Supporting Information

Copper-catalyzed 4-HO-TEMPOH mediated phosphorylation of alkenes

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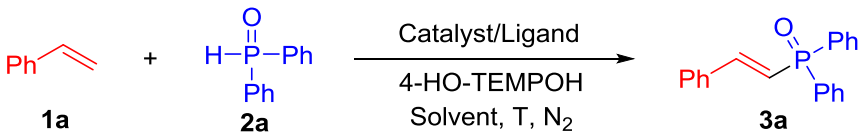
General:

All reagents were purchased from commercial suppliers and used without further purification. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^{1}H NMR（400 MHz）and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$. Chemical shifts (δ) are reported in ppm using TMS as internal standard and spin-spin coupling constants (*J*) are given in Hz. Proton, carbon and phosphorus multiplicity are recorded as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m) and broad (brs). Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and uncorrected. The high-resolution mass spectra (HRMS) were measured on a Waters LCT Premierxe^{TM} spectrometer by ESI.

Optimization Screens for alkene phosphorylation reaction conditions:

Table S1. Screening of catalysts.[a]



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

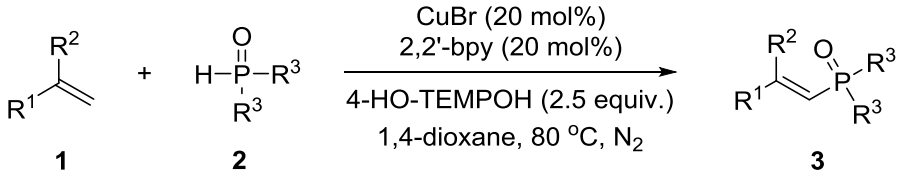
Entry	catalyst (mol%)	Ligand (mol%)	Solvent	Yield (%) [b]
1	**CuBr**	**2,2'-Bpy**	**1,4-Dioxane**	**62**
2	$CuBr_2$	2,2'-Bpy	1,4-Dioxane	51
3	$Cu(OAc)_2$	2,2'-Bpy	1,4-Dioxane	16
4	$Cu(OTf)_2$	2,2'-Bpy	1,4-Dioxane	31
5	CuI	2,2'-Bpy	1,4-Dioxane	28
6	CuCl	2,2'-Bpy	1,4-Dioxane	35
7	$Fe(acac)_3$	2,2'-Bpy	1,4-Dioxane	< 5
8	$FeCl_2$	2,2'-Bpy	1,4-Dioxane	12
9	$Fe(OAc)_2$	2,2'-Bpy	1,4-Dioxane	12
10	$Fe(OTf)_3$	2,2'-Bpy	1,4-Dioxane	19

[a] All reactions were carried out by stirring a mixture of **1a** (0.3 mmol), **2a** (0.2 mmol), catalyst (20 mol%), 2,2'-bpy (20 mol%) and 4-HO-TEMPOH (2.5 equiv.) in 2 mL 1,4-dioxane under N_2 atmosphere at 80 °C for 12 h unless noted otherwise. [b] Isolated yield.

Substrates preparation:

Unless otherwise stated, starting materials were purchased from Adamas, 3A Materials, TCI or Energy-Chemical and used without further purification. The following starting materials were prepared according to the procedures described previously in the literatures: **1ac**,[1] **1ad-ag**,[2] **1ah**,[3] 2,2,6,6-tetramethylpiperidin-1-ol (TEMPOH) and 2,2,6,6-tetramethylpiperidine-1,4-diol (4-HO-TEMPOH).[4]

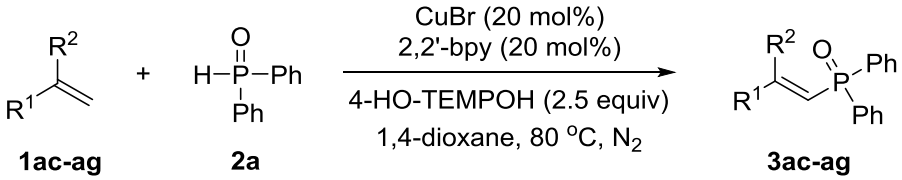
General experimental procedure:



1 **2** **3**

A 25 mL oven-dried reaction tube were charged with alkenes **1** (0.5 mmol, 2.5 equiv.), P(O)H compounds **2** (0.2 mmol), CuBr (20 mmol%), 2,2'-bpy (20 mmol%), 4-HO-TEMPOH (0.5 mmol, 2.5 equiv.) and 2 mL 1,4-dioxane. The tube was then sealed and the displacement of N_2 gas for three times. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/2 to 2/1) to give the corresponding products in yields listed in Table 2. The identity and purity of the products were confirmed by ^{1}H, ^{13}C and ^{31}P spectroscopic analysis.

Late-stage functionalization of complex bioactive molecules:



1ac-ag **2a** **3ac-ag**

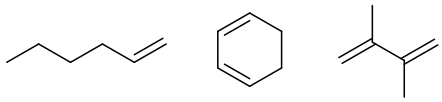
A 25 mL oven-dried reaction tube were charged with alkenes **1ac-ag** (0.5 mmol, 2.5 equiv.), diphenylphosphine oxide **2a** (0.2 mmol), CuBr (20 mmol%), 2,2'-bpy (20 mmol%), 4-HO-TEMPOH (0.5 mmol, 2.5 equiv.) and 2 mL 1,4-dioxane. The tube was then sealed and the displacement of N_2 gas for three times. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The

residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/2 to 2/1) to give the corresponding products in yields listed in Table 3. The identity and purity of the products were confirmed by ^{1}H, ^{13}C and ^{31}P spectroscopic analysis.

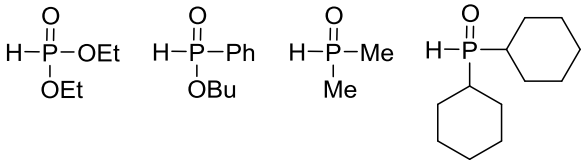
The unsuccessful substrates for this reaction:

Table S2. The unsuccessful substrates.

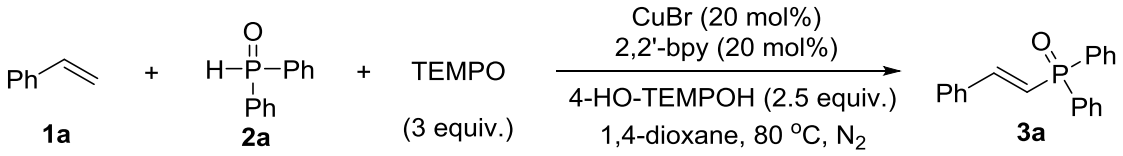
1) The alkenes



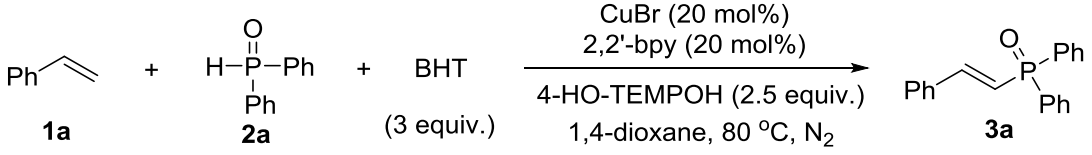
2) The P species



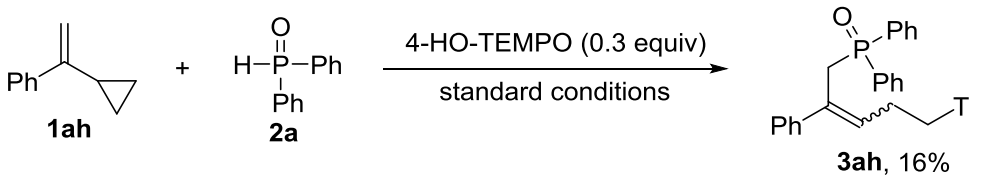
Control experiments:



A 25 mL oven-dried reaction tube were charged with alkenes **1a** (0.5 mmol, 2.5 equiv.), diphenylphosphine oxide **2a** (0.2 mmol), CuBr (20 mmol%), 2,2'-bpy (20 mmol%), 4-HO-TEMPOH (0.5 mmol, 2.5 equiv.), TEMPO (3 equiv.) and 2 mL 1,4-dioxane. The tube was then sealed and the displacement of N_2 gas for three times. Then the mixture was stirred for 12 h at 80 °C. The reaction was almost fully suppressed and a little product was detected (< 5 %).



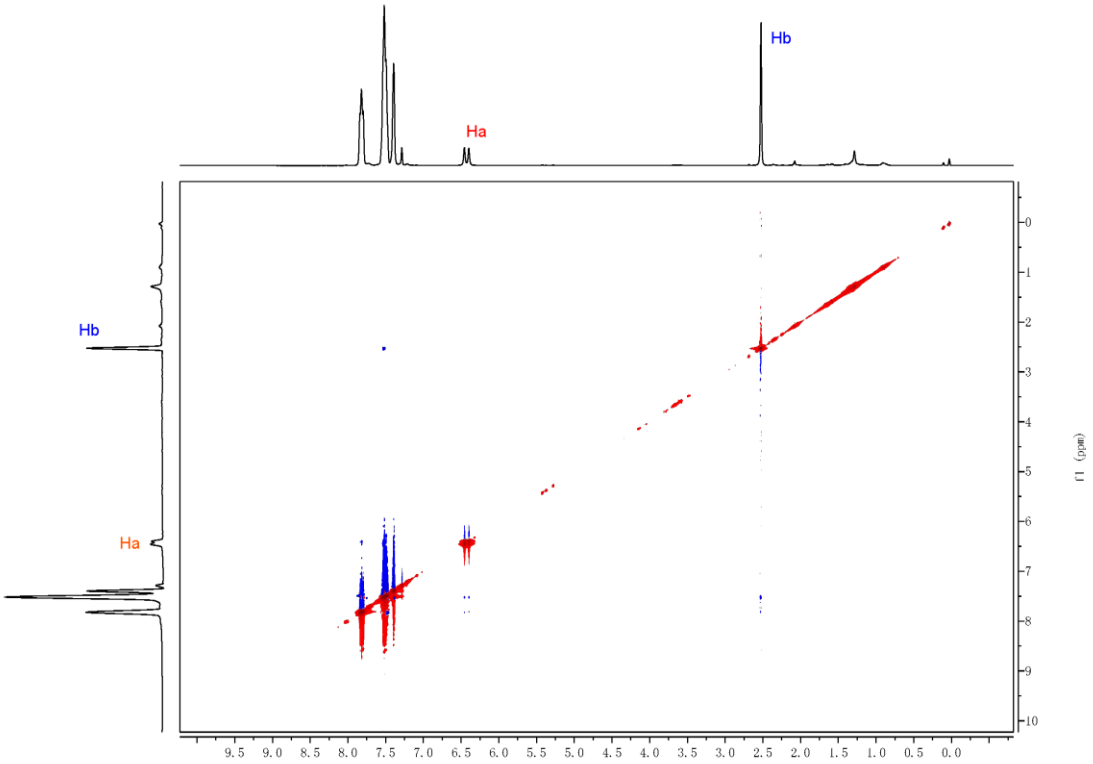
A 25 mL oven-dried reaction tube were charged with alkenes **1a** (0.5 mmol, 2.5 equiv.), diphenylphosphine oxide **2a** (0.2 mmol), CuBr (20 mmol%), 2,2'-bpy (20 mmol%), 4-HO-TEMPOH (0.5 mmol, 2.5 equiv.), BHT (3 equiv.) and 2 mL 1,4-dioxane. The tube was then sealed and the displacement of N_2 gas for three times. Then the mixture was stirred for 12 h at 80 °C. The reaction was almost fully suppressed and a little product was detected (< 5 %).

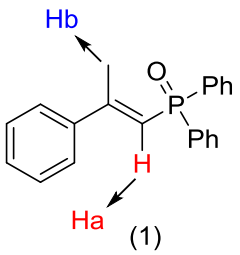


A 25 mL oven-dried reaction tube was charged with (1-cyclopropylvinyl)benzene **1ah** (0.5 mmol, 2.5 equiv), diphenylphosphine oxide (0.2 mmol), CuBr (20 mmol%), 2,2'-bpy (20 mmol%), 4-HO-TEMPOH (0.5 mmol, 2.5 equiv.), 4-HO-TEMPO (0.3 equiv.) and 2 mL 1,4-dioxane. The tube was then sealed and the displacement of N_2 gas for three times. Next, the mixture was stirred at 80 °C for 12 h. The reaction mixture was isolated by silica gel column chromatography to give the ring-opening product **3ah** in 16% yield.

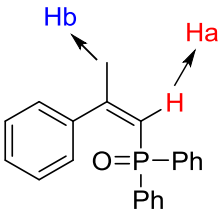
The NOE experiment of 3y:

The product **3y** may have two different structures as shown in below. In order to identify the configuration of **3y**, the NOE experiment has been conducted and the result is shown as below:





(1)



(2)

From above NOE experiment, we found that Ha and Hb are not adjacent in space. Thus, the structure 2 (Z isomer) could be excluded and the product **3y** should be E-isomer as reported.

References:

(1) M. E. Norako, M. J. Greaney and R. L. Brutchey, Synthesis and characterization of wurtzite-phase copper tin selenide nanocrystals. *J. Am. Chem. Soc.*, 2012, **134**, 23-26.

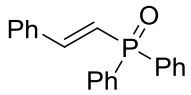
(2) (a) F. Chen, Y.-T. Tang, X.-R. Li, Y.-Y. Duan, C.-X. Chen and Y. Zheng, Oxoammonium salt-mediated vicinal oxyazidation of alkenes with NaN_3: Access to β-aminooxy azides. *Adv. Syn. Catal.*, 2021, **363**, 5079-5084. (b) X. Yin, B. Chen, F. Qiu, X. Wang, Y. Liao, M. Wang, X. Lei and J. Liao, Enantioselective palladium-catalyzed hydrofluorination of alkenylarenes. *ACS Catal.*, 2020, **10**, 1954-1960. (c) Y. Zheng, Q.-Y. Yang, L.-Y. Wu, X.-Y. Zhu, M.-J. Ge, H. Yang, S.-Y. Liu and F. Chen, Oxoammonium salt-mediated regioselective vicinal dioxidation of alkenes: relying on transient and persistent nitroxides. *Org. Lett.*, 2021, **23**, 8533-8538.

(3) (a) C. Chen, X. Shen, J. Chen, X. Hong and Z. Lu, Iron-catalyzed hydroboration of vinylcyclopropanes. *Org. Lett.*, 2017, **19**, 5422-5425. (b) W.-S. Zhang, D.-W. Ji, Y. Li, X.-X. Zhang, C.-Y. Zhao, Y.-C. Hu and Q.-A. Chen, Regio- and stereoselective diarylation of 1,3-dienes via Ni/Cr cocatalysis. *ACS Catal.*, 2022, **12**, 2158-2165.

(4) (a) W. Lu and Z. Shen, Direct synthesis of alkenylboronates from alkenes and pinacol diboron via copper catalysis. *Org. Lett.*, 2019, **21**, 142-146. (b) L. Zhu, D. Song, Y.-H. Liu, M.-D. Chen, X.-R. Zhang, M.-Y. You and J.-L. Zhan, Iron-catalyzed regioselective synthesis of (*E*)-vinyl sulfones mediated by unprotected hydroxylamines. *Org. Biomol. Chem.*, 2022, **20**, 9127-9131.

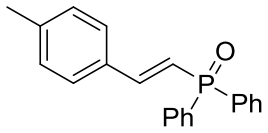
Analytical data for products:

(E)-Diphenyl(styryl)phosphine oxide (3a)



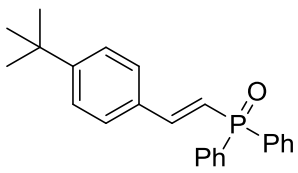
White solid; (46 mg, 75%); mp: 166–170 °C; R_f = 0.44 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 4H), 7.54–7.46 (m, 9H), 7.38–7.37 (m, 3H), 6.84 (dd, J = 17.6 Hz, J = 22.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 134.4 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.2 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.7 (d, $J_{C\text{-}P}$ = 9.0 Hz), 129.5, 128.2, 128.0 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.1, 118.5 (d, $J_{C\text{-}P}$ = 104.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.7; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{18}OP$, $[M + H]^+$: 305.1090, found 305.1087.

(E)-(4-Methylstyryl)diphenylphosphine oxide (3b)



White solid; (53 mg, 83%); mp: 210–213 °C; R_f = 0.42 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, J = 7.6 Hz, J = 12.0 Hz, 4H), 7.55–7.41 (m, 9H), 7.18 (d, J = 8.0 Hz, 2H), 6.77 (dd, J = 17.2 Hz, J = 22.4 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.5 (d, $J_{C\text{-}P}$ = 4.0 Hz), 140.4, 133.1 (d, $J_{C\text{-}P}$ = 105.0 Hz), 132.4 (d, $J_{C\text{-}P}$ = 18.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.7, 117.8 (d, $J_{C\text{-}P}$ = 105.0 Hz), 21.4; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.7; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{20}OP$, $[M + H]^+$: 319.1246, found 319.1247.

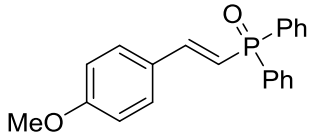
(E)-(4-(tert-Butyl)styryl)diphenylphosphine oxide (3c)



White solid; (61 mg, 85%); mp: 160–163 °C; R_f = 0.55 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, J = 7.6 Hz, J = 12.0 Hz, 4H), 7.55–7.44 (m, 9H), 7.40 (d, J = 8.0 Hz, 2H), 6.79 (dd, J = 17.2 Hz, J = 22.4 Hz, 1H), 1.32 (s , 9H); ^{13}C NMR (100 MHz,

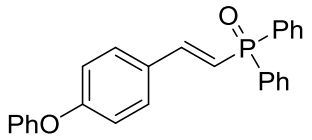
$CDCl_3$): δ 153.6, 147.5 (d, $J_{C\text{-}P}$ = 4.0 Hz), 132.9 (d, $J_{C\text{-}P}$ = 106.0 Hz), 132.4 (d, $J_{C\text{-}P}$ = 18.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.6, 125.8, 117.9 (d, $J_{C\text{-}P}$ = 105.0 Hz), 34.8, 31.3; ^{31}P NMR ($CDCl_3$, 162 MHz): 25.1; ESI-HRMS (ESI, m/z): Calcd for $C_{24}H_{26}OP$, $[M + H]^+$: 361.1716, found 361.1718.

(E)-(4-Methoxystyryl)diphenylphosphine oxide (3d)



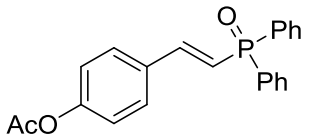
White solid; (50 mg, 74%); mp: 189–193 °C; R_f = 0.40 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58–7.73 (m, 4H), 7.55–7.51 (m, 2H), 7.49–7.38 (m, 7H), 6.89 (d, J = 8.8 Hz, 2H), 6.66 (dd, J = 17.2 Hz, J = 22.4 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.1, 147.2 (d, $J_{C\text{-}P}$ = 4.0 Hz), 133.0 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10 Hz), 129.3, 128.5 (d, $J_{C\text{-}P}$ = 12 Hz), 127.8 (d, $J_{C\text{-}P}$ = 19 Hz), 115.8 (d, $J_{C\text{-}P}$ = 106.0 Hz), 114.2, 55.3; ^{31}P NMR ($CDCl_3$, 162 MHz): 25.4; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{19}NaO_2P$, $[M + Na]^+$: 357.1015, found 357.1019.

(E)-(4-Phenoxystyryl)diphenylphosphine oxide (3e)



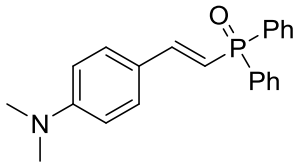
White solid; (58 mg, 73%); mp: 155–158 °C; R_f = 0.47 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (dd, J = 7.6 Hz, J = 12 Hz, 4H), 7.55–7.42 (m, 9H), 7.35 (t, J = 8.0 Hz, 2H), 7.14 (t, J = 7.2 Hz, 1H), 7.03 (d, J = 8.0 Hz, 2H), 6.98 (d, J = 8.4 Hz, 2H), 6.73 (dd, J = 17.2 Hz, J = 22.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 156.1, 146.6 (d, $J_{C\text{-}P}$ = 4.0 Hz), 132.9 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.7 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 9.0 Hz), 129.9 (d, $J_{C\text{-}P}$ = 18 Hz), 129.8, 129.3, 128.5 (d, $J_{C\text{-}P}$ = 12 Hz), 123.9, 119.4, 118.3, 117.5 (d, $J_{C\text{-}P}$ = 105.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.7; ESI-HRMS (ESI, m/z): Calcd for $C_{26}H_{22}O_2P$, $[M + H]^+$: 397.1352, found 397.1349.

(E)-4-(2-(Diphenylphosphoryl)vinyl)phenyl acetate (3f)



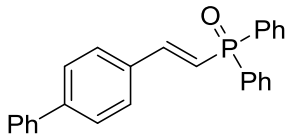
White solid; (55 mg, 75%); mp: 176–180 °C; R_f = 0.50 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.56–7.44 (m, 9H), 7.12 (d, J = 8.4 Hz, 2H), 6.79 (dd, J = 17.2 Hz, J = 22.4 Hz, 1H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.1, 151.9, 146.3 (d, $J_{C\text{-}P}$ = 4.0 Hz), 132.8 (d, $J_{C\text{-}P}$ = 18 Hz), 132.76 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10 Hz), 128.8, 128.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 122.0, 119.4 (d, $J_{C\text{-}P}$ = 104.0 Hz), 21.1; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.4; ESI-HRMS (ESI, m/z): Calcd for $C_{22}H_{19}NaO_3P$, $[M + Na]^+$: 385.0964, found 385.0968.

(E)-(4-(Dimethylamino)styryl)diphenylphosphine oxide (3g)



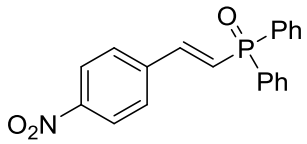
Purple solid; (42 mg, 61%); mp: 184–188 °C; R_f = 0.43 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 4H), 7.53–7.49 (m, 2H), 7.48–7.43 (m, 4H), 7.42–7.30 (m, 3H), 6.66 (d, J = 9.2 Hz, 2H), 6.52 (dd, J = 17.6 Hz, J = 22.4 Hz, 1H), 3.00 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.4, 147.7 (d, $J_{C\text{-}P}$ = 4.0 Hz), 133.4 (d, $J_{C\text{-}P}$ = 104.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.1, 128.3 (d, $J_{C\text{-}P}$ = 12.0 Hz), 122.9 (d, $J_{C\text{-}P}$ = 19.0 Hz), 112.0 (d, $J_{C\text{-}P}$ = 109.0 Hz), 111.6, 40.0; ^{31}P NMR ($CDCl_3$, 162 MHz): 26.0; ESI-HRMS (ESI, m/z): Calcd for $C_{22}H_{23}NOP$, $[M + H]^+$: 348.1512, found 348.1513.

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



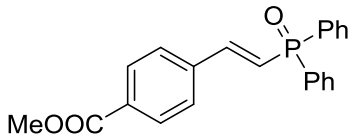
White solid; (48 mg, 63%); mp: 205–209 °C; R_f = 0.50 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80–7.75 (m, 4H), 7.64–7.59 (m, 6H), 7.57–7.43 (m, 9H), 7.37 (t, J = 7.2 Hz, 1H), 6.87 (dd, J = 17.6 Hz, J = 22.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 142.7, 140.0, 133.9 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.7, 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.1, 127.6, 127.3, 126.9, 118.8 (d, $J_{C\text{-}P}$ = 104.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.7; ESI-HRMS (ESI, m/z): Calcd for $C_{26}H_{22}OP$, $[M + H]^+$: 381.1403, found 385.1402.

(E)-(4-Nitrostyryl)diphenylphosphine oxide (3i)



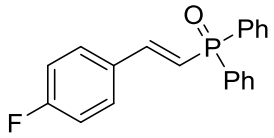
Yellow solid; (33 mg, 47%); mp: 212–215 °C; R_f = 0.46 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (d, J = 7.6 Hz, 2H), 7.78–7.67 (m, 4H), 7.61–7.51 (m, 9H), 7.09–6.99 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4, 144.6 (d, $J_{C\text{-}P}$ = 4.0 Hz), 141.0 (d, $J_{C\text{-}P}$ = 19.0 Hz), 132.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 132.1 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.8 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.4, 124.7 (d, $J_{C\text{-}P}$ = 100.0 Hz), 124.1; ^{31}P NMR ($CDCl_3$, 162 MHz): 23.5; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}NO_3P$, $[M + H]^+$: 350.0941, found 350.0946.

Methyl (E)-4-(2-(diphenylphosphoryl)vinyl)benzoate (3j)



White solid; (56 mg, 77%); mp: 182–184 °C; R_f = 0.41 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, J = 8.4 Hz, 2H), 7.79–7.74 (m, 4H), 7.60–7.47 (m, 9H), 6.97 (dd, J = 17.2 Hz, J = 22.0 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.3, 146.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 139.0 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.4 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.1, 130.0, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.5, 122.1 (d, $J_{C\text{-}P}$ = 102.0 Hz), 52.1; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.0; ESI-HRMS (ESI, m/z): Calcd for $C_{22}H_{20}O_3P$, $[M + H]^+$: 363.1145, found 363.1146.

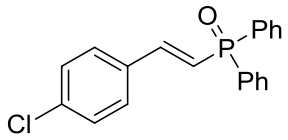
(E)-(4-Fluorostyryl)diphenylphosphine oxide (3k)



White solid; (39 mg, 61%); mp: 163–167 °C; R_f = 0.42 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, J = 7.6 Hz, J = 12 Hz, 4H), 7.56–7.43 (m, 9H), 7.06 (t, J = 8.4 Hz, 2H), 6.77 (dd, J = 17.6 Hz, J = 22.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.2 (d, $J_{C\text{-}F}$ = 249.0 Hz), 145.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 132.3 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.5 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.0 (d, $J_{C\text{-}F}$ = 3.0 Hz), 130.9 (d, $J_{C\text{-}P}$ = 10 Hz), 129.2 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 12 Hz), 118.4 (dd, $J_{C\text{-}P}$ = 104.0 Hz, $J_{C\text{-}F}$ = 2 Hz), 115.5 (d, $J_{C\text{-}F}$ = 21.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz):

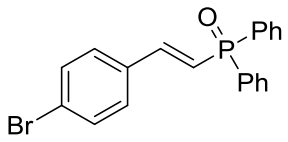
24.4; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}FOP$, $[M + H]^+$: 323.0996, found 323.1003.

(E)-(4-Chlorostyryl)diphenylphosphine oxide (3l)



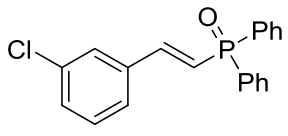
White solid; (49 mg, 72%); mp: 185–189 °C; R_f = 0.44 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.57–7.42 (m, 9H), 7.35 (d, J = 8.4 Hz, 2H), 6.82 (dd, J = 17.2 Hz, J = 22.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 136.0, 133.6 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.1, 128.9, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 120.0 (d, $J_{C\text{-}P}$ = 103.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.2; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}ClOP$, $[M + H]^+$: 339.0700, found 339.0702.

(E)-(4-Bromostyryl)diphenylphosphine oxide (3m)



White solid; (49 mg, 64%); mp: 195–199 °C; R_f = 0.27 (hexanes/ethyl acetate 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.57–7.38 (m, 11H), 6.83 (dd, J = 17.6 Hz, J = 22.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.2 (d, $J_{C\text{-}P}$ = 4.0 Hz), 134.0 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 105.0 Hz), 132.1, 132.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 9.0 Hz), 129.2, 128.7 (d, $J_{C\text{-}P}$ = 12.0 Hz), 124.3, 120.1 (d, $J_{C\text{-}P}$ = 103.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.2; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}BrOP$, $[M + H]^+$: 383.0195, found 383.0190.

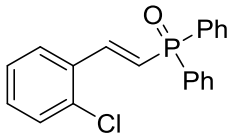
(E)-(3-Chlorostyryl)diphenylphosphine oxide (3n)



White solid; (37 mg, 54%); mp: 173–176 °C; R_f = 0.41 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.57–7.53 (m, 2H), 7.51–7.42 (m, 6H), 7.40–7.29 (m, 3H), 6.86 (dd, J = 17.2 Hz, J = 22.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 136.8 (d, $J_{C\text{-}P}$ = 17.0 Hz), 134.8, 132.5 (d, $J_{C\text{-}P}$ = 106.0 Hz), 132.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 130.1, 129.9, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.2, 126.2,

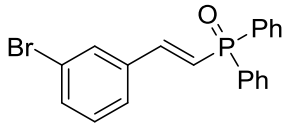
121.0 (d, $J_{C\text{-}P}$ = 102.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.4; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}ClOP$, $[M + H]^+$: 339.0700, found 339.0704.

(E)-(2-Chlorostyryl)diphenylphosphine oxide (3o)



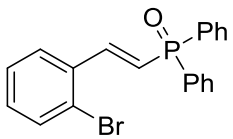
White solid; (32 mg, 47%); mp: 175–178 °C; R_f = 0.41 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83–7.74 (m, 5H), 7.65–7.62 (m, 1H), 7.57–7.53 (m, 2H), 7.51–7.47 (m, 4H), 7.41–7.36 (m, 1H), 7.31–7.25 (m, 2H), 6.88 (dd, J = 17.2 Hz, J = 20.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5 (d, $J_{C\text{-}P}$ = 5.0 Hz), 134.4, 133.4 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.4 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 130.7, 130.0, 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.6 (d, $J_{C\text{-}P}$ = 1.0 Hz), 127.0, 123.0 (, $J_{C\text{-}P}$ = 103.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.7; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}ClOP$, $[M + H]^+$: 339.0700, found 339.0703.

(E)-(3-Bromostyryl)diphenylphosphine oxide (3p)



Yellow solid; (38 mg, 50%); mp: 145–148 °C; R_f = 0.49 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.67 (s, 1H), 7.55–7.42 (m, 9H), 7.25 (t, J = 7.2 Hz, 1H); 6.85 (t, J = 26.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.3 (d, $J_{C\text{-}P}$ = 4.0 Hz), 135.6 (d, $J_{C\text{-}P}$ = 18.0 Hz), 131.4, 130.9 (d, $J_{C\text{-}P}$ = 106.0 Hz), 130.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 129.8 (d, $J_{C\text{-}P}$ = 10 Hz), 128.9, 128.7, 127.2 (d, $J_{C\text{-}P}$ = 12.0 Hz), 125.2, 121.5, 119.5 (d, $J_{C\text{-}P}$ = 103.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.3; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}BrOP$, $[M + H]^+$: 383.0195, found 383.0197.

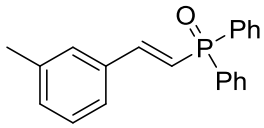
(E)-(2-Bromostyryl)diphenylphosphine oxide (3q)



White solid; (30 mg, 39%); mp: 158–161 °C; R_f = 0.45 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (dd, J = 7.6 Hz, J = 12.0 Hz, 4H), 7.74–7.64 (m, 1H), 7.61 (d,

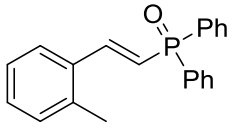
J = 8.0 Hz, 1H), 7.58–7.53 (m, 3H), 7.51–7.48 (m, 4H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 6.82 (dd, *J* = 17.6 Hz, *J* = 20.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 134.8 (d, $J_{C\text{-}P}$ = 18.0 Hz), 133.0, 131.9 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 130.6, 128.3 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.4, 127.3, 124.4, 122.8 (d, $J_{C\text{-}P}$ = 102.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 25.0; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{17}BrOP$, $[M + H]^+$: 383.0195, found 383.0196.

(E)-(3-Methylstyryl)diphenylphosphine oxide (3r)



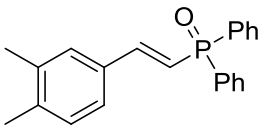
White solid; (36 mg, 57%); mp: 130–134 °C; R_f = 0.44 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, *J* = 7.6 Hz, *J* = 12 Hz, 4H), 7.56–7.42(m, 7H), 7.33 (d, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 6.82 (dd, *J* = 17.2 Hz, *J* = 22.4 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.1 (d, $J_{C\text{-}P}$ = 4.0 Hz), 137.8, 134.4 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.3 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.7 (d, $J_{C\text{-}P}$ = 9.0 Hz), 130.3, 128.1, 128.0 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.6, 124.4, 118.2 (d, $J_{C\text{-}P}$ = 104.0 Hz), 20.7; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.8; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{20}OP$, $[M + H]^+$: 319.1246, found 319.1243.

(E)-(2-Methylstyryl)diphenylphosphine oxide (3s)



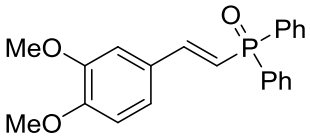
White solid; (31 mg, 49%); mp: 137–141 °C; R_f = 0.51 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79–7.72 (m, 5H), 7.57–7.48 (m, 7H), 7.26–7.20 (m, 3H), 6.78 (dd, *J* = 17.6 Hz, *J* = 22.8 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 133.3, 130.2 (d, $J_{C\text{-}P}$ = 18.0 Hz), 128.9 (d, $J_{C\text{-}P}$ = 105.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 2.0 Hz), 127.5 (d, $J_{C\text{-}P}$ = 10.0 Hz), 127.0, 126.1, 124.9 (d, $J_{C\text{-}P}$ = 12.0 Hz), 122.5, 122.4, 116.4 (d, $J_{C\text{-}P}$ = 103.0 Hz), 15.9; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.8; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{20}OP$, $[M + H]^+$: 319.1246, found 319.1248.

(E)-(3,4-Dimethylstyryl)diphenylphosphine oxide (3t)



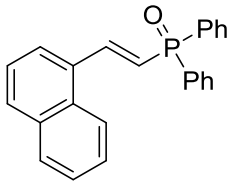
White solid; (41 mg, 62%); mp: 197–201 °C; R_f = 0.50 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 4H), 7.54–7.50 (m, 2H), 7.48–7.39 (m, 5H), 7.29–7.25 (m, 2H), 7.13 (d, J = 7.6 Hz, 1H), 7.71 (dd, J = 17.6 Hz, J = 22.4 Hz, 1H), 2.26 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.6 (d, $J_{C\text{-}P}$ = 4.0 Hz), 139.1, 137.0, 133.0 (d, $J_{C\text{-}P}$ = 105.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 18 Hz), 131.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 130.0, 128.7, 128.4 (d, $J_{C\text{-}P}$ = 12.0 Hz), 125.3, 117.5 (d, $J_{C\text{-}P}$ = 105.0 Hz), 19.64, 19.61; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.8; ESI-HRMS (ESI, m/z): Calcd for $C_{22}H_{22}OP$, $[M + H]^+$: 333.1403, found 333.1405.

