
To an overdried undivided cell equipped with a stir bar, **1a** (0.4 mmol), **2a** (0.8 mmol), TEMPO (0.8 mmol) or BHT (0.8 mmol), Mn(OAc)₂ (0.08 mmol), 2, 2'-bipyridine (0.16 mmol), *n*Bu₄N•OAc (0.8 mmol), THF (6 mL) and HOAc (2 mL) were added. The flask was equipped with a graphite rod (ϕ 7 mm, about 15 mm immersion depth in solution) as the anode and a platinum plate (15 mm × 15 mm ×

0.3 mm) as the cathode. After that, the reaction mixture was stirred and electrolyzed at a constant current of 8 mA under room temperature for 3 h. After the reaction was finished, TLC was used to detect the mount of **3aa**. The TEMPO-**2a** adduct and BHT-**2a** adduct were detected by high-resolution mass spectrometry (see below).



7. Cyclic Voltammetric Experiments

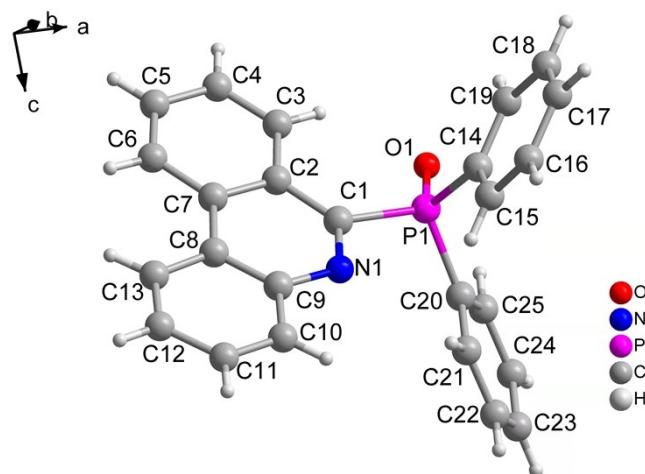
Cyclic voltammetry (CV) experiments were conducted in a 20 mL glass vial fitted with a glassy carbon working electrode (3 mm in diameter), a Ag/AgNO₃ (0.1 M in CH₃CN) reference electrode, and a platinum wire counter electrode. 15 mL THF and 5 mL HOAc containing 0.1 M ⁿBu₄N•OAc and substrate were poured into the electrochemical cell in all cyclic voltammetry experiments. The scan rate was 0.1 V/s, ranging from -1 V to 3 V.



Conditions: (1) 15 mL THF, 5 mL HOAc, 2 mmol $^{n}\text{Bu}_4\text{N}\bullet\text{OAc}$, 0.4 mmol **1a**; (2) 15 mL THF, 5 mL HOAc, 2 mmol $^{n}\text{Bu}_4\text{N}\bullet\text{OAc}$, 0.4 mmol **2a**; (3) 15 mL THF, 5 mL HOAc, 2 mmol $^{n}\text{Bu}_4\text{N}\bullet\text{OAc}$, 0.4 mmol 2,2-Bipy; (4) (a) 15 mL THF, 5 mL HOAc, 2 mmol $^{n}\text{Bu}_4\text{N}\bullet\text{OAc}$, 0.2 mmol $\text{Mn}(\text{OAc})_2$; (b) 15 mL THF, 5 mL HOAc, 2 mmol $^{n}\text{Bu}_4\text{N}\bullet\text{OAc}$, 0.2 mmol $\text{Mn}(\text{OAc})_2$, 0.4 mmol 2,2-Bipy; (c) 15 mL THF, 5 mL HOAc, 2 mmol $^{n}\text{Bu}_4\text{N}\bullet\text{OAc}$, 0.2 mmol $\text{Mn}(\text{OAc})_2$, 0.4 mmol 2,2-Bipy, 0.4 mmol **2a**.

Fig. S3: Cyclic voltammetry studies

8. Crystal Data of 3aa



Bond precision: C-C = 0.0033 Å

Wavelength=0.71073

Cell: $a=13.278(5)$ $b=8.402(3)$ $c=17.431(7)$
 $\alpha=90$ $\beta=90.161(6)$ $\gamma=90$

Temperature: 296 K

	Calculated	Reported
Volume	1944.6(13)	1944.8(13)
Space group	P 21/c	P2(1) /c
Hall group	-P 2ybc	?
Moiety formula	C25 H18 N O P	?
Sum formula	C25 H18 N O P	C25 H18 N O P
Mr	379.37	379.37
Dx, g cm ⁻³	1.296	1.296
Z	4	4
Mu (mm ⁻¹)	0.157	0.156
F000	792.0	792.0
F000'	792.70	
h, k, lmax	15, 9, 20	15, 9, 20
Nref	3413	3410
Tmin, Tmax	0.945, 0.959	0.946, 0.959
Tmin'	0.945	

Correction method= # Reported T Limits: Tmin=0.946 Tmax=0.959
AbsCorr = MULTI-SCAN

Data completeness= 0.999

Theta(max)= 25.000

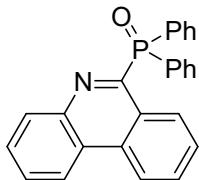
R(reflections)= 0.0444(2799)

wR2(reflections)=
0.1182(3410)

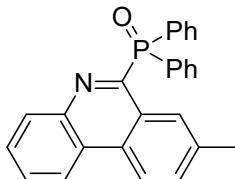
S = 1.073

Npar= 253

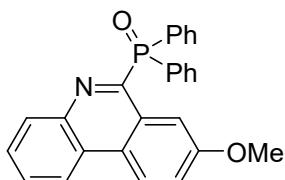
9. Characterization Data



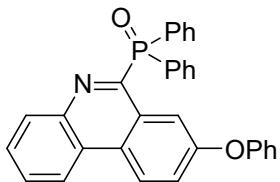
phenanthridin-6-yl diphenylphosphine oxide (3aa)^[1a]: white solid, m.p. 200.5-201.6 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.52 (d, *J* = 8.2 Hz, 1H), 8.68-8.49 (m, 2H), 8.09-8.02 (m, 1H), 8.00-7.88 (m, 4H), 7.86-7.78 (m, 1H), 7.68 (ddd, *J* = 8.6, 7.2, 2.4 Hz, 3H), 7.51 (ddd, *J* = 7.2, 2.8, 1.4 Hz, 2H), 7.48-7.40 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 156.83 (d, *J* = 128.4 Hz), 142.73 (d, *J* = 23.4 Hz), 133.41 (s), 132.59 (d, *J* = 6.9 Hz), 132.39 (s), 132.30 (s), 131.76 (d, *J* = 2.5 Hz), 131.16 (s), 131.08 (s), 128.80 (d, *J* = 12.6 Hz), 128.56 (s), 128.23 (d, *J* = 12.2 Hz), 127.93 (s), 127.73 (s), 124.34 (d, *J* = 2.2 Hz), 122.17 (d, *J* = 1.1 Hz), 122.13 (d, *J* = 0.9 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 28.32 (s).



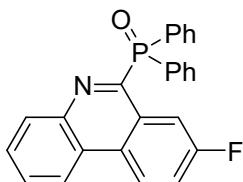
(8-methylphenanthridin-6-yl)diphenylphosphine oxide (3ba)^[1a]: white solid, m.p. 210.4-211.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.32 (s, 1H), 8.54 (dd, *J* = 7.0, 2.0 Hz, 2H), 8.10-8.01 (m, 1H), 7.99-7.88 (m, 4H), 7.69 (qd, *J* = 7.0, 3.4 Hz, 3H), 7.57-7.48 (m, 2H), 7.45 (td, *J* = 7.4, 2.9 Hz, 4H), 2.56 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 156.23 (d, *J* = 128.8 Hz), 142.43 (d, *J* = 23.4 Hz), 138.11 (s), 133.61 (s), 132.95 (s), 132.57 (s), 132.36 (d, *J* = 9.2 Hz), 131.67 (d, *J* = 2.7 Hz), 131.06 (d, *J* = 1.2 Hz), 130.50 (d, *J* = 6.9 Hz), 128.75 (s), 128.24 (s), 128.11 (s), 127.71 (s), 124.45 (d, *J* = 2.5 Hz), 122.02 (d, *J* = 1.5 Hz), 121.95 (d, *J* = 0.6 Hz), 21.99 (s). ³¹P NMR (162 MHz, CDCl₃) δ 28.23 (s).



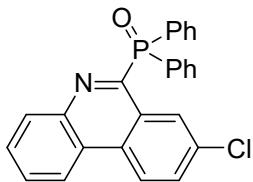
(8-methoxyphenanthridin-6-yl)diphenylphosphine oxide (3ca)^[1a]: white solid, m.p. 104.6-105.7 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.02 (d, *J* = 2.6 Hz, 1H), 8.57-8.44 (m, 2H), 8.05 (d, *J* = 7.7 Hz, 1H), 8.01-7.92 (m, 4H), 7.73-7.60 (m, 2H), 7.52 (dd, *J* = 7.3, 6.5 Hz, 2H), 7.45 (td, *J* = 7.3, 2.8 Hz, 5H), 3.94 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 158.81 (s), 155.42 (d, *J* = 129.4 Hz), 142.07 (d, *J* = 23.2 Hz), 133.53 (s), 132.49 (s), 132.33 (d, *J* = 9.2 Hz), 131.71 (d, *J* = 2.7 Hz), 131.09 (s), 129.45 (d, *J* = 23.1 Hz), 128.86 (s), 128.21 (d, *J* = 12.1 Hz), 127.68 (s), 127.07 (d, *J* = 6.8 Hz), 124.55 (d, *J* = 2.4 Hz), 123.67 (d, *J* = 1.6 Hz), 122.66 (s), 121.66 (d, *J* = 0.6 Hz), 107.48 (s), 55.67 (s). ³¹P NMR (162 MHz, CDCl₃) δ 27.67 (s).



(8-phenoxyphenanthridin-6-yl)diphenylphosphine oxide (3da): white solid, m.p. 212.4-212.8 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.93 (d, *J* = 2.4 Hz, 1H), 8.60 (d, *J* = 9.1 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.14-8.00 (m, 1H), 7.97-7.82 (m, 4H), 7.68 (td, *J* = 14.7, 7.0 Hz, 2H), 7.61-7.48 (m, 4H), 7.47-7.33 (m, 6H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.02 (d, *J* = 7.7 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 156.77 (s), 156.23 (d, *J* = 128.9 Hz), 156.09 (s), 142.33 (d, *J* = 23.0 Hz), 133.03 (s), 132.27 (d, *J* = 9.2 Hz), 131.99 (s), 131.70 (d, *J* = 2.7 Hz), 131.27 (s), 130.04 (s), 129.05 (s), 128.76 (d, *J* = 22.9 Hz), 128.36 (d, *J* = 6.7 Hz), 128.20 (d, *J* = 12.2 Hz), 128.16 (s), 124.31 (d, *J* = 2.5 Hz), 124.16 (s), 123.34 (s), 121.82 (s), 119.67 (s), 115.08 (s). ³¹P NMR (162 MHz, CDCl₃) δ 27.90 (s). HRMS (ESI-TOF, [M+H]⁺): calcd for C₃₁H₂₃NO₂P, 472.1461, found 472.1456.

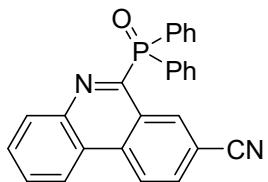


(8-fluorophenanthridin-6-yl)diphenylphosphine oxide (3ea): white solid, m.p. 229.7-230.3 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.33 (dd, *J* = 10.2, 2.6 Hz, 1H), 8.62 (dd, *J* = 8.2, 5.5 Hz, 1H), 8.55-8.46 (m, 1H), 8.13-8.03 (m, 1H), 8.01-7.90 (m, 4H), 7.77-7.65 (m, 2H), 7.61-7.39 (m, 7H). ¹³C NMR (101 MHz, CDCl₃) δ 161.26 (d, *J* = 249.1 Hz), 155.93 (dd, *J* = 128.4, 4.4 Hz), 142.42 (dd, *J* = 22.8, 1.0 Hz), 133.13 (s), 132.33 (d, *J* = 9.2 Hz), 132.09 (s), 131.88 (d, *J* = 2.8 Hz), 131.21 (s), 129.27 (s), 129.04 (dd, *J* = 23.1, 9.4 Hz), 128.64 (s), 128.28 (d, *J* = 12.2 Hz), 124.64 (dd, *J* = 8.7, 1.4 Hz), 123.95 (d, *J* = 1.4 Hz), 121.92 (s), 120.54 (d, *J* = 24.4 Hz), 113.19 (d, *J* = 23.2 Hz). ¹⁹F NMR (376 MHz, CDCl₃) δ -109.58 (s). ³¹P NMR (162 MHz, CDCl₃) δ 27.54 (s).

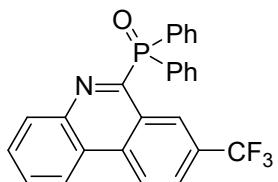


(8-chlorophenanthridin-6-yl)diphenylphosphine oxide (3fa): white solid, m.p. 234.5-235.2 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.66 (d, *J* = 2.1 Hz, 1H), 8.54 (ddd, *J* = 9.6, 7.4, 1.8 Hz, 2H), 8.07 (dd, *J* = 6.5, 3.0 Hz, 1H), 8.01-7.90 (m, 4H), 7.78 (dd, *J* = 8.9, 2.1 Hz, 1H), 7.73 (dd, *J* = 5.5, 4.1 Hz, 2H), 7.58-7.50 (m, 2H), 7.49-7.40 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 155.79 (d, *J* = 128.1 Hz), 142.63 (d, *J* = 22.8 Hz), 133.99 (s), 133.18 (s), 132.34 (d, *J* = 9.2 Hz), 132.14 (s), 131.87 (d, *J* = 2.8 Hz), 131.83 (s), 131.19 (d, *J* = 1.1 Hz), 130.98 (d, *J* = 6.7 Hz), 129.17 (d, *J* = 22.0 Hz), 128.75 (d, *J* = 23.0 Hz), 128.28 (d, *J* = 12.2 Hz), 127.67 (s), 123.80 (d, *J* = 1.5 Hz),

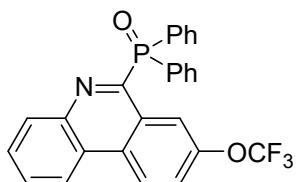
123.76 (d, $J = 2.6$ Hz), 122.02 (d, $J = 0.8$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 27.41 (s).



6-(diphenylphosphoryl)phenanthridine-8-carbonitrile (3ga)^[1a]: white solid, m.p. 261.5-262.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.08 (s, 1H), 8.71 (d, $J = 8.6$ Hz, 1H), 8.62-8.52 (m, 1H), 8.17-8.10 (m, 1H), 8.04-7.91 (m, 5H), 7.87-7.78 (m, 2H), 7.59-7.51 (m, 2H), 7.51-7.44 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.58 (d, $J = 126.6$ Hz), 143.50 (d, $J = 22.3$ Hz), 135.00 (d, $J = 6.6$ Hz), 133.98 (s), 132.71 (s), 132.37 (s), 132.30 (d, $J = 9.3$ Hz), 132.11 (d, $J = 2.8$ Hz), 131.66 (s), 131.39 (d, $J = 0.9$ Hz), 130.14 (d, $J = 82.3$ Hz), 128.41 (d, $J = 12.3$ Hz), 127.24 (d, $J = 23.0$ Hz), 123.45 (d, $J = 1.1$ Hz), 123.02 (d, $J = 2.3$ Hz), 122.65 (d, $J = 0.6$ Hz), 118.41 (s), 111.45 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 27.39 (s).

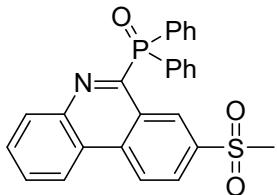


diphenyl(8-(trifluoromethyl)phenanthridin-6-yl)phosphine oxide (3ha)^[1a]: white solid, m.p. 179.3-180.3 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.03 (s, 1H), 8.77 (d, $J = 8.6$ Hz, 1H), 8.62 (dd, $J = 9.2, 3.6$ Hz, 1H), 8.14 (dd, $J = 5.4, 2.6$ Hz, 1H), 8.00 (ddd, $J = 12.4, 11.7, 5.1$ Hz, 5H), 7.81 (dd, $J = 6.3, 2.4$ Hz, 2H), 7.59-7.51 (m, 2H), 7.47 (td, $J = 7.4, 3.0$ Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.06 (d, $J = 127.3$ Hz), 143.31 (d, $J = 22.6$ Hz), 134.71 (d, $J = 6.7$ Hz), 133.03 (s), 132.33 (d, $J = 9.2$ Hz), 131.98 (s), 131.94 (d, $J = 2.8$ Hz), 131.29 (s), 129.69 (d, $J = 52.5$ Hz), 129.54 (q, $J = 33.2$ Hz), 128.32 (d, $J = 12.3$ Hz), 127.24 (d, $J = 23.0$ Hz), 126.98 (q, $J = 3.6$ Hz), 126.26 (q, $J = 4.1$ Hz), 125.21 (d, $J = 6.1$ Hz), 123.39 (d, $J = 2.3$ Hz), 123.24 (d, $J = 1.4$ Hz), 122.51 (s). ^{19}F NMR (376 MHz, CDCl_3) δ -62.21 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 26.95 (s).

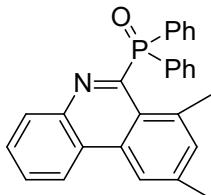


(9-diphenyl(8-(trifluoromethoxy)phenanthridin-6-yl)phosphine oxide (3ia): white solid, m.p. 169.7-170.4 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.53 (s, 1H), 8.72-8.62 (m, 1H), 8.58-8.47 (m, 1H), 8.14-8.07 (m, 1H), 8.02-7.91 (m, 4H), 7.72 (ddd, $J = 17.2, 10.0, 6.2$ Hz, 3H), 7.60-7.41 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.33 (d, $J = 128.2$ Hz), 147.88 (s), 142.75 (d, $J = 22.6$ Hz), 132.99 (s), 132.32 (d, $J = 9.2$ Hz), 131.94 (s), 131.91 (d, $J = 2.7$ Hz), 131.25 (s), 131.02 (d, $J = 6.6$ Hz), 129.28 (d, $J = 20.3$ Hz), 128.59 (s), 128.30 (d, $J = 12.2$ Hz), 124.39 (s), 123.61 (s), 122.09 (s),

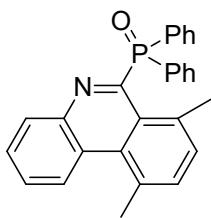
120.51 (d, $J = 258.5$ Hz), 119.61 (s). ^{19}F NMR (376 MHz, CDCl_3) δ -57.62 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 27.16 (d, $J = 1.3$ Hz). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{26}\text{H}_{18}\text{F}_3\text{NO}_2\text{P}$, 464.1022, found 464.1011.



(10-(methylsulfonyl)phenanthridin-6-yl)diphenylphosphine oxide (3ja): white solid, m.p. 238.7-239.2 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.25 (d, $J = 1.4$ Hz, 1H), 8.79 (d, $J = 8.8$ Hz, 1H), 8.60 (d, $J = 7.9$ Hz, 1H), 8.33 (d, $J = 8.7$ Hz, 1H), 8.13 (d, $J = 7.6$ Hz, 1H), 8.05-7.92 (m, 4H), 7.87-7.75 (m, 2H), 7.49 (ddd, $J = 11.5, 10.6, 5.7$ Hz, 6H), 3.18 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.26 (d, $J = 126.9$ Hz), 143.57 (d, $J = 22.3$ Hz), 139.52 (s), 135.77 (d, $J = 6.5$ Hz), 132.30 (d, $J = 9.3$ Hz), 132.17 (d, $J = 105.4$ Hz), 132.10 (d, $J = 2.6$ Hz), 131.35 (s), 130.57 (s), 129.73 (s), 128.93 (s), 128.41 (d, $J = 12.3$ Hz), 127.80 (s), 127.14 (d, $J = 22.9$ Hz), 123.87 (s), 123.09 (d, $J = 2.2$ Hz), 122.79 (s), 44.17 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 26.99 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{26}\text{H}_{21}\text{NO}_3\text{PS}$, 458.0974, found 458.0978.

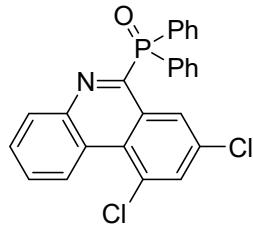


(11-(7,9-dimethylphenanthridin-6-yl)diphenylphosphine oxide (3ka): white solid, m.p. 269.2-270.4 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.55 (d, $J = 8.2$ Hz, 1H), 8.34 (s, 1H), 7.84-7.76 (m, 4H), 7.73 (dd, $J = 7.9, 1.3$ Hz, 1H), 7.63 (dt, $J = 14.9, 7.0$ Hz, 2H), 7.50 (td, $J = 7.2, 1.3$ Hz, 2H), 7.43 (ddd, $J = 7.1, 5.3, 2.3$ Hz, 4H), 7.32 (s, 1H), 2.95 (s, 3H), 2.56 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.56 (d, $J = 129.5$ Hz), 141.21 (s), 140.77 (s), 137.55 (s), 135.39 (s), 134.45 (d, $J = 6.6$ Hz), 134.33 (s), 133.42 (s), 132.01 (d, $J = 9.0$ Hz), 131.17 (d, $J = 2.7$ Hz), 130.51 (d, $J = 1.3$ Hz), 128.54 (d, $J = 30.5$ Hz), 128.03 (d, $J = 12.3$ Hz), 125.59 (d, $J = 23.7$ Hz), 124.25 (d, $J = 2.6$ Hz), 122.24 (s), 120.02 (d, $J = 1.9$ Hz), 24.93 (s), 21.93 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 36.25 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{27}\text{H}_{23}\text{NOP}$, 408.1512, found 408.1518.

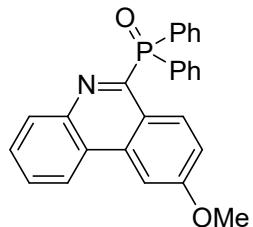


(12-(7,10-dimethylphenanthridin-6-yl)diphenylphosphine oxide (3la): white solid, m.p. 199.9-200.4 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.66 (dd, $J = 5.7, 3.8$ Hz, 1H), 7.86 (dd, $J = 11.0, 7.8$ Hz, 5H), 7.61 (dd, $J = 6.0, 3.4$ Hz, 2H), 7.49 (ddd, $J = 17.1, 12.3, 6.3$ Hz, 7H), 7.37 (d, $J = 7.4$ Hz, 1H), 3.03 (s, 3H), 2.91 (s, 3H). ^{13}C NMR (101

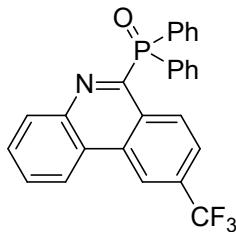
MHz, CDCl₃) δ 157.23 (d, *J* = 129.0 Hz), 142.18 (d, *J* = 24.5 Hz), 135.49 (d, *J* = 35.0 Hz), 134.44 (s), 134.25 (s), 133.81 (d, *J* = 6.4 Hz), 132.26 (d, *J* = 1.8 Hz), 132.05 (d, *J* = 8.9 Hz), 131.23 (d, *J* = 2.7 Hz), 130.41 (s), 130.10 (d, *J* = 1.0 Hz), 128.05 (d, *J* = 12.2 Hz), 127.84 (s), 127.82 (d, *J* = 22.8 Hz), 127.61 (s), 126.35 (s), 125.18 (d, *J* = 2.7 Hz), 26.08 (s), 24.60 (s). ³¹P NMR (162 MHz, CDCl₃) δ 33.87 (s). HRMS (ESI-TOF, [M+H]⁺): calcd for C₂₇H₂₃NOP, 408.1512, found 408.1517.



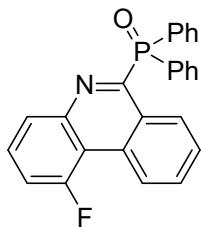
(13-(8,10-dichlorophenanthridin-6-yl)diphenylphosphine oxide (3ma): white solid, m.p. 205.1-205.9 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.77 (d, *J* = 2.1 Hz, 1H), 9.73 (ddd, *J* = 6.6, 4.0, 1.7 Hz, 1H), 8.08 (dd, *J* = 10.2, 8.2 Hz, 1H), 7.92 (ddd, *J* = 13.1, 6.7, 5.3 Hz, 5H), 7.80-7.70 (m, 2H), 7.57-7.51 (m, 2H), 7.47 (td, *J* = 7.7, 3.0 Hz, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 155.68 (d, *J* = 127.7 Hz), 143.59 (d, *J* = 22.7 Hz), 134.77 (s), 132.98 (d, *J* = 15.9 Hz), 132.32 (d, *J* = 9.3 Hz), 132.09 (d, *J* = 3.2 Hz), 132.00 (d, *J* = 2.8 Hz), 131.85 (s), 131.41 (s), 130.58 (d, *J* = 23.8 Hz), 129.38 (s), 128.86 (s), 128.33 (d, *J* = 12.3 Hz), 128.14 (d, *J* = 6.6 Hz), 127.17 (s), 126.14 (s), 122.91 (d, *J* = 2.5 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 28.34 (s). HRMS (ESI-TOF, [M+H]⁺): calcd for C₂₅H₁₇Cl₂NOP, 448.0419, found 448.0415.



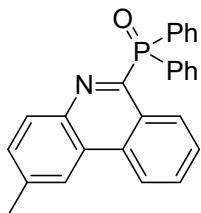
(14-(9-methoxyphenanthridin-6-yl)diphenylphosphine oxide (3na): white solid, m.p. 162.1-163.0 °C. ¹H NMR (500 MHz, CDCl₃) δ 9.47 (d, *J* = 9.2 Hz, 1H), 8.46 (dd, *J* = 6.9, 2.7 Hz, 1H), 8.07-7.98 (m, 1H), 7.98-7.87 (m, 5H), 7.65 (dq, *J* = 6.2, 3.8, 2.5 Hz, 2H), 7.53-7.45 (m, 2H), 7.43 (td, *J* = 7.6, 2.9 Hz, 4H), 7.27 (dd, *J* = 9.2, 2.5 Hz, 1H), 3.97 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 161.47, 156.00 (d, *J* = 128.8 Hz), 143.05 (d, *J* = 23.7 Hz), 135.06 (d, *J* = 7.3 Hz), 133.49, 132.66, 132.35 (d, *J* = 9.1 Hz), 131.67 (d, *J* = 2.8 Hz), 131.09, 130.49, 128.80, 128.17 (d, *J* = 12.0 Hz), 124.09 (d, *J* = 2.6 Hz), 123.18 (d, *J* = 23.6 Hz), 122.15, 117.90, 102.99, 55.54. ³¹P NMR (202 MHz, CDCl₃) δ 28.03. HRMS (ESI-TOF, [M+H]⁺): calcd for C₂₆H₂₁NO₂P, 410.1304, found 410.1312.



diphenyl(9-(trifluoromethyl)phenanthridin-6-yl)phosphine oxide (3oa)^[1b]: white solid, m.p. 166.3-167.2 °C. ¹H NMR (500 MHz, CDCl₃) δ 9.75 (d, *J* = 8.6 Hz, 1H), 8.91 (s, 1H), 8.69-8.47 (m, 1H), 8.16-8.06 (m, 1H), 8.00-7.93 (m, 4H), 7.89 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.82-7.71 (m, 2H), 7.54 (ddd, *J* = 7.4, 5.2, 1.5 Hz, 2H), 7.47 (td, *J* = 7.6, 3.2 Hz, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 156.79 (d, *J* = 127.6 Hz), 143.02 (d, *J* = 22.7 Hz), 132.92, 132.46, 132.41, 132.31 (d, *J* = 9.3 Hz), 132.20, 132.09, 131.95 (d, *J* = 2.7 Hz), 131.37, 129.82, 129.61 (d, *J* = 10.6 Hz), 129.12 (d, *J* = 23.1 Hz), 128.33 (d, *J* = 12.0 Hz), 123.87 (q, *J* = 273.1 Hz), 123.78 (dd, *J* = 4.7, 2.8 Hz), 122.11, 119.69 (d, *J* = 4.5 Hz). ¹⁹F NMR (471 MHz, CDCl₃) δ -62.65. ³¹P NMR (202 MHz, CDCl₃) δ 27.87.

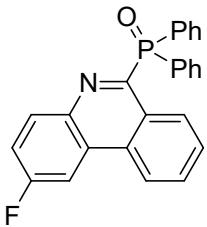


(15-(1-fluorophenanthridin-6-yl)diphenylphosphine oxide (3pa)): white solid, m.p. 196.5-197.6 °C. ¹H NMR (500 MHz, CDCl₃) δ 9.56 (d, *J* = 8.2 Hz, 1H), 9.06 (d, *J* = 8.4 Hz, 1H), 8.00 - 7.84 (m, 6H), 7.73 (t, *J* = 7.7 Hz, 1H), 7.63 (dtq, *J* = 7.9, 5.5, 2.8, 2.2 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.46 (td, *J* = 7.6, 3.0 Hz, 5H). ¹³C NMR (126 MHz, CDCl₃) δ 161.17, 158.94 (d, *J* = 49.4 Hz), 157.74, 144.59, 144.41 (d, *J* = 2.7 Hz), 133.11, 132.30 (d, *J* = 9.2 Hz), 131.81 (d, *J* = 2.8 Hz), 131.64 (d, *J* = 2.1 Hz), 130.81-130.50 (m), 128.50, 128.24 (d, *J* = 12.1 Hz), 128.10, 127.44-127.13 (m), 127.01, 115.13 (d, *J* = 24.1 Hz), 114.23 (d, *J* = 9.1 Hz). ¹⁹F NMR (471 MHz, CDCl₃) δ -110.07. ³¹P NMR (202 MHz, CDCl₃) δ 28.52 (d, *J* = 8.7 Hz). HRMS (ESI-TOF, [M+H]⁺): calcd for C₂₅H₁₈FNOP, 398.1105, found 398.1112.

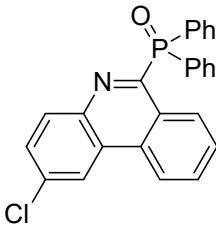


(2-methylphenanthridin-6-yl)diphenylphosphine oxide (3qa)^[1a]: white solid, m.p. 214.0-214.5 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.45 (d, *J* = 8.3 Hz, 1H), 8.65 (d, *J* = 8.3 Hz, 1H), 8.38 (s, 1H), 7.97 – 7.88 (m, 5H), 7.83 (t, *J* = 7.7 Hz, 1H), 7.67 (t, *J* = 7.3 Hz, 1H), 7.57-7.48 (m, 3H), 7.44 (ddd, *J* = 7.1, 5.4, 2.3 Hz, 4H), 2.64 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 154.53 (d, *J* = 328.7 Hz), 141.18 (d, *J* = 23.3 Hz), 139.12 (s), 133.49 (s), 132.45 (s), 132.37 (s), 132.28 (s), 131.67 (d, *J* = 2.8 Hz), 130.93 (d, *J*

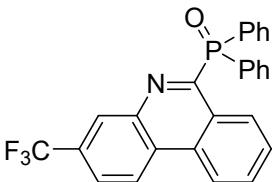
= 1.0 Hz), 130.80 (s), 130.48 (s), 128.47 (s), 128.18 (d, J = 12.2 Hz), 127.78 (s), 124.23 (d, J = 2.4 Hz), 122.11 (d, J = 1.6 Hz), 121.67 (d, J = 1.0 Hz), 22.24 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 28.50 (s).



(2-fluorophenanthridin-6-yl)diphenylphosphine oxide (3ra)^[1a]: white solid, m.p. 235.9-236.6 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.50 (d, J = 8.3 Hz, 1H), 8.49 (d, J = 7.9 Hz, 1H), 8.16 (dd, J = 10.0, 2.7 Hz, 1H), 8.03 (dd, J = 8.9, 5.8 Hz, 1H), 7.98-7.88 (m, 4H), 7.87-7.79 (m, 1H), 7.70 (dd, J = 8.2, 7.2 Hz, 1H), 7.56-7.38 (m, 7H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.43 (d, J = 250.0 Hz), 156.12 (d, J = 128.5 Hz), 139.64 (d, J = 23.5 Hz), 133.58 (d, J = 9.5 Hz), 133.27 (s), 132.29 (d, J = 9.3 Hz), 132.22 (s), 132.04 (dd, J = 6.8, 4.4 Hz), 131.81 (d, J = 2.7 Hz), 131.12 (s), 128.63 (d, J = 8.9 Hz), 128.26 (d, J = 12.2 Hz), 127.74 (d, J = 23.1 Hz), 125.98 (dd, J = 9.5, 2.3 Hz), 122.33 (s), 117.85 (d, J = 24.5 Hz), 107.11 (d, J = 24.2 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -109.33 (d, J = 2.3 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 28.46 (s).

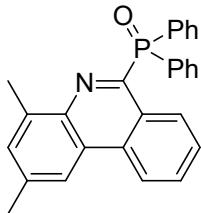


(2-chlorophenanthridin-6-yl)diphenylphosphine oxide (3sa)^[1a]: white solid, m.p. 266.1-266.7 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.50 (d, J = 8.2 Hz, 1H), 8.58-8.46 (m, 2H), 8.01-7.88 (m, 5H), 7.84 (t, J = 7.6 Hz, 1H), 7.70 (t, J = 7.5 Hz, 1H), 7.63 (dd, J = 8.7, 2.1 Hz, 1H), 7.56-7.49 (m, 2H), 7.45 (td, J = 7.3, 2.8 Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.27 (d, J = 127.6 Hz), 141.08 (d, J = 23.4 Hz), 134.92 (s), 133.17 (s), 132.60 (s), 132.30 (d, J = 9.3 Hz), 132.12 (s), 131.85 (d, J = 2.6 Hz), 131.56 (d, J = 6.7 Hz), 131.37 (s), 129.34 (s), 128.65 (d, J = 10.5 Hz), 128.28 (d, J = 12.2 Hz), 127.93 (d, J = 22.9 Hz), 125.43 (d, J = 2.2 Hz), 122.18 (s), 121.81 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 28.41 (s).

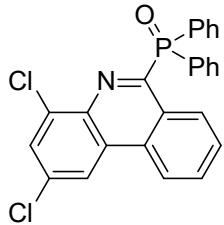


(16-diphenyl(3-(trifluoromethyl)phenanthridin-6-yl)phenanthridin-6-yl)phosphine oxide (3ta): white solid, m.p. 160.1-160.7 °C. ^1H NMR (500 MHz, CDCl_3) δ 9.59 (d, J = 8.3 Hz, 1H), 8.77-8.54 (m, 2H), 8.31 (s, 1H), 8.03-7.83 (m, 6H), 7.76 (dt, J = 8.1, 4.4 Hz, 1H), 7.55 (t, J = 7.4 Hz, 2H), 7.48 (td, J = 7.6, 2.9 Hz, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.02 (d, J = 125.9 Hz), 141.91 (d, J = 23.3 Hz), 132.86, 132.28 (d, J = 9.3 Hz),

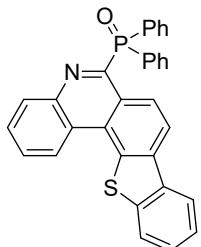
132.03, 131.97 (d, $J = 2.9$ Hz), 131.82 (d, $J = 6.8$ Hz), 131.69, 130.61 (q, $J = 32.9$ Hz), 129.01 (d, $J = 17.8$ Hz), 128.52, 128.47, 128.35 (d, $J = 12.2$ Hz), 126.63 (d, $J = 2.2$ Hz), 124.52 (q, $J = 3.3$ Hz), 123.91 (q, $J = 272.5$ Hz), 123.32, 122.51 (d, $J = 1.6$ Hz). ^{19}F NMR (471 MHz, CDCl_3) δ -62.20. ^{31}P NMR (202 MHz, CDCl_3) δ 28.35 (d, $J = 9.9$ Hz). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{26}\text{H}_{18}\text{F}_3\text{NOP}$, 448.1073, found 448.1076.



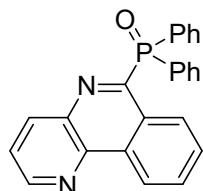
(2,4-dimethylphenanthridin-6-yl)diphenylphosphine oxide (3ua): white solid, m.p. 202.4-202.6 °C. ^1H NMR (500 MHz, CDCl_3) δ 9.39 (dd, $J = 8.6, 3.7$ Hz, 1H), 8.63 (t, $J = 7.6$ Hz, 1H), 8.22 (d, $J = 5.4$ Hz, 1H), 7.94-7.84 (m, 4H), 7.84-7.74 (m, 1H), 7.70-7.60 (m, 1H), 7.58-7.48 (m, 2H), 7.48-7.36 (m, 5H), 2.57 (t, $J = 3.5$ Hz, 3H), 2.45 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 154.00 (d, $J = 131.4$ Hz), 140.02 (d, $J = 22.6$ Hz), 138.79 (d, $J = 7.2$ Hz), 133.60, 132.77, 132.63 (d, $J = 6.9$ Hz), 132.32 (d, $J = 9.3$ Hz), 131.58 (d, $J = 2.8$ Hz), 131.32, 130.61, 128.34, 128.11 (d, $J = 12.0$ Hz), 127.64 (d, $J = 24.0$ Hz), 127.58, 124.25 (d, $J = 2.5$ Hz), 122.37 (d, $J = 1.8$ Hz), 119.43, 22.17, 18.00. ^{31}P NMR (202 MHz, CDCl_3) δ 29.68 (d, $J = 8.1$ Hz). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{27}\text{H}_{23}\text{NOP}$, 408.1512, found 408.1507.



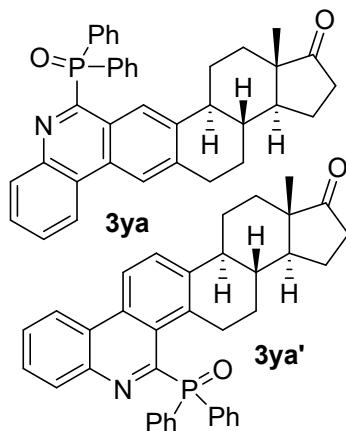
(2,4-dichlorophenanthridin-6-yl)diphenylphosphine oxide (3va)^[1b]: white solid, m.p. 275.0-275.4 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.67 (d, $J = 8.2$ Hz, 1H), 8.41 (d, $J = 8.3$ Hz, 1H), 8.36 (d, $J = 1.9$ Hz, 1H), 8.18-8.05 (m, 4H), 7.82 (t, $J = 7.6$ Hz, 1H), 7.74 (dd, $J = 13.7, 4.7$ Hz, 2H), 7.58-7.39 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.08 (d, $J = 126.2$ Hz), 137.67 (d, $J = 22.7$ Hz), 136.81 (s), 134.30 (s), 133.04 (s), 132.49 (d, $J = 9.1$ Hz), 131.99 (s), 131.88 (d, $J = 2.6$ Hz), 131.80 (s), 131.27 (d, $J = 6.6$ Hz), 129.31 (d, $J = 25.7$ Hz), 128.82 (s), 128.29 (d, $J = 12.2$ Hz), 128.17 (d, $J = 22.7$ Hz), 126.53 (d, $J = 2.4$ Hz), 122.34 (s), 120.67 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 25.46 (s).



benzo[4,5]thieno[3,2-k]phenanthridin-6-yldiphenylphosphine oxide (3wa): white solid, m.p. >325 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.72 (d, $J = 8.7$ Hz, 1H), 9.12 (d, $J = 8.3$ Hz, 1H), 8.45 (d, $J = 8.7$ Hz, 1H), 8.28 (d, $J = 6.9$ Hz, 1H), 8.14 (d, $J = 7.9$ Hz, 1H), 7.98 (dd, $J = 10.8, 8.0$ Hz, 5H), 7.88 (t, $J = 7.6$ Hz, 1H), 7.78 (t, $J = 7.4$ Hz, 1H), 7.60-7.40 (m, 8H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.85 (d, $J = 128.5$ Hz), 143.63 (d, $J = 23.6$ Hz), 140.22 (s), 137.83 (s), 134.33 (s), 133.84 (d, $J = 2.6$ Hz), 133.46 (s), 132.42 (s), 132.40 (d, $J = 9.2$ Hz), 131.79 (d, $J = 2.6$ Hz), 131.61 (s), 129.35 (d, $J = 7.1$ Hz), 129.06 (s), 128.59 (s), 128.25 (d, $J = 12.2$ Hz), 127.76 (s), 127.76 (d, $J = 23.8$ Hz), 125.46 (s), 125.14 (s), 125.03 (s), 123.91 (d, $J = 2.1$ Hz), 122.36 (d, $J = 12.7$ Hz), 121.51 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 29.39 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{27}\text{H}_{23}\text{NOP}$, 486.1076, found 486.1072.

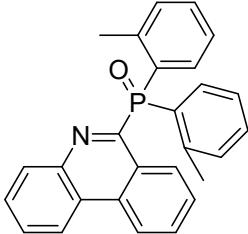


benzo[c][1,5]naphthyridin-6-yldiphenylphosphine oxide (3xa): white solid, m.p. 191.3-191.5 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.48 (d, $J = 8.3$ Hz, 1H), 9.26 (d, $J = 8.2$ Hz, 1H), 9.03 (d, $J = 4.4$ Hz, 1H), 8.41-8.27 (m, 1H), 8.03-7.87 (m, 5H), 7.86-7.76 (m, 1H), 7.64 (td, $J = 8.2, 4.3$ Hz, 1H), 7.59-7.40 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.03 (d, $J = 5.8$ Hz), 157.75 (s), 151.20 (s), 141.04 (s), 138.18 (s), 137.54 (d, $J = 23.3$ Hz), 133.52 (d, $J = 7.4$ Hz), 133.01 (s), 132.30 (d, $J = 9.3$ Hz), 132.12-131.82 (m), 131.61 (s), 129.95 (d, $J = 23.3$ Hz), 129.56 (s), 128.30 (d, $J = 12.3$ Hz), 127.96 (s), 123.83 (d, $J = 27.9$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 28.48 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{24}\text{H}_{18}\text{N}_2\text{OP}$, 381.1151, found 381.1156.



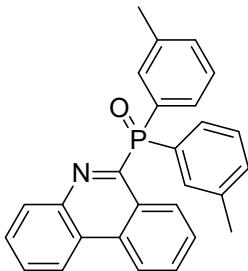
Derived from estone (3ya + 3ya'): white solid, m.p. 171.9-178.6. ^1H NMR (500 MHz, CDCl_3) δ 9.55-8.35 (m, 2H), 8.29-7.92 (m, 4H), 7.81-7.31 (m, 10H), 4.67 -2.87 (m, 2H), 2.68-1.32 (m, 13H), 0.85 (d, $J = 19.4$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 220.59, 157.07, 156.05, 142.70, 142.51, 141.02, 140.93, 140.86, 140.74, 140.65, 136.61, 136.39, 135.48, 134.53, 133.78, 133.73, 133.59, 132.95, 132.76, 132.40, 132.36, 132.32, 132.29, 132.25, 132.09, 132.04, 131.83, 131.76, 131.66, 131.64, 131.59, 131.56, 130.98, 130.87, 130.85, 130.63, 130.58, 130.04, 128.87, 128.85, 128.52, 128.49, 128.39, 128.35, 128.29, 128.21, 128.12, 127.71, 127.61, 127.40,

127.22, 126.63, 126.45, 124.86, 123.99, 123.97, 123.94, 122.04, 121.98, 121.52, 119.86, 50.60, 48.12, 47.93, 45.32, 44.61, 37.78, 37.49, 35.87, 35.84, 31.82, 31.45, 30.30, 30.00, 29.71, 26.32, 26.17, 25.70, 25.51, 21.58, 21.49, 13.99, 13.81. ^{31}P NMR (202 MHz, CDCl_3) δ 34.31 (d, $J = 6.6$ Hz), 26.61 (d, $J = 8.2$ Hz). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{37}\text{H}_{35}\text{NO}_2\text{P}$, 556.2400, found 556.1394.



