Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Silver-catalyzed Phosphonylation of Unprotected Propargylic Alcohols for Synthesis of Allenylphosphoryl Compounds

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Contents:

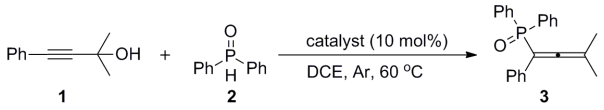
Ⅰ. General Methods and Materials

All reactions involving air- and moisture-sensitive reagents were carried out under an argon atmosphere. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker advance III 400 spectrometer (400 MHz for ^{1}H and 100 MHz for ^{13}C) in $CDCl_3$ with TMS as internal standard. Chemical shifts (δ) were measured in ppm relative to TMS δ = 0 for ^{1}H, or to chloroform δ = 77.0 for ^{13}C as internal standard. ^{31}P and ^{19}F NMR spectra were recorded on the same instrument. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), Coupling constants, *J*, are reported in hertz. Mass data were measured with Thermo Scientific DSQ II mass spectrometer. The starting materials were purchased from Aldrich, Acros Organics, J&K Chemicals or TCI and used without further purification. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". Thin-layer chromatography (TLC) was performed using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm). Substrates were prepared according to literature methods.[S1]

2. Optimization reaction conditions

2-1. Optimization varying different catalysts of reaction conditions[a]

Table S1 The experiment react with different catalysts

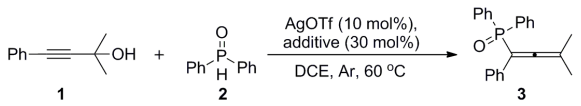


entry	catalyst	yield (%)[b]
1	AgOTf	36%
2	$Sc(OTf)_3$	21%
3	$Cu(OTf)_2$	35%
4	LiOTf	19%
5	$Sm(OTf)_3$	36%
6	CuOTf	13%
7	$AgNTf_2$	25%
8	$AgSbF_6$	28%
9	$AgBF_4$	trace
10	Ag_2CO_3	n. r.
11	$Cu(ClO_4)_2$	32%
12	$FeCl_3$	30%
13	$Fe(acac)_2$	22%
14	$Co(ClO_4)_2•6H_2O$	21%
15	$Ni(OAc)_2$	17%

[a] The reaction was carried out with catalyst (10 mol %), **1a** (0.30 mmol), and **2a** (1.5 equiv.) in DCE (1.5 mL) at 60 °C under argon for 14 h, unless otherwise noted. [b] Yield of isolated product.

2-2. Optimization varying different additives of reaction conditions[a]

Table S2 The experiment react with different additives



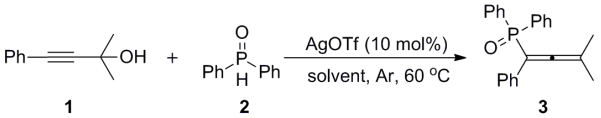
1 2 3

entry	additive	yield (%)[b]
1	Et_3N	n. r.
2	K_3PO_4	n. r.
3	K_2CO_3	n. r.
4	Pyruvic acid	35%
5	Trifluoroacetic anhydride	38%
6	CF_3SO_3H	30%
7	$TsOH•H_2O$	25%
8	Pivalic acid	28%
9[c]	$(NH_4)_2S_2O_8$	25%
10[c]	$PhI(OAc)_2$	n. r.

[a] The reaction was carried out with AgOTf (10 mol %), **1a** (0.30 mmol), and **2a** (1.5 equiv.), additive (30 mol %) in DCE (1.5 mL) at 60 °C under argon for 14 h, unless otherwise noted. [b] Yield of isolated product. [c] additive (100 mol %)

2-3. Optimization varying different solvents of reaction conditions [a]

Table S3 The experiment react with different solvents



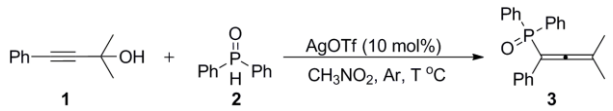
1 2 3

entry	solvent	yield (%)[b]
1	DCE	36%
2	MeCN	36%
3	THF	trace
4	dioxane	Trace
5	toluene	trace
6	DMF	n. r.
7	CF_3CH_2OH	**1** decomposition
8	DMSO	n. r.
9	CH_3NO_2	47%
10	DCM	30%

[a] The reaction was carried out with AgOTf (10 mol %), **1a** (0.30 mmol), and **2a** (1.5 equiv.) in solvent (1.5 mL) at 60 °C under argon for 14 h, unless otherwise noted. [b] Yield of isolated product.

2-4. Optimization varying different temperatures / substrate ratio of reaction conditions[a]

Table S4 The experiment react with different reaction conditions



entry	**1/2**	T (^{o}C)	yield (%)[b]
1	1/1	60	46%
2	1/2	60	48%
3	3/2	60	60%
4	2/1	60	75%
5	**2.5/1**	**60**	**87%**
6	3/1	60	87%
7	3/2	40	35%
8	3/2	80	43%
9	3/2	100	15%

[a] The reaction was carried out with AgOTf (10 mol %), **1a**, and **2a** in CH_3NO_2 (1.5 mL) at T ^{o}C under argon for 14 h, unless otherwise noted. [b] Yield of isolated product.

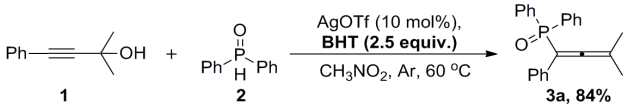
3. General Procedure for the Synthesis of 3a-3z.

To a Schlenk tube were added propargylic alcohol **1** (0.75 mmol), AgOTf (10 mmol%), P(O)H **2** (0.3 mmol) and charged with argon for three times. CH_3NO_2 (1.5 mL) was added and the mixture was stirred at 60 °C under Ar for 14 h. The substrate was consumed (monitored by TLC), After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo, and the resulting residue was purified by column chromatography using a mixture of petroleum ether / ethyl acetate and petroleum ether / Isopropanol as eluent to give **3**.

4. Preliminary mechanistic studies.

4.1 Radicals Trapping Experiments using BHT

In a Schlenk tube, 2-methyl-4-phenylbut-3-yn-2-ol **1a** (0.75 mmol), AgOTf (10 mol %), $HP(O)Ph_2$ (0.30 mmol) and BHT (2.5 equiv) were added and charged with Ar three times. Then, CH_3NO_2 (1.5 mL) were added. The mixture was allowed to stir at 60℃ for 14 hours (monitored by TLC). After substrate was consumed, the reaction was cooled to room temperature and concentrated in vacuo. The resulting residue was purified by column chromatography to give **3a** in 84% yield (Scheme S1). Thus this preliminary studies demonstrate that a radical pathway isn't involved in the reaction.

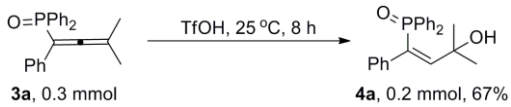


Scheme S1 Radicals Trapping Experiments using BHT

5. **Acid promoted transformations of allenes 3a into compounds 4a.**

A solution of allenes **3a** (0.3 mmol) in TfOH (1 mL) was stirred at 25 ^{o}C for 8 h. The mixture was poured into ice water (30 mL) and extracted with DCM (3 × 30 mL). The extracts were combined, washed with water, a saturated aqueous solution of $NaHCO_3$, and water again, and dried over

Na_2SO_4, and then the solvent was distilled off under reduced pressure, the resulting residue was purified by column chromatography to give **4a** in 67% yield (72 mg).



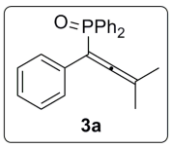
Scheme S2 Acid promoted transformations of allenes 3a into compounds 4a

(Z)-(3-hydroxy-3-methyl-1-phenylbut-1-en-1-yl)diphenylphosphine oxide (4a) white solid, **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.66 – 7.53 (m, 4H), 7.48 (td, *J* = 7.4, 1.2 Hz, 2H), 7.36 (td, *J* = 7.6, 3.0 Hz, 4H), 7.17 – 7.05 (m, 2H), 7.01 (t, *J* = 7.6 Hz, 2H), 6.83 – 6.58 (m, 3H), 1.56 (s, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ161.8 (d, *J* = 6.0 Hz), 139.7 (d, *J* = 12.6 Hz), 132.4, 132.3, 132.1, 131.8 (d, *J* = 2.7 Hz), 131.5, 131.2, 130.4, 129.6, 129.5, 128.1, 127.9, 127.7, 127.02 (d, *J* = 1.6 Hz), 70.3 (d, *J* = 5.0 Hz), 30.8. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 32.27. **MS (ESI)**: found $[M+H]^+$ 362.8.

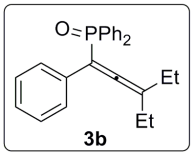
6. References

[S1] (a) Cao, C.; Li, Y.; Shi, Y.; Odom. A. L. *Chem. Commun.* **2004**, 2002. (b) Hashmi, A. S. K.; Wang, T.; Shi, S.; Rudolph, M. *J. Org. Chem.* **2012**, *77*, 7761. (c) Paegle, E.; Belyakov, S.; Petrova, M.; Liepinsh, E.; Arsenyan, P. *Eur. J. Org. Chem.* **2015**, 4389. (d) Csékei, M.; Novák, Z.; Kotschy, A. *Tetrahedron,* **2007**, *64*, 975.

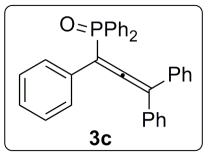
7. Characterization data of products



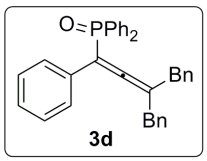
(3-methyl-1-phenylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3a) (87%) White solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (m, 4H), 7.57 (t, *J* = 10.5 Hz, 2H), 7.53 – 7.45 (m, 2H), 7.45 – 7.37 (m, 4H), 7.27 – 7.19 (m, 2H), 7.15 (t, *J* = 7.3 Hz, 1H), 1.46 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 210.0 (d, *J* = 6.2 Hz), 133.5, 133.4, 133.3, 132.4, 131.6, 131.5, 131.4, 128.4, 128.2, 128.1, 128.0, 127.2, 99.9 (d, *J* = 13.6 Hz), 99.1 (d, *J* = 103.3 Hz), 18.93 (d, *J* = 5.7 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.53. **MS (ESI)**: found $[M+H]^+$ 344.8.



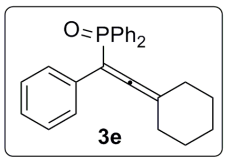
(3-ethyl-1-phenylpenta-1,2-dien-1-yl)diphenylphosphine oxide (3b) (30%) White solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 – 7.69 (m, 4H), 7.64 (d, *J* = 7.9 Hz, 2H), 7.53 – 7.45 (m, 2H), 7.45 – 7.36 (m, 4H), 7.30 – 7.20 (m, 2H), 7.16 (dd, *J* = 8.4, 6.2 Hz, 1H), 1.95 – 1.79 (m, 2H), 1.80 – 1.63 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 208.7 (d, *J* = 7.0 Hz), 133.7 (d, *J* = 8.2 Hz), 133.5, 132.5, 131.7, 131.6, 131.5, 131.4, 128.5, 128.2, 128.1, 127.8, 127.7, 127.1, 112.3 (d, *J* = 13.4 Hz), 102.5 (d, *J* = 104.3 Hz), 25.1 (d, *J* = 5.2 Hz), 12.0 (d, *J* = 1.9 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.30. **MS (ESI)**: found $[M+H]^+$ 372.9.



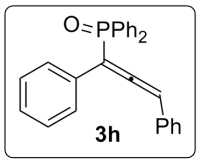
diphenyl(1,3,3-triphenylpropa-1,2-dien-1-yl)phosphine oxide (3c) (35%) White solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.70 (m, 6H), 7.40 (m, *2*H), 7.34 – 7.23 (m, 12H), 7.20 (t, *J* = 7.3 Hz, 1H), 7.03 (dd, *J* = 6.5, 2.8 Hz, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 212.48 (d, *J* = 5.3 Hz), 134.43 (d, *J* = 6.1 Hz), 132.80, 132.02 (d, *J* = 6.6 Hz), 131.76 (d, *J* = 3.0 Hz), 131.54 (d, *J* = 9.8 Hz), 128.74, 128.43, 128.26 (d, *J* = 7.2 Hz), 128.16 (d, *J* = 1.5 Hz), 128.13, 128.03, 127.91, 113.76 (d, *J* = 13.6 Hz), 104.10 (d, *J* = 99.2 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 29.59. **MS (ESI)**: found $[M+H]^+$ 469.0.



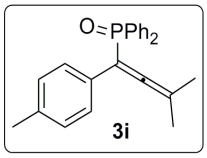
(3-benzyl-1,4-diphenylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3d) (70%) White solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.45 (m, 8H), 7.35 (m, 4H), 7.27 – 7.17 (m, 8H), 7.17 – 7.09 (m, 1H), 6.91 (m, 4H), 3.26 – 2.91 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.8 (d, *J* = 7.2 Hz), 137.5 (d, *J* = 2.1 Hz), 132.8 (d, *J* = 6.7 Hz), 131.7, 131.6, 131.5 (d, *J* = 2.7 Hz), 129.1, 128.3, 128.2, 128.1, 128.0, 127.9 (d, *J* = 4.8 Hz),, 127.2, 126.5, 108.6 (d, *J* = 13.5 Hz), 102.0 (d, *J* = 103.0 Hz), 38.4 (d, *J* = 5.0 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 30.47. **MS (ESI)**: found $[M+H]^+$ 497.2.



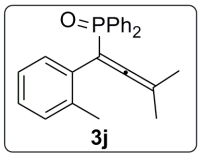
(2-cyclohexylidene-1-phenylvinyl)diphenylphosphine oxide (3e) (68%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.83 – 7.68 (m, 4H), 7.58 (d, *J* = 8.1 Hz, 2H), 7.53 – 7.46 (m, 2H), 7.42 (m, 4H), 7.24 (dd, *J* = 14.8, 7.5 Hz, 2H), 7.15 (t, *J* = 7.3 Hz, 1H), 2.06 (m, 2H), 1.95 – 1.78 (m, 2H), 1.46 (m, 2H), 1.32 (dd, *J* = 10.9, 4.7 Hz, 2H), 1.03 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 206.5 (d, *J* = 7.0 Hz), 133.6 (d, *J* = 8.1 Hz), 133.5, 132.5, 131.7, 131.6, 131.5 (d, *J* = 2.8 Hz), 128.4, 128.3, 128.2, 128.0 (d, *J* = 4.9 Hz), 127.0, 105.9 (d, *J* = 13.3 Hz), 98.9 (d, *J* = 104.8 Hz), 29.7 (d, *J* = 5.3 Hz), 26.2 (d, *J* = 3.0 Hz), 25.2. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.30. **MS (ESI)**: found $[M+H]^+$ 385.0.



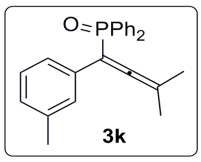
(1,3-diphenylpropa-1,2-dien-1-yl)diphenylphosphine oxide (3g) (88%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84 – 7.71 (m, 4H), 7.68 (d, *J* = 7.9 Hz, 2H), 7.49 – 7.41 (m, 1H), 7.41 – 7.33 (m, 3H), 7.33 – 7.17 (m, 8H), 7.11 (d, *J* = 7.9 Hz, 2H), 6.29 (d, *J* = 10.7 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 212.9 (d, *J* = 5.7 Hz), 132.6 (d, *J* = 38.3 Hz), 131.8 (d, *J* = 2.9 Hz), 131.7 (d, *J* = 2.6 Hz), 131.6, 131.6 (d, *J* = 1.7 Hz), 131.5, 131.4, 131.3, 128.7, 128.6, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 126.8 (d, *J* = 2.1 Hz), 105.4 (d, *J* = 98.8 Hz), 98.4 (d, *J* = 13.1 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 29.72. **MS (ESI)**: found $[M+H]^+$ 392.8.



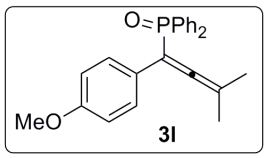
(3-methyl-1-(p-tolyl)buta-1,2-dien-1-yl)diphenylphosphine oxide (3i) (88%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.87 – 7.64 (m, 4H), 7.54 – 7.44 (m, 4H), 7.40 (m, 4H), 7.04 (d, *J* = 8.0 Hz, 2H), 2.25 (s, 3H), 1.45 (d, *J* = 6.1 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ209.6 (d, *J* = 6.2 Hz), 136.8, 132.9 (d, *J* = 105.6 Hz), 131.5, 131.4, 130.2 (d, *J* = 8.0 Hz), 129.1, 128.1, 128.0, 127.9, 99.6 (d, *J* = 13.7 Hz), 98.7 (d, *J* = 103.4 Hz), 20.9, 18.9 (d, *J* = 5.8 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.65. **MS (ESI)**: found $[M+H]^+$ 358.9.



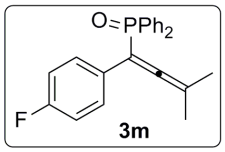
(3-methyl-1-(o-tolyl)buta-1,2-dien-1-yl)diphenylphosphine oxide (3j) (86%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (m, 4H), 7.62 – 7.35 (m, 7H), 7.22 – 6.95 (m, 3H), 2.35 (s, 3H), 1.47 (d, *J* = 6.2 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.8 (d, *J* = 6.2 Hz), 136.5 (d, *J* = 5.6 Hz), 133.2, 132.2 (d, *J* = 7.0 Hz), 132.1, 131.5, 131.4, 131.3 (d, *J* = 2.7 Hz), 130.4, 129.4 (d, *J* = 3.0 Hz), 128.0, 127.9, 127.3, 125.6, 97.3 (d, *J* = 14.0 Hz), 97.2 (d, *J* = 101.2 Hz). 20.7, 18.7 (d, *J* = 5.8 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 30.39. **MS (ESI)**: found $[M+H]^+$ 358.7.



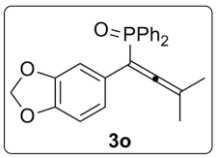
(3-methyl-1-(m-tolyl)buta-1,2-dien-1-yl)diphenylphosphine oxide (3k) (78%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.85 – 7.63 (m, 4H), 7.58 – 7.32 (m, 8H), 7.13 (m, 1H), 6.97 (d, *J* = 7.5 Hz, 1H), 2.26 (s, 3H), 1.45 (d, *J* = 6.1 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 210.0 (d, *J* = 6.2 Hz), 138.0, 133.5, 133.2 (d, *J* = 8.0 Hz), 132.5, 131.5, 131.4 (d, *J* = 4.6 Hz), 128.7 (d, *J* = 5.0 Hz), 128.3, 128.1, 128.0, 125.3 (d, *J* = 4.8 Hz), 99.7 (d, *J* = 13.6 Hz), 99.0 (d, *J* = 103.1 Hz), 21.4, 18.9 (d, *J* = 5.8 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.56. **MS (ESI)**: found $[M+H]^+$ 359.0.



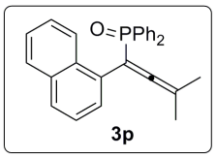
(1-(4-methoxyphenyl)-3-methylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3l) (65%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 – 7.66 (m, 4H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.49 – 7.45 (m, 2H), 7.45 – 7.39 (m, 4H), 6.82 – 6.72 (m, 2H), 3.72 (s, 3H), 1.45 (d, *J* = 6.1 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.3 (d, *J* = 6.3 Hz), 133.4, 132.3 (d, *J* = 6.8 Hz), 131.5, 131.4 (d, *J* = 3.7 Hz), 130.6, 129.3 (d, *J* = 5.0 Hz), 128.4 (d, *J* = 11.0 Hz), 128.2, 128.0, 125.4 (d, *J* = 8.2 Hz), 113.9, 113.6, 99.8 (d, *J* = 13.8 Hz), 98.4 (d, *J* = 103.6 Hz), 55.1, 19.0 (d, *J* = 5.8 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.93. **MS (ESI)**: found $[M+H]^+$ 375.0.



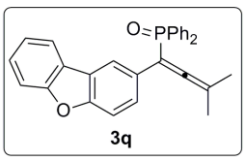
(1-(4-fluorophenyl)-3-methylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3m) (80%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (dd, *J* = 11.9, 7.7 Hz, 4H), 7.58 (dd, *J* = 8.5, 5.5 Hz, 2H), 7.53 – 7.46 (m, 2H), 7.43 (t, *J* = 7.2 Hz, 4H), 6.92 (t, *J* = 8.4 Hz, 2H), 1.46 (d, *J* = 5.9 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ209.6 (d, *J* = 6.0 Hz), 163.1, 160.6, 133.1, 132.0, 131.5 (d, *J* = 2.8 Hz), 131.5, 131.4, 129.8 (d, *J* = 4.9 Hz), 129.7 (d, *J* = 4.9 Hz), 129.3, 129.2, 128.2, 128.1, 115.4, 115.2, 100.2 (d, *J* = 13.6 Hz), 98.1 (d, *J* = 103.4 Hz), 18.9 (d, *J* = 5.7 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.76. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -114.71. **MS (ESI)**: found $[M+H]^+$ 362.8.



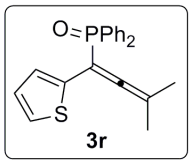
(1-(benzo[d][1,3]dioxol-5-yl)-3-methylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3o) (70%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 – 7.68 (m, 4H), 7.59 – 7.36 (m, 6H), 7.23 – 7.12 (m, 1H), 7.06 (d, *J* = 1.3 Hz, 1H), 6.68 (t, *J* = 8.4 Hz, 1H), 5.86 (s, 2H), 1.44 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.3 (d, *J* = 6.3 Hz), 147.6, 146.8, 132.7 (d, *J* = 105.8 Hz), 131.5, 131.4, 128.1 (d, *J* = 12.2 Hz), 127.0 (d, *J* = 8.3 Hz), 122.0, 121., 108.4 (d, *J* = 5.7 Hz), 108.2, 100.8, 100.0 (d, *J* = 13.5 Hz), 98.7 (d, *J* = 103.8 Hz), 18.9 (d, *J* = 5.7 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.69. **MS (ESI)**: found $[M+H]^+$ 388.8.