(E)-(3,4-Dimethoxystyryl)diphenylphosphine oxide (3u)



White solid; (46 mg, 63%); mp: 136–140 °C; R_f = 0.31 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79–7.74 (m, 4H), 7.54–7.36 (m, 7H), 7.10–7.06 (m, 2H), 6.86 (d, J = 8.0 Hz, 1H), 6.66 (dd, J = 17.6 Hz, J = 21.6 Hz, 1H), 3.90 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.9, 149.2, 147.4 (d, $J_{C\text{-}P}$ = 4.0 Hz), 133.1 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 18.0 Hz), 122.0, 116.4 (d, $J_{C\text{-}P}$ = 106.0 Hz), 111.0, 109.5, 55.94, 55.90; ^{31}P NMR ($CDCl_3$, 162 MHz): 25.0; ESI-HRMS (ESI, m/z): Calcd for $C_{22}H_{21}NaO_3P$, $[M + Na]^+$: 387.1121, found 387.1123.

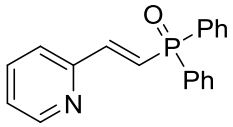
(E)-(2-(Naphthalen-1-yl)vinyl)diphenylphosphine oxide (3v)



Red solid; (51 mg, 72%); mp: 141–143 °C; R_f = 0.56 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.33 (dd, J = 13.2 Hz, J = 19.6 Hz, 1H), 8.13–8.10 (m, 1H), 7.88–7.76 (m, 7H), 7.57–7.45 (m, 9H), 6.96 (dd, J = 17.2 Hz, J = 23.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 133.5, 132.8 (d, $J_{C\text{-}P}$ = 105.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 17.0 Hz),

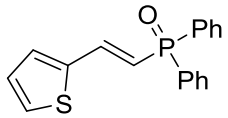
131.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.0, 130.2, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 126.7, 126.1, 125.2, 124.6 (d, $J_{C\text{-}P}$ = 1.0 Hz), 123.2, 122.4 (d, $J_{C\text{-}P}$ = 102.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.2; ESI-HRMS (ESI, m/z): Calcd for $C_{24}H_{20}OP$, $[M + H]^+$: 355.1246, found 355.1240.

(E)-Diphenyl(2-(pyridin-2-yl)vinyl)phosphine oxide (3w)



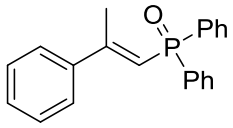
Yellow solid; (30 mg, 49%); mp: 134–138 °C; R_f = 0.21 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (d, J = 4.4 Hz, 1H), 7.80–7.75 (m, 4H), 7.71 (dt, J = 1.6 Hz, J = 8.0 Hz, 1H), 7.62–7.59 (m, 1H), 7.57–7.50 (m, 3H), 7.48–7.44 (m, 4H), 7.38 (d, J = 7.6 Hz, 1H), 7.25 (dd, J = 4.8 Hz, J = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.6 (d, $J_{C\text{-}P}$ = 18.0 Hz), 149.8, 145.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 136.9, 132.5 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 124.4, 124.1, 123.8 (d, $J_{C\text{-}P}$ = 101.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.1; ESI-HRMS (ESI, m/z): Calcd for $C_{19}H_{19}NOP$, $[M + H]^+$: 308.1199, found 308.1202.

(E)-Diphenyl(2-(thiophen-2-yl)vinyl)phosphine oxide (3x)



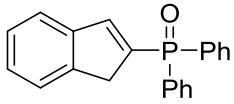
White solid; (36 mg, 58%); mp: 144–145 °C; R_f = 0.52 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.63–7.52 (m, 3H), 7.50–7.46 (m, 4H), 7.36 (d, J = 4.8 Hz, 1H), 7.20 (d, J = 3.6 Hz, 1H), 7.03 (dd, J = 3.6 Hz, J = 4.8 Hz, 1H), 6.58 (dd, J = 17.2 Hz, J = 21.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.8, 140.5, 139.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 132.7 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 130.0, 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 117.5 (d, $J_{C\text{-}P}$ = 105.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.2; ESI-HRMS (ESI, m/z): Calcd for $C_{18}H_{16}OPS$, $[M + H]^+$: 311.0654, found 311.0655.

(E)-Diphenyl(2-phenylprop-1-en-1-yl)phosphine oxide (3y)



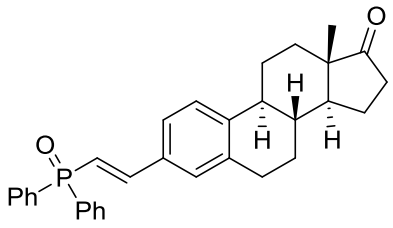
White oil; (32 mg, 51%); R_f = 0.29 (hexanes/ethyl acetate 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (dd, J = 3.6 Hz, J = 7.6 Hz, 4H), 7.50–7.44 (m, 8H), 7.37–7.35 (m, 3H), 6.40 (d, J = 23.6 Hz, 1H), 2.50 (d, J = 2.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 141.4 (d, $J_{C\text{-}P}$ = 17.0 Hz), 133.9 (d, $J_{C\text{-}P}$ = 104.0 Hz), 130.8 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.5, 128.0 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.8, 125.2, 117.6 (d, $J_{C\text{-}P}$ = 104.0 Hz), 19.0 (d, $J_{C\text{-}P}$ = 7.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 21.8; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{20}OP$, $[M + H]^+$: 319.1246, found 319.1248.

(1H-Inden-2-yl)diphenylphosphine oxide (3z)



Yellow oil; (30 mg, 47%); R_f = 0.48 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78–7.73 (m, 4H), 7.56 (dt, J = 1.6 Hz, J =7.2 Hz, 2H), 7.51–7.45 (m, 6H), 7.34–7.26 (m, 3H), 3.70 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.0 (d, $J_{C\text{-}P}$ = 9.0 Hz), 145.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 142.7 (d, $J_{C\text{-}P}$ = 16.0 Hz), 139.1 (d, $J_{C\text{-}P}$ = 109.0 Hz), 132.2 (d, $J_{C\text{-}P}$ = 105.0 Hz), 132.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.6 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.2, 126.9, 124.2 (d, $J_{C\text{-}P}$ = 1.0 Hz), 122.8, 40.3 (d, $J_{C\text{-}P}$ = 12.0 Hz); ^{31}P NMR ($CDCl_3$, 162 MHz): 24.0; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{18}OP$, $[M + H]^+$: 317.1090, found 317.1089.

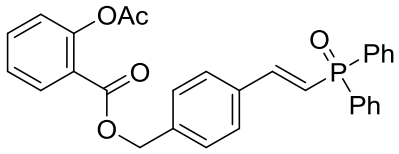
(8R,9S,13S,14S)-3-((E)-2-(Diphenylphosphoryl)vinyl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (3ac)



Yellow oil; (59 mg, 61%); R_f = 0.46 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, J = 7.2 Hz, J = 12 Hz, 4H), 7.55–7.51 (m, 2H), 7.49–7.40 (m, 5H), 7.34–7.29 (m, 2H), 7.26 (s, 1H), 6.79 (dd, J = 17.2 Hz, J = 22.4 Hz, 1H), 2.92 (t, J = 4.4 Hz, 2H), 2.50 (dd, J = 8.8 Hz, J = 18.8 Hz, 1H), 2.45–2.40 (m, 1H), 2.37–2.28 (m, 1H), 2.15 (dd, J = 8.8 Hz, J =

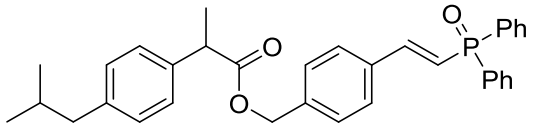
18.8 Hz, 1H), 2.10–1.96 (m, 3H), 1.68–1.59 (m, 2H), 1.56–1.40 (m, 4H), 0.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 220.5, 147.3 (d, $J_{C\text{-}P}$ = 3.0 Hz), 142.2, 137.0, 133.0 (d, $J_{C\text{-}P}$ = 105.0 Hz), 132.6 (d, $J_{C\text{-}p}$ = 18.0 Hz), 131.7 (d, $J_{C\text{-}p}$ = 2.0 Hz), 131.3 (d, $J_{C\text{-}p}$ = 9.0 Hz), 128.5 (d, $J_{C\text{-}p}$ = 12.0 Hz), 128.3, 125.8, 125.0, 118.2 (d, $J_{C\text{-}p}$ = 104.0 Hz), 50.4, 47.8, 44.5, 37.9, 35.7, 31.4, 29.2, 26.2, 25.5, 21.5, 13.7; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.6; ESI-HRMS (ESI, m/z): Calcd for $C_{32}H_{34}O_2P$, $[M + H]^+$: 481.2291, found 481.2293.

(E)-4-(2-(Diphenylphosphoryl)vinyl)benzyl 2-acetoxybenzoate (3ad)



White solid; (52 mg, 52%); mp: 129–132 °C; R_f = 0.41 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, J = 1.6 Hz, J = 7.6 Hz, 1H), 7.78–7.73 (m, 4H), 7.58–7.46 (m, 10H), 7.43 (d, J = 8.0 Hz, 2H), 7.33–7.29 (m, 1H), 7.10 (d, J = 8.0 Hz, 1H), 6.87 (dd, J = 17.6 Hz, J = 21.6 Hz, 1H), 5.31 (s, 2H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.6, 164.2, 150.7, 146.8 (d, $J_{C\text{-}P}$ = 3.0 Hz), 137.6, 135.2 (d, $J_{C\text{-}P}$ = 17.0 Hz), 134.0, 132.8 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.87, 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.7, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.0, 126.0, 123.8, 123.0, 119.9 (d, $J_{C\text{-}P}$ = 103.0 Hz), 66.3, 20.8; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.3; ESI-HRMS (ESI, m/z): Calcd for $C_{30}H_{26}O_5P$, $[M + H]^+$: 497.1512, found 497.1508.

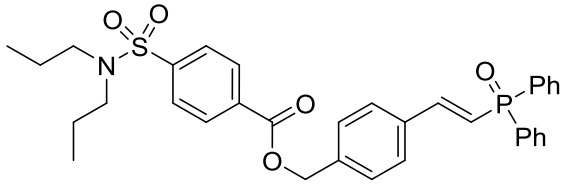
(E)-4-(2-(Diphenylphosphoryl)vinyl)benzyl 2-(4-isobutylphenyl)propanoate (3ae)



White solid; (66 mg, 63%); mp: 75–79 °C; R_f = 0.37 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77–7.72 (m, 4H), 7.56–7.43 (m, 9H), 7.20 (t, J = 7.6 Hz, 4H), 7.08 (d, J = 8.0 Hz, 2H), 6.81 (dd, J = 17.6 Hz, J = 21.6 Hz, 1H), 5.10 (s, 2H), 3.74 (q, J = 7.2 Hz, 1H), 2.44 (d, J = 7.2 Hz, 2H), 1.89–1.79 (m, 1H), 1.50 (d, J = 7.2 Hz, 3H), 0.89 (d, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.3, 146.9 (d, $J_{C\text{-}P}$ = 3.0 Hz), 140.6, 138.1, 137.4, 134.7 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.8 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 129.3, 128.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.0, 127.8, 127.1, 119.5 (d, $J_{C\text{-}P}$ = 104.0 Hz), 65.6, 45.0,

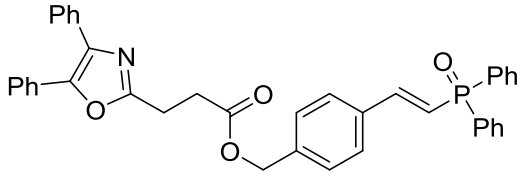
44.9, 30.1, 22.3, 18.2; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.5; ESI-HRMS (ESI, m/z): Calcd for $C_{34}H_{36}O_3P$, $[M + H]^+$: 523.2397, found 523.2398.

(E)-4-(2-(Diphenylphosphoryl)vinyl)benzyl 4-(N,N-dipropylsulfamoyl)benzoate (3af)



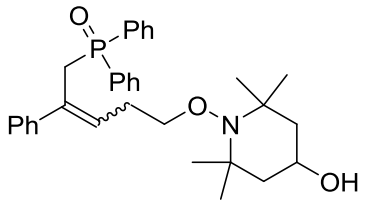
White oil; (93 mg, 77%); R_f = 0.27 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, J = 8.4 Hz, 2H), 7.87 (d, J = 8.4 Hz, 2H), 7.79–7.73 (m, 4H), 7.58–7.53 (m, 5H), 7.51–7.46 (m, 6H), 6.88 (dd, J = 17.6 Hz, J = 22.4 Hz, 1H), 5.39 (s, 2H), 3.09 (t, J = 7.6 Hz, 4H), 1.59–1.49 (m, 4H), 0.86 (t, J = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.9, 146.6 (d, $J_{C\text{-}P}$ = 4.0 Hz), 144.4, 137.3, 135.2 (d, $J_{C\text{-}P}$ = 18.0 Hz), 133.1, 132.7 (d, $J_{C\text{-}P}$ = 106.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 130.2, 128.6, 128.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.0, 126.9, 119.9 (d, $J_{C\text{-}P}$ = 103.0 Hz), 66.6, 49.8, 21.8, 11.0; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.3; ESI-HRMS (ESI, m/z): Calcd for $C_{34}H_{37}NO_5PS$, $[M + H]^+$: 602.2125, found 602.2128.

(E)-4-(2-(Diphenylphosphoryl)vinyl)benzyl 3-(4,5-diphenyloxazol-2-yl)propanoate (3ag)

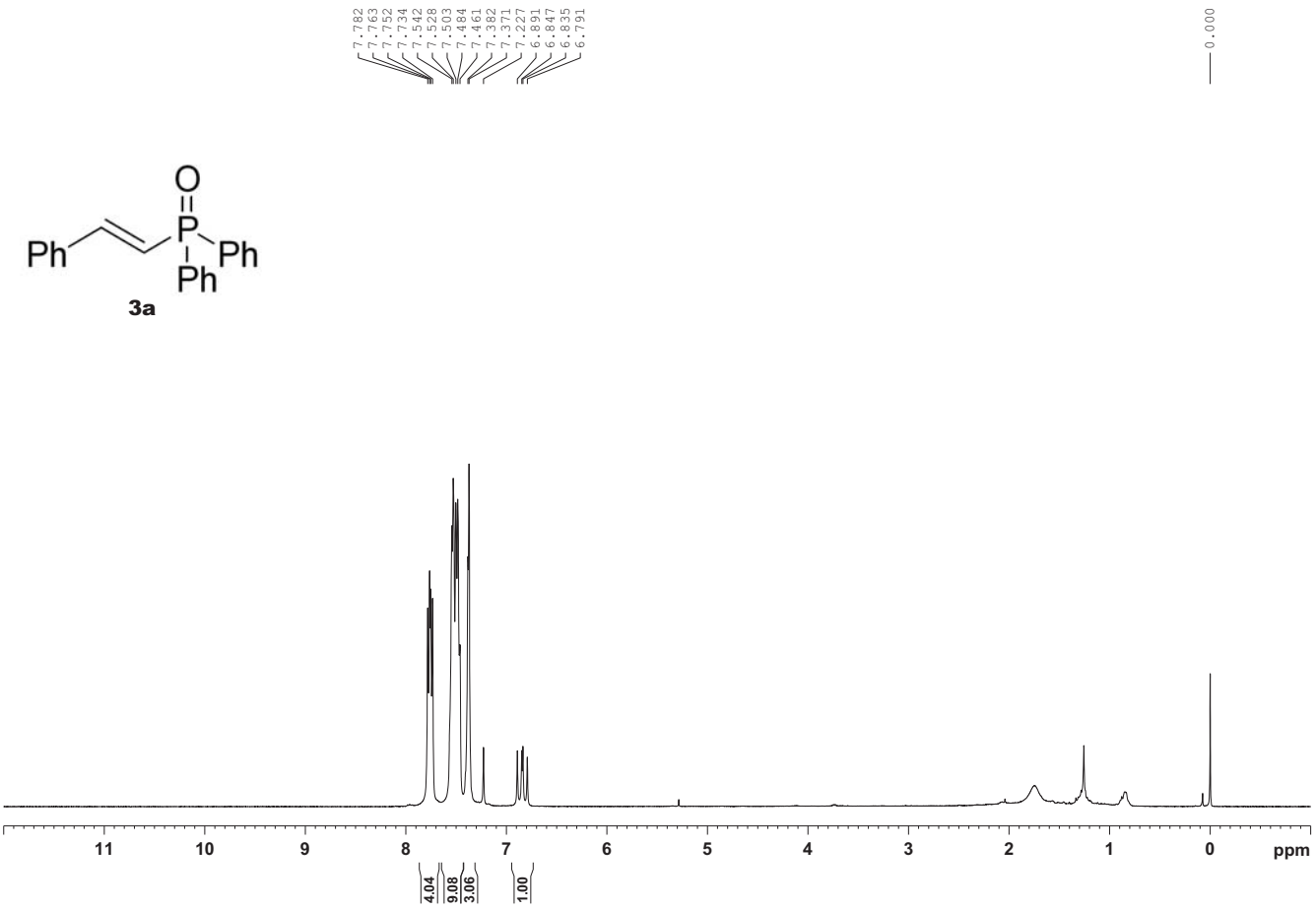


White oil; (68 mg, 56%); R_f = 0.20 (hexanes/ethyl acetate 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (dd, J = 7.6 Hz, J = 11.6 Hz, 4H), 7.60 (d, J = 7.2 Hz, 2H), 7.56–7.39 (m, 11H), 7.33–7.25 (m, 8H), 6.77 (dd, J = 17.6 Hz, J = 22.0 Hz, 1H), 5.17 (s, 2H), 3.20 (t, J = 7.2 Hz, 2H), 2.98 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 160.9, 146.3 (d, $J_{C\text{-}P}$ = 4.0 Hz), 144.8, 137.2, 134.4, 134.3 (d, $J_{C\text{-}P}$ = 18.0 Hz), 132.2 (d, $J_{C\text{-}P}$ = 105.0 Hz), 131.8, 131.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.7 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.2, 128.1 (d, $J_{C\text{-}P}$ = 12.0 Hz), 128.0, 127.9, 127.8, 127.7, 127.4, 127.3, 127.2, 125.8, 119.0 (d, $J_{C\text{-}P}$ = 103.0 Hz), 65.3, 30.4, 22.8; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.4; ESI-HRMS (ESI, m/z): Calcd for $C_{39}H_{33}NO_4P$, $[M + H]^+$: 610.2142, found 610.2145.

(5-((4-Hydroxy-2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2-phenylpent-2-en-1-yl)diphenyl phosphine oxide (3ah)



Colourless oil; (16 mg, 16%); R_f = 0.14 (hexanes/ethyl acetate 1:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65–7.60 (m, 4H), 7.41 (dt, J = 1.2 Hz, J = 7.6 Hz, 2H), 7.32 (dt, J = 3.2 Hz, J = 7.6 Hz, 4H), 7.09 (brs, 5H), 5.84 (dd, J = 7.2 Hz, J = 12.4 Hz, 1H), 3.95–3.88 (m, 1H), 3.76 (t, J = 6.4 Hz, 2H), 3.68 (d, J = 14.8 Hz, 2H), 2.40–2.34 (m, 2H), 1.77 (m, 2H), 1.43 (t, J = 12.0 Hz, 2H), 1.15 (s, 6H), 1.12 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.1 (d, $J_{C\text{-}P}$ = 1.0 Hz), 132.6 (d, $J_{C\text{-}P}$ = 87.0 Hz), 131.6 (d, $J_{C\text{-}P}$ = 9.0 Hz), 131.5 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 131.0 (d, $J_{C\text{-}P}$ = 9.0 Hz), 128.2 (d, $J_{C\text{-}P}$ = 12.0 Hz), 127.9, 126.7, 126.5, 75.8, 63.1, 60.0, 48.1, 33.7 (d, $J_{C\text{-}P}$ = 67.0 Hz), 33.1, 29.0, 21.0; ^{31}P NMR ($CDCl_3$, 162 MHz): 24.3; ESI-HRMS (ESI, m/z): Calcd for $C_{32}H_{41}NO_3P$, $[M + H]^+$: 518.2819, found 518.2815.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6.847
6.835
6.791
0.000
O
P
Ph
Ph
Ph
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ppm
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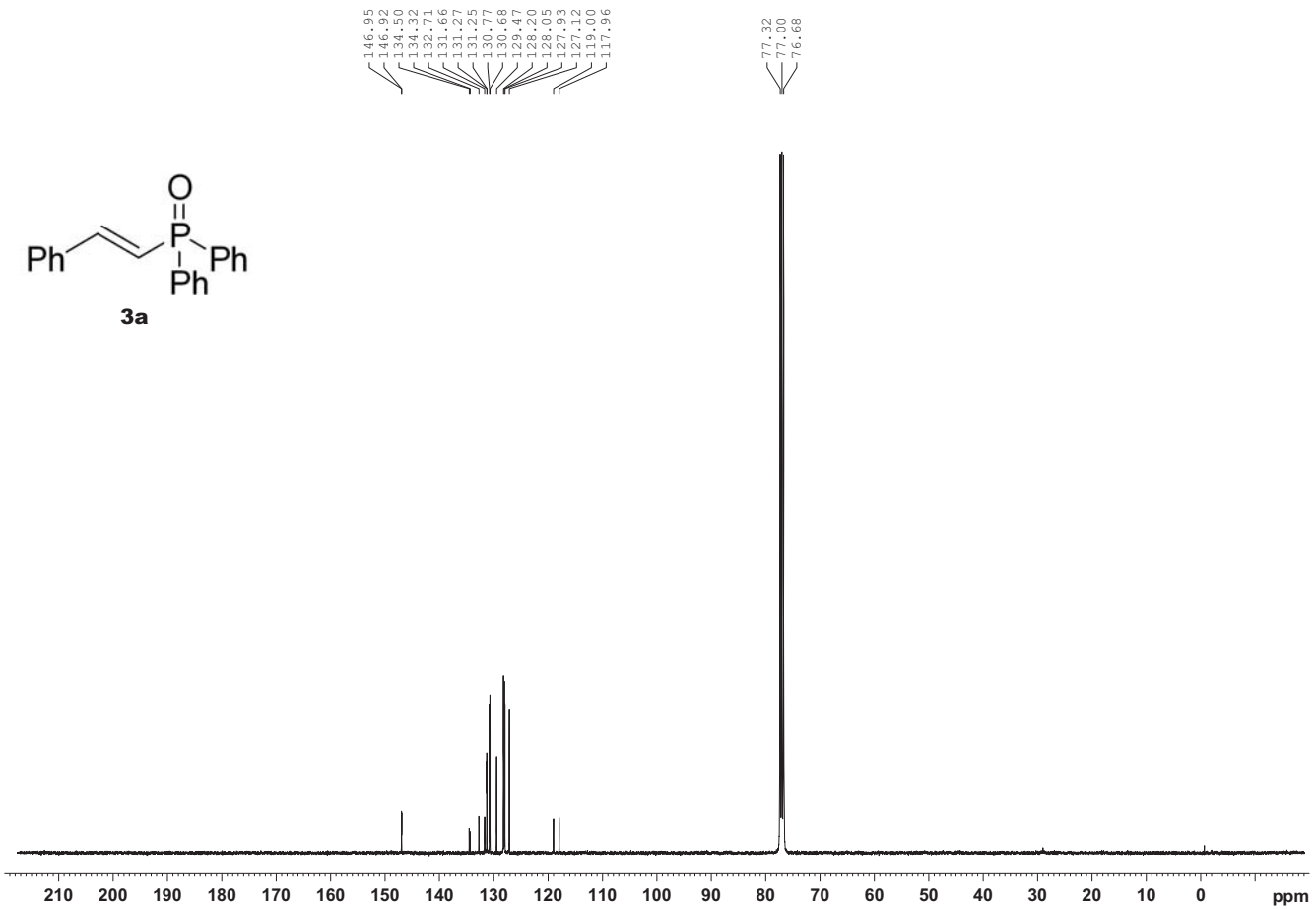
1.00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77.00
76.68
O
P
Ph
Ph
Ph
3a
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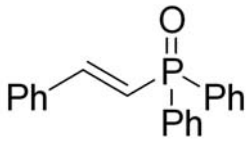
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Ph

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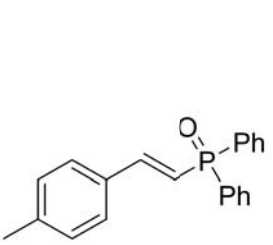
O
P
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Ph

3b

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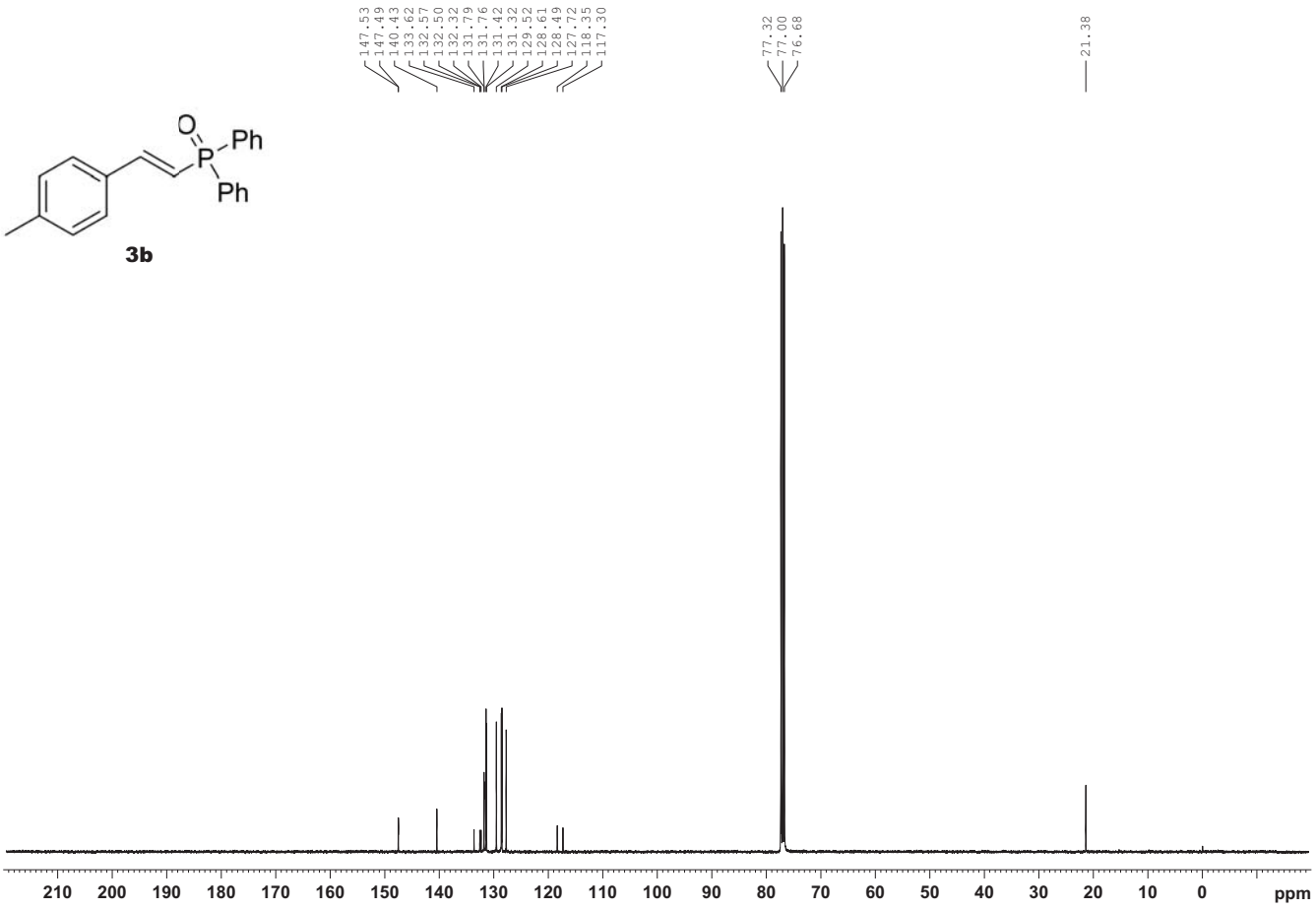
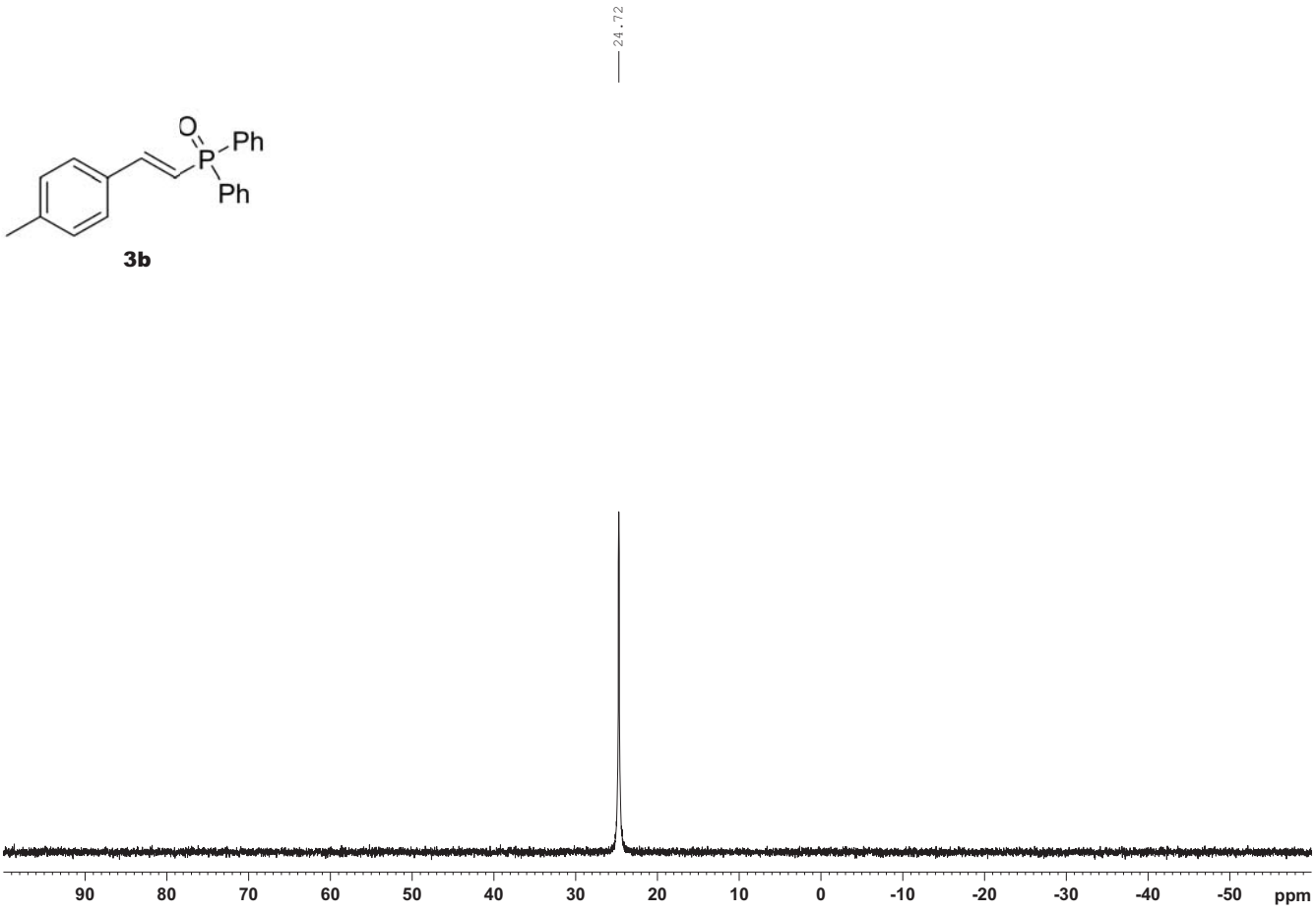
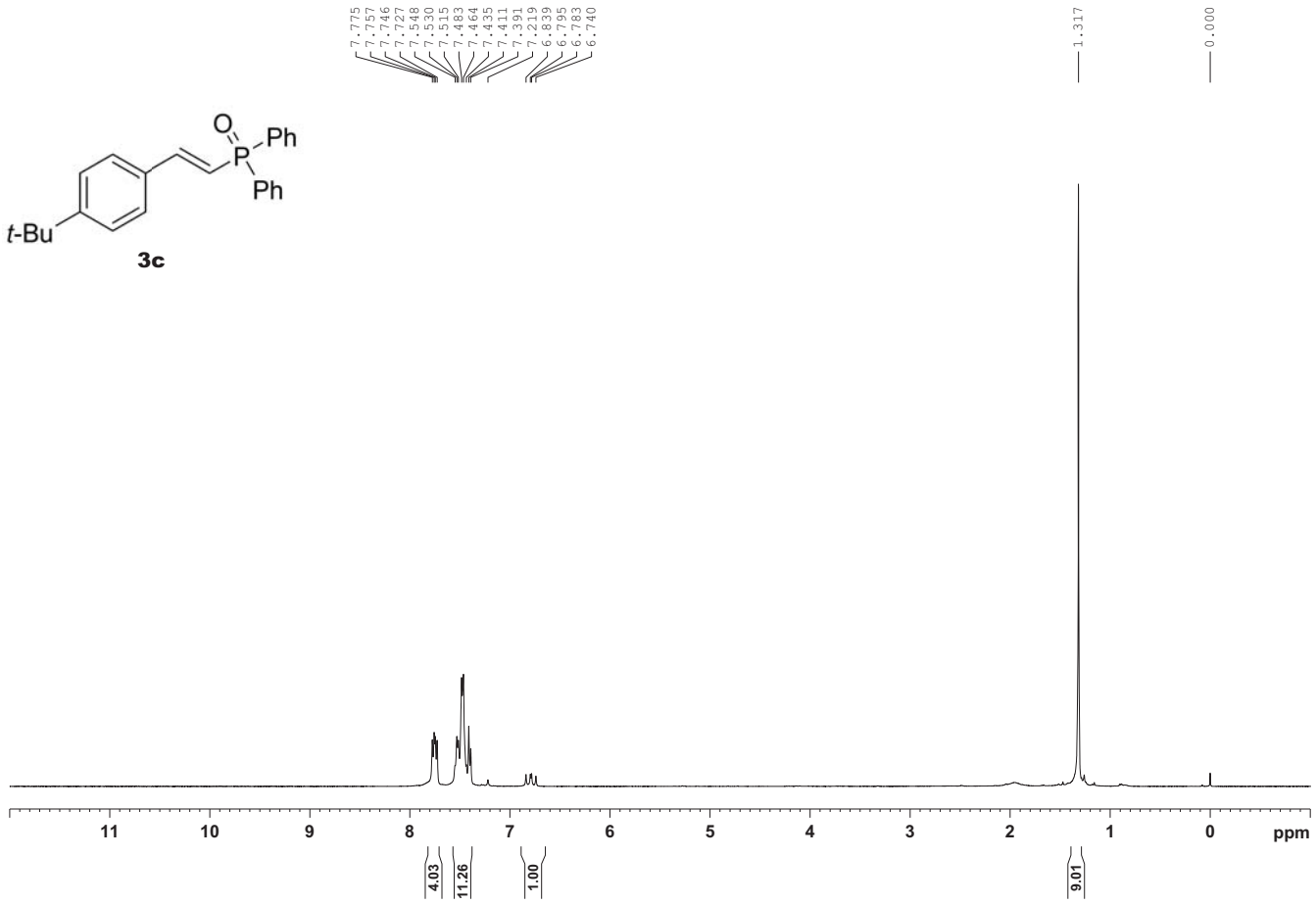
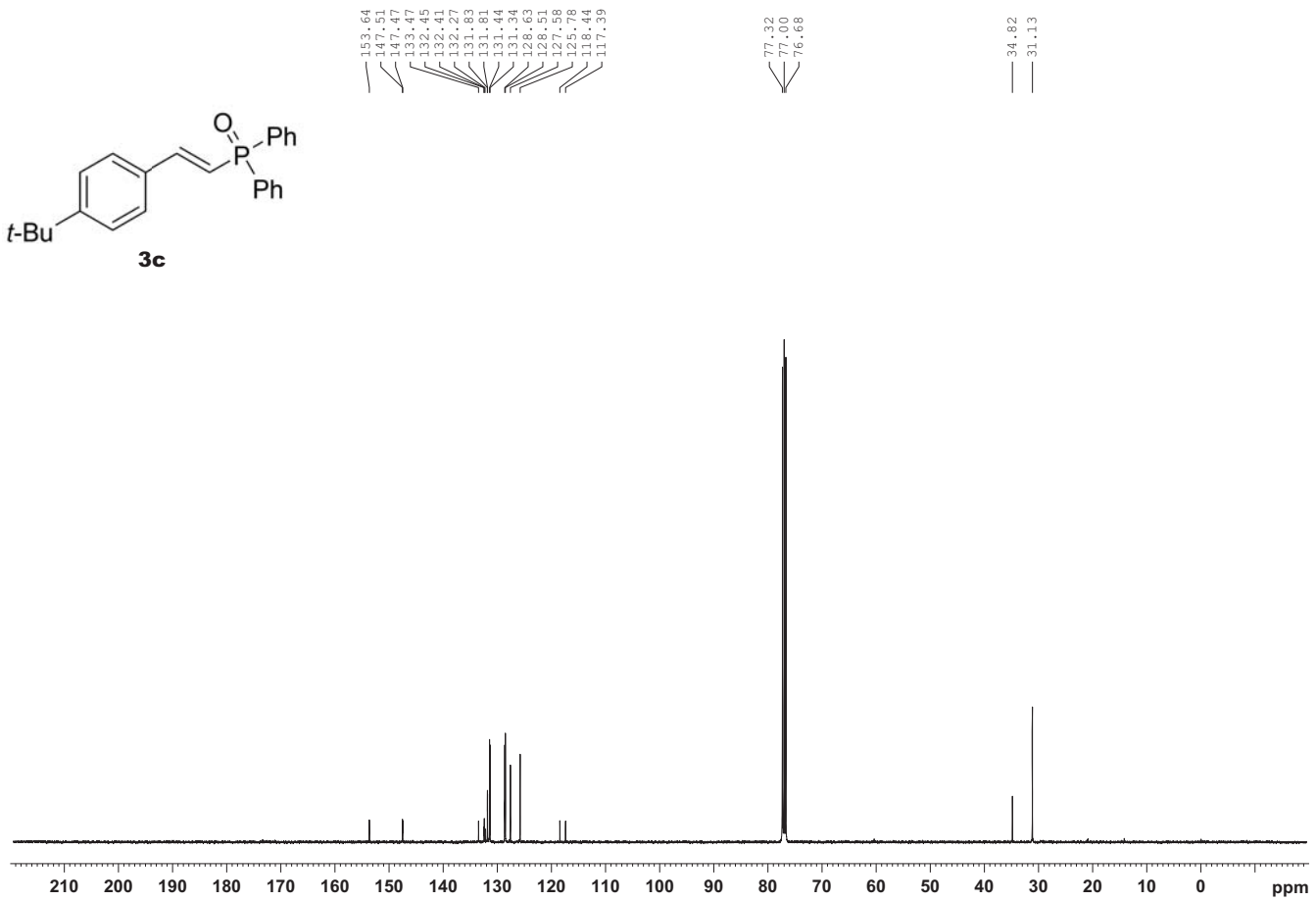
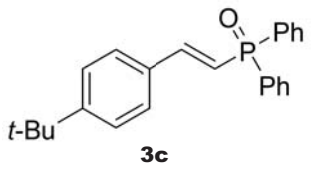
1.02
3.01


