Supplementary Information

Dehydrative allylation of P-H species under metal-free conditions

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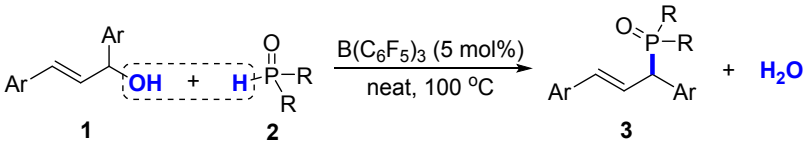
1. General information

Unless otherwise noted, all commercially available compounds were used as received. All solvents were purified according to standard procedures. The ^{1}H NMR was recorded at 400MHz, ^{13}C NMR was recorded at 101 MHz, ^{31}P NMR was recorded at 162 MHz. ^{1}H, ^{13}C NMR chemical shifts were calibrated to *tetra*-methylsilane as an external reference. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), d (doublet), t (triplet), dd (doublet of doublets), m (multiplet); coupling constants (*J*) are in Hertz (Hz). IR spectra were recorded on a Thermo Scientific Nicolet iS-5 FT-IR spectrometer and are reported in terms of frequency of absorption (cm^{-1}). HRMS were obtained on an IonSpec FT-ICR mass spectrometer with ESI resource. Melting points were measured on a RY-I apparatus and are reported uncorrected. The starting materials allylic alcohols **1** and P-H species **2** were readily prepared according to the related literatures.[1,2] The catalyst $B(C_6F_5)_3$ was purchased from *Energy Chemical Ltd* (Shanghai).

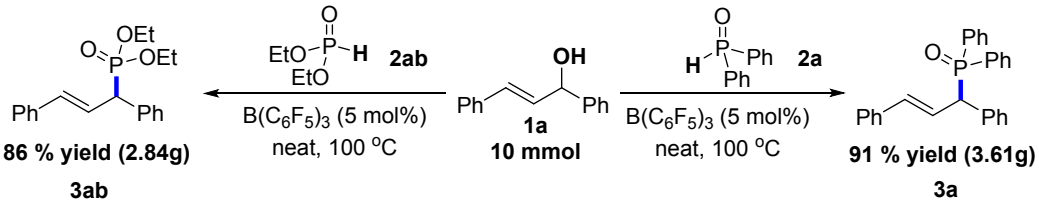
2. General procedure

2.1 General procedure for preparation of 3a-3ak



Allyl alcohols **1** (0.2 mmol) and P-H species **2** (0.3 mmol) was added to dried Schlenk tube (10 mL), $B(C_6F_5)_3$ (5 mol%) was then added subsequently. The reaction was stirred under Ar atmosphere at 60 °C-120 °C (oil bath) for 2-24 h. After complete conversion, products was purified *via* PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) to afford the corresponding products **3a-3ak**.

2.2 Procedure for gram scale reactions

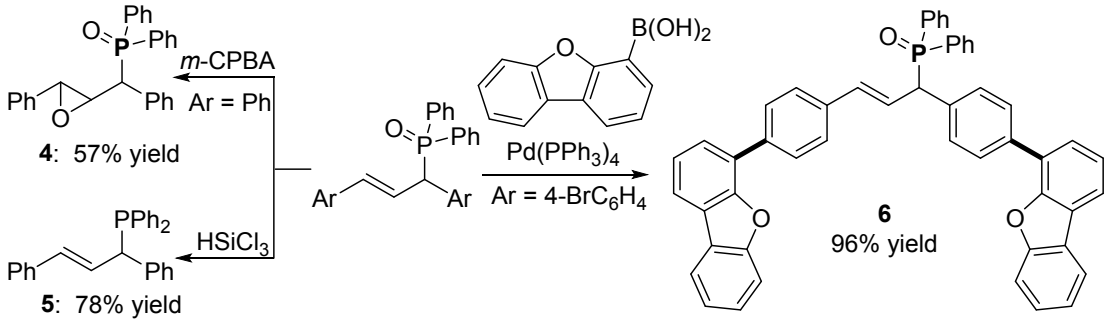


Procedure for synthesis of 3a in gram scale: (E)-1,3-diphenylprop-2-en-1-ol 1a (10.0 mmol), diphenylphosphine oxide **2a** (15.0 mmol) was added to round-bottomed flask (100 mL), and $B(C_6F_5)_3$ (0.5 mmol) was then added subsequently. The reaction was stirred under Ar atmosphere at 100 °C (oil bath) for 12 h. After complete conversion, product was purified via column chromatography

(Petroleum ether (bp: 60-90 °C)/ethyl acetate = 3/1) to afford the corresponding product **3a** (3.61 g, 91 % yield).

Procedure for synthesis of 3ab in gram scale: (*E*)-1,3-diphenylprop-2-en-1-ol **1a** (10.0 mmol), diethyl phosphite **2ab** (40.0 mmol) was added into round-bottomed flask (100 mL), and $B(C_6F_5)_3$ (0.5 mmol) was then added subsequently. The reaction was stirred under Ar atmosphere at 100 °C (sand bath) for 12 h. After complete conversion, products was purified *via* column chromatography (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 3/1) to afford the corresponding product **3ab** (2.84 g, 86 % yield).

2.3 Preliminary protocol applications.



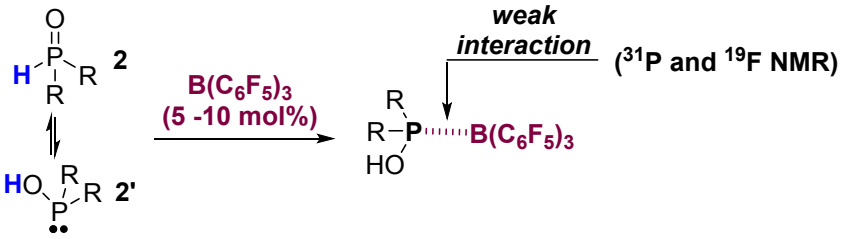
Procedure for synthesis of **4**: Allylic phosphorus compound **3a** (0.2 mmol) and *m*-CPBA (0.4 mmol) was dissolved in CH_2Cl_2 (2.0 mL) in Schlenk tube (10 mL). The reaction was stirred at room temperature for 36 h. After complete conversion, the residue was purified by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) to afford the corresponding product **4** in 57 % yield.

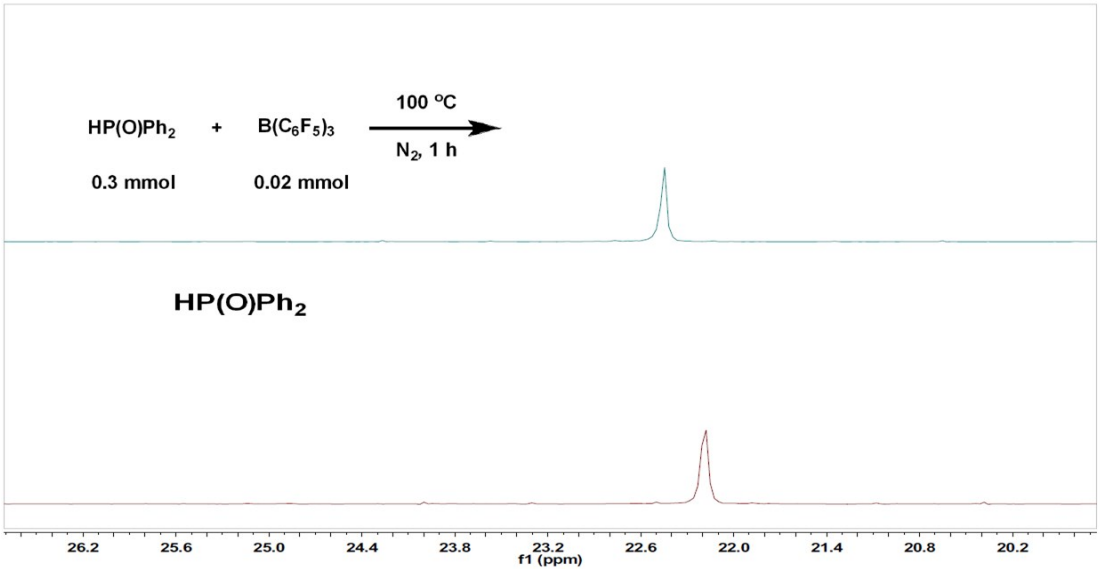
Procedure for synthesis of 5 (known compound [6]): Allylic phosphorus compound 3a (0.2 mmol), $HSiCl_3$ (4.0 mmol), and NEt_3 (2.0 mmol) was dissolved in toluene (2.0 mL) in Schlenk tube (10 mL). The reaction was stirred at 120 °C (sand bath) for 3 h. After filtration, the product was washed with MeOH (10 mL) and dried under vacuum to afford the corresponding product 5 (78 % yield).

Procedure for synthesis of **6**: Allylic phosphorus compound **3a** (0.2 mmol), dibenzo[b,d]furan-4-yl boronic acid (4.0 equiv.), $Pd(PPh_3)_4$ (10 mol%), and CsF (5.0 equiv) was dissolved in xylene (2.0 mL) in Schlenk tube (10 mL) under N_2 atmosphere. The reaction was stirred at 60 °C (oil bath) for 12 h. After complete conversion, the solvent was removed under vacuum. Then, the residue was purified by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) to afford the corresponding product **6** (96 % yield).

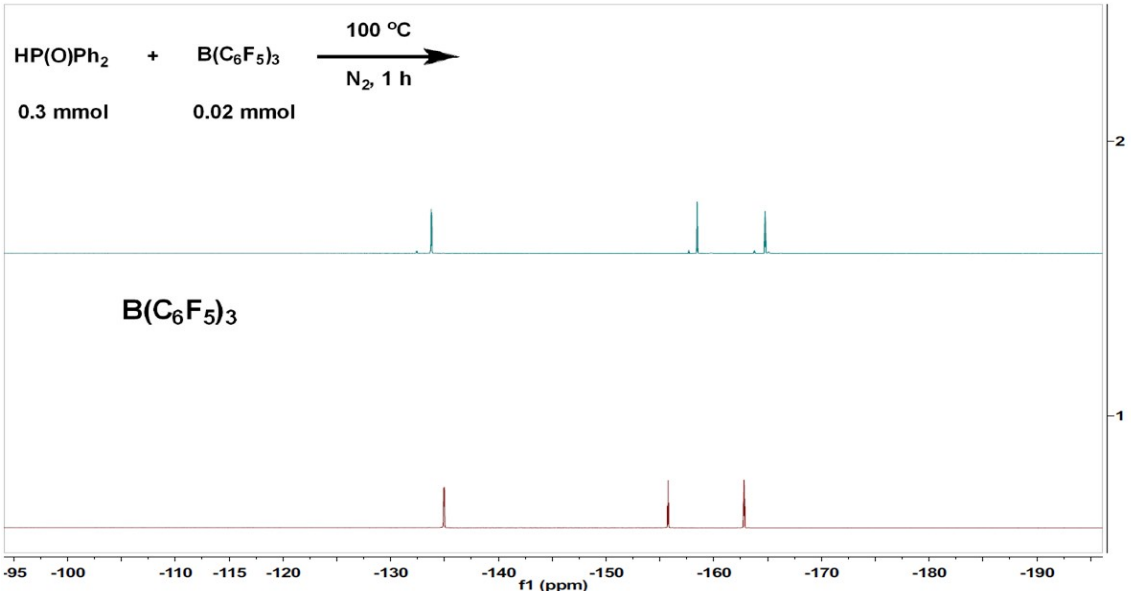
3. Control experiments.

3.1 The interaction between P-H species and $B(C_6F_5)_3$



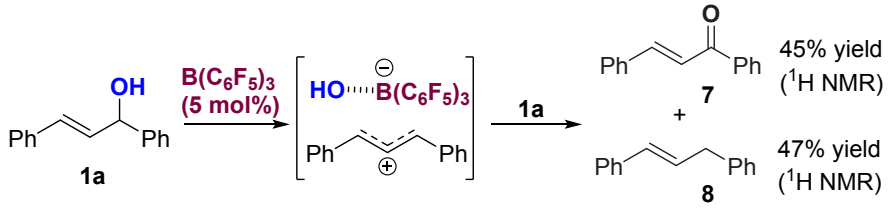


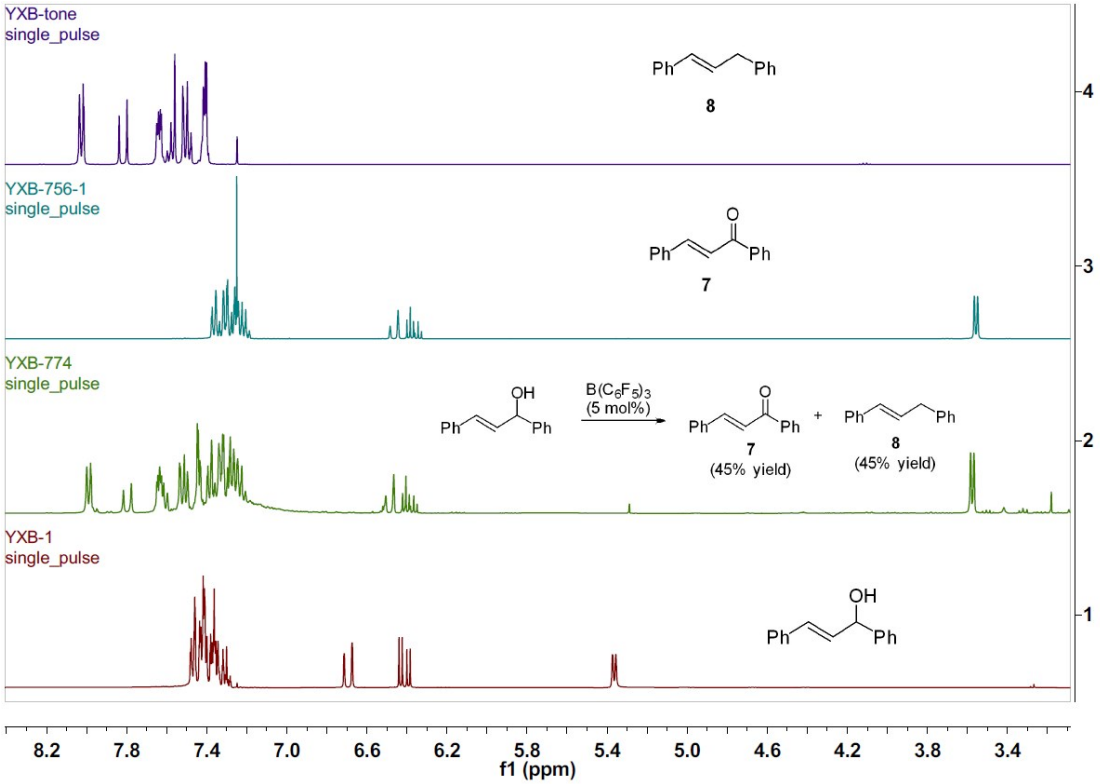
^{31}P NMR Spectra



^{19}F NMR Spectra

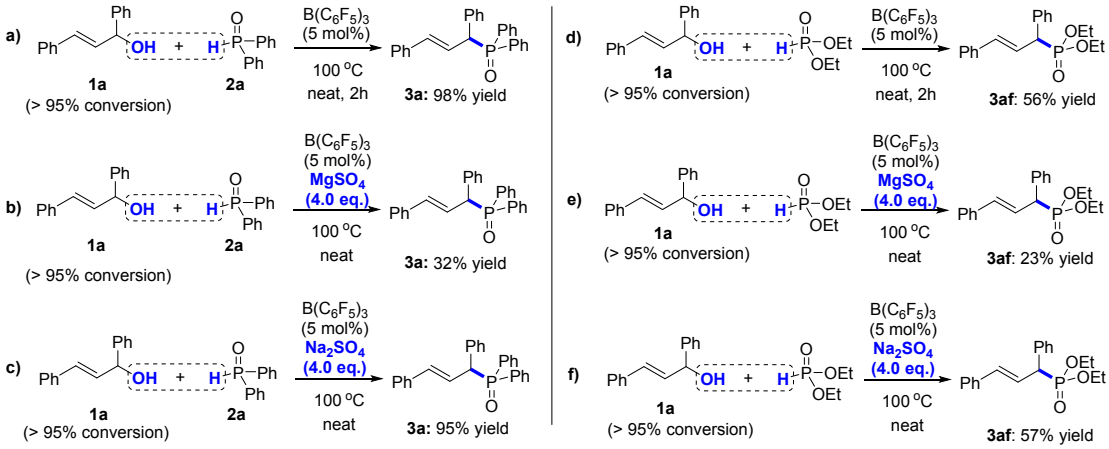
3.2 The interaction between allylic alcohol and $B(C_6H_5)_3$





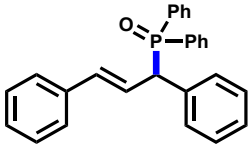
^{1}H NMR Spectra

3.3 The reaction in the presence of dehydrating agents



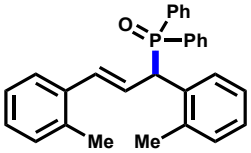
4. Analytical data for all new compounds

(E)-(1,3-diphenylallyl)diphenylphosphine oxide (3a)



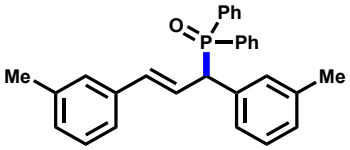
3a was known compounds[3, 4, 5]. Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3a** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (77 mg, 98% yield), Mp: 203-205 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.80 (m, 2H), 7.61 – 7.55 (m, 2H), 7.48 – 7.39 (m, 3H), 7.38 – 7.32 (m, 3H), 7.31 – 7.26 (m, 2H), 7.23 – 7.13 (m, 8H), 6.59 (ddd, *J* = 15.8, 9.1, 7.1 Hz, 1H), 6.32 (dd, *J* = 15.8, 3.8 Hz, 1H), 4.38 (t, *J* = 9.4 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.8, 136.8, 136.0 (d, J_{C-P} = 6.0 Hz), 134.6, 134. 4, 132.2 (d, J_{C-P} = 31.3 Hz), 131.9 (d, J_{C-P} = 2.9 Hz), 131.8, 131.8, 131.6 (d, J_{C-P} = 2.7 Hz), 131.5, 131.4, 131.2 (d, J_{C-P} = 31.6 Hz), 129.6, 129.5, 128.7, 128.7, 128.6, 128.5, 128.5, 128.4, 128.2, 127.7, 127.2 (d, J_{C-P} = 2.4 Hz), 126.5 (d, J_{C-P} = 1.8 Hz), 124.7 (d, J_{C-P} = 7.2 Hz), 52.4 (d, J_{C-P} = 65.0 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.06. IR (KBr): 3057.30, 2909.00, 1597.06, 1492.77, 1437.23, 1170.76, 1118.33, 1101.29, 1029.07, 972.24, 719.89, 691.76 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{27}H_{24}OP$ 395.1565, found 395.1559.

(E)-(1,3-di-o-tolylallyl)diphenylphosphine oxide (3b)



3b was known compounds[3, 5]. Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3b** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (84 mg, 99% yield), Mp: 203-205 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.85 (m, 3H), 7.57 – 7.44 (m, 5H), 7.42 – 7.34 (m, 1H), 7.27 (s, 3H), 7.20 (t, *J* = 7.3 Hz, 1H), 7.15 – 6.99 (m, 5H), 6.54 – 6.43 (m, 1H), 6.34 (dt, *J* = 15.3, 7.9 Hz, 1H), 4.64 (t, *J* = 9.4 Hz, 1H), 2.14 (s, 3H), 2.00 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.0 (d, J_{C-P} = 1.9 Hz), 135.9 (d, J_{C-P} = 7.6 Hz), 135.2, 134.5 (d, J_{C-P} = 5.4 Hz), 132.9, 132.6, 132. 4, 132.0, 132.0, 131.9 (d, J_{C-P} = 3.4 Hz), 131.6 (d, J_{C-P} = 2.7 Hz), 131.2, 131.2, 131.0, 130.5, 130.0, 129.5 (d, J_{C-P} = 4.5 Hz), 128.6, 128.5, 128.3, 128.2, 127.6, 127.2, 126.6, 126.1, 126.0, 47.9 (d, J_{C-P} = 66.3 Hz), 19.8, 19.6. ^{31}P NMR (162 MHz, Chloroform-*d*) δ 32.58. IR (KBr): 3055.20, 2924.95, 1484.92, 1460.07, 1437.11, 1185.50, 1117.12, 1070.62, 1070.62, 964.09, 720.25, 695.62 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{29}H_{28}OP$ 423.1878, found 423.1879.

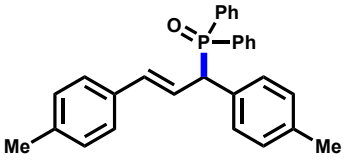
(E)-(1,3-di-m-tolylallyl)diphenylphosphine oxide(3c)



3c was known compounds[3, 4, 5]. Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3c** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (71 mg, 84% yield), Mp: 168-169 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.80 (m, 2H),

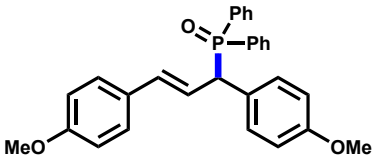
7.61 – 7.54 (m, 2H), 7.52 – 7.42 (m, 3H), 7.38 (td, *J* = 7.3, 1.4 Hz, 1H), 7.32 – 7.27 (m, 2H), 7.17 – 7.04 (m, 5H), 6.99 (t, *J* = 7.6 Hz, 3H), 6.58 (ddd, *J* = 16.0, 9.2, 7.3 Hz, 1H), 6.29 (dd, *J* = 15.7, 3.7 Hz, 1H), 4.33 (t, *J* = 9.4 Hz, 1H), 2.27 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.2, 138.1, 136.7, 135.9 (d, $J_{C\text{-}P}$ = 6.0 Hz), 134.5 (d, $J_{C\text{-}P}$ = 11.4 Hz), 132.3 (d, $J_{C\text{-}P}$ = 35.6 Hz), 131.9, 131.8, 131.6, 131.5, 131.4, 131.1, 130.3 (d, $J_{C\text{-}P}$ = 5.7 Hz), 128.6, 128.5, 128.4, 128.3, 128.2, 128.0, 127.1, 126.5 (d, $J_{C\text{-}P}$ = 5.8 Hz), 124.5 (d, $J_{C\text{-}P}$ = 7.4 Hz), 123.8, 52.4 (d, $J_{C\text{-}P}$ = 65.2 Hz), 21.5, 21.5. ^{31}P NMR (162 MHz, Chloroform-d) δ 31.99. IR (KBr): 3051.61, 2920.57, 2854.29, 1603.31, 1586.27, 1486.48, 1437.44, 1175.49, 1116.43, 1071.01, 963.48, 781.25, 718.76, 703.55 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{29}H_{28}OP$ 423.1878, found 423.1880.

(E)-(1,3-di-p-tolylallyl)diphenylphosphine oxide (3d)



3d was known compounds[3, 4, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3d** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (84 mg, 99% yield), Mp: 182-183 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.75 (m, 2H), 7.64 – 7.56 (m, 2H), 7.52 – 7.37 (m, 4H), 7.35 – 7.28 (m, 2H), 7.22 (dd, *J* = 8.1, 1.9 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 4H), 6.49 (ddd, *J* = 16.0, 9.1, 7.0 Hz, 1H), 6.25 (dd, *J* = 15.7, 3.9 Hz, 1H), 4.32 (t, *J* = 9.6 Hz, 1H), 2.28 (s, 3H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.5 (d, $J_{C\text{-}P}$ = 0.9 Hz), 136.8 (d, $J_{C\text{-}P}$ = 2.4 Hz), 134.2, 134.1, 134.1, 133.0, 132.9, 132.4 (d, $J_{C\text{-}P}$ = 30.4 Hz), 131.9, 131.8, 131.7 (d, $J_{C\text{-}P}$ = 16.9 Hz), 131.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.5, 131.4, 131.3, 129.4, 129.4, 129.4, 129.2, 128.5, 128.4, 128.3, 128.2, 126.4, 126.4, 123.8 (d, $J_{C\text{-}P}$ = 7.1 Hz), 52.0 (d, $J_{C\text{-}P}$ = 65.5 Hz), 21.3, 21.2. ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.99. IR (KBr): 3051.67, 3024.73, 2920.68, 2854.24, 1511.13, 1437.44, 1180.59, 1117.79, 1102.92, 1070.87, 968.82, 824.41, 801.93, 728.68, 718.45, 694.90 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{29}H_{28}OP$ 423.1878, found 423.1872.

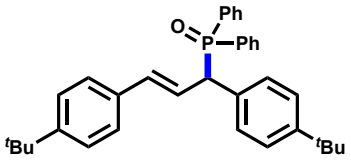
(E)-(1,3-bis(4-methoxyphenyl)allyl)diphenylphosphine oxide(3e)



3e was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3e** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (82 mg, 90% yield), Mp: 137-139 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.80 (m, 2H), 7.63 – 7.56 (m, 2H), 7.48 – 7.39 (m, 3H), 7.38 – 7.32 (m, 1H), 7.31 – 7.26 (m, 4H), 7.16 – 7.10 (m, 2H), 6.76 (d, *J* = 2.9 Hz, 2H), 6.73 (d, *J* = 3.0 Hz, 2H), 6.41 (ddd, *J* = 15.8, 8.9, 7.0 Hz, 1H), 6.25 (dd, *J* = 15.8, 3.7 Hz, 1H), 4.34 (t, *J* = 9.4 Hz, 1H), 3.70 (s, 3H), 3.68 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.3, 158.7 (d, $J_{C\text{-}P}$ = 2.3 Hz), 133.8, 133.7, 132.5 (d, $J_{C\text{-}P}$ = 29.6 Hz), 131.8, 131.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.7, 131.7, 131.6 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.5, 131.4, 130.6, 130.6, 129.7 (d, $J_{C\text{-}P}$ = 2.4 Hz), 128.6, 128.4, 128.4, 128.2, 128.1 (d, $J_{C\text{-}P}$ = 5.9 Hz), 127.6, 127.6, 122.6 (d, $J_{C\text{-}P}$ = 7.0 Hz), 114.1, 114.1, 113.9, 55.3, 55.3, 51.3 (d, $J_{C\text{-}P}$ = 66.0 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.24. IR (KBr): 3055.39, 3031.19, 3005.95, 2956.04, 2933.66, 2907.62, 2835.50, 1608.06, 1577.13, 1510.50, 1484.94, 1462.70, 1437.74, 1293.30, 1251.21, 1174.48, 1118.77, 968.80, 864.91, 828.96, 811.65,

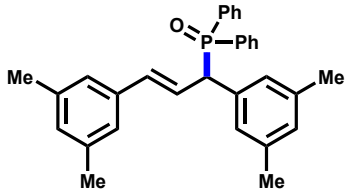
734.36, 719.08, 710.32, 694.26. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{29}H_{28}O_3P$ 455.1776, found 455.1771.

(E)-(1,3-bis(4-(tert-butyl)phenyl)allyl)diphenylphosphine oxide (3f)



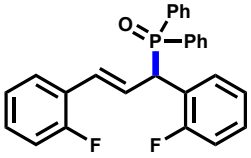
3f was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3f** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (41 mg, 40% yield), Mp: 257-258 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.81 (m, 2H), 7.58 – 7.51 (m, 2H), 7.51 – 7.42 (m, 3H), 7.39 (td, *J* = 7.3, 1.1 Hz, 1H), 7.29 (td, *J* = 6.7, 5.8, 1.9 Hz, 2H), 7.29 – 7.21 (m, 2H), 7.21 (s, 4H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.55 (ddd, *J* = 16.1, 9.3, 7.3 Hz, 1H), 6.33 (dd, *J* = 15.7, 3.7 Hz, 1H), 4.35 (t, *J* = 9.6 Hz, 1H), 1.27(s, 9H), 1.25 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.7 (d, J_{C-P} = 0.9 Hz), 150.0 (d, J_{C-P} = 2.5 Hz), 134.1 (d, J_{C-P} = 2.3 Hz), 134.1 (d, J_{C-P} = 11.3 Hz), 132.9, 132.8, 132.7, 132.0, 131.9, 131.9, 131.8 (d, J_{C-P} = 2.7 Hz), 131.7, 131.5, 131.5, 131.4, 131.1, 129.1 (d, J_{C-P} = 5.7 Hz), 128.5, 128.4, 128.2, 128.1, 126.2, 126.2, 125.5 (d, J_{C-P} = 1.5 Hz), 125.4, 124.0 (d, J_{C-P} = 6.9 Hz), 51.9 (d, J_{C-P} = 65.1 Hz), 34.6, 34.5, 31.4, 31.3. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.16. IR (KBr): 3055.47, 2963.47, 1509.51, 1436.69, 1364.81, 1181.77, 1171.09, 1117.57, 781.57, 693.11 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{35}H_{40}OP$ 507.2817, found 507.2823.

(E)-(1,3-bis(3,5-dimethylphenyl)allyl)diphenylphosphine oxide (3g)



3g was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3g** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (46 mg, 51% yield), Mp: 194-195 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.80 (m, 2H), 7.59 – 7.54 (m, 2H), 7.51 – 7.43 (m, 3H), 7.41 – 7.36 (m, 1H), 7.33 – 7.28 (m, 2H), 6.88 (d, *J* = 19.6 Hz, 4H), 6.80 (d, *J* = 8.6 Hz, 2H), 6.55 (ddd, *J* = 15.8, 9.4, 7.2 Hz, 1H), 6.24 (dd, *J* = 15.8, 3.8 Hz, 1H), 4.27 (t, *J* = 9.6 Hz, 1H), 2.24 (s, 6H), 2.19 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.0, 137.9, 136.8 (d, J_{C-P} = 1.7 Hz), 135.9 (d, J_{C-P} = 5.9 Hz), 134.4 (d, J_{C-P} = 11.5 Hz), 132.4 (d, J_{C-P} = 37.4 Hz), 131.9, 131.8, 131.8 (d, J_{C-P} = 2.4 Hz), 131.7, 131.6, 131.5, 131.3, 129.4, 128.8, 128.5, 128.4, 128.2, 128.1, 127.3, 127.3, 124.4, 52.4 (d, J_{C-P} = 65.4 Hz), 21.4, 21.3. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.12. IR (KBr): 3054.47, 3024.56, 2916.86, 1599.53, 1437.54, 1176.57, 1117.89, 1102.86, 965.14, 852.47, 837.77, 749.53, 722.66, 704.54 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{31}H_{32}OP$ 451.2191, found 451.2187.

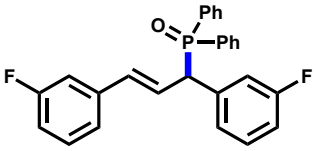
(E)-(1,3-bis(4-(tert-butyl)phenyl)allyl)diphenylphosphine oxide (3h)



3h was known compounds[3]**.** Following the general procedure, the reaction was stirred at 120 ºC (oil bath) for 12h. **3h** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white

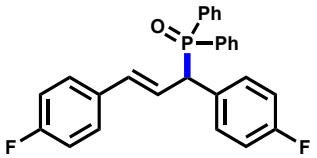
solid (35 mg, 41% yield), Mp: 180-182 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.87 (m, 3H), 7.66 – 7.61 (m, 2H), 7.52 – 7.46 (m, 3H), 7.40 – 7.28 (m, 4H), 7.16 – 7.08 (m, 3H), 7.00 (td, *J* = 7.6, 0.9 Hz, 1H), 6.95 – 6.82 (m, 2H), 6.62 (ddd, *J* = 15.5, 9.0, 6.3 Hz, 1H), 6.51 (dd, *J* = 16.0, 3.6 Hz, 1H), 4.90 (t, *J* = 8.8 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.5 (d, $J_{C\text{-}F}$= 250.48 Hz), 159.8 (dd, $J_{C\text{-}F,\ C\text{-}P}$ = 245.43, 6.7Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.0 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.5 Hz), 131.6, 131.6, 131.0, 131.0, 130.8 (d, $J_{C\text{-}P}$ = 3.5 Hz), 129.1 (d, $J_{C\text{-}P}$ = 8.4 Hz), 128.8 (d, $J_{C\text{-}P}$ = 8.7 Hz), 128.7, 128.6, 128.4, 128.3, 127.4 (dd, $J_{C\text{-}F,\ C\text{-}P}$ = 5.4, 4.5 Hz), 125.9 (dd, $J_{C\text{-}F,\ C\text{-}P}$ = 7.1, 4.3 Hz), 124.6, 124.4 (d, d, $J_{C\text{-}P}$ = 11.9 Hz), 124.1, 124.1, 123.3 (dd, $J_{C\text{-}F,\ C\text{-}P}$ = 14.5, 5.5 Hz), 115.7, 115.5, 115.3, 115.1, 43.2 (d, d, $J_{C\text{-}P}$ = 66.5 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.53. ^{19}F NMR (376 MHz, Chloroform-d) δ -118.30, -118.47. IR (KBr): 3057.51, 2925.29, 1488.34, 1455.53, 1437.75, 1231.50, 1192.29, 1117.55, 1103.38, 966.50, 805.08, 721.23, 706.71, 695.45 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{27}H_{22}F_2OP$ 431.1376, found 431.1377.

(E)-(1,3-bis(3-fluorophenyl)allyl)diphenylphosphine oxide(3i)



3i was known compounds[3, 4]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3i** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (39 mg, 45% yield), Mp: 188-191 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.83 (m, 2H), 7.62 – 7.57 (m, 2H), 7.54 – 7.45 (m, 3H), 7.43 – 7.39 (m, 1H), 7.35 – 7.31 (m, 2H), 7.21 – 7.09 (m, 4H), 6.97 (d, *J* = 7.7 Hz, 1H), 6.91 – 6.85 (m, 3H), 6.54 (ddd, *J* = 16.0, 9.1, 7.1 Hz, 1H), 6.27 (dd, *J* = 15.7, 3.8 Hz, 1H), 4.36 (t, *J* = 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.0 (d, $J_{C\text{-}F}$ = 246.4 Hz), 162.7 (d, $J_{C\text{-}F}$ = 247.5 Hz), 138.8 (dd, $J_{C\text{-}F,C\text{-}P}$ = 7.6, 1.7 Hz), 138.2 (dd, $J_{C\text{-}F,C\text{-}P}$ = 6.6 Hz), 133.7 (dd, $J_{C\text{-}F,C\text{-}P}$ = 10.9, 1.8 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.8, 131.7, 131.6, 131.3, 131.3, 130.7 (d, $J_{C\text{-}P}$ = 19.4 Hz), 130.1 (dd, $J_{C\text{-}F,C\text{-}P}$ = 12.1, 8.3 Hz), 128.7, 128.6, 128.5, 128.4, 125.6 (d, $J_{C\text{-}P}$ = 7.3 Hz), 125.2 (dd, $J_{C\text{-}F,C\text{-}P}$ = 5.3, 3.0 Hz), 122.3, 116.6 (d, $J_{C\text{-}P}$ = 5.8 Hz), 116.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 114.7 (d, $J_{C\text{-}P}$ = 21.3 Hz), 114.3 (d, $J_{C\text{-}P}$ = 21.2 Hz), 113.0 (d, $J_{C\text{-}P}$ = 21.7 Hz), 52.0 (d, $J_{C\text{-}P}$ = 64.6 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.86. ^{19}F NMR (376 MHz, Chloroform-d) δ = -112.22, -113.35. IR (KBr): 3057.01, 2917.62, 1609.67, 1584.94, 1486.34, 1447.49, 1437.53, 1253.42, 1173.94, 1118.12, 962.62, 780.96, 718.85, 699.85 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{27}H_{22}F_2OP$ 431.1376, found 431.1374.

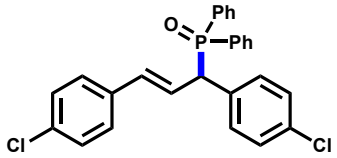
(E)-(1,3-bis(4-fluorophenyl)allyl)diphenylphosphine oxide(3j)



3j was known compounds[3, 4, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3j** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (83 mg, 96% yield), Mp: 167-168 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 – 7.84 (m, 2H), 7.60 – 7.55 (m, 2H), 7.51 – 7.44 (m, 3H), 7.41 – 7.29 (m, 5H), 7.17 – 7.14 (m, 2H), 6.91 (ddd, *J* = 8.7, 5.1, 2.8 Hz, 4H), 6.45 (ddd, *J* = 15.8, 8.8, 7.0 Hz, 1H), 6.27 (dd, *J* = 15.9, 3.7 Hz, 1H), 4.36 (t, *J* = 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.4 (d, $J_{C\text{-}F}$ = 248.5 Hz), 162.0 (d, $J_{C\text{-}F}$ = 246.4 Hz), 133.4 (d, $J_{C\text{-}P}$ = 11.2 Hz), 132.8, 132.0 (d, $J_{C\text{-}P}$ = 22.2 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.3 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.4

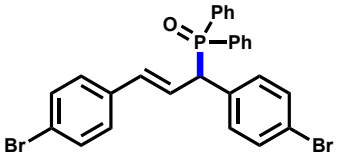
Hz), 131.7, 131.6, 131.3, 131.2, 131.1, 131.1, 131.0, 130.9, 128.7, 128.6, 128.5, 128.4, 128.0, 128.0, 124.2 (d, J_{C-P} = 6.8 Hz), 115.7, 115.6, 115.5, 115.4, 51.3 (d, J_{C-P} = 65.1 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.12. ^{19}F NMR (376 MHz, Chloroform-d) δ = -113.93, -114.95. IR (KBr): 3056.68, 2907.52, 1600.28, 1508.36, 1437.36, 1244.90, 1184.16, 1159.33, 1117.74, 1101.40, 968.44, 843.97, 819.72, 767.64, 734.17, 705.12 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{27}H_{22}F_2OP$ 431.1376, found 431.1376.

(E)-(1,3-bis(4-chlorophenyl)allyl)diphenylphosphine oxide (3k)



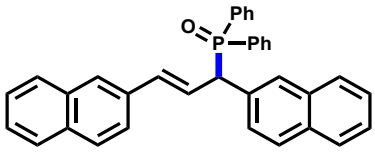
3k was known compounds[4, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3k** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (83 mg, 90% yield), Mp: 188-189 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.80 (m, 2H), 7.61 – 7.54 (m, 2H), 7.53 – 7.41 (m, 4H), 7.36 – 7.32 (m, 6H), 7.25 – 7.22 (m, 2H), 7.05 (d, *J* = 8.5Hz, 2H), 6.48 (ddd, *J* = 15.8, 8.9, 6.9 Hz, 1H), 6.20 (dd, *J* = 15.8, 3.8 Hz, 1H), 4.29 (t, *J* = 9.4 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.0, 134.4 (d, J_{C-P} = 6.3 Hz), 133.6, 133.5, 133.5, 133.3, 132.1, 131.9, 131.7, 131.6, 131.6 (d, J_{C-P} = 82.8 Hz),131.3, 131.2, 130.8, 130.8, 128.9, 128.7, 128.6, 128.5, 128.4, 127.7, 124.9 (d, J_{C-P} = 7.0 Hz), 51.6 (d, J_{C-P} = 64.6 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.86. IR (KBr): 3055.07, 2915.95, 1489.64, 1437.48, 1186.91, 1117.63, 1092.65, 1014.18, 966.73, 821.86, 797.78, 723.40, 702.49 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{27}H_{22}Cl_2OP$ 463.0785, found 463.0788.

(E)-(1,3-bis(4-bromophenyl)allyl)diphenylphosphine oxide (3l)



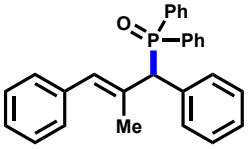
3l was known compounds[3, 4, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3l** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (104 mg, 94% yield), Mp: 203-205 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 – 7.80 (m, 2H), 7.62 – 7.55 (m, 2H), 7.54 – 7.40 (m, 4H), 7.39 – 7.30 (m, 6H), 7.24 (dd, *J* = 8.4, 1.7 Hz, 2H), 7.05 (d, *J* = 8.2 Hz, 2H), 6.49 (ddd, *J* = 16.0, 9.0, 7.0 Hz, 1H), 6.21 (dd, *J* = 15.8, 3.8 Hz, 1H), 4.30 (t, *J* = 9.2 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.4, 134.9 (d, J_{C-P} = 6.3 Hz), 133.6, 133.5, 132.1, 131.9, 131.8, 131.7, 131.6, 131.3, 131.2, 131.2, 131.1, 130.8 (d, J_{C-P} = 14.1 Hz), 128.7, 128.6, 128.6, 128.4, 128.0, 124.9 (d, J_{C-P} = 7.3 Hz), 121.7, 121.4, 51.7 (d, J_{C-P} = 64.6 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.73. IR (KBr): 3054.04, 2912.51, 1647.38, 1486.10, 1436.71, 1187.88, 1170.30, 1118.30, 1102.70, 1109.91, 971.13, 815.86, 793.13, 748.85, 721.72, 692.53 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{27}H_{22}Br_2OP$ 550.9775, found 550.9775.

(E)-(1,3-di(naphthalen-2-yl)allyl)diphenylphosphine oxide (3m)



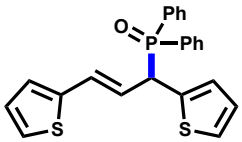
3m was known compounds[4, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3m** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (64 mg, 65% yield), Mp: 235-236 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.85 (m, 3H), 7.78 – 7.67 (m, 6H), 7.67 – 7.60 (m, 2H), 7.57 – 7.52 (m, 2H), 7.52 – 7.38 (m, 8H), 7.38 – 7.32 (m, 1H), 7.31 – 7.23 (m, 2H), 6.79 (ddd, *J* = 16.0, 9.1, 7.0 Hz, 1H), 6.48 (dd, *J* = 15.8, 3.8 Hz, 1H), 4.59 (t, *J* = 9.4 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 134.7 (d, $J_{C\text{-}P}$ = 11.4 Hz), 134.2, 133.6 (d, $J_{C\text{-}P}$ = 6.4 Hz), 133.5, 133.0, 132.5, 132.2 (d, $J_{C\text{-}P}$ = 7.1 Hz), 132.00, 1319, 131.8, 131.7, 131.5, 131.4, 131.2 (d, $J_{C\text{-}P}$ = 7.0 Hz), 128.7, 128.6, 128.6, 128.4, 128.4, 128.3, 128.2, 128.0, 127.7, 127.6 (d, $J_{C\text{-}P}$ = 4.9 Hz), 126.4, 126.3, 126.2, 126.0, 126.0, 125.0 (d, $J_{C\text{-}P}$ = 7.3 Hz), 123.7, 52.6 (d, $J_{C\text{-}P}$ = 65.3 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.07. IR (KBr): 3005.79, 2989.41, 1596.64, 1437.01, 1275.39, 1260.44, 1180.15, 1116.76, 961.42, 841.29, 764.18, 749.63, 697.48 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{35}H_{28}OP$ 495.1878, found 495.1873.

(E)-(2-methyl-1,3-diphenylallyl)diphenylphosphine oxide (3n)



3n was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3n** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (53 mg, 65% yield), Mp: 227-228 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.90 – 7.83 (m, 2H), 7.64 – 7.55 (m, 2H), 7.55 – 7.42 (m, 5H), 7.41 – 7.34 (m, 1H), 7.33 – 7.27 (m, 2H), 7.26 – 7.18 (m, 5H), 7.14 (t, *J* = 7.3 Hz, 1H), 6.95 (d, *J* = 7.3 Hz, 2H), 6.69 (s, 1H), 4.23 (d, *J* = 8.8 Hz, 1H), 1.90 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.8, 135.7 (d, $J_{C\text{-}P}$ = 4.8 Hz), 134.6 (d, $J_{C\text{-}P}$ = 6.1 Hz), 133.4 (d, $J_{C\text{-}P}$ = 26.3 Hz), 132.5, 131.6, 131.4, 131.3, 131.3, 131.2, 130.9, 130.8, 130.3, 130.2, 128.9, 128.5, 128.5, 128.4, 128.3, 128.0, 127.2, 126.4, 56.5 (d, $J_{C\text{-}P}$ = 66.7 Hz), 18.2 (d, $J_{C\text{-}P}$ = 4.9 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.31. IR (KBr): 3055.39, 3023.91, 2921.66, 1598.29, 1491.48, 1437.24, 1174.73, 1116.82, 1071.44, 920.47, 720.40, 699.48 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{28}H_{26}OP$ 409.1721, found 409.1725.

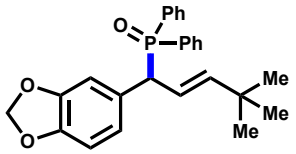
(E)-(1,3-di(thiophen-2-yl)allyl)diphenylphosphine oxide (3o)



3o was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3o** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (76 mg, 94% yield), Mp: 192-193 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 – 7.76 (m, 2H), 7.73 – 7.66 (m, 2H), 7.54 – 7.41 (m, 4H), 7.40 – 7.33 (m, 2H), 7.11 (dd, *J* = 13.4, 5.1 Hz, 2H), 7.04 (t, *J* = 2.6 Hz, 1H), 6.88 (ddd, *J* = 6.1, 5.2, 3.6 Hz, 2H), 6.80 (d, *J* = 3.5 Hz, 1H), 6.48 (dd, *J* = 15.6, 3.8 Hz, 1H), 6.27 (ddd, *J* = 15.4, 8.6, 6.4 Hz, 1H), 4.67 (dd, *J* = 12.3, 8.6 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.5 (d, $J_{C\text{-}P}$ = 3.0 Hz), 136.9 (d, $J_{C\text{-}P}$ = 5.9 Hz), 132.1 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.9 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.9, 131.8, 131.6, 131.5, 130.8 (d, $J_{C\text{-}P}$ = 30.1 Hz), 128.7, 128.6, 128.5, 128.4, 127.8 (d, $J_{C\text{-}P}$ = 10.8 Hz), 127.4, 127.3 (d, $J_{C\text{-}P}$ = 5.9 Hz), 127.2 (d, $J_{C\text{-}P}$ = 2.4 Hz), 126.0, 125.1 (d, $J_{C\text{-}P}$ = 2.7 Hz), 124.7, 123.5 (d, $J_{C\text{-}P}$ = 6.7 Hz), 47.2 (d, $J_{C\text{-}P}$ = 66.3 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.10. IR (KBr): 3054.71, 2909.16, 1486.96, 1275.02, 1260.85, 1179.22, 1118.22, 953.99, 763.24, 749.15,

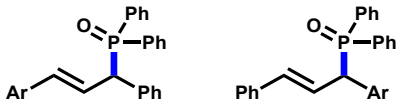
721.37, 694.51 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{23}H_{20}OPS_2$ 407.0693, found 407.0695.

(E)-(1-(benzo[d][1,3]dioxol-5-yl)-4,4-dimethylpent-1-en-3-yl)diphenylphosphine oxide (3p)



3p was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3p** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (59 mg, 71% yield), Mp: 248-251 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.77 (m, 2H), 7.66 – 7.57 (m, 2H), 7.54 – 7.38 (m, 4H), 7.38 – 7.30 (m, 2H), 6.90, 6.75 – 6.59 (m, 1H), 5.90, 5.70 – 5.59 (m, 2H), 5.34 (dd, *J* = 15.5, 3.9 Hz, 2H), 4.04 (t, *J* = 9.3 Hz, 1H), 0.78 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 147.6, 147.0, 146.9, 146.6, 132.4 (d, $J_{C\text{-}P}$ = 43.8 Hz), 131.9, 131.8, 131.7, 131.6, 131.4, 131.3, 130.1 (d, $J_{C\text{-}P}$ = 6.0 Hz), 128.4, 128.2, 122.6 (d, $J_{C\text{-}P}$ = 6.8 Hz), 119.4 (d, $J_{C\text{-}P}$ = 6.6 Hz), 109.8 (d, $J_{C\text{-}P}$ = 5.5 Hz), 108.3, 101.0, 51.8 (d, $J_{C\text{-}P}$ = 65.6 Hz), 33.3, 29.2. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.44. IR (KBr): 3054.84, 2956.76, 1501.97, 1501.97, 1486.64, 1437.90, 1363.27, 1249.76, 1175.66, 1116.40, 1039.79, 970.82, 931.36, 718.70, 698.45 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{26}H_{28}O_3P$ 419.1776, found 419.1774.

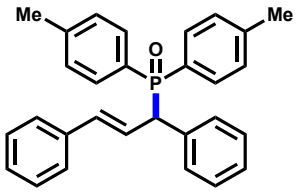
(E)-(3-(4-bromophenyl)-1-phenylallyl)diphenylphosphine oxide (3q) and (E)-(1-(4-bromophenyl)-3-phenylallyl)diphenylphosphine oxide (3q')



(Ar=4-BrC_6H_4) 3q:3q'=1:1

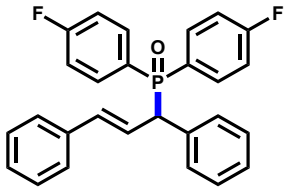
3q and **3q'** was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3q** and **3q'** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (62 mg, 65% yield), Mp: 178-179 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*, **3q**+**3q'**) δ 7.88 – 7.81 (m, 4H), 7.63 – 7.53 (m, 4H), 7.51 – 7.42 (m, 6H), 7.42 – 7.28 (m, 12H), 7.25 – 7.14 (m, 10H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.63 – 6.46 (m, 2H), 6.30 (dd, *J* = 15.8, 3.7 Hz, 1H), 6.23 (dd, *J* = 15.8, 3.8 Hz, 1H), 4.36 (t, *J* = 9.3 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*, 3q+3q') δ 136.6 (d, $J_{C\text{-}P}$ = 2.4 Hz), 135.7 (d, $J_{C\text{-}P}$ = 5.8 Hz), 135.7 (d, $J_{C\text{-}P}$ = 2.4 Hz), 135.2 (d, $J_{C\text{-}P}$ = 5.8 Hz), 134.9, 134.8, 133.3, 133.2, 132.0 (d, $J_{C\text{-}P}$ = 3.0 Hz), 132.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.8 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.8, 131.8, 131.8, 131.8, 131.7, 131.7, 131.6, 131.4, 131.4, 131.3, 131.3, 131.2, 131.2, 129.6, 129.5, 128.7, 128.7, 128.69, 128.6, 128.6, 128.5, 128.4, 128.4, 128.3, 128.0, 128.0, 127.9, 127.3 (d, $J_{C\text{-}P}$ = 2.3 Hz), 126.5 (d, $J_{C\text{-}P}$ = 1.7 Hz), 125.6 (d, $J_{C\text{-}P}$ = 7.3 Hz), 124.1 (d, $J_{C\text{-}P}$ = 7.1 Hz), 121.5 (d, $J_{C\text{-}P}$ = 1.2 Hz), 121.3 (d, $J_{C\text{-}P}$ = 2.9 Hz), 52.4 (d, $J_{C\text{-}P}$ = 64.7 Hz), 51.7 (d, $J_{C\text{-}P}$ = 64.8 Hz). ^{31}P NMR (162 MHz, Chloroform-d, 3q+3q') δ = 32.06, 31.66. ^{1}H NMR (401 MHz, Chloroform-*d*) δ 7.88 – 7.81 (m, 2H), 7.63 – 7.56 (m, 2H), 7.54 – 7.38 (m, 4H), 7.37 – 7.30 (m, 4H), 7.25 – 7.15 (m, 7H), 6.51 (ddd, *J* = 16.0, 9.0, 7.1 Hz, 1H), 6.30 (dd, *J* = 15.8, 3.7 Hz, 1H), 4.35 (d, *J* = 9.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 136.5 (d, $J_{C\text{-}P}$ = 2.3 Hz), 135.2 (d, $J_{C\text{-}P}$ = 6.2 Hz), 134.9, 134.8, 132.1, 132.0, 131.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 131.8, 131.7, 131.7, 131.4, 131.3, 131.2, 131.1, 130.9 (d, $J_{C\text{-}P}$ = 30.3 Hz), 128.7, 128.6, 128.5, 127.9, 126.5, 126.5, 124.0 (d, $J_{C\text{-}P}$ = 7.2 Hz), 121.3, 51.7 (d, $J_{C\text{-}P}$ = 64.6 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.68. IR (KBr): 3056.34, 3025.71, 2920.53, 2850.08, 1484.22, 1437.12, 1169.07, 1117.96, 1011.21, 965.62, 825.08, 721.51, 692.18 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{27}H_{23}BrOP$ 473.0670, found 473.0680.

(E)-(1,3-diphenylallyl)di-p-tolylphosphine oxide (3t)



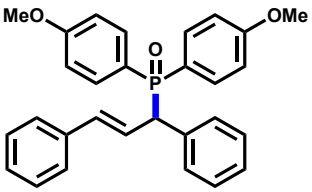
3t was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3t** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (61 mg, 72% yield), Mp: 210-211 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (dd, *J* = 10.6, 8.1 Hz, 2H), 7.44 (dd, *J* = 10.9, 8.1 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.27 – 7.19 (m, 8H), 7.19 – 7.13 (m, 2H), 7.09 (dd, *J* = 7.9, 2.8 Hz, 2H), 6.59 (ddd, *J* = 16.0, 9.1, 7.0 Hz, 1H), 6.33 (dd, *J* = 15.7, 3.7 Hz, 1H), 4.34 (t, *J* = 9.6 Hz, 1H), 2.36 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 142.2 (d, $J_{C\text{-}P}$ = 2.5 Hz), 141.9 (d, $J_{C\text{-}P}$ = 2.5 Hz), 136.9 (d, $J_{C\text{-}P}$ = 1.9 Hz), 136.3 (d, $J_{C\text{-}P}$ = 5.8 Hz), 134.3 (d, $J_{C\text{-}P}$ = 11.4 Hz), 131.8, 131.8, 131.5, 131.4, 129.6, 129.6, 129.3, 129.2, 129.1, 128.9, 128.6, 128.5, 128.1 (d, $J_{C\text{-}P}$ = 39.1 Hz), 127.6, 127.1, 126.5, 125.1 (d, $J_{C\text{-}P}$ = 7.0 Hz), 52.5 (d, $J_{C\text{-}P}$ = 65.1 Hz), 21.7, 21.6. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.41. IR (KBr): 3023.73, 2918.33, 1600.14, 1439.64, 1452.82, 1275.46, 1260.49, 1171.46, 1115.48, 964.99. 764.24, 749.21, 700.54, 660.11 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{29}H_{28}OP$ 423.1878, found 423.1873.

(E)-(1,3-diphenylallyl)bis(4-fluorophenyl)phosphine oxide (3u)



3u was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3u** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (70 mg, 81% yield), Mp: 195-197 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.78 (m, 2H), 7.54 (ddd, *J* = 10.4, 8.3, 5.5 Hz, 2H), 7.33 (d, *J* = 7.5 Hz, 2H), 7.24 – 7.12 (m, 10H), 7.03 – 6.96 (m, 2H), 6.57 (ddd, *J* = 16.1, 9.1, 7.0 Hz, 1H), 6.35 (dd, *J* = 15.9, 3.6 Hz, 1H), 4.32 (t, *J* = 9.3 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-d) δ 166.2 (d, $J_{C\text{-}P}$ = 22.5 Hz), 163.7 (d, $J_{C\text{-}P}$ = 23.8 Hz), 136.5, 135.6 (d, $J_{C\text{-}P}$ = 6.0 Hz), 134.9, 134.8, 134.0 (dd, $J_{C\text{-}F,C\text{-}P}$ = 39.2, 18.4 Hz), 134.0 (d, $J_{C\text{-}P}$ = 39.1 Hz), 129.5 (d, $J_{C\text{-}P}$ = 5.3 Hz), 128.8, 128.6, 127.9, 127.5, 126.5, 124.1 (d, $J_{C\text{-}P}$ = 7.3 Hz), 116.1 (dd, $J_{C\text{-}F,C\text{-}P}$ = 19.4, 10.3 Hz), 115.8 (dd, $J_{C\text{-}F,C\text{-}P}$ = 19.2, 10.4 Hz), 52.7 (d, $J_{C\text{-}P}$ = 66.3 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 31.13. ^{19}F NMR (376 MHz, Chloroform-d) δ = -106.32, -106.56. IR (KBr): 3060.41, 3029.41, 1593.09, 1498.76, 1453.17, 1397.47, 1234.80, 1187.25, 1161.69, 1115.85, 1094.42, 965.78, 829.92, 747.26, 699.42, 663.57 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{27}H_{22}F_2OP$ 431.1376, found 431.1379.

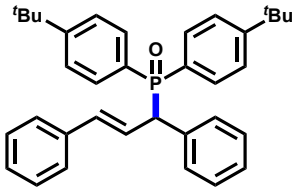
(E)-(1,3-diphenylallyl)bis(4-methoxyphenyl)phosphine oxide (3v)



3v was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 120 ºC (oil bath) for 2h. **3v** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (64 mg, 70% yield), Mp: 224-225 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 (dd, *J* = 10.2,

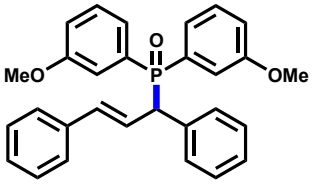
8.8 Hz, 2H), 7.46 (dd, *J* = 10.5, 8.8 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.27 – 7.14 (m, 8H), 6.95 (dd, *J* = 8.8, 2.2 Hz, 2H), 6.80 (dd, *J* = 8.8, 2.2 Hz, 2H), 6.59 (ddd, *J* = 16.0, 9.1, 7.1 Hz, 1H), 6.32 (dd, *J* = 15.7, 3.8 Hz, 1H), 4.29 (t, *J* = 9.8 Hz, 1H), 3.81 (s, 3H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.3 (d, J_{C-P} = 2.8 Hz), 162.1 (d, J_{C-P} = 2.9 Hz), 136.9, 136.4 (d, J_{C-P} = 5.9 Hz), 134.3, 134.1, 133.7, 133.6, 133.3, 133.2, 129.6, 129.5, 128.6, 128.5, 127.6, 127.1, 126.5, 125.1 (d, J_{C-P} = 6.9 Hz), 123.9, 123.3, 122.9, 122.3, 114.1, 114.0, 113.9, 113.7, 55.4, 55.3, 53.0 (d, J_{C-P} = 65.9 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.16. IR (KBr): 3025.28, 2907.17, 1596.63, 1570.61, 1501.92, 1453.00, 1292.63, 1253.68, 1178.25, 1166.68, 1119.85, 1100.45, 1025.92, 970.21, 825.83, 802.04, 746.68, 696.81 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{29}H_{28}O_3P$ 455.1776, found 455.1773.

