Supporting Information

Base Promoted Regio- and Stereoselective Hydrophosphinylation of Allenes

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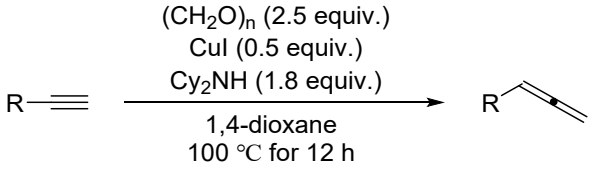
1. General information

The reactions were carried out in 10 mL Schlenk tubes under N_2. Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification. All of the allene substrates are known compounds and were prepared according to the previously reported methods. SPOs were purchased from Bidepharm. Reactions were monitored by thin layer chromatography (TLC) using silicycle precoated silica gel plates. Column chromatography was performed over silica gel (300-400 mesh). NMR spectra were recorded on a ZK-400 spectrometer or a Bruker AV-500 spectrometer, using $CDCl_3$ as the solvent. Chemical shifts are reported in parts per million with the internal TMS signal at 0.0 ppm as a standard, referenced to the residual signal of $CDCl_3$ (7.26 ppm for ^1H, 77.16 ppm for ^{13}C). The following abbreviations were used for the descriptions of splitting patterns: s = singlet, d = doublet, t = triplet, q = quartet and m = multiplet. Mass spectra were recorded with an Agilent 1290-6545XT ultra-high-performance liquid chromatography-quadrupole time-of-flight mass spectrometer using electrospray ionization.

2. Preparation of allenes

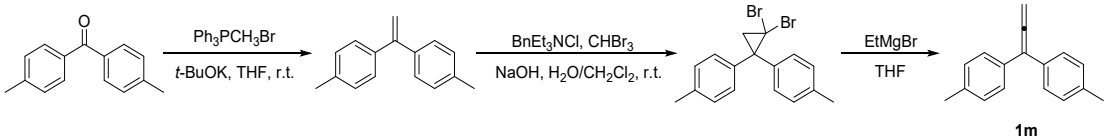
General Procedure A: Synthesis of allenes from alkyne



According to literature procedures,[S1] the corresponding aryl alkyne (10 mmol, 1.0 equiv.), paraformaldehyde (0.75 g, 25 mmol, 2.5 equiv.), CuI (0.95 g, 5.0 mmol, 0.5 equiv.) and dicyclohexylamine (3.6 mL, 18 mmol, 1.8 equiv.) were dissolved in 1,4-dioxane (50 mL) under nitrogen, and the reaction mixture was placed in an oil bath and heated for 12 hours at 100 °C. The reaction was monitored by TLC (pure Hexane). After the disappearance of the starting material, the reaction was quenched with H_2O (20 mL), followed by the addition of Et_2O (20 mL). The organic layer was separated, and the aqueous layer was extracted with Et_2O (3 × 20 mL). The gathered organic layers were dried over $MgSO_4$, and the solvents were evaporated under vacuum. The allene was obtained by purification on silica gel column chromatography.

The substrate **1a-k**,[S1, S2] and **1l**[S3] in Table 2 was prepared according to the procedures described in the literature reported before.

General Procedure B: Synthesis of allene 1m



1m

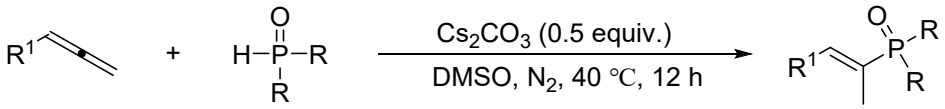
The substrate **1m** in Table 2 was prepared according to the procedures described in the literature reported before.[S4]

Step 1: Methyl triphenylphosphonium bromide (6.43 g, 18 mmol, 1.2 equiv.) was added to a dried flask followed by THF (40 mL). Then *t*-BuOK (22.5 mL, 22.5 mmol, 1.5 equiv.) was added dropwise to the solution under ice-bath and the resulting yellow suspension was stirred at room temperature for 30 min. To this suspension, a solution of 4,4′-Dimethylbenzophenone (15 mmol, 1.0 equiv.) was added and the resulting mixture was further stirred at room temperature overnight. Upon completion, water and DCM were added to the reaction mixture, and the aqueous phase was extracted with DCM. The combined organic phases were washed with brine, dried over anhydrous $MgSO_4$ and the solvent was removed under reduced pressure. The reaction mixture was purified by column chromatography over silica gel using hexanes as eluent to afford 4,4'-(ethene-1,1-diyl)bis(methylbenzene).

Step 2: To a solution of 4,4'-(ethene-1,1-diyl)bis(methylbenzene) (10 mmol, 1.0 equiv.), bromoform (6.33 g, 25 mmol, 2.5 equiv.), $BnNEt_3Cl$ (228 mg, 1 mmol, 10 mol %) and a solution of 50% NaOH (250 mmol, 25 equiv.) were added, and the mixture was stirred at room temperature overnight. Upon completion, water and DCM were added, and the aqueous phase was extracted with DCM. The combined organic phases were washed with brine, dried over anhydrous $MgSO_4$ and the solvent removed under reduced pressure. The reaction mixture was purified by column chromatography to afford 1,1′-(2,2-Dibromocyclopropylidene)bis[4-methylbenzene].

Step 3: To a pre-cooled (ice-bath) solution of 1,1′-(2,2-Dibromocyclopropylidene)bis[4-methylbenzene] (1.0 equiv.) in dry THF (1.0 mL/mmol), EtMgBr (3.0 M in THF, 1.5 equiv.) was added dropwise. The mixture was then slowly warmed to room temperature and stirred at room temperature for an additional 3 hours. Then the reaction was quenched by water and the mixture was extracted with DCM. The combined organic layers were washed with brine, and dried with anhydrous $MgSO_4$. After removing the solvent under reduced pressure, the crude product was purified by column chromatography on silica gel to afford allene **1m**.

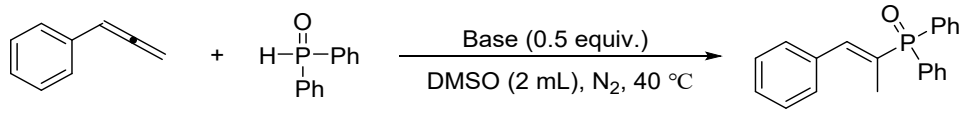
3. General procedure for the hydrophosphinylation of allenes



To an oven dried Schlenk tube (10 mL) with a magnetic stir bar, allenes (0.4 mmol, 2 equiv., if solid), phosphine oxides (0.2 mmol, 1 equiv.) and Cs_2CO_3 (0.1 mmol, 0.5 equiv.) were added. The Schlenk tube was sealed and degassed via vacuum evacuation and subsequent backfilled with N_2 for three times. Subsequently, allenes (0.4 mmol, if liquid) and anhydrous DMSO (2 mL) were added. Then the reaction mixture was placed in an oil bath and heated for 12 hours at 40 °C. Upon completion, water and EtOAc were added to the reaction mixture, and the aqueous phase was extracted with EtOAc. The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by column chromatography isolation on silica gel (petroleum ether: ethyl acetate = 2:1 *v*/*v*) to give the pure desired product.

4. Optimization of reaction conditions

Table S1. Base optimization[a]

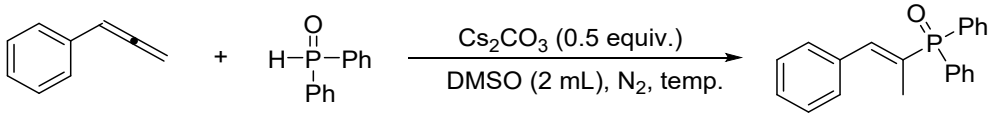


1a **2a** **3a**

Entry	Base	Yield[b] (%)
1	*t*-BuOK	9
2	*t*-BuONa	30
3	*t*-BuOLi	16
4	KOH	61
5	NaOH	55
6	Cs_2CO_3	81
7	Rb_2CO_3	64
8	K_2CO_3	20
9	Na_2CO_3	N.D.
10	CsF	26
11	DBU	8
12[c]	Cs_2CO_3	20
13	/	N.D.
14[d]	Cs_2CO_3	82
15[e]	Cs_2CO_3	56

[a]Reaction condition: phenyl allene **1a** (0.40 mmol), SPO **2a** (0.20 mmol), base (0.10 mmol, 0.5 equiv.), DMSO (2 mL), 40 °C, 12 h, N_2 atmosphere. [b]Yield was determined by ^{1}H NMR analysis using 1,3,5-trimethoxy-benzene as an internal standard. [c]Phenyl allene **1a** (0.20 mmol), SPO **2a** (0.40 mmol). [d]Base (1.0 equiv.). [e]Base (0.3 equiv.). N.D.: not detected.

Table S2. Reaction temperature optimization[a]

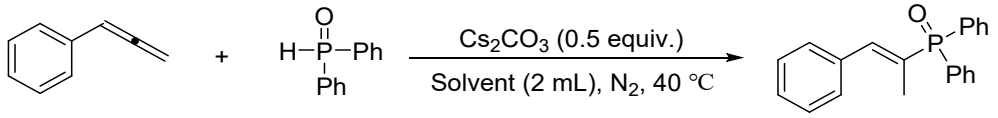


1a **2a** **3a**

Entry	Temperature	Yield[b] (%)
1	40 °C	81
2	60 °C	66
3	50 °C	73
4	r.t.	72

[a]Reaction condition: phenyl allene **1a** (0.40 mmol), SPO **2a** (0.20 mmol), Cs_2CO_3 (0.10 mmol, 0.5 equiv.), DMSO (2 mL), 12 h, N_2 atmosphere. [b]Yield was determined by ^{1}H NMR analysis using 1,3,5-trimethoxy-benzene as an internal standard.

Table S3. Solvent optimization[a]



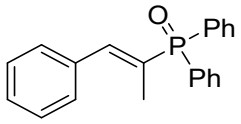
1a **2a** **3a**

Entry	Solvent	Yield[b] (%)
1	DMF	74
2	DMSO	81
3	DMA	65
4	THF	30
5	DCM	N.D.
6	MeCN	35
7	Toluene	trace
8	1,4-Dioxane	8

[a]Reaction condition: phenyl allene **1a** (0.40 mmol), SPO **2a** (0.20 mmol), Cs_2CO_3 (0.10 mmol, 0.5 equiv.), solvent (2 mL), 12 h, N_2 atmosphere, 40 ℃. [b]Yield was determined by ^{1}H NMR analysis using 1,3,5-trimethoxy-benzene as an internal standard. N.D.: not detected.

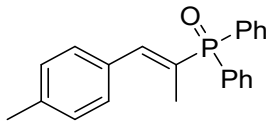
5. Analytical data for compounds

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



Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as white solid (48mg, 75%, mp 129-130 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.72 (m, 4H), 7.56-7.43 (m, 6H), 7.37-7.26 (m, 5H), 7.22 (d, *J* = 21.9, 1H), 2.10 (d, *J* = 13.8, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.7 (d, *J* = 10.8 Hz), 135.8 (d, *J* = 18.8 Hz), 132.1 (d, *J* = 9.6 Hz), 131.9 (d, *J* = 2.9 Hz), 131.3 (d, *J* = 102.1 Hz), 130.3 (d, *J* = 96.4 Hz), 129.4, 128.6 (d, *J* = 11.9 Hz), 128.5, 128.4, 15.1 (d, *J* = 10.8 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.39; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{20}OP]^+$ 319.1246, found 319.1243.

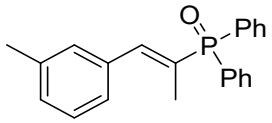
(*E*)-diphenyl(1-(p-tolyl)prop-1-en-2-yl)phosphine oxide (**3b**)



Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (45mg, 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.73 (m, 4H), 7.57-7.53 (m, 2H), 7.51-7.45 (m, 4H), 7.31-7.28 (m, 2H), 7.24-7.16 (m, 3H), 2.36 (s, 3H), 2.13 (d, *J* = 13.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.7 (d, *J* = 10.9 Hz), 138.5, 132.9 (d, *J* = 19.2 Hz), 132.0 (d, *J* = 9.6 Hz), 131.9 (d, *J* = 2.8 Hz), 131.3 (d, *J* = 102.3 Hz), 129.5, 129.1, 129.0 (d, *J* = 96.8 Hz), 128.6 (d, *J* = 11.9 Hz), 21.3, 15.1 (d, *J* = 10.8

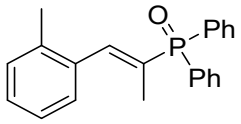
Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.70; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{22}H_{22}OP]^+$ 333.1403, found 333.1407.

(*E*)-diphenyl(1-(m-tolyl)prop-1-en-2-yl)phosphine oxide (**3c**)



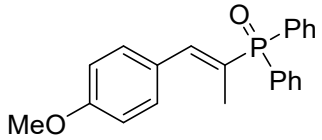
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (38 mg, 58%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78-7.73 (m, 4H), 7.57-7.54 (m, 2H), 7.51-7.47 (m, 4H), 7.29-7.24 (m, 1H), 7.20-7.11 (m, 4H), 2.35 (s, 3H), 2.11 (d, J = 13.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9 (d, J = 10.9 Hz), 138.1, 135.8 (d, J = 19.0 Hz), 132.1 (d, J = 9.8 Hz), 132.0 (d, J = 2.7 Hz), 131.3 (d, J = 102.2 Hz), 130.2, 130.0 (d, J = 97.3 Hz), 129.2, 128.6 (d, J = 11.8 Hz), 128.3, 126.5, 21.5, 15.1 (d, J = 10.9 Hz); ^{31}P NMR (202 MHz, $CDCl_3$) δ 33.58; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{22}H_{22}OP]^+$ 333.1403, found 333.1408.

(*E*)-diphenyl(1-(o-tolyl)prop-1-en-2-yl)phosphine oxide (**3d**)



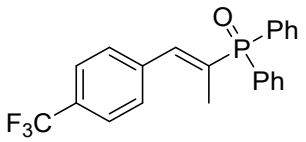
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (39 mg, 59%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81-7.73 (m, 4H), 7.59-7.47 (m, 6H), 7.25-7.17 (m, 4H), 7.13 (d, J = 22.2 Hz, 1H), 2.17 (s, 3H), 1.98 (d, J = 15.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.6 (d, J = 11.5 Hz), 136.7, 135.0 (d, J = 17.8 Hz), 132.1 (d, J = 9.6 Hz), 132.1 (d, J = 2.5 Hz), 131.4 (d, J = 96.0 Hz), 131.3 (d, J = 102.6 Hz), 130.3, 128.7 (d, J = 12.1 Hz), 128.7, 128.4, 125.7, 20.0, 14.6 (d, J = 10.9 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.68; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{22}H_{22}OP]^+$ 333.1403, found 333.1405.

(*E*)-(1-(4-methoxyphenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3e**)



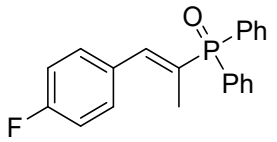
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (44 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.71 (m, 4H), 7.57-7.52 (m, 2H), 7.50-7.43 (m, 4H), 7.34 (d, J = 8.4 Hz, 2H), 7.15 (d, J = 22.2 Hz, 1H), 6.90 (d, J = 8.9 Hz, 2H), 3.81 (s, 3H), 2.11 (d, J = 13.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.8, 142.4 (d, J = 11.0 Hz), 132.2 (d, J = 9.4 Hz), 132.0 (d, J = 2.7 Hz), 131.6 (d, J = 102.6 Hz), 131.3, 128.7 (d, J = 11.9 Hz), 128.6 (d, J = 19.6 Hz), 127.5 (d, J = 98.7 Hz), 113.9, 55.4, 15.3 (d, J = 11.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.91; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{22}H_{22}O_2P]^+$ 349.1352, found 349.1349.

(*E*)-diphenyl(1-(4-(trifluoromethyl)phenyl)prop-1-en-2-yl)phosphine oxide (**3f**)



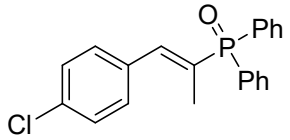
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (56 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.71 (m, 4H), 7.65-7.61 (m, 2H), 7.60-7.55 (m, 2H), 7.53-7.44 (m, 6H), 7.29 (d, J = 21.6 Hz, 1H), 2.09 (d, J = 13.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.1 (d, J = 10.7 Hz), 139.3 (d, J = 18.8 Hz), 133.2 (d, J = 95.1 Hz), 132.2 (d, J = 2.8 Hz), 132.1 (d, J = 9.7 Hz), 130.8 (d, J = 102.6 Hz), 130.1 (q, J = 32.4 Hz), 129.6, 128.8 (d, J = 11.9 Hz), 125.4 (q, J = 3.7 Hz), 124.0 (q, J = 272.7 Hz), 15.2 (d, J = 10.6 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.58; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.65; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{22}H_{19}F_3OP]^+$ 387.1120, found 387.1125.

(*E*)-(1-(4-fluorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3g**)



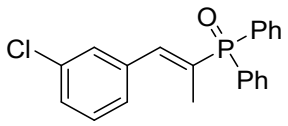
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (36 mg, 57%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77-7.71 (m, 4H), 7.58-7.54 (m, 2H), 7.52-7.46 (m, 4H), 7.39-7.33 (m, 2H), 7.19 (d, J = 21.7 Hz, 1H), 7.06 (t, J = 8.6 Hz, 2H), 2.08 (d, J = 13.8 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 162.5 (d, J = 249.2 Hz), 141.6 (d, J = 10.9 Hz), 132.1 (d, J = 9.6 Hz), 132.1 (d, J = 2.7 Hz), 131.9 (dd, J = 18.8, 3.5 Hz), 131.4 (d, J = 8.3 Hz), 131.1 (d, J = 102.4 Hz), 130.0 (d, J = 97.1 Hz), 128.7 (d, J = 11.9 Hz), 115.5 (d, J = 21.5 Hz), 15.1 (d, J = 10.9 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.24; ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.11; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{19}FOP]^+$ 337.1152, found 337.1158.

(*E*)-(1-(4-chlorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3h**)



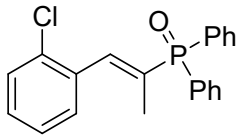
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (45 mg, 64%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77-7.70 (m, 4H), 7.59-7.53 (m, 2H), 7.51-7.46 (m, 4H), 7.36-7.32 (m, 2H), 7.31-7.27 (m, 2H), 7.19 (d, J = 21.6 Hz, 1H), 2.07 (d, J = 13.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.3 (d, J = 10.8 Hz), 134.2, 134.1 (d, J = 18.2 Hz), 132.1, 132.0 (d, J = 9.6 Hz), 131.0 (d, J = 96.9 Hz), 130.9 (d, J = 102.9 Hz), 130.7, 128.7 (d, J = 11.8 Hz), 128.6 (d, J = 2.6 Hz), 15.1 (d, J = 10.7 Hz); ^{31}P NMR (202 MHz, $CDCl_3$) δ 32.97. HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{19}ClOP]^+$ 353.0857, found 353.0853.

(*E*)-(1-(3-chlorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3i**)



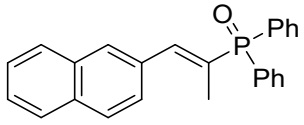
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (49 mg, 70%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72-7.63 (m, 4H), 7.52-7.46 (m, 2H), 7.45-7.40 (m, 4H), 7.27-7.14 (m, 4H), 7.09 (d, *J* = 21.5 Hz, 1H), 2.01 (d, *J* = 13.7 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.2 (d, *J* = 11.0 Hz), 137.7 (d, *J* = 19.0 Hz), 134.5, 132.4 (d, *J* = 95.7 Hz), 132.2 (d, *J* = 9.8 Hz), 132.1 (d, *J* = 2.8 Hz), 131.1 (d, *J* = 102.3 Hz), 129.8, 129.3, 128.8 (d, *J* = 11.9 Hz), 128.5, 127.6, 15.2 (d, *J* = 10.8 Hz); ^{31}P NMR (202 MHz, $CDCl_3$) δ 32.85; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{19}ClOP]^+$ 353.0857, found 353.0860.

(*E*)-(1-(2-chlorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3j**)



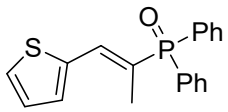
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (39 mg, 55%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.79 (m, 4H), 7.64-7.49 (m, 6H), 7.45-7.41 (m, 1H), 7.35-7.25 (m, 3H), 7.10 (d, *J* = 21.6 Hz, 1H), 2.03 (d, *J* = 13.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.2 (d, *J* = 13.3 Hz), 134.4 (d, *J* = 19.0 Hz), 133.8, 133.5 (d, *J* = 95.9 Hz), 132.2 (d, *J* = 9.6 Hz), 132.1 (d, *J* = 2.4 Hz), 131.0 (d, *J* = 102.4 Hz), 130.2, 129.7, 129.5, 128.8 (d, *J* = 12.0 Hz), 126.6, 14.5 (d, *J* = 10.4 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.82; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{19}ClOP]^+$ 353.0857, found 353.0854.

(*E*)-(1-(naphthalen-2-yl)prop-1-en-2-yl)diphenylphosphine oxide (**3k**)



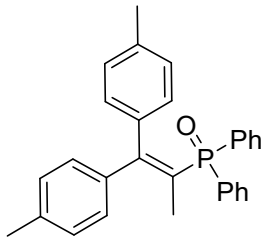
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (46 mg, 62 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.77 (m, 8H), 7.60-7.55 (m, 2H), 7.54-7.46 (m, 7H), 7.38 (d, *J* = 21.9 Hz, 1H), 2.20 (d, J = 13.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8 (d, *J* = 10.9 Hz), 133.4 (d, *J* = 19.0 Hz), 133.2, 133.0, 132.2 (d, *J* = 9.6 Hz), 132.1 (d, *J* = 2.8 Hz), 131.4 (d, *J* = 102.3 Hz), 130.7 (d, *J* = 96.8 Hz), 129.2, 128.8 (d, *J* = 11.9 Hz), 128.4, 128.1, 127.8, 127.0, 126.8, 126.6, 15.4 (d, *J* = 10.9 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.50; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{25}H_{22}OP]^+$ 369.1403, found 369.1408.

(*E*)-diphenyl(1-(thiophen-2-yl)prop-1-en-2-yl)phosphine oxide (**3l**)



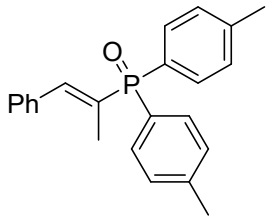
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (41 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77-7.68 (m, 4H), 7.58-7.52 (m, 2H), 7.51-7.40 (m, 6H), 7.20 (d, J = 3.6 Hz, 1H), 7.09 (dd, J = 5.1, 3.7 Hz, 1H), 2.15 (d, J = 13.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.3 (d, *J* = 21.8 Hz), 135.2 (d, *J* = 12.4 Hz), 132.2 (d, *J* = 9.7 Hz), 132.1 (d, *J* = 3.0 Hz), 131.5 (d, *J* = 104.6 Hz), 131.4, 128.8, 128.7 (d, *J* = 12.2 Hz), 127.3, 126.1 (d, *J* = 100.2 Hz), 15.9 (d, *J* = 10.7 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.29; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{19}H_{18}OPS]^+$ 325.0810, found 325.0812.

(1,1-di-p-tolylprop-1-en-2-yl)diphenylphosphine oxide (**3m**)



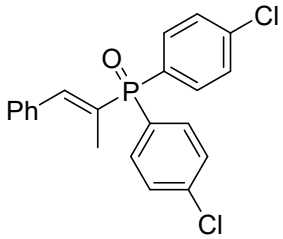
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (52 mg, 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75-7.65 (m, 4H), 7.39-7.25 (m, 6H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.08 (d, *J* = 7.9 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 6.71 (d, *J* = 7.7 Hz, 2H), 2.37 (s, 3H), 2.13 (s, 3H), 1.93 (d, *J* = 13.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9 (d, *J* = 9.2 Hz), 139.7 (d, *J* = 15.4 Hz), 138.5 (d, *J* = 8.0 Hz), 137.7 (d, *J* = 15.1 Hz), 134.4 (d, *J* = 102.2 Hz), 131.3 (d, *J* = 9.4 Hz), 130.6 (d, *J* = 2.8 Hz), 130.4, 129.3, 129.0, 128.2, 128.1, 128.1, 126.3 (d, *J* = 97.0 Hz), 21.6 (d, *J* = 12.7 Hz), 21.4, 21.2; ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.39; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{29}H_{28}OP]^+$ 423.1872, found 423.1876.

(*E*)-(1-phenylprop-1-en-2-yl)di-p-tolylphosphine oxide (**3n**)



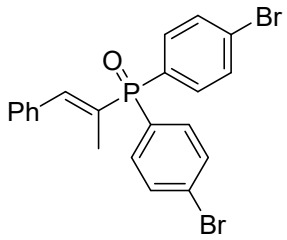
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (41 mg, 60%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66-7.59 (m, 4H), 7.38-7.33 (m, 4H), 7.30-7.27 (m, 5H), 7.19 (d, *J* = 21.8 Hz, 1H), 2.41 (s, 6H), 2.09 (d, *J* = 13.7 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.5 (d, *J* = 3.4 Hz), 142.4 (d, *J* = 12.0 Hz), 136.1 (d, *J* = 19.0 Hz), 132.2 (d, *J* = 10.0 Hz), 130.9 (d, *J* = 97.1 Hz), 129.5, 129.4 (d, *J* = 12.4 Hz), 128.5, 128.4, 128.2 (d, *J* = 104.4 Hz), 21.7, 15.2 (d, *J* = 10.8 Hz); ^{31}P NMR (202 MHz, $CDCl_3$) δ 33.61; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{23}H_{24}OP]^+$ 347.1559, found 347.1562.

(*E*)-bis(4-chlorophenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3o**)



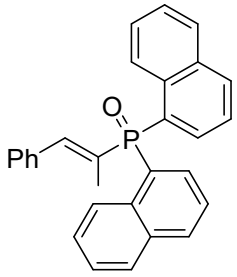
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (42 mg, 54%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.63 (m, 4H), 7.51-7.46 (m, 4H), 7.42-7.31 (m, 5H), 7.20 (d, *J* = 22.4 Hz, 1H), 2.10 (d, *J* = 14.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5 (d, *J* = 11.1 Hz), 138.9 (d, *J* = 3.2 Hz), 135.5 (d, *J* = 19.4 Hz), 133.5 (d, *J* = 10.4 Hz), 129.6, 129.5 (d, *J* = 103.6 Hz), 129.4 (d, *J* = 98.6 Hz), 129.2 (d, *J* = 12.6 Hz), 128.8, 128.6, 15.1 (d, *J* = 10.9 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.04; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{18}Cl_2OP]^+$ 387.0467, found 387.0462.

(*E*)-bis(4-bromophenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3p**)



Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (48 mg, 51%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68-7.56 (m, 8H), 7.43-7.31 (m, 5H), 7.20 (d, *J* = 22.4 Hz, 1H), 2.09 (d, *J* = 14.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5 (d, *J* = 10.9 Hz), 135.4 (d, *J* = 19.4 Hz), 133.6 (d, *J* = 10.3 Hz), 132.2 (d, *J* = 12.3 Hz), 130.0 (d, *J* = 102.9 Hz), 129.6, 129.3 (d, *J* = 98.5 Hz), 128.8, 128.6, 127.6 (d, *J* = 3.4 Hz), 15.1 (d, *J* = 11.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.29; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{21}H_{18}Br_2OP]^+$ 474.9457, found 474.9461.

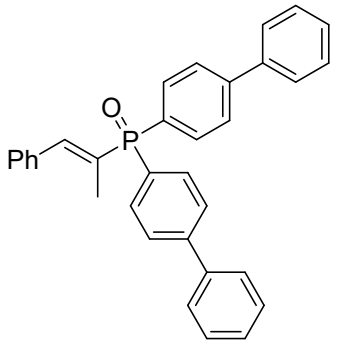
(*E*)-di(naphthalen-1-yl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3q**)



Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (54 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92-8.87 (m, 2H), 8.03 (d, *J* = 8.2 Hz, 2H), 7.95-7.90 (m, 2H), 7.60-7.49 (m, 6H), 7.45-7.35 (m, 7H), 7.34-7.29 (m, 1H), 2.15 (d, *J* = 13.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.2 (d, *J* = 10.8 Hz), 136.2 (d, *J* = 19.2 Hz), 134.3 (d, *J* = 20.8 Hz), 134.2 (d, *J* = 4.0 Hz), 133.3 (d,

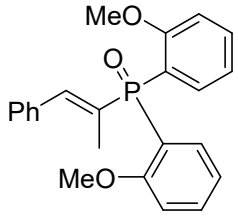
J = 9.8 Hz), 133.2, 131.1 (d, J = 96.6 Hz), 129.6, 128.9, 128.6, 128.5, 128.2 (d, *J* = 100.5 Hz), 127.9 (d, *J* = 5.2 Hz), 127.5, 126.7, 124.5 (d, *J* = 14.0 Hz), 16.0 (d, *J* = 10.4 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 40.03; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{29}H_{24}OP]^+$ 419.1559, found 419.1556.

