

Supporting Information for:

Pd-Catalyzed Divergent Regioselective Annulation of Phosphinyl Allenes Accessing Polyarylfurans and 2H- Chromenes

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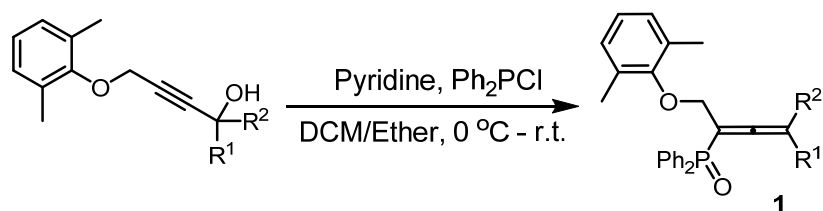
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1. General Information

Solvents and reagents were reagent grade and used without purification unless otherwise noted. Anhydrous dichloromethane and methanol were bought from Aladdin Chemicals, Shanghai, China. Column chromatography was performed using silica gel (200-300 mesh). The $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, $^{19}\text{F-NMR}$, $^{31}\text{P-NMR}$ data reported in ppm (δ) were recorded on a 400, 500 or 600 MHz NMR Bruker-DMX with tetramethylsilane (TMS) as an internal standard and CDCl_3 as solvent unless otherwise stated. Coupling constants are reported in Hertz (Hz). Spectral splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; and br, broad. High resolution mass spectroscopic data of the products were collected on a Waters Micromass GCT instrument using EI (70 eV) or an Agilent Technologies 6540 UHD Accurate-Mass Q-TOF LC/MS using ESI.

2. General Procedures for Substrates Preparation

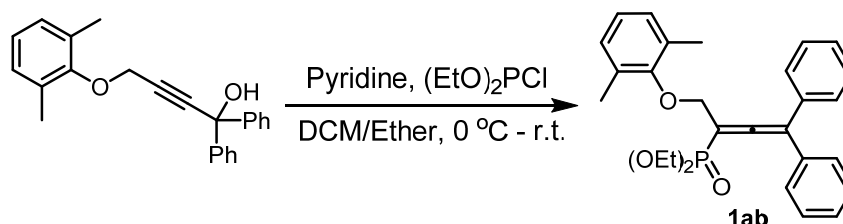
All allenylphosphine oxide substrates (**1**) were prepared according to the procedure reported in our previous works, please see more details in: Y.-T. Xia, J.-J. Wu, C.-Y. Zhang, M. Mao, Y.-G. Ji, L. Wu, *Org. Lett.* **2019**, *21*, 6383; K. Wei, K. Luo, F. Liu, L. Wu, L.-Z. Wu, *Org. Lett.* **2019**, *21*, 1994; J. Zhu, M. Mao, H.-J. Ji, J.-Y. Xu, L. Wu, *Org. Lett.* **2017**, *19*, 1946; L. Zhang, J. Zhu, J. Ma, L. Wu, W.-H. Zhang, *Org. Lett.* **2017**, *19*, 6308; Y. Li, S.-Y. Zhang, X.-L. Yan, J. Zhu, K. Luo, L. Wu, *Org. Lett.* **2023**, *25*, 4720.



To a stirred solution of acetylenic alcohol (20 mmol) at $0\text{ }^\circ\text{C}$ in anhydrous ether (25 mL) and pyridine (24 mmol, 1.9 mL) under nitrogen atmosphere, diphenylphosphine chloride (20 mmol) in CH_2Cl_2 (25 mL) was added dropwise. The mixture was stirred at $0\text{ }^\circ\text{C}$ for 1 h. Then, the mixture was stirred at room temperature overnight. After completion of the reaction monitored by TLC, cold water was added

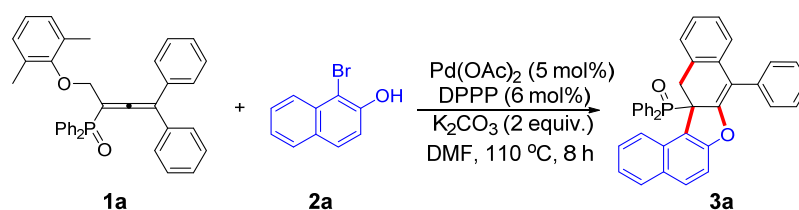
and extracted with DCM twice. The organic layer was combined, dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent to give allenylphosphine oxide.

Using (EtO)₂P(=O)Cl instead of Ph₂P(=O)Cl, under the same reaction conditions, diethoxy-substituted phosphinyl allene (**1ab**) have been obtained



1ab: A colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 7.47–7.46 (m, 4H), 7.40–7.37 (m, 4H), 7.35–7.32 (m, 2H), 7.00 (d, *J* = 5.0 Hz, 2H), 6.94–6.91 (m, 1H), 4.61 (d, *J* = 10.0 Hz, 2H), 4.19–4.11 (m, 4H), 2.29 (s, 6H), 1.29 (t, *J* = 5.0 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 212.00 (d, *J* = 2.5 Hz), 155.61, 134.75 (d, *J* = 7.2 Hz), 131.26, 128.99, 128.87 (d, *J* = 2.8 Hz), 128.77, 128.30, 124.35, 113.27 (d, *J* = 15.8 Hz), 95.47 (d, *J* = 189.8 Hz), 69.01 (d, *J* = 11.6 Hz), 62.81 (d, *J* = 6.0 Hz), 16.47. ³¹P NMR (162 MHz, CDCl₃) δ 15.37.

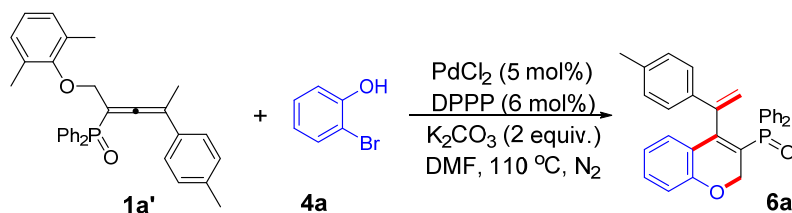
3. General Procedure for the Synthesis of Polyarylfuran **3a**



A typical experimental procedure for the synthesis of **3a** in 0.2 mmol scale: Allenylphosphine oxide **1a** (105.2 mg, 0.2 mmol, 1 equiv.), 1-bromo-2-naphthol **2a** (53.5 mg, 0.24 mmol, 1.2 equiv.), palladium acetate (2.3 mg, 0.01 mmol, 5 mol%), 1,3-bis(diphenylphosphino)propane DPPP (4.9 mg, 0.012 mmol, 6 mol%), potassium carbonate (55.3 mg, 0.4 mmol, 2.0 equiv.) were added into a 25 mL Schlenk tube. The vessel was evacuated and backfilled with N₂ three times, DMF (2 mL) was added via a syringe. Then, the mixture was stirred at 110 °C for 8 h in the oil bath. Upon completion of the reaction, water (30.0 mL) was added and extracted with EtOAc twice. Combined

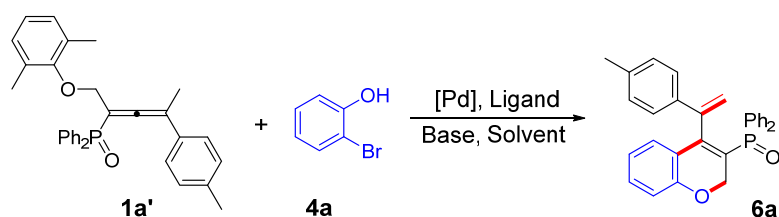
organic layer was dried over anhydrous Na_2SO_4 , and the solvent was removed under reduced pressure to afford the crude product, which was further purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/3) to afford the desired product **3a** (79.7 mg, 73%).

4. General Procedure for the Synthesis of 2*H*-Chromene **6a**



A typical experimental procedure for the synthesis of **6a** in 0.2 mmol scale: Allenylphosphine oxide **1a'** (95.6 mg, 0.2 mmol, 1 equiv.), 2-bromophenol **4a** (41.5 mg, 0.24 mmol, 1.2 equiv.), palladium chloride (1.8 mg, 0.01 mmol, 5 mol%), 1,3-bis(diphenylphosphino)propane DPPP (4.9 mg, 0.012 mmol, 6 mol%), potassium carbonate (55.3 mg, 0.4 mmol, 2.0 equiv.) were added into a 25 mL Schlenk tube. The vessel was evacuated and backfilled with N_2 three times, DMF (2 mL) was added via a syringe. Then, the mixture was stirred at 110 °C for 8 h in the oil bath. Upon completion of the reaction, water (30.0 mL) was added and extracted with EtOAc twice. Combined organic layer was dried over anhydrous Na_2SO_4 , and the solvent was removed under reduced pressure to afford the crude product, which was further purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/1) to afford the desired product **6a** (89.6 mg, 91%).

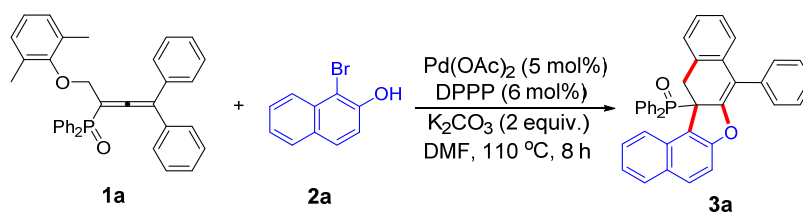
5. Optimization of the Reaction Conditions ^a



Entry	Pd(Cat.)	Ligand	Base	Solvent	Yield (%) ^b
1	Pd(OAc) ₂	DPPP	K ₂ CO ₃	DMF	63
2	Pd(PPh ₃) ₂ Cl ₂	---	K ₂ CO ₃	DMF	50
3	Pd ₂ (dba) ₃	---	K ₂ CO ₃	DMF	n.d.
4	Pd(acac) ₂	---	K ₂ CO ₃	DMF	42
5	Pd(TFA) ₂	DPPP	K ₂ CO ₃	DMF	n.d.
6	PdCl ₂	DPPP	K ₂ CO ₃	DMF	91
7	PdCl ₂	DPPP	K ₃ PO ₄	DMF	trace
8	PdCl ₂	DPPP	Na ₂ CO ₃	DMF	44
9	PdCl ₂	DPPP	NaHCO ₃	DMF	72
10	PdCl ₂	DPPP	DIPEA	DMF	trace
11	PdCl ₂	DPPP	DIPA	DMF	trace
12	PdCl ₂	DPPP	K ₂ CO ₃	Toluene	n.d.
13	PdCl ₂	DPPP	K ₂ CO ₃	THF	n.d.
14	PdCl ₂	DPPP	K ₂ CO ₃	MeCN	n.d.
15	PdCl ₂	DPPP	K ₂ CO ₃	DCE	n.d.
16	PdCl ₂	DPPP	K ₂ CO ₃	DMAc	78

^aReaction conditions: **1a'** (0.2 mmol), **4a** (0.24 mmol), Base (0.4 mmol), Catalyst (5 mol%), Ligand (6 mol%), 110 °C, N₂, 8 h. ^bIsolated yield by column chromatography. n.d.= no detect.

6. Gram-Scale Synthesis of **3a**



Allenylphosphine oxide **1a** (526.2 mg, 1.0 mmol, 1.0 equiv.), 1-bromo-2-naphthol

2a (223.1 mg, 1.2 mmol, 1.2 equiv.), palladium acetate (11.3 mg, 0.05 mmol, 5 mol%), 1,3-bis(diphenylphosphino)propane DPPP (24.7 mg, 0.06 mmol, 6 mol%), potassium carbonate (276.4 mg, 2.0 mmol, 2.0 equiv.) were added into a 25 mL Schlenk tube. The vessel was evacuated and backfilled with N₂ three times, DMF (10 mL) was added via a syringe.. Then, the mixture was stirred at 110 °C for 8 h in the oil bath. Upon completion of the reaction, water (100 mL) was added and extracted with EtOAc twice. Combined organic layer was dried over anhydrous Na₂SO₄, and the solvent was removed under reduced pressure to afford the crude product, which was further purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/3) to afford the desired product **3a** (371.4 mg, 68%).

7. The Key NOE Correlations of Compound **6i** and **6j**

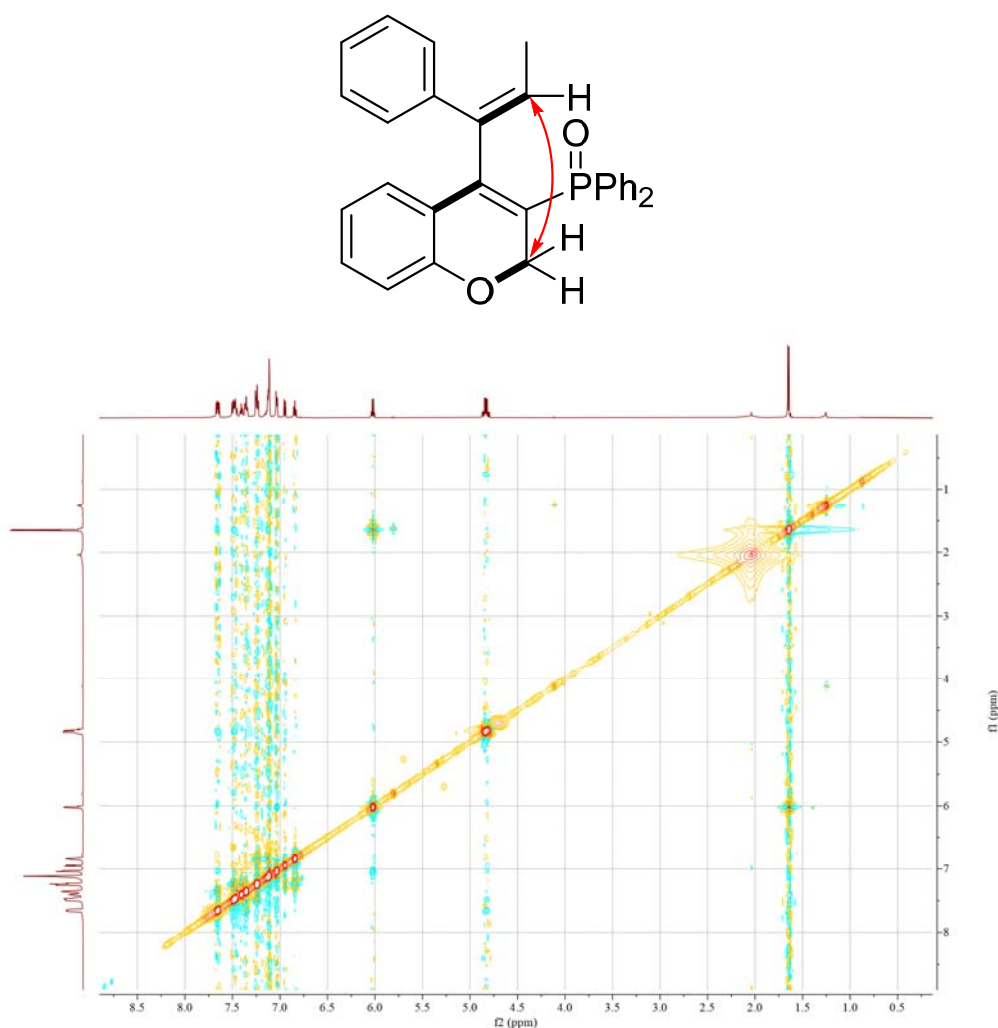


Figure S1 The key NOE correlations of compound **6i**

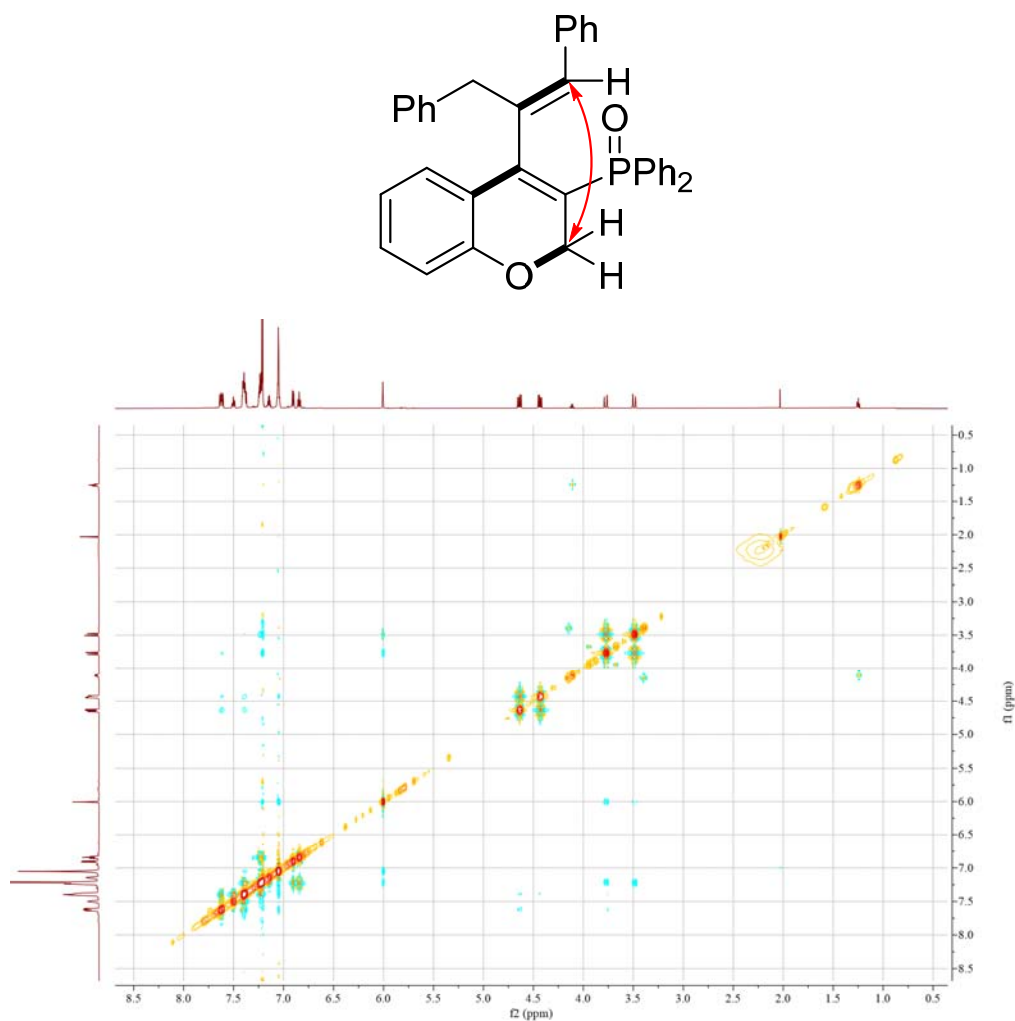


Figure S2 The key NOE correlations of compound **6j**

8. Single Crystal X-Ray Diffraction

The crystal of **3a** (CCDC 2249006) was crystallized by slow vapor diffusion of CH_2Cl_2 layered with petroleum ether at room temperature for several days.

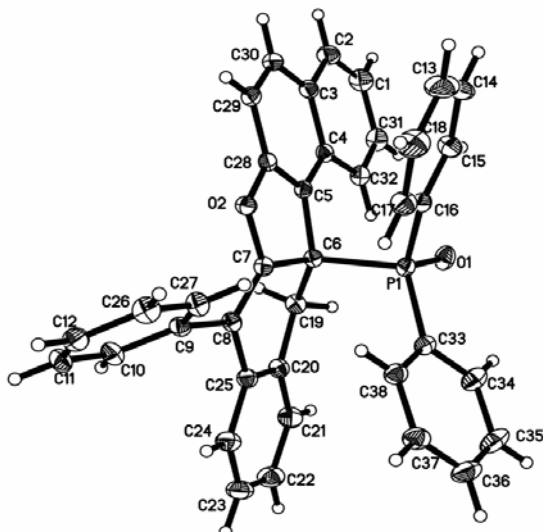


Figure S3. X-ray structures of **3a** (ellipsoids set at 50 % probability).

Table 1. Crystal data and structure refinement for Ly200524_a.

Identification code	Ly200524_a	
Empirical formula	C ₃₈ H ₂₇ O ₂ P	
Formula weight	546.56	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 10.0902(10) Å	α = 90°.
	b = 20.276(2) Å	β = 104.978(3)°.
	c = 14.0678(15) Å	γ = 90°.
Volume	2780.3(5) Å ³	
Z	4	
Density (calculated)	1.306 Mg/m ³	
Absorption coefficient	0.134 mm ⁻¹	
F(000)	1144	
Crystal size	0.250 x 0.220 x 0.200 mm ³	
Theta range for data collection	2.009 to 27.517°.	
Index ranges	-10 ≤ h ≤ 13, -26 ≤ k ≤ 26, -17 ≤ l ≤ 18	
Reflections collected	16608	
Independent reflections	6321 [R(int) = 0.0326]	
Completeness to theta = 25.242°	99.0 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.7456 and 0.6470
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6321 / 0 / 370
Goodness-of-fit on F ²	1.026
Final R indices [I>2sigma(I)]	R1 = 0.0484, wR2 = 0.1093
R indices (all data)	R1 = 0.0770, wR2 = 0.1226
Extinction coefficient	n/a
Largest diff. peak and hole	0.241 and -0.337 e.Å ⁻³

Table 2. Bond lengths [Å] and angles [°] for Ly200524_a.

P(1)-O(1)	1.4805(13)
P(1)-C(16)	1.8057(19)
P(1)-C(33)	1.8134(18)
P(1)-C(6)	1.8914(16)
O(2)-C(28)	1.389(2)
O(2)-C(7)	1.3939(19)
C(1)-C(2)	1.359(3)
C(1)-C(31)	1.404(3)
C(1)-H(1)	0.9300
C(2)-C(3)	1.415(3)
C(2)-H(2)	0.9300
C(3)-C(30)	1.412(3)
C(3)-C(4)	1.429(2)
C(4)-C(32)	1.415(2)
C(4)-C(5)	1.418(2)
C(5)-C(28)	1.368(2)
C(5)-C(6)	1.520(2)
C(6)-C(7)	1.507(2)
C(6)-C(19)	1.532(2)
C(7)-C(8)	1.341(2)
C(8)-C(25)	1.482(2)
C(8)-C(9)	1.483(2)
C(9)-C(27)	1.390(3)
C(9)-C(10)	1.394(2)
C(10)-C(11)	1.376(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.374(3)

C(11)-H(11)	0.9300
C(12)-C(26)	1.379(3)
C(12)-H(12)	0.9300
C(13)-C(14)	1.363(4)
C(13)-C(18)	1.370(4)
C(13)-H(13)	0.9300
C(14)-C(15)	1.384(3)
C(14)-H(14)	0.9300
C(15)-C(16)	1.390(3)
C(15)-H(15)	0.9300
C(16)-C(17)	1.383(3)
C(17)-C(18)	1.391(3)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
C(19)-C(20)	1.510(2)
C(19)-H(19A)	0.9700
C(19)-H(19B)	0.9700
C(20)-C(21)	1.383(3)
C(20)-C(25)	1.406(2)
C(21)-C(22)	1.386(3)
C(21)-H(21)	0.9300
C(22)-C(23)	1.366(3)
C(22)-H(22)	0.9300
C(23)-C(24)	1.378(3)
C(23)-H(23)	0.9300
C(24)-C(25)	1.396(3)
C(24)-H(24)	0.9300
C(26)-C(27)	1.379(3)
C(26)-H(26)	0.9300
C(27)-H(27)	0.9300
C(28)-C(29)	1.397(2)
C(29)-C(30)	1.367(2)
C(29)-H(29)	0.9300
C(30)-H(30)	0.9300
C(31)-C(32)	1.366(3)
C(31)-H(31)	0.9300
C(32)-H(32)	0.9300
C(33)-C(38)	1.384(3)

C(33)-C(34)	1.390(3)
C(34)-C(35)	1.382(3)
C(34)-H(34)	0.9300
C(35)-C(36)	1.377(3)
C(35)-H(35)	0.9300
C(36)-C(37)	1.372(3)
C(36)-H(36)	0.9300
C(37)-C(38)	1.384(3)
C(37)-H(37)	0.9300
C(38)-H(38)	0.9300
O(1)-P(1)-C(16)	111.93(9)
O(1)-P(1)-C(33)	110.51(8)
C(16)-P(1)-C(33)	109.55(8)
O(1)-P(1)-C(6)	108.87(7)
C(16)-P(1)-C(6)	103.85(8)
C(33)-P(1)-C(6)	111.97(8)
C(28)-O(2)-C(7)	105.51(12)
C(2)-C(1)-C(31)	120.15(19)
C(2)-C(1)-H(1)	119.9
C(31)-C(1)-H(1)	119.9
C(1)-C(2)-C(3)	121.62(19)
C(1)-C(2)-H(2)	119.2
C(3)-C(2)-H(2)	119.2
C(30)-C(3)-C(2)	121.66(18)
C(30)-C(3)-C(4)	120.14(16)
C(2)-C(3)-C(4)	118.20(18)
C(32)-C(4)-C(5)	124.42(16)
C(32)-C(4)-C(3)	118.66(16)
C(5)-C(4)-C(3)	116.92(16)
C(28)-C(5)-C(4)	119.89(16)
C(28)-C(5)-C(6)	107.26(14)
C(4)-C(5)-C(6)	132.76(15)
C(7)-C(6)-C(5)	99.96(13)
C(7)-C(6)-C(19)	109.38(13)
C(5)-C(6)-C(19)	117.43(14)
C(7)-C(6)-P(1)	113.67(11)
C(5)-C(6)-P(1)	105.60(11)

C(19)-C(6)-P(1)	110.55(11)
C(8)-C(7)-O(2)	122.77(15)
C(8)-C(7)-C(6)	126.23(15)
O(2)-C(7)-C(6)	110.55(13)
C(7)-C(8)-C(25)	116.94(15)
C(7)-C(8)-C(9)	121.75(16)
C(25)-C(8)-C(9)	121.30(15)
C(27)-C(9)-C(10)	117.55(17)
C(27)-C(9)-C(8)	121.24(16)
C(10)-C(9)-C(8)	121.19(16)
C(11)-C(10)-C(9)	121.12(18)
C(11)-C(10)-H(10)	119.4
C(9)-C(10)-H(10)	119.4
C(12)-C(11)-C(10)	120.35(19)
C(12)-C(11)-H(11)	119.8
C(10)-C(11)-H(11)	119.8
C(11)-C(12)-C(26)	119.65(18)
C(11)-C(12)-H(12)	120.2
C(26)-C(12)-H(12)	120.2
C(14)-C(13)-C(18)	120.7(2)
C(14)-C(13)-H(13)	119.7
C(18)-C(13)-H(13)	119.7
C(13)-C(14)-C(15)	120.4(2)
C(13)-C(14)-H(14)	119.8
C(15)-C(14)-H(14)	119.8
C(14)-C(15)-C(16)	119.7(2)
C(14)-C(15)-H(15)	120.2
C(16)-C(15)-H(15)	120.2
C(17)-C(16)-C(15)	119.59(19)
C(17)-C(16)-P(1)	125.15(15)
C(15)-C(16)-P(1)	115.13(16)
C(16)-C(17)-C(18)	119.9(2)
C(16)-C(17)-H(17)	120.1
C(18)-C(17)-H(17)	120.1
C(13)-C(18)-C(17)	119.9(3)
C(13)-C(18)-H(18)	120.1
C(17)-C(18)-H(18)	120.1
C(20)-C(19)-C(6)	112.51(14)

C(20)-C(19)-H(19A)	109.1
C(6)-C(19)-H(19A)	109.1
C(20)-C(19)-H(19B)	109.1
C(6)-C(19)-H(19B)	109.1
H(19A)-C(19)-H(19B)	107.8
C(21)-C(20)-C(25)	119.65(17)
C(21)-C(20)-C(19)	119.77(17)
C(25)-C(20)-C(19)	120.50(15)
C(20)-C(21)-C(22)	120.9(2)
C(20)-C(21)-H(21)	119.6
C(22)-C(21)-H(21)	119.6
C(23)-C(22)-C(21)	119.73(19)
C(23)-C(22)-H(22)	120.1
C(21)-C(22)-H(22)	120.1
C(22)-C(23)-C(24)	120.5(2)
C(22)-C(23)-H(23)	119.8
C(24)-C(23)-H(23)	119.8
C(23)-C(24)-C(25)	120.9(2)
C(23)-C(24)-H(24)	119.5
C(25)-C(24)-H(24)	119.5
C(24)-C(25)-C(20)	118.31(17)
C(24)-C(25)-C(8)	121.94(17)
C(20)-C(25)-C(8)	119.73(15)
C(27)-C(26)-C(12)	120.01(19)
C(27)-C(26)-H(26)	120.0
C(12)-C(26)-H(26)	120.0
C(26)-C(27)-C(9)	121.30(18)
C(26)-C(27)-H(27)	119.3
C(9)-C(27)-H(27)	119.3
C(5)-C(28)-O(2)	113.63(15)
C(5)-C(28)-C(29)	124.09(16)
O(2)-C(28)-C(29)	122.27(15)
C(30)-C(29)-C(28)	116.77(17)
C(30)-C(29)-H(29)	121.6
C(28)-C(29)-H(29)	121.6
C(29)-C(30)-C(3)	122.18(17)
C(29)-C(30)-H(30)	118.9
C(3)-C(30)-H(30)	118.9

C(32)-C(31)-C(1)	120.32(19)
C(32)-C(31)-H(31)	119.8
C(1)-C(31)-H(31)	119.8
C(31)-C(32)-C(4)	121.03(18)
C(31)-C(32)-H(32)	119.5
C(4)-C(32)-H(32)	119.5
C(38)-C(33)-C(34)	119.05(18)
C(38)-C(33)-P(1)	126.77(14)
C(34)-C(33)-P(1)	114.17(15)
C(35)-C(34)-C(33)	120.1(2)
C(35)-C(34)-H(34)	120.0
C(33)-C(34)-H(34)	120.0
C(36)-C(35)-C(34)	120.1(2)
C(36)-C(35)-H(35)	119.9
C(34)-C(35)-H(35)	119.9
C(37)-C(36)-C(35)	120.3(2)
C(37)-C(36)-H(36)	119.8
C(35)-C(36)-H(36)	119.8
C(36)-C(37)-C(38)	119.8(2)
C(36)-C(37)-H(37)	120.1
C(38)-C(37)-H(37)	120.1
C(33)-C(38)-C(37)	120.62(19)
C(33)-C(38)-H(38)	119.7
C(37)-C(38)-H(38)	119.7

Symmetry transformations used to generate equivalent atoms:

Table 3. Hydrogen bonds for Ly200524_a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(34)-H(34)...O(1)	0.93	2.44	2.904(2)	110.8

Symmetry transformations used to generate equivalent atoms:

The crystal of **6c** (CCDC 2249732) was crystallized by slow vapor diffusion of CH₂Cl₂ layered with petroleum ether at room temperature for several days.

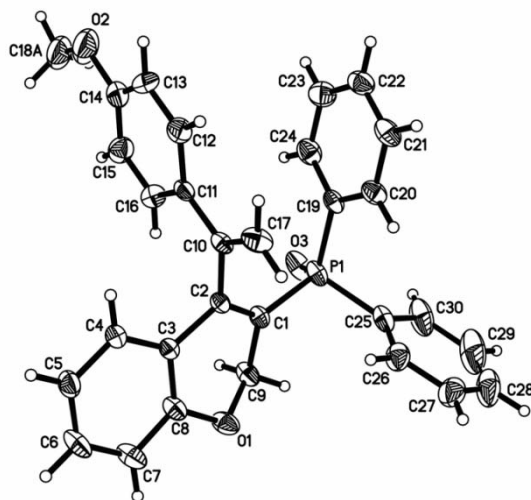


Figure S4. X-ray structures of **6c** (ellipsoids set at 50 % probability).

Table 1. Crystal data and structure refinement for Ly201215_a.

Identification code	Ly201215_a	
Empirical formula	C ₃₀ H ₂₇ O ₄ P	
Formula weight	482.48	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.9061(16) Å	α = 74.291(5)°.
	b = 10.4242(18) Å	β = 76.463(5)°.
	c = 14.312(3) Å	γ = 86.731(5)°.
Volume	1243.6(4) Å ³	
Z	2	
Density (calculated)	1.289 Mg/m ³	
Absorption coefficient	0.145 mm ⁻¹	
F(000)	508	
Crystal size	0.220 x 0.200 x 0.180 mm ³	
Theta range for data collection	1.518 to 24.996°.	
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16	
Reflections collected	15418	

Independent reflections	4344 [R(int) = 0.0382]
Completeness to theta = 24.996°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.6641
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4344 / 16 / 329
Goodness-of-fit on F ²	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0708, wR2 = 0.1859
R indices (all data)	R1 = 0.1135, wR2 = 0.2153
Extinction coefficient	n/a
Largest diff. peak and hole	0.708 and -0.495 e.Å ⁻³

Table 2. Bond lengths [Å] and angles [°] for Ly201215_a.

C(1)-C(2)	1.347(4)
C(1)-C(9)	1.517(5)
C(1)-P(1)	1.800(3)
C(2)-C(3)	1.482(4)
C(2)-C(10)	1.497(4)
C(3)-C(8)	1.384(5)
C(3)-C(4)	1.395(5)
C(4)-C(5)	1.380(5)
C(4)-H(4)	0.9300
C(5)-C(6)	1.356(6)
C(5)-H(5)	0.9300
C(6)-C(7)	1.381(6)
C(6)-H(6)	0.9300
C(7)-C(8)	1.380(5)
C(7)-H(7)	0.9300
C(8)-O(1)	1.383(4)
C(9)-O(1)	1.395(5)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(17)	1.315(5)
C(10)-C(11)	1.477(5)
C(11)-C(16)	1.383(5)
C(11)-C(12)	1.389(5)
C(12)-C(13)	1.401(7)

C(12)-H(12)	0.9300
C(13)-C(14)	1.346(7)
C(13)-H(13)	0.9300
C(14)-C(15)	1.331(6)
C(14)-O(2)	1.416(6)
C(15)-C(16)	1.382(6)
C(15)-H(15)	0.9300
C(16)-H(16)	0.9300
C(17)-H(17A)	0.9300
C(17)-H(17B)	0.9300
C(19)-C(24)	1.374(6)
C(19)-C(20)	1.383(5)
C(19)-P(1)	1.796(4)
C(20)-C(21)	1.376(5)
C(20)-H(20)	0.9300
C(21)-C(22)	1.366(6)
C(21)-H(21)	0.9300
C(22)-C(23)	1.362(6)
C(22)-H(22)	0.9300
C(23)-C(24)	1.374(6)
C(23)-H(23)	0.9300
C(24)-H(24)	0.9300
C(25)-C(26)	1.381(6)
C(25)-C(30)	1.385(6)
C(25)-P(1)	1.805(5)
C(26)-C(27)	1.391(7)
C(26)-H(26)	0.9300
C(27)-C(28)	1.345(9)
C(27)-H(27)	0.9300
C(28)-C(29)	1.362(11)
C(28)-H(28)	0.9300
C(29)-C(30)	1.400(10)
C(29)-H(29)	0.9300
C(30)-H(30)	0.9300
O(2)-C(18A)	1.394(8)
O(2)-C(18B)	1.405(9)
O(3)-P(1)	1.489(3)
O(4)-H(4A)	0.8499

O(4)-H(4B)	0.8501
C(18A)-C(18A)#1	1.877(18)
C(18A)-H(18A)	0.9600
C(18A)-H(18B)	0.9600
C(18A)-H(18C)	0.9600
C(18B)-H(18D)	0.9601
C(18B)-H(18E)	0.9600
C(18B)-H(18F)	0.9600
C(2)-C(1)-C(9)	117.8(3)
C(2)-C(1)-P(1)	130.9(3)
C(9)-C(1)-P(1)	110.6(2)
C(1)-C(2)-C(3)	117.3(3)
C(1)-C(2)-C(10)	125.3(3)
C(3)-C(2)-C(10)	117.4(3)
C(8)-C(3)-C(4)	117.6(3)
C(8)-C(3)-C(2)	118.9(3)
C(4)-C(3)-C(2)	123.4(3)
C(5)-C(4)-C(3)	120.9(4)
C(5)-C(4)-H(4)	119.6
C(3)-C(4)-H(4)	119.6
C(6)-C(5)-C(4)	120.2(4)
C(6)-C(5)-H(5)	119.9
C(4)-C(5)-H(5)	119.9
C(5)-C(6)-C(7)	120.7(4)
C(5)-C(6)-H(6)	119.6
C(7)-C(6)-H(6)	119.6
C(8)-C(7)-C(6)	119.0(4)
C(8)-C(7)-H(7)	120.5
C(6)-C(7)-H(7)	120.5
C(7)-C(8)-O(1)	117.5(4)
C(7)-C(8)-C(3)	121.6(4)
O(1)-C(8)-C(3)	120.7(3)
O(1)-C(9)-C(1)	113.4(3)
O(1)-C(9)-H(9A)	108.9
C(1)-C(9)-H(9A)	108.9
O(1)-C(9)-H(9B)	108.9
C(1)-C(9)-H(9B)	108.9

H(9A)-C(9)-H(9B)	107.7
C(17)-C(10)-C(11)	124.2(3)
C(17)-C(10)-C(2)	119.2(3)
C(11)-C(10)-C(2)	116.7(3)
C(16)-C(11)-C(12)	114.6(4)
C(16)-C(11)-C(10)	122.1(3)
C(12)-C(11)-C(10)	123.3(4)
C(11)-C(12)-C(13)	121.4(4)
C(11)-C(12)-H(12)	119.3
C(13)-C(12)-H(12)	119.3
C(14)-C(13)-C(12)	121.0(4)
C(14)-C(13)-H(13)	119.5
C(12)-C(13)-H(13)	119.5
C(15)-C(14)-C(13)	119.1(5)
C(15)-C(14)-O(2)	123.6(6)
C(13)-C(14)-O(2)	117.3(5)
C(14)-C(15)-C(16)	120.9(5)
C(14)-C(15)-H(15)	119.5
C(16)-C(15)-H(15)	119.5
C(15)-C(16)-C(11)	123.0(4)
C(15)-C(16)-H(16)	118.5
C(11)-C(16)-H(16)	118.5
C(10)-C(17)-H(17A)	120.0
C(10)-C(17)-H(17B)	120.0
H(17A)-C(17)-H(17B)	120.0
C(24)-C(19)-C(20)	118.0(4)
C(24)-C(19)-P(1)	118.3(3)
C(20)-C(19)-P(1)	123.7(3)
C(21)-C(20)-C(19)	120.5(4)
C(21)-C(20)-H(20)	119.7
C(19)-C(20)-H(20)	119.7
C(22)-C(21)-C(20)	120.7(4)
C(22)-C(21)-H(21)	119.6
C(20)-C(21)-H(21)	119.6
C(23)-C(22)-C(21)	119.1(4)
C(23)-C(22)-H(22)	120.4
C(21)-C(22)-H(22)	120.4
C(22)-C(23)-C(24)	120.6(4)

C(22)-C(23)-H(23)	119.7
C(24)-C(23)-H(23)	119.7
C(23)-C(24)-C(19)	121.0(4)
C(23)-C(24)-H(24)	119.5
C(19)-C(24)-H(24)	119.5
C(26)-C(25)-C(30)	118.9(5)
C(26)-C(25)-P(1)	122.7(3)
C(30)-C(25)-P(1)	118.4(4)
C(25)-C(26)-C(27)	120.6(5)
C(25)-C(26)-H(26)	119.7
C(27)-C(26)-H(26)	119.7
C(28)-C(27)-C(26)	120.1(7)
C(28)-C(27)-H(27)	119.9
C(26)-C(27)-H(27)	119.9
C(27)-C(28)-C(29)	120.5(7)
C(27)-C(28)-H(28)	119.7
C(29)-C(28)-H(28)	119.7
C(28)-C(29)-C(30)	120.6(6)
C(28)-C(29)-H(29)	119.7
C(30)-C(29)-H(29)	119.7
C(25)-C(30)-C(29)	119.2(7)
C(25)-C(30)-H(30)	120.4
C(29)-C(30)-H(30)	120.4
C(8)-O(1)-C(9)	113.4(3)
C(18A)-O(2)-C(14)	123.3(7)
C(18B)-O(2)-C(14)	131.6(10)
H(4A)-O(4)-H(4B)	104.5
O(3)-P(1)-C(19)	111.39(18)
O(3)-P(1)-C(1)	108.32(15)
C(19)-P(1)-C(1)	114.06(16)
O(3)-P(1)-C(25)	111.69(19)
C(19)-P(1)-C(25)	107.13(17)
C(1)-P(1)-C(25)	104.05(18)
O(2)-C(18A)-C(18A)#1	175.9(11)
O(2)-C(18A)-H(18A)	110.0
C(18A)#1-C(18A)-H(18A)	66.0
O(2)-C(18A)-H(18B)	109.7
C(18A)#1-C(18A)-H(18B)	73.3

H(18A)-C(18A)-H(18B)	109.5
O(2)-C(18A)-H(18C)	108.7
C(18A)#1-C(18A)-H(18C)	72.3
H(18A)-C(18A)-H(18C)	109.5
H(18B)-C(18A)-H(18C)	109.5
O(2)-C(18B)-H(18D)	109.5
O(2)-C(18B)-H(18E)	113.0
H(18D)-C(18B)-H(18E)	109.5
O(2)-C(18B)-H(18F)	105.8
H(18D)-C(18B)-H(18F)	109.5
H(18E)-C(18B)-H(18F)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1

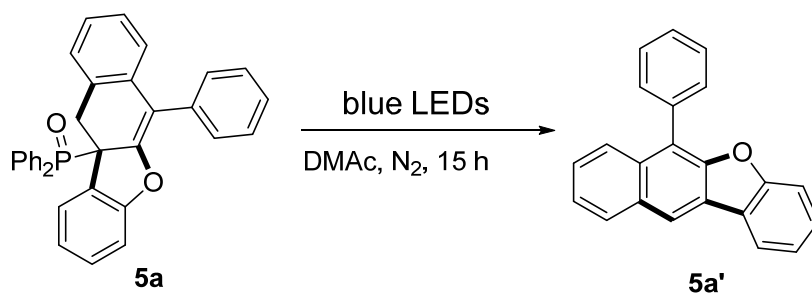
Table 3. Hydrogen bonds for Ly201215_a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(18B ^{^b})-H(18F ^{^b})...O(4)#2	0.96	2.57	3.359(18)	139.1

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1 #2 -x,-y+1,-z+1

9. Derivatization of Product 5a

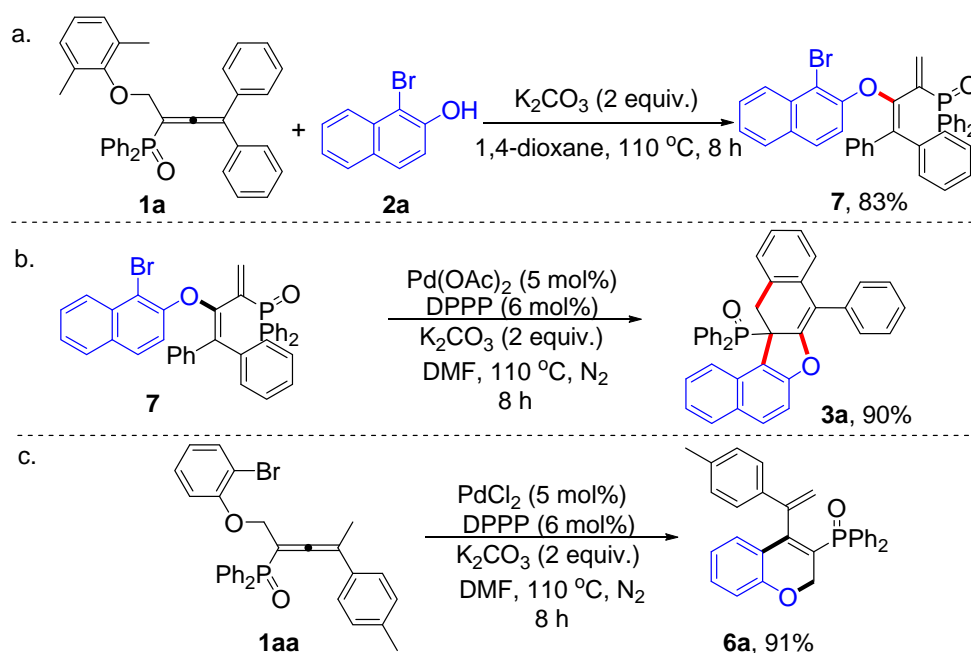


5a (100 mg, 0.2 mmol) was added into a 25 mL Schlenk tube. The vessel was evacuated and backfilled with N₂ three times, DMAc (2 mL) was added via a syringe. Then, the reaction was stirred at room temperature under the irradiation of blue LED (450 ± 5 nm, 25 W) for 20 h. Upon completion of the reaction, water (30.0 mL) was added and

extracted with EtOAc twice. Combined organic layer was dried over anhydrous Na₂SO₄, and the solvent was removed under reduced pressure to afford the crude product, which was further purified by flash column chromatography (ethyl acetate/petroleum ether = 1/50) to afford the desired product **5a'** in 81% yield (47.6 mg).

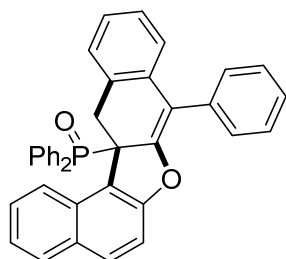
5a': A white solid, ¹H NMR (500 MHz, CDCl₃) δ 8.44 (s, 1H), 8.10-8.07 (m, 2H), 7.94 (d, *J* = 10.0 Hz, 1H), 7.65-7.60 (m, 4H), 7.55-7.45 (m, 5H), 7.38-7.35 (m, 1H).

10. Mechanistic Studies



11. Characterizations of Products

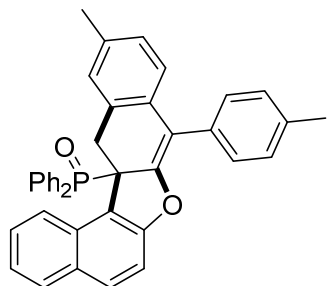
diphenyl(8-phenyldinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)phosphine oxide (**3a**)



A white solid, (80 mg, 73% yield). ¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 8.0 Hz, 1H), 7.60–7.44 (m, 6H), 7.41–7.35 (m, 5H), 7.29–7.27 (m, 2H), 7.19–7.15 (m, 1H), 7.03–6.98 (m, 1H), 6.96–6.90 (m, 5H), 6.88–6.83 (m, 2H), 6.66–6.62 (m, 1H), 6.18 (d,

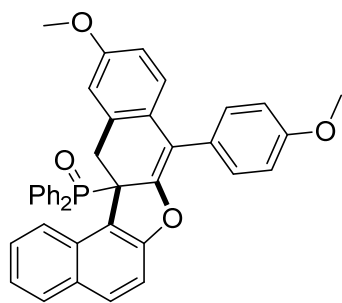
$J = 8.0$ Hz, 1H), 4.71–6.65 (m, 1H), 3.64–3.52 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.13 (d, $J = 6.1$ Hz), 153.76 (d, $J = 5.1$ Hz), 136.18 (d, $J = 5.1$ Hz), 134.90 (d, $J = 5.1$ Hz), 131.25 (d, $J = 9.1$ Hz), 131.07, 130.72, 130.69 (d, $J = 206.0$ Hz), 130.56, 130.40 (dd, $J = 11.1, 3.0$ Hz), 130.13, 130.04, 128.49, 128.38, 128.30, 127.57, 127.43, 127.27, 127.15, 126.59, 125.70, 124.57 (d, $J = 105.0$ Hz), 120.87 (d, $J = 4.0$ Hz), 116.44 (d, $J = 11.1$ Hz), 111.34 (d, $J = 3.0$ Hz), 55.55 (d, $J = 55.6$ Hz), 35.72. ^{31}P NMR (162 MHz, CDCl_3) δ 29.71. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{38}\text{H}_{28}\text{O}_2\text{P}^+$: 547.1821, Found: 547.1816.

(11-methyl-8-(p-tolyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3b)



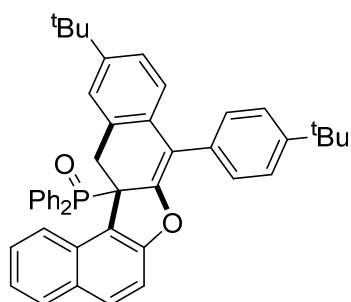
A white solid, (59 mg, 51% yield) ^1H NMR (400 MHz, CDCl_3) δ 8.59 (d, $J = 8.0$ Hz, 1H), 7.72–7.62 (m, 5H), 7.61–7.53 (m, 2H), 7.42 (d, $J = 8.0$ Hz, 2H), 7.34–7.28 (m, 3H), 7.24 (s, 1H), 7.17–7.05 (m, 5H), 7.03–6.99 (m, 2H), 6.59 (d, $J = 8.0$ Hz, 1H), 6.21 (d, $J = 8.0$ Hz, 1H), 4.77–4.70 (m, 1H), 3.74–3.62 (m, 1H), 2.48 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.21 (d, $J = 5.8$ Hz), 152.63 (d, $J = 5.8$ Hz), 137.23, 136.26, 133.48 (d, $J = 4.7$ Hz), 131.96 (d, $J = 4.3$ Hz), 131.21 (d, $J = 8.5$ Hz), 131.00 (d, $J = 2.9$ Hz), 130.60, 130.27 (d, $J = 110.1$ Hz), 130.06 (d, $J = 8.5$ Hz), 129.92 (d, $J = 3.3$ Hz), 129.10, 129.06, 128.43, 127.81, 127.33 (d, $J = 11.5$ Hz), 127.15, 127.04 (d, $J = 11.7$ Hz), 125.66, 125.09, 123.93, 120.84 (d, $J = 4.0$ Hz), 116.32 (d, $J = 11.2$ Hz), 111.39 (d, $J = 2.6$ Hz), 55.49 (d, $J = 55.9$ Hz), 35.77, 21.44, 21.08. ^{31}P NMR (162 MHz, CDCl_3) δ 29.71. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{40}\text{H}_{31}\text{NaO}_2\text{P}^+$: 597.1954, Found: 597.1949.

(11-methoxy-8-(4-methoxyphenyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3c)



A white solid, (59 mg, 49% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.55 (d, $J = 5.0$ Hz, 1H), 7.73–7.69 (m, 2H), 7.64–7.58 (m, 3H), 7.56–7.53 (m, 1H), 7.49 (d, $J = 10.0$ Hz, 1H), 7.43 (d, $J = 10.0$ Hz, 2H), 7.28–7.25 (m, 1H), 7.14–7.11 (m, 1H), 7.09–7.04 (m, 3H), 7.03–7.01 (m, 3H), 6.99–6.97 (m, 3H), 6.31 (d, $J = 10.0$ Hz, 1H), 6.22 (d, $J = 10.0$ Hz, 1H), 4.73–4.68 (m, 1H), 3.87 (s, 3H), 3.82 (s, 3H), 3.70–3.60 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 158.77 (d, $J = 58.0$ Hz), 157.36 (d, $J = 6.3$ Hz), 151.37 (d, $J = 6.3$ Hz), 132.51 (d, $J = 5.0$ Hz), 131.73, 131.22, 131.16, 130.81, 130.56, 130.46 (d, $J = 126.0$ Hz), 130.31, 130.12 (d, $J = 7.6$ Hz), 129.28 (d, $J = 5.0$ Hz), 128.53, 127.42 (d, $J = 11.3$ Hz), 127.22, 127.13, 126.88, 125.11, 123.98, 120.79 (d, $J = 3.8$ Hz), 115.99 (d, $J = 11.3$ Hz), 113.87, 113.01, 111.46 (d, $J = 2.5$ Hz), 55.58, 55.42 (d, $J = 56.7$ Hz), 55.40, 36.13. $^{31}\text{P NMR}$ (202 MHz, CDCl_3) δ 30.08. **HRMS (ESI):** ($[\text{M} + \text{Na}]^+$) Calcd for $\text{C}_{40}\text{H}_{31}\text{NaO}_4\text{P}^+$: 629.1852, Found: 629.1848.

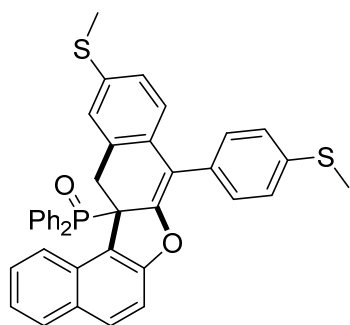
(11-(tert-butyl)-8-(4-(tert-butyl)phenyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3d)



A white solid, (79 mg, 60% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.61 (d, $J = 5.0$ Hz, 1H), 7.69–7.66 (m, 2H), 7.64–7.57 (m, 4H), 7.52–7.47 (m, 5H), 7.43 (s, 1H), 7.31–7.28 (m, 1H), 7.12–7.09 (m, 1H), 7.07–7.02 (m, 4H), 7.00–6.96 (m, 2H), 6.77 (d, $J = 10.0$ Hz, 1H), 6.29 (d, $J = 5.0$ Hz, 1H), 4.78–4.73 (m, 1H), 3.74–3.65 (m, 1H), 1.42 (s, 9H), 1.37 (s, 9H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 157.35 (d, $J = 5.7$ Hz), 152.92 (d, $J = 5.7$ Hz), 149.96 (d, $J = 98.8$ Hz), 133.56 (d, $J = 4.5$ Hz), 131.88, 131.22 (d, $J = 8.4$ Hz),

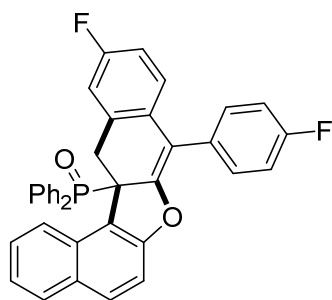
131.07, 130.87 (d, $J = 238.1$ Hz), 130.56, 130.34, 130.10 (d, $J = 8.4$ Hz), 129.75, 128.51, 127.42 (d, $J = 11.4$ Hz), 127.27, 127.19 (d, $J = 3.4$ Hz), 125.58 (d, $J = 11.4$ Hz), 125.29, 124.07 (d, $J = 13.6$ Hz), 120.84 (d, $J = 4.0$ Hz), 116.38 (d, $J = 11.3$ Hz), 111.55, 55.83 (d, $J = 55.8$ Hz), 36.19, 34.80, 34.61, 31.54, 31.48. ^{31}P NMR (202 MHz, CDCl_3) δ 29.99. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{46}\text{H}_{43}\text{NaO}_2\text{P}^+$: 681.2893, Found: 681.2879.

(11-(methylthio)-8-(4-(methylthio)phenyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3e)



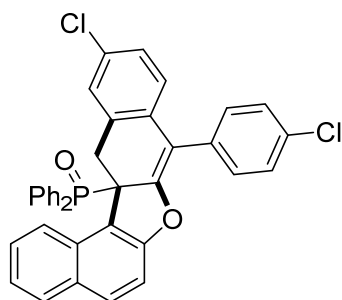
A white solid, (47 mg, 37% yield). ^1H NMR (500 MHz, CDCl_3) δ 8.52 (d, $J = 10.0$ Hz, 1H), 7.68–7.64 (m, 2H), 7.61–7.57 (m, 3H), 7.55–7.50 (m, 2H), 7.41 (d, $J = 5.0$ Hz, 2H), 7.35 (d, $J = 10.0$ Hz, 2H), 7.30–7.27 (m, 2H), 7.14–7.12 (m, 1H), 7.08–7.05 (m, 3H), 7.02 (d, $J = 10.0$ Hz, 1H), 6.99–6.96 (m, 2H), 6.65 (d, $J = 5.0$ Hz, 1H), 6.18 (d, $J = 10.0$ Hz, 1H), 4.74–4.69 (m, 1H), 3.68–3.58 (m, 1H), 2.55 (s, 3H), 2.52 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 157.15 (d, $J = 6.3$ Hz), 153.18, 137.94, 136.66, 133.27, 131.62, 131.29, 131.22, 130.90, 130.70, 130.55 (d, $J = 228.1$ Hz), 130.44, 130.06 (d, $J = 8.8$ Hz), 128.55, 127.48 (d, $J = 11.3$ Hz), 127.33, 127.23, 126.43, 126.30, 125.90, 125.07, 124.14, 120.69, 115.70 (d, $J = 10.1$ Hz), 111.35, 55.62 (d, $J = 55.4$ Hz), 35.78, 16.23, 15.76. ^{31}P NMR (202 MHz, CDCl_3) δ 30.23. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{40}\text{H}_{31}\text{NaO}_2\text{PS}_2^+$: 661.1395, Found: 661.1392.

(11-fluoro-8-(4-fluorophenyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3f)



A white solid, (68 mg, 58% yield). **¹H NMR** (400 MHz, CDCl₃) δ 8.53 (d, *J* = 8.0 Hz, 1H), 7.75–7.70 (m, 2H), 7.64–7.54 (m, 5H), 7.49–7.45 (m, 2H), 7.34–7.30 (m, 1H), 7.23–7.19 (m, 3H), 7.16–7.08 (m, 4H), 7.07–6.98 (m, 3H), 6.52–6.48 (m, 1H), 6.25–6.22 (m, 1H), 4.79–4.73 (m, 1H), 3.72–3.60 (m, 1H). **¹³C NMR** (151 MHz, CDCl₃) δ 162.65 (d, *J* = 96.6 Hz), 161.01 (d, *J* = 96.6 Hz), 156.97 (d, *J* = 4.5 Hz), 153.35 (d, *J* = 6.0 Hz), 133.22 (dd, *J* = 9.1, 4.5 Hz), 132.18, 131.59 (dd, *J* = 7.6, 3.0 Hz), 131.20, 131.14, 130.85 (dd, *J* = 31.7, 3.0 Hz), 130.51 (dd, *J* = 9.1, 4.5 Hz), 130.34 (d, *J* = 4.5 Hz), 129.99 (d, *J* = 7.6 Hz), 128.49, 127.43, 127.36, 127.27 (d, *J* = 12.1 Hz), 126.79 (d, *J* = 9.1 Hz), 124.91, 124.13, 120.46 (d, *J* = 3.0 Hz), 115.53 (d, *J* = 22.7 Hz), 115.46 (d, *J* = 21.1 Hz), 114.75 (d, *J* = 10.6 Hz), 113.77 (d, *J* = 21.1 Hz), 111.18, 55.35 (d, *J* = 55.9 Hz), 35.59. **³¹P NMR** (162 MHz, CDCl₃) δ 29.50. **¹⁹F NMR** (376 MHz, CDCl₃) δ -113.80 (s), -115.38 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₈H₂₅F₂NaO₂P⁺: 605.1452, Found: 605.1450.

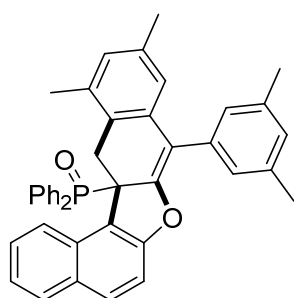
(11-chloro-8-(4-chlorophenyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3g)



A white solid, (81 mg, 66% yield). **¹H NMR** (400 MHz, CDCl₃) δ 8.53 (d, *J* = 8.0 Hz, 1H), 7.72–7.68 (m, 2H), 7.65–7.55 (m, 5H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.44–7.42 (m, 3H), 7.34–7.31 (m, 1H), 7.22–7.18 (m, 1H), 7.16–7.10 (m, 3H), 7.07–6.99 (m, 3H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.19 (d, *J* = 8.0 Hz, 1H), 4.78–4.72 (m, 1H), 3.70–3.58 (m, 1H). **¹³C NMR** (101 MHz, CDCl₃) δ 156.87 (d, *J* = 6.1 Hz), 154.38 (d, *J* = 6.1 Hz),

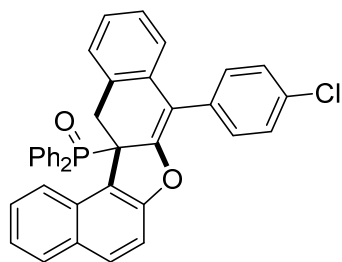
134.35 (d, $J = 5.1$ Hz), 133.48, 132.79 (d, $J = 4.0$ Hz), 132.66 (d, $J = 5.1$ Hz), 132.09, 131.26, 131.18, 130.93 (dd, $J = 18.2, 3.0$ Hz), 130.38, 130.23, 129.97 (d, $J = 8.1$ Hz), 128.74, 128.53, 128.40, 127.40 (d, $J = 25.3$ Hz), 127.40, 127.33, 126.46, 124.91, 124.24, 120.42, 114.56 (d, $J = 10.1$ Hz), 111.14, 55.52 (d, $J = 54.5$ Hz), 35.34. **^{31}P NMR** (162 MHz, CDCl_3) δ 29.60. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{38}\text{H}_{25}\text{Cl}_2\text{NaO}_2\text{P}^+$: 637.0861, Found: 637.0856.

(8-(3,5-dimethylphenyl)-10,12-dimethyldinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3h)



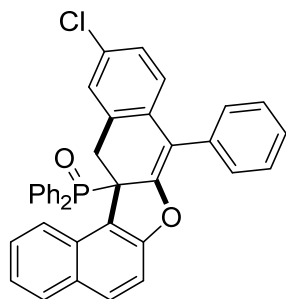
A white solid, (60 mg, 50% yield). **^1H NMR** (500 MHz, CDCl_3) δ 8.61 (d, $J = 5.0$ Hz, 1H), 7.63–7.55 (m, 6H), 7.52 (d, $J = 10.0$ Hz, 1H), 7.29–7.26 (m, 1H), 7.18–7.15 (m, 1H), 7.09–7.04 (m, 7H), 7.00–6.96 (m, 2H), 6.75 (s, 1H), 5.83 (s, 1H), 4.92–4.87 (m, 1H), 3.38–3.28 (m, 1H), 2.61 (s, 3H), 2.42 (s, 6H), 1.97 (s, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 157.33 (d, $J = 5.0$ Hz), 153.39 (d, $J = 6.3$ Hz), 137.74, 136.74 (d, $J = 3.8$ Hz), 135.76 (d, $J = 65.5$ Hz), 135.28 (d, $J = 5.0$ Hz), 131.48 (d, $J = 95.8$ Hz), 131.01 (d, $J = 2.5$ Hz), 130.84 (d, $J = 8.8$ Hz), 130.53, 130.34, 130.01 (d, $J = 8.8$ Hz), 129.89 (d, $J = 3.8$ Hz), 129.47 (d, $J = 44.1$ Hz), 128.52, 127.90, 127.46, 127.34 (d, $J = 6.3$ Hz), 127.17 (d, $J = 11.3$ Hz), 126.06 (d, $J = 3.8$ Hz), 125.09, 124.74, 124.00, 120.95, 116.67 (d, $J = 11.3$ Hz), 111.61 (d, $J = 2.5$ Hz), 55.79 (d, $J = 55.4$ Hz), 31.40, 21.63, 20.87, 20.31. **^{31}P NMR** (202 MHz, CDCl_3) δ 31.11. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{42}\text{H}_{35}\text{NaO}_2\text{P}^+$: 625.2267, Found: 625.2259.

(8-(4-chlorophenyl)dinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3i-1)



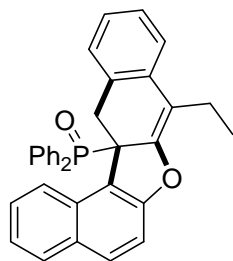
A white solid, (38 mg, 33% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.51 (d, $J = 10.0$ Hz, 1H), 7.62–7.51 (m, 7H), 7.46–7.38 (m, 5H), 7.30–7.27 (m, 1H), 7.14–7.11 (m, 1H), 7.07–7.02 (m, 5H), 6.97–6.95 (m, 2H), 7.78–6.75 (m, 1H), 6.23 (d, $J = 10.0$ Hz, 1H), 4.79–4.74 (m, 1H), 3.70–3.61 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 157.01 (d, $J = 6.3$ Hz), 154.21 (d, $J = 6.3$ Hz), 135.79, 133.32, 131.47, 131.16, 130.75, 130.42, 130.08 (d, $J = 8.8$ Hz), 128.68, 128.57, 128.47, 127.49, 127.40, 127.32, 127.23, 126.85, 125.49, 125.05, 124.21, 120.84, 115.36 (d, $J = 11.3$ Hz), 111.28, 55.61 (d, $J = 55.4$ Hz), 35.69. $^{31}\text{P NMR}$ (202 MHz, CDCl_3) δ 30.47. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{38}\text{H}_{26}\text{ClNaO}_2\text{P}^+$: 603.1251, Found: 603.1241.

(11-chloro-8-phenyldinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3i-2)



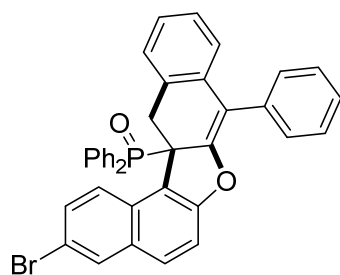
A white solid, (30 mg, 26% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.52 (d, $J = 10.0$ Hz, 1H), 7.72–7.68 (m, 2H), 7.62–7.45 (m, 9H), 7.41–7.38 (m, 1H), 7.37 (s, 1H), 7.31–7.28 (m, 1H), 7.17–7.14 (m, 1H), 7.12–7.07 (m, 3H), 7.02–6.97 (m, 3H), 6.71 (d, $J = 5.0$ Hz, 1H), 6.20 (d, $J = 10.0$ Hz, 1H), 4.75–4.70 (m, 1H), 3.68–3.59 (m, 1H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 157.08, 154.01, 134.66 (d, $J = 47.9$ Hz), 132.73, 131.93, 131.41, 131.34, 131.05, 130.81 (d, $J = 2.5$ Hz), 130.40, 130.11, 130.04, 128.55, 128.34, 127.83, 127.44 (d, $J = 32.8$ Hz), 127.43, 127.41 (d, $J = 17.6$ Hz), 126.79, 125.05, 124.21, 120.52, 115.76 (d, $J = 11.3$ Hz), 111.35 (d, $J = 2.5$ Hz), 55.51 (d, $J = 55.4$ Hz), 35.46. $^{31}\text{P NMR}$ (202 MHz, CDCl_3) δ 30.24. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{38}\text{H}_{26}\text{ClNaO}_2\text{P}^+$: 603.1251, Found: 603.1242.

(8-ethylidnaptho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3j)



A white solid, (32 mg, 32% yield). **¹H NMR** (500 MHz, CDCl₃) δ 8.52 (d, *J* = 5.0 Hz, 1H), 7.62–7.58 (m, 3H), 7.55–7.51 (m, 4H), 7.30–7.27 (m, 2H), 7.18–7.15 (m, 1H), 7.09–7.03 (m, 4H), 7.01–6.98 (m, 1H), 6.96–6.93 (m, 2H), 6.86–6.83 (m, 1H), 6.58 (d, *J* = 5.0 Hz, 1H), 4.73–4.68 (m, 1H), 3.61–3.51 (m, 1H), 2.70–2.57 (m, 2H), 1.19 (t, *J* = 7.5 Hz, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 157.08 (d, *J* = 5.0 Hz), 152.83 (d, *J* = 5.0 Hz), 135.43 (d, *J* = 5.0 Hz), 131.53 (d, *J* = 7.6 Hz), 131.03, 130.69, 130.60, 130.18 (d, *J* = 8.8 Hz), 130.09 (d, *J* = 88.2 Hz), 128.50, 128.35, 127.34 (d, *J* = 11.3 Hz), 127.30 (d, *J* = 13.9 Hz), 127.12, 127.03, 126.17, 125.23, 123.98, 122.68, 121.17 (d, *J* = 3.8 Hz), 115.65 (d, *J* = 11.4 Hz), 111.21 (d, *J* = 2.5 Hz), 54.57 (d, *J* = 56.7 Hz), 35.75, 19.09 (d, *J* = 2.5 Hz), 13.48 (d, *J* = 6.3 Hz). **³¹P NMR** (202 MHz, CDCl₃) δ 30.22. **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₄H₂₈O₂P⁺: 499.1821, Found: 499.1815.

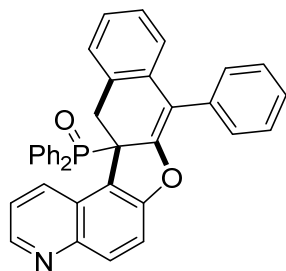
(3-bromo-8-phenyldinaphtho[2,1-b:2',3'-d]furan-13a(13H)-yl)diphenylphosphine oxide (3k)



A white solid, (30 mg, 24% yield). **¹H NMR** (400 MHz, CDCl₃) δ 8.51 (d, *J* = 8.0 Hz, 1H), 7.78 (s, 1H), 7.74–7.64 (m, 5H), 7.53–7.52 (m, 4H), 7.44–7.38 (m, 3H), 7.14–7.04 (m, 8H), 6.80–6.77 (m, 1H), 6.33 (d, *J* = 8.0 Hz, 1H), 4.78–4.72 (m, 1H), 3.75–3.63 (m, 1H). **¹³C NMR** (101 MHz, CDCl₃) δ 157.35 (d, *J* = 6.1 Hz), 153.22 (d, *J* = 6.1 Hz), 135.98 (d, *J* = 4.0 Hz), 134.69 (d, *J* = 4.0 Hz), 131.41, 131.23, 131.15, 130.83 (d, *J* = 3.0 Hz), 130.48, 130.43, 130.05, 129.96, 129.64 (d, *J* = 3.0 Hz), 128.88 (d, *J* = 3.0 Hz), 128.42, 128.24, 127.68, 127.58 (d, *J* = 11.1 Hz), 127.37, 127.26 (d, *J* = 12.1 Hz), 126.80

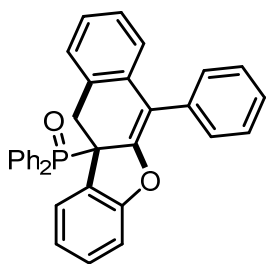
(d, $J = 17.2$ Hz), 125.79, 121.29 (d, $J = 4.0$ Hz), 117.86, 116.77 (d, $J = 11.1$ Hz), 112.44 (d, $J = 2.0$ Hz), 55.37 (d, $J = 55.6$ Hz), 35.61. ^{31}P NMR (162 MHz, CDCl_3) δ 29.62. **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{38}\text{H}_{26}\text{BrNaO}_2\text{P}^+$: 647.0746, Found: 647.0742.

diphenyl(8-phenylnaphtho[2',3':4,5]furo[3,2-f]quinolin-13a(13H)-yl)phosphine oxide (3l)



A white solid, (47 mg, 43% yield). ^1H NMR (400 MHz, CDCl_3) δ 8.96 (d, $J = 8.0$ Hz, 1H), 8.76 (d, $J = 4.0$ Hz, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.73–7.68 (m, 2H), 7.65–7.60 (m, 2H), 7.51–7.43 (m, 6H), 7.35 (d, $J = 8.0$ Hz, 1H), 7.24 (d, $J = 8.0$ Hz, 1H), 7.16–7.03 (m, 7H), 6.79–6.76 (m, 1H), 6.34 (d, $J = 4.0$ Hz, 1H), 4.74–4.68 (m, 1H), 3.78–3.66 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.07 (d, $J = 5.1$ Hz), 153.19 (d, $J = 5.1$ Hz), 148.55, 145.25, 135.89 (d, $J = 5.1$ Hz), 134.57 (d, $J = 4.0$ Hz), 133.36, 132.02, 131.45, 131.18 (d, $J = 9.1$ Hz), 130.60 (d, $J = 63.6$ Hz), 129.99 (d, $J = 4.0$ Hz), 129.88, 128.42, 128.16, 127.72 (d, $J = 3.0$ Hz), 127.41 (d, $J = 36.4$ Hz), 127.35, 126.76, 125.87, 125.78, 121.80, 120.87 (d, $J = 4.0$ Hz), 117.02 (d, $J = 11.1$ Hz), 114.58, 55.49 (d, $J = 56.6$ Hz), 35.62. ^{31}P NMR (162 MHz, CDCl_3) δ 28.08. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{37}\text{H}_{27}\text{NO}_2\text{P}^+$: 548.1774, Found: 548.1774.

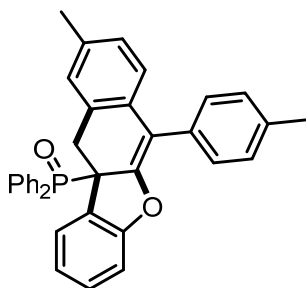
diphenyl(6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)phosphine oxide (5a)



A white solid, (87 mg, 76% yield). ^1H NMR (500 MHz, CDCl_3) δ 7.78–7.73 (m, 4H), 7.52–7.47 (m, 5H), 7.39–7.38 (m, 1H), 7.30–7.28 (m, 1H), 7.24–7.21 (m, 2H), 7.13–7.10

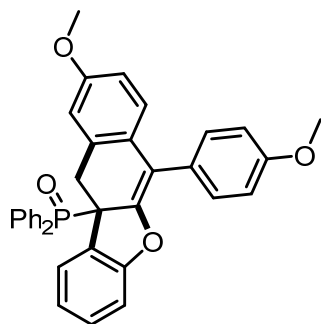
(m, 2H), 7.04-6.99 (m, 3H), 6.91-6.86 (m, 2H). 6.75-6.72 (m, 1H), 6.65 (d, $J = 10.0$ Hz, 1H), 6.46 (d, $J = 6.0$ Hz, 1H), 4.29-4.24 (m, 1H), 3.59-3.49 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.20 (d, $J = 5.0$ Hz), 152.99 (d, $J = 5.0$ Hz), 136.31 (d, $J = 3.8$ Hz), 134.92, 131.61 (d, $J = 8.8$ Hz), 131.52, 131.15 (d, $J = 3.8$ Hz), 130.89 (d, $J = 3.8$ Hz), 130.72, 130.65, 130.16, 130.04 (d, $J = 89.5$ Hz), 129.35, 128.49, 128.08, 127.96, 127.86, 127.69, 127.49 (d, $J = 12.6$ Hz), 127.17, 126.63, 126.08, 125.53 (d, $J = 3.8$ Hz), 122.77, 116.44 (d, $J = 10.1$ Hz), 110.08, 53.28 (d, $J = 58.0$ Hz), 34.97. ^{31}P NMR (202 MHz, CDCl_3) δ 27.13 (s). **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{34}\text{H}_{26}\text{O}_2\text{P}^+$: 497.1665, Found: 497.1657.

(9-methyl-6-(p-tolyl)naphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5b)



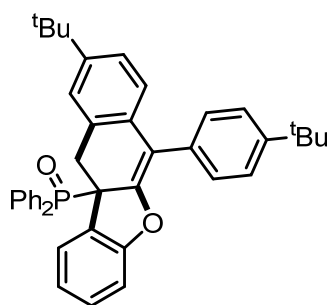
A white solid, (67 mg, 64% yield). ^1H NMR (500 MHz, CDCl_3) δ 7.78-7.72 (m, 4H), 7.51 (d, $J = 10.0$ Hz, 1H), 7.37 (d, $J = 10.0$ Hz, 2H), 7.31-7.28 (m, 3H), 7.25-7.21 (m, 2H), 7.13-7.10 (m, 1H), 7.04-6.99 (m, 3H), 6.90 (s, 1H), 6.89-6.86 (m, 1H), 6.64 (d, $J = 10.0$ Hz, 1H), 6.54 (d, $J = 10.0$ Hz, 1H), 6.38 (d, $J = 10.0$ Hz, 1H), 4.19-4.14 (m, 1H), 3.55-3.46 (m, 1H), 2.43 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.33 (d, $J = 3.8$ Hz), 151.93 (d, $J = 5.0$ Hz), 137.36, 136.22, 133.59 (d, $J = 5.0$ Hz), 132.02 (d, $J = 3.8$ Hz), 131.65 (d, $J = 8.8$ Hz), 131.49, 131.01 (d, $J = 2.5$ Hz), 130.74, 130.68, 129.98 (d, $J = 3.8$ Hz), 129.18, 128.99, 127.87 (d, $J = 11.3$ Hz), 127.69, 127.30 (d, $J = 11.3$ Hz), 126.08, 125.63 (d, $J = 3.8$ Hz), 122.59 (d, $J = 2.5$ Hz), 116.35 (d, $J = 11.3$ Hz), 110.02, 53.33 (d, $J = 59.2$ Hz), 35.08, 21.50, 21.01. ^{31}P NMR (202 MHz, CDCl_3) δ 26.87 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{36}\text{H}_{29}\text{NaO}_2\text{P}^+$: 547.1797, Found: 547.1793.

(9-methoxy-6-(4-methoxyphenyl)naphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5c)



A white solid, (68 mg, 61% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.81-7.76 (m, 4H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.35-7.31 (m, 1H), 7.28-7.23 (m, 2H), 7.17-7.14 (m, 1H), 7.10-7.01 (m, 5H), 6.92-6.88 (m, 1H), 6.69-6.65 (m, 2H), 6.45 (d, *J* = 8.0 Hz, 1H), 6.31 (d, *J* = 8.0 Hz, 1H), 4.21-4.14 (m, 1H), 3.90 (s, 3H), 3.75 (s, 3H), 3.60-3.47 (m, 1H). **¹³C NMR** (101 MHz, CDCl₃) δ 159.29 (d, *J* = 4.0 Hz), 158.94, 158.24, 150.53 (d, *J* = 6.1 Hz), 132.64 (d, *J* = 4.0 Hz), 131.54 (d, *J* = 8.1 Hz), 131.39 (d, *J* = 2.0 Hz), 131.13, 130.68, 130.57, 130.28 (d, *J* = 89.9 Hz), 129.20, 127.78 (d, *J* = 11.1 Hz), 127.29 (d, *J* = 12.1 Hz), 127.14, 125.52 (d, *J* = 4.0 Hz), 122.44 (d, *J* = 2.0 Hz), 115.84 (d, *J* = 11.1 Hz), 113.90, 113.82, 112.41, 109.89, 55.36, 55.32, 53.11 (d, *J* = 59.6 Hz), 35.29. **³¹P NMR** (162 MHz, CDCl₃) δ 28.38 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₆H₂₉NaO₄P⁺: 579.1696, Found: 579.1696.

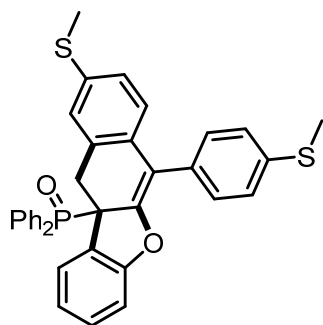
(9-(tert-butyl)-6-(4-(tert-butyl)phenyl)naphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5d)



A white solid, (88 mg, 72% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.77-7.72 (m, 4H), 7.55 (d, *J* = 10.0 Hz, 1H), 7.50 (d, *J* = 10.0 Hz, 2H), 7.45 (d, *J* = 5.0 Hz, 2H), 7.31-7.28 (m, 1H), 7.23-7.20 (m, 2H), 7.12 (s, 1H), 7.10-7.07 (m, 1H), 7.02-6.98 (m, 3H), 6.90-6.87 (m, 1H), 6.76 (d, *J* = 10.0 Hz, 1H), 6.63 (d, *J* = 5.0 Hz, 1H), 6.50 (d, *J* = 10.0 Hz, 1H), 4.23-4.18 (m, 1H), 3.58-3.48 (m, 1H), 1.40 (s, 9H), 1.24 (s, 9H). **¹³C NMR** (126 MHz, CDCl₃) δ 159.32 (d, *J* = 3.8 Hz), 152.16 (d, *J* = 6.3 Hz), 150.39, 149.33, 133.56 (d, *J* = 5.0 Hz), 131.88 (d, *J* = 3.8 Hz), 131.62 (d, *J* = 8.8 Hz), 131.41 (d, *J* = 2.5 Hz),

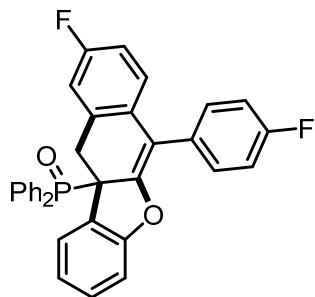
130.92, 130.66 (d, $J = 8.8$ Hz), 129.77 (d, $J = 3.8$ Hz), 129.23 (d, $J = 2.5$ Hz), 127.82 (d, $J = 11.3$ Hz), 127.45 (d, $J = 11.3$ Hz), 126.02, 125.66 (d, $J = 3.8$ Hz), 125.32, 125.24, 123.88, 122.59 (d, $J = 3.8$ Hz), 116.28 (d, $J = 11.3$ Hz), 110.03, 53.54 (d, $J = 59.2$ Hz), 35.29, 34.79, 34.45, 31.52, 31.36. ^{31}P NMR (202 MHz, CDCl_3) δ 26.11 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{42}\text{H}_{41}\text{NaO}_2\text{P}^+$: 631.2736, Found: 631.2730.

(9-(methylthio)-6-(4-(methylthio)phenyl)naphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5e)



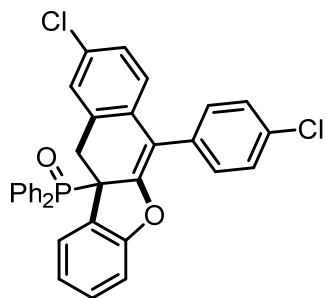
A white solid, (72 mg, 61% yield). ^1H NMR (500 MHz, CDCl_3) δ 7.75-7.70 (m, 4H), 7.49 (d, $J = 5.0$ Hz, 1H), 7.38-7.28 (m, 5H), 7.25-7.20 (m, 2H), 7.15-7.12 (m, 1H), 7.06-6.98 (m, 4H), 6.89-6.86 (m, 1H), 6.65-6.61 (m, 2H), 6.38 (d, $J = 10.0$ Hz, 1H), 4.19-4.14 (m, 1H), 3.53-3.43 (m, 1H), 2.53 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.16 (d, $J = 3.8$ Hz), 152.44 (d, $J = 5.0$ Hz), 138.02, 136.52, 133.31 (d, $J = 3.8$ Hz), 131.78 (d, $J = 3.8$ Hz), 131.66, 131.59, 131.27, 130.65 (d, $J = 8.8$ Hz), 130.45, 129.65, 129.41, 127.93 (d, $J = 12.6$ Hz), 127.70 (d, $J = 3.8$ Hz), 127.47 (d, $J = 12.6$ Hz), 126.33, 125.65, 122.80, 115.62 (d, $J = 11.3$ Hz), 110.04, 53.35 (d, $J = 58.0$ Hz), 35.01, 16.11, 15.76. ^{31}P NMR (202 MHz, CDCl_3) δ 26.79 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{36}\text{H}_{29}\text{NaO}_2\text{PS}_2^+$: 611.1239, Found: 611.1238.