(E)-bis(4-(tert-butyl)phenyl)(1,3-diphenylallyl)phosphine oxide (3w)



3w was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3w** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (92 mg, 91% yield), Mp: 248-251 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.77 – 7.70 (m, 2H), 7.52 – 7.44 (m, 4H), 7.35 – 7.29 (m, 4H), 7.24 – 7.16 (m, 8H), 6.57 (ddd, *J* = 16.0, 9.1, 7.0 Hz, 1H), 6.27 (dd, *J* = 15.8, 3.7 Hz, 1H), 4.33 (t, *J* = 9.7 Hz, 1H), 1.31 (s, 9H), 1.25 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.1 (d, J_{C-P} = 2.5 Hz), 154.9 (d, J_{C-P} = 2.5 Hz), 137.0, 136.4 (d, J_{C-P} = 5.9 Hz), 134.3, 134.2, 131.8, 131.7, 131.4, 131.3, 129.6, 129.6, 129.1 (d, J_{C-P} = 35.7 Hz), 128.5, 128.5, 128.1 (d, J_{C-P} = 35.9 Hz), 127.6, 127.0, 126.5, 125.5, 125.4, 125.3, 125.2, 52.7 (d, J_{C-P} = 64.7 Hz), 35.0, 35.0, 31.2, 31.1. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.35. IR (KBr): 3060.08, 3026.50, 2964.24, 2904.71, 2868.32, 1599.07, 1493.86, 1454.41, 1392.95, 1363.00, 1267.38, 1185.22, 1092.89, 1017.79, 968.89, 825.80, 777.20, 747.32, 699.20 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{35}H_{40}OP$ 507.2817, found 507.2820.

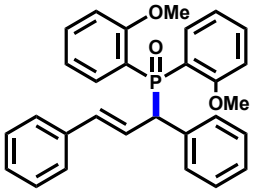
(E)-(1,3-diphenylallyl)bis(3-methoxyphenyl)phosphine oxide (3x)



3x was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3x** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (65 mg, 71% yield), Mp: 164-165 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (dd, *J* = 12.2, 2.5 Hz, 1H), 7.39 – 7.35 (m, 4H), 7.24 – 7.21 (m, 6H), 7.21 – 7.13 (m, 3H), 7.13 – 7.04 (m, 2H), 7.02 (dt, *J* = 6.0, 2.6 Hz, 1H), 6.93 – 6.88 (m, 1H), 6.60 (ddd, *J* = 16.0, 9.1, 7.2 Hz, 1H), 6.34 (dd, *J* = 15.7, 3.8 Hz, 1H), 4.34 (t, *J* = 9.4 Hz, 1H), 3.73 (s, 3H), 3.63 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.6 (d, J_{C-P} = 14.1 Hz), 159.3 (d, J_{C-P} = 14.3 Hz), 136.8 (d, J_{C-P} = 2.4 Hz), 136.1 (d, J_{C-P} = 5.8 Hz), 134.5 (d, J_{C-P} = 11.4 Hz), 133.4 (d, J_{C-P} = 36.0 Hz), 132.5 (d, J_{C-P} = 36.2 Hz), 129.7, 129.6, 129.6, 129.5, 129.3, 128.7, 128.5, 127.7, 127.2, 126.5, 124.7 (d, J_{C-P} = 7.1 Hz), 123.7 (d, J_{C-P} = 9.2 Hz), 123.4 (d, J_{C-P} = 8.9 Hz), 118.4, 118.3 (d, J_{C-P} = 2.9 Hz), 116.7 (d, J_{C-P} = 9.3 Hz), 116.1 (d, J_{C-P} = 9.0 Hz), 55.5, 55.4, 52.5 (d, J_{C-P} = 64.9 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.51. IR (KBr):3059.27,

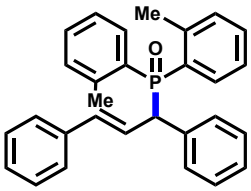
3025.72, 2937.56, 2835.31, 1589.88, 1576.55, 1481.81, 1421.49, 1286.54, 1252.18, 1184.24, 1112.21, 1043.81, 965.92, 776.33, 747.80, 696.46 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{29}H_{28}O_3P$ 455.1776, found 455.1775.

(E)-(1,3-diphenylallyl)bis(2-methoxyphenyl)phosphine oxide (3y)



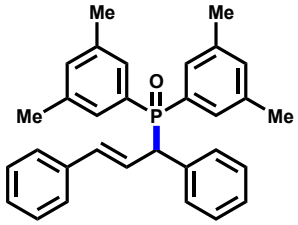
3y was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3y** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (72 mg, 79% yield), Mp: 181-182 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (ddd, *J* = 12.8, 7.6, 1.5 Hz, 1H), 7.62 (ddd, *J* = 13.1, 7.6, 1.6 Hz, 1H), 7.45 (d, *J* = 7.7 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 1H), 7.29 (t, *J* = 7.8 Hz, 1H), 7.23 (dd, *J* = 11.5, 7.3 Hz, 4H), 7.18 (d, *J* = 3.3 Hz, 1H), 7.16 – 7.12 (m, 2H), 7.07 (t, *J* = 7.3 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.88 – 6.73 (m, 3H), 6.73 – 6.66 (m, 1H), 6.46 (dd, *J* = 15.8, 3.5 Hz,1H), 4.97 (dd, *J* = 12.4, 9.2 Hz, 1H), 3.69 (s, 3H), 3.62 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.3 (d, J_{C-P} = 3.5 Hz), 159.9 (d, J_{C-P} = 3.8 Hz), 138.0 (d, J_{C-P} = 5.9 Hz), 137.4, 134.6, 134.5, 134.3, 134.2, 133.3, 133.2, 132.9, 132.7, 129.5, 129.4, 128.4, 128.2, 127.3, 127.2, 127.1, 126.7, 126.4, 121.8 (d, J_{C-P} = 14.4 Hz), 120.7 (d, J_{C-P} = 10.5 Hz), 120.5 (d, J_{C-P} = 11.1 Hz), 111.0 (d, J_{C-P} = 6.7 Hz), 110.5 (d, J_{C-P} = 6.8 Hz), 55.5, 55.3, 51.0 (d, J_{C-P} = 69.3 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 33.36. IR (KBr): 3059.39, 3024.97, 2936.64, 2835.44, 1589.12, 1576.63, 1478.99, 1430.95, 1275.65, 1251.35, 1179.29, 1162.34, 1073.01, 1021.86, 964.98, 802.37, 751.50, 697.26 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{29}H_{28}O_3P$ 455.1776, found 455.1776.

(E)-(1,3-diphenylallyl)di-o-tolylphosphine oxide (3z)



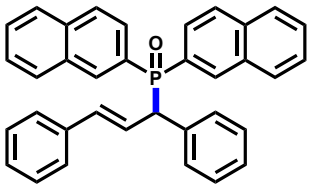
3z was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3z** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (69 mg, 82% yield), Mp: 167-169 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 (d, *J* = 11.4 Hz, 1H), 7.59 (t, *J* = 8.92 Hz, 1H), 7.42 – 7.26 (m, 6H), 7.25 – 7.19 (m, 6H), 7.20 – 7.13 (m, 4H), 6.58 (dt, *J* = 16.0, 7.8 Hz, 1H), 6.31 (dd, *J* = 15.7, 3.7 Hz, 1H), 4.35 (*t*, J = 9.5 Hz, 1H), 2.33 (s, 3H), 2.23 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.4 (d, J_{C-P} = 11.4 Hz), 138.1 (d, J_{C-P} = 11.5 Hz), 136.9, 136.2 (d, J_{C-P} = 6.2 Hz), 134.5, 134.4, 132.7, 132.6, 132.4 (d, J_{C-P} = 2.0 Hz), 132.3, 132.2, 131.9, 131.1 (d, J_{C-P} = 29.4 Hz), 129.6, 129.6, 128.6, 128.5, 128.3 (d, J_{C-P} = 8.6 Hz), 128.2 (d, J_{C-P} = 5.6 Hz), 128.0 (d, J_{C-P} = 12.1 Hz), 127.6, 127.2, 126.5, 124.9 (d, J_{C-P} = 7.3 Hz), 52.4 (d, J_{C-P} = 64.6 Hz), 21.5, 21.4. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.45. IR (KBr): 3057.53, 3025.25, 2925.79, 1595.19, 1492.93, 1451.70, 1283.46, 1180.49, 1137.13, 1083.49, 1030.14, 967.20, 805.91, 748.14, 694.01 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{29}H_{28}OP$ 423.1878, found 423.1873.

(E)-bis(3,5-dimethylphenyl)(1,3-diphenylallyl)phosphine oxide (3aa)



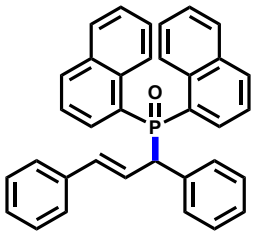
3aa was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 120 ºC (oil bath) for 2h. **3aa** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (37 mg, 41% yield), Mp: 210-212 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 11.1 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.23 – 7.11 (m, 10H), 7.09 (s, 1H), 6.97 (s, 1H), 6.57 (ddd, *J* = 15.9, 9.1, 6.9 Hz, 1H), 6.31 (dd, *J* = 15.8, 3.9 Hz, 1H), 4.35 (t, *J* = 9.5 Hz, 1H), 2.29 (s, 6H), 2.19(s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.1, 138.0, 137.9, 137.7, 137.0 (d, J_{C-P} = 2.3 Hz), 136.4 (d, J_{C-P} = 5.9 Hz), 134.4, 134.3, 133.5 (d, J_{C-P} = 2.9 Hz), 133.3 (d, J_{C-P} = 2.8 Hz), 131.9 (d, J_{C-P} = 25.0 Hz), 131.0 (d, J_{C-P} = 25.1 Hz), 129.7, 129.6, 129.5, 129.4, 129.2, 129.1, 128.5, 128.5, 127.6, 127.1, 126.5, 125.1 (d, J_{C-P} = 7.0 Hz), 52.4 (d, J_{C-P} = 64.4 Hz), 21.5, 21.3. ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.80. IR (KBr): 3055.77, 3024.09, 2914.61, 1598.33, 1488.99, 1452.91, 1417.99, 1273.05, 1168.37, 1124.81, 963.68, 868.21, 849.85, 765.94, 697.76 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{31}H_{32}OP$ 451.2191, found 451.2185.

(E)-(1,3-diphenylallyl)di(naphthalen-2-yl)phosphine oxide (3ab)



3ab was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 2h. **3ab** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as white solid (40 mg, 40% yield), Mp: 213-214 ºC. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.51 (d, *J* = 12.8 Hz, 1H), 8.21 (d, *J* = 13.0 Hz, 1H), 7.95 – 7.81 (m, 4H), 7.76 (t, *J* = 8.1 Hz, 3H), 7.63 (td, *J* = 8.7, 1.5 Hz, 1H), 7.58 – 7.39 (m, 6H), 7.23 – 7.10 (m, 8H), 6.68 (ddd, *J* = 16.0, 9.1, 7.1 Hz, 1H), 6.40 (dd, *J* = 15.7, 3.8 Hz, 1H), 4.62 (t, *J* = 9.5 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.7 (d, J_{C-P} = 1.8 Hz), 136.0 (d, J_{C-P} = 5.9 Hz), 134.7, 134.6, 134.5 (d, J_{C-P} = 1.7 Hz), 134.3, 134.2, 133.8, 133.7, 132.7 (d, J_{C-P} = 12.6 Hz), 132.5 (d, J_{C-P} = 12.6 Hz), 129.7, 129.6, 129.3, 129.1, 129.0, 128.8, 128.5, 128.3, 128.2, 128.1, 128.1, 127.9, 127.9, 127.8, 127.7, 127.3, 127.0, 126.8, 126.5, 126.4, 126.3, 126.2, 124.7 (d, J_{C-P} = 6.9 Hz), 52.4 (d, J_{C-P} = 65.1 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.32. IR (KBr): 3054.29, 3022.52, 2915.90, 1590.17, 1491.22, 1453.51, 1272.69, 1169.23, 1133.87, 1087.42, 968.81, 859.29, 819.43, 745.84, 719.60 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{35}H_{28}OP$ 495.1878, found 495.1877.

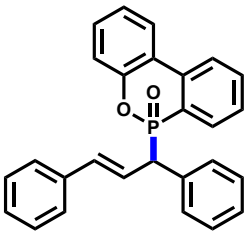
(E)-(1,3-diphenylallyl)di(naphthalen-1-yl)phosphine oxide (3ac)



3ac was known compounds[3, 5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil

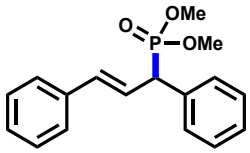
bath) for 2h. **3ac** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (44 mg, 44% yield), Mp: 235-237 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.76 (d, *J* = 8.5 Hz, 1H), 8.58 (d, *J* = 8.4 Hz, 1H), 8.13 (dd, *J* = 14.3, 7.1 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.90 – 7.78 (m, 2H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.46 (td, *J* = 7.7, 2.6 Hz, 1H), 7.42 – 7.22 (m, 7H), 7.22 – 7.01 (m, 8H), 6.78 (dt, *J* = 15.9, 8.8 Hz, 1H), 6.38 (dd, *J* = 15.9, 3.3 Hz, 1H), 4.79 (t, *J* = 9.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.9 (d, $J_{C\text{-}P}$ = 5.5 Hz), 136.8, 134.4, 134.3, 134.2, 134.0 (d, $J_{C\text{-}P}$ = 5.0 Hz), 133.9, 133.7, 133.6, 133.1 (d, $J_{C\text{-}P}$ = 2.4 Hz), 132.9 (d, $J_{C\text{-}P}$ = 2.2 Hz), 132.2, 132.1, 130.0 (d, $J_{C\text{-}P}$ = 22.8 Hz), 129.7, 129.7, 129.1 (d, $J_{C\text{-}P}$ = 23.3 Hz), 128.9, 128.7, 128.5, 128.4, 127.6, 127.3, 127.1, 126.7 (d, $J_{C\text{-}P}$ = 4.5 Hz), 126.5, 126.3, 126.2, 125.8 (d, $J_{C\text{-}P}$ = 6.5 Hz), 124.4 (d, $J_{C\text{-}P}$ = 13.7 Hz), 124.2 (d, $J_{C\text{-}P}$ = 13.7 Hz), 52.1 (d, $J_{C\text{-}P}$ = 66.7 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 36.77. IR (KBr): 3056.69, 1594.63, 1505.53, 1492.54, 1452.35, 1264.41, 1211.45, 1177.12, 1159.10, 1026.67, 986.49, 830.40, 800.29, 773.78, 747.24, 697.58 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{35}H_{28}OP$ 495.1878, found 495.1880.

6-((E)-1,3-diphenylallyl)dibenzo[c,e][1,2]oxaphosphinine 6-oxide (3ad)



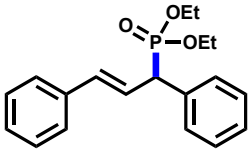
3ad was known compounds[3]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3ad** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (45 mg, 55% yield), Mp: 165-168 °C. Then the two isomer can be further separated by carefully PTLC(Petroleum ether (bp: 60-90 °C)/ethyl acetate = 2:1) for five times, one of the pure isomer was obtained in 6 mg and the NMR was checked and set as reference to assign picks of this two isomer. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.89 (m, 2H), 7.81 – 7.74 (m, 1H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.44 – 7.37 (m, 1H), 7.36 – 7.27 (m, 6H), 7.27 – 7.20 (m, 4H), 7.18 (d, *J* = 7.0 Hz, 2H), 6.98 (dt, *J* = 8.2, 1.9 Hz, 1H), 6.52 – 6.40 (m, 1H), 6.21 (dd, *J* = 15.7, 4.8 Hz, 1H), 4.01 (dd, *J* = 16.1, 9.7 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.9 (d, $J_{C\text{-}P}$ = 8.2 Hz), 136.5 (d, $J_{C\text{-}P}$ = 3.3 Hz), 136.2 (d, $J_{C\text{-}P}$ = 7.0 Hz), 135.3, 135.2, 134.3 (d, $J_{C\text{-}P}$ = 8.6 Hz), 133.6 (d, $J_{C\text{-}P}$ = 2.3 Hz), 132.5, 132.4, 130.8, 129.2, 129.1, 128.8 (d, $J_{C\text{-}P}$ = 2.4 Hz), 128.5 (d, $J_{C\text{-}P}$ = 0.9 Hz), 128.1 (d, $J_{C\text{-}P}$ = 12.3 Hz), 127.9 (d, $J_{C\text{-}P}$ = 1.0 Hz), 127.6 (d, $J_{C\text{-}P}$ = 2.9 Hz), 126.5 (d, $J_{C\text{-}P}$ = 1.9 Hz), 125.2, 124.6, 123.9, 123.7 (d, $J_{C\text{-}P}$ = 10.0 Hz), 122.7 (d, $J_{C\text{-}P}$ = 13.7 Hz), 122.5 (d, $J_{C\text{-}P}$ = 7.3 Hz), 120.7 (d, $J_{C\text{-}P}$ = 6.6 Hz), 51.7 (d, $J_{C\text{-}P}$ = 86.9 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 33.53. Another diastereoisomer: ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.83 (m, 2H), 7.59 (t, *J* = 7.8 Hz, 1H), 7.42-7.33 (m, 2H), 7.32 – 7.27 (m, 4H), 7.26 – 7.16 (m, 9H), 6.57 (ddd, *J* = 15.9, 9.0, 7.0 Hz, 1H), 6.57-6.38 (m, 1H), 4.01 (dd, *J* = 16.1, 9.7 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.9 (d, $J_{C\text{-}P}$ = 7.9 Hz), 136.6 (d, $J_{C\text{-}P}$ = 17.5 Hz), 136.2 (d, $J_{C\text{-}P}$ = 6.8 Hz), 134.7 (d, $J_{C\text{-}P}$ = 12.2 Hz), 134.2 (d, $J_{C\text{-}P}$ = 5.4 Hz), 133.6 (d, $J_{C\text{-}P}$ = 11.5 Hz), 131.7 (d, $J_{C\text{-}P}$ = 8.6 Hz), 130.8, 129.3 (d, $J_{C\text{-}P}$ = 6.3 Hz), 128.8, 128.8, 128.6 (d, $J_{C\text{-}P}$ = 5.2 Hz), 128.1 (d, $J_{C\text{-}P}$ = 12.4 Hz), 128.0, 127.7, 126.5 (d, $J_{C\text{-}P}$ = 12.8 Hz), 125.4, 124.7, 124.1, 123.8 (d, $J_{C\text{-}P}$ = 10.0 Hz), 122.9, 122.8, 122.7 (d $J_{C\text{-}P}$ = 2.0 Hz), 120.7 (d, $J_{C\text{-}P}$ = 6.6 Hz), 51.8 (d, $J_{C\text{-}P}$ = 86.8 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 34.18. IR (KBr): 3060.19, 3026. 41, 2924.92, 1594.74, 1581.87, 1493.73, 1475.21, 1447.53, 1430.59, 1275.52, 1256.69, 1236.47, 1116.99, 912.82, 749.72, 697.48 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{27}H_{22}O_2P$ 409.1357, found 409.1355.

dimethyl (E)-(1,3-diphenylallyl)phosphonate (3ae)



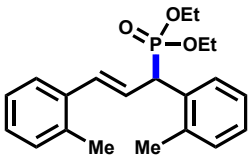
3ae was known compounds[5]**.** Following the general procedure, the reaction was stirred at 60 ºC (oil bath) for 12h. **3ae** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as yellow liquid (34 mg, 56% yield) (94% yield, in case 4.0 equiv. dimethyl phosphonate was introduced). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.43 (m, 2H), 7.40 – 7.33 (m, 4H), 7.30 (t, *J* = 7.4 Hz, 3H), 7.22 (t, *J* = 7.2 Hz, 1H), 6.64 – 6.47 (m, 2H), 4.03 (dd, *J* = 24.9, 7.9 Hz, 1H), 3.73 (d, *J* = 10.8 Hz, 3H), 3.55 (d, *J* = 10.5 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.7 (d, $J_{C\text{-}P}$ = 2.9 Hz), 135.7 (d, $J_{C\text{-}P}$ = 7.4 Hz), 134.0, 133.9, 129.1, 129.0, 128.9 (d, $J_{C\text{-}P}$ = 1.6 Hz), 128.6, 127.9, 127.5 (d, $J_{C\text{-}P}$ = 2.9 Hz), 126.6, 124.3 (d, $J_{C\text{-}P}$ = 9.7 Hz), 53.7, 53.6, 49.1 (d, $J_{C\text{-}P}$ = 137.5 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 27.74. IR (KBr): 3026.51, 2951.96, 2849.82, 1598.71, 1494.14, 1453.33, 1274.96, 1258.33, 1182.57, 1028.37, 966.21, 824.73, 764.40, 749.19, 698.20 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{17}H_{20}O_3P$ 303.1150, found 303.1148.

diethyl (E)-(1,3-diphenylallyl)phosphonate (3af)



3af was known compounds[5]**.** Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3af** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as yellow liquid (37 mg, 56% yield) (86% yield, in case 4.0 equiv. diethyl phosphonate was introduced). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.43 (m, 2H), 7.35 (q, *J* = 7.6 Hz, 4H), 7.31 – 7.23 (m, 3H), 7.21 (t, *J* = 7.3 Hz, 1H), 6.61 – 6.48 (m, 2H), 4.08 (pd, *J* = 7.0, 1.5 Hz, 2H), 4.04 – 3.90 (m, 2H), 3.86 – 3.74 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.8 (d, $J_{C\text{-}P}$ = 2.9 Hz), 136.1 (d, $J_{C\text{-}P}$ = 7.0 Hz), 133.8, 133.7, 129.2, 129.1, 128.8, 128.6, 127.8, 127.4 (d, $J_{C\text{-}P}$ = 2.9 Hz), 126.5, 124.7 (d, $J_{C\text{-}P}$ = 9.6 Hz), 62.9 (d, $J_{C\text{-}P}$ = 7.1 Hz), 62.7 (d, $J_{C\text{-}P}$ = 7.5 Hz), 49.5 (d, $J_{C\text{-}P}$ = 136.9 Hz), 16.6 (d, $J_{C\text{-}P}$ = 5.8 Hz), 16.4 (d, $J_{C\text{-}P}$ = 5.7 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 25.44. IR (KBr): 3059.36, 3027.22, 2980.70, 2929.15, 2905.49, 1598.64, 1494.34, 1452.85, 1390.39, 1247.58, 1163.17, 1051.42, 1025.90, 963.45, 794.98, 747.34, 697.74 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{19}H_{24}O_3P$ 331.1463, found 331.1457.

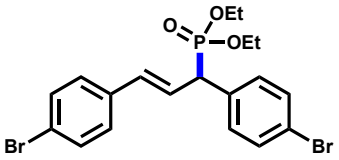
diethyl (E)-(1,3-di-o-tolylallyl)phosphonate (3ag)



Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3ag** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as yellow liquid (61 mg, 85% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (d, *J* = 7.7 Hz, 1H), 7.47 – 7.41 (m, 1H), 7.24 – 7.08 (m, 6H), 6.79 (dd, *J* = 15.6, 4.8 Hz, 1H), 6.36 (dt, *J* = 15.6, 8.5 Hz, 1H), 4.30 (dd, *J* = 25.6, 8.7 Hz, 1H), 4.11 (p, *J* = 7.1 Hz, 2H), 3.95 (dp, *J* = 10.1, 7.1 Hz, 1H), 3.84 – 3.72 (m, 1H), 2.42 (s, 3H), 2.30 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.3 (d, $J_{C\text{-}P}$ = 8.9

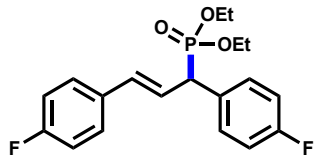
Hz), 136.1 (d, $J_{C\text{-}P}$ = 2.8 Hz), 135.4 (d, $J_{C\text{-}P}$ = 1.3 Hz), 134.4 (d, $J_{C\text{-}P}$ = 6.7 Hz), 131.7 (d, $J_{C\text{-}P}$ = 13.6 Hz), 130.7, 130.3, 128.9 (d, $J_{C\text{-}P}$ = 5.0 Hz), 127.7, 127.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 126.4 (d, $J_{C\text{-}P}$ = 2.8 Hz), 126.3 (d, $J_{C\text{-}P}$ = 9.7 Hz), 126.2, 125.9 (d, $J_{C\text{-}P}$ = 1.4 Hz), 62.9 (d, $J_{C\text{-}P}$ = 6.9 Hz), 62.6 (d, $J_{C\text{-}P}$ = 7.4 Hz), 45.0 (d, $J_{C\text{-}P}$ = 137.9 Hz), 20.1, 19.9, 16.6 (d, $J_{C\text{-}P}$ = 6.0 Hz), 16.4 (d, $J_{C\text{-}P}$ = 5.8 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 26.27. IR (KBr): 3019.79, 2979.71, 2928.74, 1490.59, 1461.36, 1390.20, 1248.76, 1163.36, 1097.05, 1050.88, 1025.85, 963.49, 751.68 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{21}H_{28}O_3P$ 359.1776, found 359.1770.

diethyl (E)-(1,3-bis(4-bromophenyl)allyl)phosphonate (3ah)



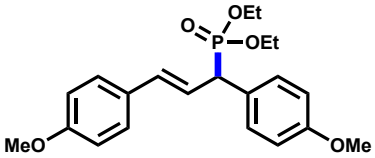
Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3ah** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as yellow liquid (70 mg, 72% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 8.5 Hz, 2H), 7.32 (dd, *J* = 8.5, 2.1 Hz, 2H), 7.22 (d, *J* = 8.4 Hz, 2H), 6.53 – 6.38 (m, 2H), 4.14 – 4.03 (m, 2H), 4.03 – 3.88 (m, 2H), 3.87 – 3.79 (m, 1H), 1.26 (t, *J* = 7.0 Hz, 3H), 1.15 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.5 (d, $J_{C\text{-}P}$ = 2.8 Hz), 134.9, 134.9, 133.0, 132.9, 131.9, 131.8, 130.8, 130.8, 128.0, 125.0, 124.9, 121.7, 121.5 (d, $J_{C\text{-}P}$ = 3.8 Hz), 63.0 (d, $J_{C\text{-}P}$ = 7.5 Hz), 62.9 (d, $J_{C\text{-}P}$ = 7.6 Hz), 48.8 (d, $J_{C\text{-}P}$ = 137.9 Hz), 16.6 (d, $J_{C\text{-}P}$ = 5.7 Hz), 16.4 (d, $J_{C\text{-}P}$ = 5.7 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 24.44. IR (KBr): 3027.76, 2980.16, 2928.69, 2905.17, 1486.28, 1391.21, 1246.63, 1163.14, 1071.31, 1050.27, 1024.57, 964.54, 795.54, 746.17 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{19}H_{22}Br_2O_3P$ 486.9673, found 486.9670.

diethyl (E)-(1,3-bis(4-fluorophenyl)allyl)phosphonate (3ai)



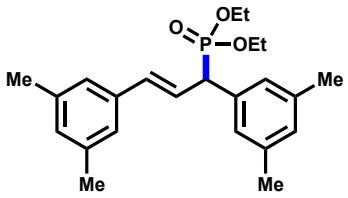
Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 14h. **3ai** was isolated by PTLC (Petroleum ether (bp: 60-90 ºC)/ethyl acetate = 1/1) as yellow liquid (48 mg, 65% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (ddd, *J* = 7.7, 5.2, 2.2 Hz, 2H), 7.34 (dd, *J* = 8.6, 5.5 Hz, 2H), 7.02 (dt, *J* = 24.2, 8.6 Hz, 4H), 6.52 (dd, *J* = 15.8, 4.1 Hz, 1H), 6.40 (dt, *J* = 15.9, 8.2 Hz, 1H), 4.15 – 4.03 (m, 2H), 4.02 – 3.90 (m, 2H), 3.89 – 3.78 (m, 1H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.5 (d, $J_{C\text{-}F}$ = 34.8 Hz), 161.1 (d, $J_{C\text{-}F}$ = 32.1 Hz), 132.8, 132.7 (d, $J_{C\text{-}P}$ = 14.1 Hz), 131.7 (dd, $J_{C\text{-}F,\ C\text{-}P}$ = 7.3, 3.0 Hz), 130.7, 130.7 (d, $J_{C\text{-}P}$ = 14.14 Hz), 128.1, 128.0, 124.2 (d, $J_{C\text{-}P}$ = 9.7 Hz), 115.8, 115.7, 115.6, 115.5, 62.9 (d, $J_{C\text{-}P}$ = 7.0 Hz), 62.8 (d, $J_{C\text{-}P}$ = 7.2 Hz), 48.5 (d, $J_{C\text{-}P}$ = 138.5 Hz), 16.5 (d, $J_{C\text{-}P}$ = 5.8 Hz), 16.4 (d, $J_{C\text{-}P}$ = 5.8 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 25.07. ^{19}F NMR (376 MHz, Chloroform-d) δ -113.96, -115.02. IR (KBr): 2982.77, 2930.15, 1601.01, 1508.56, 1227.81, 1159.10, 1097.06, 1051.31, 1025.95, 965.14, 845.81, 787.30, 748.45 cm^{-1}. HRMS (ESI/[M+H]$^+$) Calcd. for: $C_{19}H_{22}F_2O_3P$ 367.1275, found 367.1273.

diethyl (E)-(1,3-bis(4-methoxyphenyl)allyl)phosphonate (3aj)



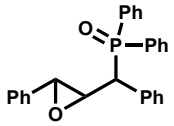
Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 12h. **3aj** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as yellow liquid (41 mg, 53% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.34 (m, 2H), 7.31 (d, *J* = 8.7 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 6.86 – 6.80 (m, 2H), 6.49 (dd, *J* = 15.8, 4.2 Hz, 1H), 6.40 – 6.31 (m, 1H), 4.14 – 4.03 (m, 2H), 4.02 – 3.85 (m, 2H), 3.85 – 3.80 (m, 1H), 3.79 (s, 3H), 3.79 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.3, 158.8 (d, $J_{C\text{-}P}$ = 2.8 Hz), 133.0, 132.9, 130.2, 130.1, 129.7 (d, $J_{C\text{-}P}$ = 2.7 Hz), 128.1, 128.1, 127.7, 122.6, 122.5, 114.2, 114.0, 62.8 (d, $J_{C\text{-}P}$ = 7.1 Hz), 62.7 (d, $J_{C\text{-}P}$ = 7.7 Hz), 55.4, 48.5 (d, $J_{C\text{-}P}$ = 137.8 Hz), 16.6 (d, $J_{C\text{-}P}$ = 5.8 Hz), 16.4 (d, $J_{C\text{-}P}$ = 5.8 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 26.01. IR (KBr): 2979.40, 2931.54, 1607.38, 1510.42, 1463.19, 1290.93, 1249.30, 1175.64, 1627.83, 963.42, 829.84, 776.03, 746.48, 697.88 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{21}H_{28}O_5P$ 391.1674, found 391.1681.

diethyl (E)-(1,3-bis(3,5-dimethylphenyl)allyl)phosphonate (3ak)



Following the general procedure, the reaction was stirred at 100 ºC (oil bath) for 14h. **3ak** was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as yellow liquid (15 mg, 20% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.05 (s, 2H), 7.00 (s, 2H), 6.89 (s, 1H),6.87 (s, 1H), 6.54 – 6.42 (m, 2H), 4.14 – 4.03 (m, 2H), 4.03 – 3.85 (m, 2H), 3.85 – 3.76 (m, 1H), 2.31 (s, 6H), 2.29 (s, 6H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.2 (d, $J_{C\text{-}P}$ = 1.7 Hz), 138.0, 136.8 (d, $J_{C\text{-}P}$ = 2.2 Hz), 135.9 (d, $J_{C\text{-}P}$ = 7.2 Hz), 133.8, 133.6, 129.4, 129.0 (d, $J_{C\text{-}P}$ = 3.2 Hz), 126.9, 126.8, 124.5, 124.4, 62.8 (d, $J_{C\text{-}P}$ = 5.5 Hz), 62.7 (d, $J_{C\text{-}P}$ = 5.4 Hz), 49.5 (d, $J_{C\text{-}P}$ = 136.8 Hz), 21.4, 21.3, 16.6 (d, $J_{C\text{-}P}$ = 5.9 Hz), 16.4 (d, $J_{C\text{-}P}$ = 5.9 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 25.80. IR (KBr): 2980.17, 2919.13, 1600.83, 1508.29, 1454.71, 1390.52, 1247.22, 1160.68, 1052.91, 1026.04, 962.62, 786.89, 748.93, 698.60 cm^{-1}. HRMS (ESI/[M+H]$^{+}$) Calcd. for: $C_{23}H_{32}O_3P$ 387.2089, found 387.2081.

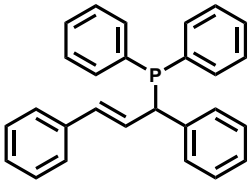
diphenyl(phenyl(3-phenyloxiran-2-yl)methyl)phosphine oxide (4)



4 was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (47 mg, 57% yield), Mp: 149-151 °C. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.84 (m, 2H), 7.63 – 7.51 (m, 5H), 7.46 (dd, *J* = 11.1, 8.3 Hz, 2H), 7.33 – 7.17 (m, 11H), 4.39 (t, *J* = 8.0 Hz, 1H), 4.23 (d, *J* = 8.0 Hz, 1H), 4.13 (d, *J* = 9.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.6, 132.7 (d, $J_{C\text{-}P}$ = 3.9 Hz), 132.2 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.7 (d, $J_{C\text{-}P}$ = 4.7 Hz), 131.6, 131.3, 131.2, 130.9, 130.8, 129.1, 129.0, 128.4, 128.3, 128.3 (d, $J_{C\text{-}P}$ = 1.4 Hz), 128.1 (d, $J_{C\text{-}P}$ = 4.2 Hz), 127.3 (d, $J_{C\text{-}P}$ = 1.9 Hz), 127.0, 74.4 (d, $J_{C\text{-}P}$ = 10.9 Hz), 74.3 (d, $J_{C\text{-}P}$ = 3.3 Hz), 46.1 (d, $J_{C\text{-}P}$ = 68.9 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 39.00. IR (KBr): 3059.55, 3029.90, 2915.81, 1708.78, 1493.89, 1437.61, 1275.42, 1260.96, 1154.22, 1117.66,

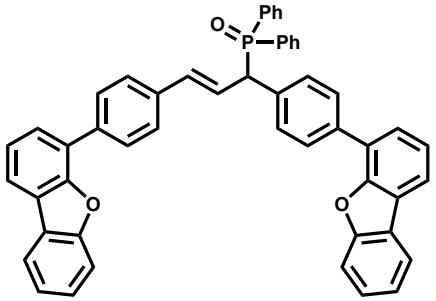
750.44, 698.80 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{27}H_{24}O_2P$ 411.1514, found 411.1515.

(E)-(1,3-diphenylallyl)diphenylphosphane (5)



5 was white solid (59 mg, 78% yield). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.49 (m, 3H), 7.29 – 7.10 (m, 17H), 6.38 (dq, *J* = 11.5, 5.9, 4.3 Hz, 1H), 6.20 (dd, *J* = 15.7, 3.3 Hz, 1H), 4.31 (dd, *J* = 8.6, 5.6 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.8 (d, J_{C-P} = 2.5 Hz), 136.0 (d, J_{C-P} = 6.1 Hz), 134.5 (d, J_{C-P} = 11.2 Hz), 131.9 (d, J_{C-P} = 2.5 Hz), 131.8, 131.8, 131.7 (d, J_{C-P} = 128.7 Hz), 131.6 (d, J_{C-P} = 2.9 Hz), 131.5, 131.4, 129.6, 129.5, 128.7, 128.6, 128.6, 128.5, 128.5, 128.3, 128.2, 127.7 (d, J_{C-P} = 0.8 Hz), 127.2 (d, J_{C-P} = 2.4 Hz), 126.5, 126.5, 124.7 (d, J_{C-P} = 7.3 Hz), 52.4 (d, J_{C-P} = 65.0 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = -0.84. IR (KBr): 3056.55, 3025.25, 2922.63, 1493.54, 1437.38, 1275.39, 1260.69, 1172.96, 1117.25, 764.20, 749.42, 721.55, 698.48 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{27}H_{24}P$ 379.1616, found 379.1610.

(E)-(1,3-bis(4-(dibenzo[b,d]furan-4-yl)phenyl)allyl)diphenylphosphine oxide (6)



6 was isolated by PTLC (Petroleum ether (bp: 60-90 °C)/ethyl acetate = 1/1) as white solid (140 mg, 96% yield), Mp: 228-230 °C. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.98 (dd, *J* = 7.7, 0.7 Hz, 2H), 7.94 – 7.89 (m, 4H), 7.84 (dd, *J* = 8.1, 1.4 Hz, 4H), 7.72 – 7.65 (m, 2H), 7.62 – 7.50 (m, 10H), 7.49 – 7.33 (m, 12H), 6.76 (ddd, *J* = 16.2, 9.2, 7.2 Hz, 1H), 6.49 (dd, *J* = 15.7, 3.7 Hz, 1H), 4.53 (t, *J* = 9.6 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.2, 153.4 (d, J_{C-P} = 1.5 Hz), 136.2 (d, J_{C-P} = 2.5 Hz), 135.8, 135.4 (d, J_{C-P} = 6.3 Hz), 135.3 (d, J_{C-P} = 2.6 Hz), 134.3 (d, J_{C-P} = 11.3 Hz), 131.9 (d, J_{C-P} = 8.5 Hz), 131.6 (d, J_{C-P} = 141.7 Hz), 131.5 (d, J_{C-P} = 8.7 Hz), 129.8 (d, J_{C-P} = 5.7 Hz), 129.0 (d, J_{C-P} = 1.7 Hz), 128.9, 128.7, 128.6, 128.5, 128.4, 127.3 (d, J_{C-P} = 1.4 Hz), 126.8 (d, J_{C-P} = 1.4 Hz), 126.7 (d, J_{C-P} = 8.7 Hz), 125.4 (d, J_{C-P} = 5.3 Hz), 125.0, 124.2, 123.3 (d, J_{C-P} = 3.4 Hz), 122.9 (d, J_{C-P} = 2.5 Hz), 120.8, 119.8 (d, J_{C-P} = 2.4 Hz), 112.0, 52.4 (d, J_{C-P} = 65.0 Hz). ^{31}P NMR (162 MHz, Chloroform-d) δ = 32.15. IR (KBr): 3054.19, 2923.67, 1512.06, 1450.72, 1438.23, 1396.11, 1275.20, 1259.77, 1190.23, 1173.12, 1117.21, 1099.51, 1028.15, 969.93, 749.77, 701.21 cm^{-1}. HRMS (ESI/$[M+H]^+$) Calcd. for: $C_{51}H_{36}O_3P$ 727.2402, found 727.2393.

5. References

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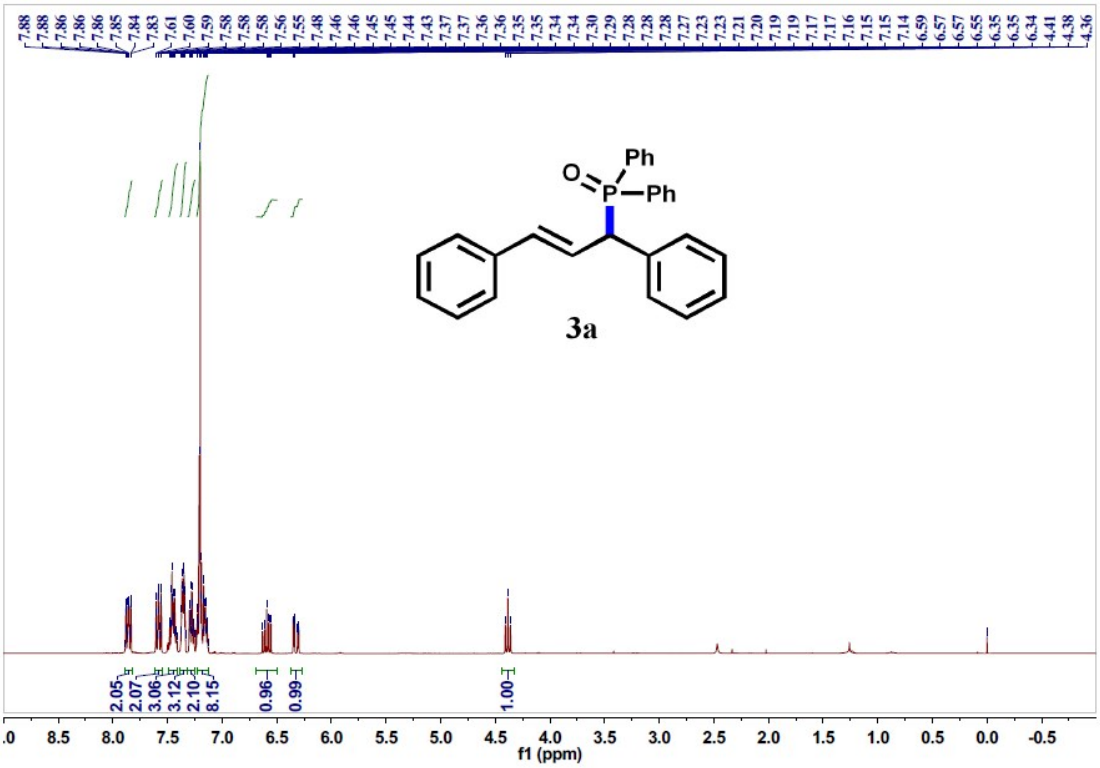
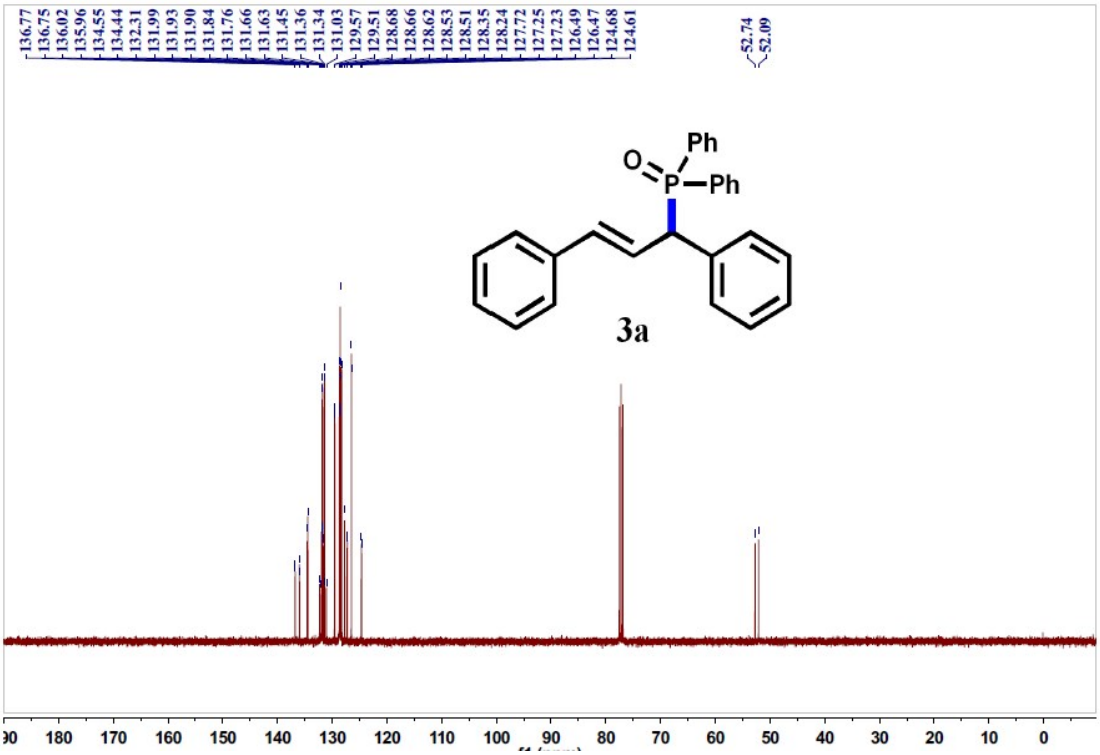
(5) Q. Chen, C.-X. Wen, X.-F. Wang, G.-D. Yu, C. OuYang, Y.-P. Huo and K. Zhang, *Adv. Synth. Catal.*, 2018, **360**, 3590-3594.
(6) P. Butti, R. Rochat, A. D. Sadow and A. Tongi, *Angew. Chem. Int. Ed.*, 2008, **47**, 4878-4881.