(*E*)-di([1,1'-biphenyl]-4-yl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3r**)



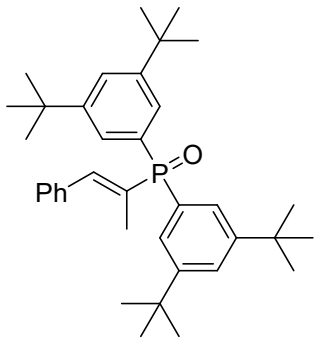
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (68 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.84 (m, 4H), 7.76-7.72 (m, 4H), 7.66-7.61 (m, 4H), 7.50-7.45 (m, 4H), 7.44-7.37 (m, 6H), 7.36-7.29 (m, 2H), 2.19 (d, J = 13.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9 (d, *J* = 2.6 Hz), 142.9 (d, *J* = 10.7 Hz), 140.1, 136.0 (d, *J* = 19.0 Hz), 132.8 (d, *J* = 9.9 Hz), 130.4 (d, *J* = 97.5 Hz), 130.0 (d, *J* = 102.7 Hz), 129.6, 129.1, 128.6, 128.5 (d, *J* = 3.5 Hz), 128.3, 127.5, 127.4, 15.3 (d, *J* = 10.8 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.24; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{33}H_{28}OP]^+$ 471.1872, found 471.1876.

(*E*)-bis(2-methoxyphenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3s**)



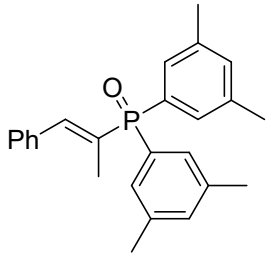
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (61 mg, 80%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.55 (m, 2H), 7.41-7.38 (m, 2H), 7.32-7.25 (m, 5H), 7.18 (d, *J* = 7.6 Hz, 1H), 6.96-6.93 (m, 2H), 6.85-6.82 (m, 2H), 3.59 (s, 6H), 2.00 (d, *J* = 14.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.2 (d, *J* = 2.7 Hz), 140.2 (d, *J* = 10.7 Hz), 136.8 (d, *J* = 19.4 Hz), 134.3 (d, *J* = 7.7 Hz), 133.4, 131.9 (d, *J* = 101.9 Hz), 129.4, 128.3, 127.7, 120.8 (d, *J* = 12.1 Hz), 120.6 (d, *J* = 104.7 Hz), 111.0 (d, *J* = 6.4 Hz), 55.4, 15.1 (d, *J* = 12.0 Hz); ^{31}P NMR (202 MHz, $CDCl_3$) δ 29.89; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{23}H_{24}O_3P]^+$ 379.1458, found 379.1453.

(*E*)-bis(3,5-di-tert-butylphenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3t**)



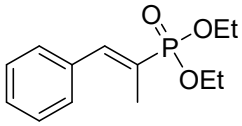
Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (67 mg, 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.55 (m, 6H), 7.41-7.34 (m, 4H), 7.32-7.25 (m, 2H), 2.07 (d, *J* = 13.6 Hz, 3H), 1.31 (s, 36H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.9 (d, *J* = 11.5 Hz), 142.0 (d, *J* = 10.3 Hz), 136.4 (d, *J* = 18.2 Hz), 131.3 (d, *J* = 95.4 Hz), 130.4 (d, *J* = 102.3 Hz), 129.3, 128.4, 128.1, 126.3 (d, *J* = 10.3 Hz), 125.9 (d, *J* = 2.7 Hz), 35.1, 31.4, 15.1 (d, *J* = 11.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 35.37; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{37}H_{52}OP]^+$ 543.3750, found 543.3754.

(*E*)-bis(3,5-dimethylphenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3u**)



Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (49 mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.29 (m, 9H), 7.23-7.15 (m, 3H), 2.34 (s, 12H), 2.11 (d, *J* = 13.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.3 (d, *J* = 11.0 Hz), 138.3 (d, *J* = 12.3 Hz), 136.2 (d, *J* = 18.8 Hz), 133.7 (d, *J* = 2.9 Hz), 131.2 (d, *J* = 101.4 Hz), 130.9 (d, *J* = 96.0 Hz), 129.7 (d, *J* = 9.5 Hz), 129.6, 128.5, 128.3, 21.5, 15.3 (d, *J* = 10.7 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.96; HRMS (ESI) m/z $[M+H]^+$ calcd for $[C_{25}H_{28}OP]^+$ 375.1872, found 375.1875.

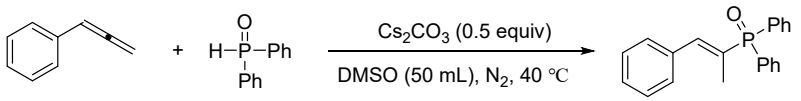
diethyl (*E*)-(1-phenylprop-1-en-2-yl)phosphonate (**3v**)



Prepared following the general procedure, purified by column chromatography (2/1 petroleum ether/ ethyl acetate), and isolated as a colorless oil (30 mg, 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 24.8 Hz, 1H), 7.39-7.36 (m, 4H), 7.34-7.28 (m, 1H), 4.21-4.02 (m, 4H), 2.06 (d, *J* = 15.3 Hz, 3H), 1.35 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8 (d, *J* = 11.8 Hz), 135.8 (d, *J* = 23.8 Hz), 129.6, 128.5, 126.0 (d, *J* = 178.1 Hz), 61.9 (d, *J* = 5.6 Hz), 16.5 (d, *J* = 6.2 Hz), 14.4 (d, *J* = 9.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 22.12; HRMS (ESI) m/z

$[M+H]^+$ calcd for $[C_{13}H_{20}O_3P]^+$ 255.1145, found 255.1140.

6. Procedure of Gram-Scale Experiment

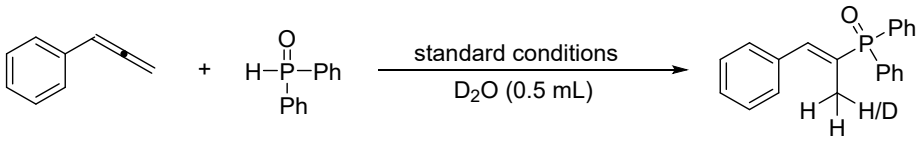


1a 10 mmol | **2a** 5 mmol | **3a** 1.14 g, 72% yield

To an oven dried Schlenk tube (100 mL) containing a magnetic stir bar, phosphine oxides (1.01 g, 5 mmol, 1 equiv) and Cs_2CO_3 (0.82 g, 2.5 mmol, 0.5 equiv) were added. The Schlenk tube was sealed and degassed via vacuum evacuation and subsequent backfilled with N_2 for three times. Subsequently, allenes (1.16 g, 10 mmol, 2 equiv) and anhydrous DMSO (50 mL) were added. Then the reaction mixture was placed in an oil bath and heated for 24 hours at 40 °C. Upon completion, water and EtOAc were added to the reaction mixture, and the aqueous phase was extracted with EtOAc. The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by column chromatography isolation on silica gel (petroleum ether: ethyl acetate = 2:1 *v*/*v*) to afford white solid (*E*)-diphenyl(1-phenylprop-1-en-2-yl)phosphine oxide **3a** in 72% yield (1.14 g).

7. Preliminary mechanistic studies

Deuterium labeling experiment



70% yield, 92% D

To an oven dried Schlenk tube (10 mL) with a magnetic stir bar, phosphine oxides (0.2 mmol, 1.0 equiv.), Cs_2CO_3 (0.1 mmol, 0.5 equiv.) were added. The Schlenk tube was sealed and degassed via vacuum evacuation and subsequent backfilled with N_2 for three times. Subsequently, allenes (0.4 mmol, 2.0 equiv.), D_2O (0.5 mL) and anhydrous DMSO (2 mL) were added. Then the reaction mixture was placed in an oil bath and heated for 12 hours at 40 °C. Upon completion, water and EtOAc were added to the reaction mixture, and the aqueous phase was extracted with EtOAc. The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by column chromatography isolation on silica gel (petroleum ether: ethyl acetate = 2:1 *v*/*v*) to give the D-labelled product.

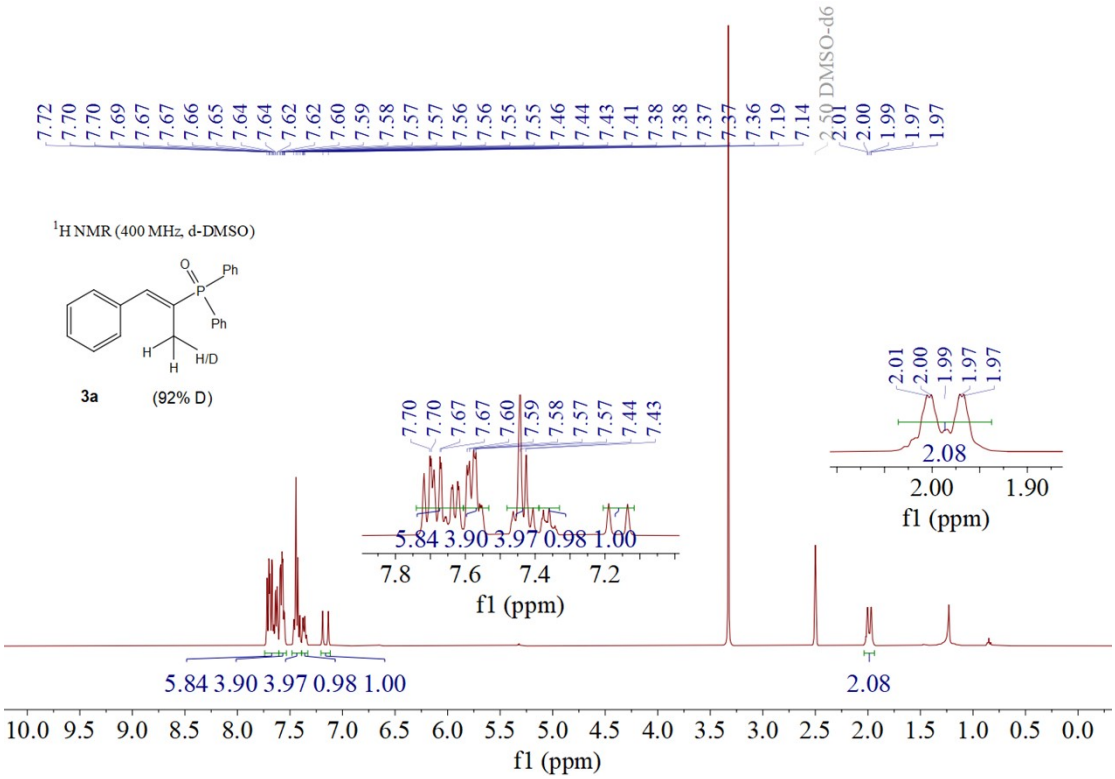


Figure S1. ^{1}H NMR Spectra of compound **3a** (0.5 mL D_2O)

8. References

[S1] S. Rej, H. F. T. Klare, M. Oestreich, *Org. Lett*., 2022, **24**, 1346-1350.

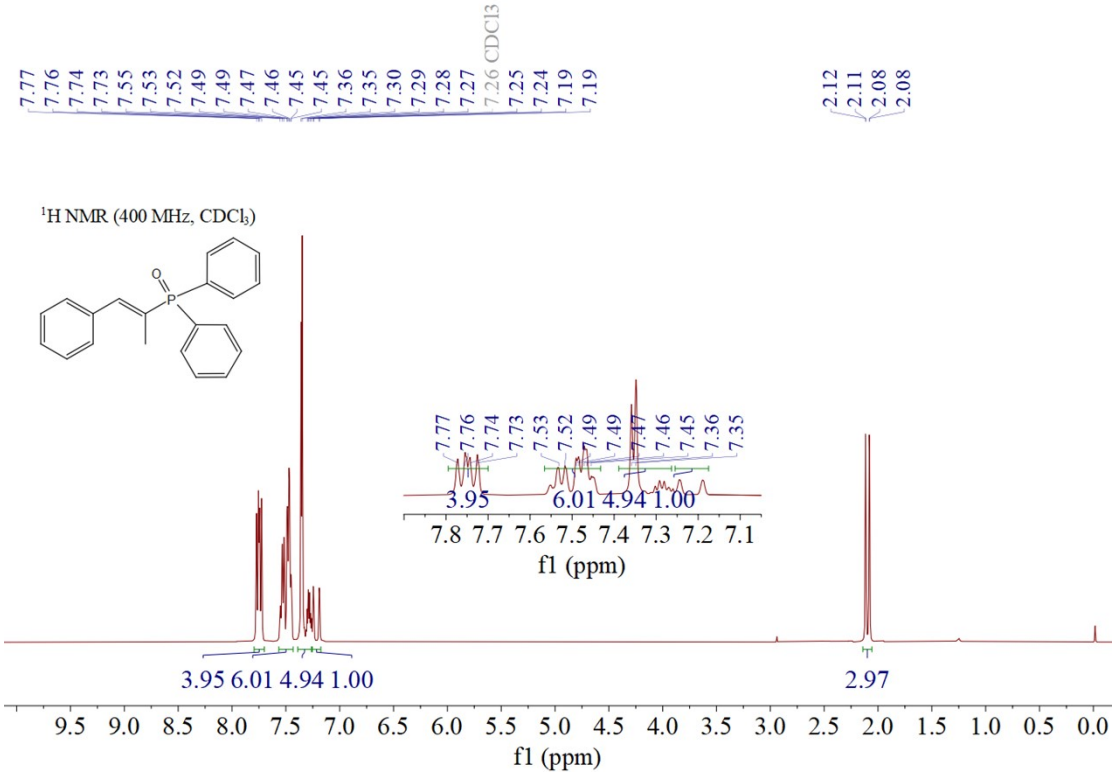
[S2] M. Brochetta, T. Borsari, A. Gandini, S. Porey, A. Deb, E. Casali, A. Chakraborty, G. Zanoni, D. Maiti, *Chem. Eur. J*., 2019, **25**, 750-753.

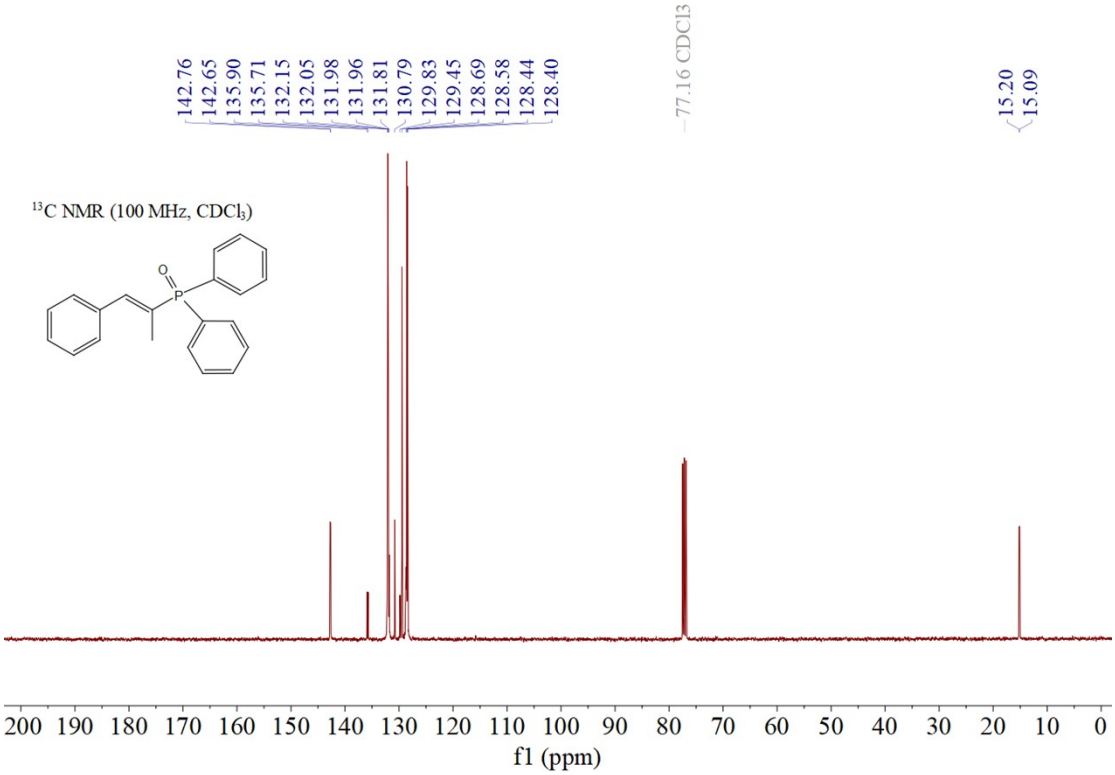
[S3] J. Jim, J. Choi, H. S. Kim, I. S. Kim, K. C. Nam, J. Kim, S. Lee, *J. Org. Chem*., 2016, **81**, 303-308.

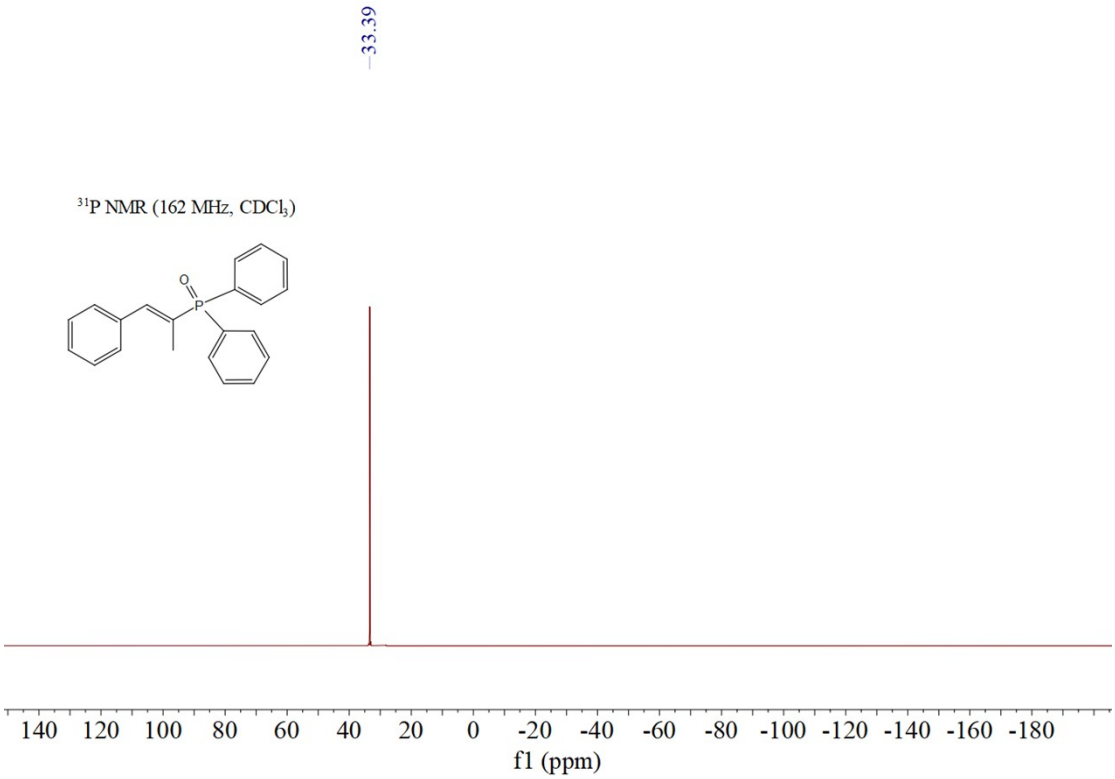
[S4] S. Yamazaki, Y. Yamamoto, Y. Fukushima, M. Takebayashi, T. Ukai, Y. Mikata, *J. Org. Chem*., 2010, **75**, 5216-5222.

9. NMR spectra

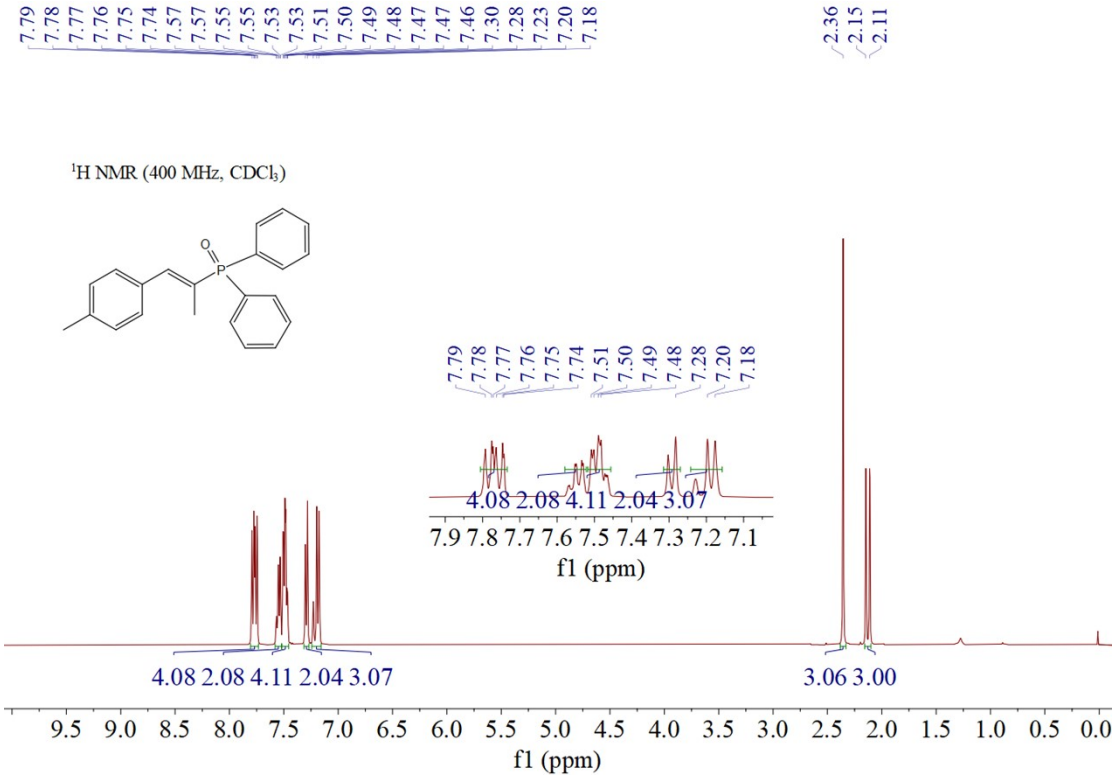
(*E*)-diphenyl(1-phenylprop-1-en-2-yl)phosphine oxide (**3a**)

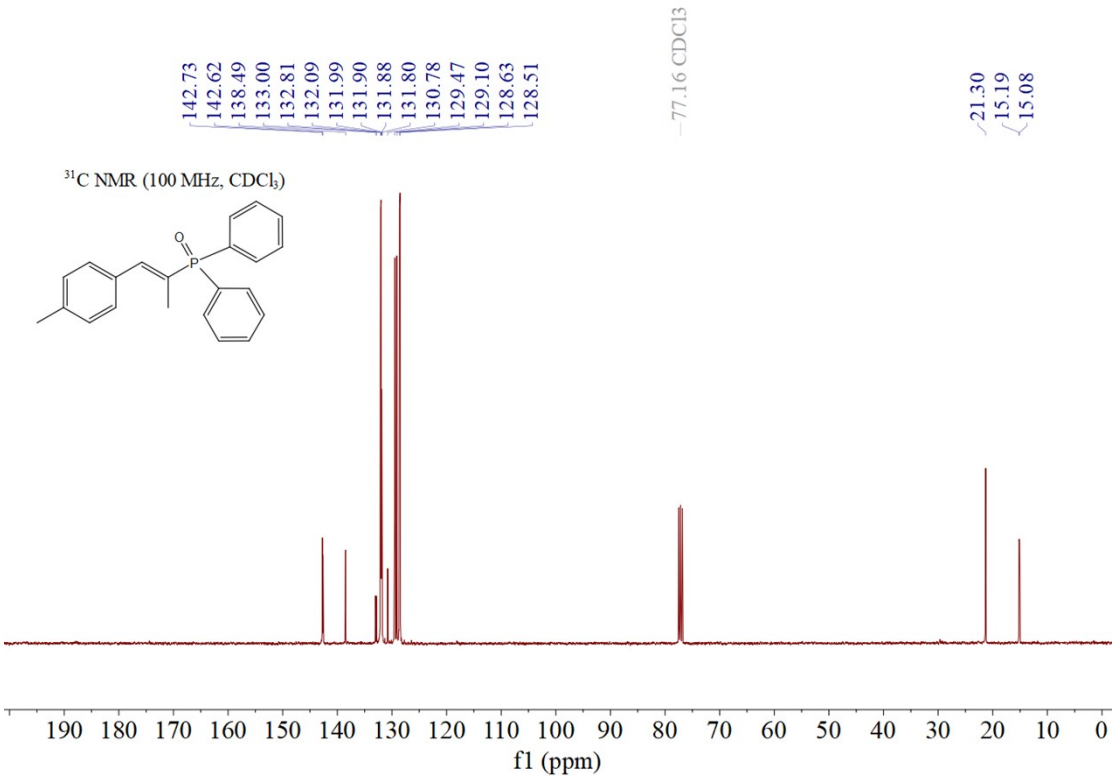


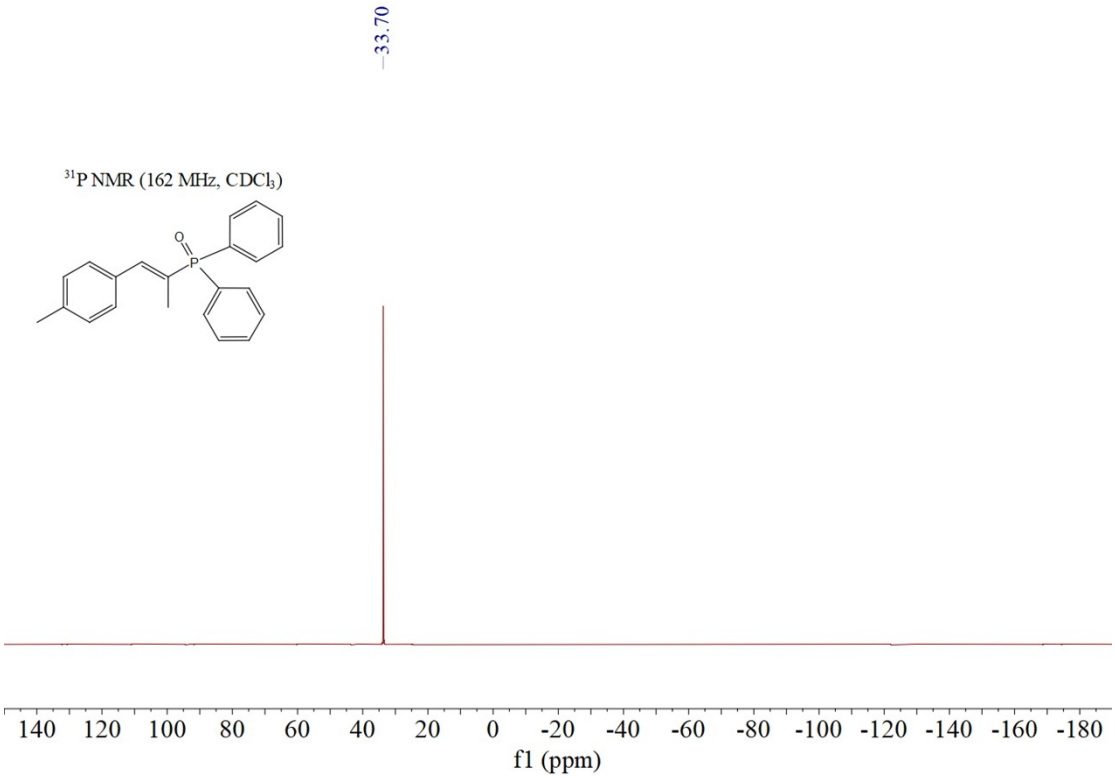




(*E*)-diphenyl(1-(p-tolyl)prop-1-en-2-yl)phosphine oxide (**3b**)