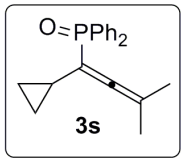
(3-methyl-1-(naphthalen-1-yl)buta-1,2-dien-1-yl)diphenylphosphine oxide (3p) (86%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.22 (t, *J* = 7.8 Hz, 1H), 7.87 – 7.73 (m, 6H), 7.68 (d, *J* = 8.2 Hz, 1H), 7.54 – 7.46 (m, 1H), 7.38 (m, 8H), 1.52 (d, *J* = 6.1 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 211.1 (d, *J* = 5.9 Hz), 133.9, 133.1, 131.8 (d, *J* = 50.6 Hz), 131.5 (d, *J* = 9.3 Hz), 131.4 (d, *J* = 2.8 Hz), 130.4 (d, *J* = 7.4 Hz), 128.5, 128.0 (d, *J* = 12.1 Hz), 127.9, 127.3 (d, *J* = 3.6 Hz), 125.9, 125.4, 125.2, 124.8, 97.6 (d, *J* = 13.9 Hz), 96.4 (d, *J* = 101.1 Hz), 18.8 (d, *J* = 5.8 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 30.63. **MS (ESI)**: found $[M+H]^+$ 394.8.



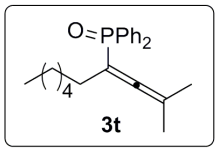
(1-(dibenzo[b,d]furan-2-yl)-3-methylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3q) (82%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.26 (s, 1H), 7.90 (d, *J* = 7.5 Hz, 1H), 7.85 – 7.75 (m, 4H), 7.72 (dd, *J* = 8.6, 1.3 Hz, 1H), 7.54 – 7.36 (m, 9H), 7.25 (dd, *J* = 14.8, 7.6 Hz, 1H), 1.50 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.9 (d, *J* = 6.2 Hz), 156.3, 155.4, 132.8 (d, *J* = 105.8 Hz),, 131.6, 131.5 (d, *J* = 2.8 Hz), 131.4, 128.3, 128.2, 128.1, 127.6 (d, *J* = 5.2 Hz), 127.0, 124.4, 124.0, 122.5, 120.9, 120.4, 120.3, 111.5 (d, *J* = 12.9 Hz), 100.0 (d, *J* = 13.6 Hz), 98.9 (d, *J* = 103.0 Hz), 19.0 (d, *J* = 5.7 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.93. **MS (ESI)**: found $[M+H]^+$ 435.0.



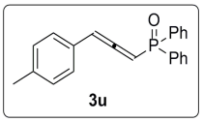
(3-methyl-1-(thiophen-2-yl)buta-1,2-dien-1-yl)diphenylphosphine oxide (3r) (40%) Light yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (m, 4H), 7.57 – 7.48 (m, 2H), 7.48 – 7.37 (m, 4H), 7.34 – 7.23 (m, 1H), 7.19 – 7.07 (m, 1H), 6.86 (dd, *J* = 5.1, 3.7 Hz, 1H), 1.47 (d, *J* = 5.9 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 208.3 (d, *J* = 5.5 Hz), 136.4 (d, *J* = 12.1 Hz), 132.8, 131.8, 131.7 (d, *J* = 2.8 Hz), 131.6, 131.5, 128.2 (d, *J* = 12.3 Hz), 127.7, 126.8 (d, *J* = 2.5 Hz), 124.8, 100.9 (d, *J* = 13.1 Hz), 94.5 (d, *J* = 103.3 Hz), 19.1 (d, *J* = 5.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 30.77. **MS (ESI)**: found $[M+H]^+$ 351.0.



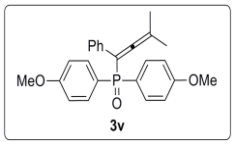
(1-cyclopropyl-3-methylbuta-1,2-dien-1-yl)diphenylphosphine oxide (3s) (86%) Colorless liquid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.90 – 7.64 (m, 4H), 7.62 – 7.38 (m, 6H), 1.43 (s, 3H), 1.40 (s, 3H), 0.90 – 0.64 (m, 2H), 0.61 – 0.35 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 204.9 (d, *J* = 7.7 Hz), 132.9, 131.9, 131.5, 131.4, 128.0, 127.9, 100.6 (d, *J* = 105.1 Hz).100.5 (d, *J* = 14.0 Hz), 19.2 (d, *J* = 5.9 Hz), 8.6 (d, *J* = 3.2 Hz), 8.4 (d, *J* = 12.5 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 32.09. **MS (ESI)**: found $[M+H]^+$ 308.7.



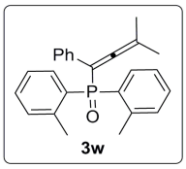
(2-methyldeca-2,3-dien-4-yl)diphenylphosphine oxide (3t) (72%) Colorless liquid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.82 – 7.59 (m, 4H), 7.58 – 7.35 (m, 6H), 2.21 (dd, *J* = 15.3, 8.0 Hz, 2H), 1.55 – 1.37 (m, 8H), 1.35 – 1.13 (m, 6H), 0.84 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 207.5 (d, *J* = 6.8 Hz), 132.6 (d, *J* = 103.3 Hz), 131.5, 131.4, 131.4 (d, *J* = 2.7 Hz), 128.1, 128.0, 98.4 (d, *J* = 14.7 Hz), 96.5 (d, *J* = 102.9 Hz), 31.6, 28.7, 28.3 (d, *J* = 5.9 Hz), 27.4 (d, *J* = 8.1 Hz), 22.5, 19.2 (d, *J* = 6.1 Hz), 14.0. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.71. **MS (ESI)**: found $[M+H]^+$ 352.9.



Diphenyl(3-p-tolylpropa-1,2-dien-1-yl)phosphine oxide (3u) (60%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.84 – 7.71 (m, 4H), 7.49 (ddd, *J* = 14.7, 7.3, 1.4 Hz, 2H), 7.45 – 7.37 (m, 4H), 7.05 (dd, *J* = 18.8, 8.1 Hz, 4H), 6.35 – 6.19 (m, 2H), 2.31 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 213.1, 137.7, 132.9, 132.7, 131.9 (d, *J* = 2.5 Hz), 131.8, 131.7, 131.4, 131.3 (d, *J* = 3.0 Hz), 131.2, 129.4 (d, *J* = 1.3 Hz), 128.4 (d, *J* = 2.2 Hz), 128.3 (d, *J* = 2.2 Hz), 128.2, 128.1, 126.9 (d, *J* = 2.2 Hz), 96.1 (d, *J* = 13.6 Hz), 89.5 (d, *J* = 103.1 Hz), 21.2. **^{31}P NMR (162 MHz, $CDCl_3$)** δ 23.93. **MS (ESI)**: found $[M+H]^+$ 331.1.

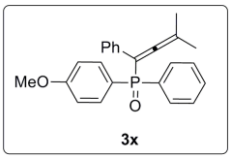


bis(4-methoxyphenyl)(3-methyl-1-phenylbuta-1,2-dien-1-yl)phosphine oxide (3v) (40%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 – 7.61 (m, 4H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 2H), 7.15 (dd, *J* = 8.4, 6.3 Hz, 1H), 6.97 – 6.87 (m, 4H), 3.82 (s, 6H), 1.50 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.8 (d, *J* = 6.1 Hz), 162.0 (d, *J* = 2.8 Hz), 133.6 (d, *J* = 7.9 Hz), 133.3, 133.2, 128.4, 128.2 (d, *J* = 4.9 Hz), 127.0, 125.1, 124.0, 113.7 (d, *J* = 13.2 Hz), 99.6 (d, *J* = 103.5 Hz), 99.4 (d, *J* = 13.5 Hz), 55.2, 19.1 (d, *J* = 5.7 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.28. **MS (ESI)**: found $[M+H]^+$ 404.9.



3w

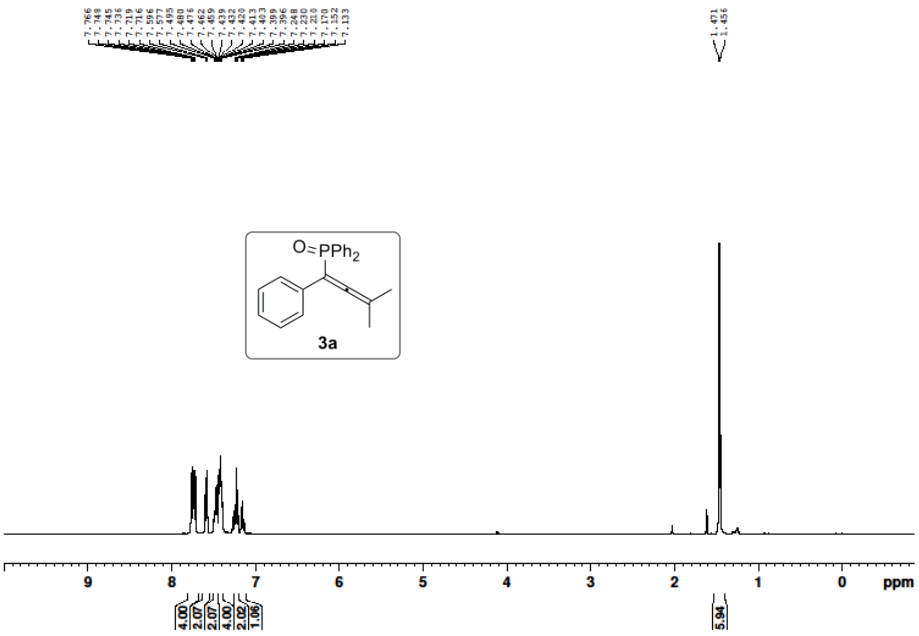
(3-methyl-1-phenylbuta-1,2-dien-1-yl)di-o-tolylphosphine oxide (3w) (48%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 (d, *J* = 8.0 Hz, 2H), 7.42 – 7.32 (m, 4H), 7.27 (dt, *J* = 7.6, 6.3 Hz, 4H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 2H), 2.60 (s, 6H), 1.38 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 208.7 (d, *J* = 6.2 Hz), 143.0 (d, *J* = 7.6 Hz), 134.0 (d, *J* = 7.3 Hz), 132.4 (d, *J* = 12.4 Hz), 131.9, 131.7, 131.6, 131.5, 131.4, 130.9, 128.5 (d, *J* = 3.0 Hz), 127.1, 125.1 (d, *J* = 12.9 Hz), 99.9 (d, *J* = 13.6 Hz), 98.5 (d, *J* = 101.6 Hz), 21.8 (d, *J* = 4.1 Hz), 18.5 (d, *J* = 5.7 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 37.73. **MS (ESI)**: found $[M+H]^+$ 372.8.



3x

(4-methoxyphenyl)(3-methyl-1-phenylbuta-1,2-dien-1-yl)(phenyl)phosphine oxide (3x) (51%) white solid; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78 – 7.63 (m, 4H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.52 – 7.35 (m, 3H), 7.23 (t, *J* = 7.5 Hz, 2H), 7.15 (t, *J* = 7.3 Hz, 1H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 1.48 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.8 (d, *J* = 6.1 Hz), 162.2 (d, *J* = 2.8 Hz), 133.5 (d, *J* = 106.9 Hz), 133.4 (d, *J* = 8.0 Hz), 133.3, 133.0, 131.4, 131.3 (d, *J* = 4.5 Hz), 128.4, 128.2, 128.1, 128.0, 127.9, 127.1, 123.9 (d, *J* = 111.5 Hz), 113.8 (d, *J* = 13.2 Hz), 99.7 (d, *J* = 13.5 Hz), 99.3 (d, *J* = 103.4 Hz), 55.2, 19.0 (dd, *J* = 5.7, 3.6 Hz). **^{31}P NMR (162 MHz, $CDCl_3$)** δ 31.47. **MS (ESI)**: found $[M+H]^+$ 374.8.

8. Copies of NMR spectra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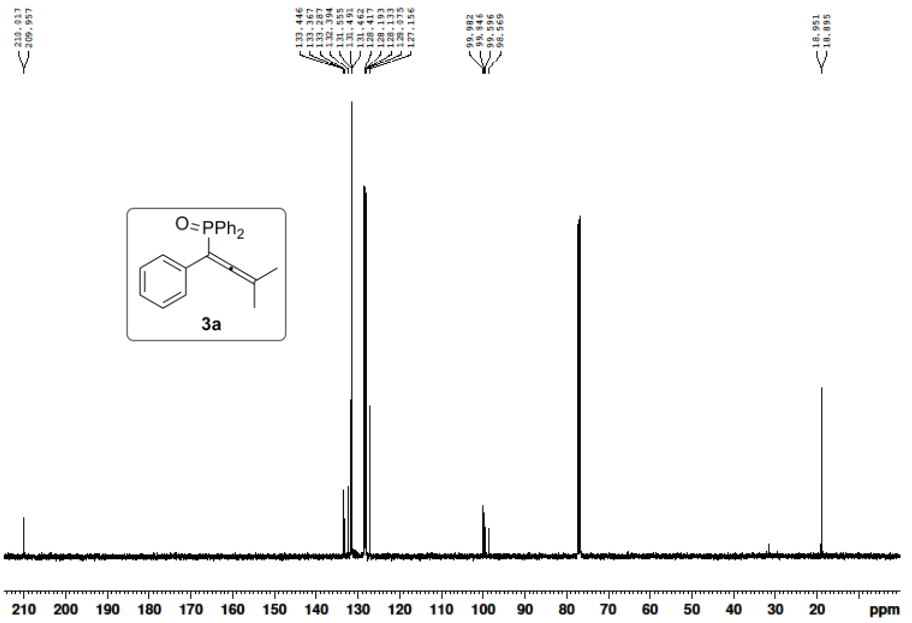
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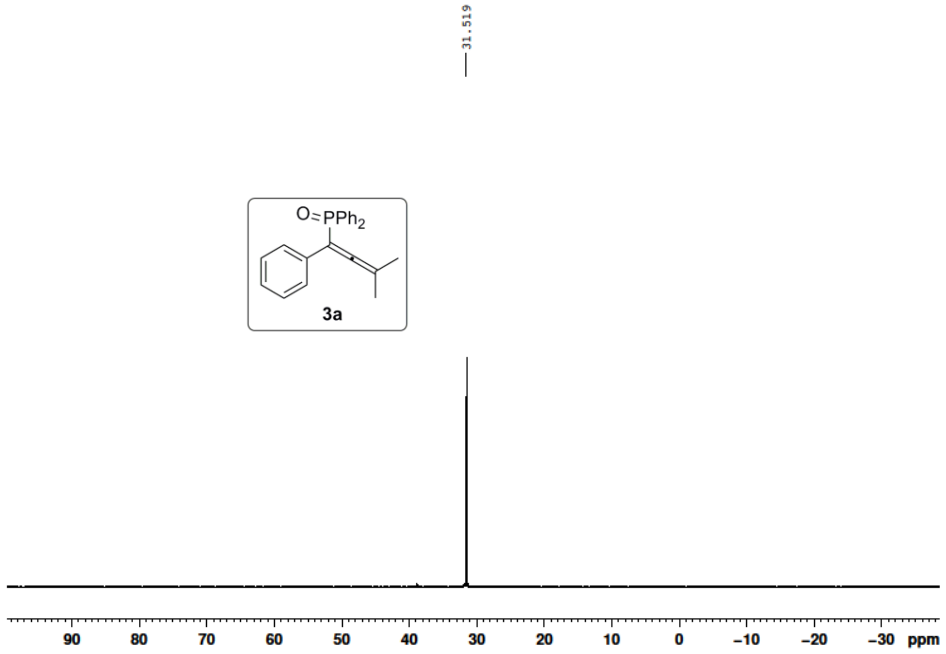
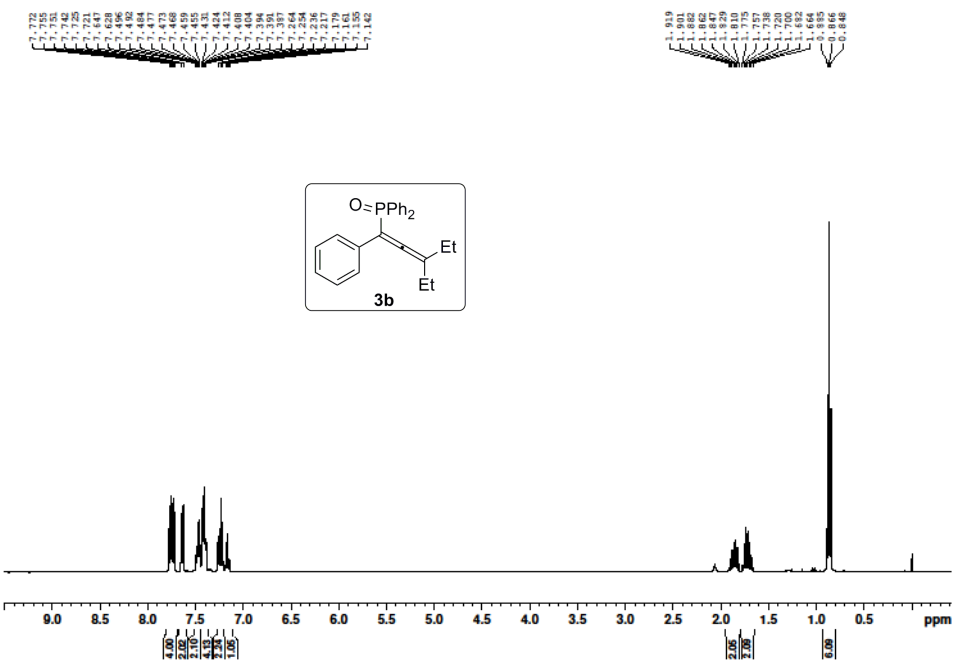
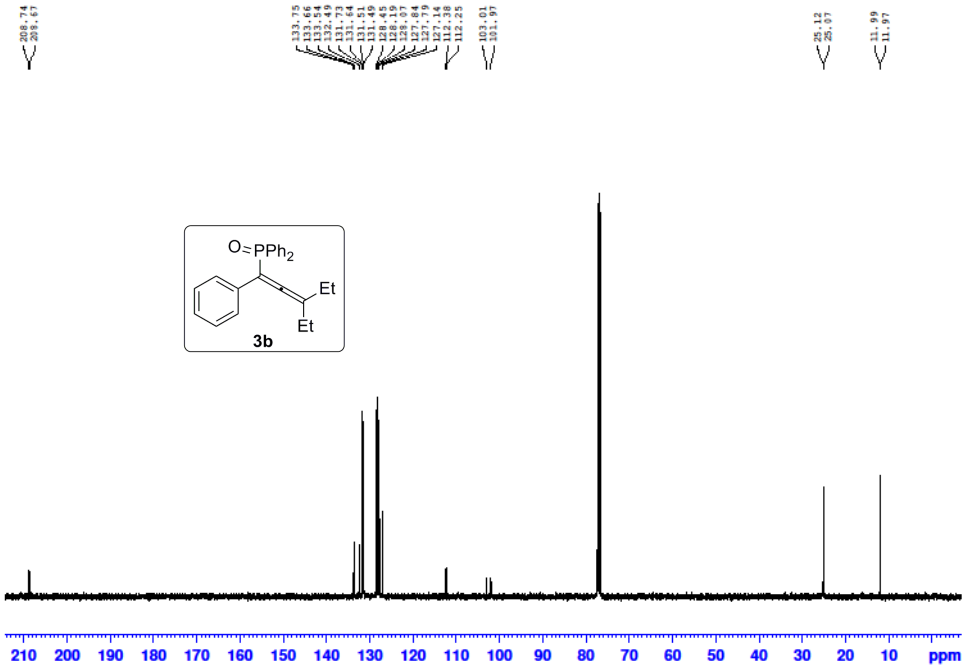

