Supporting Information

Electrochemically Driven Hydrophosphination of Alkenes and Alkynes with diphenylphosphine

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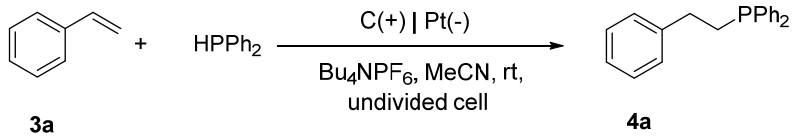
1. General information

All air-sensitive compounds were handled using standard Schlenk-line or glove box techniques under high-purity nitrogen. ^{1}H and $^{31}P\{^{1}H\}$ NMR spectra were recorded at 25 °C on Bruker Avance III spectrometers (600 or 400 MHz) in deuterated solvents, with chemical shifts calibrated using $CDCl_3$ as an internal standard. The abbreviations used to denote the multiplicities are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Electrolysis experiments were conducted using a CHI 760E potentiostat (Shanghai Chenhua), and cyclic voltammograms were obtained on the same instrument. All reagents and solvents were used as received without further purification unless otherwise stated.

2. Screening of partial reaction conditions

Table S1 Optimization of $HPPh_2$ in electrochemical hydrophosphination of alkene.

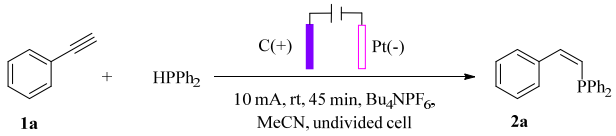


3a **4a**

Entry	$HPPh_2$	Time	Conversion[a]
1	1.0	8 h	45%
2	1.2	8 h	65%
3	1.5	8 h	63%
4	1.2	5 h	60%
5	1.2	6 h	61%
6	*1.2*	*7 h*	*84%(72%[b])*
7	1.2	7 h	37%[c]
8	1.2	7 h	44%[d]
9	1.5	7 h	26%[e]
10	1.5	7 h	64%[f]
11	1.5	7 h	28%[g]
12	1.5	7 h	trace[h]

[a] Conversion was determined by ^{1}H NMR spectroscopy. [b] isolated yield. [c] I = 5 mA, [d] I = 15 mA, [e] Et_4NBF_4 as the supporting electrolyte. [f] $^{n}Bu_4NBF_4$ as the supporting electrolyte. [g] CH_3OH as the solvent. [h] THF as the solvent.

Table S2 Optimization of the reaction conditions.

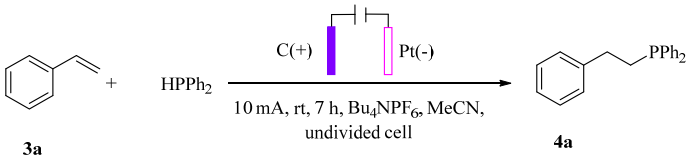


1a 2a

Entry	Variation from the reaction conditions	Conversion[a]
1	None	91%(80%[b])
2	No electric current	0%
3	C(+)|C(-) instead of C(+)|Pt(-)	68%
4	Pt(+)|Pt(-) instead of C(+)|Pt(-)	76%
5	SST(+)|SST(-) instead of C(+)|Pt(-)	36%
6	Air instead of N_2	0%

[a] Reaction conditions: **1a** (0.5 mmol), $HPPh_2$ (0.75 mmol), $^nBu_4NPF_6$ (0.5 mmol), I = 10 mA, MeCN (3.0 mL), N_2, rt, 45 min, isolated yield. Conversion was determined by 1 H NMR spectroscopy. [b] isolated yield.

Table S3 Optimization of the reaction conditions.

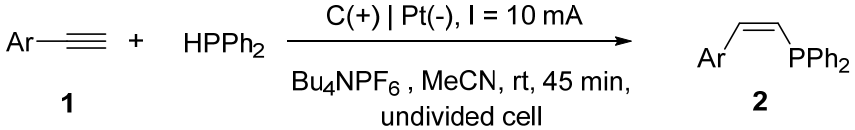


3a 4a

Entry	Variation from the reaction conditions	Conversion[a]
1	None	84%(72%[b])
2	No electric current	0%
3	C(+)|C(-) instead of C(+)|Pt(-)	40%
4	Pt(+)|Pt(-) instead of C(+)|Pt(-)	19%
5	C(+)|Ni(-) instead of C(+)|Pt(-)	27%
6	Air instead of N_2	0%

[a] Reaction conditions: **3a** (0.5 mmol), $HPPh_2$ (0.6 mmol), $^nBu_4NPF_6$ (0.5 mmol), I = 10 mA, MeCN (3.0 mL), N_2, rt, 45 min, isolated yield. Conversion was determined by 1 H NMR spectroscopy. [b] isolated yield.

3. General procedure for the hydrophosphination of alkynes

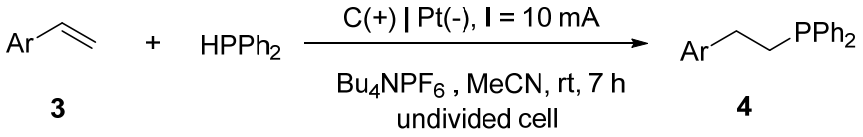


1 2

In a 10 mL over-dried three-necked flask equipped with a magnetic stir bar, alkyne (0.5 mmol), $HPPh_2$ (130 μL, 0.75 mmol), Bu_4NPF_6 (193.7 mg, 0.5 mmol) and MeCN

(3.0 mL) were added, respectively. The flask was equipped with graphite felt electrode (10 × 10 × 3 mm^3) as the anode and platinum plate (10 × 10 × 0.10 mm^3) as the cathode. The mixture was stirred for 45 min under a continuous current of 10 mA at room temperature.

4. General procedure for the hydrophosphination of alkenes

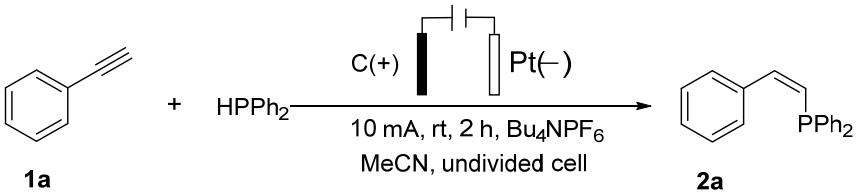


3 **4**

In a 10 mL over-dried three-necked flask equipped with a magnetic stir bar, alkene (0.5 mmol), $HPPh_2$ (87 μL, 0.6 mmol), Bu_4NPF_6 (193.7 mg, 0.5 mmol) and MeCN (3.0 mL) were added, respectively. The flask was equipped with graphite felt electrode (10 × 10 × 3 mm^3) as the anode and platinum plate (10 × 10 × 0.10 mm^3) as the cathode. The mixture was stirred for 7 h under a continuous current of 10 mA at room temperature.

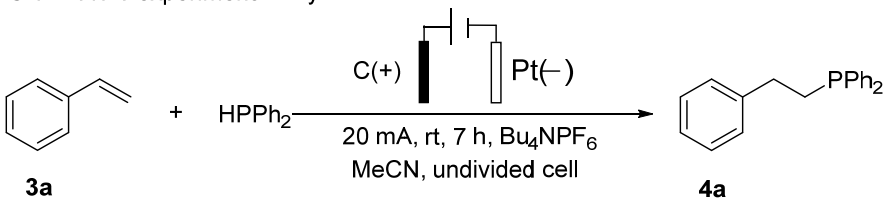
5. Gram-scale hydrophosphination of phenylacetylene and styrene

(A) Gram-scale experiment of phenylacetylene



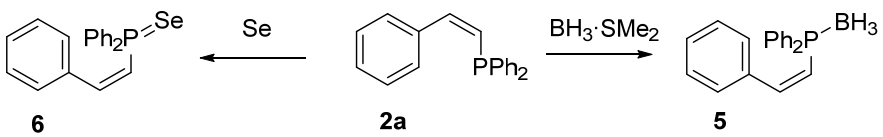
1a (10 mmol, 1.02 g) **2a** (1.76 g, 61% yield)

(B) Gram-scale experiment of styrene



3a (10 mmol, 1.04 g) **4a** (1.63 g, 56% yield)

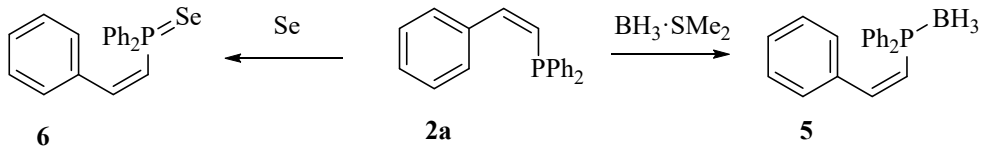
(C) The conversion of vinylphosphines



6 **2a** **5**

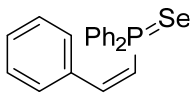
In a 25 mL over-dried flask equipped with a magnetic stirbar, phenylacetylene or styrene (10 mmol), $HPPh_2$ (2.6 mL, 15 mmol), Bu_4NPF_6 (1.94 g, 10 mmol) and MeCN (10 mL) were added, respectively. The flask was equipped with graphite felt electrode ($15 \times 15 \times 3$ mm^3) as the anode and platinum plate ($15 \times 15 \times 0.10$ mm^3) as the cathode. The alkyne mixture was stirred for 2 h under a continuous current of 10 mA at room temperature. The alkenylphosphine products (1.76 g, 61% yield) was obtained by flash column chromatography on silica gel with ethyl acetate/petroleum ether as eluents. The alkene mixture was stirred for 7 h under a continuous current of 20 mA at room temperature. The alkylphosphine products (1.63 g, 56% yield) was obtained by flash column chromatography on silica gel with ethyl acetate/petroleum ether as eluents.

6. Product transformations



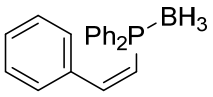
6 **2a** **5**

Se powder (15.8 mg,0.2 mmol, for **2a-Se**) or $BH_3 \cdot SMe_2$ (20 μL, 0.2 mmol, 10 M in Me_2S, for **2a-BH_3**) were added to 5 mL flask containing **2a** (57.6 mg,0.2 mmol) and 3.0 mL CH_3CN. The mixture was stirred 0.5 h at room temperature. The reaction mixture was subjected directly to preparative thin-layer chromatography purification to afford the corresponding product **5** (82% yield) and **6** (86% yield).



(Z)-diphenyl(styryl)phosphine selenide (6)

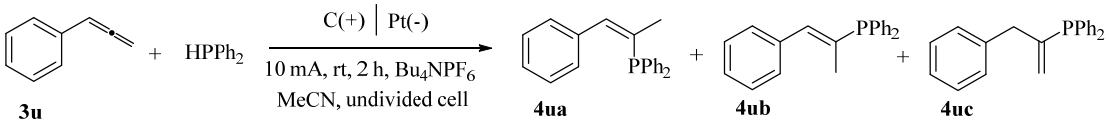
The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4** (63.6 mg, 86% yield). ^1H NMR (600 MHz, $CDCl_3$): δ 7.93 – 7.89 (m, 4H), 7.52 – 7.46 (m, 3H), 7.37 – 7.28 (m, 5H), 7.07 – 7.03 (m, 2H), 6.46 (d, J =18.0, 13.4 Hz, 1H). $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$): δ 145.9 (d, J =2.7 Hz), 134.3 (d, J =6.7 Hz), 131.8 (d, J =10.9 Hz), 131.3 (d, J = 3.1 Hz), 130.2, 128.8, 128.4, 127.5, 122.3, 121.8. $^{31}P\{^1H\}$ NMR (243 MHz, $CDCl_3$): δ 20.1. HRMS (ESI): $[M+H]^+$ 369.0311, found 365.0316.



(Z) -Diphenyl (styryl) phosphonium boride (5)

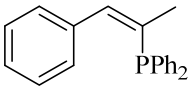
The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **5** (49.6 mg, 82% yield). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.74 – 7.71 (m, 4H), 7.62 – 7.54 (m, 2H), 7.41 – 7.27 (m, 8H), 7.10 – 7.05 (m, 3H), 6.21 (t, *J* =12.9 Hz, 1H), 1.59 – 1.13 (m, 3H). ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$): δ 147.4, 134.8 (d, *J* =5.2 Hz), 132.4 (d, *J* =9.2 Hz), 131.0 (d, *J* =1.8 Hz), 129.5, 129.4, 128.6, 127.6, 119.9, 119.6. ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ 10.8 (d, *J* = 67.1 Hz). HRMS (ESI): $[M+H]^+$ 303.1474, found 303.1467.

7. Reaction with diene substrate



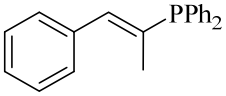
3u **4ua** **4ub** **4uc**

When **3u** as substrate, 68% isolated yield (**4ua**:**4ub**:**4uc**=90:1:9) of hydrophosphinated mixture products was aobtained. Signals belonging to the product **4ua** are denoted with ●, product 4ub are denoted with ●, 4uc are denoted with ● .



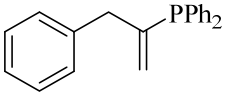
(Z)-diphenyl(1-phenylprop-1-en-2-yl)phosphane (4ua) [10]

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.46 – 7.25 (m, 15H), 6.69 (d, 1H, *J* = 13.8 Hz), 2.00 (dd, 3H, *J* = 9.1, 1.3 Hz). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ 8.1.



(E)-diphenyl(1-phenylprop-1-en-2-yl)phosphane (4ub) [1]

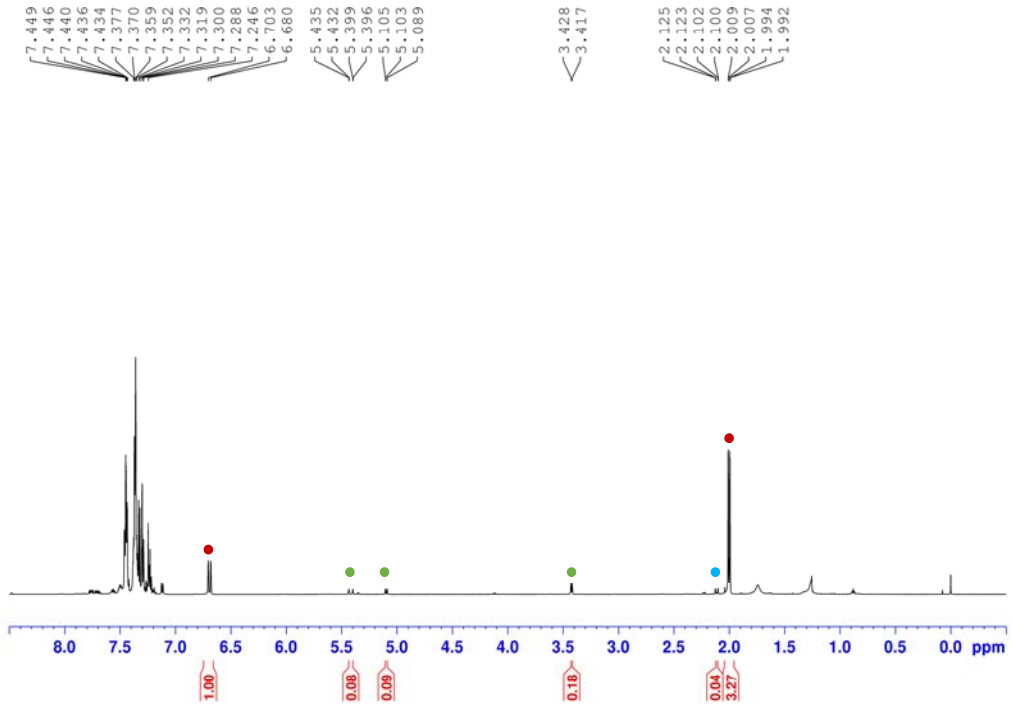
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.46 – 7.25 (m, 16H), 2.11 (dd, 3H, *J* = 13.8, 1.4 Hz). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 13.5.



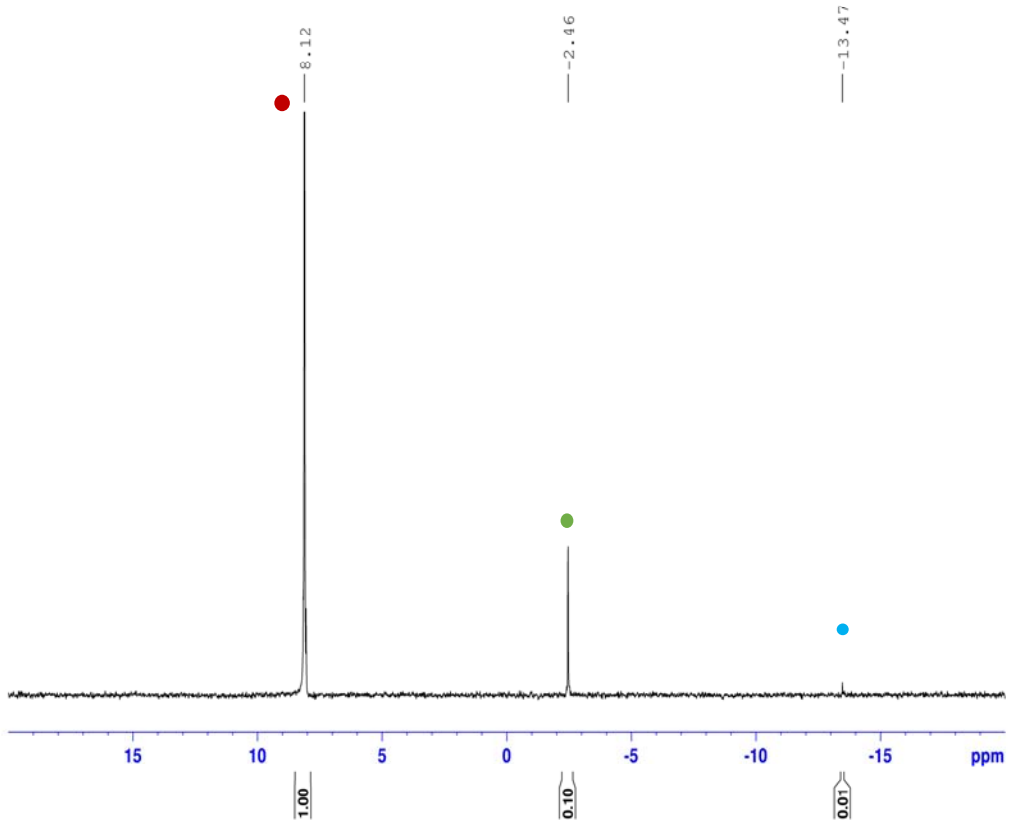
Diphenyl(3-phenylprop-1-en-2-yl)phosphane (4uc) [10]

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.46 – 7.25 (m, 15H), 5.42 (dd, 1H, *J* = 21.4, 1.5 Hz), 5.10 (dd, 1H, *J* = 9.2, 0.9 Hz), 3.42 (d, 2H, *J* = 7.0 Hz). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 2.5.

^{1}H NMR

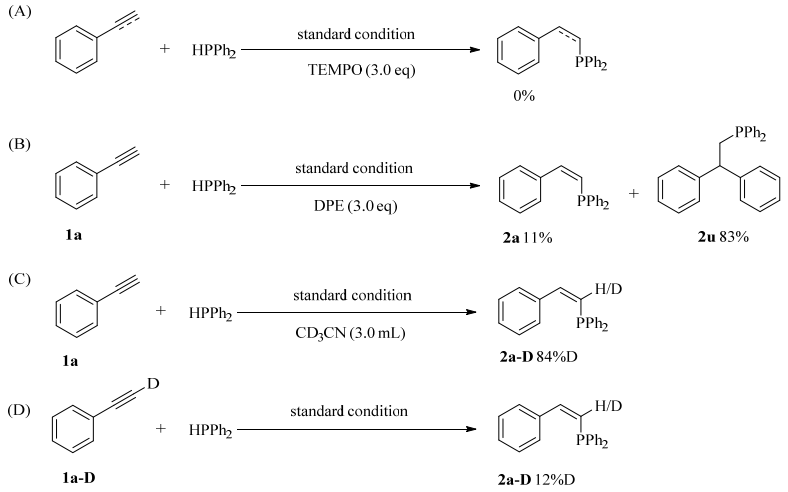


^{31}P{^{1}H}NMR

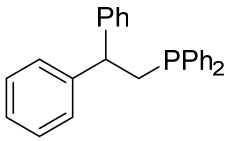


8. Mechanistic Studies

I. Control experiment



In a 10 mL over-dried three-necked flask equipped with a magnetic stir bar, phenylacetylene or styrene (0.5 mmol), $HPPh_2$ (0.75 or 0.6 mmol), Bu_4NPF_6 (0.5 mmol), TEMPO (1.5 mmol) and MeCN (3.0 mL) were added, respectively. The flask was equipped with graphite felt electrode (10 × 10 × 3 mm^3) as the anode and platinum plate (10 × 10 × 0.10 mm^3) as the cathode. The mixture was stirred for 45 min or 7 h under a continuous current of 10 mA at room temperature. The reaction was completely suppressed. The addition of DPE to the phenylacetylene model reaction enabled the isolation and purification of the the radical trapping product **2u** (Conversion 83%). When deuterated acetonitrile was used in the template reaction, the deuterium incorporation rate was 84%, while when deuterated phenylacetylene is used as substrate, the deuteration rate is only 12%, indicating that most of the hydrogen in the product came from acetonitrile.



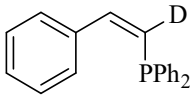
(2,2-diphenylethyl)diphenylphosphane (2u)

Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2u** (128.2 mg, 70% yield).

^1H NMR (600 MHz, $CDCl_3$): δ 7.47 – 7.44 (m, 4H), 7.37 – 7.36 (m, 6H), 7.33 – 7.30 (m, 4H), 7.29

– 7.21 (m, 6H), 4.03 – 3.98 (m, 1H), 2.91 – 2.89 (m, 2H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 20.8.

Spectroscopic data matches that reported in the literature [9].



(Z)-diphenyl(2-phenylvinyl-1-d)phosphane (2a-D)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2a-D** (94.0 mg, 63% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 – 7.50 (m, 2H), 7.45 – 7.43 (m, 4H), 7.33 – 7.23 (m, 10H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 25.2.

II. Procedures for cyclic voltammetry (CV)

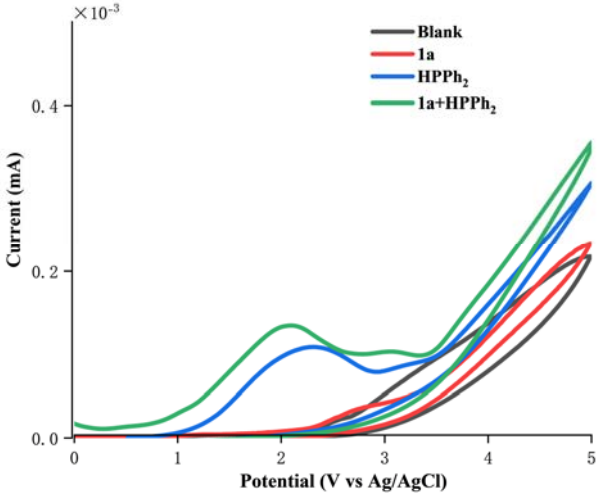
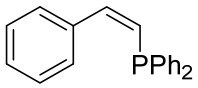


Fig S1. Cyclic voltammetry measurements.

Cyclic voltammetry experiments were conducted in a 25 mL three-electrode cell equipped with a glassy carbon working electrode, an Ag/AgCl reference electrode submerged in saturated aqueous KCl, and a platinum wire counter electrode. All experiments were performed at a scan rate of 100 mV s^{-1} in MeCN containing 0.1 M Bu_4NPF_6 as the supporting electrolyte. The potential range was set to −5 to 5 V, and the following systems were studied: Background electrolyte (Bu_4NPF_6, 0.1 M in MeCN); $HPPh_2$ (0.1 M in MeCN); Phenylacetylene (0.1 M in MeCN); A mixture of

phenylacetylene (0.1 M in MeCN) and $HPPh_2$ (0.1 M in MeCN). Currents and potentials were reported in mA and V, respectively. The CV analysis of the phenylacetylene in CH_3CN (blue) displayed the oxidation wave at 2.8 V (vs Ag/AgCl) respectively. However, the mixture of diphenylphosphine and phenylacetylene in CH_3CN (green) shows an oxidation peak at 2.0 and 3.0 V (vs Ag/AgCl). The attenuation of the diphenylphosphine oxidation peak suggests its preferential oxidation in the mixed system.

9. Spectroscopic Data of Products

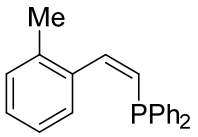


(Z)-Diphenyl(styryl)phosphane (2a)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2a** (115.4 mg, 80% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.46 – 7.36 (m, 6H), 7.34 – 7.26 (m, 10H), 6.46 (dd, *J* = 12.7, 2.8 Hz, 1H). $^{31}P\{^1H\}$ NMR (243 MHz, $CDCl_3$): δ –24.8.

Spectroscopic data matches that reported in the literature [1].

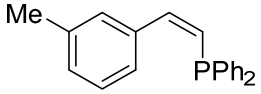


(Z)-(2-Methylstyryl)diphenylphosphane (2b)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2b** (98.7 mg, 65% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.47 – 7.43 (m, 1H), 7.42 – 7.40 (m, 4H), 7.32 – 7.29 (m, 7H), 7.18 – 7.10 (m, 3H), 6.54 (dd, *J* = 12.2, 2.9 Hz, 1H), 2.30 (s, 3H). $^{31}P\{^1H\}$ NMR (243 MHz, $CDCl_3$): δ – 27.0.

Spectroscopic data matches that reported in the literature [2].

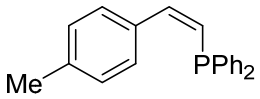


(Z)-(3-Methylstyryl)diphenylphosphane (2c)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2c** (93.4 mg, 62% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.43 – 7.36 (m, 4H), 7.34 – 7.28 (m, 9H), 7.22 – 7.06 (m, 1H), 6.43 (dd, *J* = 12.7, 2.6 Hz, 1H) ,2.30 (s, 3H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 24.5.

Spectroscopic data matches that reported in the literature [1].

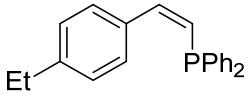


(Z)-(4-Methylstyryl)diphenylphosphane (2d)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2d** (117.9 mg, 78% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 – 7.47 (m, 6H), 7.39 – 7.36 (m, 7H), 7.18 – 7.17 (m, 2H), 6.44 (dd, *J* = 12.7, 2.9 Hz, 1H) ,2.38 (s, 3H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ –24.8.

Spectroscopic data matches that reported in the literature [1].

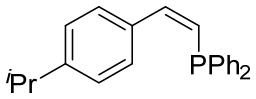


(Z)-(4-Ethylstyryl)diphenylphosphane (2e)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2e** (93.9 mg, 59% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.45 – 7.43 (m, 6H), 7.38 – 7.30 (m, 7H), 7.25 – 7.14 (m, 2H), 6.39 (dd, *J* = 12.7, 3.0 Hz, 1H) ,2.62 (q, *J* = 7.6 Hz, 2H), 1.21 (t, *J* = 7.6 Hz, 3H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 24.8.

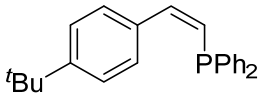
Spectroscopic data matches that reported in the literature [3].



(Z)-(4-Isopropylstyryl)diphenylphosphane (2f)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2f** (110.5 mg, 67% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.45 – 7.43 (m, 6H), 7.34 – 7.31 (m, 7H), 7.25 – 7.17 (m, 2H), 6.39 (dd, *J* = 12.7, 2.9 Hz, 1H), 2.90 – 2.86 (m, 1H), 1.23 (d, *J* = 6.9 Hz, 6H). ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$): δ 149.0, 144.1 (d, *J* = 19.0 Hz), 139.5 (d, *J* = 9.3 Hz), 134.5, 132.7 (d, *J* = 18.7 Hz), 129.6 (d, *J* = 8.7 Hz), 128.5 (d, *J* = 6.6 Hz), 128.4, 128.2 (d, *J* = 15.6 Hz), 126.2, 33.9, 23.9. ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 24.7. HRMS (ESI): $[M+H]^+$ 331.1616, found 331.1613.

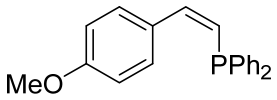


(Z)-(4-(Tert-butyl)styryl)diphenylphosphane (2g)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2g** (103.4 mg, 60% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.52 – 7.49 (m, 6H), 7.40 – 7.36 (m, 9H), 6.45 (dd, *J* = 12.7, 3.0 Hz, 1H), 1.35 (s, 9H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ –24.7.

Spectroscopic data matches that reported in the literature [4].

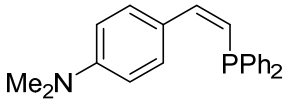


(Z)-(4-Methoxystyryl)diphenylphosphane (2h)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2h** (105.1 mg, 66% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.52 – 7.48 (m, 6H), 7.38 – 7.34 (m, 7H), 6.89 (d, *J* = 8.7 Hz, 2H), 6.36 (dd, *J* = 12.6, 2.8 Hz, 1H), 3.82 (s, 3H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 24.6.

Spectroscopic data matches that reported in the literature [1].

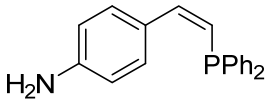


(Z)-4-(2-(Diphenylphosphaneyl)vinyl)-N,N-dimethylaniline (2i)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2i** (88.2 mg, 53% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.53 – 7.51 (m, 6H), 7.37 – 7.36 (m, 7H), 6.71 – 6.69 (m, 2H), 6.23 – 6.20 (m, 1H), 2.99 (s, 6H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 23.9.

Spectroscopic data matches that reported in the literature [1].

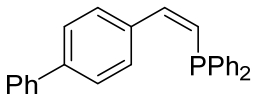


(Z)-4-(2-(Diphenylphosphaneyl)vinyl)aniline (2j)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2j** (110.9 mg, 73% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 – 7.46 (m, 4H), 7.42 – 7.39 (m, 2H), 7.36 – 7.33 (m, 7H), 6.64 – 6.63 (m, 2H), 6.23 (dd, *J* = 12.7, 2.8 Hz, 1H), 3.7 (br, 2H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ –24.2.

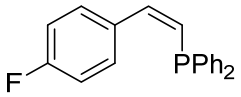
Spectroscopic data matches that reported in the literature [2].



(Z)-(2-([1,1'-Biphenyl]-4-yl)vinyl)diphenylphosphane (2k)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2k** (159.6 mg, 88% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.47 – 7.45 (m, 5H), 7.43 – 7.40 (m, 7H), 7.35 – 7.32 (m, 8H), 6.48 (dd, *J* = 12.7, 2.7 Hz, 1H). $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$): δ 143.6 (d, *J* = 18.9 Hz), 140.7 (d, *J* = 26.6 Hz), 139.3 (d, *J* = 9.0 Hz), 135.9, 133.1 (d, *J* = 18.7 Hz), 132.8 (d, *J* = 18.8 Hz), 130.0 (d, *J* = 8.6 Hz), 129.5 (d, *J* = 16.0 Hz), 128.8, 128.6 (d, *J* = 6.9 Hz), 127.4, 127.0, 126.8. $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 24.5. HRMS (ESI): $[M+H]^+$ 365.1459, found 365.1461.

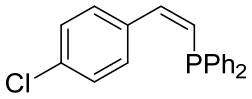


(Z)-(4-Fluorostyryl)diphenylphosphane (2l)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2l** (124.5 mg, 81% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 – 7.48 (m, 6H), 7.40 – 7.36 (m, 7H), 7.06 – 7.03 (m, 2H), 6.50 (dd, *J* = 12.7, 2.3 Hz, 1H). ^{31}P NMR (243 MHz, $CDCl_3$): δ –25.0. ^{19}F{^{1}H} NMR (565 MHz, $CDCl_3$): δ –113.1.

Spectroscopic data matches that reported in the literature [2].

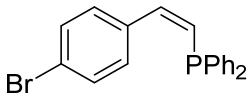


(Z)-(4-Chlorostyryl)diphenylphosphane (2m)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2m** (101.9 mg, 63% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.48 – 7.46 (m, 6H), 7.38 – 7.35 (m, 7H), 7.32 – 7.28 (m, 2H), 6.53 (dd, *J* = 12.7, 2.5 Hz, 1H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 24.9.

Spectroscopic data matches that reported in the literature [2].

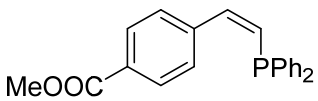


(Z)-(4-Bromostyryl)diphenylphosphane (2n)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2n** (108.3 mg, 59% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.47 – 7.45 (m, 6H), 7.40 – 7.28 (m, 9H), 6.54 (d, *J* = 12.7 Hz, 1H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 25.0.

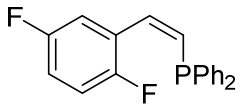
Spectroscopic data matches that reported in the literature [2].



Methyl (Z)-4-(2-(diphenylphosphaneyl)vinyl)benzoate (2o)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2o** (115.6 mg, 67% yield).

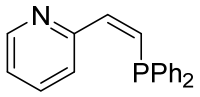
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.05 – 8.03 (d, *J* = 8.3 Hz, 2H), 7.53 – 7.50 (m, 6H), 7.41 – 7.40 (m, 6H), 7.13 (dd, *J* = 17.0, 11 Hz, 1H), 6.90 (dd, *J* = 17.0, 12 Hz, 1H), 3.95 (s, 3H). ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$): δ 166.8, 141.6 (d, *J* = 26.8 Hz), 141.3 (d, *J* = 11.2 Hz), 137.4 (d, *J* = 9.2 Hz), 133.3 (d, *J* = 19.0 Hz), 130.4 (d, *J* = 13.9 Hz), 130.0, 129.0, 128.7 (d, *J* = 6.7 Hz), 126.7, 52.2. ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 10.8. HRMS (ESI): $[M+H]^+$ 347.1196, found 347.1194.