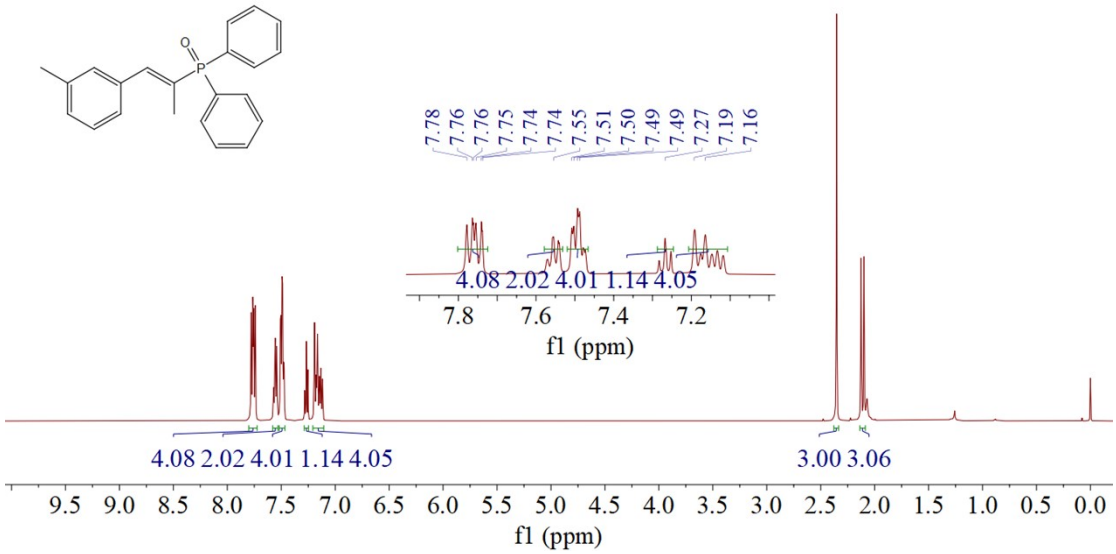


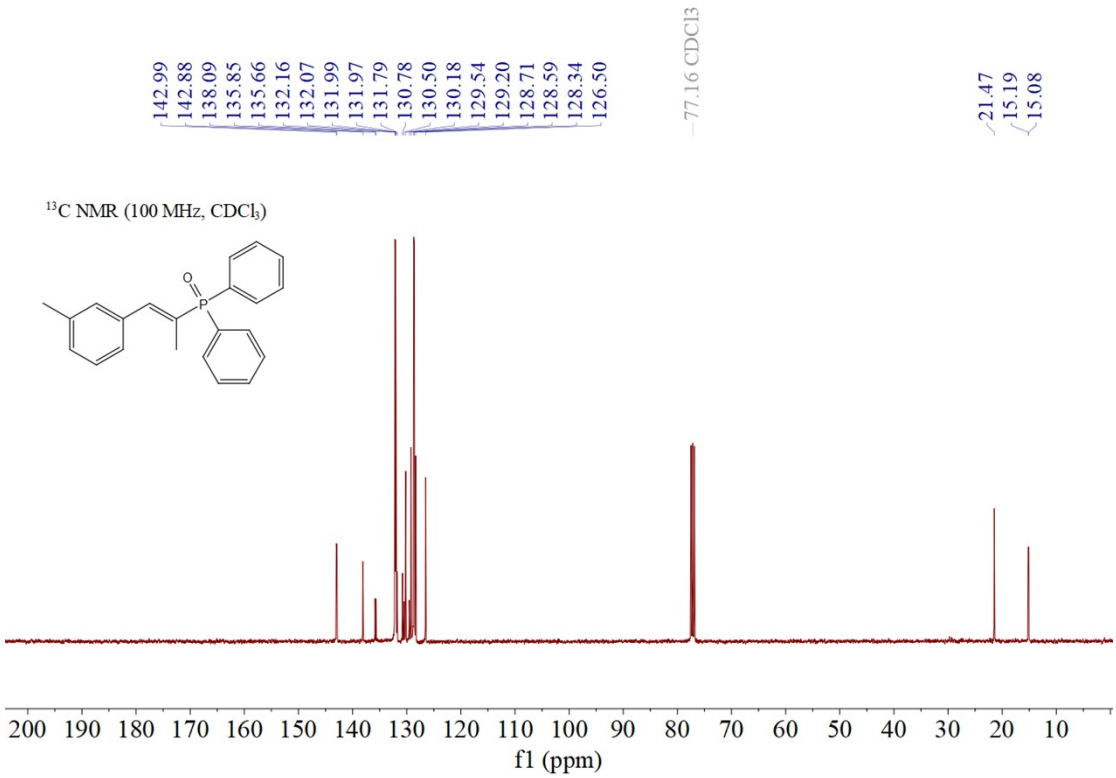




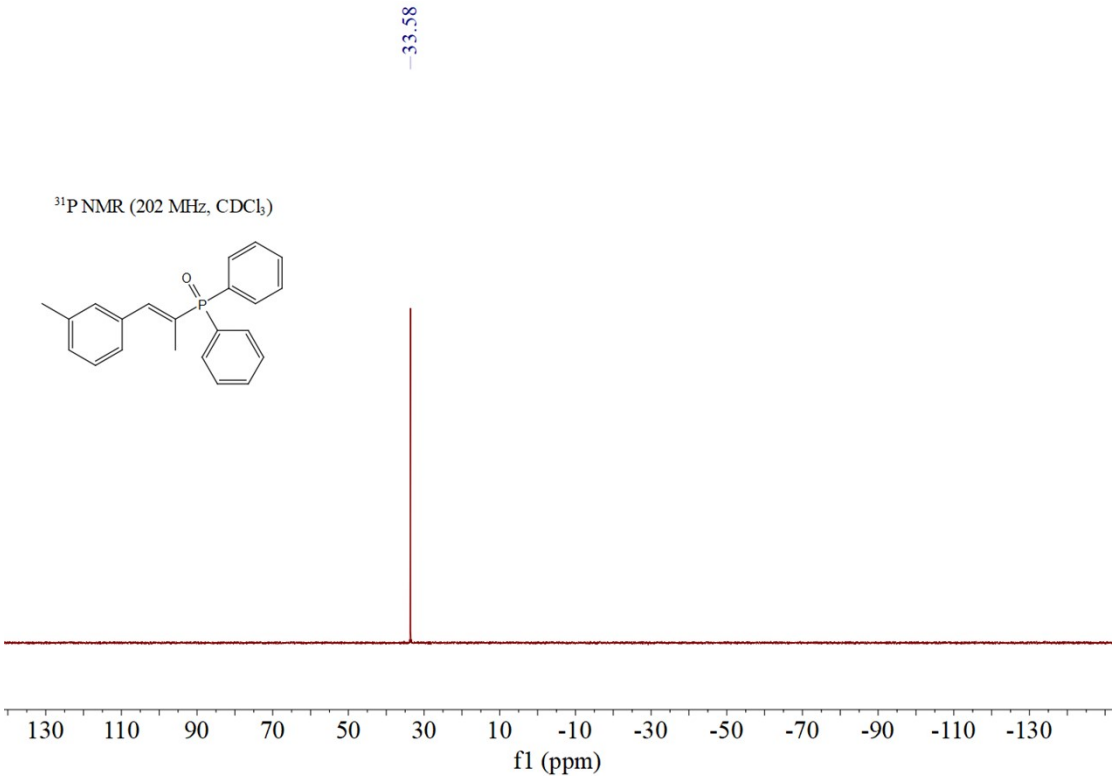
(*E*)-diphenyl(1-(m-tolyl)prop-1-en-2-yl)phosphine oxide (**3c**)

^{1}H NMR (500 MHz, $CDCl_3$)

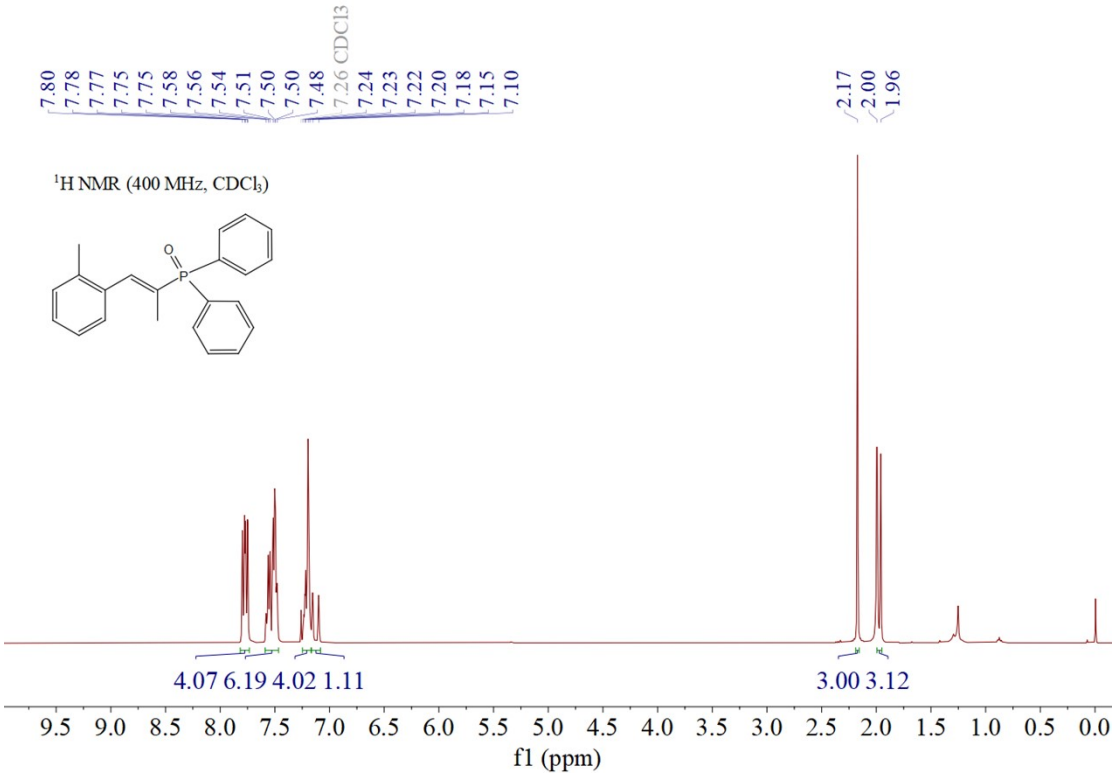


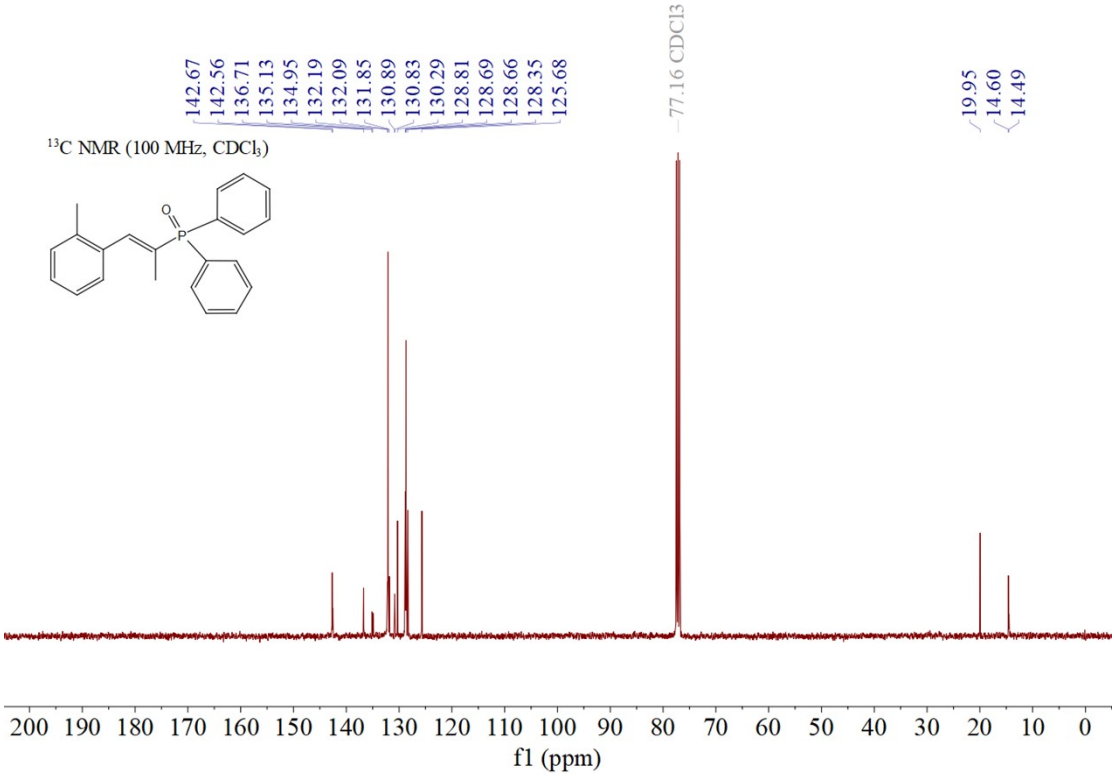


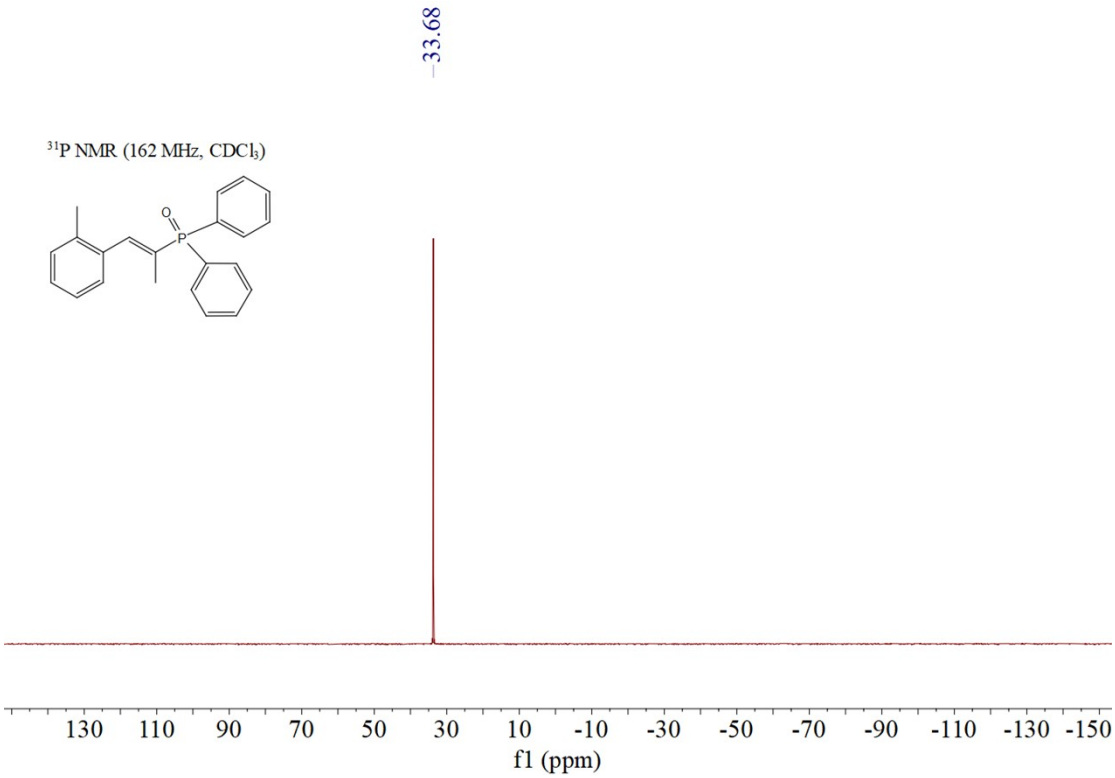
^{13}C NMR (100 MHz, $CDCl_3$)



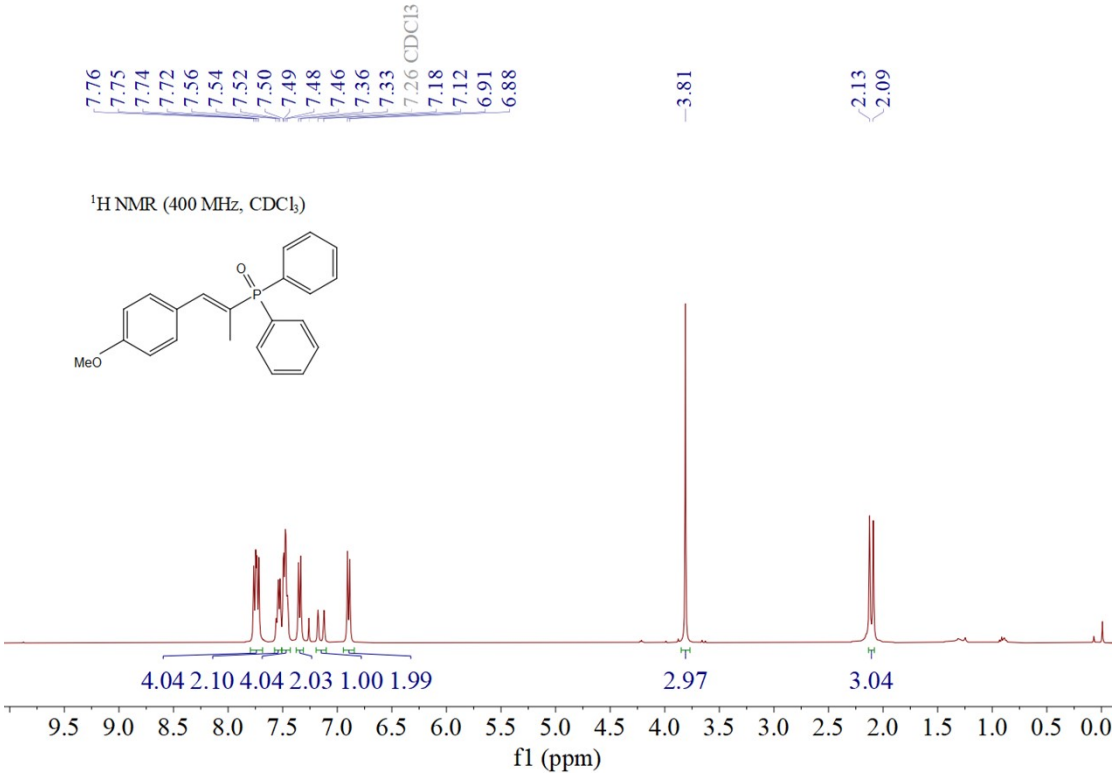
(*E*)-diphenyl(1-(o-tolyl)prop-1-en-2-yl)phosphine oxide (**3d**)

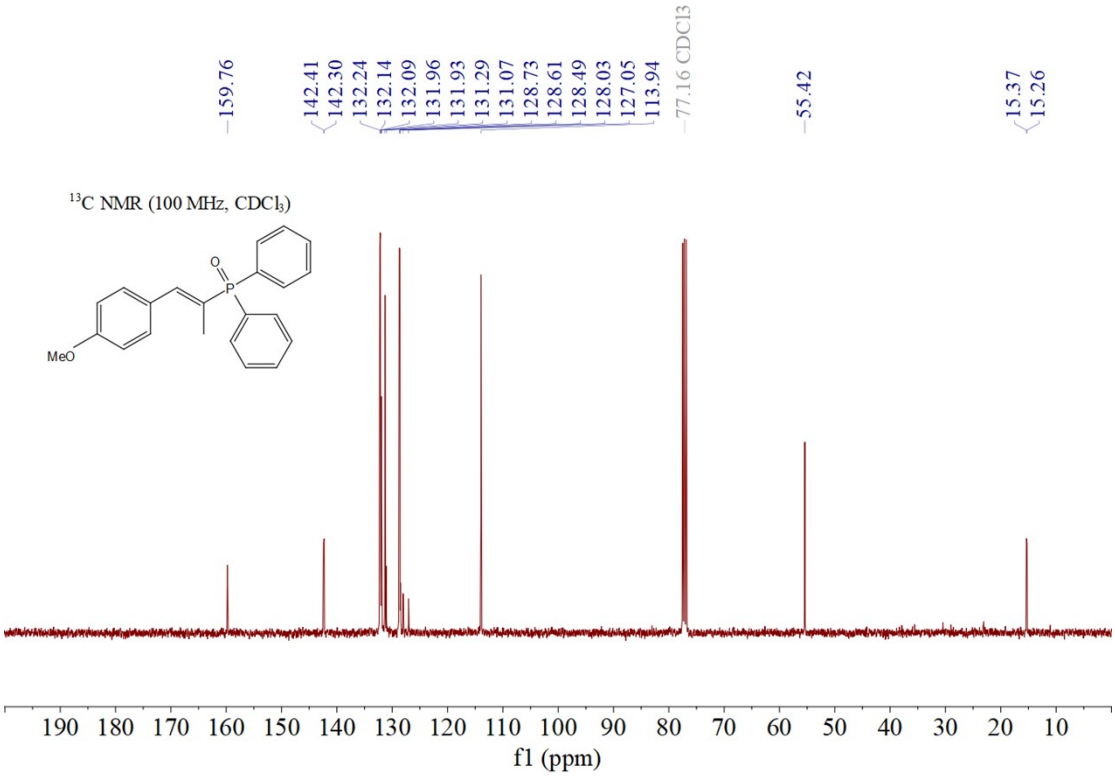


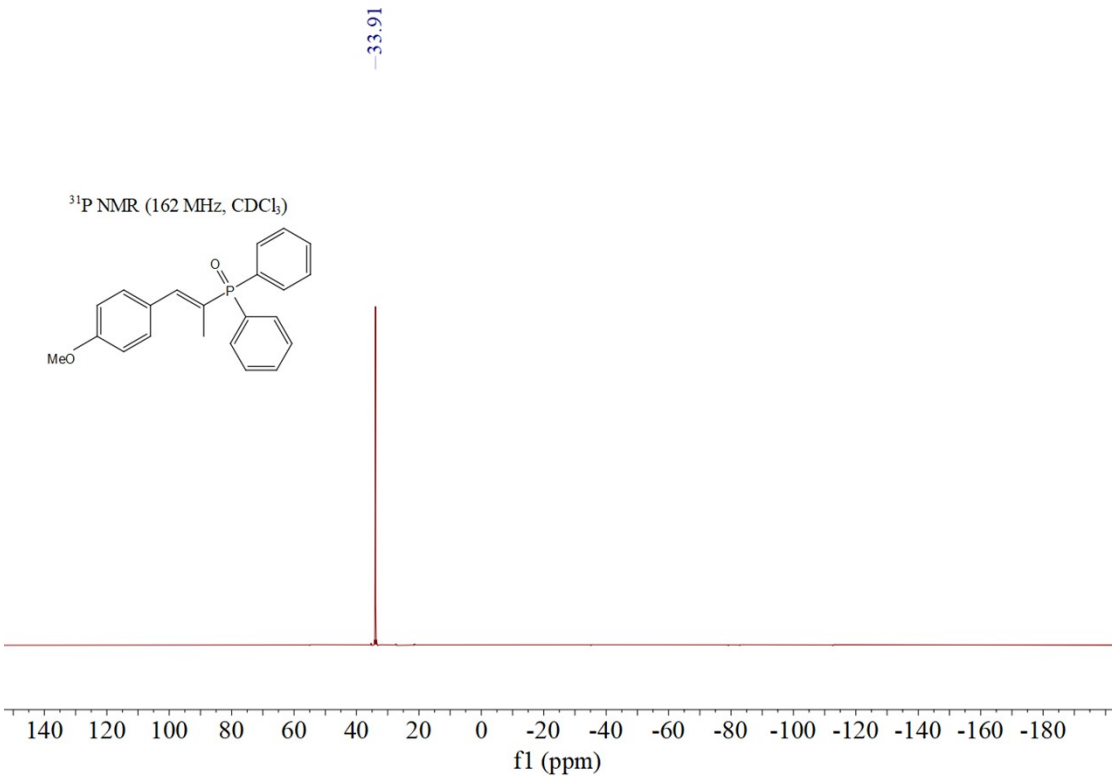




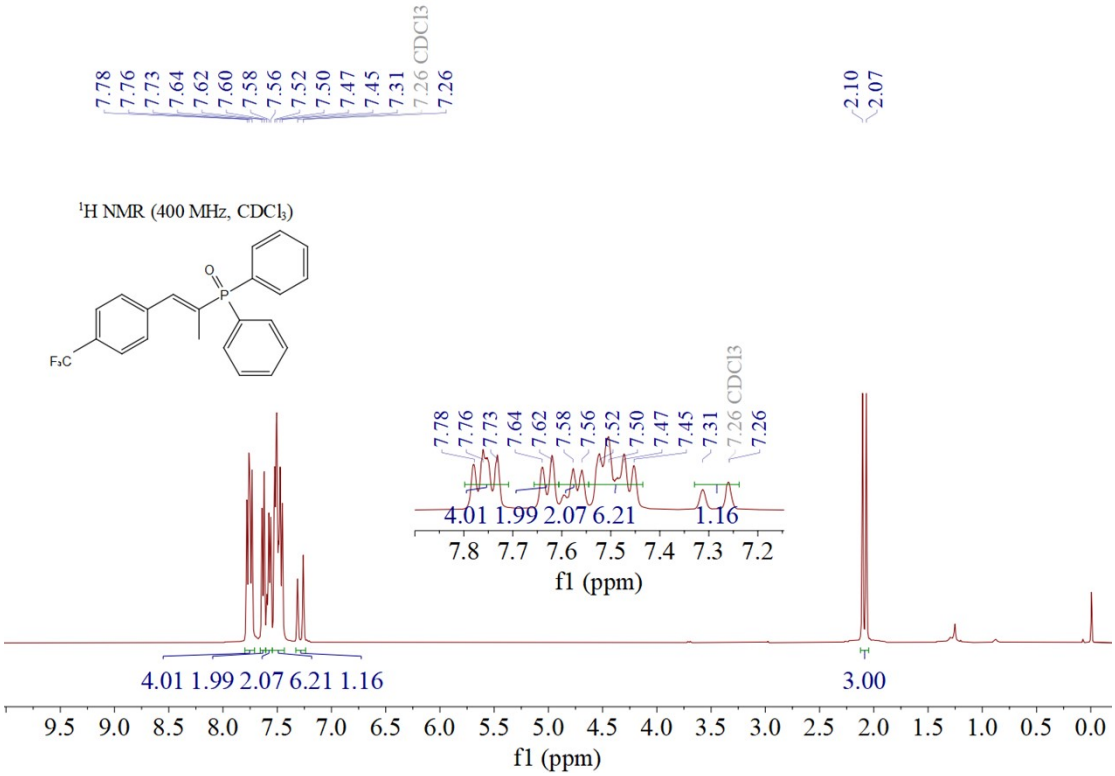
(*E*)-(1-(4-methoxyphenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3e**)

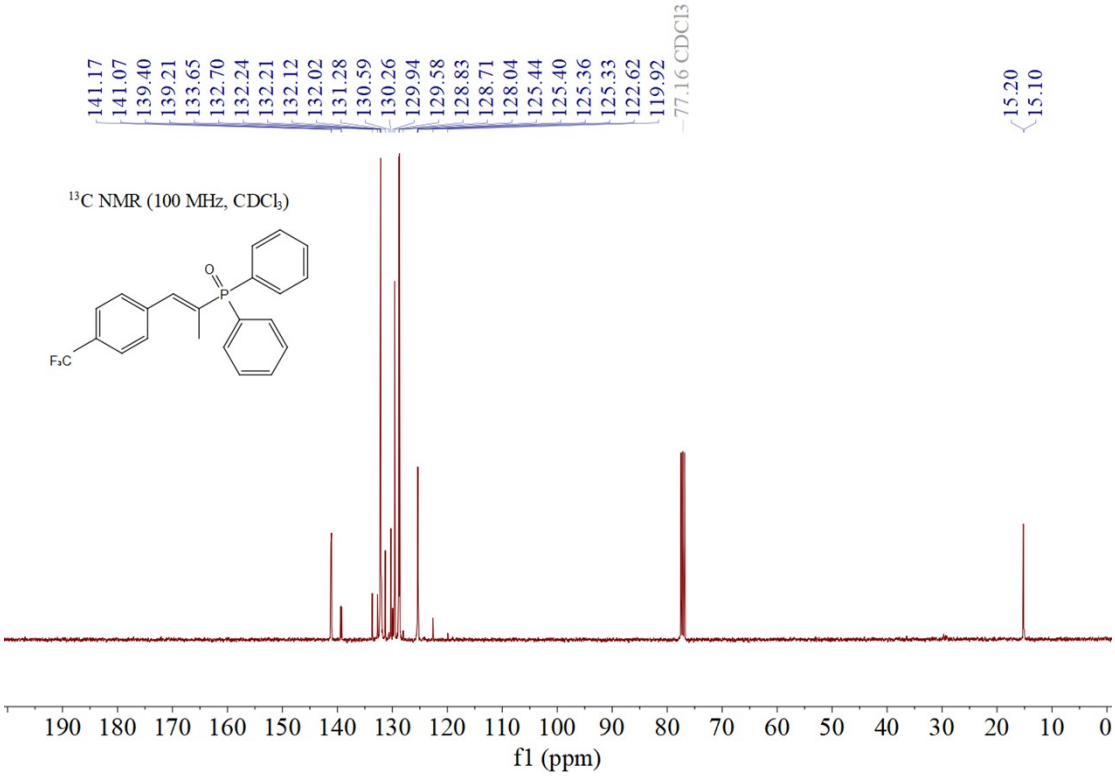

7.76
7.75
7.74
7.72
7.56
7.54
7.52
7.50
7.49
7.48
7.46
7.36
7.33
7.26 CDCl3
7.18
7.12
6.91
6.88
3.81
2.13
2.09
1H NMR (400 MHz, CDCl3)
MeO
4.04 2.10 4.04 2.03 1.00 1.99
2.97
3.04
f1 (ppm)

