Support Information

Electrochemical oxidation of intramolecular annulation of aryl phosphine compounds: an efficient approach towards π-conjugated phosphonium salts

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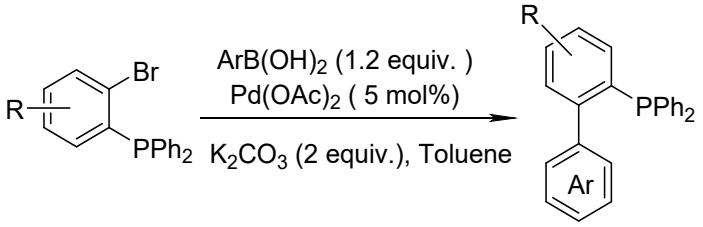
Content

I. General Information

All reagents used for experiments are commercially available and used directly without any further purification. The solvents need to be dehydrated and partly deoxidized before using, CH_3OH was treated with Mg and I_2, CH_2Cl_2 was treated with CaH_2, toluene and 1,4-dioxane were treated with sodium and benzophenone respectively. All experiments involving electrochemistry were carried out on the LK2010Z electrochemical workstation (made in China). Thin layer chromatography (TLC) was performed on pre-coated, glass-backed silica gel plates. Column chromatography was performed on silica gel 200 ~ 300 mesh. ^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz), ^{31}P NMR (162 M Hz) and ^{19}F (376 M Hz), and were recorded on JEOL JNM-ECZ400S NMR spectrometer with $CDCl_3$ or CD_3OD as solvent. Chemical shifts of all ^{1}H, ^{13}C ,^{31}P and ^{19}F NMR data spectra were reported in delta (δ) units, parts per million (ppm). The residual solvent signals were used as references and the chemical shifts converted to the TMS scale ($CDCl_3$: 7.26 ppm for ^{1}H NMR, 77.2 ppm for ^{13}C NMR, CD_3OD: 3.35 ppm for ^{1}H NMR, 49.8 ppm for ^{13}C NMR, Trimethyl phosphate: 2.8-3.2 ppm for ^{31}P NMR). Multiplicities are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; dd, doubled doublet; dt, doubled triplet; m, multiplet. All coupling constants (*J* values) were reported in Hertz (Hz). High-resolution mass spectra (HRMS) were obtained with a mass spectrometer.

II. General procedure for the preparation of phosphines substrates

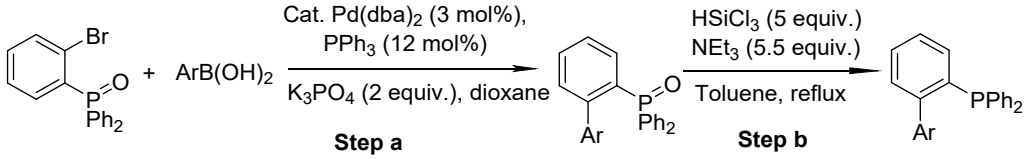
Compounds **1b-i** and **1n-t** were prepared based on previous literature[1].
Compounds **1j-m** and **1w** were prepared based on previous literature[2].
Method 1[1]: Suzuki cross coupling reaction for the synthesis of **1b-i**, **1n-t.**



The representative procedure is as follows: In an over- dried two- necked flask equipped with a stir bar, (2-bromophenyl) diphenylphosphane (1 mmol), aryl boronic acid (1.2 mmol), potassium carbon ate (2 mmol), $Pd(PPh_3)_4$ (5 mmol%) and 1,4-dioxane

(20 mL) was added. The reaction mixture was stirred and heated at 100 ℃ under N_2 atmosphere for 72 hours. After completion of the reaction, remove volatile components from crude products, and then water was added and extracted with CH_2Cl_2 (3 × 30 mL). The organic phase was dried over anhydrous magnesium sulfate, filtered and concentrated in *vacuo*. The desired phosphines substrates **1b-i** and **1n-t** were purified by silica gel column chromatography, using *n*-hexane: DCM = 25 : 1 – 5 : 1 as eluent.

Method 2[2]: Suzuki cross coupling reaction for the synthesis of **1j-m** and **1w**



Step a：The representative procedure is as follows: A mixture of (2-bromophenyl) diphenyl phosphine oxide (2.4 g, 6.8 mmol), 4-cyanophenylboronic acid (1.0 g, 6.8 mmol), $Pd(dba)_2$ (120 mg, 0.2 mmol), PPh_3 (220 mg, 0.82 mmol), K_3PO_4 (2 equiv., 2.9 g) and dried 1,4-dioxane (30 mL) were mixed and heated at 105 ℃ under N_2 atmosphere for 12 h. The reaction system was cooled to room temperature, volatile components were evaporated in a rotary evaporator, the remaining components were extracted with H_2O and DCM. The organic phase was washed with saturated salt water, dried with anhydrous magnesium sulfate, filtered, and concentrated in *vacuo*. The desired phosphine oxides were purified by column chromatography, using *n*-hexane: EtOAc = 4 : 1 – 2 : 1 as eluent.

Step b: The representative procedure is as follows: A solution of phosphine oxide in dried toluene was frozen using an EtOH/liquid nitrogen bath, to which trichlorosilane (2.1 mL, 21 mmol) and triethylamine (3.2 mL, 23 mmol) were added. The mixture was heated at 110 ℃ under N_2 atmosphere overnight. After cooling to room temperature, add 5 mL saturated $NaHCO_3$ solution to the mixture and stirred for 5 min. The mixture was filtered through a pad of alumina and concentrated in *vacuo*. The desired **1j-m** and **1w** were purified by column chromatography, using *n*-hexane: DCM = 10 : 1~ 5 : 1 as eluent.

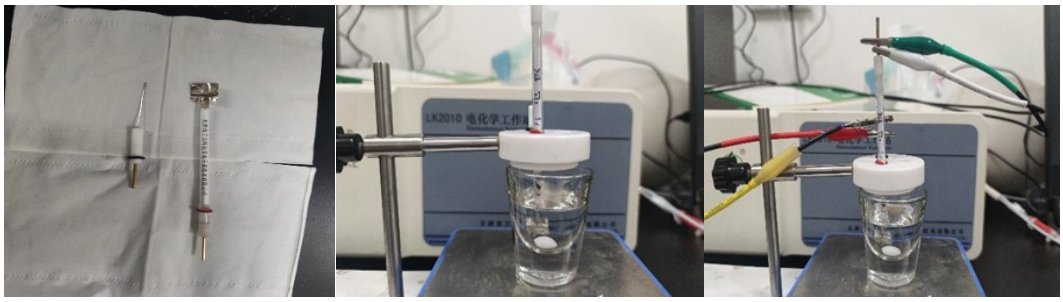
III. Reference

1. Li, G.; An, J.; Jia, C.; Yan, B.; Zhong, L.; Wang, J.; Yang, S. *Org. Lett.* **2020,** *22*, 9450-9455.

2. Baba, K.; Tobisu, M.; Chatani, N. *Angew. Chem. Int. Ed.* **2013,** *52*, 11892-11895.

IV. General procedure for the synthesis of phosphonium salts 2a-x

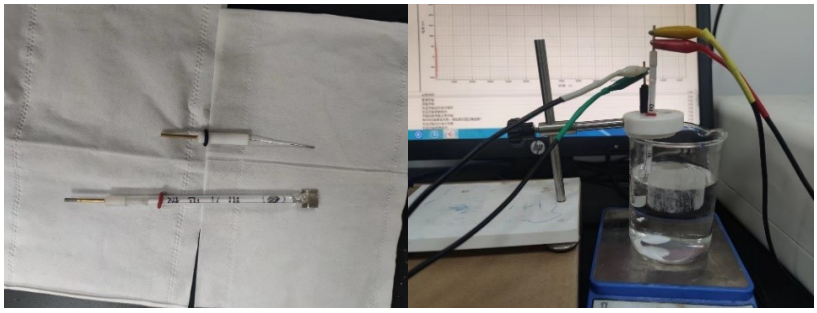
2-(2'-diphenylphosphine) diphenyl **2a** (0.2 mmol, 0.0677 g), NaOTf (0.4 mmol, 0.0688 g), HFIP/DCM/MeOH (0.75 mL/2.25 mL/8.00 mL) were mixed and added into an oven-dried undivided electrolytic cell (25 mL) equipped with a stir bar. The cell was equipped with platinum plate (10 mm × 10 mm × 1 mm) as the anode and platinum wire as the cathode, and the distance between them is 1.5 cm. The reaction mixture was stirred and electrolyzed at a constant current of 4 mA at room temperature for 4 h. When the reaction was finished, the solvent was evaporated under reduced pressure and the residue was absorbed onto small amounts of silica gel. The pure product was obtained by column chromatography on silica gel, using DCM : MeOH = 200 : 1 ~ 50 : 1 as eluent.



V. Procedure for Gram-scale synthesis of 2a

2-(2'-diphenylphosphine) diphenyl **2a** (5 mmol, 1.6919 g), NaOTf (10 mmol, 1.7205 g), HFIP/DCM/MeOH (18.75 mL/56.25 mL/200 mL) were mixed and added into an oven-dried undivided beaker (500 mL) equipped with a stir bar. The beaker was equipped with platinum plate (15 mm × 15 mm × 1 mm) as the anode and platinum wire as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under room temperature for 20 h. When the reaction was finished, the solvent was evaporated under reduced pressure and the residue was absorbed onto small amounts of silica gel. The pure product was obtained by column chromatography on

silica gel, using DCM : MeOH = 200 : 1 ~ 50 : 1 as eluent. Pure product **2a** was obtained in 77% isolated yield (1.8872 g).

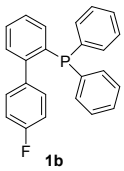


VI. Procedure for control experiment.

In an over-dried undivided electrolytic cell (25 mL) equipped with a stir bar, 2-(2'-diphenylphosphine) diphenyl **2a** (0.2 mmol, 0.0677 g), NaOTf (0.4 mmol, 0.0688 g), 2,2,6,6-Tetramethylpiperidine-1-oxy (TEMPO) (0.4 mmol, 0.0625 g) or 2,6-di-*tert*-butyl-4-methylphenol (BHT) (0.4 mmol, 0.0881 g), HFIP/DCM/MeOH (0.75 mL/2.25 mL/8.00 mL) was added. The cell was equipped platinum plate (10 mm × 10 mm×1 mm) as the anode and platinum wire as the cathode, the reaction mixture was stirred and electrolyzed at a constant current of 4 mA under room temperature for 4 h and stopped until complete consumption of **1a** (monitored by TCL, *n*-hexane: ethyl acetate = 10 : 1). The pure product was obtained by flash column chromatography on silica gel (DCM : MeOH = 200 : 1 ~ 50 : 1 as eluent).

VII. Data and spectra of ^{1}H NMR, ^{13}C NMR, ^{31}P NMR and ^{19}F NMR.

(4'-fluoro-[-biphenyl]-2-yl) diphenylphosphine (1b)



1b

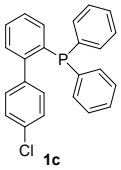
White solid. R_f 0.4 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 – 7.36 (m, 1H), 7.35 – 7.26 (m, 8H), 7.24 – 7.18 (m, 4H), 7.15 – 7.09 (m, 2H), 7.05 – 7.00 (m, 1H), 6.93 (t, J = 8.7 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.2 (d, J = 246.1 Hz), 147.2 (d, J = 28.1 Hz), 137.7 (dd, J = 5.8, 3.0 Hz), 137.3 (d, J = 11.3 Hz), 136.2 (d, J = 13.8 Hz), 134.2 (s), 134.0 (d, J = 14.3 Hz), 131.4 (dd, J = 8.0, 3.6 Hz), 130.3 (d, J = 4.6 Hz), 128.8 (s), 128.7 (s), 128.6 (d, J = 7.0 Hz), 127.6 (s), 114.6 (d, J = 21.3 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.80.

(4'-chloro-[-biphenyl]-2-yl) diphenylphosphine (1c)



1c

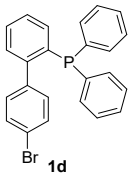
White solid. R_f 0.4 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.39 (m, 1H), 7.33 – 7.17 (m, 14H), 7.12 – 7.08 (m, 2H), 7.07 – 7.03 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.0 (d, J = 28.5 Hz), 140.2 (d, J = 6.2 Hz), 137.3 (d, J = 11.0 Hz), 136.0 (d, J = 13.9 Hz), 134.3, 134.0 (d, J = 19.7 Hz), 133.4, 131.1 (d, J = 3.6 Hz), 130.2 (d, J = 4.6 Hz), 129.0, 128.8, 128.6 (d, J = 6.9 Hz), 127.9, 127.8.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -13.10.

(4'-bromo-[-biphenyl]-2-yl) diphenylphosphine (1d)



1d

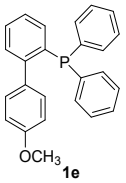
White solid. R_f 0.4 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41–7.36 (m, 3H), 7.31–7.24 (m, 8H), 7.22–7.18 (m, 4H), 7.04–7.02 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.1 (d, J = 28.5 Hz), 140.8 (d, J = 6.3 Hz), 137.4 (d, J = 11.4 Hz), 136.1 (d, J = 14.5 Hz), 134.4, 134.1 (d, J = 20.0 Hz), 131.6 (d, J = 3.7 Hz), 131.0, 130.2 (d, J = 4.6 Hz), 129.1, 128.9, 128.7 (d, J = 6.8 Hz), 128.0, 121.8.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -13.2.

(4'-methoxy-[-biphenyl]-2-yl) diphenylphosphine (1e)



1e

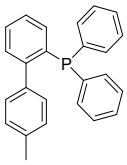
White solid. R_f 0.2 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.40 (t, J = 7.8 Hz, 1H), 7.37 – 7.31 (m, 7H), 7.30 – 7.23 (m,5H), 7.15 (d, J = 7.9 Hz, 2H), 7.11 – 7.06 (m, 1H), 6.84 (d, J = 8.6 Hz, 2H), 3.82 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.8, 148.0 (d, J = 28.9 Hz), 137.8 (d, J = 11.6 Hz), 135.8 (d, J = 13.2 Hz), 134.2, 134.1, 133.9 (d, J = 19.6 Hz), 130.8 (d, J = 3.6 Hz), 130.3 (d, J = 4.8 Hz), 128.7, 128.4 (d, J = 2.7 Hz), 128.4, 127.1, 113.0, 55.2.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -13.05.

(4'-methyl-[-biphenyl]-2-yl) diphenylphosphine (1f)



1f

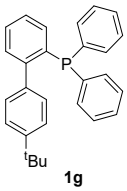
Colorless oil. R_f 0.2 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.37 – 7.30 (m, 2H), 7.29 – 7.18 (m, 11H), 7.10 – 7.03 (m, 5H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.7, 148.4, 139.0 (d, J = 6.6 Hz), 138.0 (d, J = 11.7 Hz), 137.0, 135.8 (d, J = 13.4 Hz), 134.5, 134.0 (d, J = 19.6 Hz), 130.3 (d, J = 5.1 Hz), 129.7 (d, J = 3.7 Hz), 128.9, 128.5, 128.5, 128.4, 127.3, 21.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -13.37.

(4'-(tert-butyl)-[-biphenyl]-2-yl) diphenylphosphine (1g)



1g

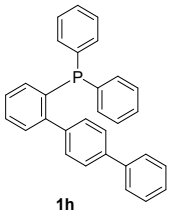
White solid. R_f 0.3 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (m, 2H), 7.41 – 7.29 (m, 13H), 7.23 (d, J = 7.4 Hz, 2H), 7.16 (m, 1H), 1.42 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.8, 148.3, 148.0, 138.6 (d, J = 6.1 Hz), 137.8 (d, J = 11.7 Hz), 136.0, 135.8, 134.0, 133.8, 130.2 (d, J = 4.7 Hz), 129.4 (d, J = 3.7 Hz), 128.7, 128.4, 128.4, 128.3, 127.1, 124.5, 34.5, 31.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.28.

[1,1':4',1''-terphenyl]-2-yldiphenylphosphine (1h)



1h

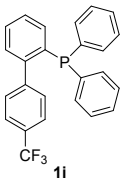
White solid. R_f 0.4 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71 (d, J = 6.7 Hz, 1H), 7.61 (d, J = 7.2 Hz, 2H), 7.51 (d, J = 7.9 Hz, 2H), 7.43 (m, 3H), 7.29 (m, 13H), 7.10 (d, J = 3.0 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.0 (d, J = 28.4 Hz), 140.9 (d, J = 18.0 Hz), 140.0, 137.6 (d, J = 11.1 Hz), 135.9 (d, J = 13.6 Hz), 134.3, 134.2, 134.0, 130.3, 128.9(d, J = 8.3 Hz), 128.6, 128.6, 128.5, 127.5, 127.3 (d, J = 10.5 Hz), 126.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.77.

(4'-(trifluoromethyl)-[-biphenyl]-2-yl) diphenylphosphine (1i)



1i

White solid. R_f 0.2 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53 (d, J = 8.1 Hz, 2H), 7.43 (t, J = 6.9 Hz, 1H)., 7.38 – 7.28 (m, 10H), 7.25 (m, 4H), 7.12 (m, 1H).

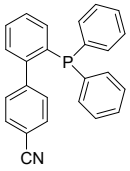
^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.7 (d, J = 27.9 Hz), 145.4 (d, J = 5.7 Hz), 137.0 (d, J = 11.2 Hz), 136.2 (d, J = 15.1 Hz), 134.2 (d, J = 19.0 Hz), 133.9, 130.2, 130.1, 129.5, 129.2, 128.9, 128.8, 128.6, 128.5, 128.3 – 128.0 (m), 125.8, 124.6 (d, J = 3.4 Hz), 123.0, 120.3.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.54.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.21.

Exact Mass (ESI): Calcd for $C_{25}H_{19}F_3P$ $[M+H]^+$, 407.11765, found 407.11631.

2'-(diphenylphosphanyl)-[1,1'-biphenyl]-4-carbonitrile (1j)



1j

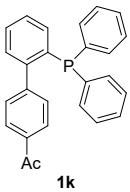
White solid. R_f 0.1 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53 (d, J = 1.9 Hz, 1H), 7.51 (d, J = 2.0 Hz, 1H)., 7.45 – 7.36 (m, 1H), 7.35 – 7.12 (m, 14H), 7.11 – 6.99 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.4 (d, J = 5.7 Hz), 146.1, 145.8, 136.5 (d, J = 11.1 Hz), 136.0 (d, J = 15.4 Hz), 134.0 (d, J = 20.1 Hz), 131.4, 130.5 (d, J = 3.9 Hz), 129.7 (d, J = 4.5 Hz), 128.9, 128.8, 128.5 (d, J = 7.0 Hz), 128.3, 119.0, 110.9.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.84.

(4'-(acetyl)-[-biphenyl]-2-yl) diphenylphosphine (1k)



1k

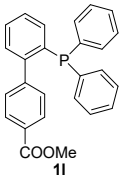
White solid. R_f 0.1 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93 – 7.85 (m, 2H), 7.47 – 7.39 (m, 1H), 7.38 – 7.28 (m, 10H), 7.26 (m, 4H), 7.11 (m, 1H), 2.61 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 198.0, 147.1 (d, J = 28.7 Hz), 146.8(d, J = 5.9 Hz), 137.2 (d, J = 11.3 Hz), 136.0, 135.8, 134.3, 134.0 (d, J = 20.0 Hz), 130.0 (d, J = 3.8 Hz), 130.0 (d, J = 4.8 Hz), 129.0, 128.8, 128.6 (d, J = 6.9 Hz), 128.0, 127.8, 26.8.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -13.41.

(4'-(carboxylate)-[-biphenyl]-2-yl) diphenylphosphine (1l)



1l

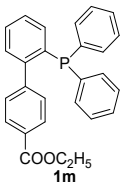
White solid. R_f 0.2 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (d, J = 8.3 Hz, 2H), 7.45 – 7.38 (m, 1H), 7.34 – 7.25 (m, 10H), 7.21 (m, 4H), 7.09 (m, 1H), 3.91 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.1, 147.3, 147.0, 146.5, 137.2 (d, J = 11.5 Hz), 136.0 (d, J = 15.1 Hz), 134.2, 134.0 (d, J = 19.9 Hz), 130.0, 129.9 (d, J = 4.0 Hz), 129.0, 128.9, 128.7, 128.5 (d, J = 7.0 Hz), 128.0, 52.2.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.79.

(4'-(ethoxycarbonyl)-[-biphenyl]-2-yl) diphenylphosphine (1m)



1m

White solid. R_f 0.2 (hexane/DCM = 5 : 1)

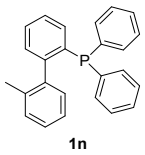
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.99 – 7.92 (m, 2H), 7.45 – 7.38 (m, 1H), 7.36 – 7.25 (m, 10H), 7.22 (m, 4H), 7.12 – 7.05 (m, 1H), 4.38 (q, J = 7.1 Hz, 2H), 1.39 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.6, 147.3 (d, J = 28.5 Hz), 146.4 (d, J = 5.9 Hz), 137.3 (d, J = 11.5 Hz), 136.0 (d, J = 14.8 Hz), 134.3, 134.0 (d, J = 19.8 Hz), 130.0 (d, J = 4.7 Hz), 129.8 (d, J = 3.7 Hz), 129.2, 129.0 (d, J = 7.9 Hz), 128.7, 128.5 (d, J = 6.8 Hz), 128.0.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.99.

Exact Mass (ESI): Calcd for $C_{27}H_{24}O_2P^+$ $[M+H]^+$, 411.15139, found 411.15058.

(2'-(methyl)-[-biphenyl]-2-yl) diphenylphosphine (1n)



1n

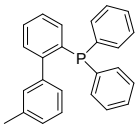
White solid. R_f 0.3 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 (m, 1H), 7.32 – 7.24 (m, 7H), 7.22 – 7.13 (m, 7H), 7.08 (m, 1H), 7.00 – 6.95 (m, 1H), 6.79 (d, J = 7.5 Hz, 1H), 2.04 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.7 (d, J = 30.8 Hz), 141.2 (d, J = 6.8 Hz), 137.4 (d, J = 12.3 Hz), 137.2 (d, J = 11.2 Hz), 136.8, 135.9, 134.0 (d, J = 20.3 Hz), 133.6, 130.4, 129.8 (d, J = 5.3 Hz), 129.7, 128.8, 128.5 (d, J = 4.2 Hz), 128.4 (d, J = 7.1 Hz), 128.3, 127.5 (d, J = 17.6 Hz), 124.9, 20.6 (d, J = 4.0 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.72.

(3'-(methyl)-[-biphenyl]-2-yl) diphenylphosphine (1o)



1o

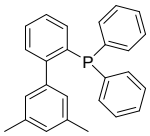
Colorless oil. R_f 0.3 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.33 (m, 2H), 7.25 (m, 11H), 7.15 (t, J = 7.5 Hz, 1H), 7.10 – 7.04 (m, 2H), 6.99 (d, J = 7.7 Hz, 1H), 6.90 (s, 1H), 2.21 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.6 (d, J = 29.0 Hz), 141.8 (d, J = 6.4 Hz), 138.0 (d, J = 11.8 Hz), 137.2, 136.1 (d, J = 13.9 Hz), 134.3, 134.1 (d, J = 20.0 Hz), 130.8 (d, J = 3.2 Hz), 130.2 (d, J = 4.8 Hz), 128.8, 128.6 (d, J = 1.8 Hz), 128.5, 128.1, 127.6, 127.4, 126.8 (d, J = 3.6 Hz), 21.6.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.84.

(3', 5'-(dimethyl)-[-biphenyl]-2-yl) diphenylphosphine (1p)



1p

White solid. R_f 0.2 (hexane/DCM = 5 : 1)

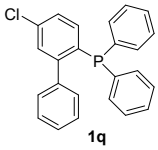
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 – 7.27 (m, 13H), 7.09 (m, 1H), 6.94 (s, 1H), 6.74 (s, 2H), 2.22 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.6 (d, J = 29.5 Hz), 141.7 (d, J = 6.7 Hz), 138.2 (d, J = 11.5 Hz), 137.0, 136.0 (d, J = 13.8 Hz), 134.2 (d, J = 3.7 Hz), 134.0, 130.0 (d, J = 4.9 Hz), 128.8 (d, J = 27.1 Hz), 128.5, 128.4, 127.7 (d, J = 3.4 Hz), 127.7 (d, J = 3.4 Hz), 127.2, 127.2, 21.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.96.

Exact Mass (ESI): Calcd for $C_{26}H_{24}P^+$ $[M+H]^+$, 367.16156, found 367.15079.

(5-chloro-[1,1'-biphenyl]-2-yl) diphenylphosphine (1q)



1q

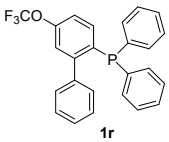
White solid. R_f 0.2 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 – 7.30 (m, 9H), 7.30 – 7.27 (m, 2H), 7.27 – 7.21 (m, 4H), 7.21 – 7.17 (m, 2H), 7.01 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.0, 149.7, 140.5 (d, J = 6.2 Hz), 137.3 (d, J = 11.6 Hz), 135.6, 134.9 (d, J = 9.5 Hz), 134.7, 134.0 (d, J = 20.0 Hz), 130.2 (d, J = 4.7 Hz), 129.6 (d, J = 3.7 Hz), 128.8, 128.7, 128.6, 127.9, 127.8, 127.6.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -18.03.

diphenyl (5-trifluoromethoxyo)-[1,1'-biphenyl]-2-yl) phosphine (1r)



1r

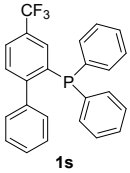
White solid. R_f 0.1 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.37 – 7.27 (m, 9H), 7.21 (m, 7H), 7.15 – 7.10 (m, 1H), 7.08 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.2, 150.0, 149.5, 140.4 (d, J = 5.8 Hz), 137.2 (d, J = 11.5 Hz), 135.8, 134.9 (d, J = 15.7 Hz), 134.0 (d, J = 20.1 Hz), 129.6 (d, J = 3.4 Hz), 128.8, 128.7, 128.6, 127.9, 122.1(d, J = 4.4 Hz), 121.8, 119.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -14.04.

diphenyl (4-trifluoromethyl)-[1,1'-biphenyl]-2-yl) phosphine (1s)



1s

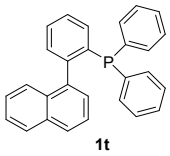
White solid. R_f 0.3 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62 (d, J = 9.2 Hz, 1H), 7.41 (m, 1H), 7.36 – 7.30 (m, 6H), 7.30 – 7.23 (m, 4H), 7.22 (s, 4H), 7.16 – 7.12 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.6, 151.4, 140.5 (d, J = 5.8 Hz), 138.1(d, J = 17.9 Hz), 136.4 (d, J = 11.5 Hz), 134.1, 133.9, 130.5 (d, J = 4.0 Hz), 130.4 (d, J = 3.7 Hz), 129.5 (d, J = 3.6 Hz), 129.0, 128.7 (d, J = 6.9 Hz), 127.9, 125.4 (d, J = 3.6 Hz), 122.8.

^{31}P NMR (162 MHz, $CDCl_3$) δ: -12.27.

1-(2-diphenylphosphinophenyl) naphthalene (1t)



1t

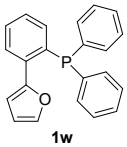
White solid. R_f 0.2 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.83 (d, J = 8.3 Hz, 1H), 7.77 (d, J = 8.4 Hz, 1H), 7.62 – 7.58 (m, 1H), 7.54 (s, 1H), 7.48 – 7.37 (m, 5H), 7.34 – 7.22 (m, 11H), 7.17 – 7.10 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.5, 148.2, 139.3 (d, J = 6.3 Hz), 137.8 (d, J = 11.2 Hz), 136.2 (d, J = 13.8 Hz), 134.3 (d, J = 20.8 Hz), 134.0, 132.8, 132.6, 130.5 (d, J = 4.9 Hz), 129.0 (d, J = 3.9 Hz), 129.0, 128.7, 128.6, 128.5, 128.3, 128.1 (d, J = 3.0 Hz), 127.8, 127.6, 127.3, 126.1 (d, J = 12.0 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: -13.07.

2-(2-diphenylphosphinylphenyl) furan (1w)



1w

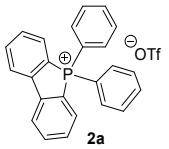
Colorless oil. R_f 0.3 (hexane/DCM = 5 : 1)

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (m, 1H), 7.38 (m, 2H), 7.33 – 7.24 (m, 10H), 7.16 (m, 1H), 6.97 (m, 1H), 6.53 – 6.47 (m, 1H), 6.35 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.8 (d, J = 4.1 Hz), 137.6 (d, J = 11.1 Hz), 136.4 (d, J = 26.2 Hz), 134.8, 134.6 (d, J = 19.3 Hz), 134.2 (d, J = 19.7 Hz), 129.0, 129.0 (d, J = 6.3 Hz), 128.8, 128.5 (d, J = 5.0 Hz), 127.9, 111.6, 111.1 (d, J = 12.5 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: -9.13.

5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2a)



2a

White solid (75.8 mg, 78%). M.p. 124 – 125 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^1H NMR (400 MHz, $CDCl_3$) δ: 8.15 (m, 4H), 7.90 (m, 2H), 7.80 (m, 6H), 7.70 (m, 6H).

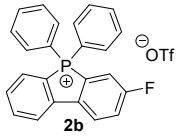
^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.2 (d, J = 19.2 Hz), 136.9, 136.1, 133.5 (d, J = 11.8 Hz), 132.6 (d, J = 9.9 Hz), 131.8 (d, J = 11.9 Hz), 131.0 (d, J = 13.6 Hz), 123.9 (d, J = 10.0 Hz), 120.9 (d, J = 95.1 Hz), 116.3 (d, J = 87.7 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.79.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.02.

Exact Mass (ESI): Calcd for $C_{24}H_{18}P^+$ [M-OTf] $^+$ 337.11406, found 337.11442.

3-fluoro-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2b)



2b

White solid (62.8 mg, 62%). M.p. 252 – 253 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^1H NMR (400 MHz, $CDCl_3$) δ: 8.26 (m, 1H), 8.17 (m, 1H), 8.11 (t, J = 8.7 Hz, 1H), 7.88 (t, J = 7.6 Hz, 1H), 7.84 – 7.75 (m, 7H), 7.73 – 7.63 (m, 5H), 7.58 (t, J = 8.3 Hz, 1H).

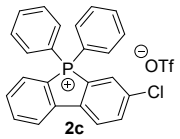
^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.7 (d, J = 16.8 Hz), 162.2 (d, J = 17.4 Hz), 143.0 (d, J = 18.4 Hz), 140.2 (d, J = 18.9 Hz), 136.9, 136.0, 133.1 (d, J = 11.8 Hz), 132.3 (d, J = 10.2 Hz), 131.0 (d, J = 12.4 Hz), 130.8 (d, J = 13.7 Hz), 126.2, 126.1, 126.0, 124.1 (d, J = 22.3 Hz), 123.8 (d, J = 9.9 Hz), 123.4, 122.4, 122.2, 120.6, 119.7, 119.0, 119.0, 118.7 (d, J = 11.2 Hz), 115.7, 114.8.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.20.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.04, -105.73.

Exact Mass (ESI): Calcd for $C_{24}H_{17}FP^+$ [M-OTf] $^+$ 355.10464, found 355.10490.

3-chloro-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2c)



2c

White solid (66.6 mg, 64%). M.p. 151 – 152 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^1H NMR (400 MHz, $CDCl_3$) δ: 8.26 (s, 3H), 8.01 (d, J = 9.6 Hz, 1H), 7.93 (s, 1H), 7.86 (m, J = 6.8 Hz, 7H), 7.74 (m, 5H).

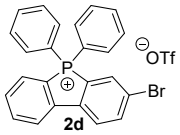
^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.2 (d, J = 18.7 Hz), 142.8 (d, J = 19.1 Hz), 137.4 (d, J = 15.6 Hz), 137.2 (d, J = 5.0 Hz), 136.4 (d, J = 2.9 Hz), 133.5 (d, J = 11.7 Hz), 132.8 (d, J = 10.1 Hz), 131.9 (d, J = 12.2 Hz), 131.5 (d, J = 11.1 Hz), 131.2 (d, J = 13.6 Hz), 129.4, 127.2, 125.8 (d, J = 10.9 Hz), 124.5 (d, J = 9.9 Hz), 123.6, 122.7 (d, J = 4.9 Hz), 121.0, 119.7 (d, J = 55.7 Hz), 115.6 (d, J = 87.6 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.37.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.97.

Exact Mass (ESI): Calcd for $C_{24}H_{17}ClP^+$ [M-OTf] $^+$ 371.07509, found 371.07546.

3-bromo-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2d)



White solid (67.7 mg, 60%). M.p. 219 – 220 °C, R_f 0.4 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27 – 8.15 (m, 2H), 8.14 – 8.07 (m, 2H), 8.01 (d, J = 8.5 Hz, 1H), 7.91 (m, 1H), 7.87 – 7.77 (m, 6H), 7.72 (m, 5H).

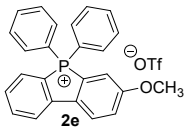
^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.3 (d, J = 8.6 Hz), 143.1 (d, J = 8.1 Hz), 140.0, 137.3, 136.5 (d, J = 2.7 Hz), 134.3 (d, J = 10.7 Hz), 133.5 (d, J = 12.0 Hz), 132.9 (d, J = 10.3 Hz), 132.2 (d, J = 12.1 Hz), 131.2 (d, J = 13.8 Hz), 125.8 (d, J = 10.6 Hz), 125.2 (d, J = 15.1 Hz), 124.4 (d, J = 9.8 Hz), 123.9, 122.8 (d, J = 41.5 Hz), 120.8, 119.6 (d, J = 53.0 Hz), 115.5 (d, J = 87.6 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.32.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.95.

Exact Mass (ESI): Calcd for $C_{24}H_{17}BrP^+$ [M-OTf] $^+$ 415.02458, found 415.02489.

3-methoxy-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2e)



White solid (60.9 mg, 59%). M.p. 162 – 163 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.03 (m, 3H), 7.82 (m, 3.9 Hz, 7H), 7.72 – 7.65 (m, 4H), 7.63 – 7.55 (m, 2H), 7.38 (m, 1H), 3.93 (s, 3H).

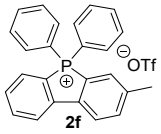
^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.2 (d, J = 15.3 Hz), 144.5 (d, J = 19.2 Hz), 136.9, 136.4, 136.3, 136.0 (d, J = 2.9 Hz), 133.5 (d, J = 11.7 Hz), 132.3 (d, J = 10.0 Hz), 131.0 (d, J = 13.6 Hz), 130.3 (d, J = 12.0 Hz), 125.3 (d, J = 12.0 Hz), 123.2 (d, J = 10.1 Hz), 122.4 (d, J = 41.3 Hz), 120.7, 119.6 (d, J = 38.4 Hz), 116.8 (d, J = 12.2 Hz), 116.3 (d, J = 63.6 Hz), 56.7.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.08.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.02.

Exact Mass (ESI): Calcd for $C_{25}H_{20}OP^+$ [M-OTf] $^+$ 367.12463, found 367.12423.

3-methyl-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2f)



White solid (70.0 mg, 70%). M.p. 166 – 167 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.09 (d, J = 8.7 Hz, 2H), 8.02 (d, J = 4.5 Hz, 1H), 7.88 (t, J = 9.4 Hz, 2H), 7.84 – 7.75 (m, 6H), 7.74 – 7.64 (m, 6H), 2.51 (s, 3H).

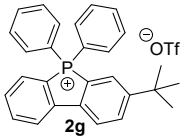
^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.3 (d, J = 19.5 Hz), 142.7 (d, J = 11.8 Hz), 141.6 (d, J = 19.3 Hz), 137.8, 136.9, 136.0 (d, J = 2.7 Hz), 133.4 (d, J = 11.6 Hz), 132.5 (d, J = 6.7 Hz), 132.4 (d, J = 6.7 Hz), 131.2 (d, J = 12.2 Hz), 131.0 (d, J = 13.6 Hz), 125.8, 123.8 (d, J = 10.6 Hz), 123.7 (d, J = 10.1 Hz), 122.6, 121.2 (d, J = 30.4 Hz), 120.5, 119.8 (d, J = 72.6 Hz), 116.5 (d, J = 87.4 Hz), 21.6.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.63.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.96.

Exact Mass (ESI): Calcd for $C_{25}H_{20}P^+$ [M-OTf] $^+$ 351.12971, found 351.13052.

3-(tert-butyl)-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2g)



White solid (82.4 mg, 76%). M.p. 93 – 94 °C, R_f 0.4 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (m, 3H), 7.95 (d, J = 2.8 Hz, 1H), 7.93 (d, J = 1.7 Hz, 1H), 7.91 – 7.76 (m, 7H), 7.75 – 7.66 (m, 5H), 1.38 (s, 9H).

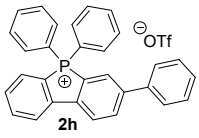
^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.8 (d, J = 10.6 Hz), 144.1 (d, J = 19.4 Hz), 141.9 (d, J = 19.6 Hz), 136.9, 136.1, 134.5, 133.4 (d, J = 11.5 Hz), 132.4 (d, J = 9.8 Hz), 131.3 (d, J = 12.4 Hz), 131.1 (d, J = 13.4 Hz), 128.3 (d, J = 10.0 Hz), 123.9 (q, J = 10.2 Hz), 122.6, 121.4 (d, J = 4.7 Hz), 119.9 (d, J = 96.3 Hz), 116.5 (d, J = 87.2 Hz), 35.6, 31.2.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.94.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.92.

Exact Mass (ESI): Calcd for $C_{28}H_{26}P^+$ [M-OTf] $^+$ 393.17666, found 393.17669.

3,5,5-triphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2h)



White solid (78.7 mg, 70%). M.p. 234 – 235 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (s, 1H), 8.20 (d, J = 15.5 Hz, 2H), 8.13 (s, 2H), 7.96 – 7.77 (m, 7H), 7.76 – 7.59 (m, 7H), 7.47 (d, J = 5.6 Hz, 2H), 7.39 (s, 1H).

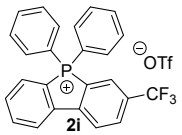
^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.7 (d, J = 11.6 Hz), 143.9 (d, J = 19.2 Hz), 143.0 (d, J = 19.3 Hz), 137.9, 137.0, 136.2 (d, J = 2.8 Hz), 135.4, 133.5 (d, J = 11.9 Hz), 132.7 (d, J = 10.0 Hz), 131.7 (d, J = 11.9 Hz), 131.1 (d, J = 13.6 Hz), 130.1 (d, J = 10.3 Hz), 129.3 (d, J = 30.0 Hz), 127.2, 124.6 (d, J = 10.4 Hz), 124.1 (d, J = 9.8 Hz), 122.6 (d, J = 10.3 Hz), 121.5 (d, J = 14.7 Hz), 119.9 (d, J = 107.3 Hz), 116.2 (d, J = 87.3 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.05.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.91.

Exact Mass (ESI): Calcd for $C_{30}H_{22}P^+$ [M-OTf] $^+$ 413.14536, found 413.14668.

5,5-diphenyl-3-(trifluoromethyl)-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2i)



White solid (29.9/57.6 mg, 27/57%). M.p. 177 – 178 °C, R_f 0.4 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.47 (d, J = 9.8 Hz, 1H), 8.34 (d, J = 19.9 Hz, 1H), 8.28 (t, J = 9.7 Hz, 1H), 8.19 (q, J = 9.1 Hz, 2H), 7.97 (t, J = 7.6 Hz, 1H), 7.89 – 7.77 (m, 7H), 7.73 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8 (d, J = 19.1 Hz), 142.6 (d, J = 18.4 Hz), 137.4, 136.6 (d, J = 2.8 Hz), 134.2, 133.8, 133.8, 133.6, 133.5, 133.2 (d, J = 21.5 Hz), 133.0, 132.9 (d, J = 3.8 Hz), 131.4, 131.2, 128.5 (d, J = 7.8 Hz), 125.4 (q, J = 4.0 Hz), 124.3, 123.1, 122.6, 122.1, 121.7, 121.6, 120.8, 119.4, 115.2

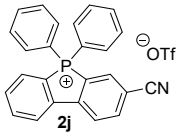
(d, J = 87.9 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 23.74.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -62.48, -78.06.

Exact Mass (ESI): Calcd for $C_{25}H_{17}F_3P^+$ [M-OTf] $^+$ 405.10145, found 405.10097.

3-cyano-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2j)



2j

White solid (51.1 mg, 50%). M.p. 218 – 219 °C, R_f 0.2 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, CD_3OD) δ: 8.79 (m, 1H), 8.48 (m, 1H), 8.42 (m, 1H), 8.34 (d, J = 7.9 Hz, 1H), 8.30 (d, J = 7.4 Hz, 1H), 8.04 (t, J = 7.7 Hz, 1H), 7.92 (dd, J = 14.9, 7.6 Hz, 6H), 7.83 (m, 1H), 7.75 (m, 4H).

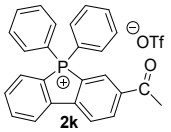
^{13}C NMR (100 MHz, CD_3OD) δ: 148.0 (d, J = 19.0 Hz), 142.9 (d, J = 18.3 Hz), 140.1, 137.0, 136.1, 133.6 (d, J = 12.0 Hz), 132.5 (d, J = 12.0 Hz), 130.7 (d, J = 13.9 Hz), 125.1, 124.6, 123.8, 122.6 (d, J = 50.3 Hz), 121.7 (d, J = 65.9 Hz), 116.4 (d, J = 54.1 Hz), 115.3, 114.8 (d, J = 14.1 Hz).

^{31}P NMR (162 MHz, CD_3OD) δ: 23.84.

^{19}F NMR (376 MHz, CD_3OD) δ: -76.01.

Exact Mass (ESI): Calcd for $C_{25}H_{17}NP^+$ [M-OTf]$^+$ 362.10931, found 362.11211.

3-acetyl-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2k)



2k

White solid (63.4 mg, 60%). M.p. 97 – 98 °C, R_f 0.2 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.77 (d, J = 9.6 Hz, 1H), 8.53 (s, 1H), 8.42 (s, 1H), 8.33 (s, 1H), 8.21 (s, 1H), 7.97 (s, 1H), 7.84 (m, 7H), 7.72 (m, 4H), 2.75 (s, 3H).

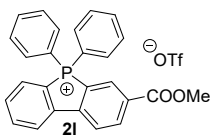
^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.5, 147.7 (d, J = 20.0 Hz), 143.1 (d, J = 18.6 Hz), 139.2 (d, J = 10.8 Hz), 137.2, 136.4, 133.8 (d, J = 11.9 Hz), 133.0, 132.7 (d, J = 11.9 Hz), 132.3 (d, J = 10.5 Hz), 131.2 (d, J = 13.7 Hz), 125.1 (d, J = 63.9 Hz), 122.5 (d, J = 33.4 Hz), 121.6 (d, J = 32.9 Hz), 119.4, 115.7 (d, J = 87.7 Hz), 27.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.98.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.00.

Exact Mass (ESI): Calcd for $C_{26}H_{20}OP^+$ [M-OTf] $^+$ 379.12463, found 379.13001.

3-(methoxycarbonyl)-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2l)



2l

White solid (79.4 mg, 73%). M.p. 160 – 161 °C, R_f 0.2 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.57 (d, J = 10.1 Hz, 1H), 8.52 (d, J = 7.9 Hz, 1H), 8.42 – 8.27 (m, 2H), 8.27 – 8.17 (m, 1H), 7.94 (t, J = 7.3 Hz, 1H), 7.78 (m, 7H), 7.70 (m, 4H), 3.93 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.8, 148.1 (d, J = 19.5 Hz), 142.9 (d, J = 18.6 Hz), 138.0, 137.3, 136.4 (d, J = 2.5 Hz), 133.5 (d, J = 11.8 Hz), 132.9, 132.9, 132.8, 131.2 (d, J = 13.7 Hz), 125.8, 125.3 (d, J =

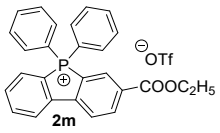
9.7 Hz), 124.6 (d, *J* = 9.8 Hz), 122.6, 122.1 (d, *J* = 28.8 Hz), 121.3, 120.2 (d, *J* = 163.7 Hz), 115.6 (d, *J* = 87.7 Hz), 53.1.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.84.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.00.

Exact Mass (ESI): Calcd for $C_{26}H_{20}O_2P^+$ [M-OTf] $^+$ 395.11954, found 395.11921.

3-(ethoxycarbonyl)-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2m)



White solid (65.8 mg, 59%). M.p. 90 – 91 °C, R_f 0.2 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.62 – 8.51 (m, 2H), 8.39 – 8.24 (m, 3H), 7.96 (t, *J* = 7.7 Hz, 1H), 7.90 – 7.76 (m, 7H), 7.72 (m, 4H), 4.43 (q, *J* = 8.9 Hz, 2H), 1.41 (t, *J* = 8.9, 3H).

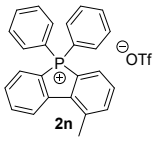
^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.4, 148.2 (d, *J* = 19.6 Hz), 142.8 (d, *J* = 17.0 Hz), 137.9, 137.2, 136.4, 133.6 (d, *J* = 11.8 Hz), 133.2 (d, *J* = 11.5 Hz), 133.0, 132.8 (d, *J* = 11.4 Hz), 131.2 (d, *J* = 13.7 Hz), 125.1, 124.5, 122.6, 122.2 (d, *J* = 24.1 Hz), 121.3 (d, *J* = 24.7 Hz), 119.4, 115.6 (d, *J* = 87.8 Hz), 62.4, 14.4.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.95.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.02.

Exact Mass (ESI): Calcd for $C_{27}H_{22}O_2P^+$ [M-OTf] $^+$ 409.13519, found 409.13766.

1-methyl-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2n)



White solid (51.0 mg, 51%). M.p. 148 – 149 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (m, 1H), 8.15 (m, 1H), 7.97 – 7.89 (m, 2H), 7.82 – 7.75 (m, 5H), 7.74 (d, *J* = 1.2 Hz, 1H), 7.71 (dd, *J* = 7.9, 4.2 Hz, 1H), 7.67 (m, 5H), 7.60 (m, 1H), 2.84 (s, 3H).

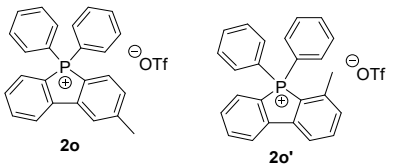
^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.4 (d, *J* = 19.4 Hz), 141.9 (d, *J* = 19.1 Hz), 140.2 (d, *J* = 1.4 Hz), 137.8 (d, *J* = 9.9 Hz), 136.8 (d, *J* = 1.4 Hz), 136.0 (d, *J* = 2.8 Hz), 133.4 (d, *J* = 11.6 Hz), 132.9 (d, *J* = 9.7 Hz), 131.0, 130.9 (d, *J* = 4.2 Hz), 130.7, 130.4 (d, *J* = 10.2 Hz), 127.6 (d, *J* = 10.2 Hz), 122.6, 121.6 (d, *J* = 33.4 Hz), 120.7 (d, *J* = 33.2 Hz), 119.4, 116.5 (d, *J* = 88.1 Hz), 22.8.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 20.88.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.96.

Exact Mass (ESI): Calcd for $C_{25}H_{20}P^+$ [M-OTf] $^+$ 351.12971, found 351.12975.

2-methyl-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2o)
4-methyl-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2o')



White solid (81.0 mg, 81%). M.p. 144 – 145 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.12 (m, 4H), 8.05 – 8.00 (m, 1H), 8.00 – 7.92 (m, 4H), 7.91 – 7.80 (m, 7H), 7.80 – 7.69 (m, 18H), 7.69 – 7.58 (m, 8H), 7.51 (m, 1H), 7.42 (m, 1H), 2.54 (s, 3.93H, **2o'**), 2.37 (s, 3H, **2o**).

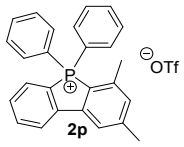
^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.7, 145.0 (d, J = 20.2 Hz), 144.5, 144.3 (d, J = 4.7 Hz), 144.2, 144.1 (d, J = 4.2 Hz), 143.9 (d, J = 19.3 Hz), 137.6, 136.8, 136.4 (d, J = 2.9 Hz), 136.0 (d, J = 2.8 Hz), 1336 (d, J = 11.7 Hz), 133.3 (d, J = 11.6 Hz), 133.0 (d, J = 10.6 Hz), 132.7, 132.5 (d, J = 8.1 Hz), 132.3 (d, J = 6.8 Hz), 132.2, 131.9, 131.7 (d, J = 3.4 Hz), 131.6, 131.5, 131.2 (d, J = 13.5 Hz), 131.0 (d, J = 13.6 Hz), 124.7 (d, J = 10.2 Hz), 124.0, 123.9, 122.7 (d, J = 4.6 Hz), 122.0 (d, J = 9.8 Hz), 121.8 (d, J = 9.9 Hz), 120.9, 119.6 (d, J = 19.4 Hz), 118.7, 117.7, 117.2, 116.5 (d, J = 35.3 Hz), 114.9 (d, J = 86.5 Hz), 22.3, 21.5 (d, J = 4.0 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 24.75 (2o), 22.15 (2o').

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.95.

Exact Mass (ESI): Calcd for $C_{25}H_{20}P^+$ [M-OTf] $^+$ 351.12971, found 351.12975.

2,4-dimethyl-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate（2p）



White solid (87.4 mg, 85%). M.p. 201 – 202 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.13 (m, 1H), 7.94 (m, 1H), 7.90 – 7.83 (m, 4H), 7.83 – 7.77 (m, 3H), 7.77 – 7.70 (m, 5H), 7.64 (m, 1H), 7.27 (d, J = 5.5 Hz, 1H), 2.56 (s, 3H), 2.36 (s, 3H).

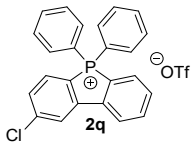
^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.4 (d, J = 1.5 Hz), 145.2 (d, J = 20.5 Hz), 144.0 (d, J = 13.7 Hz), 143.8 (d, J = 4.2 Hz), 136.6, 136.2 (d, J = 2.8 Hz), 133.8 (d, J = 10.9 Hz), 133.5 (d, J = 11.8 Hz), 131.7, 131.6 (d, J = 9.7 Hz), 131.1 (d, J = 13.5 Hz), 125.8, 123.7 (d, J = 9.8 Hz), 123.2, 122.8 (d, J = 10.2 Hz), 122.4 (d, J = 40.5 Hz), 119.4, 116.0 (d, J = 39.8 Hz), 115.0 (d, J = 31.1 Hz), 22.1, 21.4 (d, J = 3.8 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 24.12.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.95.

Exact Mass (ESI): Calcd for $C_{26}H_{22}P^+$ [M-OTf] $^+$ 365.14536, found 365.14524.

2-chloro-5,5-diphenyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2q)



White solid (65.5 mg, 63%). M.p. 130 – 131 °C, R_f 0.4 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.23 (t, J = 8.8 Hz, 1H), 8.19 – 8.09 (m, 2H), 8.07 (s, 1H), 7.93 (t, J = 7.5 Hz, 1H), 7.85 – 7.72 (m, 7H), 7.68 (m, 5H).

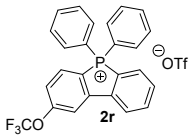
^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.8 (d, J = 20.5 Hz), 144.1, 142.9 (d, J = 18.6 Hz), 137.0, 136.2 (d, J = 2.7 Hz), 134.0 (d, J = 10.9 Hz), 133.5 (d, J = 11.9 Hz), 132.8 (d, J = 9.8 Hz), 132.4 (d, J = 11.8 Hz), 132.0 (d, J = 12.5 Hz), 131.1 (d, J = 13.6 Hz), 124.2 (d, J = 11.0 Hz), 122.6, 121.9, 121.0, 119.1 (d, J = 97.0 Hz), 115.9 (d, J = 88.1 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.57.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.03.

Exact Mass (ESI): Calcd for $C_{24}H_{17}ClP^+$ [M-OTf] $^+$ 371.07509, found 371.07817.

5,5-diphenyl-2-(trifluoromethoxy)-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2r)



White solid (71.8 mg, 63%). M.p. 151 – 152 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.41 (t, J = 8.8 Hz, 1H), 8.16 (dd, J = 7.9 Hz, 2H), 8.02 – 7.92 (m, 1H), 7.90 – 7.74 (m, 8H), 7.68 (d, J = 3.0 Hz, 4H), 7.54 (d, J = 7.0 Hz, 1H).

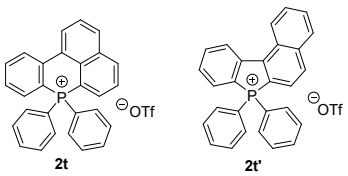
^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.8, 146.9, 142.8, 137.1, 136.2, 135.2, 133.5 (d, J = 11.8 Hz), 132.7 (q, J = 10.8 Hz), 131.1 (d, J = 13.6 Hz), 124.3, 123.1, 122.1, 121.2, 118.9 (d, J = 97.6 Hz), 116.2, 115.5 (d, J = 29.5 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 22.31.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -57.24, -78.10.

Exact Mass (ESI): Calcd for $C_{25}H_{17}F_3OP^+$ [M-OTf] $^+$ 421.09636, found 421.09607.

7,7-diphenyl-7H-benzo[kl]acridophosphin-7-ium trifluoromethanesulfonate (2t)
7,7-diphenyl-7H-dibenzo[b,e]phosphindol-7-ium trifluoromethanesulfonate (2t')



Yellow solid (71.8 mg, 67%). M.p. 121 – 122 °C, R_f 0.2 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.84 (d, J = 11.7 Hz, 1H), 8.59 (d, J = 3.2 Hz, 1H), 8.47 (dd, J = 8.5, 0.9 Hz, 9H), 8.37 – 8.29 (m, 5H), 8.26 (dd, J = 7.7, 3.2 Hz, 4H), 8.18 – 8.02 (m, 3H), 7.91 (dd, J = 17.3, 9.6 Hz, 3H), 7.87 – 7.78 (m, 14H), 7.76 (dd, J = 10.1, 3.6 Hz, 1H), 7.69 (td, J = 7.7, 3.6 Hz, 14H), 7.60 (dd, J = 15.7, 5.3 Hz, 6H).

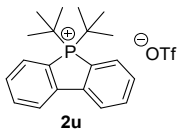
^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.2 (d, J = 18.8 Hz), 144.7, 143.9 (d, J = 20.2 Hz), 139.0, 137.0, 136.8, 136.3, 135.9, 134.4 (d, J = 9.6 Hz), 133.8 (d, J = 11.8 Hz), 133.5 (d, J = 11.8 Hz), 132.4 (d, J = 9.5 Hz), 132.2, 132.0 (d, J = 11.1 Hz), 131.3, 131.2, 131.0, 130.9, 130.7, 130.2, 129.2, 128.9, 128.5, 124.5, 124.3 (d, J = 5.1 Hz), 124.1 (d, J = 12.9 Hz), 123.5 (d, J = 9.7 Hz), 123.1, 122.7, 120.7 (d, J = 11.3 Hz), 119.5 , 117.7, 116.9, 116.0, 115.1 (d, J = 10.0 Hz).

^{31}P NMR (162 MHz, $CDCl_3$) δ: 24.75, 21.57.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.98.

Exact Mass (ESI): Calcd for $C_{28}H_{20}P^+$ [M-OTf] $^+$ 387.12971, found 387.13144.

5,5-di-tert-butyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2u)



White solid (83.0 mg, 93%). M.p. 124 – 125 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (dd, J = 4.9, 2.5 Hz, 2H), 8.10 – 8.05 (m, 2H), 7.92 – 7.83 (m, 2H), 7.73 (m, 2H), 1.51 (d, J = 3.6 Hz, 9H), 1.47 (d, J = 3.6 Hz, 9H).

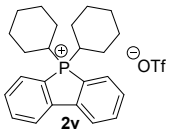
^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.0 (d, J = 12.9 Hz), 136.0, 132.1 (d, J = 8.7 Hz), 131.2 (d, J = 10.4 Hz), 123.7 (d, J = 8.3 Hz), 119.5 (d, J = 77.2 Hz), 36.1 (d, J = 31.9 Hz), 27.0.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 51.87.

^{19}F NMR (376 MHz, $CDCl_3$) δ: -77.91.

Exact Mass (ESI): Calcd for $C_{20}H_{26}P^+$ [M-OTf] $^+$ 297.17666, found 297.17670.