6. NMR spectra for new compounds

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134.44
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131.90
131.84
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131.63
131.45
131.36
131.34
131.03
129.57
129.51
128.68
128.66
128.62
128.53
128.51
128.35
128.24
127.72
127.25
127.23
126.49
126.47
124.68
124.61
52.74
52.09
Ph
O
P
Ph
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110
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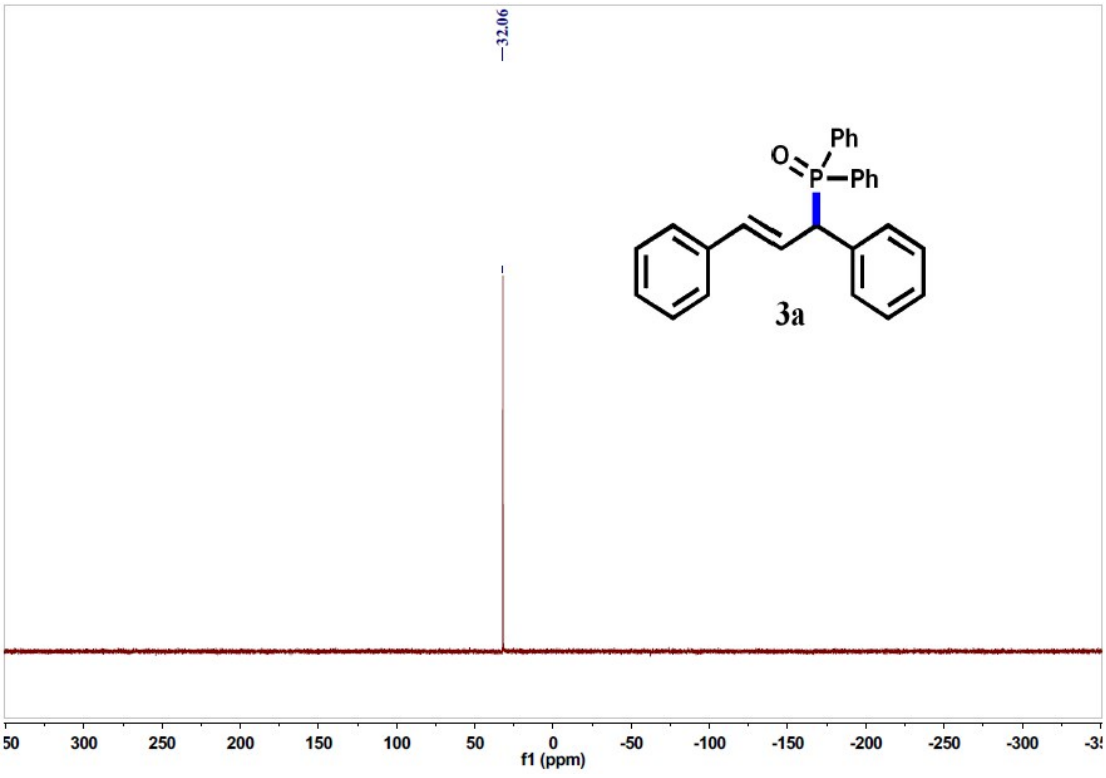
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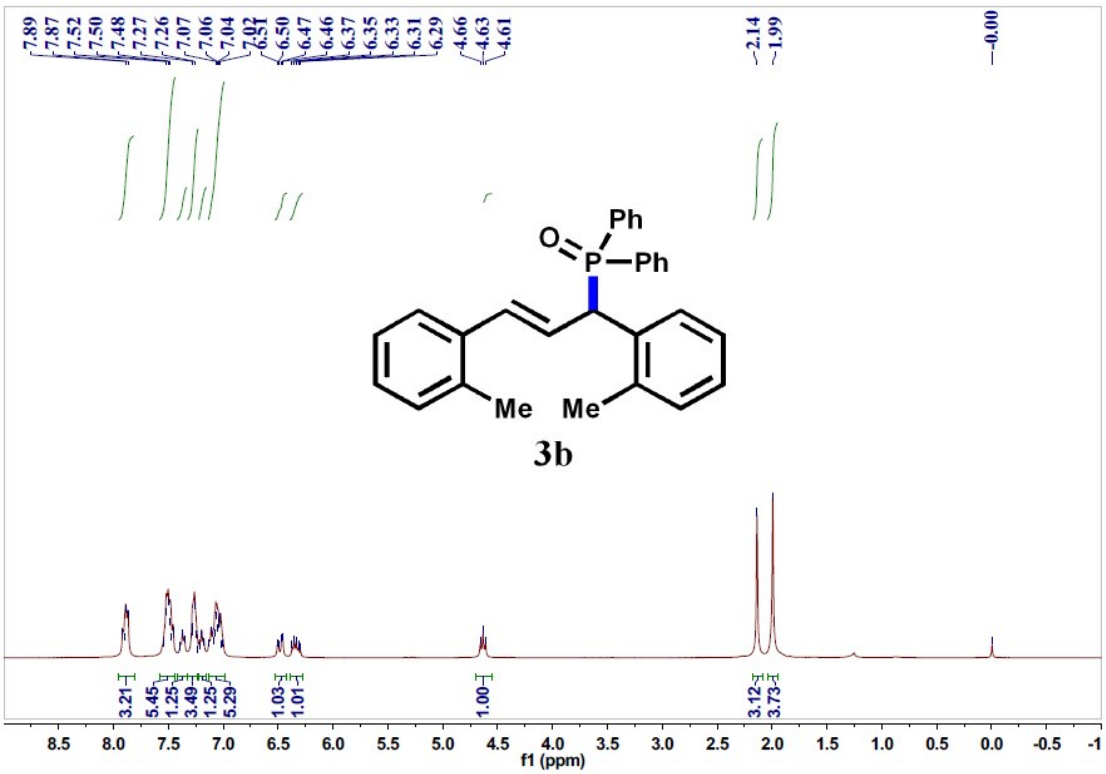
f1 (ppm)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6.37
6.35
6.33
6.31
6.29
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4.61
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O
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3b
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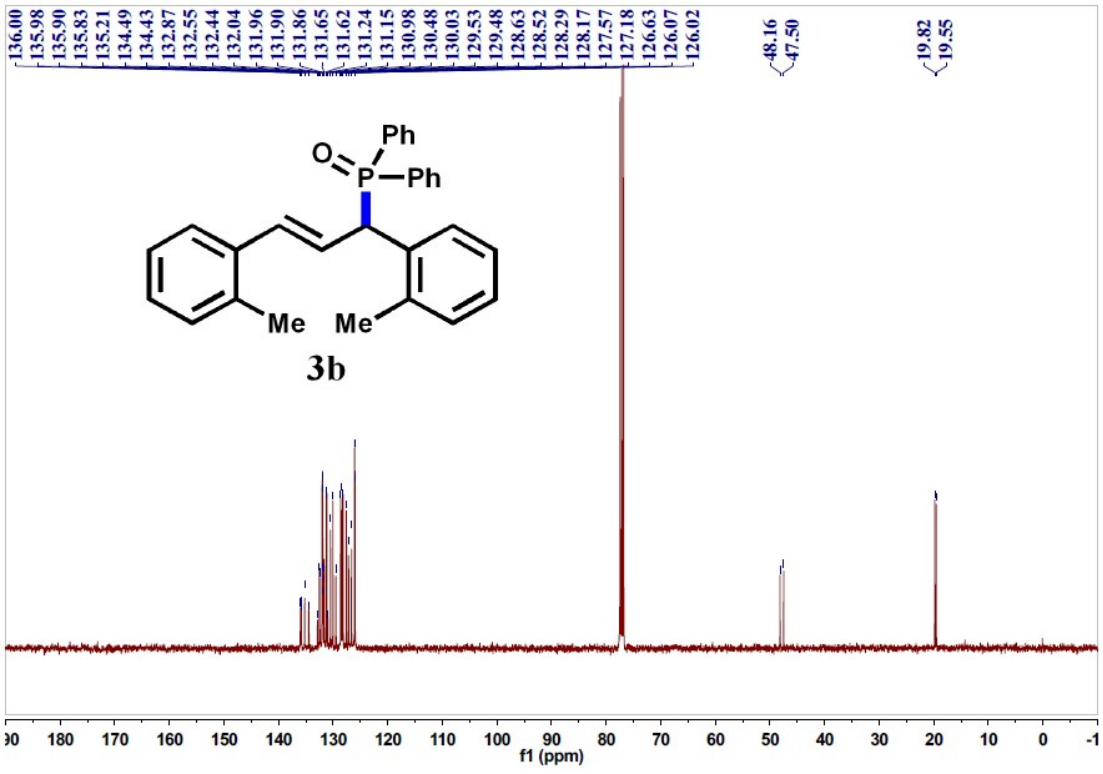
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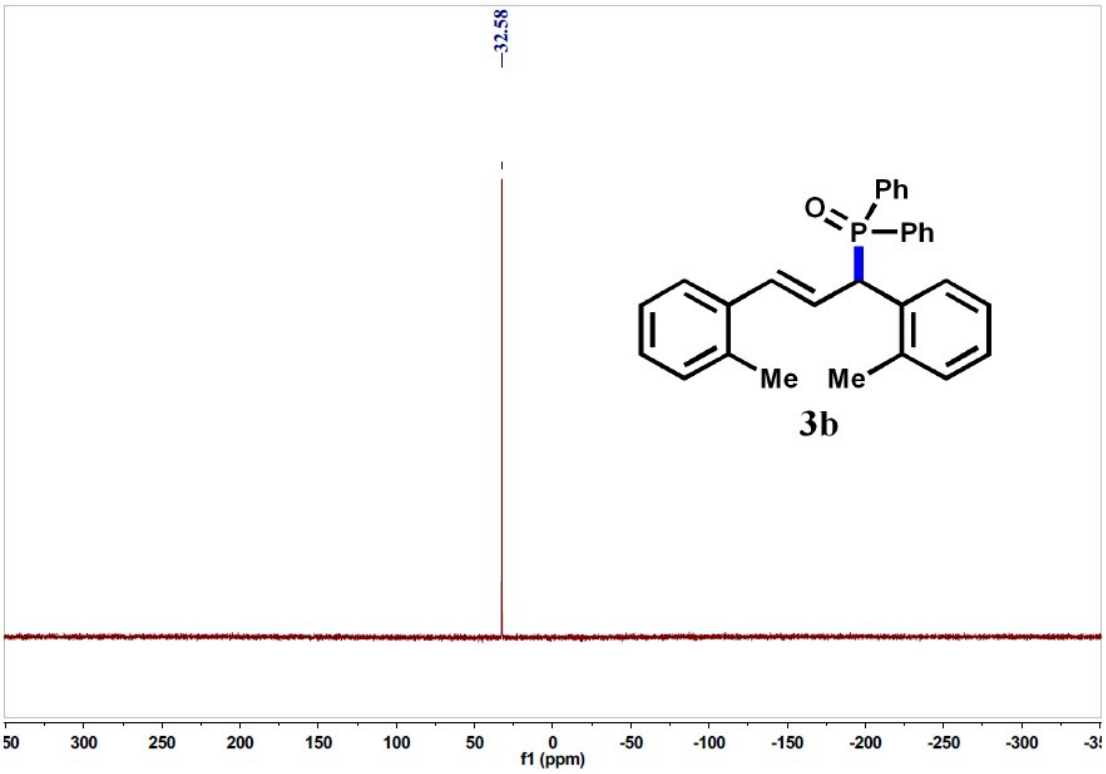
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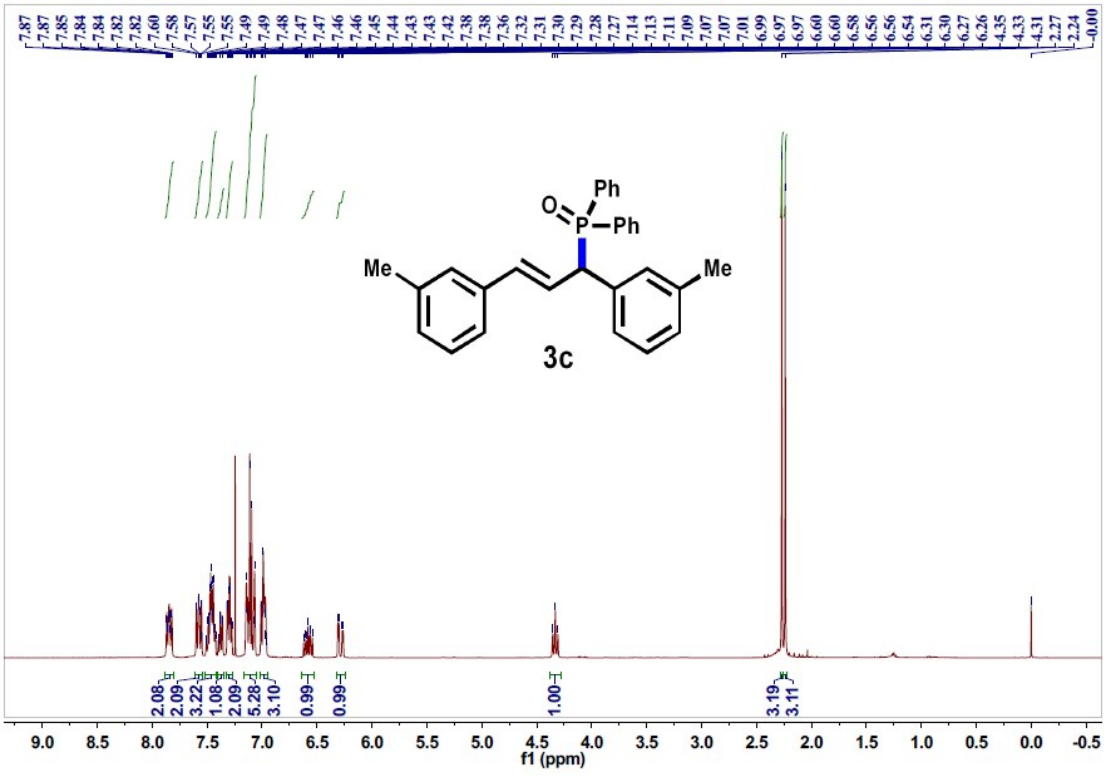
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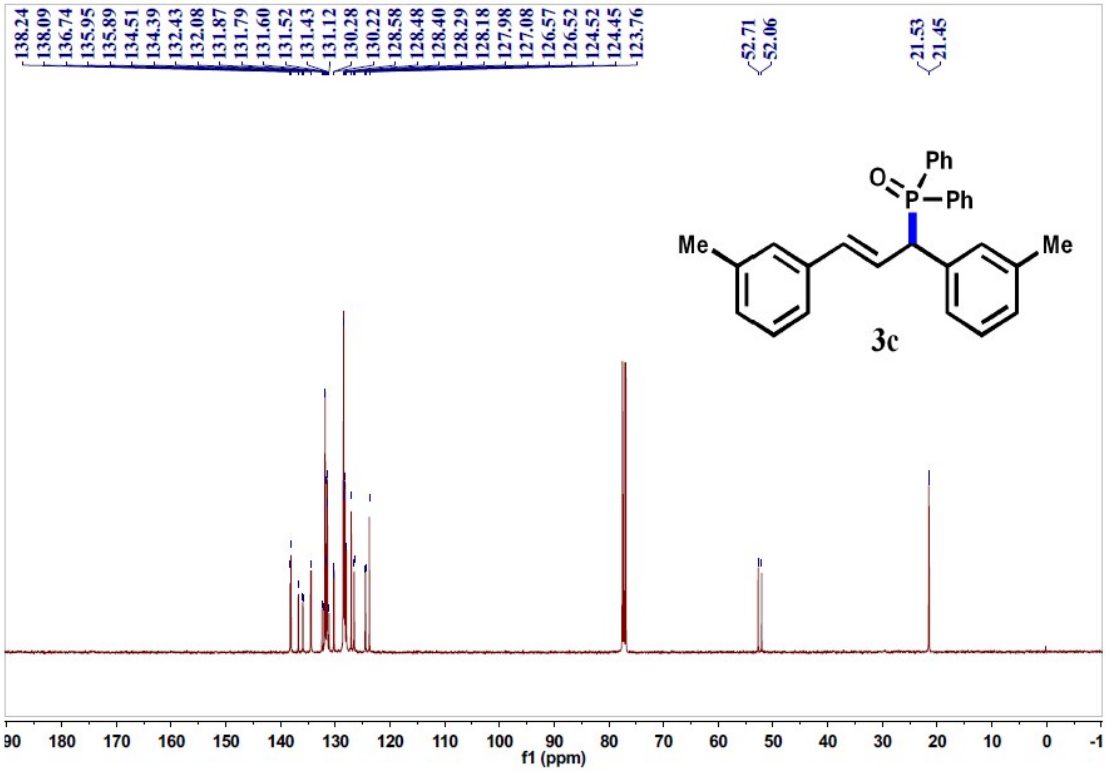
f1 (ppm)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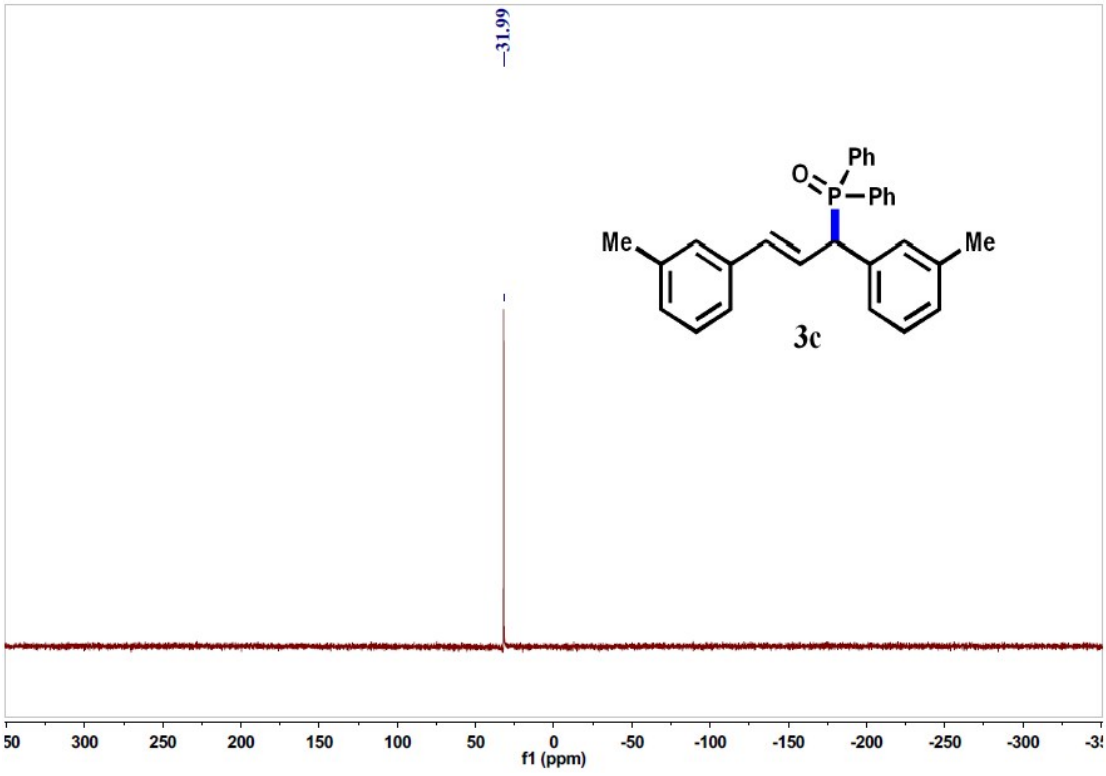
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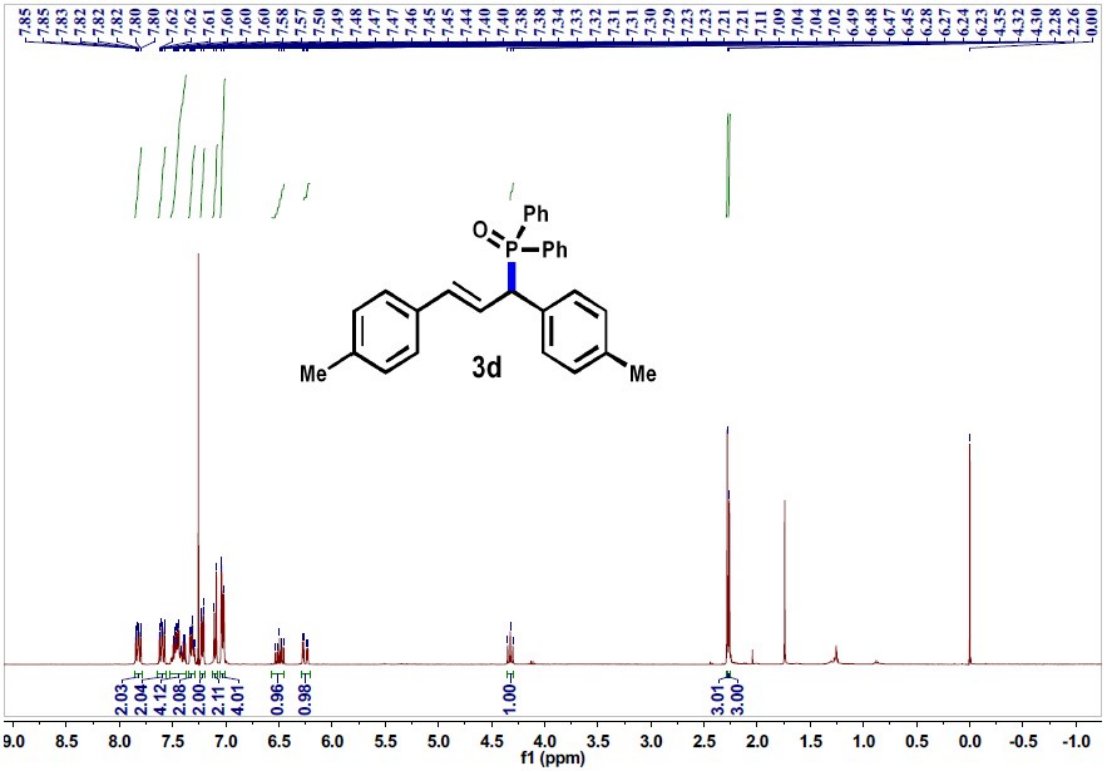
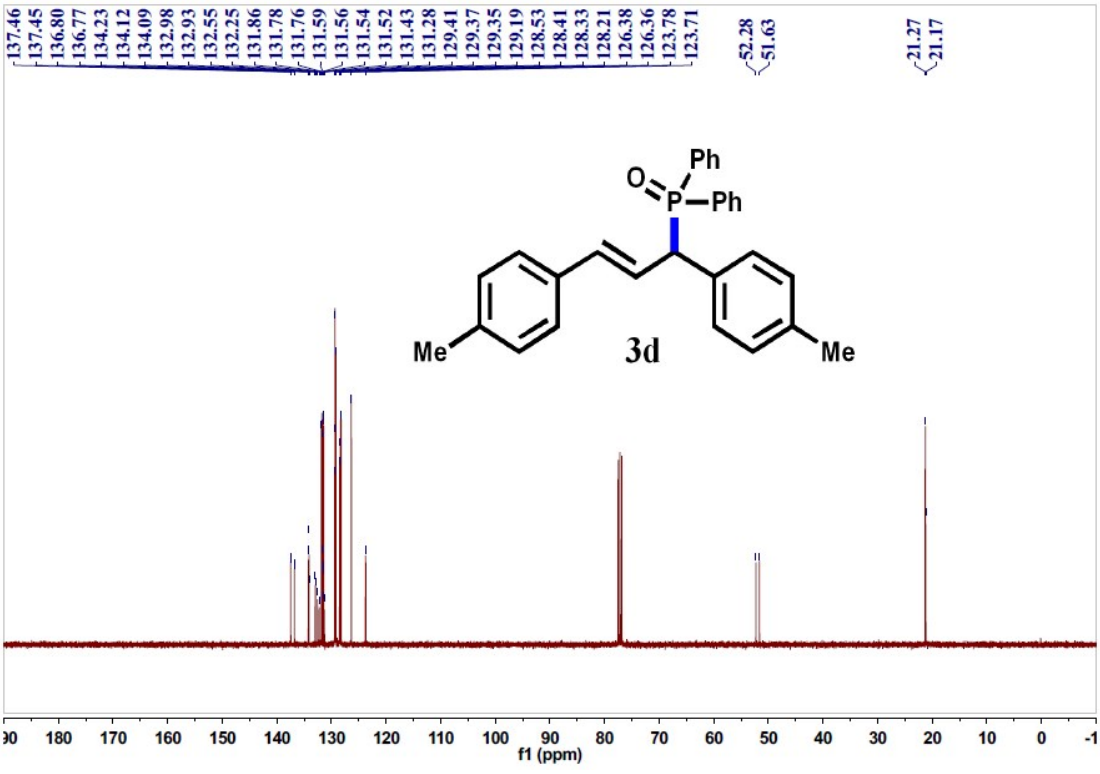
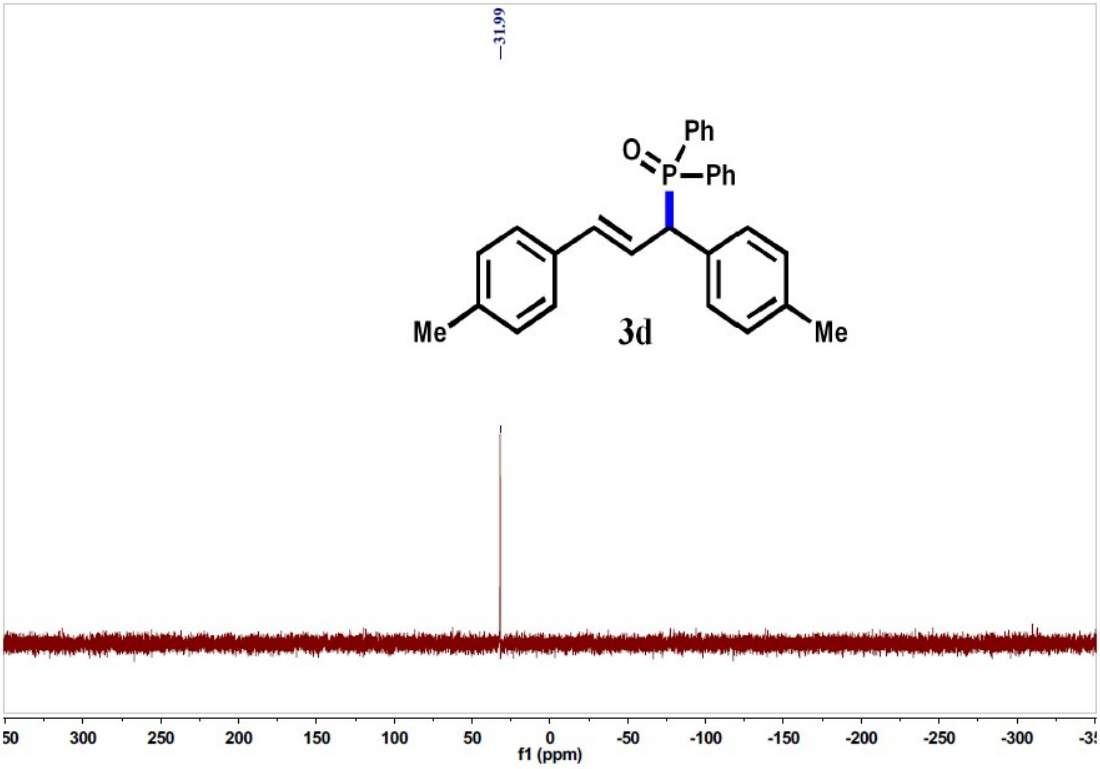
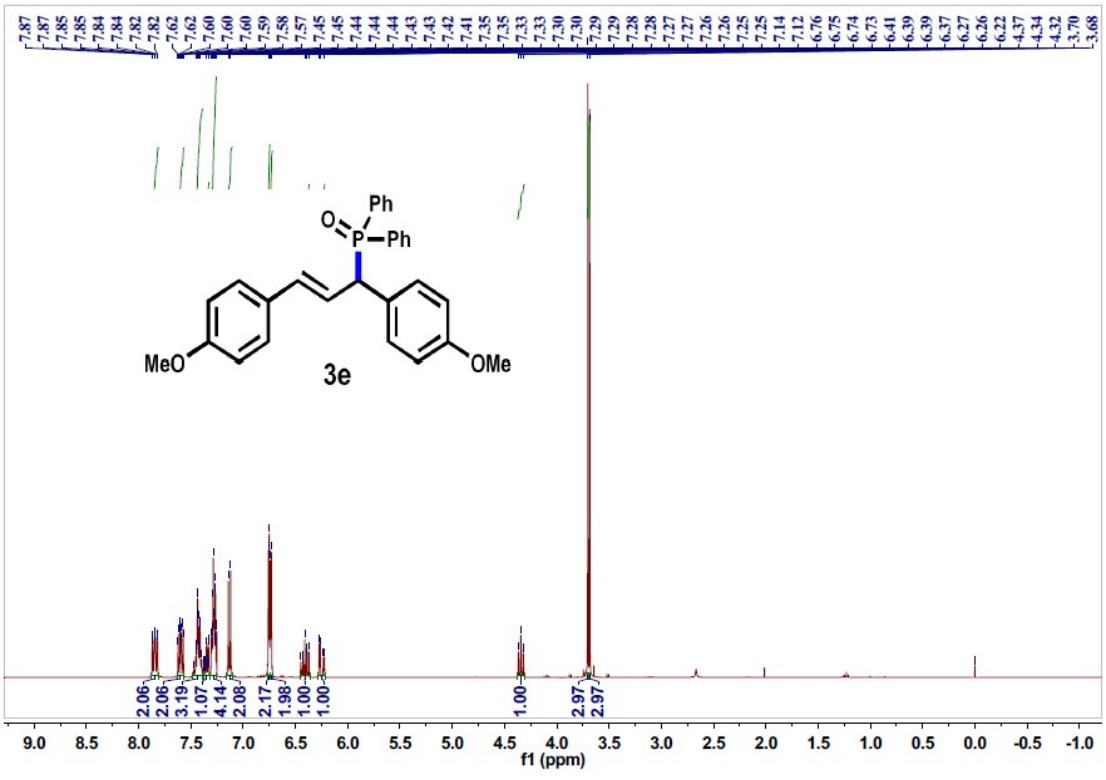
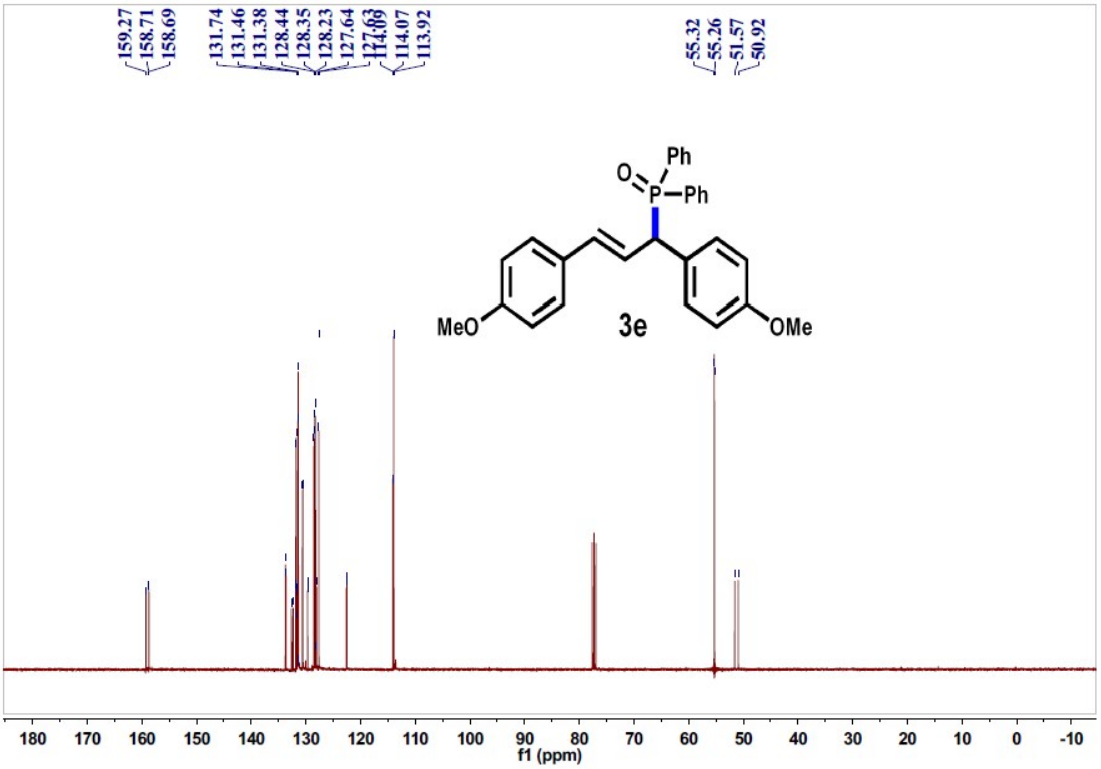
3c
f1 (ppm)

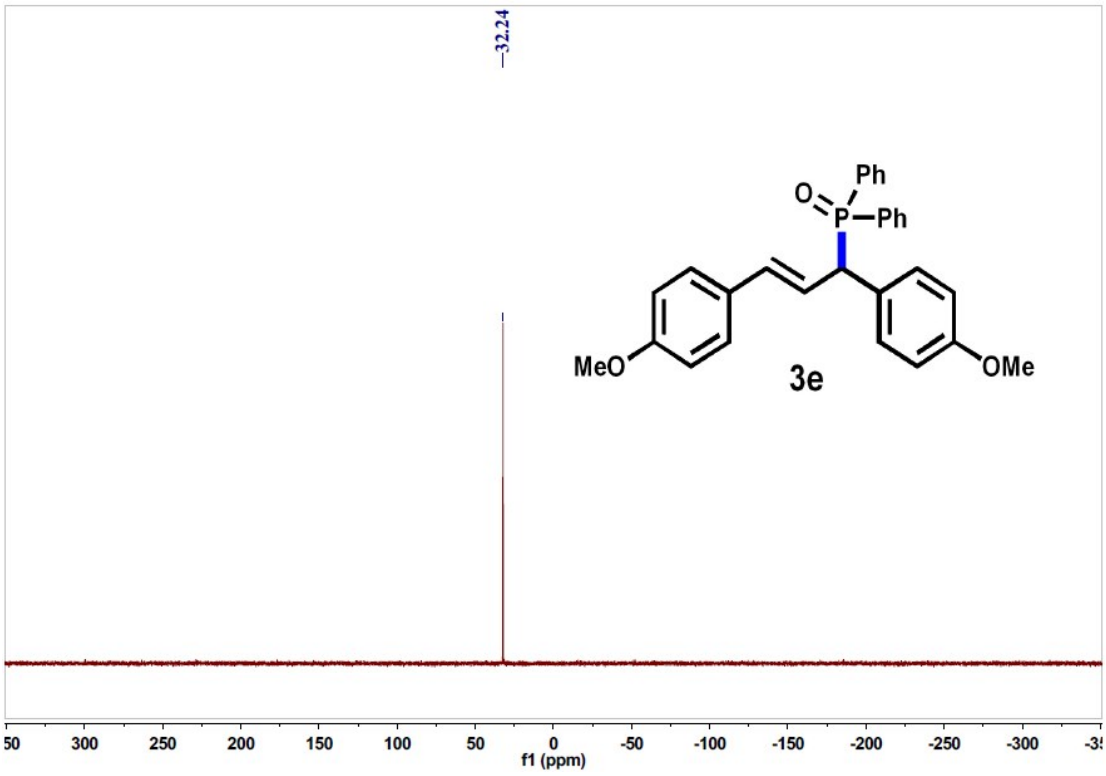

Ph
O
P
Ph
Me
Me
3d
f1 (ppm)

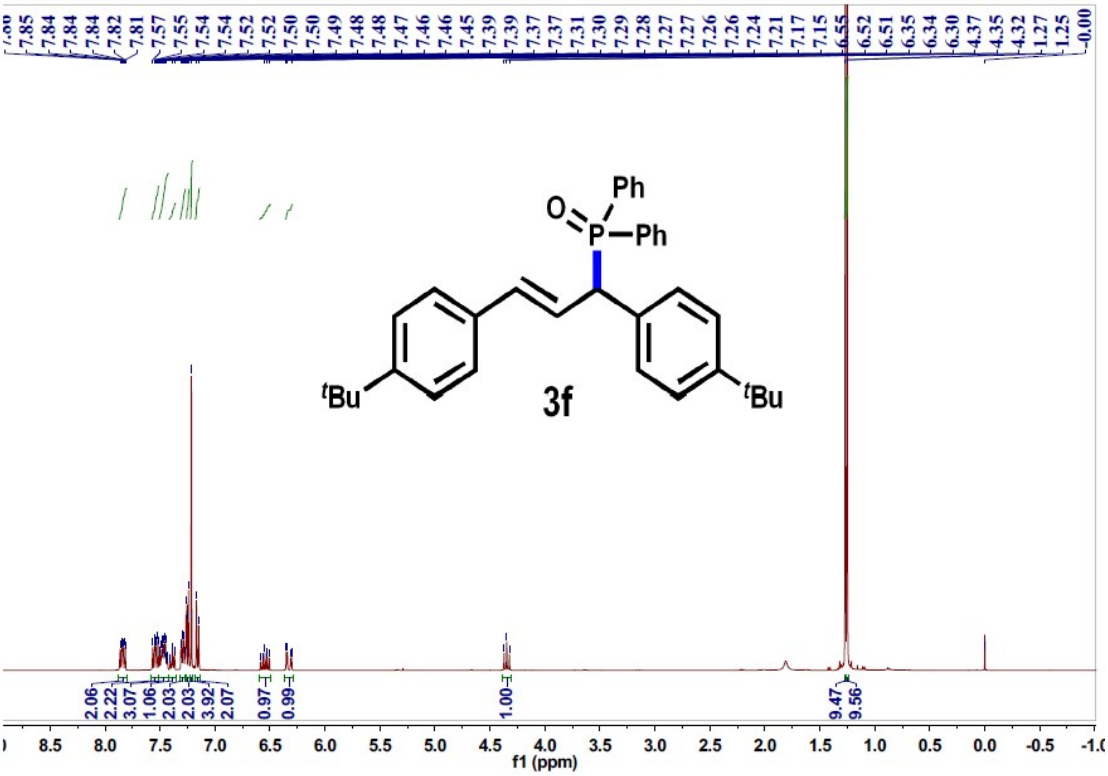

Ph
O
P
Ph
Me
Me
3d
f1 (ppm)

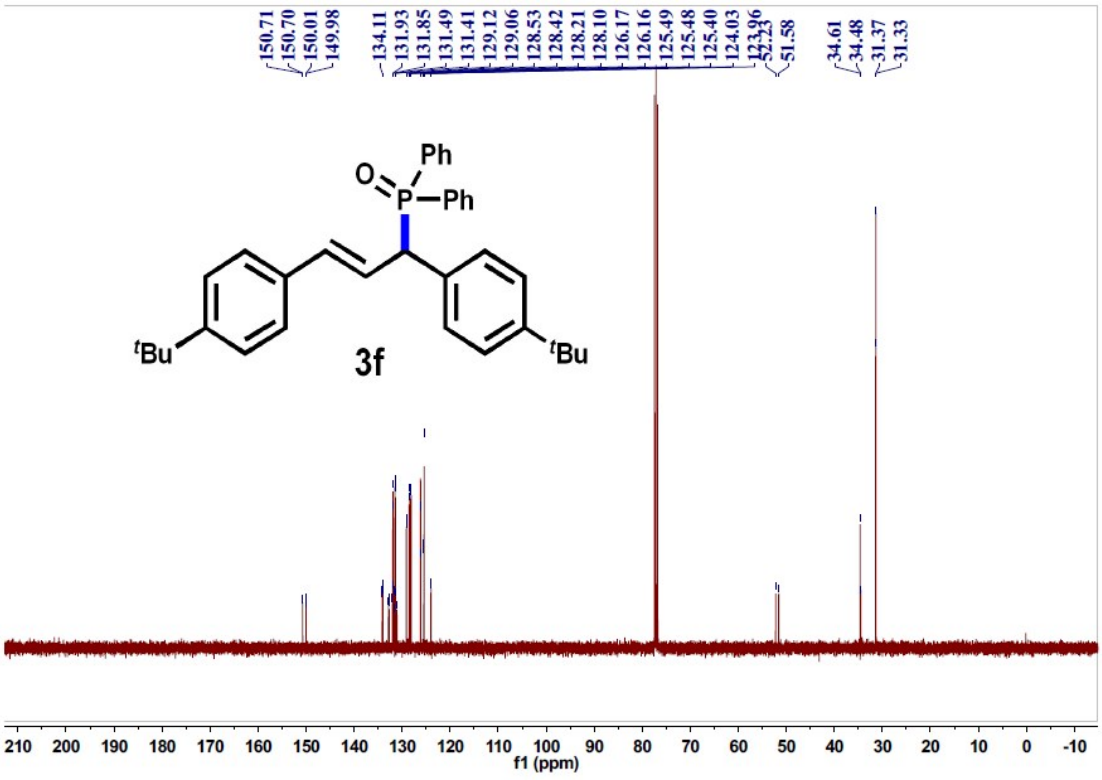

31.99
Ph
O
P
Ph
Me
Me
3d
f1 (ppm)

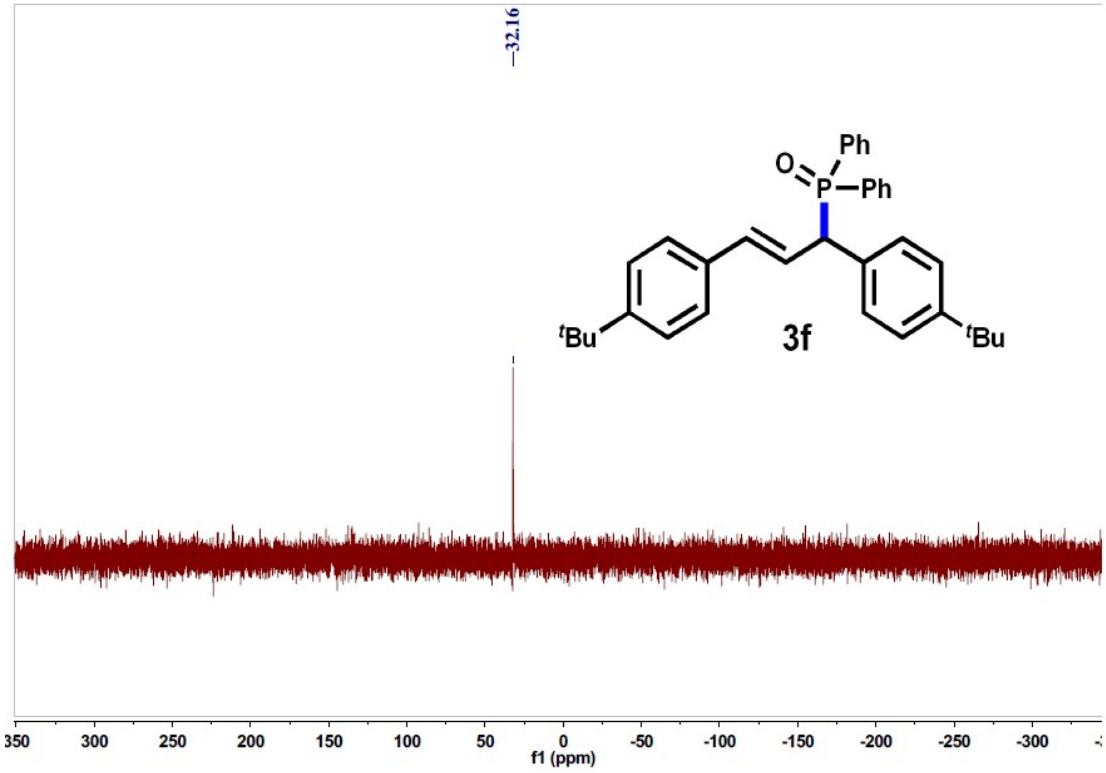

Ph
O
P
Ph
MeO
OMe
3e
f1 (ppm)

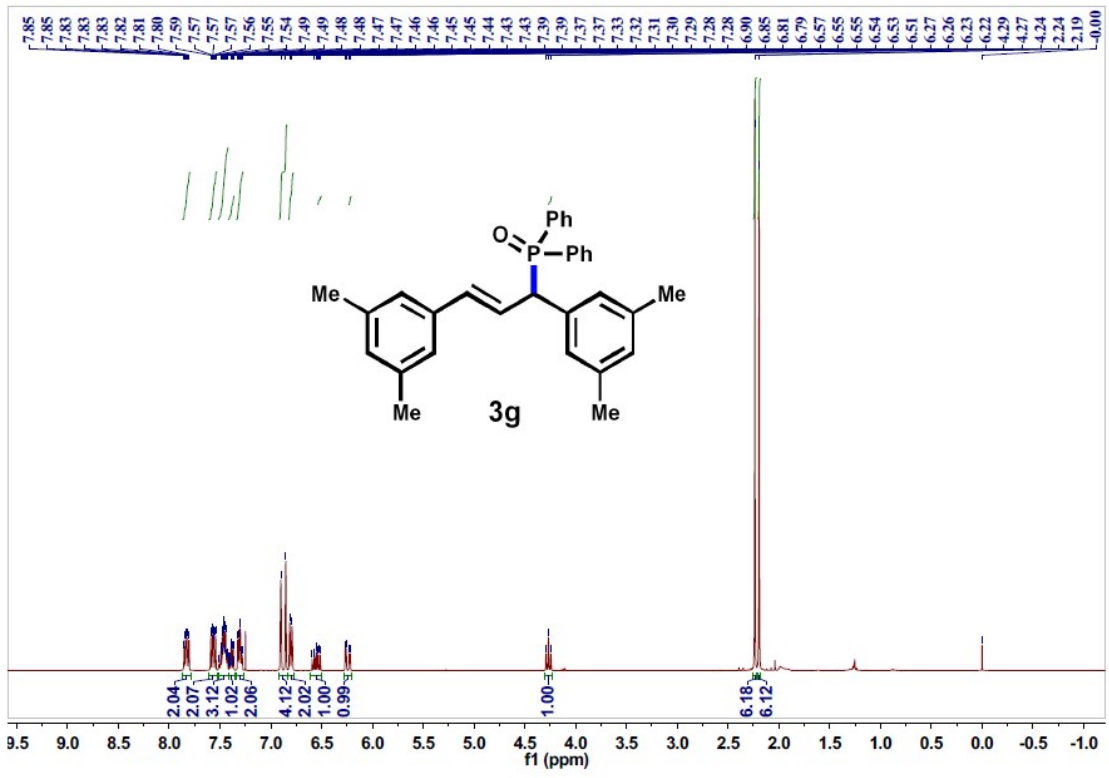

159.27
158.71
158.69
131.74
131.46
131.38
128.44
128.35
128.23
127.64
114.07
113.92
55.32
55.26
51.57
50.92
Ph
O
P
Ph
MeO
3e
OMe
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

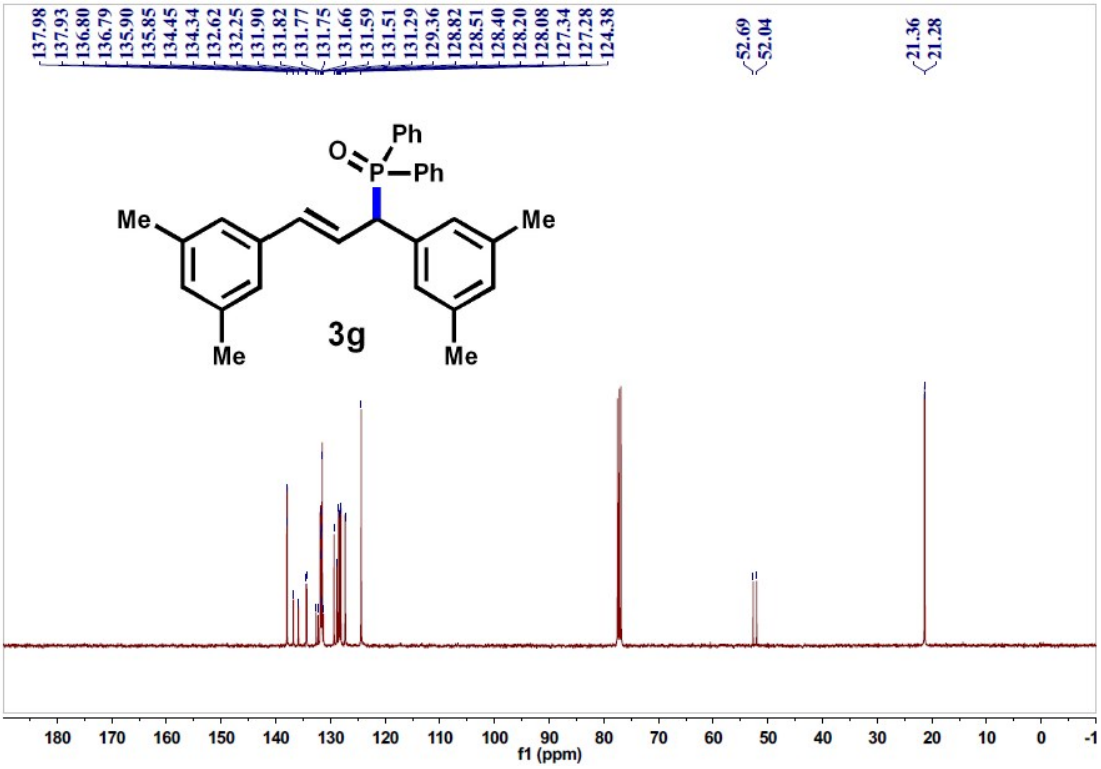

32.24
Ph
O
P
Ph
MeO
3e
OMe
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)

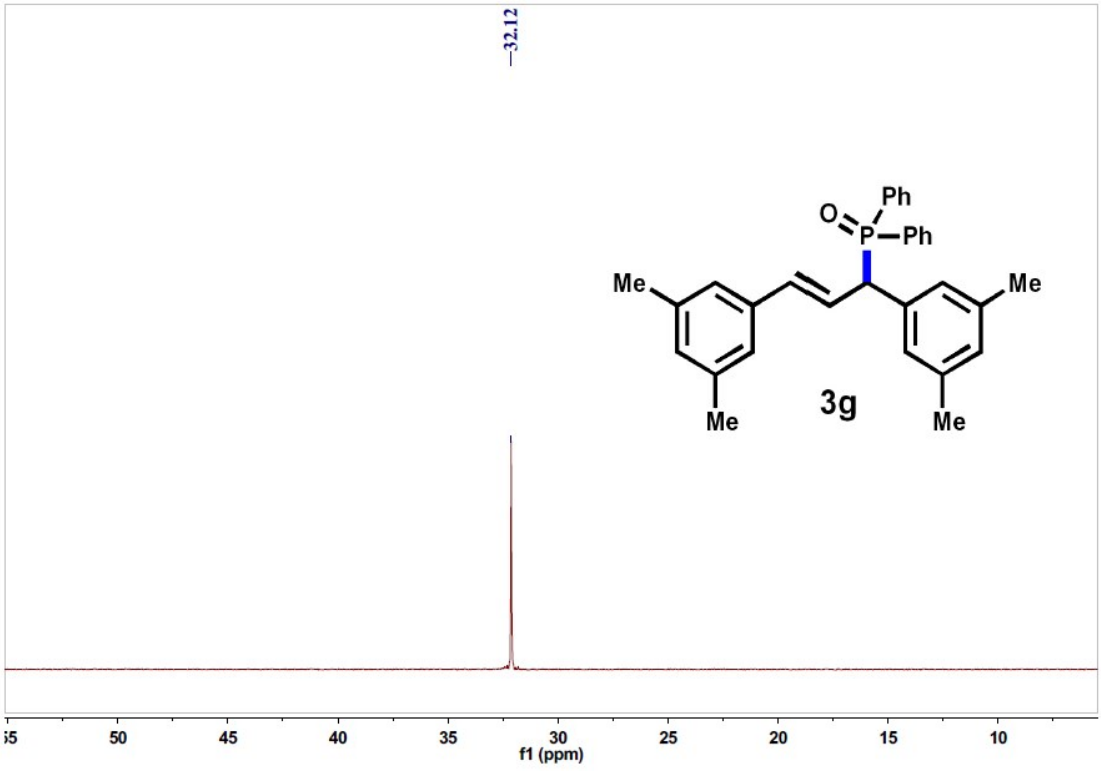

Ph
O
P
Ph
tBu
tBu
3f
f1 (ppm)

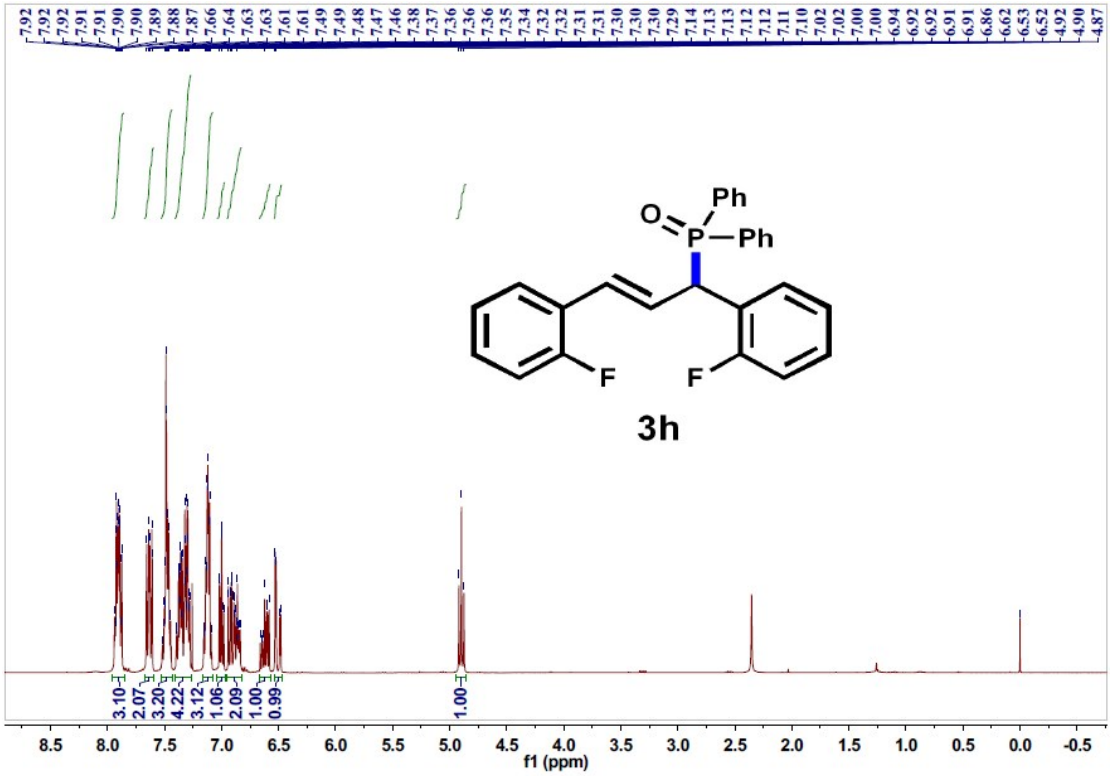

Ph
O
P
Ph
tBu
tBu
3f
f1 (ppm)

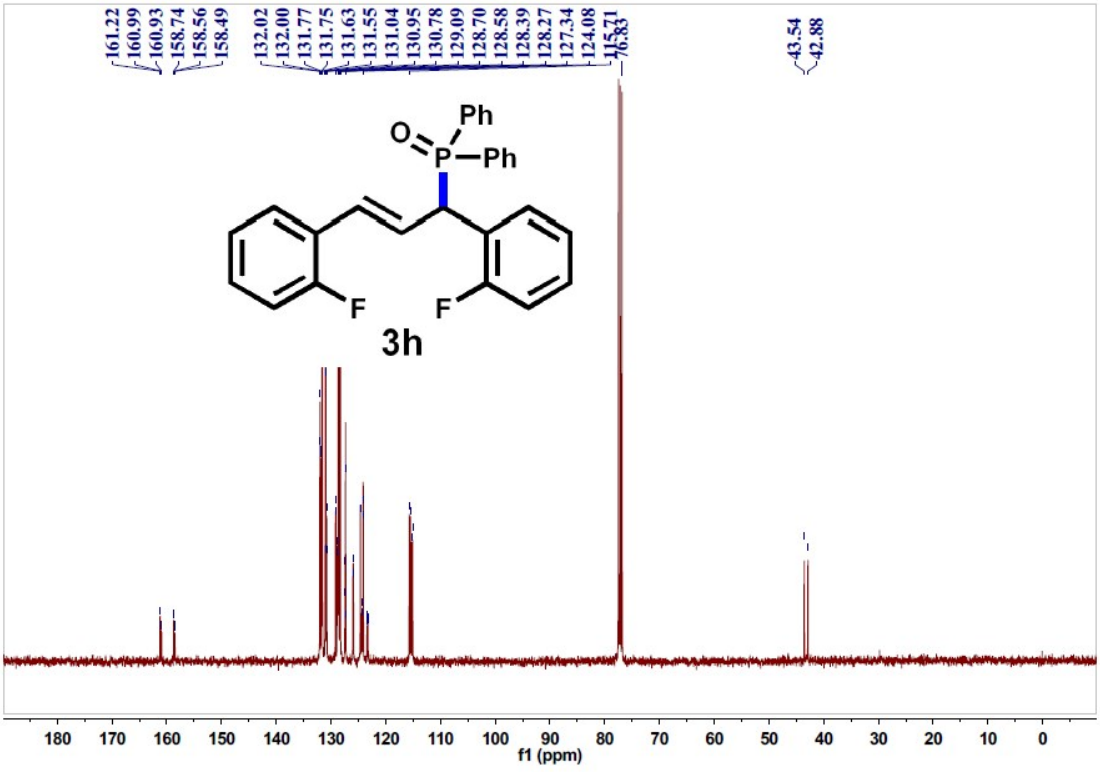

32.16
Ph
O
P
Ph
tBu
tBu
3f
f1 (ppm)

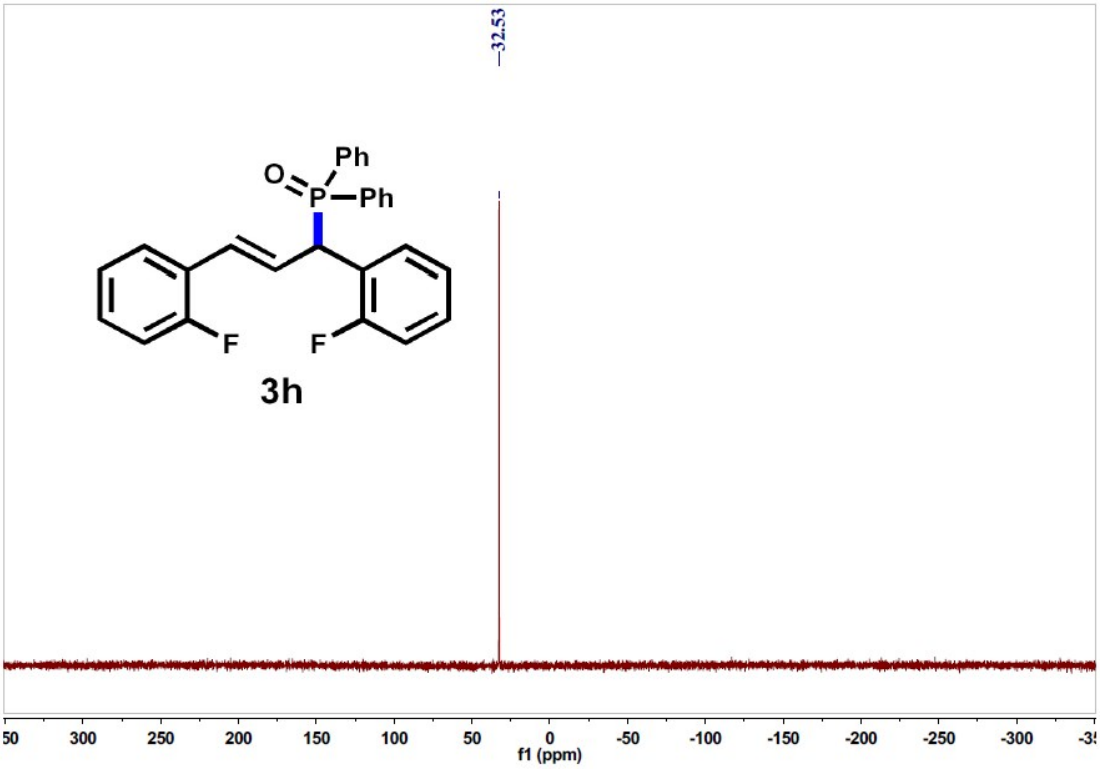

Ph
O
P
Ph
Me
Me
Me
Me
3g
2.04
2.07
3.12
1.02
2.06
4.12
2.02
1.00
0.99
1.00
6.18
6.12
f1 (ppm)

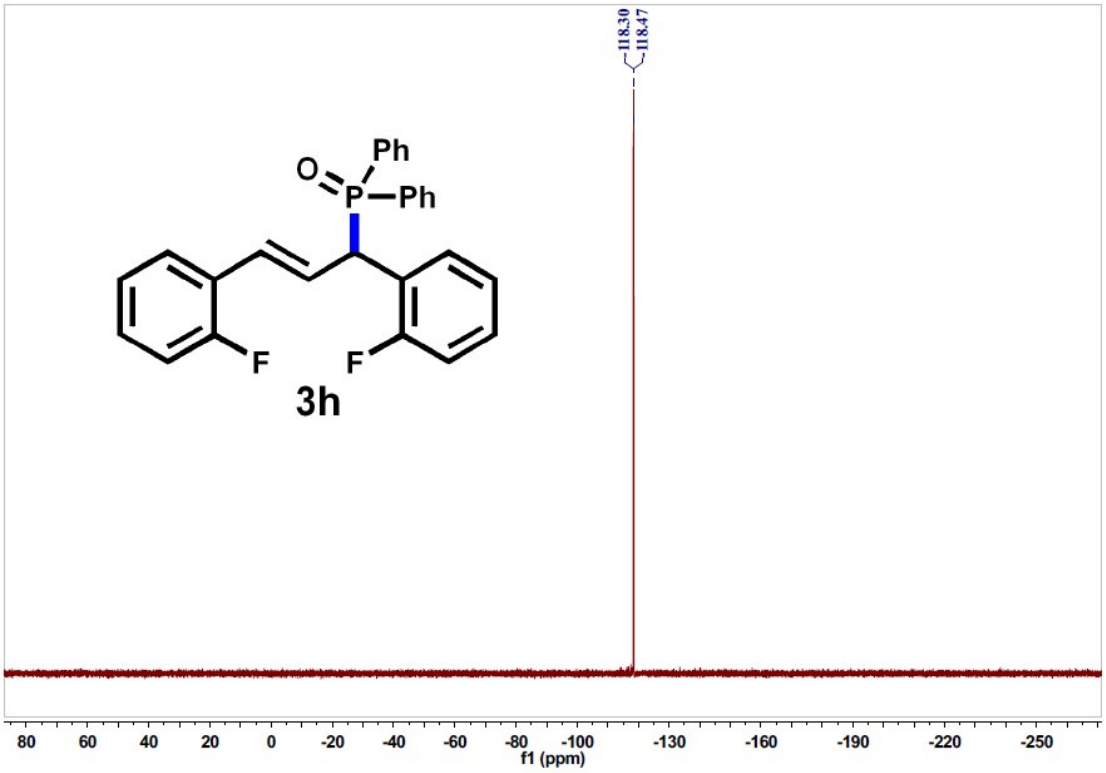

137.98
137.93
136.80
136.79
135.90
135.85
134.45
134.34
132.62
132.25
131.90
131.82
131.77
131.75
131.66
131.59
131.51
131.29
129.36
128.82
128.51
128.40
128.20
128.08
127.34
127.28
124.38
52.69
52.04
21.36
21.28
Ph
O
P
Ph
Me
Me
Me
Me
3g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

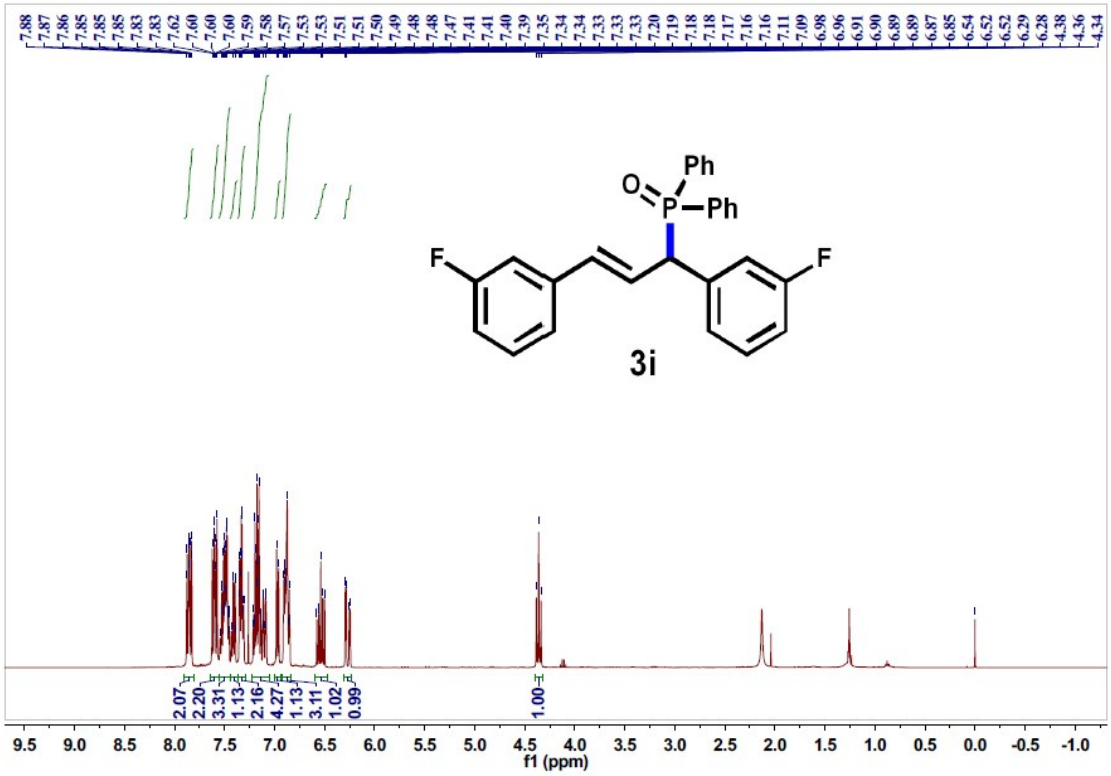

32.12
Ph
O
P
Ph
Me
Me
Me
Me
3g
50
45
40
35
30
25
20
15
10
f1 (ppm)

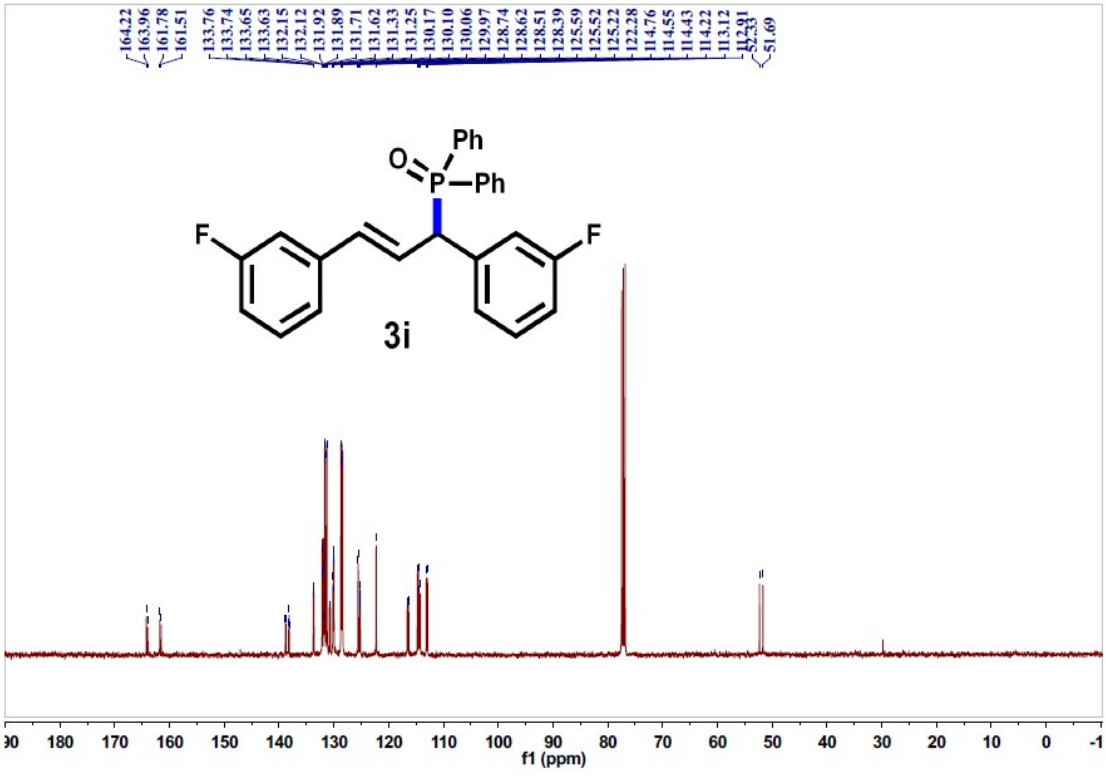

Ph
O
P
Ph
F
F
3h
f1 (ppm)