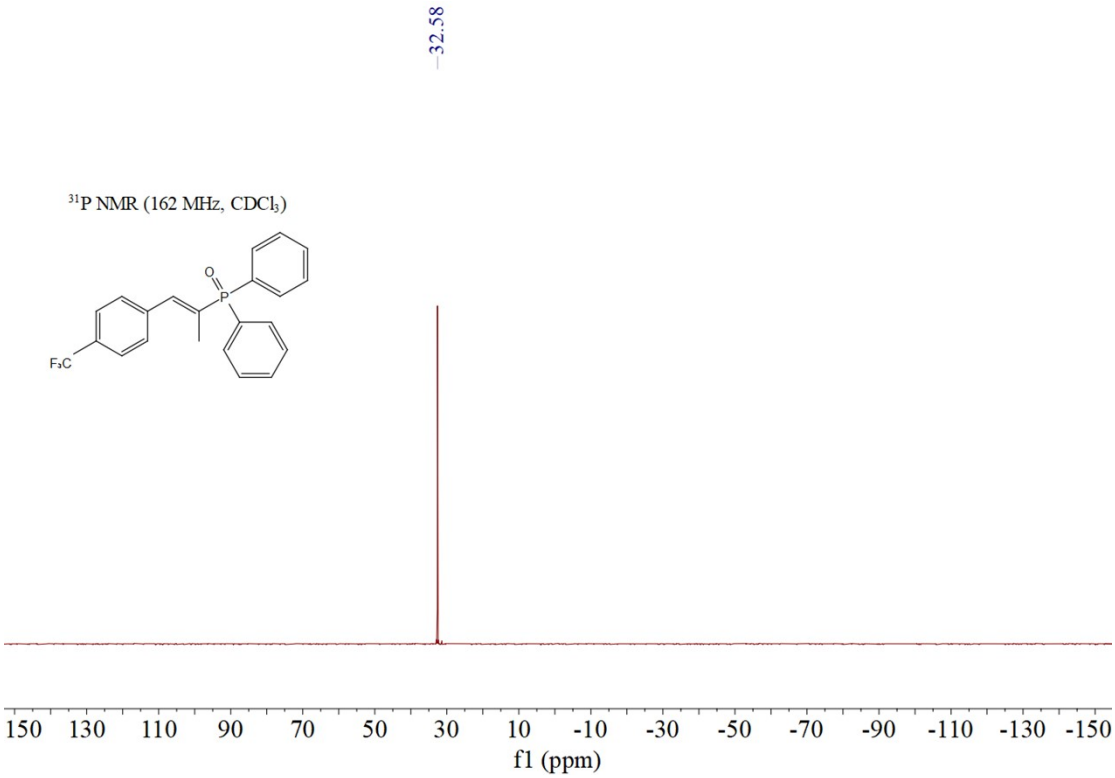

159.76
142.41
142.30
132.24
132.14
132.09
131.96
131.93
131.29
131.07
128.73
128.61
128.49
128.03
127.05
113.94
77.16 CDCl3
55.42
15.37
15.26
13C NMR (100 MHz, CDCl3)
MeO
f1 (ppm)

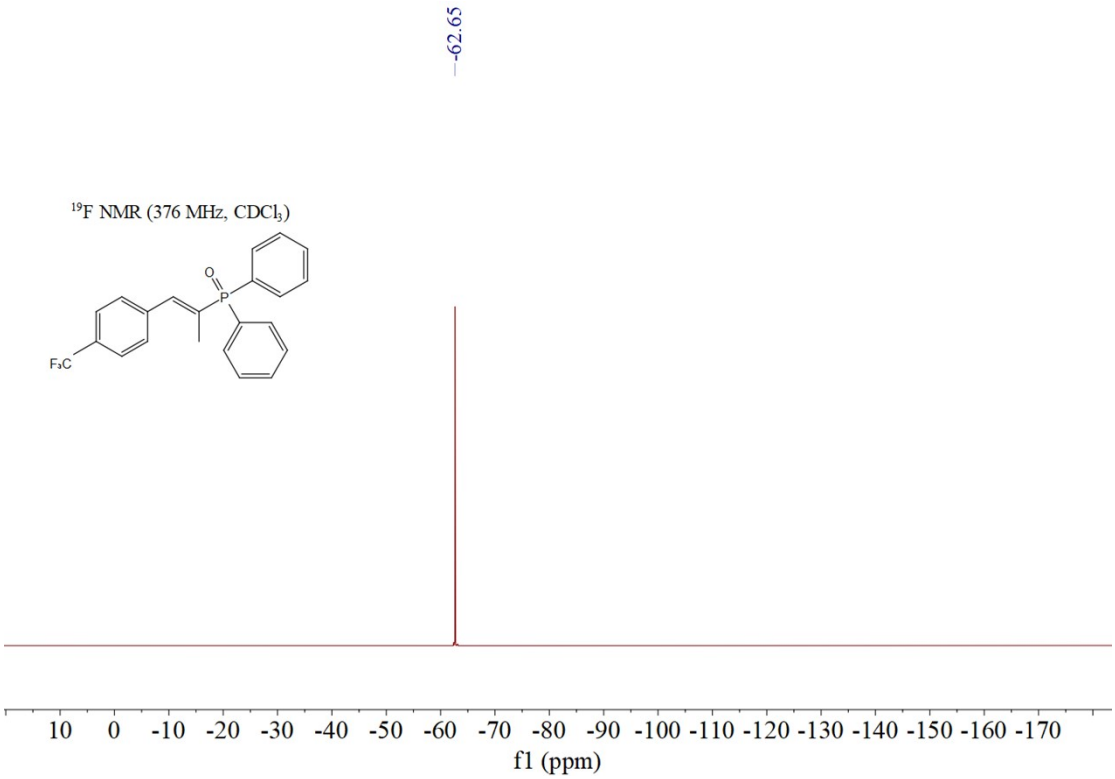


(*E*)-diphenyl(1-(4-(trifluoromethyl)phenyl)prop-1-en-2-yl)phosphine oxide (**3f**)

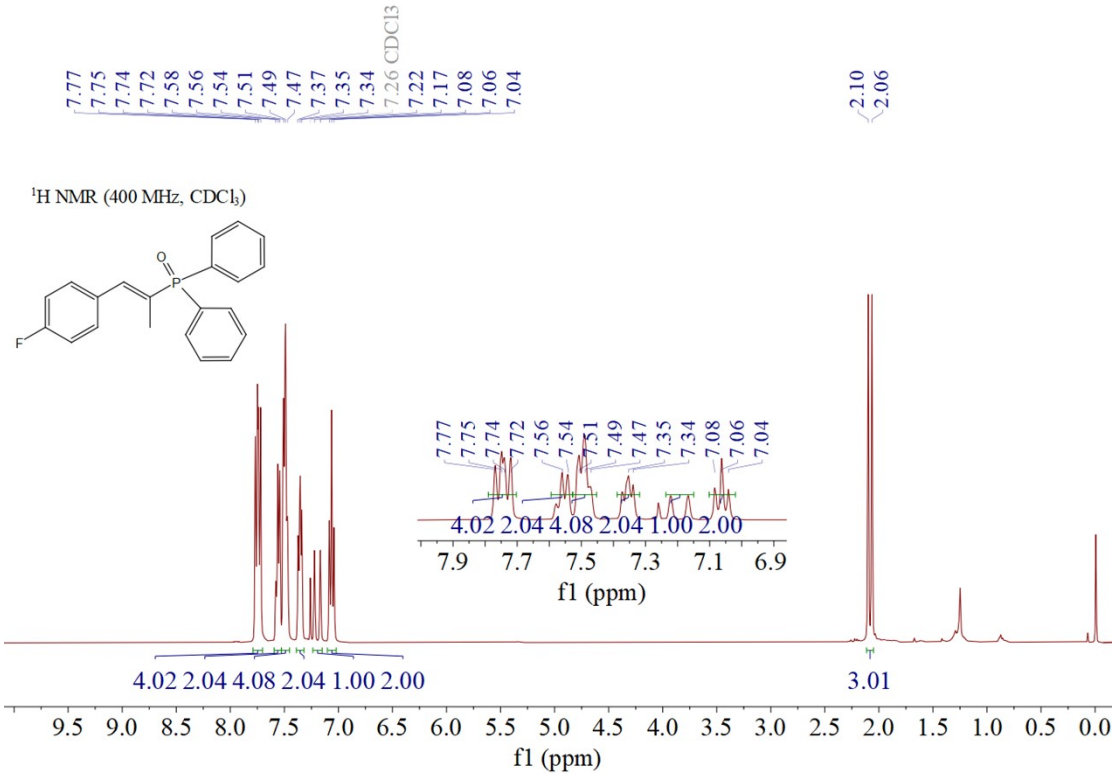


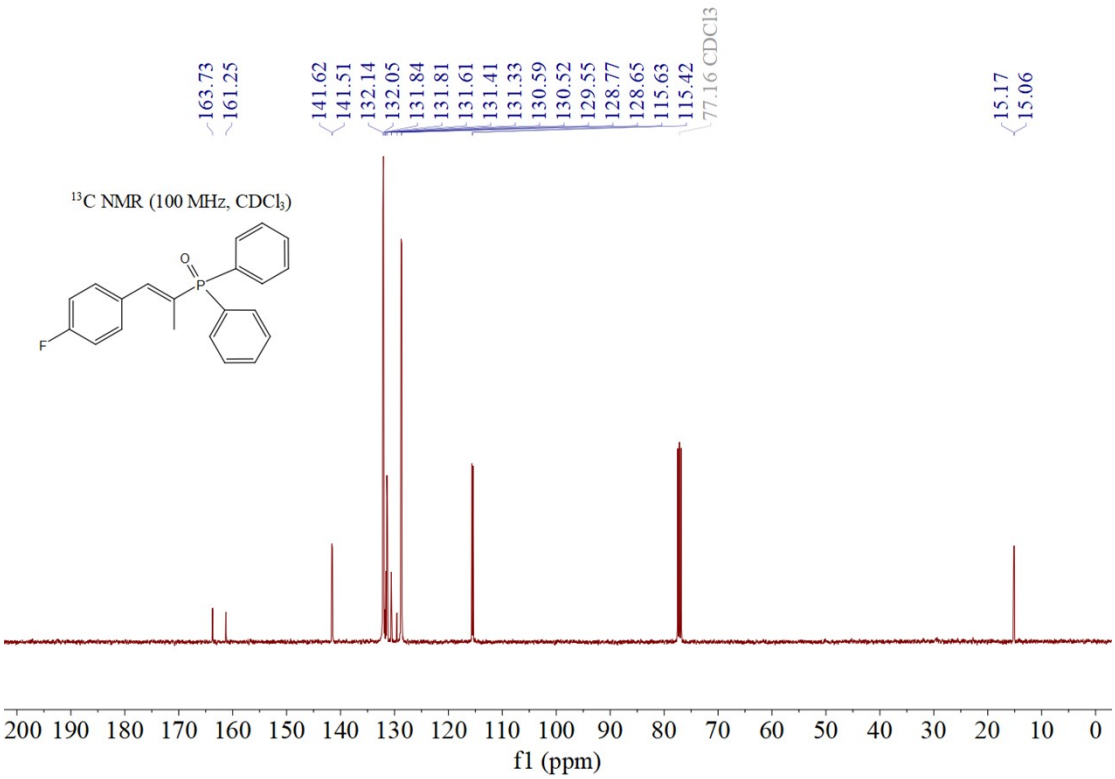

141.17
141.07
139.40
139.21
133.65
132.70
132.24
132.21
132.12
132.02
131.28
130.59
130.26
129.94
129.58
128.83
128.71
128.04
125.44
125.40
125.36
125.33
122.62
119.92
77.16 CDCl3
15.20
15.10
13C NMR (100 MHz, CDCl3)
O
P
F3C
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

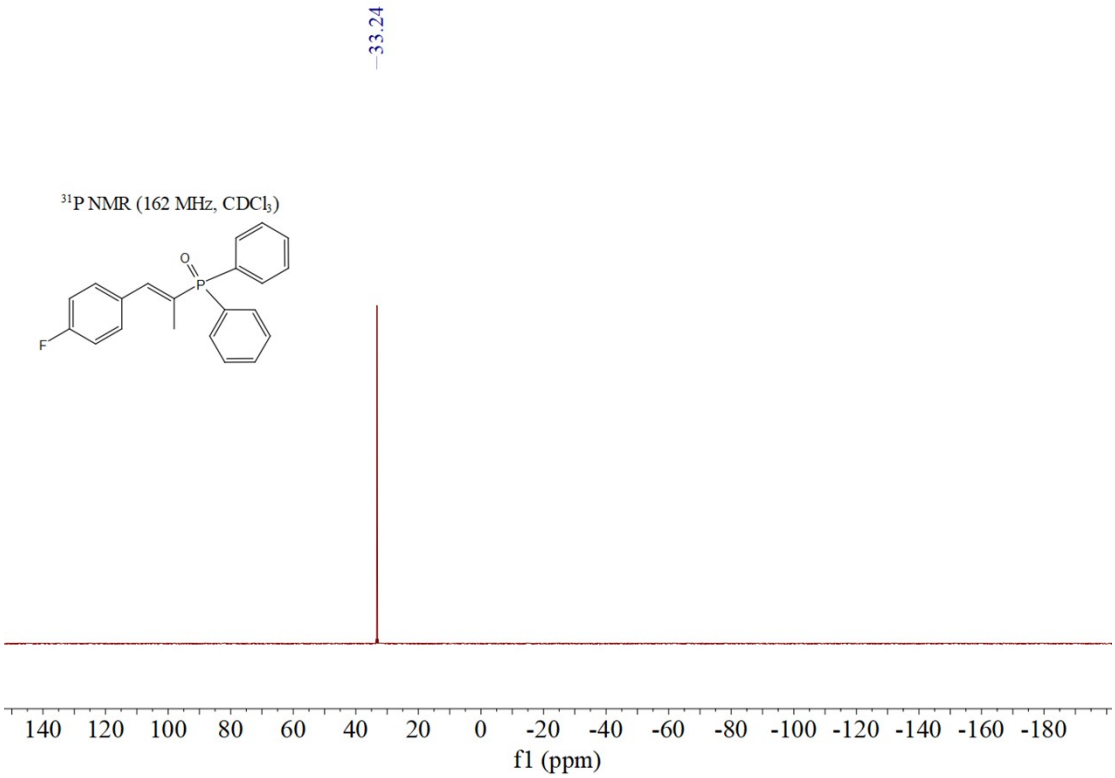

32.58
31P NMR (162 MHz, CDCl3)
O
P
F3C
150 130 110 90 70 50 30 10 -10 -30 -50 -70 -90 -110 -130 -150
f1 (ppm)

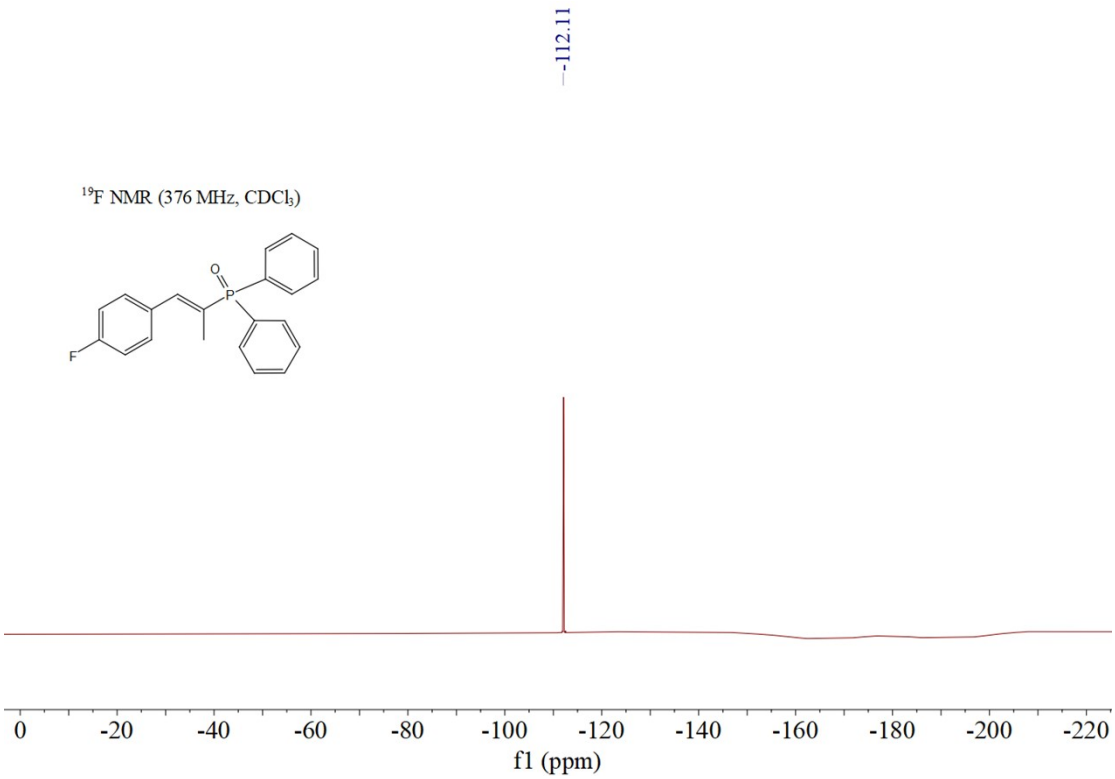


(*E*)-(1-(4-fluorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3g**)

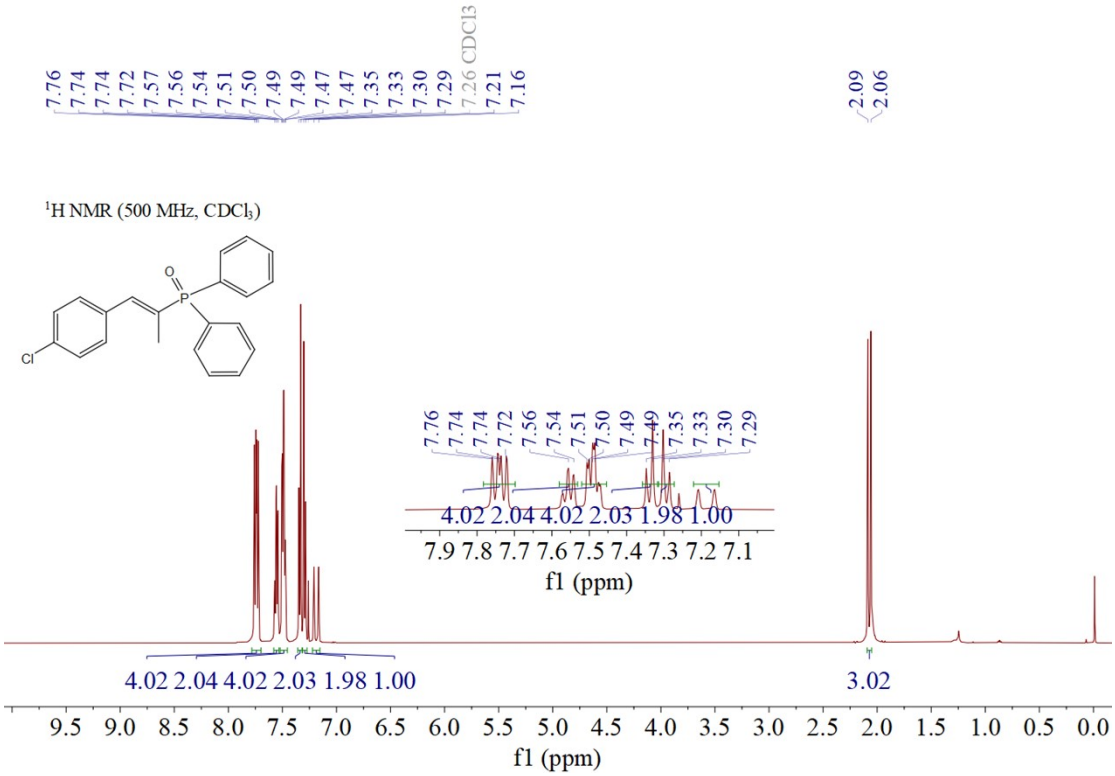


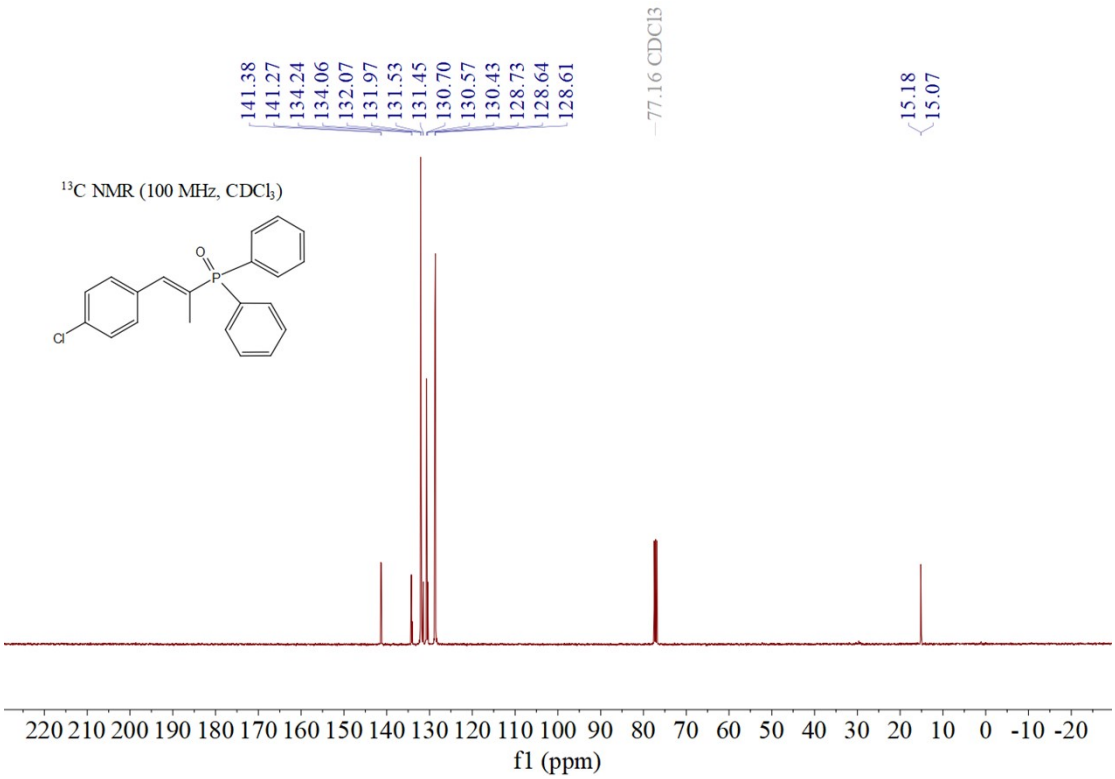

163.73
161.25
141.62
141.51
132.14
132.05
131.84
131.81
131.61
131.41
131.33
130.59
130.52
129.55
128.77
128.65
115.63
115.42
77.16 CDCl3
15.17
15.06
13C NMR (100 MHz, CDCl3)
O
P
F
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

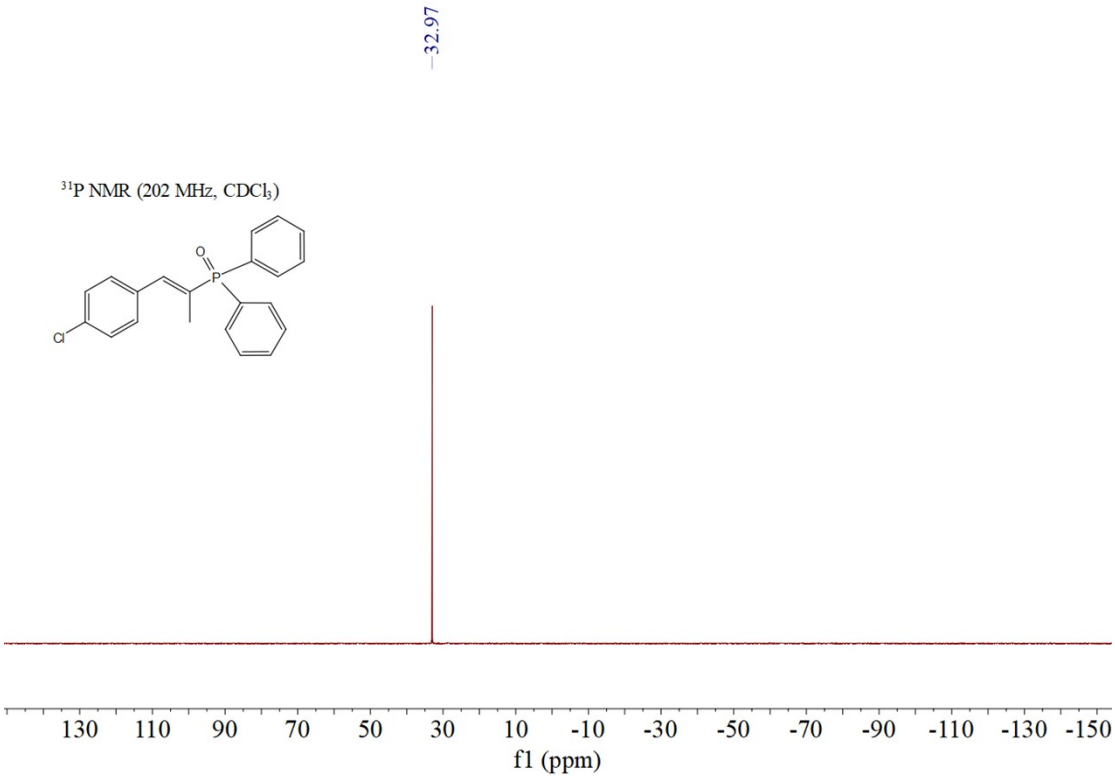

33.24
31P NMR (162 MHz, CDCl3)
O
P
F
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180
f1 (ppm)



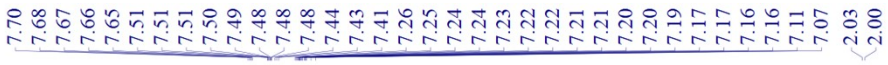
(*E*)-(1-(4-chlorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3h**)