147.53
147.49
140.43
133.62
132.57
132.50
132.32
131.79
131.76
131.42
131.32
129.52
128.61
128.49
127.72
118.35
117.30
77.32
77.00
76.68
21.38
O
P
Ph
Ph
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


24.72
O
P
Ph
Ph
3b
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

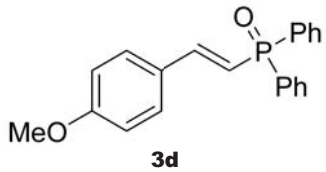

7.775
7.757
7.746
7.727
7.548
7.530
7.515
7.483
7.464
7.435
7.411
7.391
7.219
6.839
6.795
6.783
6.740
1.317
0.000
O
P
Ph
Ph
t-Bu
3c
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
11.26
1.00
9.01


153.64
147.51
147.47
133.47
132.45
132.41
132.27
131.83
131.81
131.44
131.34
128.63
128.51
127.58
125.78
118.44
117.39
77.32
77.00
76.68
34.82
31.13
O
P
Ph
Ph
t-Bu
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


O
P
Ph
Ph
t-Bu

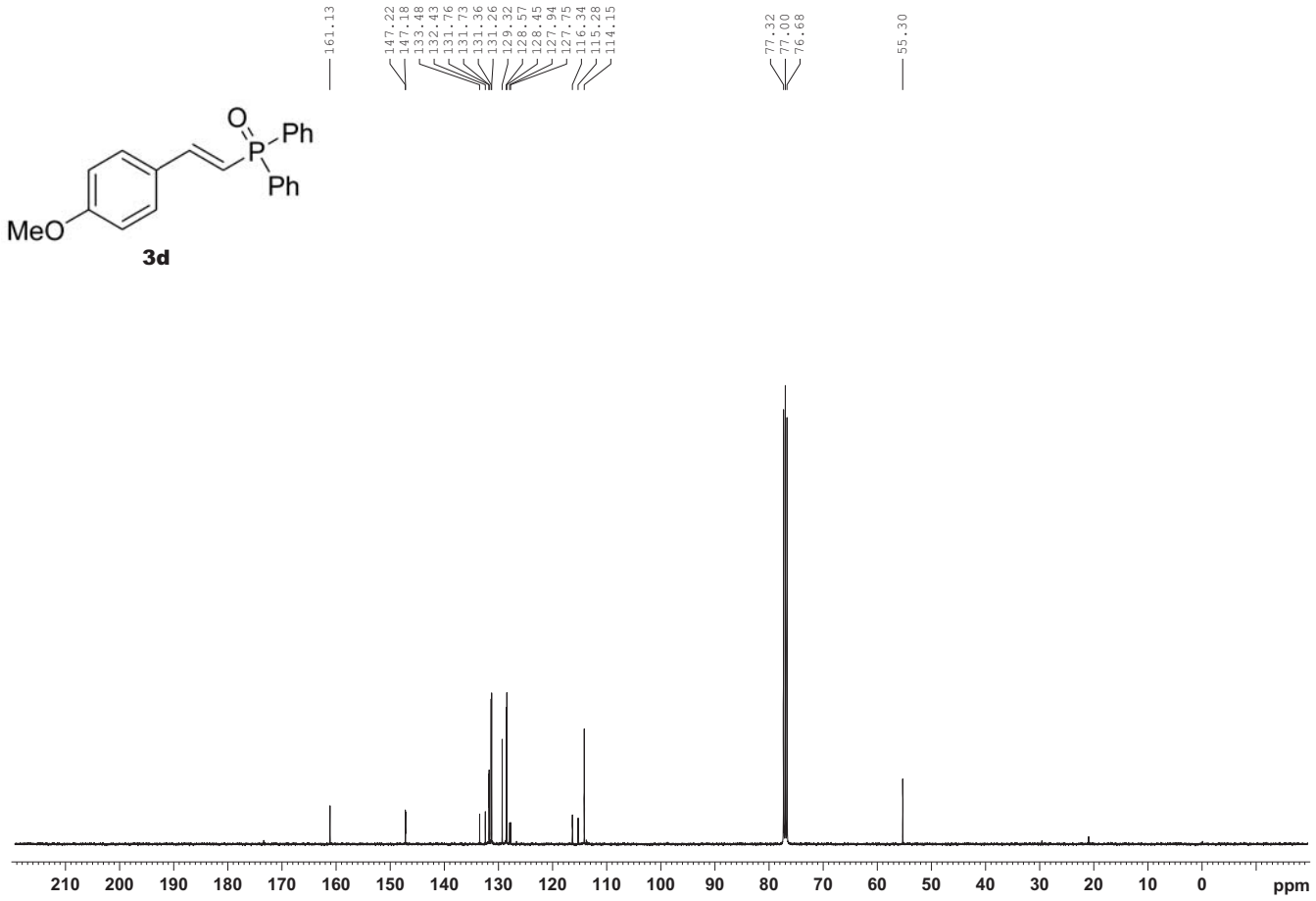
3c

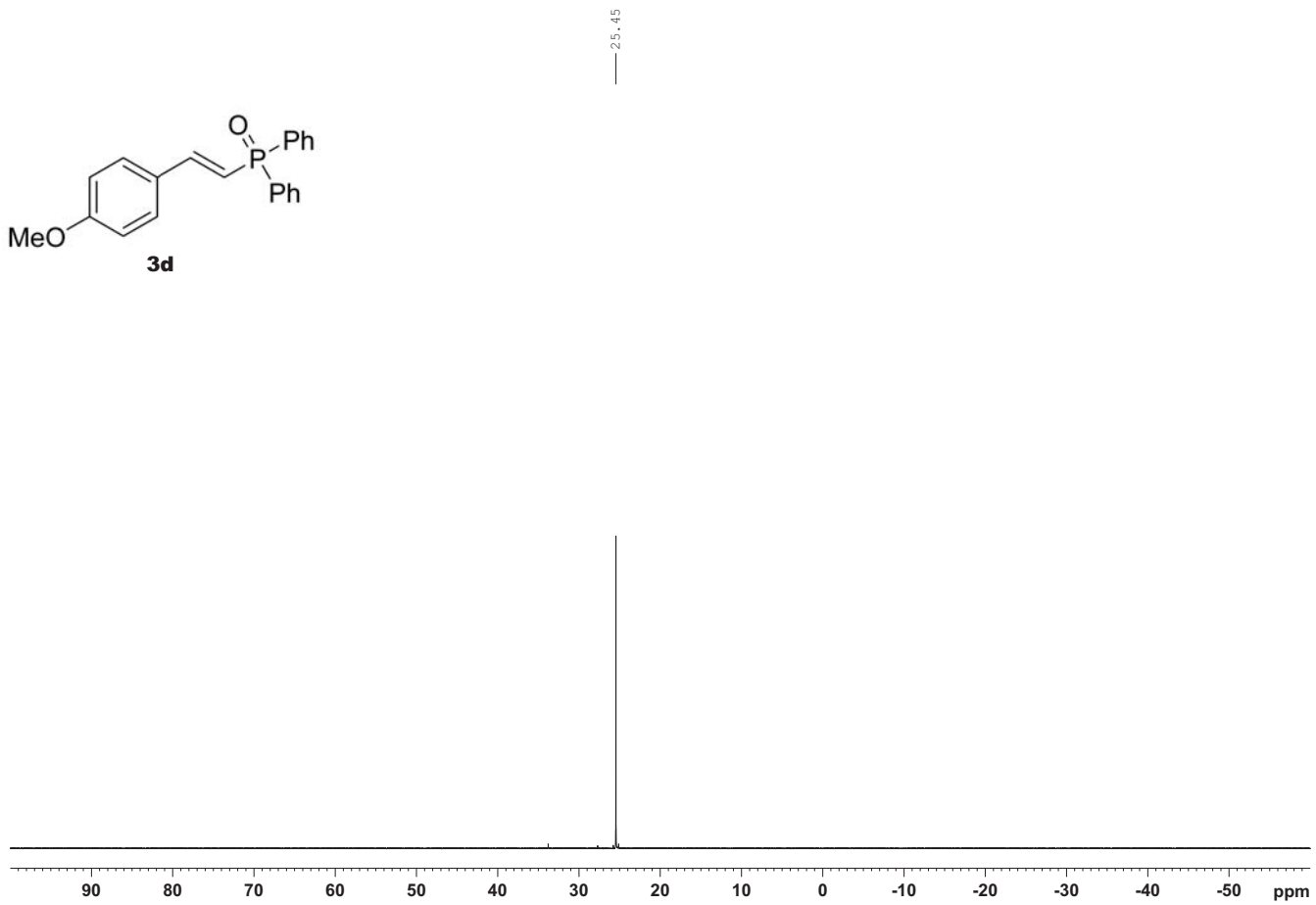
25.14
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

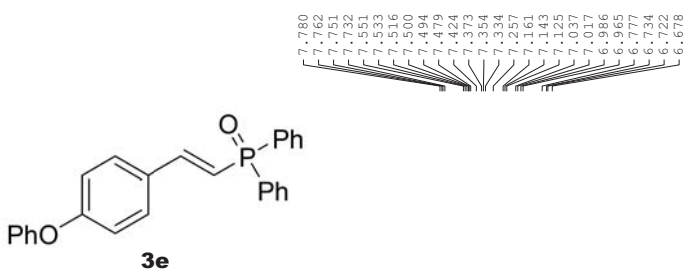

O
P
Ph
Ph
MeO

3d

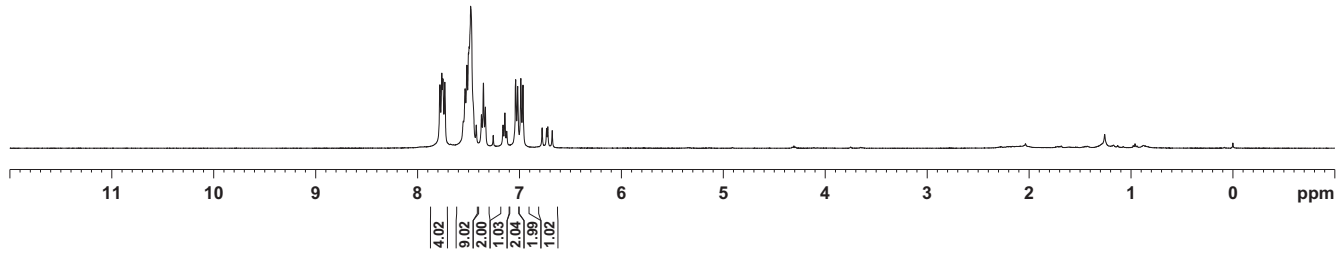
7.778
7.761
7.757
7.748
7.731
7.727
7.554
7.551
7.536
7.532
7.526
7.518
7.514
7.493
7.486
7.475
7.467
7.463
7.458
7.450
7.434
7.428
7.384
7.266
6.904
6.882
6.715
6.672
6.659
6.616
3.823
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
2.04
7.06
1.99
1.00
3.01

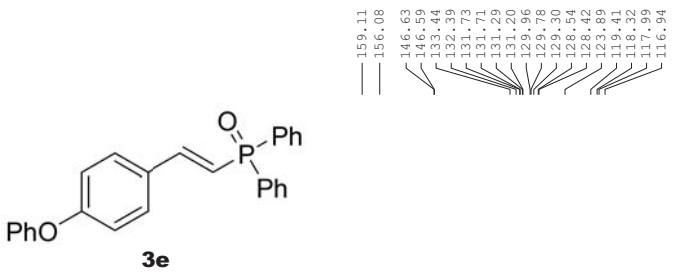

161.13
147.22
147.18
133.48
132.43
131.76
131.73
131.36
131.26
129.32
128.57
128.45
127.94
127.75
116.34
115.28
114.15
77.32
77.00
76.68
55.30
O
P
Ph
Ph
MeO
3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


25.45
O
P
Ph
Ph
MeO
3d
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

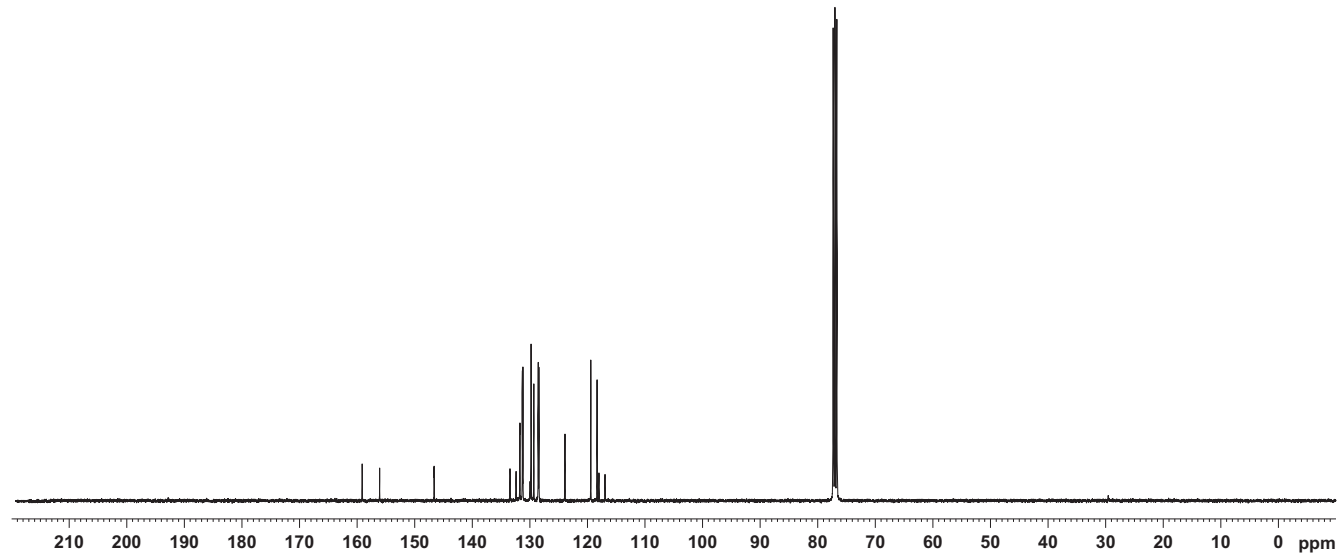

7.780
7.762
7.751
7.732
7.551
7.533
7.516
7.500
7.494
7.479
7.424
7.373
7.354
7.334
7.257
7.161
7.143
7.125
7.037
7.017
6.986
6.965
6.777
6.734
6.722
6.678
-0.000
O
P
Ph
Ph
PhO

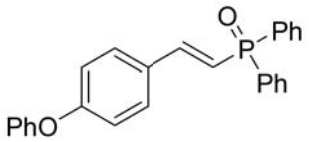
3e


11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.02
9.02
2.00
1.03
2.04
1.99
1.02


159.11
156.08
146.63
146.59
133.44
132.39
131.73
131.71
131.29
131.20
129.96
129.78
129.30
128.54
128.42
123.89
119.41
118.32
117.99
116.94
77.32
77.00
76.68
O
P
Ph
Ph
PhO

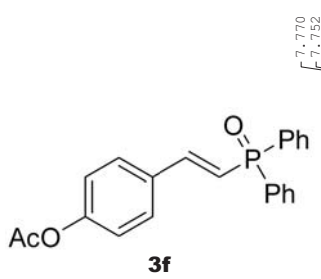
3e


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


O
P
Ph
Ph
PhO

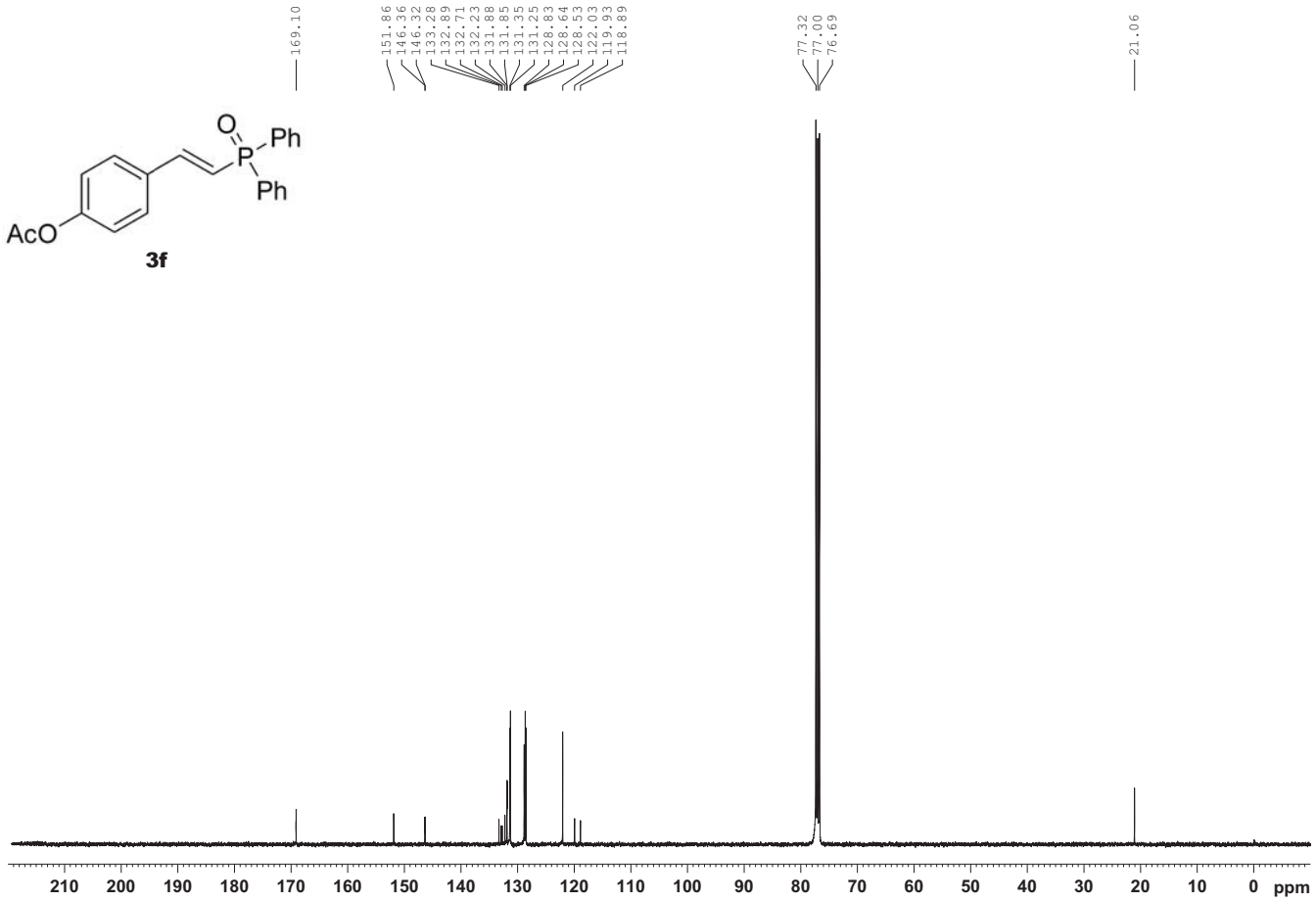
3e

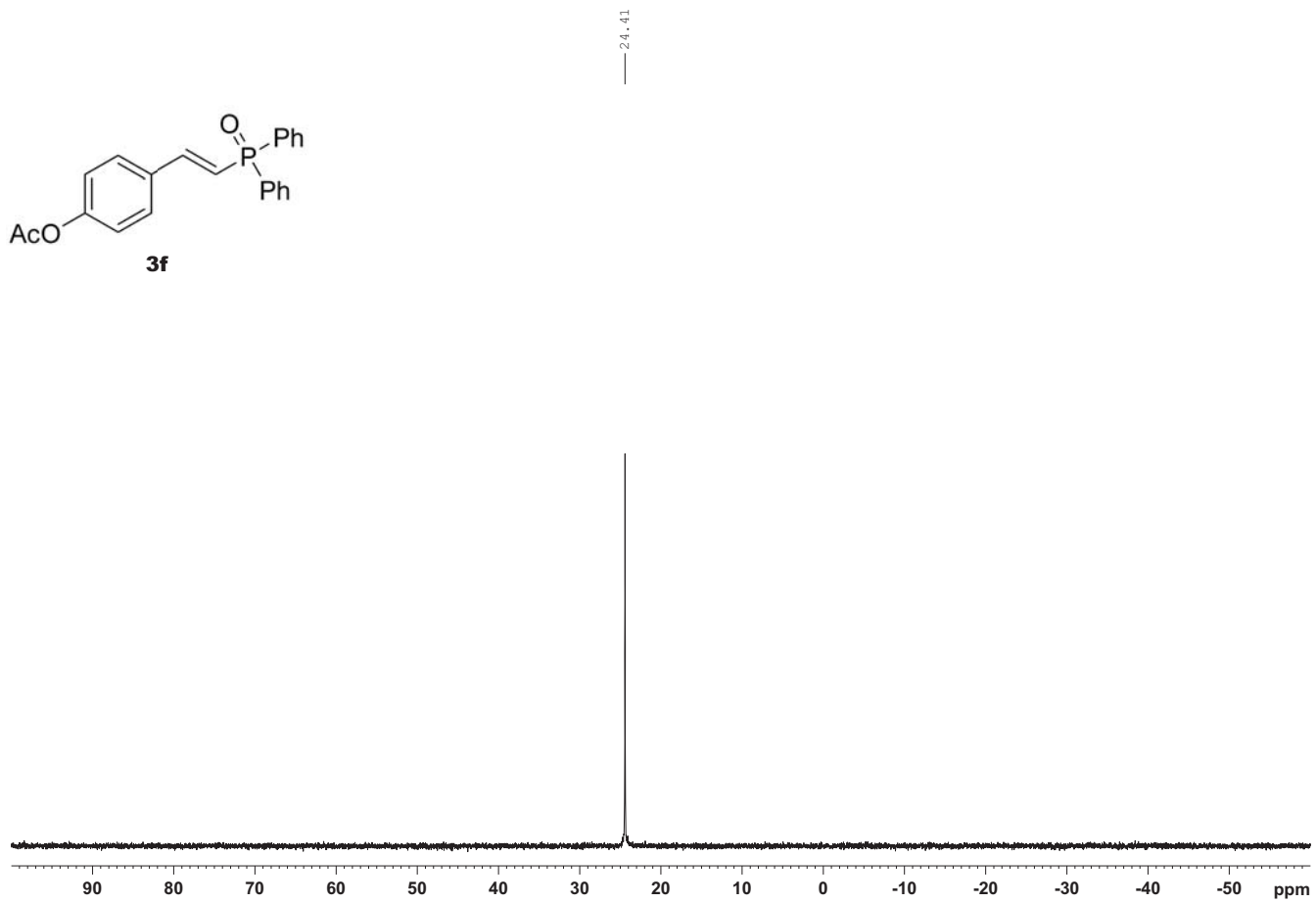
24.68
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

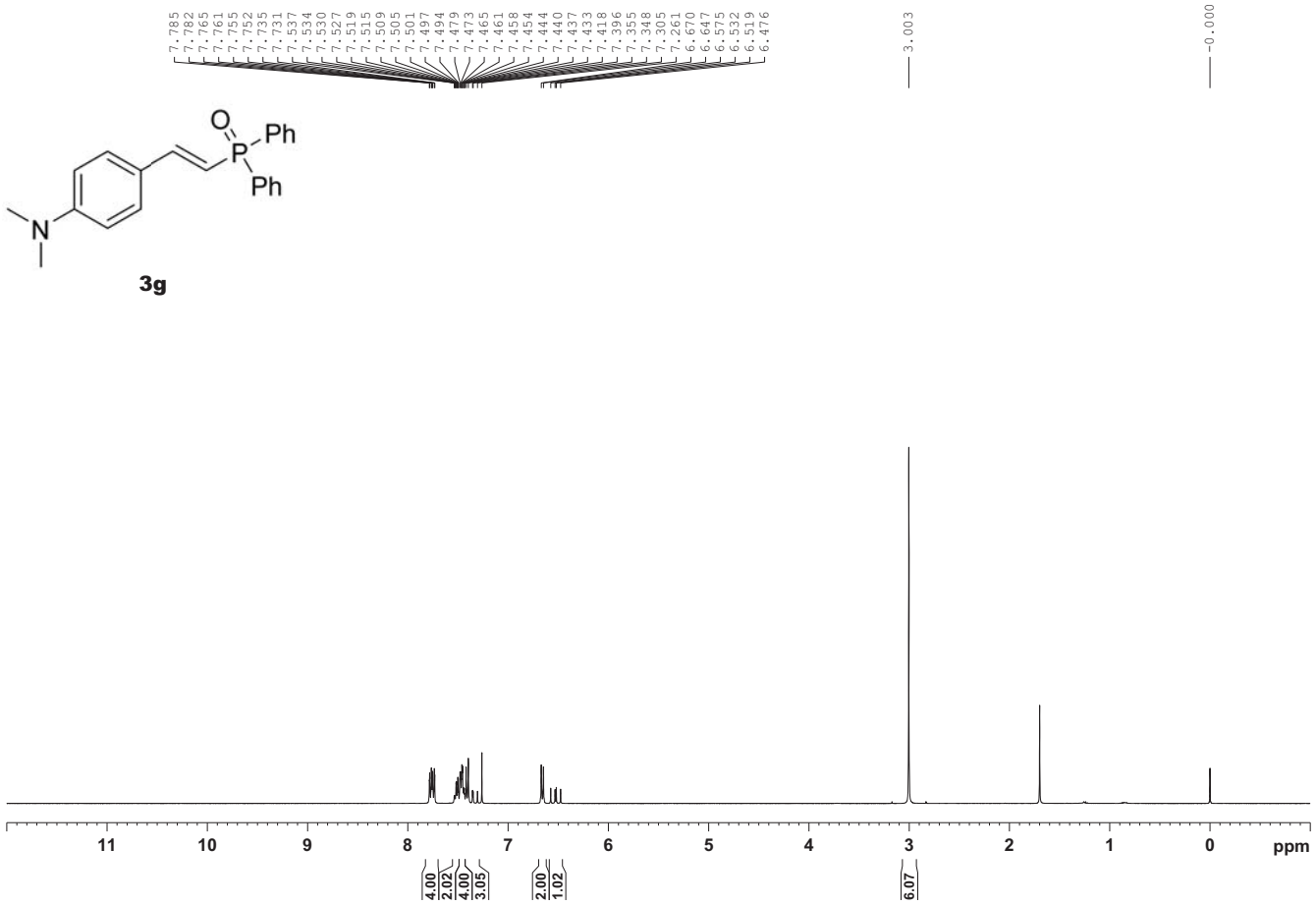

O
P
Ph
Ph
AcO

3f

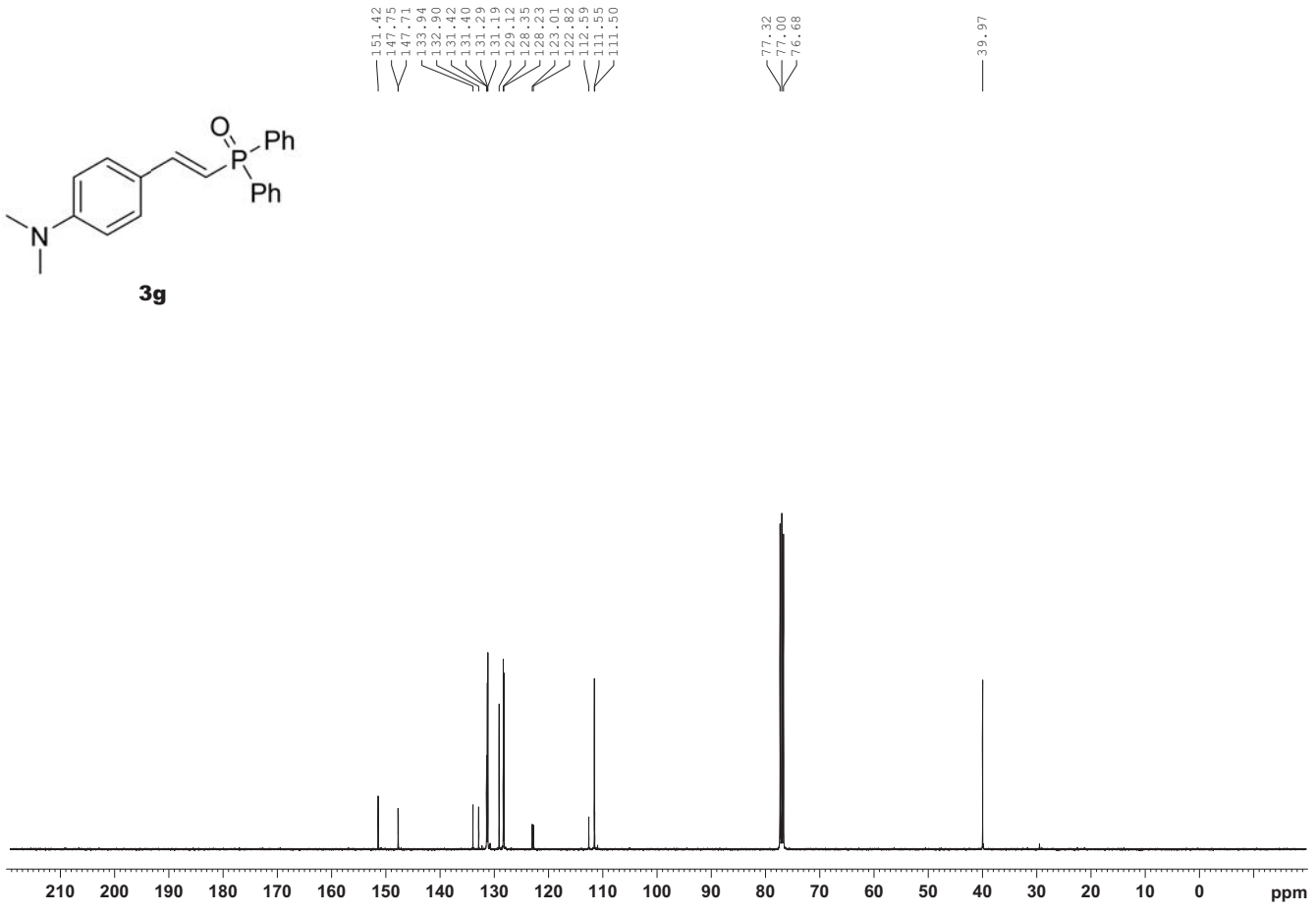
7.770
7.752
7.749
7.740
7.722
7.719
7.562
7.555
7.549
7.533
7.503
7.496
7.485
7.478
7.467
7.464
7.461
7.444
7.264
7.127
7.106
6.841
6.798
6.786
6.742
2.304
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.05
9.08
2.03
1.02
3.00


