Supporting Information

Chloroform-Based Atherton-Todd-Type Reactions of Alcohols and Thiols with Secondary Phosphine Oxides Generating Phosphinothioates and Phosphinates

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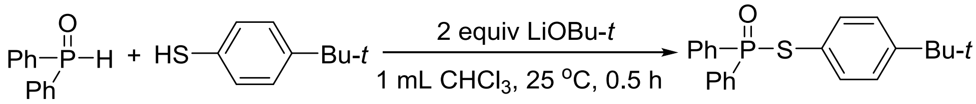
General information

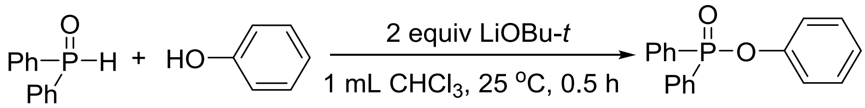
All reactions were carried out in oven-dried Schlenk tubes under N_2 atmosphere. Dry solvents were obtained by purification according to standard methods. Reagents were used as received unless otherwise noted. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR data were obtained on a Bruker-400 spectrometer (400 MHz for 1H, 100 MHz for 13C, and 162 MHz for 31P NMR spectroscopy). Data are report as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q= quartet, m = multiplet), Coupling constants (*J*) are reported in hertz. Mass spectra were measured on a Shimadzu GCMS-QP2010 Plus spectrometer (EI). HRMS were conducted in the Analytical Center at Hunan University, China.

Typical procedure for the synthesis of phosphinothioate



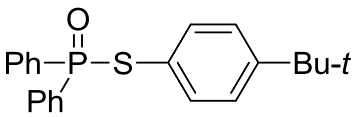
Under N_2 atmosphere, 0.2 mmol diphenylphosphine oxide, 0.1 mmol 4-butylthiophenol, 2 equiv LiOBu-*t* and 1 mL $CHCl_3$ were charged into a 25 mL schlenck tube, and the mixture was stirred at 25 °C for 0.5 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ ethyl acetate=4/1) to afford analytically pure product.

Typical procedure for the synthesis of phosphinates



Under N_2 atmosphere, 0.2 mmol diphenylphosphine oxide, 0.1 mmol phenol, 2 equiv LiOBu-*t* and 1 mL $CHCl_3$ were charged into a 25 mL schlenck tube, and the mixture was stirred at 25 °C for 0.5 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ ethyl acetate=5/1) to afford analytically pure product.

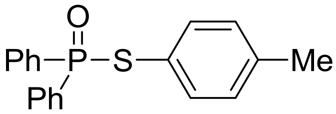
Characterization data of products phosphinothioates and phosphinonates



3a

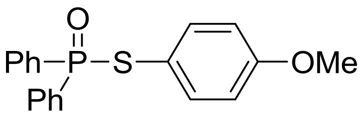
***S*-4-(*tert*-butyl)phenyl) diphenylphosphinothioate.** White solid, m.p.: 122.3-123.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.81 (m, 4H), 7.53-7.49 (m, 2H), 7.46-7.41 (m, 4H), 7.35 (dd, *J* = 8.4, 1.6 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 1.24 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7 (d, *J* = 2.6 Hz), 135.6 (d, *J* = 3.8 Hz), 133.1 (d, *J* = 106.0 Hz), 132.6 (d, *J* = 3.0 Hz), 132.0 (d, *J* = 10.2 Hz), 128.9 (d, *J* = 13.0 Hz), 126.7 (d, *J* = 1.8 Hz), 122.6 (d, *J* =5.3 Hz), 35.0, 31.5. ^{31}P NMR (162 MHz, $CDCl_3$) δ 41.68.

HRMS (EI): calcd for $C_{18}H_{14}OPS$: 366.1207; found: 366.1193.



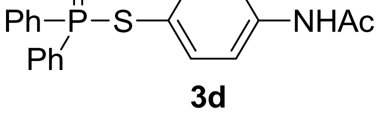
3b

***S*-*p*-tolyl diphenylphosphinothioate.**[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.82 (m, 4H), 7.53-7.49 (m, 2H), 7.46-7.42 (m, 4H), 7.32 (dd, *J* = 8.4, 1.6 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 2H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2 (d, *J* = 2.4 Hz), 135.4 (d, *J* = 3.7 Hz), 132.7 (d, *J* = 106.0 Hz), 132.3 (d, *J* = 3.0 Hz), 131.7 (d, *J* = 10.2 Hz), 123.0 (d, *J* = 1.8 Hz), 128.5 (d, *J* = 13.0 Hz), 122.3 (d, *J* = 5.2 Hz), 21.2. ^{31}P NMR (162 MHz, $CDCl_3$) δ 41.29.



3c

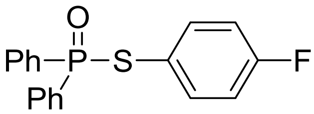
***S*-(4-methoxyphenyl) diphenylphosphinothioate.**[2,3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.81 (m, 4H), 7.53-7.49 (m, 2H), 7.47-7.42 (m, 4H), 7.33 (dd, *J* = 8.8, 1.6 Hz, 2H), 6.73 (d, *J* = 8.8 Hz, 2H), 3.74 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5 (d, *J* = 2.3 Hz), 137.1 (d, *J* = 3.5 Hz), 132.7 (d, *J* = 105.6 Hz), 132.2 (d, *J* = 3.0 Hz), 131.7 (d, *J* = 10.1 Hz), 128.5 (d, *J* = 13.0 Hz), 116.0 (d, *J* = 5.3 Hz), 114.8 (d, *J* = 1.8 Hz), 55.3. ^{31}P NMR (162 MHz, $CDCl_3$) δ 41.33.



3d

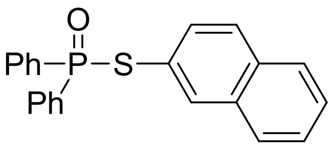
***S*-(4-acetamidophenyl) diphenylphosphinothioate.**[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.75 (s, 1H), 7.86-7.81 (m, 4H), 7.57-7.54 (m, 2H), 7.50-7.43 (m, 6H), 7.21 (d, *J* = 7.2 Hz, 2H), 2.12 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 140.4 (d, *J* = 2.3 Hz), 136.4 (d, *J* = 3.6 Hz), 132.6 (d, *J* = 3.2 Hz), 132.1 (d, *J* = 105.6 Hz), 131.4 (d, *J* = 10.3 Hz), 128.8 (d, *J* = 13.1 Hz), 120.4 (d, *J* = 1.4 Hz), 117.9 (d, *J* = 5.3 Hz), 24.5. ^{31}P

NMR (162 MHz, $CDCl_3$) δ 42.69.



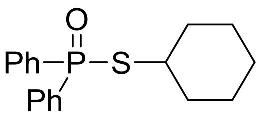
3e

***S*-(4-fluorophenyl) diphenylphosphinothioate.**[4] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.81 (m, 4H), 7.55-7.50 (m, 2H), 7.47-7.39 (m, 6H), 6.90 (t, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4 (dd, *J* = 248.2, 2.5 Hz), 137.4 (dd, *J* = 8.5, 3.7 Hz), 132.5 (d, *J* = 3.0 Hz), 132.3 (d, *J* = 106.3 Hz), 131.6 (d, *J* = 10.2 Hz), 128.6 (d, *J* = 13.1 Hz), 121.2 (dd, *J* = 5.0, 3.3 Hz), 116.4 (dd, *J* = 22.0, 1.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 41.58 (d, $J_{F\text{-}P}$ = 4.2 Hz).



3g

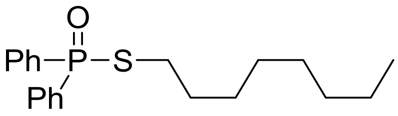
***S*-naphthalen-2-yl diphenylphosphinothioate.**[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.90-7.85 (m, 4H), 7.75-7.69 (m, 2H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.52-7.41 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.4 (d, *J* = 5.0 Hz), 133.5 (d, *J* = 1.8 Hz), 133.0 (d, *J* = 1.4 Hz), 132.5 (d, *J* = 106.2 Hz), 132.4 (d, *J* = 3.0 Hz), 131.7 (d, *J* = 10.3 Hz), 131.6 (d, *J* = 3.1 Hz), 128.7 (d, *J* = 1.2 Hz), 128.6 (d, *J* = 13.1 Hz), 127.8, 127.6, 126.9, 126.5, 123.5 (d, *J* = 5.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 41.55.



3h

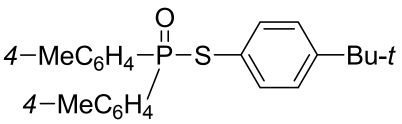
***S*-cyclohexyl diphenylphosphinothioate.**[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.85 (m, 4H), 7.52-7.43 (m, 6H), 3.35-3.25 (m, 1H), 1.96-1.92 (m, 2H), 1.68-1.64 (m, 2H), 1.56-1.46 (m, 3H), 1.32-1.21 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.1 (d, *J* = 106.1 Hz), 132.1 (d, *J* = 2.9 Hz), 131.4 (d, *J* = 10.3 Hz), 128.6 (d, *J* = 13.0 Hz), 44.4

(d, J = 2.1 Hz), 35.6 (d, J = 3.9 Hz), 25.7, 25.3. ^{31}P NMR (162 MHz, $CDCl_3$) δ 41.87.



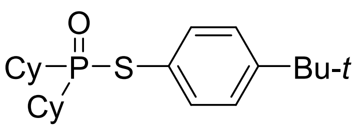
3i

***S*-octyl diphenylphosphinothioate.**[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.897.83 (m, 4H), 7.52-7.42 (m, 6H), 2.80-2.74 (m, 2H), 1.63-1.55 (m, 2H), 1.30-1.17 (m, 10H), 0.84 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.5 (d, J = 106.3 Hz), 132.2 (d, J = 3.0 Hz), 131.5 (d, J = 10.3 Hz), 128.6 (d, J = 12.9 Hz), 31.7, 30.5 (d, J = 4.9 Hz), 29.3 (d, J = 2.3 Hz), 29.0, 28.9, 28.6, 22.6, 14.1. ^{31}P NMR (162 MHz, $CDCl_3$) δ 43.01.



3j

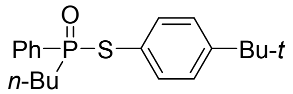
***S*-(4-(*tert*-butyl)phenyl) di-p-tolylphosphinothioate.** White solid, m.p.: 151.1-152.0 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (dd, J = 12.8, 8.0 Hz, 4H), 7.36-7.33 (m, 2H), 7.25-7.20 (m, 6H), 2.38 (s, 6H), 1.24 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.0 (d, J = 2.4 Hz), 142.7 (d, J = 3.0 Hz), 135.1 (d, J = 3.7 Hz), 131.7 (d, J = 10.6 Hz), 129.7 (d, J = 108.7 Hz), 129.2 (d, J = 13.5 Hz), 126.3 (d, J = 1.7 Hz), 122.8 (d, J = 5.2 Hz), 34.6, 31.2, 21.6. ^{31}P NMR (162 MHz, $CDCl_3$) δ 42.09. HRMS (EI): calcd for $C_{24}H_{27}OPS$: 394.1520; found: 394.1503.



3k

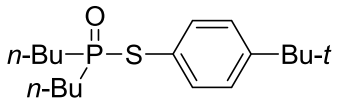
***S*-(4-(*tert*-butyl)phenyl) dicyclohexylphosphinothioate.** Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 2.04-1.82 (m, 12H), 1.70 (bs, 2H), 1.53-1.38 (m, 4H), 1.30 (s, 9H), 1.22 (bs, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.8 (d, J = 1.8 Hz), 135.4 (d, J = 3.1 Hz), 126.3 (d, J = 1.0 Hz), 123.0 (d,

J = 5.1 Hz), 40.1 (d, J = 62.1 Hz), 34.6, 31.2, 26.5 (d, J = 13.6 Hz), 26.2 (d, J = 3.5 Hz), 26.1 (d, J = 3.4 Hz), 25.9 (d, J = 1.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 67.46. HRMS (EI): calcd for $C_{22}H_{35}OPS$: 378.5515; found: 378.2154.



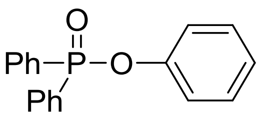
3l

***S*-(4-(*tert*-butyl)phenyl) butyl(phenyl)phosphinothioate.** Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, J = 12.1, 7.5 Hz, 2H), 7.56 – 7.48 (m, 1H), 7.46-7.41 (m, 2H), 7.36 (d, J = 7.7 Hz, 2H), 7.27 (d, J = 5.0 Hz, 2H), 2.24-2.07 (m, 2H), 1.57-1.45 (m, 2H), 1.39-1.29 (m, 2H), 1.27 (s, 9H), 0.85 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.3 (d, J = 2.3 Hz), 135.2 (d, J = 3.5 Hz), 132.5 (d, J = 98.2 Hz), 132.1 (d, J = 2.9 Hz), 131.2 (d, J = 9.7 Hz), 128.4 (d, J = 12.5 Hz), 126.4 (d, J = 1.6 Hz), 122.3 (d, J = 5.1 Hz), 34.6, 32.9 (d, J = 71.0 Hz), 31.2, 24.3 (d, J = 4.6 Hz), 23.8 (d, J = 16.1 Hz), 13.5. ^{31}P NMR (162 MHz, $CDCl_3$) δ 50.39. HRMS (EI): calcd for $C_{20}H_{27}OPS$: 346.1520; found: 346.1507.



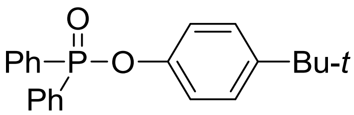
3m

***S*-(4-(*tert*-butyl)phenyl) dibutylphosphinothioate.** Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 1.92-1.85 (m, 4H), 1.68-1.56 (m, 4H), 1.42-1.37 (m, 4H), 1.31 (s, 9H), 0.91 (t, J = 7.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.4 (d, J = 2.2 Hz), 135.3 (d, J = 3.3 Hz), 126.6 (d, J = 1.5 Hz), 122.4 (d, J = 5.0 Hz), 34.7, 31.2, 31.1 (d, J = 65.6 Hz), 24.5 (d, J = 4.2 Hz), 23.9 (d, J = 16.0 Hz), 13.6. ^{31}P NMR (162 MHz, $CDCl_3$) δ 61.84. ^{31}P NMR (162 MHz, $CDCl_3$) δ 67.46. HRMS (EI): calcd for $C_{18}H_{31}OPS$: 326.1833; found: 326.1826.



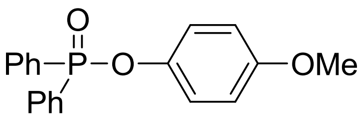
4a

phenyl diphenylphosphinate.[6] **1**H NMR (400 MHz, $CDCl_3$) δ 7.92-7.86 (m, 4H), 7.51-7.47 (m, 2H), 7.45-7.40 (m, 4H), 7.21 (d, *J* = 4.4 Hz, 4H), 7.07-7.02 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.9 (d, *J* = 8.1 Hz), 132.5 (d, *J* = 2.8 Hz), 131.8 (d, *J* = 10.3 Hz), 131.0 (d, *J* = 137.4 Hz), 129.7, 128.6 (d, *J* = 13.5 Hz), 124.6, 120.8 (d, *J* = 4.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.72.



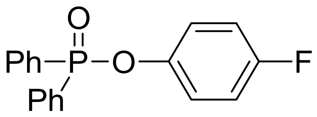
4b

4-(*tert*-butyl)phenyl diphenylphosphinate.[7] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.87 (m, 4H), 7.56-7.52 (m, 2H), 7.49-7.44 (m, 4H), 7.23 (d, *J* = 8.8 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 1.25 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.4 (d, *J* = 8.2 Hz), 147.4 (d, *J* = 0.8 Hz), 132.4 (d, *J* = 2.8 Hz), 131.8 (d, *J* = 10.3 Hz), 131.3 (d, *J* = 137.4 Hz), 128.6 (d, *J* = 13.3 Hz), 126.5, 120.1 (d, *J* = 4.6 Hz), 34.3, 31.4. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.19.



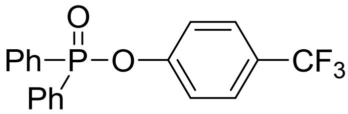
4c

4-methoxyphenyl diphenylphosphinate.[7] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.85 (m, 4H), 7.51-7.47 (m, 2H), 7.44-7.40 (m, 4H), 7.11 (d, *J* = 8.8 Hz, 2H), 6.72 (d, *J* = 9.2 Hz, 2H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4 (d, *J* = 0.9 Hz), 144.3 (d, *J* = 8.3 Hz), 132.4 (d, *J* = 2.9 Hz), 131.8 (d, *J* = 10.2 Hz), 131.0 (d, *J* = 137.1 Hz), 128.6 (d, *J* = 13.3 Hz), 121.7 (d, *J* = 4.4 Hz), 114.6, 55.5. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.52.



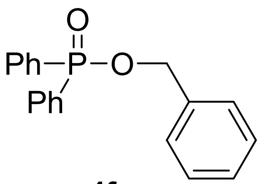
4d

4-fluorophenyl diphenylphosphinate.[7] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.85 (m, 4H), 7.55-7.51 (m, 2H), 7.48-7.43 (m, 4H), 7.17-7.14 (m, 2H), 6.0 (t, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5 (dd, *J* = 241.9, 1.2 Hz), 146.6 (dd, *J* = 8.2, 2.7 Hz), 132.6 (d, *J* = 2.8 Hz), 131.8 (d, *J* = 10.4 Hz), 130.6 (d, *J* = 137.2 Hz), 128.7 (d, *J* = 13.4 Hz), 122.2 (dd, *J* = 8.3, 4.5 Hz), 116.2 (d, *J* = 23.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.21.



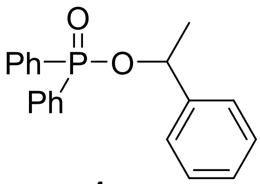
4e

4-(trifluoromethyl)phenyl diphenylphosphinate.[8] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.87 (m, 4H), 7.58-7.46 (m, 8H), 7.33 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.5 (db, *J* = 7.9 Hz), 132.8 (d, *J* = 2.8 Hz), 131.7 (d, *J* = 10.5 Hz), 130.4 (d, *J* = 137.3 Hz), 128.78 (d, *J* = 13.5 Hz), 127.1 (q, *J* = 3.7 Hz), 126.9 (q, *J* = 33.4 Hz), 123.9 (q, *J* = 270.1 Hz), 121.0 (d, *J* = 5.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.72.



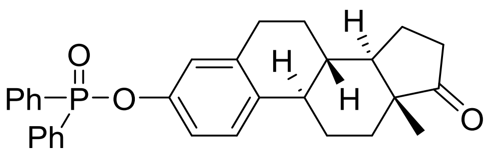
4f

benzyl diphenylphosphinate.[9] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.81 (m, 4H), 7.51-7.47 (m, 2H), 7.44-7.39 (m, 4H), 7.37-7.26 (m, 5H), 5.06 (d, *J* = 6.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.4 (d, *J* = 7.5 Hz), 132.3 (d, *J* = 2.8 Hz), 131.7 (d, *J* = 10.2 Hz), 131.3 (d, *J* = 136.0 Hz), 128.7, 128.6 (d, *J* = 4.0 Hz), 128.3, 127.9, 66.4 (d, *J* = 5.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.42.



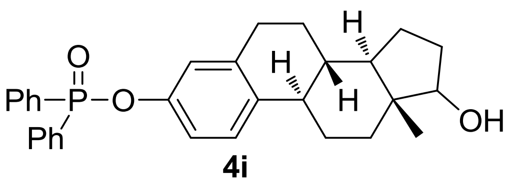
4g

1-phenylethyl diphenylphosphinate. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.81 (m, 2H), 7.69-7.6(m, 2H), 7.54-7.36 (m, 4H), 7.34-7.13 (m, 7H), 5.58-5.45 (m, 1H), 1.66 (d, J = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1 (d, J = 5.0 Hz), 132.7 (d, J = 93.3 Hz), 132.1 (d, J = 2.8 Hz), 132.0 (d, J = 2.8 Hz), 131.9 (d, J = 10.2 Hz), 131.5 (d, J = 10.1 Hz), 131.4 (d, J = 99.0 Hz), 128.5, 128.5, 128.4 (d, J = 3.3Hz), 128.1 (d, J = 30.7Hz), 125.9, 74.5 (d, J = 5.6 Hz), 25.1 (d, J = 3.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.00.



4h

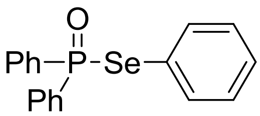
13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl diphenylphosphinate. White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, J = 12.4, 7.6 Hz, 4H), 7.54-7.43 (m, 6H), 7.12 (d, J = 8.4 Hz, 1H), 6.97 (s, 1H), 6.92 (d, J = 8.4 Hz, 1H), 2.82-2.80 (m, 2H), 2.48 (dd, J = 18.8, 8.8 Hz, 1H), 2.33-2.30 (m, 1H), 2.19-1.91 (m, 5H), 1.61-1.35 (m, 6H), 0.88 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 220.8, 148.7 (d, J = 8.4 Hz), 138.2, 136.1, 132.4 (d, J = 2.6 Hz), 131.8 (d, J = 10.3 Hz), 131.2 (d, J = 137.3 Hz), 128.6 (d, J = 13.3 Hz), 126.5, 120.8 (d, J = 4.4 Hz), 117.9 (d, J = 4.6 Hz), 50.4, 47.9, 44.0, 38.0, 35.9, 31.5, 29.4, 26.3, 25.7, 21.6, 13.8. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.13. HRMS (EI): calcd for $C_{30}H_{31}O_3P$: 470.2011; found: 470.1996.



4i

17-hydroxy-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-

cyclopenta[a]phenanthren-3-yl diphenylphosphinate.[10] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 12.5, 7.2 Hz, 4H), 7.54-7.43 (m, 6H), 7.12 (d, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 10.0 Hz, 2H), 3.70 (t, *J* = 8.8 Hz, 1H), 2.77-2.75 (m, 2H), 2.24-2.05 (m, 3H), 1.93-1.90 (m, 1H), 1.84-1.80 (m, 1H), 1.70-1.62 (m, 1H), 1.52-1.09 (m, 8H), 0.74 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5 (d, *J* = 8.4 Hz), 138.5, 136.7, 132.4 (d, *J* = 2.8 Hz), 131.8 (d, *J* = 10.3 Hz), 131.3 (d, J = 137.5 Hz), 128.6 (d, *J* = 13.3 Hz), 126.5, 120.7 (d, *J* = 4.7 Hz), 117.7 (d, *J* = 4.7 Hz), 81.8, 50.0, 44.0, 43.2, 38.5, 36.7, 30.5, 29.5, 27.0, 26.1, 23.1, 11.1. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.03.



5a

***Se*-phenyl diphenylphosphinoselenoate.**[11] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.80 (m, 4H), 7.51-7.43 (m, 8H), 7.25-7.23 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0 (d, *J* = 3.3 Hz), 133.1 (d, *J* = 97.3 Hz), 131.9 (d, *J* = 3.2 Hz), 131.0 (d, *J* = 10.5 Hz), 128.9 (d, *J* = 1.5 Hz), 128.4 (d, *J* = 2.0 Hz), 128.2 (d, *J* = 13.1 Hz), 123.4 (d, *J* = 5.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 39.93.

Reference

1. G. Kumaraswamy, and R. Raju, *Adv. Synth. Catal.*, 2014, **356**, 2591-2598.
2. J. Wang, X. Huang, Z. Ni, S. Wang, J. Wu and Y. Pan, *Green Chem.*, 2015, **17**, 314-319.
3. L. Y. Kuo, A. P. Blum and M. Sabat, *Inorg. Chem.*, 2005, **44**, 5537-554.
4. R. D. Cook and L. Rahhal-Arabi, *Tetrahedron Lett.*, 1985, **26**, 3147-3150.
5. H. Schindlbauer and W. Prikoszovich, *Monatsh. Chem.*, 1968, **99**, 1792-1798.
6. Z. S. Han, L. Zhang, Y. Xu, J. D. Sieber, M. A. Marsini, Z. Li, J. T. Reeves, K. R. Fandrick, N. D. Patel, J.-N. Desrosiers, B. Qu, A. Chen, D. M. Rudzinski, L. P. Samankumara, S. Ma, N. Grinberg, F. Roschangar, N. K. Yee, G. Wang, J. J. Song and C. H. Senanayake, *Angew. Chem. Int. Ed.*, 2015, **54**, 5474-5477.
7. B. Xiong, X. Feng, L. Zhu, T. Chen, Y. Zhou, C. T. Au and S. Yin, *ACS Catal.*, 2015, **5**, 537-

543.

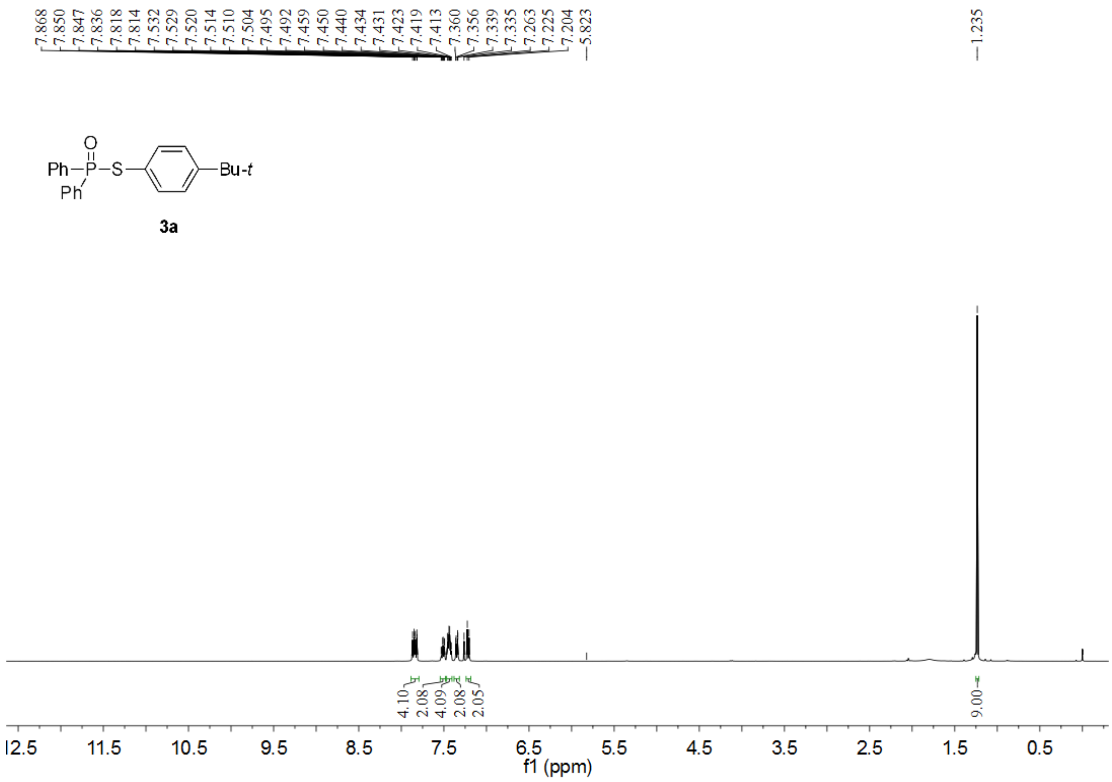
8. E. Buncel, A. Chen, M. Decouzon, S. A. Fancy, J. F. Gal, M. Herreros and P. C. Maria, *J. Mass Spectrom.*, 1998, **33**, 757-765.

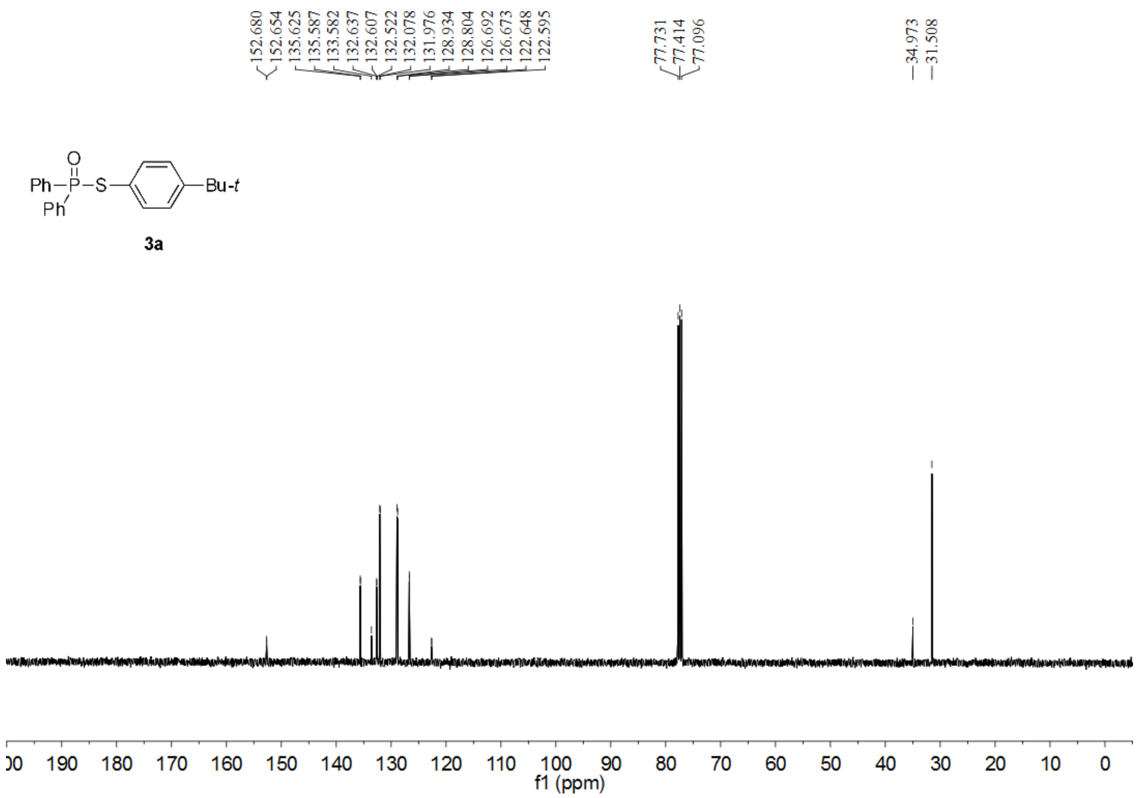
9. J. Xu, P. Zhang, X. Li, Y. Gao, J. Wu, G. Tang and Y. Zhao, *Adv. Synth. Catal.*, 2014, **356**, 3331-3335.