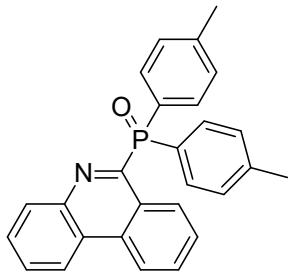
phenanthridin-6-yl-di-o-tolyphosphine oxide (3ab): white solid, m.p. 244.2-244.9 °C.

^1H NMR (400 MHz, CDCl_3) δ 9.23 (d, $J = 8.3$ Hz, 1H), 8.69 (d, $J = 8.3$ Hz, 1H), 8.62 (d, $J = 8.0$ Hz, 1H), 7.93 (d, $J = 7.9$ Hz, 1H), 7.85 (t, $J = 7.7$ Hz, 1H), 7.78-7.61 (m, 3H), 7.47-7.35 (m, 4H), 7.31 (dd, $J = 7.3, 4.2$ Hz, 2H), 7.16 (t, $J = 7.2$ Hz, 2H), 2.46 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.32 (d, $J = 128.3$ Hz), 143.39 (d, $J = 7.9$ Hz), 142.90 (d, $J = 23.4$ Hz), 133.09 (d, $J = 12.1$ Hz), 132.69 (d, $J = 6.6$ Hz), 131.85 (d, $J = 2.5$ Hz), 131.62 (d, $J = 10.7$ Hz), 131.35 (s), 130.98 (s), 130.33 (s), 128.87 (s), 128.84 (s), 128.68 (s), 127.84 (s), 127.54 (d, $J = 22.9$ Hz), 125.37 (d, $J = 12.8$ Hz), 124.43 (d, $J = 2.4$ Hz), 122.26 (d, $J = 1.2$ Hz), 122.07 (s), 22.06 (d, $J = 3.6$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 38.27 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{27}\text{H}_{23}\text{NOP}$, 408.1512, found 408.1508.

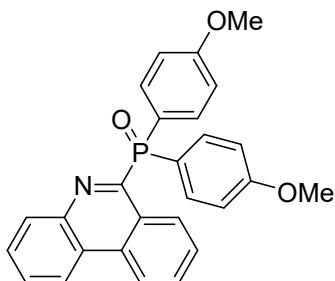


phenanthridin-6-yl-di-m-tolyphosphine oxide (3ac): white solid, m.p. 246.0-246.9 °C.

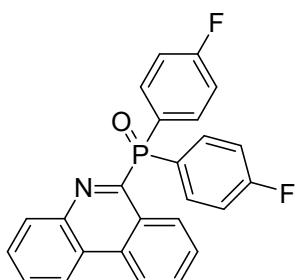
^1H NMR (400 MHz, CDCl_3) δ 9.51 (d, $J = 8.2$ Hz, 1H), 8.70-8.55 (m, 2H), 8.13-8.05 (m, 1H), 7.89-7.79 (m, 3H), 7.70 (tdd, $J = 10.1, 5.8, 4.3$ Hz, 5H), 7.40-7.29 (m, 4H), 2.37 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.05 (d, $J = 127.6$ Hz), 142.76 (d, $J = 23.1$ Hz), 138.04 (d, $J = 12.1$ Hz), 133.28 (s), 132.67 (s), 132.61 (s), 132.56 (d, $J = 3.1$ Hz), 132.52 (s), 132.24 (s), 131.21 (d, $J = 1.3$ Hz), 131.00 (s), 129.53 (d, $J = 9.4$ Hz), 128.71 (d, $J = 10.1$ Hz), 128.01 (d, $J = 12.9$ Hz), 127.98 (s), 127.88 (s), 127.75 (s), 124.35 (d, $J = 2.4$ Hz), 122.12 (s), 21.53 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 28.66 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{27}\text{H}_{23}\text{NOP}$, 408.1512, found 408.1519.



phenanthridin-6-yl-di-p-tolylphosphine oxide (3ad)^[1a]: white solid, m.p. 202.4-203.3 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.50 (d, *J* = 8.3 Hz, 1H), 8.76-8.51 (m, 2H), 8.11-8.04 (m, 1H), 7.87-7.77 (m, 5H), 7.75-7.65 (m, 3H), 7.24 (s, 4H), 2.38 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 157.27 (d, *J* = 127.8 Hz), 142.77 (d, *J* = 23.3 Hz), 142.08 (d, *J* = 2.8 Hz), 132.56 (d, *J* = 6.7 Hz), 132.32 (d, *J* = 9.6 Hz), 131.21 (d, *J* = 1.1 Hz), 130.98 (s), 130.27 (s), 129.20 (s), 128.97 (d, *J* = 12.6 Hz), 128.70 (d, *J* = 3.9 Hz), 128.64 (s), 127.93 (s), 127.87 (s), 127.70 (s), 124.33 (d, *J* = 2.5 Hz), 122.10 (s), 21.67 (s). ³¹P NMR (162 MHz, CDCl₃) δ 28.96 (s).

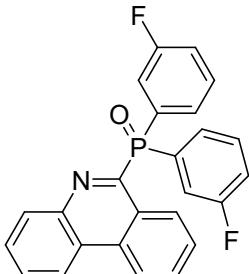


bis(4-methoxyphenyl)(phenanthridin-6-yl)phosphine oxide (3ae)^[1a]: colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 9.49 (d, *J* = 8.3 Hz, 1H), 8.68-8.50 (m, 2H), 8.15-7.99 (m, 1H), 7.84 (dd, *J* = 11.0, 8.7 Hz, 5H), 7.73-7.61 (m, 3H), 6.95 (dd, *J* = 8.7, 1.9 Hz, 4H), 3.80 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 162.31 (d, *J* = 2.9 Hz), 157.47 (d, *J* = 129.0 Hz), 142.76 (d, *J* = 23.2 Hz), 134.14 (d, *J* = 10.6 Hz), 132.57 (d, *J* = 6.8 Hz), 131.07 (d, *J* = 17.6 Hz), 130.40 (s), 128.72 (s), 128.67 (d, *J* = 2.0 Hz), 127.85 (s), 127.33 (d, *J* = 59.4 Hz), 124.81 (s), 124.31 (d, *J* = 2.4 Hz), 123.70 (s), 122.10 (s), 113.79 (d, *J* = 13.2 Hz), 113.66 (d, *J* = 4.6 Hz), 55.30 (s). ³¹P NMR (162 MHz, CDCl₃) δ 28.85 (s).

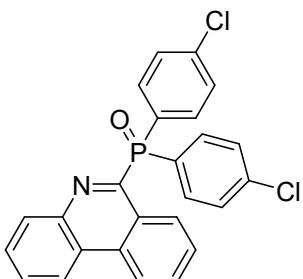


bis(4-fluorophenyl)(phenanthridin-6-yl)phosphine oxide (3af)^[1a]: white solid, m.p. 144.2-145.1 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.47 (d, *J* = 8.3 Hz, 1H), 8.75-8.56 (m, 2H), 8.12-8.03 (m, 1H), 7.99-7.84 (m, 5H), 7.79-7.65 (m, 3H), 7.15 (t, *J* = 7.8 Hz, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 165.09 (dd, *J* = 253.1, 3.3 Hz), 156.33 (d, *J* = 130.9 Hz), 142.65 (d, *J* = 23.6 Hz), 134.78 (d, *J* = 1.7 Hz), 134.78 (d, *J* = 19.4 Hz), 132.66 (d, *J* = 7.1 Hz), 131.28 (s), 131.06 (d, *J* = 1.0 Hz), 129.16 (d, *J* = 3.4 Hz),

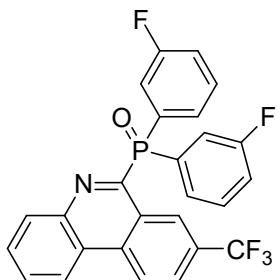
129.08 (s), 128.90 (s), 128.36 (s), 128.06 (s), 127.71 (d, J = 23.5 Hz), 124.39 (d, J = 2.4 Hz), 122.55-121.65 (m), 115.69 (dd, J = 21.4, 13.4 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -106.92 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 26.69 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{25}\text{H}_{17}\text{F}_2\text{NOP}$, 416.1010, found 416.1012.



bis(3-fluorophenyl)(phenanthridin-6-yl)phosphine oxide (3ag): white solid, m.p. 159.8-160.9 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.48 (d, J = 8.3 Hz, 1H), 8.68 (d, J = 8.3 Hz, 1H), 8.62 (dd, J = 8.8, 3.2 Hz, 1H), 8.15-8.06 (m, 1H), 7.93-7.84 (m, 1H), 7.80-7.62 (m, 7H), 7.50-7.40 (m, 2H), 7.23 (td, J = 8.4, 1.9 Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.61 (d, J = 17.2 Hz), 161.13 (d, J = 17.2 Hz), 155.67 (d, J = 131.5 Hz), 142.65 (d, J = 23.7 Hz), 135.64 (d, J = 6.2 Hz), 134.60 (d, J = 6.0 Hz), 132.71 (d, J = 7.1 Hz), 131.35 (s), 131.11 (s), 130.25 (dd, J = 14.2, 7.4 Hz), 129.08 (d, J = 22.9 Hz), 128.17 (d, J = 10.9 Hz), 128.00 (dd, J = 8.7, 3.2 Hz), 127.76 (d, J = 23.9 Hz), 124.44 (d, J = 2.4 Hz), 122.25 (dd, J = 8.0, 1.2 Hz), 119.27 (d, J = 1.8 Hz), 119.25 (d, J = 22.7 Hz), 119.06 (d, J = 3.0 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -111.39 (d, J = 6.0 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 24.92 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{25}\text{H}_{17}\text{F}_2\text{NOP}$, 416.1010, found 416.1014.



bis(4-chlorophenyl)(phenanthridin-6-yl)phosphine oxide (3ah)^[1a]: white solid, m.p. 229.3-230.1 °C. ^1H NMR (400 MHz, CDCl_3) δ 9.45 (d, J = 8.3 Hz, 1H), 8.67 (d, J = 8.3 Hz, 1H), 8.61 (dd, J = 6.1, 3.4 Hz, 1H), 8.07 (dd, J = 5.8, 3.7 Hz, 1H), 7.87 (dd, J = 11.3, 8.4 Hz, 5H), 7.79-7.67 (m, 3H), 7.44 (dd, J = 8.5, 2.3 Hz, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.93 (d, J = 130.8 Hz), 142.65 (d, J = 23.6 Hz), 138.60 (d, J = 3.4 Hz), 133.68 (d, J = 10.0 Hz), 132.68 (d, J = 7.1 Hz), 131.62 (s), 131.34 (s), 131.07 (d, J = 1.1 Hz), 130.56 (s), 129.05 (d, J = 21.5 Hz), 128.70 (d, J = 12.8 Hz), 128.18 (d, J = 15.7 Hz), 127.81 (s), 127.58 (s), 124.42 (d, J = 2.5 Hz), 122.30 (d, J = 1.4 Hz), 122.22 (s). ^{31}P NMR (162 MHz, CDCl_3) δ 26.48 (s).



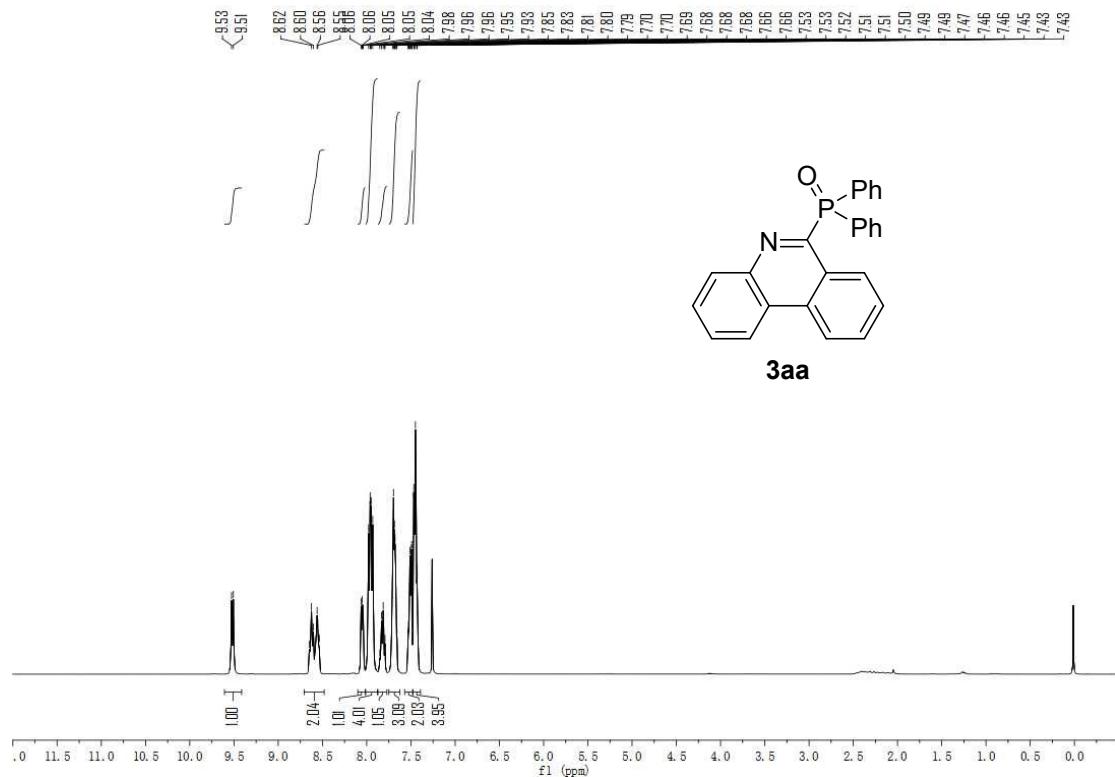
bis(3-fluorophenyl)(8-(trifluoromethyl)phenanthridin-6-yl)phosphine oxide (3jg): white solid, m.p. 173.8-174.3 °C. ^1H NMR (400 MHz, CDCl_3) δ 10.02 (s, 1H), 8.76 (d, J = 8.7 Hz, 1H), 8.66-8.57 (m, 1H), 8.22-8.14 (m, 1H), 8.05 (dd, J = 8.7, 1.5 Hz, 1H), 7.82 (qd, J = 6.7, 4.8 Hz, 4H), 7.76-7.67 (m, 2H), 7.52-7.41 (m, 2H), 7.23 (dd, J = 8.4, 2.5 Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.39 (dd, J = 249.8, 17.3 Hz), 155.83 (d, J = 130.2 Hz), 143.19 (d, J = 23.0 Hz), 135.31 (d, J = 5.8 Hz), 134.78 (d, J = 7.7 Hz), 134.27 (d, J = 5.8 Hz), 131.18 (d, J = 1.1 Hz), 130.41 (dd, J = 14.3, 7.4 Hz), 130.02 (d, J = 40.7 Hz), 129.98-129.16 (m), 128.02 (dd, J = 8.6, 3.2 Hz), 127.22 (q, J = 3.2 Hz), 127.17 (d, J = 23.6 Hz), 125.90 (q, J = 4.3 Hz), 123.78 (dd, J = 545.5, 272.8 Hz), 123.44 (d, J = 2.5 Hz), 122.98 (dd, J = 82.1, 1.0 Hz), 119.51 (d, J = 2.6 Hz), 119.30 (dd, J = 6.3, 3.7 Hz), 119.07 (d, J = 10.0 Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -62.22 (s), -111.09 (d, J = 6.4 Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 23.45 (s). HRMS (ESI-TOF, $[\text{M}+\text{H}]^+$): calcd for $\text{C}_{26}\text{H}_{16}\text{F}_5\text{NOP}$, 484.0884, found 484.0889.

10. References

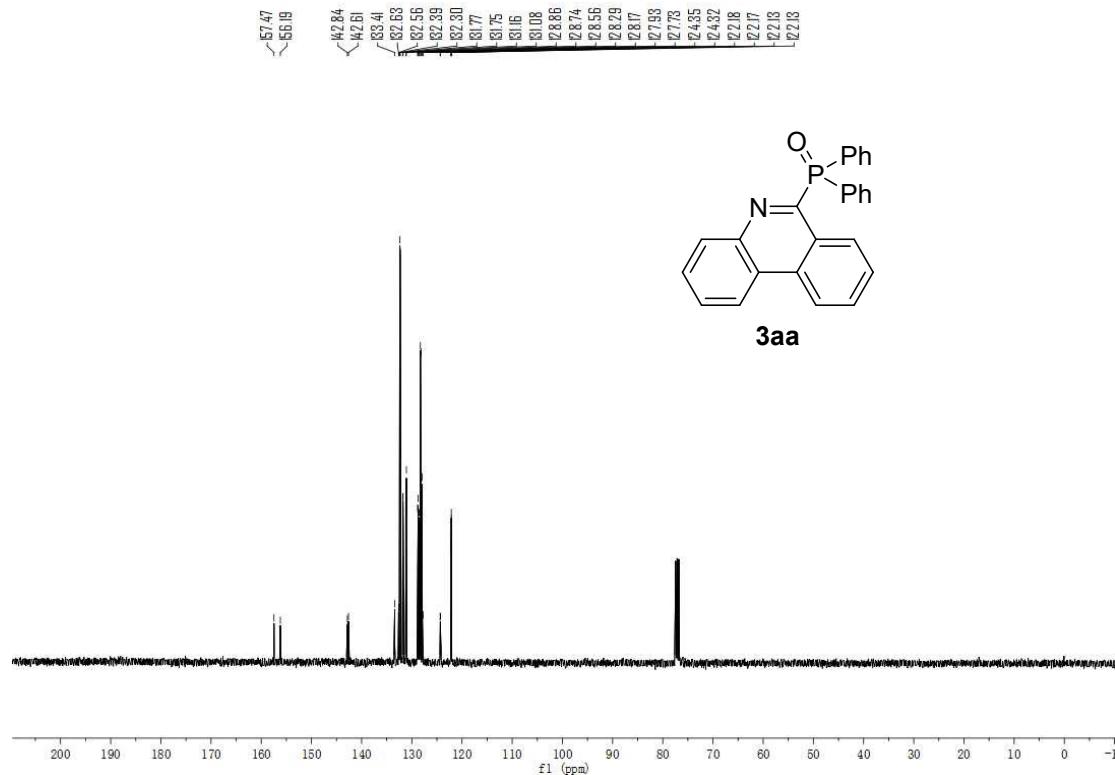
- [1] (a) Y. Liu, X.-L. Chen, X.-Y. L, S.-S. Zhu, S.-J. Li, Y. Song, L.-B. Qu and B. Yu, *J. Am. Chem. Soc.*, **2021**, *143*, 964-972; (b) W.-S. Guo, Q. Dou, J. Hou, L.-R. Wen and M. Li, *J. Org. Chem.*, **2017**, *82*, 7015-7022.
- [2] C. C. Chen and J. Waser, *Chem. Commun.*, **2014**, *50*, 12923-12926.