5,5-dicyclohexyl-5H-benzo[b]phosphindol-5-ium trifluoromethanesulfonate (2v)



2v

White solid (87.7 mg, 88%). M.p. 216 – 217 °C, R_f 0.3 (DCM/CH_3OH = 50 : 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.15 (t, J = 7.9 Hz, 2H), 7.97 (dd, J = 7.6, 5.7 Hz, 2H), 7.82 (t, J = 7.6 Hz, 2H), 7.71 – 7.56 (m, 2H), 3.51 – 3.29 (m, 2H), 1.91 (s, 4H), 1.72 (d, J = 23.0 Hz, 6H), 1.58 – 1.40 (m, 4H), 1.28 – 1.05 (m, 6H).

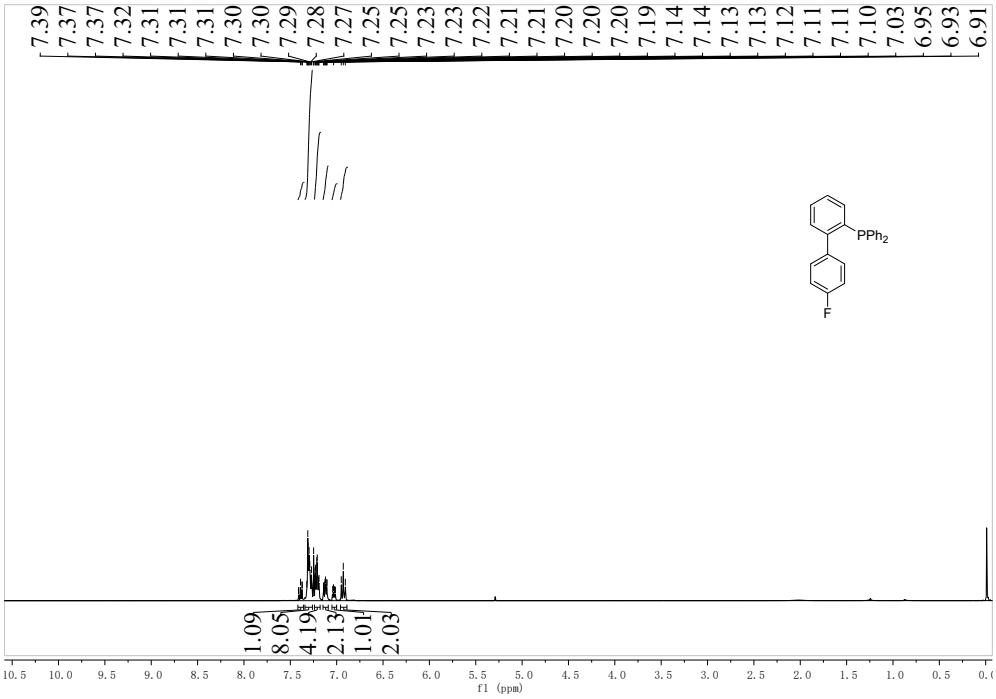
^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.2, 135.6, 132.9 (d, J = 9.4 Hz), 130.8 (d, J = 10.9 Hz), 122.9 (d, J = 8.5 Hz), 118.4 (d, J = 81.5 Hz), 30.6 (d, J = 39.2 Hz), 26.1, 25.6 (d, J = 14.1 Hz), 25.2.

^{31}P NMR (162 MHz, $CDCl_3$) δ: 43.10.

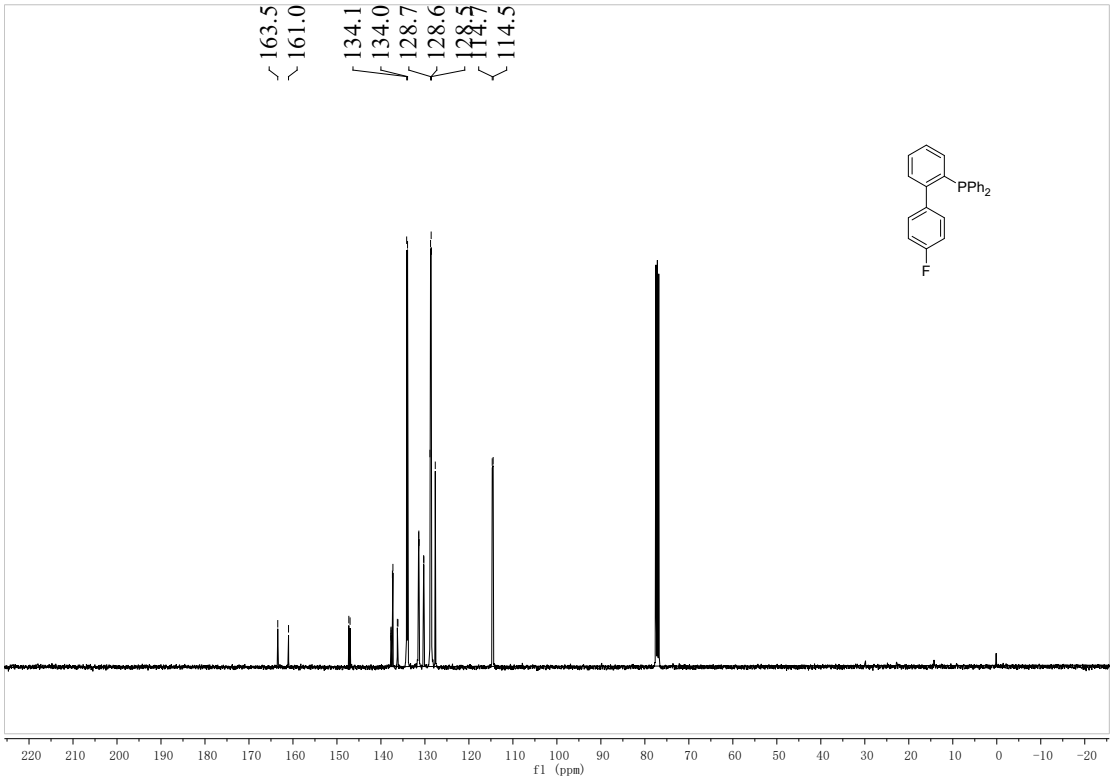
^{19}F NMR (376 MHz, $CDCl_3$) δ: -78.02.

Exact Mass (ESI): Calcd for $C_{24}H_{30}P^+$ [M-OTf] $^+$ 349.20796, found 349.20940.

