Supporting Information

Nickel-Catalysed P-C Bonds Formation via P-H/C-CN Cross Couplings

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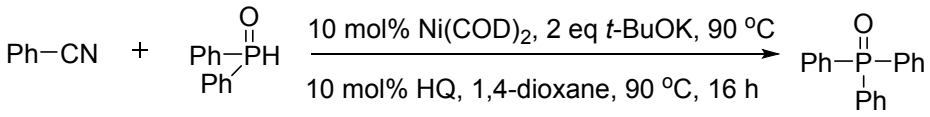
General information

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).

General procedure for the synthesis of phosphine oxides

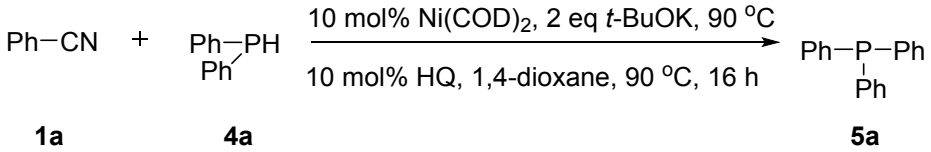


1a **2a** **3a**

Under N_2 atmosphere, 0.26 mmol diphenylphosphine oxide, 0.2 mmol benzonitrile, 10 mol% $Ni(COD)_2$, 10 mol% 8-hydroxyquinoline (HQ), 0.4 mmol *t*-BuOK and 1.0 mL 1,4-

dioxane were charged into a 25 mL schlenk tube, and the mixture was stirred at 90 °C for 16 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ethyl acetate as eluent) to afford analytically pure triphenylphosphine oxide **3**.

General procedure for the synthesis of phosphines



Under N_2 atmosphere, 0.26 mmol diphenylphosphine oxide, 0.2 mmol benzonitrile, 10 mol% $Ni(COD)_2$, 10 mol% 8-hydroxyquinoline (HQ), 0.4 mmol *t*-BuOK and 1.0 mL 1,4-dioxane were charged into a 25 mL schlenk tube, and the mixture was stirred at 90 °C for 16 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ethyl acetate as eluent) to afford analytically pure triphenylphosphine **5**.

Detection of KCN

(a) By trapping with PhCHO

After the reaction of benzonitrile (0.2 mmol) with diphenylphosphine oxide (0.2 mmol) completed under standard reaction conditions, acetic acid (1 mmol) and PhCHO (0.2 mmol) were added to the mixture at room temperature. The mixture was stirred for 6 h. No 2-hydroxy-2-phenylacetonitrile was detected by GC-MS.

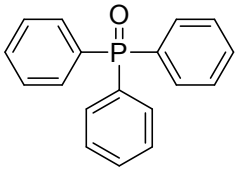
(b) By color change test

Picrate paper was by wetting filter paper with a solution of 5.0 g sodium bicarbonate and 0.5 g picric acid in 100 mL water. After drying the paper, it was cut into strips for use. Under N_2 atmosphere, 0.26 mmol diphenylphosphine oxide, 0.2 mmol benzonitrile, 10 mol% $Ni(COD)_2$, 10 mol% 8-hydroxyquinoline (HQ), 0.4 mmol *t*-BuOK and 1.0 mL 1,4-dioxane were charged into a 25 mL schlenk tube, and the mixture was stirred at 90 °C for

16 h. Tartaric acid (0.2 g) were added and a strip was placed above the reaction mixture. The schlenk tube was heated 80 °C for 1h. However no color change of the strip was observed (the strip should change to red if cyanide anion was present).

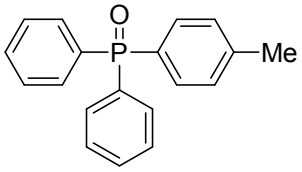
Ref. (a) J. Kim et al, *J. Am. Chem. Soc.*, **2012**, *134*, 2528; (b) G. Zhang et al, *Org. Lett.*, **2011**, *13*, 5004.

Characterization data of 3



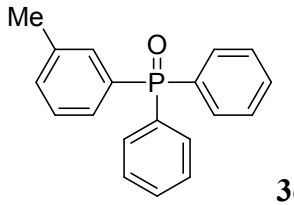
3a[1]

Following the general procedure (90 °C, 16 h), **3a** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62-7.67 (m, 6H),7.49-7.53(m, 3H), 7.40-7.45 (m, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.14; ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.54 (d, J_{P-C} = 101.9 Hz), 132.08 (d, J_{P-C} = 9.8 Hz), 131.95 (d, J_{P-C} = 2.8 Hz), 128.51 (d, J_{P-C} = 12.1 Hz). MS (EI): 278.



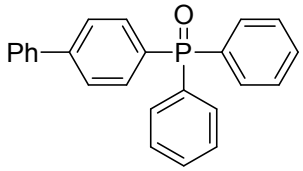
3b[1]

Following the general procedure (90 °C, 16 h), **3b** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.69 (m, 4H), 7.49-7.58 (m, 4H), 7.42-7.47 (m, 4H), 7.25-7.28 (m, 2H), 2.39 (s, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.21; ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.44 (d, J_{P-C} = 2.8 Hz), 132.78 (d, J_{P-C} = 103.3 Hz), 132.10 (d, J_{P-C} = 10.1 Hz), 132.03 (d, J_{P-C} = 9.8 Hz), 131.82 (d, J_{P-C} = 2.8 Hz), 129.24 (d, J_{P-C} = 12.4 Hz), 129.11 (d, J_{P-C} = 105.9 Hz), 128.43 (d, J_{P-C} = 12.0 Hz), 21.59 (d, J_{P-C} = 1.0 Hz). MS (EI): 292.



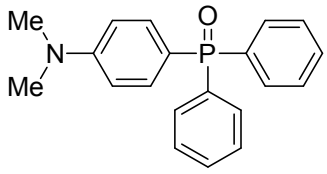
3c[2]

Following the general procedure (90 °C, 16 h), **3c** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.69 (m, 4H), 7.52-7.59 (m, 3H), 7.43-7.48 (m, 4H), 7.29-7.40 (m, 3H), 2.36 (s, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.55; ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.47 (d, J_{P-C} = 11.9 Hz), 132.54 (d, J_{P-C} = 102.4 Hz), 132.80 (d, J_{P-C} = 2.7 Hz), 132.48 (d, J_{P-C} = 9.4 Hz), 132.14 (d, J_{P-C} = 103.3 Hz), 132.08 (d, J_{P-C} = 9.9 Hz), 131.90 (d, J_{P-C} = 2.7 Hz), 129.17 (d, J_{P-C} = 10.2 Hz), 128.47 (d, J_{P-C} = 12.1 Hz), 128.31 (d, J_{P-C} = 12.9 Hz), 21.42 (b). MS (EI): 292.



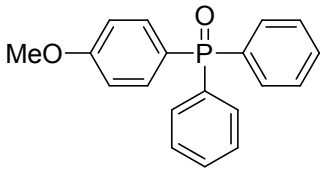
3e[1]

Following the general procedure (90 °C, 16 h), **3e** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.76 (m, 8H), 7.53-7.60 (m, 4H), 7.38-7.50 (m, 7H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.20; ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.76 (d, J_{P-C} = 2.6 Hz), 139.89 (b), 132.63 (d, J_{P-C} = 10.2 Hz), 132.57 (d, J_{P-C} = 97.6 Hz), 132.13 (d, J_{P-C} = 9.8 Hz), 132.03 (d, J_{P-C} = 2.6 Hz), 131.05 (d, J_{P-C} = 104.4 Hz), 128.99, 128.59 (d, J_{P-C} = 12.0 Hz), 128.21, 127.29, 127.16. MS (EI): 354.



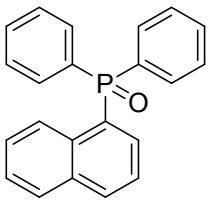
3f[12]

Following the general procedure (90 °C, 16 h), **3f** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.70 (m, 4H), 7.42-7.53 (m, 8H), 6.71 (dd, 2H, J = 8.8 Hz, J = 2.4 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.76; ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.54 (d, J_{P-C} = 2.1 Hz), 133.83 (d, J_{P-C} = 103.3 Hz), 133.57 (d, J_{P-C} = 11.2 Hz), 132.16 (d, J_{P-C} = 9.7 Hz), 131.61 (d, J_{P-C} = 2.5 Hz), 128.41 (d, J_{P-C} = 11.9 Hz), 116.76 (d, J_{P-C} = 114.9 Hz), 111.34 (d, J_{P-C} = 12.9 Hz), 40.05. MS (EI): 321.



3g[1]

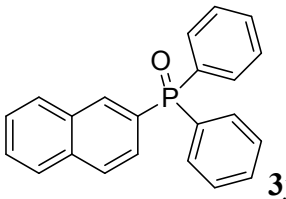
Following the general procedure (90 °C, 16 h), **3g** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.68 (m, 12H), 6.95-6.95 (m, 2H), 3.84 (s, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.03; ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.49 (d, J_{P-C} = 2.8 Hz), 133.99 (d, J_{P-C} = 11.2 Hz), 133.00 (d, J_{P-C} = 103.7 Hz), 132.06 (d, J_{P-C} = 9.8 Hz), 131.80 (d, J_{P-C} = 2.7Hz), 128.45 (d, J_{P-C} = 12.0 Hz), 123.60 (d, J_{P-C} = 109.7 Hz), 114.08 (d, J_{P-C} = 13.1 Hz), 55.36. MS (EI): 308.



3i[2]

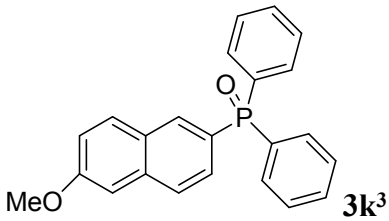
Following the general procedure (90 °C, 16 h), **3i** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (d, 1H, J = 8.4 Hz), 8.02(d, 1H, J = 8.4 Hz), 7.89 (d, 1H, J = 8 Hz), 7.66-7.71 (m, 4H), 7.27-7.57 (m, 10 H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.58; ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.90 (d, J_{P-C} = 8.4 Hz), 133.80 (d, J_{P-C} = 10.7 Hz), 133.71 (d, J_{P-C} = 8.0 Hz), 133.33 (d, J_{P-C} = 2.6 Hz), 132.72 (d, J_{P-C} = 104.1 Hz), 132.09 (d, J_{P-C} = 9.8 Hz), 131.93 (d, J_{P-C} = 2.5 Hz), 128.83 (d, J_{P-C} = 101.0 Hz), 128.79. 128.61 (d, J_{P-C}

= 12.1 Hz), 127.59 (d, J_{P-C} = 5.7 Hz), 127.38, 126.52, 124.16 (d, J_{P-C} = 14.2 Hz). MS (EI): 328.