(Z)-(2,5-Difluorostyryl)diphenylphosphane (2p)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2p** (114.2 mg, 70% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.61 – 7.57 (m, 1H), 7.48 – 7.45 (m, 5H), 7.40 – 7.36 (m, 6H), 6.84 – 6.81 (m, 2H), 6.62 (dd, *J* = 12.7, 2.0 Hz, 1H). ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$): δ 162.7 (dd, *J* = 250.8, 12.2 Hz), 161.9(dd, *J* = 251.7, 11.9 Hz), 138.6 (d, *J* = 8.9 Hz), 135.1 (d, *J* = 19.4, 4.2 Hz), 132.8, 132.7, 131.8, 131.7, 128.6 (t, *J* = 6.9 Hz), 121.2 (dt, *J* = 13.1, 2.6 Hz), 110.8 (dd, *J* = 21.4, 3.4 Hz), 103.7 (t, *J* = 25.5 Hz). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 25.2 (d, *J* = 5.8 Hz). ^{19}F{^{1}H} NMR (565 MHz, $CDCl_3$): δ – 109.3 (d, *J* = 8.1 Hz), – 111.2 (d, *J* = 7.7 Hz). HRMS (ESI): $[M+H]^+$ 325.0958, found 325.0961.



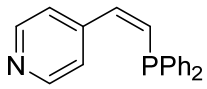
(Z)-2-(2-(Diphenylphosphaneyl)vinyl)pyridine (2q)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2q** (113.4 mg, 78% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.60 – 8.59 (d, *J* = 4.6 Hz), 7.67 – 7.64 (m, 1H), 7.53 – 7.37 (m, 10H), 7.31 – 7.28 (m, 2H), 7.19 – 7.16 (m, 1H), 6.84 (dd, *J* =16.9, 11.2 Hz, 1H). ^{31}P{^{1}H} NMR (243

MHz, $CDCl_3$): δ – 11.2.

Spectroscopic data matches that reported in the literature [5].

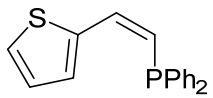


(Z)-4-(2-(Diphenylphosphaneyl)vinyl)pyridine (2r)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2r** (119.8 mg, 83% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.54 (d, *J* = 6.0 Hz, 2H), 7.46 – 7.40 (m, 4H), 7.37 – 7.36 (m, 6H), 7.24 – 7.23 (m, 3H), 7.27 – 7.26 (m, 3H), 6.65 (dd, *J* =17.1, 10.9 Hz, 1H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 10.7.

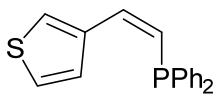
Spectroscopic data matches that reported in the literature [5].



(Z)-Diphenyl(2-(thiophen-2-yl)vinyl)phosphane (2s)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2s** (101.8 mg, 69% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.54 – 7.51 (m, 5H), 7.40 – 7.35 (m, 7H), 7.22 – 7.21 (m, 1H), 7.05 – 7.03 (m, 1H), 6.40 – 6.37 (m, 1H). ^{13}C{1H} NMR (151 MHz, $CDCl_3$): δ 140.6, 138.7 (d, *J* = 9.0 Hz), 136.5 (d, *J* = 22.4 Hz), 132.7 (d, *J* = 18.8 Hz), 130.5 (d, *J* = 3.9 Hz),128.6, 128.5 (d, *J* = 2.1 Hz), 128.3 (d, *J* = 10.8 Hz), 126.8, 126.4 (d, *J* = 15.6 Hz). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 26.4. HRMS (ESI): $[M+H]^+$ 295.0705, found 295.0722.

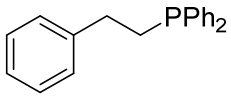


(Z)-diphenyl(2-(thiophen-3-yl)vinyl)phosphane (2t)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **2t** (87.0 mg, 59% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 – 7.46 (m, 6H), 7.40 – 7.37 (m, 7H), 7.30 – 7.25 (m, 1H), 6.40 (d, *J* =12.6 Hz, 1H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 23.9.

Spectroscopic data matches that reported in the literature [4].

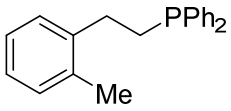


Phenethyldiphenylphosphane(4a)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4a** (104.3 mg, 72% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.46 – 7.43 (m, 4H), 7.35 – 7.32 (m, 6H), 7.28 – 7.25 (m, 2H), 7.19 – 7.16 (m, 3H), 2.74 – 2.70 (m, 2H), 2.38 – 2.35 (m, 2H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 15.9.

Spectroscopic data matches that reported in the literature [6].

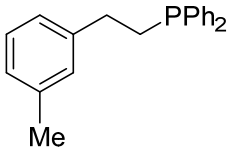


(2-methylphenethyl)diphenylphosphine(4b)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4b** (104.3 mg, 75% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58 – 7.55 (m, 4H), 7.43 – 7.41 (m, 6H), 7.22 – 7.18 (m, 4H), 2.81 – 2.77 (m, 2H), 2.41 – 2.38 (m, 2H), 2.27 (s, 3H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 15.4.

Spectroscopic data matches that reported in the literature [6].



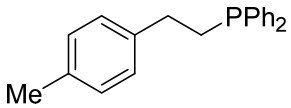
(3-methylphenethyl)diphenylphosphane（4c）

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4c** (104.3 mg, 63% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.45 – 7.35 (m, 4H), 7.35 – 7.33 (m, 6H), 7.17 – 7.14 (m, 1H), 7.00

– 6.69 (m, 3H), 2.70 – 2.66 (m, 2H), 2.37 – 2.34 (m, 2H), 2.31 (s, 3H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 15.7.

Spectroscopic data matches that reported in the literature [6].

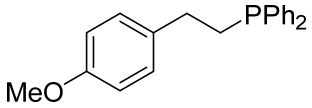


4-methylphenethyl)diphenylphosphane(4d)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4d** (85.5 mg, 56% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.45 – 7.43 (m, 4H), 7.35 – 7.32 (m, 6H), 7.10 – 7.05 (m, 4H), 2.70 – 2.66 (m, 2H), 2.36 – 2.33 (m, 2H), 2.31 (s, 3H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 16.0.

Spectroscopic data matches that reported in the literature [6].

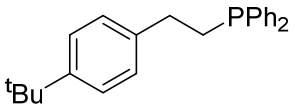


(4-methoxyphenethyl)diphenylphosphane (4e)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4e** (91.2 mg, 57% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.45 – 7.43 (m, 4H), 7.35 – 7.32 (m, 6H), 7.10 – 7.09 (m, 2H), 6.82 – 6.81 (m, 2H), 3.78 (s, 3H), 2.78 – 2.65 (m, 2H), 2.35 – 2.32 (m, 2H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 16.2.

Spectroscopic data matches that reported in the literature [6].



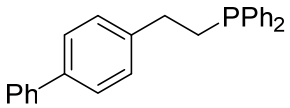
(4-tert-butylphenethyl)diphenylphosphane (4f)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4f** (125.2 mg, 72% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 – 7.46 (m, 4H), 7.36 – 7.35 (m, 6H), 7.33 – 7.31 (m, 2H), 7.15

– 7.14 (m, 2H), 2.75 – 2.71 (m, 2H), 2.41 – 2.38 (m, 2H), 1.33 (s, 9H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 15.7.

Spectroscopic data matches that reported in the literature [6].

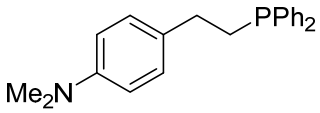


{2-[(1,1'-Biphenyl)-4-yl]ethyl}diphenylphosphane (4g)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4g** (135.6 mg, 74% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.67 – 7.66 (m, 2H), 7.60 – 7.50 (m, 8H), 7.44 – 7.42 (m, 7H), 7.34 – 7.33 (m, 2H), 2.89 – 2.87 (m, 2H), 2.52 – 2.49 (m, 2H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 15.8.

Spectroscopic data matches that reported in the literature [6].

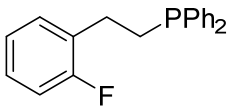


(4-dimethylaminophenethyl)diphenylphosphane (4h)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4h** (73.8 mg, 44% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.53 – 7.51 (m, 4H), 7.41 – 7.39 (m, 6H), 7.13 – 7.11 (m, 2H), 6.76 – 6.74 (m, 2H), 2.97 (s, 6H), 2.73 – 2.70 (m, 2H), 2.42 – 2.39 (m, 2H). $^{31}P\{^{1}H\}$ NMR (243 MHz, $CDCl_3$): δ – 16.0.

Spectroscopic data matches that reported in the literature [6].

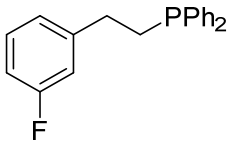


(2-fluorophenethyl)diphenylphosphane (4i)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4i** (103.4 mg, 67% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 – 7.49 (m, 4H), 7.40 – 7.37 (m, 6H), 7.22 – 7.18 (m, 2H), 7.09 – 7.01 (m, 2H), 2.83 – 2.78 (m, 2H), 2.42 – 2.38 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 15.8. ^{19}F{^{1}H} NMR (565 MHz, $CDCl_3$): δ – 118.5.

Spectroscopic data matches that reported in the literature [6].

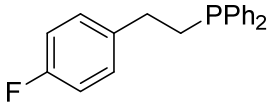


(3-fluorophenethyl)diphenylphosphane (4j)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4j** (87.6 mg, 57% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.53 – 7.51 (m, 4H), 7.41 – 7.39 (m, 6H), 7.30 – 7.25 (m, 1H), 7.01 – 7.00 (m, 1H), 6.95 – 6.92 (m, 1H), 2.81 – 2.77 (m, 2H), 2.44 – 2.41 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 16.1. ^{19}F{^{1}H} NMR (565 MHz, $CDCl_3$): δ – 113.4.

Spectroscopic data matches that reported in the literature [6].

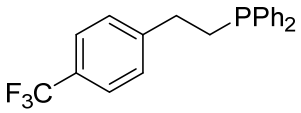


(4-fluorophenethyl)diphenylphosphane (4k)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4k** (93.9 mg, 61% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 – 7.46 (m, 4H), 7.38 – 7.37 (m, 6H), 7.16 – 7.14 (m, 2H), 7.00 – 6.97 (m, 2H), 2.75 – 2.71 (m, 2H), 2.39 – 2.36 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 16.3. ^{19}F{^{1}H} NMR (565 MHz, $CDCl_3$): δ – 117.4.

Spectroscopic data matches that reported in the literature [6].



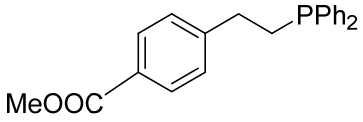
(4-trifluoromethylphenethyl)diphenylphosphane (4l)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4l**

(75.2 mg, 42% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.54 – 7.53 (m, 2H), 7.48 – 7.45 (m, 4H), 7.38 – 7.37 (m, 6H), 7.30 – 7.28 (m, 2H), 2.82 – 2.78 (m, 2H), 2.40 – 2.38 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 16.0. ^{19}F{^{1}H} NMR (565 MHz, $CDCl_3$): δ – 62.3.

Spectroscopic data matches that reported in the literature [6].

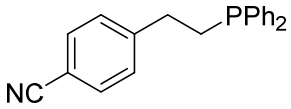


Methyl 4-(2-(diphenylphosphino)ethyl)benzoate (4m)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4m** (116.0 mg, 64% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.97 – 7.96 (m, 2H), 7.49 – 7.46 (m, 4H), 7.39 – 7.36 (m, 5H), 7.26 – 7.25 (m, 2H), 3.93 (s, 3H), 2.81 – 2.77 (m, 2H), 2.41 – 2.38 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 15.9.

Spectroscopic data matches that reported in the literature [7].

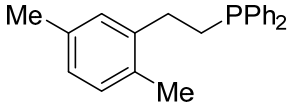


4-(2-(diphenylphosphino)ethyl)benzonitrile (4n)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4n** (75.9 mg, 48% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58 – 7.57 (m, 2H), 7.48 – 7.46 (m, 4H), 7.39 – 7.38 (m, 6H), 7.29 – 7.28 (m, 2H), 2.83 – 2.79 (m, 2H), 2.41 – 2.38 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 16.2.

Spectroscopic data matches that reported in the literature [7].



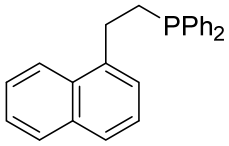
(2,5-dimethylphenethyl)diphenylphosphane (4o)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material

was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4o** (71.4 mg, 45% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58 – 7.56 (m, 4H), 7.44 – 7.40 (m, 6H), 7.10 – 7.00 (m, 3H), 2.78 – 2.76 (m, 2H), 2.41 – 2.40 (m, 2H), 2.38 (s, 3H), 2.23 (s, 3H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 15.2.

Spectroscopic data matches that reported in the literature [7].

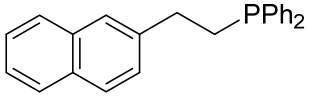


[2-(naphthalen-1-yl)ethyl]diphenylphosphane (4p)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4p** (83.7 mg, 49% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.91 – 7.90 (m, 1H), 7.85 – 7.83 (m, 1H), 7.78 – 7.77 (m, 1H), 7.59 – 7.39 (m, 14H), 3.26 – 3.22 (m, 2H), 2.57 – 2.54 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 15.3.

Spectroscopic data matches that reported in the literature [7].

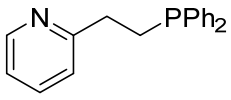


[2-(naphthalen-2-yl)ethyl]diphenylphosphane (4q)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4q** (97.1 mg, 57% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.89 – 7.84 (m, 3H), 7.70 (s, 1H), 7.85 – 7.83 (m, 1H), 7.60 – 7.50 (m, 6H), 7.46 – 7.39 (m, 7H), 3.01 – 2.98 (m, 2H), 2.58 – 2.54 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 15.6.

Spectroscopic data matches that reported in the literature [7].

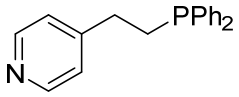


2-(2-(diphenylphosphino)ethyl)pyridine (4r)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4r** (109.5 mg, 75% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.55 (d, *J* = 4.8 Hz 1H), 7.57 – 7.55 (m, 1H), 7.51 – 7.49 (m, 4H), 7.37 – 7.32 (m, 6H), 7.13 – 7.09 (m, 2H), 2.96 – 2.92 (m, 2H), 2.56 – 2.54 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 15.4.

Spectroscopic data matches that reported in the literature [7].

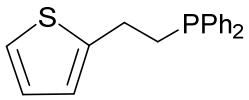


4-(2-(diphenylphosphino)ethyl)pyridine (4s)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4s** (97.7 mg, 67% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.50 – 8.49 (m, 2H), 7.48 – 7.45 (m, 4H), 7.38 – 7.37 (m, 6H), 7.12 – 7.11 (m, 2H), 2.75 – 2.71 (m, 2H), 2.40 – 2.36 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 16.0.

Spectroscopic data matches that reported in the literature [7].



Diphenyl(2-(thiophen-2-yl)ethyl)phosphane (4t)

On 0.5 mmol scale. Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ethyl acetate) to give **4t** (59.3 mg, 40% yield).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 – 7.46 (m, 4H), 7.39 – 7.36 (m, 6H), 7.14 (d, 1H), 6.94 – 6.93 (m, 1H), 6.82 (d, 1H), 2.99 – 2.95 (m, 2H), 2.48 – 2.45 (m, 2H). ^{31}P{^{1}H} NMR (243 MHz, $CDCl_3$): δ – 16.4.

Spectroscopic data matches that reported in the literature [8].

10. Spectra of prepared compounds

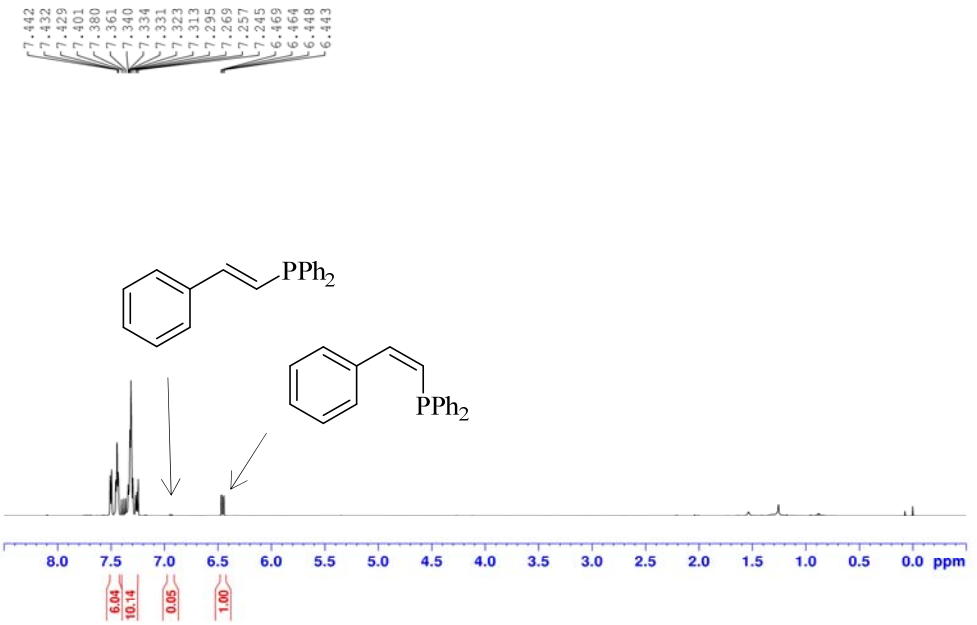


Figure S2. ^{1}H NMR spectrum of compound **2a**

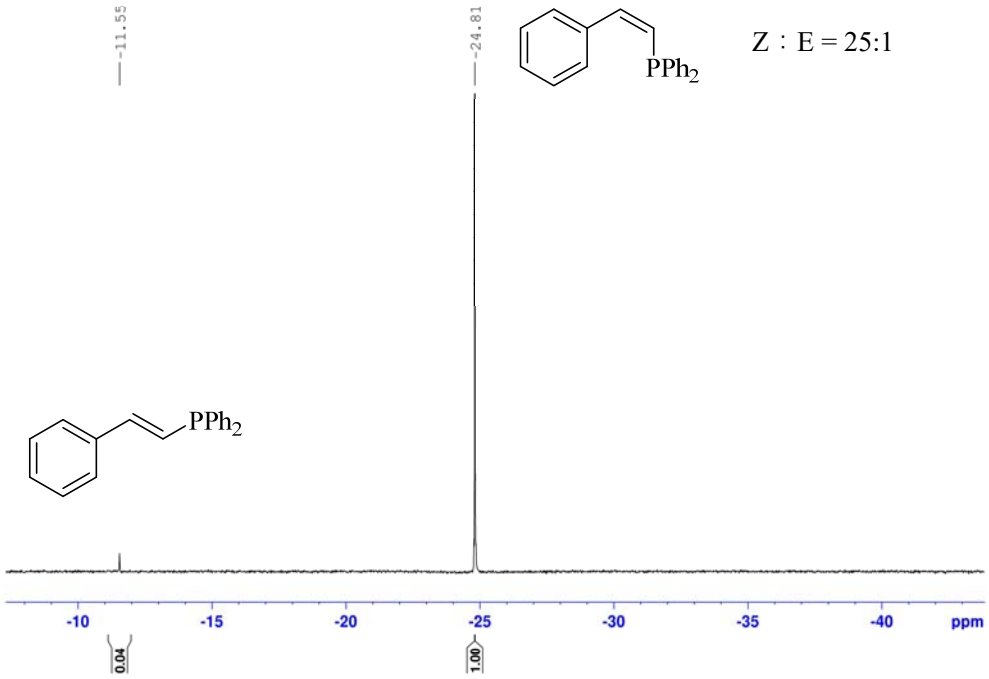


Figure S3. ^{31}P NMR spectrum of compound **2a**

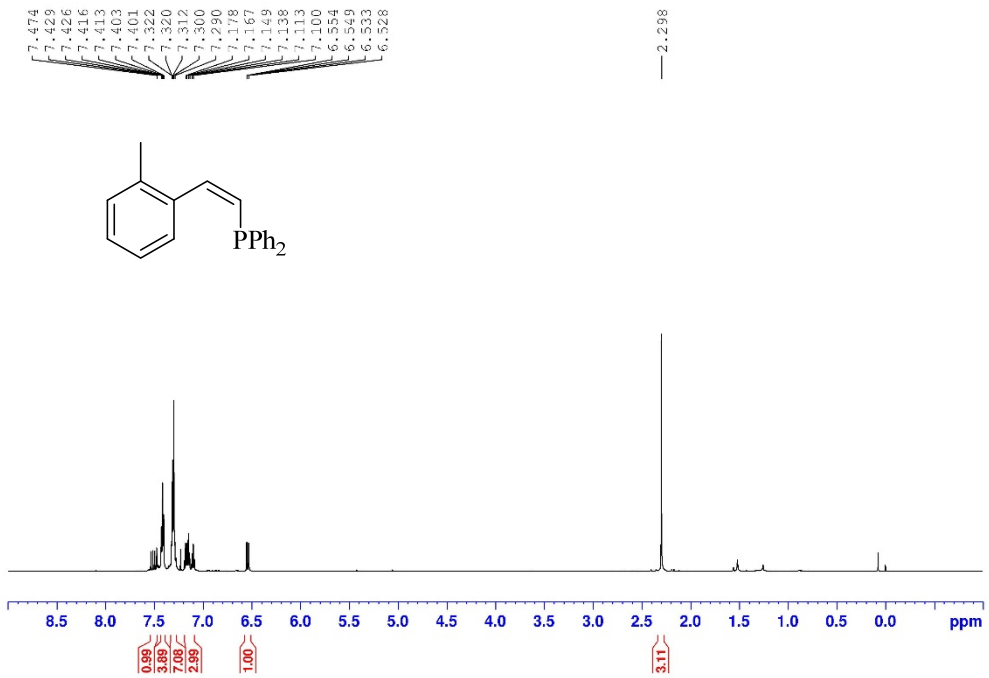


Figure S4. [1]H NMR spectrum of compound **2b**

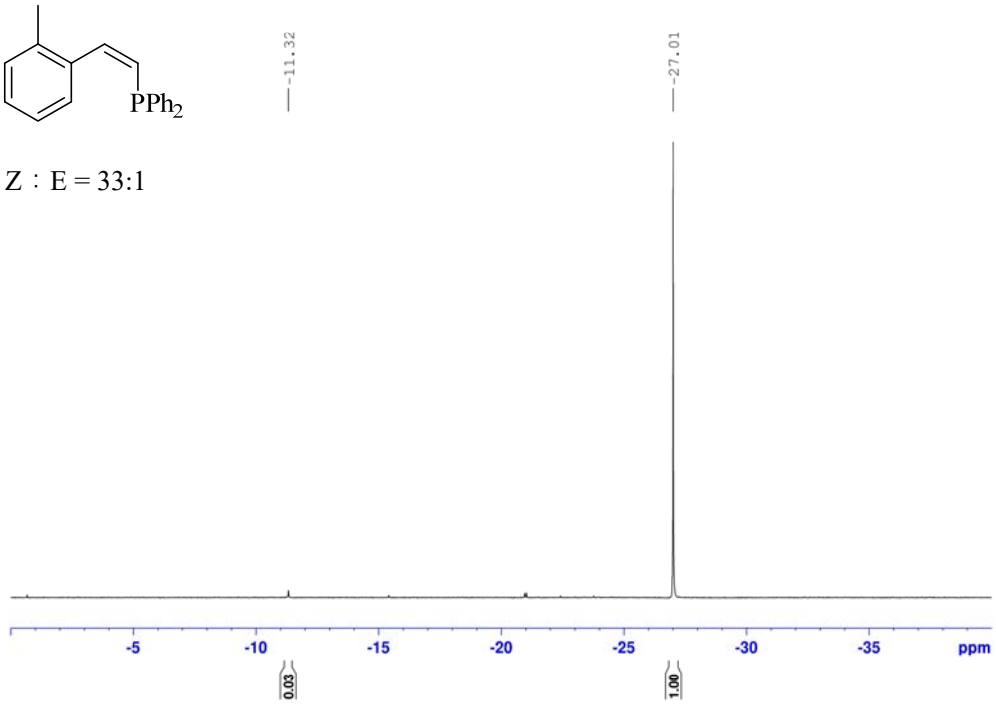


Figure S5. [31]P NMR spectrum of compound **2b**

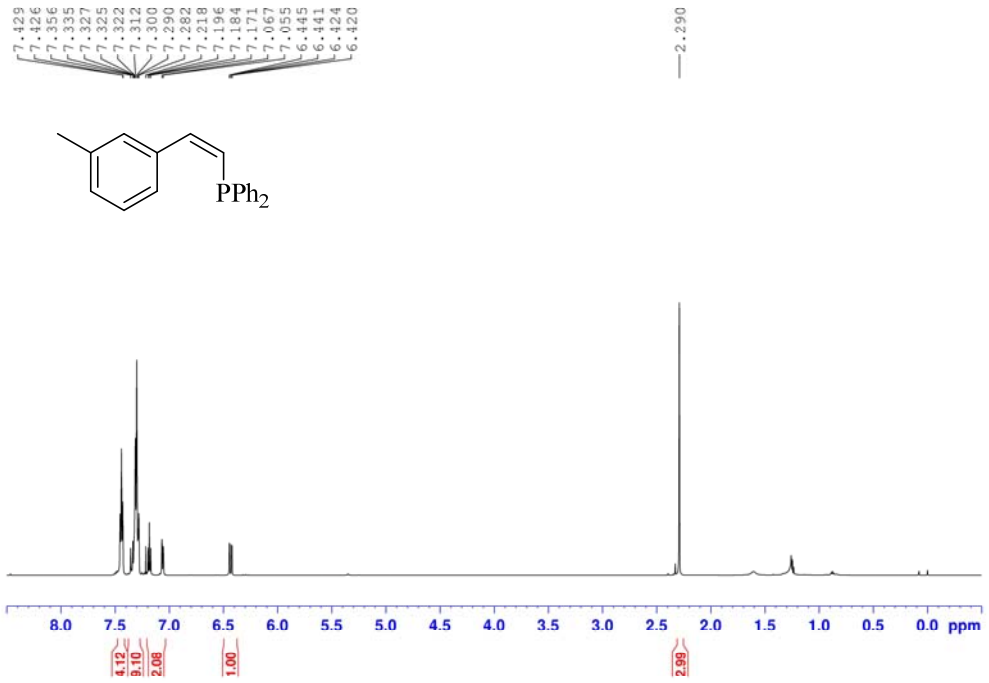
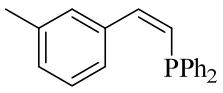