(9-fluoro-6-(4-fluorophenyl)naphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5f)



A white solid, (56 mg, 53% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.80-7.74 (m, 4H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.45-7.41 (m, 2H), 7.36-7.33 (m, 1H), 7.28-7.24 (m, 2H), 7.21-7.17 (m, 3H), 7.14-7.09 (m, 2H), 7.07-7.03 (m, 1H), 6.93-6.89 (m, 1H), 6.86 (d *J* = 8.0 Hz, 1H), 6.68 (d *J* = 8.0 Hz, 1H), 6.49-6.40 (m, 2H), 4.26-4.19 (m, 1H), 3.58-3.46 (m, 1H). **¹³C NMR** (126 MHz, CDCl₃) δ 162.16 (d, *J* = 248.5 Hz), 161.33 (d, *J* = 248.5 Hz), 158.99 (d, *J* = 4.0 Hz), 152.55, 133.35 (d, *J* = 8.1 Hz), 132.24, 131.56, 131.48, 131.36 (d, *J* = 3.0 Hz), 130.60, 130.52, 129.43, 127.88 (d, *J* = 11.1 Hz), 127.49 (d, *J* = 11.1 Hz), 127.17 (d, *J* = 9.1 Hz), 125.47 (d, *J* = 3.0 Hz), 122.83 (d, *J* = 3.0 Hz), 115.52 (d, *J* = 21.2 Hz), 115.15, 114.70 (d, *J* = 11.1 Hz), 113.58 (d, *J* = 21.2 Hz), 109.97 (d, *J* = 2.0 Hz), 53.06 (d, *J* = 58.6 Hz), 34.83. **³¹P NMR** (162 MHz, CDCl₃) δ 27.89 (s). **¹⁹F NMR** (376 MHz, CDCl₃) δ -113.76 (s), -115.67 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₄H₂₃F₂NaO₂P⁺: 555.1296, Found: 555.1295.

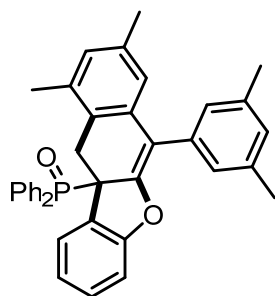
(9-chloro-6-(4-chlorophenyl)naphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5g)



A white solid, (77 mg, 68% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.74-7.69 (m, 4H), 7.46-7.42 (m, 3H), 7.35 (d, *J* = 10.0 Hz, 2H), 7.33-7.30 (m, 1H), 7.25-7.21 (m, 2H), 7.19-7.16 (m, 1H), 7.10-7.07 (m, 3H), 7.04-7.01 (m, 1H), 6.90-6.87 (m, 1H), 6.70 (dd, *J* = 5.0, 10.0 Hz, 1H), 6.66 (d, *J* = 10.0 Hz, 1H), 6.34 (d, *J* = 10.0 Hz, 1H), 4.21-4.15 (m, 1H), 3.51-3.41 (m, 1H). **¹³C NMR** (126 MHz, CDCl₃) δ 158.97 (d, *J* = 5.0 Hz), 153.67 (d, *J* = 6.3 Hz), 134.51 (d, *J* = 3.8 Hz), 133.59, 132.88, 132.07, 131.63 (d, *J* = 8.8 Hz), 131.62 (d, *J* = 27.7 Hz), 131.31 (d, *J* = 3.8 Hz), 130.64 (d, *J* = 8.8 Hz), 129.58, 128.86, 128.22, 128.01 (d, *J* = 11.3 Hz), 127.59 (d, *J* = 11.3 Hz), 127.21, 126.90, 125.56 (d, *J* = 3.8 Hz), 123.05 (d, *J* = 2.5 Hz), 114.54 (d, *J* = 10.1 Hz), 110.10, 53.31 (d, *J* = 58.0 Hz), 34.68. **³¹P NMR** (202 MHz, CDCl₃) δ 26.99 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₄H₂₃Cl₂NaO₂P⁺: 587.0705, Found: 587.0701.

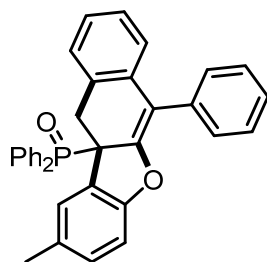
(6-(3,5-dimethylphenyl)-8,10-dimethylnaphtho[2,3-b]benzofuran-11a(11H)-

yl)diphenylphosphine oxide (5h)



A white solid, (66 mg, 60% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.77-7.74 (m, 2H), 7.67-7.63 (m, 2H), 7.50 (d, *J* = 5.0 Hz, 1H), 7.31-7.28 (m, 1H), 7.25-7.21 (m, 2H), 7.16-7.13 (m, 1H), 7.04-6.98 (m, 6H), 6.86-6.83 (m, 1H), 6.69 (d, *J* = 10.0 Hz, 1H), 6.57 (s, 1H), 6.01 (s, 1H), 4.40-4.35 (m, 1H), 3.22-3.12 (m, 1H), 2.40 (s, 6H), 2.38 (s, 3H), 1.95 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 159.26, 152.59, 137.77, 136.80, 135.85, 135.24, 135.05, 131.38, 131.03 (d, *J* = 8.8 Hz), 130.57 (d, *J* = 8.8 Hz), 130.19 (d, *J* = 2.5 Hz), 129.55, 129.32, 129.17, 127.93, 127.83, 127.32 (d, *J* = 12.6 Hz), 125.47 (d, *J* = 3.8 Hz), 125.06, 122.61, 116.57, 110.18, 53.43 (d, *J* = 59.2 Hz), 30.86, 21.62, 20.82, 20.16. **³¹P NMR** (202 MHz, CDCl₃) δ 27.73 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₈H₃₃NaO₂P⁺: 575.2110, Found: 575.2106.

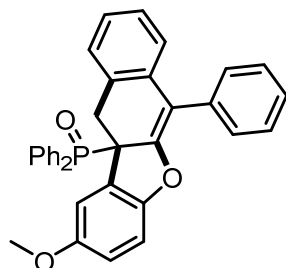
(2-methyl-6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5i)



A white solid, (74 mg, 73% yield) **¹H NMR** (400 MHz, CDCl₃) δ 7.83-7.75 (m, 4H), 7.51-7.50 (m, 4H), 7.43-7.39 (m, 1H), 7.35 (s, 1H), 7.33-7.25 (m, 3H), 7.17-7.12 (m, 2H), 7.08-7.04 (m, 2H), 6.94-6.91 (m, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.78-6.75 (m, 1H), 6.58 (d, *J* = 8.0 Hz, 1H), 6.50 (d, *J* = 4.0 Hz, 1H), 4.32-4.25 (m, 1H), 3.62-3.49 (m, 1H), 2.25 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 157.14 (d, *J* = 4.0 Hz), 153.35 (d, *J* = 5.0 Hz), 136.34 (d, *J* = 4.0 Hz), 134.97 (d, *J* = 4.0 Hz), 132.21 (d, *J* = 3.0 Hz), 131.58, 131.50, 131.19 (dd, *J* = 39.4, 2.0 Hz), 130.64 (d, *J* = 9.1 Hz), 130.10 (d, *J* = 3.0 Hz), 129.64, 128.39, 127.98, 127.82, 127.71, 127.48 (d, *J* = 12.1 Hz), 127.30, 127.06, 126.43,

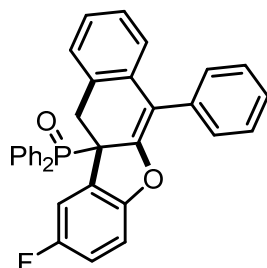
125.93, 116.09 (d, $J = 10.1$ Hz), 109.57, 53.23 (d, $J = 58.6$ Hz), 35.00, 20.89. ^{31}P NMR (162 MHz, CDCl_3) δ 26.69 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{35}\text{H}_{27}\text{NaO}_2\text{P}^+$: 533.1641, Found: 533.1639.

(2-methoxy-6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5j)



A white solid, (59 mg, 56% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.84-7.73 (m, 4H), 7.52-7.49 (m, 4H), 7.43-7.39 (m, 1H), 7.36-7.32 (m, 1H), 7.29-7.26 (m, 2H), 7.17-7.12 (m, 2H), 7.09-7.04 (m, 3H), 6.95-6.91 (m, 1H), 6.78-6.74 (m, 1H), 6.61-6.56 (m, 2H), 6.47 (d, $J = 8.0$ Hz, 1H), 4.29-4.23 (m, 1H), 3.74 (s, 3H), 3.63-3.50 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.57 (d, $J = 3.0$ Hz), 153.47 (d, $J = 6.1$ Hz), 153.16 (d, $J = 5.1$ Hz), 136.32 (d, $J = 5.1$ Hz), 134.91 (d, $J = 4.0$ Hz), 131.52, 131.44, 131.03 (d, $J = 3.0$ Hz), 130.63, 130.54, 130.06 (d, $J = 3.0$ Hz), 128.37, 127.95, 127.83 (d, $J = 11.1$ Hz), 127.53, 127.38 (d, $J = 12.1$ Hz), 127.10, 126.42, 125.91 (d, $J = 2.0$ Hz), 116.05 (d, $J = 11.1$ Hz), 115.48 (d, $J = 3.0$ Hz), 110.40 (d, $J = 3.0$ Hz), 56.11, 53.77 (d, $J = 58.6$ Hz), 34.95. ^{31}P NMR (162 MHz, CDCl_3) δ 26.91 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{35}\text{H}_{27}\text{NaO}_3\text{P}^+$: 549.1590, Found: 549.1592.

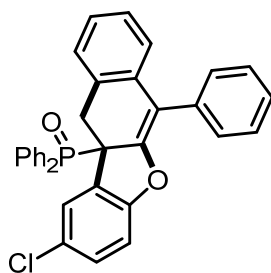
(2-fluoro-6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5k)



A white solid, (67 mg, 65% yield). ^1H NMR (500 MHz, CDCl_3) δ 7.81-7.72 (m, 4H), 7.49-7.44 (m, 4H), 7.40-7.37 (m, 1H), 7.34-7.31 (m, 1H), 7.28-7.21 (m, 3H), 7.13-7.09

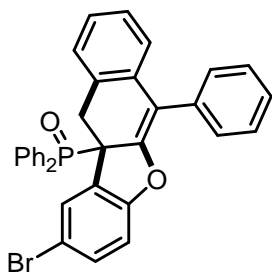
(m, 2H), 7.04-7.01 (m, 2H), 6.90-6.87 (m, 1H), 6.74-6.67 (m, 2H), 6.57-6.55 (m, 1H), 6.45 (d, $J = 10.0$ Hz, 1H), 4.23-4.18 (m, 1H), 3.57-3.48 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 158.55 (d, $J = 240.7$ Hz), 154.08 (d, $J = 254.5$ Hz), 136.08 (d, $J = 5.0$ Hz), 134.72 (d, $J = 3.8$ Hz), 131.70 (d, $J = 3.8$ Hz), 131.59 (d, $J = 8.8$ Hz), 130.77 (d, $J = 98.3$ Hz), 130.67 (d, $J = 8.8$ Hz), 130.09, 129.37, 128.51, 128.08, 127.88 (d, $J = 25.2$ Hz), 127.54 (d, $J = 12.6$ Hz), 127.25, 126.74, 126.15, 116.66 (d, $J = 10.1$ Hz), 115.72 (d, $J = 23.9$ Hz), 112.82 (d, $J = 29.0$ Hz), 110.51 (d, $J = 8.8$ Hz), 53.79 (d, $J = 56.7$ Hz), 34.77. ^{31}P NMR (202 MHz, CDCl_3) δ 26.78 (s). ^{19}F NMR (471 MHz, CDCl_3) δ -120.12 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{34}\text{H}_{24}\text{FNaO}_2\text{P}^+$: 537.1390, Found: 537.1388.

(2-chloro-6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5l)



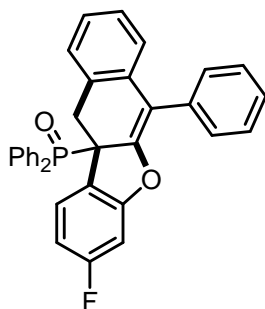
A white solid, (64 mg, 60% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.84-7.76 (m, 4H), 7.53-7.47 (m, 5H), 7.44-7.41 (m, 1H), 7.39-7.35 (m, 1H), 7.32-7.28 (m, 2H), 7.18-7.12 (m, 2H), 7.09-7.05 (m, 2H), 6.99 (d, $J = 8.0$ Hz, 1H), 6.95-6.91 (m, 1H), 6.79-6.76 (m, 1H), 6.58 (d, $J = 8.0$ Hz, 1H), 6.50 (d, $J = 8.0$ Hz, 1H), 4.27-4.20 (m, 1H), 3.62-3.49 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.64 (d, $J = 4.0$ Hz), 152.57 (d, $J = 6.1$ Hz), 135.89 (d, $J = 4.0$ Hz), 134.53 (d, $J = 4.0$ Hz), 131.68 (d, $J = 3.0$ Hz), 131.56 (d, $J = 9.1$ Hz), 131.26 (d, $J = 3.0$ Hz), 130.63 (d, $J = 9.1$ Hz), 130.02 (d, $J = 3.0$ Hz), 129.15, 128.44, 128.02, 127.91, 127.76, 127.49 (d, $J = 11.1$ Hz), 127.18, 126.75, 126.13 (d, $J = 2.0$ Hz), 125.56 (d, $J = 3.0$ Hz), 116.79 (d, $J = 10.1$ Hz), 110.90 (d, $J = 2.0$ Hz), 53.49 (d, $J = 57.6$ Hz), 34.70. ^{31}P NMR (162 MHz, CDCl_3) δ 26.16 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{34}\text{H}_{24}\text{ClNaO}_2\text{P}^+$: 553.1095, Found: 553.1096.

(2-bromo-6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5m)



A white solid, (53 mg, 46% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.84-7.77 (m, 4H), 7.65-7.64 (m, 1H), 7.53-7.47 (m, 4H), 7.44-7.41 (m, 1H), 7.39-7.35 (m, 1H), 7.33-7.28 (m, 2H), 7.18-7.12 (m, 3H), 7.10-7.05 (m, 2H), 6.94-6.91 (m, 1H), 6.79-6.76 (m, 1H), 6.54-6.50 (m, 2H), 4.27-4.20 (m, 1H), 3.61-3.49 (m, 1H). **¹³C NMR** (101 MHz, CDCl₃) δ 158.15 (d, *J* = 4.0 Hz), 152.46 (d, *J* = 6.1 Hz), 135.87 (d, *J* = 4.0 Hz), 134.51 (d, *J* = 4.0 Hz), 131.87 (dd, *J* = 34.3, 2.0 Hz), 131.57 (d, *J* = 8.1 Hz), 131.27 (d, *J* = 3.0 Hz), 130.65 (d, *J* = 8.1 Hz), 130.39 (dd, *J* = 28.3, 4.1 Hz), 130.14 (d, *J* = 192.9 Hz), 130.02 (d, *J* = 3.0 Hz), 128.45, 128.03, 127.91, 127.77, 127.50 (d, *J* = 11.1 Hz), 127.19, 126.77, 126.14 (d, *J* = 2.0 Hz), 116.79 (d, *J* = 11.1 Hz), 114.79 (d, *J* = 3.0 Hz), 111.44 (d, *J* = 2.0 Hz), 53.46 (d, *J* = 58.6 Hz), 34.71. **³¹P NMR** (162 MHz, CDCl₃) δ 25.80 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₄H₂₄BrNaO₂P⁺: 597.0590, Found: 597.0590.

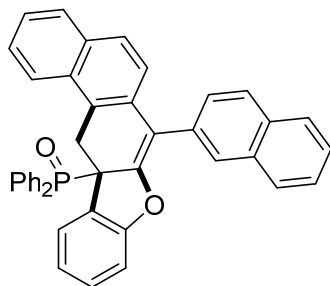
(3-fluoro-6-phenylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5n)



A white solid, (72 mg, 70% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.79-7.73 (m, 4H), 7.49-7.43 (m, 5H), 7.41-7.38 (m, 1H), 7.35-7.32 (m, 1H), 7.28-7.25 (m, 2H), 7.13-7.08 (m, 2H), 7.05-7.01 (m, 2H), 6.90-6.87 (m, 1H), 6.76-6.73 (m, 1H), 6.61-6.58 (m, 1H), 6.49 (d, *J* = 10.0 Hz, 1H), 6.38-6.36 (m, 1H), 4.25-4.19 (m, 1H), 3.56-3.46 (m, 1H). **¹³C NMR** (126 MHz, CDCl₃) δ 163.54 (d, *J* = 250.0 Hz), 159.95 (d, *J* = 12.6 Hz), 153.05 (d, *J* = 5.0 Hz), 135.93 (d, *J* = 5.0 Hz), 134.59 (d, *J* = 3.8 Hz), 131.74 (d, *J* = 3.8 Hz), 131.61 (d, *J* = 8.8 Hz), 131.26, 130.63 (d, *J* = 7.6 Hz), 130.11 (d, *J* = 2.5 Hz), 129.97 (d, *J* = 89.5 Hz), 128.54, 128.09, 128.00, 127.87, 127.57 (d, *J* = 11.3 Hz), 127.05

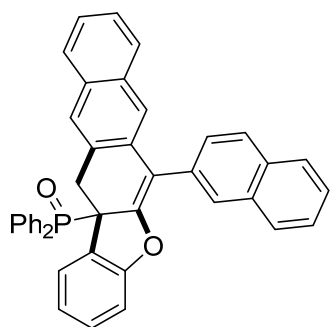
(d, $J = 45.4$ Hz), 126.23, 123.80, 117.07 (d, $J = 10.1$ Hz), 109.54 (d, $J = 22.7$ Hz), 98.79 (d, $J = 26.5$ Hz), 52.71 (d, $J = 59.2$ Hz), 35.00. ^{31}P NMR (202 MHz, CDCl_3) δ 26.56 (s). ^{19}F NMR (471 MHz, CDCl_3) δ -110.55 (s). **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{34}\text{H}_{25}\text{FO}_2\text{P}^+$: 515.1571, Found: 515.1571.

(7-(naphthalen-2-yl)phenanthro[2,3-b]benzofuran-12b(13H)-yl)diphenylphosphine oxide (5o-1)



A white solid, (39 mg, 33% yield). ^1H NMR (500 MHz, CDCl_3) δ 8.29 (d, $J = 5.0$ Hz, 1H), 8.00 (s, 1H), 7.97-7.91 (m, 3H), 7.89-7.85 (m, 2H), 7.67-7.64 (m, 2H), 7.62-7.54 (m, 6H), 7.45-7.42 (m, 1H), 7.37-7.34 (m, 1H), 7.31-7.28 (m, 2H), 7.23 (d, $J = 10.0$ Hz, 1H), 7.07-7.04 (m, 1H), 6.97-6.92 (m, 2H), 6.79-6.75 (m, 2H), 6.70-6.67 (m, 2H), 5.09-5.03 (m, 1H), 3.75-3.65 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.45 (d, $J = 5.0$ Hz), 153.55 (d, $J = 6.3$ Hz), 134.07 (d, $J = 3.8$ Hz), 133.49, 132.87 (d, $J = 13.9$ Hz), 132.64, 131.68, 131.06, 130.99, 130.84 (d, $J = 104.6$ Hz), 130.81 (d, $J = 7.6$ Hz), 129.46, 129.25, 128.26, 128.20, 128.03, 127.93, 127.24, 126.90 (d, $J = 60.5$ Hz), 126.35 (d, $J = 7.6$ Hz), 125.82 (d, $J = 3.8$ Hz), 125.33, 124.56, 124.19, 122.89, 116.49 (d, $J = 10.1$ Hz), 110.12, 53.68 (d, $J = 58.0$ Hz), 30.52. ^{31}P NMR (202 MHz, CDCl_3) δ 26.47 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{42}\text{H}_{29}\text{NaO}_2\text{P}^+$: 619.1797, Found: 619.1789.

(6-(naphthalen-2-yl)anthra[2,3-b]benzofuran-13a(13H)-yl)diphenylphosphine oxide (5o-2)

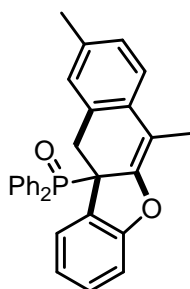


A white solid, (25 mg, 21% yield). ^1H NMR (500 MHz, CDCl_3) δ 8.04 (s, 1H), 7.99-

7.93 (m, 3H), 7.85-7.81 (m, 2H), 7.72-7.68 (m, 2H), 7.66 (d, $J = 10.0$ Hz, 1H), 7.62 (d, $J = 10.0$ Hz, 1H), 7.59 (s, 1H), 7.57-7.55 (m, 3H), 7.37-7.27 (m, 6H), 7.05-7.02 (m, 1H), 6.93-6.90 (m, 1H), 6.89 (s, 1H), 6.78-6.75 (m, 1H), 6.70-6.67 (m, 3H), 4.49-4.44 (m, 1H), 3.74-3.65 (m, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.02 (d, $J = 3.8$ Hz), 154.24, 134.44 (d, $J = 3.8$ Hz), 133.53, 132.98 (d, $J = 3.8$ Hz), 132.63, 132.45, 131.81 (d, $J = 7.6$ Hz), 131.65, 130.78, 130.72, 129.93 (d, $J = 6.3$ Hz), 129.45, 128.27, 128.06 (d, $J = 7.6$ Hz), 127.94, 127.36, 127.15, 127.06, 126.96, 126.38, 125.74, 125.57 (d, $J = 3.8$ Hz), 125.16, 122.91, 116.86 (d, $J = 10.1$ Hz), 110.12, 53.59 (d, $J = 59.2$ Hz), 35.89. ^{31}P NMR (202 MHz, CDCl_3) δ 27.89 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{42}\text{H}_{29}\text{NaO}_2\text{P}^+$: 619.1797, Found: 619.1794.

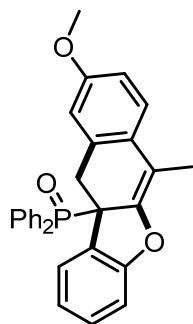
(6,9-dimethylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide

(5p)



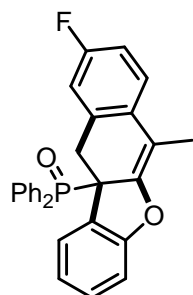
A white solid, (28 mg, 31% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.71-7.65 (m, 4H), 7.51 (d, $J = 8.0$ Hz, 1H), 7.32-7.26 (m, 1H), 7.22-7.15 (m, 3H), 7.08-7.03 (m, 3H), 6.92-6.89 (m, 1H), 6.84 (s, 1H), 6.73 (d, $J = 8.0$ Hz, 1H), 6.67 (d, $J = 8.0$ Hz, 1H), 6.59 (d, $J = 8.0$ Hz, 1H), 4.17-4.10 (m, 1H), 3.45-3.33 (m, 1H), 2.20 (s, 3H), 2.13 (d, $J = 4.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.12 (d, $J = 4.0$ Hz), 151.53 (d, $J = 5.1$ Hz), 135.74, 133.71 (d, $J = 4.0$ Hz), 131.56 (d, $J = 9.1$ Hz), 131.12 (dd, $J = 36.4, 3.0$ Hz), 130.72 (d, $J = 8.1$ Hz), 129.22 (dd, $J = 190.9, 4.0$ Hz), 129.17 (d, $J = 3.0$ Hz), 128.67 (d, $J = 1.4$ Hz), 127.72, 127.60, 127.04 (d, $J = 11.1$ Hz), 125.62 (d, $J = 4.0$ Hz), 122.89 (d, $J = 2.0$ Hz), 122.36 (d, $J = 3.0$ Hz), 109.57 (d, $J = 2.0$ Hz), 109.23 (d, $J = 11.1$ Hz), 52.39 (d, $J = 59.6$ Hz), 34.84, 20.93, 11.40 (d, $J = 4.0$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 26.07 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{30}\text{H}_{25}\text{NaO}_2\text{P}^+$: 471.1484, Found: 471.1484.

(9-methoxy-6-methylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5q)



A white solid, (19 mg, 20% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.69-7.64 (m, 4H), 7.47 (d, *J* = 6.0 Hz, 1H), 7.26-7.23 (m, 1H), 7.17-7.12 (m, 3H), 7.05-7.00 (m, 3H), 6.87-6.84 (m, 1H), 6.69 (d, *J* = 6.0 Hz, 1H), 6.59-6.57 (m, 2H), 6.37-6.35 (m, 1H), 4.12-4.07 (m, 1H), 3.68 (s, 3H), 3.40-3.31 (m, 1H), 2.08 (d, *J* = 6.0 Hz, 3H). **¹³C NMR** (151 MHz, CDCl₃) δ 159.17 (d, *J* = 4.5 Hz), 158.05, 150.58 (d, *J* = 4.0 Hz), 131.95 (d, *J* = 3.0 Hz), 131.54 (d, *J* = 7.6 Hz), 131.19 (dd, *J* = 31.7, 3.1 Hz), 130.71 (d, *J* = 9.1 Hz), 129.20, 127.67 (d, *J* = 10.6 Hz), 127.13 (d, *J* = 12.1 Hz), 125.55 (d, *J* = 3.0 Hz), 123.99, 122.32 (d, *J* = 3.0 Hz), 113.88, 112.24, 109.57, 109.06 (d, *J* = 10.6 Hz), 55.34, 52.30 (d, *J* = 60.4 Hz), 35.15, 11.45 (d, *J* = 3.0 Hz). **³¹P NMR** (243 MHz, CDCl₃) δ 25.78 (s). **HRMS (ESI):** ([M+Na]⁺) Calcd for C₃₀H₂₅NaO₃P⁺: 487.1434, Found: 487.1433.

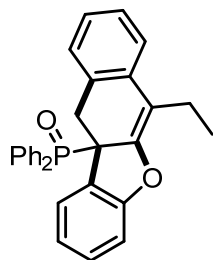
(9-fluoro-6-methylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5r)



A white solid, (23 mg, 25% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.72-7.66 (m, 4H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.32-7.29 (m, 1H), 7.23-7.19 (m, 3H), 7.13-7.05 (m, 3H), 6.92-6.88 (m, 1H), 6.78-6.73 (m, 2H), 6.65-6.61 (m, 1H), 6.57-6.52 (m, 1H), 4.21-4.14 (m, 1H), 3.44-3.32 (m, 1H), 2.12 (d, *J* = 4.0 Hz, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 161.10 (d, *J* = 246.4 Hz), 158.98 (d, *J* = 4.0 Hz), 151.92, 132.70, 131.54 (d, *J* = 8.1 Hz), 131.34 (dd, *J* = 12.1, 3.0 Hz), 130.67 (d, *J* = 8.1 Hz), 130.26 (d, *J* = 19.2 Hz), 129.30 (d, *J* = 3.0 Hz), 127.94 (d, *J* = 4.0 Hz), 127.72 (d, *J* = 11.1 Hz), 127.27 (d, *J* = 12.1 Hz), 125.48

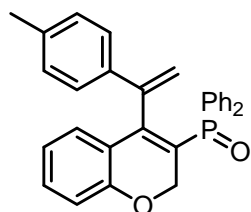
(d, $J = 3.0$ Hz), 124.18 (d, $J = 8.1$ Hz), 122.52 (d, $J = 2.0$ Hz), 114.16 (dd, $J = 161.6$, 22.2 Hz), 109.66 (d, $J = 2.0$ Hz), 108.56 (d, $J = 11.1$ Hz), 52.16 (d, $J = 58.6$ Hz), 34.70, 11.50 (d, $J = 4.0$ Hz). **^{31}P NMR** (162 MHz, CDCl_3) δ 26.06 (s). **^{19}F NMR** (376 MHz, CDCl_3) δ -116.69 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{29}\text{H}_{22}\text{FNaO}_2\text{P}^+$: 475.1234, Found: 475.1231.

(6-ethylnaphtho[2,3-b]benzofuran-11a(11H)-yl)diphenylphosphine oxide (5s)



A white solid, (30 mg, 33% yield). **^1H NMR** (500 MHz, CDCl_3) δ 7.69-7.64 (m, 4H), 7.49 (d, $J = 10.0$ Hz, 1H), 7.27-7.24 (m, 1H), 7.19-7.15 (m, 2H), 7.15-7.11 (m, 1H), 7.04-6.99 (m, 4H), 6.89-6.86 (m, 1H), 6.84-6.81 (m, 2H), 6.74-6.73 (m, 1H), 6.70 (d, $J = 5.0$ Hz, 1H), 4.19-4.14 (m, 1H), 3.43-3.34 (m, 1H), 2.69-2.59 (m, 2H), 1.22 (t, $J = 7.5$ Hz, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 159.13 (d, $J = 3.8$ Hz), 152.01 (d, $J = 5.0$ Hz), 135.56 (d, $J = 5.0$ Hz), 131.77 (d, $J = 8.8$ Hz), 131.26 (dd, $J = 34.0$, 3.8 Hz), 130.77, 130.70, 129.28 (d, $J = 2.5$ Hz), 128.08, 127.74 (d, $J = 12.6$ Hz), 127.29 (d, $J = 11.3$ Hz), 127.18, 126.16, 125.67 (d, $J = 3.8$ Hz), 122.92, 122.51 (d, $J = 3.8$ Hz), 115.51 (d, $J = 11.3$ Hz), 109.69, 52.24 (d, $J = 59.2$ Hz), 34.94, 19.21 (d, $J = 2.5$ Hz), 13.64 (d, $J = 6.3$ Hz). **^{31}P NMR** (202 MHz, CDCl_3) δ 26.55 (s). **HRMS (ESI):** ($[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{30}\text{H}_{25}\text{NaO}_2\text{P}^+$: 471.1484, Found: 471.1481

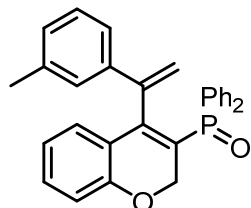
diphenyl(4-(1-(p-tolyl)vinyl)-2H-chromen-3-yl)phosphine oxide (6a)



A white solid, (82 mg, 91% yield). **^1H NMR** (500 MHz, CDCl_3) δ 7.69-7.58 (m, 4H), 7.40-7.37 (m, 2H), 7.31-7.26 (m, 4H), 7.20-7.17 (m, 1H), 7.06 (d, $J = 10.0$ Hz, 1H), 7.00 (d, $J = 5.0$ Hz, 2H), 6.92-6.89 (m, 3H), 6.78-6.75 (m, 1H), 5.55 (s, 1H), 5.11 (s, 1H), 4.90-4.86 (m, 1H), 4.80-4.77 (m, 1H), 2.26 (s, 3H). **^{13}C NMR** (126 MHz, CDCl_3)

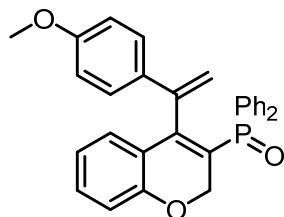
δ 155.26, 148.47 (d, $J = 6.3$ Hz), 141.33 (d, $J = 5.0$ Hz), 137.80, 134.69, 132.04, 131.91, 131.75, 131.59, 128.88, 128.32 (d, $J = 8.8$ Hz), 127.72, 125.93, 123.80 (d, $J = 12.6$ Hz), 121.98, 121.84 (d, $J = 102.1$ Hz), 116.65, 116.41, 66.31 (d, $J = 17.6$ Hz), 21.23. **^{31}P NMR** (202 MHz, CDCl_3) δ 26.65. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{30}\text{H}_{26}\text{O}_2\text{P}^+$: 449.1665, Found: 449.1665.

diphenyl(4-(1-(m-tolyl)vinyl)-2H-chromen-3-yl)phosphine oxide (6b)



A white solid, (74 mg, 83% yield). **^1H NMR** (500 MHz, CDCl_3) δ 7.67–7.58 (m, 4H), 7.41–7.38 (m, 2H), 7.30–7.26 (m, 4H), 7.21–7.18 (m, 1H), 7.08 (d, $J = 5.0$ Hz, 1H), 7.01–6.98 (m, 1H), 6.95 (d, $J = 10.0$ Hz, 1H), 6.92–6.85 (m, 3H), 6.80–6.77 (m, 3H), 5.61 (s, 1H), 5.17 (s, 1H), 4.90–4.86 (m, 1H), 4.82–4.79 (m, 1H), 2.20 (s, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 155.24, 148.36, 141.58, 137.43 (d, $J = 7.6$ Hz), 132.76, 132.12, 131.93, 131.76, 131.61, 128.75, 128.31, 128.21, 128.06, 127.71, 126.37, 123.90 (d, $J = 11.3$ Hz), 123.39, 121.99, 117.49, 116.40, 66.29 (d, $J = 17.6$ Hz), 21.55. **^{31}P NMR** (202 MHz, CDCl_3) δ 26.78. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{30}\text{H}_{26}\text{O}_2\text{P}^+$: 449.1665, Found: 449.1667.

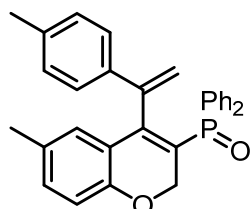
(4-(1-(4-methoxyphenyl)vinyl)-2H-chromen-3-yl)diphenylphosphine oxide (6c)



A white solid, (82 mg, 88% yield). **^1H NMR** (500 MHz, CDCl_3) δ 7.68–7.59 (m, 4H), 7.41–7.38 (m, 2H), 7.31–7.26 (m, 4H), 7.20–7.17 (m, 1H), 7.06 (d, $J = 10.0$ Hz, 1H), 7.02 (d, $J = 10.0$ Hz, 2H), 6.90 (d, $J = 10.0$ Hz, 1H), 6.79–6.76 (m, 1H), 6.64 (d, $J = 10.0$ Hz, 2H), 5.45 (s, 1H), 5.07 (s, 1H), 4.91–4.87 (m, 1H), 4.80–4.76 (m, 1H), 3.74 (s, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 159.48, 155.28, 148.52, 140.89 (d, $J = 6.3$ Hz), 132.07, 131.89, 131.81, 131.61, 130.26, 128.29 (d, $J = 11.3$ Hz), 127.69, 127.31, 123.72

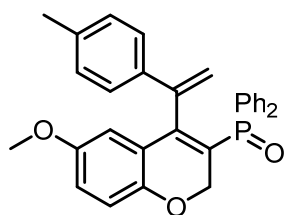
(d, $J = 11.3$ Hz), 121.98, 121.74 (d, $J = 100.8$ Hz), 116.42, 115.68, 113.52, 66.28 (d, $J = 17.6$ Hz), 55.33. **^{31}P NMR** (202 MHz, CDCl_3) δ 26.83. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{30}\text{H}_{26}\text{O}_3\text{P}^+$: 465.1614, Found: 465.1617.

(6-methyl-4-(1-(p-tolyl)vinyl)-2H-chromen-3-yl)diphenylphosphine oxide (6d)



A white solid, (80 mg, 87% yield). **^1H NMR** (500 MHz, CDCl_3) δ 7.67–7.56 (m, 4H), 7.40–7.38 (m, 2H), 7.31–7.25 (m, 4H), 7.01–6.98 (m, 3H), 6.92 (d, $J = 5.0$ Hz, 2H), 6.88 (s, 1H), 6.81 (d, $J = 10.0$ Hz, 1H), 5.56 (s, 1H), 5.09 (s, 1H), 4.86–4.82 (m, 1H), 4.75–4.72 (m, 1H), 2.27 (s, 3H), 2.09 (s, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 153.15, 148.63 (d, $J = 6.3$ Hz), 141.35 (d, $J = 5.0$ Hz), 137.71, 134.85, 132.29, 131.92, 131.84, 131.71, 131.23, 128.86, 128.28 (d, $J = 8.8$ Hz), 127.74, 125.92, 123.65 (d, $J = 12.6$ Hz), 121.63 (d, $J = 102.1$ Hz), 116.47, 116.12, 66.33 (d, $J = 17.6$ Hz), 21.23, 20.80. **^{31}P NMR** (202 MHz, CDCl_3) δ 26.74. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{31}\text{H}_{28}\text{O}_2\text{P}^+$: 463.1821, Found: 463.1823.

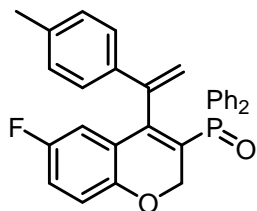
(6-methoxy-4-(1-(p-tolyl)vinyl)-2H-chromen-3-yl)diphenylphosphine oxide (6e)



A white solid, (78 mg, 82% yield). **^1H NMR** (500 MHz, CDCl_3) δ 7.69–7.57 (m, 4H), 7.41–7.38 (m, 2H), 7.31–7.25 (m, 4H), 6.99 (d, $J = 10.0$ Hz, 2H), 6.91 (d, $J = 10.0$ Hz, 2H), 6.84 (d, $J = 10.0$ Hz, 1H), 6.76–6.74 (m, 1H), 6.61 (d, $J = 3.0$ Hz, 1H), 5.54 (s, 1H), 5.11 (s, 1H), 4.83–4.80 (m, 1H), 4.73–4.69 (m, 1H), 3.53 (s, 3H), 2.25 (s, 3H). **^{13}C NMR** (126 MHz, CDCl_3) δ 154.29, 149.30, 148.53 (d, $J = 6.3$ Hz), 141.37 (d, $J = 6.3$ Hz), 137.75, 134.75, 131.89, 128.88, 128.33, 125.99, 124.35 (d, $J = 11.3$ Hz), 122.63 (d, $J = 100.8$ Hz), 117.38, 116.97, 116.81, 112.19, 66.49 (d, $J = 17.6$ Hz), 55.66, 21.22. **^{31}P NMR** (202 MHz, CDCl_3) δ 26.66. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{31}\text{H}_{28}\text{O}_3\text{P}^+$:

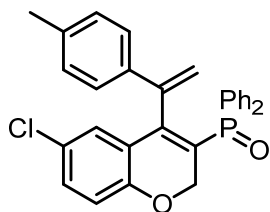
479.1771, Found: 479.1773.

(6-fluoro-4-(1-(p-tolyl)vinyl)-2H-chromen-3-yl)diphenylphosphine oxide (6f)



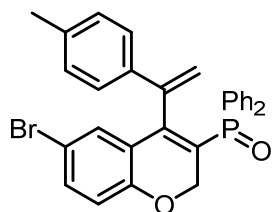
A white solid, (75 mg, 81% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.67–7.57 (m, 4H), 7.41–7.38 (m, 2H), 7.31–7.25 (m, 4H), 6.98 (d, *J* = 10.0 Hz, 2H), 6.93 (d, *J* = 5.0 Hz, 2H), 6.87–6.83 (m, 2H), 6.77–6.75 (m, 1H), 5.55 (s, 1H), 5.09 (s, 1H), 4.89–4.85 (m, 1H), 4.77–4.73 (m, 1H), 2.26 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 157.55 (d, *J* = 239.4 Hz), 151.24, 147.50 (d, *J* = 7.6 Hz), 140.92 (d, *J* = 6.3 Hz), 138.04, 134.29, 131.90, 131.82, 128.98, 128.42, 128.32, 125.88, 124.84 (dd, *J* = 12.0, 8.2 Hz), 123.55 (d, *J* = 99.5 Hz), 118.08 (d, *J* = 23.9 Hz), 117.44 (d, *J* = 7.6 Hz), 116.87, 113.65 (d, *J* = 25.2 Hz), 66.52 (d, *J* = 17.6 Hz), 21.22. **³¹P NMR** (202 MHz, CDCl₃) δ 26.58. **¹⁹F NMR** (471 MHz, CDCl₃) δ -120.71 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₀H₂₅FO₂P⁺: 467.1571, Found: 467.1577.

(6-chloro-4-(1-(p-tolyl)vinyl)-2H-chromen-3-yl)diphenylphosphine oxide (6g)



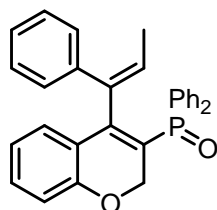
A white solid, (79 mg, 82% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.65–7.56 (m, 4H), 7.41–7.38 (m, 2H), 7.30–7.27 (m, 4H), 7.13–7.11 (m, 1H), 7.03 (d, *J* = 5.0 Hz, 1H), 6.97 (d, *J* = 5.0 Hz, 2H), 6.92 (d, *J* = 10.0 Hz, 2H), 6.84 (d, *J* = 5.0 Hz, 1H), 5.55 (s, 1H), 5.08 (s, 1H), 4.91–4.87 (m, 1H), 4.78–4.74 (m, 1H), 2.27 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 153.78, 147.15 (d, *J* = 6.3 Hz), 140.72 (d, *J* = 5.0 Hz), 138.05, 134.29, 131.94, 131.82, 131.26, 129.00, 128.42, 128.32, 127.05, 126.94, 125.84, 125.17 (d, *J* = 12.6 Hz), 123.35 (d, *J* = 99.5 Hz), 117.84, 116.92, 66.45 (d, *J* = 17.6 Hz), 21.24. **³¹P NMR** (202 MHz, CDCl₃) δ 26.57. **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₀H₂₅ClO₂P⁺: 483.1275, Found: 483.1281.

(6-bromo-4-(1-(p-tolyl)vinyl)-2H-chromen-3-yl)diphenylphosphine oxide (6h)



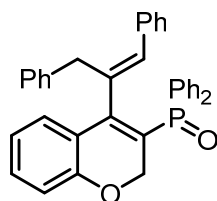
A white solid, (78 mg, 73% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.64–7.55 (m, 4H), 7.42–7.39 (m, 2H), 7.31–7.27 (m, 5H), 7.16 (s, 1H), 6.97–6.92 (m, 4H), 6.79 (d, *J* = 10.0 Hz, 1H), 5.55 (s, 1H), 5.08 (s, 1H), 4.91–4.87 (m, 1H), 4.78–4.74 (m, 1H), 2.28 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 154.29, 147.01, 140.65, 138.05, 134.29, 134.17, 131.95, 131.82, 129.94, 128.99, 128.41, 128.31, 125.83, 123.29 (d, *J* = 100.8 Hz), 118.28, 116.95, 66.39 (d, *J* = 17.6 Hz), 21.23. **³¹P NMR** (202 MHz, CDCl₃) δ 26.59. **HRMS (ESI):** ([*M*+*H*]⁺) Calcd for C₃₀H₂₅BrO₂P⁺: 527.0770, Found: 527.0776.

(E)-diphenyl(4-(1-phenylprop-1-en-1-yl)-2H-chromen-3-yl)phosphine oxide (6i)



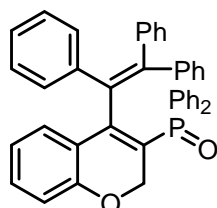
A white solid, (76 mg, 85% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.67–7.63 (m, 2H), 7.50–7.44 (m, 3H), 7.41–7.38 (m, 1H), 7.36–7.32 (m, 2H), 7.25–7.21 (m, 3H), 7.12–7.10 (m, 4H), 7.03–7.01 (m, 2H), 6.94 (d, *J* = 10.0 Hz, 1H), 6.85–6.82 (m, 1H), 6.01 (q, *J* = 5.0 Hz, 1H), 4.87–4.79 (m, 2H), 1.64 (d, *J* = 10.0 Hz, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 155.45, 146.56 (d, *J* = 6.5 Hz), 139.13, 134.75 (d, *J* = 5.0 Hz), 132.52, 131.93 (d, *J* = 15.1 Hz), 131.73, 131.66, 128.47 (d, *J* = 12.6 Hz), 128.16, 128.07, 127.09 (d, *J* = 30.2 Hz), 126.07, 125.55, 123.44 (d, *J* = 12.6 Hz), 122.25, 121.97 (d, *J* = 102.1 Hz), 116.53, 66.28 (d, *J* = 17.6 Hz), 16.25. **³¹P NMR** (202 MHz, CDCl₃) δ 25.76. **HRMS (ESI):** ([*M*+*H*]⁺) Calcd for C₃₀H₂₆O₂P⁺: 449.1665, Found: 449.1669.

(E)-(4-(1,3-diphenylprop-1-en-2-yl)-2H-chromen-3-yl)diphenylphosphine oxide (6j)



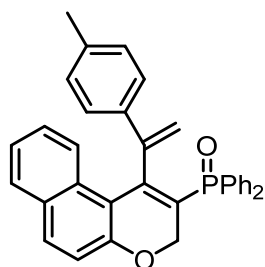
A white solid, (87 mg, 83% yield). **¹H NMR** (500 MHz, CDCl₃) δ 7.66–7.62 (m, 2H), 7.52–7.49 (m, 1H), 7.42–7.39 (m, 5H), 7.28–7.24 (m, 8H), 7.16 (br, 1H), 7.07 (br, 5H), 6.93 (d, *J* = 5.0 Hz, 1H), 6.87–6.84 (m, 1H), 6.03 (s, 1H), 4.69–4.64 (m, 1H), 4.48–4.44 (m, 1H), 3.81 (d, *J* = 15.0 Hz, 1H), 3.52 (d, *J* = 15.0 Hz, 1H). **¹³C NMR** (126 MHz, CDCl₃) δ 155.56, 150.51 (d, *J* = 5.0 Hz), 137.49 (d, *J* = 171.4 Hz), 136.94 (d, *J* = 5.0 Hz), 132.09 (d, *J* = 3.8 Hz), 131.84, 131.63, 131.63 (d, *J* = 20.2 Hz), 130.31, 130.06, 128.81 (d, *J* = 12.6 Hz), 128.66, 128.45, 128.34, 128.18, 127.20, 126.78, 126.46, 122.51 (d, *J* = 12.6 Hz), 122.20, 118.06 (d, *J* = 99.5 Hz), 116.54, 66.25 (d, *J* = 18.9 Hz), 45.86. **³¹P NMR** (202 MHz, CDCl₃) δ 25.19. **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₆H₃₀O₂P⁺: 525.1978, Found: 525.1974.

diphenyl(4-(1,2,2-triphenylvinyl)-2H-chromen-3-yl)phosphine oxide (6k)



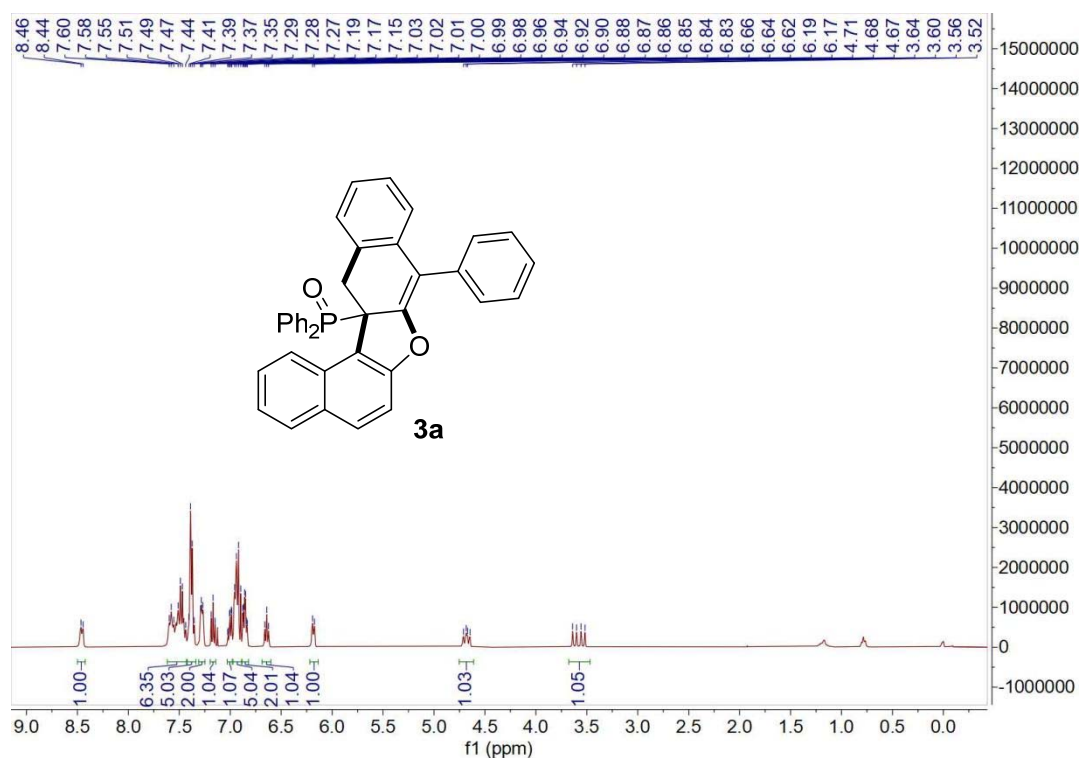
A white solid, (105 mg, 90% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.70–7.65 (m, 3H), 7.56–7.52 (m, 1H), 7.48–7.45 (m, 3H), 7.39–7.37 (m, 2H), 7.30–7.27 (m, 4H), 7.24–7.11 (m, 9H), 7.04–7.01 (m, 5H), 6.98–6.94 (m, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 4.41–4.30 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 155.33 (d, *J* = 2.0 Hz), 151.72 (d, *J* = 4.0 Hz), 143.70, 143.32 (d, *J* = 4.0 Hz), 140.67, 133.55, 132.51 (d, *J* = 3.0 Hz), 131.75, 131.63, 131.56, 131.45, 131.25, 130.82, 130.14, 128.68, 128.56, 128.08, 127.68, 127.52 (d, *J* = 9.1 Hz), 126.84, 126.54 (d, *J* = 21.2 Hz), 125.04 (d, *J* = 12.1 Hz), 122.03, 118.90 (d, *J* = 100.0 Hz), 116.31, 66.44 (d, *J* = 19.2 Hz). **³¹P NMR** (162 MHz, CDCl₃) δ 24.19. **HRMS (ESI):** ([M+H]⁺) Calcd for C₄₁H₃₂O₂P⁺: 587.2134, Found: 587.2130.

diphenyl(1-(1-(p-tolyl)vinyl)-3H-benzo[f]chromen-2-yl)phosphine oxide (6l)

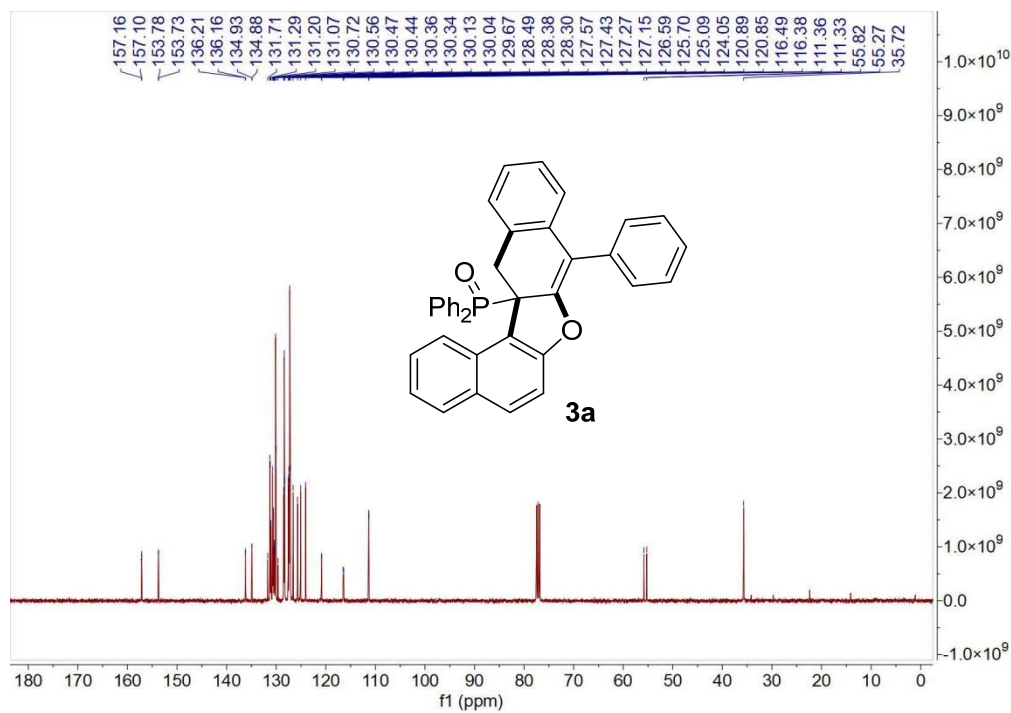


A white solid, (79 mg, 79% yield). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.42–8.39 (m, 1H), 7.83 (br, 2H), 7.70 (d, $J = 10.0$ Hz, 1H), 7.67–7.62 (m, 2H), 7.58–7.56 (m, 1H), 7.44–7.40 (m, 3H), 7.25 (br, 1H), 7.18–7.14 (m, 5H), 6.79 (d, $J = 5.0$ Hz, 2H), 6.68 (d, $J = 5.0$ Hz, 2H), 5.76 (s, 1H), 5.56 (s, 1H), 4.84 (br, 1H), 4.50 (br, 1H), 2.10 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 156.29, 150.11 (d, $J = 6.3$ Hz), 144.88 (d, $J = 6.3$ Hz), 137.42, 134.88, 133.55, 131.63, 131.53, 131.38, 129.87, 128.51, 128.43, 126.68, 126.19, 125.52, 124.00, 120.75 (d, $J = 103.3$ Hz), 120.05, 117.73 (d, $J = 11.3$ Hz), 117.15, 66.85 (d, $J = 17.6$ Hz), 21.12. $^{31}\text{P NMR}$ (202 MHz, CDCl_3) δ 26.39. **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{34}\text{H}_{28}\text{O}_2\text{P}^+$: 499.1821, Found: 499.1819.

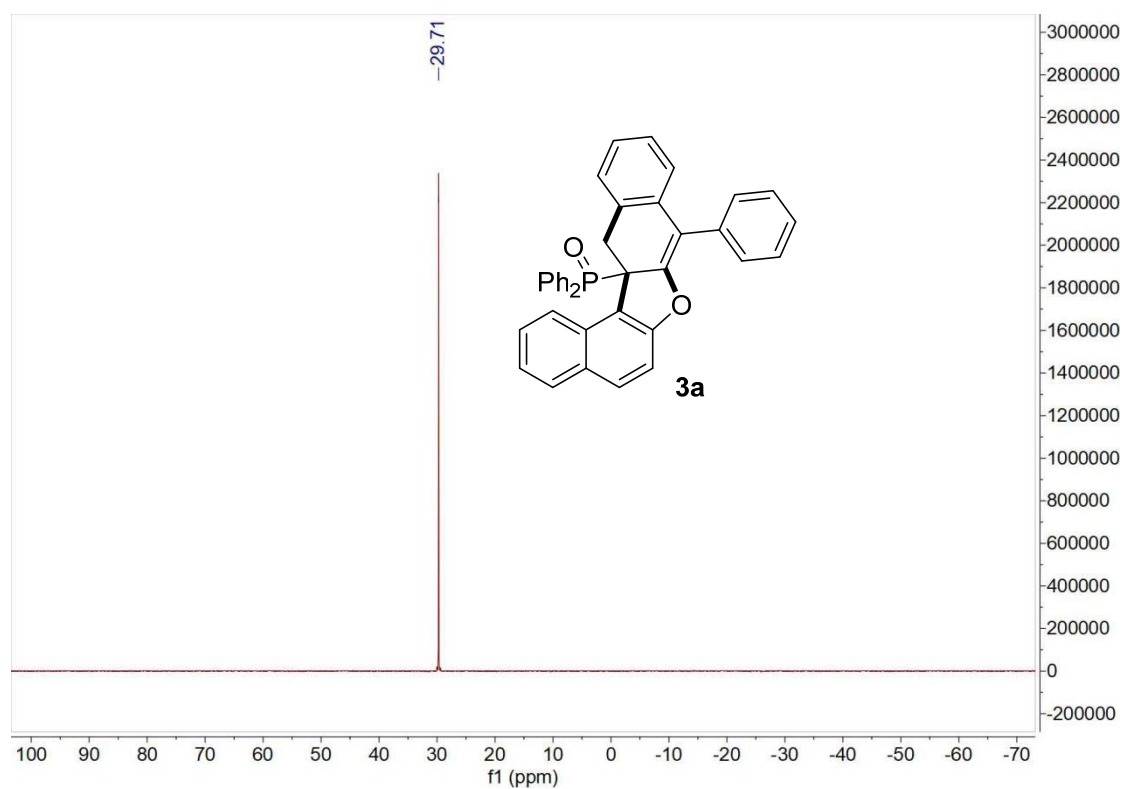
12. $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, $^{31}\text{P-NMR}$, $^{19}\text{F-NMR}$, and HRMS Spectra for Products



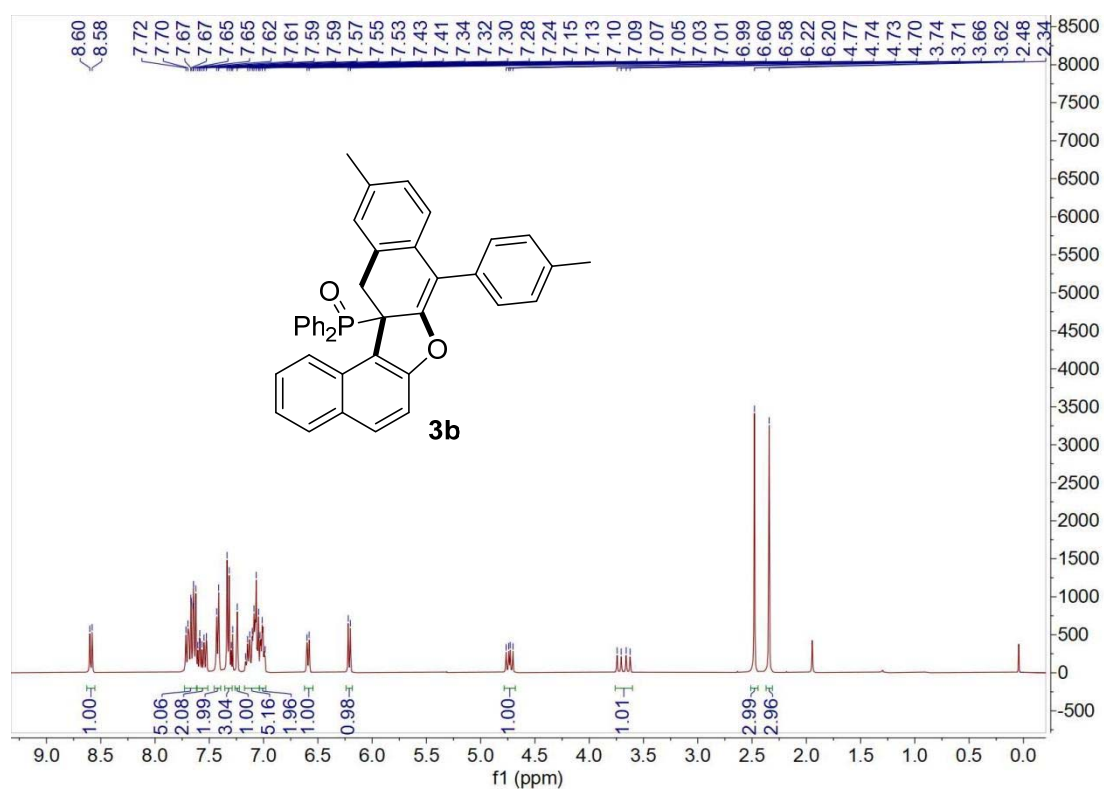
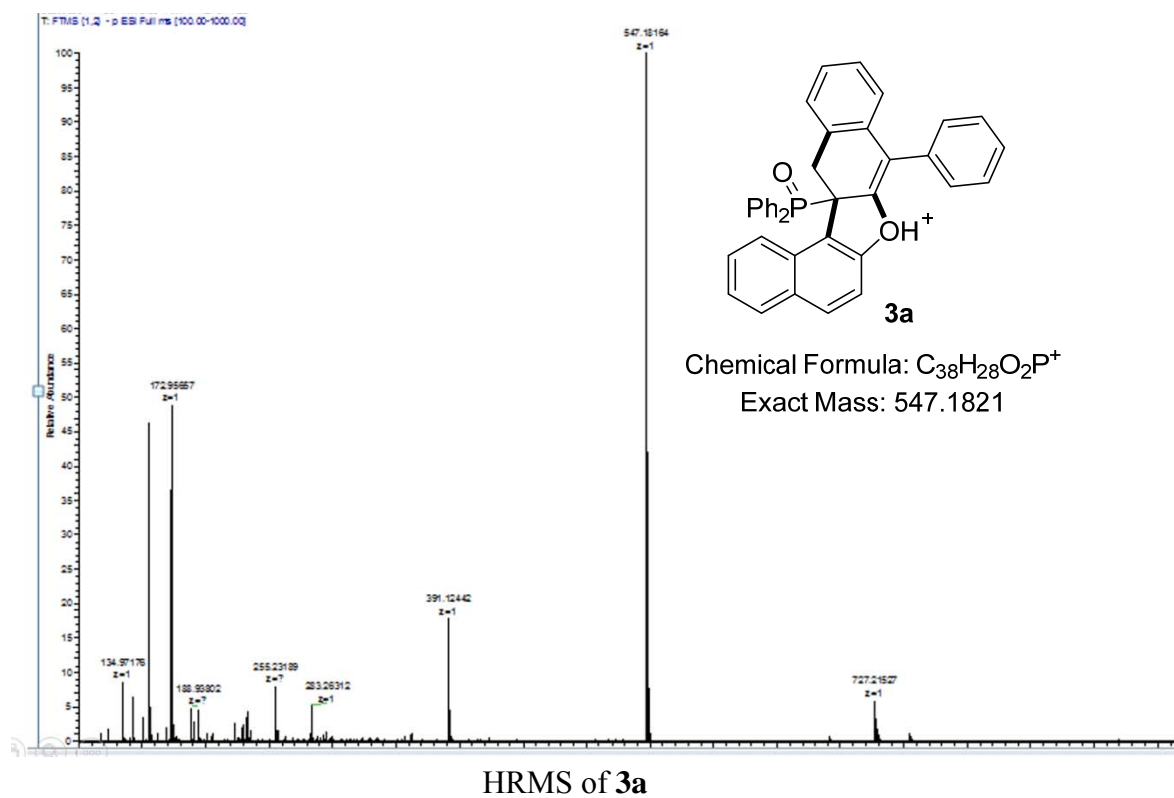
¹H-NMR (400 MHz, CDCl₃) of **3a**

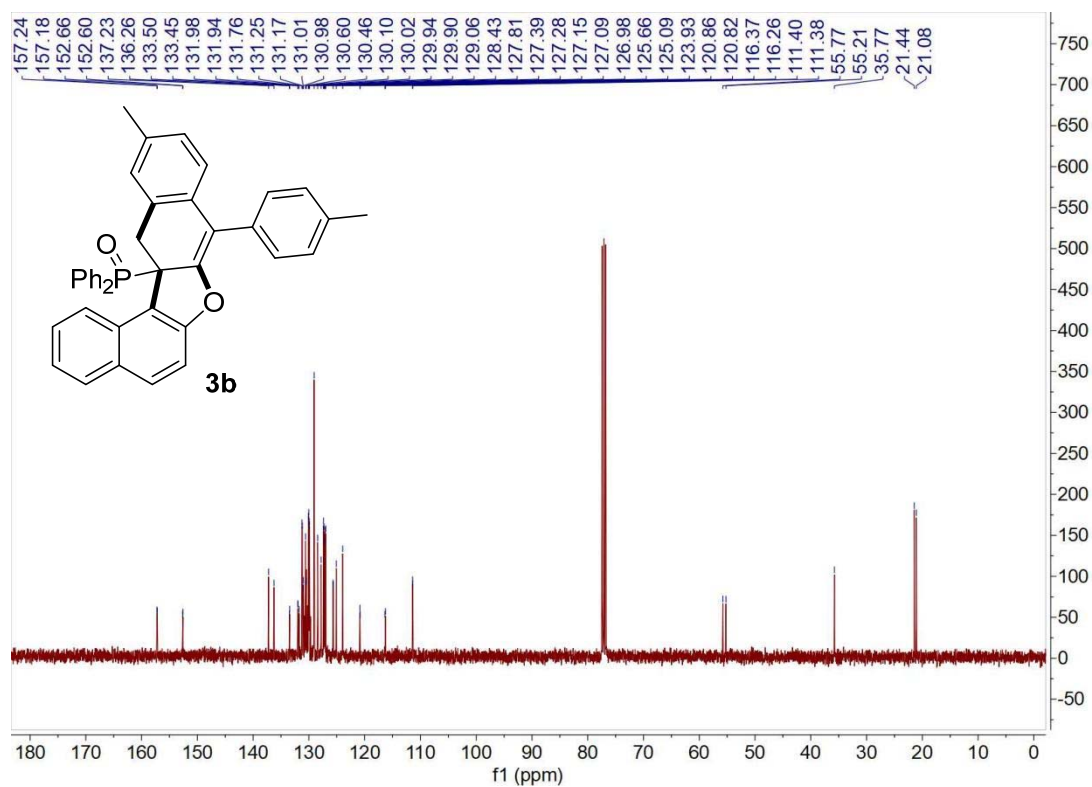


¹³C-NMR (101 MHz, CDCl₃) of **3a**

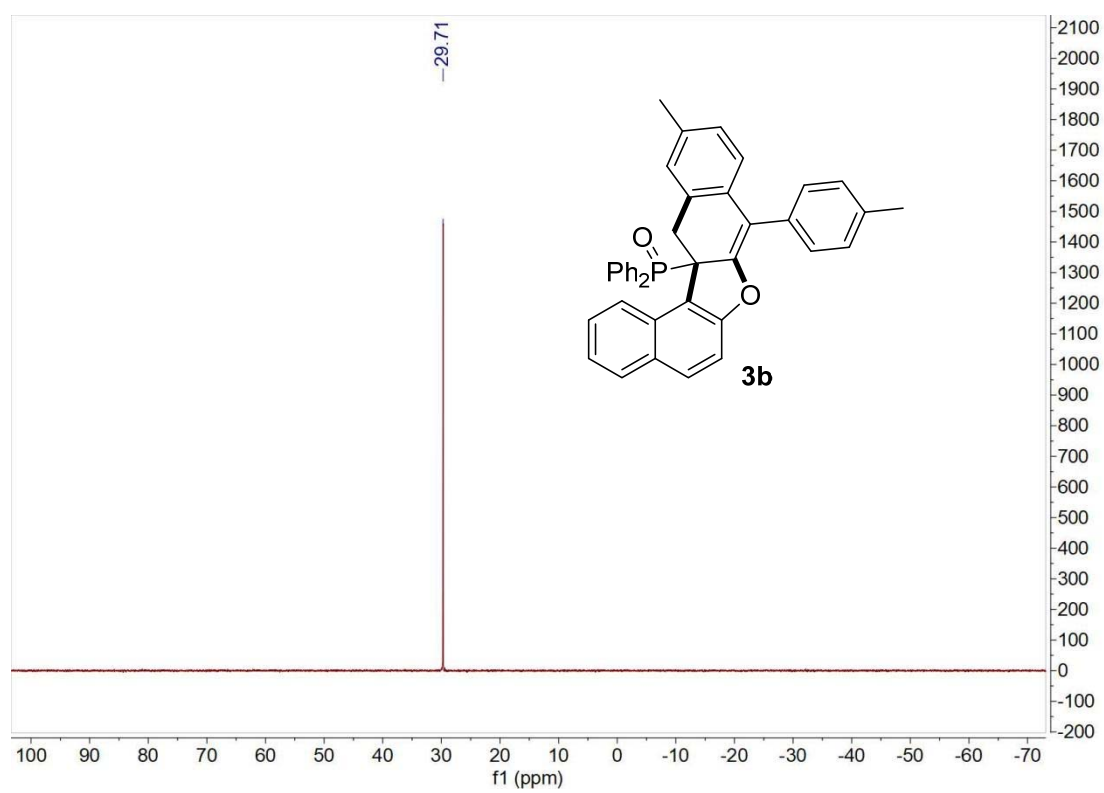


³¹P-NMR (162 MHz, CDCl₃) of **3a**



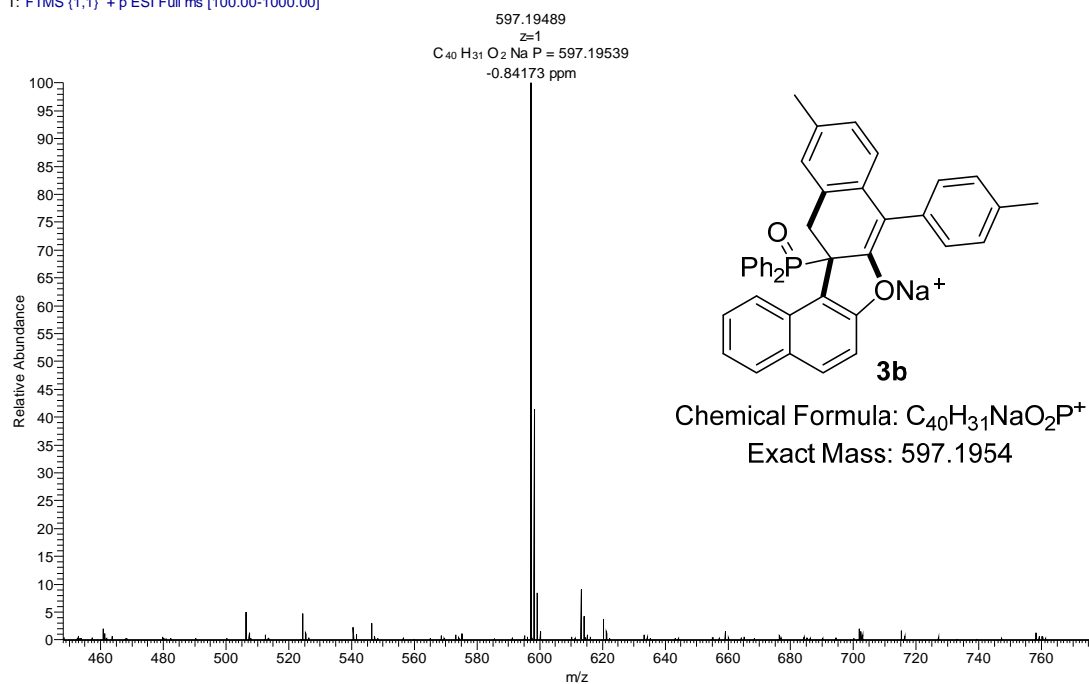


¹³C-NMR (101 MHz, CDCl₃) of **3b**

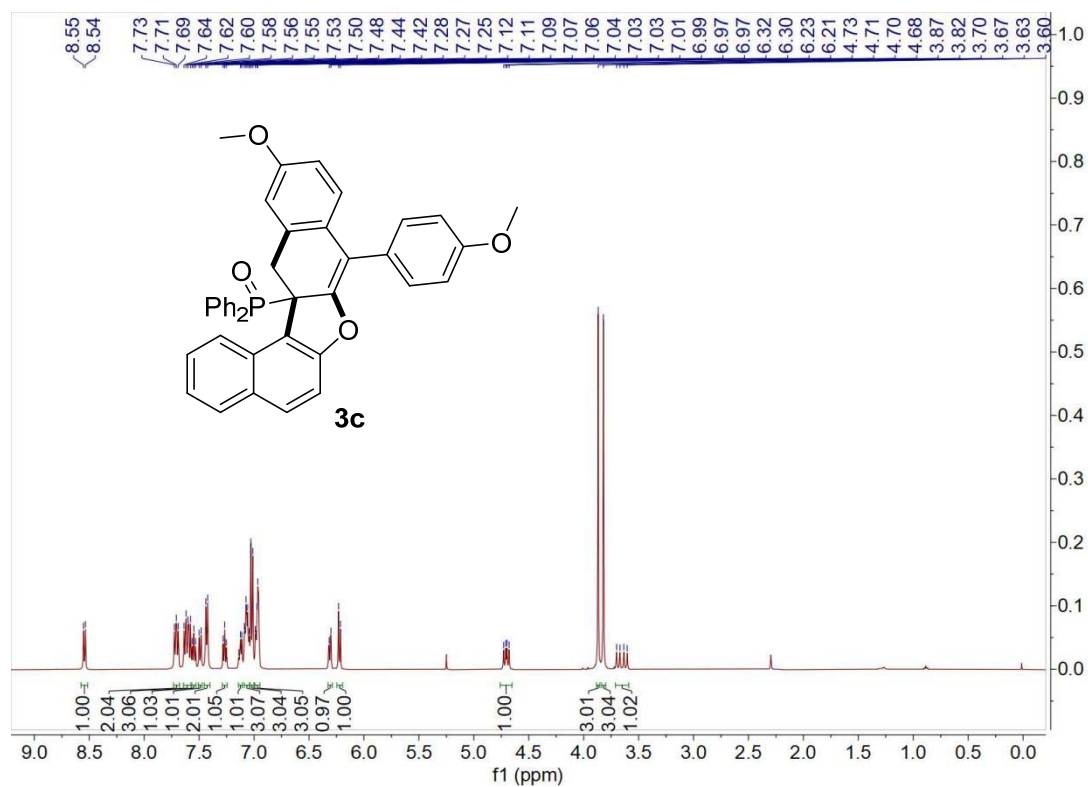


³¹P-NMR (162 MHz, CDCl₃) of **3b**

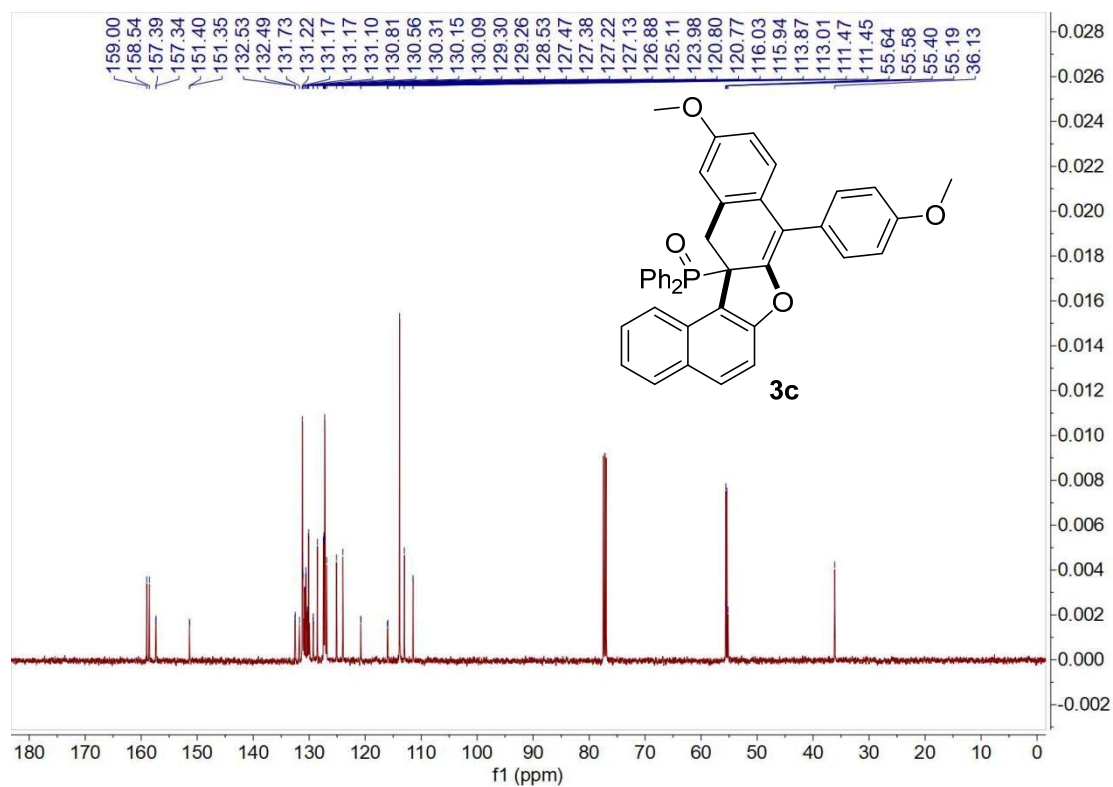
20210515-26 #35 RT: 0.46 AV: 1 NL: 8.08E4
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