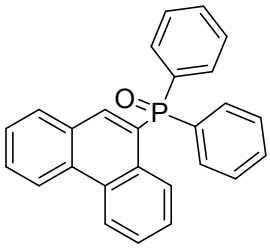
3j[1]

Following the general procedure (90 °C, 16 h), **3j** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (d, 1H, J = 13.6 Hz), 7.87-7.92 (m, 3H), 7.45-7.74 (m, 13H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.34; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.73 (d, J_{P-C} = 2.3 Hz), 134.04 (d, J_{P-C} = 9.3 Hz), 132.53 (d, J_{P-C} = 103.8 Hz), 132.44 (d, J_{P-C} = 13.1 Hz), 132.17 (d, J_{P-C} = 9.8 Hz), 132.03 (d, J_{P-C} = 2.7 Hz), 129.53 (d, J_{P-C} = 106.6 Hz), 129.00, 128.58 (d, J_{P-C} = 12.0 Hz), 128.34 (d, J_{P-C} = 10.2 Hz), 128.29, 127.85, 126.98, 126.86 (d, J_{P-C} = 10.6 Hz). MS (EI): 328.



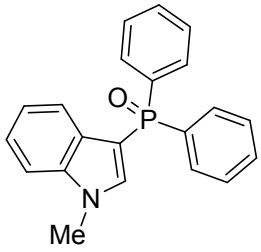
3k[3]

Following the general procedure (90 °C, 16 h), **3k** was isolated as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, 1H, J = 13.6 Hz), 7.68-7.80 (m, 6H), 7.44-7.62 (m, 7H), 7.14-7.20 (m, 2H), 3.92 (s, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.60; ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.45, 135.29 (d, J_{P-C} = 2.4 Hz), 132.67 (d, J_{P-C} = 9.6 Hz), 131.68 (d, J_{P-C} = 102.2 Hz), 131.12 (d, J_{P-C} = 9.8 Hz), 130.90 (d, J_{P-C} = 2.6 Hz), 129.49, 127.49 (d, J_{P-C} = 12.0 Hz), 126.88 (d, J_{P-C} = 13.3 Hz), 126.53 (d, J_{P-C} = 10.8 Hz), 126.08 (d, J_{P-C} = 12 Hz), 125.69 (d, J_{P-C} = 105.9 Hz), 118.85, 104.62, 54.40. MS (EI): 358.



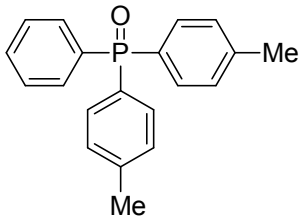
3l[4]

Following the general procedure (90 °C, 16 h), **3l** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68-8.73 (m, 2H), 8.62 (d, 1H, J = 8.4 Hz), 7.45-7.77 (m, 16H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.91; ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.92 (d, J_{P-C} = 11.2 Hz), 132.53 (d, J_{P-C} = 104.2 Hz), 132.21, 132.11, 132.05 (d, J_{P-C} = 2.7 Hz), 130.92 (d, J_{P-C} = 8.4 Hz), 130.76 (d, J_{P-C} = 8.4 Hz), 130.08, 129.68 (d, J_{P-C} = 14.8 Hz), 129.17, 128.75, 128.69 (d, J_{P-C} = 5.5 Hz), 128.63, 127.75 (d, J_{P-C} = 100.4 Hz), 127.22 (d, J_{P-C} = 5.4 Hz), 127.04, 123.06, 122.66. MS (EI): 378.



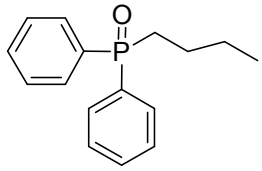
3m[7]

Following the general procedure (90 °C, 16 h), **3m** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75-7.80 (m, 4H), 7.33-7.52 (m, 8H), 7.20-7.27 (m, 2H), 7.06 (t, 1 H, J = 7.6 Hz), 3.76 (s, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 21.55; ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.26 (d, J_{P-C} = 10.3 Hz), 137.36 (d, J_{P-C} = 19.2 Hz), 133.90 (d, J_{P-C} = 107.3 Hz), 131.74 (d, J_{P-C} = 10.3 Hz), 131.63 (d, J_{P-C} = 2.6 Hz), 128.74 (d, J_{P-C} = 9.2 Hz), 128.40 (d, J_{P-C} = 12.1 Hz), 122.83, 121.36, 121.18, 109.93, 104.57 (d, J_{P-C} = 127.6 Hz), 33.31. MS (EI): 331.



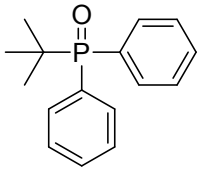
3n[1]

Following the general procedure (90 °C, 16 h), **3n** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.66 (m, 2H), 7.47-7.55 (m, 5H), 7.39-7.43 (m, 2H), 7.23 (d, 4H, J = 2.4 Hz), 2.39 (s, 6 H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.37; ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.36 (d, J_{P-C} = 2.8 Hz), 132.98 (d, J_{P-C} = 103.5 Hz), 132.07 (d, J_{P-C} = 10.2 Hz), 132.01 (d, J_{P-C} = 9.8 Hz), 131.75 (d, J_{P-C} = 2.7 Hz), 129.32 (d, J_{P-C} = 106.0 Hz), 129.21 (d, J_{P-C} = 12.4 Hz), 128.40 (d, J_{P-C} = 12.0 Hz), 21.59 (d, J_{P-C} = 0.9 Hz). MS (EI): 306.



3o[6]

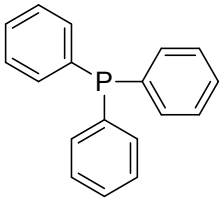
Following the general procedure (90 °C, 16 h), **3o** was isolated as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68-7.73 (m, 4H), 7.41-7.50 (m, 6H), 2.20-2.27 (m, 2H), 1.51-1.62 (m, 2H), 1.35-1.44 (m, 2H), 0.86 (t, 3H, J = 7.2 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.80; ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.28 (d, J_{P-C} = 135.6 Hz), 130.62 (d, J_{P-C} = 2.6 Hz), 129.74 (d, J_{P-C} = 9.2 Hz), 127.60 (d, J_{P-C} = 11.5 Hz), 28.42 (d, J_{P-C} = 71.8 Hz), 23.07 (d, J_{P-C} = 15.0 Hz), 22.44 (d, J_{P-C} = 4.0 Hz), 12.57. MS (EI): 258.



3p[5]

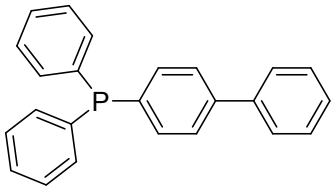
Following the general procedure (90 °C, 16 h), **3p** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (t, 4H, J = 8.4 Hz), 7.43-7.51 (m, 6H), 1.22 (d, 9H, J = 14.8 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ 38.70; ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.40 (d, J_{P-C} = 8.0 Hz), 131.69 (d, J_{P-C} = 2.7 Hz), 131.36 (d, J_{P-C} = 89.8 Hz), 128.49 (d, J_{P-C} = 10.8 Hz), 34.17 (d, J_{P-C} = 70.3 Hz), 25.41. MS (EI): 258.

Characterization data of 5



5a[8]

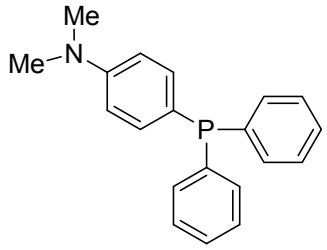
Following the general procedure (90 °C, 16 h), **5a** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (bm, 15H); ^{31}P NMR (162 MHz, $CDCl_3$) δ -5.37; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.46 (d, J_{P-C} = 10.7 Hz), 133.99 (d, J_{P-C} = 19.4 Hz), 128.97, 128.76 (d, J_{P-C} = 6.9 Hz). MS (EI): 262.



5d[10]

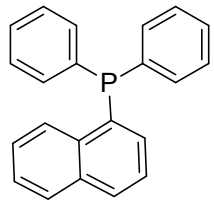
Following the general procedure (90 °C, 16 h), **5d** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.65 (m, 4H), 7.37-7.50 (m, 15H); ^{31}P NMR (162 MHz, $CDCl_3$) δ -6.10; ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.76, 140.76, 137.38 (d, J_{P-C} = 10.4 Hz), 136.28 (d, J_{P-C} = 10.5 Hz), 134.45 (d, J_{P-C} = 19.4 Hz), 134.04 (d, J_{P-C} = 19.3 Hz),

129.24, 129.10, 128.82 (d, J_{P-C} = 6.9 Hz), 127.83, 127.45 (d, J_{P-C} = 7.0 Hz), 127.34. MS (EI): 338.



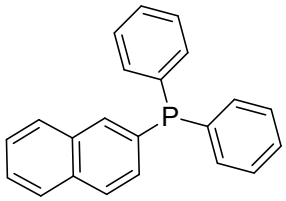
5e[10]

Following the general procedure (90 °C, 16 h), **5e** was isolated as oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.48 (m, 12H), 6.88 (d, 2H, J = 7.6 Hz), 3.14 (s, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ -7.31; ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.10 (b), 138.96 (d, J_{P-C} = 10.0 Hz), 135.84 (d, J_{P-C} = 21.5 Hz), 133.65, 133.47, 128.57 (d, J_{P-C} = 606 Hz), 128.45, 112.56 (b), 40.51. MS (EI): 305.



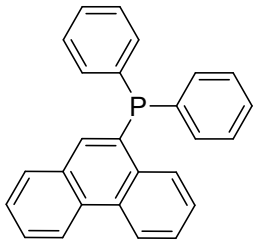
5f[8]

Following the general procedure (90 °C, 16 h), **5f** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (dd, 1H, J = 4.0 Hz, J = 8.0 Hz), 7.87 (t, 2H, J = 8.8 Hz), 7.44-7.54 (m, 2H), 7.33-7.39 (m, 11H), 7.03 (t, 1H, J = 6.0 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ -14.23; ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.62(d, J_{P-C} = 9.5 Hz), 135.59 (d, J_{P-C} = 22 Hz), 134.83 (d, J_{P-C} = 13.9 Hz), 134.49 (d, J_{P-C} = 19.7 Hz), 133.69 (d, J_{P-C} = 4.4 Hz), 132.32, 129.75, 109.02 (d, J_{P-C} =22.1Hz), 128.87 (d, J_{P-C} =7 Hz), 128.79 (d, J_{P-C} = 8.2 Hz) ,127.23 (d, J_{P-C} = 126 Hz), 126.56 (d, J_{P-C} = 3.4 Hz), 125.88 (d, J_{P-C} = 1.4 Hz), 125.36 (d, J_{P-C} = 25.8 Hz). MS (EI): 312.