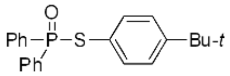
10. Q. Yang, Y. Wang, G. Wang, J. Gao, X. Zhao, D. Liu and H. Mi, *J. Appl. Polym. Sci.*, 2013, *130*, 595-602.

11. S. Kawaguchi, M. Kotani, S. Atobe, A. Nomoto, M. Sonoda and A. Ogawa, *Organometallics*, 2011, **30**, 6766−6769.

Copies of 1H, 13C and 31P NMR spectroscopes



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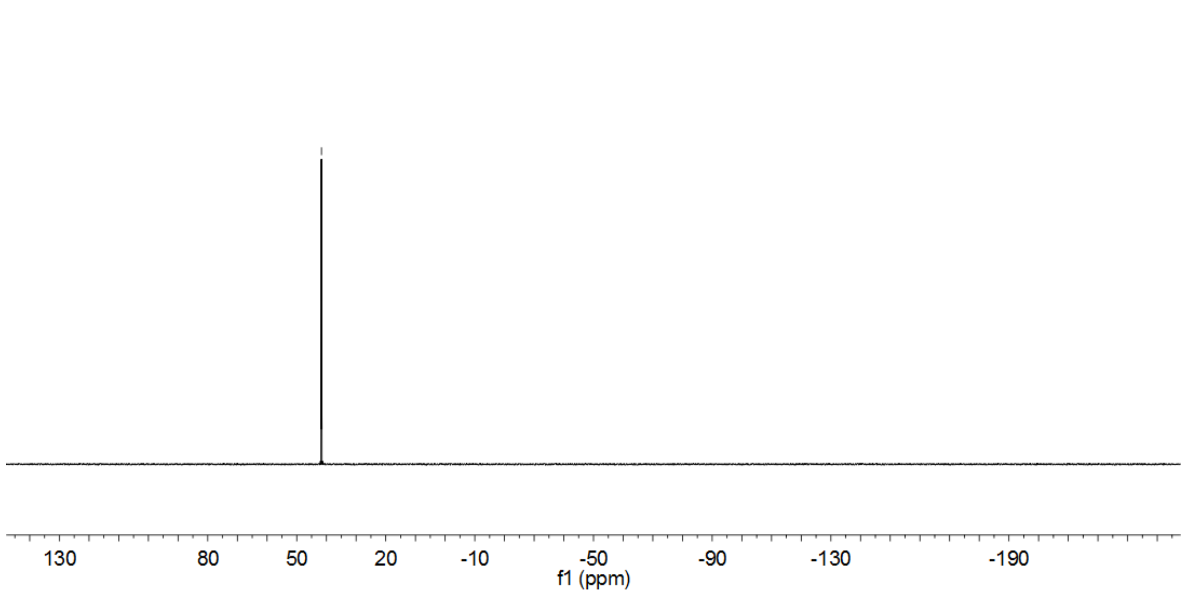
P
S
Bu-t
Ph

3a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80
50
20
-10
-50
-90
-130
-190
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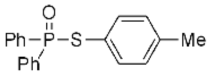
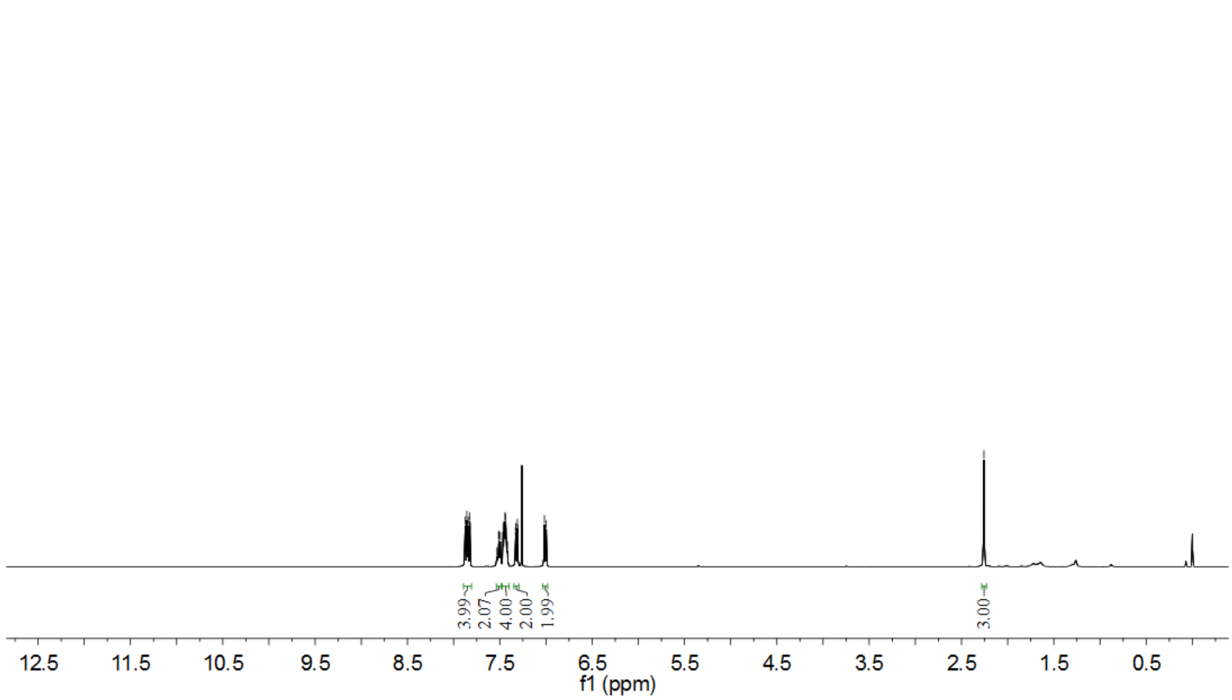

O
Ph
P
S
Me
Ph

3b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7.825
7.821
7.531
7.514
7.509
7.494
7.464
7.455
7.444
7.435
7.427
7.423
7.417
7.331
7.327
7.310
7.306
7.017
6.997
2.257
3.99
2.07
4.00
2.00
1.99
3.00
12.5
11.5
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7.5
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5.5
4.5
3.5
2.5
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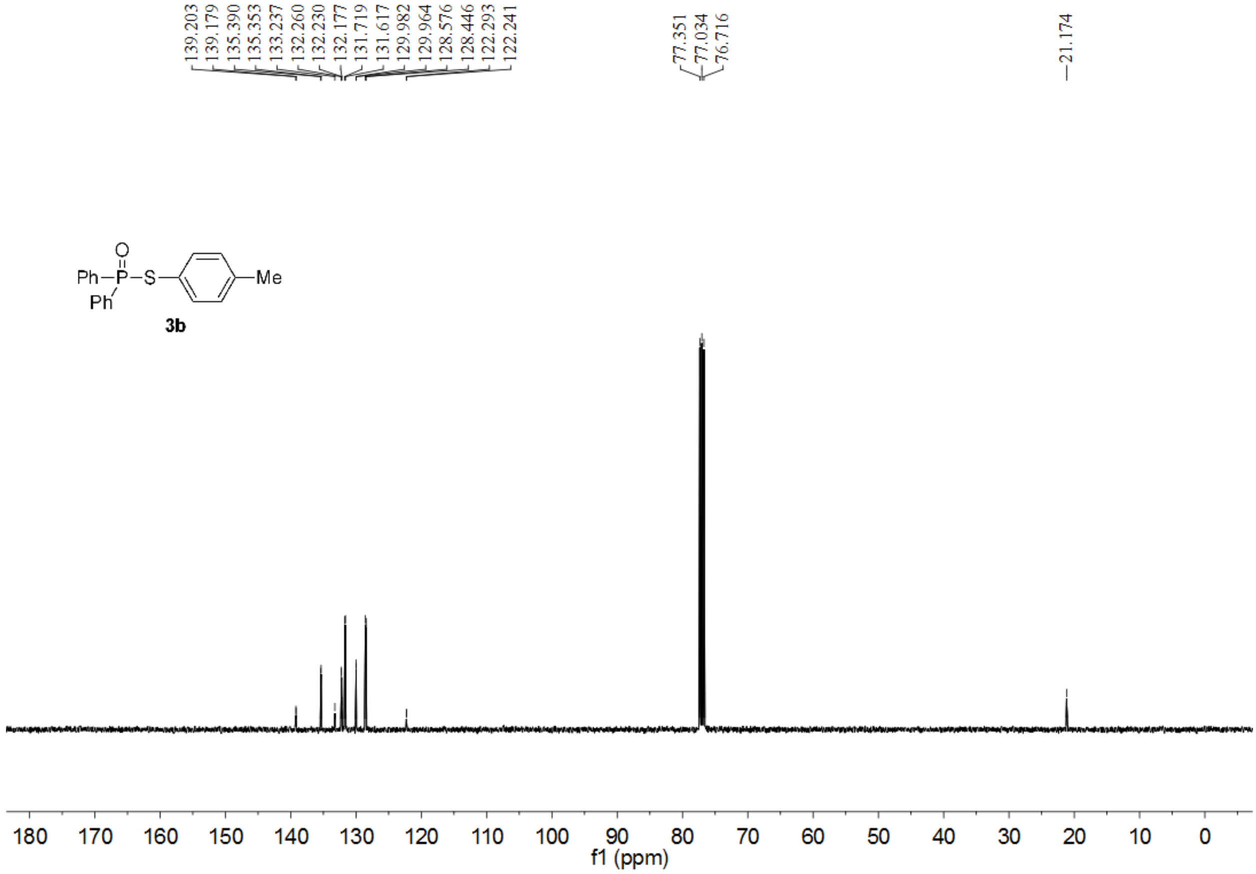
0.5
f1 (ppm)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128.576
128.446
122.293
122.241
77.351
77.034
76.716
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Ph
O
P
S
Me
Ph
3b
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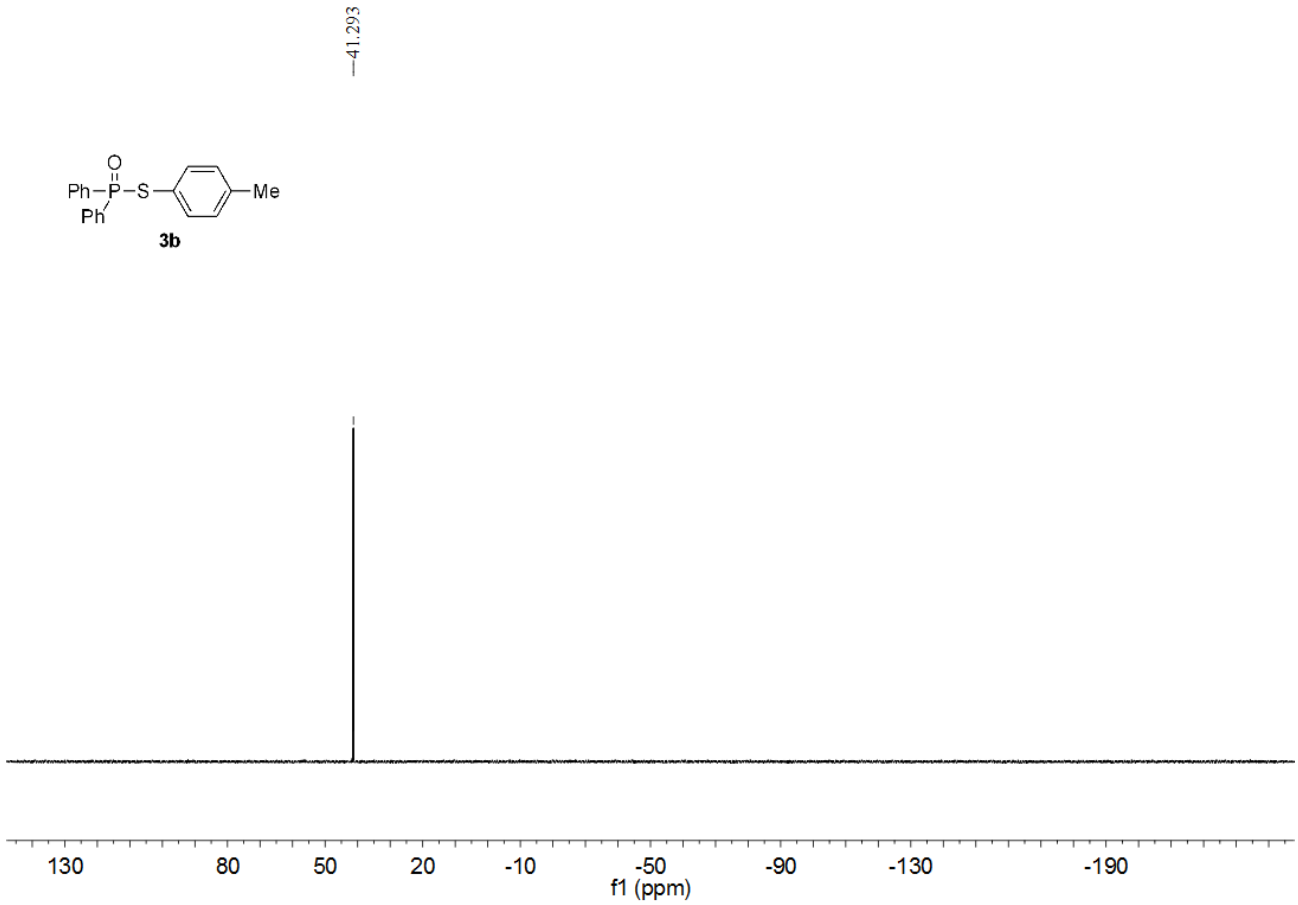
f1 (ppm)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Ph
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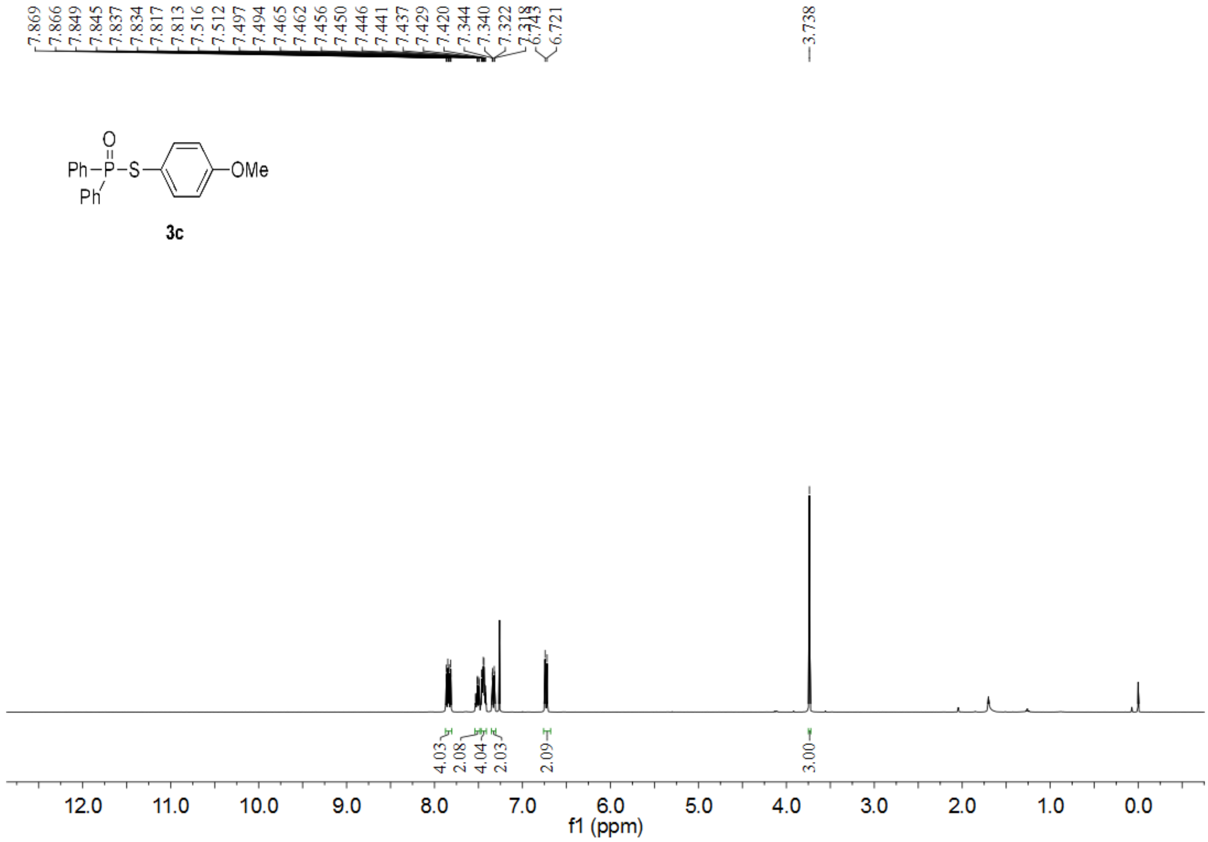

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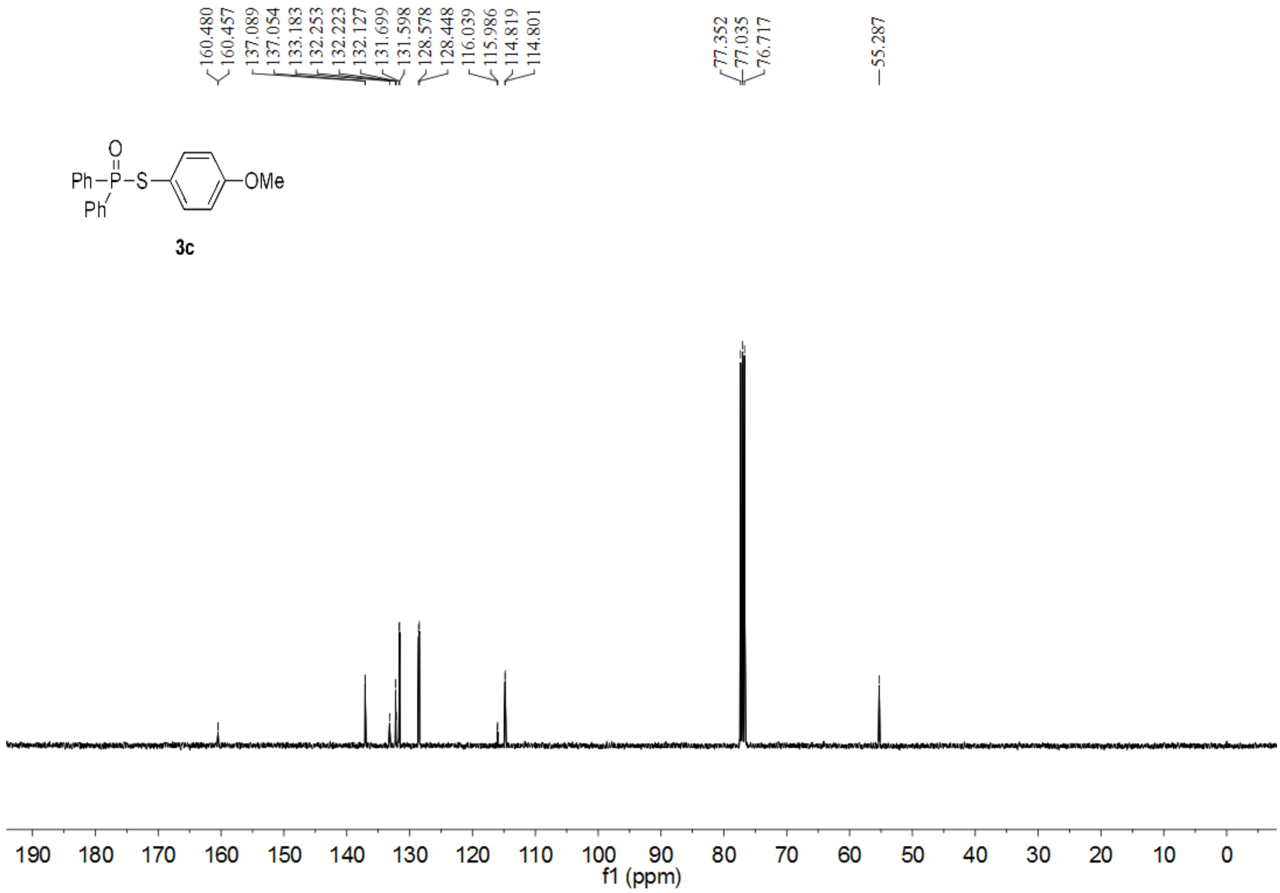

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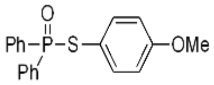
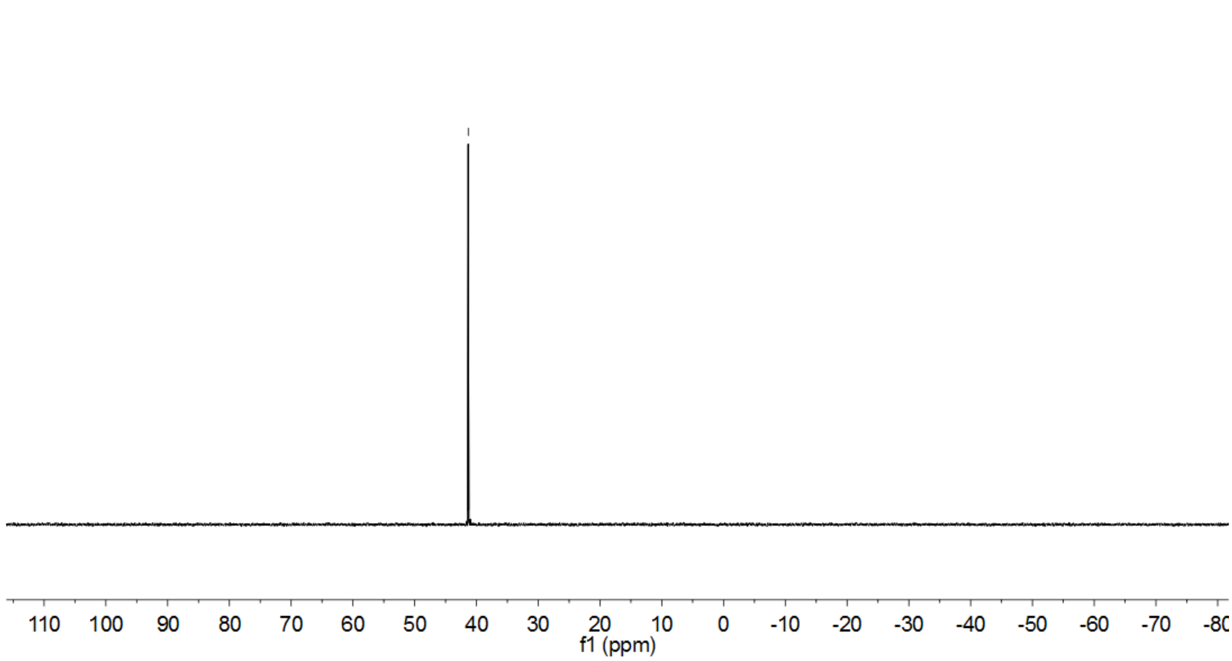


O
Ph
P
S
OMe
Ph

3c



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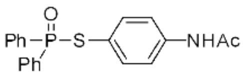
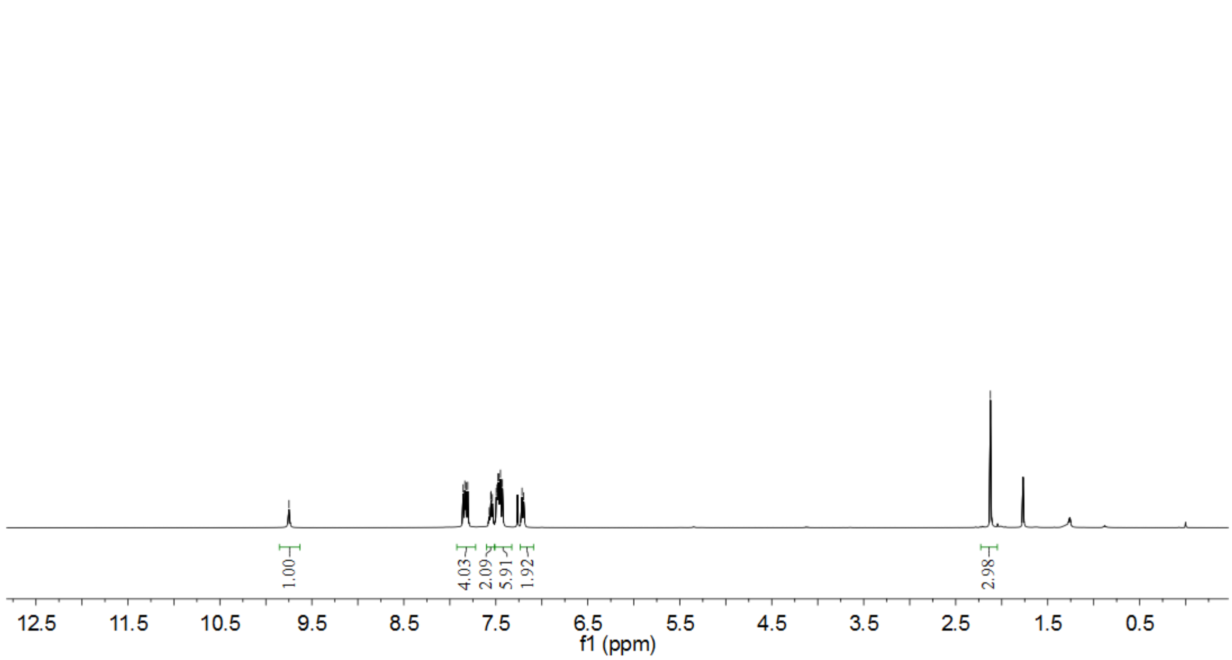
f1 (ppm)