11. NMR Spectra

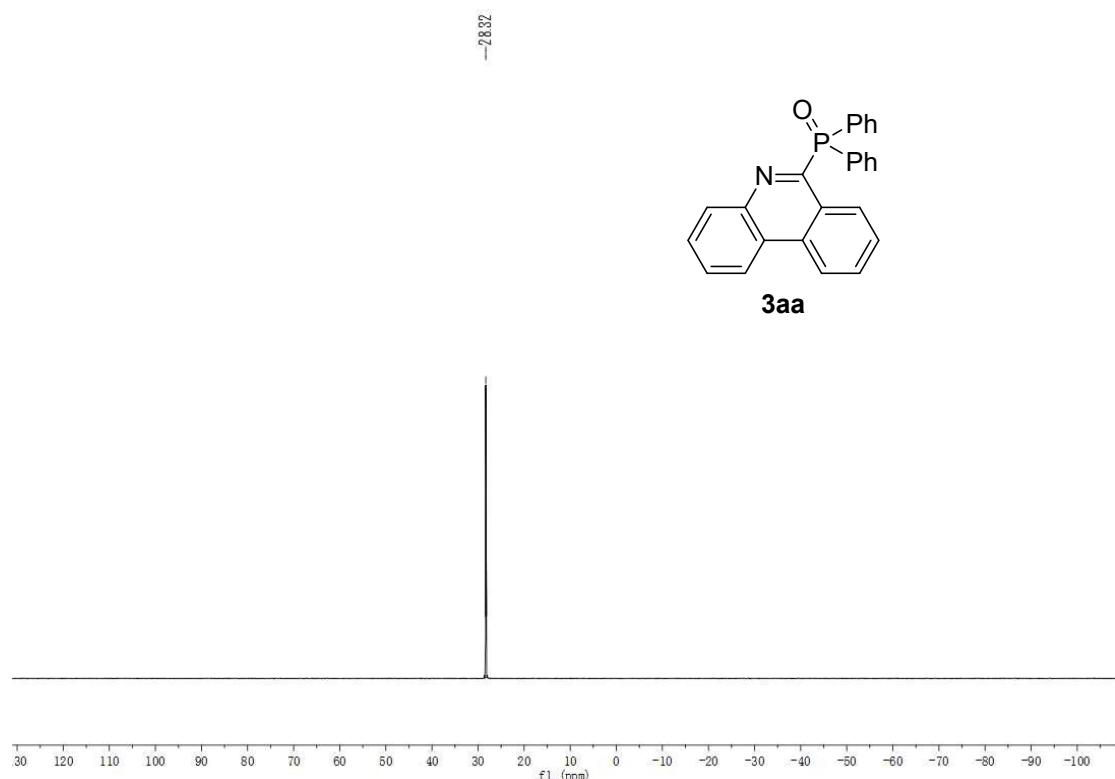
¹H NMR spectrum of 3aa



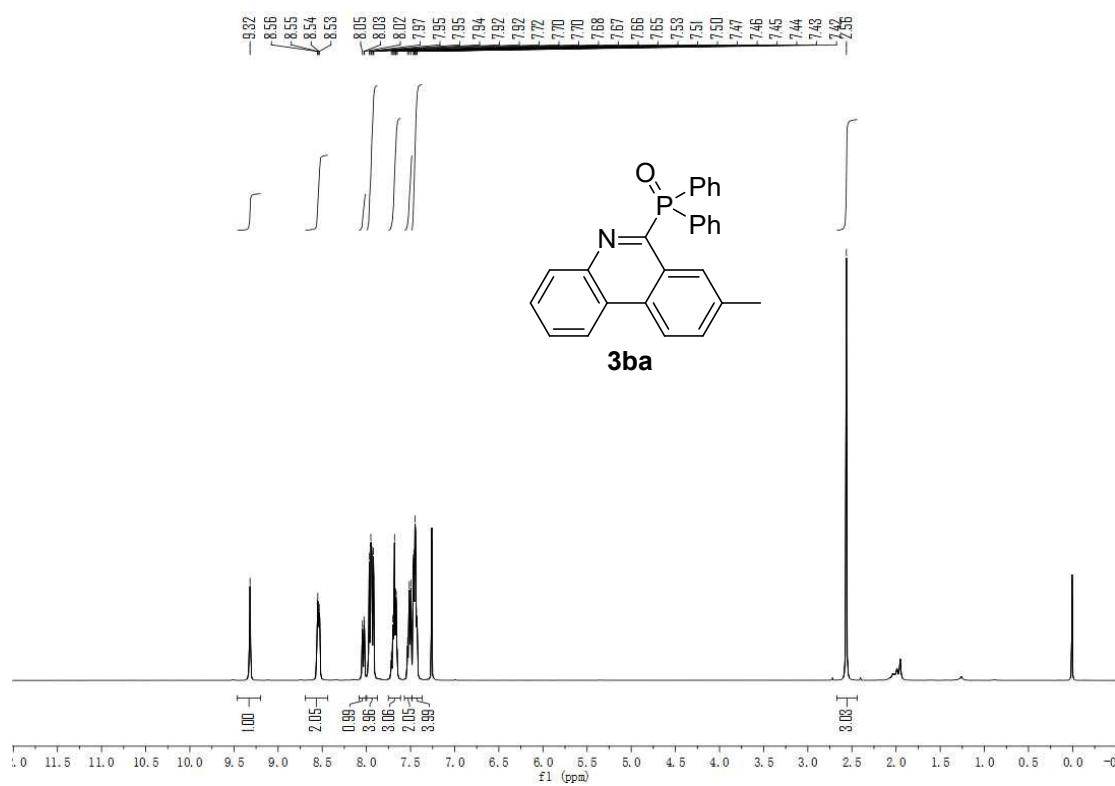
¹³C NMR spectrum of 3aa



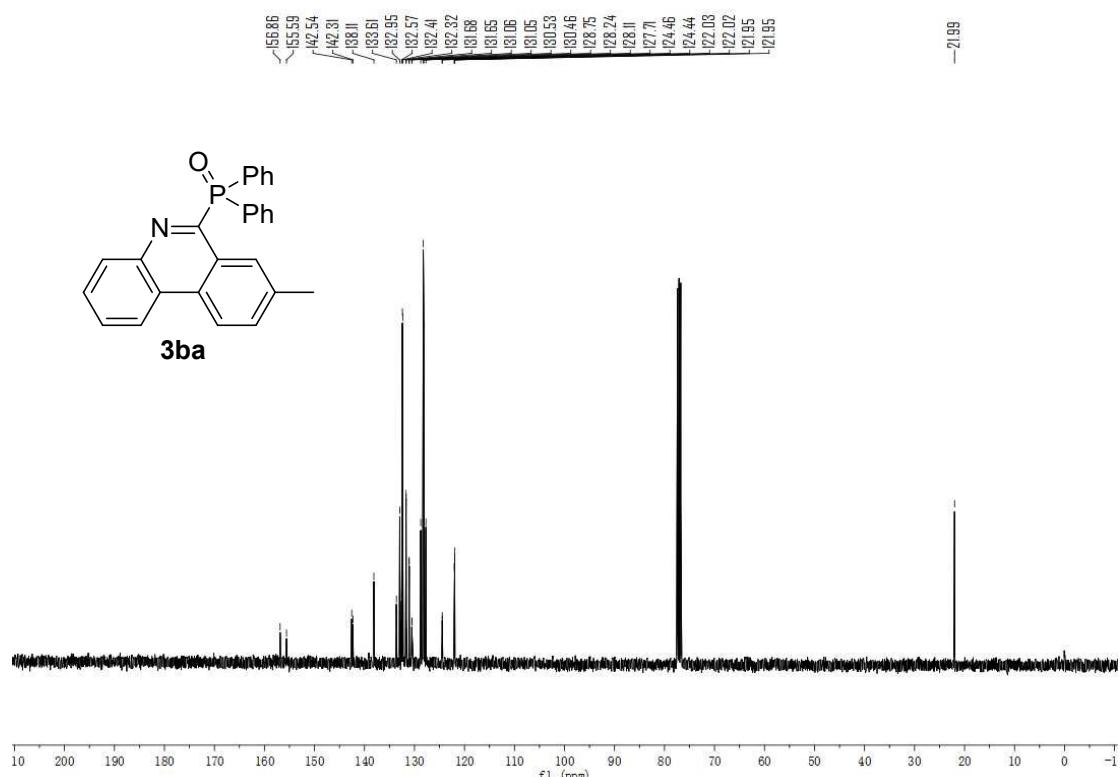
^{31}P NMR spectrum of 3aa



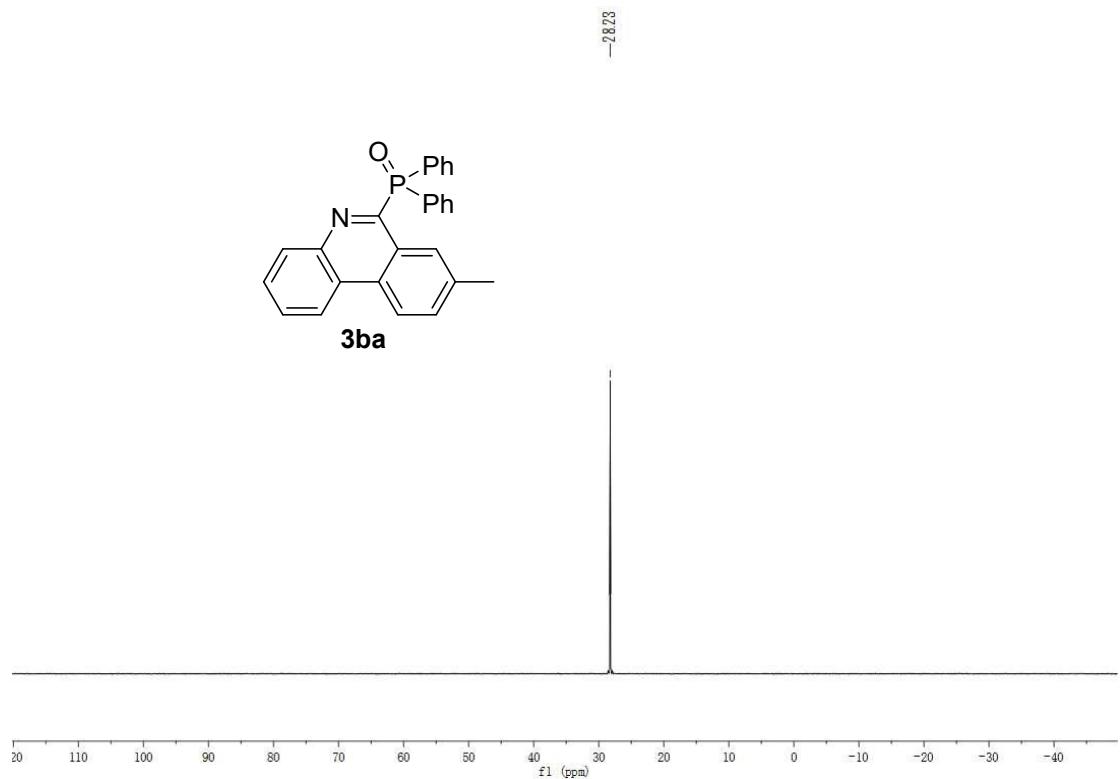
^1H NMR spectrum of 3ba



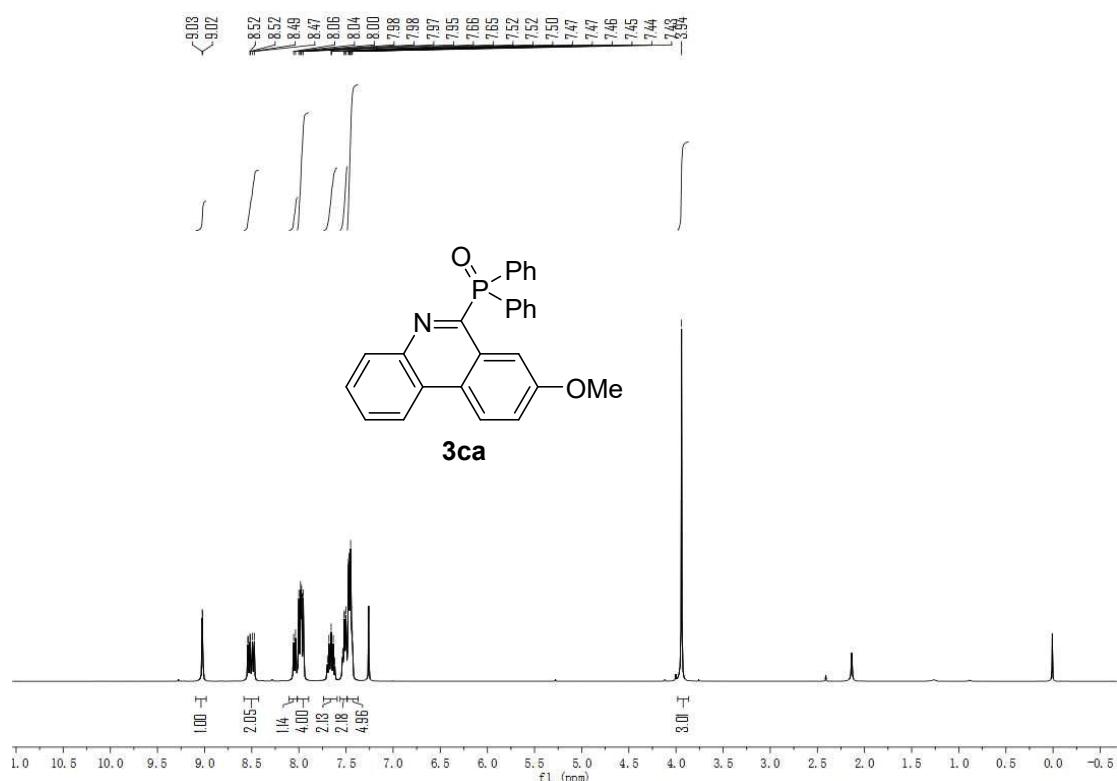
¹³C NMR spectrum of 3ba



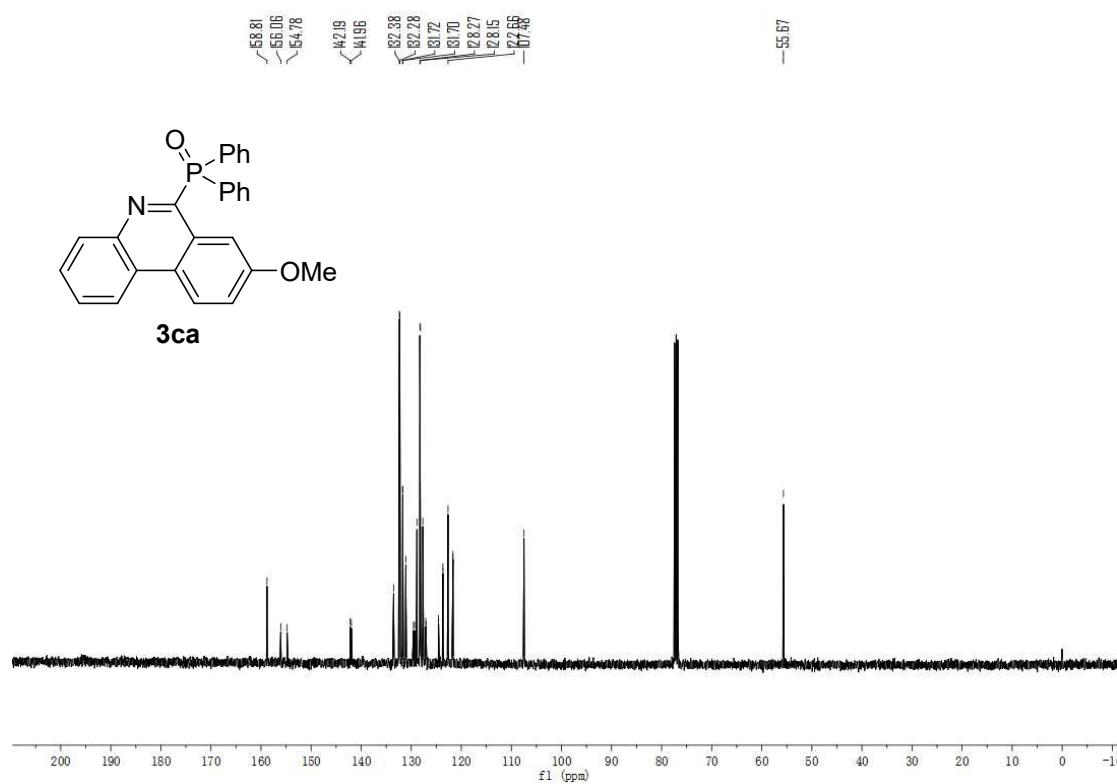
³¹P NMR spectrum of 3ba



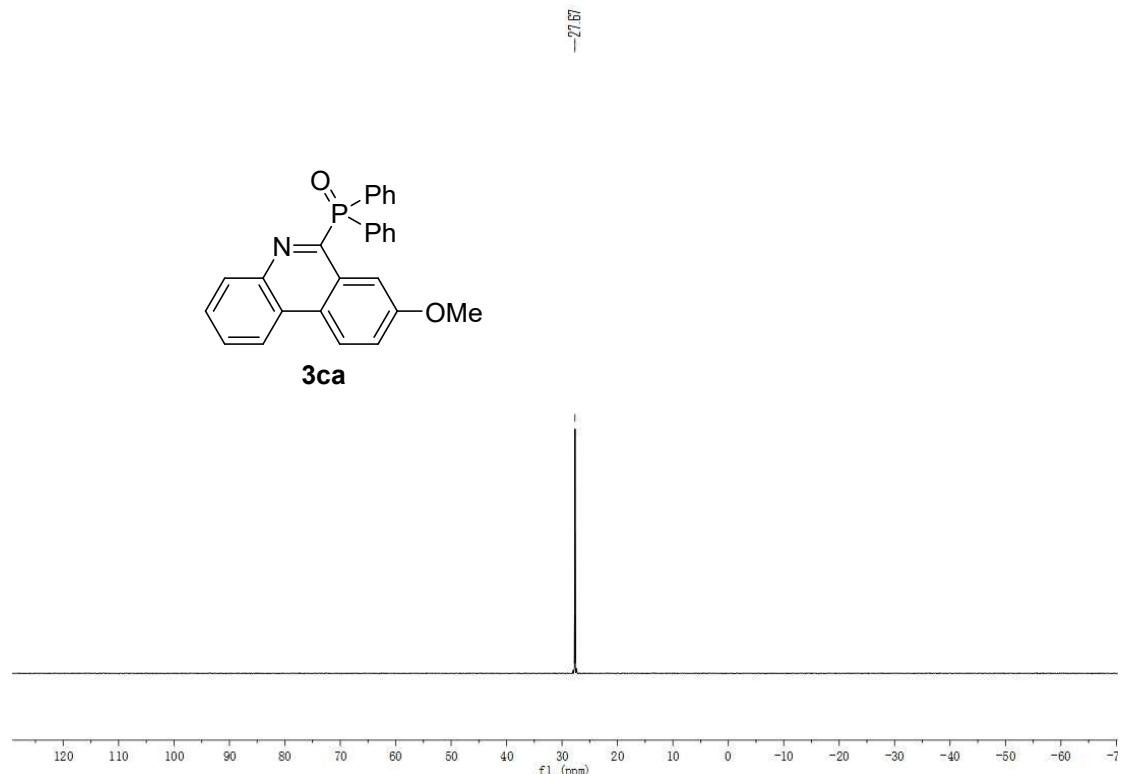
¹H NMR spectrum of 3ca



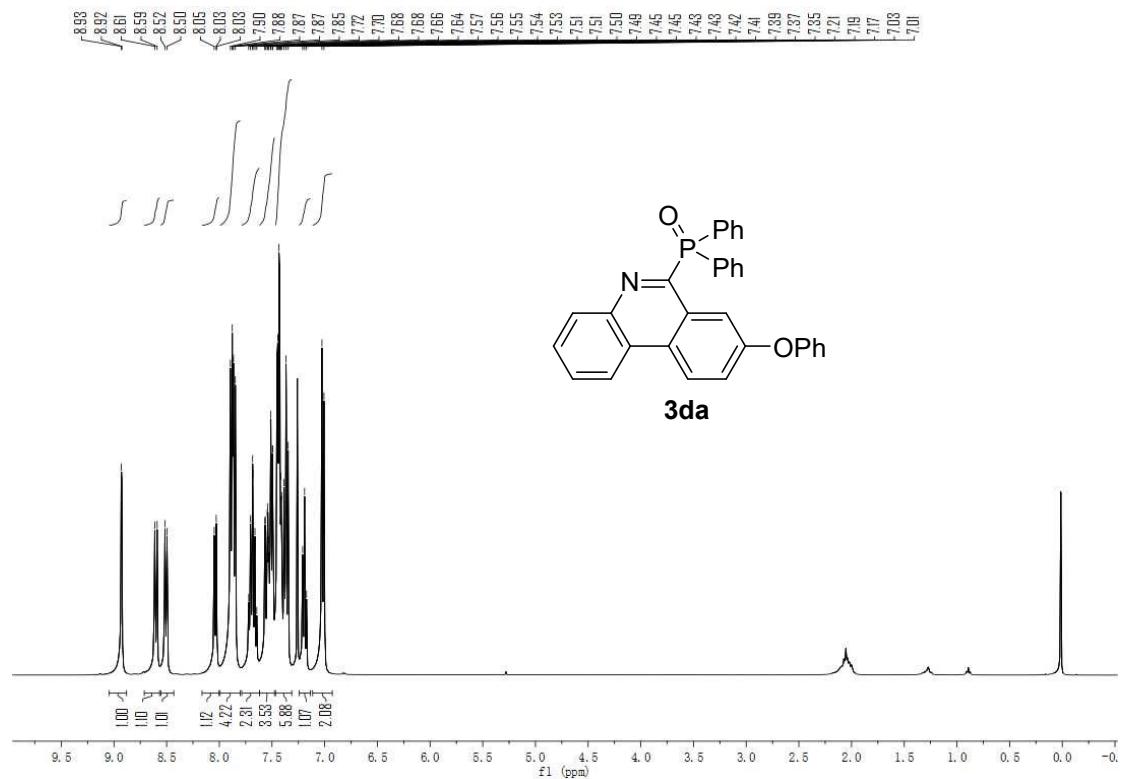
¹³C NMR spectrum of 3ca



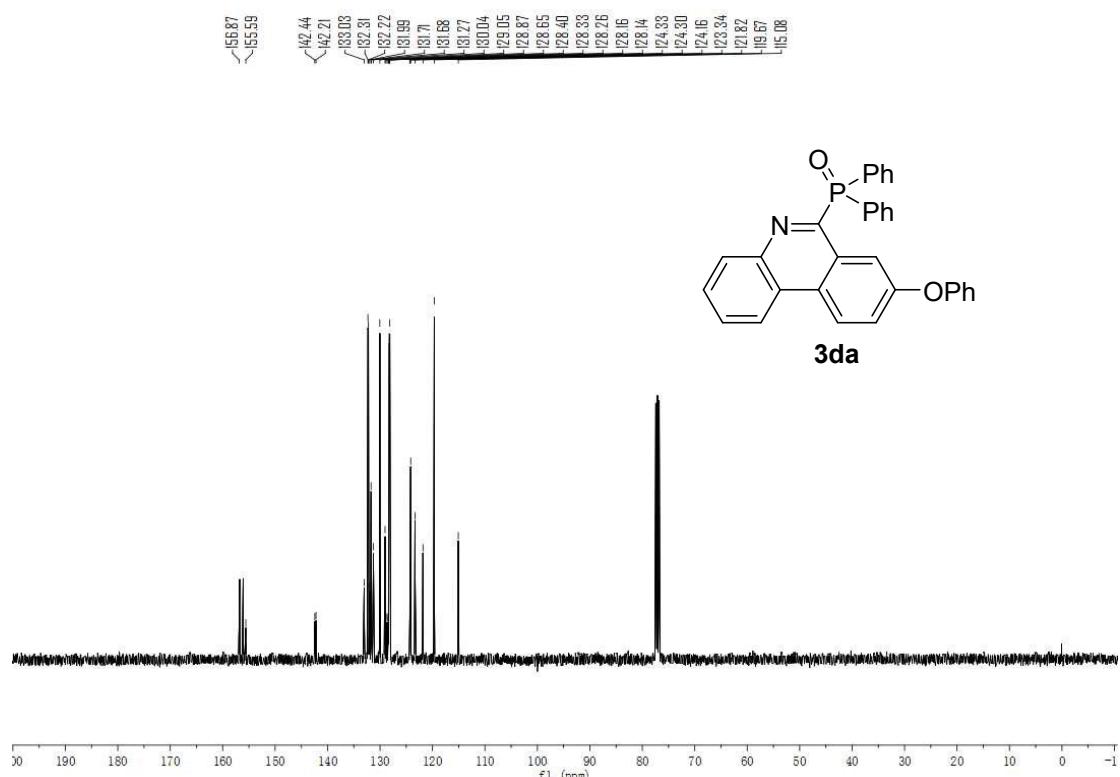
^{31}P NMR spectrum of 3ca



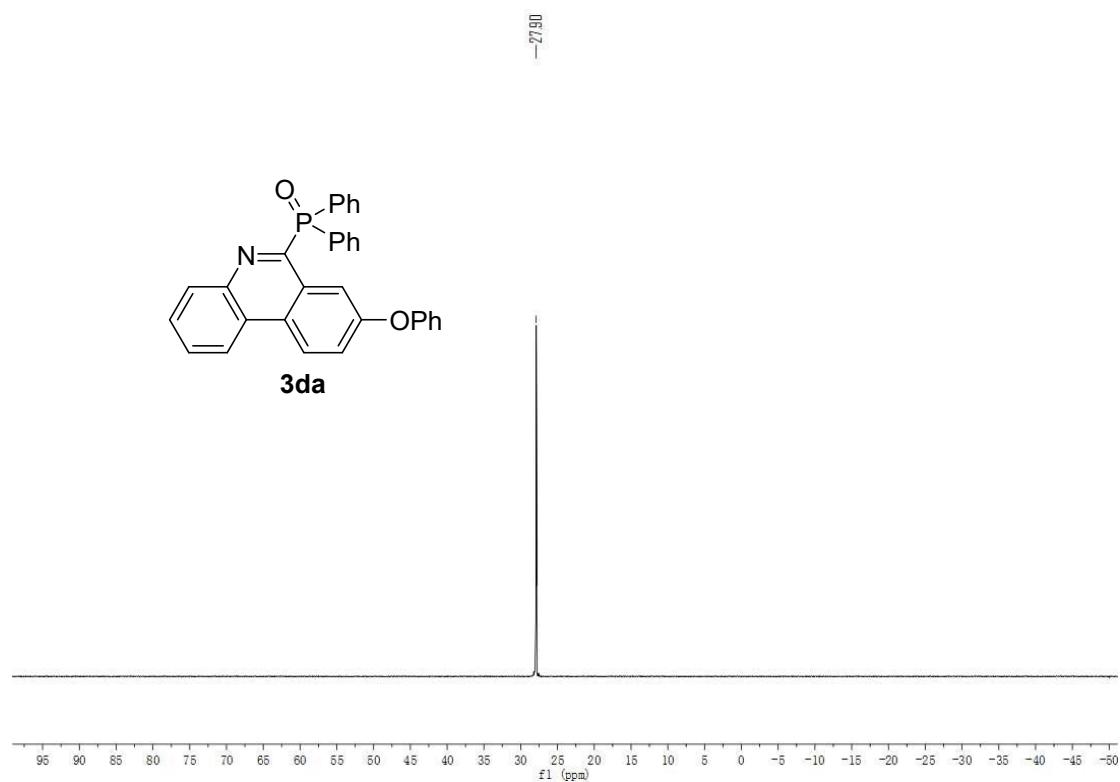
^1H NMR spectrum of 3da



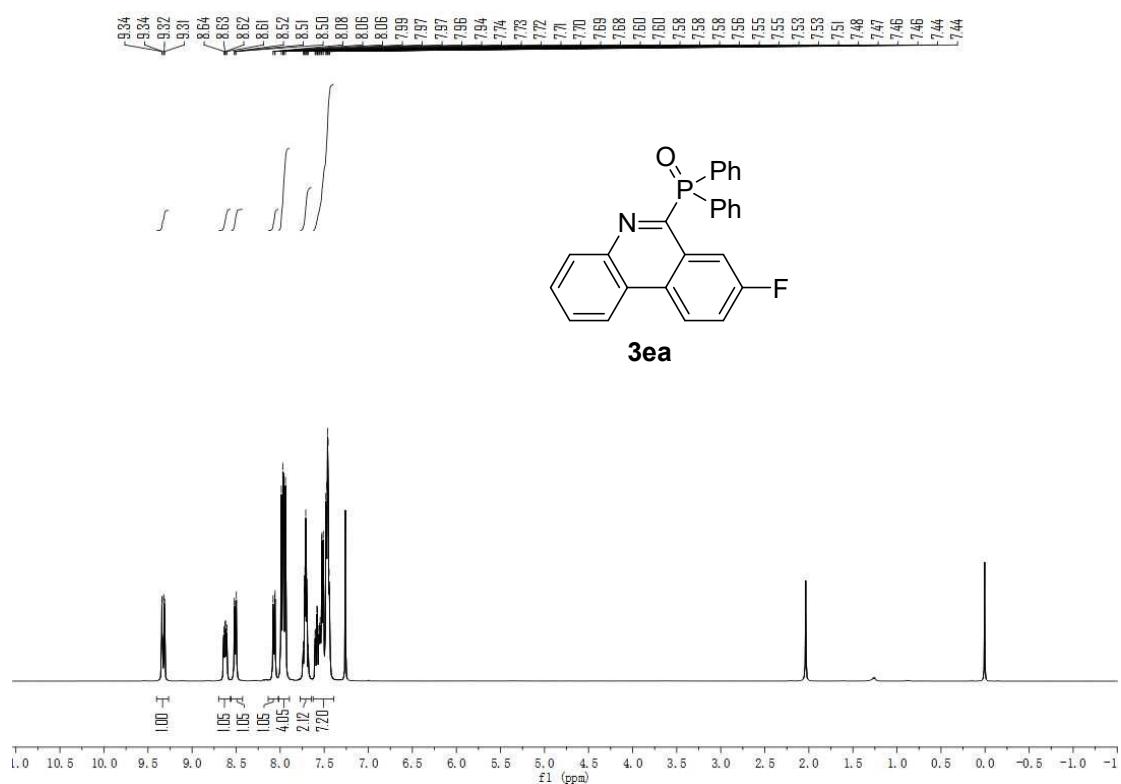
^{13}C NMR spectrum of 3da



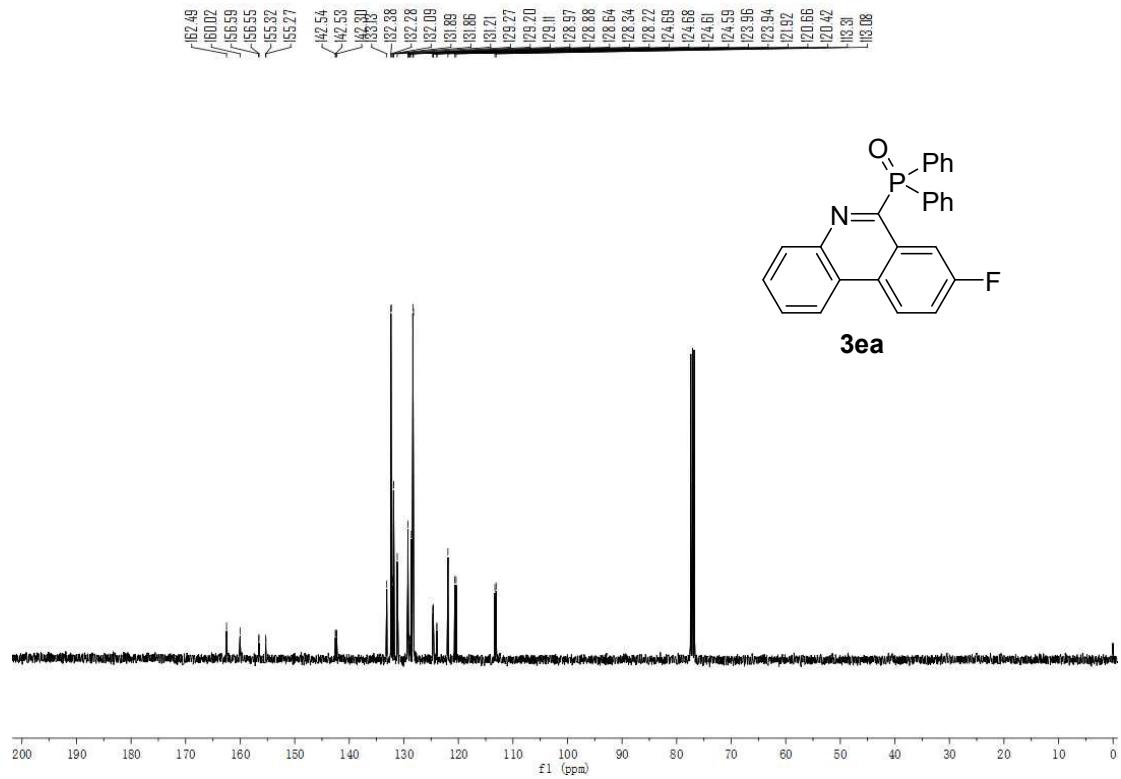
^{31}P NMR spectrum of 3da



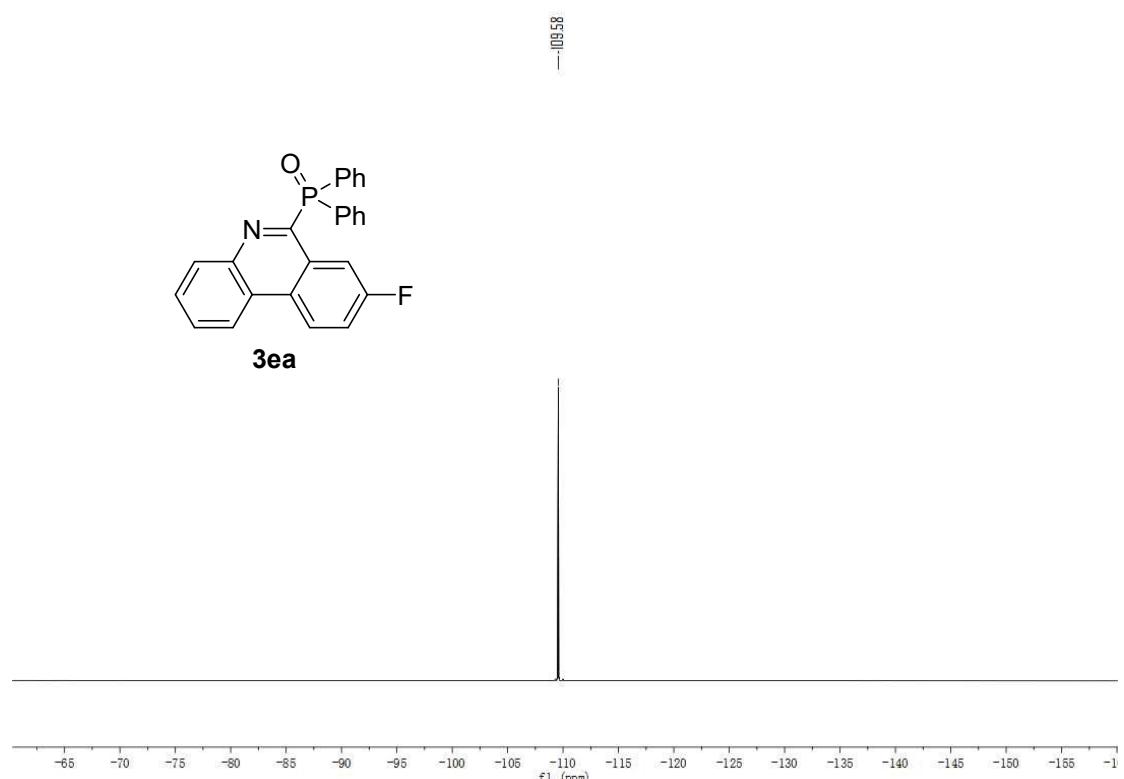
¹H NMR spectrum of 3ea



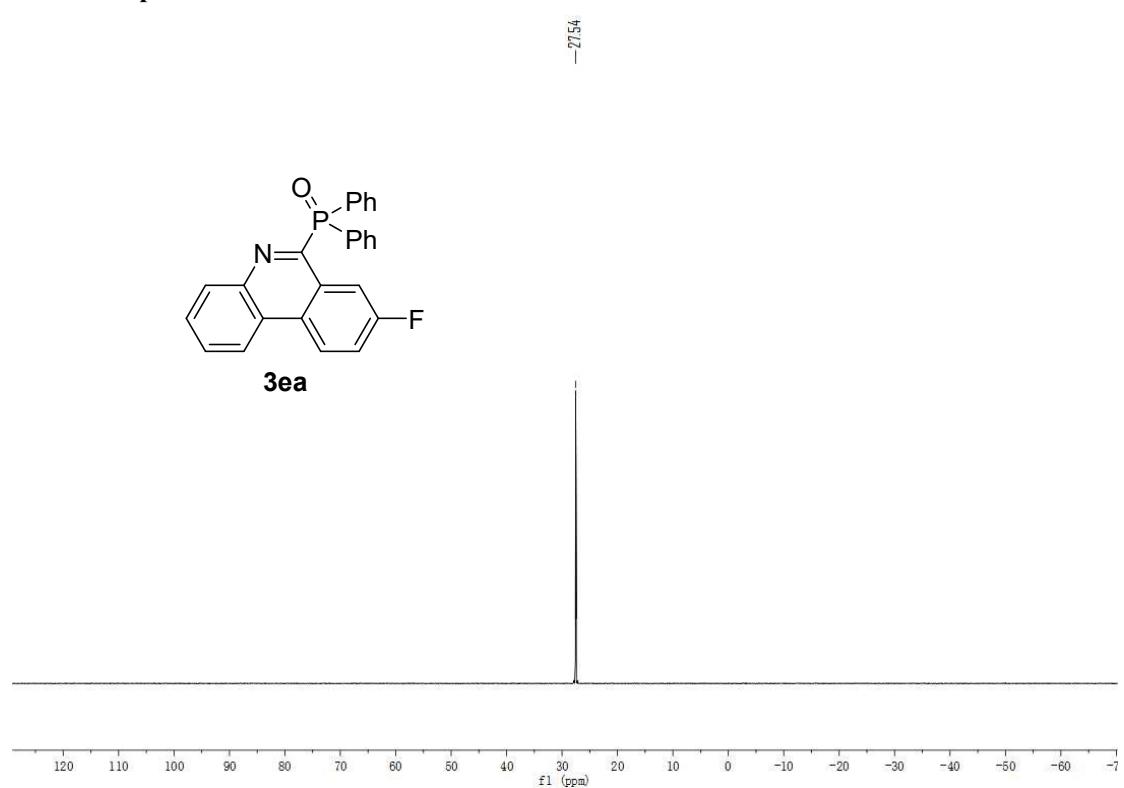
¹³C NMR spectrum of 3ea



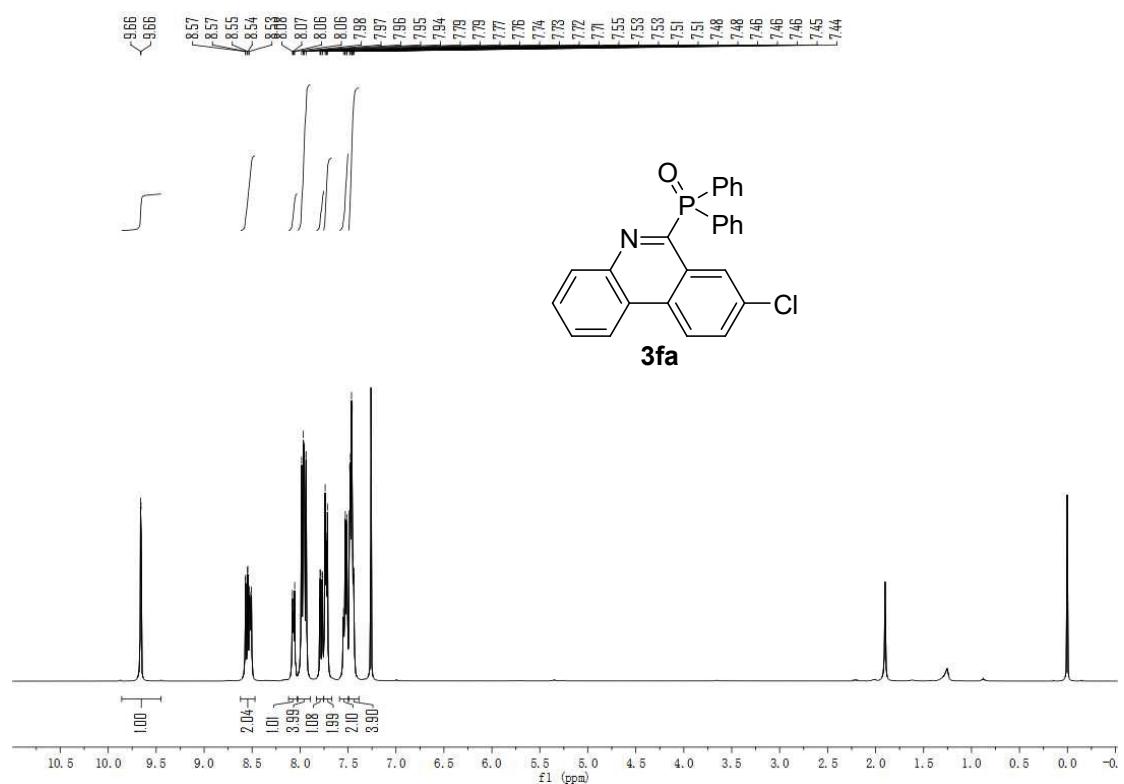
¹⁹F NMR spectrum of 3ea



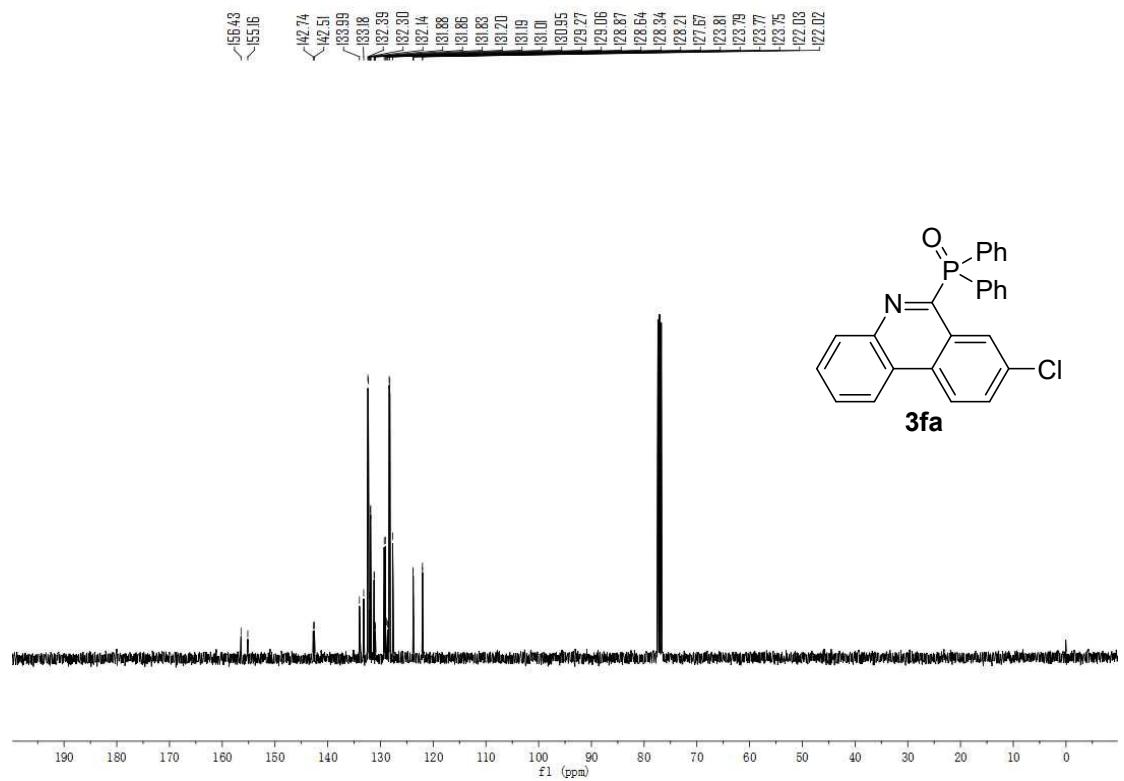
³¹P NMR spectrum of 3ea



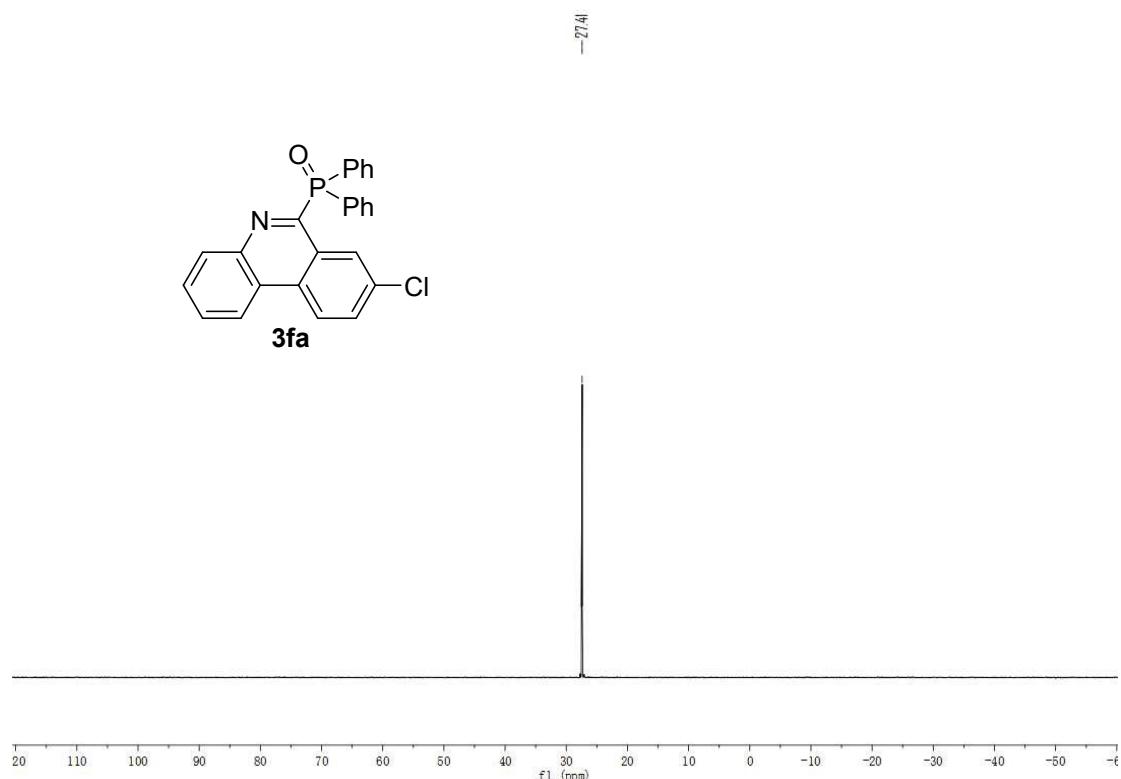
¹H NMR spectrum of 3fa



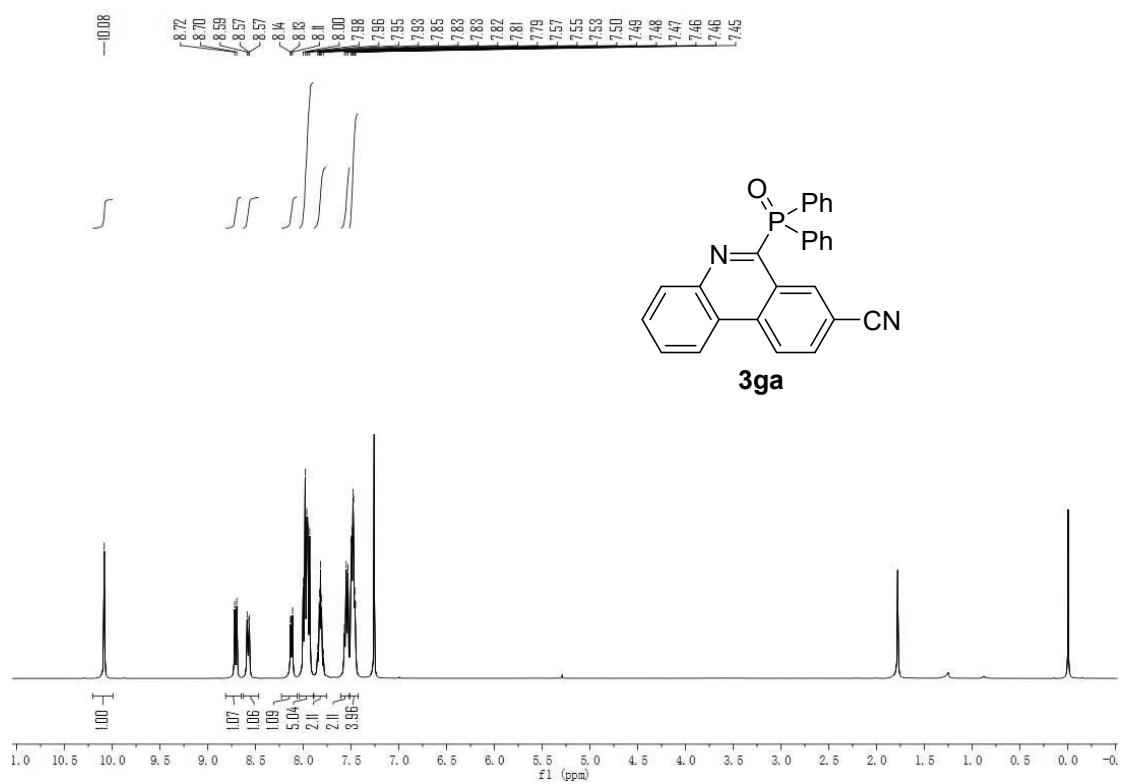
¹³C NMR spectrum of 3fa



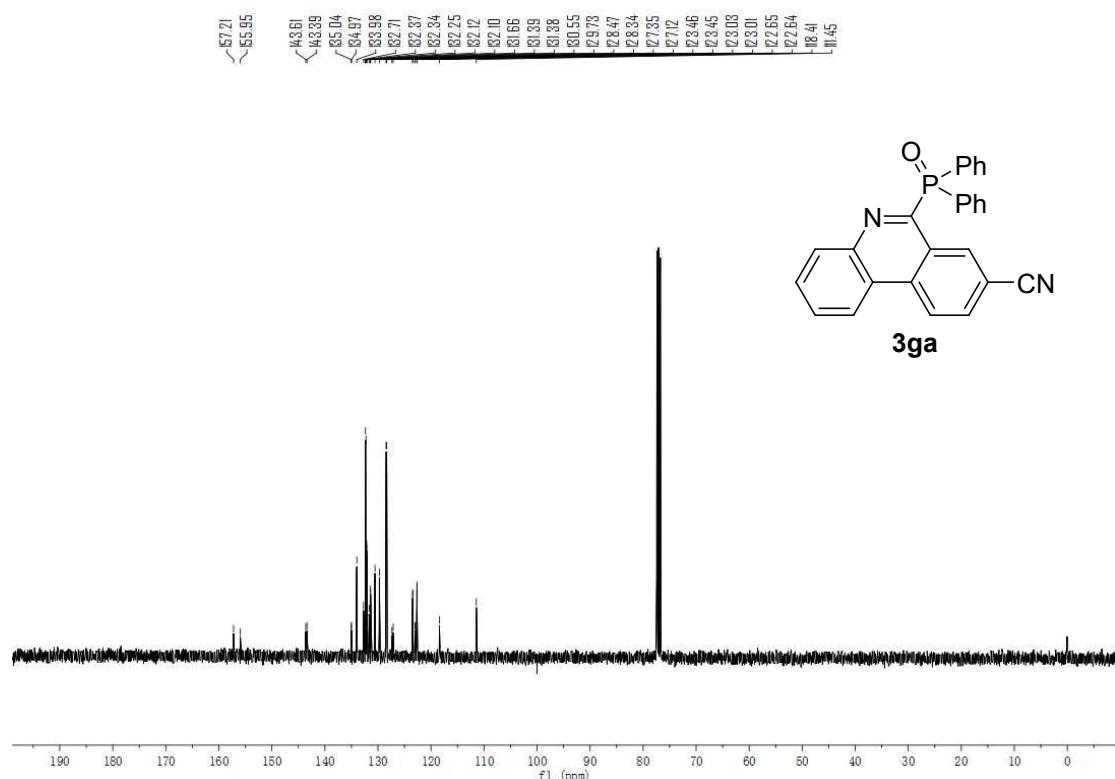