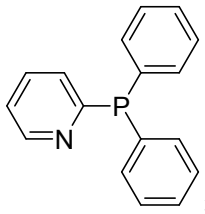
5g[8]

Following the general procedure (90 °C, 16 h), **5g** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.84 (m, 3H), 7.75 (d, 2H, J = 7.6 Hz), 7.46-7.53 (m, 2H), 7.35-7.43 (m, 11H); ^{31}P NMR (162 MHz, $CDCl_3$) δ -4.90; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.39 (d, J_{P-C} = 10.7 Hz), 134.98 (d, J_{P-C} = 11.0 Hz), 134.49 (d, J_{P-C} = 22.2 Hz), 134.11 (d, J_{P-C} = 19.3 Hz), 133.65, 133.55 (d, J_{P-C} = 8.2 Hz), 130.34 (d, J_{P-C} =17.4 Hz), 129.09, 128.86 (d, J_{P-C} = 6.8 Hz), 128.41, 128.26 (d, J_{P-C} = 6.7 Hz), 127.99, 127.01, 126.58. MS (EI): 312.



5h[9]

Following the general procedure (90 °C, 16 h), **5h** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, 1H, J = 8.4 Hz), 8.59 (d, 1H, J = 8.4 Hz), 8.37 (dd, 1H, J = 4.8 Hz, J = 8.0 Hz), 7.55 (q, 3H, J = 7.2 Hz), 7.40-7.46 (m, 2H), 7.26-7.31 (m, 10H), 7.17 (d, 1H, J = 5.6 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ -12.95; ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.31 (d, J_{P-C} = 9.3 Hz), 134.67 (d, J_{P-C} = 19.8 Hz), 134.10, 133.72 (d, J_{P-C} = 3.6 Hz), 133.55 (d, J_{P-C} = 10.7 Hz), 131.69 (d, J_{P-C} = 2.1 Hz), 131.06, 130.45 (d, J_{P-C} = 2.3 Hz), 129.28, 129.23, 128.94 (d, J_{P-C} = 7.2 Hz), 127.59, 127.44, 127.18, 127.01 (d, J_{P-C} = 2.1 Hz), 126.95, 123.26 (d, J_{P-C} = 1.5 Hz), 122.79. MS (EI): 362.



5i[8]

Following the general procedure (90 ^{o}C, 16 h), **5i** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (d, 1H, J = 4.8 Hz), 7.52-7.58 (m, 1H), 7.35-7.42 (m, 10H), 7.15-7.18 (m, 1H), 7.08 (d, 1H, J = 7.6 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ -4.00; ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.27 (d, J_{P-C} = 4.4 Hz), 150.59 (d, J_{P-C} = 12.6 Hz), 136.45 (d, J_{P-C} = 10.7 Hz), 135.96 (d, J_{P-C} = 2.1 Hz), 134.44 (d, J_{P-C} = 19.7 Hz), 129.32, 128.88 (d, J_{P-C} =7.1 Hz), 128.07 (d, J_{P-C} =15.3 Hz), 122.42. MS (EI): 263.

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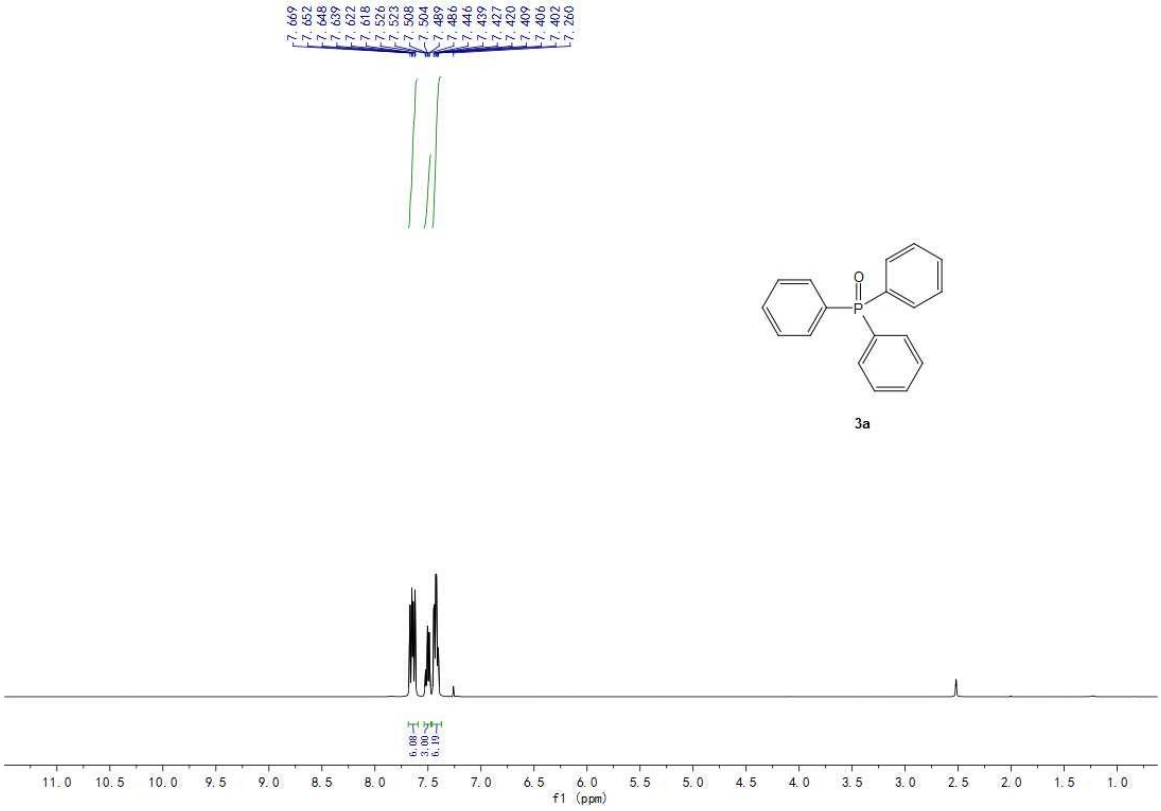
Copies of ^{1}H NMR, ^{31}P NMR and ^{13}C NMR spectra



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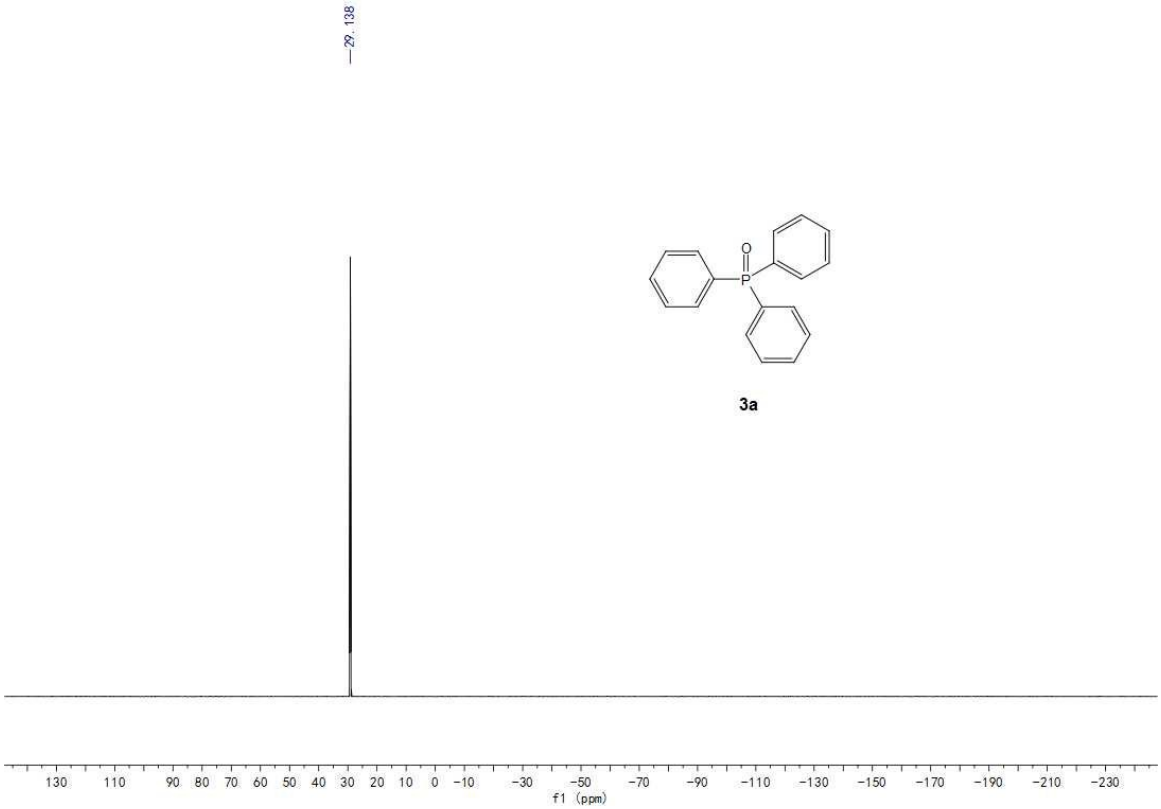