O
Ph
P
S
NHAc
Ph

3d



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7.855 7.837 7.823 7.805 7.571 7.556 7.552 7.537 7.496 7.487 7.477 7.468 7.459 7.449 7.428 7.215 7.197
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1.00
4.03
2.09
5.91
1.92
2.98
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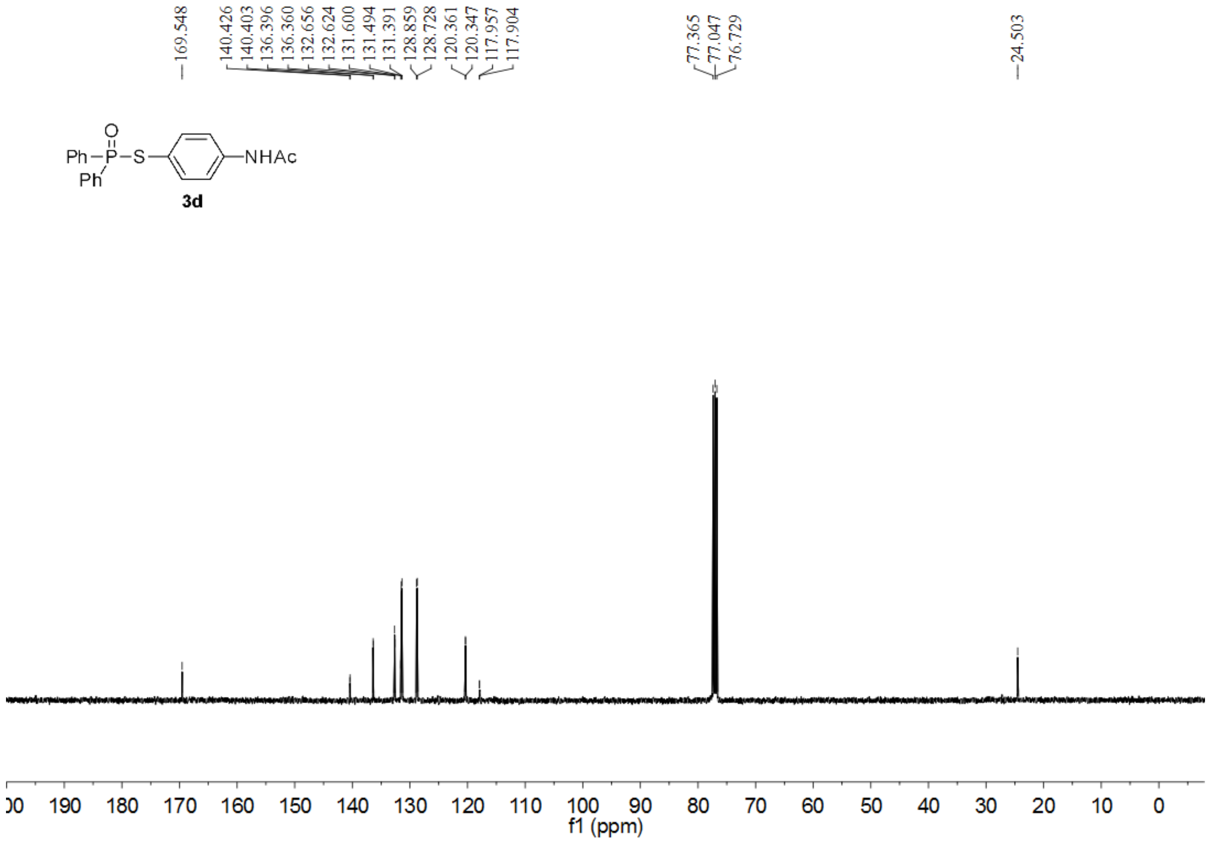
f1 (ppm)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120.347
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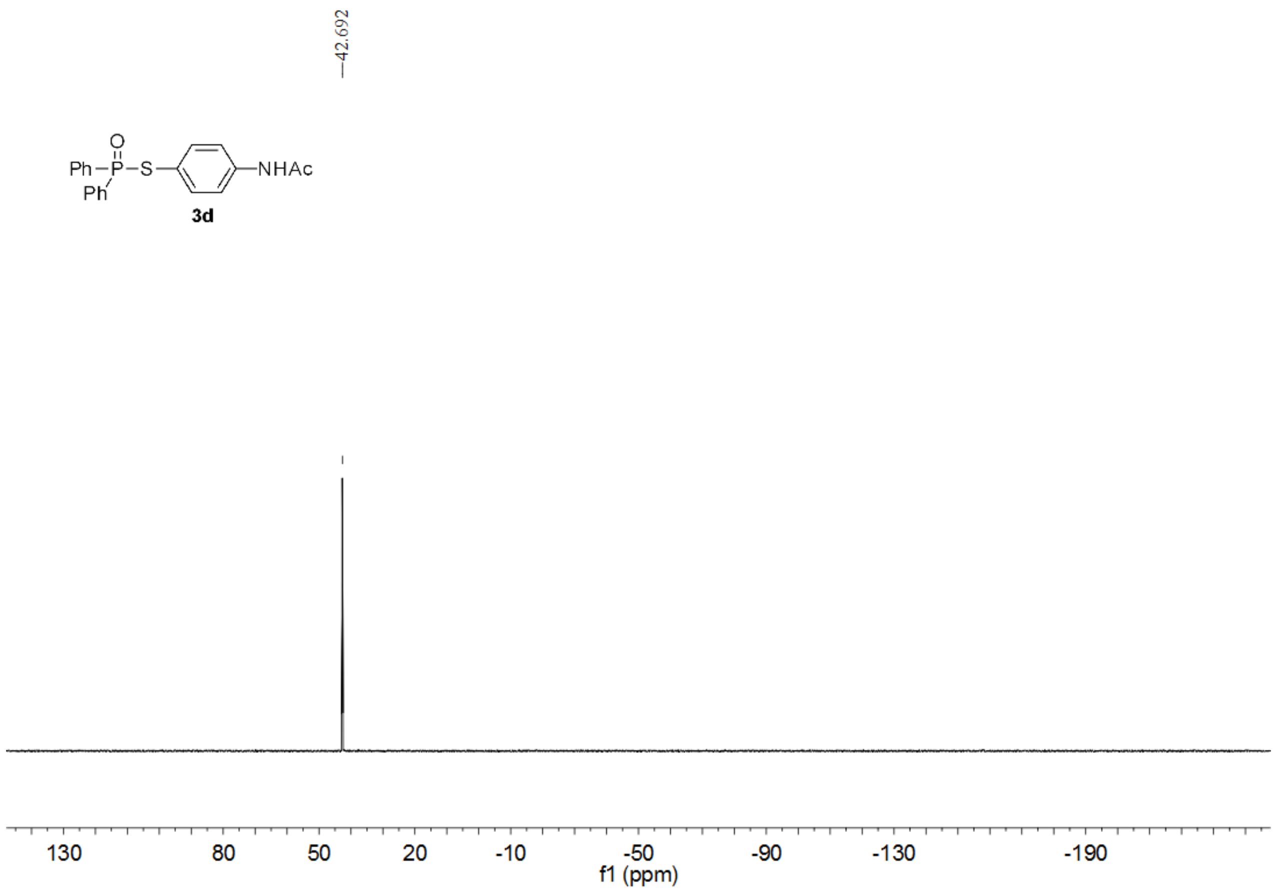
24.503
Ph
P
O
S
NHAc
Ph
3d
f1 (ppm)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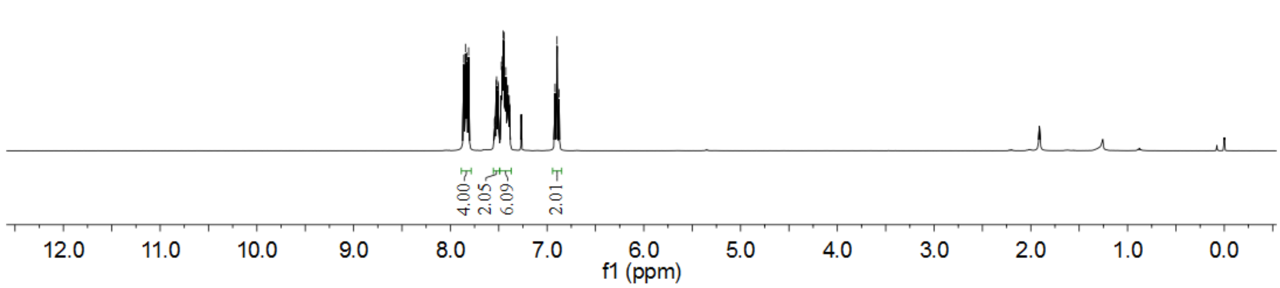
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7.423
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7.409
7.404
7.400
7.391
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2.05
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11.0
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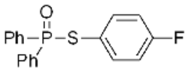
f1 (ppm)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P
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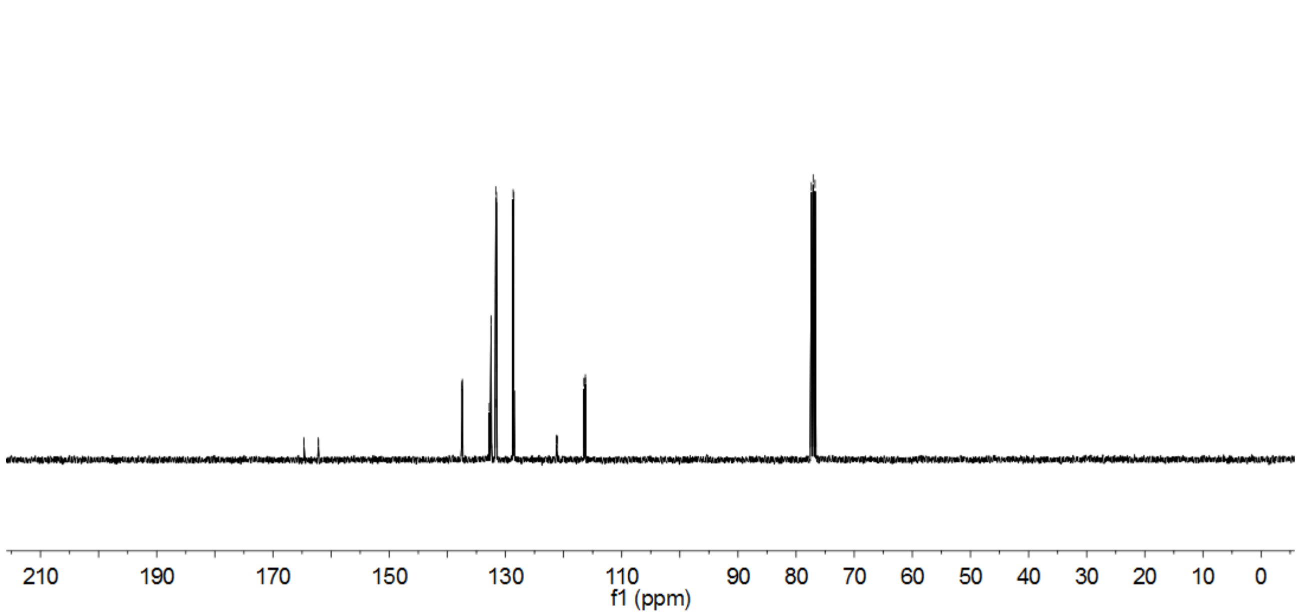
Ph

3e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131.552
128.682
128.551
121.204
121.171
121.120
116.470
116.452
116.250
116.233
77.377
77.059
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50
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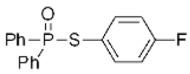
f1 (ppm)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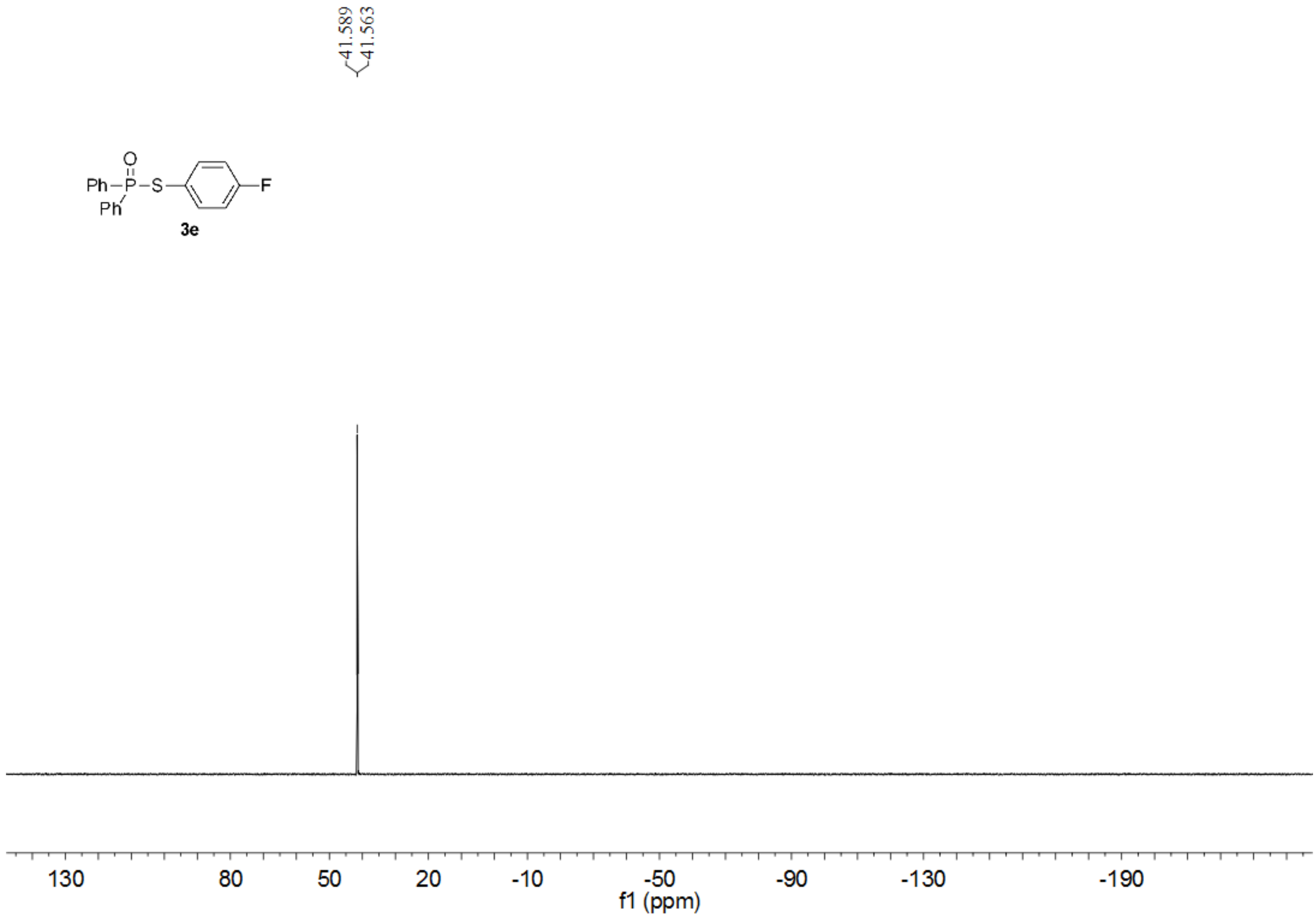
P
O
S
F
Ph

3e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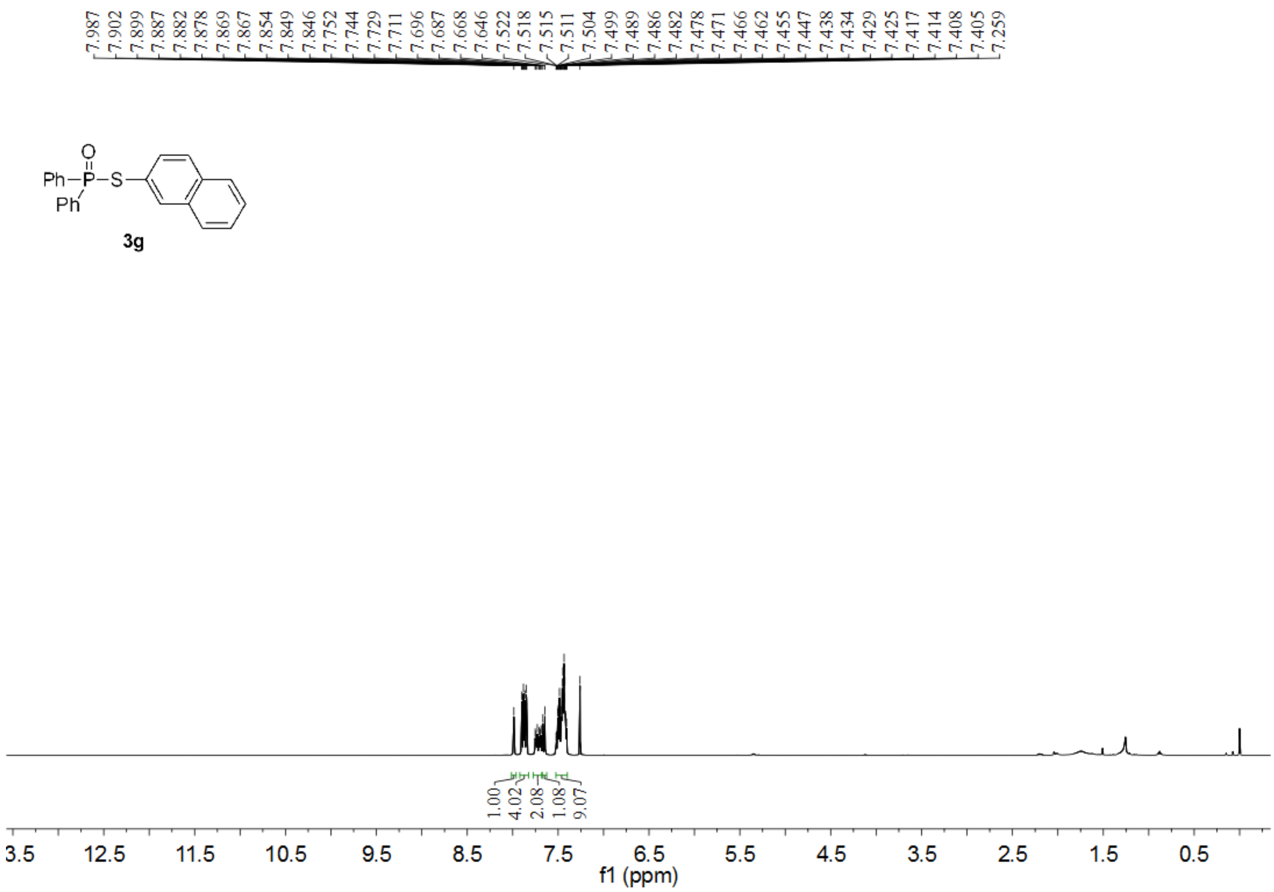
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7.511
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7.499
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O
P
S
Ph
3g
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4.02
2.08
1.08
9.07
3.5
12.5
11.5
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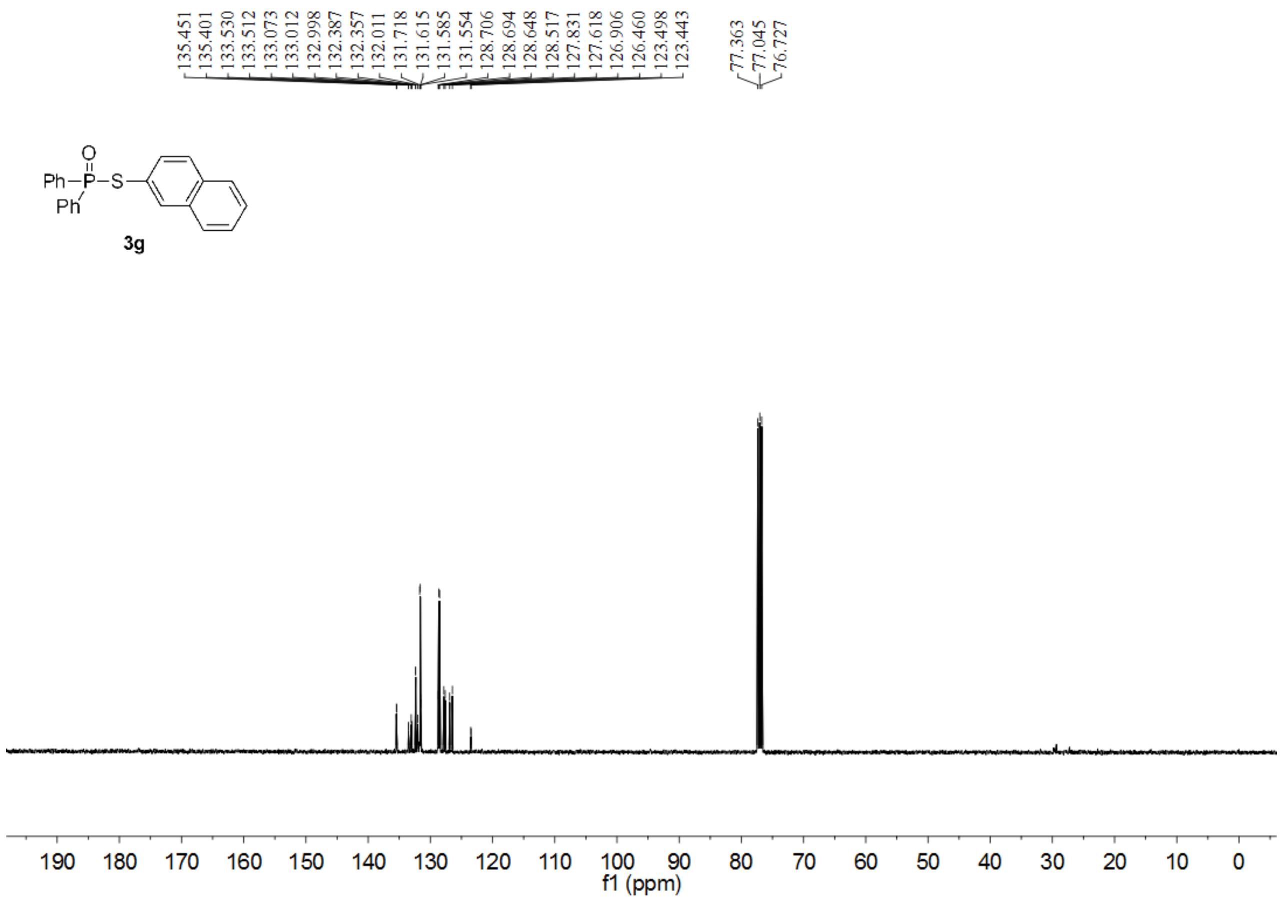

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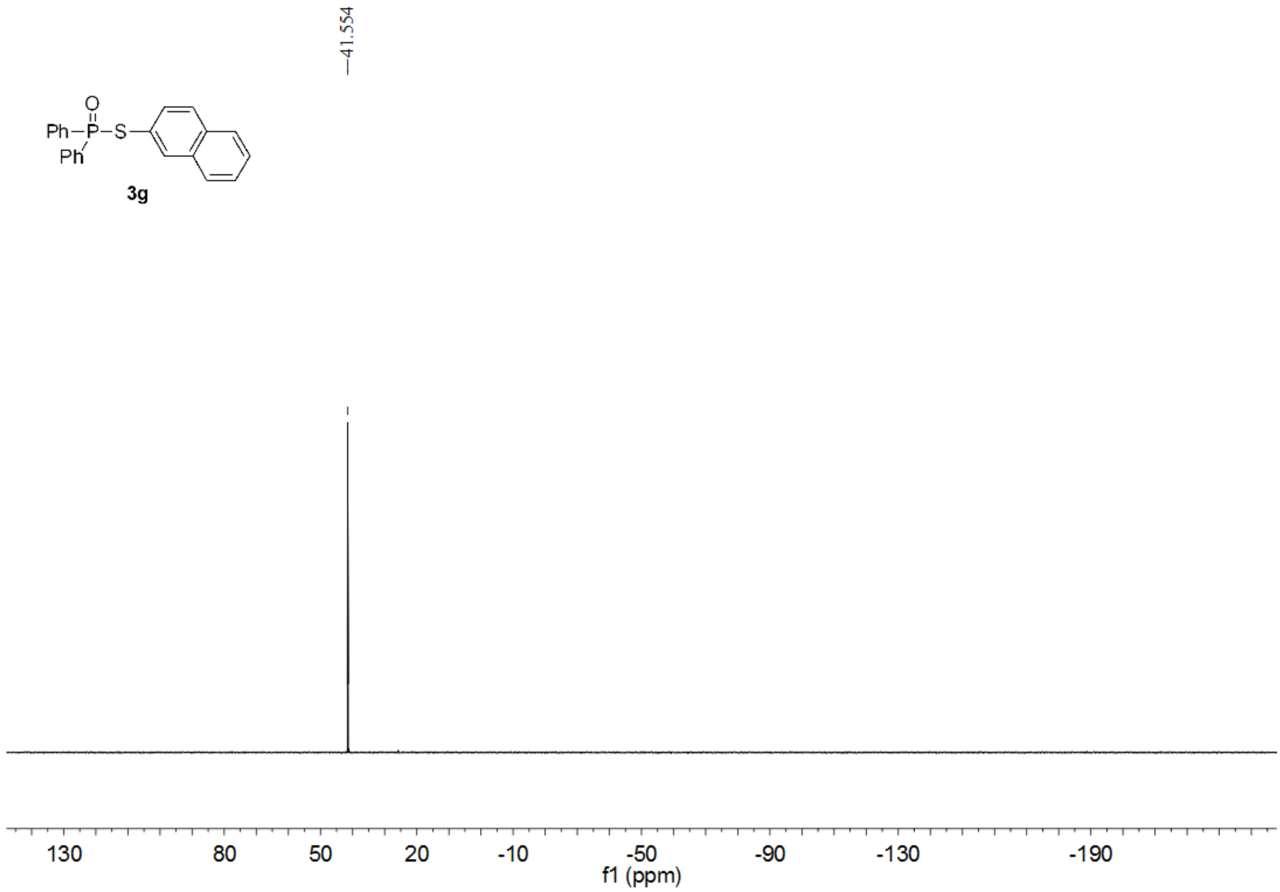

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S
Ph
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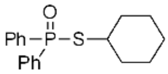
f1 (ppm)



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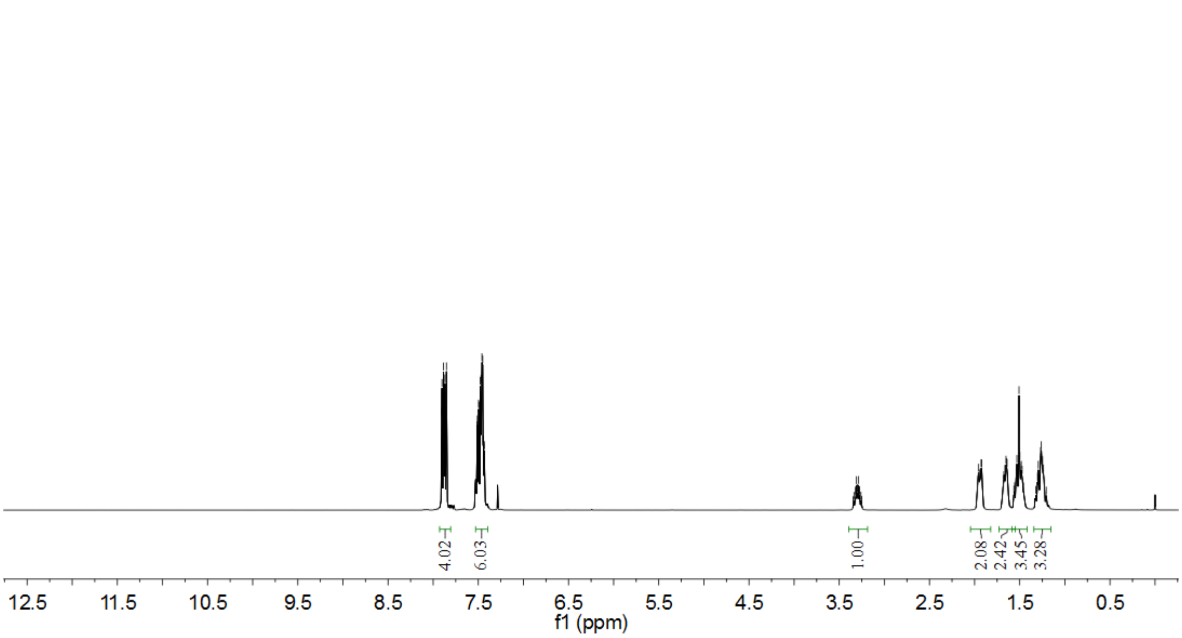
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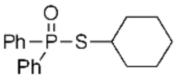
4.02
6.03
1.00
2.08
2.42
3.45
3.28
12.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)



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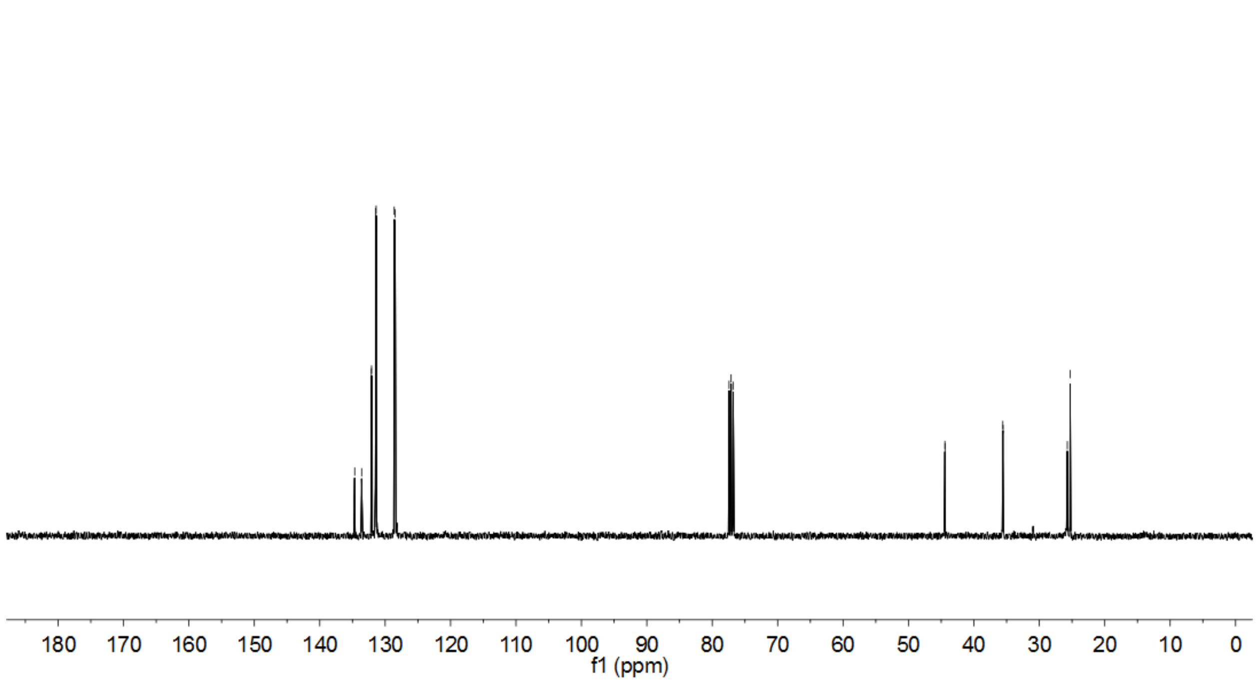
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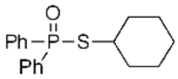