169.10
151.86
146.36
146.32
133.28
132.89
132.71
132.23
131.88
131.85
131.35
131.25
128.83
128.64
128.53
122.03
119.93
118.89
77.32
77.00
76.69
21.06
O
P
Ph
Ph
AcO
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

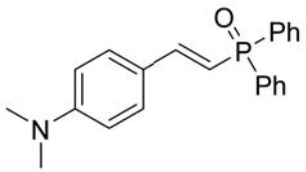

24.41
O
P
Ph
Ph
AcO
3f
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm


7.785
7.782
7.765
7.761
7.755
7.752
7.735
7.731
7.537
7.534
7.530
7.527
7.519
7.515
7.509
7.505
7.501
7.497
7.494
7.479
7.473
7.465
7.461
7.458
7.454
7.444
7.440
7.437
7.433
7.418
7.396
7.355
7.348
7.305
7.261
6.670
6.647
6.575
6.532
6.519
6.476
3.003
-0.000
O
P
Ph
Ph
N
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.00
2.02
4.00
3.05
2.00
1.02
6.07

3g

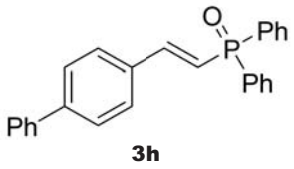

151.42
147.75
147.71
133.94
132.90
131.42
131.40
131.29
131.19
129.12
128.35
128.23
123.01
122.82
112.59
111.55
111.50
77.32
77.00
76.68
39.97
O
P
Ph
Ph
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3g

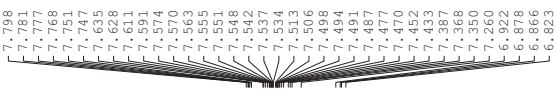


3g

25.97
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm



3h


7.798
7.781
7.777
7.768
7.751
7.747
7.635
7.628
7.611
7.591
7.574
7.570
7.563
7.555
7.551
7.548
7.542
7.537
7.534
7.513
7.506
7.498
7.494
7.491
7.487
7.477
7.470
7.452
7.433
7.387
7.368
7.350
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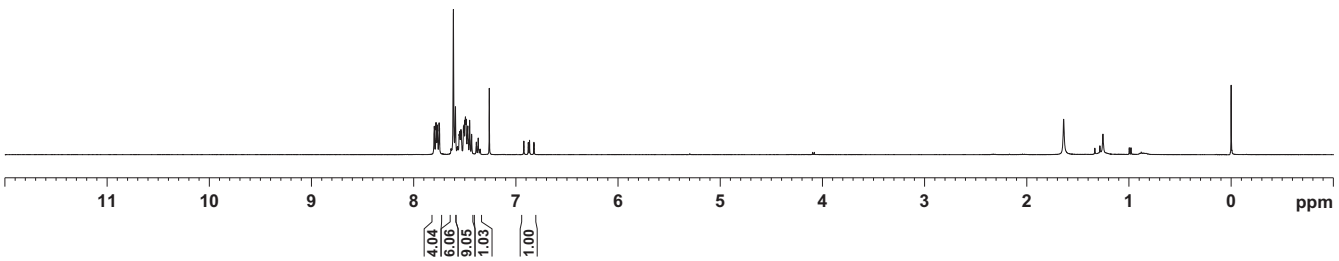
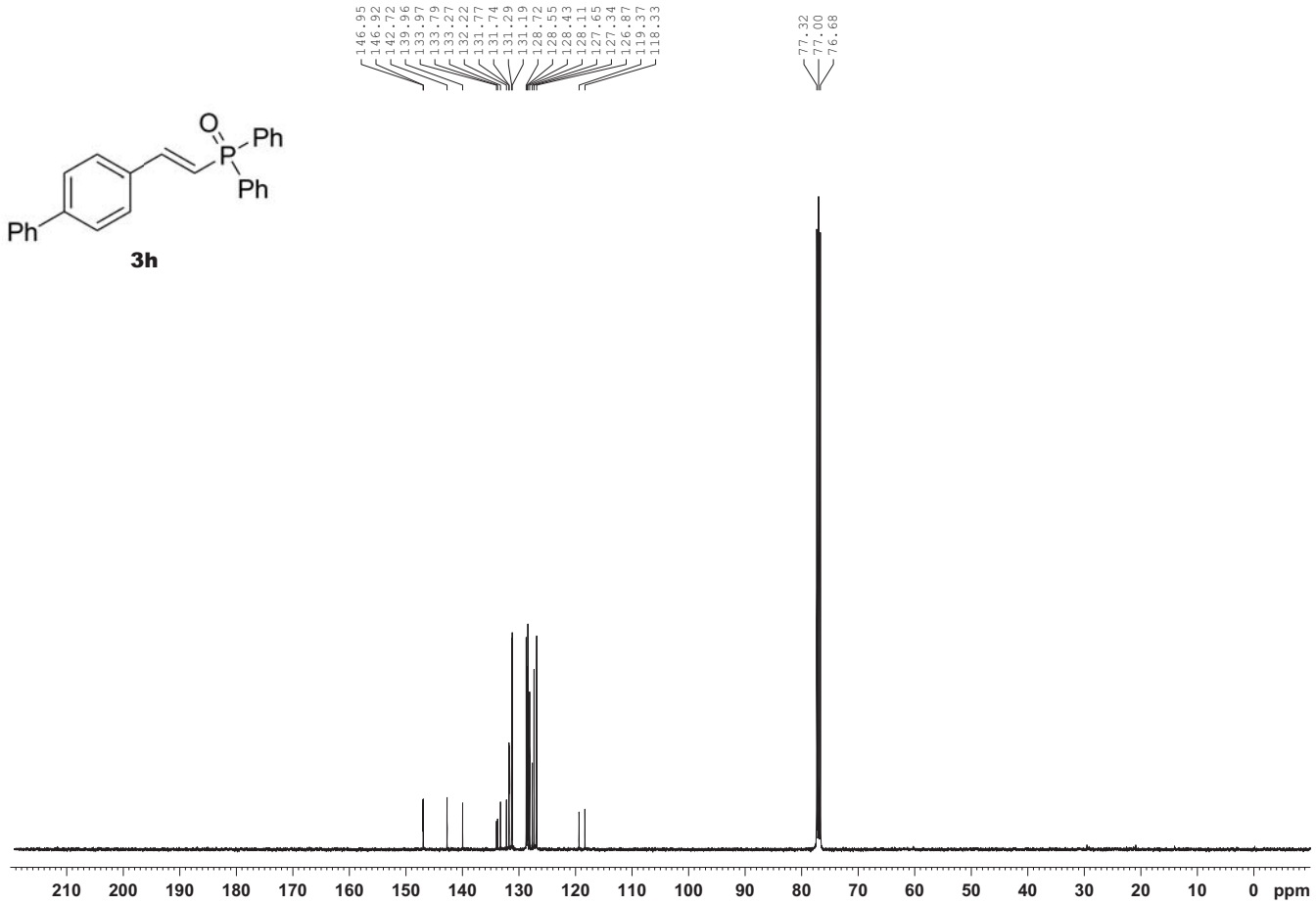
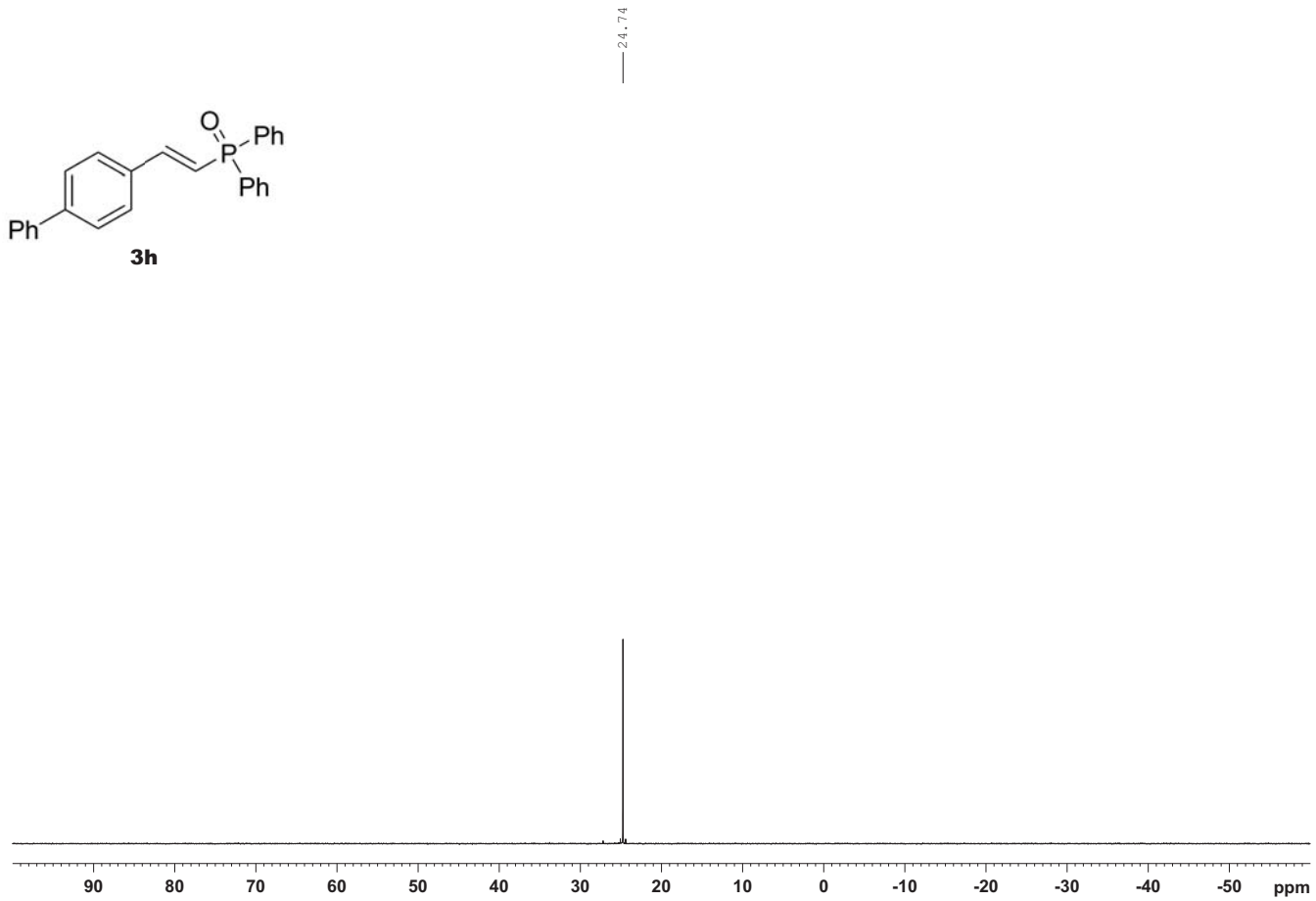
6.922
6.878
6.866
6.823


-0.000

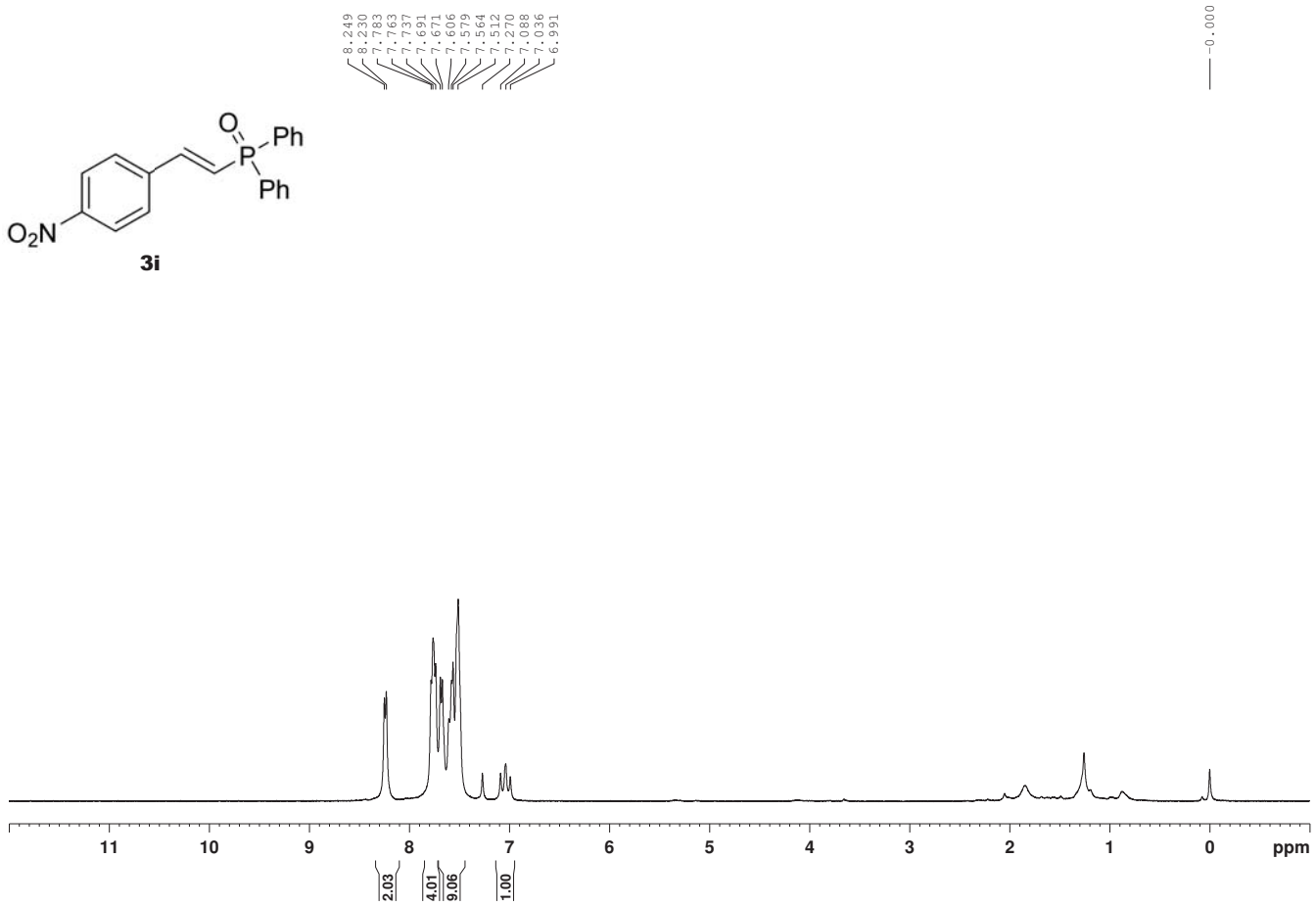

11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.04
6.06
9.05
1.03
1.00

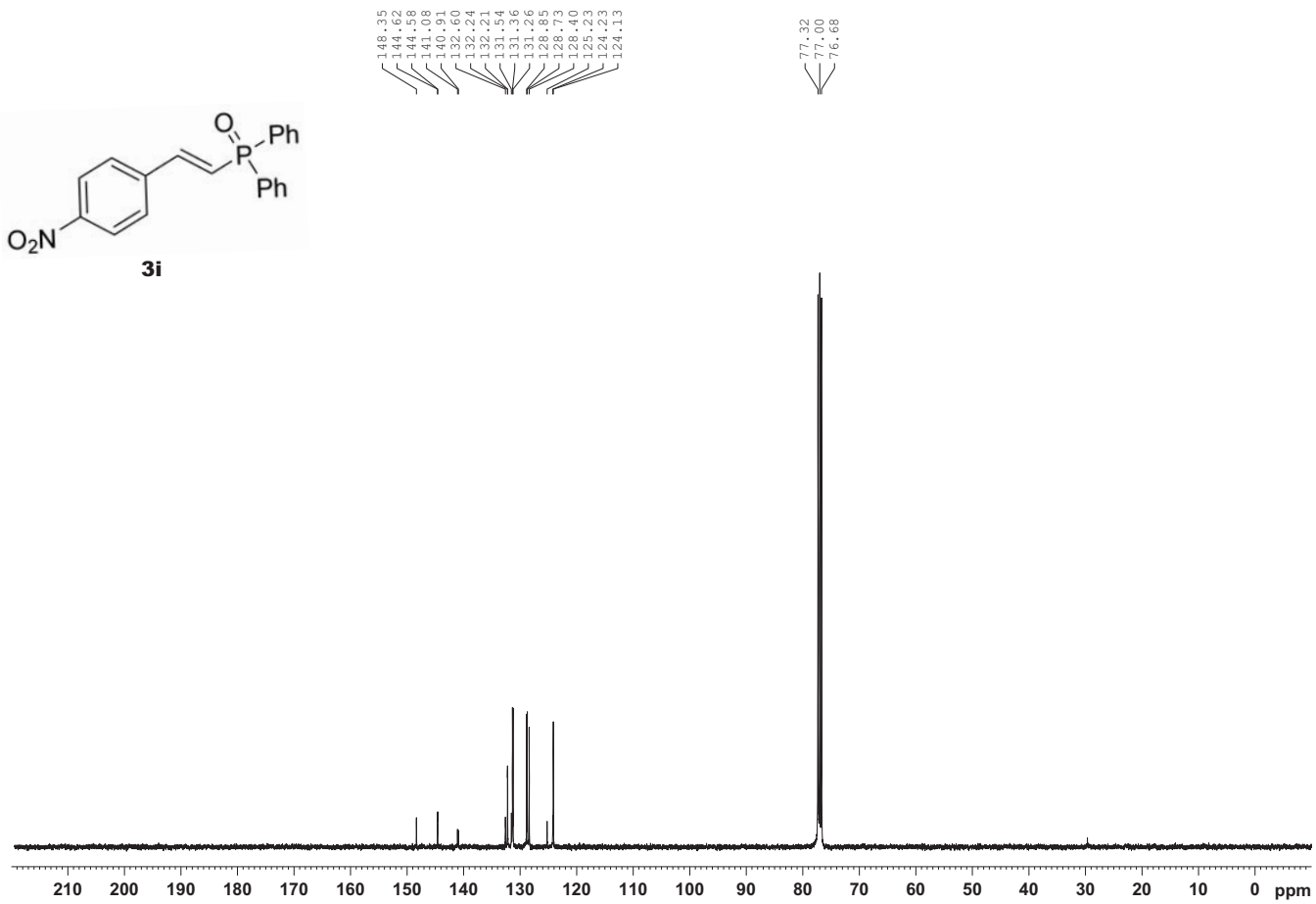

146.95
146.92
142.72
139.96
133.97
133.79
133.27
132.22
131.77
131.74
131.29
131.19
128.72
128.55
128.43
128.11
127.65
127.34
126.87
119.37
118.33
77.32
77.00
76.68
O
P
Ph
Ph
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

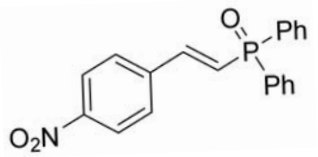
3h


24.74
O
P
Ph
Ph
Ph
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

3h

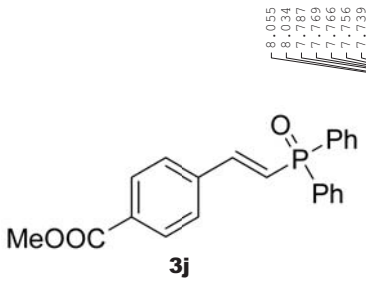

8.249
8.230
7.783
7.763
7.737
7.691
7.671
7.606
7.579
7.564
7.512
7.270
7.088
7.036
6.991
-0.000
O
P
Ph
Ph
O2N
3i
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.03
4.01
9.06
1.00


148.35
144.62
144.58
141.08
140.91
132.60
132.24
132.21
131.54
131.36
131.26
128.85
128.73
128.40
125.23
124.23
124.13
77.32
77.00
76.68
O
P
Ph
Ph
O2N
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


O
P
Ph
Ph
O_2N

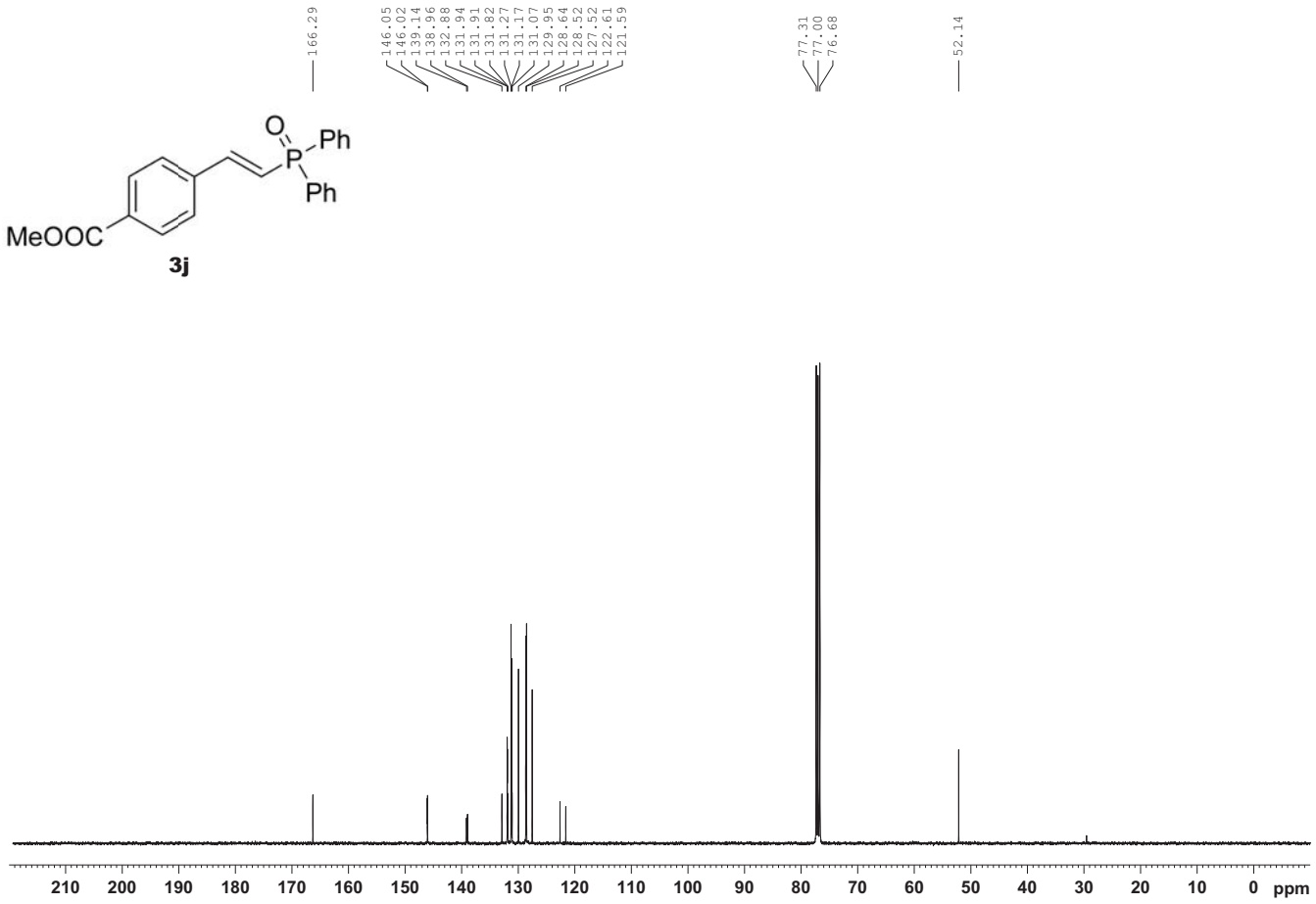
3i

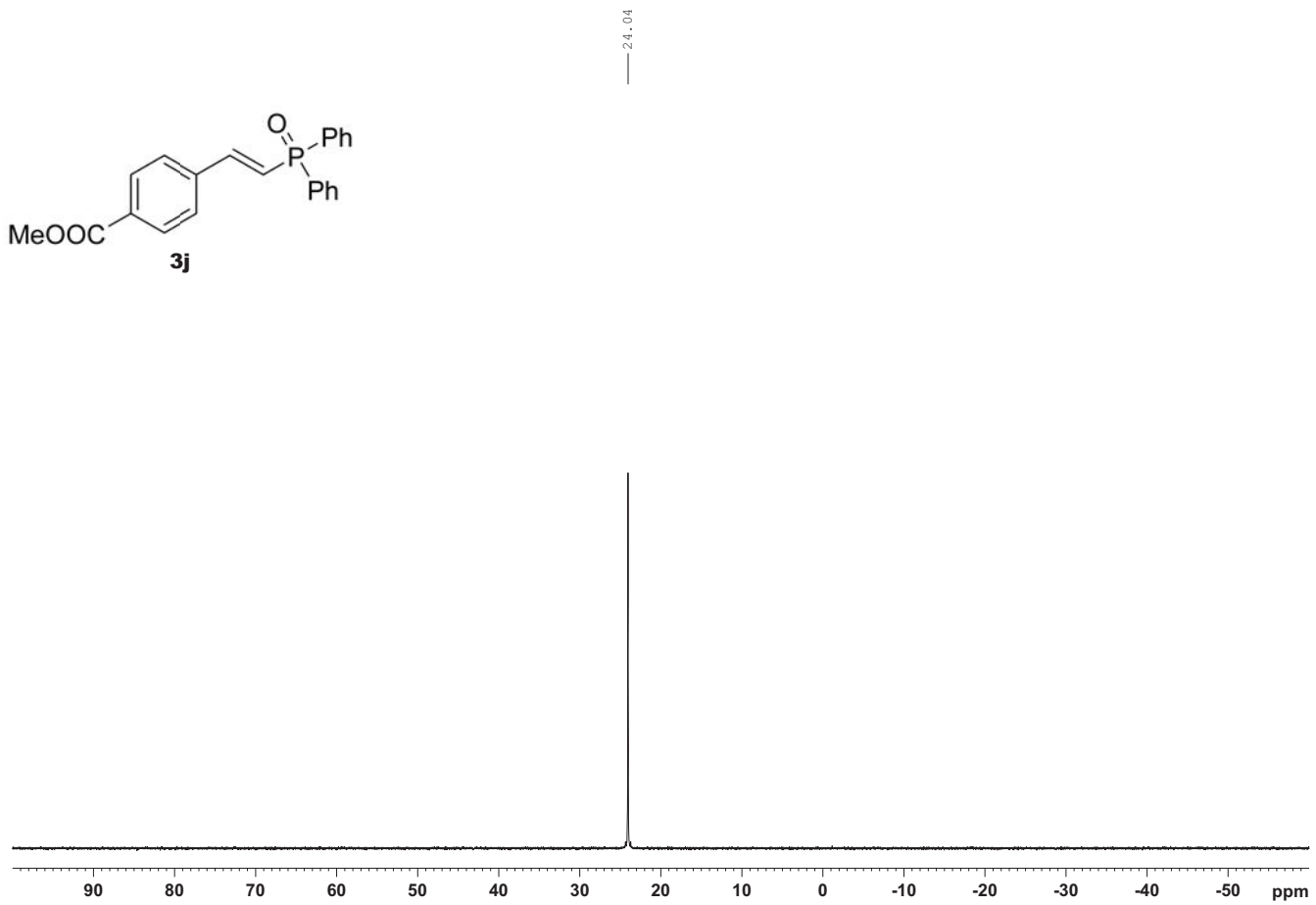
23.46
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

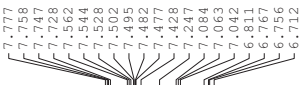

O
P
Ph
Ph
MeOOC

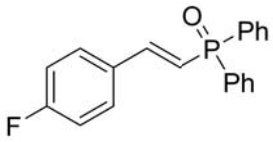
3j

8.055
8.034
7.787
7.769
7.766
7.756
7.739
7.735
7.599
7.578
7.560
7.556
7.542
7.538
7.515
7.509
7.496
7.492
7.489
7.479
7.475
7.471
7.274
7.020
6.977
6.965
6.922
3.920
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
4.10
9.02
1.02
3.01

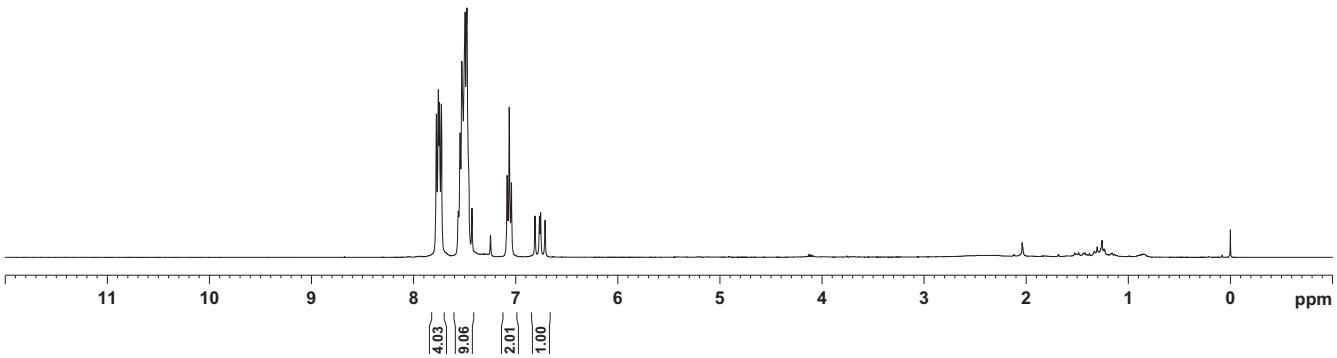

166.29
146.05
146.02
139.14
138.96
132.88
131.94
131.91
131.82
131.27
131.17
131.07
129.95
128.64
128.52
127.52
122.61
121.59
77.31
77.00
76.68
52.14
O
P
Ph
Ph
MeOOC
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


24.04
O
P
Ph
Ph
MeOOC
3j
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm


7.777
7.758
7.747
7.728
7.562
7.544
7.528
7.502
7.495
7.482
7.477
7.428
7.247
7.084
7.063
7.042
6.811
6.767
6.756
6.712
0.000


O
Ph
P
Ph
F

3k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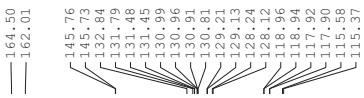
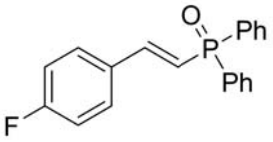
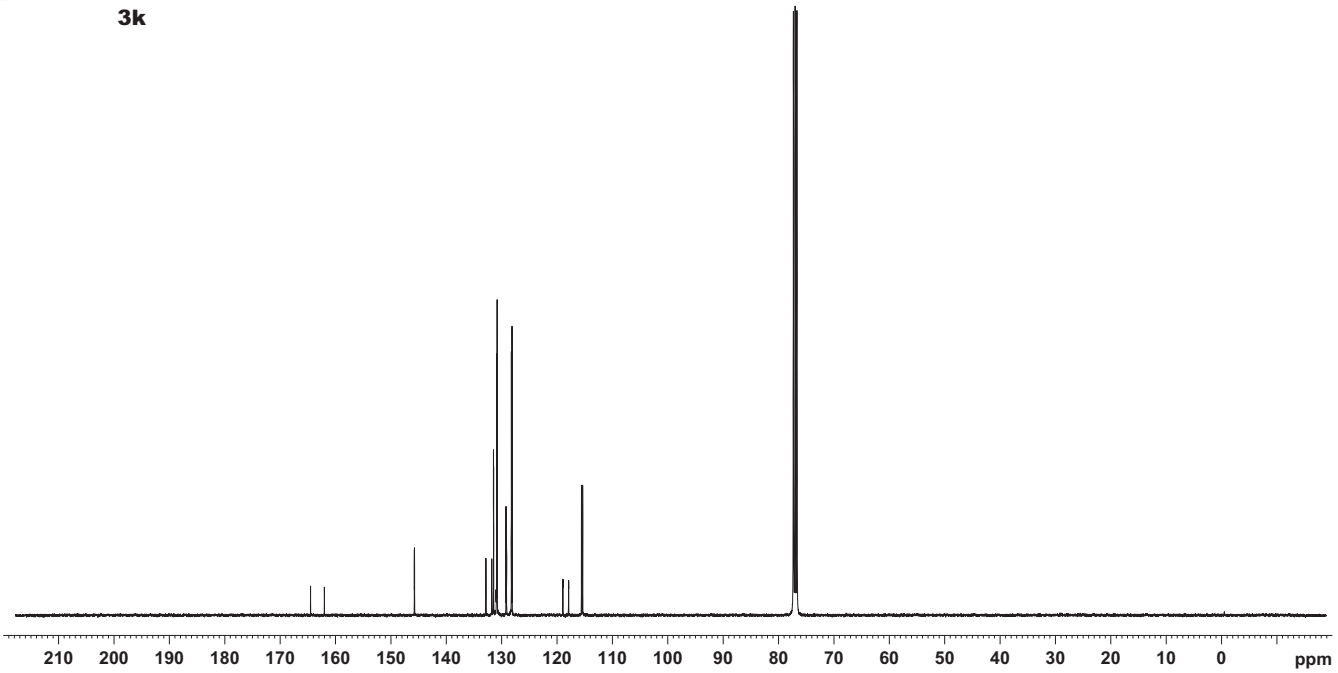
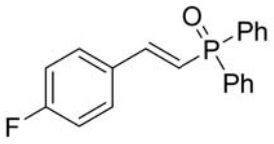
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
9.06
2.01
1.00


164.50
162.01
145.76
145.73
132.84
131.79
131.48
131.45
130.99
130.96
130.91
130.81
129.21
129.13
128.24
128.12
118.96
118.94
117.92
117.90
115.58
115.37
77.32
77.00
76.68