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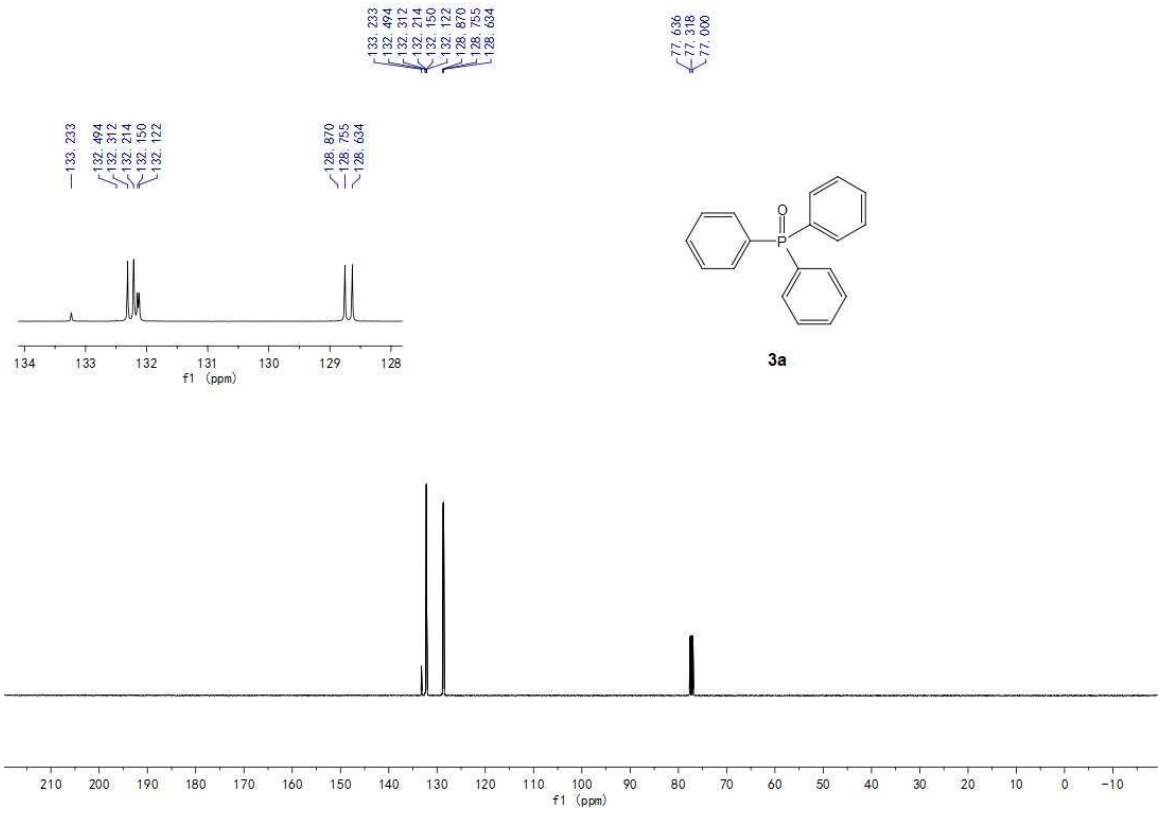
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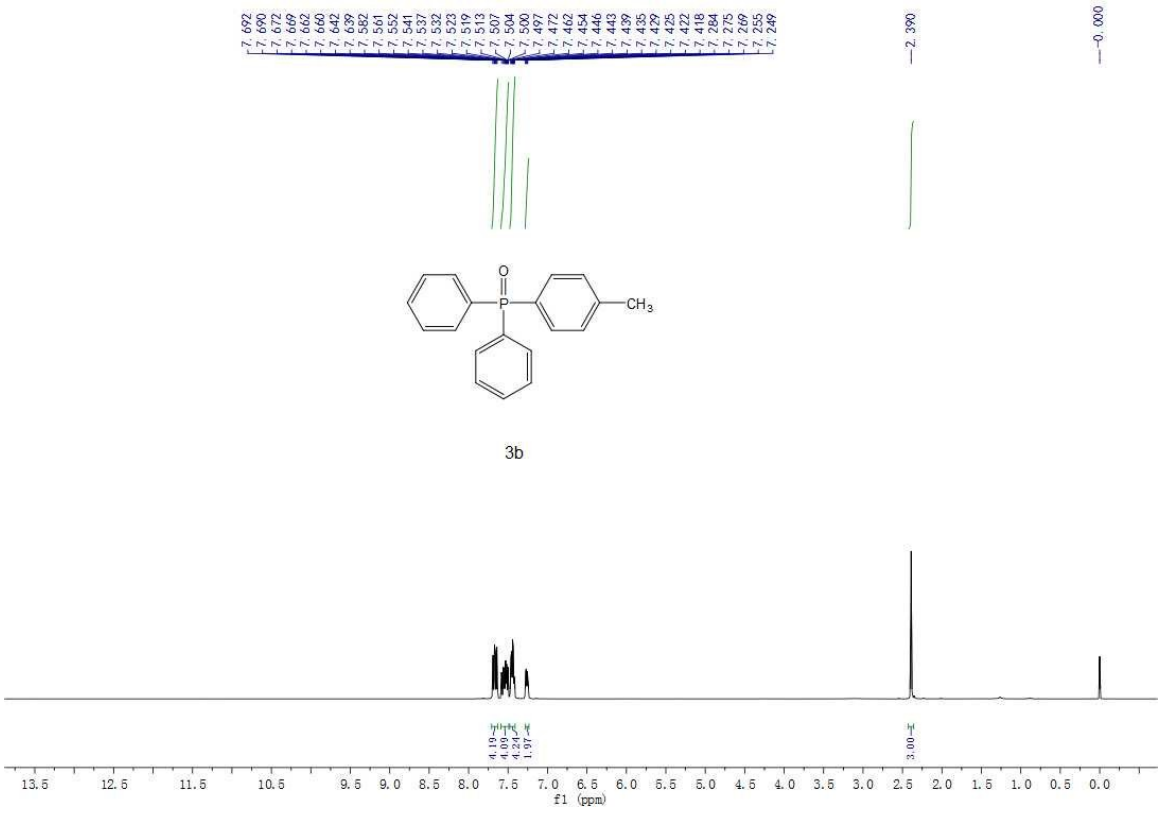
77.318
77.000
f1 (ppm)
3a

f1 (ppm)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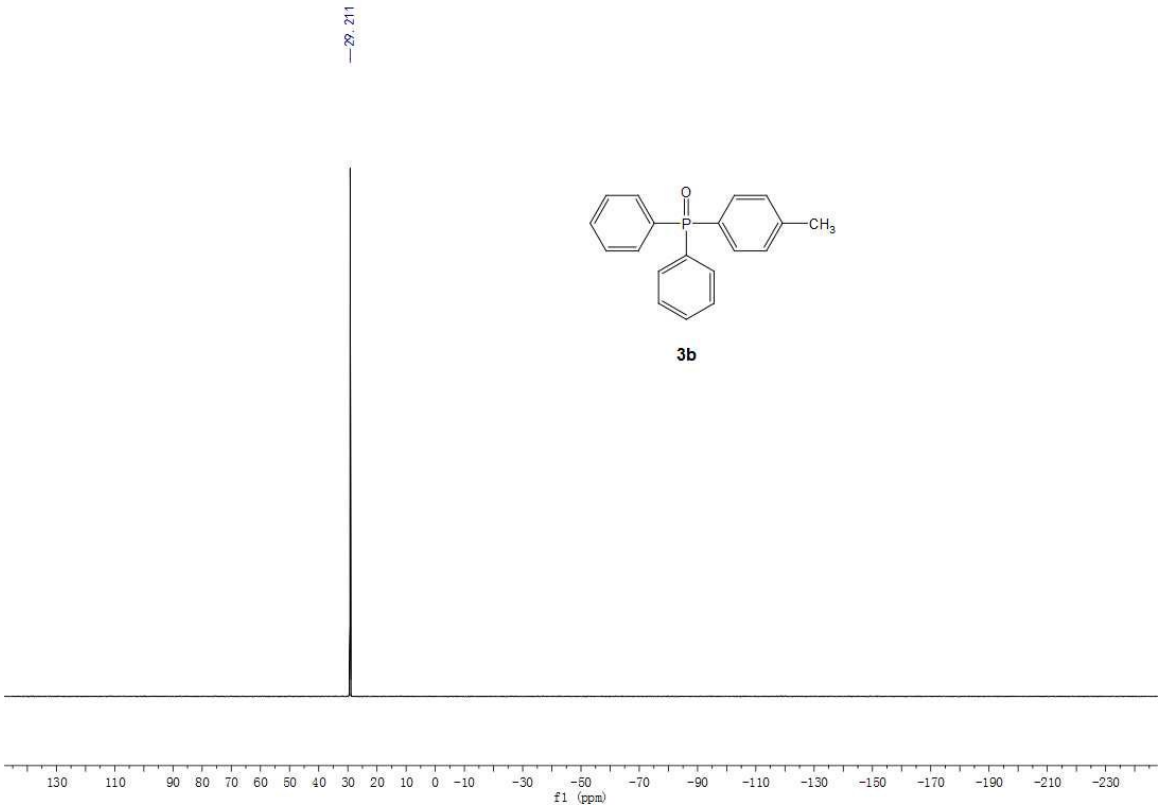
7.537
7.532
7.523
7.519
7.513
7.507
7.504
7.500
7.497
7.472
7.462
7.454
7.446
7.443
7.439
7.435
7.429
7.425
7.422
7.418
7.284
7.275
7.269
7.255
7.249
2.390
0.000
CH3
3b
4.19
4.09
4.24
1.97
3.00
f1 (ppm)


29.211
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

3b


142.759
142.731
133.591
132.558
132.447
132.380
132.346
132.282
132.215
132.134
132.106
129.937
129.605
129.481
128.878
128.790
128.670
77.725
77.407
77.089
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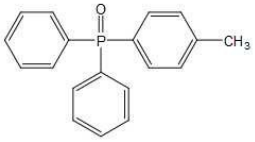
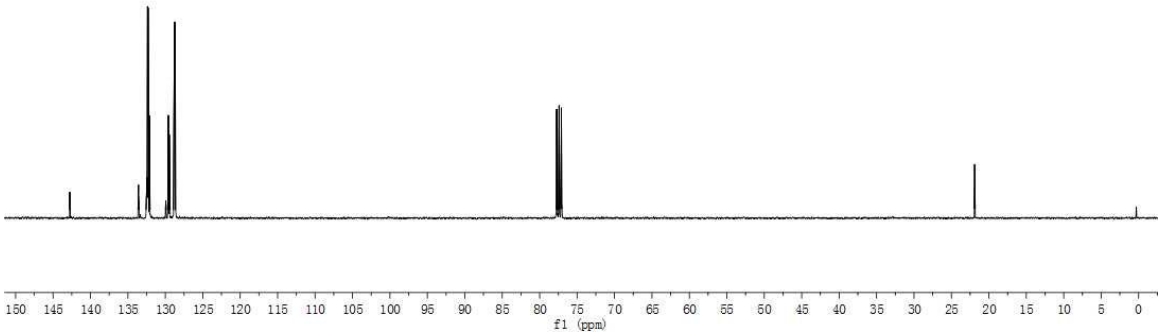
21.888
0.299



3b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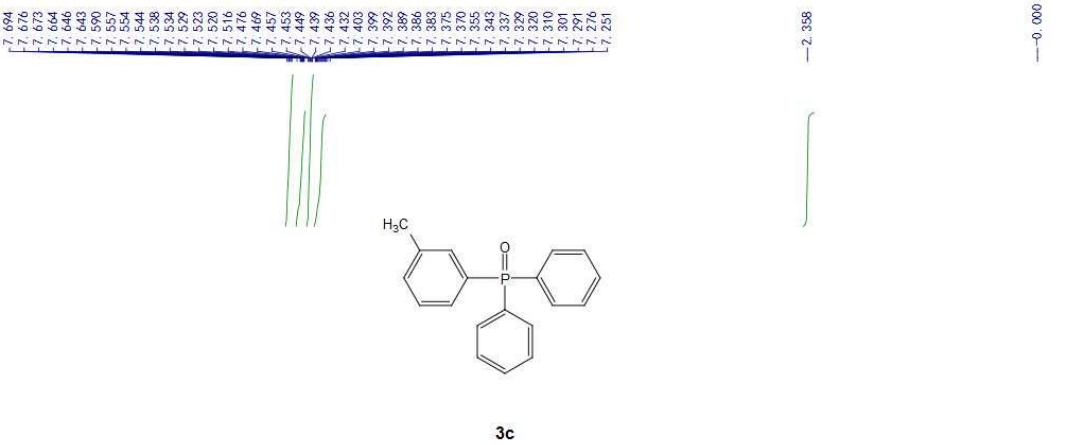
f1 (ppm)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7.523
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7.516
7.476
7.469
7.457
7.453
7.449
7.439
7.436
7.432
7.403
7.399
7.392
7.389
7.386
7.383
7.375
7.370
7.355
7.343
7.337
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7.310
7.301
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7.276
7.251
2.358
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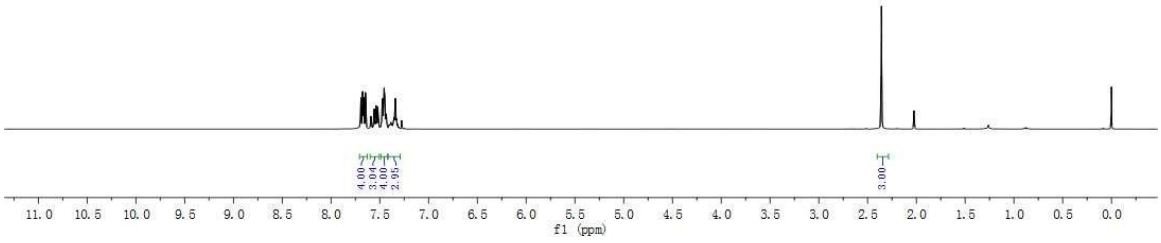
H3C
O
P