Figure S6. ^{1}H NMR spectrum of compound **2c**



Z：E = 33:1

Figure S7. ^{31}P NMR spectrum of compound **2c**

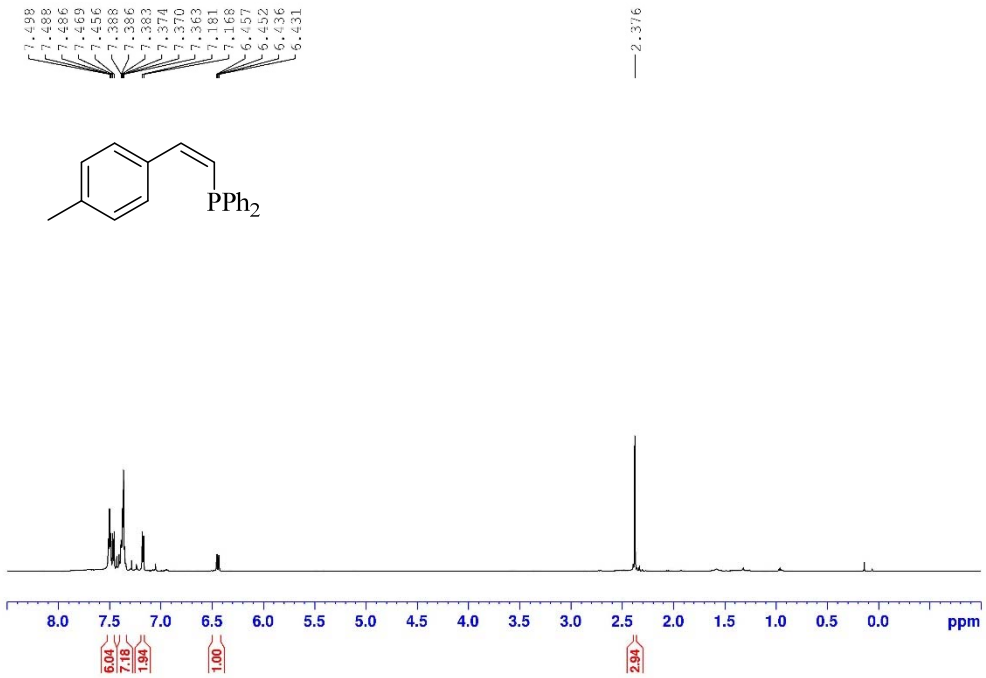


Figure S8. ^{1}H NMR spectrum of compound **2d**

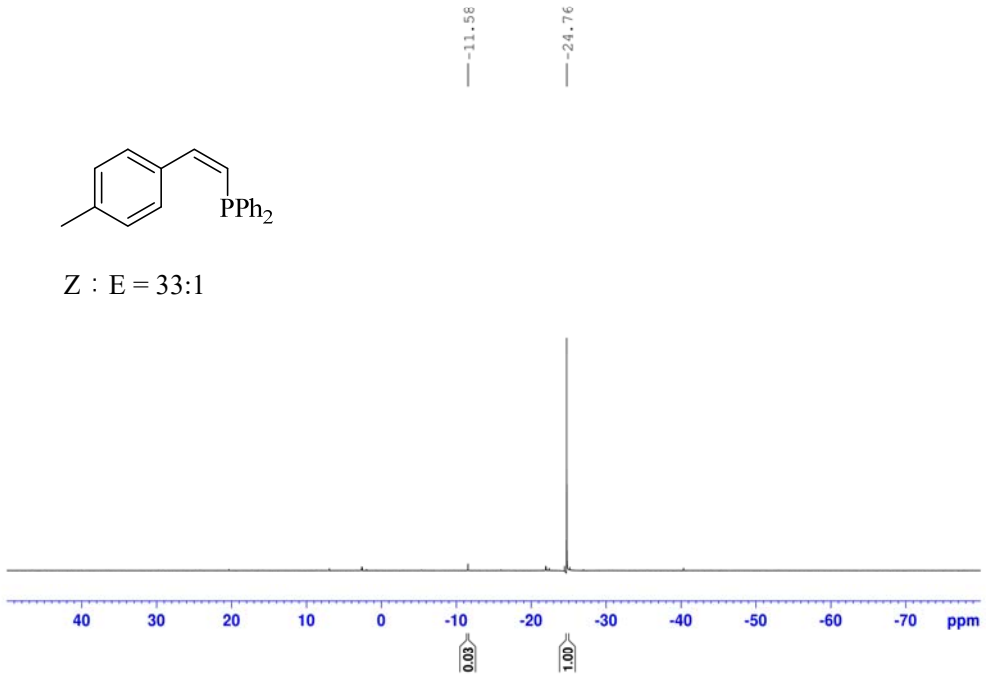


Figure S9. ^{31}P NMR spectrum of compound **2d**

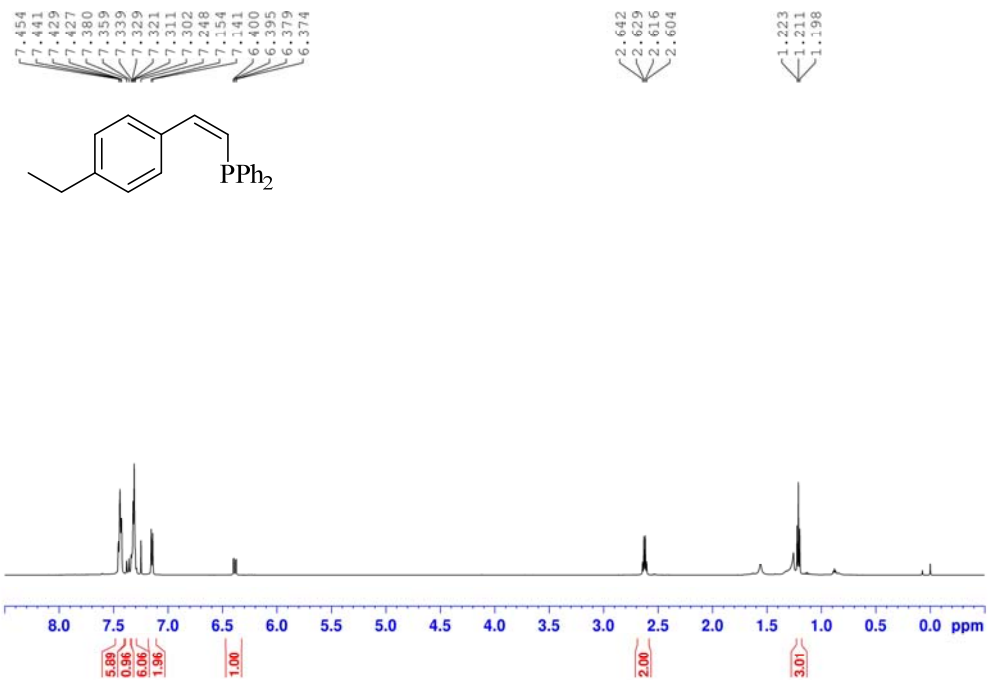


Figure S10. ^{1}H NMR spectrum of compound **2e**

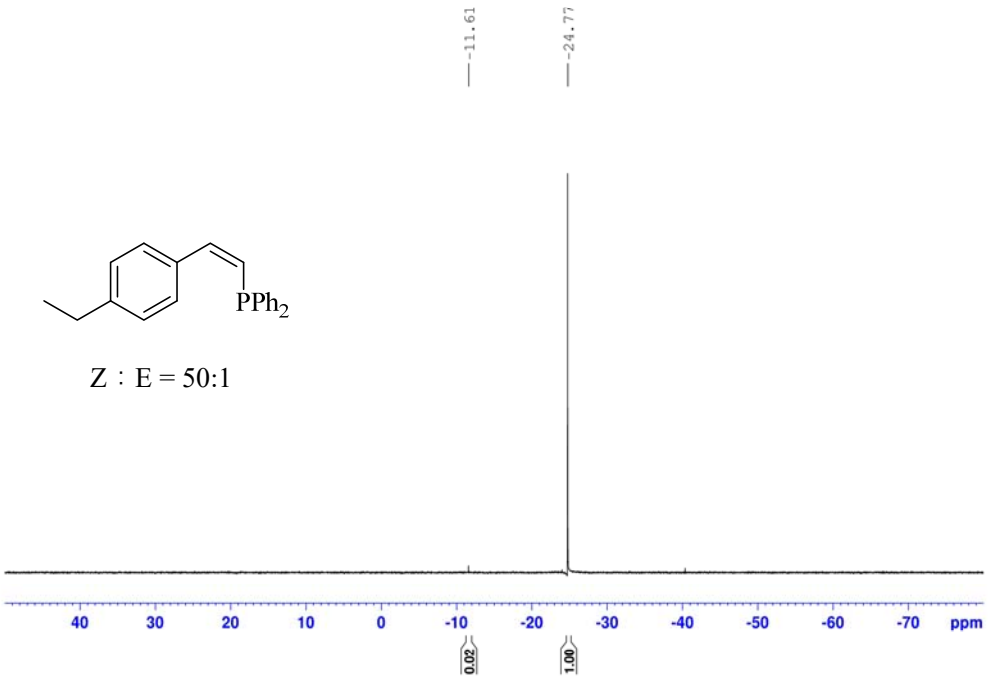


Figure S11. ^{31}P NMR spectrum of compound **2e**

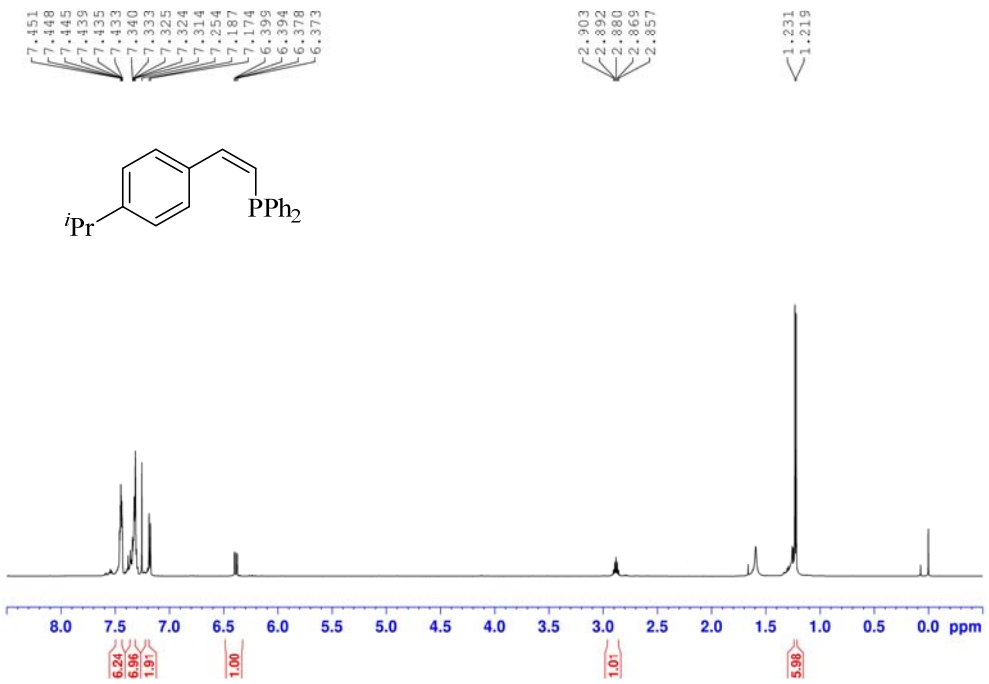


Figure S12. ^{1}H NMR spectrum of compound **2f**

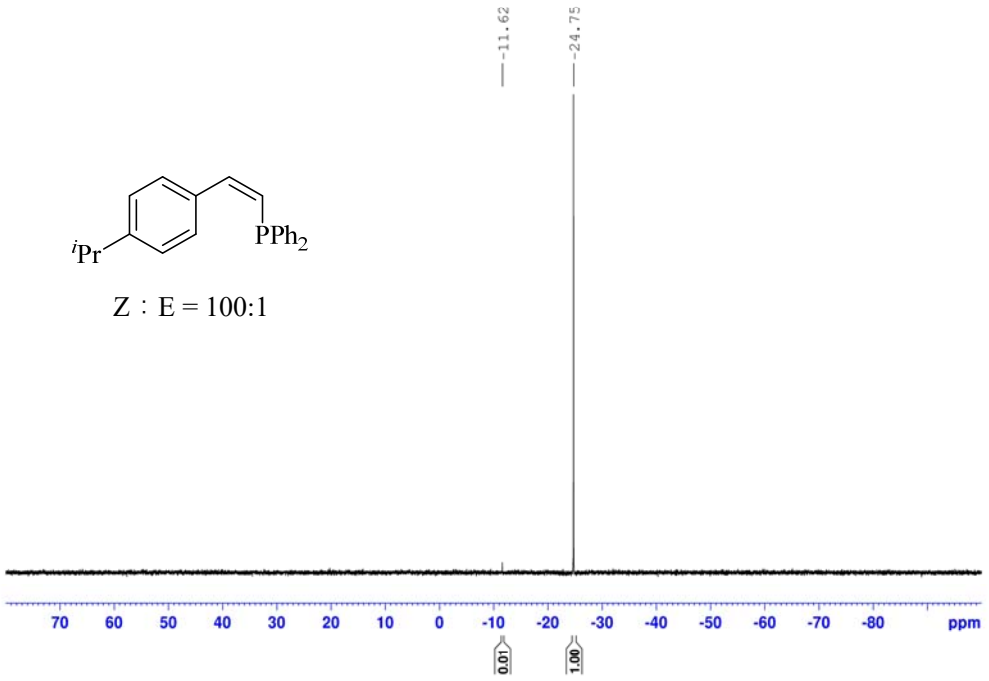


Figure S13. ^{31}P NMR spectrum of compound **2f**

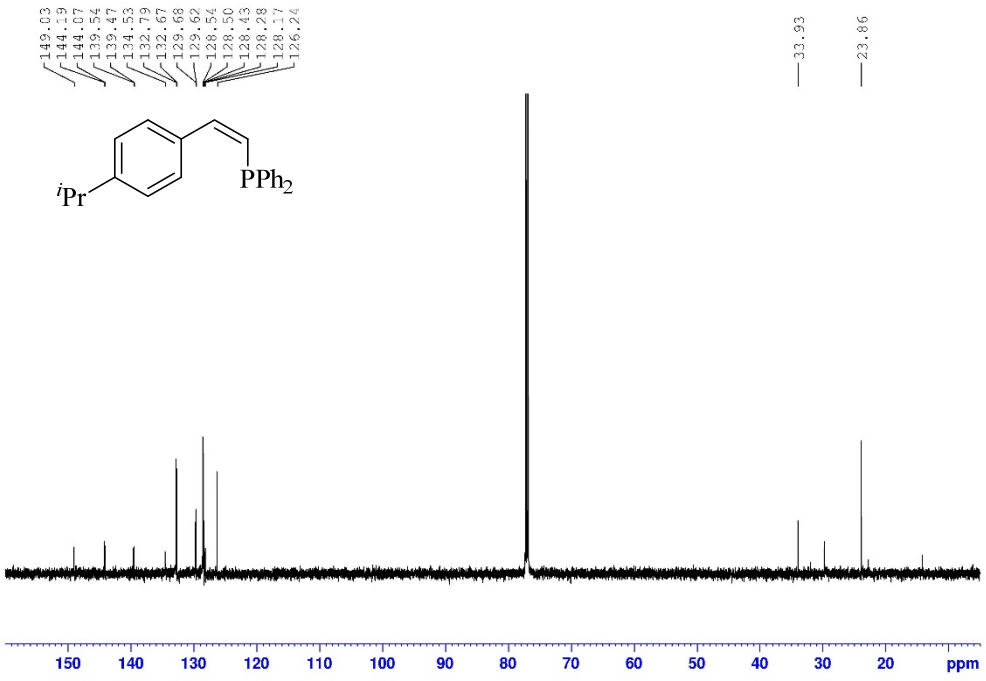


Figure S14. ^{13}C NMR spectrum of compound **2f**

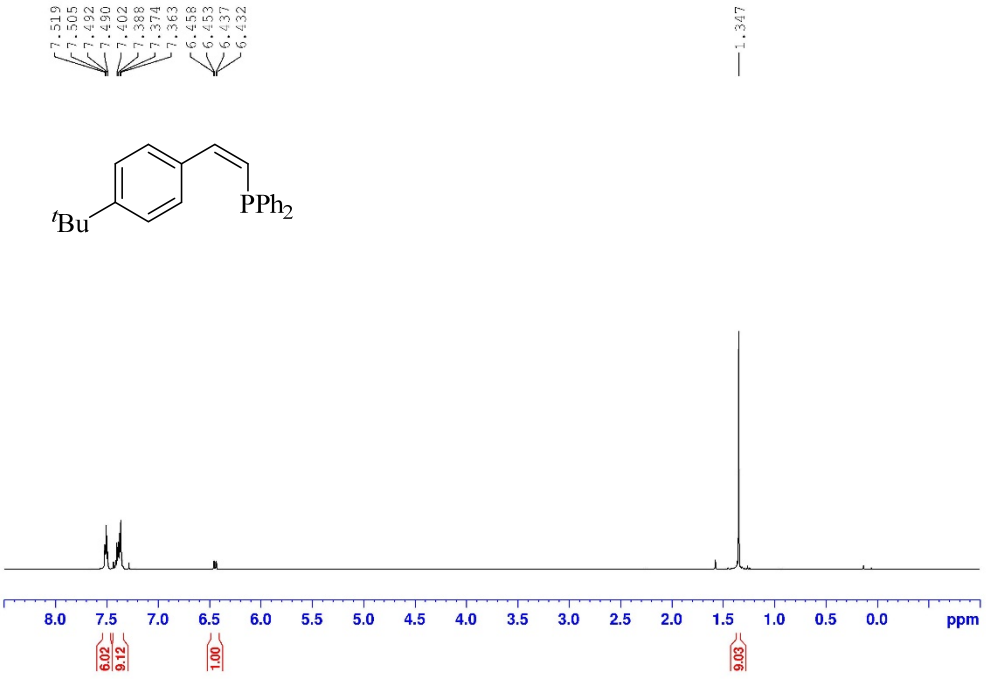


Figure S15. ^{1}H NMR spectrum of compound **2g**

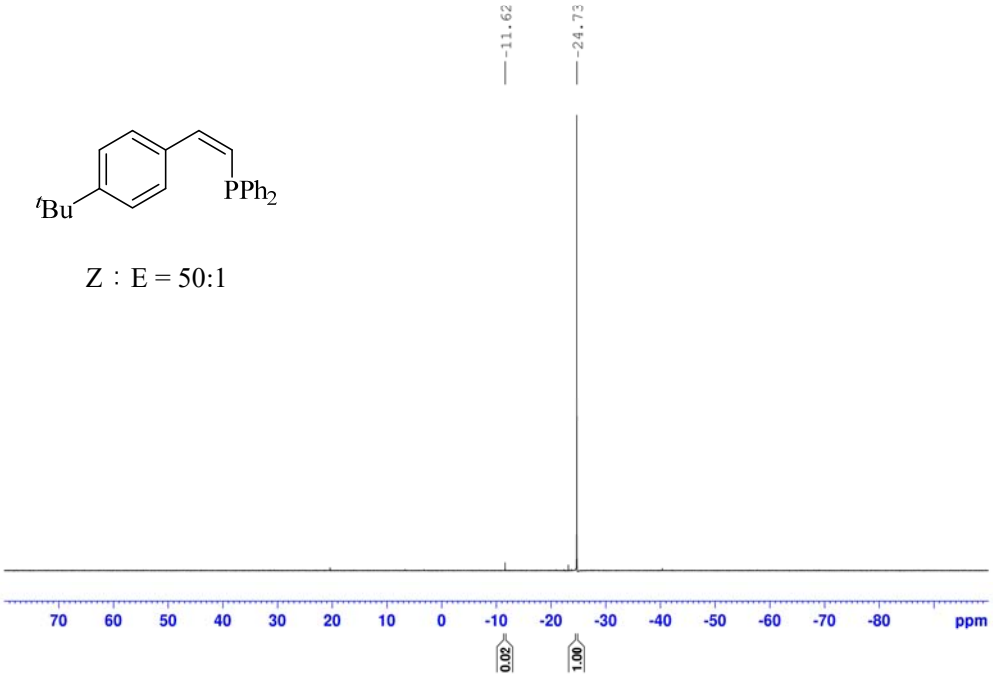


Figure S16. ^{31}P NMR spectrum of compound **2g**

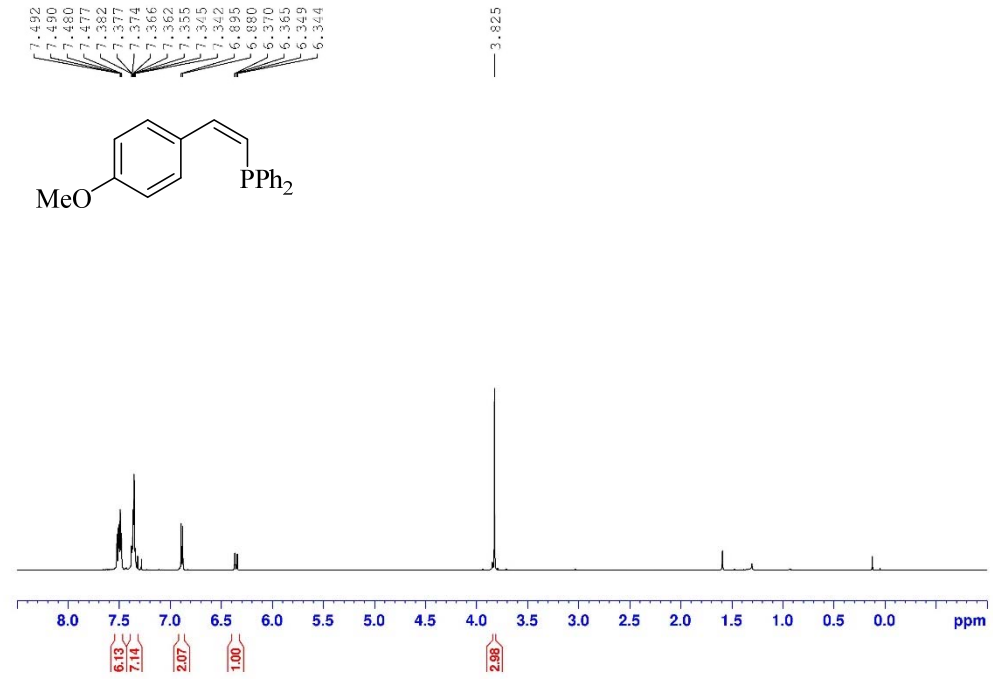


Figure S17. ^{1}H NMR spectrum of compound **2h**

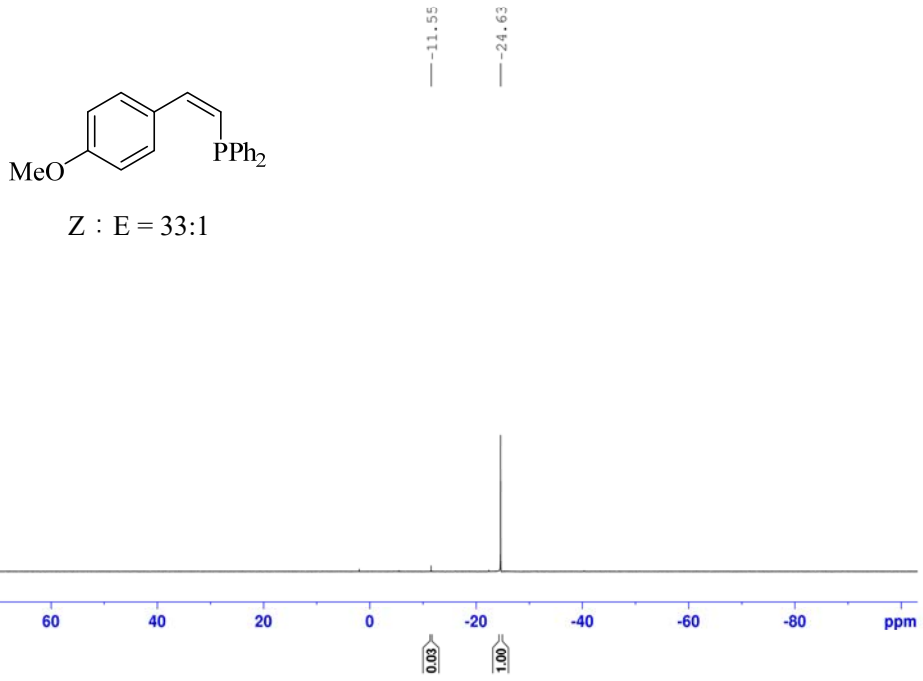


Figure S18. ^{31}P NMR spectrum of compound **2h**

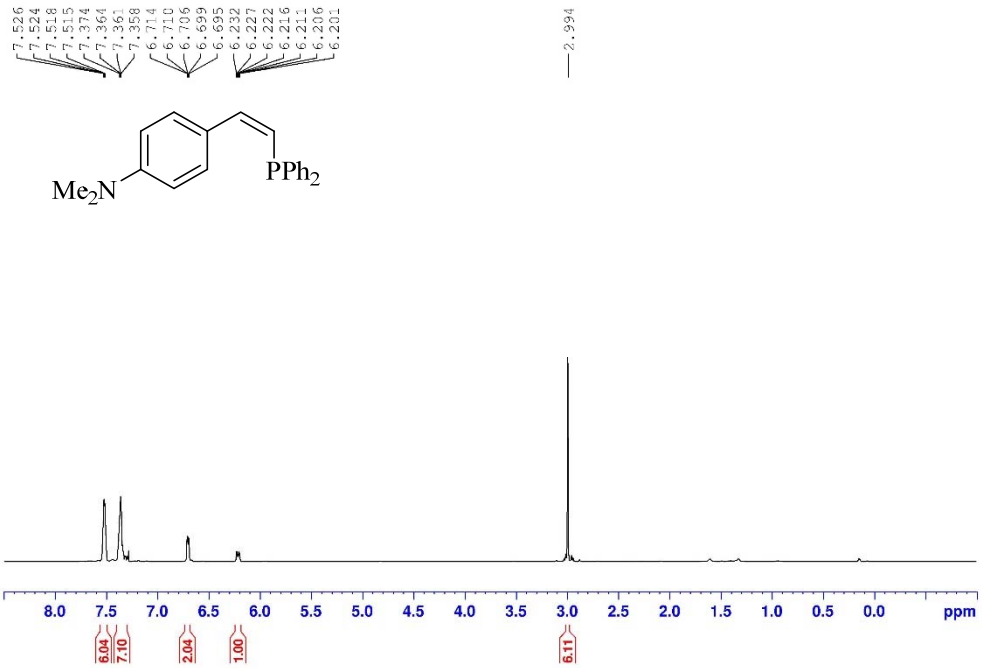


Figure S19. ^{1}H NMR spectrum of compound **2i**

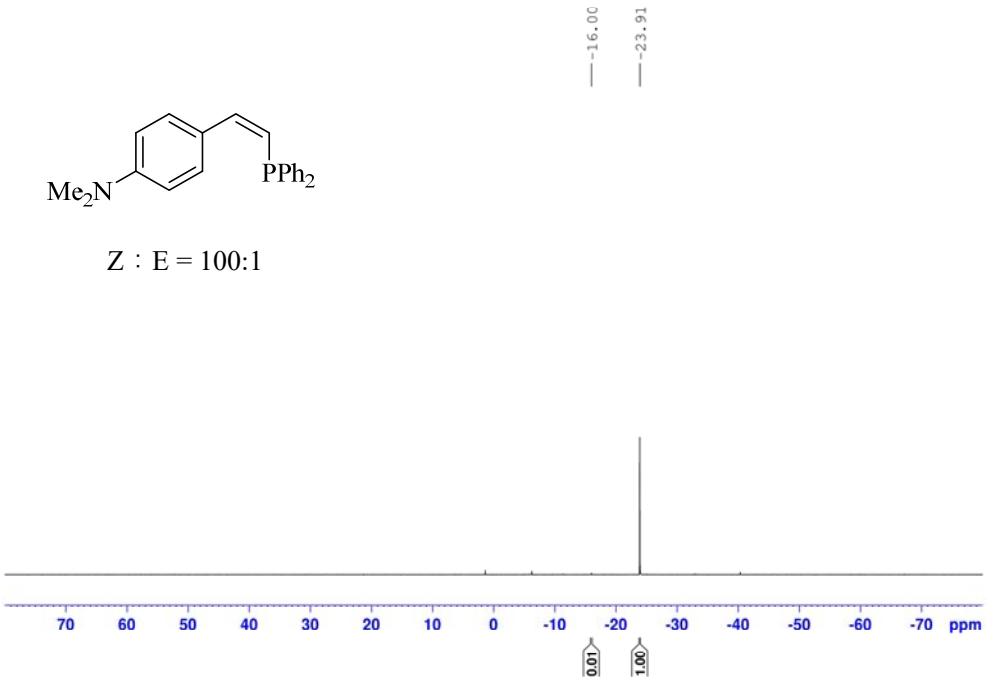


Figure S20. ^{31}P NMR spectrum of compound **2i**

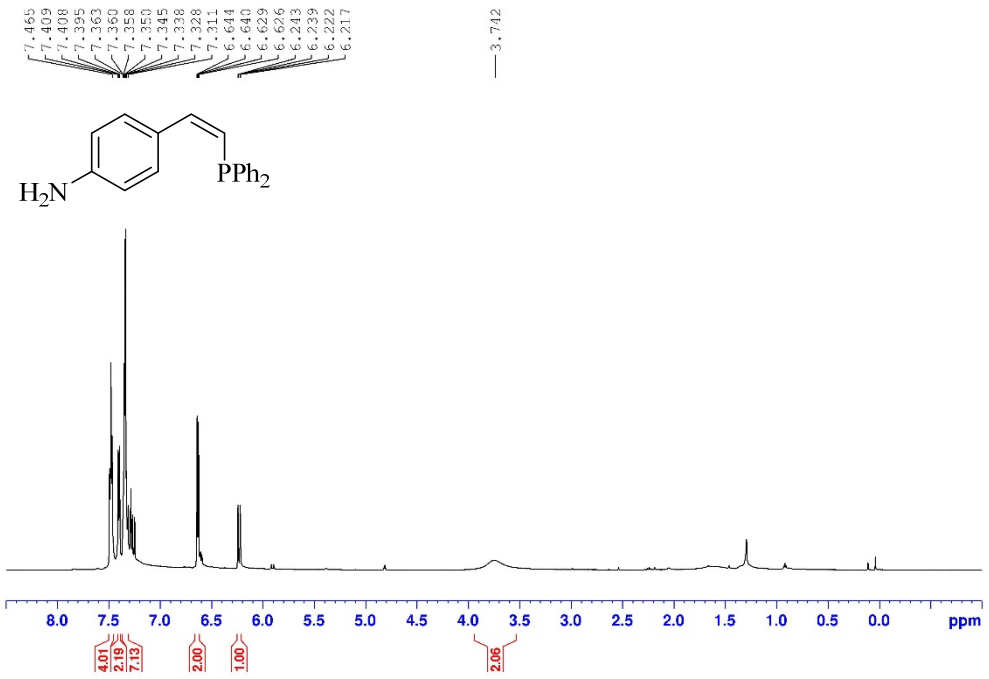


Figure S21. ^{1}H NMR spectrum of compound **2j**

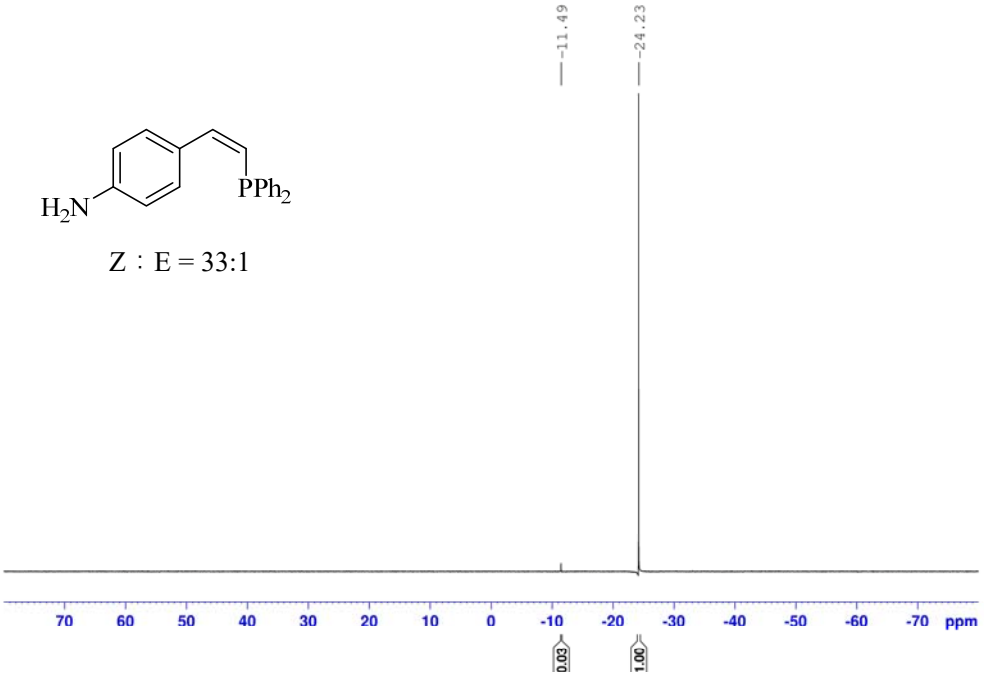


Figure S22. ^{31}P NMR spectrum of compound **2j**

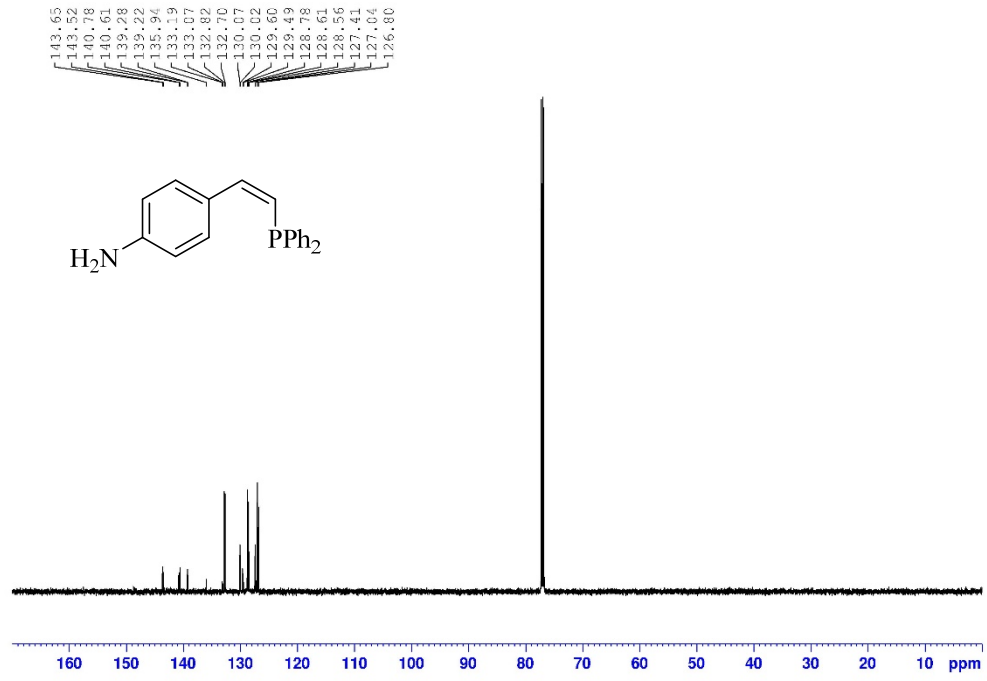


Figure S23. ^{13}C NMR spectrum of compound **2j**

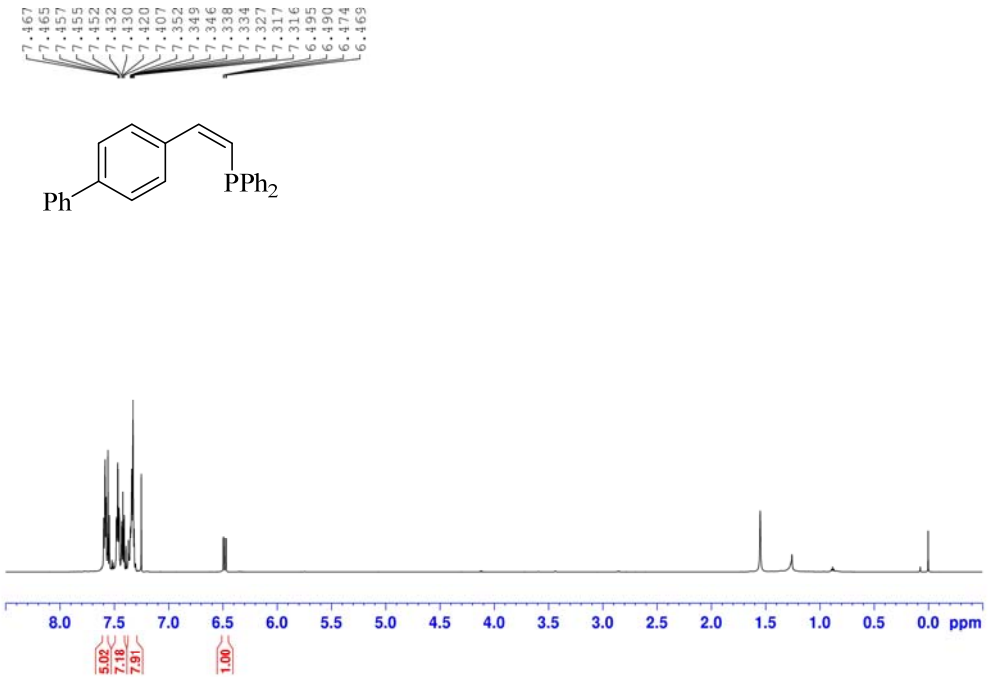


Figure S24. ^{1}H NMR spectrum of compound **2k**

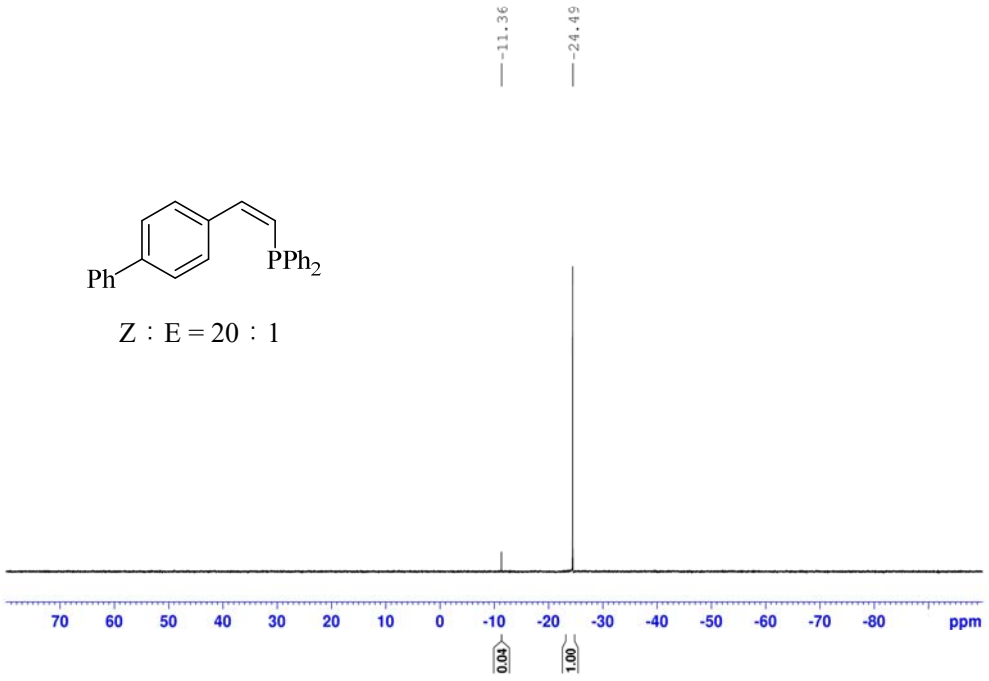


Figure S25. ^{31}P NMR spectrum of compound **2k**

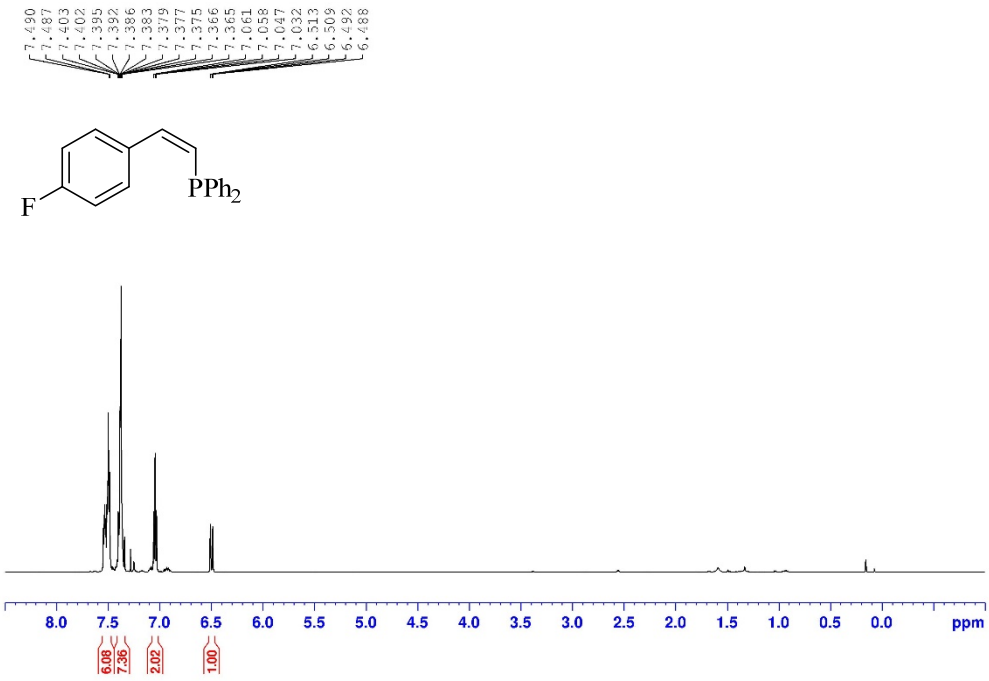
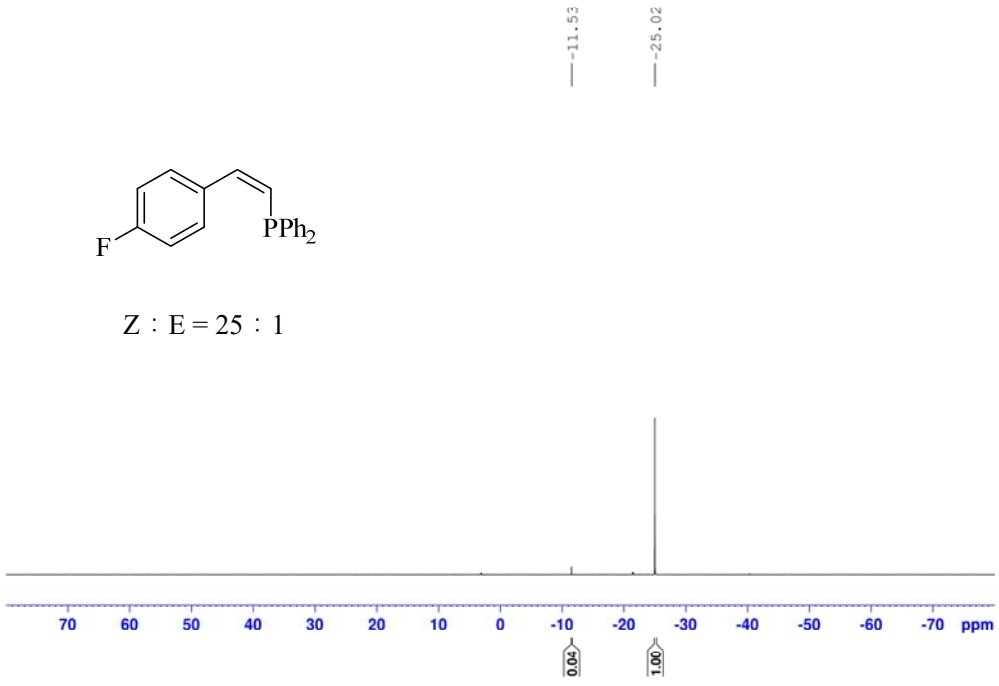