O
Ph
P
Ph
F

3k


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


O
Ph
P
Ph
F

3k

24.44
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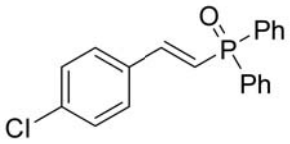
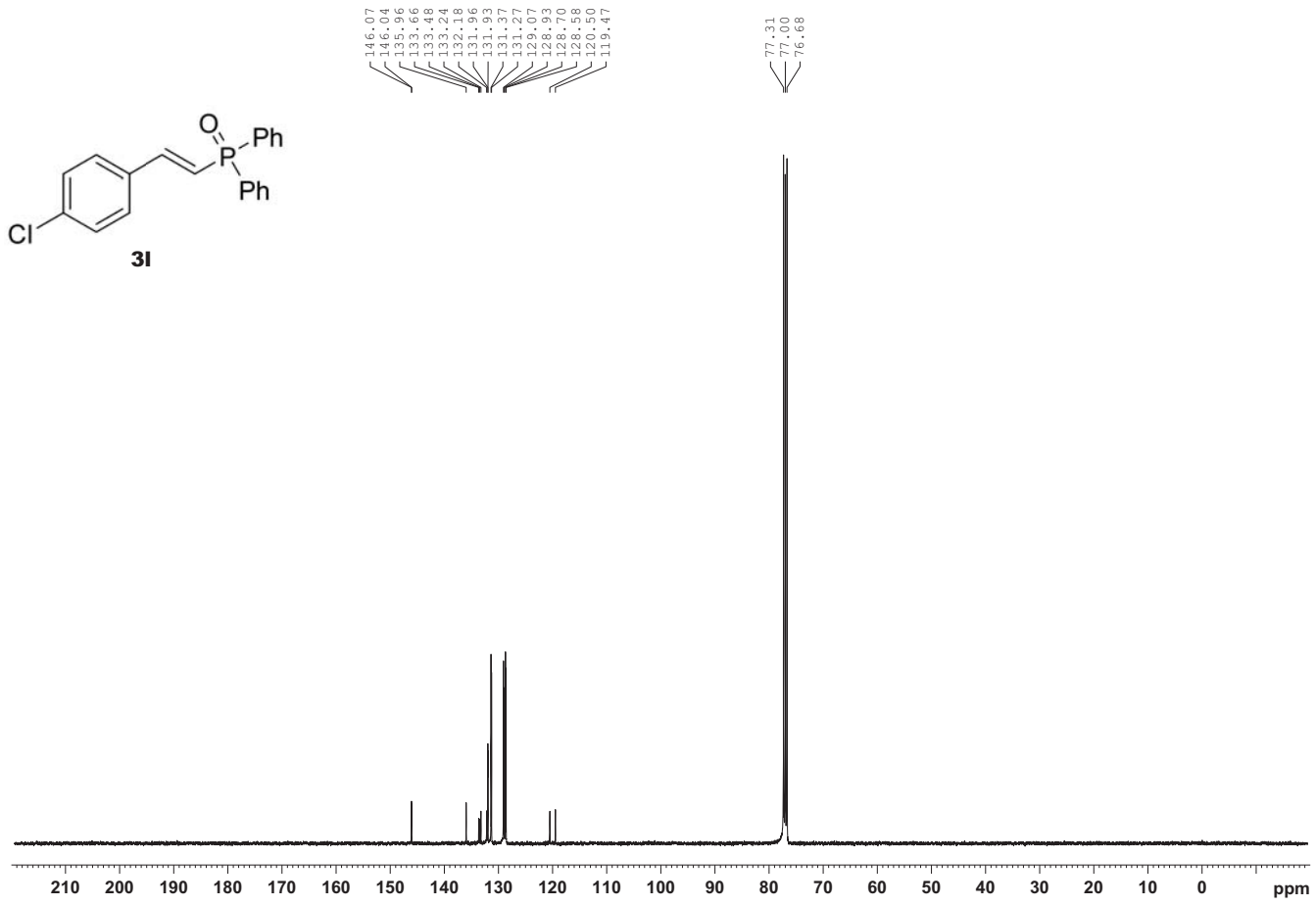
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

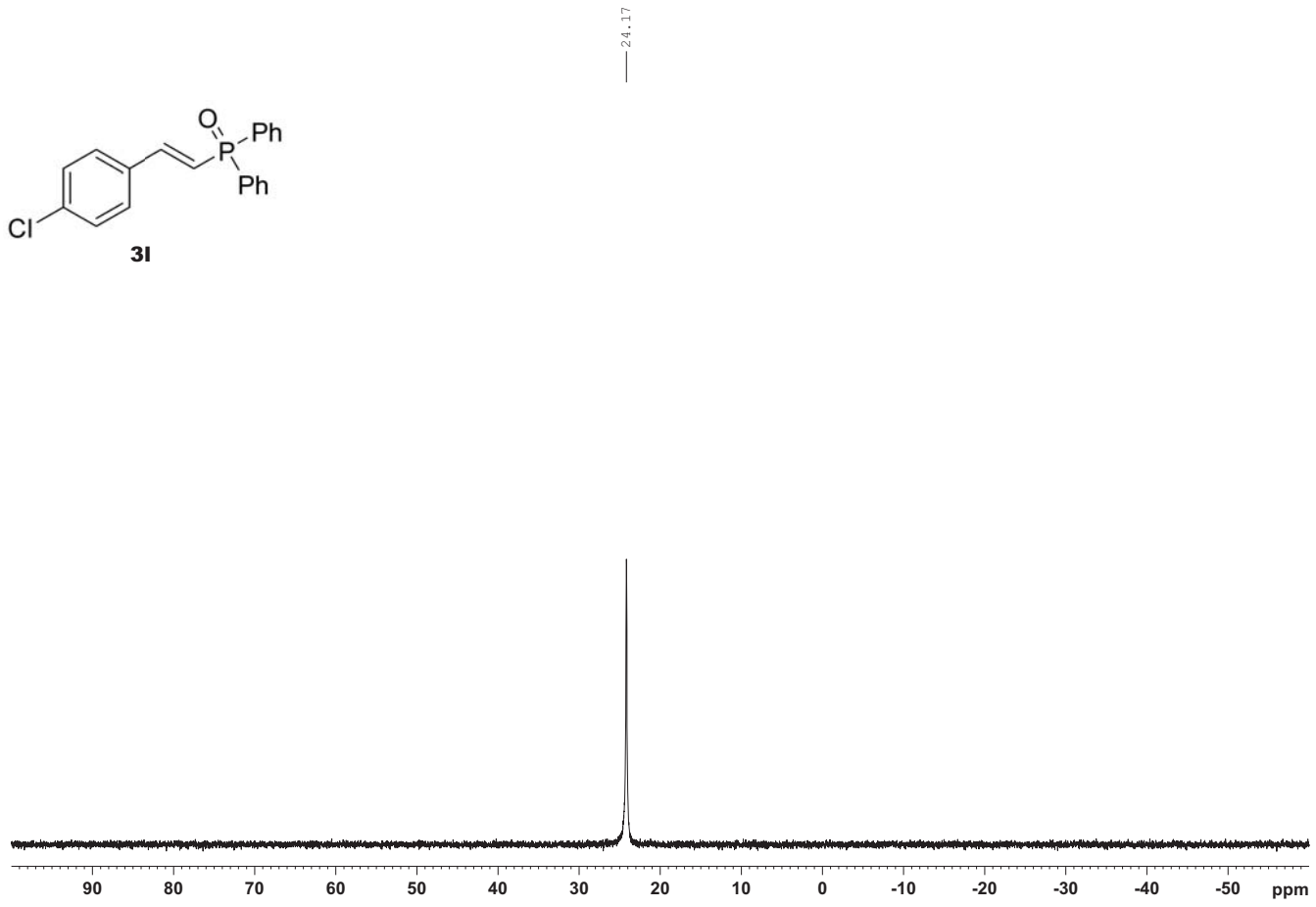

O
Ph
P
Ph
Cl

3l

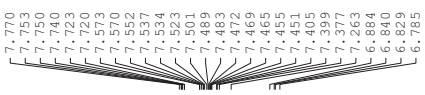
7.773
7.755
7.752
7.742
7.725
7.568
7.549
7.534
7.531
7.506
7.500
7.487
7.481
7.466
7.445
7.421
7.359
7.338
7.266
6.869
6.826
6.814
6.771
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.05
9.07
2.00
1.01


146.07
146.04
135.96
133.66
133.48
133.24
132.18
131.96
131.93
131.37
131.27
129.07
128.93
128.70
128.58
120.50
119.47
77.31
77.00
76.68
O
P
Ph
Ph
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

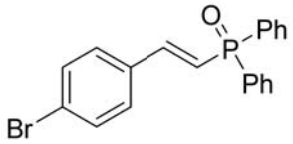
3l


24.17
O
P
Ph
Ph
Cl
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

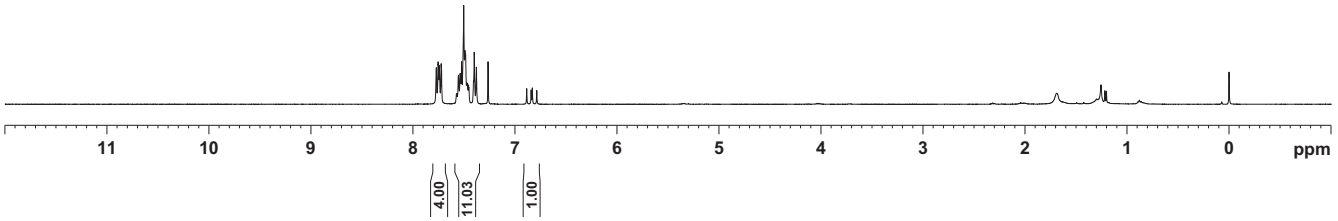
3l

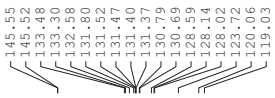

7.770
7.753
7.750
7.740
7.723
7.720
7.573
7.570
7.552
7.537
7.534
7.523
7.501
7.489
7.483
7.472
7.469
7.465
7.455
7.451
7.405
7.399
7.377
7.263
6.884
6.840
6.829
6.785

0.000


O
P
Ph
Ph
Br

3m


11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.00
11.03
1.00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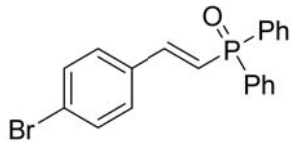
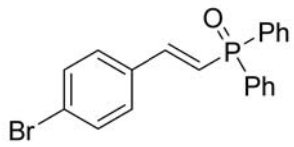
145.52
133.48
133.30
132.58
131.80
131.52
131.47
131.40
131.37
130.79
130.69
128.59
128.14
128.02
123.72
120.06
119.03


77.32
77.00
76.68


O
P
Ph
Ph
Br

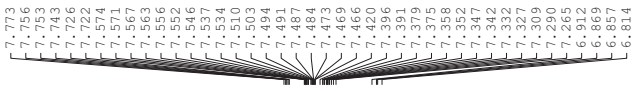
3m

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

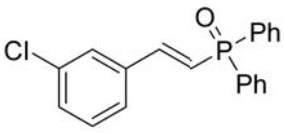

O
Ph
P
Ph
Br

3m

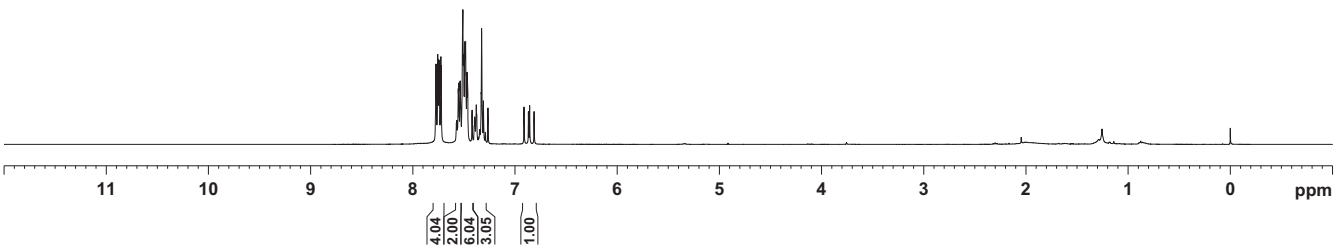
24.19
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

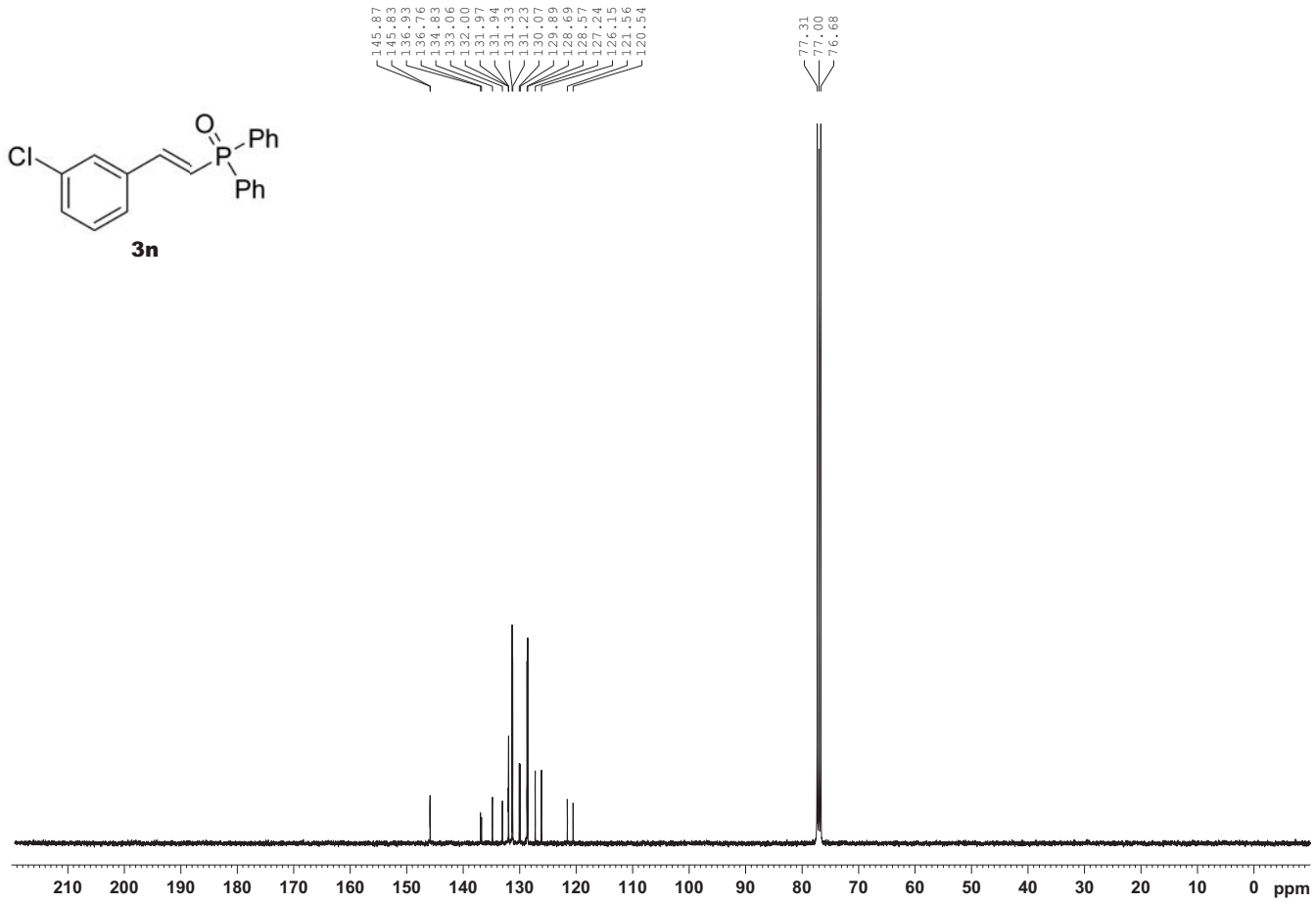

7.773
7.756
7.753
7.743
7.726
7.722
7.574
7.571
7.567
7.563
7.556
7.552
7.546
7.537
7.534
7.510
7.503
7.494
7.491
7.487
7.484
7.473
7.469
7.466
7.420
7.396
7.391
7.379
7.375
7.358
7.352
7.347
7.342
7.332
7.327
7.309
7.290
7.265
6.912
6.869
6.857
6.814

0.000

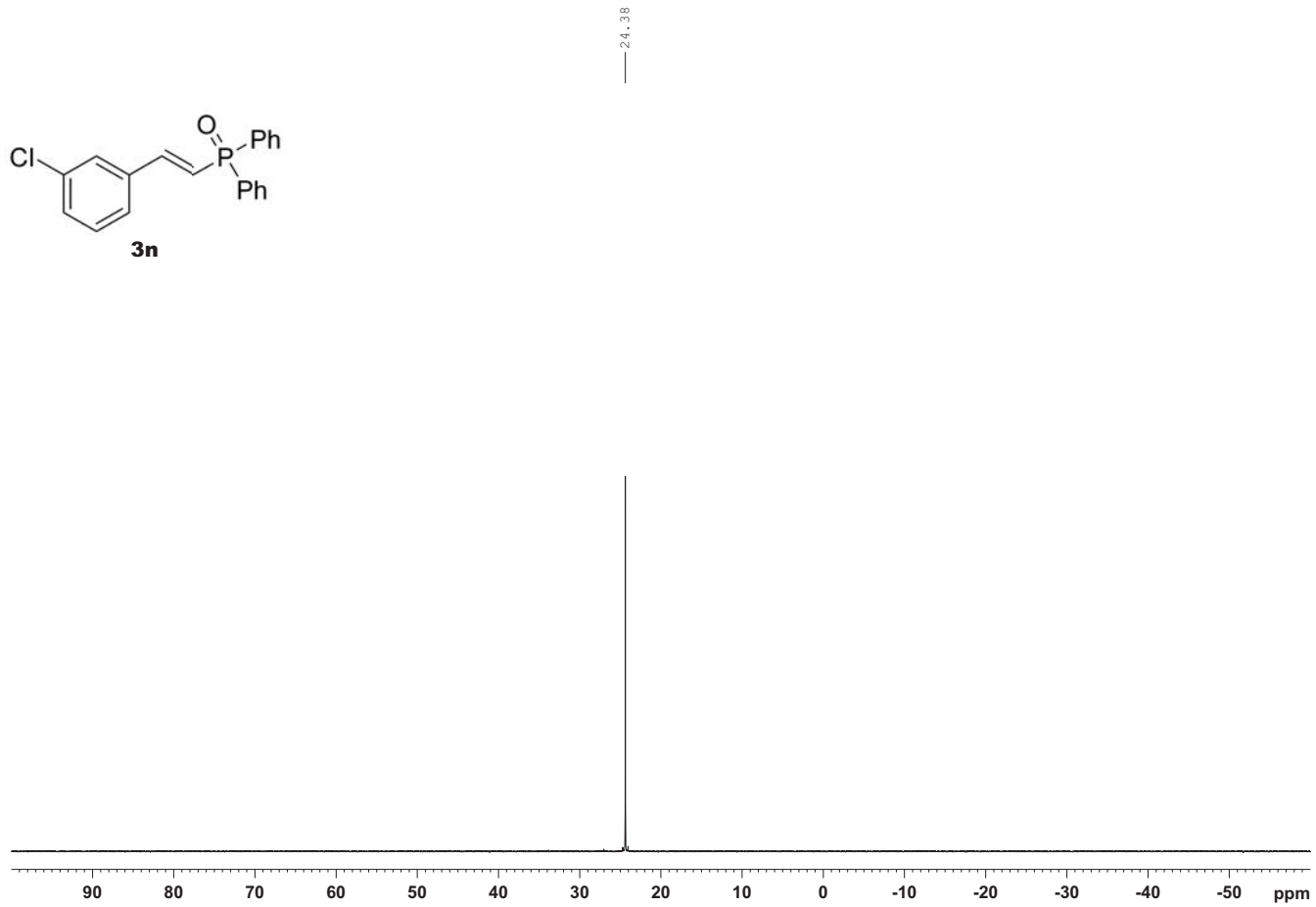

O
Ph
Cl
P
Ph

3n

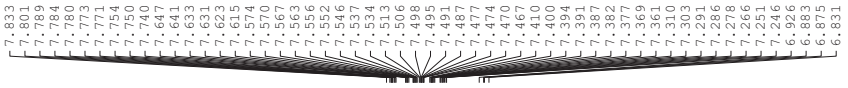

11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.04
2.00
6.04
3.05
1.00

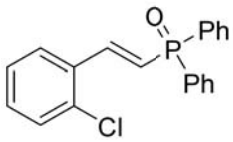

145.87
145.83
136.93
136.76
134.83
133.06
132.00
131.97
131.94
131.33
131.23
130.07
129.89
128.69
128.57
127.24
126.15
121.56
120.54
77.31
77.00
76.68
Cl
O
P
Ph
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3n

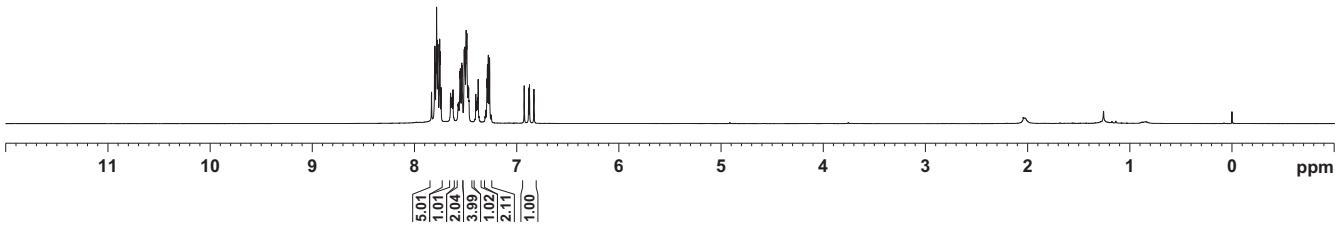

24.38
Cl
O
P
Ph
Ph
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

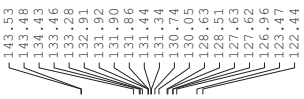
3n


7.833
7.801
7.789
7.784
7.780
7.773
7.771
7.754
7.750
7.740
7.647
7.641
7.633
7.631
7.623
7.615
7.574
7.570
7.567
7.563
7.556
7.552
7.546
7.537
7.534
7.513
7.506
7.498
7.495
7.491
7.487
7.477
7.474
7.470
7.467
7.410
7.400
7.394
7.391
7.387
7.382
7.377
7.369
7.361
7.310
7.303
7.291
7.286
7.278
7.266
7.251
7.246
6.926
6.883
6.875
6.831
0.000

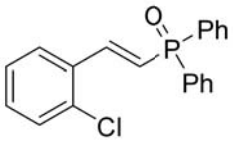

O
Ph
P
Ph
Cl

3o


11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.01
1.01
2.04
3.99
1.02
2.11
1.00

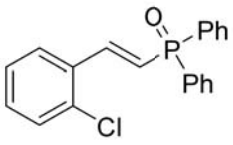

143.53
143.48
134.43
133.46
133.28
132.91
131.92
131.90
131.86
131.44
131.34
130.74
130.05
128.63
128.51
127.63
127.62
126.96
123.47
122.44


77.32
77.00
76.68


O
Ph
P
Ph
Cl

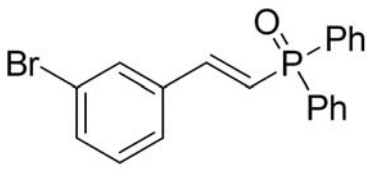
3o

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



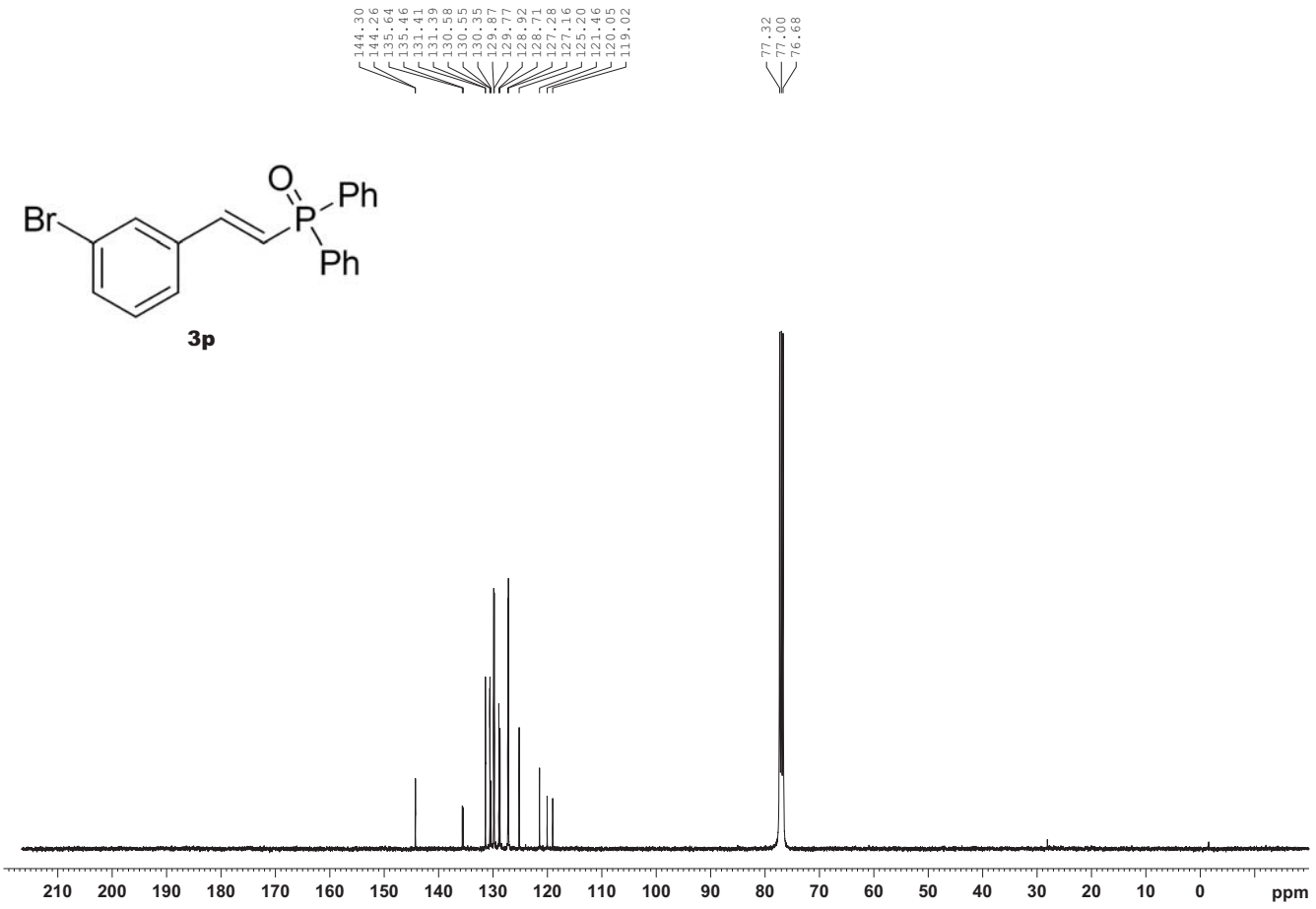
3o

24.66
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

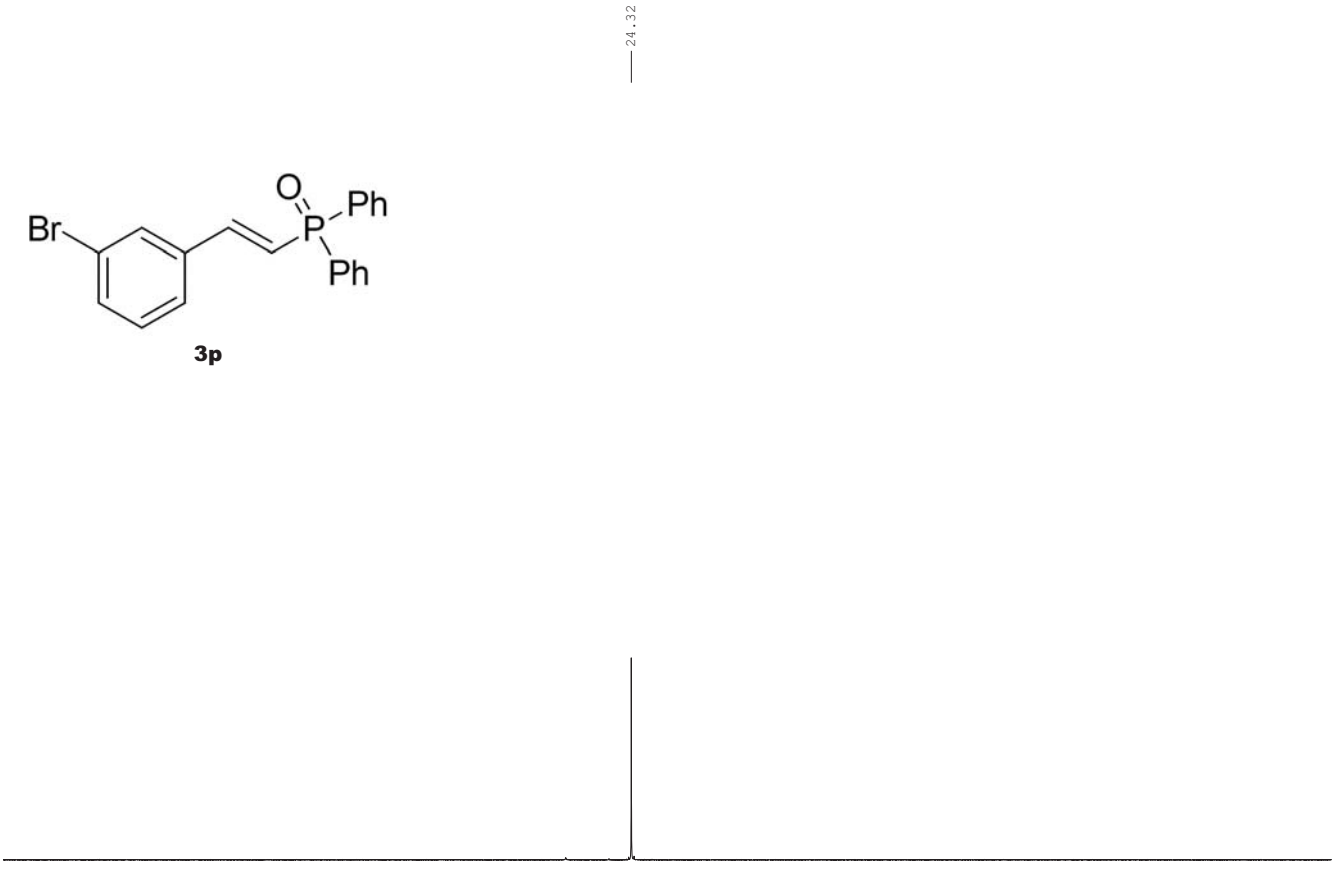


3p

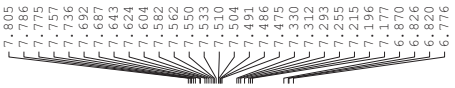
7.768
7.750
7.739
7.722
7.668
7.552
7.537
7.490
7.440
7.420
7.265
7.248
7.230
7.186
6.901
6.850
6.804
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.04
1.08
9.04
1.05
1.00


144.30
144.26
135.64
135.46
131.41
131.39
130.58
130.55
130.35
129.87
129.77
128.92
128.71
127.28
127.16
125.20
121.46
120.05
119.02
77.32
77.00
76.68
O
P
Ph
Ph
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

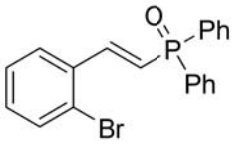
3p


24.32
O
P
Ph
Ph
Br
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

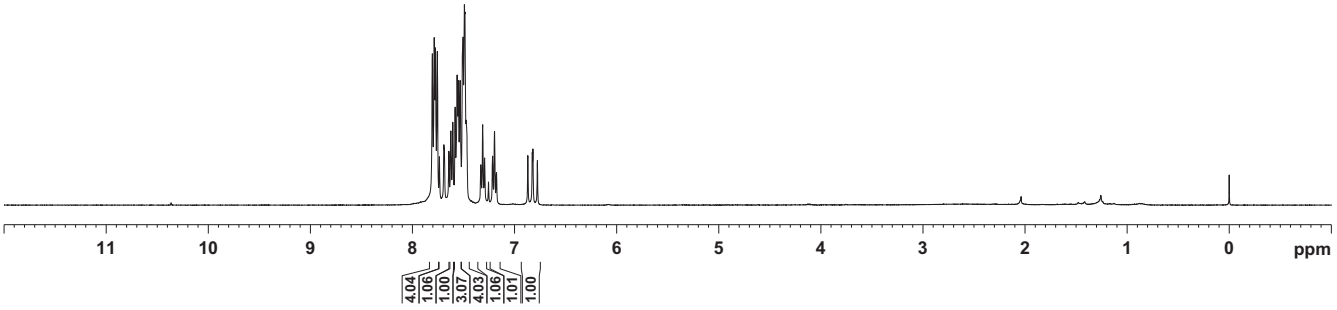
3p

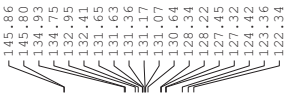

7.805
7.786
7.775
7.757
7.736
7.692
7.687
7.643
7.624
7.604
7.582
7.562
7.550
7.533
7.510
7.504
7.491
7.486
7.475
7.330
7.312
7.293
7.255
7.215
7.196
7.177
6.870
6.826
6.820
6.776


0.000

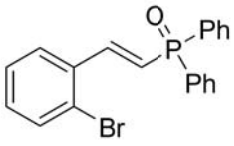

O
Ph
P
Ph
Br

3q

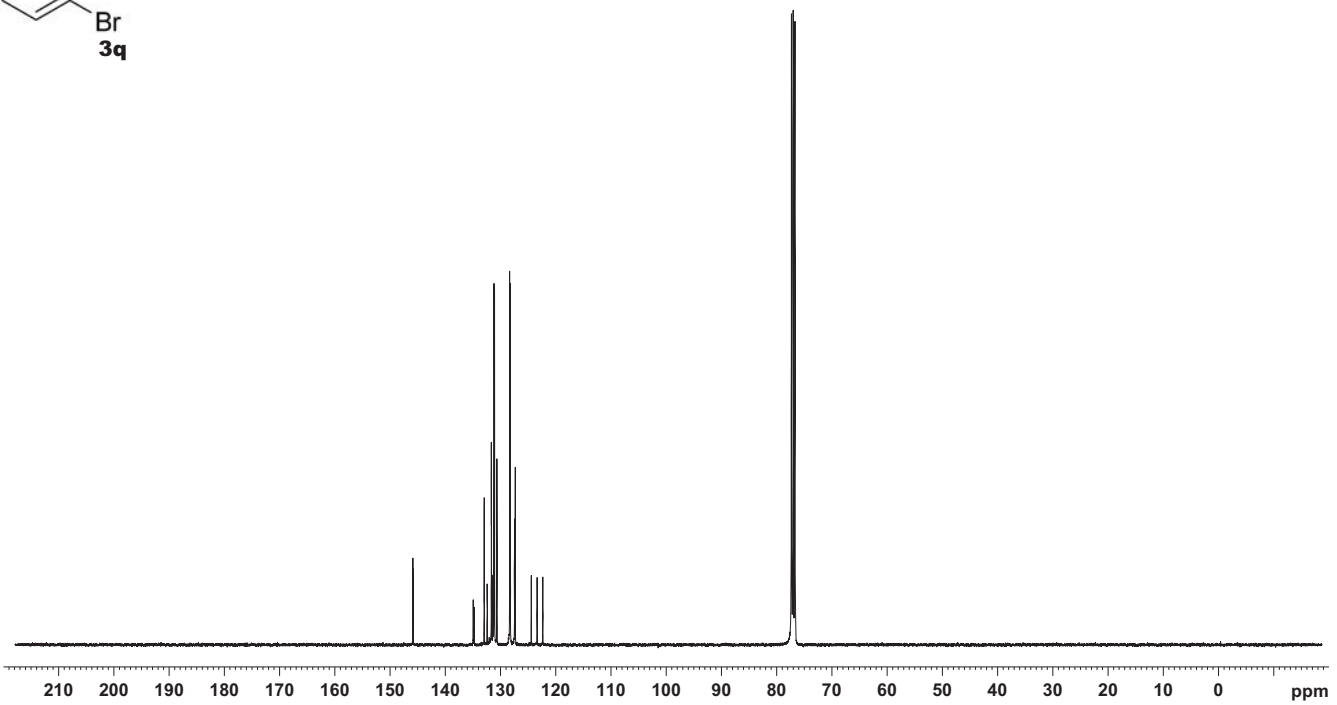

11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.04
1.06
1.00
3.07
4.03
1.06
1.01
1.00