3c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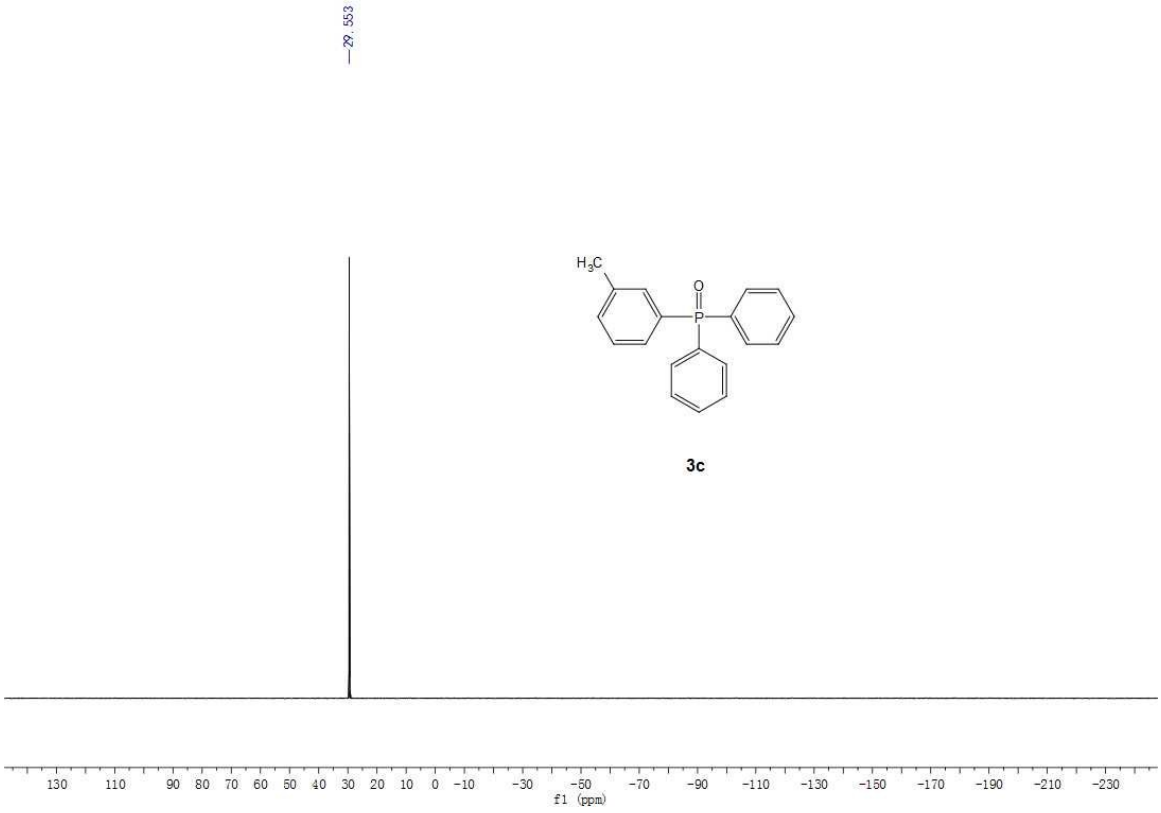
3.04
4.00
2.95
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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O
P
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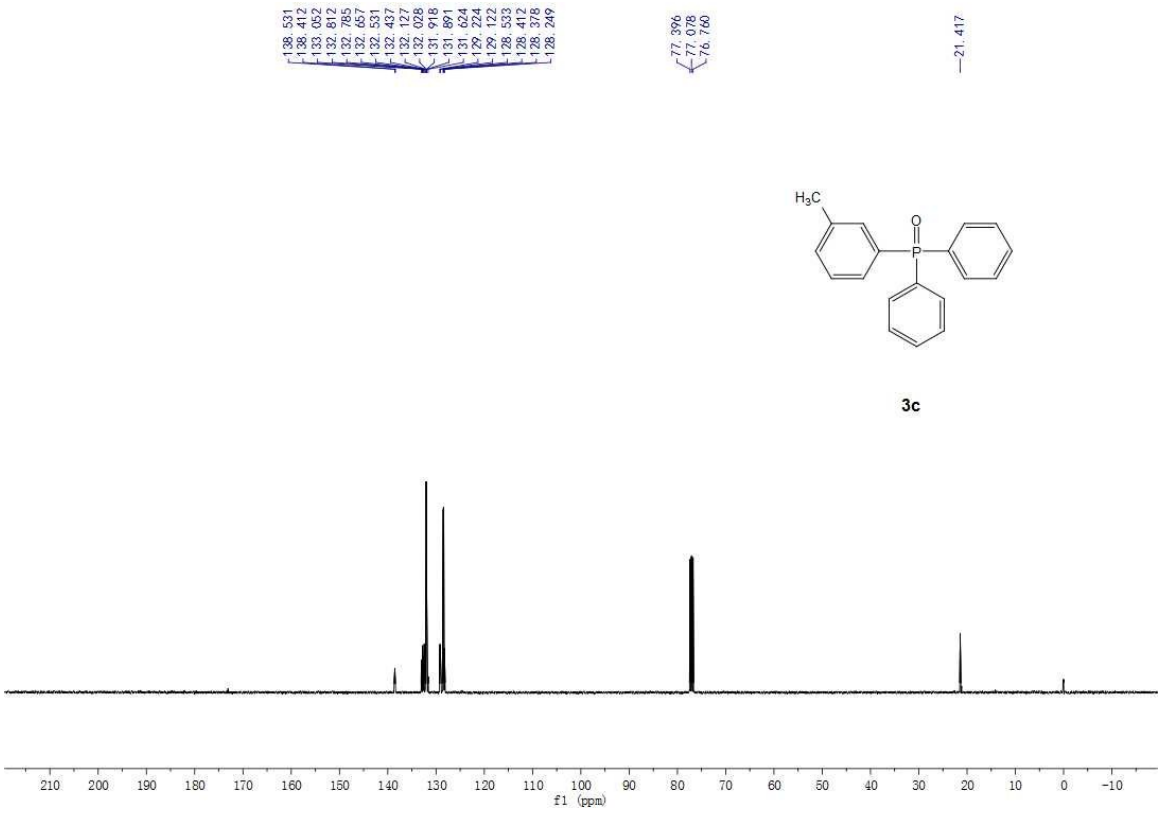
f1 (ppm)

3c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132.785
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132.531
132.437
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132.028
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131.891
131.624
129.224
129.122
128.533
128.412
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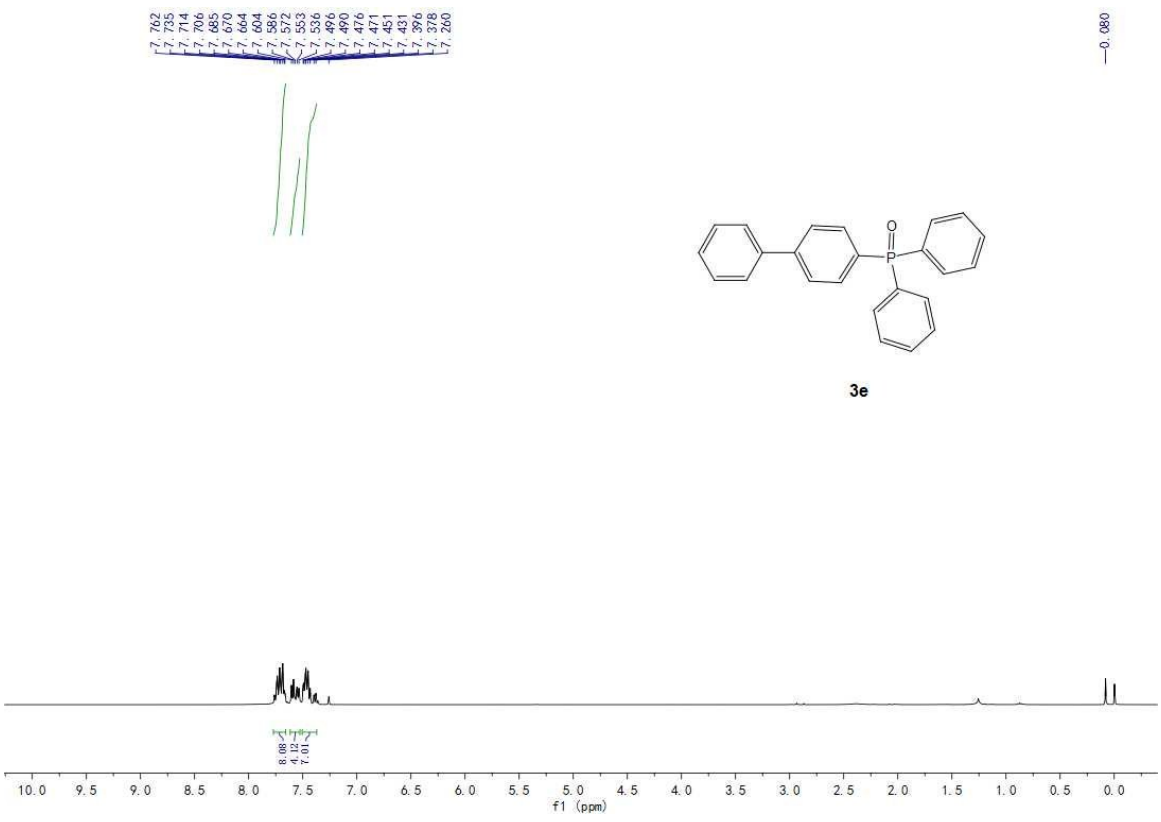
128.249
77.396
77.078
76.760
21.417
H3C
O
P
f1 (ppm)