31.519
O=PPh2
3a
90 80 70 60 50 40 30 20 10 0 −10 −20 −30 ppm


O=PPh2
Et
Et
3b
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 ppm


208.74
209.67
133.75
133.66
133.44
132.49
131.73
131.64
131.61
131.46
128.45
128.19
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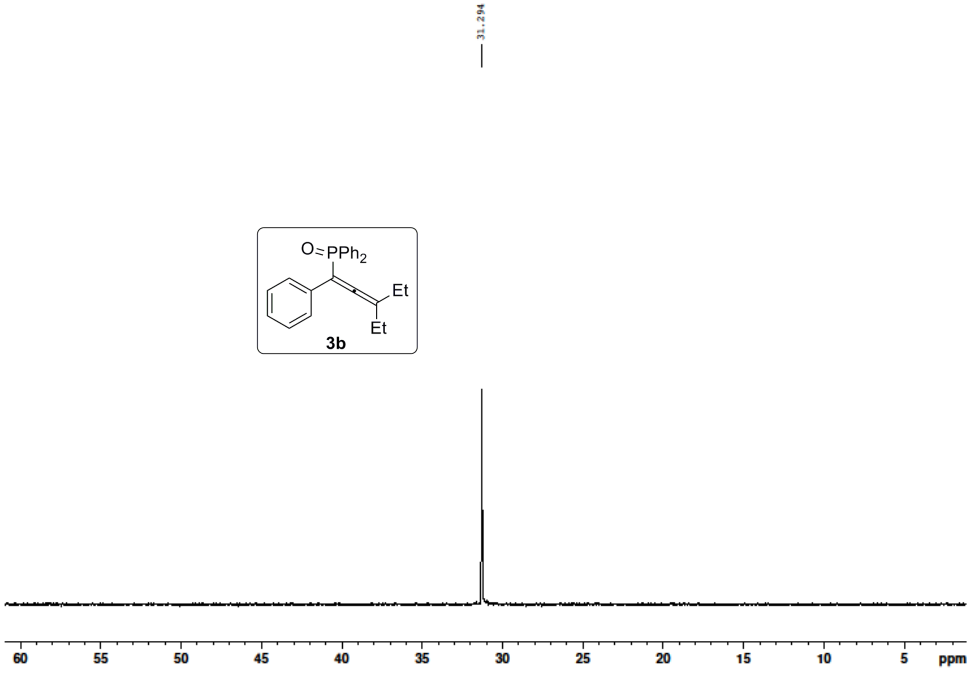
127.84
127.79
127.14
112.38
112.25
103.01
101.97
25.12
25.07
11.99
11.97
O≈PPh2
Et
Et
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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Et
Et
3b
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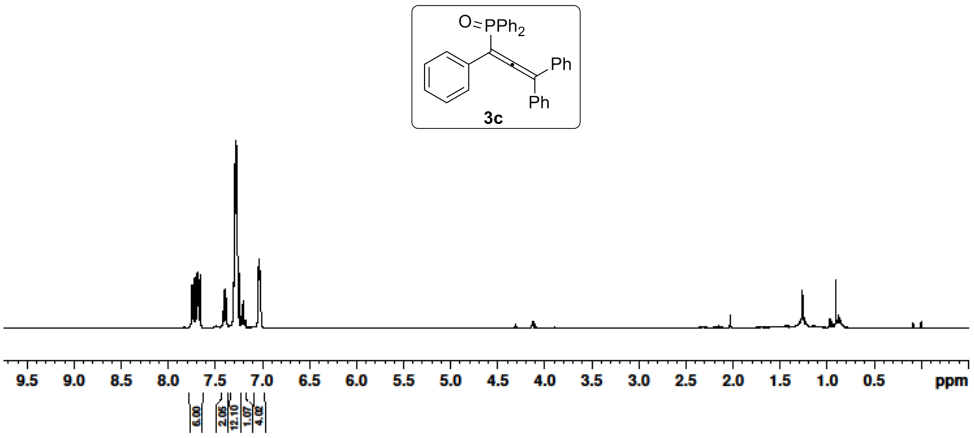
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Ph
Ph
3c
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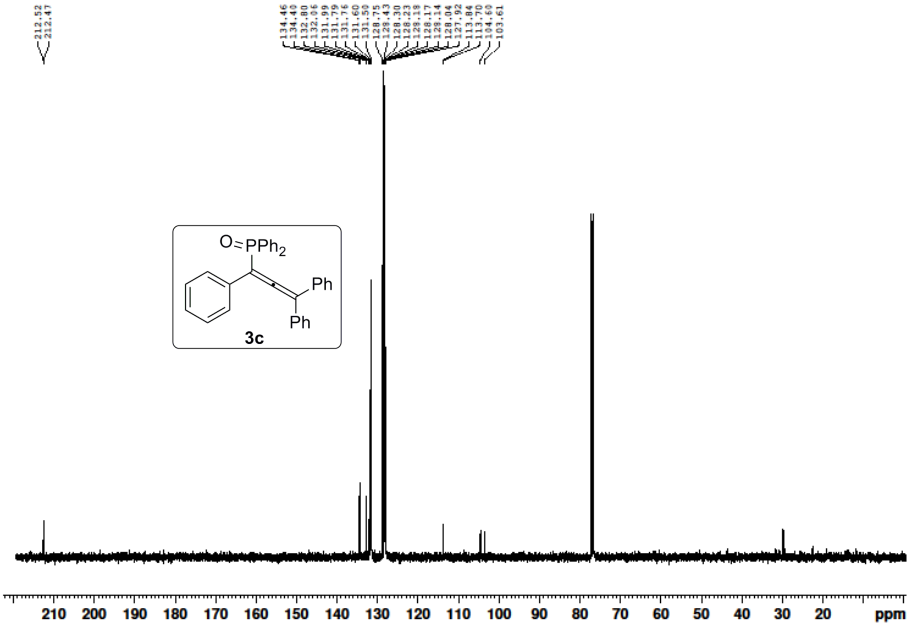
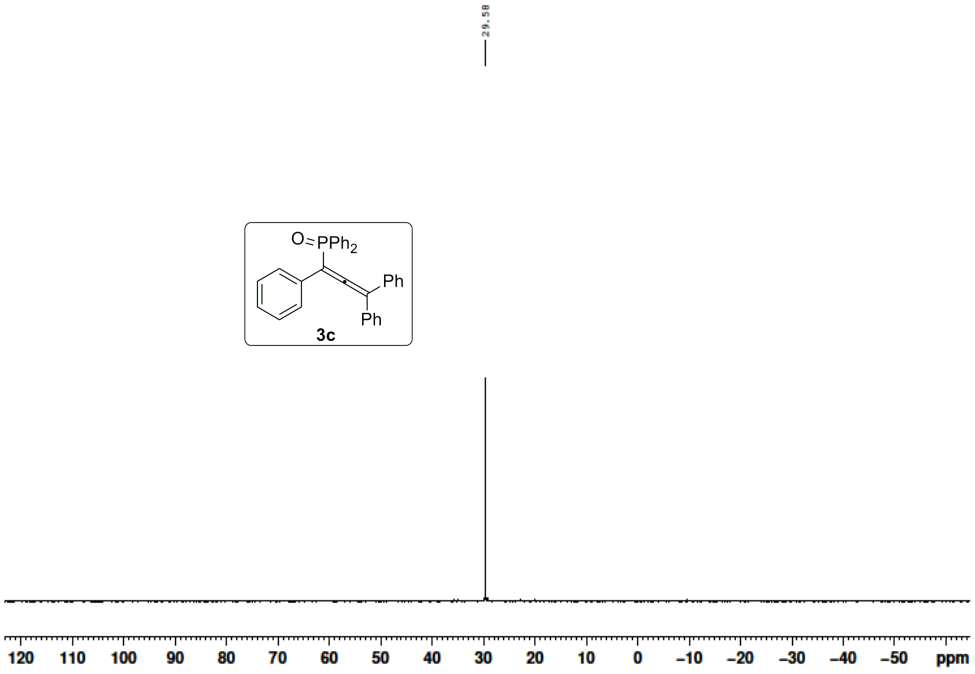
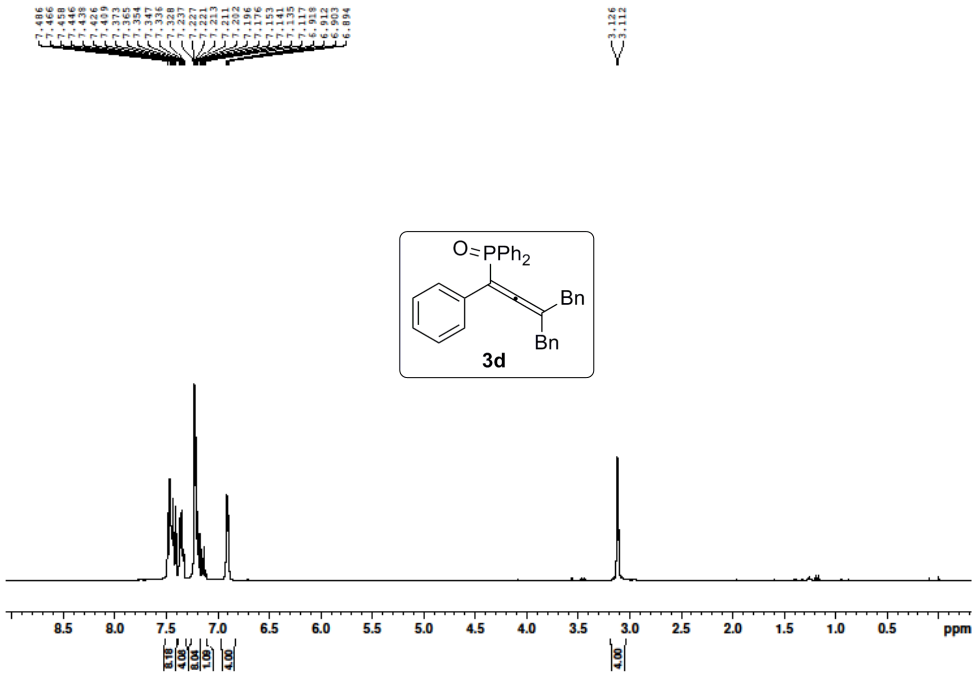
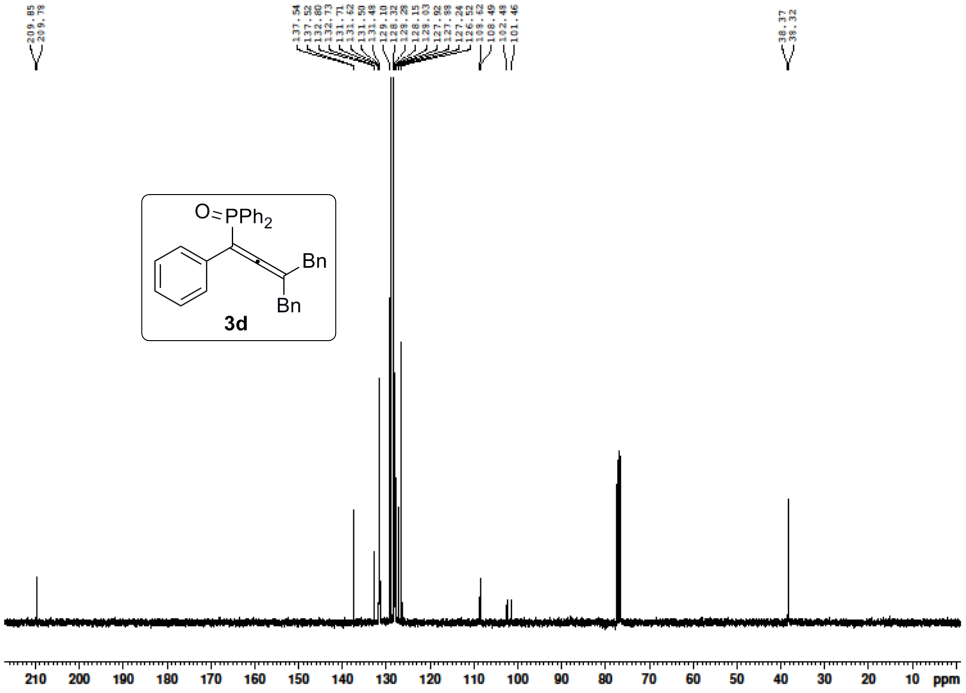
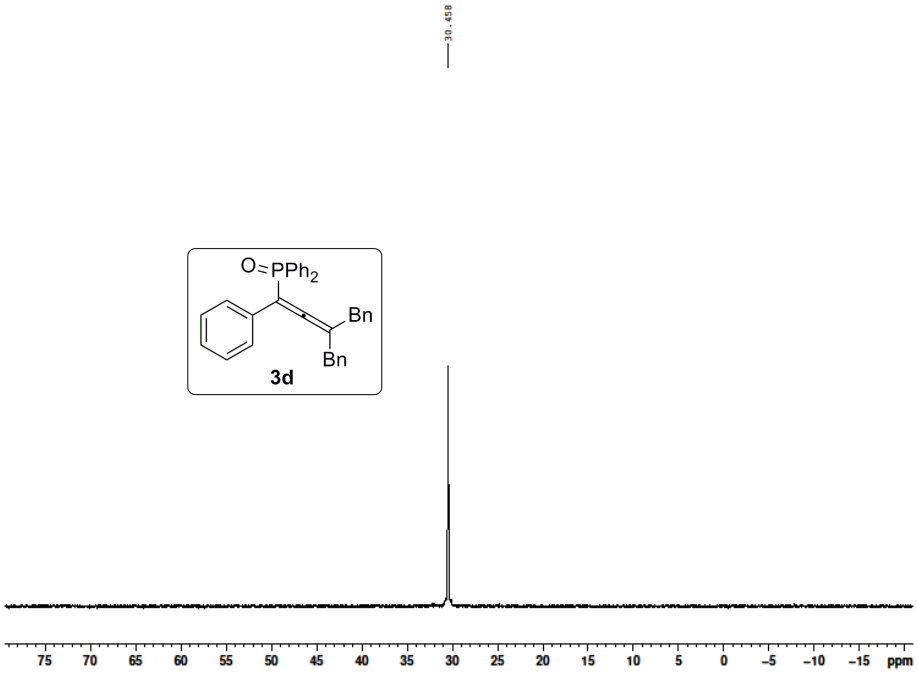
4.00
2.05
12.10
1.07
4.08

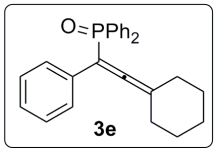

O≃PPh2
Ph
Ph
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

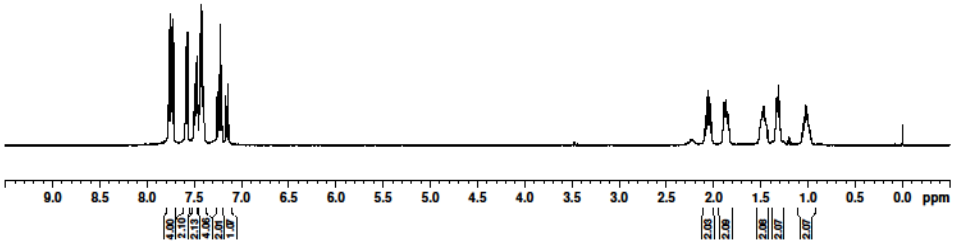

29.58
O≃PPh2
Ph
Ph
3c
120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm

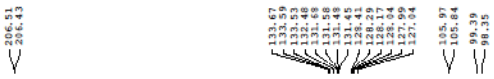

7.486
7.466
7.458
7.446
7.438
7.426
7.409
7.373
7.365
7.364
7.347
7.336
7.328
7.227
7.221
7.213
7.211
7.202
7.196
7.176
7.153
7.141
7.135
7.117
6.918
6.912
6.903
6.894
3.126
3.112
O≃PPh2
Bn
Bn
3d
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.18
4.00
4.04
8.04
1.00
4.00
4.00