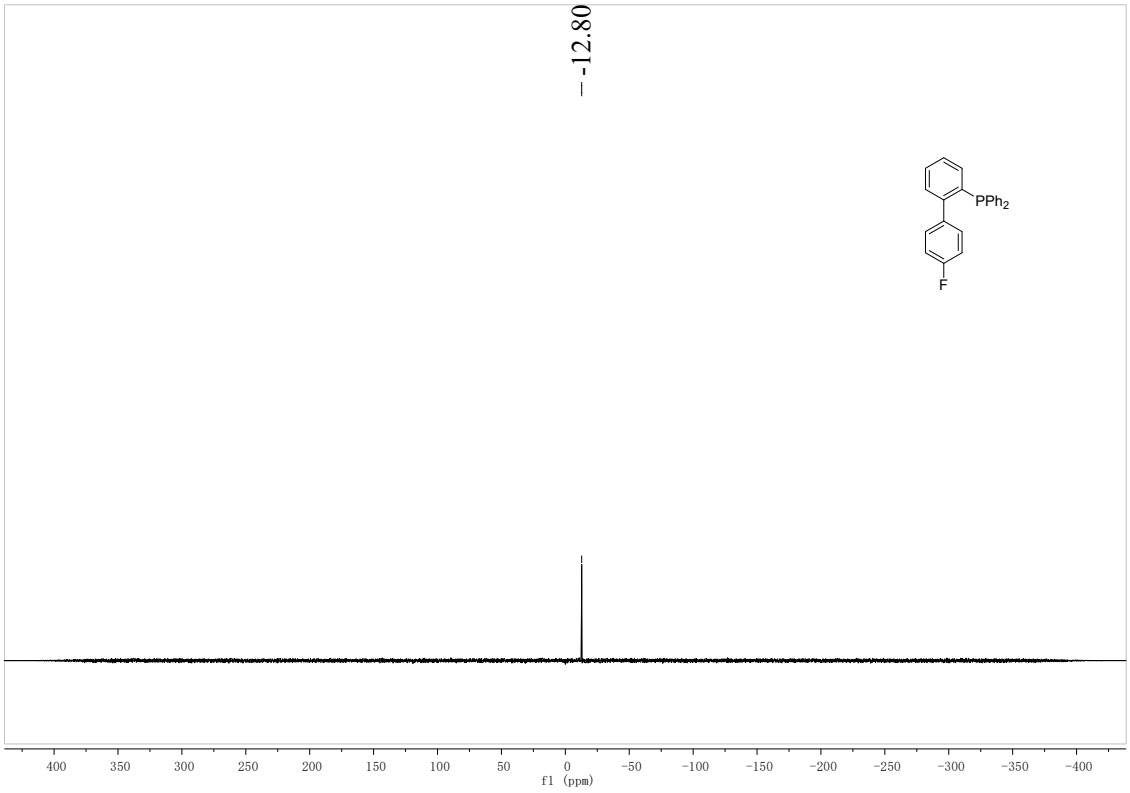
^{1}H NMR spectrum of 1b



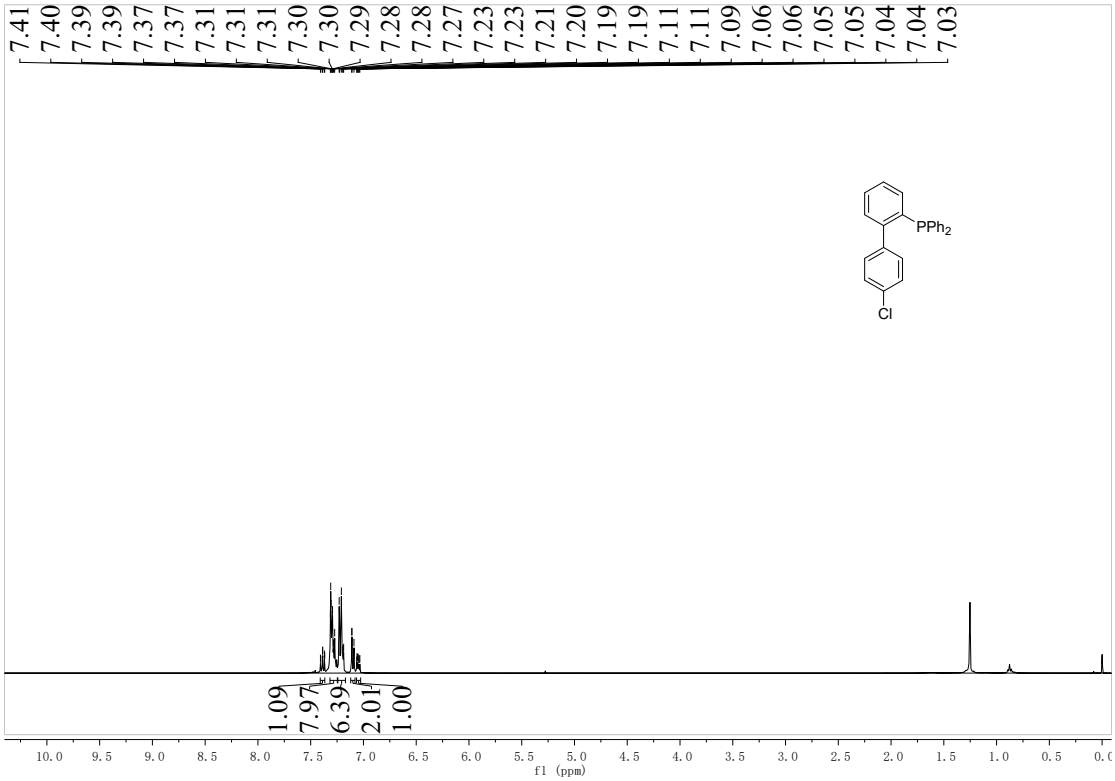
^{13}C NMR spectrum of 1b



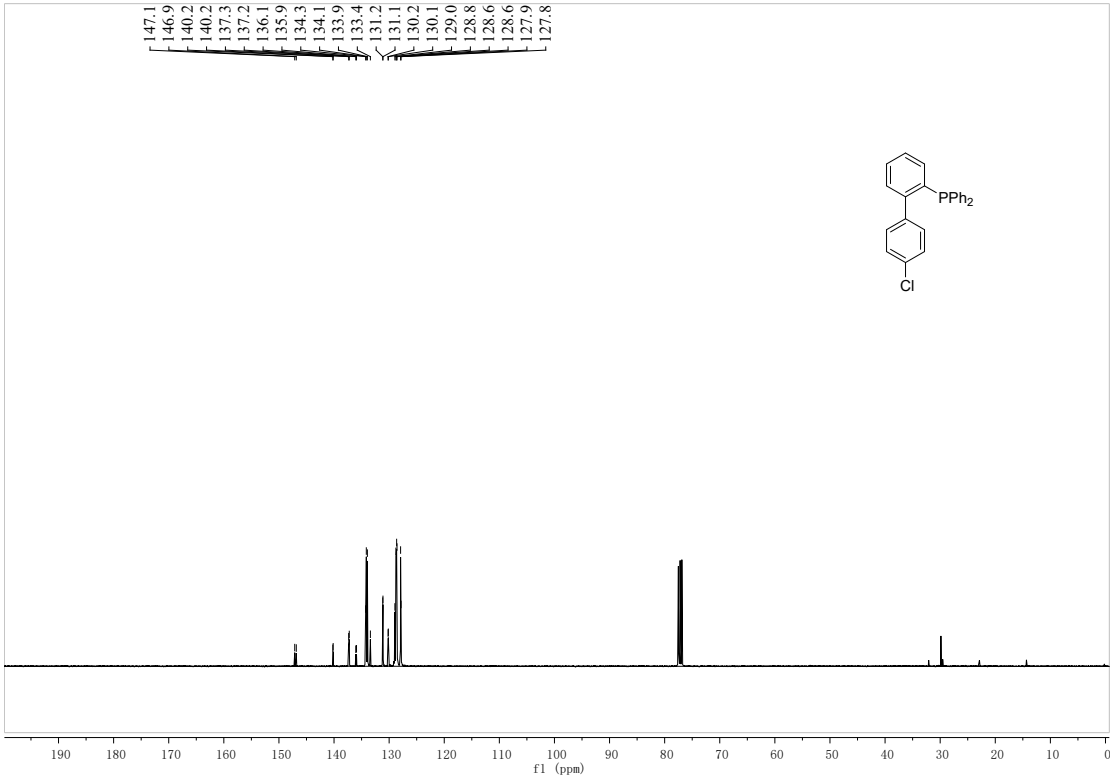
^{31}P NMR spectrum of 1b



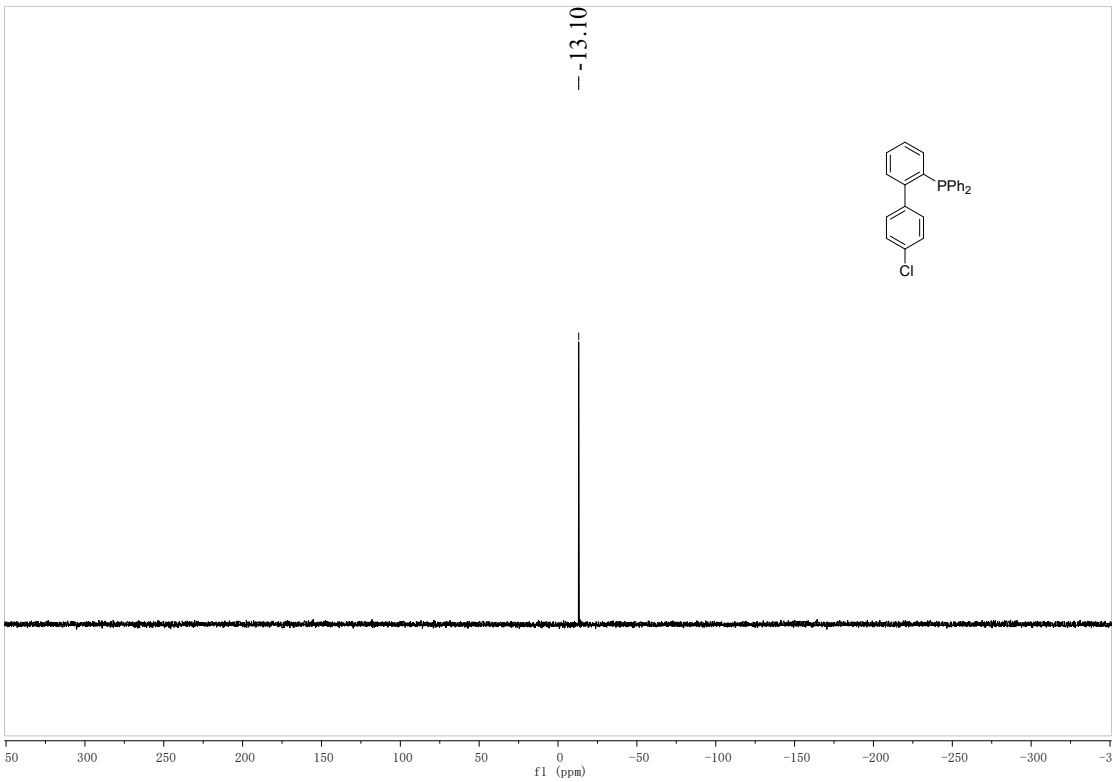
^{1}H NMR spectrum of 1c



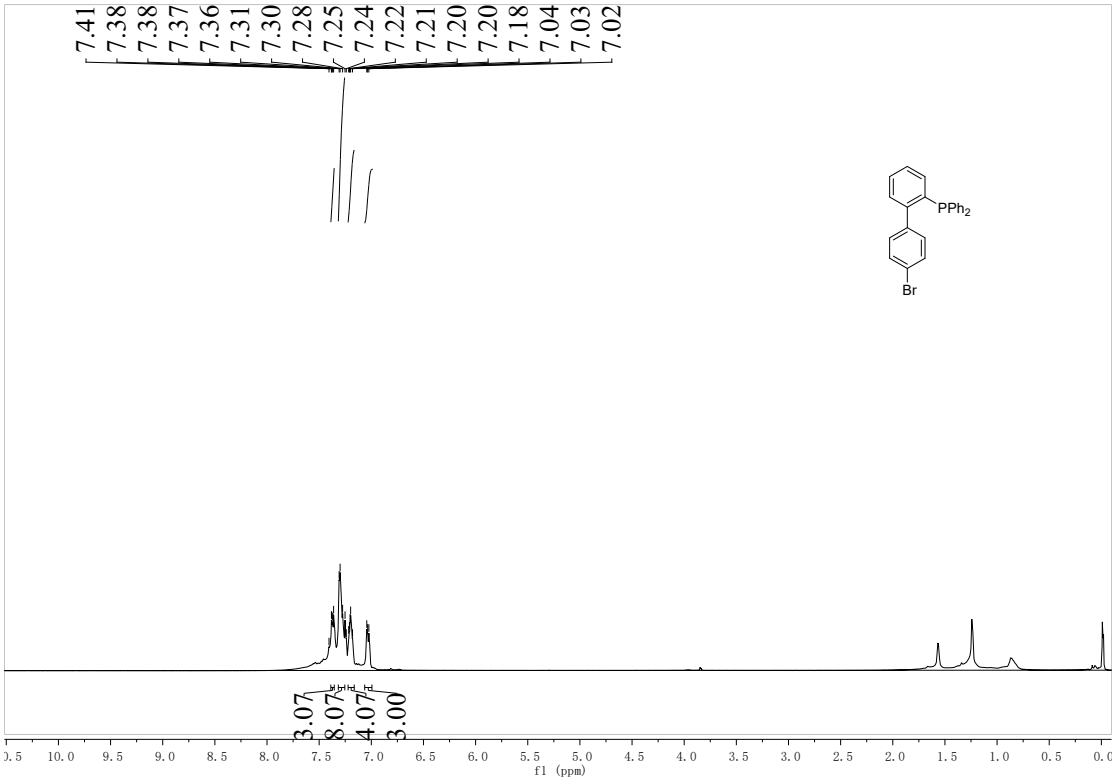
^{13}C NMR spectrum of 1c



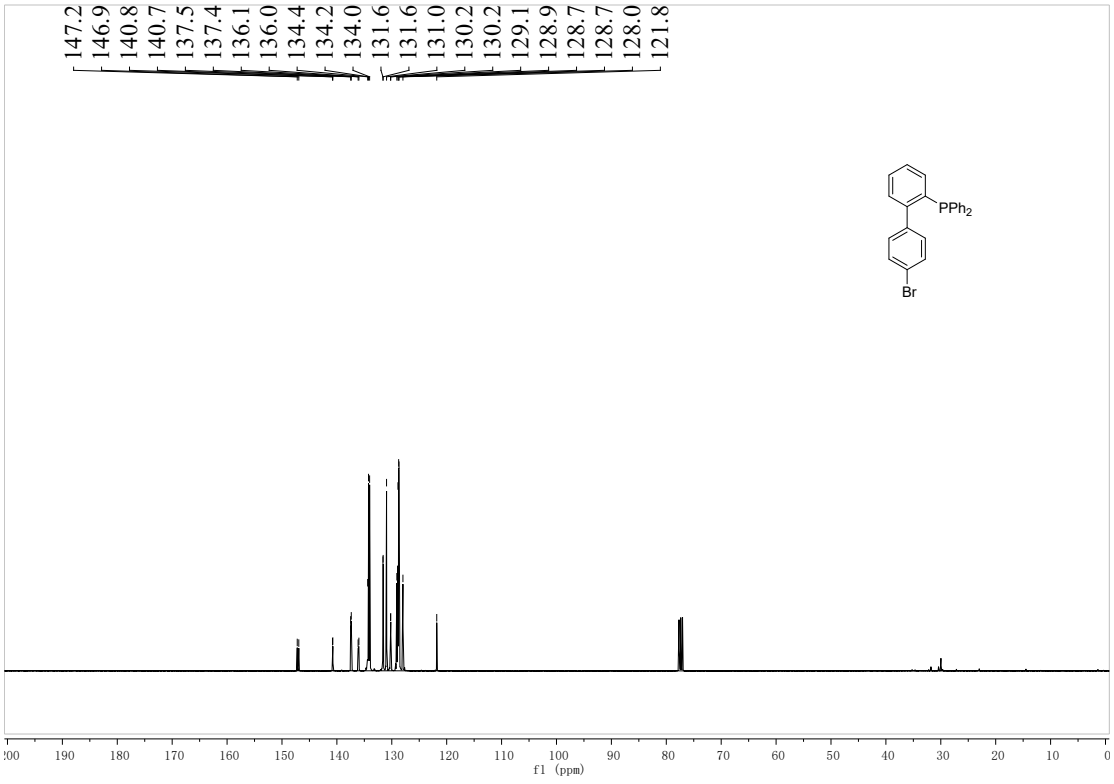
^{31}P NMR spectrum of 1c



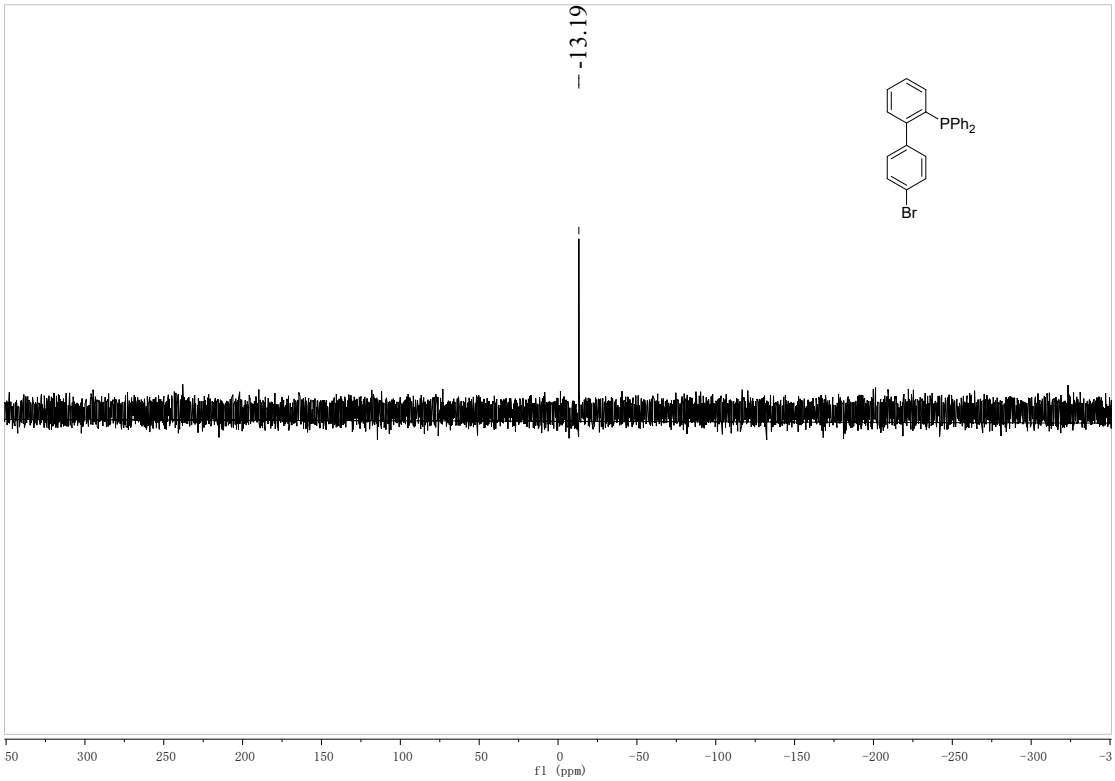
^{1}H NMR spectrum of 1d



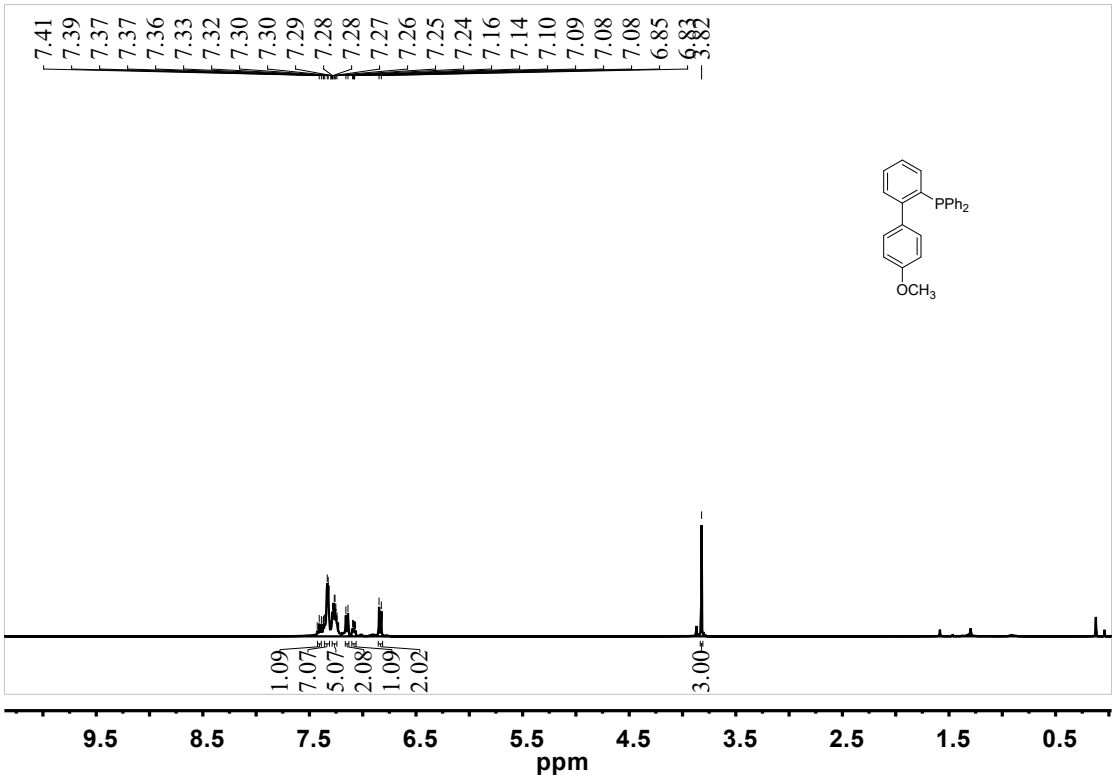
^{13}C NMR spectrum of 1d



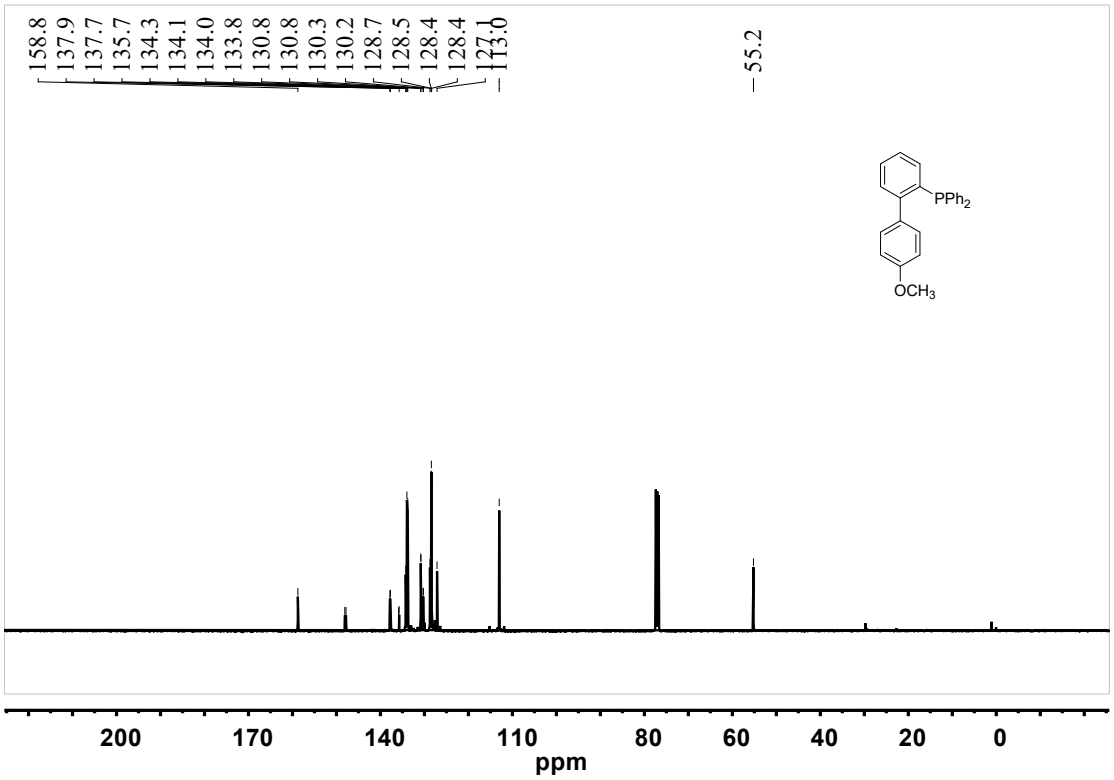
^{31}P NMR spectrum of 1d



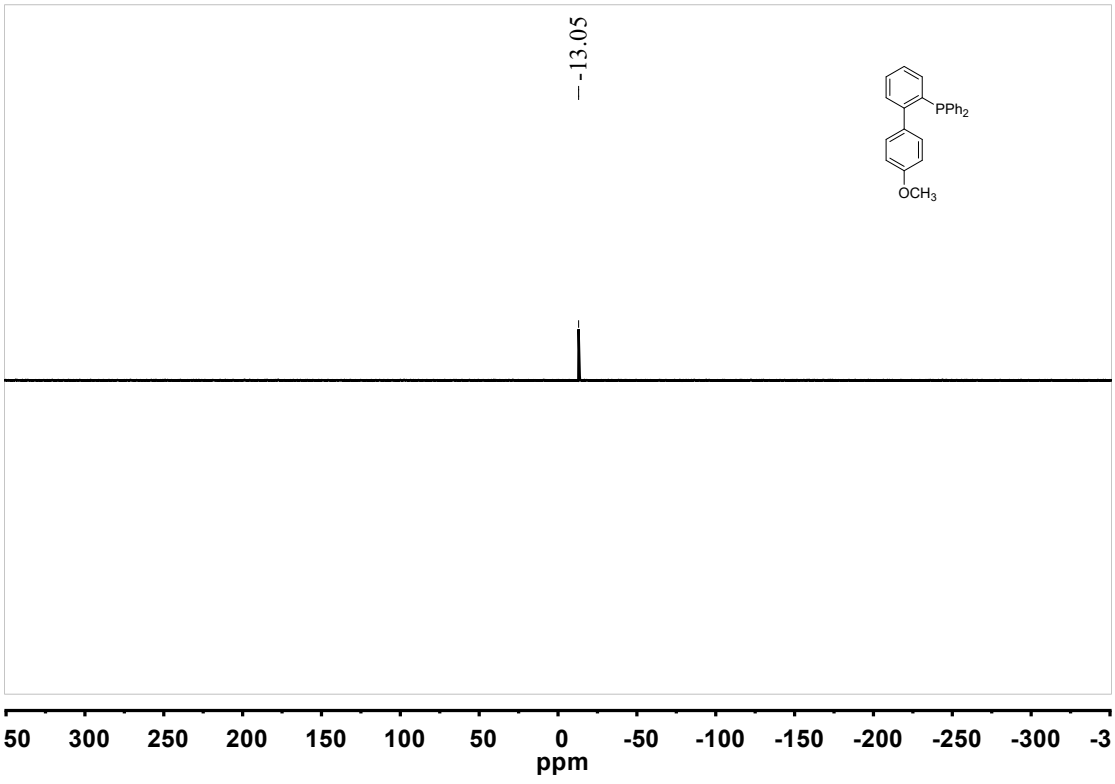
^{1}H NMR spectrum of 1e



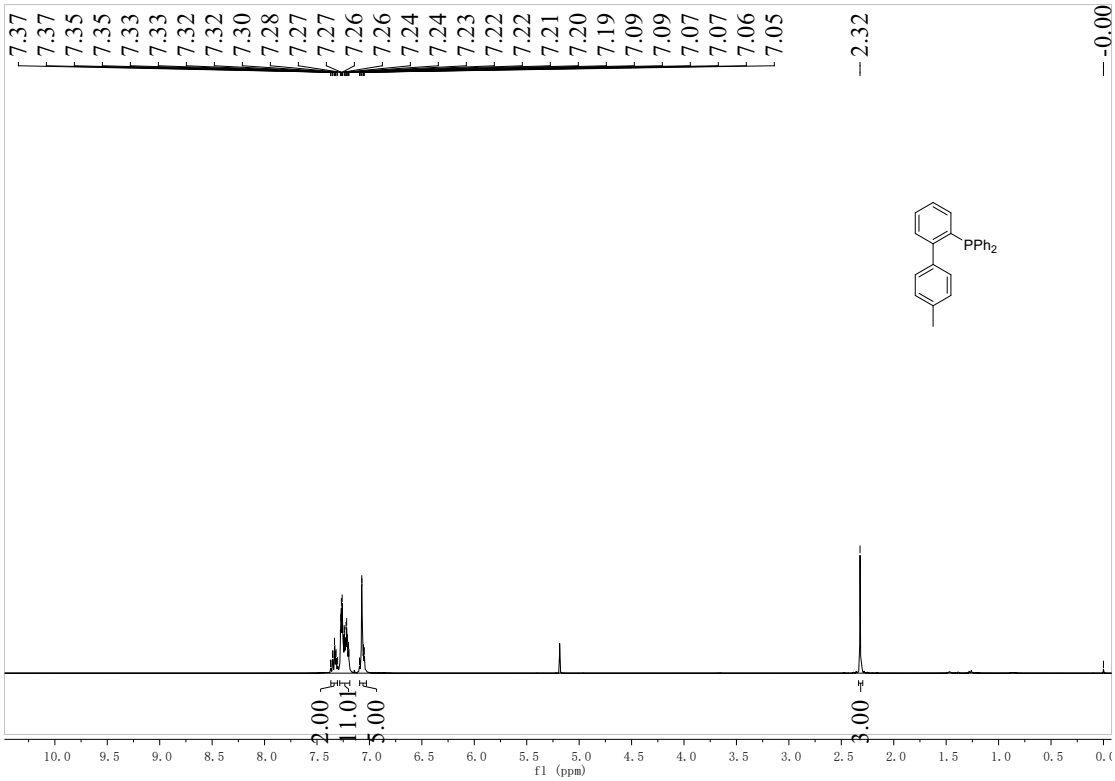
^{13}C NMR spectrum of 1e



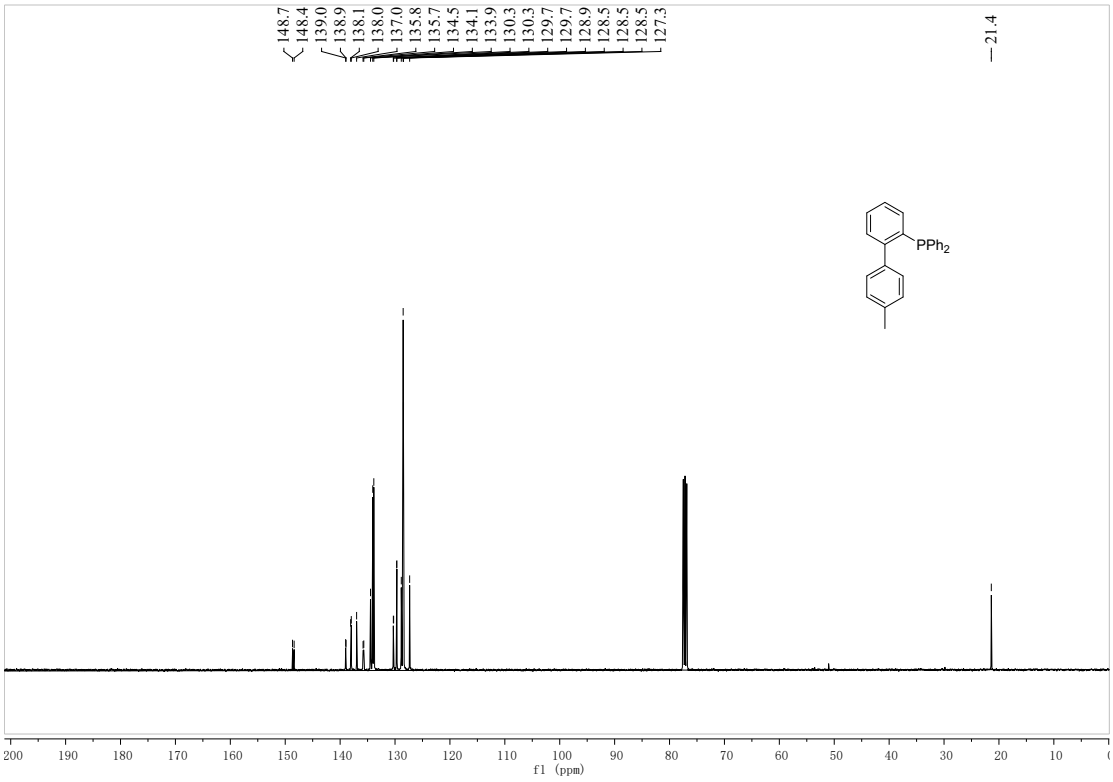
^{31}P NMR spectrum of 1e



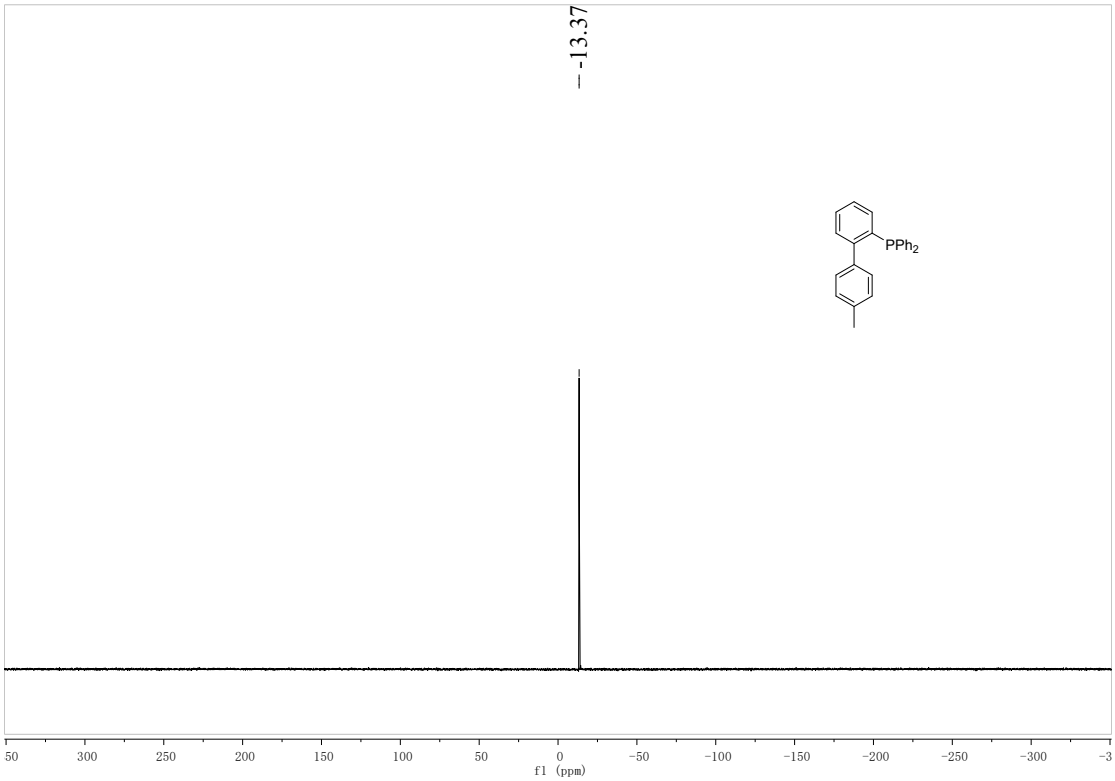
^{1}H NMR spectrum of 1f



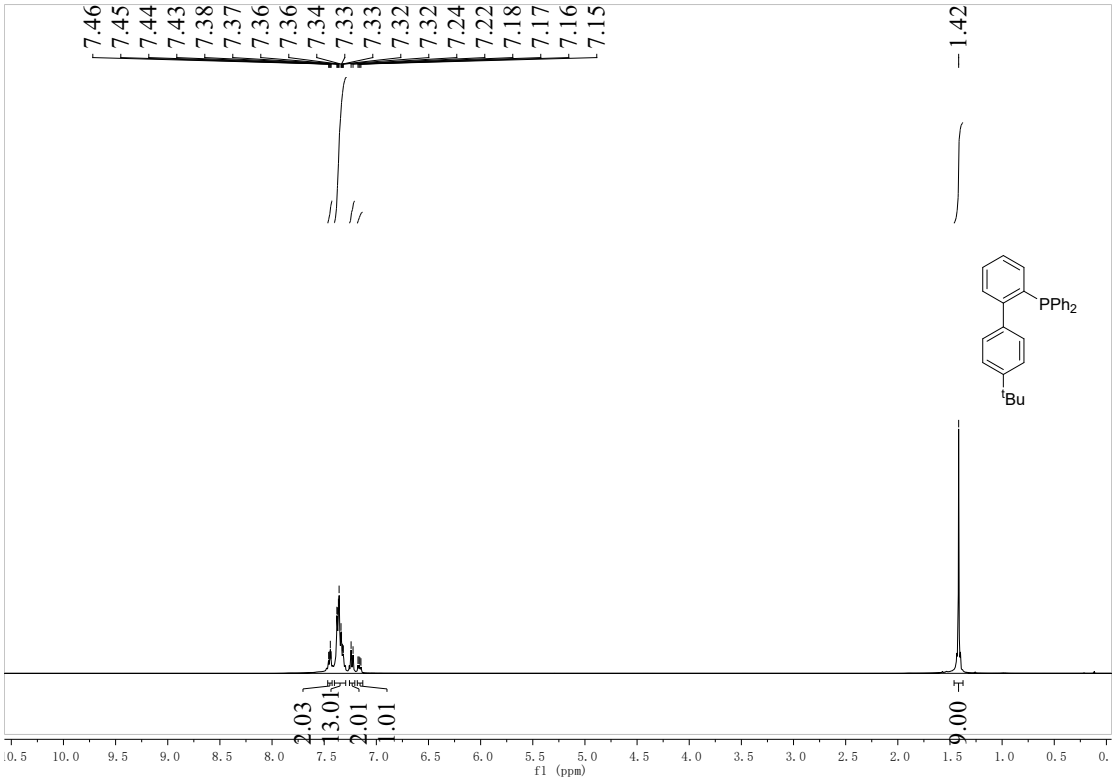
^{13}C NMR spectrum of 1f



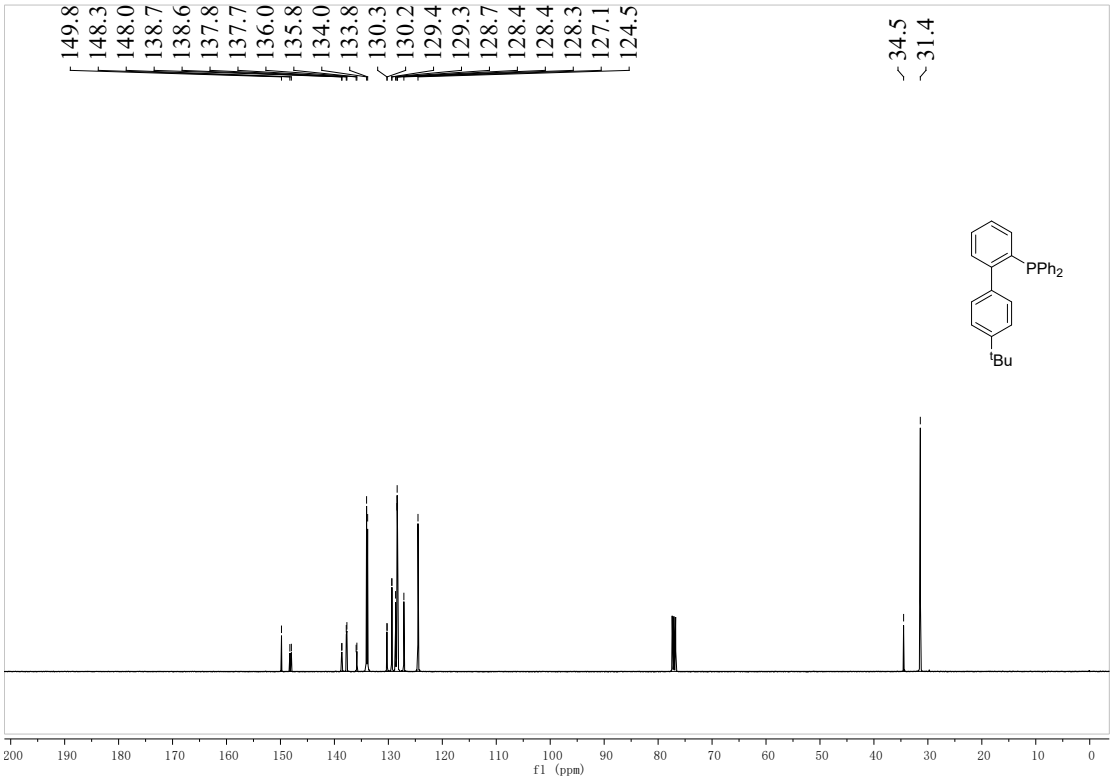
^{31}P NMR spectrum of 1f



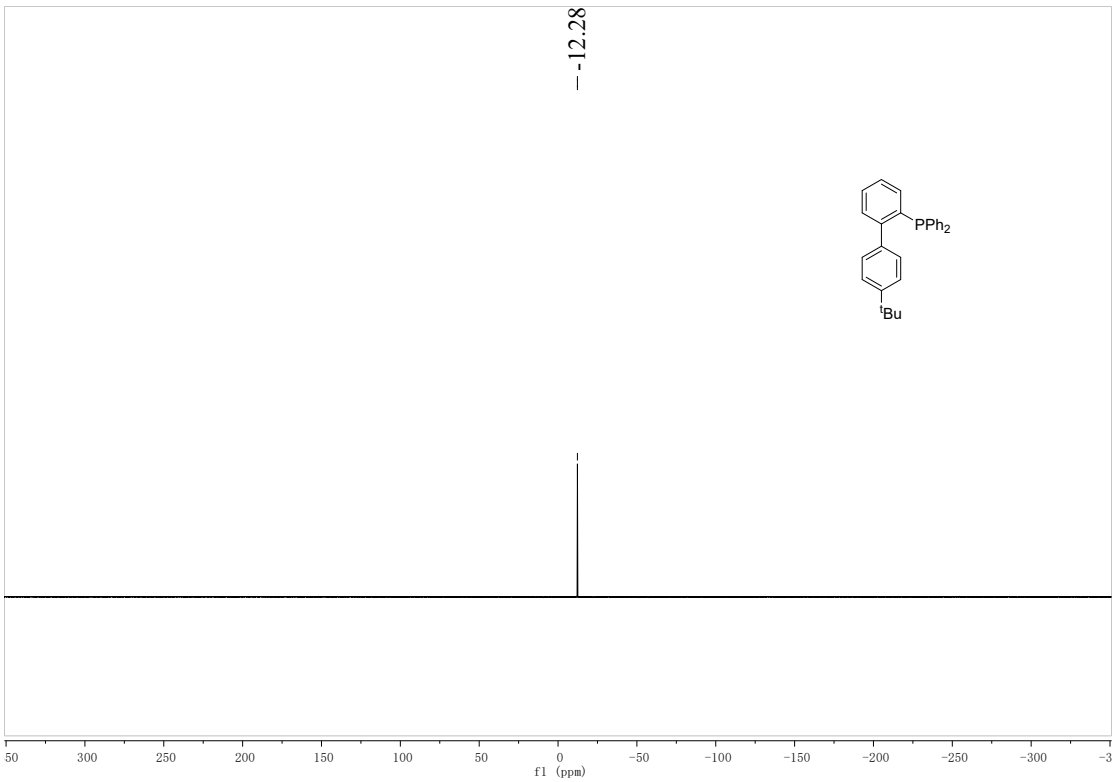
^{1}H NMR spectrum of 1g



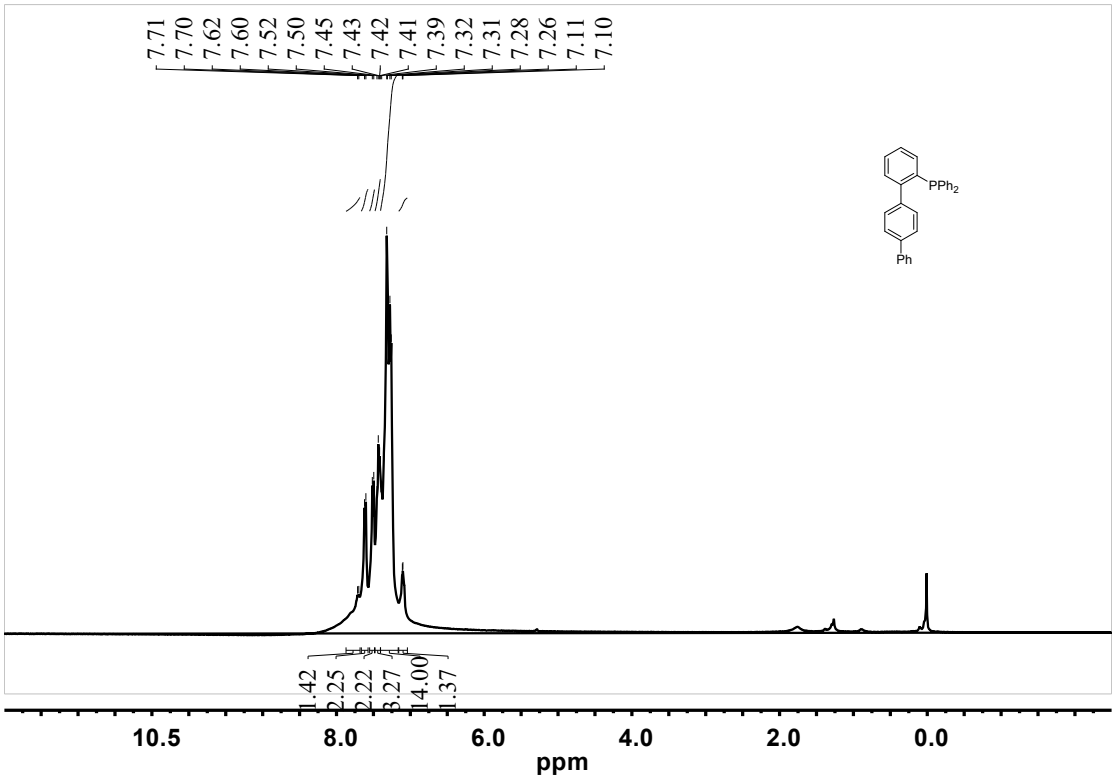
^{13}C NMR spectrum of 1g



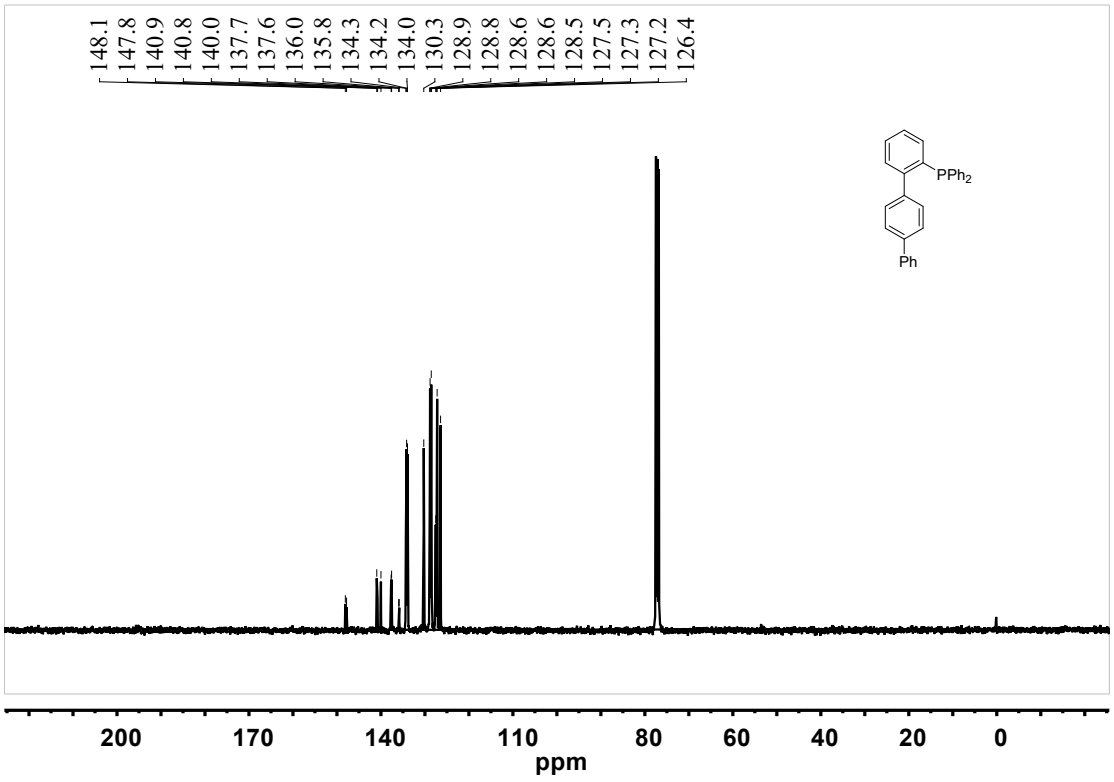
^{31}P NMR spectrum of 1g



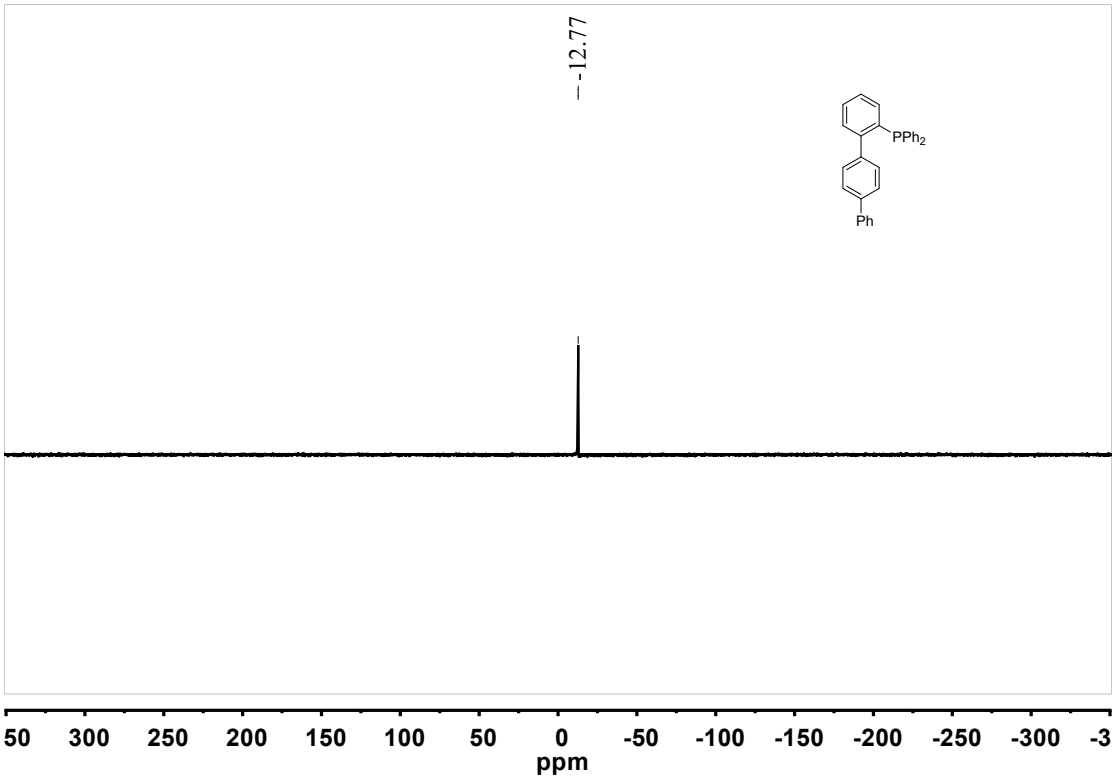
^{1}H NMR spectrum of 1h



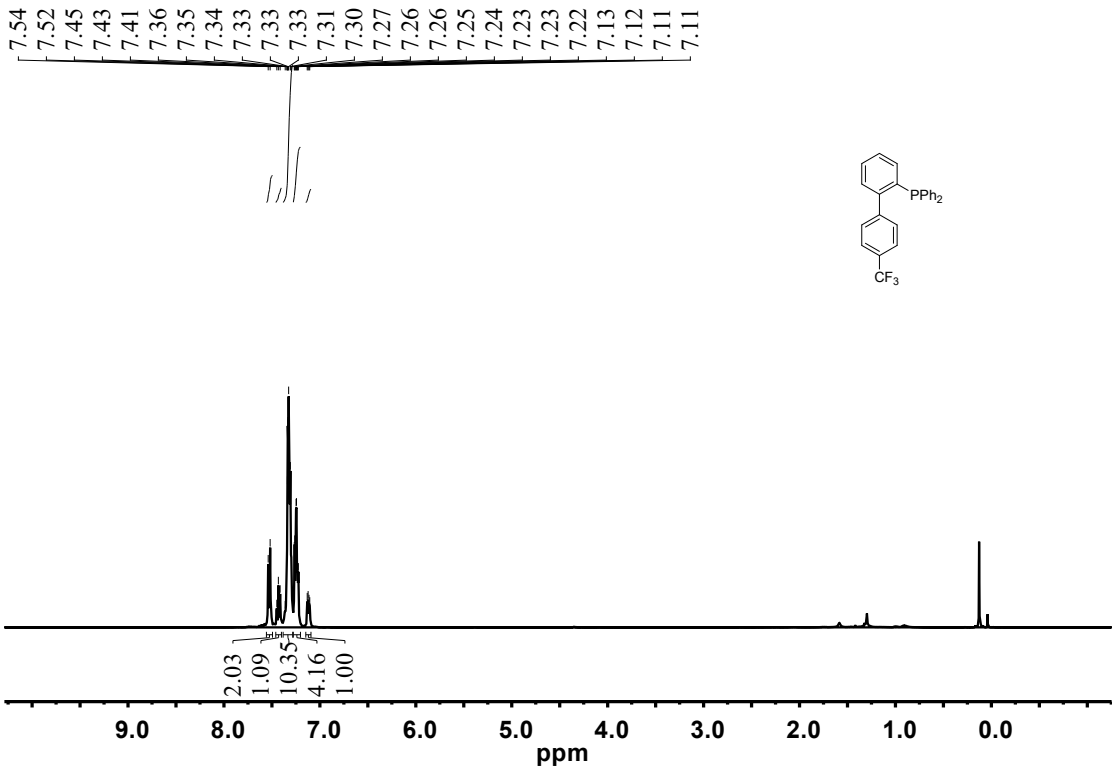
^{13}C NMR spectrum of 1h



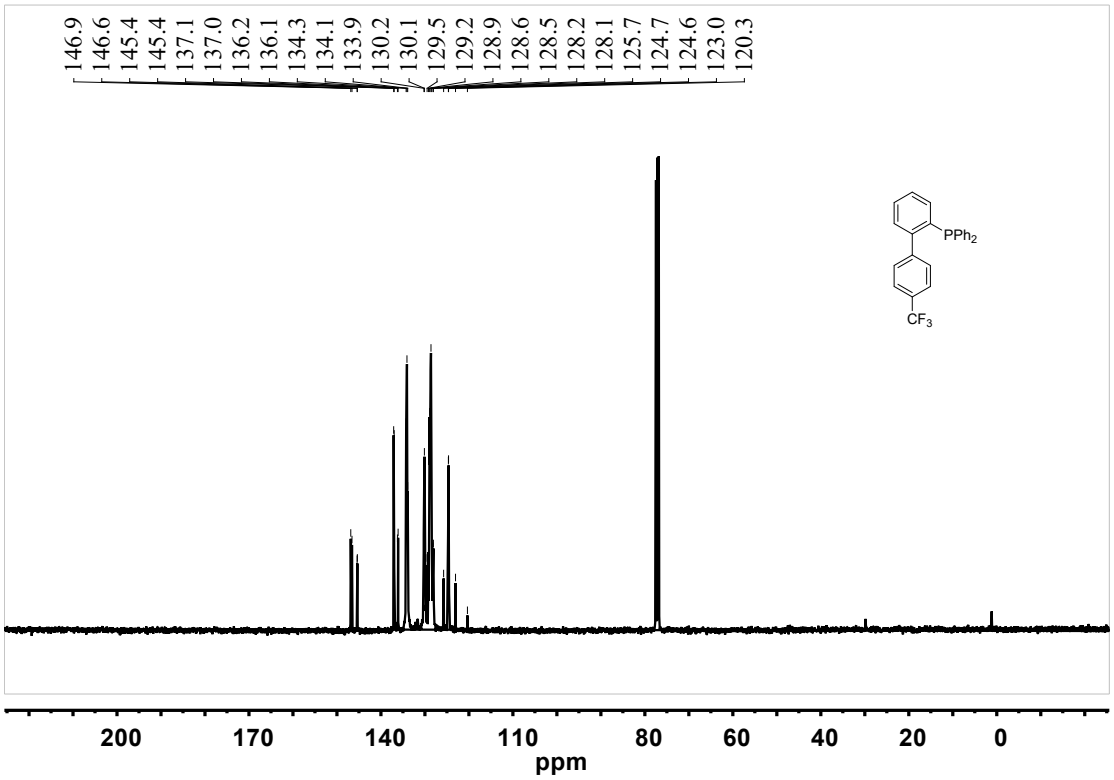