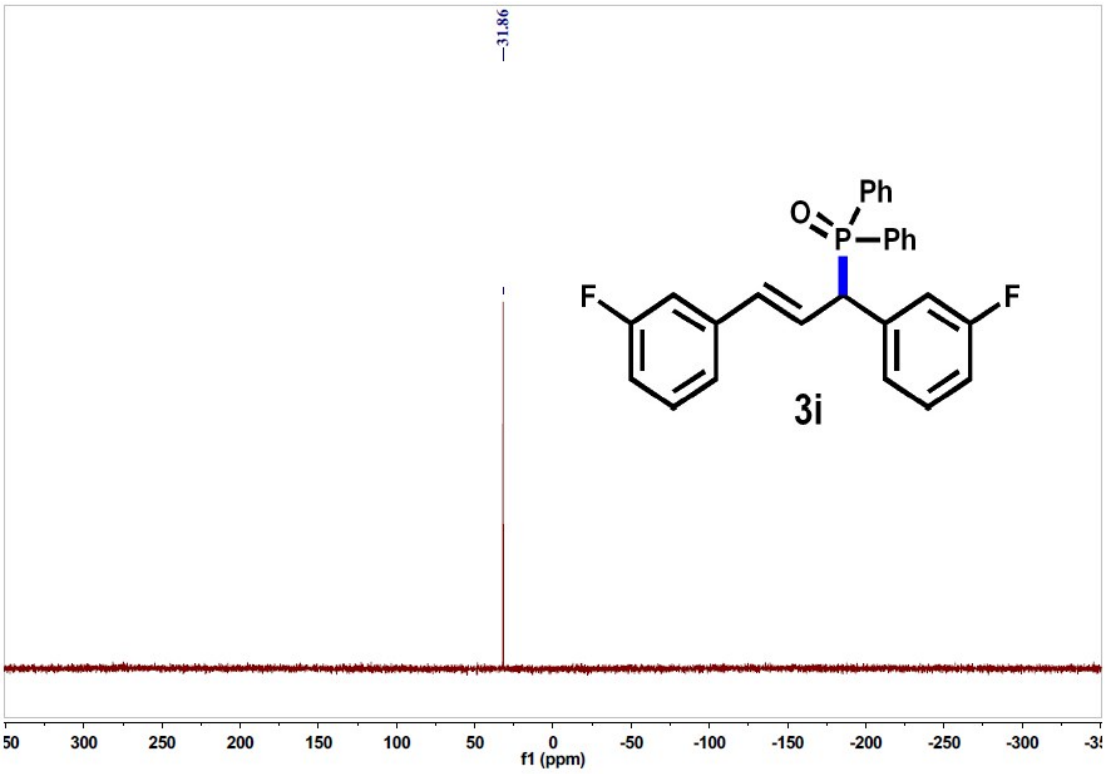

Ph
O
P
Ph
F
F
3h
f1 (ppm)

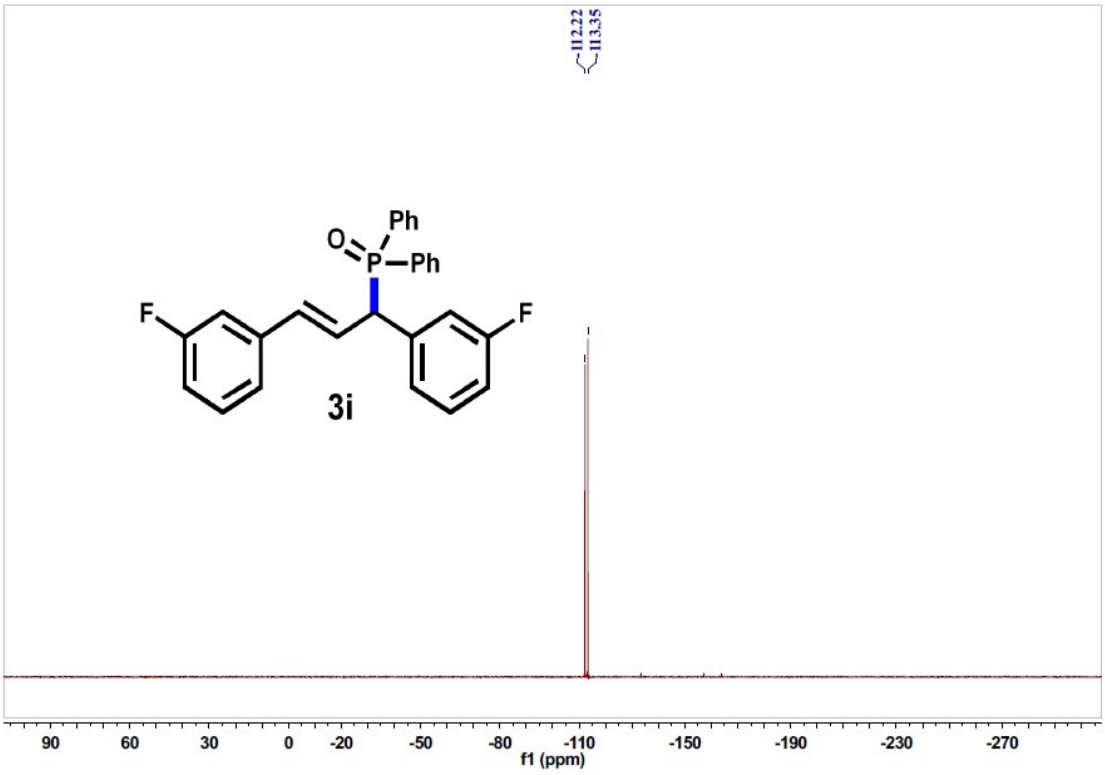

32.53
Ph
O
P
Ph
F
F
3h
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

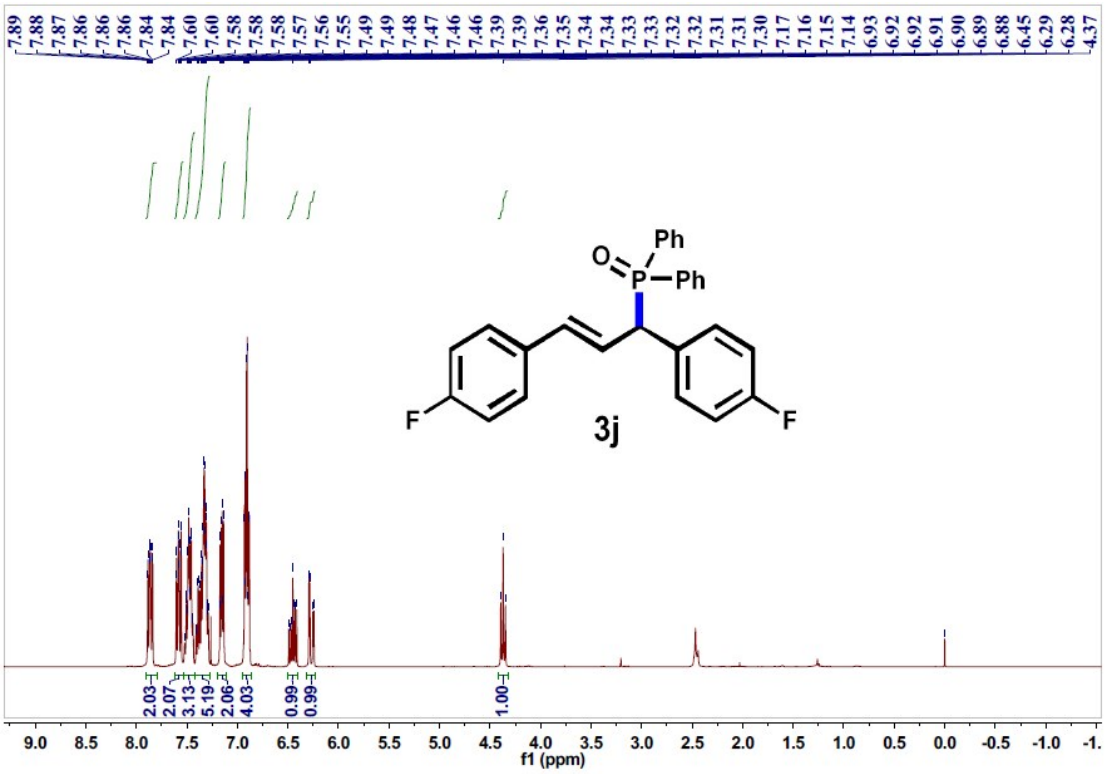

118.30
118.47
Ph
O
P
Ph
F
F
3h
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
-250
f1 (ppm)

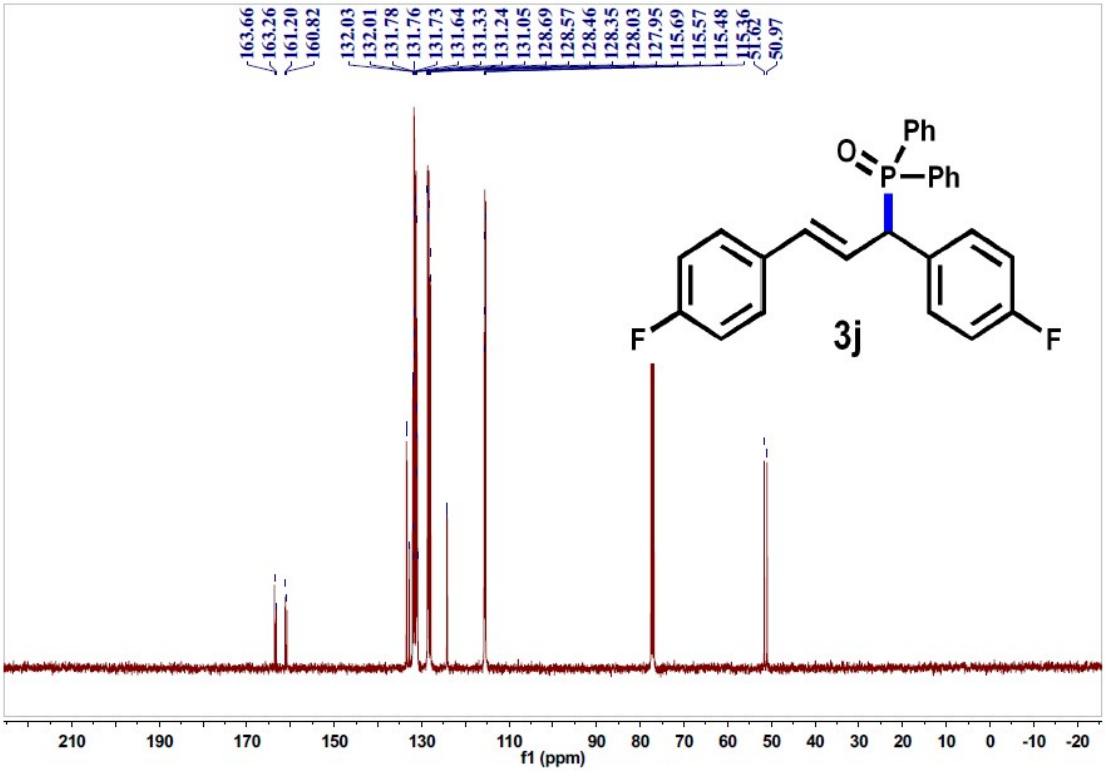

Ph
O
P
Ph
F
F
3i
f1 (ppm)

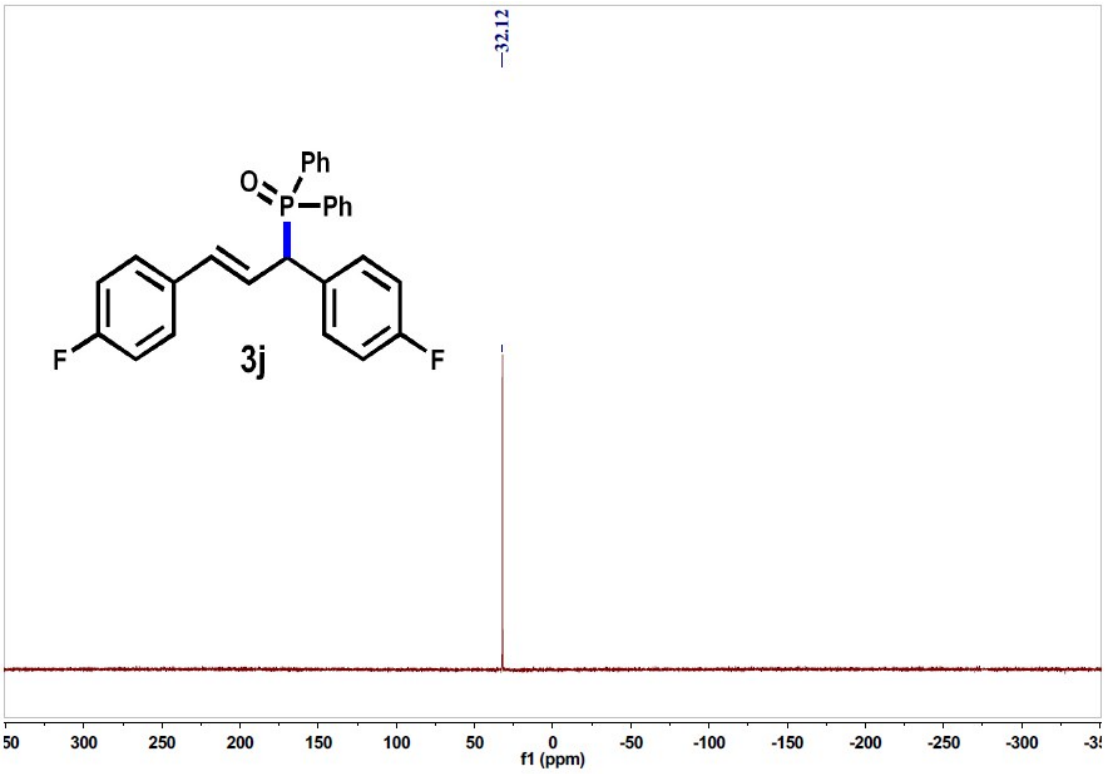

Ph
O
P
Ph
F
F
3i
f1 (ppm)

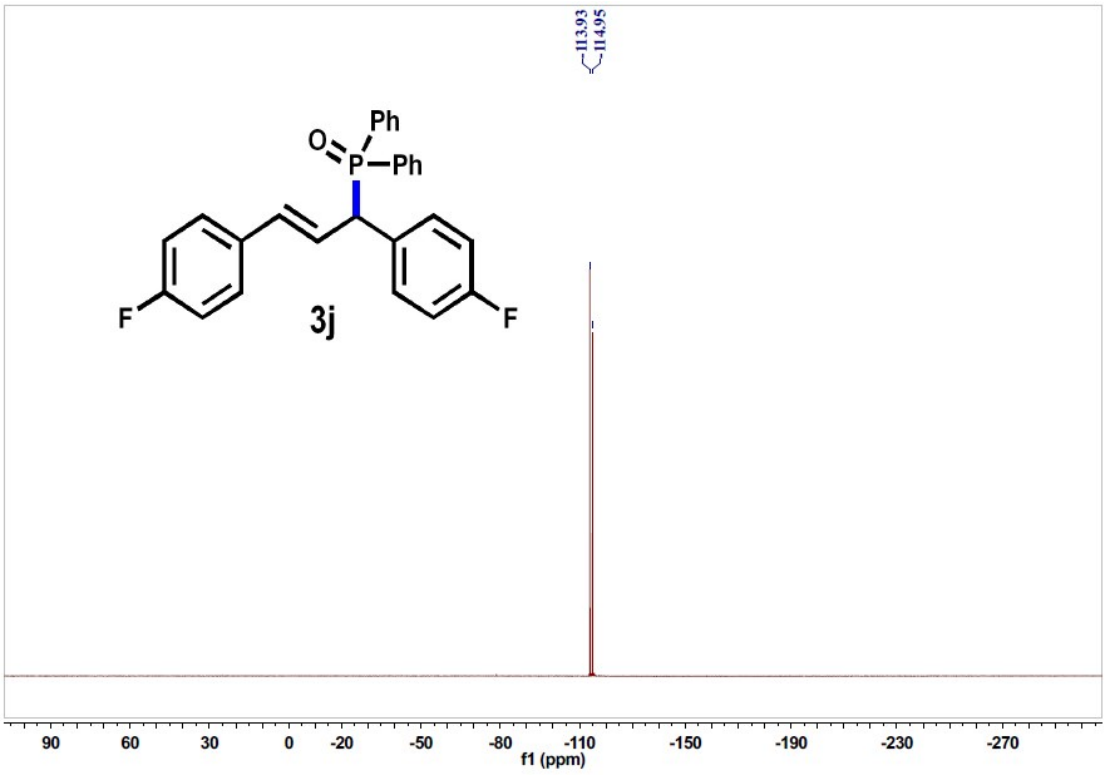

31.86
Ph
O
P
Ph
F
F
3i
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

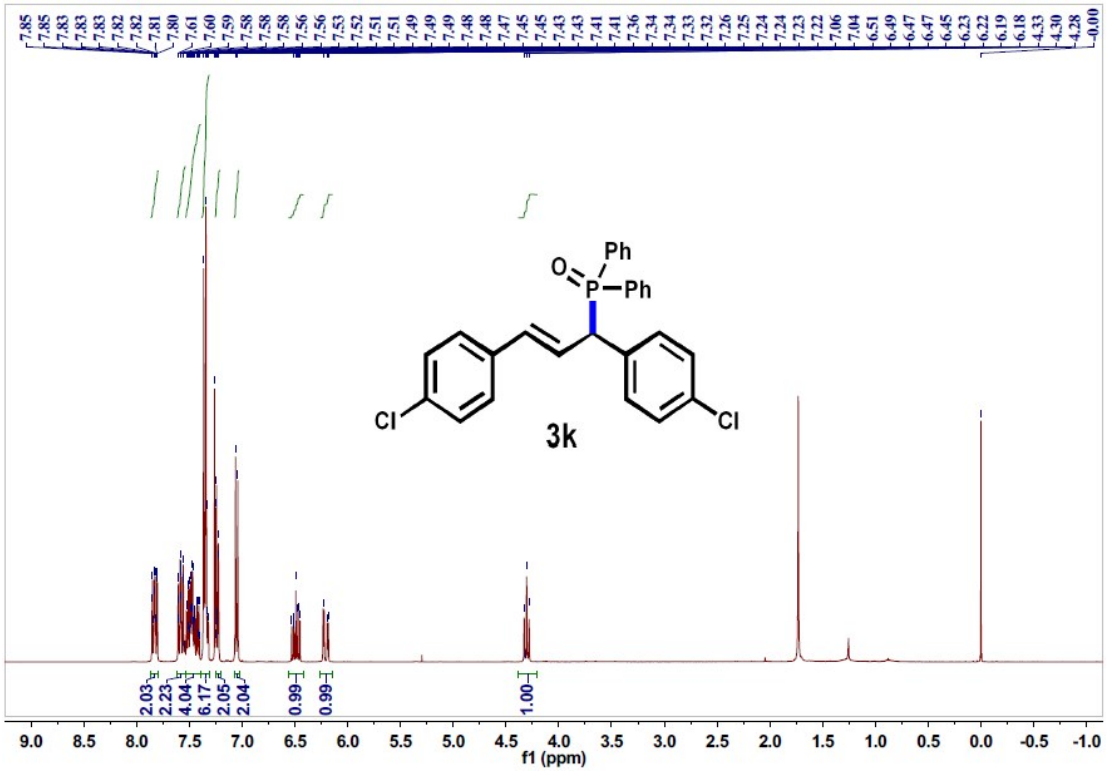

112.22
113.35
Ph
O
P
Ph
F
F
3i
90
60
30
0
-20
-50
-80
-110
-150
-190
-230
-270
f1 (ppm)

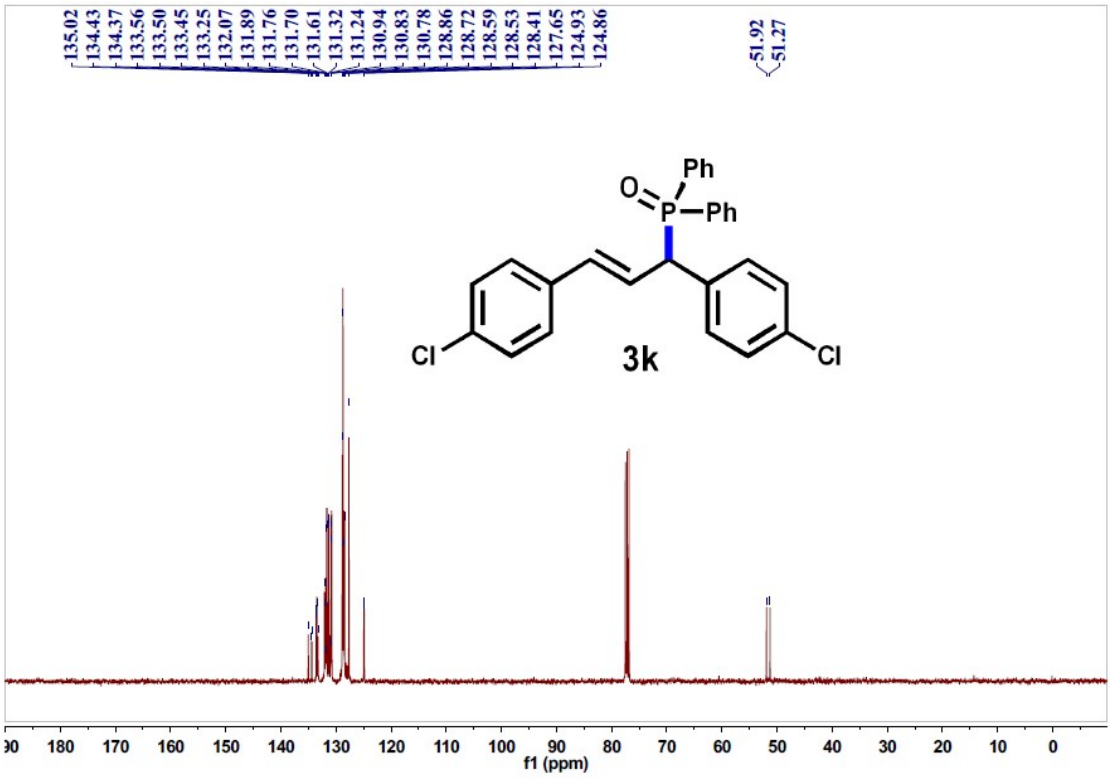

7.89 7.88 7.87 7.86 7.86 7.86 7.84 7.84 7.60 7.60 7.58 7.58 7.58 7.57 7.56 7.55 7.49 7.49 7.48 7.47 7.46 7.46 7.39 7.39 7.36 7.35 7.34 7.34 7.33 7.33 7.32 7.32 7.31 7.31 7.30 7.17 7.16 7.15 7.14 6.93 6.92 6.92 6.91 6.90 6.89 6.88 6.45 6.29 6.28 4.37
Ph
O
P
Ph
F
F
3j
2.03 2.07 3.13 5.19 2.06 4.03 0.99 0.99 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

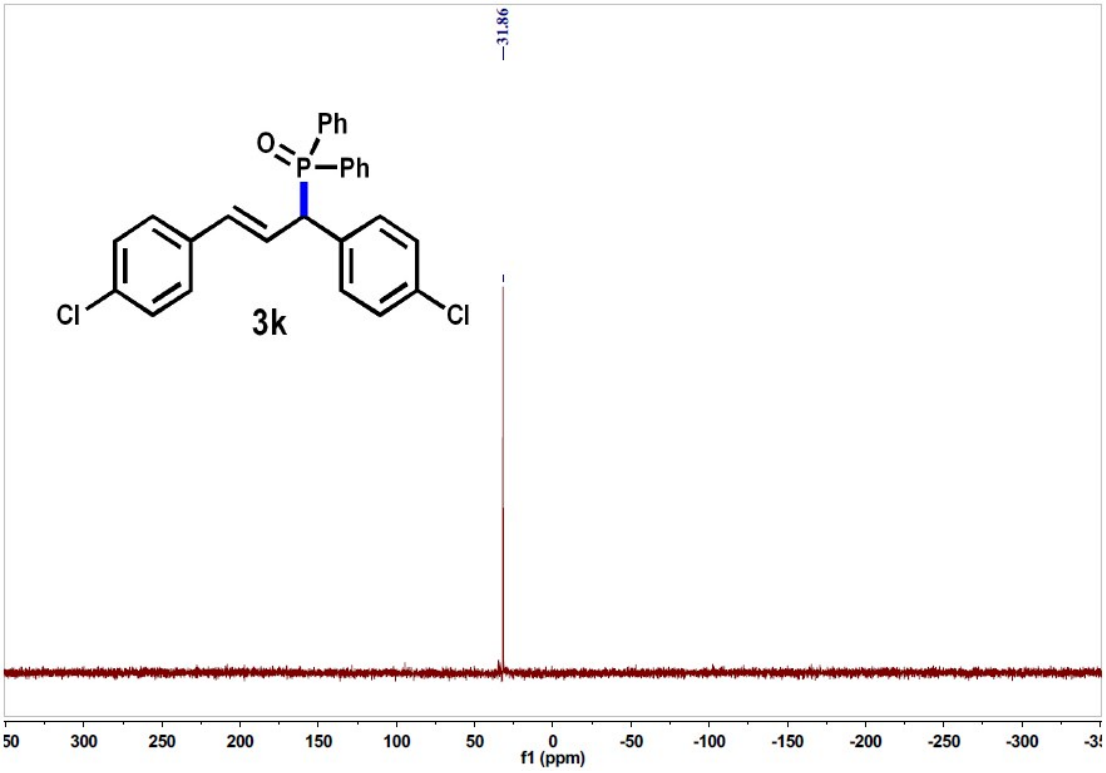

163.66 163.26 161.20 160.82 132.03 132.01 131.78 131.76 131.73 131.64 131.33 131.24 131.05 128.69 128.57 128.46 128.35 128.03 127.95 115.69 115.57 115.48 115.36 51.62 50.97
Ph
O
P
Ph
F
F
3j
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

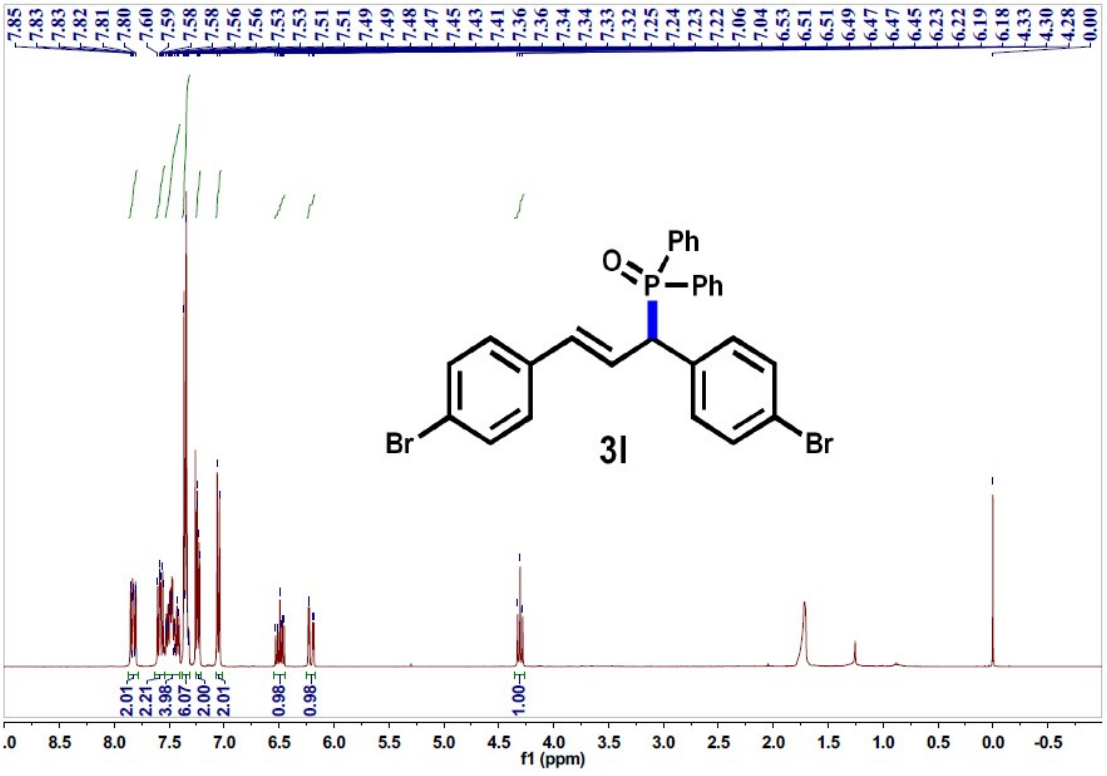

32.12
Ph
O
P
Ph
F
3j
F
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

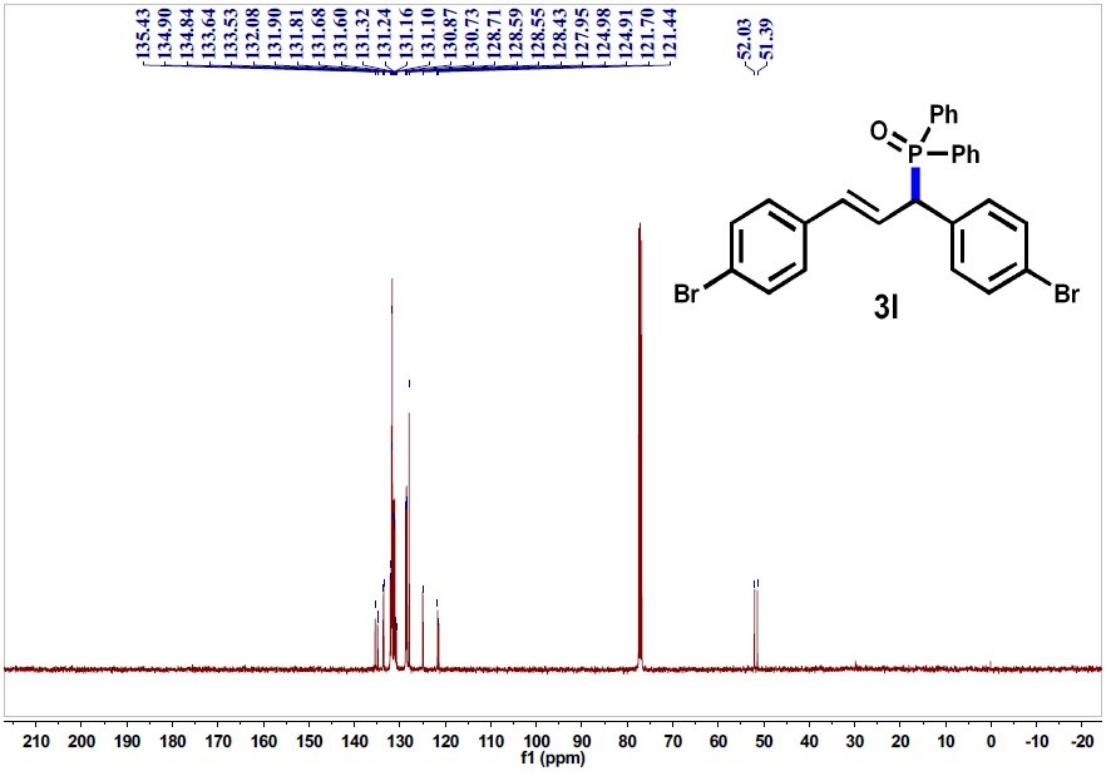

113.93
114.95
Ph
O
P
Ph
F
3j
F
90
60
30
0
-20
-50
-80
-110
-150
-190
-230
-270
f1 (ppm)


7.85 7.85 7.83 7.83 7.83 7.82 7.82 7.81 7.80 7.61 7.60 7.59 7.58 7.58 7.56 7.56 7.53 7.52 7.51 7.51 7.49 7.49 7.49 7.48 7.48 7.47 7.45 7.45 7.43 7.43 7.41 7.41 7.36 7.34 7.34 7.33 7.32 7.26 7.25 7.24 7.24 7.23 7.22 7.06 7.04 6.51 6.49 6.47 6.47 6.45 6.23 6.22 6.19 6.18 4.33 4.30 4.28 0.00
Ph
O
P
Ph
Cl
Cl
3k
2.03 2.23 4.04 6.17 2.05 2.04 0.99 0.99 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


135.02 134.43 134.37 133.56 133.50 133.45 133.25 132.07 131.89 131.76 131.70 131.61 131.32 131.24 130.94 130.83 130.78 128.86 128.72 128.59 128.53 128.41 127.65 124.93 124.86
51.92 51.27
Ph
O
P
Ph
Cl
Cl
3k
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


31.86
Ph
O
P
Ph
Cl
Cl
3k
f1 (ppm)


7.85 7.83 7.83 7.82 7.81 7.80 7.60 7.59 7.58 7.58 7.56 7.56 7.53 7.53 7.51 7.51 7.49 7.49 7.48 7.47 7.45 7.43 7.41 7.36 7.36 7.34 7.34 7.33 7.32 7.25 7.24 7.23 7.22 7.06 7.04 6.53 6.51 6.51 6.49 6.47 6.47 6.45 6.23 6.22 6.19 6.18 4.33 4.30 4.28 0.00
Ph
O
P
Ph
Br
Br
3l
2.01 2.21 3.98 6.07 2.00 2.01 0.98 0.98 1.00
f1 (ppm)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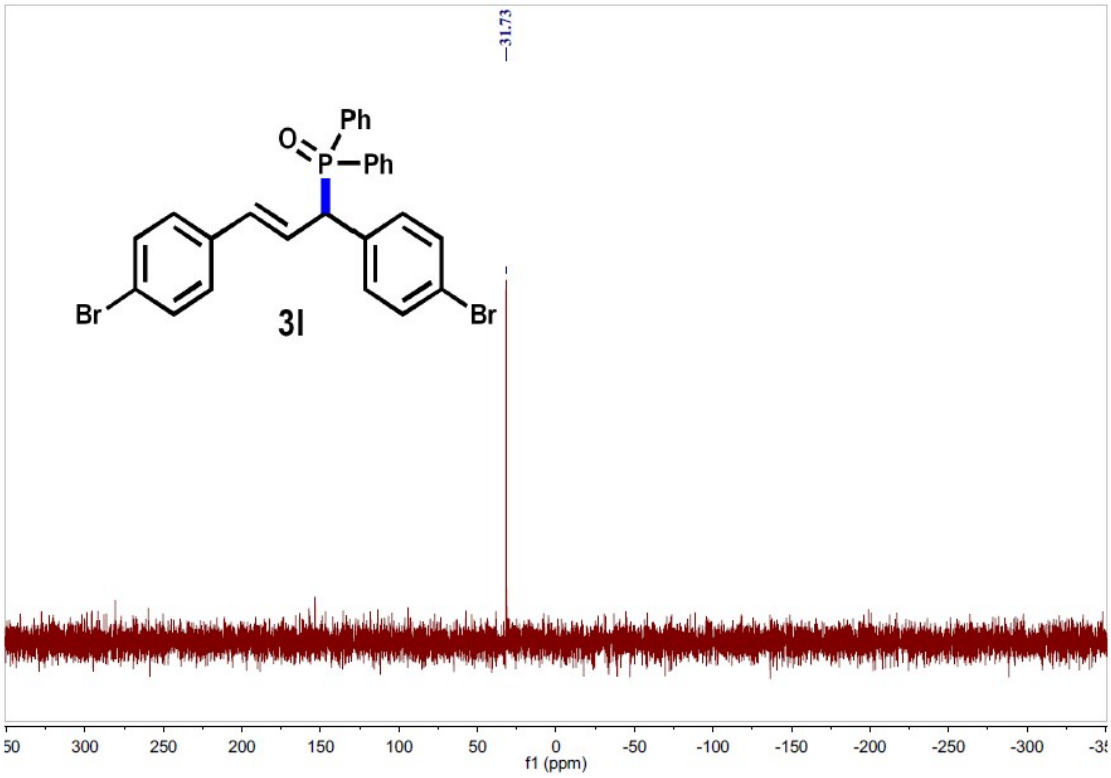
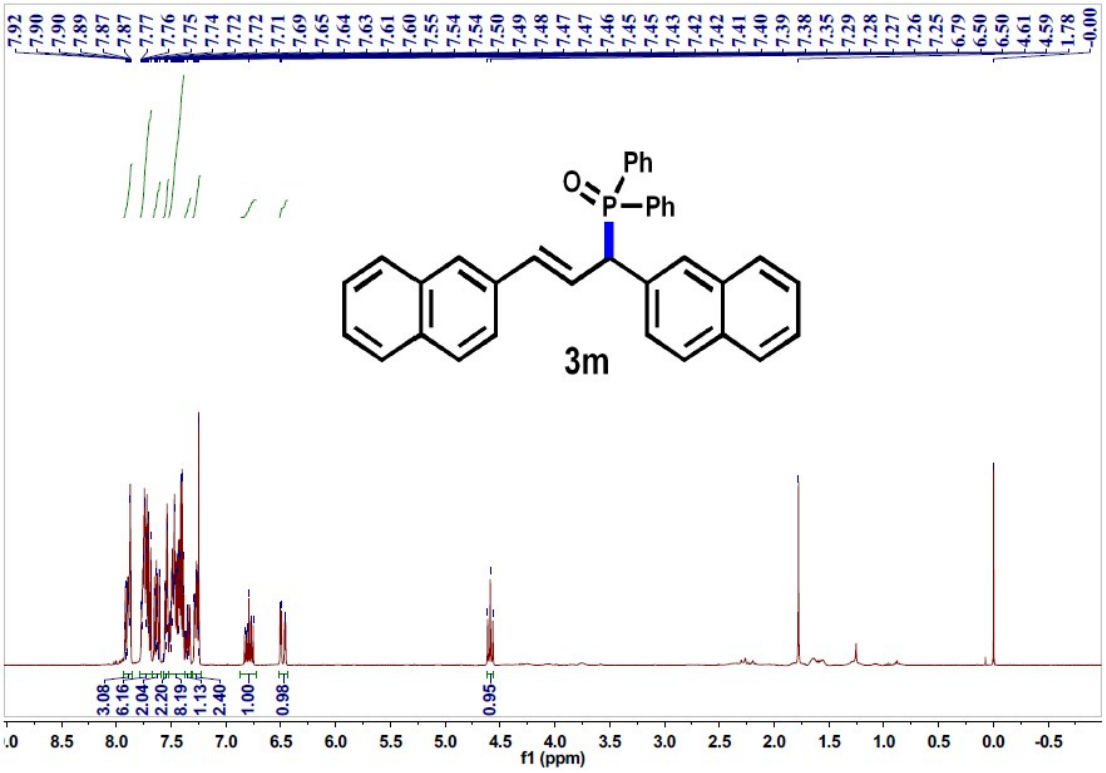
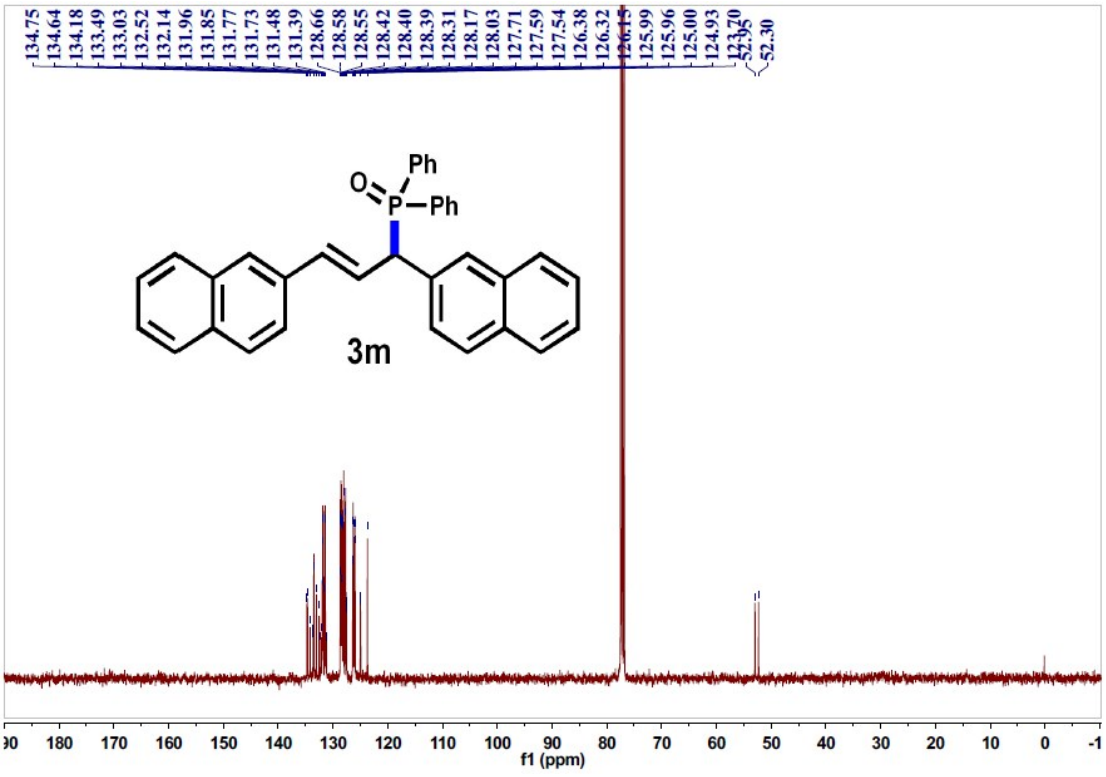
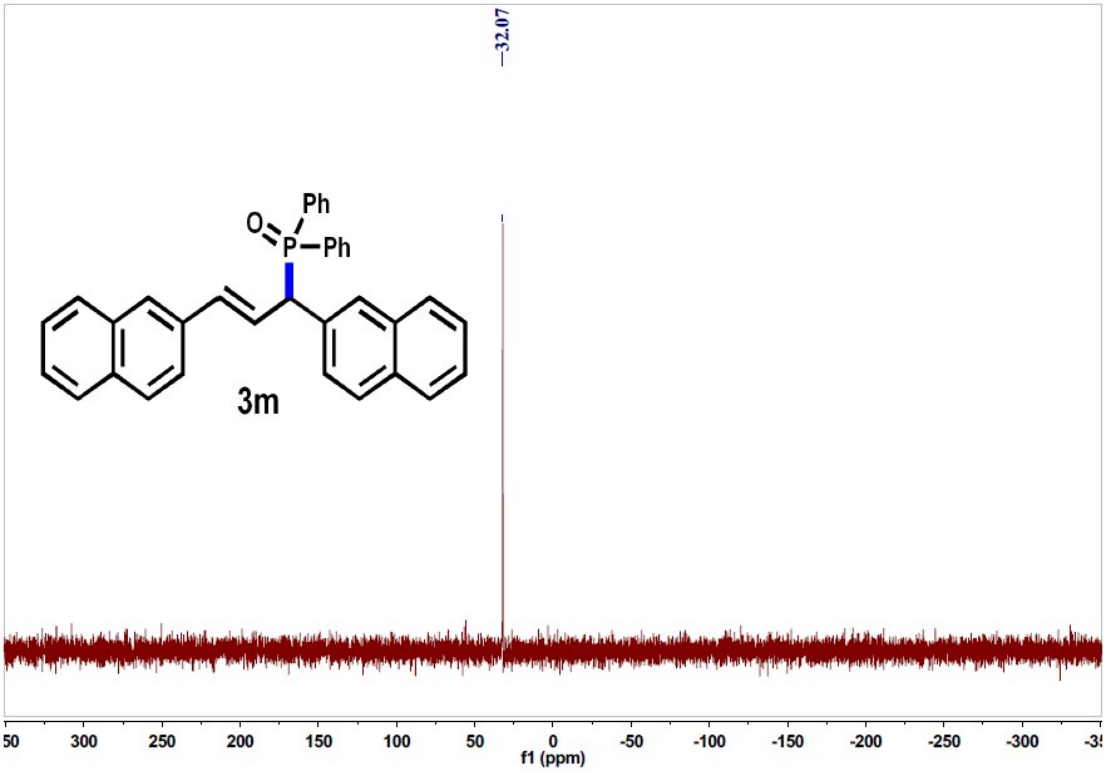
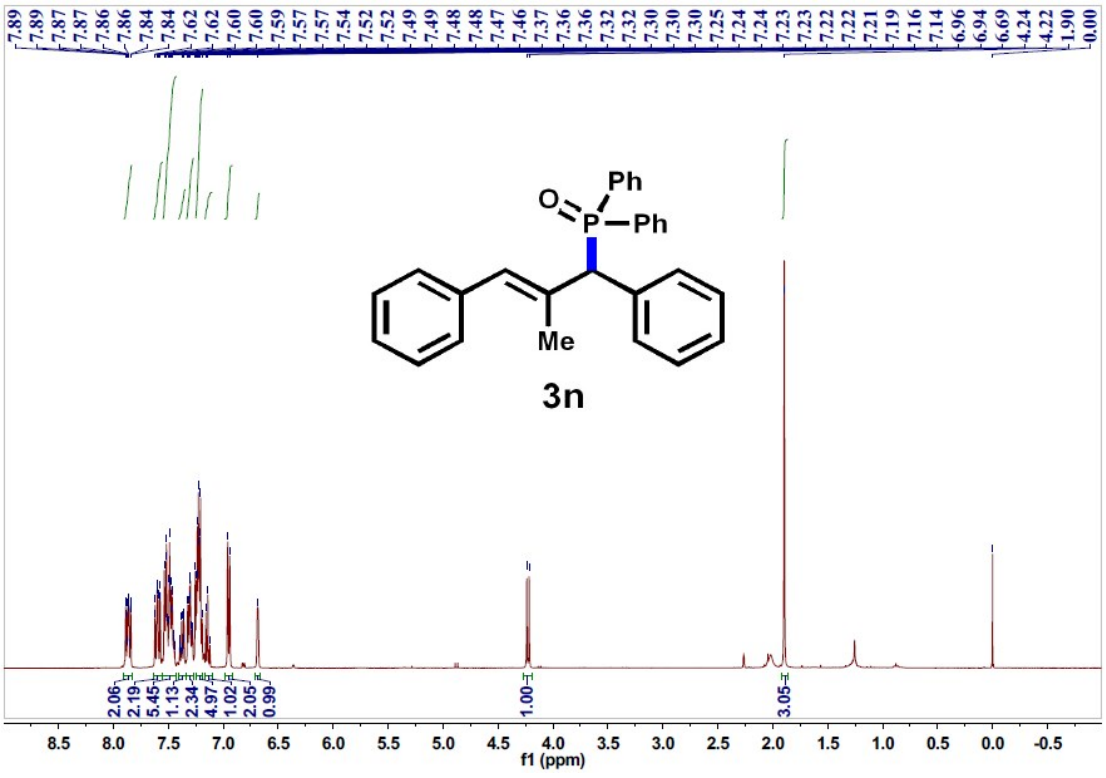
134.90
134.84
133.64
133.53
132.08
131.90
131.81
131.68
131.60
131.32
131.24
131.16
131.10
130.87
130.73
128.71
128.59
128.55
128.43
127.95
124.98
124.91
121.70
121.44
52.03
51.39
Ph
O
P
Ph
Br
Br
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


31.73
Ph
O
P
Ph
Br
Br
3l
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)


Ph
O
P
Ph
3m
f1 (ppm)


Ph
O
P
Ph
3m
f1 (ppm)


32.07
Ph
O
P
Ph
3m
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)


7.89
7.89
7.87
7.87
7.86
7.86
7.84
7.84
7.62
7.62
7.60
7.60
7.59
7.57
7.57
7.54
7.52
7.52
7.49
7.49
7.48
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7.46
7.37
7.36
7.36
7.32
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7.30
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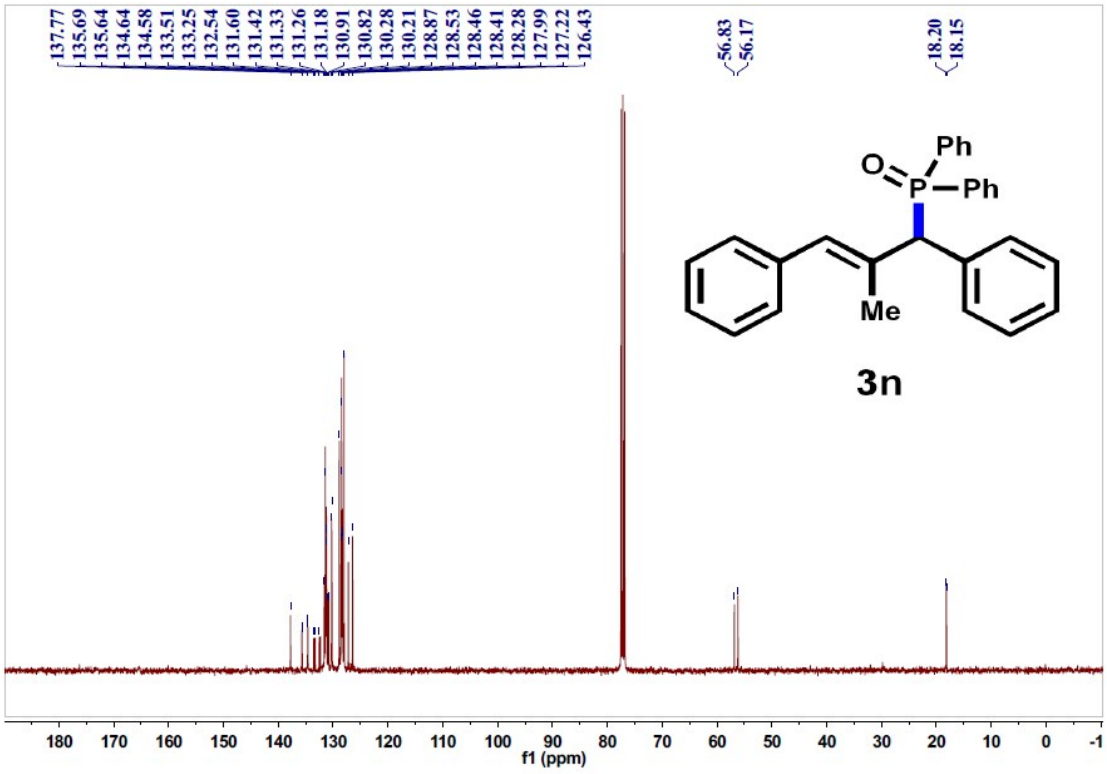
7.30
7.25
7.24
7.24
7.23
7.23
7.22
7.22
7.21
7.19
7.16
7.14
6.96
6.94
6.69
4.24
4.22
1.90
0.00
Ph
O
P
Ph
Me
3n
2.06
2.19
5.45
1.13
2.34
4.97
1.02
2.05
0.99
1.00
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


137.77
135.69
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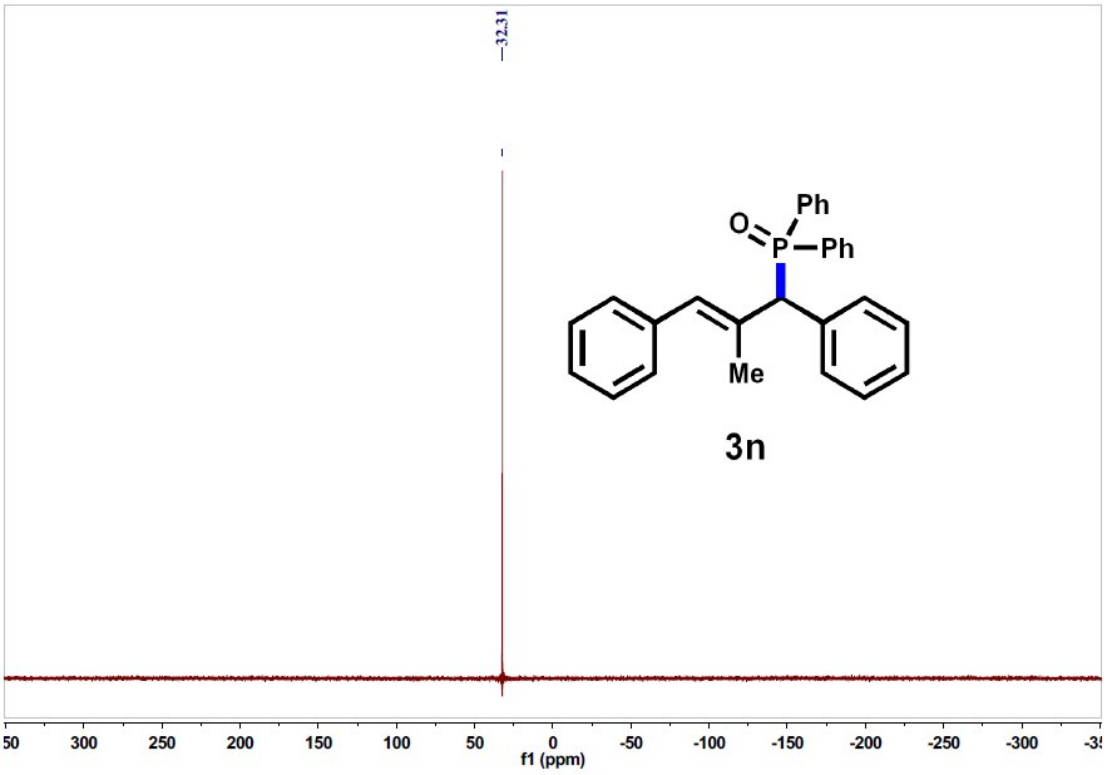
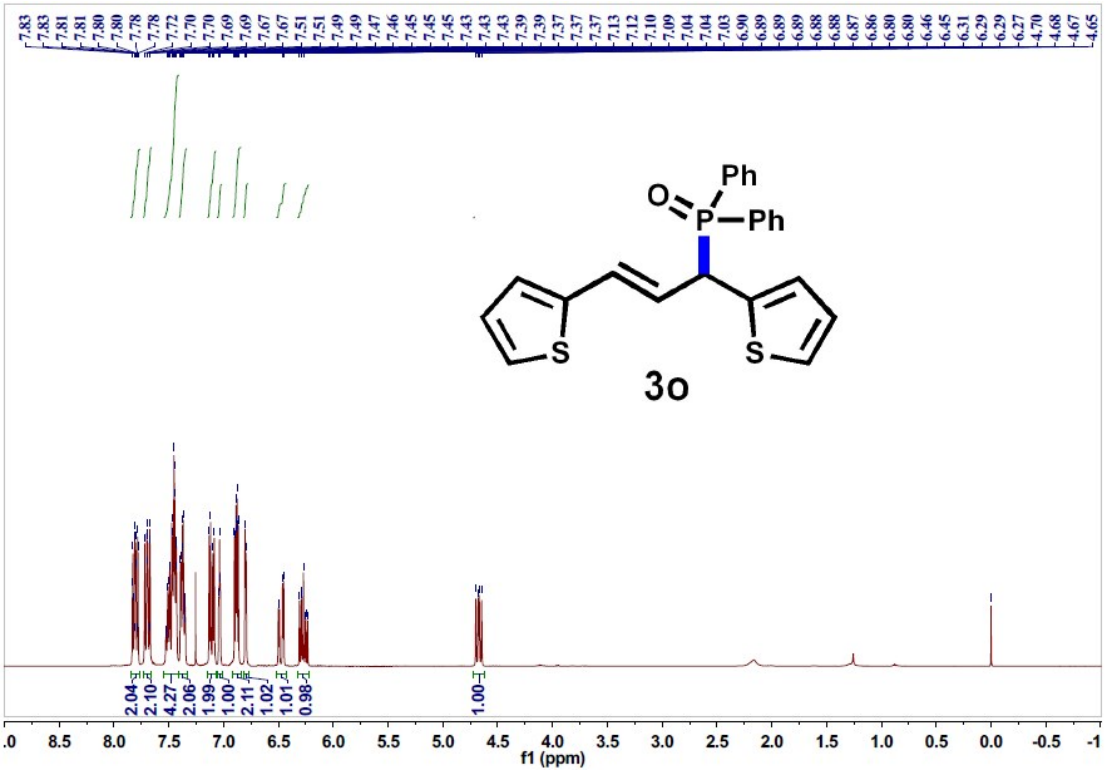
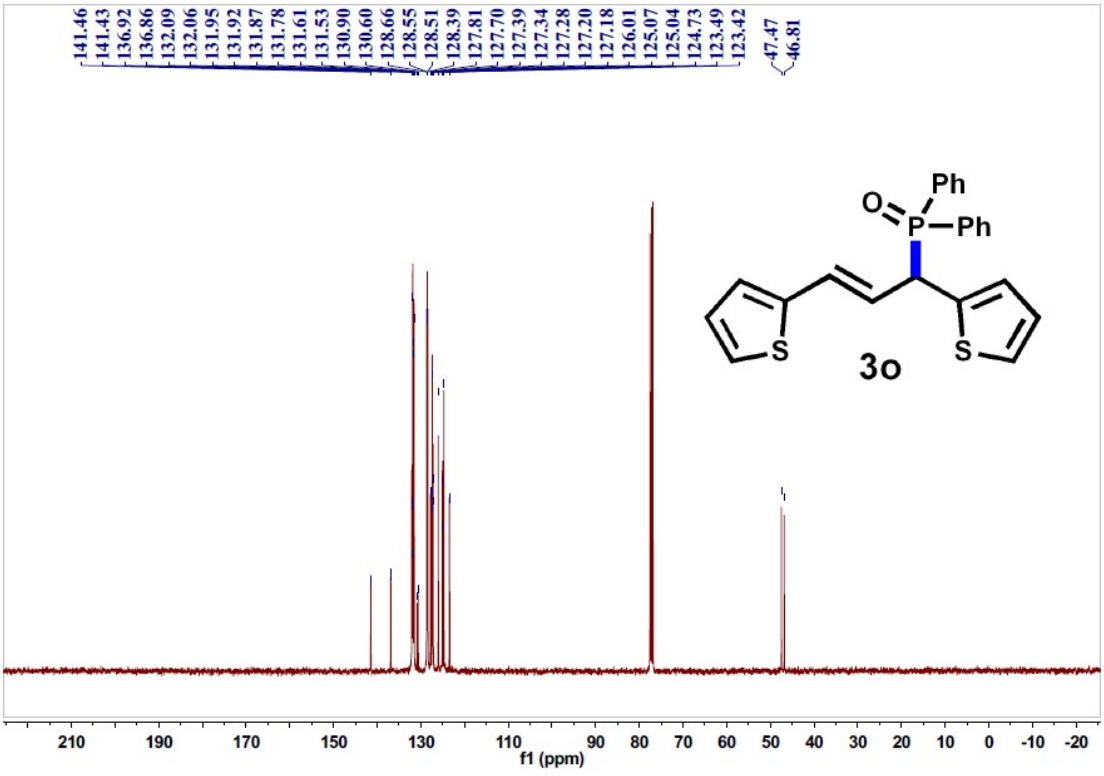
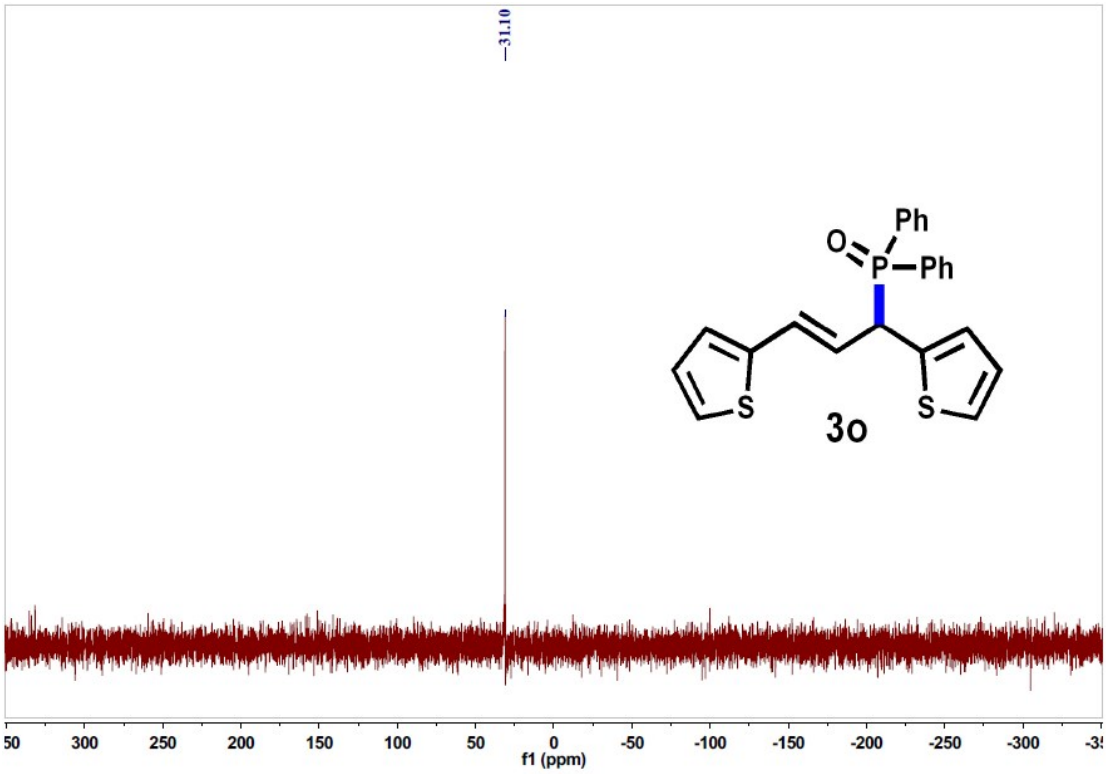
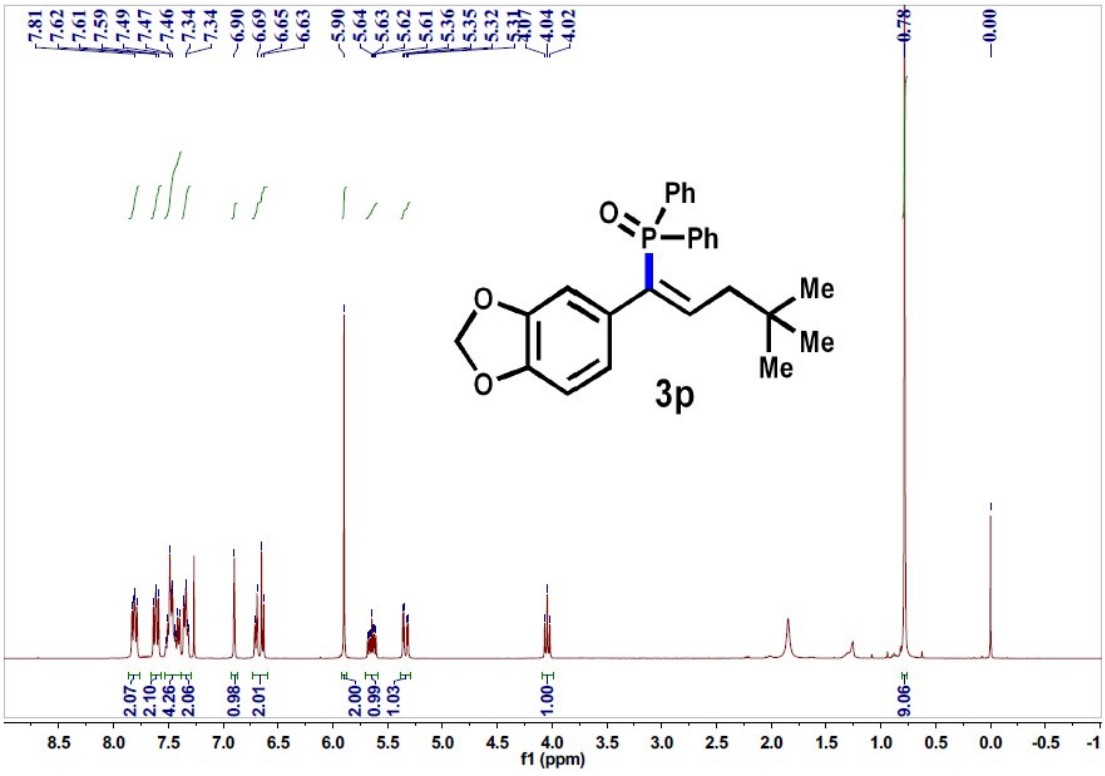
134.64
134.58
133.51
133.25
132.54
131.60
131.42
131.33
131.26
131.18
130.91
130.82
130.28
130.21
128.87
128.53
128.46
128.41
128.28
127.99
127.22
126.43
56.83
56.17
18.20
18.15
Ph
O
P
Ph
Me
3n
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

