Electrochemical Phosphorylation of α, β-Unsaturated Carboxylic Acids via Decarboxylative Cross-Coupling Reaction

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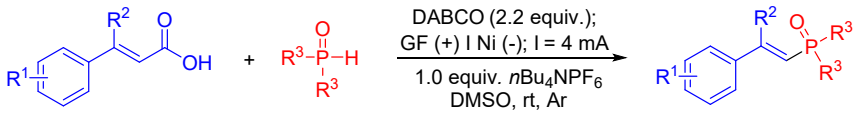
1. General information:

All reactions were carried out under Ar. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were measured on Bruker AVIII 500M spectrometers with $CDCl_3$ as solvent and the residual protonated solvent as internal standard or 85% H_3PO_4 as external standard for ^{31}P NMR (202 MHz). Chemical shifts were reported in units (ppm) by assigning the residual protonated solvent of $CDCl_3$ resonance in the 1H spectrum as 7.26 ppm and $CDCl_3$ resonance in the ^{13}C spectrum as 77.16 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). Chemical shifts of common trace ^{1}H NMR impurities (ppm): H_2O: 1.56, $CHCl_3$: 7.26. Column chromatography was performed on silica gel 300-400 mesh. The unknown products were further characterized by HRMS-ESI. Highresolution mass spectra (HRMS) were recorded with an Thermo Scientific Q Exactive Plus Orbitrap LC-MS/MS System by ESI on a quadrupole mass analyzer. All crystals were grown via a slow evaporation method. Each compound was dissolved in a 2 mL solution of either DCM within a 5 mL brown flask, which was covered with a film at the flask's mouth. Ensure not to seal it too tightly. Allow the solvent to gradually evaporate over the next 2-3 days, resulting in the formation of high-quality crystals. The single-crystal X-ray diffraction data were collected on a Bruker D8 QUEST diffractometer. Electrolysis experiments were performed using a HSPY power supply. Graphite felt was cut into 25 x 10 x 2 mm^3 pieces before use. Nickel plate was cut into 10 x 10 x 0.2 mm^3 pieces before use, and was connected to electrical feed-through on the Teflon cap of the electrochemical cell via PTFE electrode holder. Saturated calomel electrode (CHI150), platinum wire counter electrode (CHI115) and platinum working electrode (CHI102) were obtained from CH Instruments and Saturated calomel electrode was stored in 3.0 M KCl aqueous solution before use.

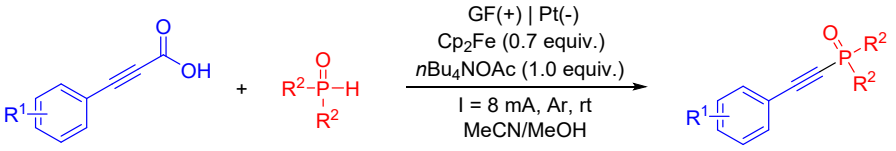
General Procedure A



1 **2** **3**

An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1** (0.3 mmol, 1.0 equiv.), diphenylphosphine oxide **2** (0.9 mmol, 3.0 equiv.), DABCO (0.66 mmol, 2.2 equiv.) and nBu$_4$PF$_6$ (0.3 mmol, 1.0 equiv.). The flask was equipped with a graphite felt anode (25 mm x 10 mm x 2 mm) and a nickel plate cathode (10 mm x 10 mm x 0.2 mm). The cell was sealed and flushed with Argon for 15 minutes, followed by the addition via syringe of DMSO (7.5 mL). The mixture was charged by constant current (I = 4 mA). The complete consumption of the starting material **1** was checked by TLC (30 % AcOEt/petroleum ether). Once the starting material was fully consumed, the power was cut. The reaction solution was concentrated in vacuo and extracted with EtOAc and H_2O (3 × 10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to give the corresponding products **3**.

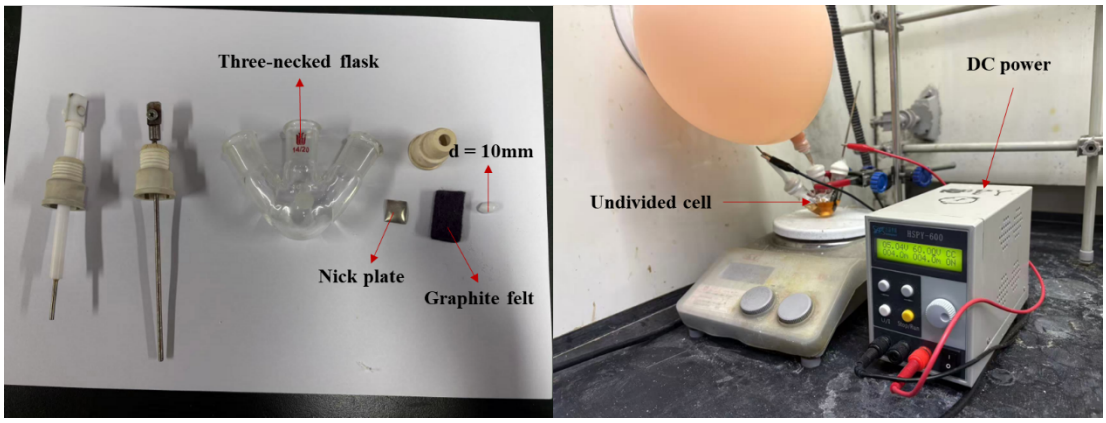
General Procedure B



4 **2** **5**

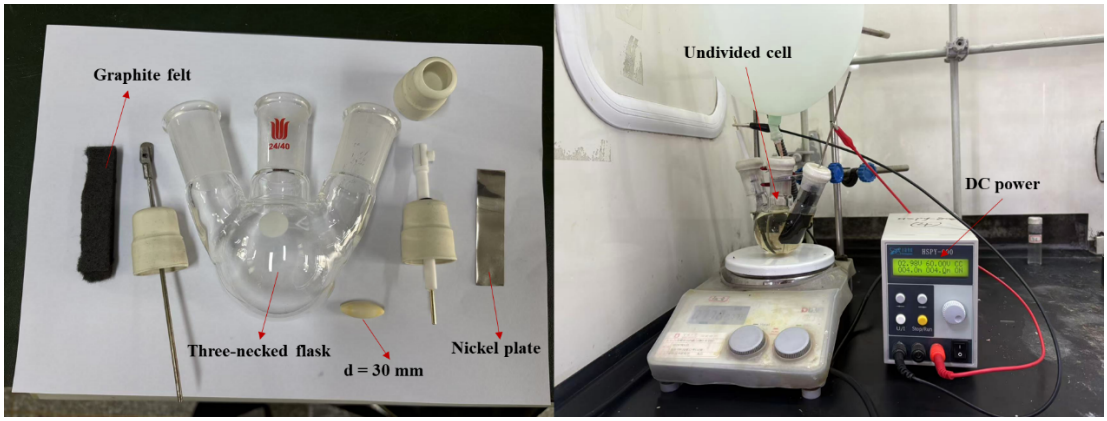
An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **4** (0.3 mmol, 1.0 equiv.), diphenylphosphine oxide **2** (0.9 mmol, 3.0 equiv.), Cp_2Fe (0.21 mmol, 0.7 equiv.) and nBu$_4$NOAc (0.3 mmol, 1.0 equiv.). The flask was equipped with a graphite felt anode (25 mm x 10 mm x 2 mm) and a platinum plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 15 minutes, followed by the addition via syringe of MeCN (7.5 mL) and MeOH (1.5 mL). The mixture was charged by constant current (I = 8 mA). The complete consumption of the starting material **4** was checked by TLC (50 % AcOEt/petroleum ether). Once the starting material was fully consumed, the reaction solution was concentrated in vacuo and extracted with EtOAc and H_2O (3 × 10 mL). The combined organic layer was dried over $MgSO_4$, filtered andconcentrated in vacuo. The residue was purified by silica gel column chromatography to give the corresponding products **5**.

The reaction setup:

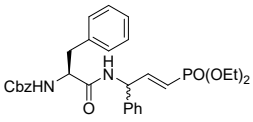


Scale-up experiment: An oven-dried 100 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1a** (4.39 mmol, 0.6497 g, 1.0 equiv.), diphenylphosphine oxide **2a** (13.17 mmol, 3.0 equiv.), DABCO (9.66 mmol, 2.2 equiv.) and n-Bu$_4$NPF$_6$ (4.39 mmol, 1.0 equiv.). The flask was equipped with a graphite felt anode (70 mm x 15 mm x 10 mm) and a nickel plate cathode (70 mm x 15 mm x 0.2 mm). The cell was sealed and flushed with Argon for 15 minutes, followed by the addition via syringe of DMSO (110 mL). The mixture was charged by constant current (I = 4 mA). The complete consumption of the starting material **1a** was checked by TLC (30 % AcOEt/petroleum ether). The reaction solution was concentrated in vacuo and extracted with EtOAc and H_2O (3 x 10 mL). The combined organic layer was dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography to give the corresponding products **3a** (694.6 mg, 2.28 mmol, 52%).

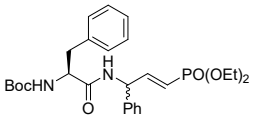
Gram-scale experiment:



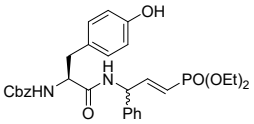
Peptidyl-vinylaminophosphonates[ref.2a]



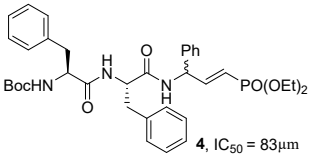
1, IC_{50} = 30μm



2, IC_{50} = 40μm

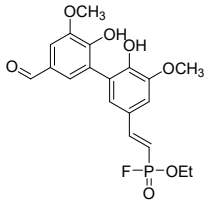


3, IC_{50} = 54μm

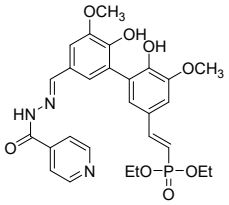


4, IC_{50} = 83μm

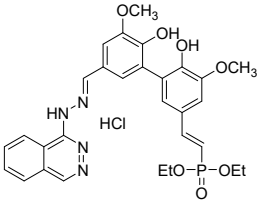
Antioxidant Effect (TBARS) and Residual Cytotoxicity (MTT) of Biaryls Derivatives[ref.2b]



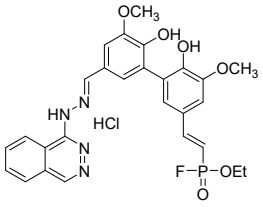
5, TBARS 135.1 ± 8.9 %,
MTT 26.8 ± 4.6 %



6, TBARS 67.5 ± 6.9 %
MTT 31.2 ± 5.5 %



7, TBARS 4.1 ± 0.6 %
MTT 61.2 ± 1.5%

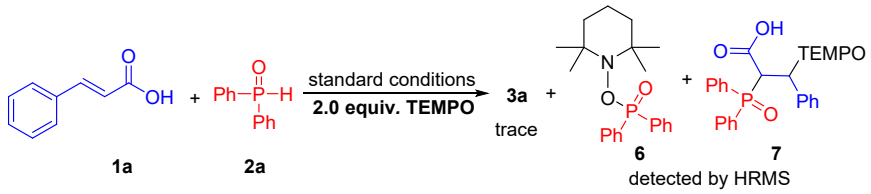


8, TBARS 2.6 ± 0.6 %
MTT 68.9 ± 0.9%

Some bioactive molecules containing alkenylphosphorus moiety

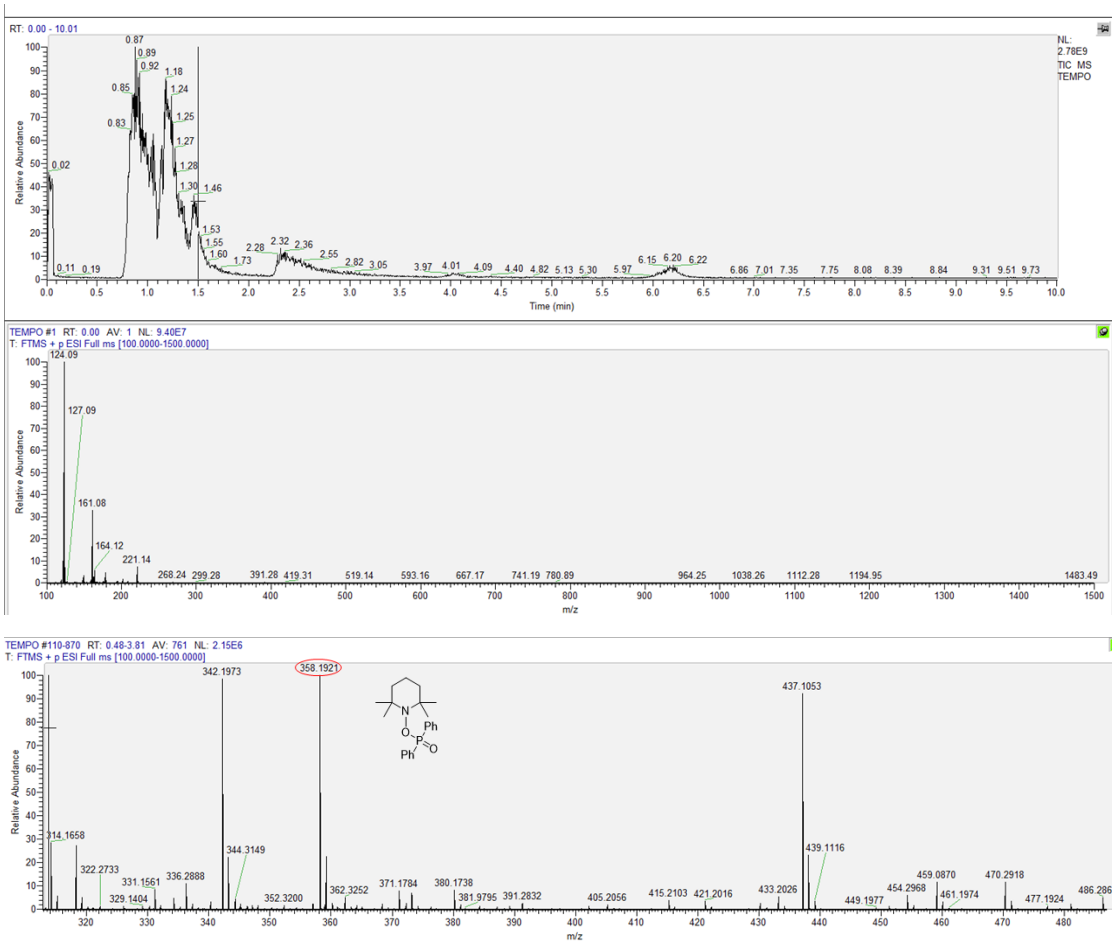
2. Control experiments

Control experiment 1:

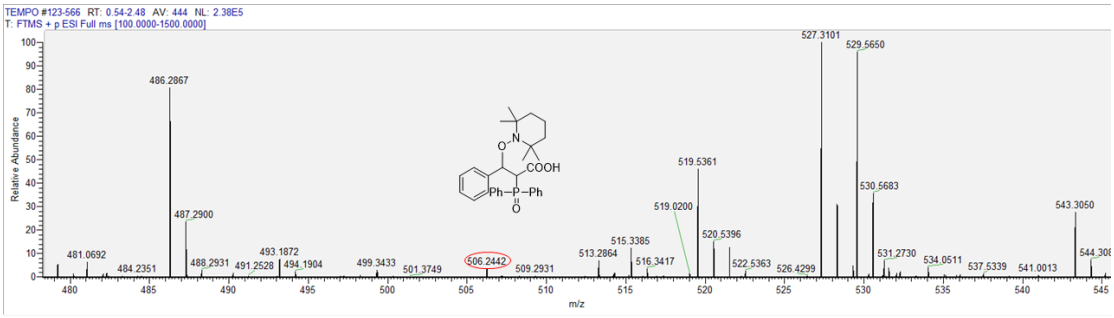


An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with **1a** (0.3 mmol, 1.0 equiv.), diphenylphosphine oxide **2a** (0.9 mmol, 3.0 equiv.), DABCO (0.66 mmol, 2.2 equiv.), TEMPO ((2,2,6,6-tetramethylpiperidin-1-yl)oxyl, 0.6 mmol, 2.0 equiv.) and *n*-Bu_4NPF_6 (0. mmol, 1.0 equiv.). The flask was equipped with a graphite felt anode (25 mm x 10 mm x 2 mm) and a nickel plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 15 minutes, followed by the addition via syringe of DMSO (7.5 mL). The mixture was charged by constant current (I = 4 mA) for 6 hours. Only trace amount of **3a** was detected, the recovery was 90%. The crude mixture was analyzed by LC-MS**.**

The mixture was checked by LC-MS:

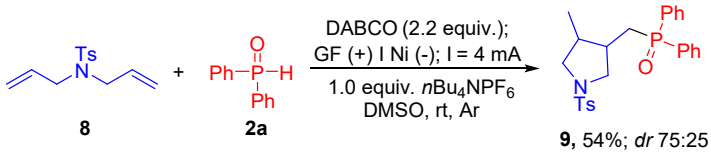


HRMS-ESI: Calcd for $C_{21}H_{29}NO_2P^+$ $[M+H]^+$ 358.1930, found 358.1921



HRMS-ESI: Calcd for $C_{30}H_{37}NO_4P^+$ $[M+H]^+$506.2455, found 506.2442

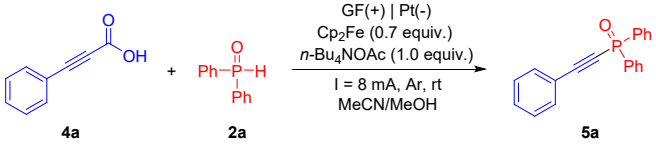
Control experiment 2:



9, 54%; *dr* 75:25

An oven-dried 10 mL three-necked round-bottomed flask with a magnetic stir bar was charged with *N*, *N*-diallyl-4-methylbenzenesulfonamide **8** (75.3 mg. 0.3 mmol), diphenylphosphine oxide **2a** (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol) The flask was equipped with a graphite felt anode (25 mm x 10 mm x 2 mm) and a nickel plate cathode (10 mm x 10 mmx 0.2 mm). The cell was sealed and flushed with Argon for 15 minutes, followed by the addition via syringe of DMSO (7.5 mL). The mixture was charged by constant current (I = 4 mA) for 6 hours. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **9** (73.4 mg, 0.16 mmol, 54%, dr 75:25) as colorless solid.

The optimization of electrochemical synthesis of alkynylphosphine oxides

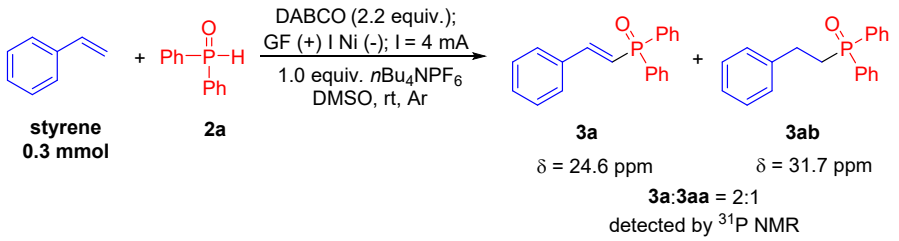


Entry	Variation from the standard conditions	Yield (%)[b]
1	None	62
2	Cp_2Fe (1.0 equiv.) instead of Cp_2Fe (0.7 equiv.)	38
3	Cp_2Fe (0.5 equiv.) instead of Cp_2Fe (0.7 equiv.)	41
4	Cp_2Fe (0.2 equiv.) instead of Cp_2Fe (0.7 equiv.)	35
5	without Cp_2Fe	n.r.
6	Et_3N instead of Cp_2Fe	trace
7	DABCO instead of Cp_2Fe	45
8	tris(4-bromophenyl)amine instead of Cp_2Fe	32
9	2.0 mA instead of 8.0 mA	26
10	5.0 mA instead of 8.0 mA	45
11	10.0 mA instead of 8.0 mA	43
12	CH_3CN/H_2O instead of CH_3CN/MeOH	22
13	CH_3CN/HOAc instead of CH_3CN/MeOH	26

14	DMF/MeOH instead of CH_3CN/MeOH	n.r.
15	THF/MeOH instead of CH_3CN/MeOH	45
16	CH_3CN/MeOH/DMF (15:3:1) instead of CH_3CN/MeOH	46
17	CH_3CN/MeOH/HOAc (15:3:1) instead of CH_3CN/MeOH	40
18	*n*-Bu_4NBF_4 instead of *n*-Bu_4NOAc	32
19	*n*-Bu_4NPF_6 instead of *n*-Bu_4NOAc	41
20	*n*-Bu_4NBr instead of *n*-Bu_4NOAc	8
21	$LiCLO_4$ instead of *n*-Bu_4NOAc	45
22	Ni (-) instead of Pt (-)	58
23	Cu (-) instead of Pt (-)	52
24	C (-) instead of Pt (-)	55
25	Pt (+) instead of GF (+)	42
26	C (+) instead of GF (+)	35
27	Cu (+) instead of GF (+)	trace
28	Without electricity	n.r.

[a] Constant current mode: undivided cell, graphite felt (+) | Pt (-), I = 8.0 mA, **4a** (0.3 mmol, 1.0 equiv.), **2a** (0.9 mmol, 3.0 equiv.), and Cp_2Fe (0.21 mmol, 0.7 equiv.), CH_3CN/MeOH: 7.5 mL + 1.5 mL, *n*-Bu_4NOAc (0.3 mmol, 1.0 equiv.) were used; the isolated yields were given.

The experimental details of reaction with styrene



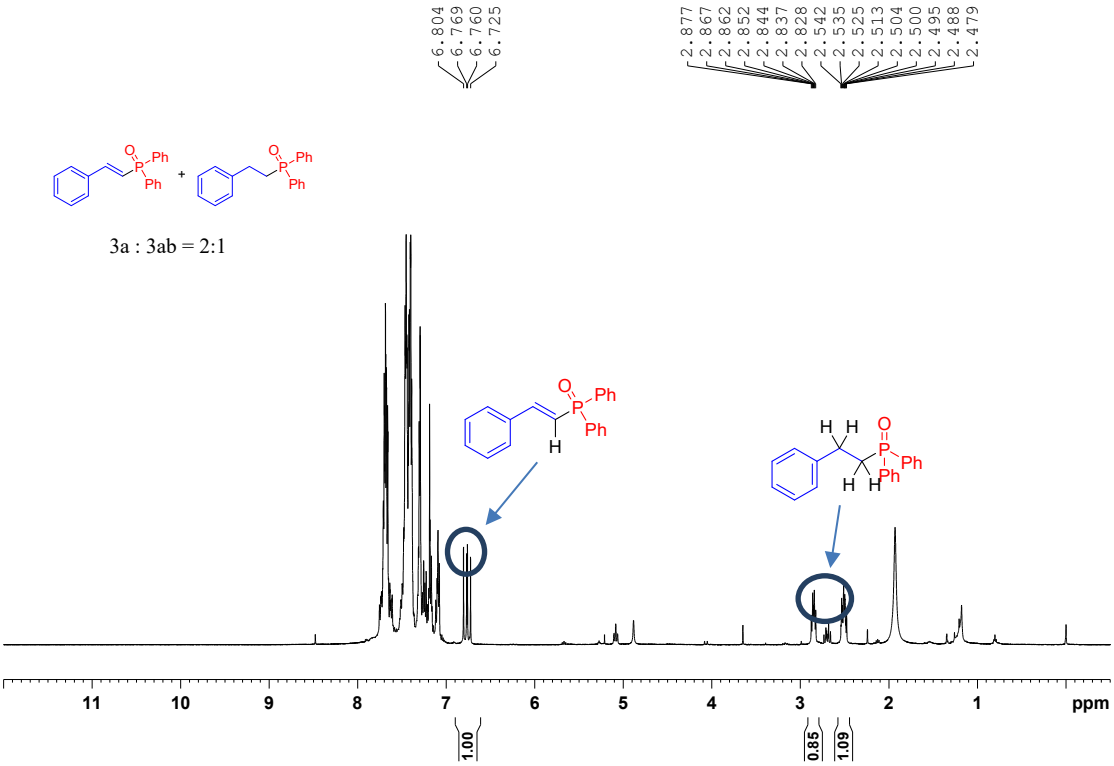
Compound **3a** and **3ab** was prepared from styrene (31.2 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3a** and **3ab** (33 mg, 21.9% and 10.9%; **3a**:**3ab**=2:1) as a mixture.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 31.7; 24.6

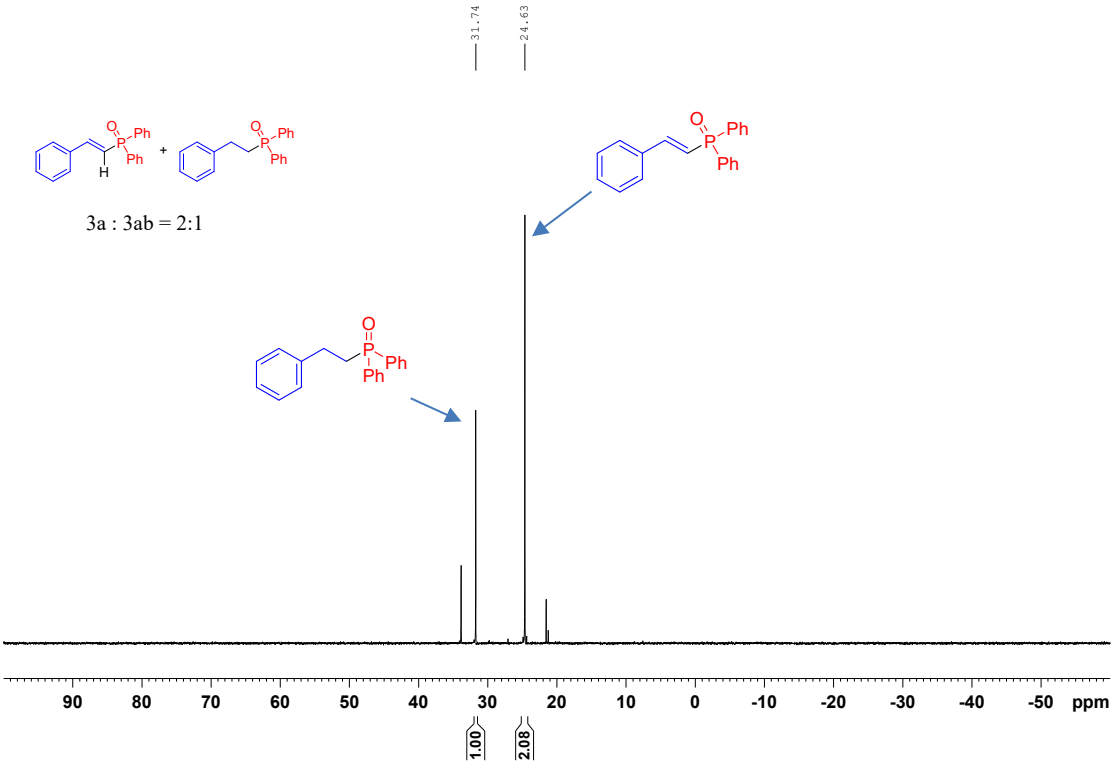
3a: HRMS-ESI: Calcd for $C_{20}H_{18}OP^+$ $[M+H]^+$305.1090, found 305.1084

3ab: **HRMS-ESI:** Calcd for $C_{20}H_{20}OP^+$ $[M+H]^+$307.1246, found 307.1251

^{1}H NMR (500 MHz, $CDCl_3$, 300K) the mixture: 3a:3ab = 2:1

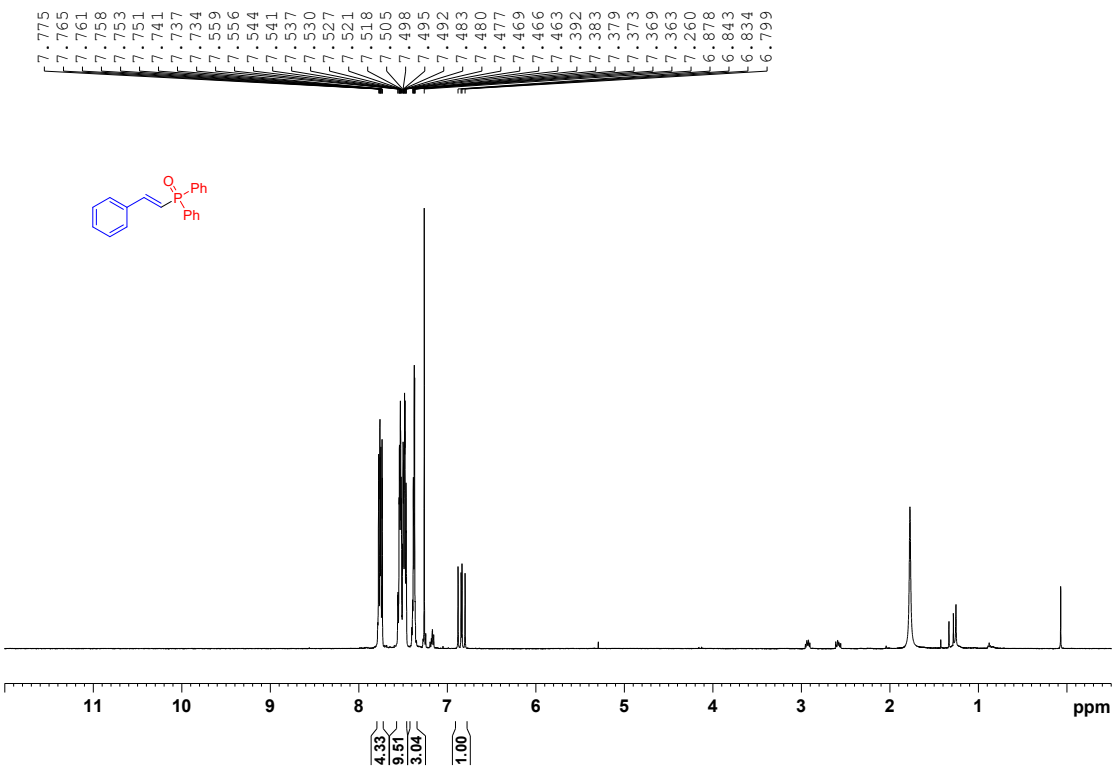


^{31}P NMR (165 MHz, $CDCl_3$, 300K) the mixture: 3a:3ab = 2:1

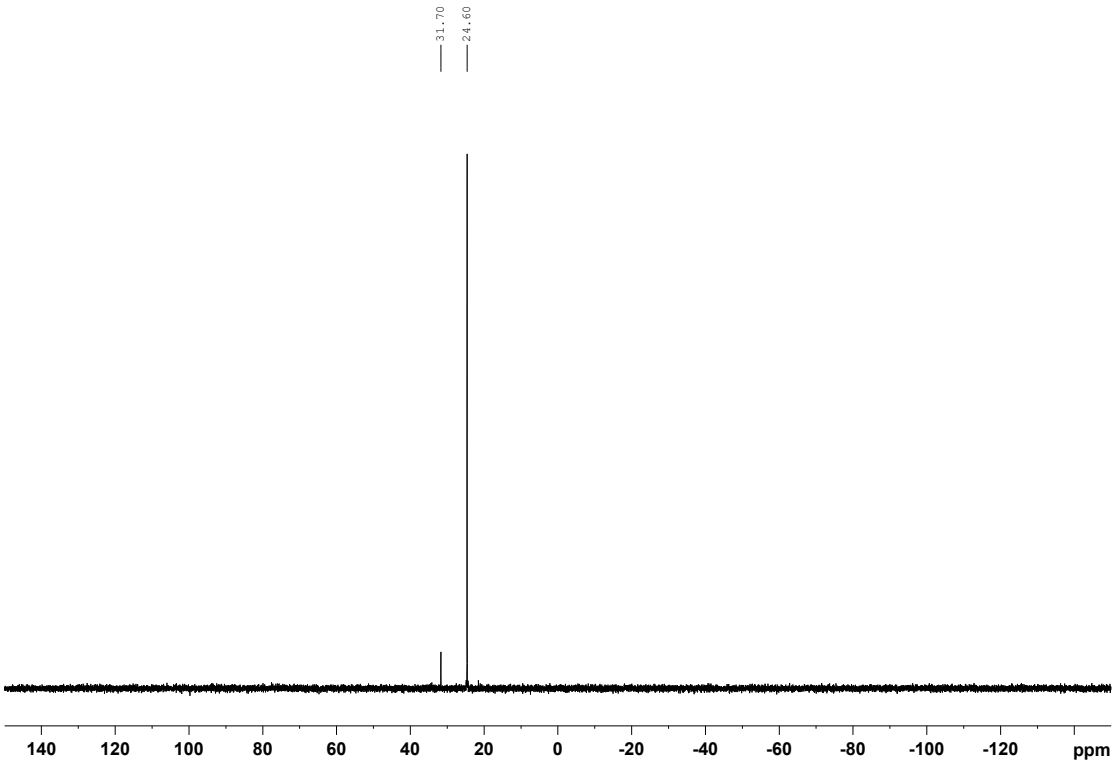


After three rounds of purification:

^{1}H NMR (500 MHz, $CDCl_3$, 300K) the mixture: 3a:3ab = 9:1

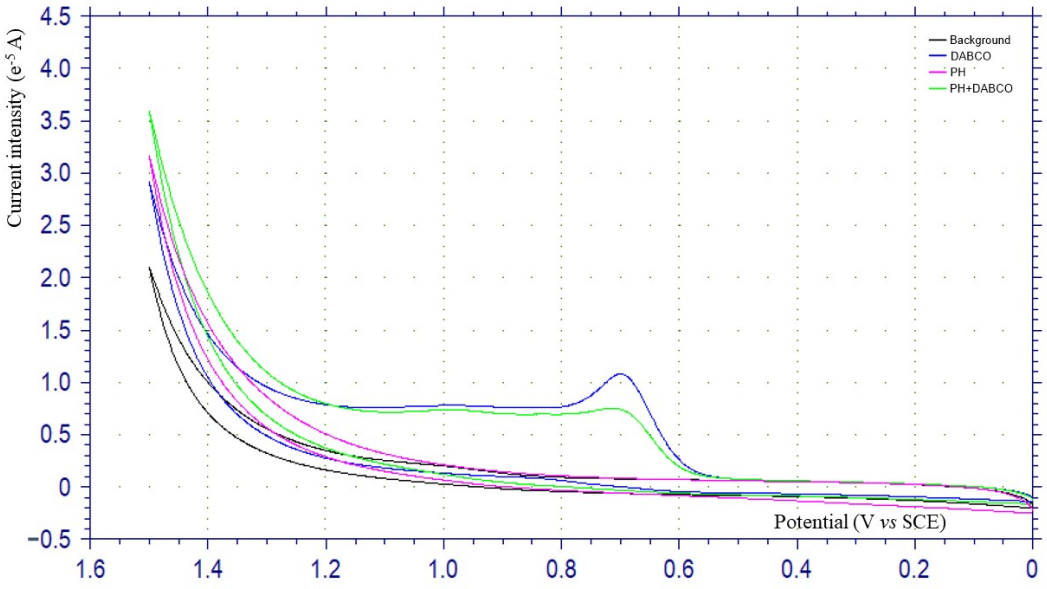


^{31}P NMR (165 MHz, $CDCl_3$, 300K) the mixture: 3a:3ab = 9:1



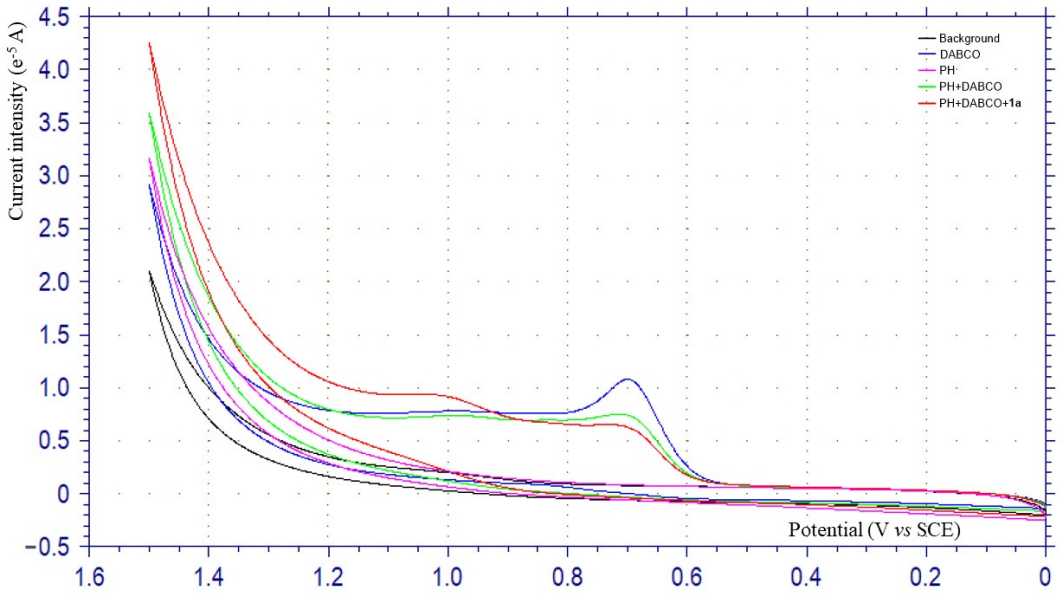
3. Cyclic voltammetry

Cyclic voltammetry was performed with CHI760E Electrochemical Workstation using the cyclic voltammetry mode. A platinum disc (diameter 3 mm) working electrode, a platinum wire counter electrode and a reference electrode (saturated calomel electrode (in a 3.0 M KCl aqueous solution) were used at a scan rate of 100 mV/s. All electrodes are purchased from CH Instruments. The experiments were conducted in a 25 mL four neck vial (undivided cell) without stirring in DMSO (20 mL) with nBu$_4$NPF$_6$ (0.1 M) as electrolyte under Ar. These experiments were carried out at room temperature, starting from 0 V to 1.5 V, positive scan. The working electrode was polished by a commercially available polishing pad and alumina (Al_2O_3) (purchased from CH Instruments), and sonicated in deionized water before data collection. The solution of interest was sparged with Argon for 5 minutes.



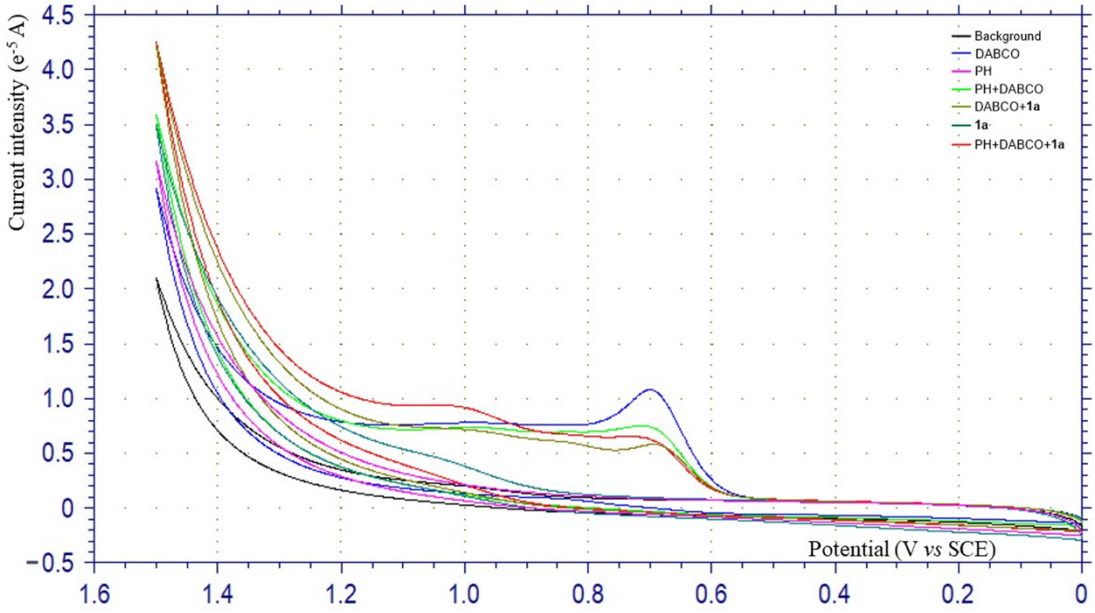
Note: background: 0.1 M n-Bu$_4$NPF$_6$ in DMSO under Ar; DABCO (4.0 mM); diphenylphosphine oxide **2a** (8.0 mM).

Figure S1: *Cyclic voltammetry of DABCO,* ***2a****, and DABCO+****2a*** *in the [0V, +1.5V] range*



Note: background: background: 0.1 M *n*-Bu_4NPF_6 in DMSO under Ar; DABCO (4.0 mM); diphenylphosphine oxide **2a** (8.0 mM); **1a** (2.0 mM).

Figure S2: *Cyclic voltammetry of DABCO,* ***2a****, DABCO+****2a****, DABCO+****2a****+****1a*** *in the [0V, +1.5V] range*

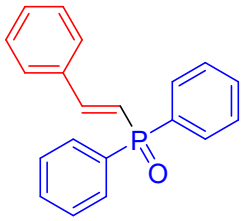


Note: background: background: 0.1 M *n*-Bu_4NPF_6 in DMSO under Ar; DABCO (4.0 mM); diphenylphosphine oxide **2a** (8.0 mM); **1a** (2.0 mM).

Figure S3: *Cyclic voltammetry of DABCO,* ***1a****,* ***2a****, DABCO+****2a****, and DABCO+****1a****+****2a*** *in the [0V, +1.5V] range*

4. Spectral Data

(*E*)-diphenyl(styryl)phosphine oxide (3a)



Compound **3a** was prepared from *trans*-cinnamic acid (**1a**, 44.4 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3a** (73 mg, 0.24 mmol, 80%) as white solid.

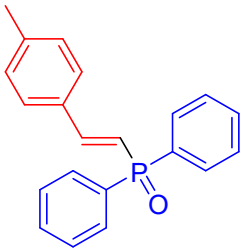
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.78-7.74 (m, 4H), 7.55-7.46 (m, 9H), 7.40-7.37 (m, 3H), 6.84 (dd, *J* = 22.3 Hz, *J* = 17.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 147.7 (d, *J* = 2.8 Hz), 135.3 (d, *J* = 17.4 Hz), 133.1 (d, *J* = 108.5 Hz), 132.0 (d, *J* = 1.4 Hz), 131.5 (d, *J* = 9.8 Hz), 130.2, 129.0, 128.8 (d, *J* = 12.0 Hz), 127.9, 119.4 (d, *J* = 100.9 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.5

HRMS-ESI: Calcd for $C_{20}H_{18}OP^+$ $[M+H]^+$305.1090, found 305.1079

(*E*)-(4-methylstyryl)diphenylphosphine oxide (3b)



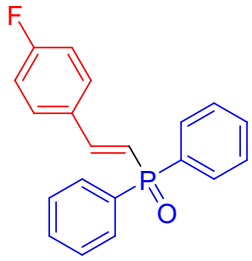
Compound **3b** was prepared from (*E*)-3-(*p*-tolyl)acrylic acid (**1b**, 48.6 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3b** (65 mg, 0.20 mmol, 68%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.77-7.73 (m, 4H), 7.53 (t, *J* = 7.3 Hz, 2H), 7.48 (t, *J* = 7.4 Hz, 5H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.82-6.74 (m, 1H), 2.37 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 147.5 (d, *J* = 3.66 Hz), 140.4, 133.1 (d, *J* = 105.6 Hz), 132.4 (d, *J* = 18.3 Hz), 131.8 (d, *J* = 2.5 Hz), 131.4 (d, *J* = 9.8 Hz), 129.5, 128.6 (d, *J* = 12.1 Hz), 127.7, 117.8 (d, *J* = 105.0 Hz), 21.4.

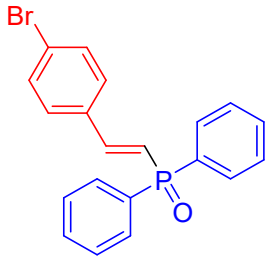
^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.8
HRMS-ESI: Calcd for $C_{21}H_{20}OP^+$ $[M+H]^+$319.1246, found 319.1236

(*E*)-(4-fluorostyryl)diphenylphosphine oxide (3c)



Compound **3c** was prepared from (*E*)-3-(4-fluorophenyl)acrylic acid(**1c**, 49.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3c** (62 mg, 0.19 mmol, 64%) as white solid.
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.79-7.75 (m, 4H), 7.58-7.45 (m, 9H), 7.09 (t, *J* = 8.6 Hz, 2H), 6.77 (dd, *J* = 22.1 Hz, *J* = 17.4 Hz, 1H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 163.9 (d, *J* = 252.0 Hz), 146.4 (d, *J* = 3.6 Hz), 133.0 (d, *J* = 106.3 Hz), 132.1 (d, *J* = 2.6 Hz), 131.6 (d, *J* = 3.4 Hz, overlapped), 131.5 (d, *J* = 10.0 Hz), 129.8 (d, *J* = 8.5 Hz), 128.8 (d, *J* = 12.0 Hz), 119.1 (d, *J* = 102.5 Hz), 116.1 (d, *J* = 22.0 Hz).
^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.4
^{19}F NMR (470 MHz, $CDCl_3$, 300K): δ (ppm) -110.0
HRMS-ESI: Calcd for $C_{20}H_{17}FOP^+$ $[M+H]^+$323.0996, found 323.0984

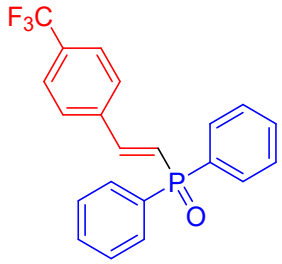
(*E*)-(4-bromostyryl)diphenylphosphine oxide (3d)



Compound **3d** was prepared from (*E*)-3-(4-bromophenyl)acrylic acid (**1d**, 68.1 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3d** (64 mg, 0.17 mmol, 56%) as white solid.
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.76-7.72(m, 4H), 7.57-7.45 (m, 9H), 7.41-7.38 (m, 2H), 6.83 (dd, *J* = 22.0 Hz, *J* = 17.4 Hz, 1H).

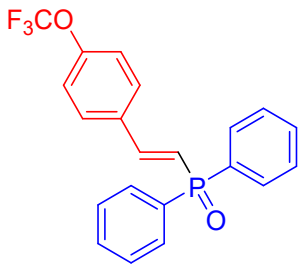
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 146.1 (d, *J* = 3.7 Hz), 134.9 (d, *J* = 17.9 Hz), 132.7 (d, *J* = 106.1 Hz), 132.1, 132.0 (d, *J* = 2.4 Hz), 131.3 (d, *J* = 10.0 Hz), 129.2, 128.7 (d, *J* = 12.4 Hz), 124.3, 120.2 (d, *J* = 103.7 Hz).
^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.2
HRMS-ESI: Calcd for $C_{20}H_{17}{}^{79}BrOP^+$ $[M+H]^+$383.0195, found 383.0178

(*E*)-diphenyl(4-(trifluoromethyl)styryl)phosphine oxide (3e)



Compound **3e** was prepared from (*E*)-3-(4 -(trifluoromethyl)phenyl) acrylic acid (**1e**, 64.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3e** (60.3 mg, 0.16 mmol, 54%) as colorless solid.
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.77-7.73 (m, 4H), 7.62-7.49 (m, 11H), 6.95 (dd, *J* = 21.7 Hz, *J* = 17.4 Hz, 1H).
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 145.9 (d, *J* = 3.2 Hz), 138.5 (d, *J* = 17.3 Hz), 132.4 (d, *J* = 106.4 Hz), 132.2 (d, *J* = 2.4 Hz), 131.7 (d, *J* = 32.8 Hz), 131.5 (d, *J* = 9.8 Hz), 128.9 (d, *J* = 12.0 Hz), 128.0, 126.0 (q, *J* = 3.7 Hz), 123.9 (q, *J* = 272.0 Hz), 122.6 (d, *J* = 101.8 Hz).
^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 23.9
^{19}F NMR (470 MHz, $CDCl_3$, 300K): δ (ppm) -62.8
HRMS-ESI: Calcd for $C_{21}H_{17}F_3OP^+$ $[M+H]^+$373.0964, found 373.0950

(*E*)-diphenyl(4-(trifluoromethoxy)styryl)phosphine oxide (3f)



Compound **3f** was prepared from (*E*)-3-(4- (trifluoromethoxy)phenyl) acrylic acid (**1f**, 69.6 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3f** (38.4 mg, 0.10

mmol, 33%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.76-7.72 (m, 4H), 7.57-7.47 (m, 9H), 7.22 (d, *J* = 8.2 Hz, 2H), 6.82 (dd, *J* = 22.0 Hz, *J* = 17.5 Hz, 1H).

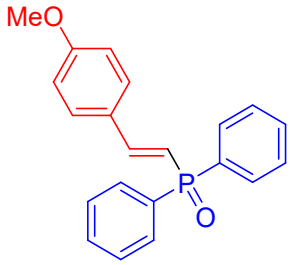
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 150.4, 145.9 (d, *J* = 3.5 Hz), 133.9 (d, *J* = 18.3 Hz), 132.8 (d, *J* = 107.0 Hz), 132.2 (d, *J* = 2.0 Hz), 131.5 (d, *J* = 9.9 Hz), 129.4, 128.8 (d, *J* = 12.1 Hz), 121.3, 120.5 (q, *J* = 257.4 Hz), 120.6 (d, *J* = 104.0 Hz).

^{31}P NMR (162 MHz, CDCl3, 300K): δ (ppm) 24.1

^{19}F NMR (470 MHz, $CDCl_3$, 300K): δ (ppm) -57.8

HRMS-ESI: Calcd for $C_{21}H_{17}F_3O_2P^+$ $[M+H]^+$389.0913, found 389.0897

(*E*)-(4-methoxystyryl)diphenylphosphine oxide (3g)



Compound **3g** was prepared from (*E*)-3-(4-methoxyphenyl)acrylic acid (**1g**, 53.4 mg, 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3g** (34 mg, 0.10 mmol, 34%) as white solid.

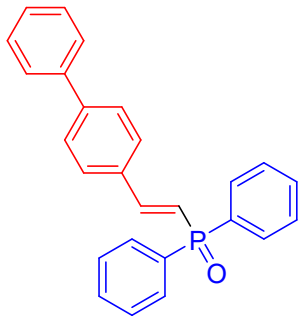
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.74 (dd, *J* = 11.8 Hz, *J* = 7.8 Hz, 4H), 7.53-7.39 (m, 9H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.66 (dd, *J* = 22.1 Hz, *J* = 17.4 Hz, 1H), 3.80 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 161.3, 147.2 (d, *J* = 3.8 Hz), 133.4 (d, *J* = 106.1 Hz), 131.8 (d, *J* = 2.6 Hz), 131.5 (d, *J* = 10.0 Hz), 129.5, 128.7 (d, *J* = 12.4 Hz), 128.1 (d, *J* = 18.4 Hz), 116.3 (d, *J* = 107.1 Hz), 114.3, 55.5.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.8

HRMS-ESI: Calcd for $C_{21}H_{20}OP^+$ $[M+H]^+$335.1195, found 335.1182

(*E*)-(2-([1,1'-biphenyl]-4-yl)vinyl)diphenylphosphine oxide (3h)



Compound **3h** was prepared from (*E*)-3-([1,1'-biphenyl]-3-yl) acrylic acid (**1h**, 67.2 mg. 0.3 mmol), diphenyl phosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1

mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3h** (53.6 mg, 0.14 mmol, 47%) as white solid.