^{31}P NMR spectrum of 1h



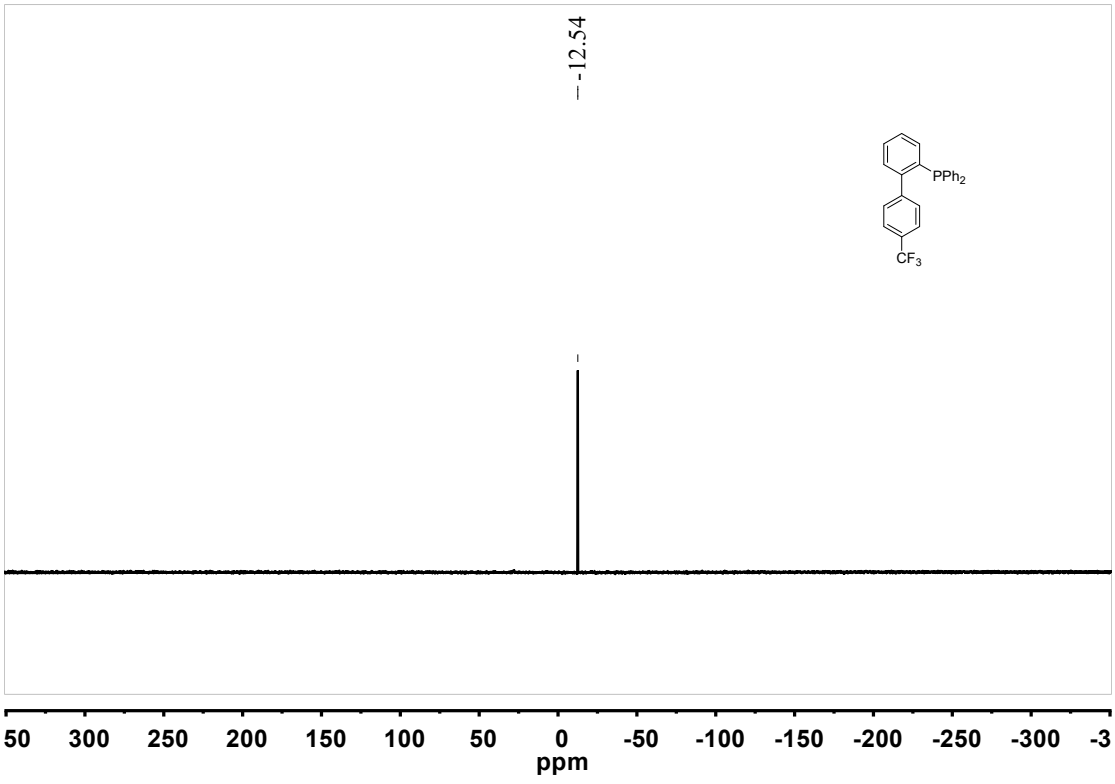
^{1}H NMR spectrum of 1i



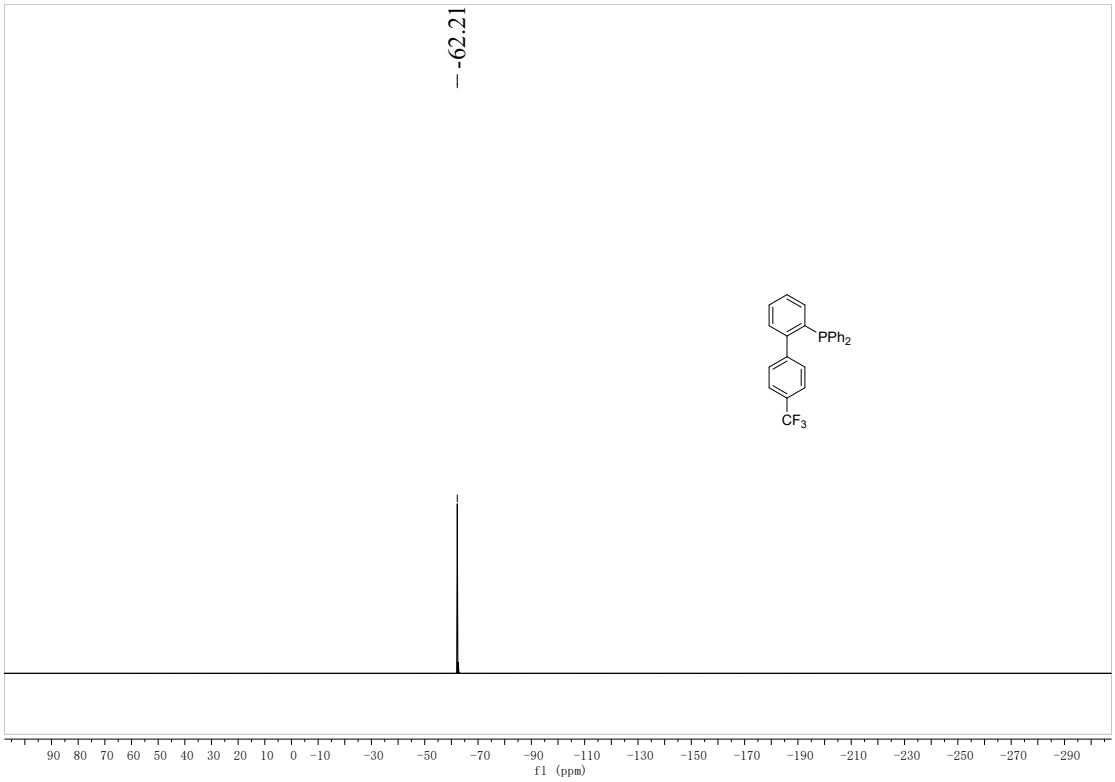
^{13}C NMR spectrum of 1i



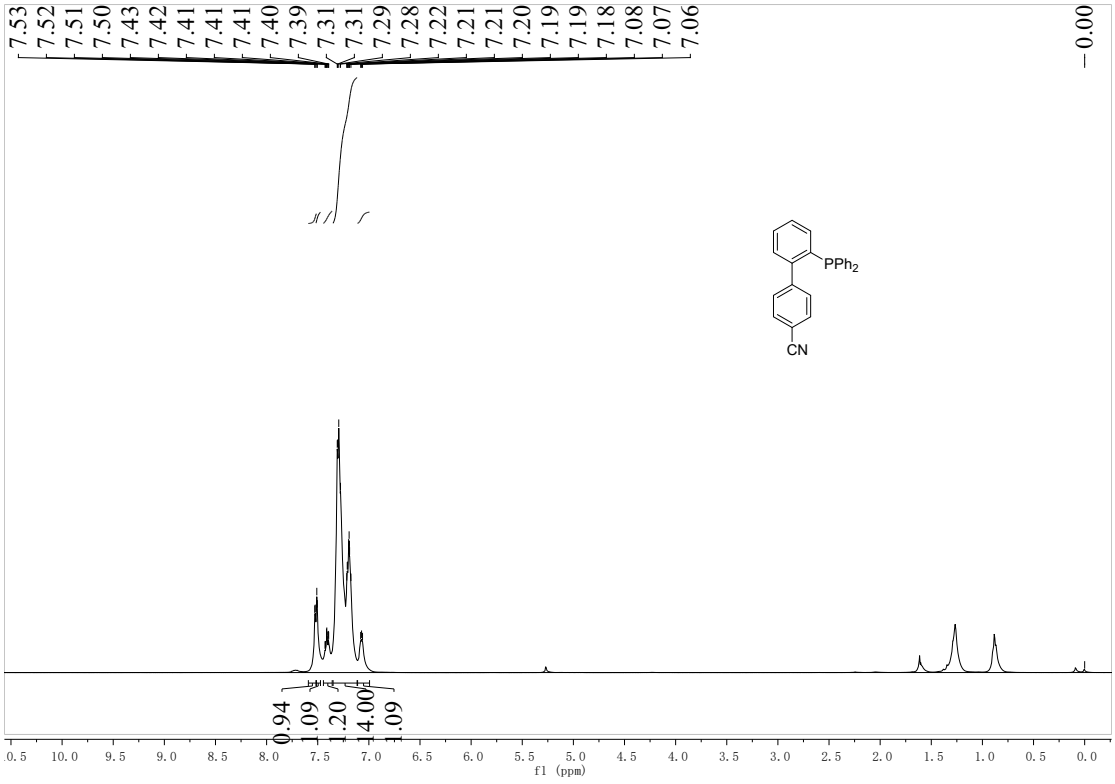
^{31}P NMR spectrum of 1i



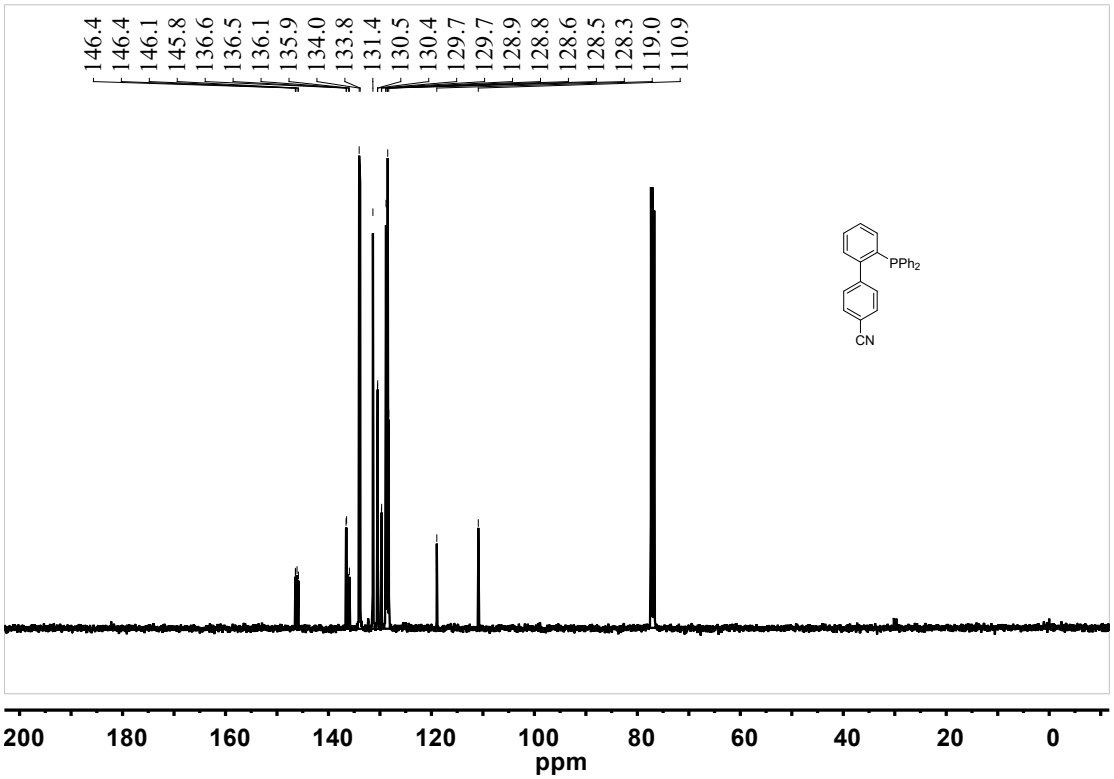
^{19}F NMR spectrum of 1i



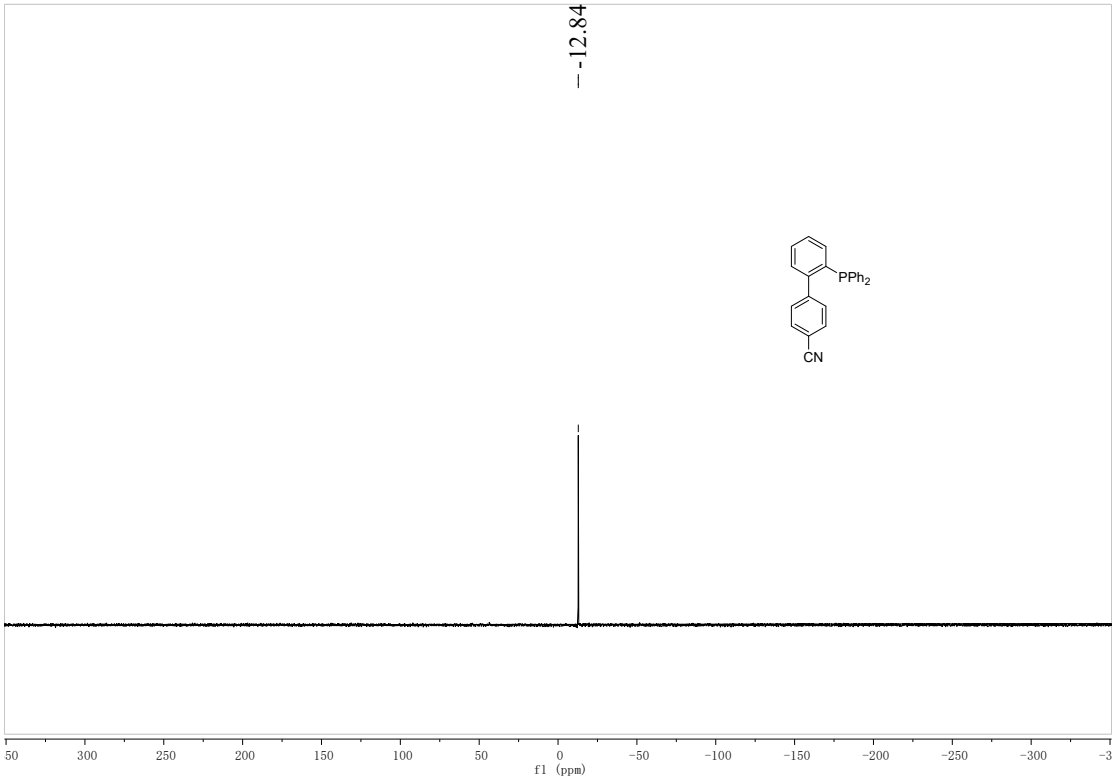
^{1}H NMR spectrum of 1j



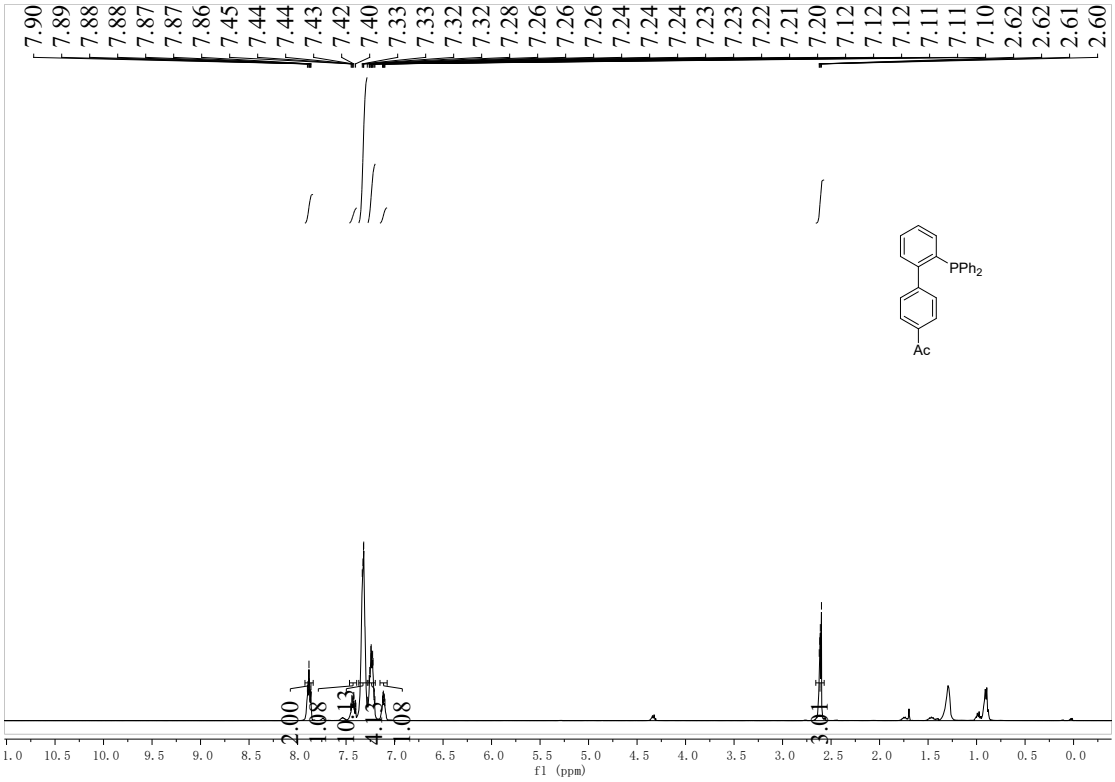
^{13}C NMR spectrum of 1j



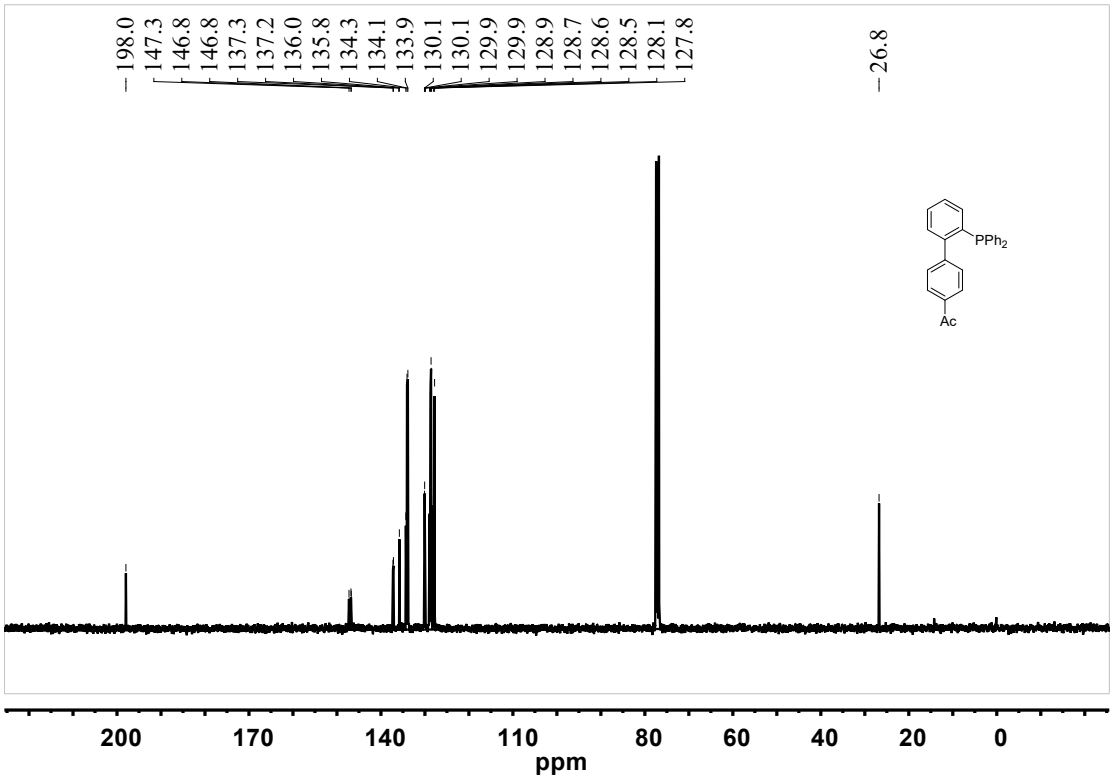
^{31}P NMR spectrum of 1j



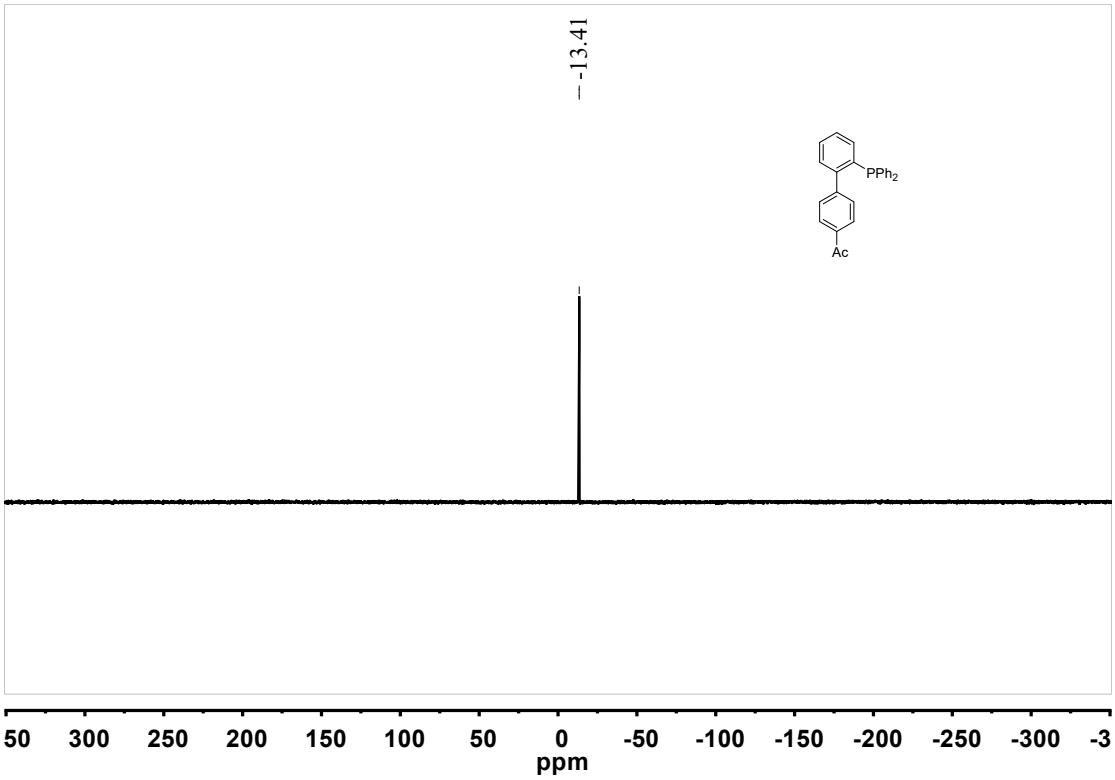
^{1}H NMR spectrum of 1k



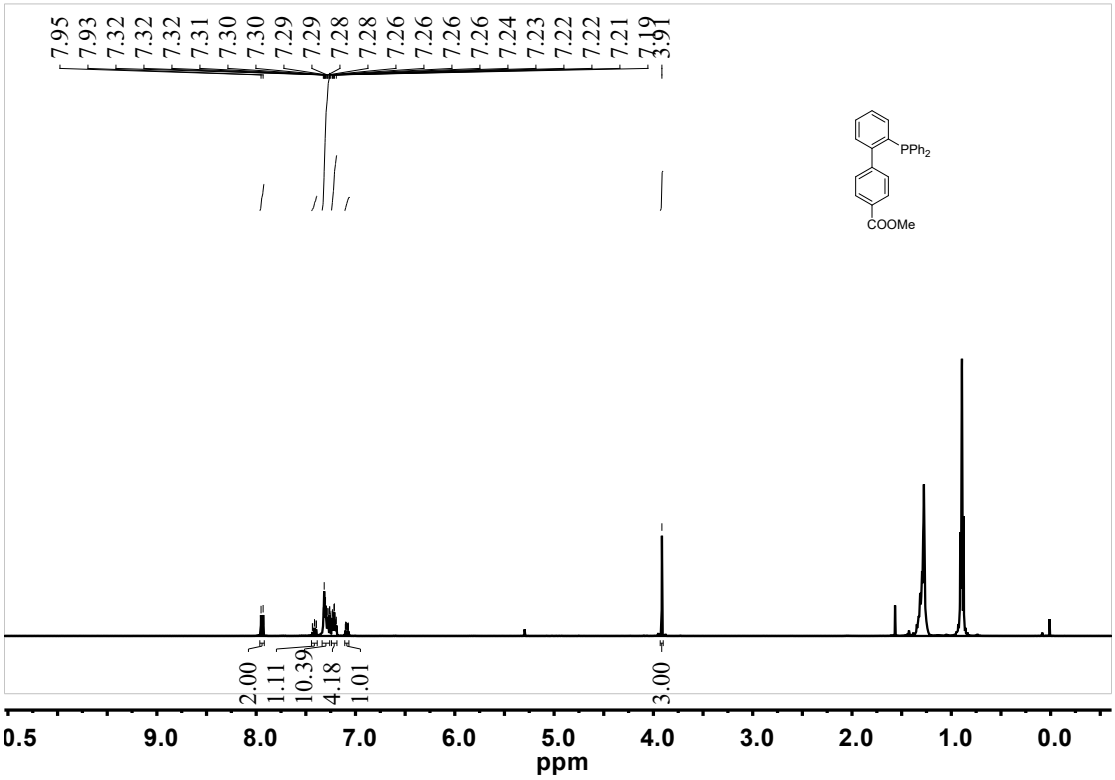
^{13}C NMR spectrum of 1k



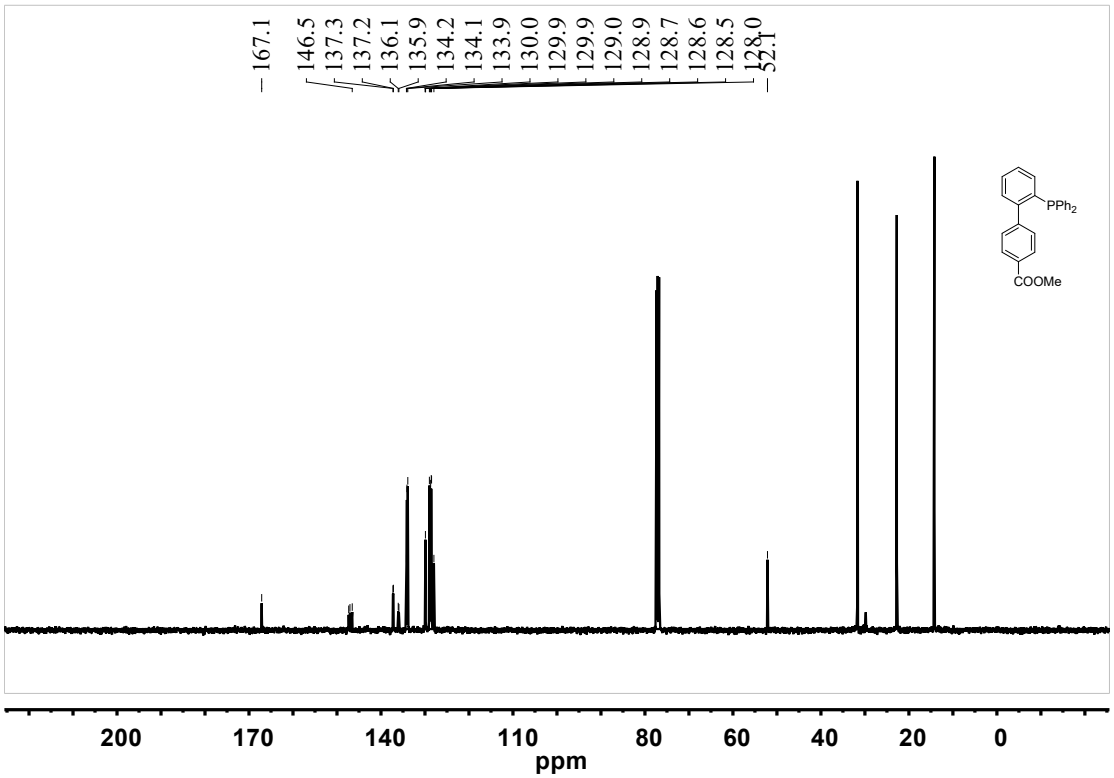
^{31}P NMR spectrum of 1k



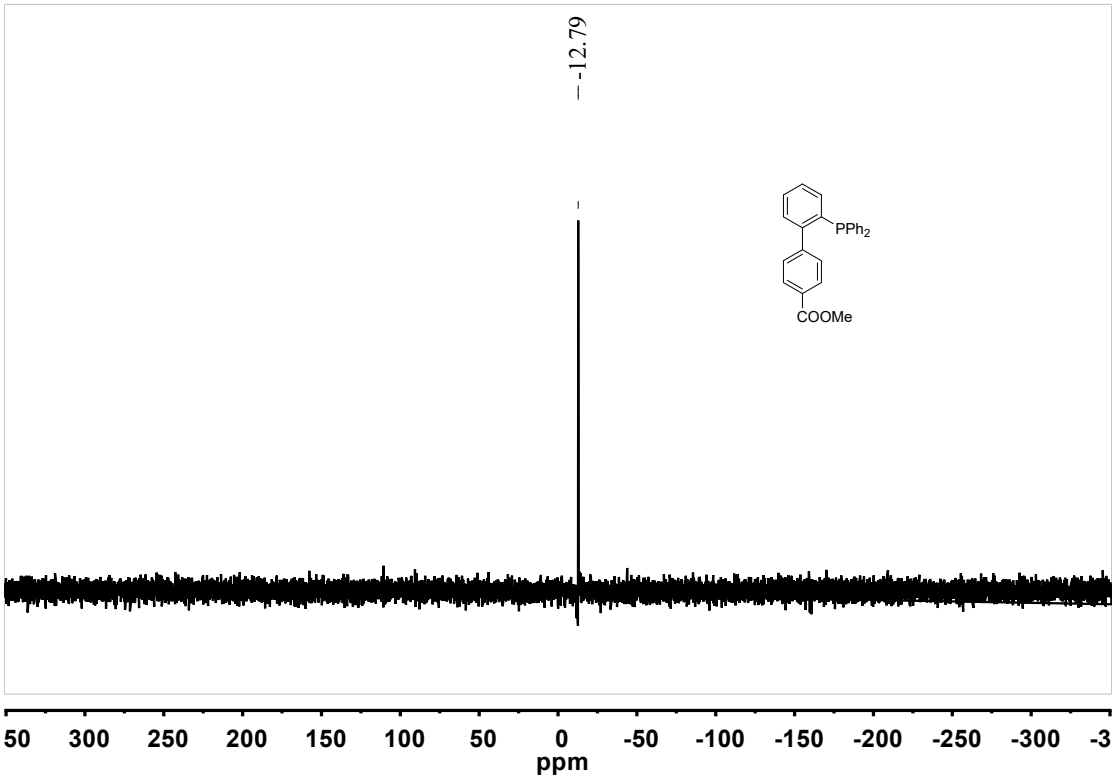
^{1}H NMR spectrum of 1l



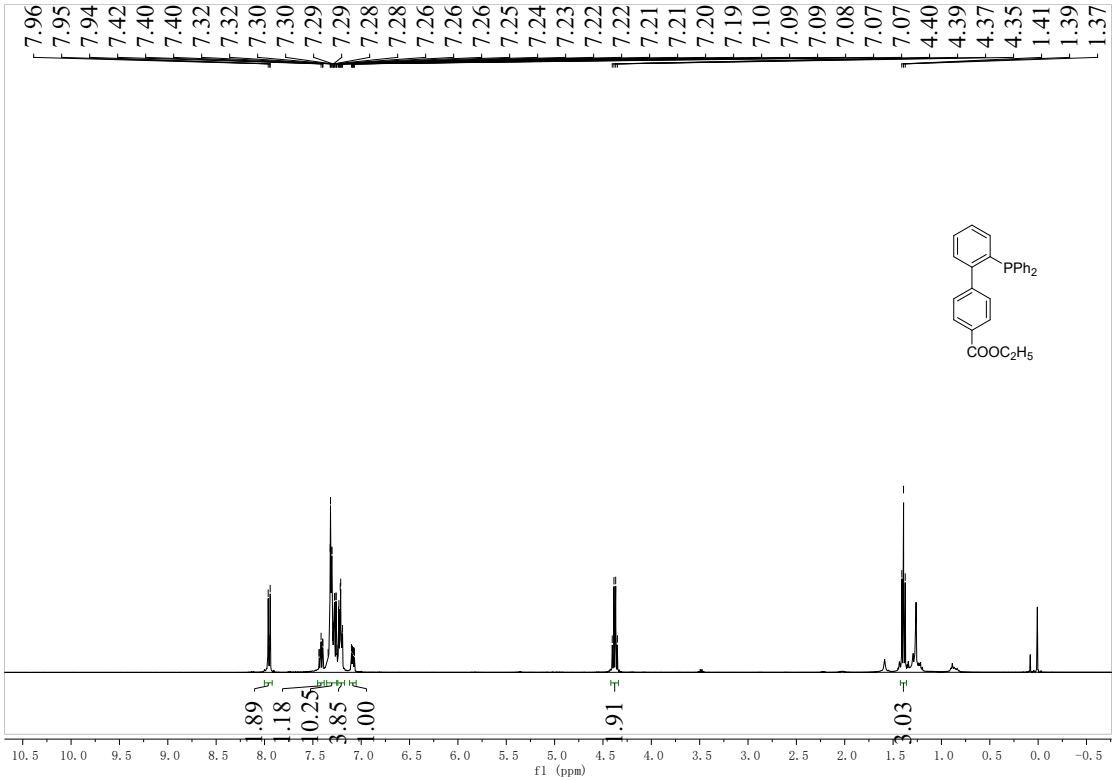
^{13}C NMR spectrum of 1l



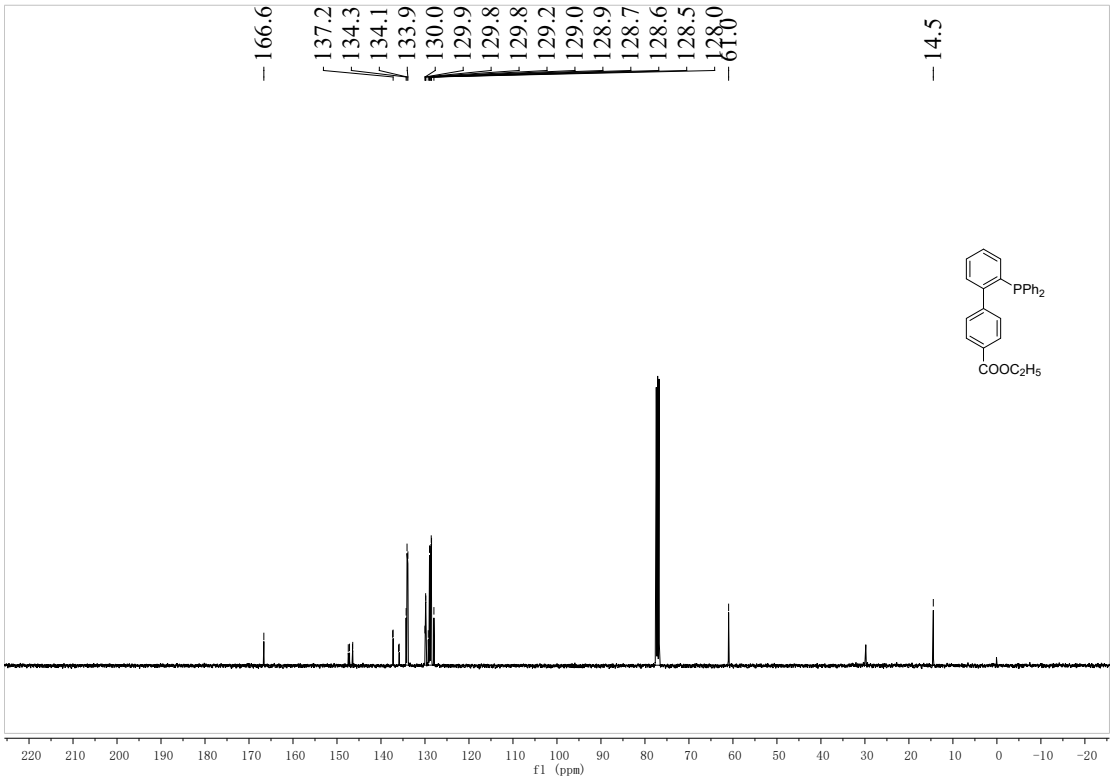
^{31}P NMR spectrum of 1l



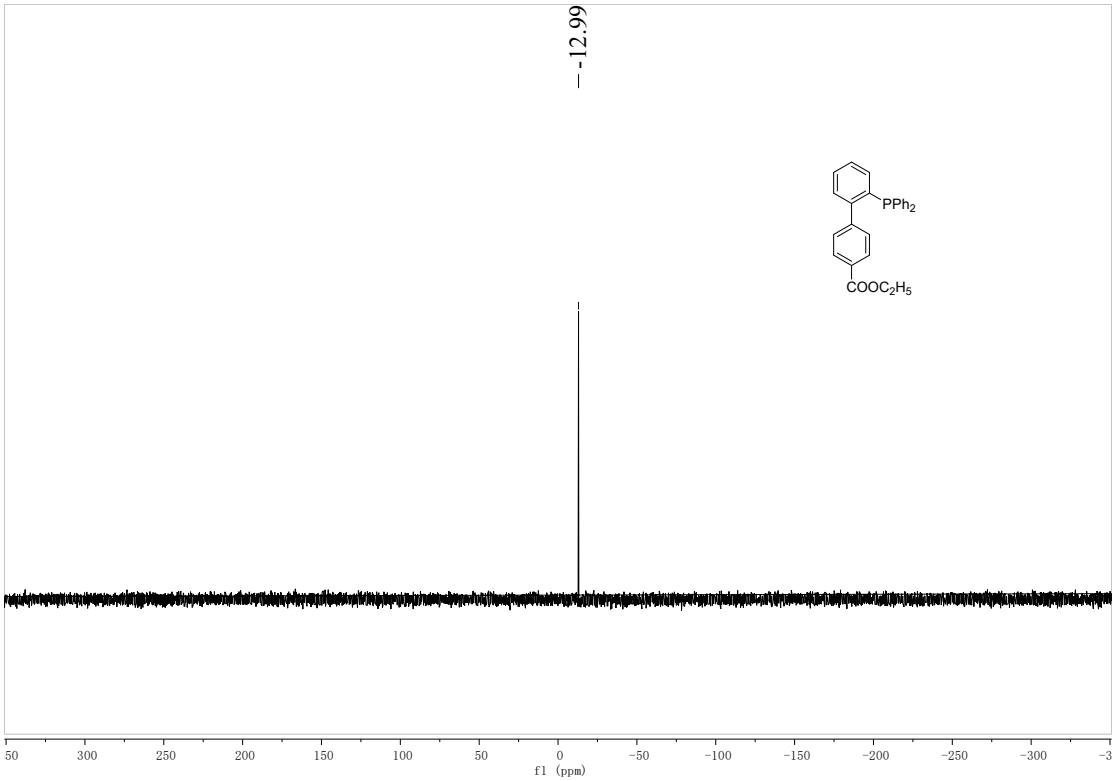
^{1}H NMR spectrum of 1m



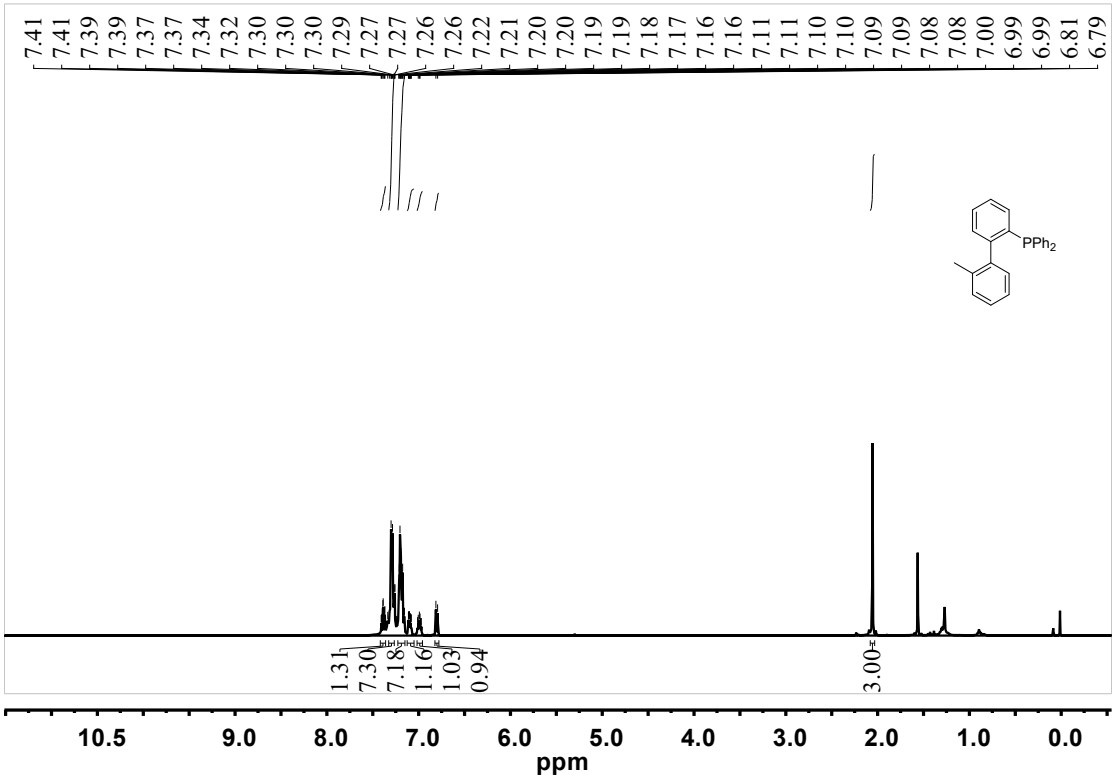
^{13}C NMR spectrum of 1m



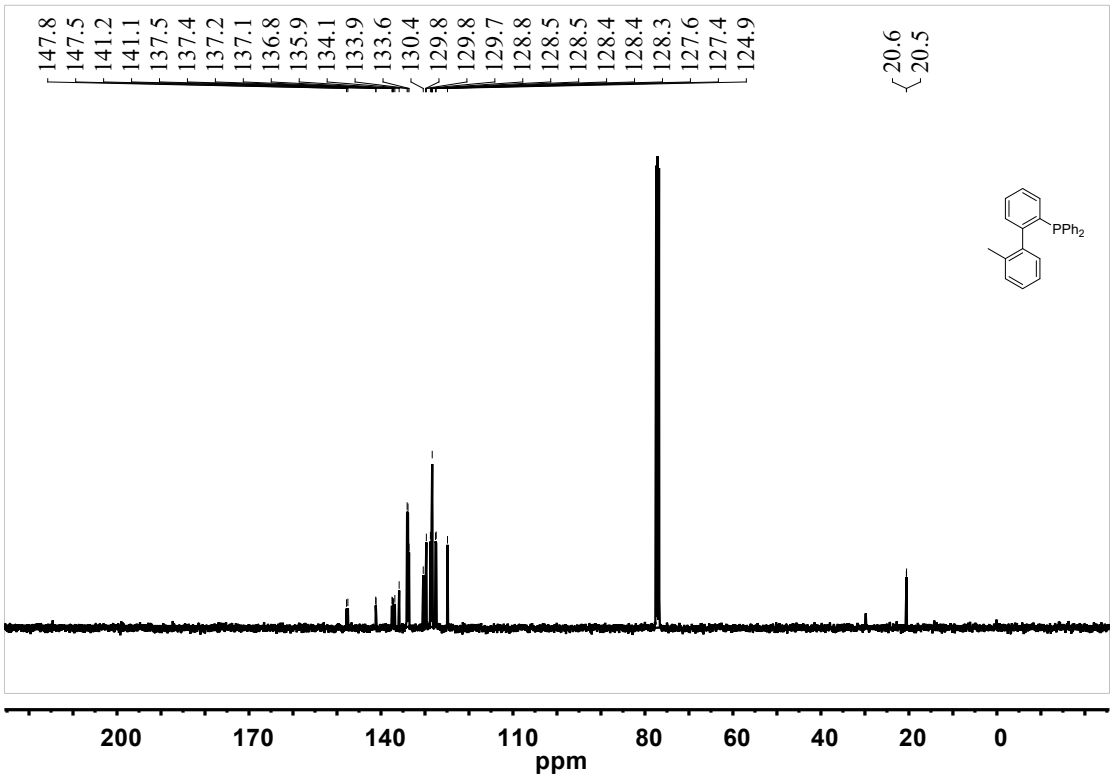
^{31}P NMR spectrum of 1m



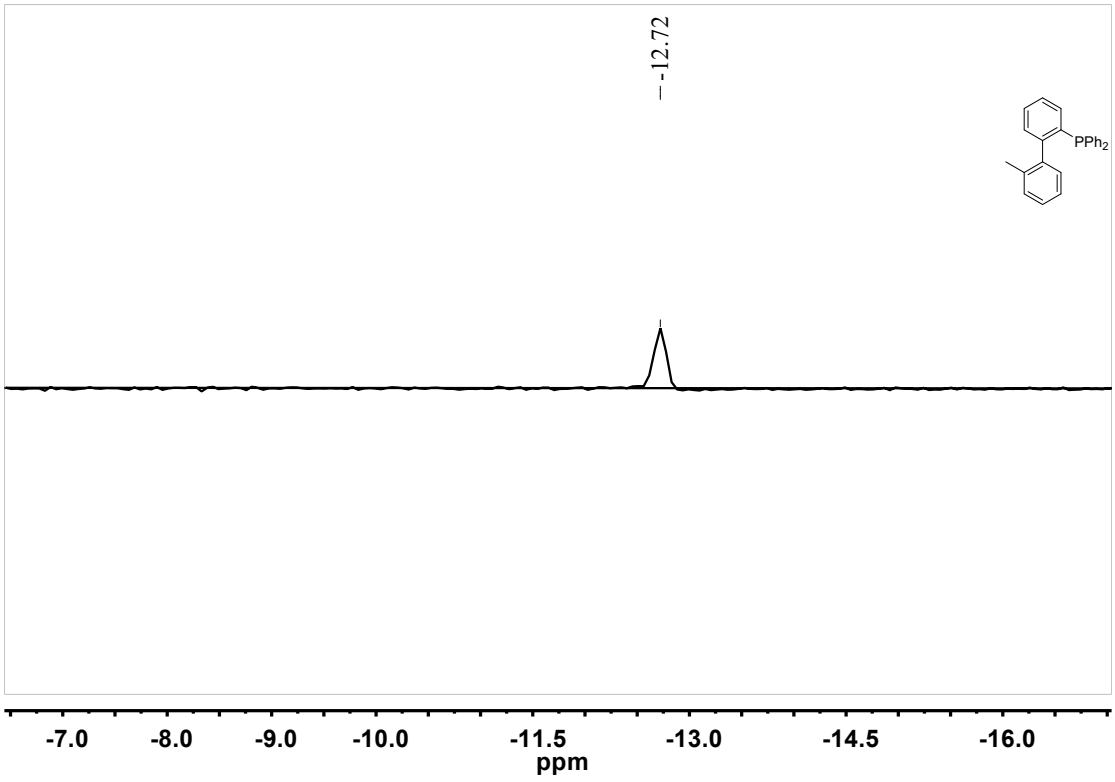
^{1}H NMR spectrum of 1n



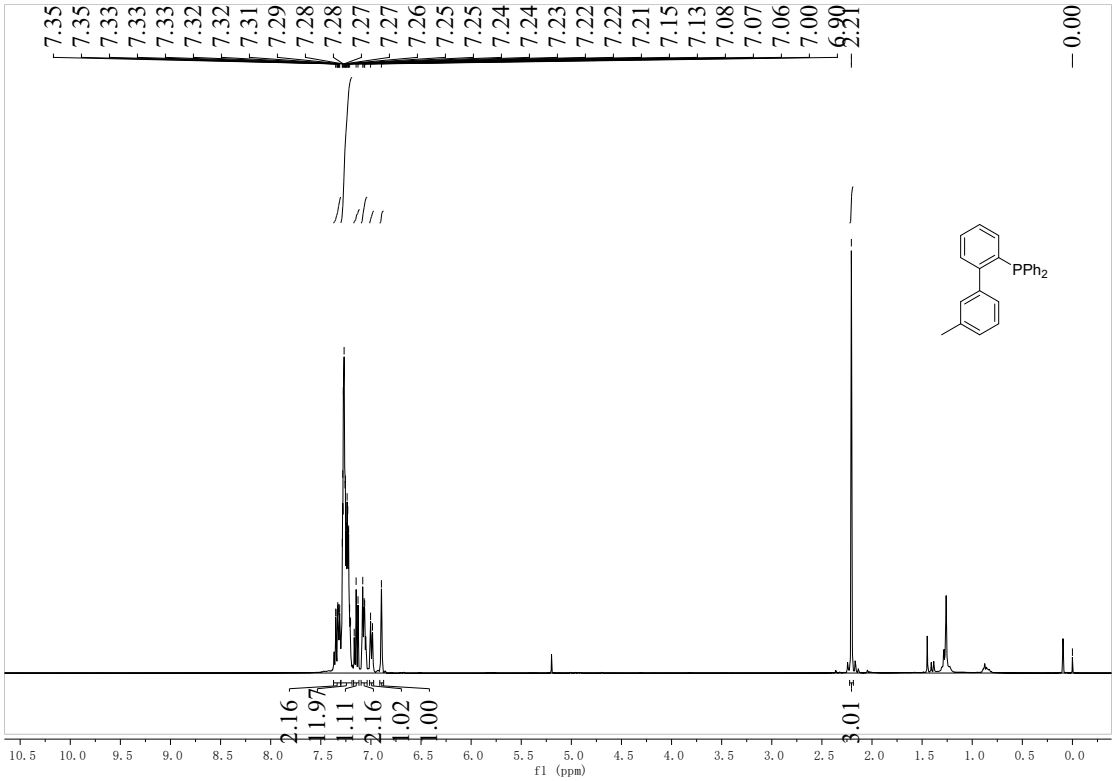
^{13}C NMR spectrum of 1n



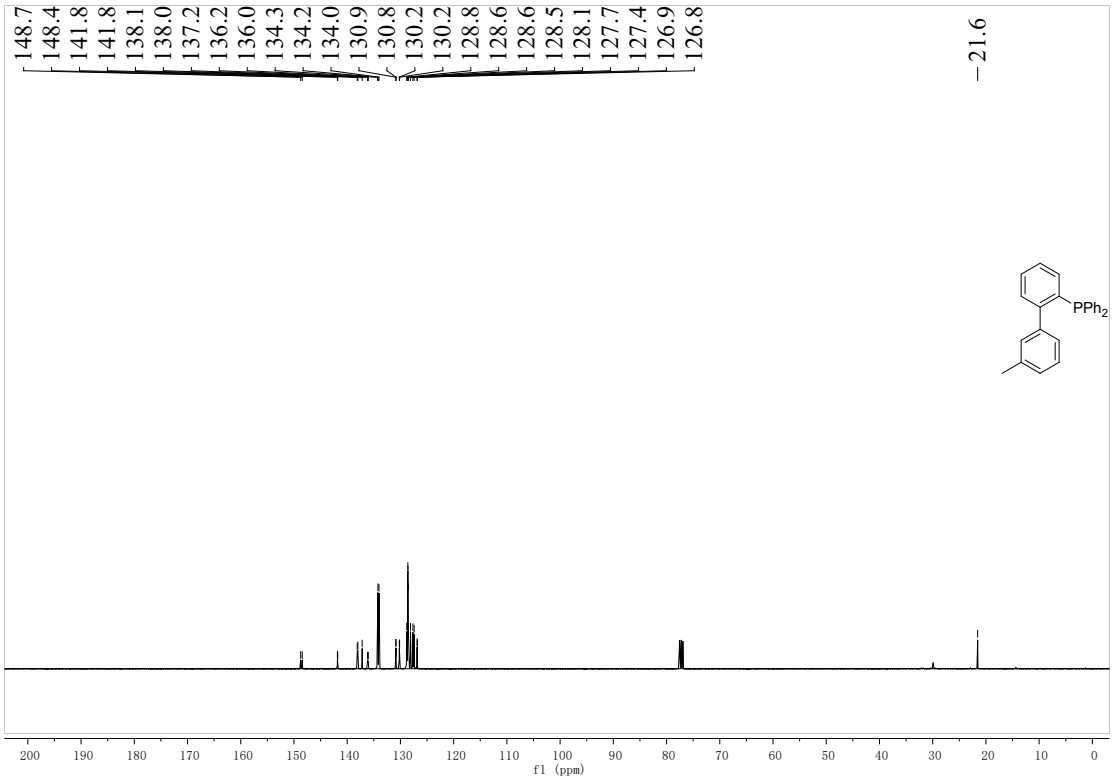
^{31}P NMR spectrum of 1n



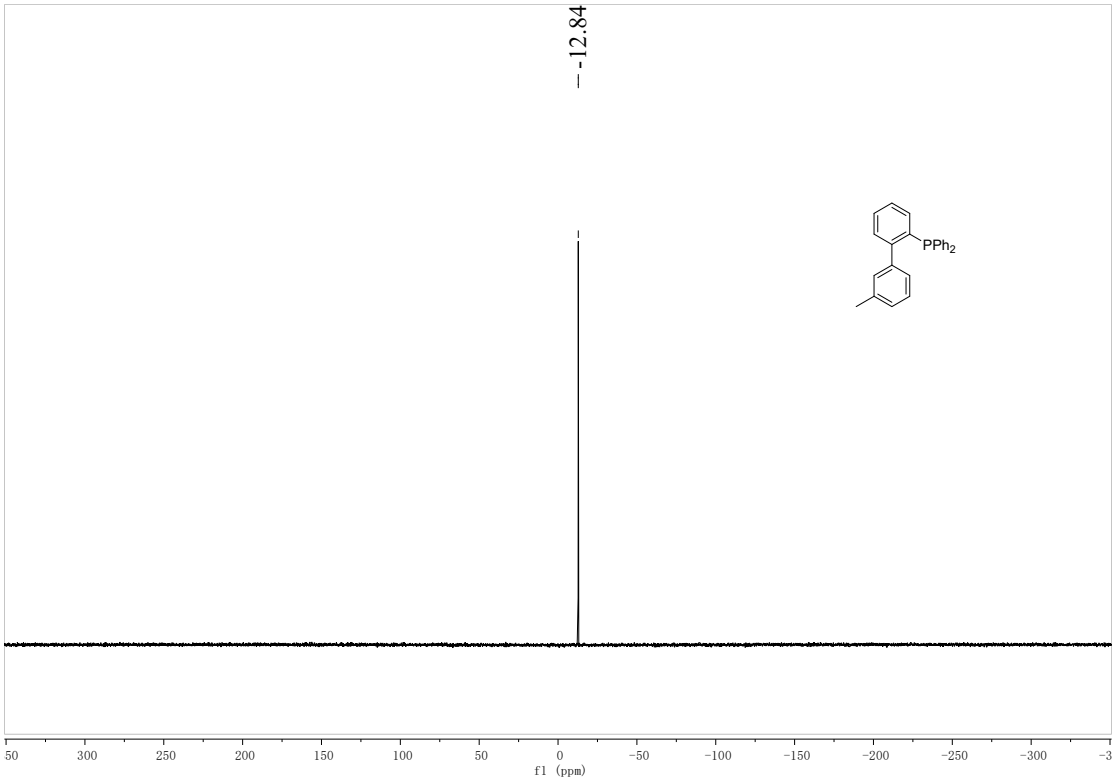
^{1}H NMR spectrum of 1o



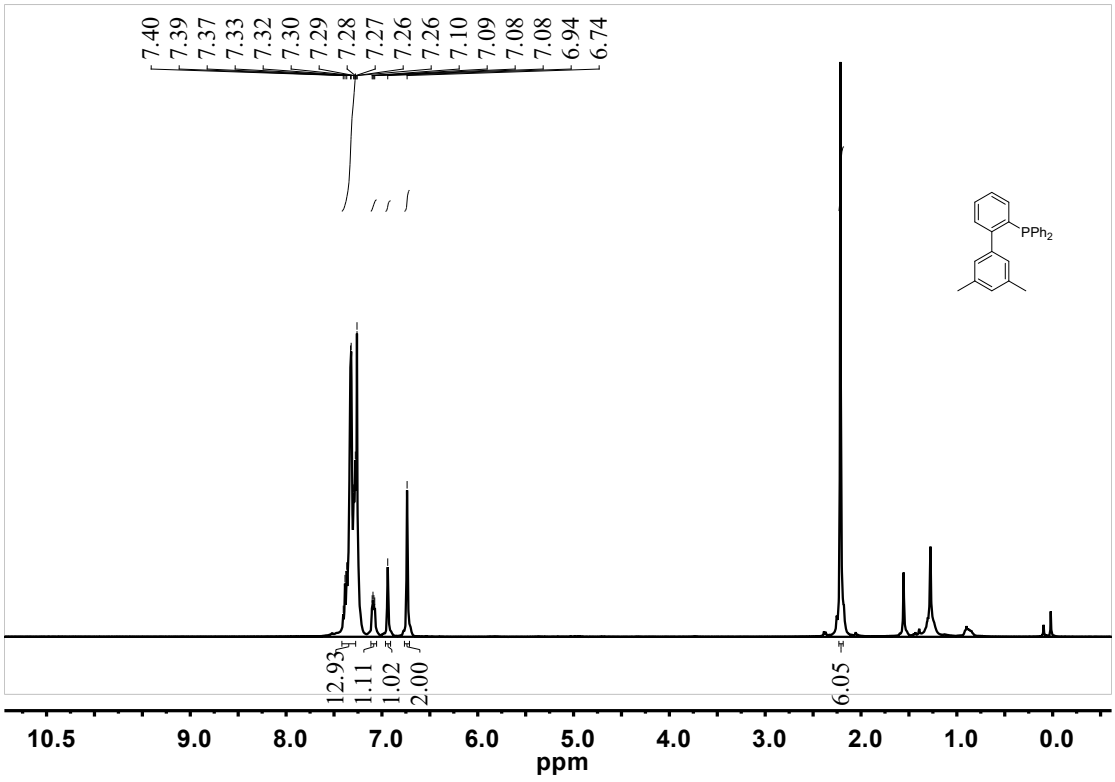