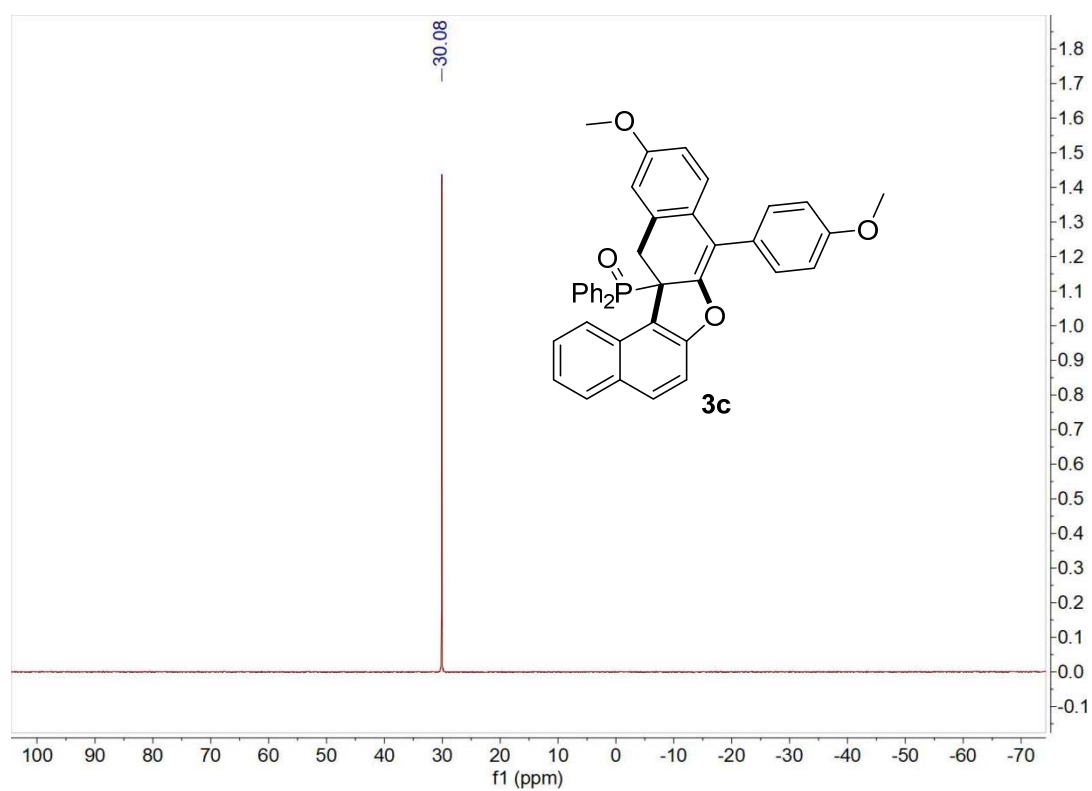
HRMS of **3b**



¹H-NMR (500 MHz, CDCl₃) of **3c**

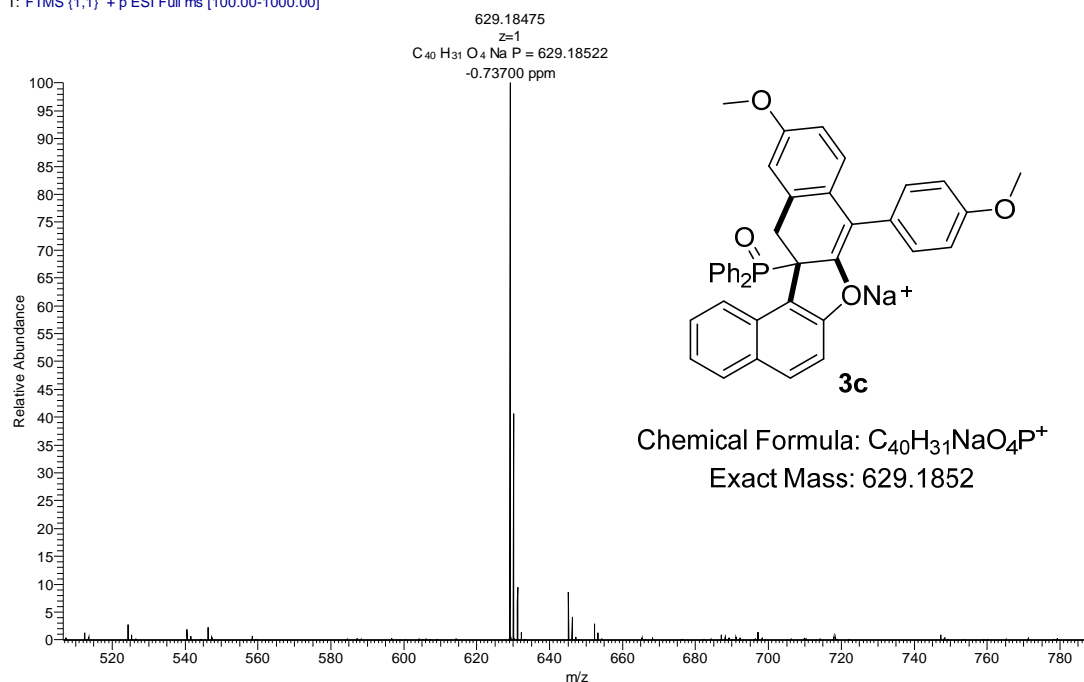


^{13}C -NMR (126 MHz, CDCl_3) of **3c**

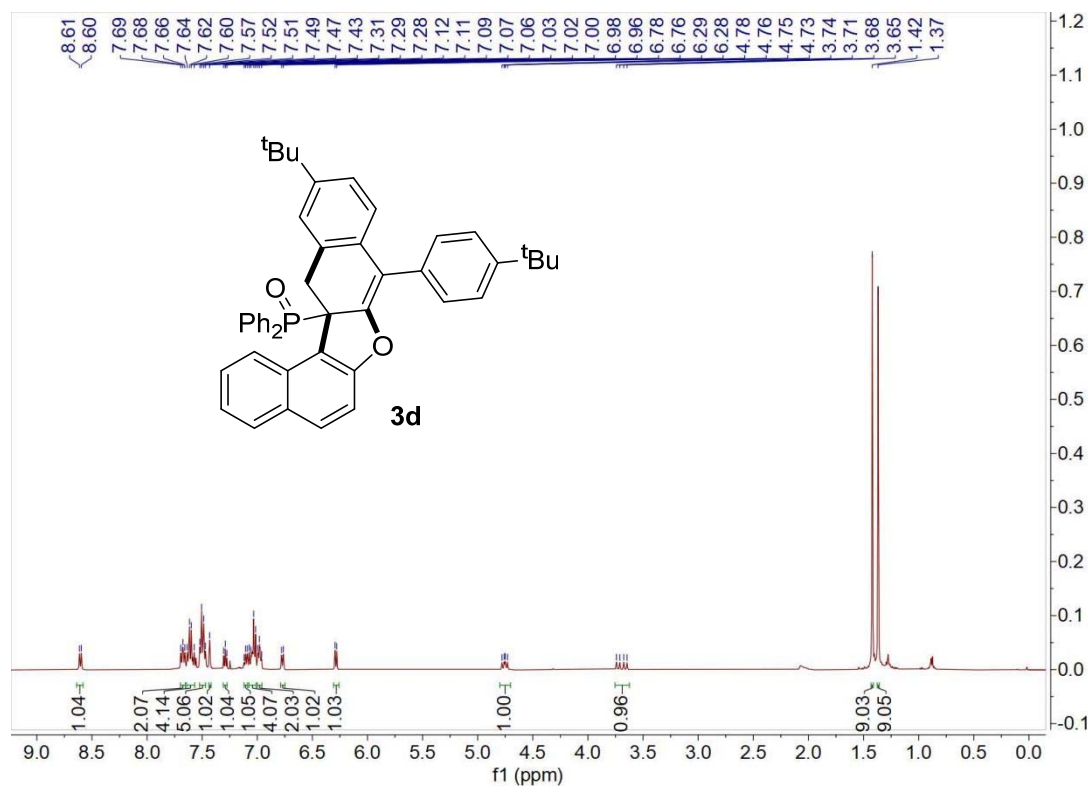


^{31}P -NMR (202 MHz, CDCl_3) of **3c**

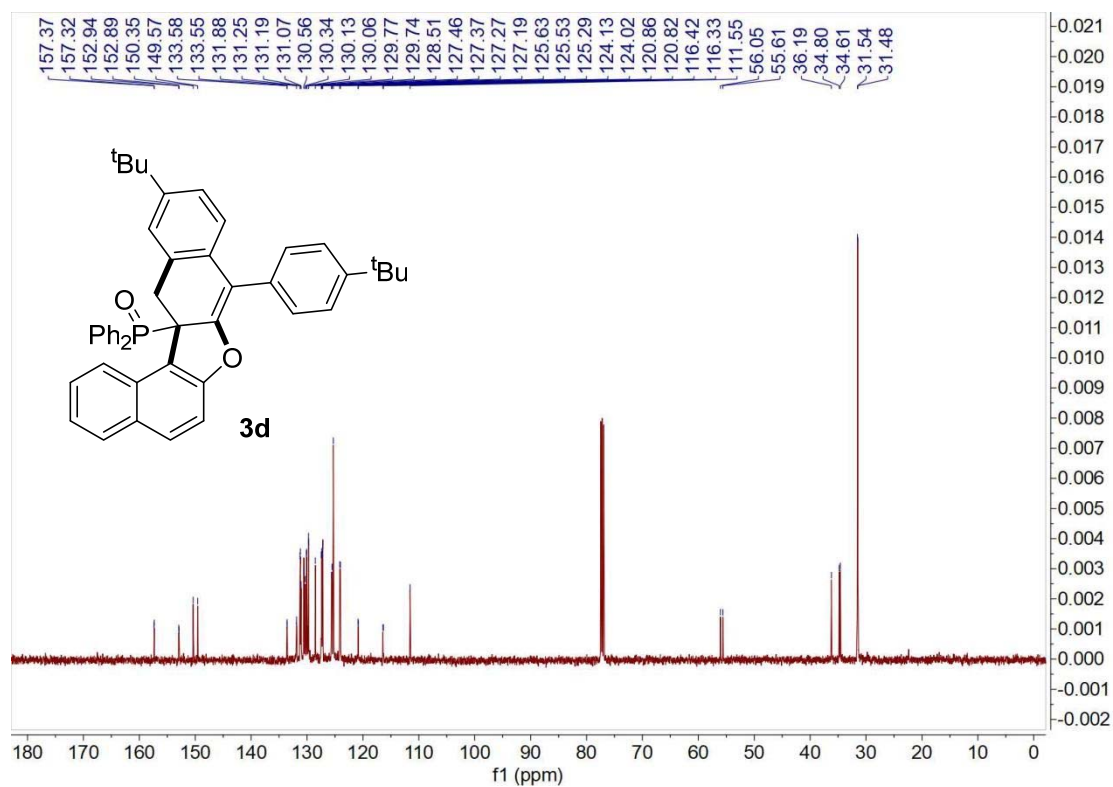
20210515-24 #25 RT: 0.40 AV: 1 NL: 1.39E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



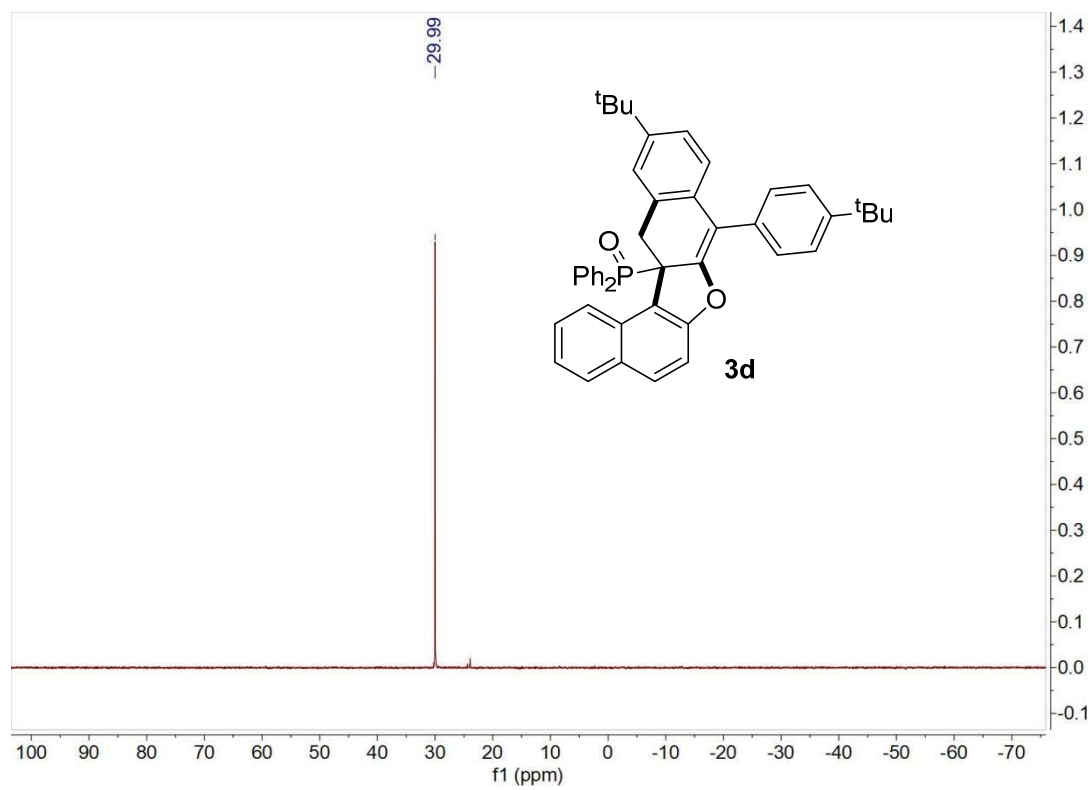
HRMS of **3c**



¹H-NMR (500 MHz, CDCl₃) of **3d**

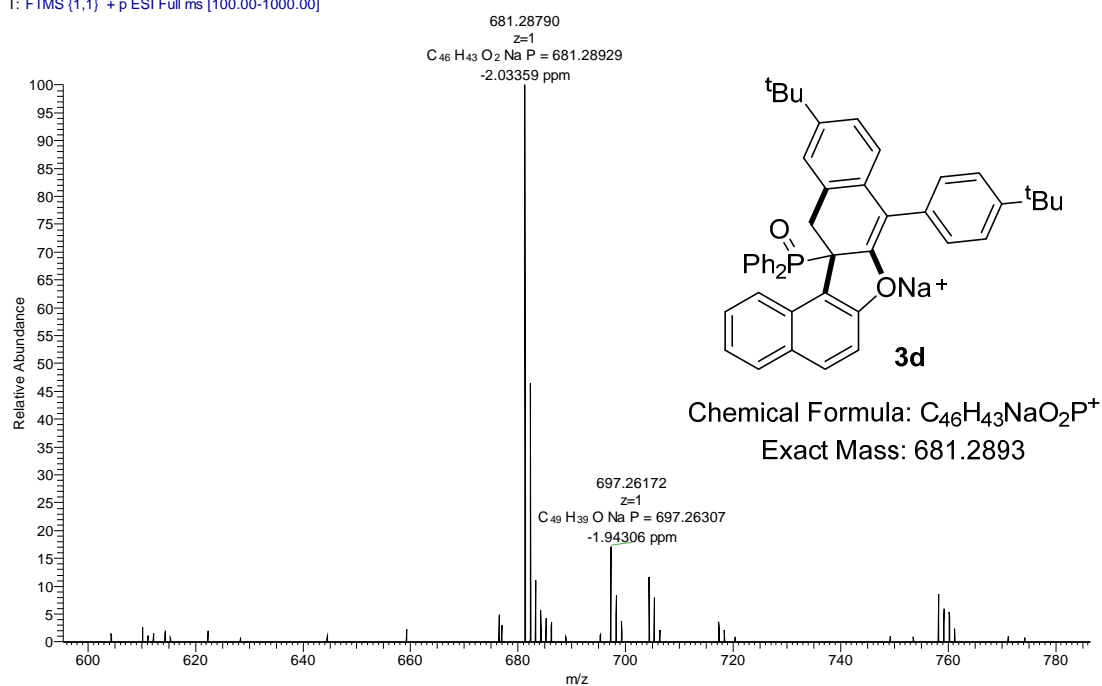


^{13}C -NMR (126 MHz, CDCl_3) of **3d**

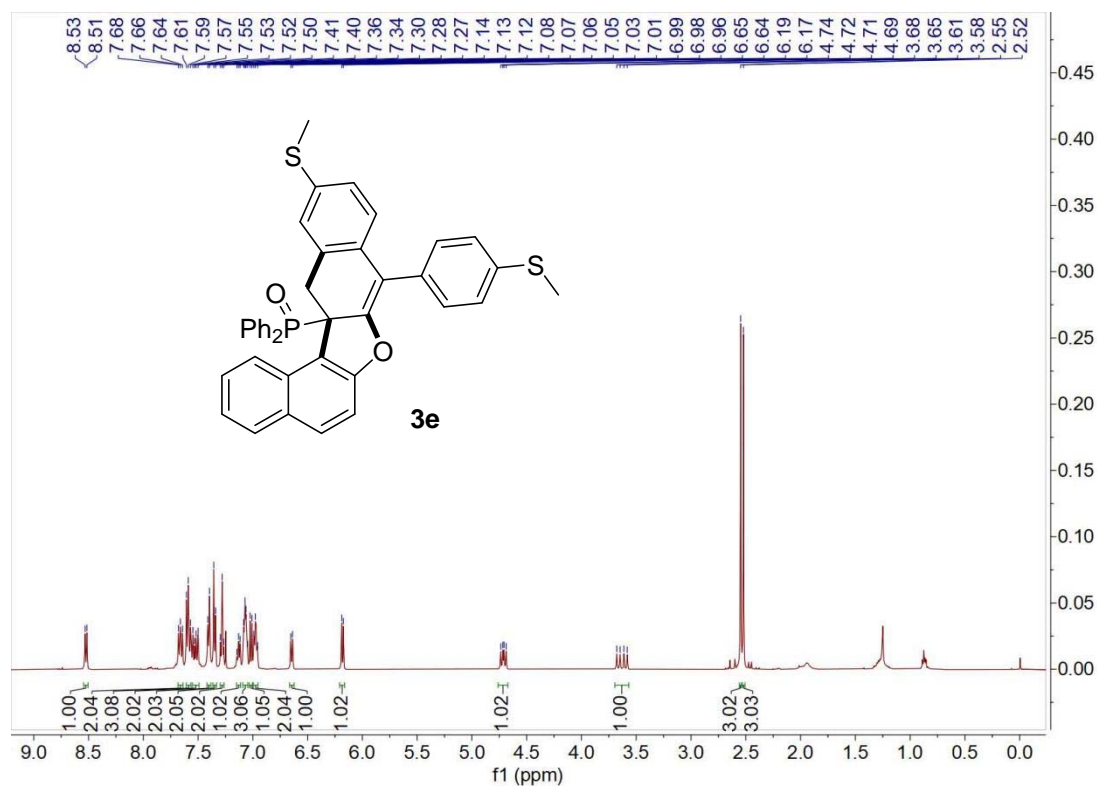


^{31}P -NMR (202 MHz, CDCl_3) of **3d**

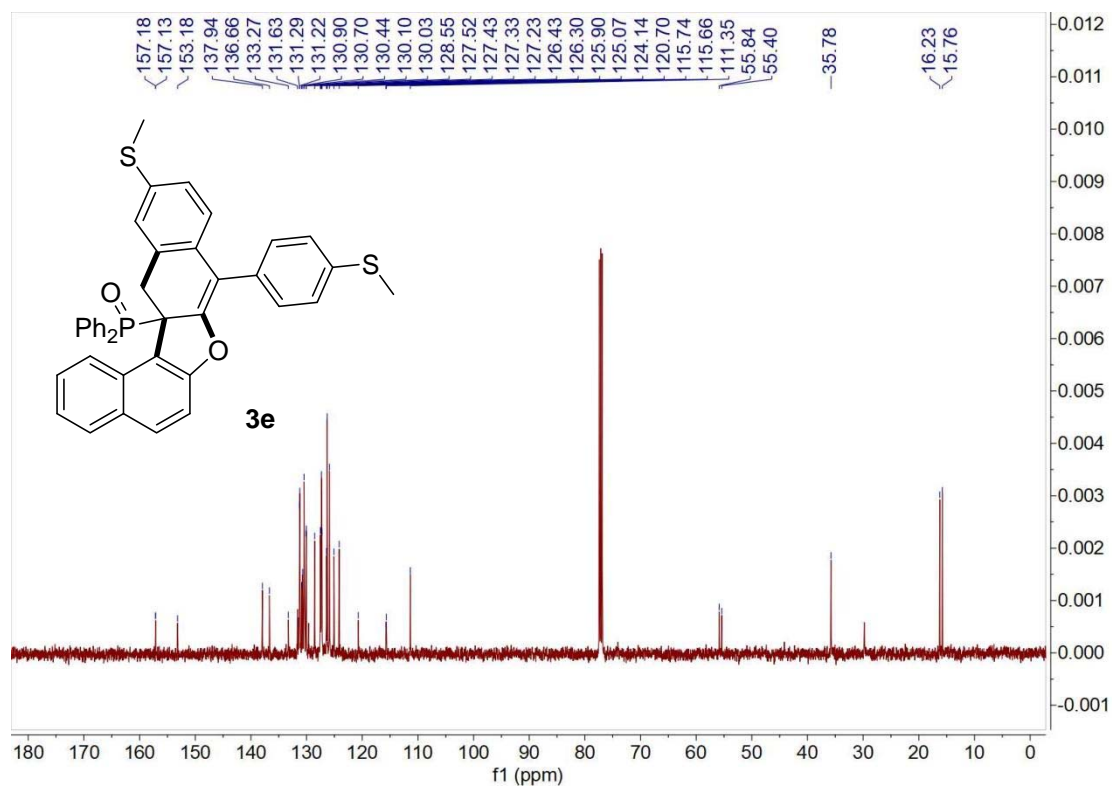
20210515-18_210517102502 #41 RT: 0.54 AV: 1 NL: 2.24E4
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



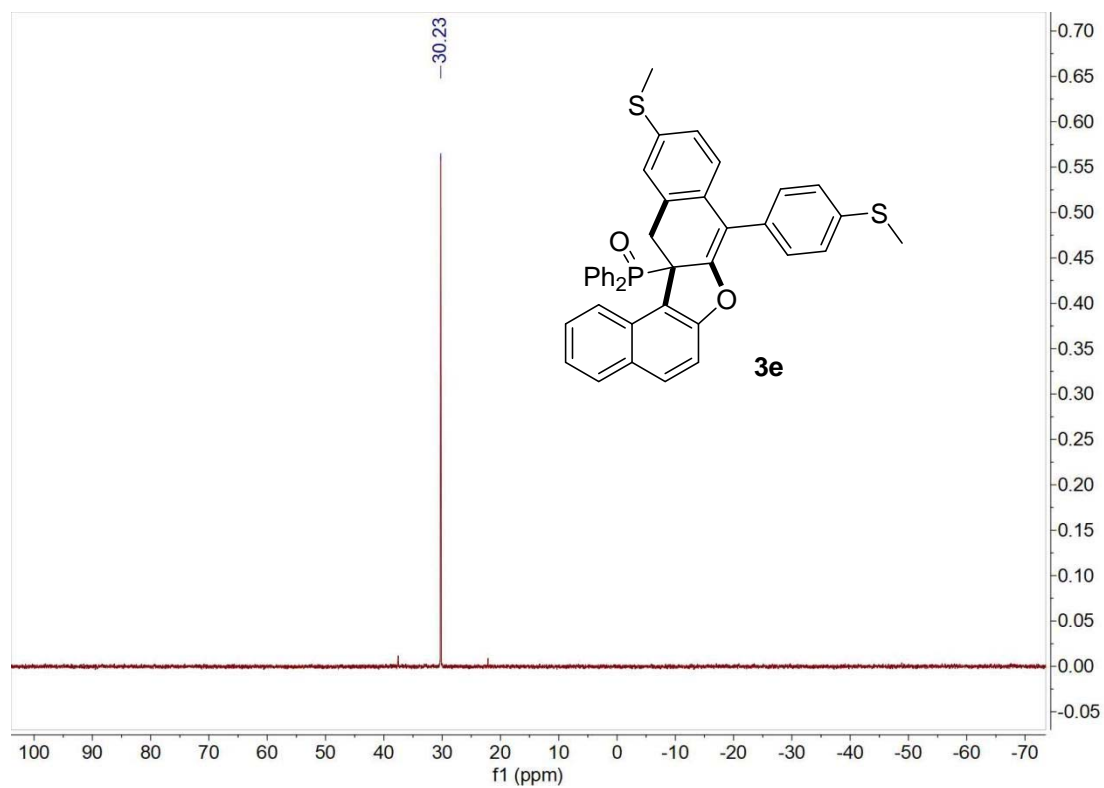
HRMS of **3d**



¹H-NMR (500 MHz, CDCl₃) of **3e**

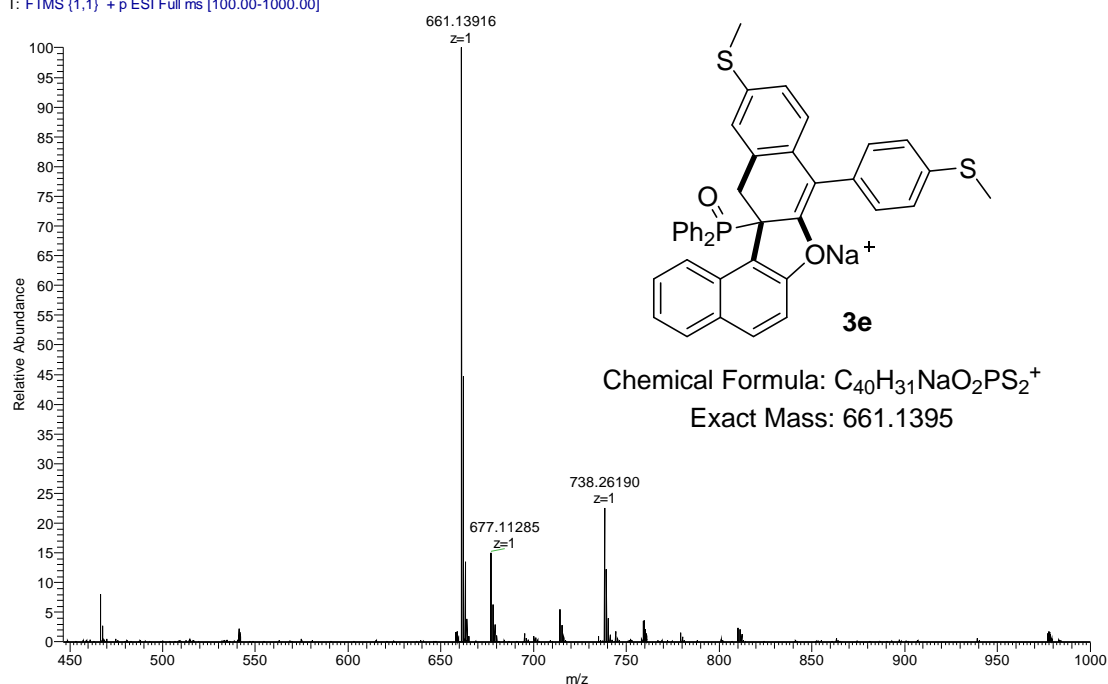


^{13}C -NMR (126 MHz, CDCl_3) of **3e**

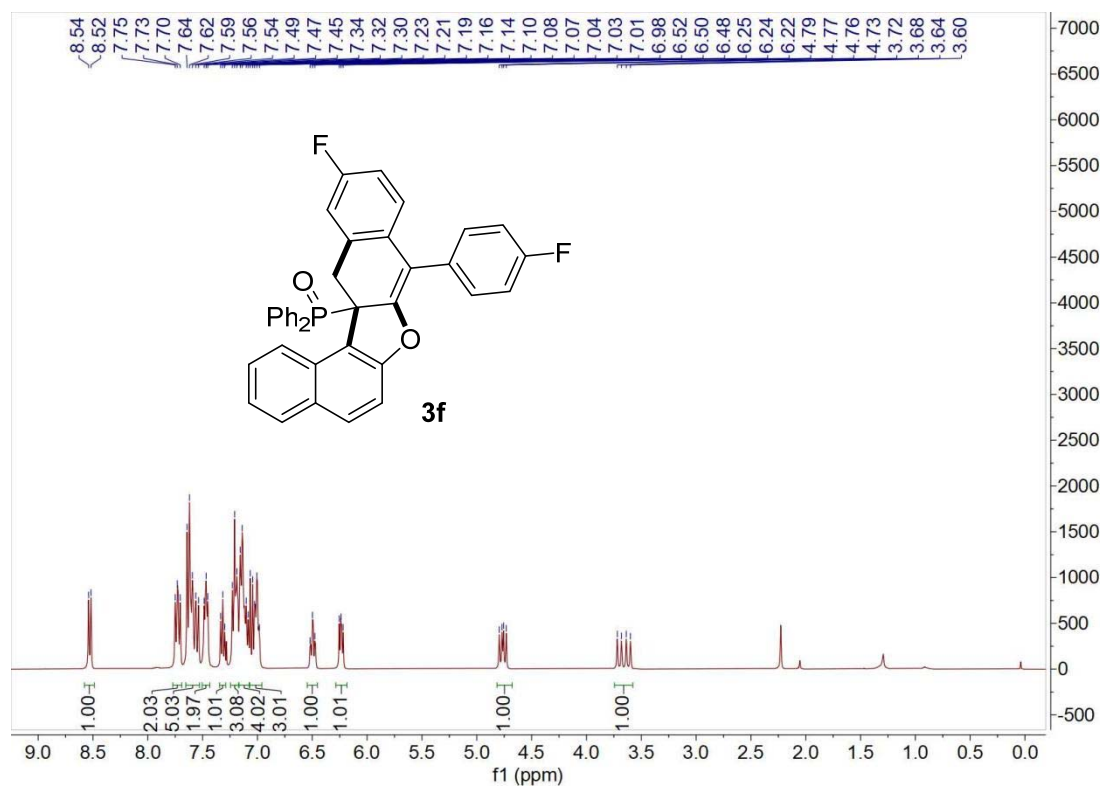


^{31}P -NMR (202 MHz, CDCl_3) of **3e**

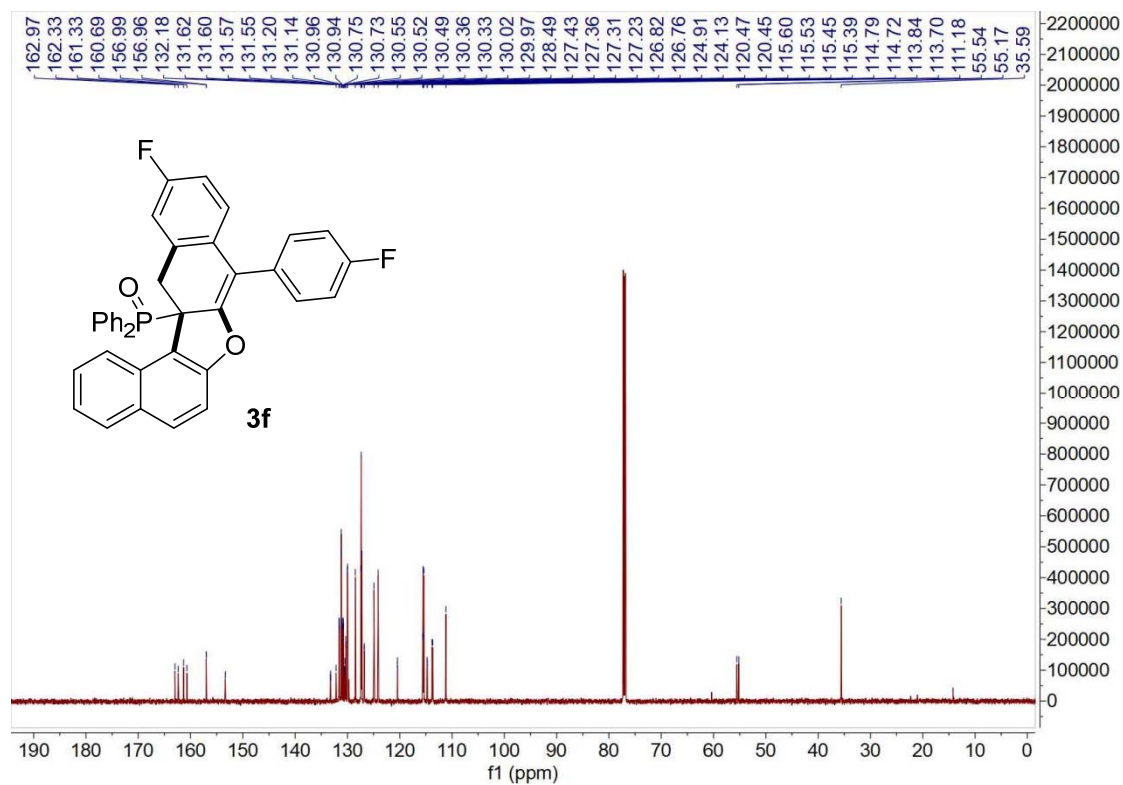
20210719-22 #25 RT: 0.31 AV: 1 NL: 4.37E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



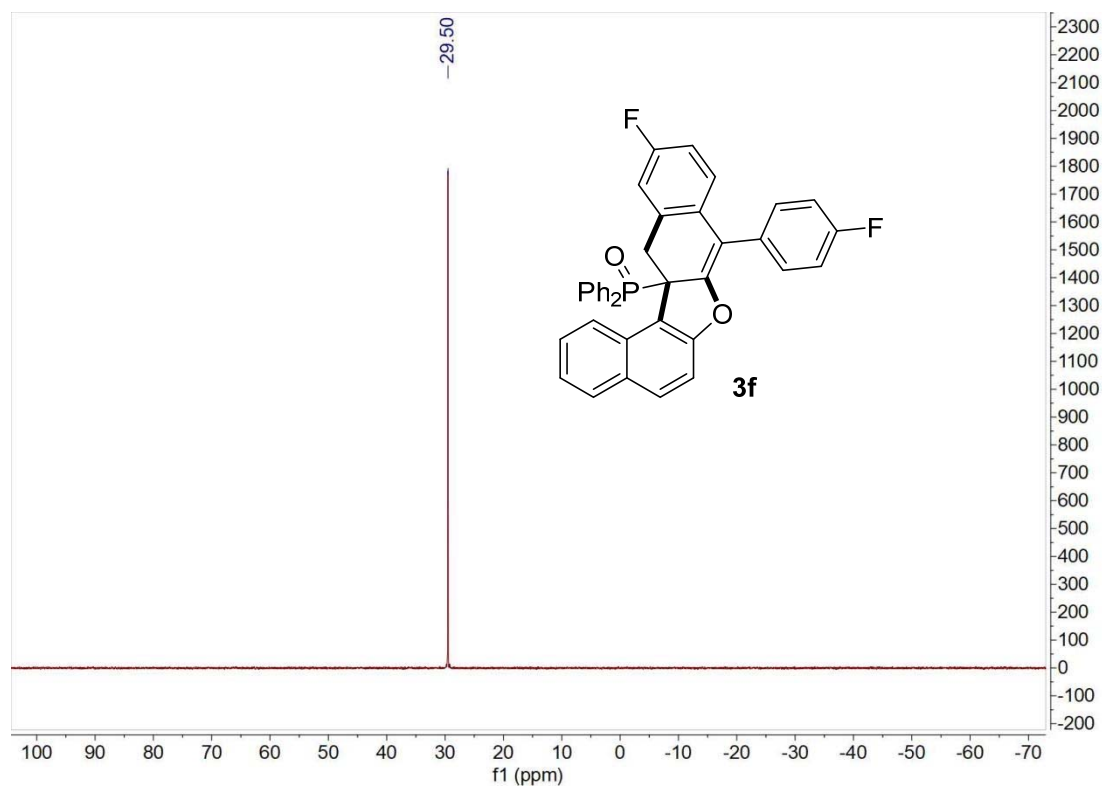
HRMS of **3e**



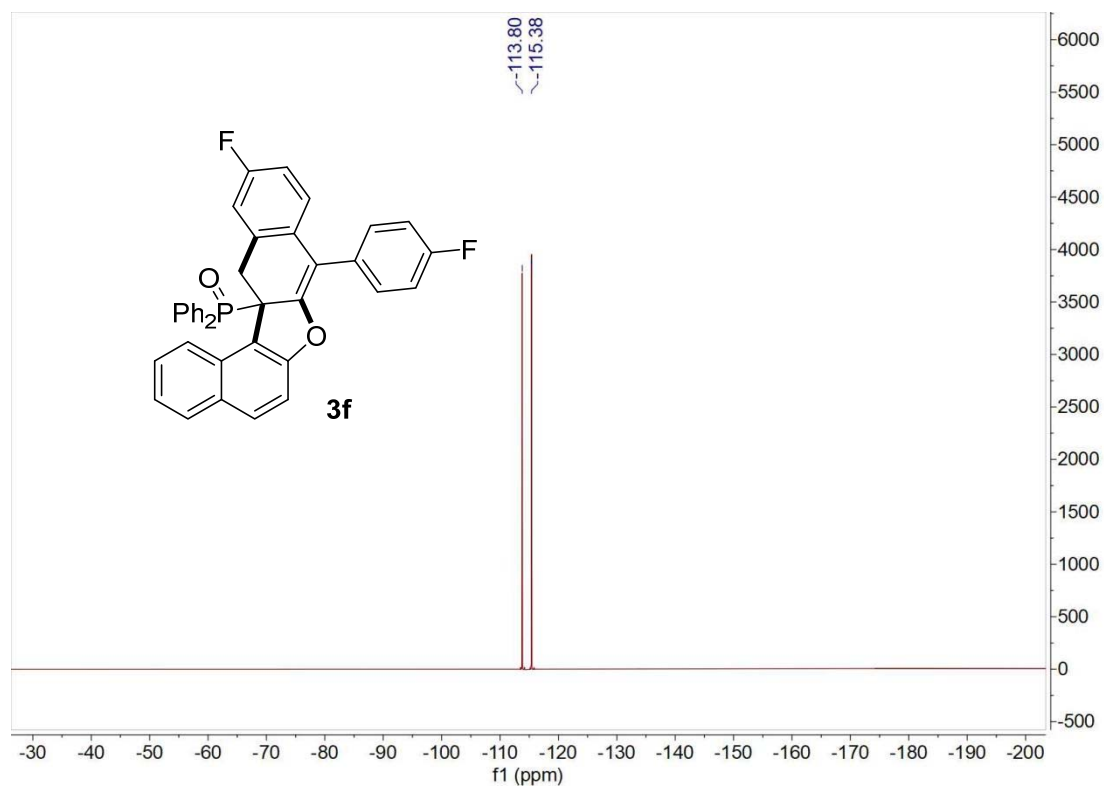
¹H-NMR (400 MHz, CDCl₃) of **3f**



^{13}C -NMR (151 MHz, CDCl_3) of **3f**

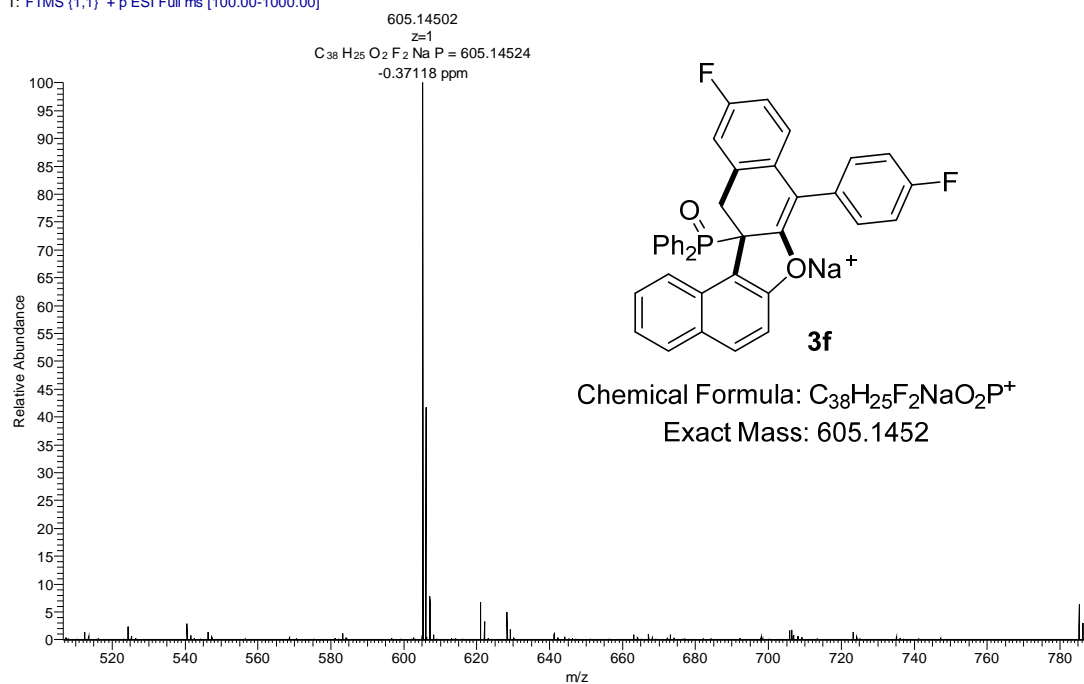


^{31}P -NMR (162 MHz, CDCl_3) of **3f**

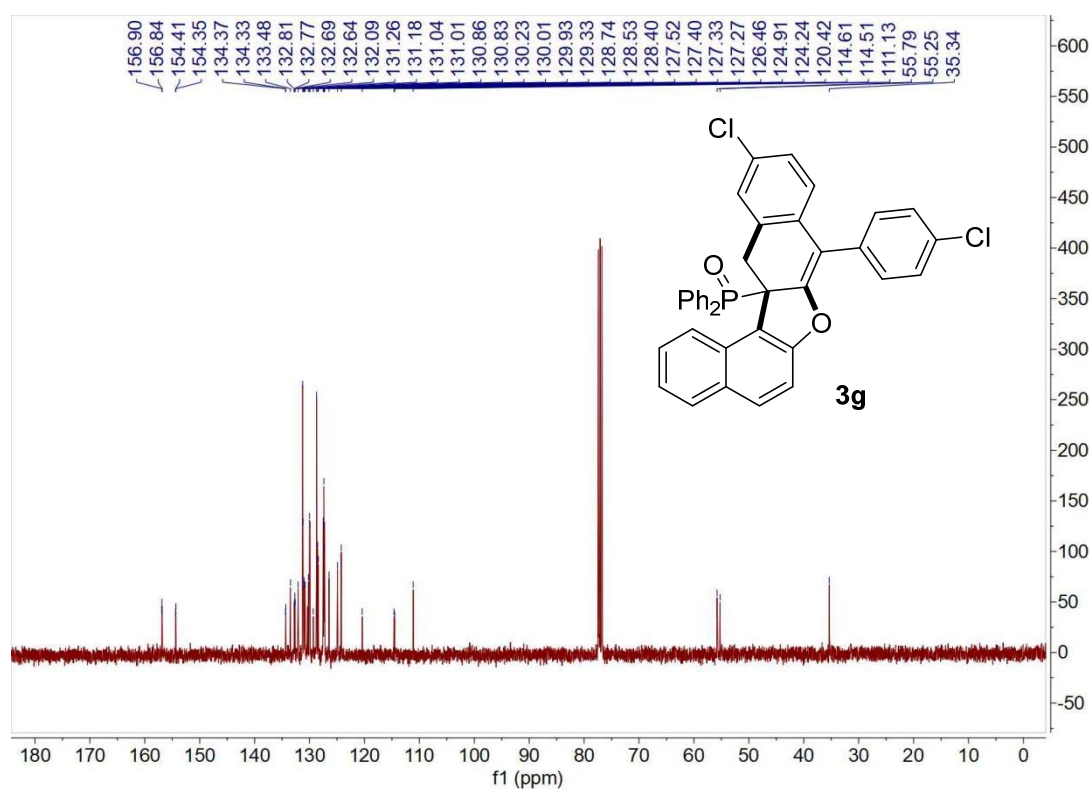
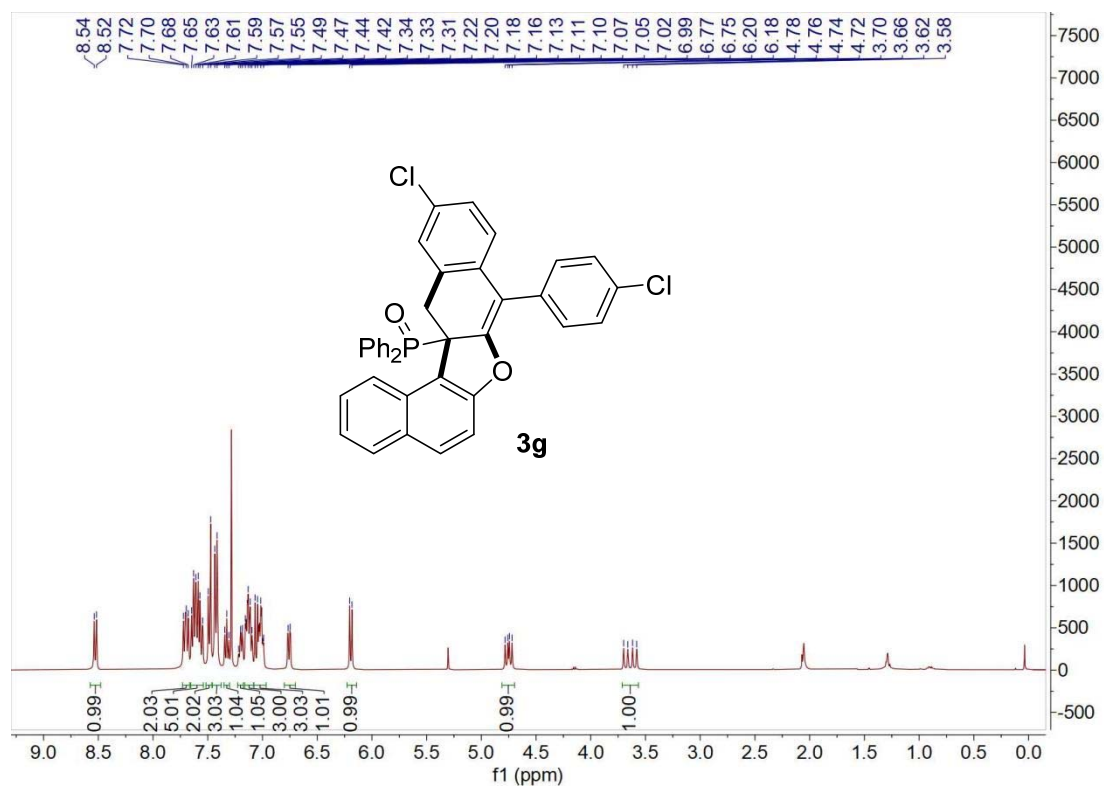


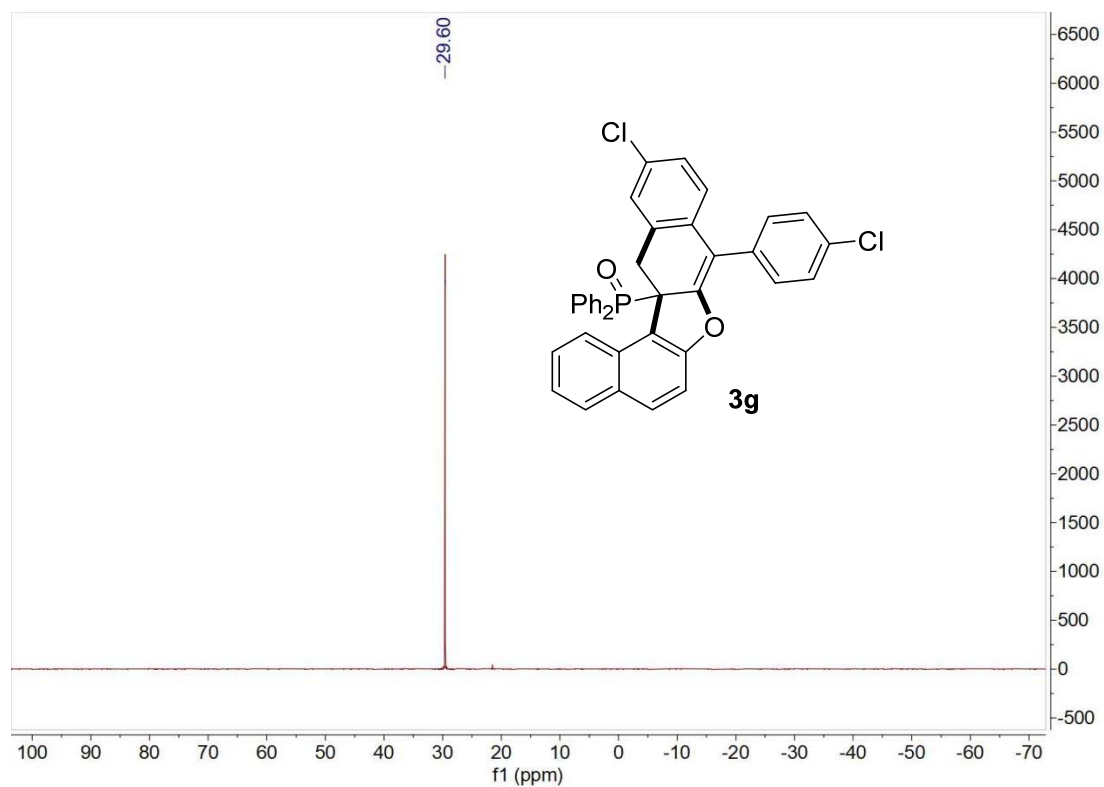
^{19}F -NMR (376 MHz, CDCl_3) of **3f**

20210515-25 #25 RT: 0.39 AV: 1 NL: 1.70E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



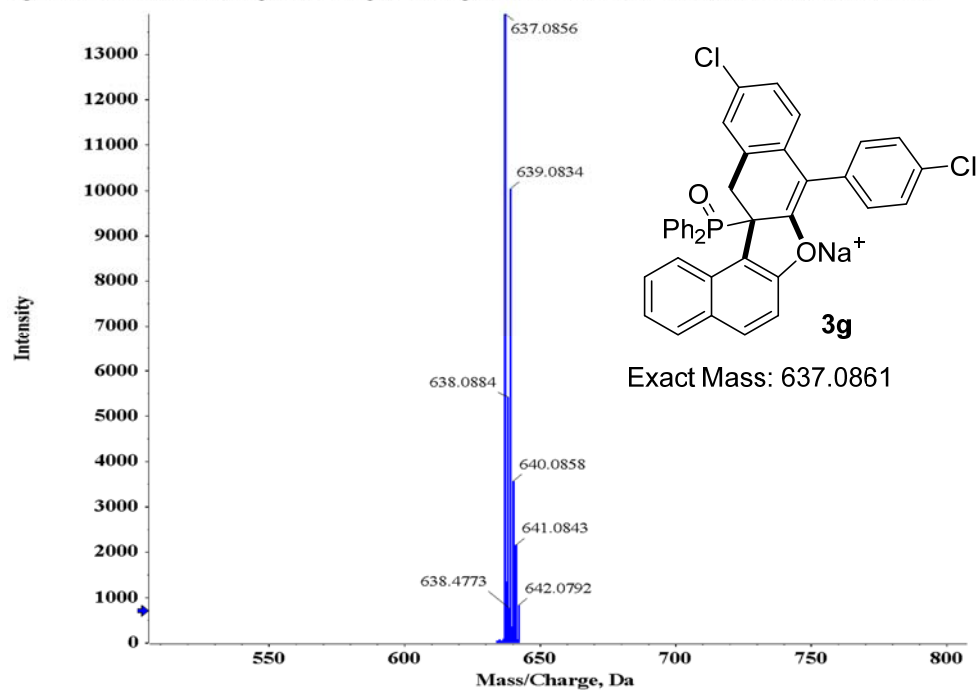
HRMS of **3f**



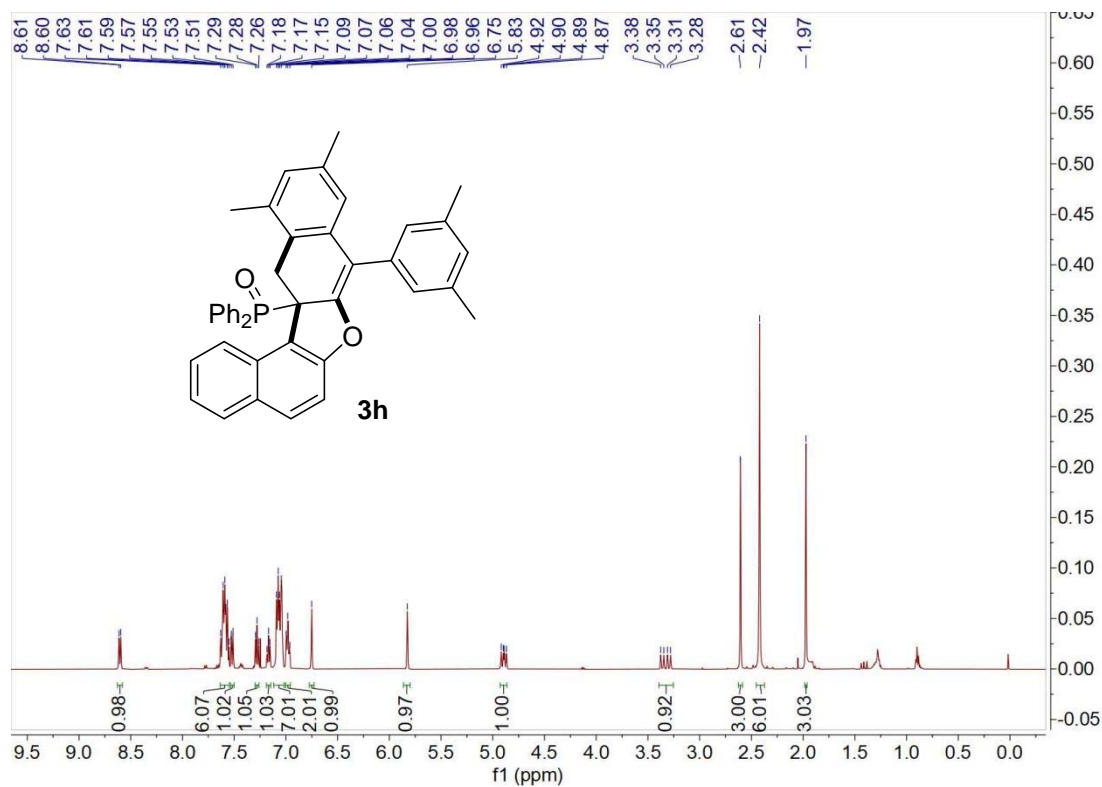


^{31}P -NMR (162 MHz, CDCl_3) of **3g**

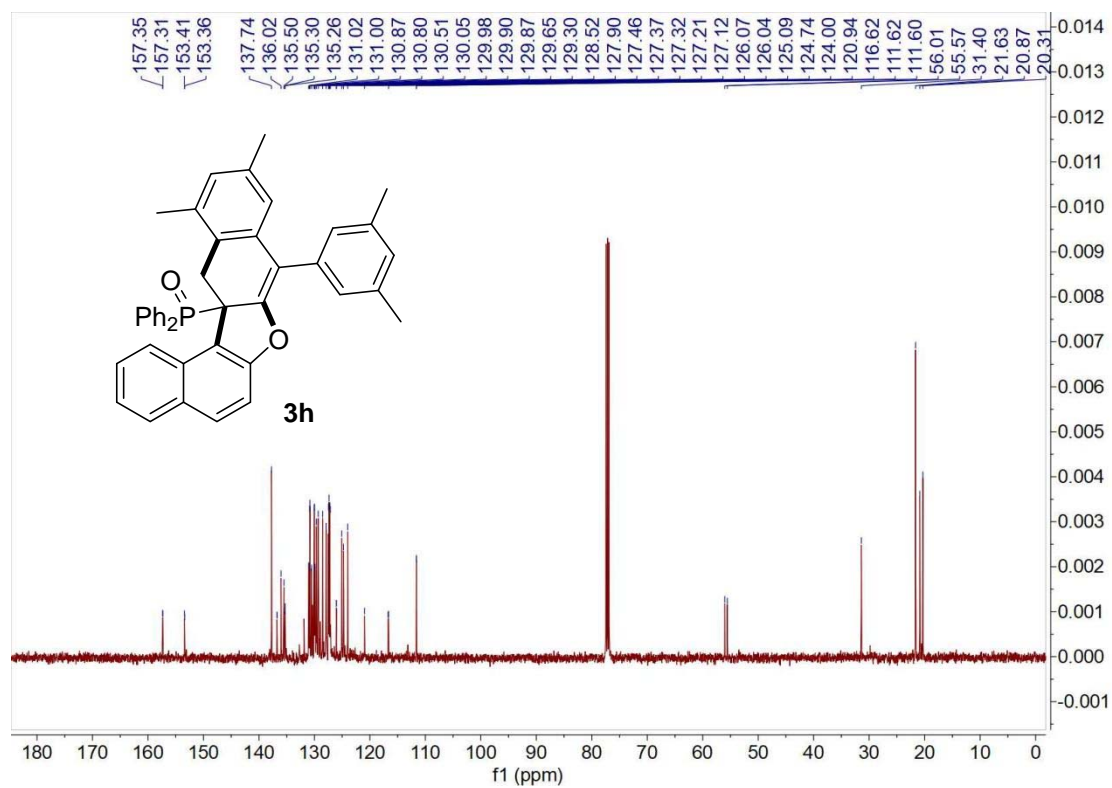
Spectrum from 05.wiff (sample 1) - Sample011, Exp...t 1, +TOF MS (80 - 1000) from 1.174 to 1.218 min



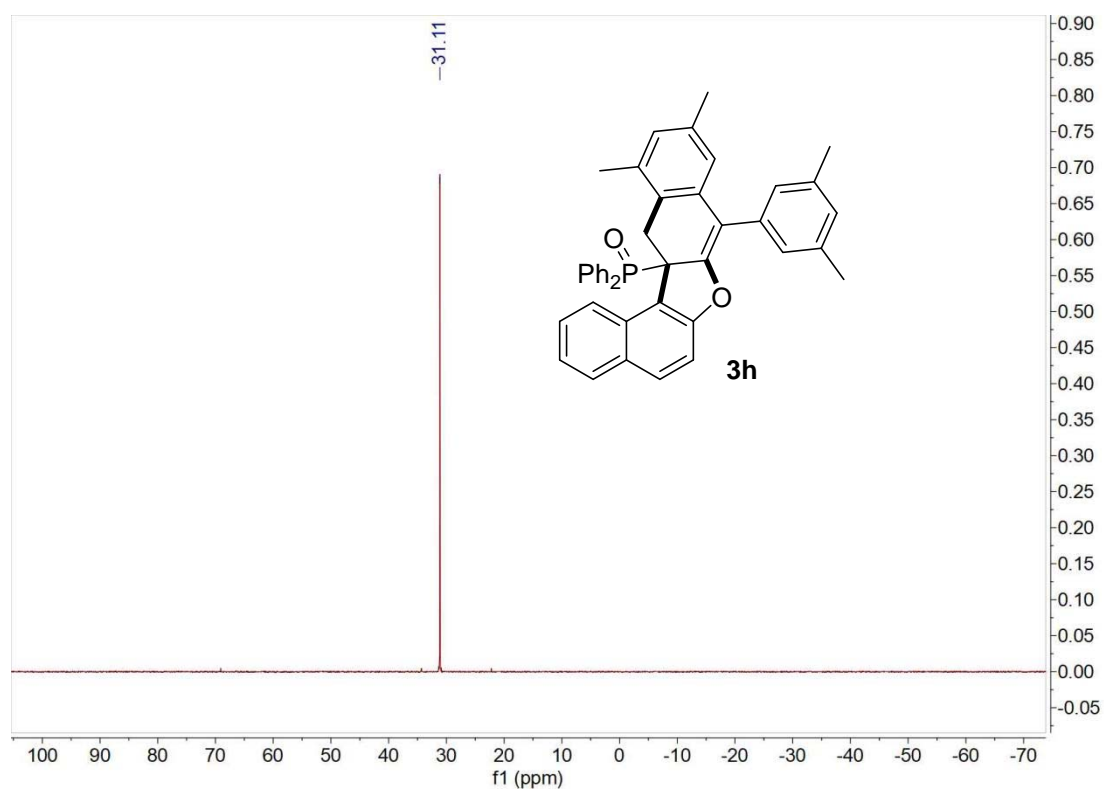
HRMS of **3g**



$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **3h**

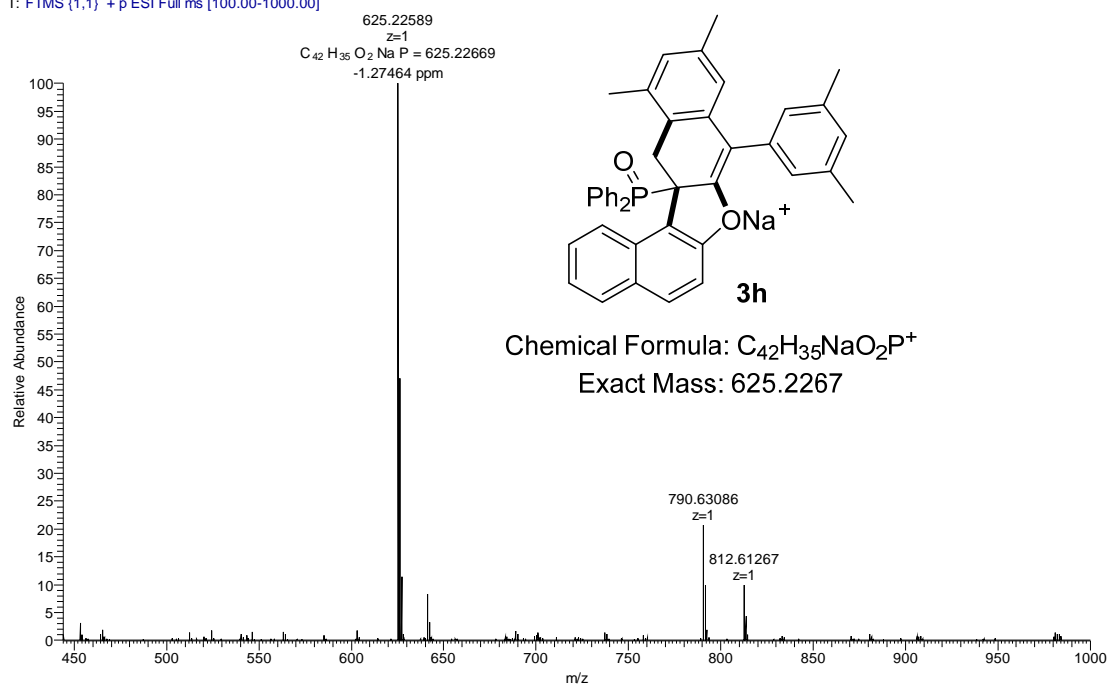


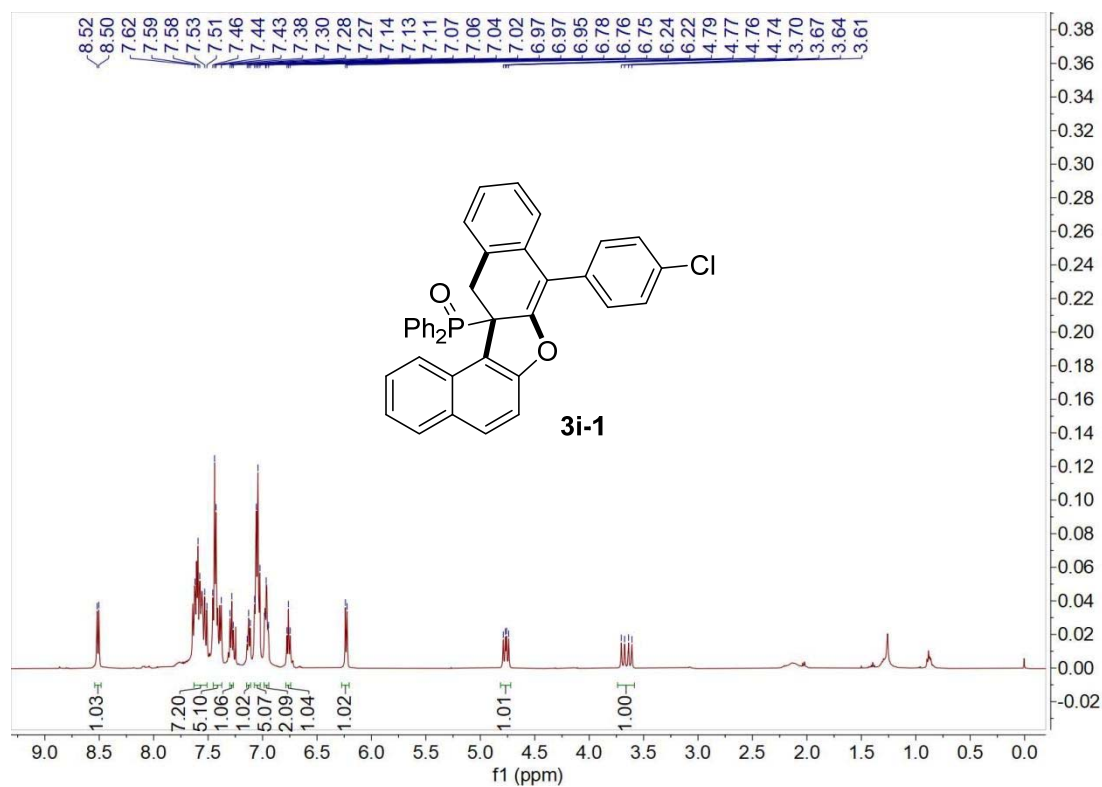
$^{13}\text{C-NMR}$ (126 MHz, CDCl_3) of **3h**



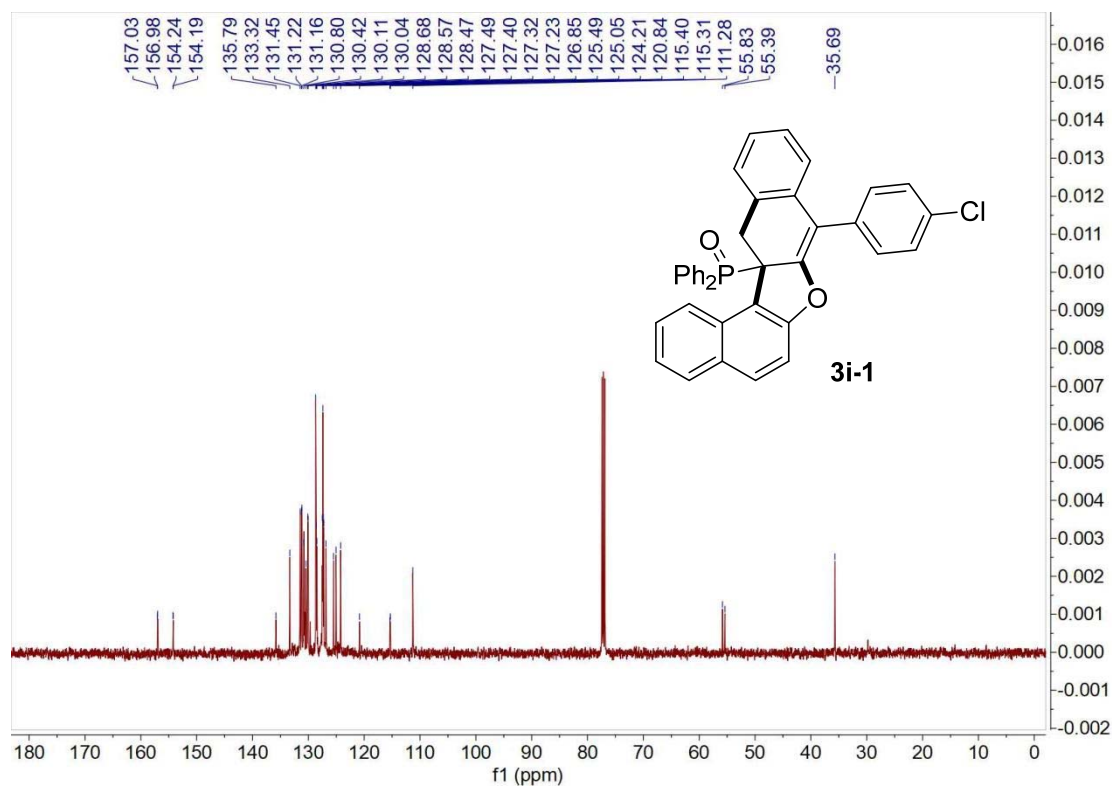
31P-NMR (202 MHz, CDCl₃) of **3h**

20210609-21 #23 RT: 0.38 AV: 1 NL: 5.01E4
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

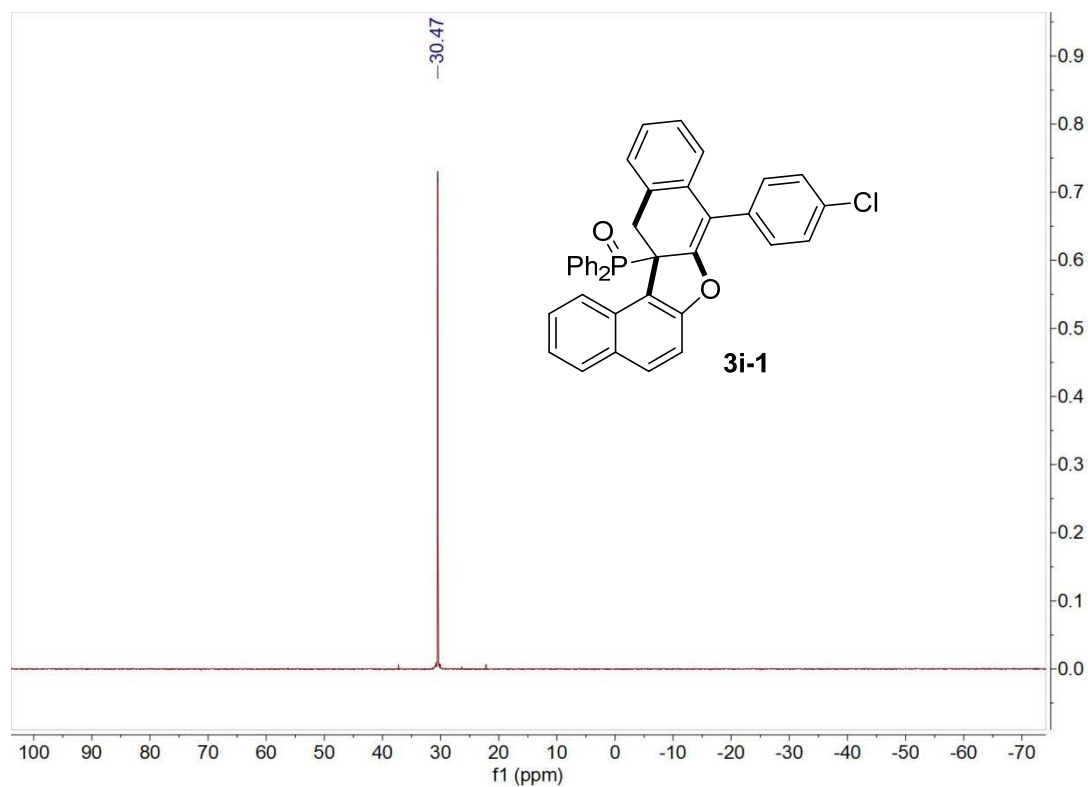




$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **3i-1**

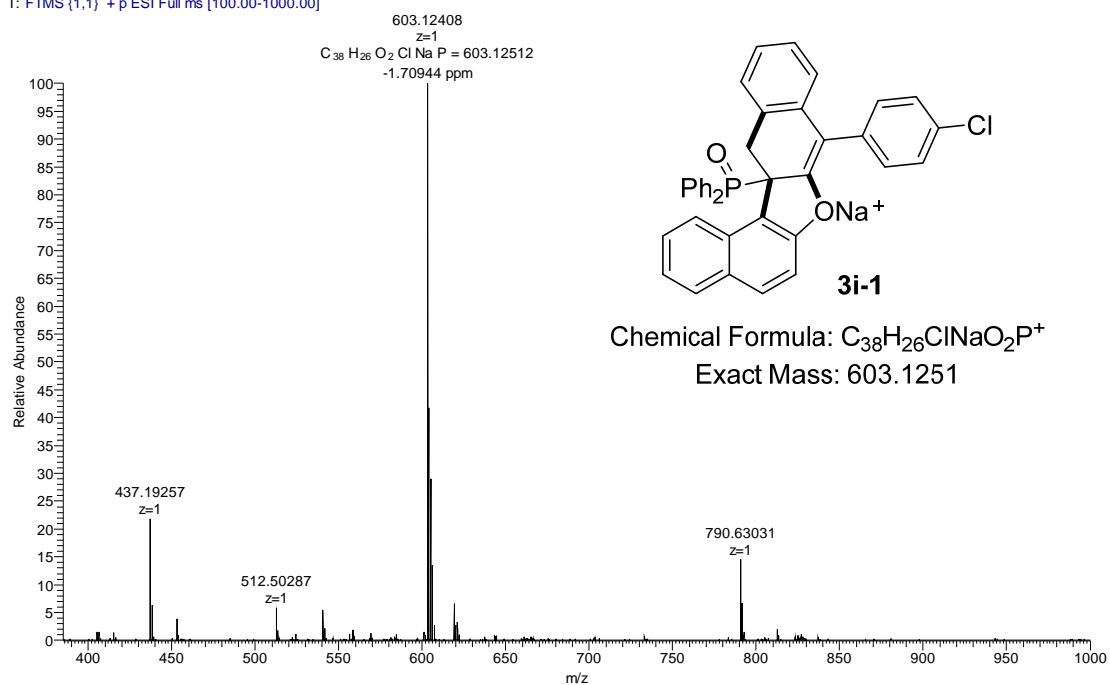


$^{13}\text{C-NMR}$ (126 MHz, CDCl_3) of **3i-1**

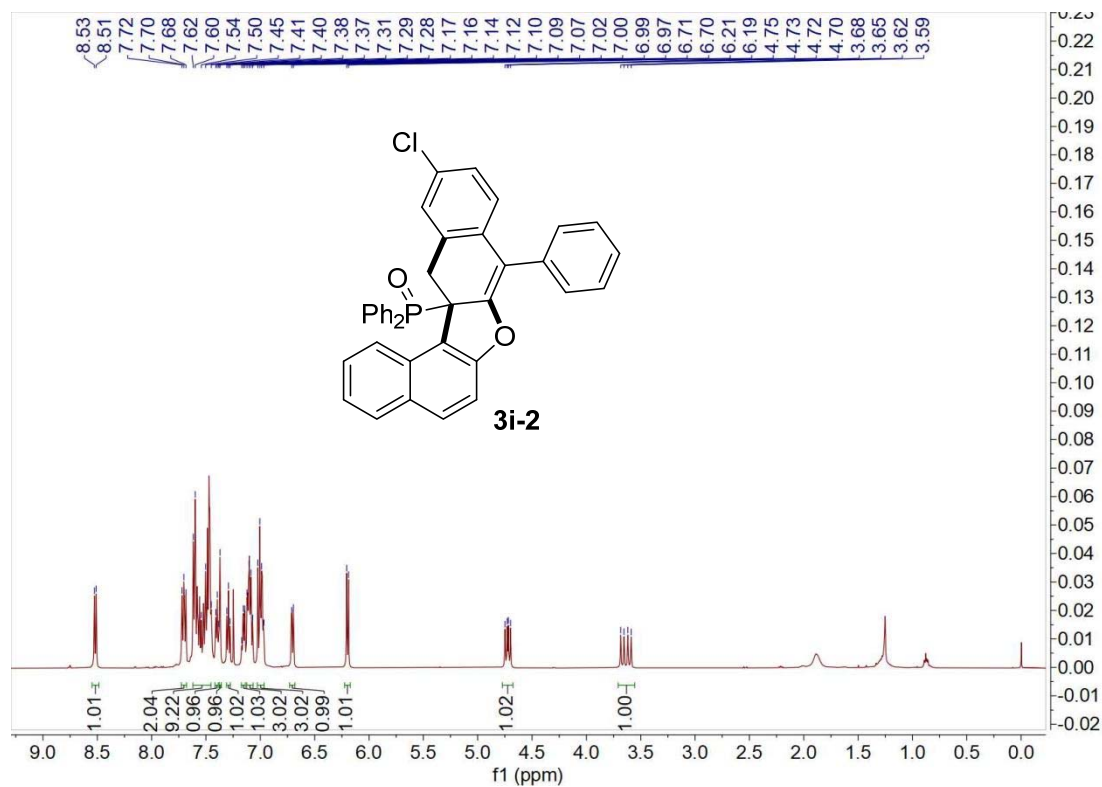


³¹P-NMR (202 MHz, CDCl₃) of **3i-1**

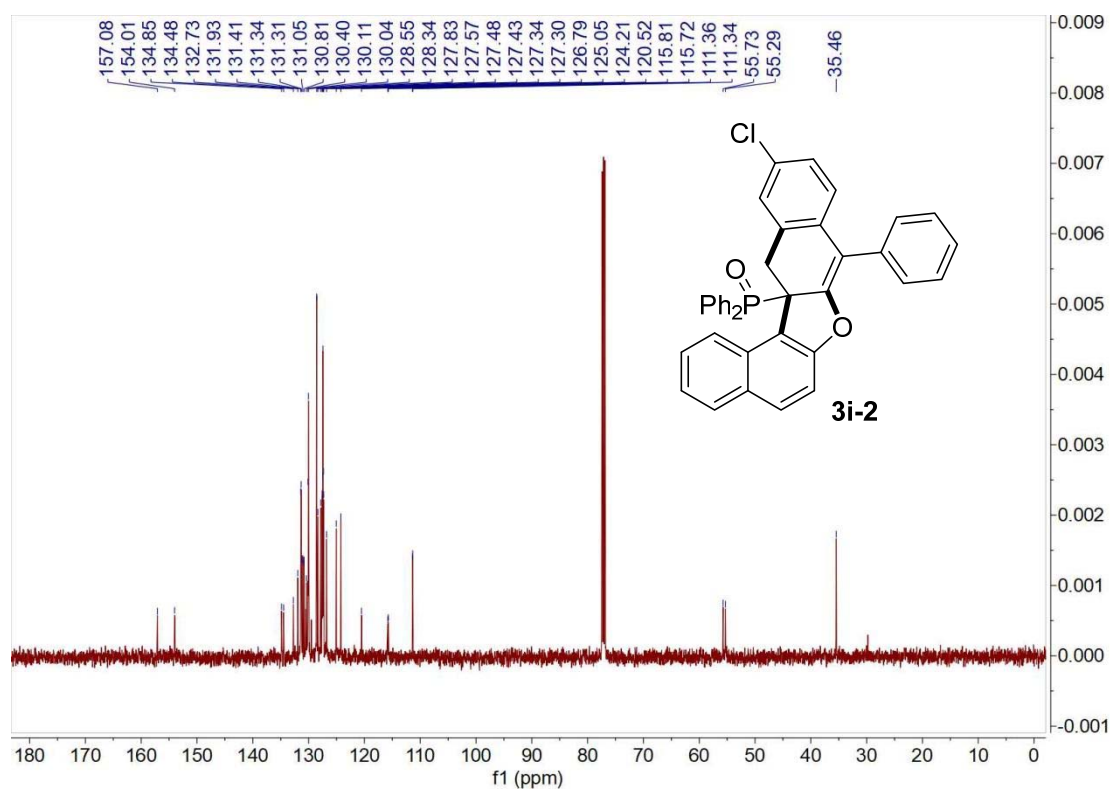
20210609-20 #23 RT: 0.36 AV: 1 NL: 1.03E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



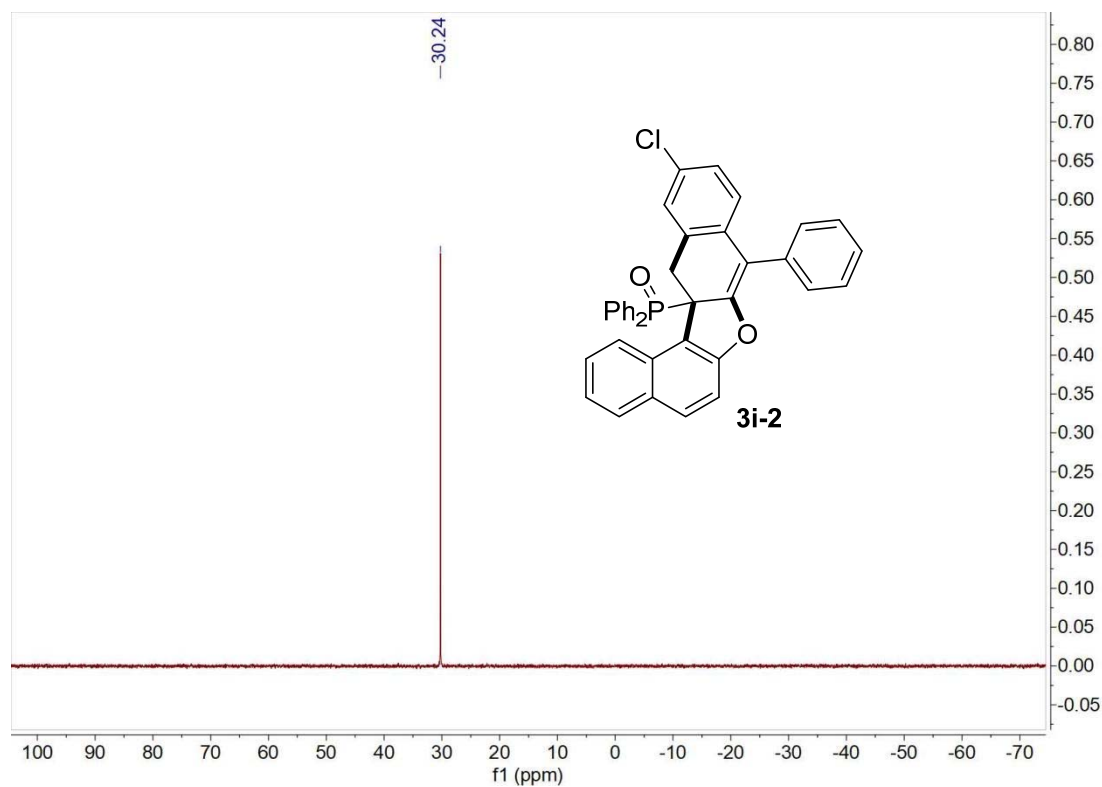
HRMS of **3i-1**



$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **3i-2**

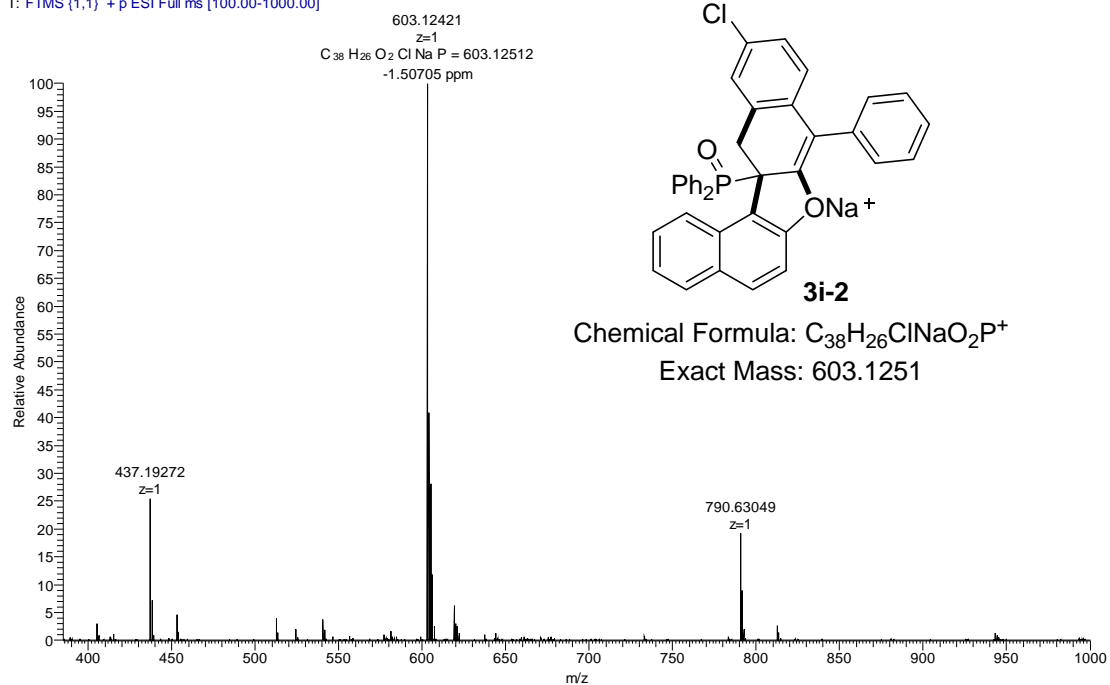


$^{13}\text{C-NMR}$ (126 MHz, CDCl_3) of **3i-2**

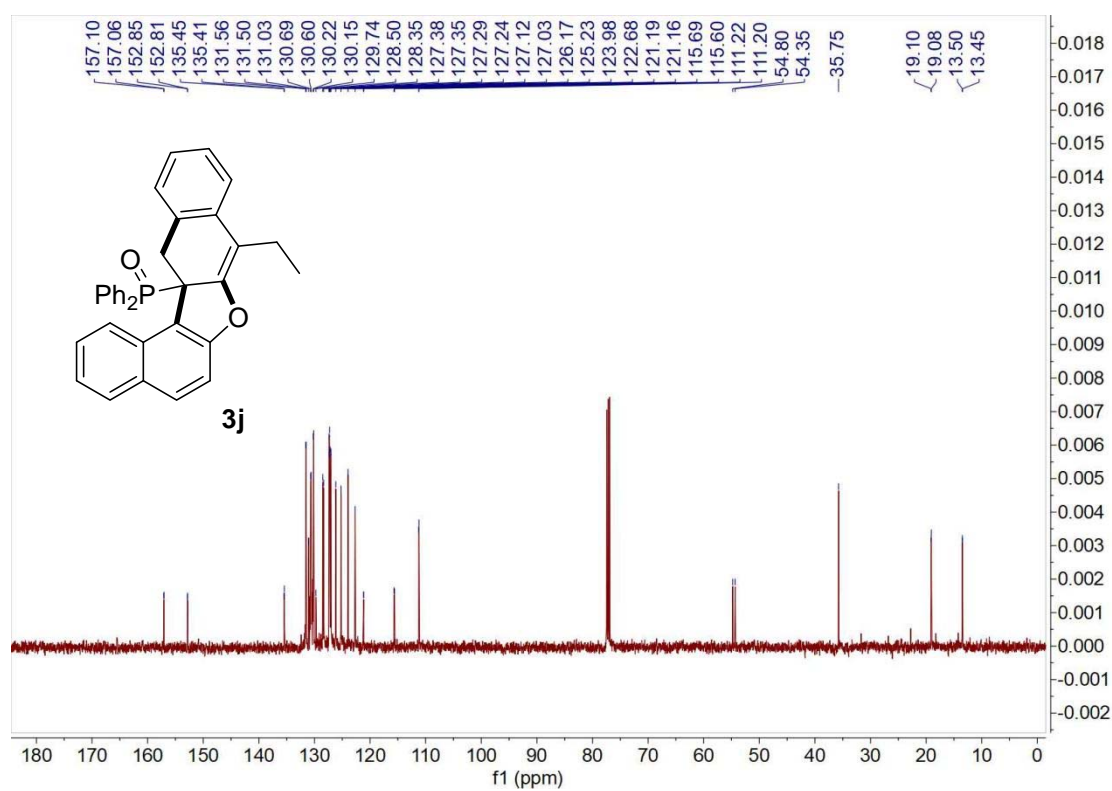
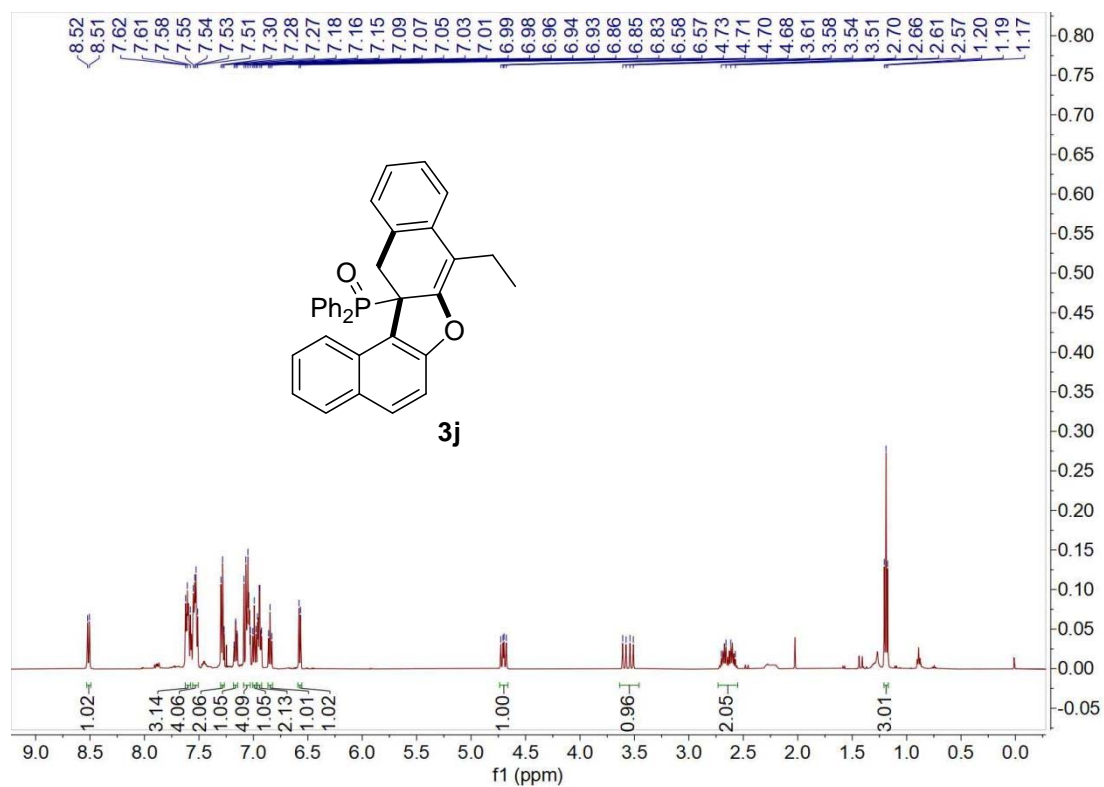