Figure S26. ^{1}H NMR spectrum of compound **2l**



Z : E = 25 : 1

Figure S27. ^{31}P NMR spectrum of compound **2l**

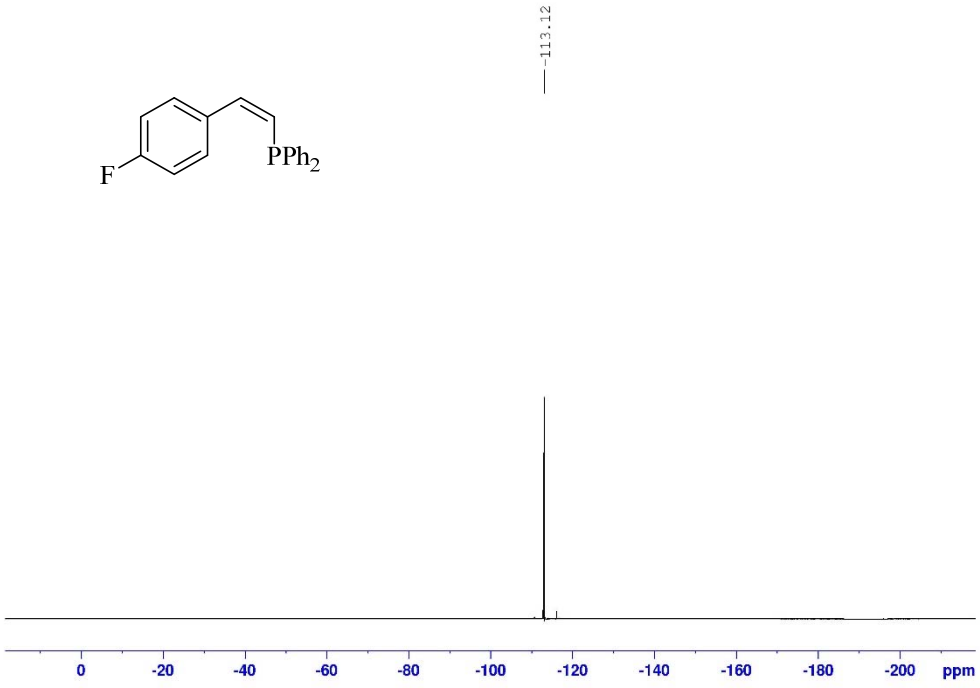


Figure S28. ^{19}F NMR spectrum of compound **2l**

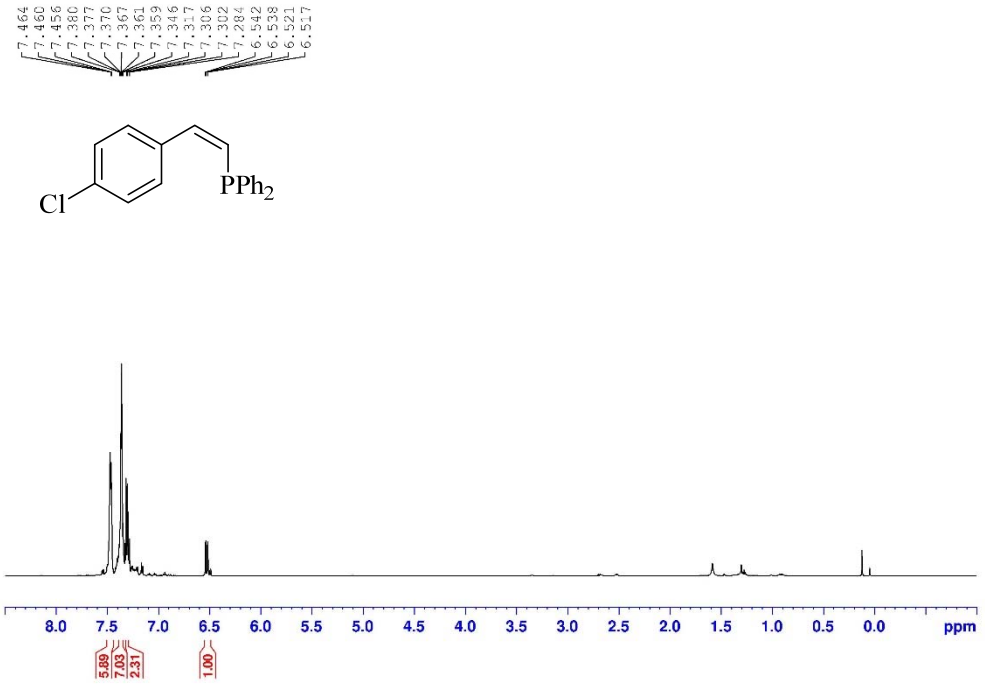


Figure S29. ^{1}H NMR spectrum of compound **2m**

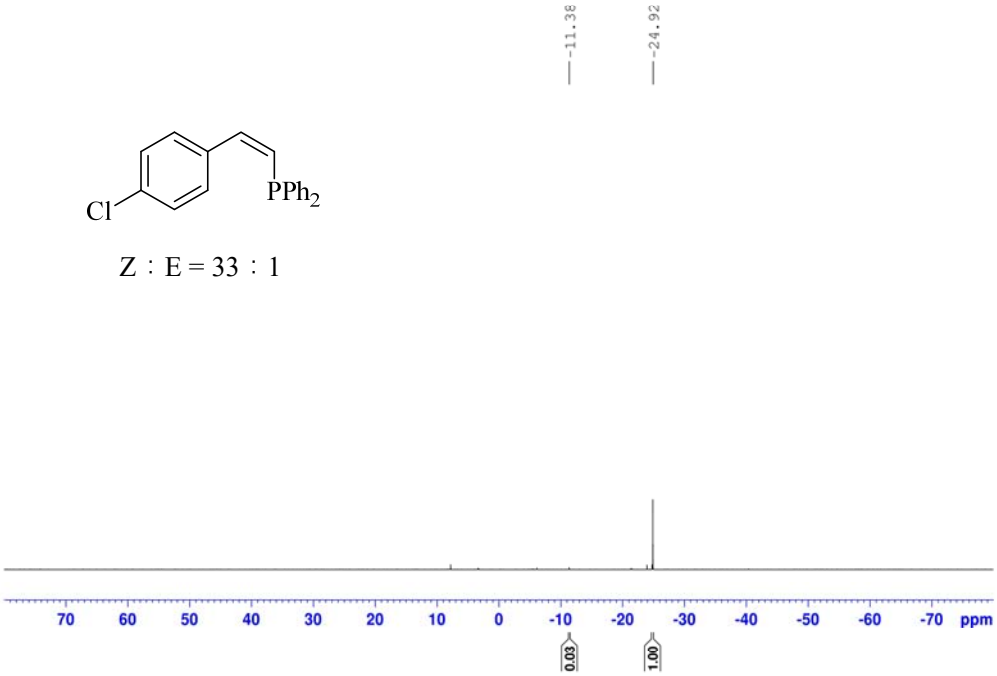


Figure S30. ^{31}P NMR spectrum of compound **2m**

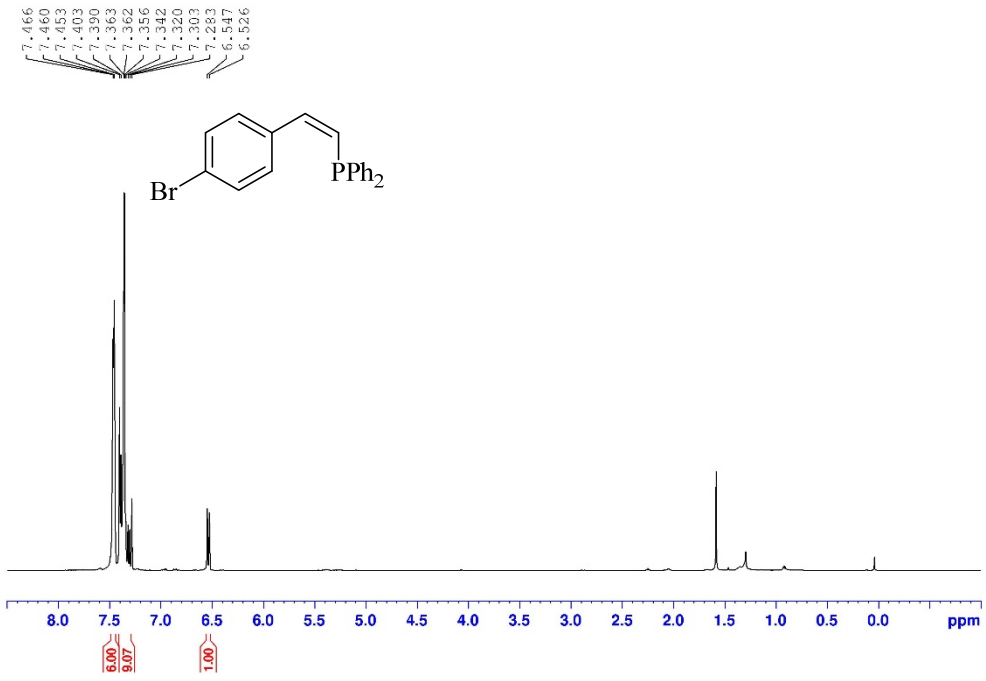


Figure S31. ^{1}H NMR spectrum of compound **2n**

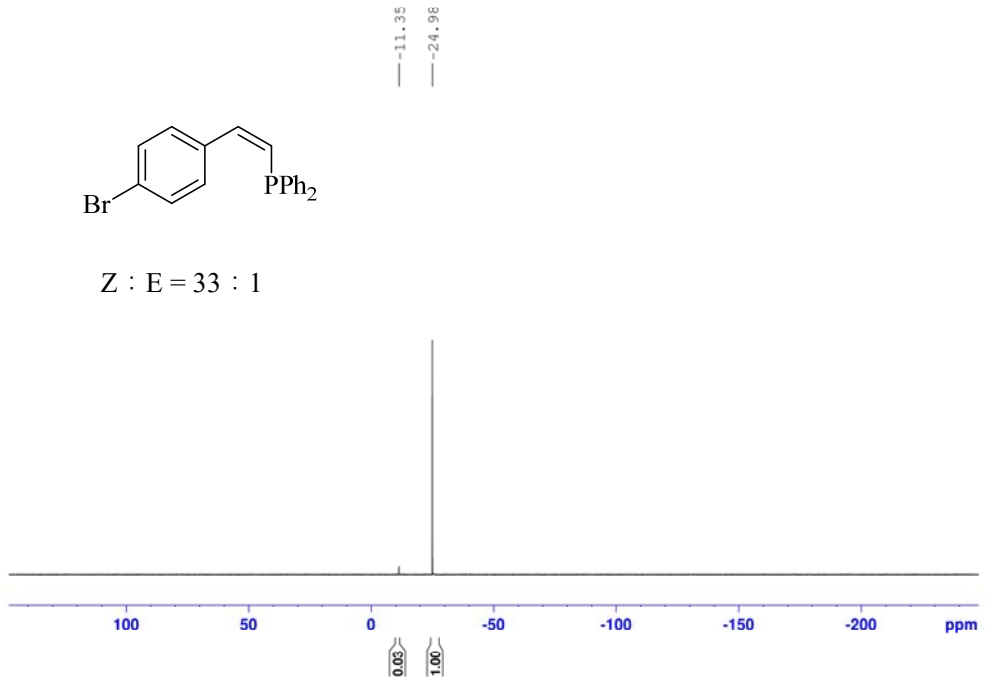


Figure S32. ^{31}P NMR spectrum of compound **2n**

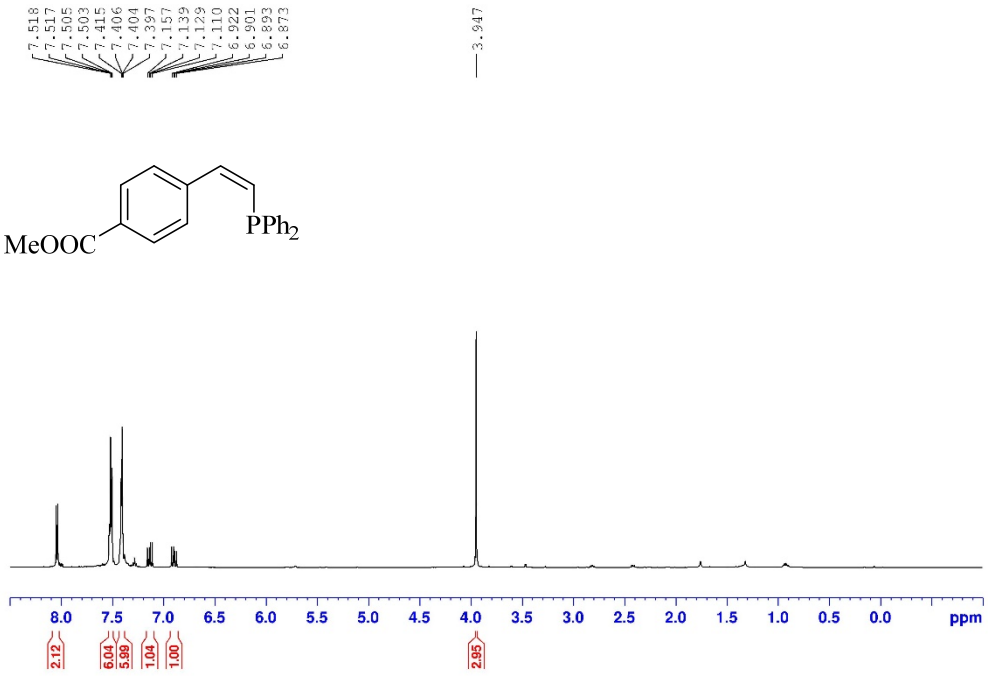


Figure S33. ^{1}H NMR spectrum of compound **2o**

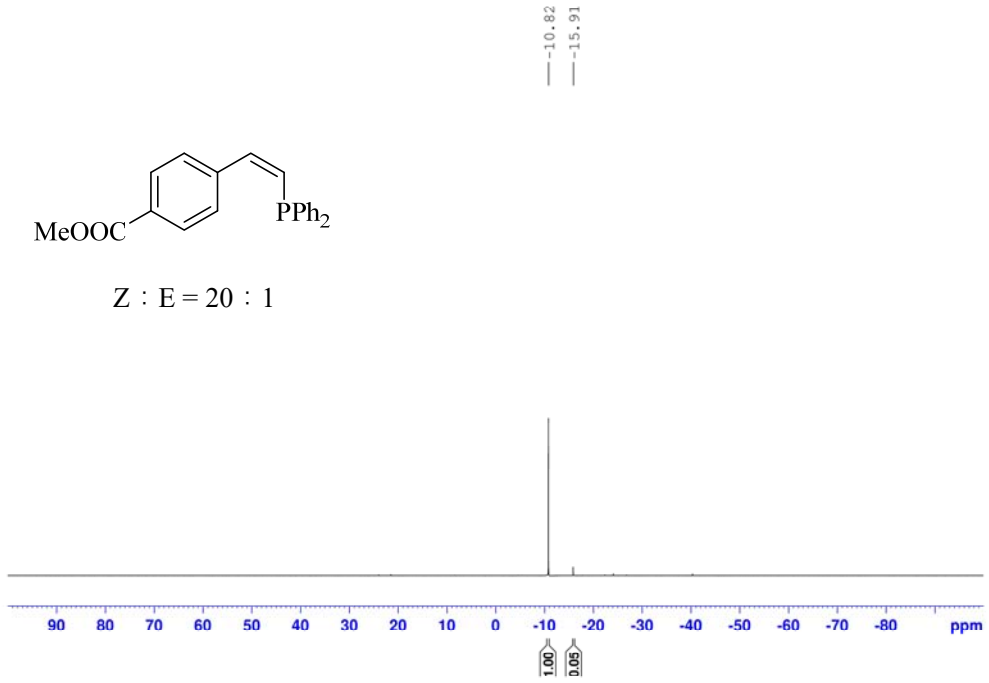


Figure S34. ^{31}P NMR spectrum of compound **2o**

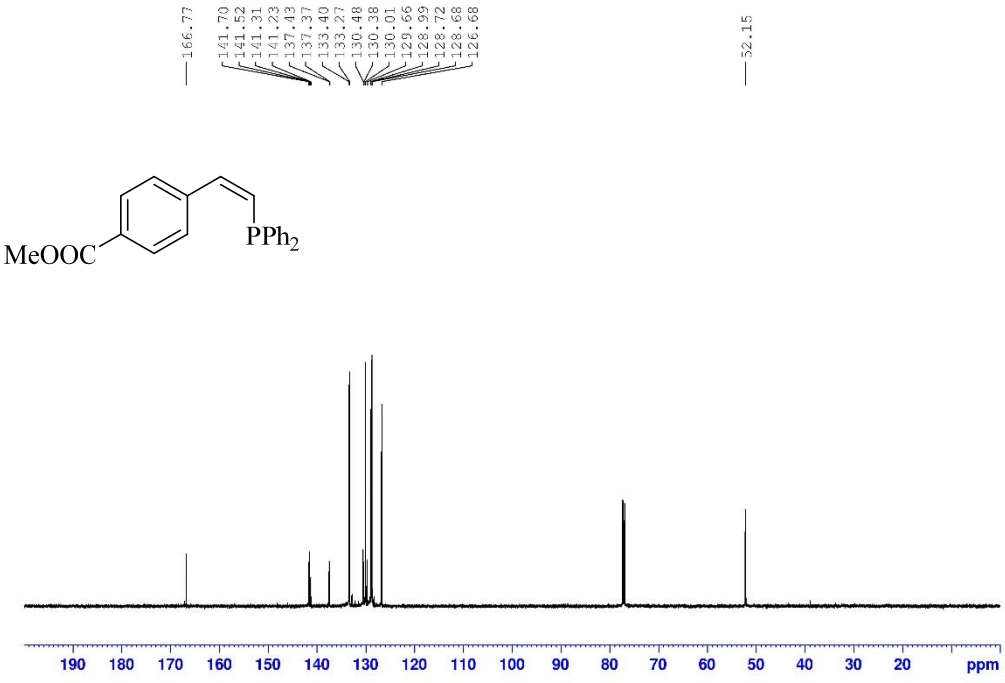


Figure S35. ^{13}C NMR spectrum of compound **2o**

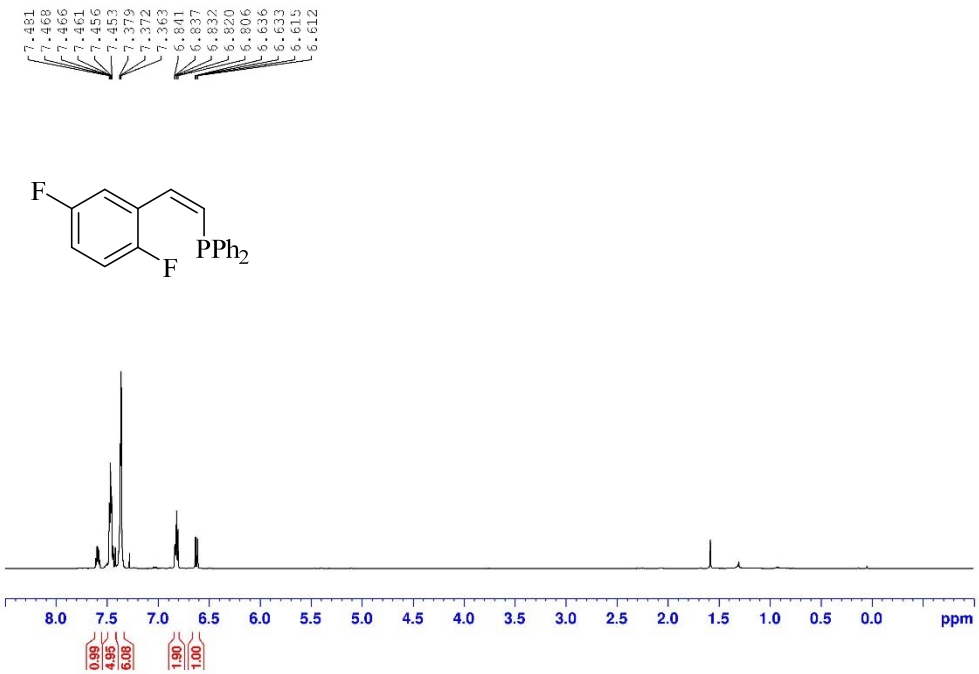
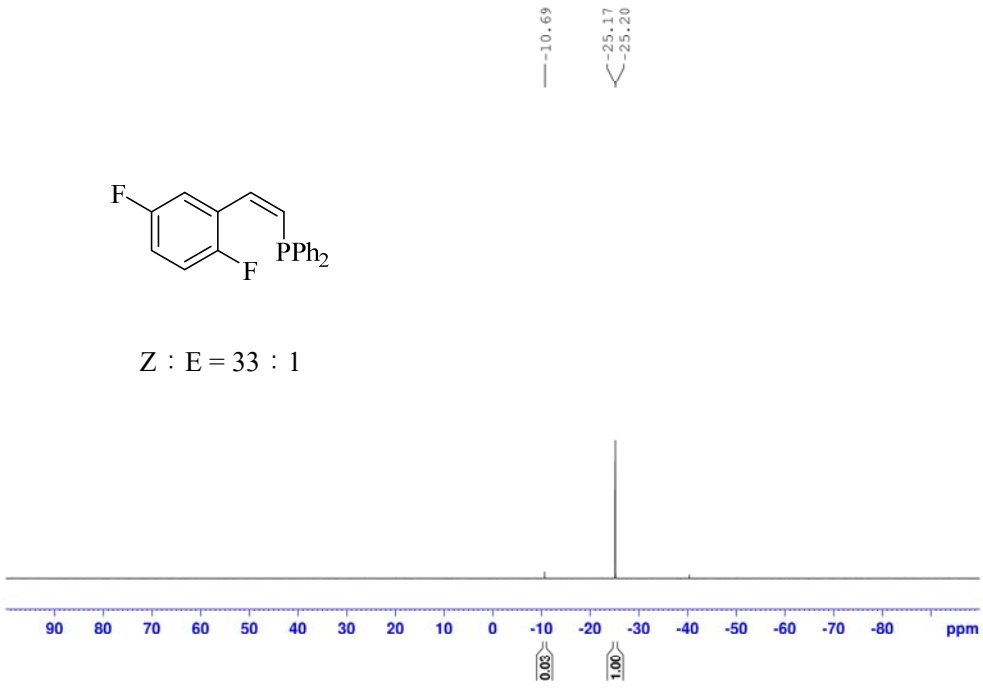


Figure S36. ^{1}H NMR spectrum of compound **2p**



Z : E = 33 : 1

Figure S37. ^{31}P NMR spectrum of compound **2p**

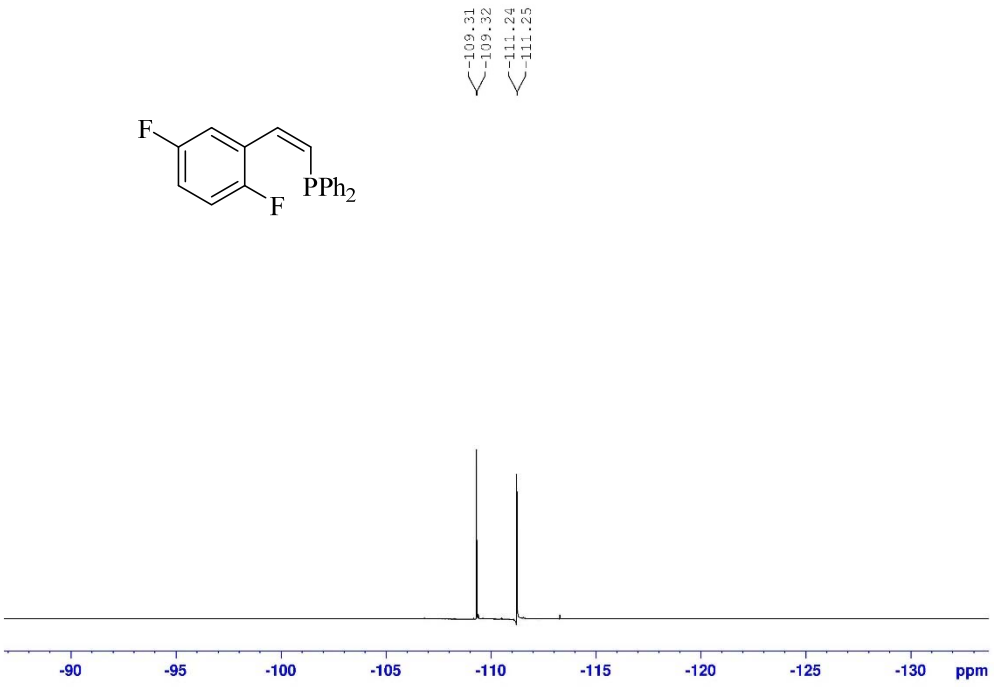


Figure S38. ^{19}F NMR spectrum of compound **2p**

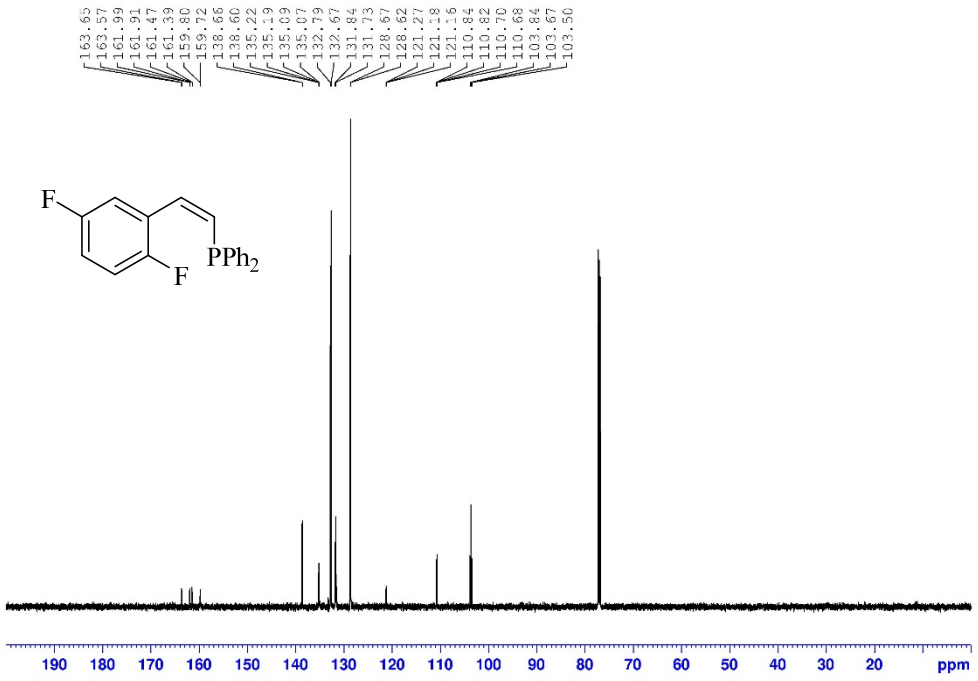


Figure S39. ^{13}C NMR spectrum of compound **2p**

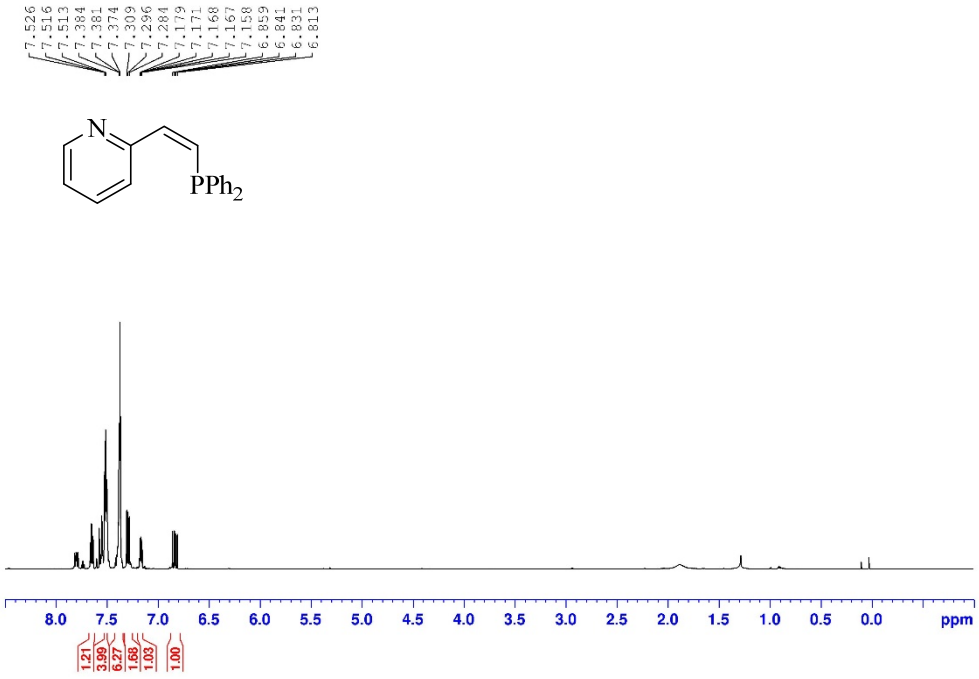
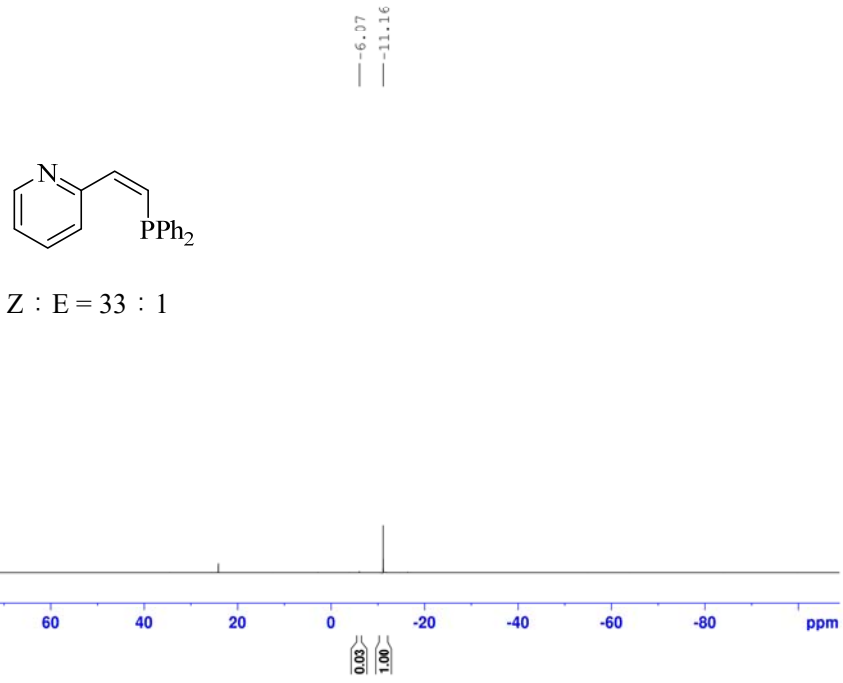