^{13}C NMR spectrum of 1o



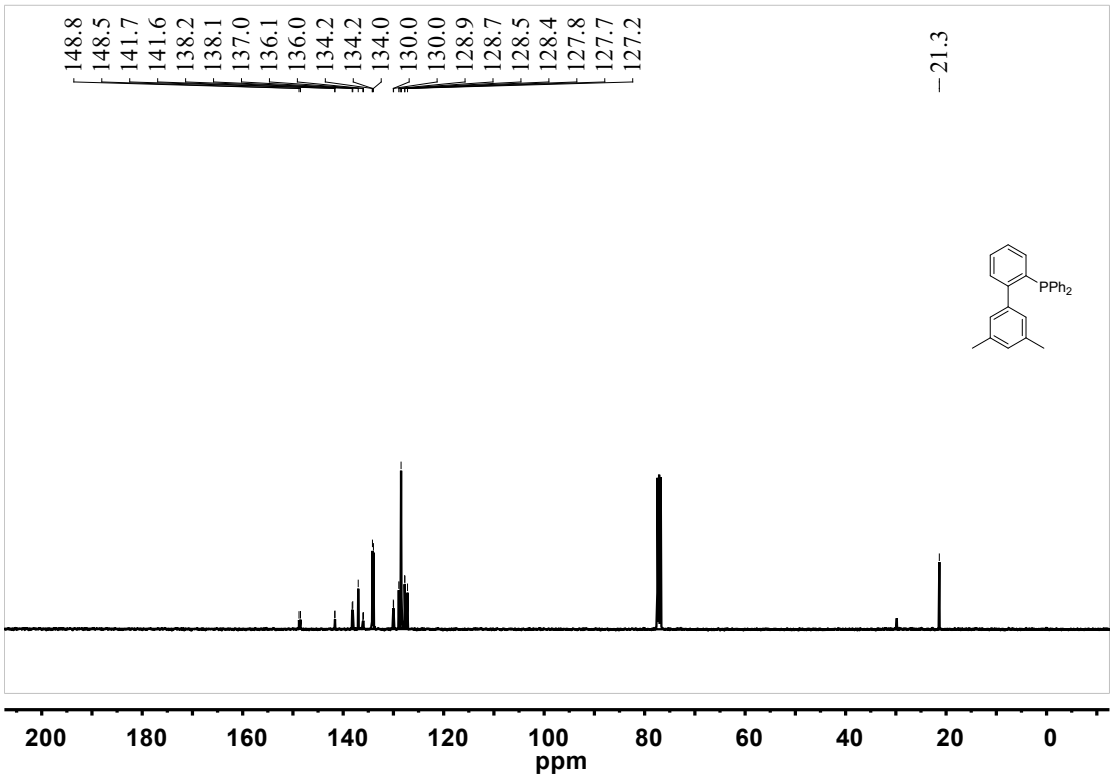
^{31}P NMR spectrum of 1o



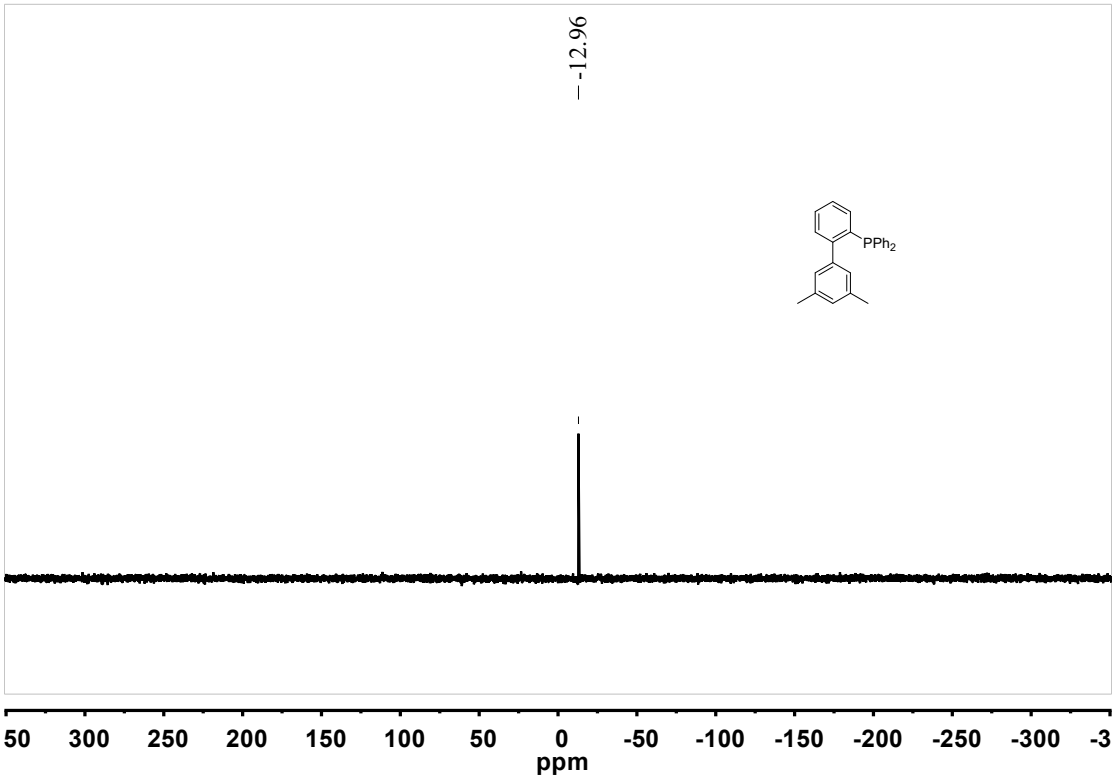
^{1}H NMR spectrum of 1p



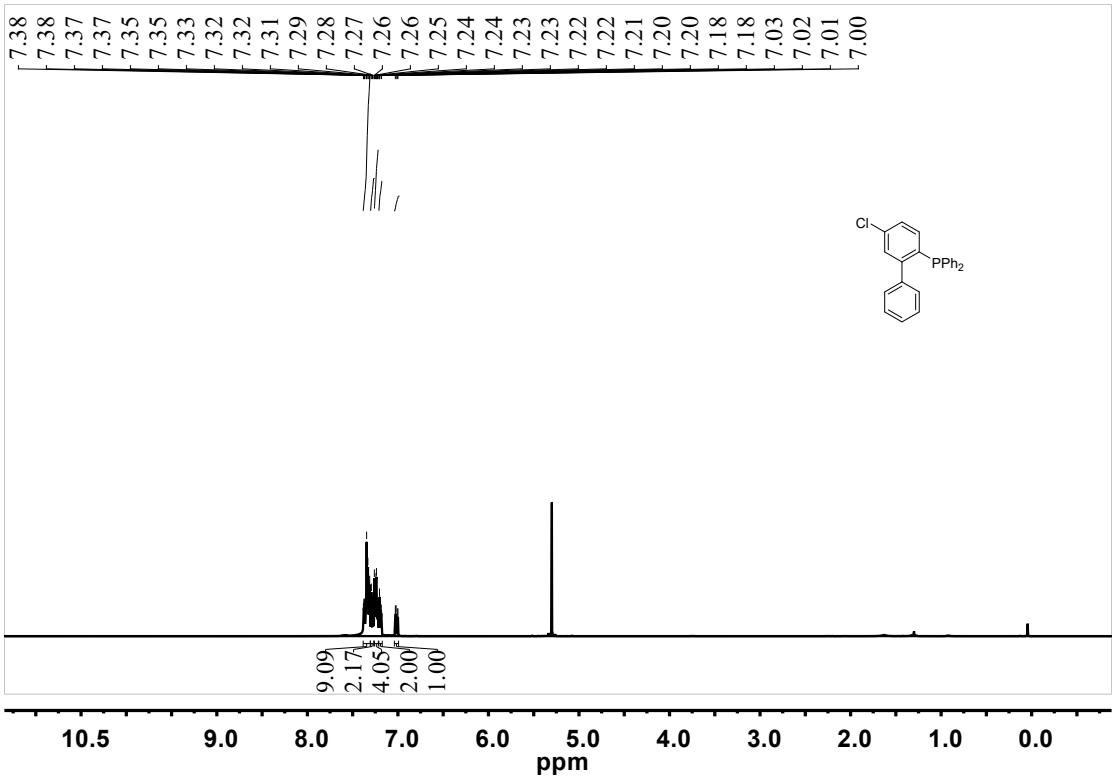
^{13}C NMR spectrum of 1p



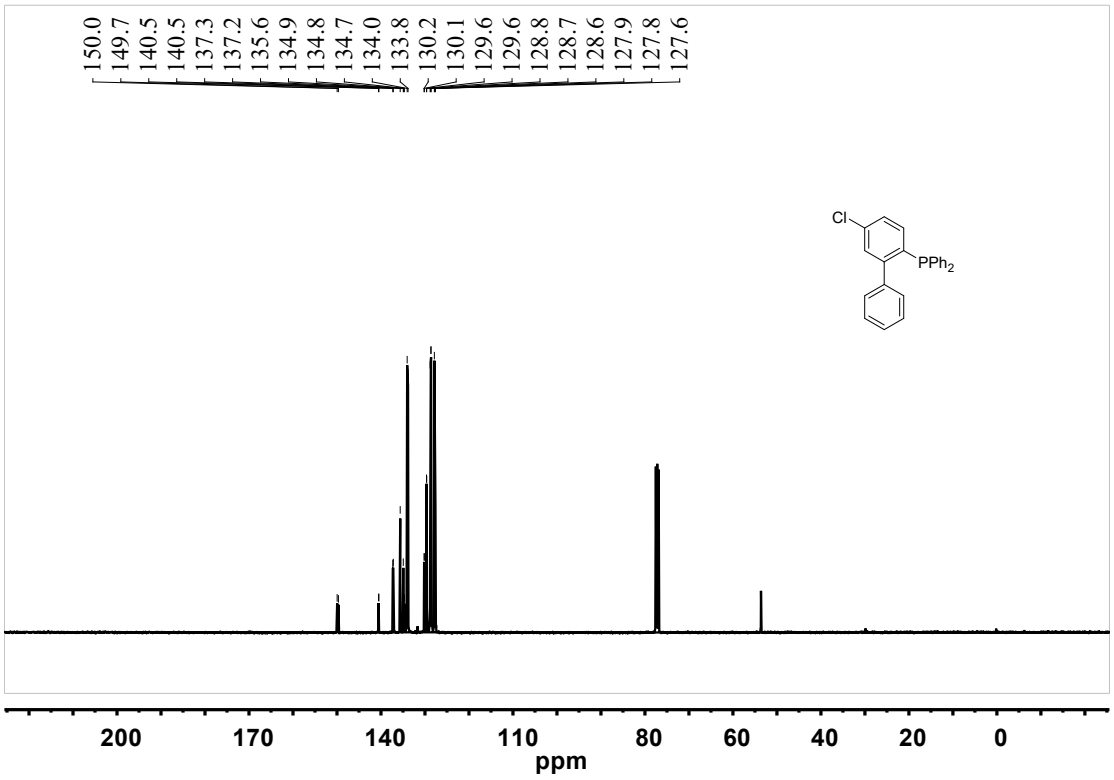
^{31}P NMR spectrum of 1p



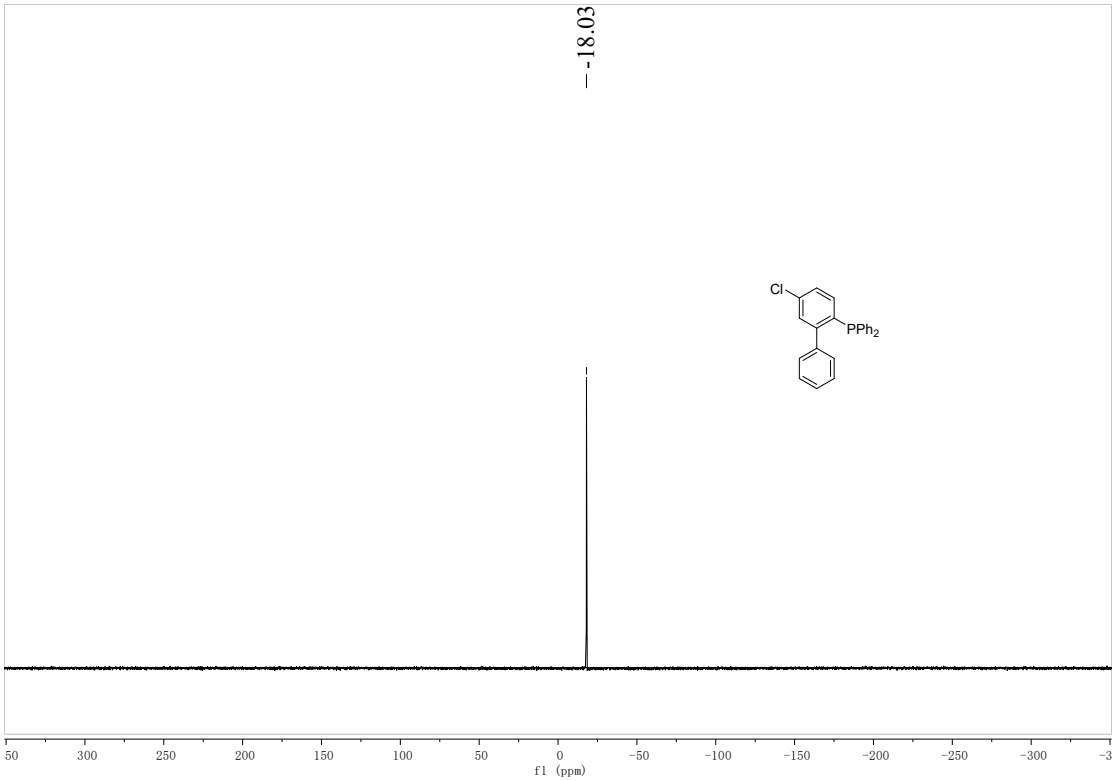
^{1}H NMR spectrum of 1q



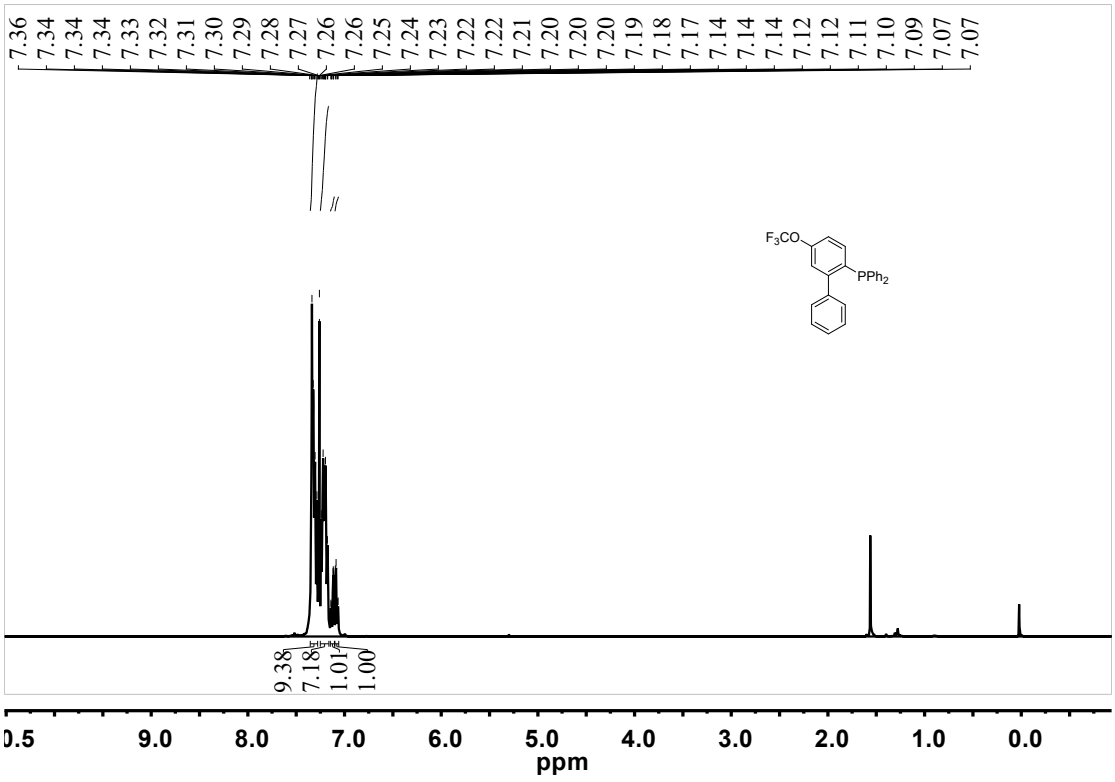
^{13}C NMR spectrum of 1q



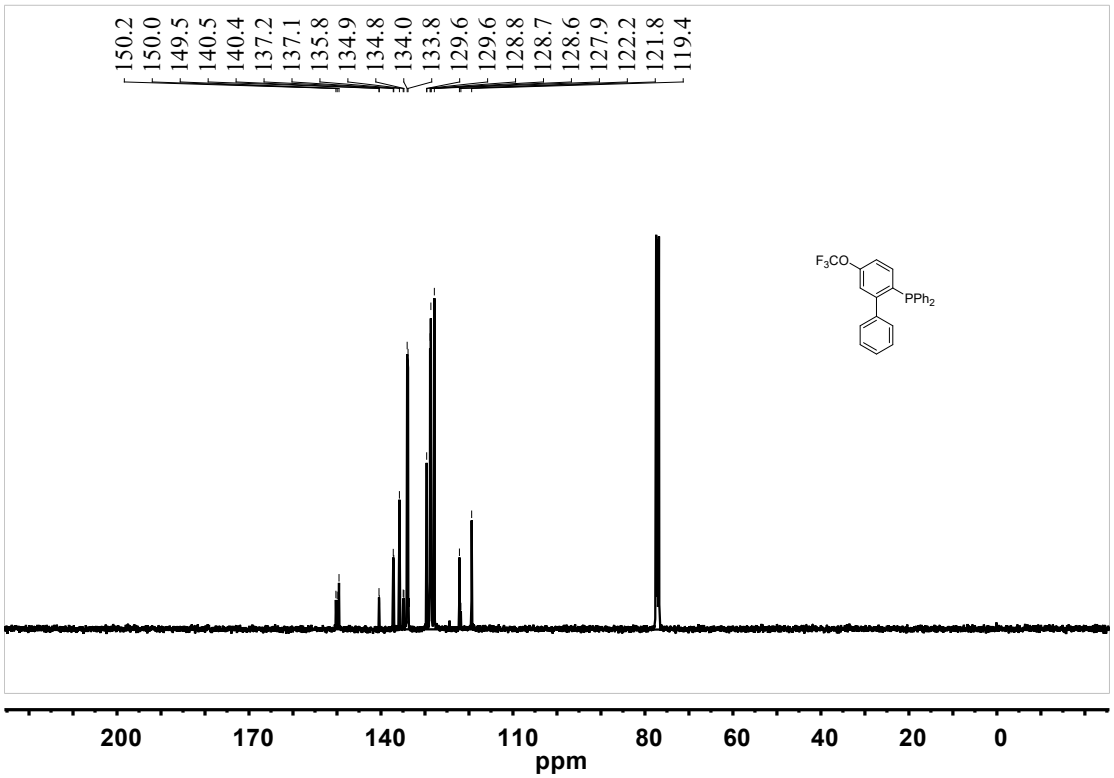
^{31}P NMR spectrum of 1q



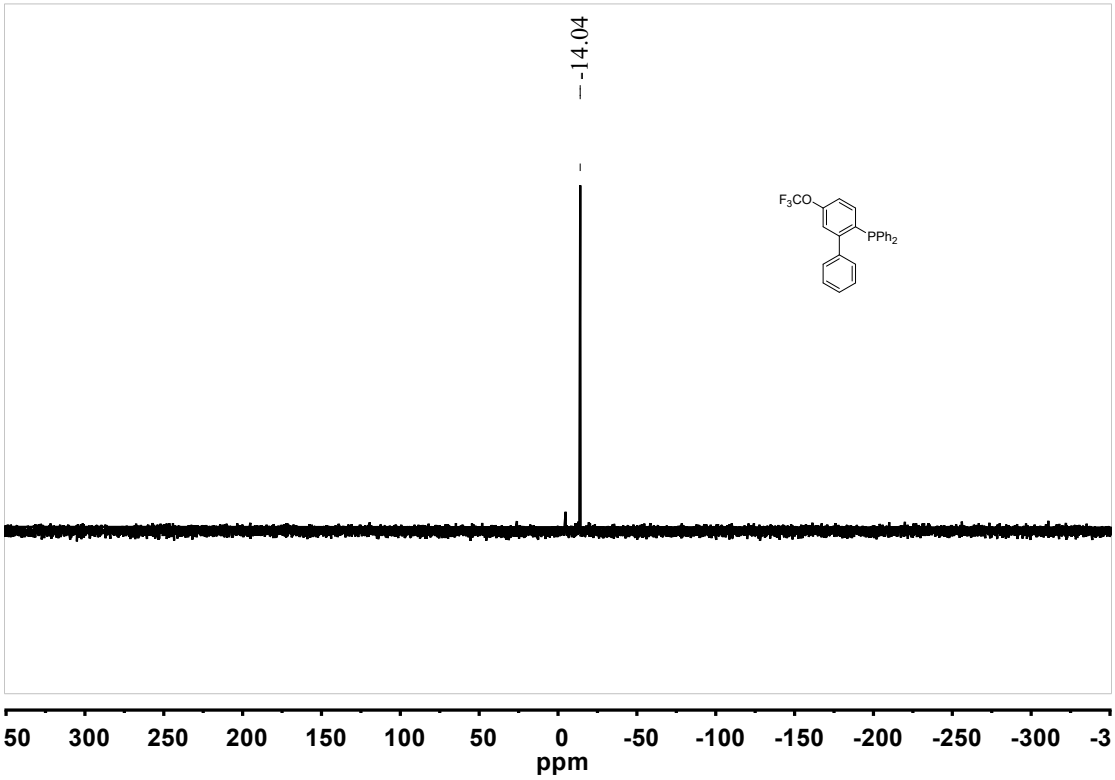
^{1}H NMR spectrum of 1r



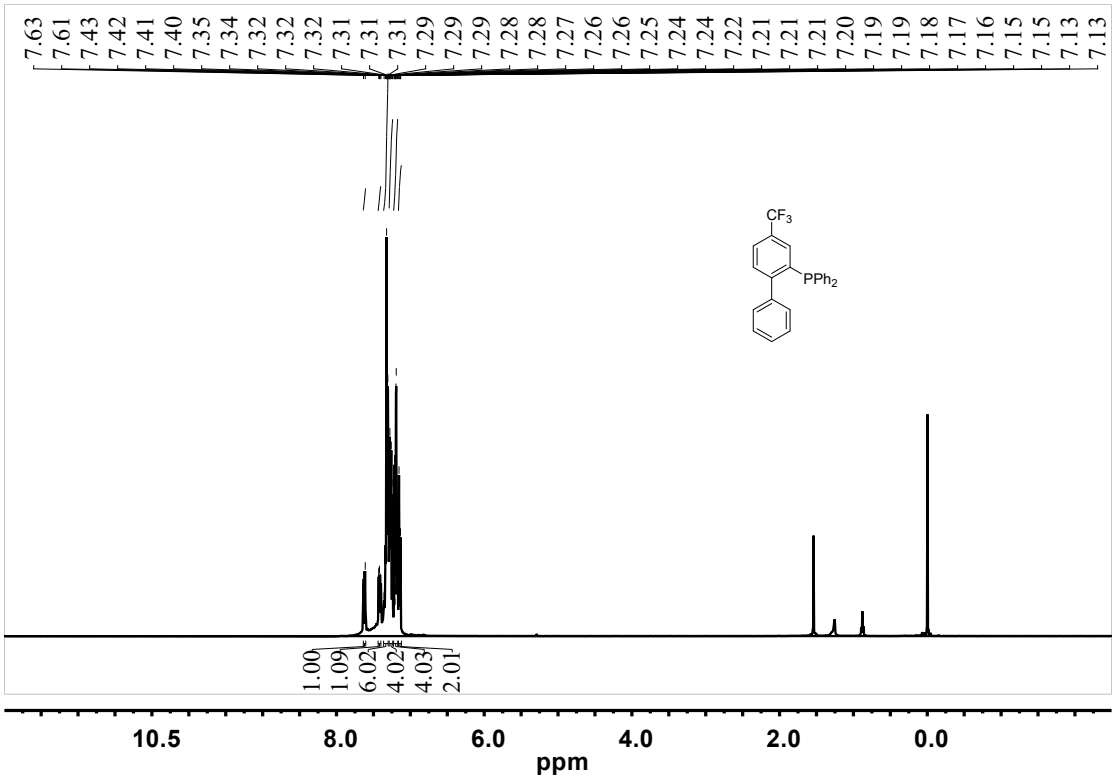
^{13}C NMR spectrum of 1r



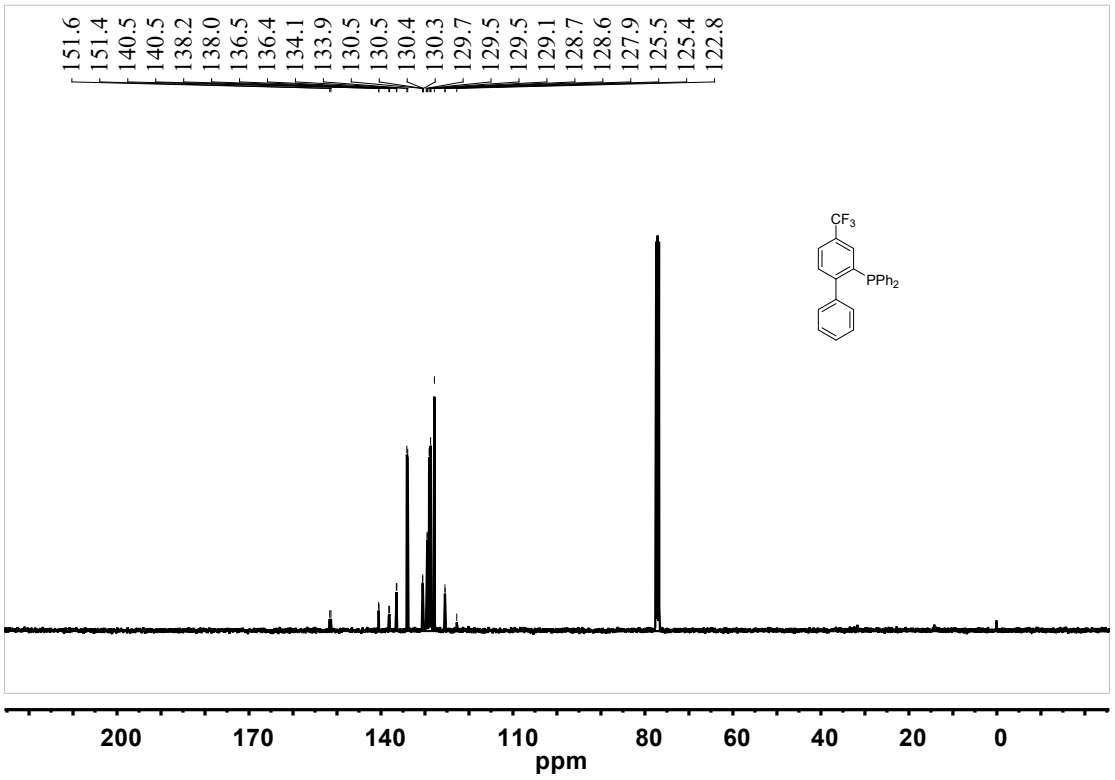
^{31}P NMR spectrum of 1r



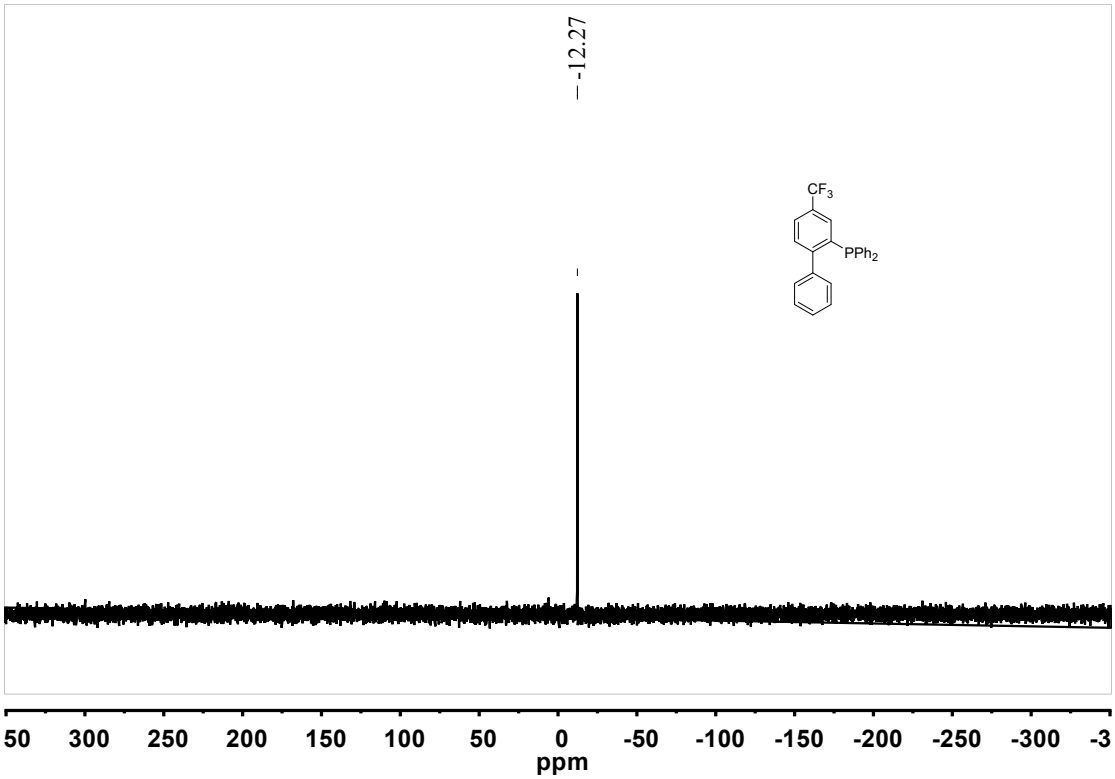
^{1}H NMR spectrum of 1s



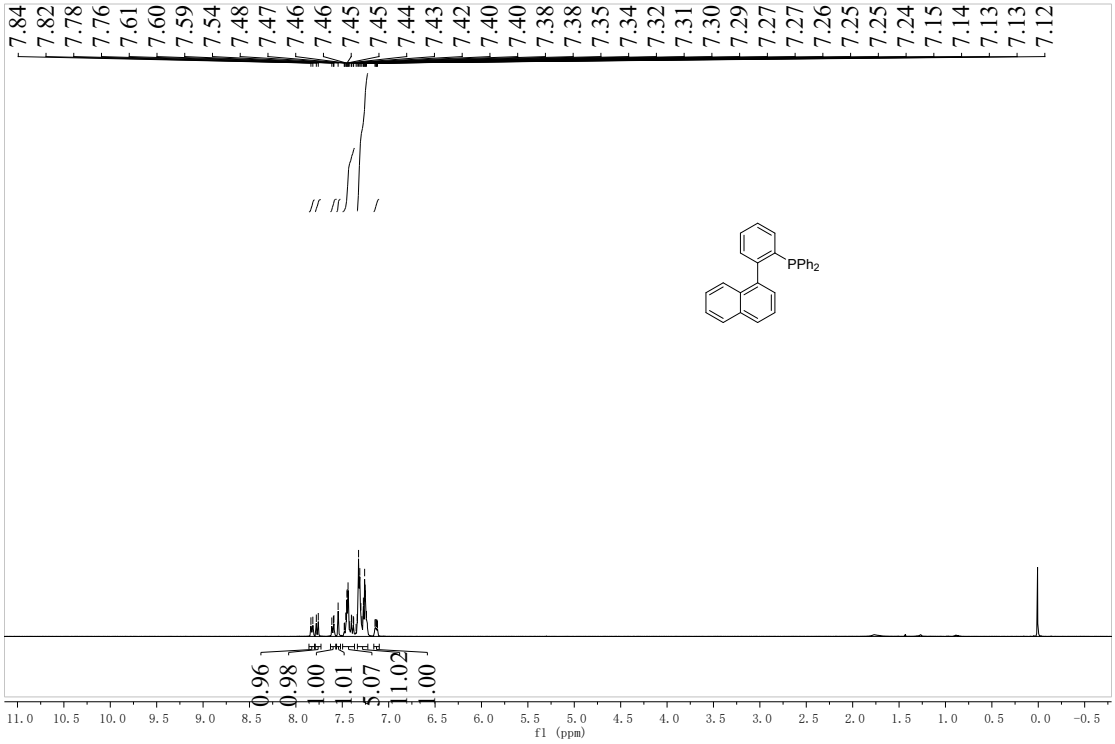
^{13}C NMR spectrum of 1s



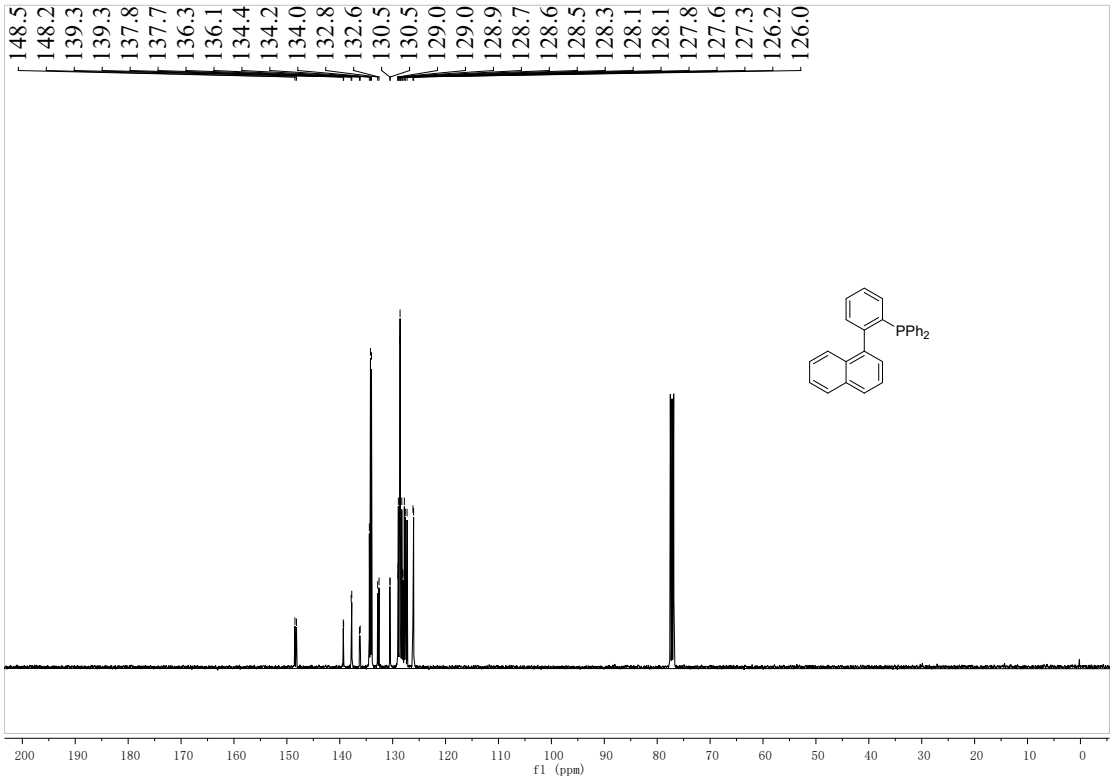
^{31}P NMR spectrum of 1s



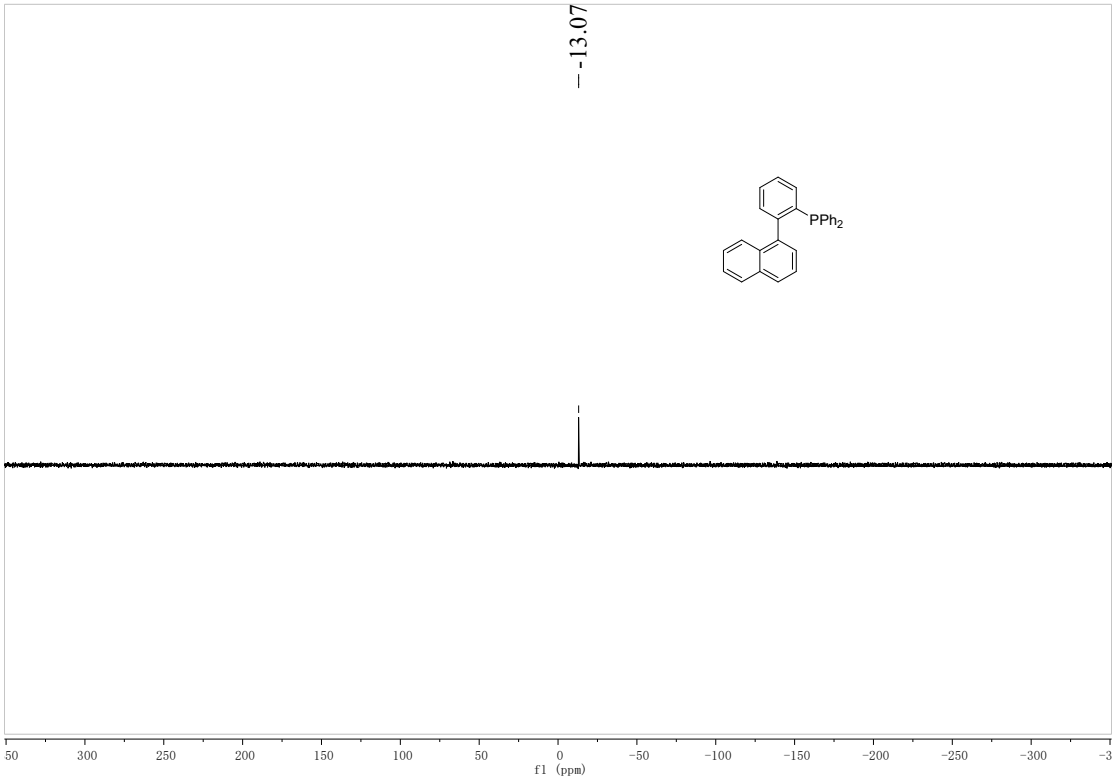
^{1}H NMR spectrum of 1t



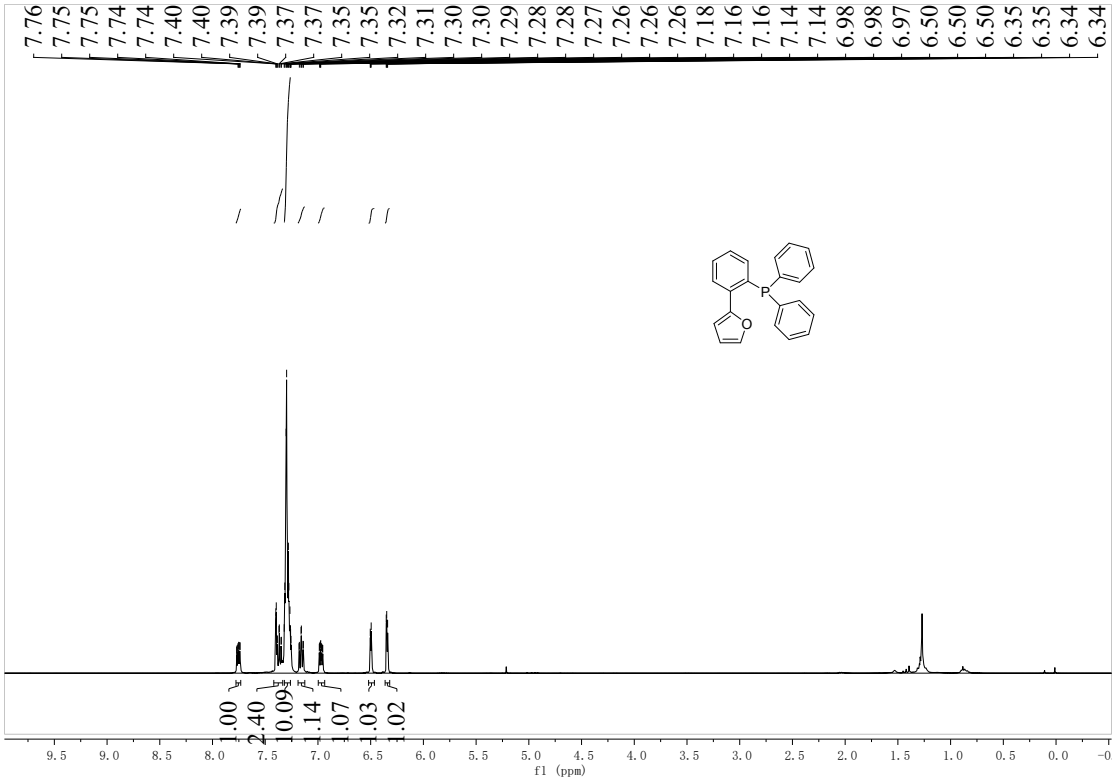
^{13}C NMR spectrum of 1t



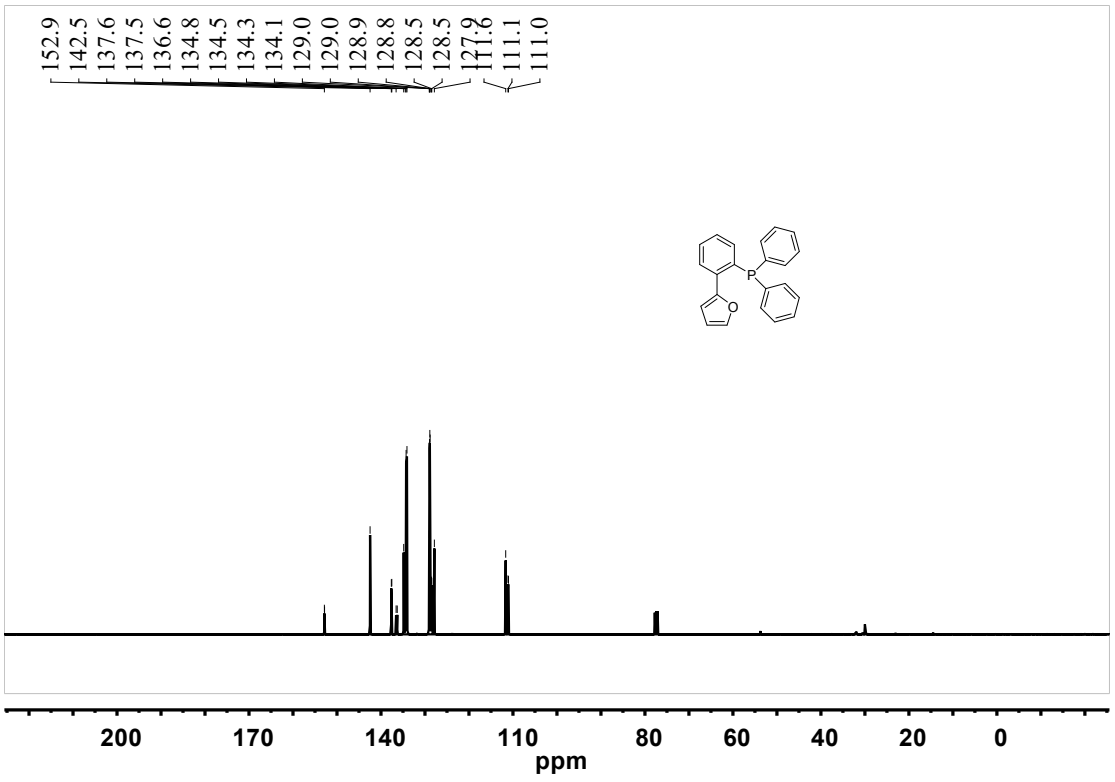
^{31}P NMR spectrum of 1t



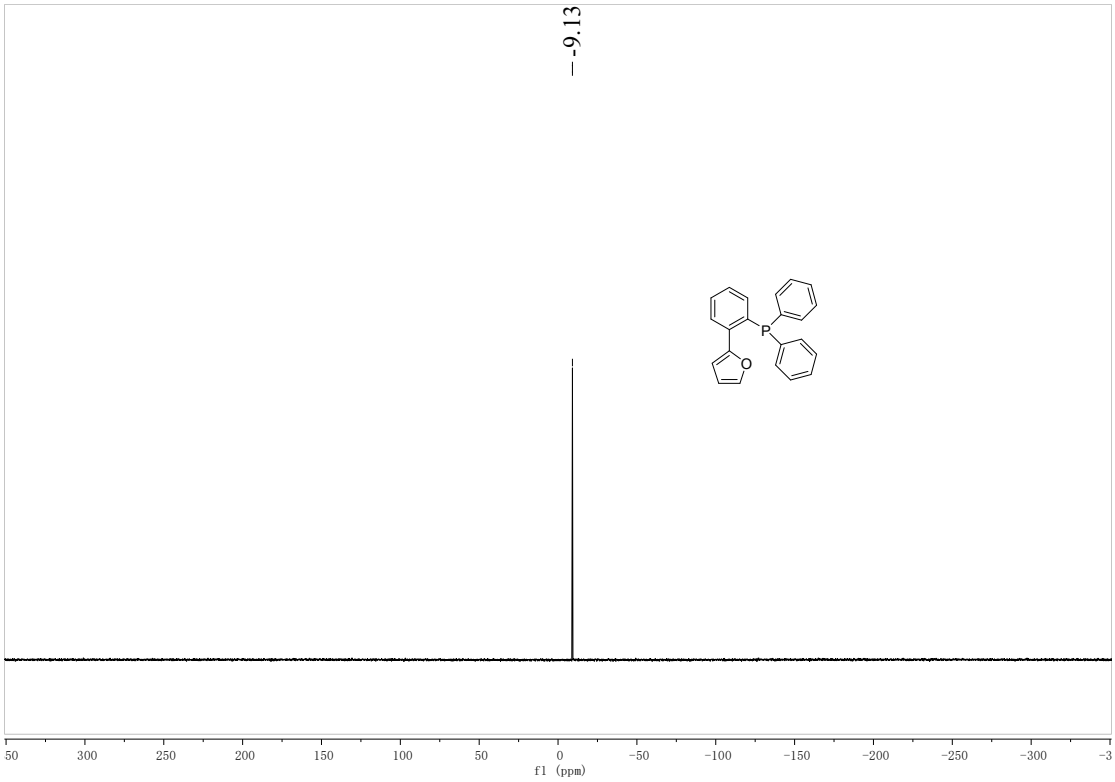
^{1}H NMR spectrum of 1w



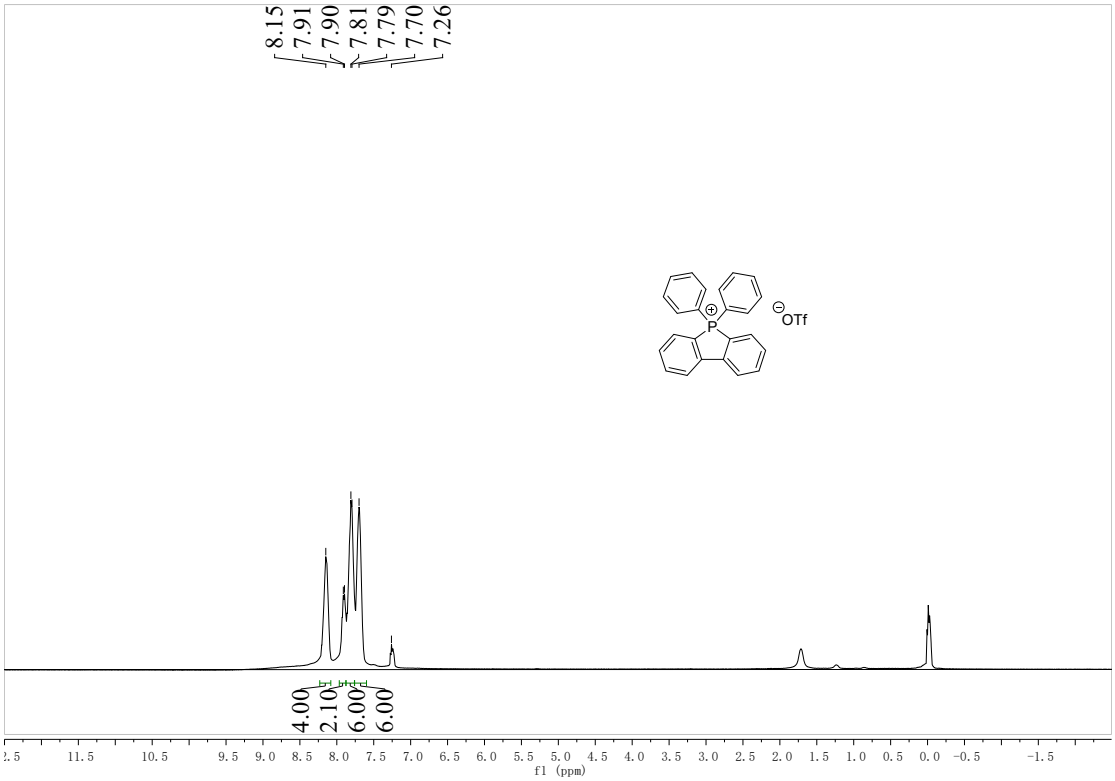
^{13}C NMR spectrum of 1w



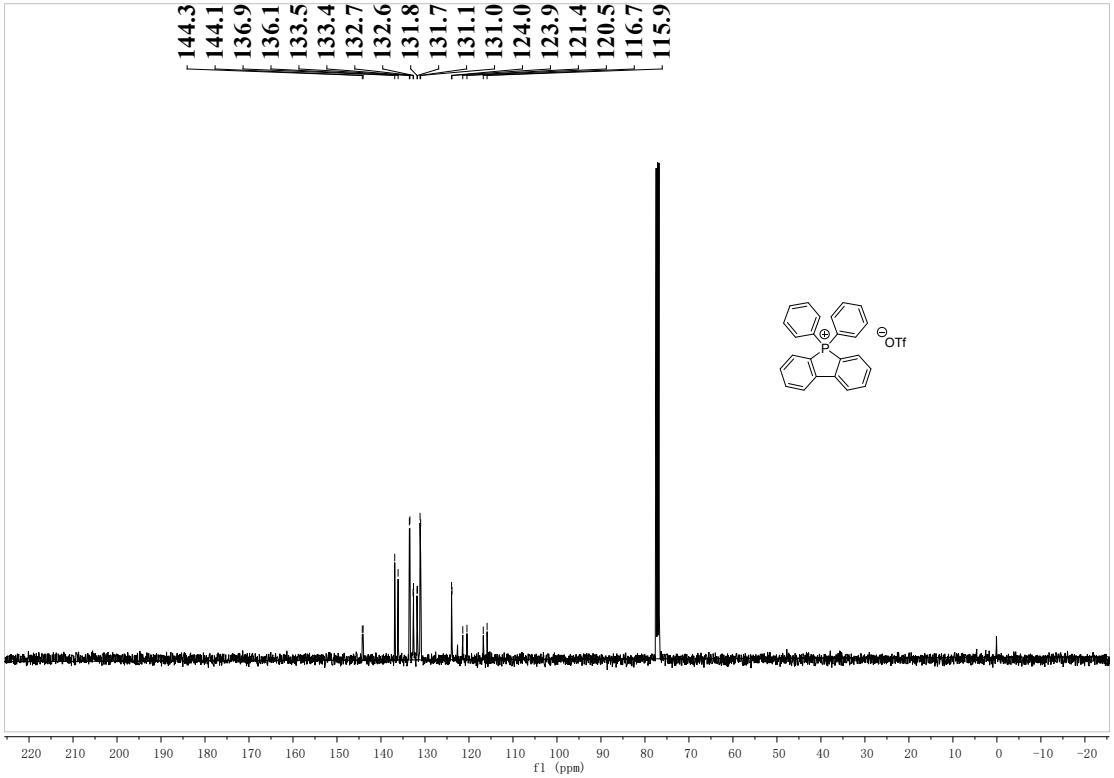
^{31}P NMR spectrum of 1w


-9.13
P
O
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
-3
f1 (ppm)