3c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7.490
7.476
7.471
7.451
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7.378
7.260
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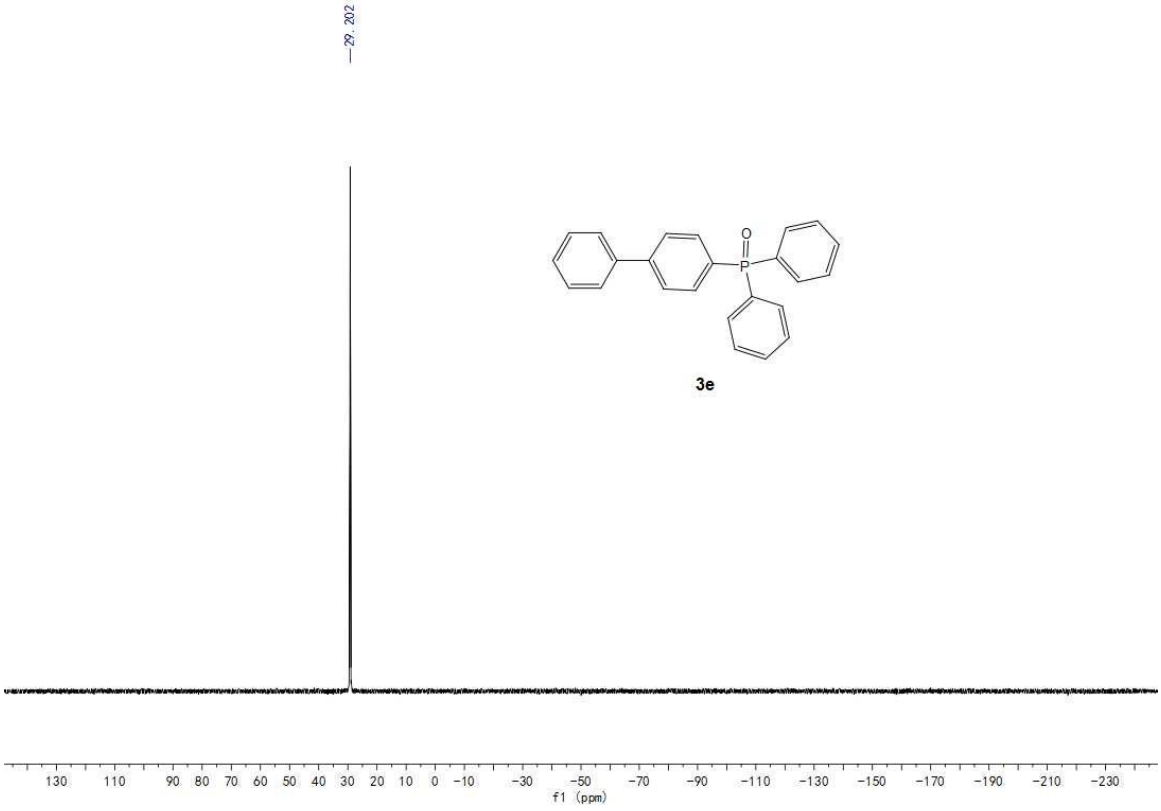
O
P
8.68
4.12
7.01
f1 (ppm)

3e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3e
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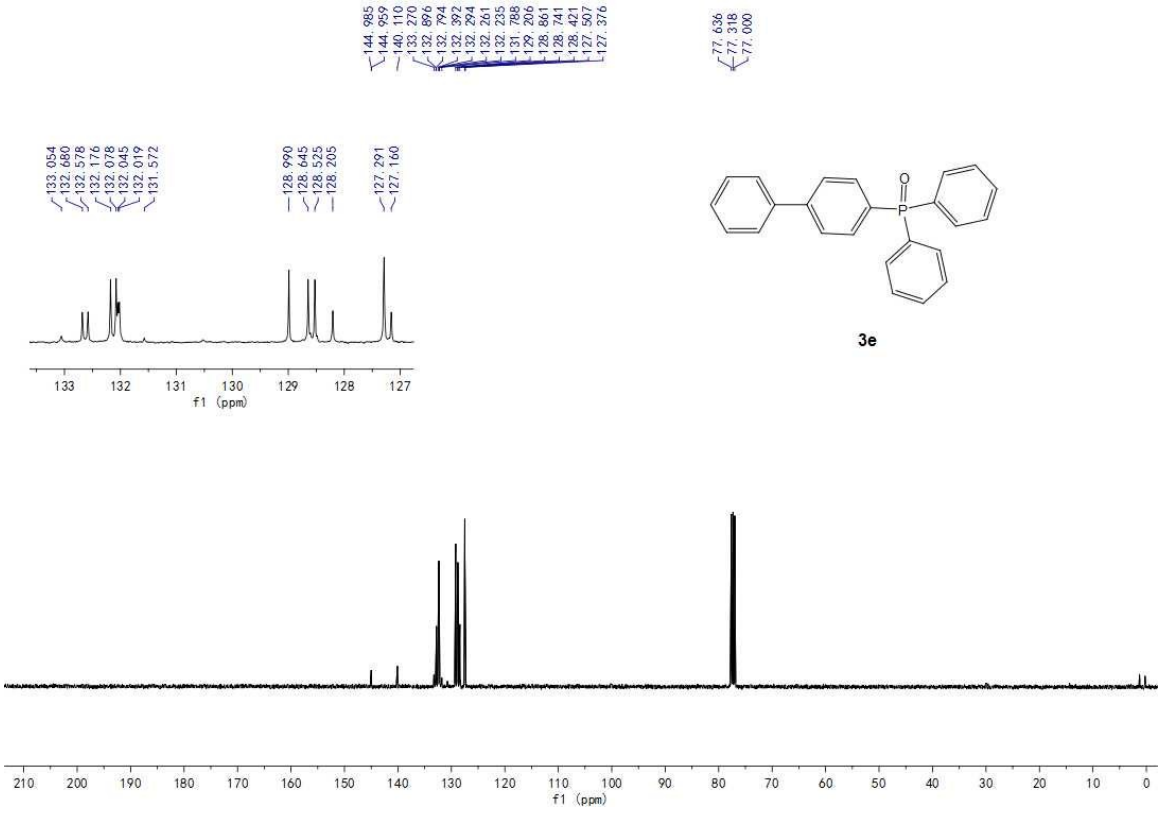
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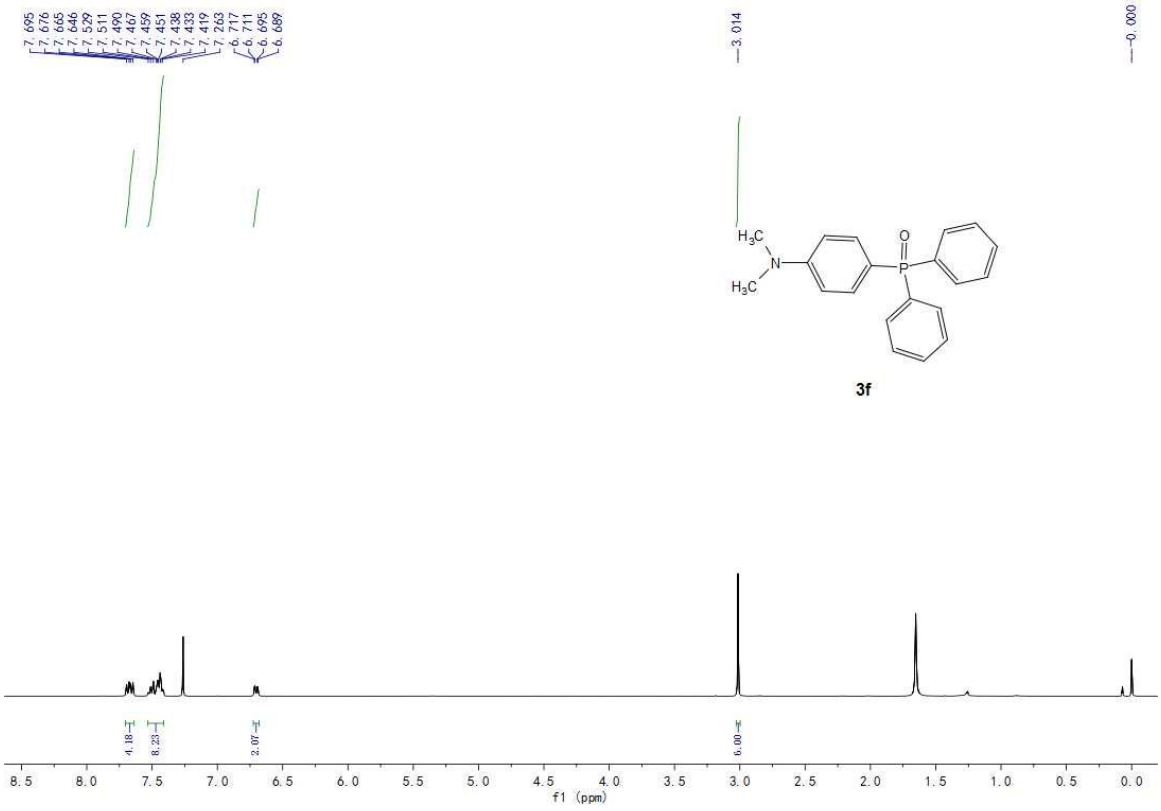
127.160
3e
f1 (ppm)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7.438
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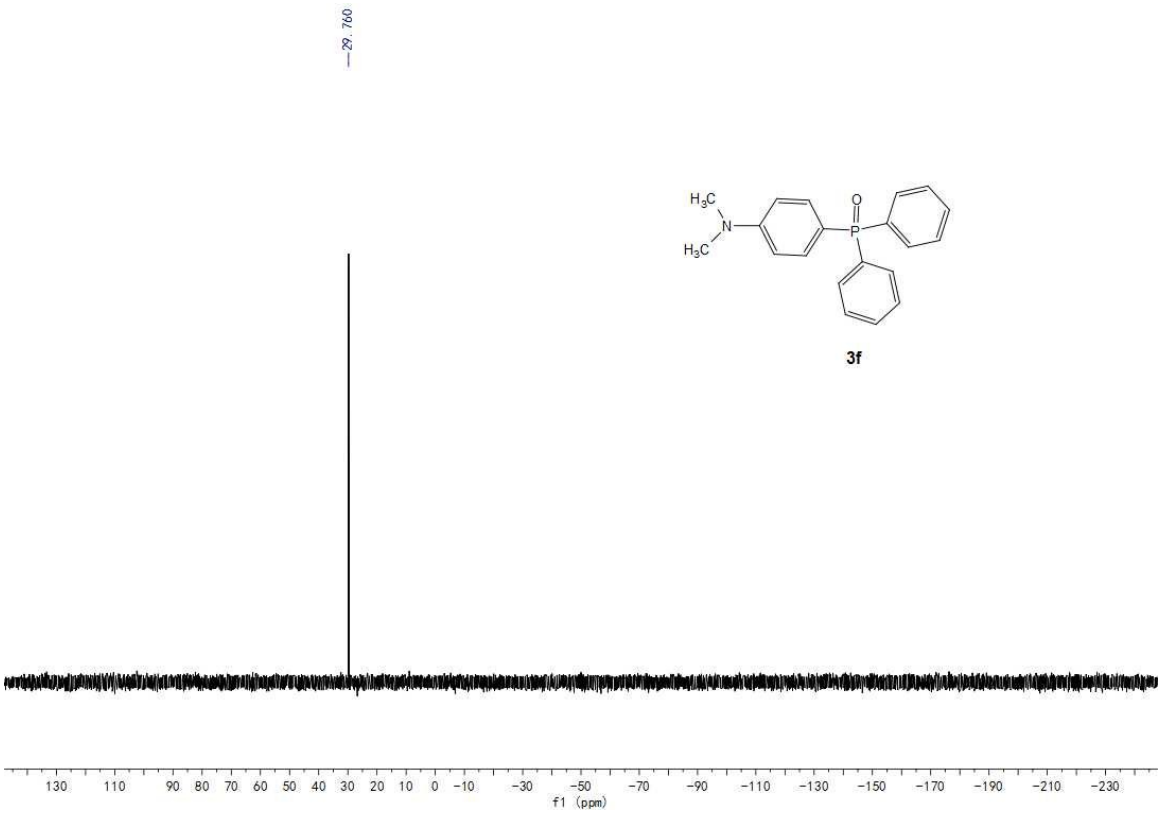
7.419
7.263
6.717
6.711
6.695
6.689
3.014
0.000
H3C
N
H3C
O
P
4.18
8.23
2.07
6.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3f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H3C
O
P
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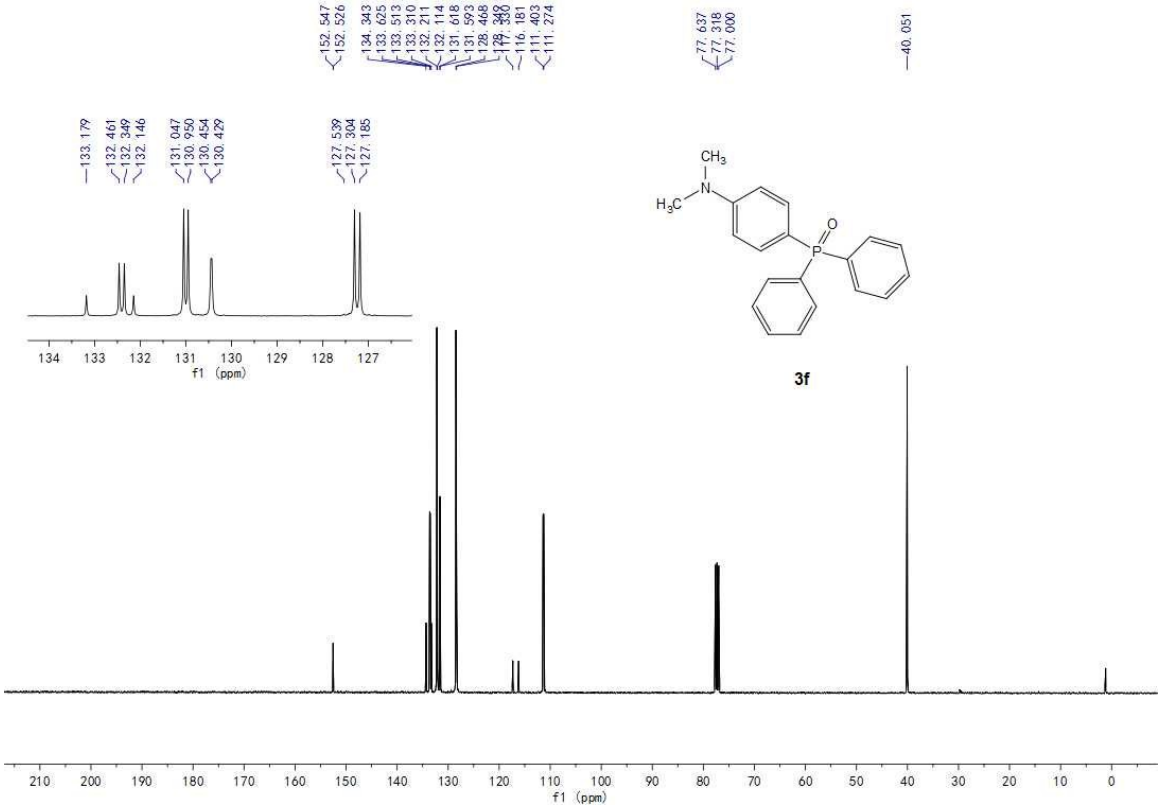
f1 (ppm)