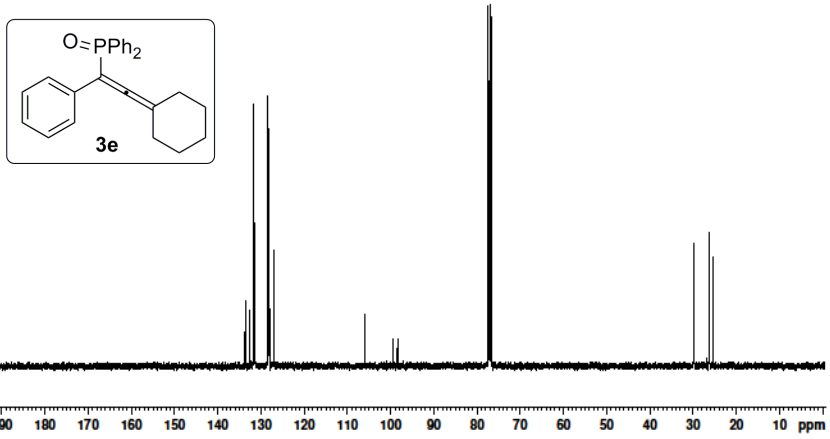

O≃PPh2
Bn
Bn
3d
ppm

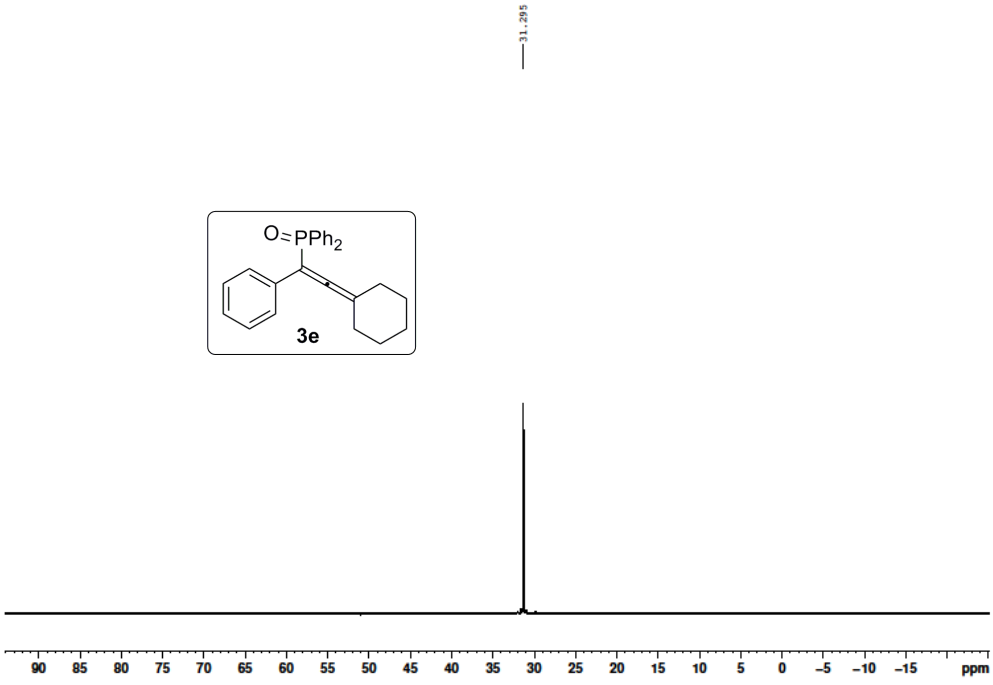

O≃PPh2
Bn
Bn
3d
ppm

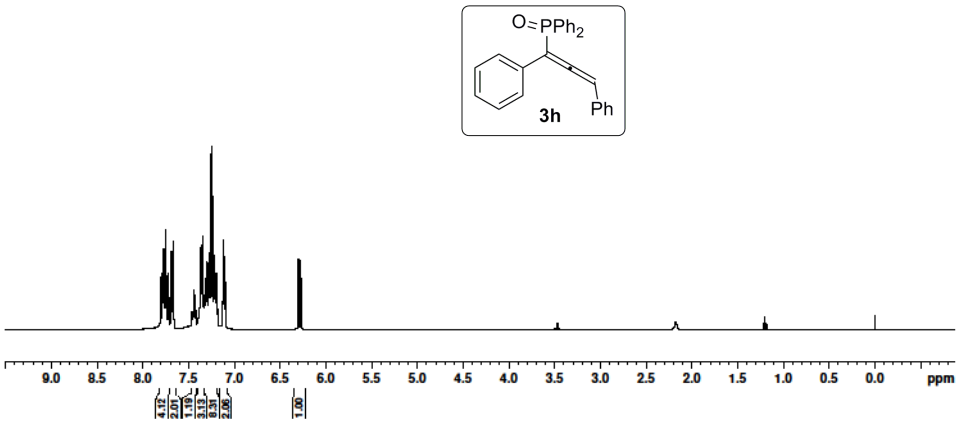

O=PPh2
3e

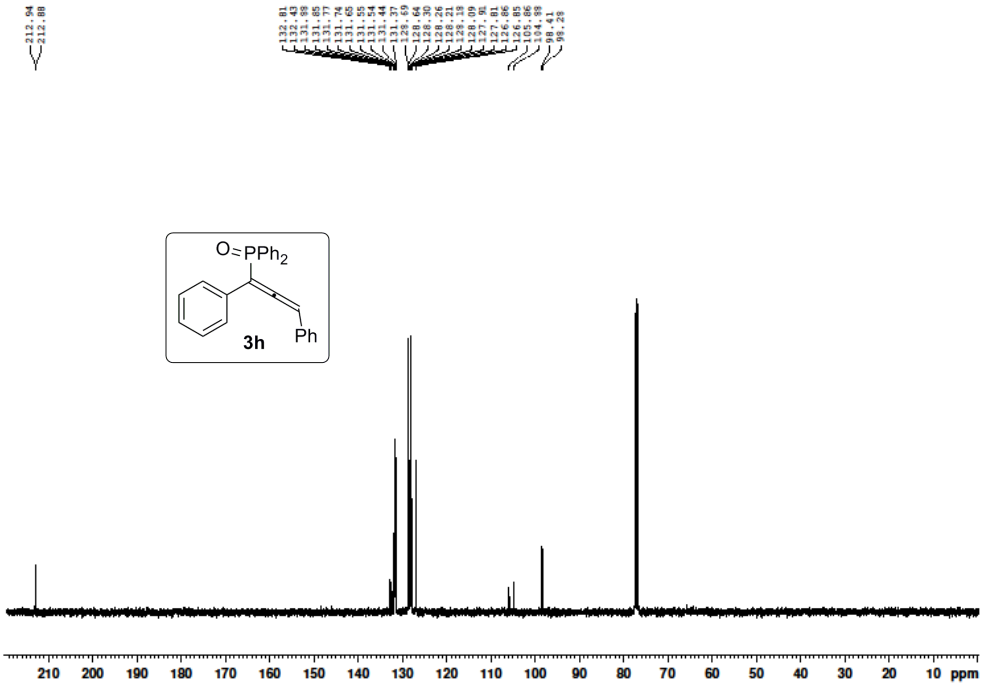

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

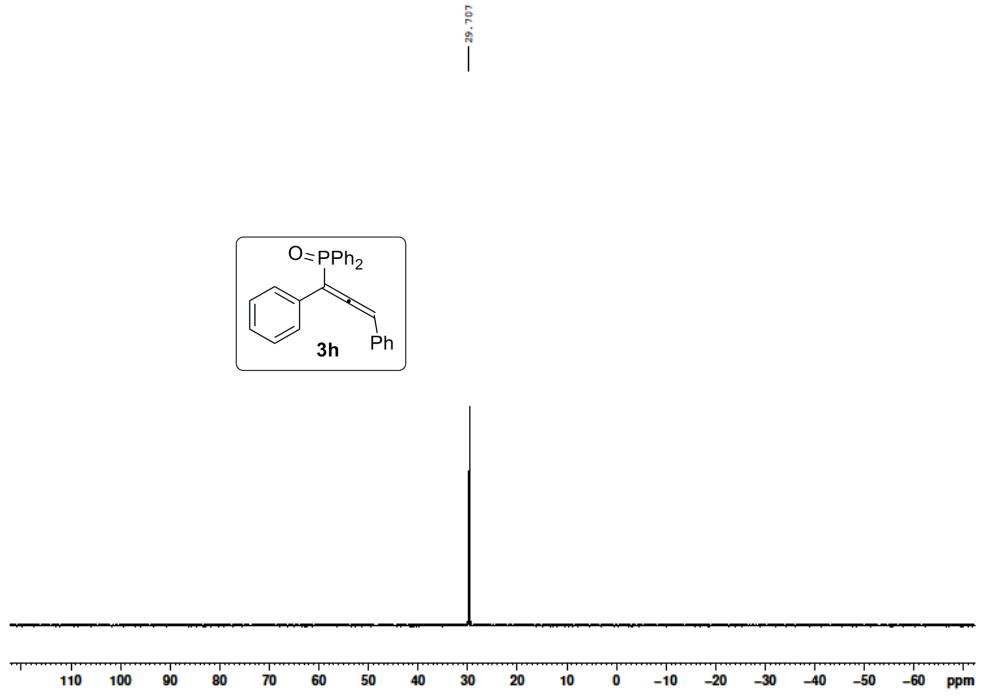


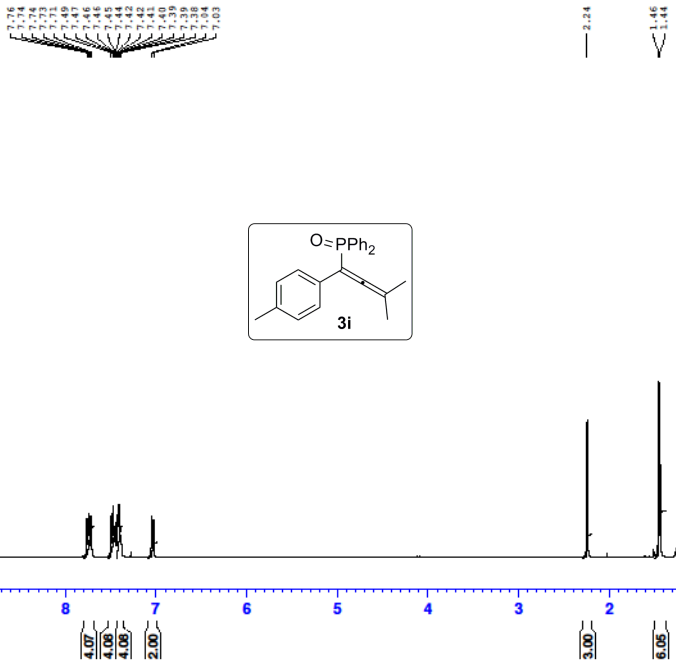

O=PPh2
3e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

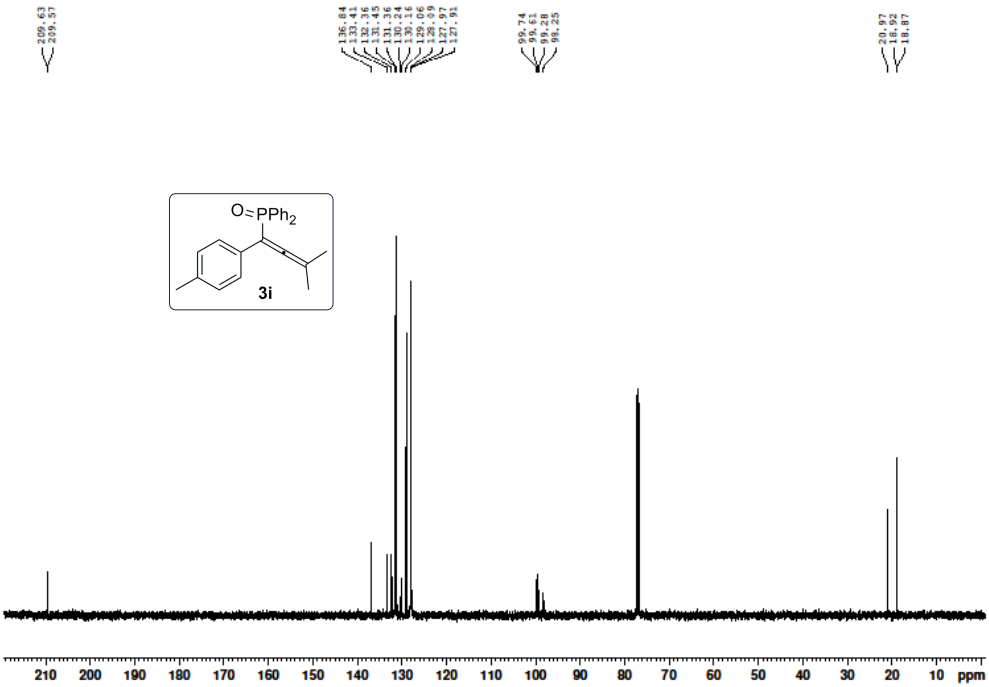

31.295
O=PPh2
3e
90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15 ppm

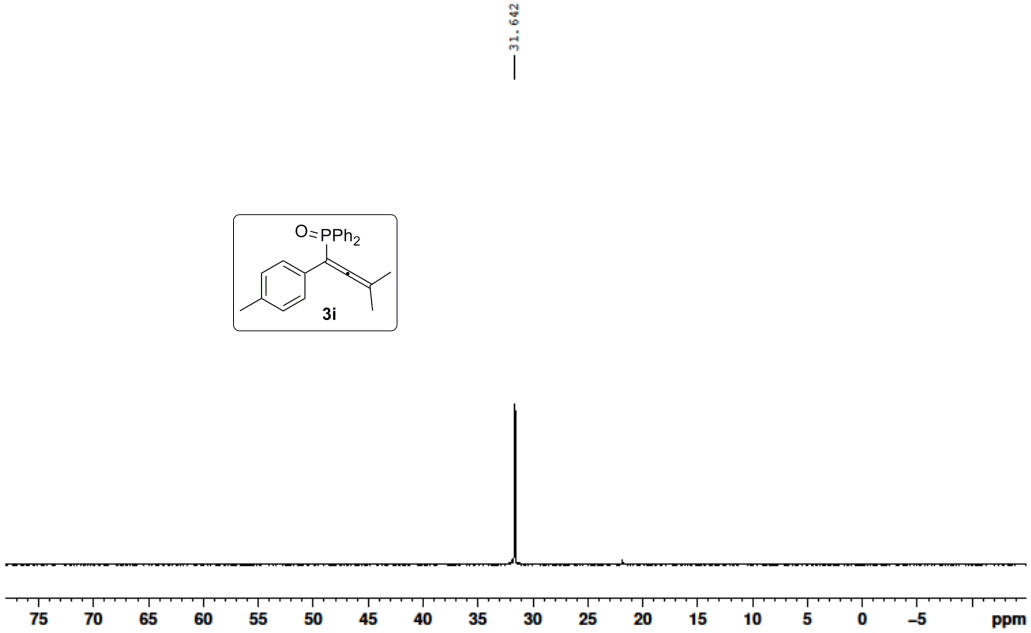

O=PPh2
Ph
3h
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

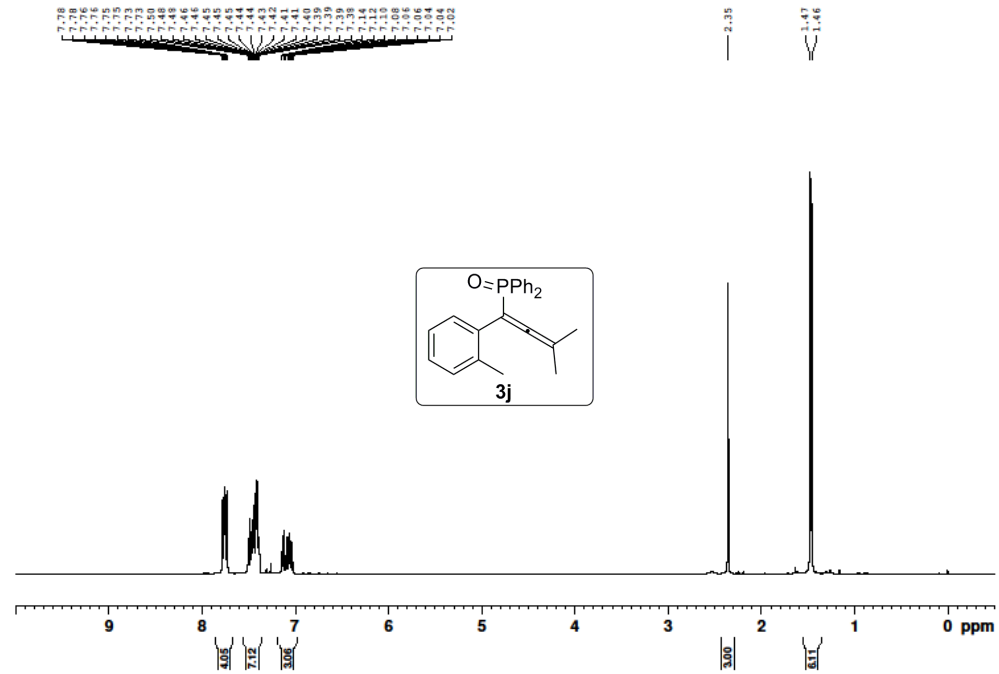

212.94
212.88
132.81
132.43
131.98
131.85
131.77
131.74
131.65
131.55
131.54
131.44
131.37
129.69
128.64
128.30
128.26
128.21
128.13
128.09
127.91
127.81
126.86
126.85
105.86
84.89
84.11
98.28
O=PPh2
3h
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

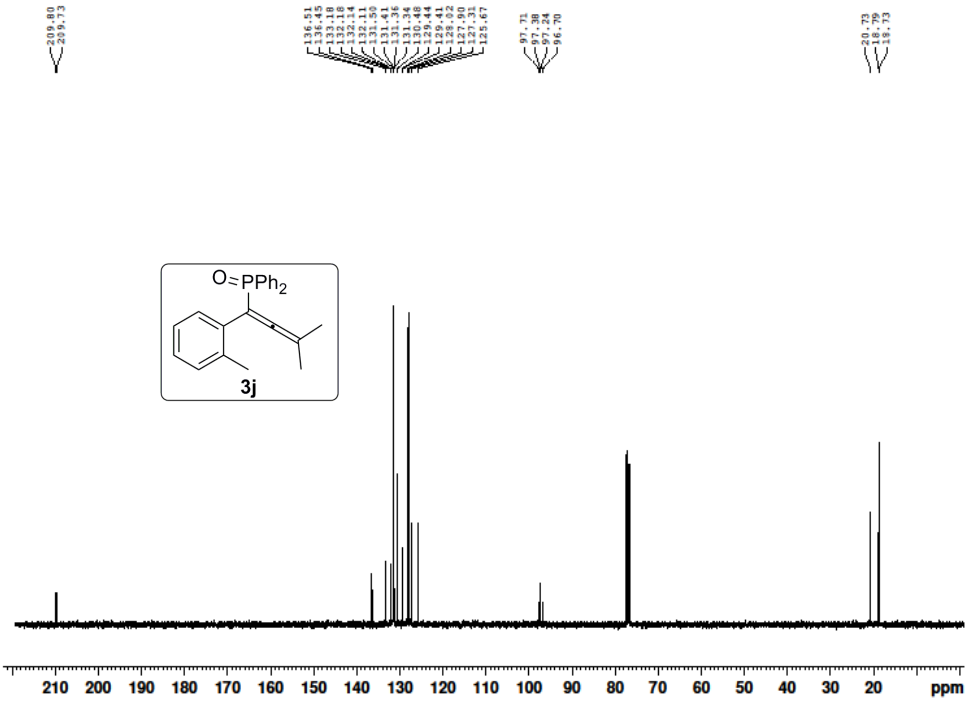

29.707
O=PPh2
3h
Ph
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
−60
ppm

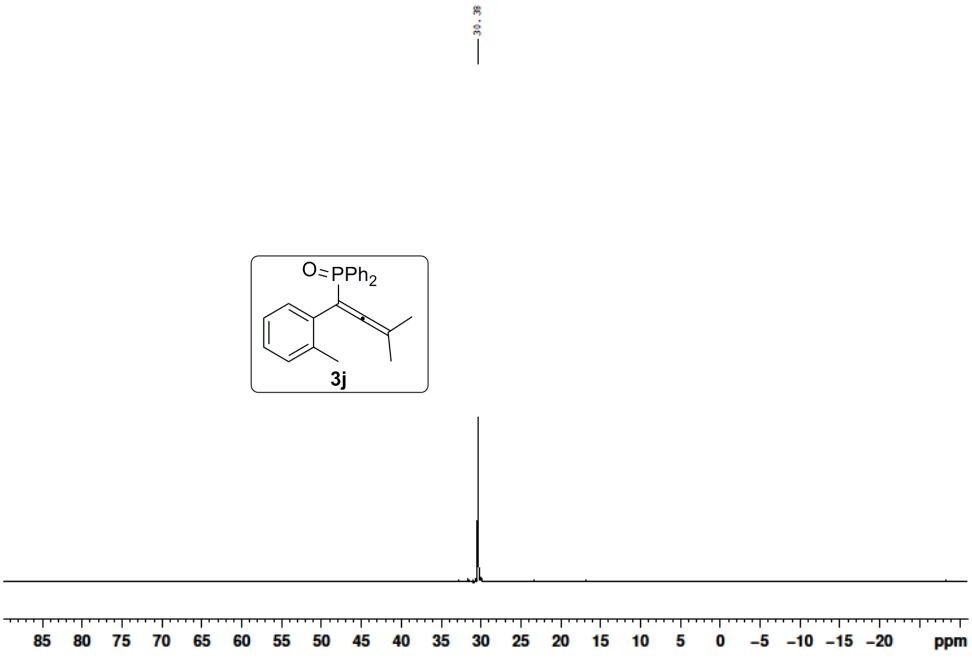

7.76
7.74
7.74
7.73
7.71
7.49
7.47
7.46
7.46
7.45
7.44
7.42
7.42
7.41
7.40
7.39
7.39
7.38
7.03
2.24
1.46
1.44
O≈PPh2
3i
9
8
7
6
5
4
3
2
1
0
ppm
4.07
4.08
4.06
2.00
3.00
6.05

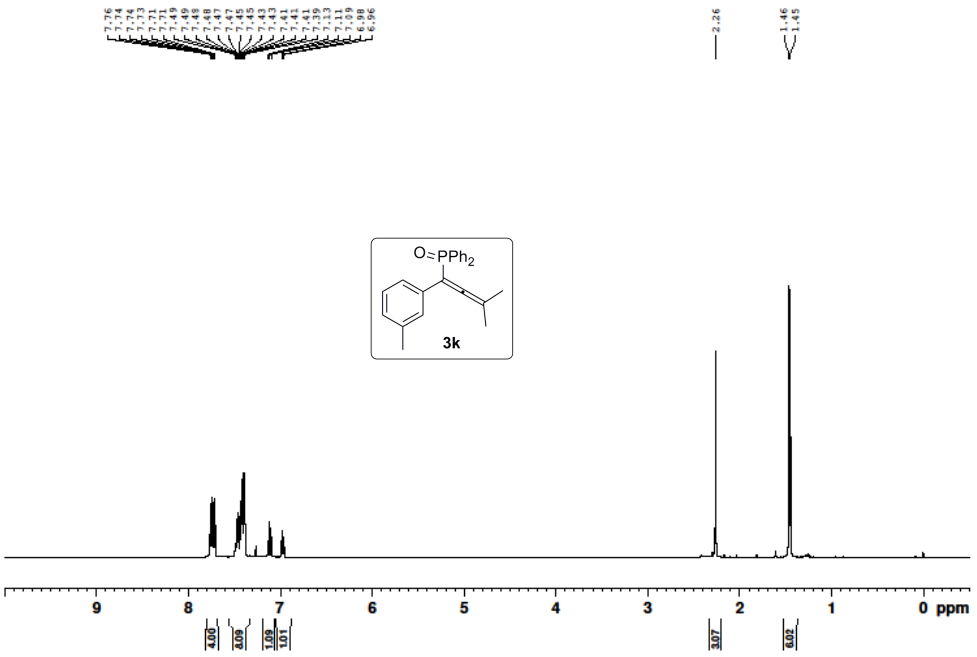

209.63
209.57
136.84
133.41
132.36
131.45
131.36
130.24
130.16
129.06
128.99
127.97
127.91
127.78
99.74
99.61
99.28
99.25
20.97
16.92
16.87
O≈PPh2
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