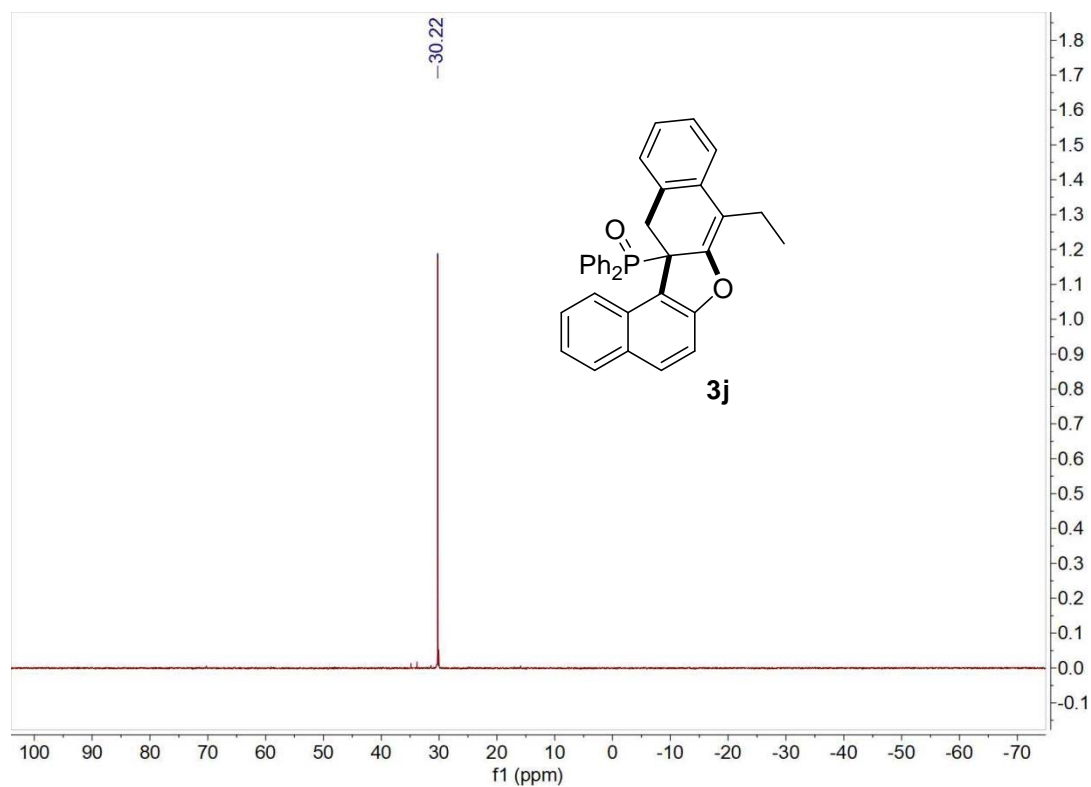
31P-NMR (202 MHz, CDCl₃) of **3i-2**

20210609-19 #23 RT: 0.36 AV: 1 NL: 9.01E4
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



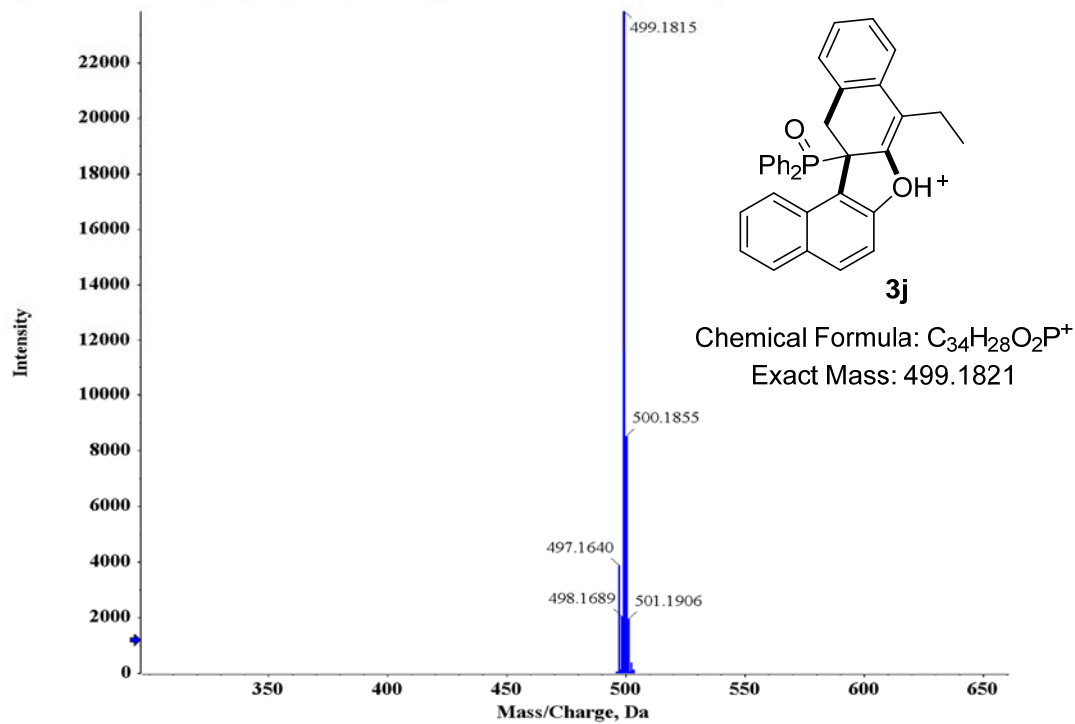
HRMS of **3i-2**



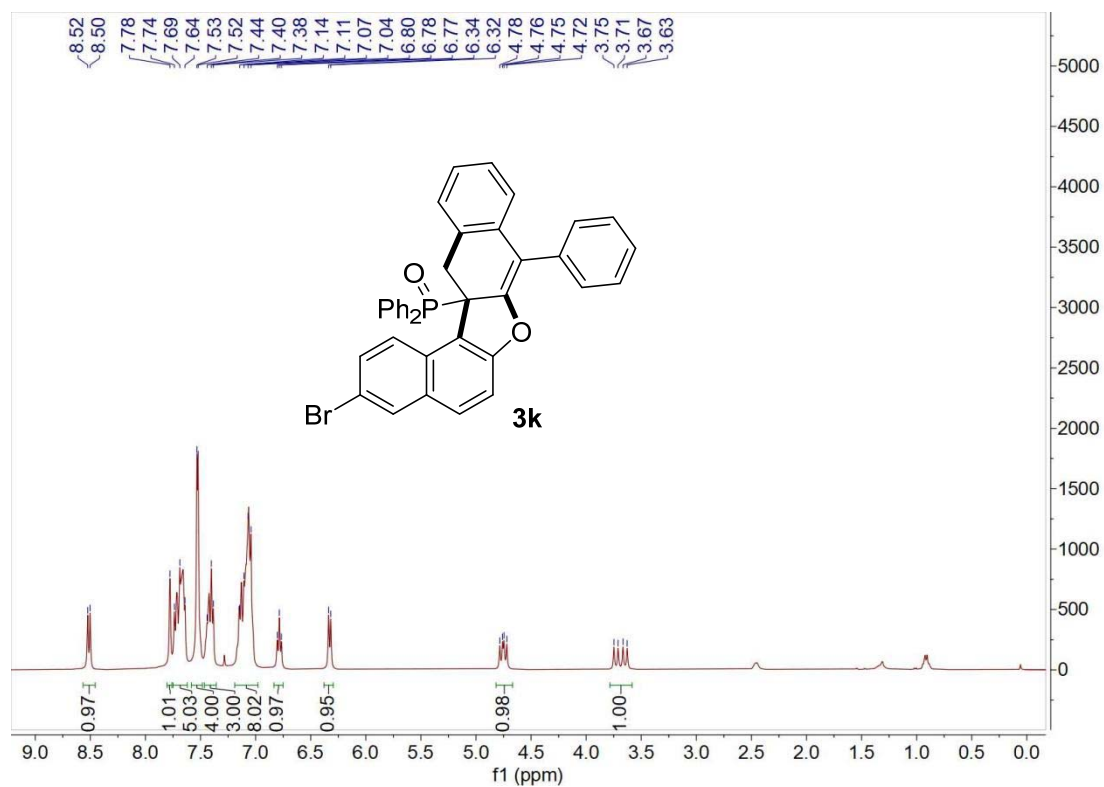


31P-NMR (202 MHz, CDCl₃) of **3j**

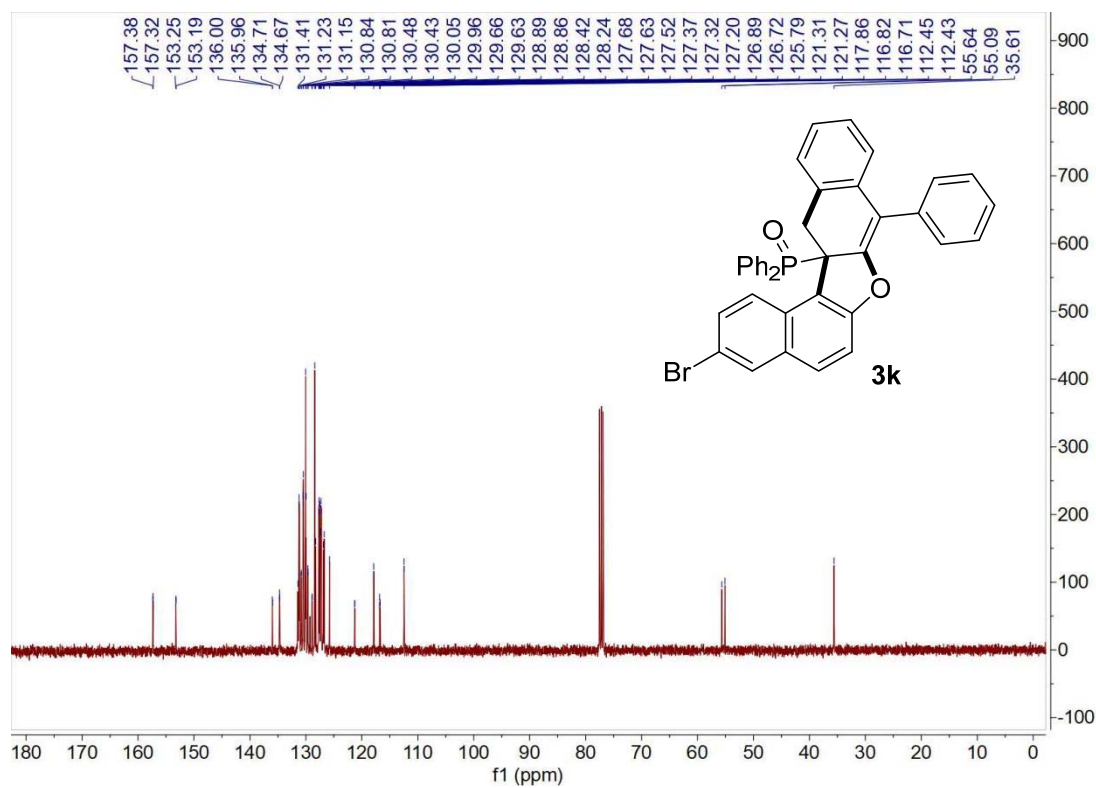
Spectrum from 06.wiff (sample 1) - Sample012, Experiment 1, +TOF MS (80 - 1000) from 0.611 to 0.655 min



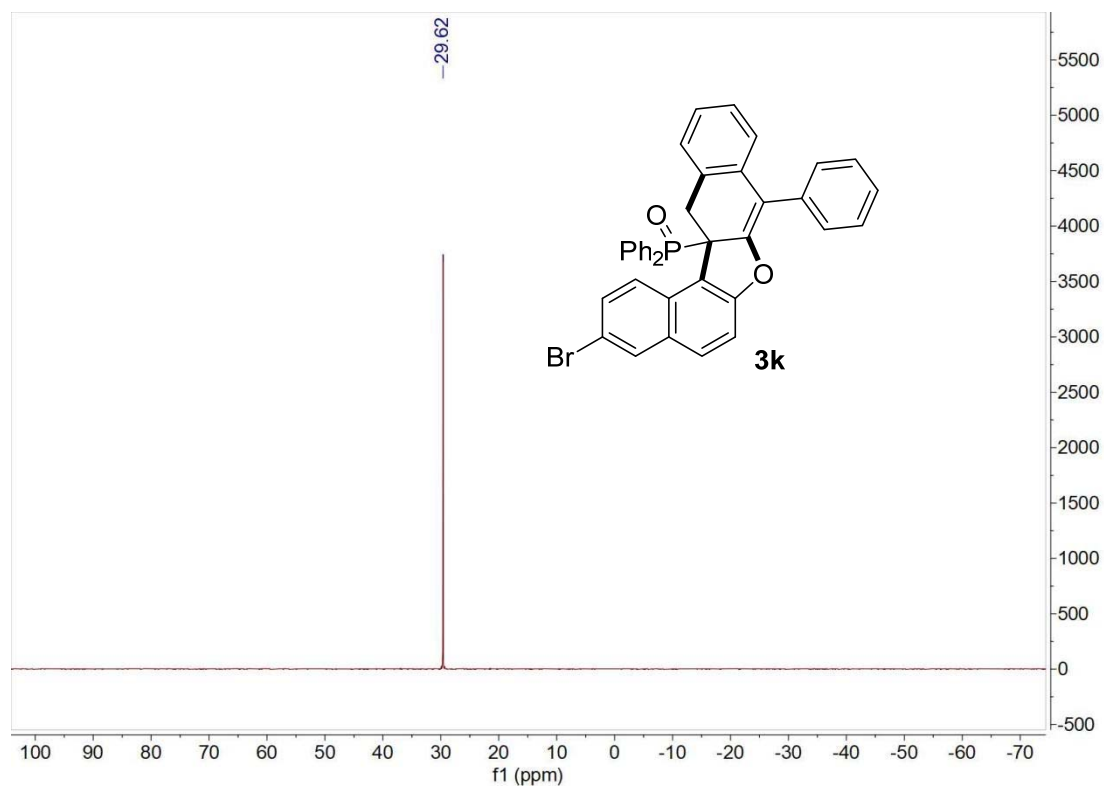
HRMS of **3j**



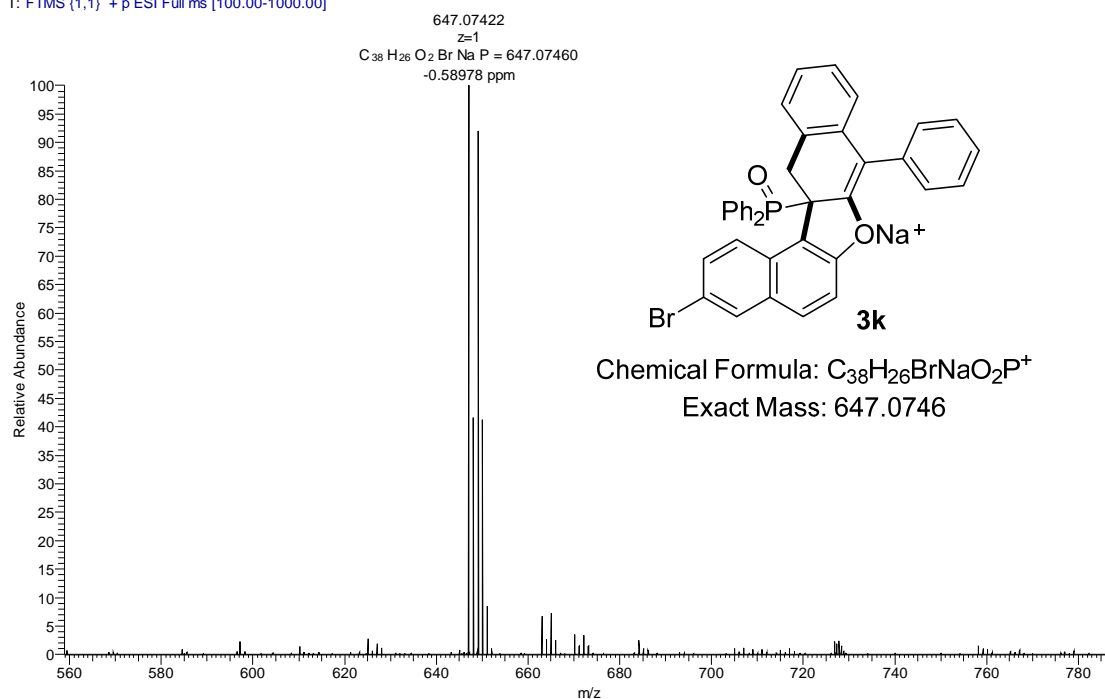
$^1\text{H-NMR}$ (400 MHz, CDCl_3) of **3k**



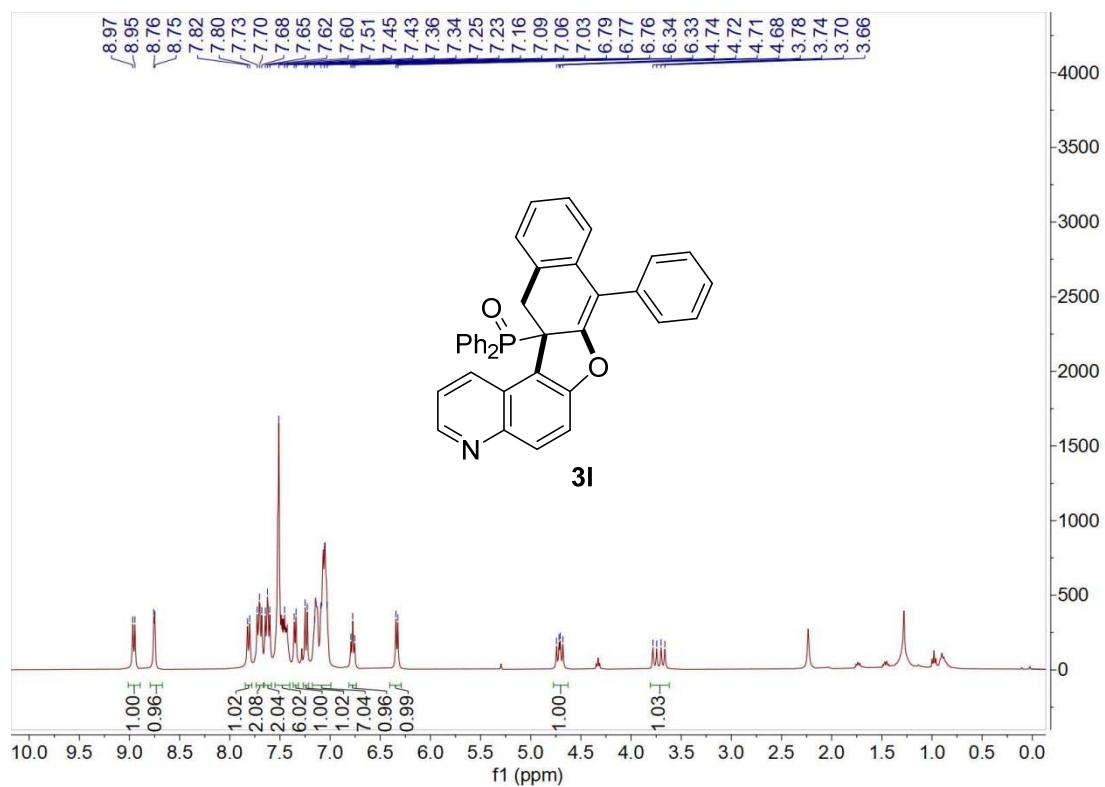
$^{13}\text{C-NMR}$ (101 MHz, CDCl_3) of **3k**



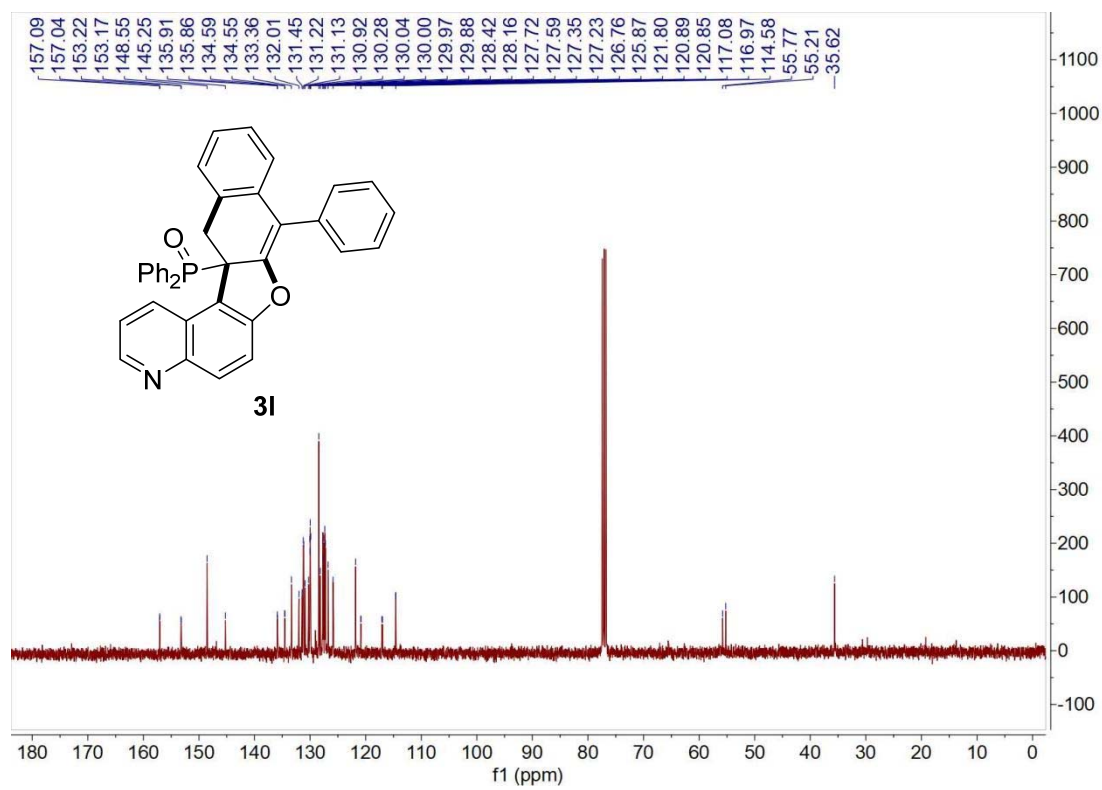
20210515-27 #21 RT: 0.34 AV: 1 NL: 6.70E4
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



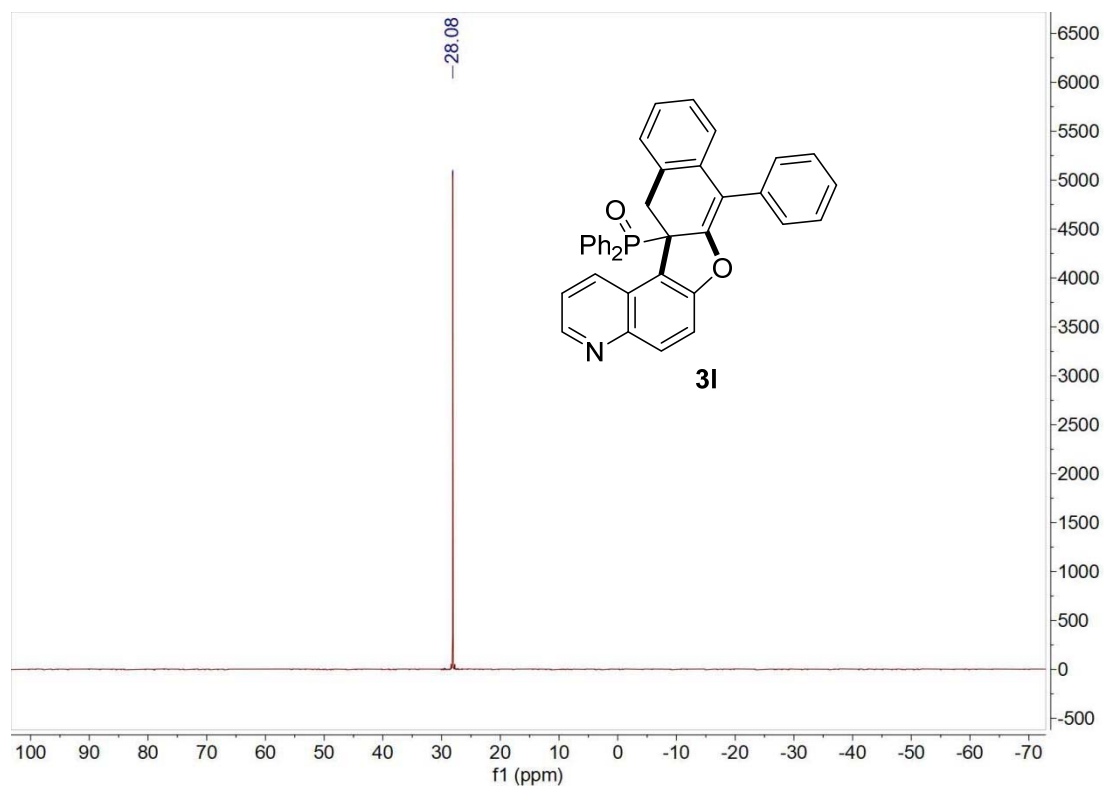
HRMS of **3k**



$^1\text{H-NMR}$ (400 MHz, CDCl_3) of **3I**

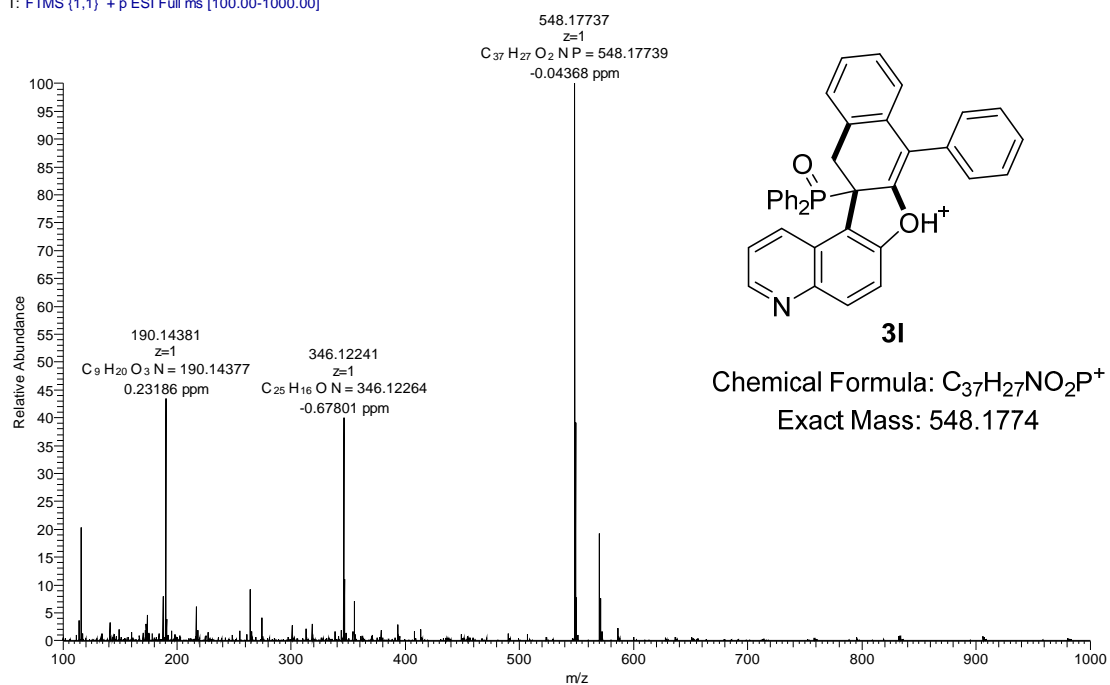


$^{13}\text{C-NMR}$ (101 MHz, CDCl_3) of **3I**

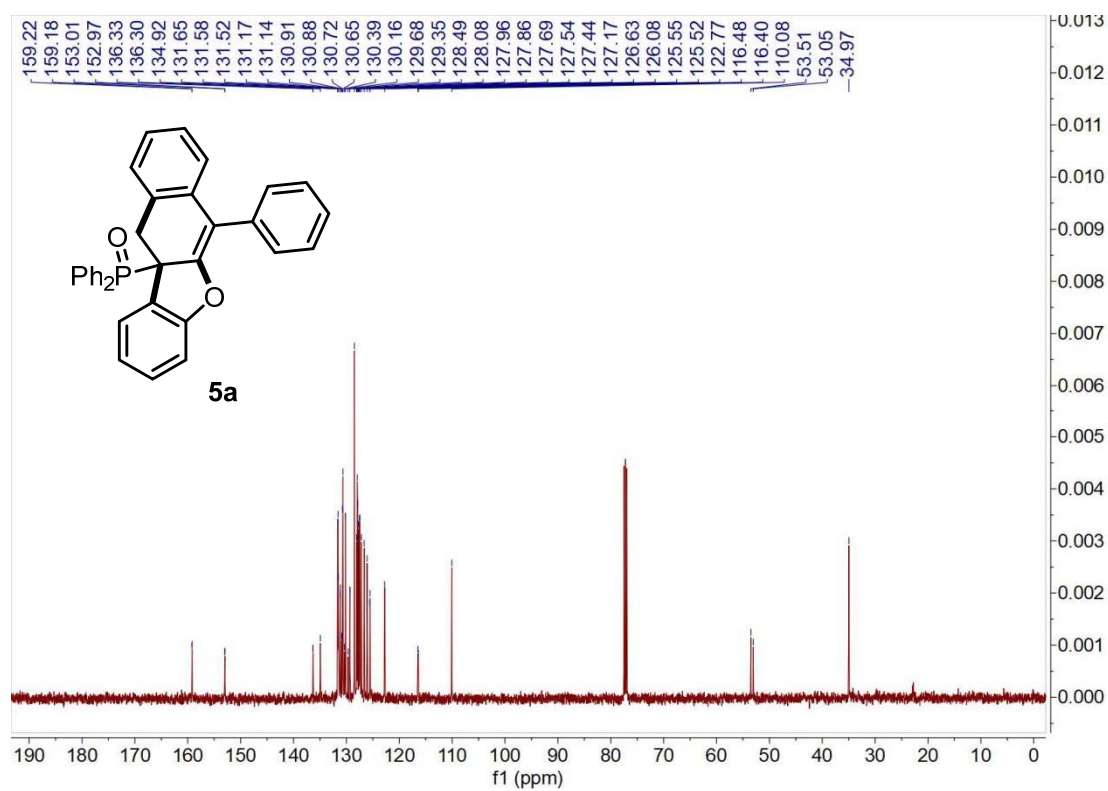
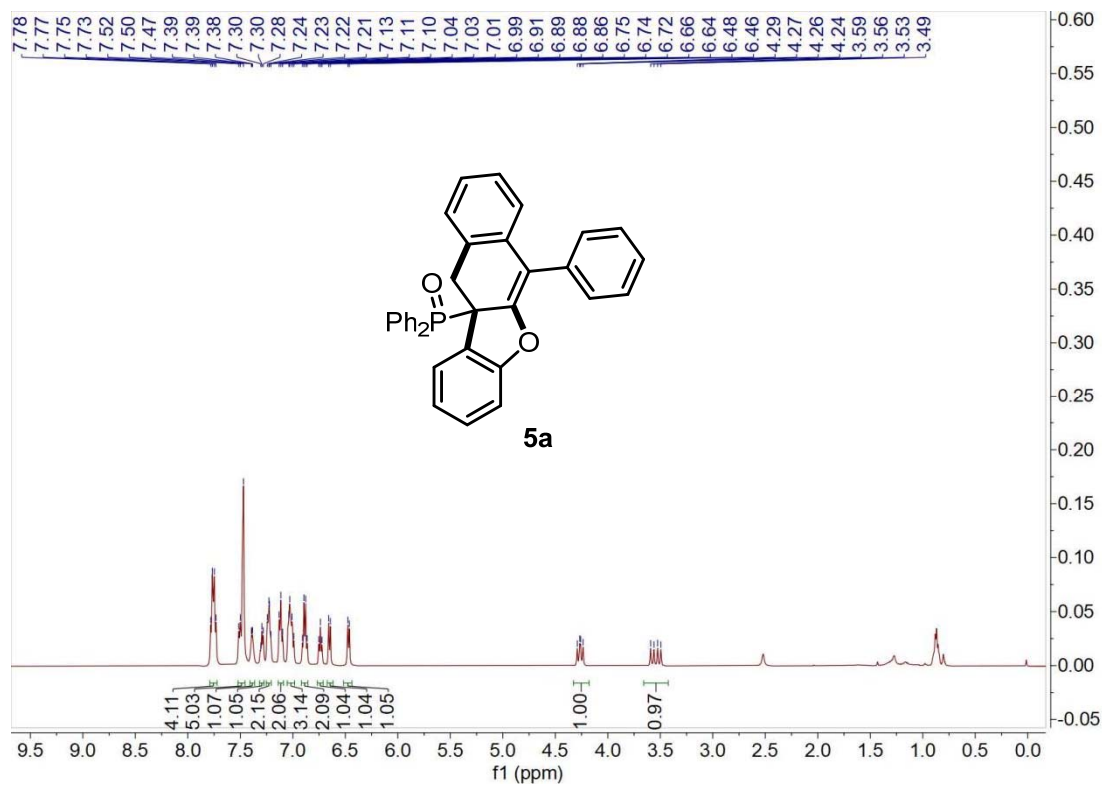


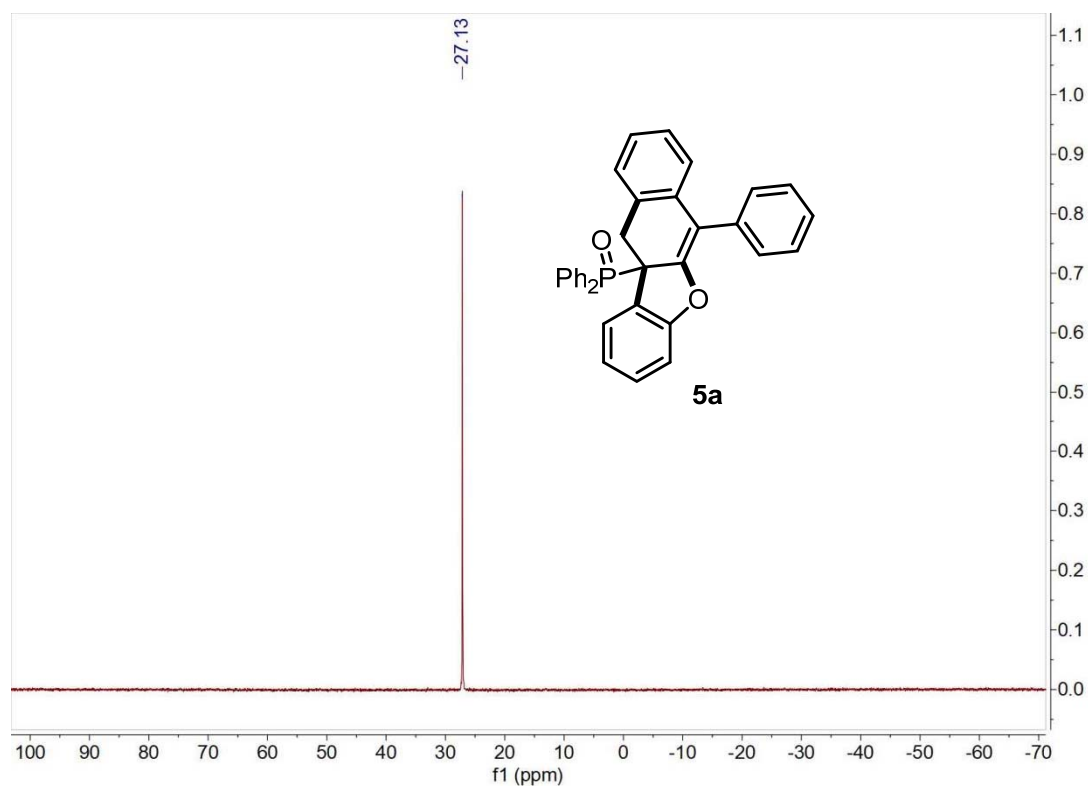
^{31}P -NMR (162 MHz, CDCl_3) of **3I**

20210624-12 #35 RT: 0.45 AV: 1 NL: 1.95E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



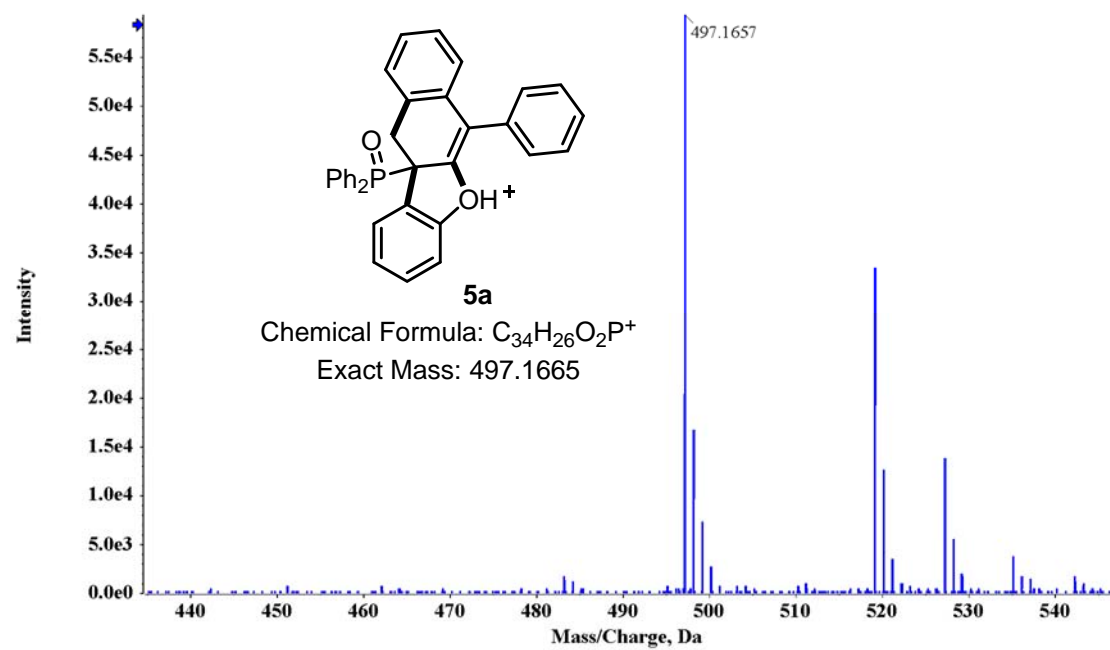
HRMS of **3I**



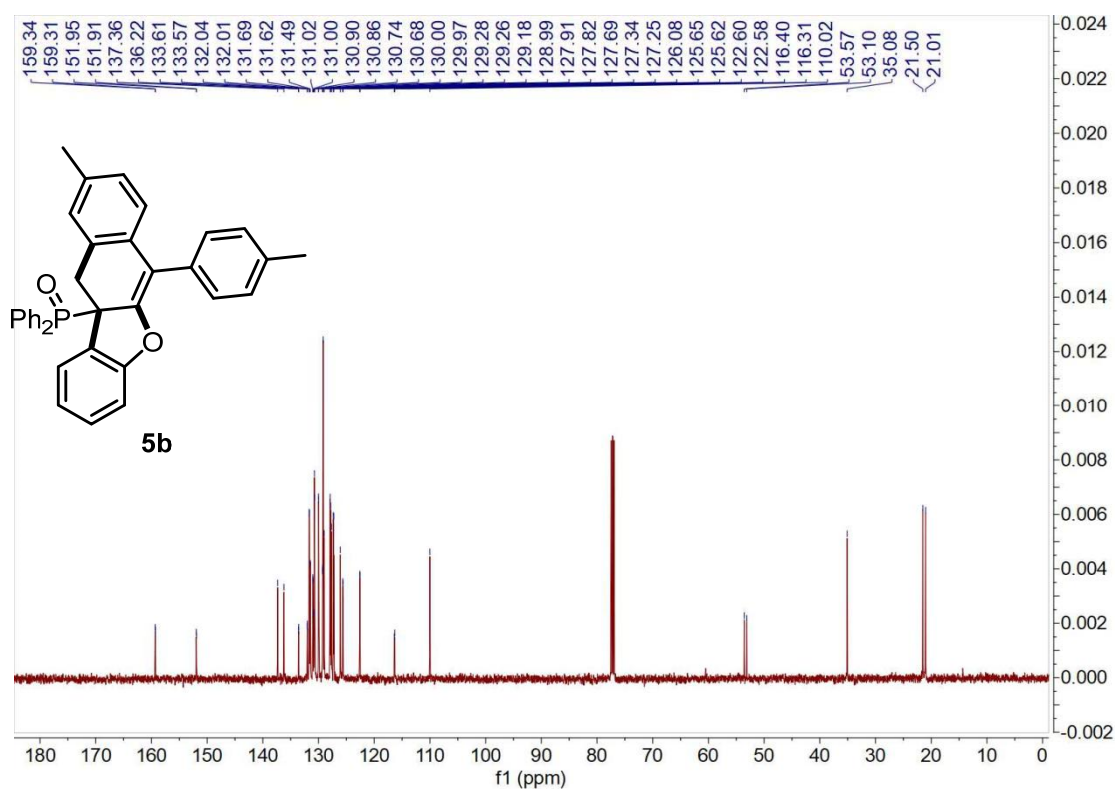
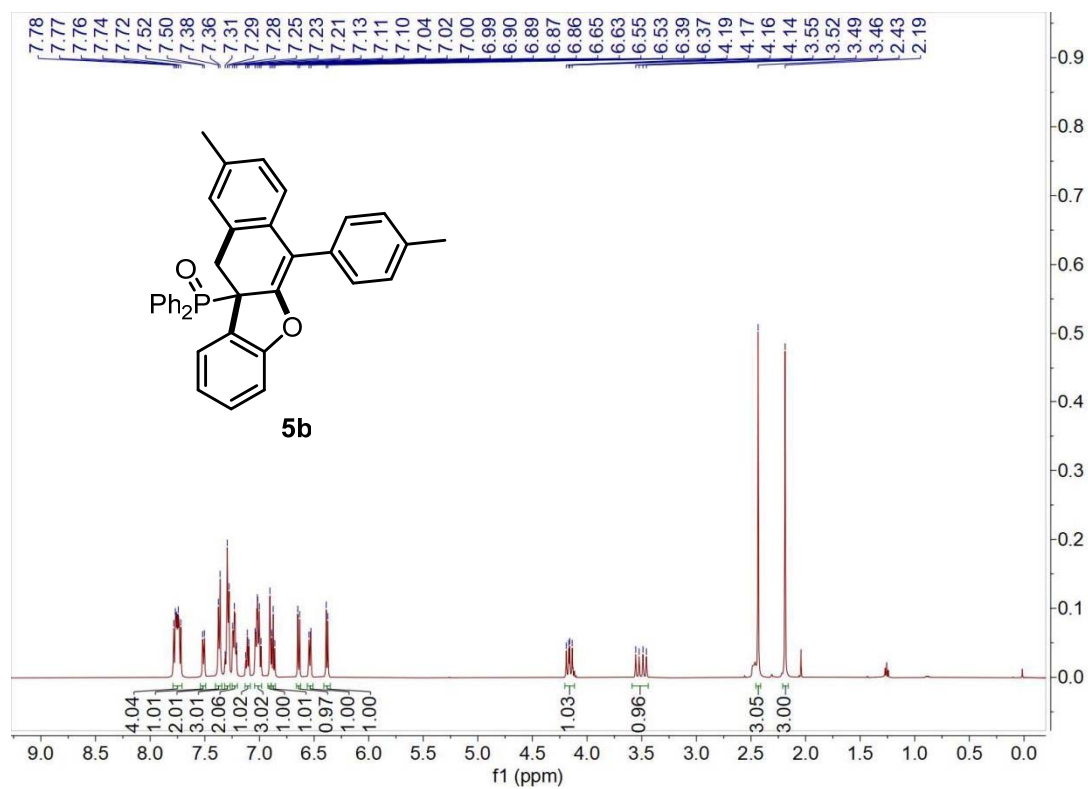


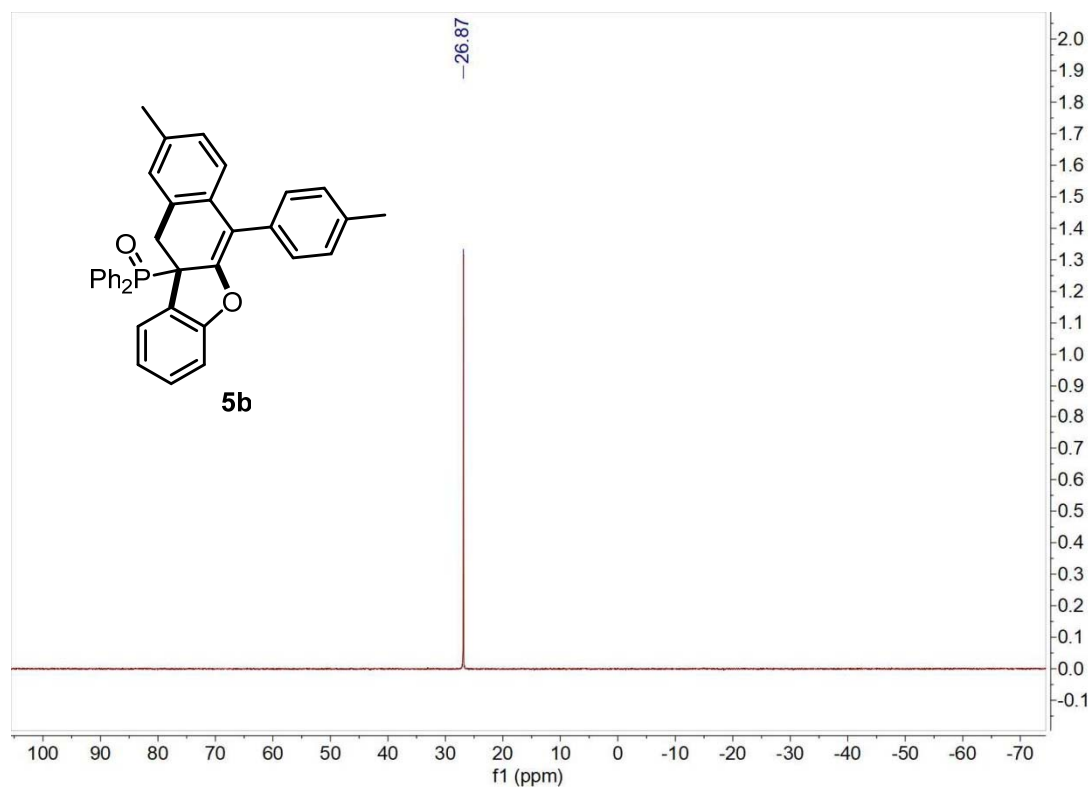
^{31}P NMR (202 MHz, CDCl_3) of **5a**

Spectrum from 16.wiff (sample 1) - Sample016, Experiment 1, +TOF MS (80 - 1000) from 0.651 min

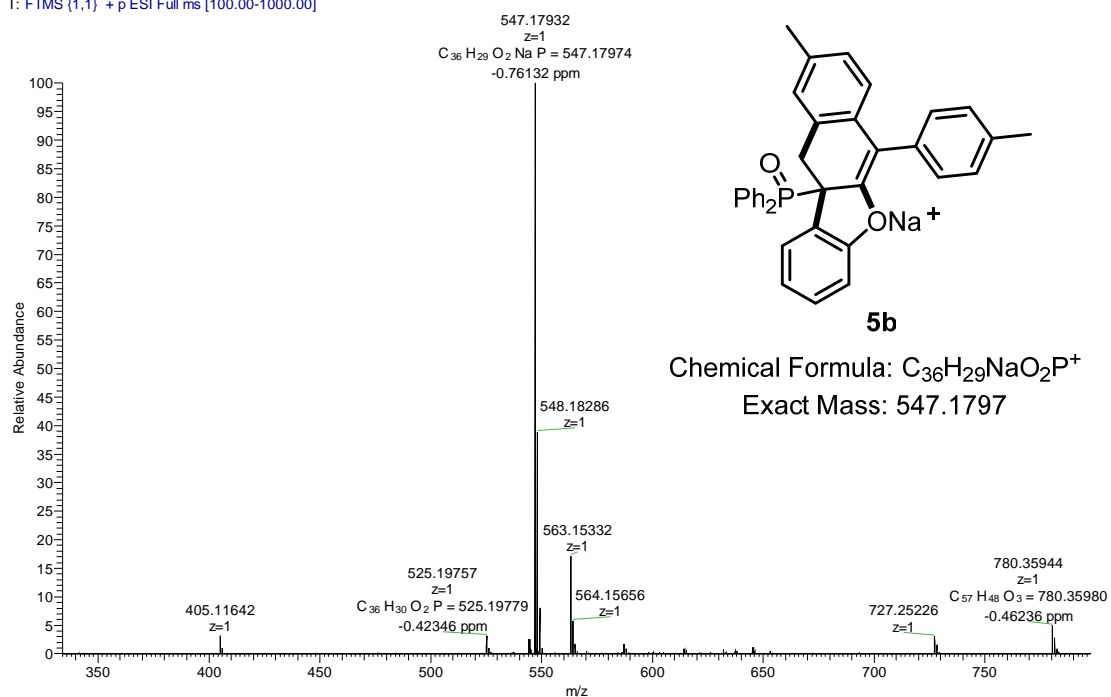


HRMS of **5a**

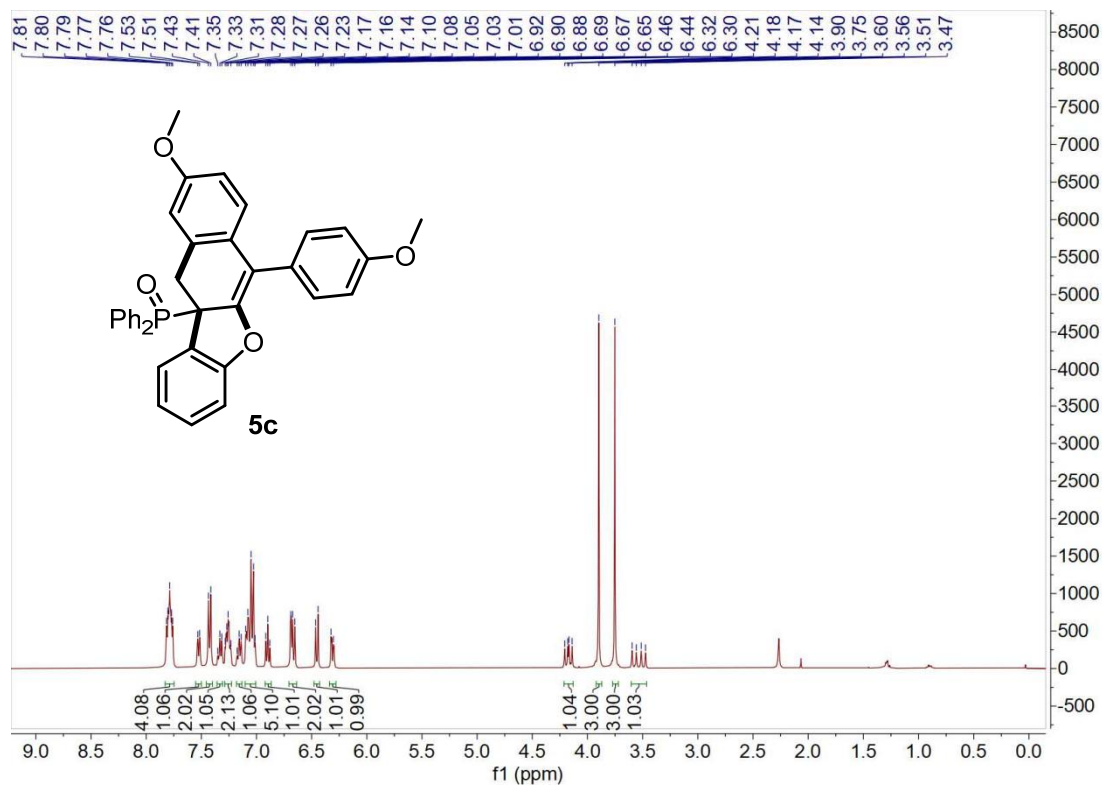




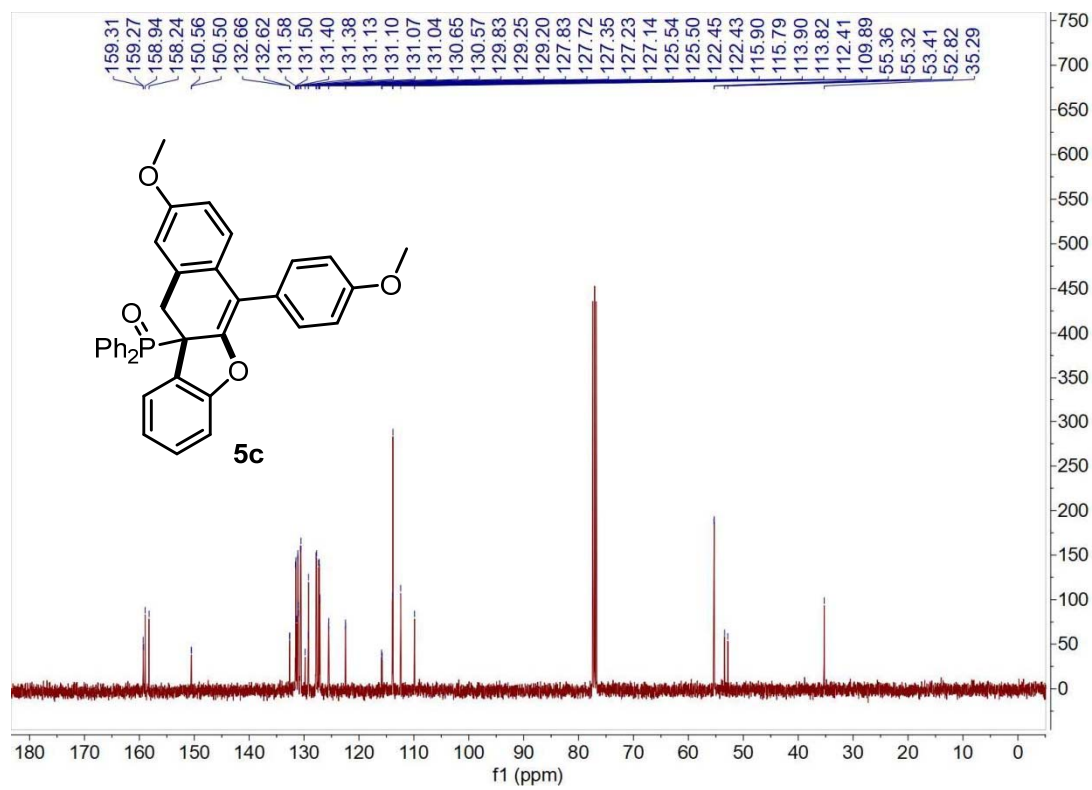
25 #27 RT: 0.32 AV: 1 NL: 1.75E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



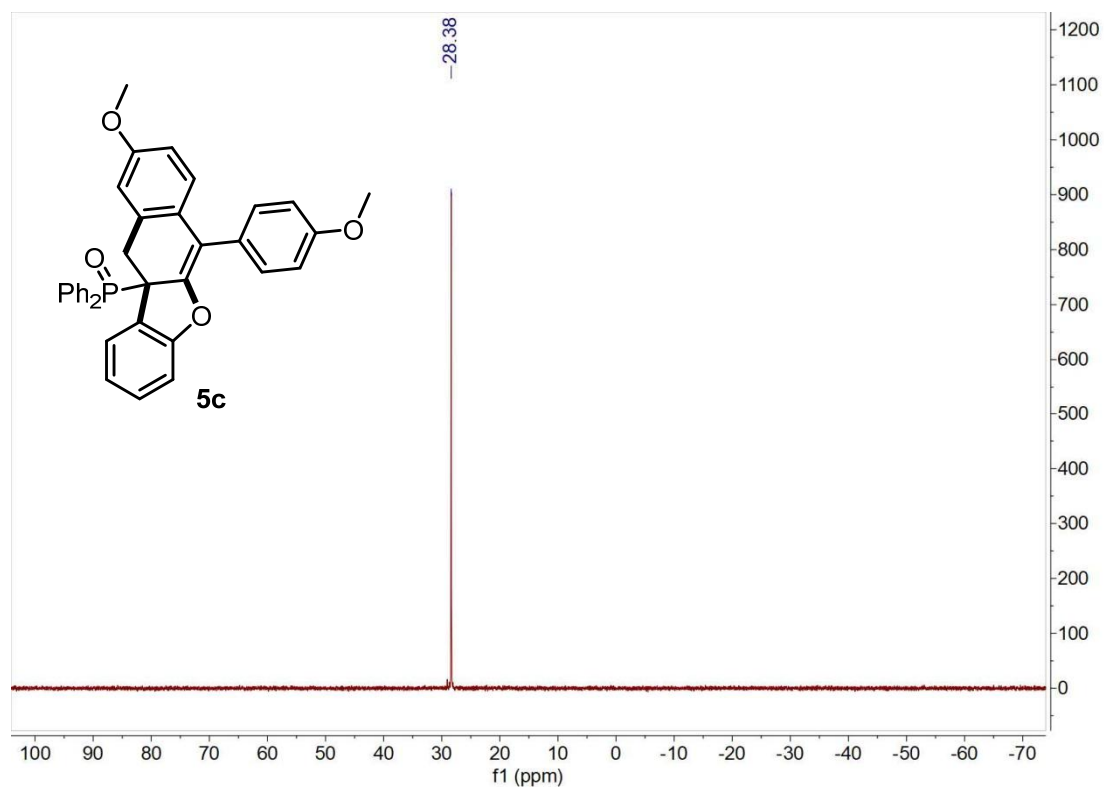
HRMS of **5b**



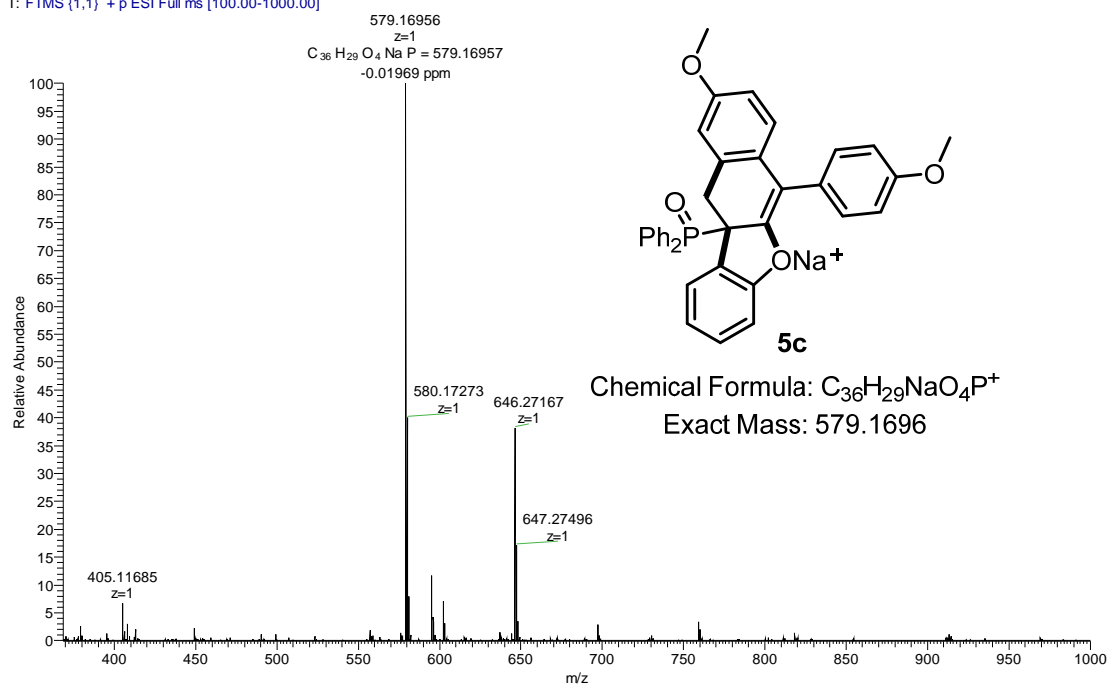
$^1\text{H-NMR}$ (400 MHz, CDCl_3) of **5c**



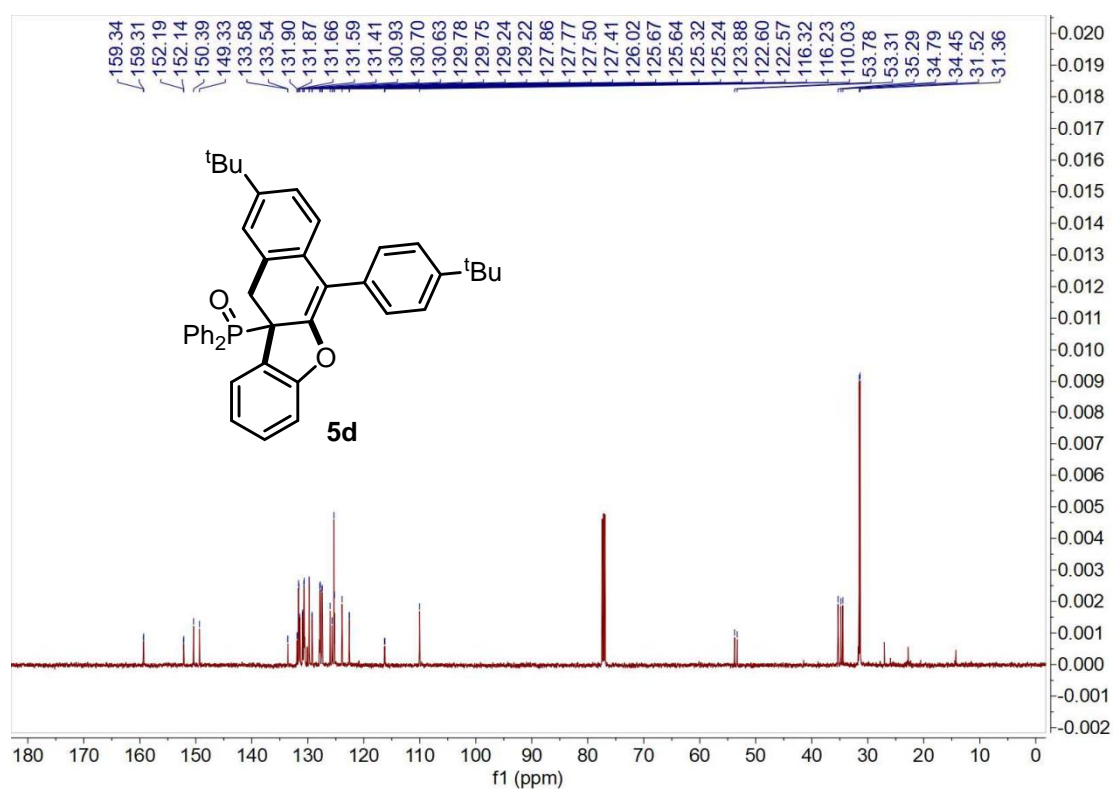
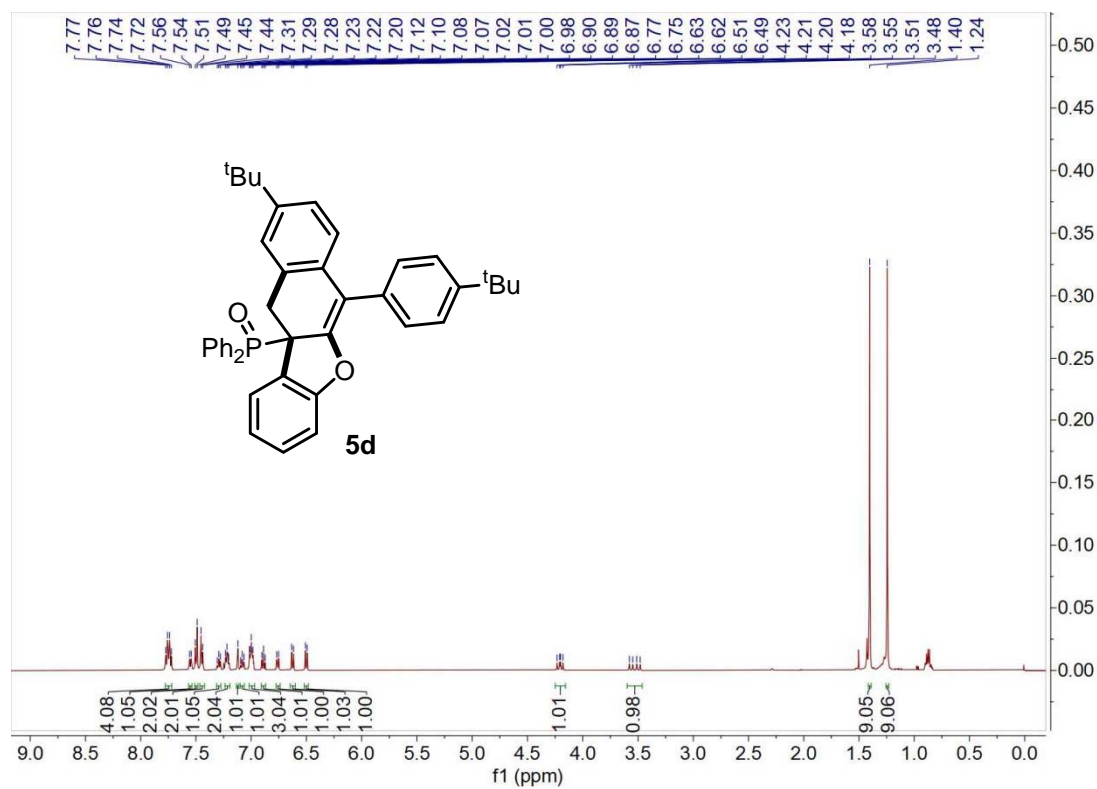
$^{13}\text{C-NMR}$ (101 MHz, CDCl_3) of **5c**

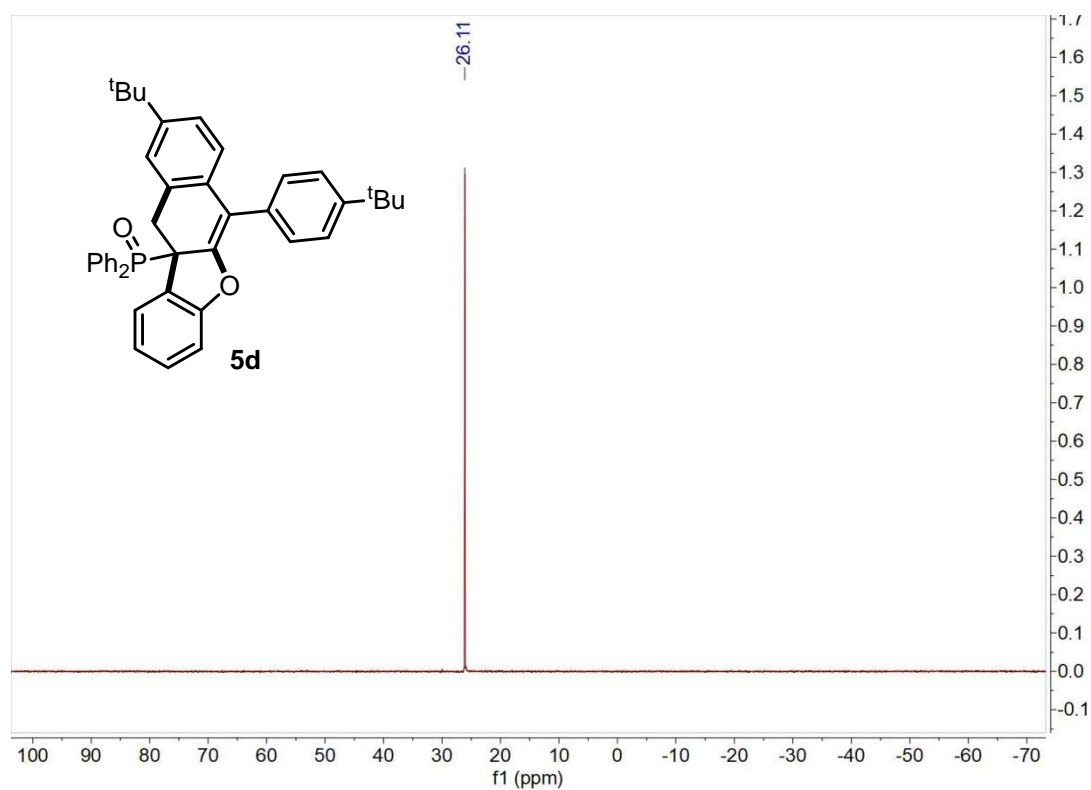


20210624-15 #33 RT: 0.42 AV: 1 NL: 1.22E5
 T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



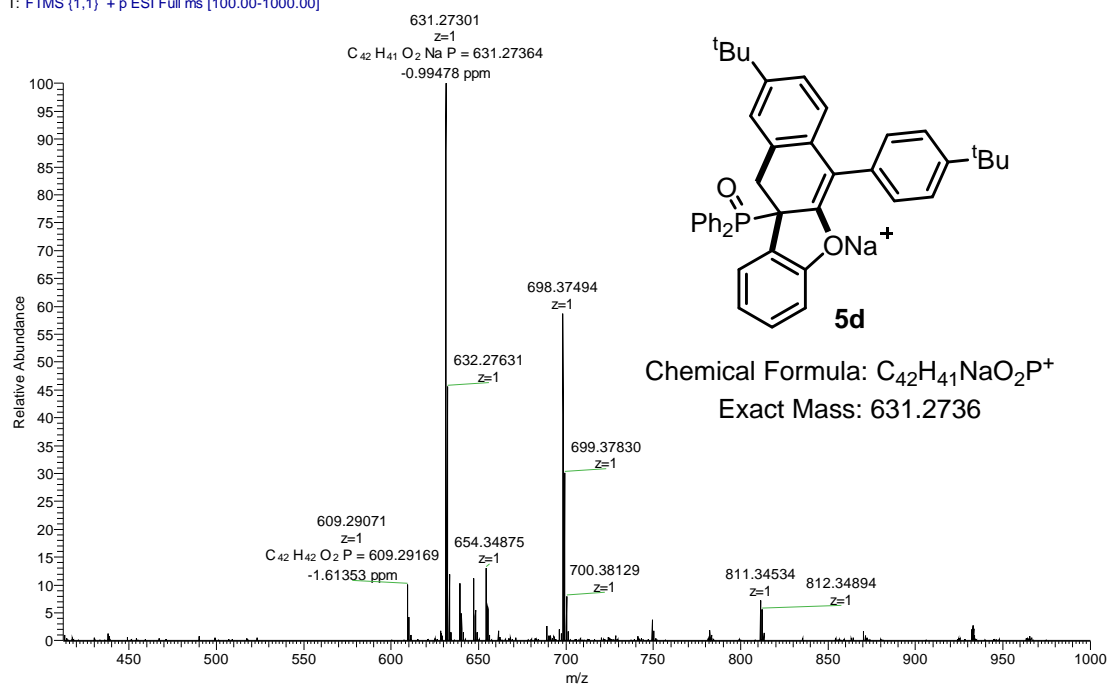
HRMS of 5c



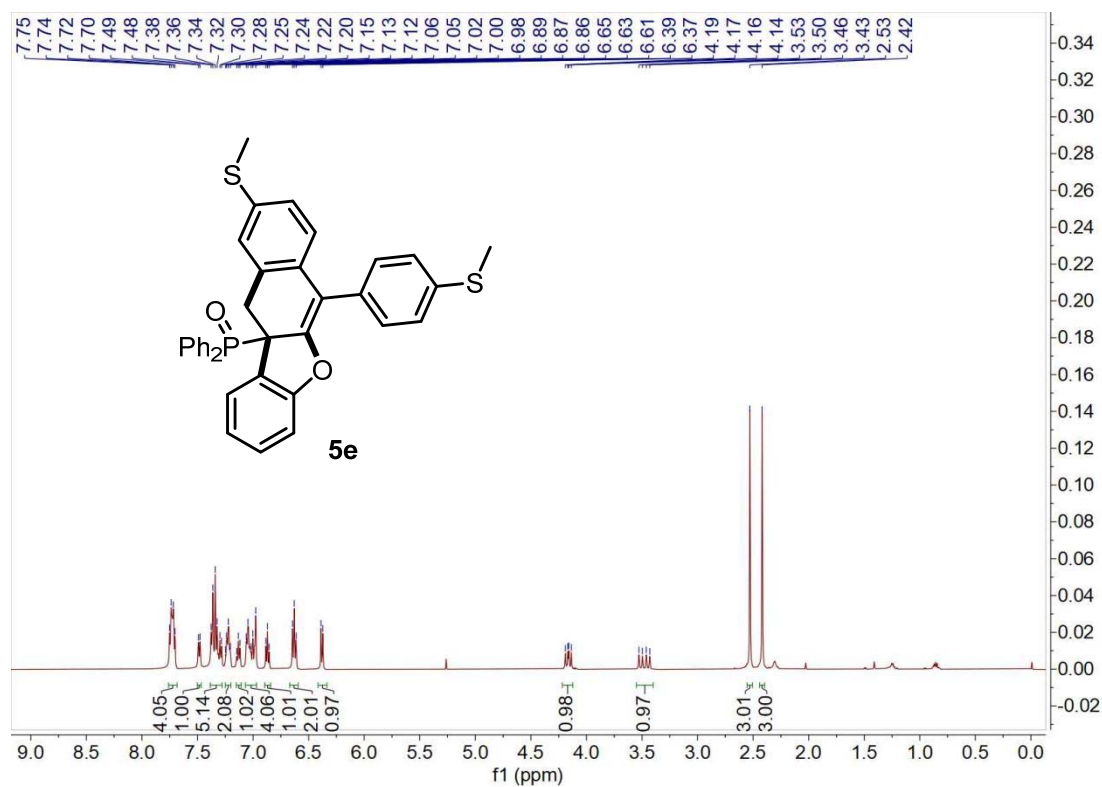


^{31}P -NMR (202 MHz, CDCl_3) of **5d**

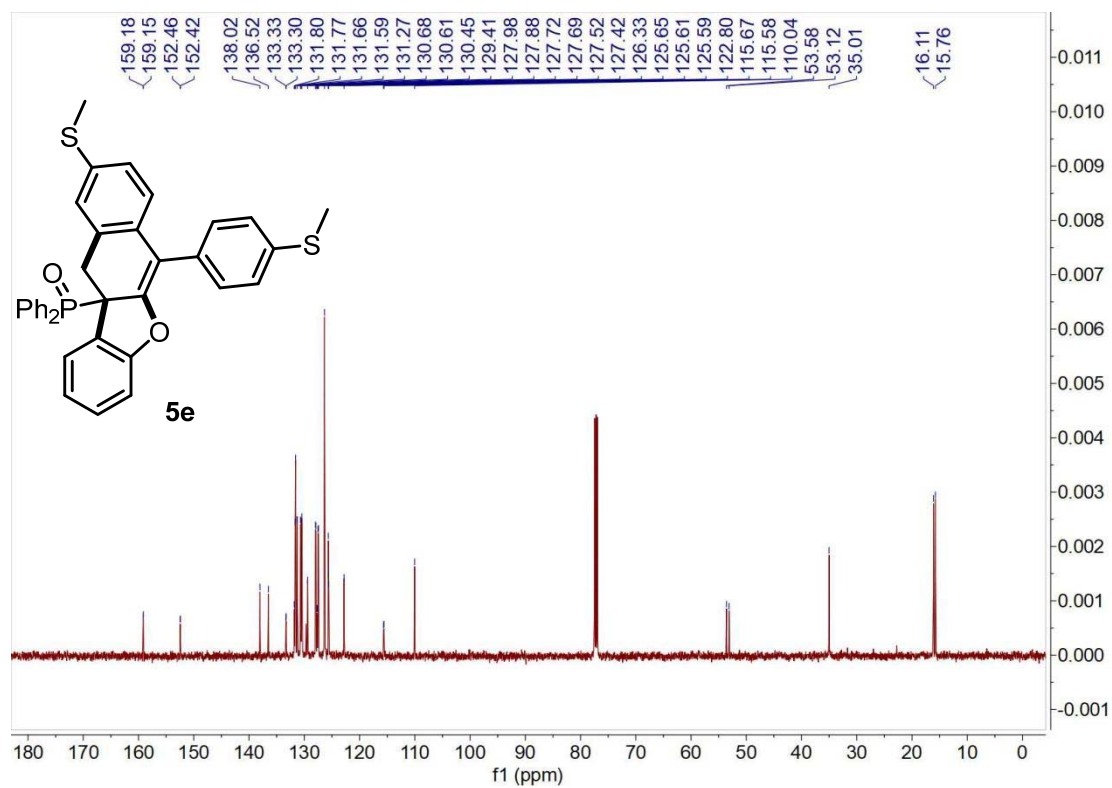
20210624-16 #49 RT: 0.65 AV: 1 NL: 1.50E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