³¹P NMR spectrum of 3fa



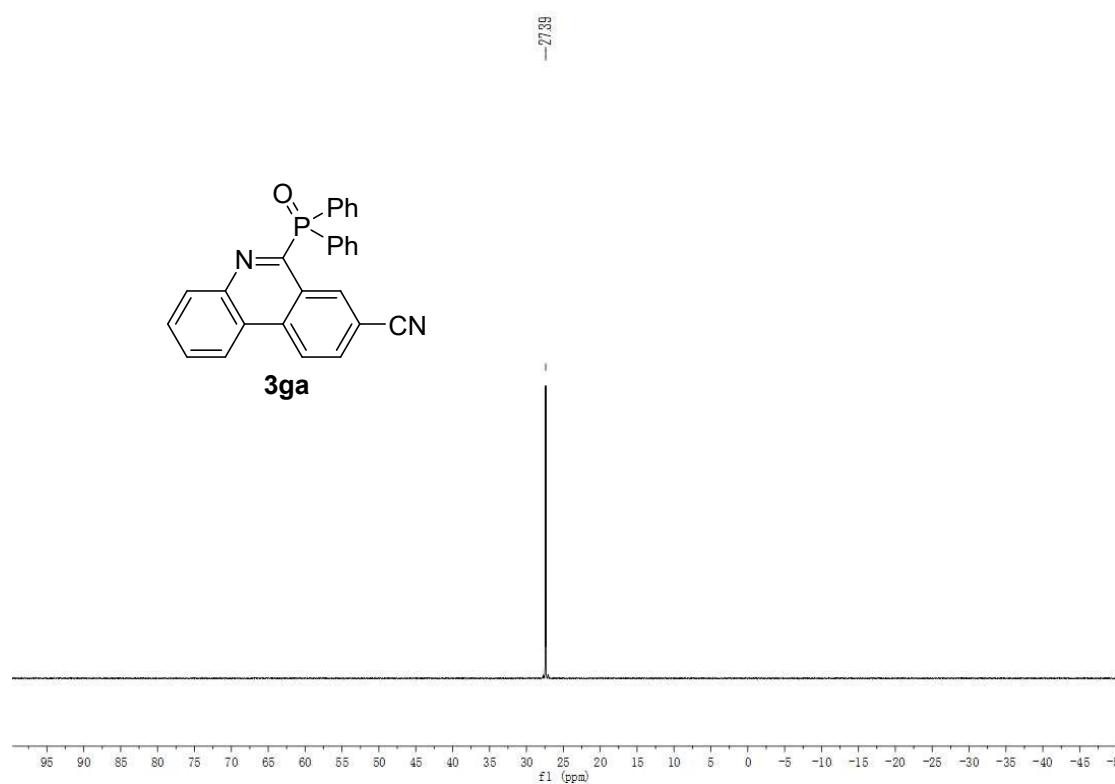
¹H NMR spectrum of 3ga



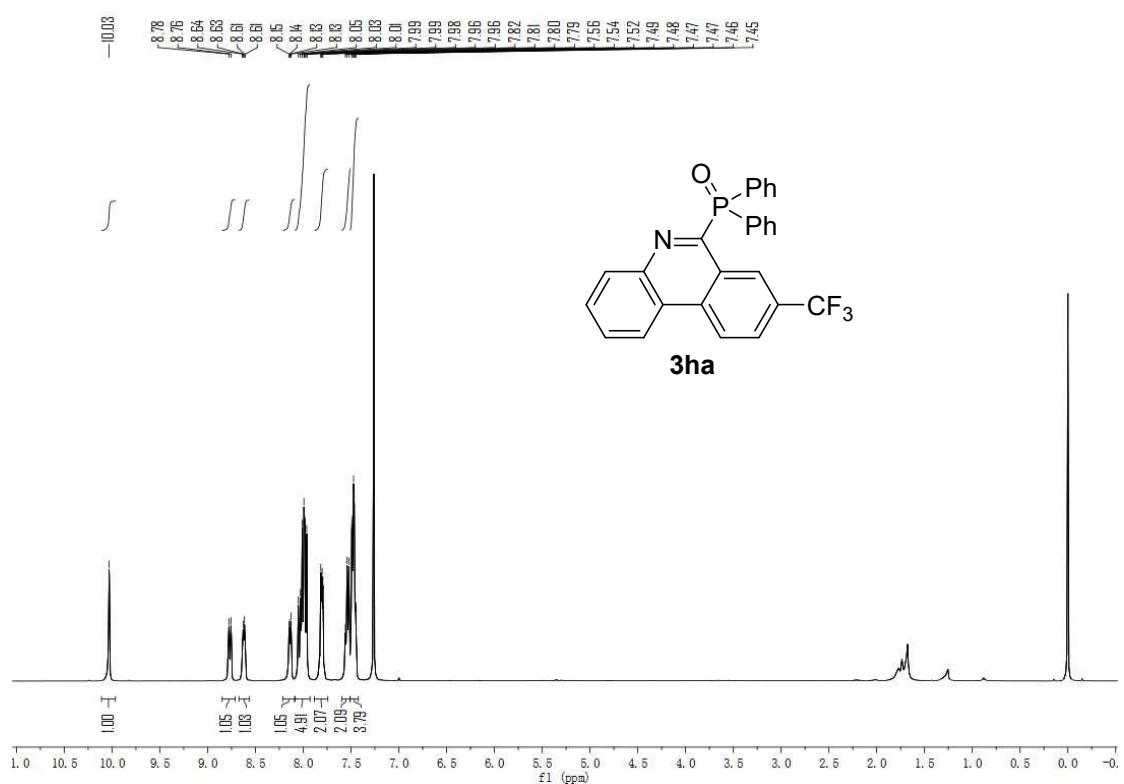
¹³C NMR spectrum of 3ga



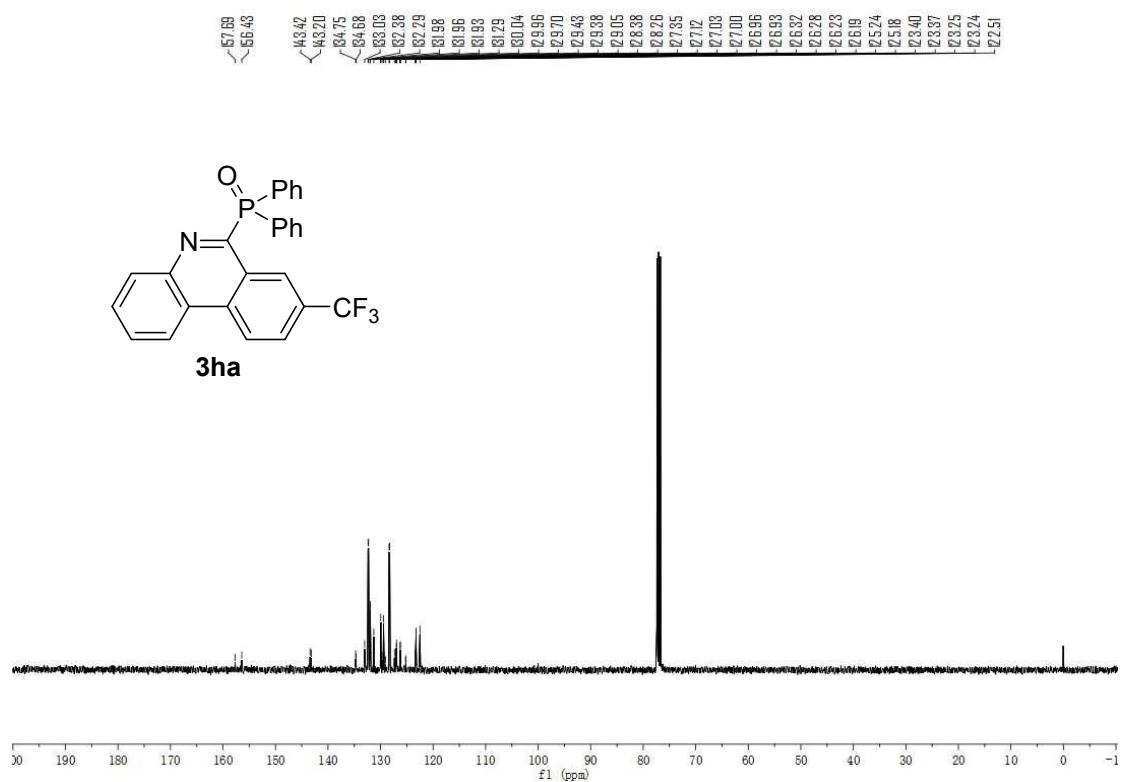
³¹P NMR spectrum of 3ga



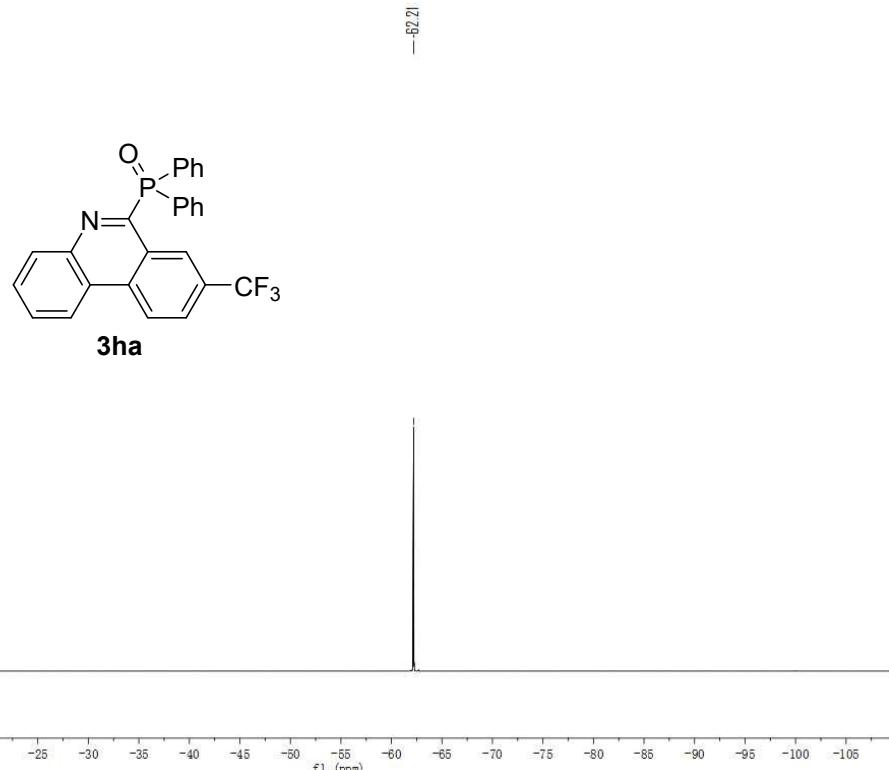
¹H NMR spectrum of 3ha



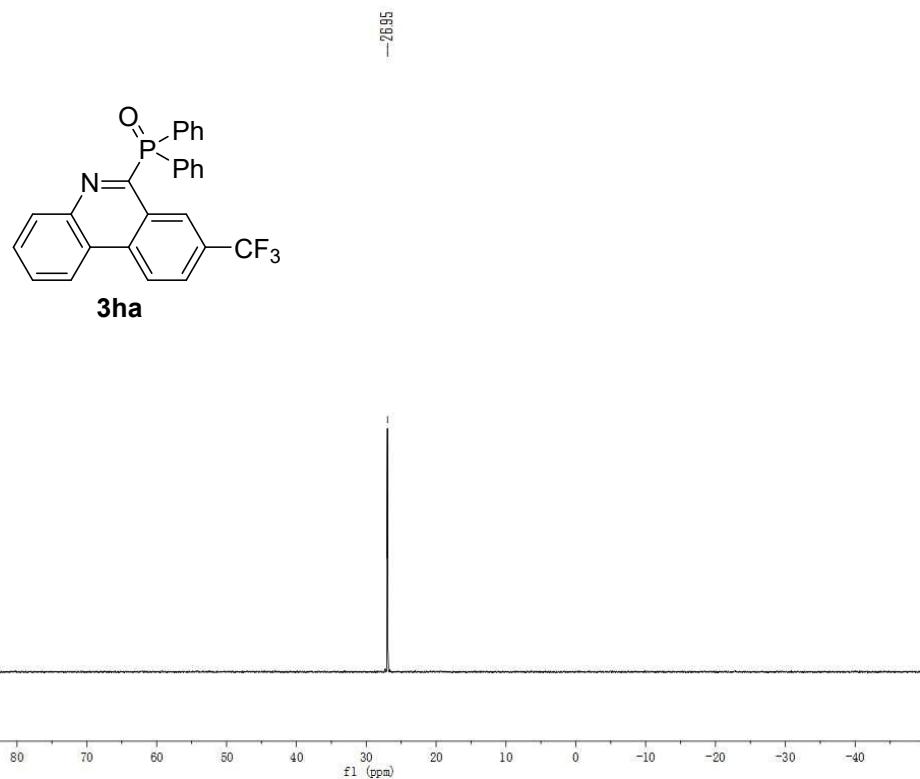
¹³C NMR spectrum of 3ha



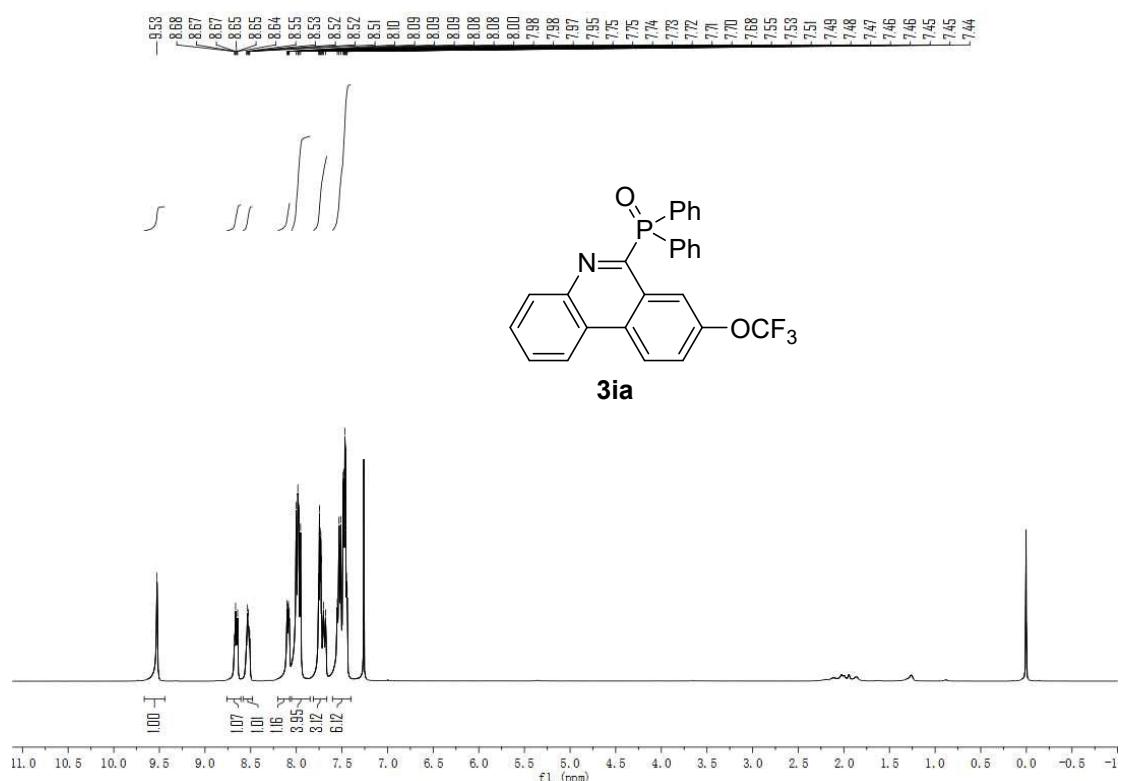
¹⁹F NMR spectrum of 3ha



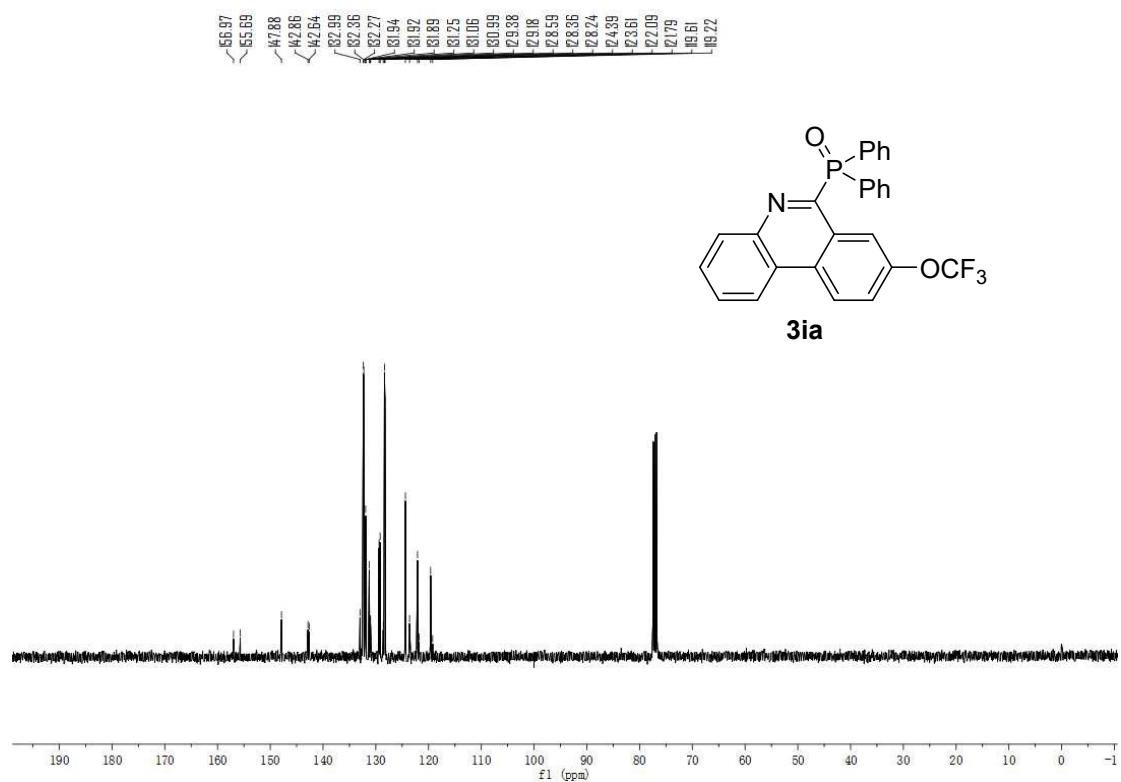
³¹P NMR spectrum of 3ha



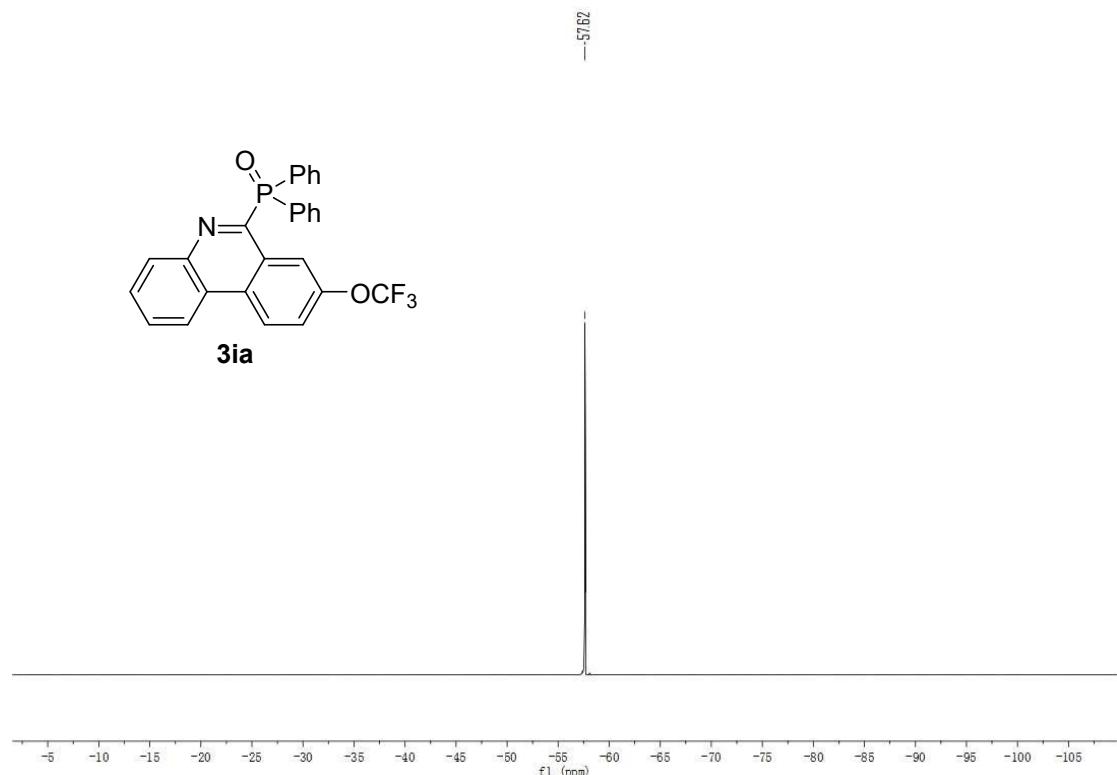
¹H NMR spectrum of 3ia



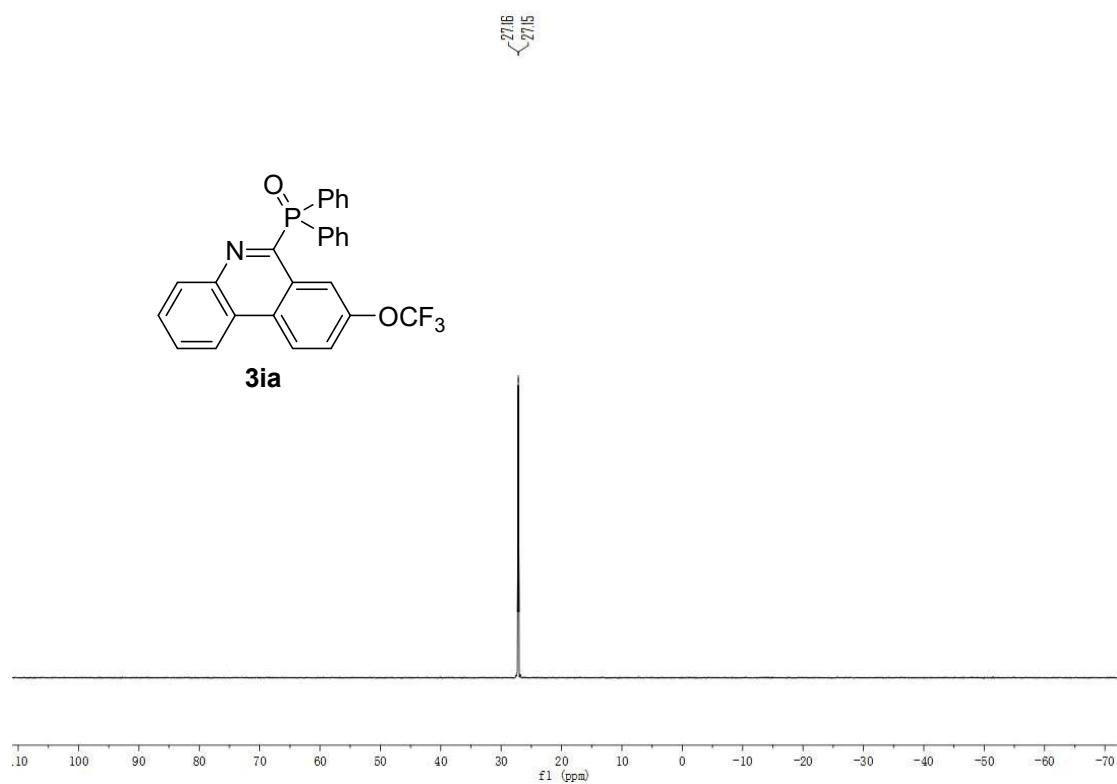
¹³C NMR spectrum of 3ia



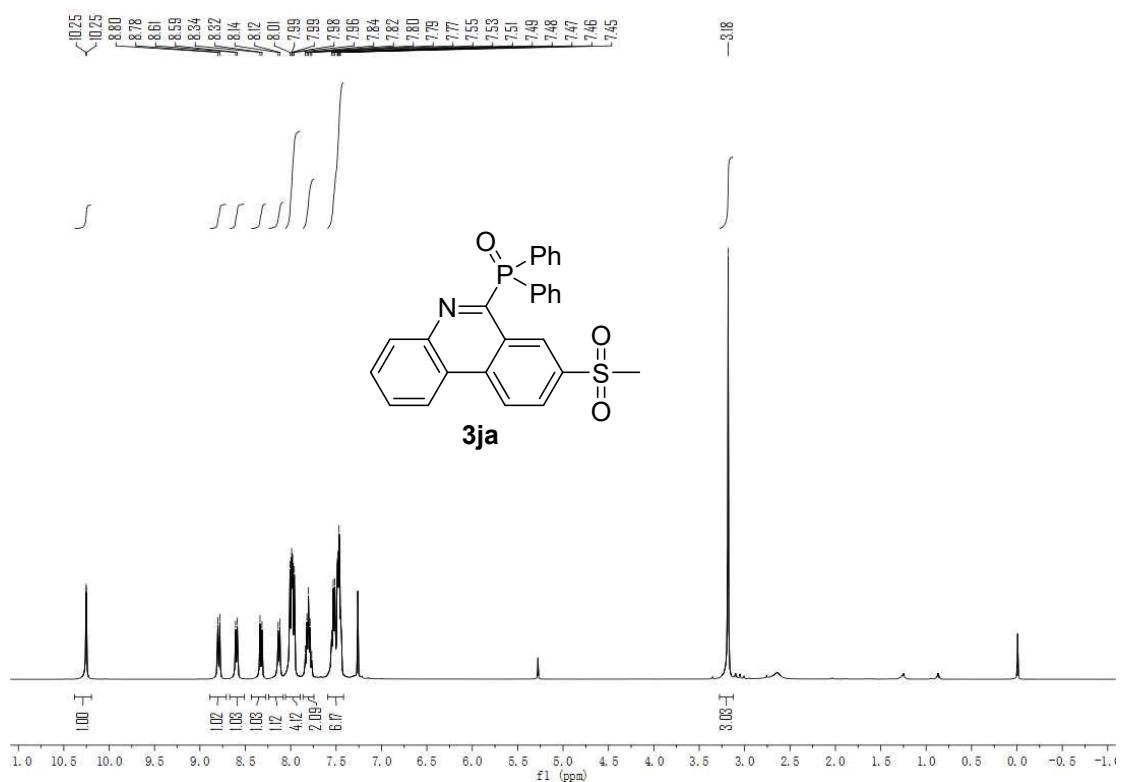
¹⁹F NMR spectrum of 3ia



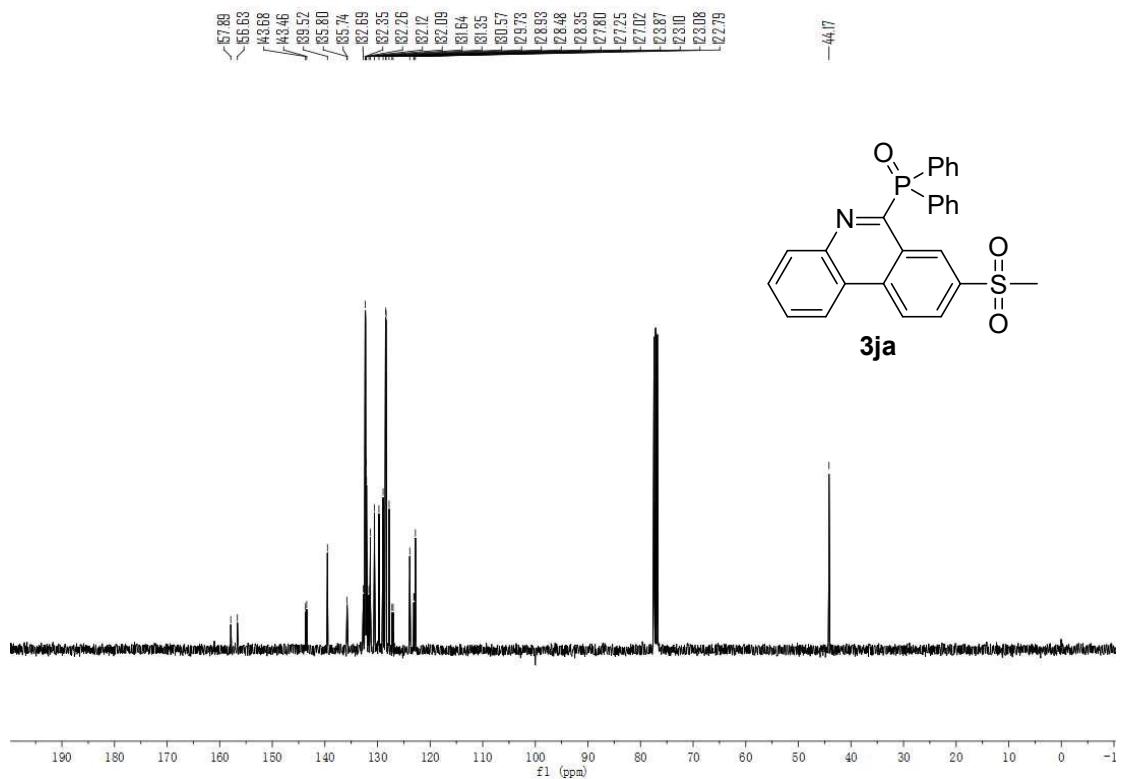
³¹P NMR spectrum of 3ia



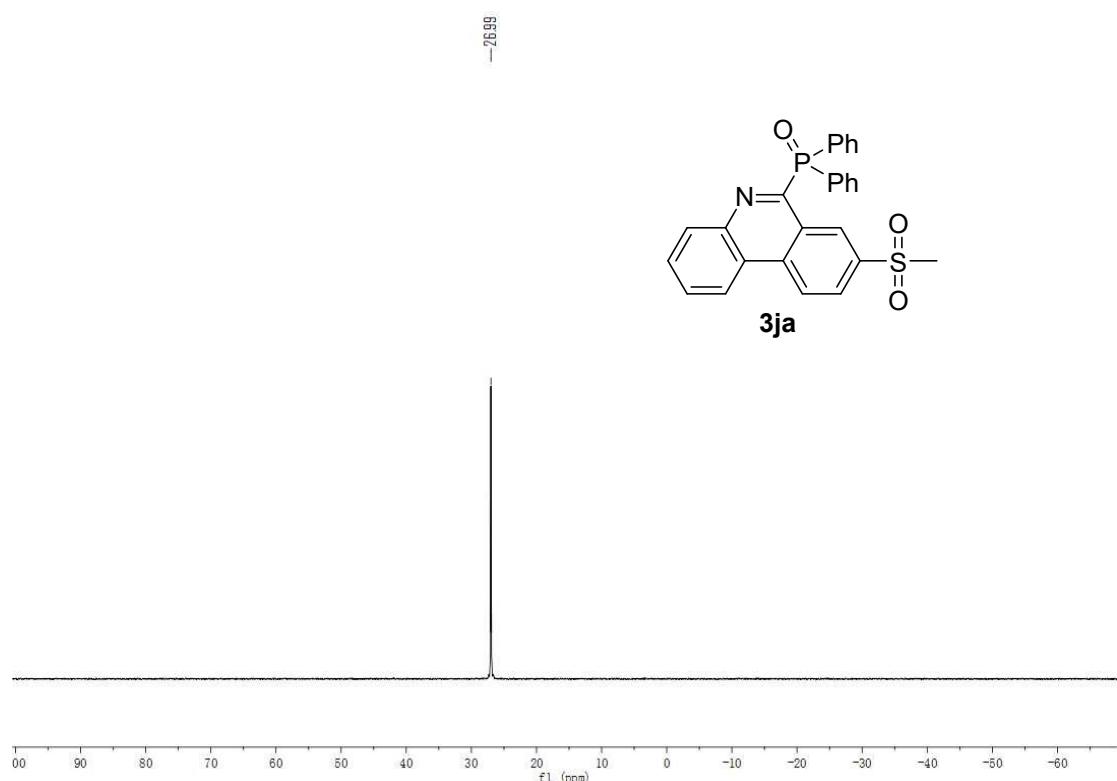
¹H NMR spectrum of 3ja



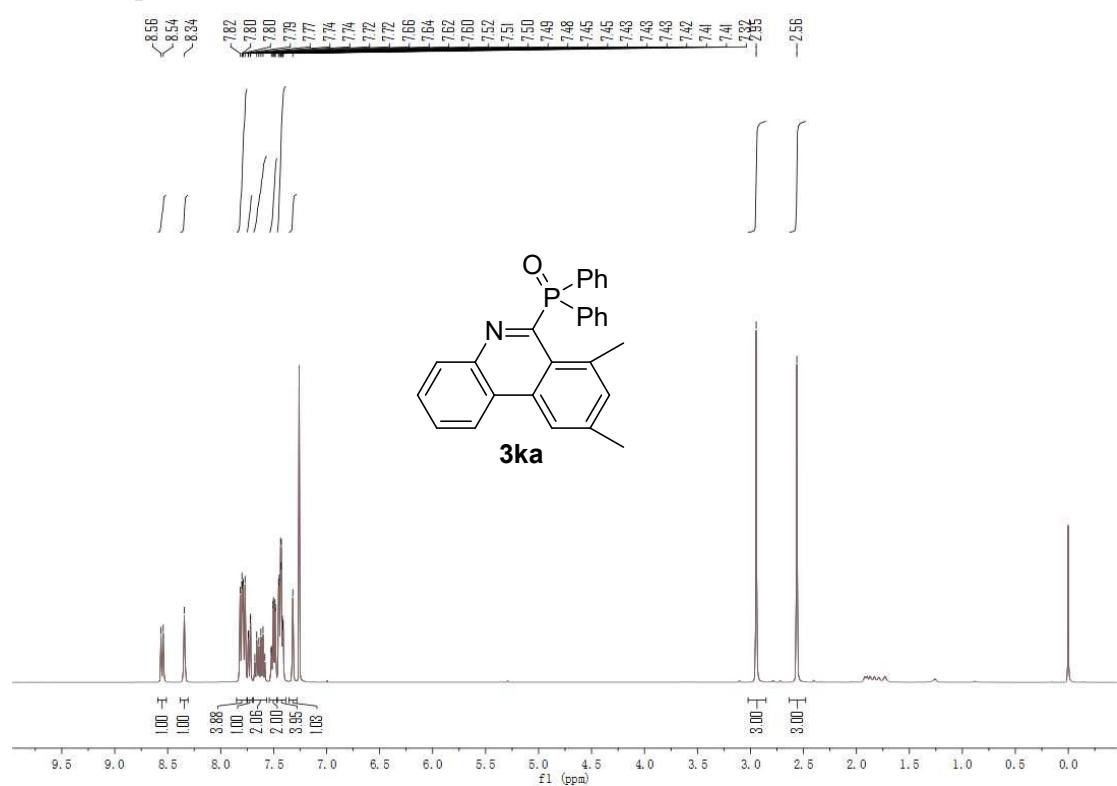
¹³C NMR spectrum of 3ja



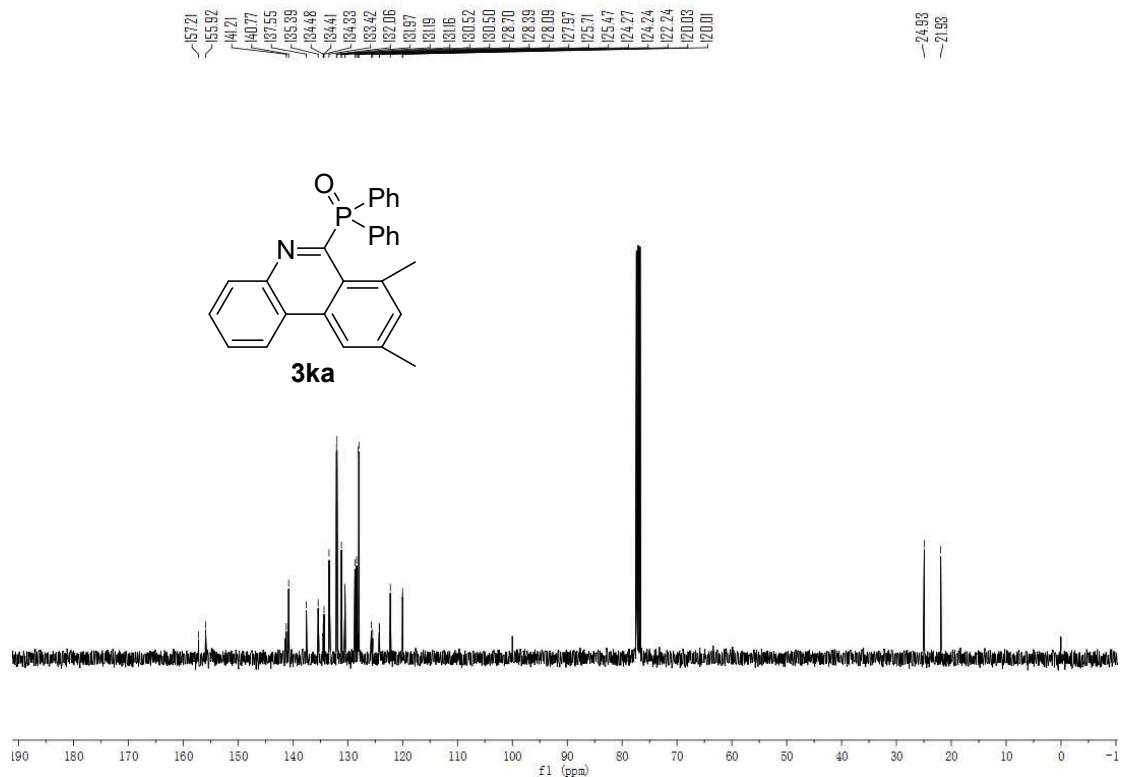
³¹P NMR spectrum of 3ja



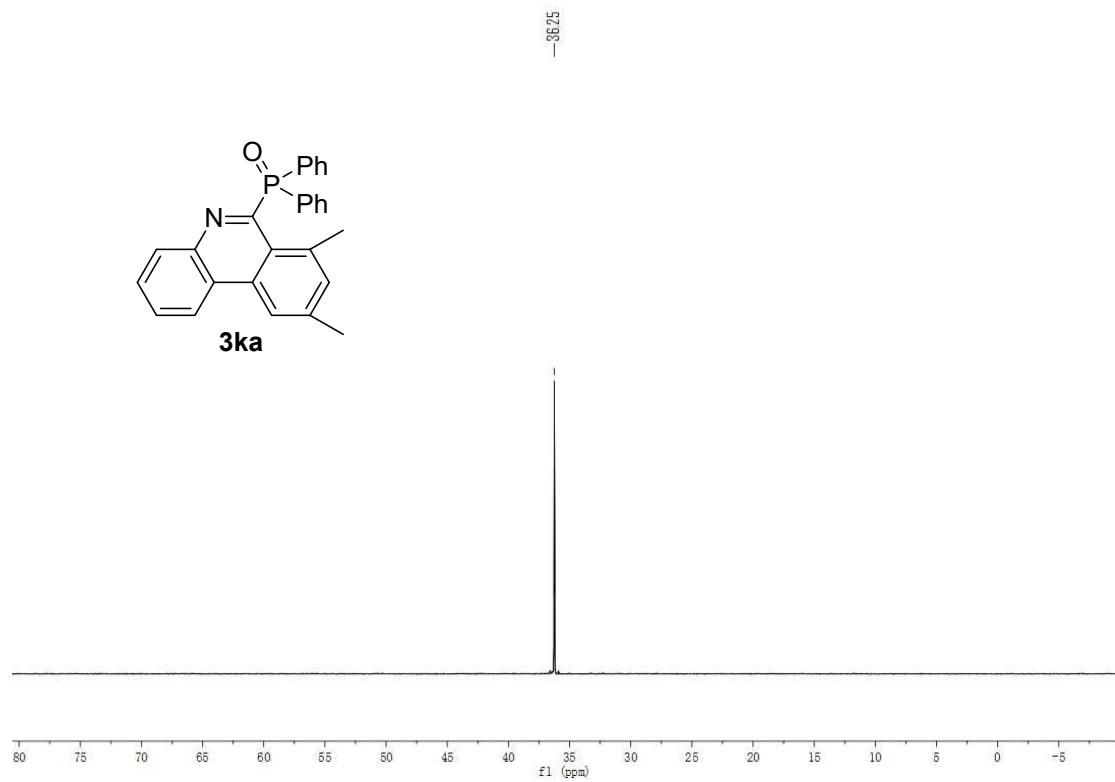
¹H NMR spectrum of 3ka



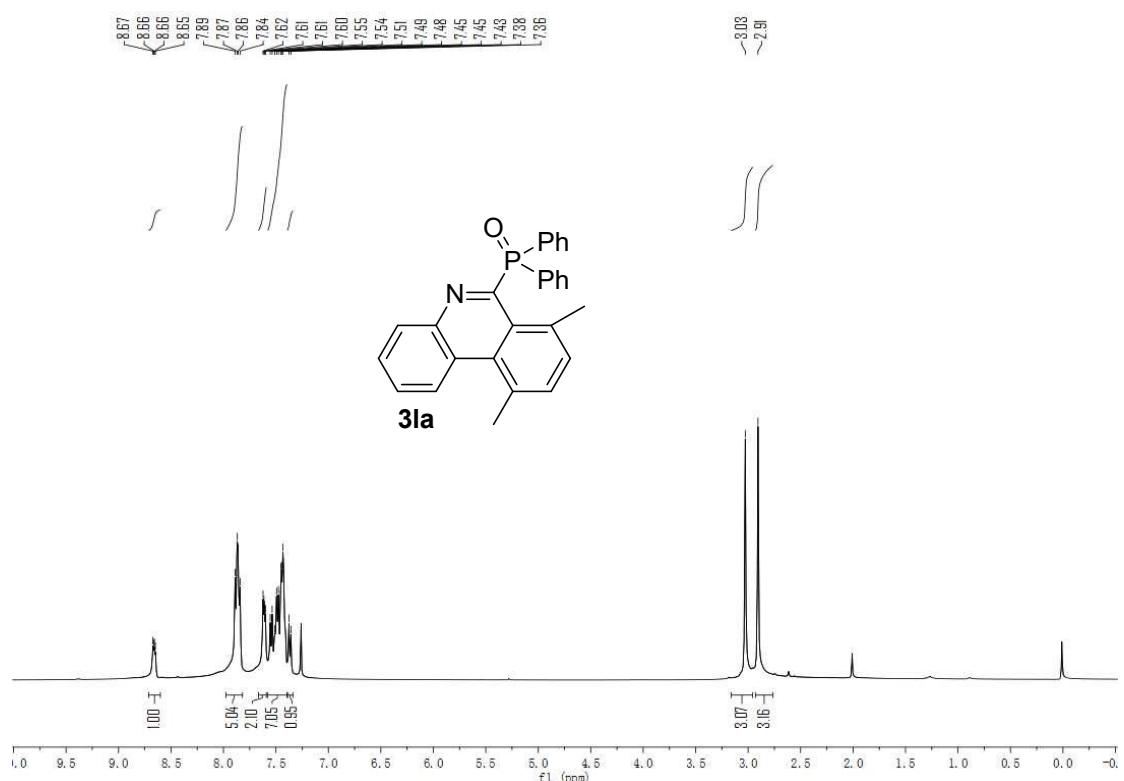
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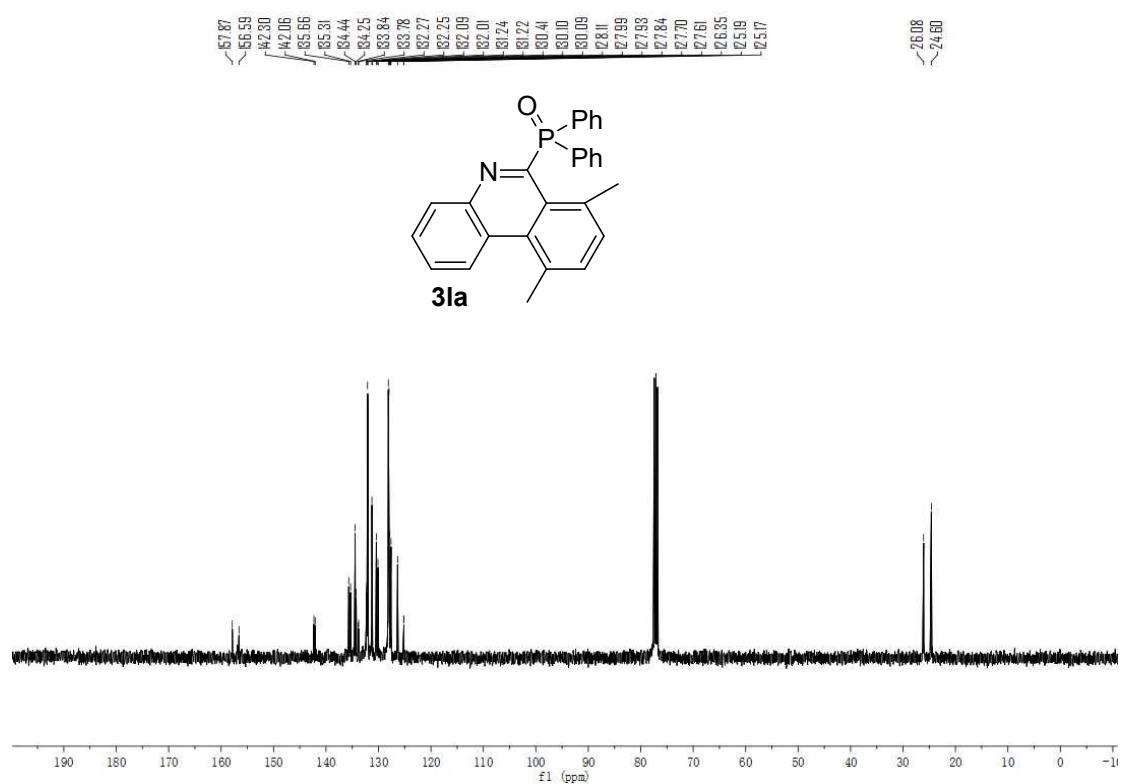
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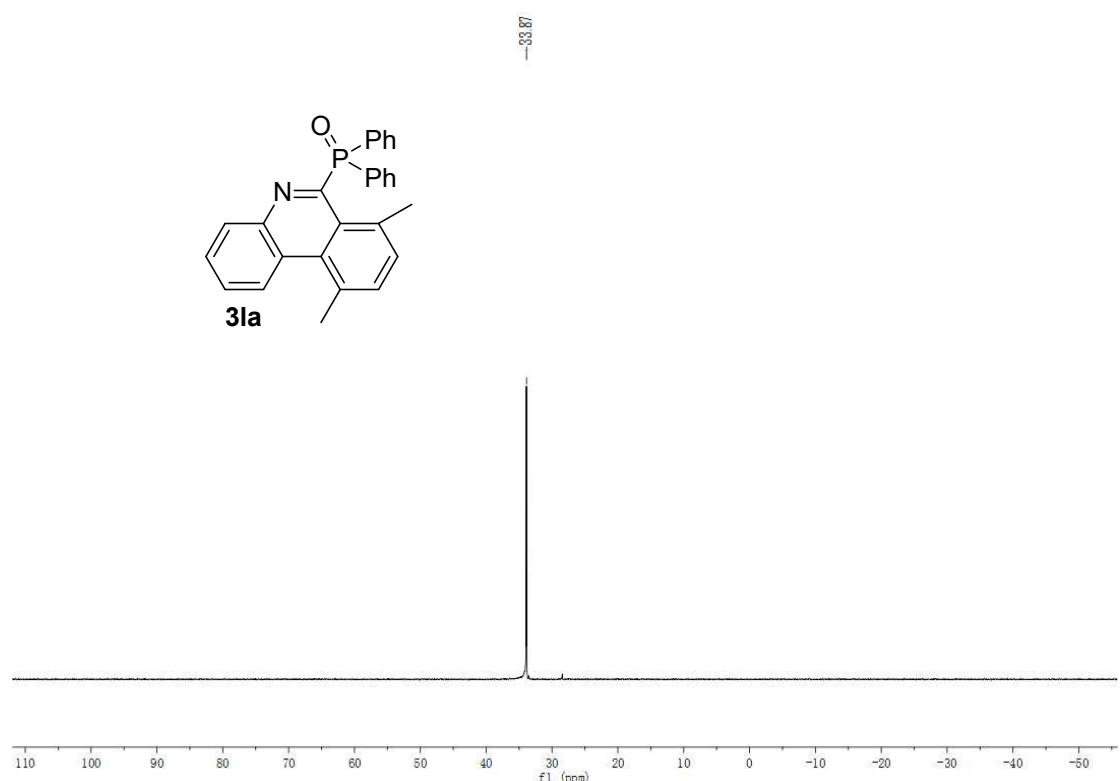
¹H NMR spectrum of 3la