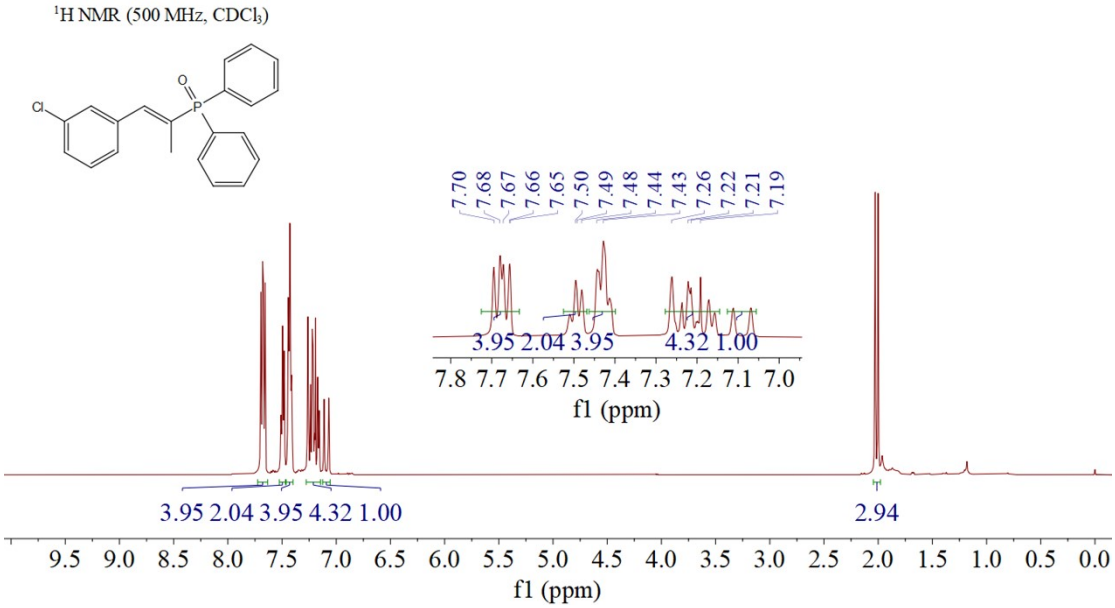


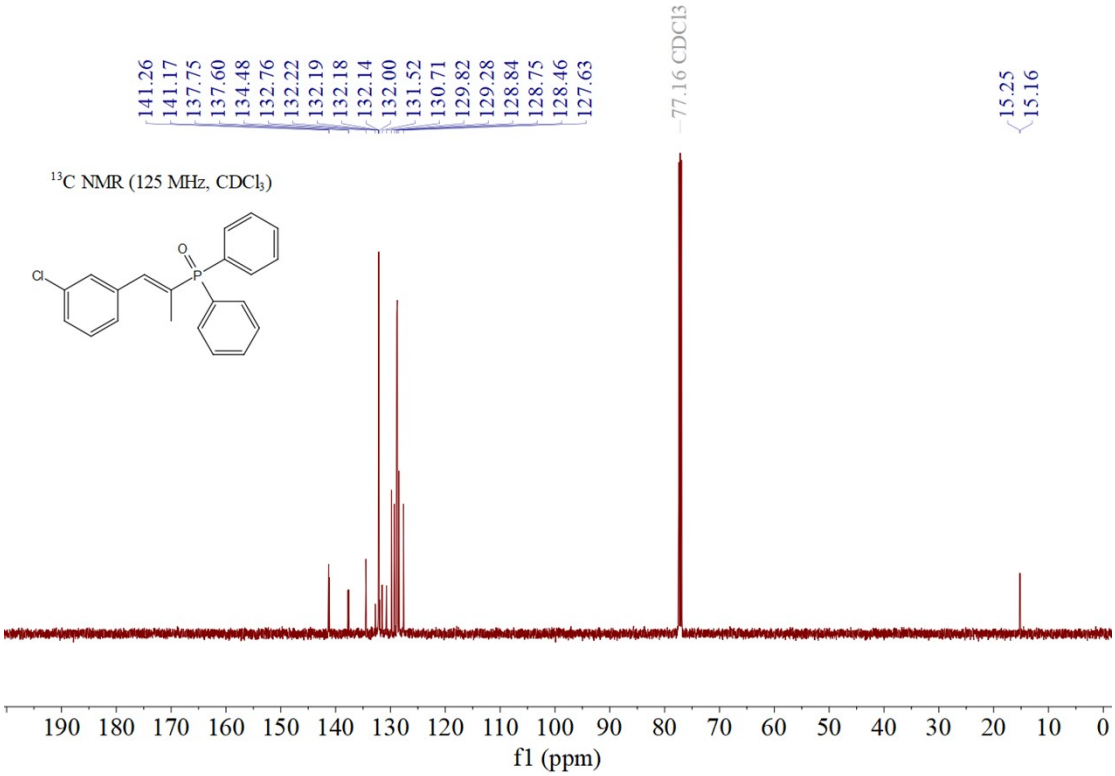


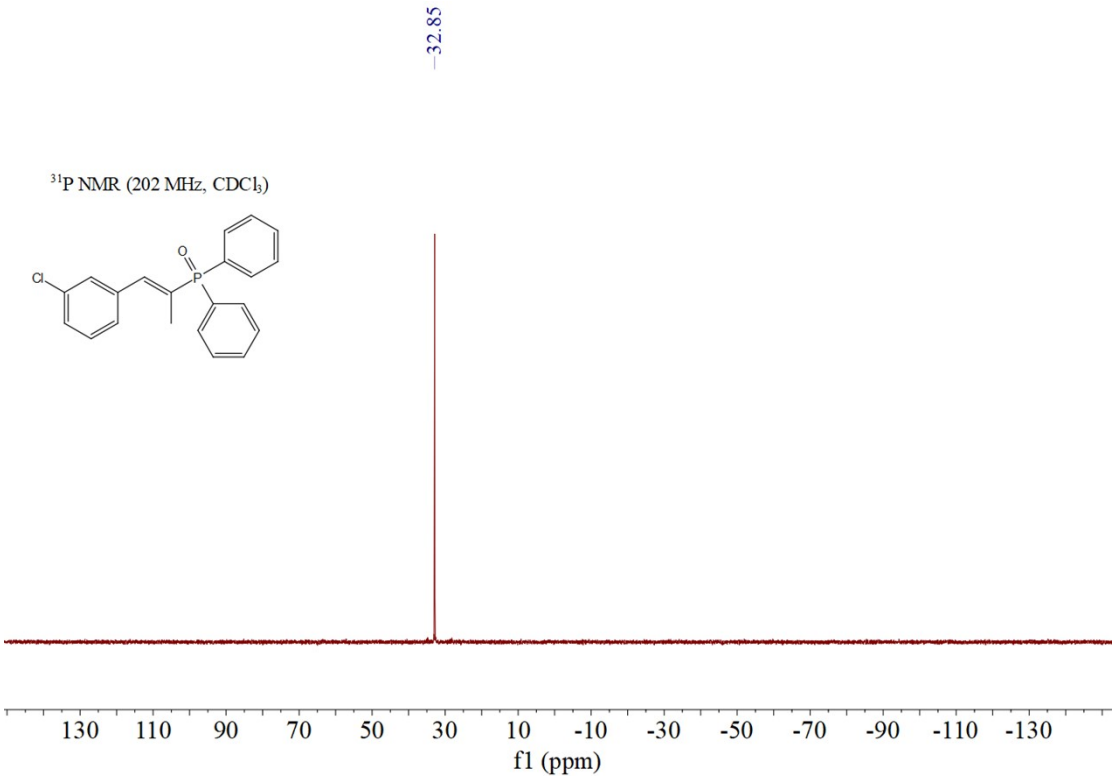


(*E*)-(1-(3-chlorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3i**)

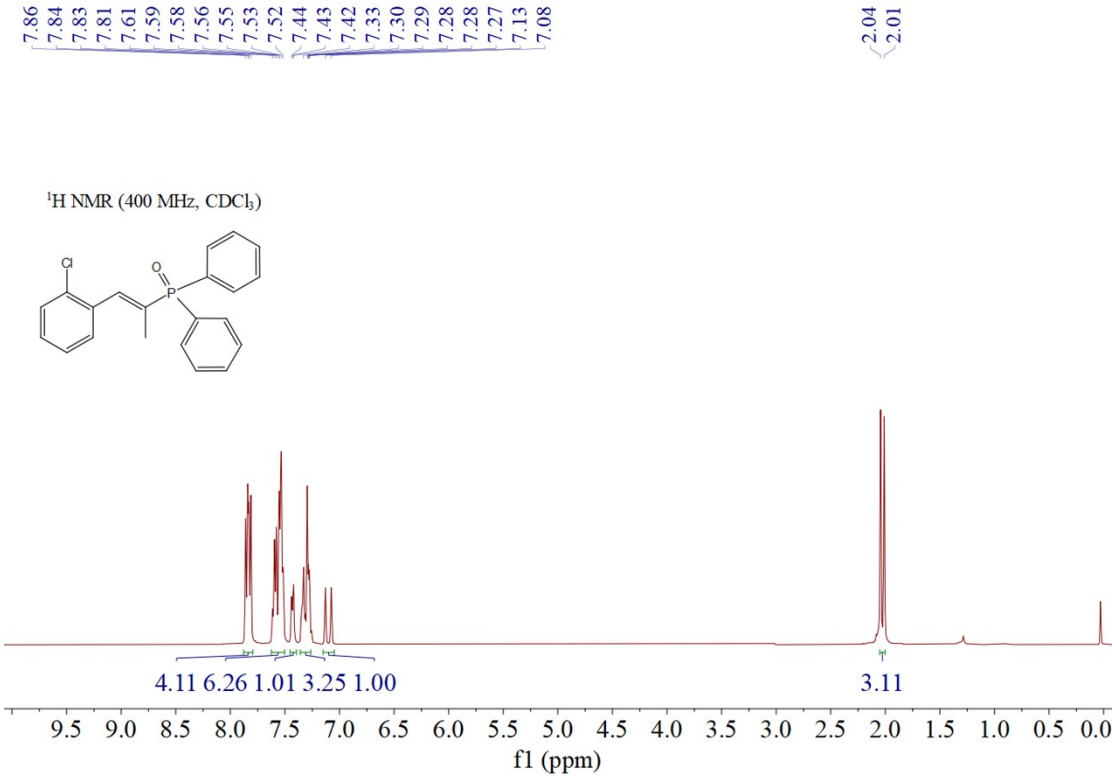

7.70
7.68
7.67
7.66
7.65
7.51
7.51
7.51
7.50
7.49
7.48
7.48
7.48
7.44
7.43
7.41
7.26
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.20
7.19
7.17
7.17
7.16
7.16
7.11
7.07
2.03
2.00

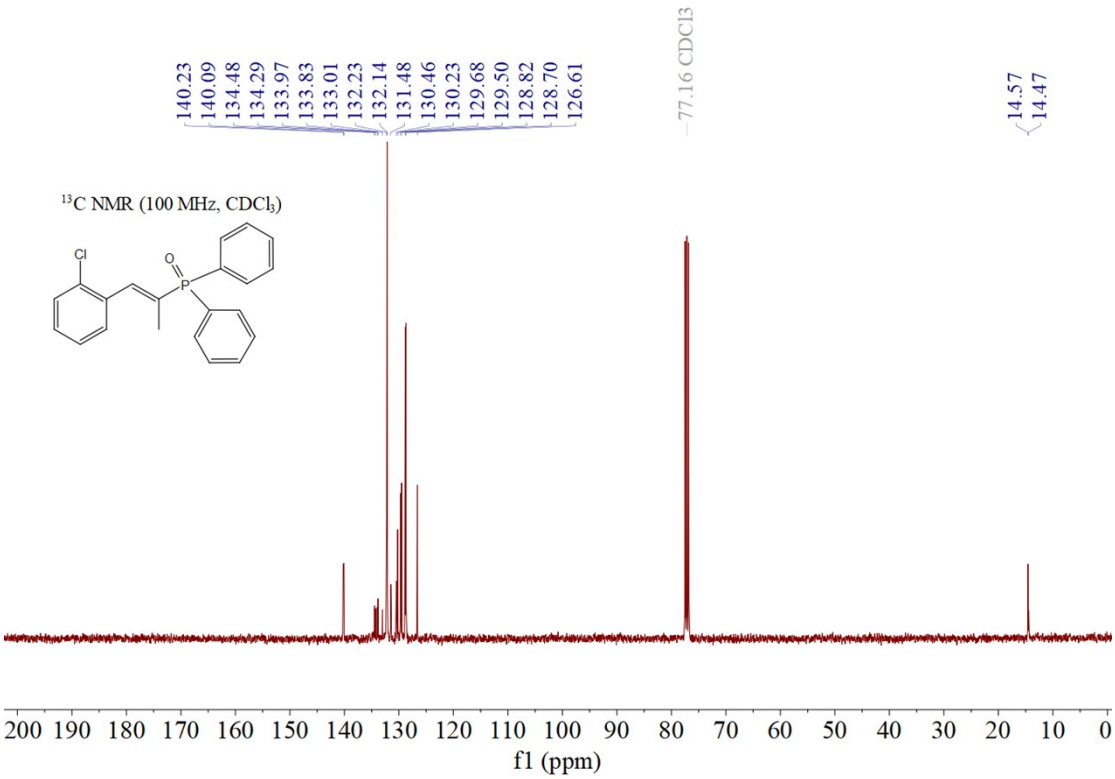

^{1}H NMR (500 MHz, $CDCl_3$)
Cl
O
P
7.70
7.68
7.67
7.66
7.65
7.50
7.49
7.48
7.44
7.43
7.26
7.22
7.21
7.19
3.95 2.04 3.95
4.32 1.00
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
3.95 2.04 3.95 4.32 1.00
2.94
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

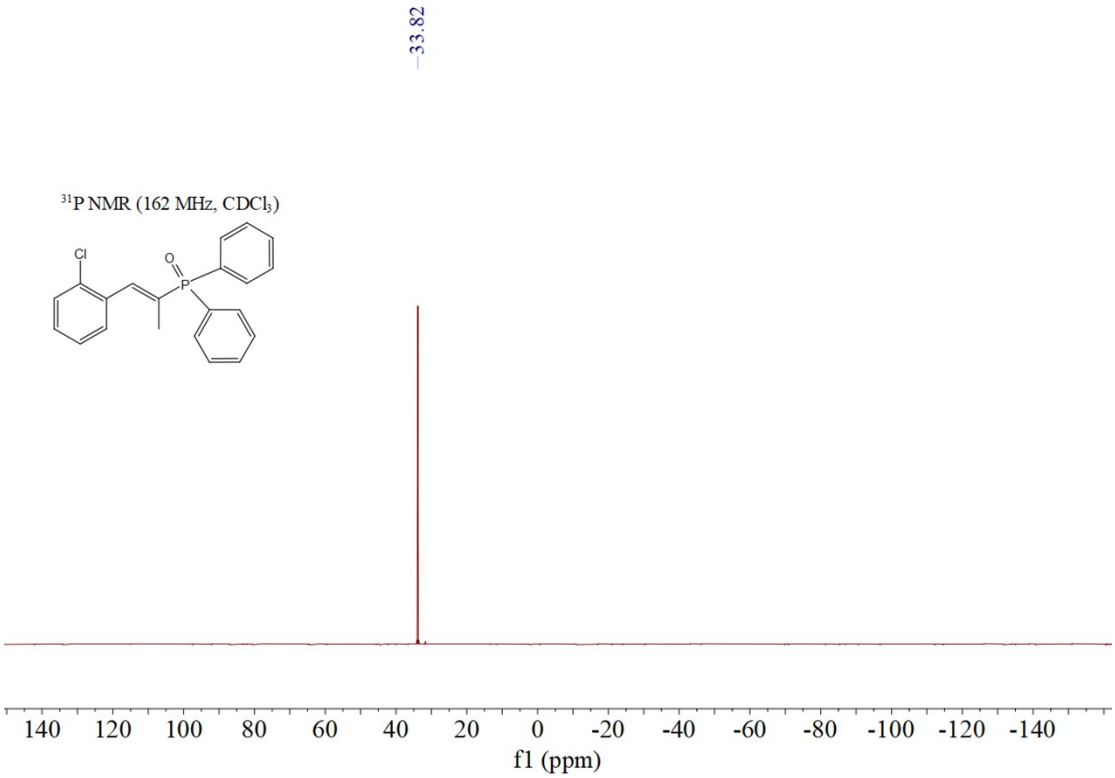

141.26
141.17
137.75
137.60
134.48
132.76
132.22
132.19
132.18
132.14
132.00
131.52
130.71
129.82
129.28
128.84
128.75
128.46
127.63
77.16 CDCl3
15.25
15.16
^{13}C NMR (125 MHz, $CDCl_3$)
Cl
O
P
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



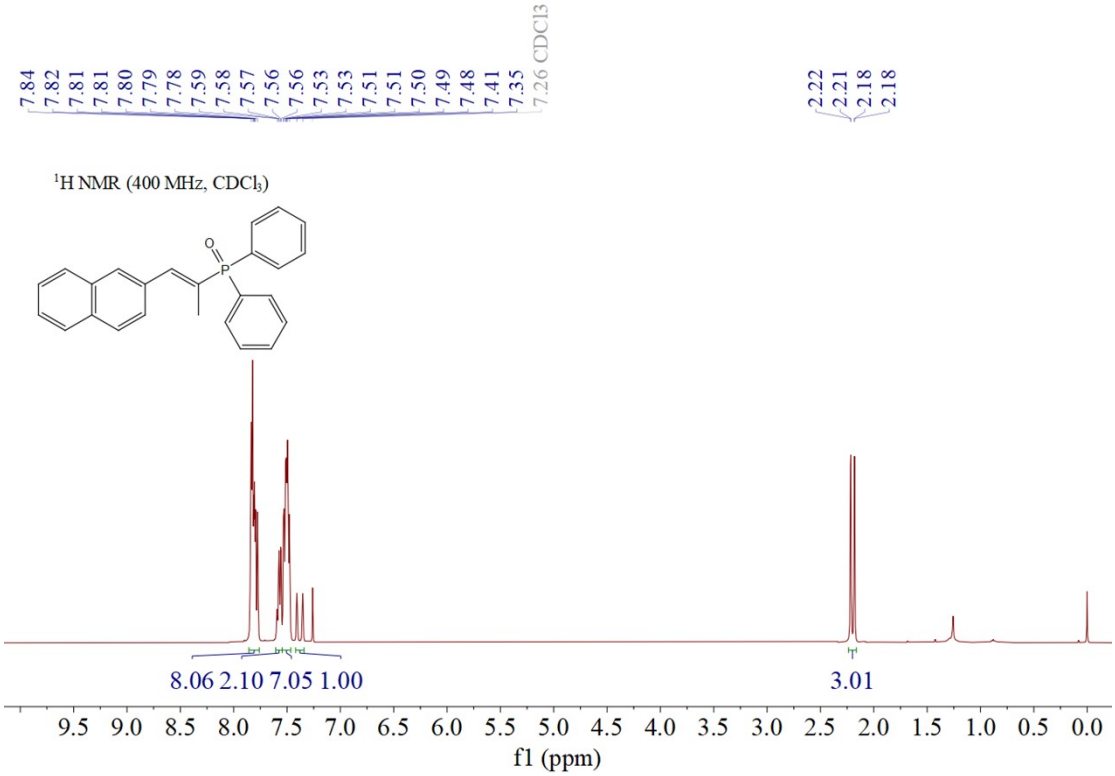
(*E*)-(1-(2-chlorophenyl)prop-1-en-2-yl)diphenylphosphine oxide (**3j**)

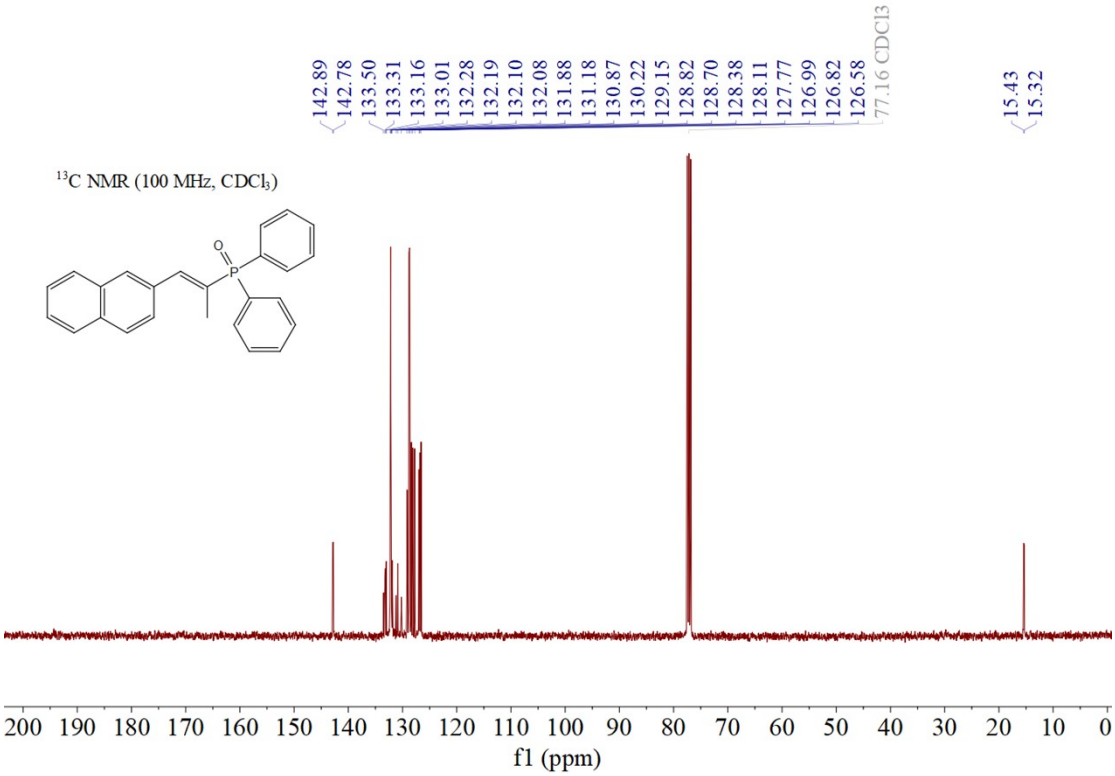


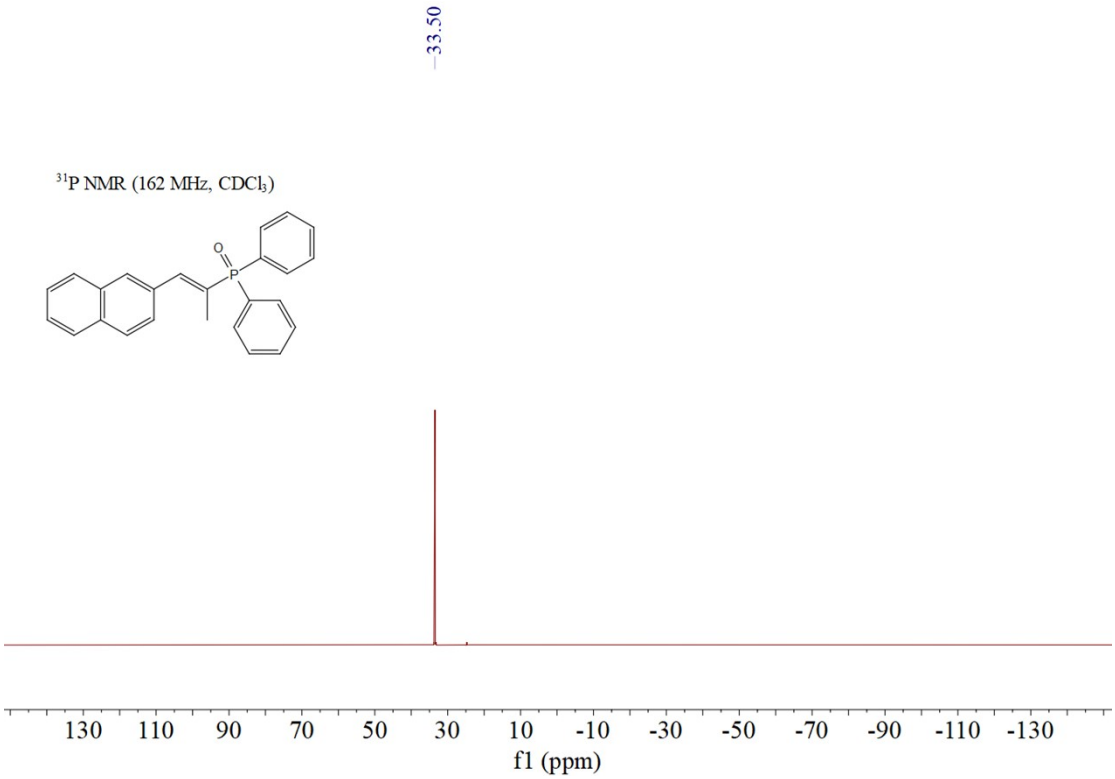




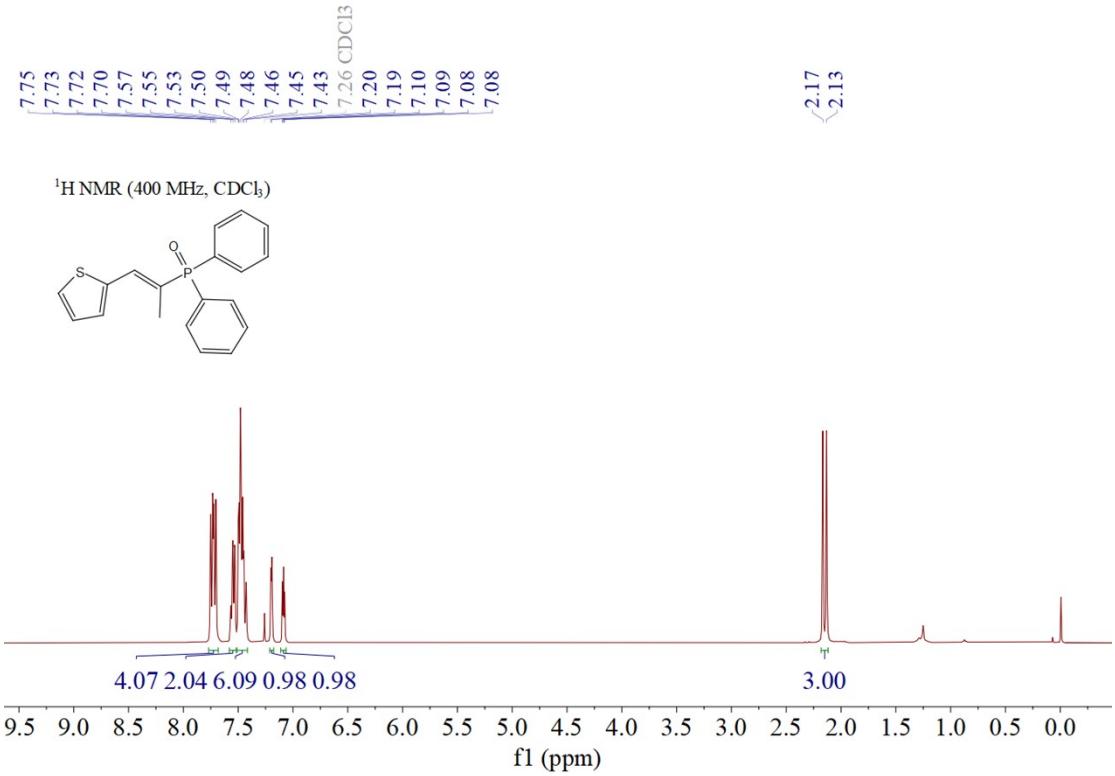
(*E*)-(1-(naphthalen-2-yl)prop-1-en-2-yl)diphenylphosphine oxide (**3k**)