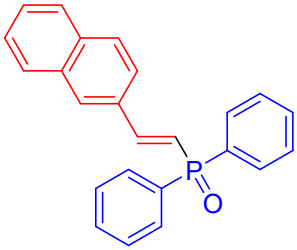
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.78 (dd, *J* = 11.9 Hz, *J* = 7.4 Hz, 4H), 7.63-7.43 (m, 15H), 7.37 (t, *J* = 7.3 Hz, 1H), 6.88 (dd, *J* = 22.2 Hz, *J* = 17.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 147.2 (d, *J* = 3.6 Hz), 143.0, 140.3, 134.2 (d, *J* = 18.5 Hz), 133.1 (d, *J* = 106.0 Hz), 132.1 (d, *J* = 2.6 Hz), 131.5 (d, *J* = 10.0 Hz), 129.0, 128.8 (d, *J* = 12.0 Hz), 128.4, 127.9, 127.6, 127.1, 119.2 (d, *J* = 104.1 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.5

HRMS-ESI: Calcd for $C_{26}H_{22}OP^+$ $[M+H]^+$381.1403, found 381.1388

(*E*)-(2-(naphthalen-2-yl)vinyl)diphenylphosphine oxide (3i)



Compound **3i** was prepared from (*E*)-3-(naphthalen-2-yl)acrylic acid (**1i**, 59.4 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3i** (49.9 mg, 0.14 mmol, 47%) as white solid.

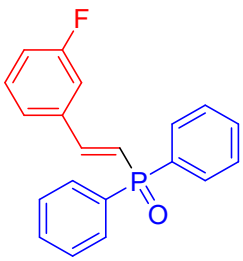
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.93 (s, 1H), 7.87-7.80 (m, 7H), 7.73-7.72 (m, 1H), 7.68-7.64 (m, 1H), 7.60-7.51 (m, 8H), 6.98 (dd, *J* = 22.0 Hz, *J* = 17.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 147.7 (*J* = 3.6 Hz), 134.2, 133.4, 133.1 (*J* = 106.0 Hz), 132.7 (*J* = 18.0 Hz), 132.0 (*J* = 2.1 Hz), 131.6 (*J* = 10.0 Hz), 129.5, 128.8 (*J* = 12.0 Hz), 128.8, 128.7, 127.9, 127.3, 126.8, 123.5, 119.5 (*J* = 105.1 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.6

HRMS-ESI: Calcd for $C_{24}H_{20}OP^+$ $[M+H]^+$355.1246, found 355.1230

(*E*)-(3-fluorostyryl)diphenylphosphine oxide (3j)



Compound **3j** was prepared from (*E*)-3-(3-fluorophenyl)acrylic acid (**1j**, 49.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3j** (45.8 mg, 0.14 mmol, 46%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.77-7.73 (m, 4H), 7.56-7.45 (m, 7H), 7.35-7.32 (m, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.24-7.21 (m, 1H), 7.06 (td, *J* = 8.3 Hz, *J* = 1.9 Hz, 1H), 6.85 (dd, *J* = 22.0 Hz, *J* = 17.3 Hz, 1H).

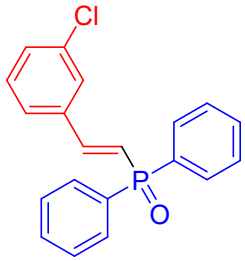
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 163.2 (d, *J* = 247.5 Hz), 146.3, 137.5 (dd, *J* = 18.2 Hz, *J* = 7.4 Hz), 132.8 (d, *J* = 106.5 Hz), 132.1 (d, *J* = 2.3 Hz), 131.5 (d, *J* = 9.8 Hz), 130.6 (d, *J* = 8.5 Hz), 128.8 (d, *J* = 12.4 Hz), 124.0 (d, *J* = 2.6 Hz), 121.1 (d, *J* = 103.2 Hz), 117.1 (d, *J* = 21.4 Hz), 114.1 (d, *J* = 22.0 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 23.9

^{19}F NMR (470 MHz, $CDCl_3$, 300K): δ (ppm) -112.5

HRMS-ESI: Calcd for $C_{20}H_{17}FOP^+$ $[M+H]^+$323.0996, found 323.0984

(*E*)-(3-chlorostyryl)diphenylphosphine oxide (3k)



Compound **3k** was prepared from (*E*)-3-(3-chlorophenyl)acrylic acid (**1k**, 54.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3k** (55.9 mg, 0.17 mmol, 55%) as white solid.

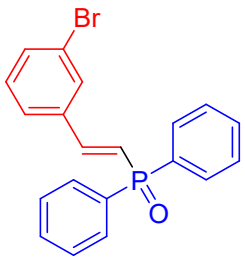
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.77-7.73 (m, 4H), 7.55 (td, *J* = 7.4 Hz, *J* = 1.1 Hz, 2H), 7.51-7.46 (m, 6H), 7.39-7.38 (m, 1H), 7.35-7.30 (m, 2H), 6.86 (dd, *J* = 22.0 Hz, *J* = 17.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 146.1 (d, *J* = 3.2 Hz), 137.1 (d, *J* = 17.2 Hz), 135.0, 132.8 (d, *J* = 107.0 Hz), 132.2 (d, *J* = 1.9 Hz), 131.5 (d, *J* = 9.9 Hz), 130.3, 130.1, 128.9 (d, *J* = 12.2 Hz), 127.5, 126.4, 121.3 (d, *J* = 100.5 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.0

HRMS-ESI: Calcd for $C_{20}H_{17}ClOP^+$ $[M+H]^+$339.0700, found 339.0690

(*E*)-(3-bromostyryl)diphenylphosphine oxide (3l)



Compound **3l** was prepared from (*E*)-3-(3-bromophenyl)acrylic acid (**1l**, 68.1 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3l** (50.6 mg, 0.13 mmol, 44%) as white solid.

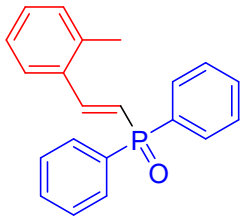
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.75-7.71 (m, 4H), 7.65 (s, 1H), 7.54-7.40 (m, 9H), 7.22 (t, *J* = 7.8 Hz, 1H), 6.84 (dd, *J* = 22.0 Hz, *J* = 17.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 145.9 (d, *J* = 3.5 Hz), 137.3 (d, *J* = 18.1 Hz), 133.0, 132.7 (d, *J* = 106.7 Hz), 132.1 (d, *J* = 2.5 Hz), 131.5 (d, *J* = 10.0 Hz), 130.5, 130.3, 128.8 (d, *J* = 12.0 Hz), 126.8, 123.1, 121.3 (d, *J* = 102.9 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.0

HRMS-ESI: Calcd for $C_{20}H_{17}{}^{79}BrOP^+$ $[M+H]^+$383.0195, found 383.0184

(*E*)-(2-methylstyryl)diphenylphosphine oxide (3m)



Compound **3m** was prepared from (*E*)-3-(o-tolyl)acrylic acid (**1m**, 48.6 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3m** (68.7 mg, 0.22 mmol, 74%) as white solid.

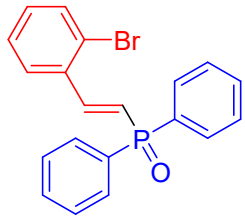
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.80-7.73 (m, 5H), 7.58 (d, *J* = 7.4 Hz, 1H), 7.55-7.46 (m, 6H), 7.27-7.24 (m, 1H), 7.20 (q, *J* = 7.5 Hz, 2H), 6.77 (dd, *J* = 23.2 Hz, *J* = 17.3 Hz, 1H), 2.37 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 145.5 (d, *J* = 3.9 Hz), 137.4, 134.4 (d, *J* = 16.9 Hz), 133.2 (d, *J* = 105.0 Hz), 132.0 (d, *J* = 2.5 Hz), 131.5 (d, *J* =10.0 Hz), 130.9, 130.0, 128.7 (d, *J* = 11.9 Hz), 126.4, 126.2, 121.2 (d, *J* = 104.0 Hz), 19.8.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.6

HRMS-ESI: Calcd for $C_{21}H_{20}OP^+$ $[M+H]^+$319.1246, found 319.1236

(*E*)-(2-bromostyryl)diphenylphosphine oxide (3n)



Compound **3n** was prepared from (*E*)-3-(2-bromophenyl)acrylic acid (**1n**, 68.1 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3n** (60.9 mg, 0.16 mmol, 53%) as white solid.

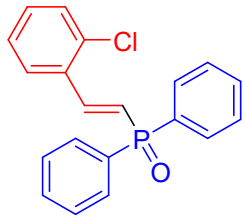
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.79-7.75 (m, 4H), 7.69 (dd, *J* = 19.6 Hz, *J* = 17.1 Hz, 1H), 7.61 (dd, *J* = 7.7 Hz, *J* = 1.1 Hz, 1H), 7.58-7.53 (m, 3H), 7.50-7.47 (m, 4H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.19 (td, *J* = 7.3 Hz, *J* = 1.3 Hz, 1H), 6.81 (td, *J* = 20.0 Hz, *J* = 17.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 146.3 (d, *J* = 5.5 Hz), 135.4 (d, *J* = 18.2 Hz), 133.5, 132.5 (d, *J* = 104.0 Hz), 132.1 (d, *J* = 2.6 Hz), 131.6 (d, *J* = 10.0 Hz), 131.1, 128.8 (d, *J* = 12.0 Hz), 127.9, 127.8, 124.9, 123.5 (d, *J* = 102.6 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.8

HRMS-ESI: Calcd for $C_{20}H_{17}{}^{79}BrOP^+$ $[M+H]^+$383.0195, found 383.0179

(*E*)-(2-chlorostyryl)diphenylphosphine oxide (3o)



Compound **3o** was prepared from (*E*)-3-(2-chlorophenyl)acrylic acid (**1o**, 54.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3o** (55.9 mg, 0.17 mmol, 55%) as white solid.

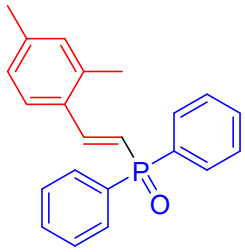
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.82-7.74 (m, 5H), 7.64-7.62 (m, 1H), 7.57-7.47 (m, 6H), 7.40-7.38 (m, 1H), 7.30-7.25 (m, 2H), 6.88 (dd, *J* = 20.6 Hz, *J* = 17.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 143.8 (d, *J* = 5.2 Hz), 134.7, 133.6 (d, *J* = 17.9 Hz), 132.6 (d, *J* = 102.6 Hz), 132.1 (d, *J* = 2.1 Hz), 131.6 (d, *J* = 9.9 Hz), 131.0, 130.3, 128.8 (d, *J* = 12.3 Hz), 127.9, 127.2, 123.2 (d, *J* = 102.3 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.7

HRMS-ESI: Calcd for $C_{20}H_{17}ClOP^+$ $[M+H]^+$339.0700, found 339.0690

(*E*)-(2,4-dimethylstyryl)diphenylphosphine oxide (3p)



Compound **3p** was prepared from (*E*)-3-(2,4-dimethylphenyl)acrylic acid (**1p**, 52.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3p** (39.9 mg, 0.12 mmol, 40%) as white solid.

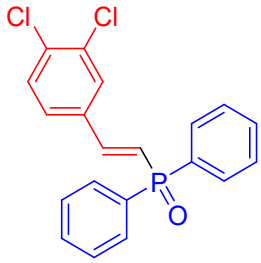
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.78-7.72 (m, 5H), 7.55-7.46 (m, 7H), 7.03-7.01 (m, 2H), 6.72 (dd, *J* = 23.2 Hz, *J* = 17.3 Hz, 1H), 2.33 (s, 3H), 2.32 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 145.5 (d, *J* = 3.8 Hz), 140.2, 137.4, 133.3 (d, *J* = 105.1 Hz), 131.9 (d, *J* = 2.5 Hz), 131.7, 131.5 (d, *J* = 9.9 Hz), 131.46 (overlapped), 128.7 (d, *J* = 12.2 Hz), 127.2, 126.2, 119.2 (d, *J* = 104.8 Hz), 21.4, 19.7.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 25.0

HRMS-ESI: Calcd for $C_{22}H_{22}OP^+$ $[M+H]^+$333.1403, found 333.1390

(*E*)-(3,4-dichlorostyryl) diphenylphosphine oxide (3q)



Compound **3q** was prepared from (*E*)-3-(3,4-dichlorophenyl) acrylic acid (**1q**, 65.1 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3q** (49.3 mg, 0.13 mmol, 44%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.76-7.72 (m, 4H), 7.60 (d, *J* = 1.9 Hz, 1H), 7.55 (td, *J* = 7.4 Hz, *J* = 1.2 Hz, 2H), 7.50-7.38 (m, 6H), 7.33 (dd, *J* = 8.3 Hz, *J* = 2.0 Hz, 1H), 6.85 (dd, *J* = 21.6 Hz, *J* = 17.4 Hz, 1H).

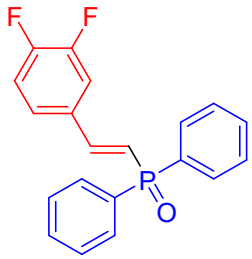
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 144.9 (d, *J* = 3.6 Hz), 135.3 (d, *J* = 18.6 Hz), 134.1,

133.3, 132.6 (d, J = 106.3 Hz), 132.2 (d, J = 2.2 Hz), 131.5 (d, J = 10.0 Hz), 131.0, 129.3, 128.9 (d, J = 12.2 Hz), 127.1, 121.9 (d, J = 102.0 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 23.7

HRMS-ESI: Calcd for $C_{20}H_{16}Cl_2OP^+$ $[M+H]^+$373.0310, found 373.0295

(*E*)-(3,4-difluorostyryl)diphenylphosphine oxide (3r)



Compound **3r** was prepared from (*E*)-3-(3,4-difluorophenyl) acrylic acid (**1r**, 55.2 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3r** (50 mg, 0.15 mmol, 49%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.76-7.72 (m, 4H), 7.55 (td, J = 7.3 Hz, J = 1.4 Hz, 2H), 7.50-7.40 (m, 5H), 7.37-7.33 (m, 1H), 7.26-7.23 (m, 1H), 7.19-7.14 (m, 1H), 6.76 (dd, J = 21.8 Hz, J = 17.3 Hz, 1H).

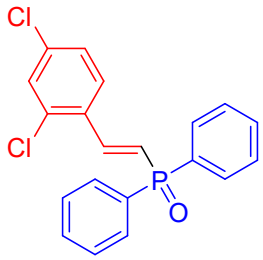
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 152.1 (dd, J = 97.0 Hz, J = 13.0 Hz), 150.1 (dd, J = 93.6 Hz, J = 13.0 Hz), 145.3, 132.7 (d, J = 106.3 Hz), 132.5 (td, J = 17.8 Hz, J = 5.2 Hz), 132.2 (d, J = 2.2 Hz), 131.5 (d, J = 9.9 Hz), 128.9 (d, J = 12.1 Hz), 124.7 (d, J = 3.1 Hz), 120.9 (d, J = 101.6 Hz), 117.5 (d, J = 17.5 Hz), 116.1 (d, J = 17.1 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 23.8

^{19}F NMR (470 MHz, $CDCl_3$, 300K): δ (ppm) -134.5 (d, J = 20.9 Hz), -136.6 (d, J = 20.9 Hz).

HRMS-ESI: Calcd for $C_{20}H_{16}F_2OP^+$ $[M+H]^+$341.0901, found 341.0889.

(*E*)-(2,4-dichlorostyryl) diphenylphosphine oxide (3s)



Compound **3s** was prepared from (*E*)-3-(2,4-dichlorophenyl)acrylic acid (**1s**, 65.1 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column

chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3s** (47.0 mg, 0.13 mmol, 42%) as white solid.

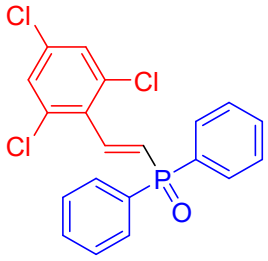
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.78-7.68 (m, 5H), 7.58-7.48 (m, 7H), 7.42 (d, *J* = 2.1Hz, 1H), 7.26 (dd, *J* = 8.4 Hz, *J*=1.9 Hz, 1H), 6.86 (dd, *J* = 20.6 Hz, *J* = 17.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 142.5 (d, *J* = 5.2 Hz), 136.3, 135.3, 132.4 (d, *J* = 106.4 Hz), 132.2 (d, *J* = 2.5 Hz), 132.1, 131.6 (d, *J* = 9.9 Hz), 130.1, 128.9 (d, *J* = 12.0 Hz), 128.6, 127.6, 123.8 (d, *J* = 101.9 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.6

HRMS-ESI: Calcd for $C_{20}H_{16}Cl_2OP^+$ $[M+H]^+$373.0310, found373.0324.

(*E*)-diphenyl(2,4,6-trichlorostyryl)phosphine oxide (3t)



Compound **3t** was prepared from (*E*)-3-(2,4,6-trichlorophenyl)acrylic acid (**1t**, 75.4 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3t** (52.6 mg, 0.13 mmol, 43%) as white solid.

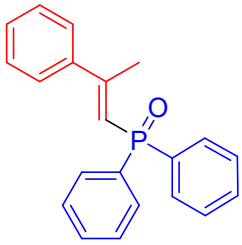
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.80-7.76 (m, 4H), 7.56 (td, *J* = 7.4 Hz, *J* = 1.3 Hz, 2H), 7.51-7.48 (m, 4H), 7.43 (dd, *J* = 20.3 Hz, *J* = 17.8 Hz, 1H), 7.37 (s, 2H), 7.03 (dd, *J* = 21.9 Hz, *J* = 17.8 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 142.5 (d, *J* = 4.5 Hz), 136.3, 135.3, 132.4 (d, *J* = 104.7 Hz), 132.2, 132.1, 131.6 (d, *J* = 9.8 Hz), 130.1, 128.9 (d, *J* = 12.1 Hz), 128.7, 127.6, 123.8 (d, *J* = 105.4 Hz)

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.1

HRMS-ESI: Calcd for $C_{20}H_{15}Cl_3OP^+$ $[M+H]^+$406.9921, found 406.9932.

(*E*)-diphenyl(2-phenylprop-1-en-1-yl)phosphine oxide (3u)



Compound **3u** was prepared from (*E*)-3-phenylbut-2-enoic acid (**1u**, 48.6 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode

(General Procedure A). The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3u** (49.7 mg, 0.16 mmol, 52%) as white solid.

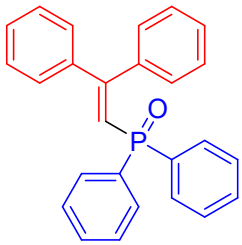
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.81-7.77 (dd, *J* = 11.5 Hz, *J* = 7.8 Hz, 4H), 7.50-7.47 (m, 8H), 7.37-7.36 (m, 3H), 6.40 (d, *J* = 23.6 Hz, 1H), 2.50 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 159.5 (d, *J* = 2.3 Hz), 142.3 (d, *J* = 17.1 Hz), 134.8 (d, *J* = 104.4 Hz), 131.7 (d, *J* = 2.2 Hz), 131.1 (d, *J* = 9.7 Hz), 129.3, 128.8, 128.7 (d, *J* = 8.0 Hz), 126.1, 118.5 (d, *J* = 105.0 Hz), 19.8 (d, *J* = 7.8 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 21.6

HRMS-ESI: Calcd for $C_{21}H_{20}OP^+$ $[M+H]^+$319.1246, found 319.1236

(2,2-diphenylvinyl) diphenyl phosphine oxide (3v)



Compound **3v** was prepared from 3,3-diphenylacrylic acid (**1v**, 67.3 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3v** (39.9 mg, 0.11 mmol, 35%) as white solid.

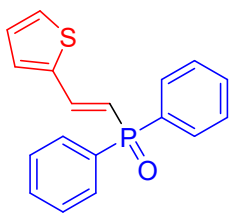
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.68 (dd, *J* = 11.6 Hz, *J* = 7.3 Hz, 4H), 7.39-7.30 (m, 11H), 7.22 (d, *J* = 7.0 Hz, 2H), 7.15-7.07 (m, 3H), 6.79 (d, *J* = 18.3 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 162.1, 142.0 (d, *J* = 16.1 Hz), 138.1(d, *J* = 6.8 Hz), 134.5 (d, *J* = 106.1 Hz), 131.2, 131.0 (d, *J* = 9.3 Hz), 130.4, 129.6, 128.7, 128.5 (d, *J* = 8.4 Hz), 128.4, 128.3, 123.7, 120.5 (d, *J* = 105.5 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 18.7

HRMS-ESI: Calcd for $C_{26}H_{22}OP^+$ $[M+H]^+$381.1403, found 381.1388

(*E*)-diphenyl(2-(thiophen-2-yl)vinyl)phosphine oxide (3w)



Compound **3w** was prepared from (*E*)-3-(thiophen-2-yl)acrylic acid (**1w**, 46.2 mg, 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode

(General Procedure A). The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3w** (52.7 mg, 0.17 mmol, 55%) as white solid.

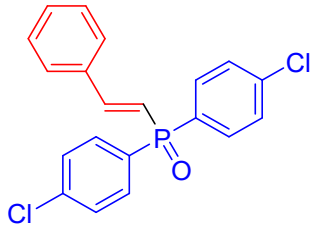
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.74 (dd, *J* = 11.8 Hz, *J* = 7.6 Hz, 4H), 7.60 (d, *J* = 17.9 Hz, 1H), 7.54 (t, *J* = 7.1 Hz, 2H), 7.48 (t, *J* = 6.8 Hz, 4H), 7.35 (d, *J* = 5.0 Hz, 1H), 7.19 (d, *J* = 3.3 Hz, 1H), 7.03 (dd, *J* = 4.8 Hz, *J* = 3.8 Hz, 1H), 6.58 (dd, *J* = 21.1 Hz, *J* = 17.6 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 141.0, 140.8, 140.1 (d, *J* = 3.8 Hz), 133.0 (d, *J* = 106.3 Hz), 132.0 (d, *J* = 2.0 Hz), 131.5 (d, *J* = 9.8 Hz), 130.3, 128.8 (d, *J* = 12.1 Hz), 128.2 (d, *J* = 5.0 Hz), 117.8 (d, *J* = 106.5 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.1

HRMS-ESI: Calcd for $C_{18}H_{16}OPS^+$ $[M+H]^+$311.0654, found 311.0642

(*E*)-bis(4-chlorophenyl)(styryl)phosphine oxide (3x)



Compound **3x** was prepared from *trans*-cinnamic acid (**1x**, 44.4 mg. 0.3 mmol), bis(4-chlorophenyl)phosphine oxide (243.9 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3x** (54.9 mg, 0.15 mmol, 49%) as white solid.

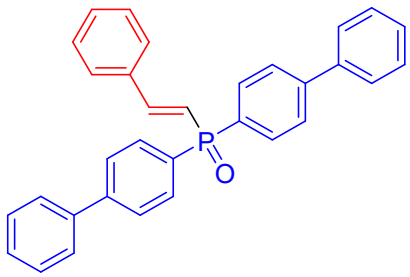
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.67 (dd, *J* = 11.7 Hz, *J* = 8.4 Hz, 4H), 7.53-7.50 (m, 3H), 7.47 (dd, *J* = 8.4 Hz, *J* = 2.1 Hz, 4H), 7.40-7.38 (m, 3H), 6.77 (dd, *J* = 23.0 Hz, *J* = 17.4 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 148.7, 138.9, 134.9 (d, *J* = 18.3 Hz), 132.9 (d, *J* = 10.5 Hz), 131.2 (d, *J* = 109.3 Hz), 130.6, 129.3 (d, *J* = 12.4 Hz), 129.1, 128.0, 118.1 (d, *J* = 106.9 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 23.2

HRMS-ESI: Calcd for $C_{20}H_{16}Cl_2OP^+$ $[M+H]^+$373.0310, found 373.0294

(*E*)-di([1,1'-biphenyl]-4-yl)(styryl)phosphine oxide (3y)



Compound **3y** was prepared from *trans*-cinnamic acid (**1y**, 44.4 mg. 0.3 mmol), di([1,1'-biphenyl]-4-yl)phosphine oxide (318.9 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **3y** (68.5 mg, 0.15 mmol, 50%) as white solid.

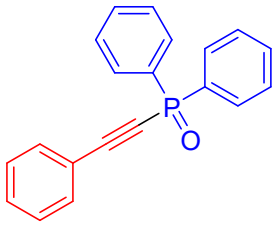
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.87 (dd, *J* = 11.6 Hz, *J* = 8.0 Hz, 4H), 7.72 (d, *J* = 6.4 Hz, 4H), 7.62 (d, *J* = 7.4 Hz, 4H), 7.58-7.55 (m, 3H), 7.47 (t, *J* = 7.5 Hz, 4H), 7.41-7.39 (m, 5H), 6.92 (dd, *J* = 22.4 Hz, *J* = 17.5 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 147.7 (d, *J* = 3.6 Hz), 144.9 (d, *J* = 2.6 Hz), 140.1, 135.3 (d, *J* = 18.3 Hz), 132.1 (d, *J* = 10.2 Hz), 131.7 (d, *J* = 107 Hz), 130.3, 129.1, 129.0 (d, *J* = 9.4 Hz), 128.3, 128.0, 127.5 (d, *J* = 12.6 Hz), 127.4, 119.4 (d, *J* = 104.5 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 24.2

HRMS-ESI: Calcd for $C_{32}H_{26}OP^+$ $[M+H]^+$457.1716, found 457.1696

Diphenyl(phenylethynyl)phosphine oxide (5a)



Compound **5a** was prepared from 3-phenylpropiolic acid (**4a**, 43.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5a** (56 mg, 0.19 mmol, 62%) as white solid.

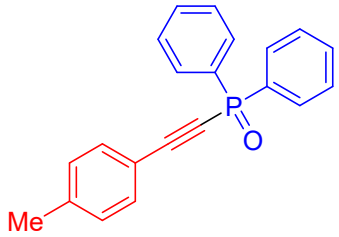
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.92-7.88 (m, 4H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.55 (td, *J* = 7.5 Hz, *J* = 1.1 Hz, 2H), 7.51-7.43 (m, 5H), 7.37 (t, *J* = 7.9 Hz, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 133.4, 132.4 (d, *J* = 1.4 Hz), 132.2 (d, *J* = 2.6 Hz), 130.8 (d, *J* = 11.2 Hz), 130.7, 128.7, 128.5 (d, *J* = 4.4 Hz), 119.8 (d, *J* = 3.8 Hz), 105.4 (d, *J* = 30.4 Hz), 80.8 (d, *J* = 170.2 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.3

HRMS-ESI: Calcd for $C_{20}H_{16}OP^+$ $[M+H]^+$303.0933, found 303.0921

diphenyl(p-tolylethynyl)phosphine oxide (5b)



Compound **5b** was prepared from 3-(*p*-tolyl)propiolic acid (**4b**, 48.0 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5b** (43 mg, 0.14 mmol, 45%) as white solid.

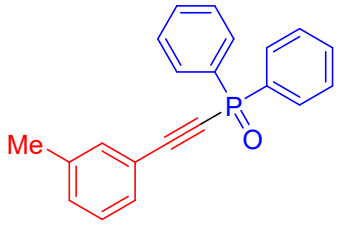
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.91-7.87 (m, 4H), 7.51 (td, *J* = 7.5 Hz, *J* = 1.4 Hz, 2H), 7.48-7.44 (m, 6H), 7.15 (d, *J* = 7.9 Hz, 2H), 2.34 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 141.4, 133.2 (d, *J* = 122.4 Hz), 132.5 (d, *J* = 1.5 Hz), 132.2 (d, *J* = 2.7 Hz), 130.9 (d, *J* = 11.1 Hz), 129.3, 128.6 (d, *J* = 13.2 Hz), 116.8 (d, *J* = 3.8 Hz), 106.0 (d, *J* = 30.6 Hz), 82.3 (d, *J* = 171.9 Hz), 21.7.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.2

HRMS-ESI: Calcd for $C_{21}H_{18}OP^+$ $[M+H]^+$317.1090, found 317.1079

diphenyl(m-tolylethynyl)phosphine oxide (5c)



Compound **5c** was prepared from 3-(*m*-tolyl)propiolic acid (**4c**, 48.0 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5c** (39 mg, 0.12 mmol, 41%) as white solid.

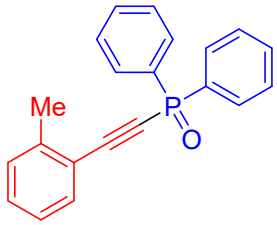
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.92-7.88 (m, 4H), 7.55 (td, *J* = 5.9 Hz, *J* = 1.6 Hz 2H), 7.51-7.47 (m, 4H), 7.42-7.40 (m, 2H), 7.27-7.26 (m, 2H), 2.35 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 138.6, 133.3 (d, *J* = 122.3 Hz), 133.1 (d, *J* = 1.4 Hz), 132.3 (d, *J* = 2.7 Hz), 132.7, 131.1 (d, *J* = 11.4 Hz), 129.8 (d, *J* = 1.4 Hz), 128.8 (d, *J* = 13.5 Hz), 128.6, 119.9 (d, *J* = 4.2 Hz), 105.9 (d, *J* = 30.0 Hz), 82.6 (d, *J* = 170.8 Hz), 21.3.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.3

HRMS-ESI: Calcd for $C_{21}H_{18}OP^+$ $[M+H]^+$317.1090, found 317.1079

diphenyl(o-tolylethynyl)phosphine oxide (5d)



Compound **5d** was prepared from 3-(*o*-tolyl)propiolic acid (**4d**, 48.0 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5d** (49 mg, 0.16 mmol, 52%) as white solid.

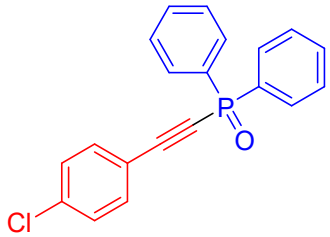
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.93-7.89 (m, 4H), 7.55 (td, *J* = 7.6 Hz, *J* = 1.4 Hz, 3H), 7.51-7.47 (m, 4H), 7.33 (td, *J* = 7.6 Hz, *J* = 1.2 Hz, 1H), 7.24 (d, *J* = 7.7 Hz, 1H), 7.19 (t, *J* =7.5 Hz, 1H), 2.47 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 141.9, 133.3 (d, *J* = 122.7 Hz), 133.1 (d, *J* = 1.6 Hz), 132.3 (d, *J* = 3.0 Hz), 131.1 (d, *J* = 11.2 Hz), 130.8, 129.9, 128.8 (d, *J* = 13.6 Hz), 125.9, 119.9 (d, *J* = 4.2 Hz), 104.8 (d, *J* = 30.1 Hz), 86.7 (d, *J* = 170.0 Hz), 20.8.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.2

HRMS-ESI: Calcd for $C_{21}H_{18}OP^+$ $[M+H]^+$317.1090, found 317.1079

((4-chlorophenyl)ethynyl)diphenylphosphine oxide (5e)



Compound **5e** was prepared from 3-(4-chlorophenyl)propiolic acid (**4e**, 54.0 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5e** (40 mg, 0.12 mmol, 40%) as white solid.

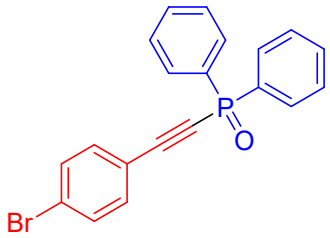
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.91-7.86 (m, 4H), 7.57 (td, *J* = 7.6 Hz, *J* = 1.3 Hz, 2H), 7.53-7.48 (m, 6H), 7.36 (d, *J* = 8.5 Hz, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 137.2, 133.9 (d, *J* = 1.3 Hz), 132.9 (d, *J* = 122.2 Hz), 132.5 (d, *J* = 2.6 Hz), 131.1 (d, *J* = 13.1 Hz), 129.2, 128.8 (d, *J* = 13.5 Hz), 118.5 (d, *J* = 4.0 Hz), 104.2 (d, *J* = 29.0 Hz), 84.1 (d, *J* = 169.8 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.4

HRMS-ESI: Calcd for $C_{20}H_{15}ClOP^+$ $[M+H]^+$337.0544, found 337.0532

((4-bromophenyl)ethynyl)diphenylphosphine oxide (5f)



Compound **5f** was prepared from 3-(4-bromophenyl)propiolic acid (**4f**, 67.5 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5f** (48 mg, 0.13 mmol, 42%) as white solid.

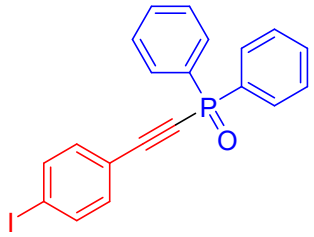
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.89 (dd, *J* = 13.9 Hz, *J* = 7.4 Hz, 4H), 7.55 (d, *J* = 6.5 Hz, 2H), 7.52-7.47 (m, 6H), 7.44 (d, *J* = 8.4 Hz, 2H).

^{13}C NMR (125 MHz, CDCl3, 300K): δ (ppm) 133.9, 132.9 (d, *J* = 122.4 Hz), 132.5 (d, *J* = 2.7 Hz), 132.1, 131.1 (d, *J* = 11.1 Hz), 128.8 (d, *J* = 13.6 Hz), 125.6, 118.9 (d, *J* = 3.7 Hz), 104.2 (d, *J* = 30.4 Hz), 84.3 (d, *J* = 166.8 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.3

HRMS-ESI: Calcd for $C_{20}H_{14}{}^{79}BrNaOP^+$ $[M+Na]^+$402.9858, found 402.9842

((4-iodophenyl)ethynyl)diphenylphosphine oxide (5g)



Compound **5g** was prepared from 3-(4-iodophenyl)propiolic acid (**4g**, 81.6 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5g** (45 mg, 0.11 mmol, 35%) as white solid.

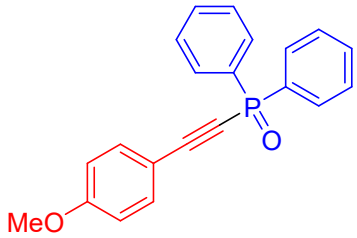
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.90-7.86 (m, 4H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.55 (td, *J* = 7.6 Hz, *J* = 1.4 Hz, 2H), 7.51-7.47 (m, 4H), 7.30 (d, *J* = 8.4 Hz, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 138.0, 133.8, 132.9 (d, *J* = 122.1 Hz), 132.5 (d, *J* = 2.9 Hz), 131.1 (d, *J* = 11.0 Hz), 128.8 (d, *J* = 13.7 Hz), 119.5 (d, *J* = 3.8 Hz), 104.3 (d, *J* = 29.3 Hz), 97.6, 84.5 (d, *J* = 166.8 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.3

HRMS-ESI: Calcd for $C_{20}H_{15}IOP^+$ $[M+H]^+$428.9900, found 428.9885

((4-methoxyphenyl)ethynyl)diphenylphosphine oxide (5h)



Compound **5h** was prepared from 3-(4-methoxyphenyl)propiolic acid (**4h**, 52.8 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5h** (45 mg, 0.14 mmol, 46%) as white solid.

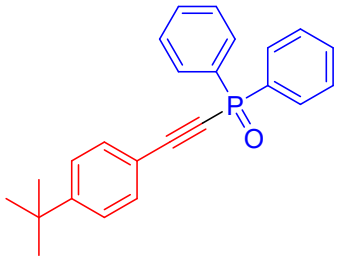
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.91-7.87 (m, 4H), 7.54-7.45 (m, 8H), 6.87 (d, *J* = 7.0 Hz, 2H), 3.81 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 161.5, 134.4 (d, *J* = 1.6 Hz), 133.4 (d, *J* = 121.9 Hz), 132.2 (d, *J* = 2.8 Hz), 131.0 (d, *J* = 11.1 Hz), 128.7 (d, *J* = 13.4 Hz), 114.3, 111.8 (d, *J* = 4.2 Hz), 106.3 (d, *J* = 31.1 Hz), 81.8 (d, *J* = 173.1 Hz), 55.5.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.2

HRMS-ESI: Calcd for $C_{21}H_{18}O_2P^+$ $[M+H]^+$333.1039, found 333.1029

((4-(tert-butyl)phenyl)ethynyl)diphenylphosphine oxide (5i)



Compound **5i** was prepared from 3-(4-(*tert*-butyl)phenyl)propiolic acid (**4i**, 60.6 mg, 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5i** (53 mg, 0.15 mmol, 49%) as white solid.

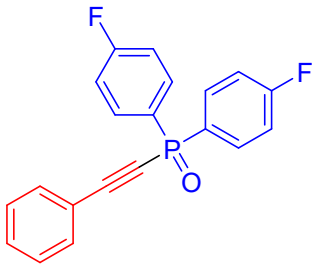
^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.90 (dd, *J* = 13.2 Hz, *J* = 7.2 Hz, 4H), 7.55-7.53 (d, *J* = 7.7 Hz, 4H), 7.50-7.49 (m, 4H), 7.40 (d, *J* = 7.9 Hz, 2H), 1.31 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 154.5, 133.3 (d, *J* = 122.2 Hz), 132.5 (d, *J* = 1.4 Hz), 132.3 (d, *J* = 2.8 Hz), 131.1 (d, *J* = 11.1 Hz), 138.7 (d, *J* = 13.3 Hz), 125.7, 116.9 (d, *J* = 4.0 Hz), 106.1 (d, *J* = 30.5 Hz), 82.3 (d, *J* = 171.5 Hz), 35.2, 31.1.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 8.3

HRMS-ESI: Calcd for $C_{24}H_{24}OP^+$ $[M+H]^+$359.1559, found 359.1546

bis(4-fluorophenyl)(phenylethynyl)phosphine oxide (5j)



Compound **5j** was prepared from 3-phenylpropiolic acid (**4j**, 43.8 mg, 0.3 mmol), bis(4-fluorophenyl)phosphine oxide (214.2 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5j** (30 mg, 0.09 mmol, 30%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.91-7.86 (m, 4H), 7.59-7.58 (m, 2H), 7.46 (t, J = 7.4 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.19 (td, J = 8.7 Hz, J = 1.2 Hz, 4H).

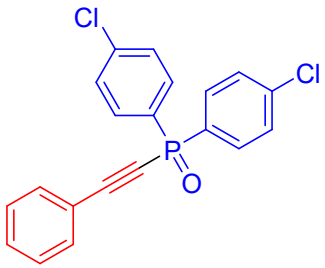
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 165.4 (dd, J = 254.1 Hz, J = 3.1 Hz), 133.6 (dd, J = 12.9 Hz, J = 8.8 Hz), 132.7 (d, J = 1.6 Hz), 131.1, 129.0 (dd, J = 126.3 Hz, J = 3.4 Hz), 128.8, 119.7 (d, J = 4.2 Hz), 116.3 (dd, J = 21.6 Hz, J = 15.1 Hz), 106.2 (d, J = 31.2 Hz), 82.5 (d, J = 173.4 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 6.2

^{19}F NMR (470 MHz, $CDCl_3$, 300K): δ (ppm) -105.7

HRMS-ESI: Calcd for $C_{20}H_{14}F_2OP^+$ $[M+H]^+$339.0745, found 339.0736

bis(4-chlorophenyl)(phenylethynyl)phosphine oxide (5k)



Compound **5k** was prepared from 3-phenylpropiolic acid (**4k**, 43.8 mg. 0.3 mmol), bis(4-chlorophenyl)phosphine oxide (242.1 mg, 0.9 mmol), tetrabutylammonium acetate (90.5 mg, 0.3 mmol), Cp_2Fe (39.1 mg, 0.21 mmol), the solution was charged in constant current mode **(General Procedure B)**. The consumption of the starting material was checked by TLC (40% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **5k** (39 mg, 0.11 mmol, 35%) as white solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.83-7.79 (m, 4H), 7.60-7.59 (m, 2H), 7.50-7.47 (m, 5H), 7.40 (t, J = 7.5 Hz, 2H).

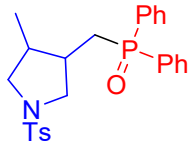
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 139.3 (d, J = 3.8 Hz), 132.8, 132.5 (d, J = 12.3 Hz), 131.4 (d, J = 124.6 Hz), 131.2, 129.3 (d, J = 14.4 Hz), 128.8, 119.6 (d, J = 4.3 Hz), 106.5 (d, J =

30.2 Hz), 82.2 (d, J = 175.7 Hz).

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 6.2

HRMS-ESI: Calcd for $C_{20}H_{14}Cl_2OP^+$ $[M+H]^+$337.0544, found 337.0532

((4-methyl-1-tosylpyrrolidin-3-yl)methyl)diphenylphosphine oxide (9)



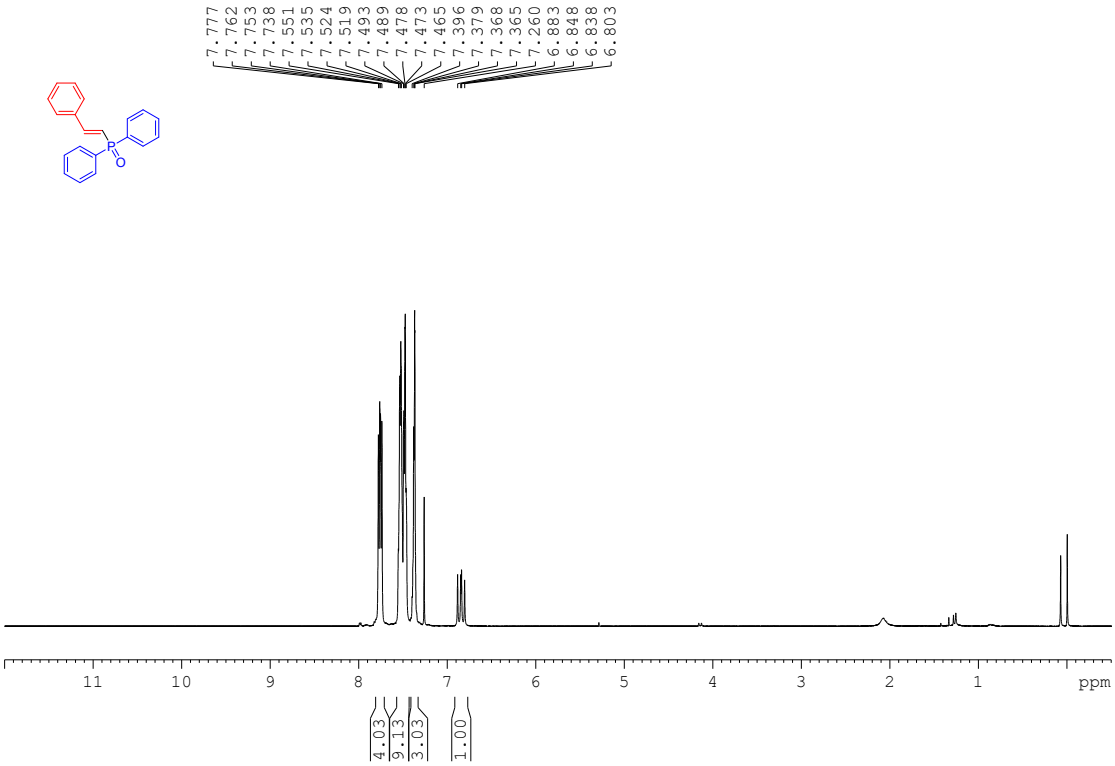
Compound **9** was prepared from *N, N*-diallyl-4-methylbenzenesulfonamide (**8**, 75.3 mg. 0.3 mmol), diphenylphosphine oxide (181.8 mg, 0.9 mmol), tetrabutylammonium hexafluorophosphate (116.1 mg, 0.3 mmol), DABCO (73.9 mg, 0.66 mmol), the solution was charged in constant current mode **(General Procedure A)**. The consumption of the starting material was checked by TLC (50% EtOAc/petroleum ether), followed by aqueous work-up, purified by silica gel column chromatography using 20% to 40% EtOAc/petroleum ether as the eluent to give **9** (73.4 mg, 0.16 mmol, 54%) as colorless solid.

^{1}H NMR (500 MHz, $CDCl_3$, 300K): δ (ppm) 7.70-7.67 (m, 4H), 7.64-7.60 (m, 2H), 7.56-7.45 (m, 6H), 7.27-7.26 (m, 2H), 3.48-3.41 (m, 0.4H), 3.30 (dd, J = 9.9 Hz, J = 7.7 Hz, 0.8H), 3.25 (dd, J = 9.6 Hz, J = 6.4 Hz, 0.8H), 3.06-2.99 (m, 1.6H), 2.89-2.85 (m, 0.2H), 2.69-2.66 (m, 0.2H), 2.40 (s, 0.8H), 2.39 (s, 2.2H), 2.34-2.30 (m, 1H), 2.26-2.19 (m, 2H), 2.06-1.97 (m, 1H), 0.87 (d, J = 5.7 Hz, 0.7H), 0.75 (d, J = 7.1 Hz, 2.3H).

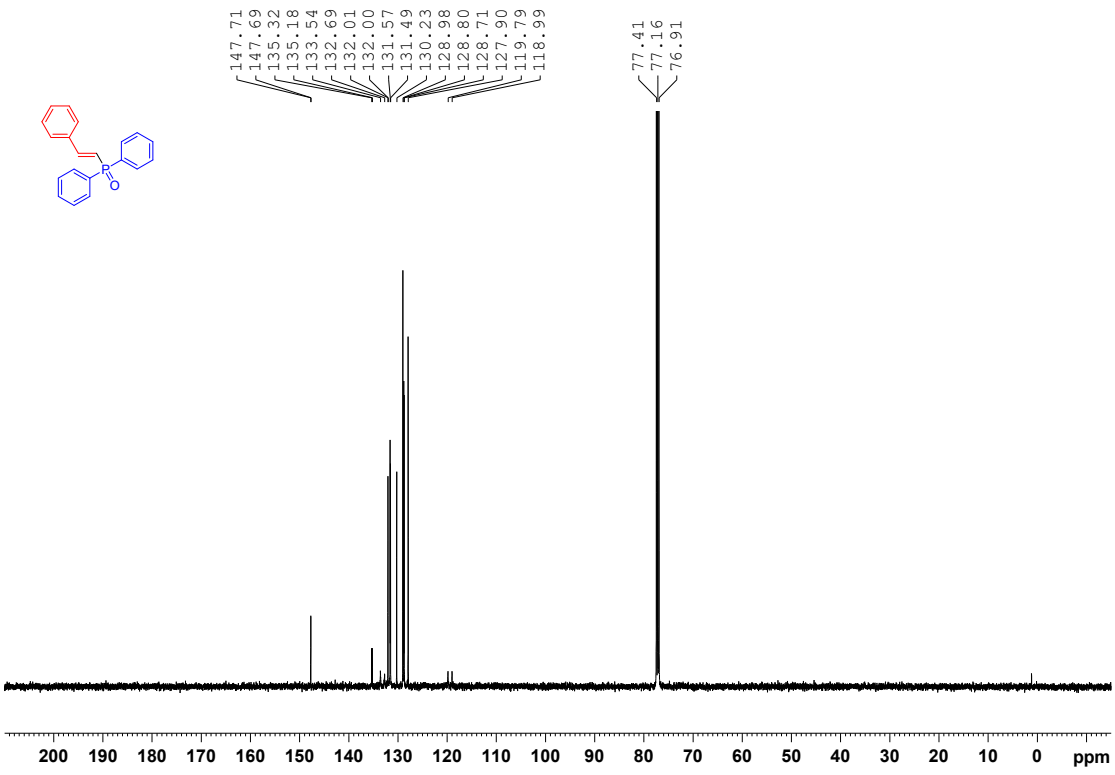
^{13}C NMR (125 MHz, $CDCl_3$, 300K): δ (ppm) 143.41 (minor), 143.39, 133.9, 133.6 (minor), 133.1, 133.0 (minor), 132.98 (d, J = 98.7 Hz, minor), 132.9 (d, J = 98.1 Hz), 132.3, 132.11 (minor, overlapped), 132.1 (d, J = 2.4 Hz), 132.0 (d, J = 2.4 Hz), 130.7 (d, J = 2.9 Hz), 130.67 (d, J = 2.7 Hz), 129.7, 129.7 (minor, overlapped), 128.9 (d, J = 1.4 Hz), 128.8 (d, J = 1.5 Hz), 127.57, 127.49 (minor), 54.2, 53.8 (minor), 53.6 (d, J = 3.0 Hz, minor), 51.6 (d, J = 5.8 Hz), 40.4 (d, J = 12.7 Hz, minor), 39.8 (d, J = 3.9 Hz, minor), 36.3 (d, J = 9.5 Hz), 35.9 (d, J = 3.5 Hz), 32.3 (d, J = 70.0 Hz, minor), 28.2 (d, J = 71.6 Hz), 21.58 (minor, overlapped), 21.57, 15.9 (minor), 13.4.