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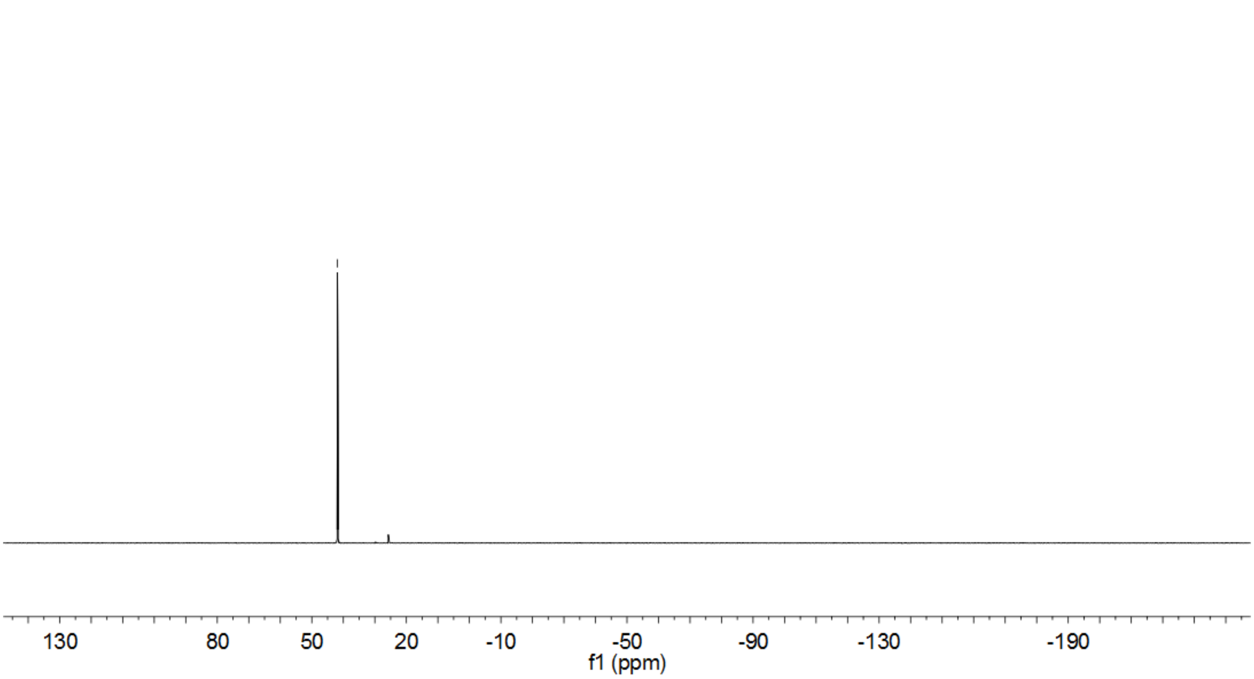

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80
50
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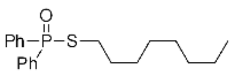
-190
f1 (ppm)



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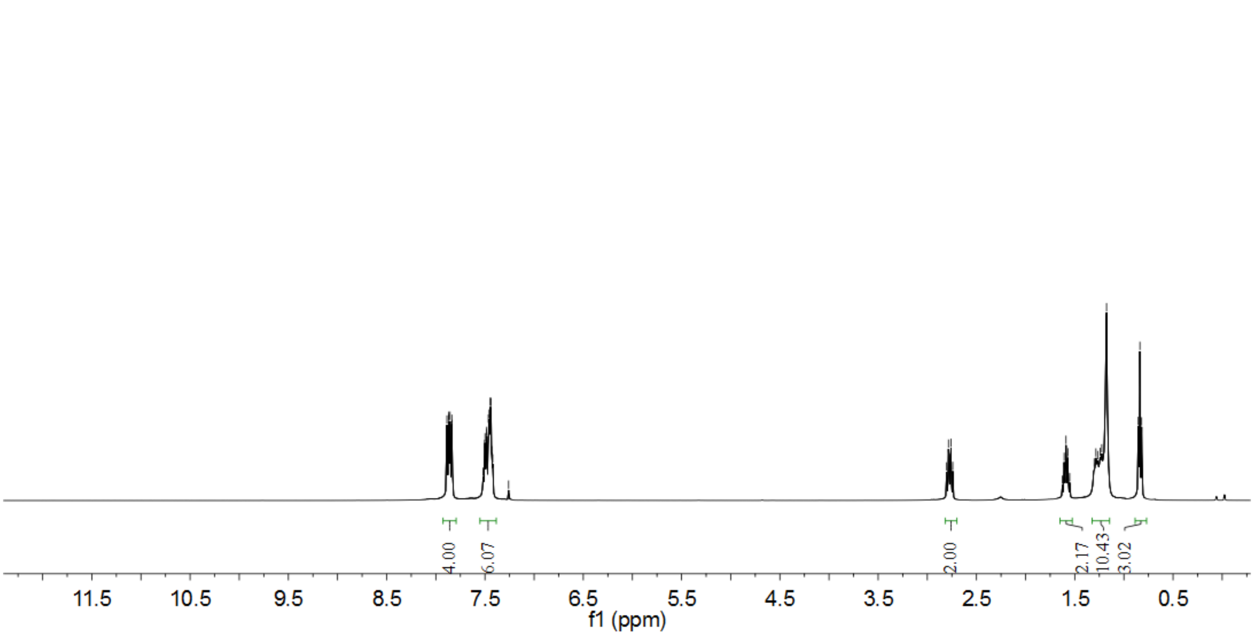

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7.870
7.866
7.855
7.837
7.834
7.519
7.512
7.504
7.500
7.485
7.482
7.466
7.457
7.447
7.443
7.439
7.431
7.426
7.421
7.260
2.803
2.785
2.778
2.766
2.759
2.741
1.608
1.589
1.570
1.285
1.267
1.243
1.225
1.176
0.854
0.837
4.00
6.07
2.00
2.17
10.43
3.02
11.5
10.5
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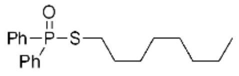
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)



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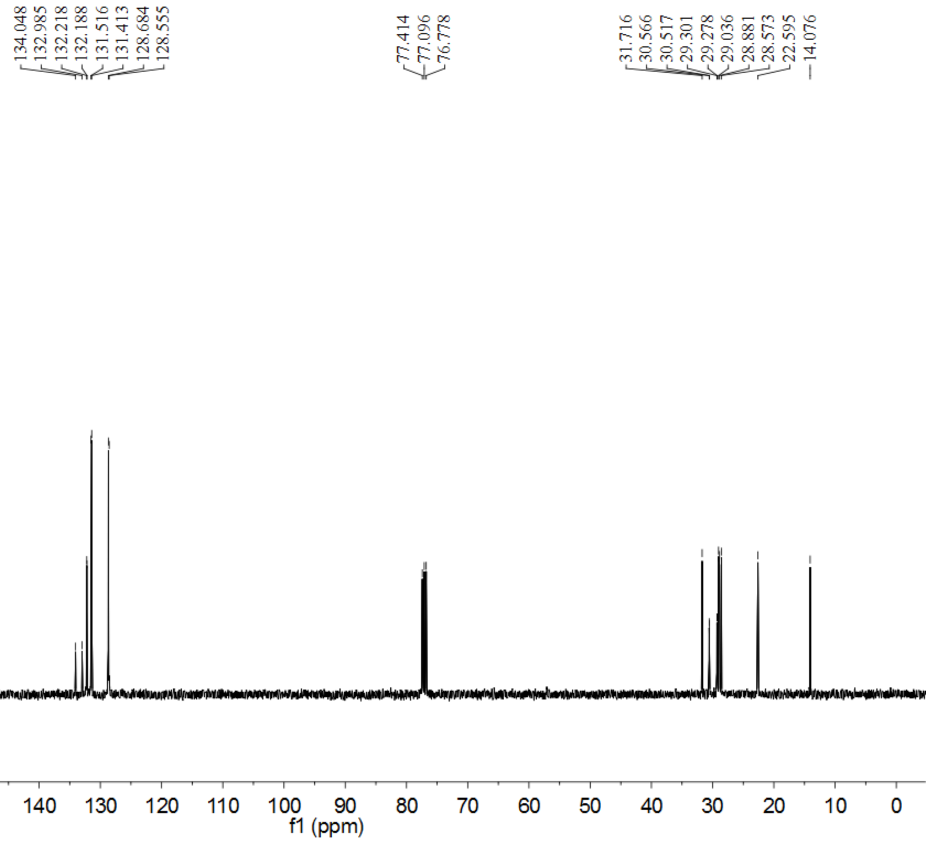
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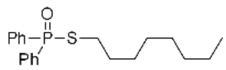
132.985
132.218
132.188
131.516
131.413
128.684
128.555
77.414
77.096
76.778
31.716
30.566
30.517
29.301
29.278
29.036
28.881
28.573
22.595
14.076
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



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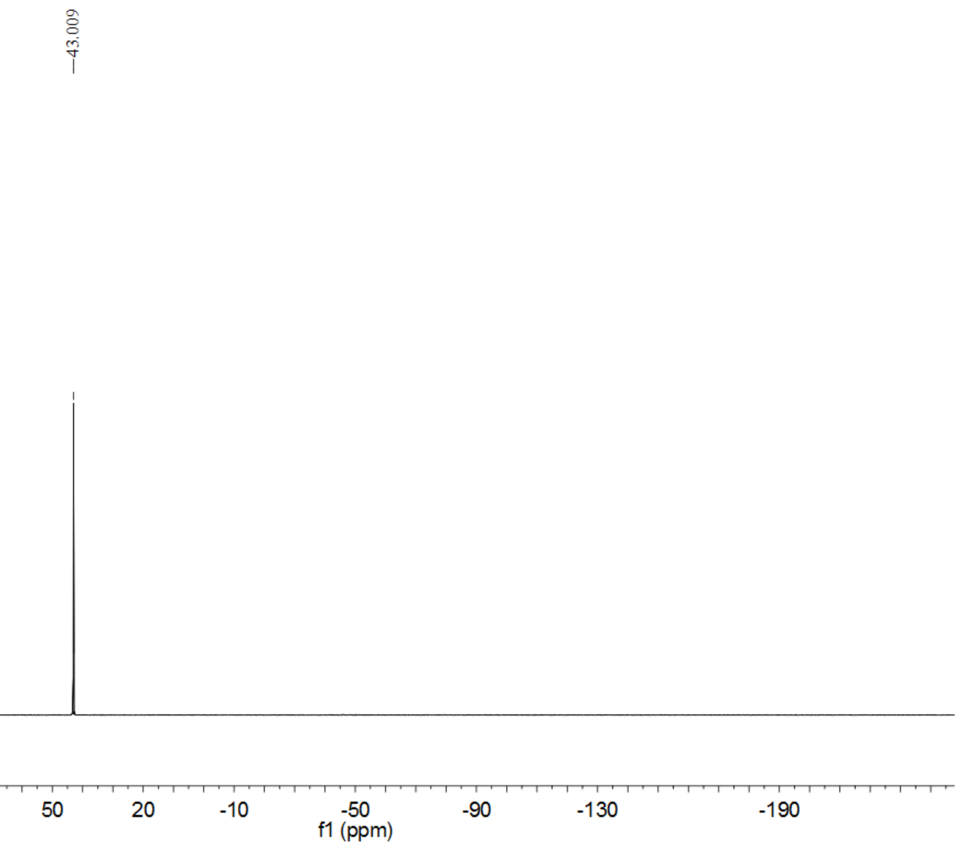
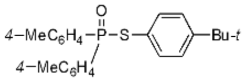
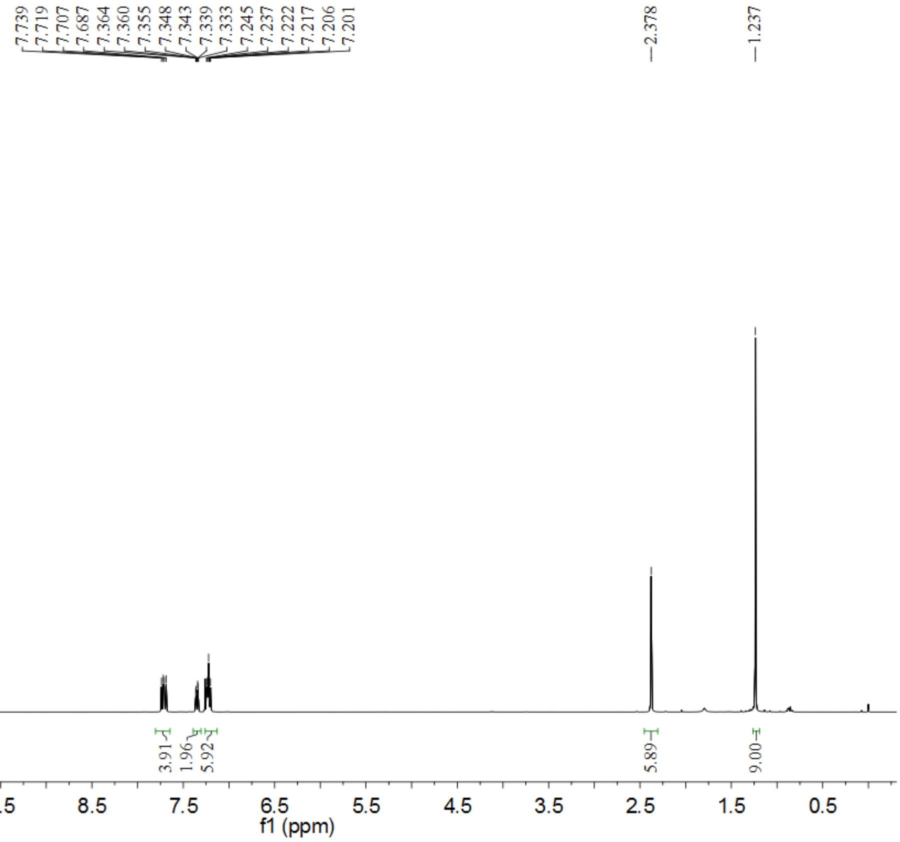
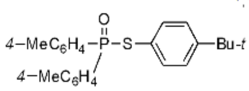

43.009
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)

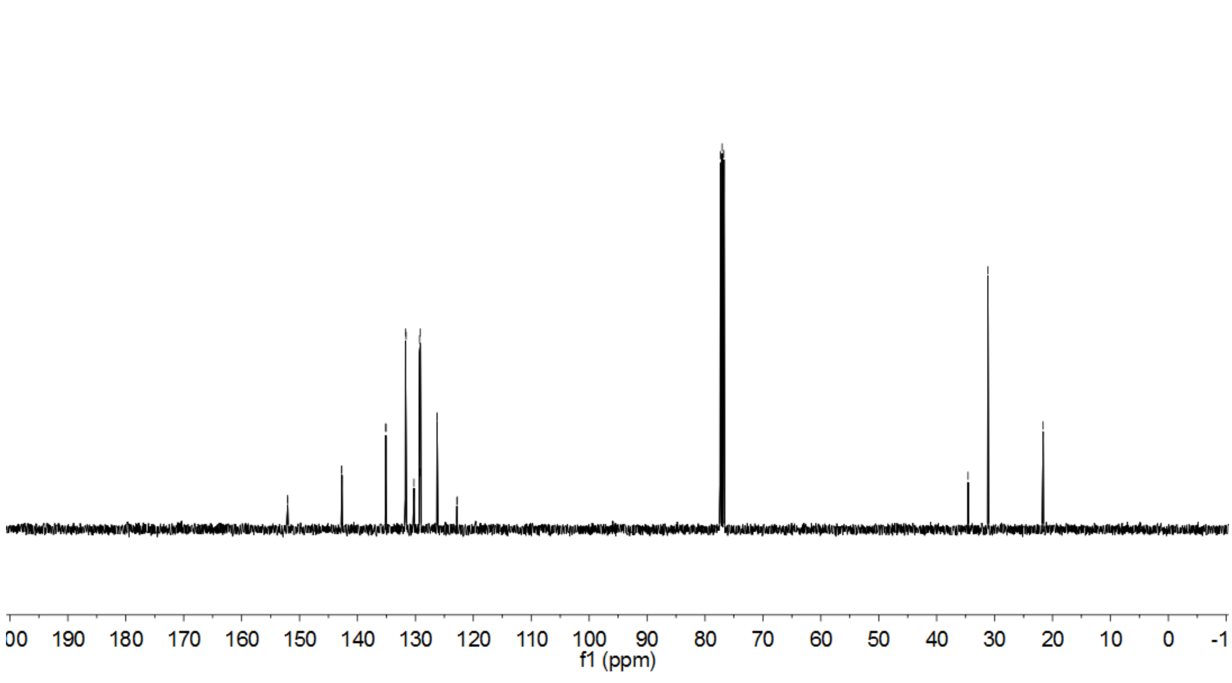

4−MeC6H4
4−MeC6H4
O
P
S
Bu-t

3j


7.739
7.719
7.707
7.687
7.364
7.360
7.355
7.348
7.343
7.339
7.333
7.245
7.237
7.222
7.217
7.206
7.201
2.378
1.237
3.91
1.96
5.92
5.89
9.00
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


4−MeC6H4
4−MeC6H4
O
P
S
Bu-t

3j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152.028
142.733
142.703
135.122
135.085
131.716
131.610
130.253
129.271
129.166
129.136
126.261
126.244
122.838
122.786
77.360
77.042
76.724
34.586
31.154
21.619
190
180
170
160
150
140
130
120
110
100
90
80
70
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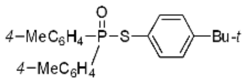
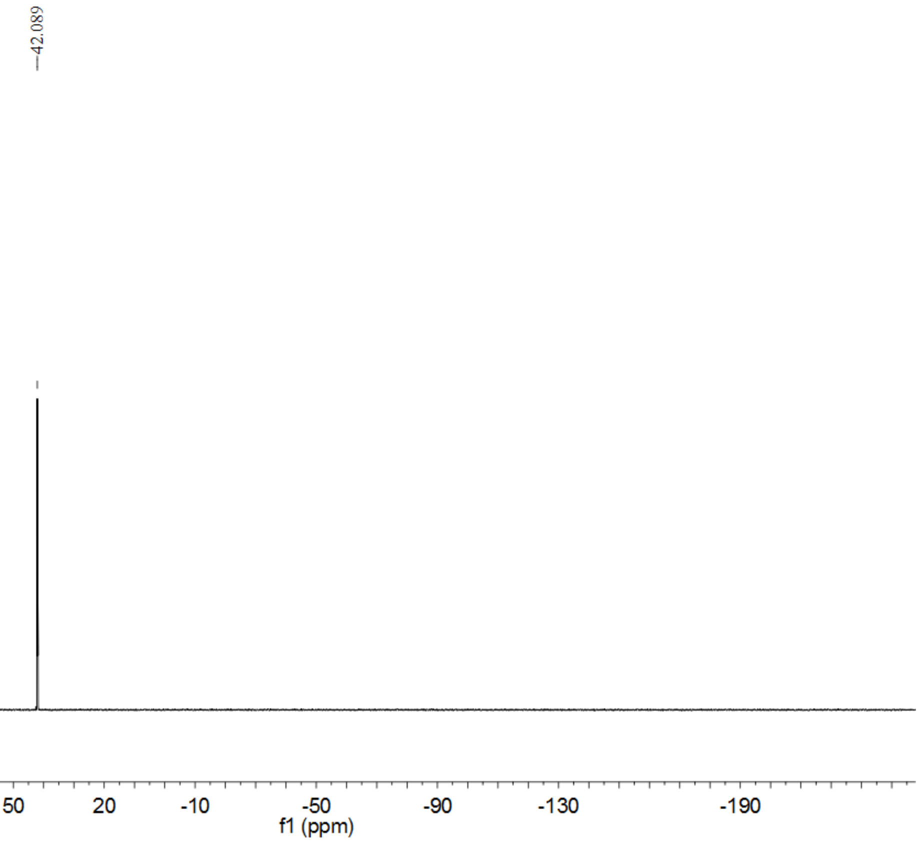
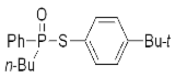
50
40
30
20
10
0
f1 (ppm)

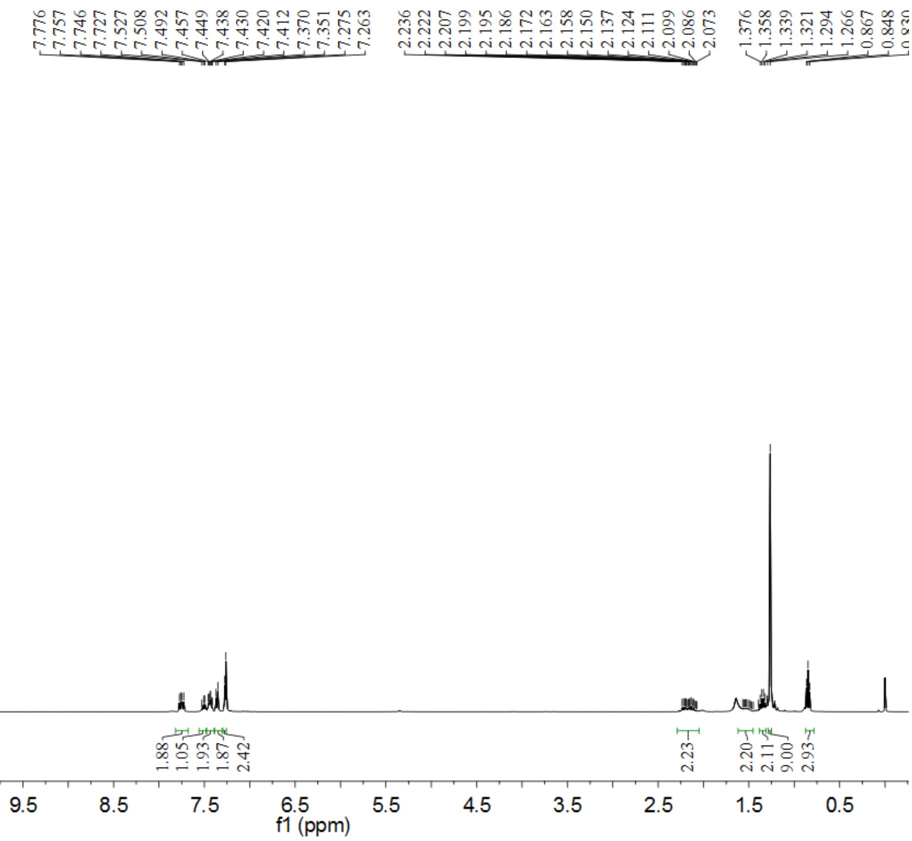

4−MeC6H4
4−MeC6H4
O
P
S
Bu-t

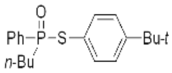
3j


42.089
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)

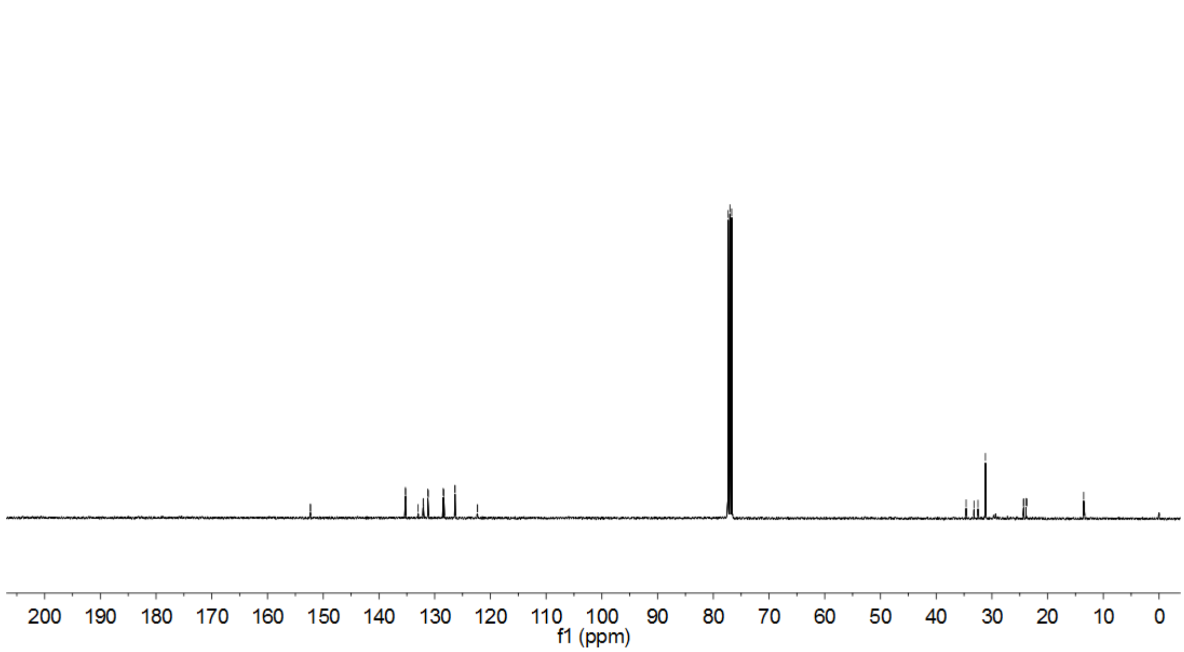

Ph
n-Bu
O
P
S
Bu-t

3k

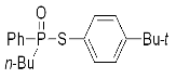

7.776
7.757
7.746
7.727
7.527
7.508
7.492
7.457
7.449
7.438
7.430
7.420
7.412
7.370
7.351
7.275
7.263
2.236
2.222
2.207
2.199
2.195
2.186
2.172
2.163
2.158
2.150
2.137
2.124
2.111
2.099
2.086
2.073
1.376
1.358
1.339
1.321
1.294
1.266
0.867
0.848
1.88
1.05
1.93
1.87
2.42
2.23
2.20
2.11
9.00
2.93
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)



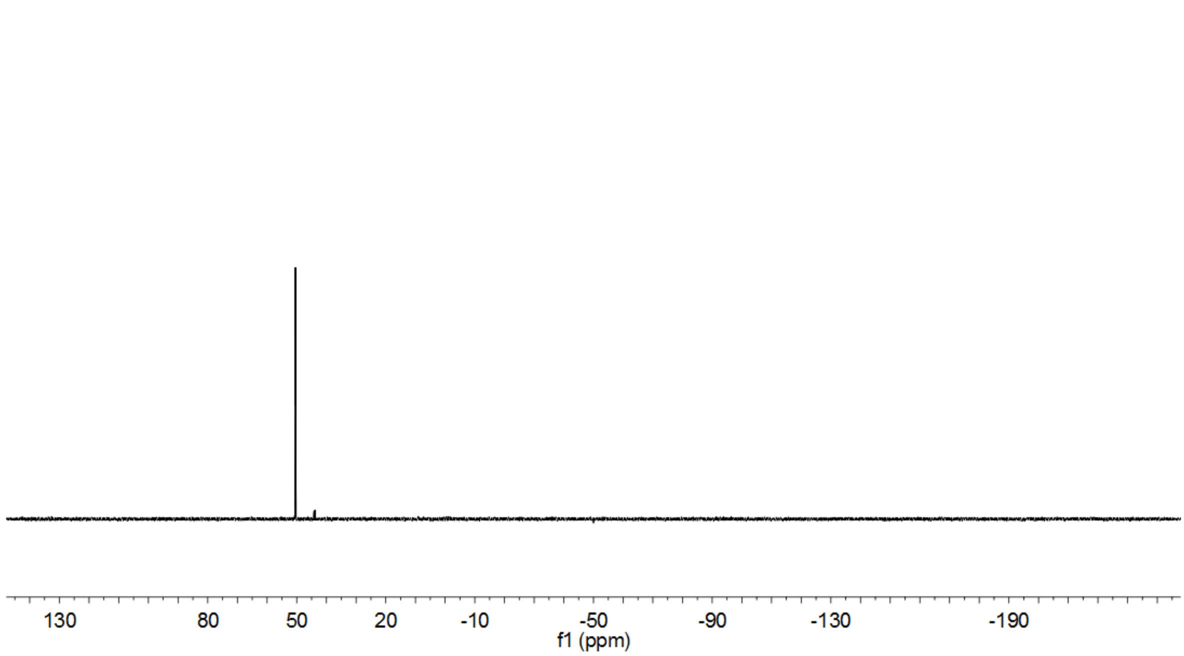
3k



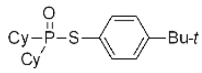
152.308
152.285
135.251
135.216
132.975
132.068
132.039
131.993
131.232
131.135
128.479
128.354
126.364
126.348
122.350
122.299
77.334
77.016
76.699
34.631
33.213
32.503
31.162
24.343
24.297
23.877
23.716
13.530
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



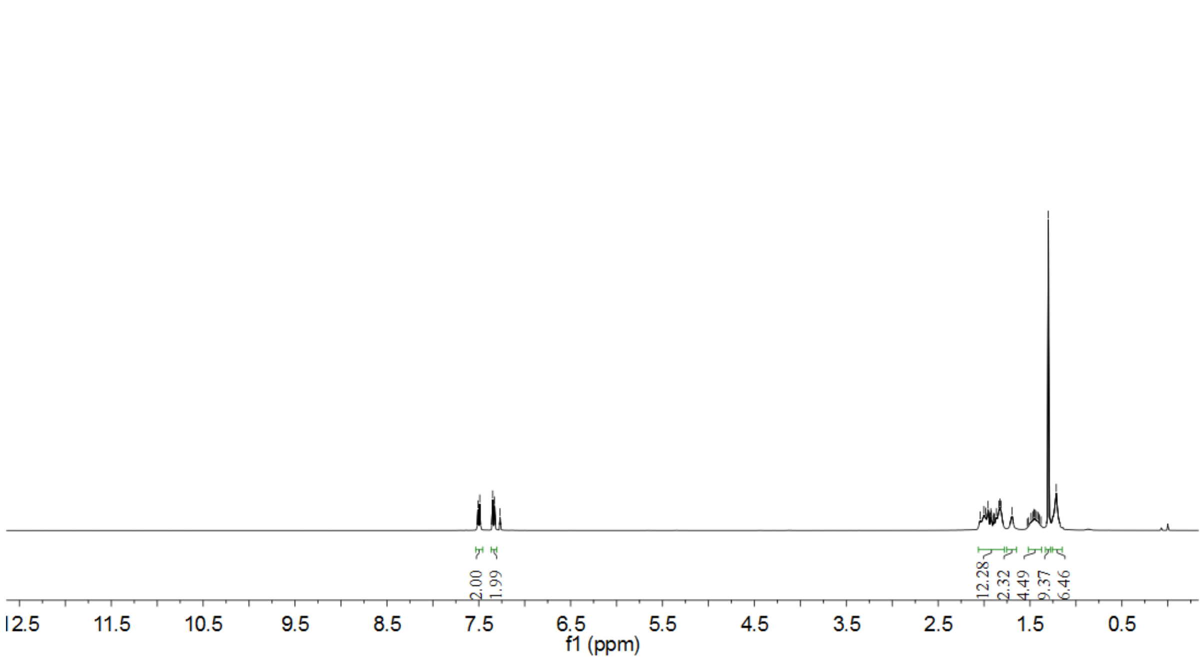
3k



50.390
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)