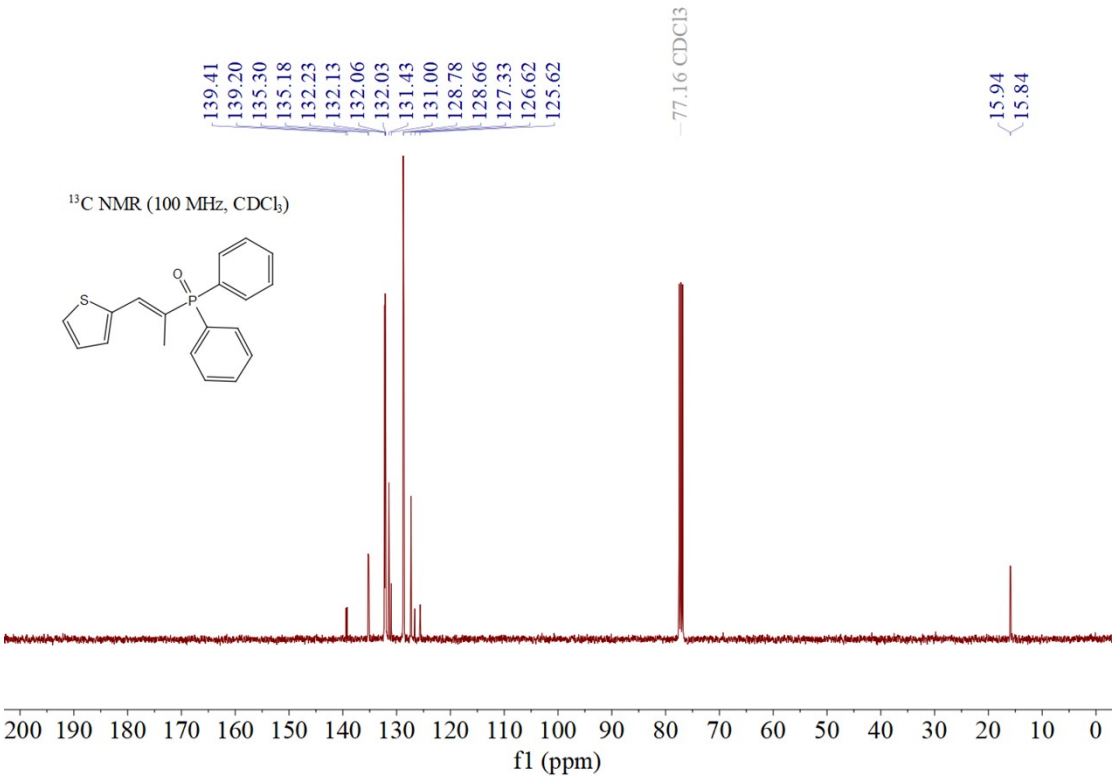

7.84
7.82
7.81
7.81
7.80
7.79
7.78
7.59
7.58
7.57
7.56
7.56
7.53
7.53
7.51
7.51
7.50
7.49
7.48
7.41
7.35
7.26 CDCl3
2.22
2.21
2.18
2.18
1H NMR (400 MHz, CDCl3)
O
P
8.06 2.10 7.05 1.00
3.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

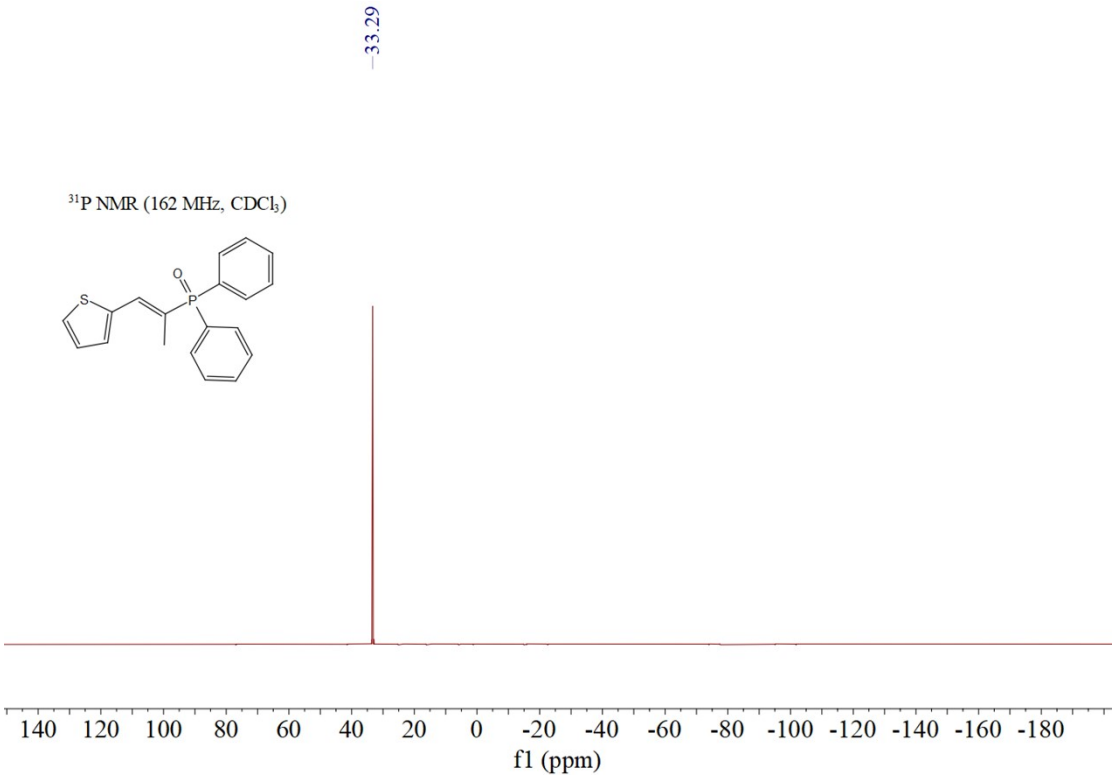

142.89
142.78
133.50
133.31
133.16
133.01
132.28
132.19
132.10
132.08
131.88
131.18
130.87
130.22
129.15
128.82
128.70
128.38
128.11
127.77
126.99
126.82
126.58
77.16 CDCl3
15.43
15.32
13C NMR (100 MHz, CDCl3)
O
P
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



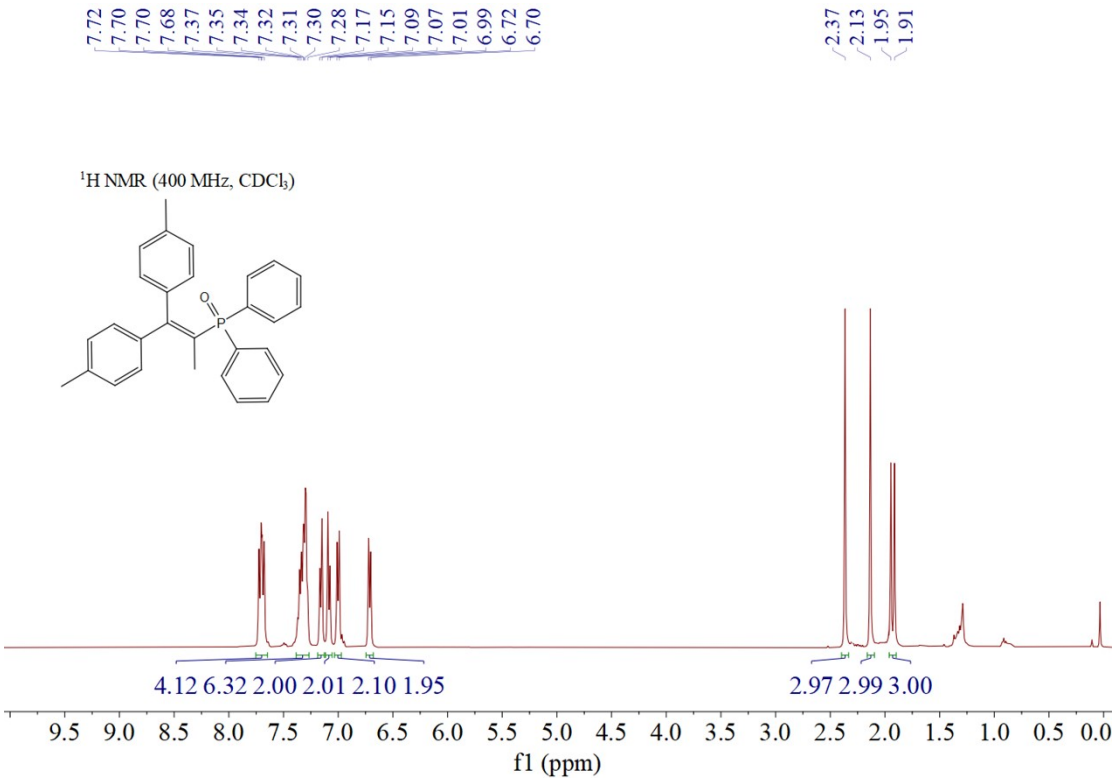
(*E*)-diphenyl(1-(thiophen-2-yl)prop-1-en-2-yl)phosphine oxide (**3l**)

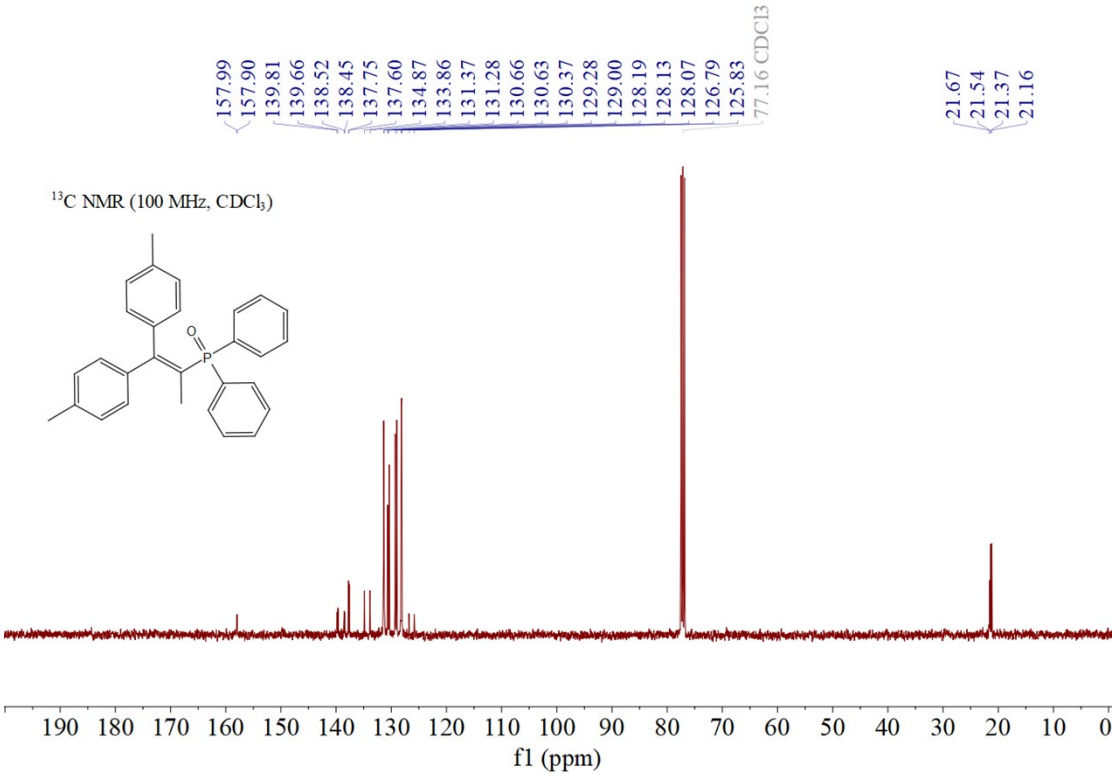


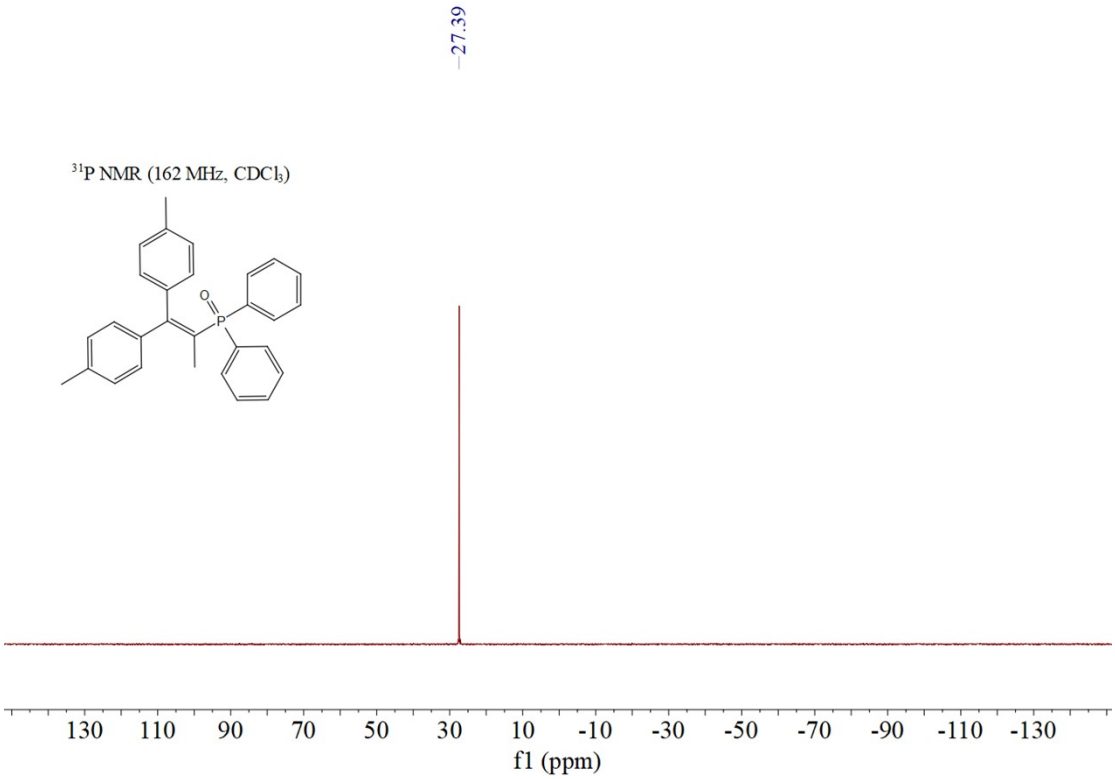




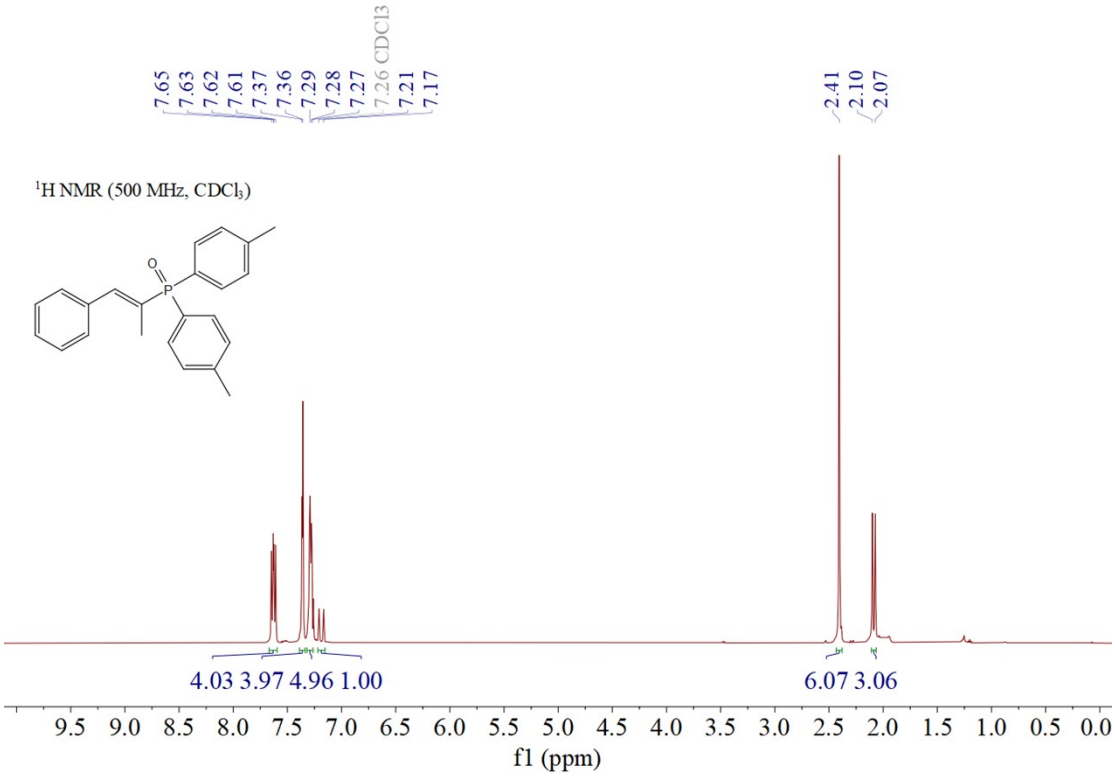
(1,1-di-p-tolylprop-1-en-2-yl)diphenylphosphine oxide (**3m**)

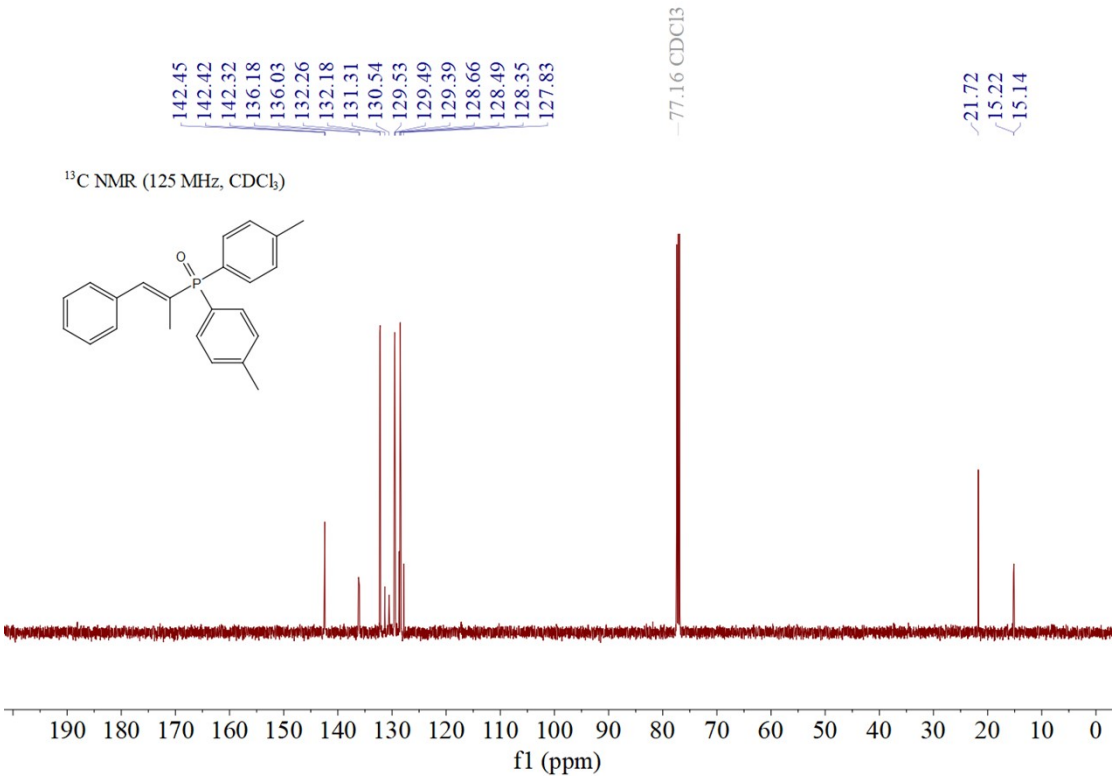

7.72
7.70
7.70
7.68
7.37
7.35
7.34
7.32
7.31
7.30
7.28
7.17
7.15
7.09
7.07
7.01
6.99
6.72
6.70
2.37
2.13
1.95
1.91
^{1}H NMR (400 MHz, $CDCl_3$)
O
P
4.12 6.32 2.00 2.01 2.10 1.95
2.97 2.99 3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

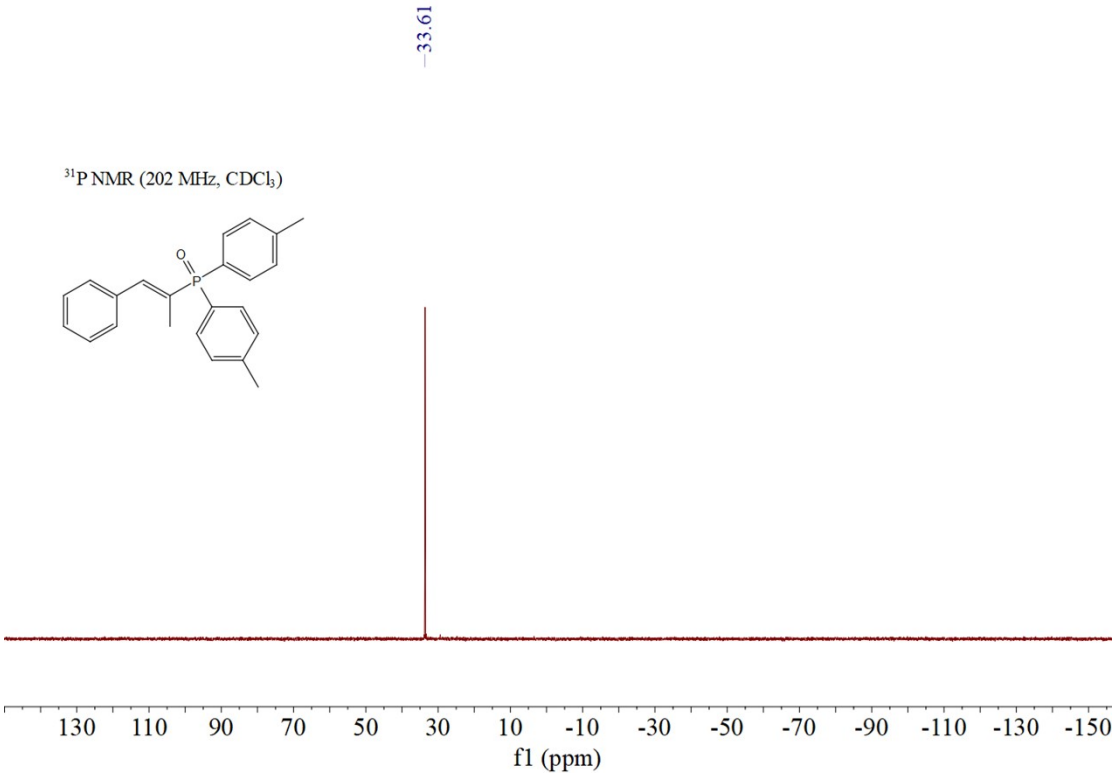

157.99
157.90
139.81
139.66
138.52
138.45
137.75
137.60
134.87
133.86
131.37
131.28
130.66
130.63
130.37
129.28
129.00
128.19
128.13
128.07
126.79
125.83
77.16 CDCl3
21.67
21.54
21.37
21.16
^{13}C NMR (100 MHz, $CDCl_3$)
O
P
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



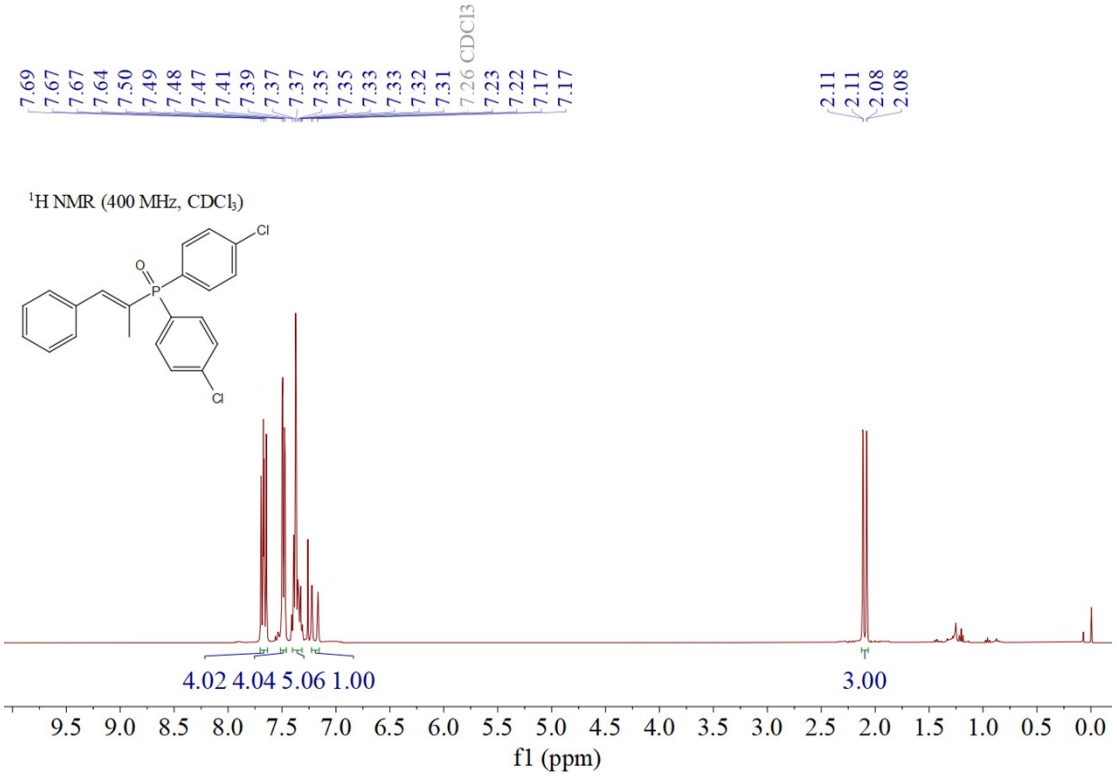
(*E*)-(1-phenylprop-1-en-2-yl)di-p-tolylphosphine oxide (**3n**)