^{31}P NMR (162 MHz, $CDCl_3$, 300K): δ (ppm) 30.7, 30.2 (minor)

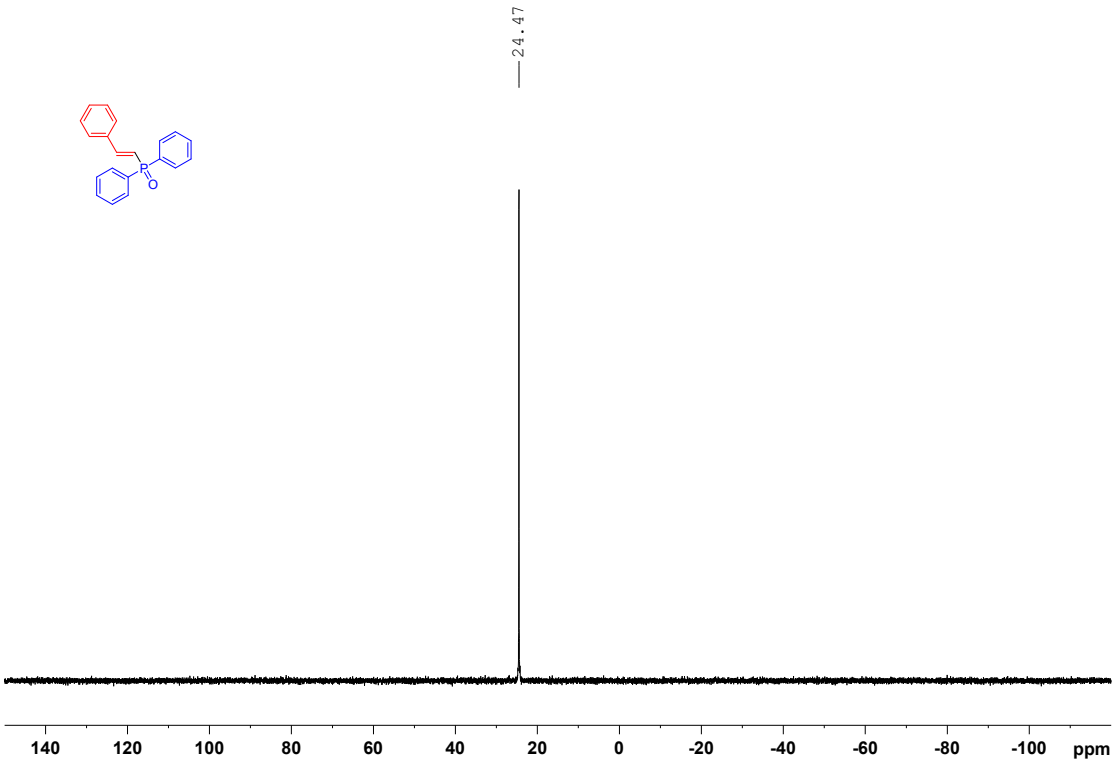
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3a**



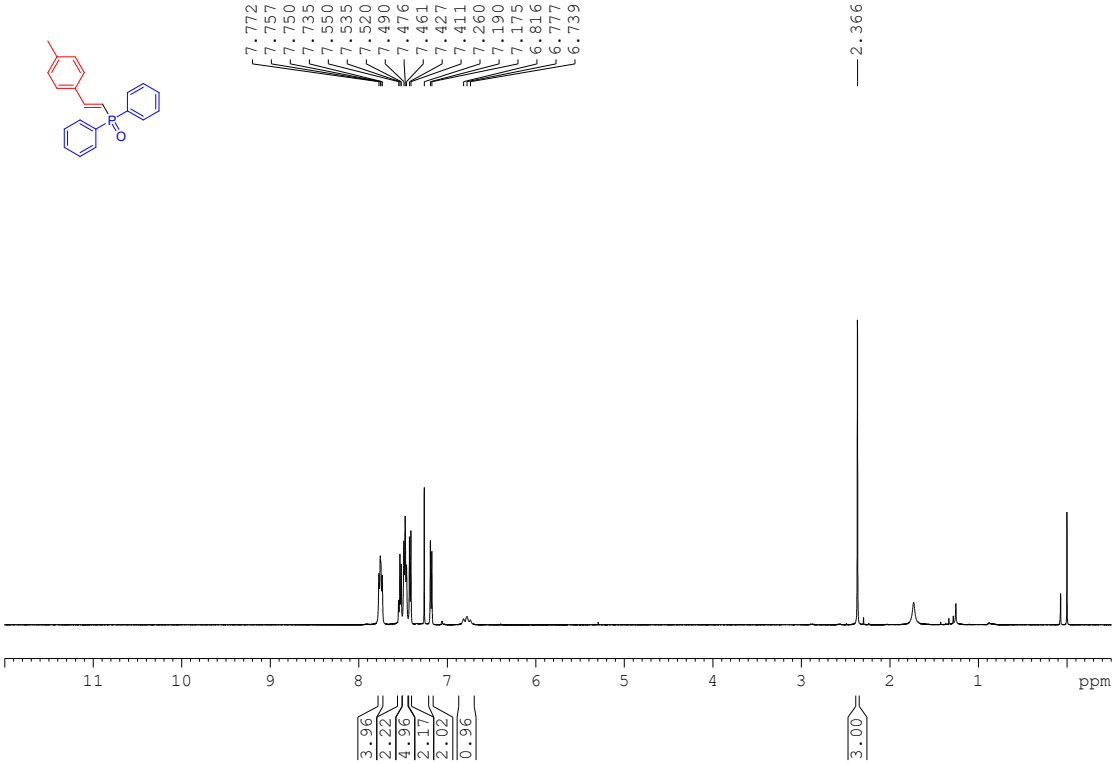
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3a**



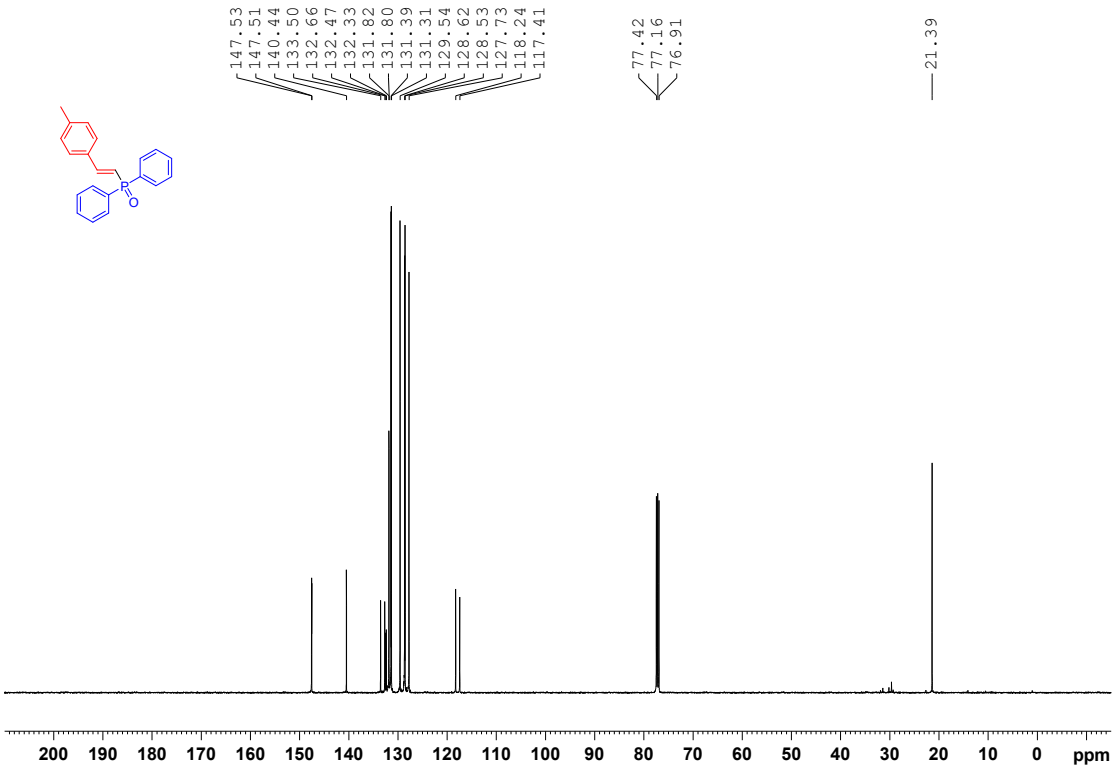
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3a**



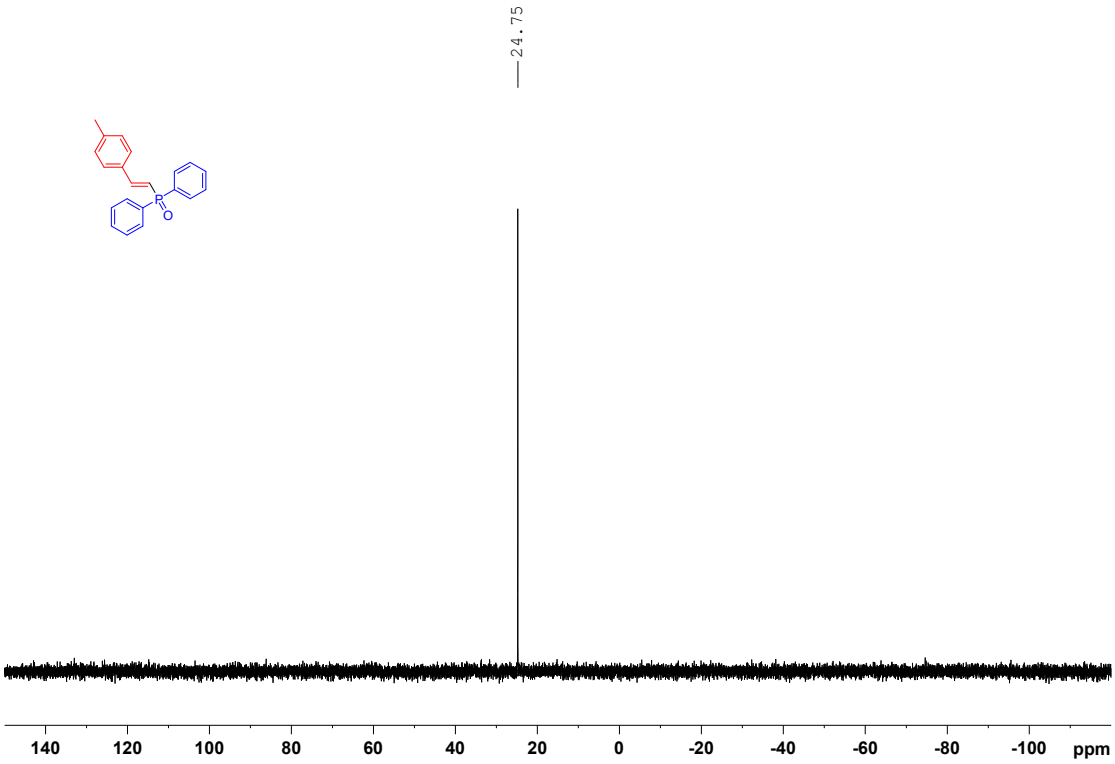
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3b**



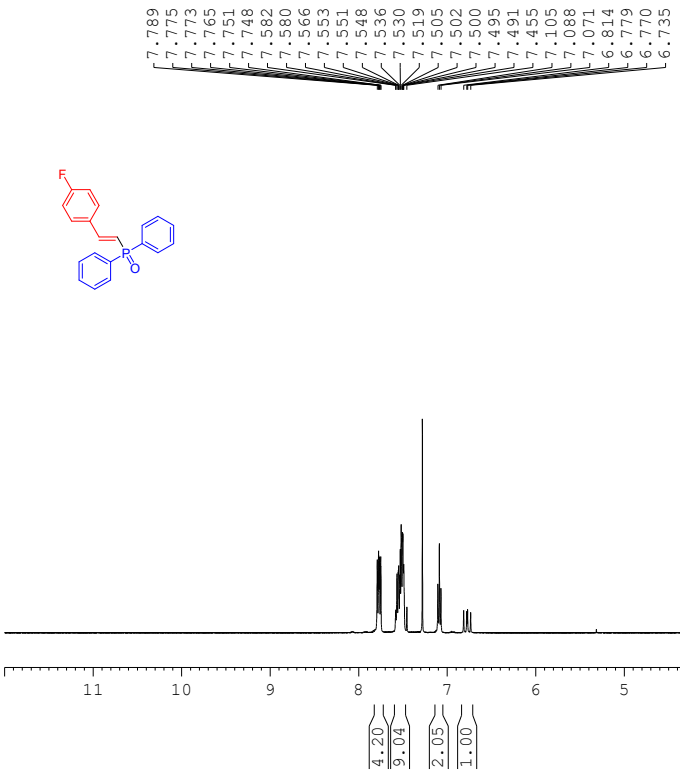
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3b**



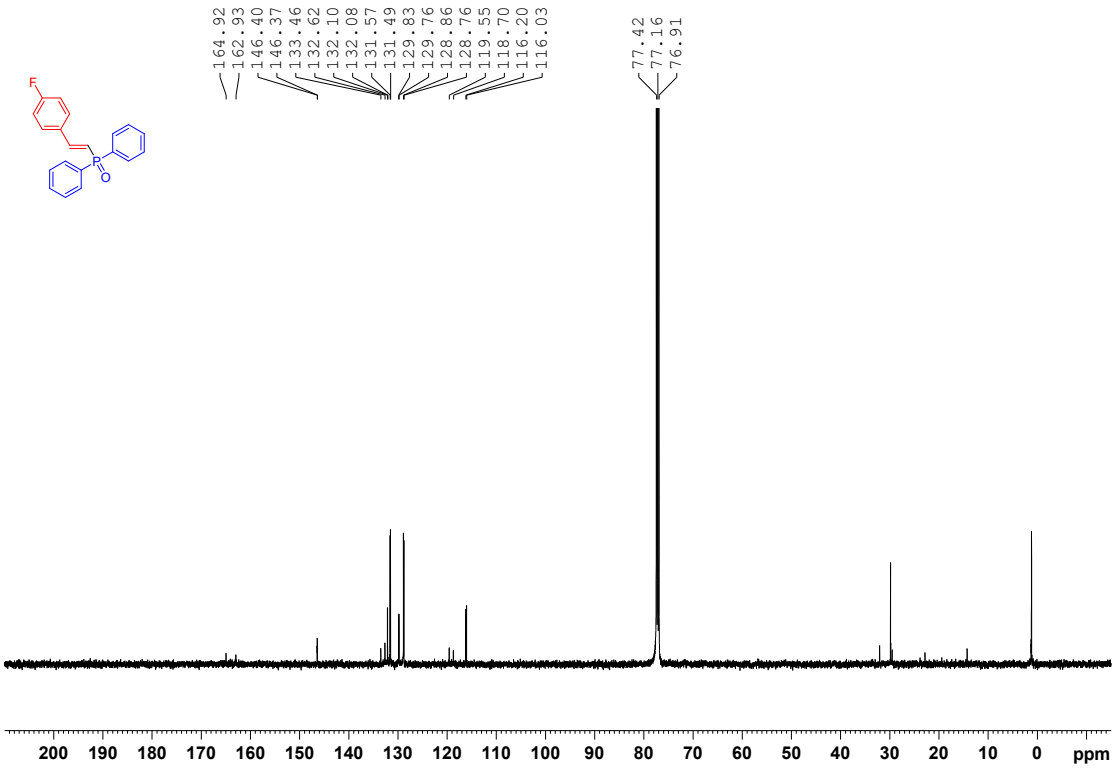
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3b**



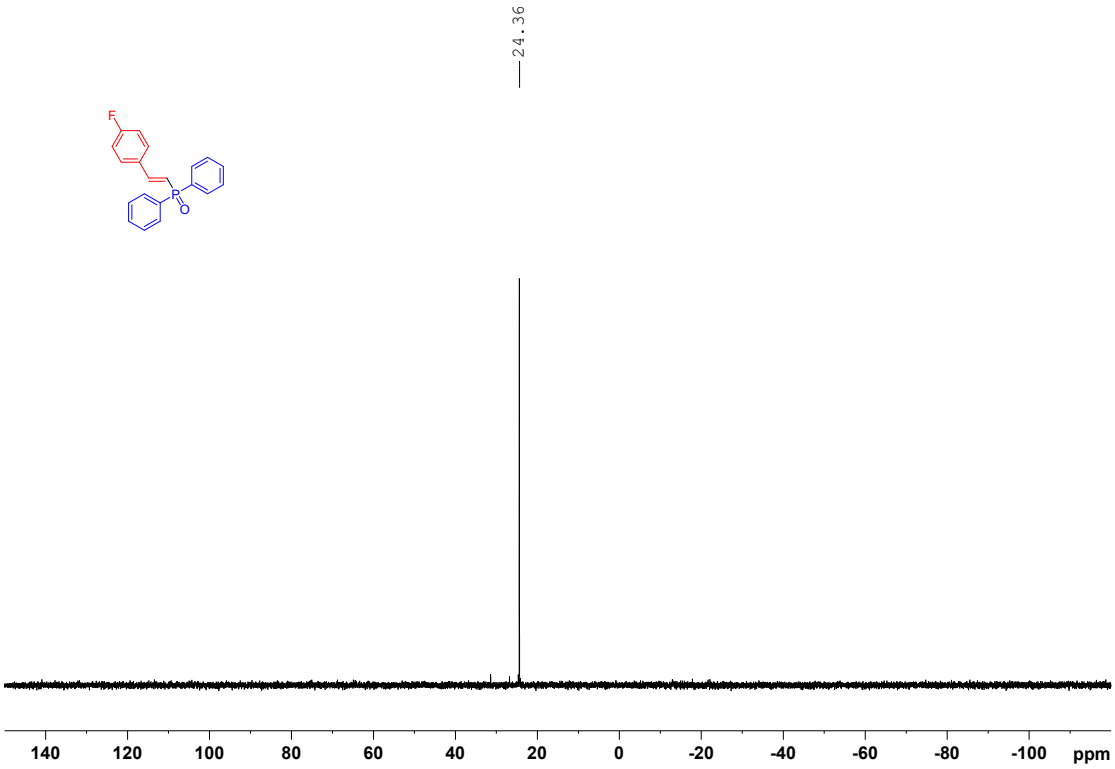
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3c**



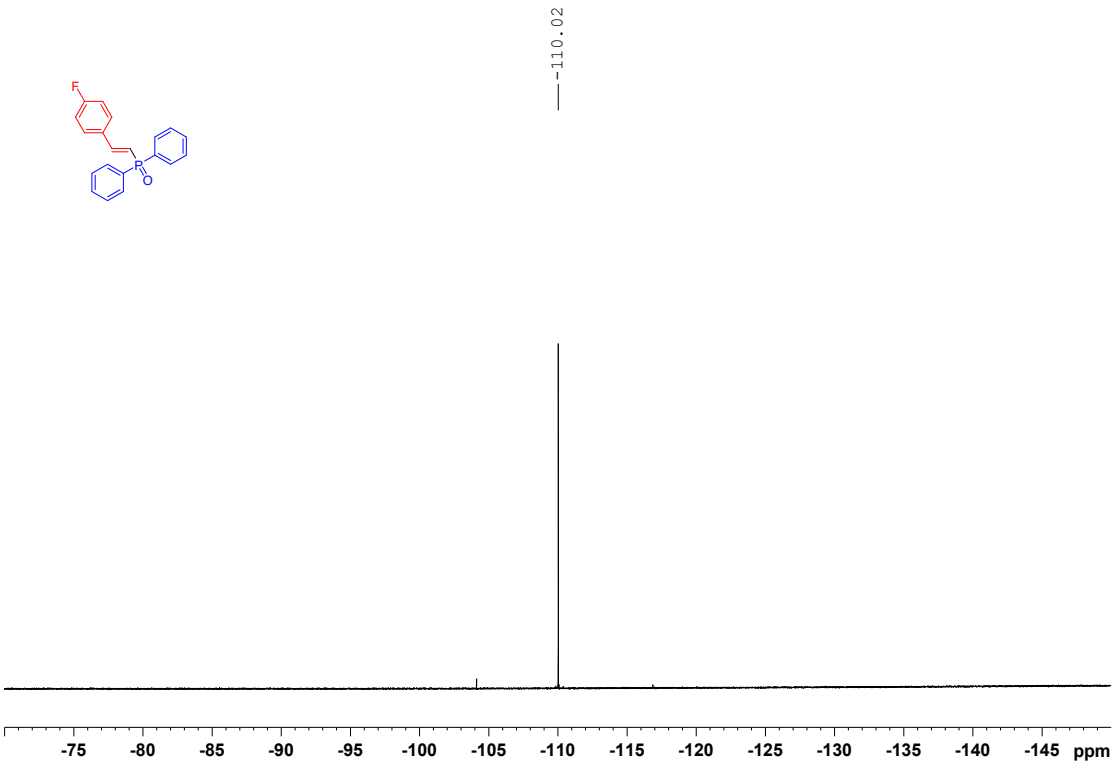
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3c**



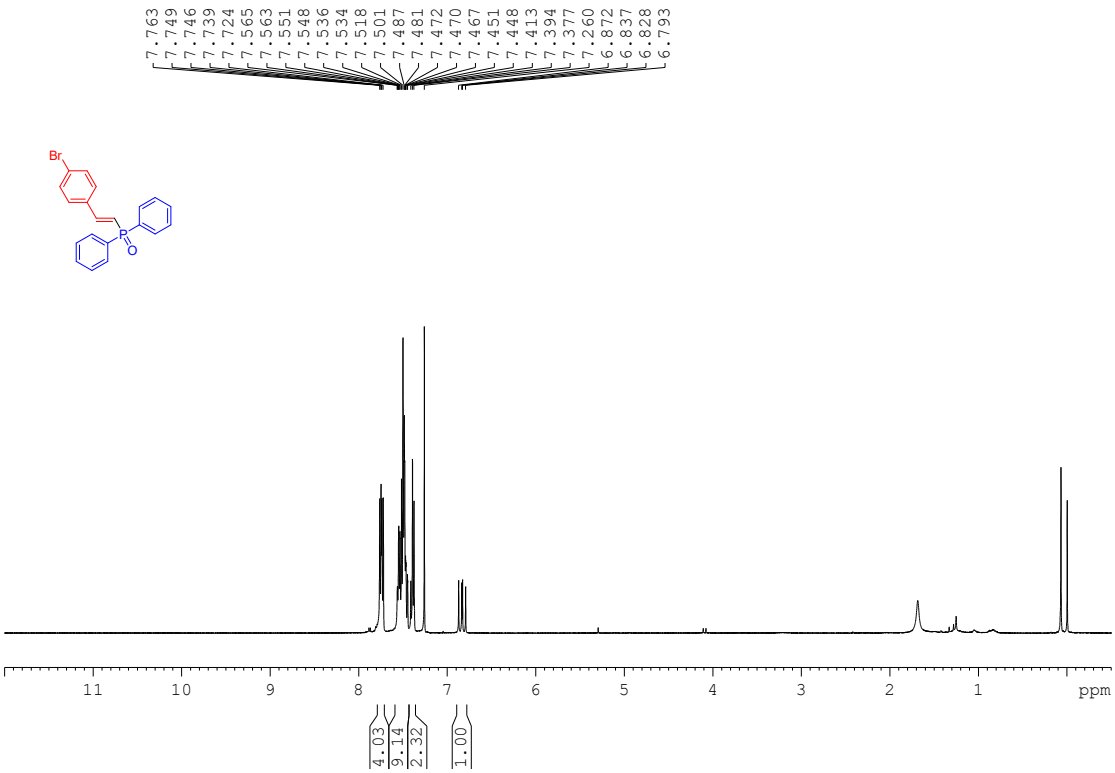
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3c**



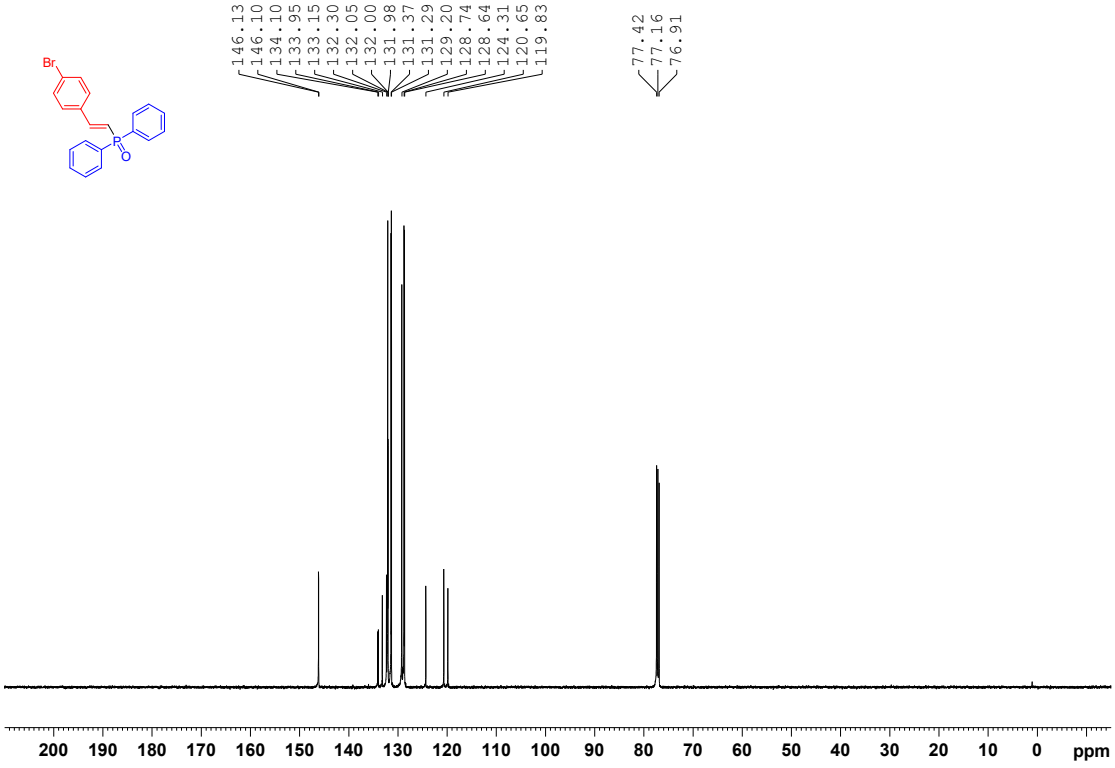
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **3c**



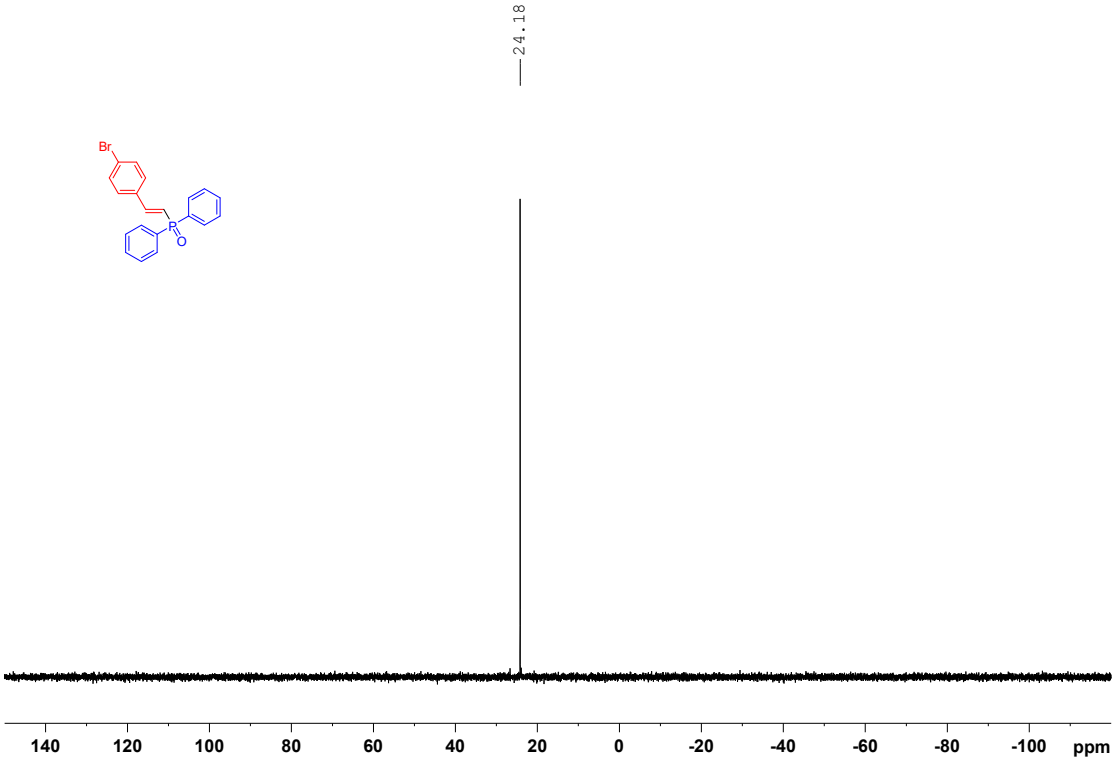
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3d**



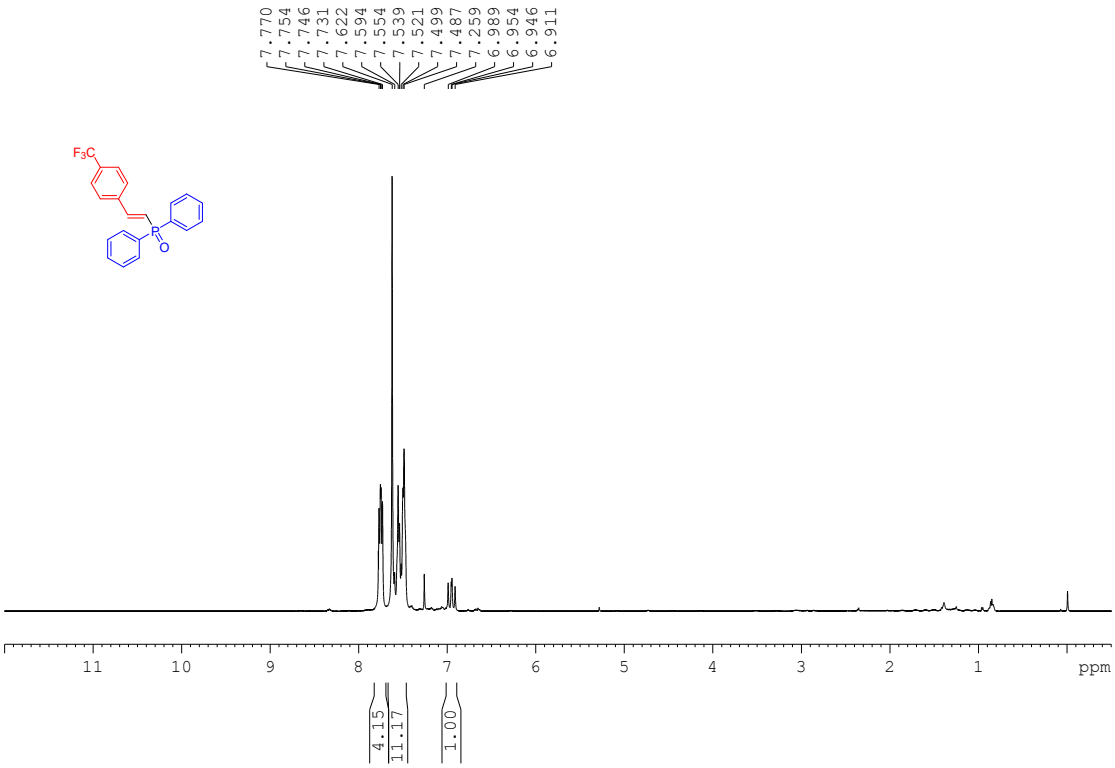
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3d**



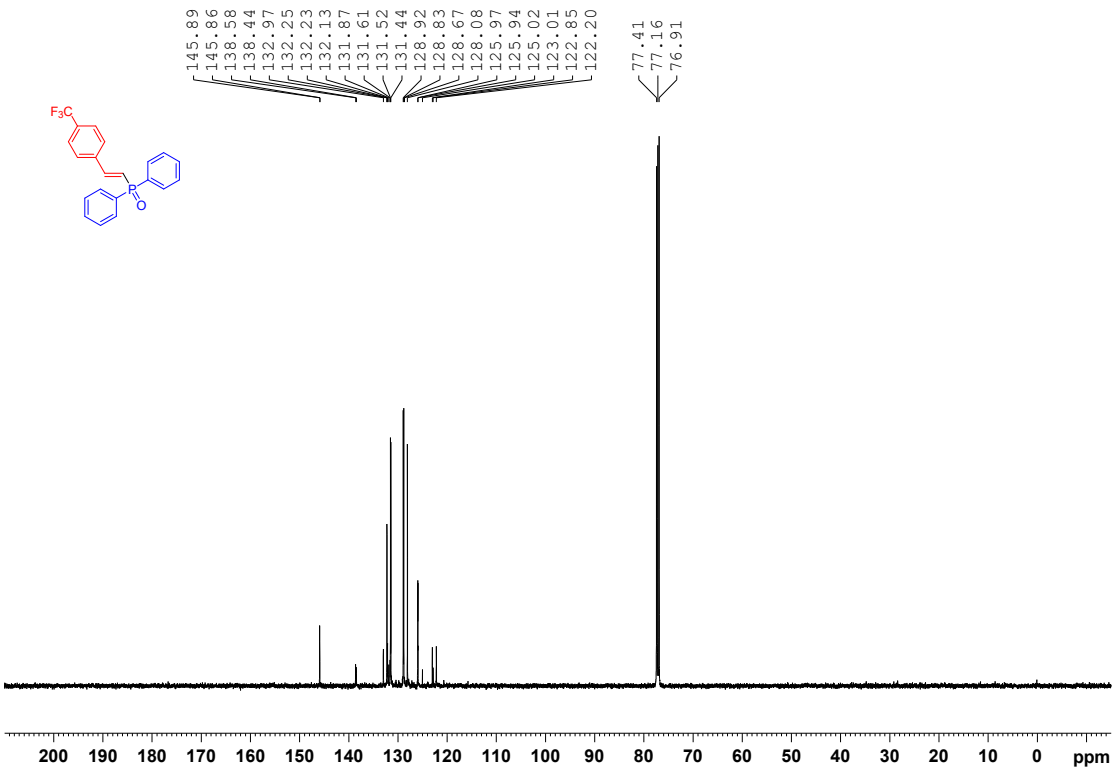
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3d**



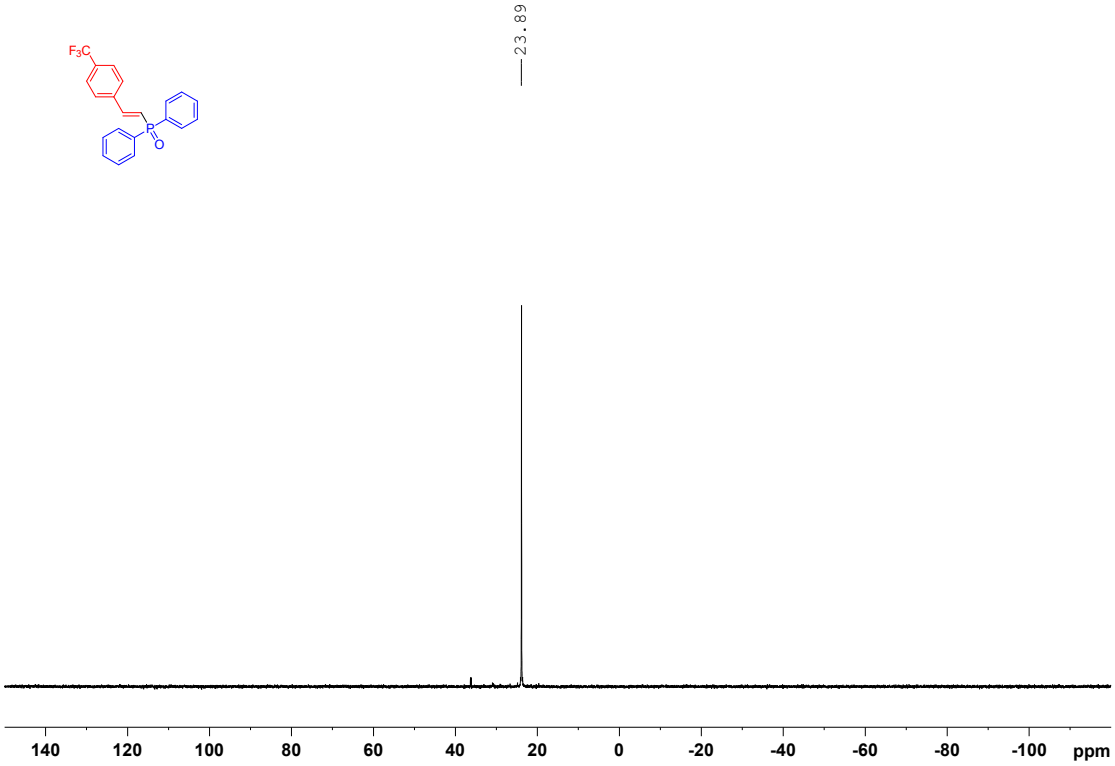
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3e**



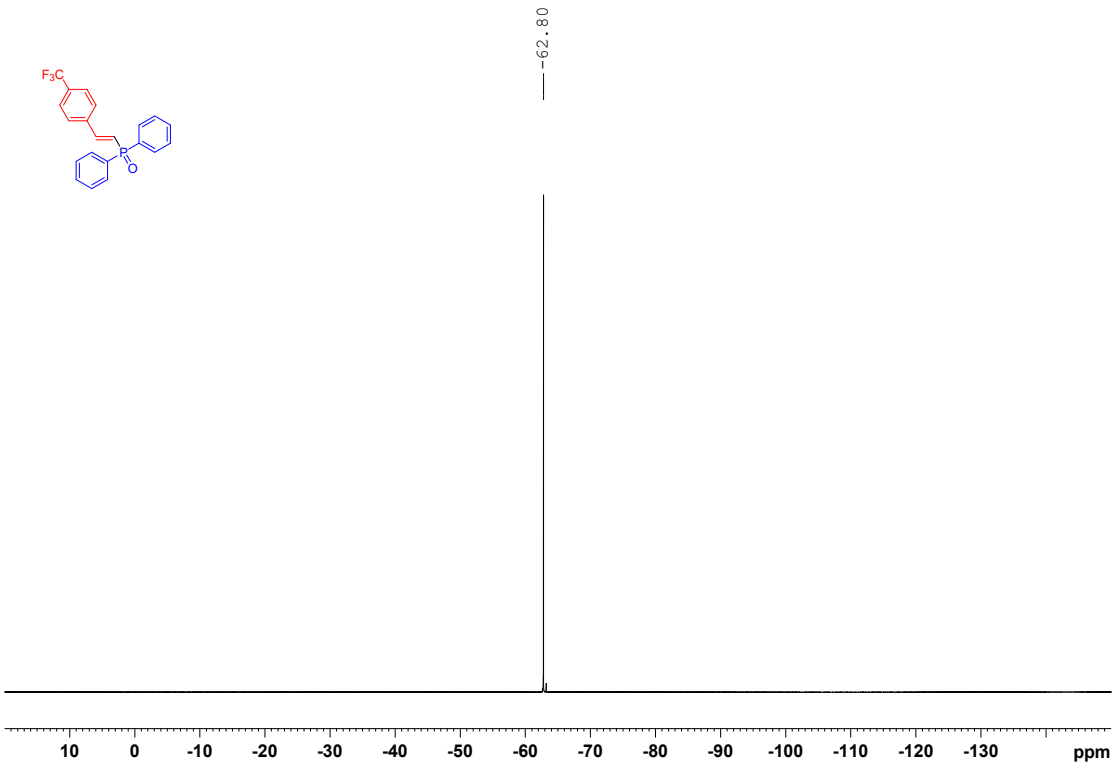
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3e**



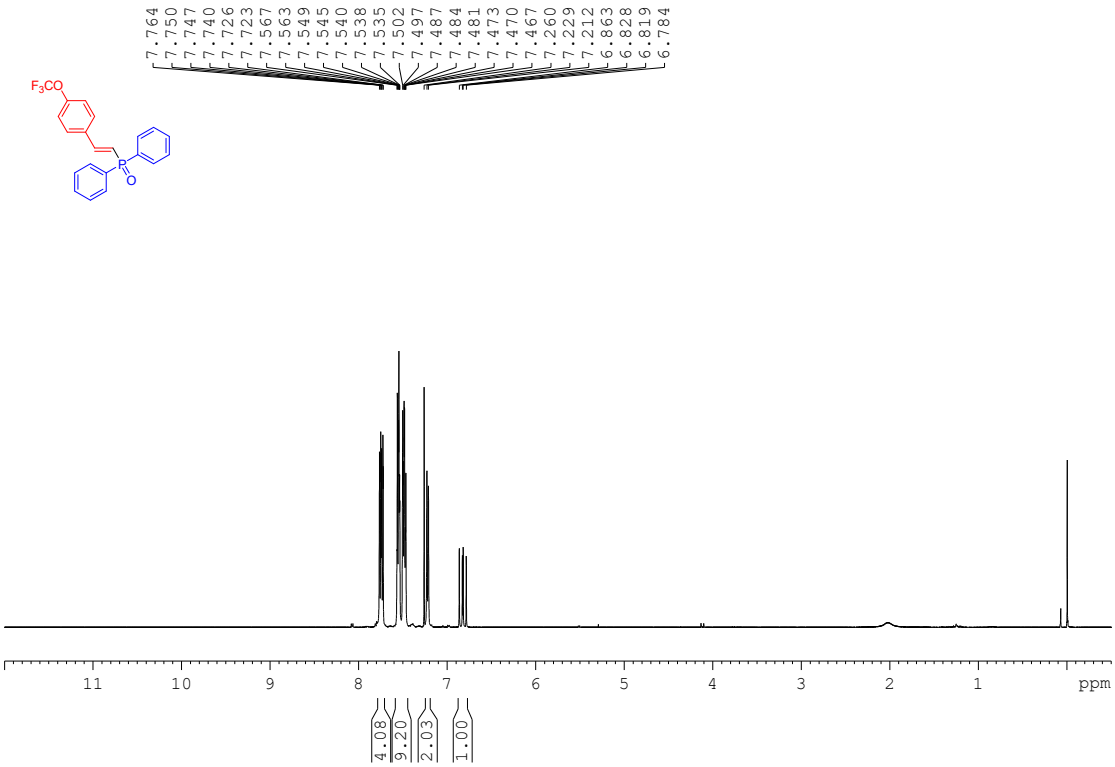
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3e**



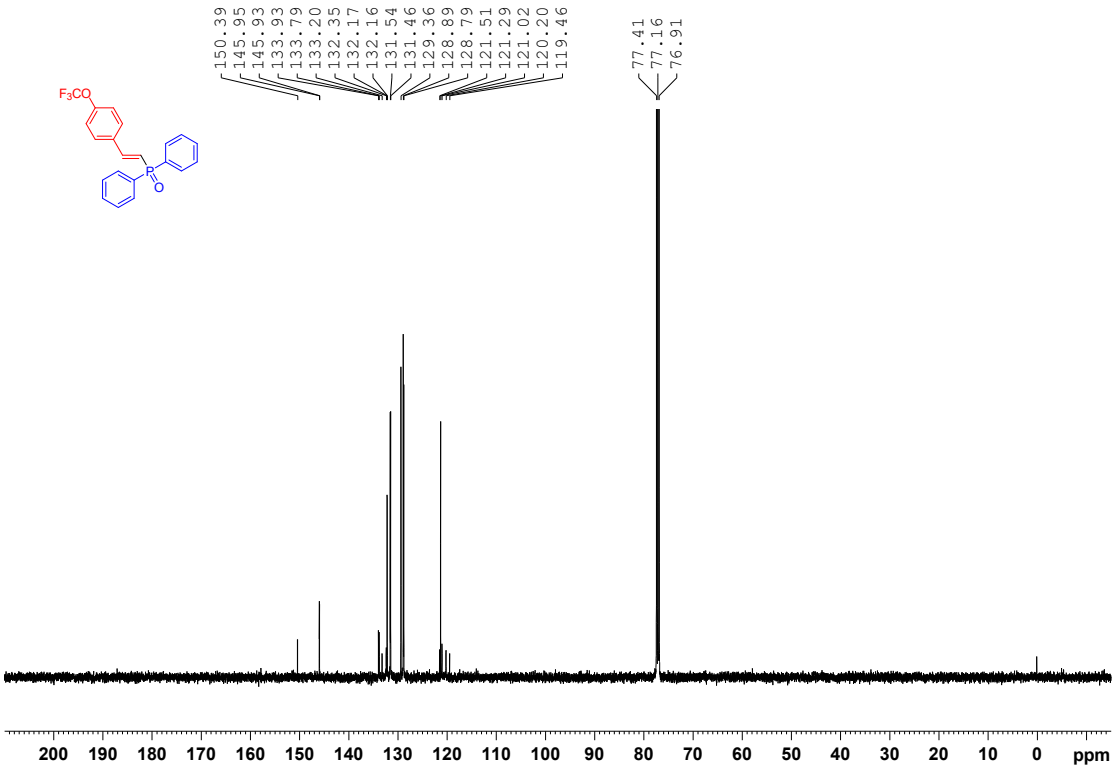
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **3e**



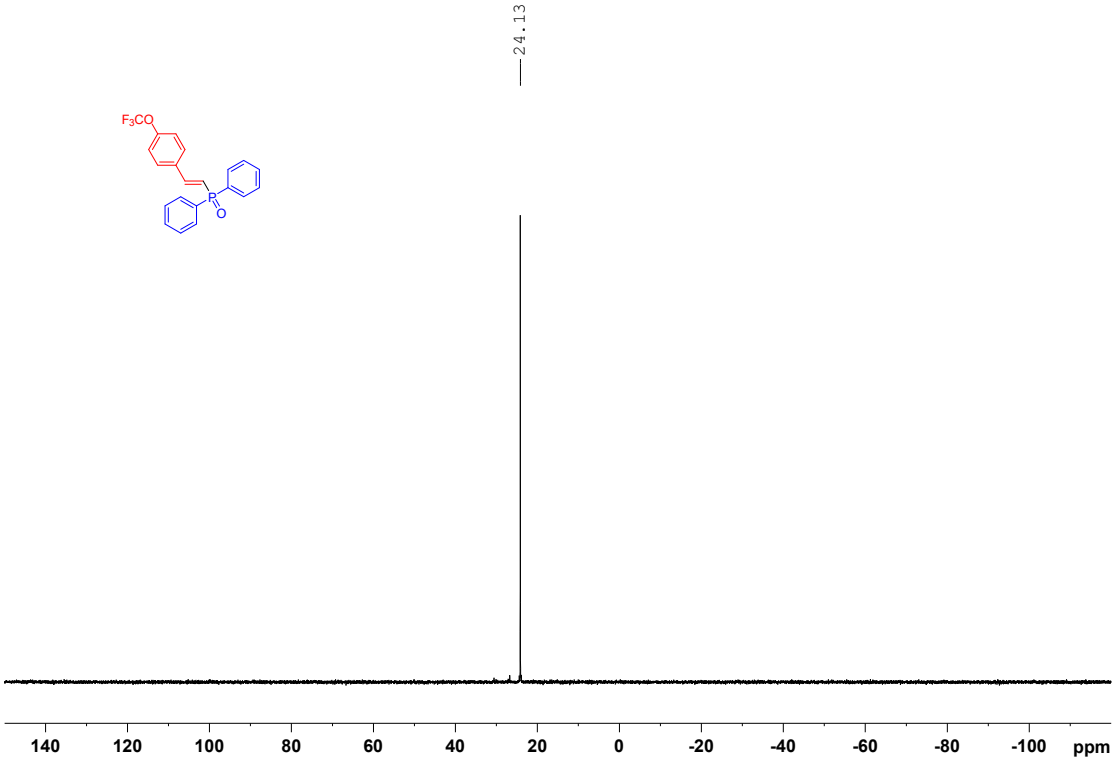
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3f**



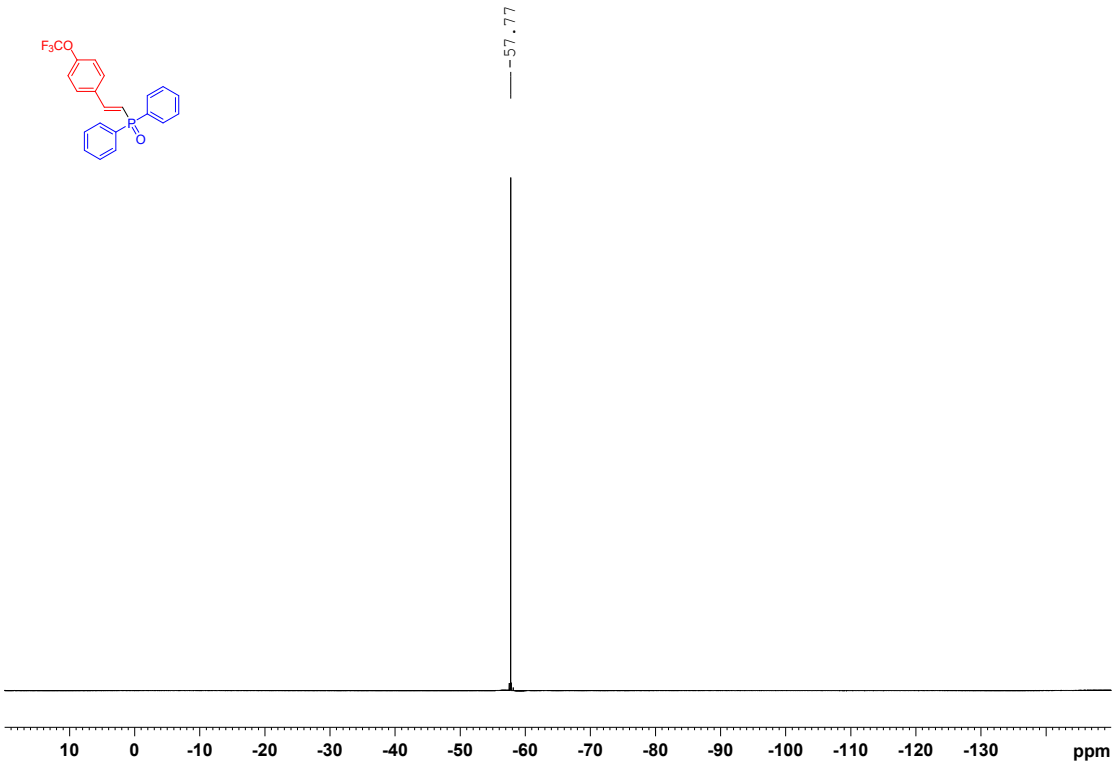
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3f**



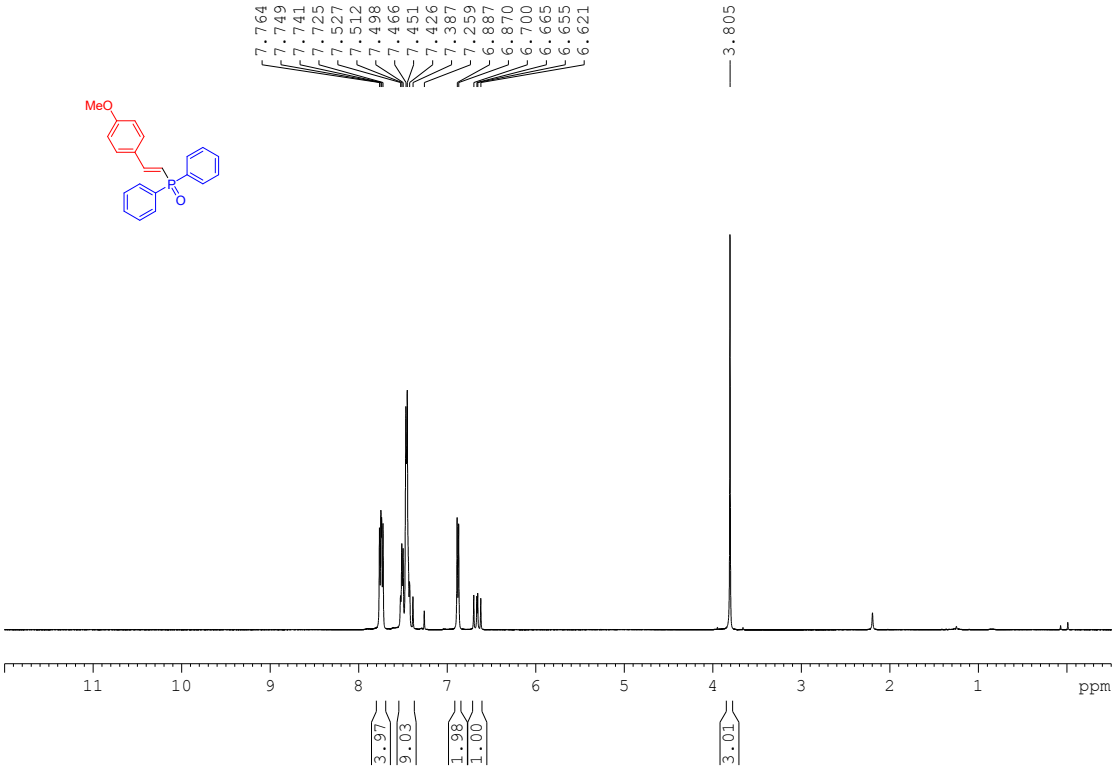
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3f**