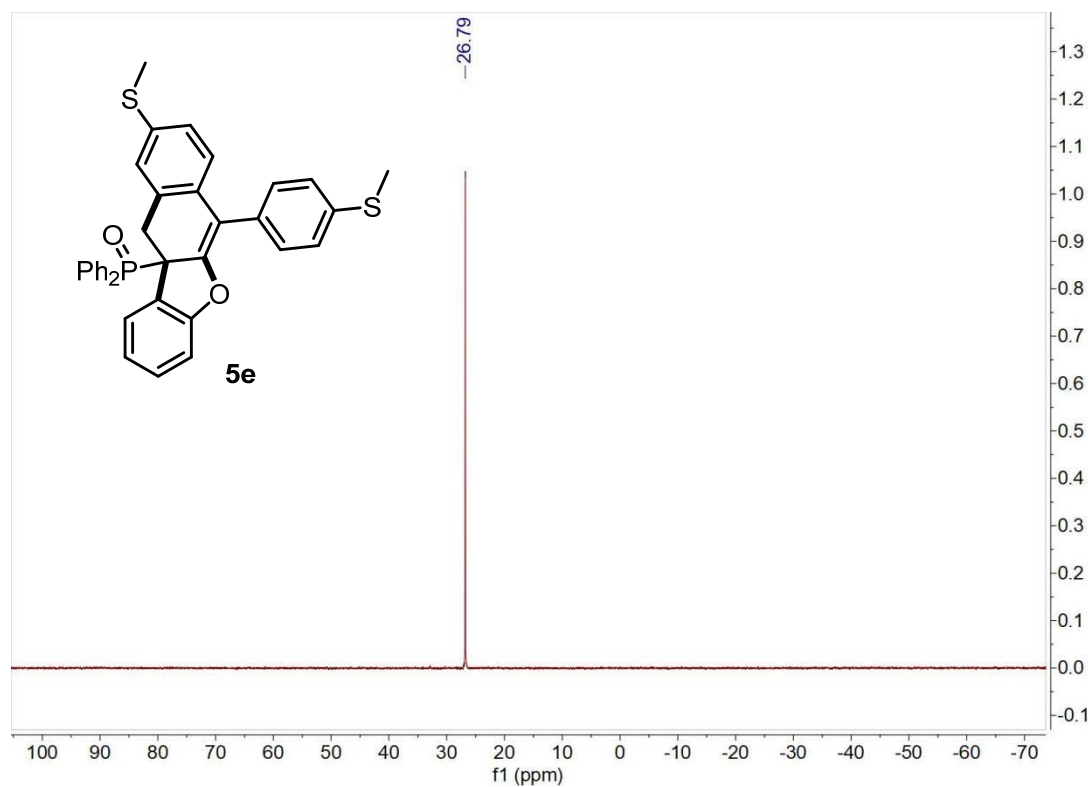
HRMS of **5d**



¹H-NMR (500 MHz, CDCl₃) of **5e**

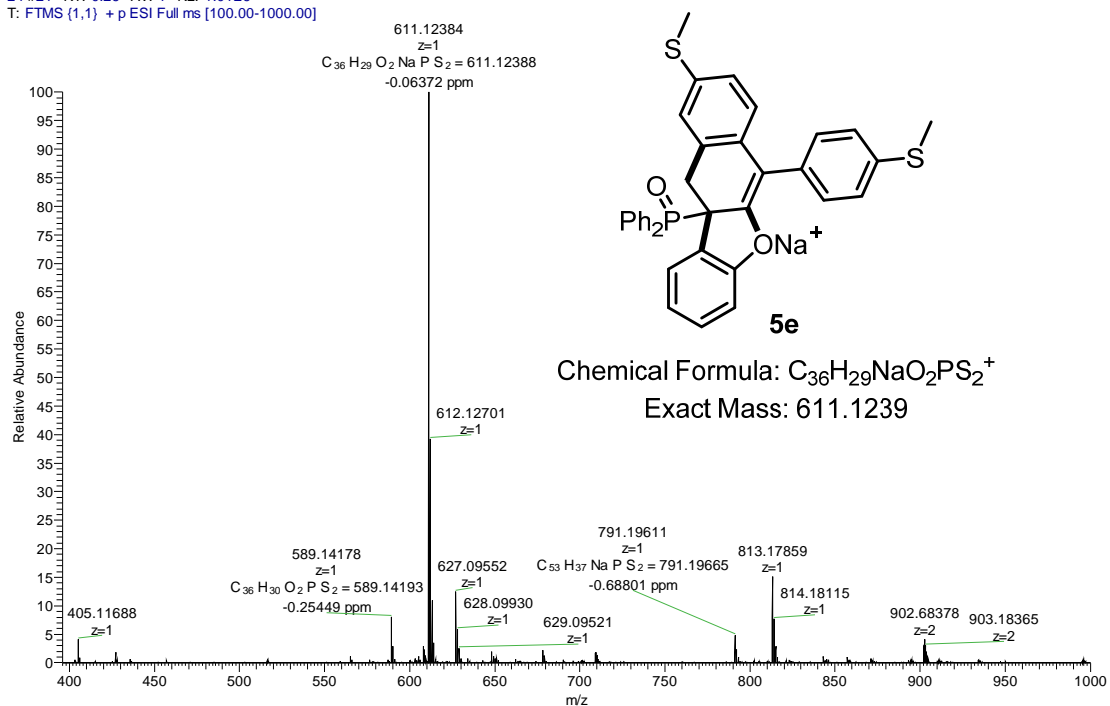


¹³C-NMR (126 MHz, CDCl₃) of **5e**

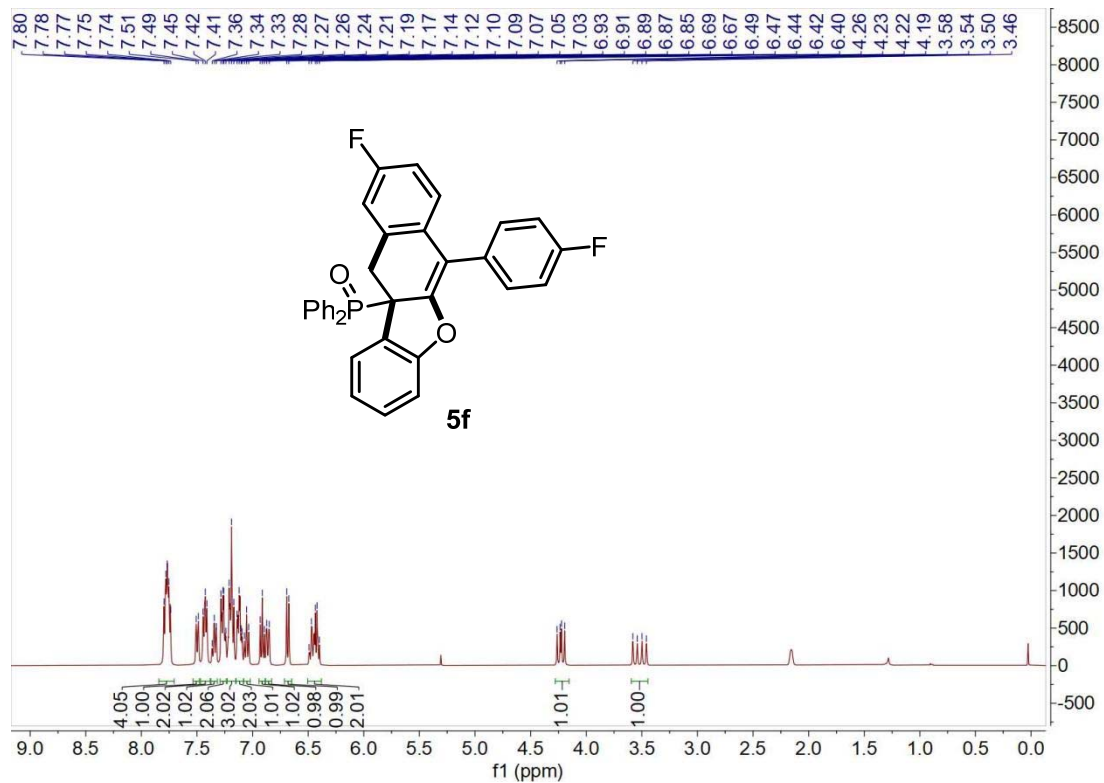


^{31}P -NMR (202 MHz, CDCl_3) of **5e**

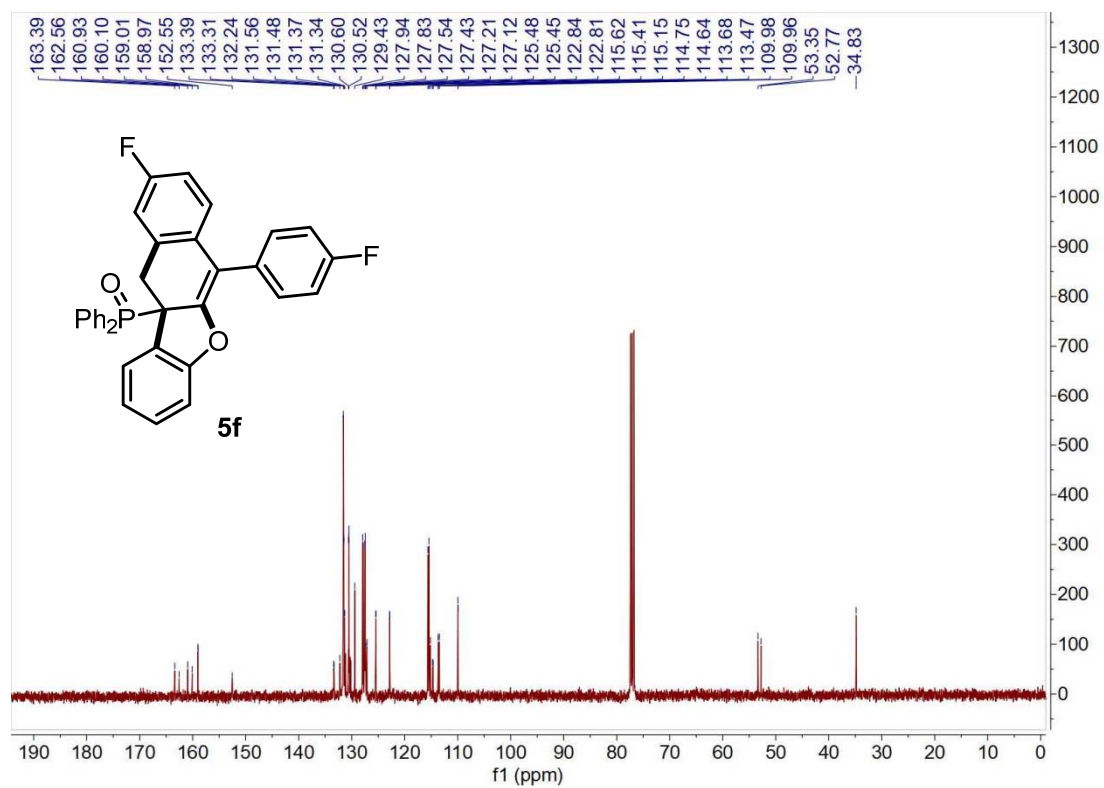
24 #21 RT: 0.26 AV: 1 NL: 1.01E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



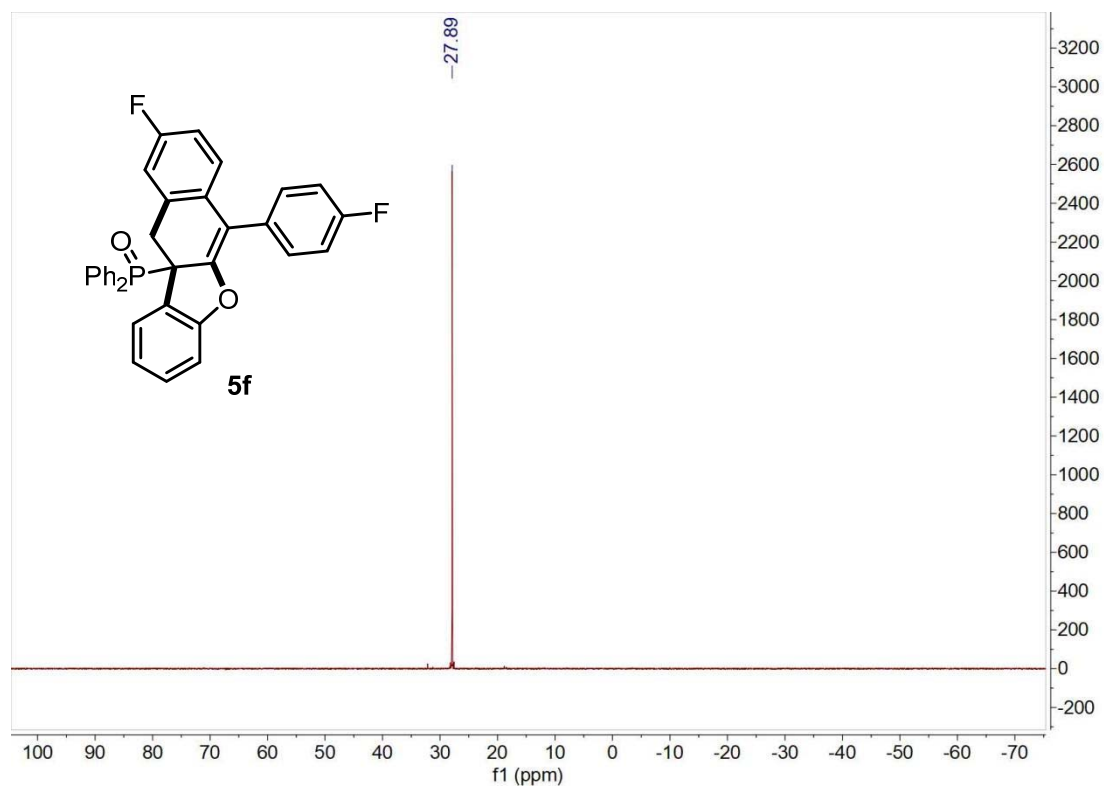
HRMS of **5e**



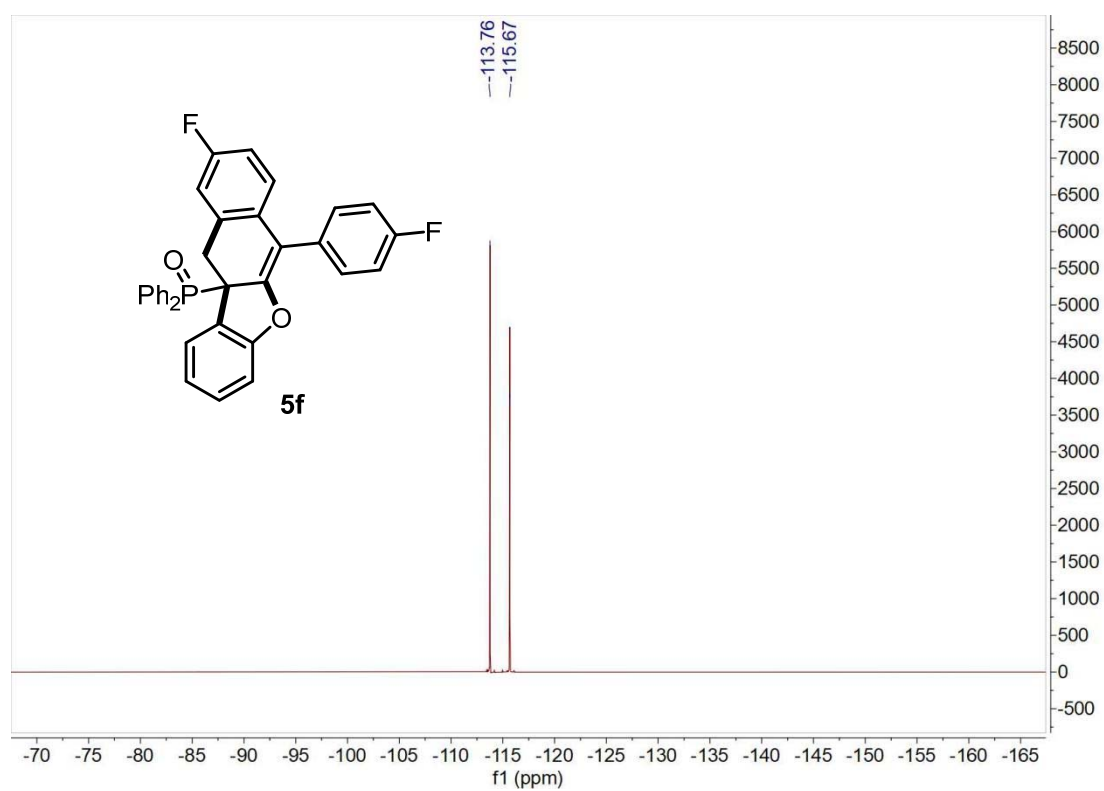
¹H-NMR (400 MHz, CDCl₃) of **5f**



¹³C-NMR (101 MHz, CDCl₃) of **5f**

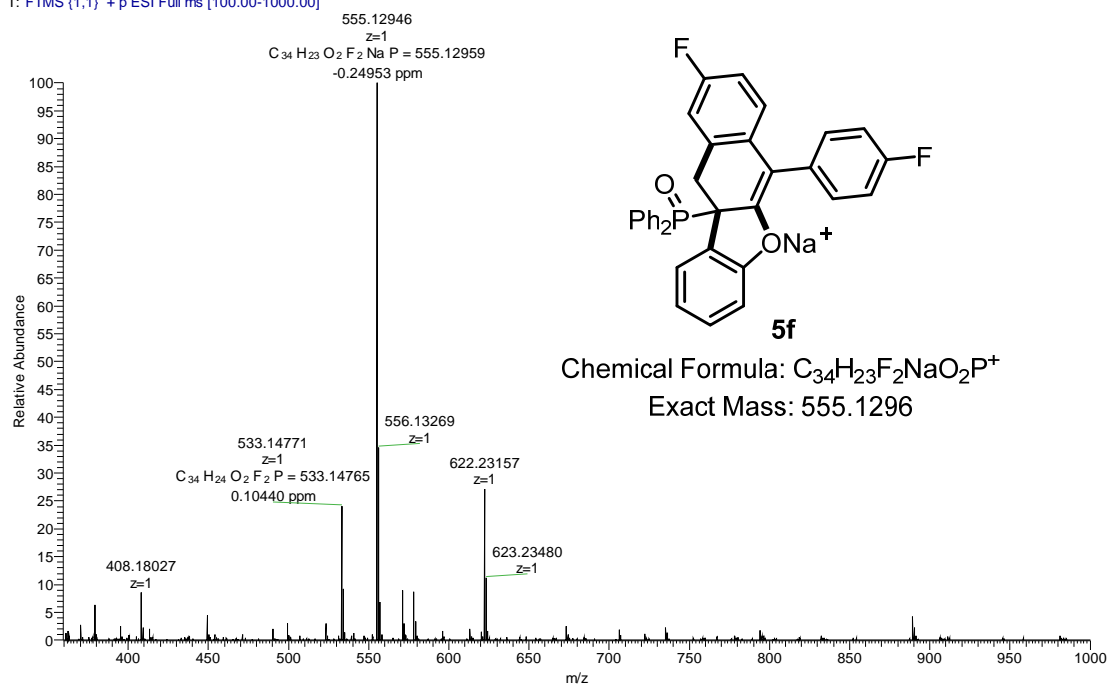


^{31}P -NMR (162 MHz, CDCl_3) of **5f**

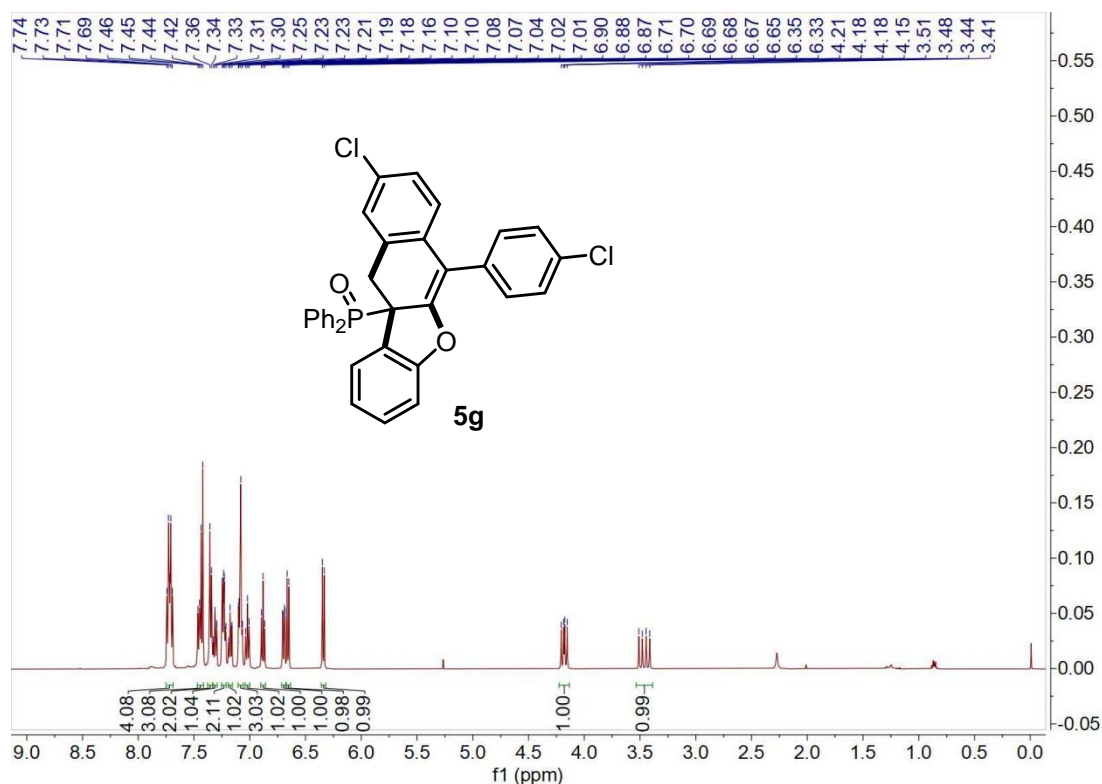


^{19}F -NMR (376 MHz, CDCl_3) of **5f**

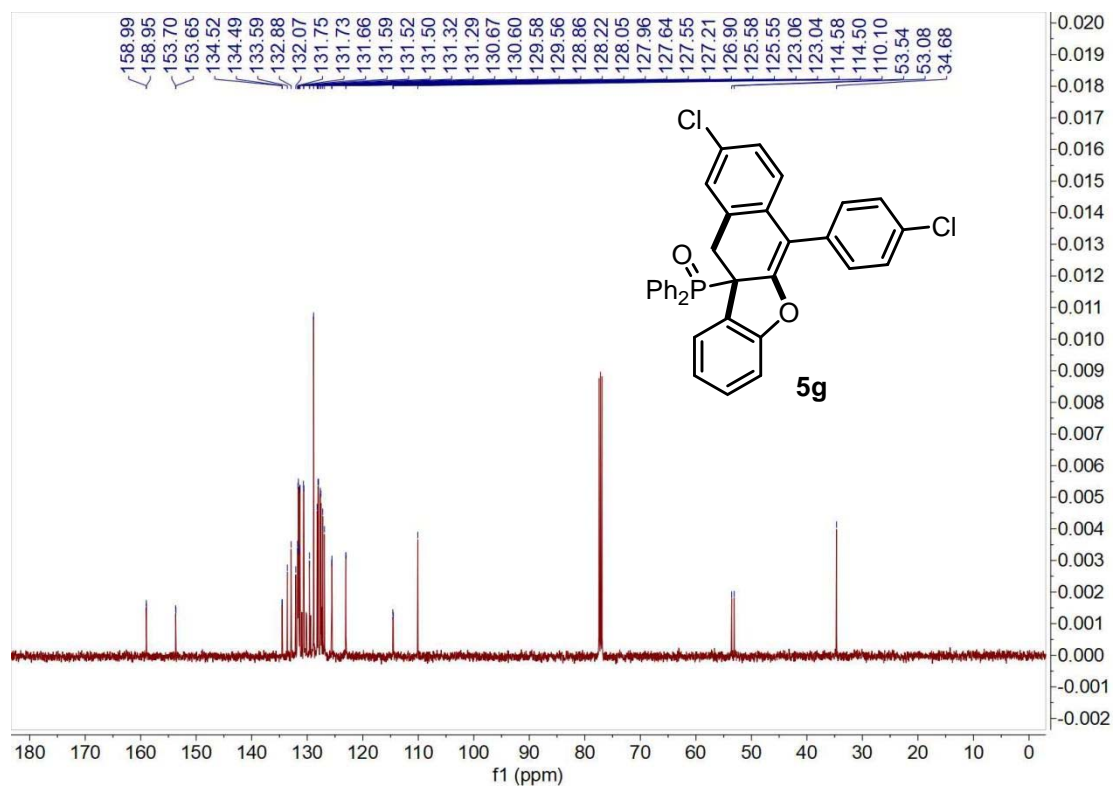
20210624-13 #35 RT: 0.46 AV: 1 NL: 1.03E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



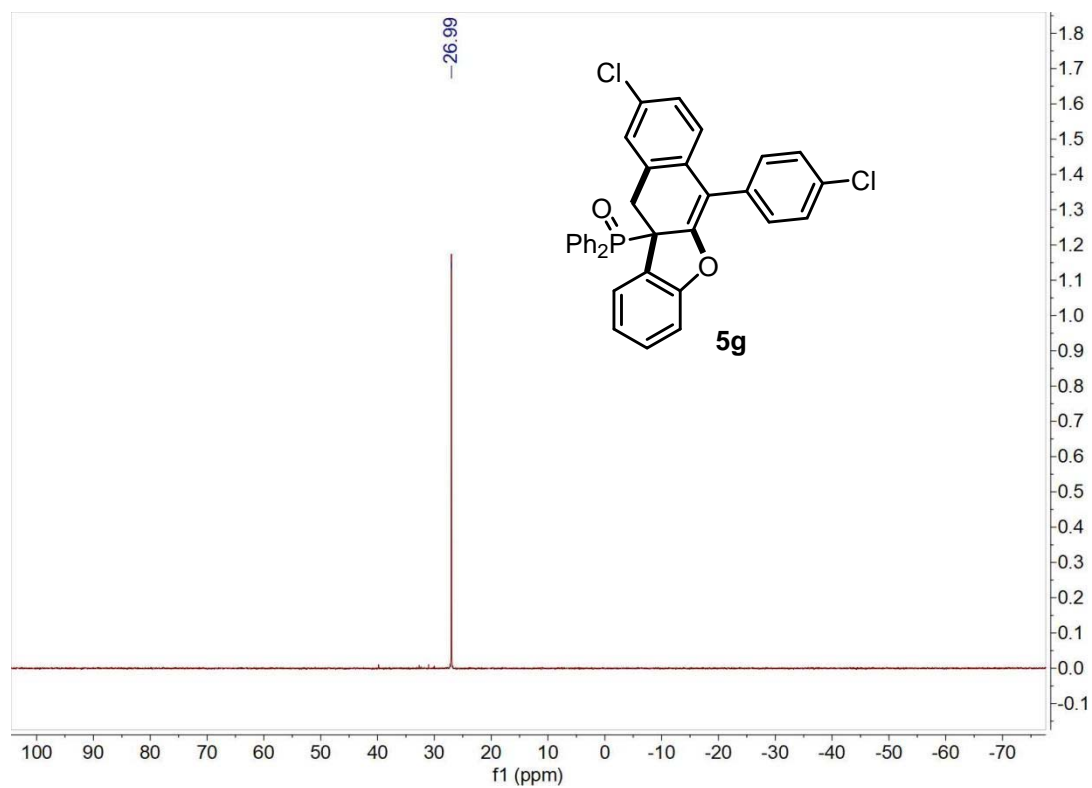
HRMS of **5f**



¹H-NMR (500 MHz, CDCl₃) of **5g**

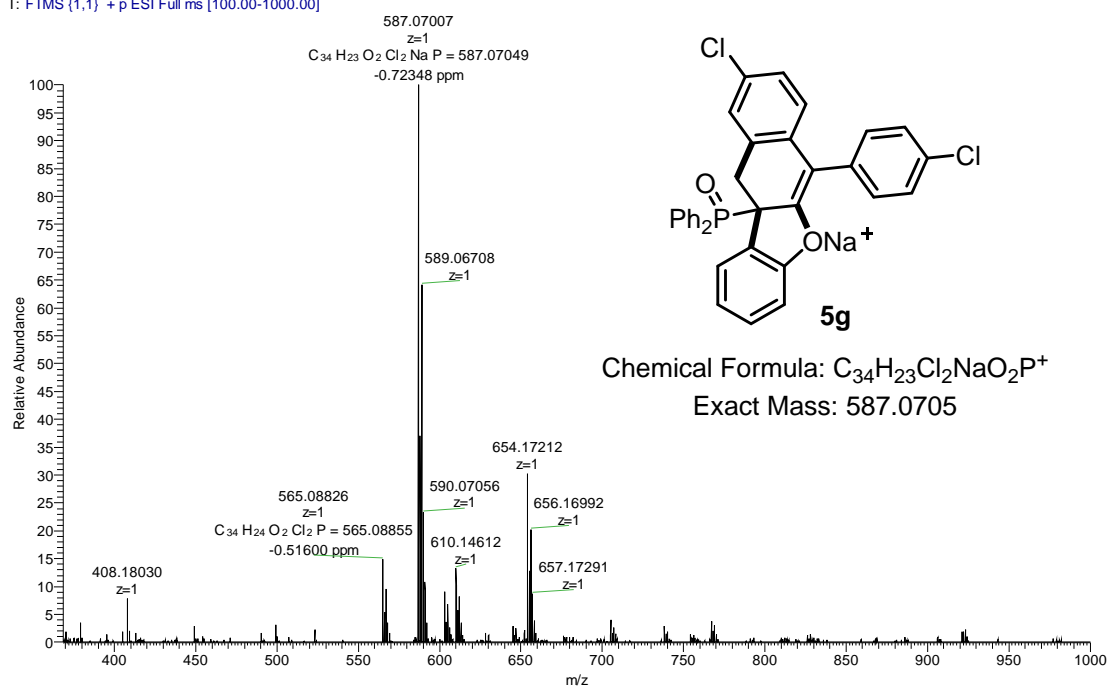


^{13}C -NMR (126 MHz, CDCl_3) of **5g**

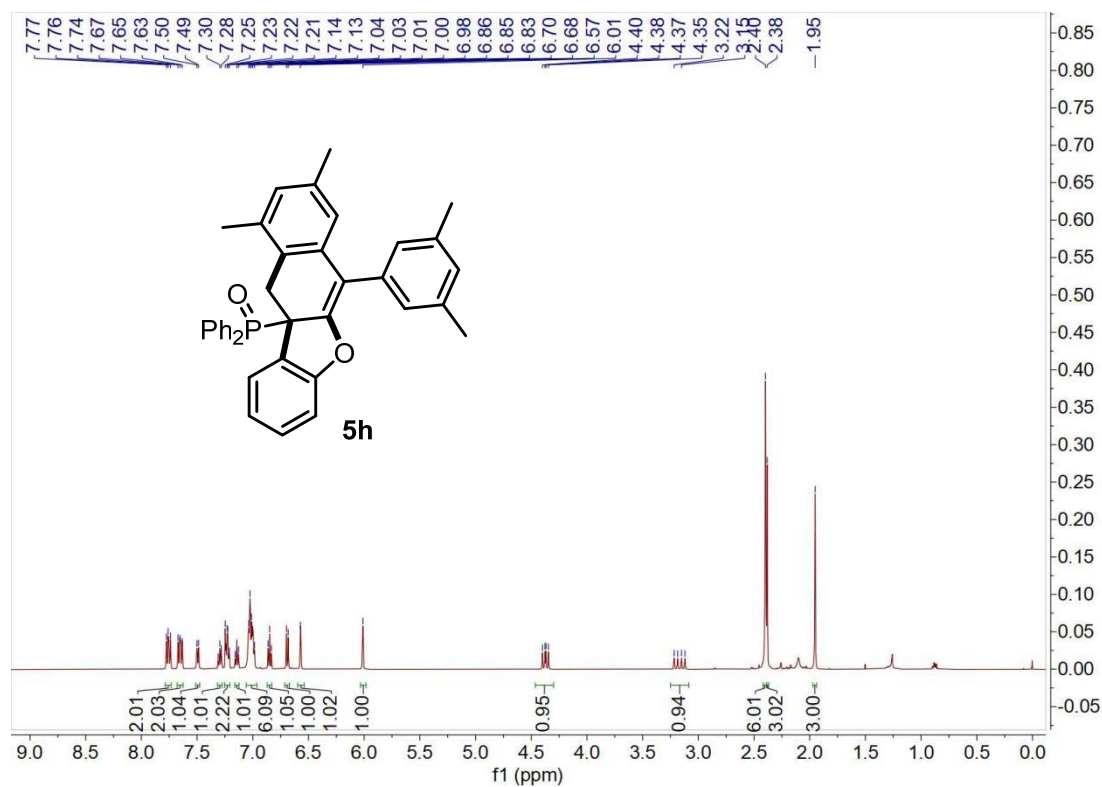


^{31}P -NMR (202 MHz, CDCl_3) of **5g**

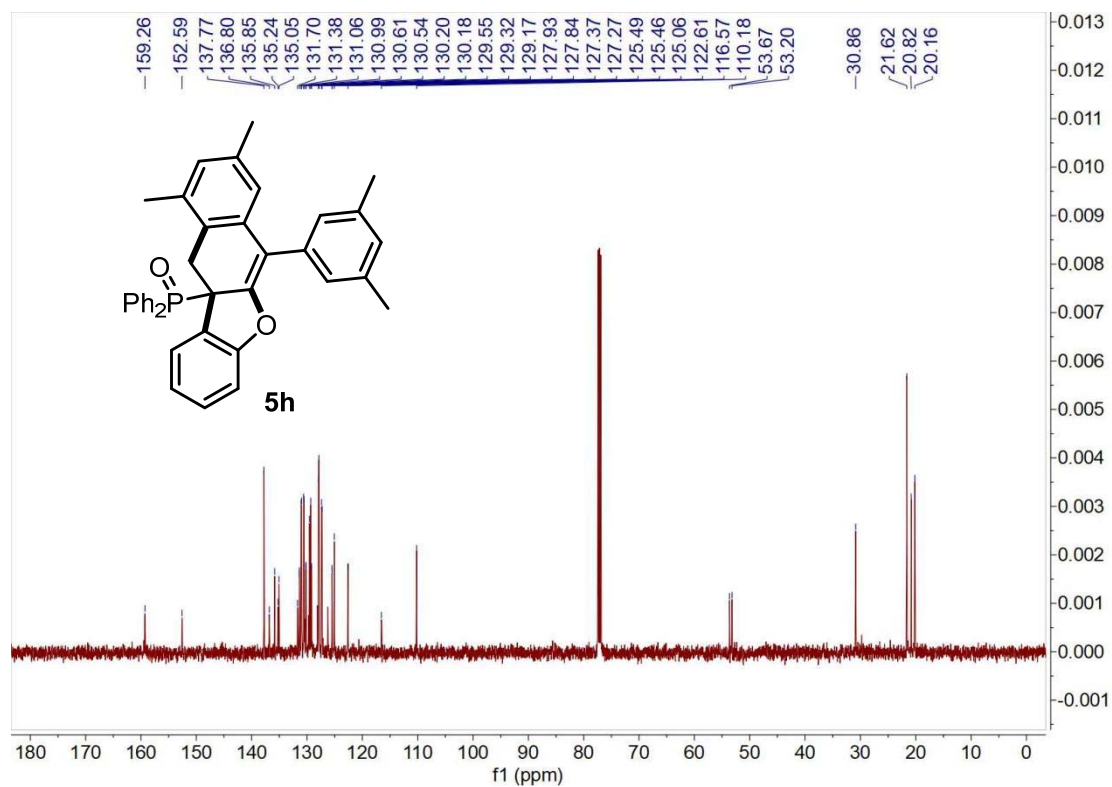
20210624-14 #37 RT: 0.49 AV: 1 NL: 8.79E4
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



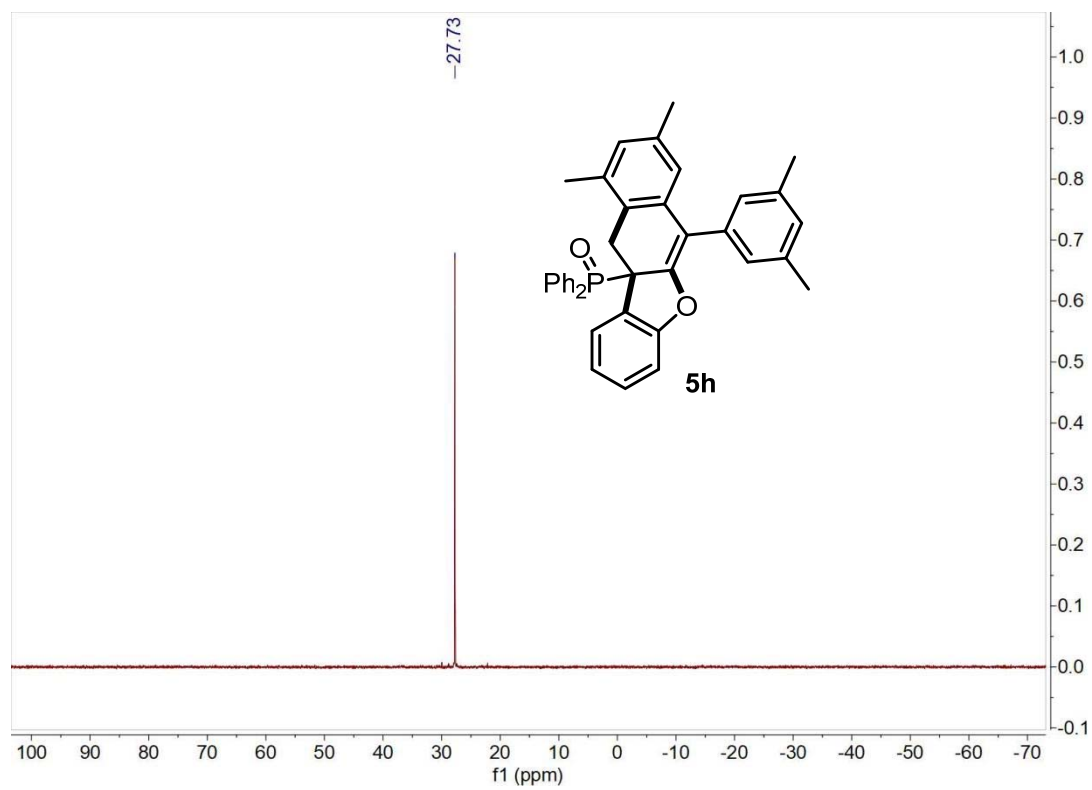
HRMS of **5g**



¹H-NMR (500 MHz, CDCl₃) of **5h**

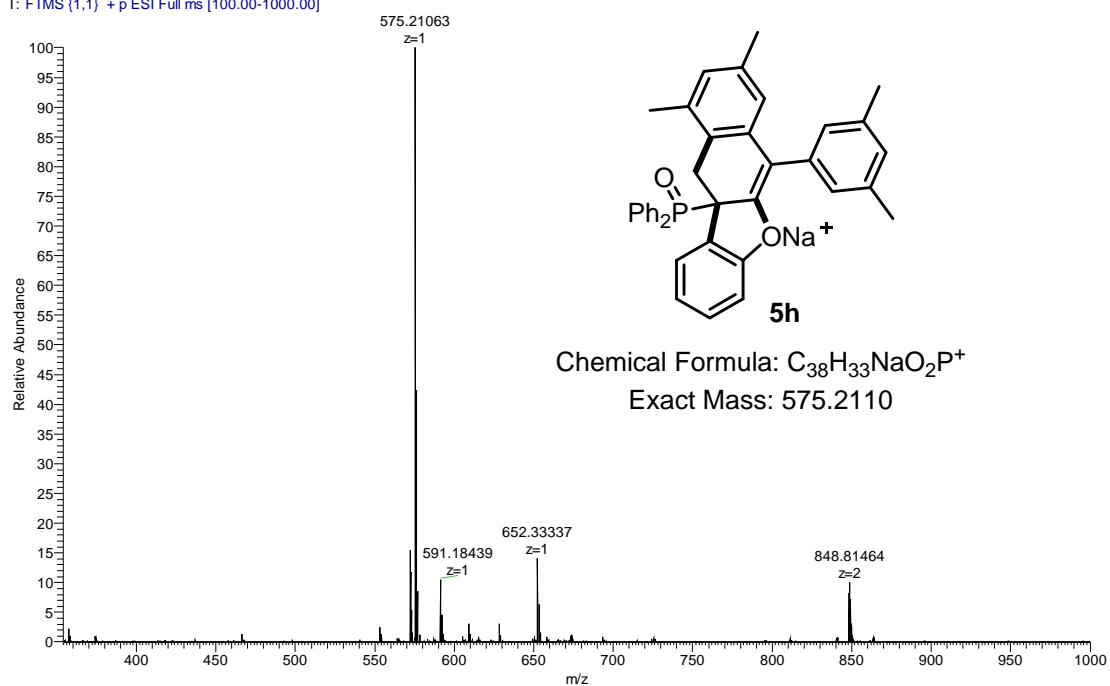


^{13}C -NMR (126 MHz, CDCl_3) of **5h**

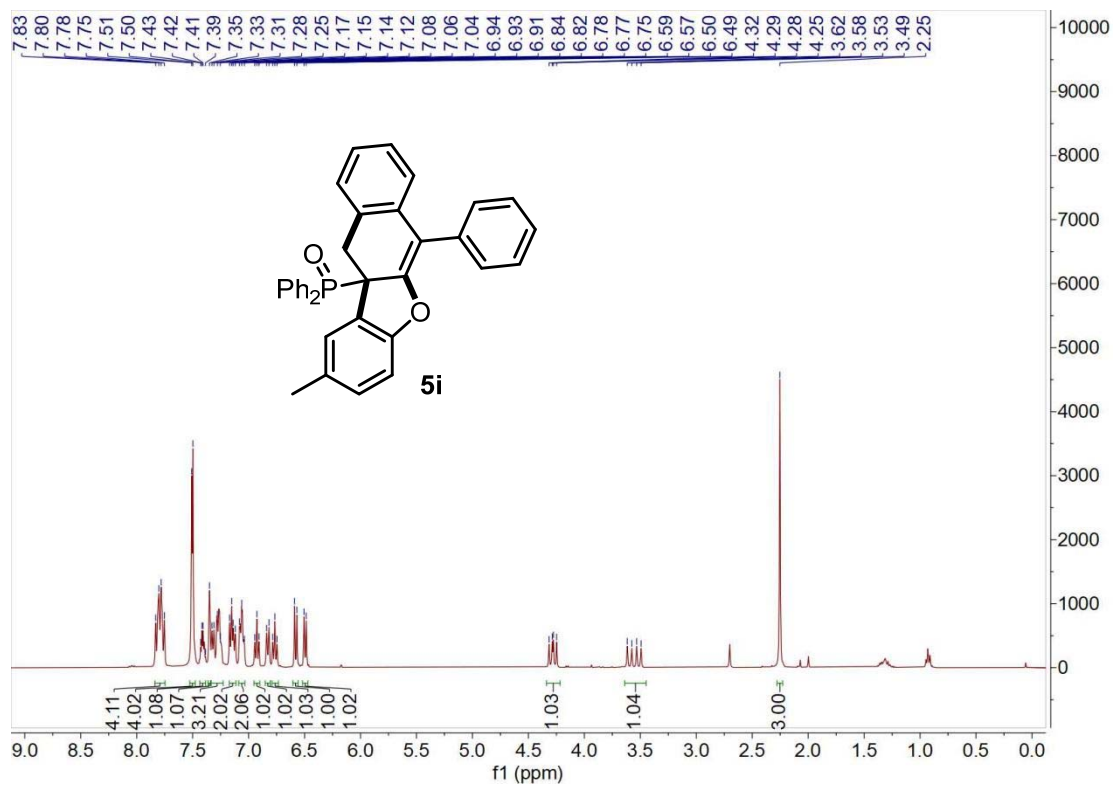


^{31}P -NMR (202 MHz, CDCl_3) of **5h**

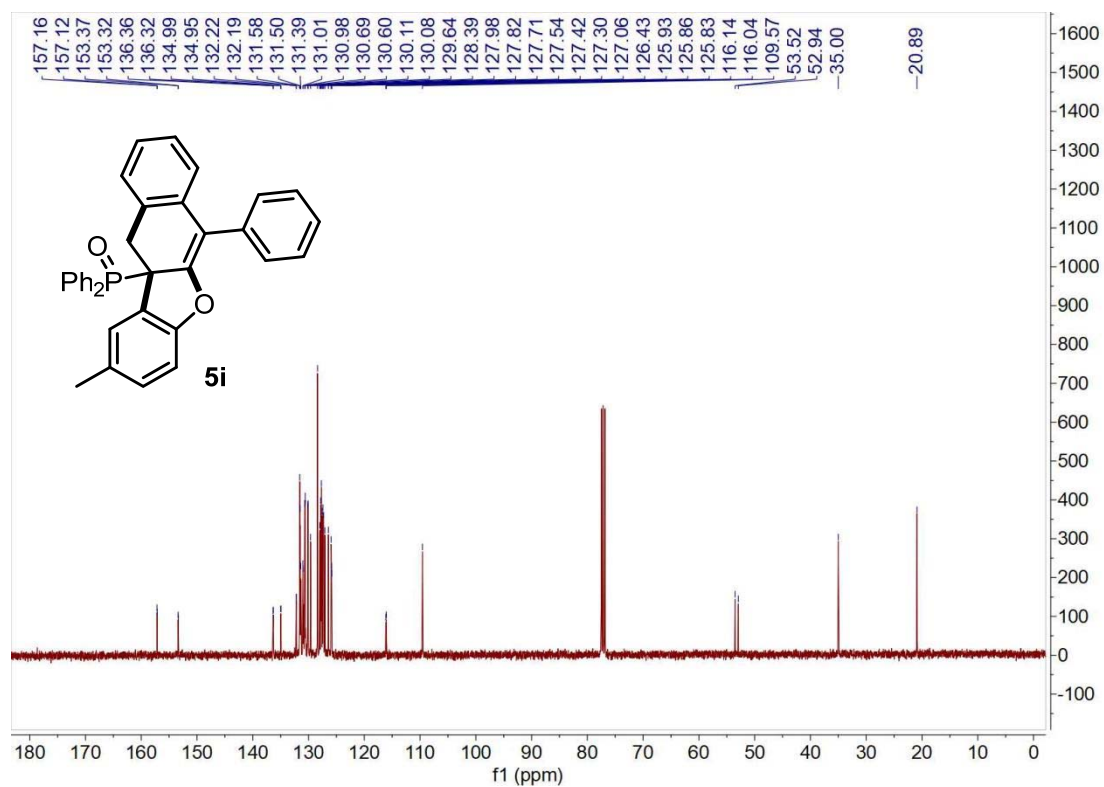
20210719-21 #29 RT: 0.34 AV: 1 NL: 9.87E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



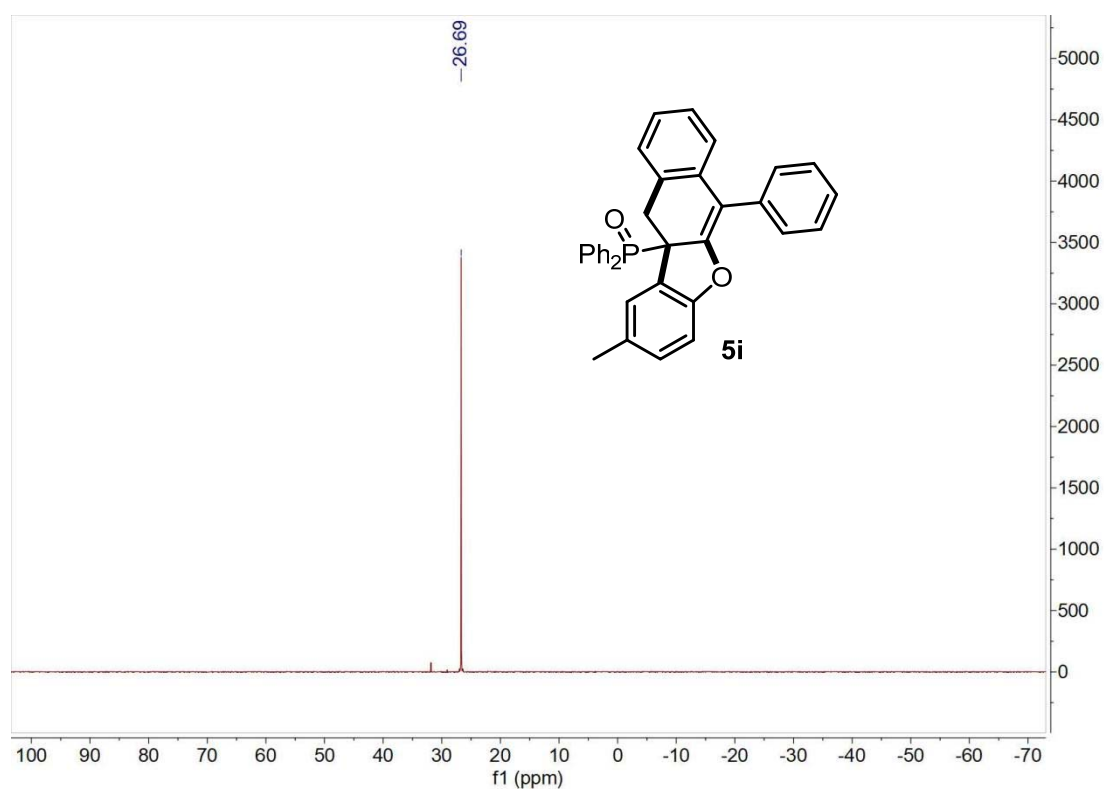
HRMS of **5h**



1H -NMR (400 MHz, $CDCl_3$) of **5i**

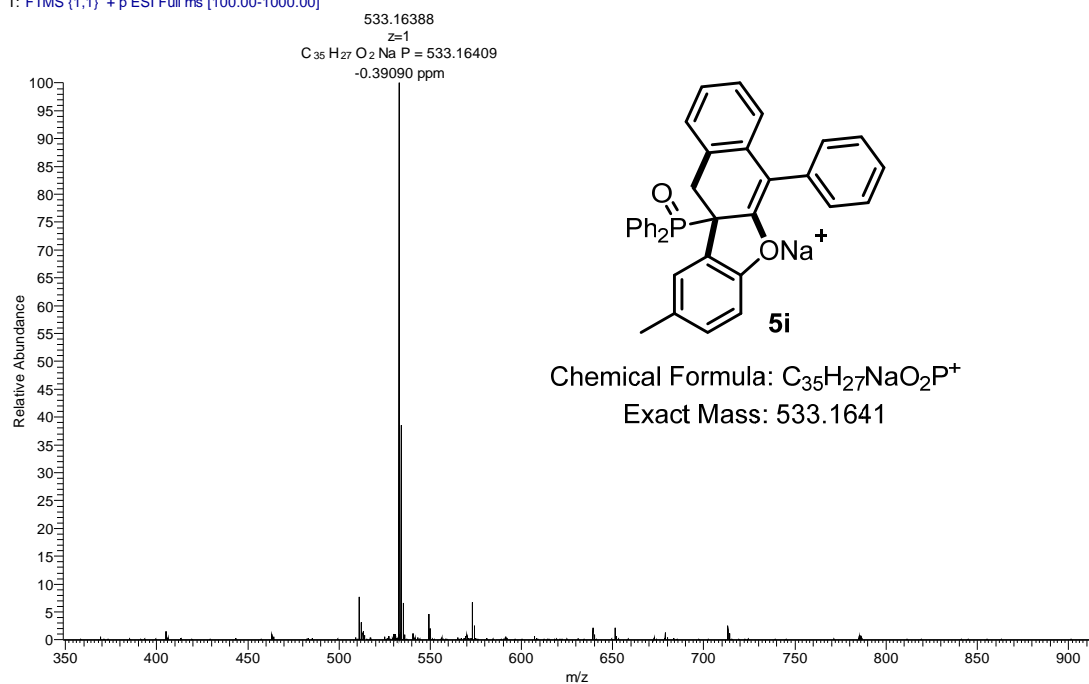


$^{13}\text{C-NMR}$ (101 MHz, CDCl_3) of **5i**

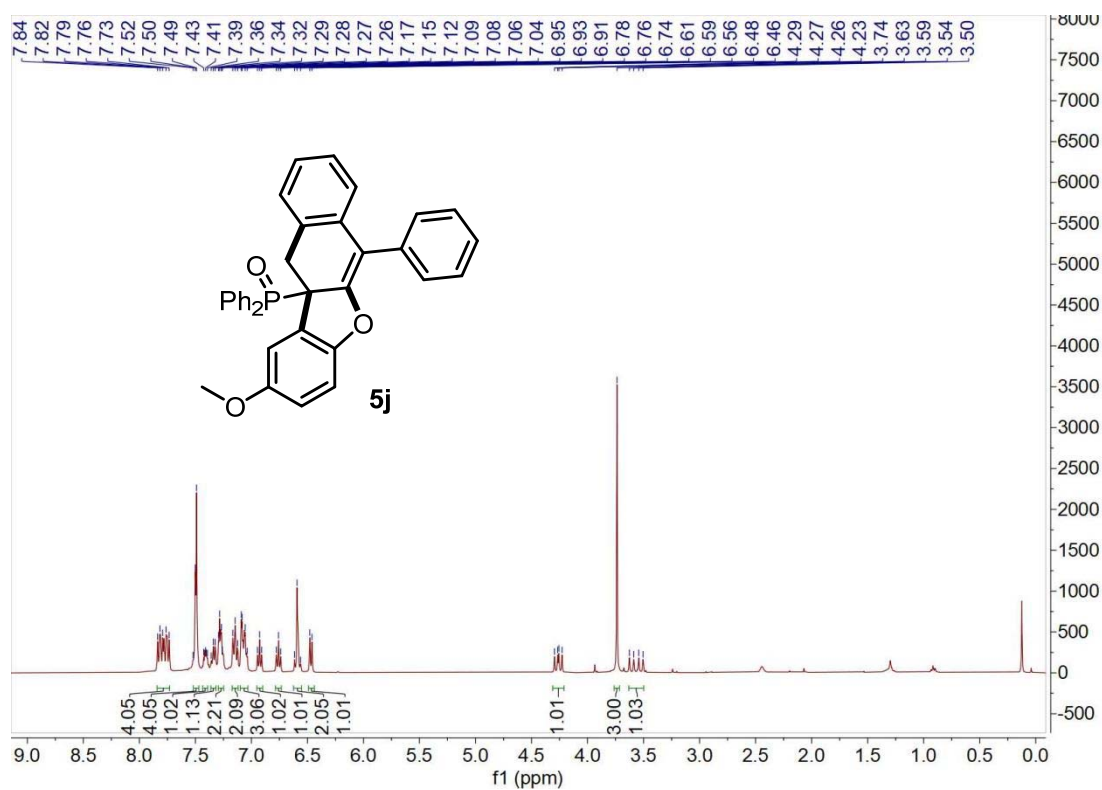


$^{31}\text{P-NMR}$ (162 MHz, CDCl_3) of **5i**

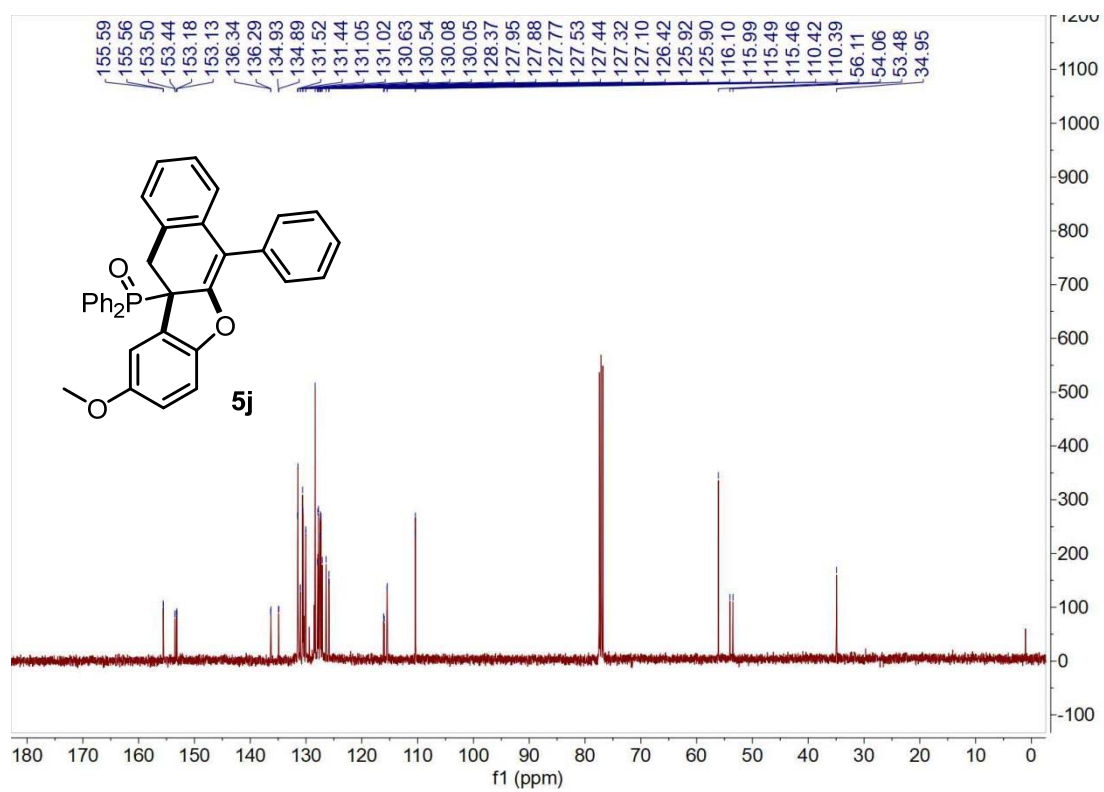
0129-14 #15 RT: 0.23 AV: 1 NL: 4.77E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



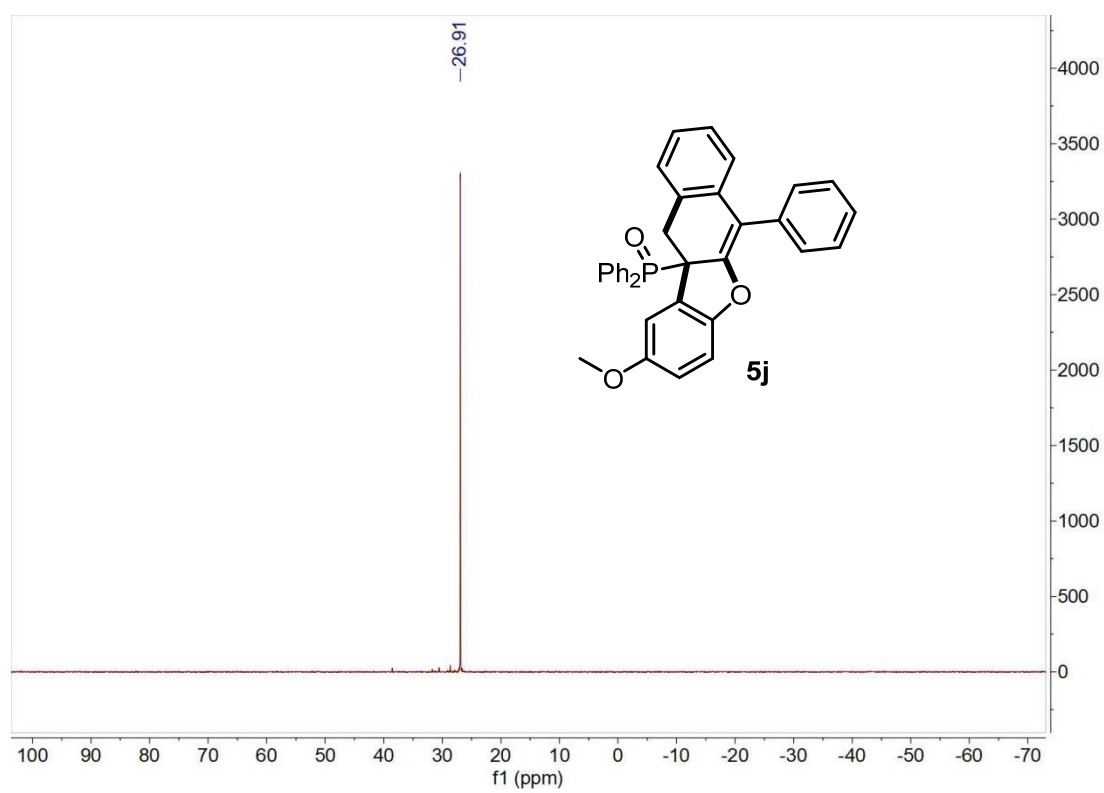
HRMS of **5i**



¹H-NMR (400 MHz, CDCl₃) of **5j**

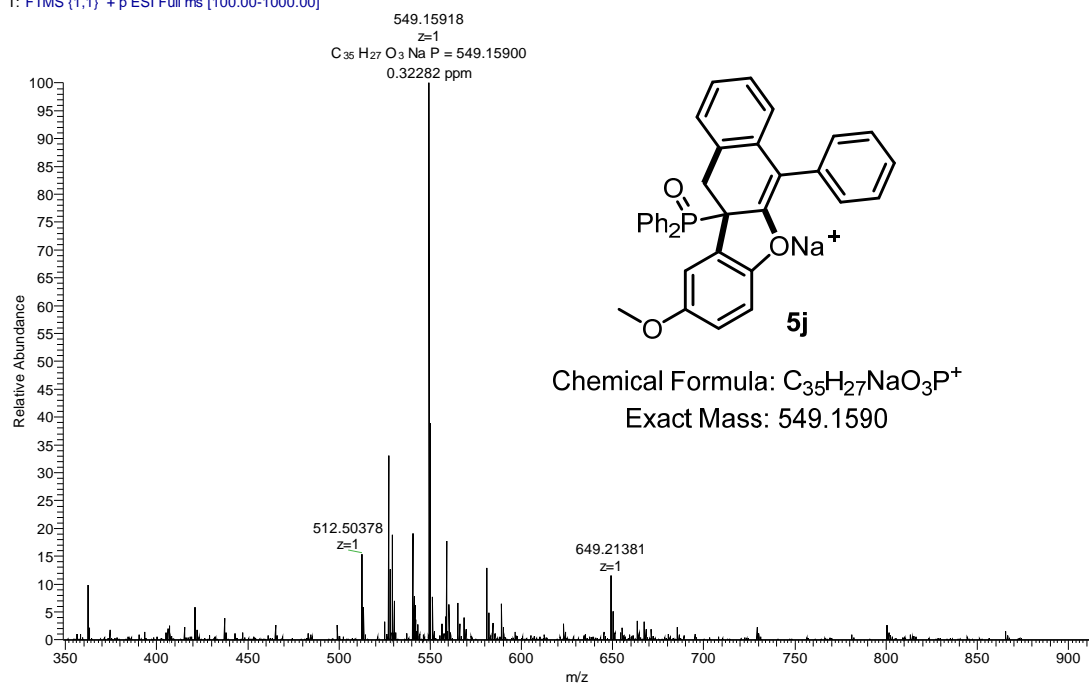


^{13}C -NMR (101 MHz, CDCl_3) of **5j**

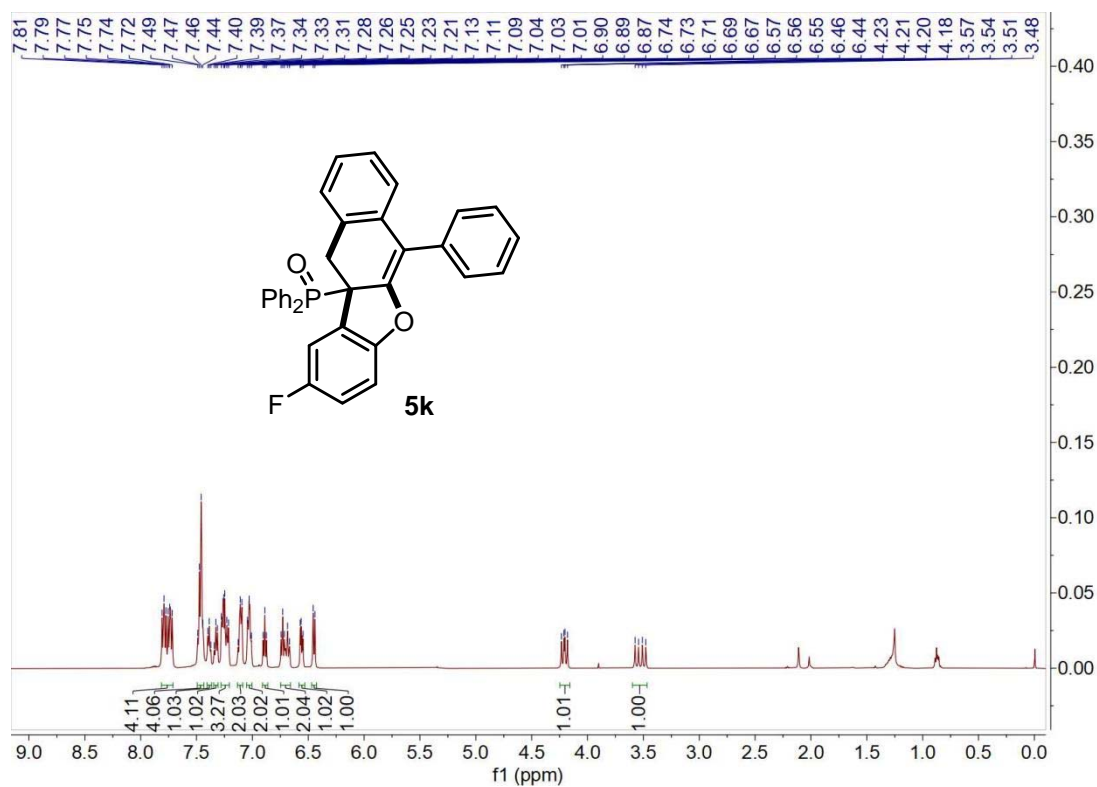


^{31}P -NMR (162 MHz, CDCl_3) of **5j**

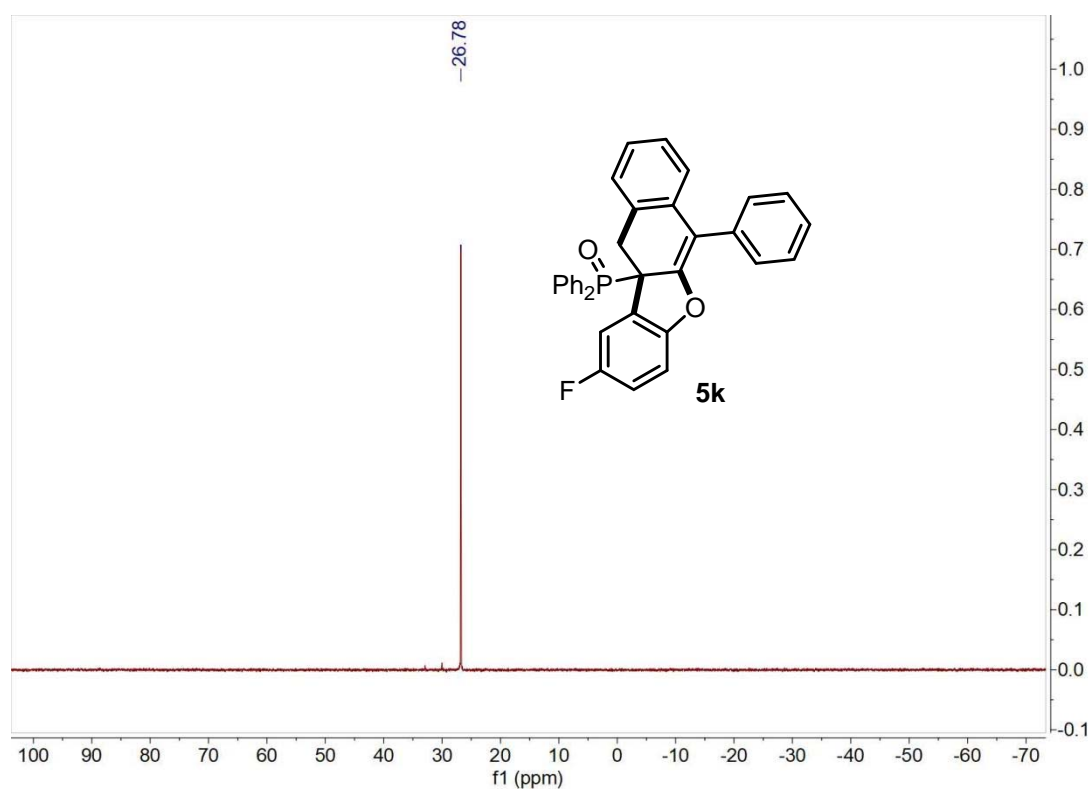
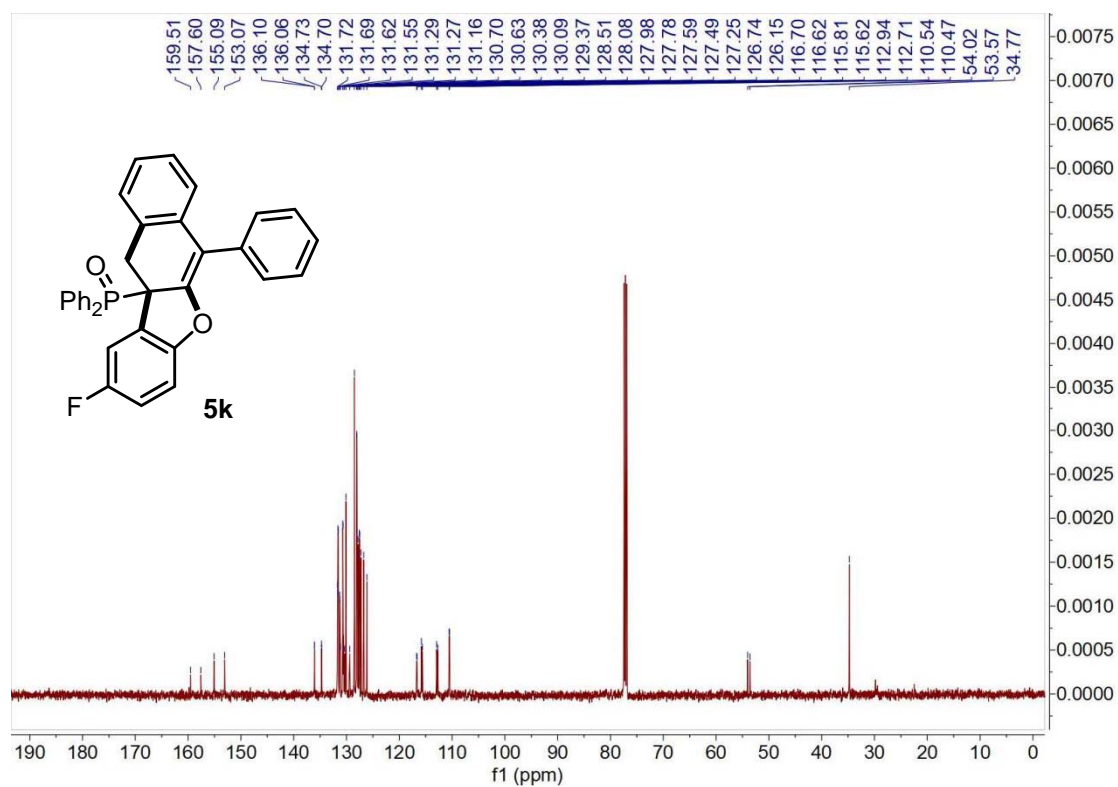
0129-13 #21 RT: 0.27 AV: 1 NL: 2.02E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

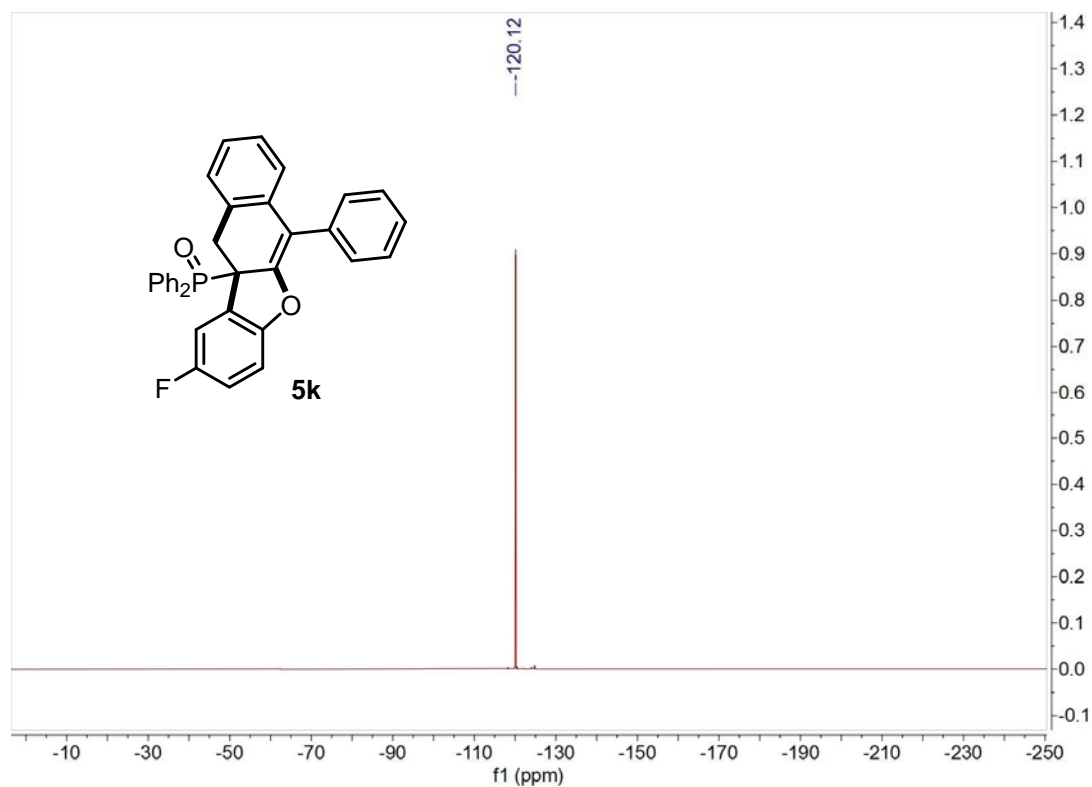


HRMS of **5j**

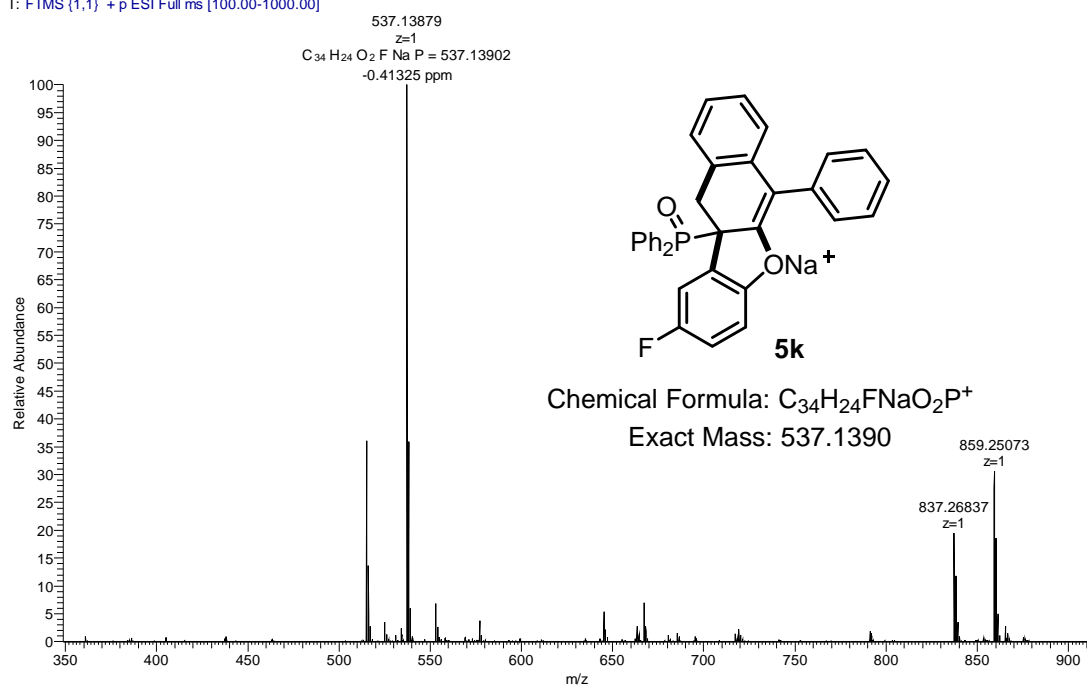


¹H-NMR (500 MHz, CDCl₃) of **5k**

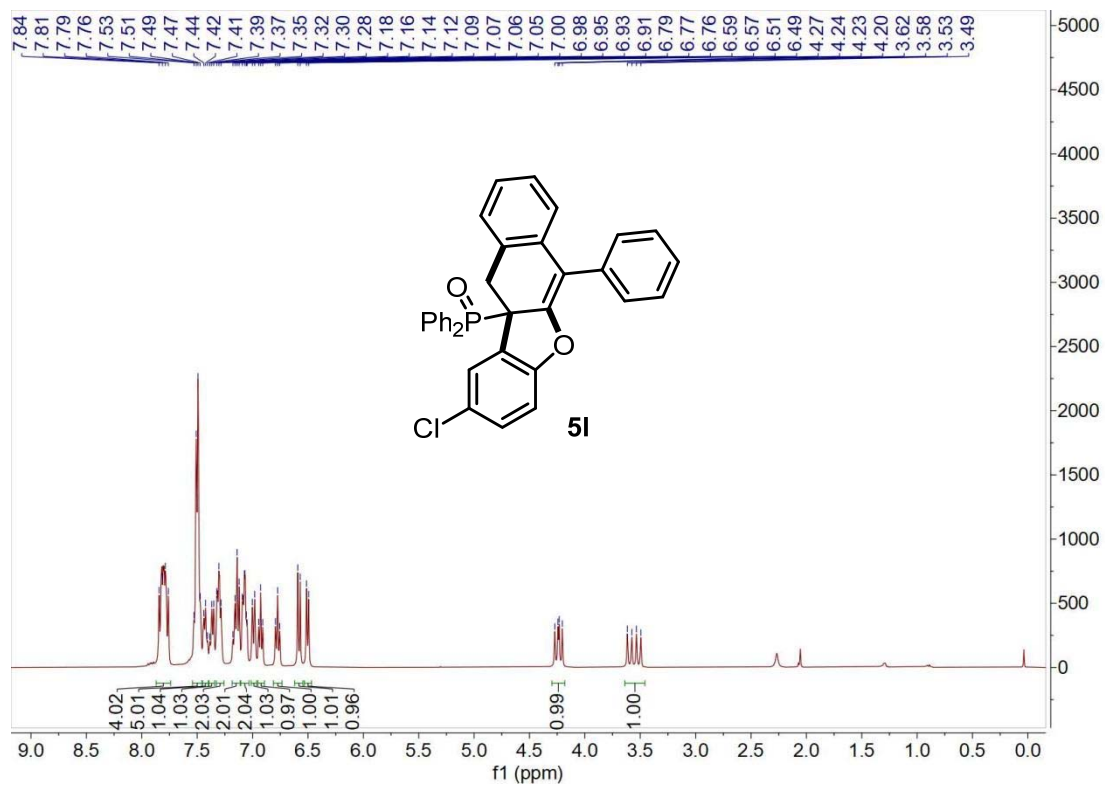




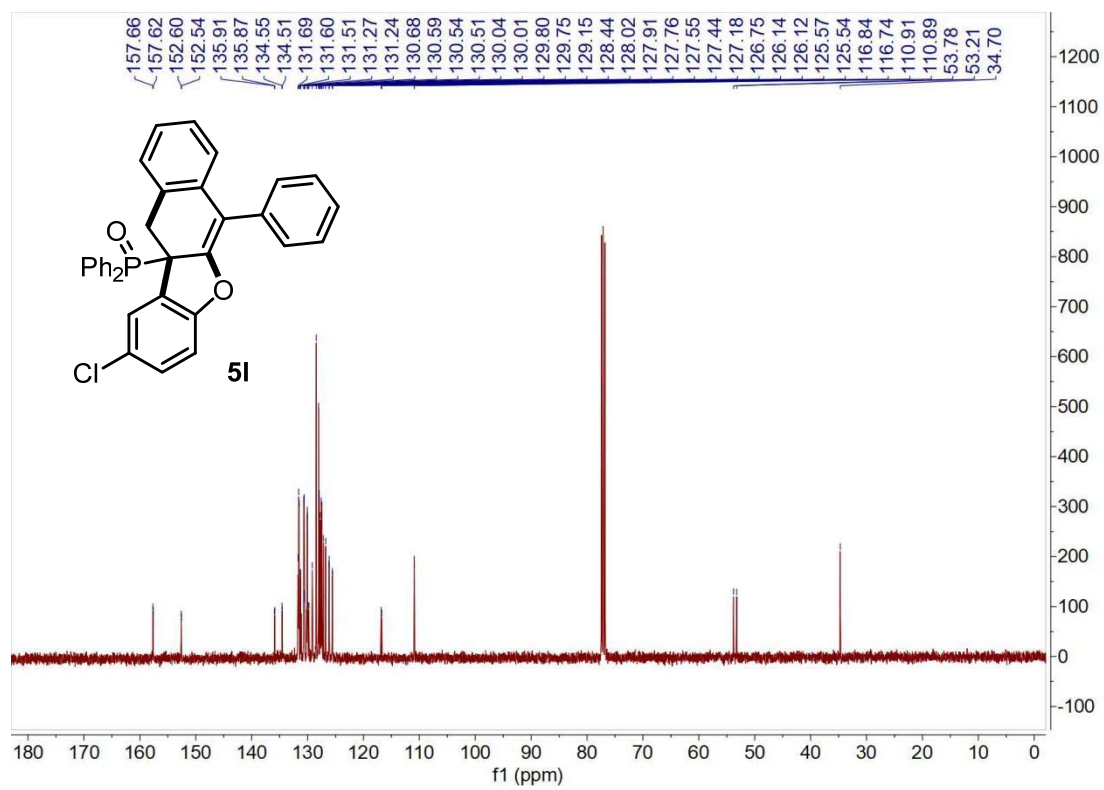
0129-12 #19 RT: 0.24 AV: 1 NL: 5.60E5
 T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