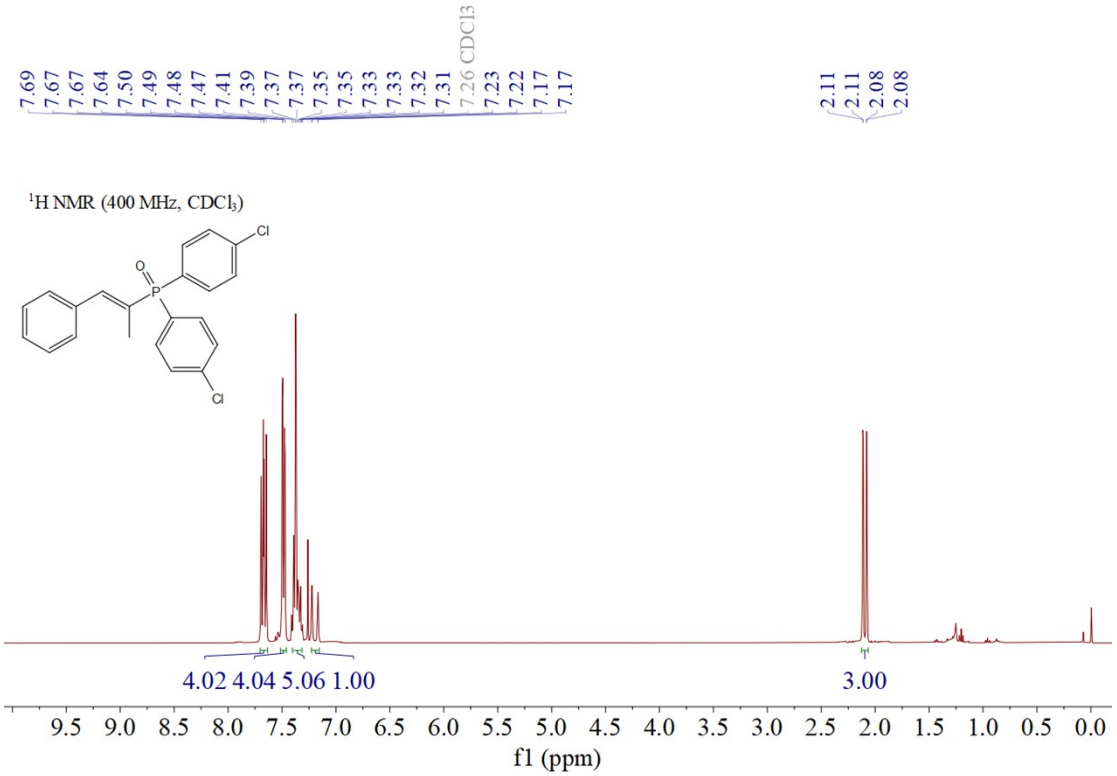


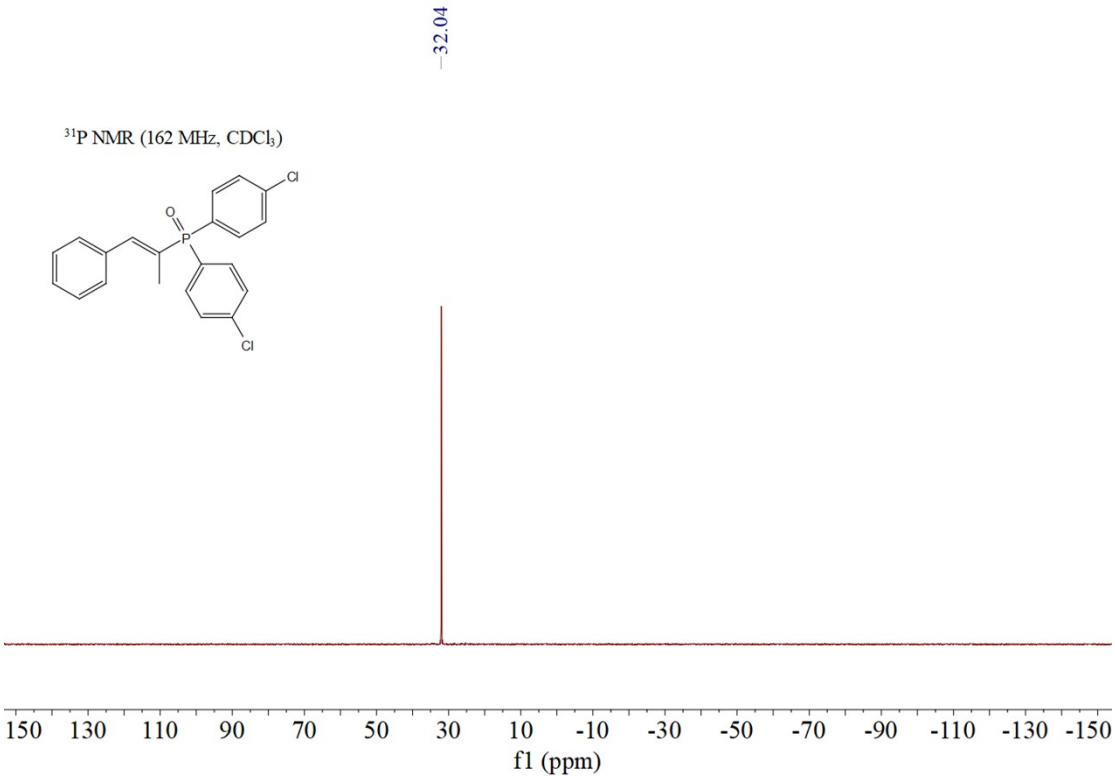




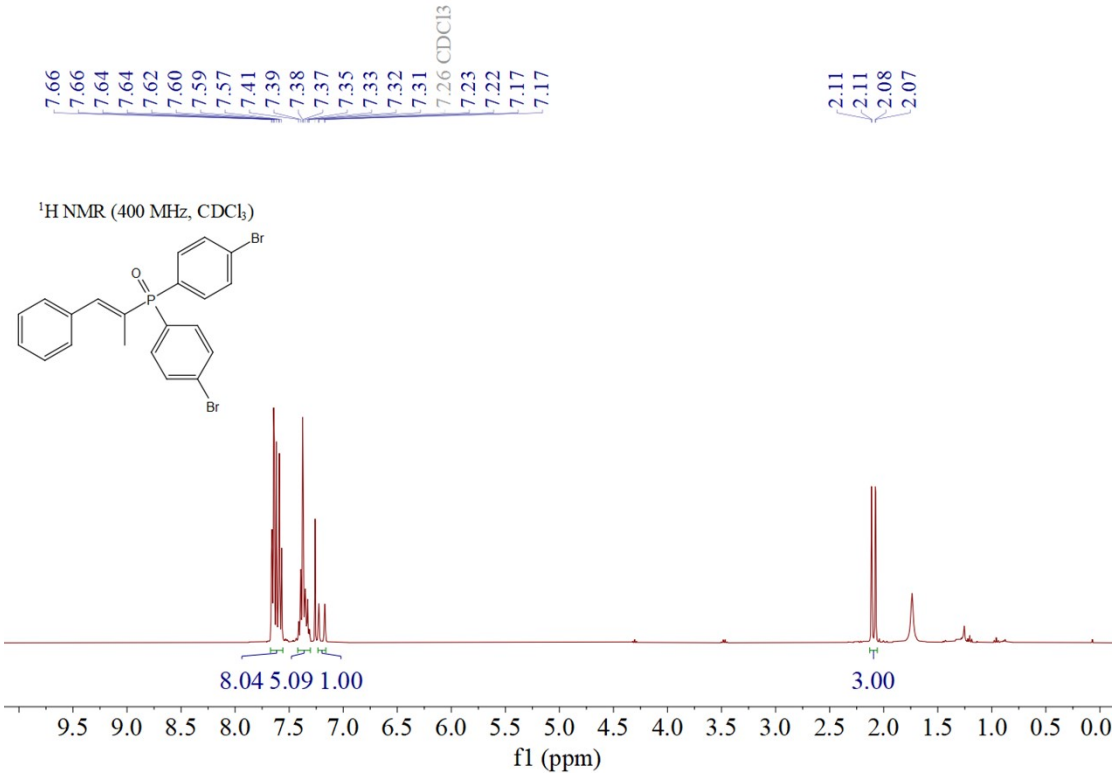
(*E*)-bis(4-chlorophenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3o**)

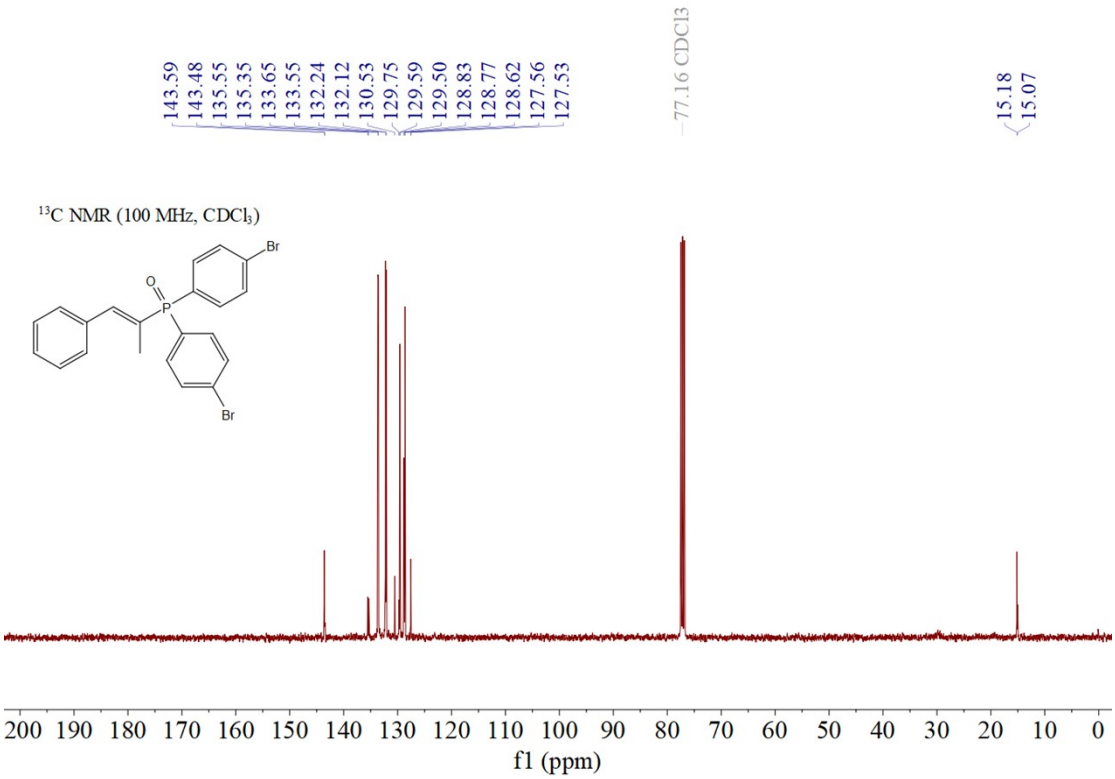

7.69 7.67 7.67 7.64 7.50 7.49 7.48 7.47 7.41 7.39 7.37 7.37 7.35 7.35 7.33 7.33 7.32 7.31 7.26 CDCl3 7.23 7.22 7.17 7.17
2.11 2.11 2.08 2.08
1H NMR (400 MHz, CDCl3)
Cl
O
P
Cl
4.02 4.04 5.06 1.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

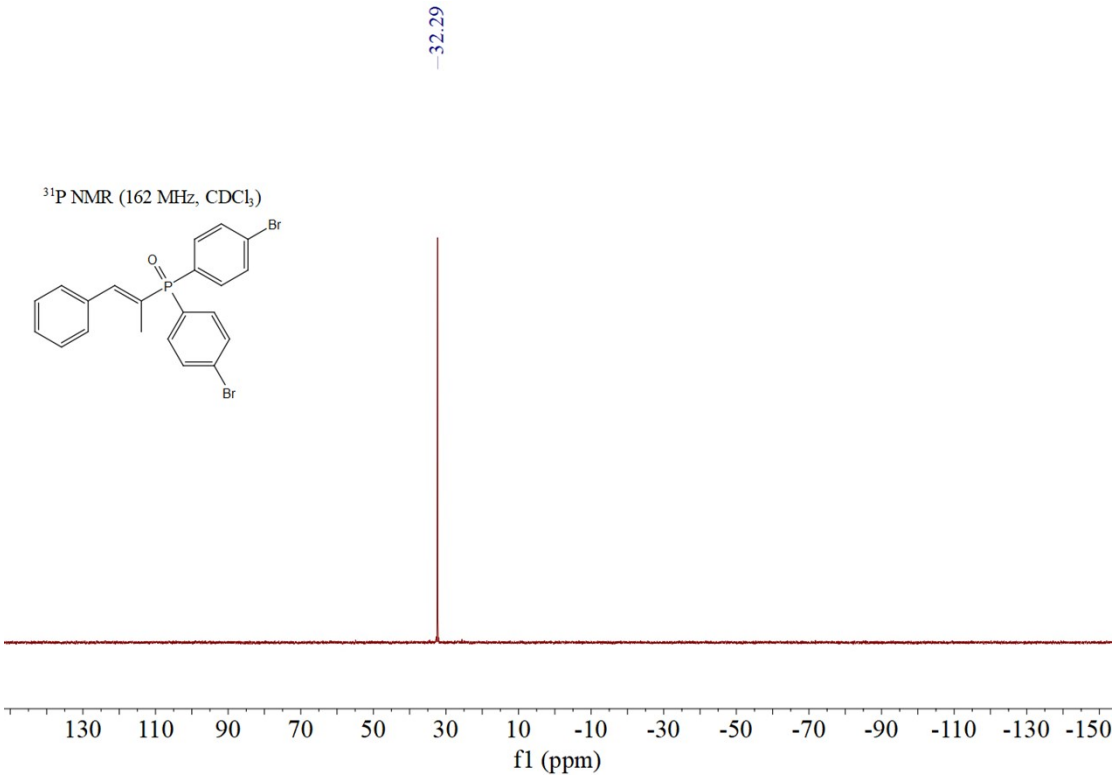

7.69 7.67 7.67 7.64 7.50 7.49 7.48 7.47 7.41 7.39 7.37 7.37 7.35 7.35 7.33 7.33 7.32 7.31 7.26 CDCl3 7.23 7.22 7.17 7.17
2.11 2.11 2.08 2.08
1H NMR (400 MHz, CDCl3)
Cl
O
P
Cl
4.02 4.04 5.06 1.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



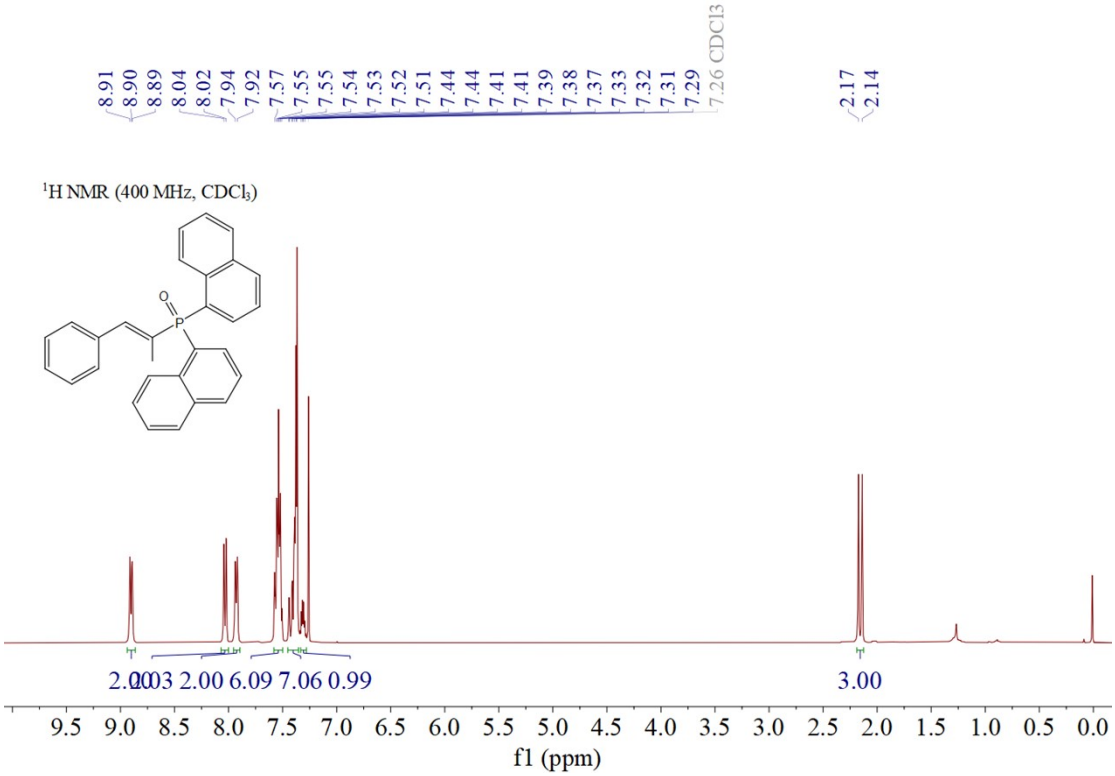
(*E*)-bis(4-bromophenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3p**)

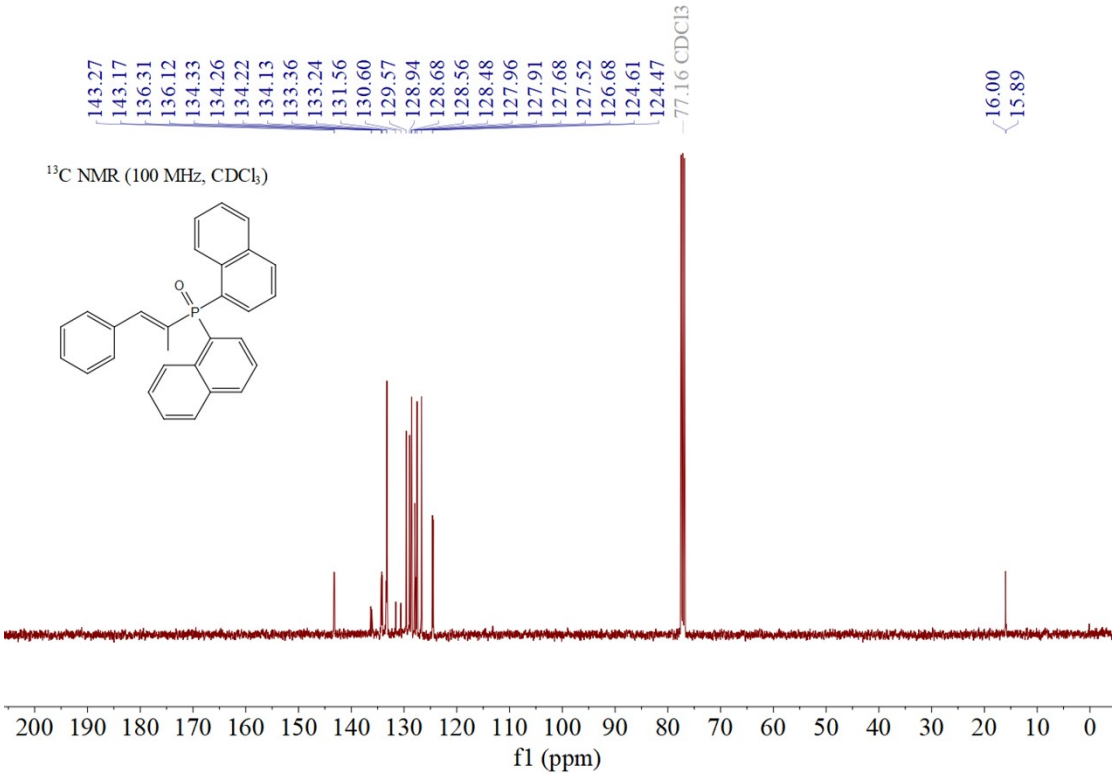


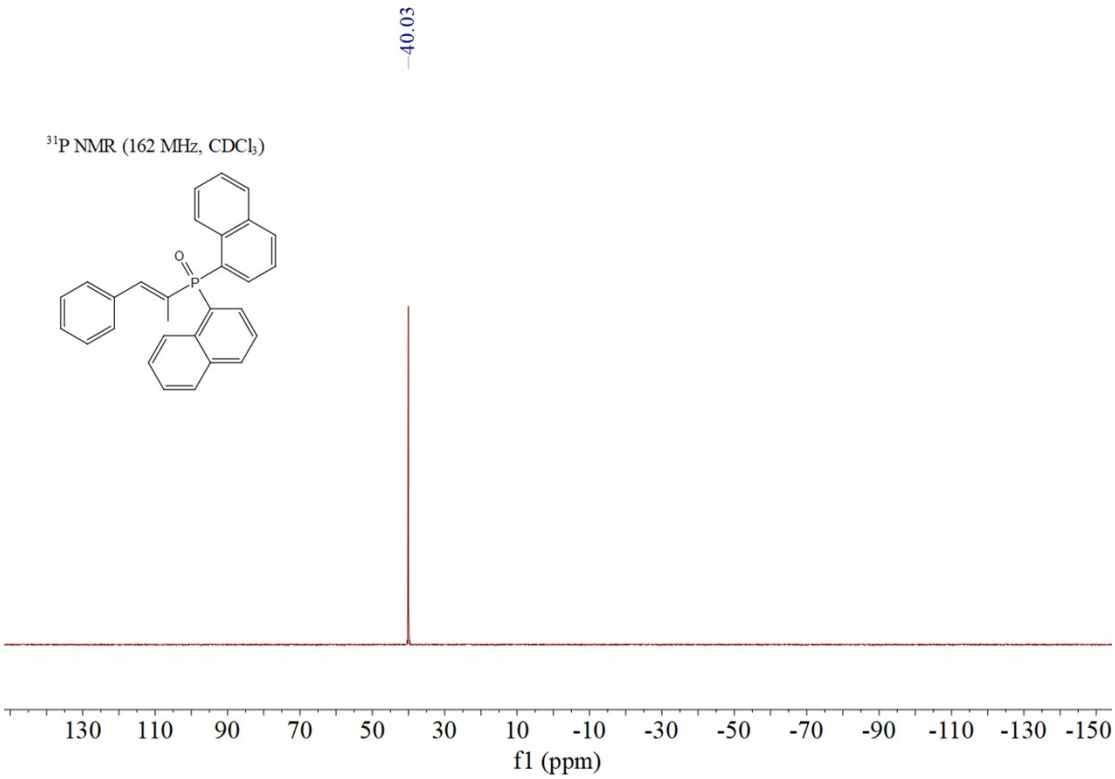




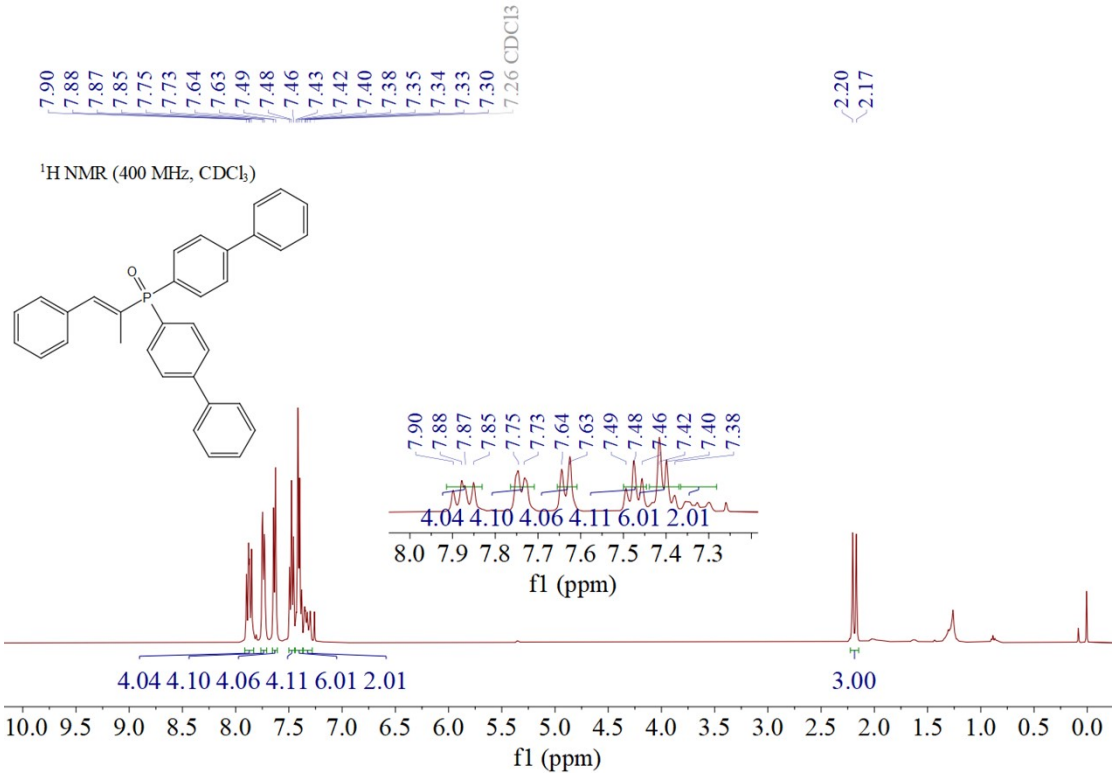
(*E*)-di(naphthalen-1-yl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3q**)

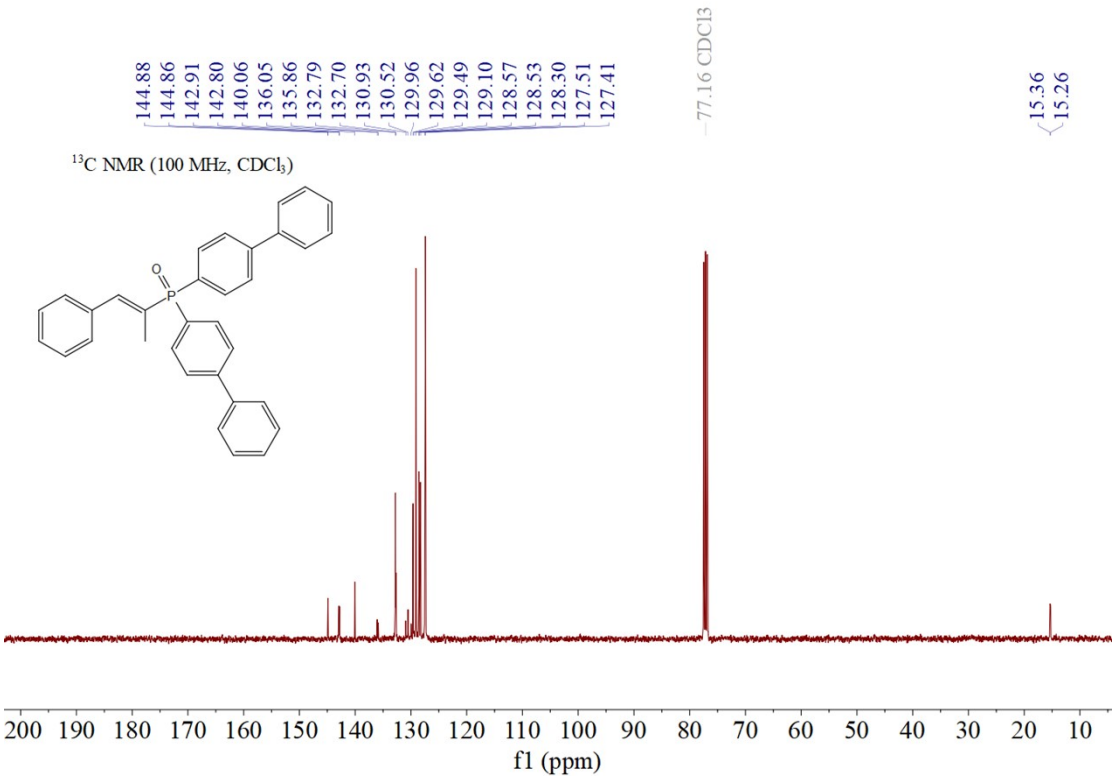

8.91
8.90
8.89
8.04
8.02
7.94
7.92
7.57
7.55
7.55
7.54
7.53
7.52
7.51
7.44
7.44
7.41
7.41
7.39
7.38
7.37
7.33
7.32
7.31
7.29
7.26 CDCl3
2.17
2.14
1H NMR (400 MHz, CDCl3)
2.00 2.00 2.00 6.09 7.06 0.99
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

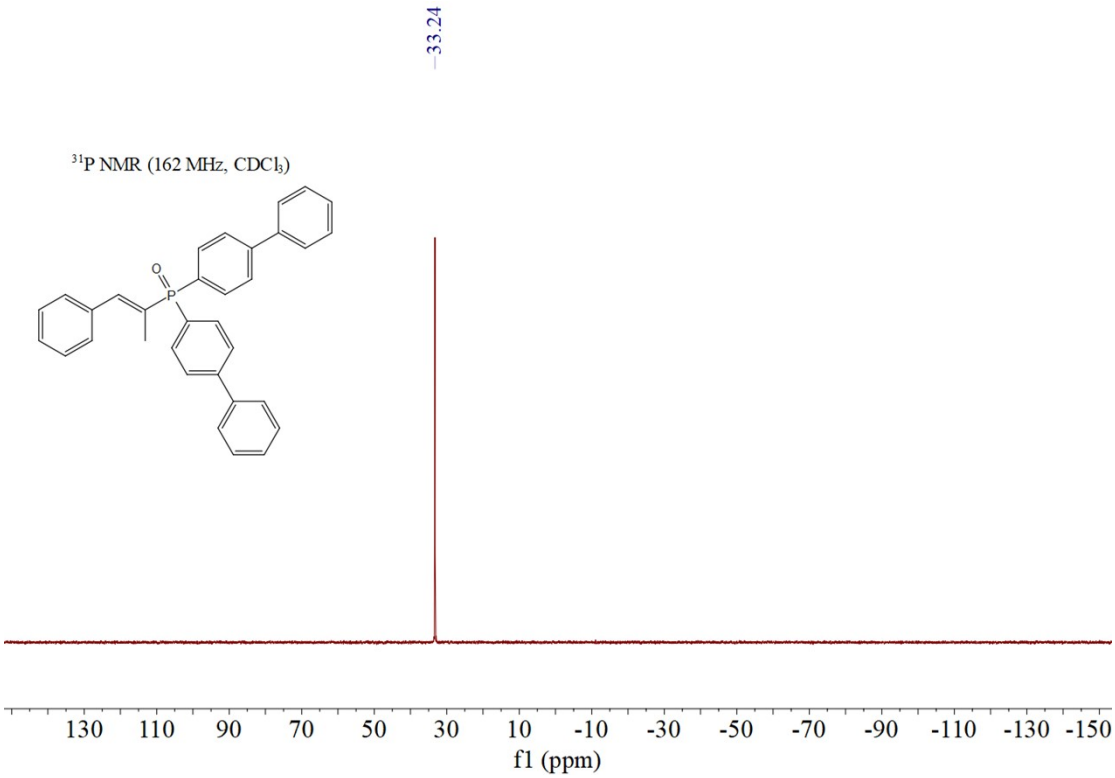

143.27
143.17
136.31
136.12
134.33
134.26
134.22
134.13
133.36
133.24
131.56
130.60
129.57
128.94
128.68
128.56
128.48
127.96
127.91
127.68
127.52
126.68
124.61
124.47
77.16 CDCl3
16.00
15.89
13C NMR (100 MHz, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