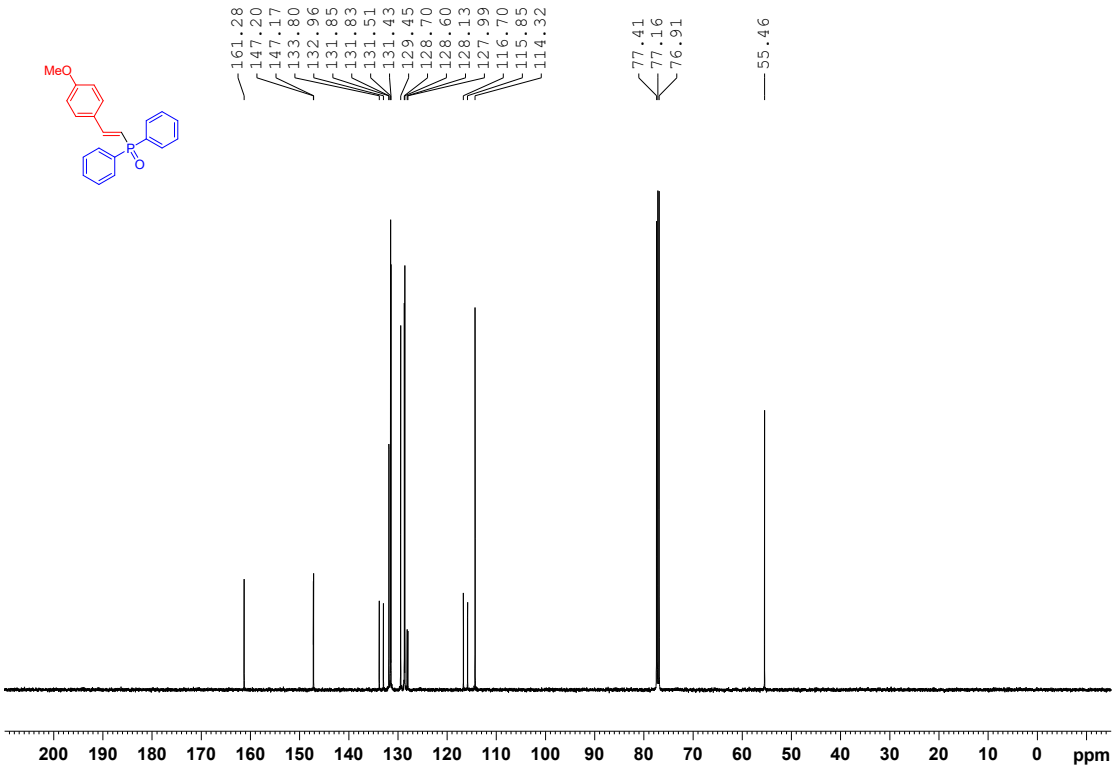
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **3f**



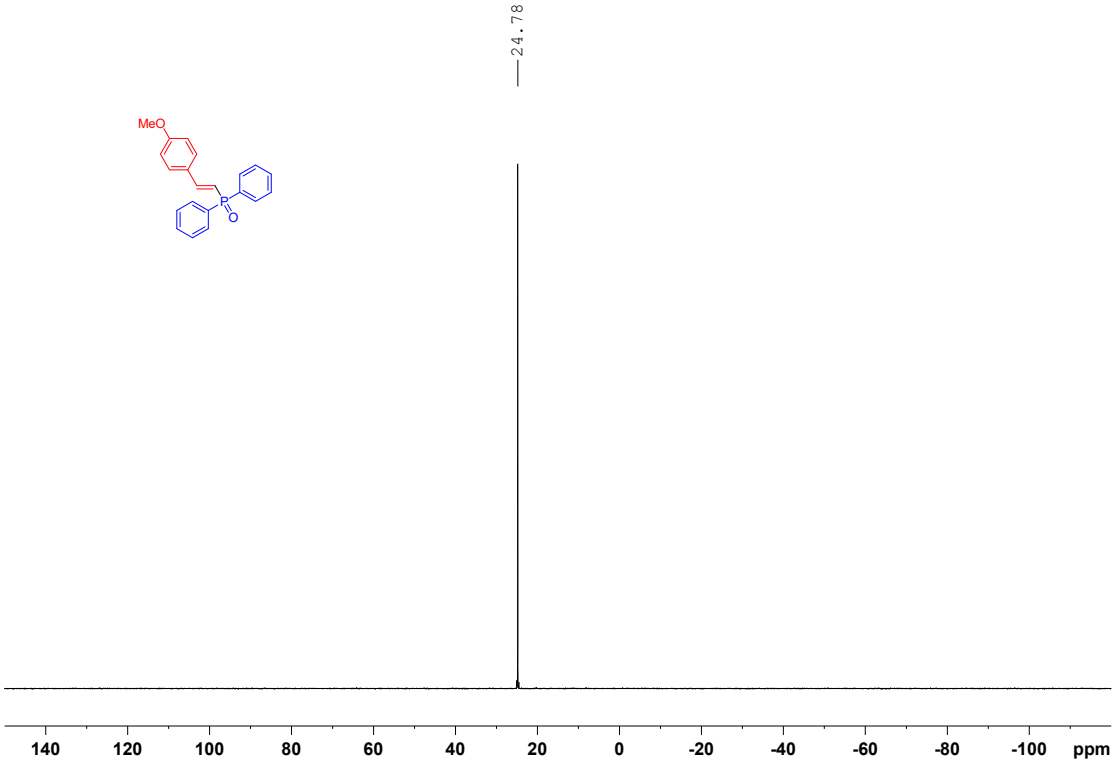
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3g**



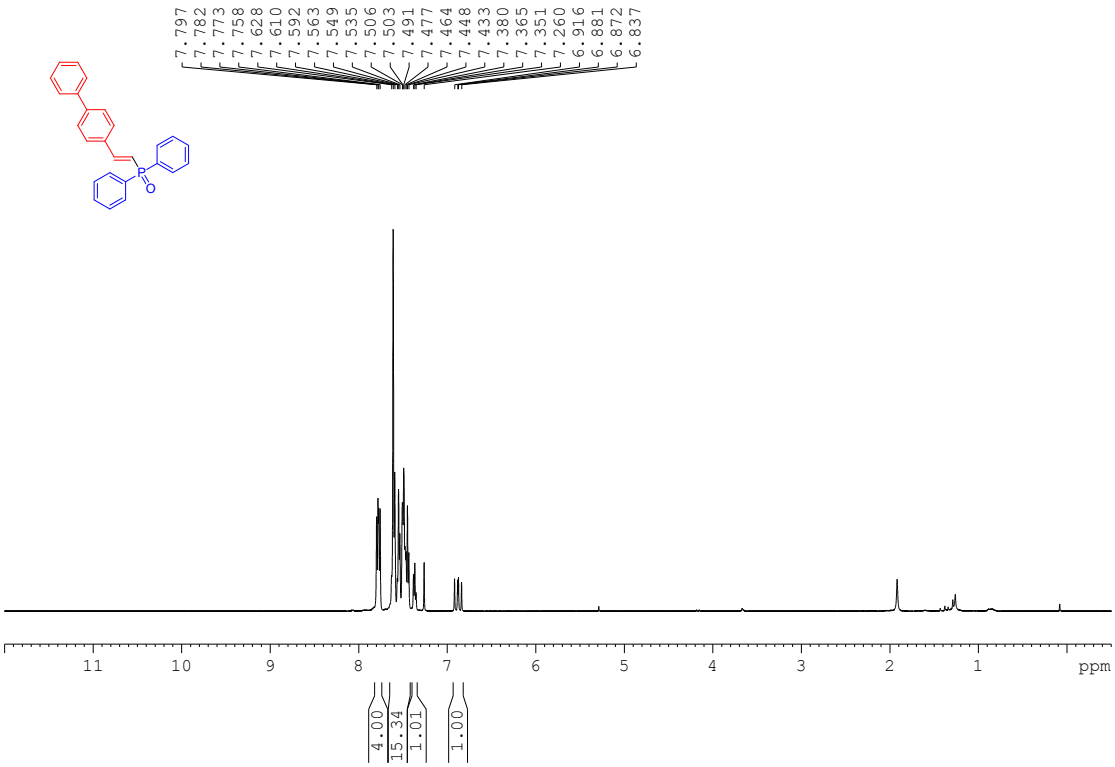
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3g**



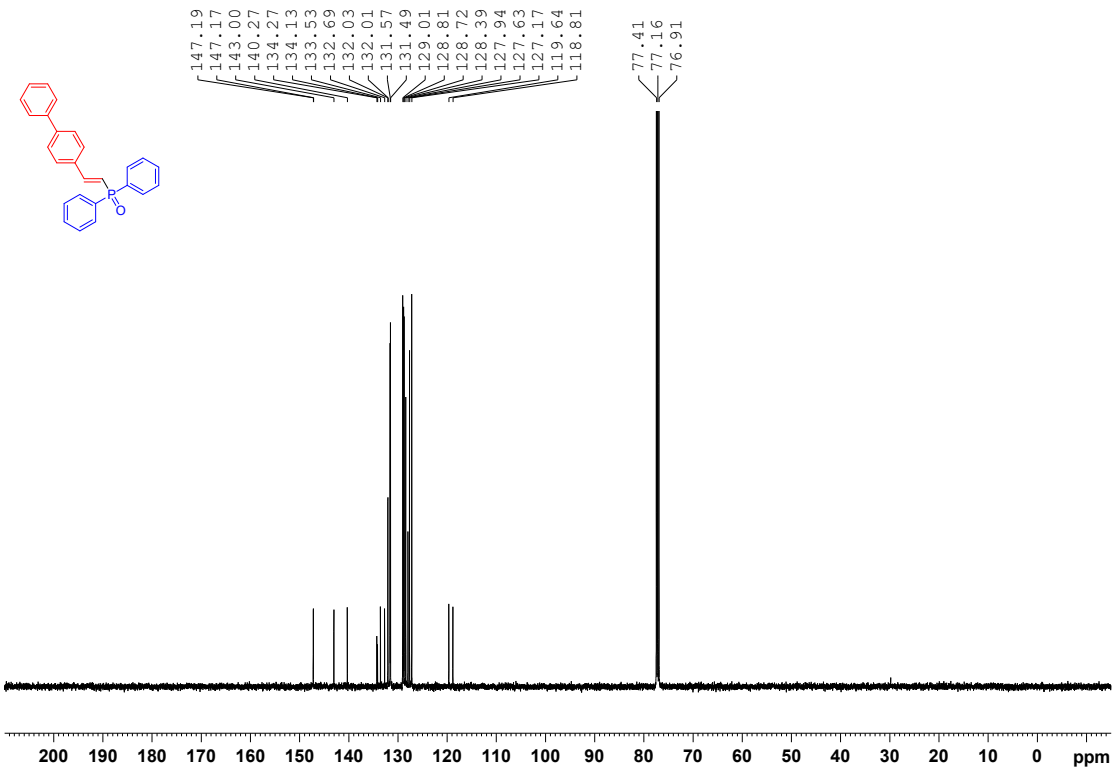
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3g**



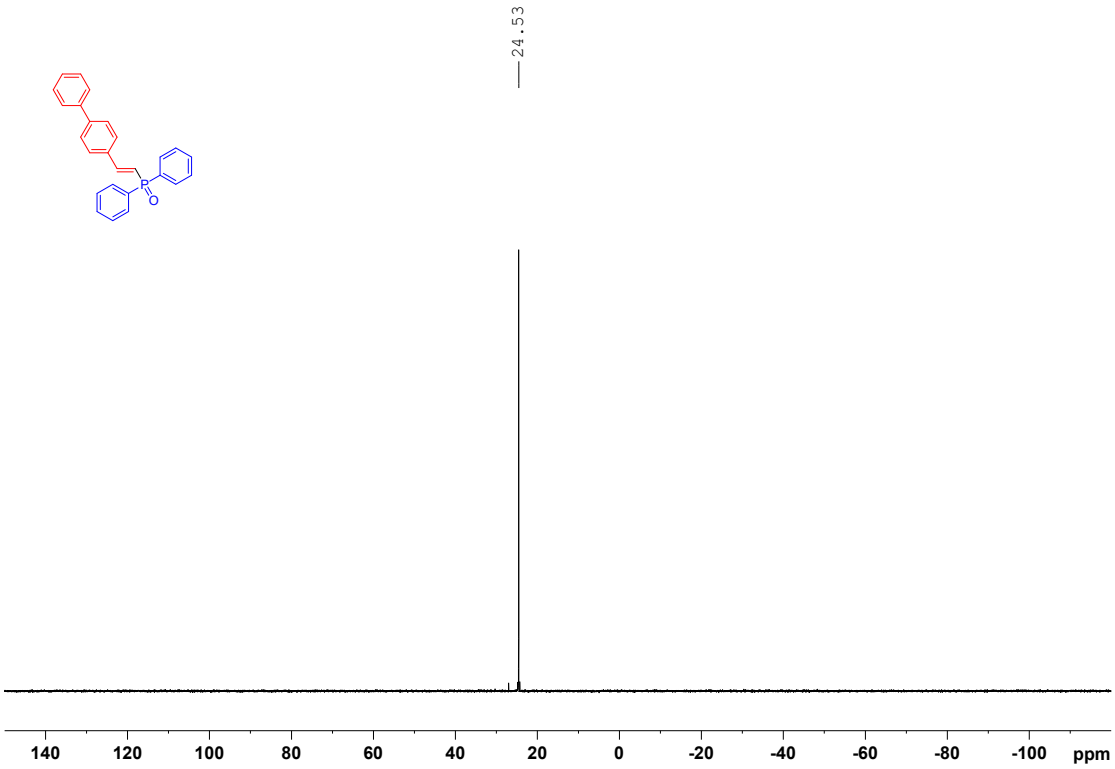
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3h**



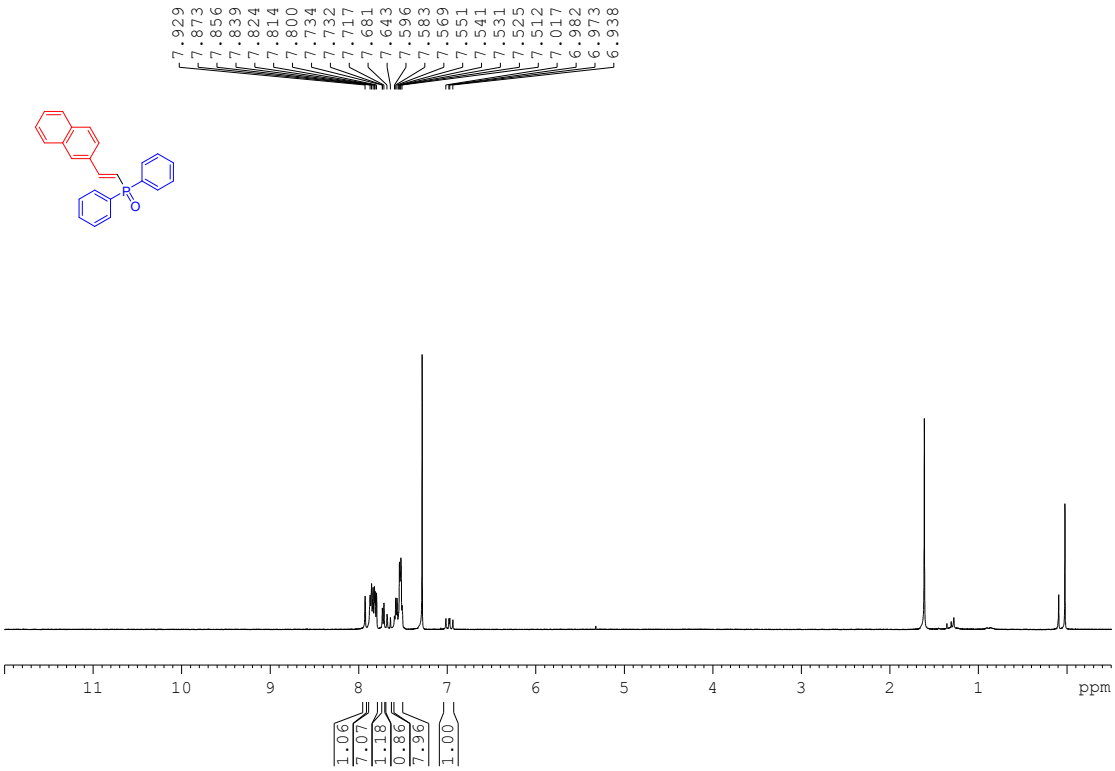
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3h**



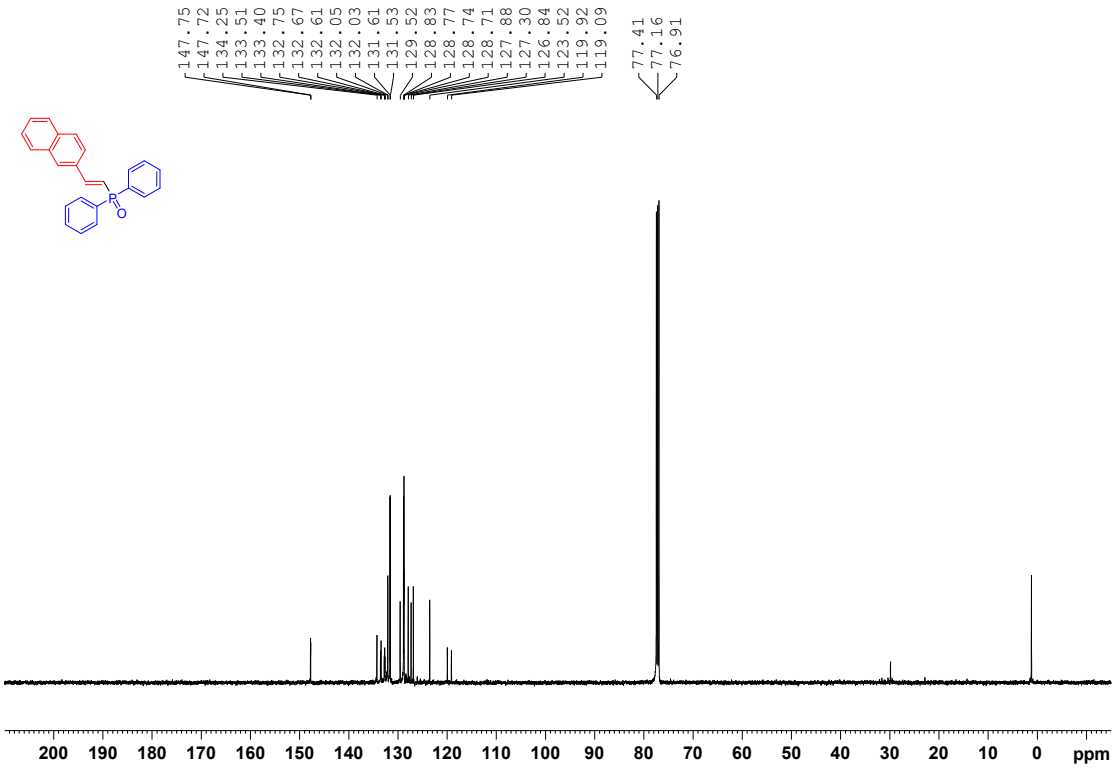
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3h**



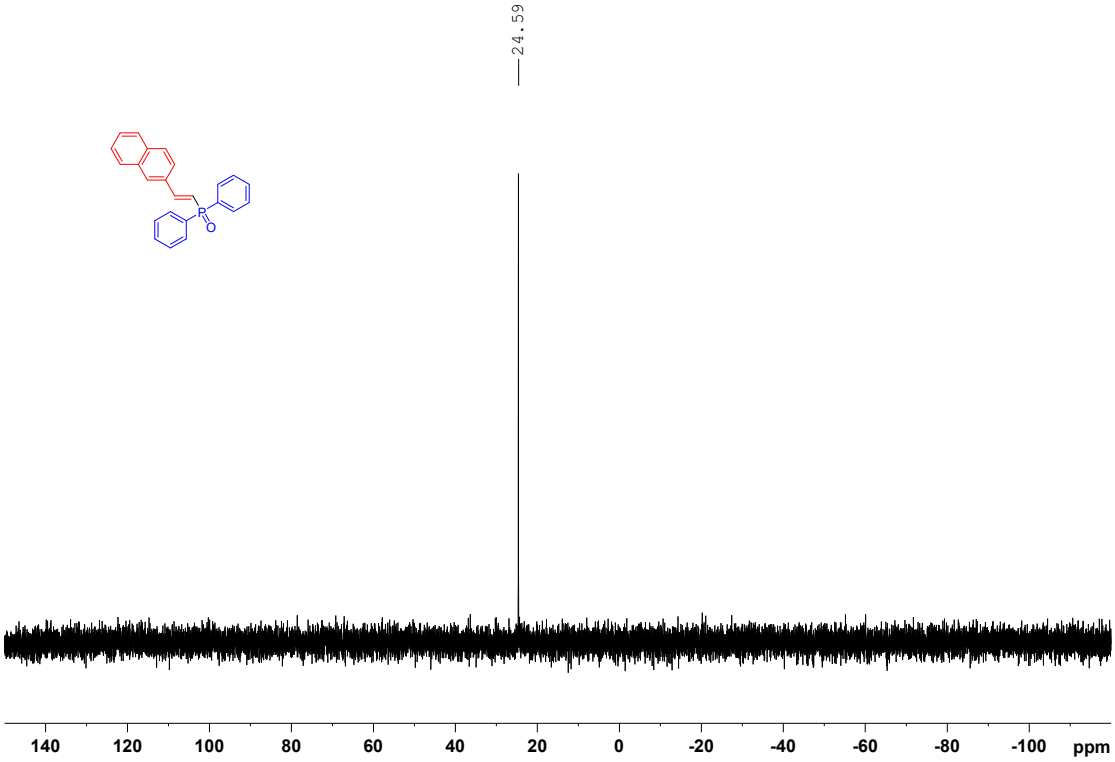
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3i**



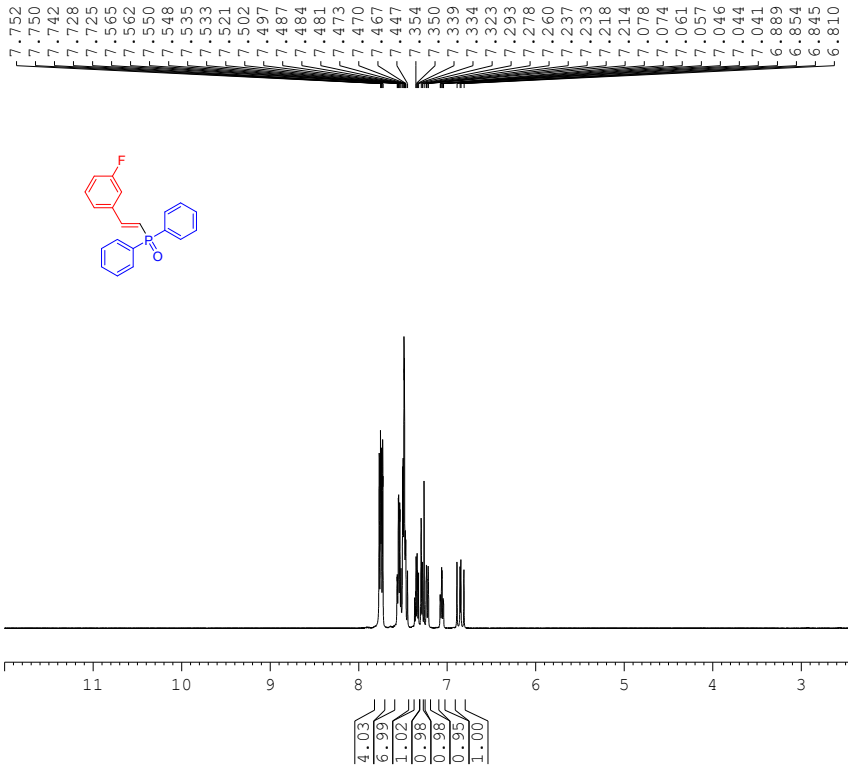
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3i**



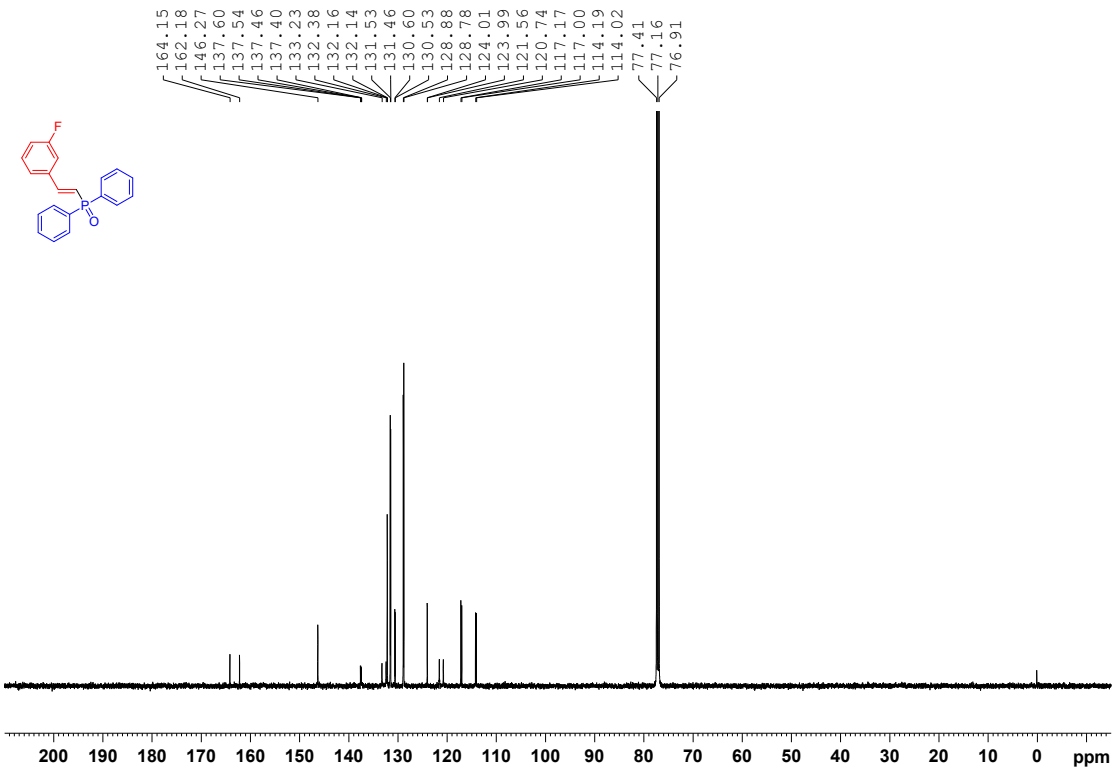
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3i**



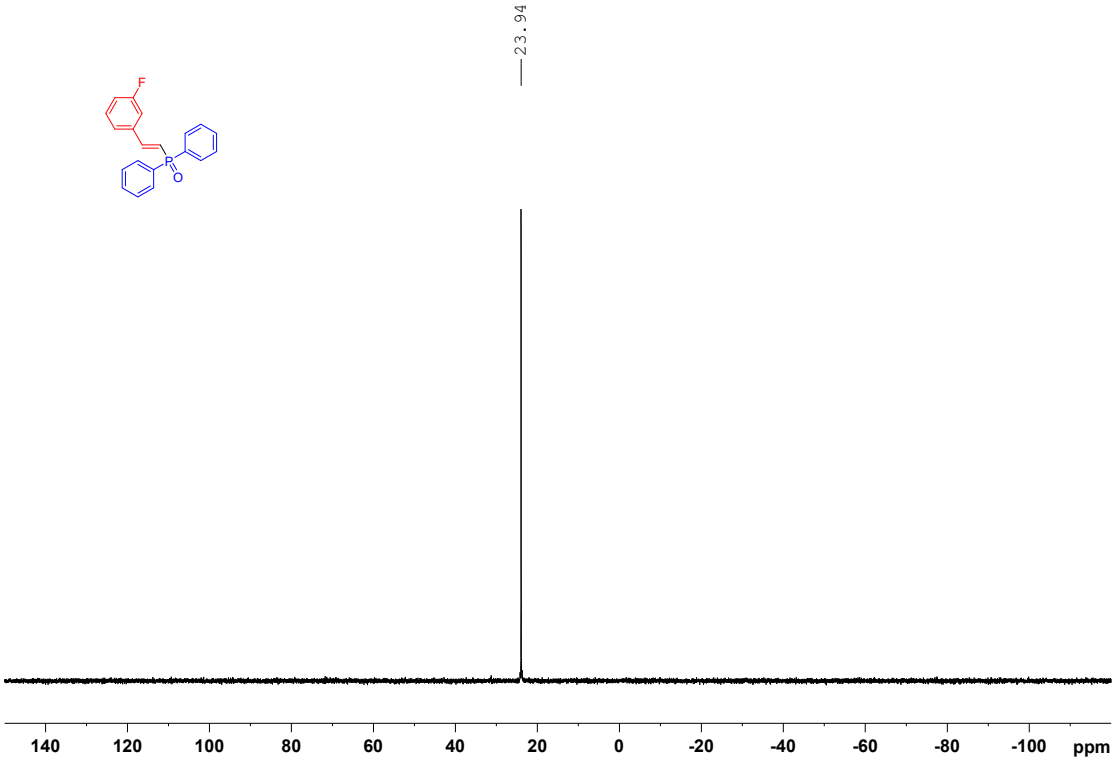
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3j**



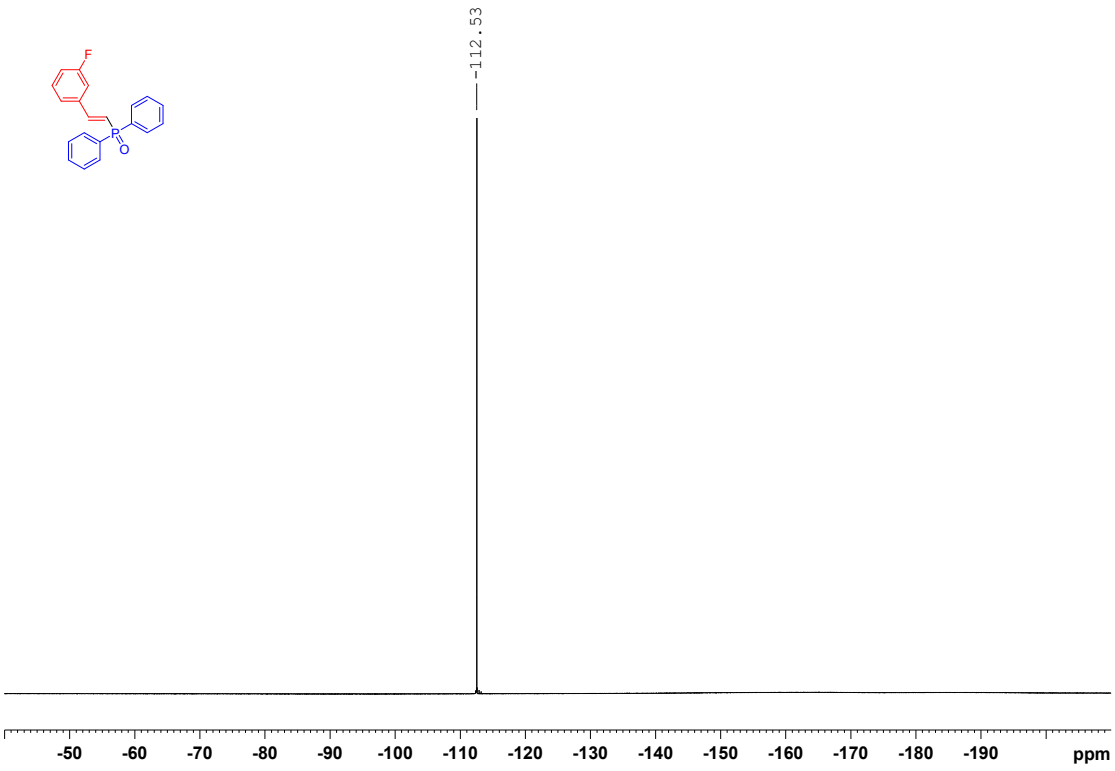
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3j**



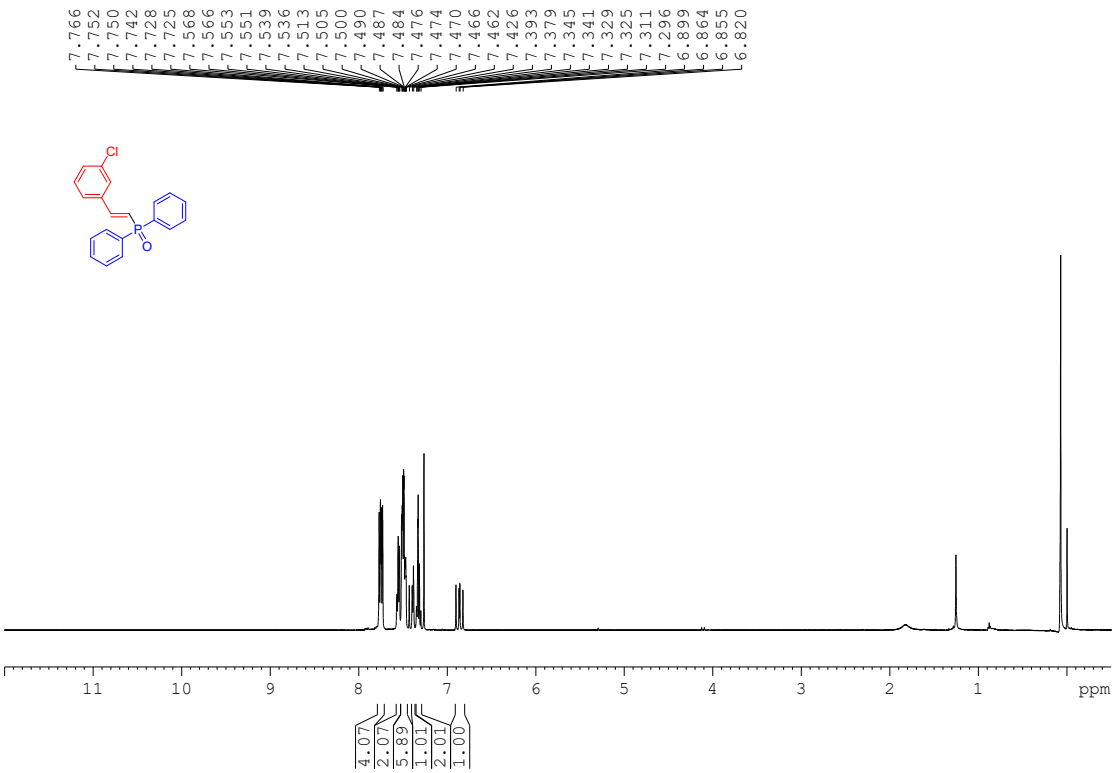
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3j**



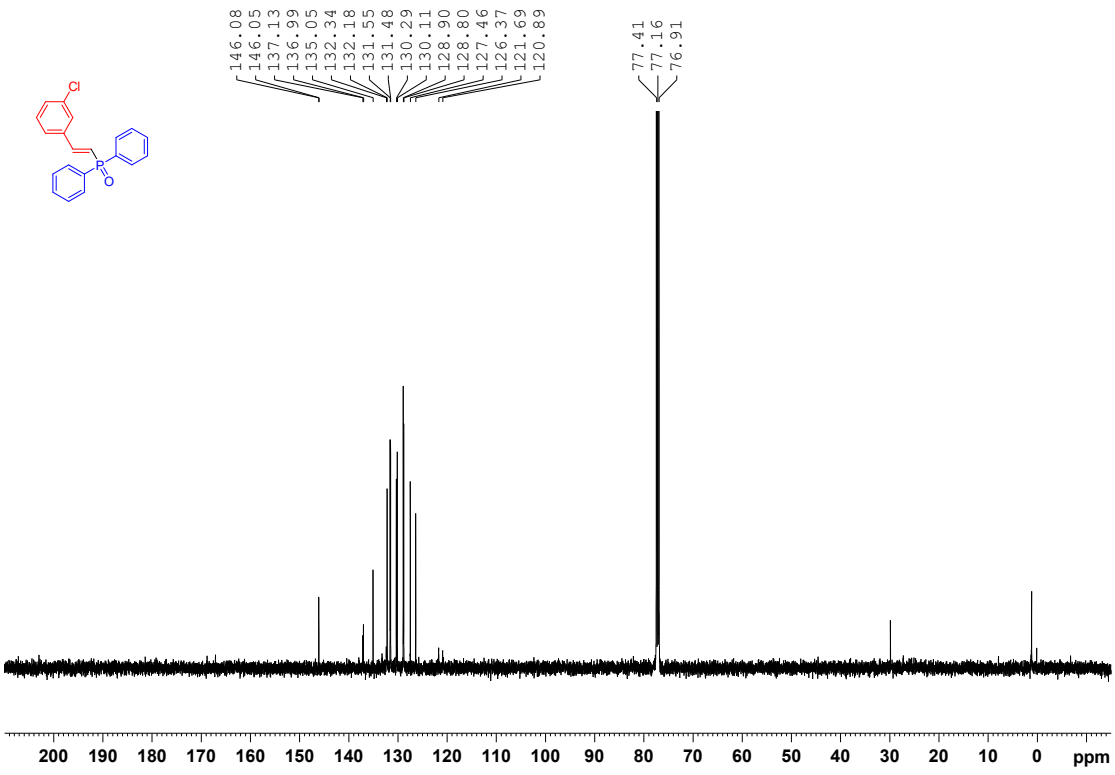
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **3j**



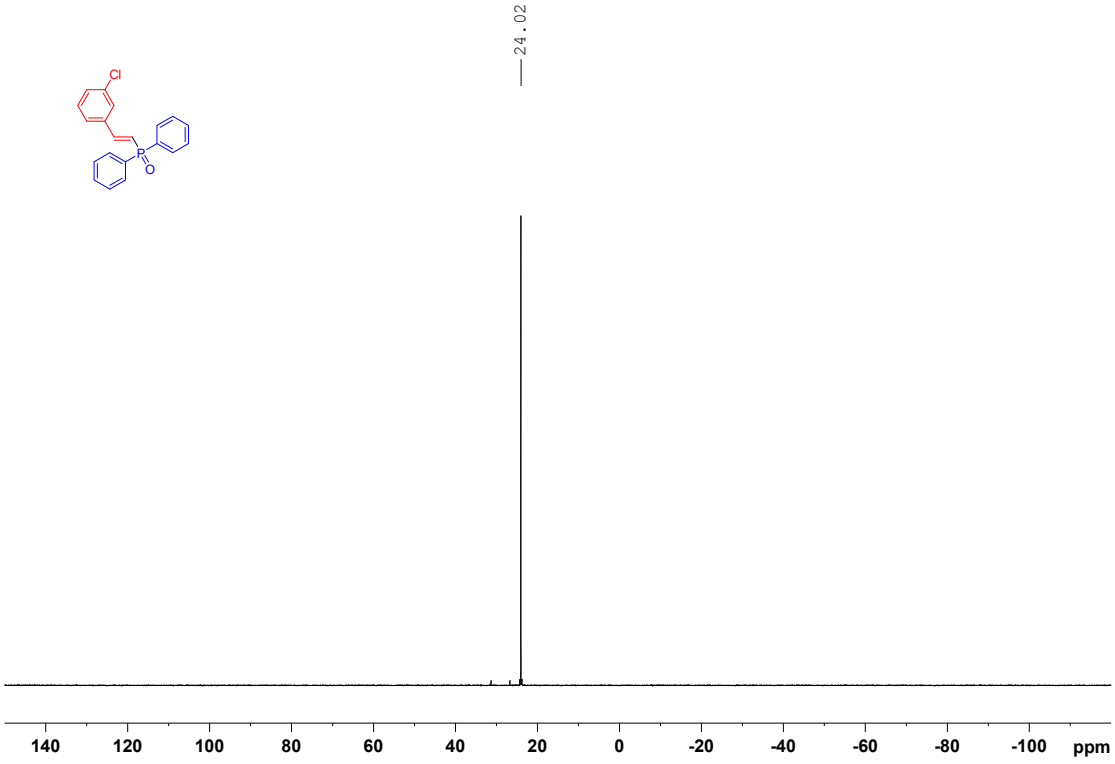
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3k**



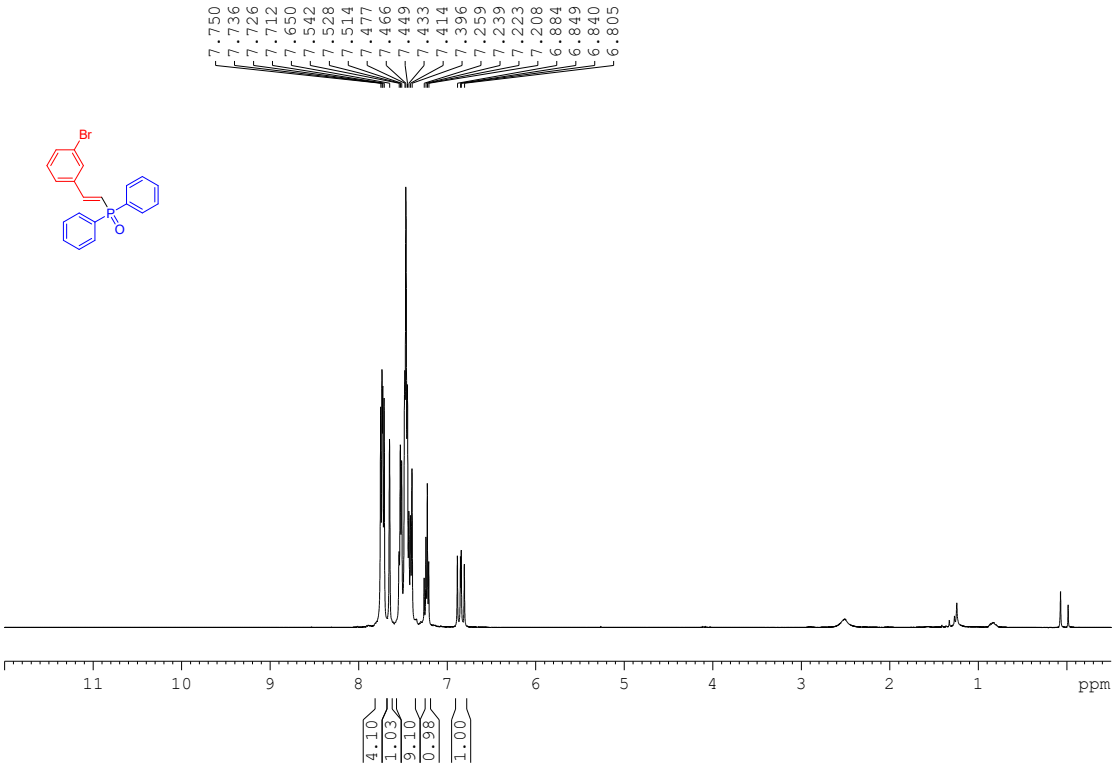
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3k**



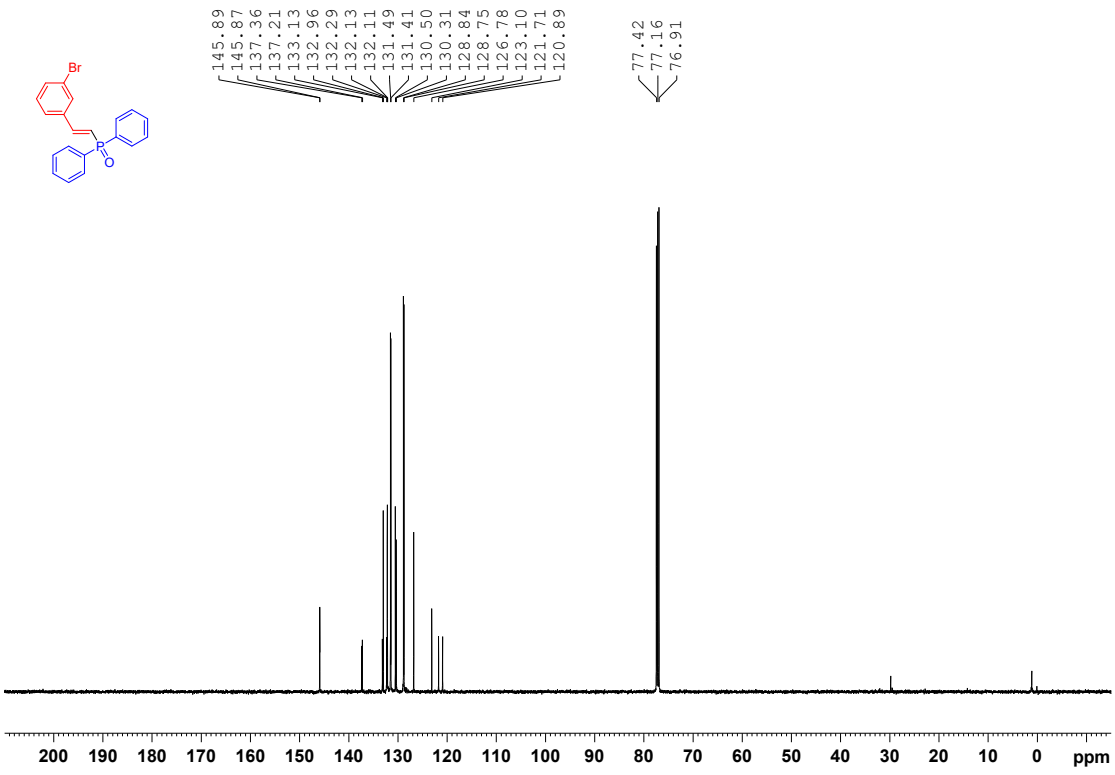
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3k**



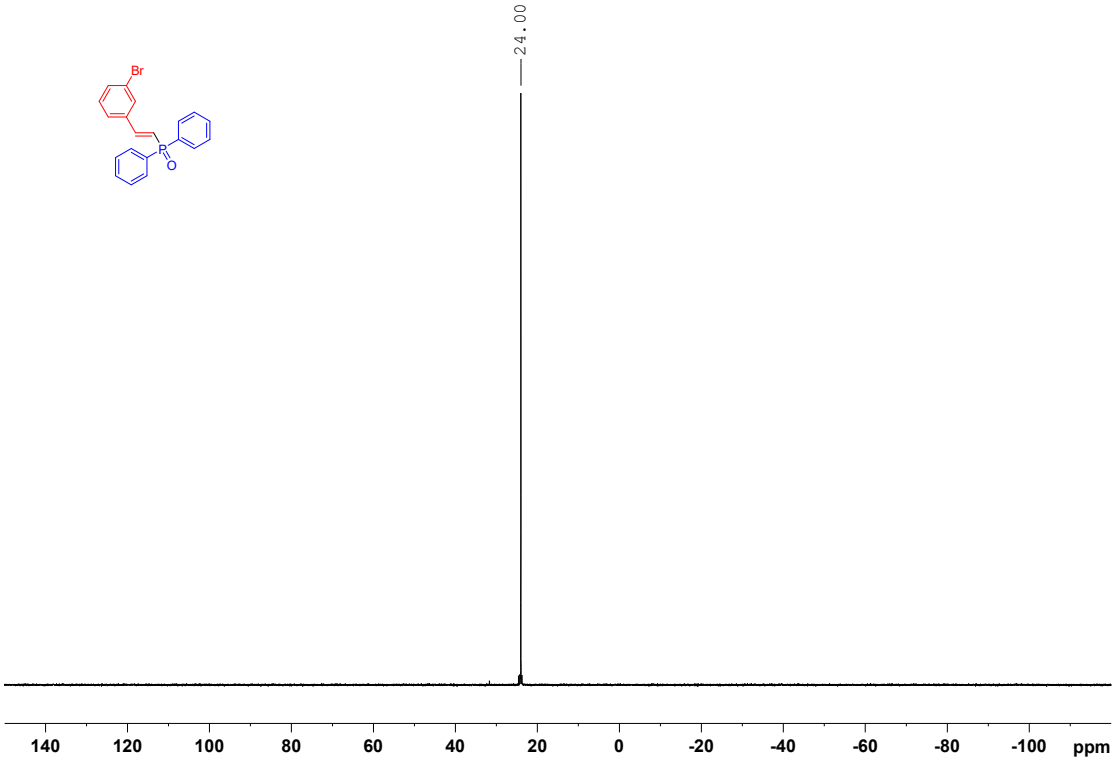
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3l**



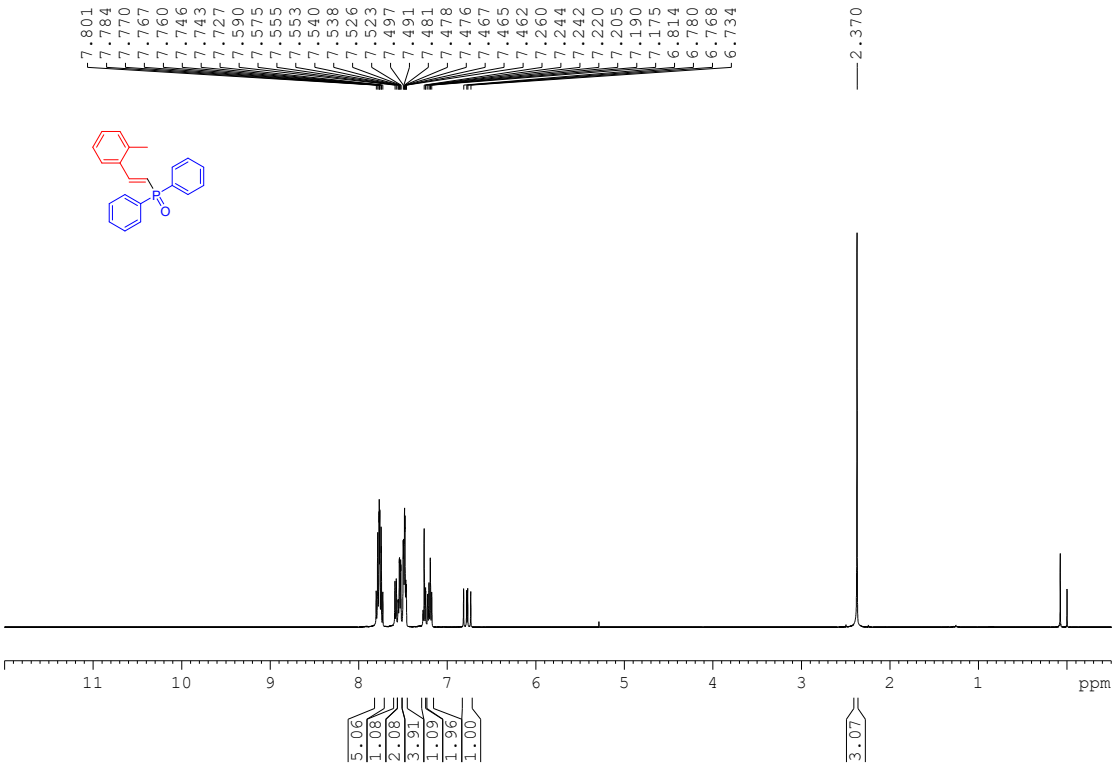
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3l**