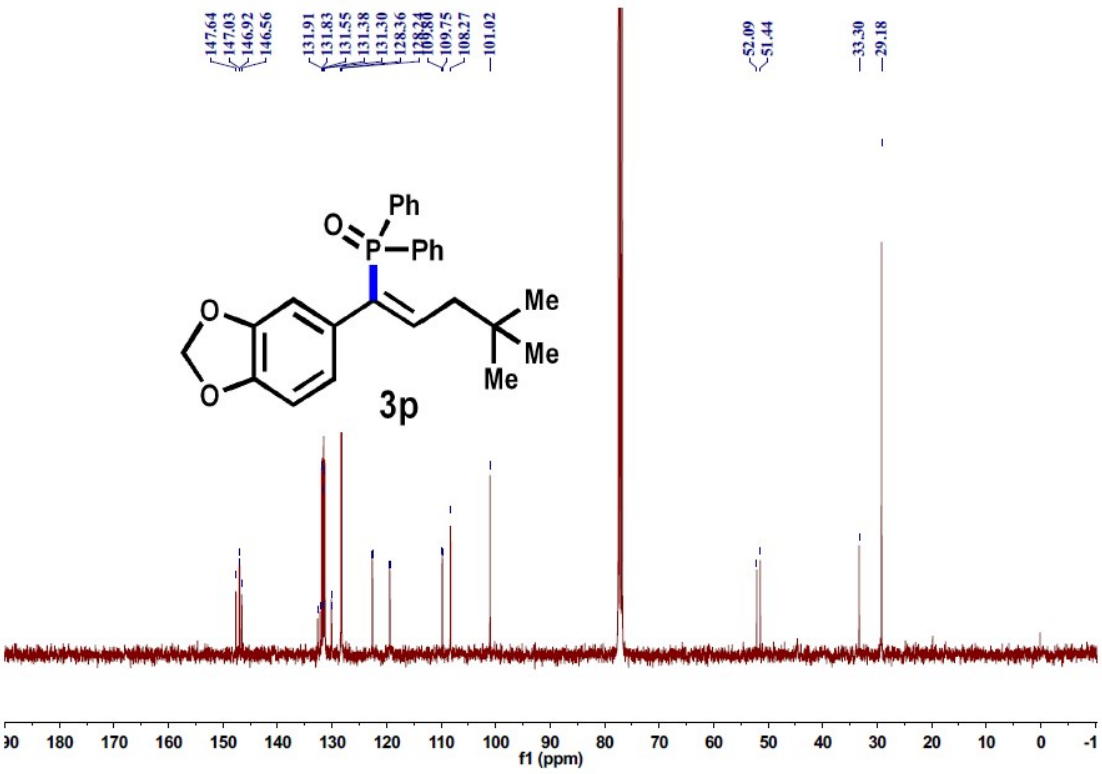

32.31
Ph
O
P
Ph
Me
3n
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -3
f1 (ppm)

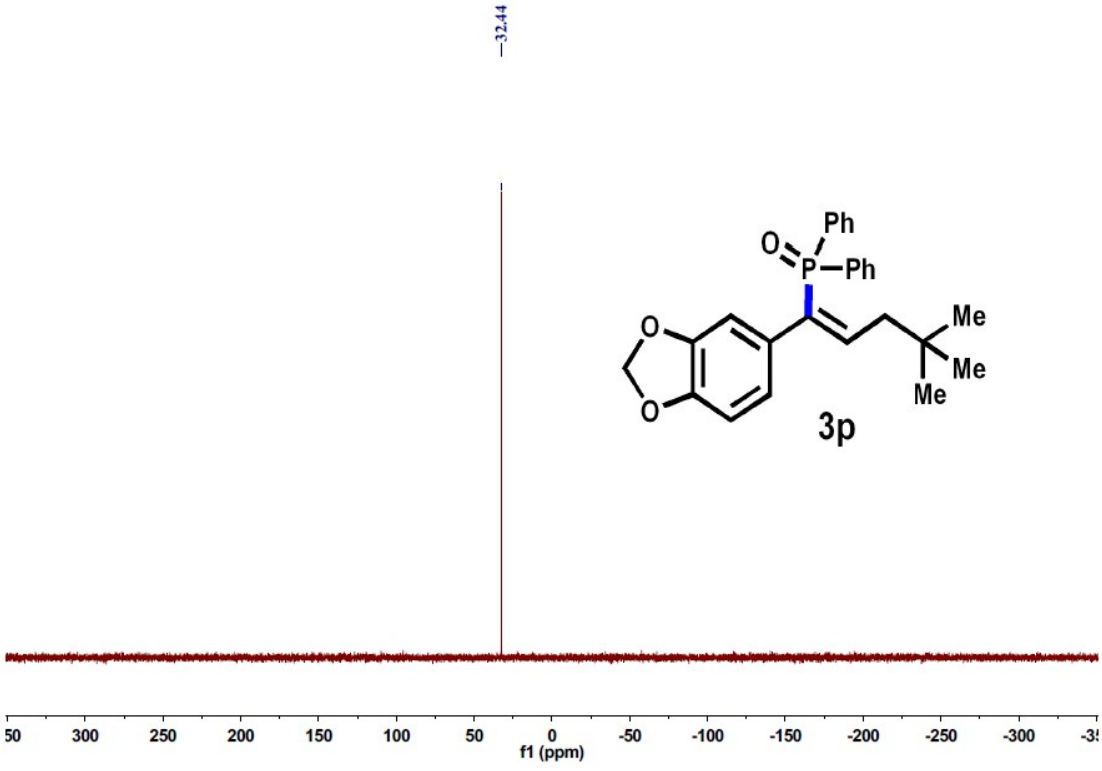

Ph
O
P
Ph
S
S
3o
2.04
2.10
4.27
2.06
1.99
1.00
2.11
1.02
1.01
0.98
1.00
f1 (ppm)

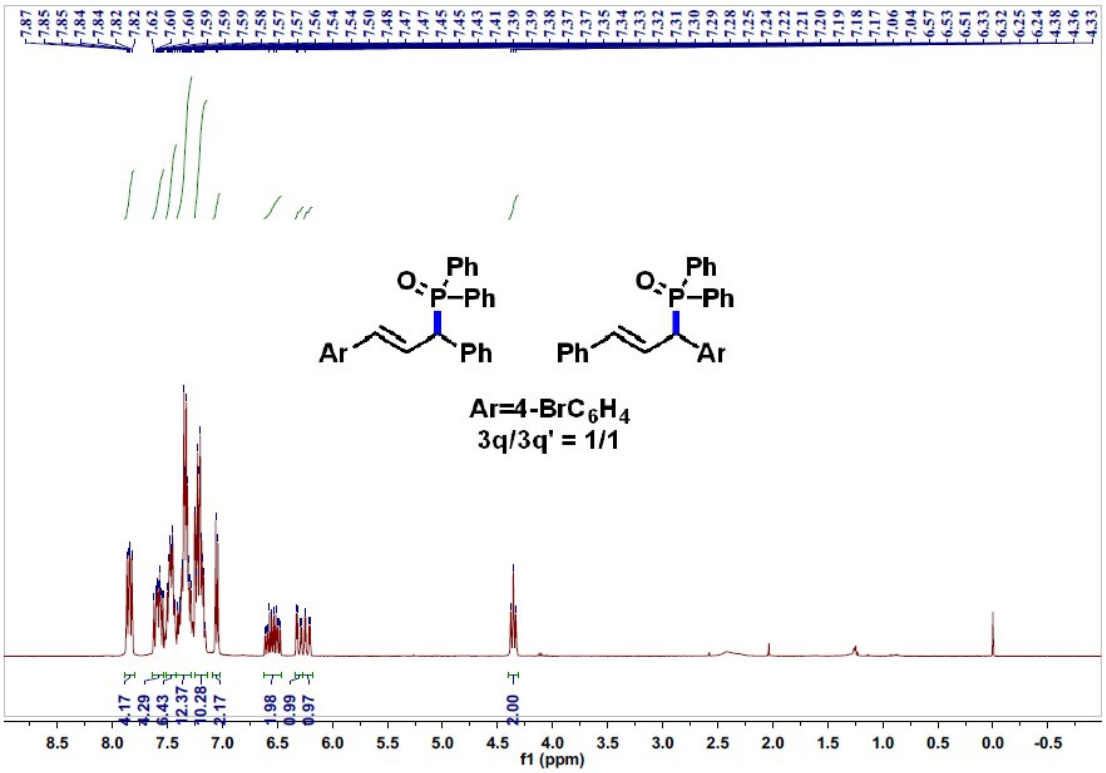

Ph
O
P
Ph
S
S
3o
f1 (ppm)

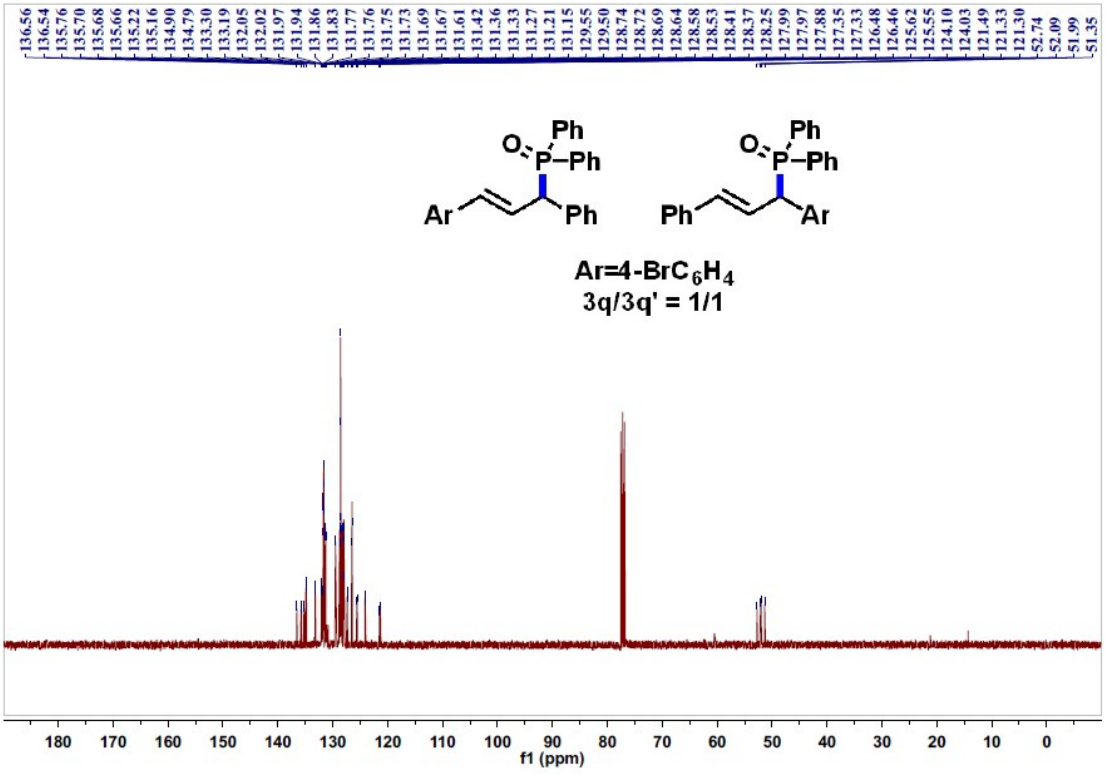

31.10
Ph
O
P
Ph
S
S
3o
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

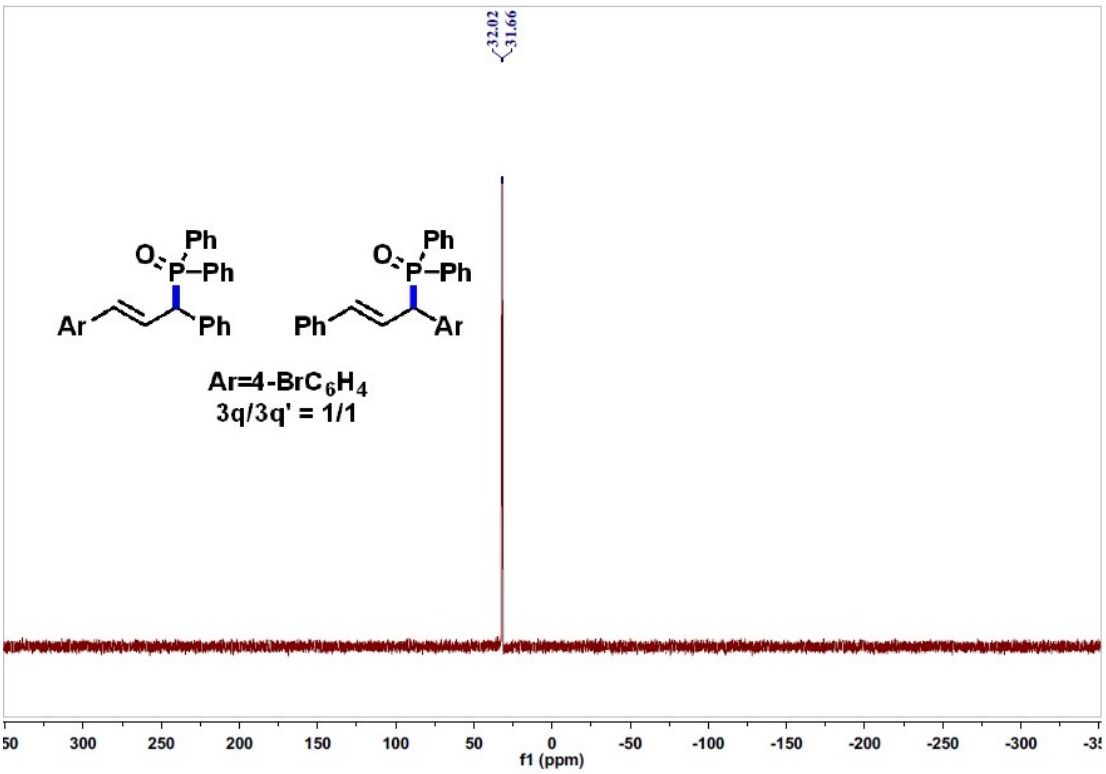

7.81
7.62
7.61
7.59
7.49
7.47
7.46
7.34
7.34
6.90
6.69
6.65
6.63
5.90
5.64
5.63
5.62
5.61
5.36
5.35
5.32
4.07
4.04
4.02
0.78
0.00
Ph
O
P
Ph
Me
Me
Me
O
O
3p
2.07
2.10
4.26
2.06
0.98
2.01
2.00
0.99
1.03
1.00
9.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

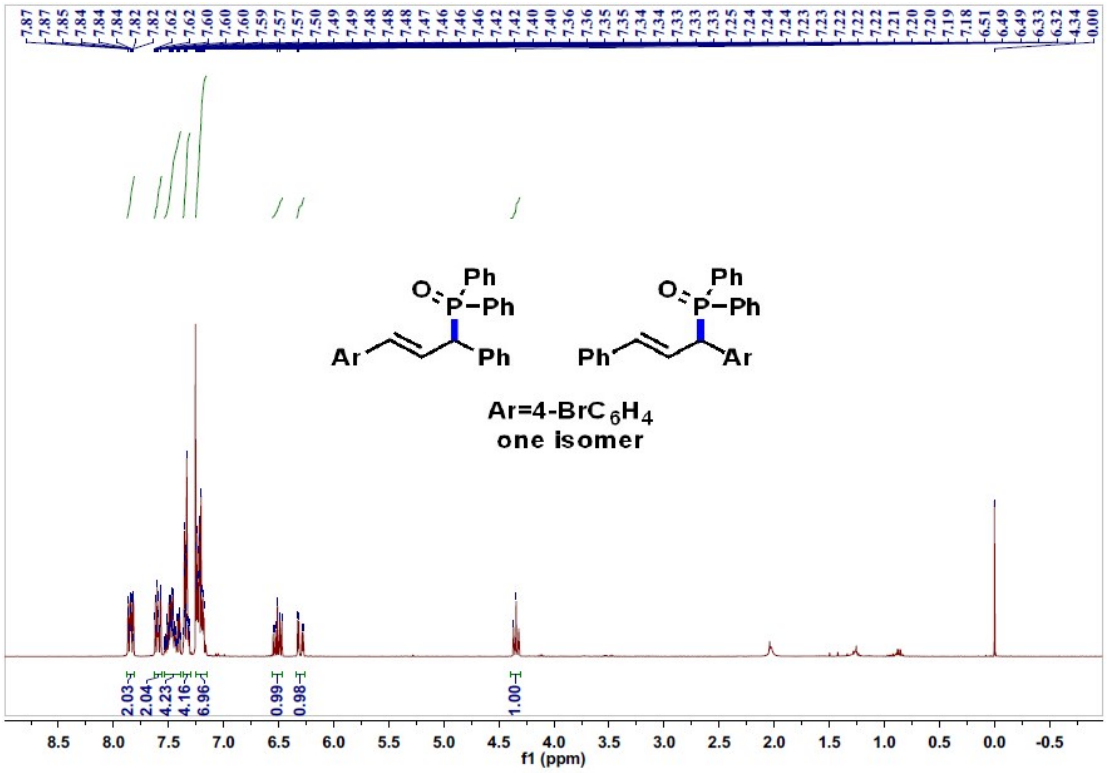

147.64
147.03
146.92
146.56
131.91
131.83
131.55
131.38
131.30
128.36
109.75
108.27
101.02
52.09
51.44
33.30
29.18
Ph
O
P
Ph
Me
Me
Me
O
O
3p
f1 (ppm)

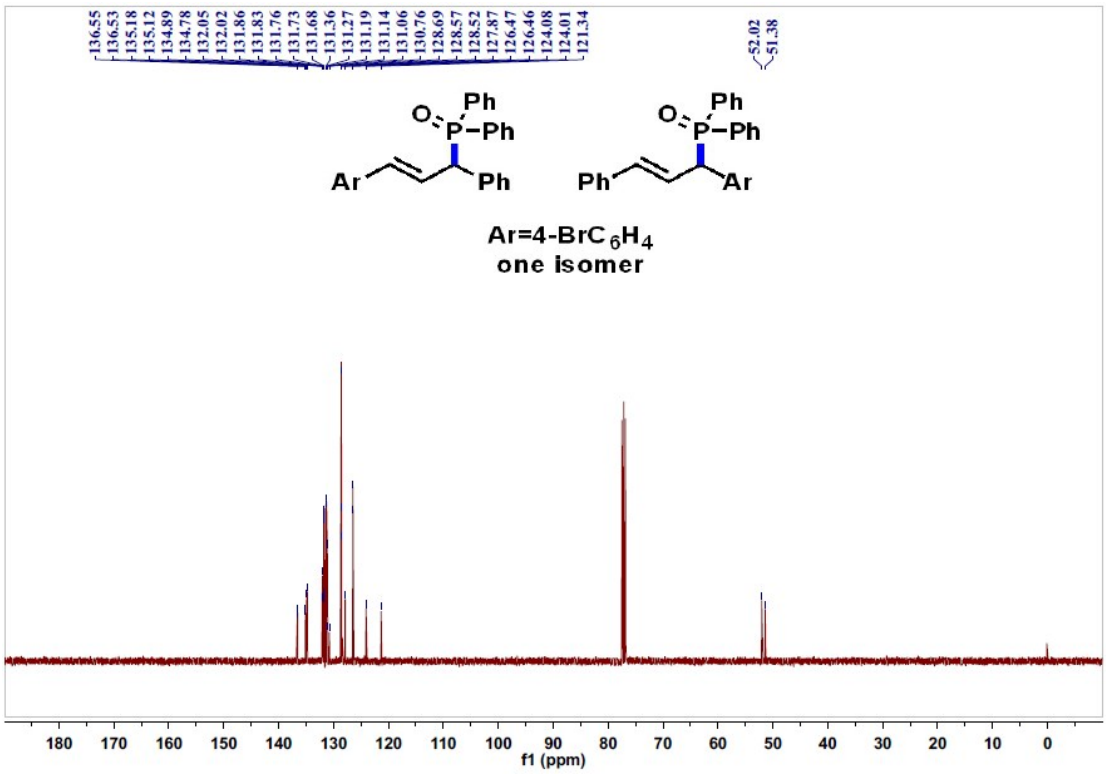

32.44
Ph
O
P
Ph
Me
Me
Me
O
O
3p
f1 (ppm)

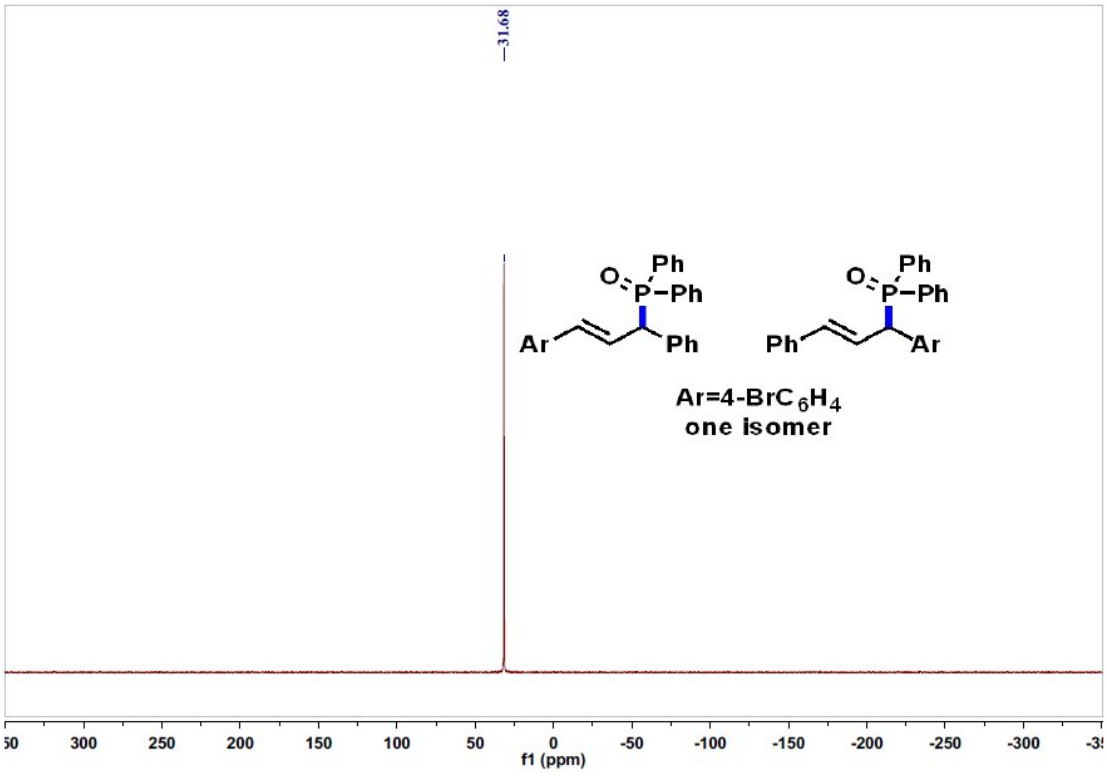

Ar=4-BrC6H4
3q/3q' = 1/1
f1 (ppm)

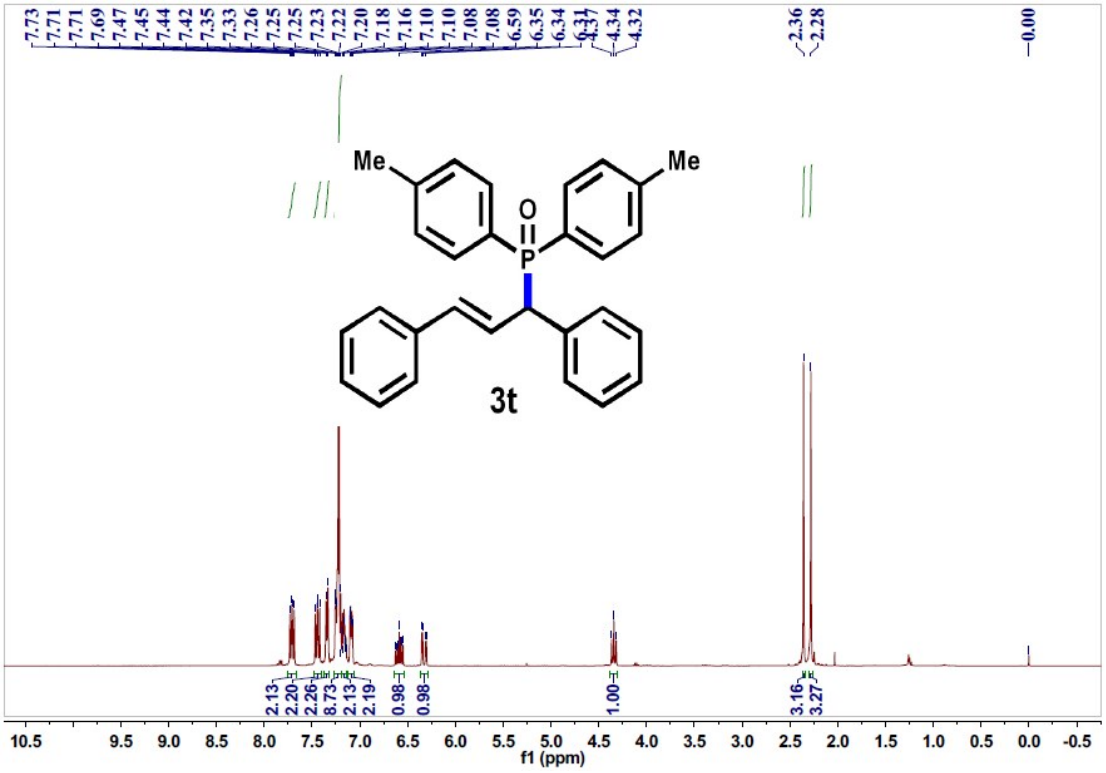

Ar=4-BrC6H4
3q/3q' = 1/1
f1 (ppm)

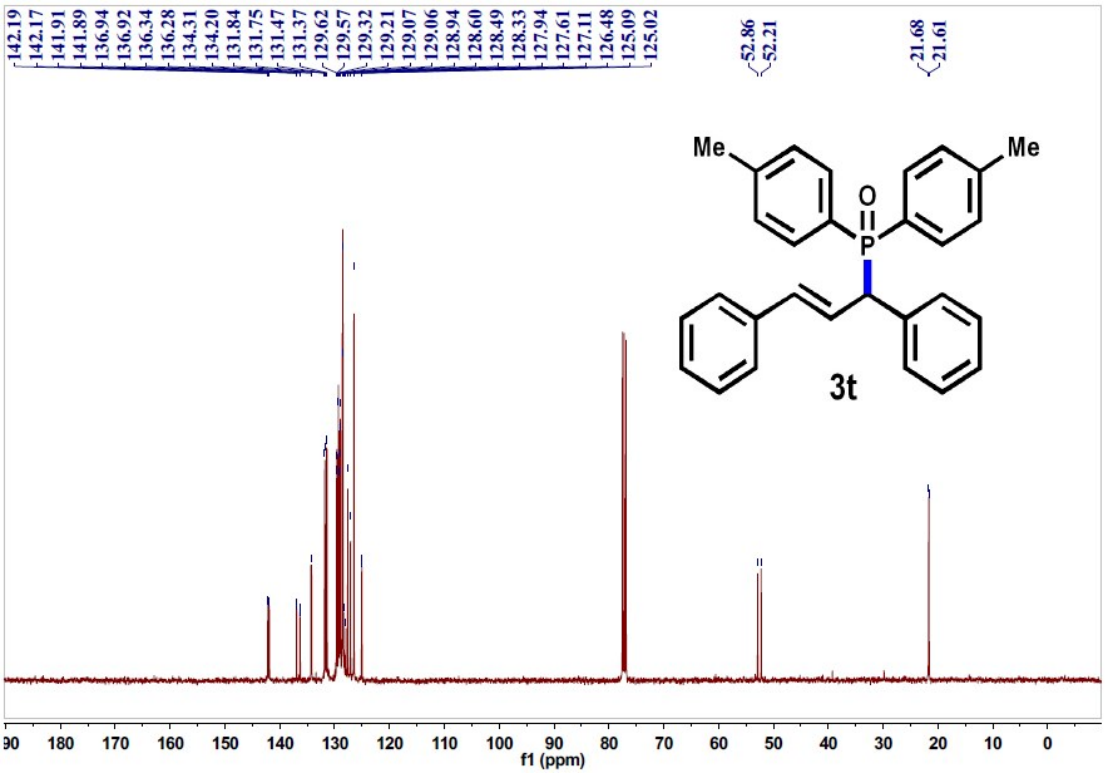

32.02
31.66
Ph
O
P–Ph
Ar
Ph
Ph
O
P–Ph
Ph
Ar
Ar=4-BrC6H4
3q/3q' = 1/1
f1 (ppm)

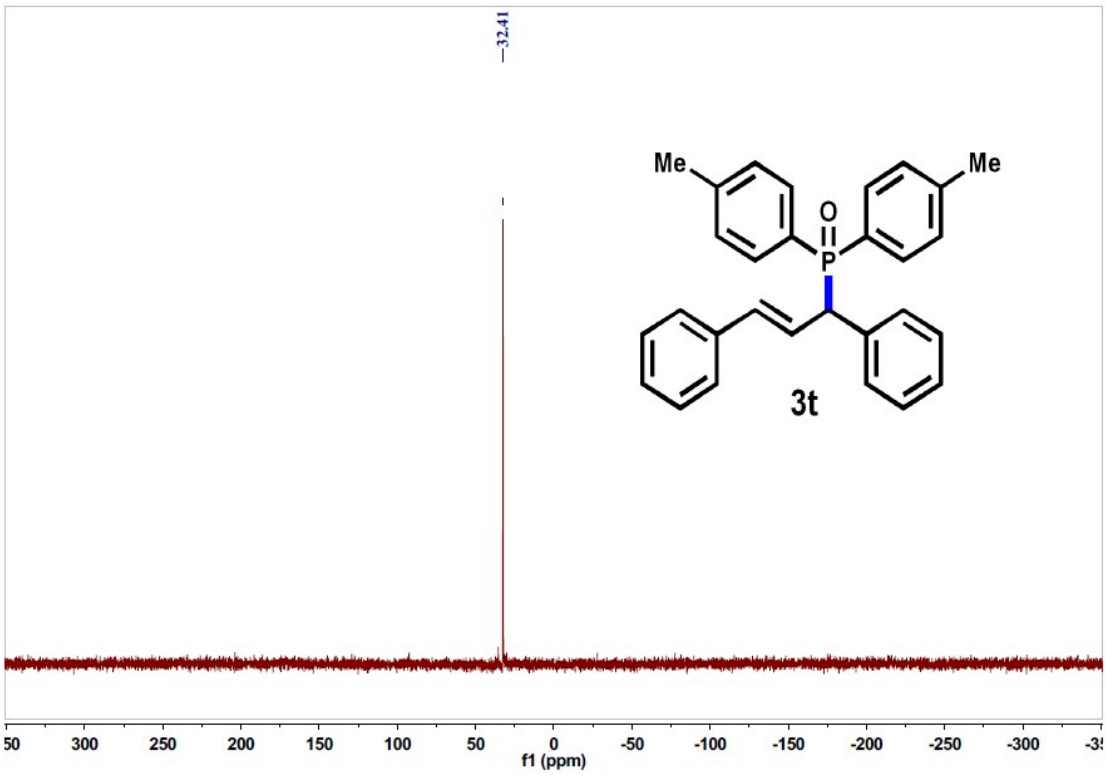

Ph
O
P–Ph
Ar
Ph
Ph
O
P–Ph
Ph
Ar
Ar=4-BrC6H4
one isomer
2.03
2.04
4.23
4.16
6.96
0.99
0.98
1.00
f1 (ppm)

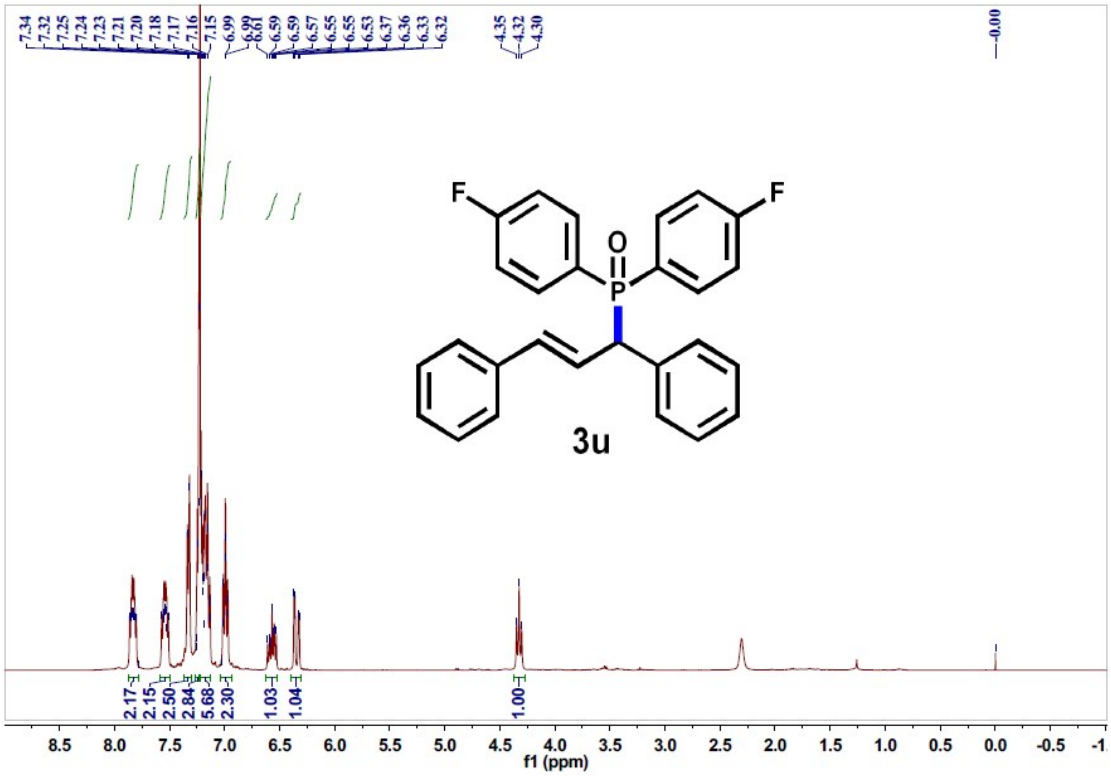

136.55
136.53
135.18
135.12
134.89
134.78
132.05
132.02
131.86
131.83
131.76
131.73
131.68
131.36
131.27
131.19
131.14
131.06
130.76
128.69
128.57
128.52
127.87
126.47
126.46
124.08
124.01
121.34
52.02
51.38
Ph
O
P
Ph
Ar
Ph
Ph
O
P
Ph
Ph
Ar
Ar=4-BrC6H4
one isomer
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

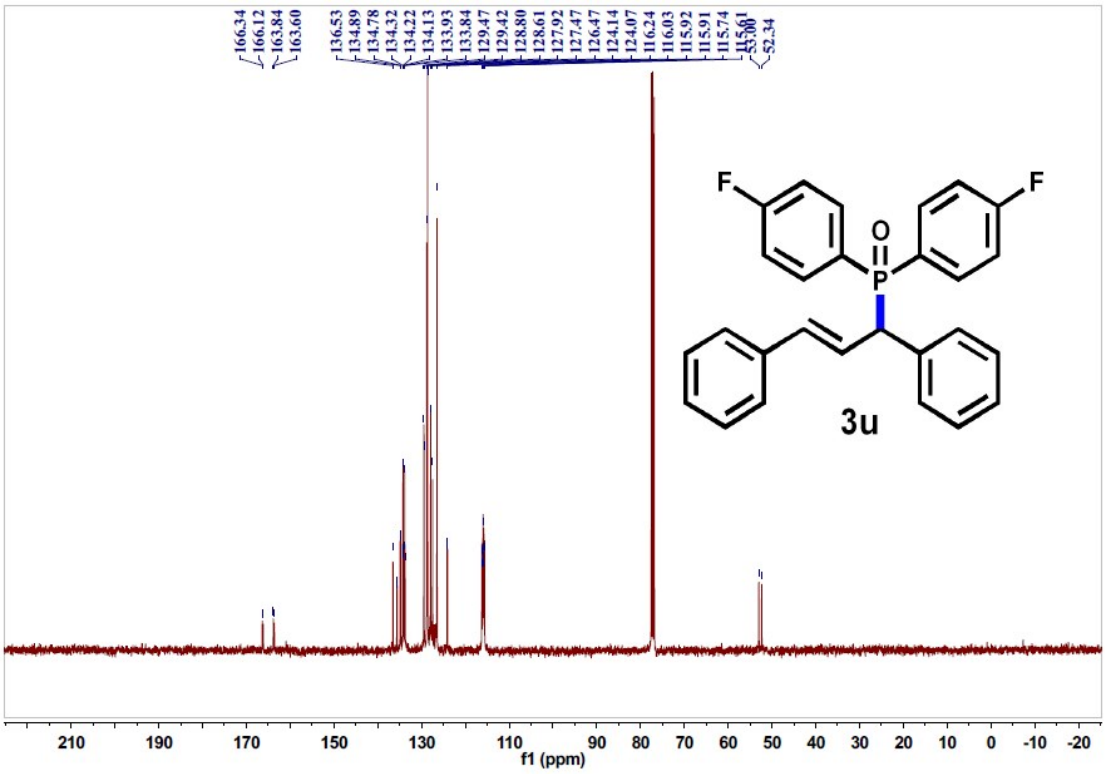

31.68
Ph
O
P
Ph
Ar
Ph
Ph
O
P
Ph
Ph
Ar
Ar=4-BrC6H4
one isomer
300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)

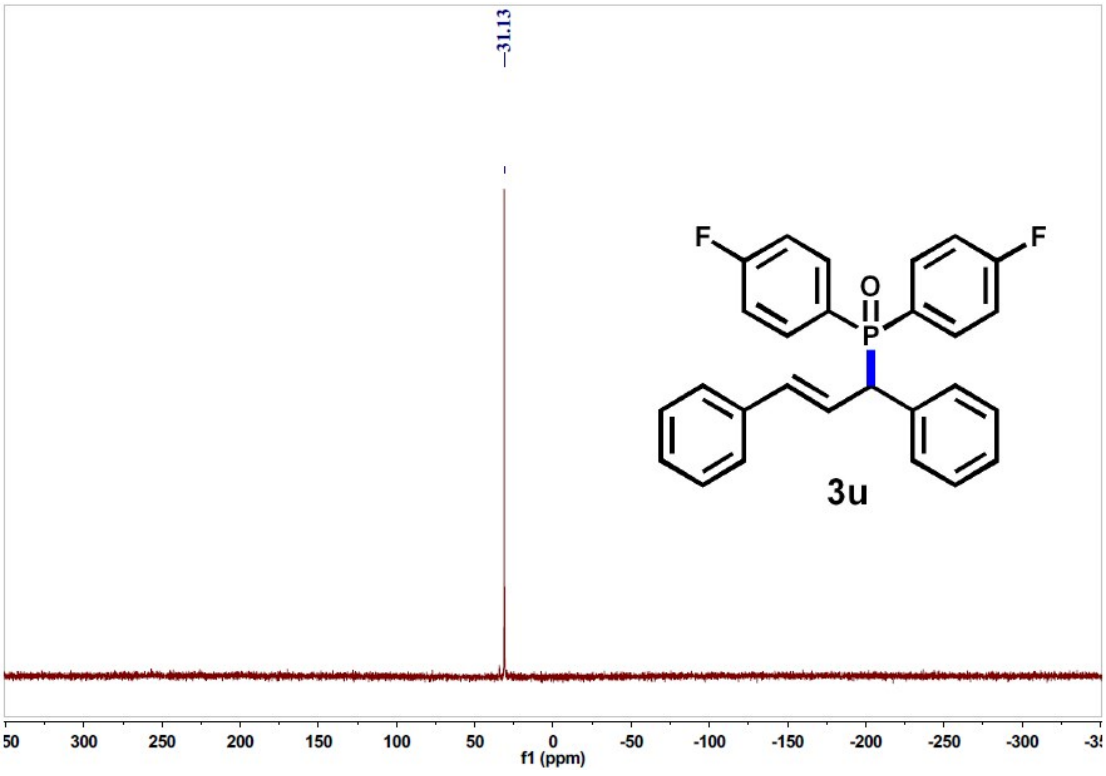

7.73
7.71
7.71
7.69
7.47
7.45
7.44
7.42
7.35
7.33
7.26
7.25
7.25
7.23
7.22
7.20
7.18
7.16
7.10
7.10
7.08
7.08
6.59
6.35
6.34
6.31
4.37
4.34
4.32
2.36
2.28
0.00
Me
Me
O
P
3t
2.13
2.20
2.26
8.73
2.13
2.19
0.98
0.98
1.00
3.16
3.27
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

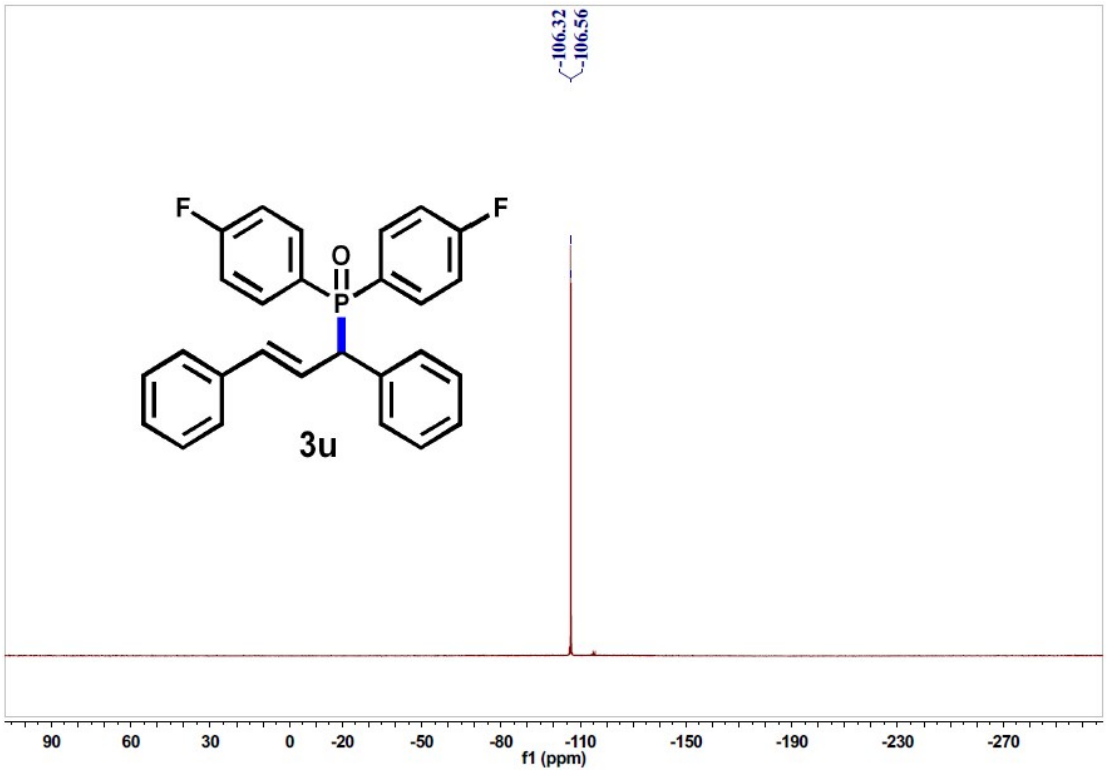

142.19
142.17
141.91
141.89
136.94
136.92
136.34
136.28
134.31
134.20
131.84
131.75
131.47
131.37
129.62
129.57
129.32
129.21
129.07
129.06
128.94
128.60
128.49
128.33
127.94
127.61
127.11
126.48
125.09
125.02
52.86
52.21
21.68
21.61
Me
Me
O
P
3t
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

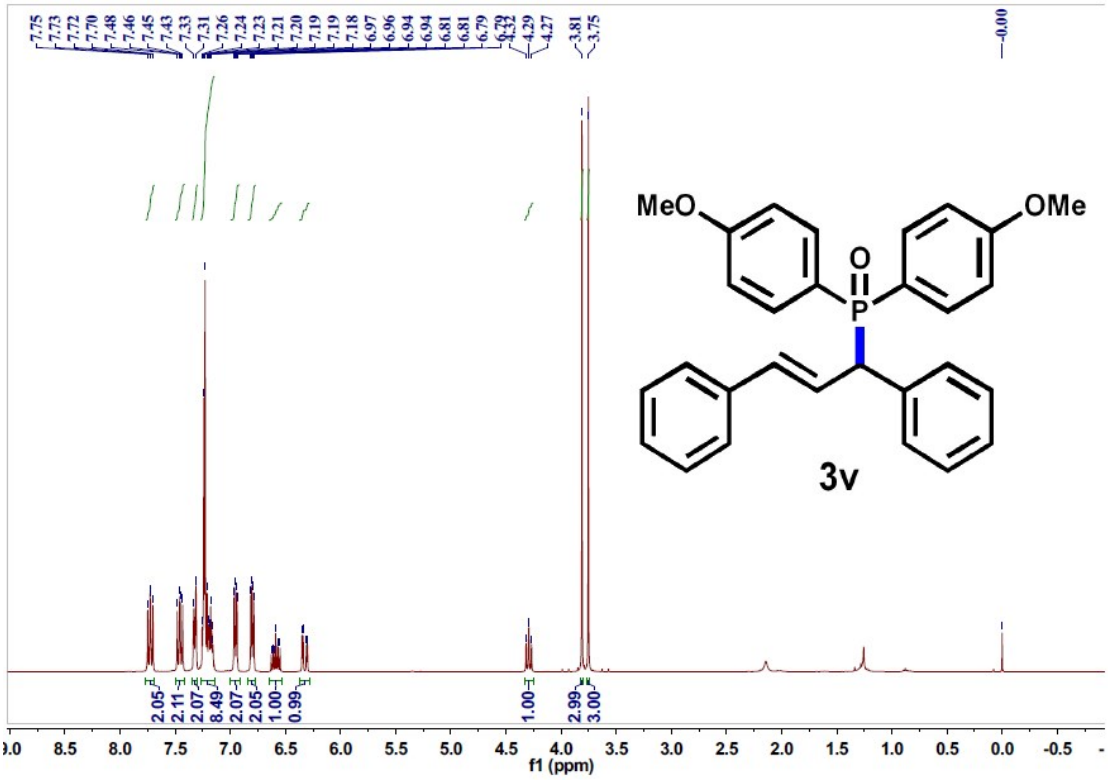

32.41
Me
Me
O
P
3t
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

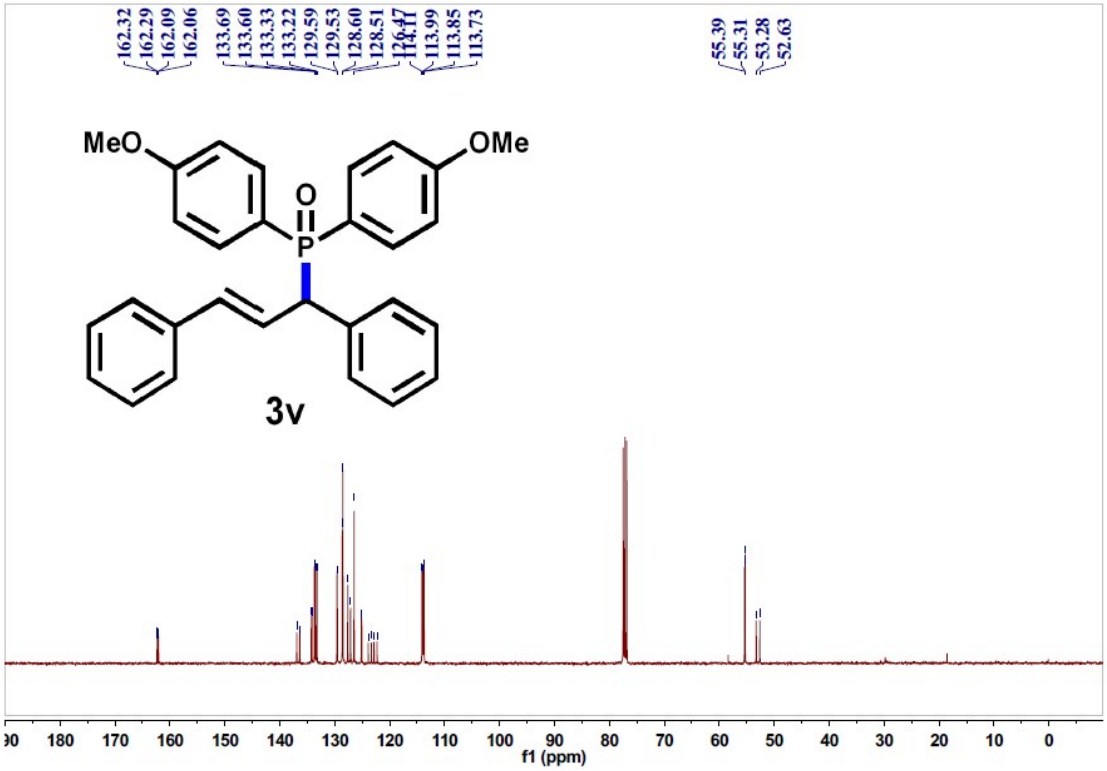

7.34
7.32
7.25
7.24
7.23
7.21
7.20
7.18
7.17
7.16
7.15
6.99
6.98
6.61
6.59
6.59
6.57
6.55
6.55
6.53
6.37
6.36
6.33
6.32
4.35
4.32
4.30
0.00
F
F
O
P
3u
2.17
2.15
2.50
2.84
5.68
2.30
1.03
1.04
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