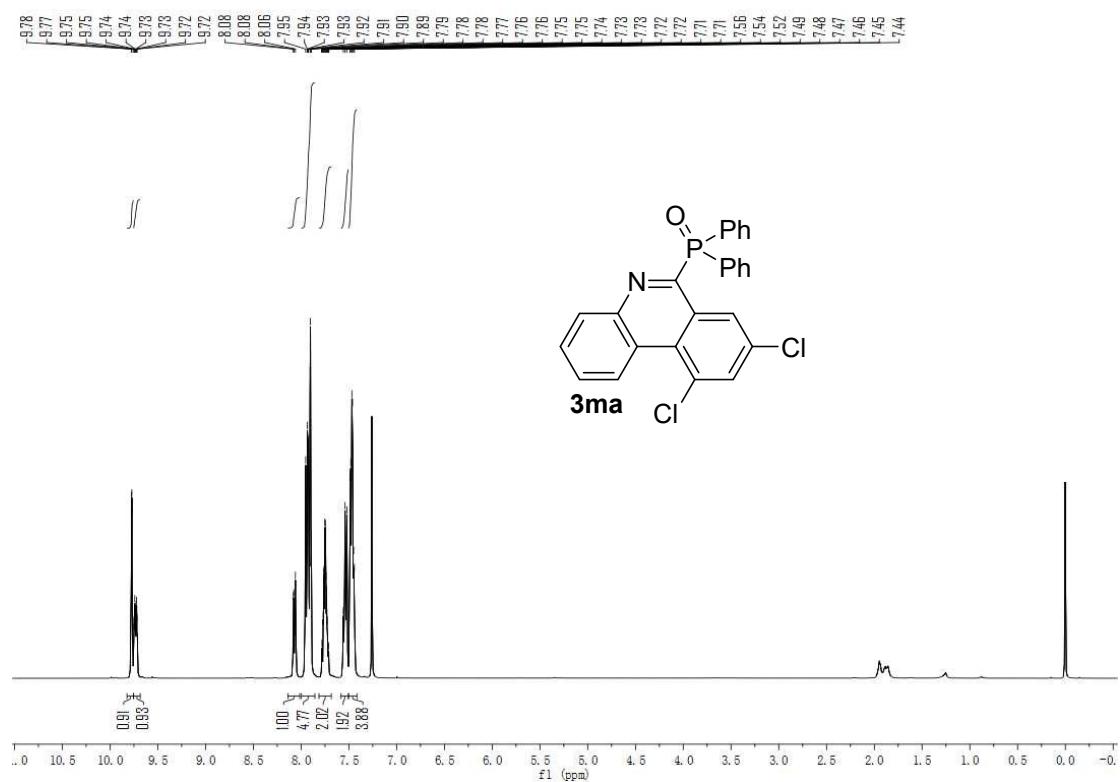
¹³C NMR spectrum of 3la



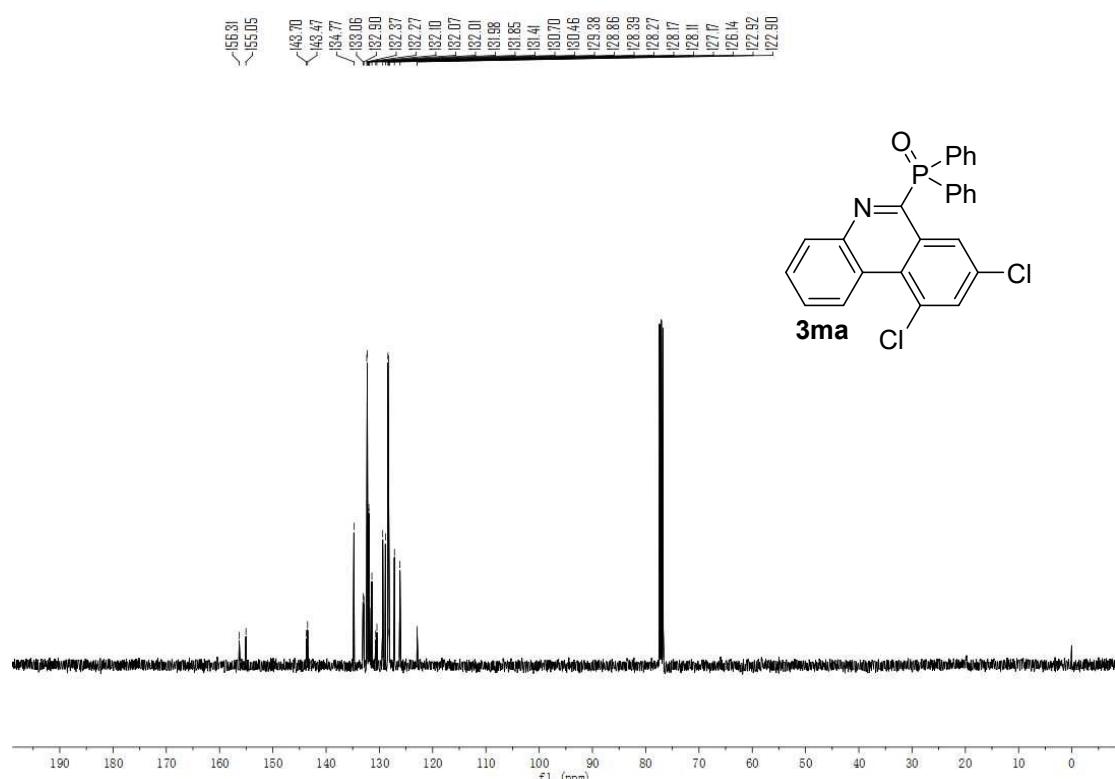
^{31}P NMR spectrum of 3la



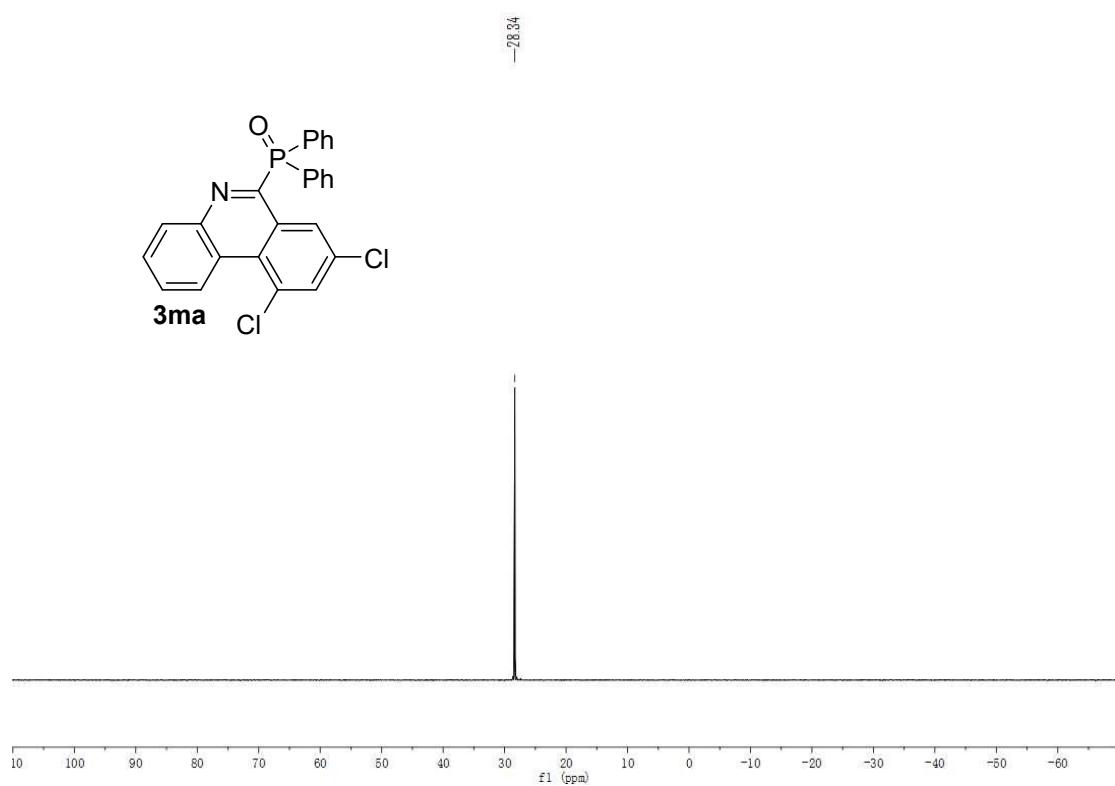
^1H NMR spectrum of 3ma



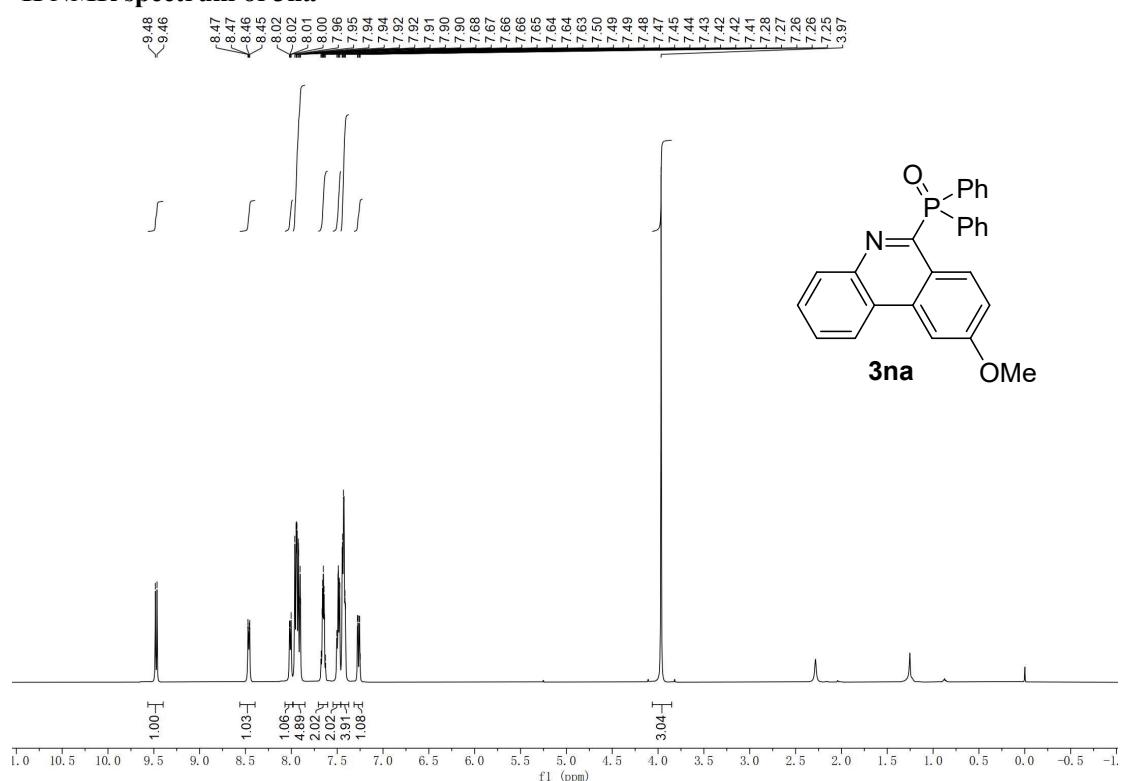
¹³C NMR spectrum of 3ma



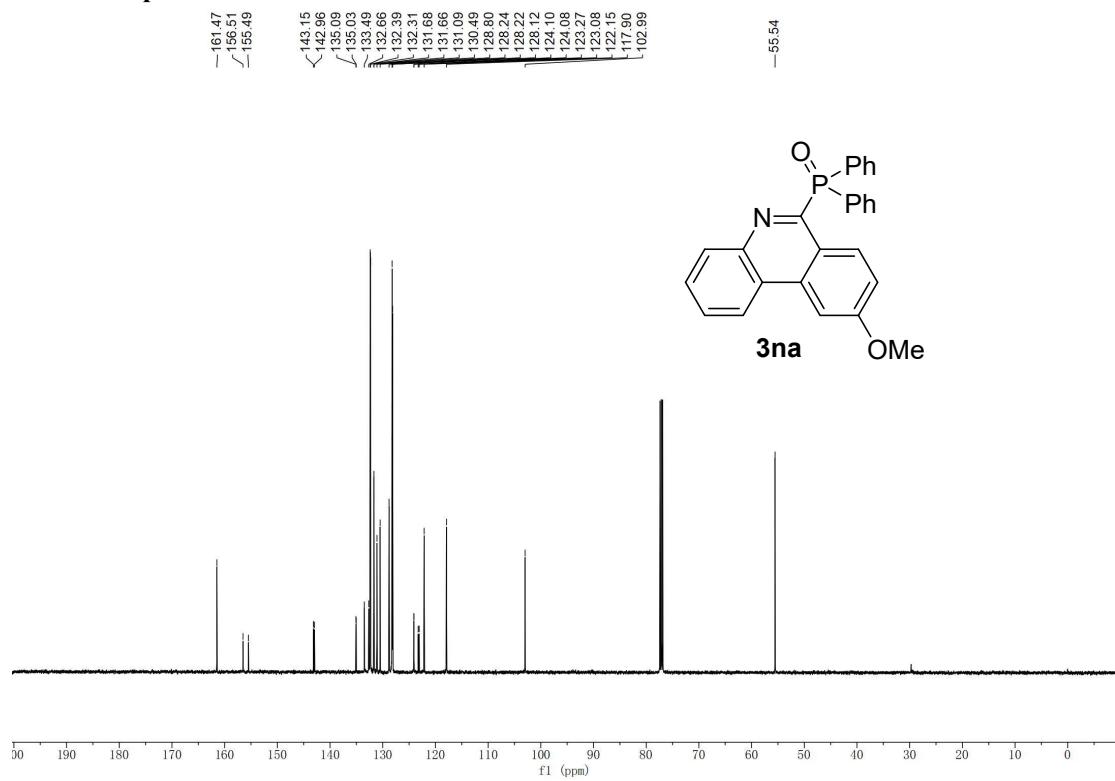
³¹P NMR spectrum of 3ma



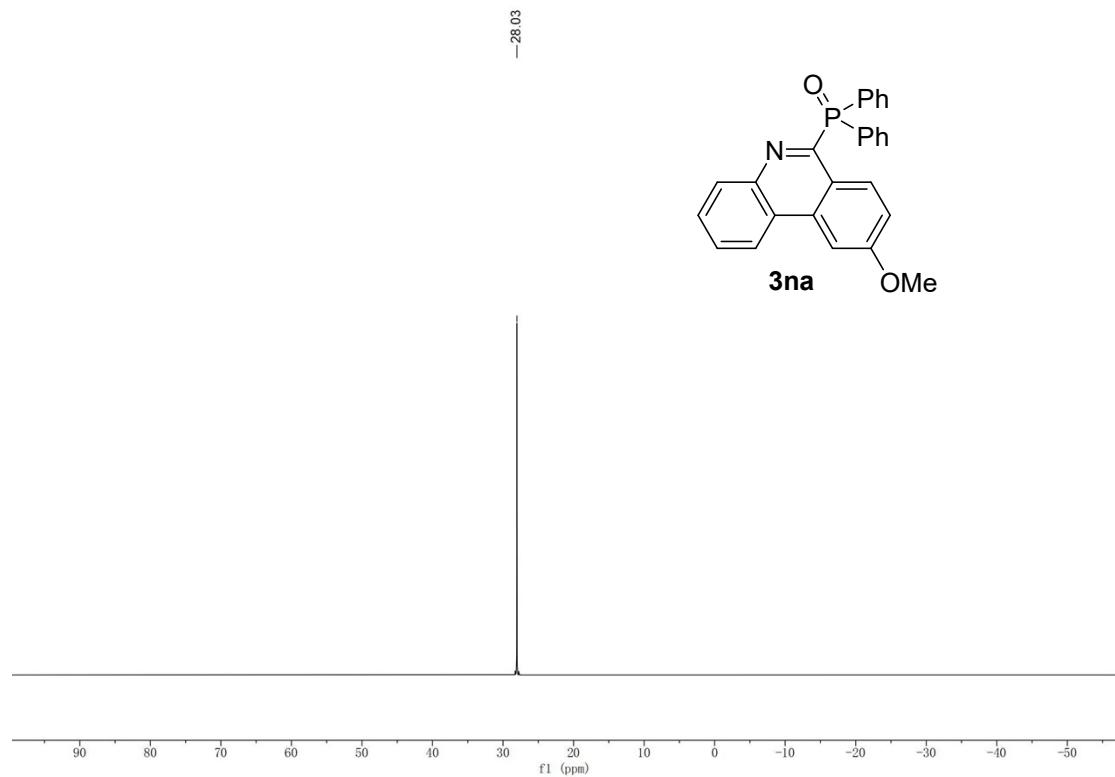
¹H NMR spectrum of 3na



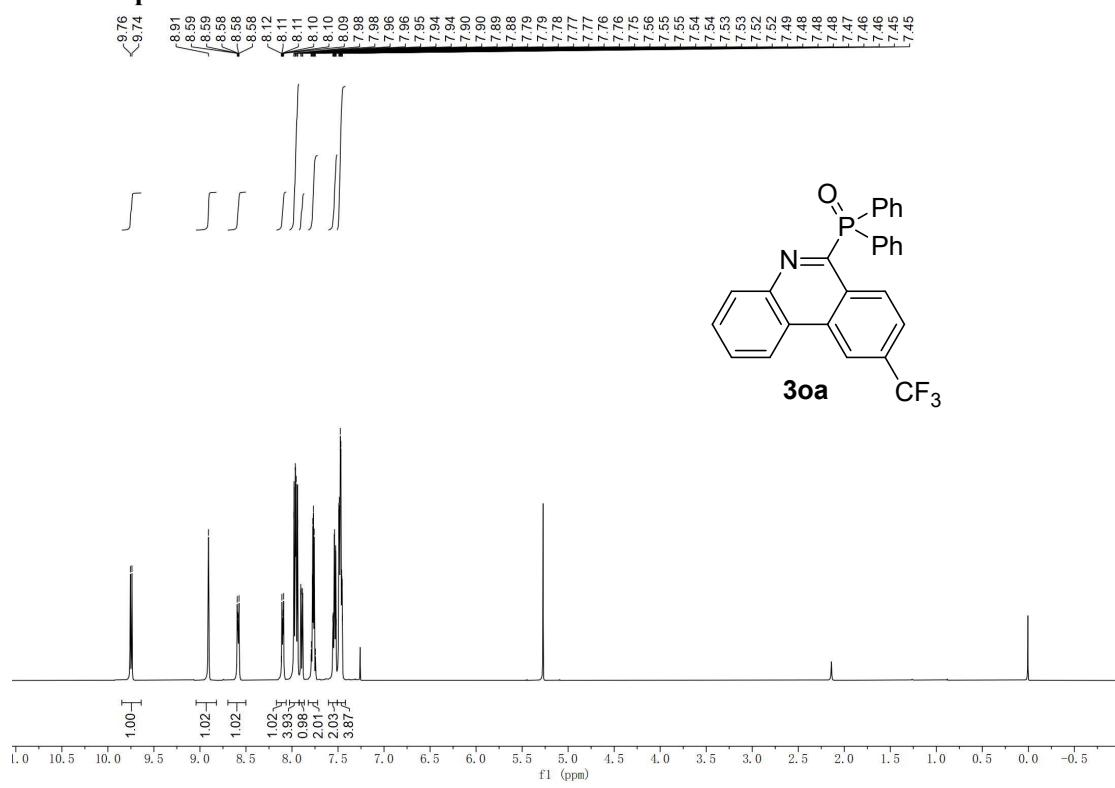
¹³C NMR spectrum of 3na



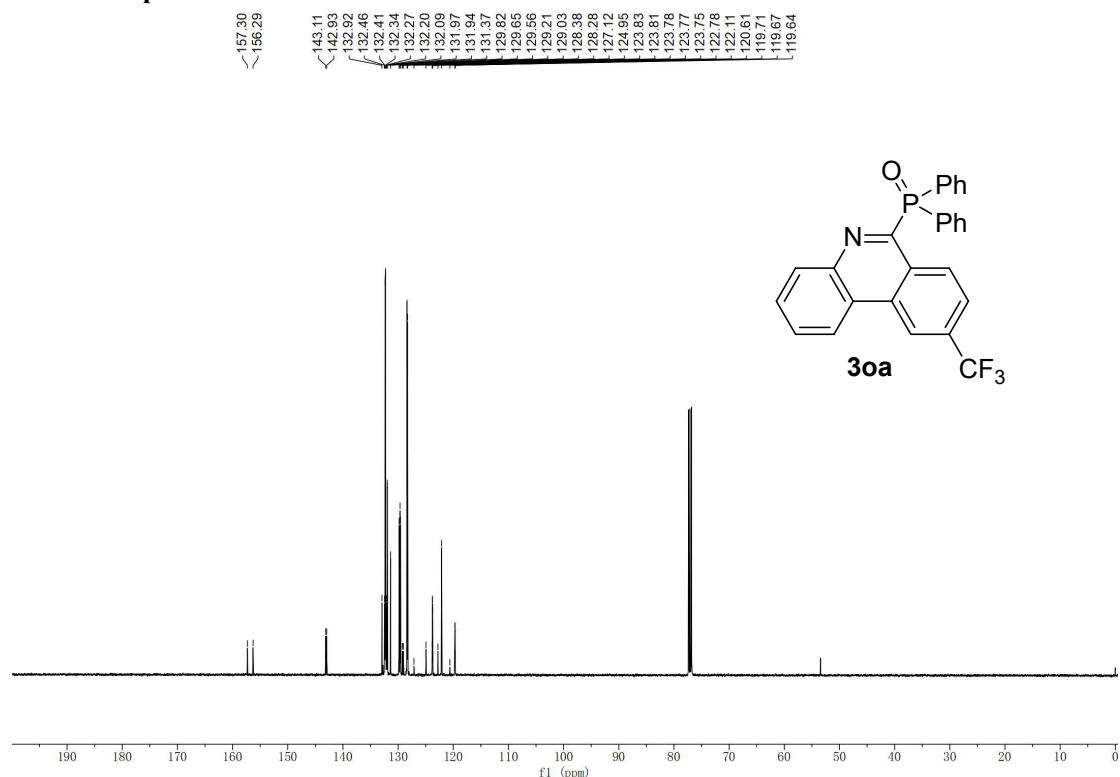
^{31}P NMR spectrum of 3na



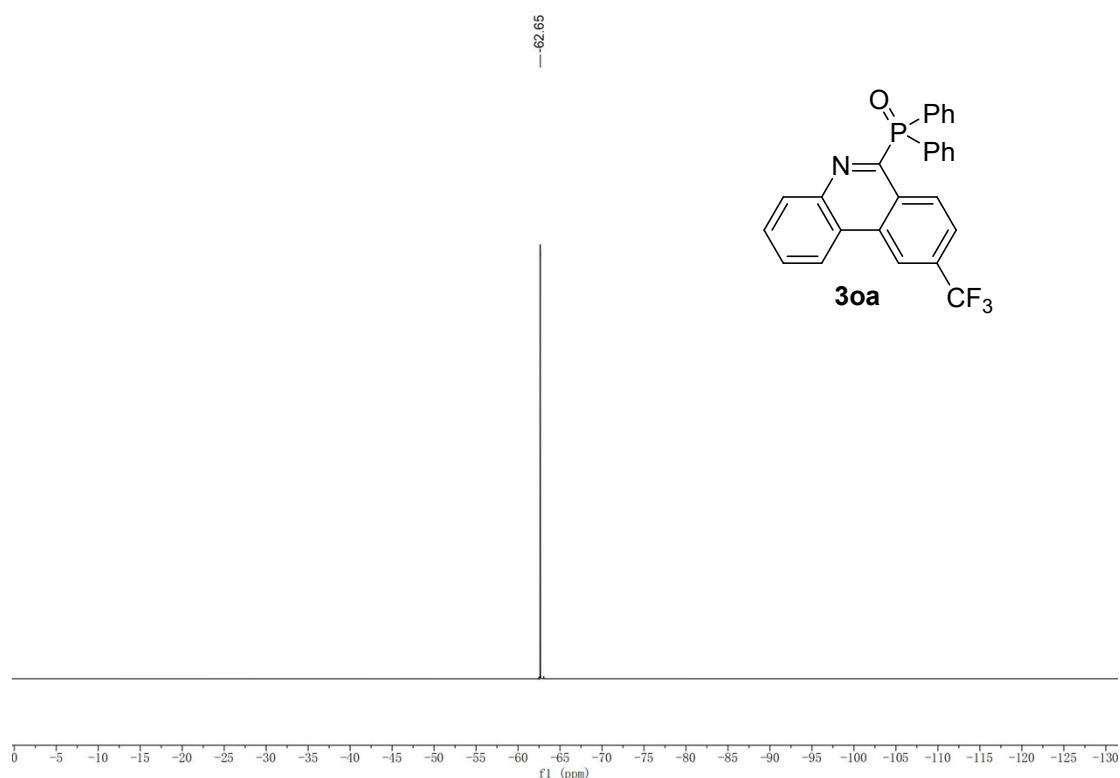
^1H NMR spectrum of 3oa



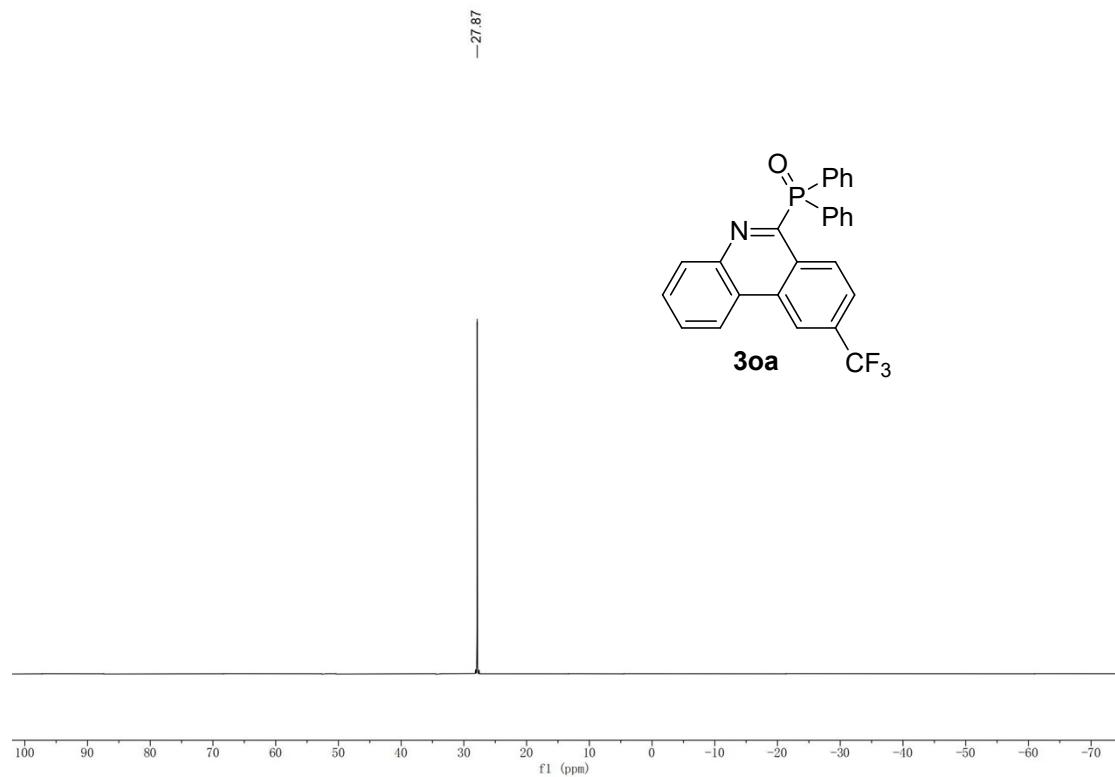
¹³C NMR spectrum of 3oa



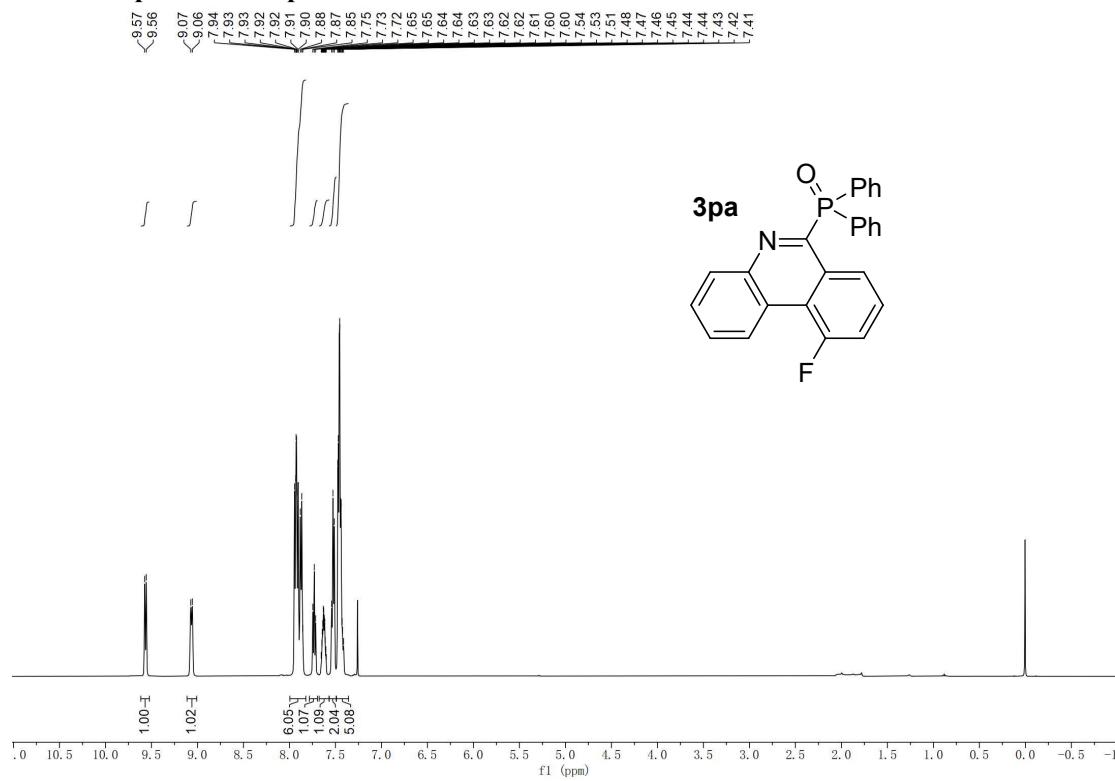
¹⁹F NMR spectrum of 3oa



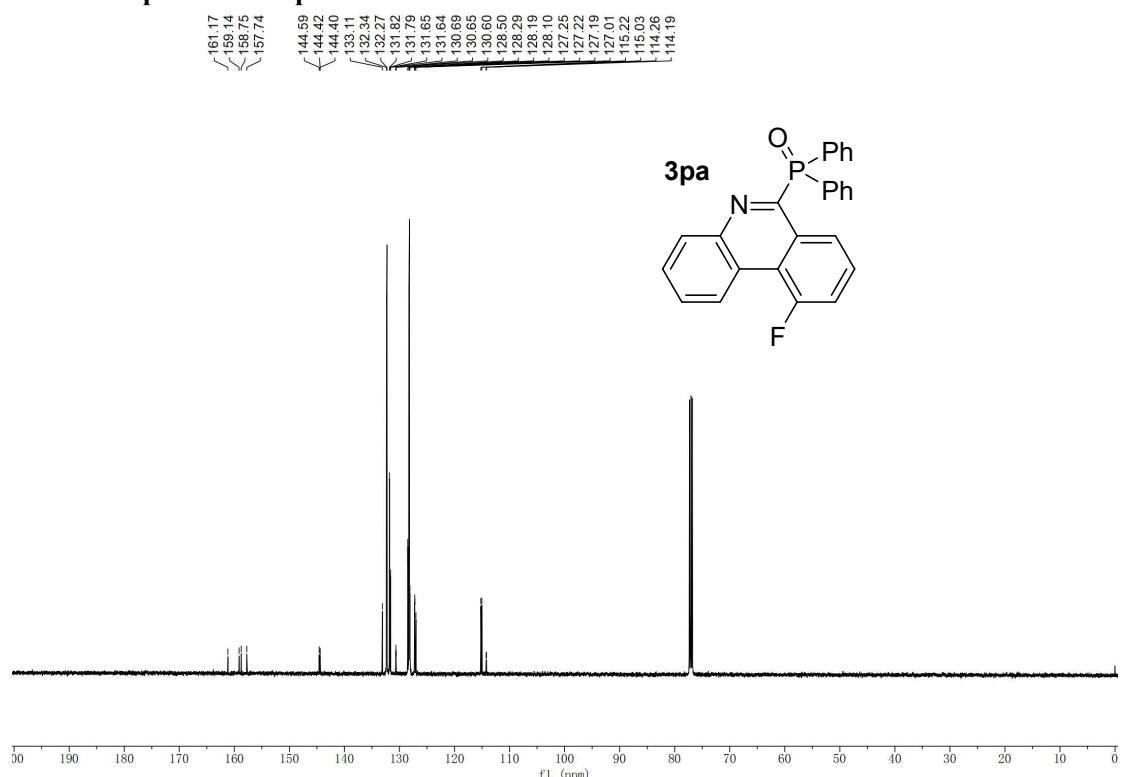
^{31}P NMR spectrum of 3oa



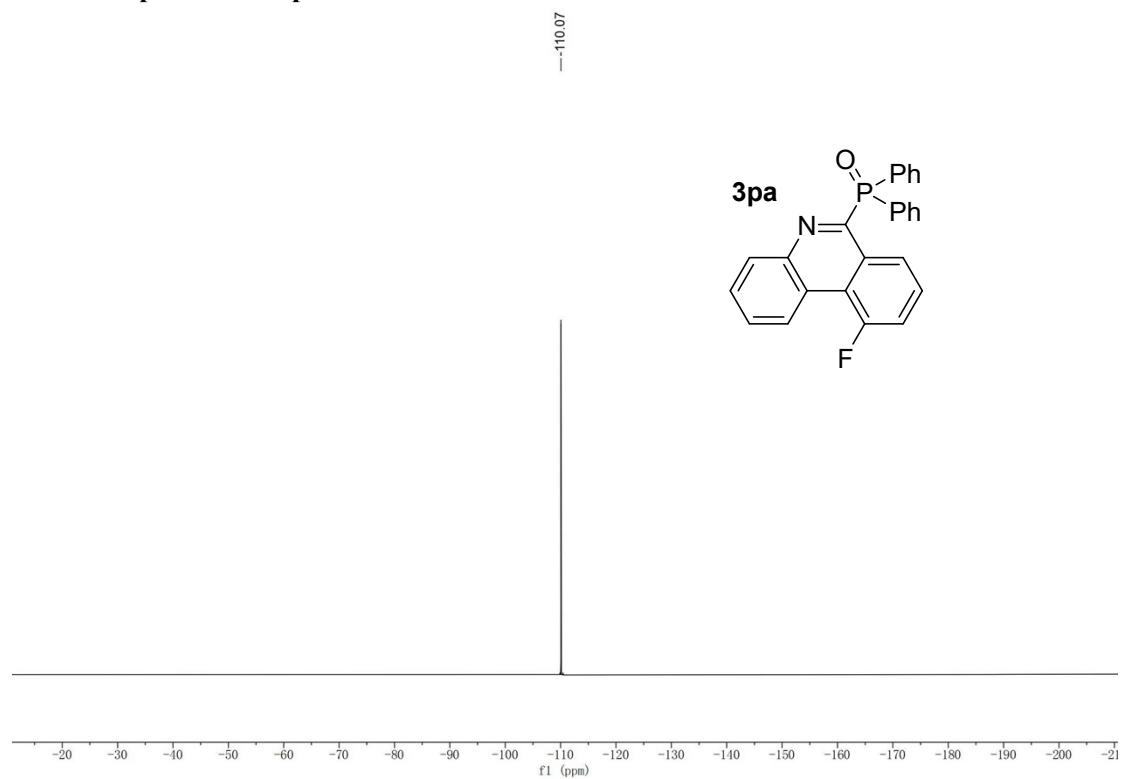
^1H NMR spectrum of 3pa



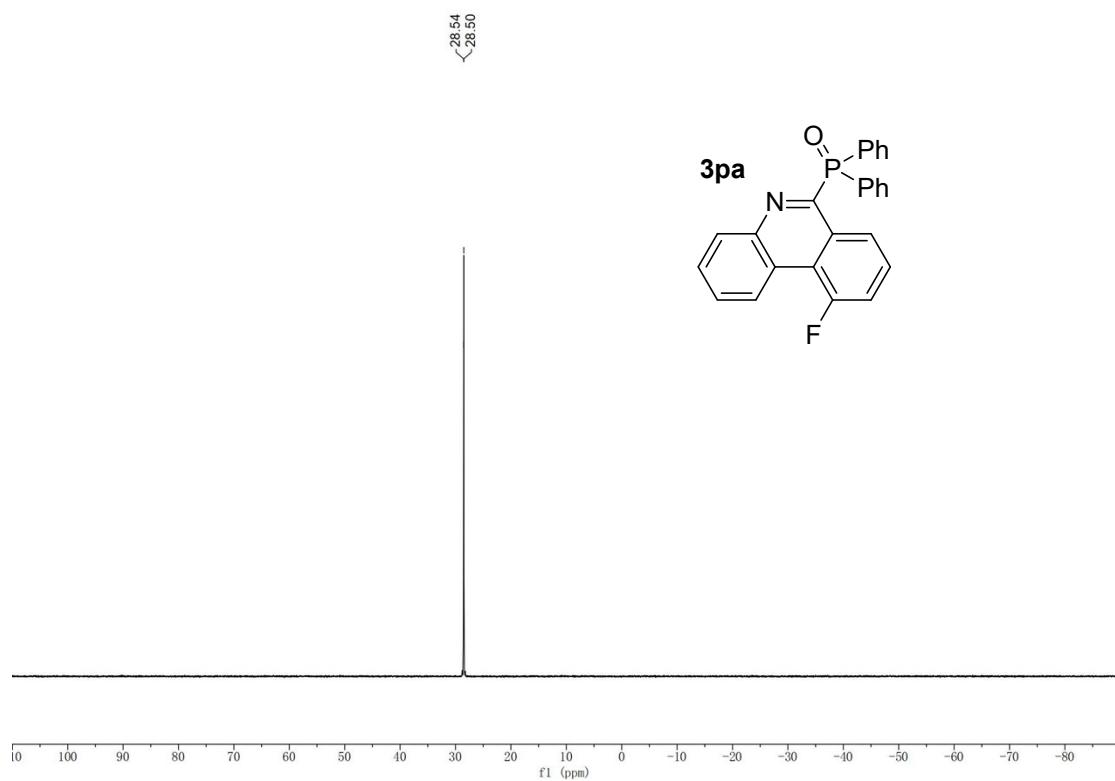
¹³C NMR spectrum of 3pa



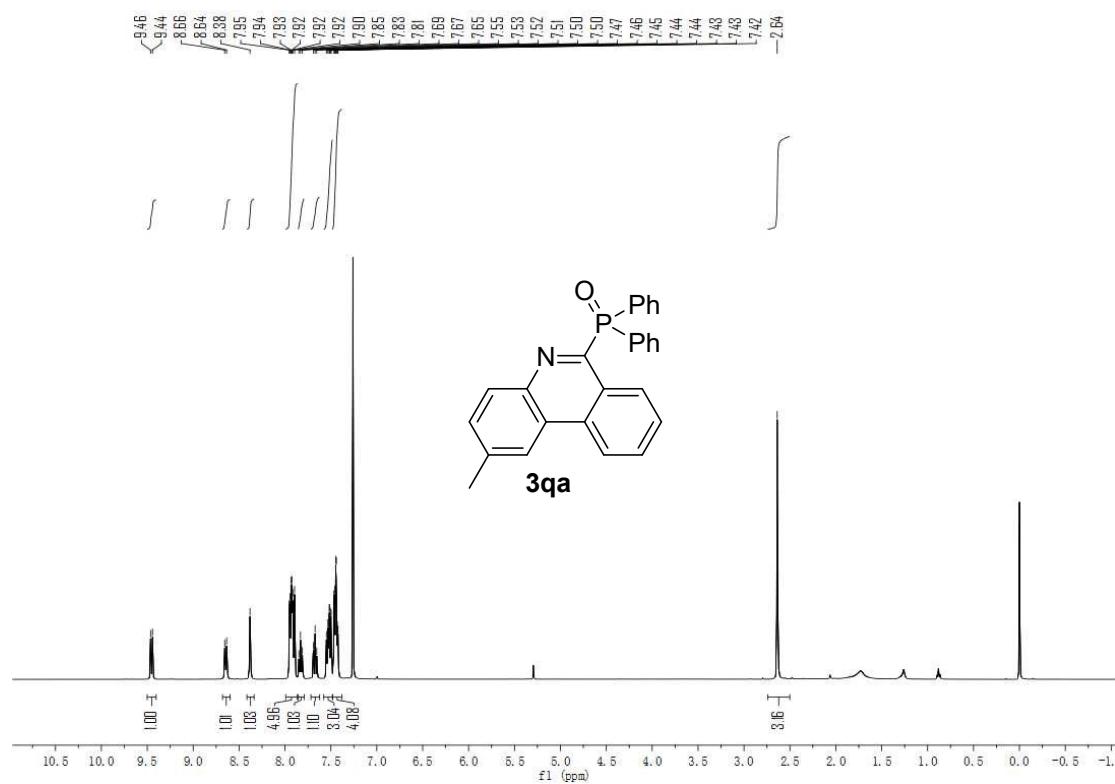