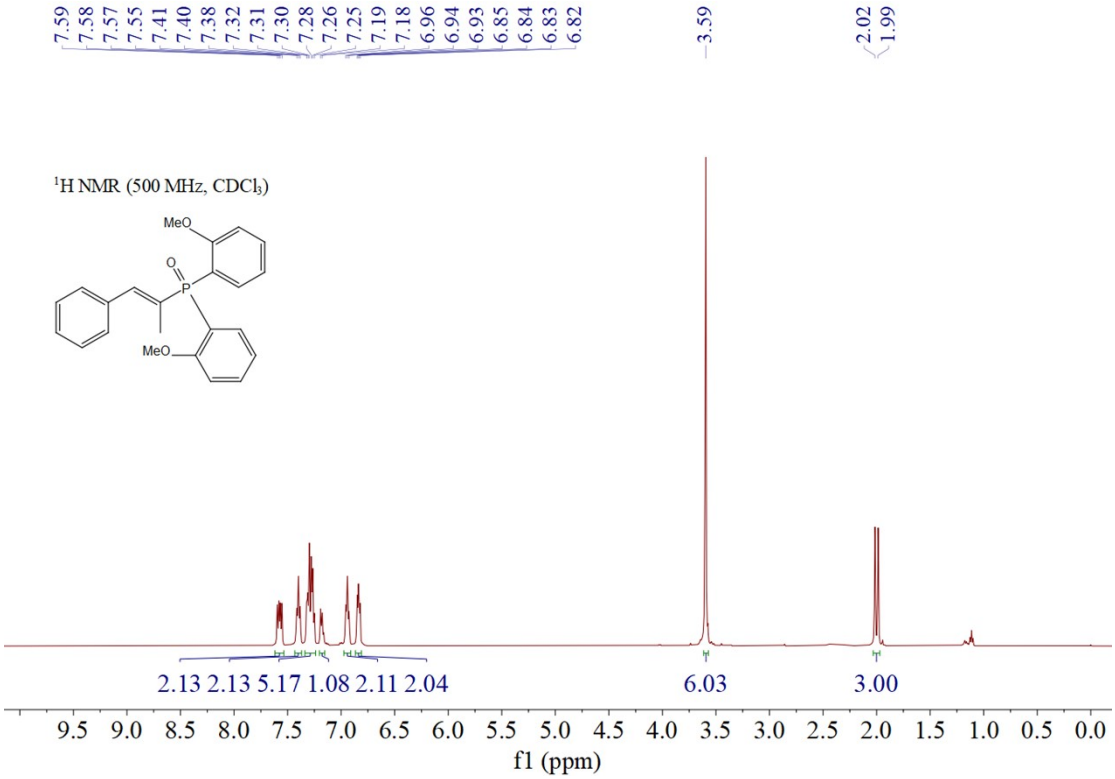
(*E*)-di([1,1'-biphenyl]-4-yl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3r**)

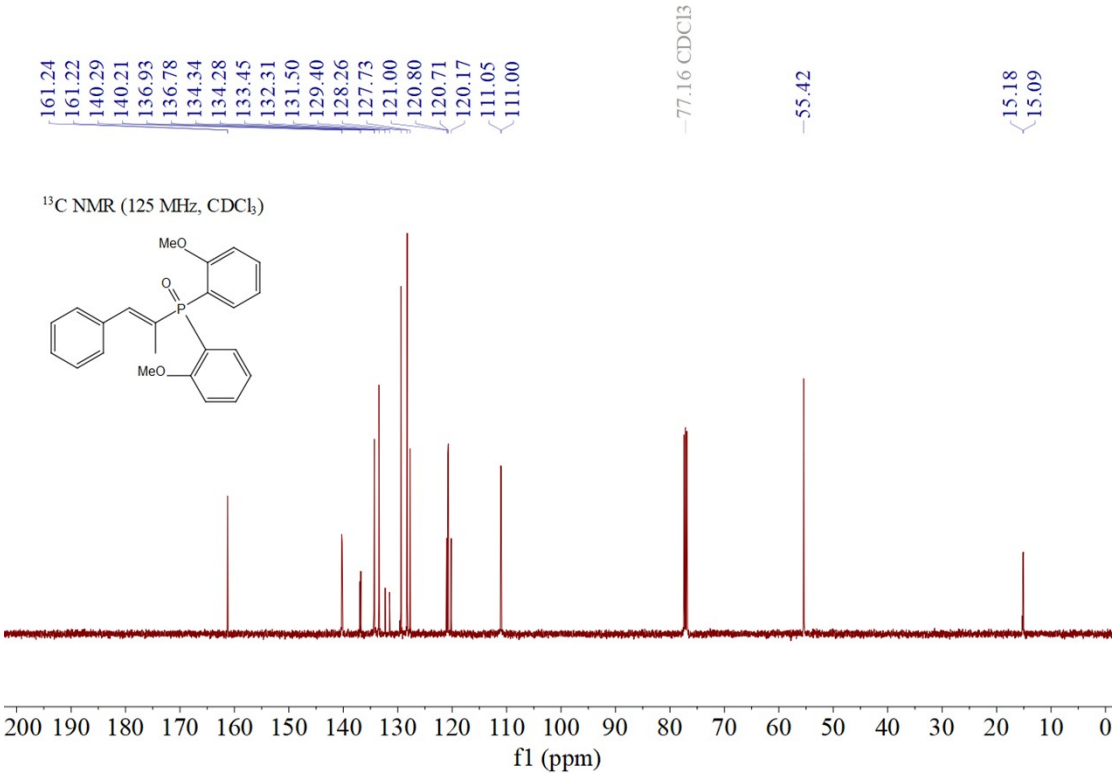


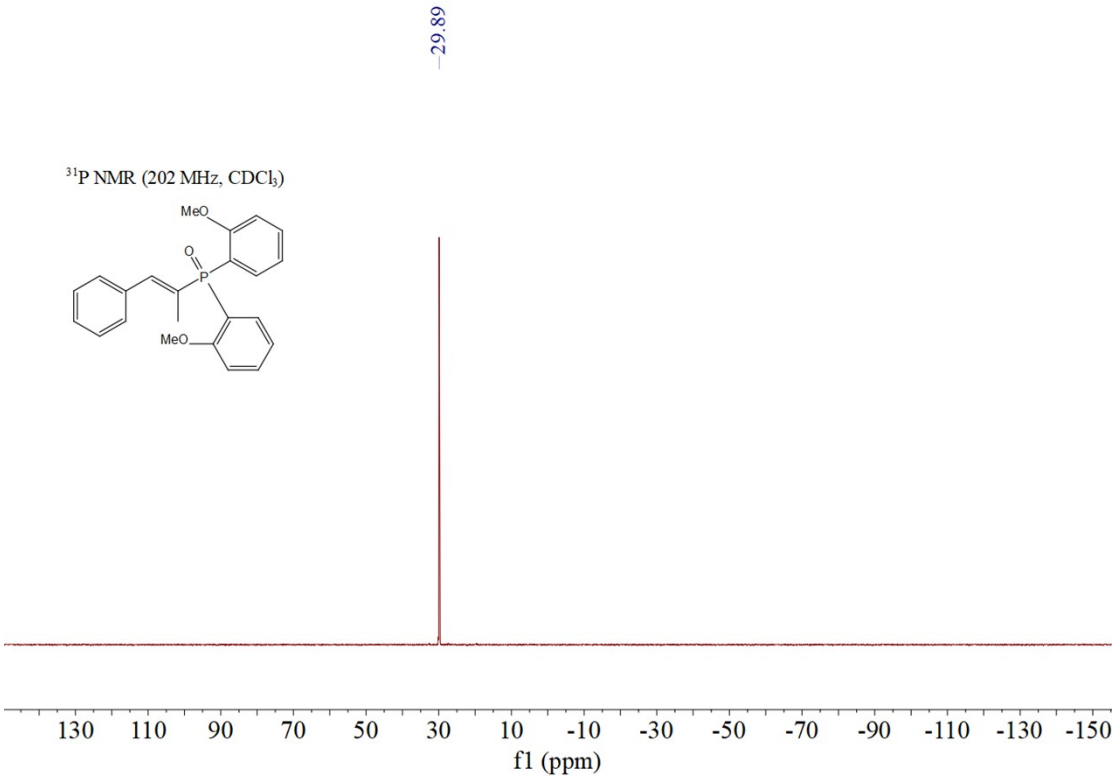




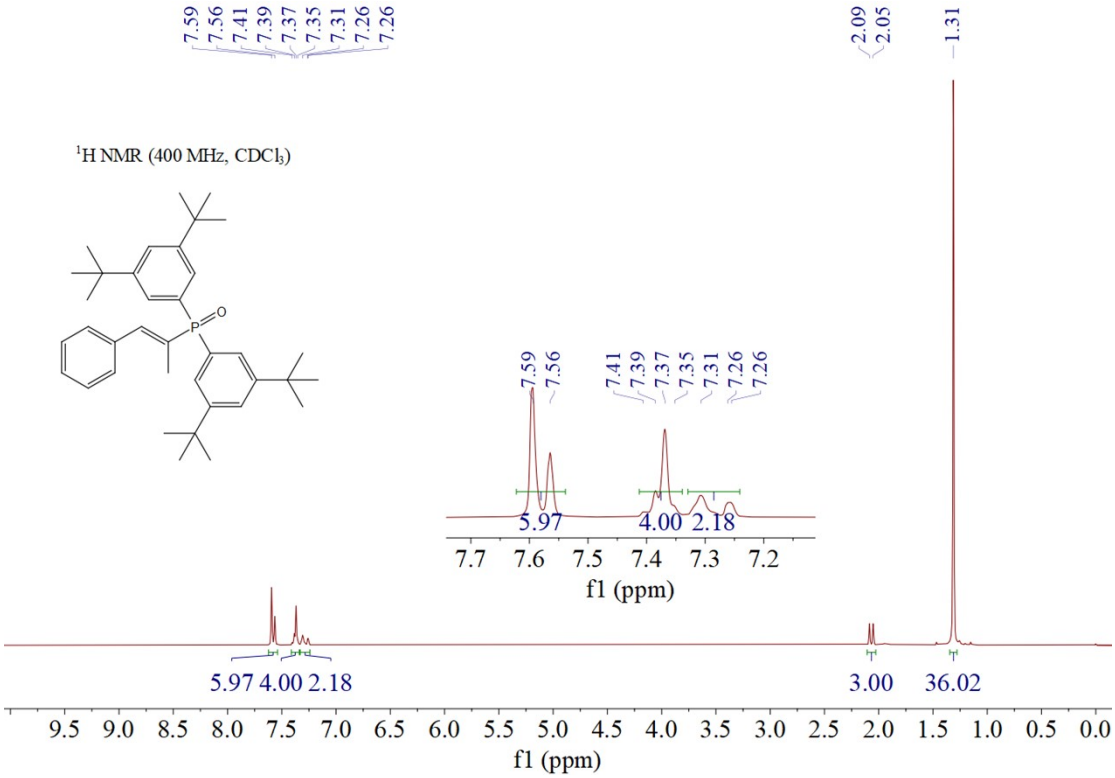
(*E*)-bis(2-methoxyphenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3s**)

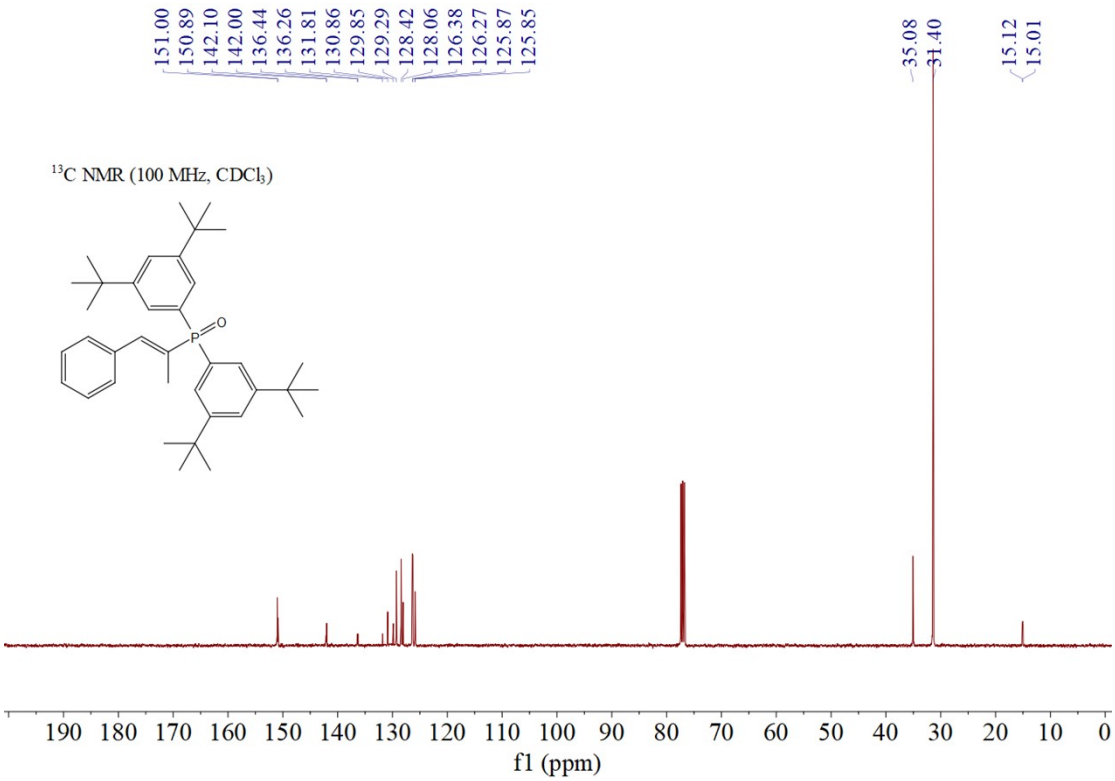

7.59
7.58
7.57
7.55
7.41
7.40
7.38
7.32
7.31
7.30
7.28
7.26
7.25
7.19
7.18
6.96
6.94
6.93
6.85
6.84
6.83
6.82
3.59
2.02
1.99
1H NMR (500 MHz, CDCl3)
MeO
O
P
MeO
2.13 2.13 5.17 1.08 2.11 2.04
6.03
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

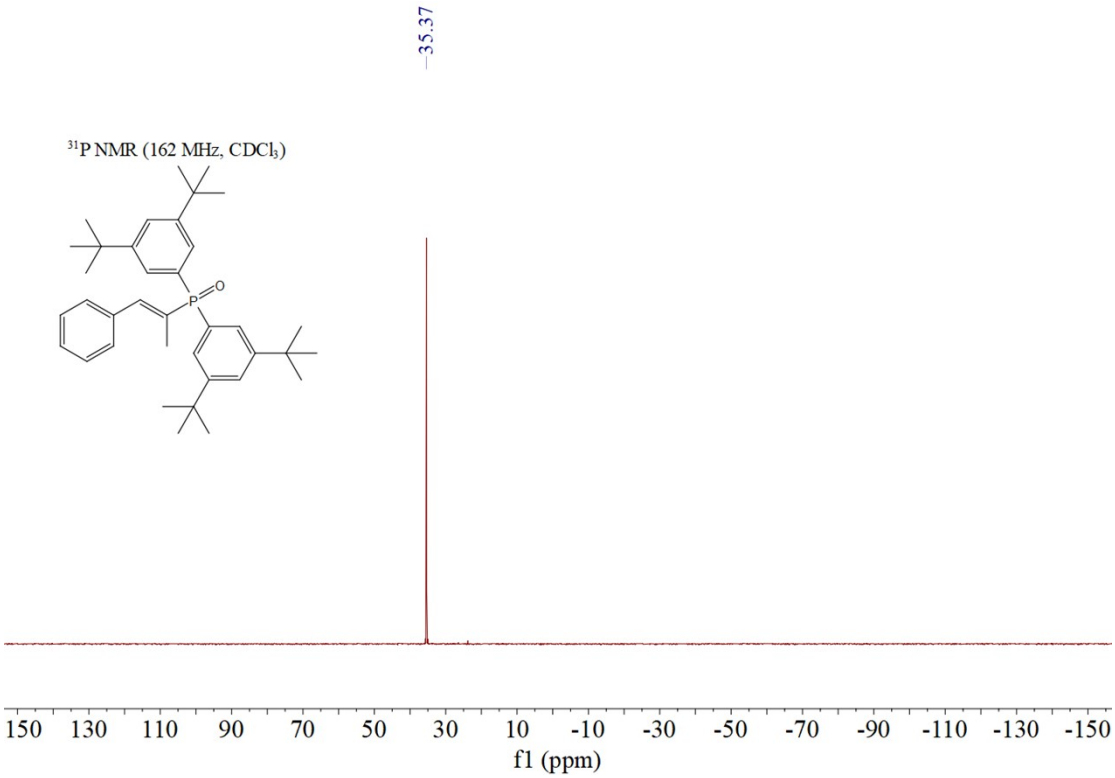

161.24
161.22
140.29
140.21
136.93
136.78
134.34
134.28
133.45
132.31
131.50
129.40
128.26
127.73
121.00
120.80
120.71
120.17
111.05
111.00
77.16 CDCl3
55.42
15.18
15.09
13C NMR (125 MHz, CDCl3)
MeO
O
P
MeO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



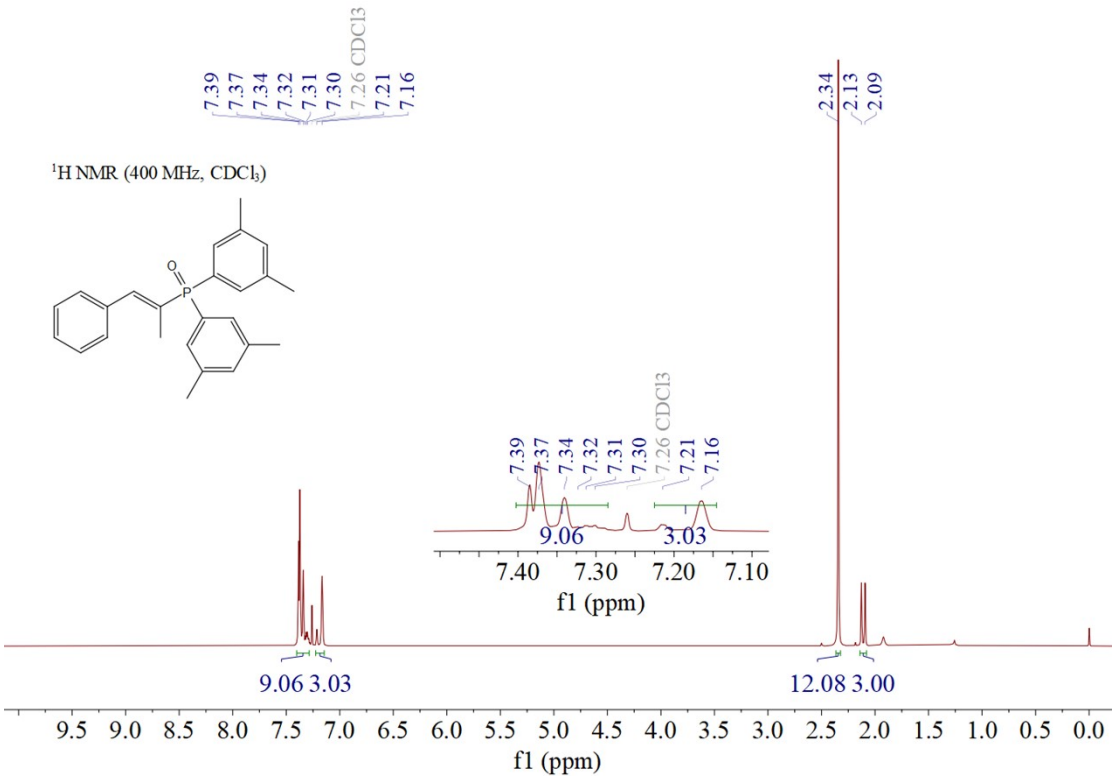
(*E*)-bis(3,5-di-tert-butylphenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3t**)

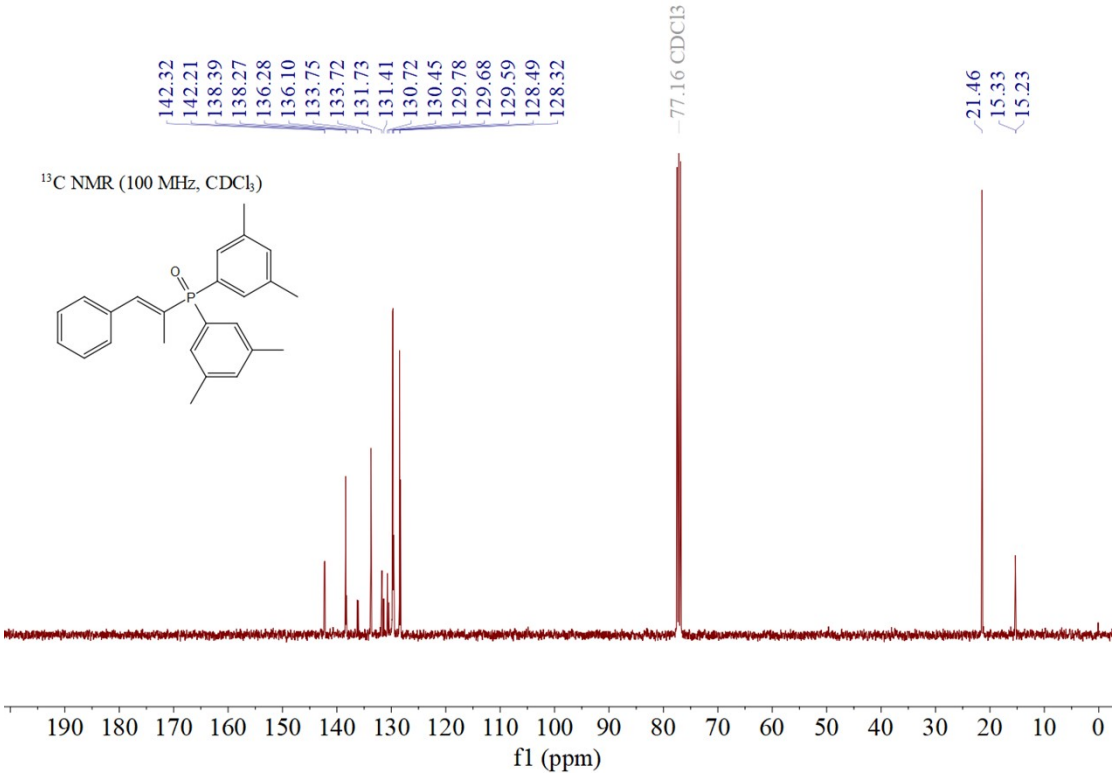


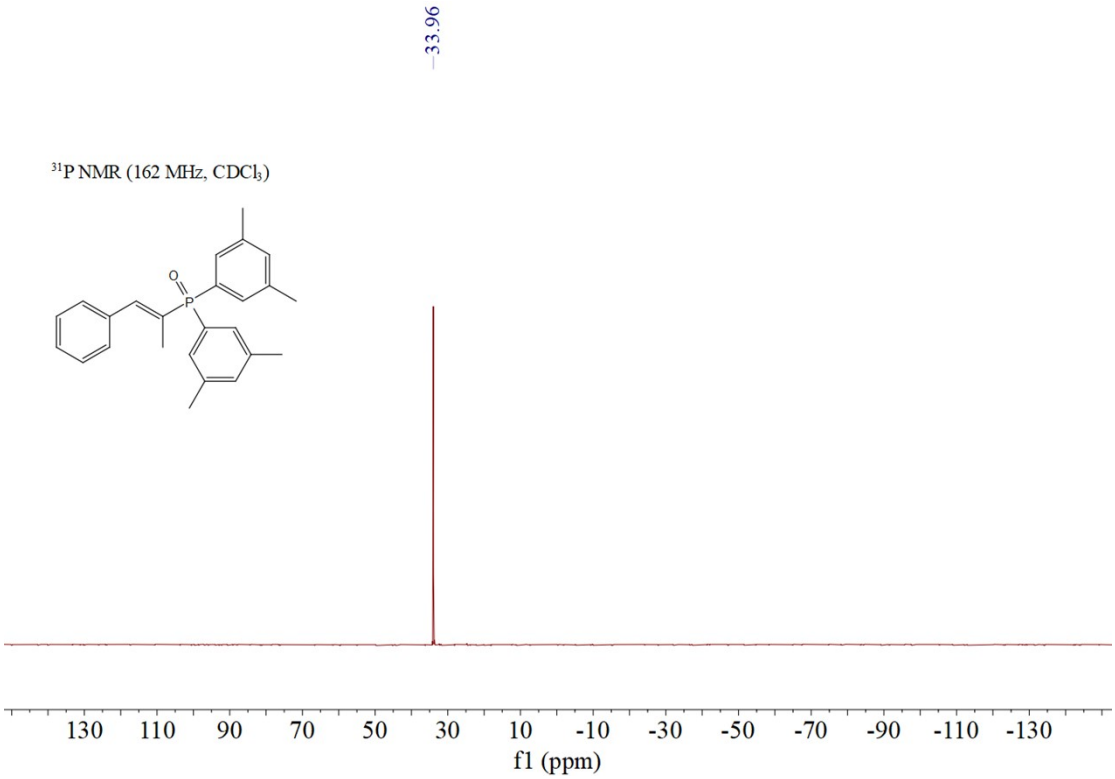




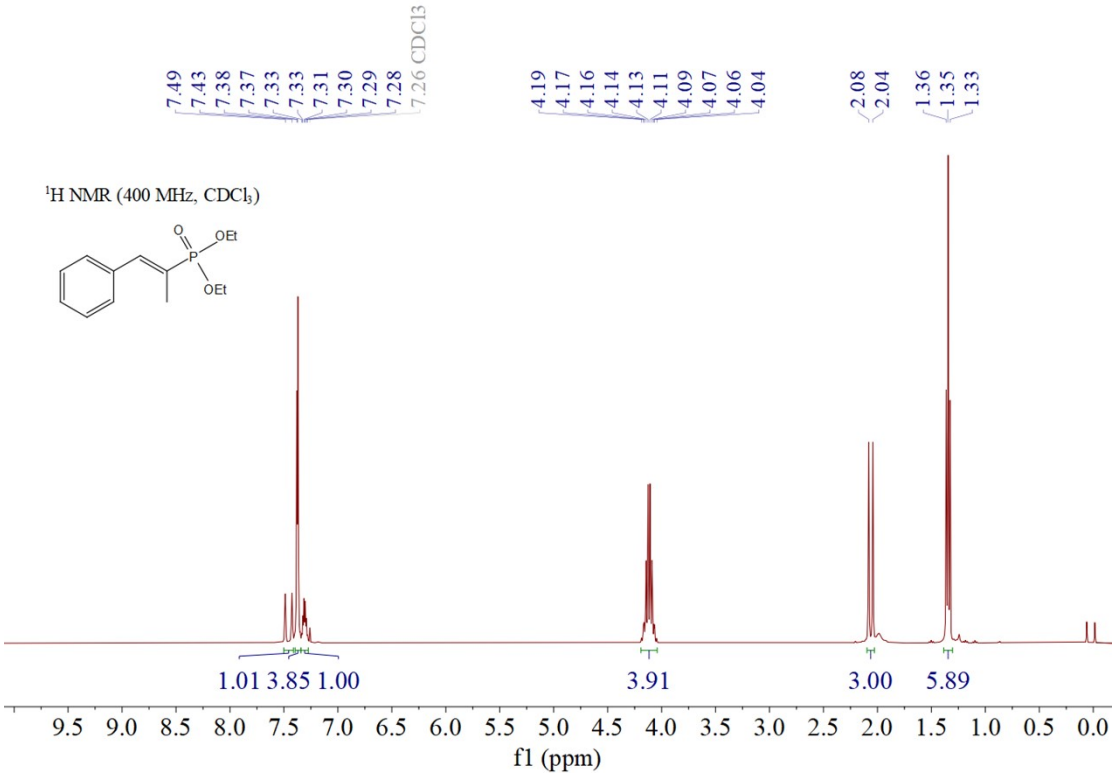
(*E*)-bis(3,5-dimethylphenyl)(1-phenylprop-1-en-2-yl)phosphine oxide (**3u**)


7.39
7.37
7.34
7.32
7.31
7.30
7.26 CDCl3
7.21
7.16
2.34
2.13
2.09
1H NMR (400 MHz, CDCl3)
9.06
3.03
7.40
7.30
7.20
7.10
f1 (ppm)
9.06 3.03
12.08 3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


142.32
142.21
138.39
138.27
136.28
136.10
133.75
133.72
131.73
131.41
130.72
130.45
129.78
129.68
129.59
128.49
128.32
77.16 CDCl3
21.46
15.33
15.23
13C NMR (100 MHz, CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

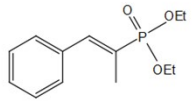


diethyl (*E*)-(1-phenylprop-1-en-2-yl)phosphonate (**3v**)



142.84
142.72
135.88
135.64
129.55
128.49
126.91
125.14
77.16 CDCl3
61.93
61.87
16.52
16.46
14.44
14.35

^{13}C NMR (100 MHz, $CDCl_3$)



22.12

^{31}P NMR (162 MHz, $CDCl_3$)