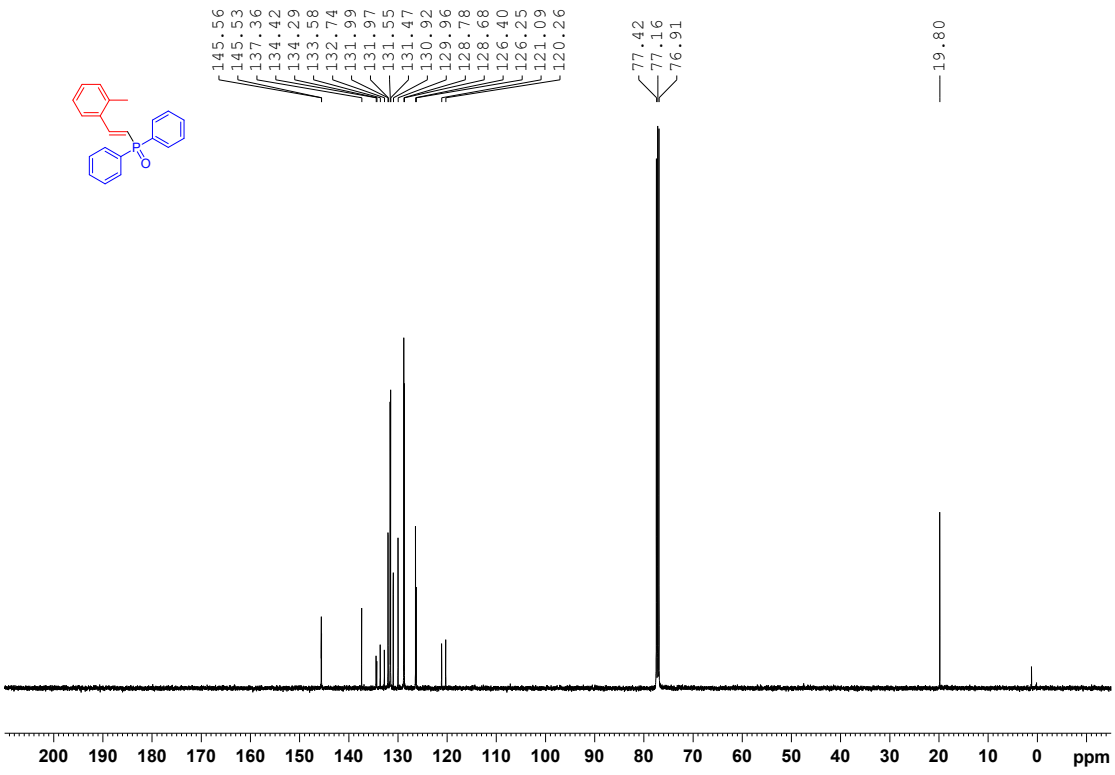
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3l**



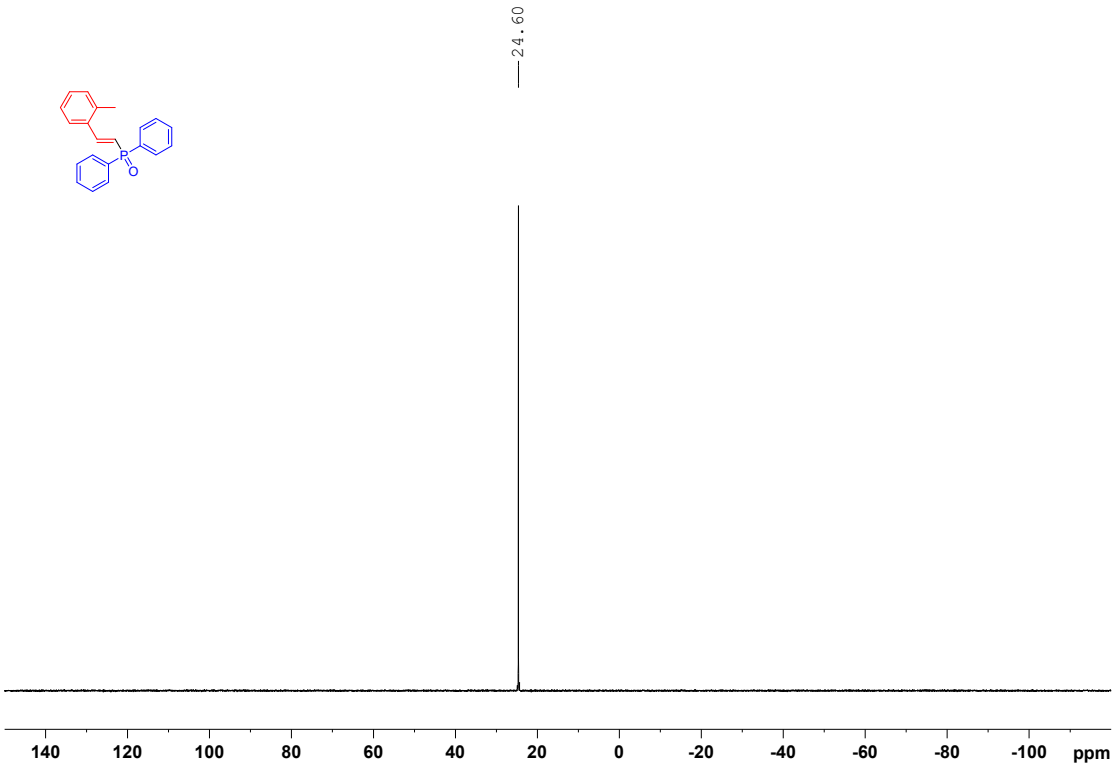
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3m**



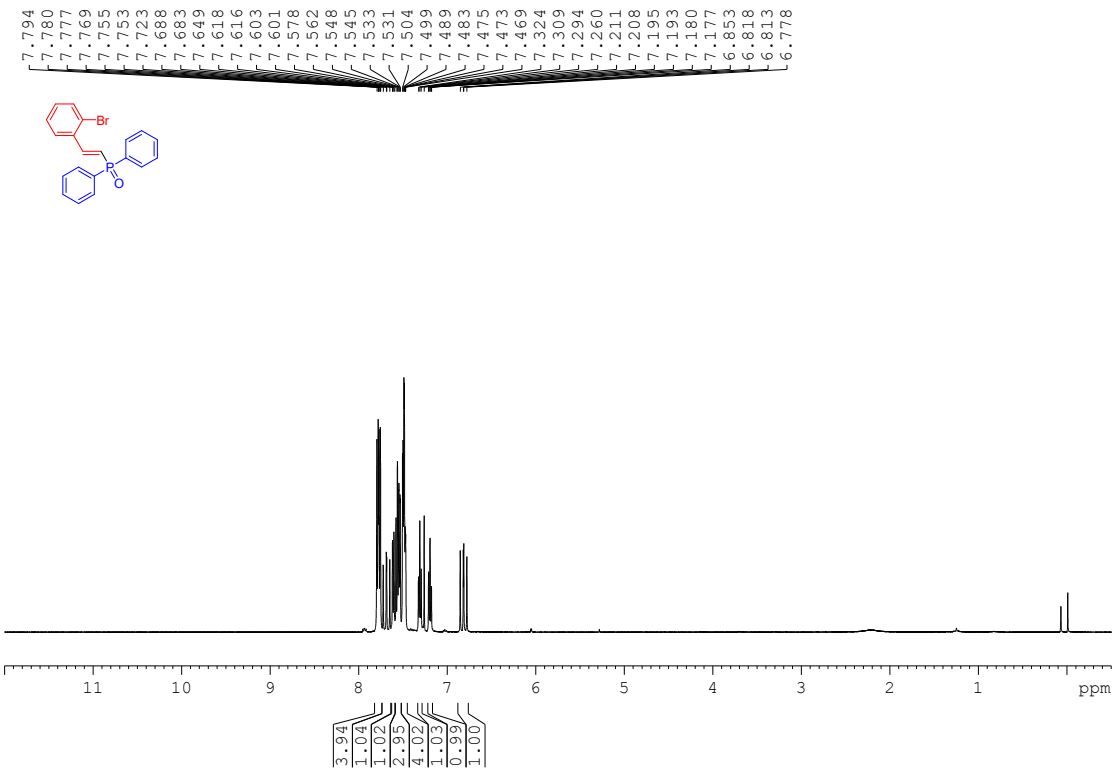
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3m**



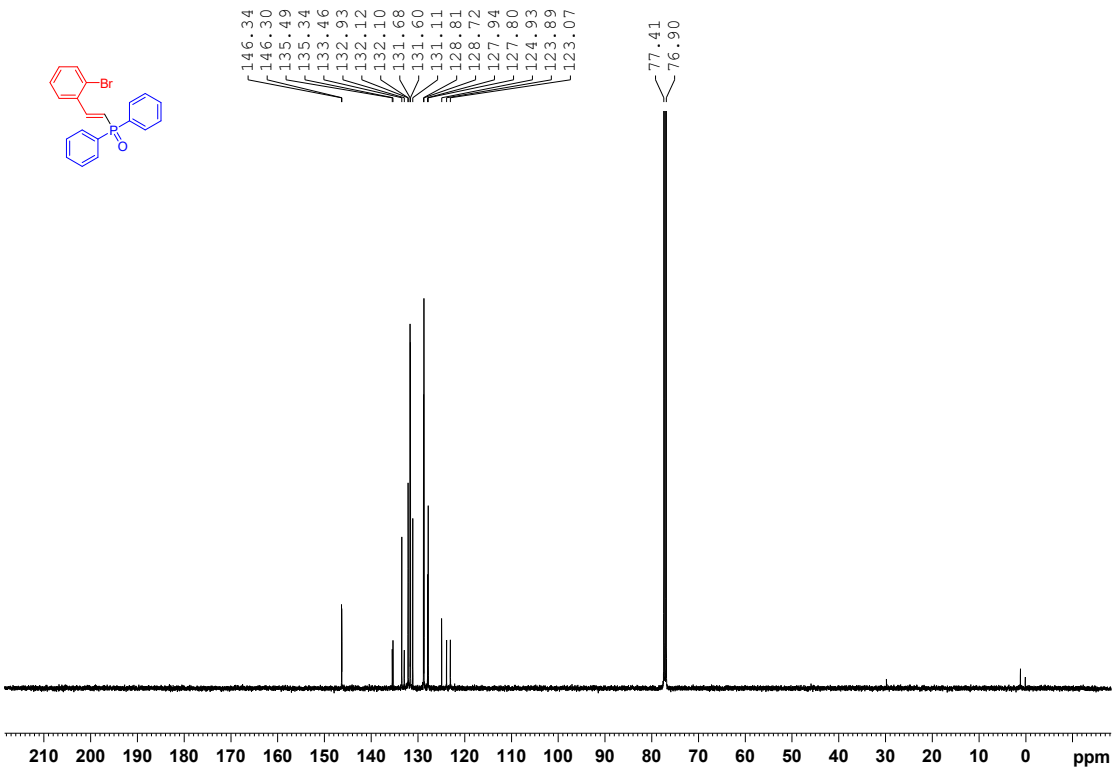
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3m**



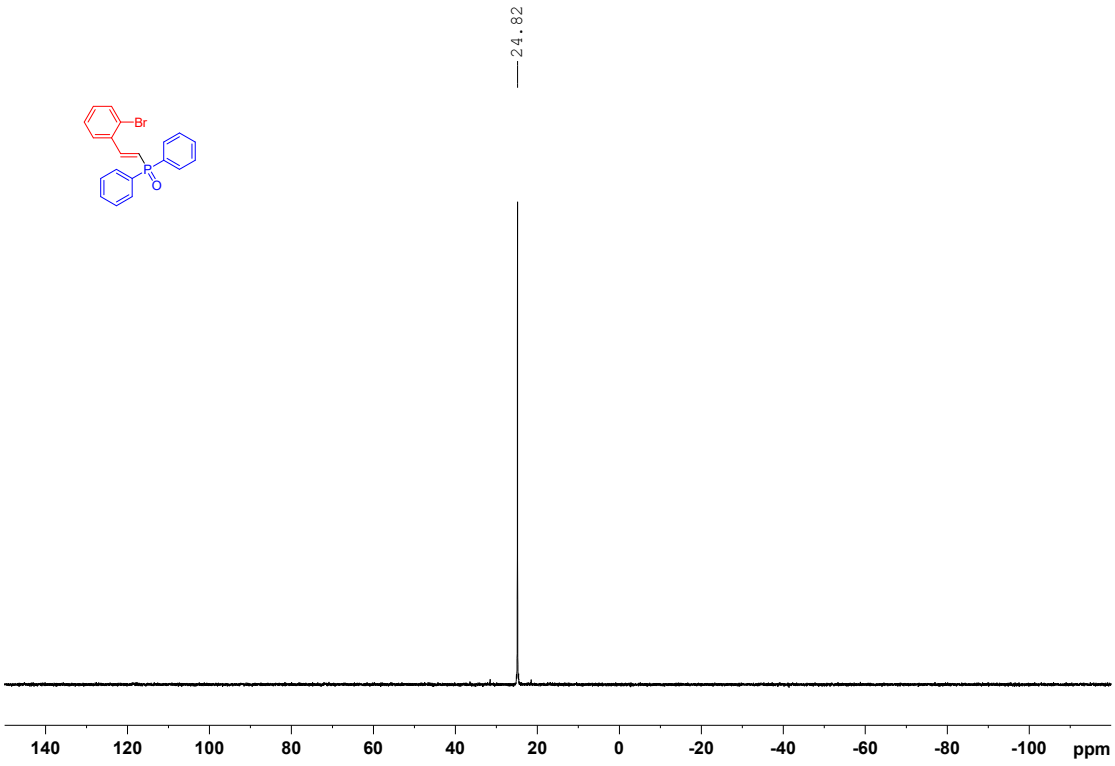
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3n**



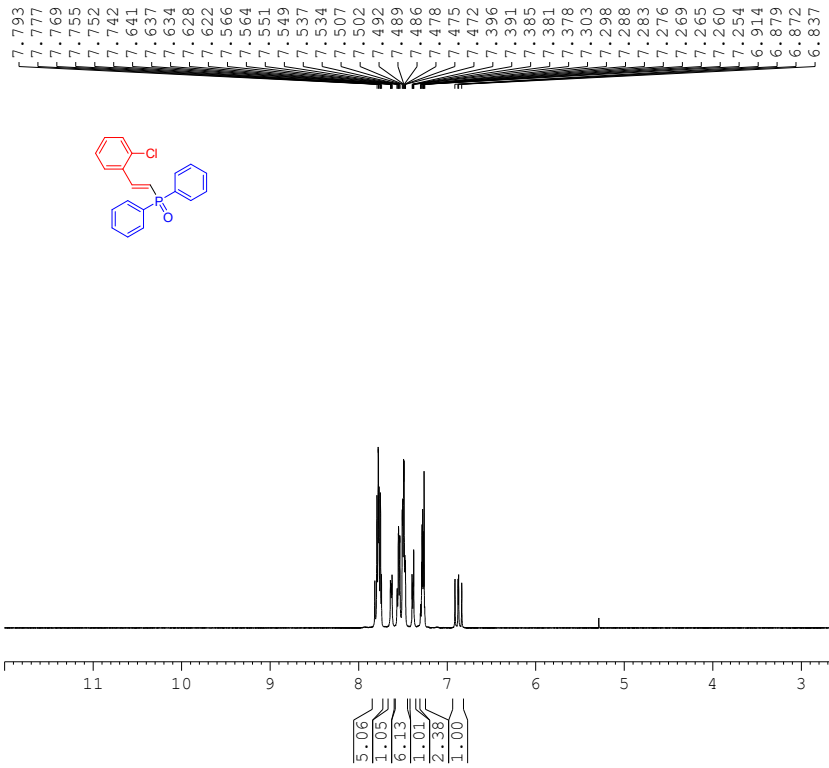
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3n**



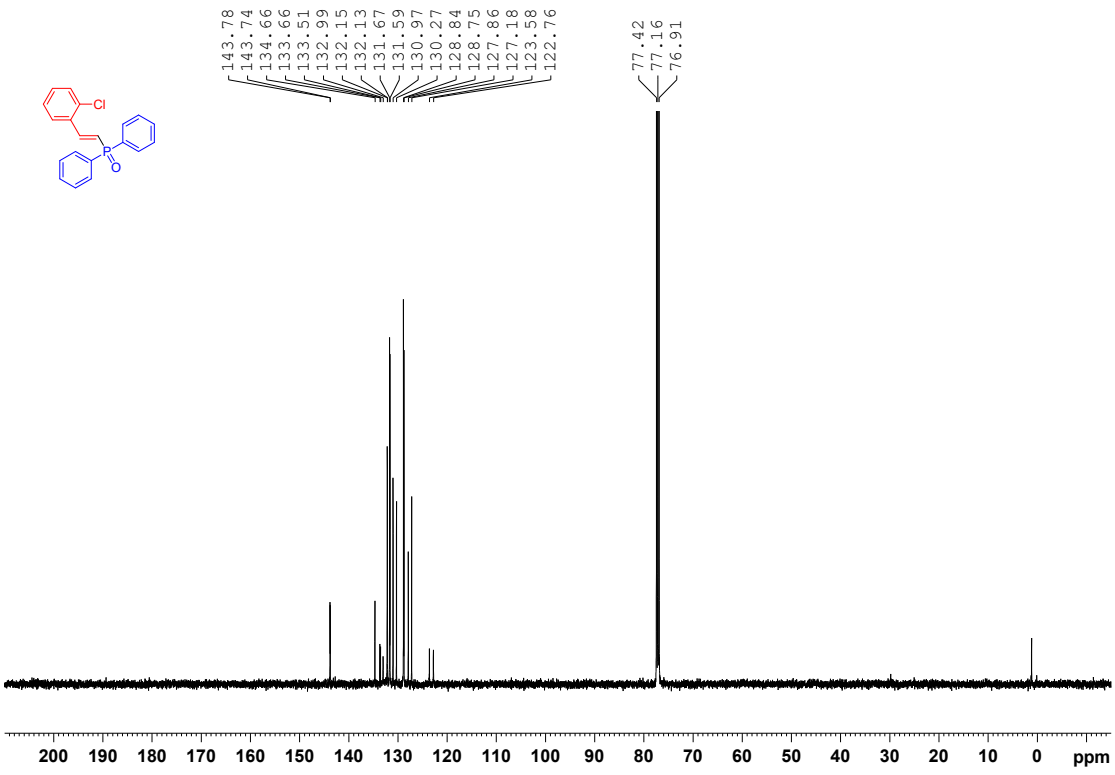
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3n**



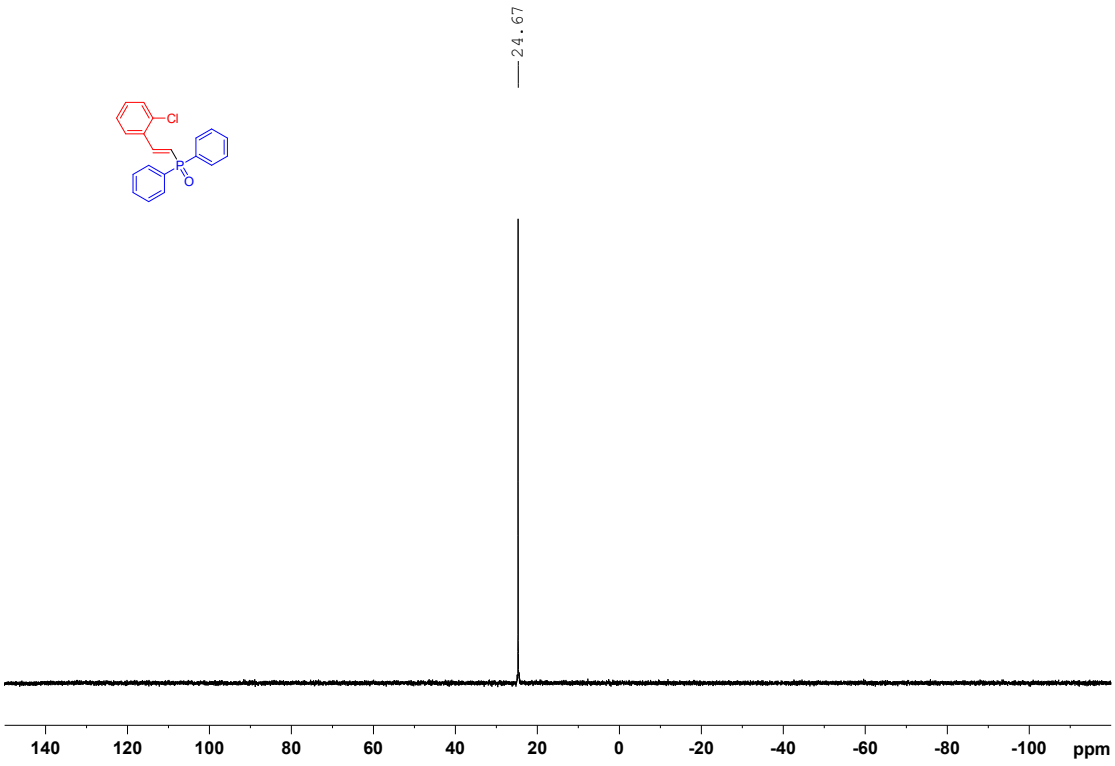
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3o**



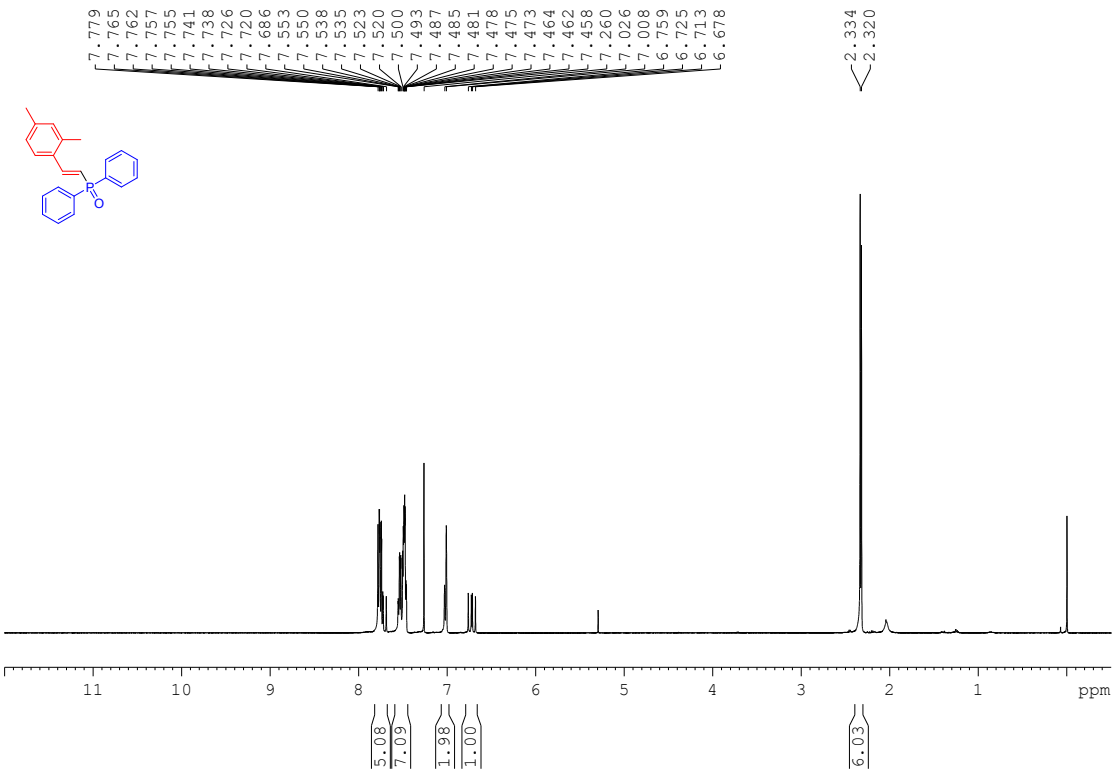
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3o**



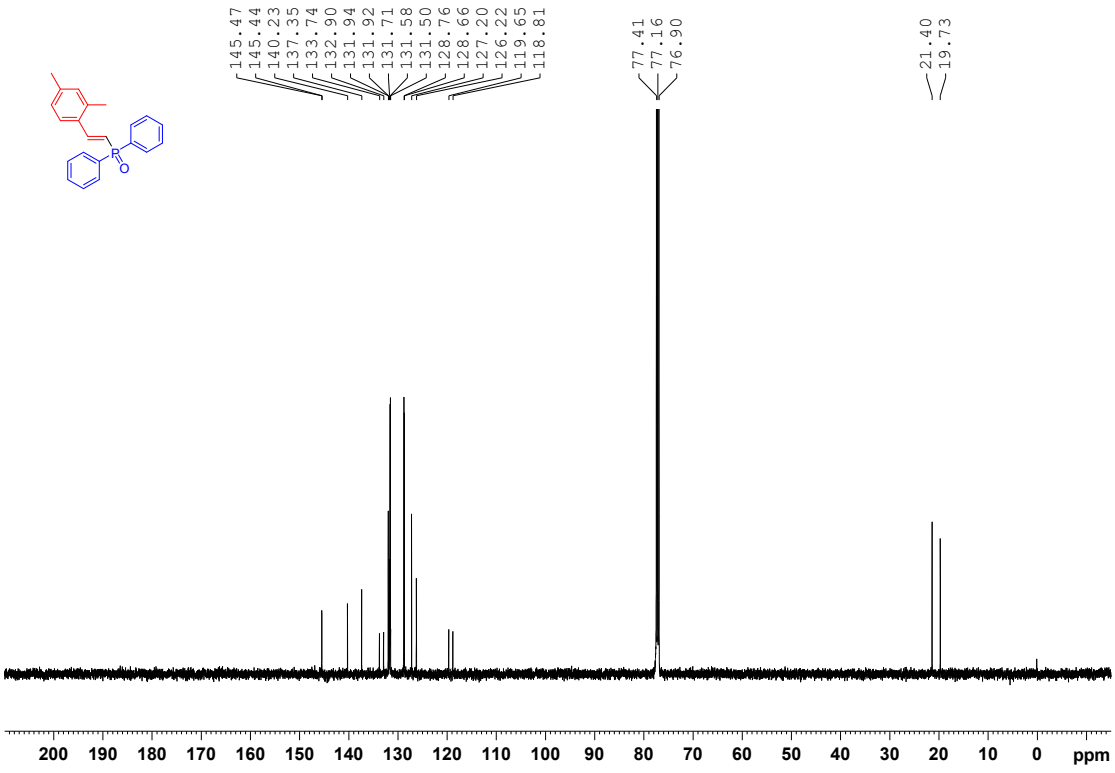
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3o**



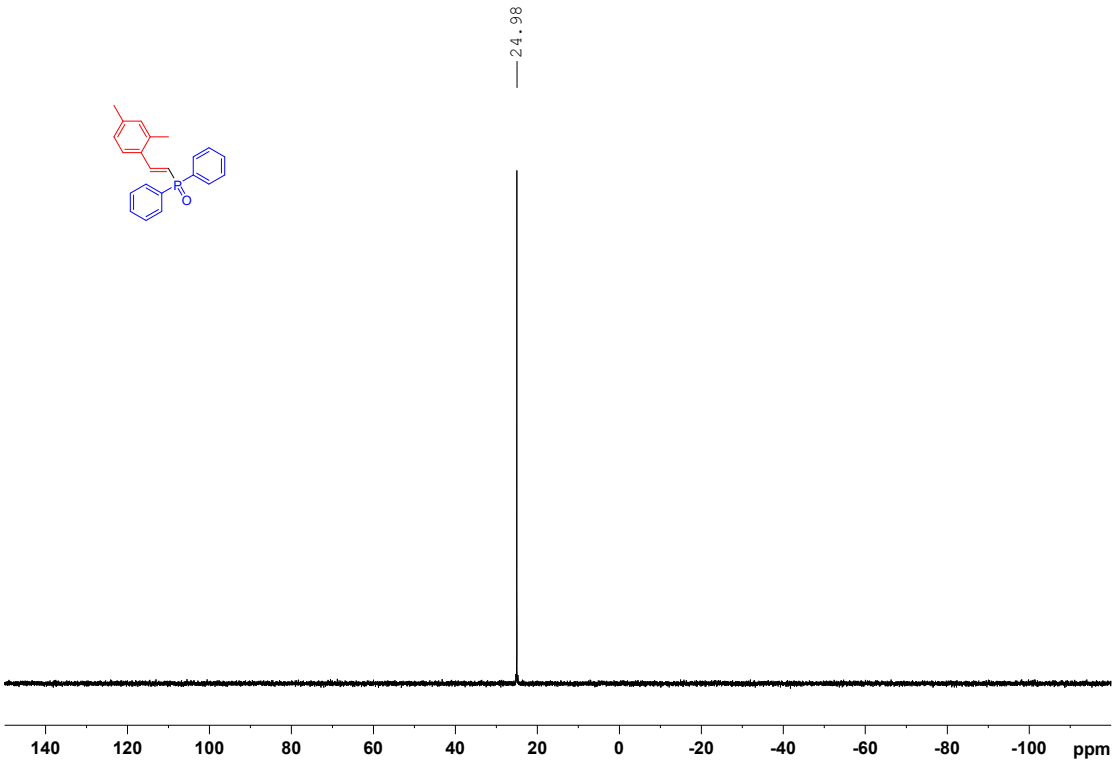
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3p**



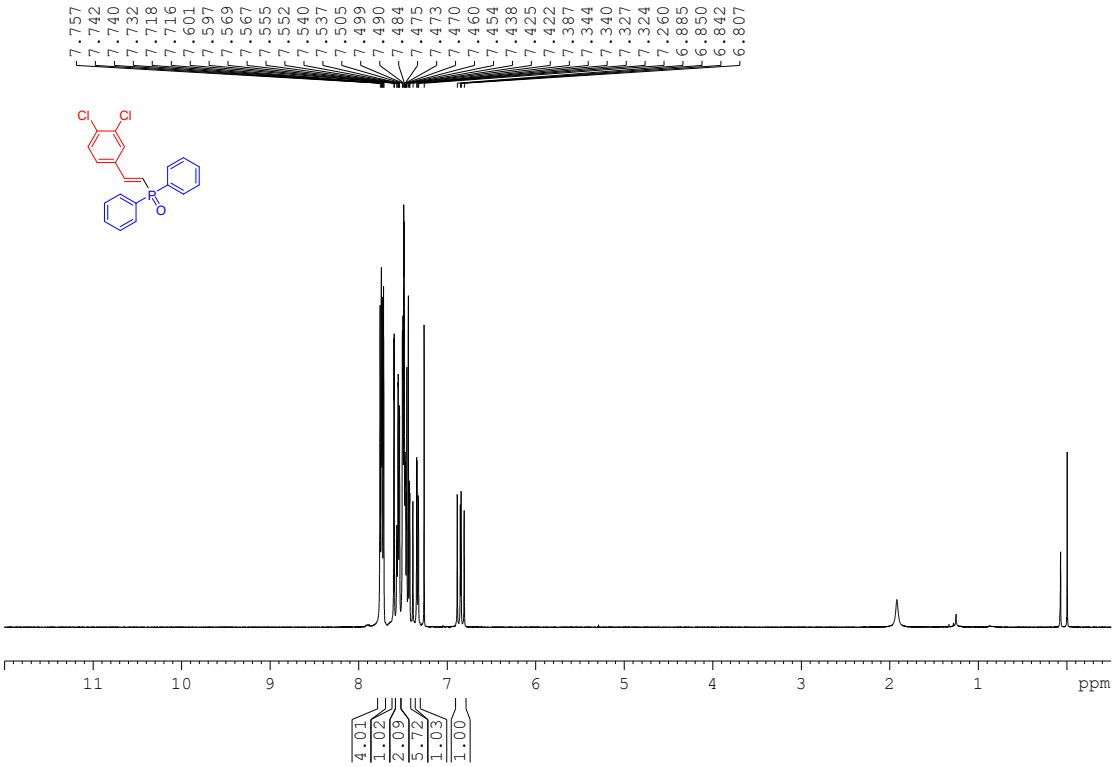
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3p**



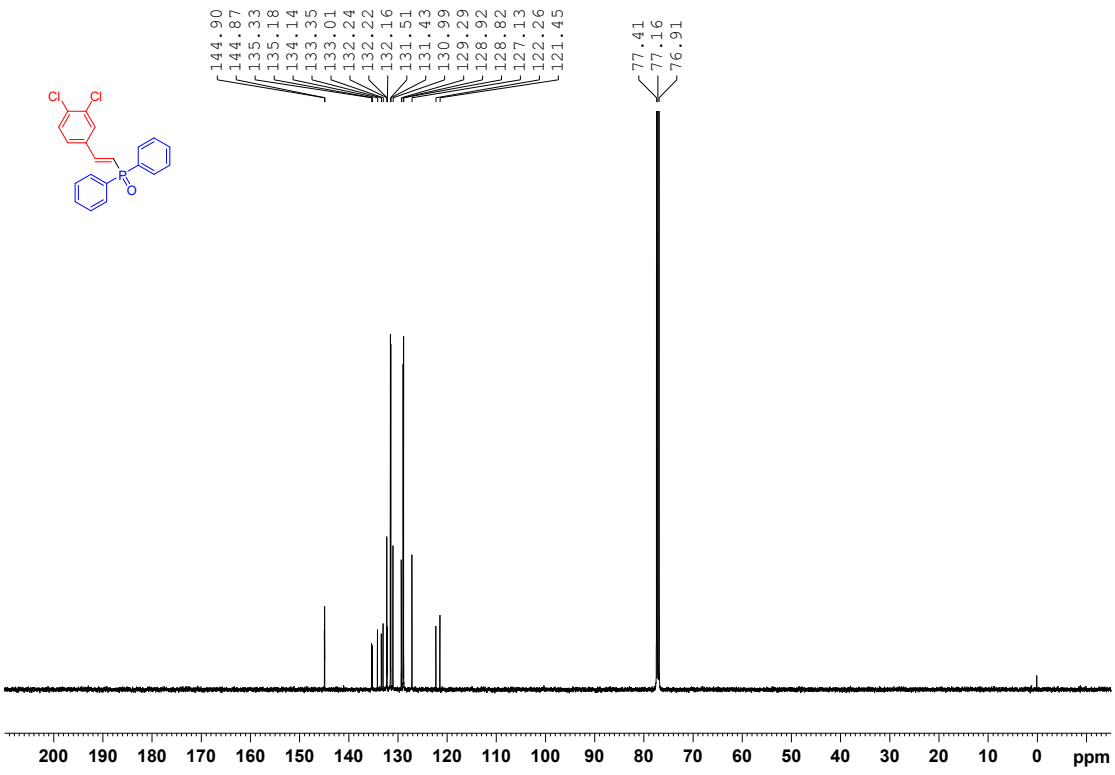
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3p**



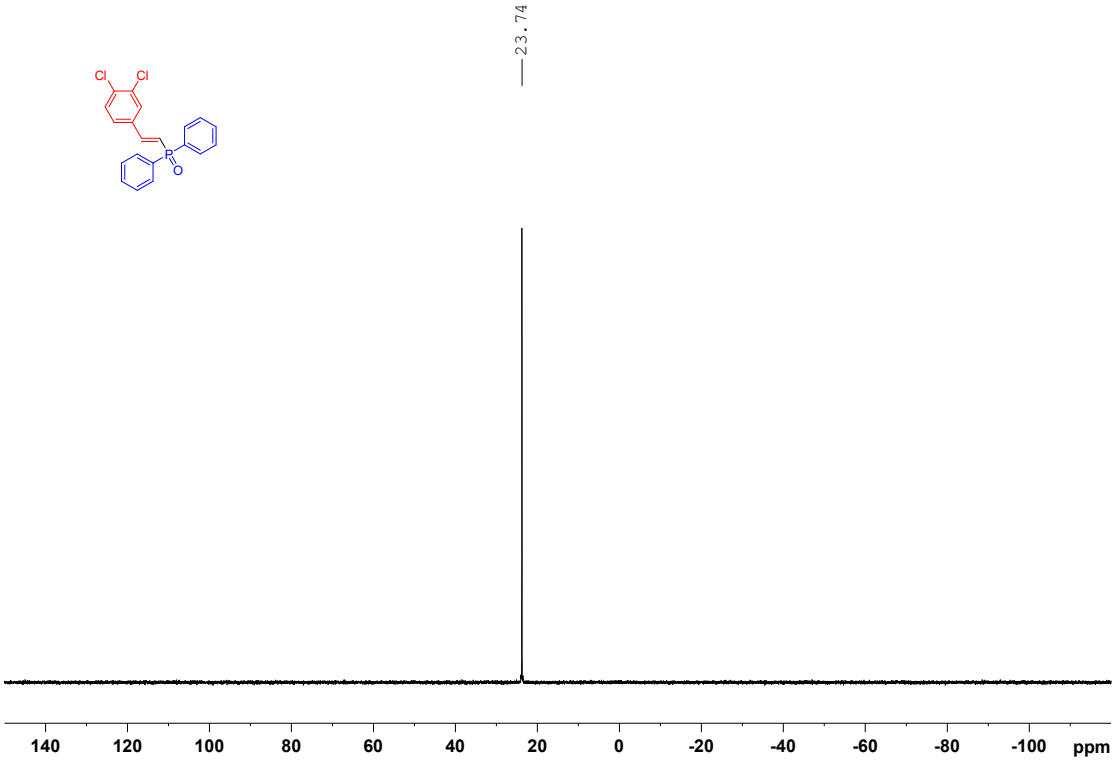
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3q**



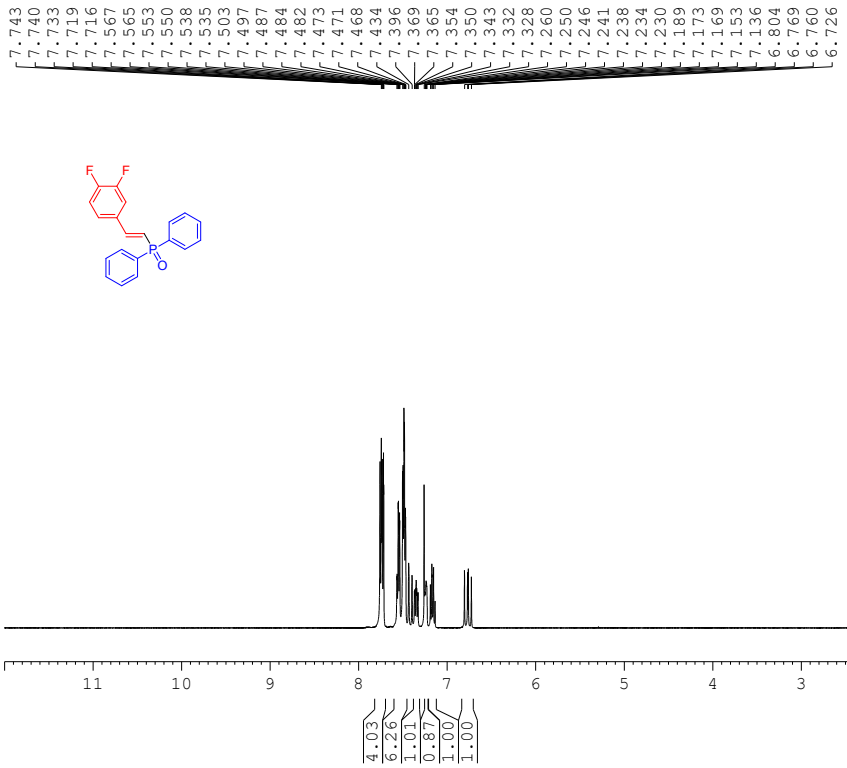
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3q**



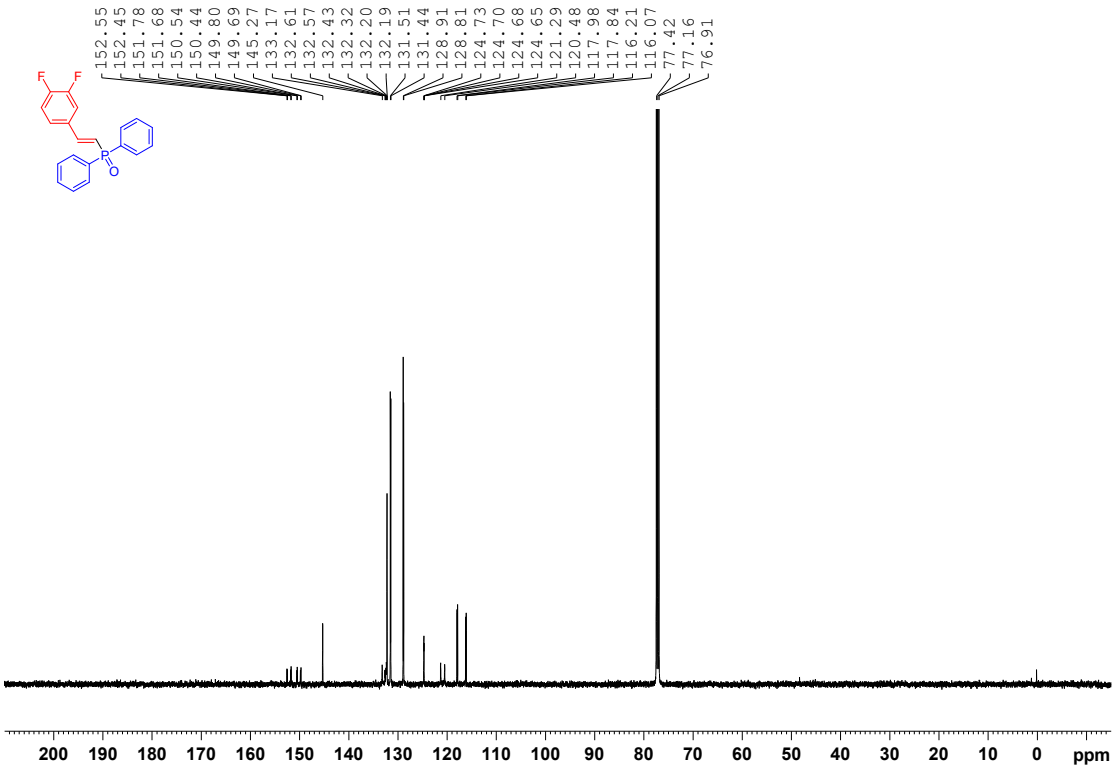
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3q**



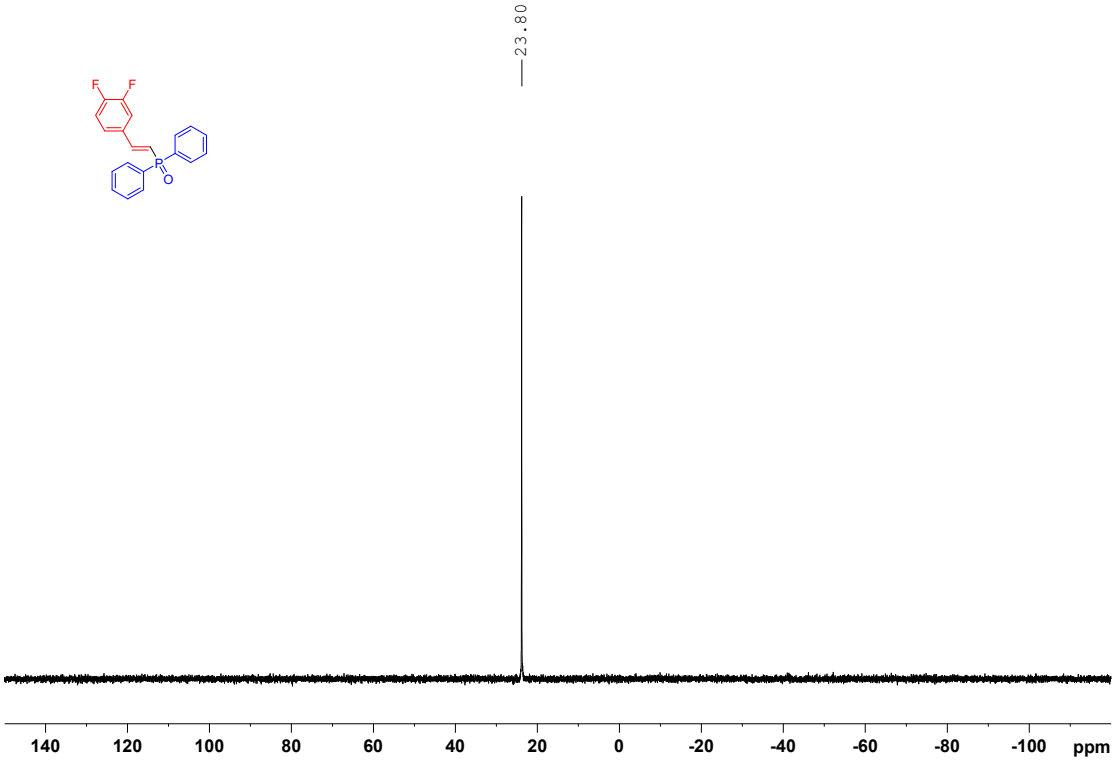
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3r**



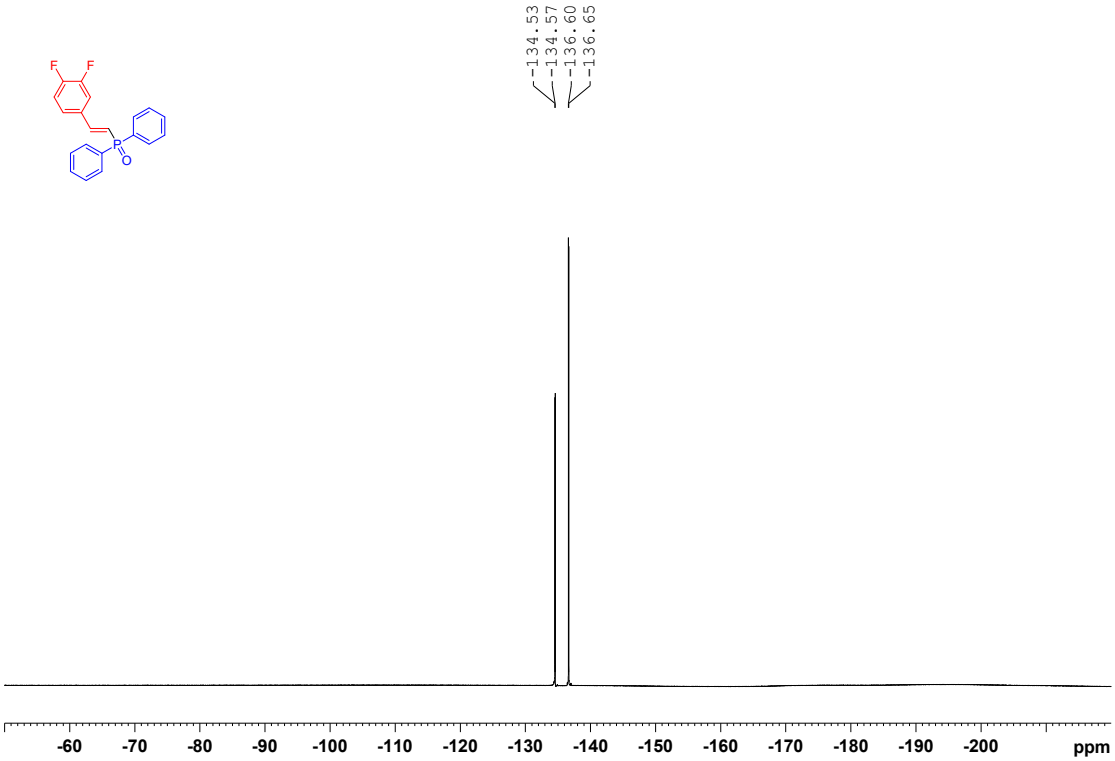
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3r**



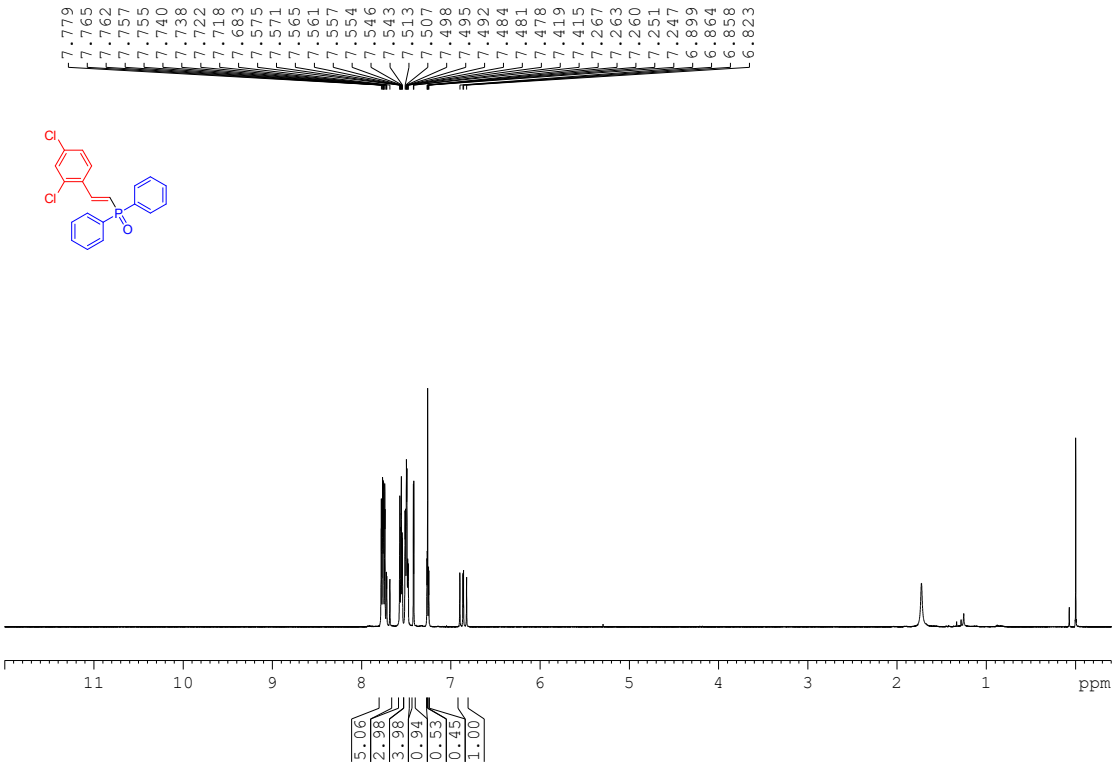
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3r**