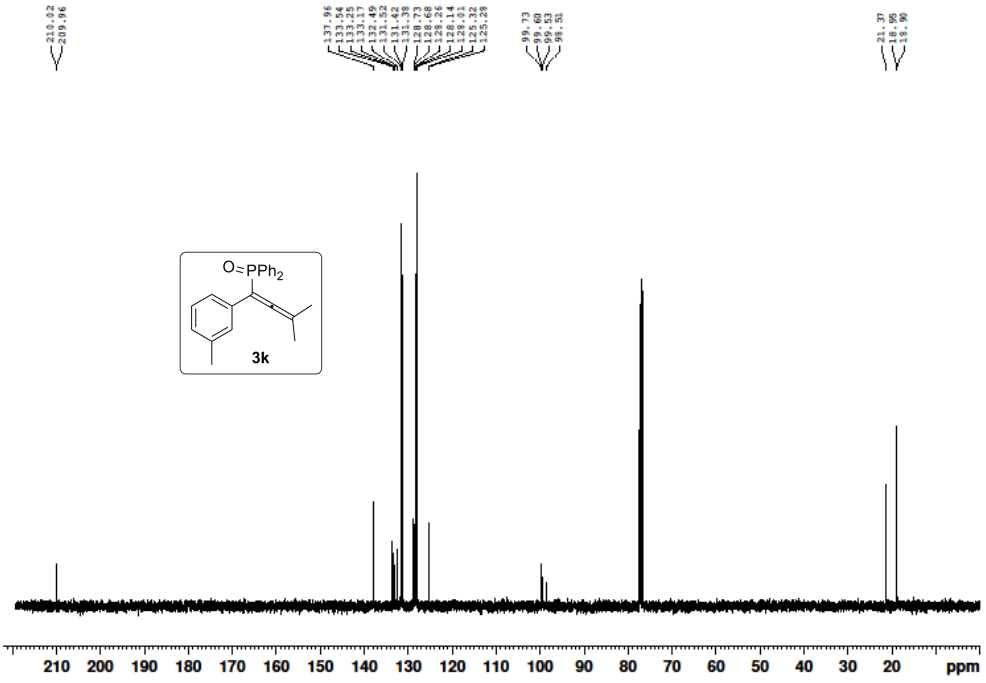

31.642
O=PPh2
3i
75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 ppm

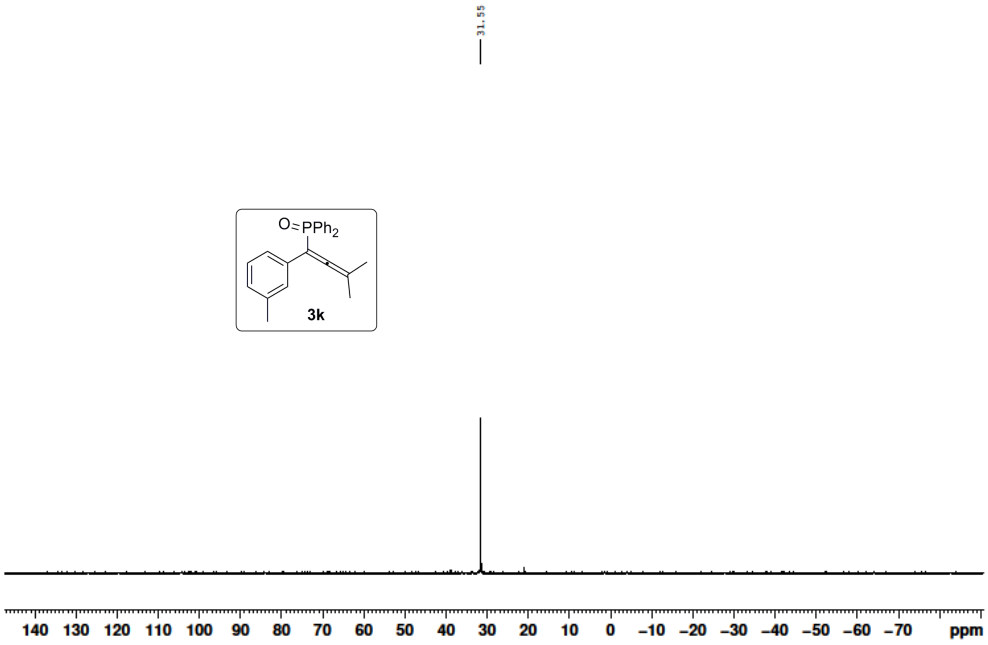

2.35
1.47
1.46
O=PPh2
3j
9 8 7 6 5 4 3 2 1 0 ppm
4.05
7.12
3.06
3.00
6.11

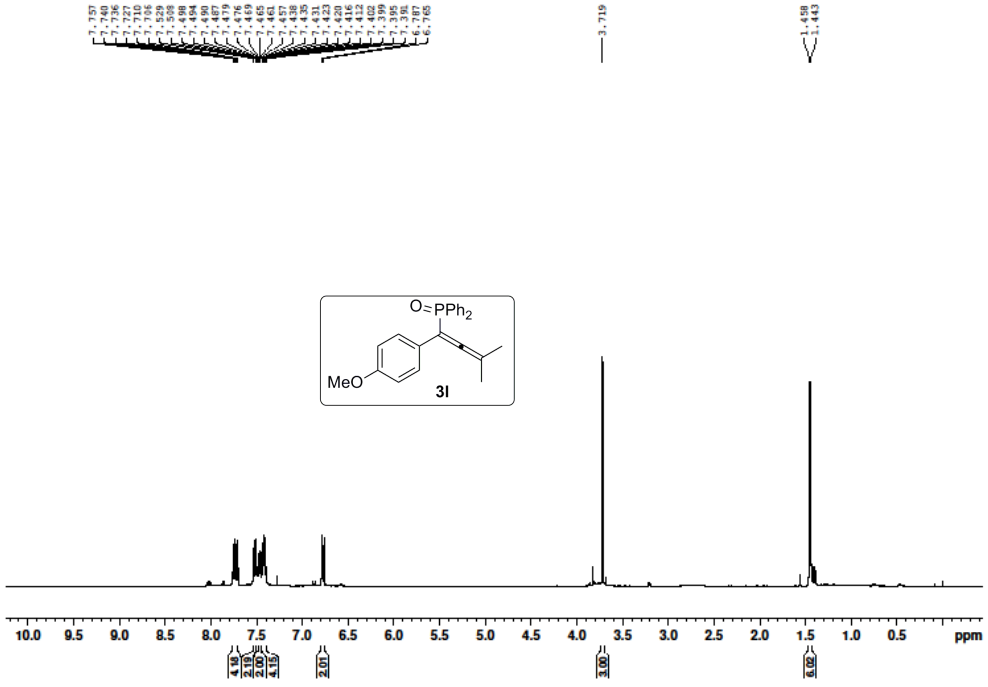

O=PPh2
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

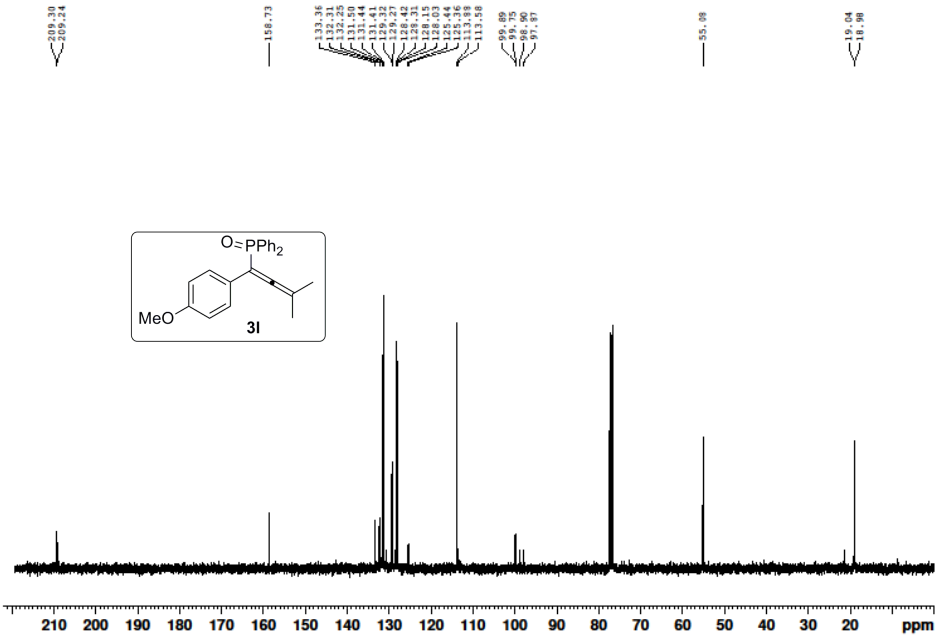

O=PPh2
3j
85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 ppm

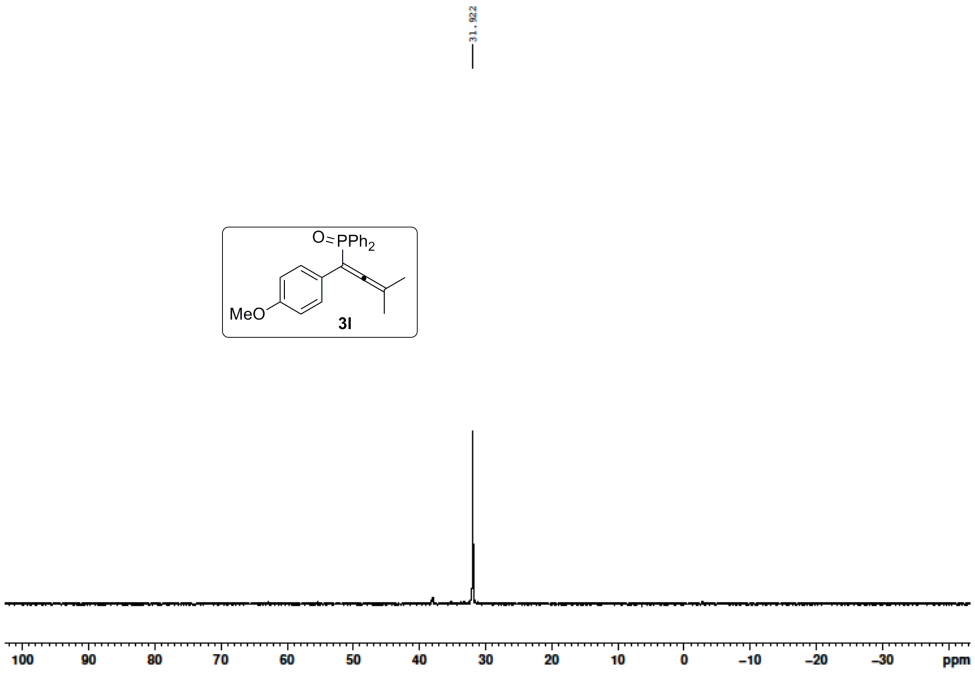

O=PPh2
3k
9
8
7
6
5
4
3
2
1
0 ppm

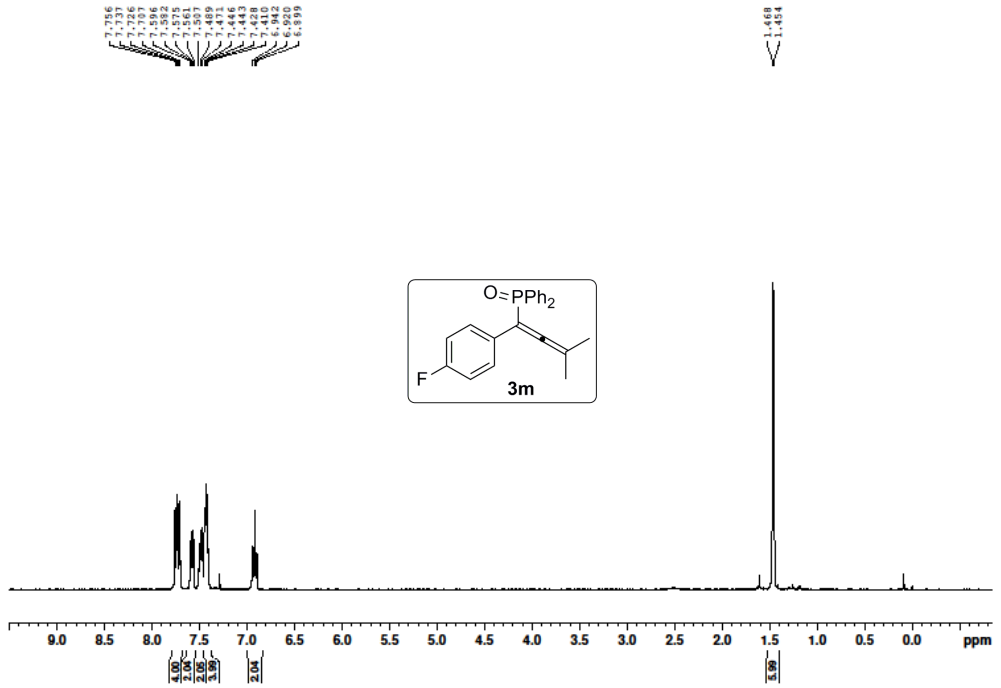

O=PPh2
3k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

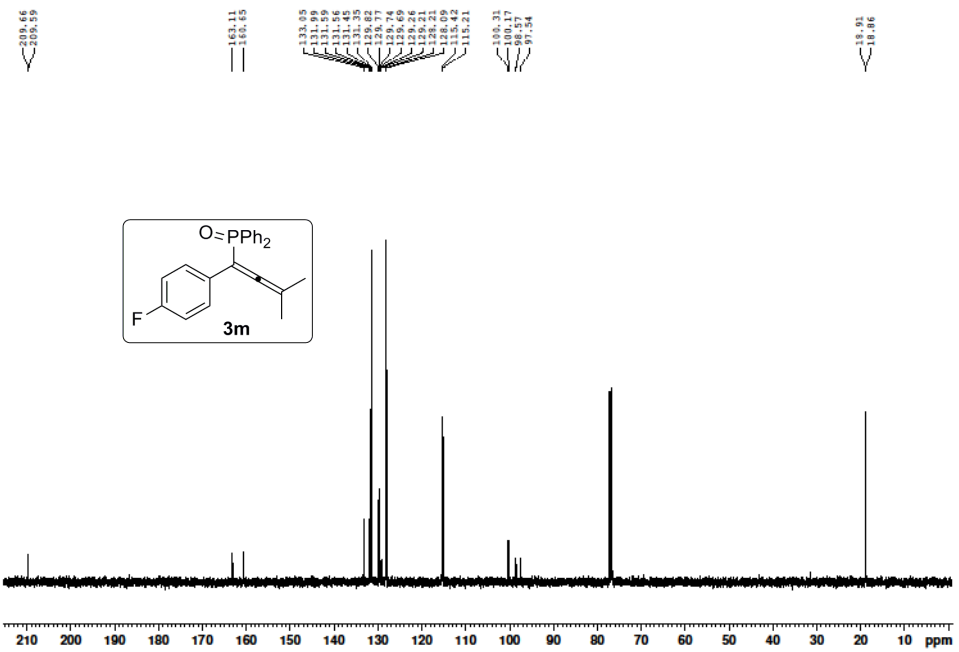

31.55
O≈PPh2
3k
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 −60 −70 ppm

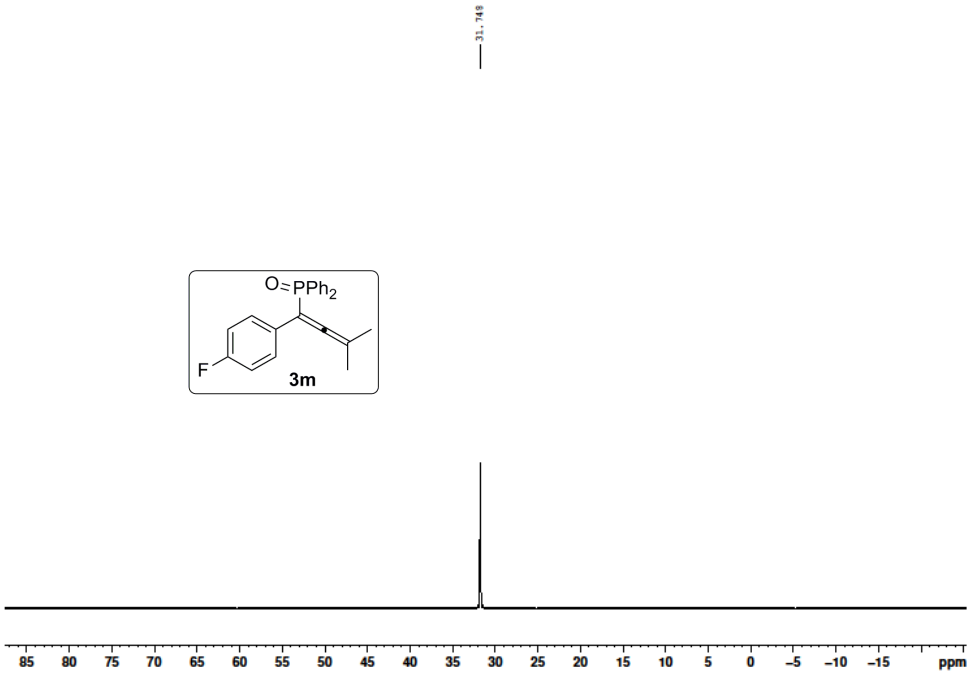

7.757
7.740
7.736
7.727
7.710
7.706
7.539
7.528
7.498
7.494
7.490
7.487
7.479
7.476
7.469
7.465
7.461
7.457
7.438
7.435
7.431
7.423
7.420
7.416
7.412
7.402
7.399
7.395
7.391
6.787
6.765
3.719
1.458
1.443
O≈PPh2
MeO
3l
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.19
2.19
2.05
4.15
2.01
3.00
6.00

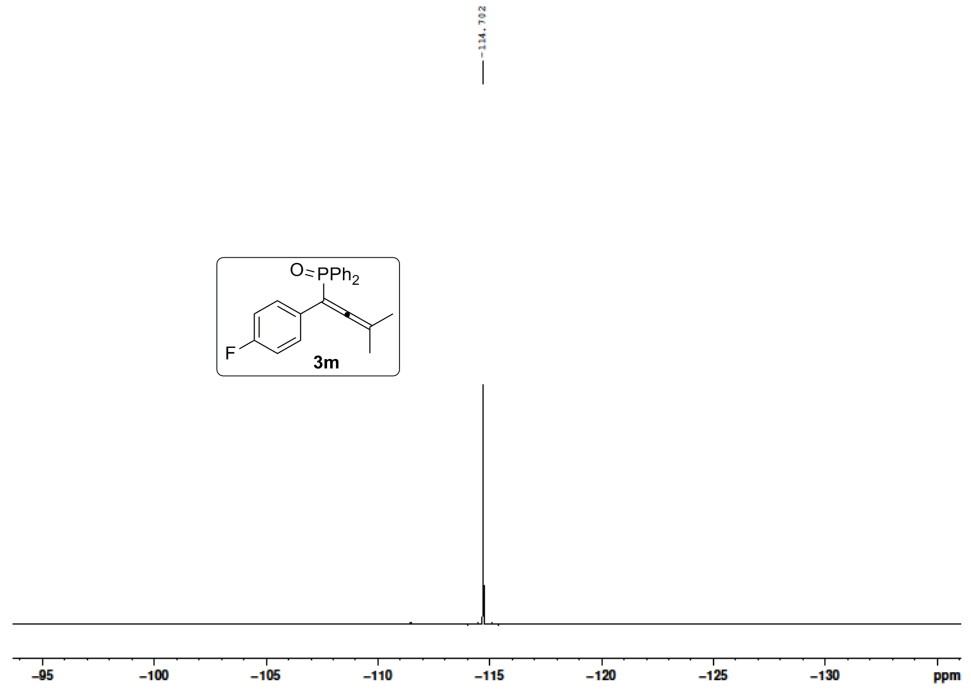

209.30
209.24
158.73
133.36
132.31
132.25
131.50
131.44
131.41
129.32
129.27
128.42
128.31
128.15
128.03
125.56
125.46
113.98
113.58
99.89
99.75
98.90
97.97
55.08
19.04
18.98
O≈PPh2
MeO
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