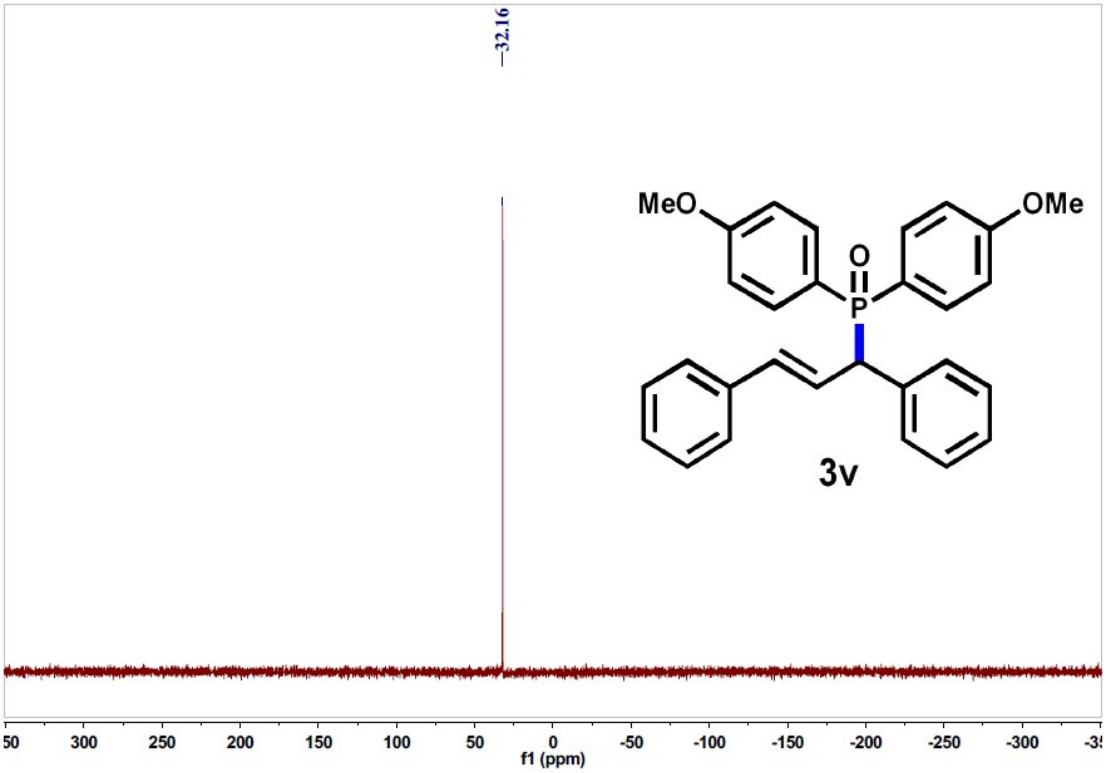

166.34
166.12
163.84
163.60
136.53
134.89
134.78
134.32
134.22
134.13
133.93
133.84
129.47
129.42
128.80
128.61
127.92
127.47
126.47
124.14
124.07
116.24
116.03
115.92
115.91
115.74
53.00
52.34
F
F
O
P
3u
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

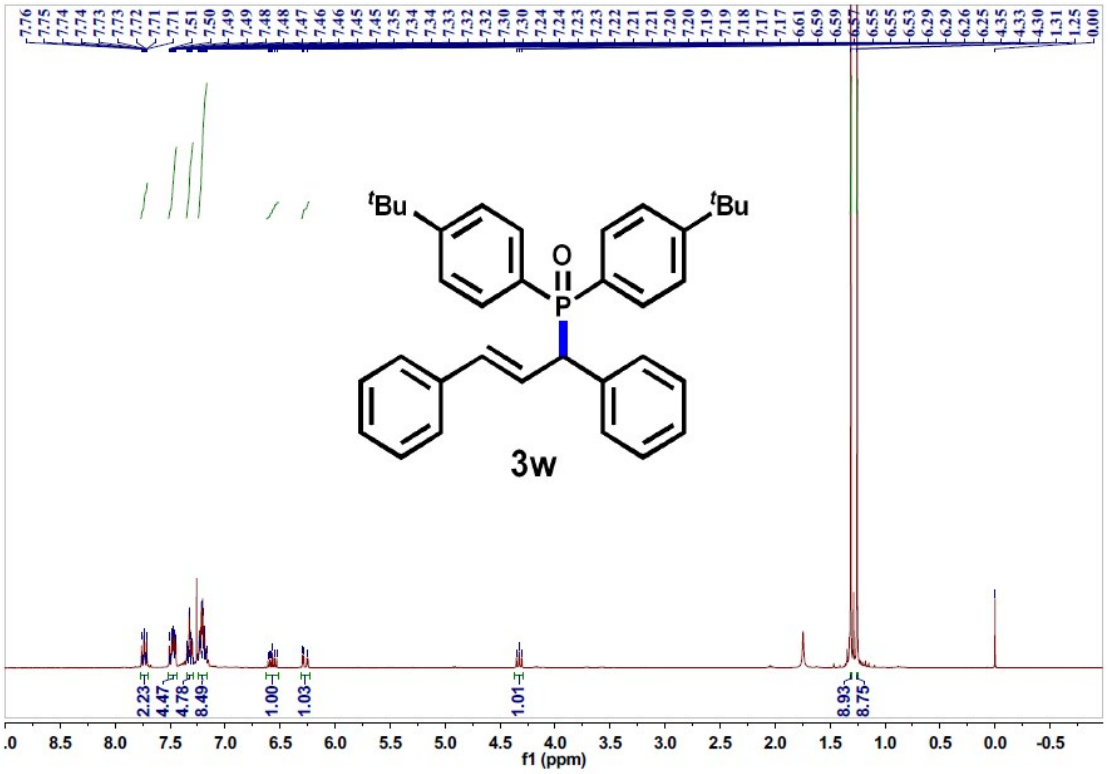

31.13
F
F
O
P
3u
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

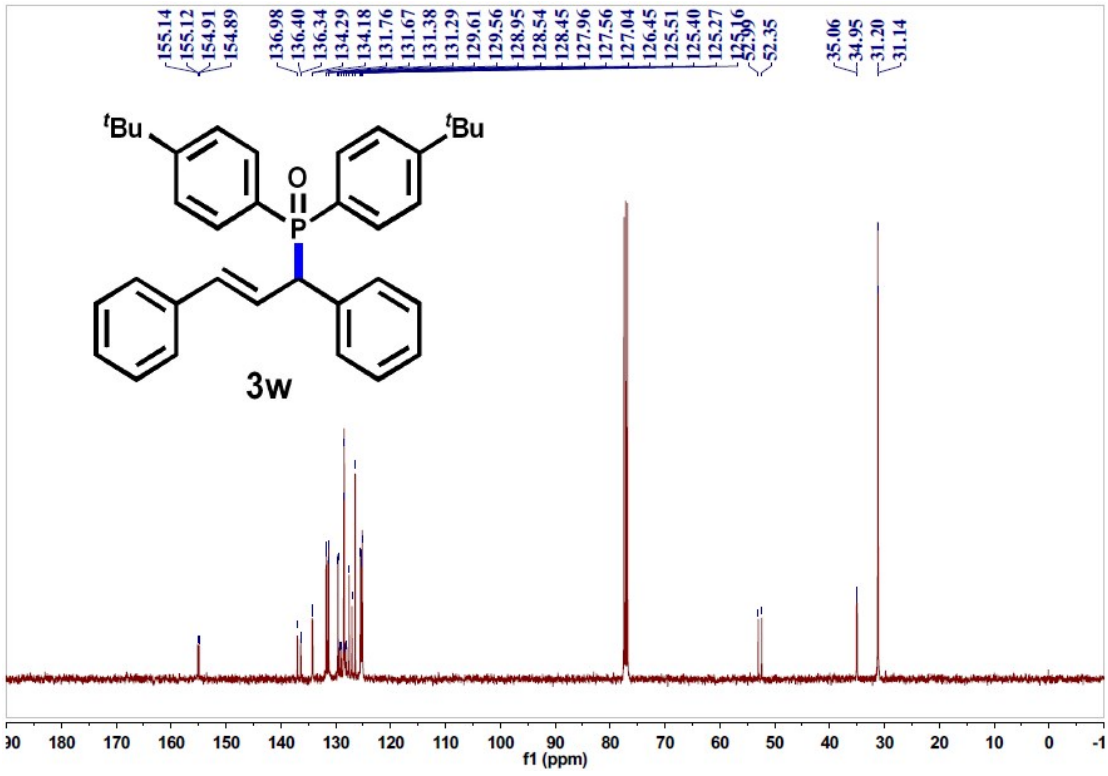

106.32
106.56
F
F
O
P
3u
90
60
30
0
-20
-50
-80
-110
-150
-190
-230
-270
f1 (ppm)

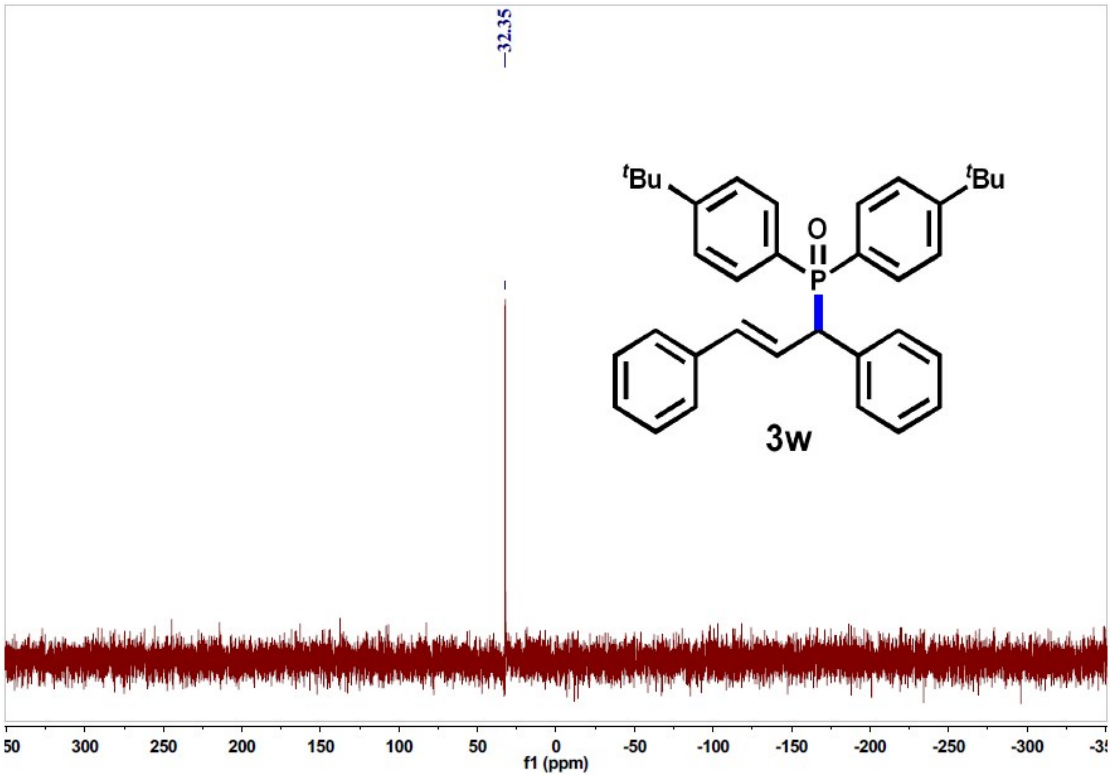

7.75
7.73
7.72
7.70
7.48
7.46
7.45
7.43
7.33
7.31
7.26
7.24
7.23
7.21
7.20
7.19
7.19
7.18
6.97
6.96
6.94
6.94
6.81
6.81
6.79
4.29
4.27
3.81
3.75
0.00
MeO
OMe
O
P
3v
2.05
2.11
2.07
8.49
2.07
2.05
1.00
0.99
1.00
2.99
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

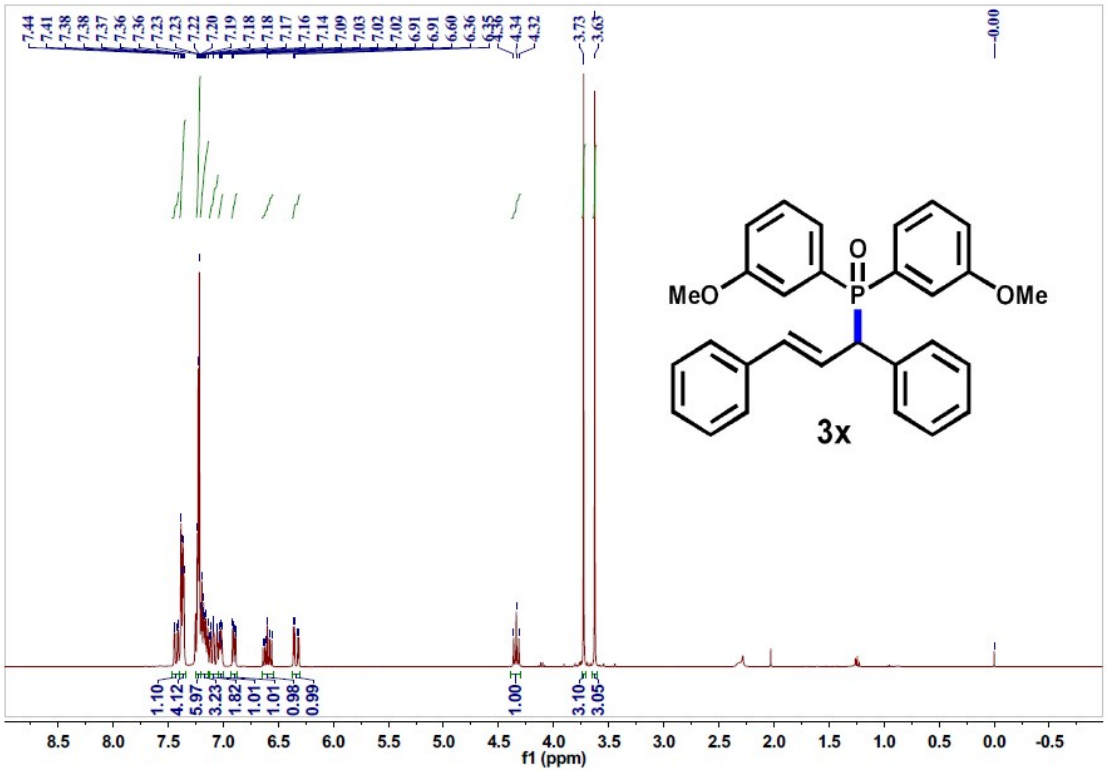

162.32
162.29
162.09
162.06
133.69
133.60
133.33
133.22
129.59
129.53
128.60
128.51
126.47
114.11
113.99
113.85
113.73
55.39
55.31
53.28
52.63
MeO
OMe
O
P
3v
f1 (ppm)

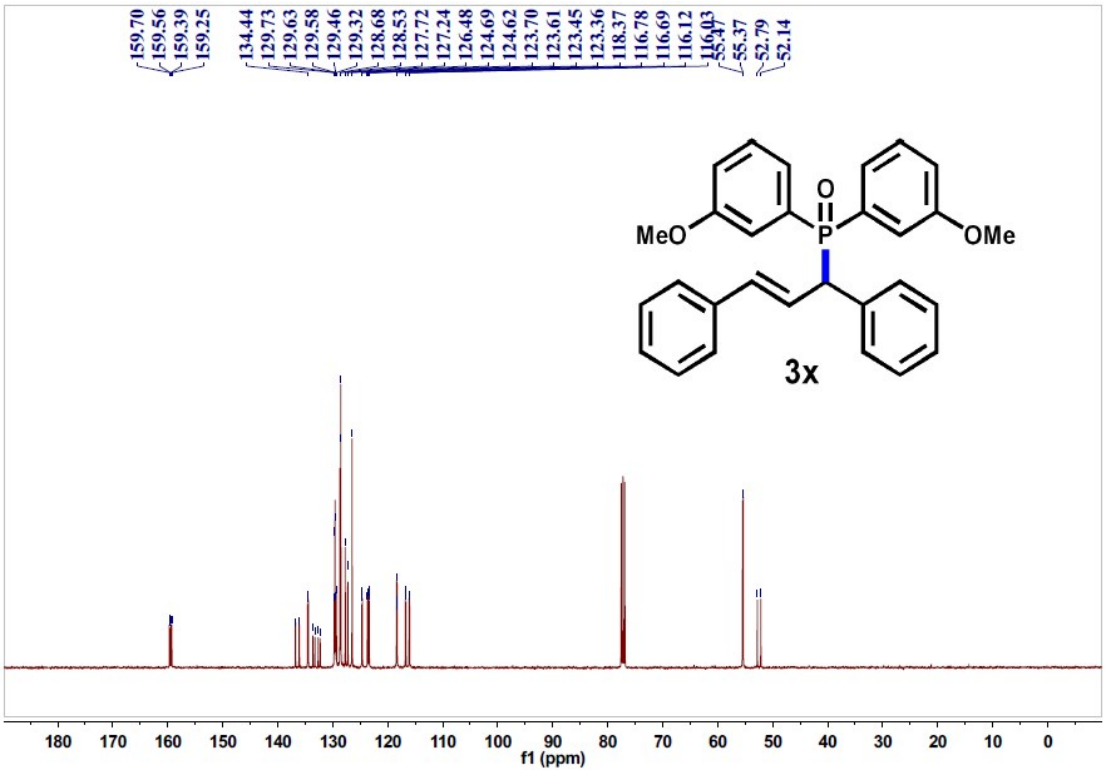

32.16
MeO
OMe
O
P
3v
f1 (ppm)

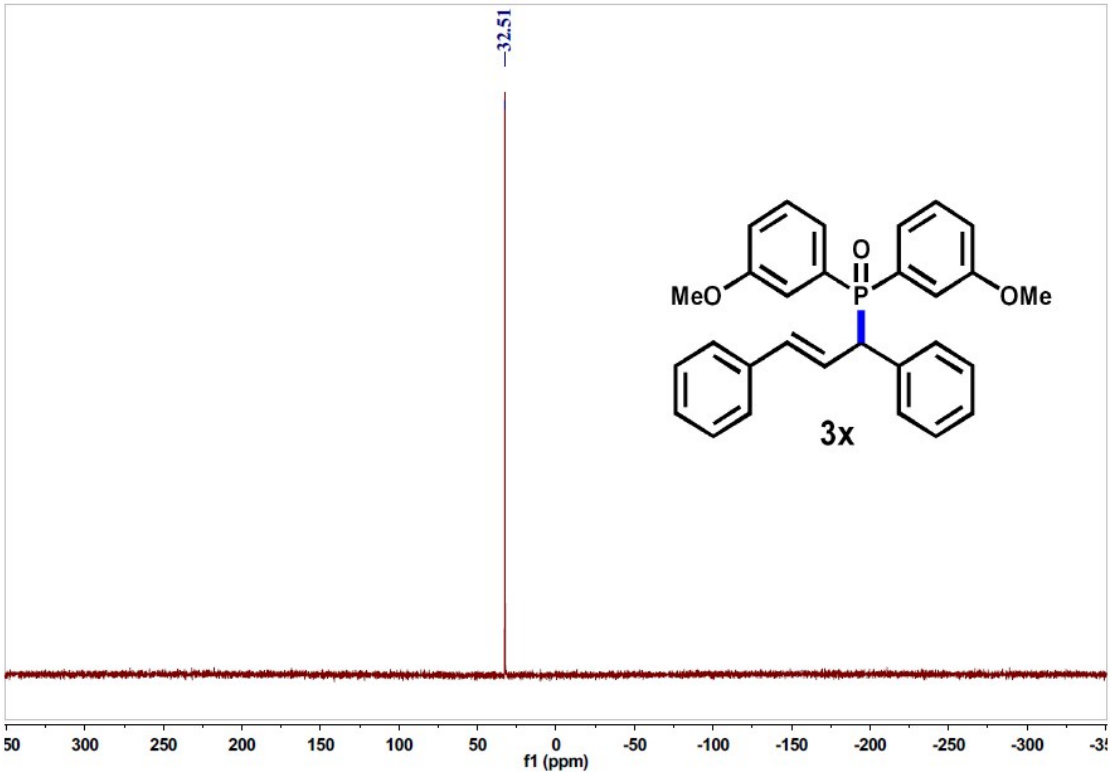

tBu
tBu
O
P
3w
f1 (ppm)

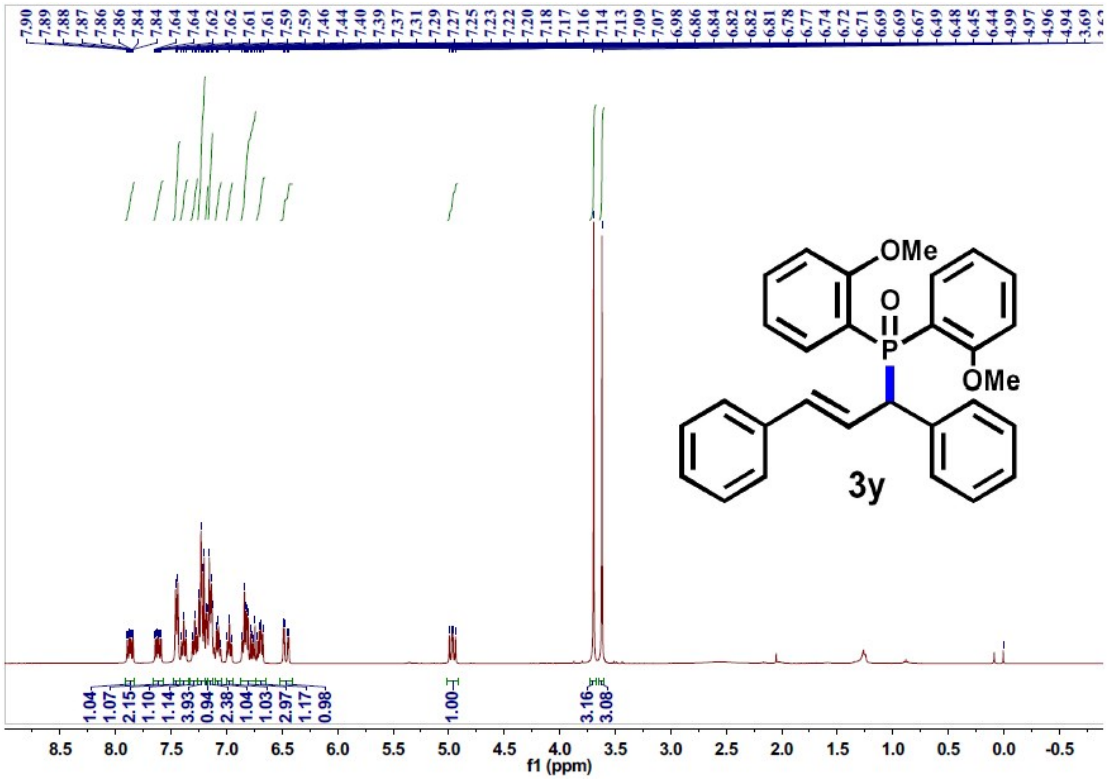

tBu
tBu
O
P
3w
f1 (ppm)

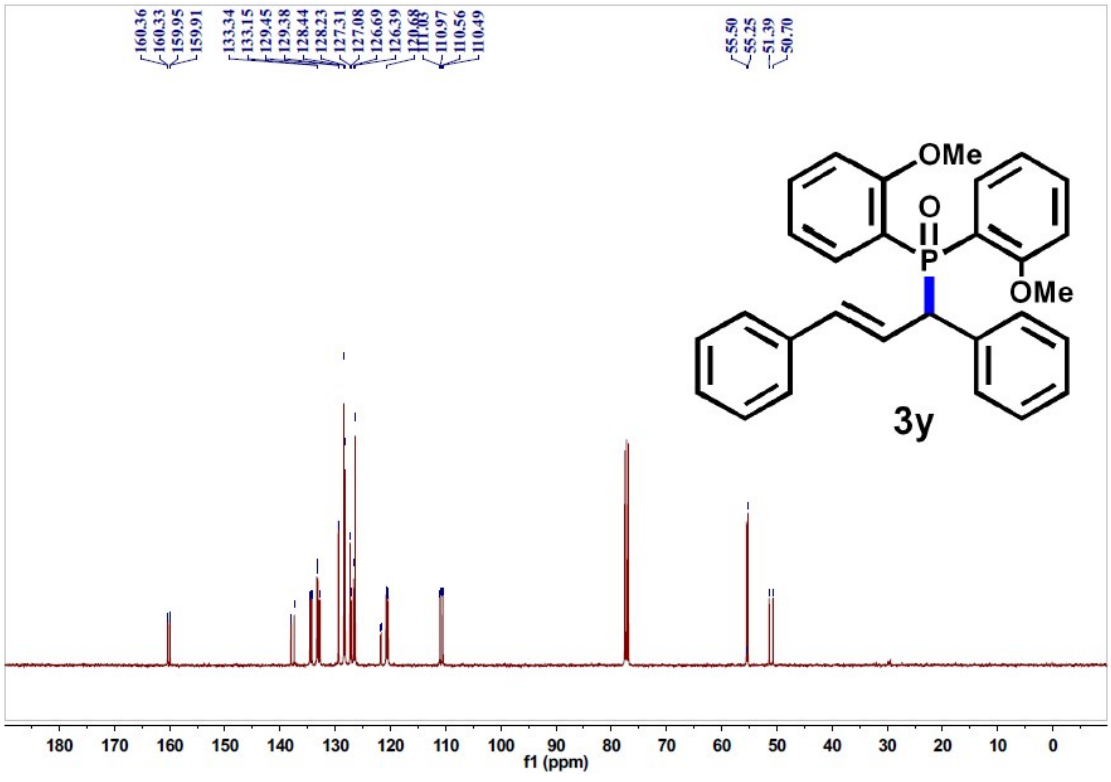

32.35
tBu
tBu
O
P
3w
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

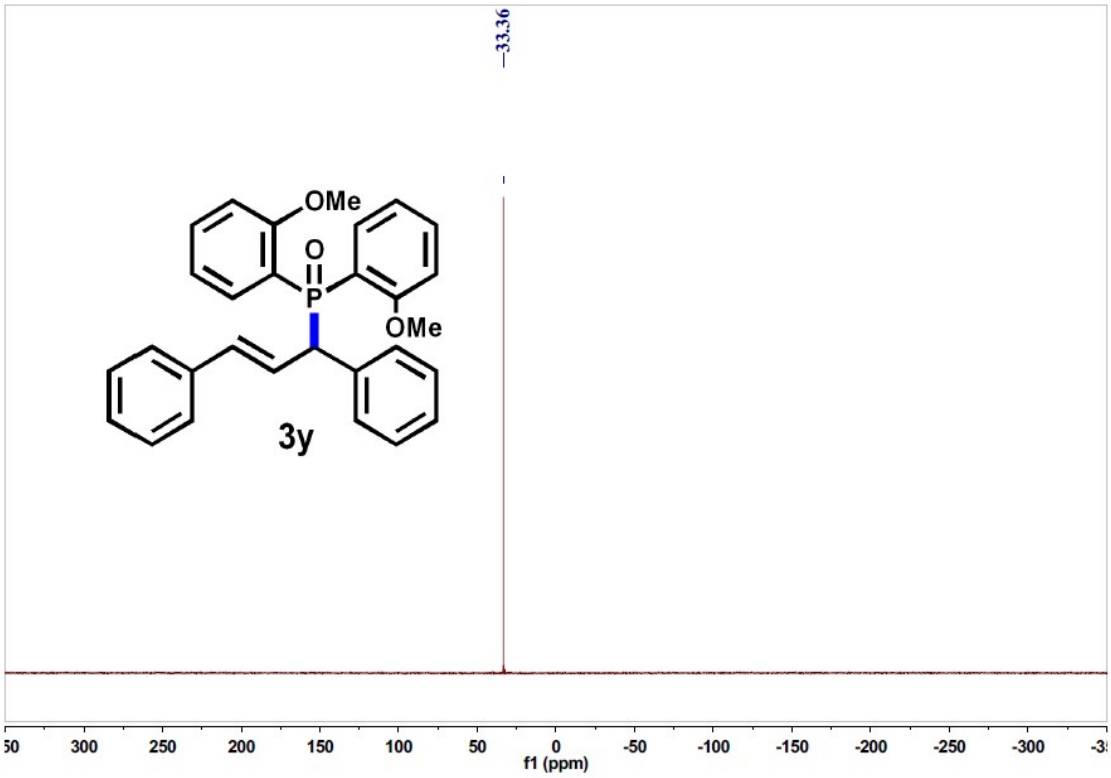

7.44
7.41
7.38
7.38
7.37
7.36
7.36
7.23
7.23
7.22
7.20
7.19
7.18
7.18
7.17
7.16
7.14
7.09
7.03
7.02
7.02
6.91
6.91
6.60
6.36
6.35
6.35
4.34
4.32
3.73
3.63
0.00
MeO
O
P
OMe
3x
1.10
4.12
5.97
3.23
1.82
1.01
1.01
0.98
0.99
1.00
3.10
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

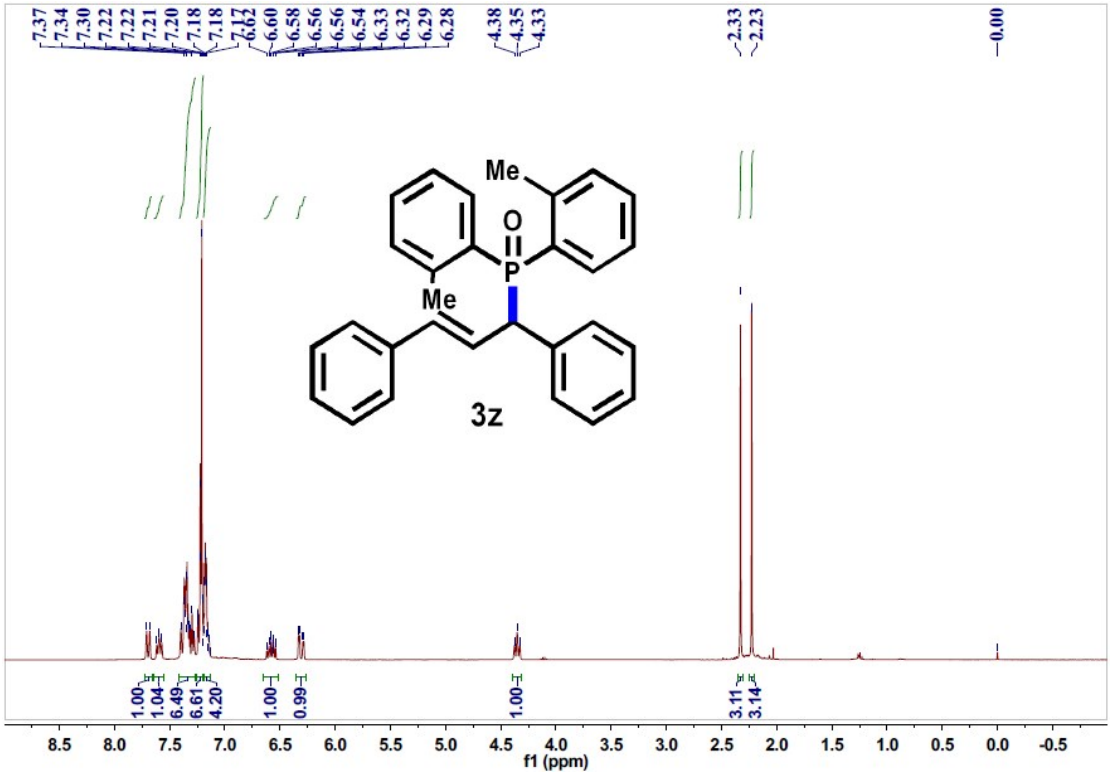

159.70
159.56
159.39
159.25
134.44
129.73
129.63
129.58
129.46
129.32
128.68
128.53
127.72
127.24
126.48
124.69
124.62
123.70
123.61
123.45
123.36
118.37
116.78
116.69
116.12
116.03
55.47
55.37
52.79
52.14
MeO
O
P
OMe
3x
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

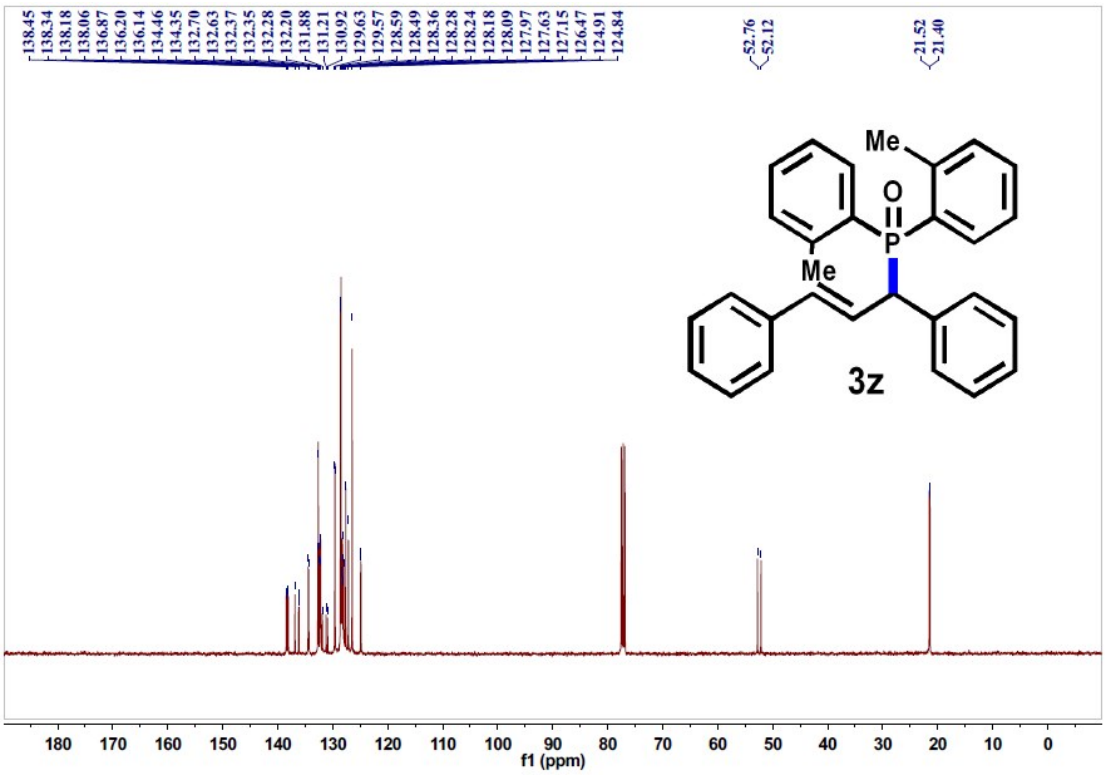

32.51
MeO
O
P
OMe
3x
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)

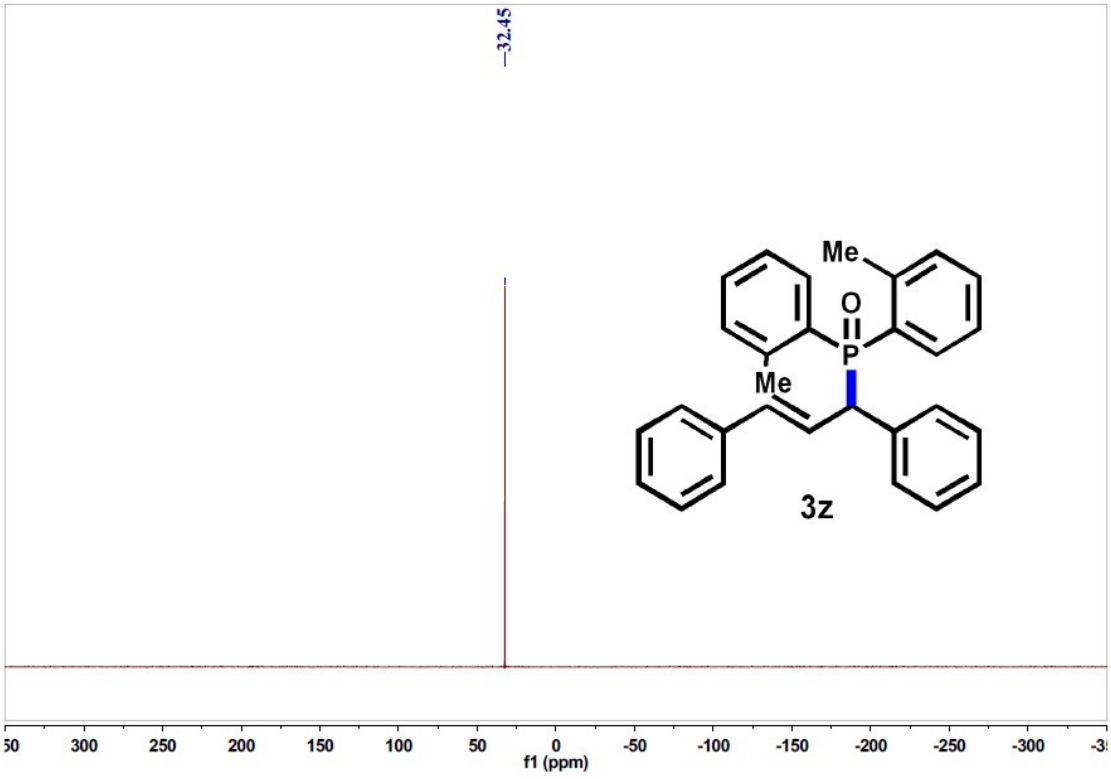

OMe
O
P
OMe
3y
f1 (ppm)

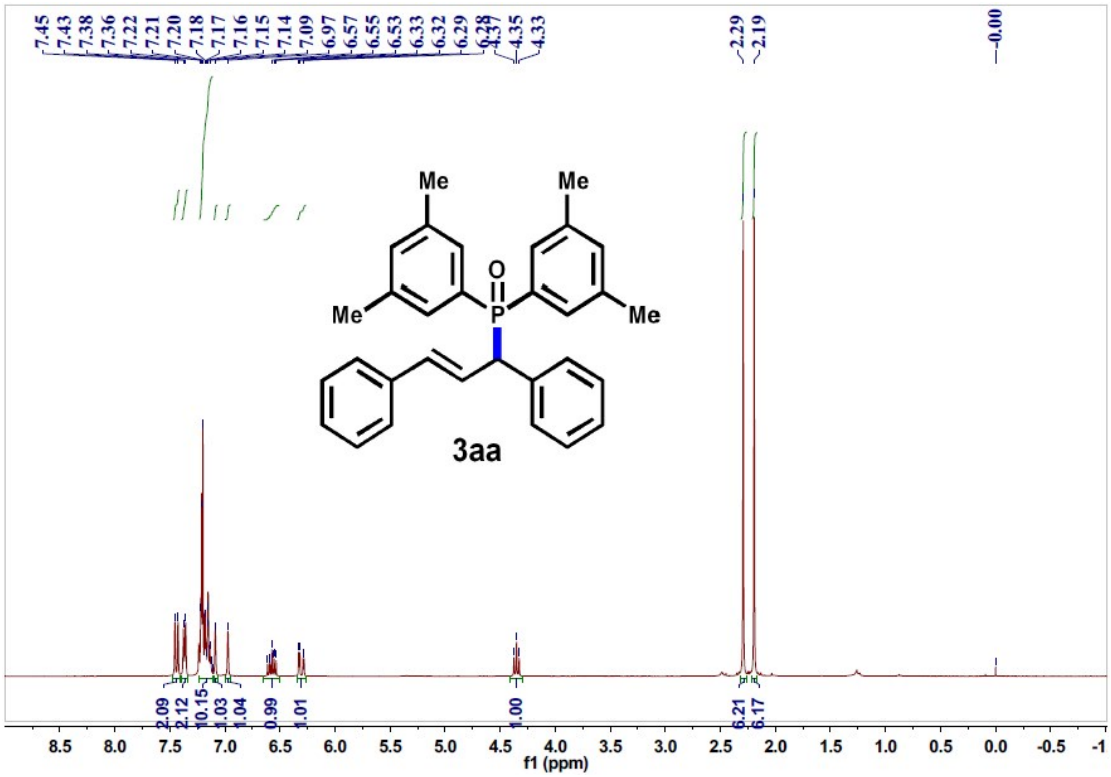

OMe
O
P
OMe
3y
f1 (ppm)

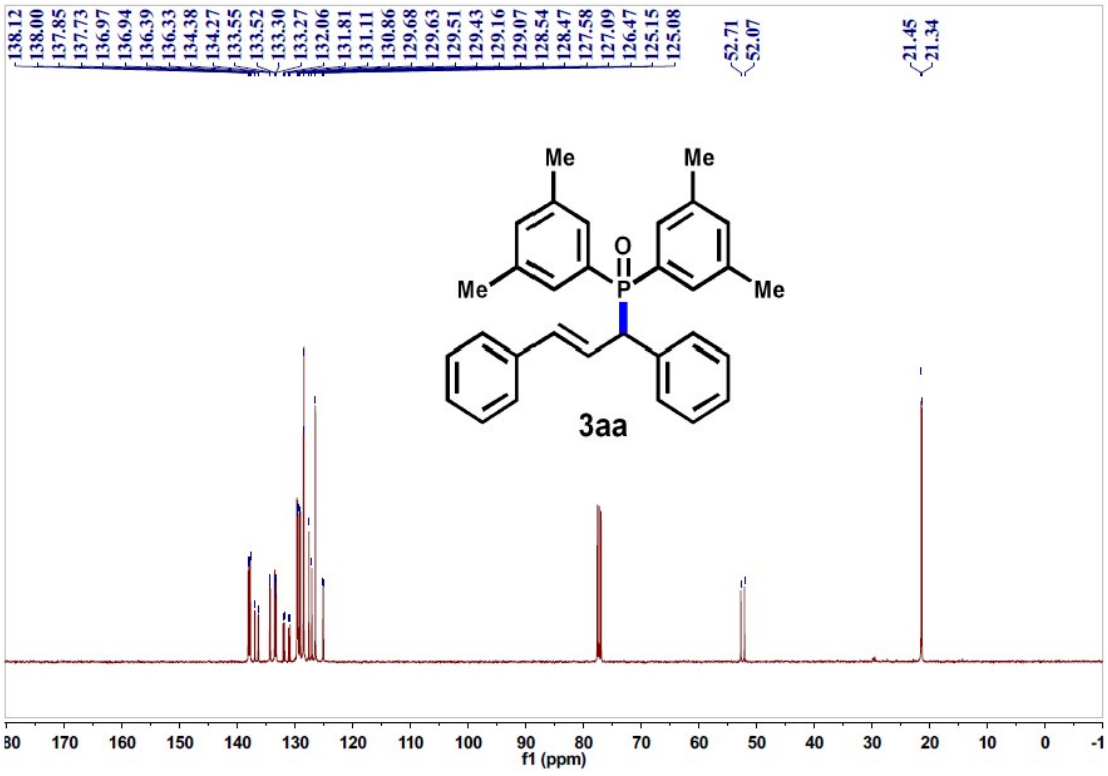

33.36
OMe
O
P
OMe
3y
f1 (ppm)

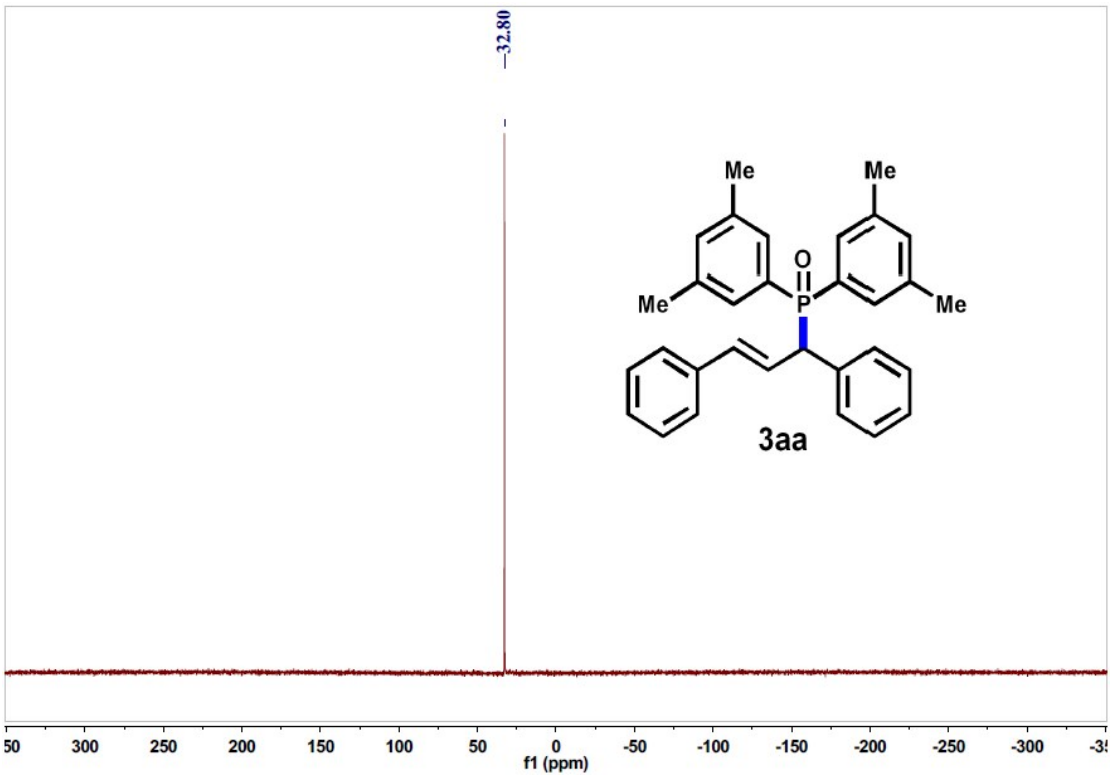

7.37
7.34
7.30
7.22
7.22
7.21
7.20
7.18
7.18
6.62
6.60
6.58
6.56
6.56
6.54
6.33
6.32
6.29
6.28
4.38
4.35
4.33
2.33
2.23
0.00
Me
O
P
Me
3z
1.00
1.04
6.49
6.61
4.20
1.00
0.99
1.00
3.11
3.14
f1 (ppm)

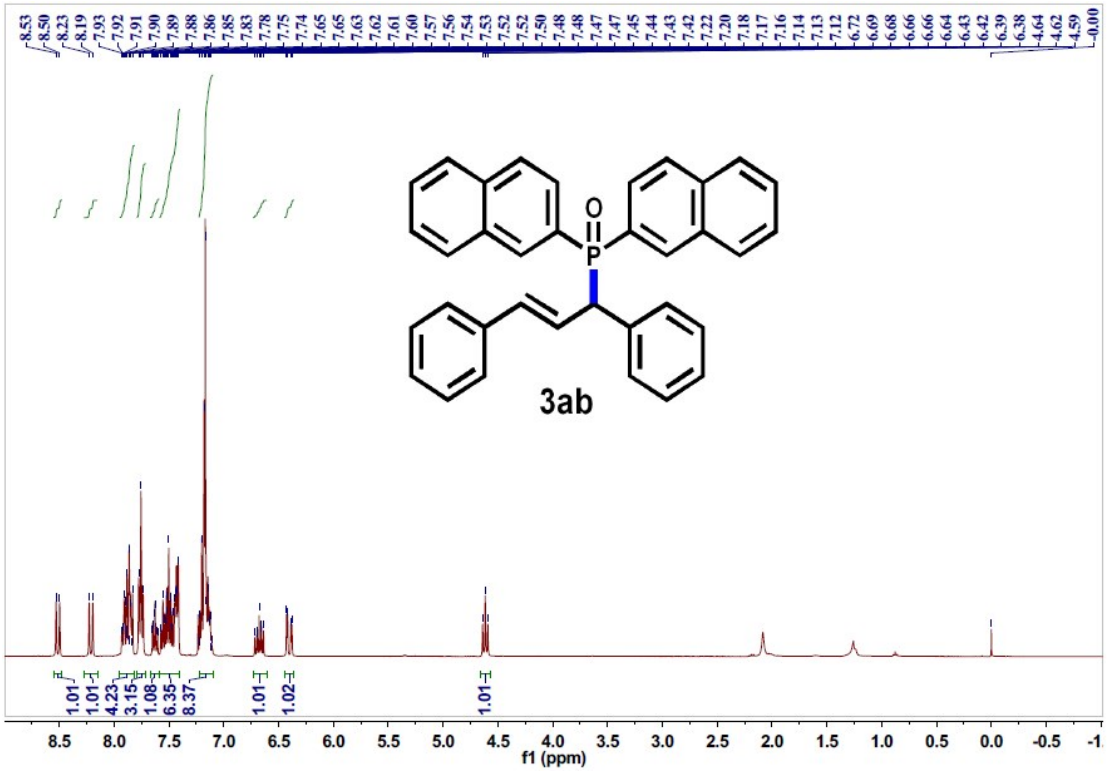

138.45
138.34
138.18
138.06
136.87
136.20
136.14
134.46
134.35
132.70
132.63
132.37
132.35
132.28
132.20
131.88
131.21
130.92
129.63
129.57
128.59
128.49
128.36
128.28
128.24
128.18
128.09
127.97
127.63
127.15
126.47
124.91
124.84
52.76
52.12
21.52
21.40
Me
O
P
Me
3z
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

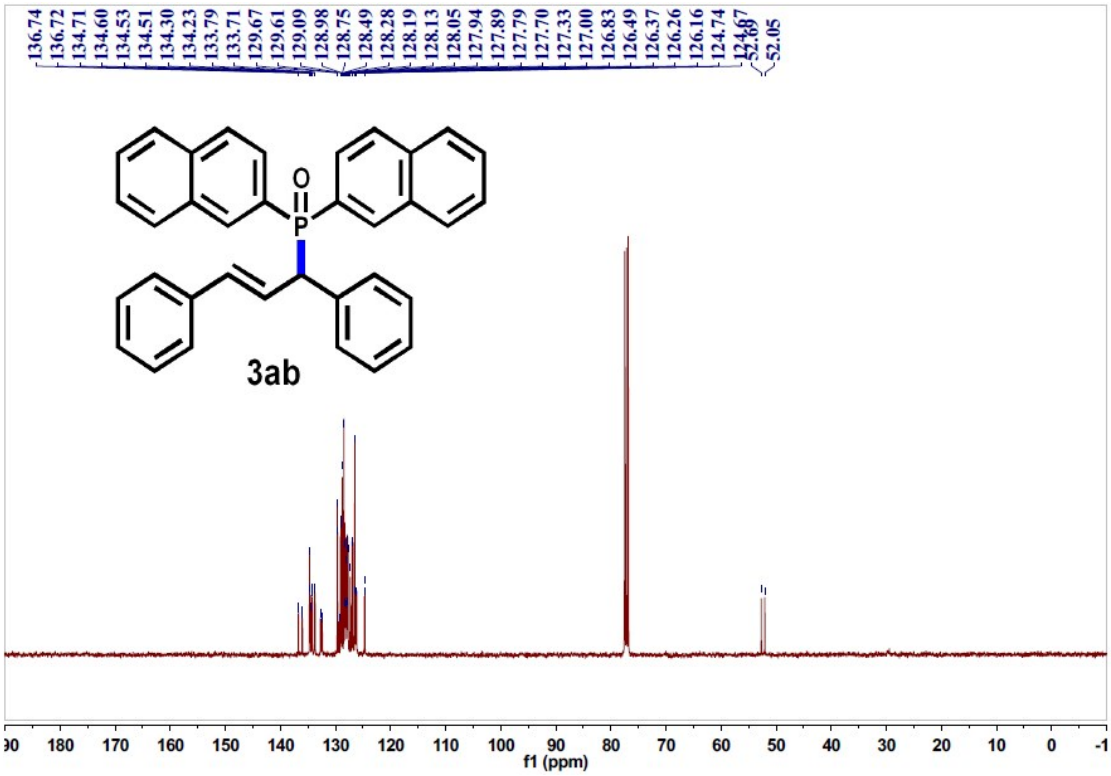

32.45
Me
O
P
Me
3z
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300
f1 (ppm)

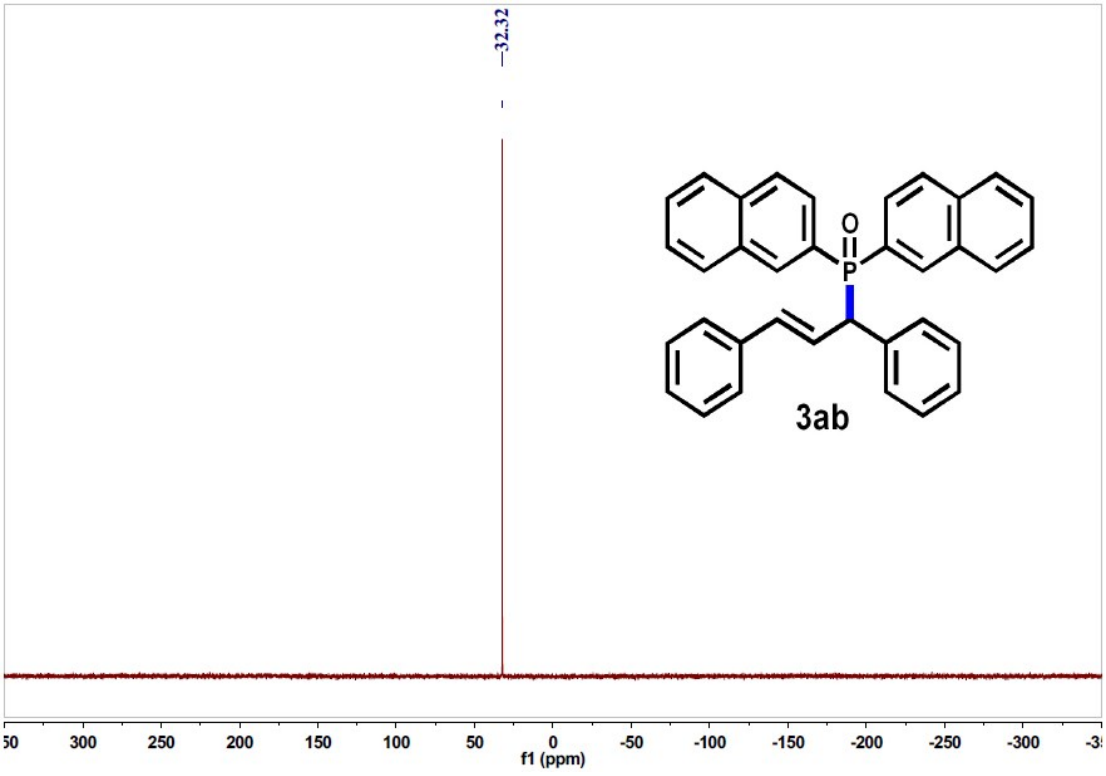

7.45
7.43
7.38
7.36
7.22
7.21
7.20
7.18
7.17
7.16
7.15
7.14
7.09
6.97
6.57
6.55
6.53
6.33
6.32
6.29
6.28
4.35
4.33
2.29
2.19
0.00
Me
Me
Me
Me
O
P
3aa
2.09
2.12
10.15
1.03
1.04
0.99
1.01
1.00
6.21
6.17
f1 (ppm)

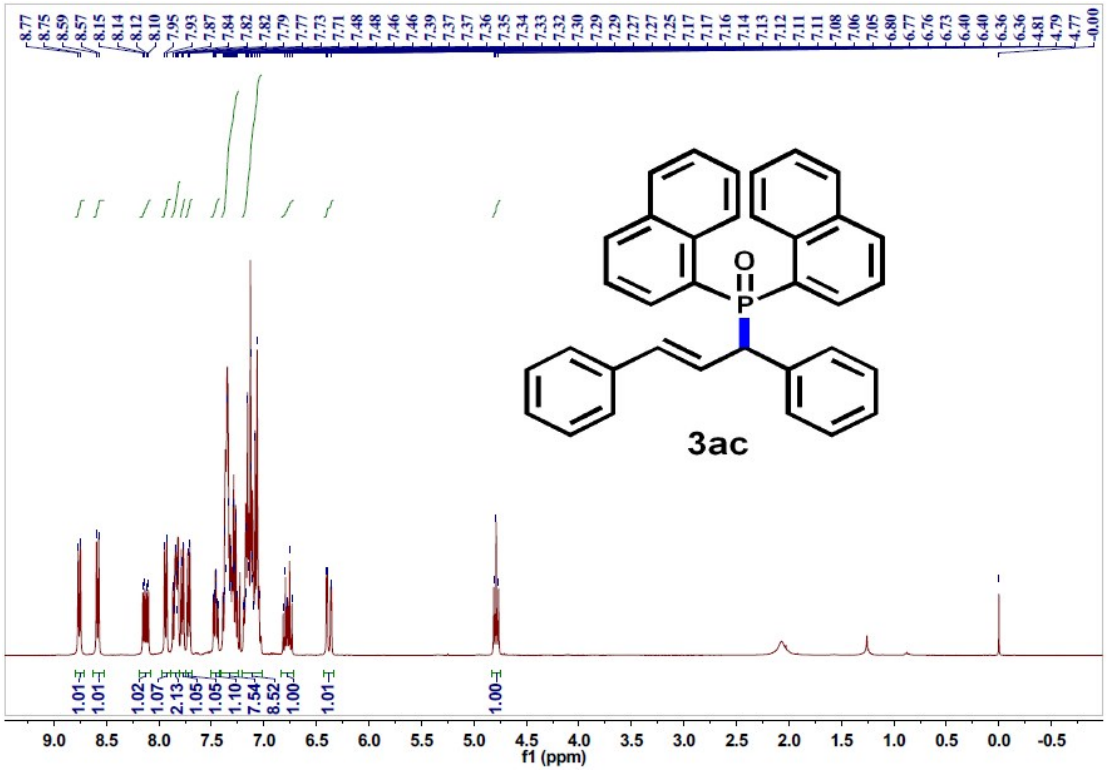

138.12
138.00
137.85
137.73
136.97
136.94
136.39
136.33
134.38
134.27
133.55
133.52
133.30
133.27
132.06
131.81
131.11
130.86
129.68
129.63
129.51
129.43
129.16
129.07
128.54
128.47
127.58
127.09
126.47
125.15
125.08
52.71
52.07
21.45
21.34
Me
Me
Me
Me
O
P
3aa
f1 (ppm)