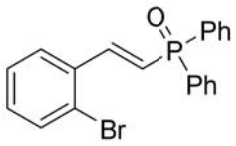

145.86
145.80
134.93
134.75
132.95
132.41
131.65
131.63
131.36
131.17
131.07
130.64
128.34
128.22
127.45
127.32
124.42
123.36
122.34


77.32
77.00
76.68


O
Ph
P
Ph
Br

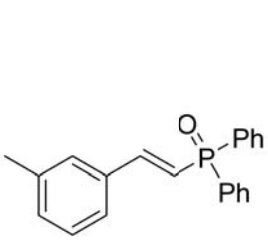
3q


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


O
Ph
P
Ph
Br

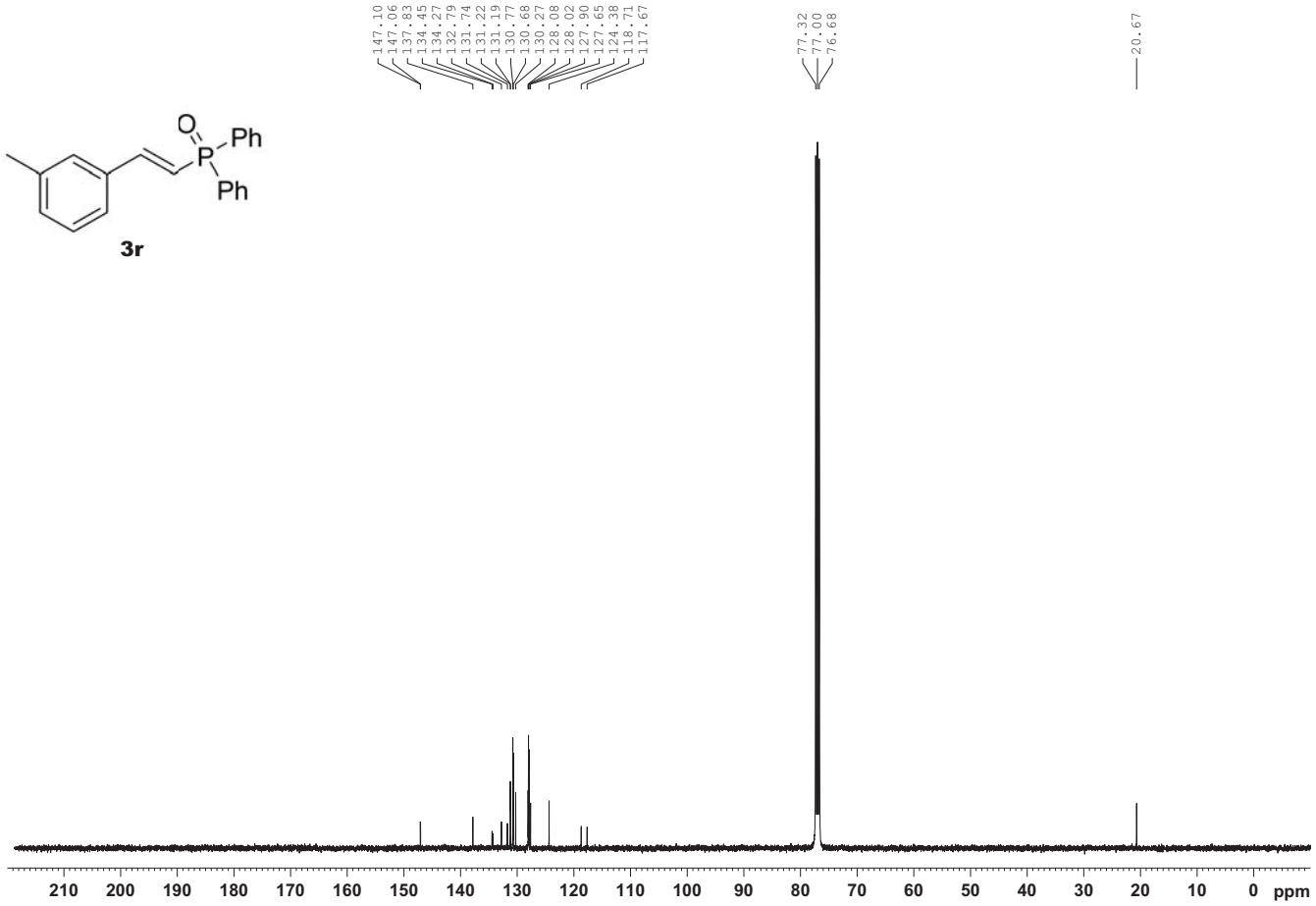
3q

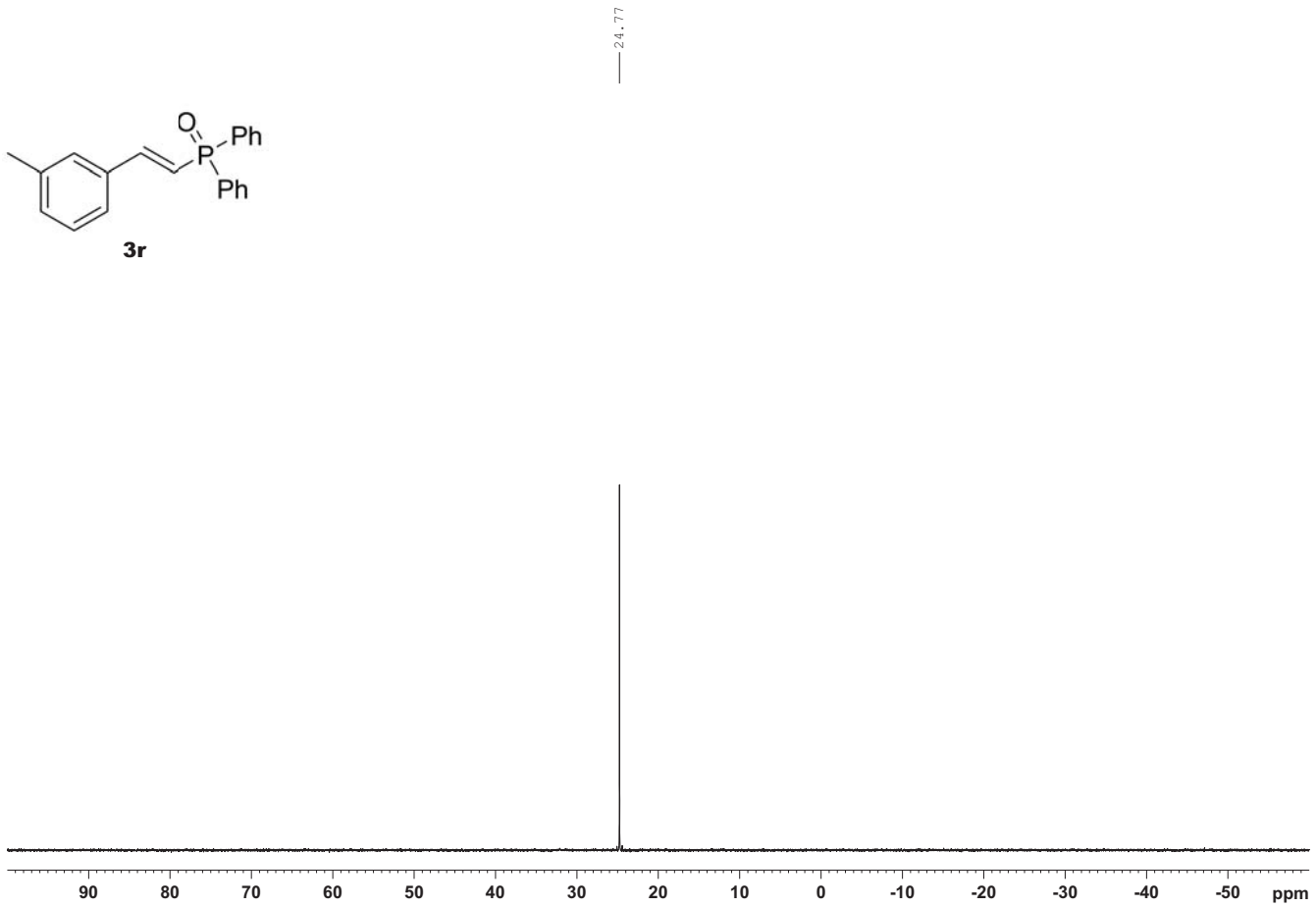
24.99
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

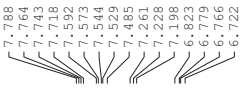

O
Ph
P
Ph

3r

7.780
7.761
7.750
7.732
7.560
7.542
7.525
7.513
7.495
7.482
7.420
7.338
7.320
7.288
7.269
7.250
7.225
7.193
7.175
6.869
6.826
6.813
6.770
2.360
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.08
7.05
2.03
1.08
1.06
1.00
3.07

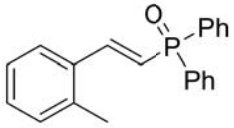

147.10
147.06
137.83
134.45
134.27
132.79
131.74
131.22
131.19
130.77
130.68
130.27
128.08
128.02
127.90
127.65
124.38
118.71
117.67
77.32
77.00
76.68
20.67
O
P
Ph
Ph
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


24.77
O
P
Ph
Ph
3r
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

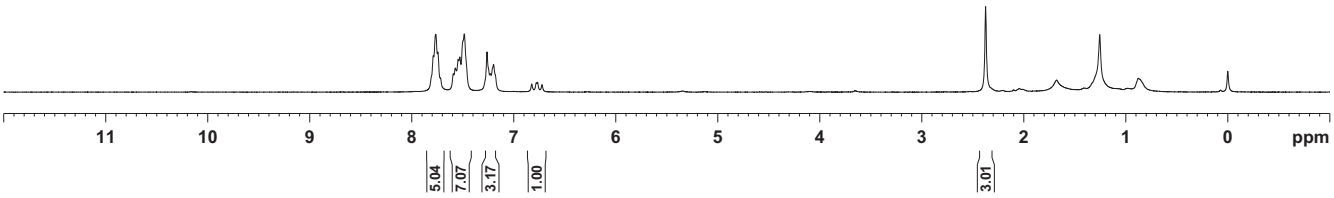

7.788
7.764
7.743
7.718
7.592
7.573
7.544
7.529
7.485
7.261
7.228
7.198
6.823
6.779
6.766
6.722

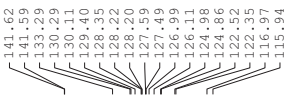
2.374

0.000


O
Ph
P
Ph

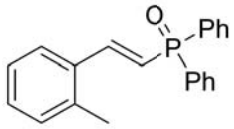
3s


11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.04
7.07
3.17
1.00
3.01

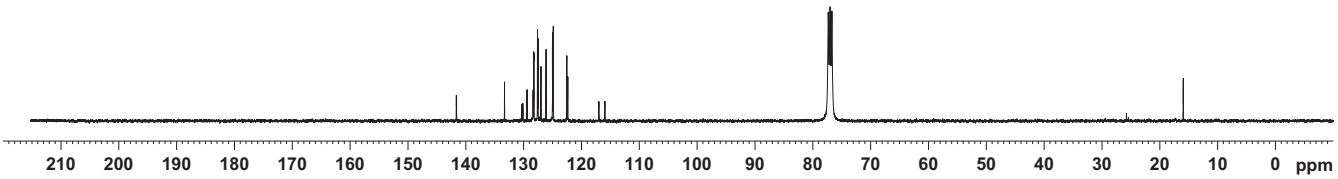

141.62
141.59
133.29
130.29
130.11
129.40
128.35
128.22
128.20
127.59
127.49
126.99
126.11
124.98
124.86
122.52
122.35
116.97
115.94

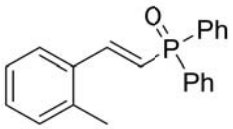

77.32
77.00
76.69

15.94


O
Ph
P
Ph

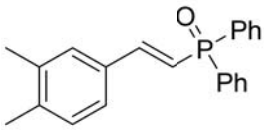
3s


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



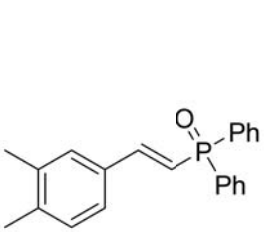
3s

24.80
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm



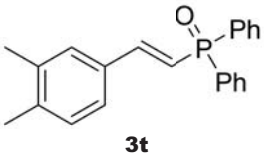
3t

7.779
7.762
7.758
7.749
7.731
7.729
7.540
7.525
7.521
7.507
7.504
7.483
7.478
7.465
7.458
7.448
7.439
7.434
7.389
7.294
7.270
7.264
7.250
7.138
7.119
6.821
6.777
6.765
6.721
2.259
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.00
2.05
5.01
2.09
1.01
1.00
6.00


O
Ph
P
Ph

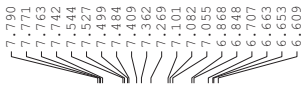
3t

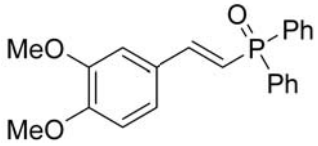
147.63
147.59
139.09
136.96
133.57
132.79
132.61
132.52
131.68
131.65
131.32
131.22
129.99
128.74
128.51
128.39
125.31
118.01
116.96
77.32
77.00
76.69
19.64
19.61
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


O
Ph
P
Ph

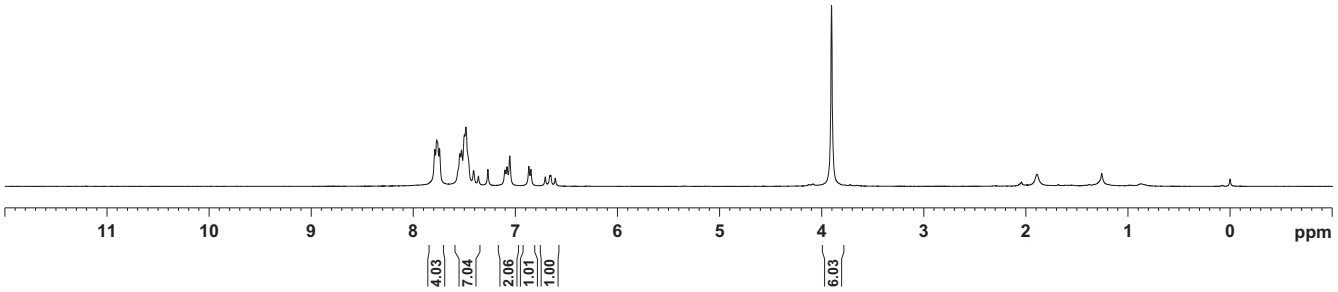
3t

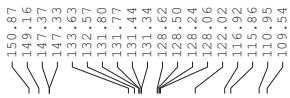
24.84
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

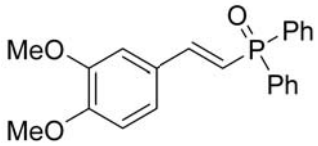

7.790
7.771
7.763
7.742
7.544
7.527
7.499
7.484
7.409
7.362
7.269
7.101
7.082
7.055
6.868
6.848
6.707
6.663
6.653
6.609
3.903
0.000


MeO
MeO
O
P
Ph
Ph

3u

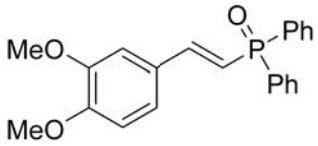

11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.03
7.04
2.06
1.01
1.00
6.03


150.87
149.16
147.37
147.33
133.63
132.57
131.80
131.77
131.44
131.34
128.62
128.50
128.24
128.06
122.02
116.92
115.86
110.95
109.54
77.32
77.00
76.68
55.94
55.90


MeO
MeO
O
P
Ph
Ph

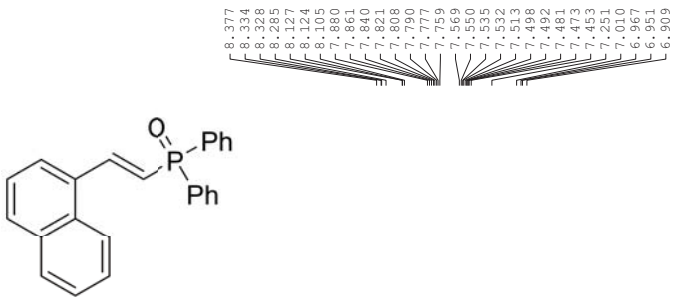
3u

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

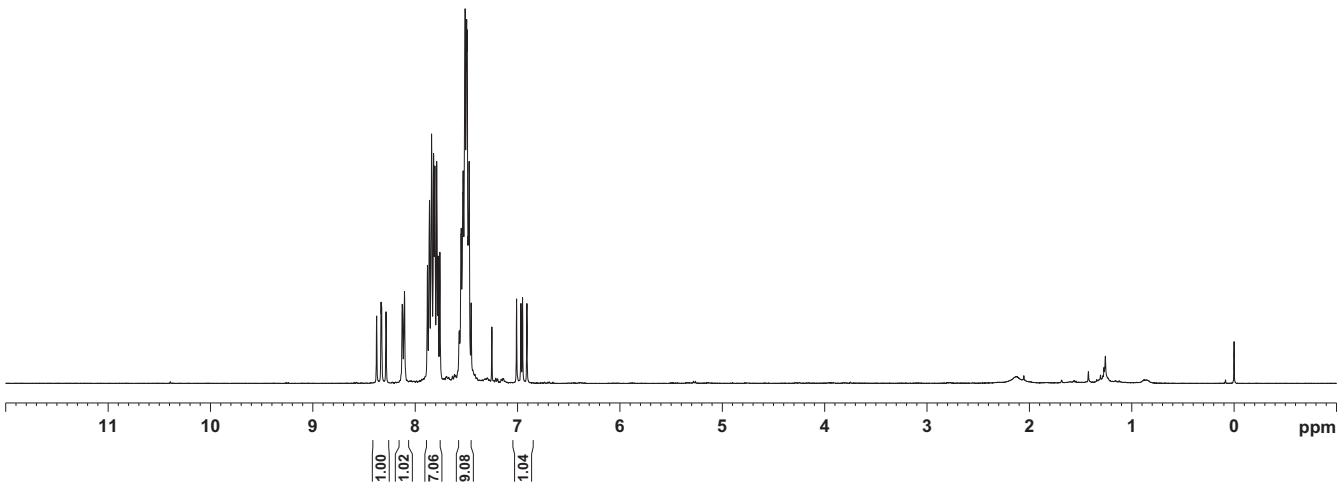

MeO
MeO
O
P
Ph
Ph

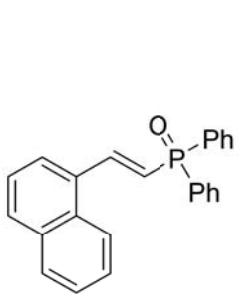
3u

25.00
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm


8.377
8.334
8.328
8.285
8.127
8.124
8.105
7.880
7.861
7.840
7.821
7.808
7.790
7.777
7.759
7.569
7.550
7.535
7.532
7.513
7.498
7.492
7.481
7.473
7.453
7.251
7.010
6.967
6.951
6.909
0.000
O
P
Ph
Ph

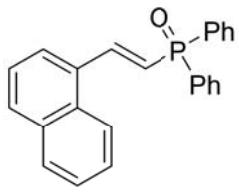
3v


11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
7.06
9.08
1.04


O
P
Ph
Ph

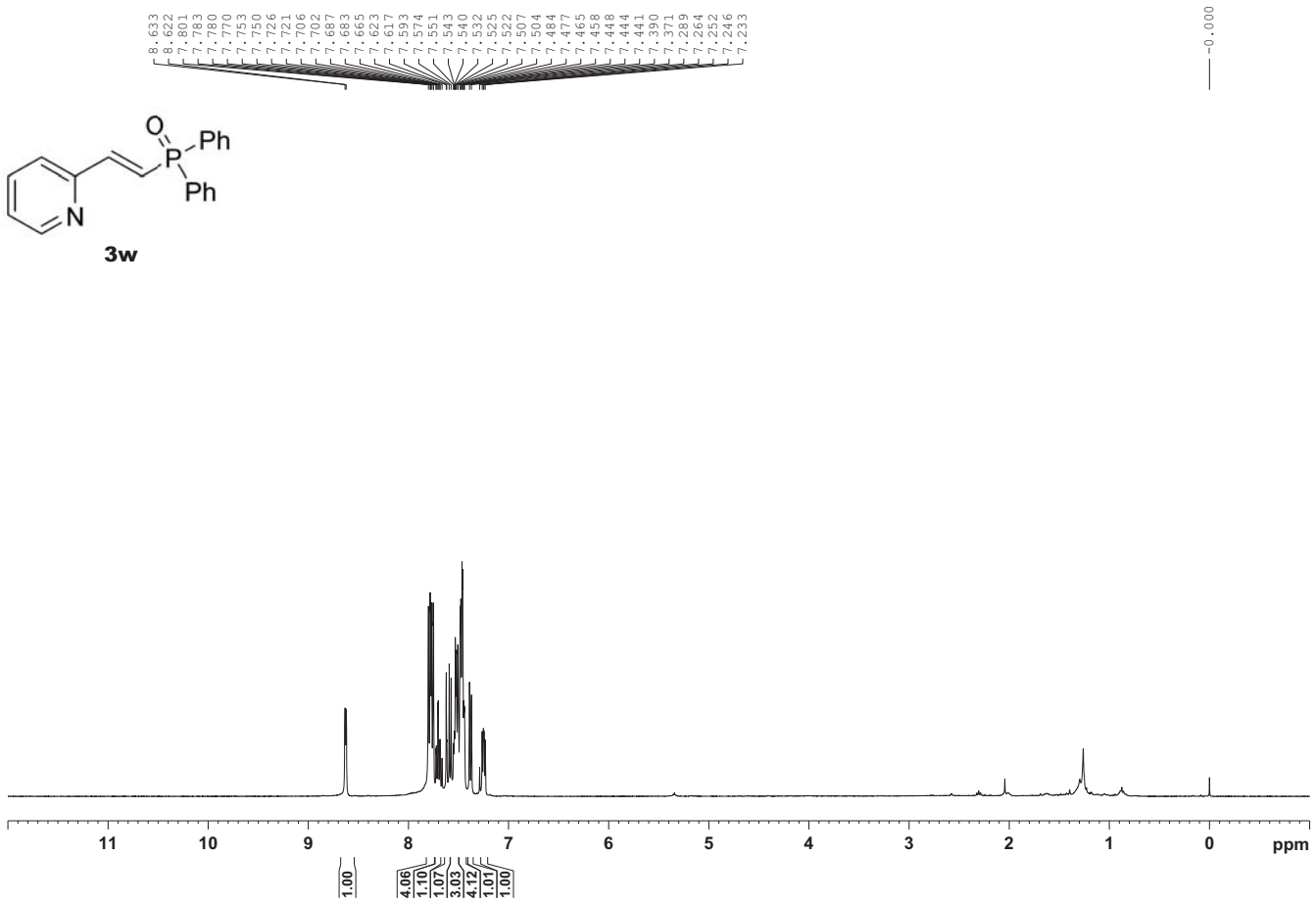
3v

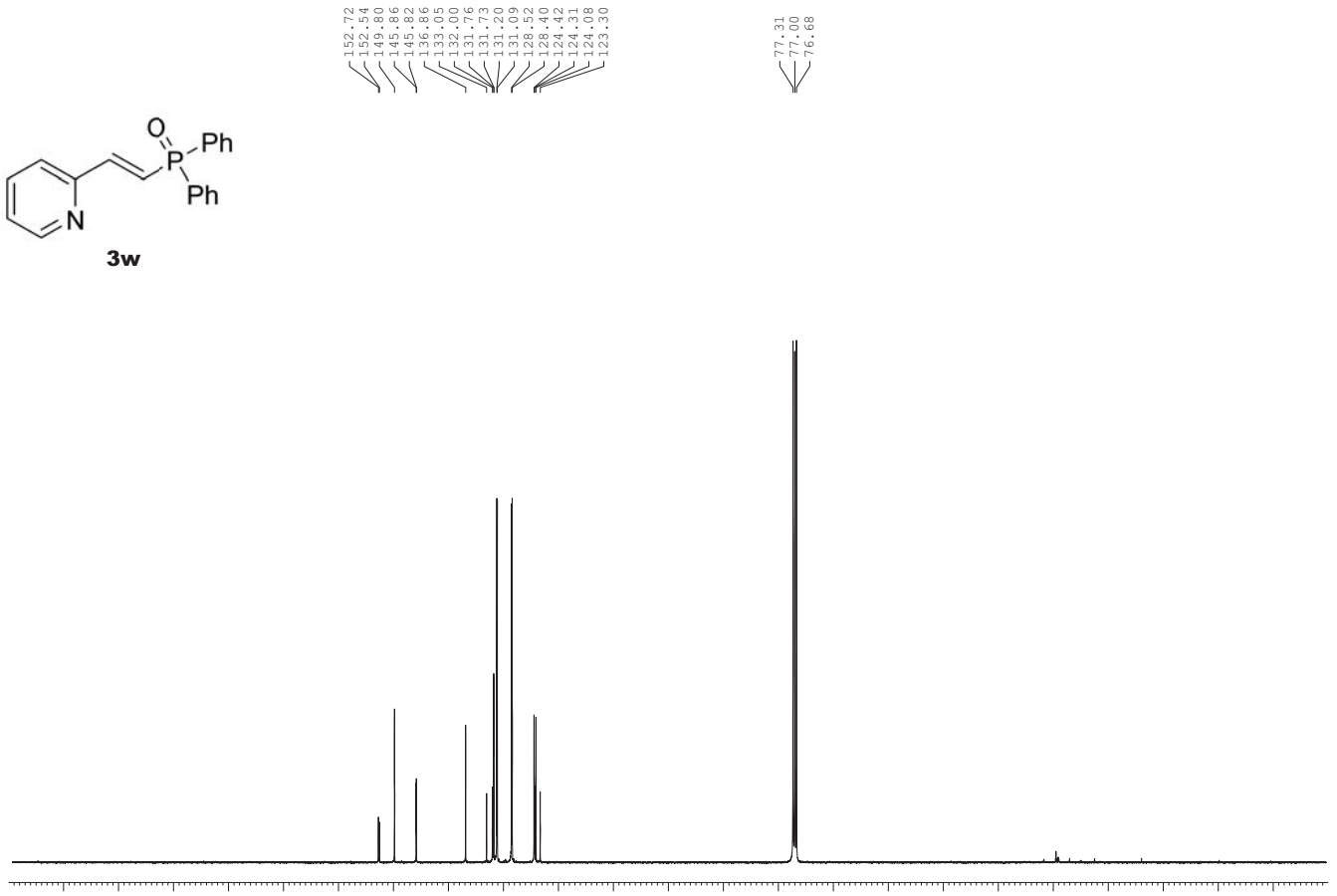
144.77
144.73
133.47
133.35
132.76
132.59
132.30
131.82
131.79
131.33
131.23
130.95
130.18
128.61
128.49
126.71
126.08
125.24
124.64
124.63
123.22
122.95
121.93
77.32
77.00
76.68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

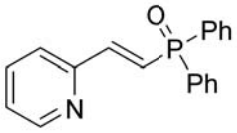

O
P
Ph
Ph

3v

24.19
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

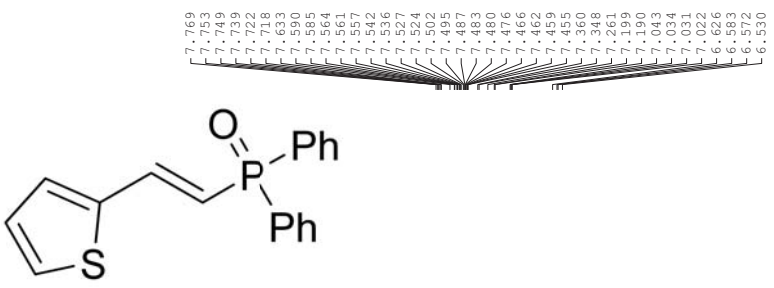

8.633
8.622
7.801
7.783
7.780
7.770
7.753
7.750
7.726
7.721
7.706
7.702
7.687
7.683
7.665
7.623
7.617
7.593
7.574
7.551
7.543
7.540
7.532
7.525
7.522
7.507
7.504
7.484
7.477
7.465
7.458
7.448
7.444
7.441
7.390
7.371
7.289
7.264
7.252
7.246
7.233
-0.000
O
P
Ph
Ph
N
3w
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
4.06
1.10
1.07
3.03
4.12
1.01
1.00


152.72
152.54
149.80
145.86
145.82
136.86
133.05
132.00
131.76
131.73
131.20
131.09
128.52
128.40
124.42
124.31
124.08
123.30
77.31
77.00
76.68
O
P
Ph
Ph
N
3w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



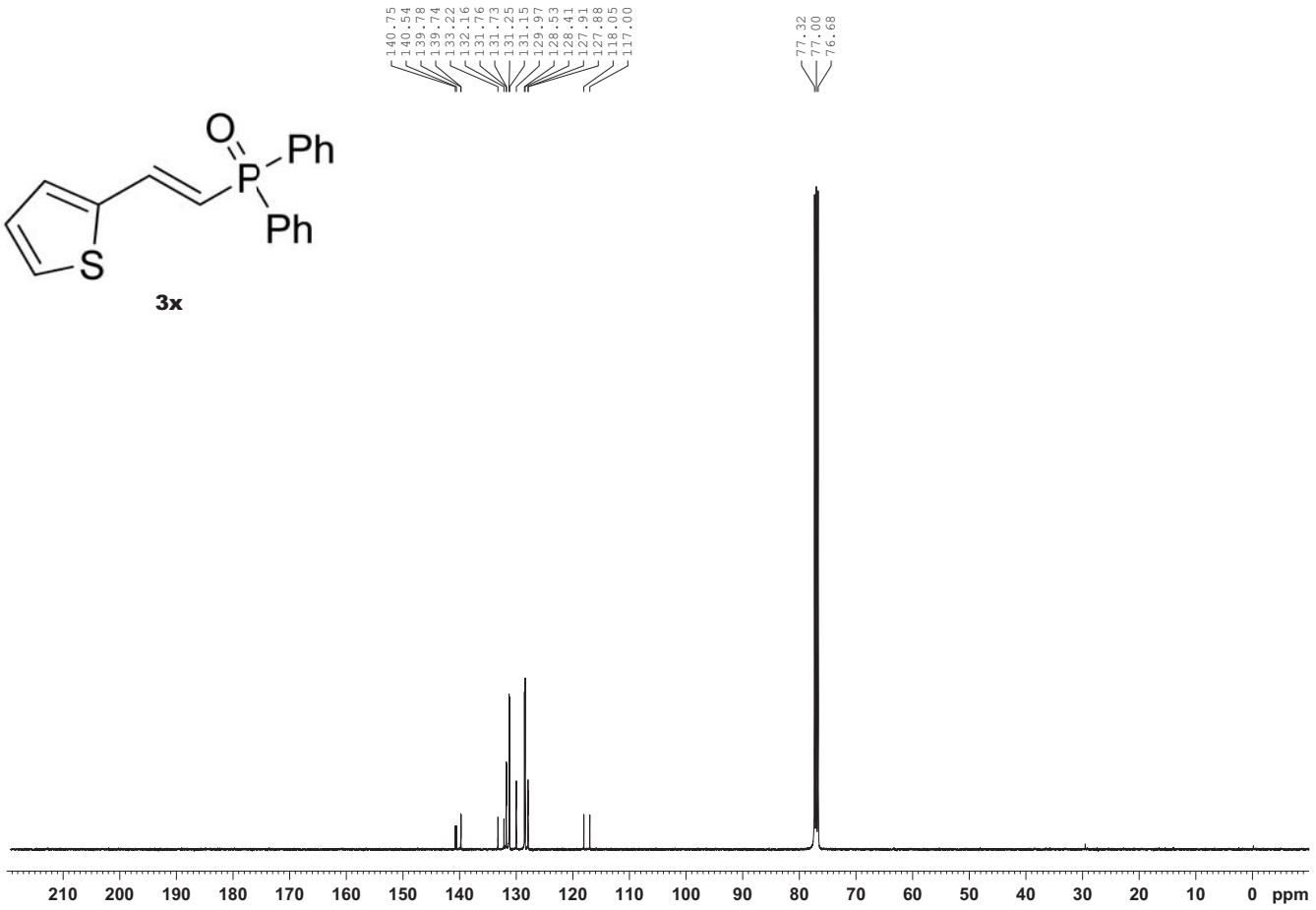
3w

24.10
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

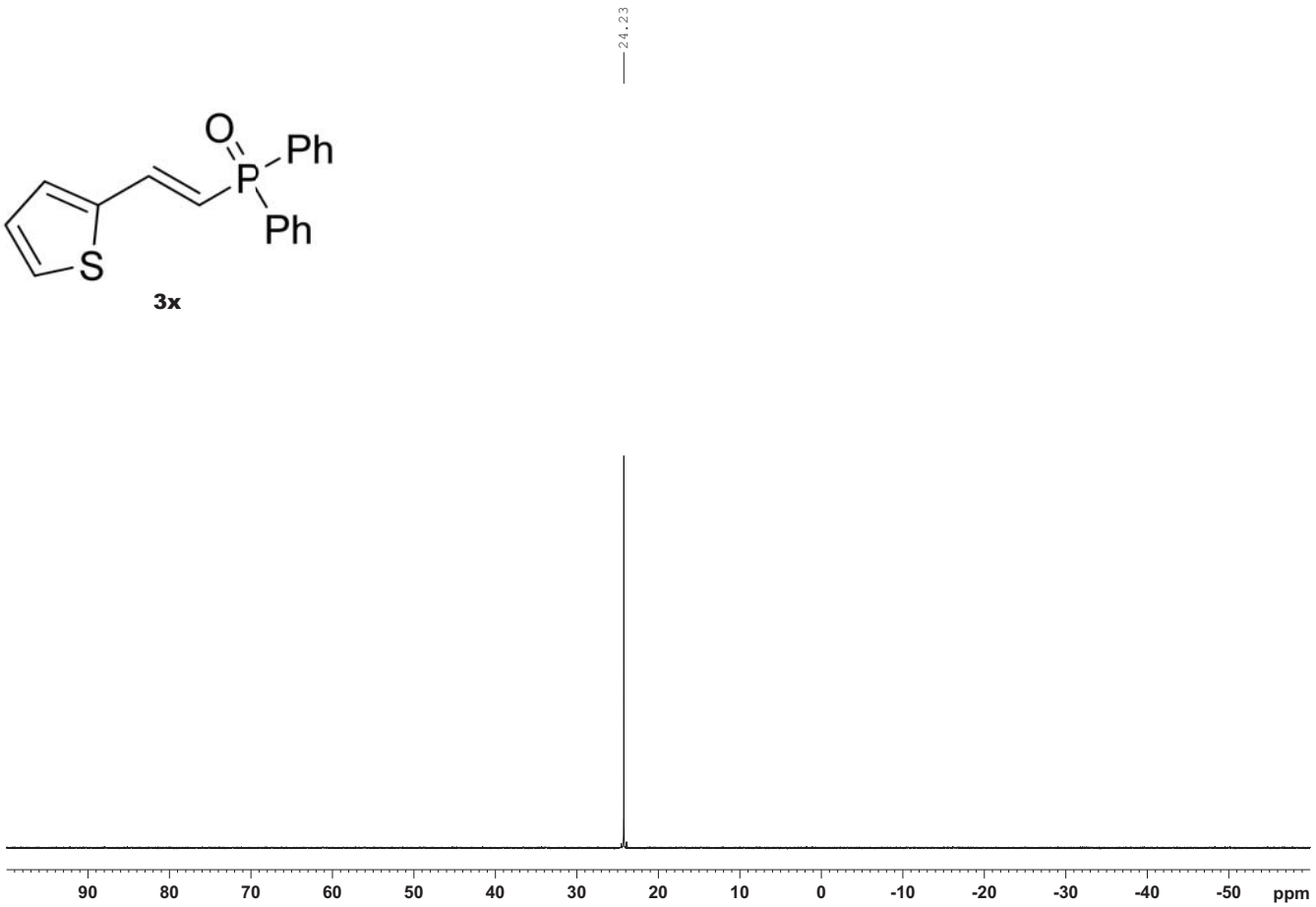


3x

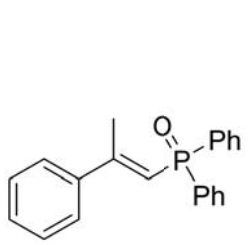
7.769
7.753
7.749
7.739
7.722
7.718
7.633
7.590
7.585
7.564
7.561
7.557
7.542
7.536
7.527
7.524
7.502
7.495
7.487
7.483
7.480
7.476
7.466
7.462
7.459
7.455
7.360
7.348
7.261
7.199
7.190
7.043
7.034
7.031
7.022
6.626
6.583
6.572
6.530
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.07
3.04
4.03
1.03
1.01
1.01
1.00