Cy
O
P
S
Bu-t
Cy

3l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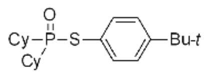
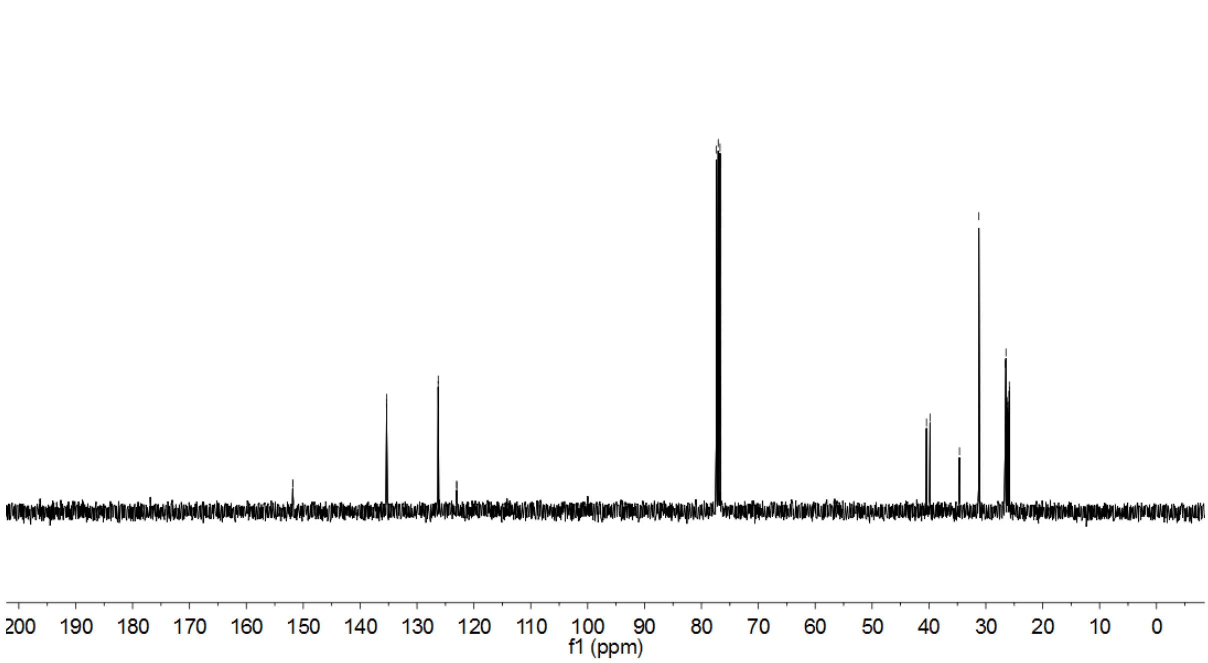
7.489
7.348
7.327
7.268
2.044
2.002
1.983
1.957
1.934
1.927
1.921
1.903
1.896
1.888
1.867
1.838
1.827
1.817
1.696
1.531
1.519
1.489
1.471
1.456
1.451
1.441
1.427
1.411
1.399
1.380
1.301
1.215
2.00
1.99
12.28
2.32
4.49
9.37
6.46
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


Cy
O
P
S
Bu-t
Cy

3l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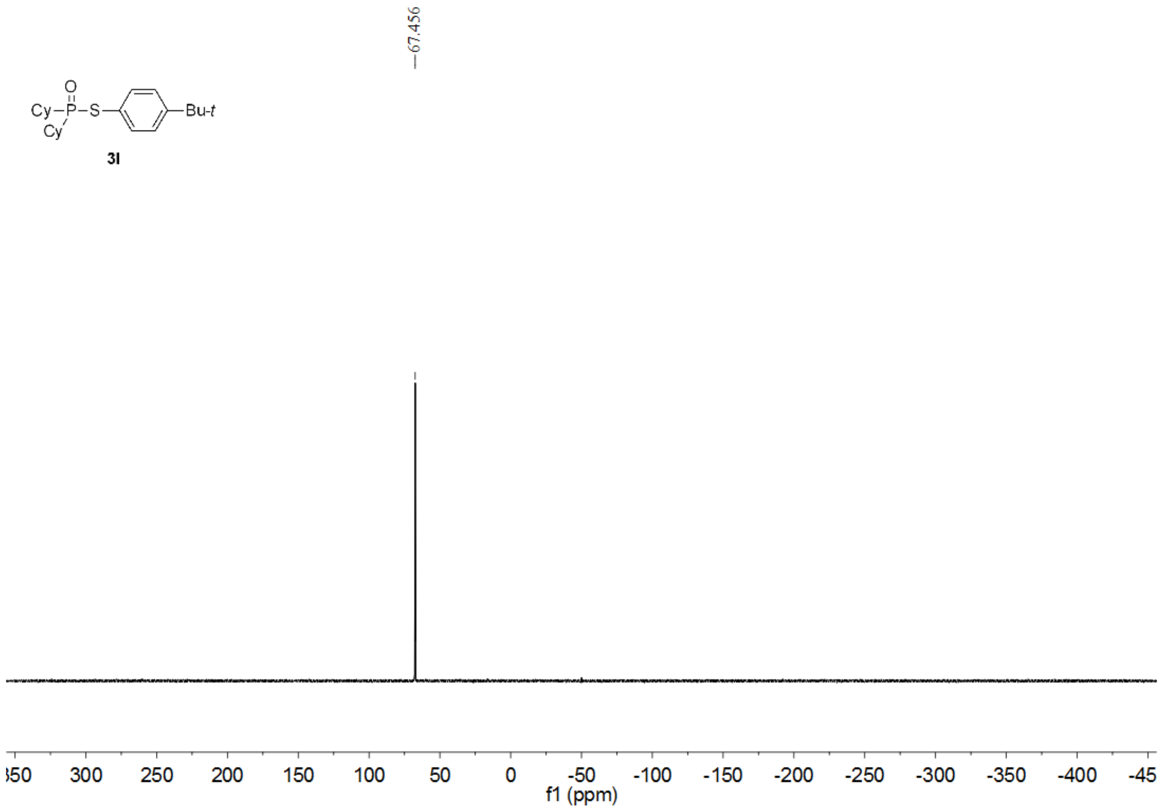
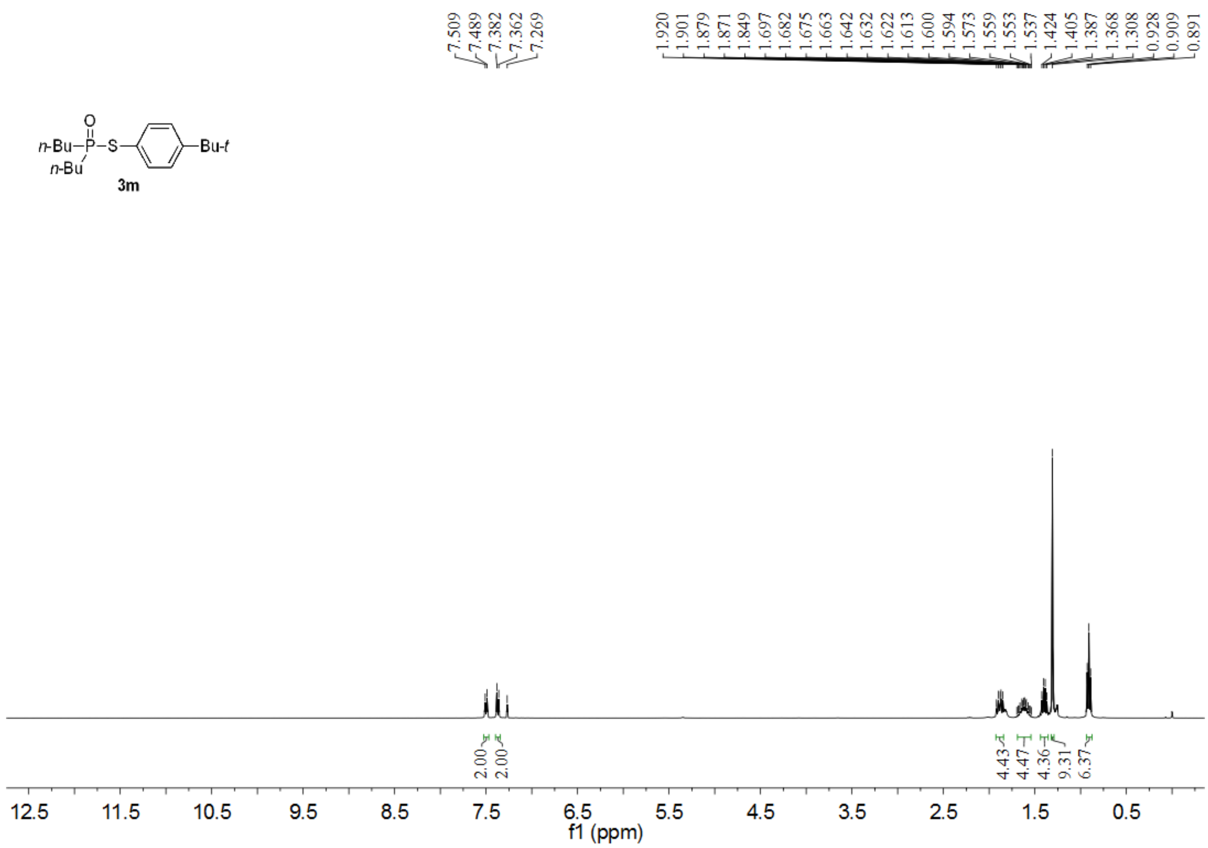
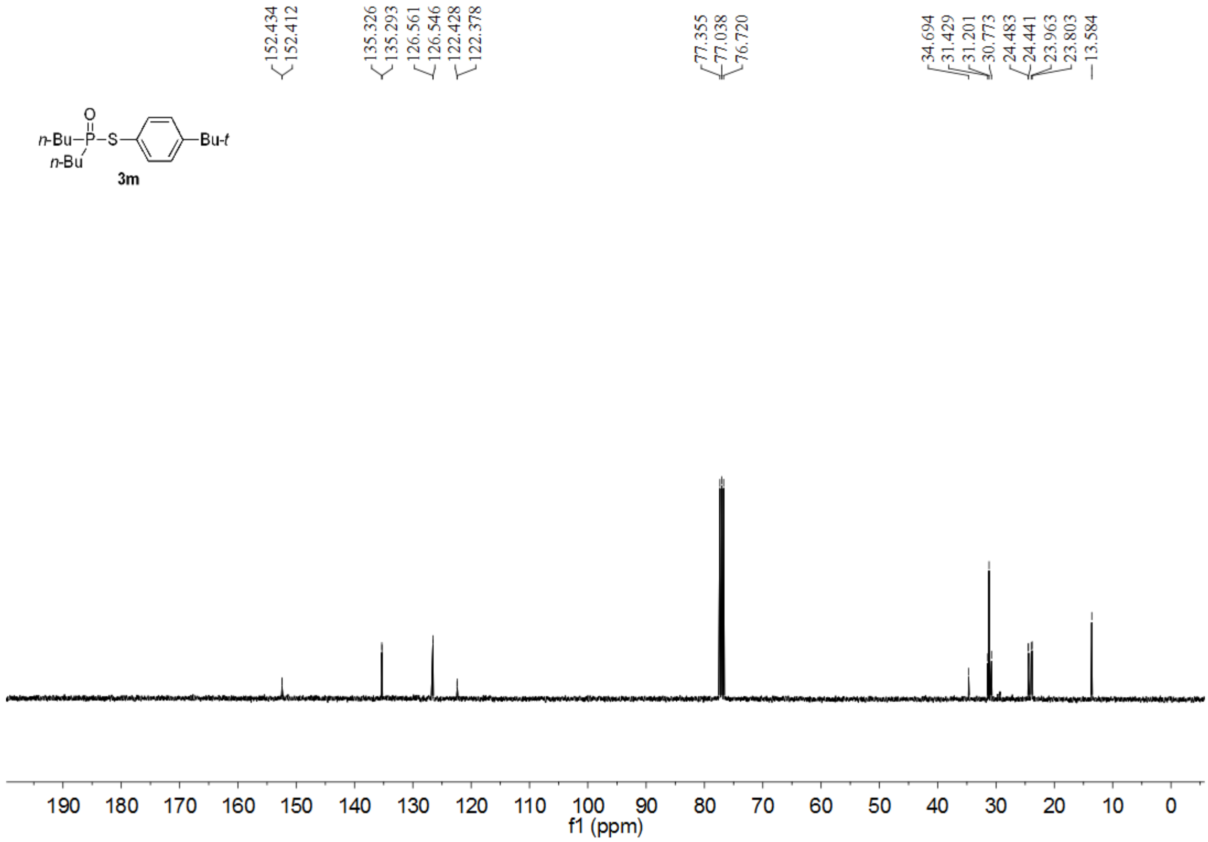
135.366
135.335
126.280
126.270
123.041
122.990
77.360
77.042
76.725
40.429
39.808
34.637
31.229
26.592
26.456
26.260
26.225
26.070
26.036
25.871
25.857
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

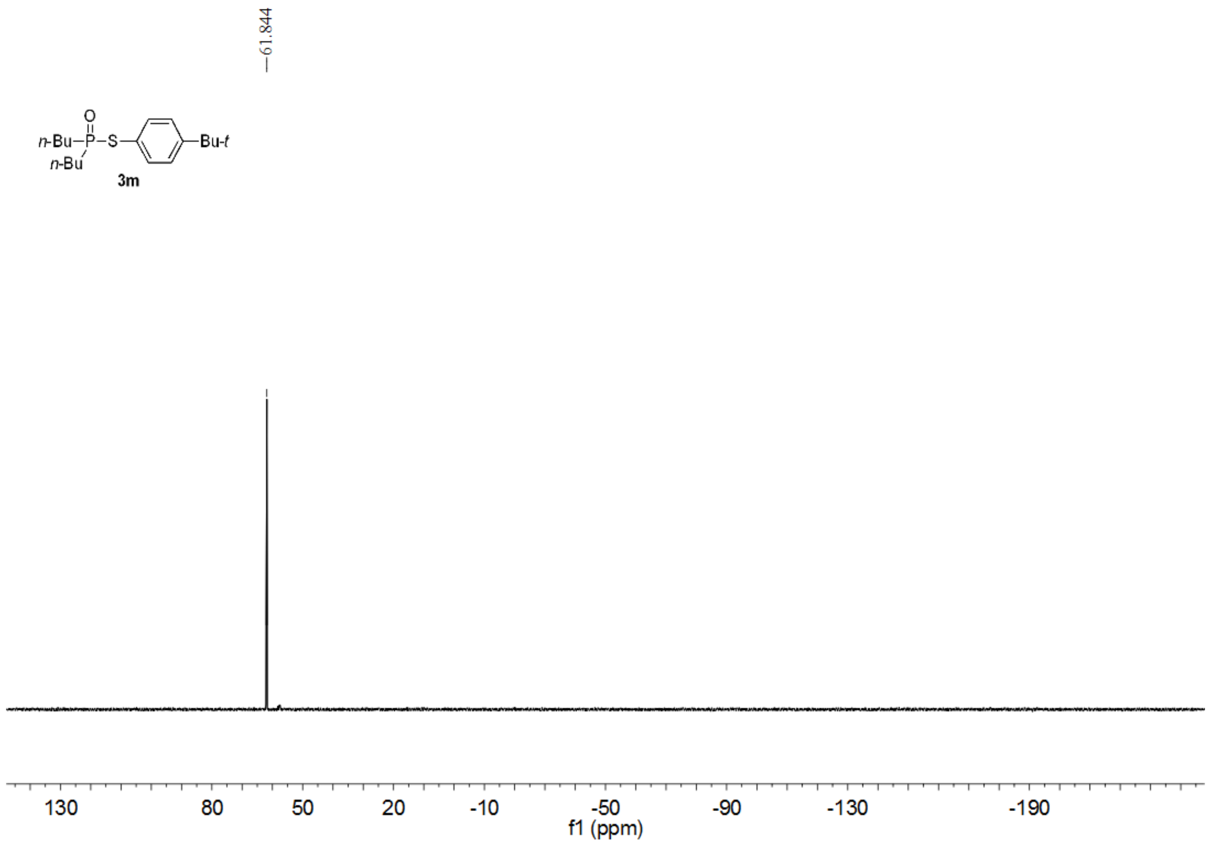


—67.456
Cy
Cy
P
O
S
Bu-t
3l
350 300 250 200 150 100 50 0 -50 -100 -150 -200 -250 -300 -350 -400 -450
f1 (ppm)



7.509
7.489
7.382
7.362
7.269
1.920
1.901
1.879
1.871
1.849
1.697
1.682
1.675
1.663
1.642
1.632
1.622
1.613
1.600
1.594
1.573
1.559
1.553
1.537
1.424
1.405
1.387
1.368
1.308
0.928
0.909
0.891
n-Bu
n-Bu
P
O
S
Bu-t
3m
2.00
2.00
4.43
4.47
4.36
9.31
6.37
12.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)


152.434
152.412
135.326
135.293
126.561
126.546
122.428
122.378
77.355
77.038
76.720
34.694
31.429
31.201
30.773
24.483
24.441
23.963
23.803
13.584
n-Bu
n-Bu
O
P
S
Bu-t
3m
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


61.844
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n-Bu
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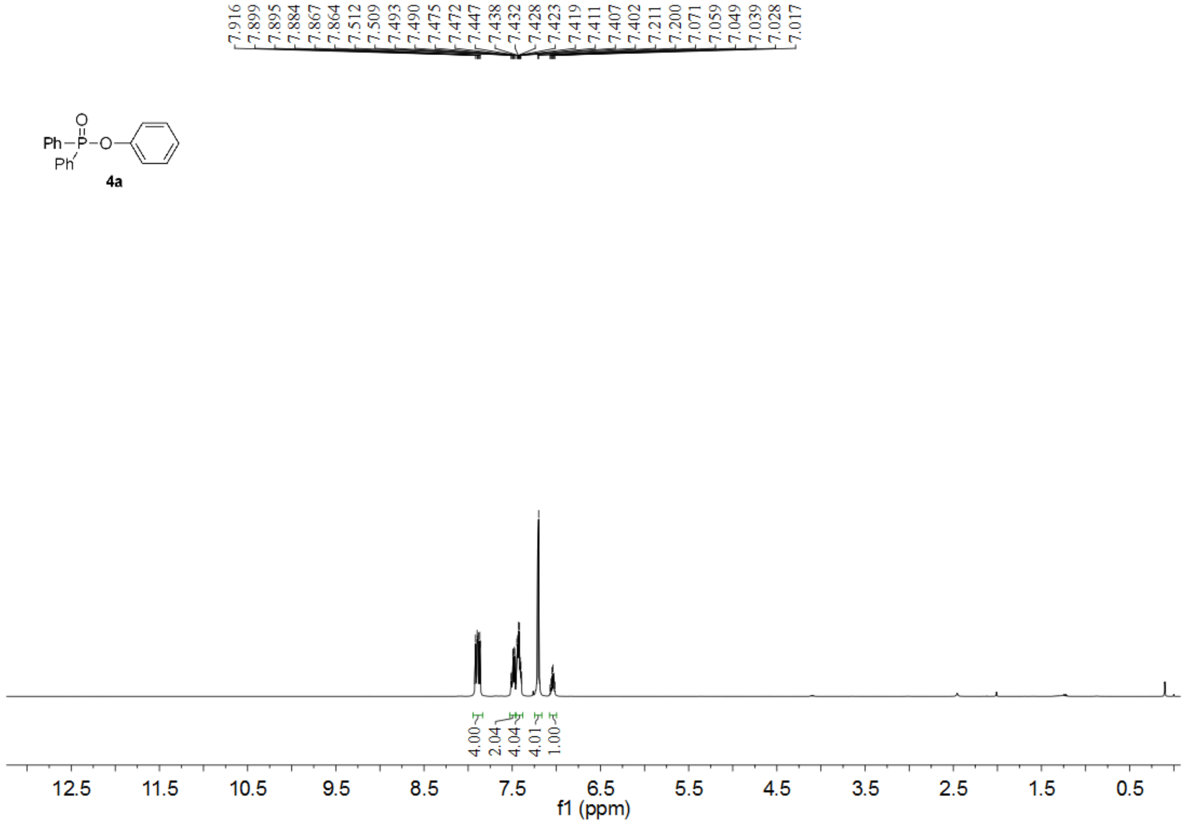
P
S
Bu-t
3m
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)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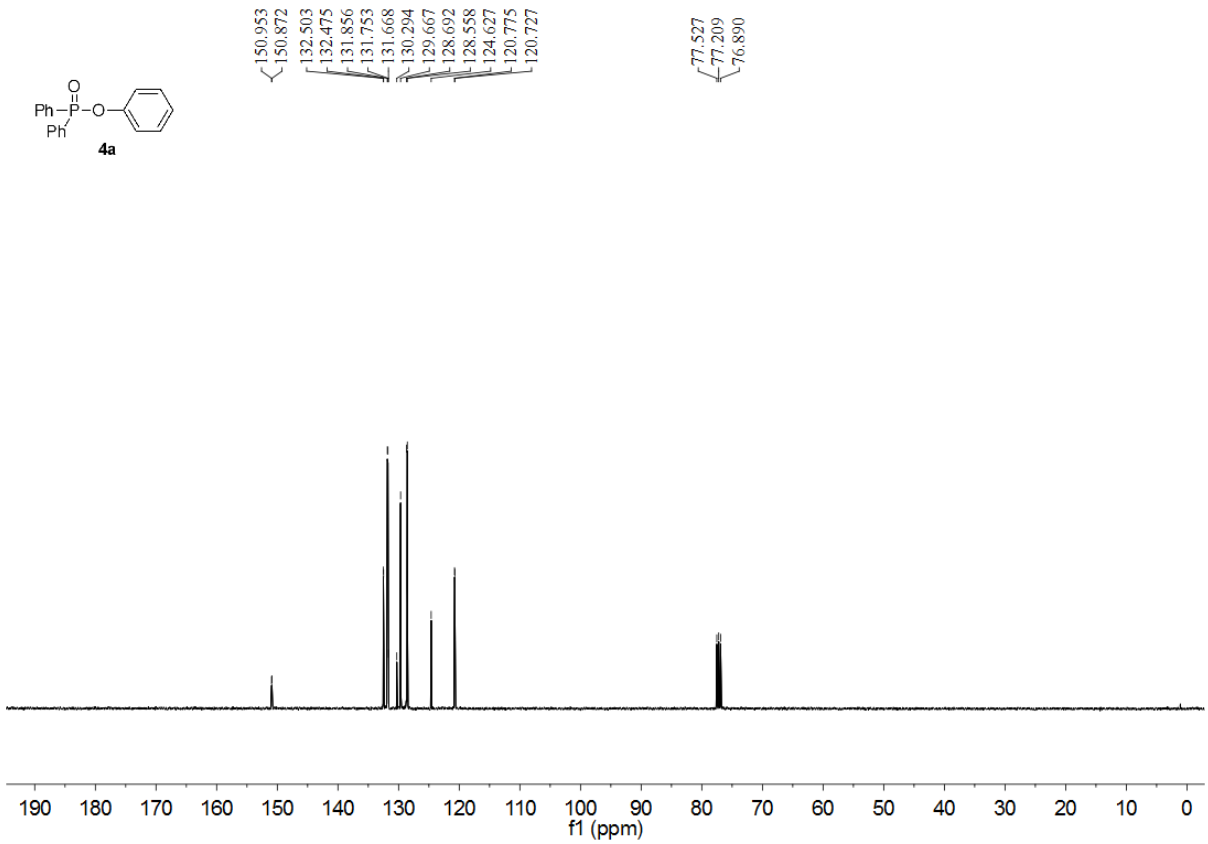
7.884
7.867
7.864
7.512
7.509
7.493
7.490
7.475
7.472
7.447
7.438
7.432
7.428
7.423
7.419
7.411
7.407
7.402
7.211
7.200
7.071
7.059
7.049
7.039
7.028
7.017
Ph
O
P
O
Ph
4a
4.00
2.04
4.04
4.01
1.00
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)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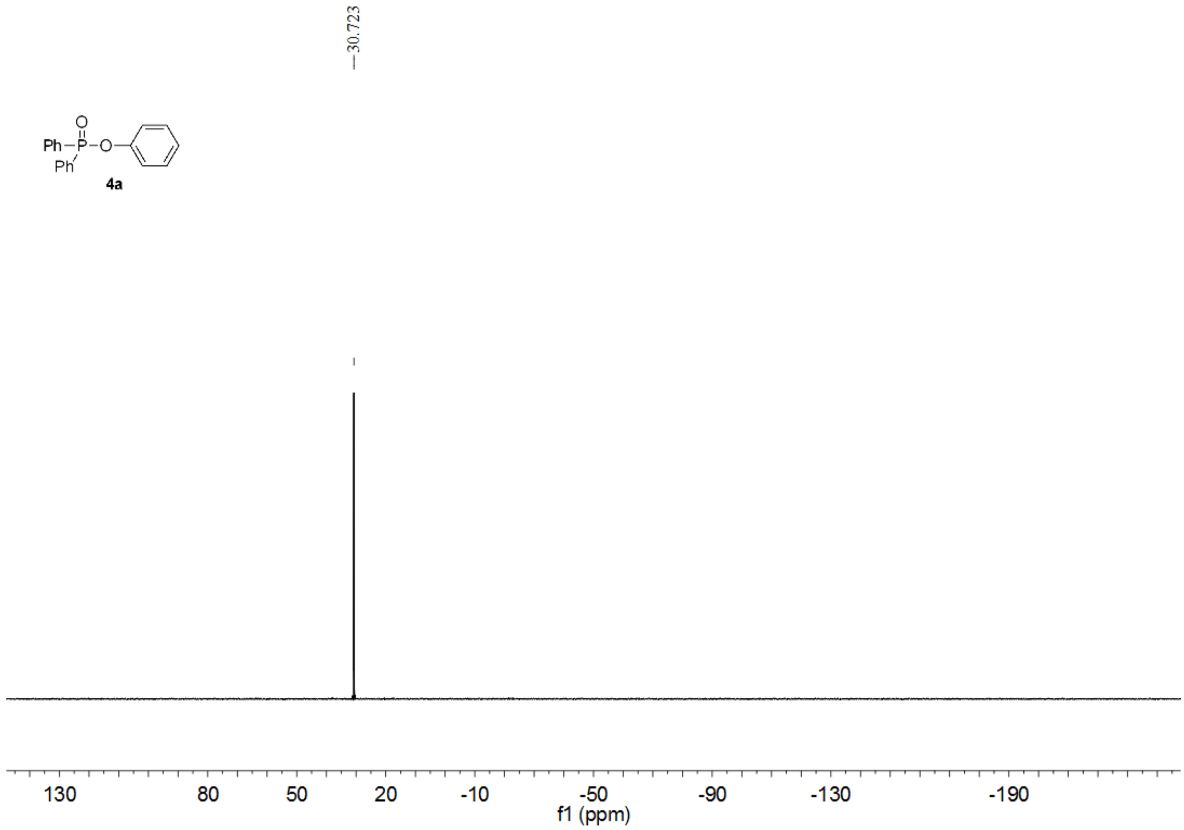
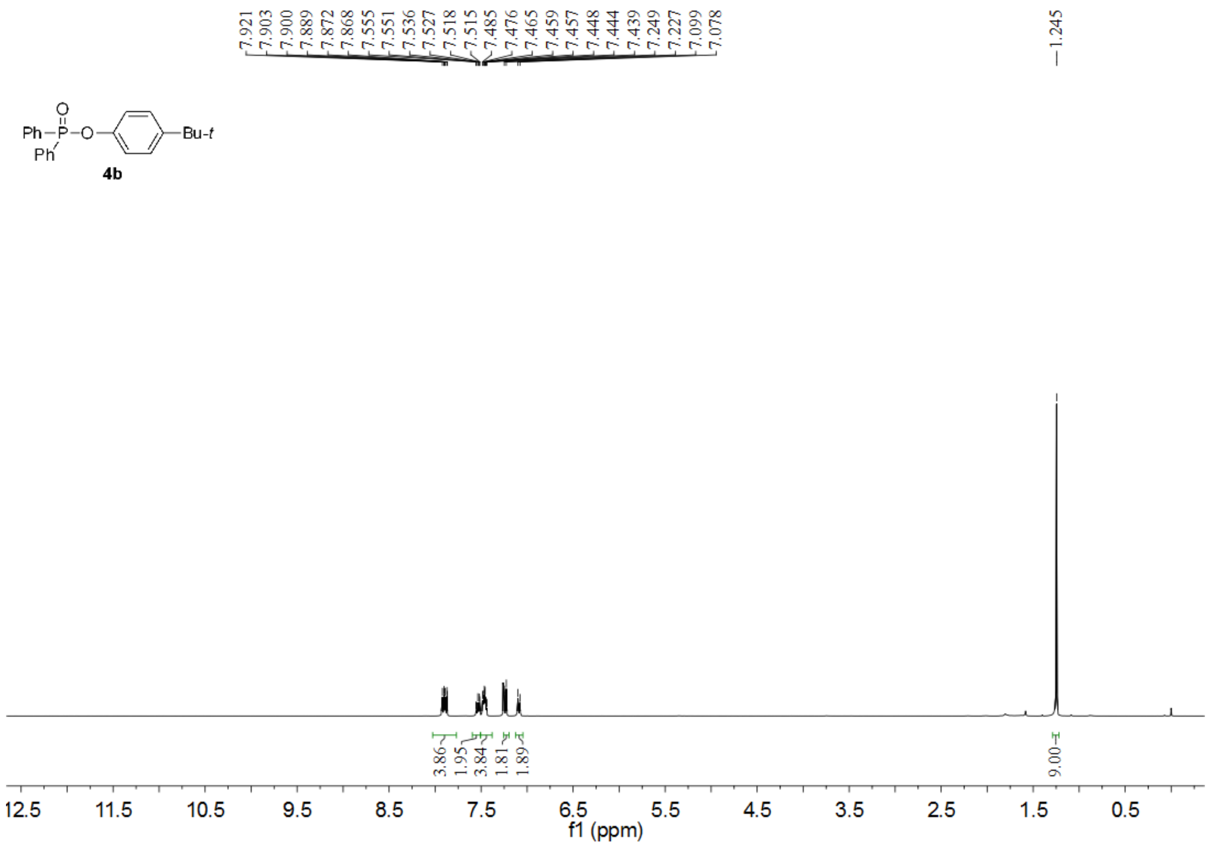
131.753
131.668
130.294
129.667
128.692
128.558
124.627
120.775
120.727
77.527
77.209
76.890
Ph
O
P
O
Ph
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