¹⁹F NMR spectrum of 3pa



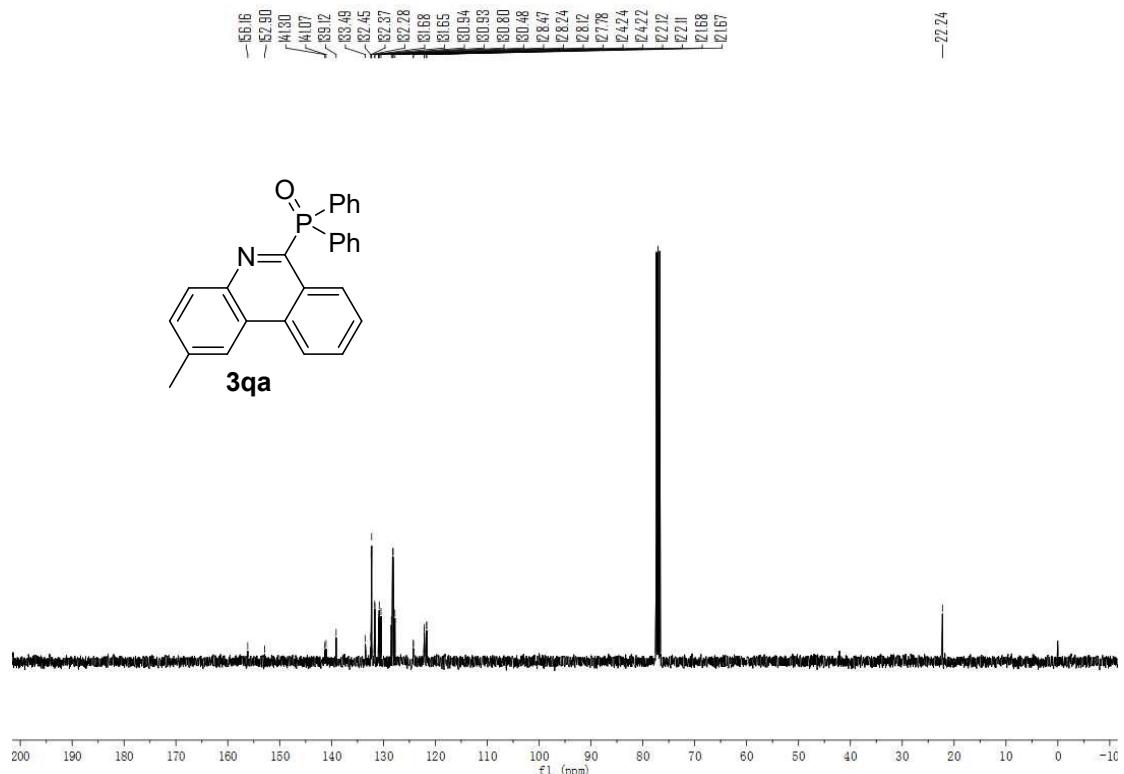
¹³P NMR spectrum of 3pa



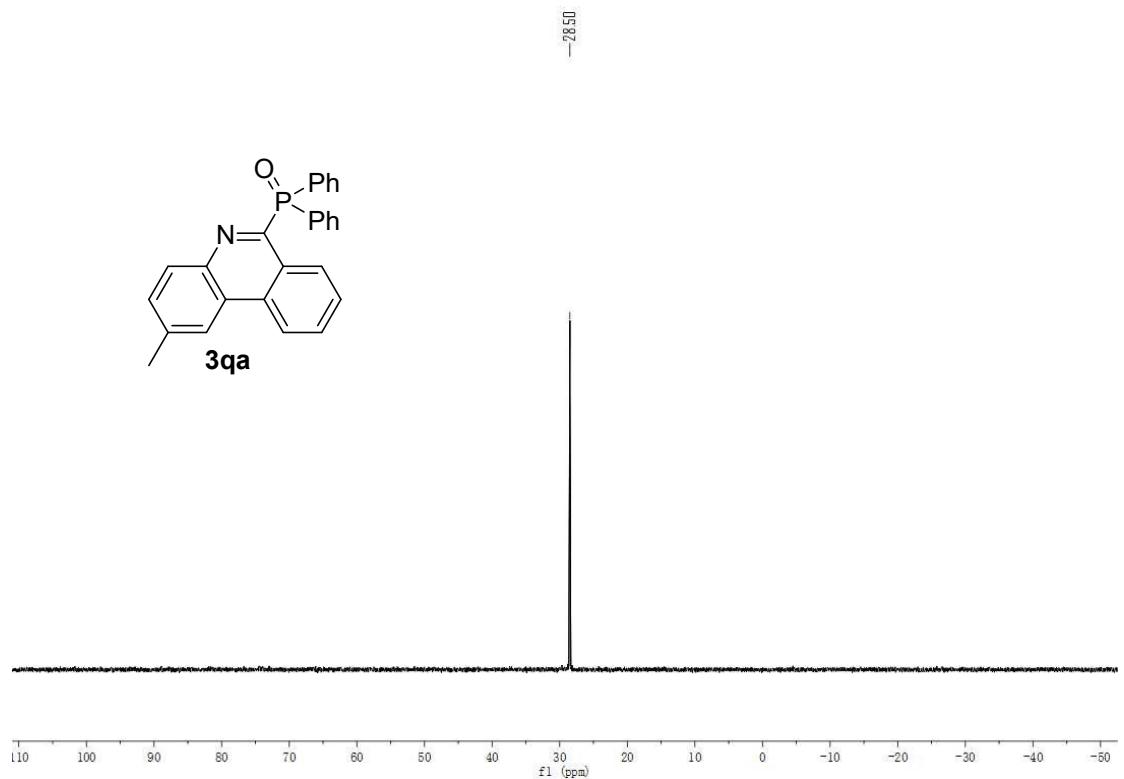
¹H NMR spectrum of 3qa



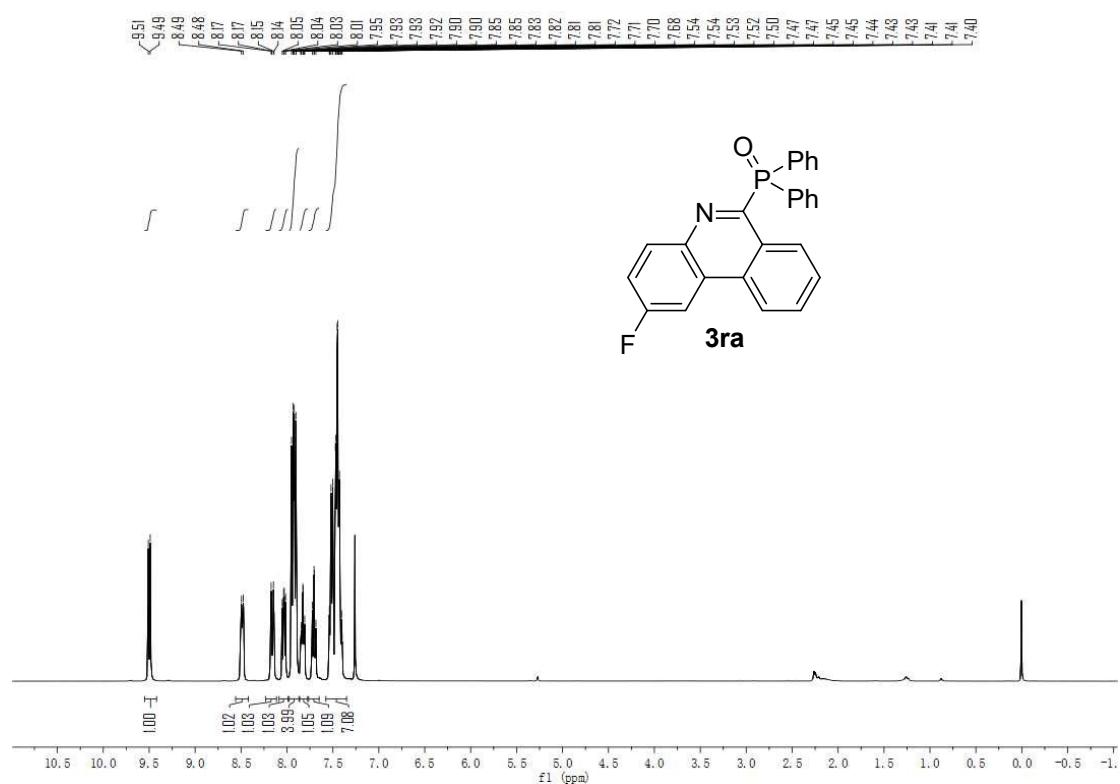
^{13}C NMR spectrum of 3qa



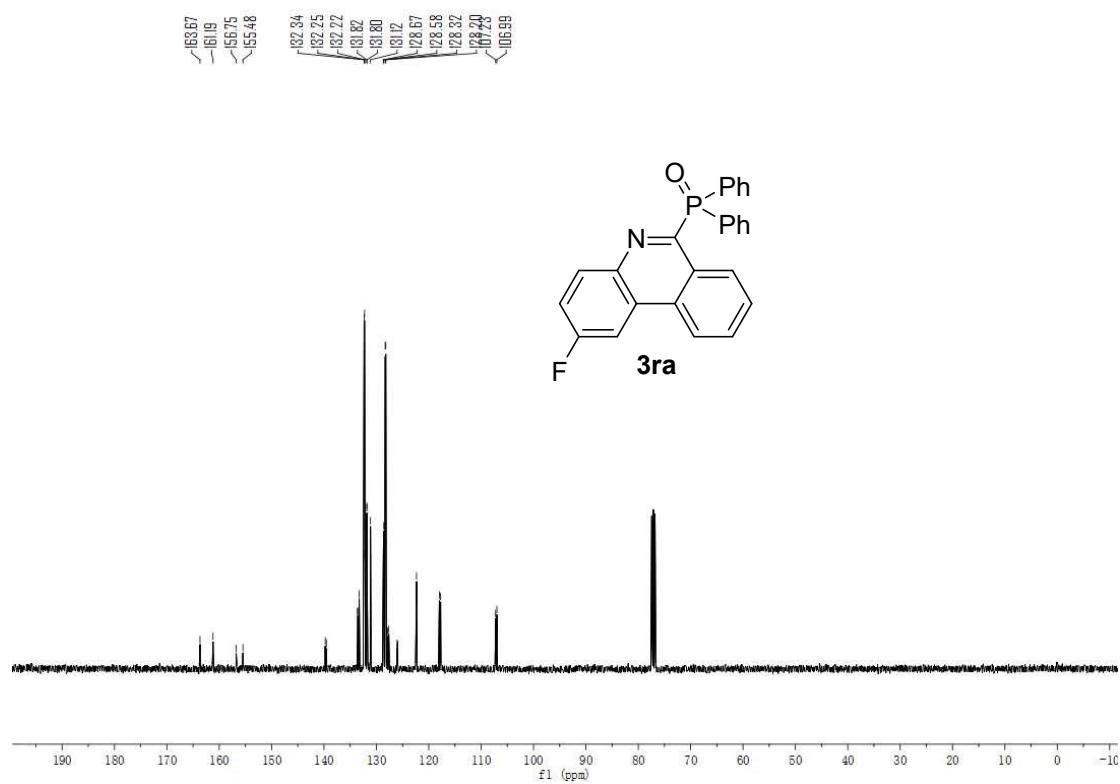
^{31}P NMR spectrum of 3qa



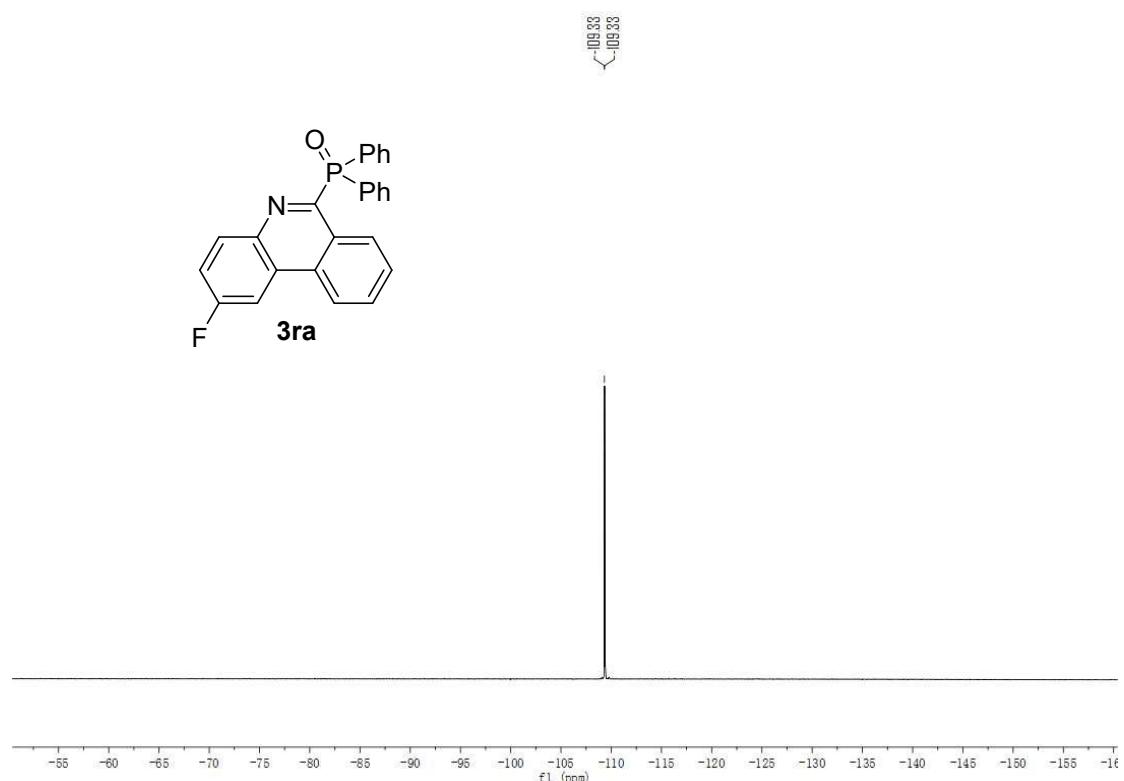
¹H NMR spectrum of 3ra



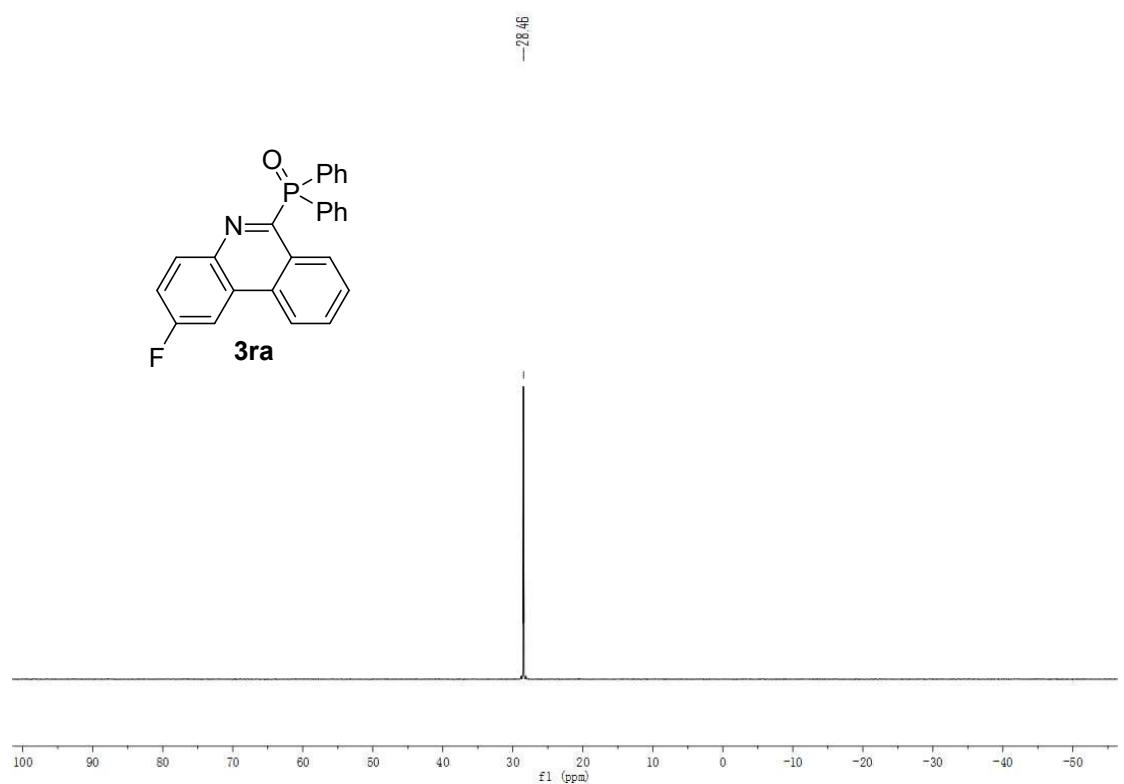
¹³C NMR spectrum of 3ra



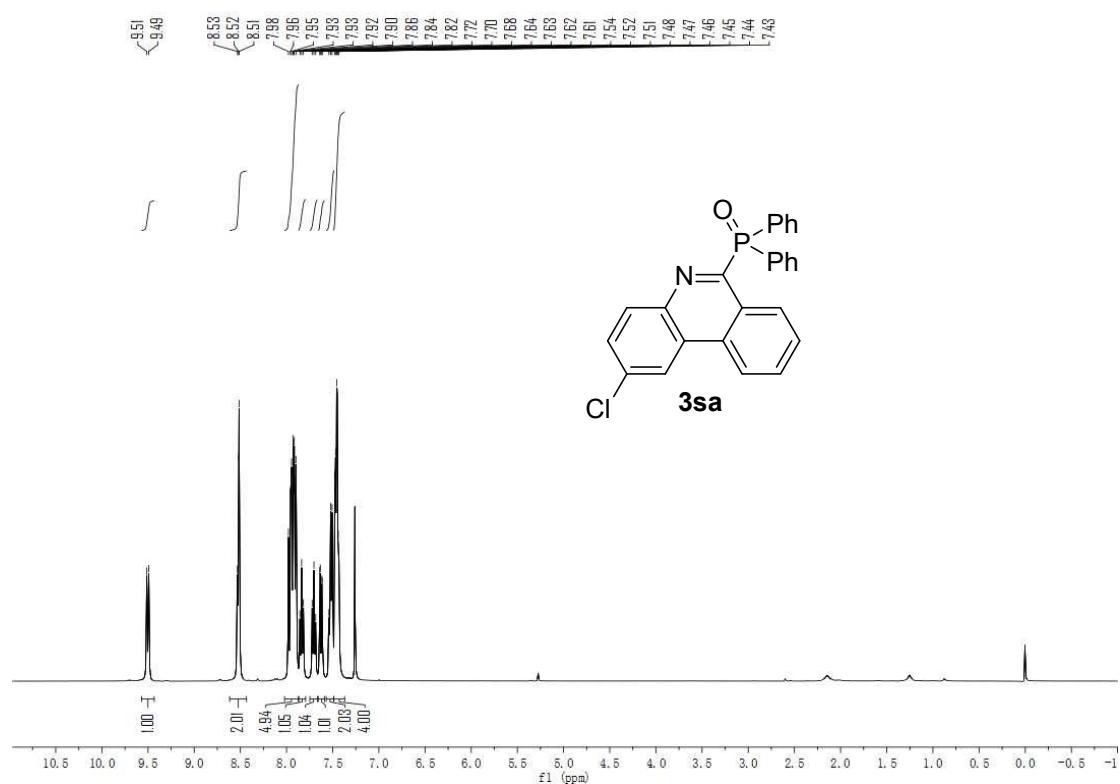
¹⁹F NMR spectrum of 3ra



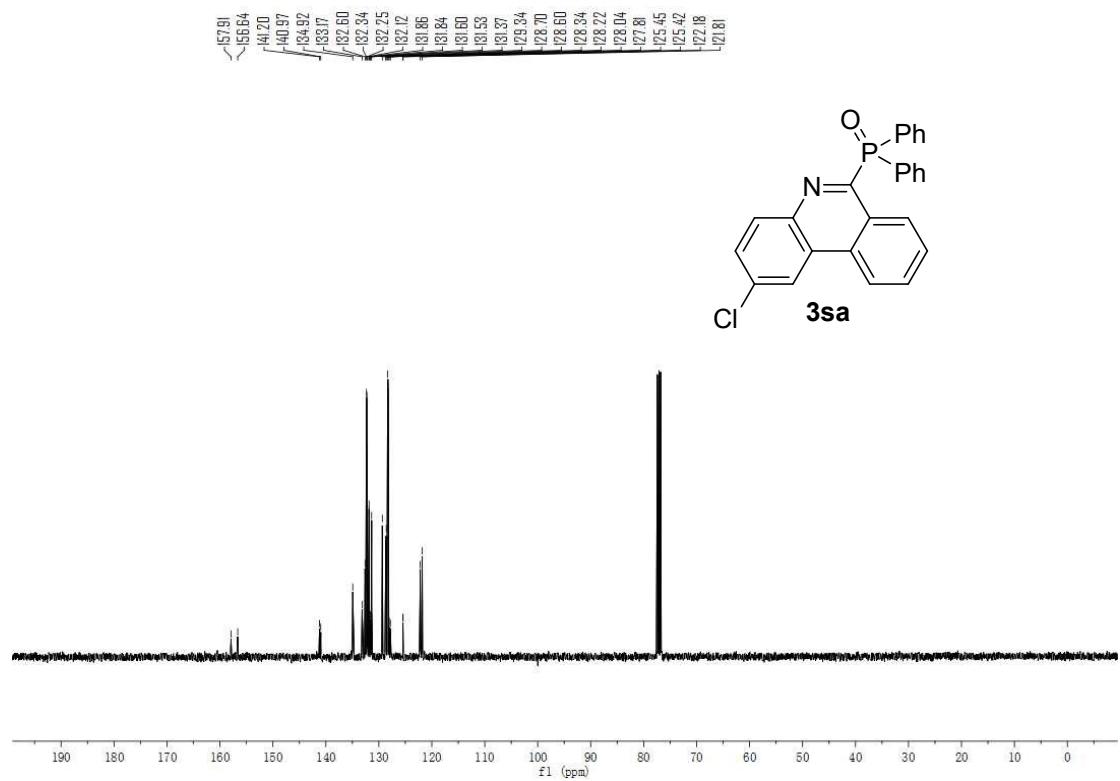
³¹P NMR spectrum of 3ra



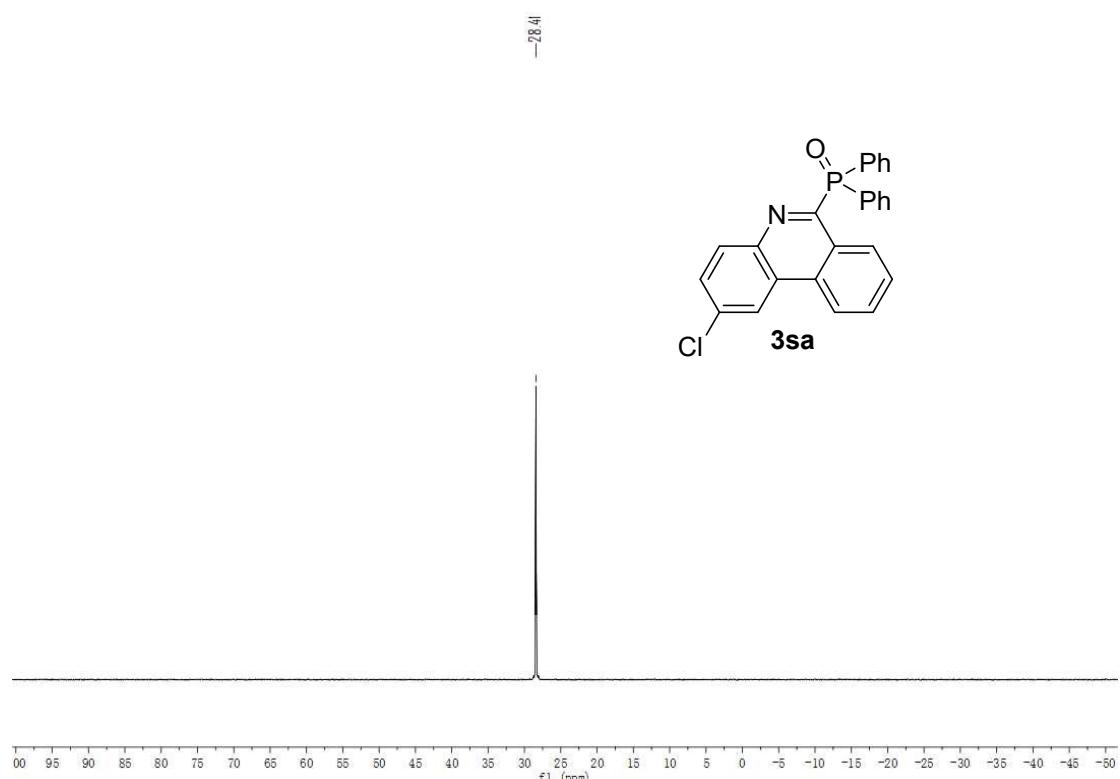
¹H NMR spectrum of 3sa



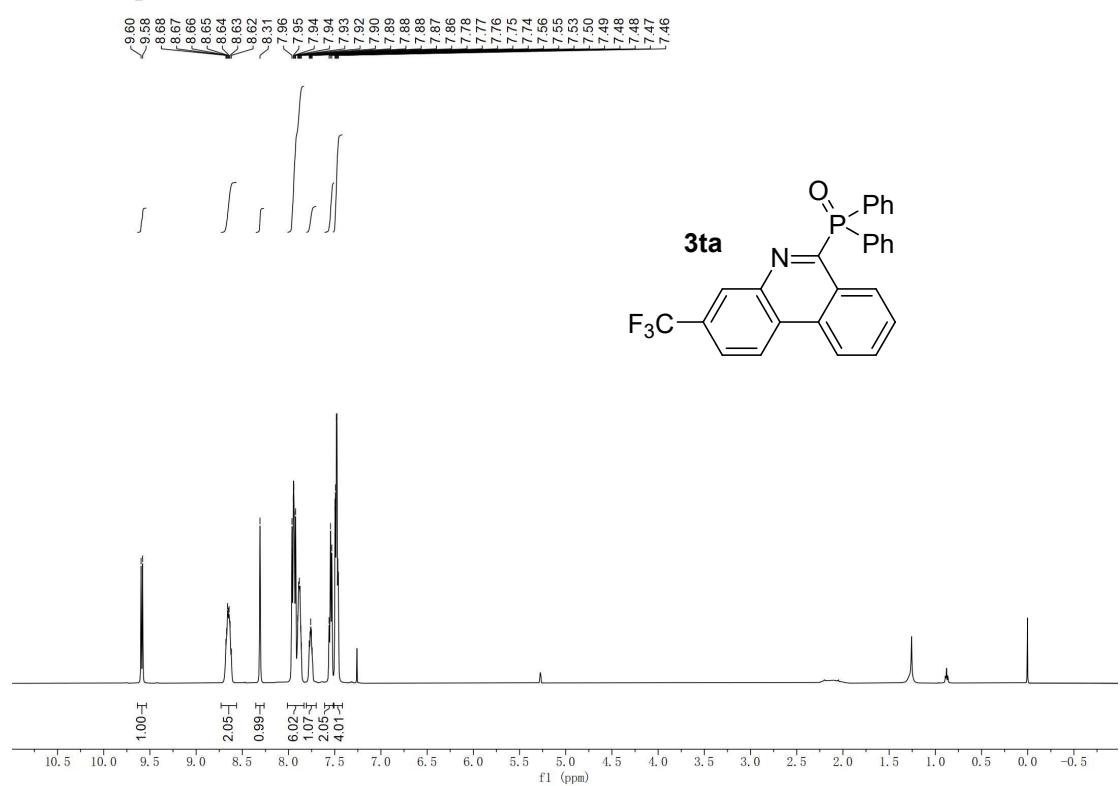
¹³C NMR spectrum of 3sa



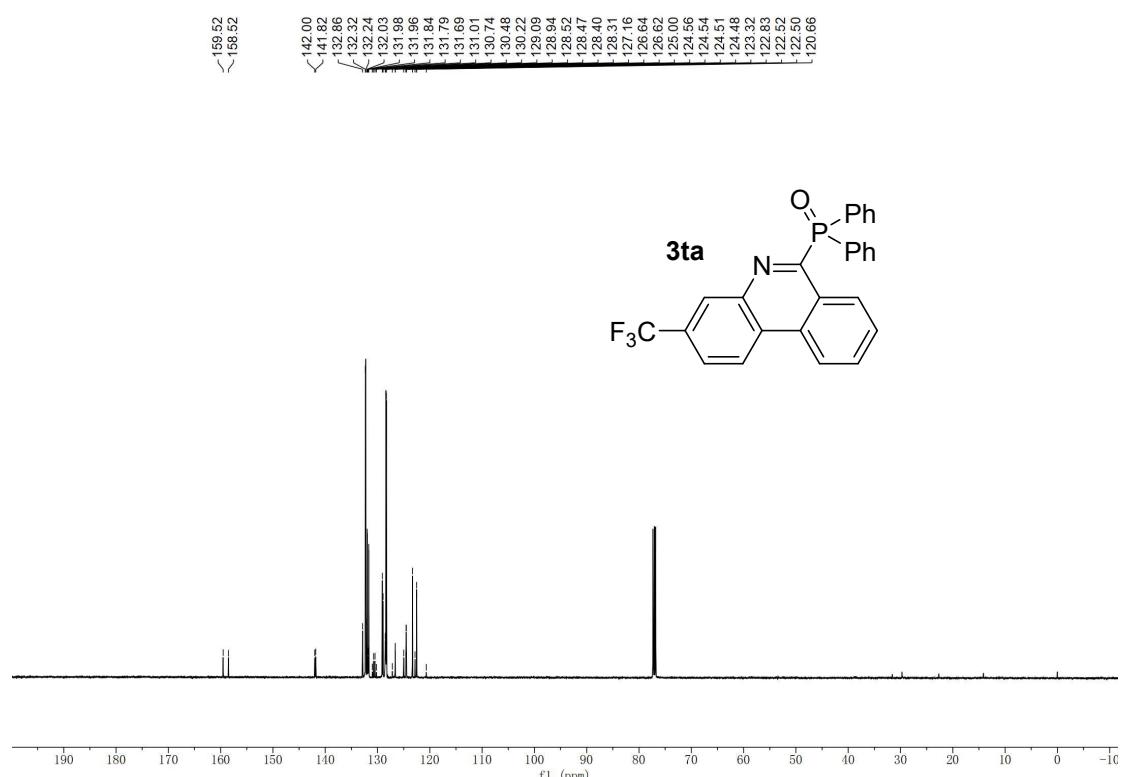
³¹P NMR spectrum of 3sa



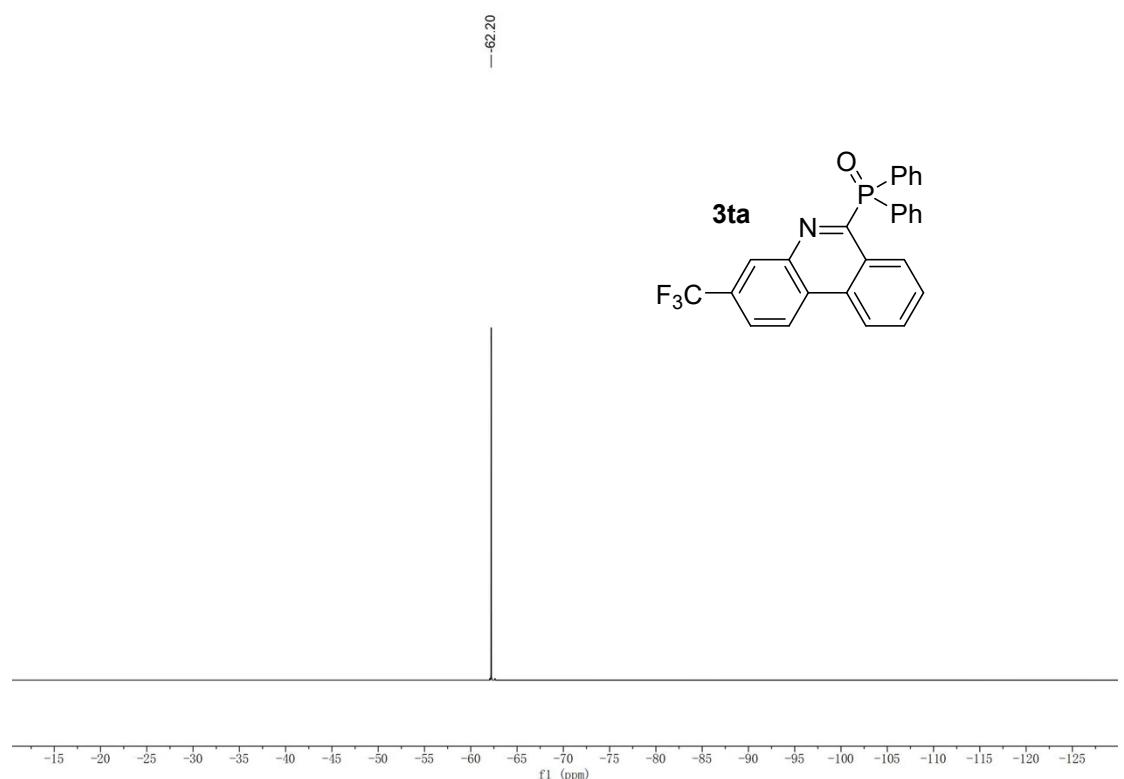
¹H NMR spectrum of 3ta



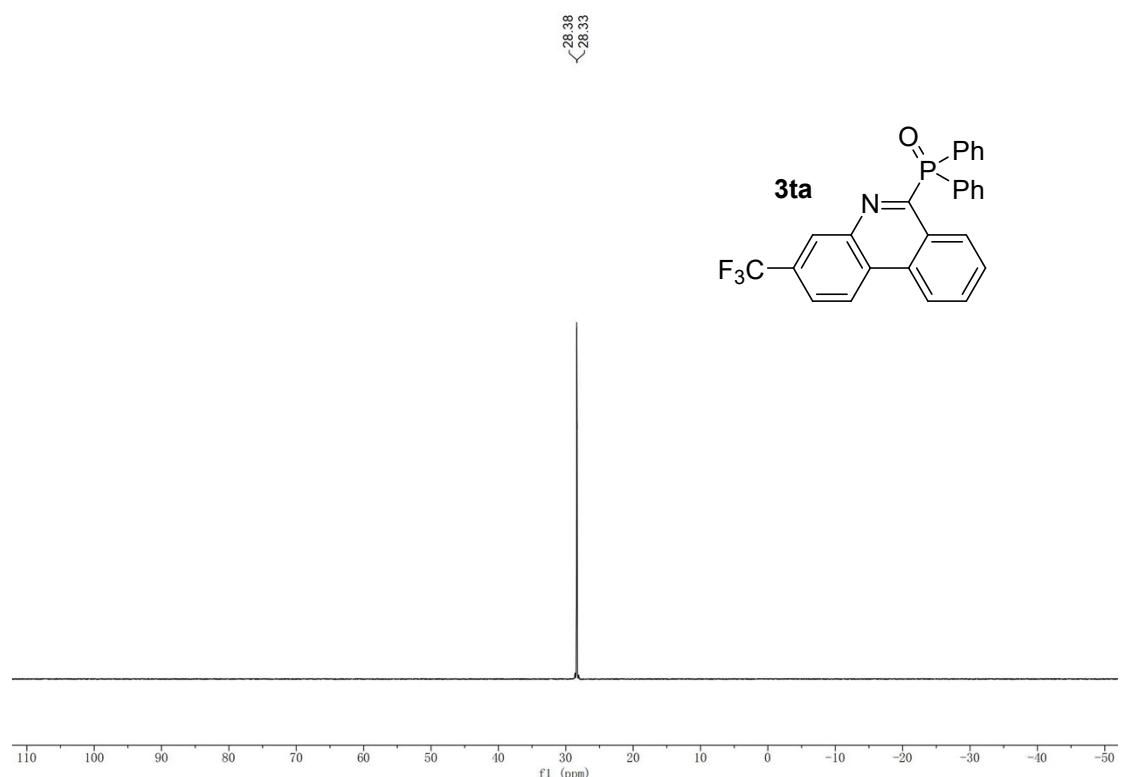
¹³C NMR spectrum of 3ta



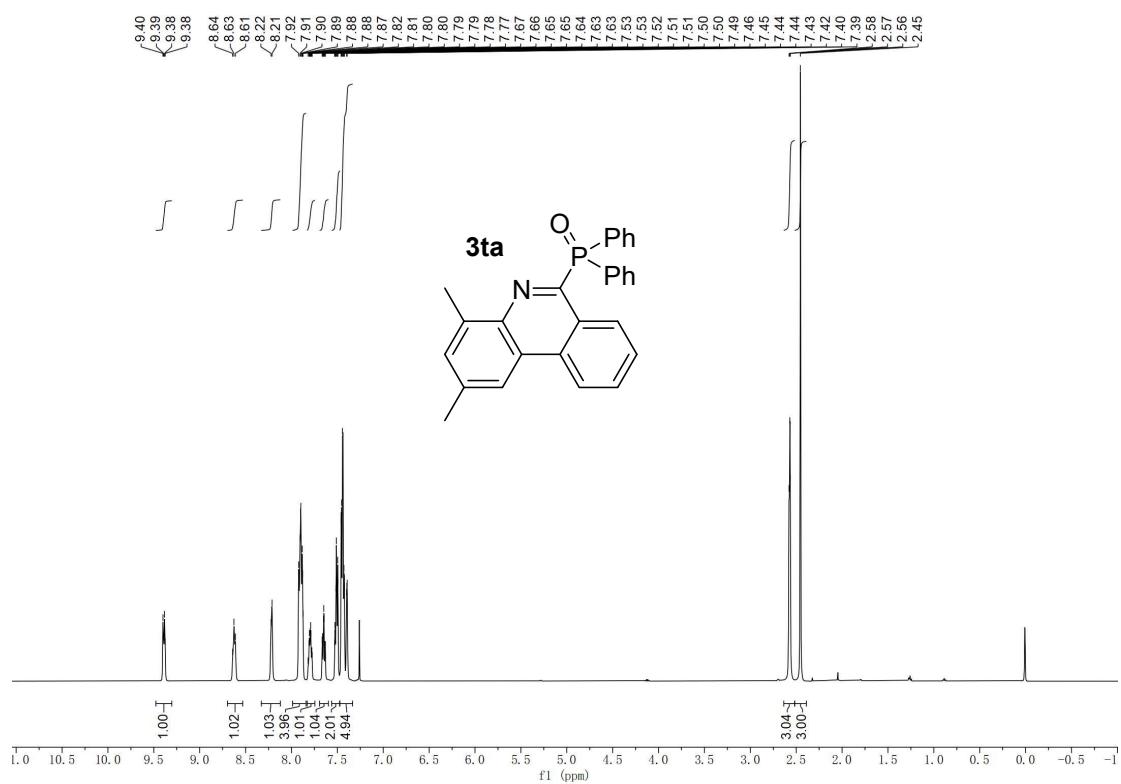
¹⁹F NMR spectrum of 3ta



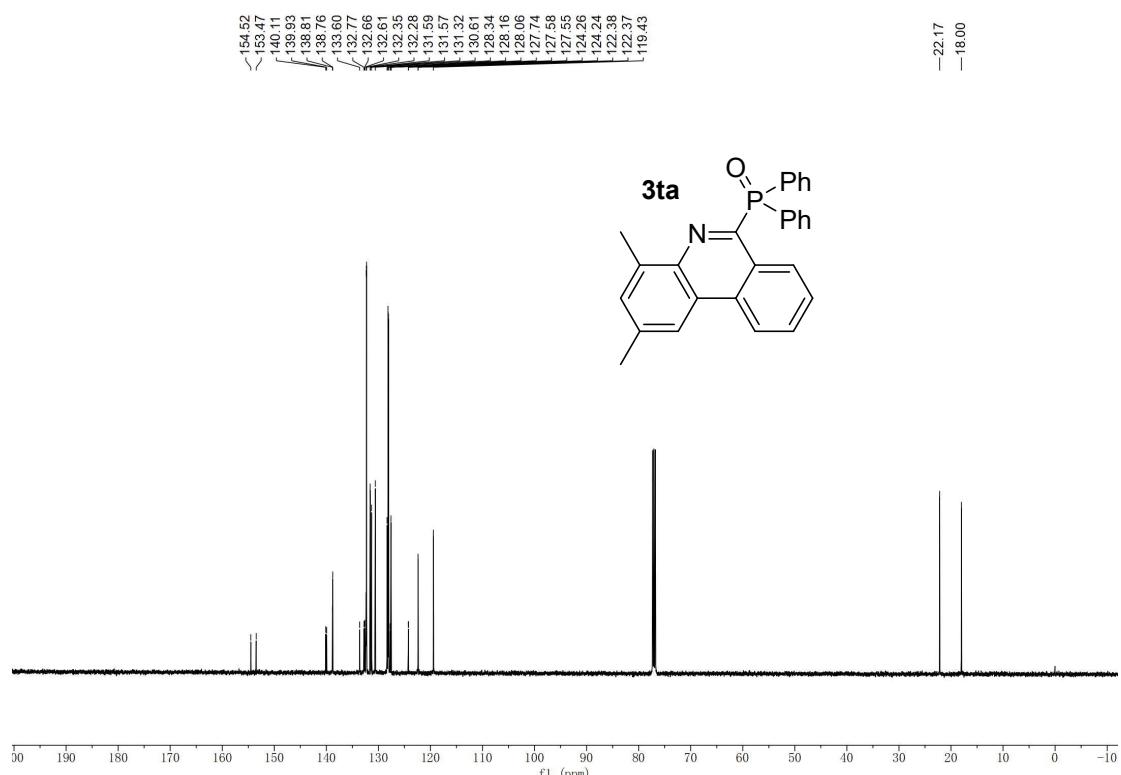