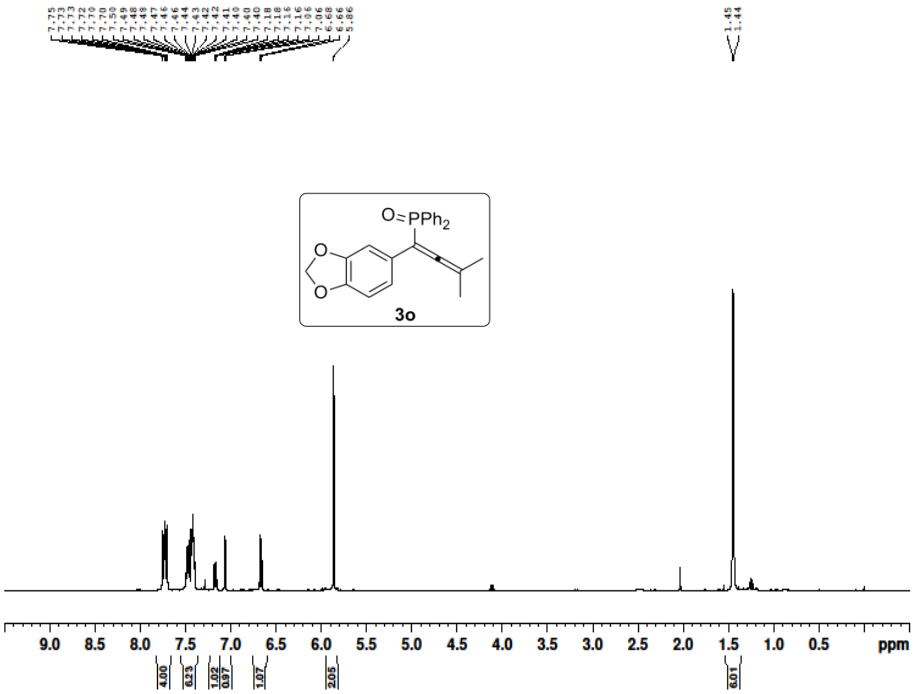

31.922
O≈PPh2
MeO
3l
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 ppm

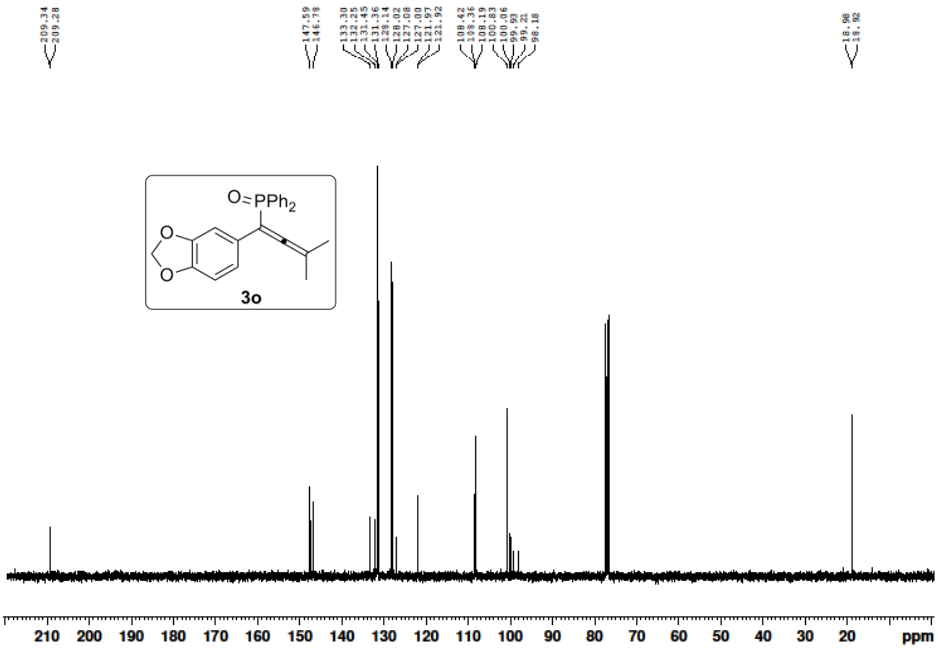

O=PPh2
F
3m
ppm

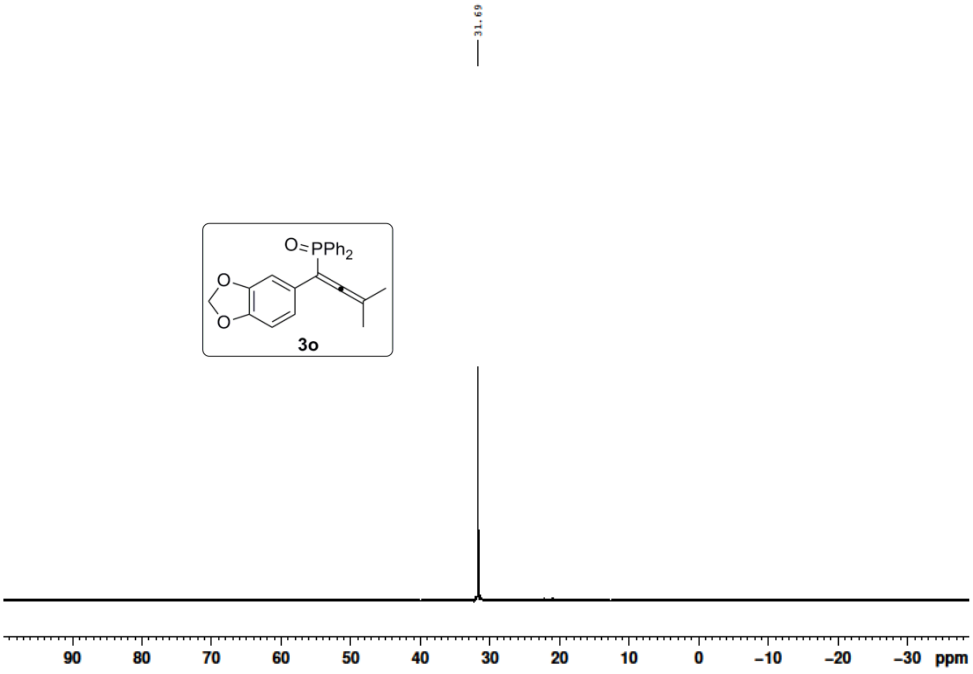

O=PPh2
F
3m
ppm

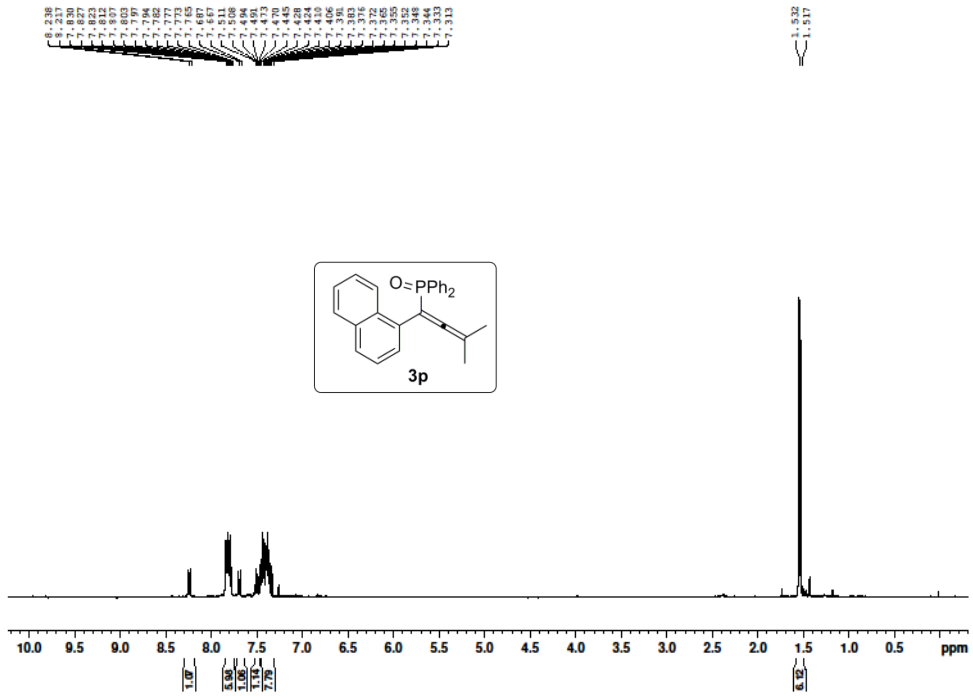

31.748
O≈PPh2
F
3m
85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 –5 –10 –15 ppm

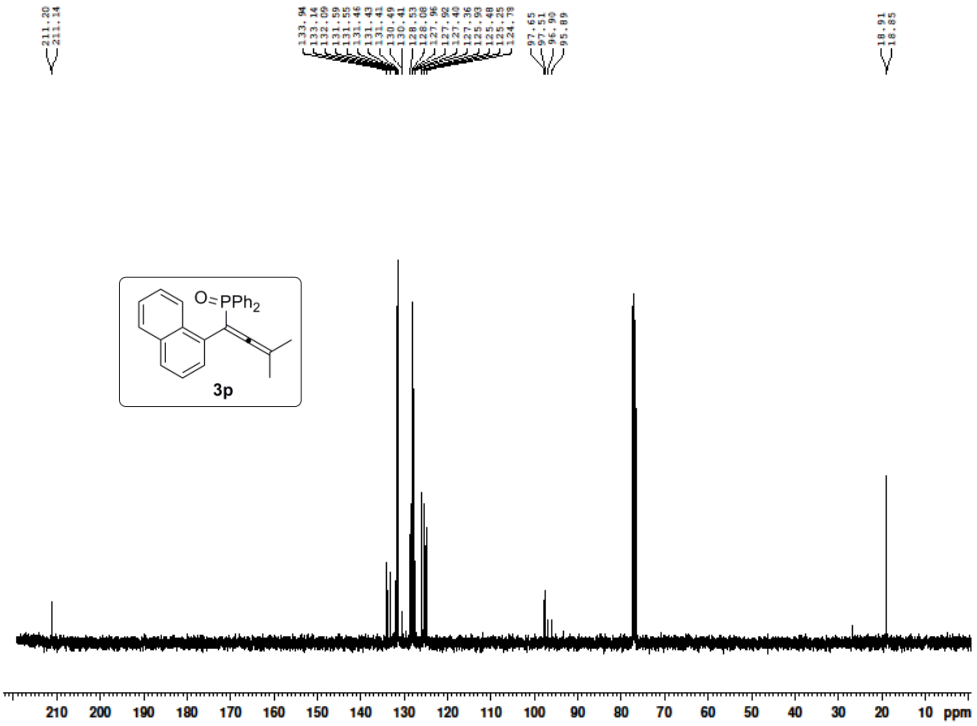

-114.702
O≈PPh2
F
3m
–95 –100 –105 –110 –115 –120 –125 –130 ppm

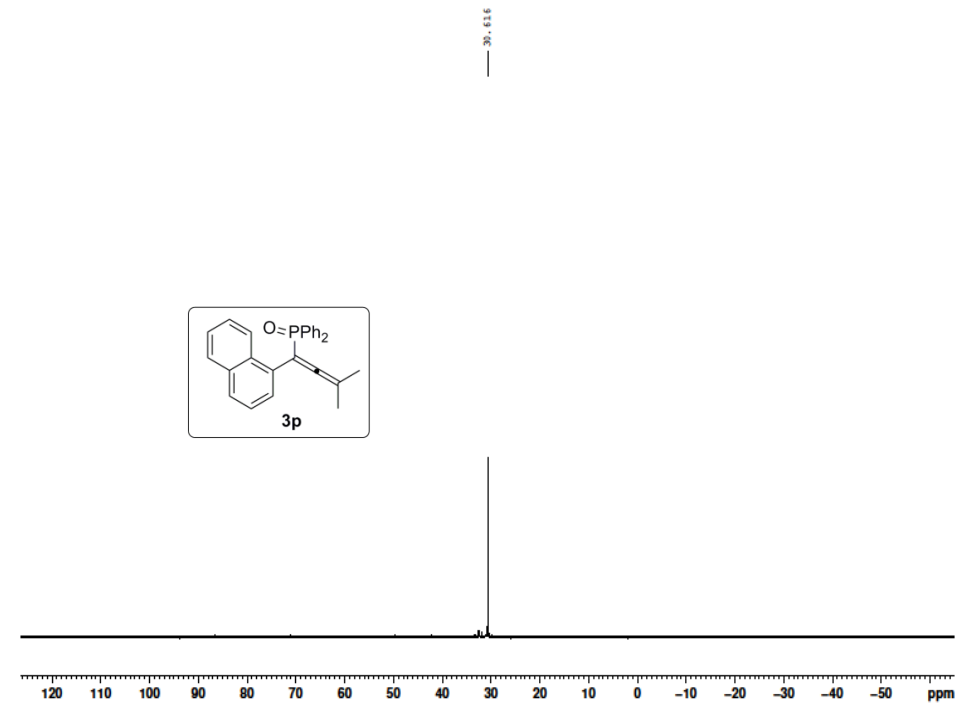

O=PPh2
3o
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.00
6.25
1.02
0.97
1.07
2.05
6.01

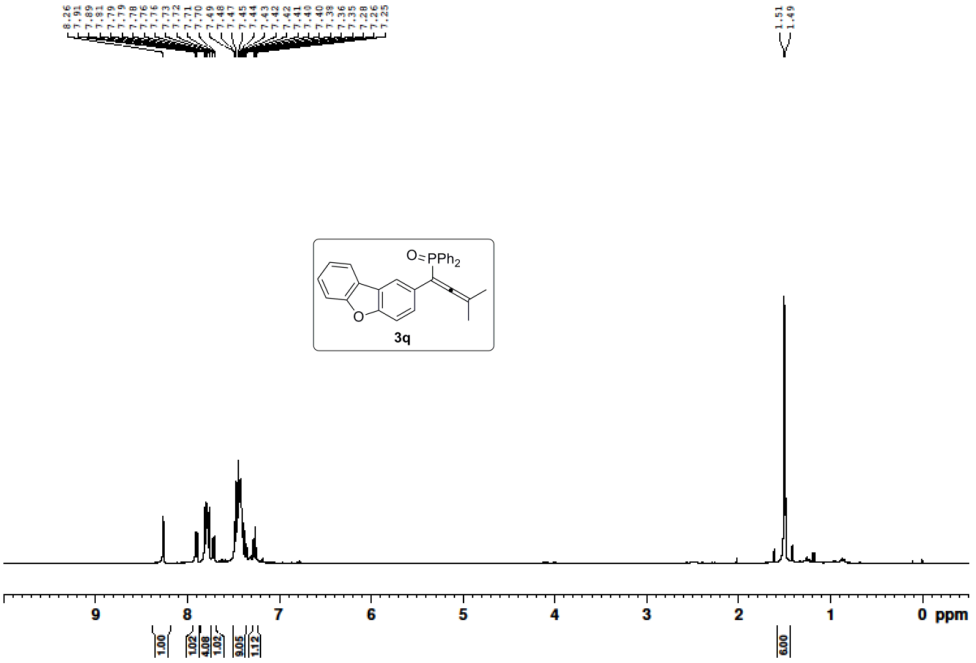

O=PPh2
3o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

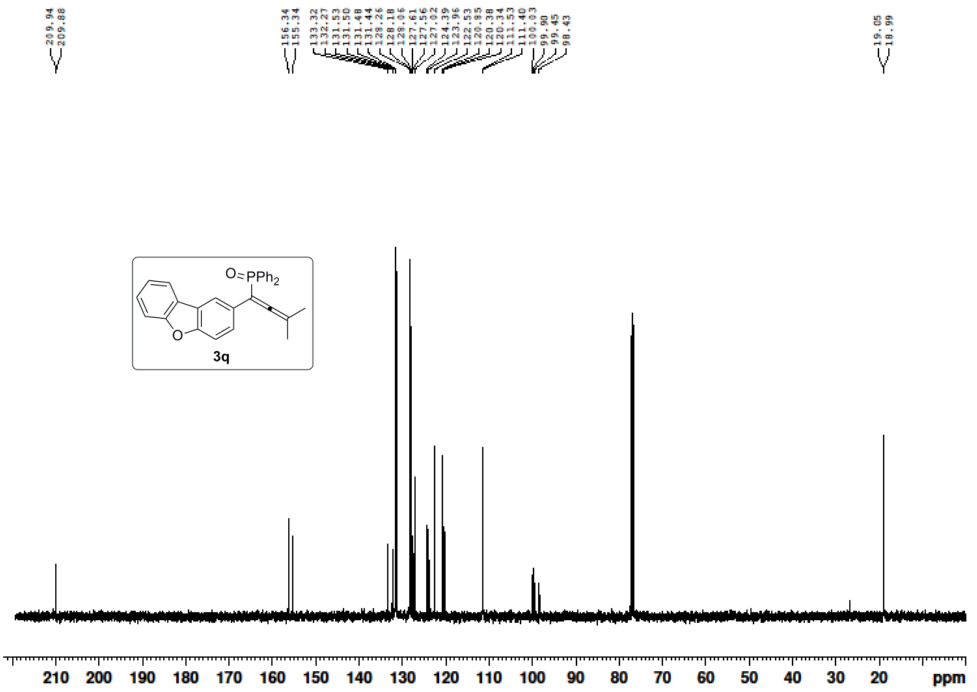

31.69
O≃PPh2
3o
90 80 70 60 50 40 30 20 10 0 −10 −20 −30 ppm

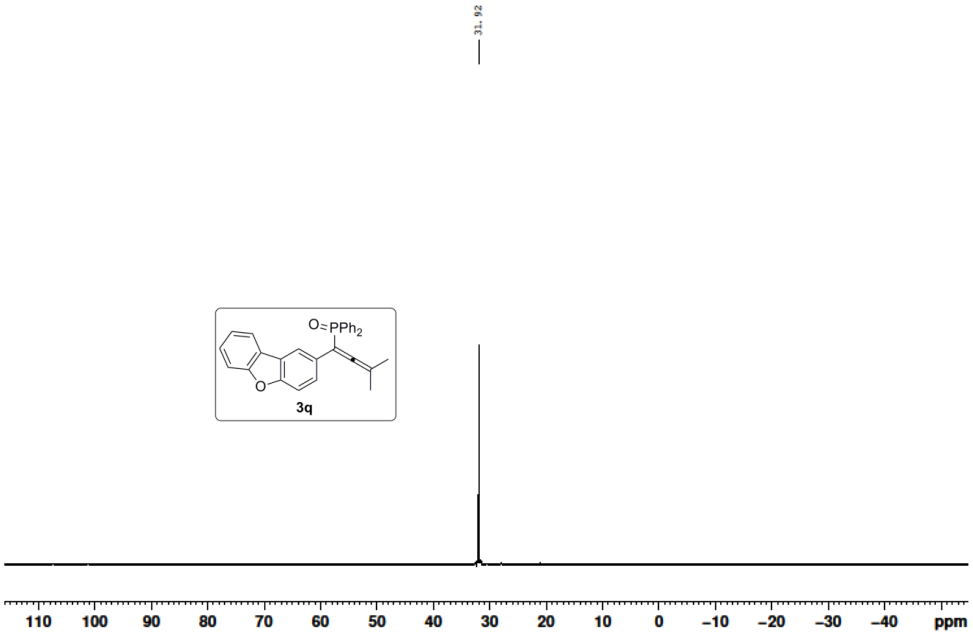

8.238
8.217
7.830
7.827
7.823
7.812
7.807
7.803
7.797
7.794
7.782
7.777
7.773
7.765
7.687
7.667
7.511
7.508
7.494
7.491
7.473
7.470
7.445
7.428
7.414
7.410
7.406
7.391
7.383
7.376
7.372
7.365
7.355
7.353
7.348
7.344
7.333
7.313
1.532
1.517
O≃PPh2
3p
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
5.93
1.06
1.15
7.79
6.12

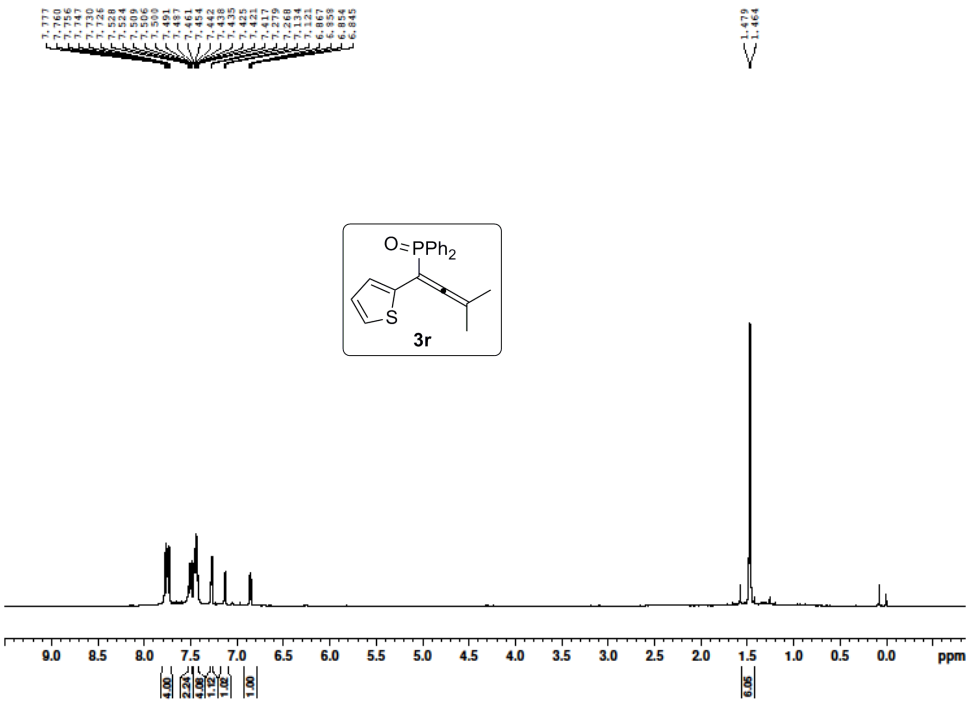

211.20
211.14
133.94
133.14
132.09
131.59
131.58
131.46
131.43
131.41
130.49
130.41
128.53
128.08
127.96
127.40
127.36
125.93
125.68
125.25
124.78
97.65
97.51
96.99
95.89
18.91
18.85
O≈PPh2
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

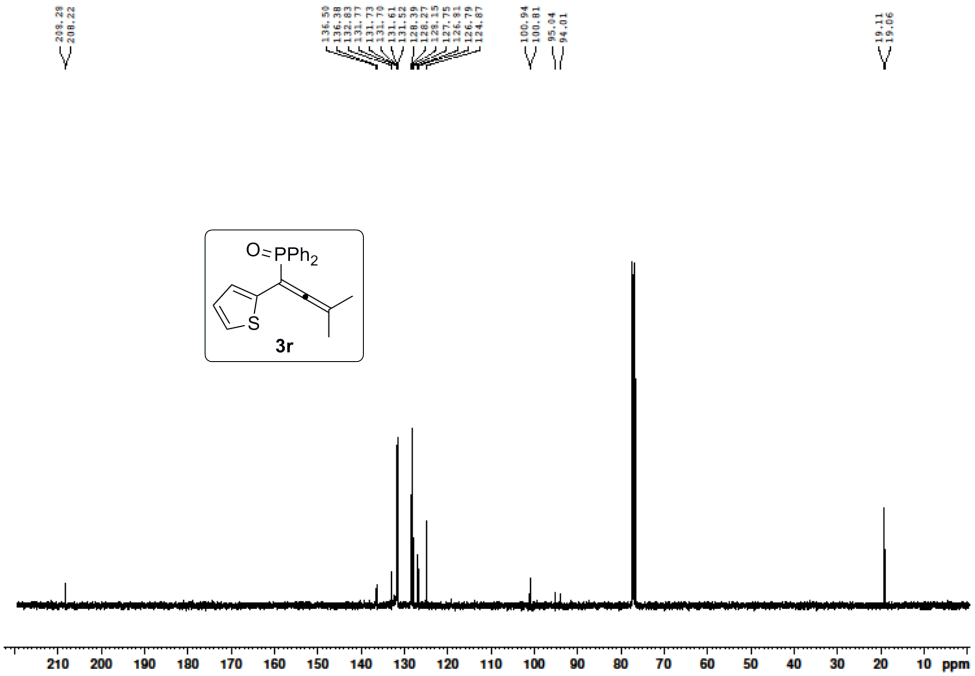

30.616
O≈PPh2
3p
120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm

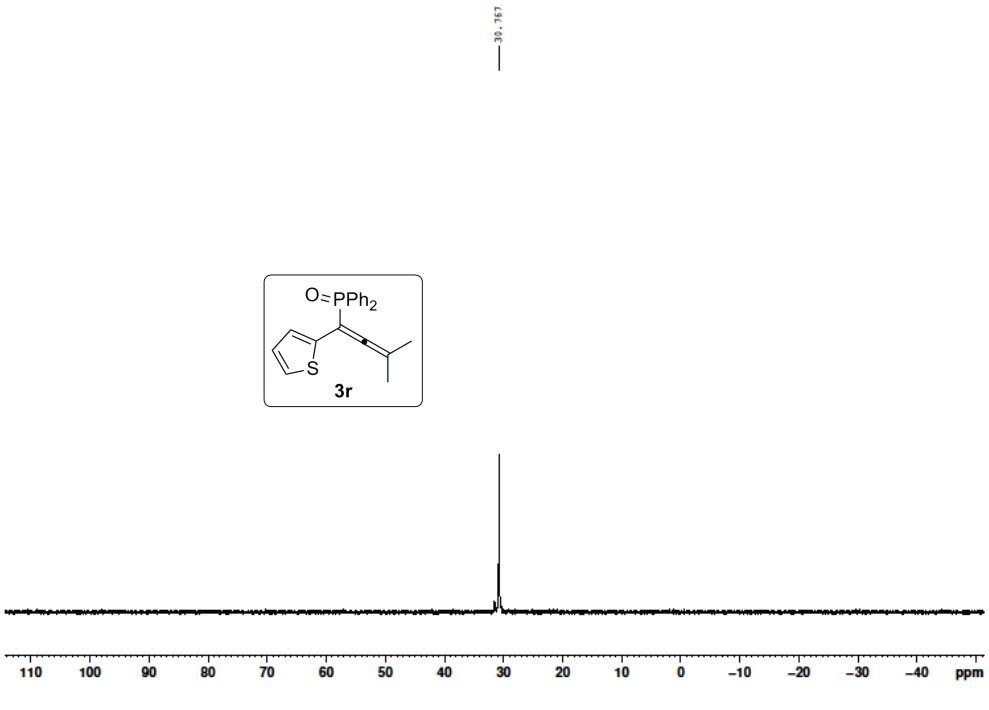

8.26
7.91
7.89
7.81
7.79
7.78
7.76
7.73
7.72
7.71
7.70
7.48
7.47
7.45
7.44
7.43
7.42
7.41
7.40
7.40
7.38
7.36
7.35
7.28
7.26
7.25
1.51
1.49
O≈PPh2
3q
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.02
4.08
1.05
8.05
1.12
6.00

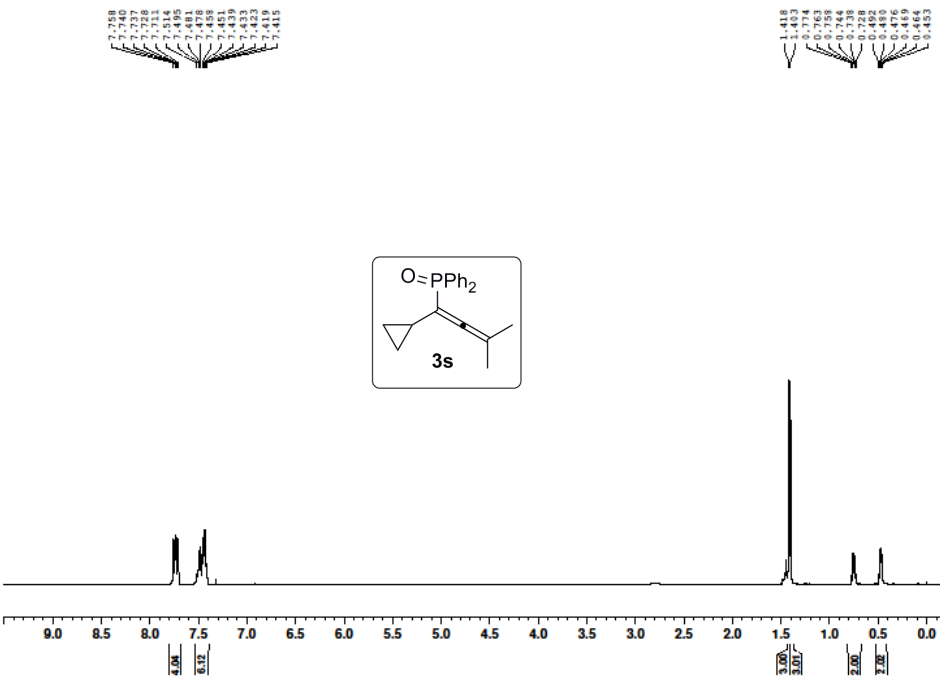

209.94
209.88
156.34
155.34
133.32
132.27
131.53
131.50
131.48
131.44
129.26
128.18
128.06
127.61
127.56
127.02
124.39
123.96
122.53
120.95
120.38
120.34
111.53
111.40
99.83
99.80
99.45
98.43
19.05
18.99
O≈PPh2
3q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