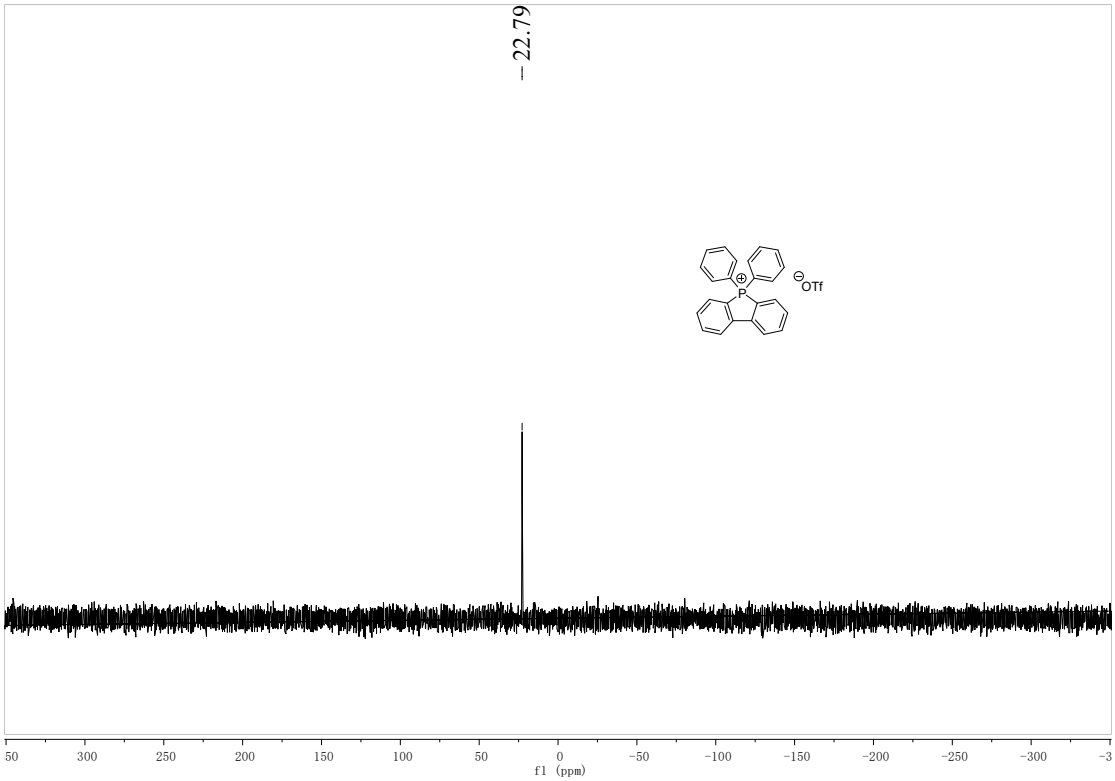
^{1}H NMR spectrum of 2a



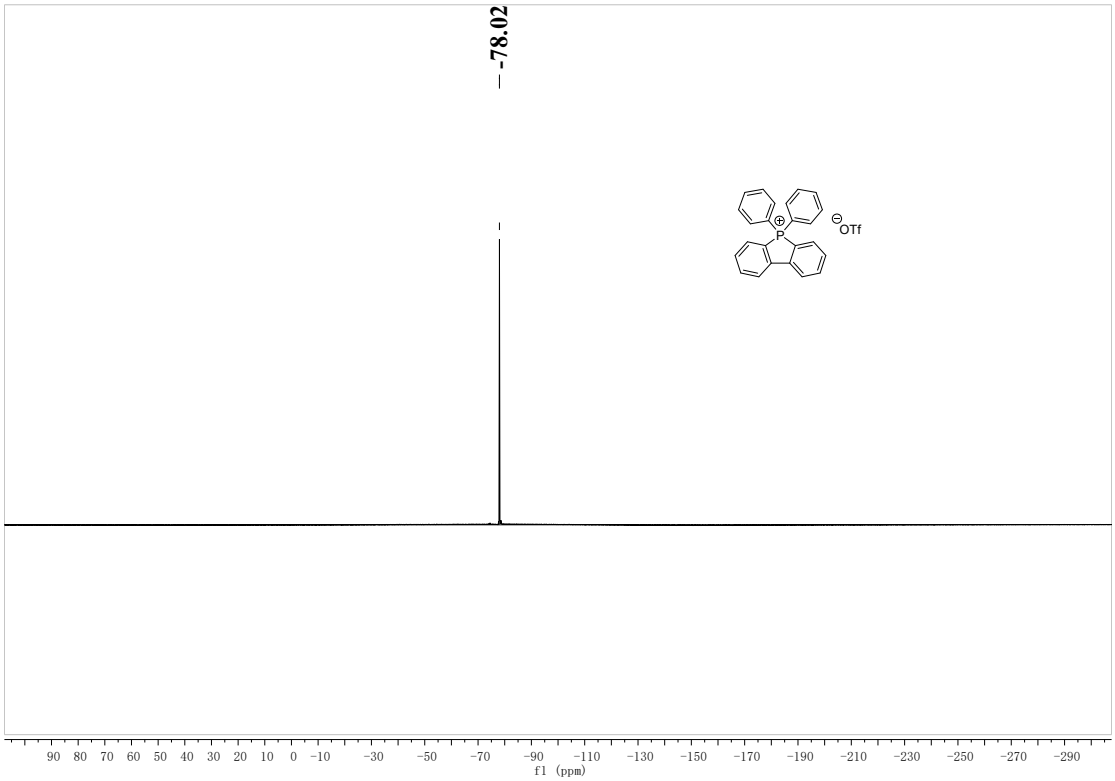
^{13}C NMR spectrum of 2a



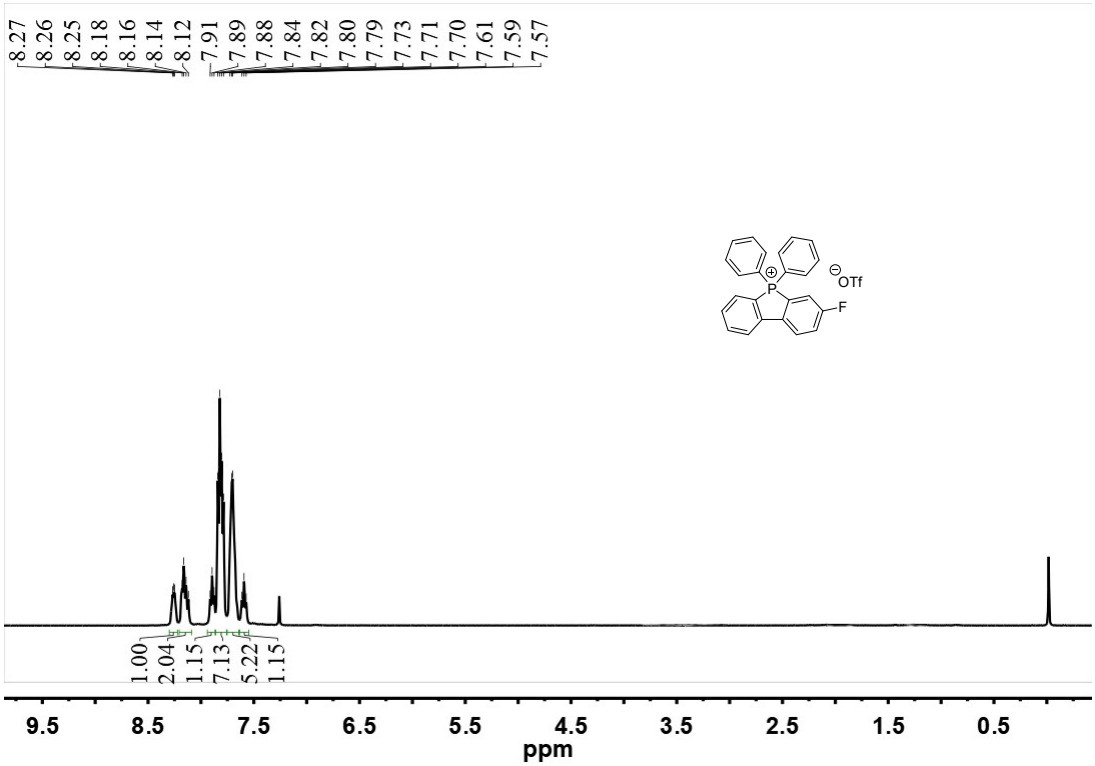
^{31}P NMR spectrum of 2a



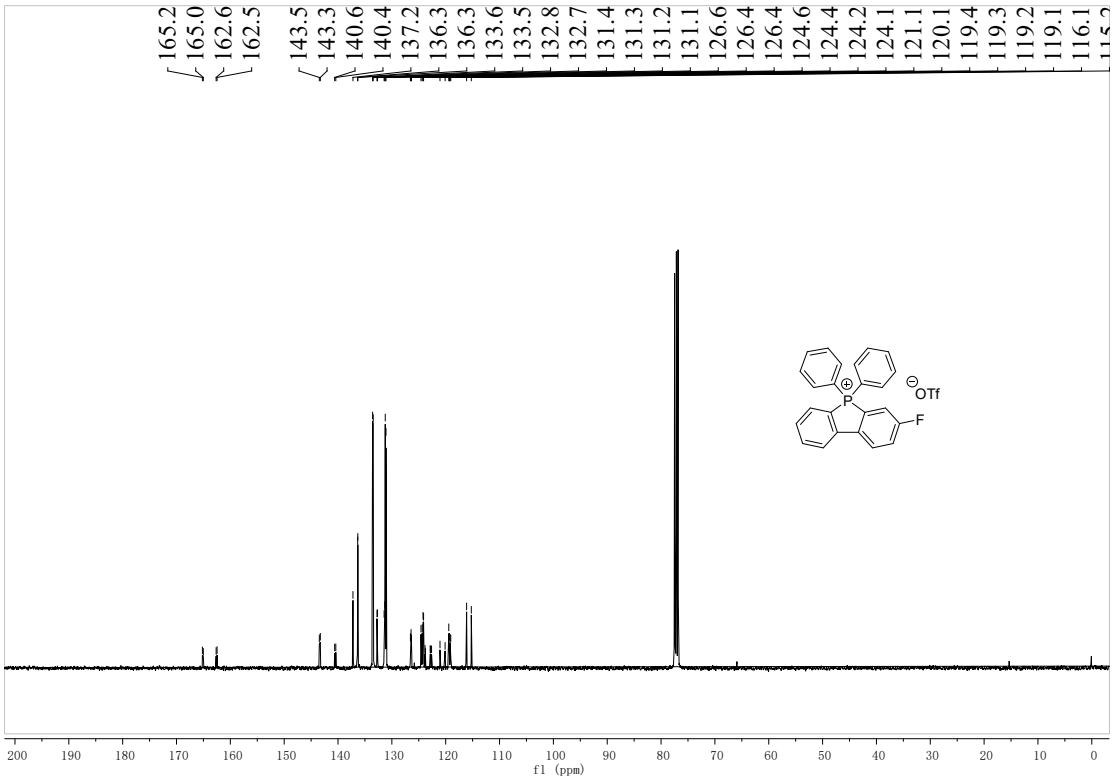
^{19}F NMR spectrum of 2a



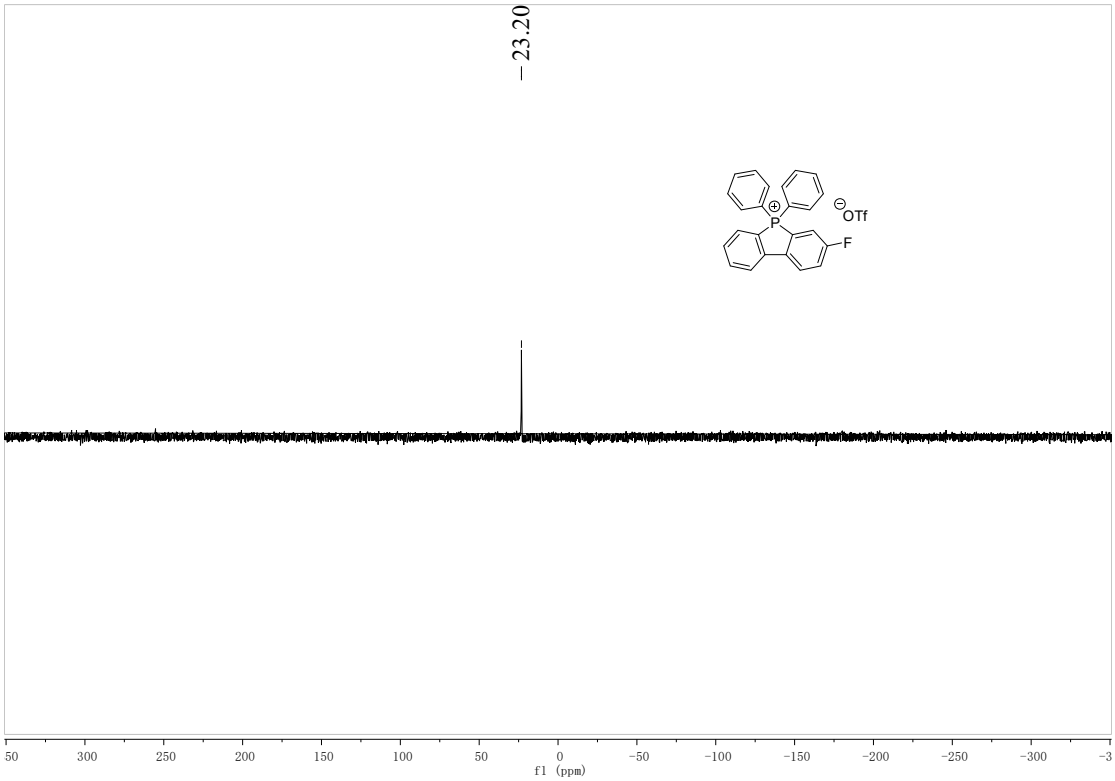
^{1}H NMR spectrum of 2b



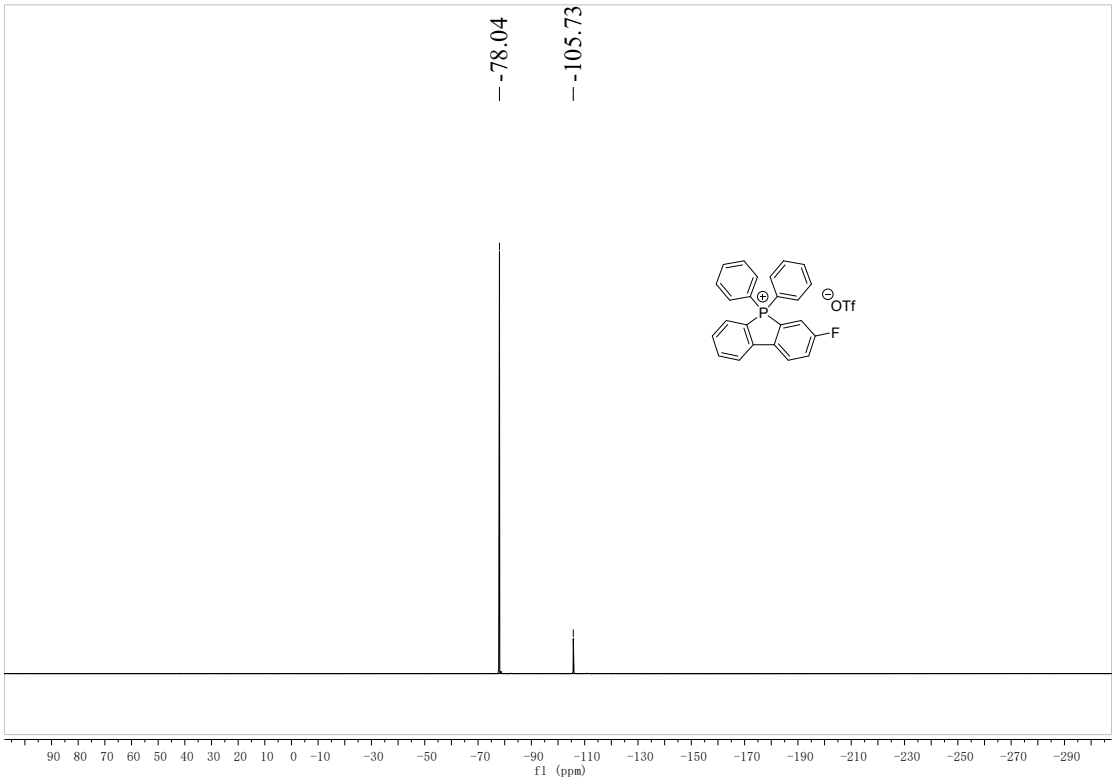
^{13}C NMR spectrum of 2b



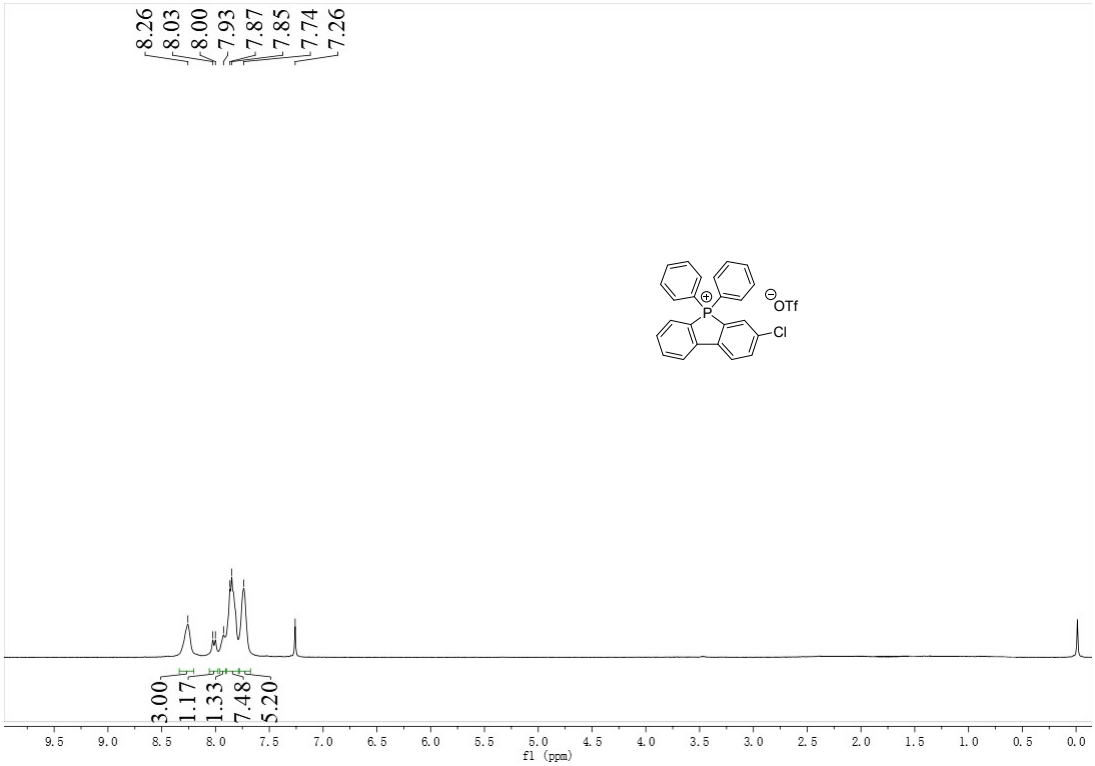
^{31}P NMR spectrum of 2b



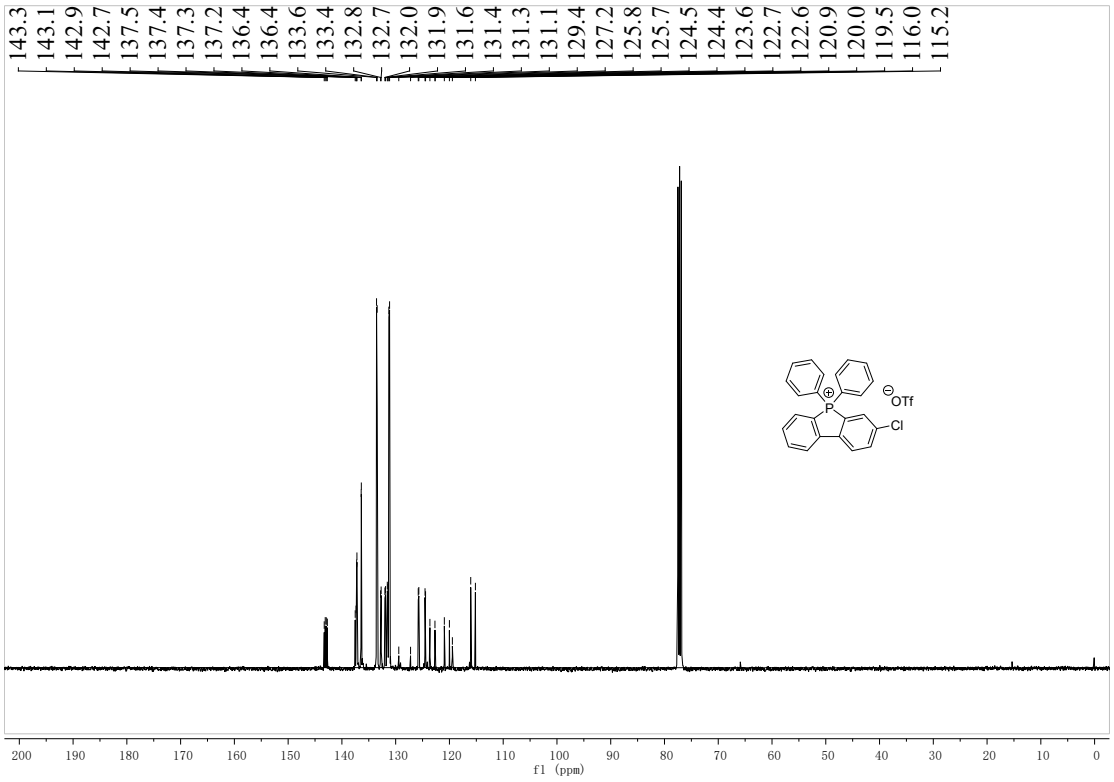
^{19}F NMR spectrum of 2b



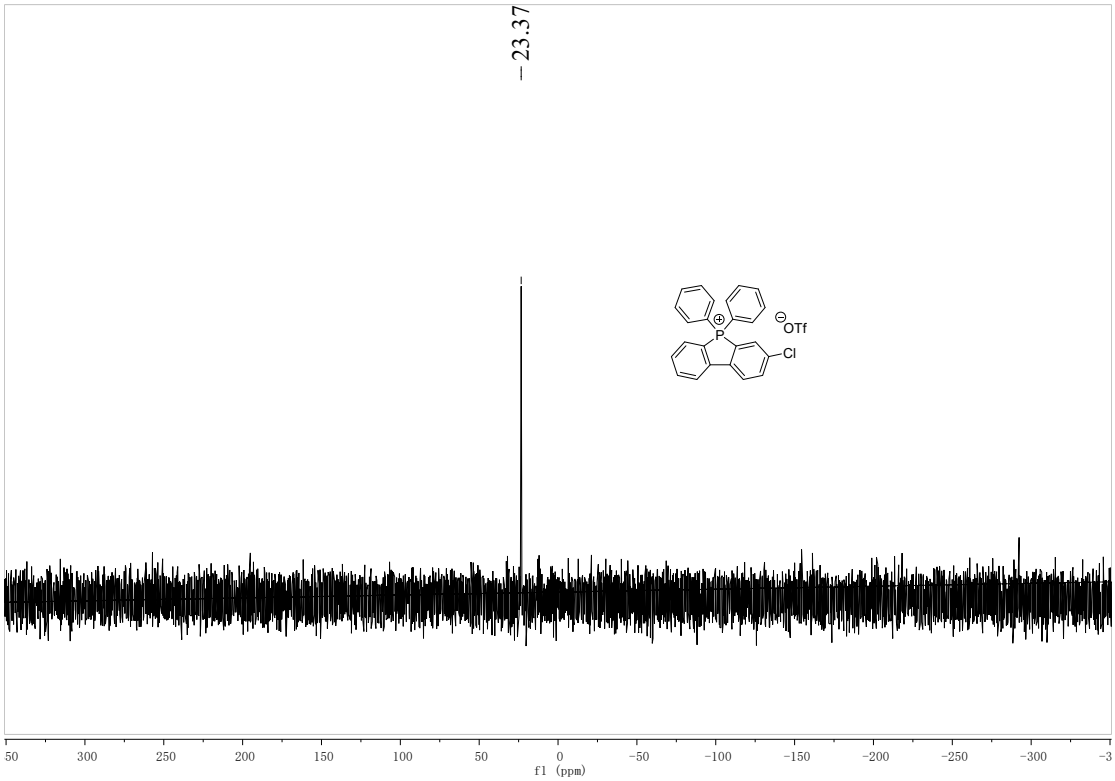
^{1}H NMR spectrum of 2c



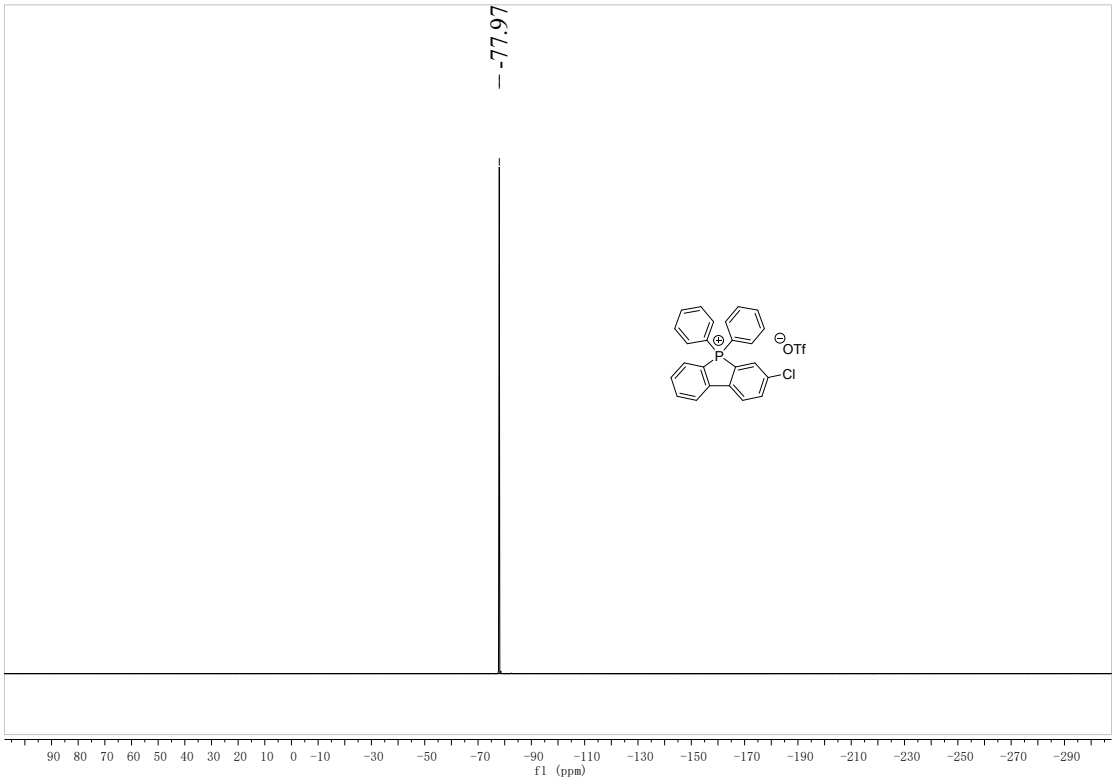
^{13}C NMR spectrum of 2c



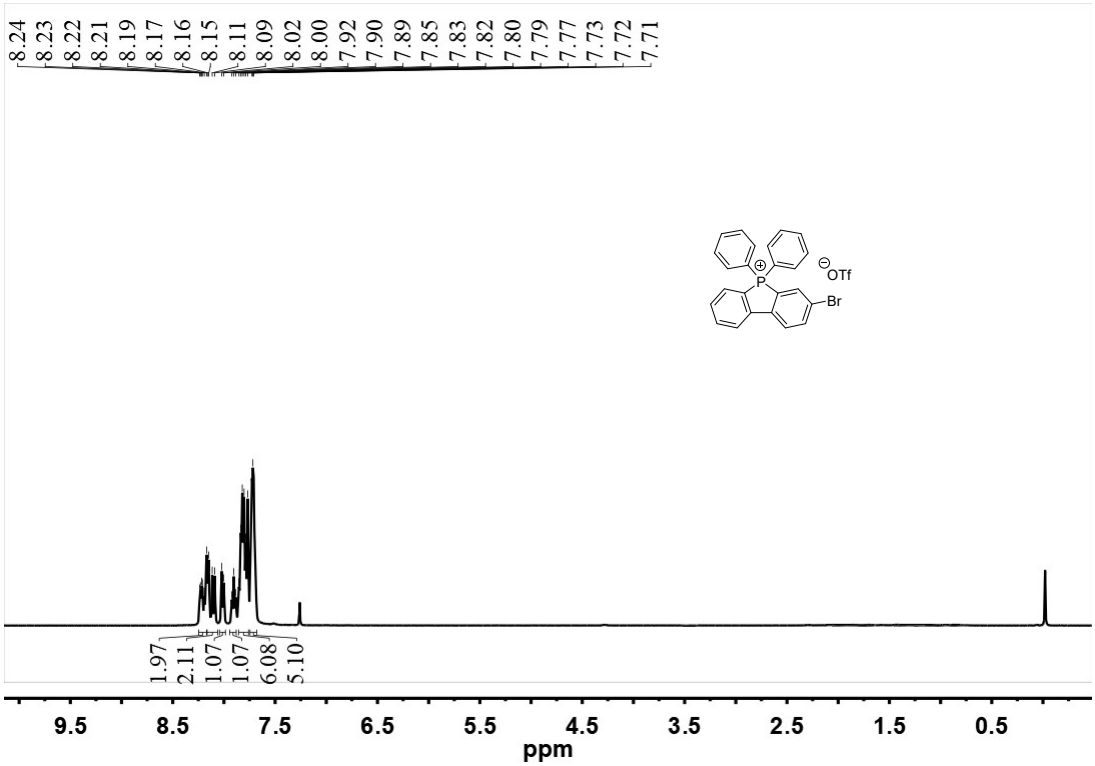
^{31}P NMR spectrum of 2c



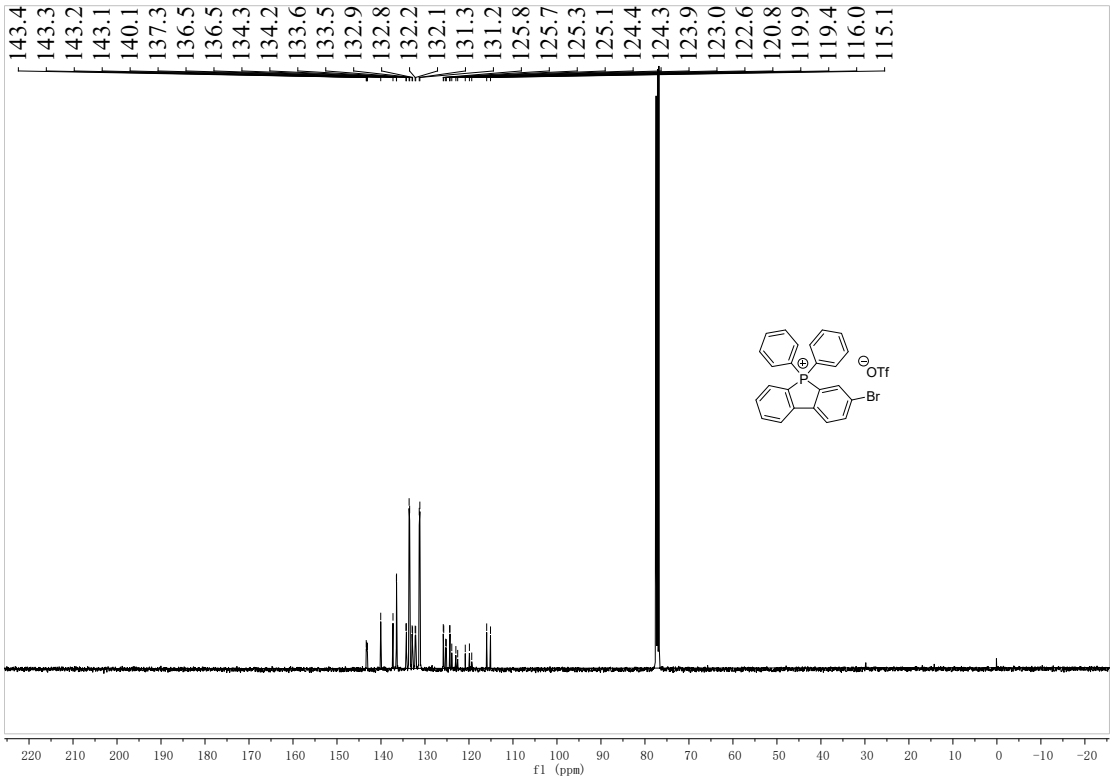
^{19}F NMR spectrum of 2c



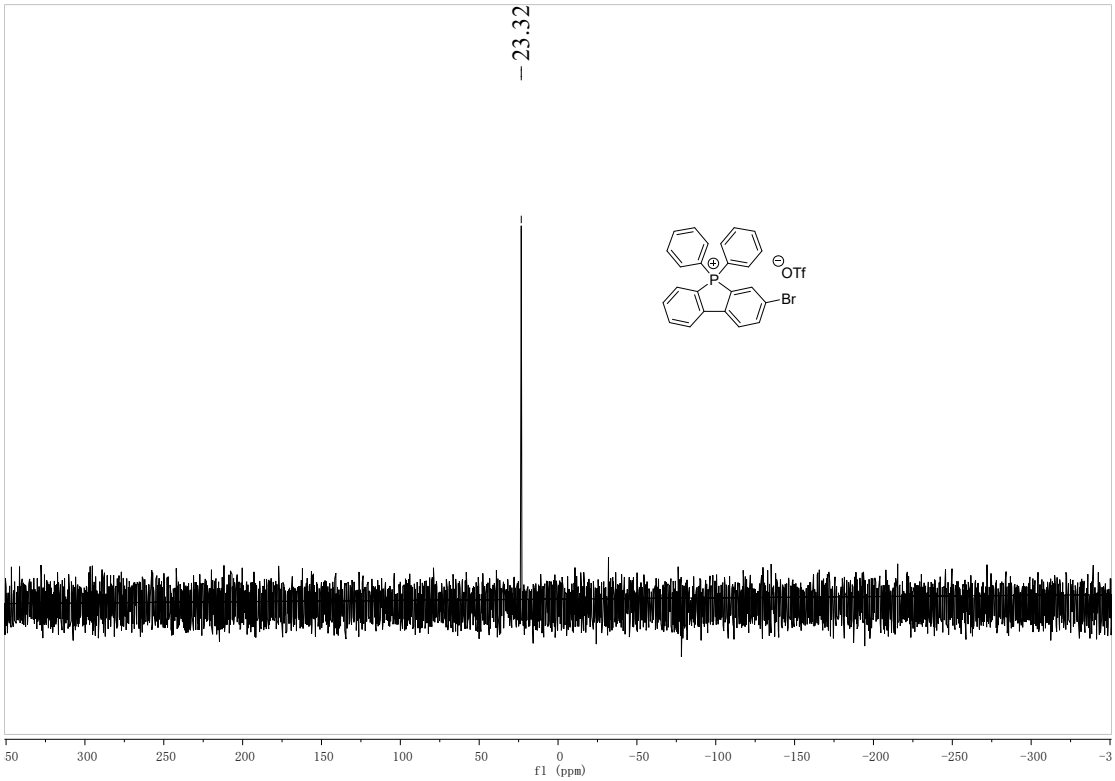
^{1}H NMR spectrum of 2d



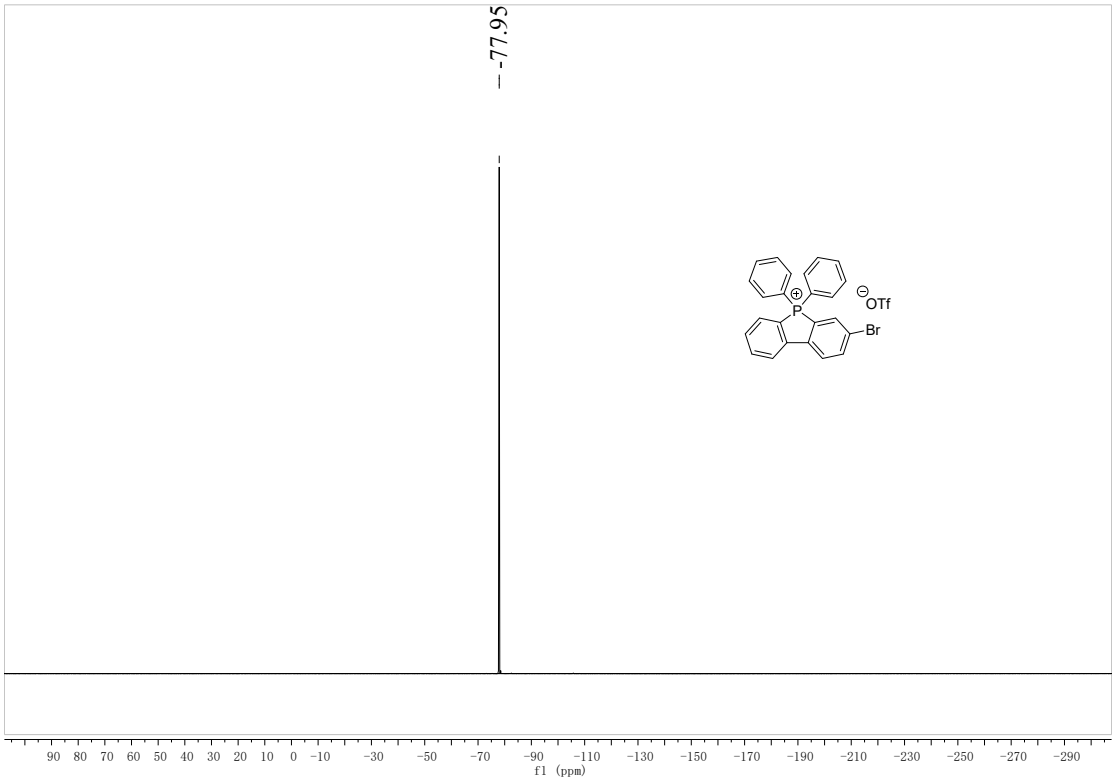
^{13}C NMR spectrum of 2d



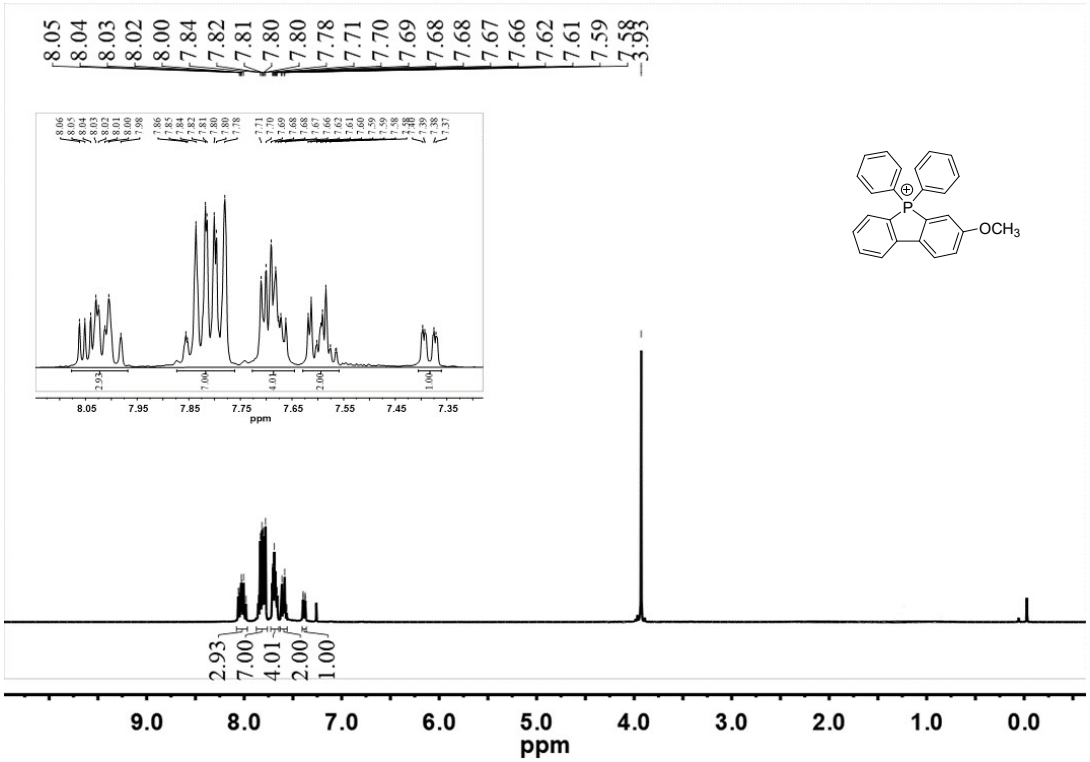
^{31}P NMR spectrum of 2d



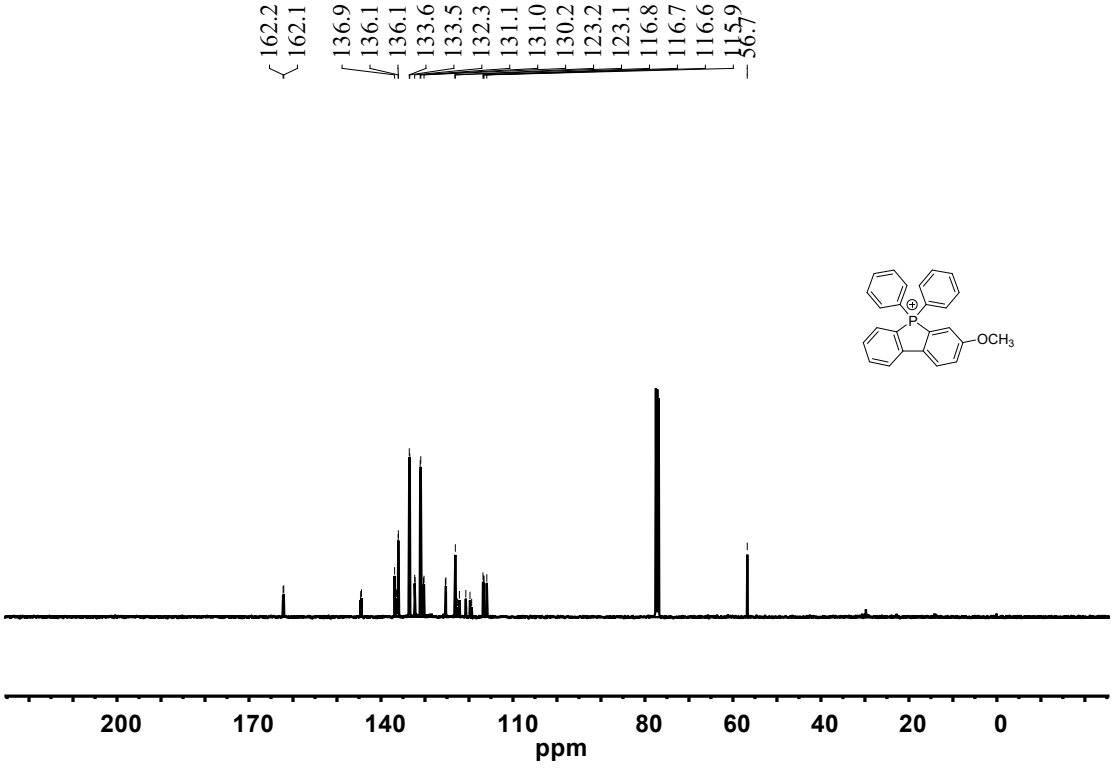
^{19}F NMR spectrum of 2d



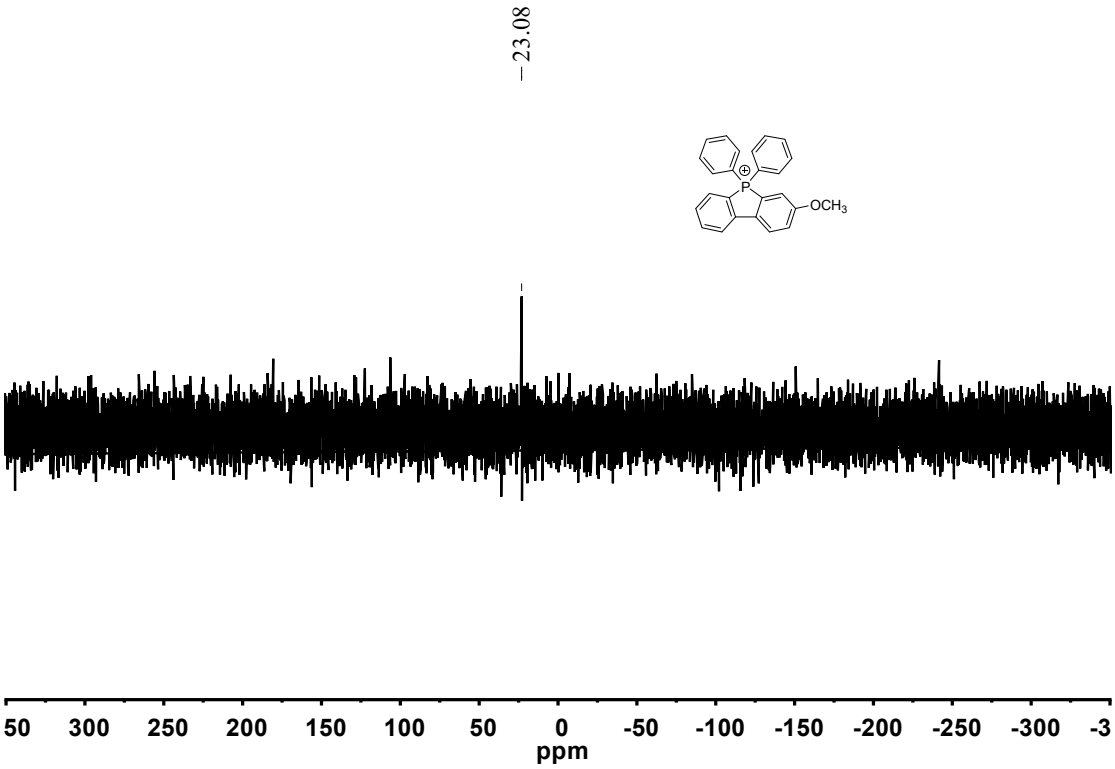
^{1}H NMR spectrum of 2e



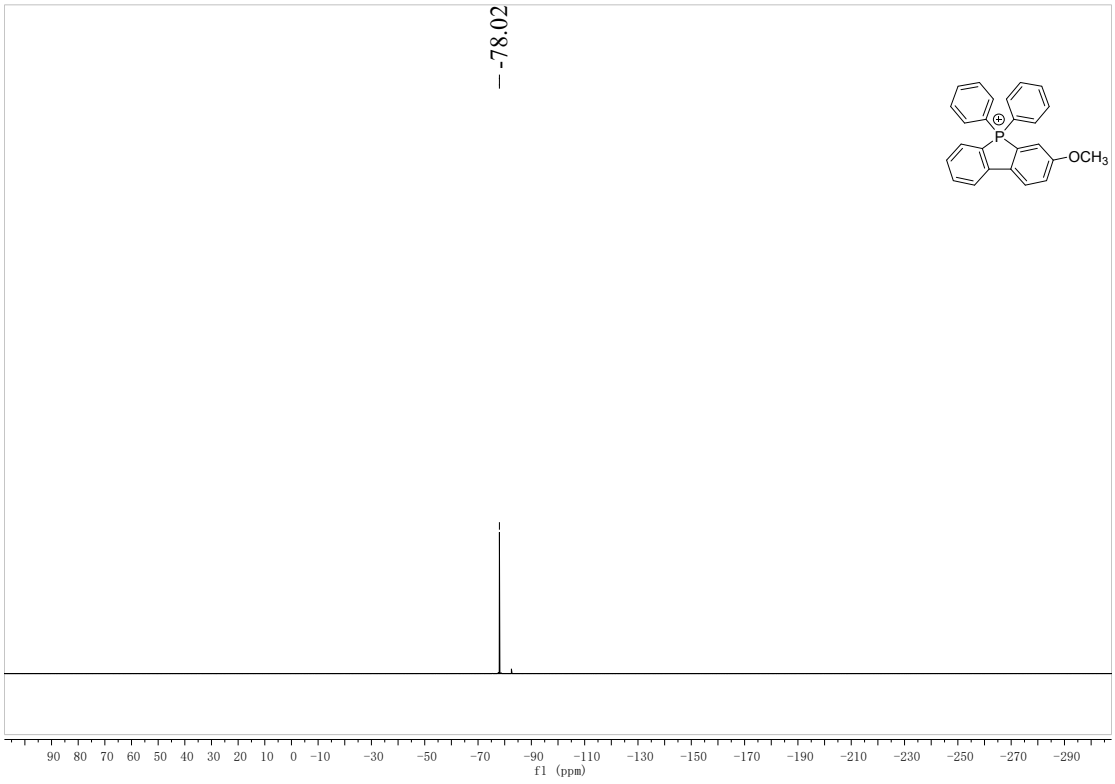
^{13}C NMR spectrum of 2e



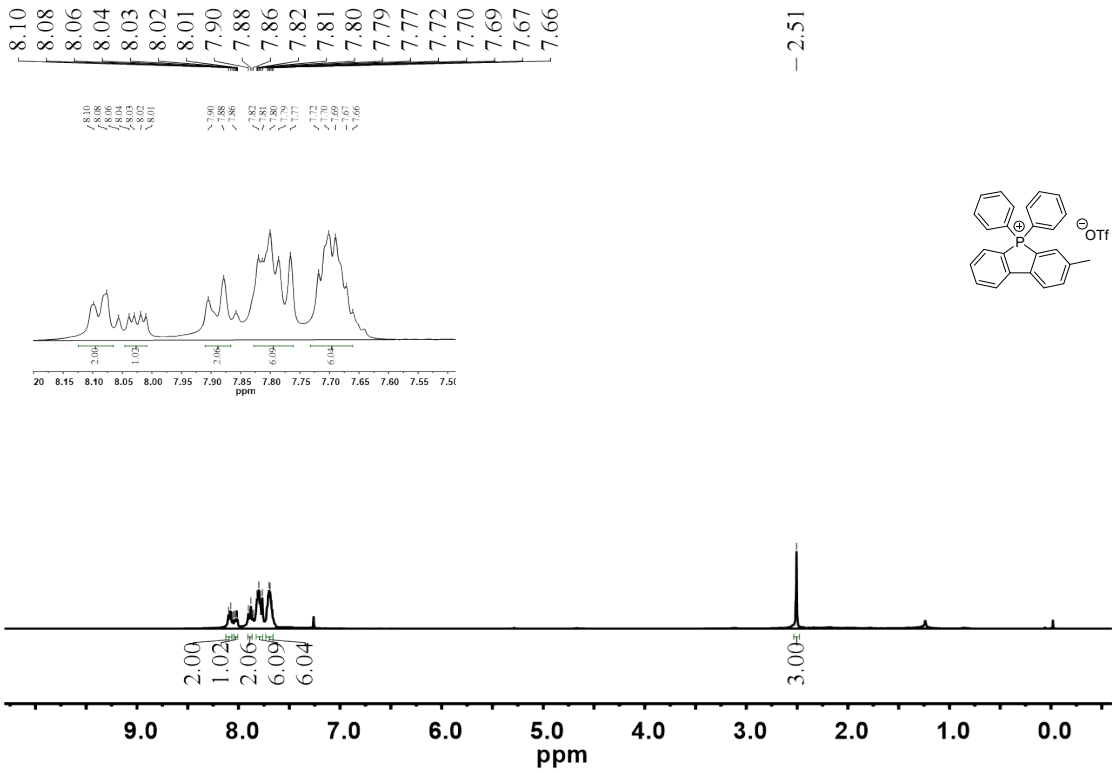
^{31}P NMR spectrum of 2e



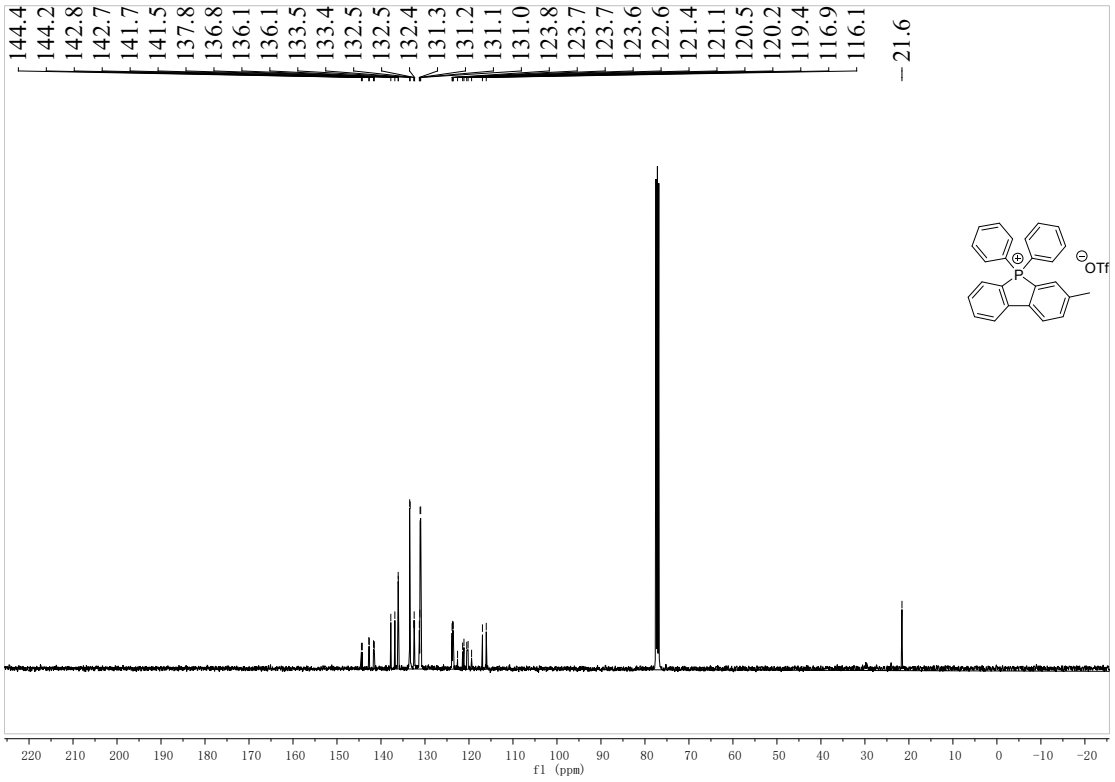
^{19}F NMR spectrum of 2e



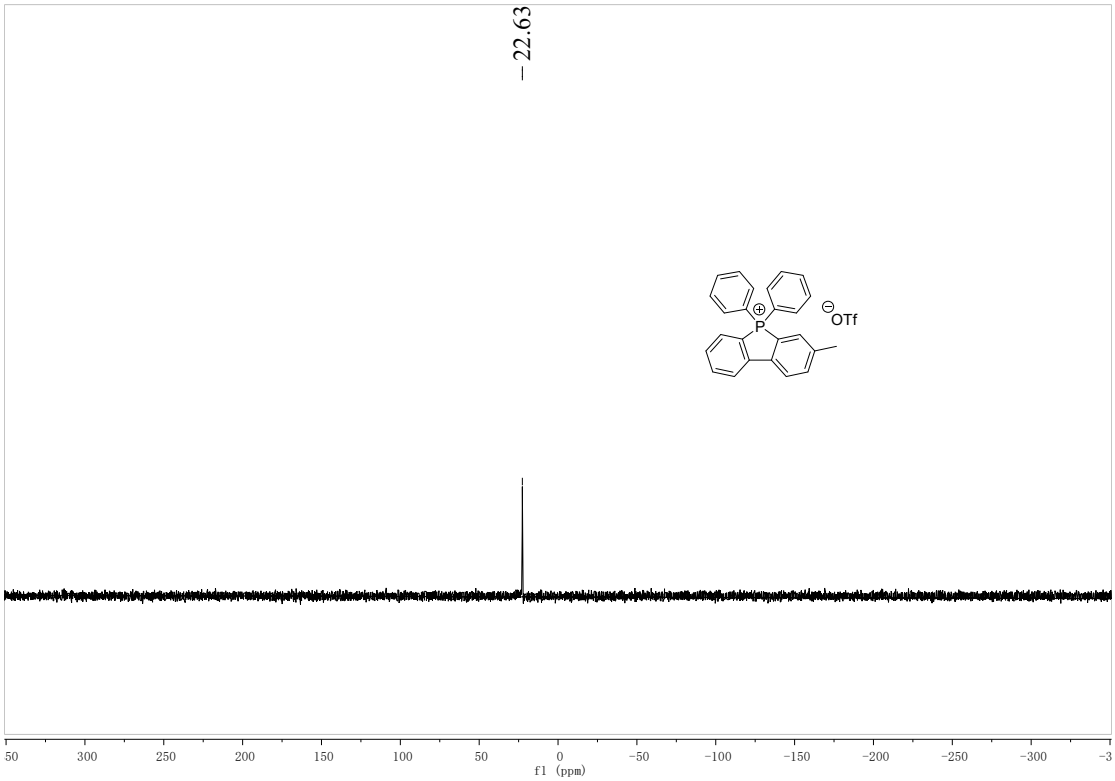
^{1}H NMR spectrum of 2f



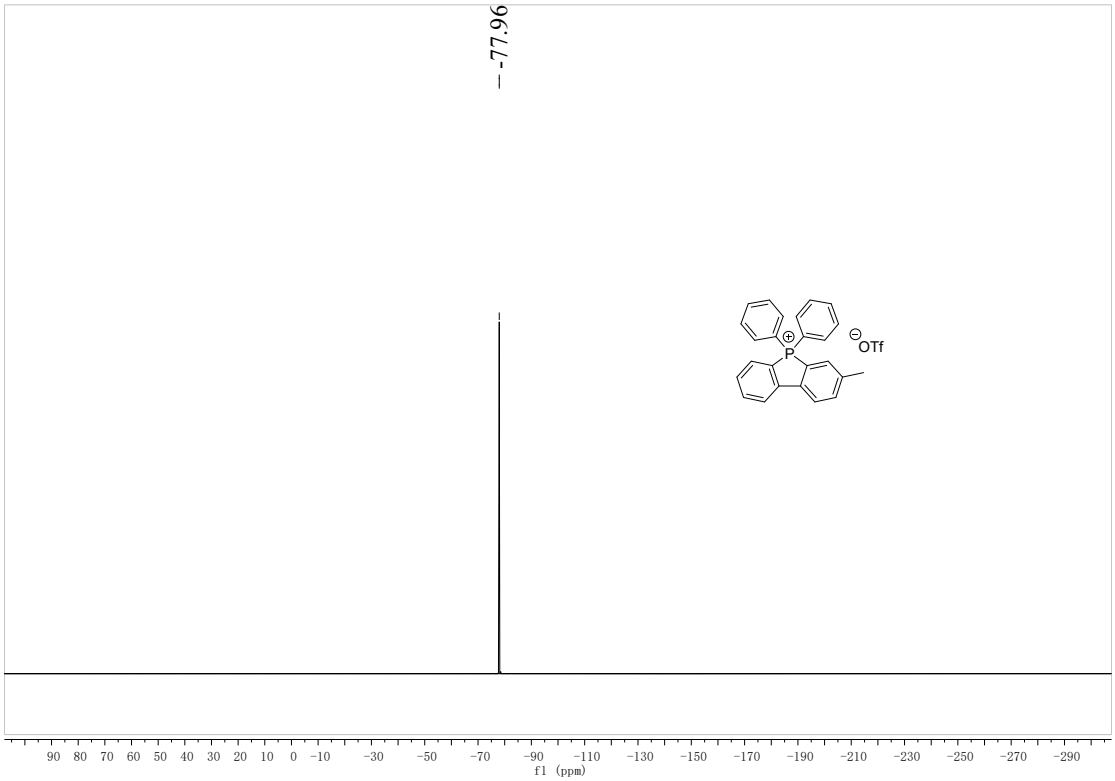
^{13}C NMR spectrum of 2f



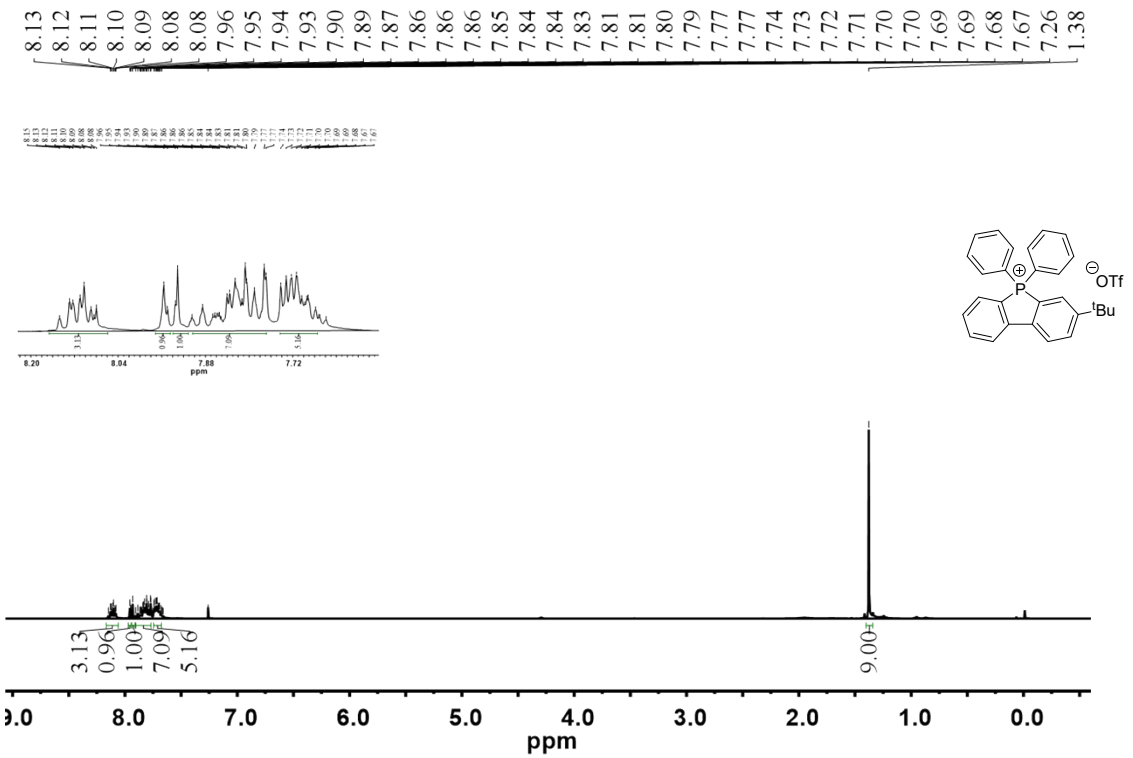
^{31}P NMR spectrum of 2f



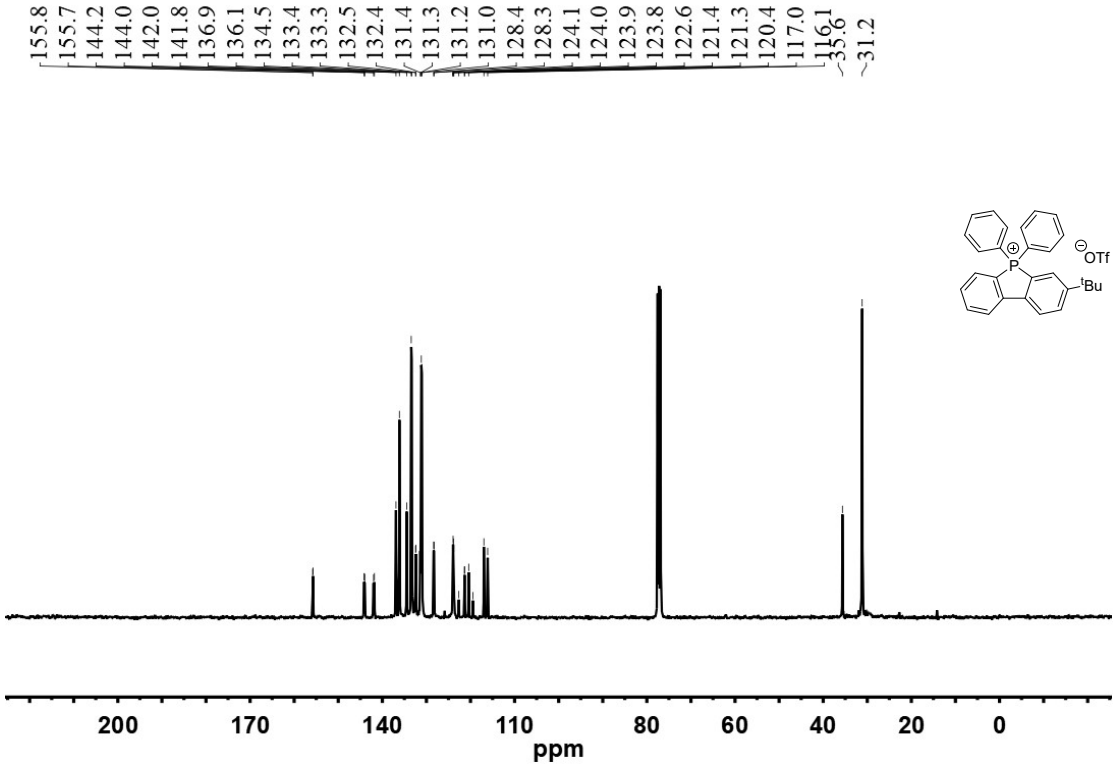
^{19}F NMR spectrum of 2f



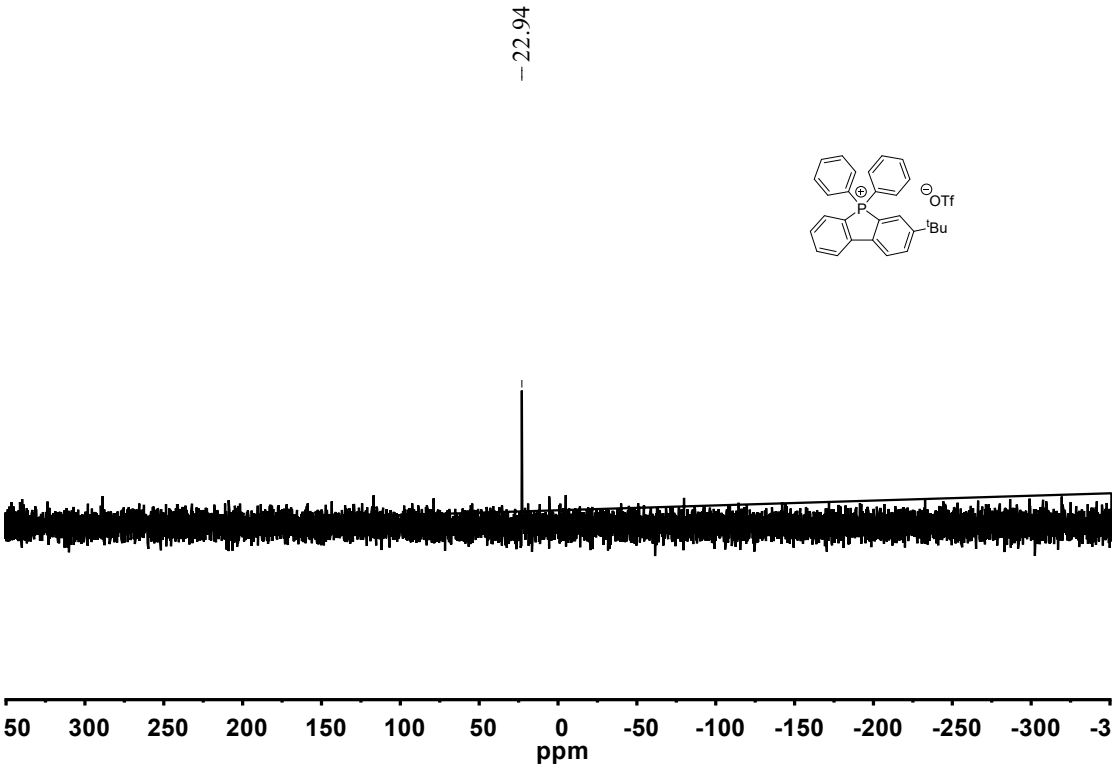
^{1}H NMR spectrum of 2g



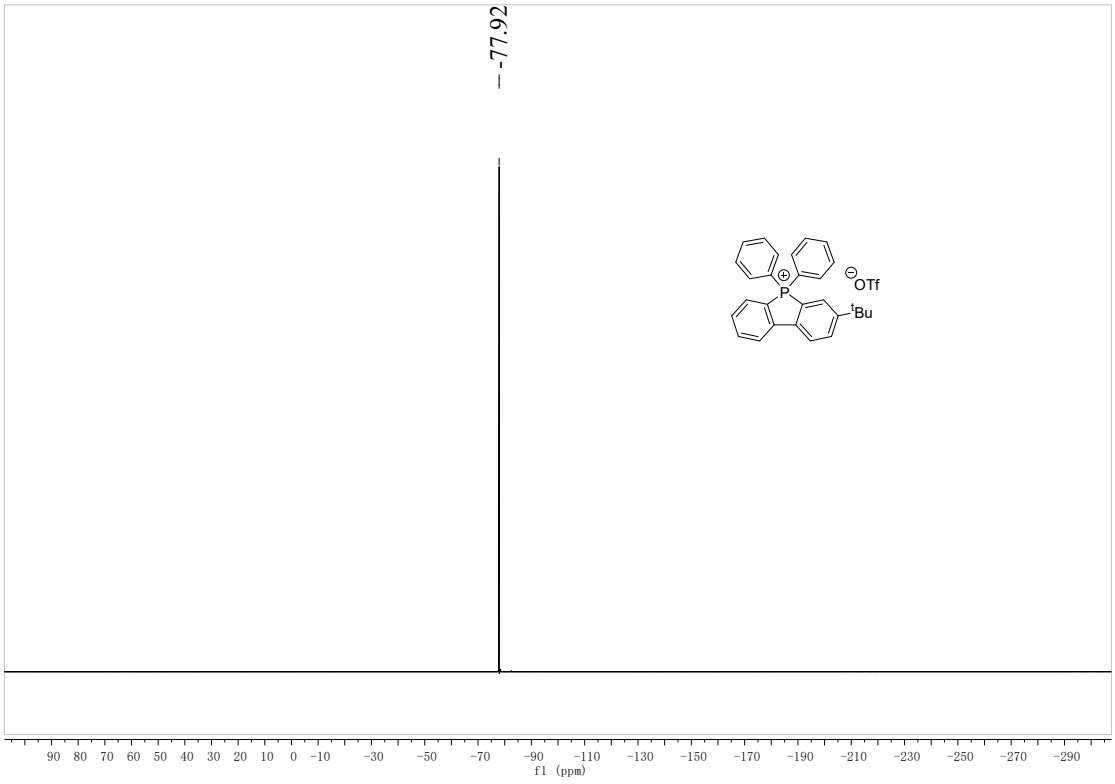
^{13}C NMR spectrum of 2g



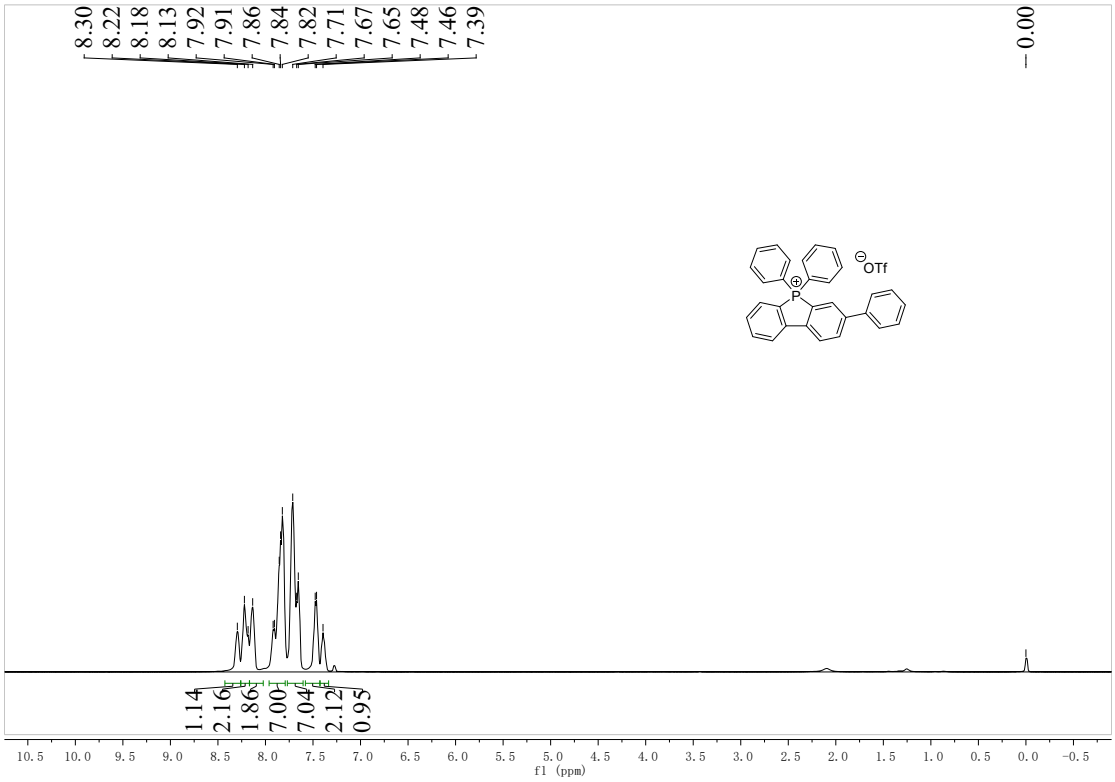
^{31}P NMR spectrum of 2g



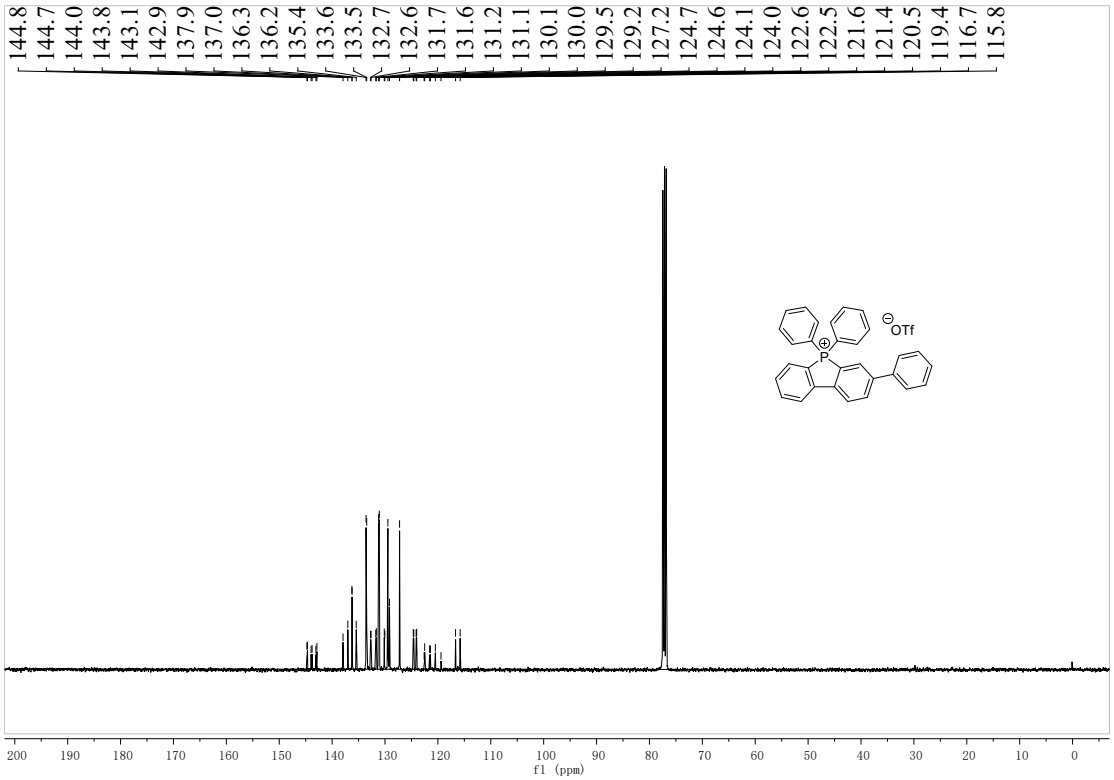
^{19}F NMR spectrum of 2g



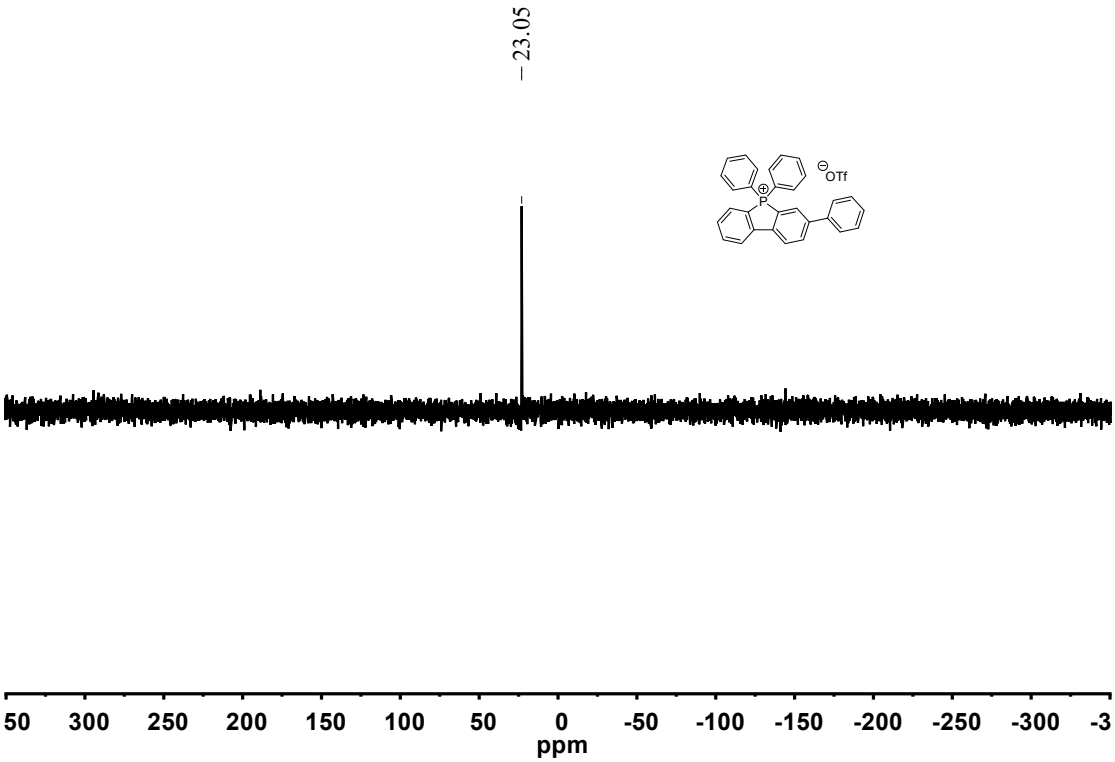
^{1}H NMR spectrum of 2h



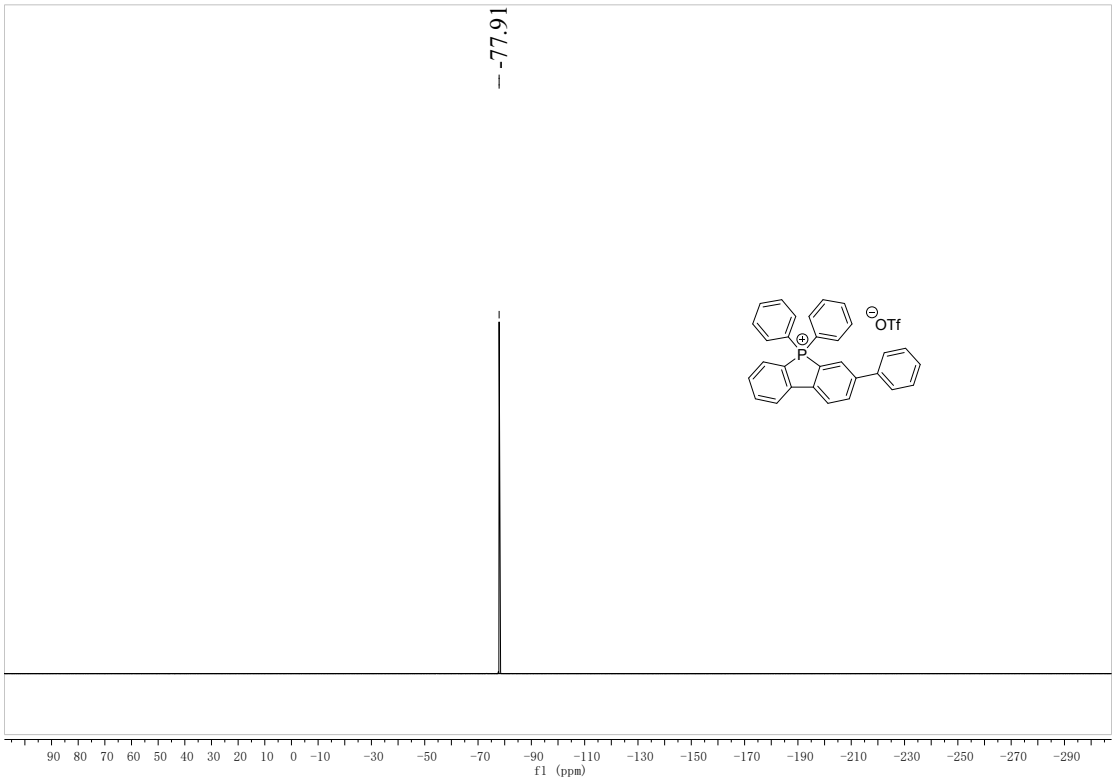
^{13}C NMR spectrum of 2h