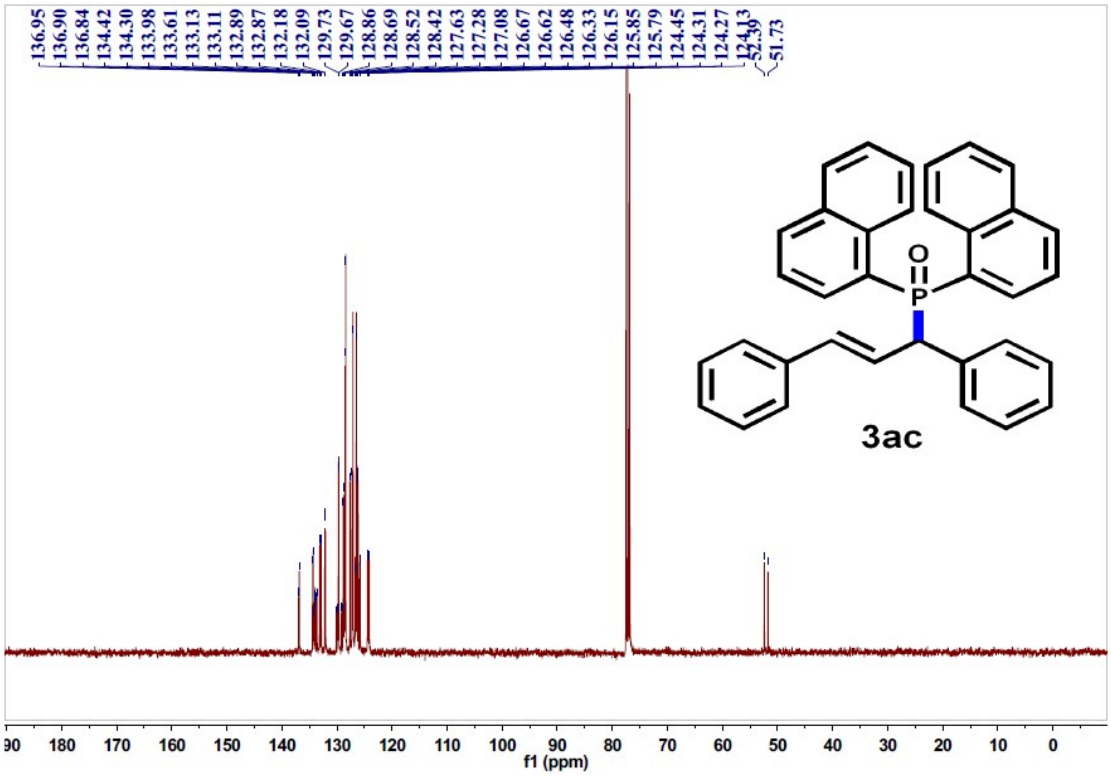

32.80
Me
Me
Me
Me
O
P
3aa
f1 (ppm)

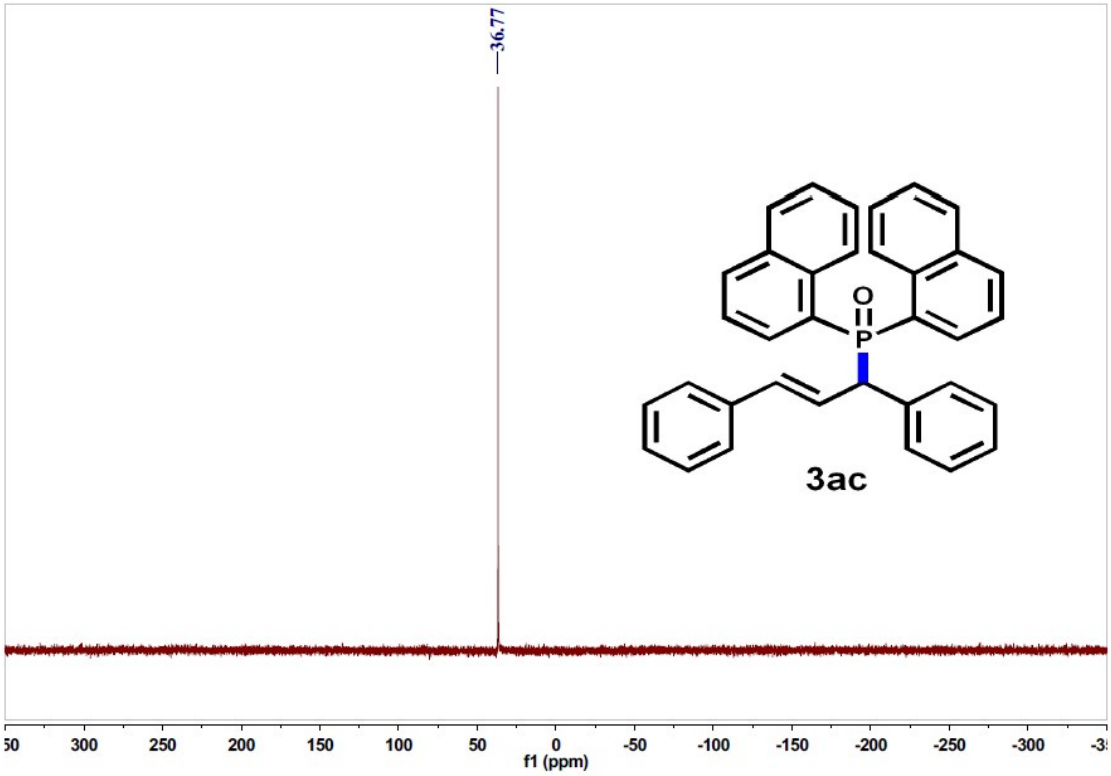

O
P
3ab
1.01
1.01
4.23
3.15
1.08
6.35
8.37
1.01
1.02
1.01
f1 (ppm)

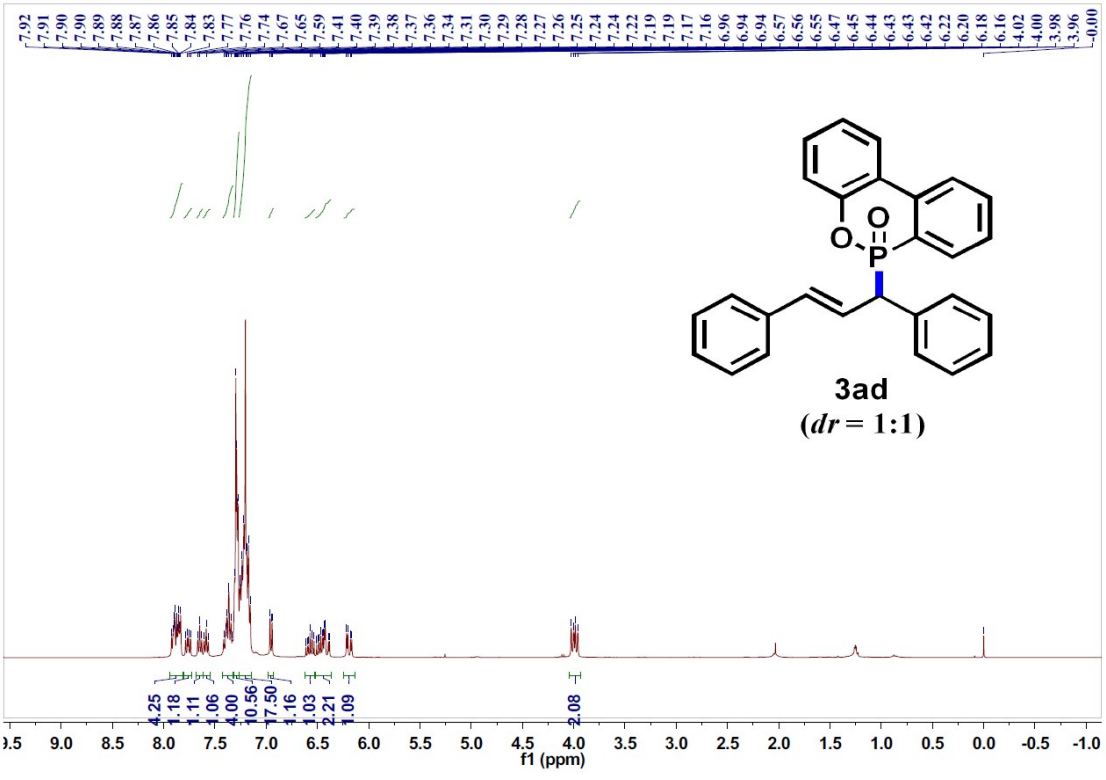

136.74
136.72
134.71
134.60
134.53
134.51
134.30
134.23
133.79
133.71
129.67
129.61
129.09
128.98
128.75
128.49
128.28
128.19
128.13
128.05
127.94
127.89
127.79
127.70
127.33
127.00
126.83
126.49
126.37
126.26
126.16
124.74
52.69
52.05
O
P
3ab
f1 (ppm)

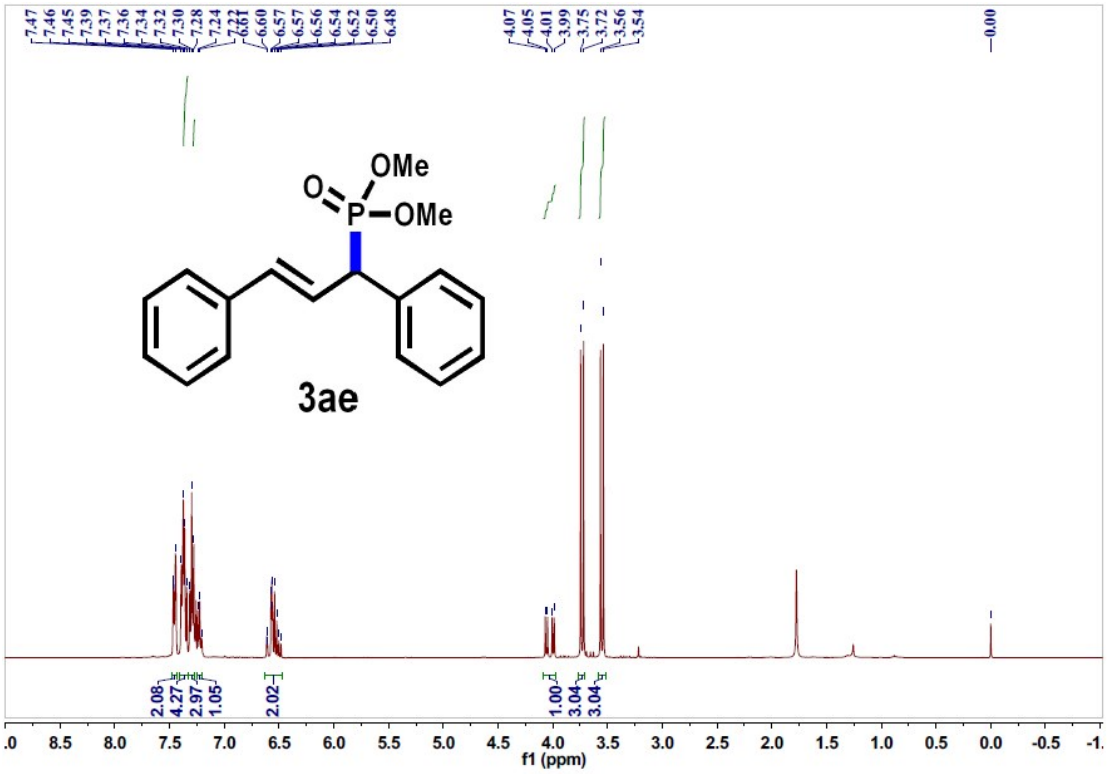

32.32
O
P
3ab
f1 (ppm)

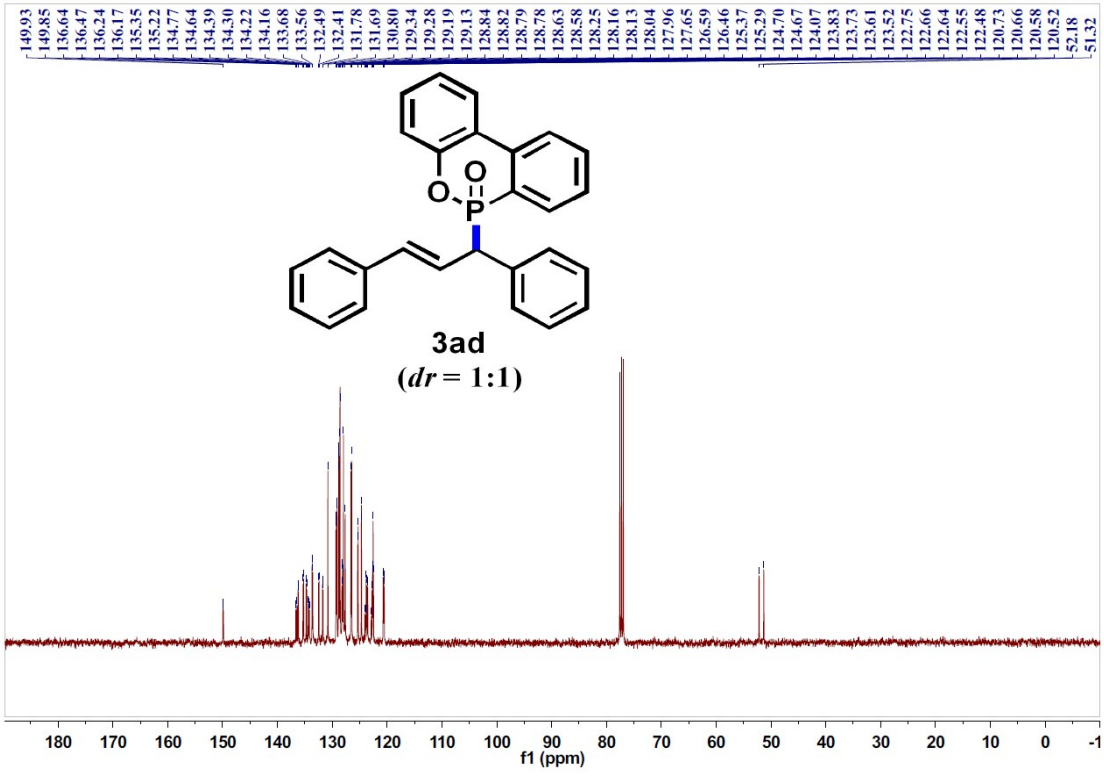

3ac
f1 (ppm)

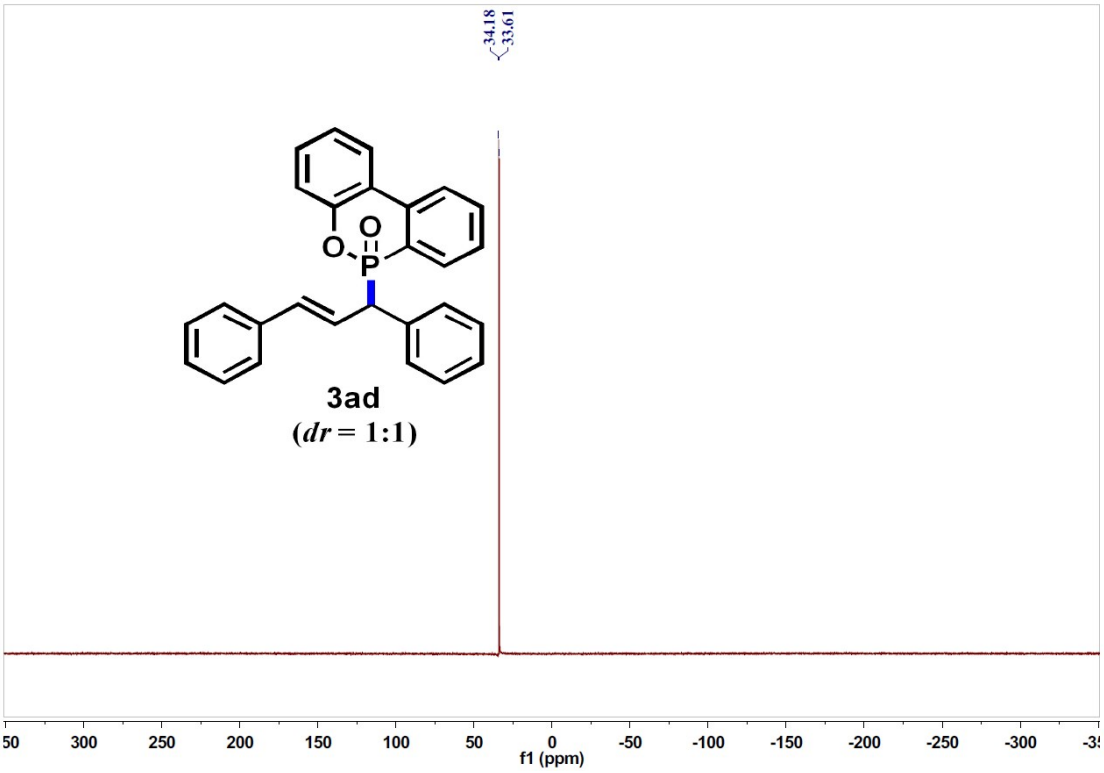

3ac
f1 (ppm)

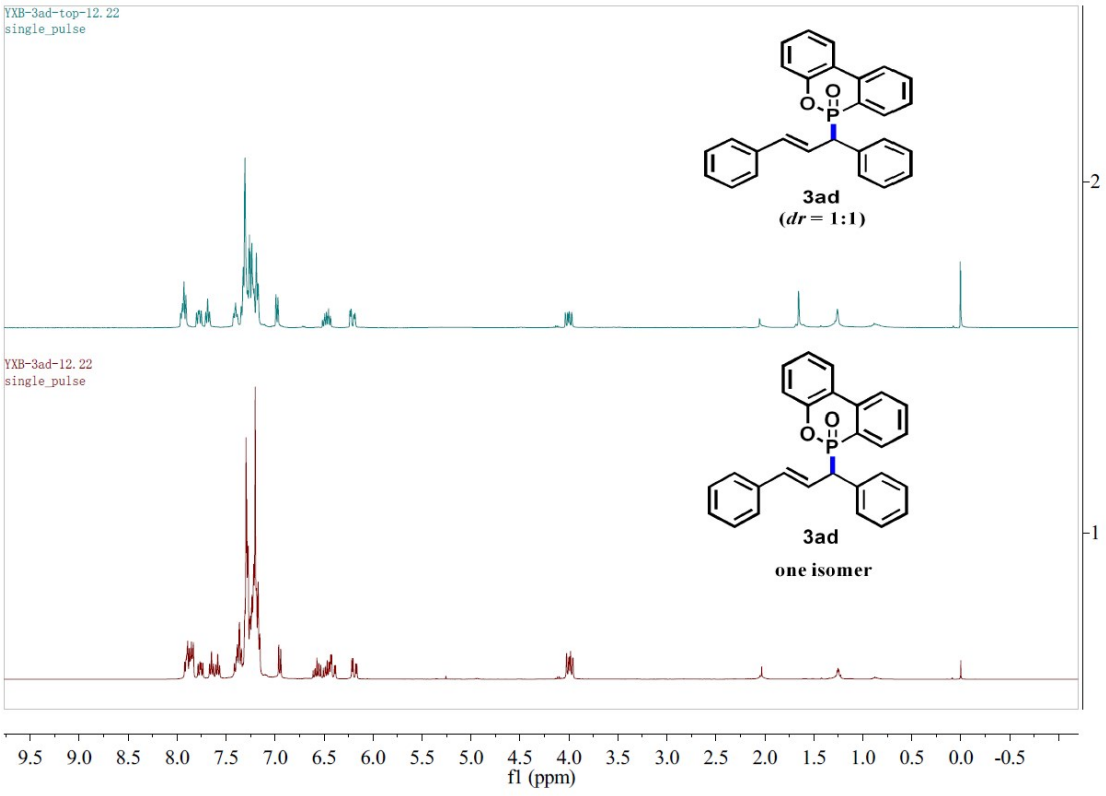

36.77
O
P
3ac
f1 (ppm)

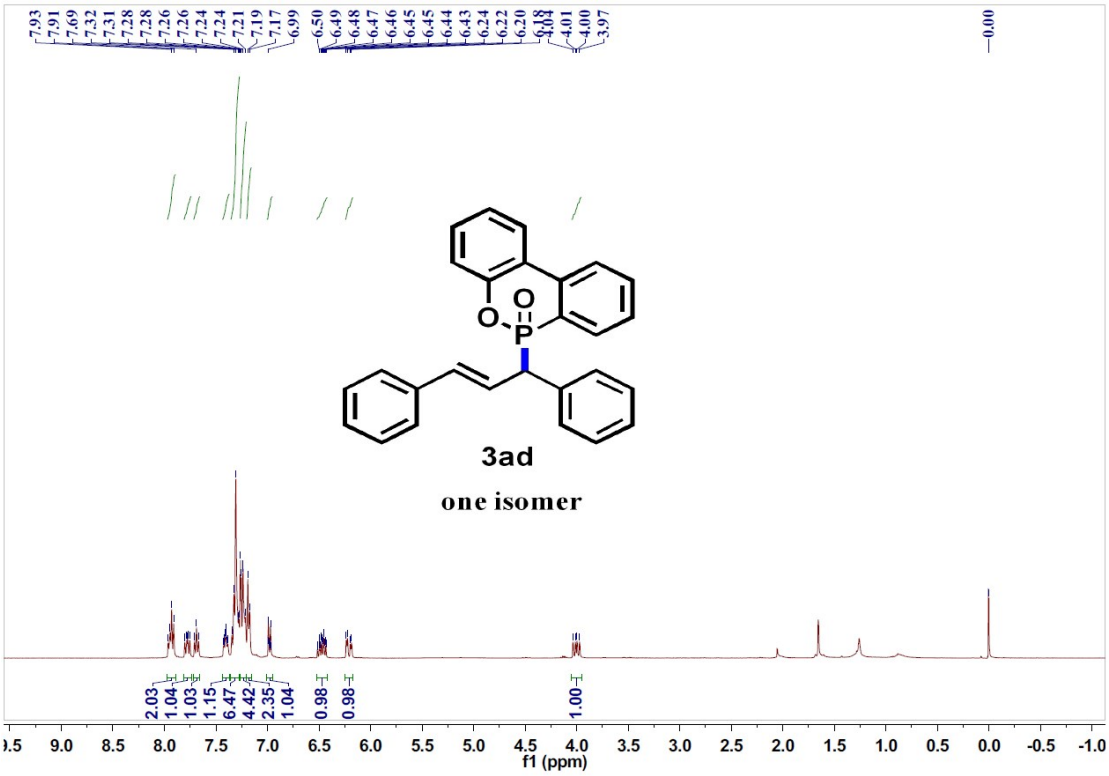

O
O
P
3ad
(dr = 1:1)
4.25
1.18
1.11
1.06
4.00
10.56
17.50
1.16
1.03
2.21
1.09
2.08
f1 (ppm)

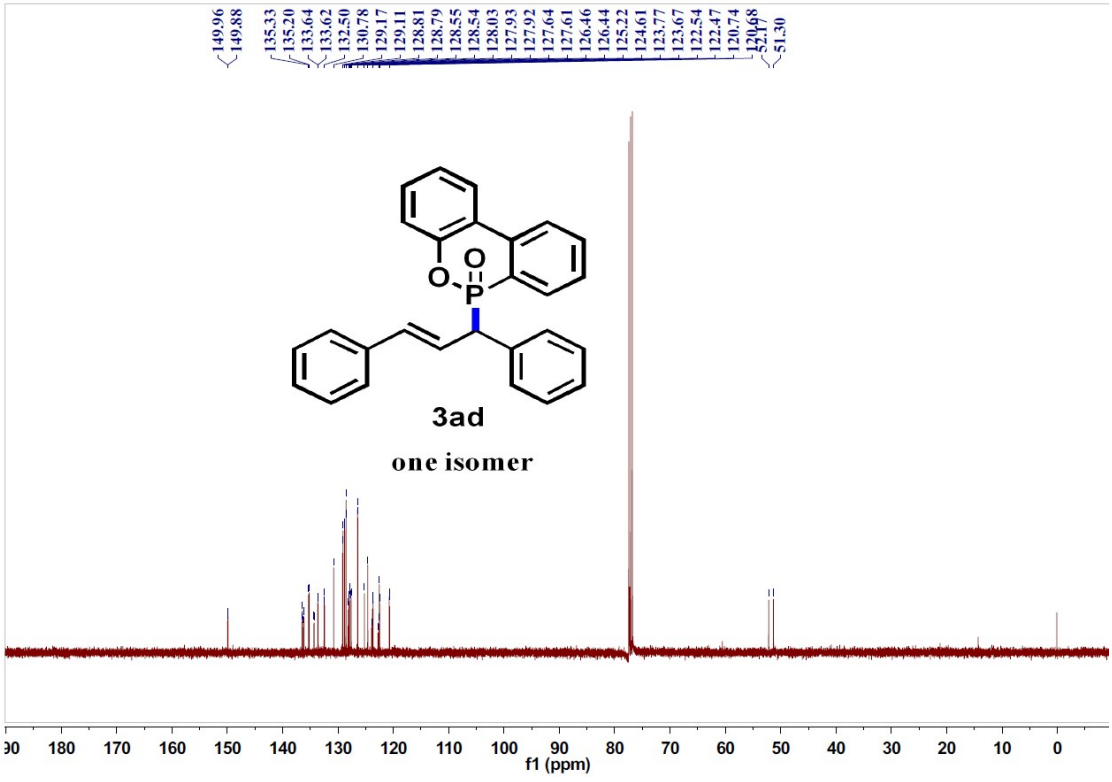

OMe
O
P
OMe
3ae
f1 (ppm)

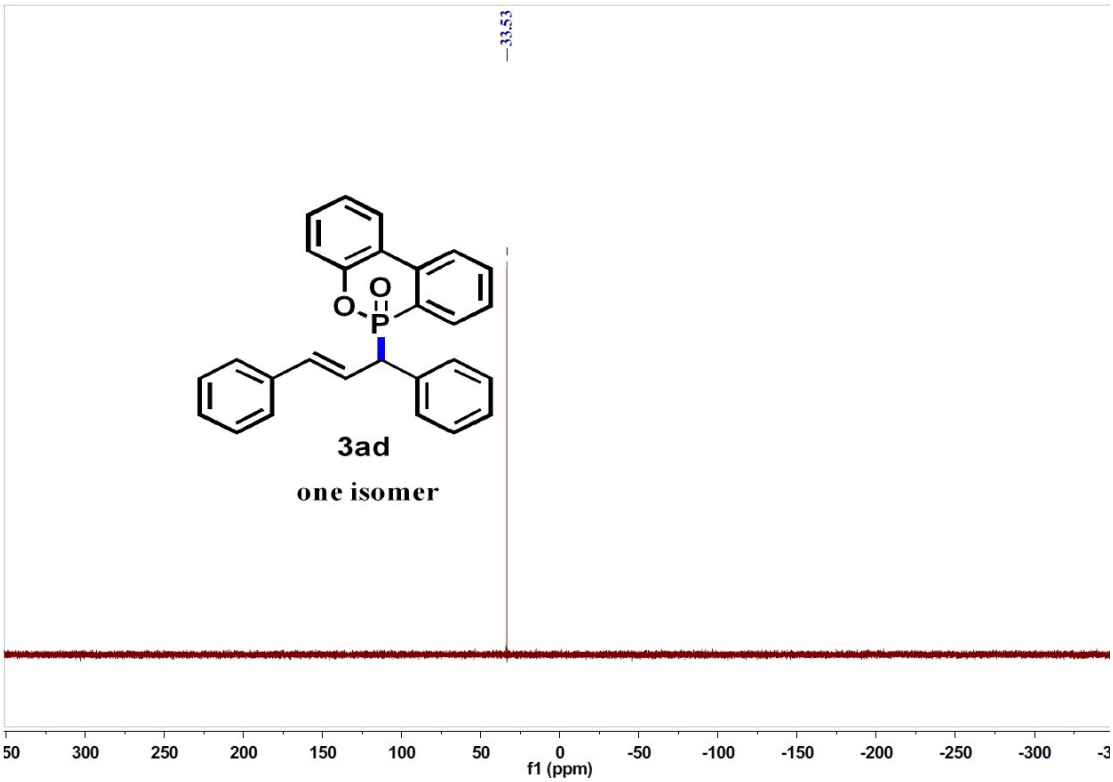

O
O
P
3ad
(dr = 1:1)
f1 (ppm)

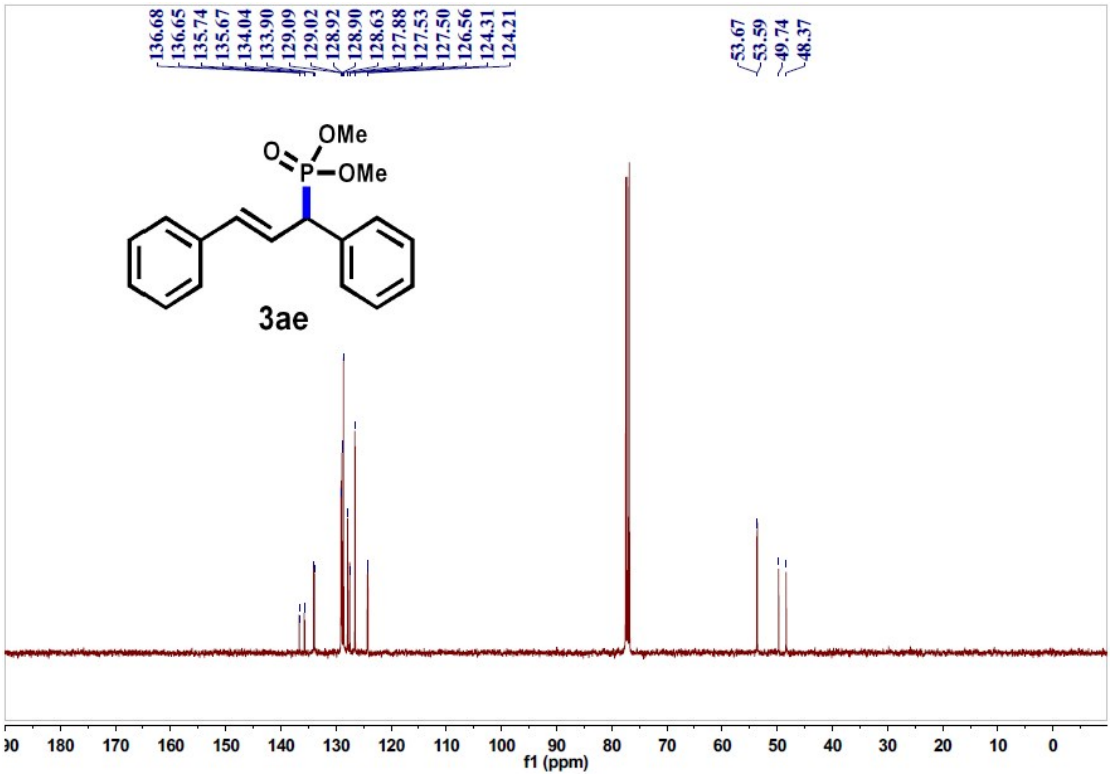

34.18
33.61
3ad
(dr = 1:1)
f1 (ppm)

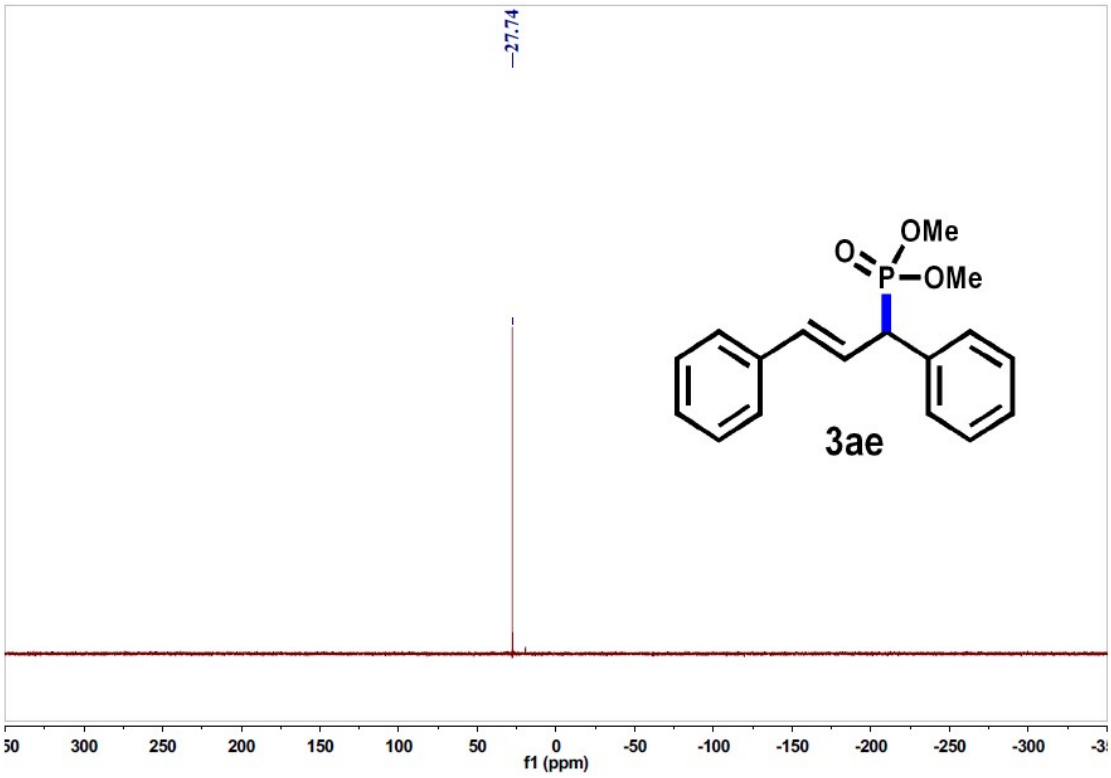

YXB-3ad-top-12.22
single_pulse
3ad
(dr = 1:1)
YXB-3ad-12.22
single_pulse
3ad
one isomer
f1 (ppm)

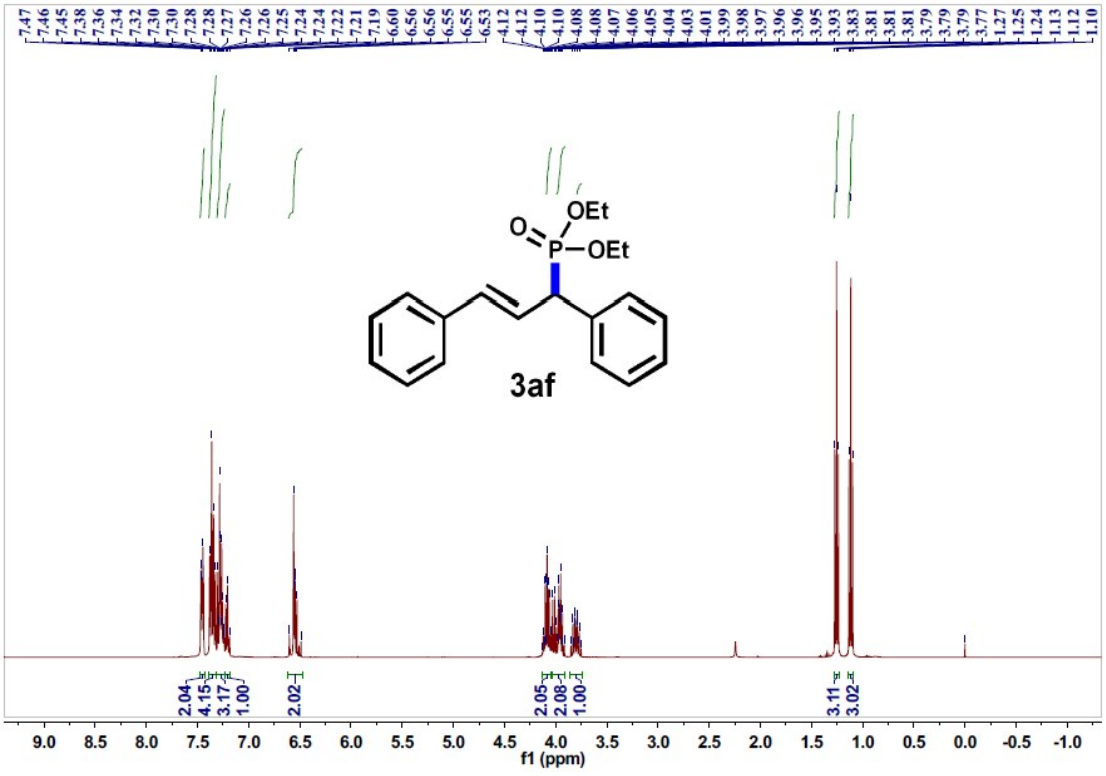

7.93
7.91
7.69
7.32
7.31
7.28
7.28
7.26
7.26
7.24
7.24
7.21
7.19
7.17
6.99
6.50
6.49
6.48
6.47
6.46
6.45
6.45
6.44
6.43
6.24
6.22
6.20
4.01
4.00
3.97
0.00
O
O
P
3ad
one isomer
2.03
1.04
1.03
1.15
6.47
4.42
2.35
1.04
0.98
0.98
1.00
f1 (ppm)

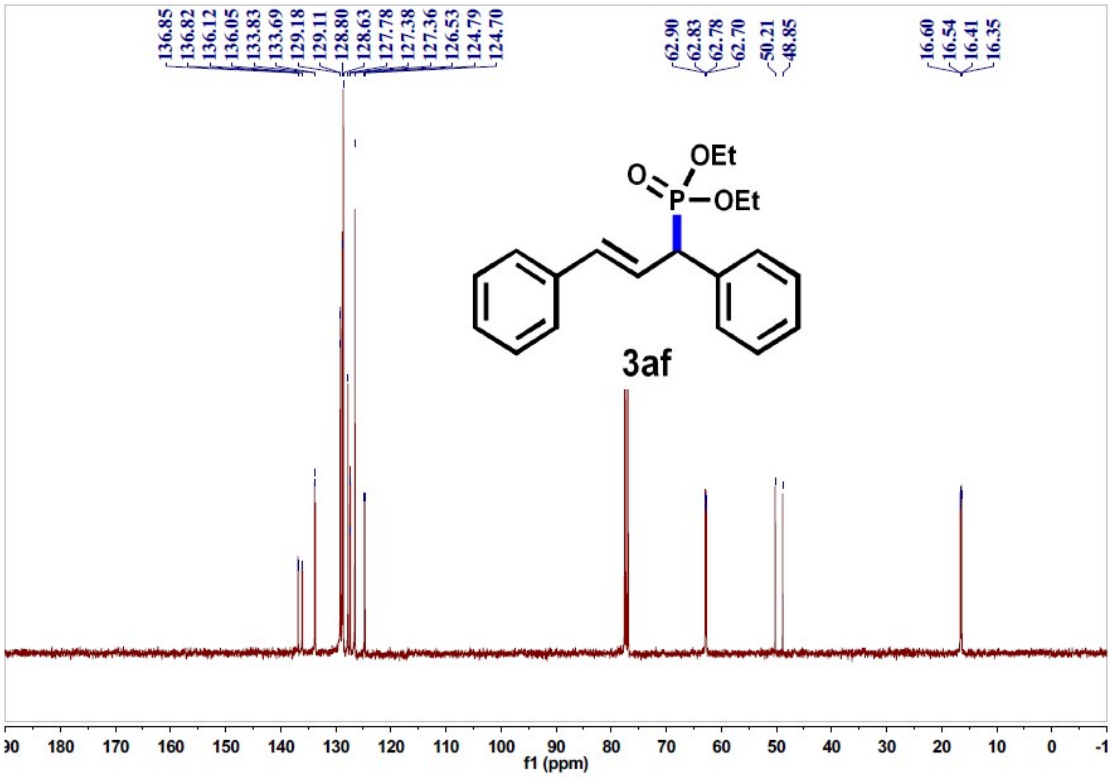

149.96
149.88
135.33
135.20
133.64
133.62
132.50
130.78
129.17
129.11
128.81
128.79
128.55
128.54
128.03
127.93
127.92
127.64
127.61
126.46
126.44
125.22
124.61
123.77
123.67
122.54
122.47
120.74
120.68
52.17
51.30
O
O
P
3ad
one isomer
f1 (ppm)

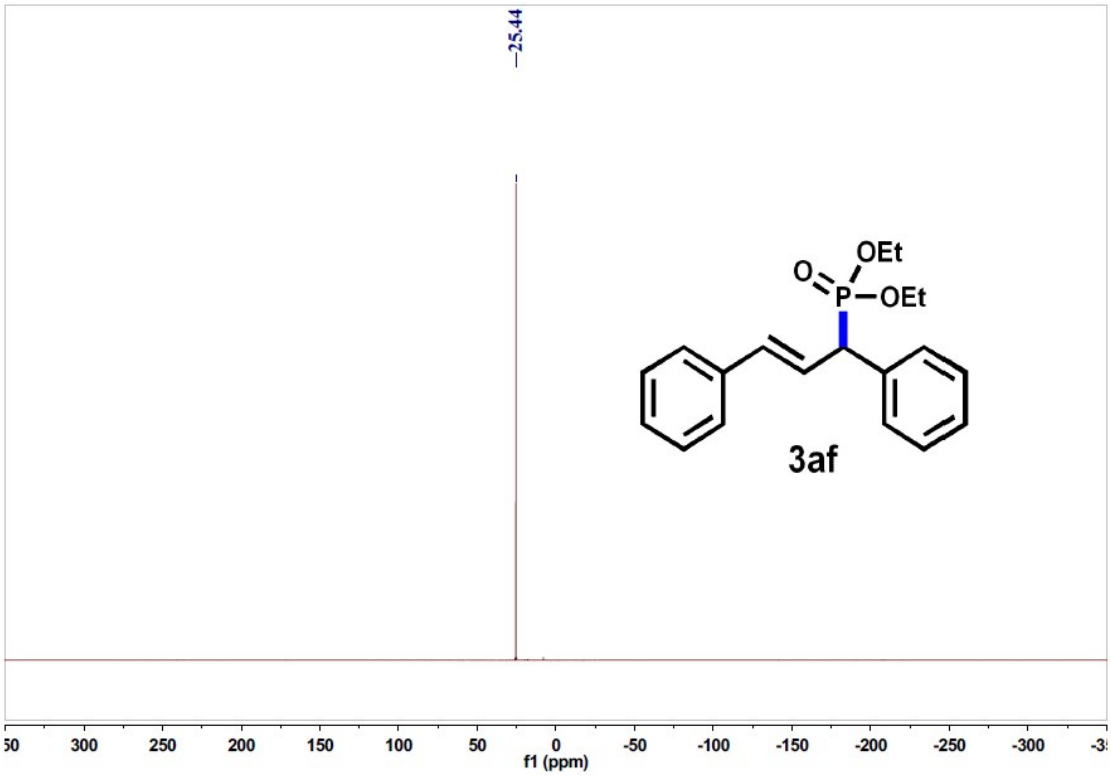

33.53
3ad
one isomer
f1 (ppm)

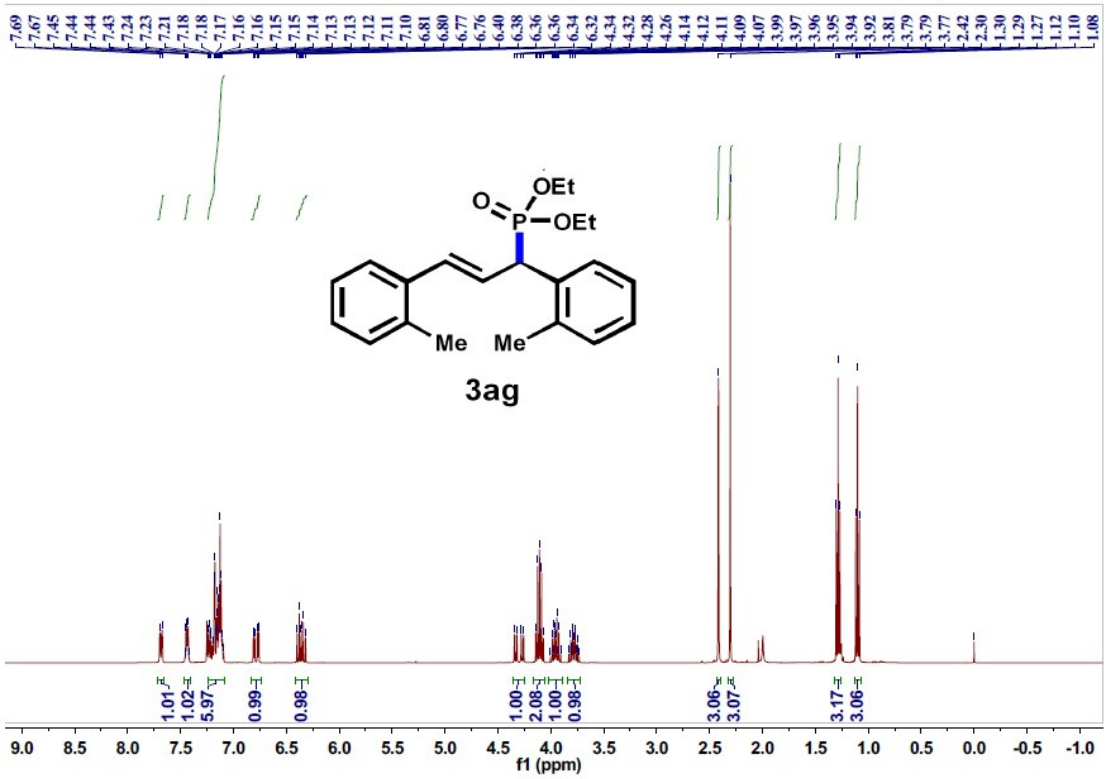

136.68
136.65
135.74
135.67
134.04
133.90
129.09
129.02
128.92
128.90
128.63
127.88
127.53
127.50
126.56
124.31
124.21
53.67
53.59
49.74
48.37
OMe
OMe
3ae
f1 (ppm)

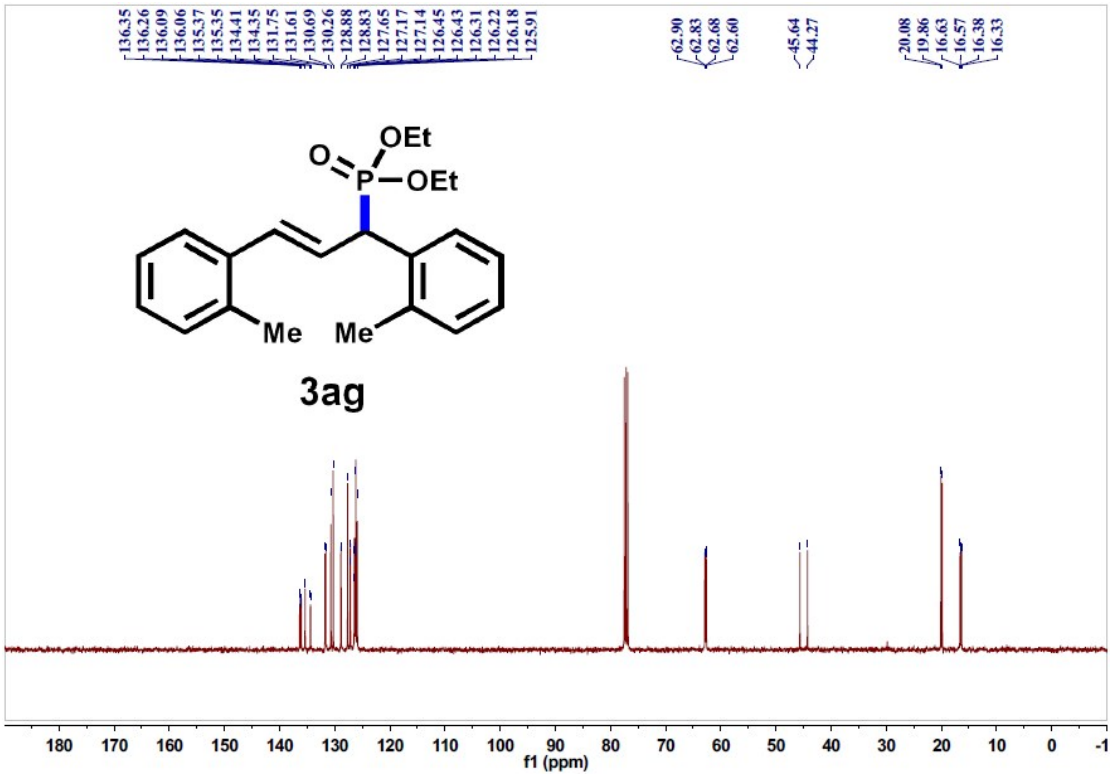

27.74
OMe
O
P
OMe
3ae
f1 (ppm)

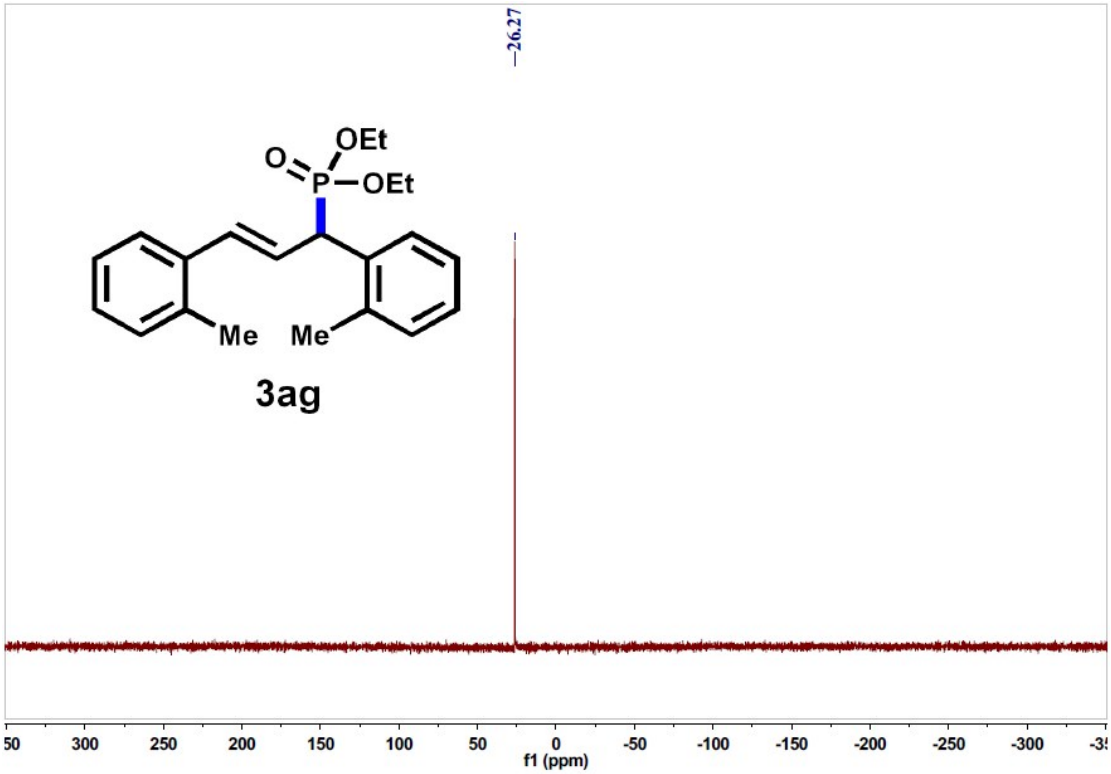

OEt
O
P
OEt
3af
2.04
4.15
3.17
1.00
2.02
2.05
2.08
1.00
3.11
3.02
f1 (ppm)

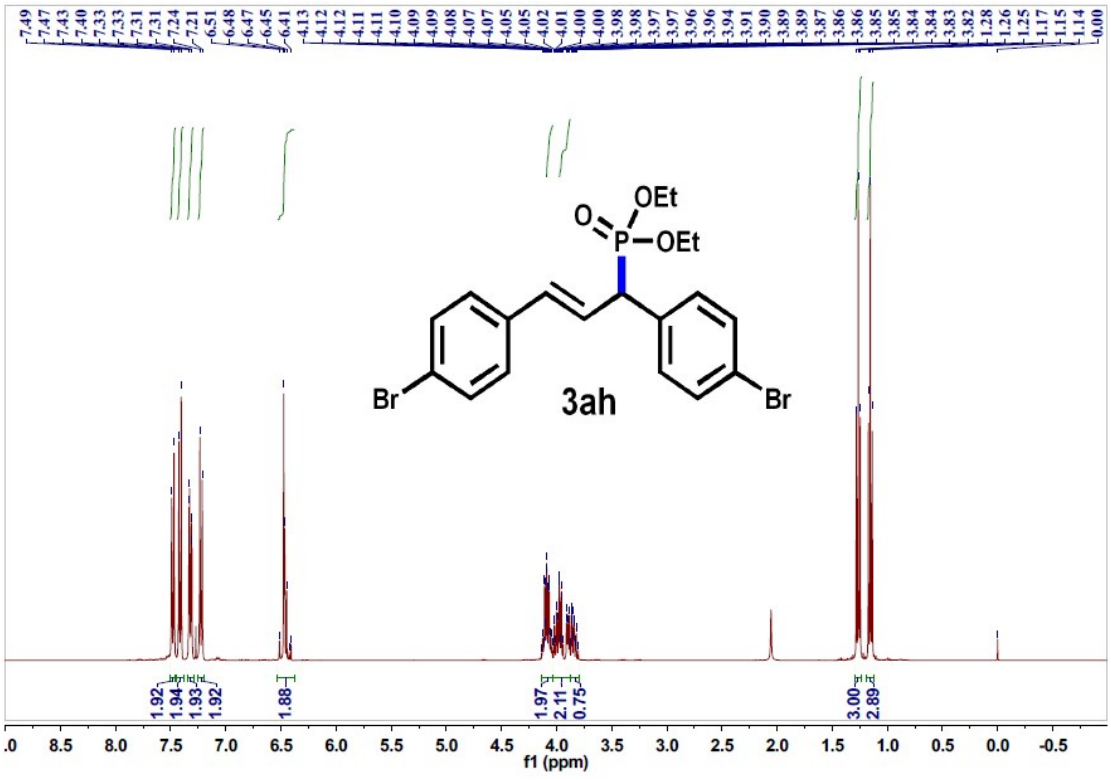

136.85
136.82
136.12
136.05
133.83
133.69
129.18
129.11
128.80
128.63
127.78
127.38
127.36
126.53
124.79
124.70
62.90
62.83
62.78
62.70
50.21
48.85
16.60
16.54
16.41
16.35
OEt
O
P
OEt
3af
f1 (ppm)

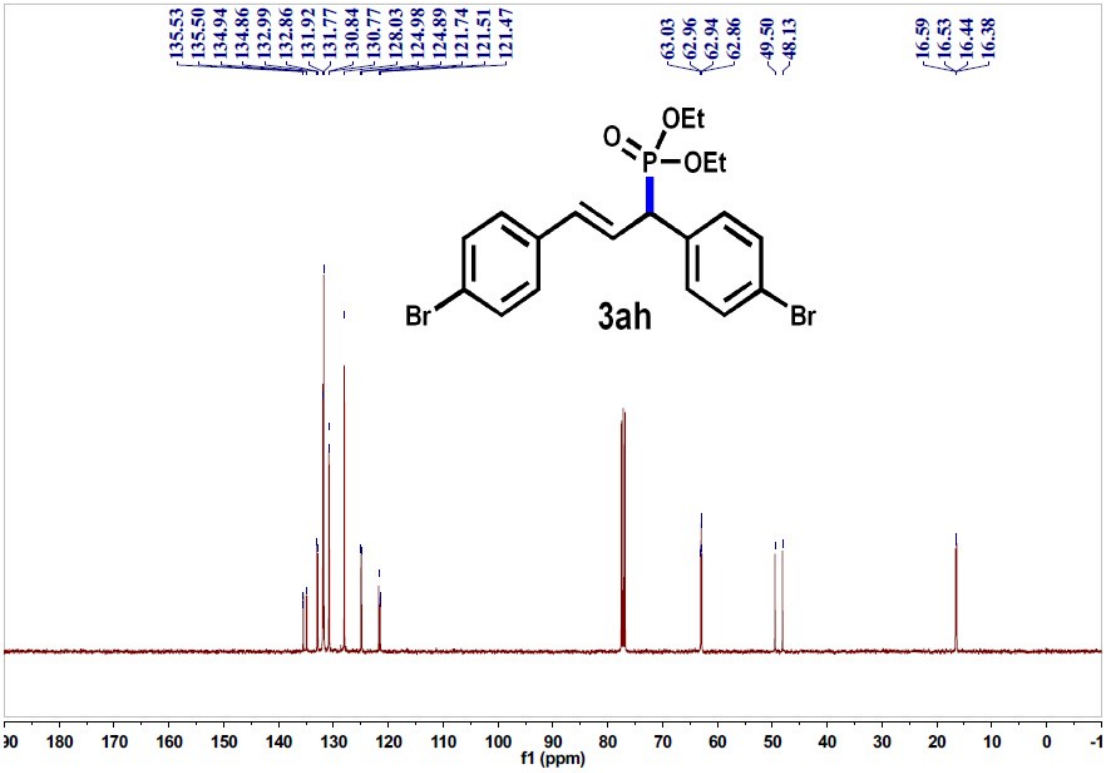

25.44
OEt
O
P
OEt
3af
f1 (ppm)

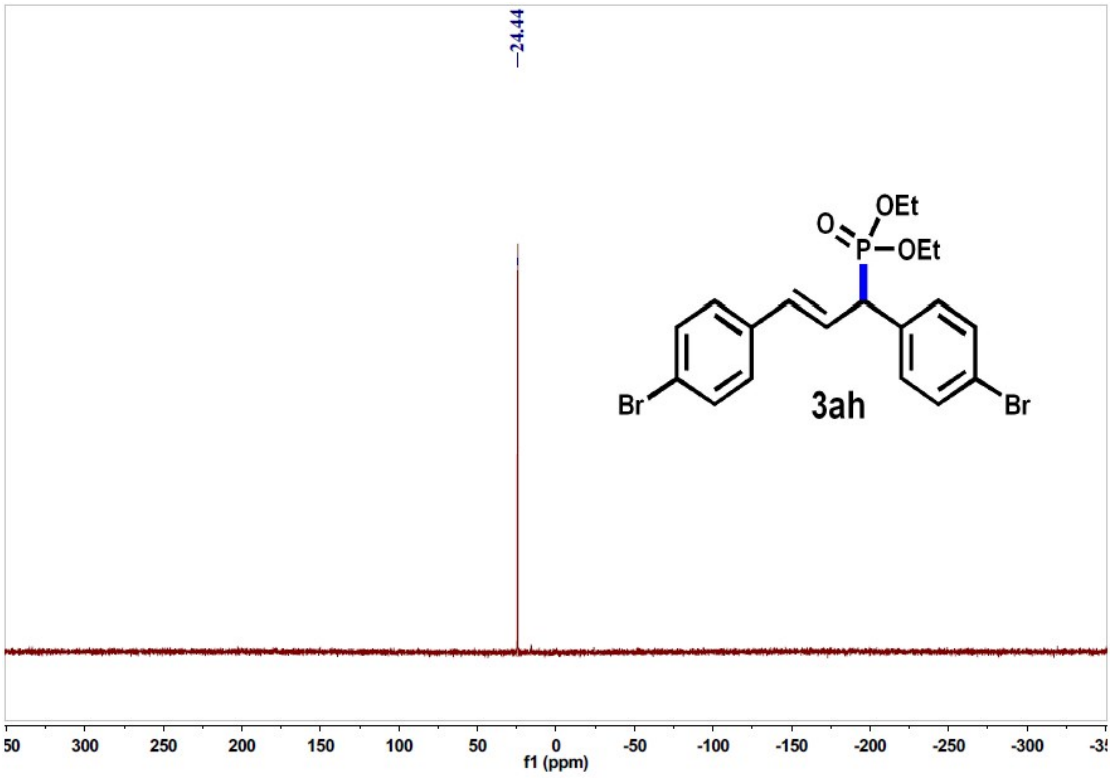

OEt
O
P
OEt
Me
Me
3ag
f1 (ppm)