Figure S40. ^{1}H NMR spectrum of compound **2q**



Z：E = 33：1

Figure S41. ^{31}P NMR spectrum of compound **2q**

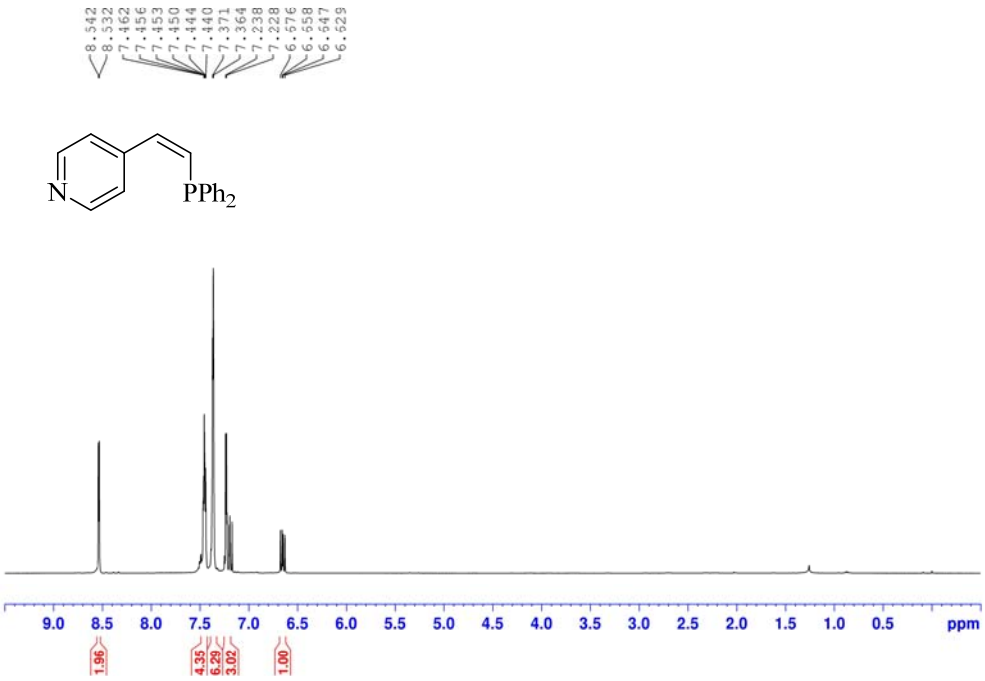


Figure S42. ^{1}H NMR spectrum of compound **2r**

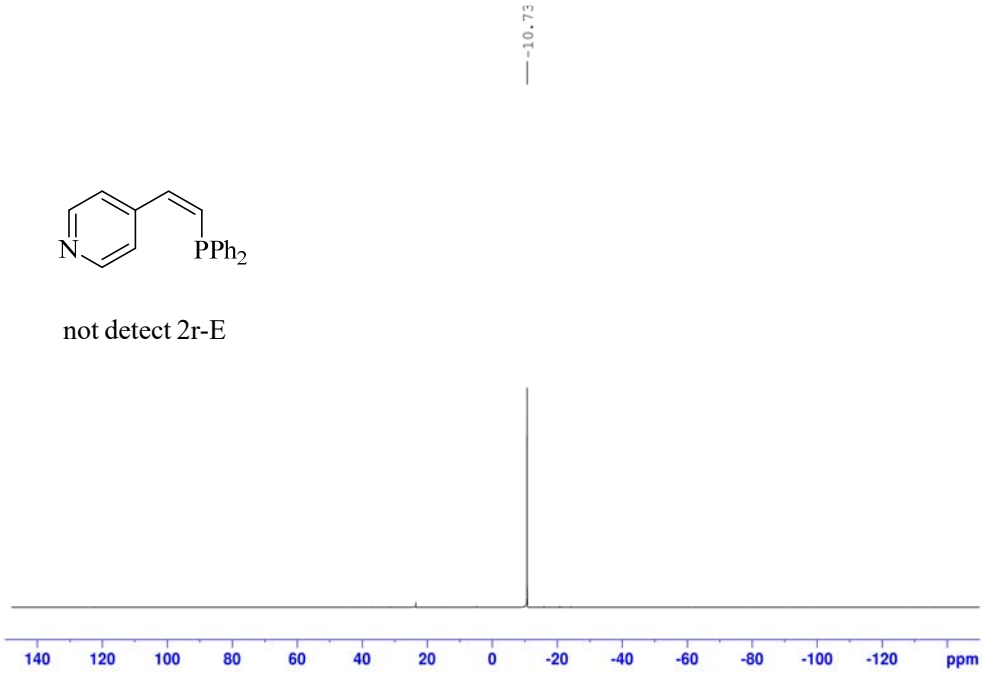


Figure S43. ^{31}P NMR spectrum of compound **2r**

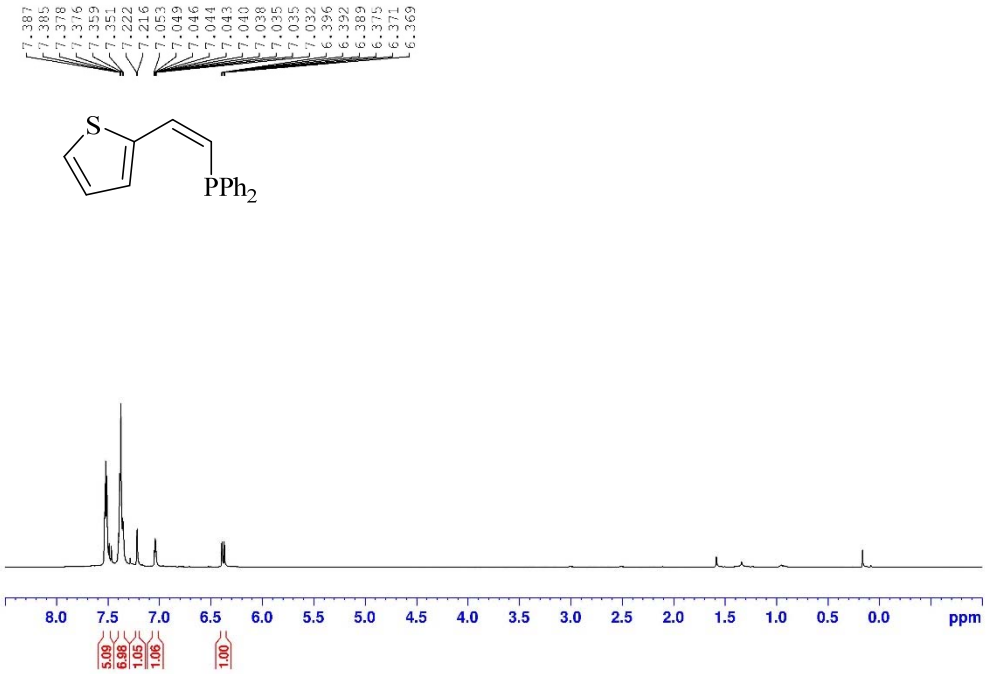


Figure S44. ^{1}H NMR spectrum of compound **2s**

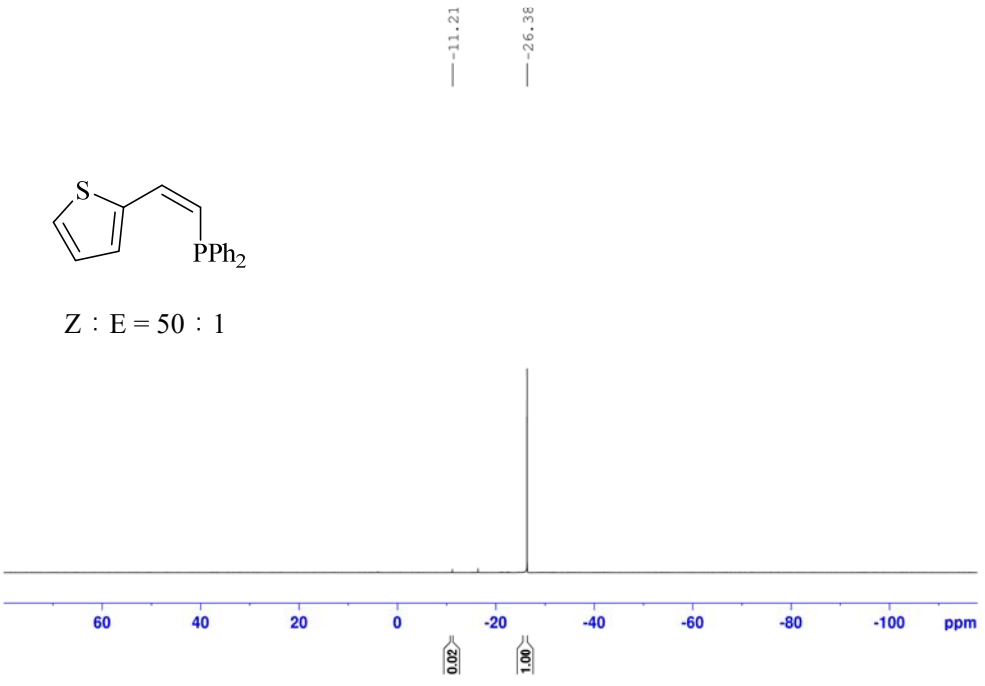


Figure S45. ^{31}P NMR spectrum of compound **2s**

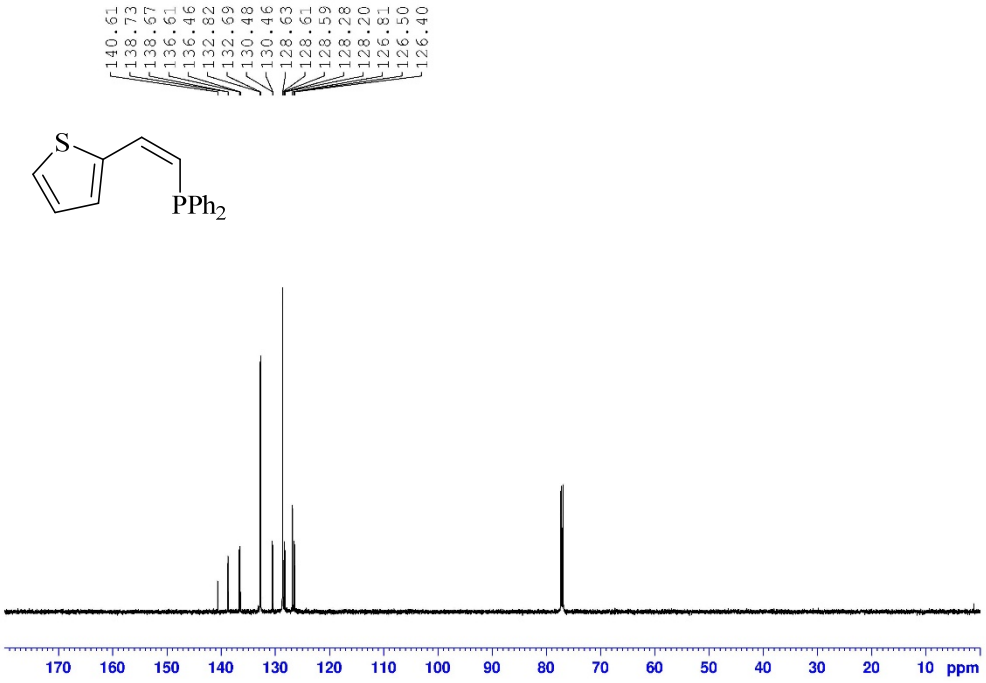


Figure S46. ^{13}C NMR spectrum of compound **2s**

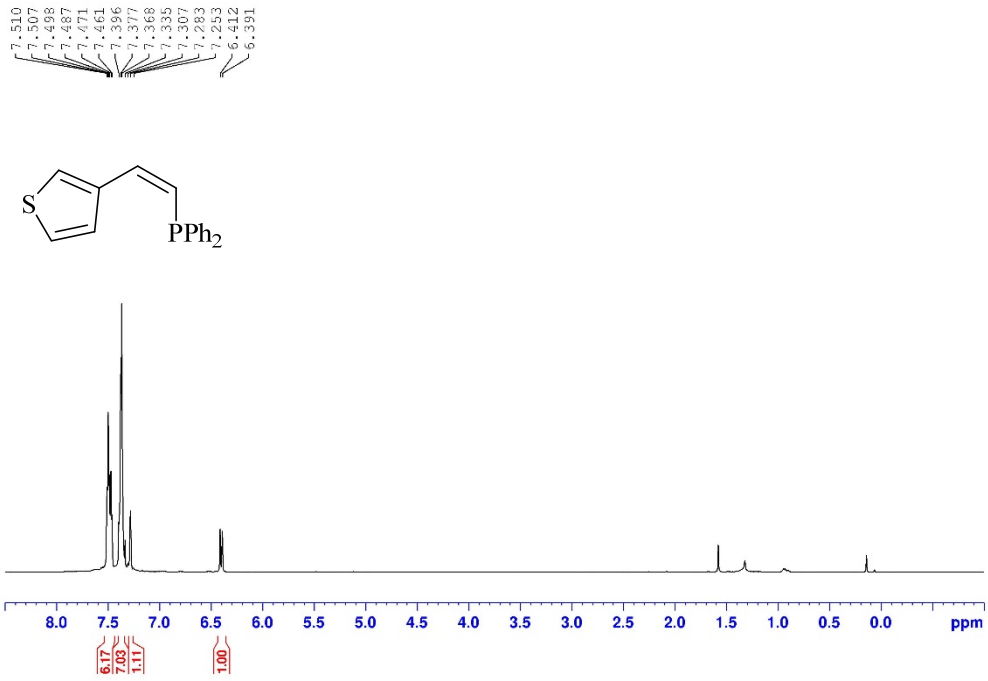


Figure S47. ^{1}H NMR spectrum of compound **2t**

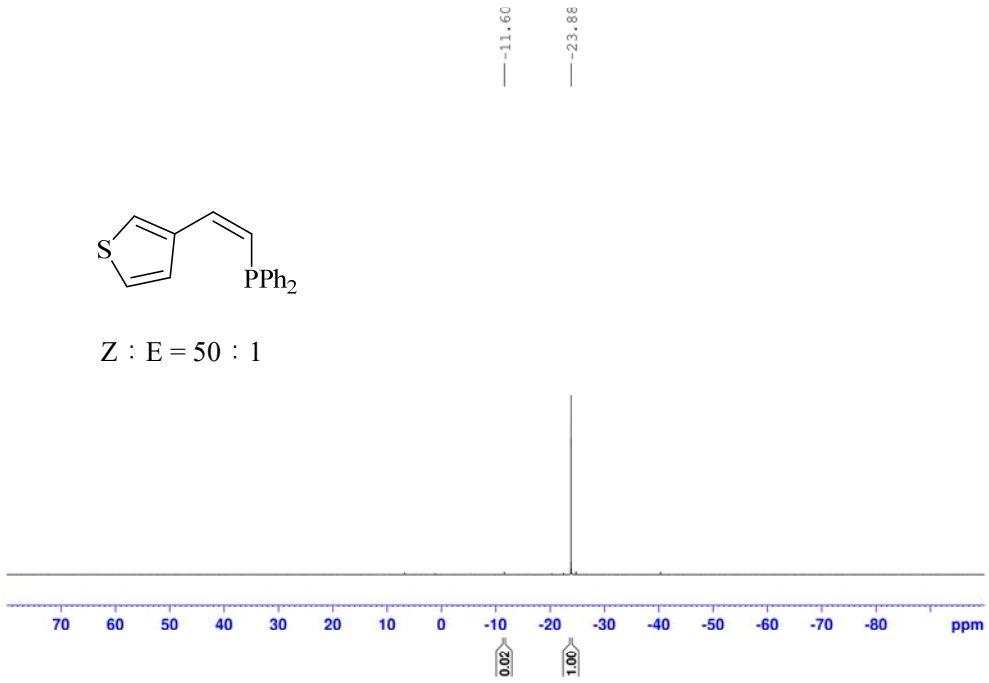


Figure S48. ^{31}P NMR spectrum of compound **2t**

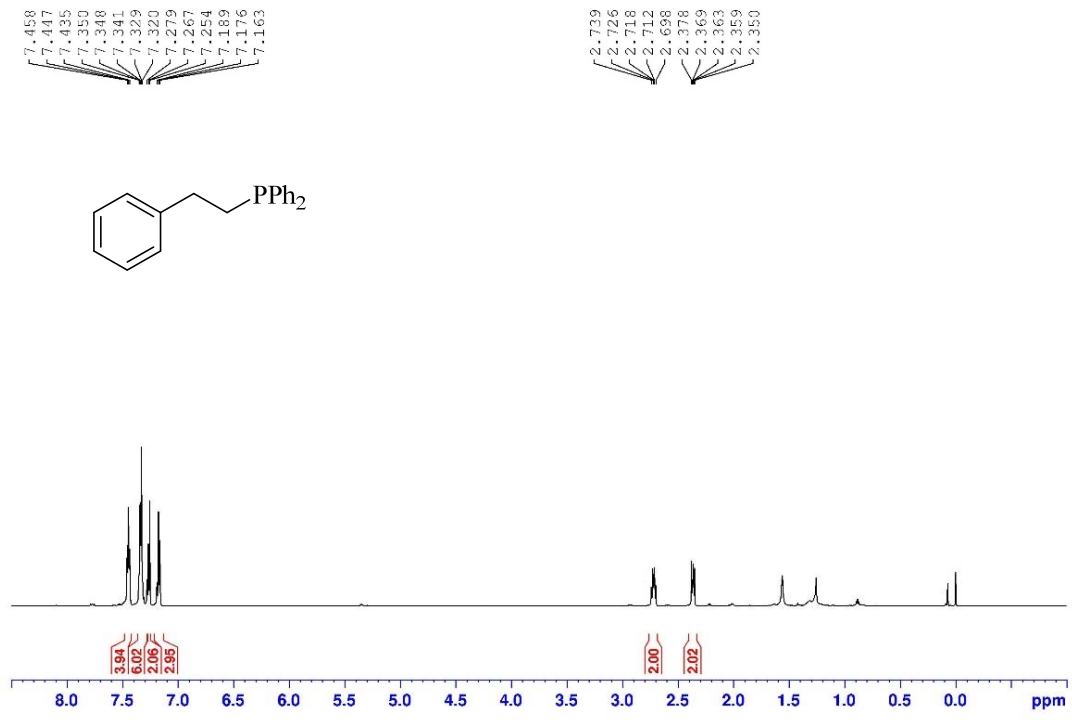


Figure S49. ^{1}H NMR spectrum of compound **4a**

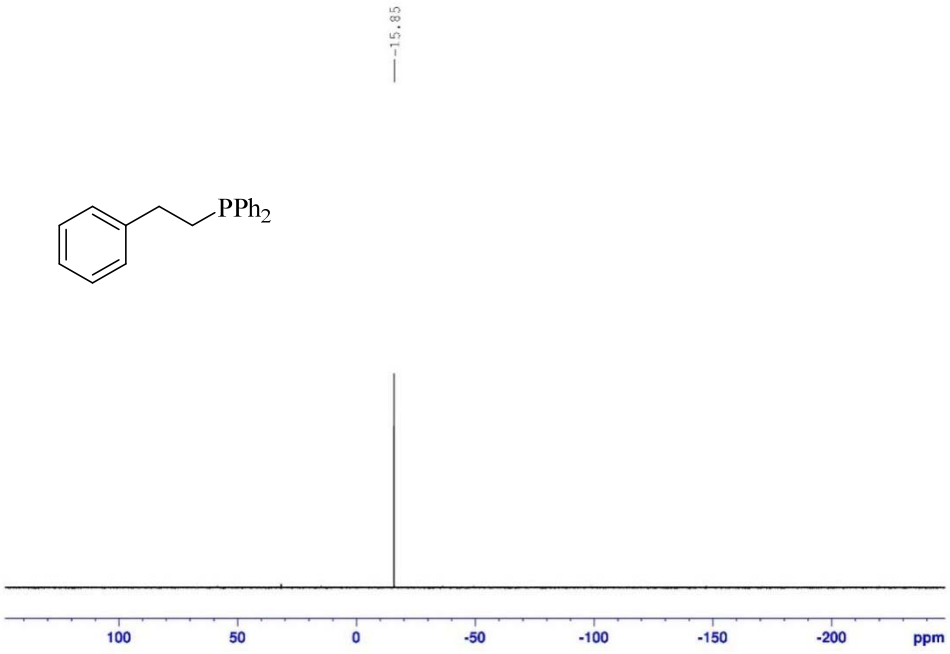


Figure S50. ^{31}P NMR spectrum of compound **4a**

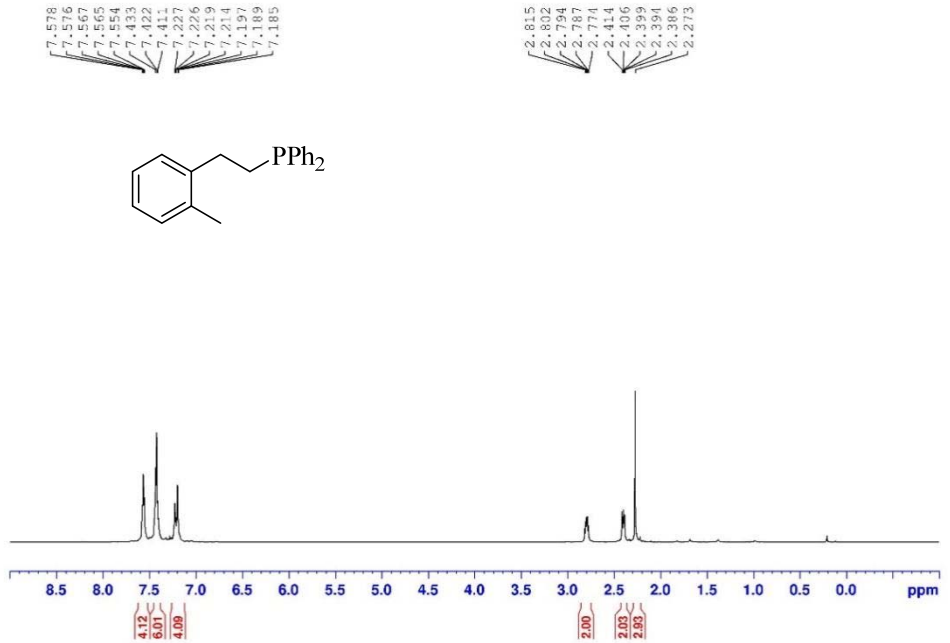


Figure S51. ^{1}H NMR spectrum of compound **4b**

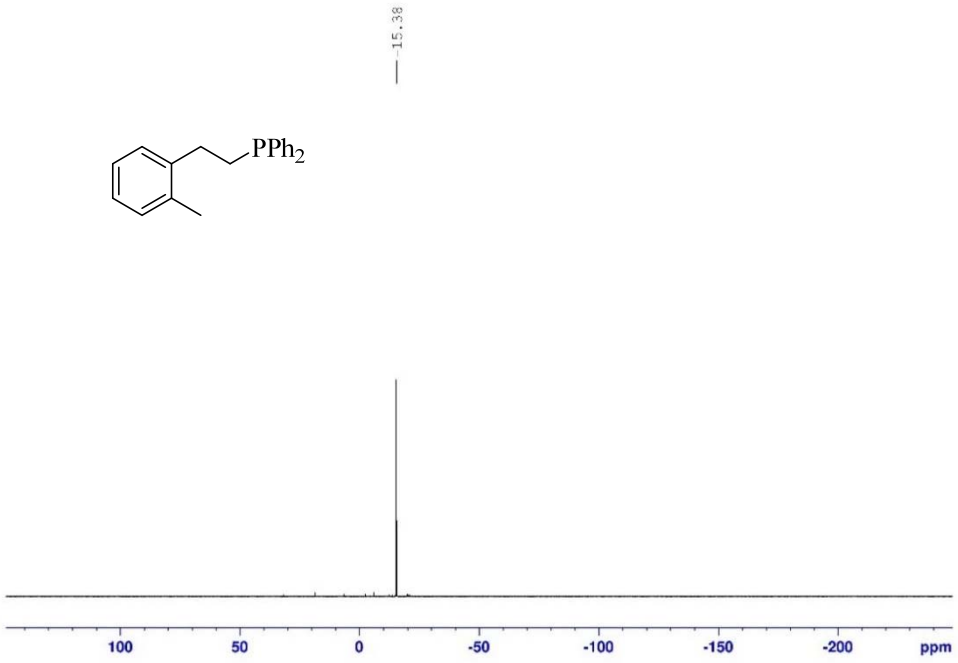


Figure S52. ^{31}P NMR spectrum of compound **4b**

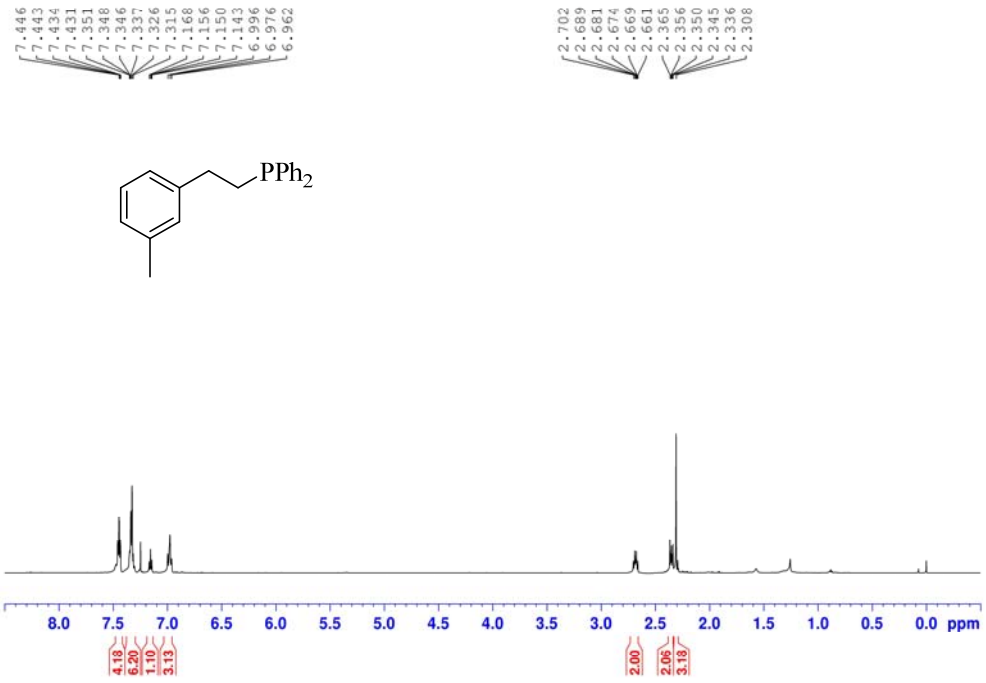


Figure S53. ^{1}H NMR spectrum of compound **4c**

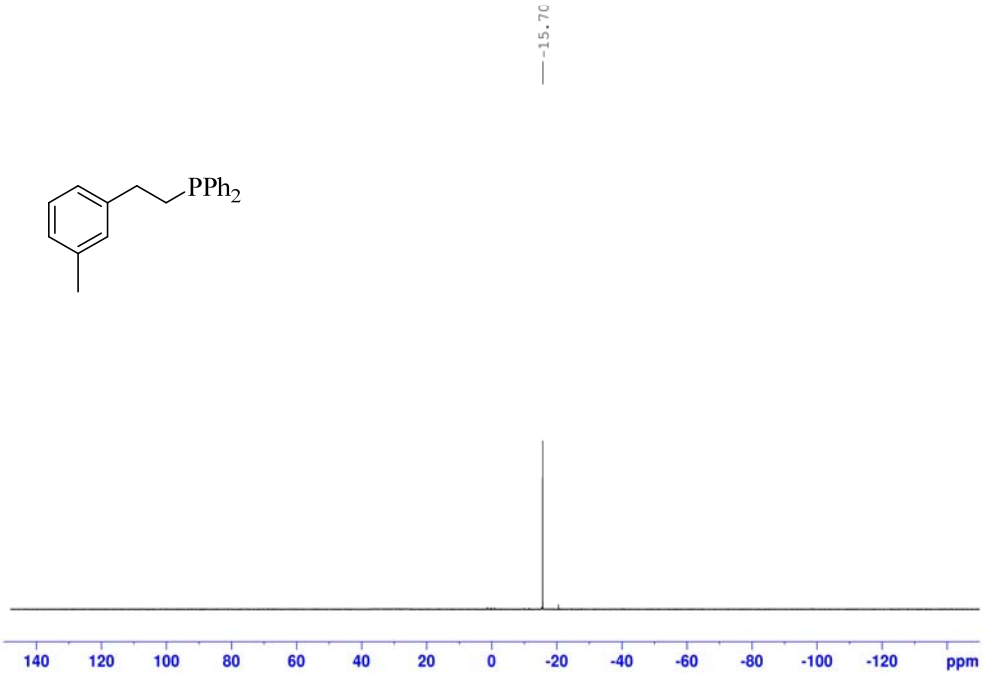


Figure S54. ^{31}P NMR spectrum of compound **4c**

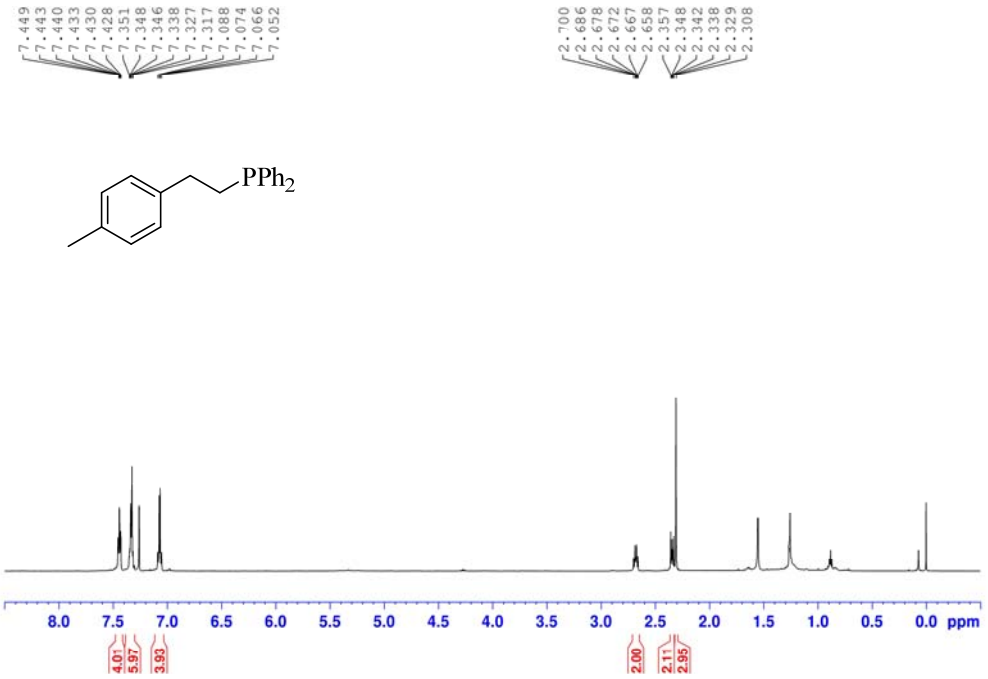


Figure S55. ^{1}H NMR spectrum of compound **4d**

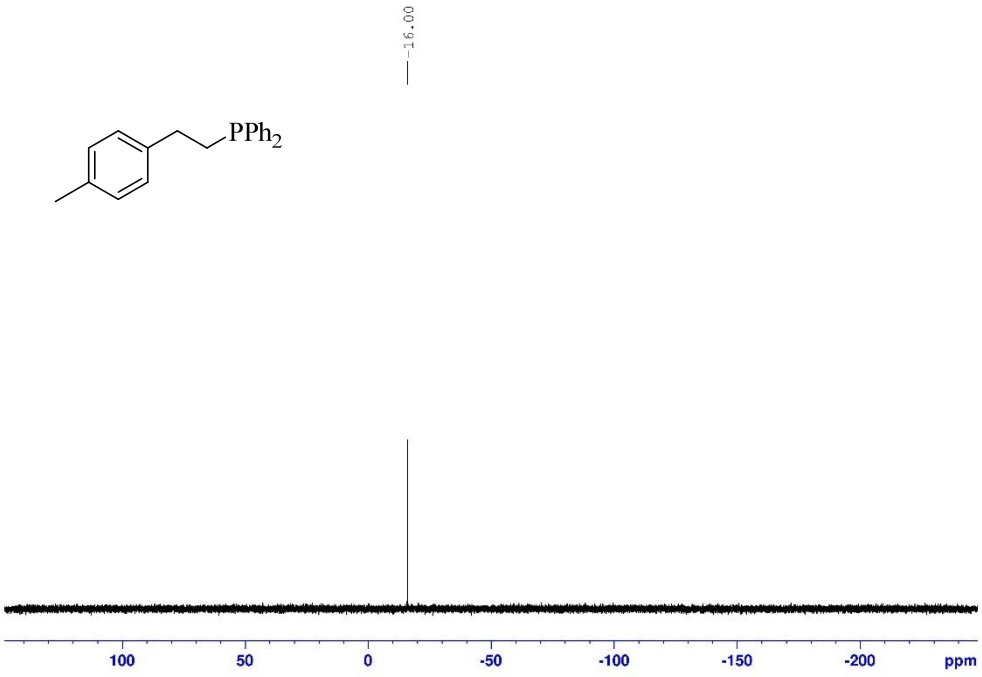