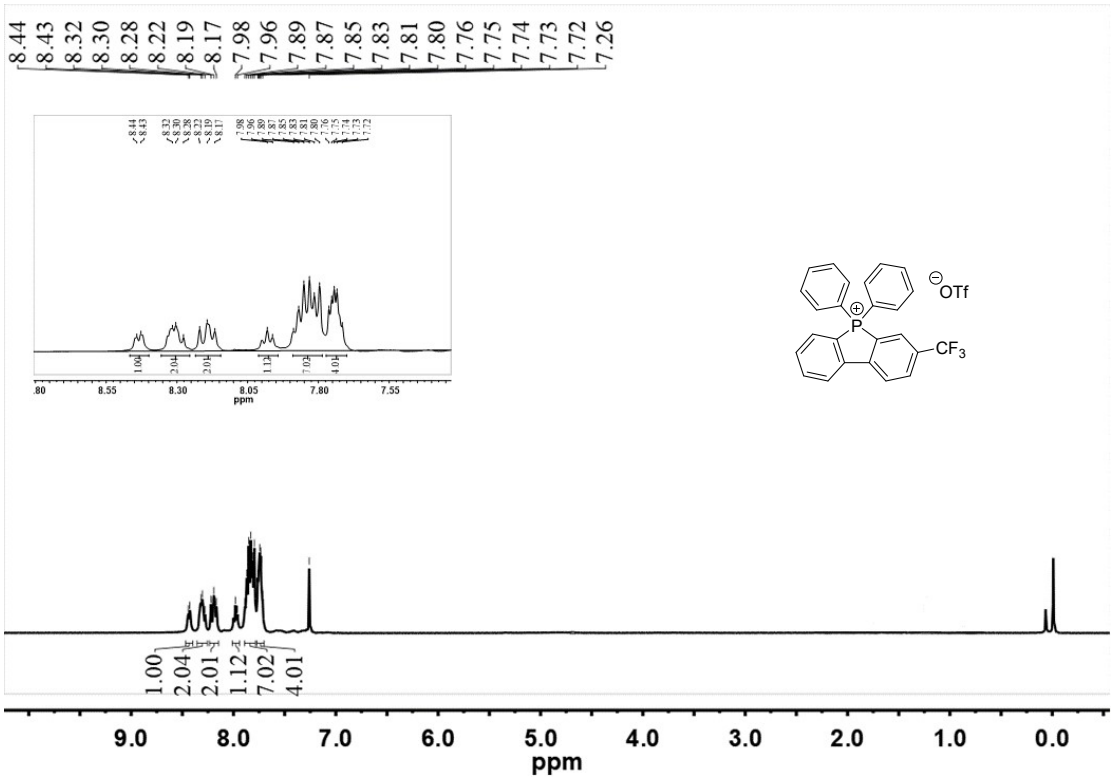
^{31}P NMR spectrum of 2h



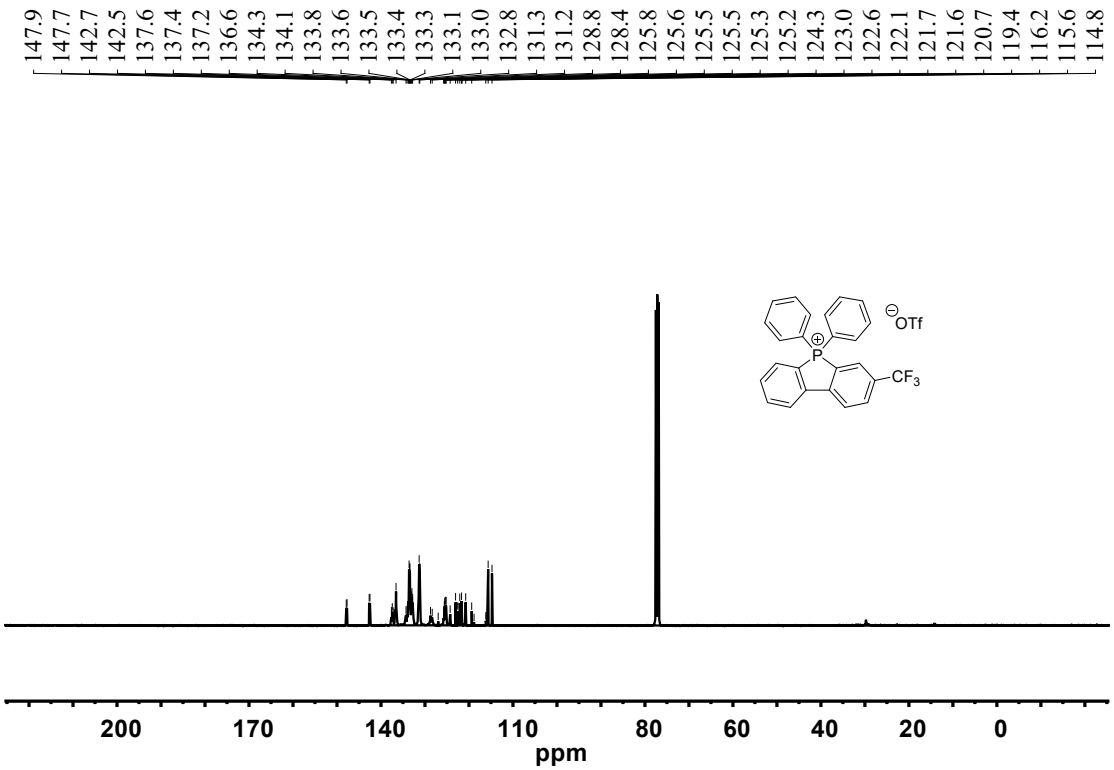
^{19}F NMR spectrum of 2h



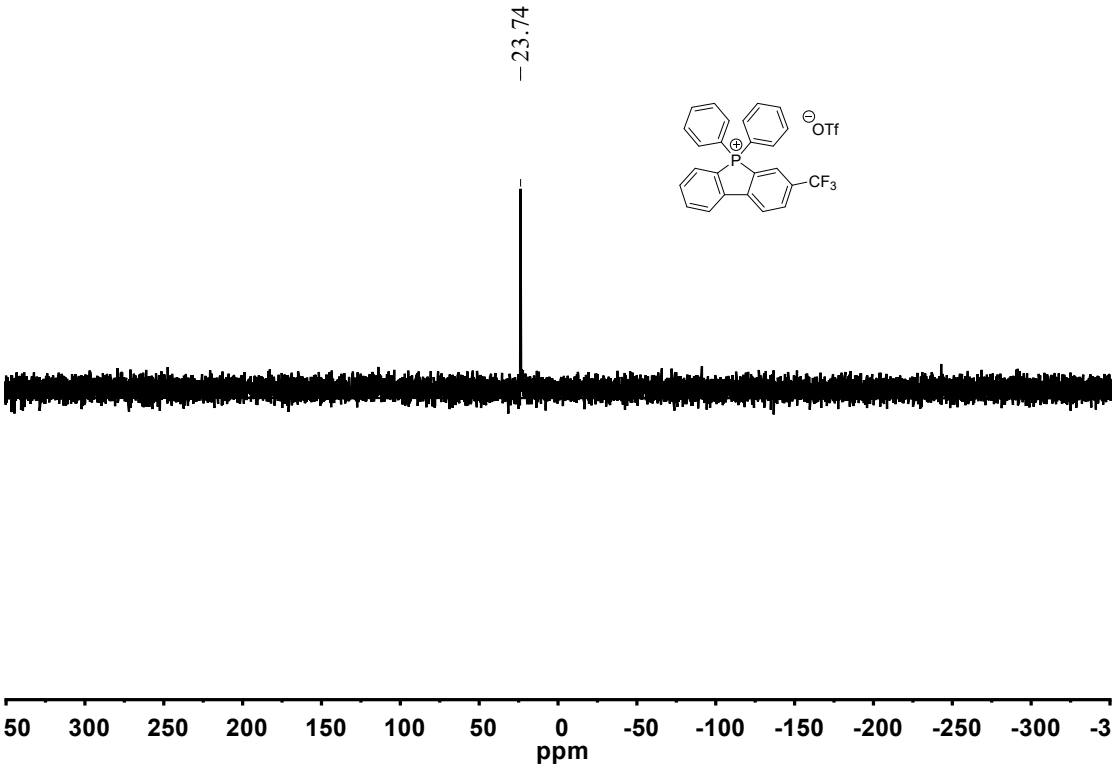
^{1}H NMR spectrum of 2i



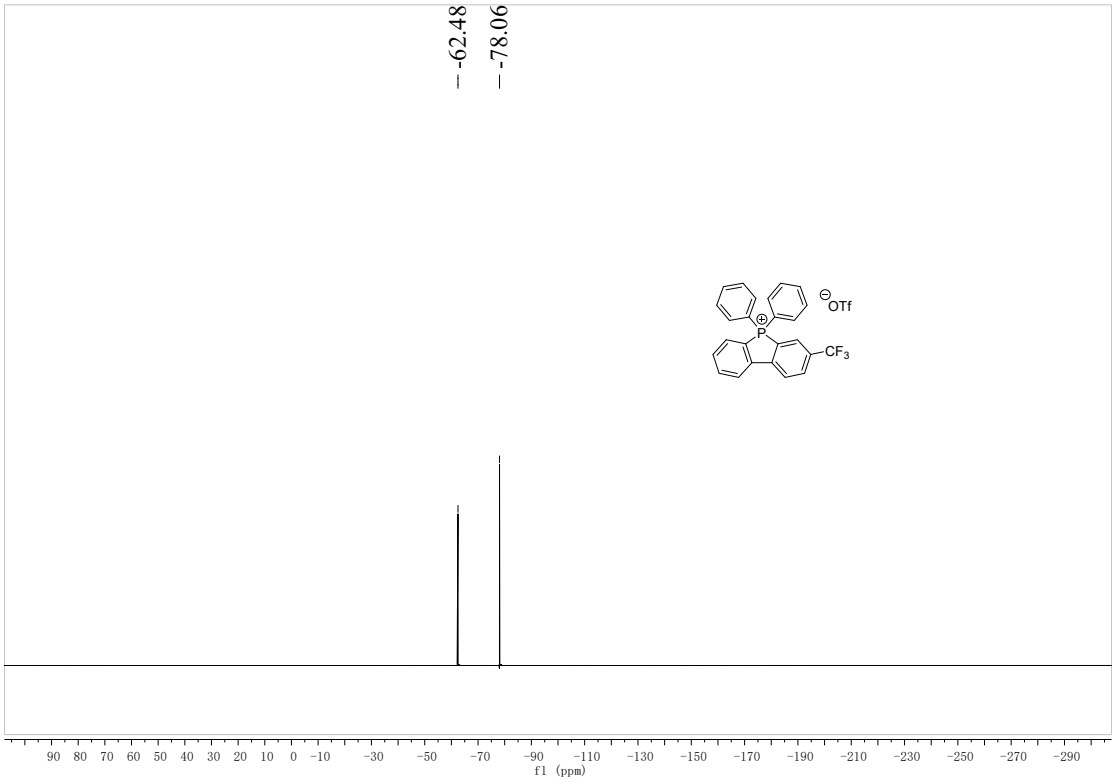
^{13}C NMR spectrum of 2i



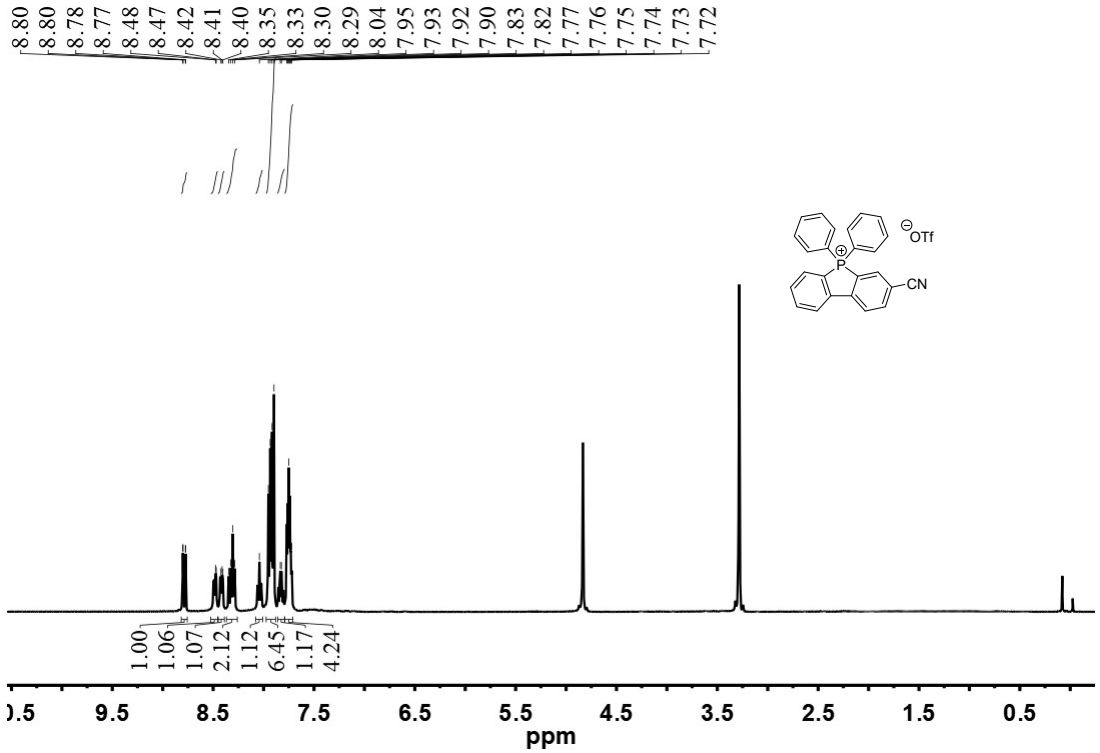
^{31}P NMR spectrum of 2i



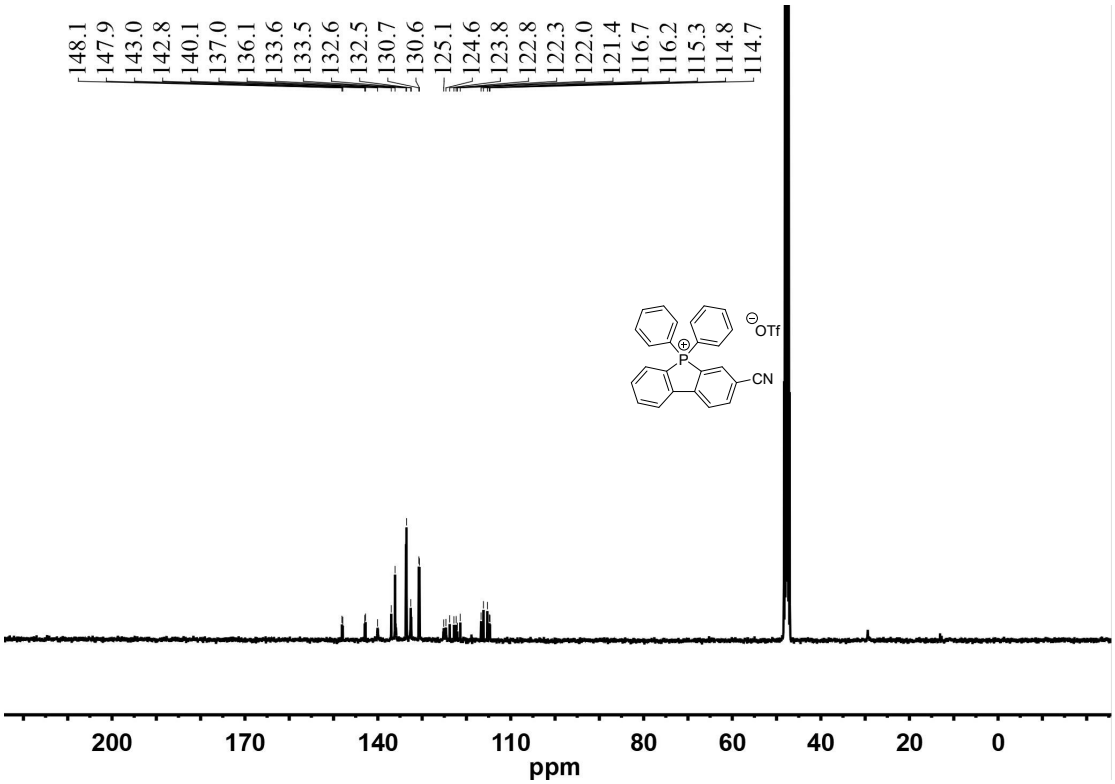
^{19}F NMR spectrum of 2i



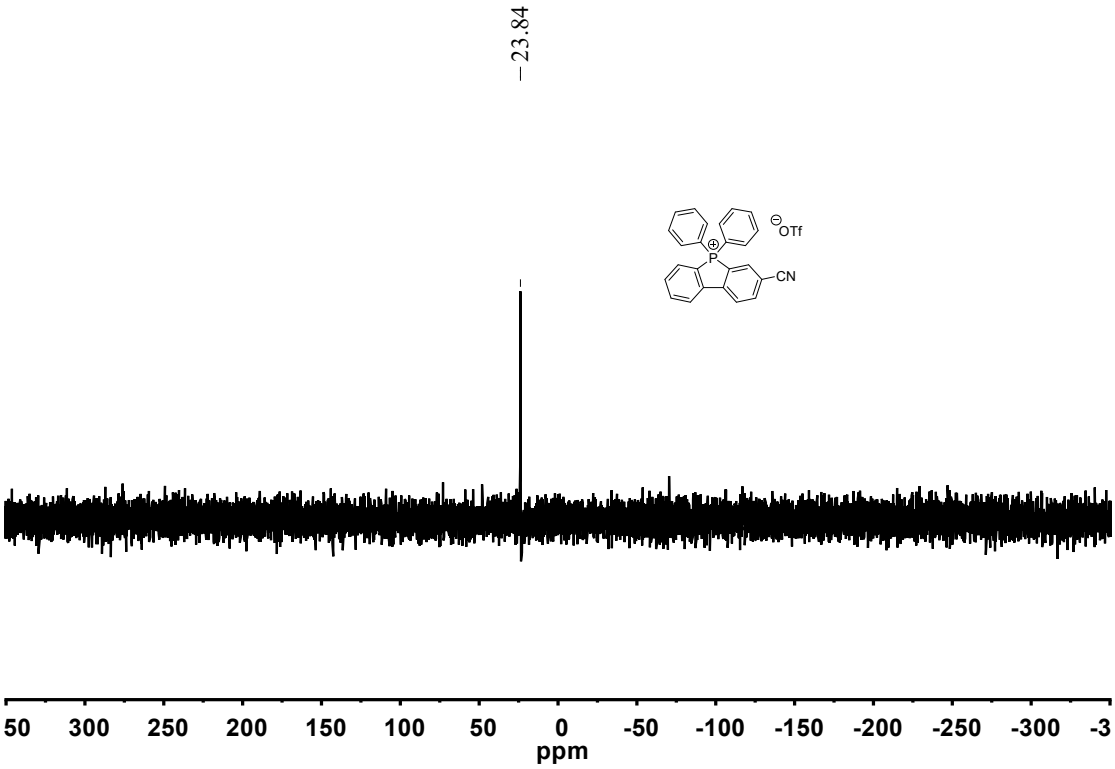
^{1}H NMR spectrum of 2j



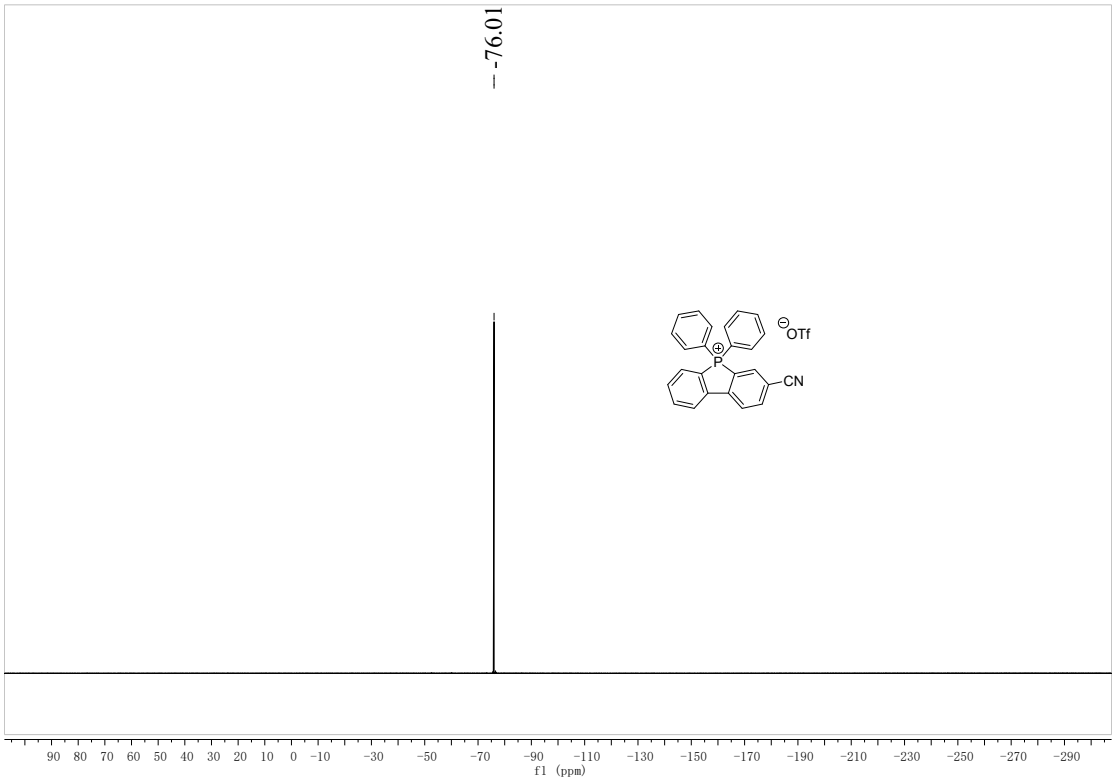
^{13}C NMR spectrum of 2j



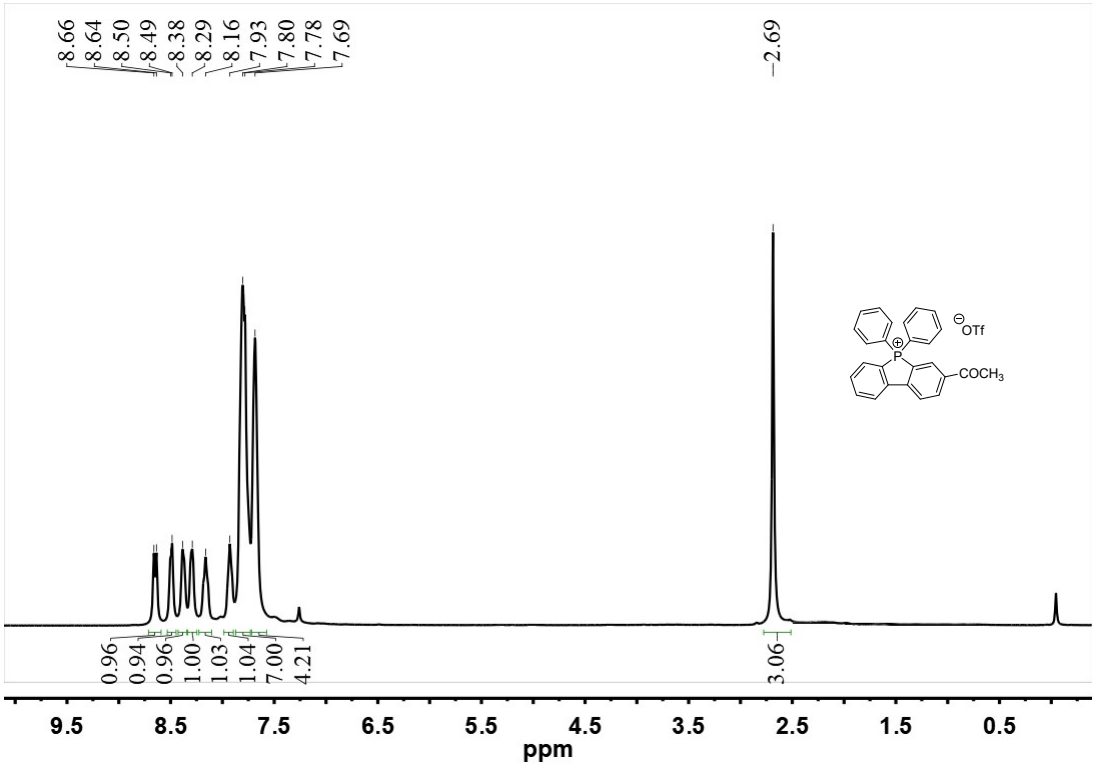
^{31}P NMR spectrum of 2j



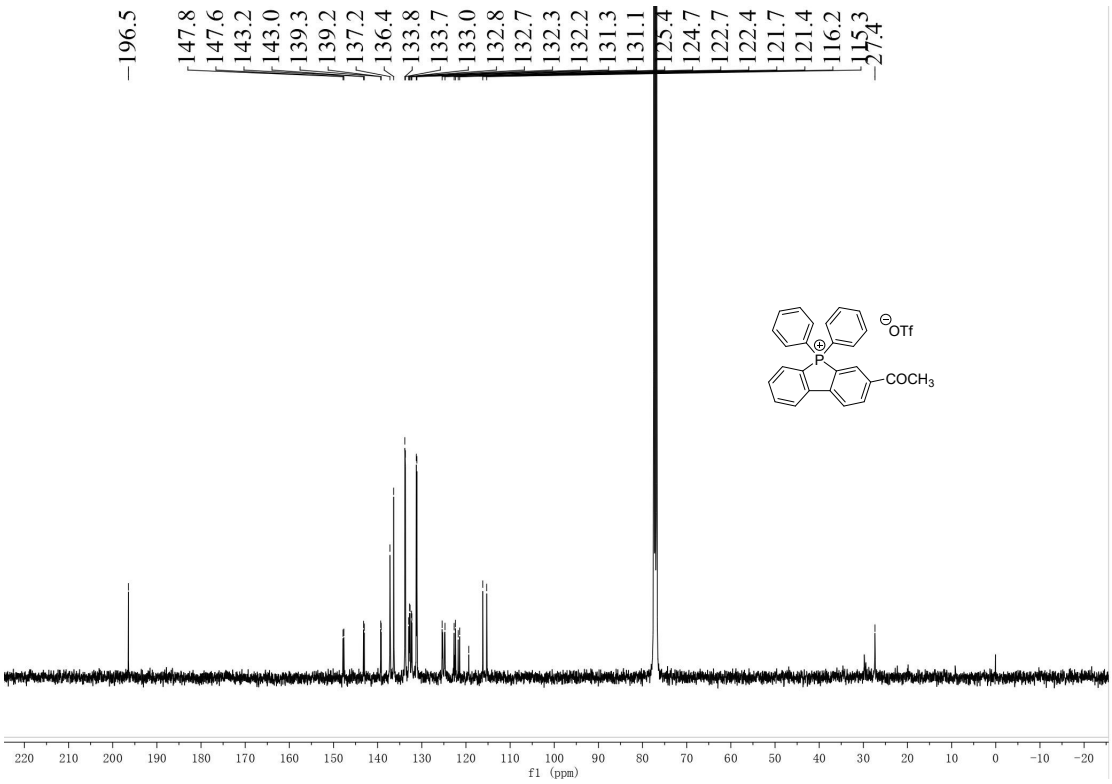
^{19}F NMR spectrum of 2j



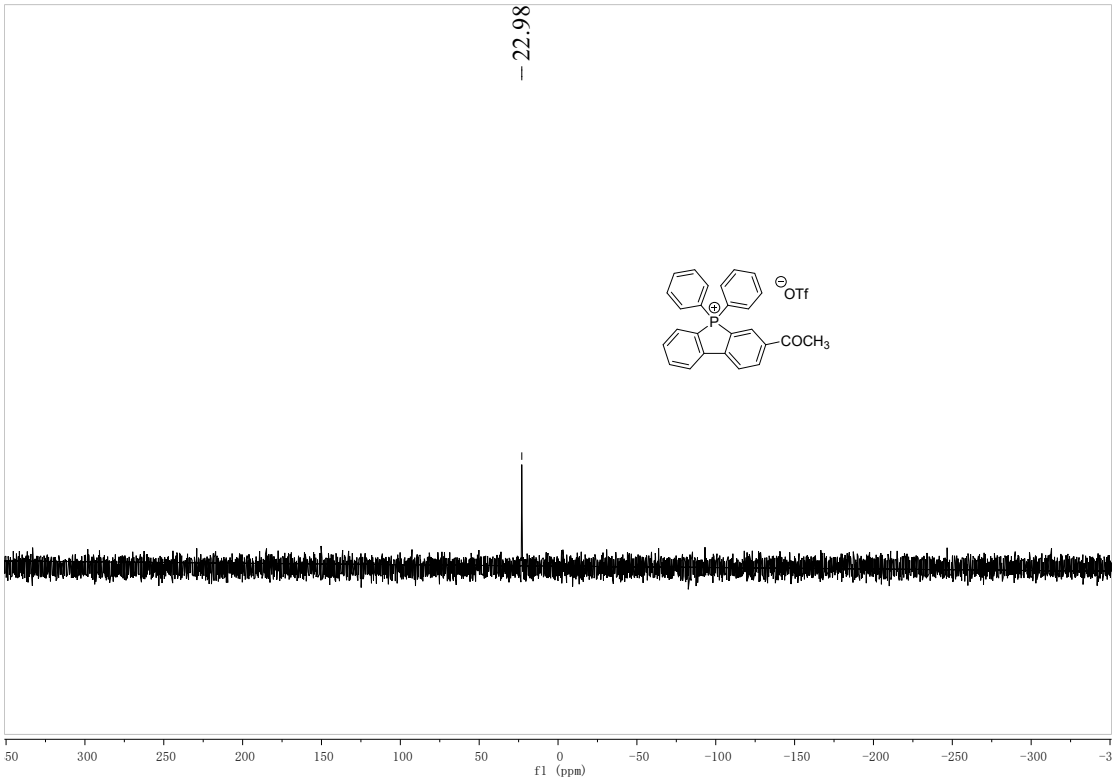
^{1}H NMR spectrum of 2k



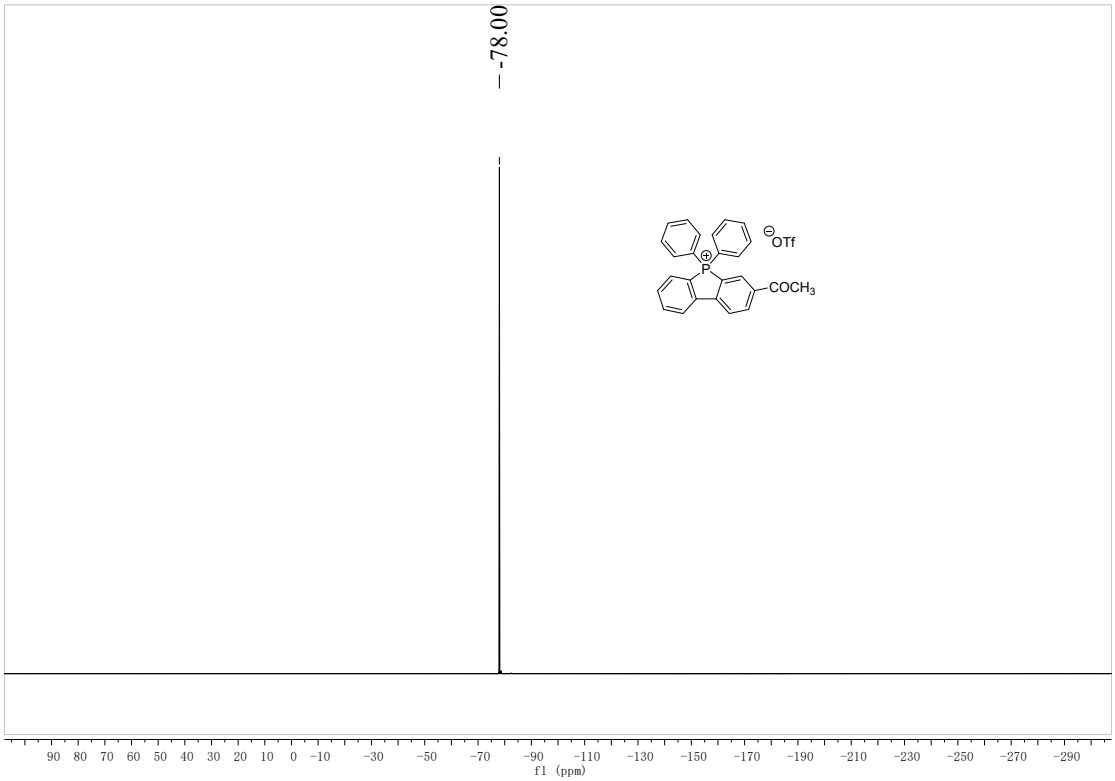
^{13}C NMR spectrum of 2k



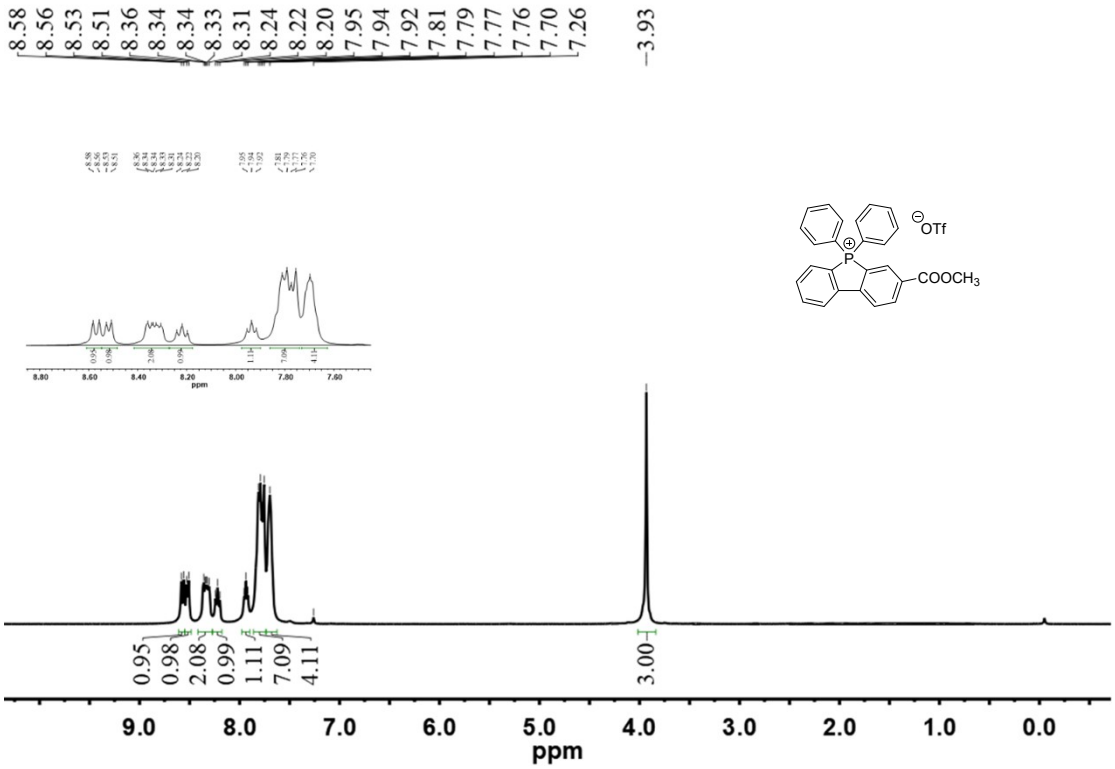
^{31}P NMR spectrum of 2k



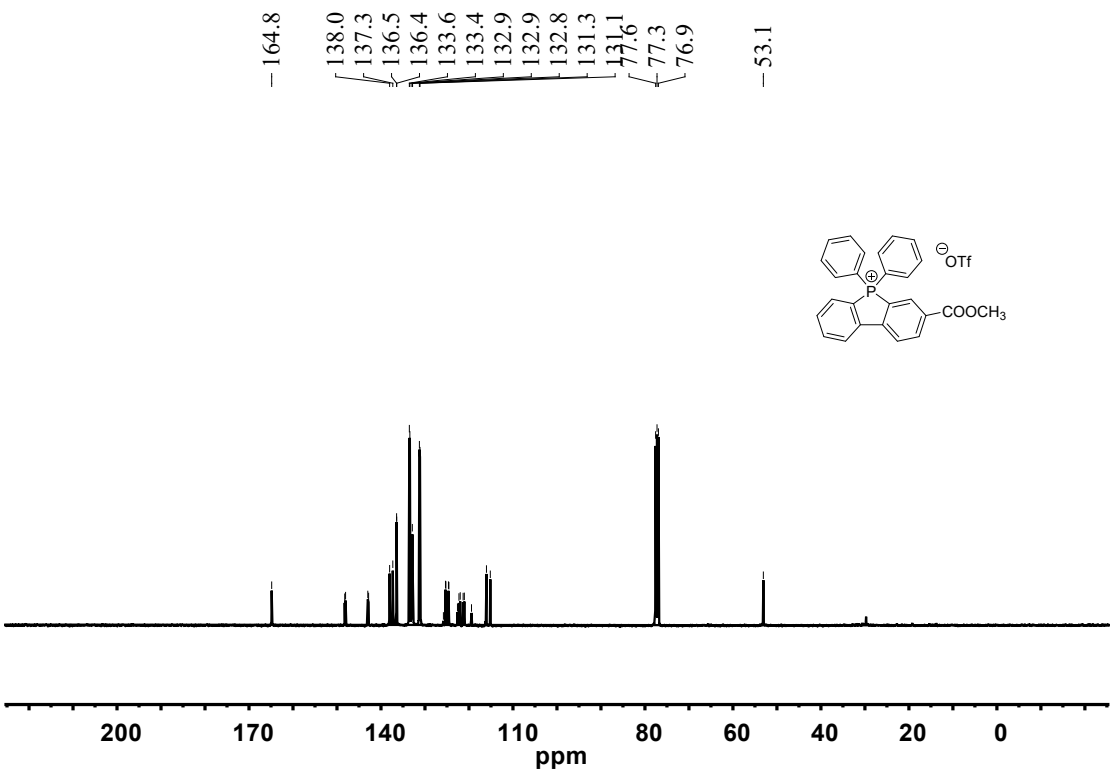
^{19}F NMR spectrum of 2k



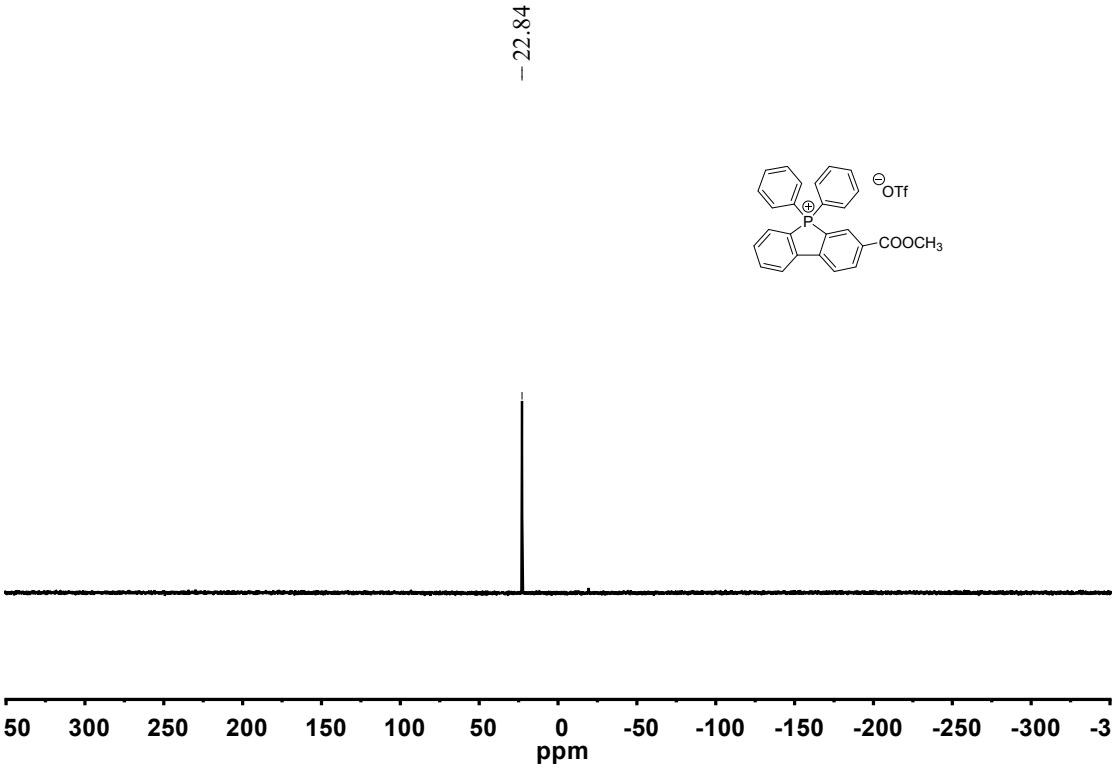
^{1}H NMR spectrum of 2l



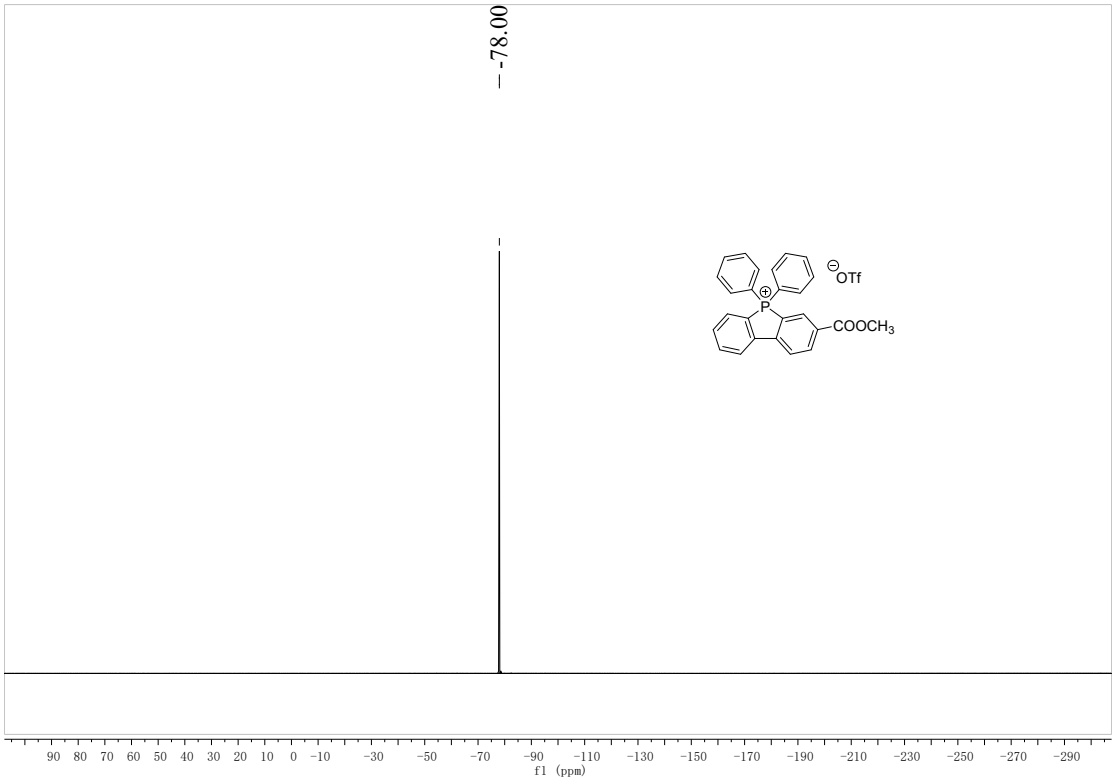
^{13}C NMR spectrum of 2l



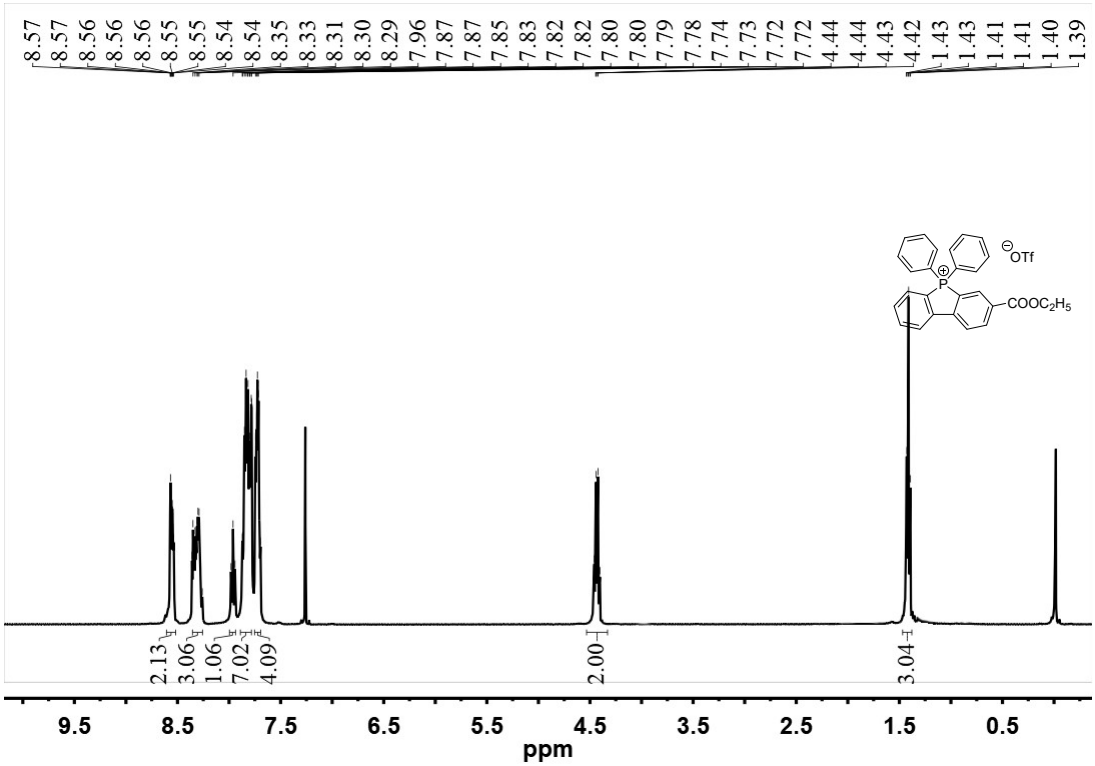
^{31}P NMR spectrum of 2l



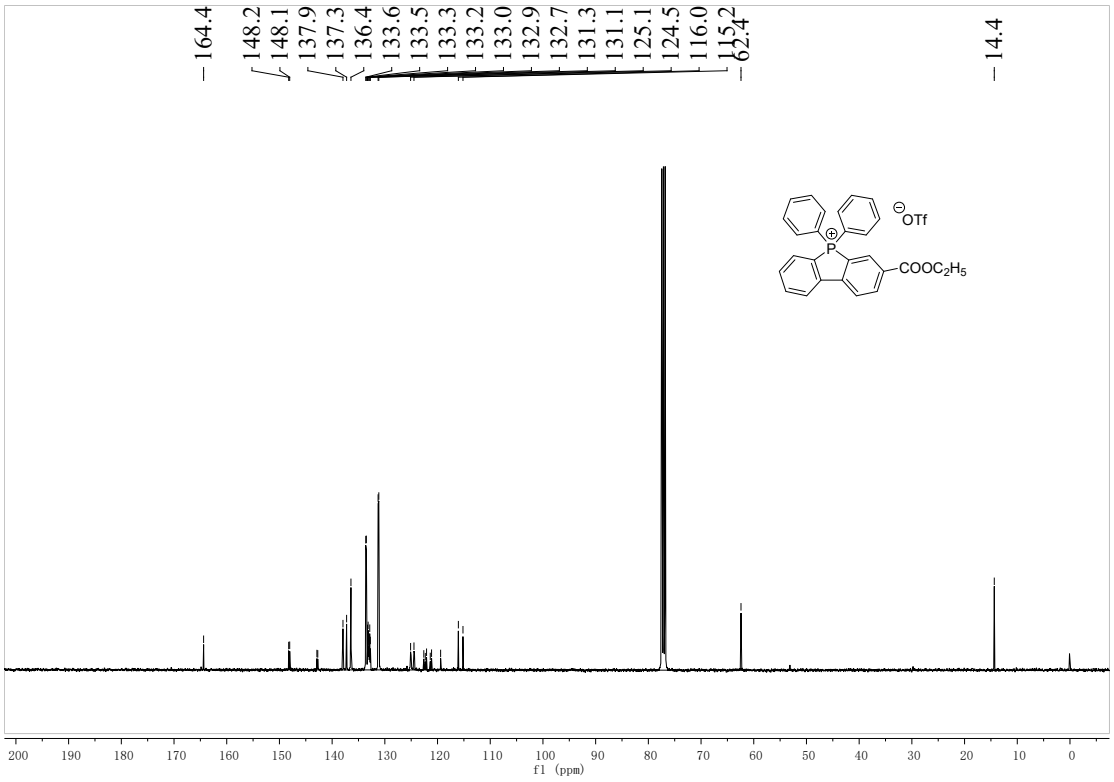
^{19}F NMR spectrum of 2l



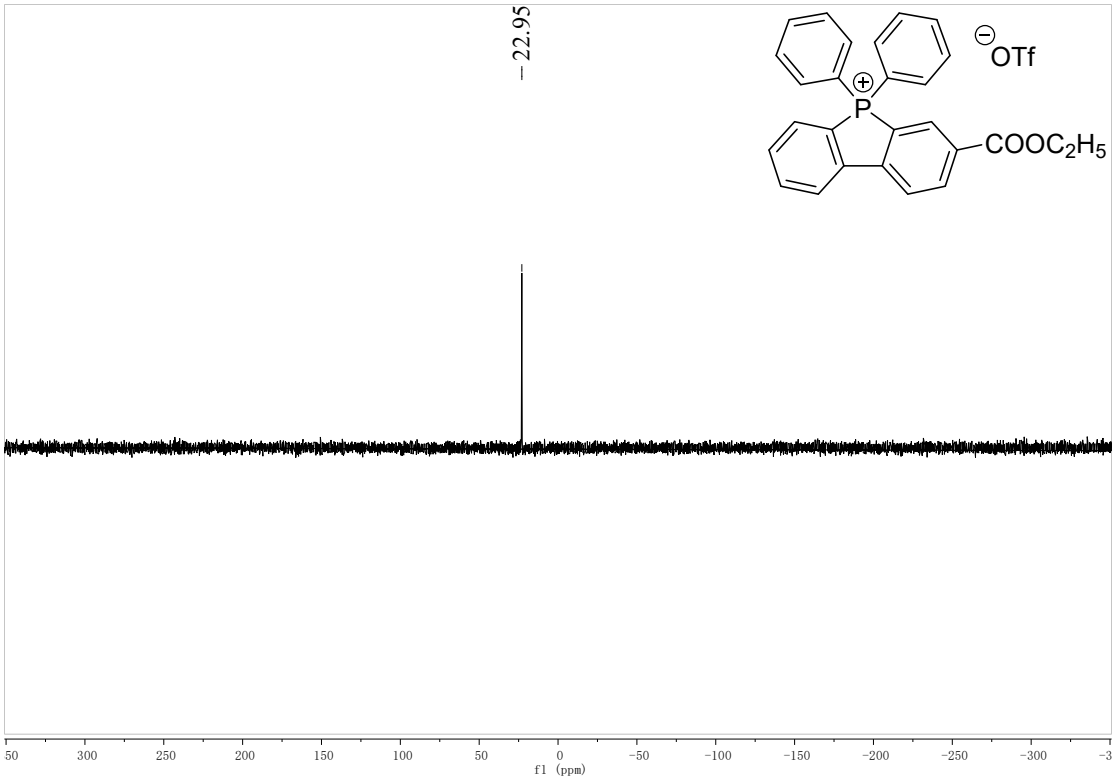
^{1}H NMR spectrum of 2m



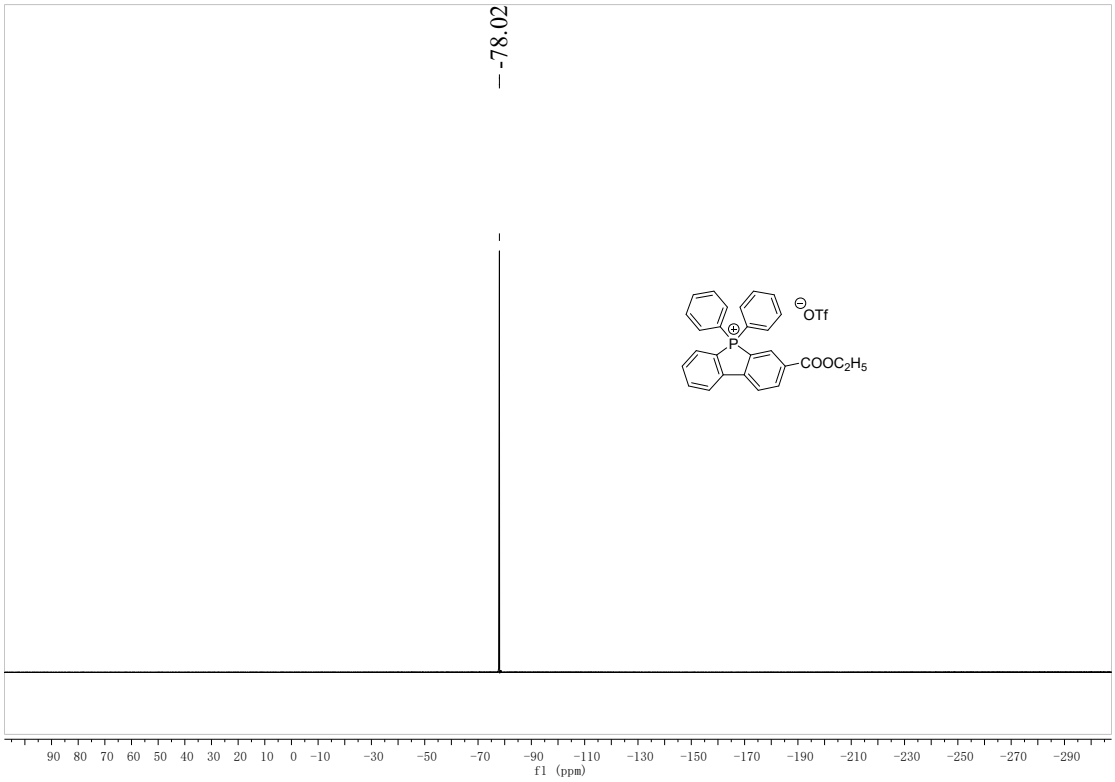
^{13}C NMR spectrum of 2m



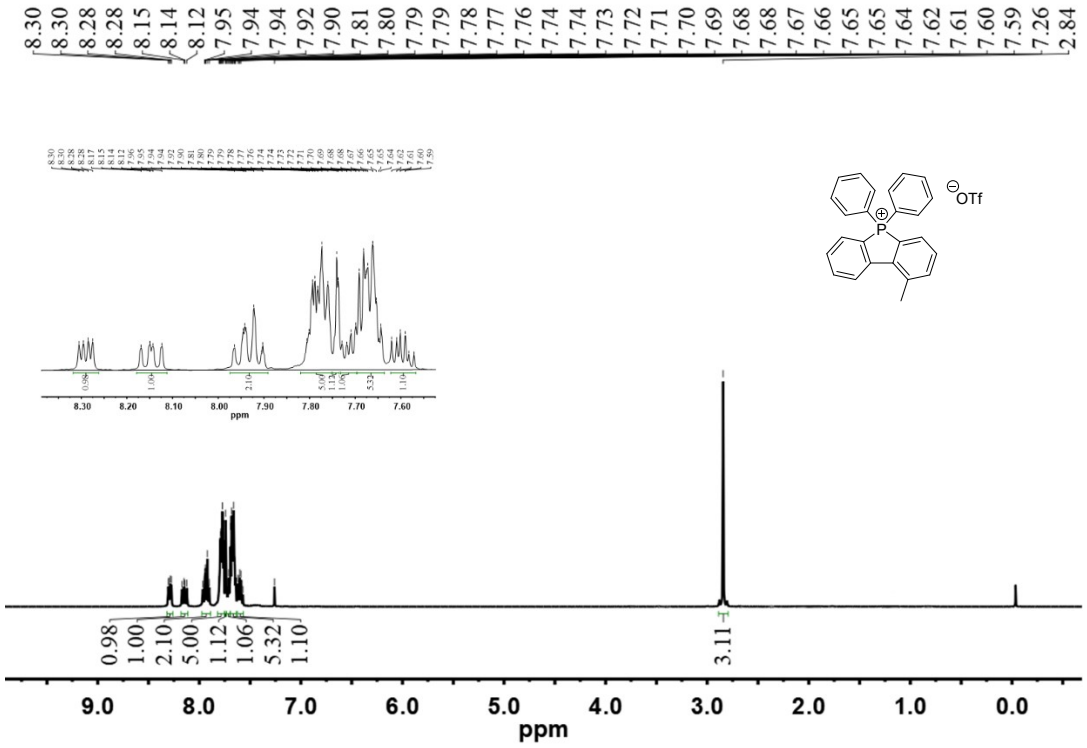
^{31}P NMR spectrum of 2m



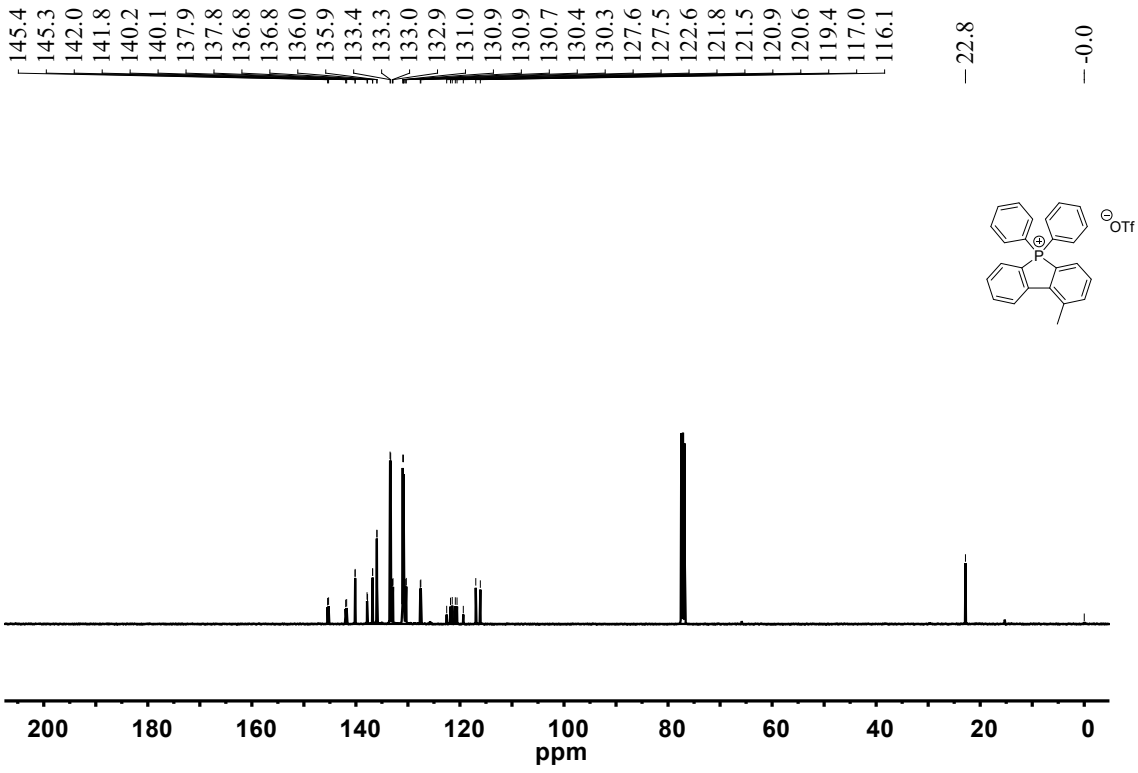
^{19}F NMR spectrum of 2m



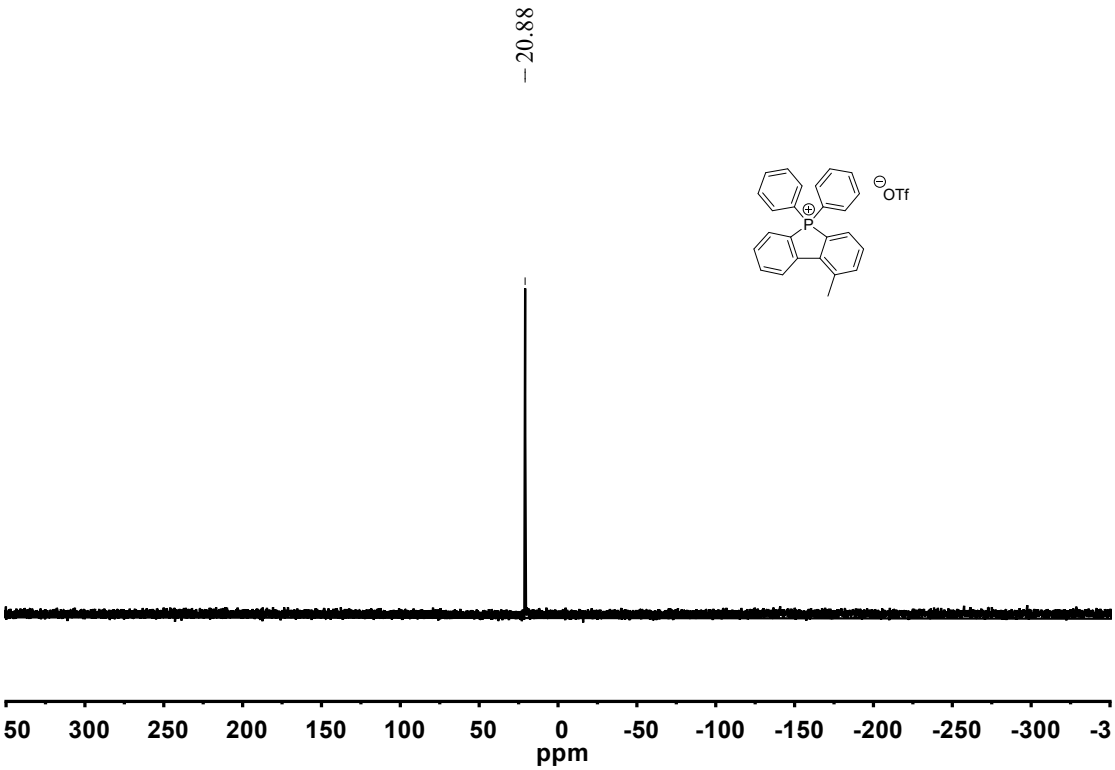
^{1}H NMR spectrum of 2n



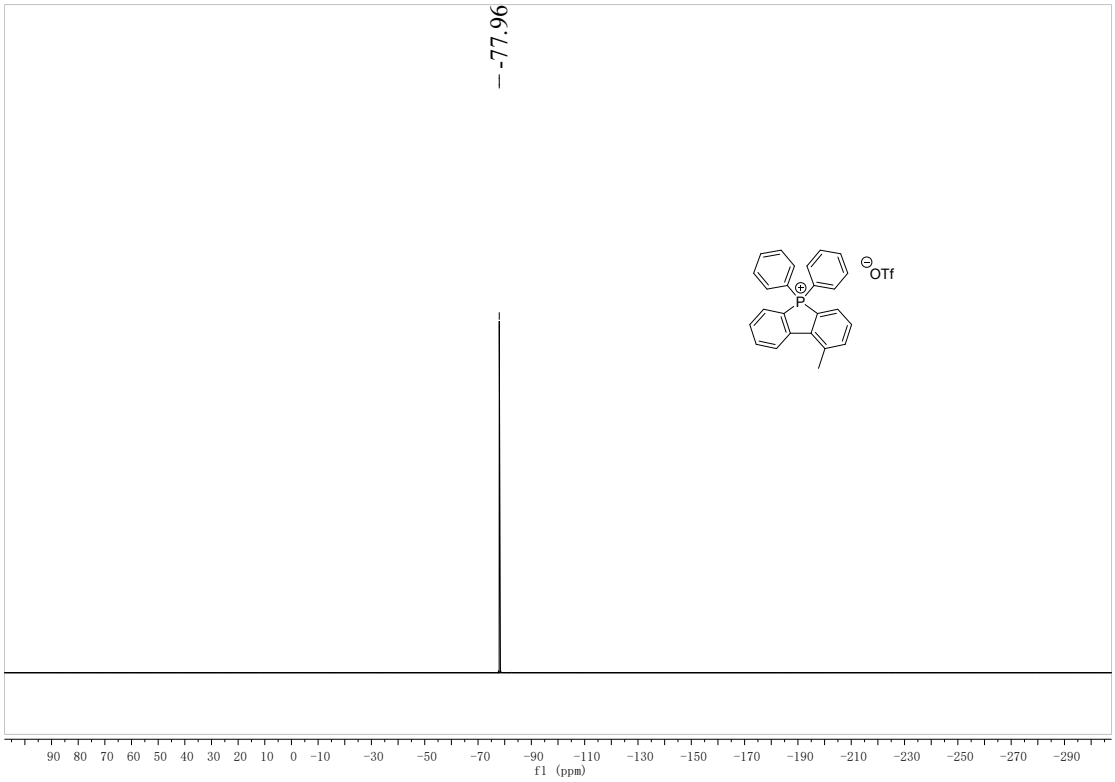
^{13}C NMR spectrum of 2n



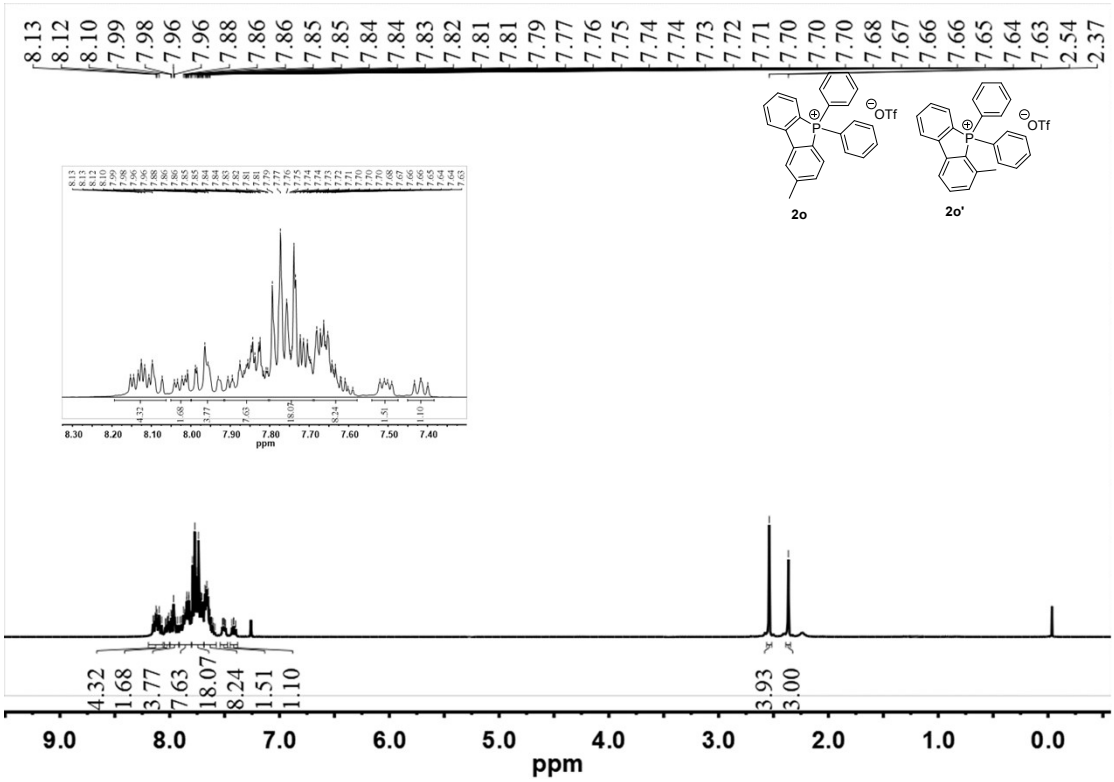
^{31}P NMR spectrum of 2n



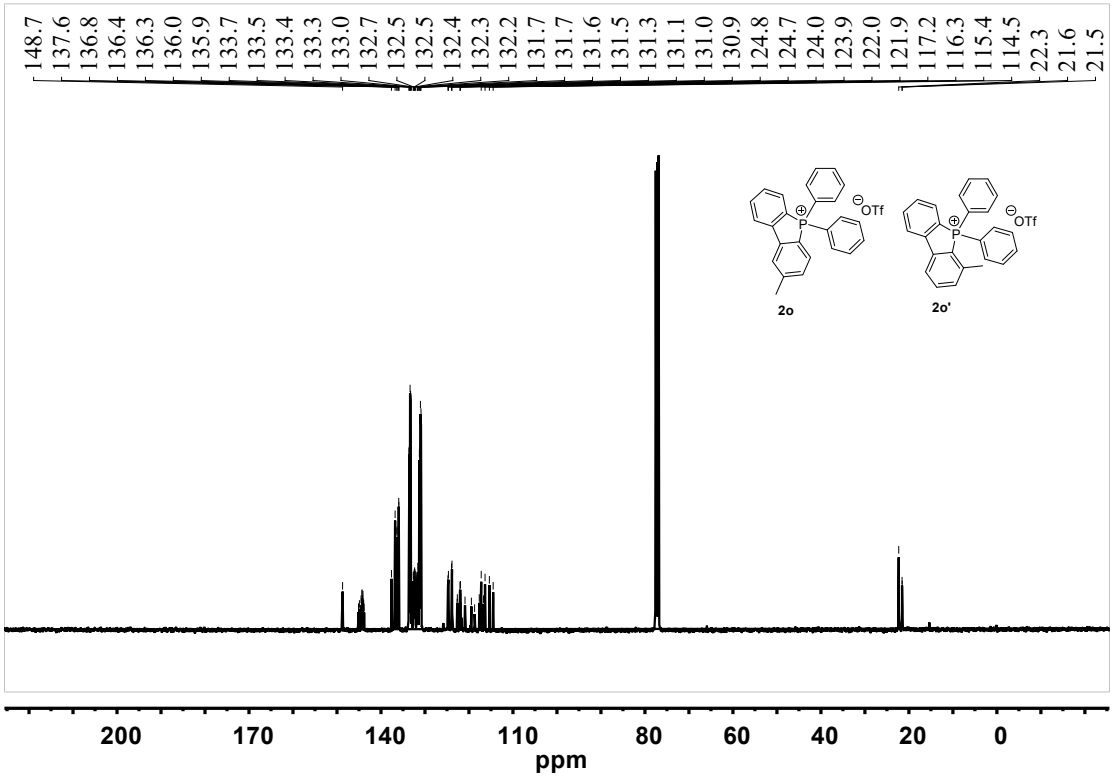
^{19}F NMR spectrum of 2n



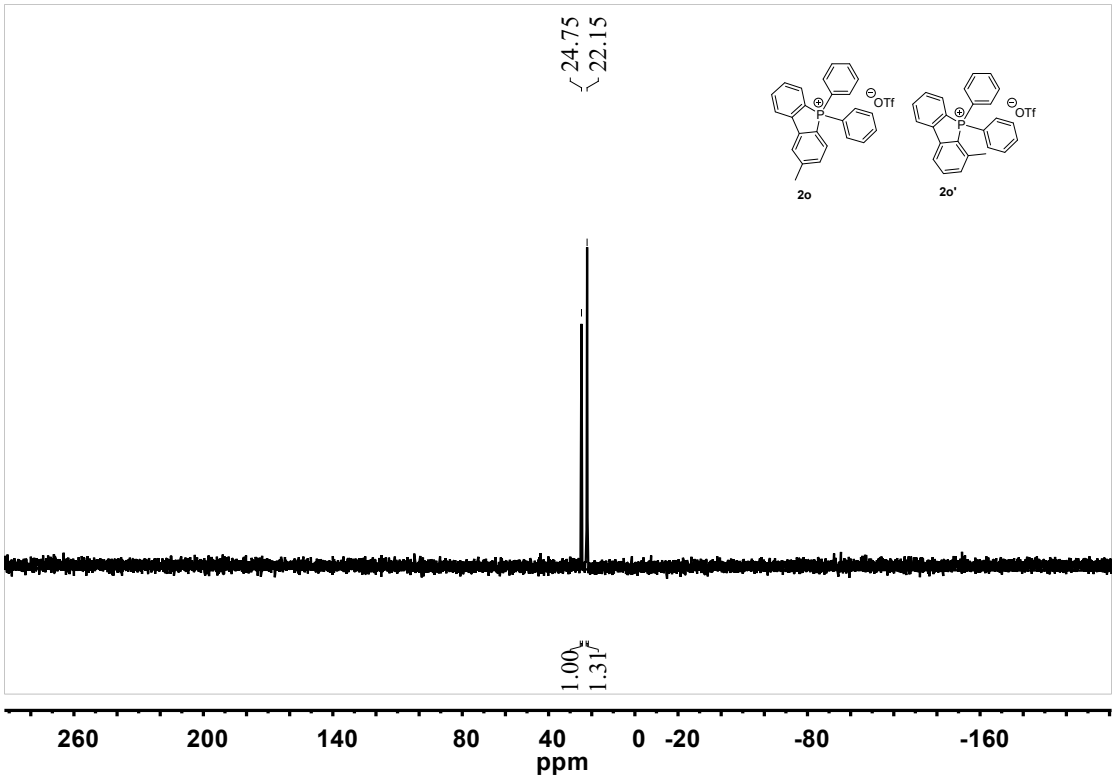
^{1}H NMR spectrum of 2o and 2o’



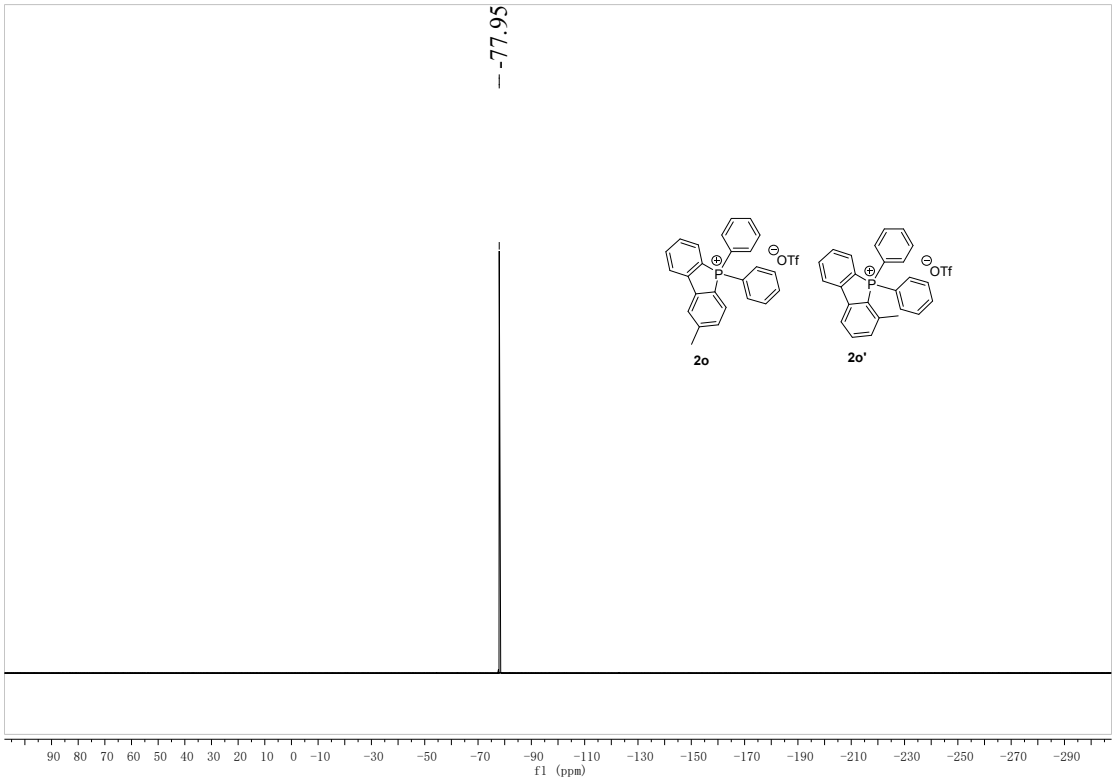
^{13}C NMR spectrum of 2o and 2o’



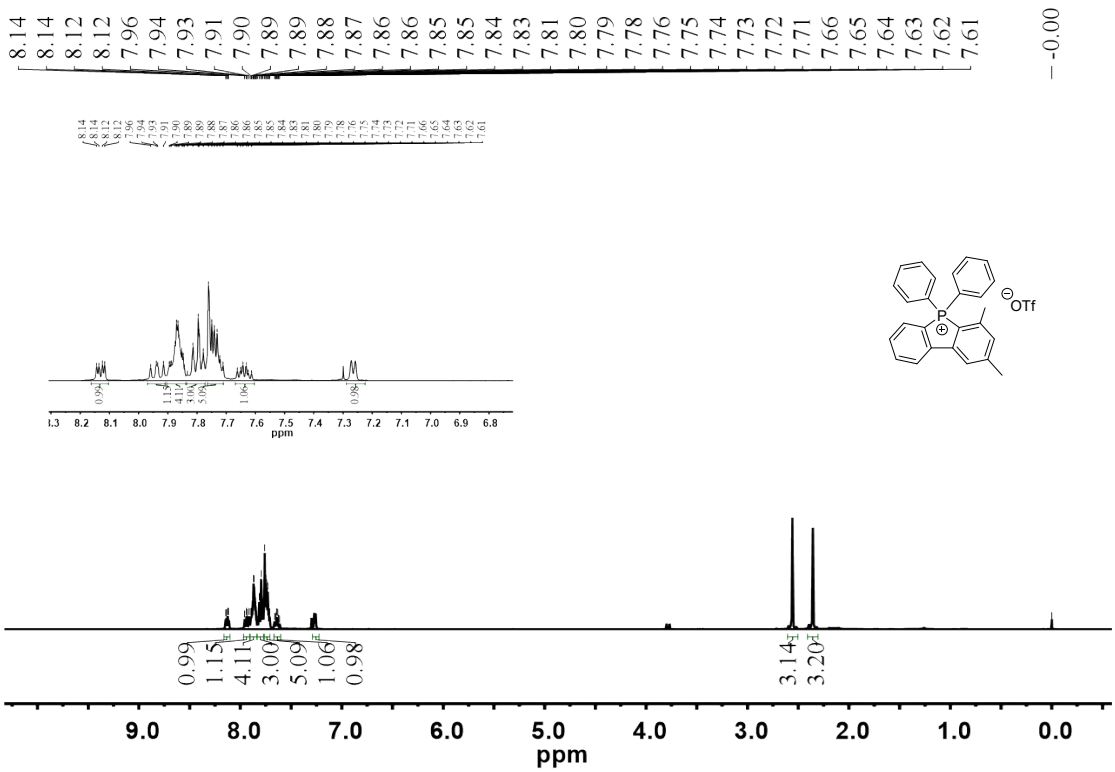
^{31}P NMR spectrum of 2o and 2o’



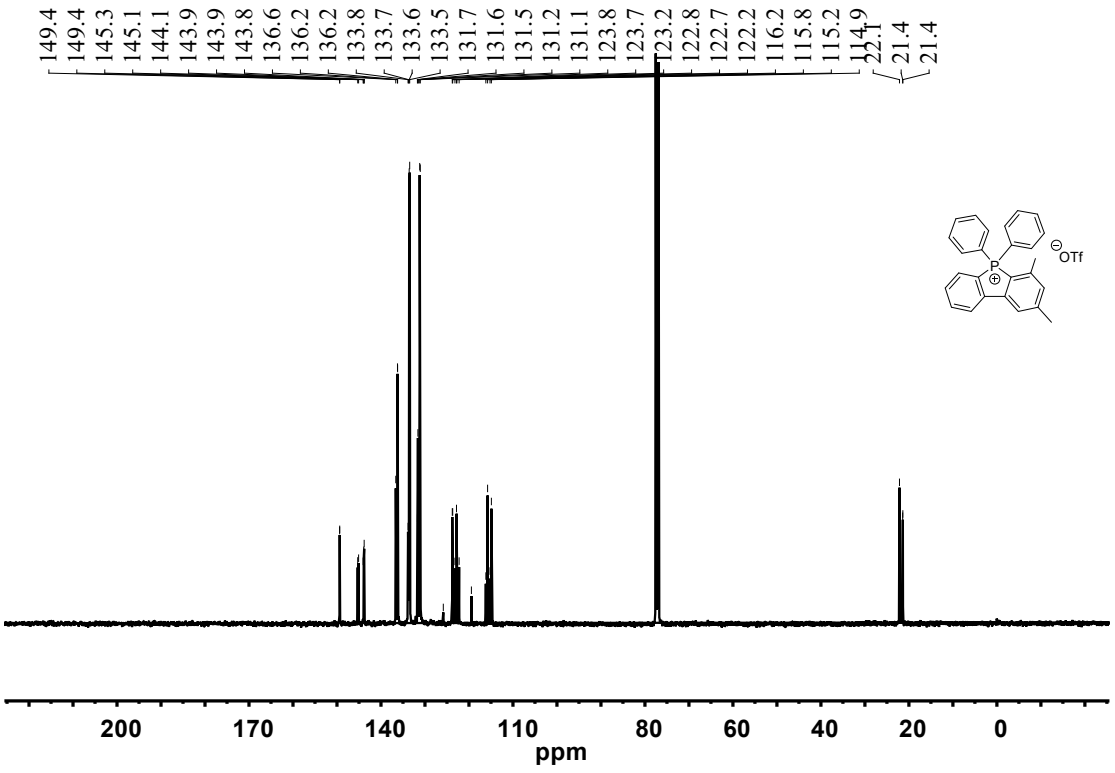
[19] F NMR spectrum of 2o and 2o’