^{31}P NMR spectrum of 3ta



^1H NMR spectrum of 3ua



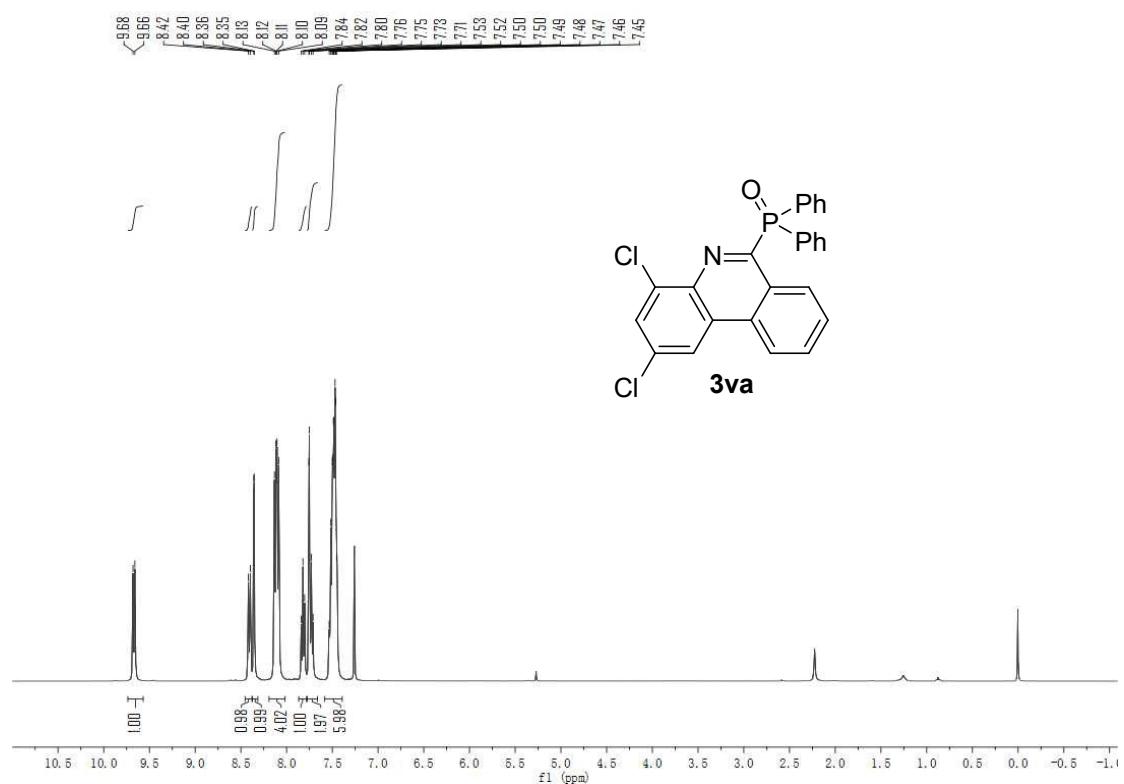
¹³C NMR spectrum of 3ua



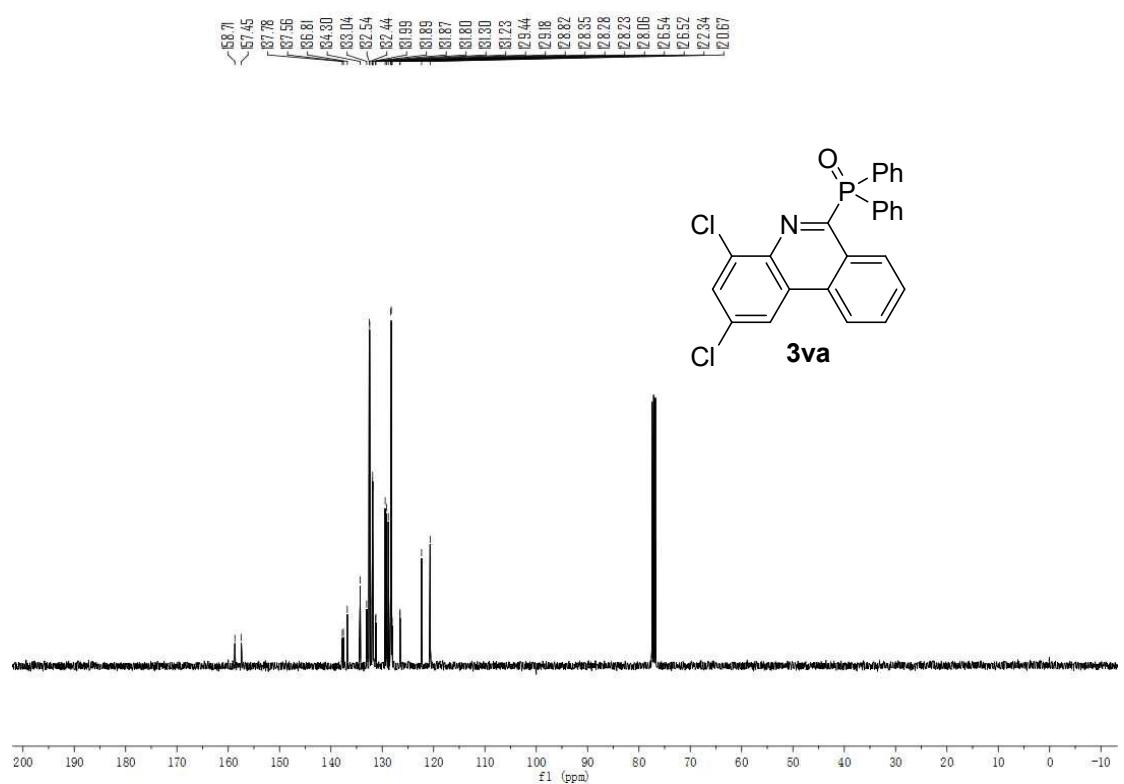
³¹P NMR spectrum of 3ua



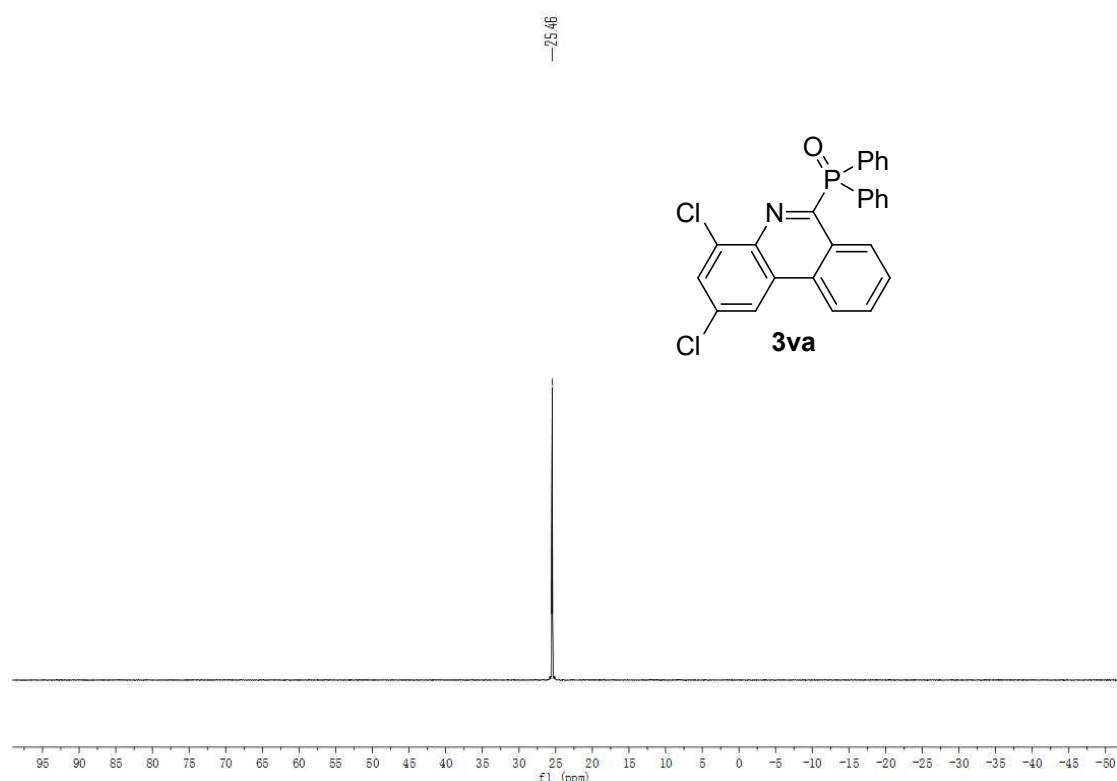
¹H NMR spectrum of 3va



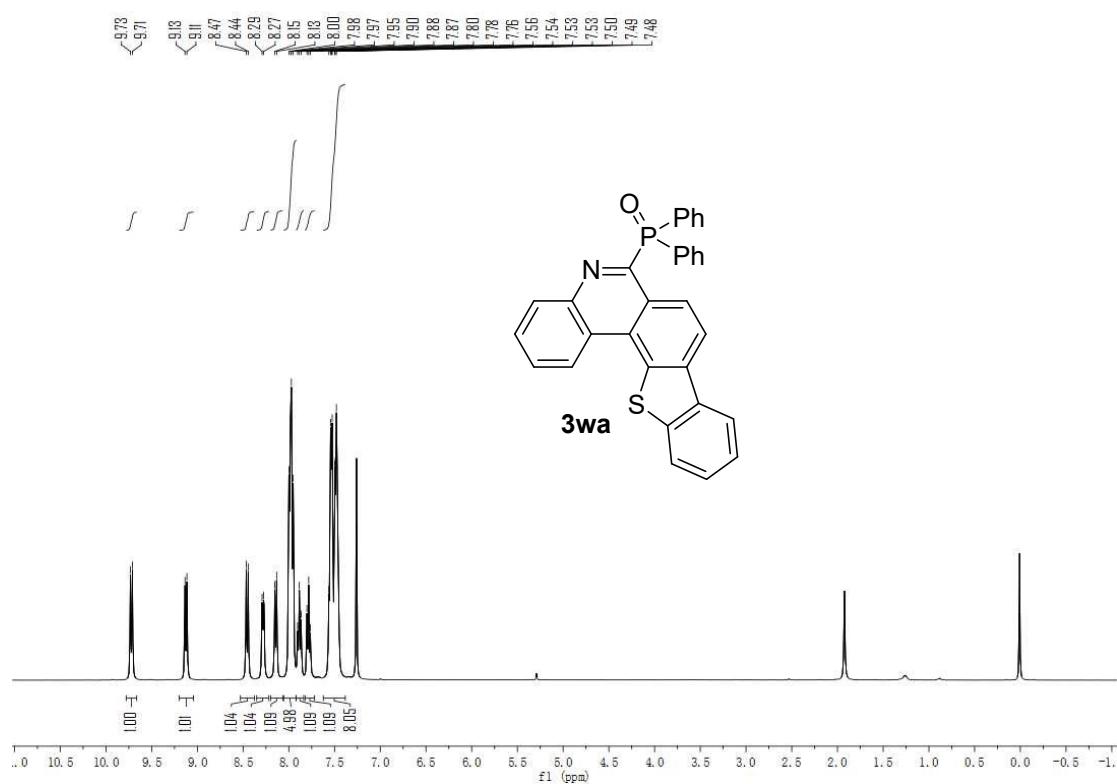
¹³C NMR spectrum of 3va



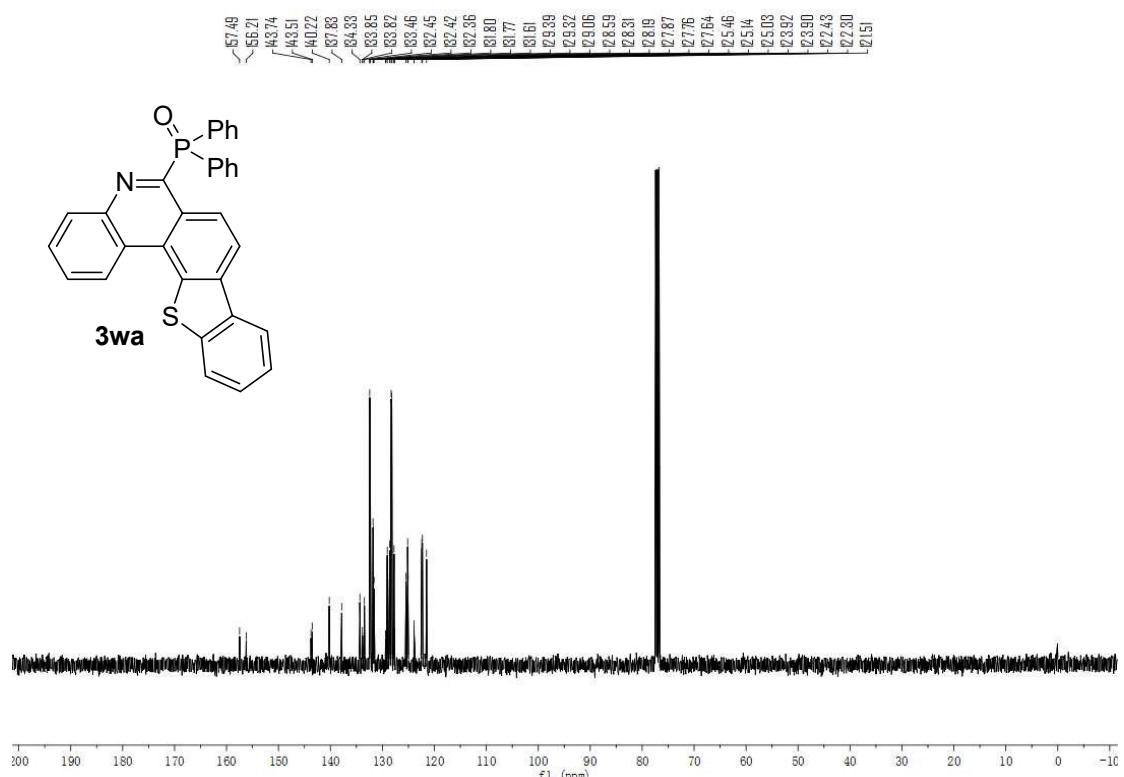
^{31}P NMR spectrum of 3va



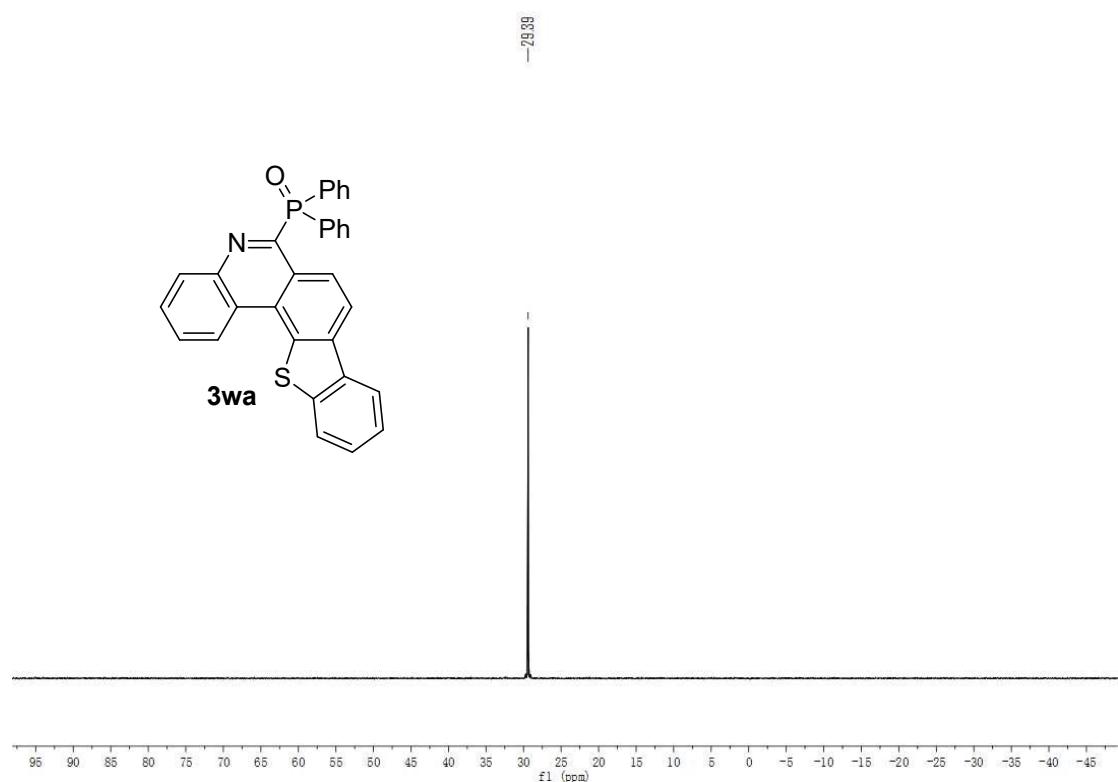
^1H NMR spectrum of 3wa



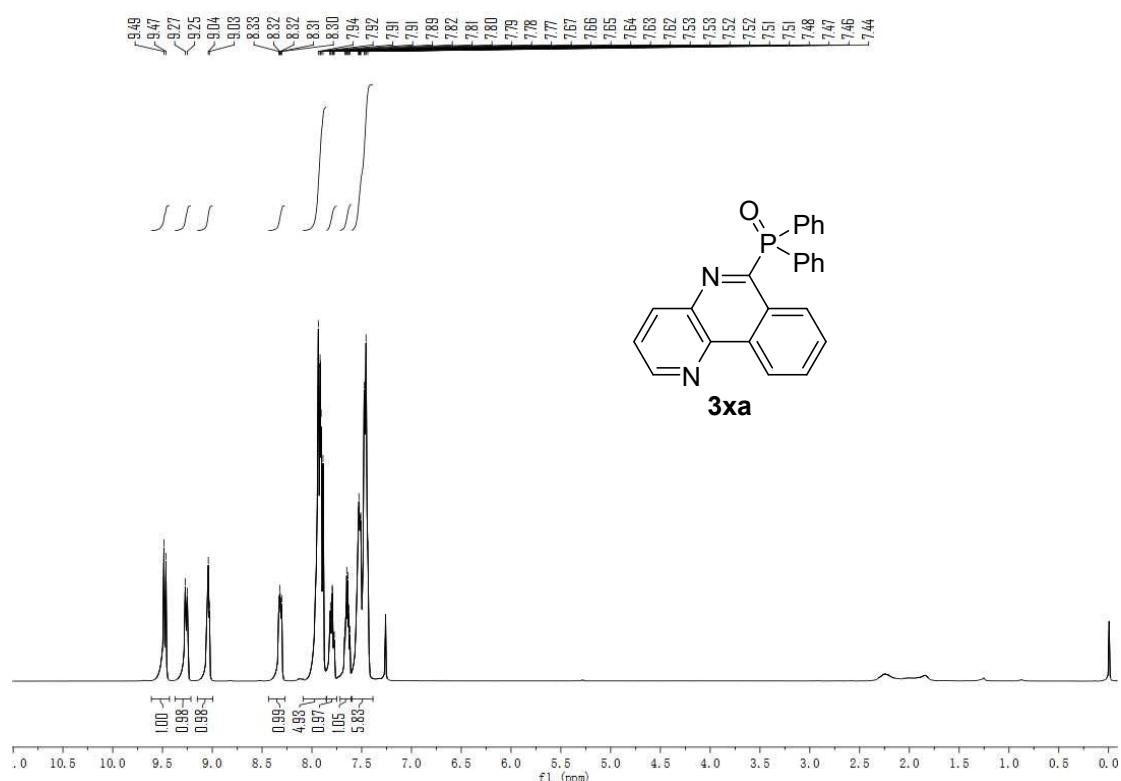
¹³C NMR spectrum of 3wa



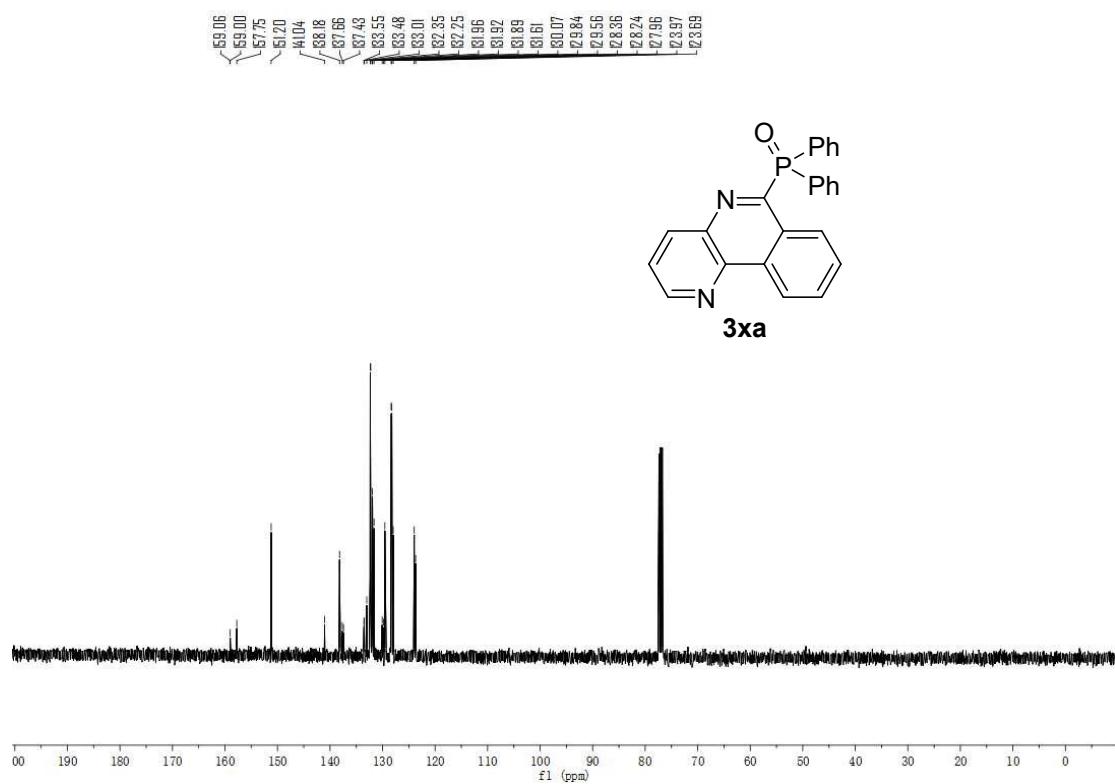
³¹P NMR spectrum of 3wa



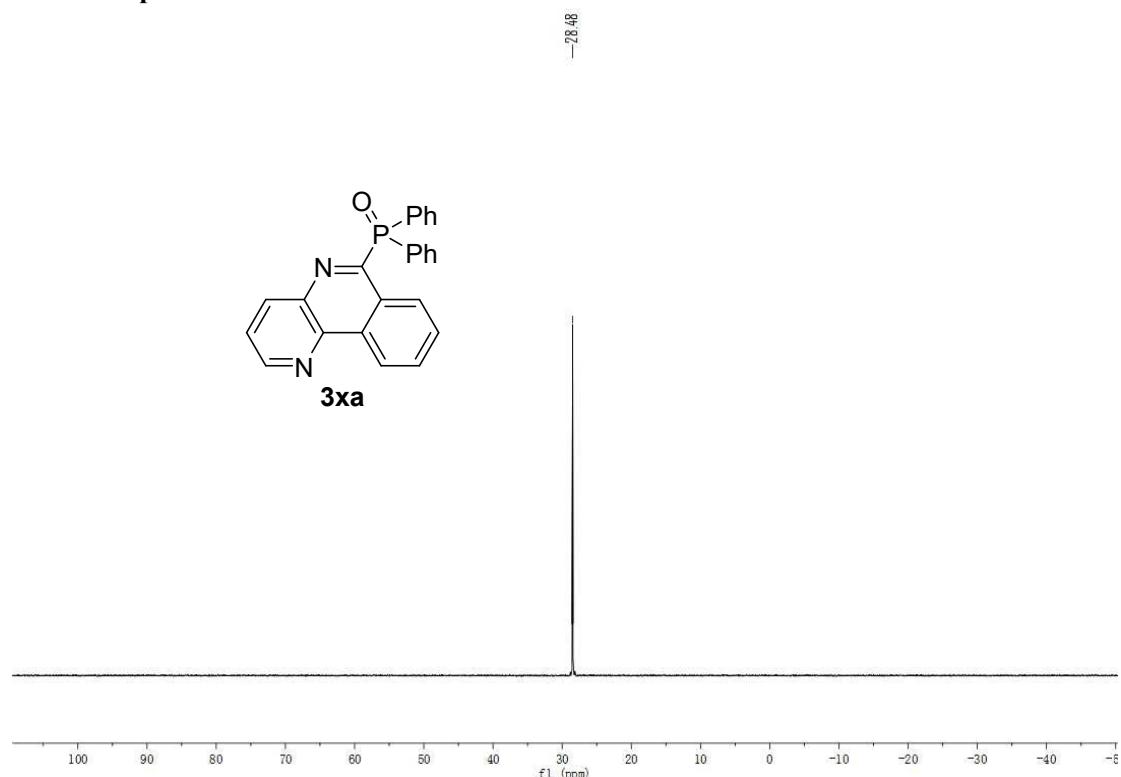
¹H NMR spectrum of 3ya



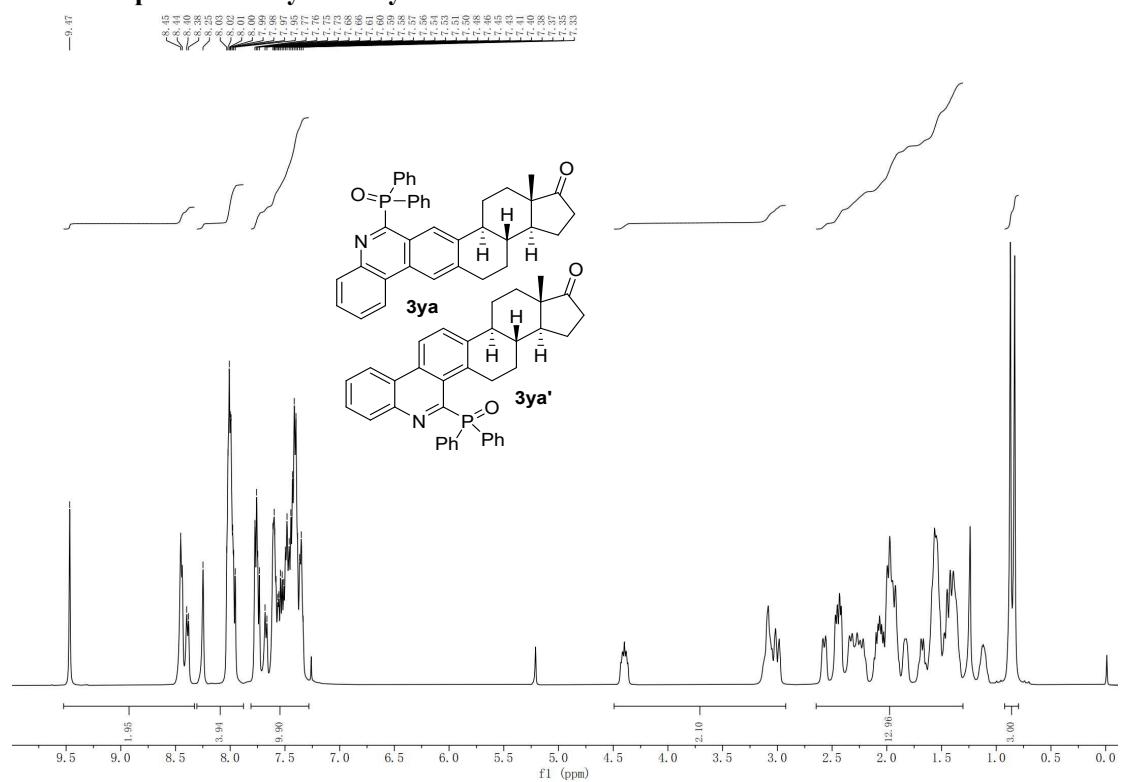
¹³C NMR spectrum of 3xa



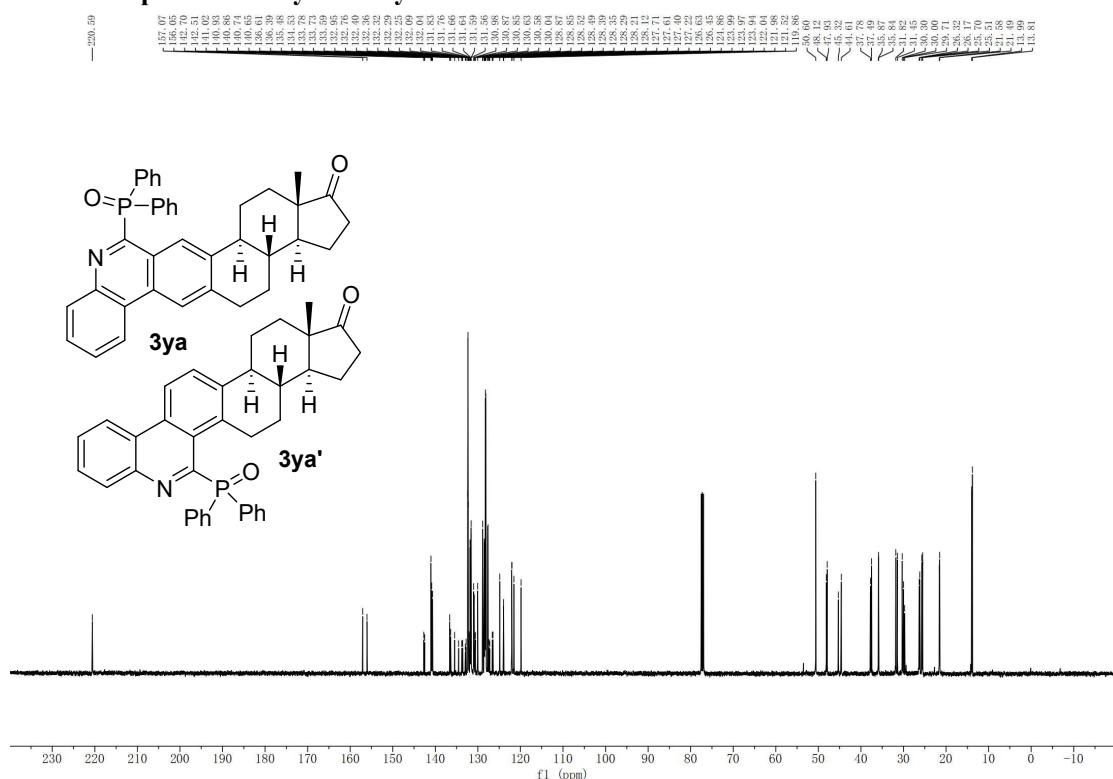
^{31}P NMR spectrum of 3xa



^1H NMR spectrum of 3ya and 3ya'



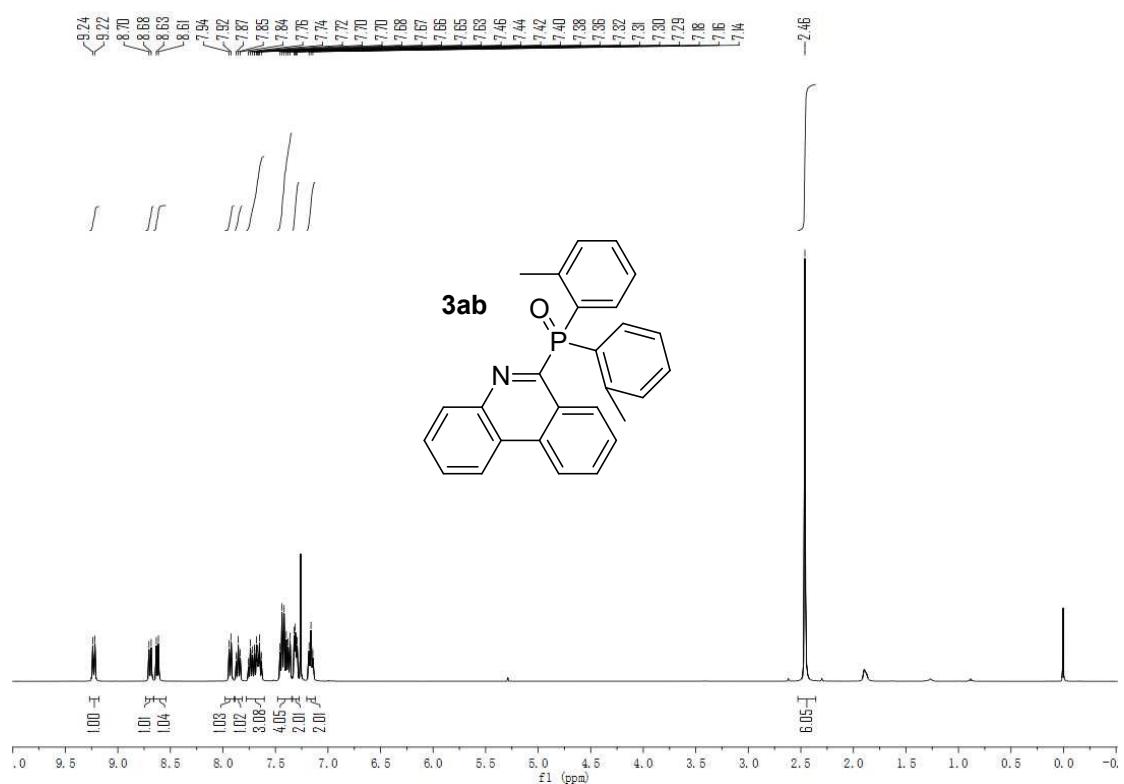
¹³C NMR spectrum of 3ya and 3ya'



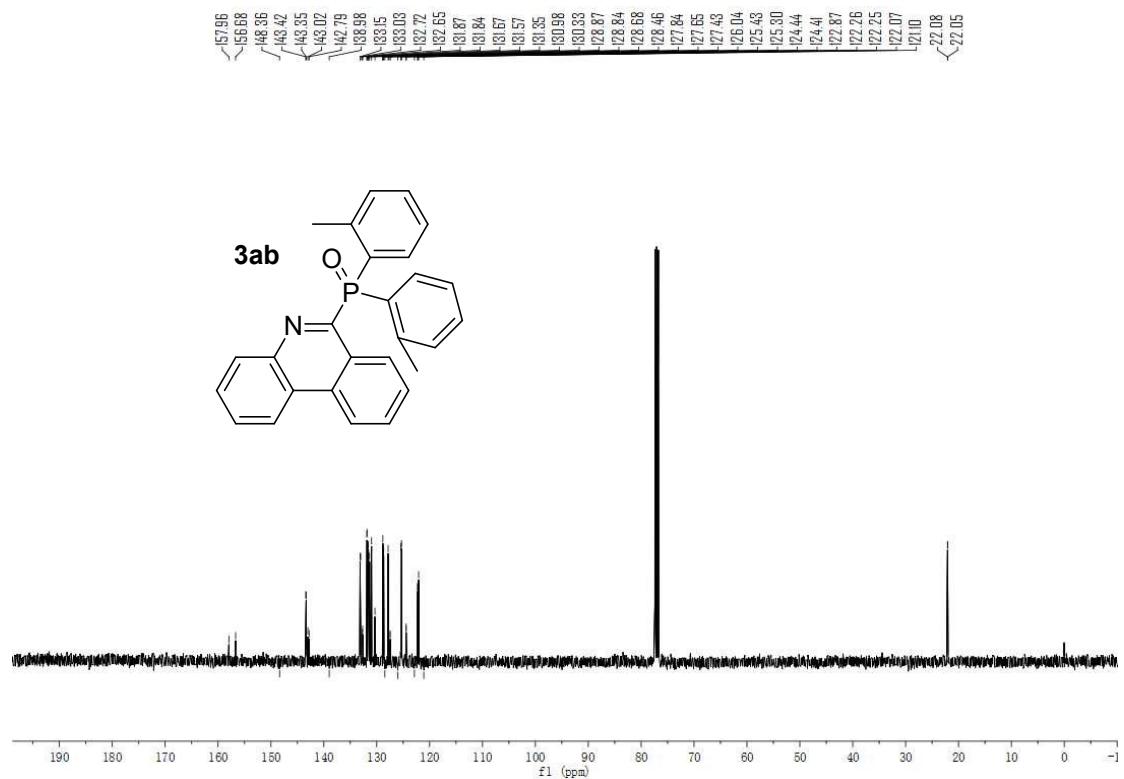
³¹P NMR spectrum of 3ya and 3ya'