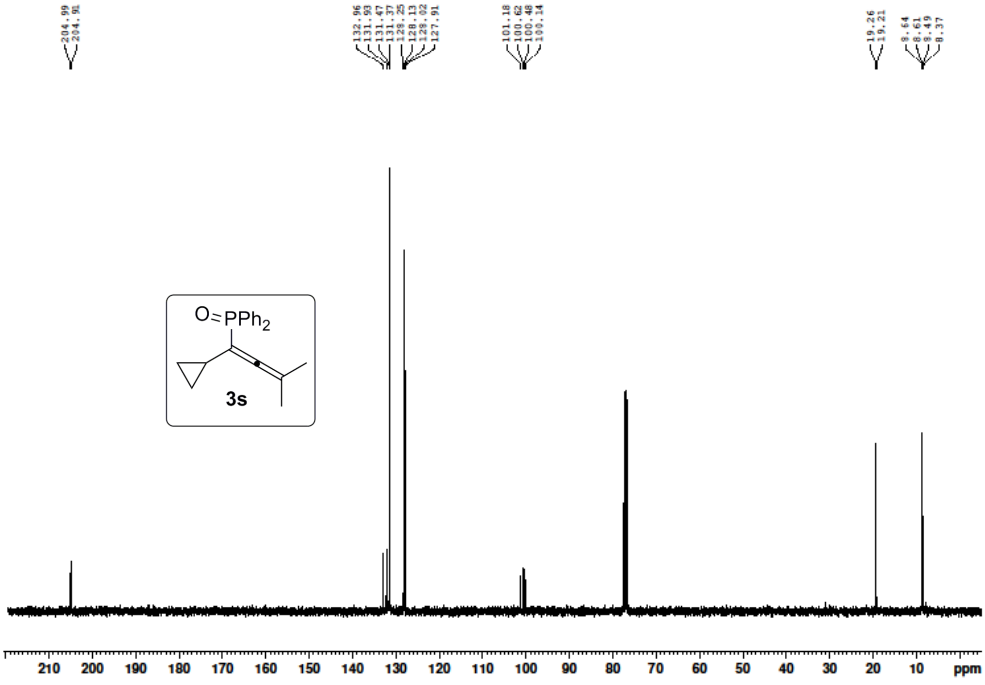

31.92
O≃PPh2
O
3q
110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 ppm

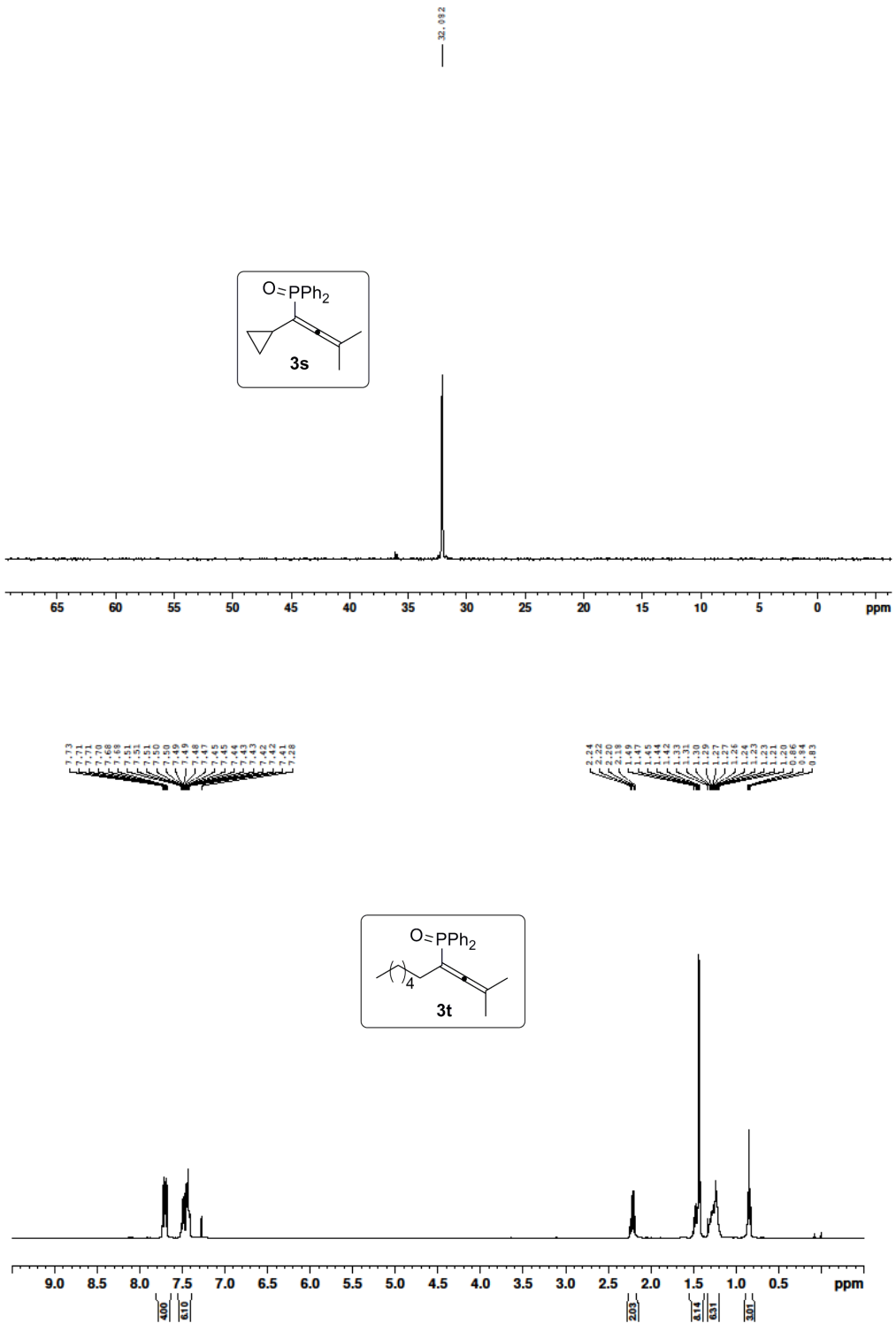

7.777
7.760
7.756
7.747
7.730
7.726
7.524
7.509
7.506
7.490
7.481
7.461
7.454
7.442
7.438
7.435
7.425
7.421
7.417
7.279
7.268
7.134
7.121
6.867
6.858
6.854
6.845
1.479
1.464
O≃PPh2
S
3r
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
4.00
2.24
4.00
1.19
1.08
1.00
6.06

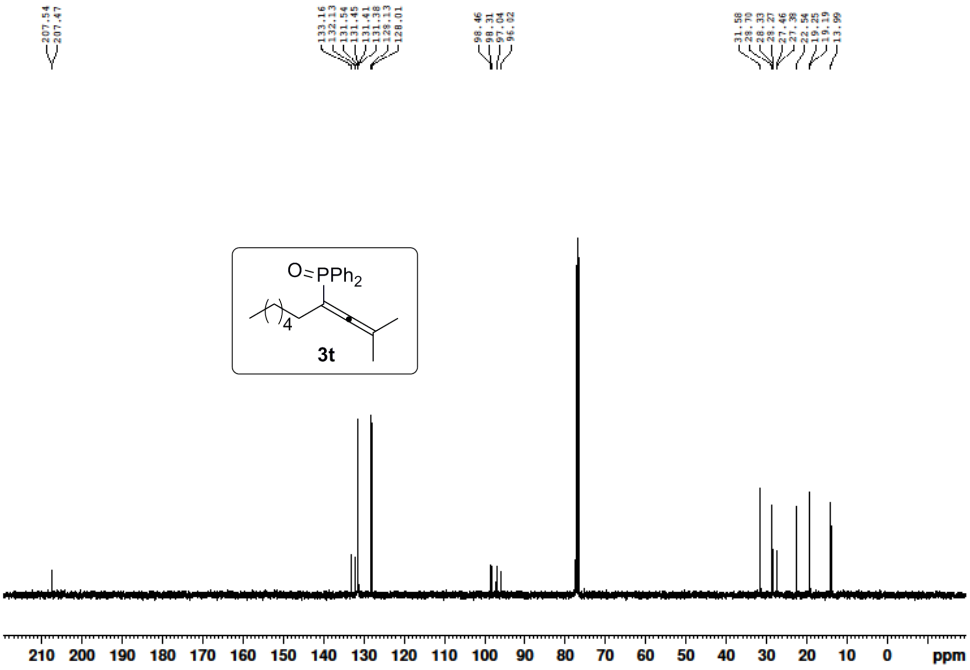

209.28
208.22
136.50
135.38
132.83
131.77
131.73
131.70
131.61
131.52
128.39
128.27
128.15
127.75
126.91
126.79
124.87
100.94
100.81
95.84
94.01
19.11
19.06
O≃PPh2
S
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

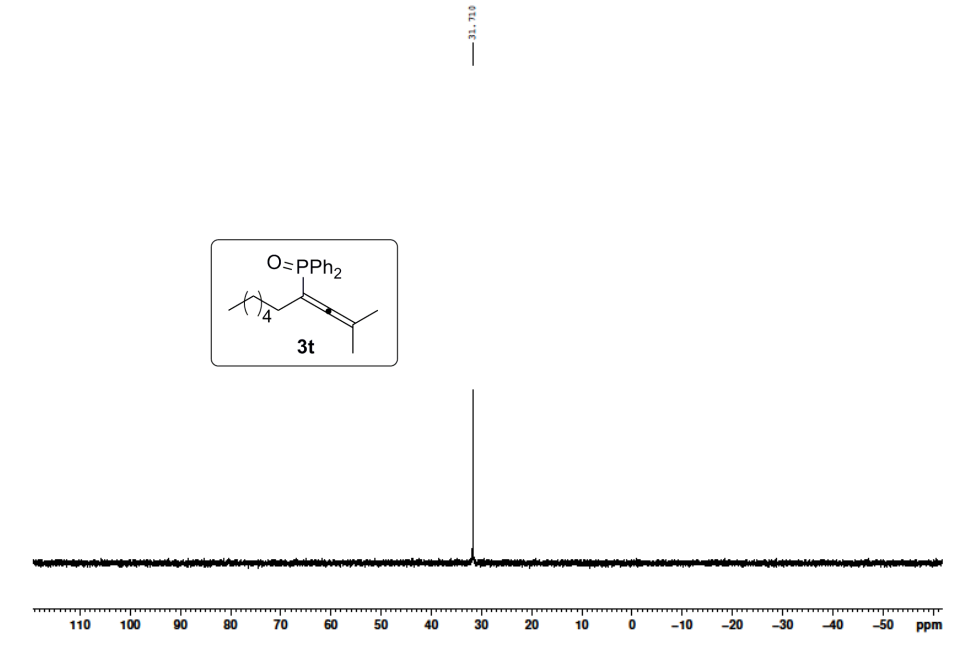

30.767
O≃PPh2
S
3r
110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 ppm

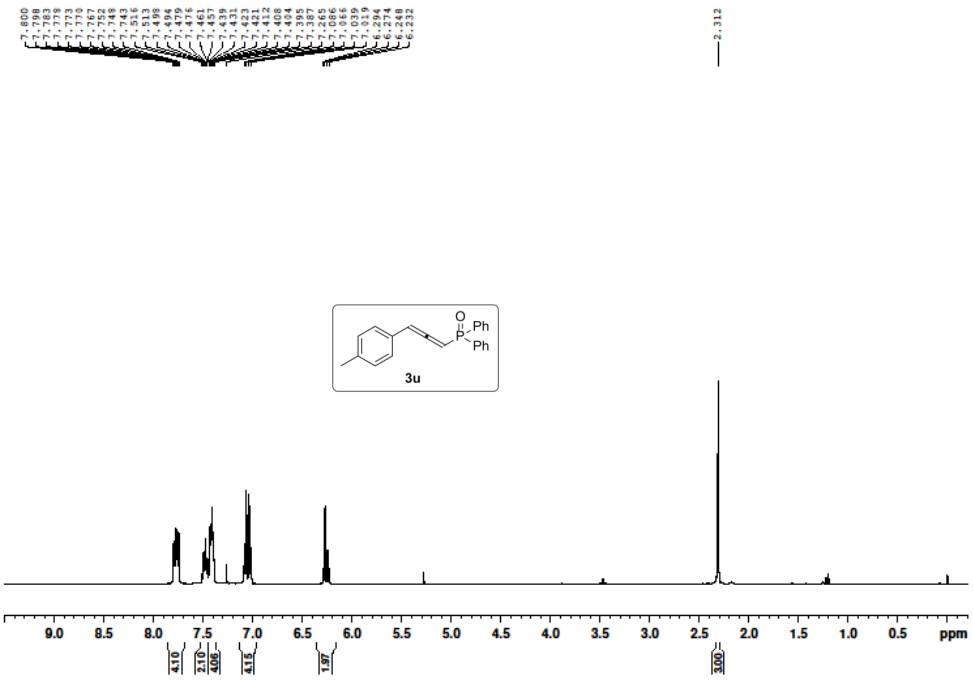

7.758
7.740
7.737
7.728
7.511
7.514
7.495
7.481
7.478
7.458
7.451
7.439
7.433
7.423
7.419
7.415
1.418
1.403
0.774
0.763
0.758
0.744
0.738
0.728
0.492
0.482
0.476
0.469
0.464
0.453
O≃PPh2
3s
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
4.04
6.12
3.00
3.07
2.00
1.08

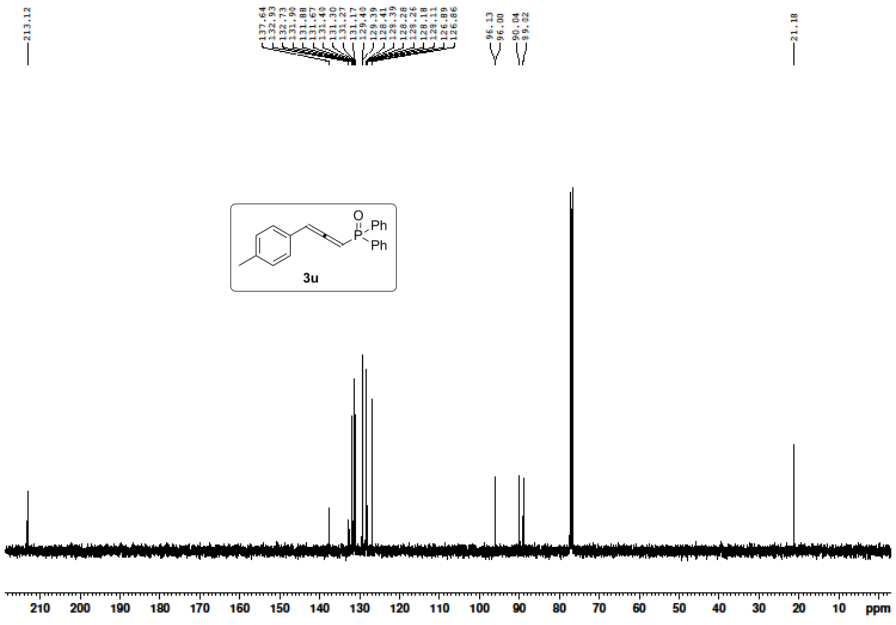

204.99
204.91
132.96
131.69
131.47
131.37
128.25
128.13
128.02
127.91
101.18
100.62
100.48
100.34
19.26
19.21
9.64
8.61
8.49
8.37
O≃PPh2
3s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

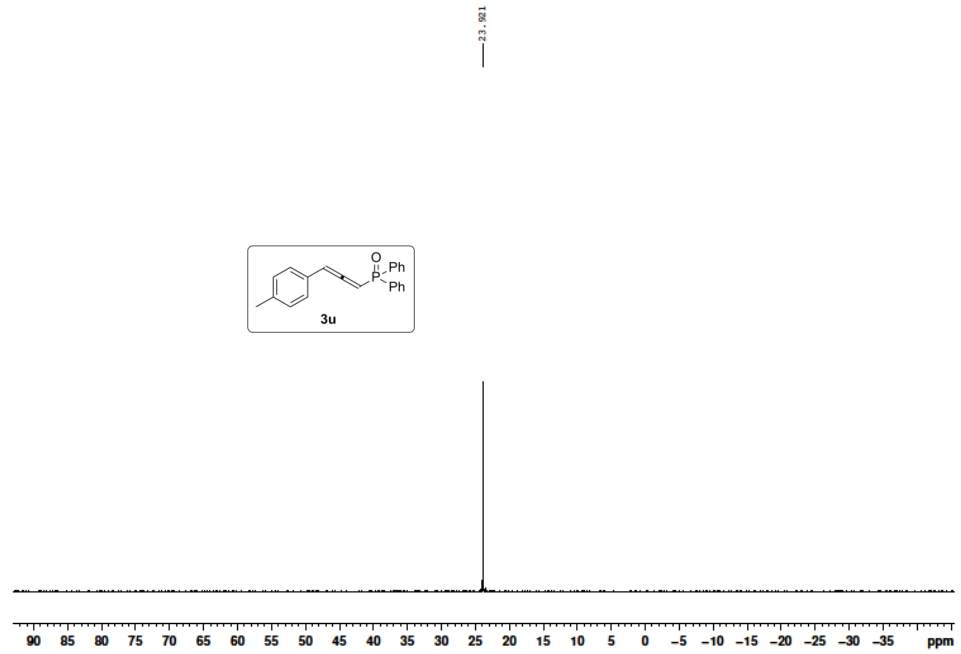

32.092
O≃PPh2
3s
65 60 55 50 45 40 35 30 25 20 15 10 5 0 ppm
O≃PPh2
4
3t
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.00
6.10
2.03
8.14
6.51
3.01

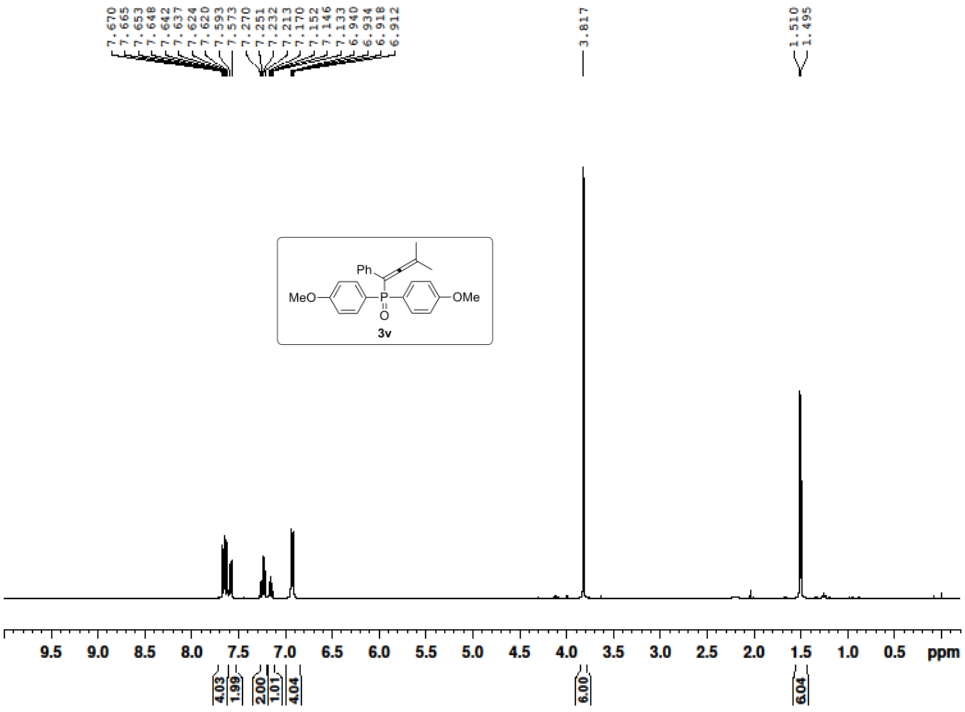

207.54
207.47
133.16
132.13
131.54
131.45
131.41
131.38
128.13
128.01
98.46
98.31
97.04
96.02
31.58
28.70
28.33
28.27
27.46
27.38
22.54
19.25
19.19
13.99
O=PPh2
4
3t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

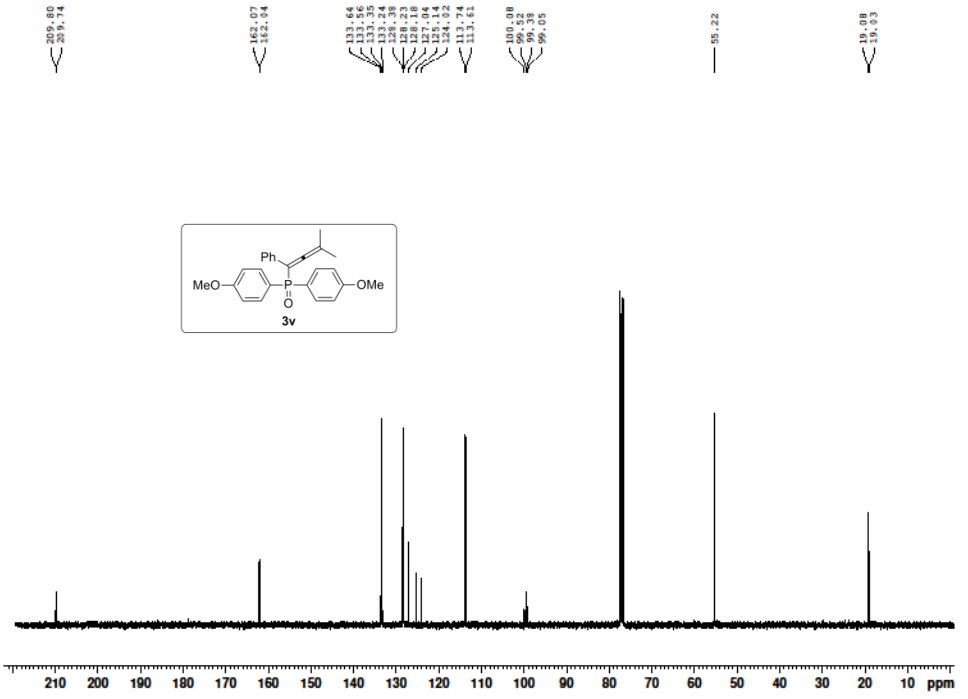

31.710
O=PPh2
4
3t
110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 ppm

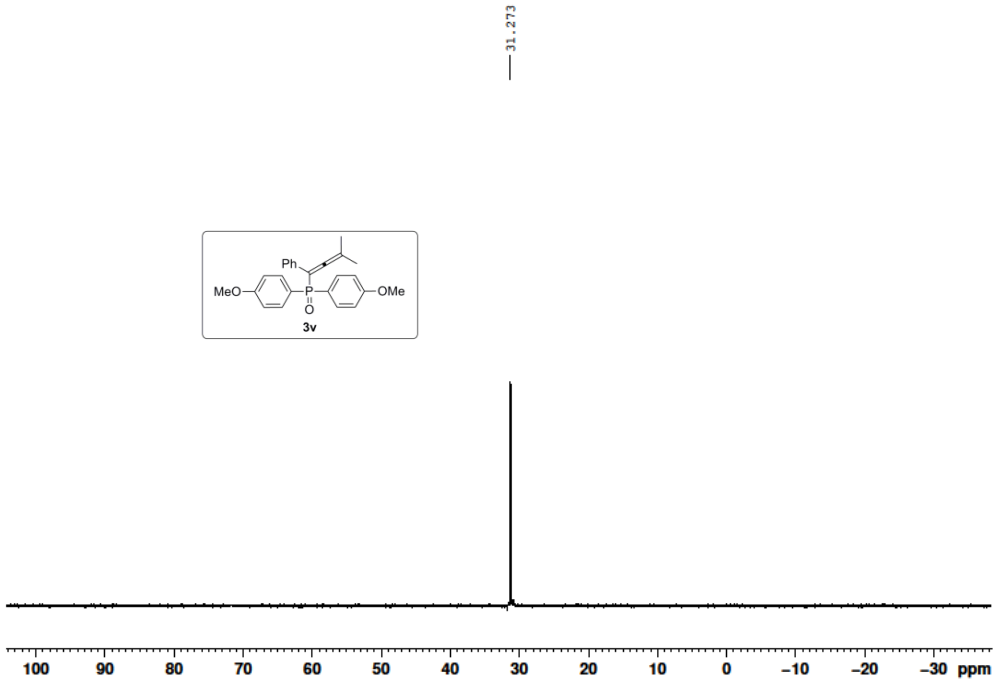

3u
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.10
2.10
4.06
4.15
1.97
3.00