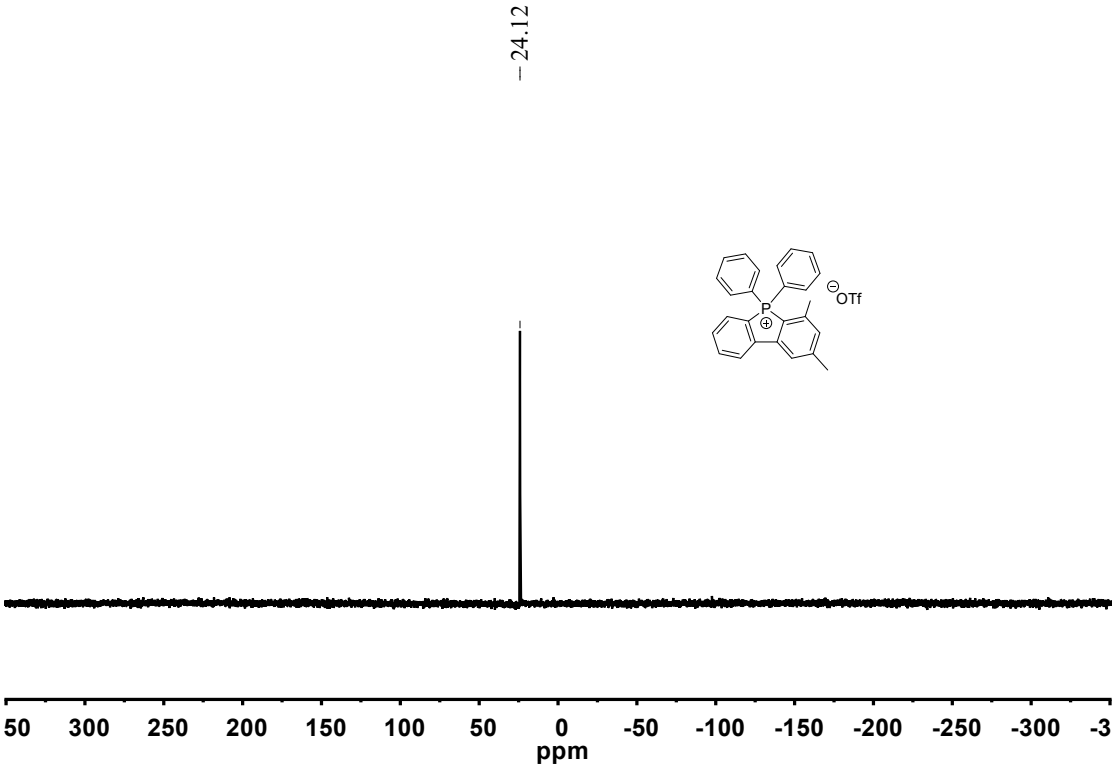
[1]H NMR spectrum of 2p



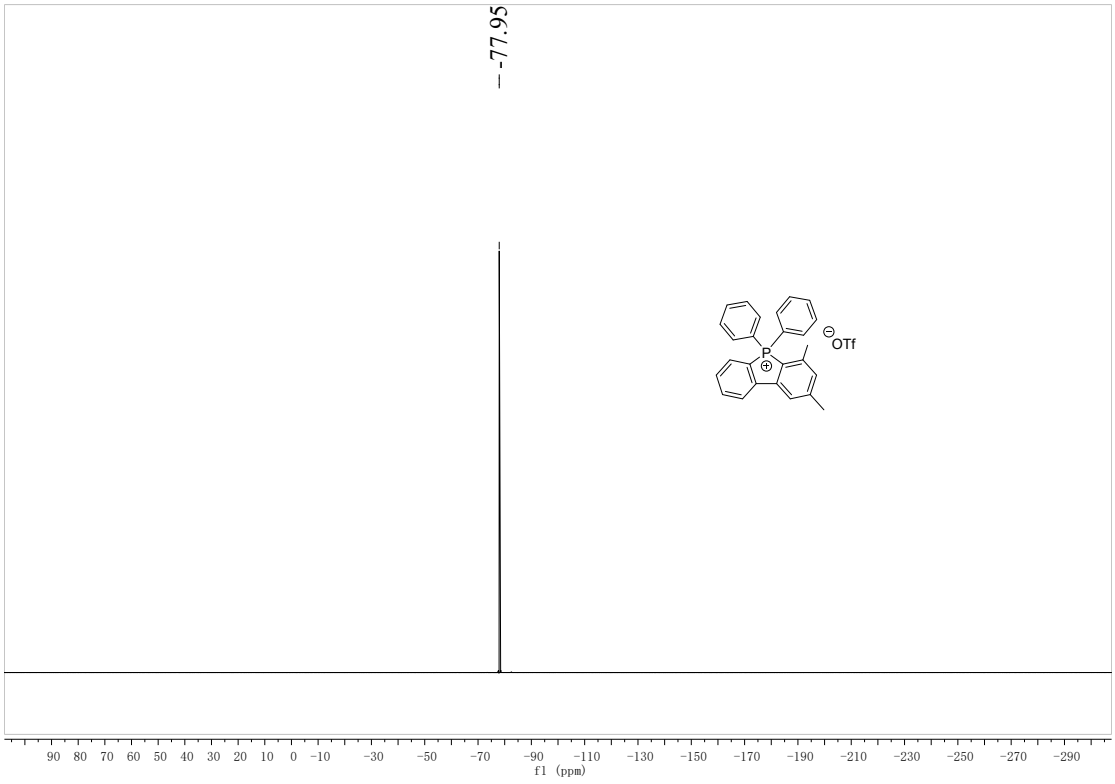
^{13}C NMR spectrum of 2p



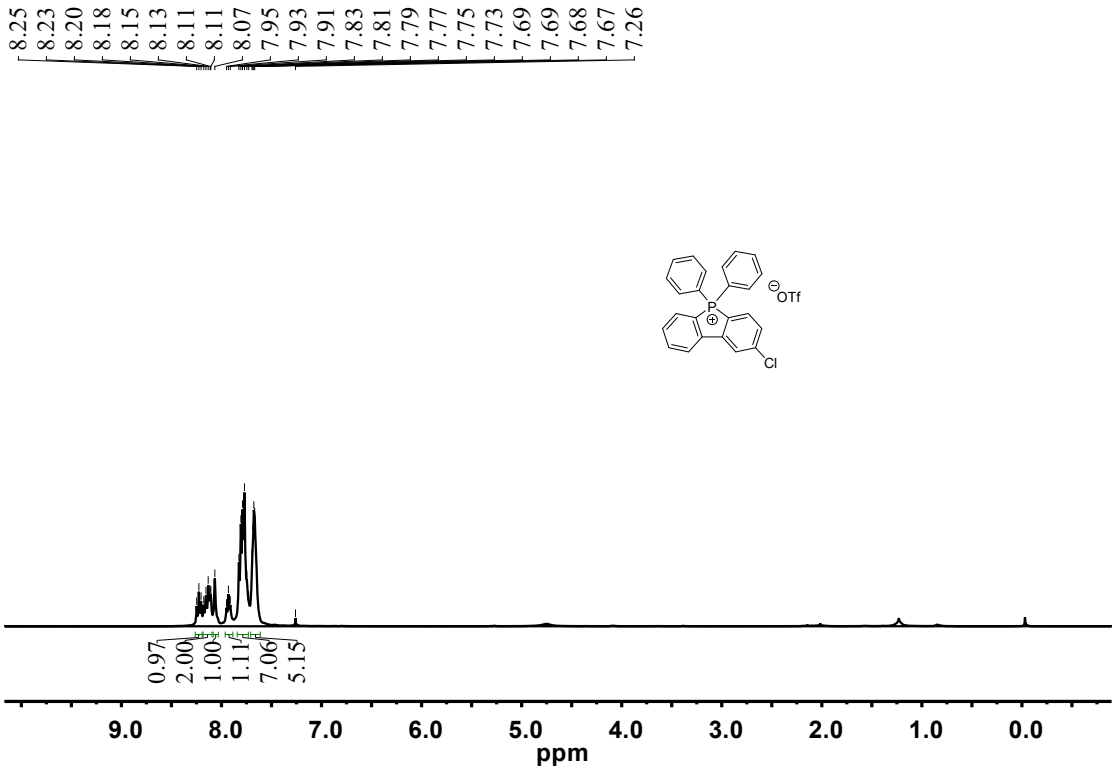
^{31}P NMR spectrum of 2p



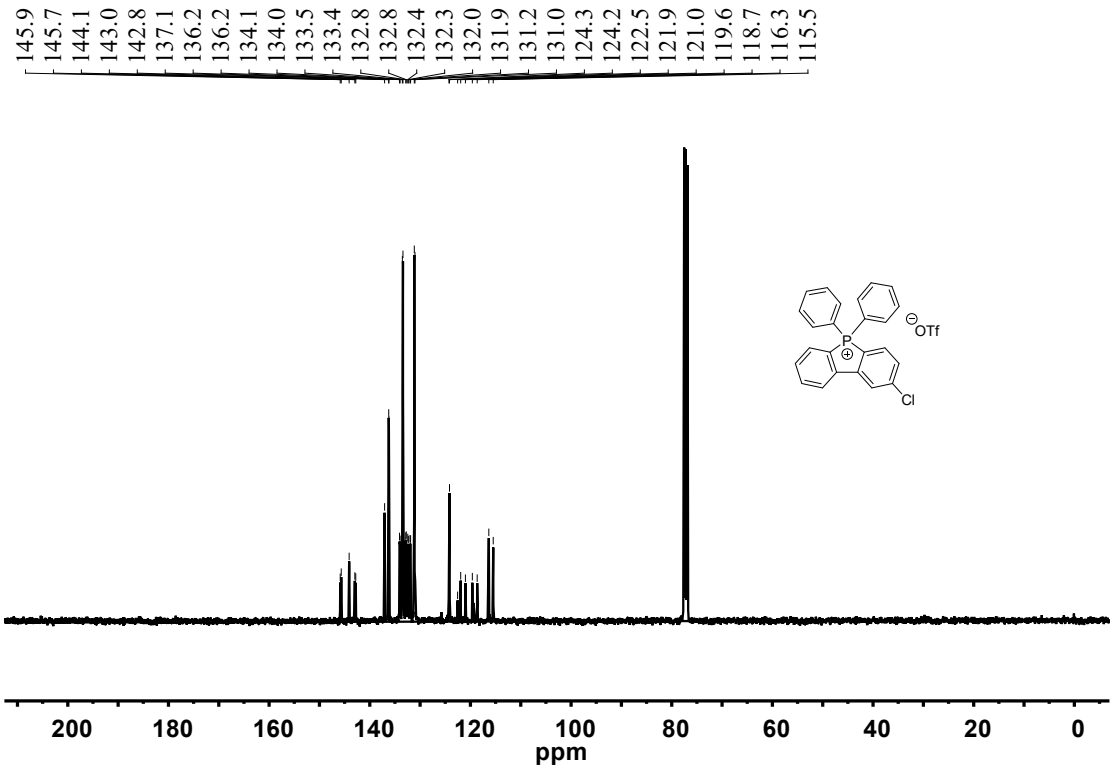
^{19}F NMR spectrum of 2p



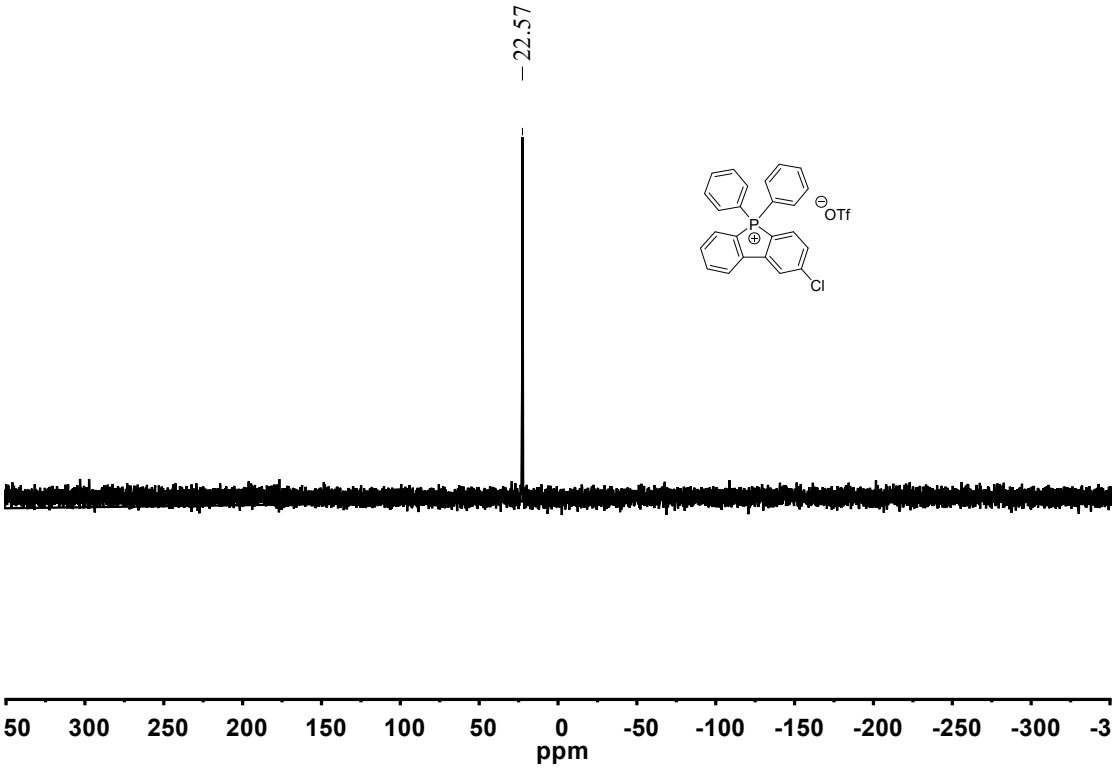
^{1}H NMR spectrum of 2q



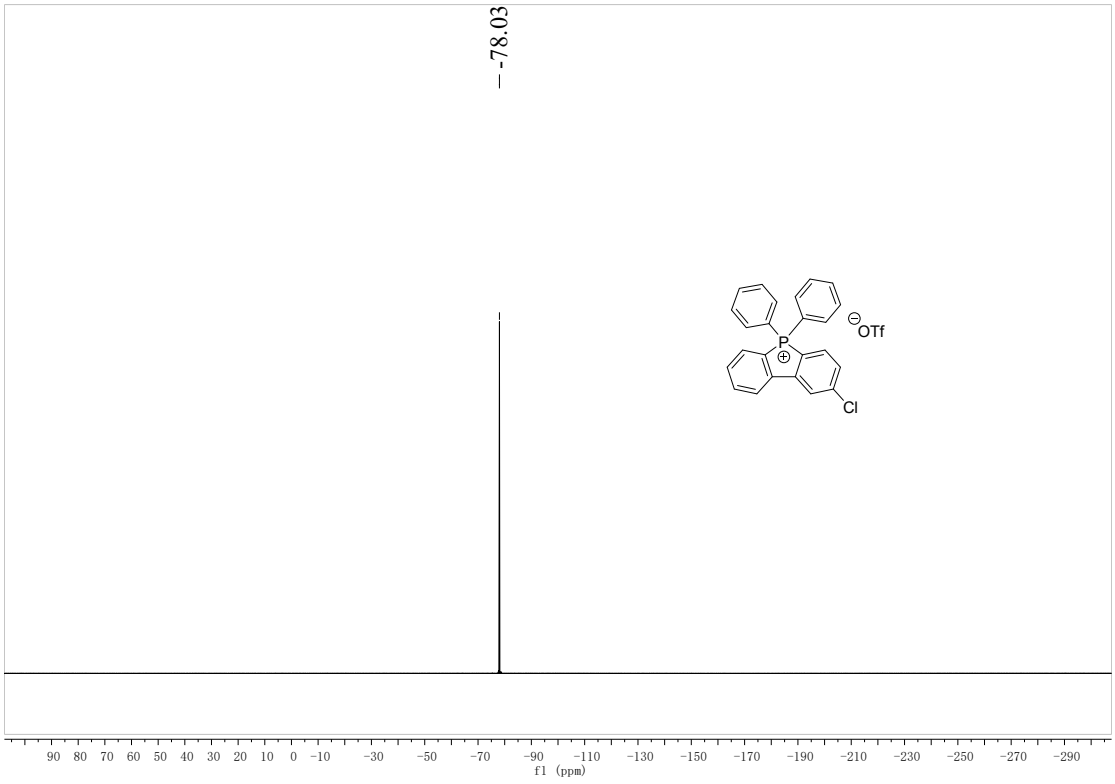
^{13}C NMR spectrum of 2q



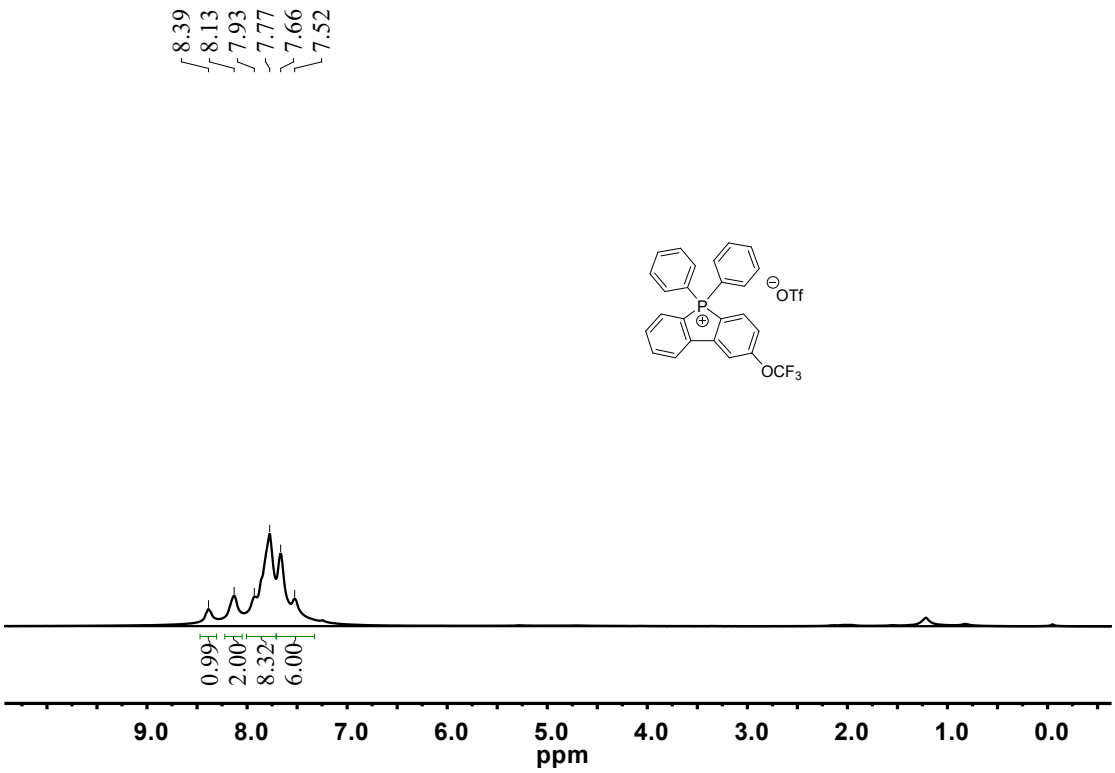
^{31}P NMR spectrum of 2q



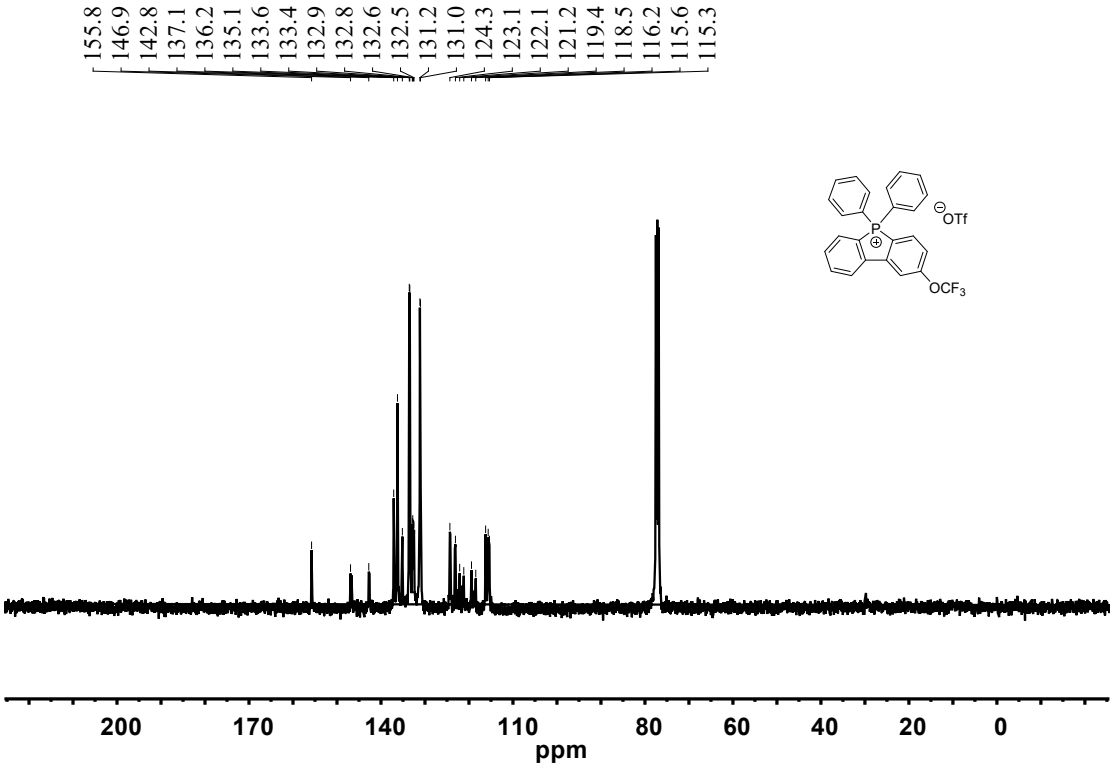
^{19}F NMR spectrum of 2q



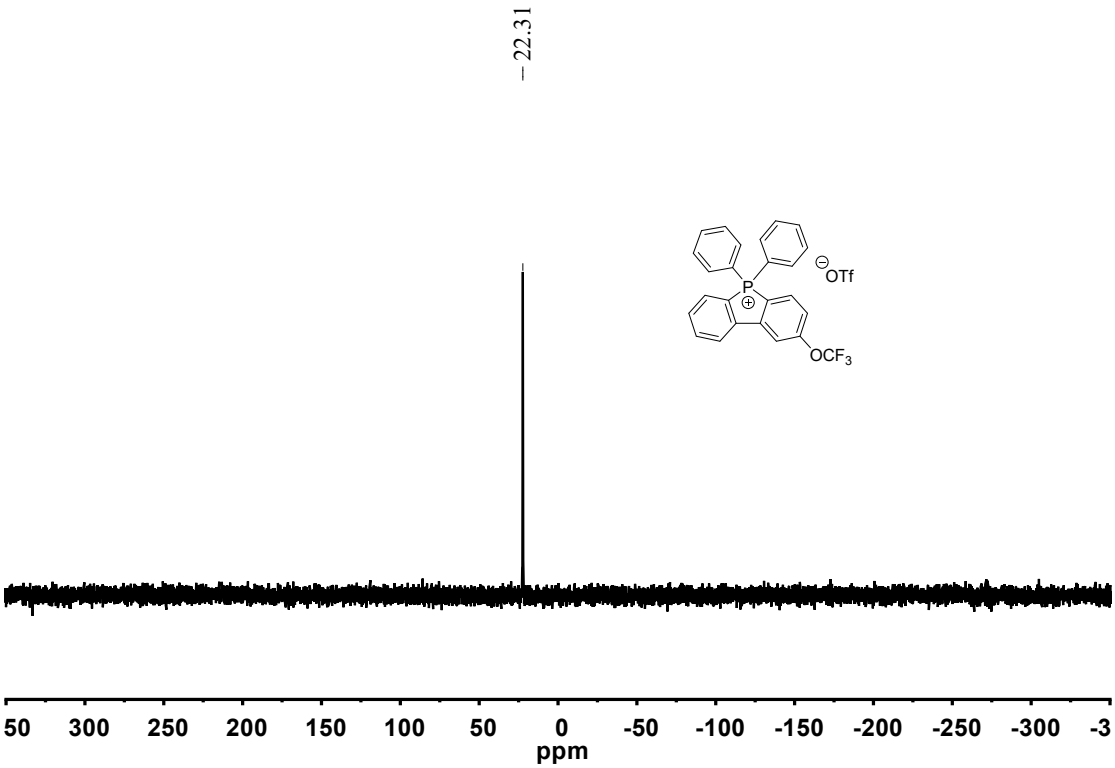
^{1}H NMR spectrum of 2r



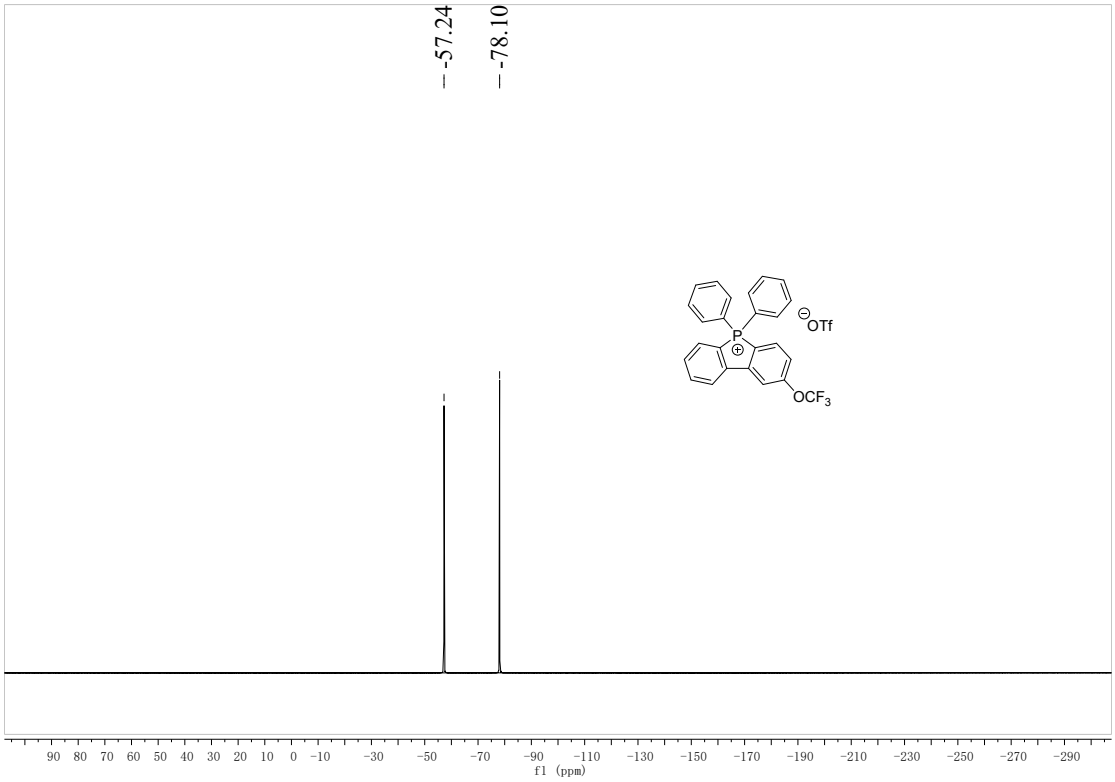
^{13}C NMR spectrum of 2r



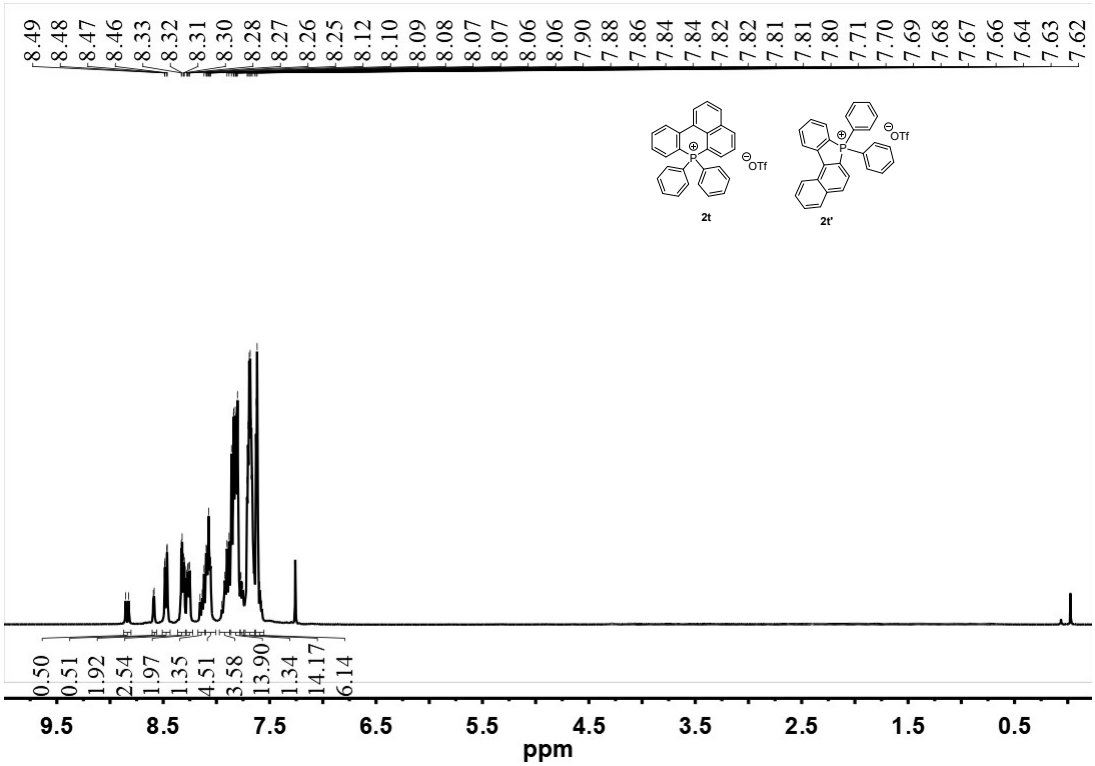
^{31}P NMR spectrum of 2r



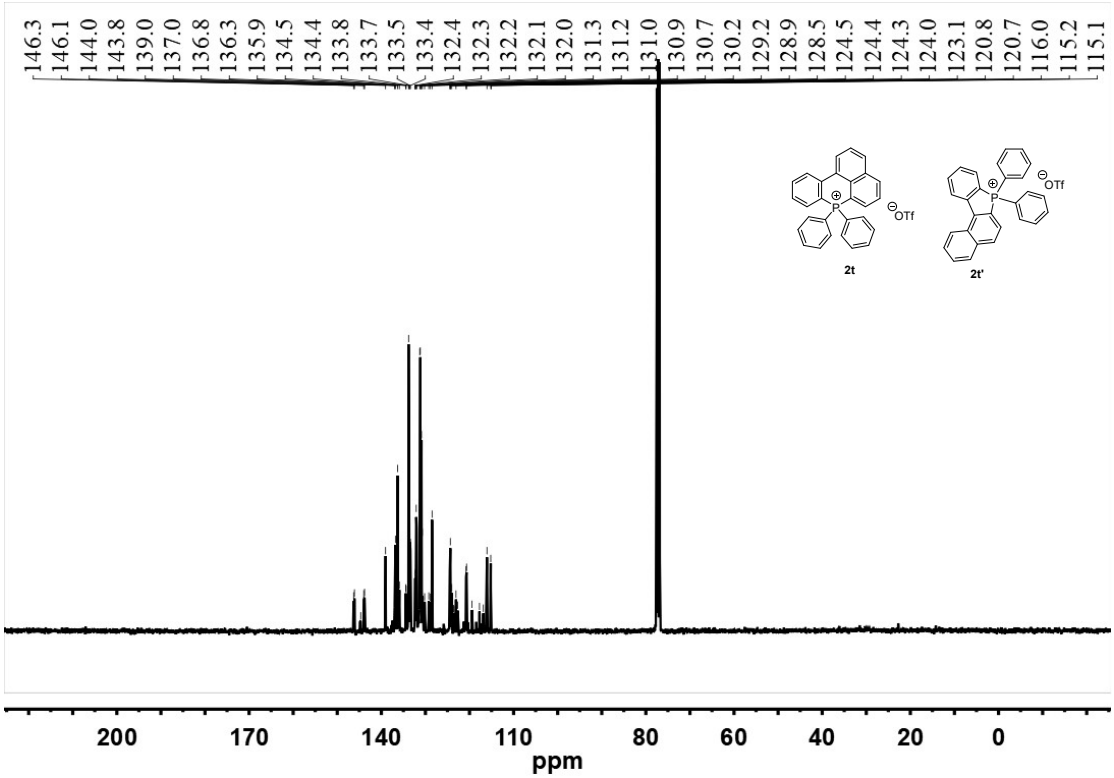
^{19}F NMR spectrum of 2r



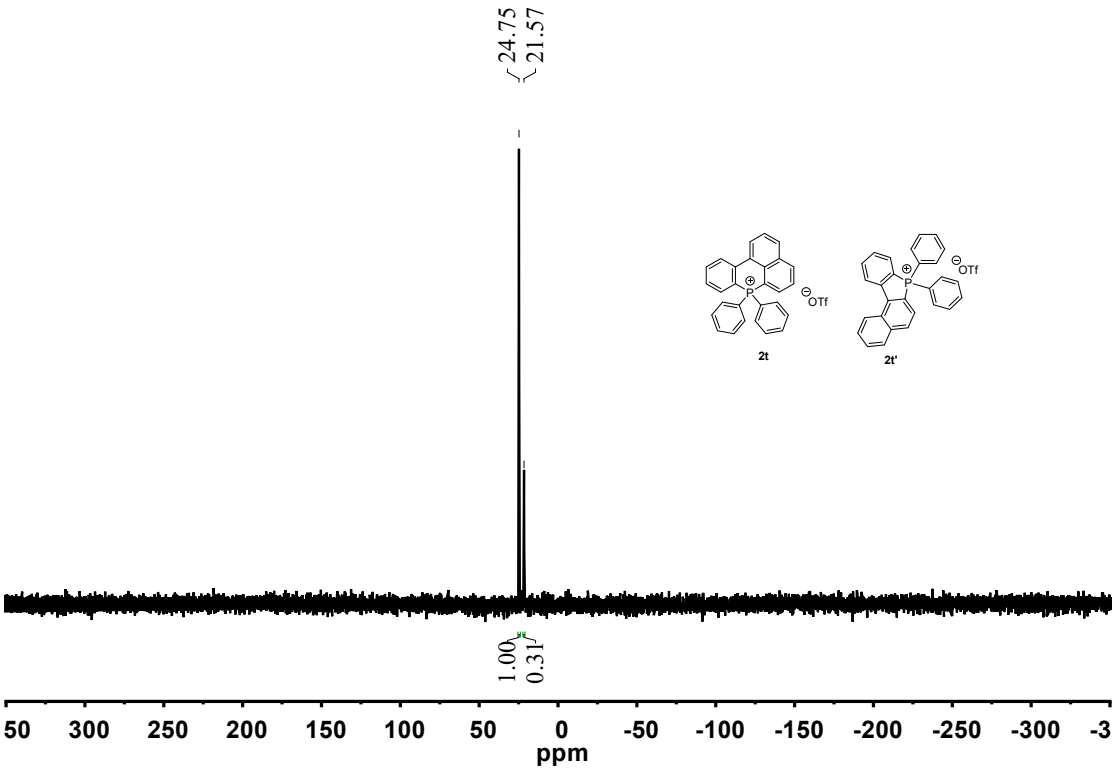
^{1}H NMR spectrum of 2t and 2t’



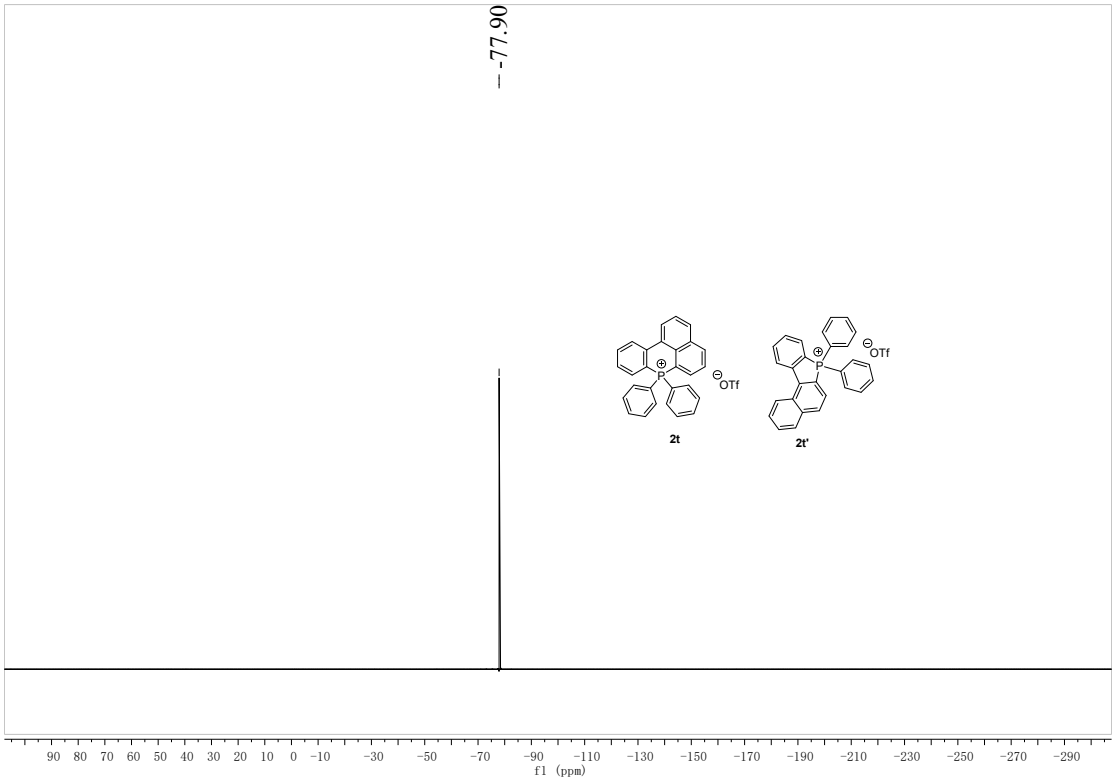
^{13}C NMR spectrum of 2t and 2t'



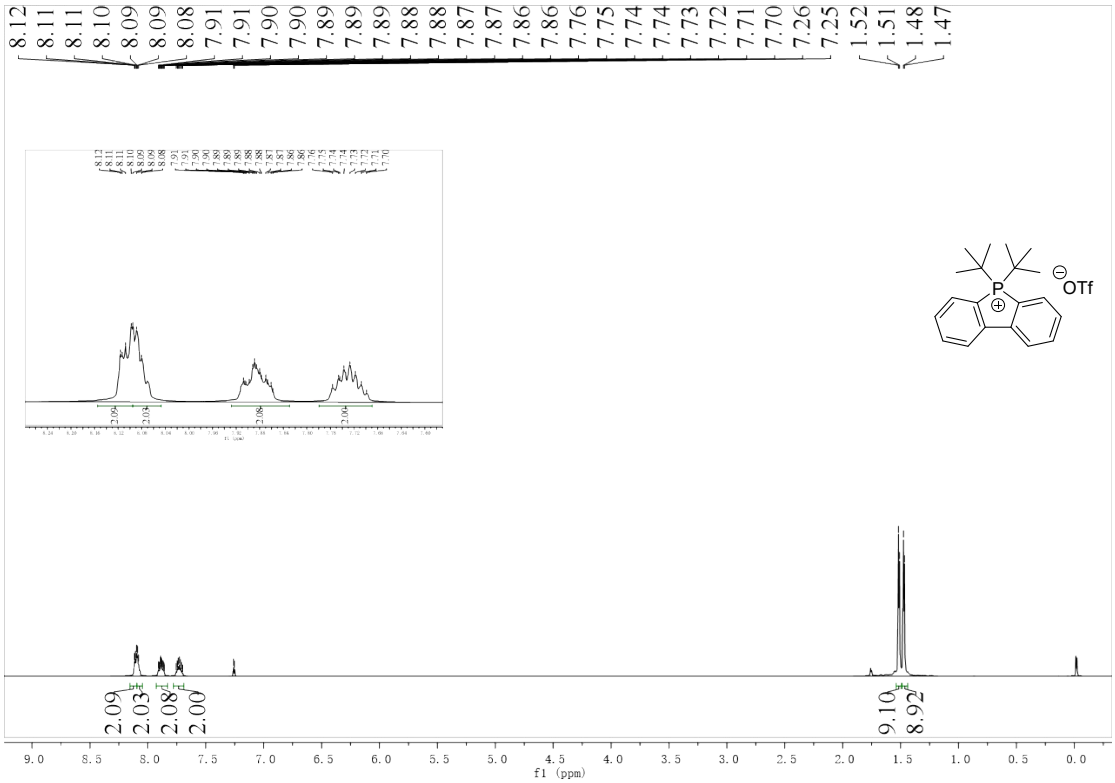
^{31}P NMR spectrum of 2t and 2t'



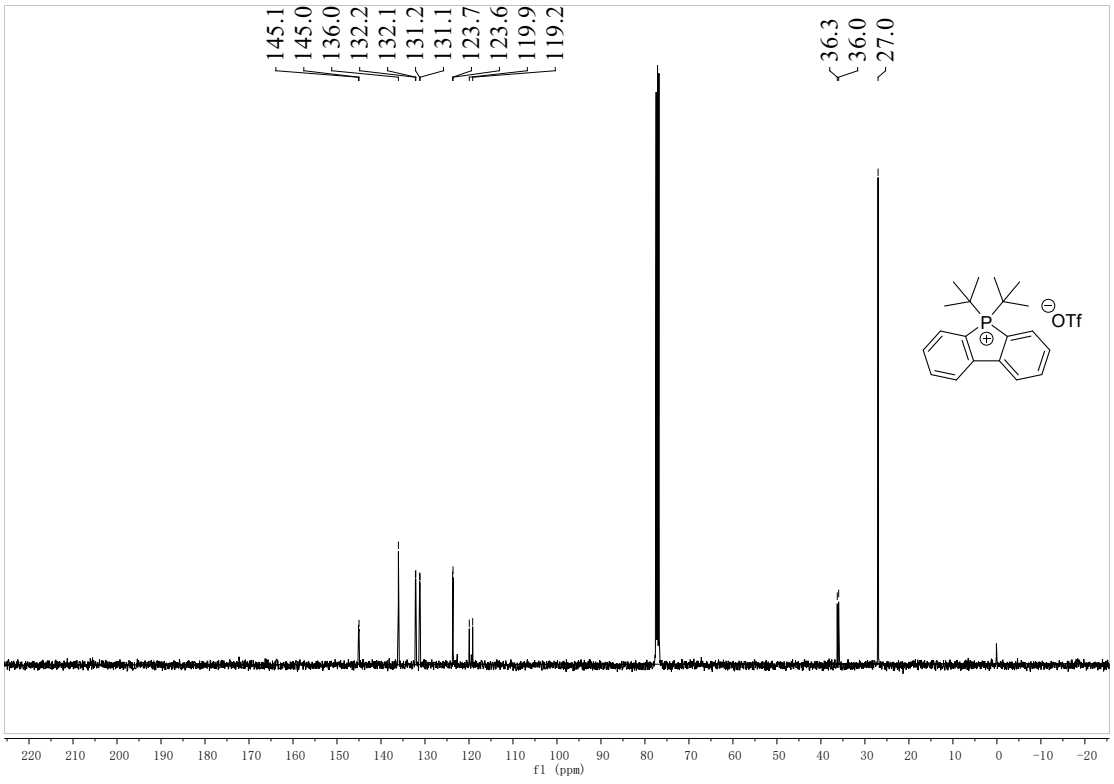
^{19}F NMR spectrum of 2t and 2t'



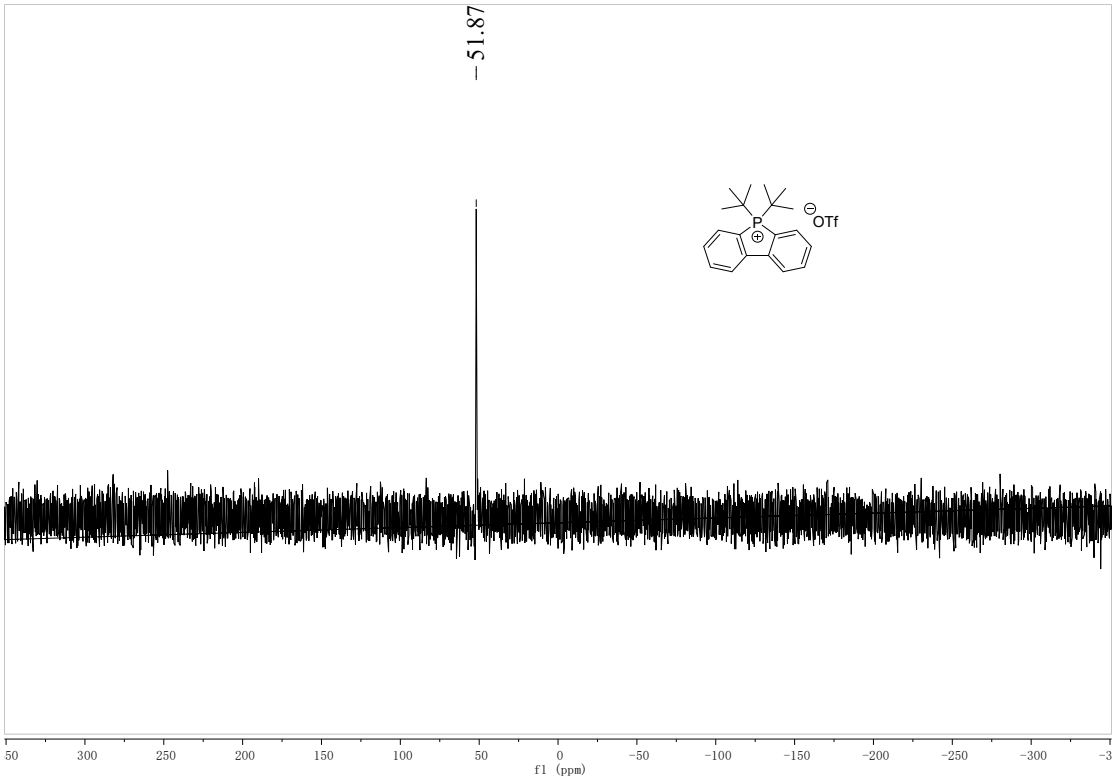
^{1}H NMR spectrum of 2u



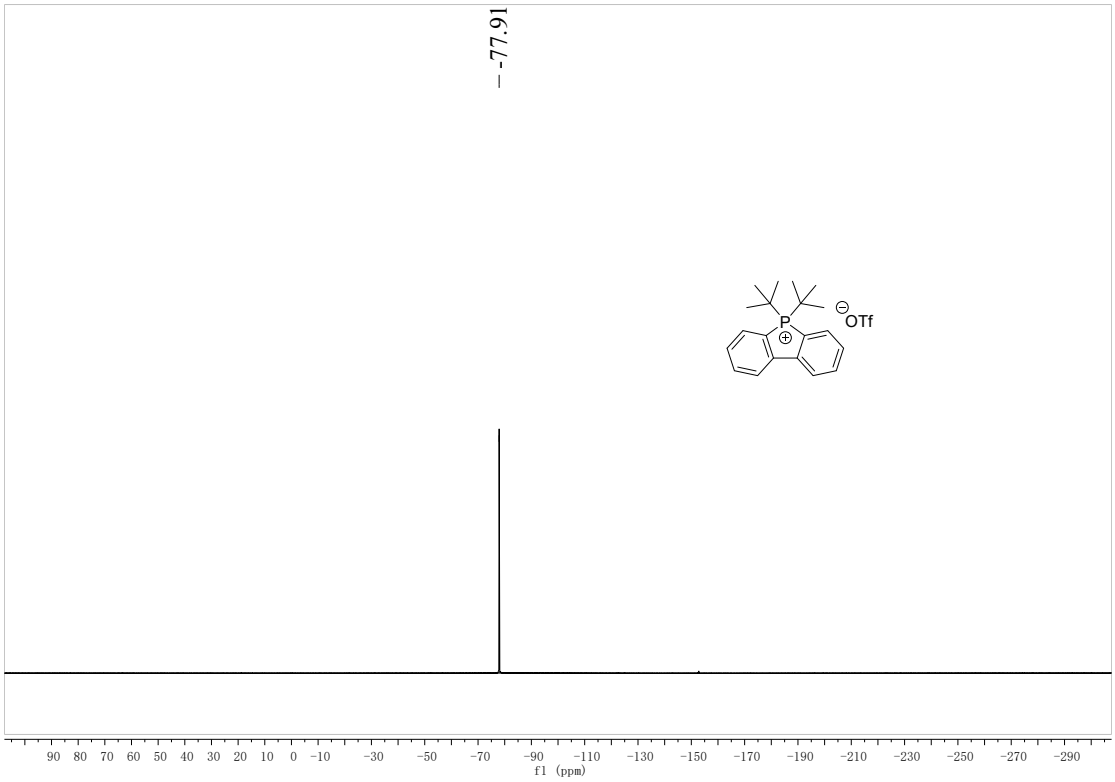
^{13}C NMR spectrum of 2u



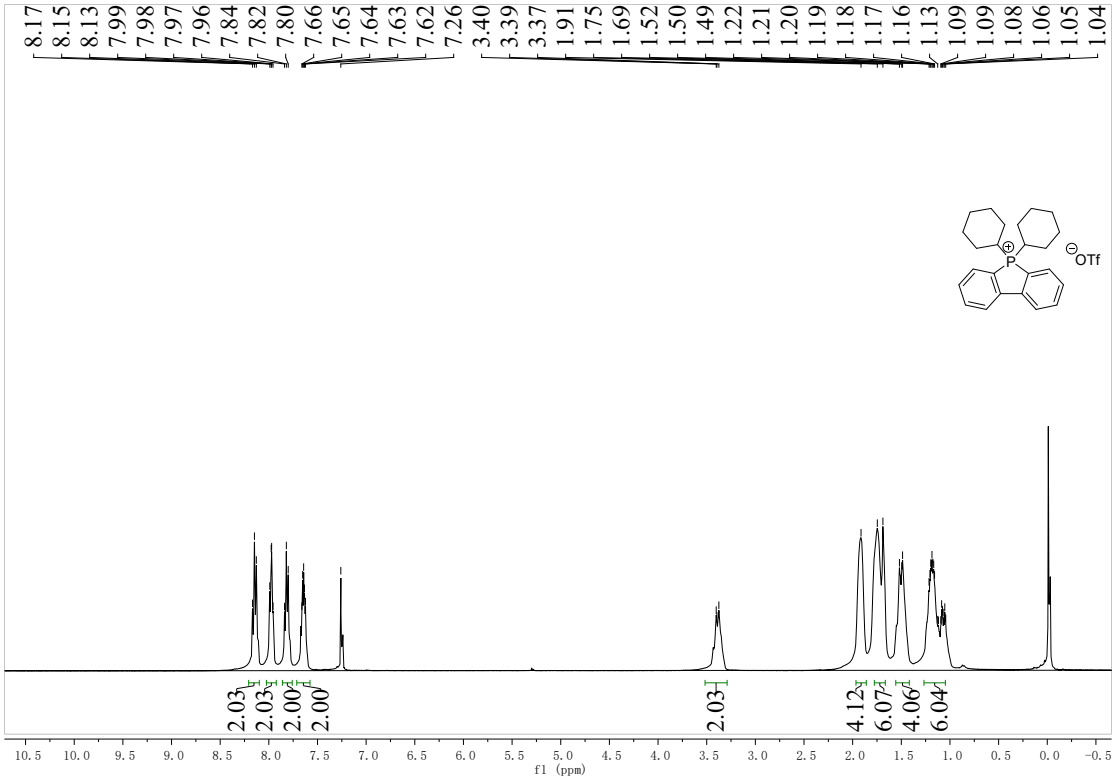
^{31}P NMR spectrum of 2u



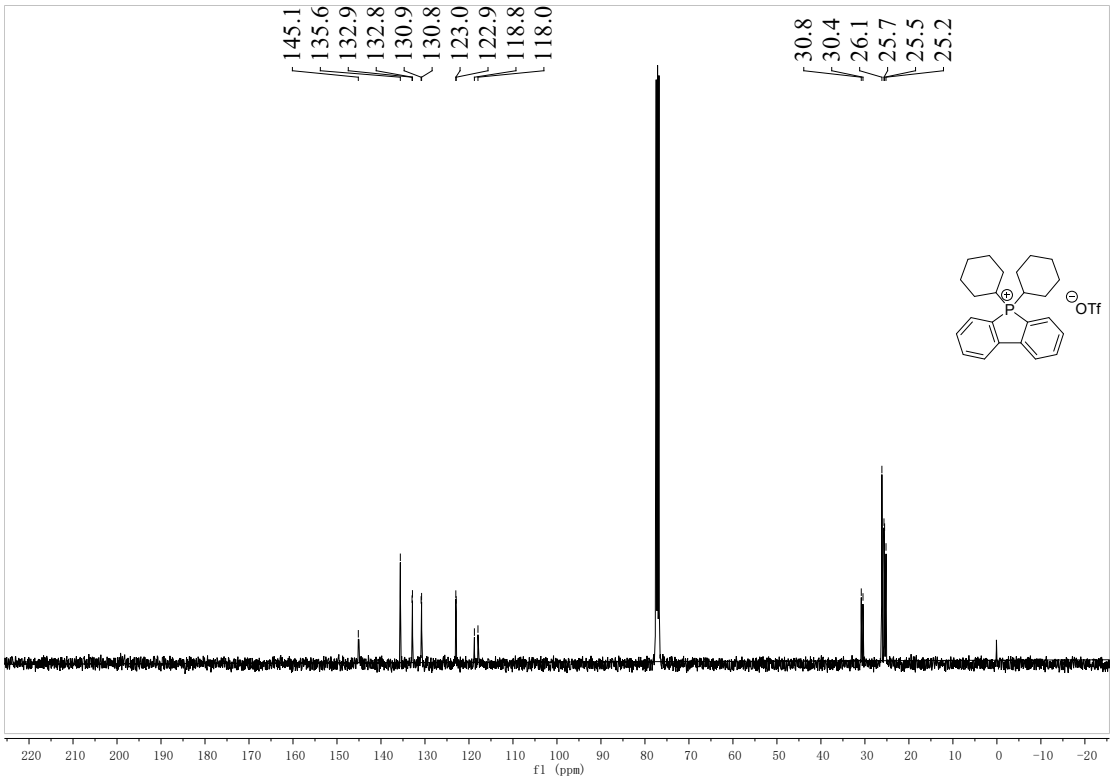
^{19}F NMR spectrum of 2u



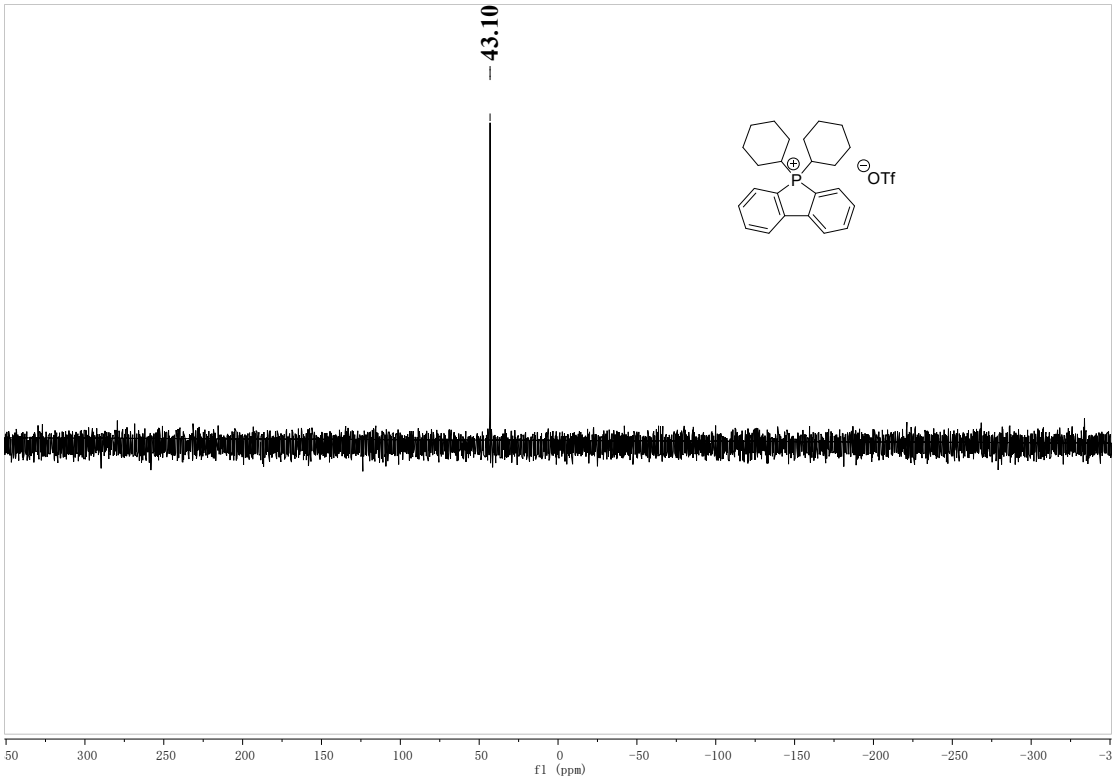
^{1}H NMR spectrum of 2v



^{13}C NMR spectrum of 2v



^{31}P NMR spectrum of 2v



^{19}F NMR spectrum of 2v