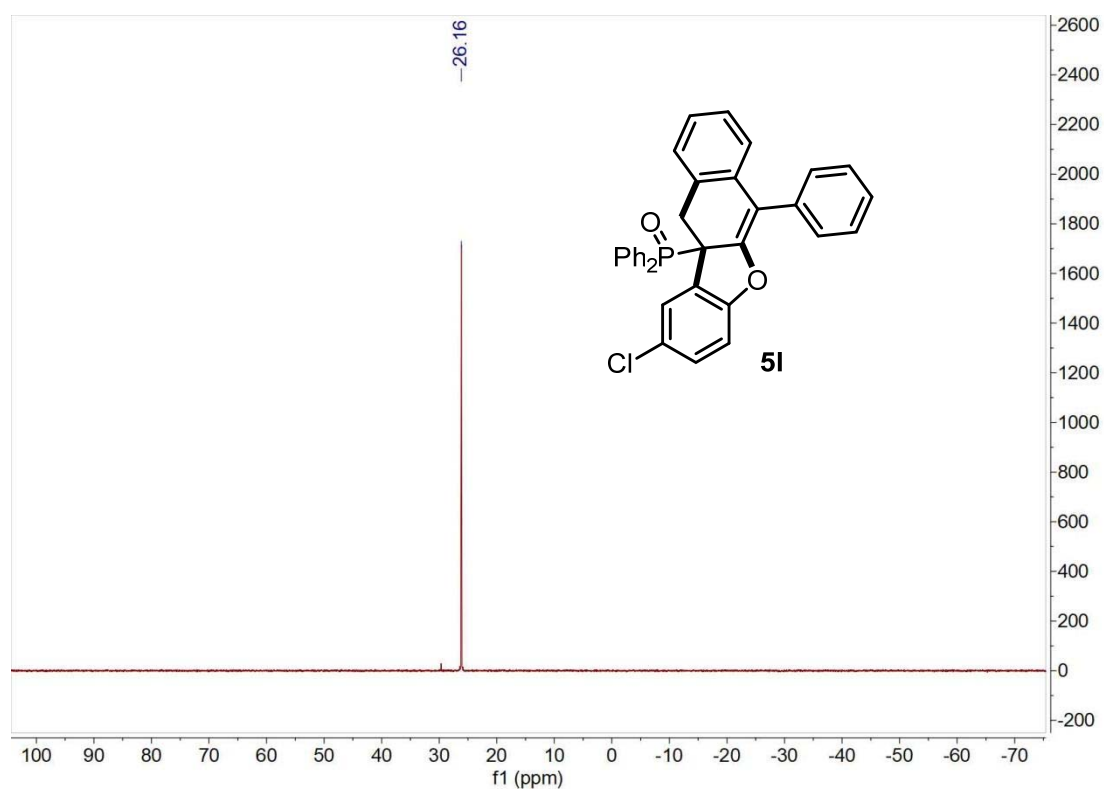
HRMS of **5k**



¹H-NMR (400 MHz, CDCl₃) of **5I**

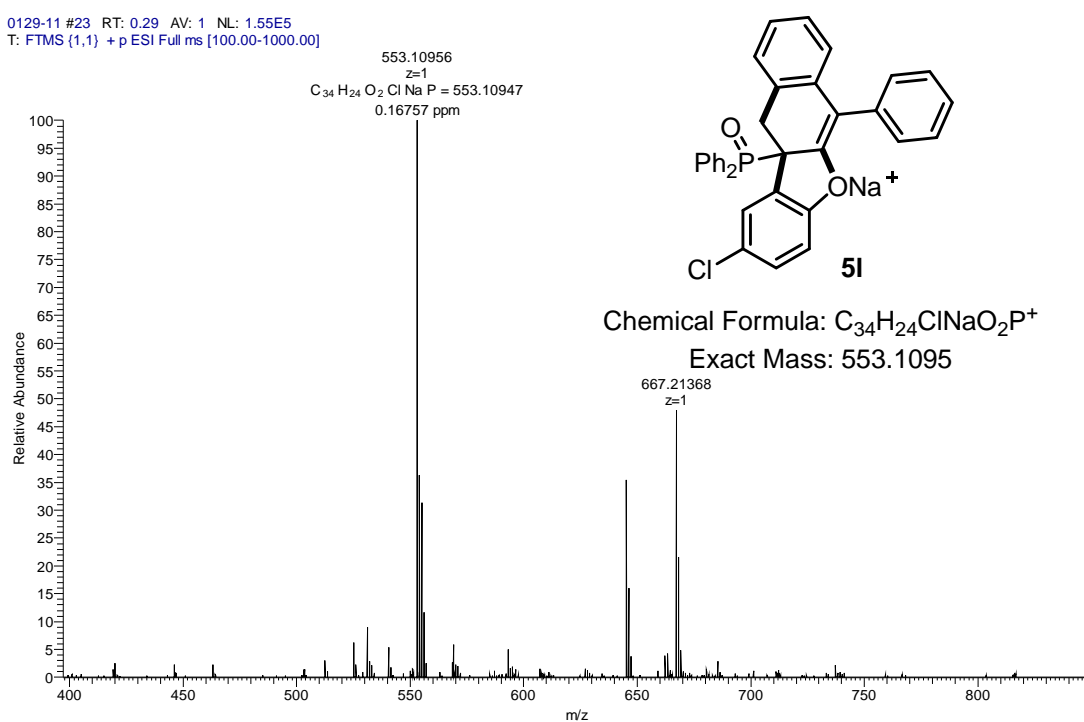


¹³C-NMR (101 MHz, CDCl₃) of **5I**

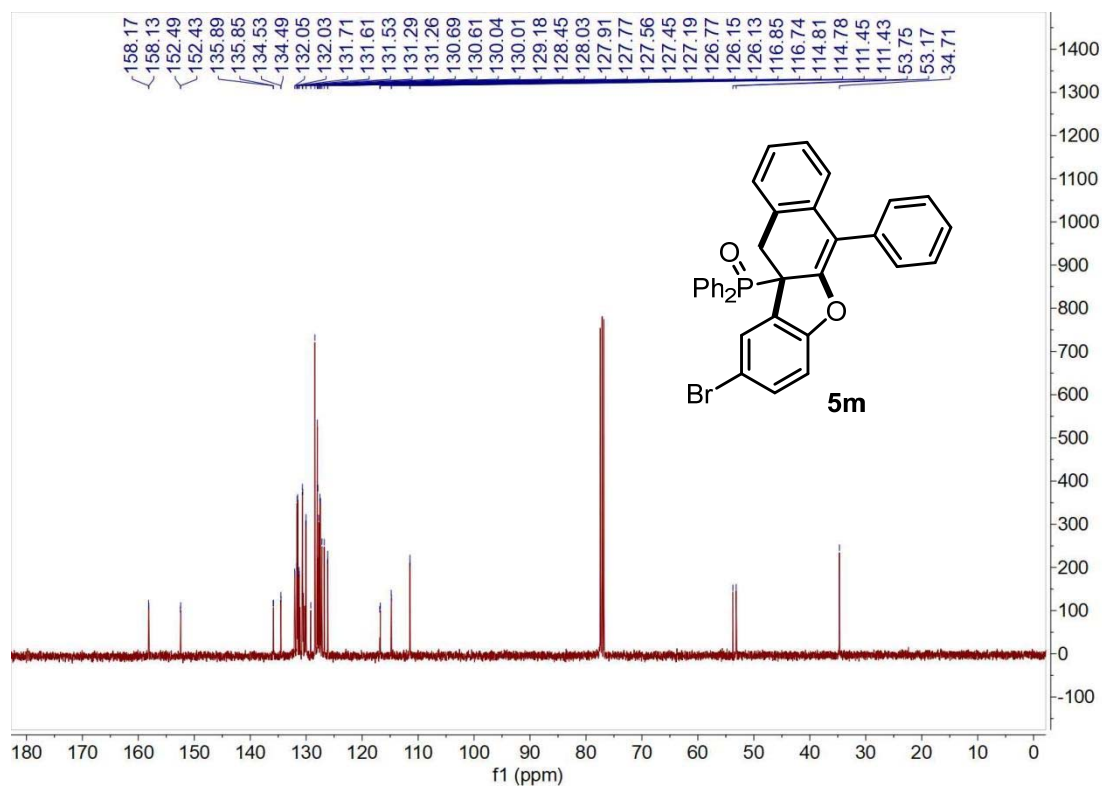
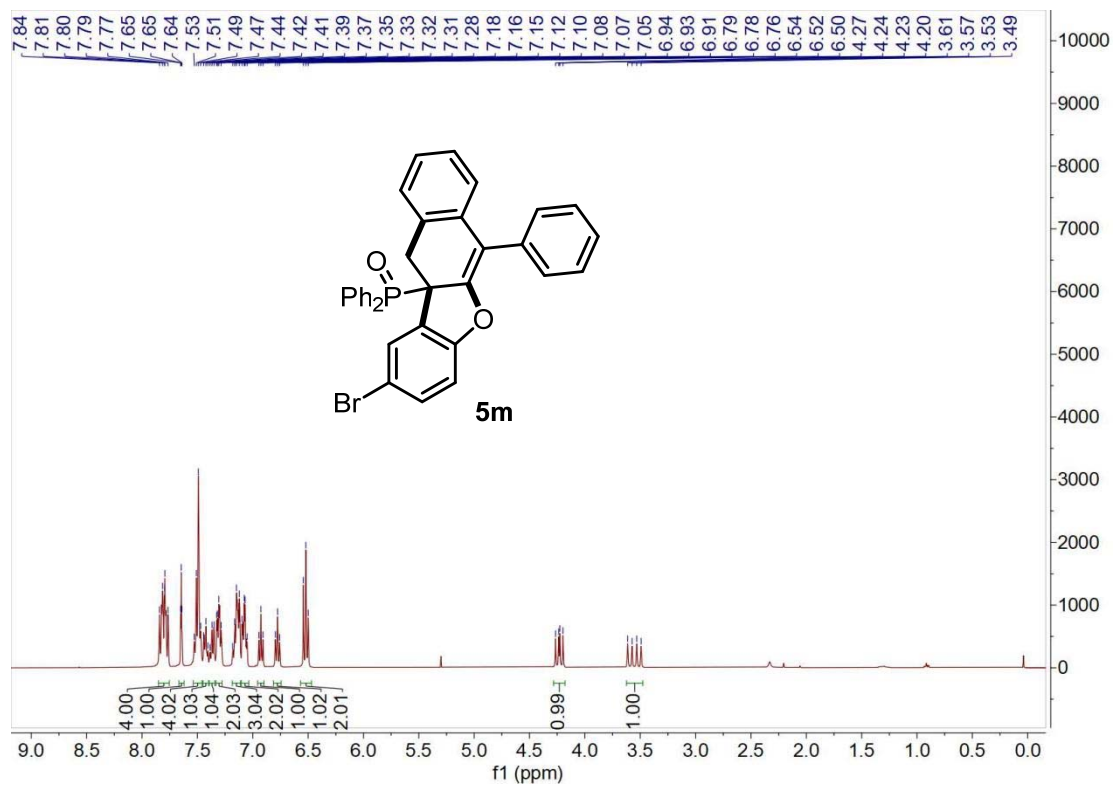


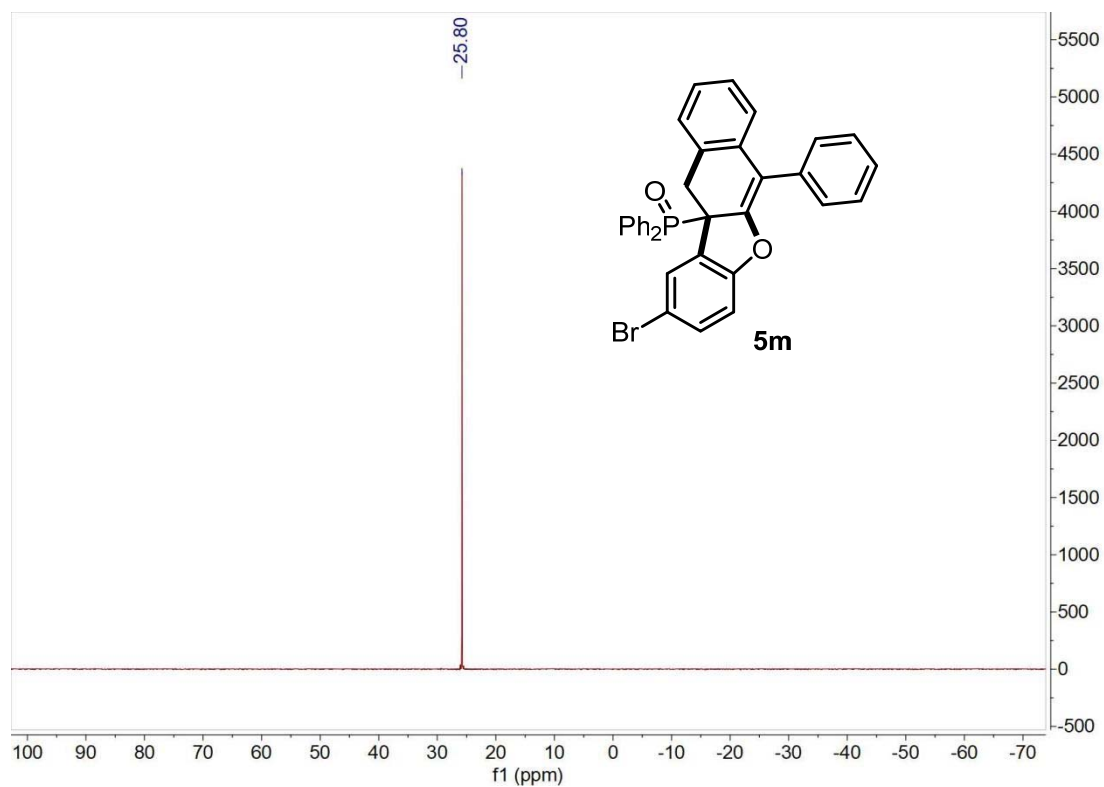
31P-NMR (162 MHz, CDCl₃) of **5I**

0129-11 #23 RT: 0.29 AV: 1 NL: 1.55E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

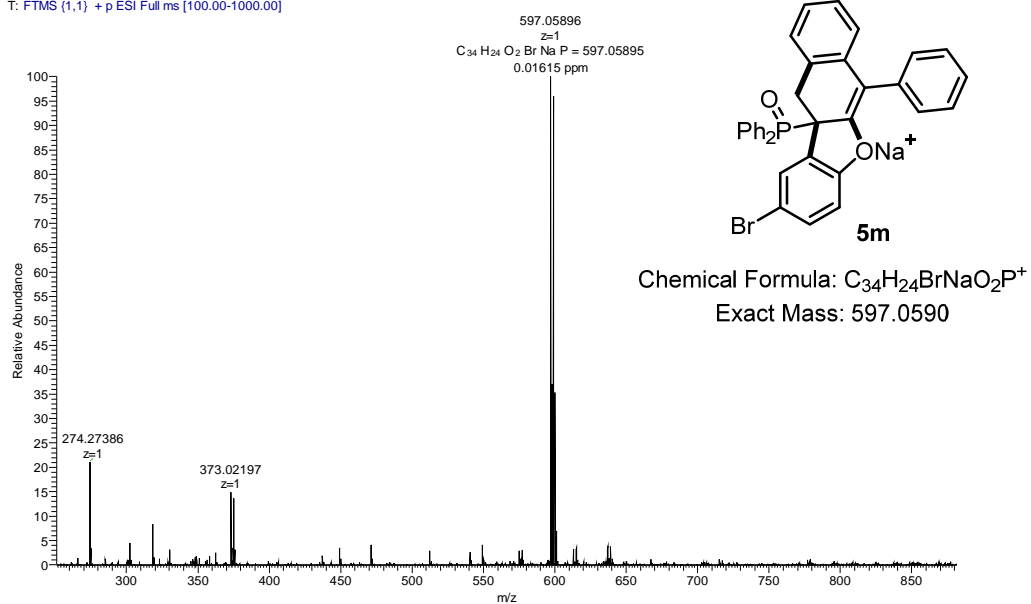


HRMS of **5I**

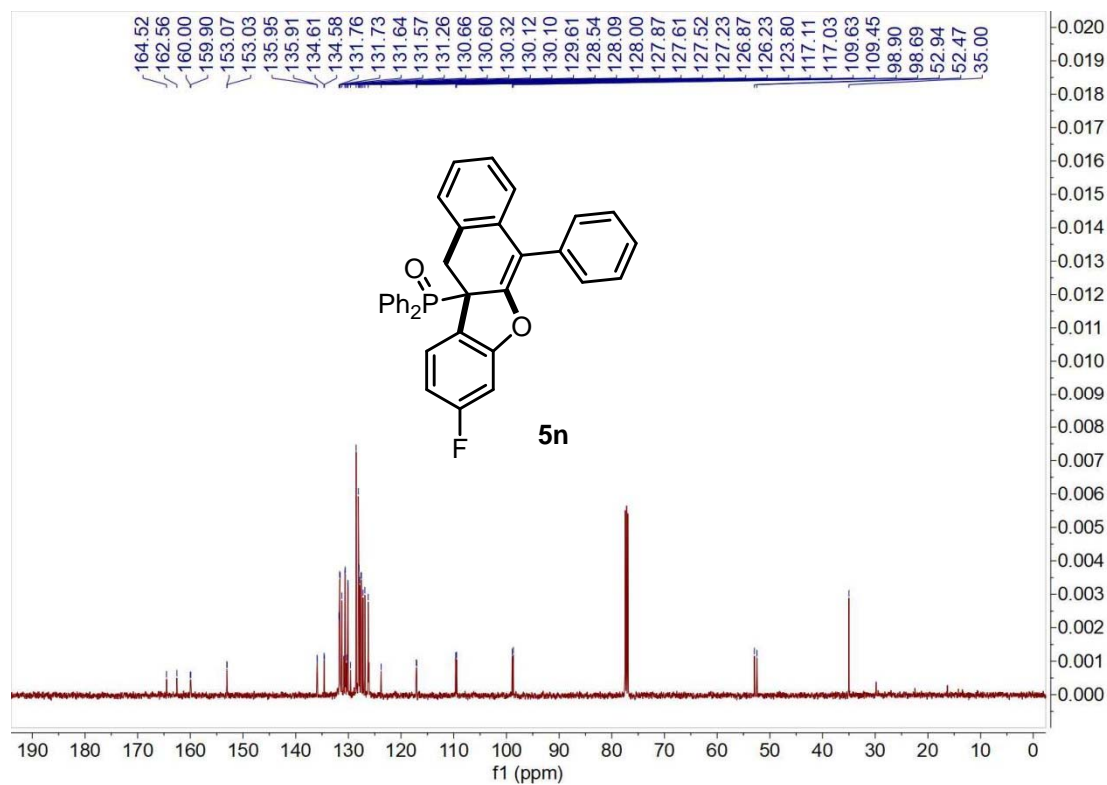
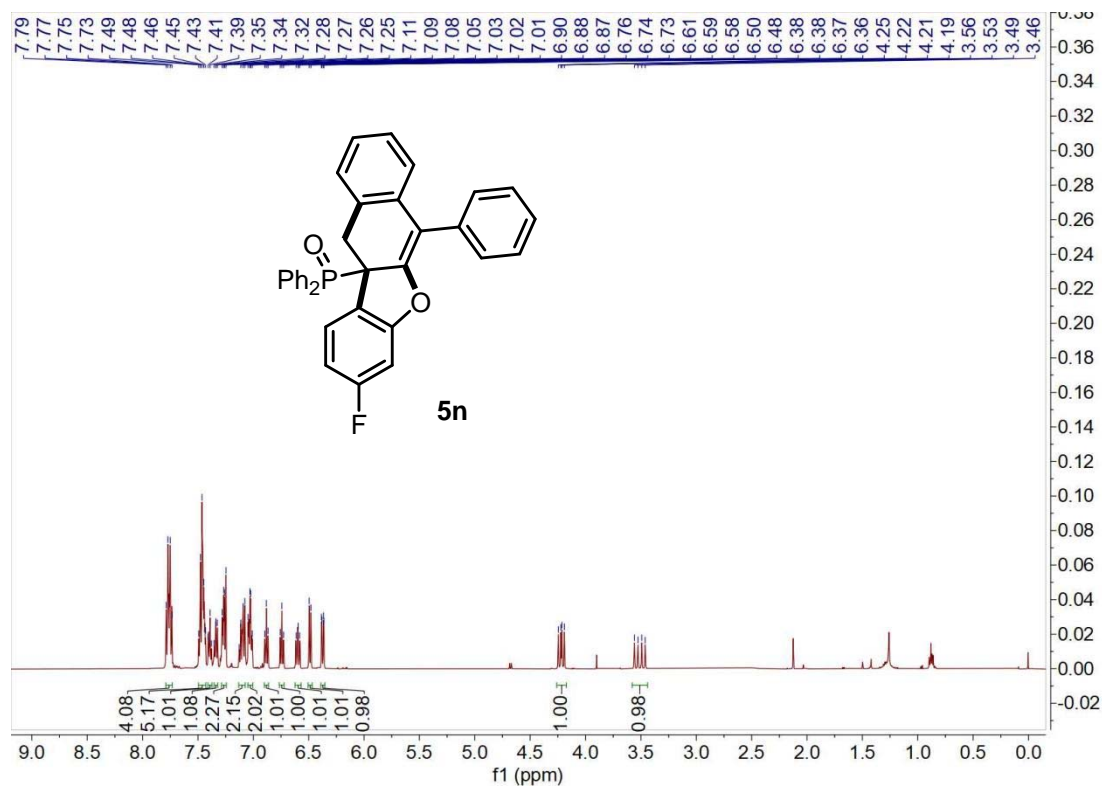


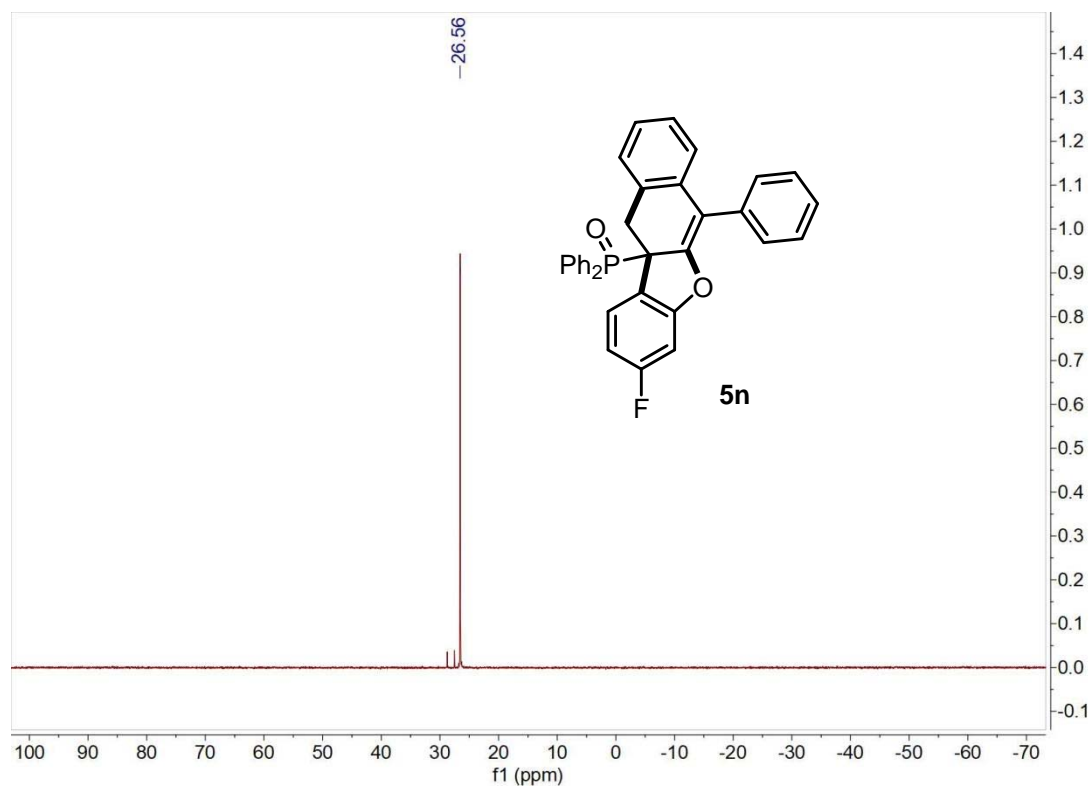


0129-10 #15 RT: 0.20 AV: 1 NL: 3.12E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

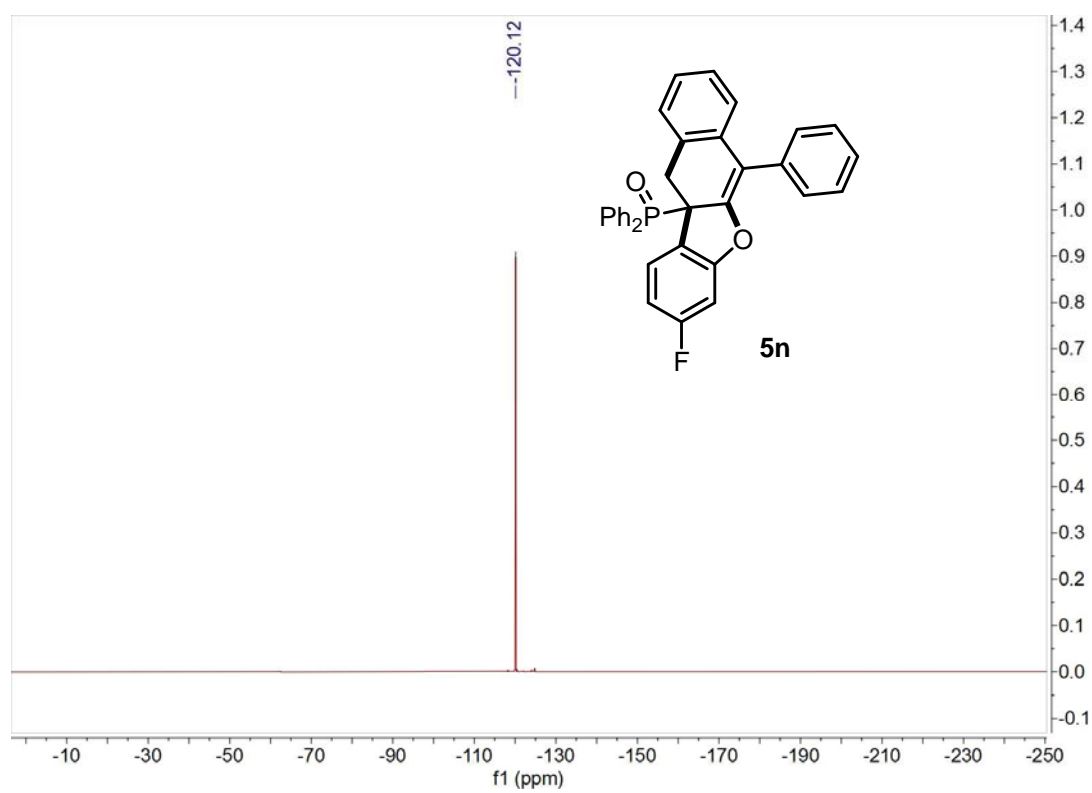


HRMS of **5m**



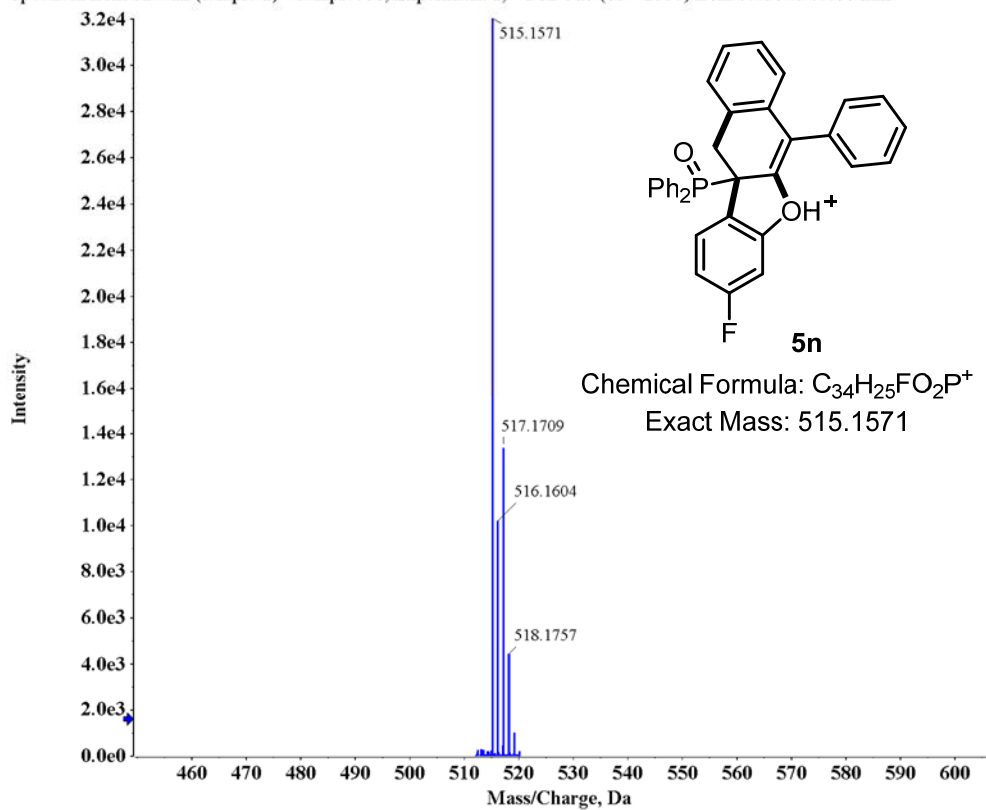


^{31}P -NMR (202 MHz, CDCl_3) of **5n**

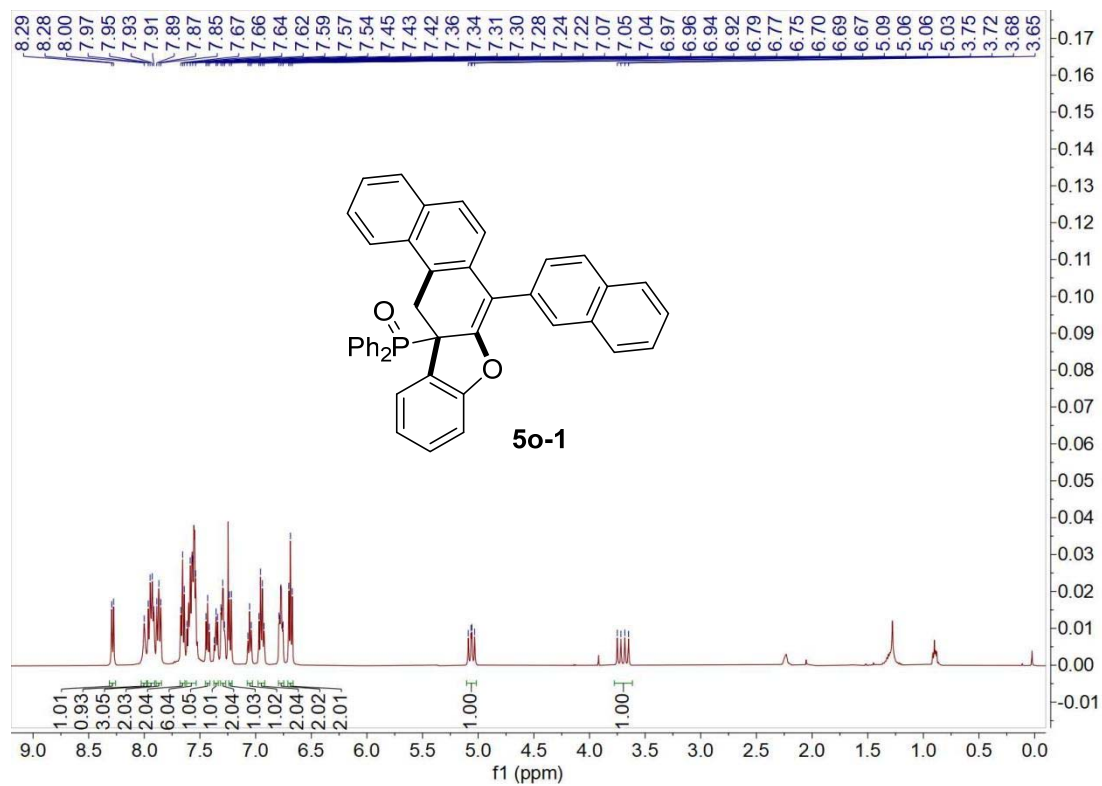


^{19}F -NMR (471 MHz, CDCl_3) of **5n**

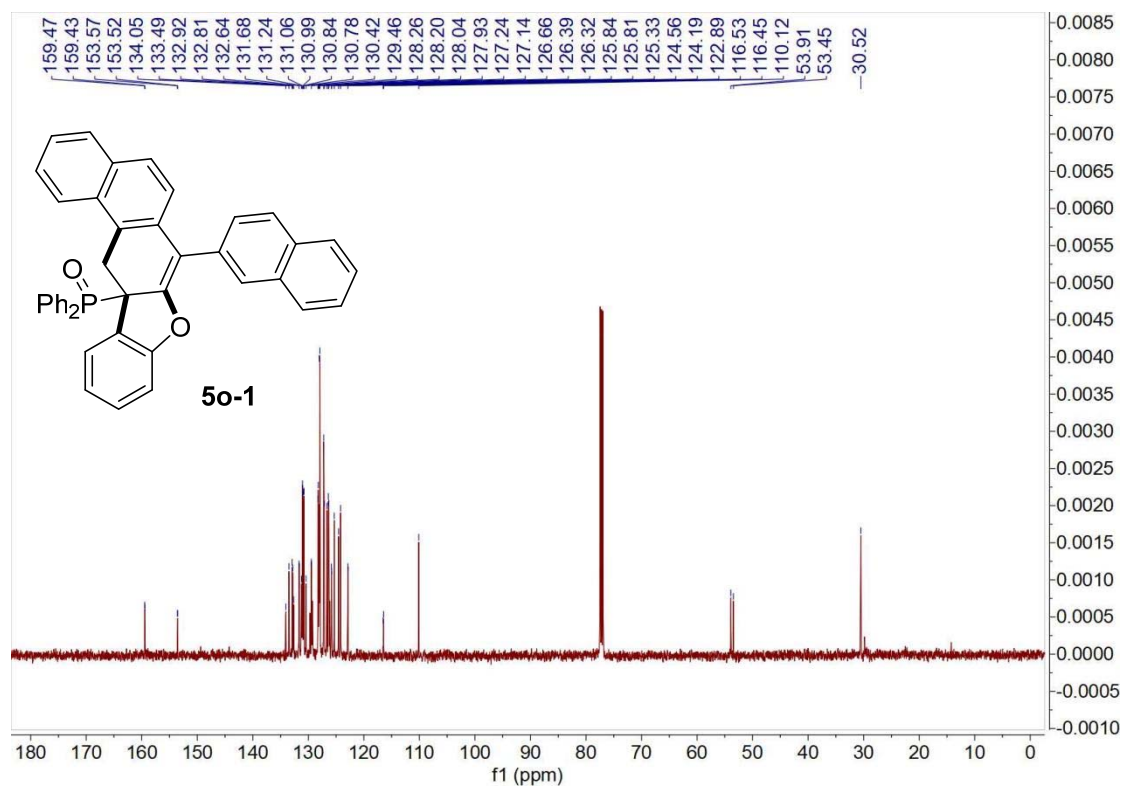
Spectrum from 02.wiff (sample 1) - Sample008, Experiment 1, +TOF MS (80 - 1000) from 0.436 to 0.480 min



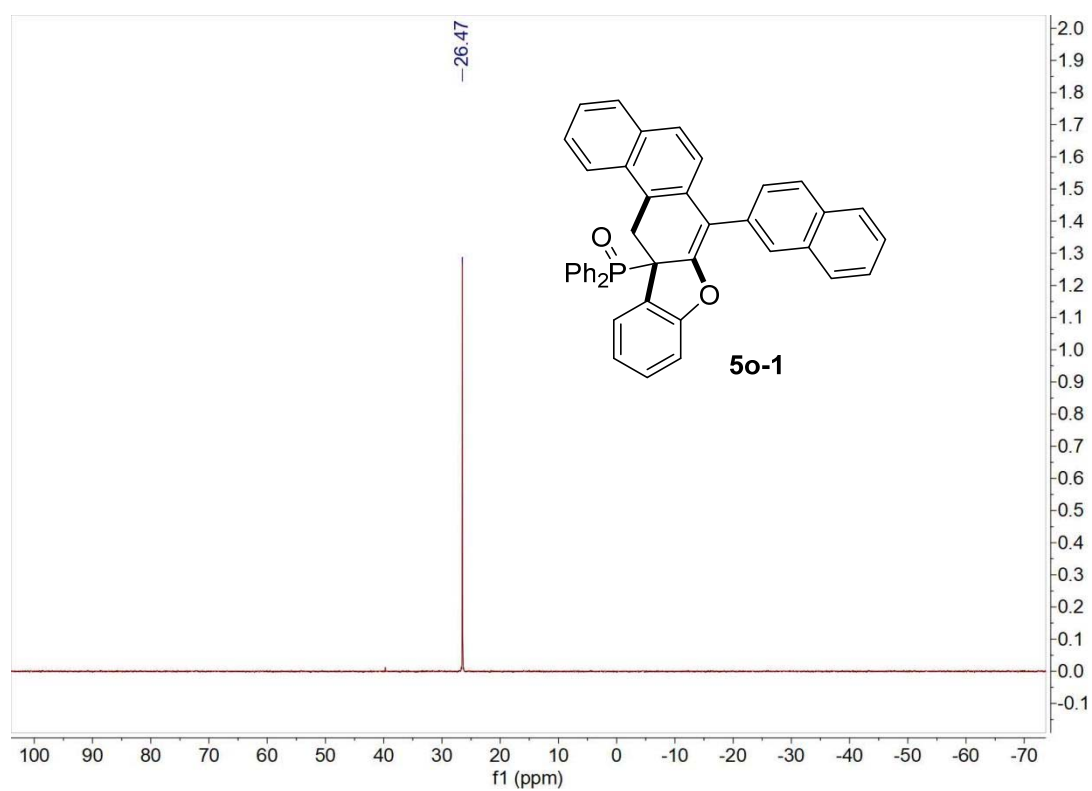
HRMS of **5n**



1H -NMR (500 MHz, $CDCl_3$) of **5o-1**

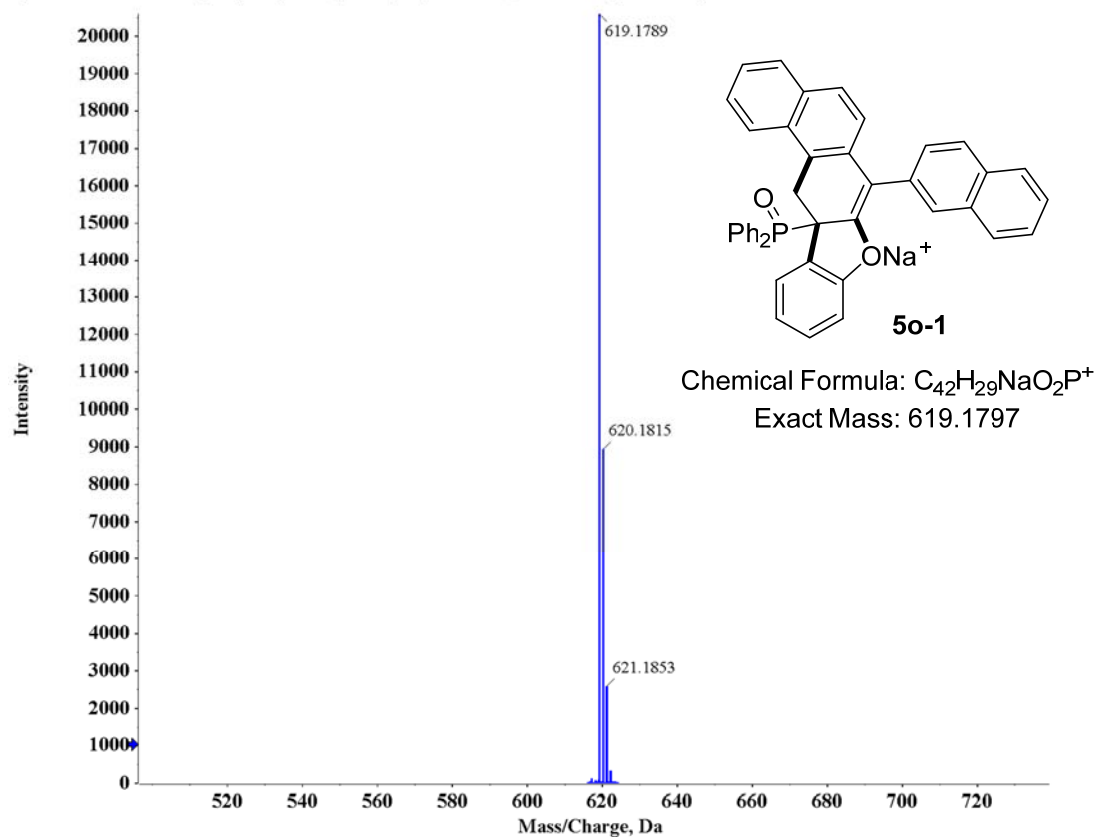


^{13}C -NMR (126 MHz, CDCl_3) of **5o-1**

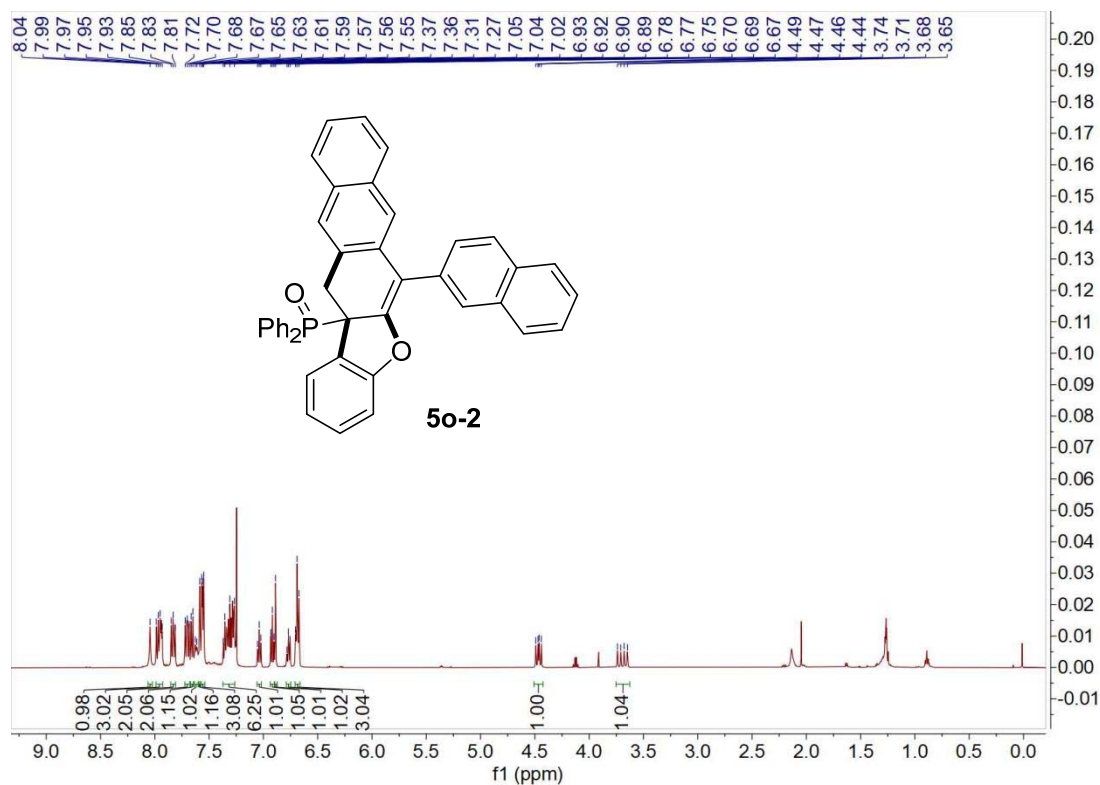


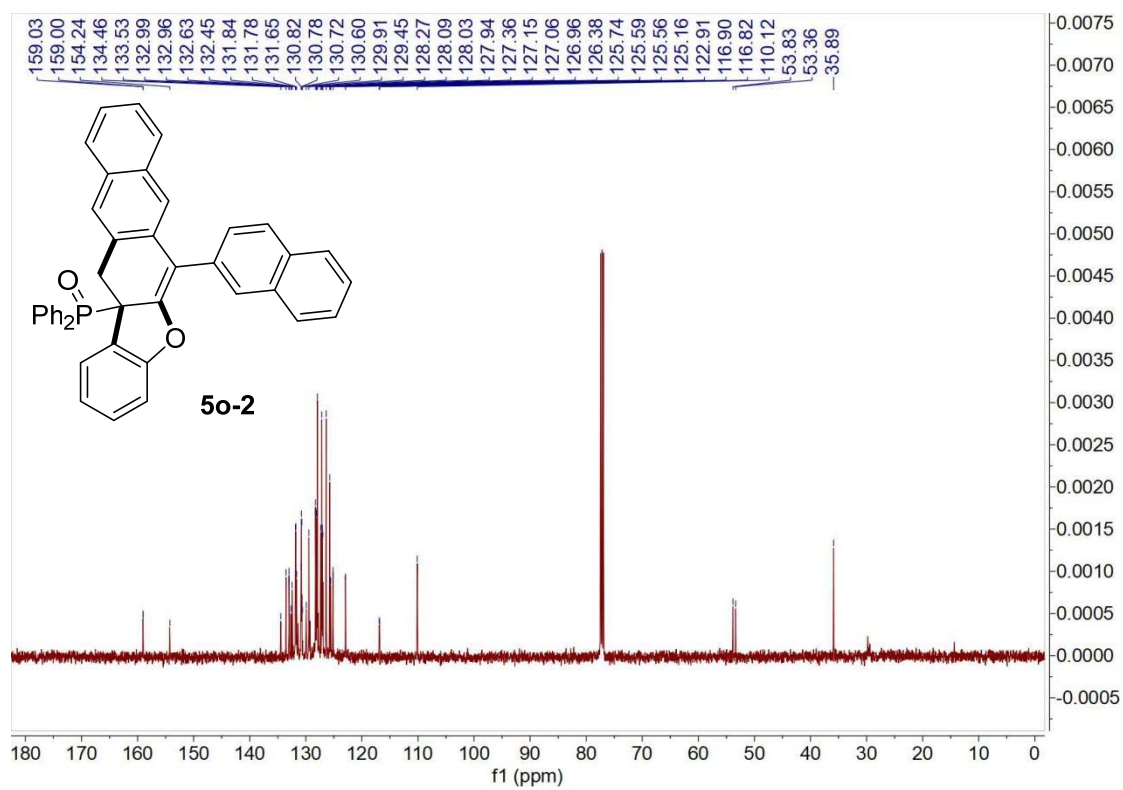
^{31}P -NMR (202 MHz, CDCl_3) of **5o-1**

Spectrum from 03.wiff (sample 1) - Sample009, Experiment 1, +TOF MS (80 - 1000) from 1.011 to 1.056 min

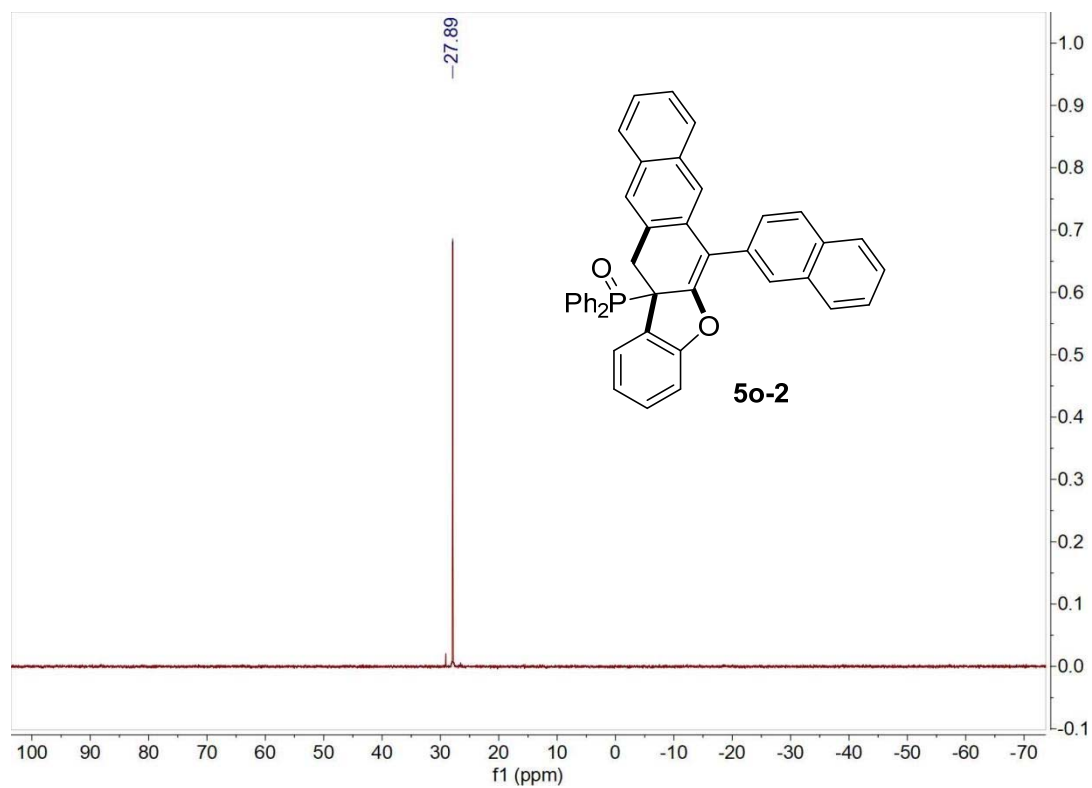


HRMS of **5o-1**



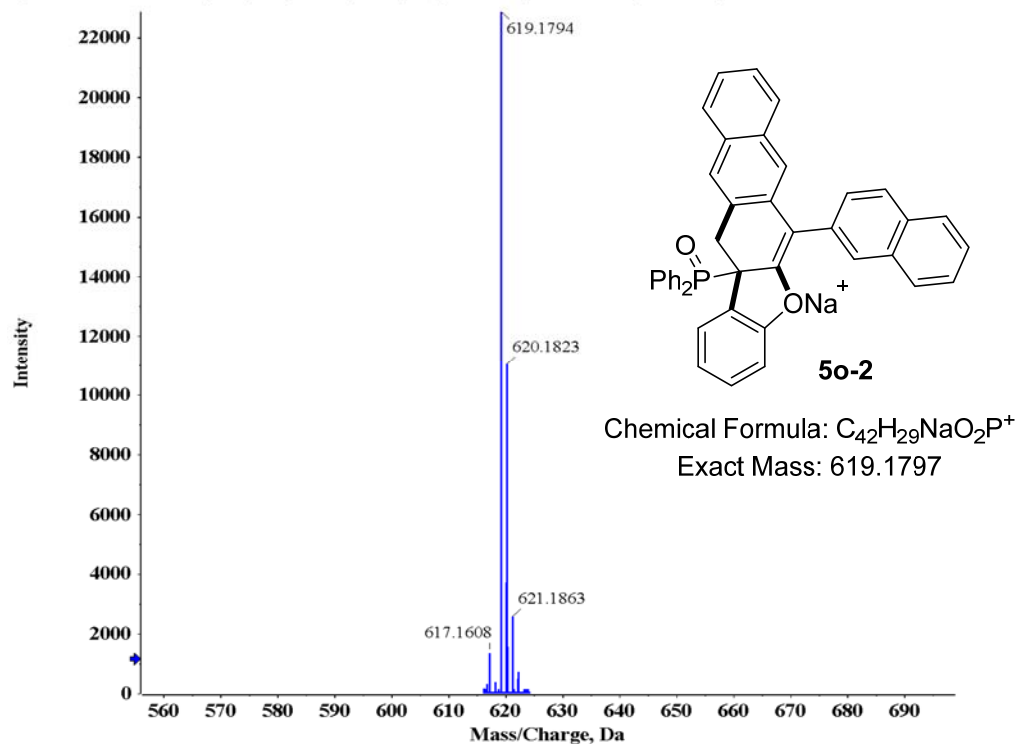


^{13}C -NMR (126 MHz, CDCl_3) of **5o-2**

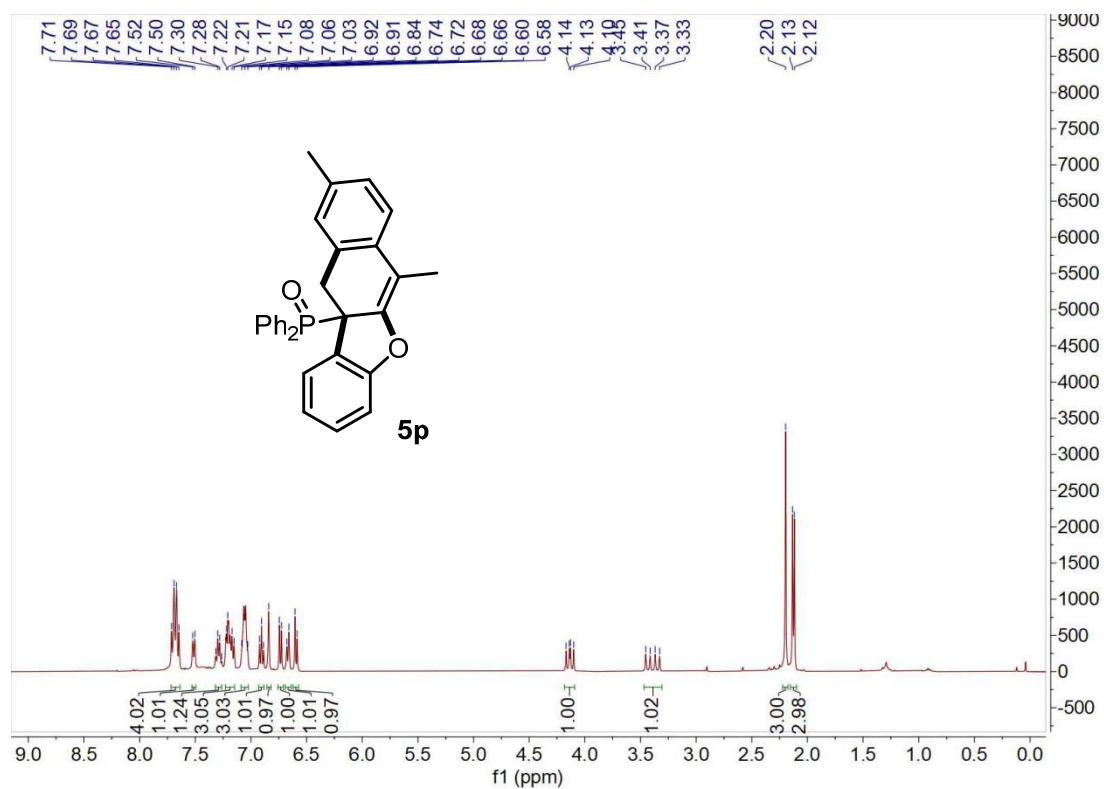


^{31}P -NMR (202 MHz, CDCl_3) of **5o-2**

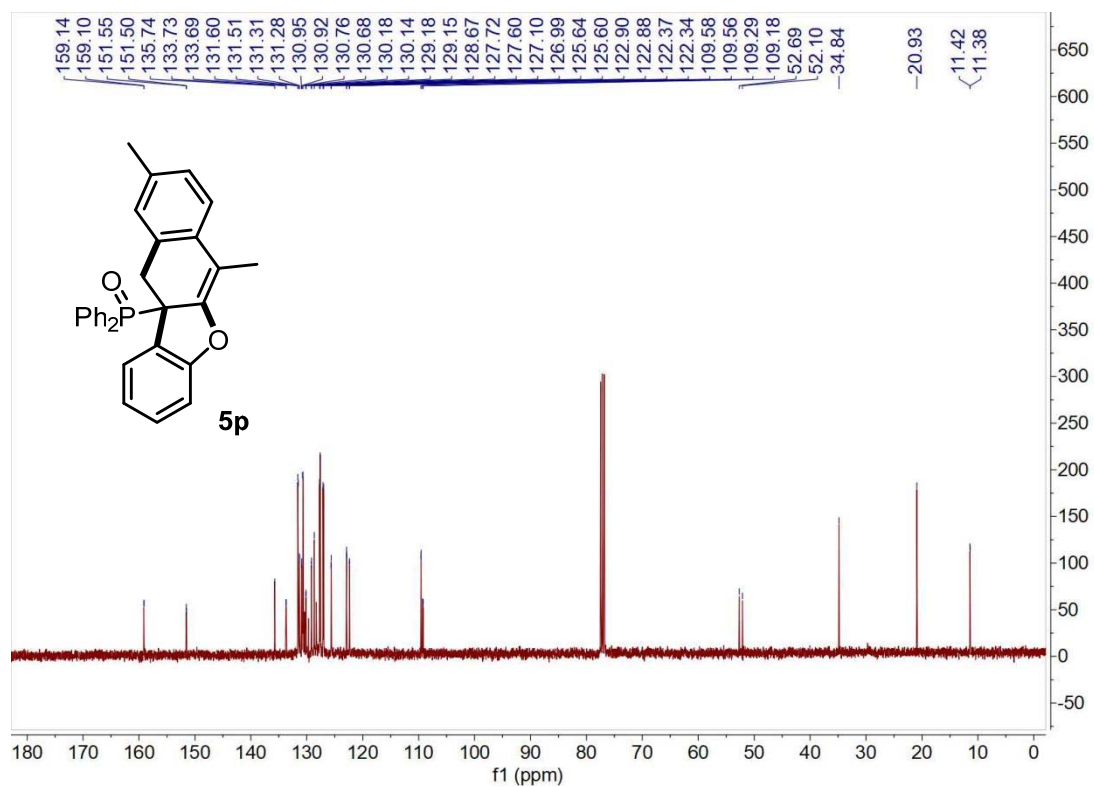
Spectrum from 04.wiff (sample 1) - Sample010, Experiment 1, +TOF MS (80 - 1000) from 1.433 to 1.477 min



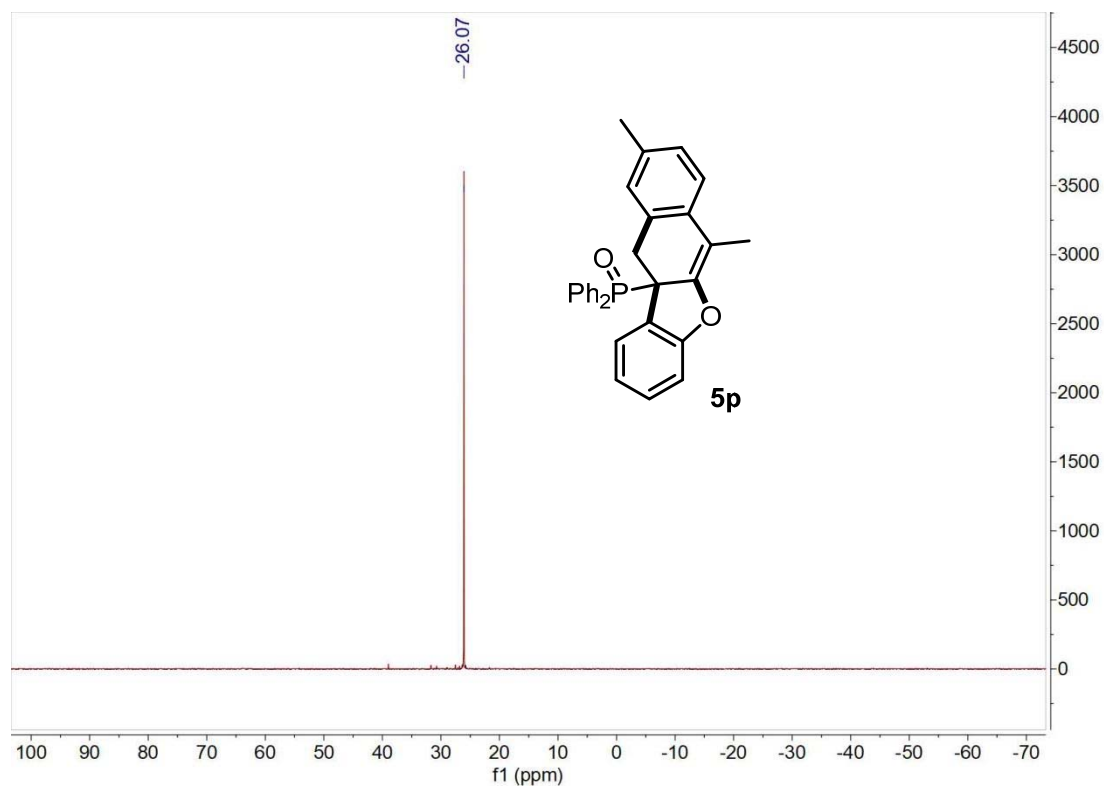
HRMS of **5o-2**



1H -NMR (400 MHz, $CDCl_3$) of **5p**

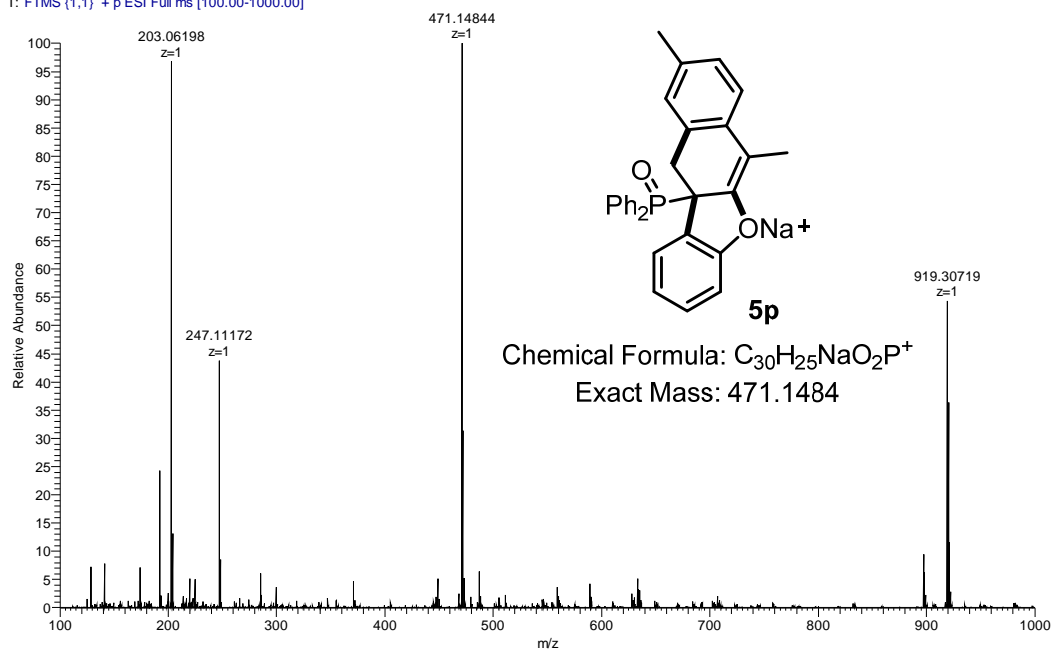


^{13}C -NMR (101 MHz, CDCl_3) of **5p**

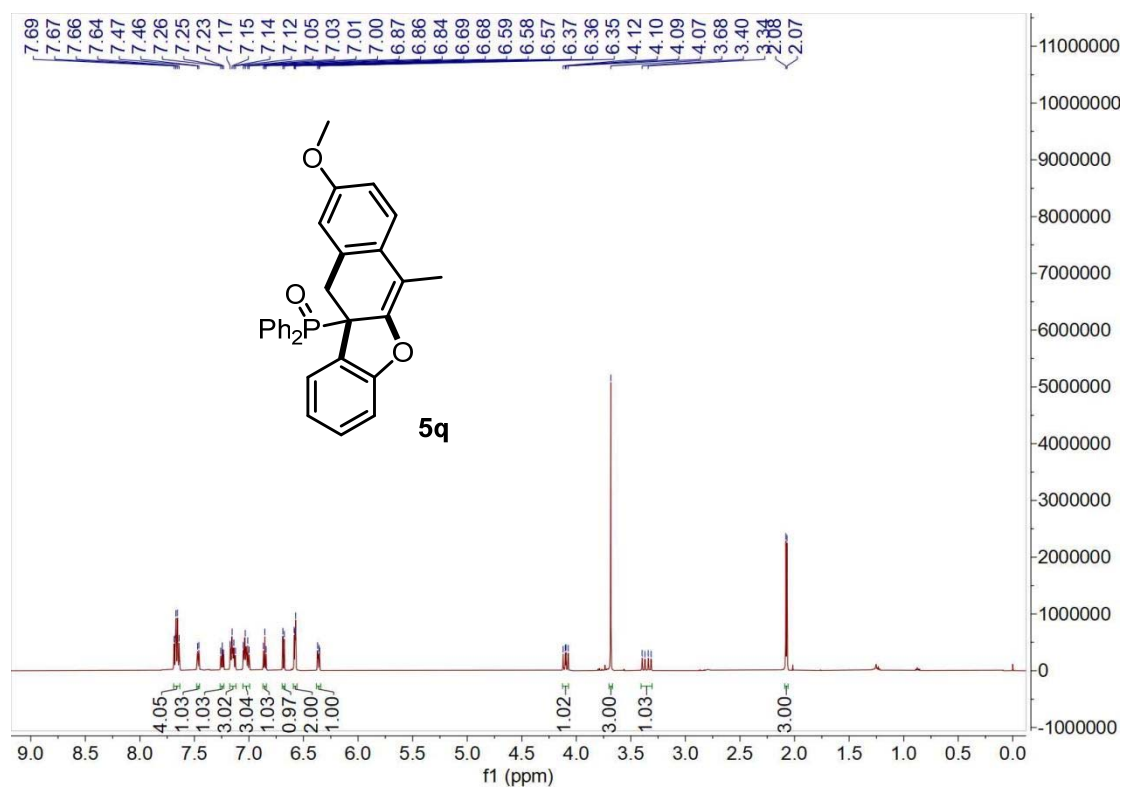


^{31}P -NMR (162 MHz, CDCl_3) of **5p**

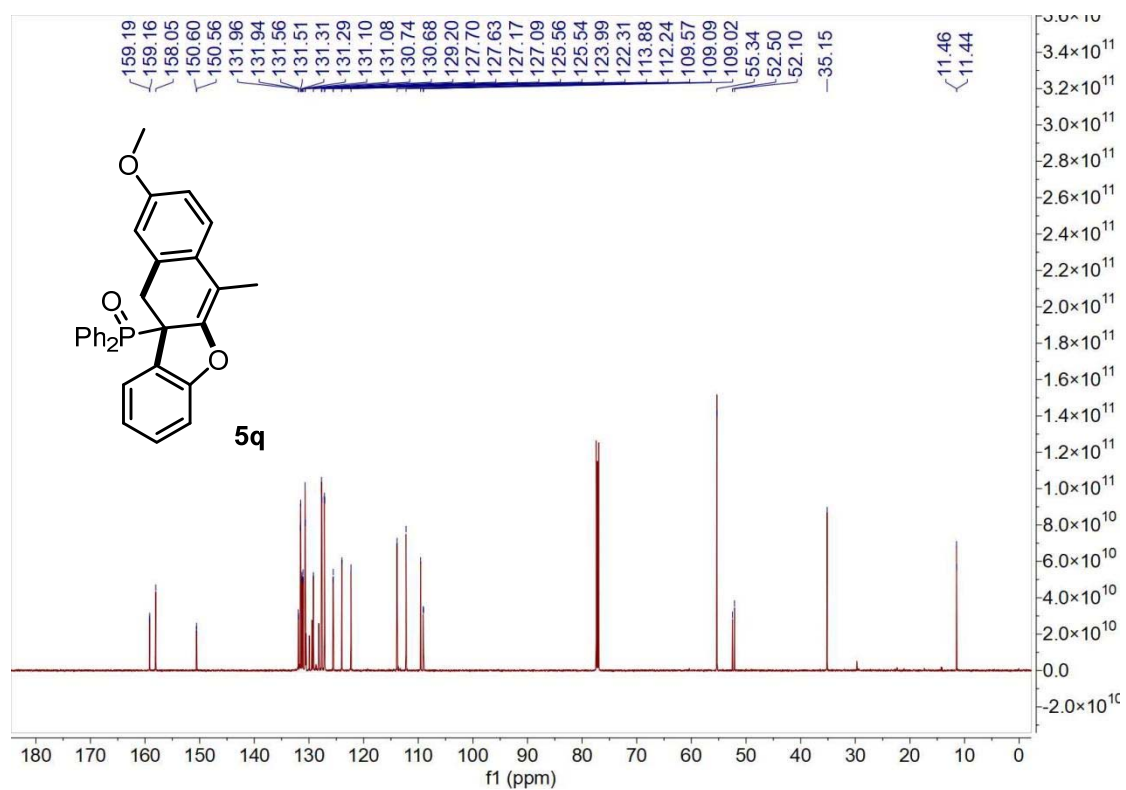
20210118-27 #31 RT: 0.39 AV: 1 NL: 1.77E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



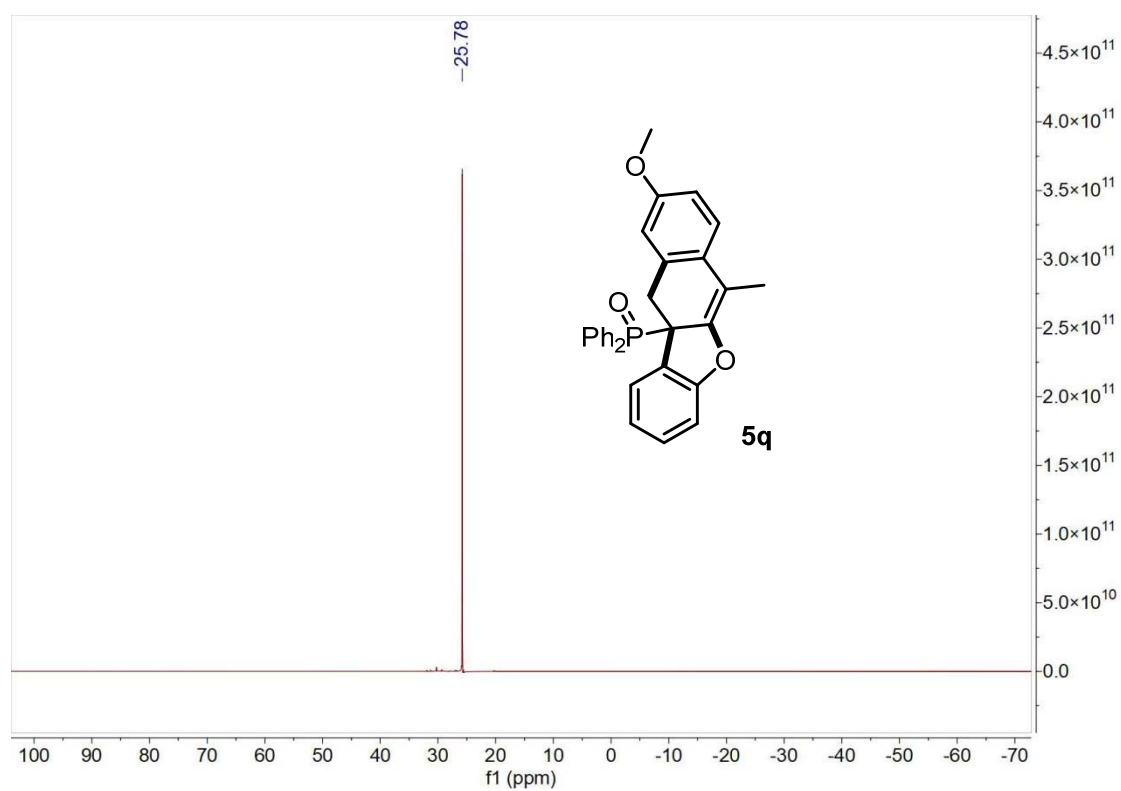
HRMS of **5p**



1H -NMR (600 MHz, $CDCl_3$) of **5q**

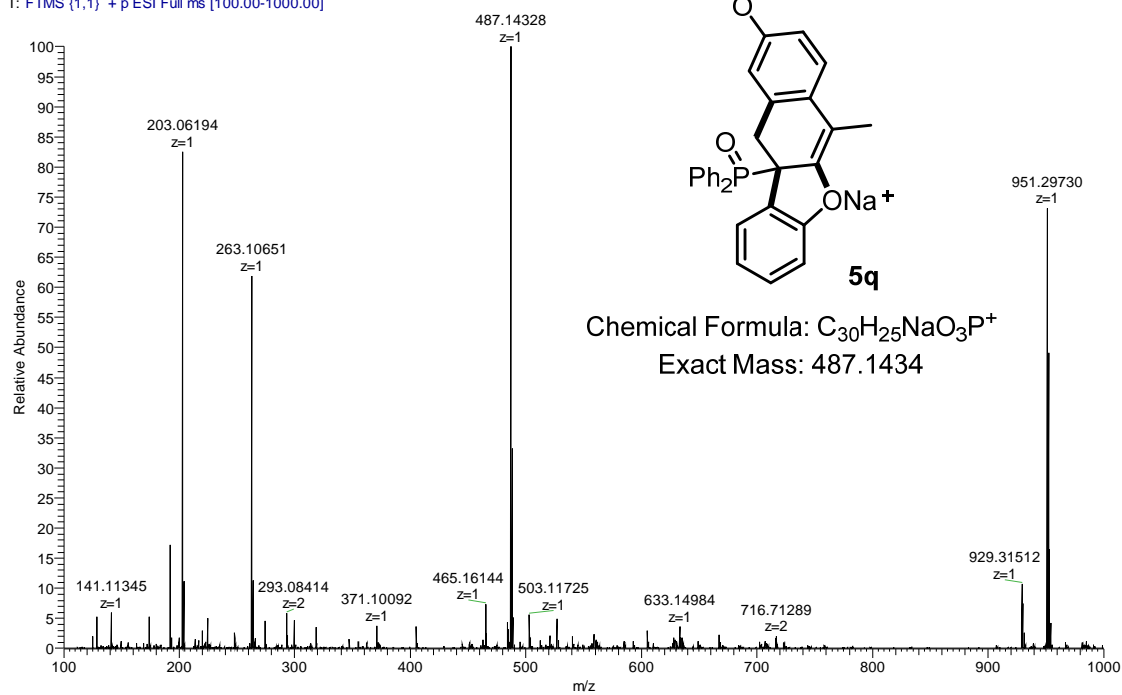


^{13}C -NMR (151 MHz, CDCl_3) of **5q**

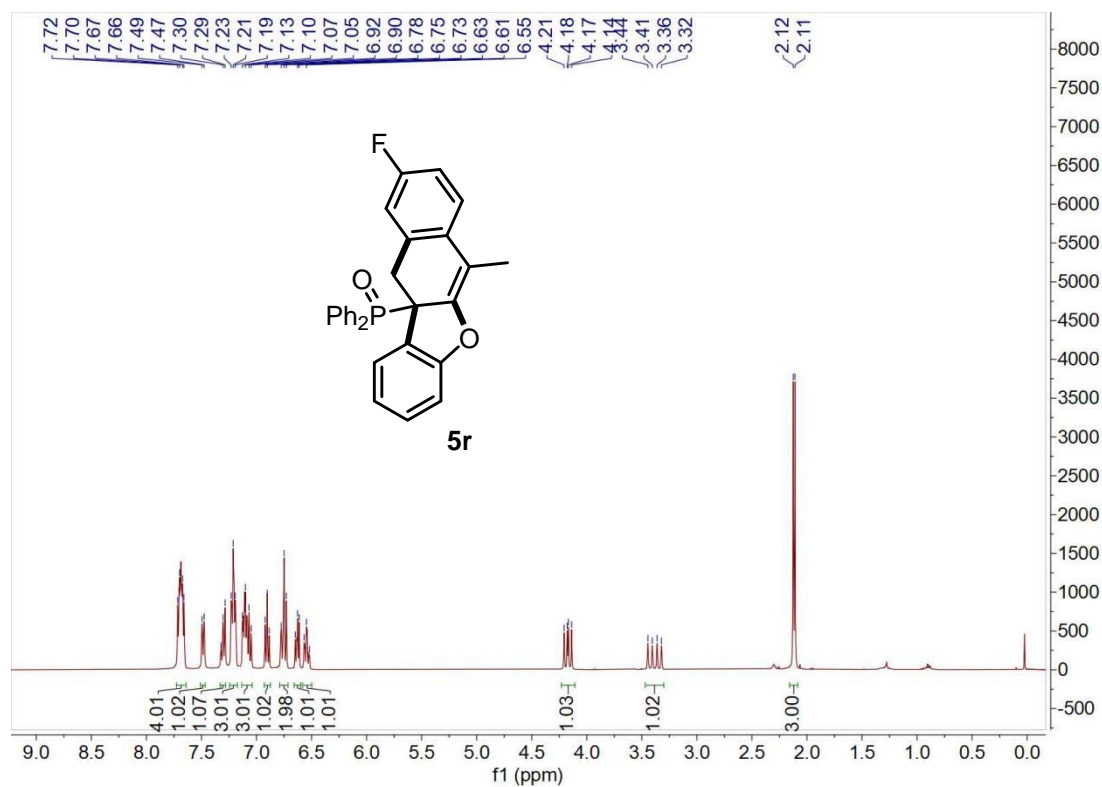


^{31}P -NMR (243 MHz, CDCl_3) of **5q**

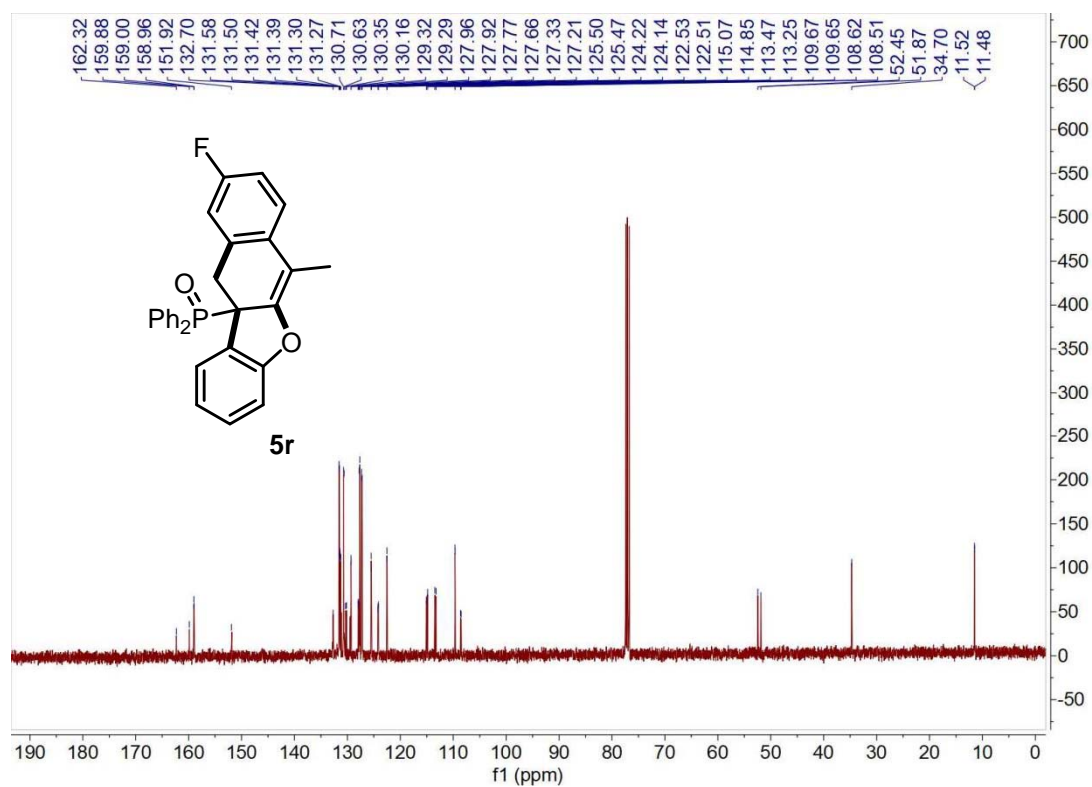
20210118-29 #25 RT: 0.31 AV: 1 NL: 2.15E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