Figure S56. ^{31}P NMR spectrum of compound **4d**

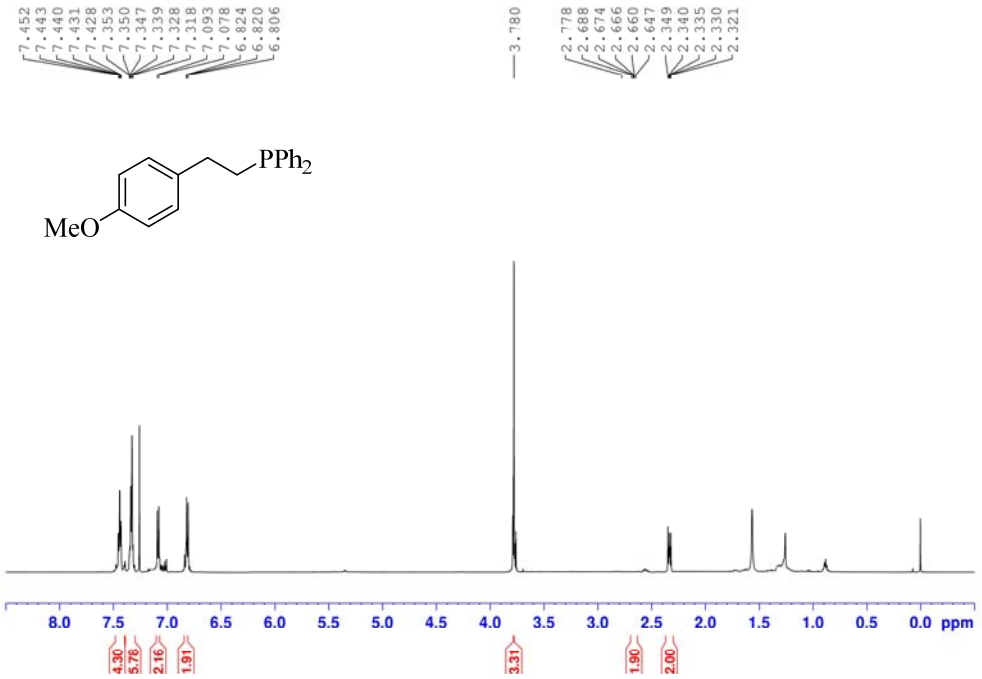


Figure S57. ^{1}H NMR spectrum of compound **4e**

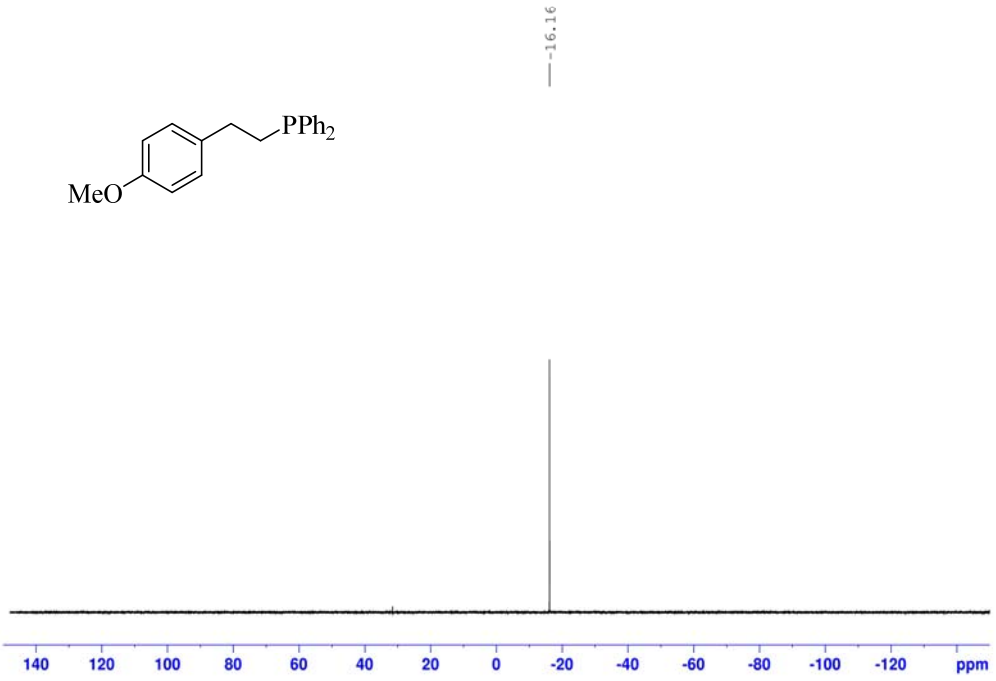


Figure S58. ^{31}P NMR spectrum of compound **4e**

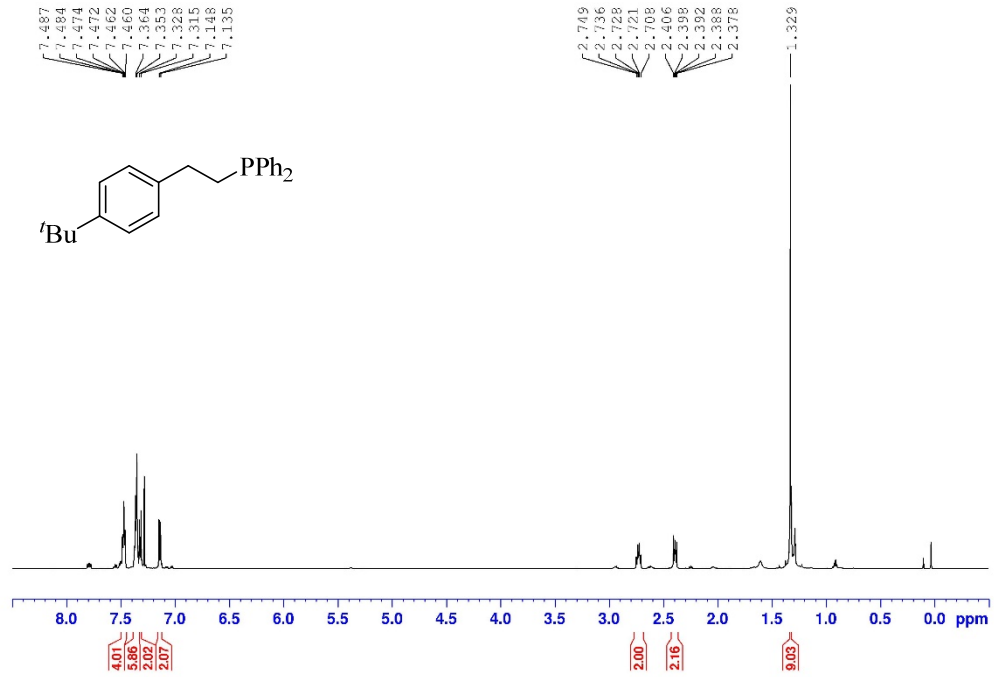


Figure S59. ^{1}H NMR spectrum of compound **4f**

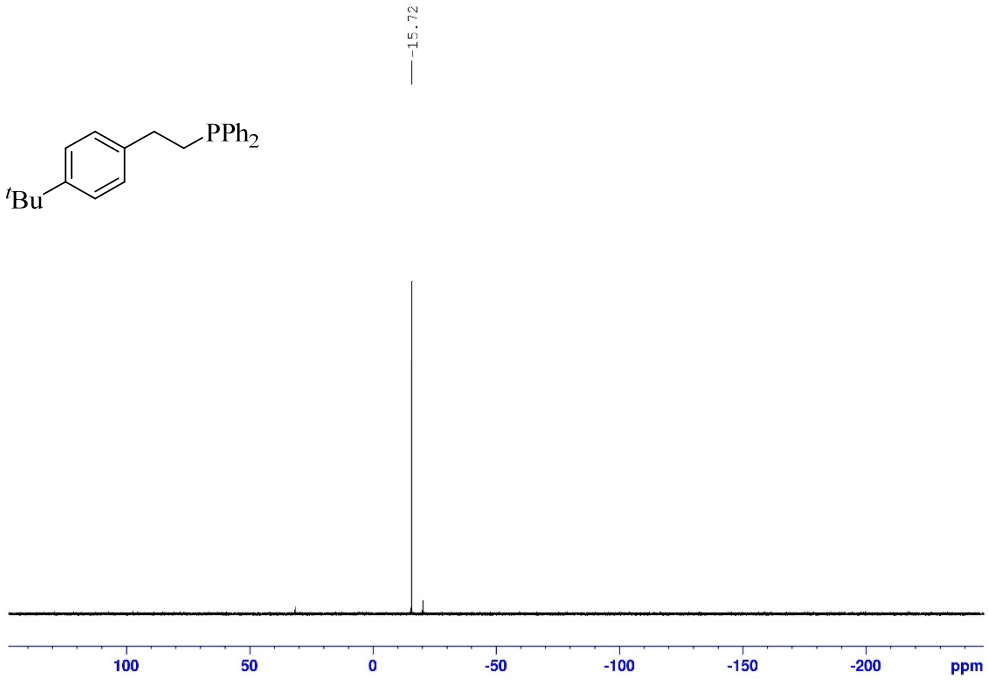


Figure S60. ^{31}P NMR spectrum of compound **4f**

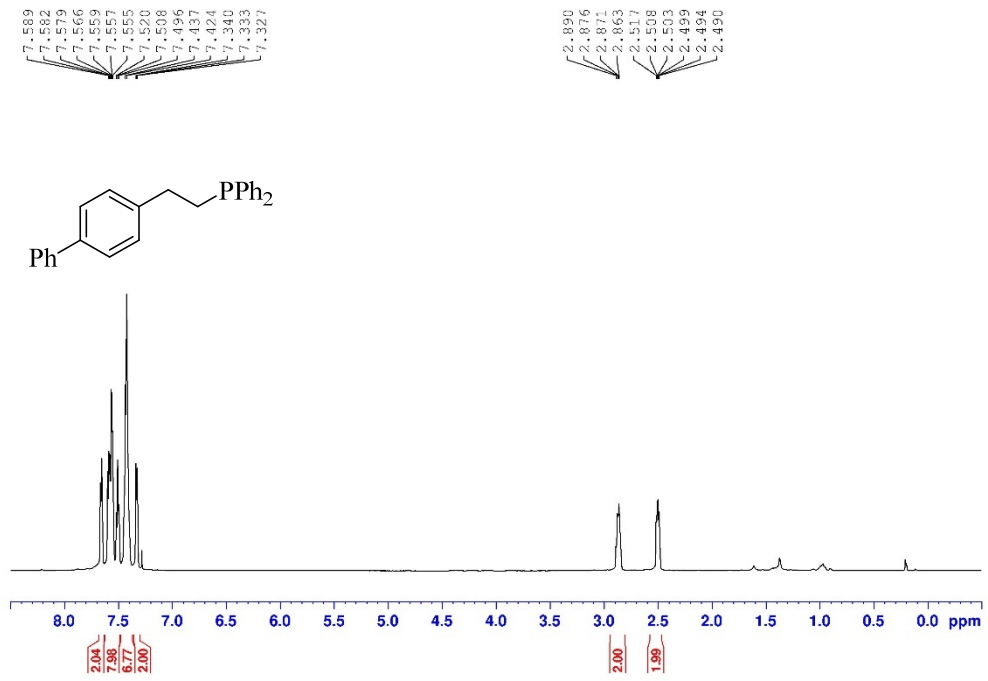


Figure S61. ^{1}H NMR spectrum of compound **4g**

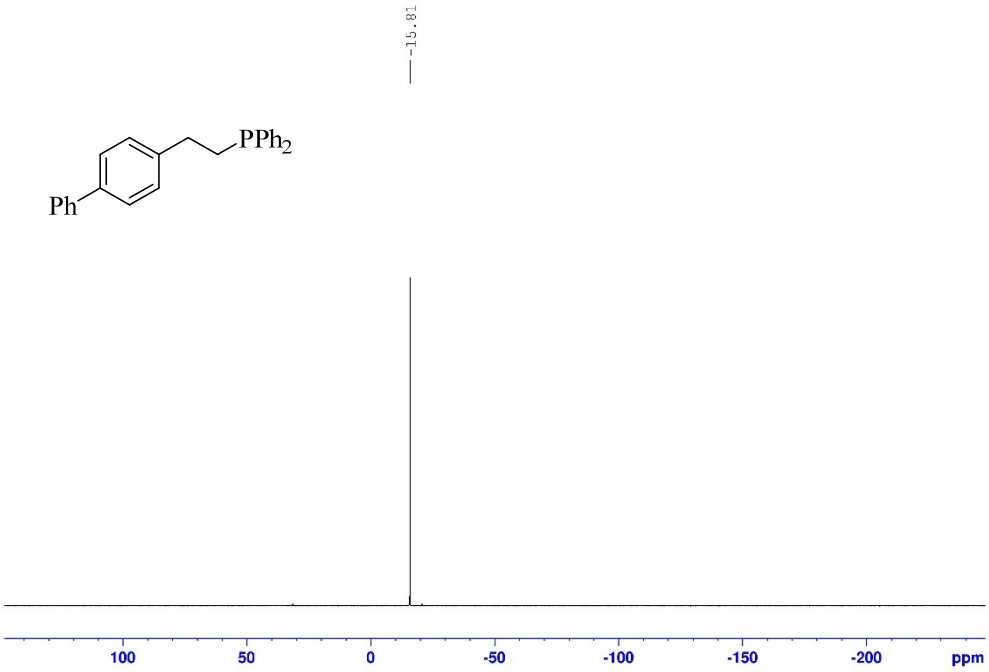


Figure S62. ^{31}P NMR spectrum of compound **4g**

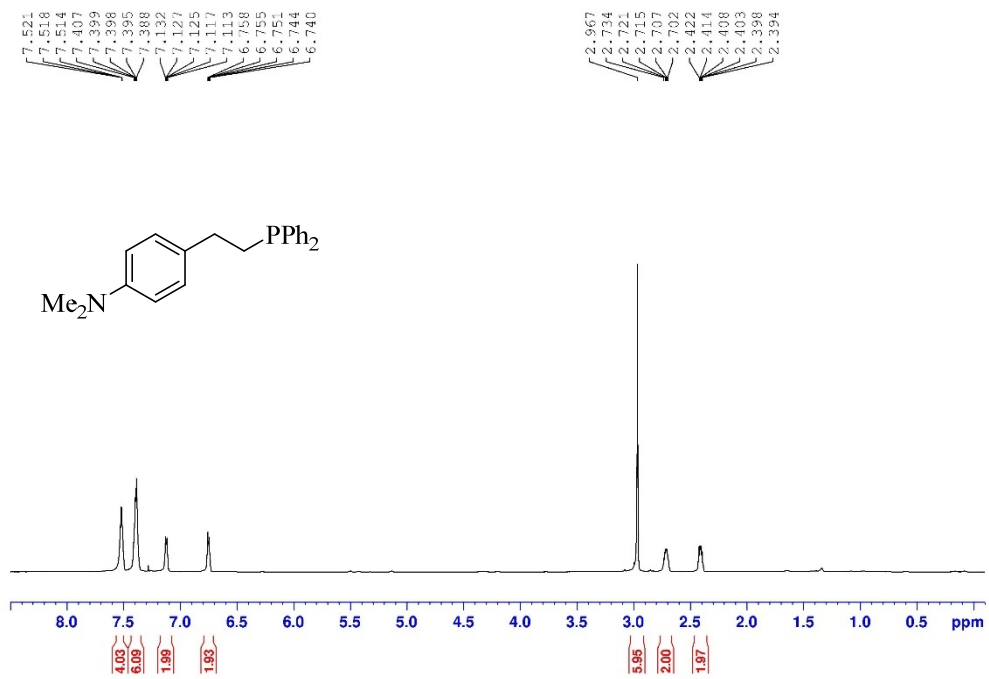


Figure S63. ^{1}H NMR spectrum of compound **4h**

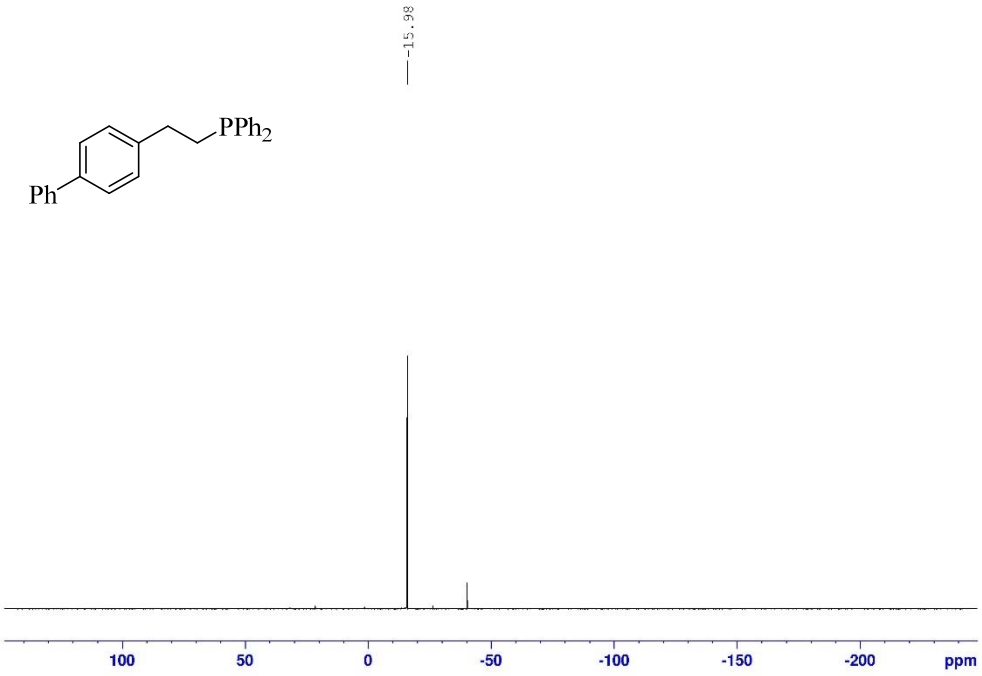


Figure S64. ^{31}P NMR spectrum of compound **4h**

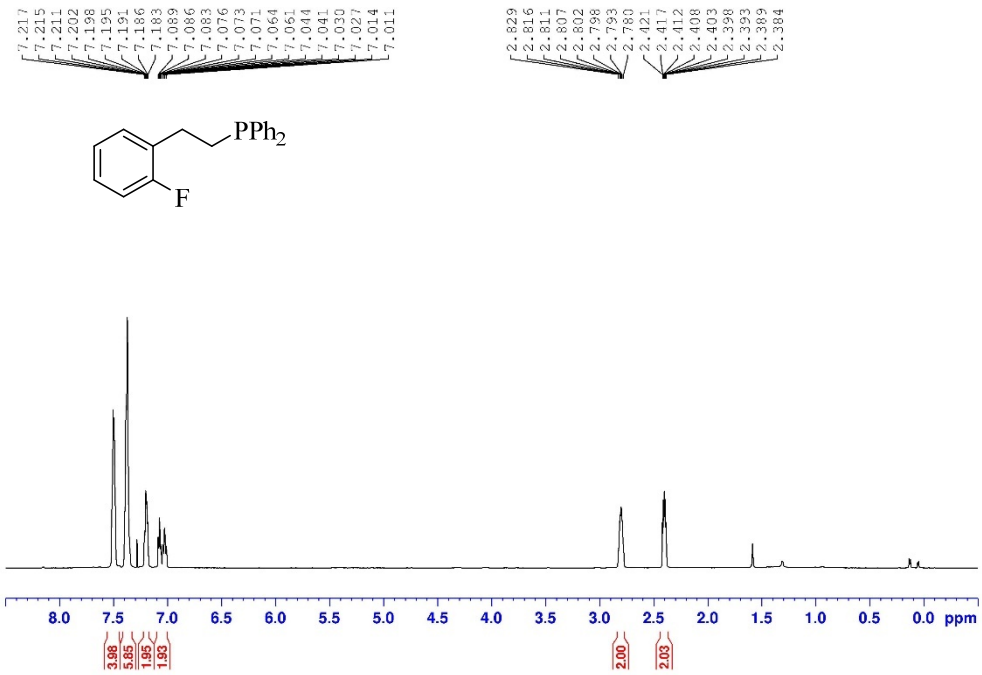


Figure S65. ^{1}H NMR spectrum of compound **4i**

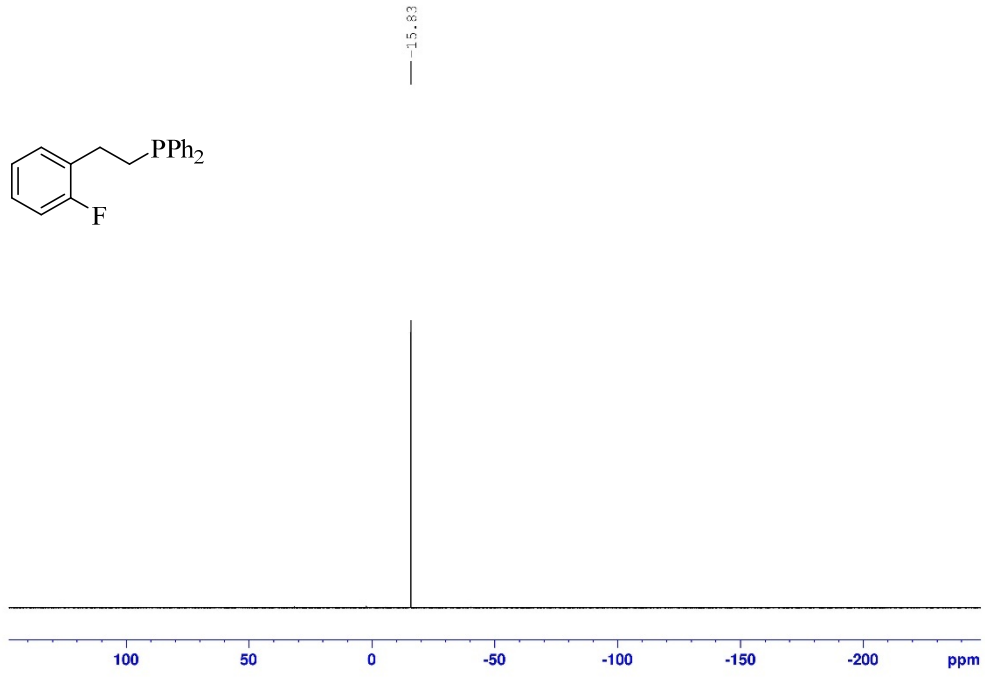


Figure S66. ^{31}P NMR spectrum of compound **4i**

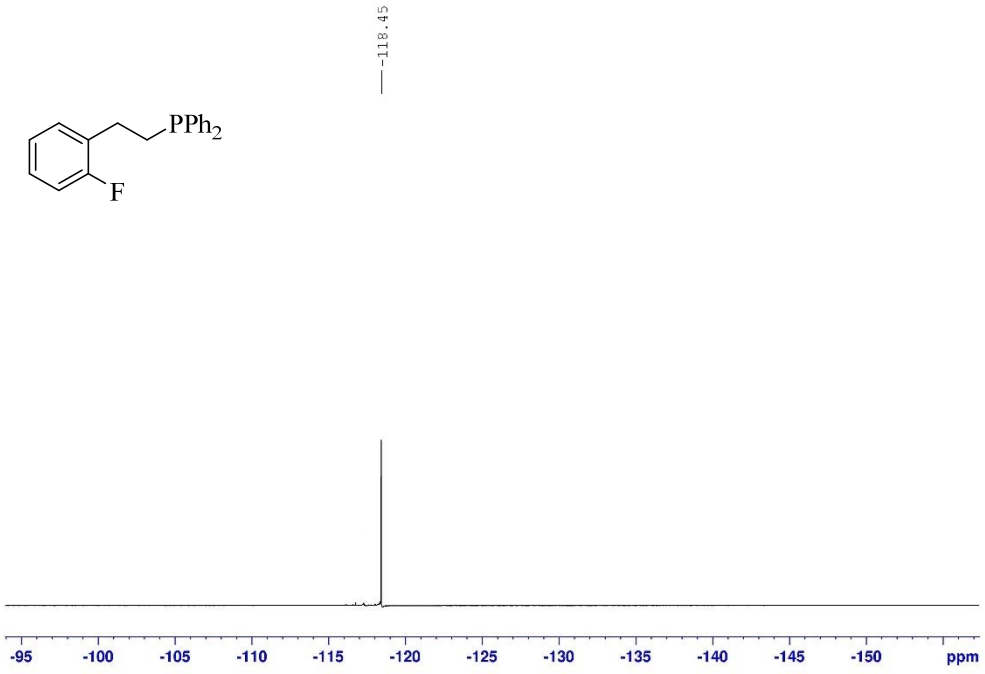


Figure S67. ^{19}F NMR spectrum of compound **4i**

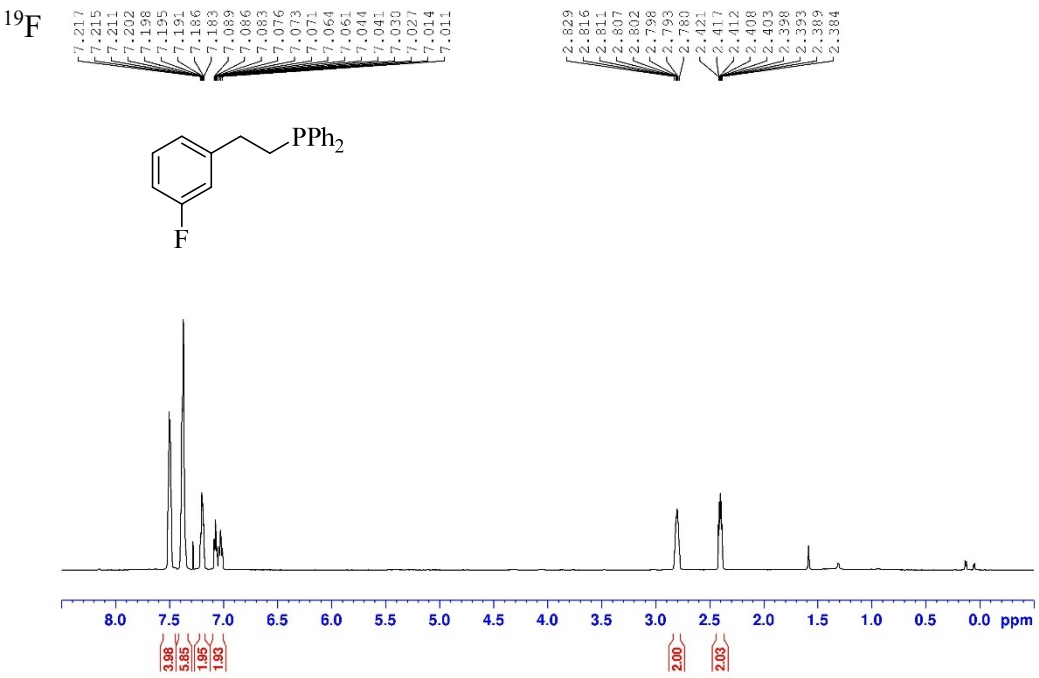


Figure S68. ^{1}H NMR spectrum of compound **4j**

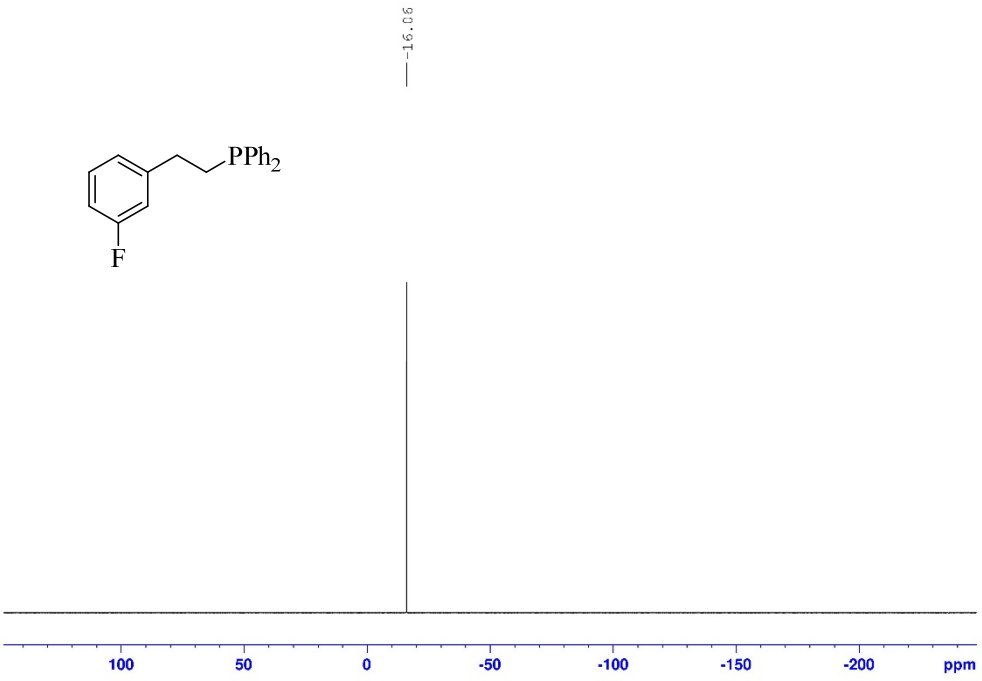


Figure S69. ^{31}P NMR spectrum of compound **4j**

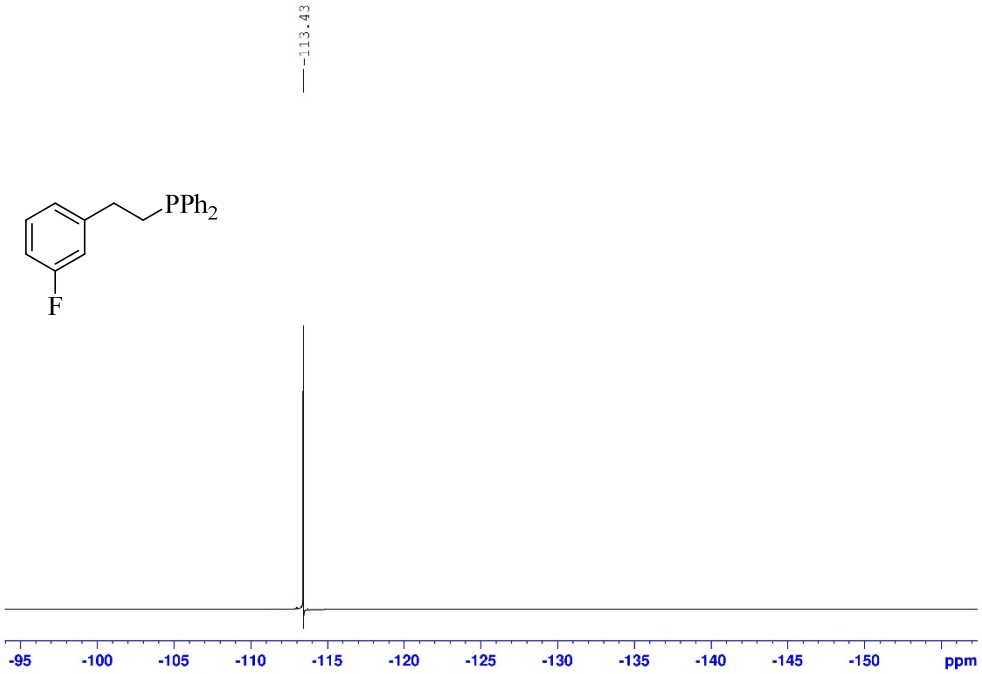


Figure S70. ^{19}F NMR spectrum of compound **4j**

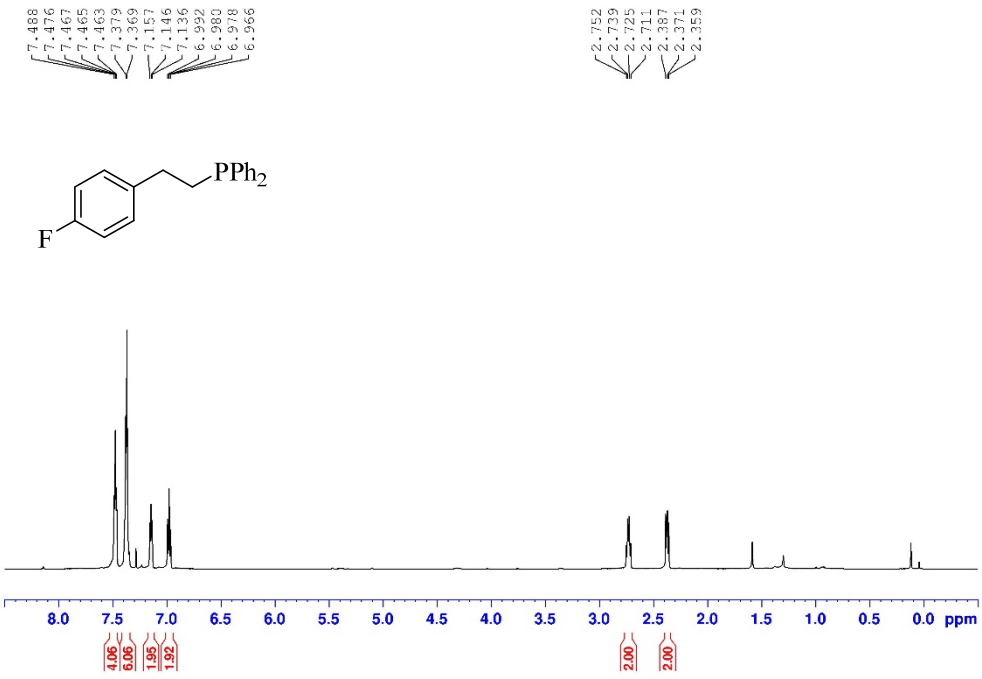


Figure S71. ^{1}H NMR spectrum of compound **4k**

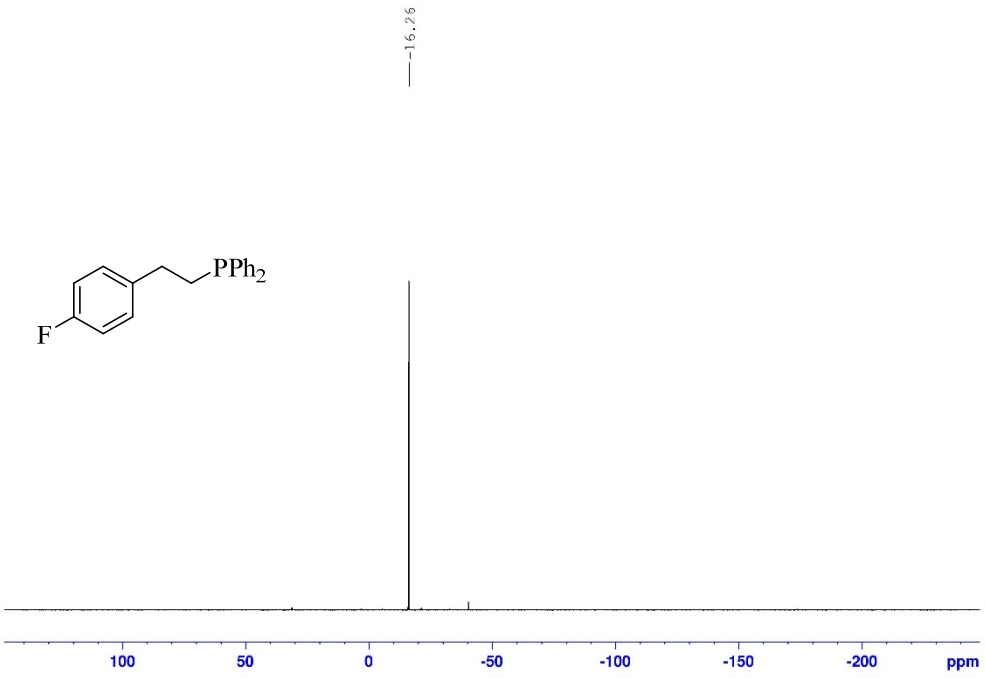