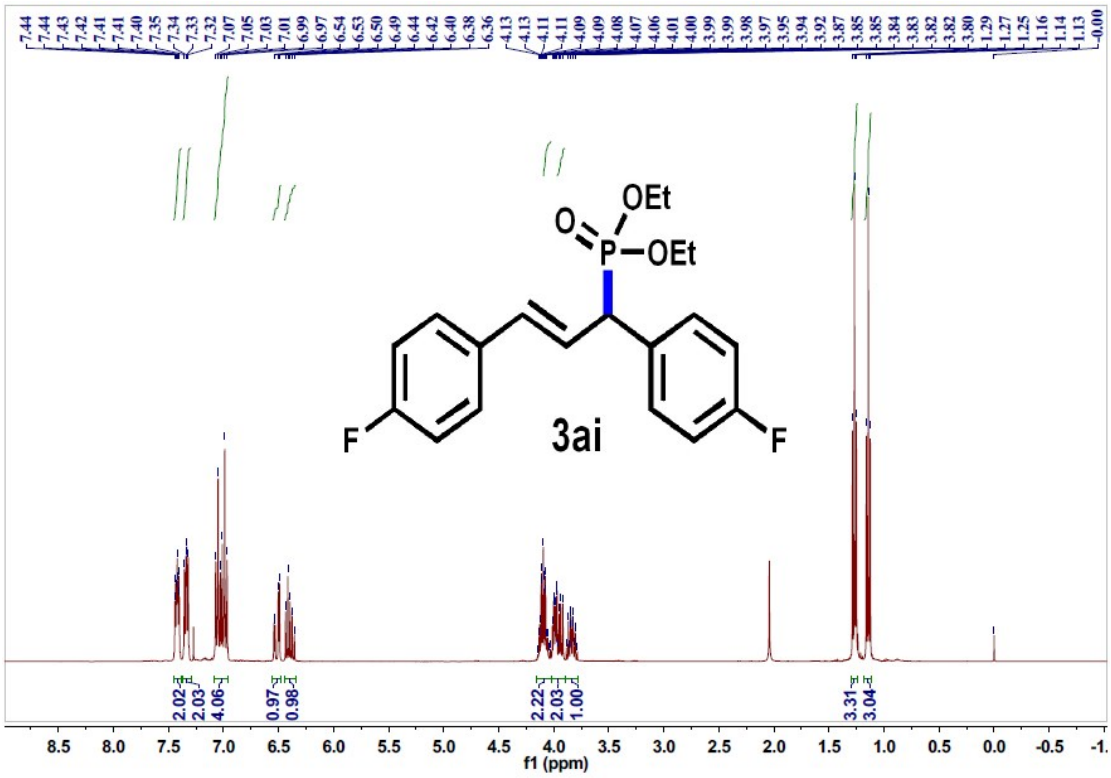

OEt
O
P
OEt
Me
Me
3ag
f1 (ppm)

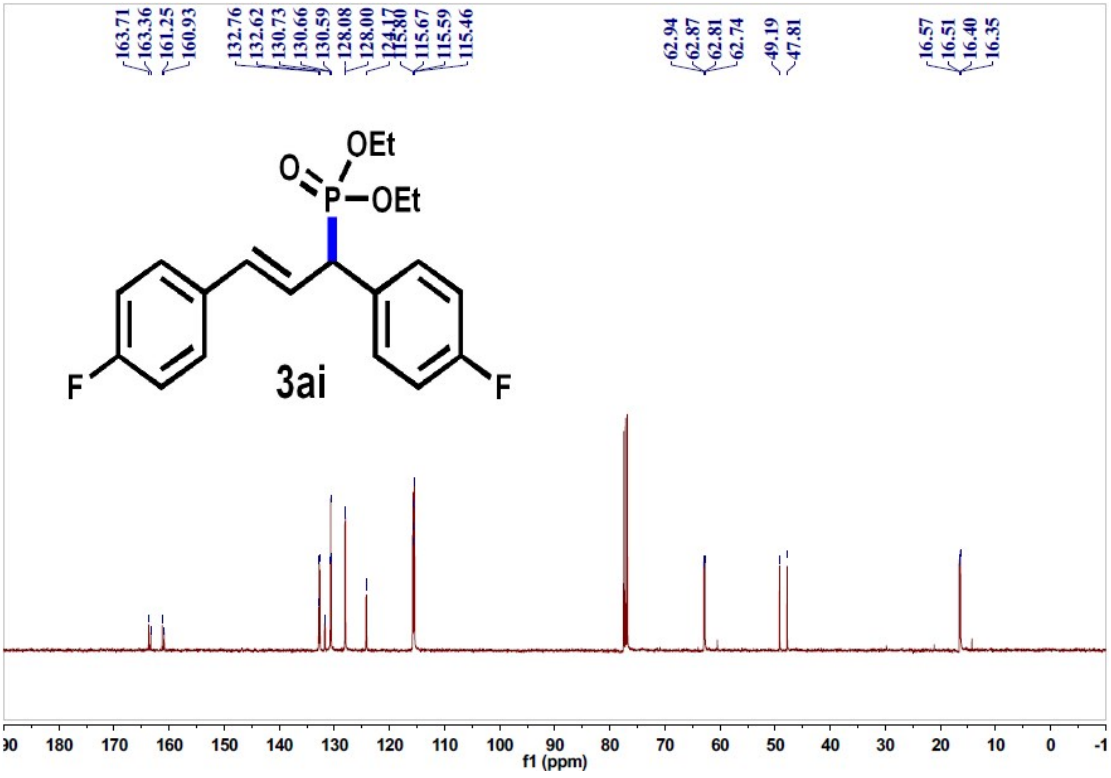

-26.27
OEt
O
P
OEt
Me
Me
3ag
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

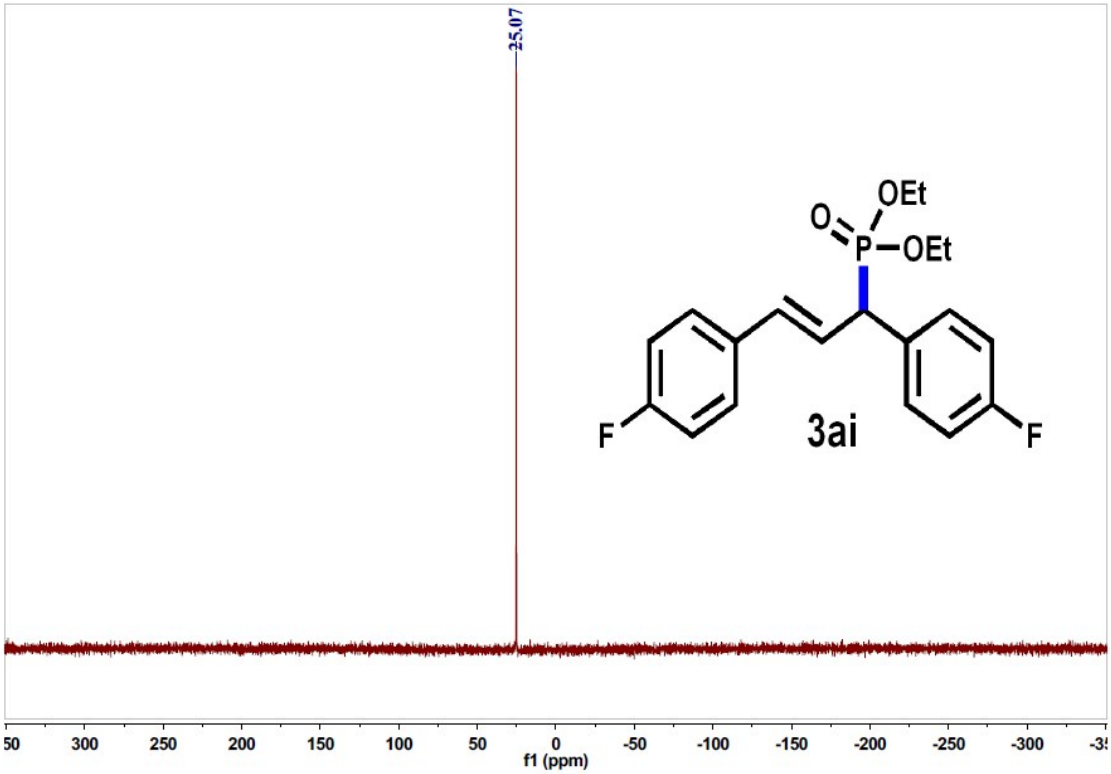

OEt
O
P
OEt
Br
Br
3ah
1.92
1.94
1.93
1.92
1.88
1.97
2.11
0.75
3.00
2.89
f1 (ppm)

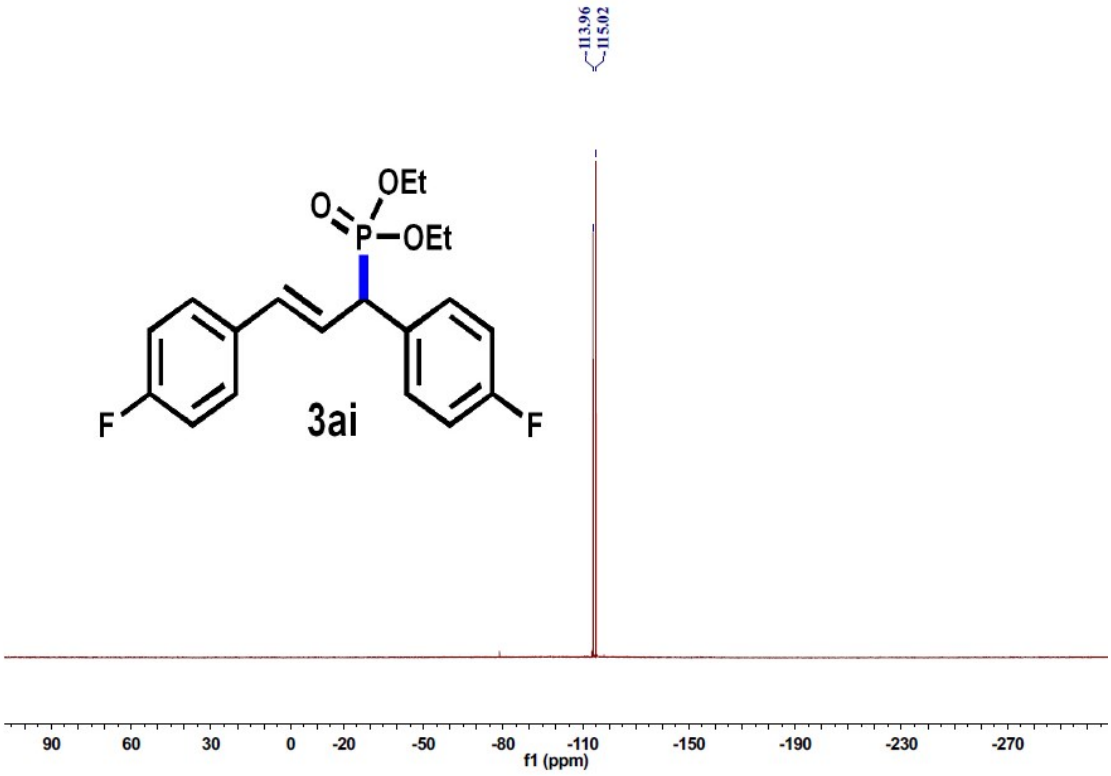

135.53
135.50
134.94
134.86
132.99
132.86
131.92
131.77
130.84
130.77
128.03
124.98
124.89
121.74
121.51
121.47
63.03
62.96
62.94
62.86
49.50
48.13
16.59
16.53
16.44
16.38
OEt
O
P
OEt
Br
3ah
Br
f1 (ppm)

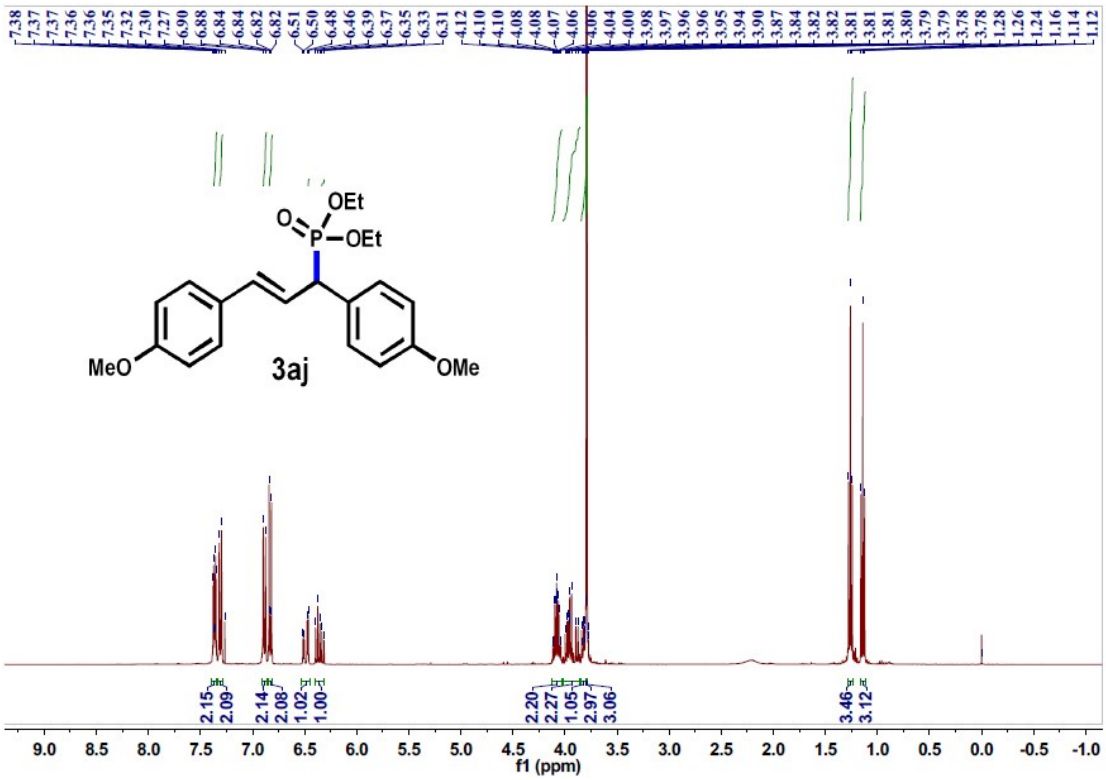

24.44
OEt
O
P
OEt
Br
3ah
Br
f1 (ppm)

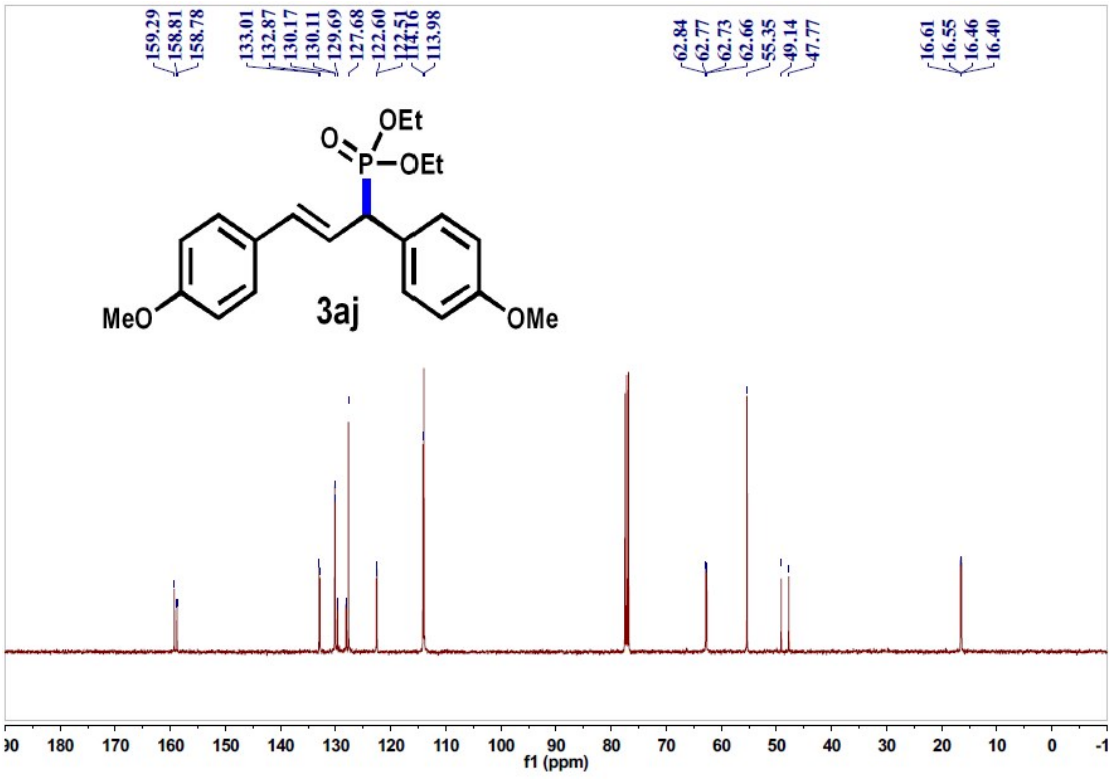

OEt
O
P
OEt
F
F
3ai
f1 (ppm)

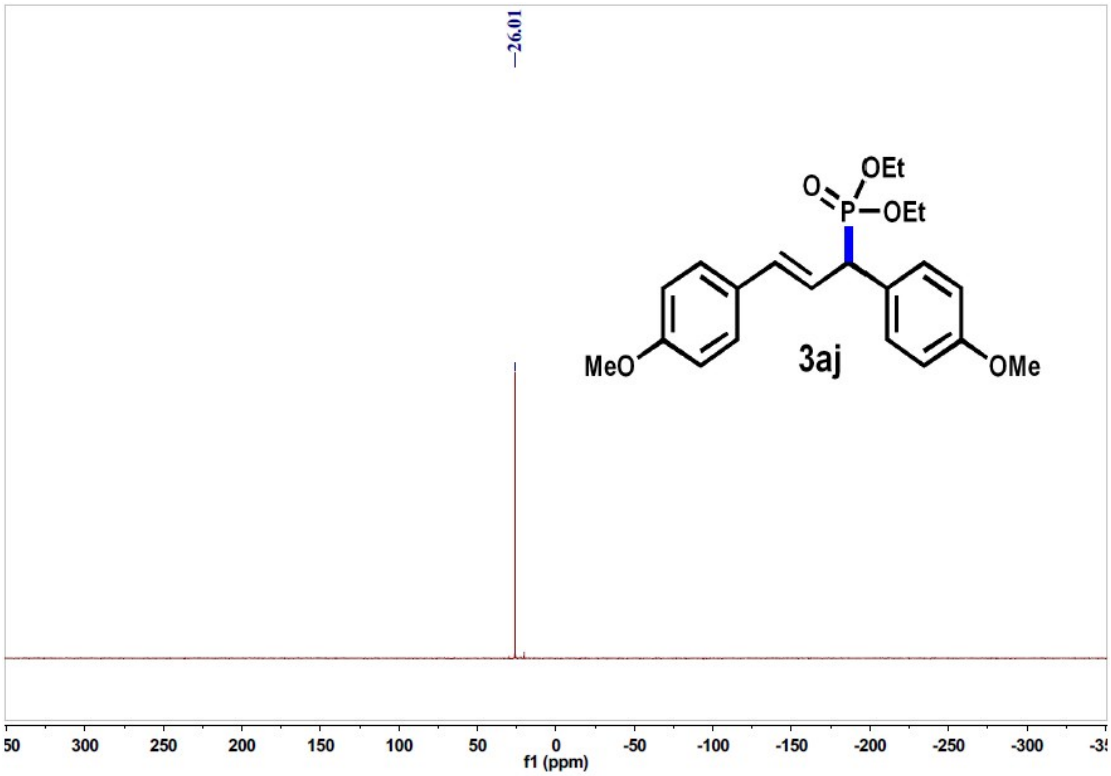

OEt
O
P
OEt
F
F
3ai
f1 (ppm)

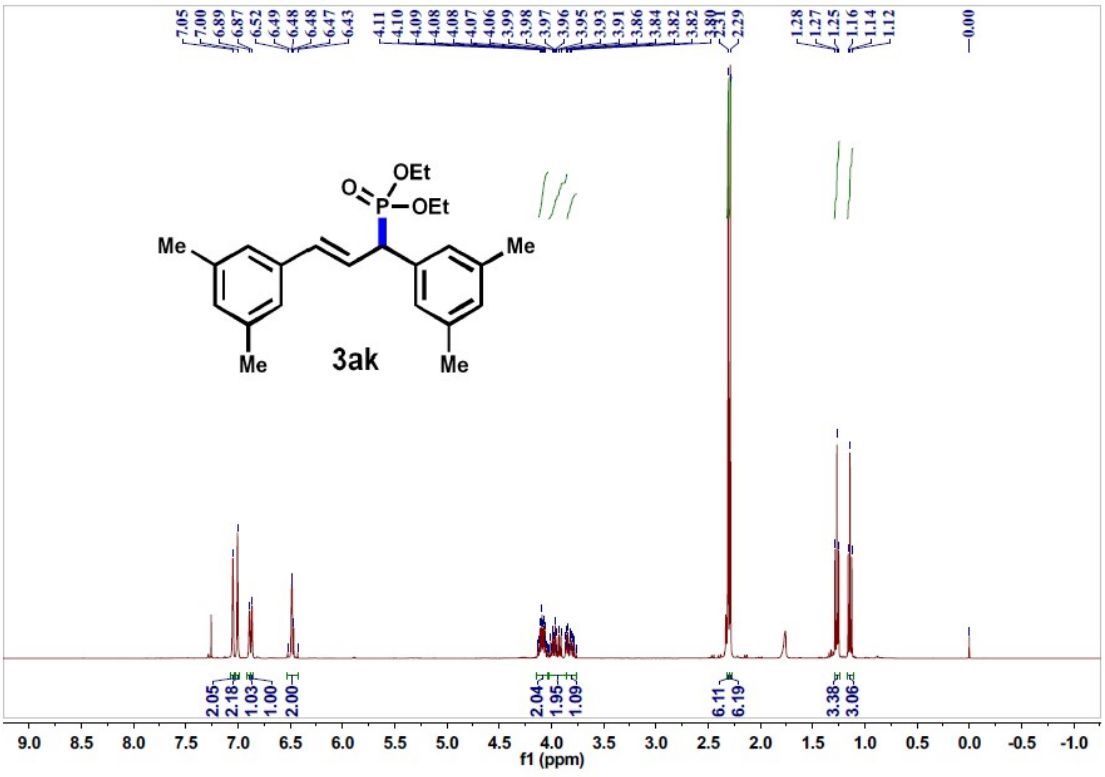

25.07
OEt
O
P
OEt
F
3ai
F
50
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

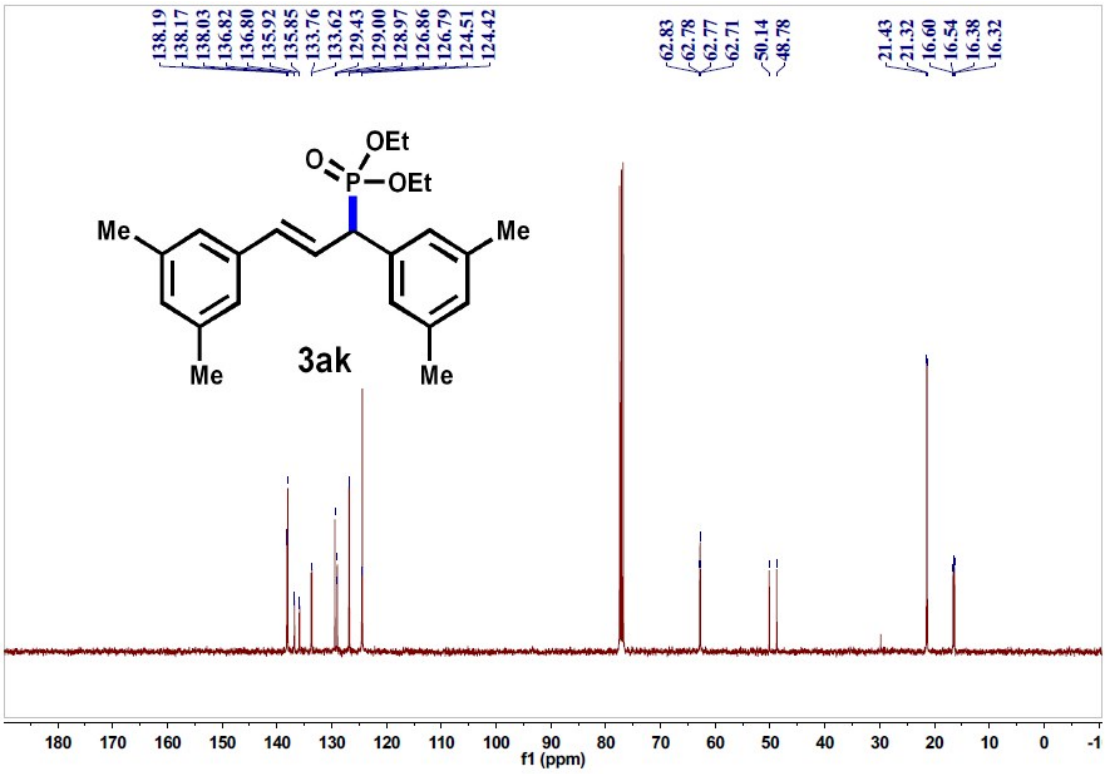

113.96
115.02
OEt
O
P
OEt
F
3ai
F
90
60
30
0
-20
-50
-80
-110
-150
-190
-230
-270
f1 (ppm)

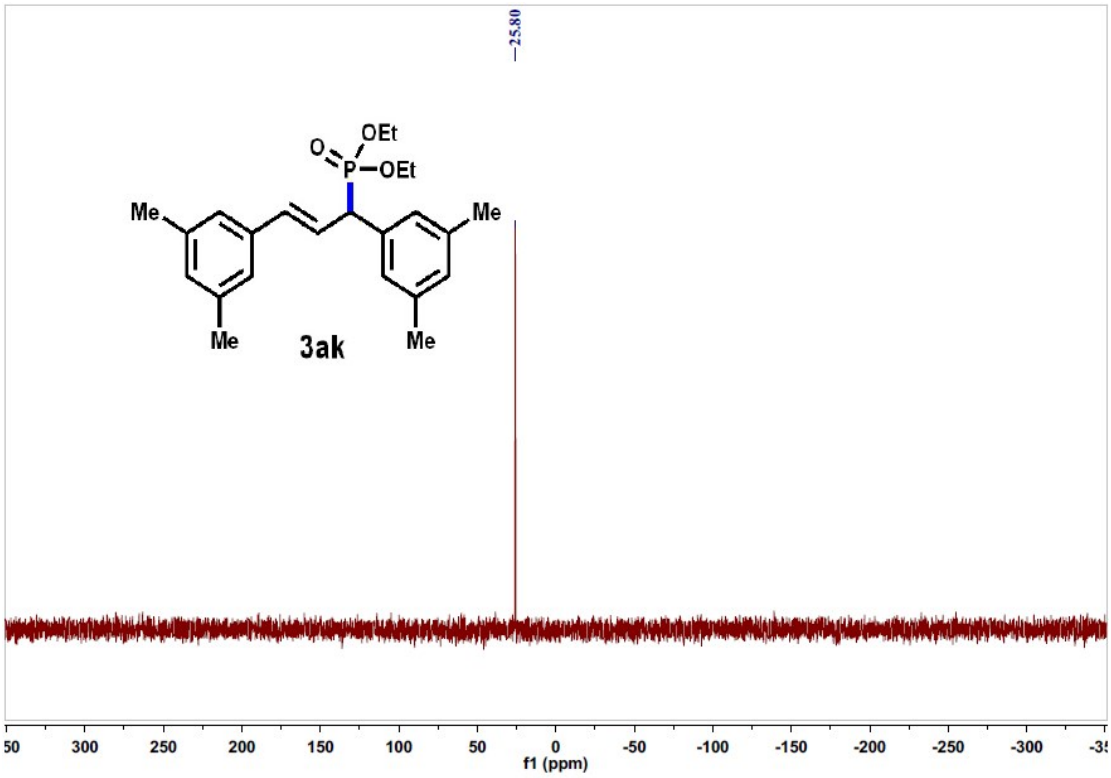

OEt
O
P
OEt
MeO
OMe
3aj
f1 (ppm)

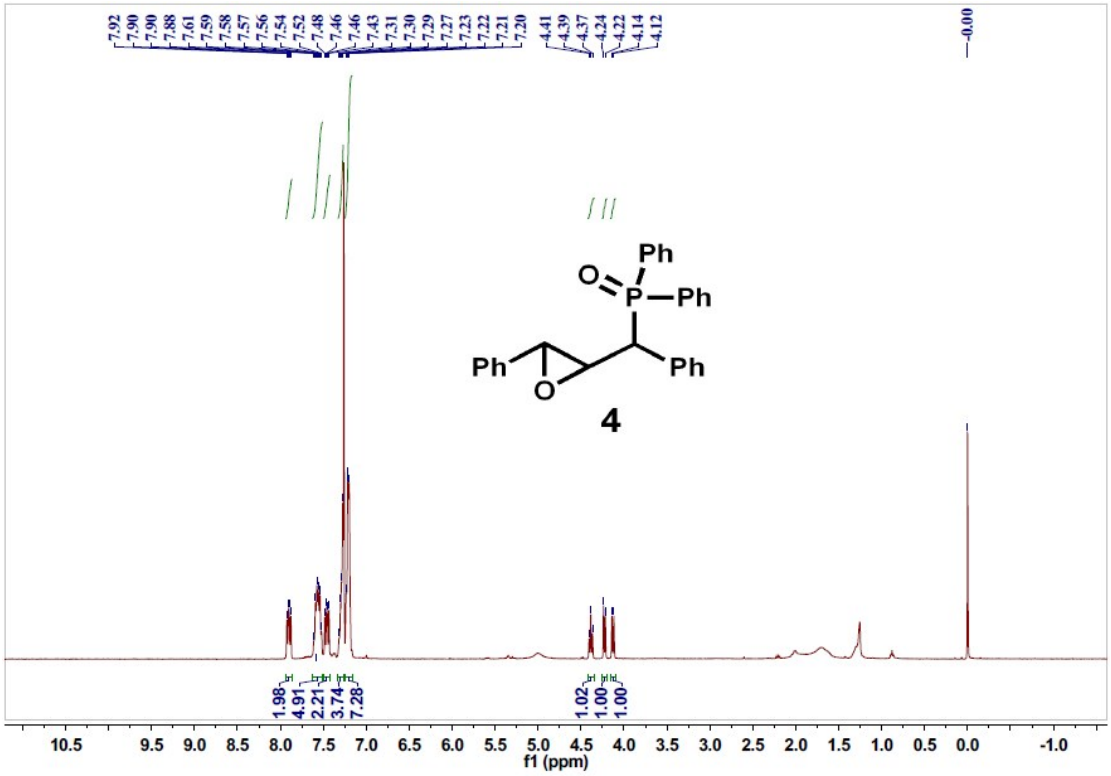

OEt
O
P
OEt
MeO
OMe
3aj
f1 (ppm)

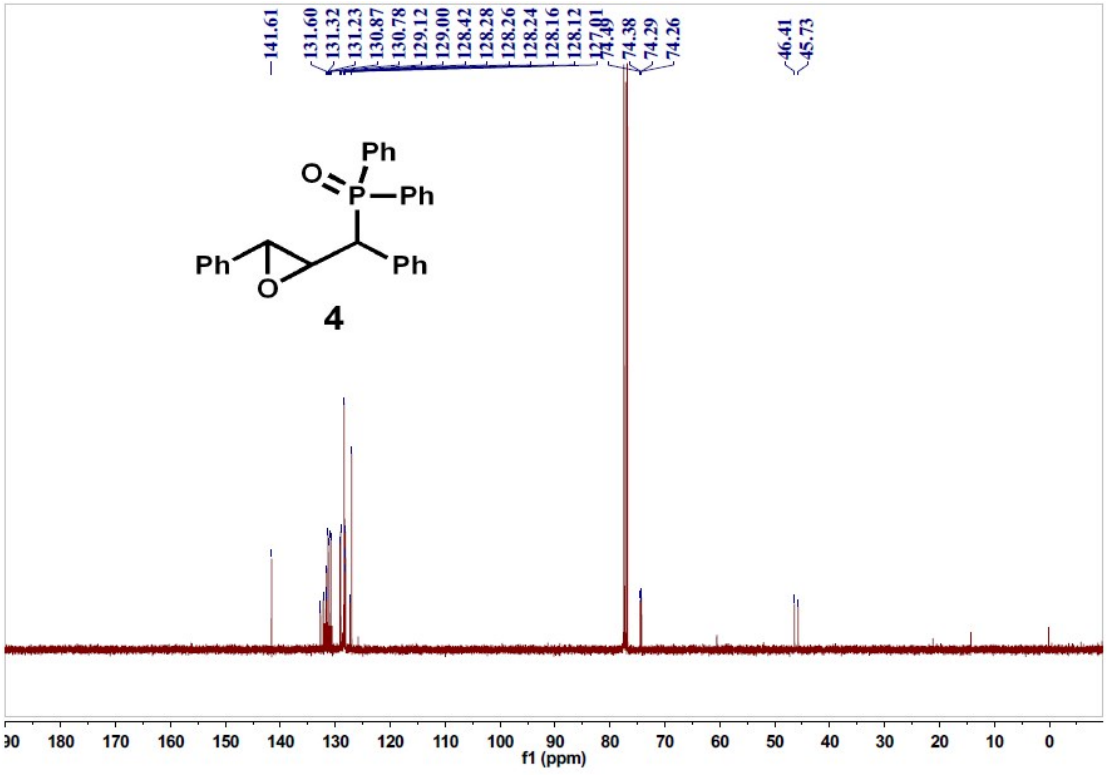

26.01
OEt
O
P
OEt
MeO
3aj
OMe
f1 (ppm)

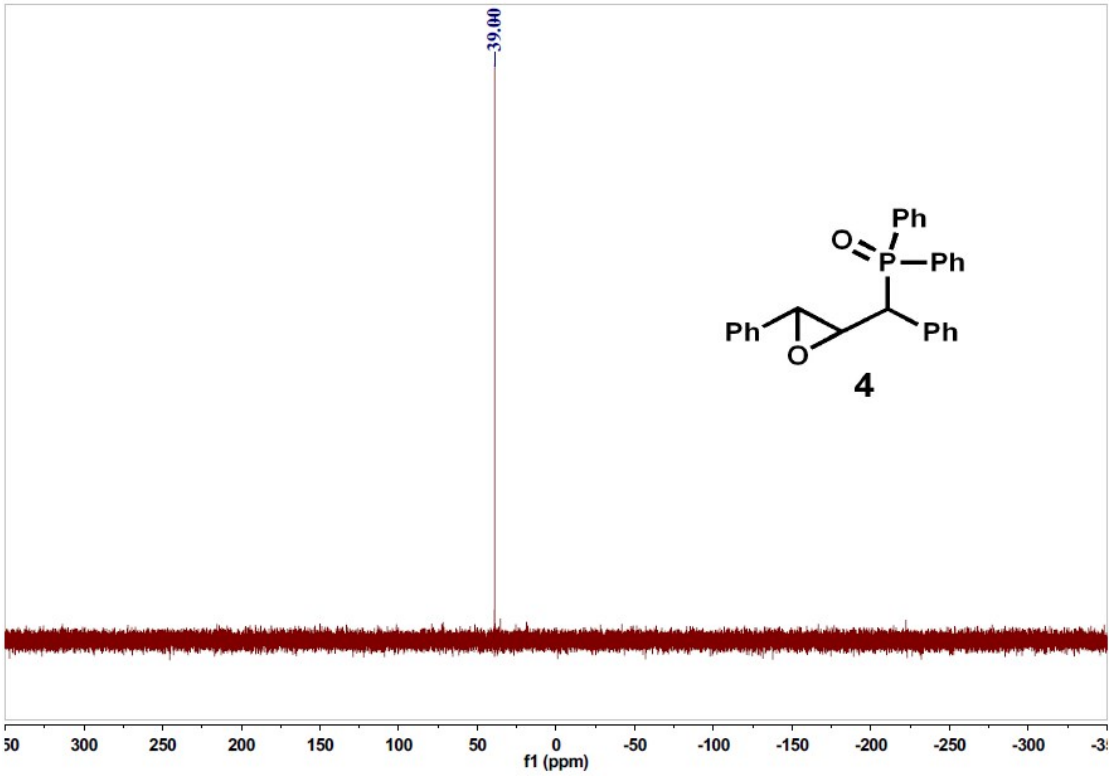

OEt
O
P
OEt
Me
Me
Me
Me
3ak
f1 (ppm)

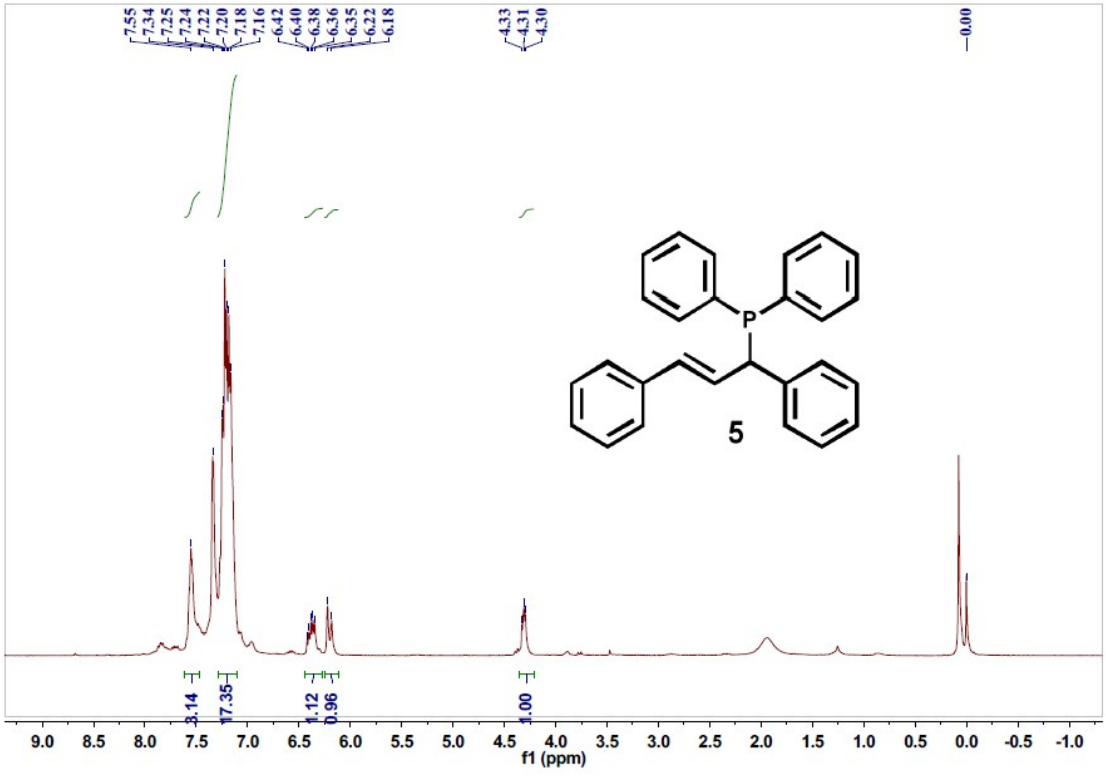

138.19
138.17
138.03
136.82
136.80
135.92
135.85
133.76
133.62
129.43
129.00
128.97
126.86
126.79
124.51
124.42
62.83
62.78
62.77
62.71
50.14
48.78
21.43
21.32
16.60
16.54
16.38
16.32
OEt
O
P
OEt
Me
Me
Me
Me
3ak
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

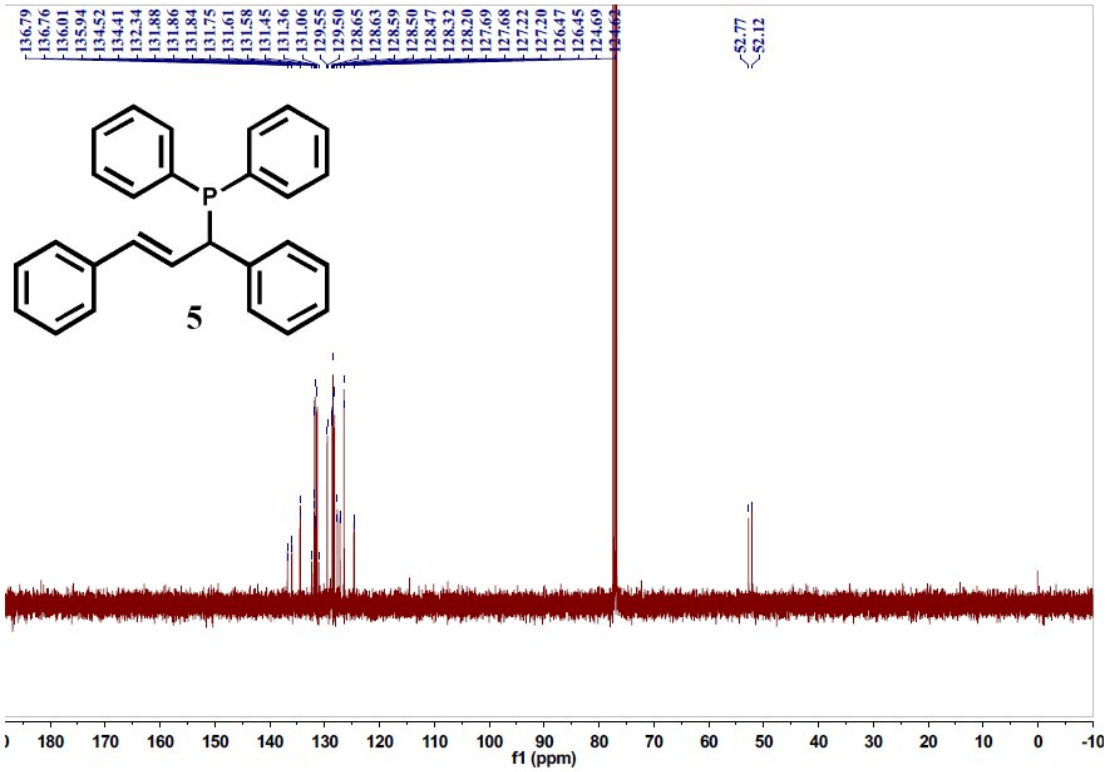

25.80
OEt
O
P
OEt
Me
Me
Me
Me
3ak
50 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -35
f1 (ppm)

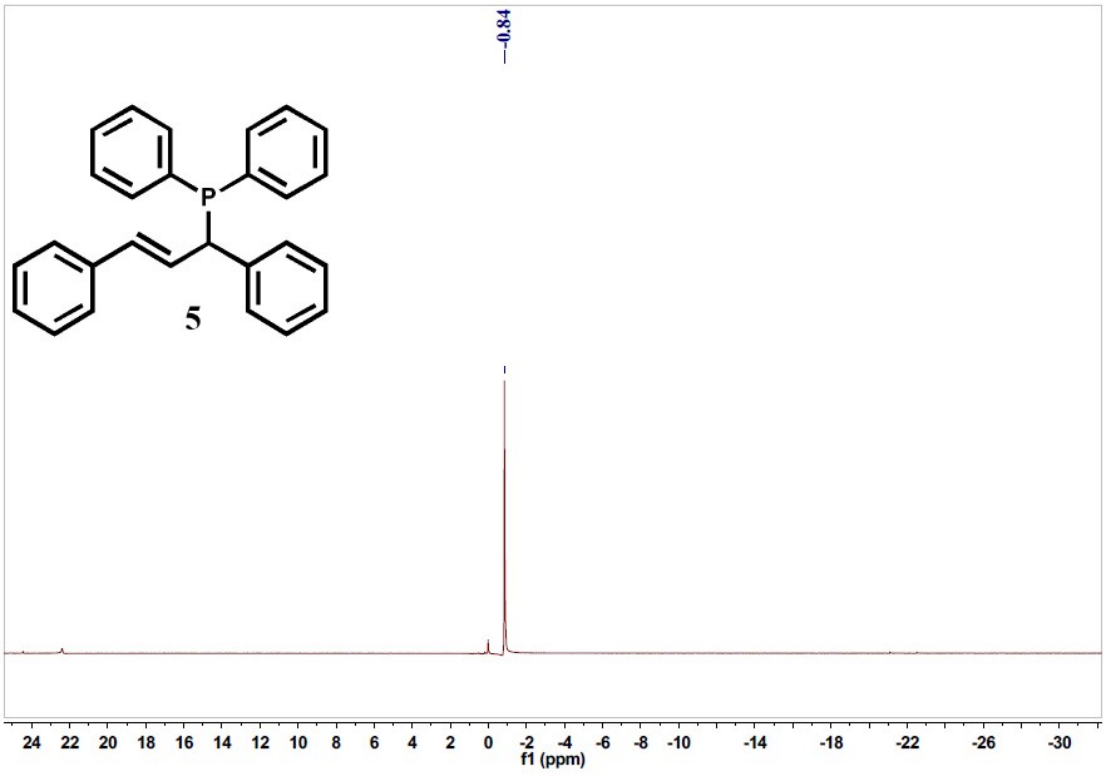

7.92
7.90
7.90
7.88
7.61
7.59
7.58
7.57
7.56
7.54
7.52
7.48
7.46
7.46
7.43
7.31
7.30
7.29
7.27
7.23
7.22
7.21
7.20
4.41
4.39
4.37
4.24
4.22
4.14
4.12
0.00
Ph
O
P
Ph
Ph
Ph
O
4
1.98
4.91
2.21
3.74
7.28
1.02
1.00
1.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-1.0
f1 (ppm)

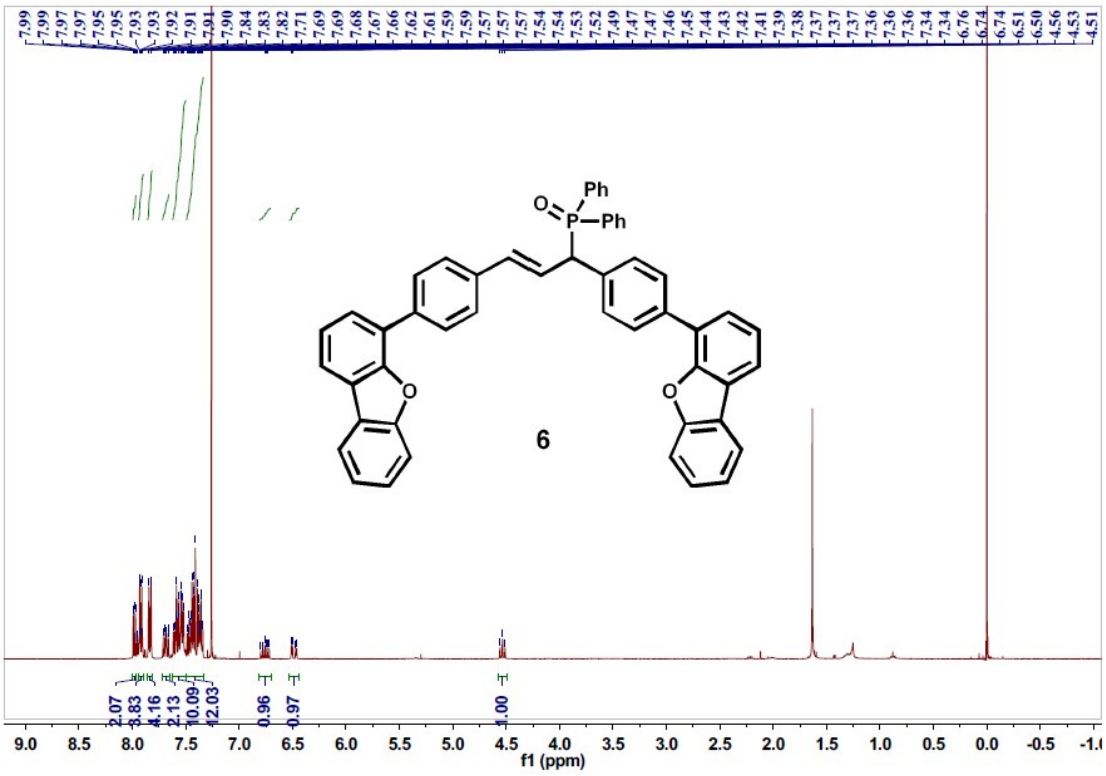

141.61
131.60
131.32
131.23
130.87
130.78
129.12
129.00
128.42
128.28
128.26
128.24
128.16
128.12
127.01
74.38
74.29
74.26
46.41
45.73
Ph
O
P
Ph
Ph
Ph
O
4
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

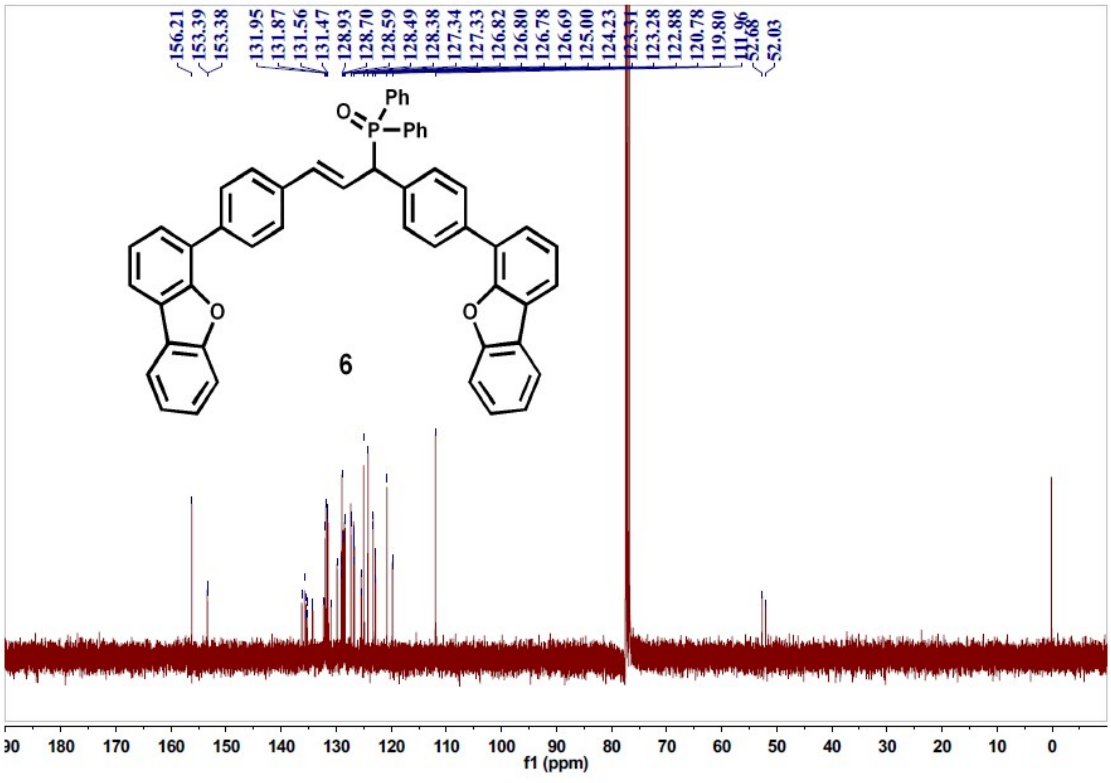

39.00
Ph
O
P
Ph
Ph
Ph
O
4
f1 (ppm)

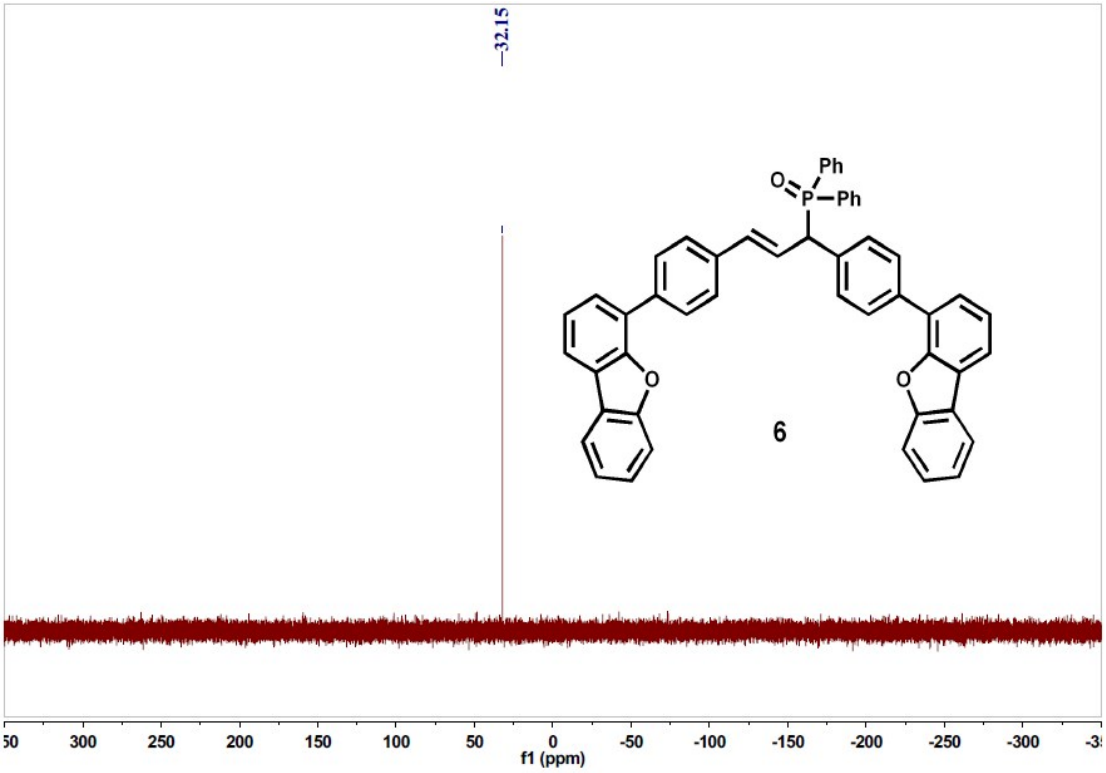

7.55
7.34
7.25
7.24
7.22
7.20
7.18
7.16
6.42
6.40
6.38
6.36
6.35
6.22
6.18
4.33
4.31
4.30
0.00
P
5
3.14
17.35
1.12
0.96
1.00
f1 (ppm)


136.79
136.76
136.01
135.94
134.52
134.41
132.34
131.88
131.86
131.84
131.75
131.61
131.58
131.45
131.36
131.06
129.55
129.50
128.65
128.63
128.59
128.50
128.47
128.32
128.20
127.69
127.68
127.22
127.20
126.47
126.45
124.69
52.77
52.12
P
5
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-0.84
P
5
24 22 20 18 16 14 12 10 8 6 4 2 0 -2 -4 -6 -8 -10 -14 -18 -22 -26 -30
f1 (ppm)


7.99 7.99 7.97 7.97 7.95 7.95 7.93 7.93 7.92 7.91 7.91 7.90 7.84 7.83 7.82 7.71 7.69 7.69 7.68 7.67 7.66 7.62 7.61 7.59 7.59 7.57 7.57 7.57 7.54 7.54 7.53 7.52 7.49 7.47 7.47 7.46 7.45 7.44 7.43 7.42 7.41 7.39 7.38 7.37 7.37 7.37 7.36 7.36 7.36 7.34 7.34 6.76 6.74 6.74 6.51 6.50 4.56 4.53 4.51
Ph
O
P
Ph
6
O
O
2.07 3.83 4.16 2.13 10.09 12.03 0.96 0.97 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


156.21 153.39 153.38 131.95 131.87 131.56 131.47 128.93 128.70 128.59 128.49 128.38 127.34 127.33 126.82 126.80 126.78 126.69 125.00 124.23 123.51 123.28 122.88 120.78 119.80 111.96 111.68 52.03
Ph
O
P
Ph
6
O
O
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


32.15
Ph
O
P
Ph
O
O
6
300
250
200
150
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)