30.723
Ph
P
O
Ph
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)

4a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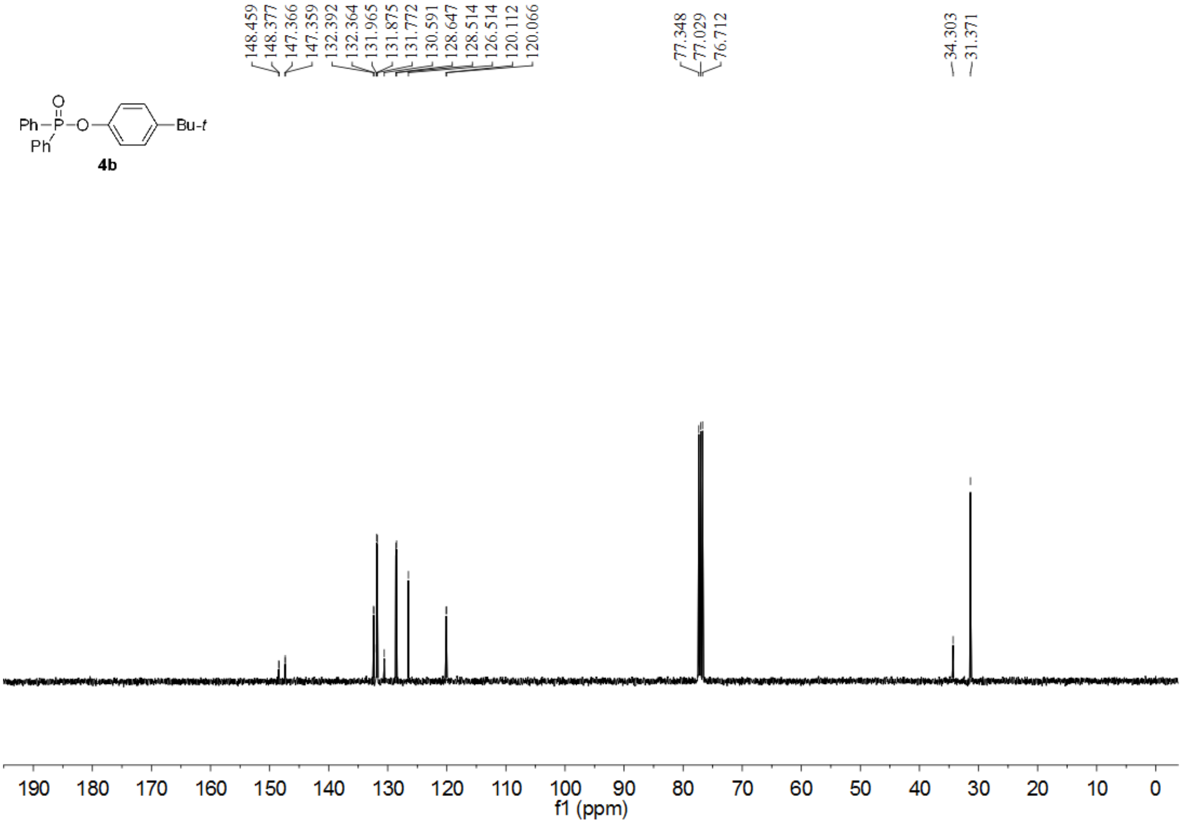
7.900
7.889
7.872
7.868
7.555
7.551
7.536
7.527
7.518
7.515
7.485
7.476
7.465
7.459
7.457
7.448
7.444
7.439
7.249
7.227
7.099
7.078
1.245
Ph
P
O
Ph
Bu-*t*
3.86
1.95
3.84
1.81
1.89
9.00
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

4b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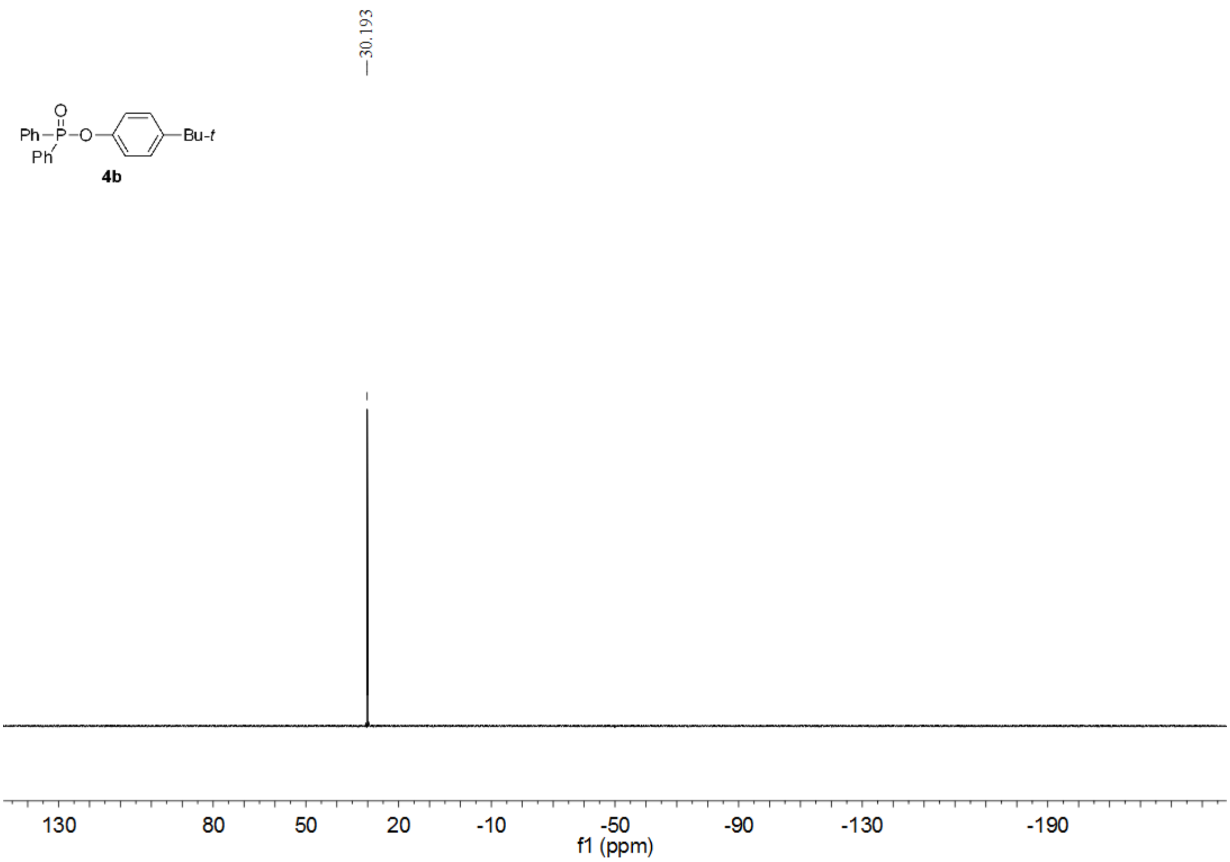
147.366
147.359
132.392
132.364
131.965
131.875
131.772
130.591
128.647
128.514
126.514
120.112
120.066
77.348
77.029
76.712
34.303
31.371
Ph
O
P
O
Bu-t
Ph
4b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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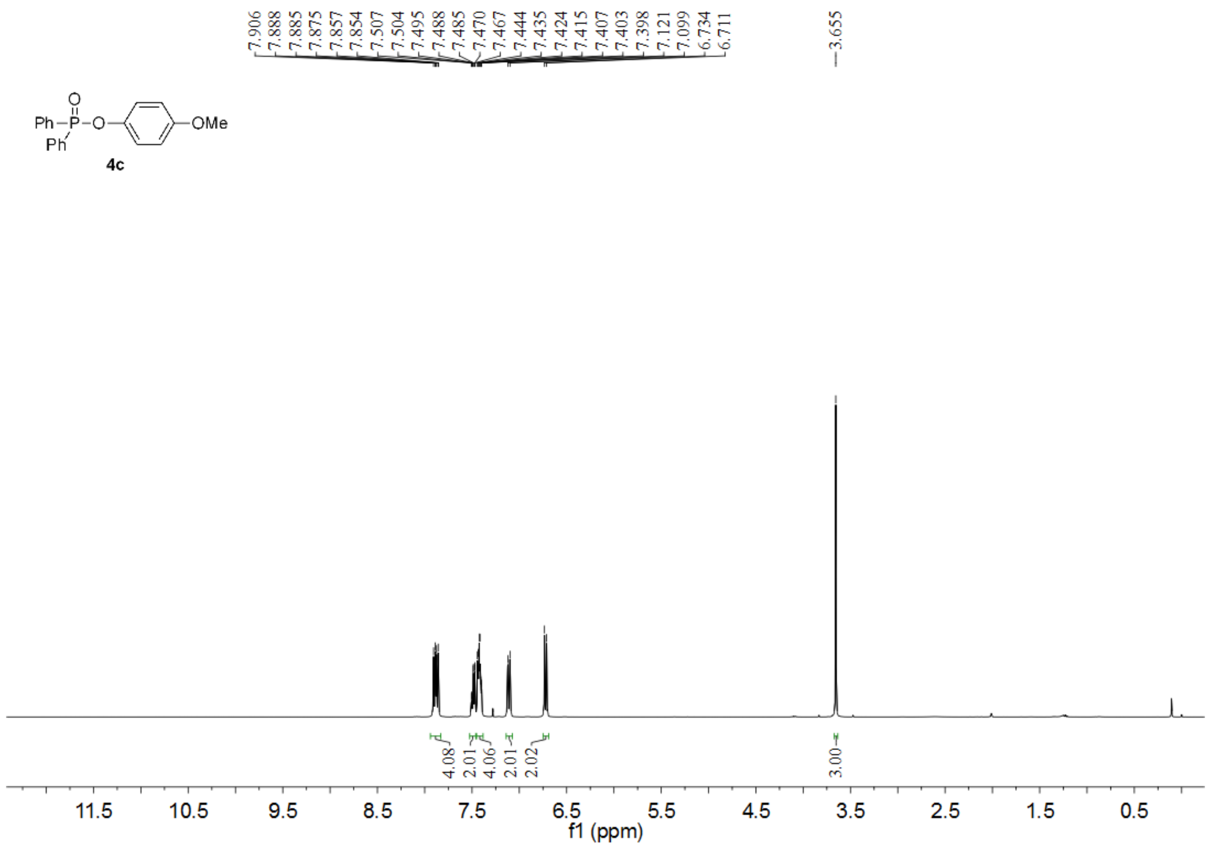
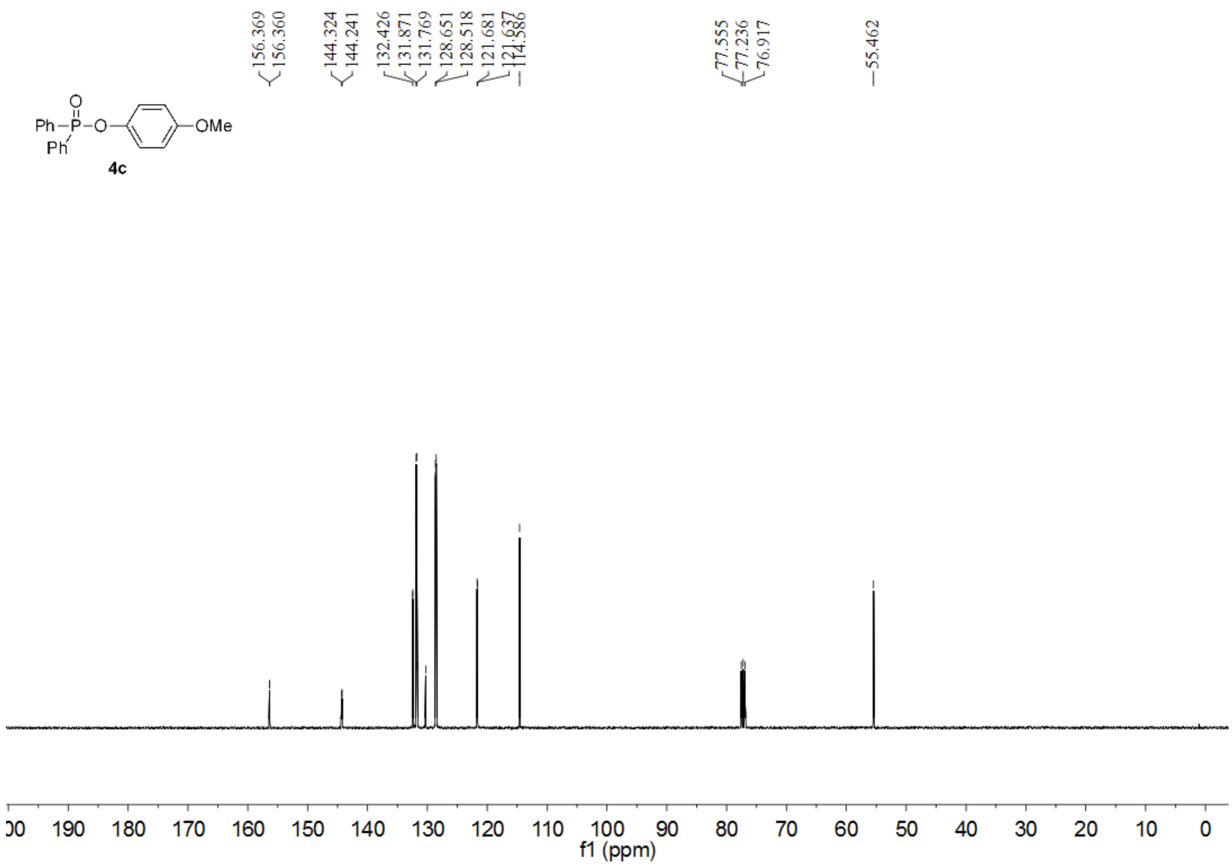
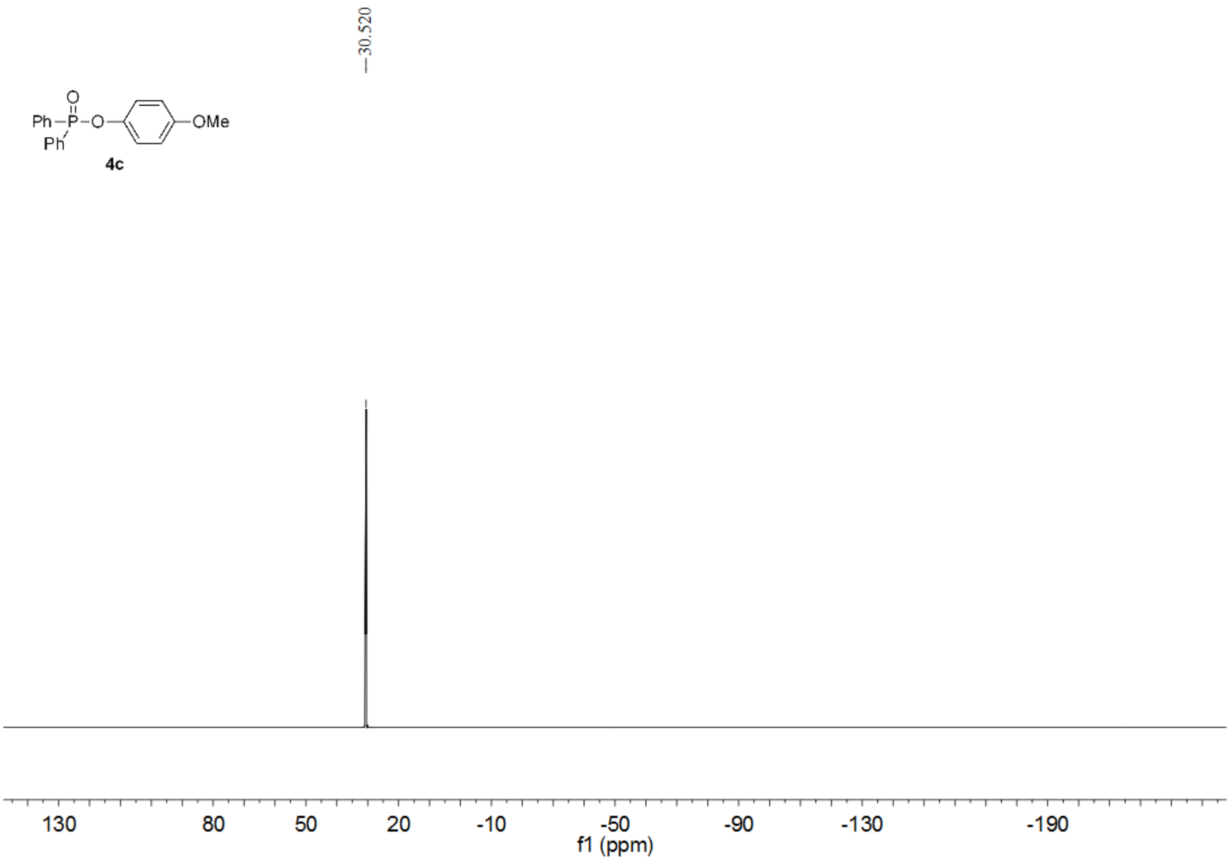
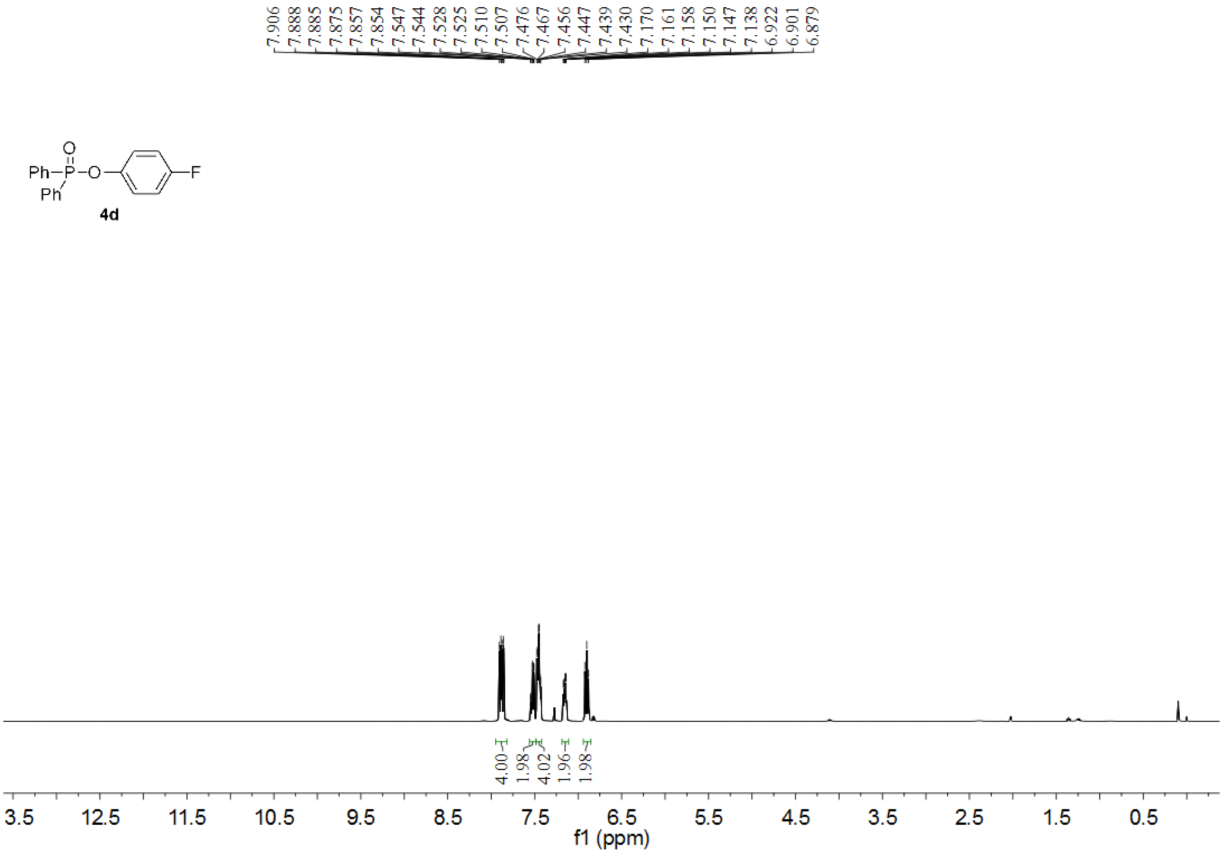
O
P
O
Bu-t
Ph
4b
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)


7.906
7.888
7.885
7.875
7.857
7.854
7.507
7.504
7.495
7.488
7.485
7.470
7.467
7.444
7.435
7.424
7.415
7.407
7.403
7.398
7.121
7.099
6.734
6.711
3.655
Ph
P
O
Ph
OMe
4c
4.08
2.01
4.06
2.01
2.02
3.00
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

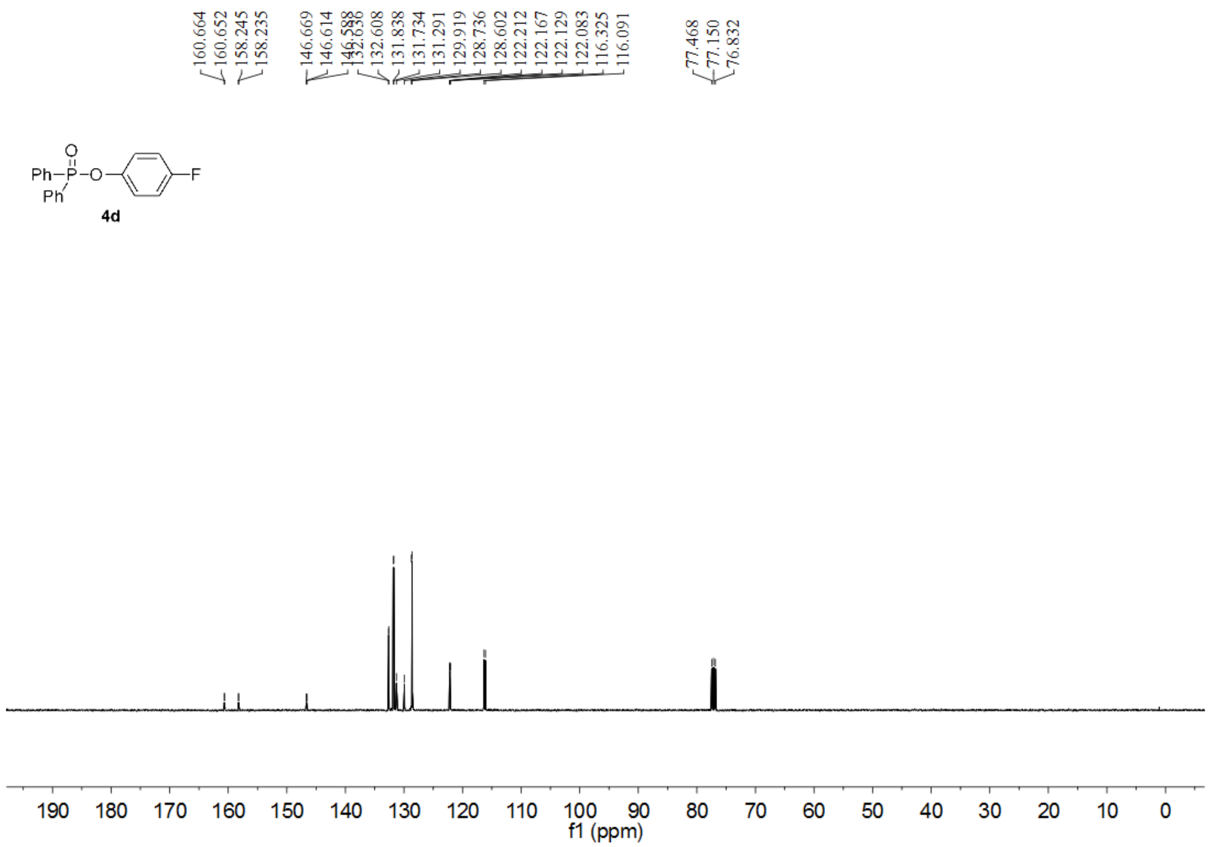

156.369
156.360
144.324
144.241
132.426
131.871
131.769
128.651
128.518
121.681
121.637
114.586
77.555
77.236
76.917
55.462
Ph
P
O
Ph
OMe
4c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

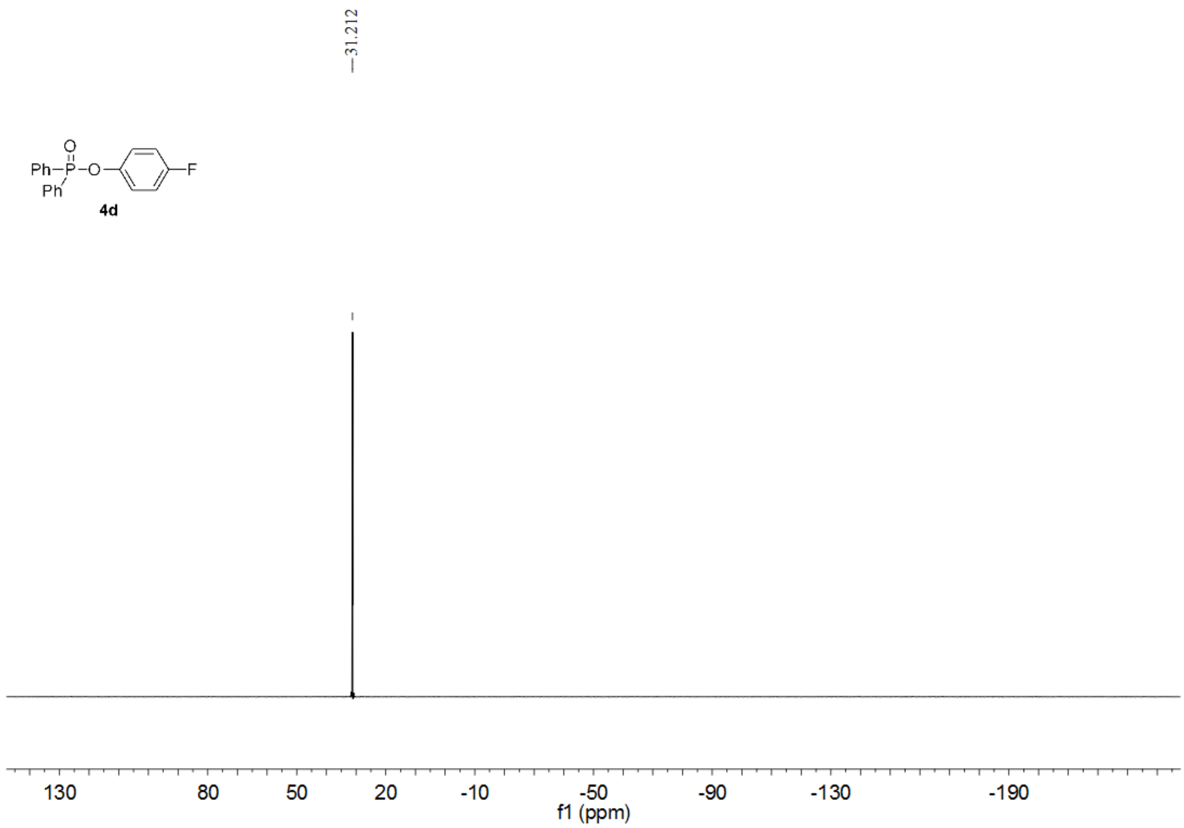


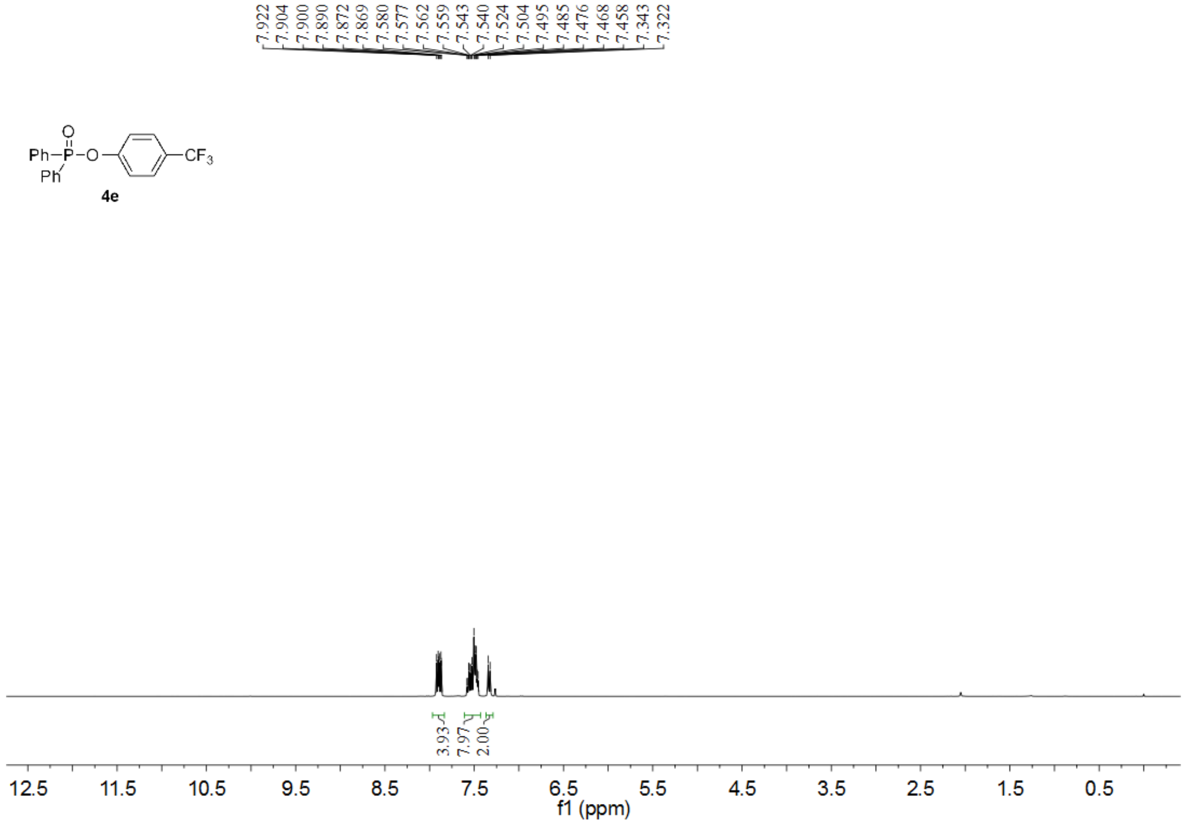
30.520
Ph
O
P
O
OMe
Ph
4c
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)