Figure S72. ^{31}P NMR spectrum of compound **4k**

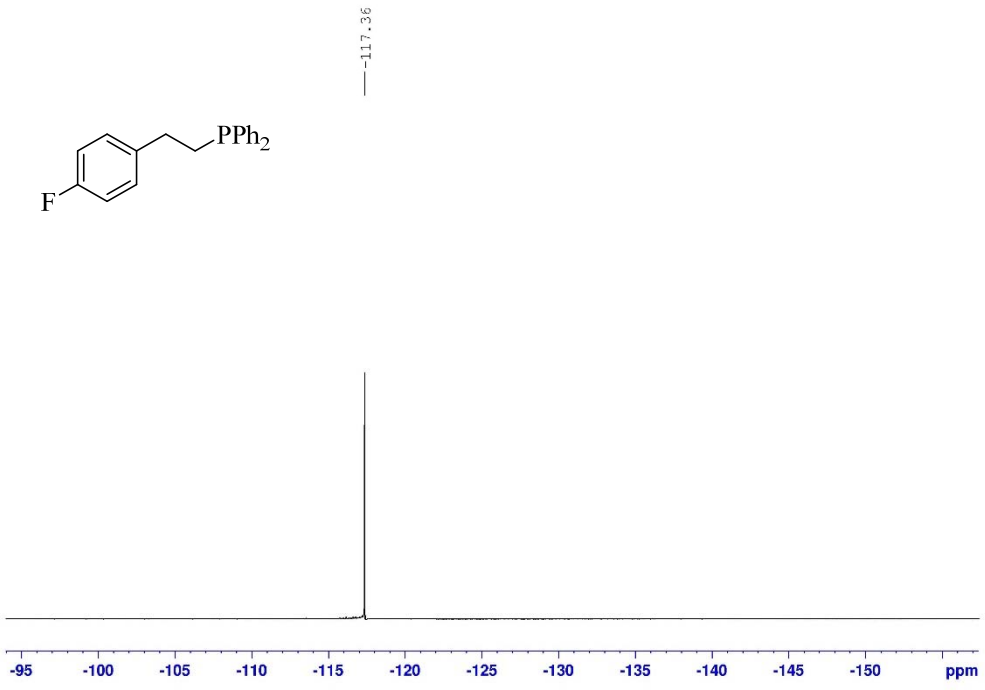


Figure S73. ^{19}F NMR spectrum of compound **4k**

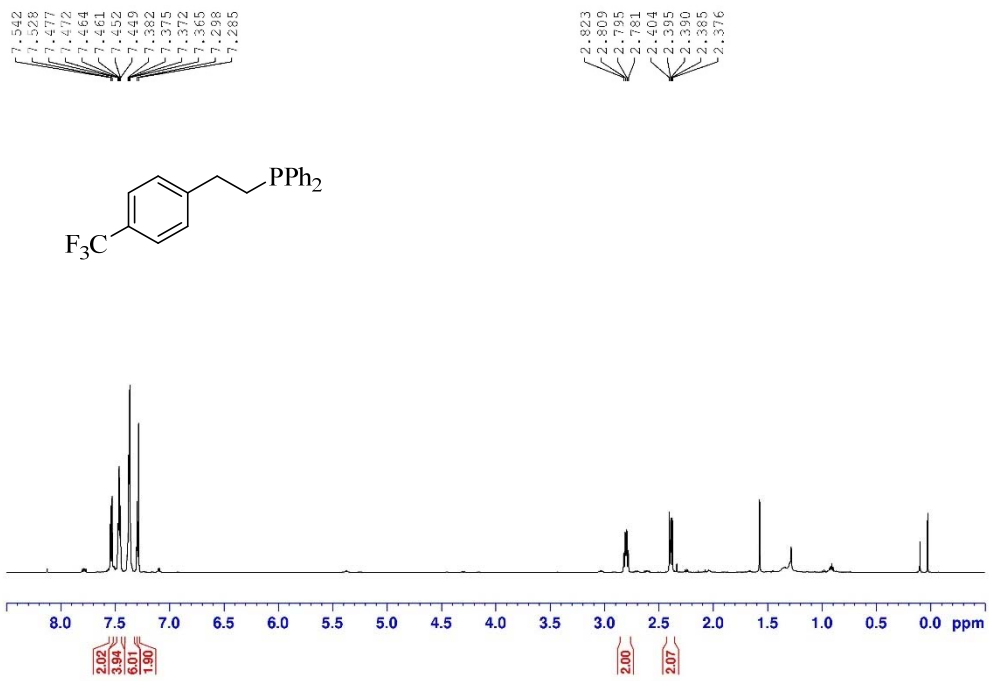


Figure S74. ^{1}H NMR spectrum of compound **4l**

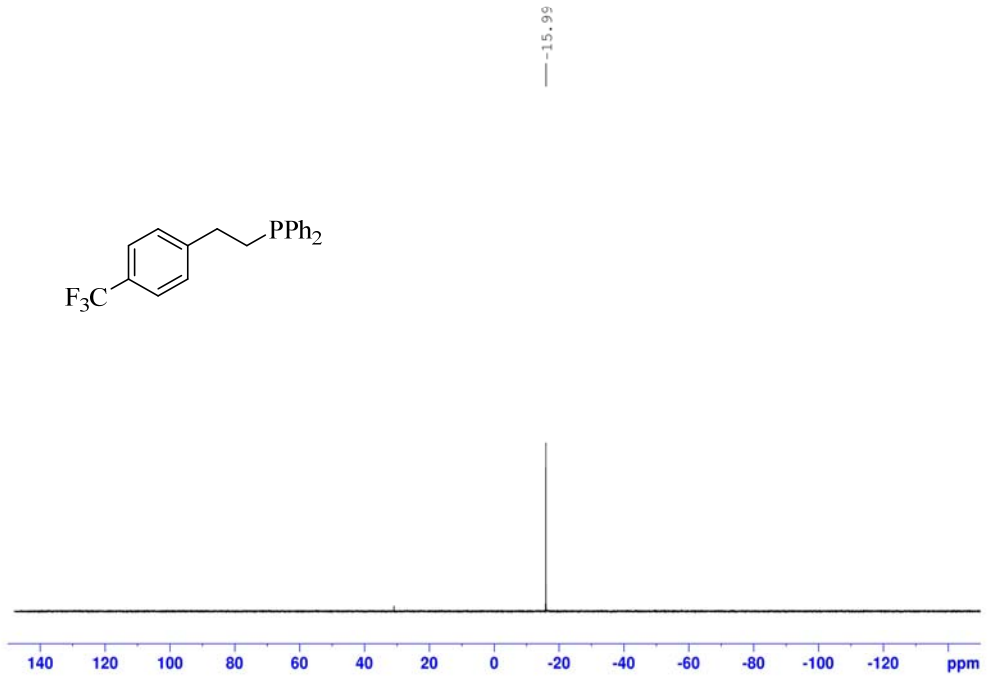


Figure S75. ^{31}P NMR spectrum of compound **4l**

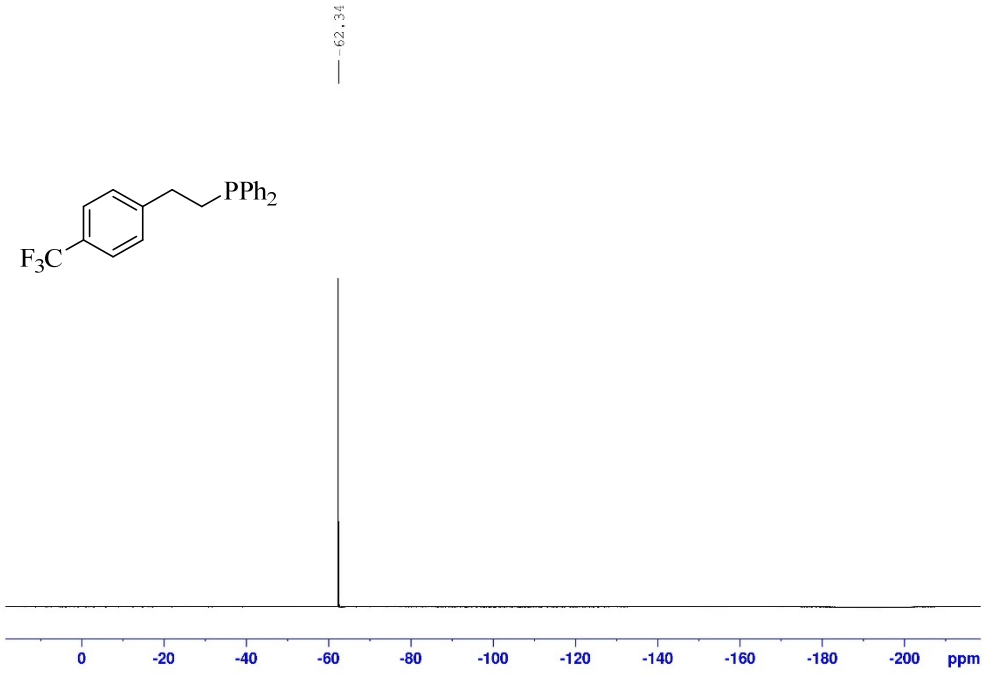


Figure S76. ^{19}F NMR spectrum of compound **4l**

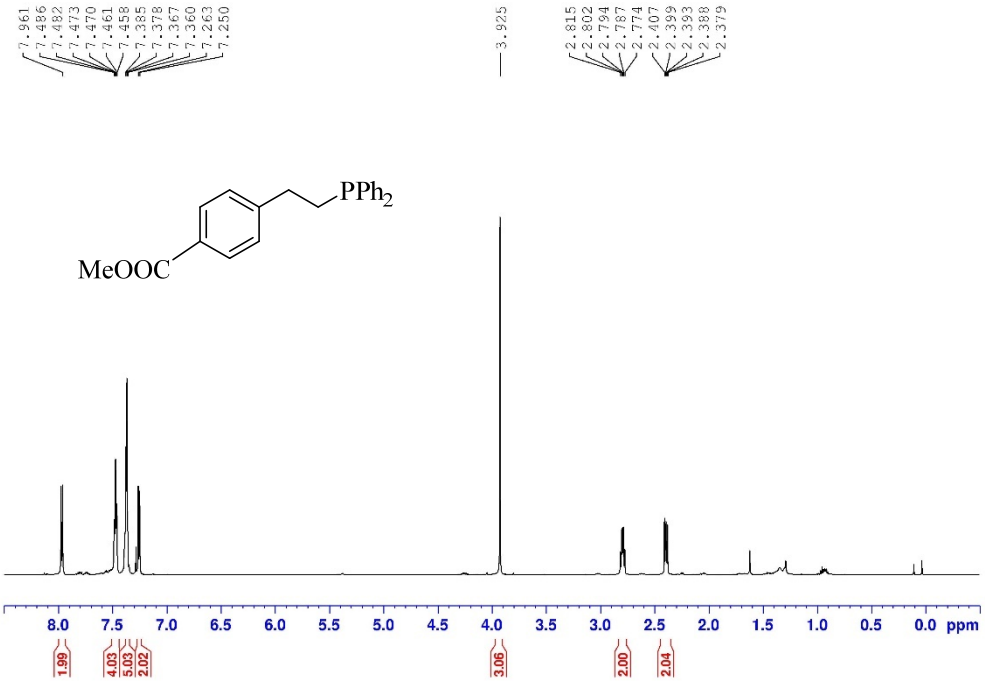


Figure S77. ^{1}H NMR spectrum of compound **4m**

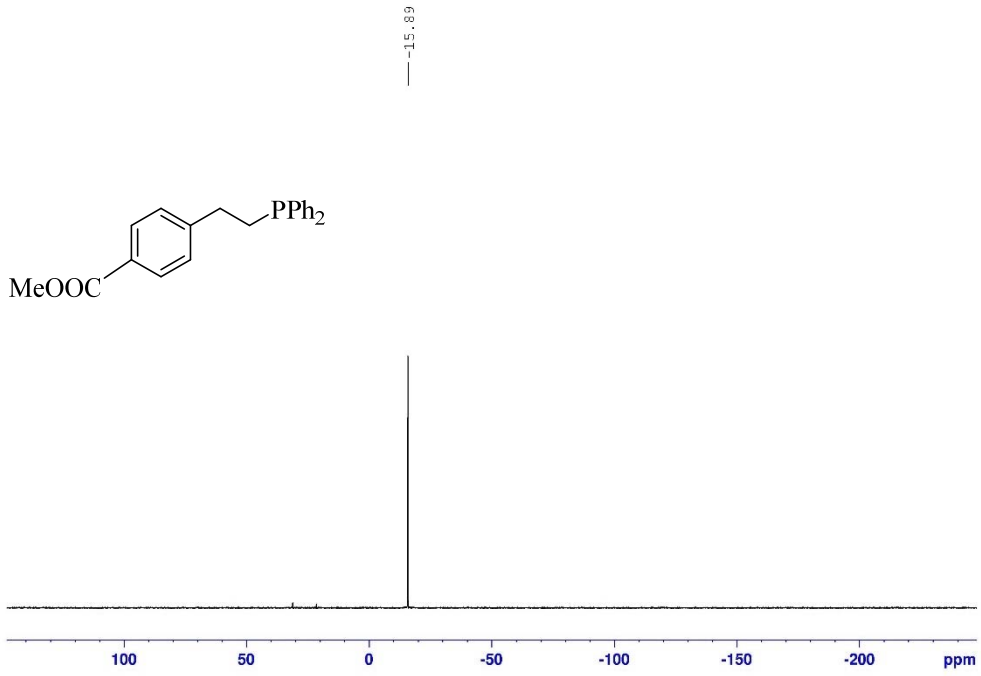


Figure S78. ^{31}P NMR spectrum of compound **4m**

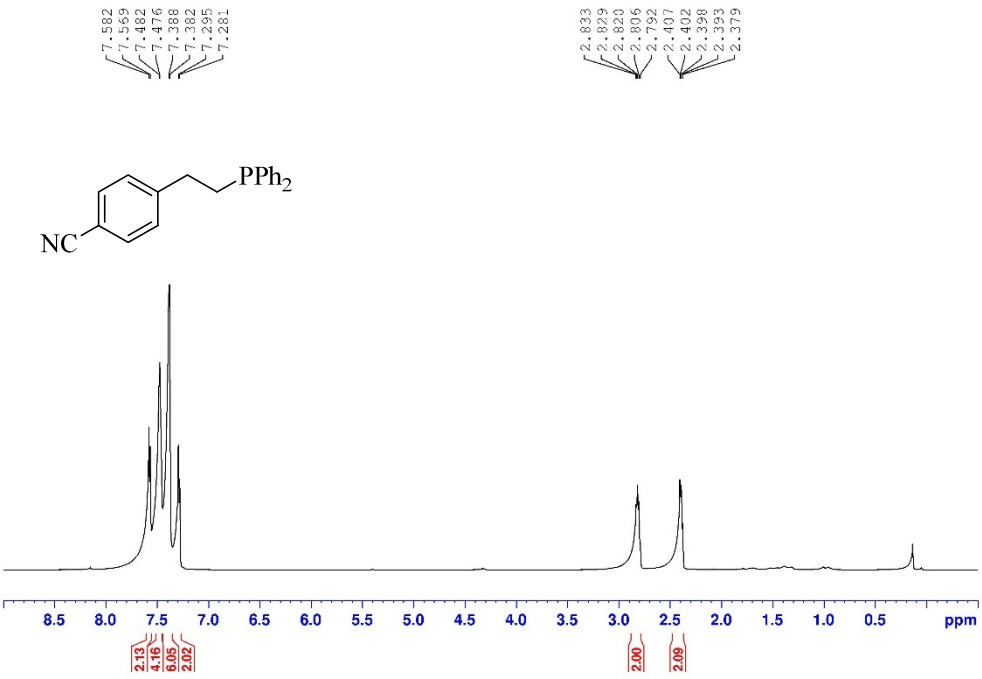


Figure S79. ^{1}H NMR spectrum of compound **4n**

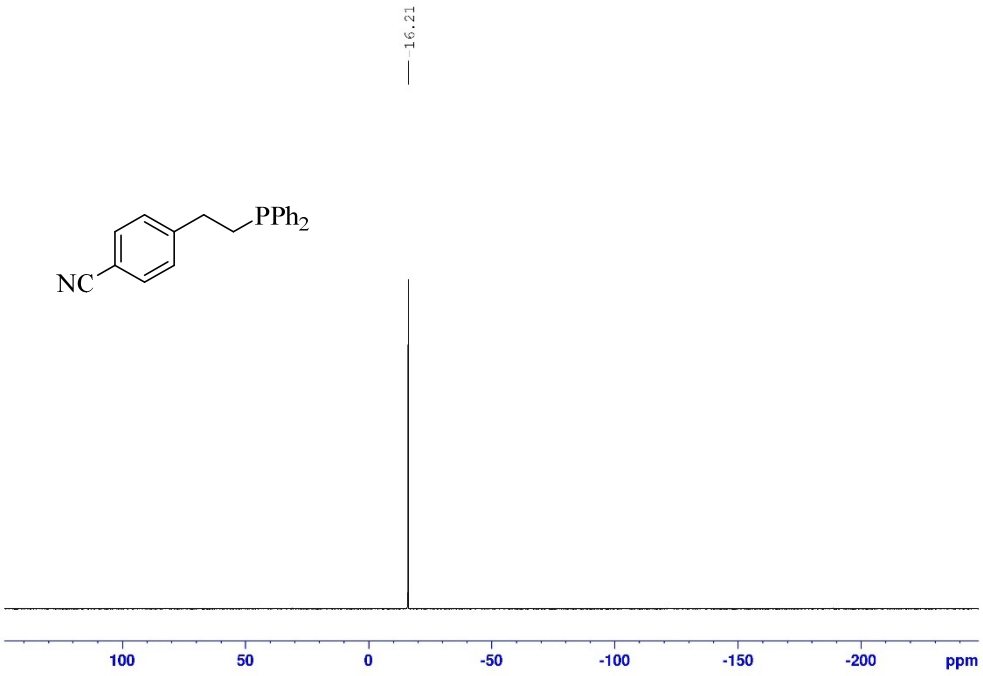


Figure S80. ^{31}P NMR spectrum of compound **4n**

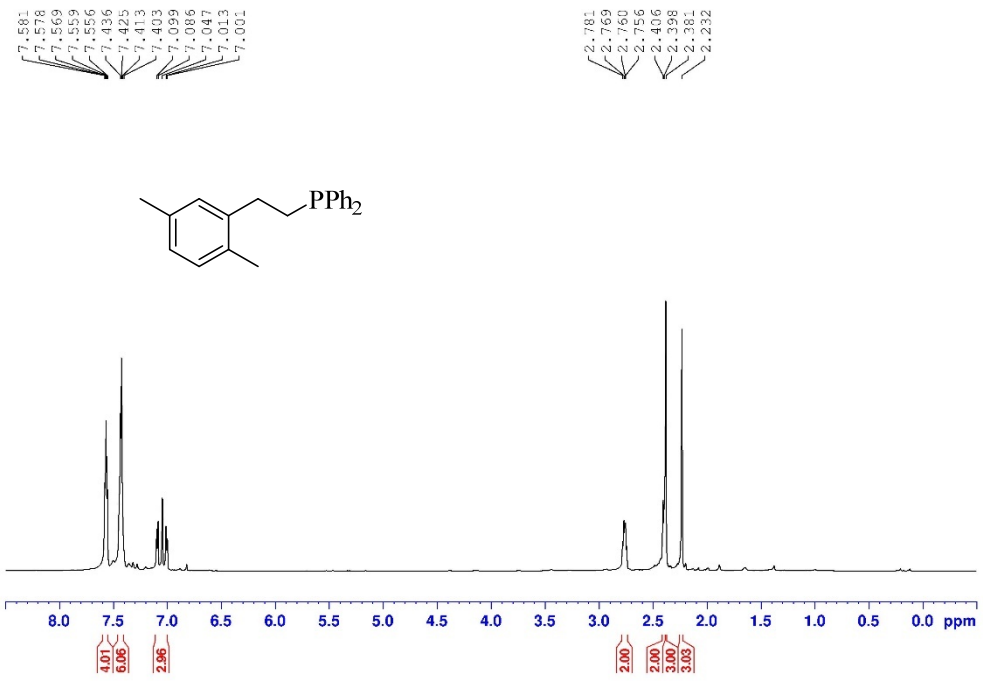


Figure S81. ^{1}H NMR spectrum of compound **4o**

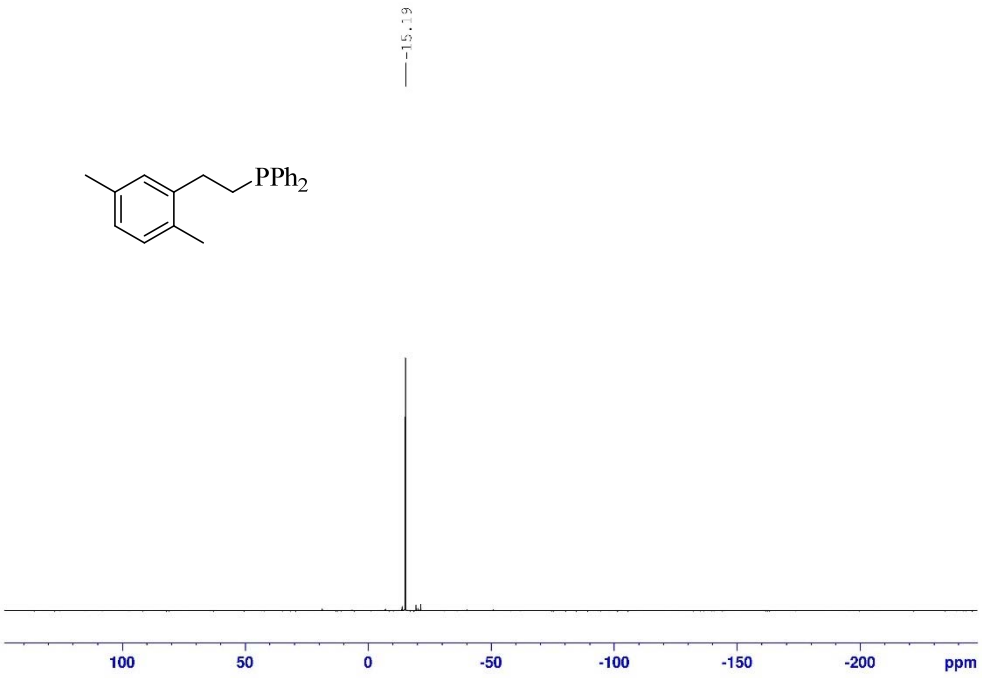


Figure S82. ^{31}P NMR spectrum of compound **4o**

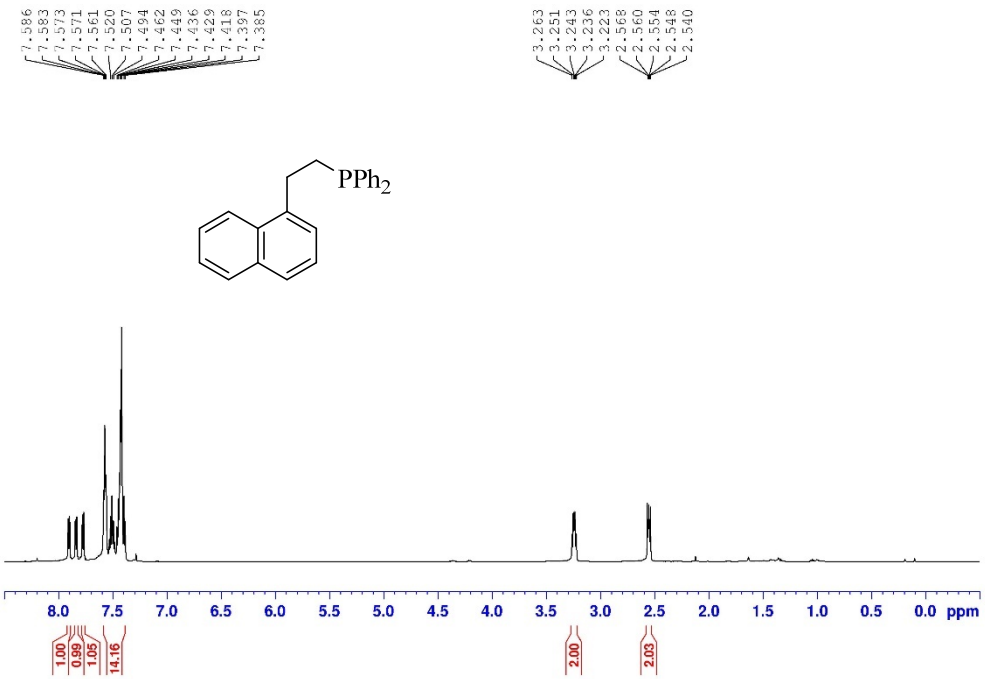


Figure S83. ^{1}H NMR spectrum of compound **4p**

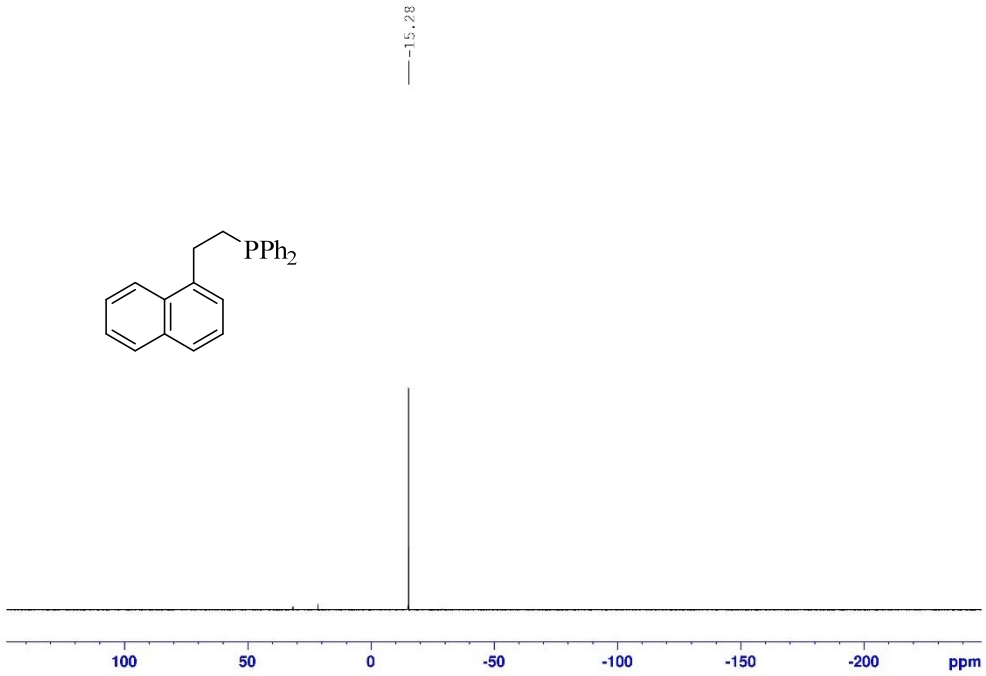


Figure S84. ^{31}P NMR spectrum of compound **4p**

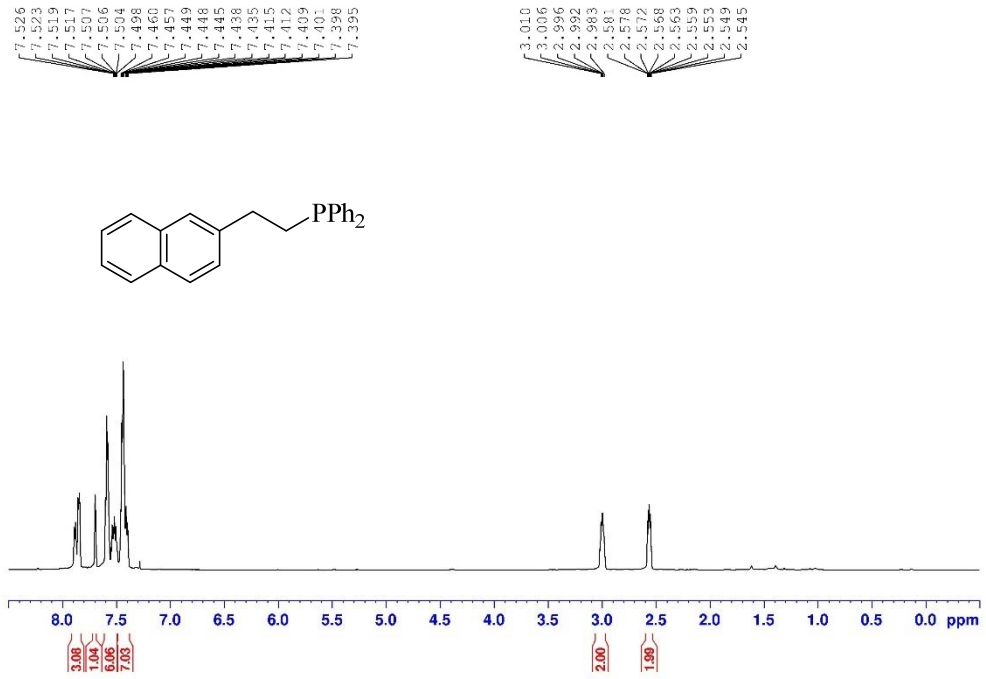


Figure S85. ^{1}H NMR spectrum of compound **4q**

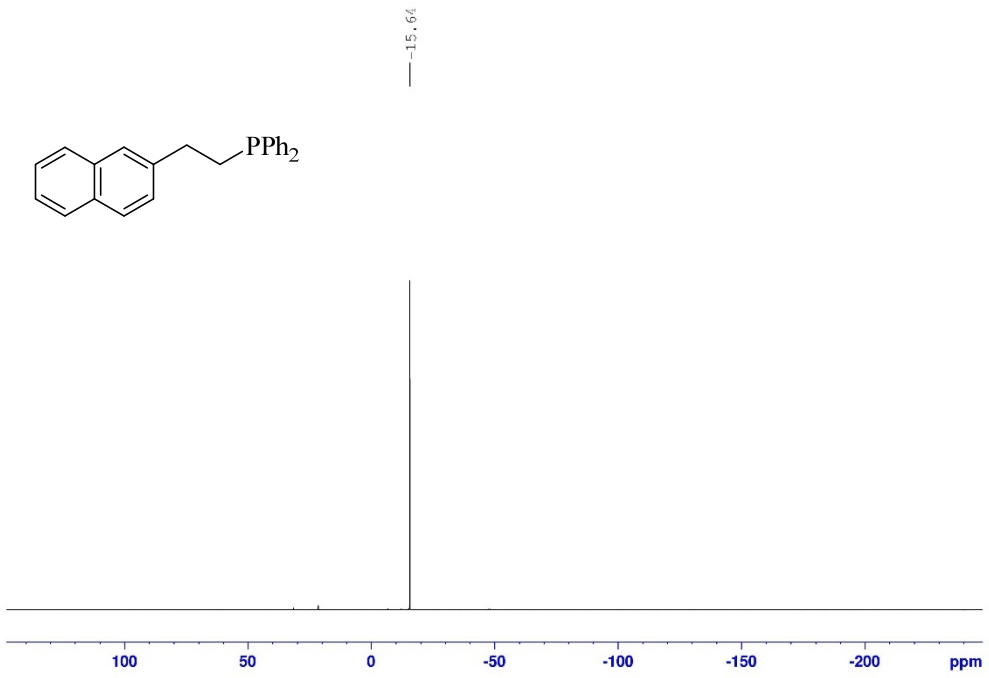


Figure S86. ^{31}P NMR spectrum of compound **4q**

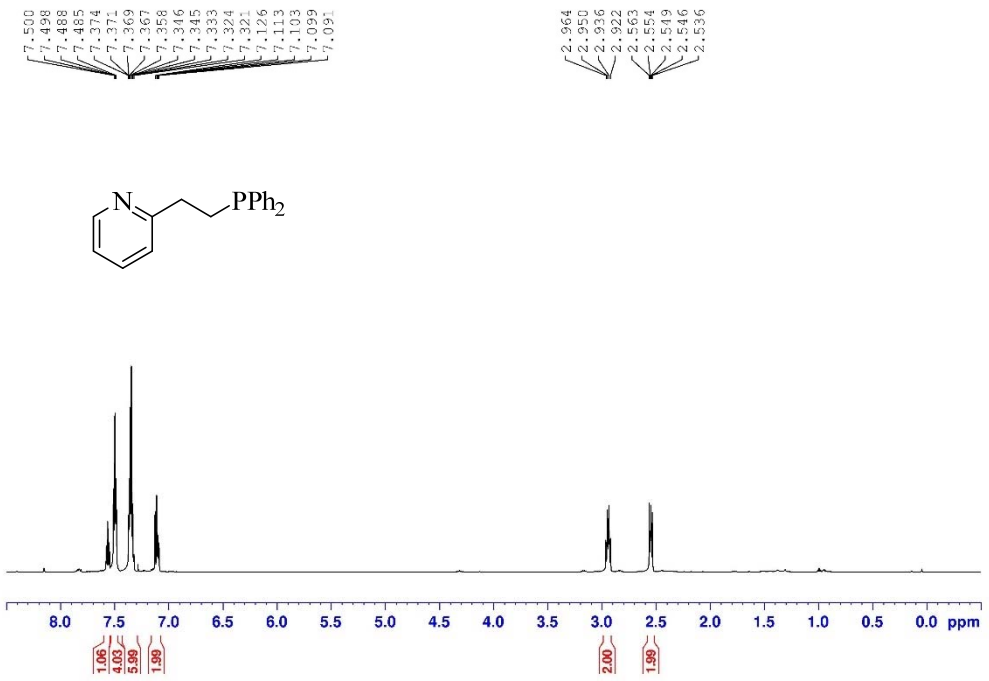


Figure S87. ^{1}H NMR spectrum of compound **4r**

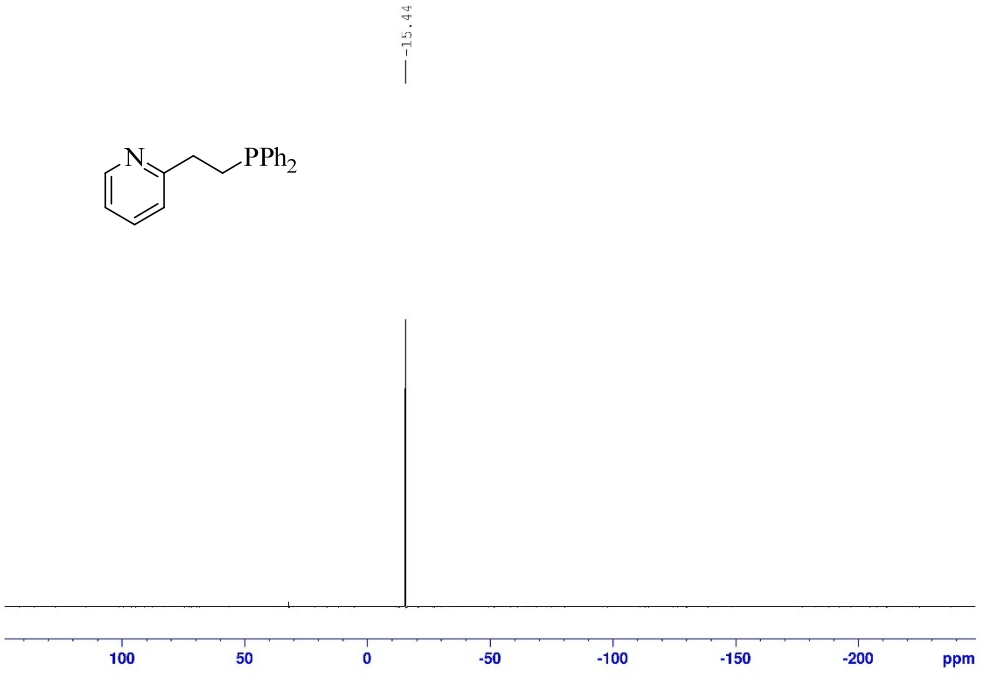