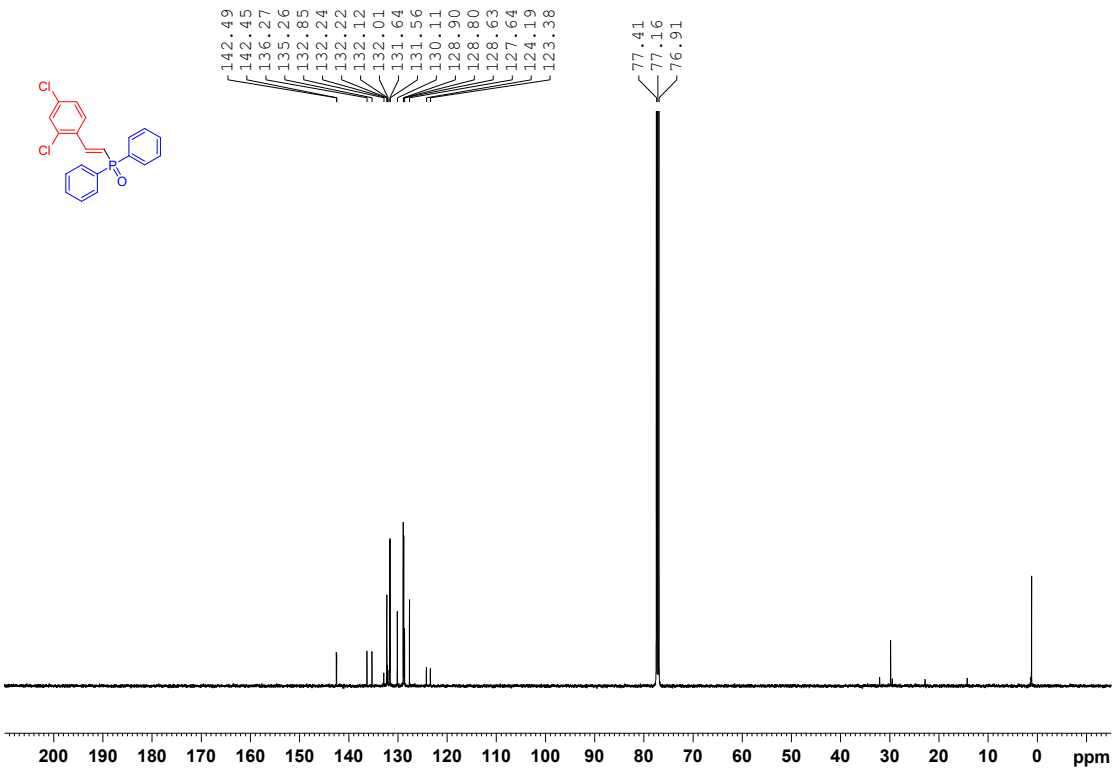
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **3r**



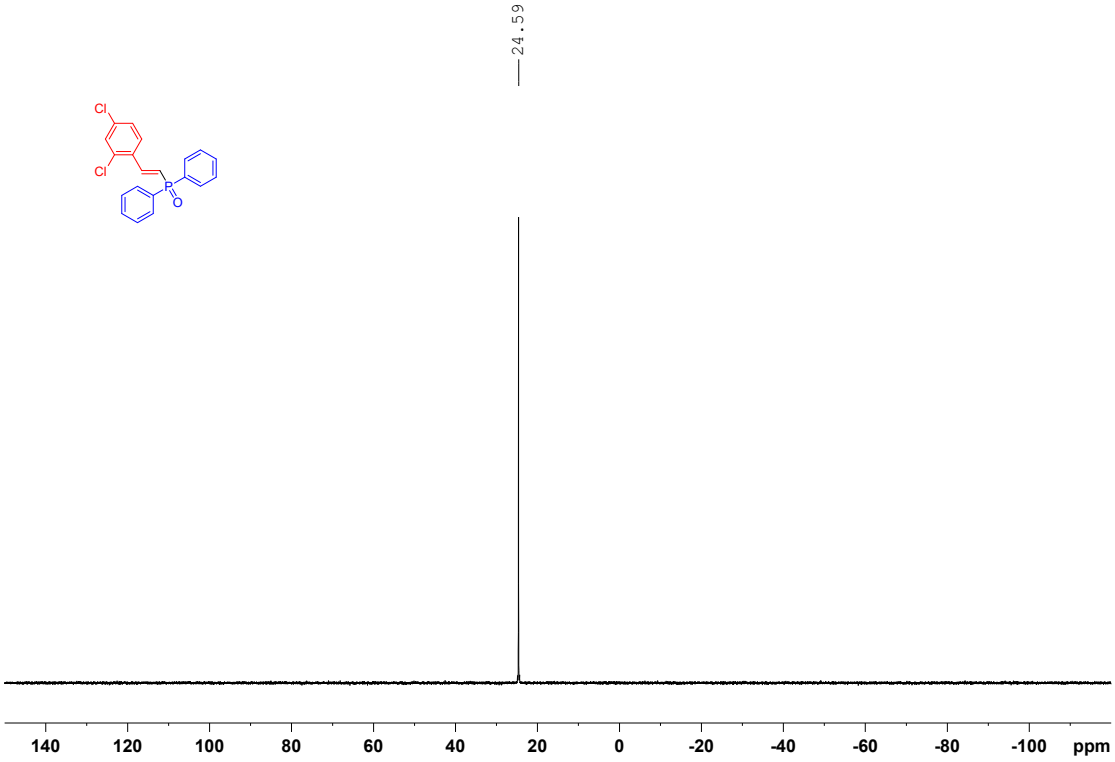
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3s**



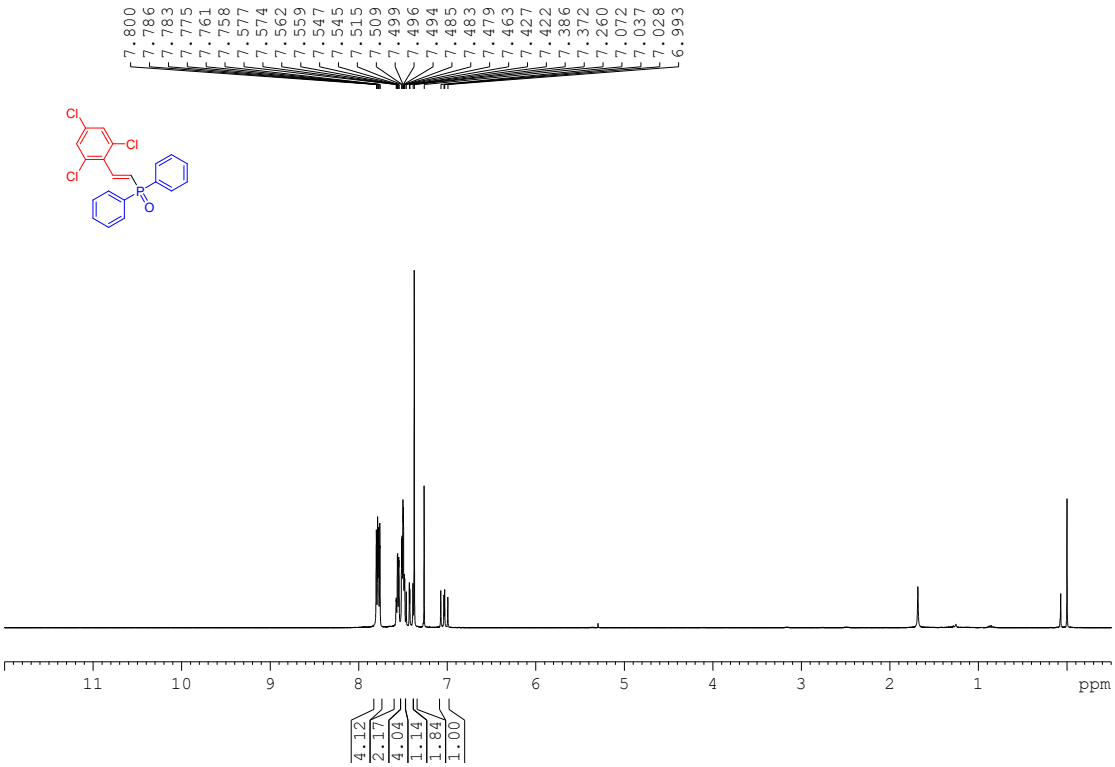
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3s**



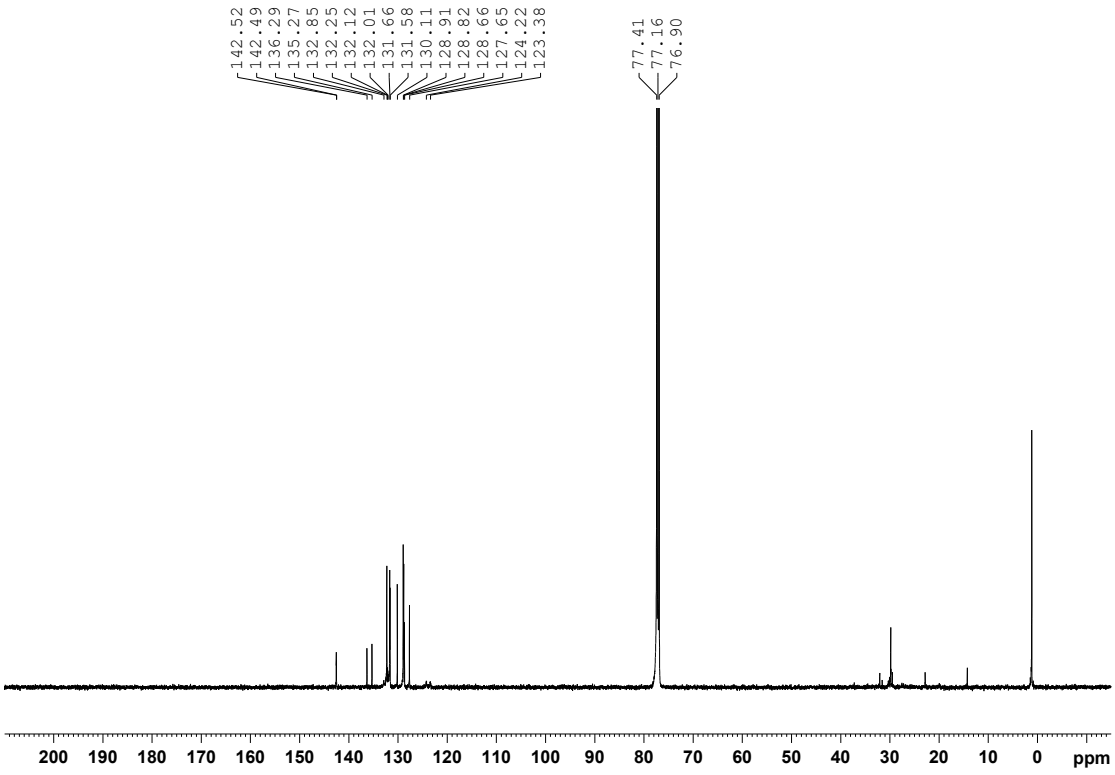
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3s**



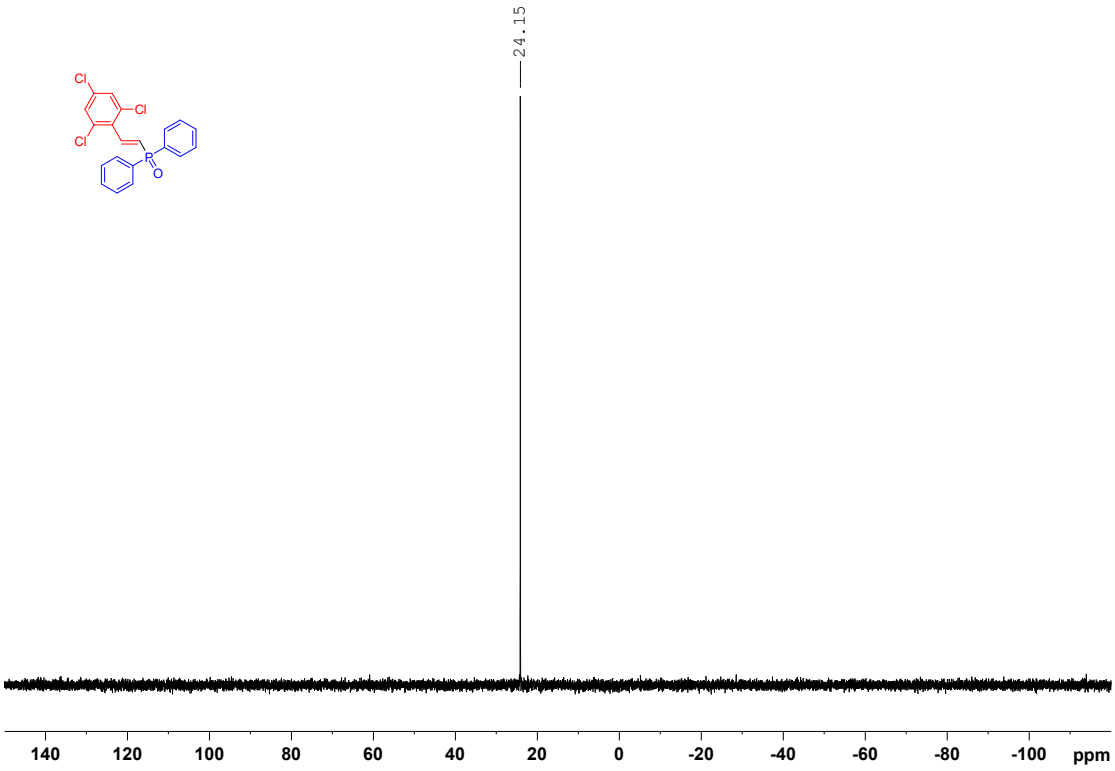
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3t**



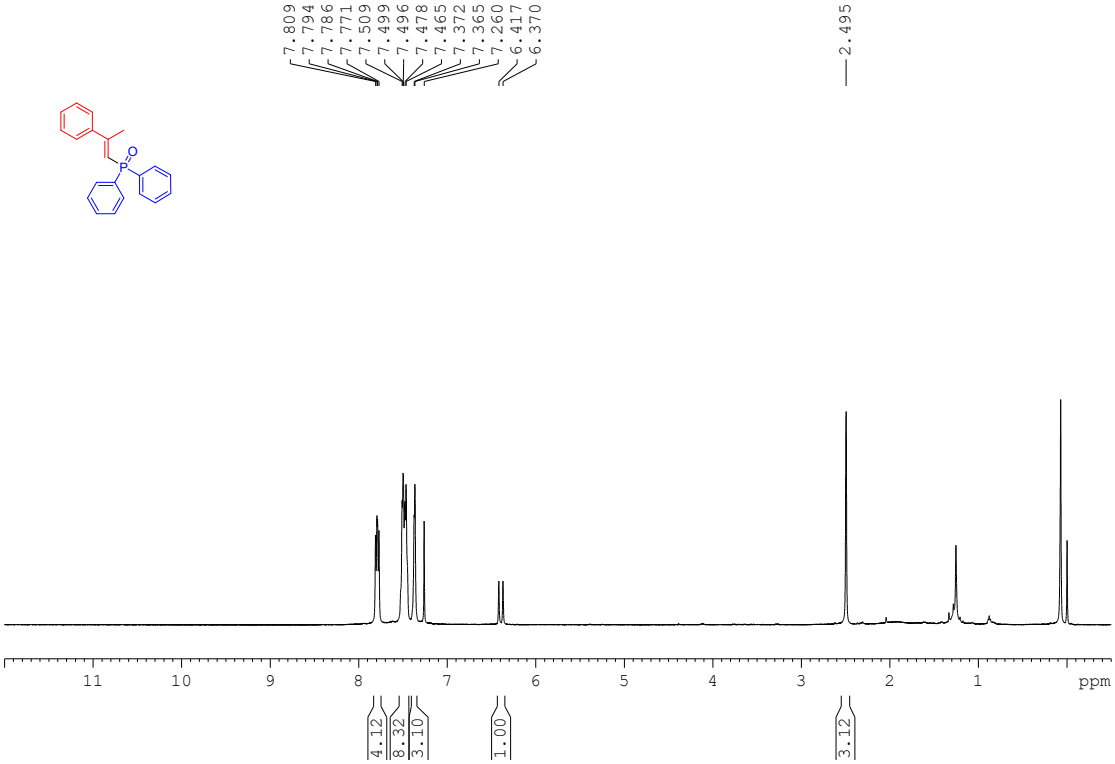
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3t**



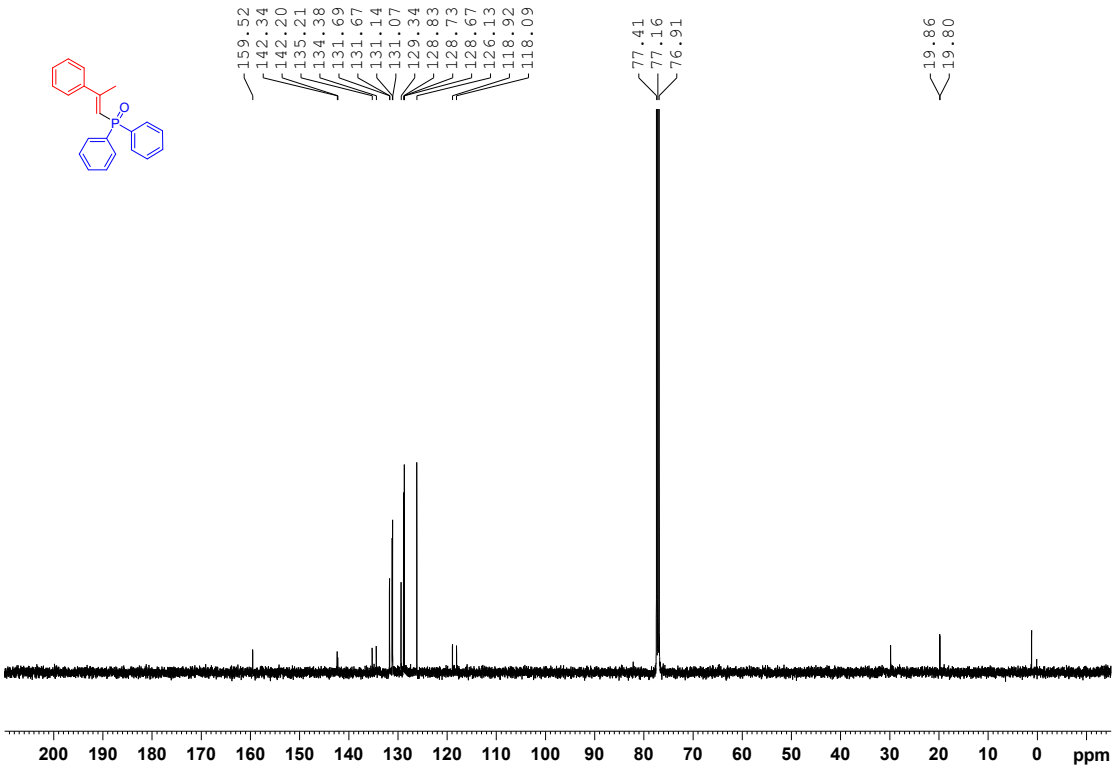
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3t**



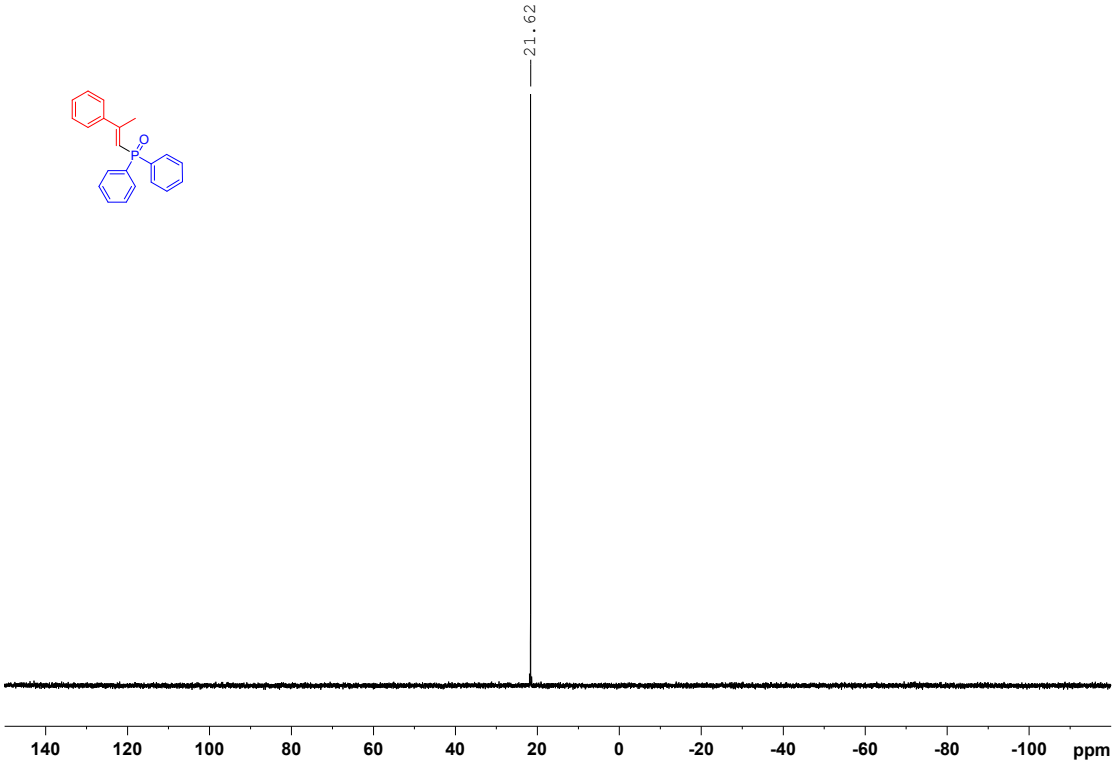
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3u**



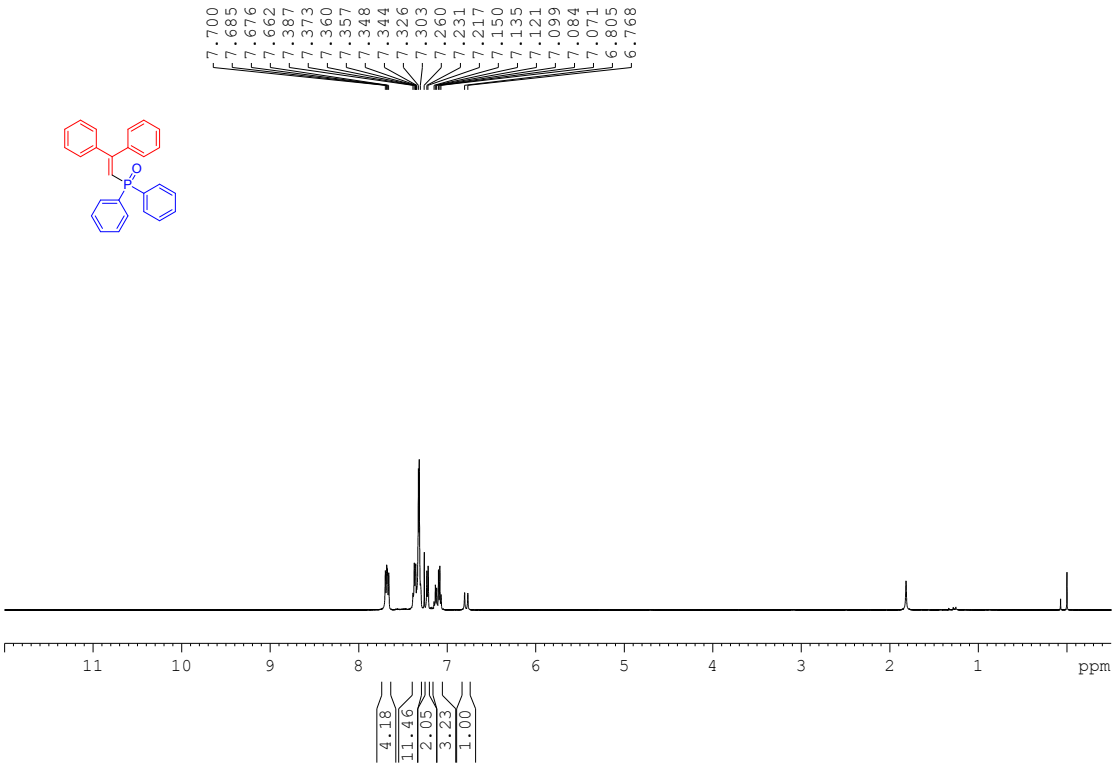
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3u**



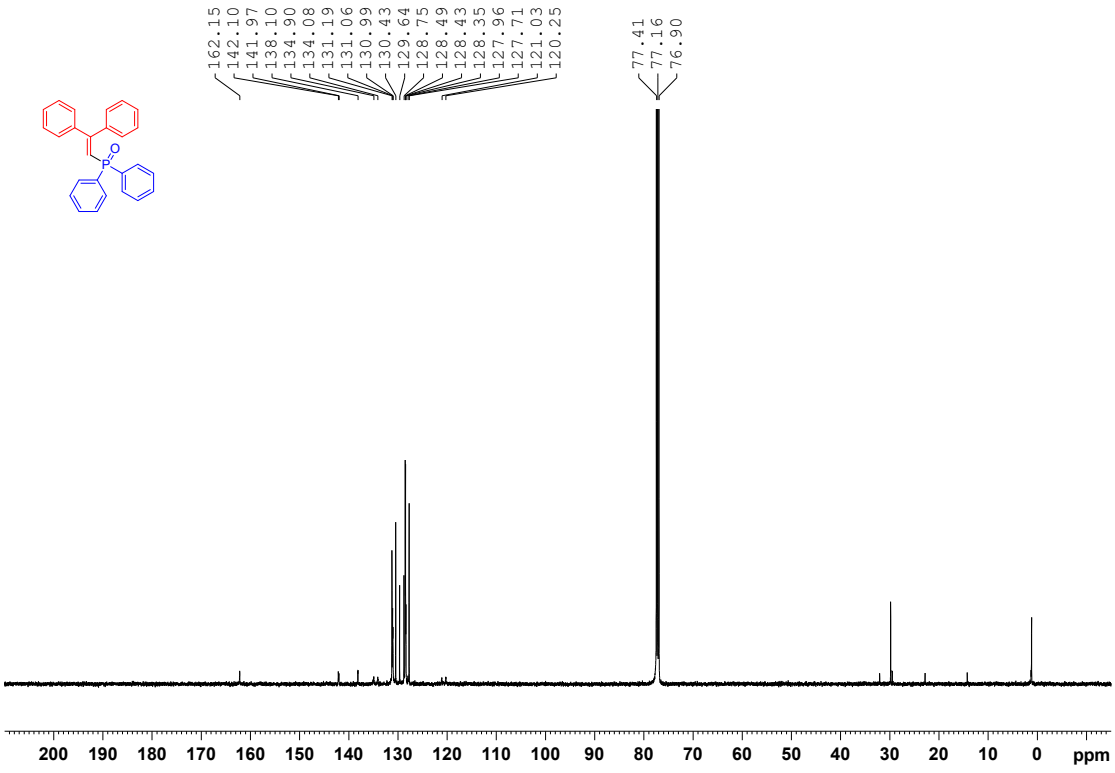
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3u**



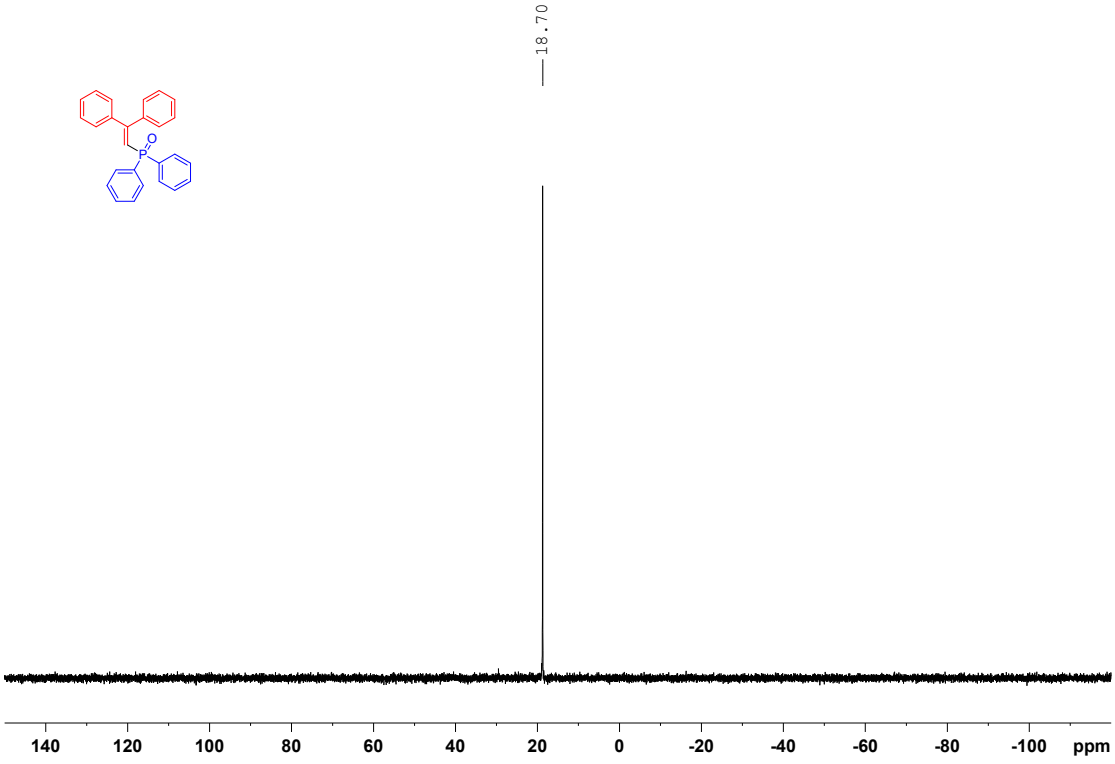
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3v**



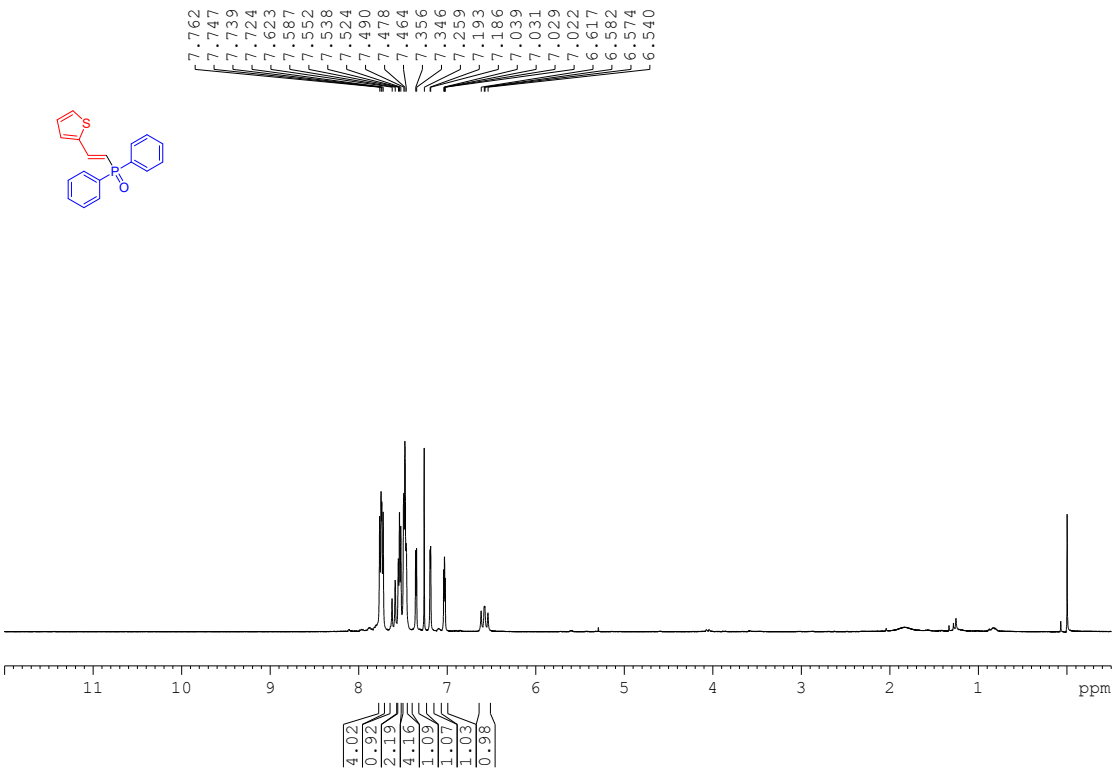
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3v**



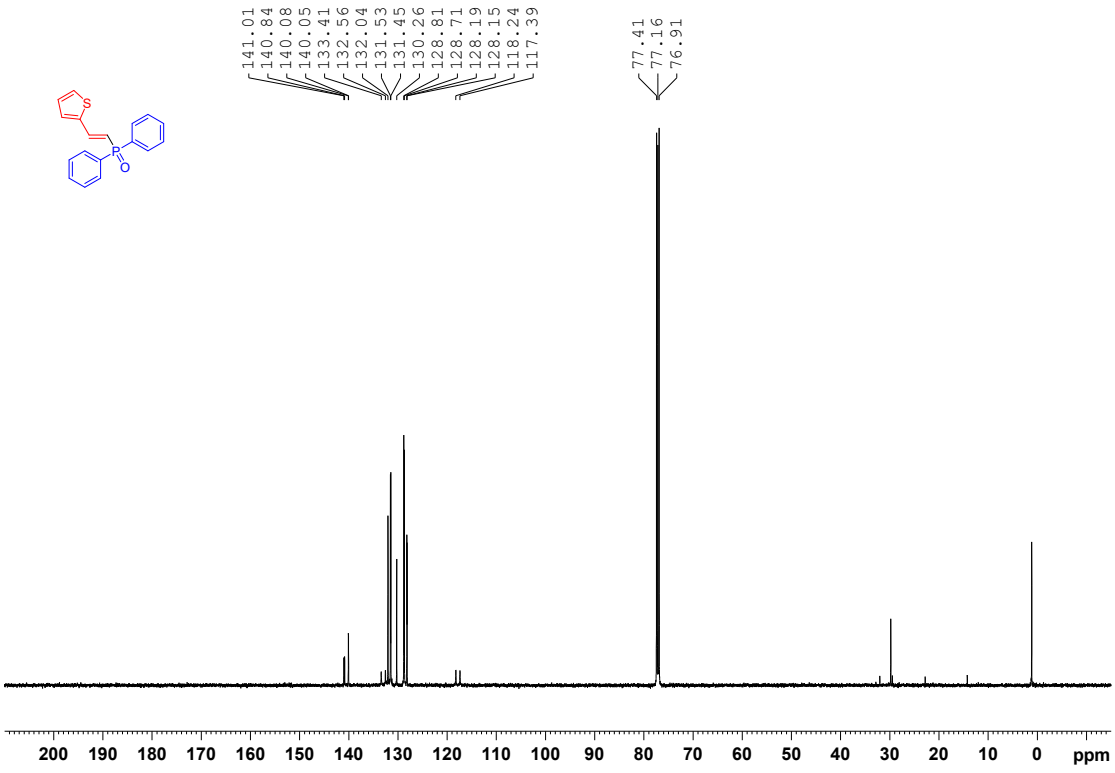
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3v**



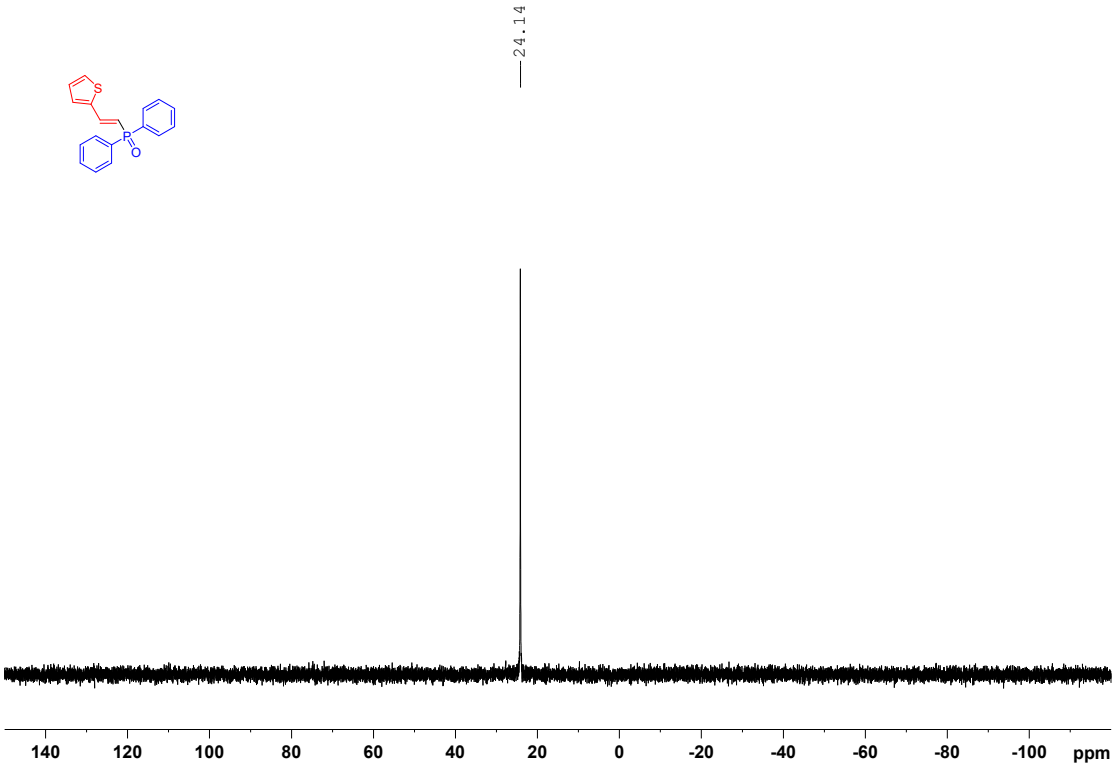
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3w**



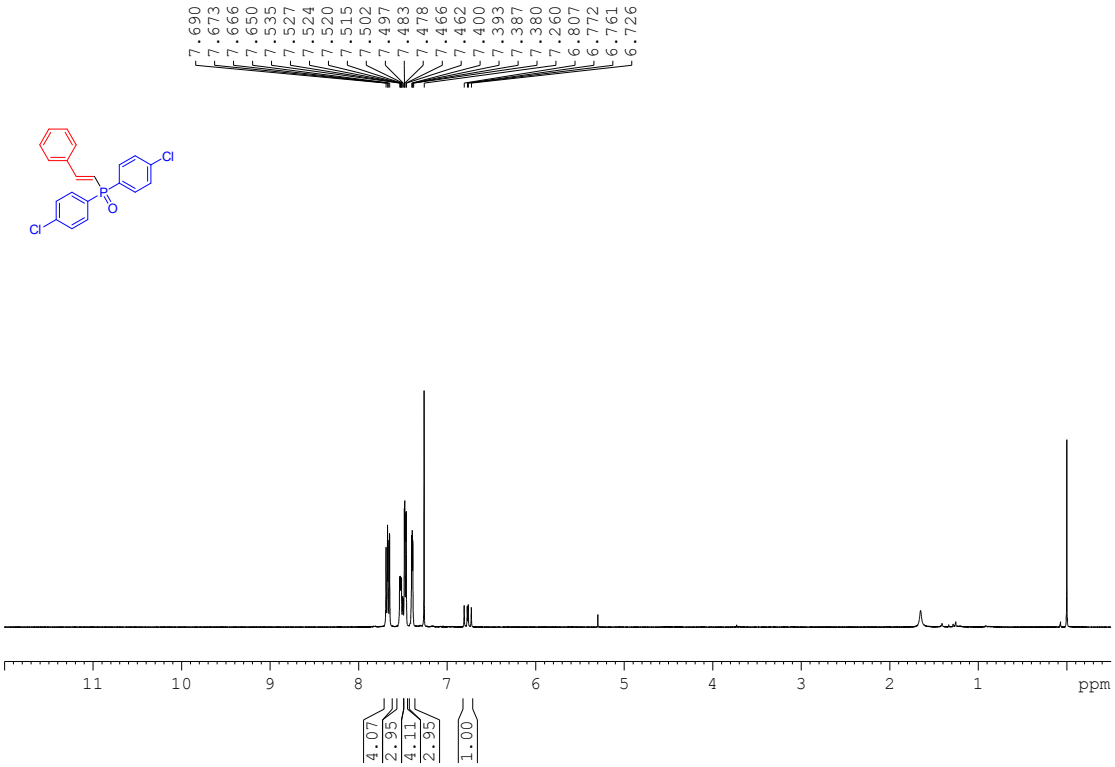
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3w**



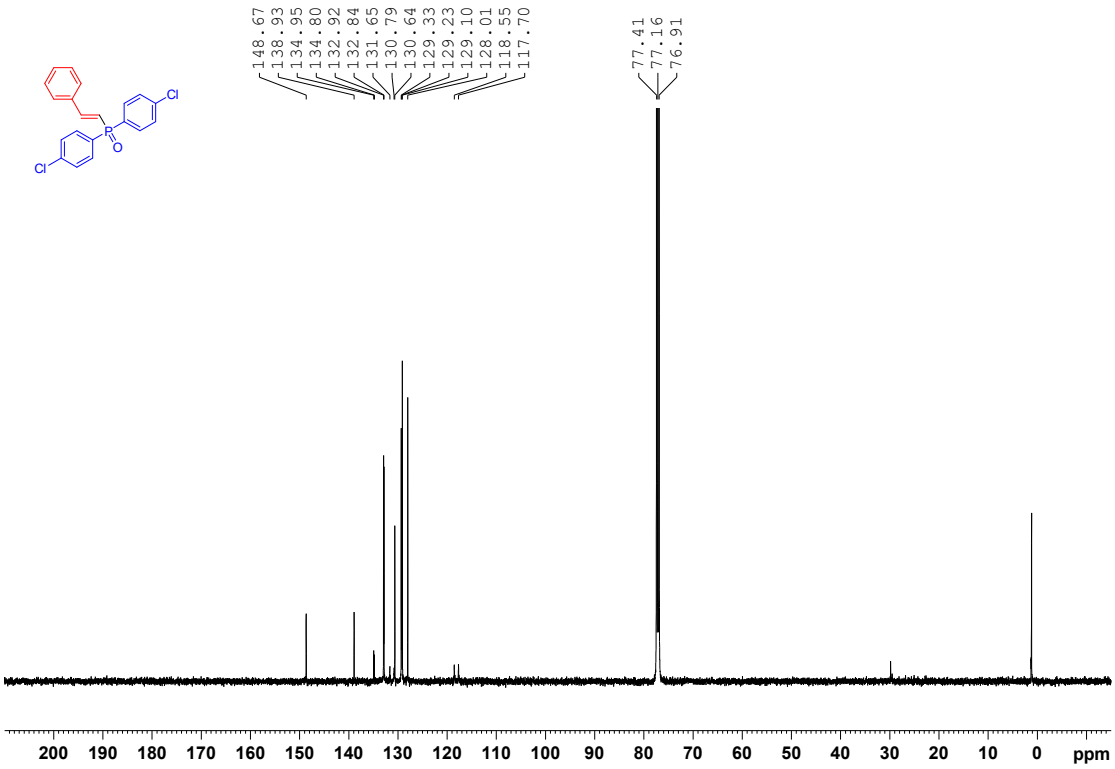
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3w**



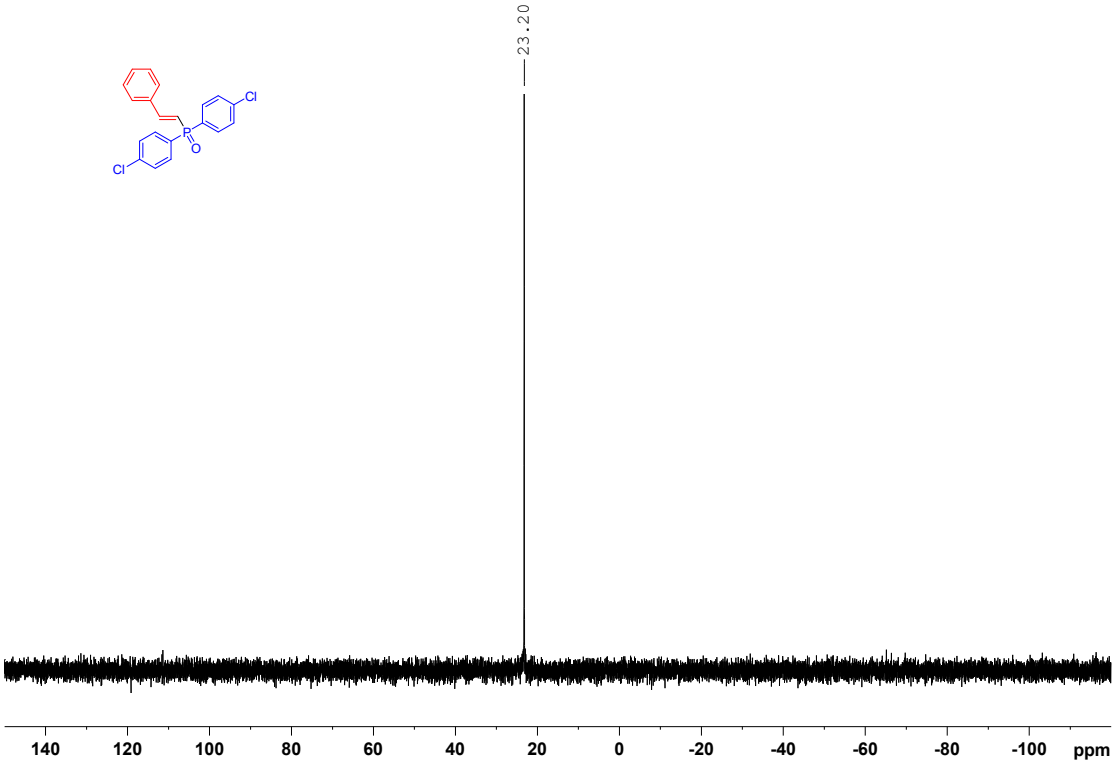
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3x**



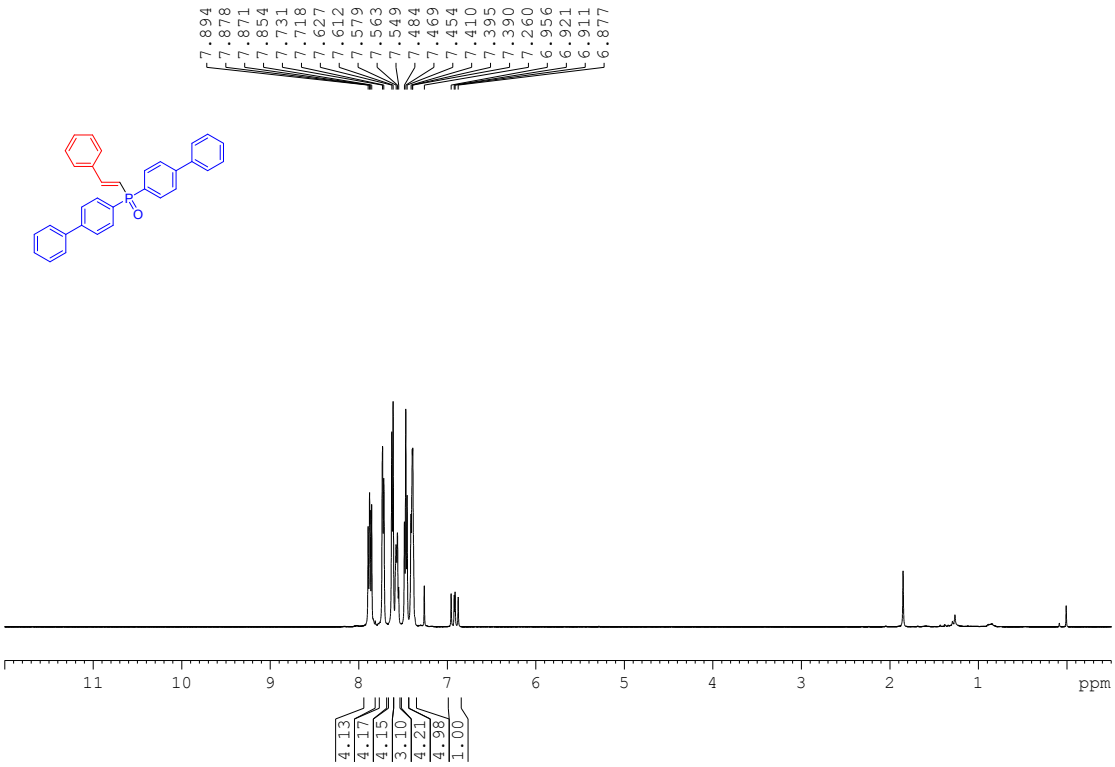
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3x**



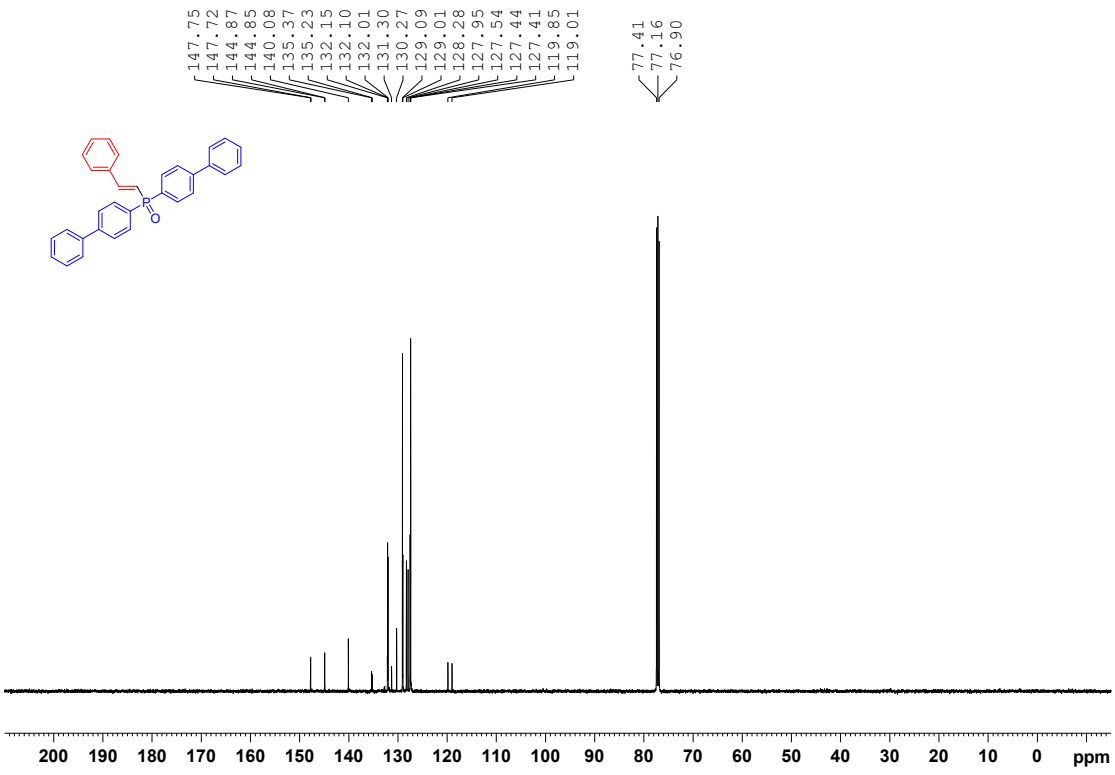
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3x**