140.75
140.54
139.78
139.74
133.22
132.16
131.76
131.73
131.25
131.15
129.97
128.53
128.41
127.91
127.88
118.05
117.00
77.32
77.00
76.68
O
P
Ph
Ph
S
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

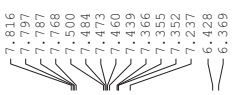
3x


24.23
O
P
Ph
Ph
S
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

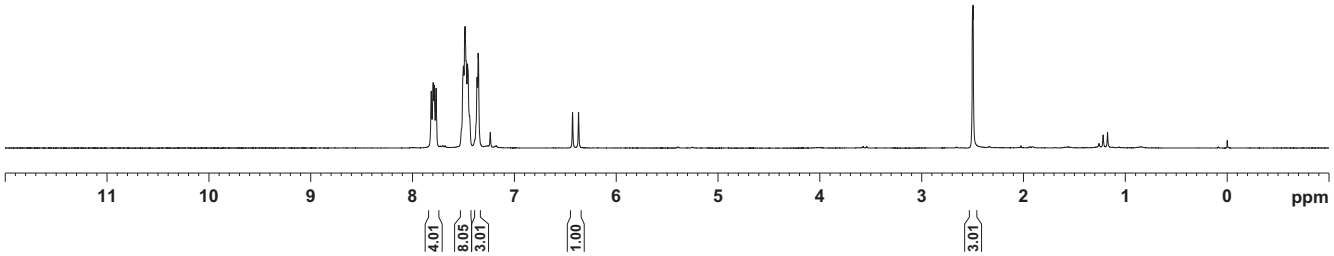
3x

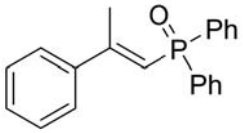

O
P
Ph
Ph

3y

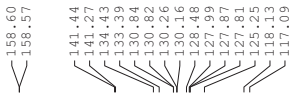

7.816
7.797
7.787
7.768
7.500
7.484
7.473
7.460
7.439
7.366
7.355
7.352
7.237
6.428
6.369


2.502
2.496
-0.000


11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.01
8.05
3.01
1.00
3.01

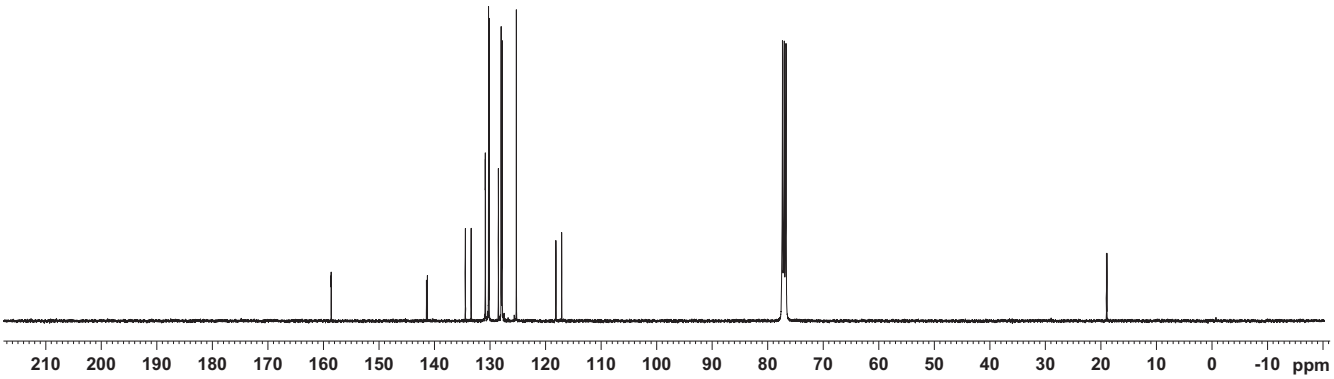

O
P
Ph
Ph

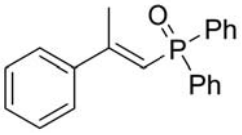
3y


158.60
158.57
141.44
141.27
134.43
133.39
130.84
130.82
130.26
130.16
128.48
127.99
127.87
127.81
125.25
118.13
117.09


77.32
77.00
76.68

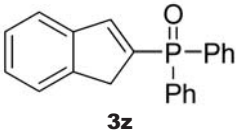

18.99
18.92


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm



3y

21.76
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm



3z

7.779
7.762
7.758
7.749
7.731
7.728
7.584
7.580
7.565
7.562
7.547
7.544
7.512
7.505
7.493
7.486
7.476
7.471
7.469
7.465
7.456
7.449
7.345
7.330
7.324
7.316
7.307
7.288
7.263
3.702
0.000
11
10
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6
5
4
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2
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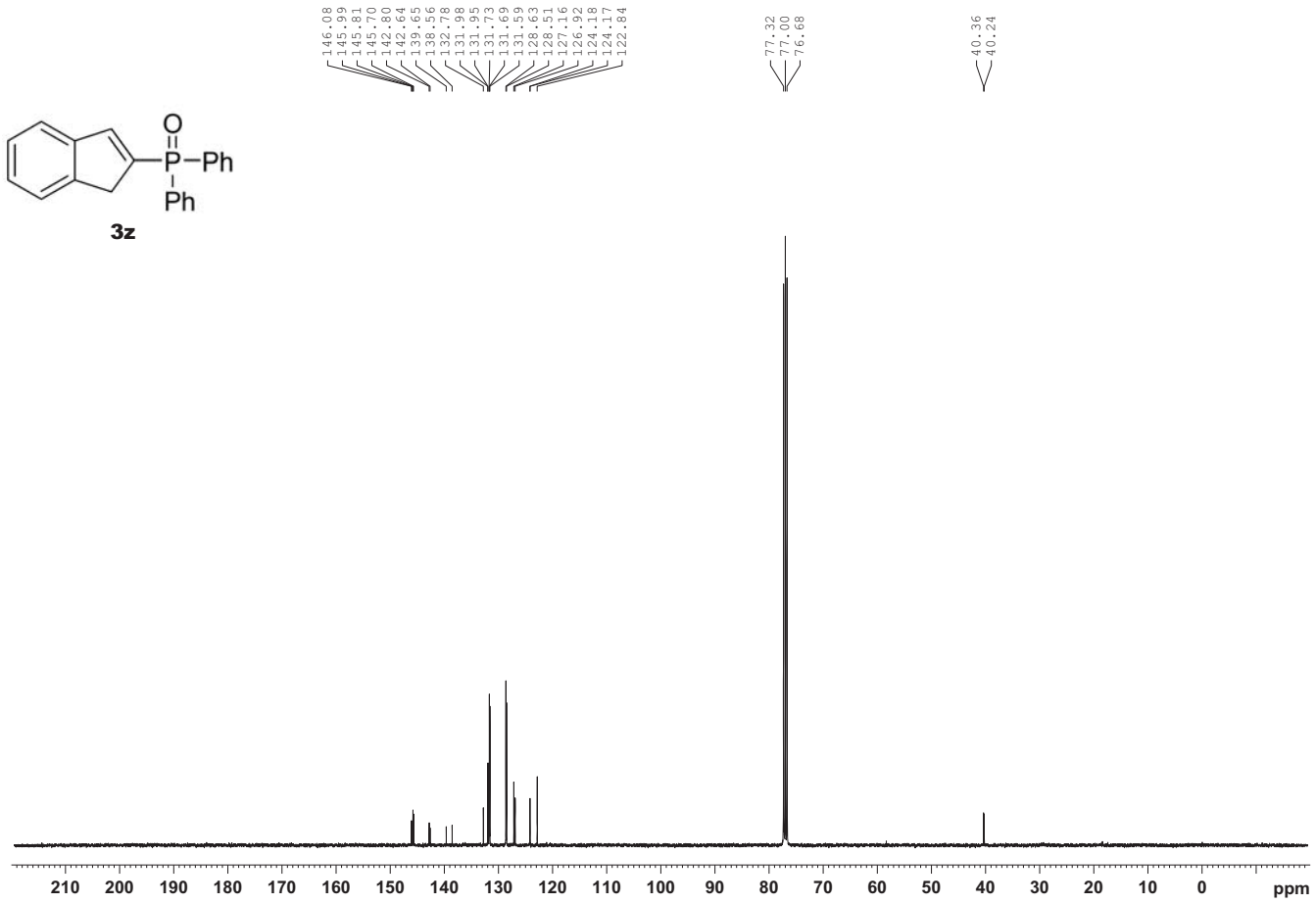
ppm
4.00
2.05
6.02
3.21
2.01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145.99
145.81
145.70
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142.64
139.65
138.56
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131.98
131.95
131.73
131.69
131.59
128.63
128.51
127.16
126.92
124.18
124.17
122.84
77.32
77.00
76.68
40.36
40.24
O
P
Ph
Ph
3z
210
200
190
180
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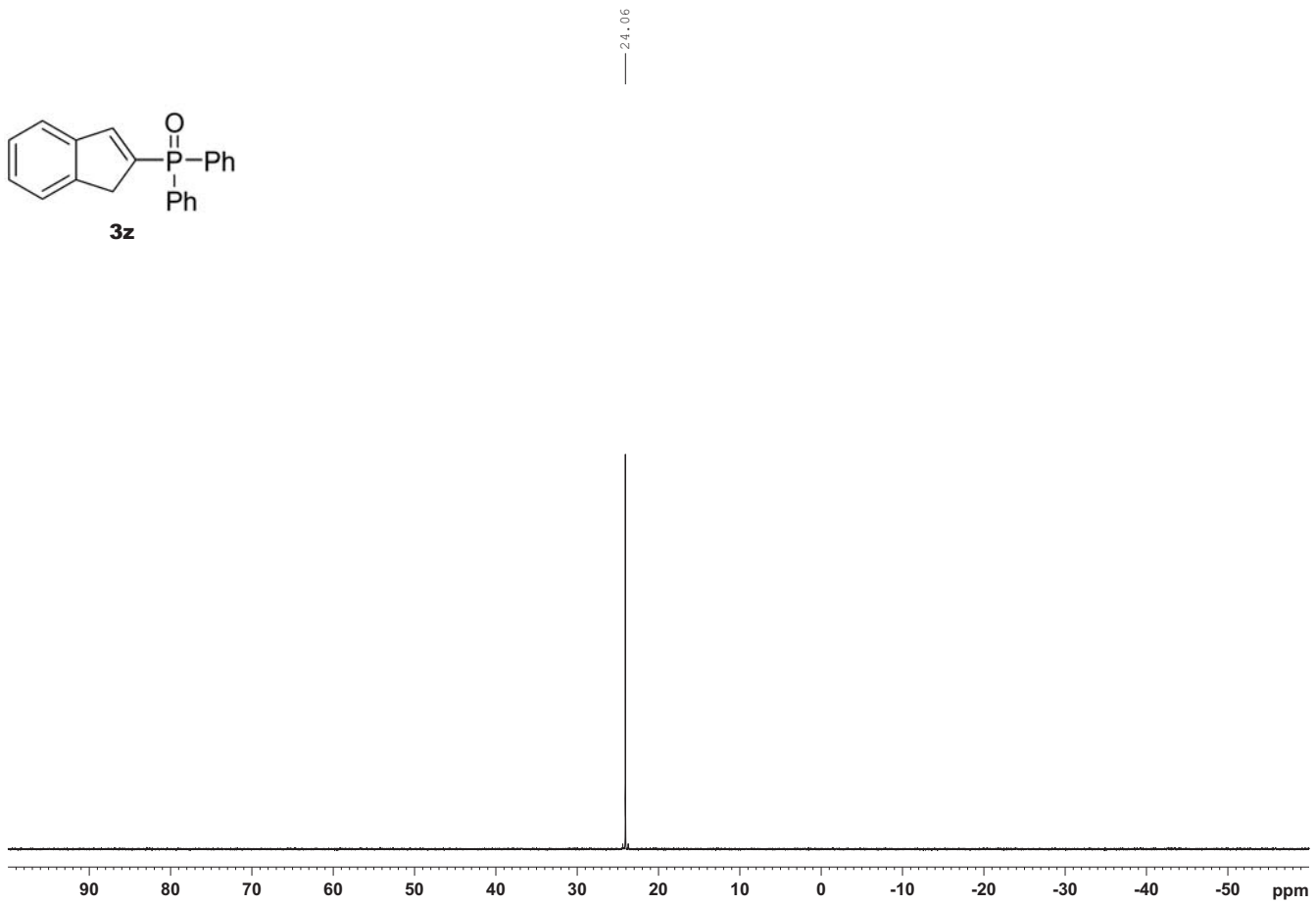
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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P
Ph
Ph
3z
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40
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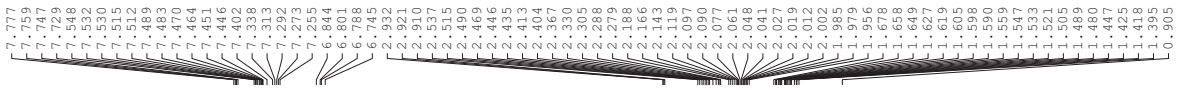
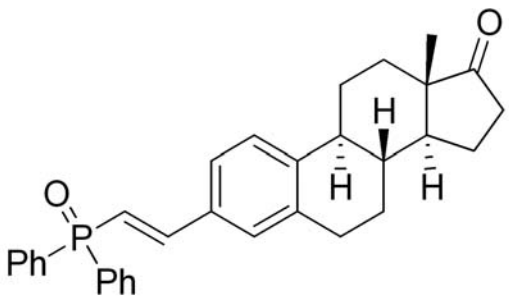
-40
-50
ppm





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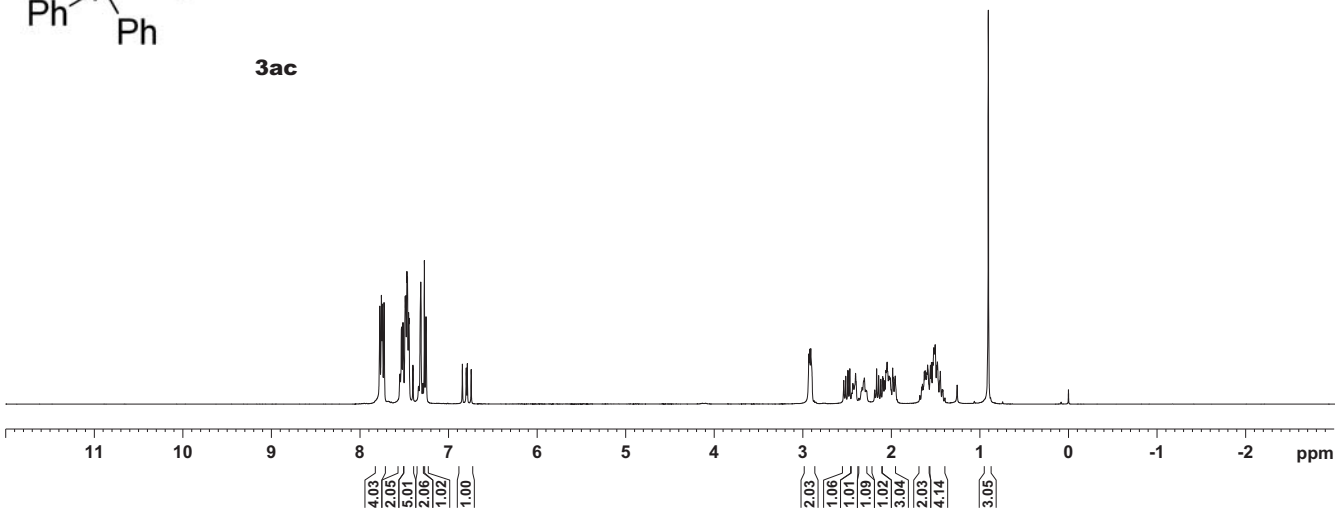
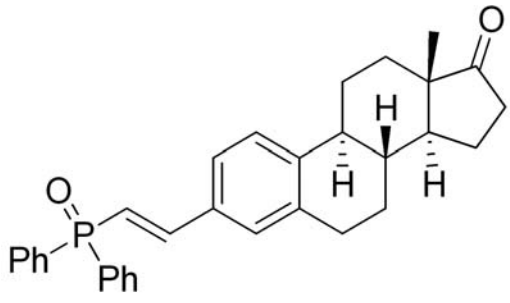
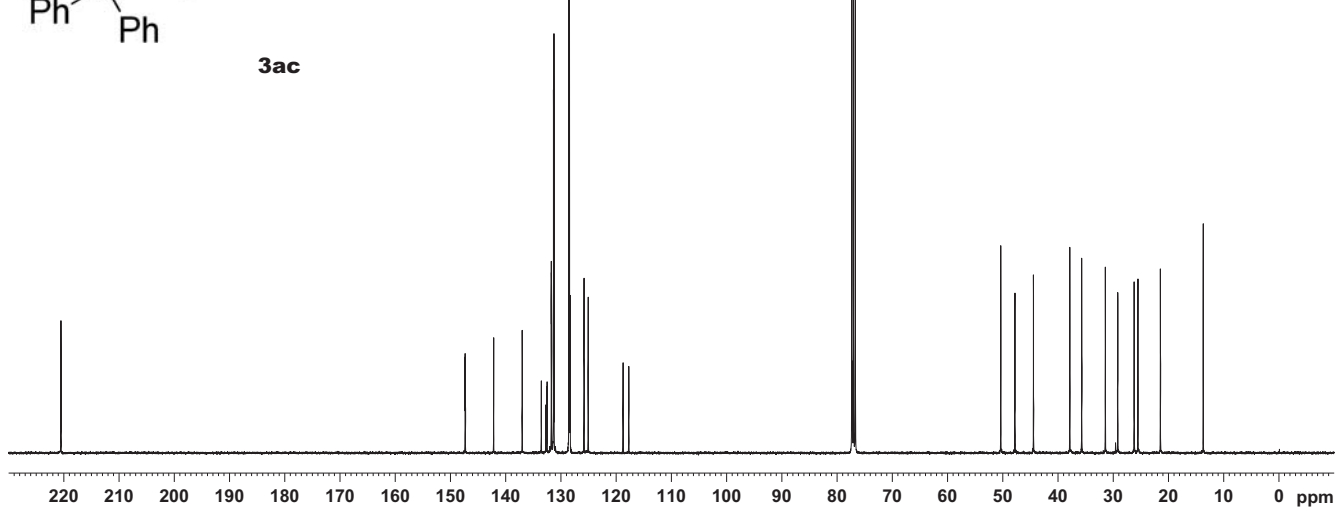
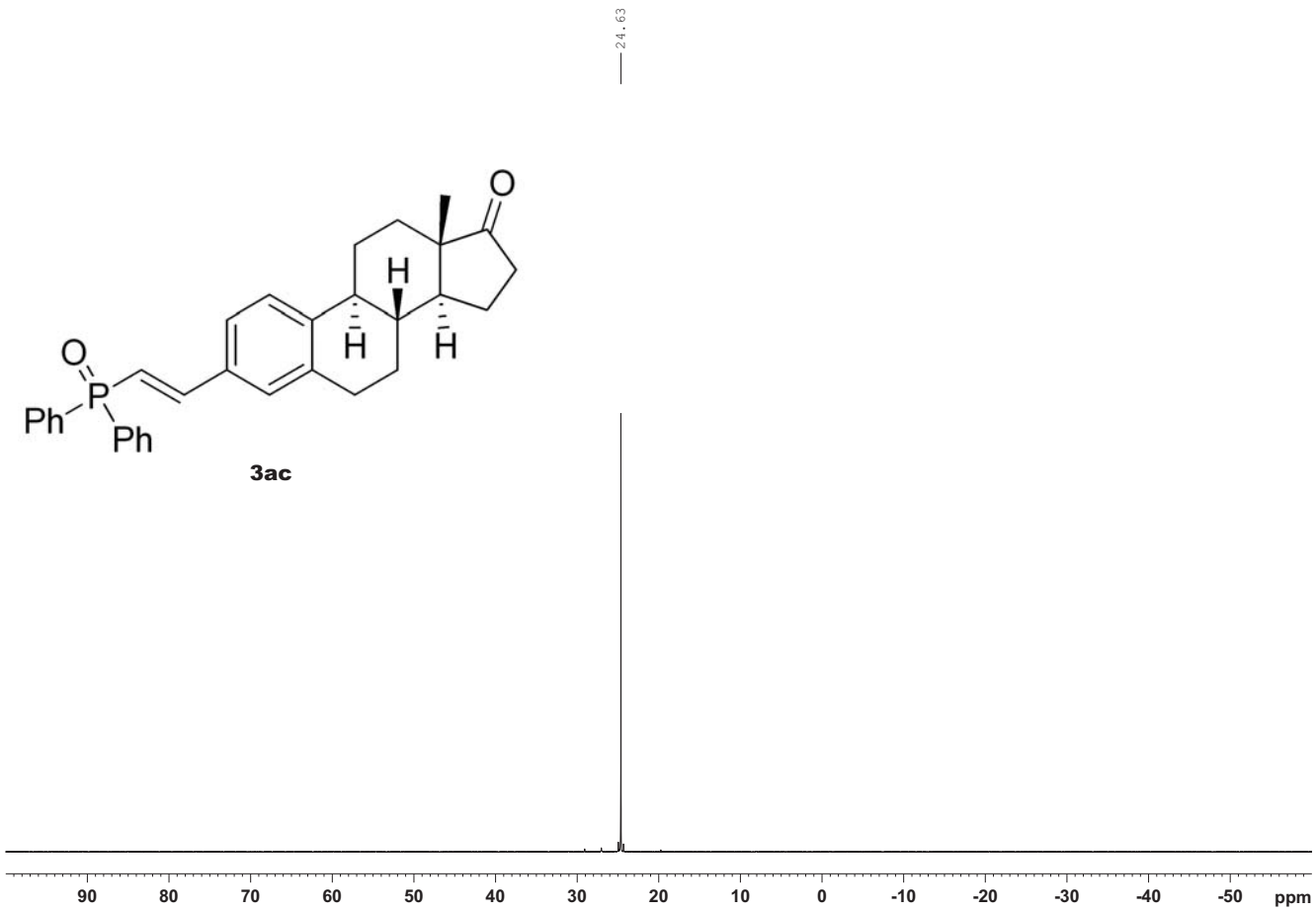




3ac

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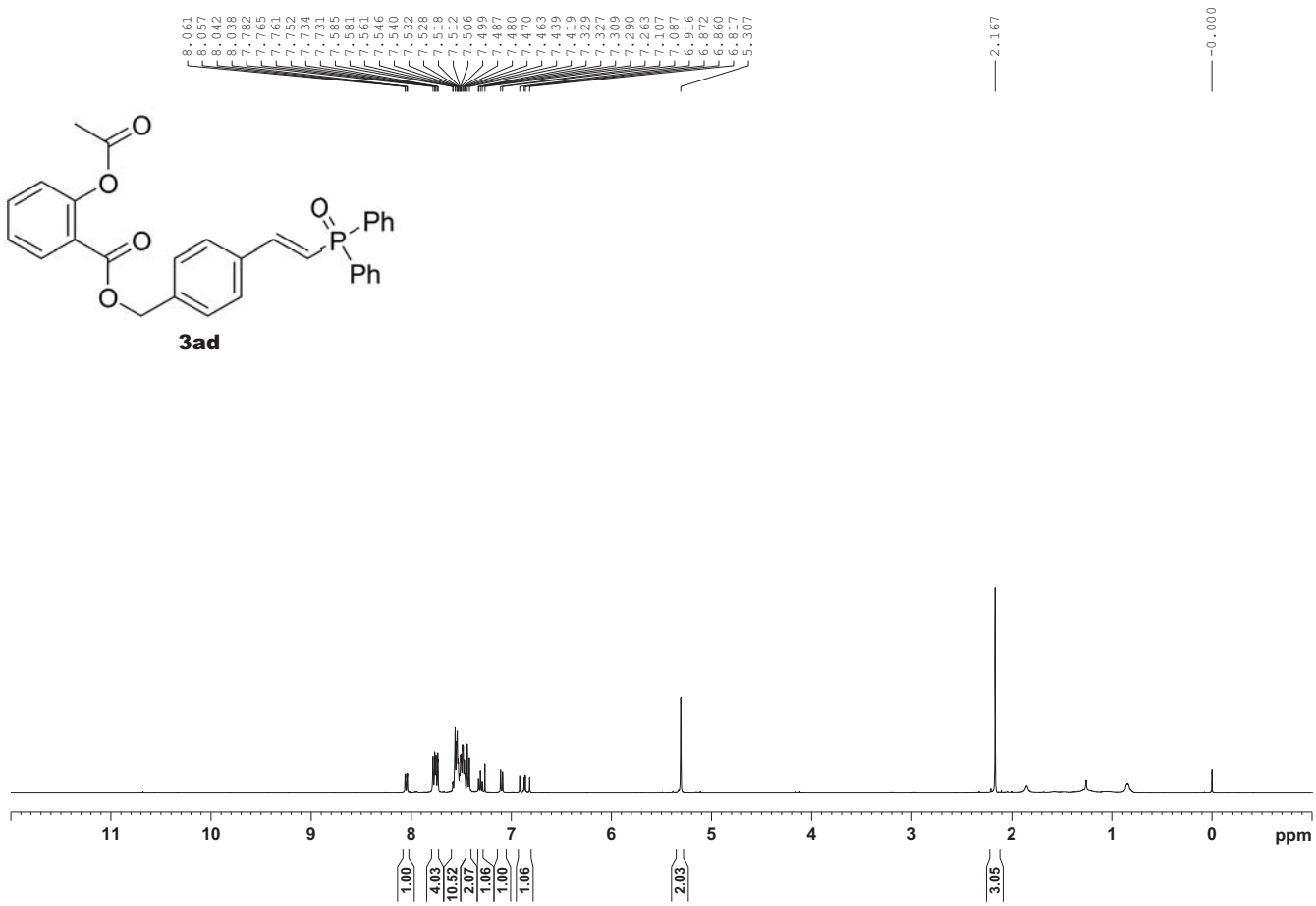
O
H
H
H
O
P
Ph
Ph
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