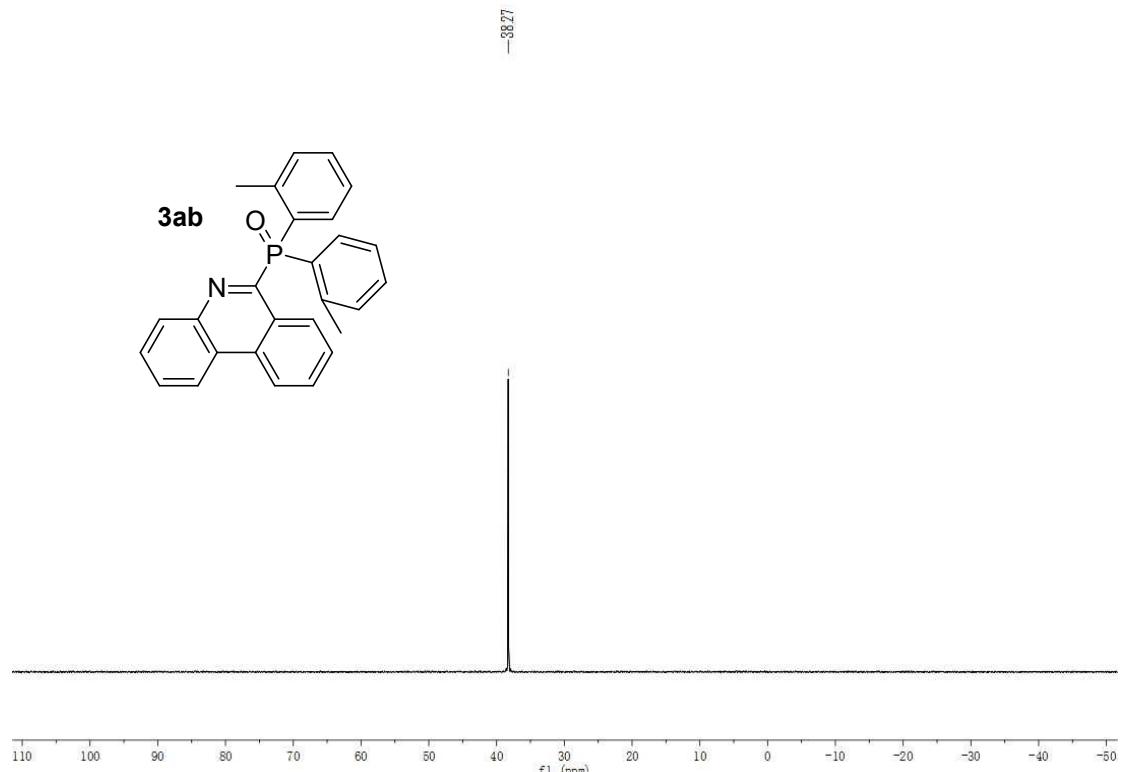
¹H NMR spectrum of 3ab



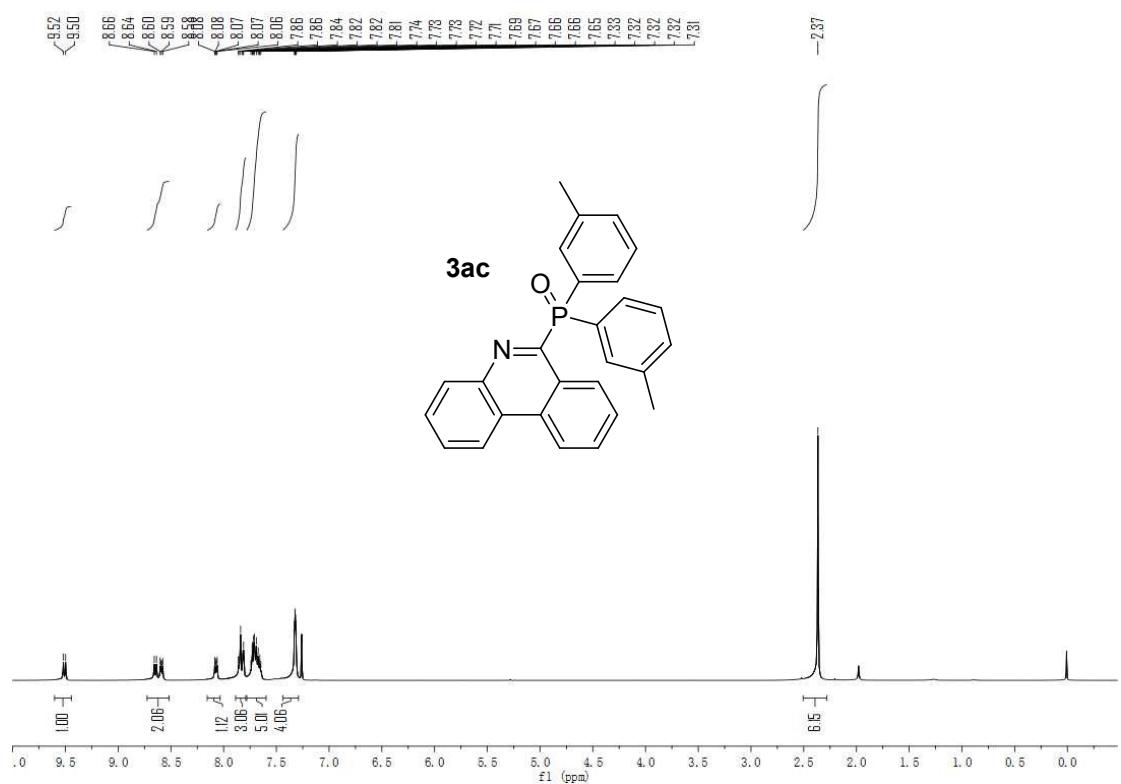
¹³C NMR spectrum of 3ab



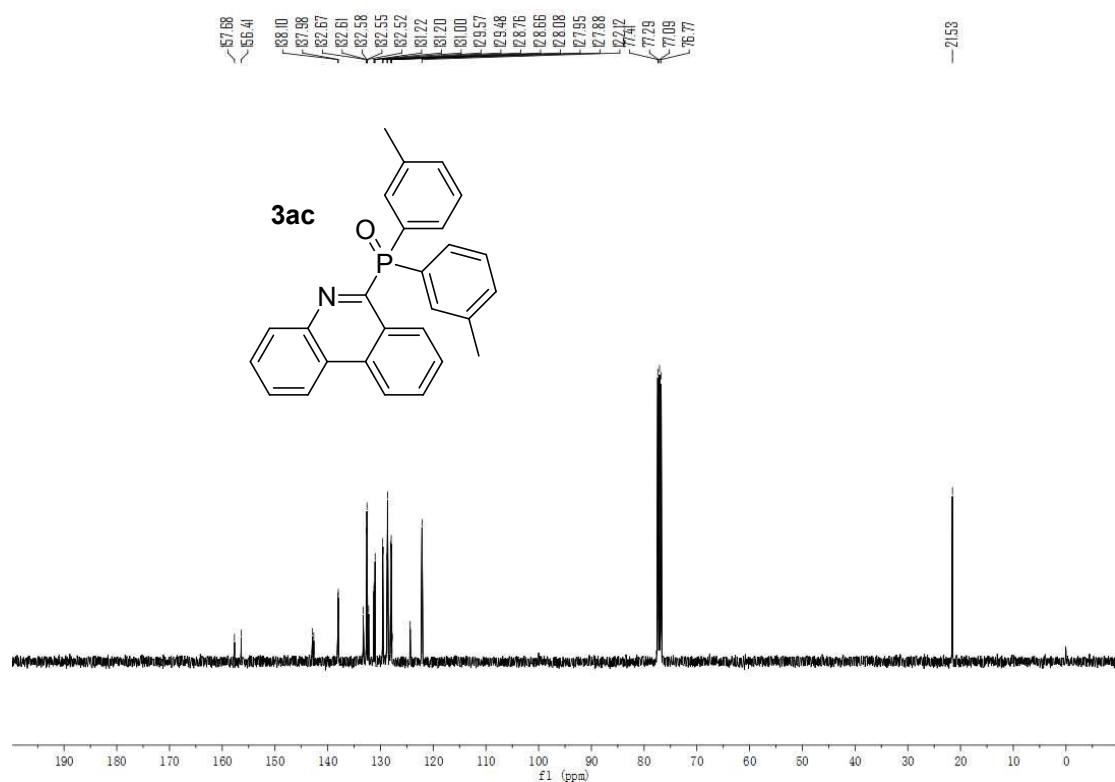
^{31}P NMR spectrum of 3ab



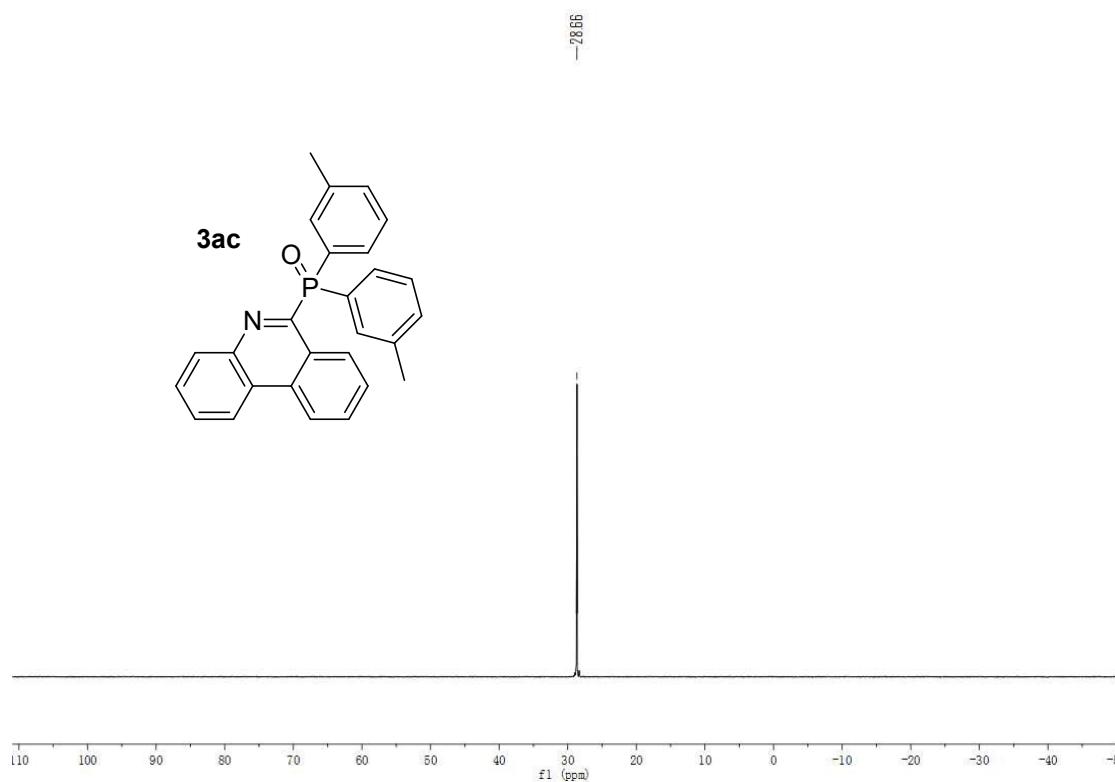
^1H NMR spectrum of 3ac



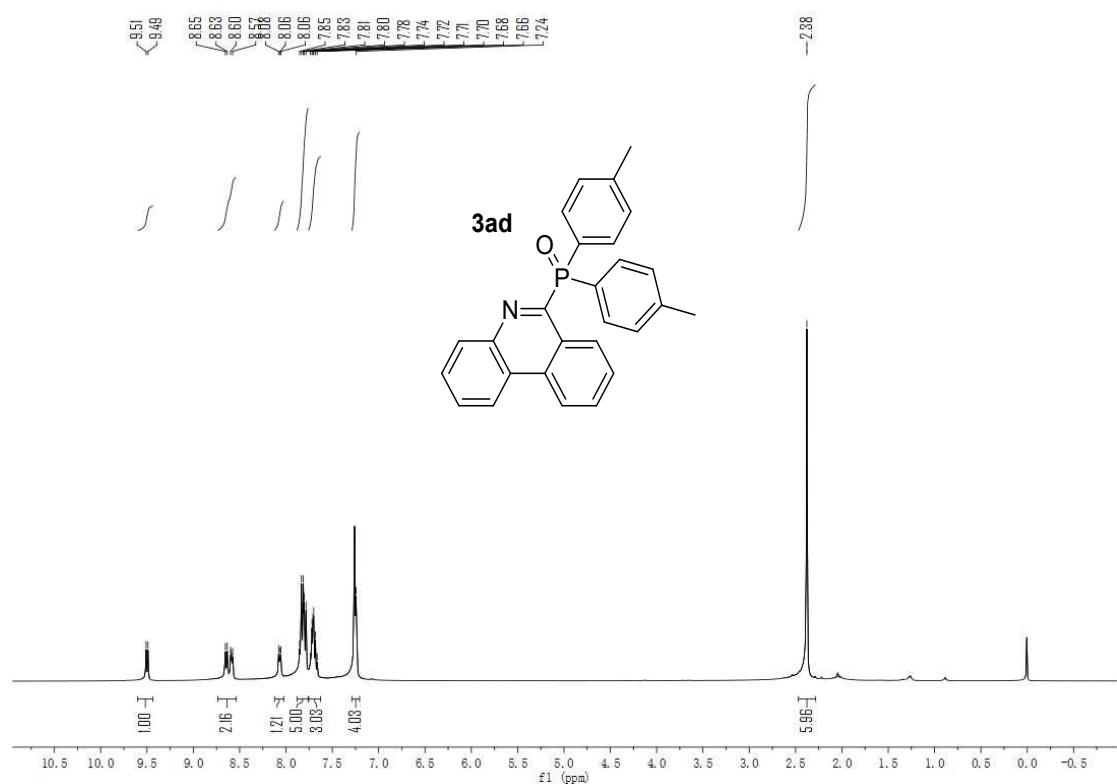
¹³C NMR spectrum of 3ac



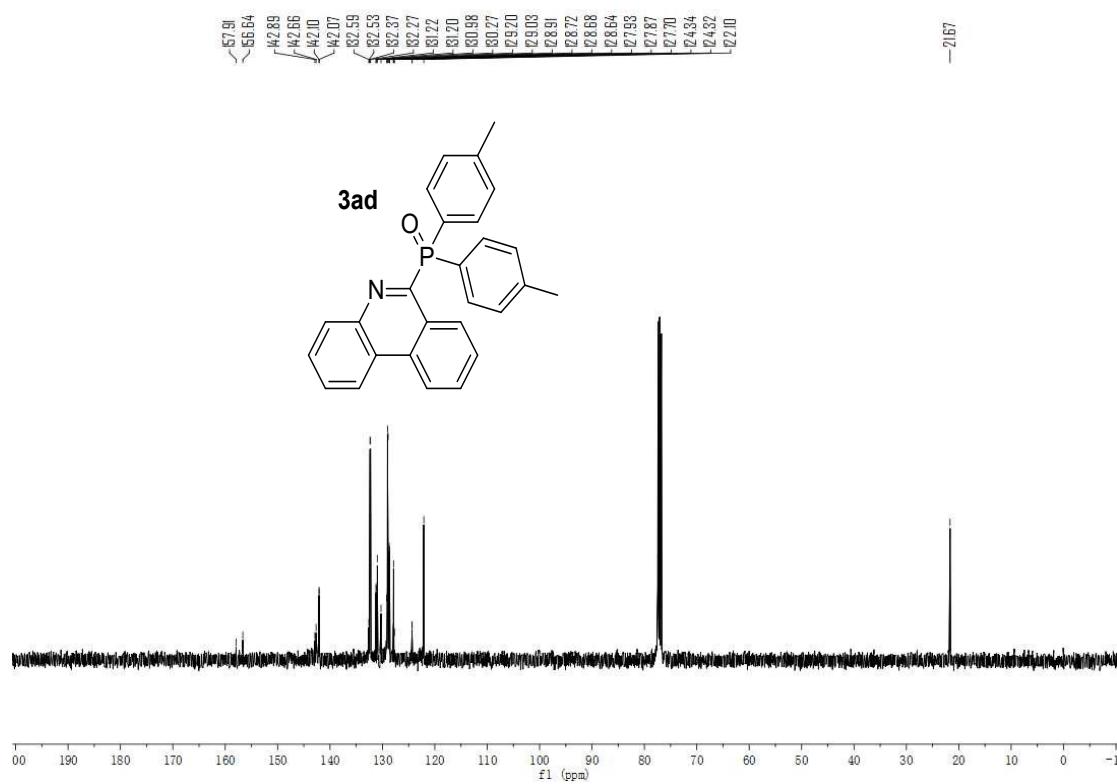
³¹P NMR spectrum of 3ac



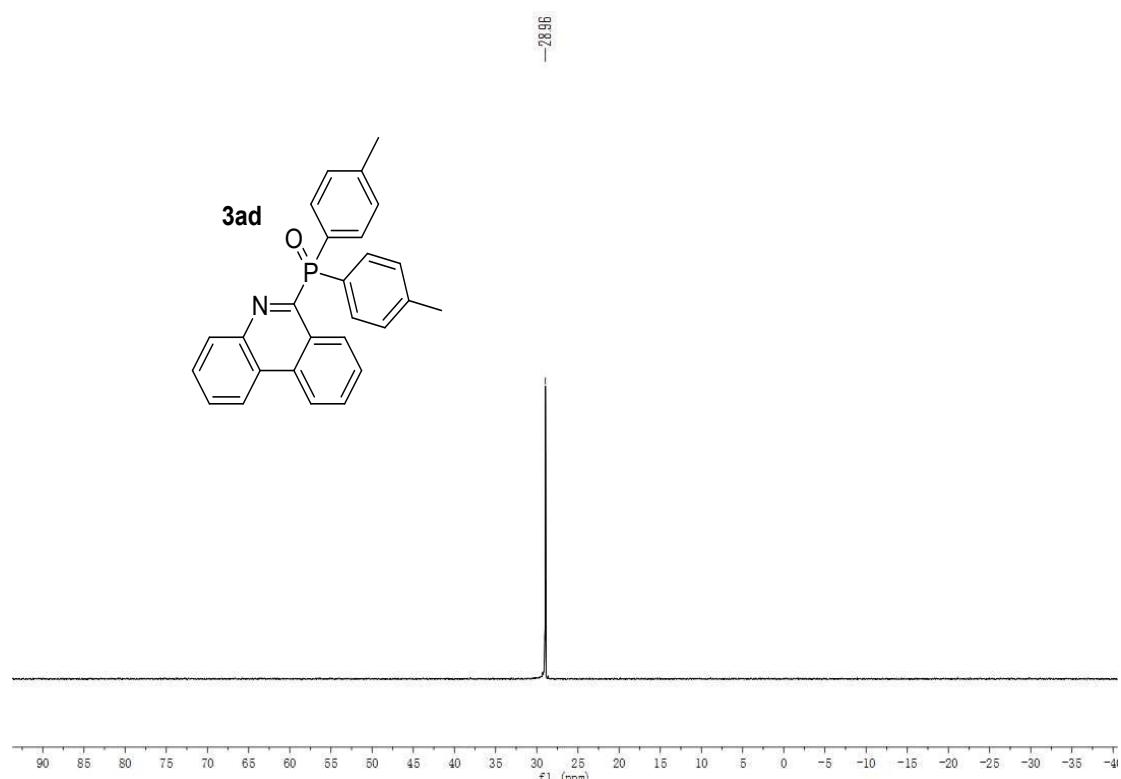
¹H NMR spectrum of 3ad



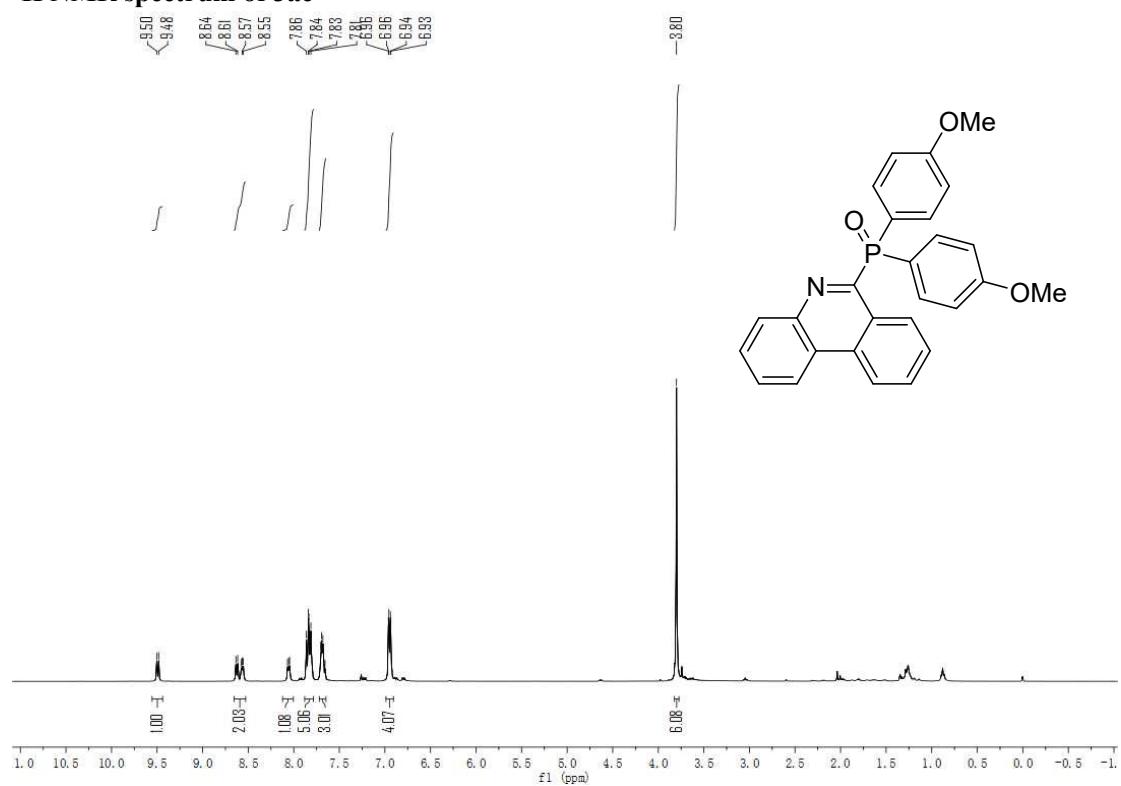
¹³C NMR spectrum of 3ad



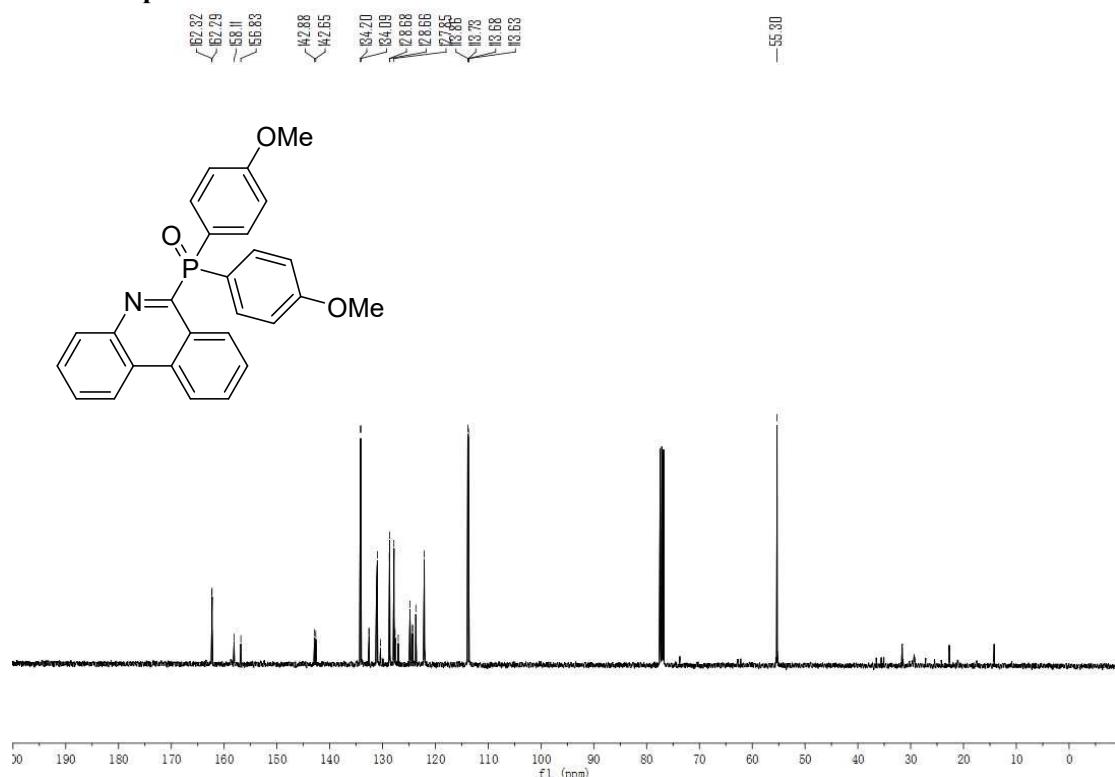
^{31}P NMR spectrum of 3ad



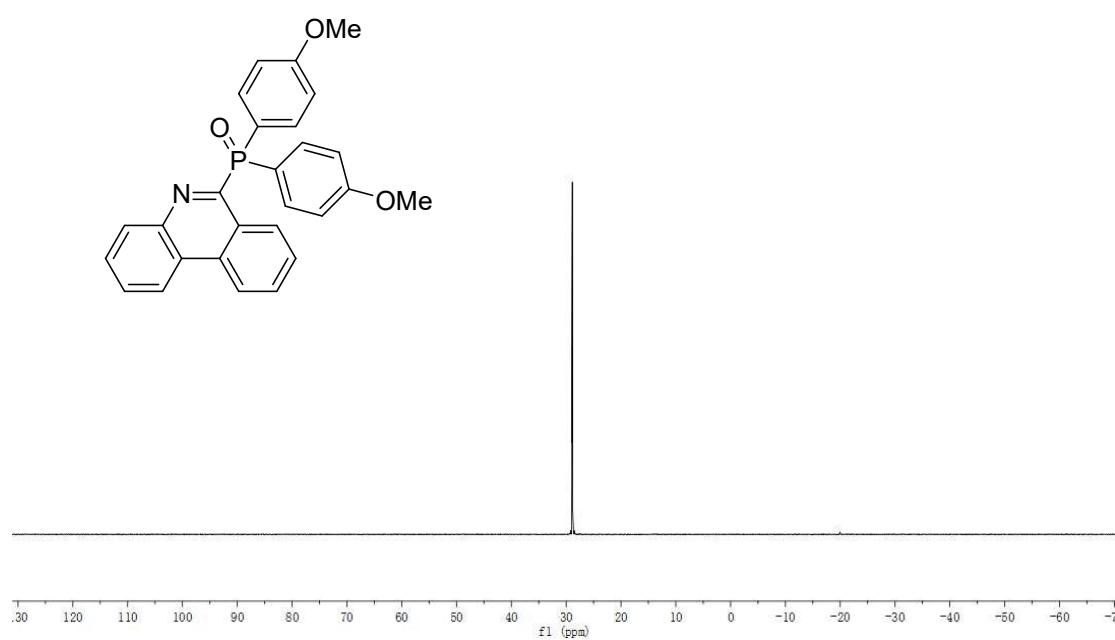
^1H NMR spectrum of 3ae



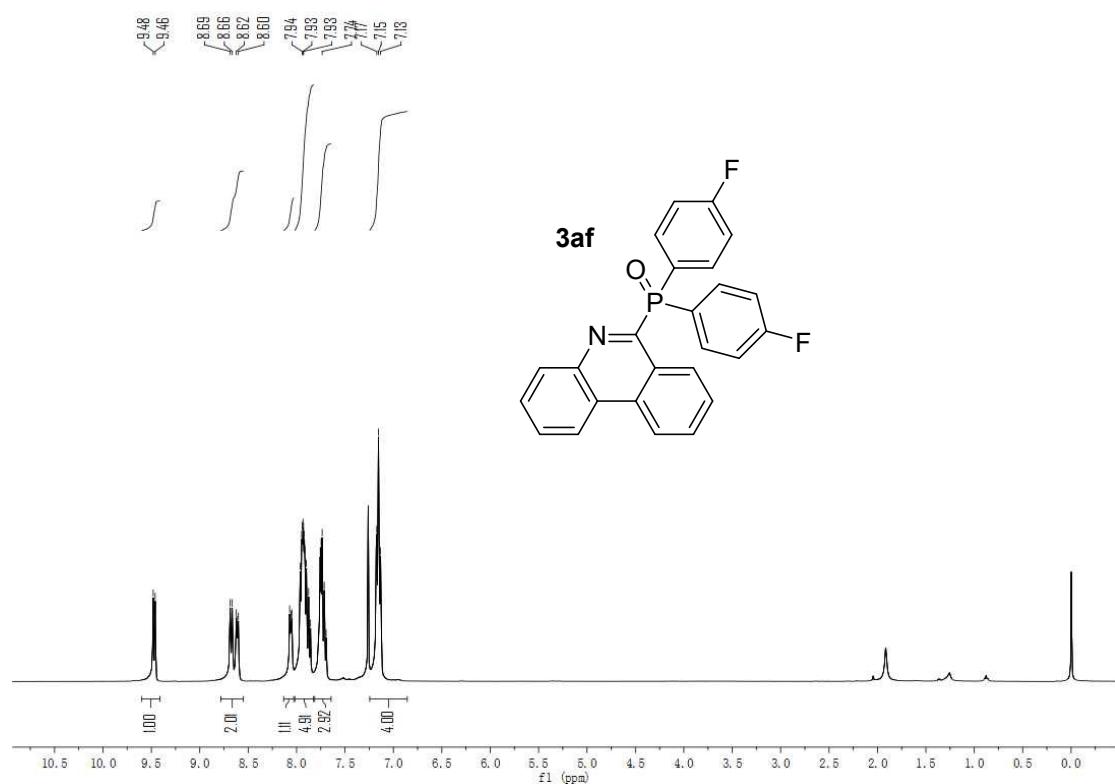
¹³C NMR spectrum of 3ae



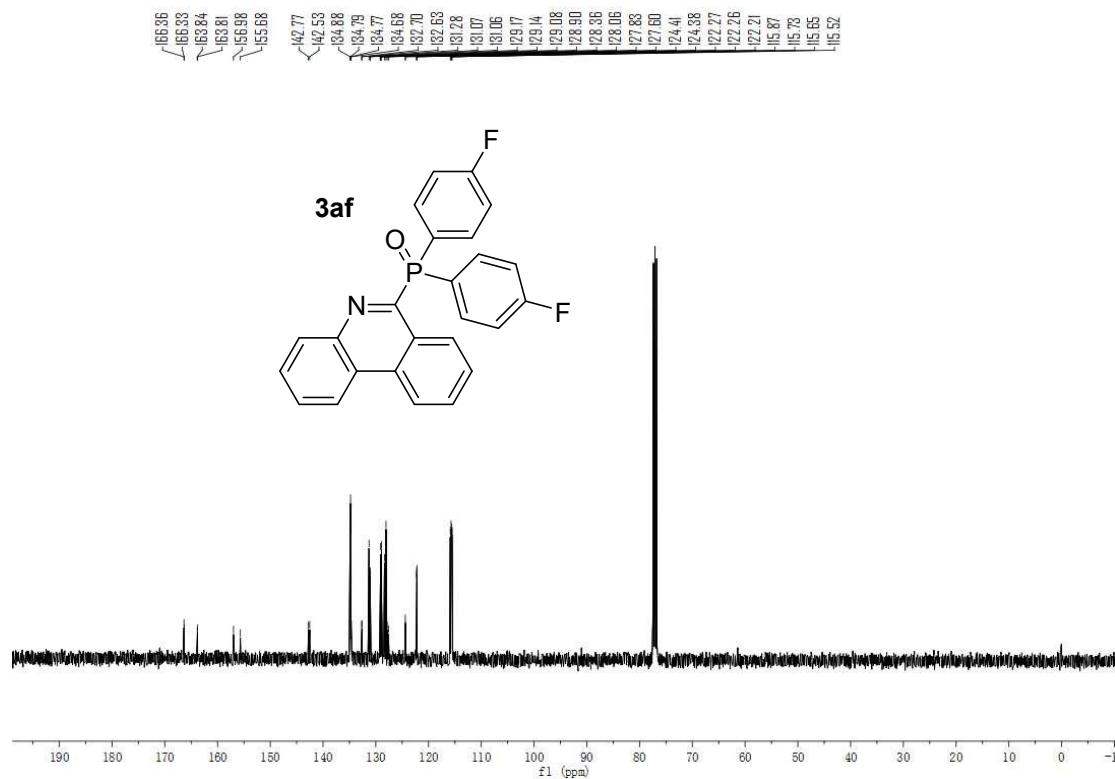
³¹P NMR spectrum of 3ae



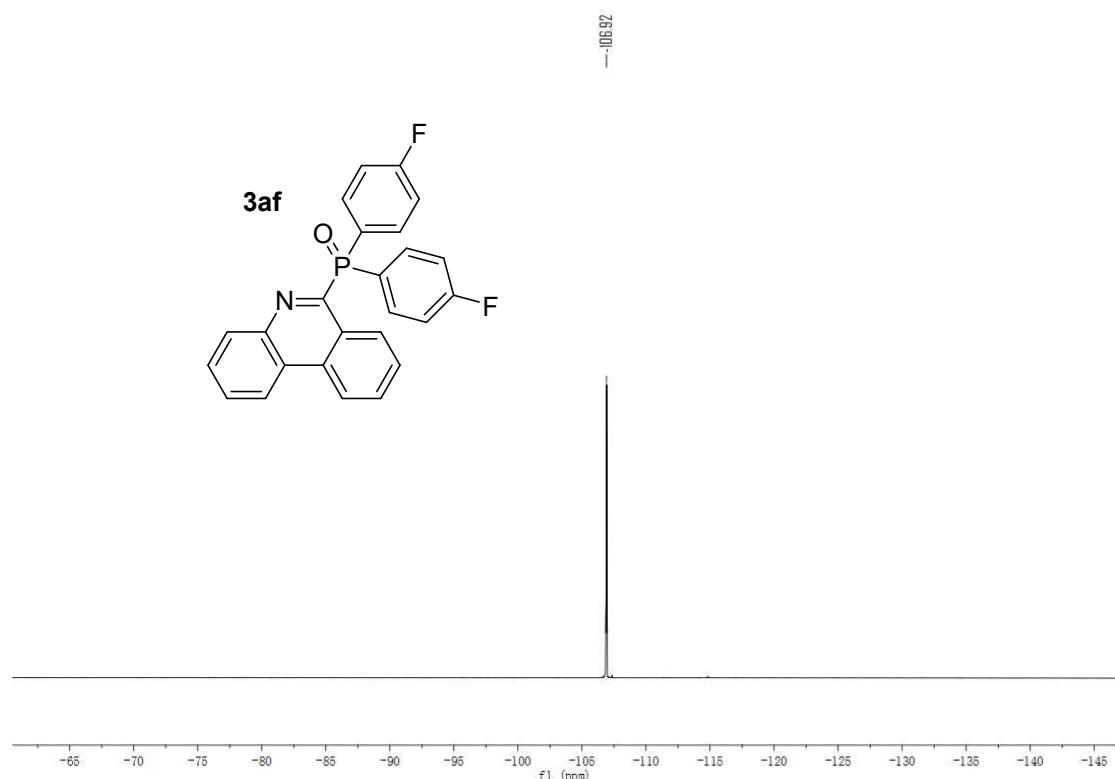
¹H NMR spectrum of 3af



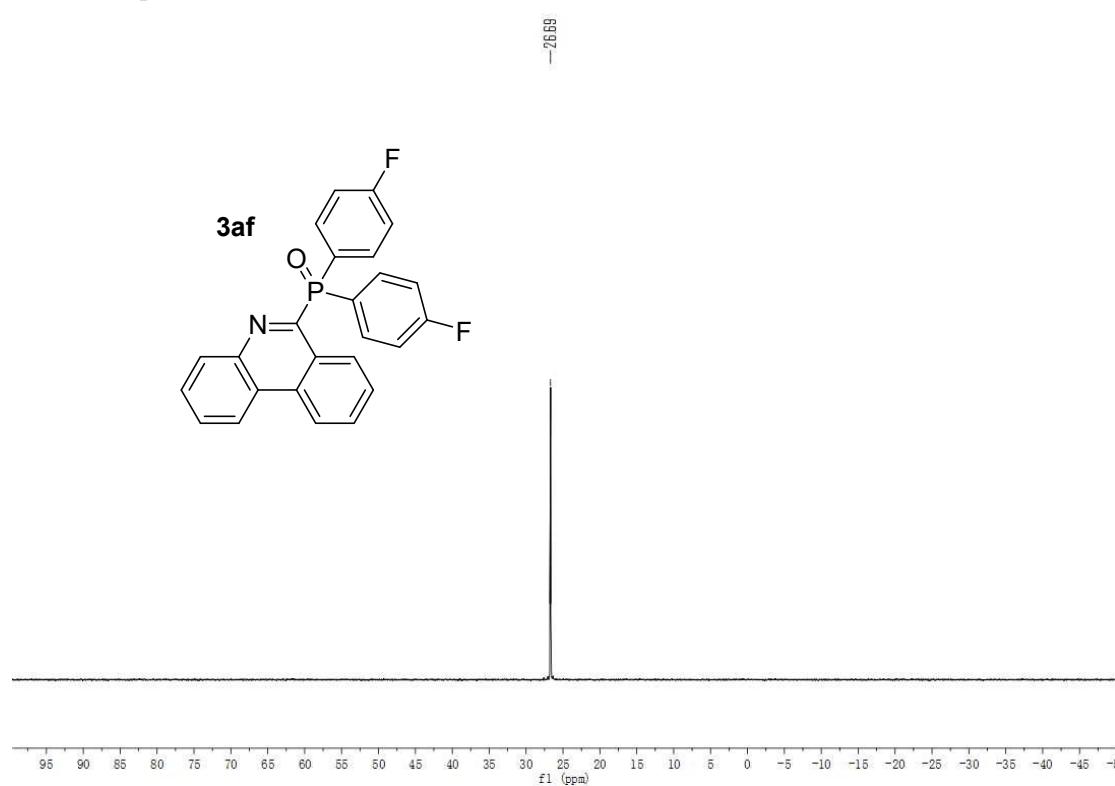
¹³C NMR spectrum of 3af



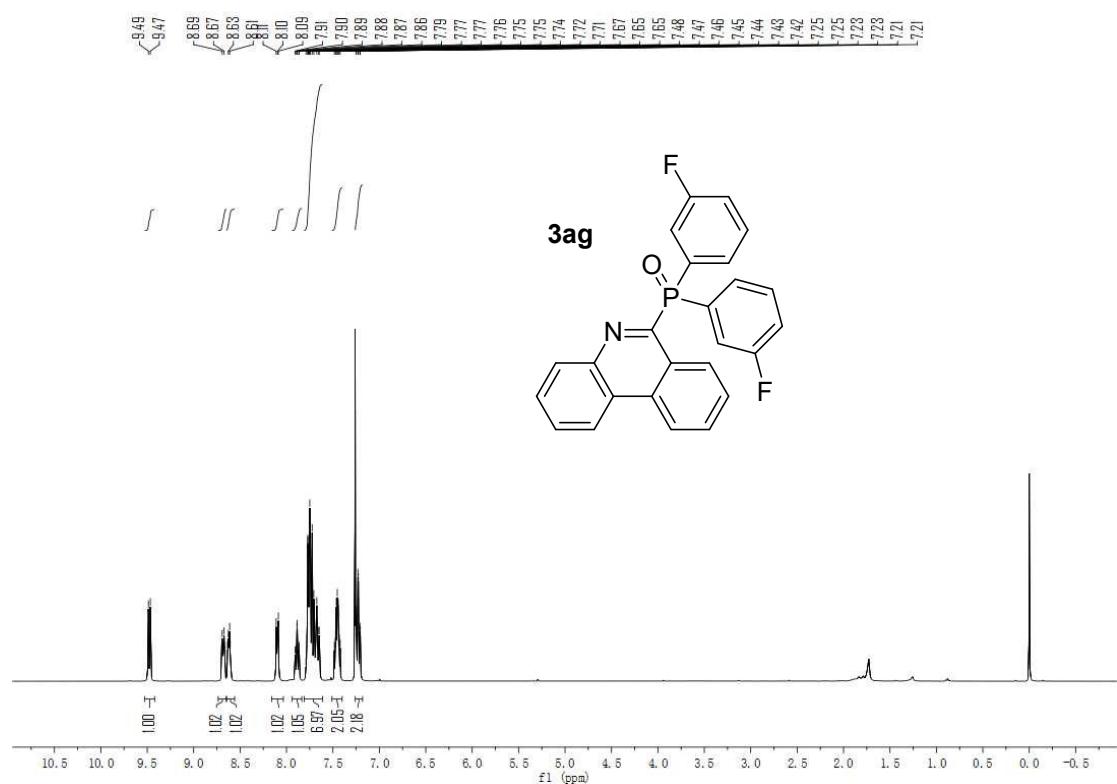
¹⁹F NMR spectrum of 3af



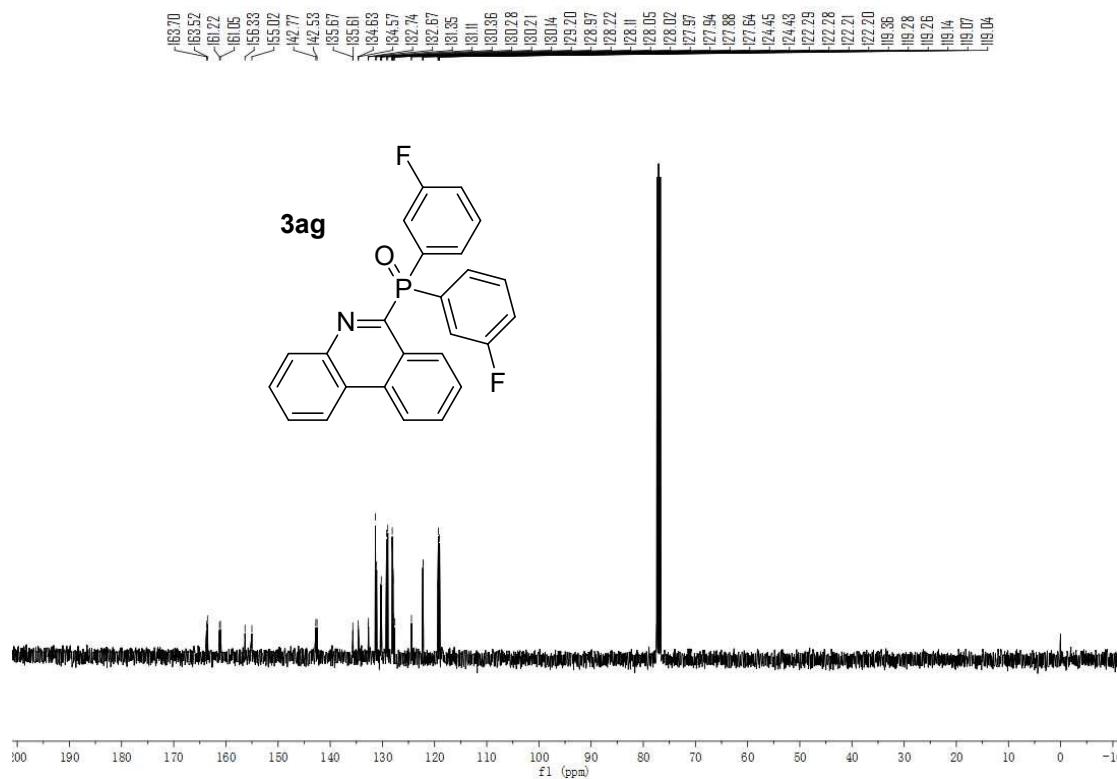
³¹P NMR spectrum of 3af



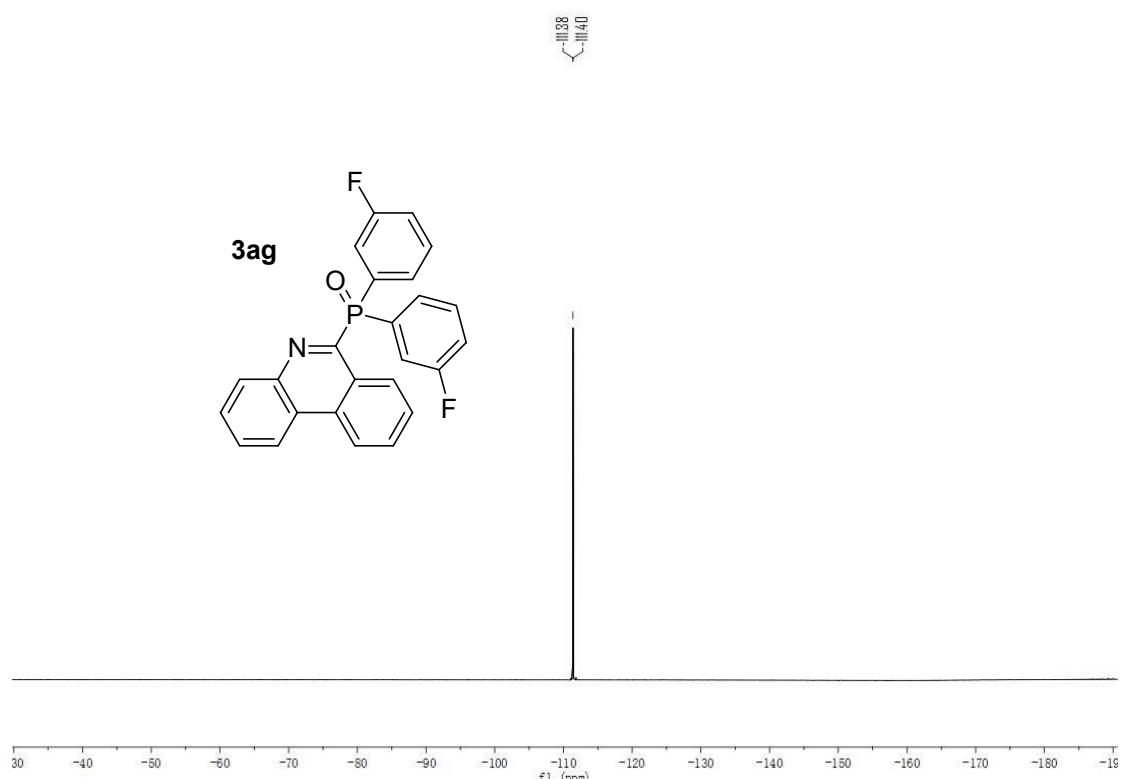
¹H NMR spectrum of 3ag



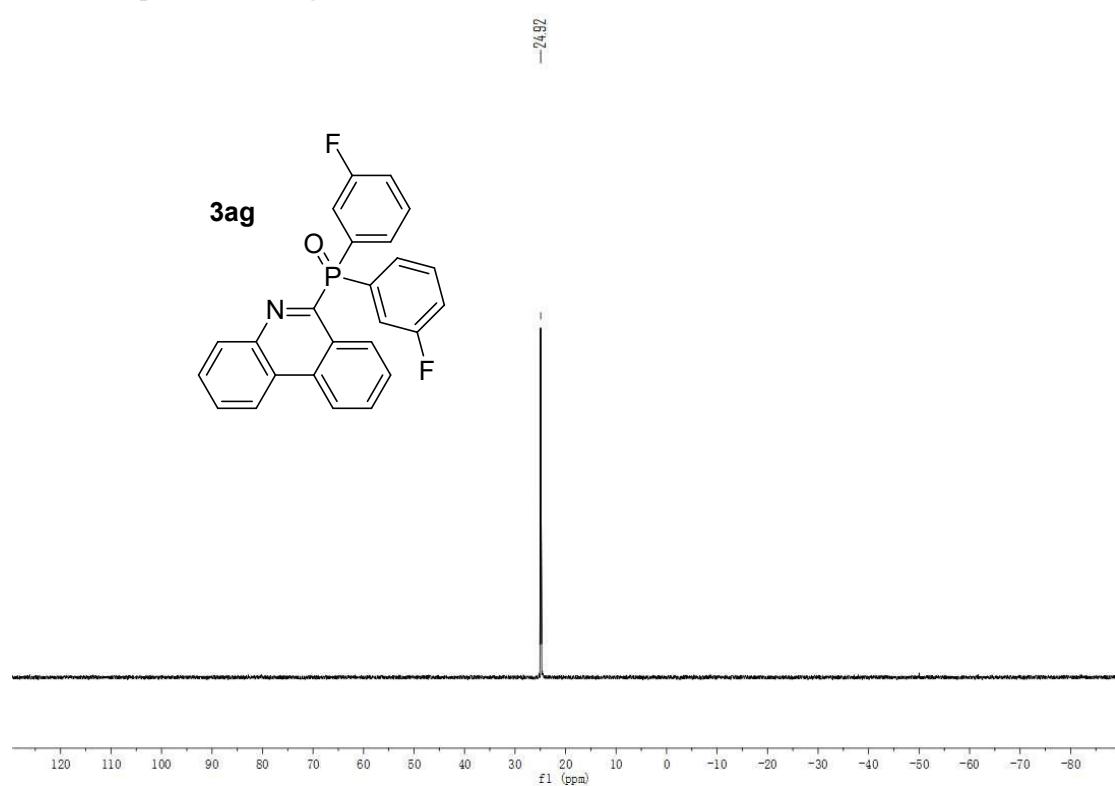
¹³C NMR spectrum of 3ag



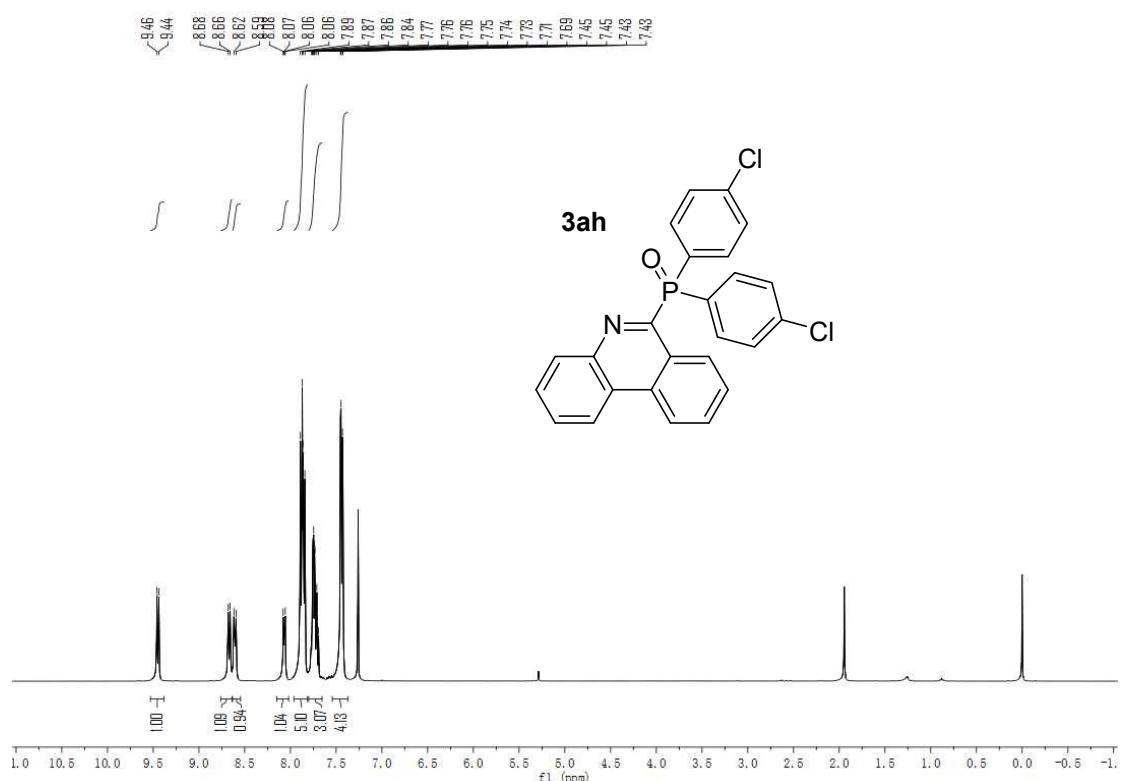
¹⁹F NMR spectrum of 3ag



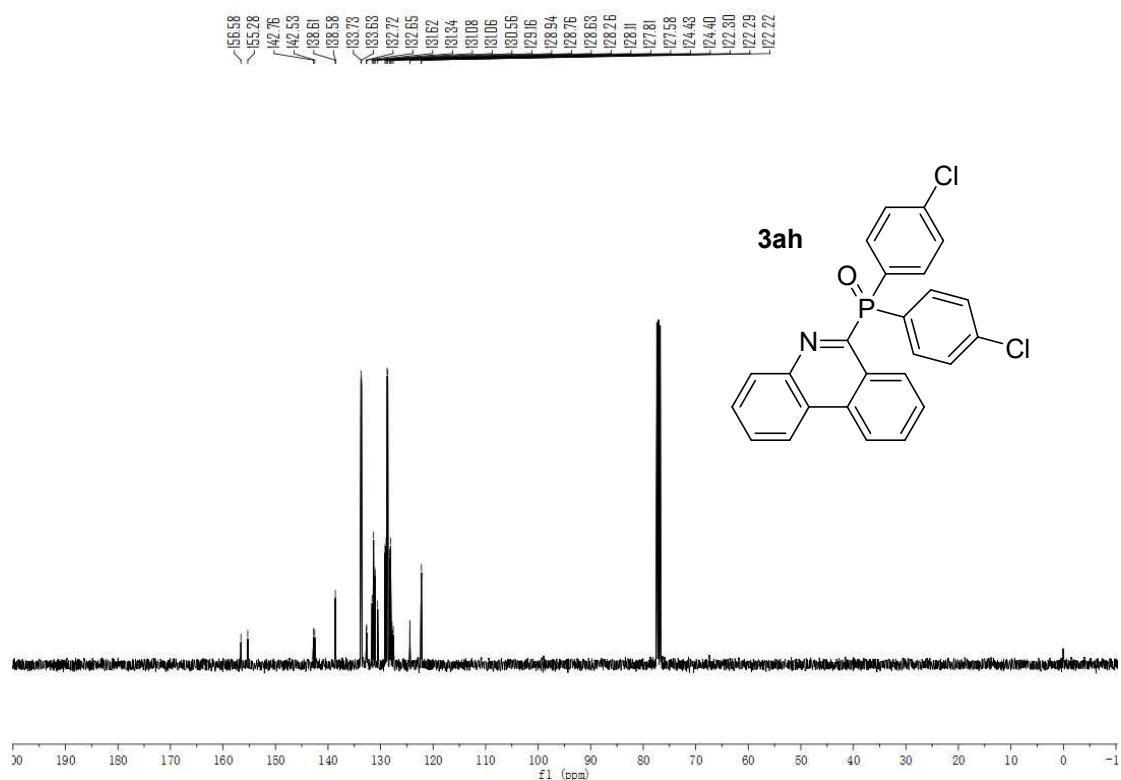
³¹P NMR spectrum of 3ag



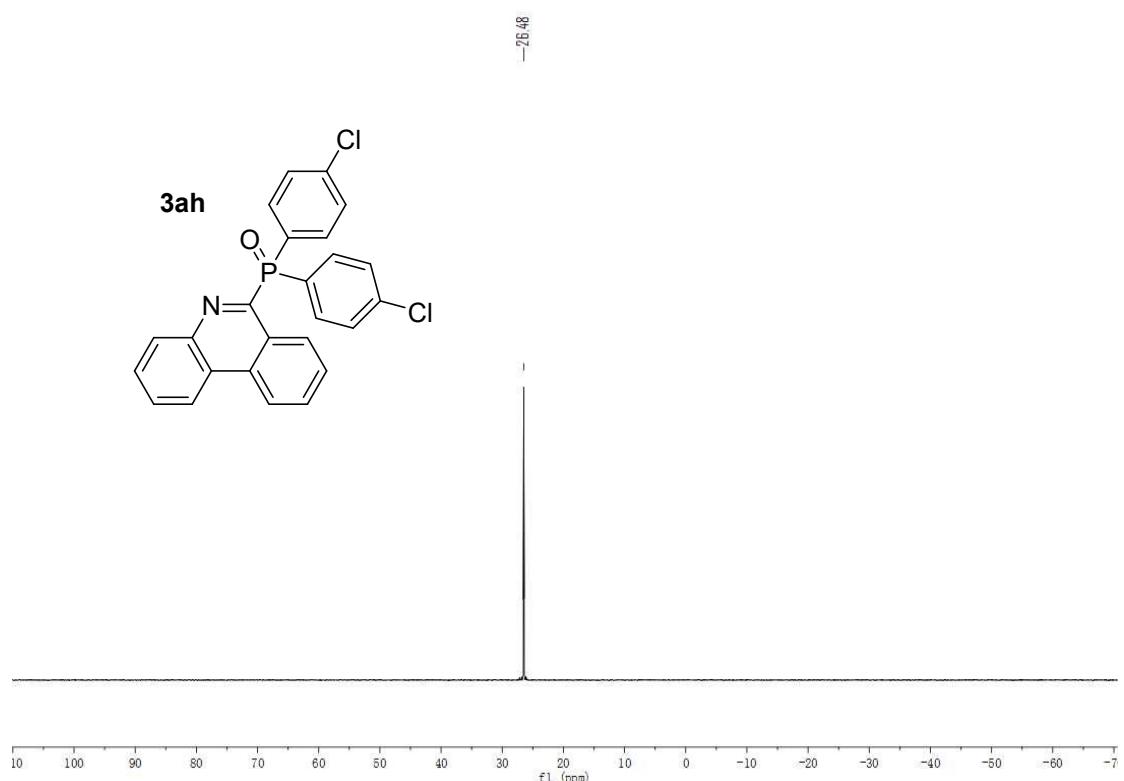
¹H NMR spectrum of 3ah



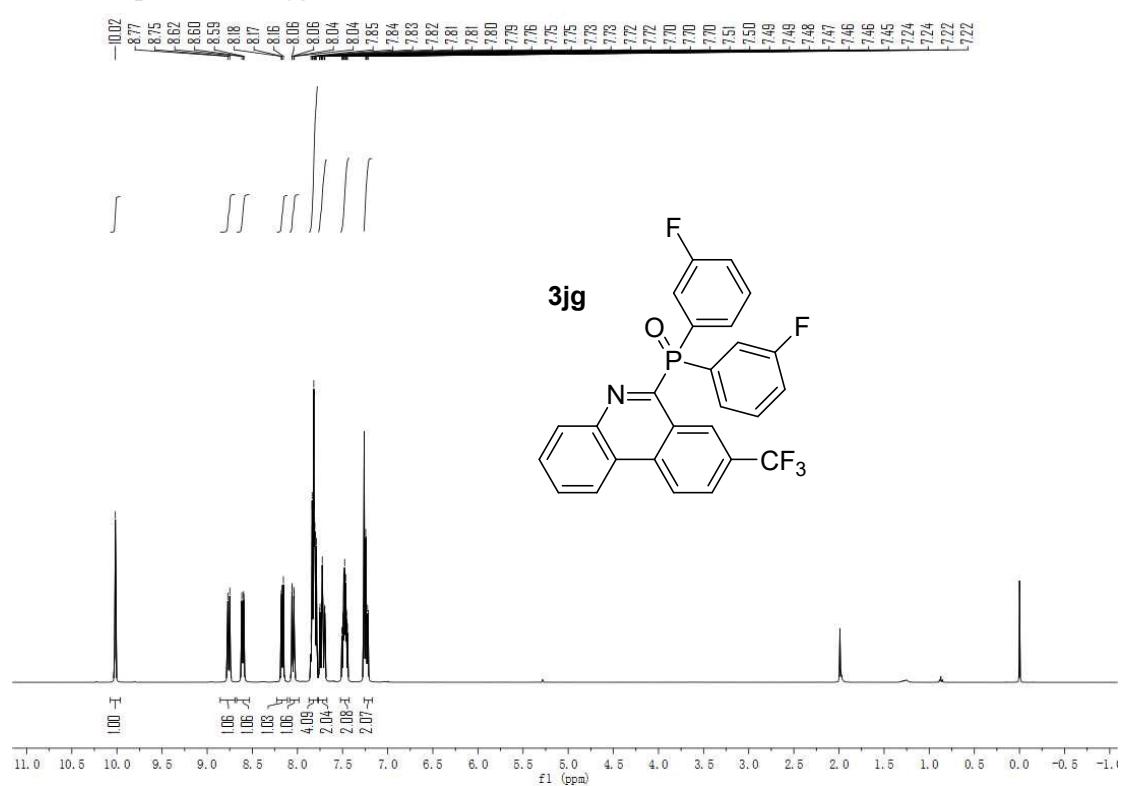
¹³C NMR spectrum of 3ah



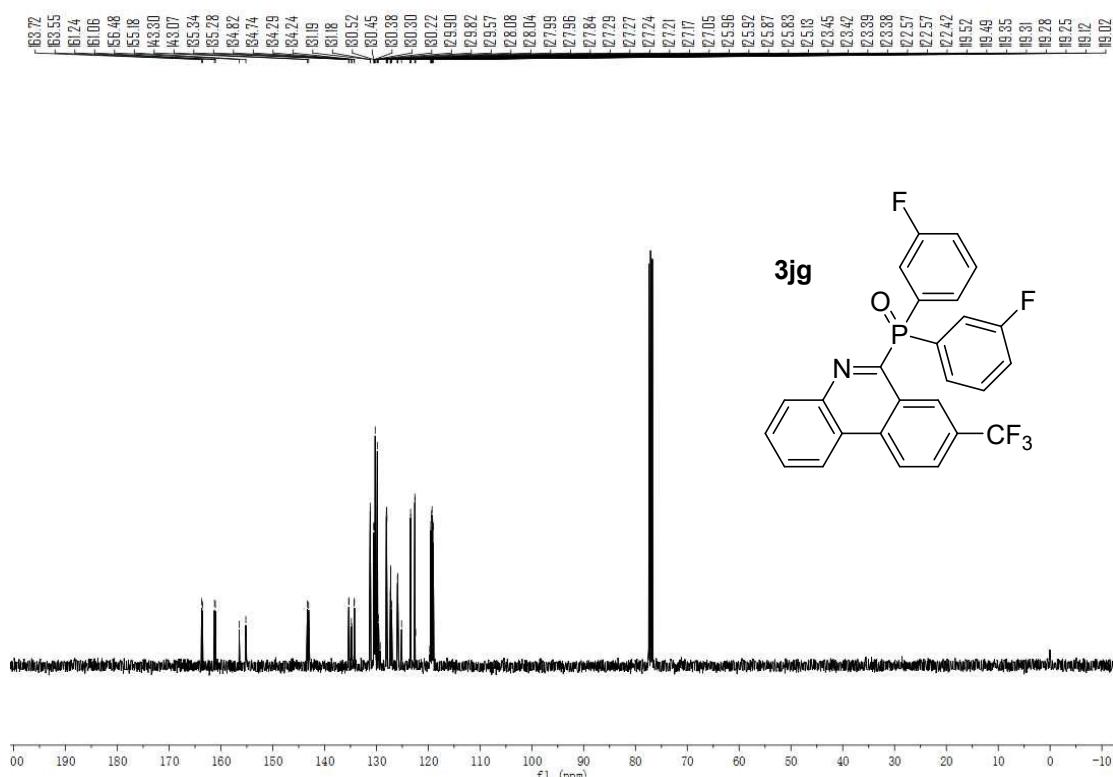
^{31}P NMR spectrum of 3ah



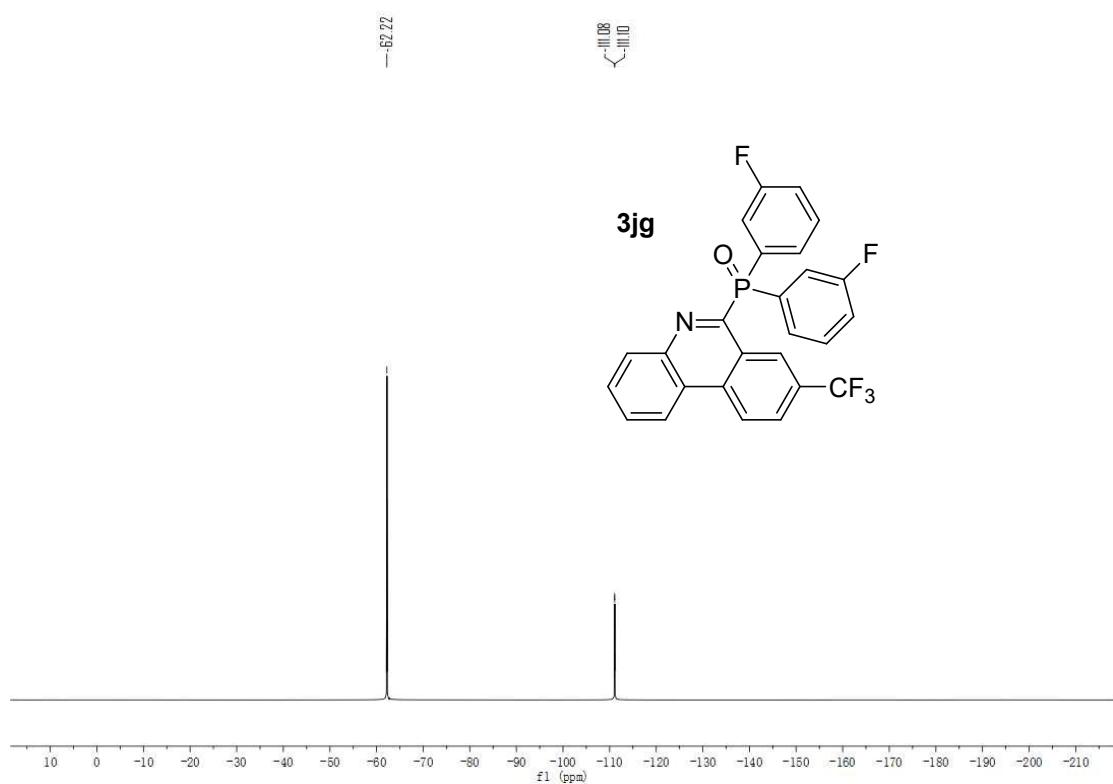
^1H NMR spectrum of 3jg



¹³C NMR spectrum of 3jg



¹⁹F NMR spectrum of 3jg



³¹P NMR spectrum of 3jg