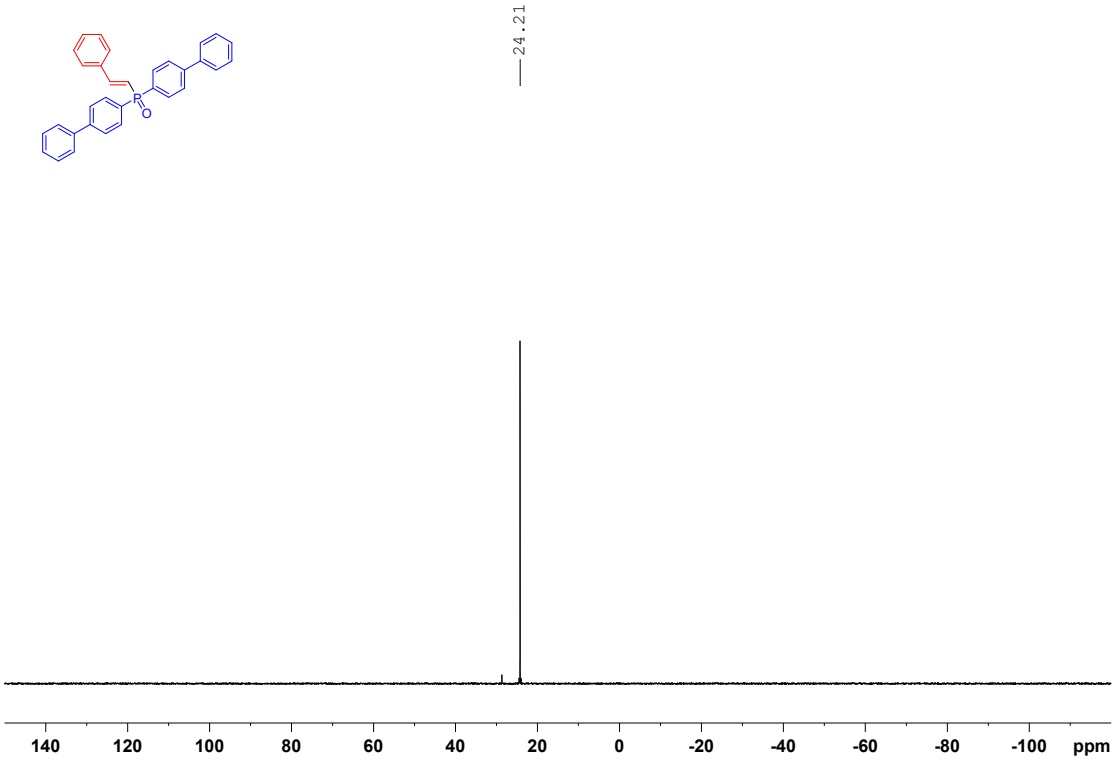
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **3y**



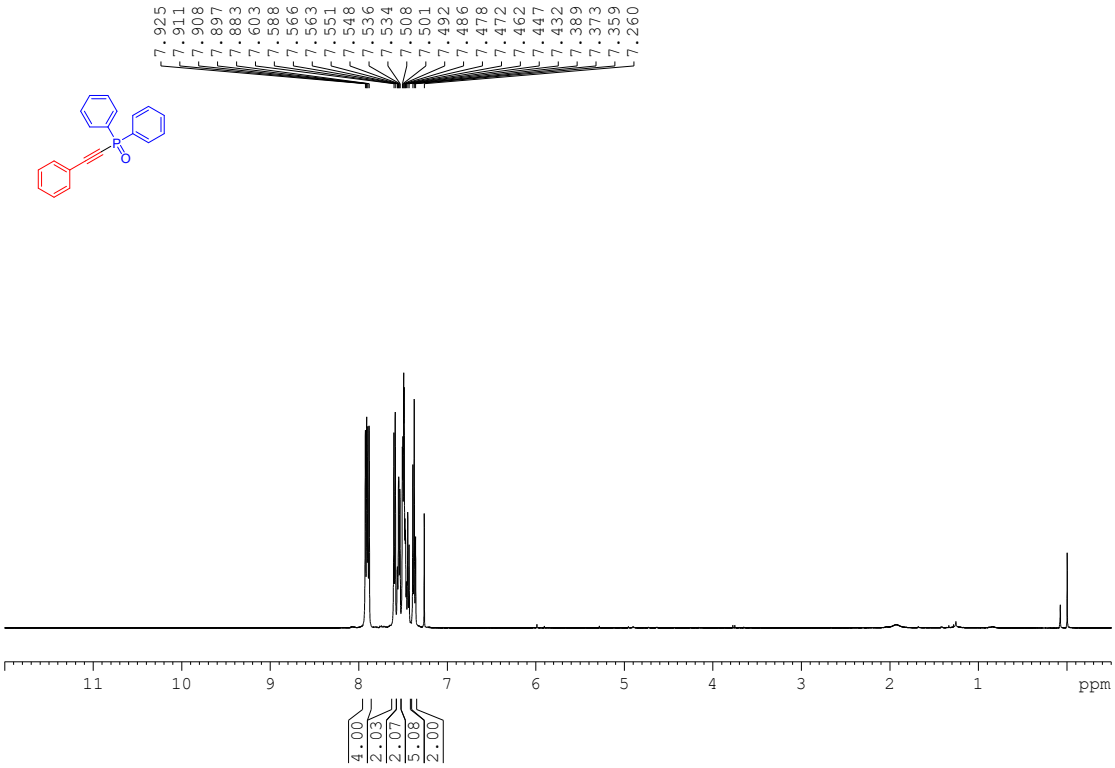
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **3y**



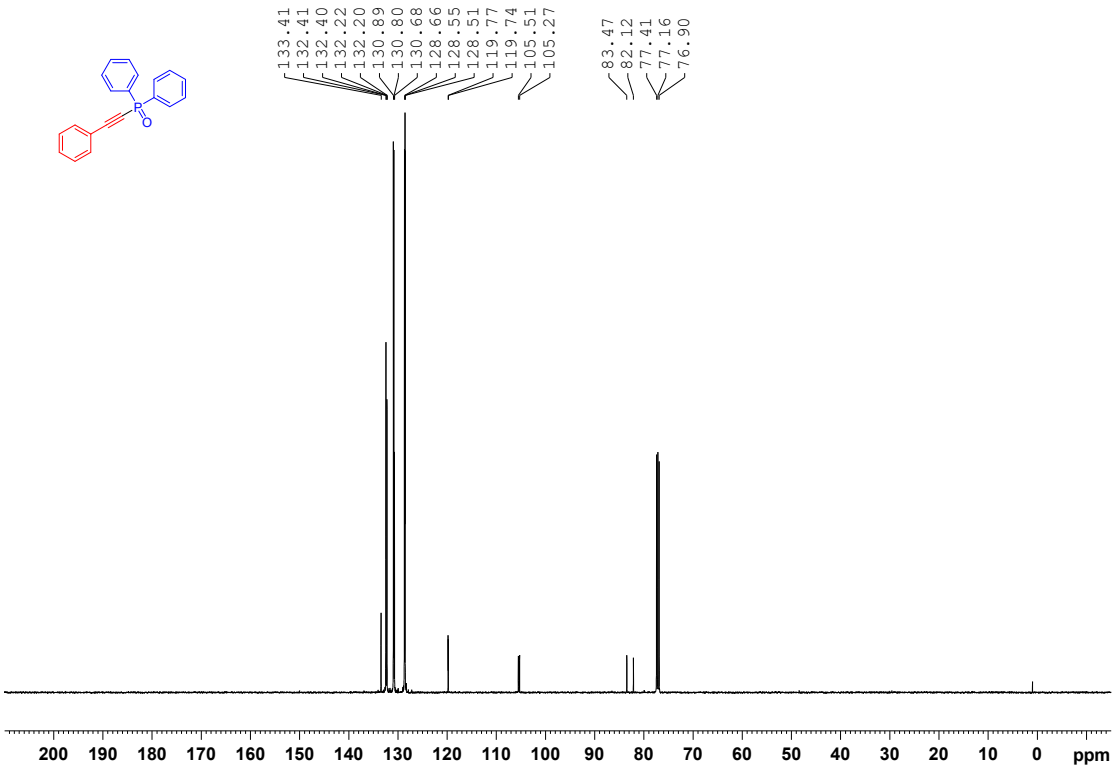
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **3y**



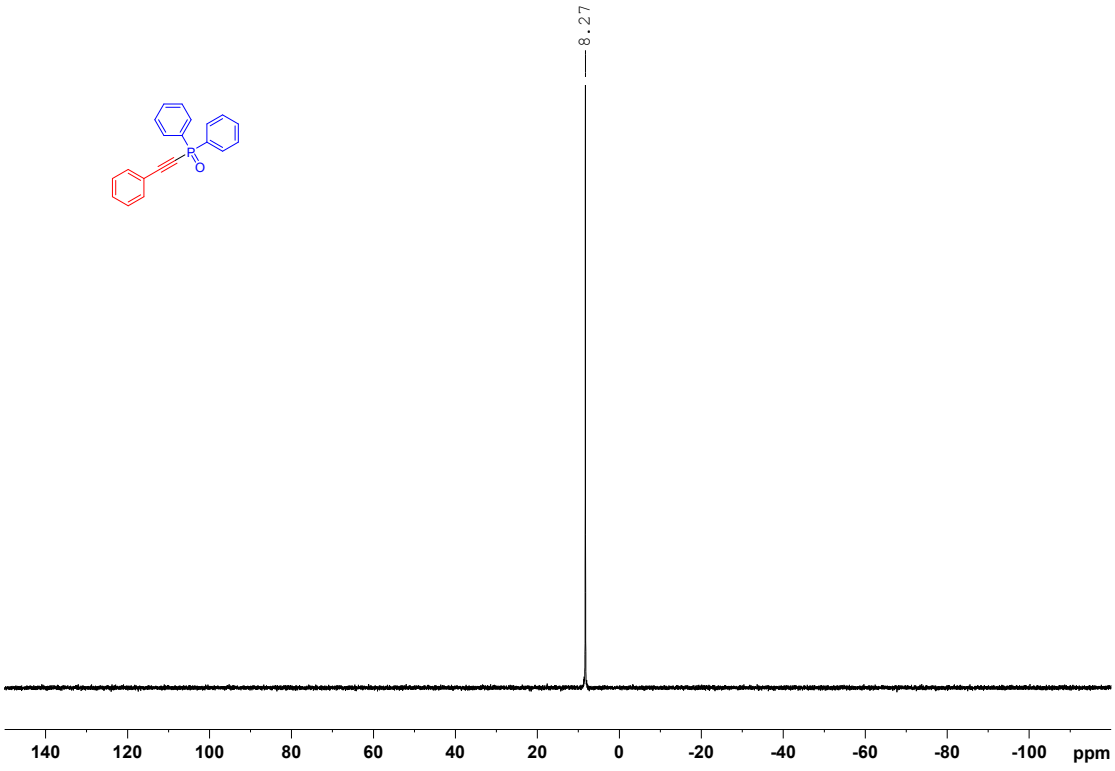
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5a**



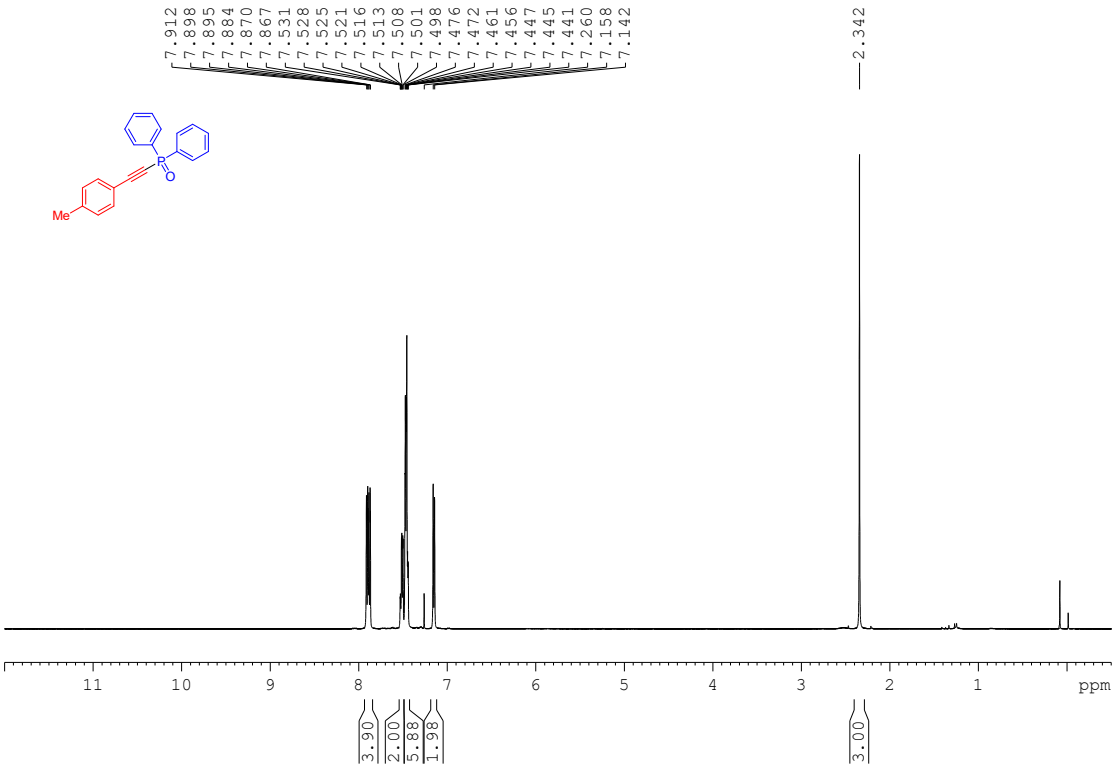
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5a**



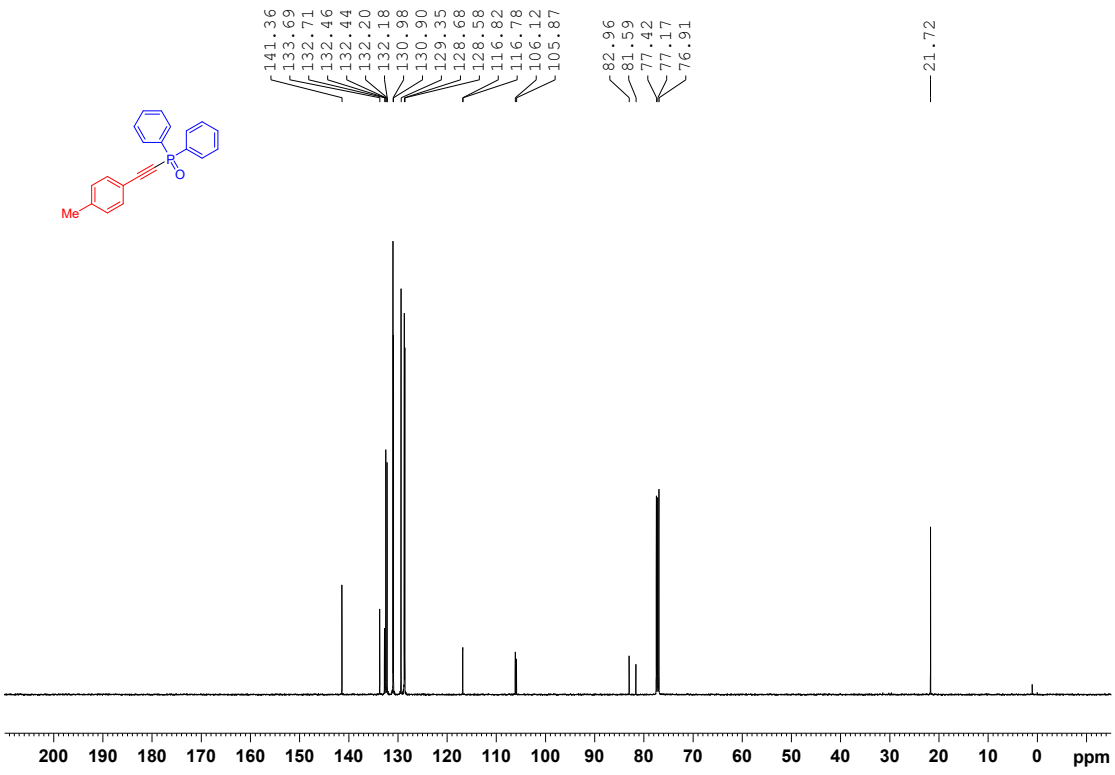
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5a**



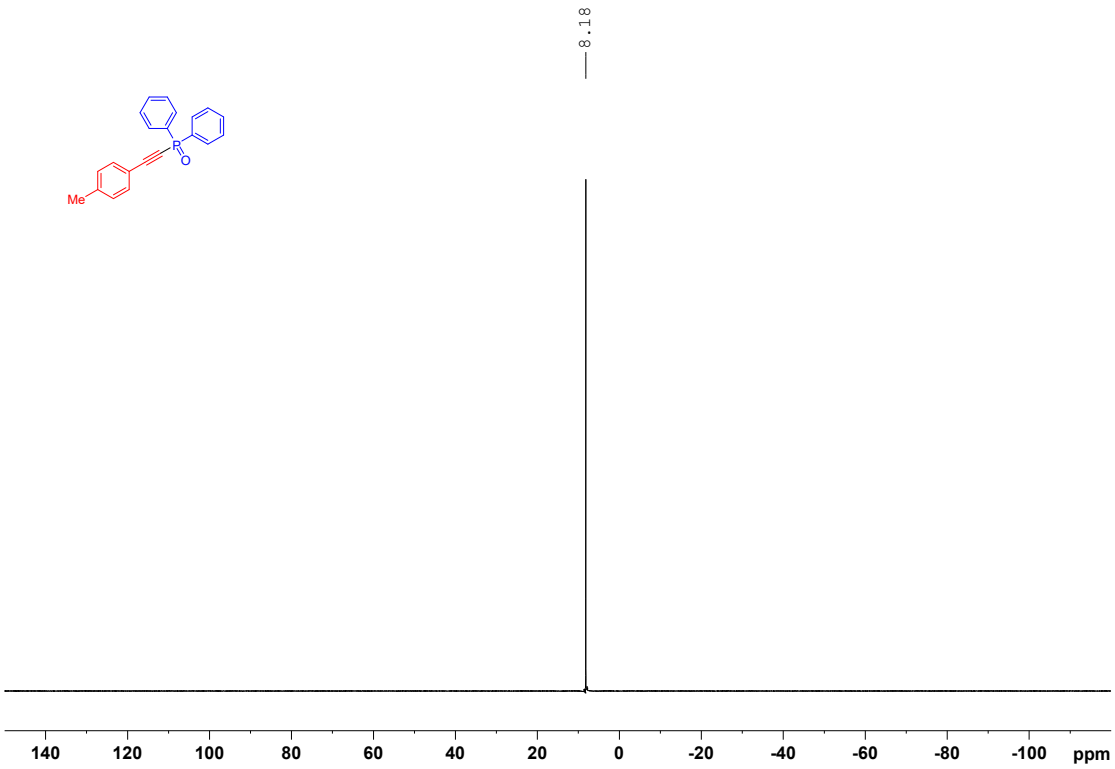
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5b**



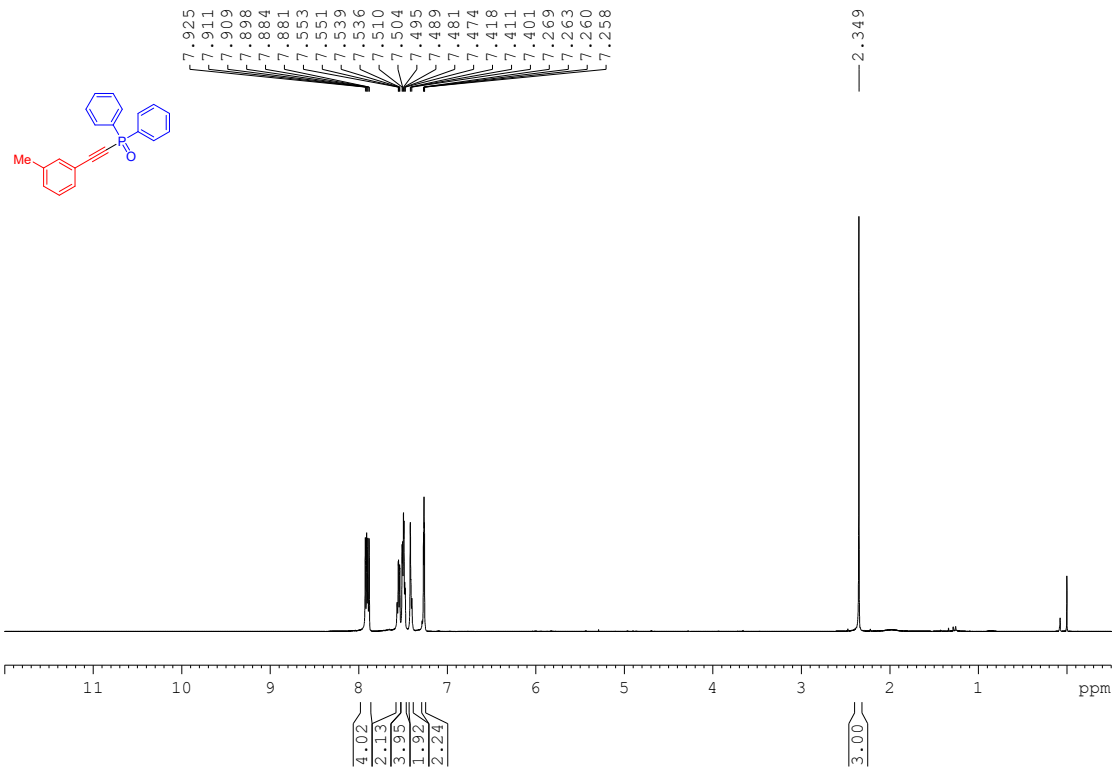
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5b**



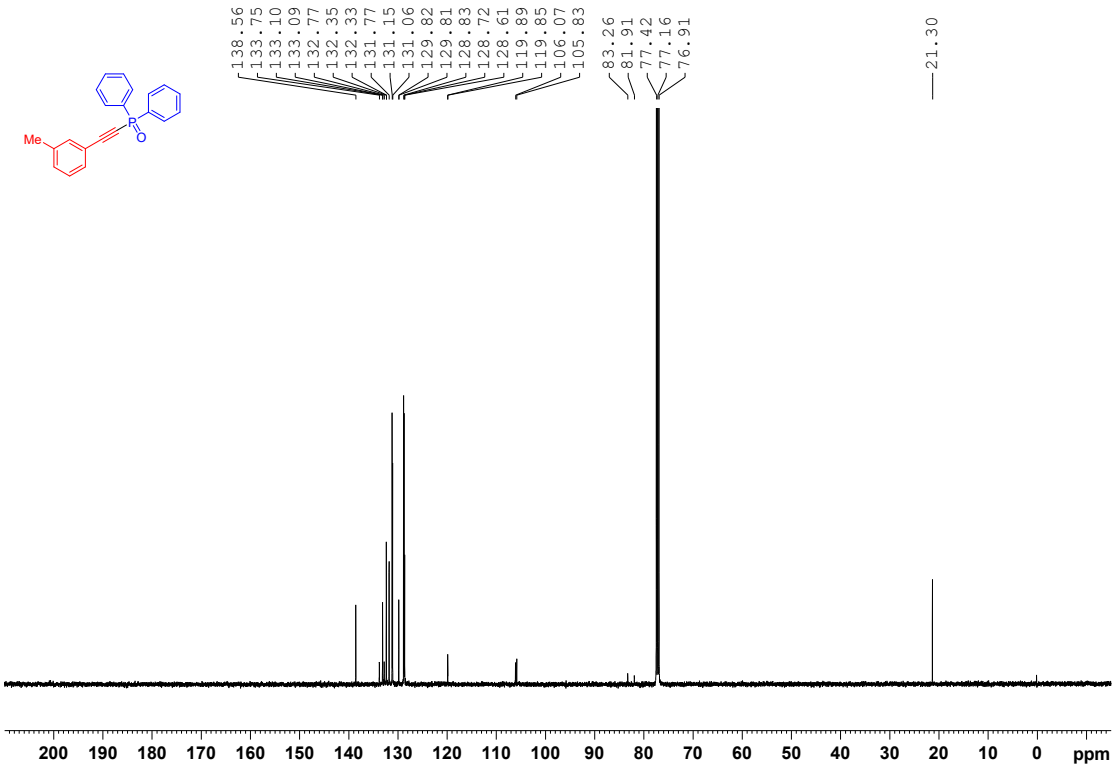
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5b**



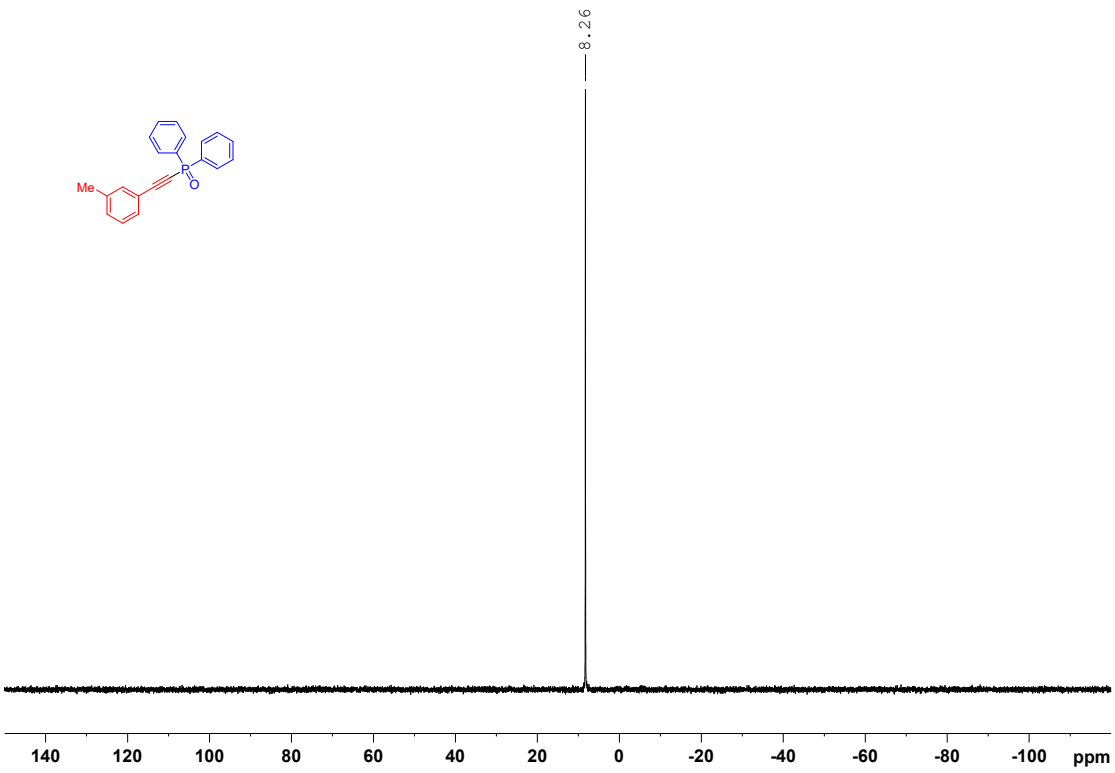
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5c**



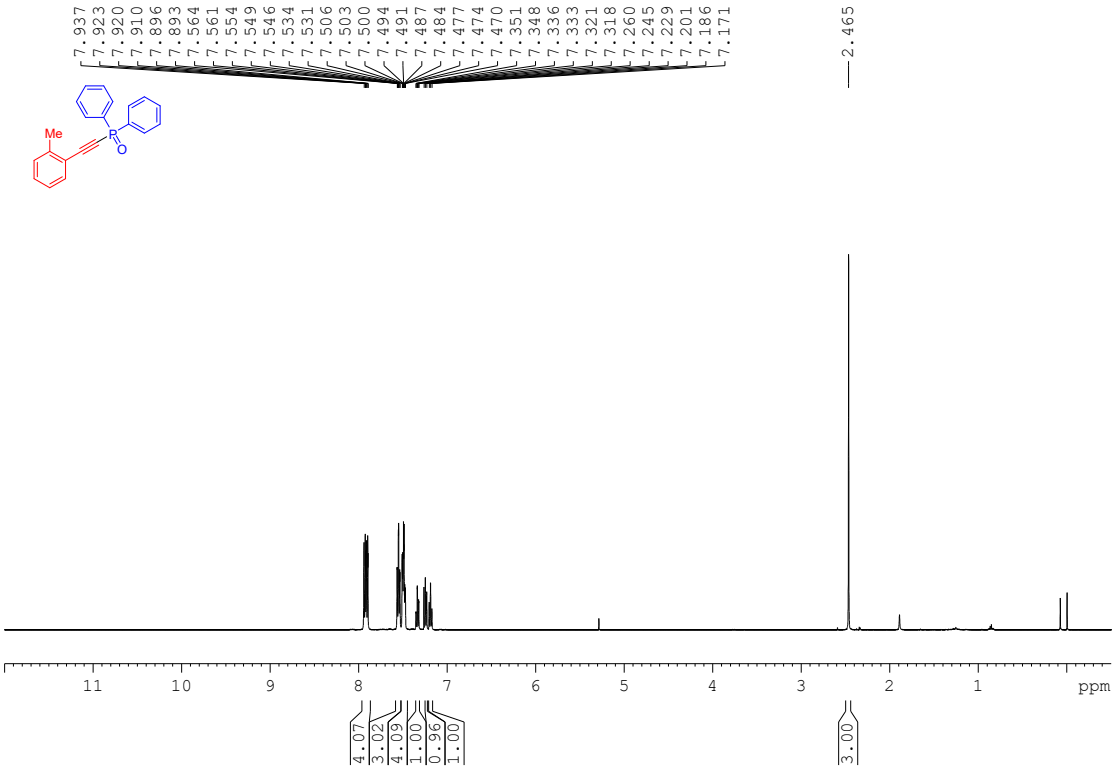
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5c**



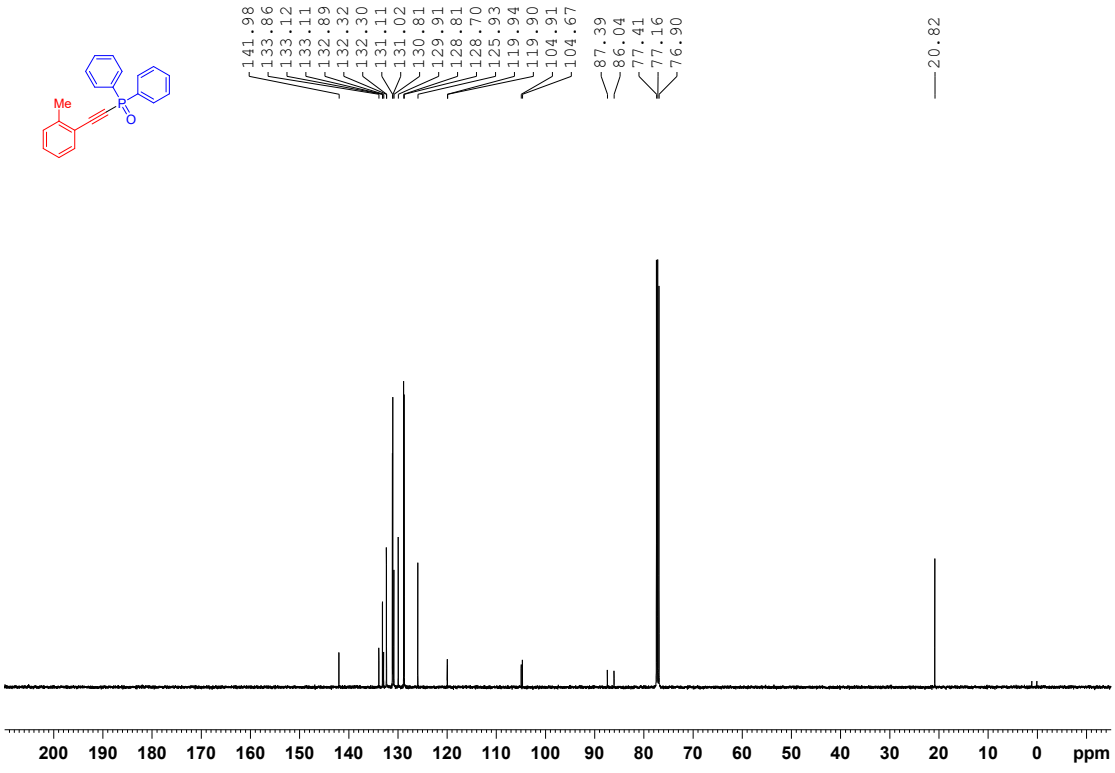
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5c**



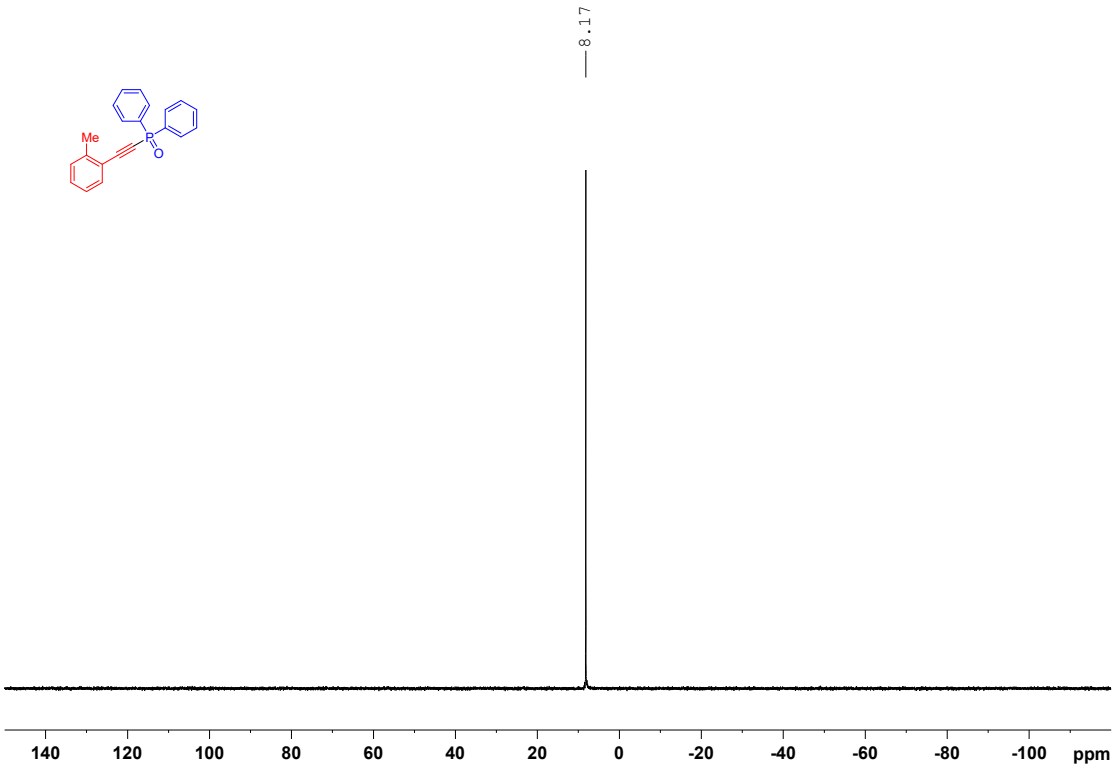
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5d**



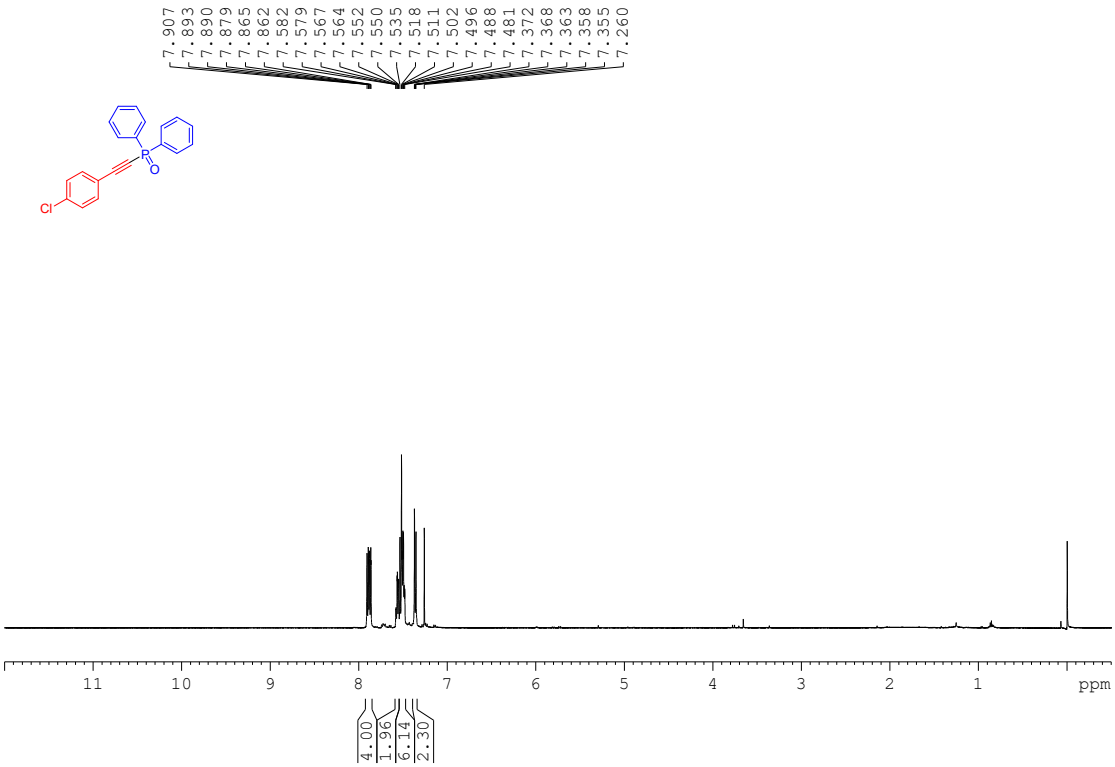
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5d**



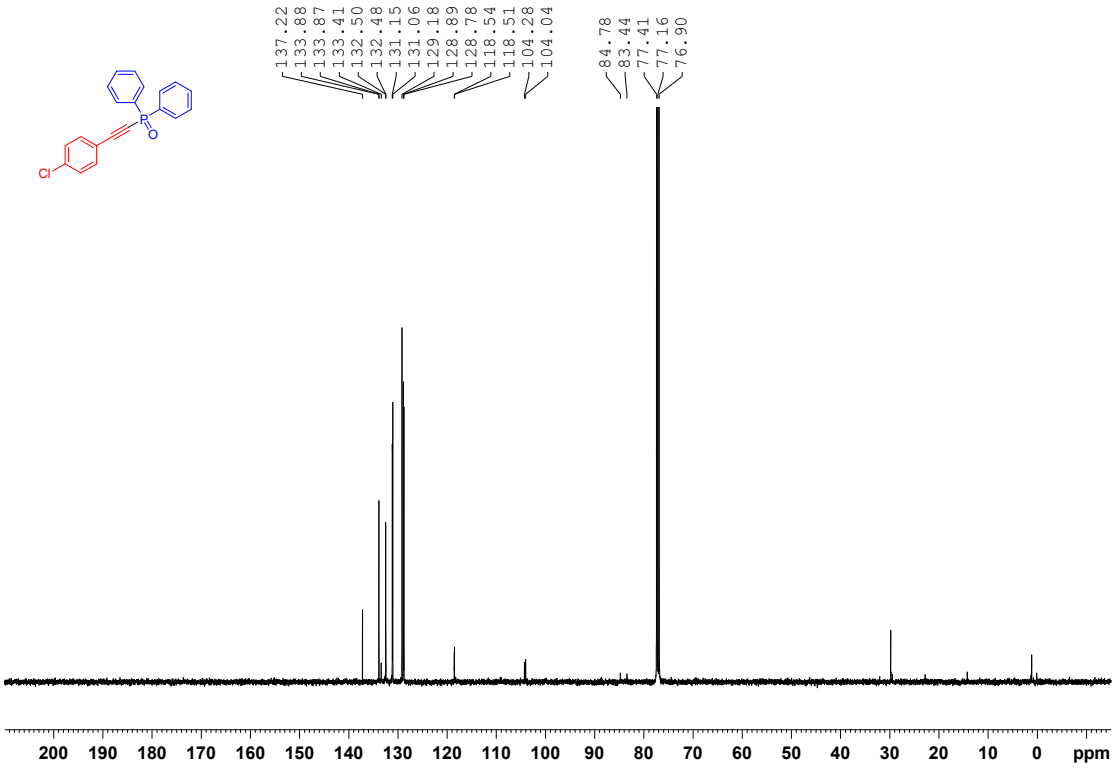
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5d**



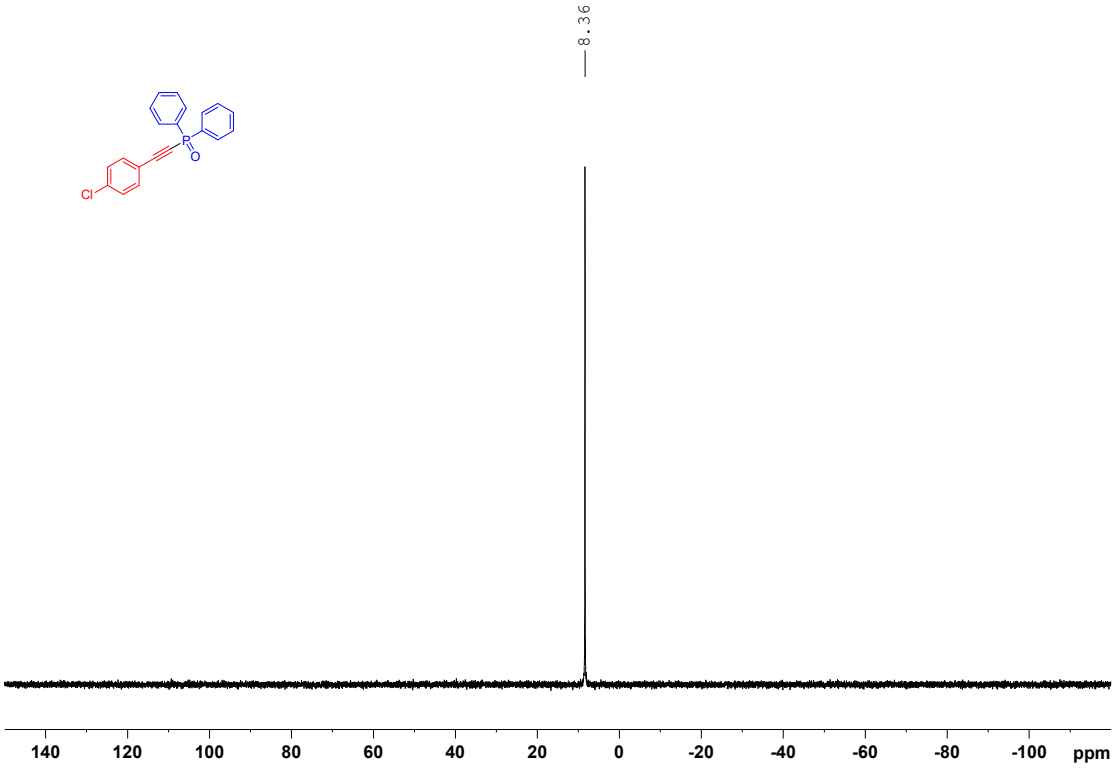
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5e**



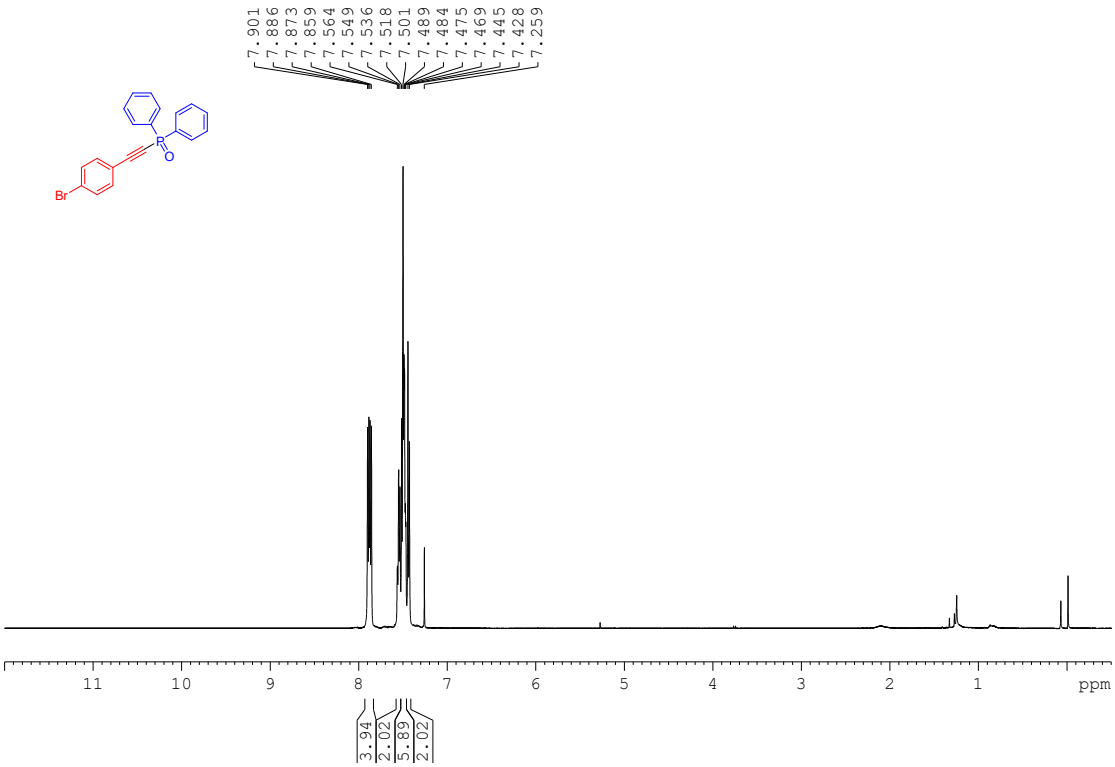
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5e**



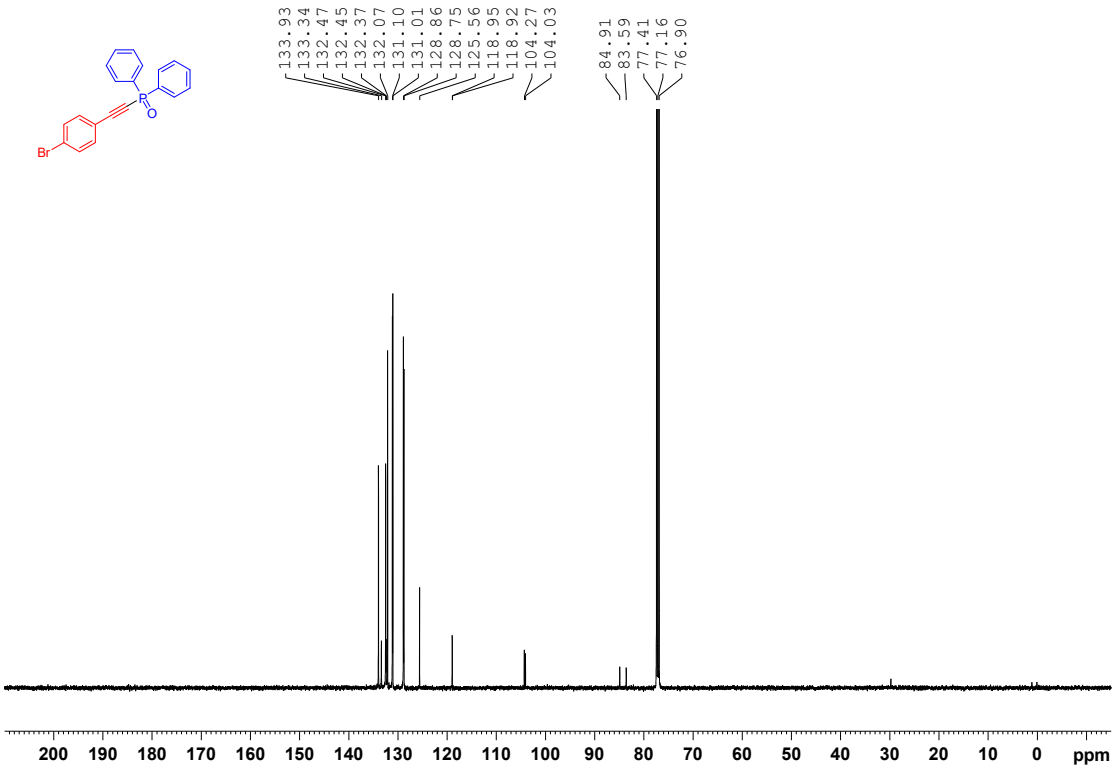
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5e**