3ac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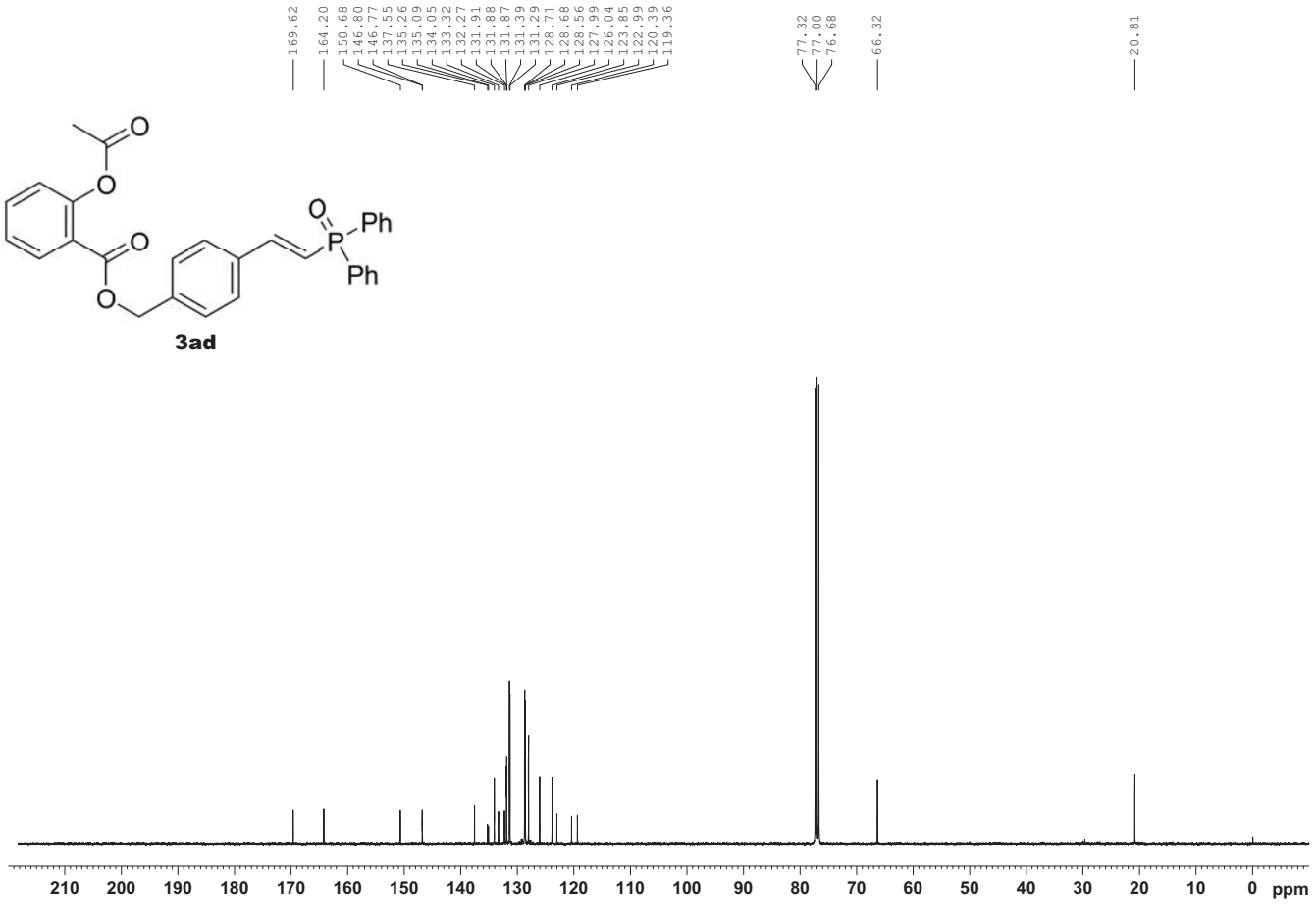
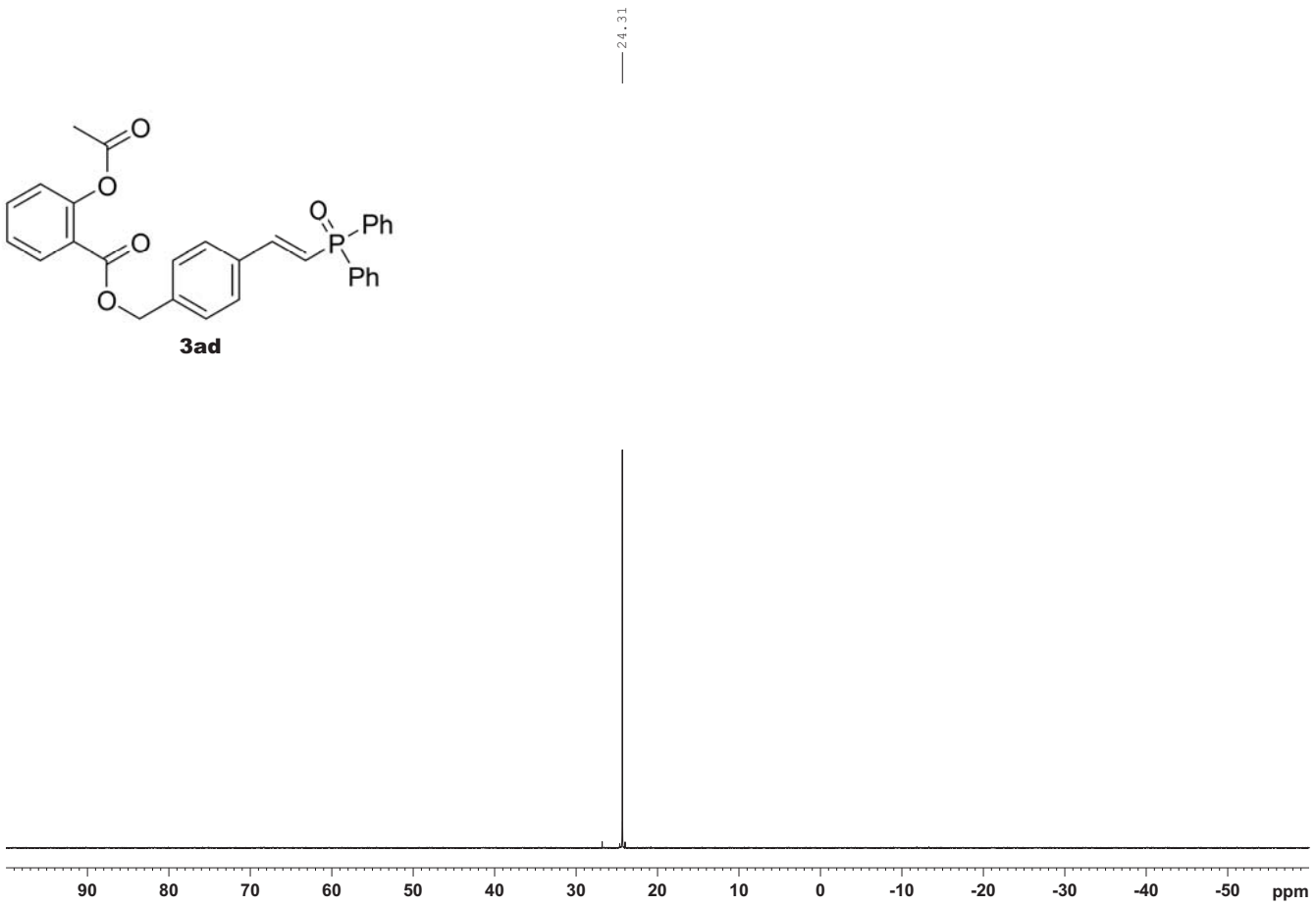
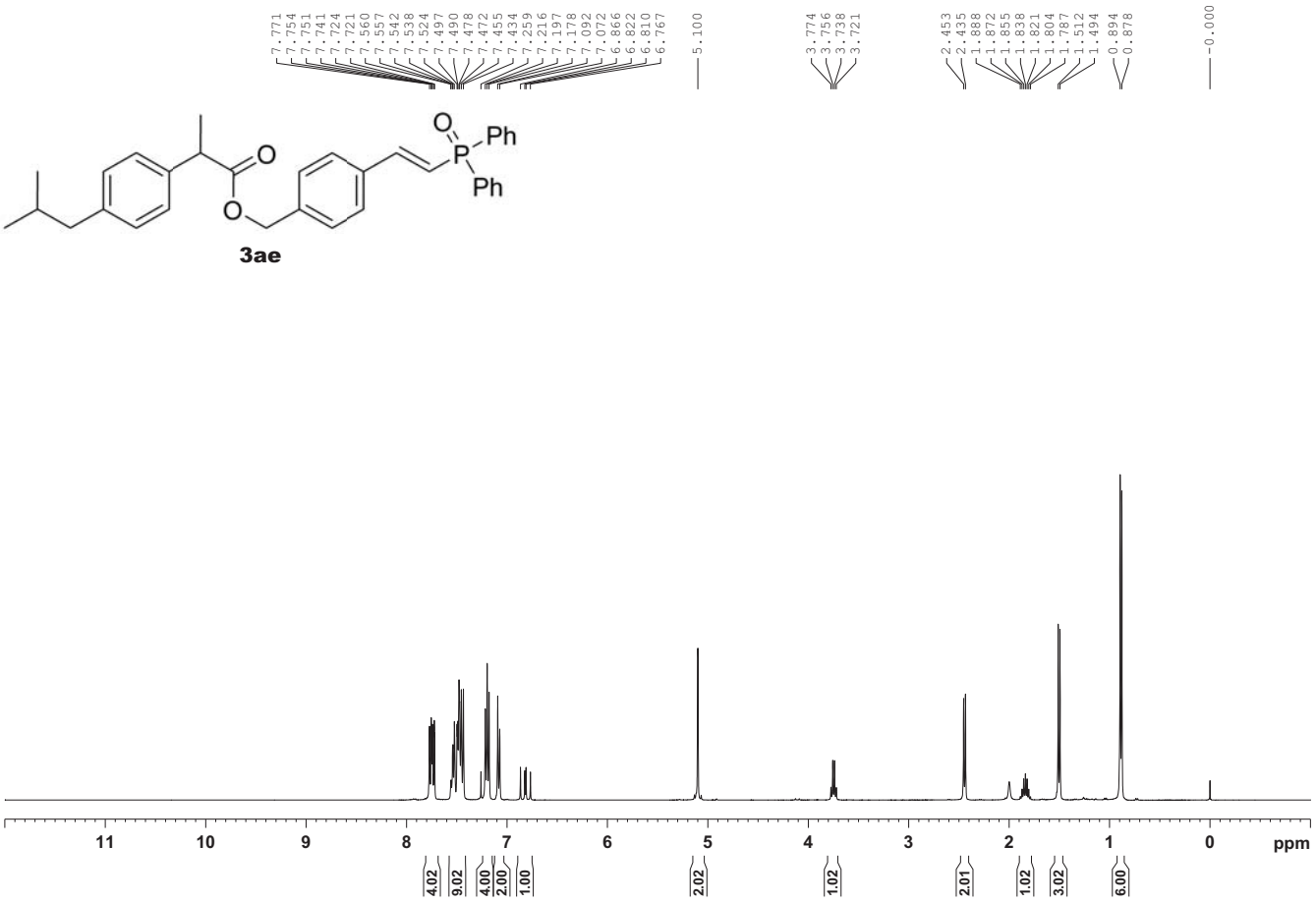
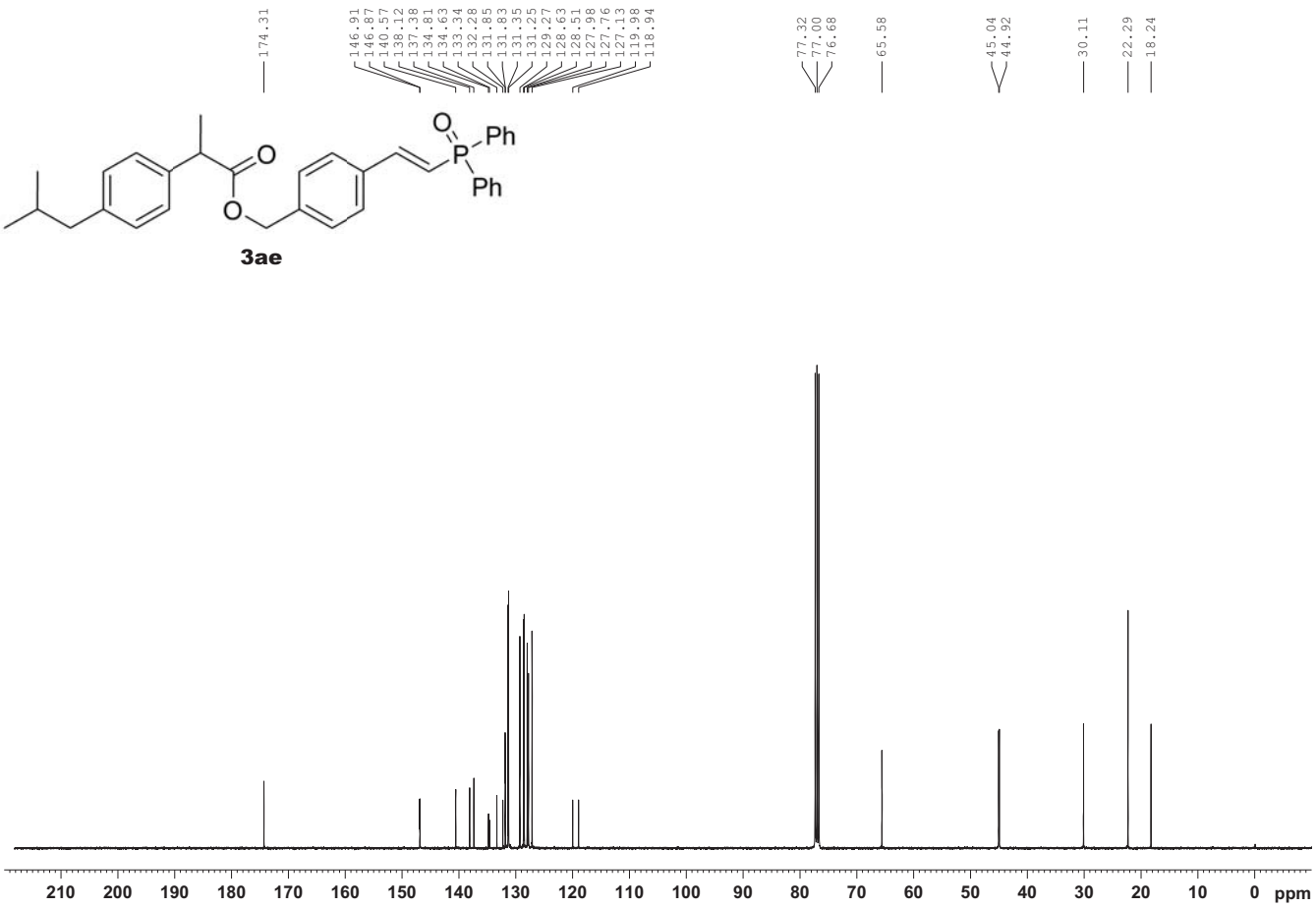
8.057
8.042
8.038
7.782
7.765
7.761
7.752
7.734
7.731
7.585
7.581
7.561
7.546
7.540
7.532
7.528
7.518
7.512
7.506
7.499
7.487
7.480
7.470
7.463
7.439
7.419
7.329
7.327
7.309
7.290
7.263
7.107
7.087
6.916
6.872
6.860
6.817
5.307
2.167
-0.000
O
O
O
O
O
P
Ph
Ph
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
4.03
10.52
2.07
1.06
1.00
1.06
2.03
3.05

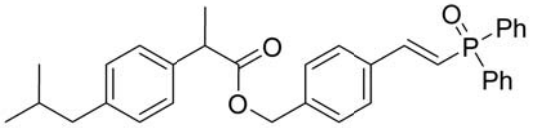
3ad


169.62
164.20
150.68
146.80
146.77
137.55
135.26
135.09
134.05
133.32
132.27
131.91
131.88
131.87
131.39
131.29
128.71
128.68
128.56
127.99
126.04
123.85
122.99
120.39
119.36
77.32
77.00
76.68
66.32
20.81
O
O
O
O
O
P
Ph
Ph
3ad
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


24.31
O
O
O
O
O
P
Ph
Ph
3ad
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm


7.771
7.754
7.751
7.741
7.724
7.721
7.560
7.557
7.542
7.538
7.524
7.497
7.490
7.478
7.472
7.455
7.434
7.259
7.216
7.197
7.178
7.092
7.072
6.866
6.822
6.810
6.767
5.100
3.774
3.756
3.738
3.721
2.453
2.435
1.888
1.872
1.855
1.838
1.821
1.804
1.787
1.512
1.494
0.894
0.878
-0.000
O
O
O
P
Ph
Ph
3ae
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.02
9.02
4.00
2.00
1.00
2.02
1.02
2.01
1.02
3.02
6.00


174.31
146.91
146.87
140.57
138.12
137.38
134.81
134.63
133.34
132.28
131.85
131.83
131.35
131.25
129.27
128.63
128.51
127.98
127.76
127.13
119.98
118.94
77.32
77.00
76.68
65.58
45.04
44.92
30.11
22.29
18.24
O
O
O
P
Ph
Ph
3ae
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



3ae

24.46
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm


8.185
8.164
7.883
7.862
7.779
7.762
7.758
7.749
7.732
7.728
7.575
7.555
7.531
7.506
7.499
7.487
7.480
7.477
7.467
7.462
7.456
7.267
6.922
6.879
6.867
6.823
5.391
3.112
3.093
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1.568
1.550
1.531
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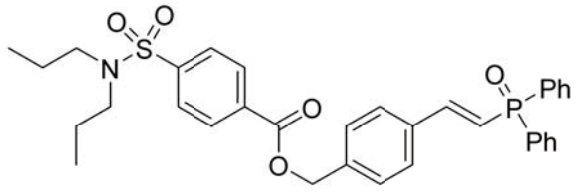
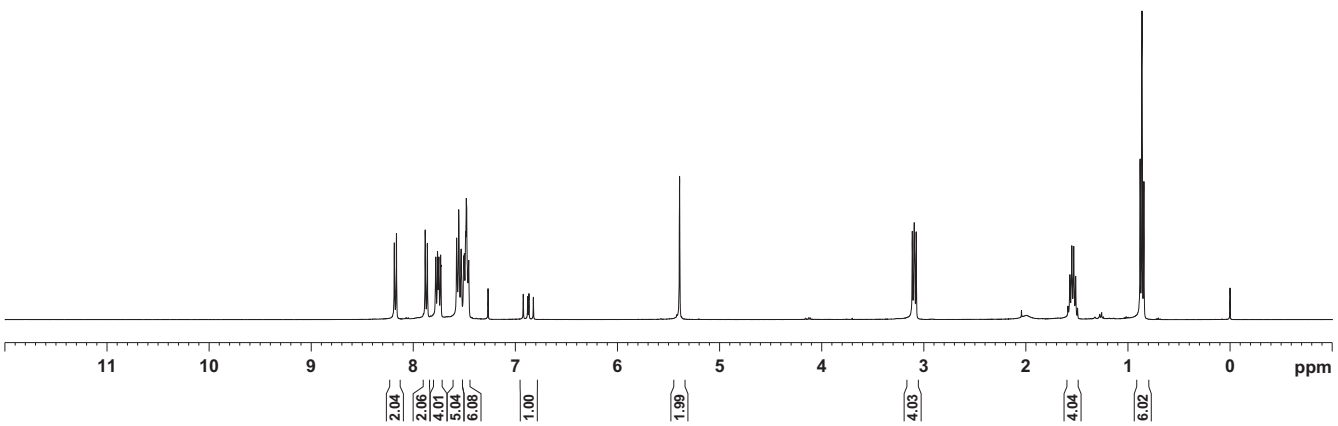
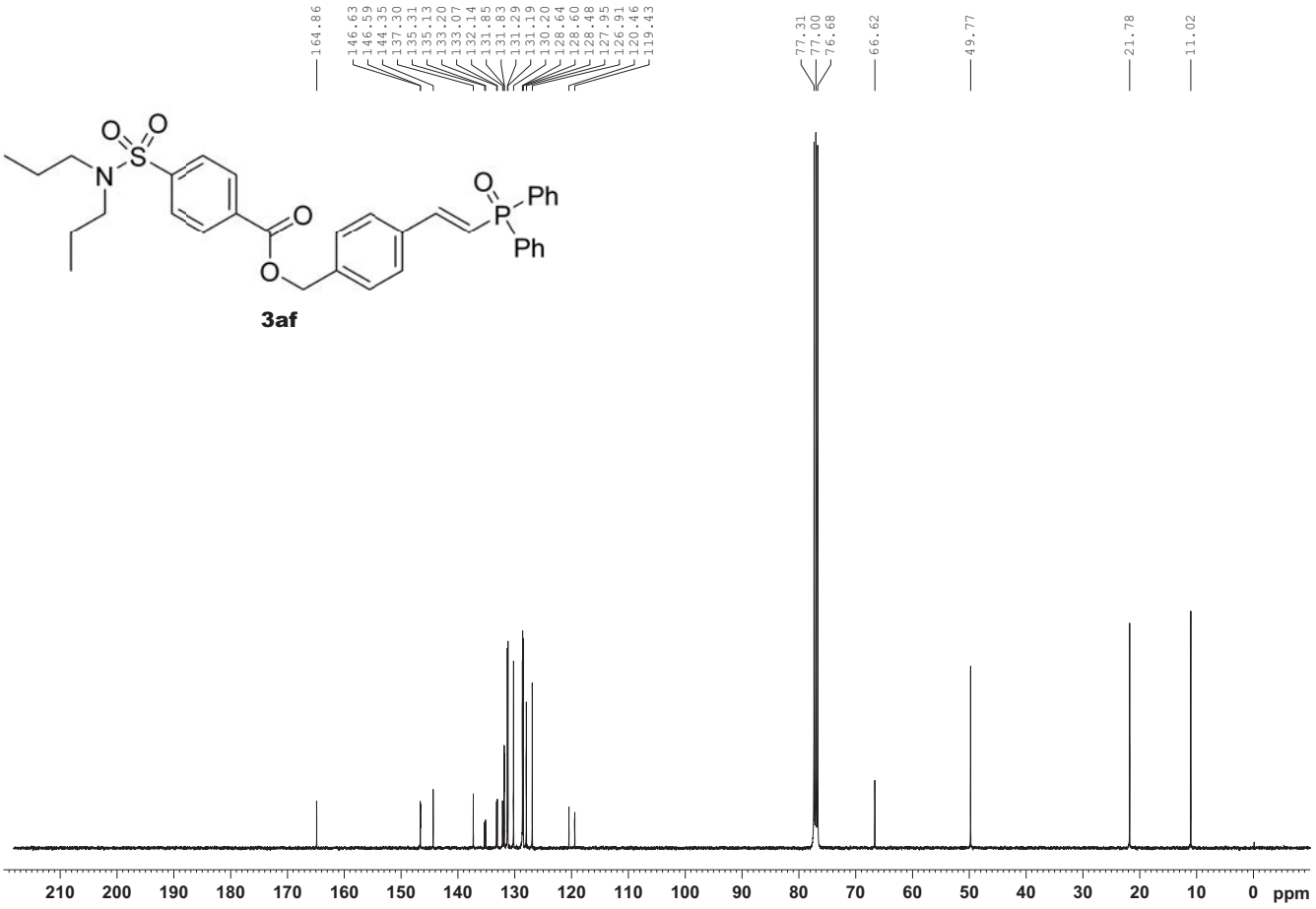
1.493
0.880
0.862
0.844
-0.000



3af

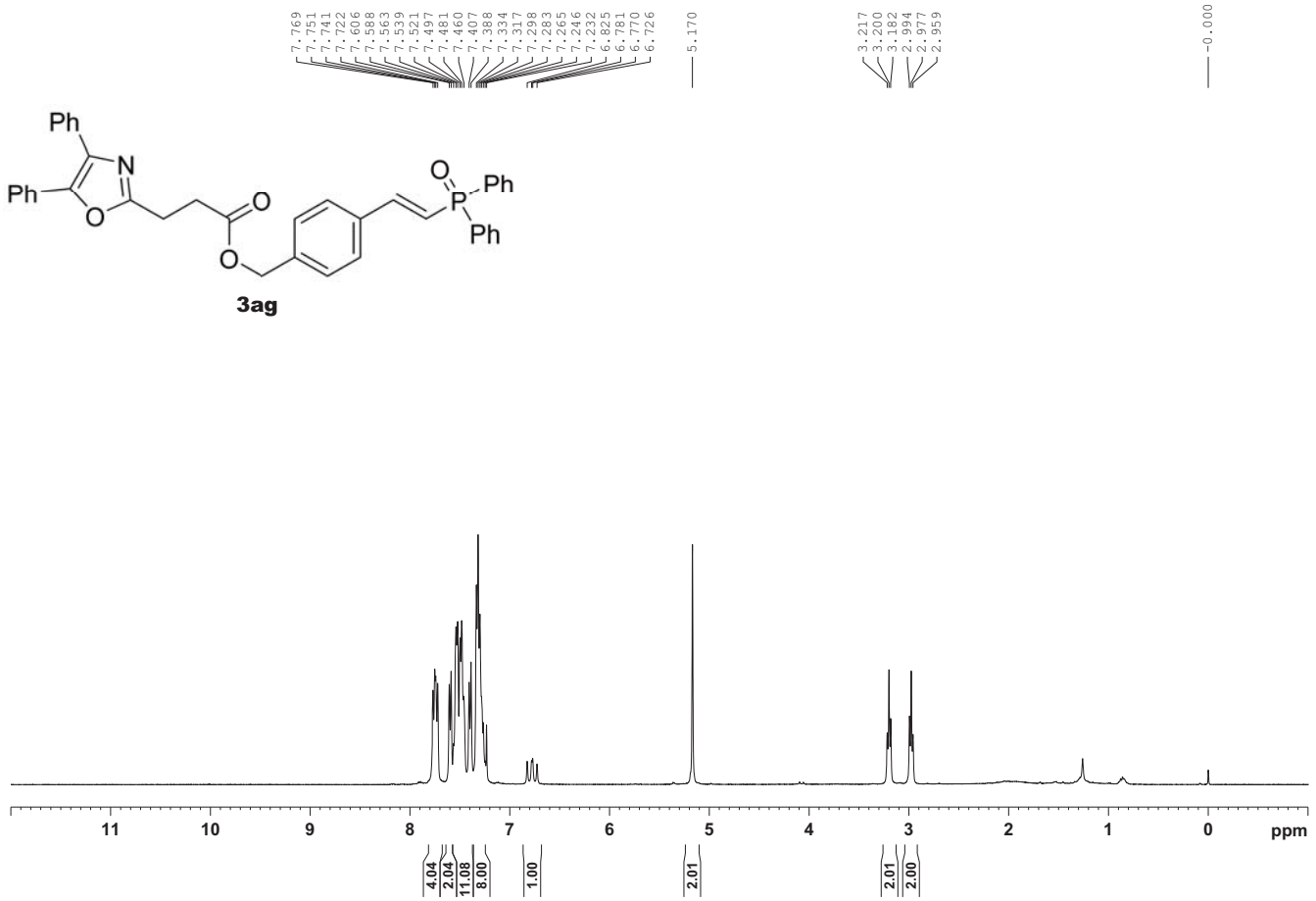

11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.04
2.06
4.01
5.04
6.08
1.00
1.99
4.03
4.04
6.02


164.86
146.63
146.59
144.35
137.30
135.31
135.13
133.20
133.07
132.14
131.85
131.83
131.29
131.19
130.20
128.64
128.60
128.48
127.95
126.91
120.46
119.43
77.31
77.00
76.68
66.62
49.77
21.78
11.02
O
O
S
N
O
O
Ph
P
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3af


24.29
O
O
S
N
O
O
Ph
P
Ph
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 ppm

3af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7.722
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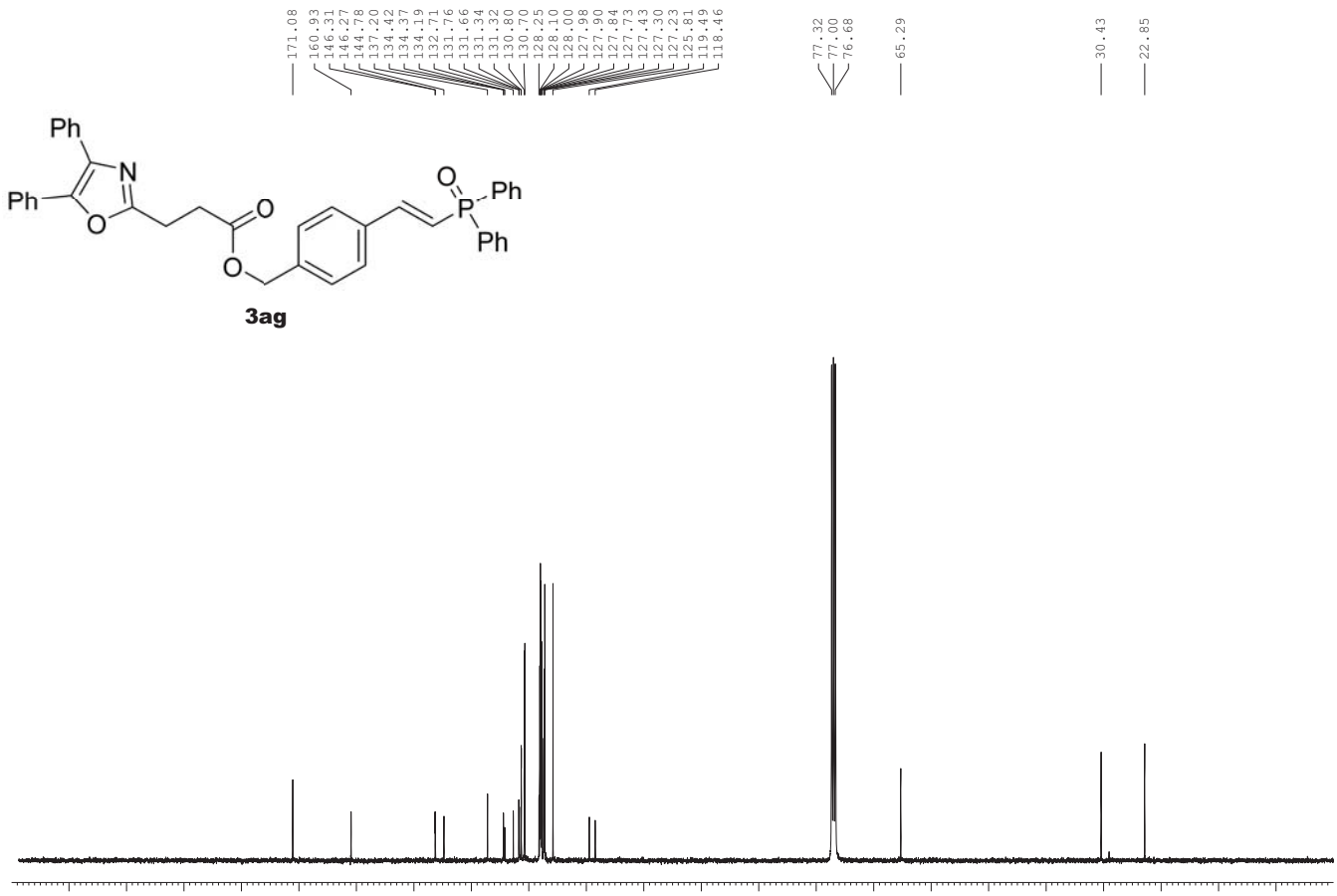
7.588
7.563
7.539
7.521
7.497
7.481
7.460
7.407
7.388
7.334
7.317
7.298
7.283
7.265
7.246
7.232
6.825
6.781
6.770
6.726
5.170
3.217
3.200
3.182
2.994
2.977
2.959
-0.000
Ph
N
Ph
O
O
O
O
P
Ph
Ph
3ag
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.04
2.04
11.08
8.00
1.00
2.01
2.01
2.00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144.78
137.20
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134.37
134.19
132.71
131.76
131.66
131.34
131.32
130.80
130.70
128.25
128.10
128.00
127.98
127.90
127.84
127.73
127.43
127.30
127.23
125.81
119.49
118.46
77.32
77.00
76.68
65.29
30.43
22.85
Ph
N
Ph
O
O
O
O
P
Ph
Ph
3ag
210
200
190
180
170
160
150
140
130
120
110
100
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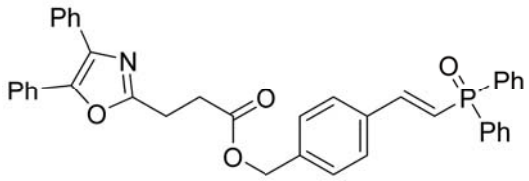
80
70
60
50
40
30
20
10
0
ppm



3ag

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70
60
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40
30
20
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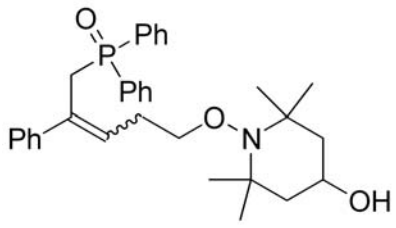
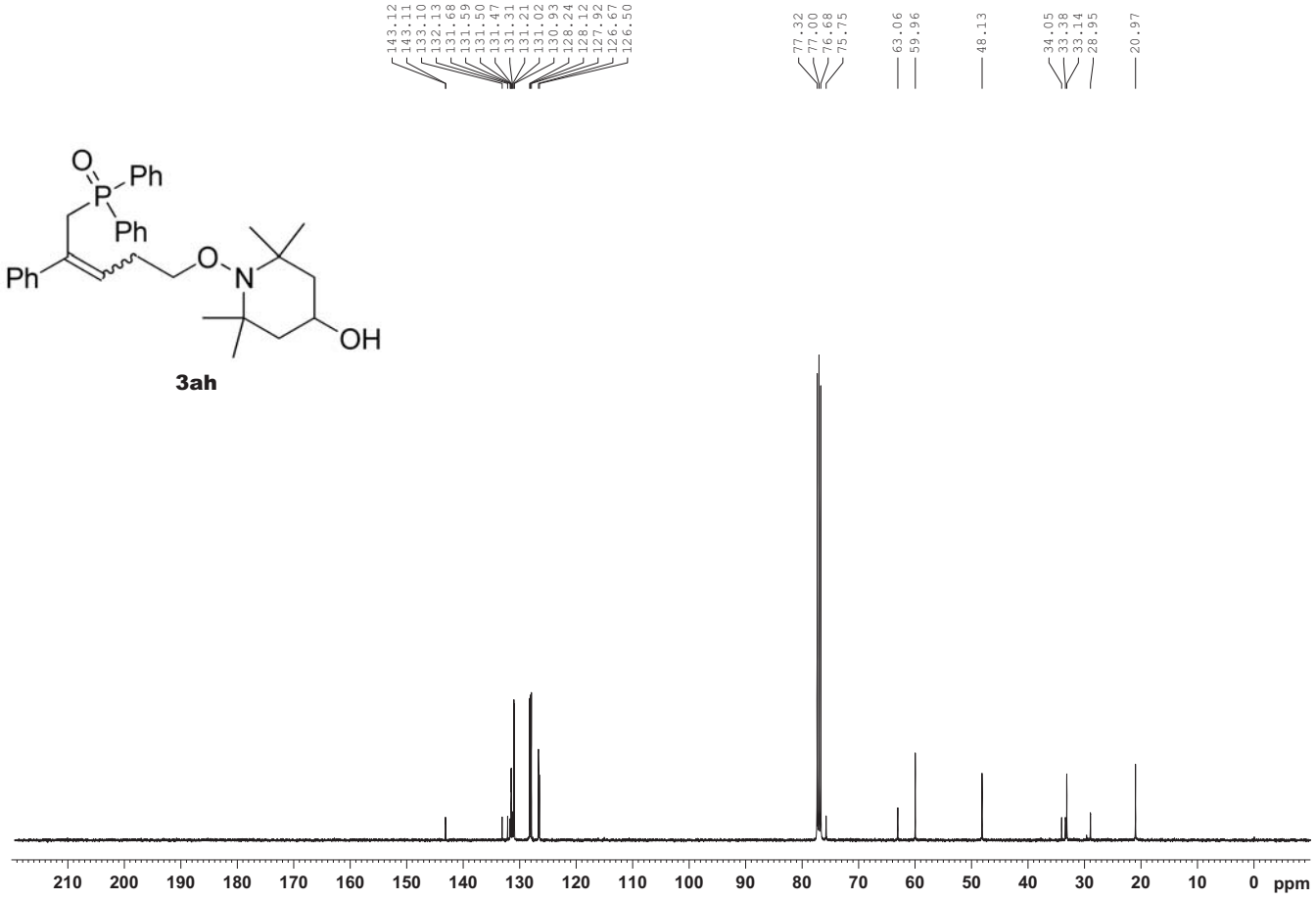
0
-10
-20
-30
-40
-50
ppm



3ah

7.649
7.632
7.628
7.621
7.603
7.600
7.432
7.429
7.413
7.410
7.395
7.392
7.338
7.331
7.319
7.312
7.301
7.293
7.266
7.086
5.870
5.852
5.839
5.821
3.952
3.942
3.924
3.913
3.903
3.885
3.778
3.762
3.746
3.695
3.658
2.397
2.381
2.372
2.364
2.355
2.339
1.785
1.779
1.757
1.748
1.461
1.432
1.402
1.146
1.116
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
4.05
2.07
4.08
5.03
1.00
1.05
2.03
2.06
2.02
2.05
2.09
12.04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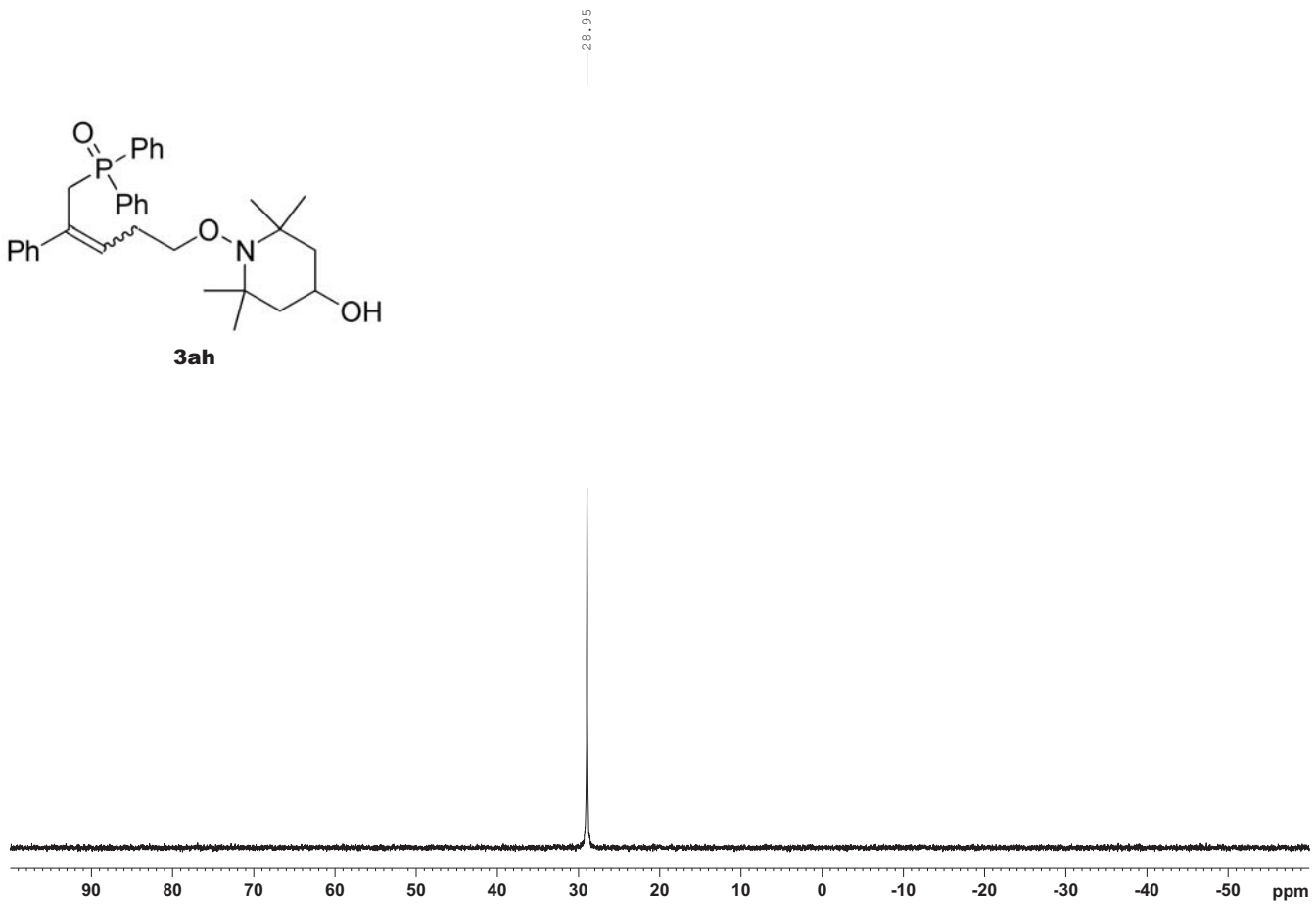
143.11
133.10
132.13
131.68
131.59
131.50
131.47
131.31
131.21
131.02
130.93
128.24
128.12
127.92
126.67
126.50
77.32
77.00
76.68
75.75
63.06
59.96
48.13
34.05
33.38
33.14
28.95
20.97
O
P
Ph
Ph
Ph
O
N
OH
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3ah


28.95
O
P
Ph
Ph
Ph
O
N
OH
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
ppm

3ah