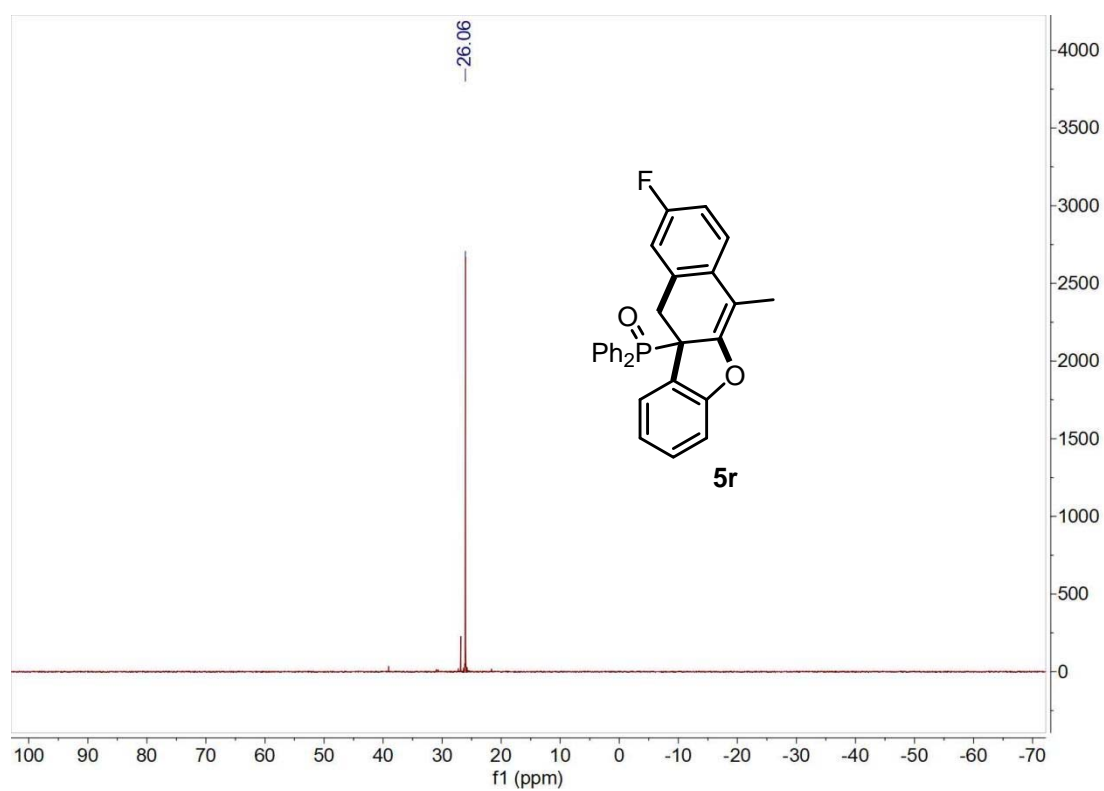
HRMS of **5q**



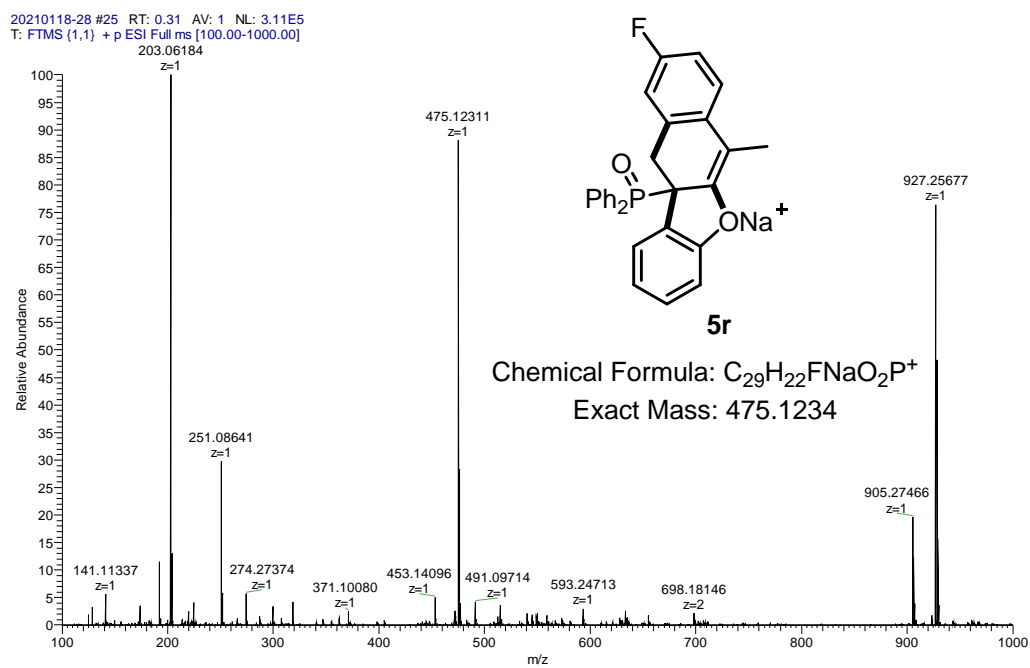
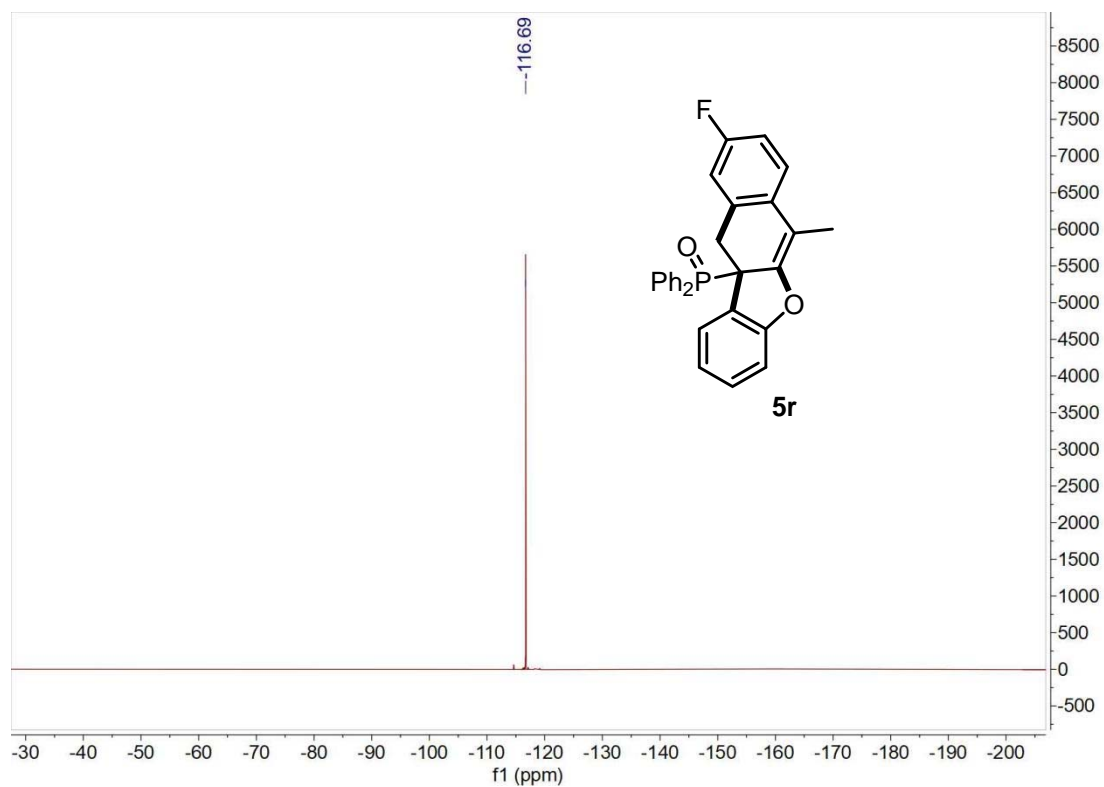
1H -NMR (400 MHz, $CDCl_3$) of **5r**



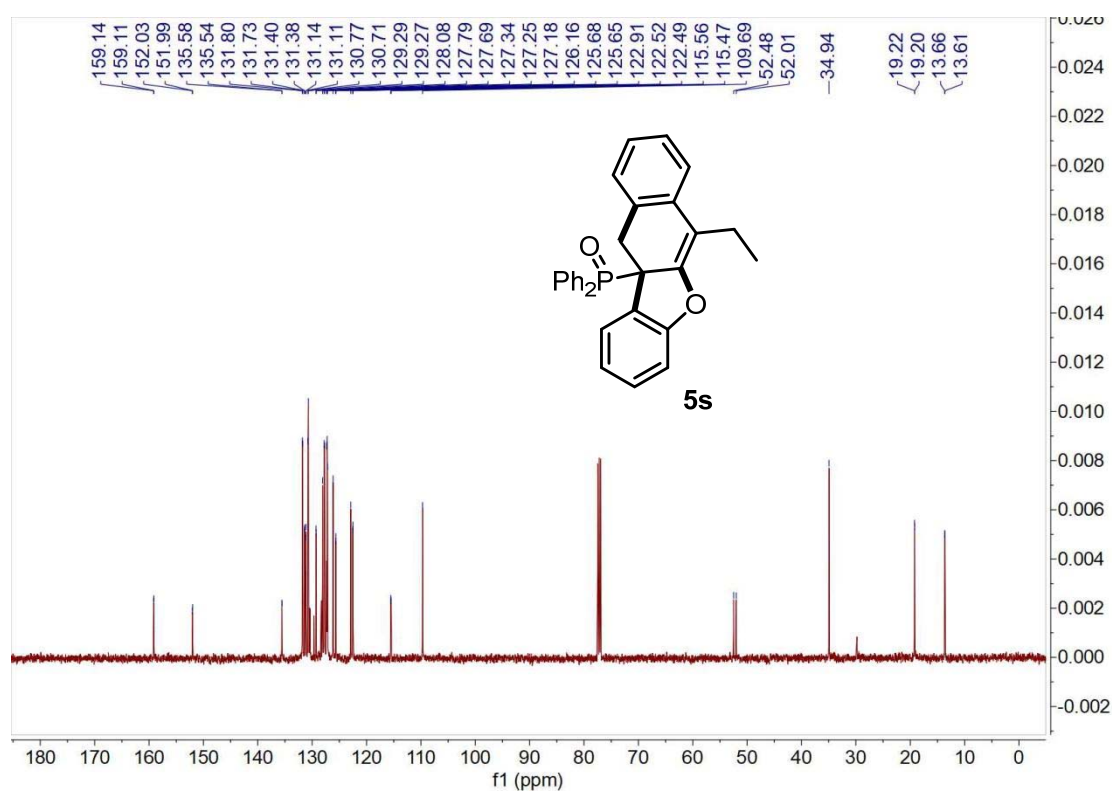
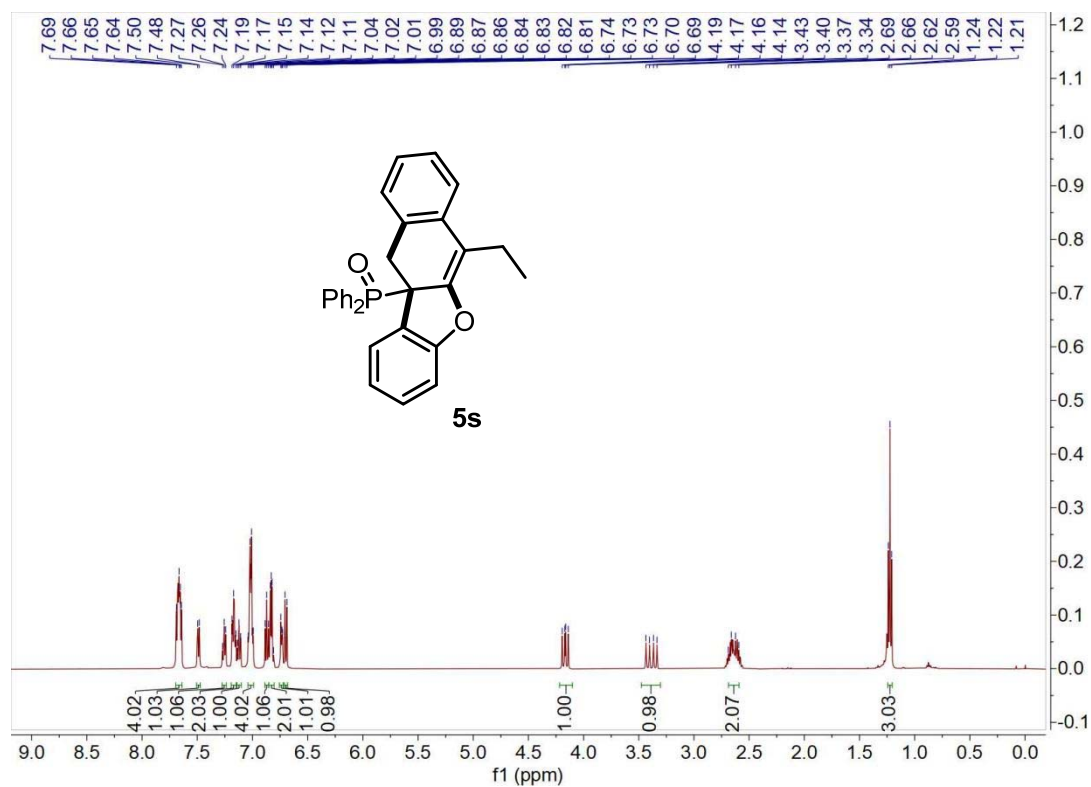
^{13}C -NMR (101 MHz, CDCl_3) of **5r**

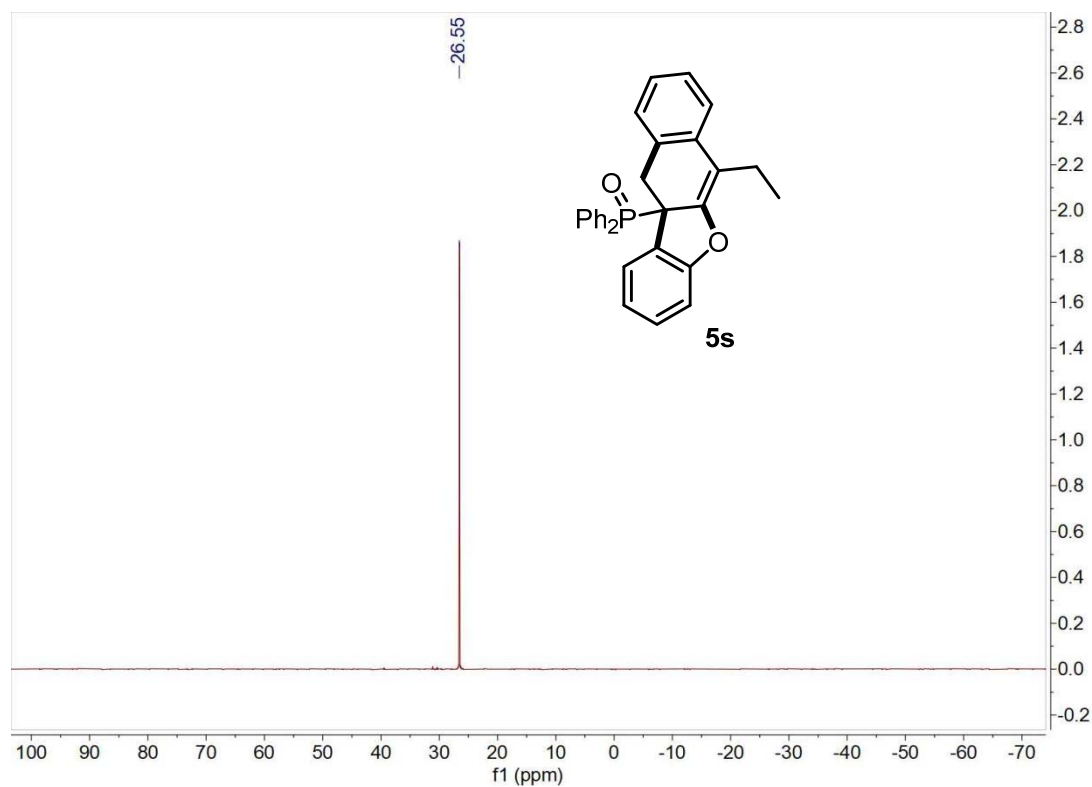


^{31}P -NMR (162 MHz, CDCl_3) of **5r**

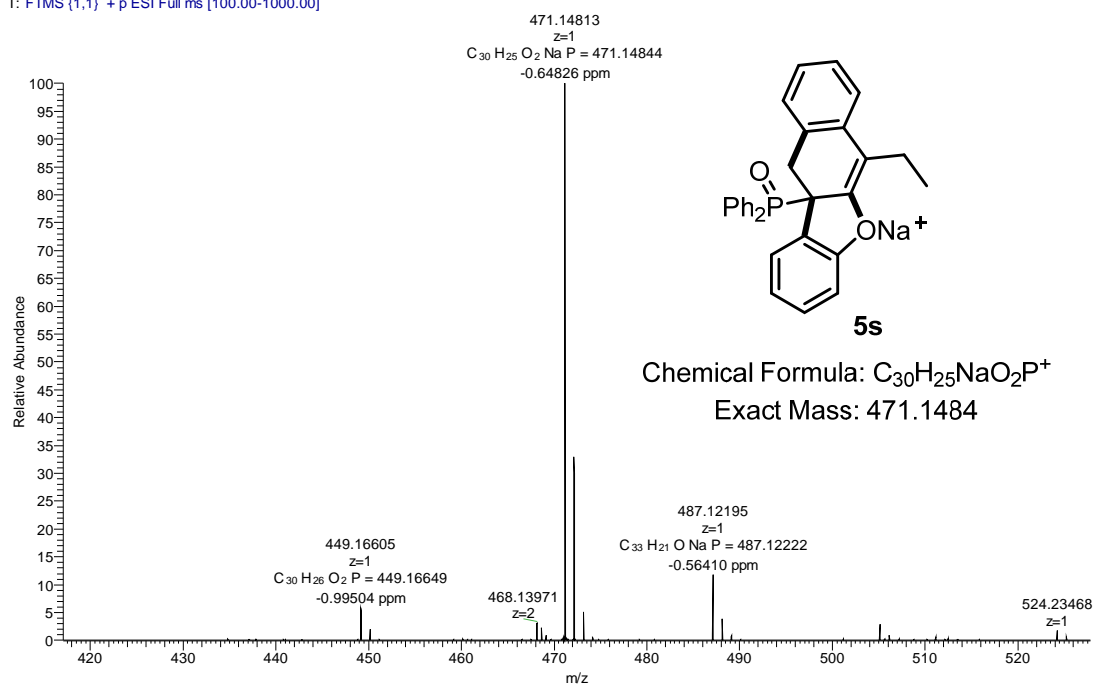


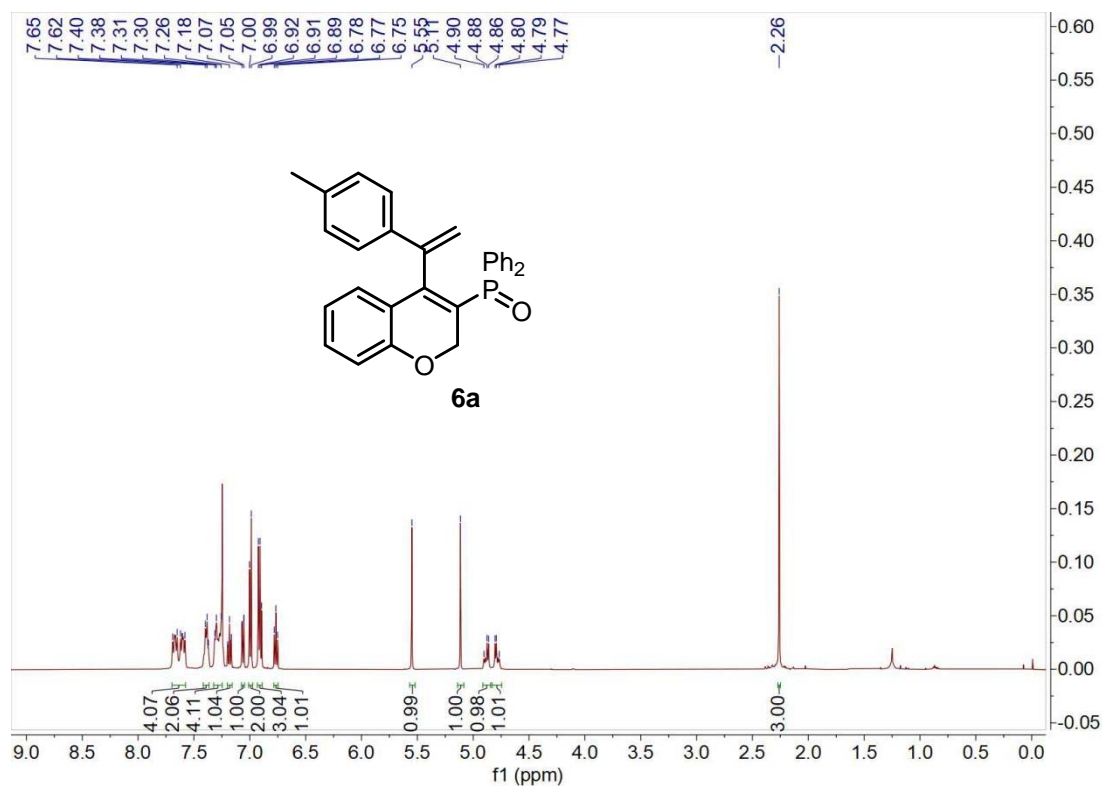
HRMS of **5r**



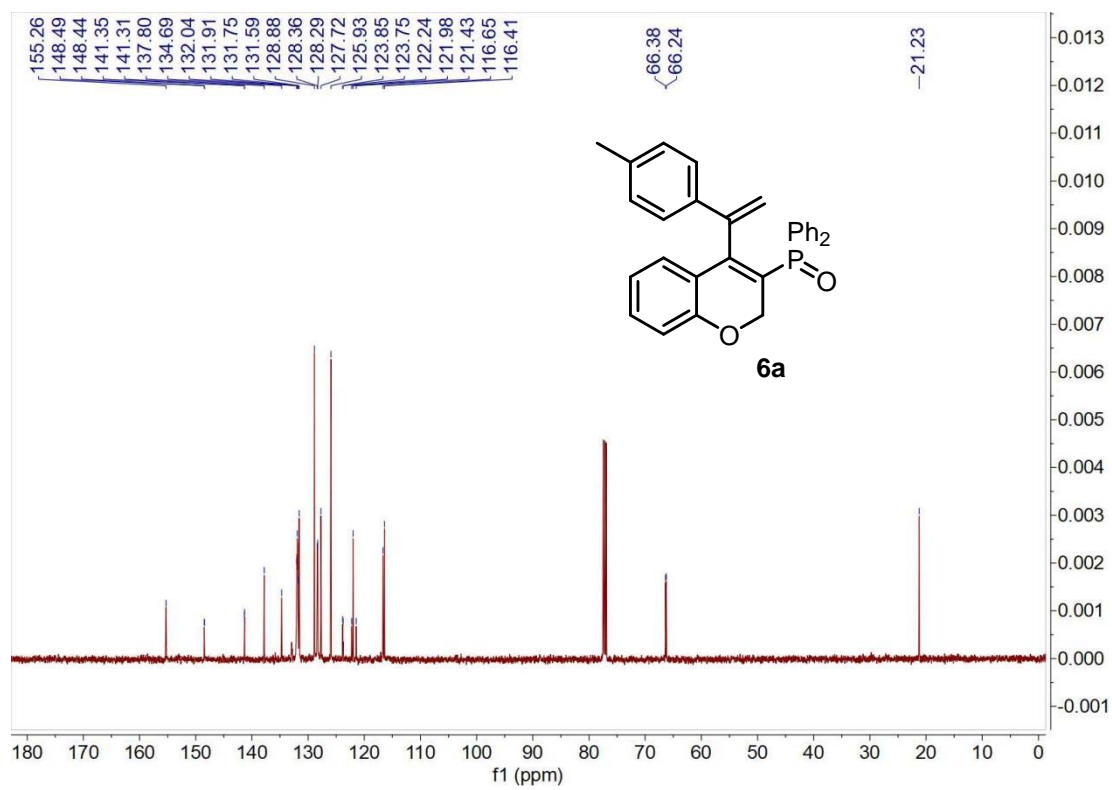


20210719-23 #35 RT: 0.41 AV: 1 NL: 9.37E5
 T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

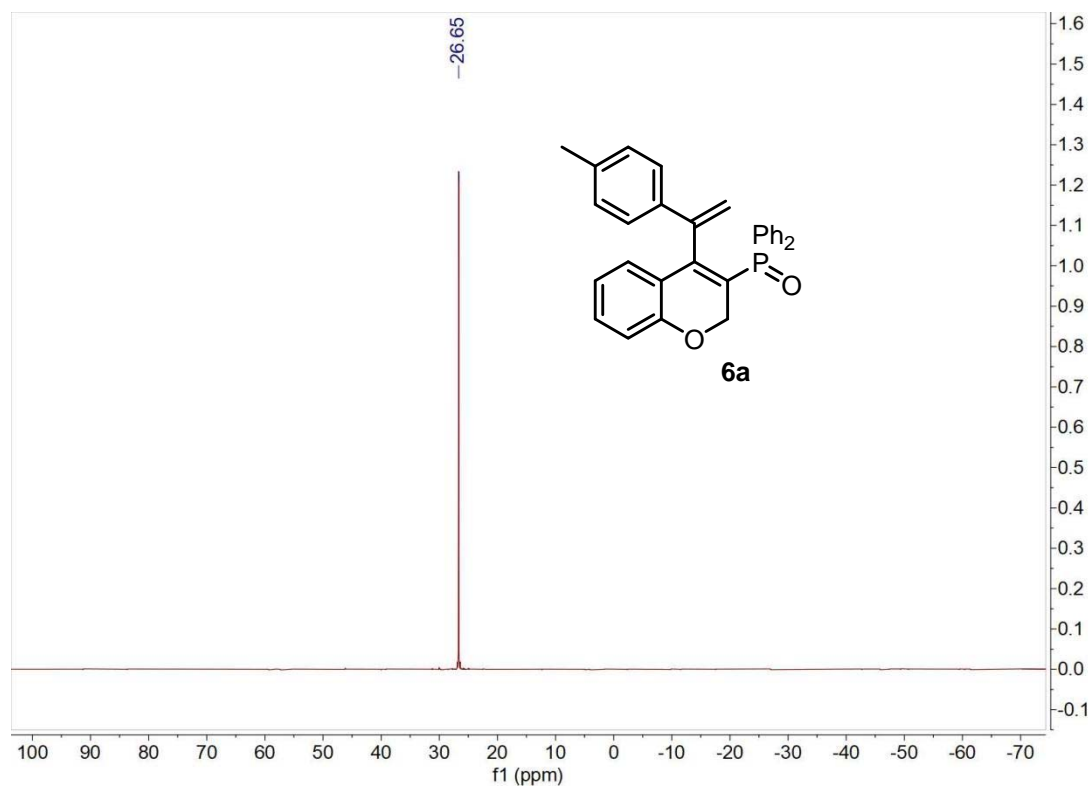




¹H-NMR (500 MHz, CDCl₃) of **6a**

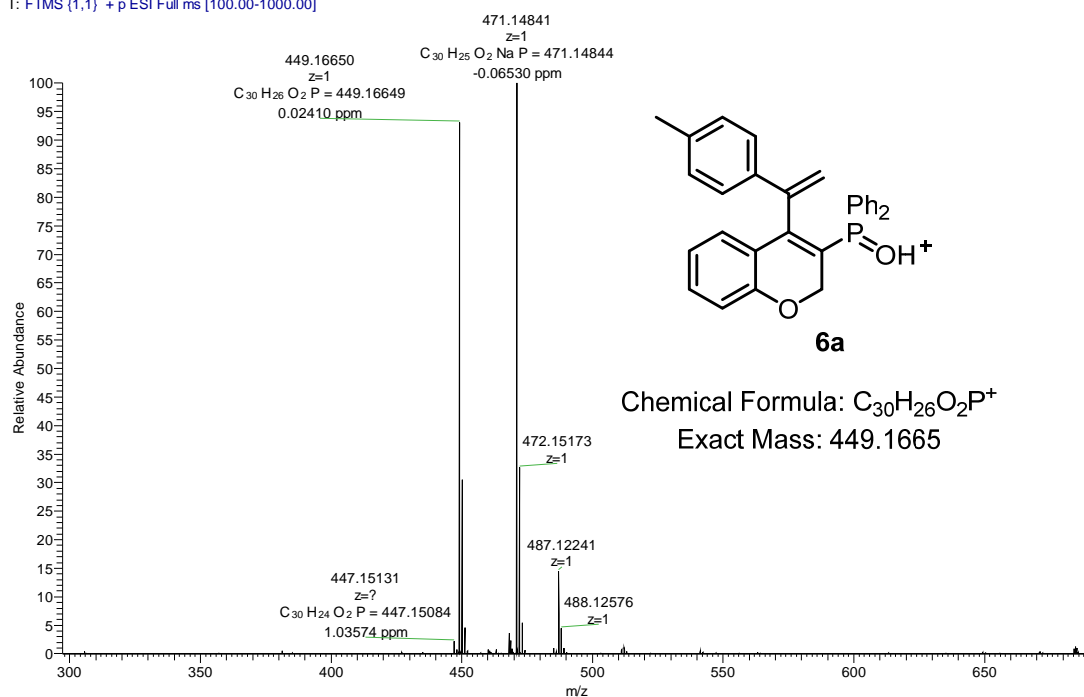


¹³C-NMR (126 MHz, CDCl₃) of **6a**

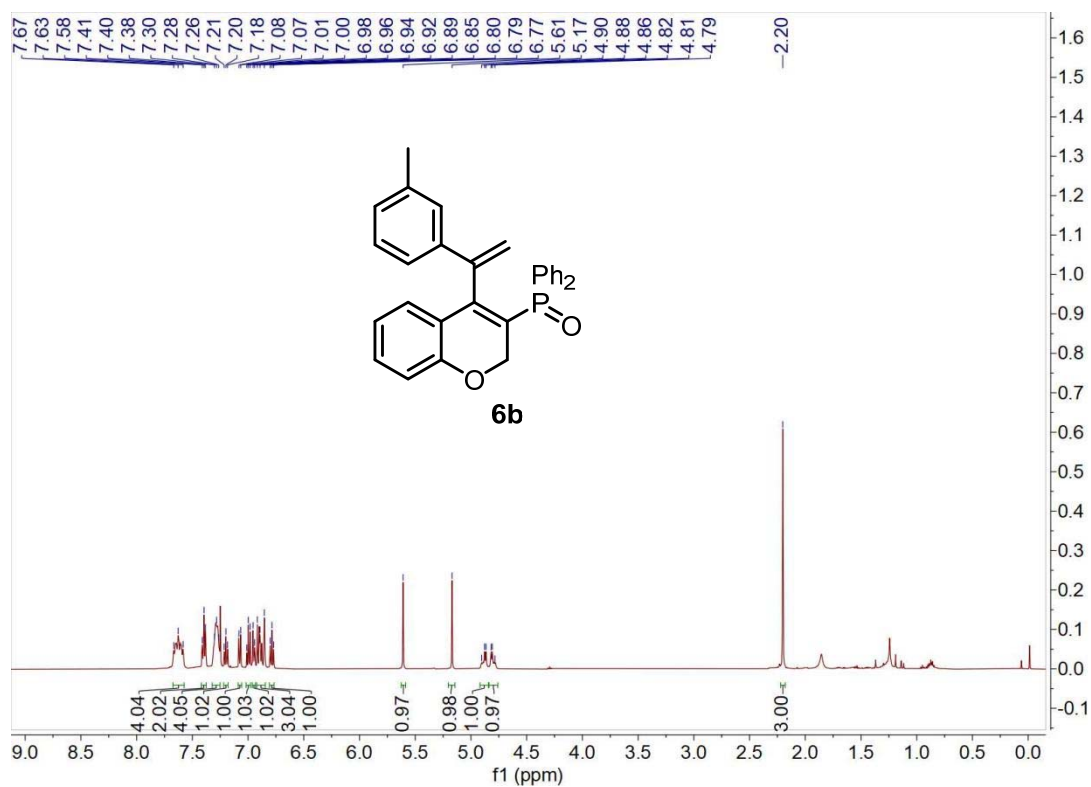


^{31}P -NMR (202 MHz, CDCl_3) of **6a**

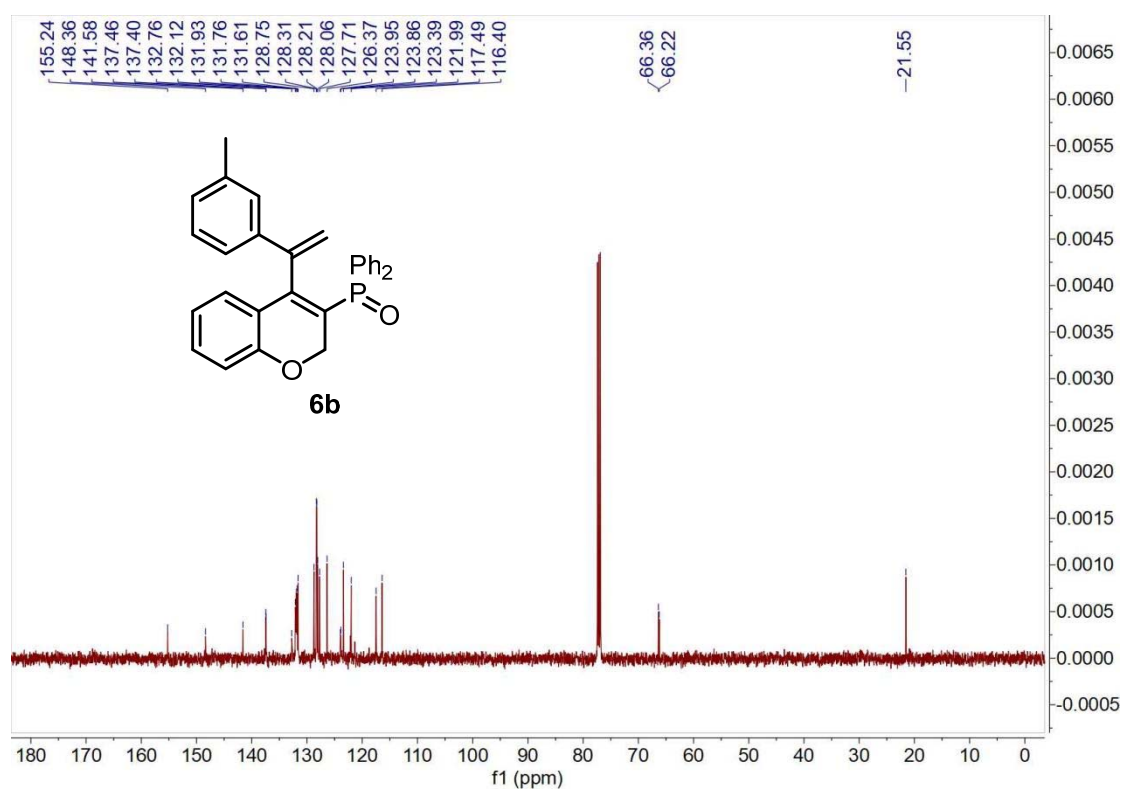
26 #39 RT: 0.46 AV: 1 NL: 4.86E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



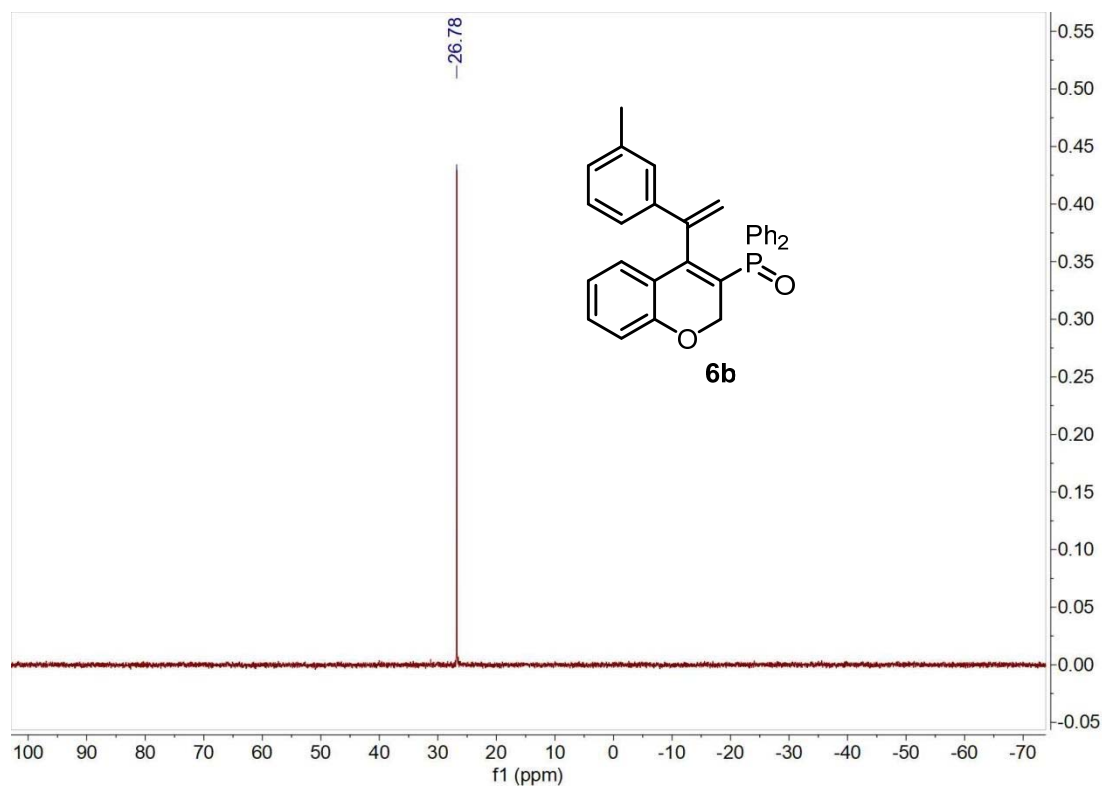
HRMS of **6a**



¹H-NMR (500 MHz, CDCl₃) of **6b**

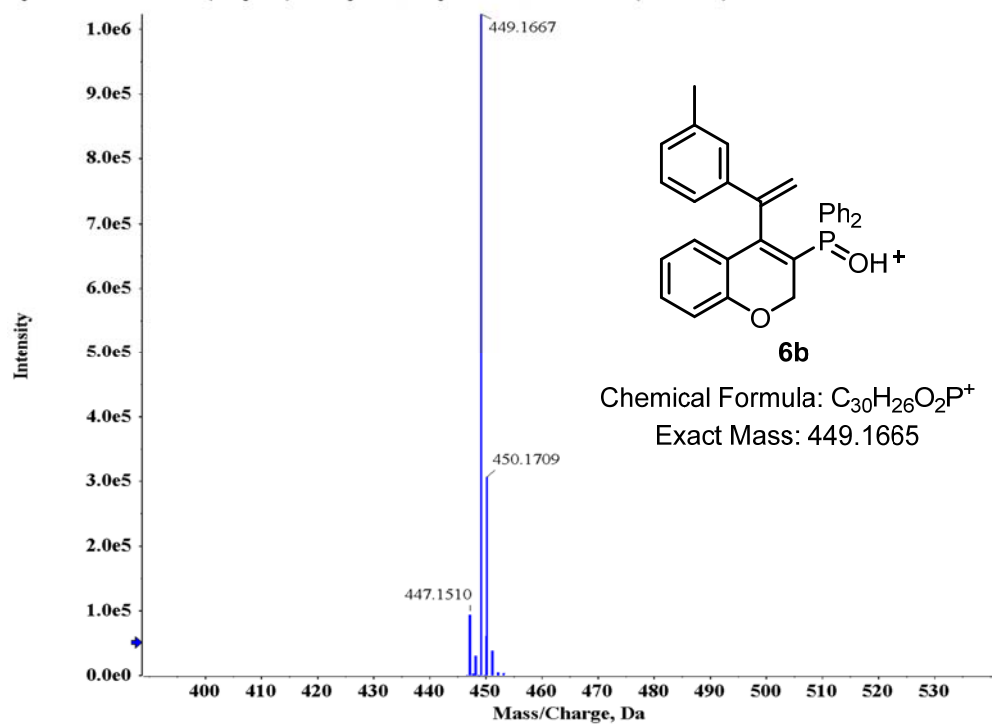


¹³C-NMR (126 MHz, CDCl₃) of **6b**

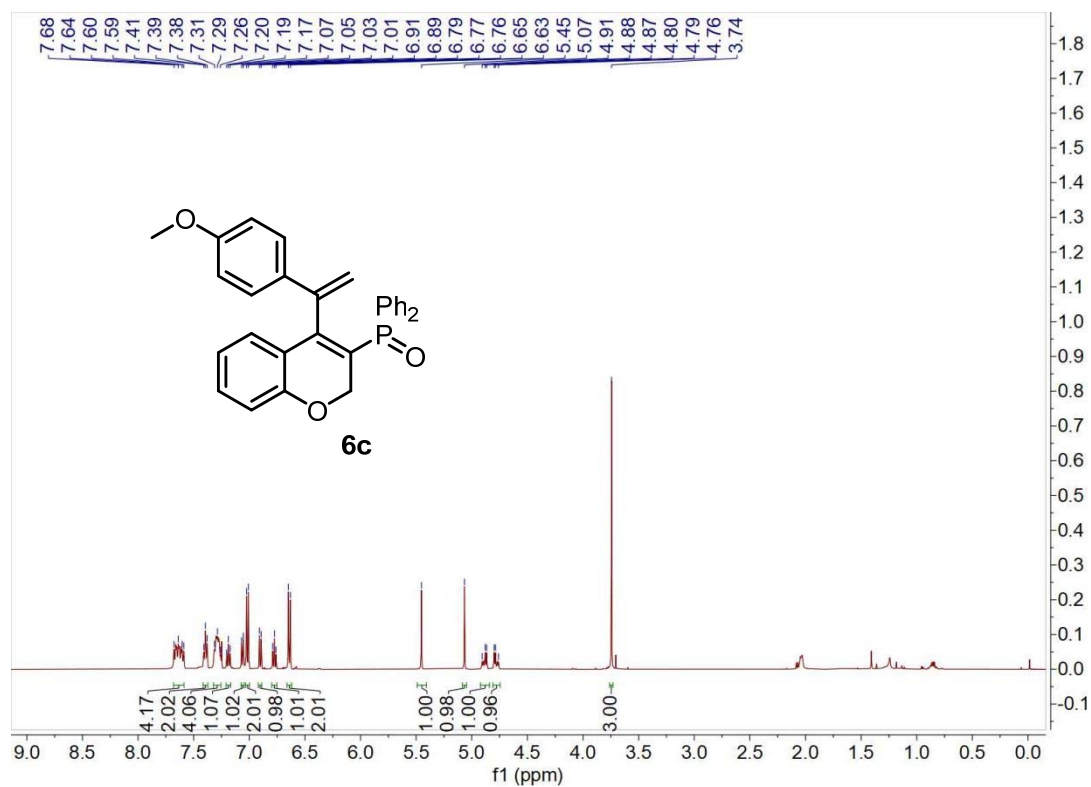


^{31}P -NMR (202 MHz, CDCl_3) of **6b**

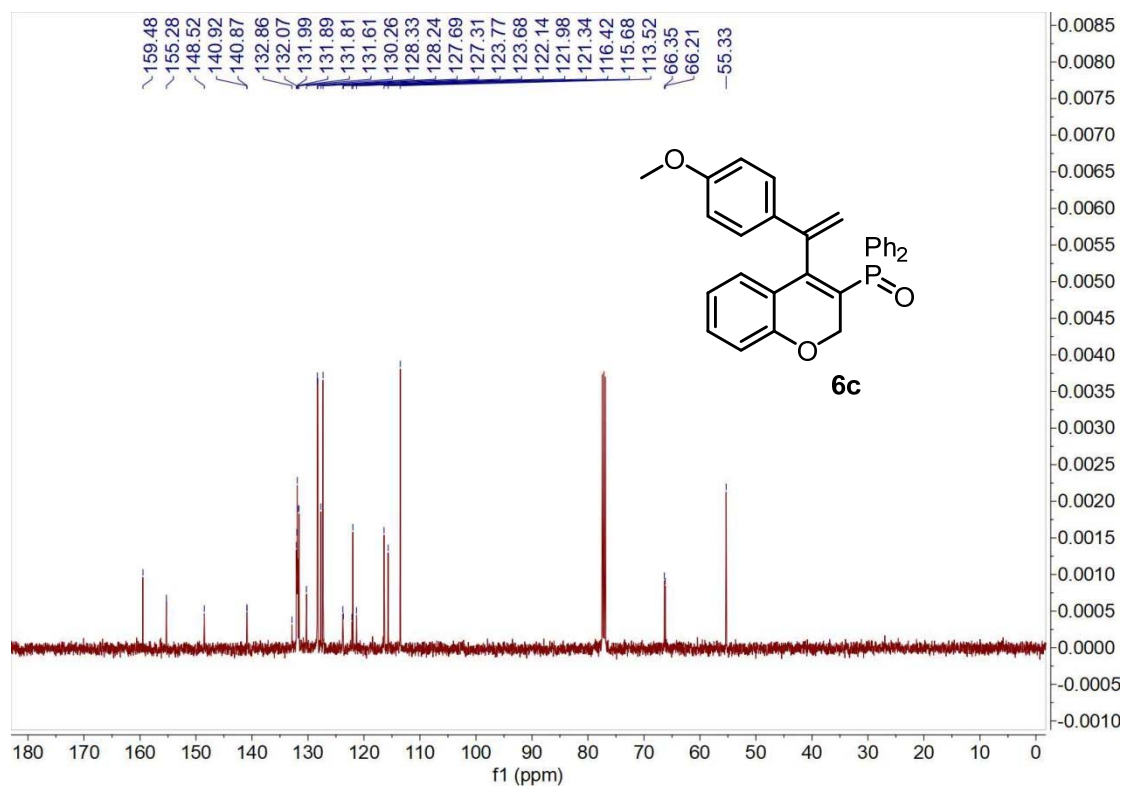
Spectrum from 07.wiff (sample 1) - Sample013, Experiment 1, +TOF MS (80 - 1000) from 0.792 to 0.837 min



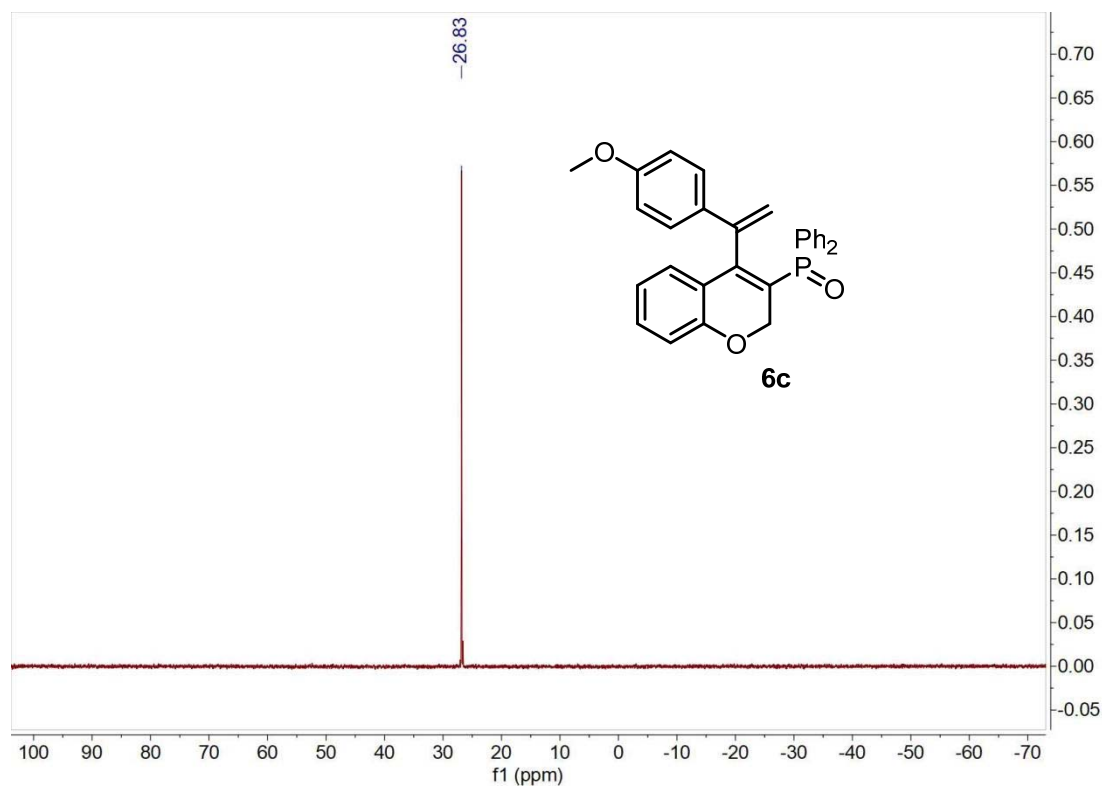
HRMS of **6b**



¹H-NMR (500 MHz, CDCl₃) of **6c**

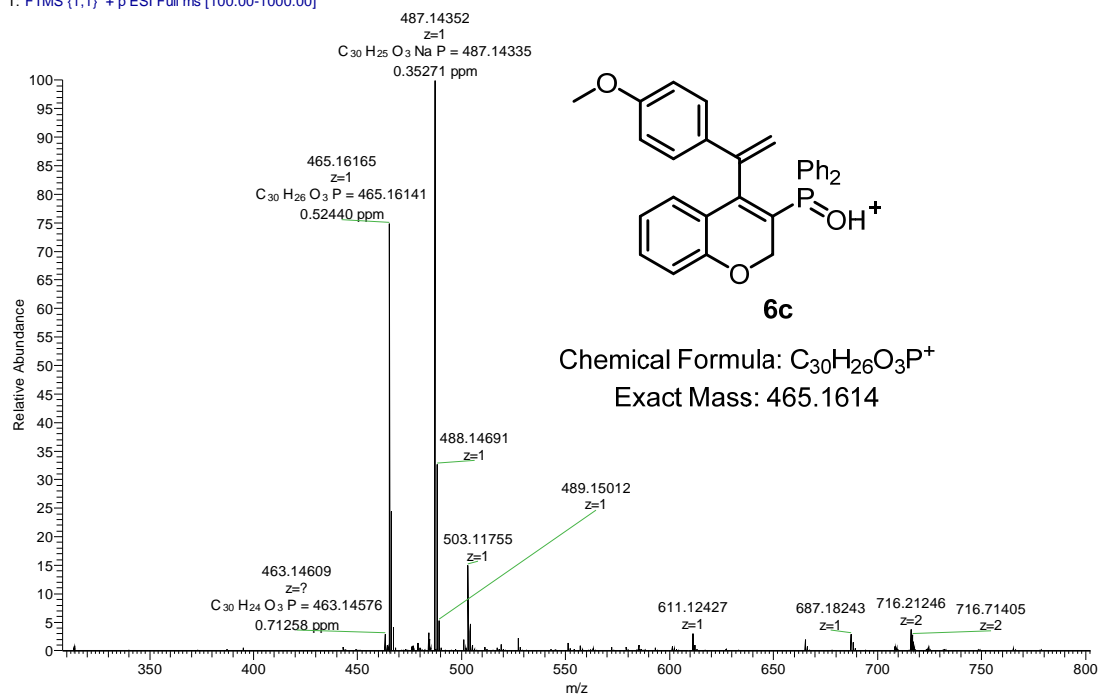


¹³C-NMR (126 MHz, CDCl₃) of **6c**

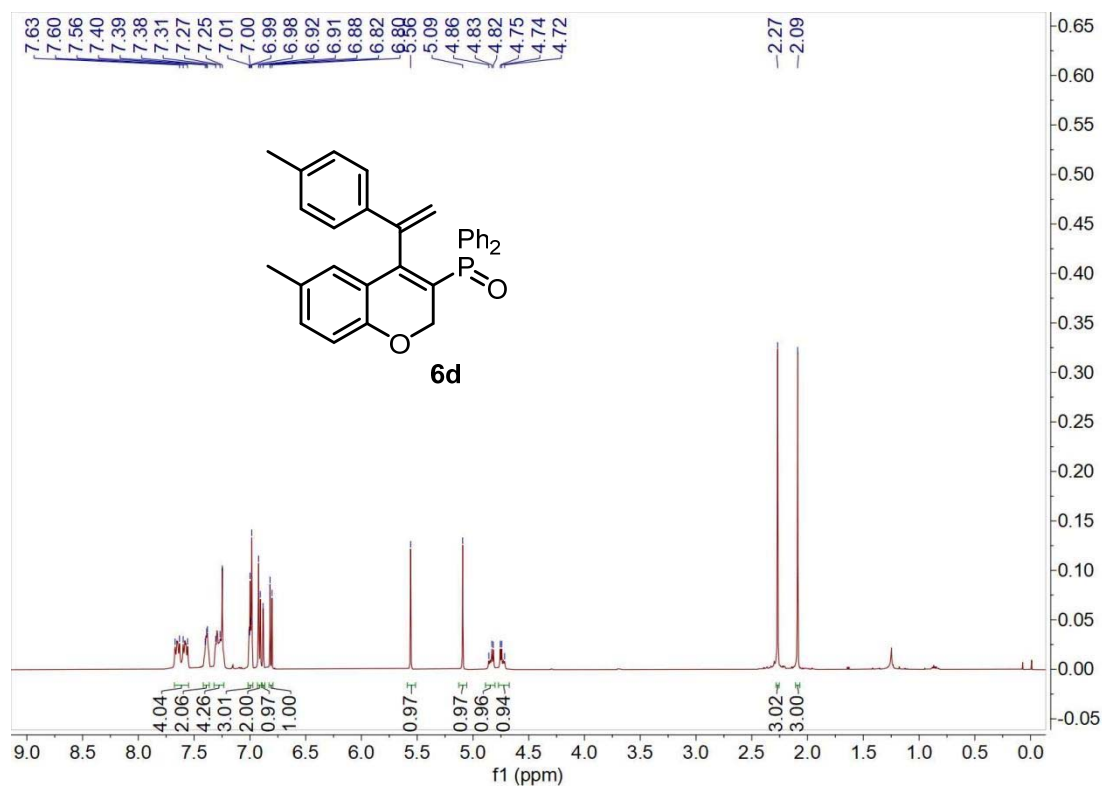


^{31}P -NMR (202 MHz, CDCl_3) of **6c**

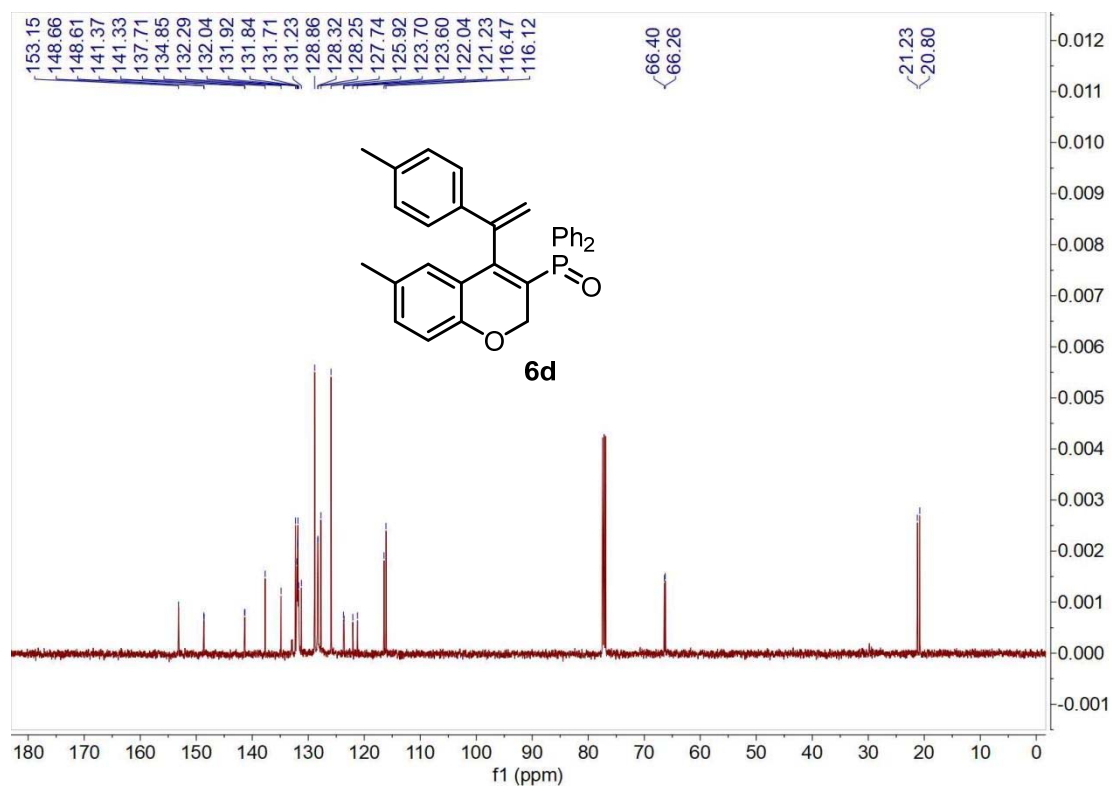
27 #27 RT: 0.33 AV: 1 NL: 1.68E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



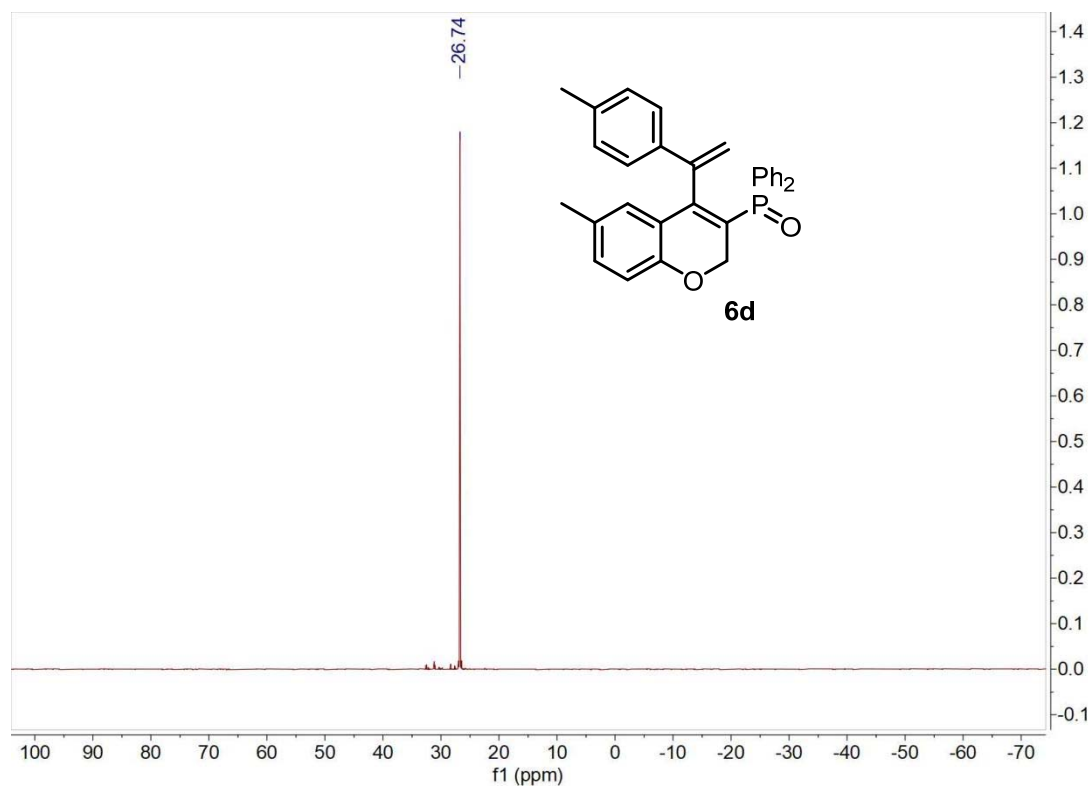
HRMS of **6c**



$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **6d**

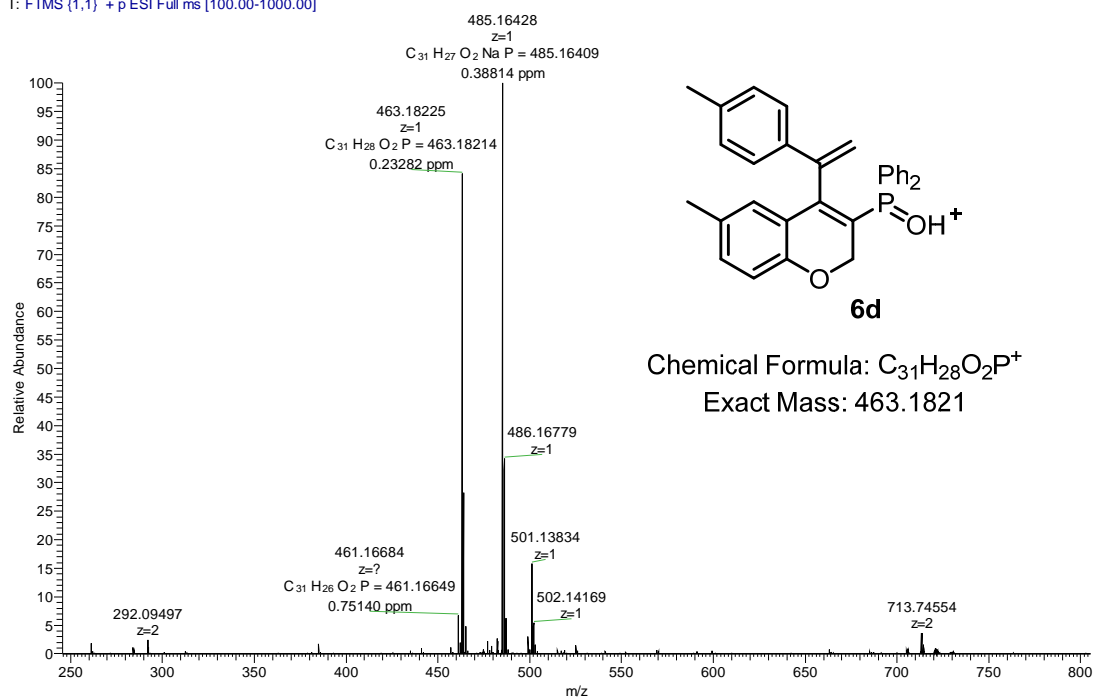


$^{13}\text{C-NMR}$ (126 MHz, CDCl_3) of **6d**

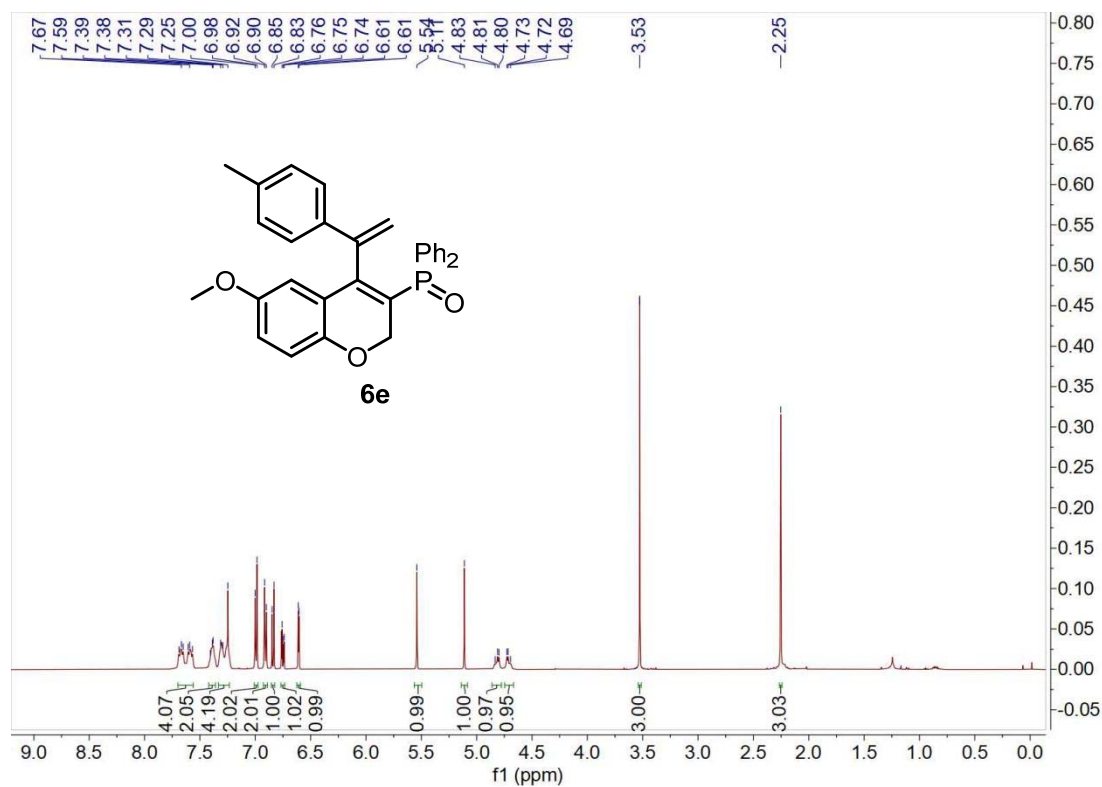


^{31}P -NMR (202 MHz, CDCl_3) of **6d**

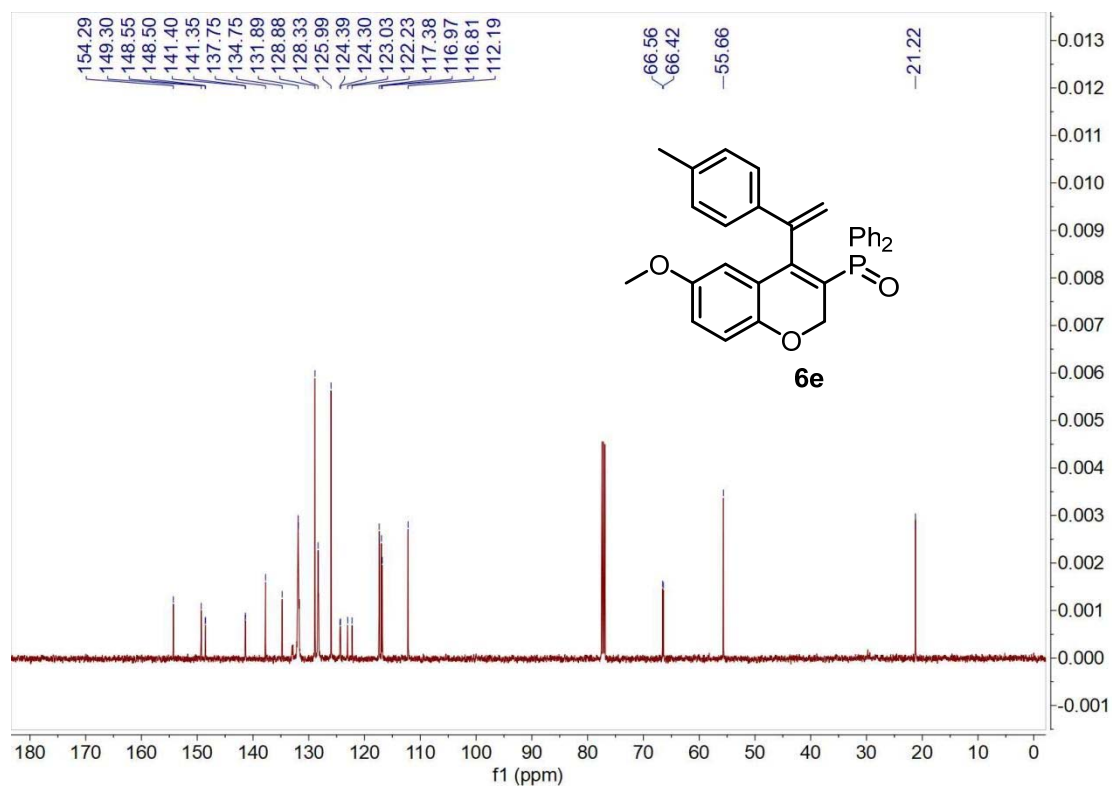
28 #27 RT: 0.32 AV: 1 NL: 1.58E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



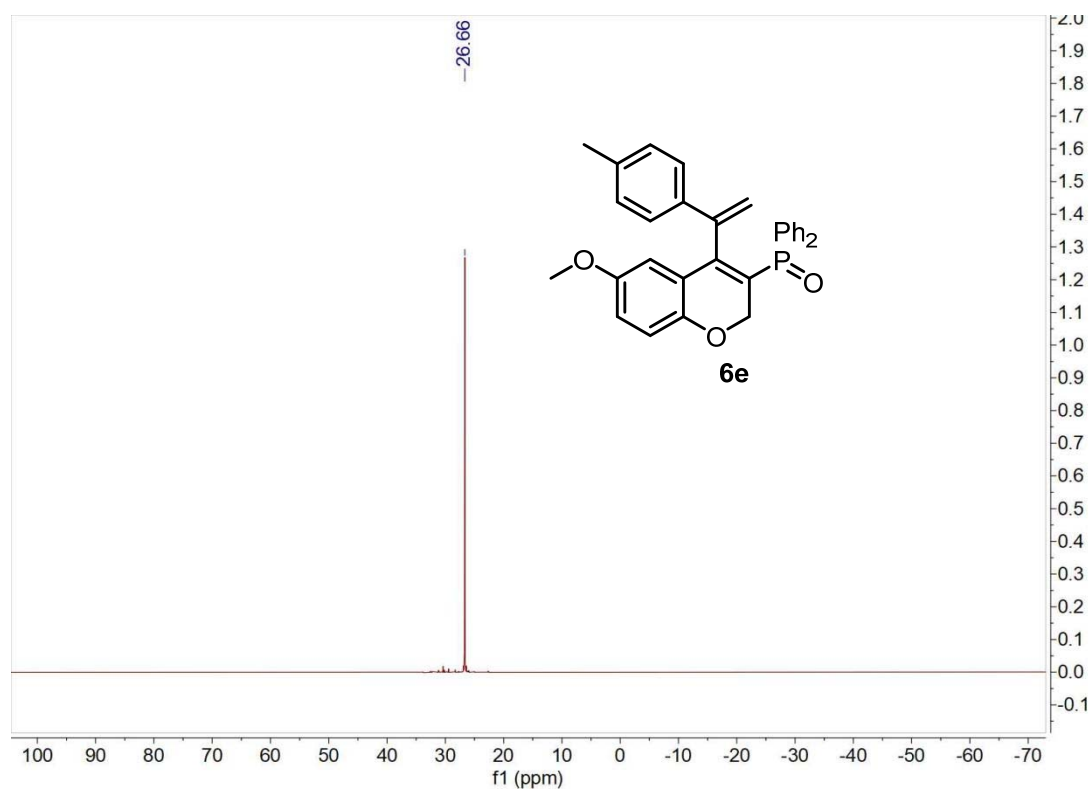
HRMS of **6d**



$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **6e**

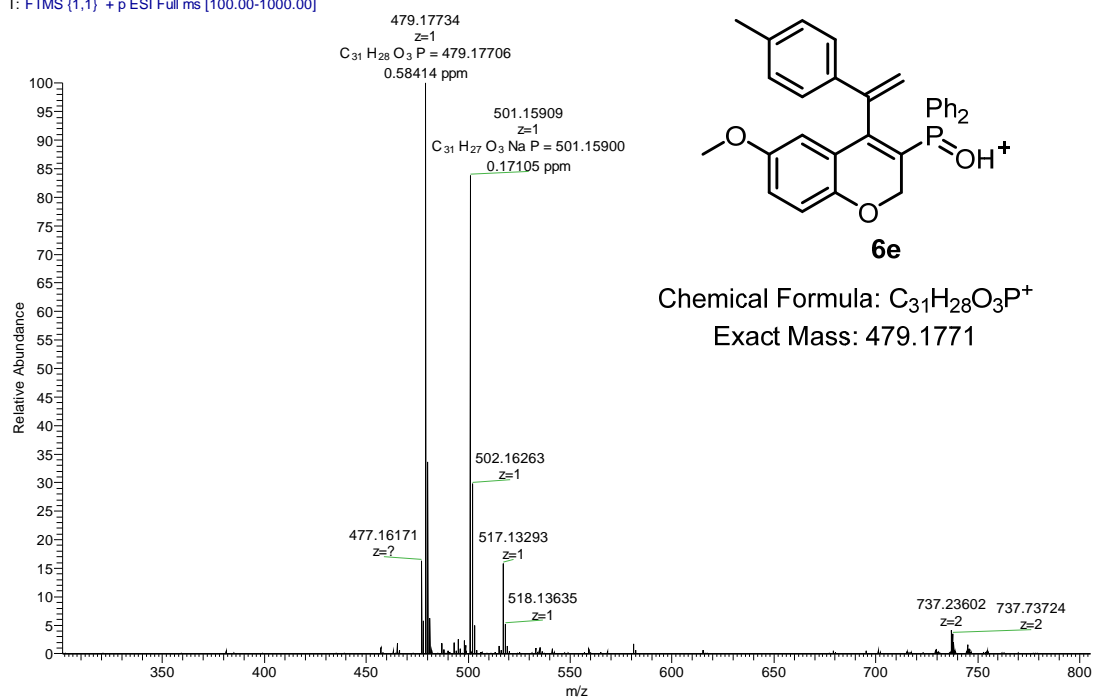


$^{13}\text{C-NMR}$ (126 MHz, CDCl_3) of **6e**

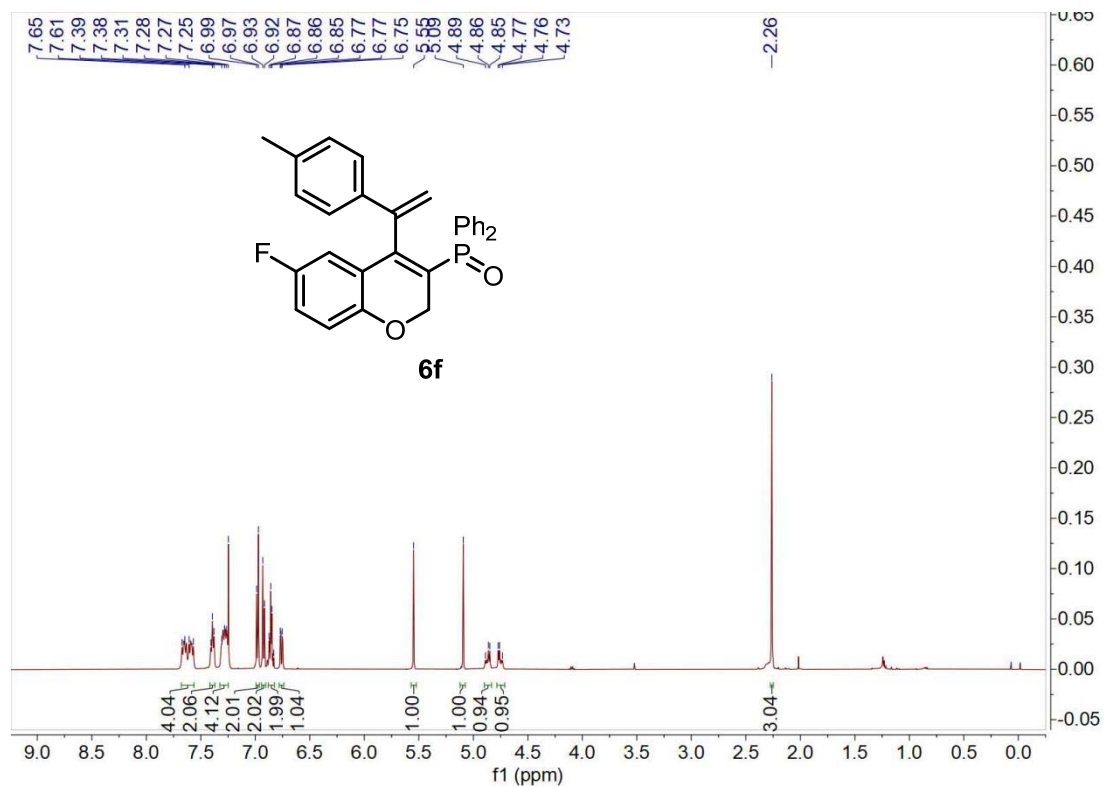


^{31}P -NMR (202 MHz, CDCl_3) of **6e**

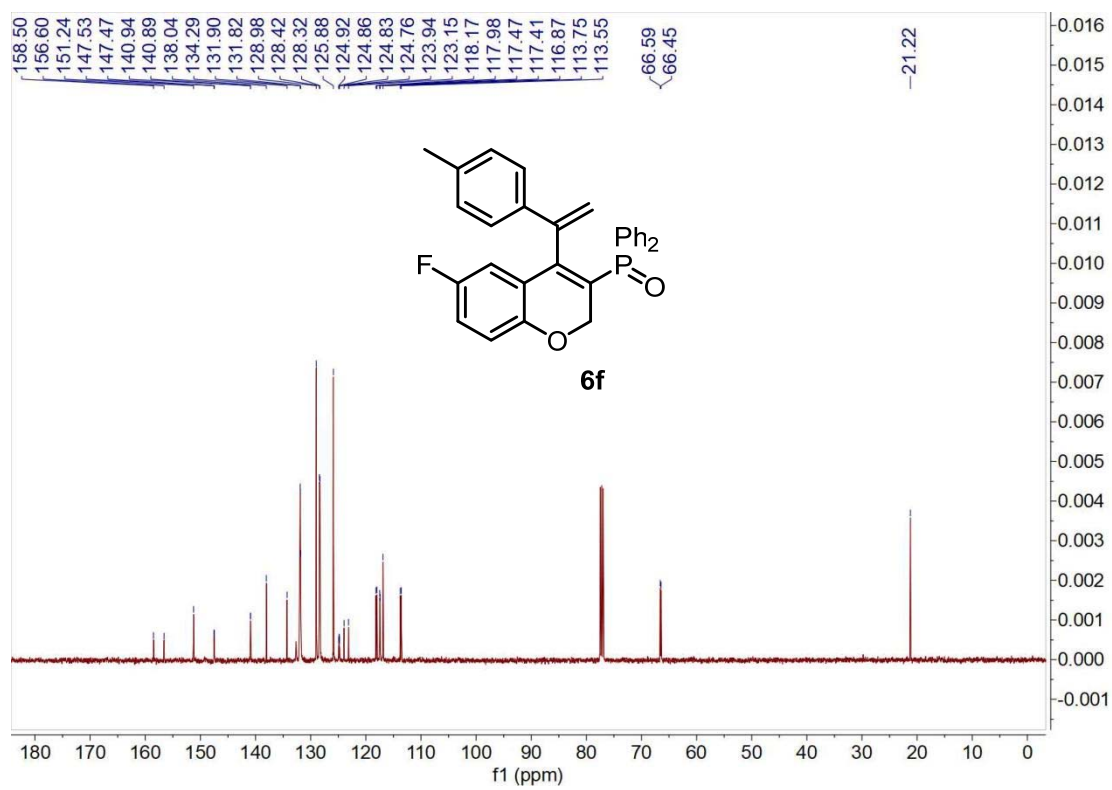
29 #9 RT: 0.11 AV: 1 NL: 1.71E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]



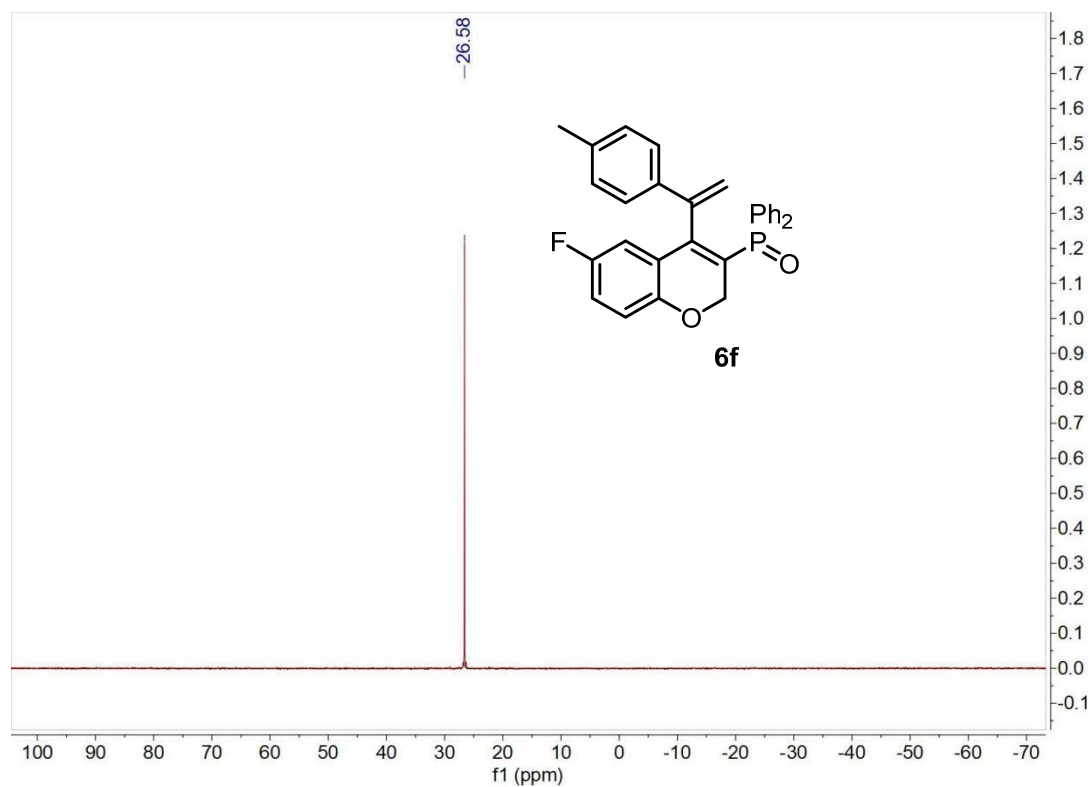
HRMS of **6e**



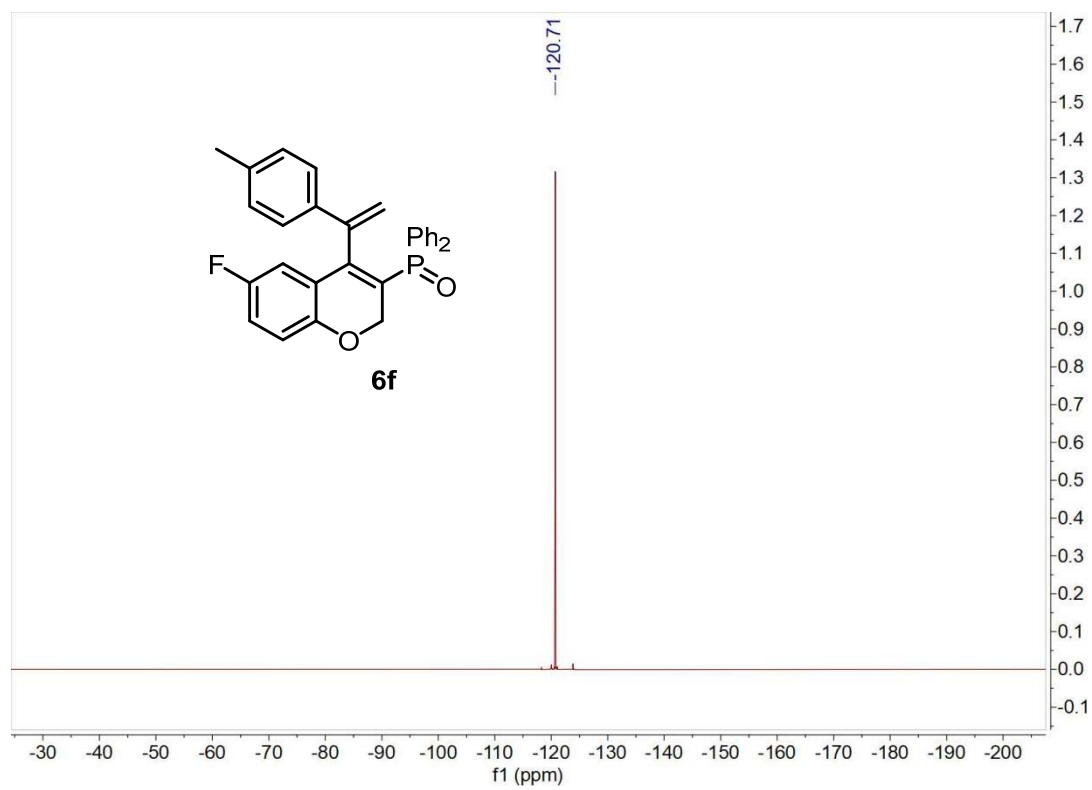
$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **6f**