3f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77.318
77.000
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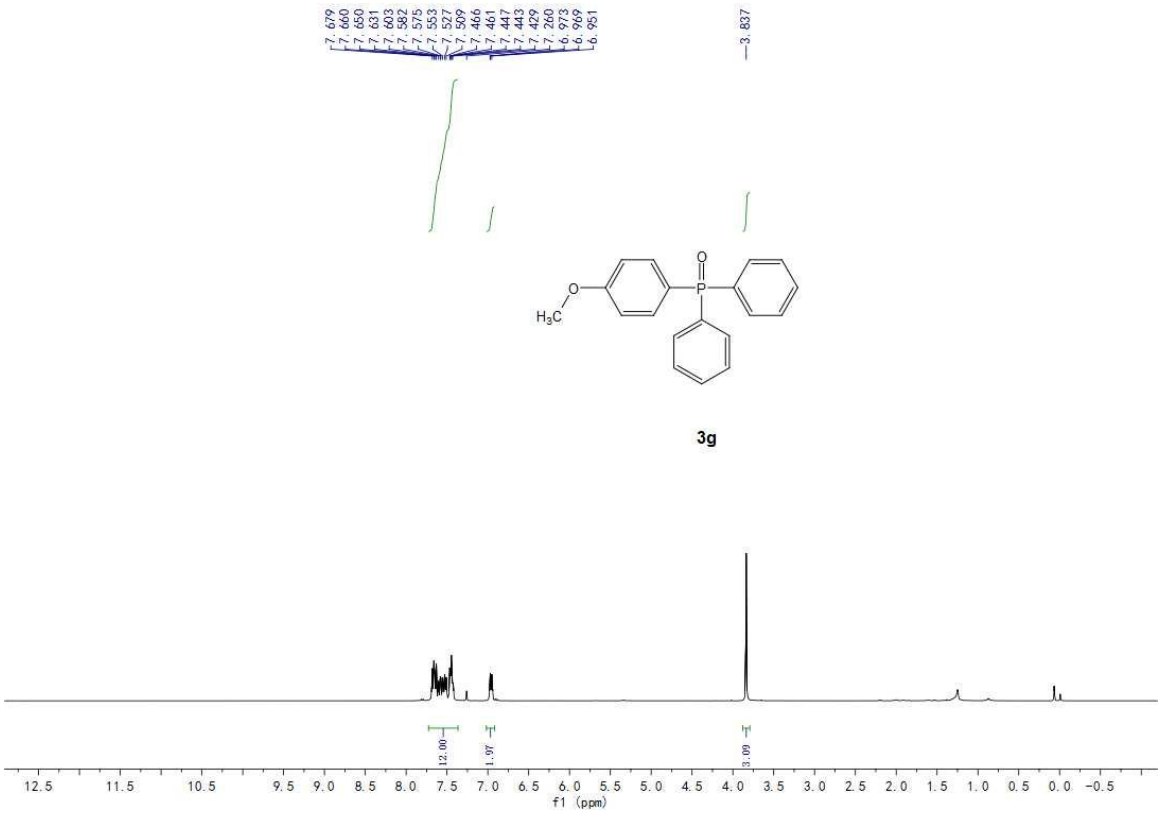
CH3
N
H3C
O
P
3f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7.447
7.443
7.429
7.260
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O
H3C
P
3g
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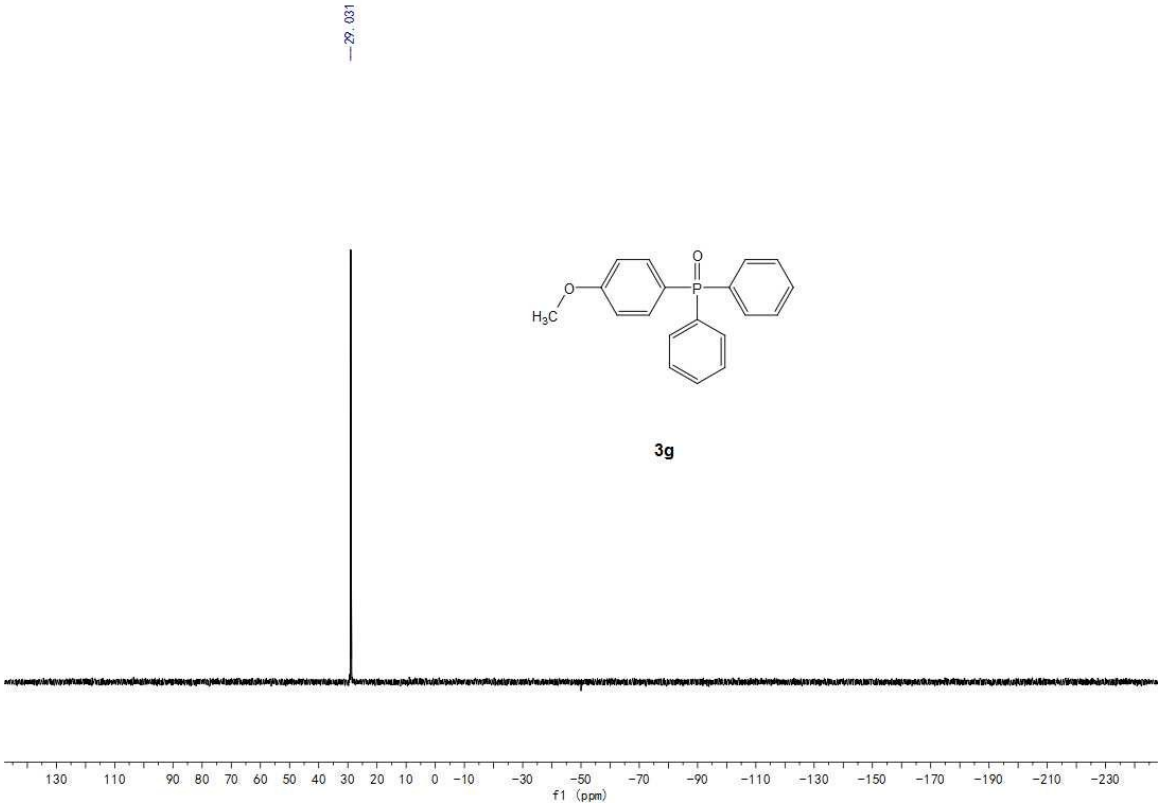
3.09
f1 (ppm)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3g
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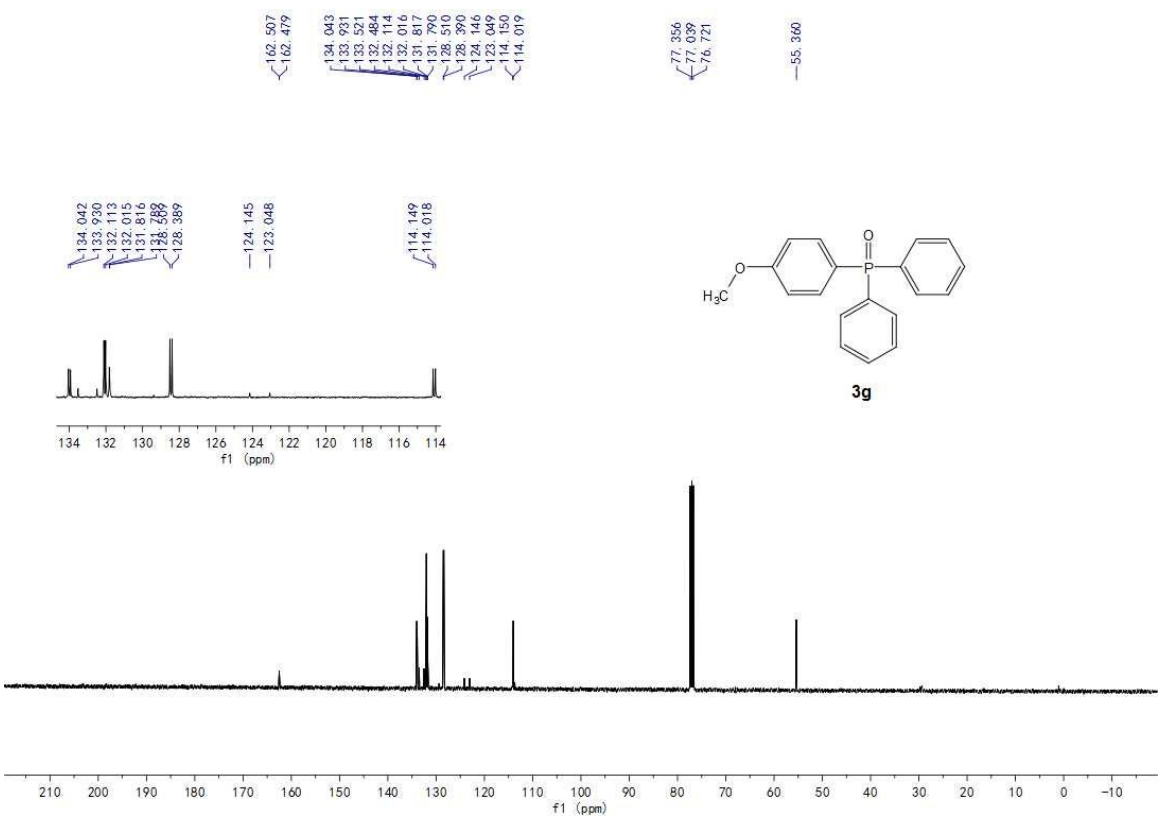
f1 (ppm)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114.150
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132.015
131.816
128.389
124.145
123.048
114.149
114.018
O
P
O
H3C
3g
134 132 130 128 126 124 122 120 118 116 114
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.587
8.566
8.026
8.005
7.898
7.878
7.707
7.689
7.678
7.659
7.566
7.548
7.531
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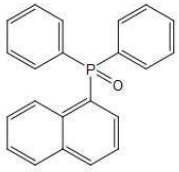
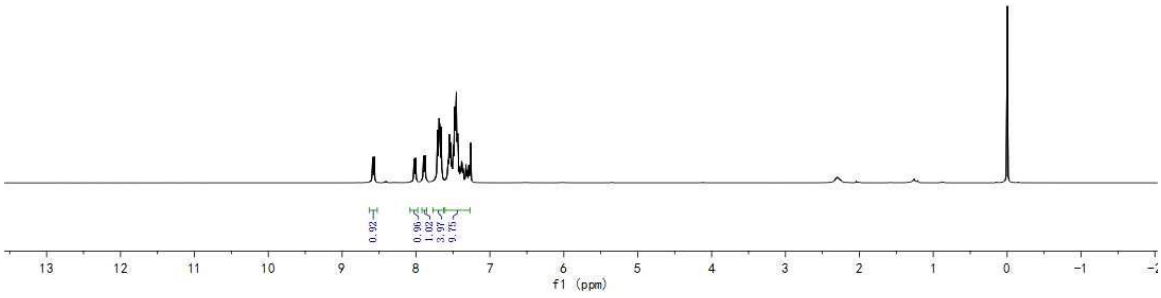