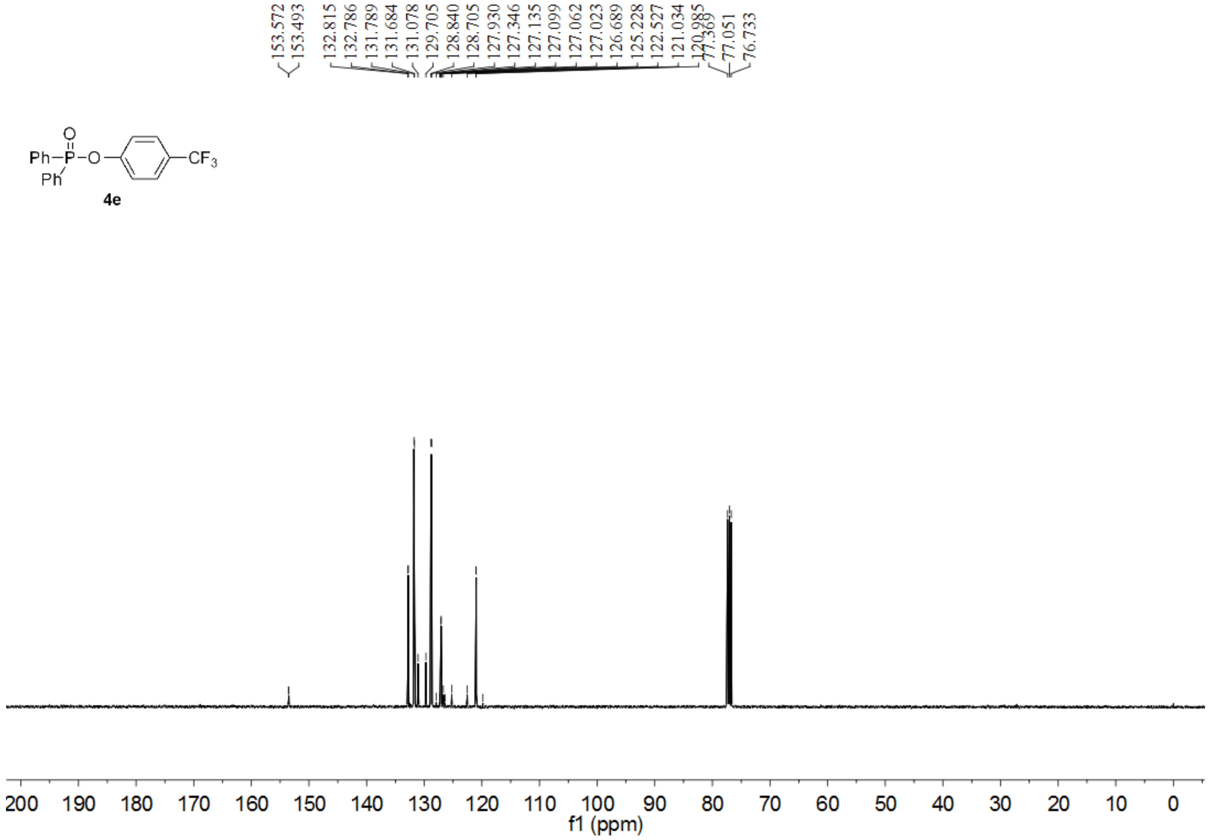
7.906
7.888
7.885
7.875
7.857
7.854
7.547
7.544
7.528
7.525
7.510
7.507
7.476
7.467
7.456
7.447
7.439
7.430
7.170
7.161
7.158
7.150
7.147
7.138
6.922
6.901
6.879
Ph
O
P
O
F
Ph
4d
4.00
1.98
4.02
1.96
1.98
3.5
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


160.664
160.652
158.245
158.235
146.669
146.614
146.588
132.636
132.608
131.838
131.734
131.291
129.919
128.736
128.602
122.212
122.167
122.129
122.083
116.325
116.091
77.468
77.150
76.832
Ph
O
P
O
F
Ph
4d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

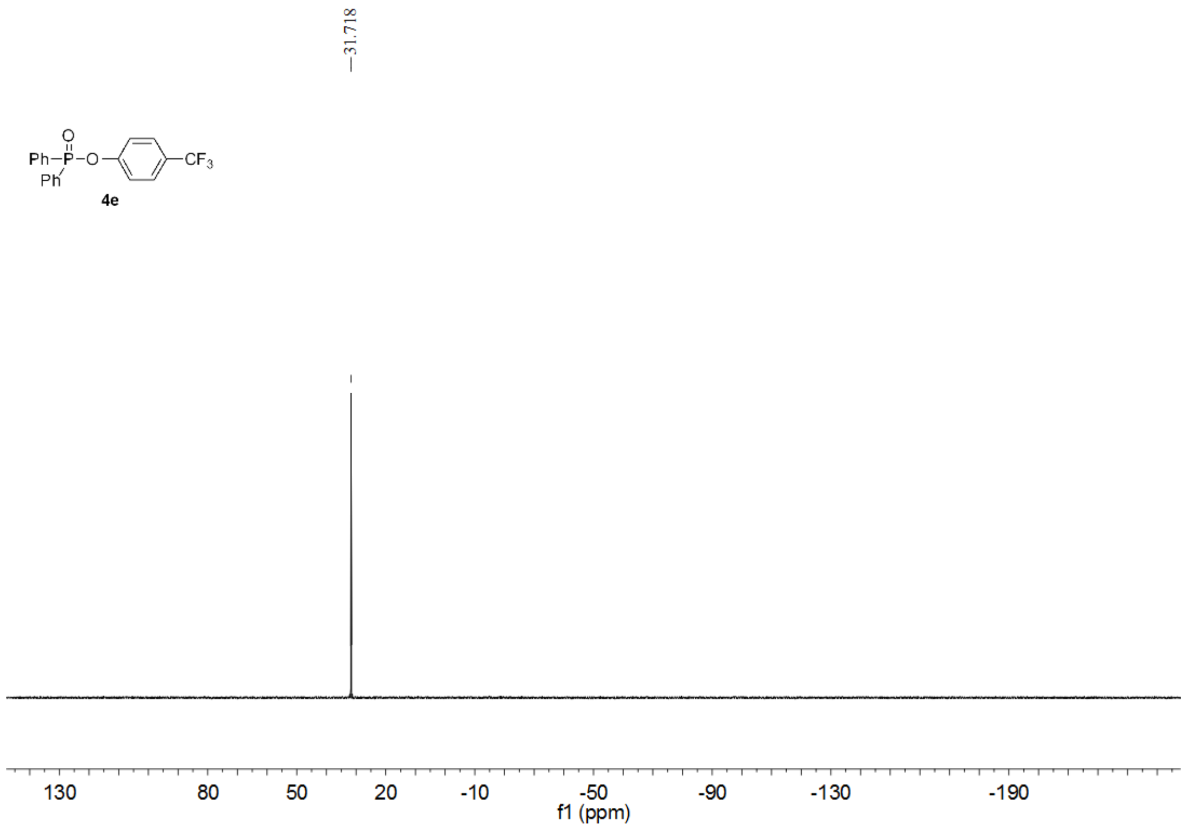

31.212
Ph
O
P
O
F
Ph
4d
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)

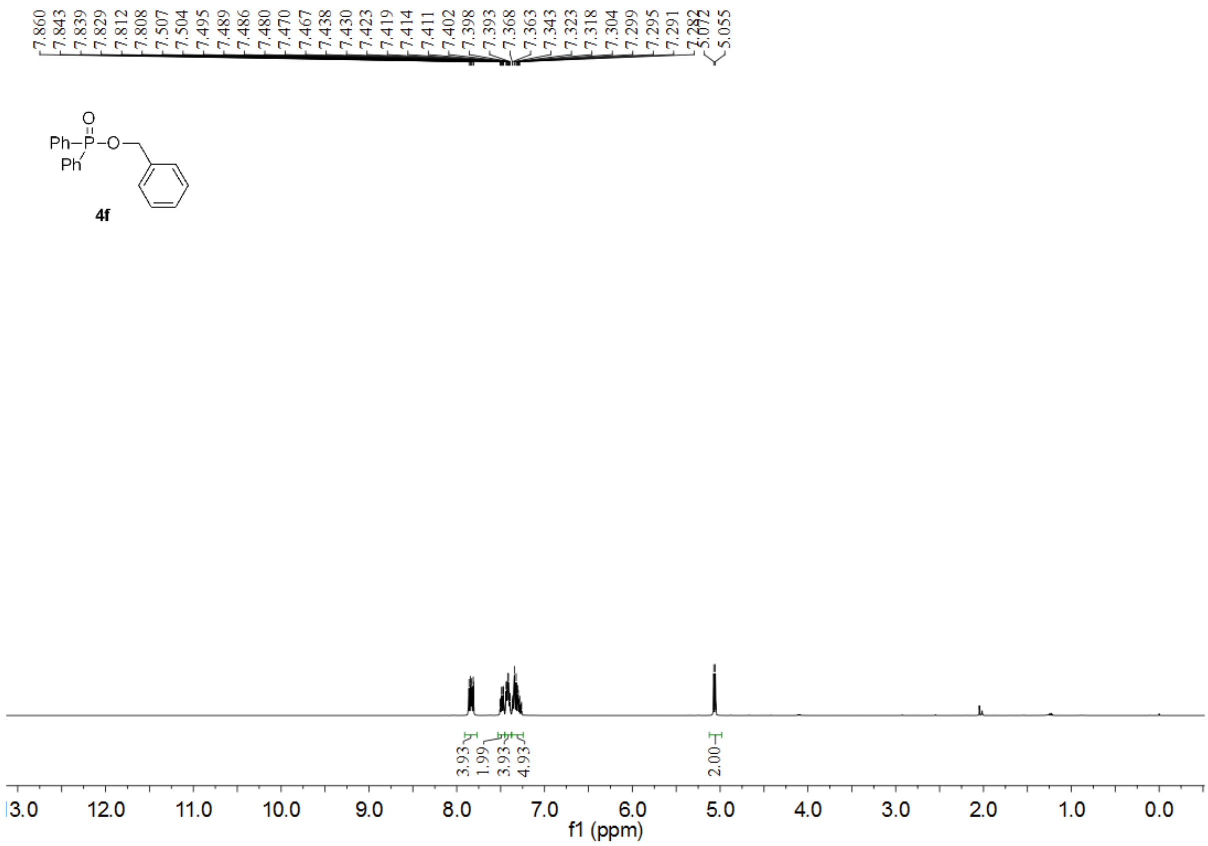


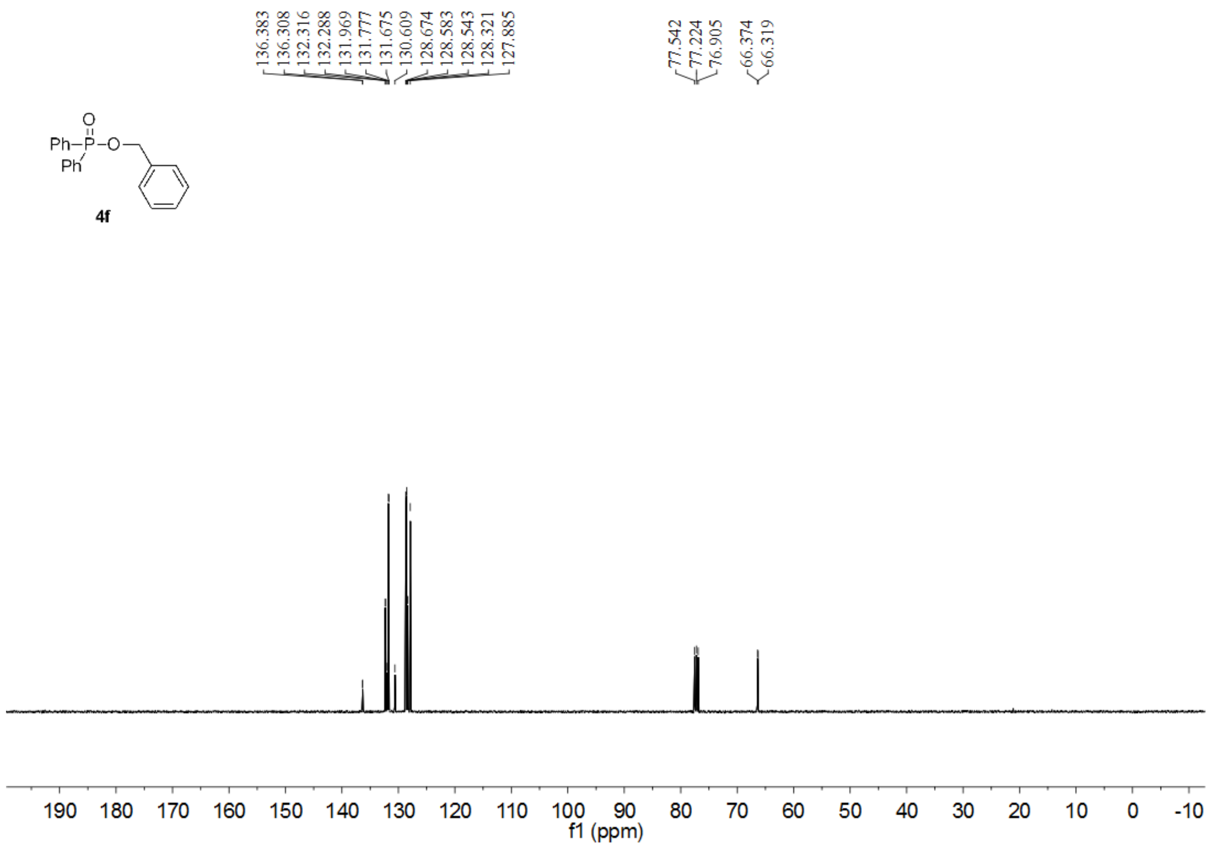
7.922
7.904
7.900
7.890
7.872
7.869
7.580
7.577
7.562
7.559
7.543
7.540
7.524
7.504
7.495
7.485
7.476
7.468
7.458
7.343
7.322
Ph
P
O
Ph
CF$_3$
4e
3.93
7.97
2.00
12.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)

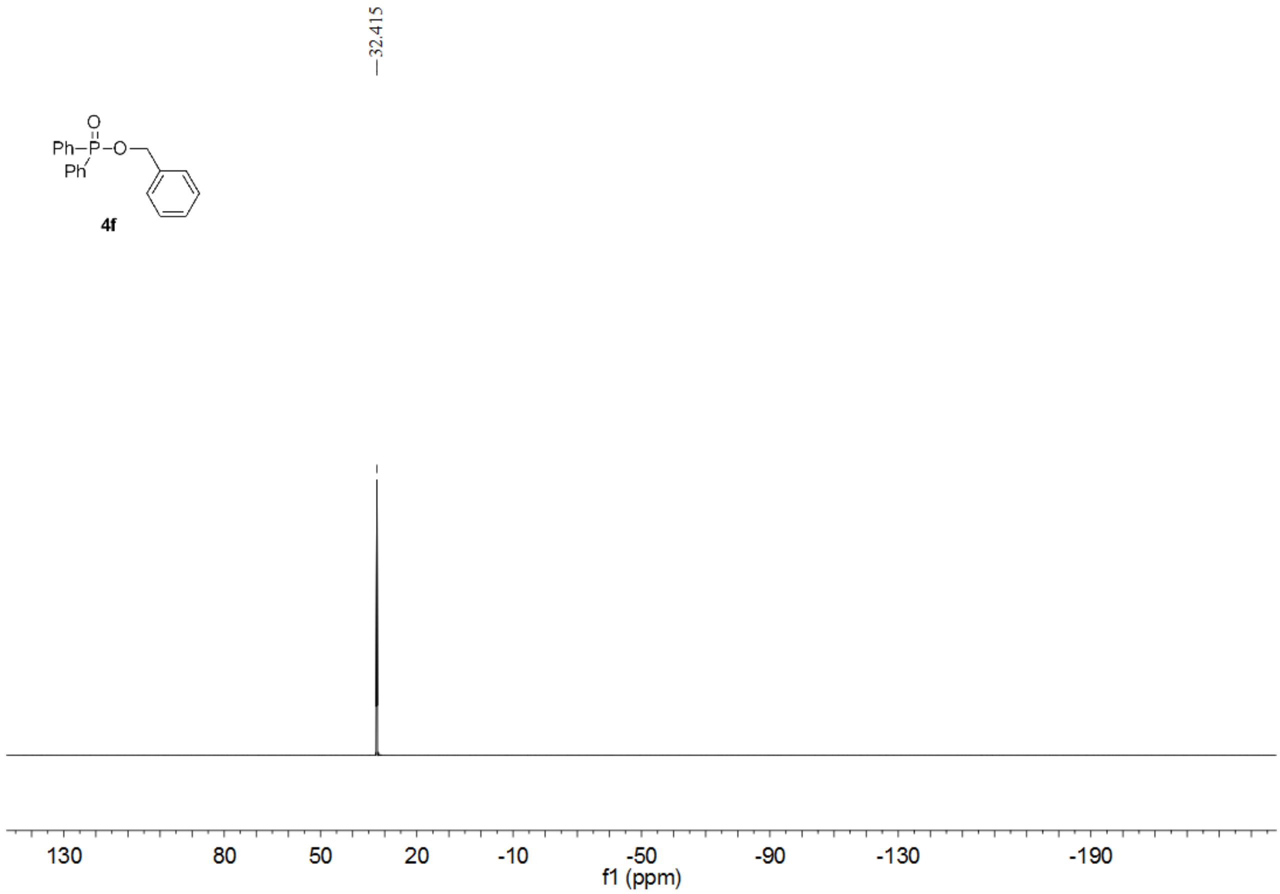


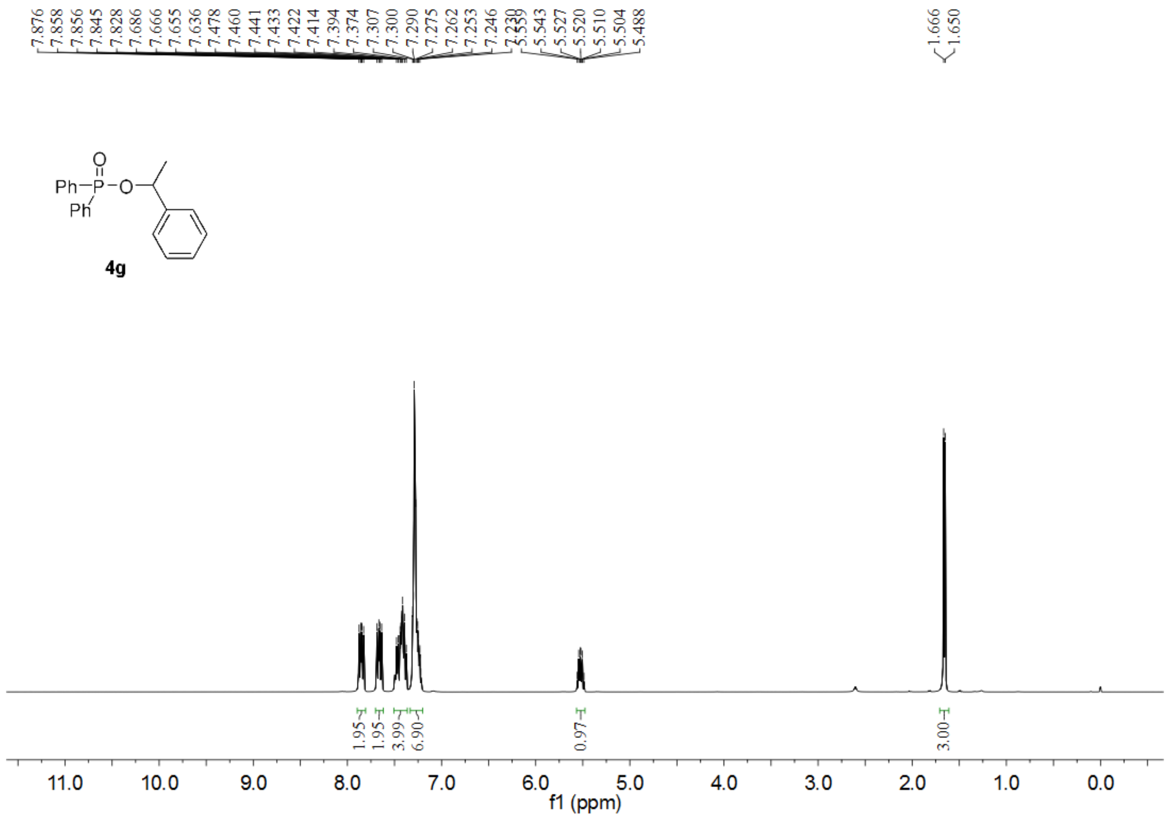
153.572
153.493
132.815
132.786
131.789
131.684
131.078
129.705
128.840
128.705
127.930
127.346
127.135
127.099
127.062
127.023
126.689
125.228
122.527
121.034
120.985
77.369
77.051
76.733
Ph
P
O
Ph
CF$_3$
4e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

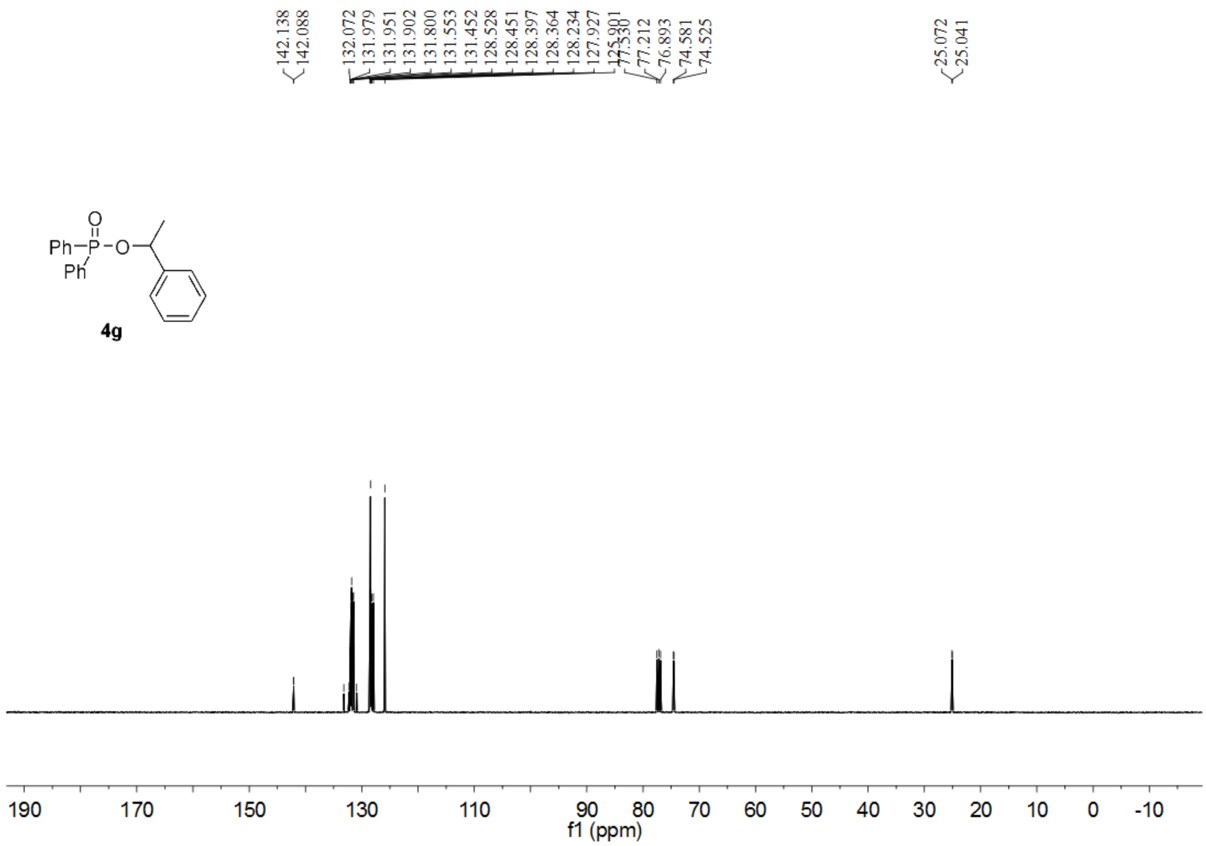

31.718
Ph
P
O
Ph
O
CF3
4e
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)

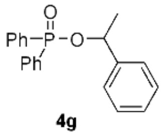

7.860
7.843
7.839
7.829
7.812
7.808
7.507
7.504
7.495
7.489
7.486
7.480
7.470
7.467
7.438
7.430
7.423
7.419
7.414
7.411
7.402
7.398
7.393
7.368
7.363
7.343
7.323
7.318
7.304
7.299
7.295
7.291
7.282
5.072
5.055
Ph
P
O
Ph
O
4f
3.93
1.99
3.93
4.93
2.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)



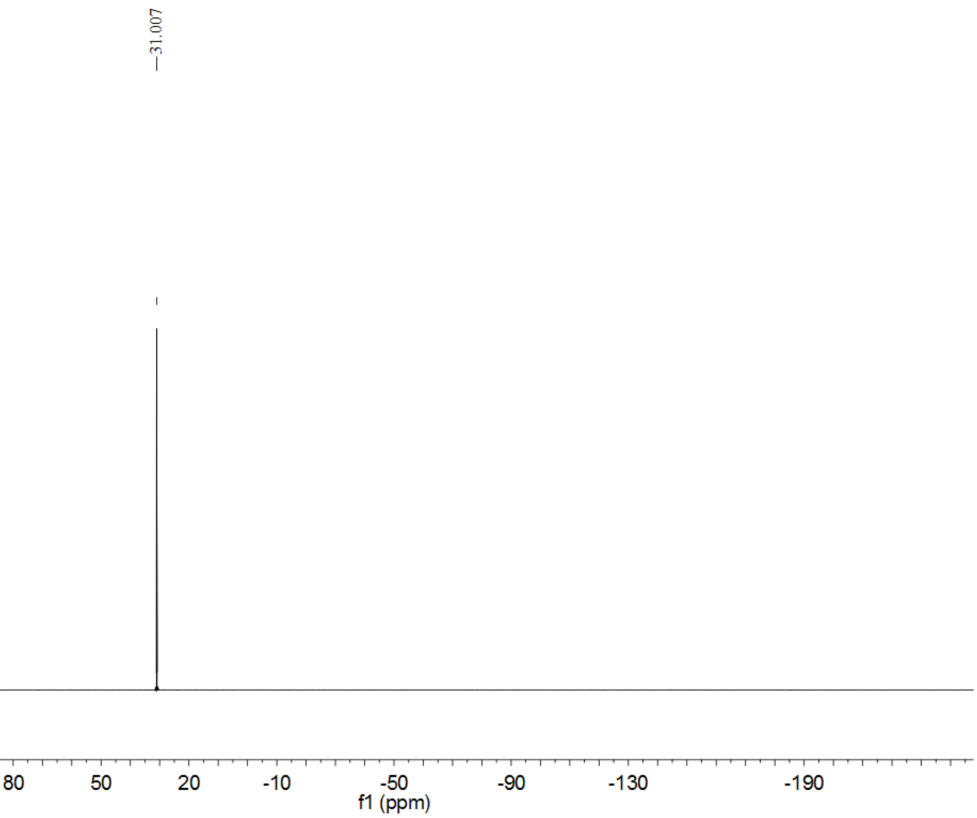



7.876
7.858
7.856
7.845
7.828
7.686
7.666
7.655
7.636
7.478
7.460
7.441
7.433
7.422
7.414
7.394
7.374
7.307
7.300
7.290
7.275
7.262
7.253
7.246
7.239
5.559
5.543
5.527
5.520
5.510
5.504
5.488
1.666
1.650
Ph
P
O
O
Ph
4g
1.95
1.95
3.99
6.90
0.97
3.00
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