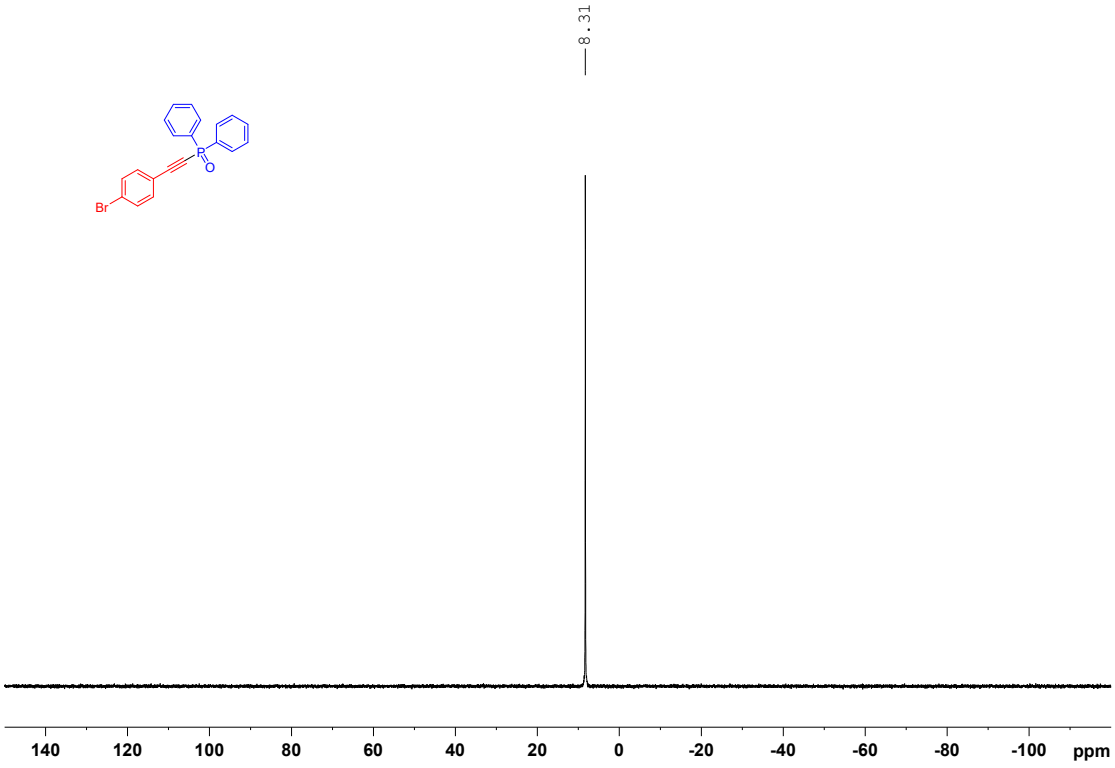
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5f**



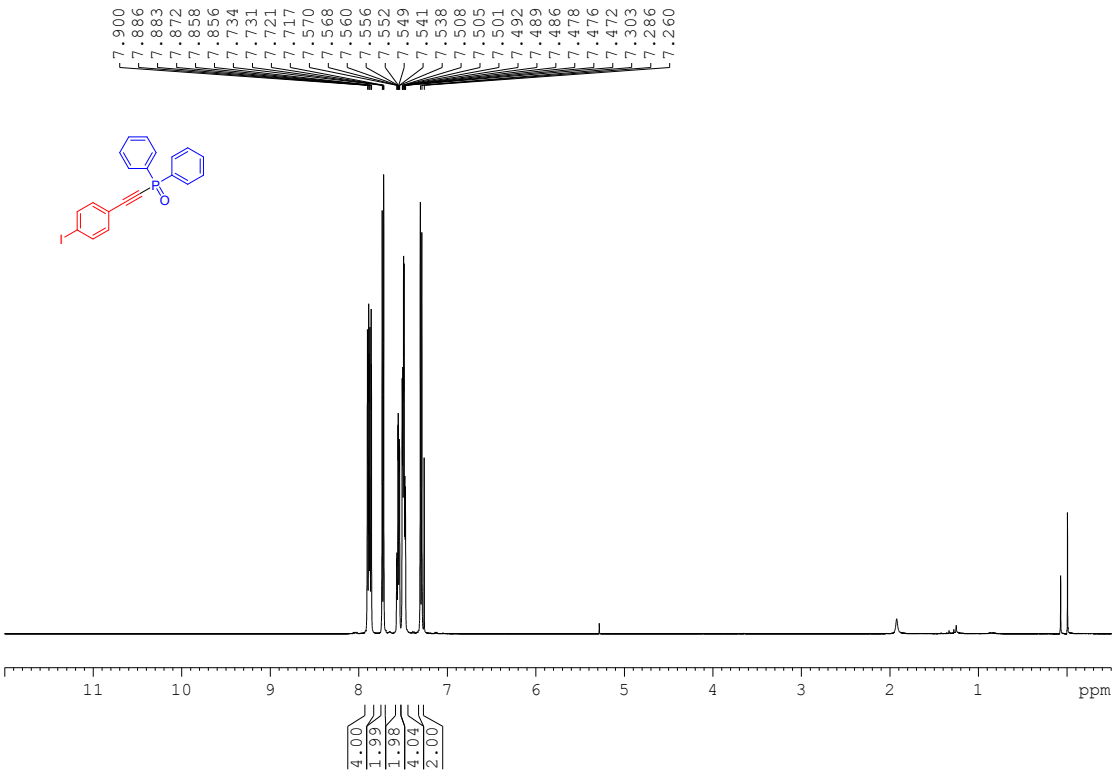
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5f**



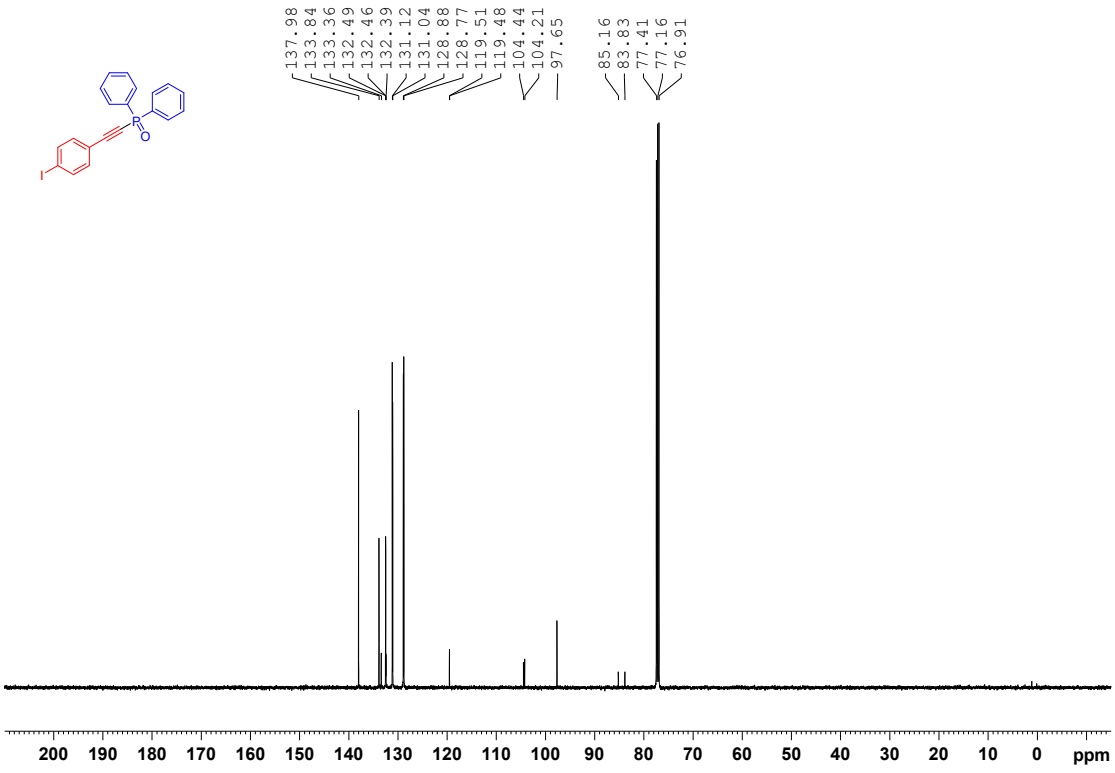
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5f**



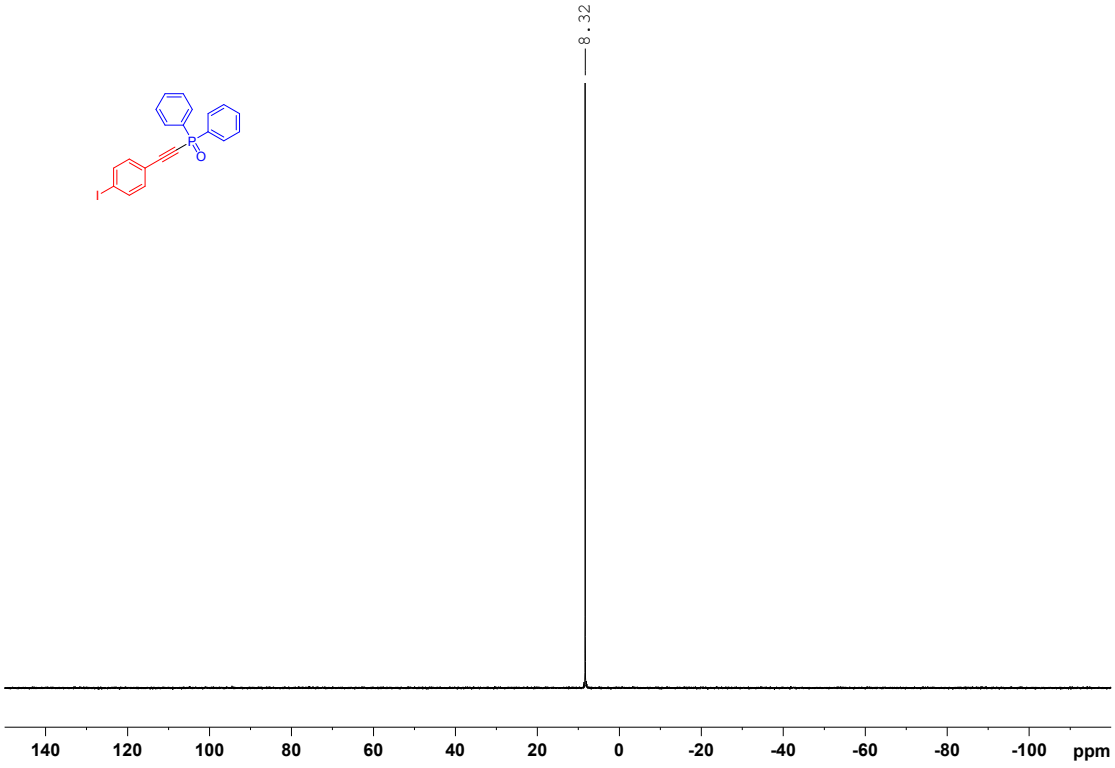
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5g**



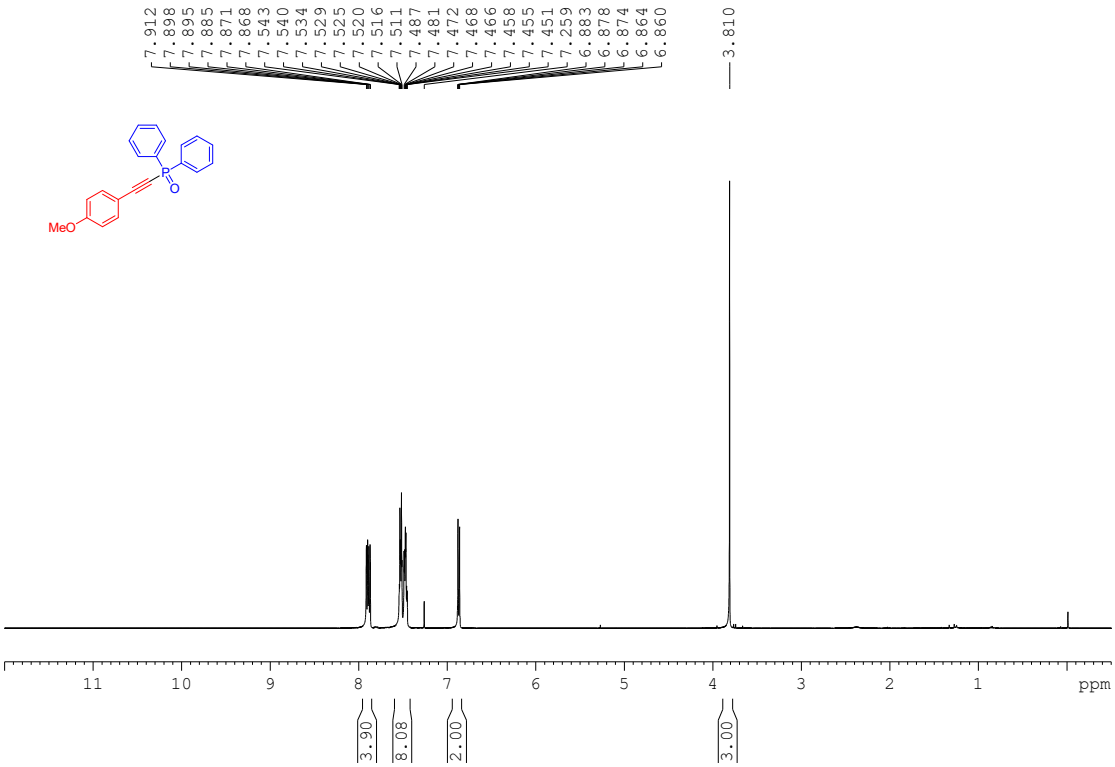
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5g**



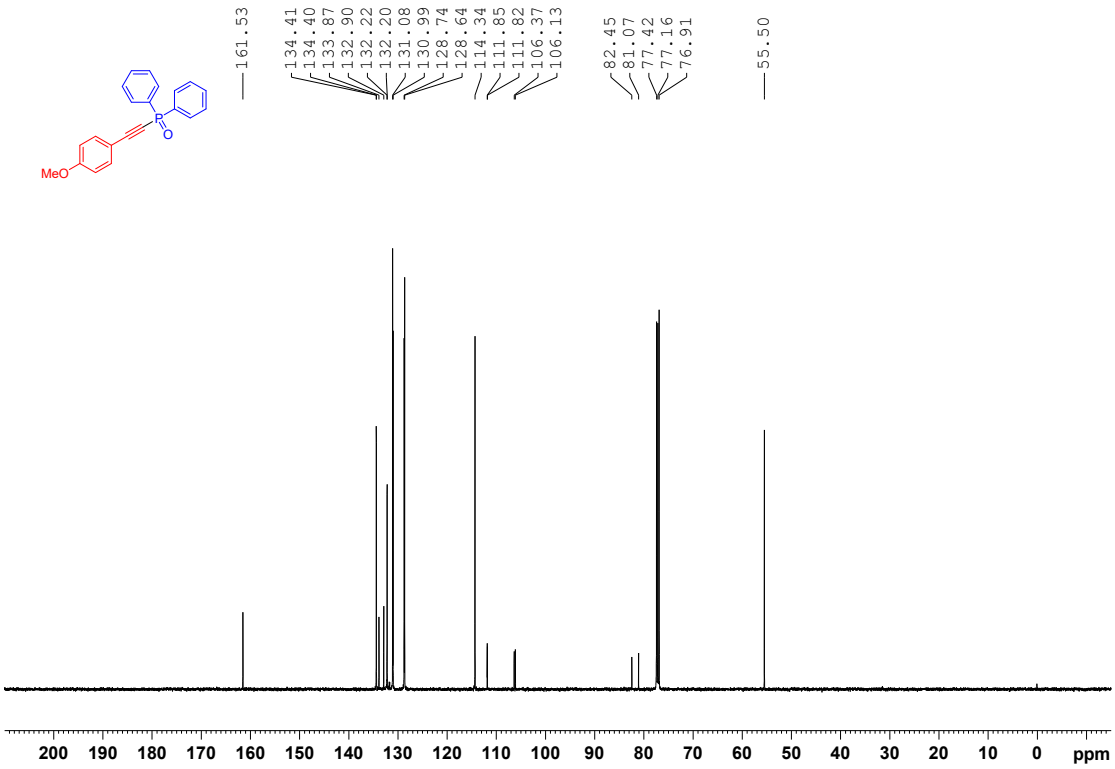
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5g**



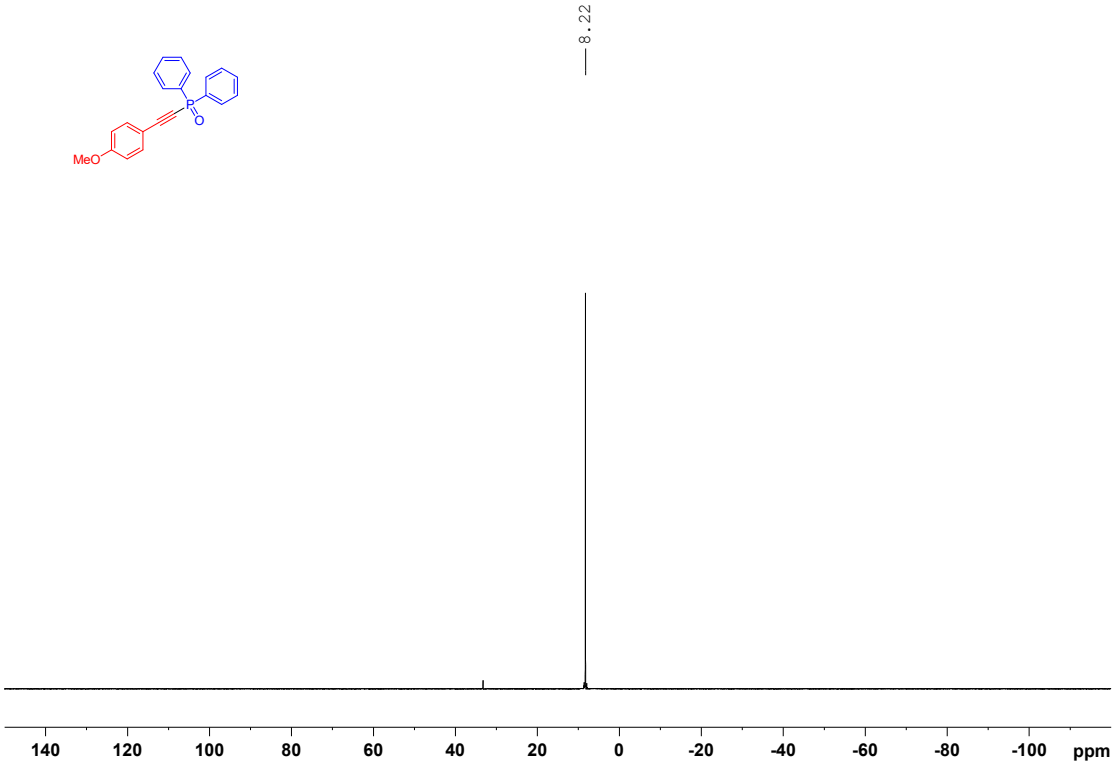
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5h**



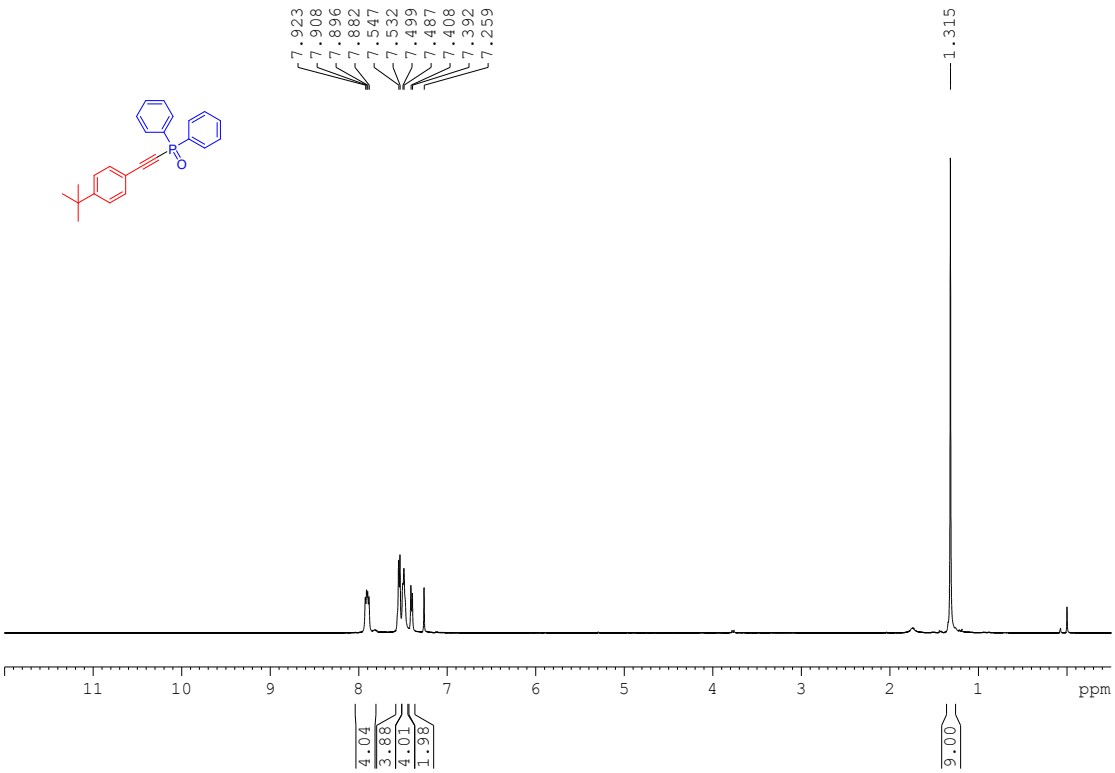
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5h**



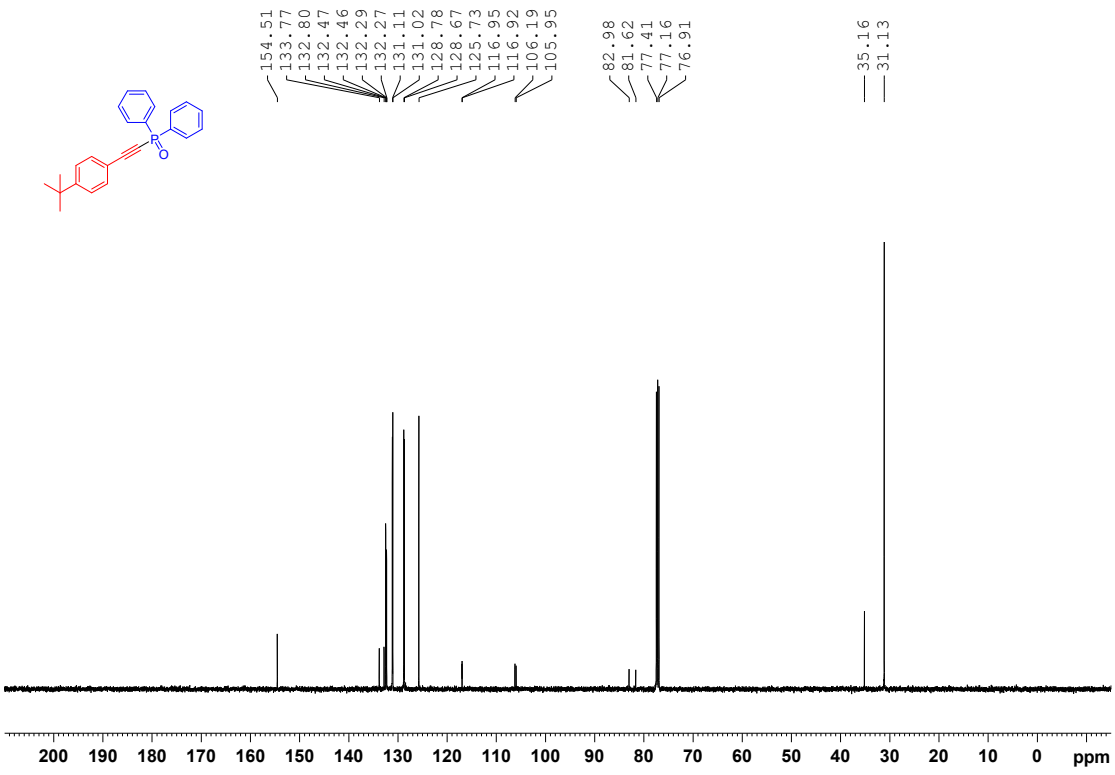
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5h**



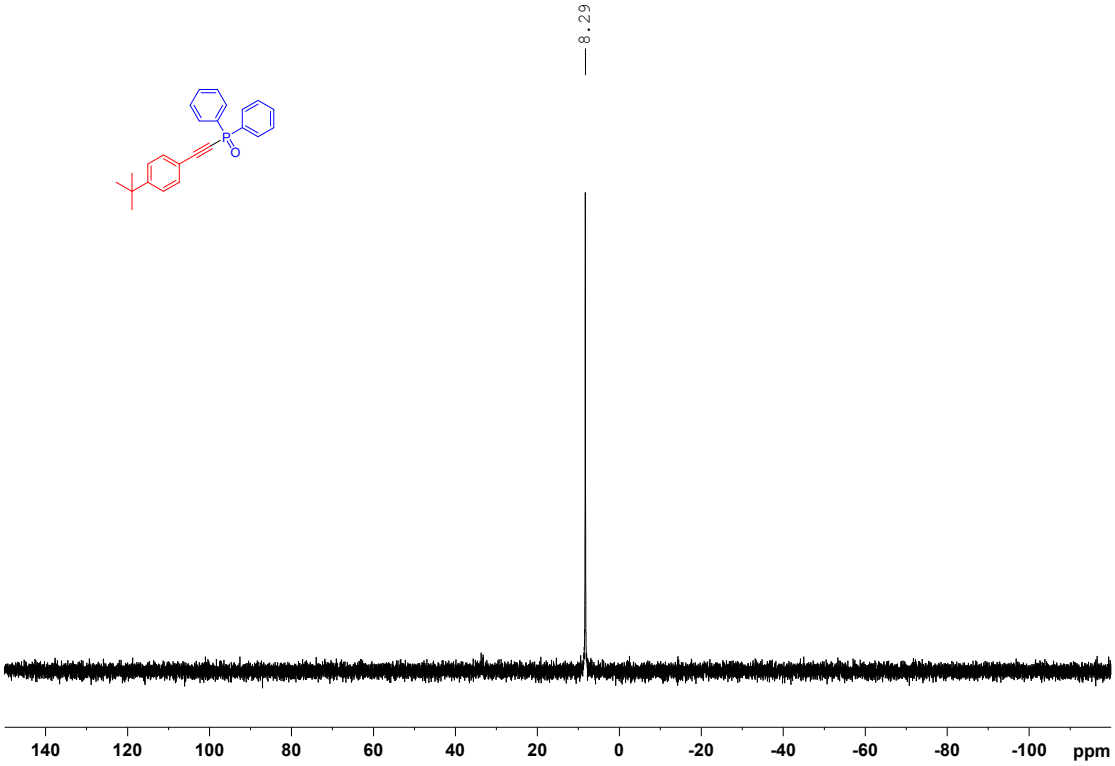
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5i**



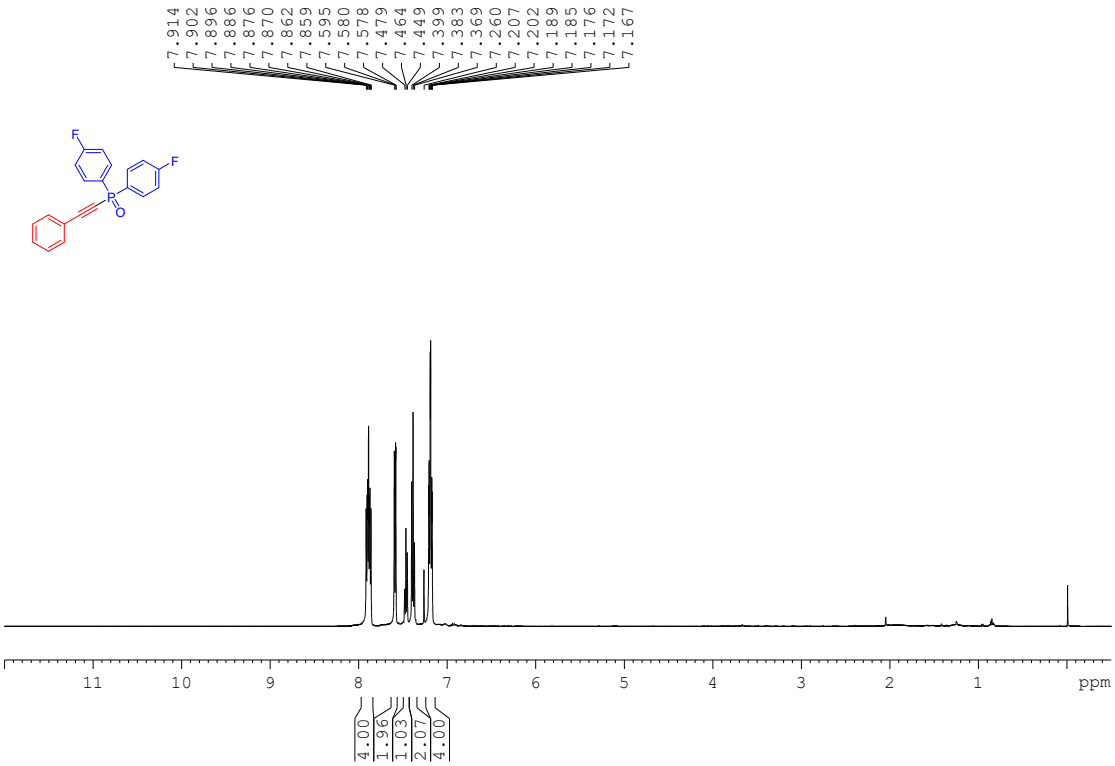
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5i**



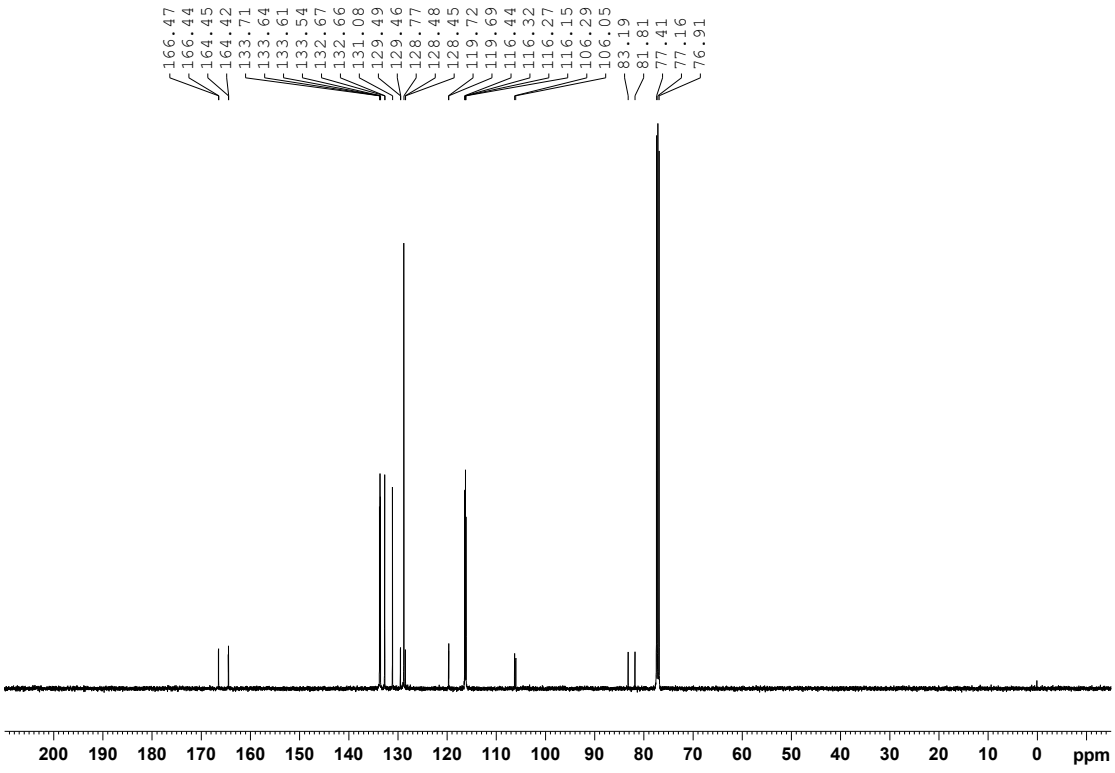
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5i**



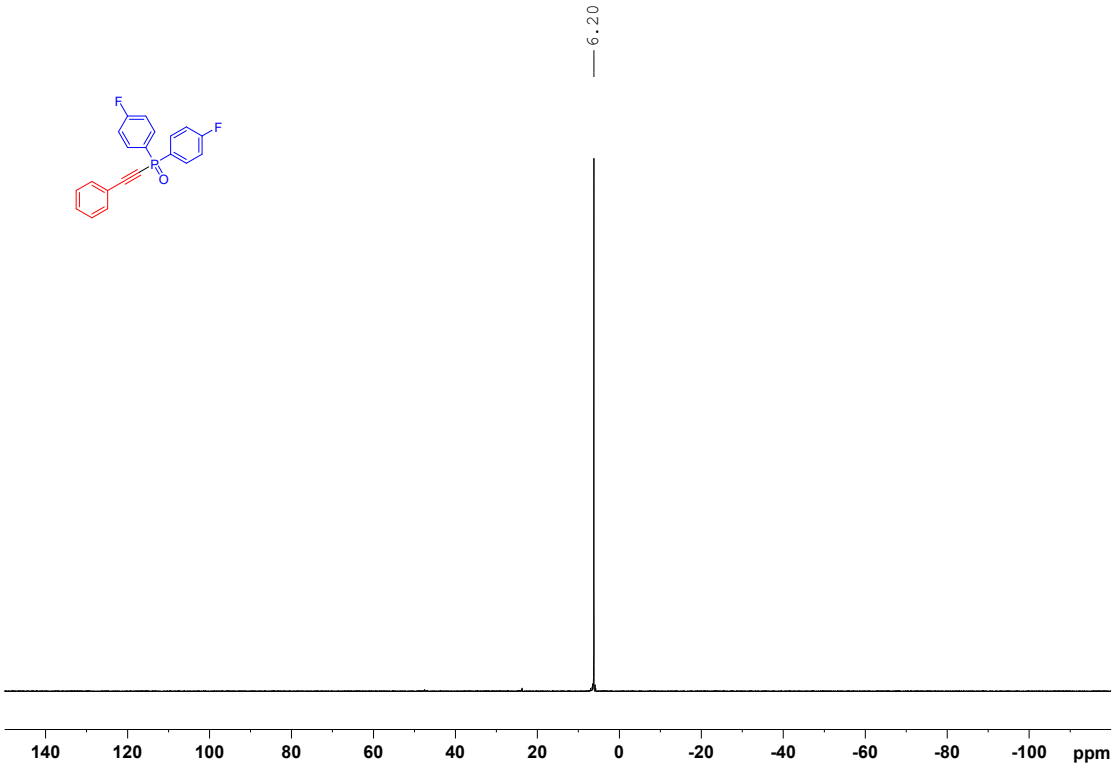
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5j**



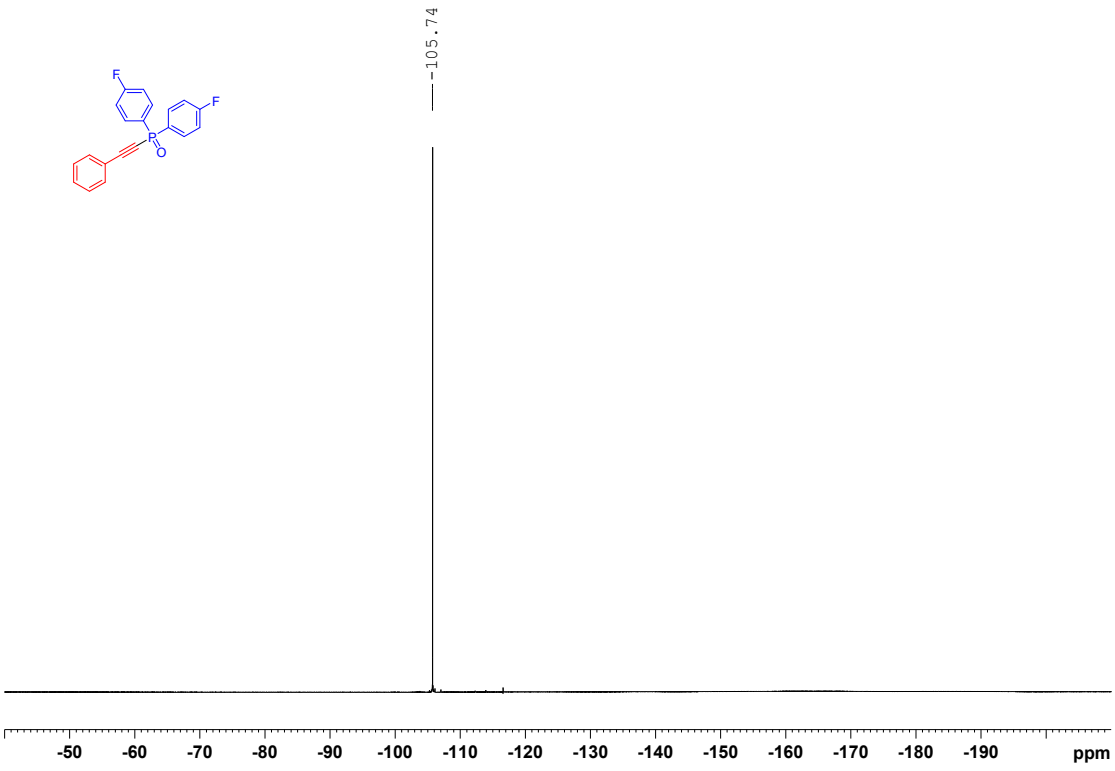
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5j**



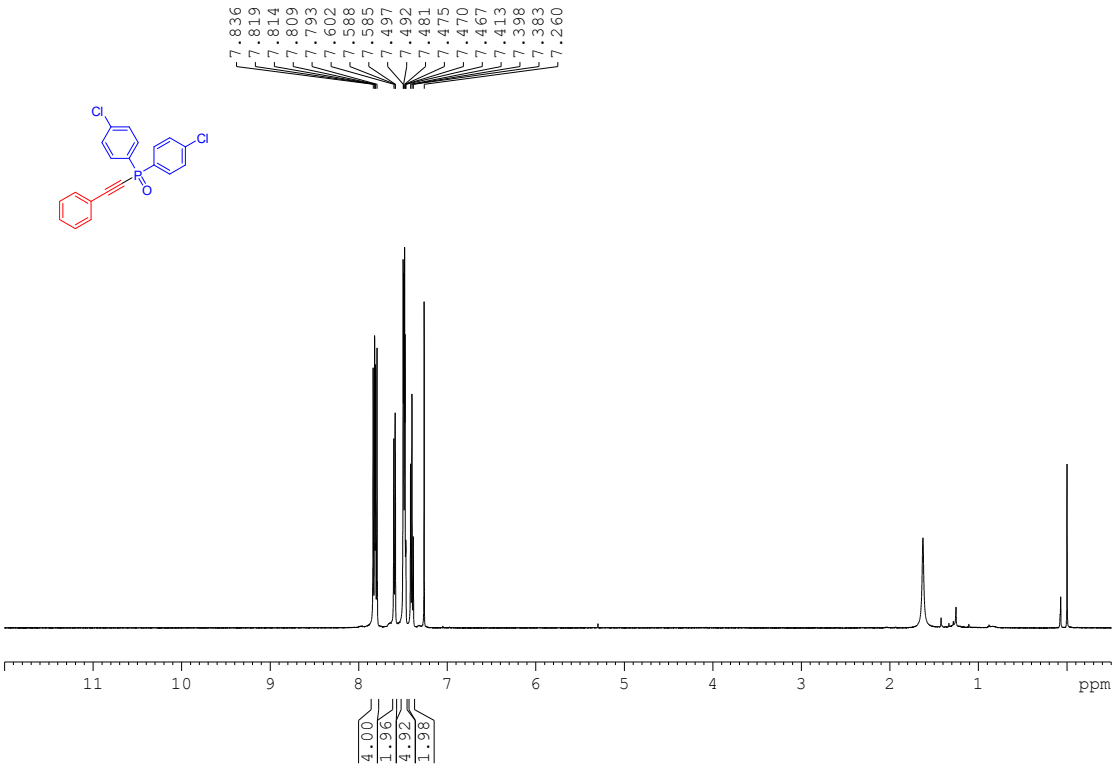
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5j**



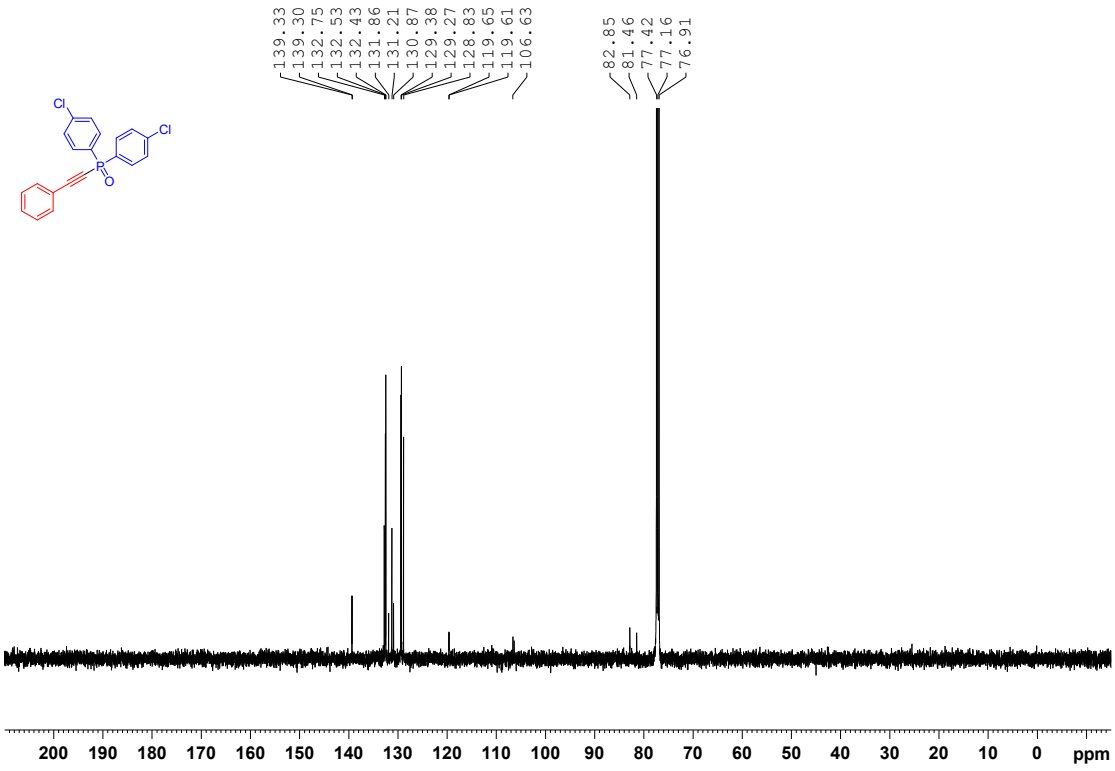
^{19}F NMR (470 MHz, $CDCl_3$, 300K), **5j**



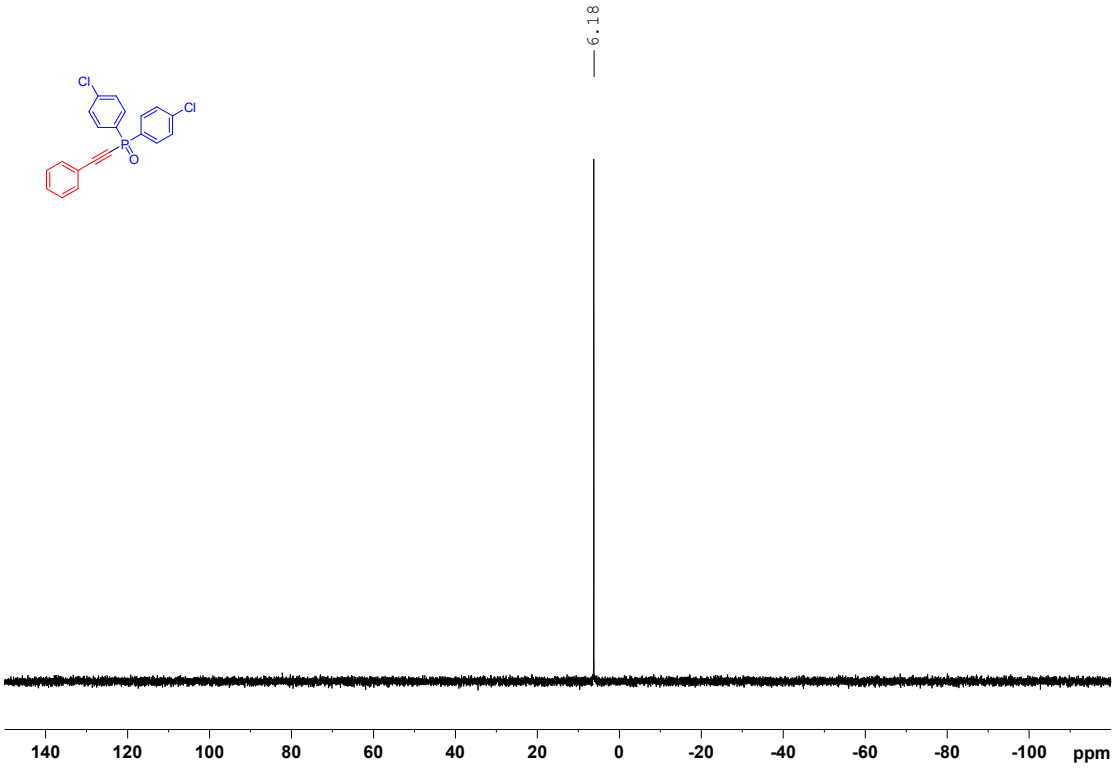
^{1}H NMR (500 MHz, $CDCl_3$, 300K), **5k**



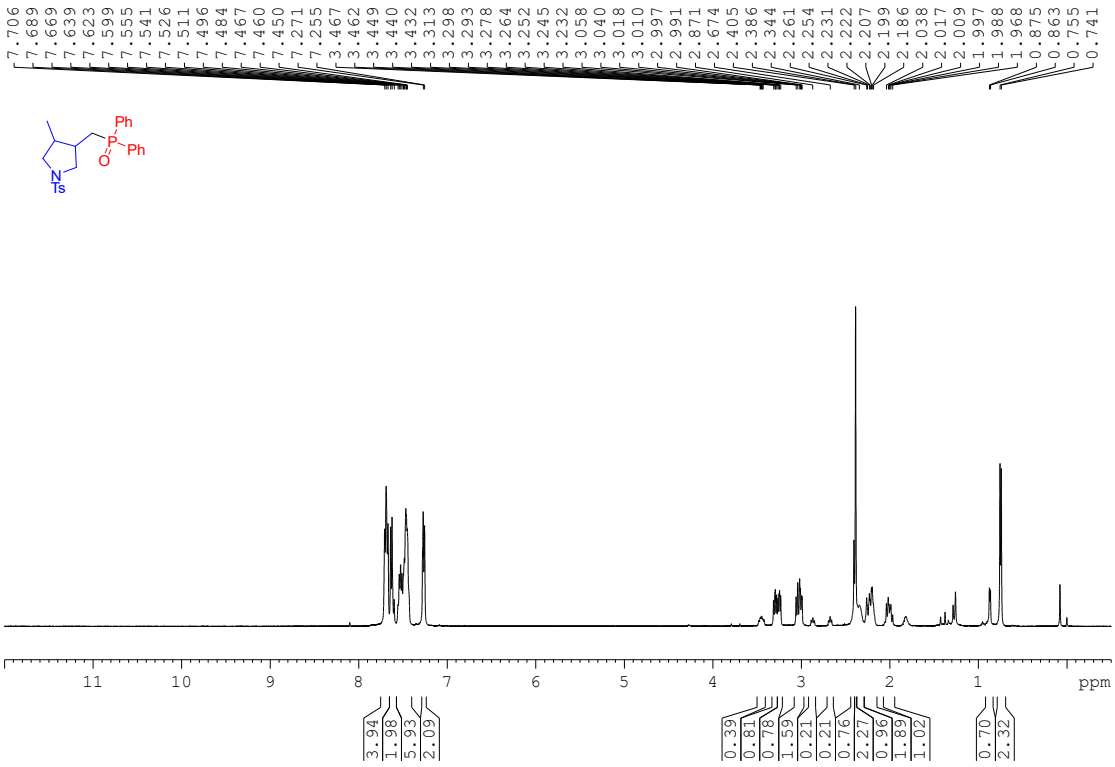
^{13}C NMR (125 MHz, $CDCl_3$, 300K), **5k**



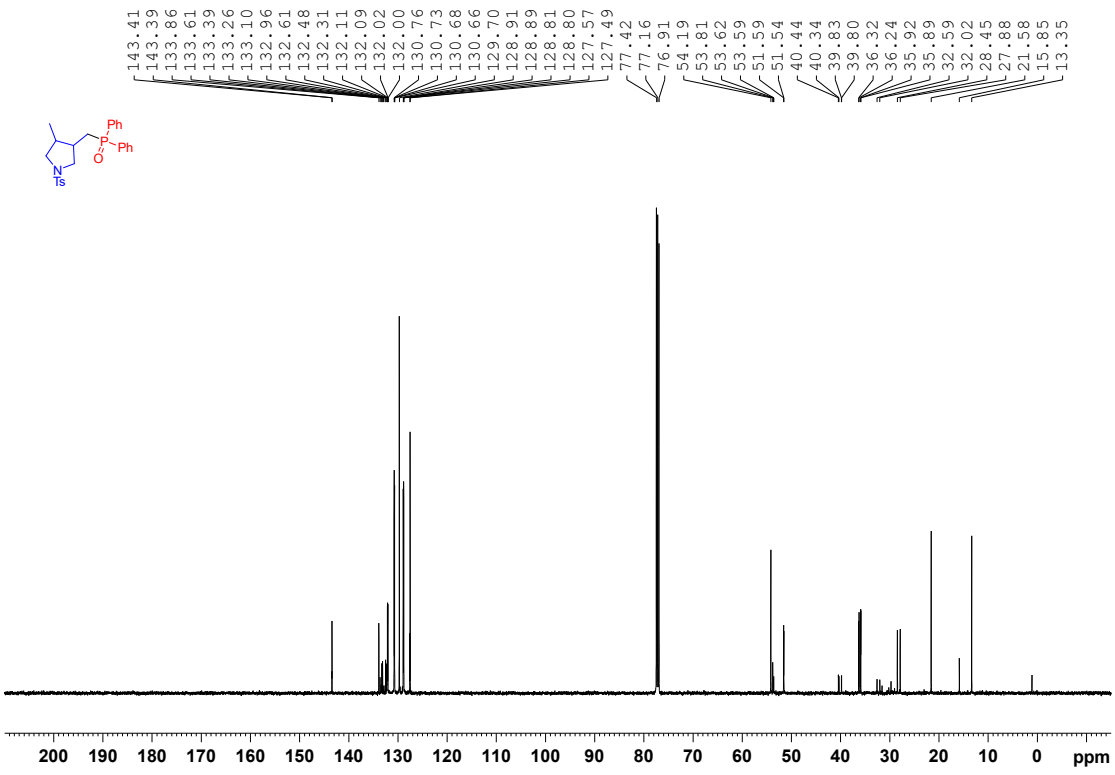
^{31}P NMR (162 MHz, $CDCl_3$, 300K), **5k**



^{1}H NMR (500 MHz, $CDCl_3$, 300K), **9** *(a mixture of diastereoisomers)*



^{13}C NMR (125 MHz, $CDCl_3$, 300K), **9** *(a mixture of diastereoisomers)*



^{31}P NMR (162 MHz, $CDCl_3$, 300K), **9** *(a mixture of diastereoisomers)*