$^{13}\text{C-NMR}$ (126 MHz, CDCl_3) of **6f**

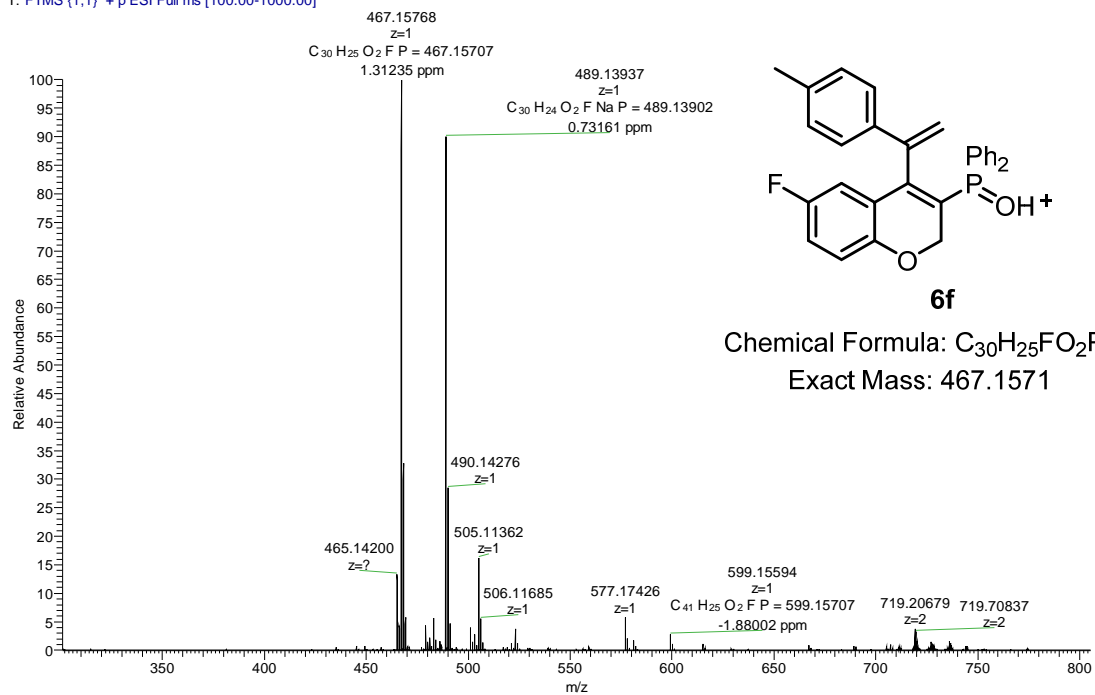


^{31}P -NMR (202 MHz, CDCl_3) of **6f**

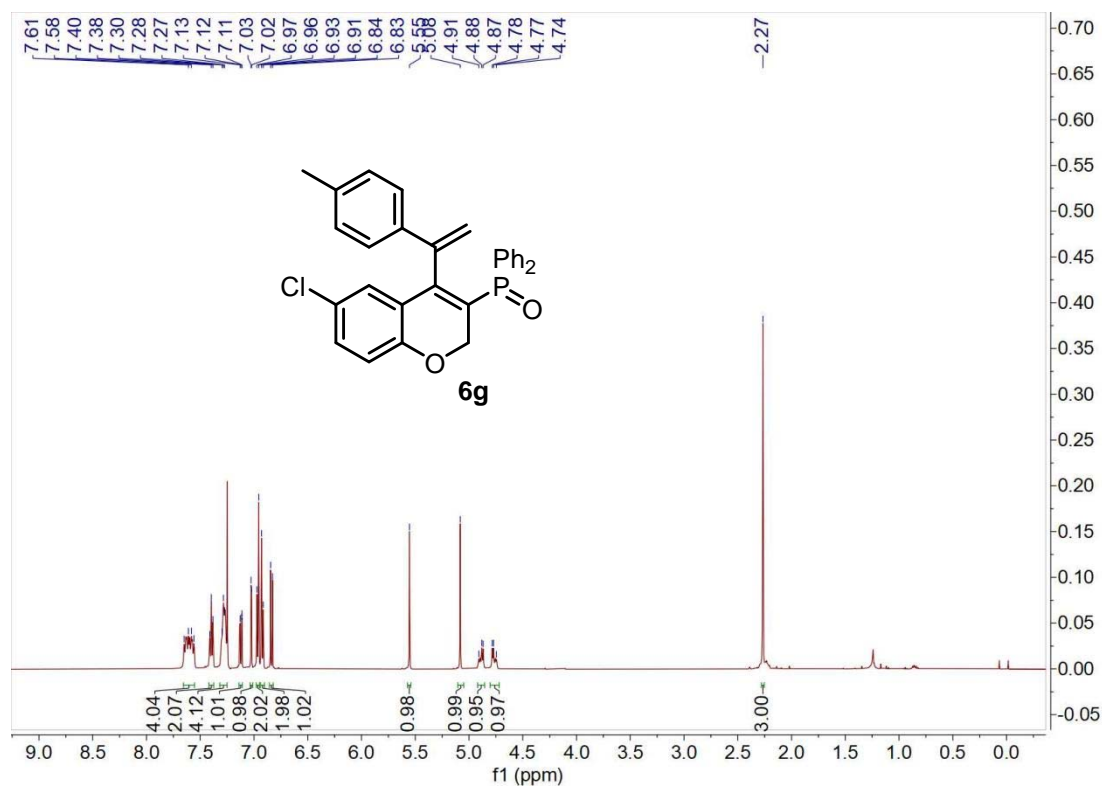


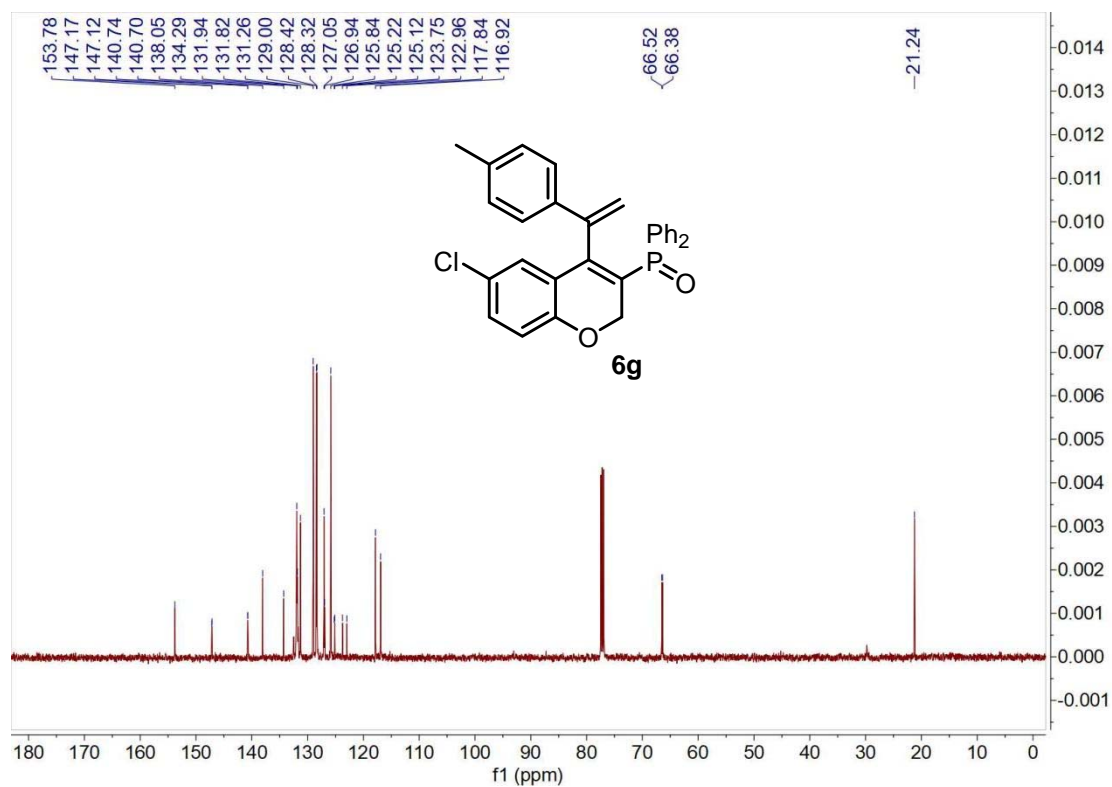
^{19}F -NMR (471 MHz, CDCl_3) of **6f**

30 #9 RT: 0.12 AV: 1 NL: 1.67E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

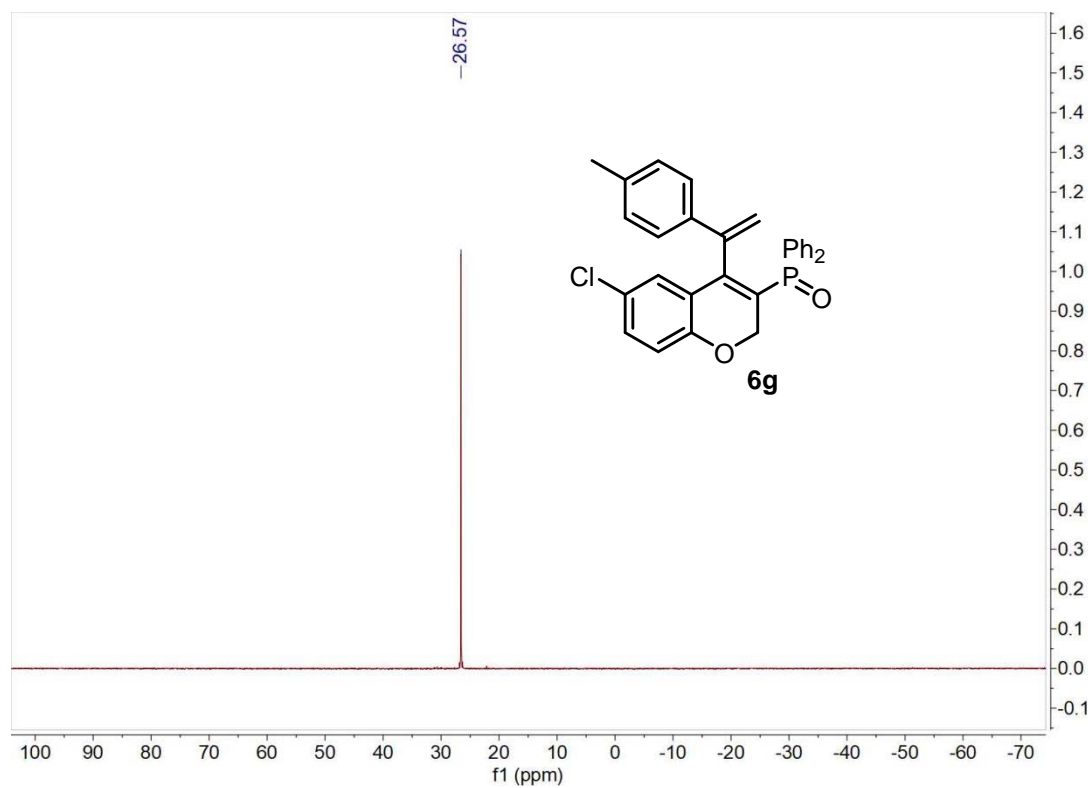


HRMS of **6f**



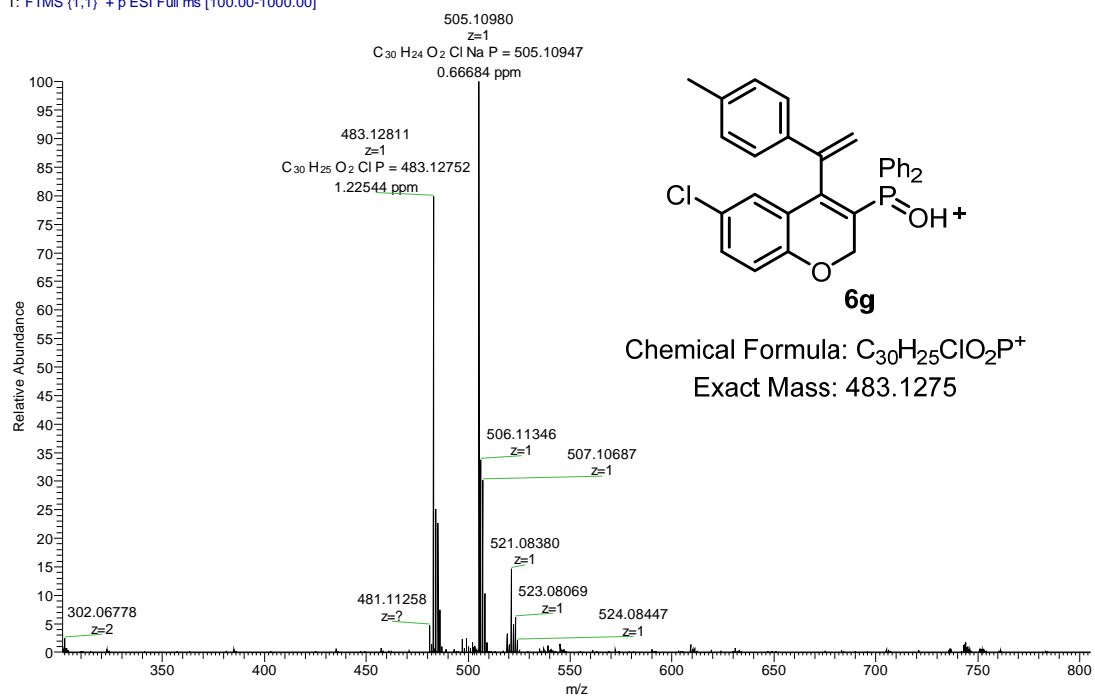


¹³C-NMR (126 MHz, CDCl₃) of **6g**

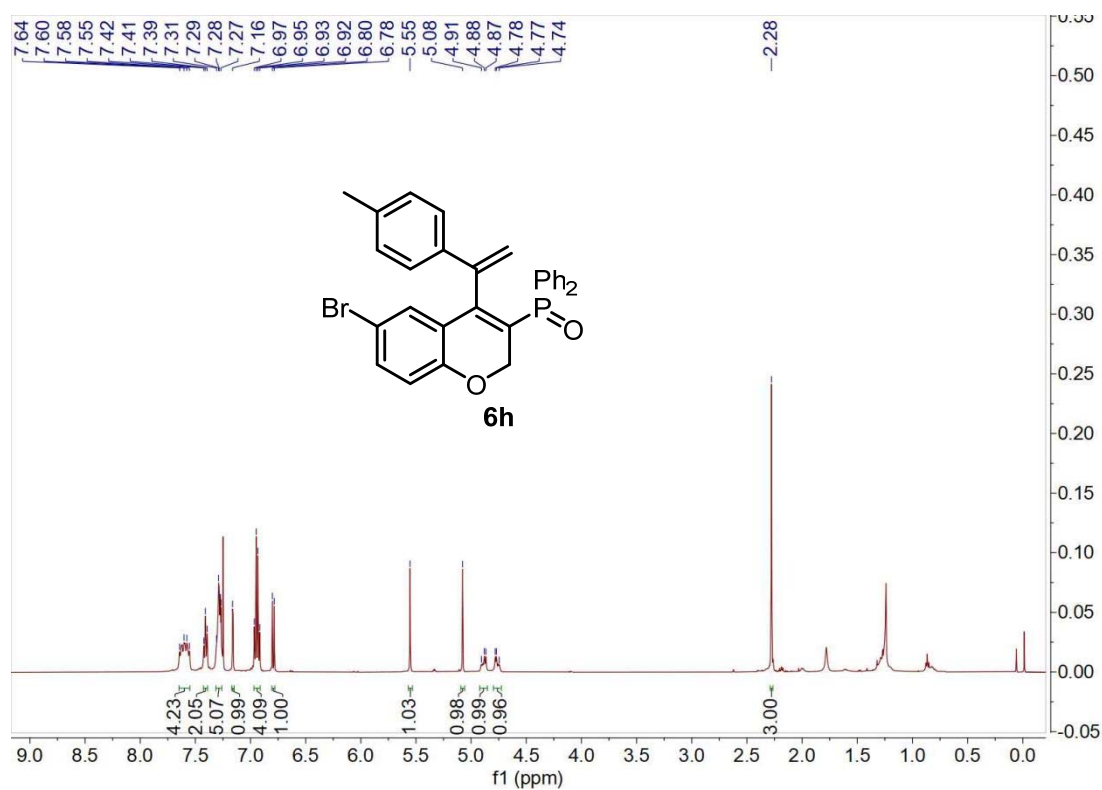


³¹P-NMR (202 MHz, CDCl₃) of **6g**

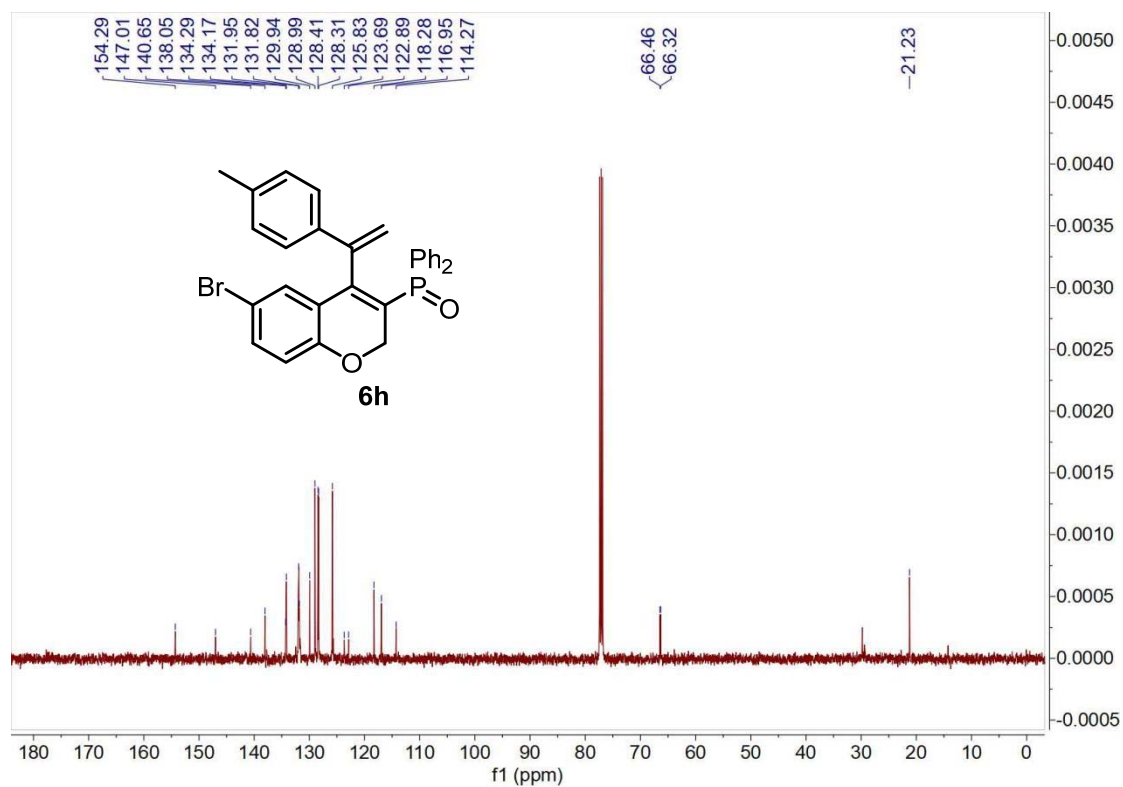
31 #33 RT: 0.40 AV: 1 NL: 9.46E5
T: FTMS (1,1) +p ESI Full ms [100.00-1000.00]



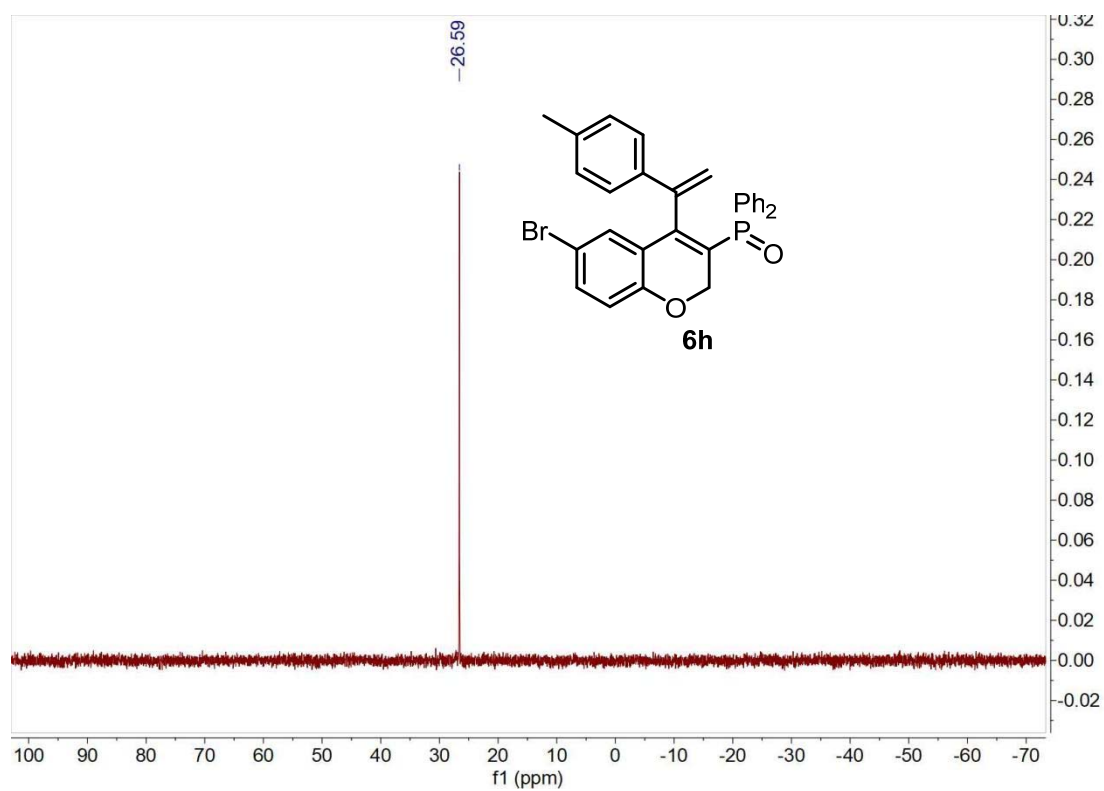
HRMS of **6g**



¹H-NMR (500 MHz, CDCl₃) of **6h**

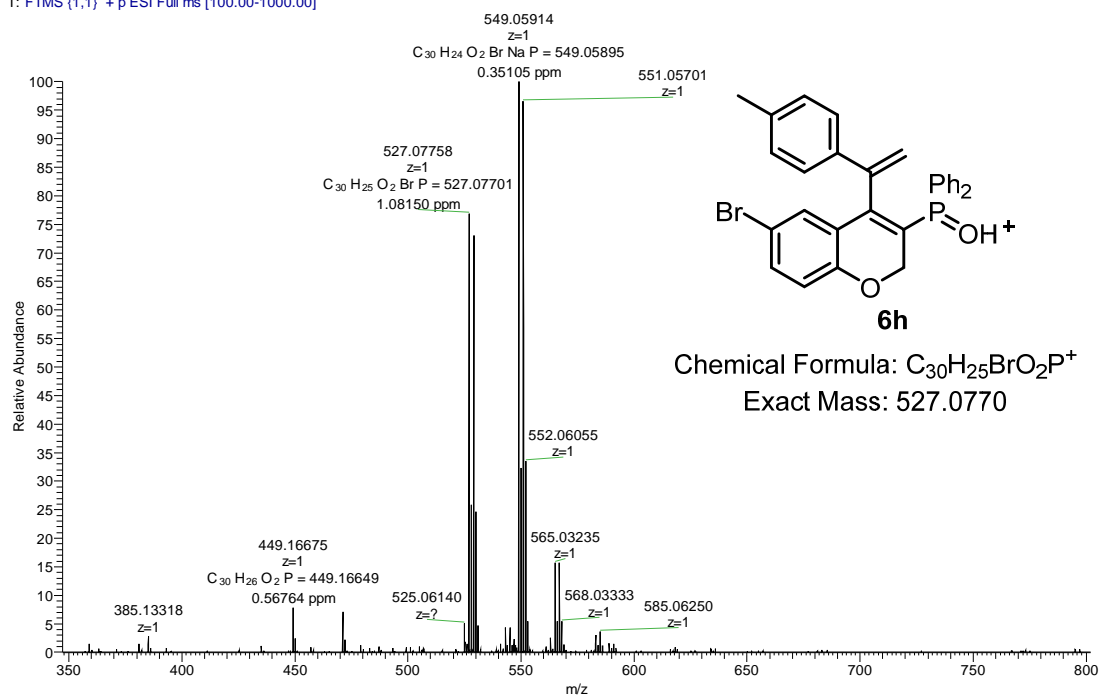


¹³C-NMR (126 MHz, CDCl₃) of **6h**

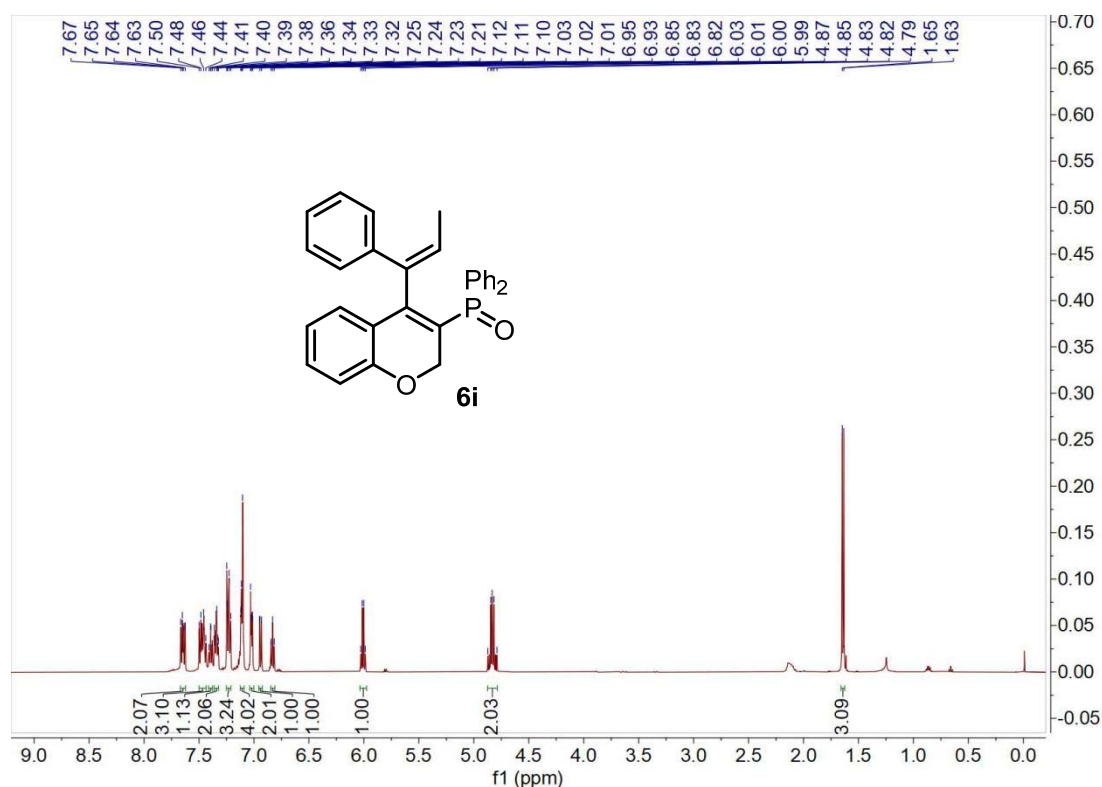


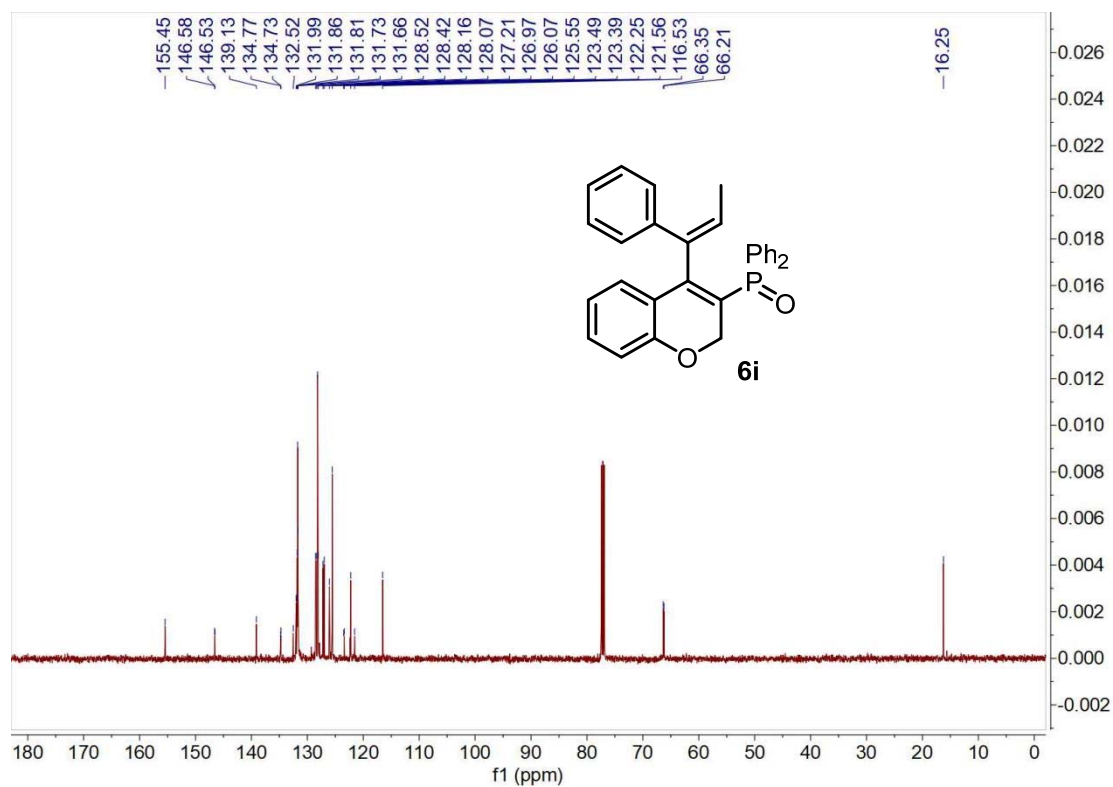
³¹P-NMR (202 MHz, CDCl₃) of **6h**

32 #27 RT: 0.33 AV: 1 NL: 6.18E5
 T: FTMS (1,1) +p ESI Full ms [100.00-1000.00]

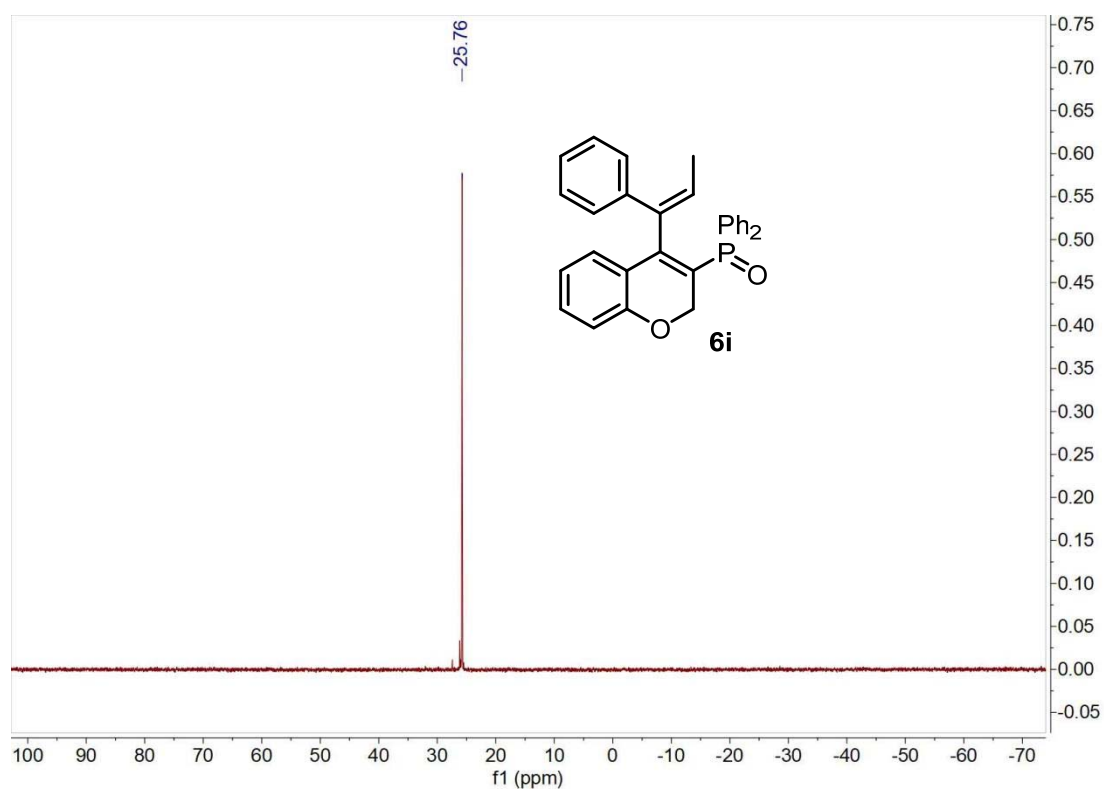


HRMS of **6h**



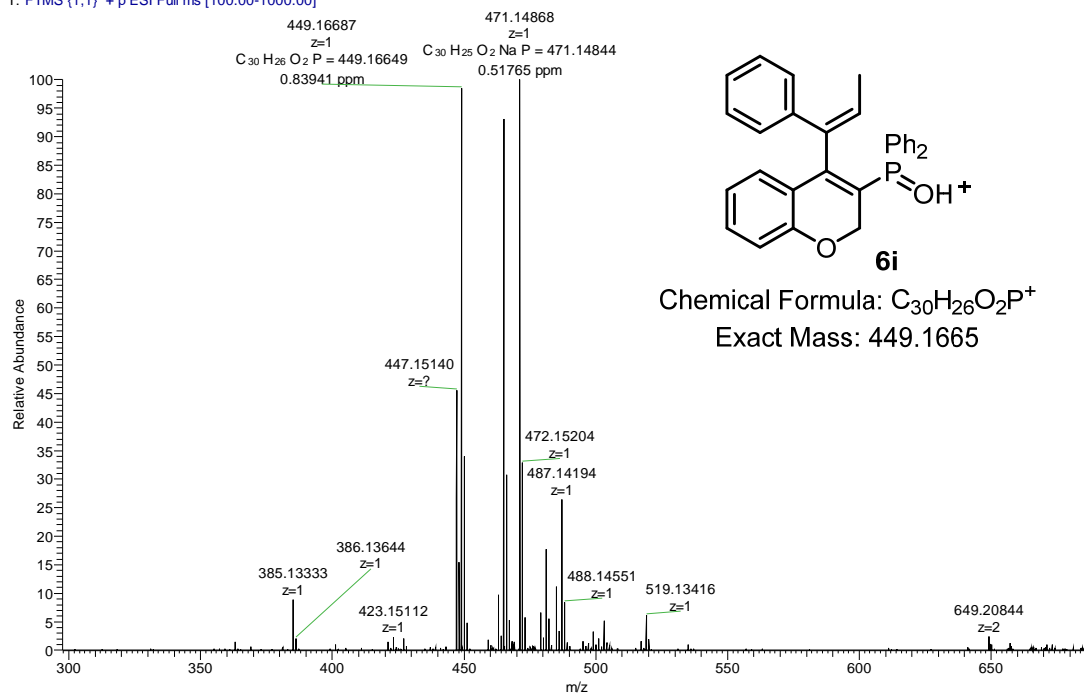


¹³C-NMR (126 MHz, CDCl₃) of **6i**

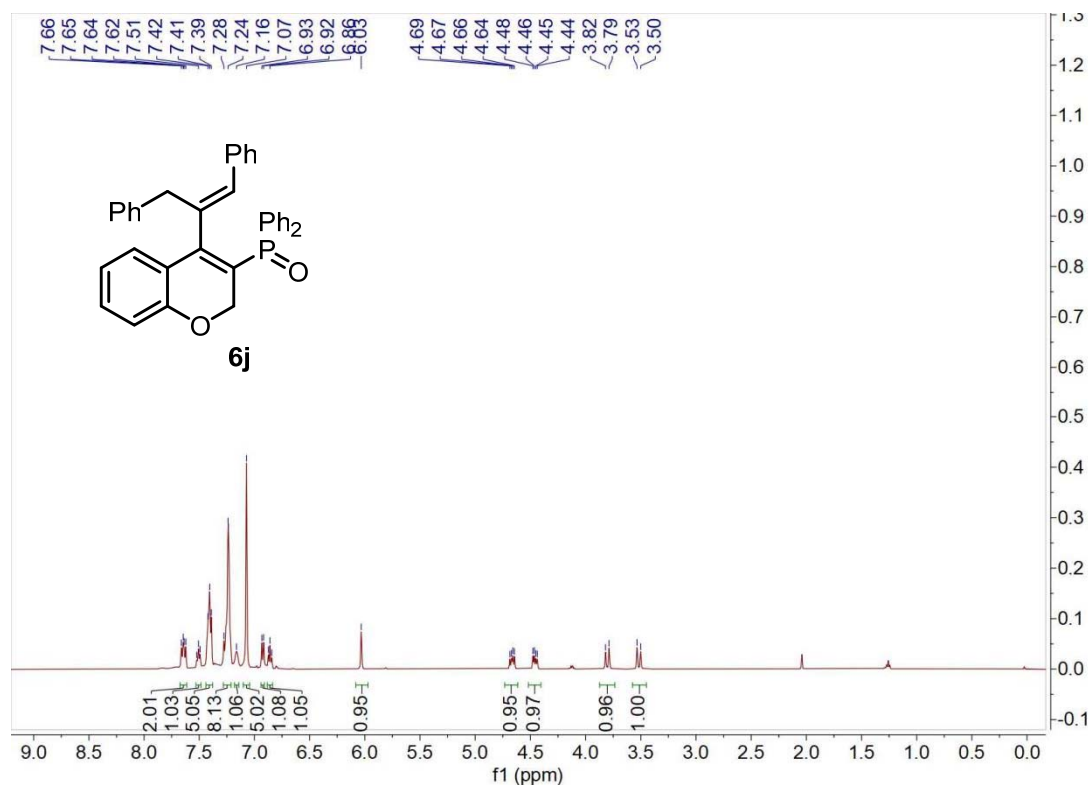


³¹P-NMR (202 MHz, CDCl₃) of **6i**

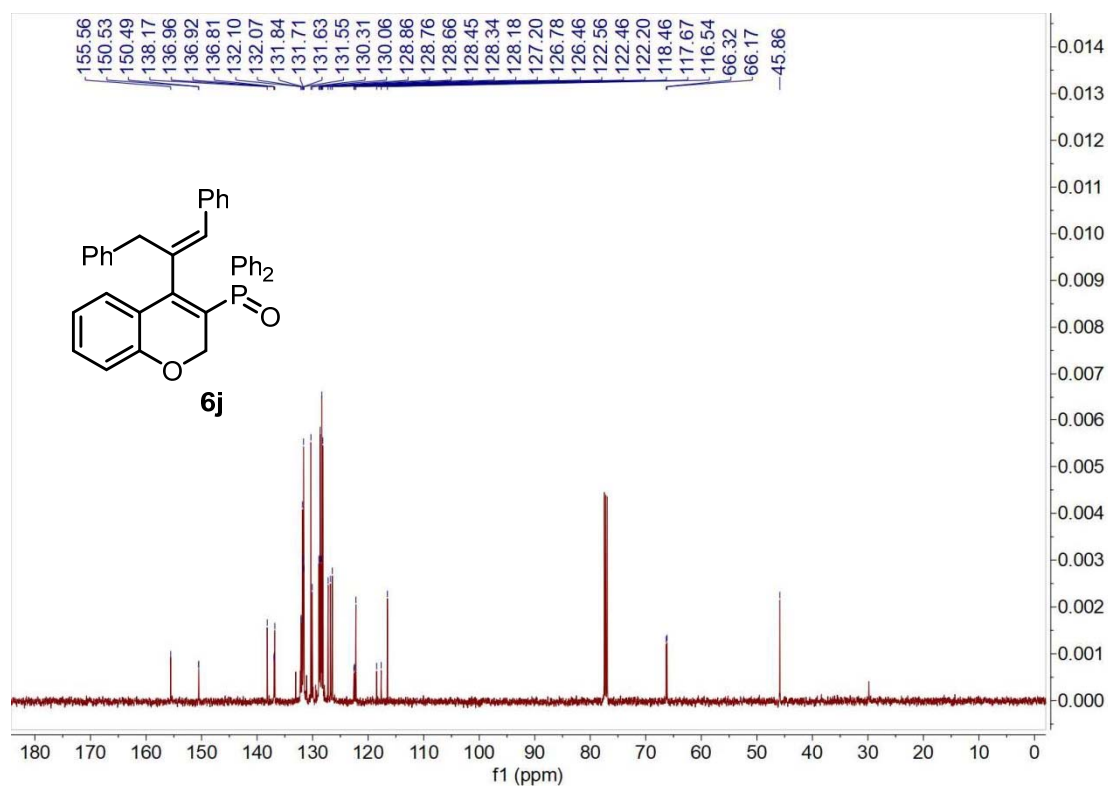
33 #21 RT: 0.25 AV: 1 NL: 1.44E6
T: FTMS (1,1) +p ESI Full ms [100.00-1000.00]



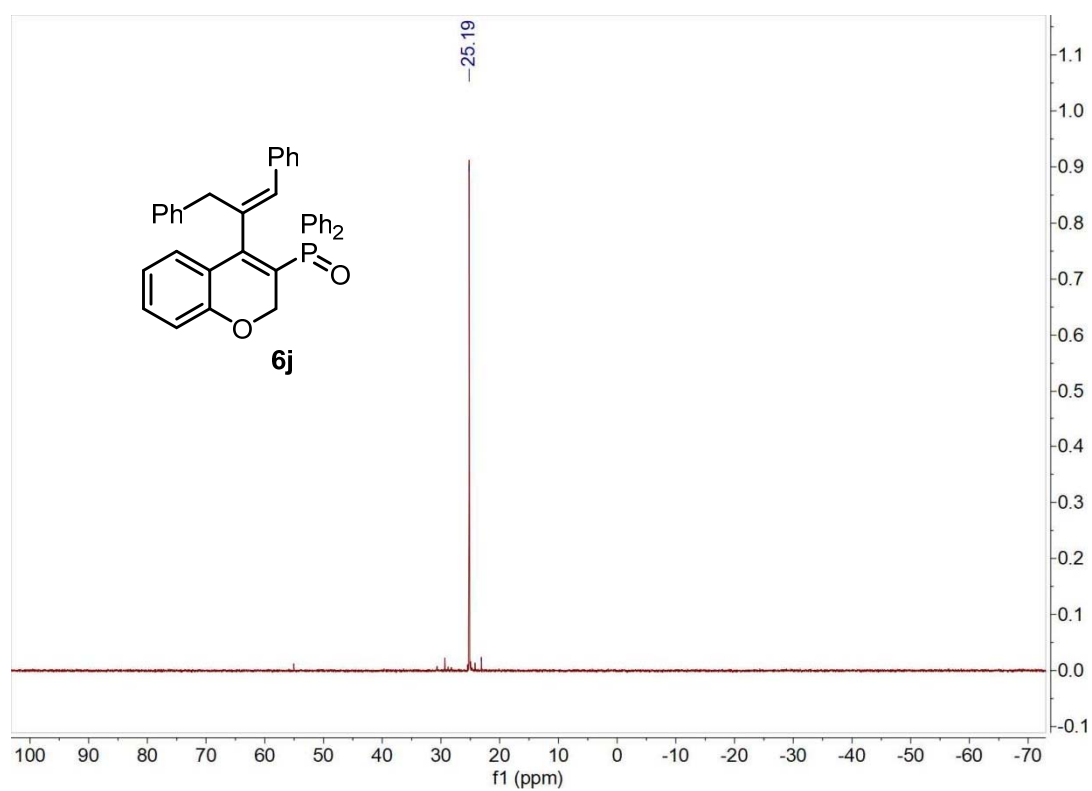
HRMS of **6i**



¹H-NMR (500 MHz, CDCl₃) of **6j**

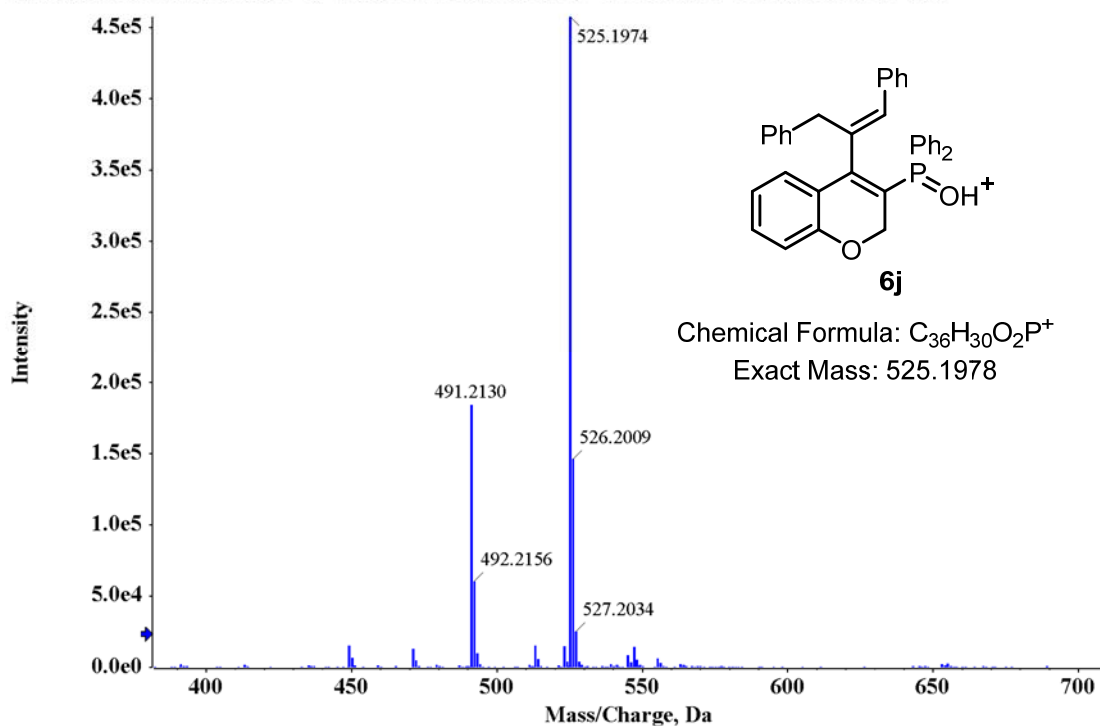


^{13}C -NMR (126 MHz, CDCl_3) of **6j**

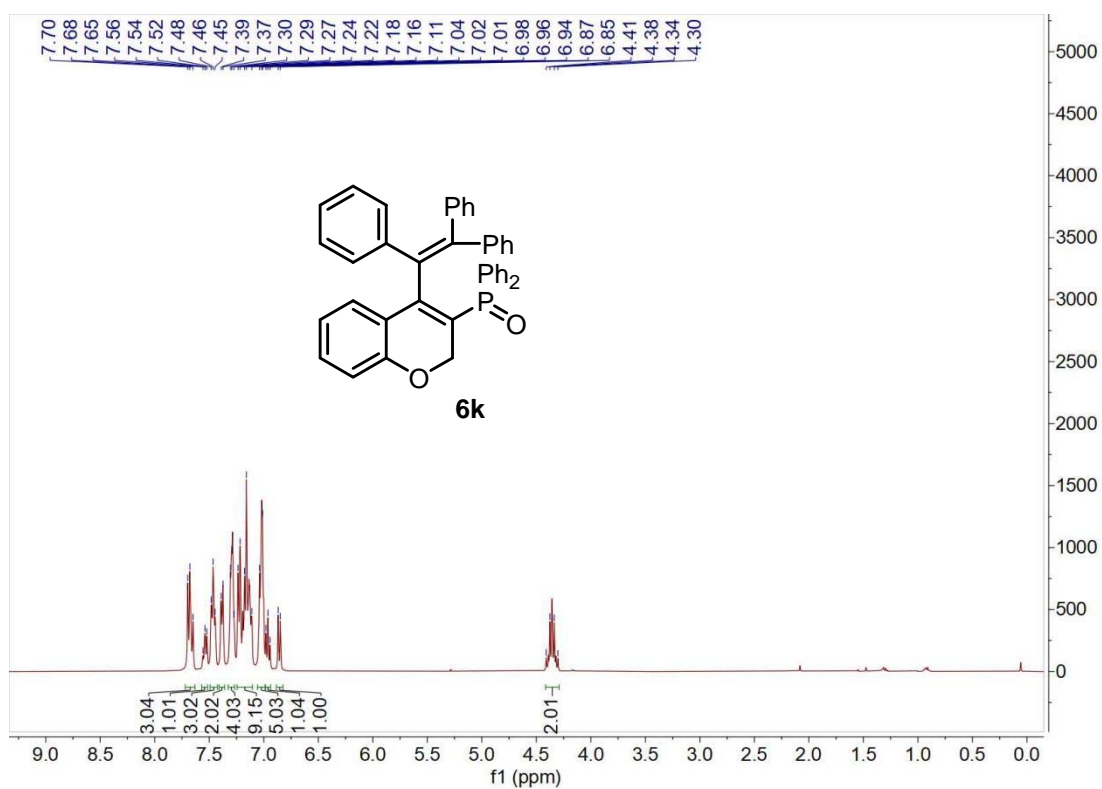


^{31}P -NMR (202 MHz, CDCl_3) of **6j**

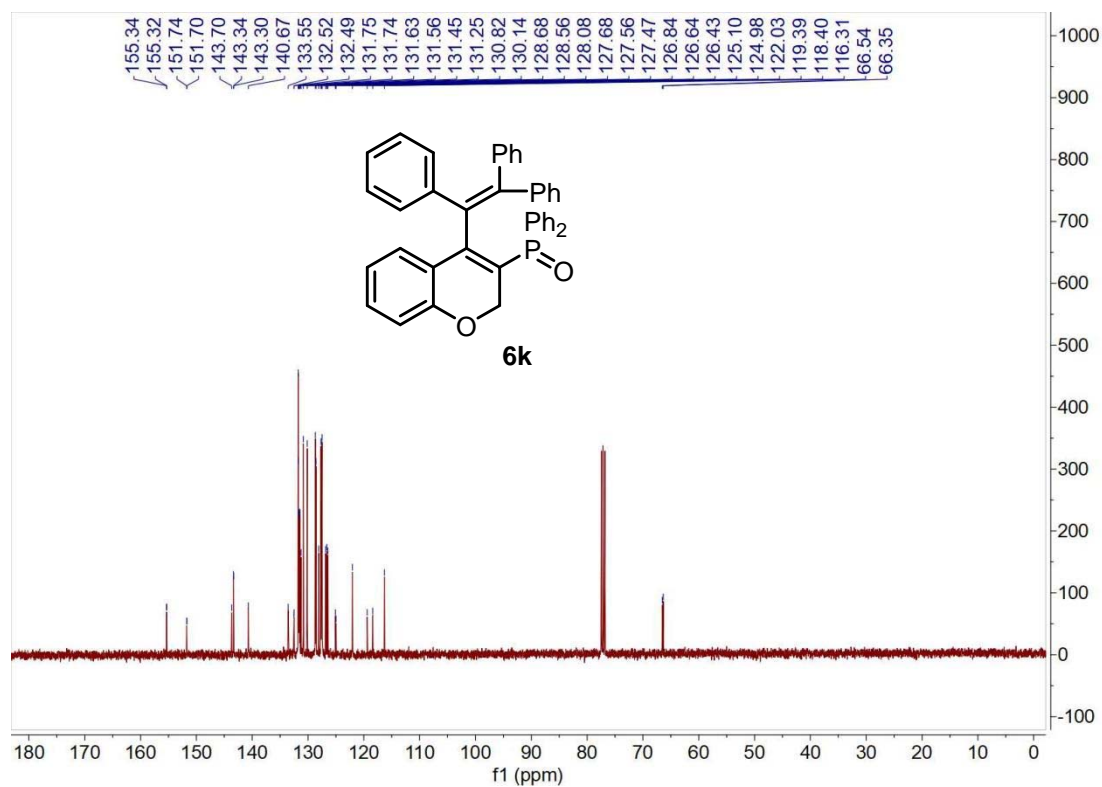
Spectrum from 08.wiff (sample 1) - Sample014, Experiment 1, +TOF MS (80 - 1000) from 1.101 min



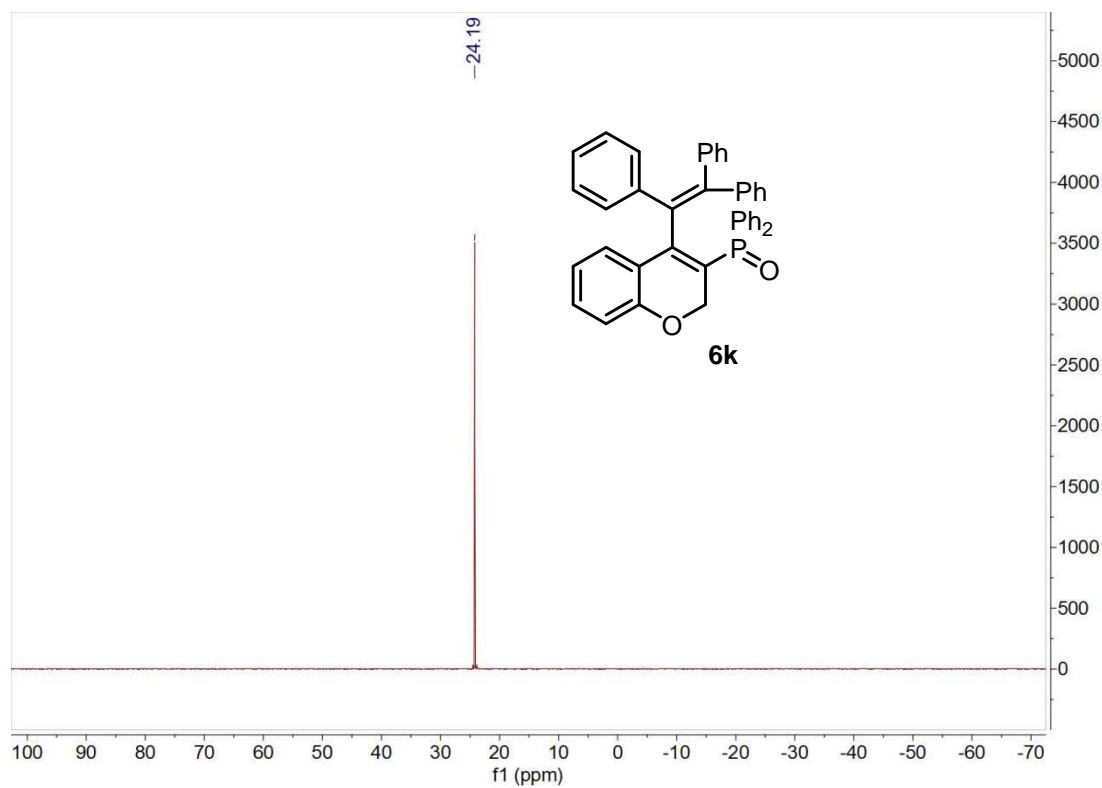
HRMS of **6j**



¹H-NMR (400 MHz, CDCl₃) of **6k**

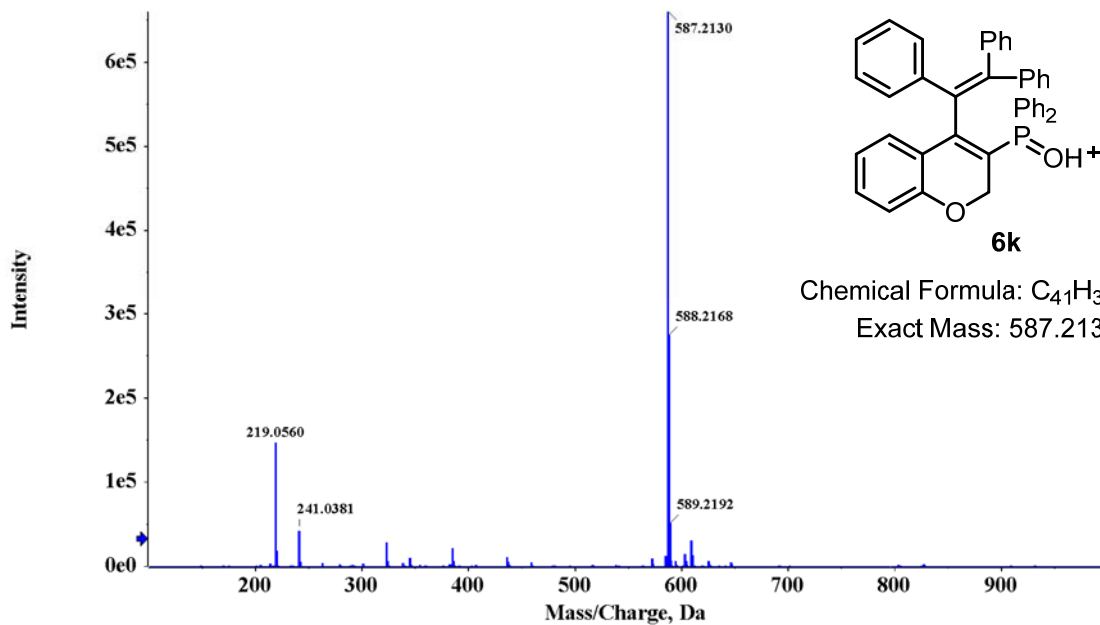


^{13}C -NMR (101 MHz, CDCl_3) of **6k**

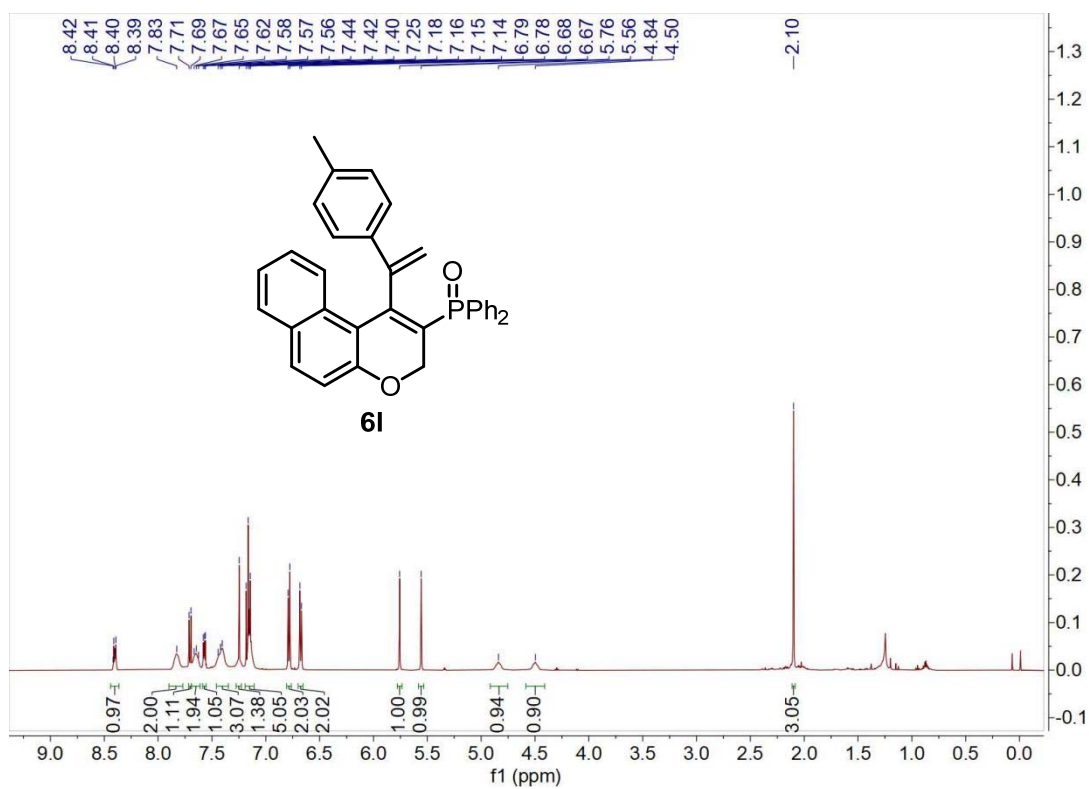


^{31}P -NMR (162 MHz, CDCl_3) of **6k**

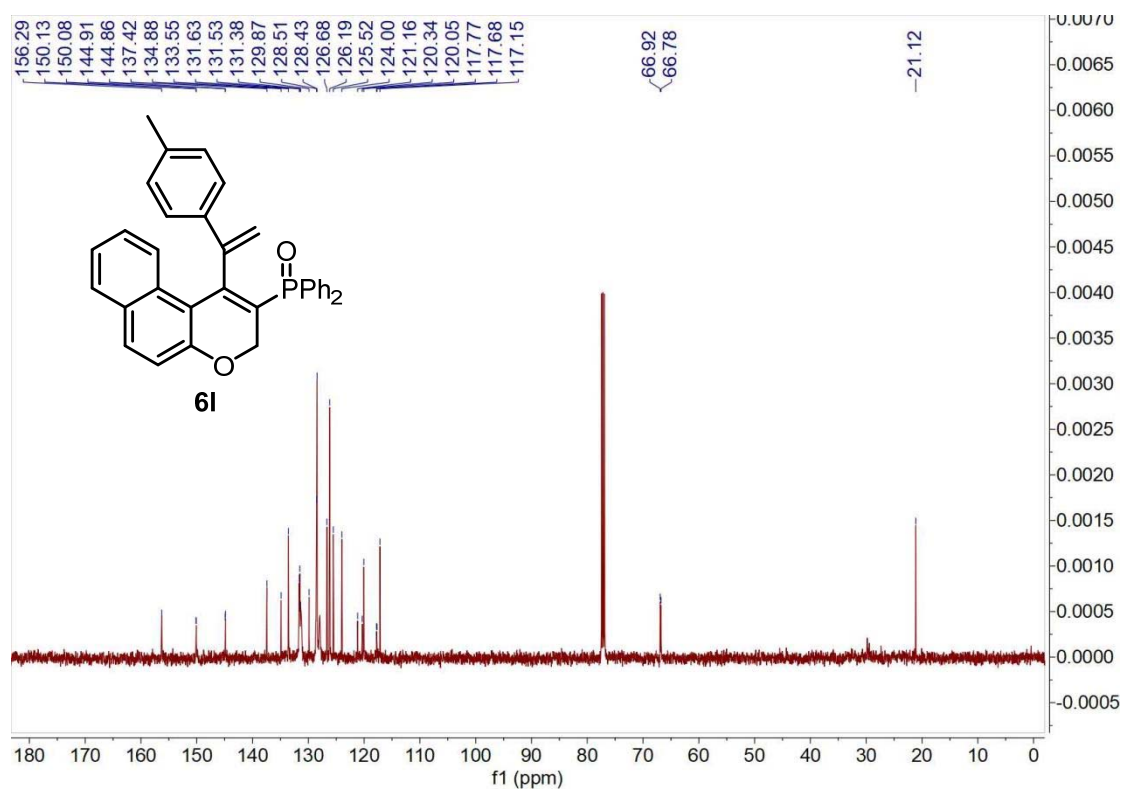
Spectrum from 1.wiff (sample 1) - Sample001, +TOF MS (100 - 1000) from 0.219 min



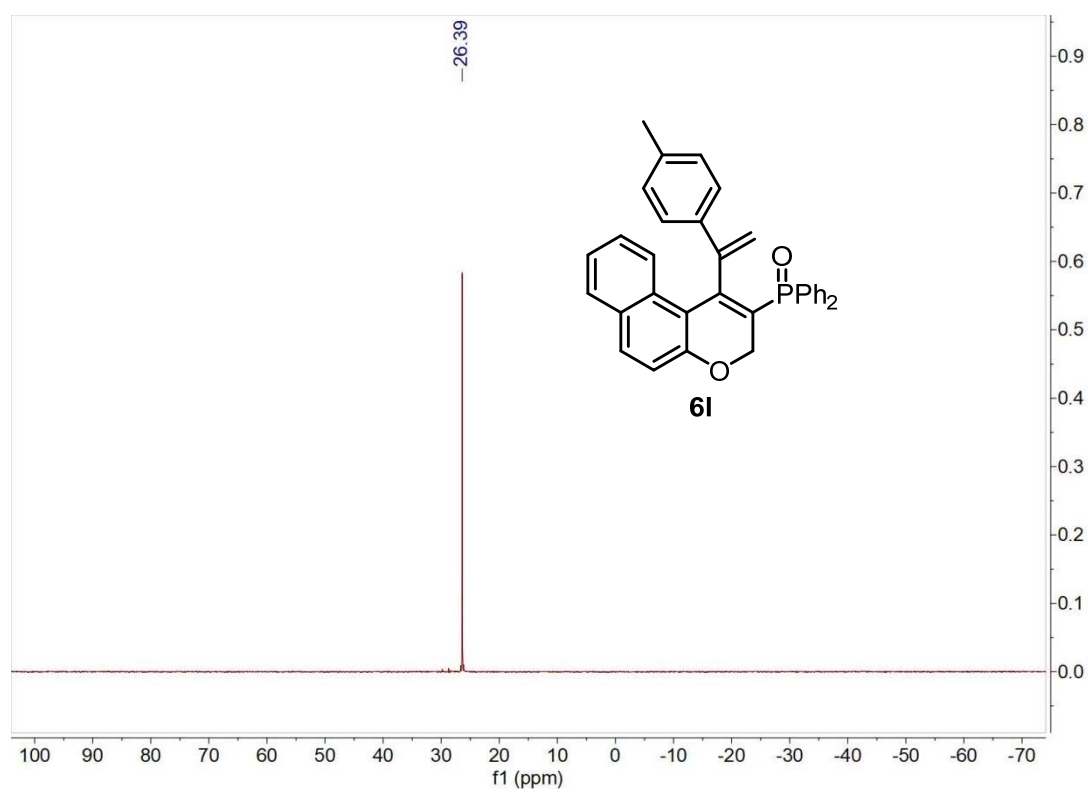
HRMS of **6k**



1H -NMR (500 MHz, $CDCl_3$) of **6l**

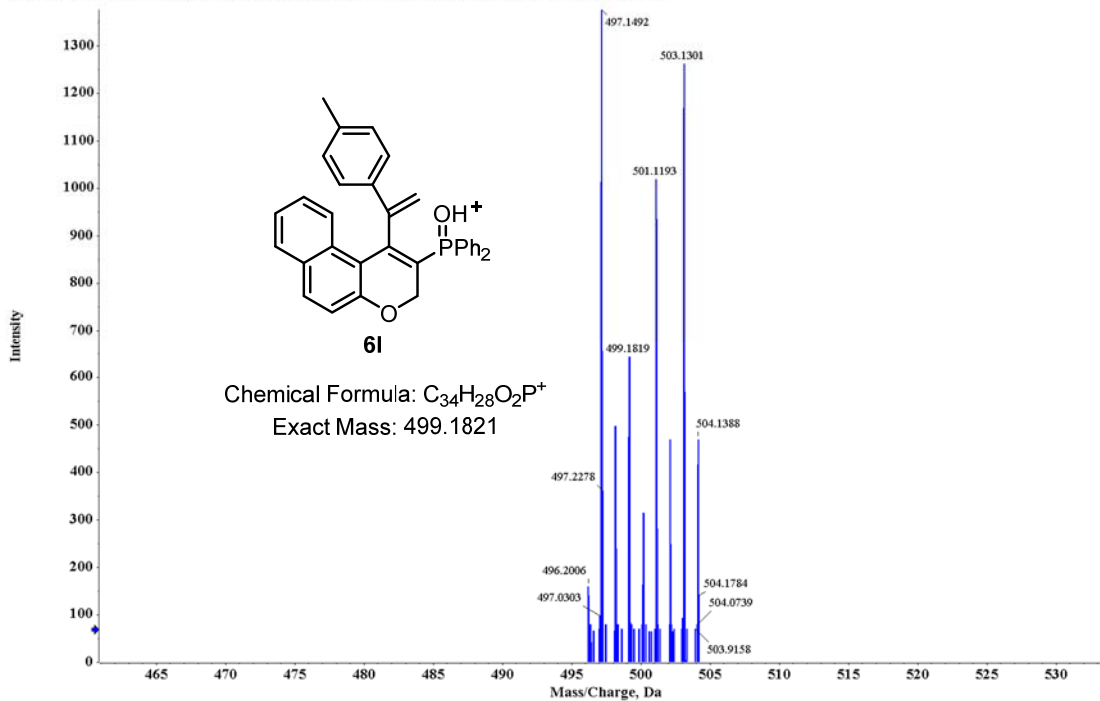


^{13}C -NMR (126 MHz, CDCl_3) of **6l**

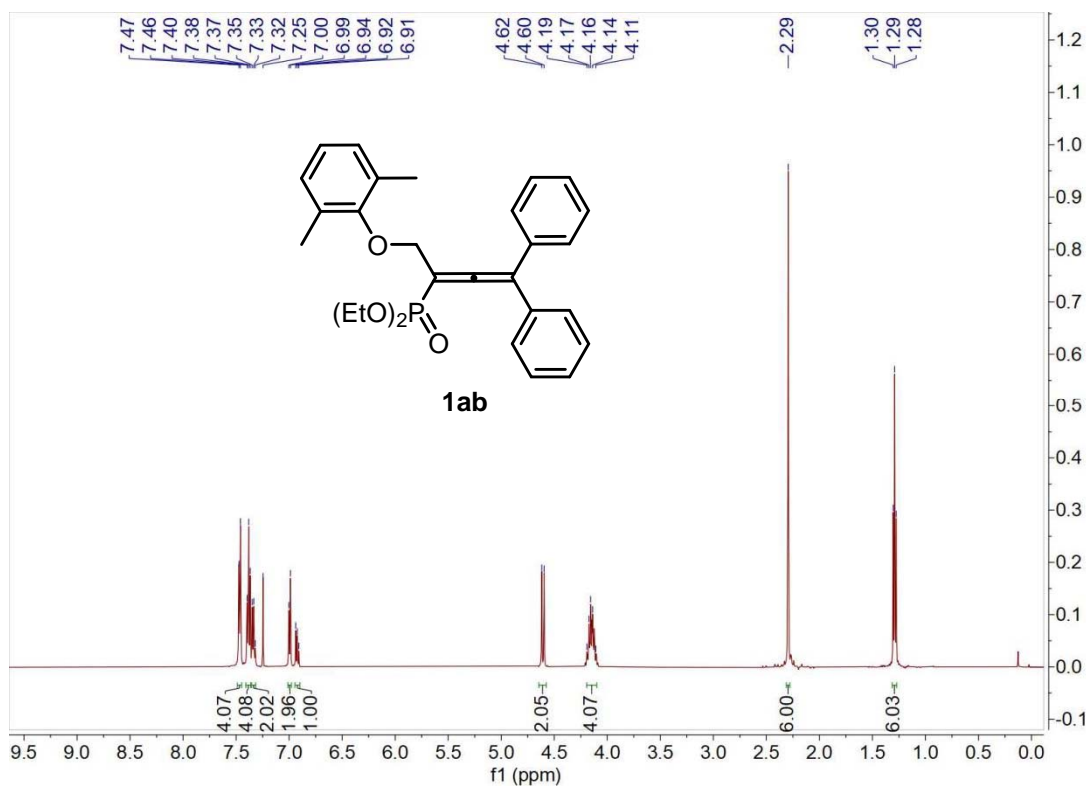


^{31}P -NMR (202 MHz, CDCl_3) of **6l**

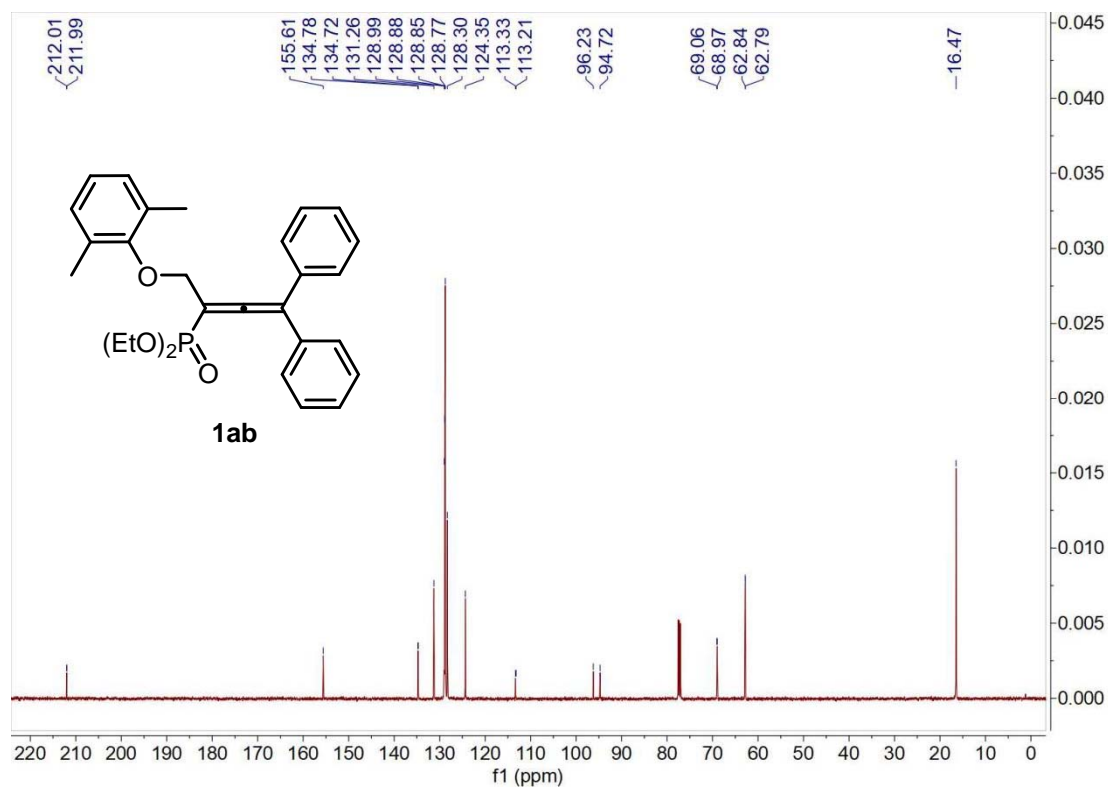
Spectrum from 08.wiff (sample 1) - Sample014, Experiment 1, +TOF MS (80 - 1000) from 0.457 to 0.501 min



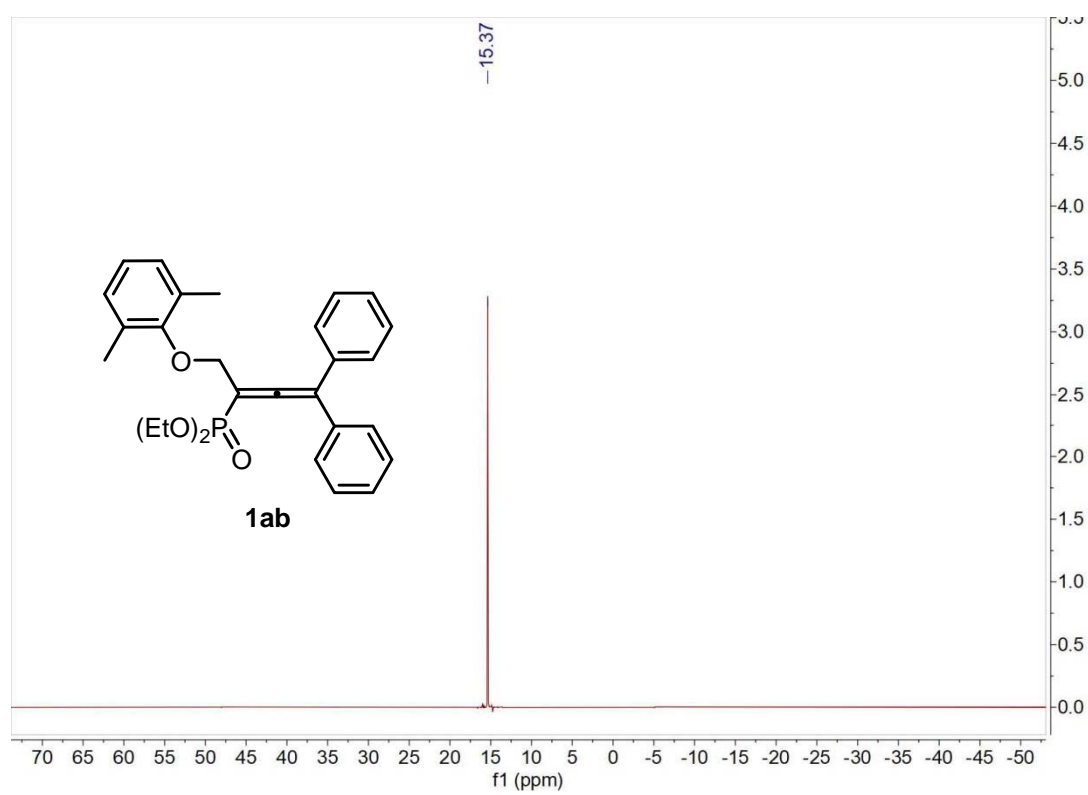
HRMS of **61**



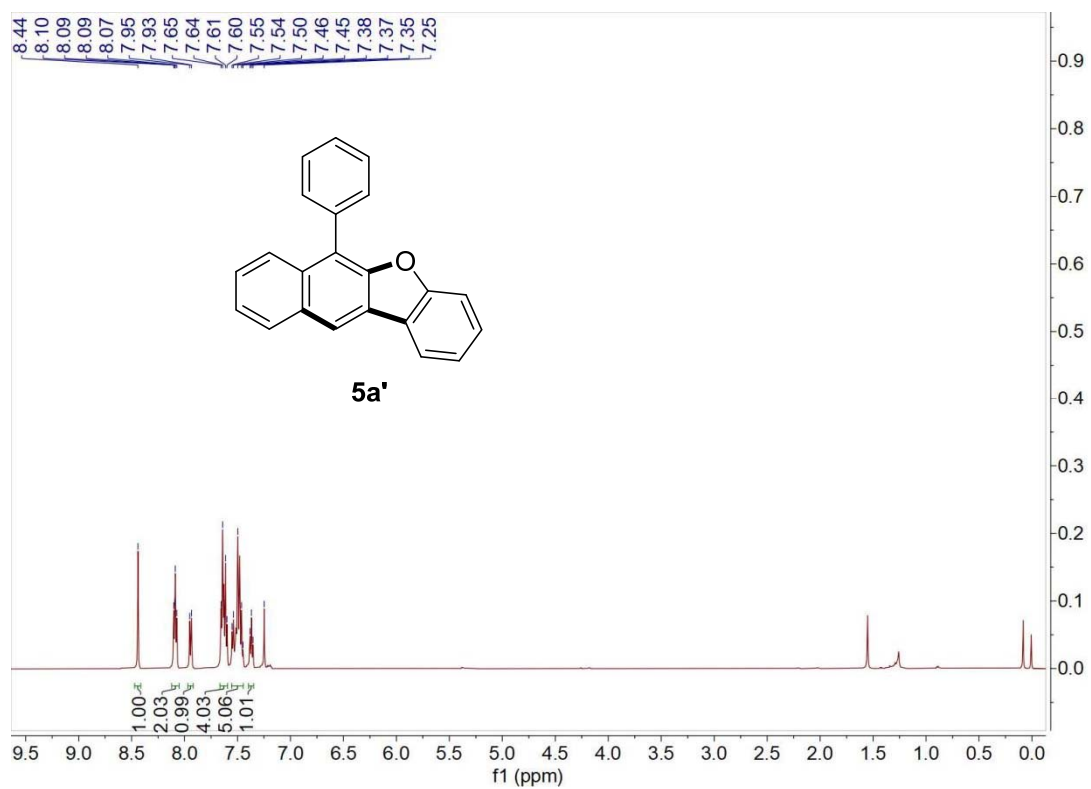
1H -NMR (500 MHz, $CDCl_3$) of **1ab**



^{13}C -NMR (126 MHz, CDCl_3) of **1ab**



^{31}P -NMR (202 MHz, CDCl_3) of **1ab**



$^1\text{H-NMR}$ (500 MHz, CDCl_3) of **5a'**