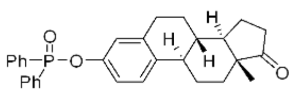

142.138
142.088
132.072
131.979
131.951
131.902
131.800
131.553
131.452
128.528
128.451
128.397
128.364
128.234
127.927
125.901
77.530
77.212
76.893
74.581
74.525
25.072
25.041
Ph
P
O
O
Ph
4g
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



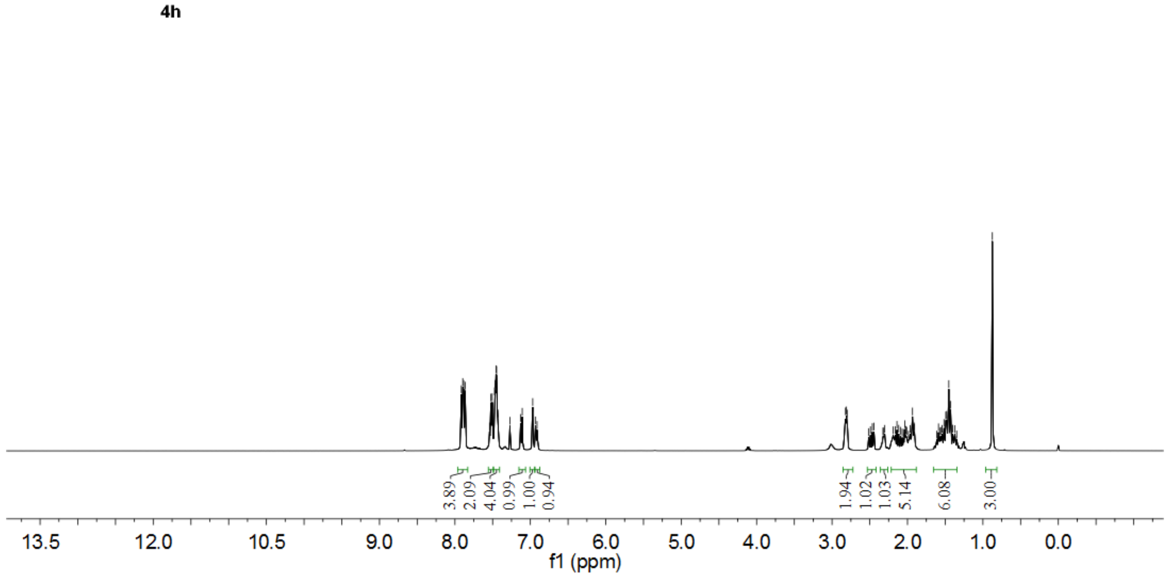
4g



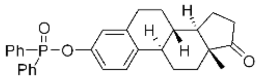
31.007
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)



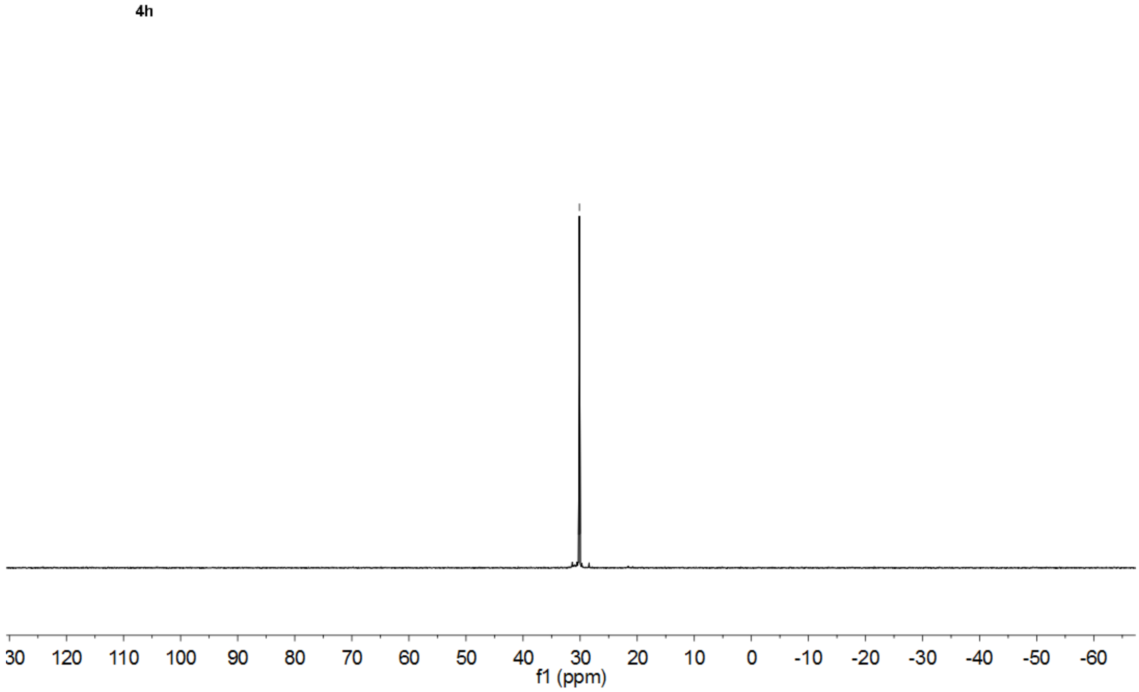
4h



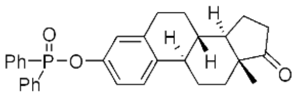
7.915
7.896
7.884
7.865
7.541
7.523
7.505
7.474
7.466
7.456
7.448
7.430
7.271
7.129
7.108
6.969
6.932
6.911
2.821
2.809
2.800
2.466
2.444
2.301
2.140
2.116
2.035
2.022
1.965
1.933
1.913
1.907
1.588
1.541
1.516
1.492
1.481
1.452
1.429
1.401
1.372
3.89
2.09
4.04
0.99
1.00
0.94
1.94
1.02
1.03
5.14
6.08
3.00
13.5
12.0
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)



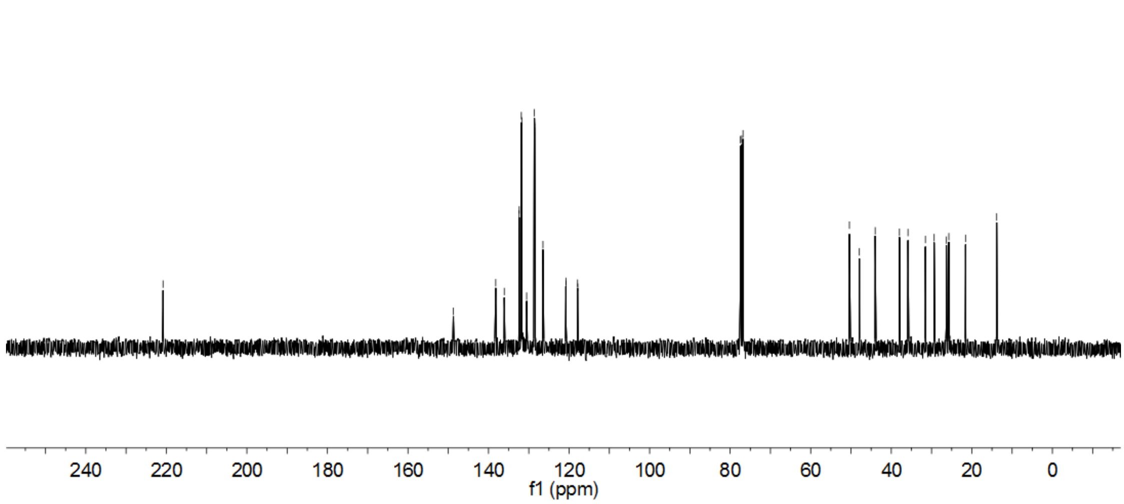
4h



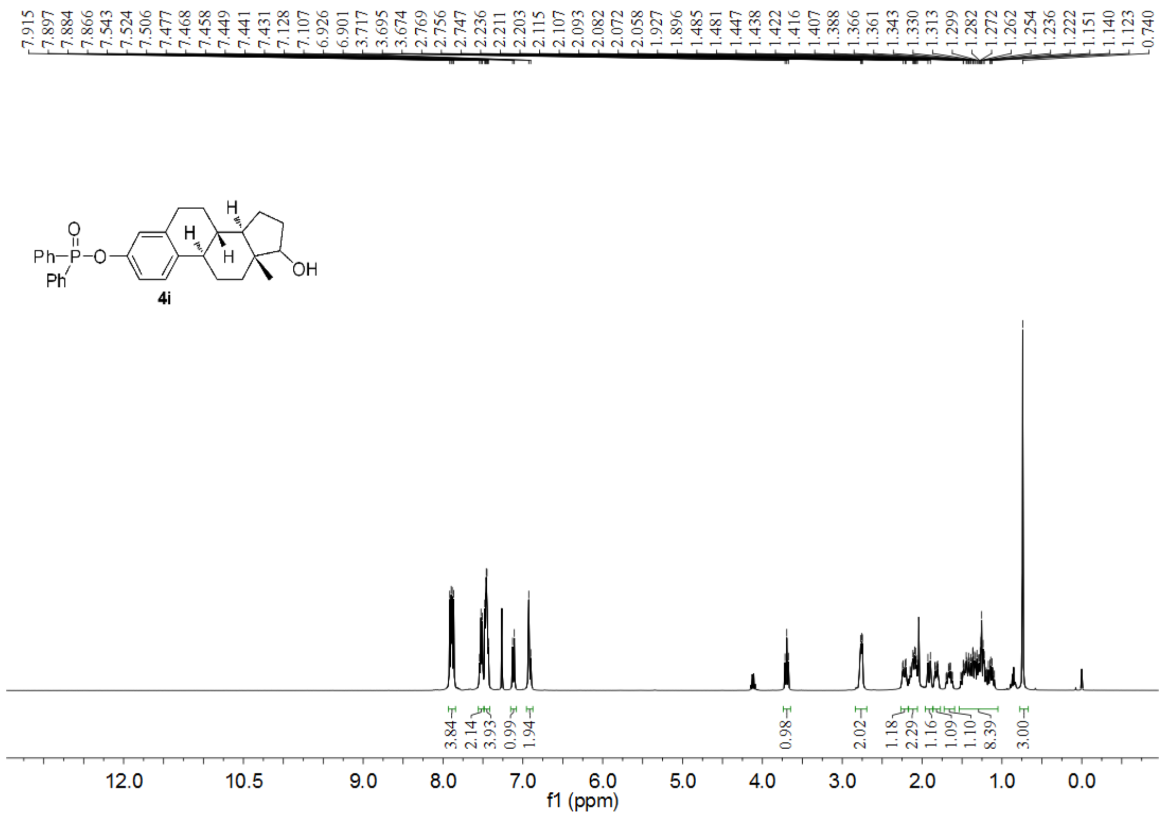
30.126
130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60
f1 (ppm)

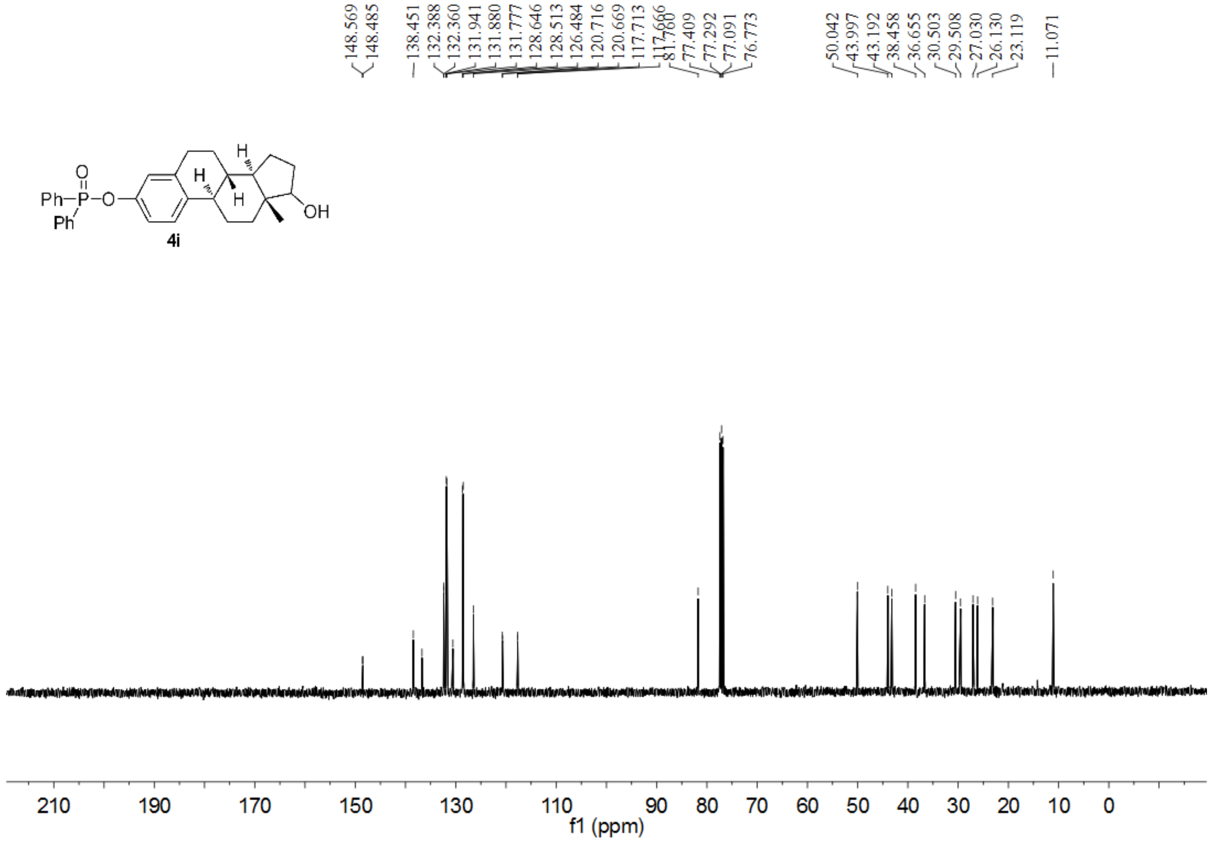


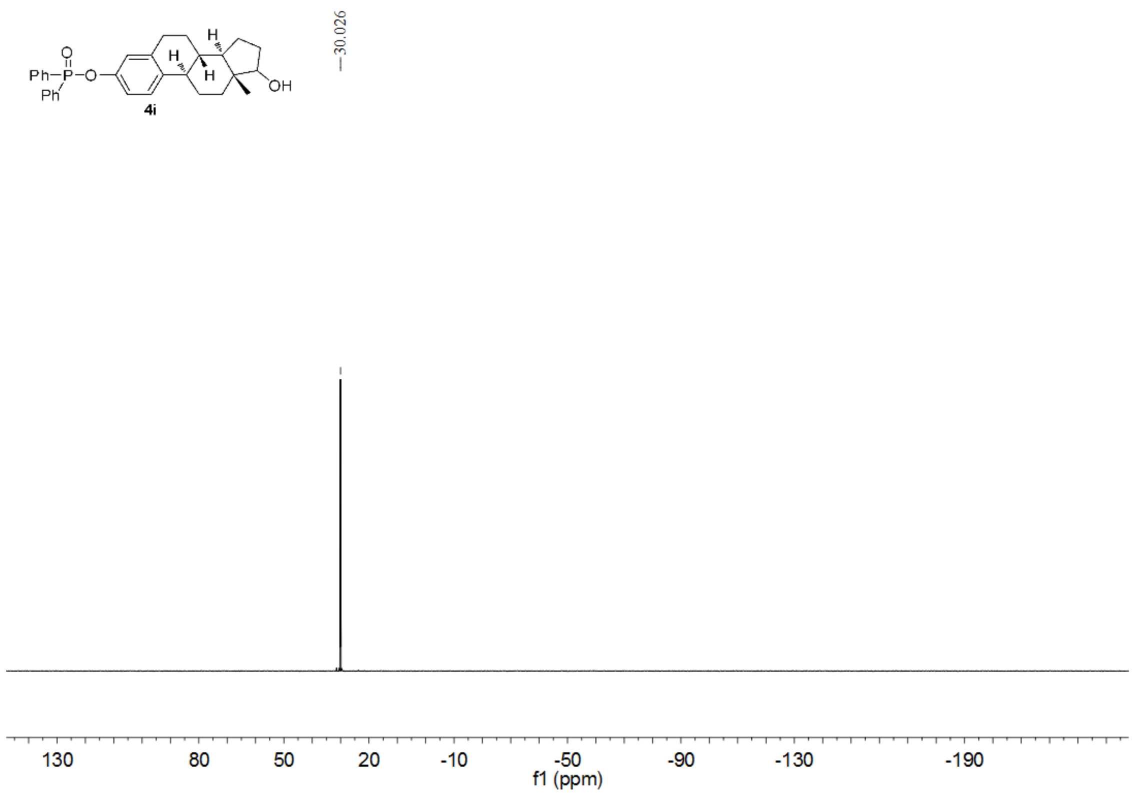
4h



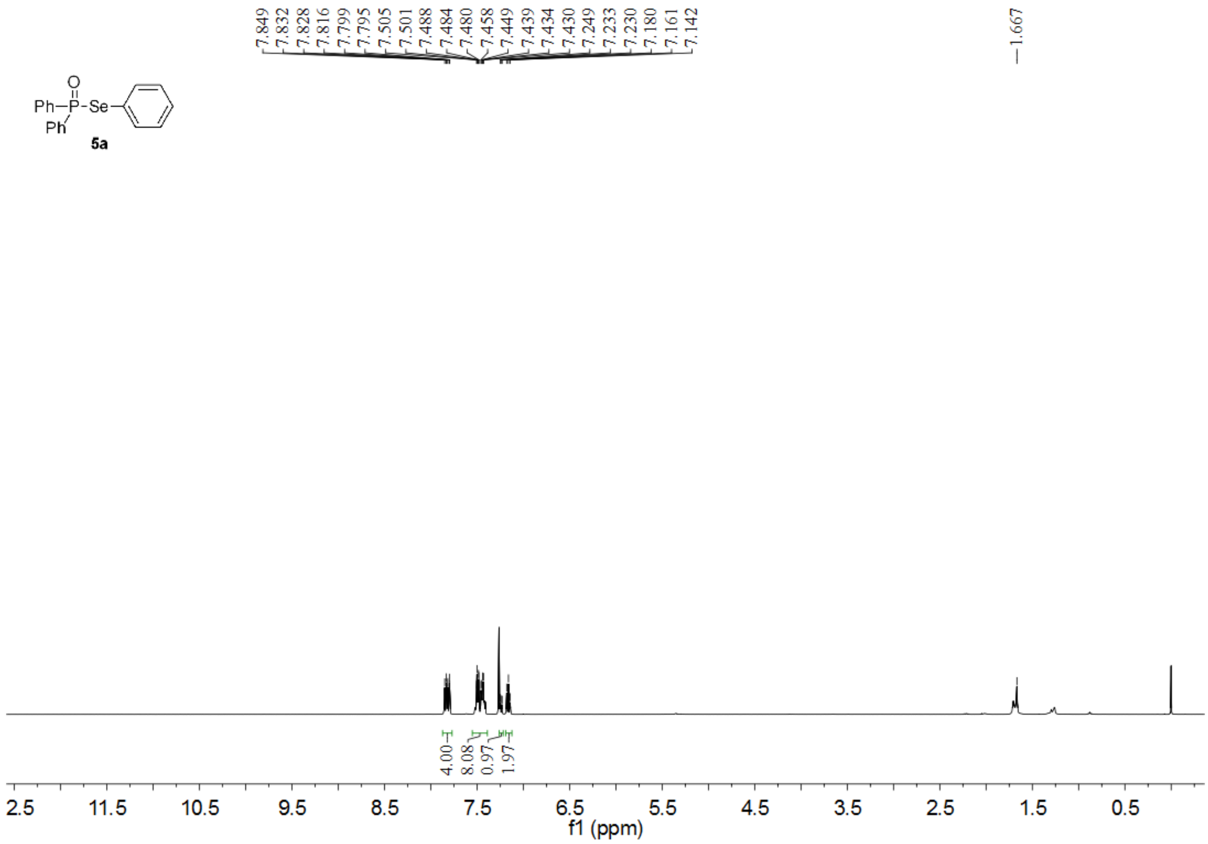
220.816
148.758
148.674
138.215
132.409
132.383
131.906
131.854
131.751
128.653
128.520
126.487
120.794
120.750
117.892
77.451
77.133
76.815
50.396
47.925
43.991
37.968
35.847
31.525
29.350
26.306
25.706
21.563
13.831
240 220 200 180 160 140 120 100 80 60 40 20 0
f1 (ppm)


7.915 7.897 7.884 7.866 7.543 7.524 7.506 7.477 7.468 7.458 7.449 7.441 7.431 7.128 7.107 6.926 6.901 3.717 3.695 3.674 2.769 2.756 2.747 2.236 2.211 2.203 2.115 2.107 2.093 2.082 2.072 2.058 1.927 1.896 1.485 1.481 1.447 1.438 1.422 1.416 1.407 1.388 1.366 1.361 1.343 1.330 1.313 1.299 1.282 1.272 1.262 1.254 1.236 1.222 1.151 1.140 1.123 0.740
O
Ph P O
Ph
H
H
H
OH
4i
3.84 2.14 3.93 0.99 1.94 0.98 2.02 1.18 2.29 1.16 1.09 1.10 8.39 3.00
12.0 10.5 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)

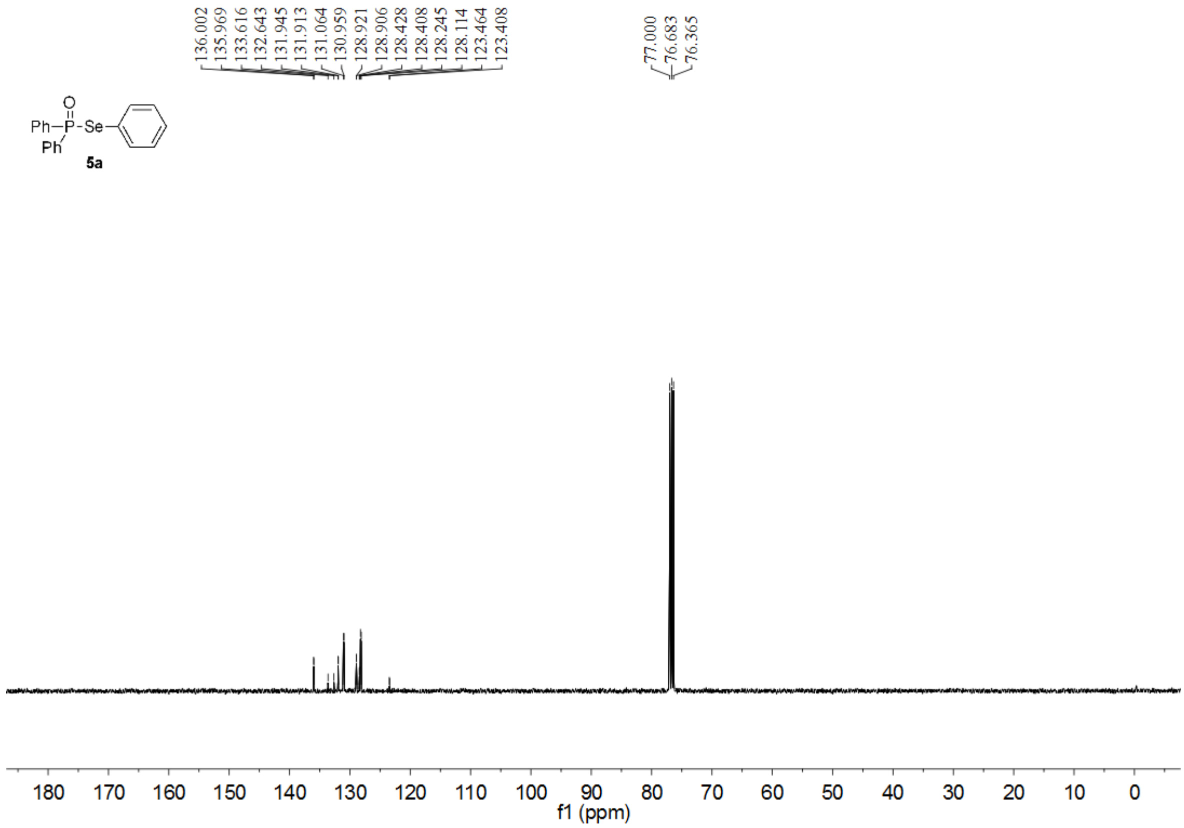

148.569 148.485 138.451 132.388 132.360 131.941 131.880 131.777 128.646 128.513 126.484 120.716 120.669 117.713 117.666 81.760 77.409 77.292 77.091 76.773 50.042 43.997 43.192 38.458 36.655 30.503 29.508 27.030 26.130 23.119 11.071
O
Ph P O
Ph
H
H
H
OH
4i
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

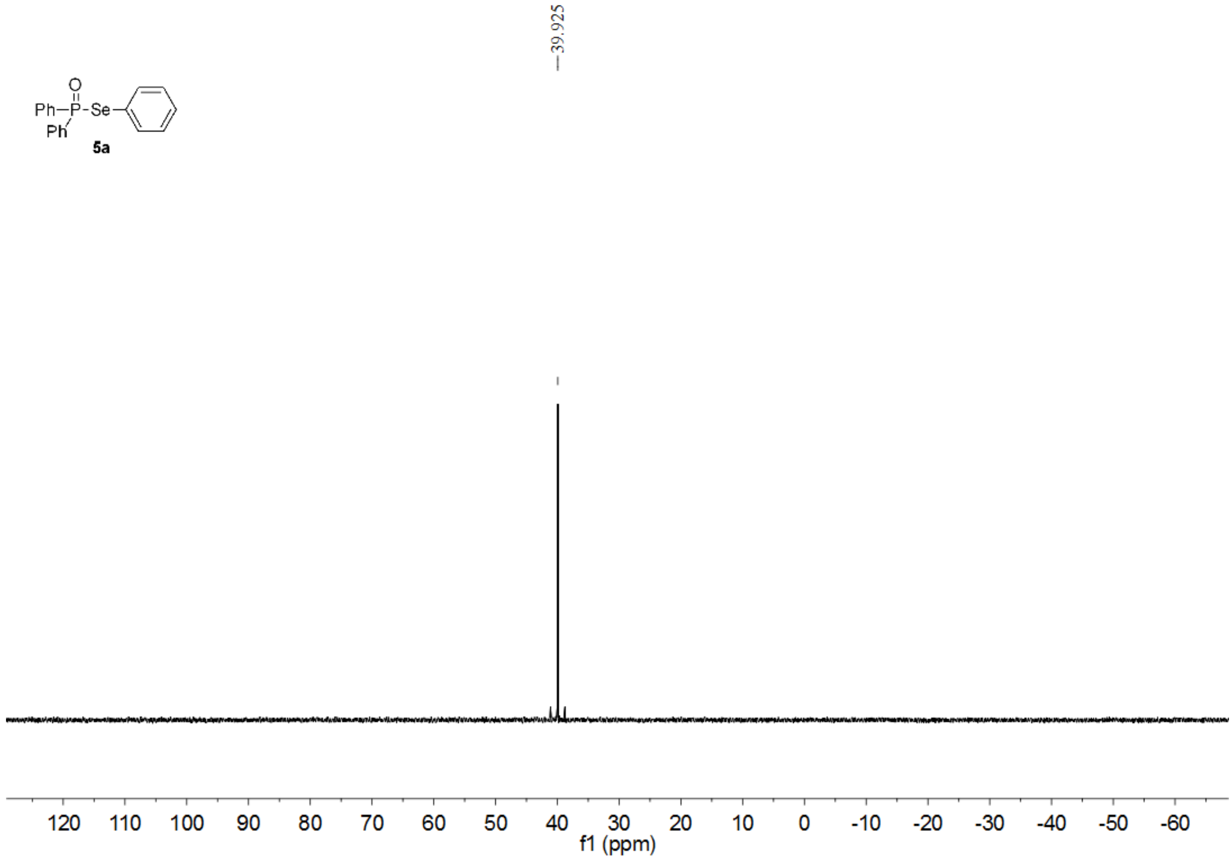


Ph–P–O
Ph
4i
OH
30.026
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)



Ph–P–Se
Ph
5a
7.849
7.832
7.828
7.816
7.799
7.795
7.505
7.501
7.488
7.484
7.480
7.458
7.449
7.439
7.434
7.430
7.249
7.233
7.230
7.180
7.161
7.142
1.667
4.00
8.08
0.97
1.97
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


136.002
135.969
133.616
132.643
131.945
131.913
131.064
130.959
128.921
128.906
128.428
128.408
128.245
128.114
123.464
123.408
77.000
76.683
76.365
Ph
P
O
Se
Ph
5a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


39.925
Ph
P
O
Se
Ph
5a
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
f1 (ppm)