Figure S88. ^{31}P NMR spectrum of compound **4r**

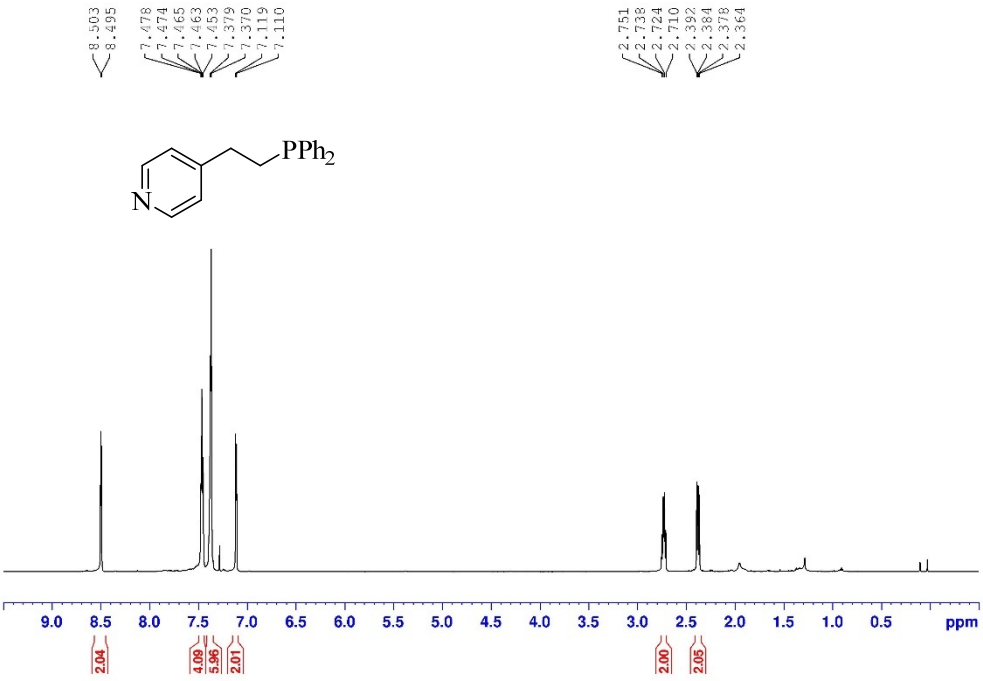


Figure S89. ^{1}H NMR spectrum of compound **4s**

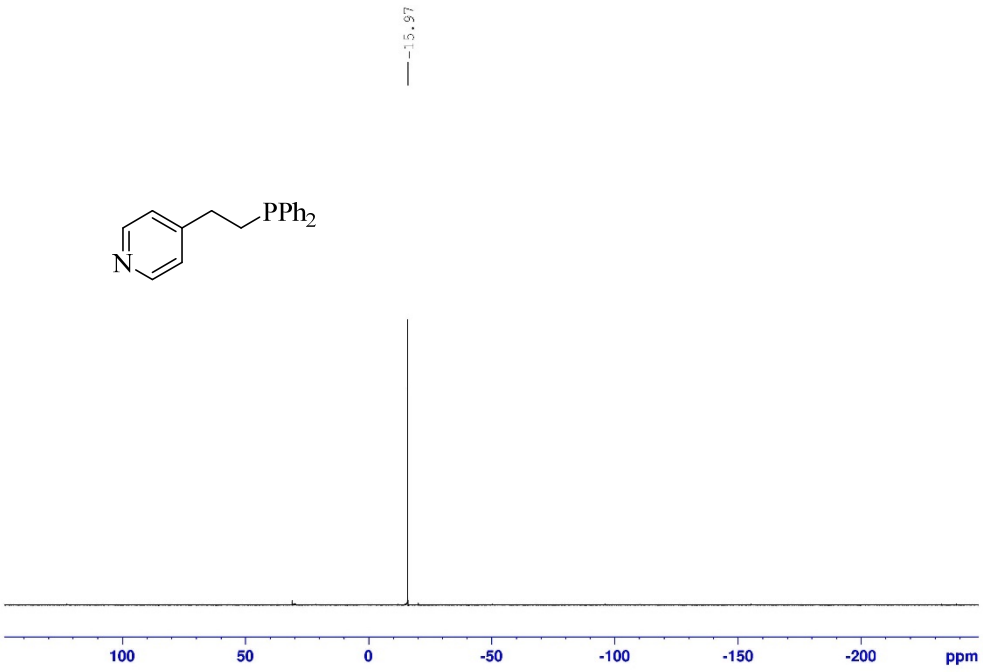


Figure S90. ^{31}P NMR spectrum of compound **4s**

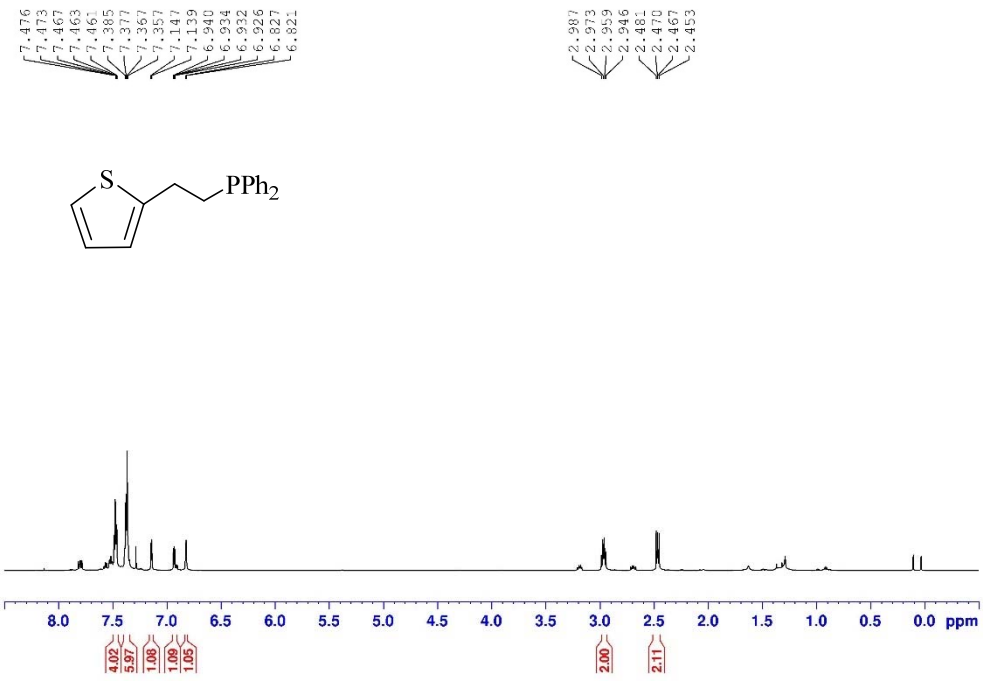


Figure S91. ^{1}H NMR spectrum of compound **4t**

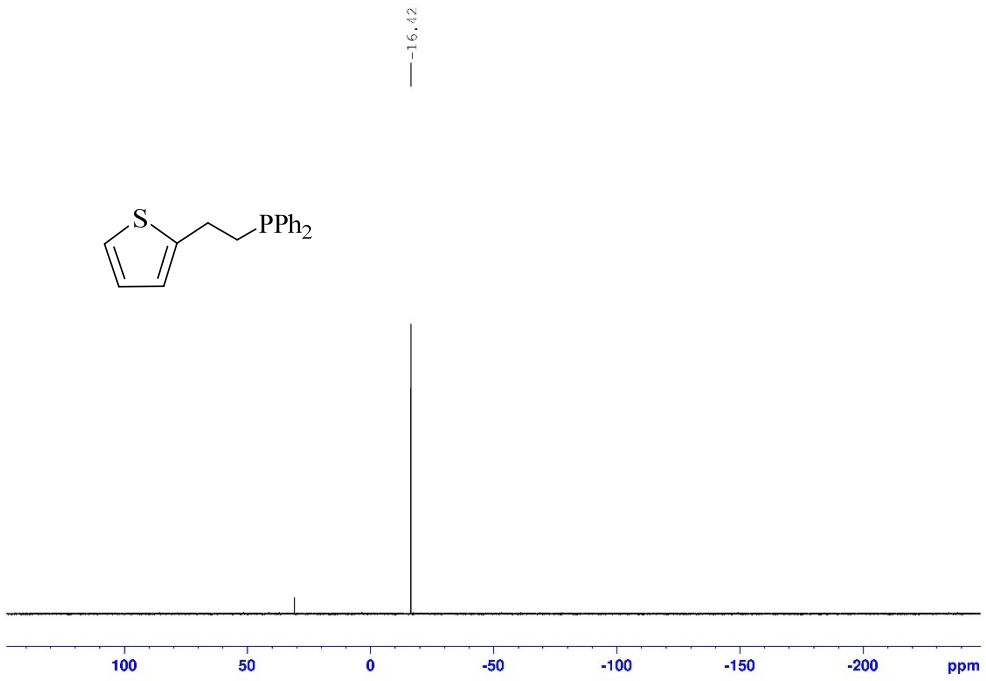


Figure S92. ^{31}P NMR spectrum of compound **4t**

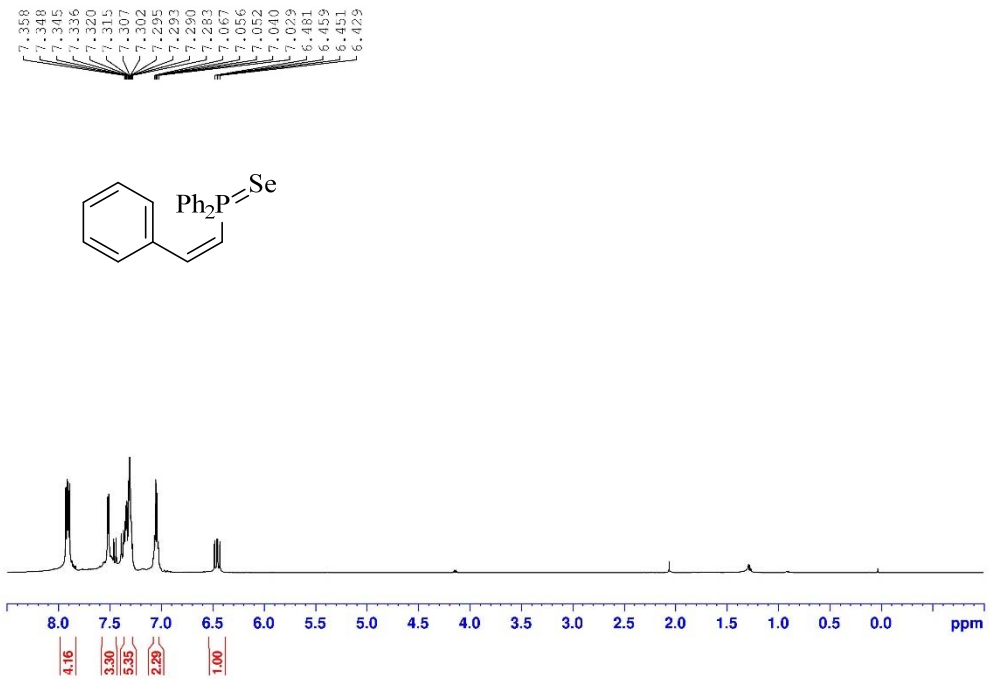


Figure S93. ^{1}H NMR spectrum of compound **2a-Se**

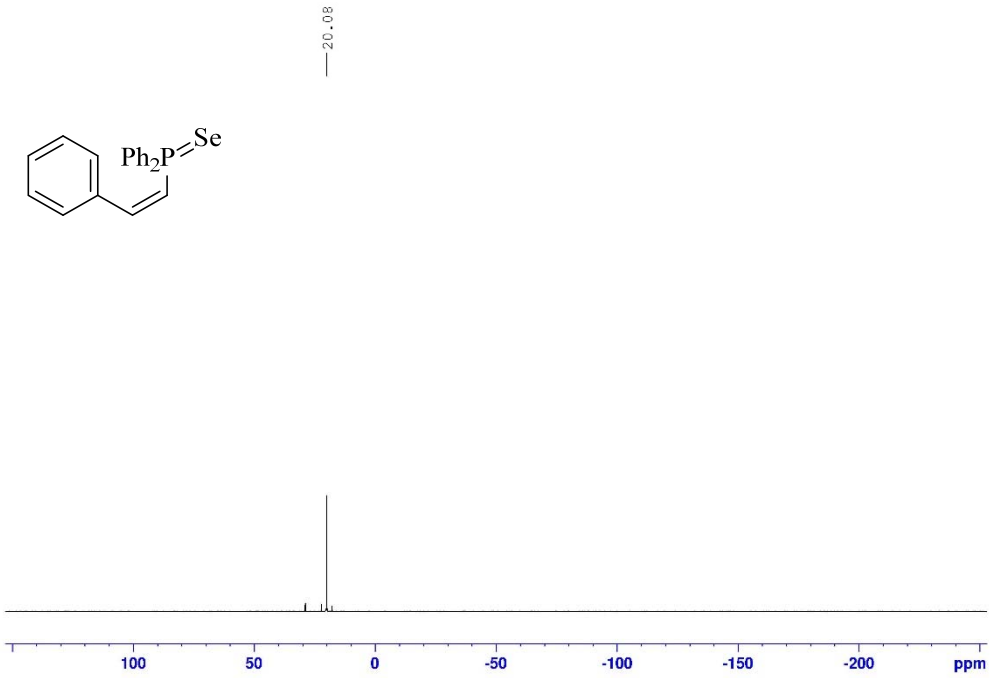


Figure S94. ^{31}P NMR spectrum of compound **2a-Se**

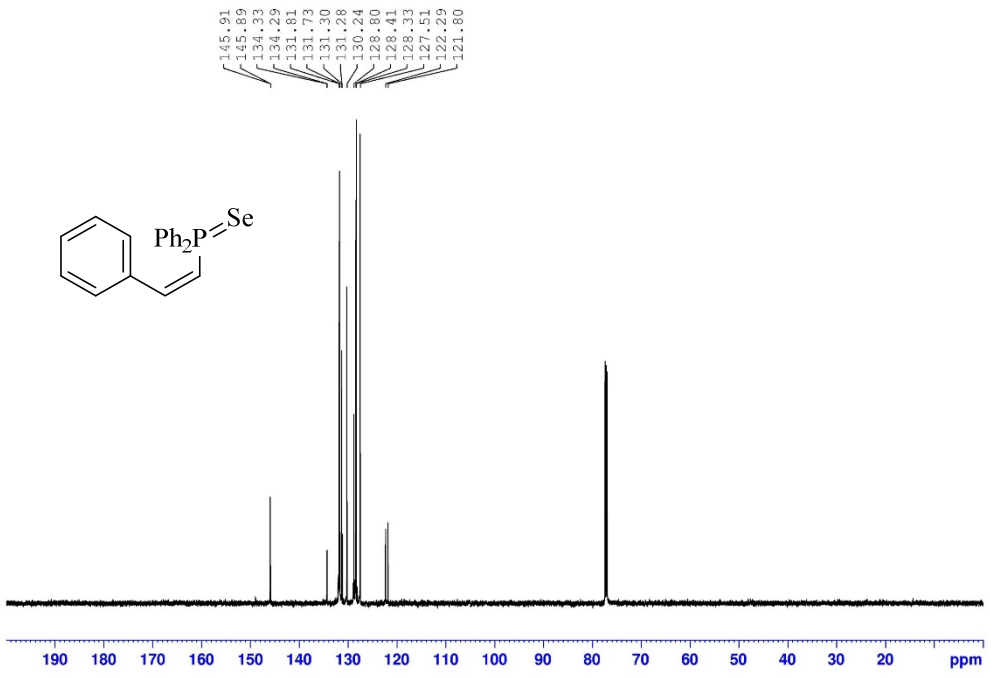


Figure S95. ^{13}C NMR spectrum of compound **2a-Se**

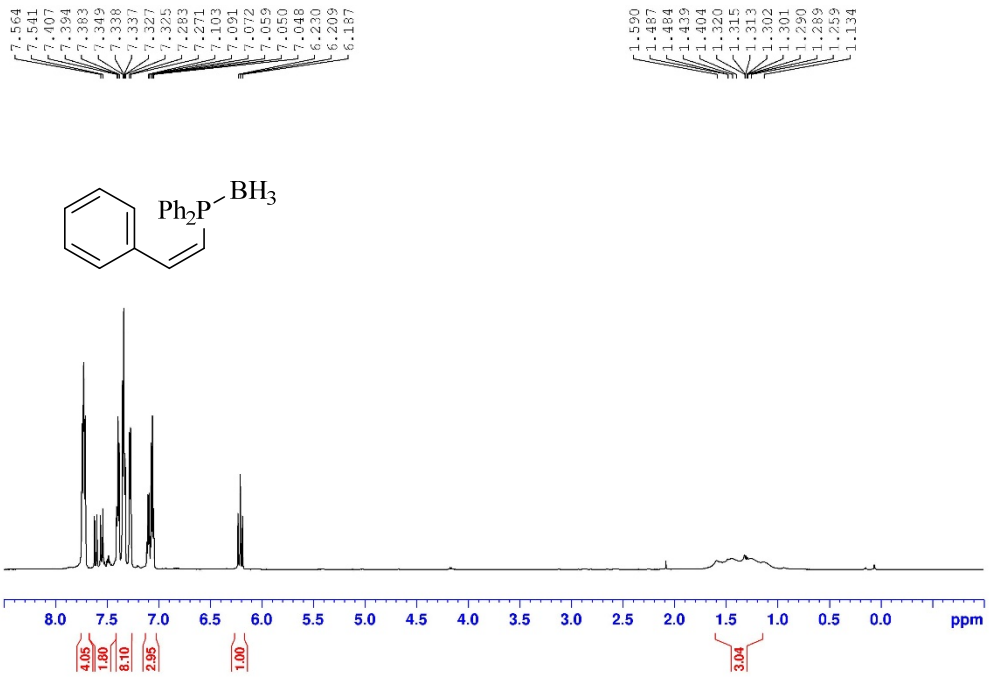


Figure S96. ^{1}H NMR spectrum of compound **2a-BH$_3$**

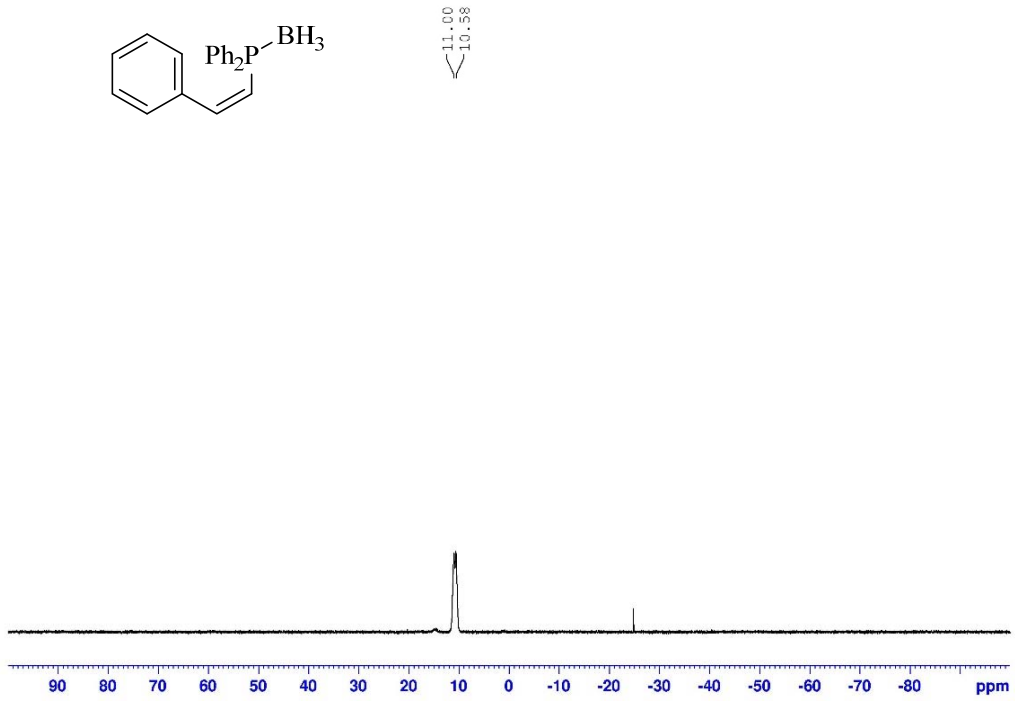


Figure S97. ^{31}P NMR spectrum of compound **2a-BH$_3$**

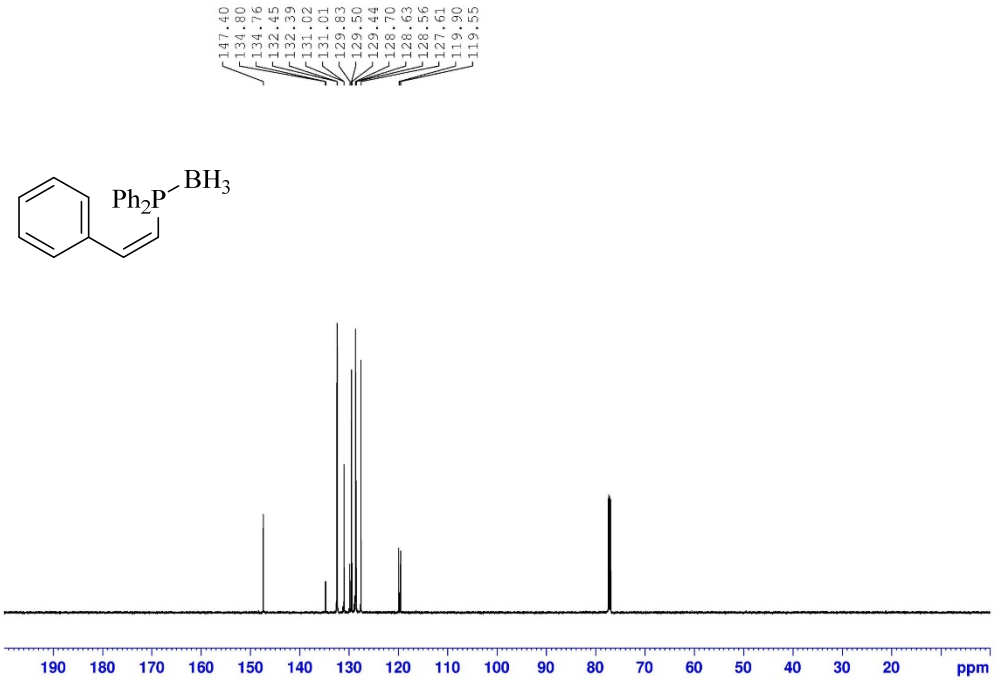


Figure S98. ^{13}C NMR spectrum of compound **2a-BH$_3$**

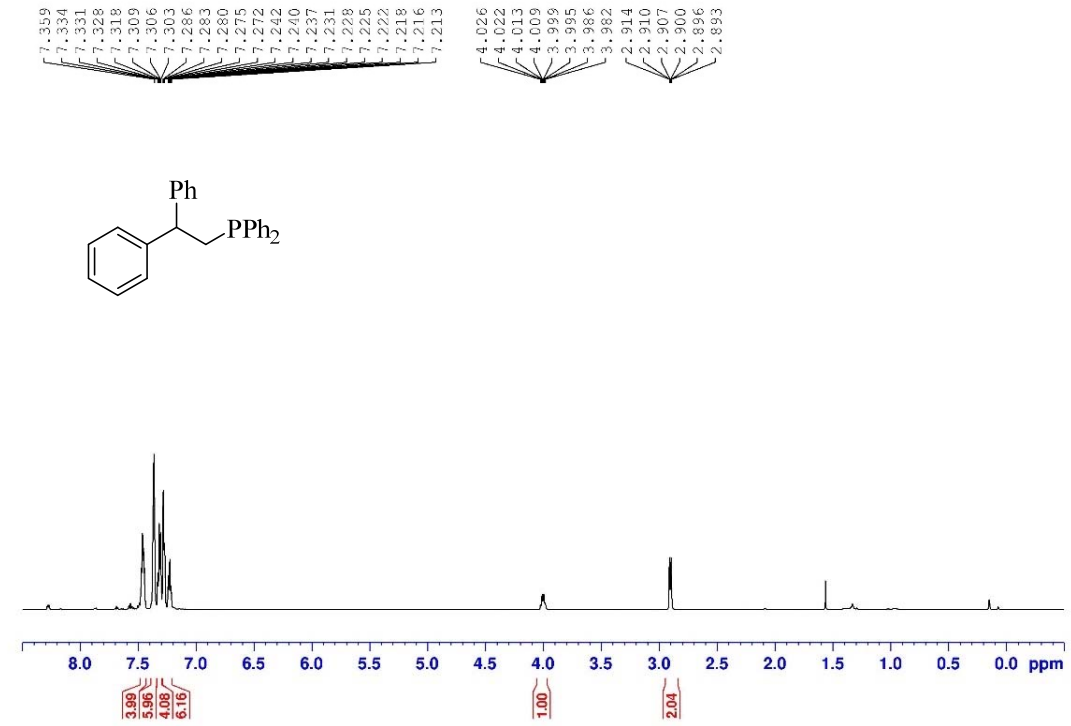


Figure S99. ^{1}H NMR spectrum of compound **2u**

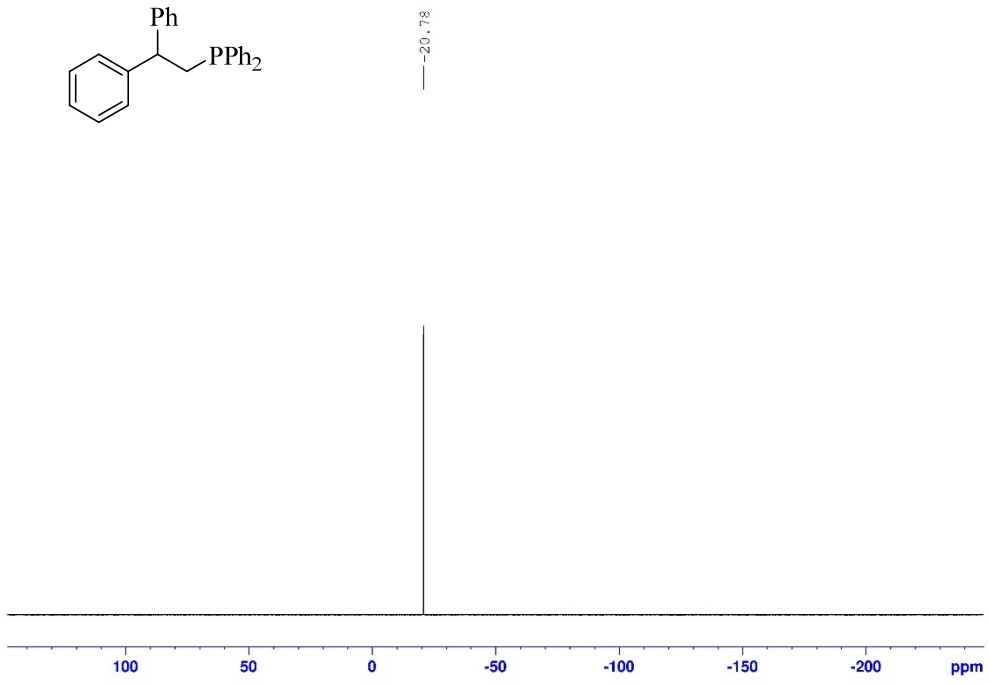


Figure S100. ^{31}P NMR spectrum of compound **2u**

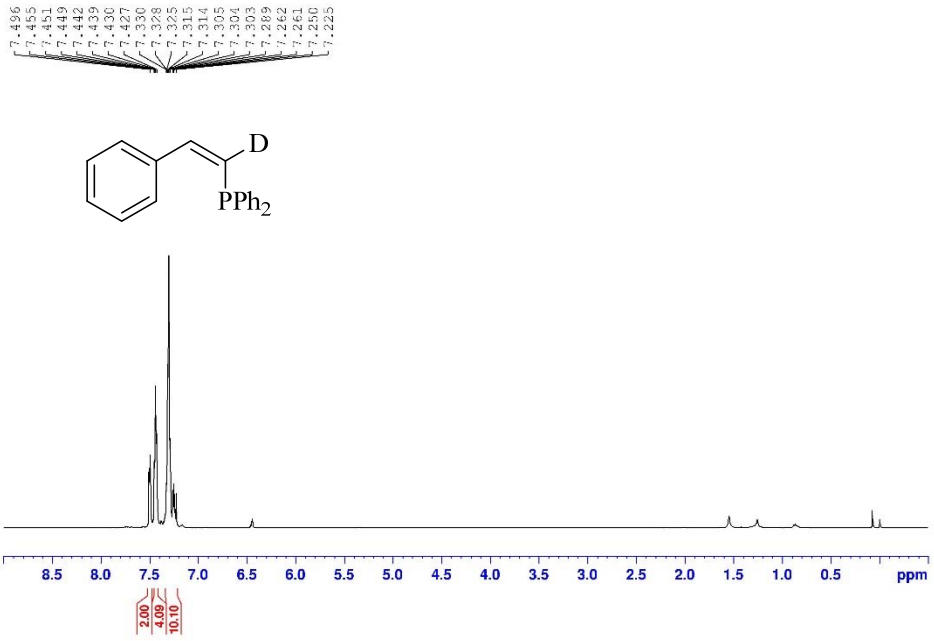


Figure S101. ^{1}H NMR spectrum of compound **2a-D**

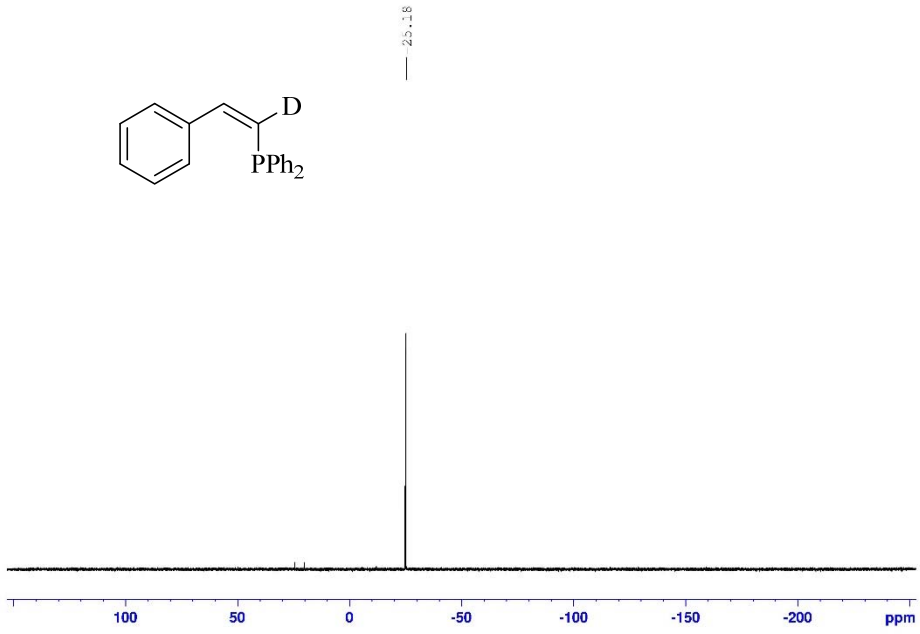


Figure S102. ^{31}P NMR spectrum of compound **2a-D**

11. References

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