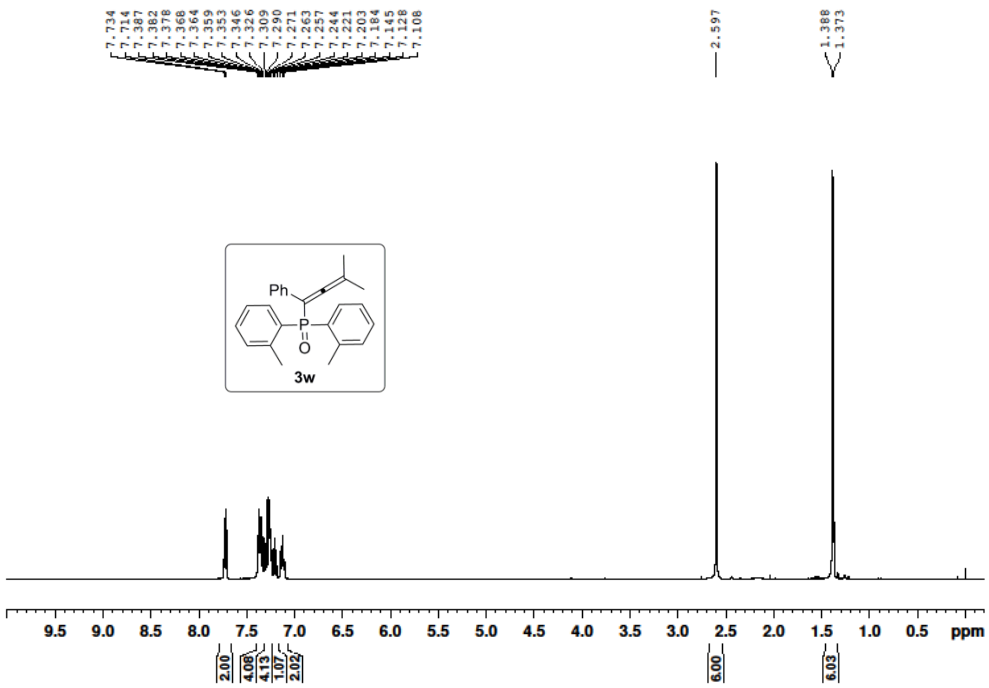

3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

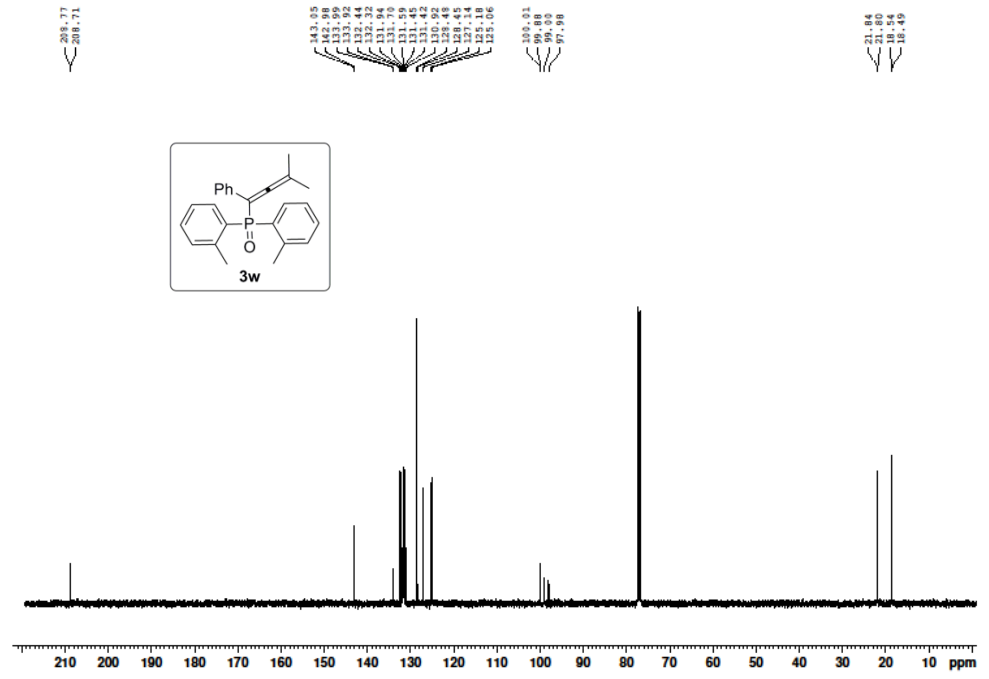

23.921
3u
90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 −5 −10 −15 −20 −25 −30 −35 ppm

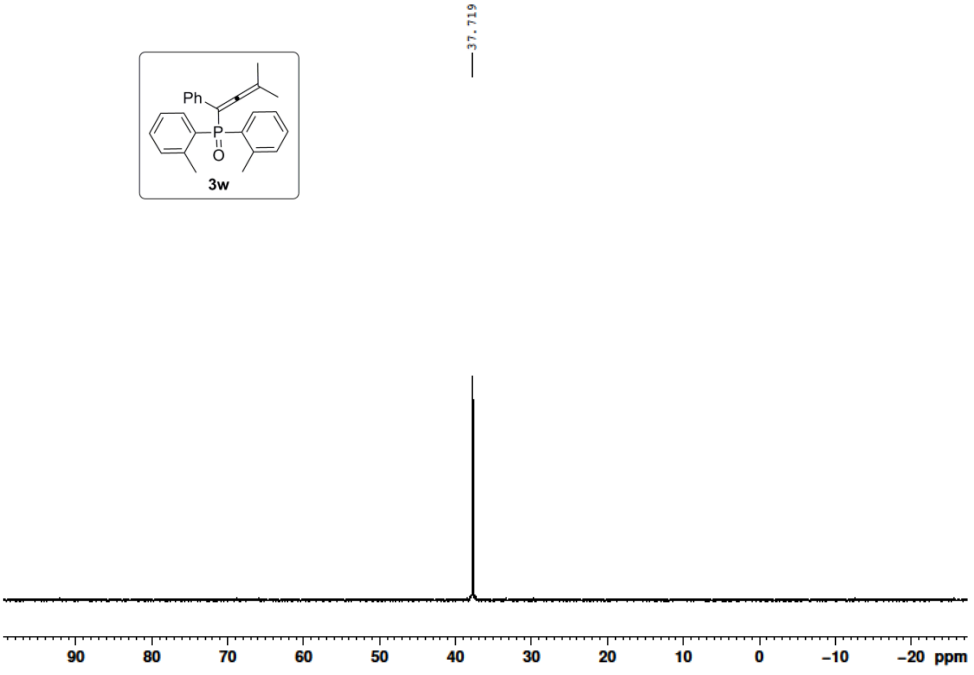

7.670
7.665
7.653
7.648
7.642
7.637
7.624
7.620
7.593
7.573
7.270
7.251
7.232
7.213
7.170
7.152
7.146
7.133
6.940
6.934
6.918
6.912
3.817
1.510
1.495
3v
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.03
1.99
2.00
1.01
4.04
6.00
6.04

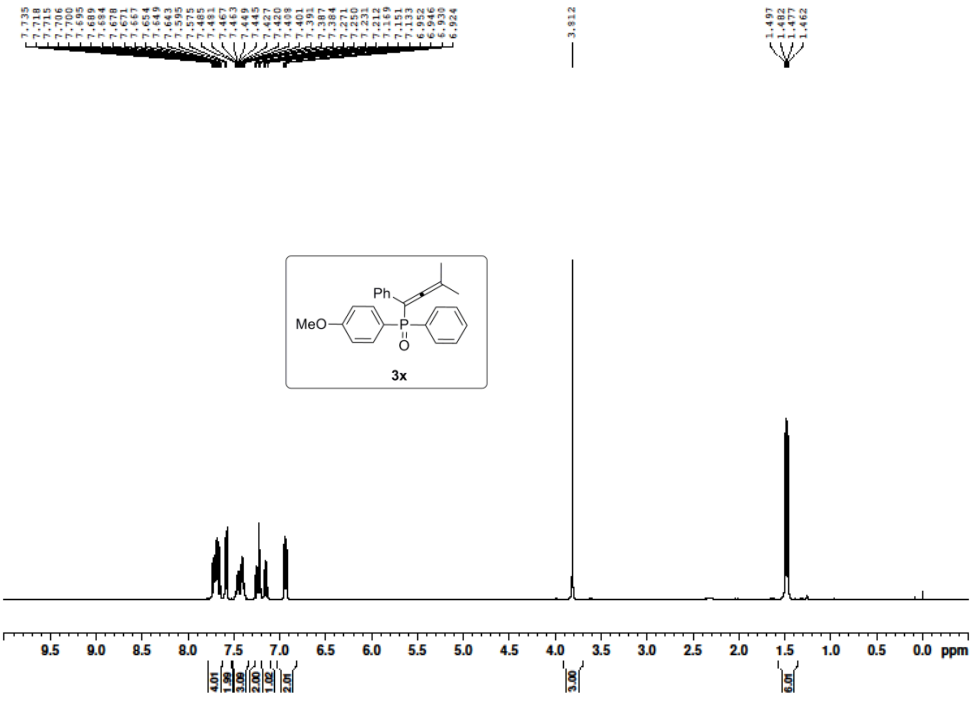

209.80
29.74
162.07
162.04
133.64
133.56
133.35
133.24
128.38
128.23
128.18
128.04
125.14
124.02
113.74
113.61
100.08
99.52
99.38
99.05
55.22
19.08
19.03
Ph
MeO
OMe
3v
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

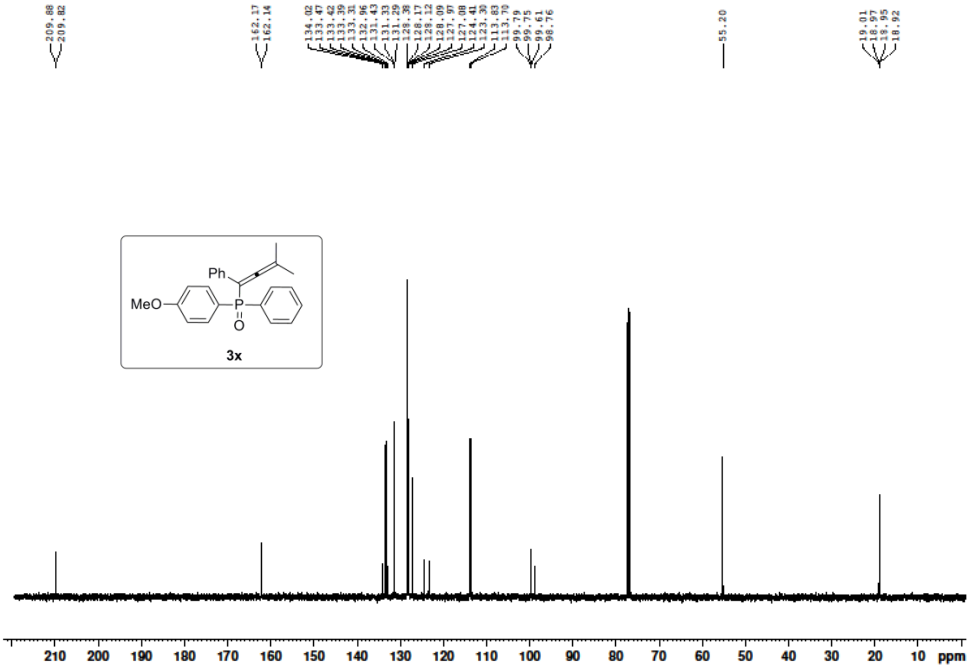

31.273
Ph
MeO
OMe
3v
100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 ppm

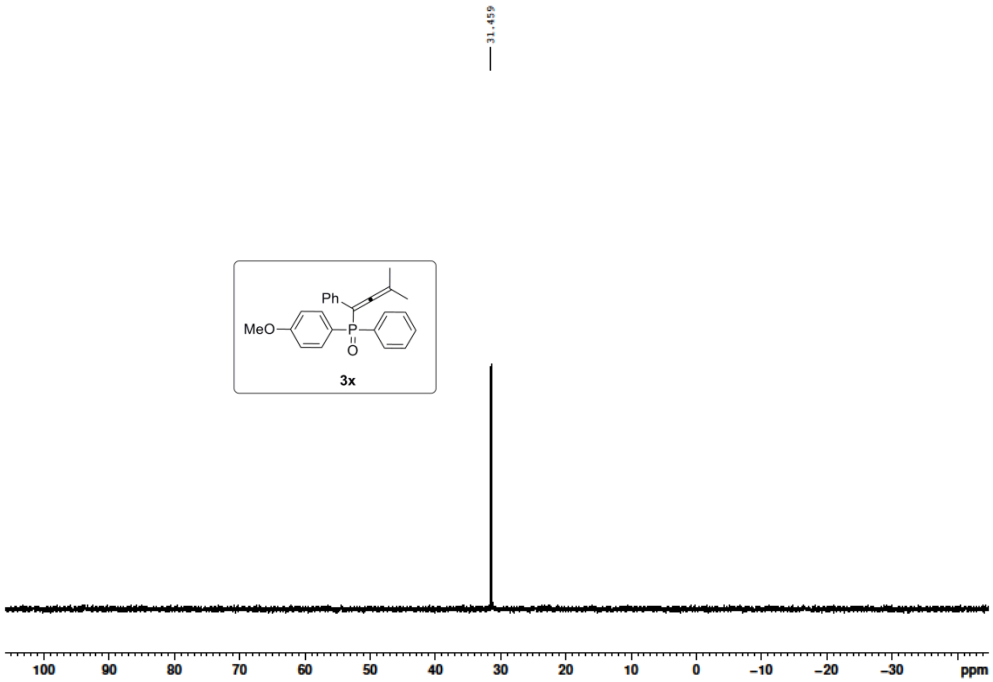

7.734
7.714
7.387
7.382
7.378
7.368
7.364
7.359
7.353
7.346
7.326
7.309
7.290
7.271
7.263
7.257
7.244
7.221
7.203
7.184
7.145
7.128
7.108
2.597
1.388
1.373
Ph
P
O
3w
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
4.08
4.13
1.07
2.02
6.00
6.03

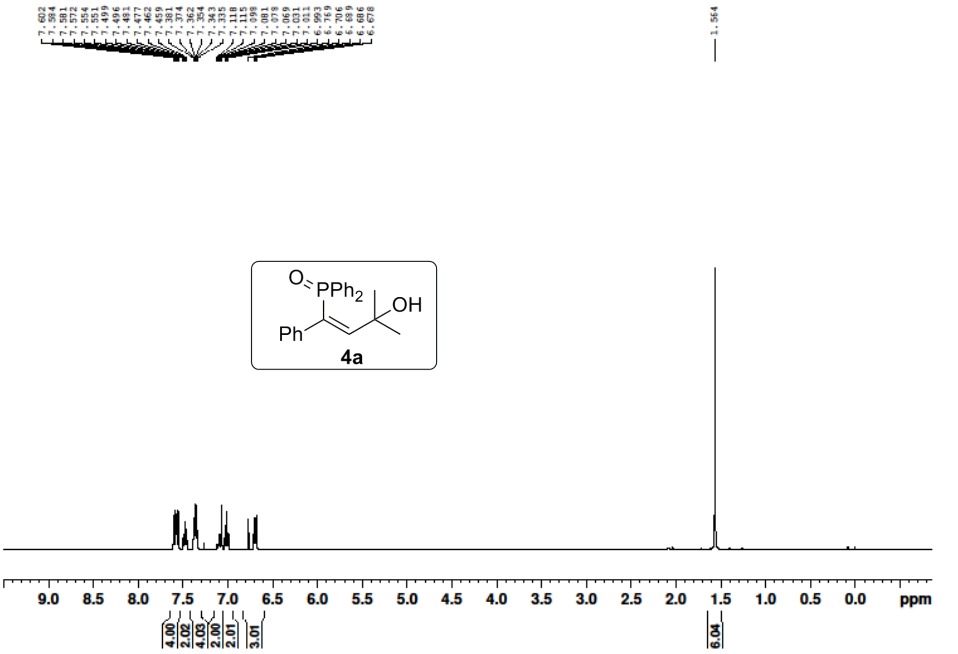

208.77
208.71
143.05
142.98
133.99
133.92
133.44
132.32
131.84
131.70
131.59
131.45
131.42
130.92
128.48
128.45
127.34
125.18
125.06
100.01
99.88
99.00
97.98
21.84
21.80
18.54
18.49
Ph
P
O
3w
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

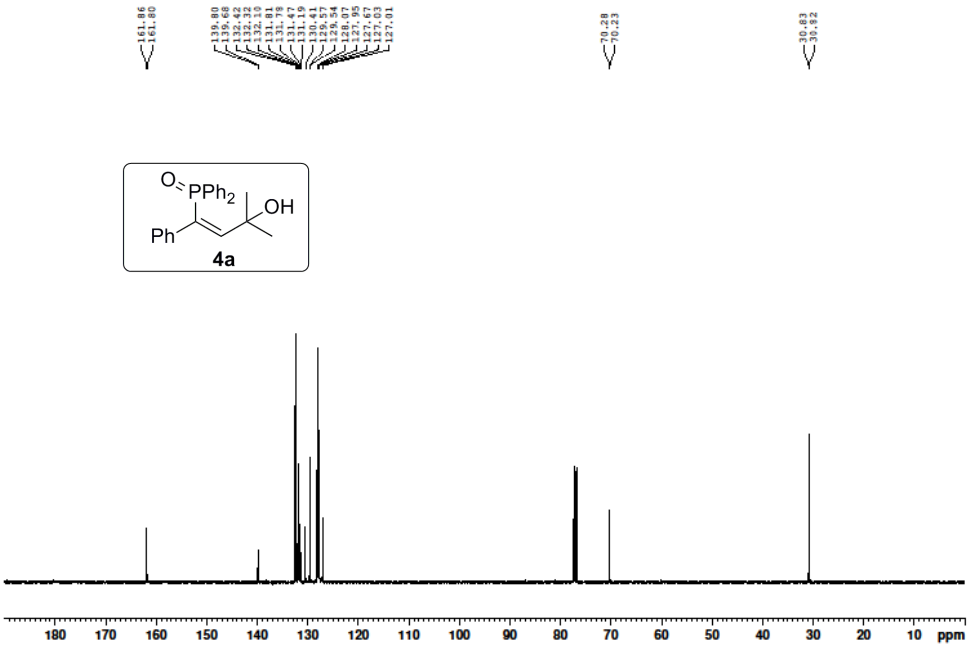

Ph
P
O
3w
37.719
90
80
70
60
50
40
30
20
10
0
−10
−20 ppm

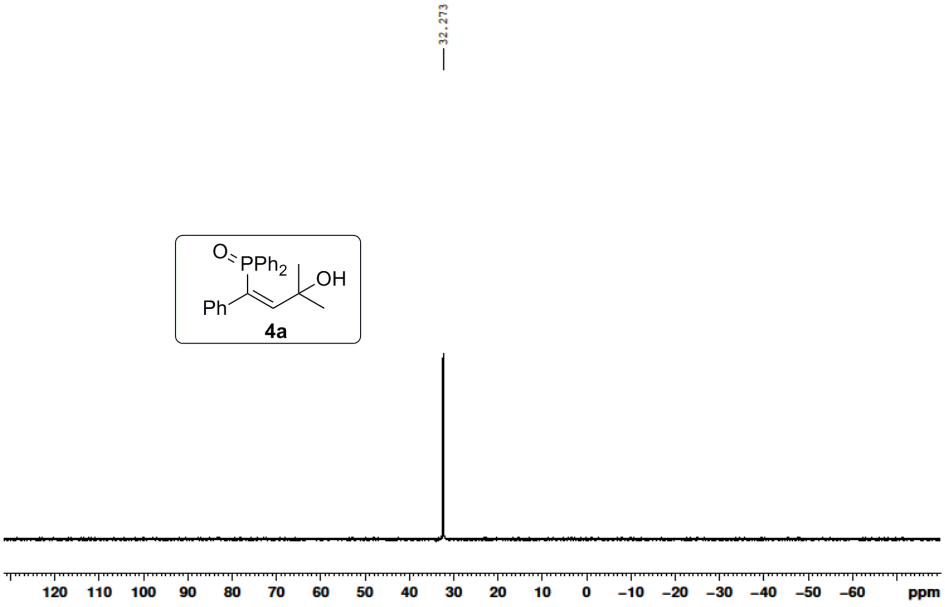

7.735
7.718
7.715
7.706
7.700
7.695
7.689
7.684
7.678
7.671
7.667
7.654
7.649
7.643
7.595
7.575
7.485
7.481
7.467
7.463
7.449
7.445
7.427
7.420
7.409
7.401
7.391
7.387
7.384
7.271
7.250
7.231
7.213
7.169
7.151
7.133
7.132
6.946
6.946
6.930
6.924
3.812
1.497
1.482
1.477
1.462
Ph
MeO
P
O
3x
9.5
9.0
8.5
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 ppm
4.01
1.98
3.08
2.00
1.02
2.01
3.00
6.01


209.88
209.82
162.17
162.14
134.02
133.47
133.42
133.39
133.31
132.86
131.43
131.33
131.29
128.38
128.17
128.12
128.09
127.97
127.88
124.41
123.30
113.83
113.70
99.79
99.75
99.61
98.76
55.20
19.01
18.97
18.95
18.92
Ph
MeO
P
O
3x
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


31.459
Ph
MeO
P
O
3x
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
ppm


O
PPh2
OH
Ph
4a
ppm


O
PPh2
OH
Ph
4a
ppm


32.273
O
PPh2
OH
Ph
4a
120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 −60 ppm