P
O

3i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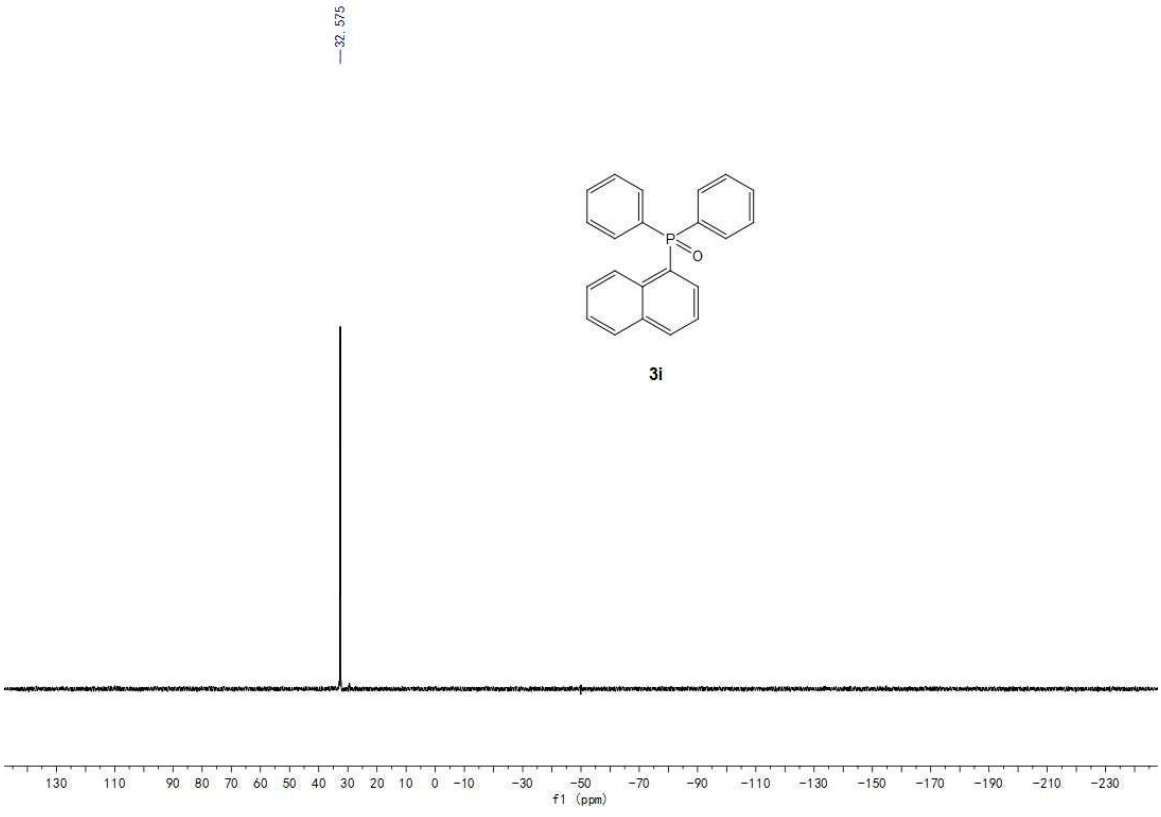
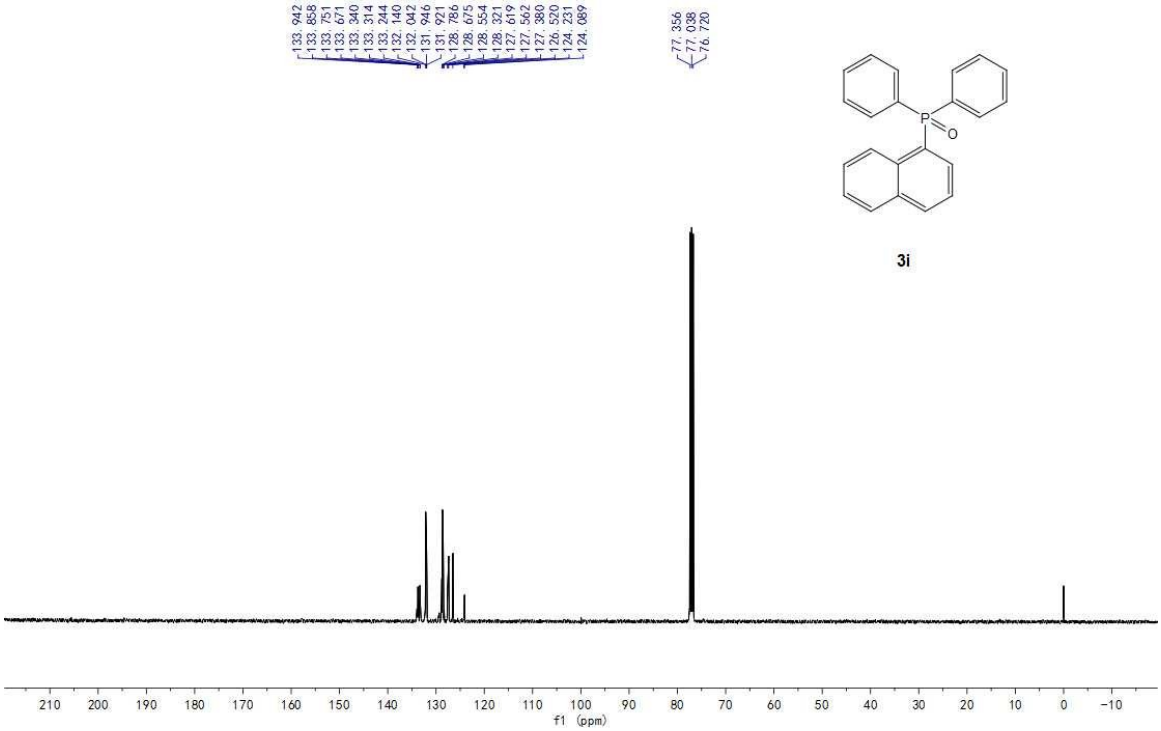
3.97
9.75
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
f1 (ppm)

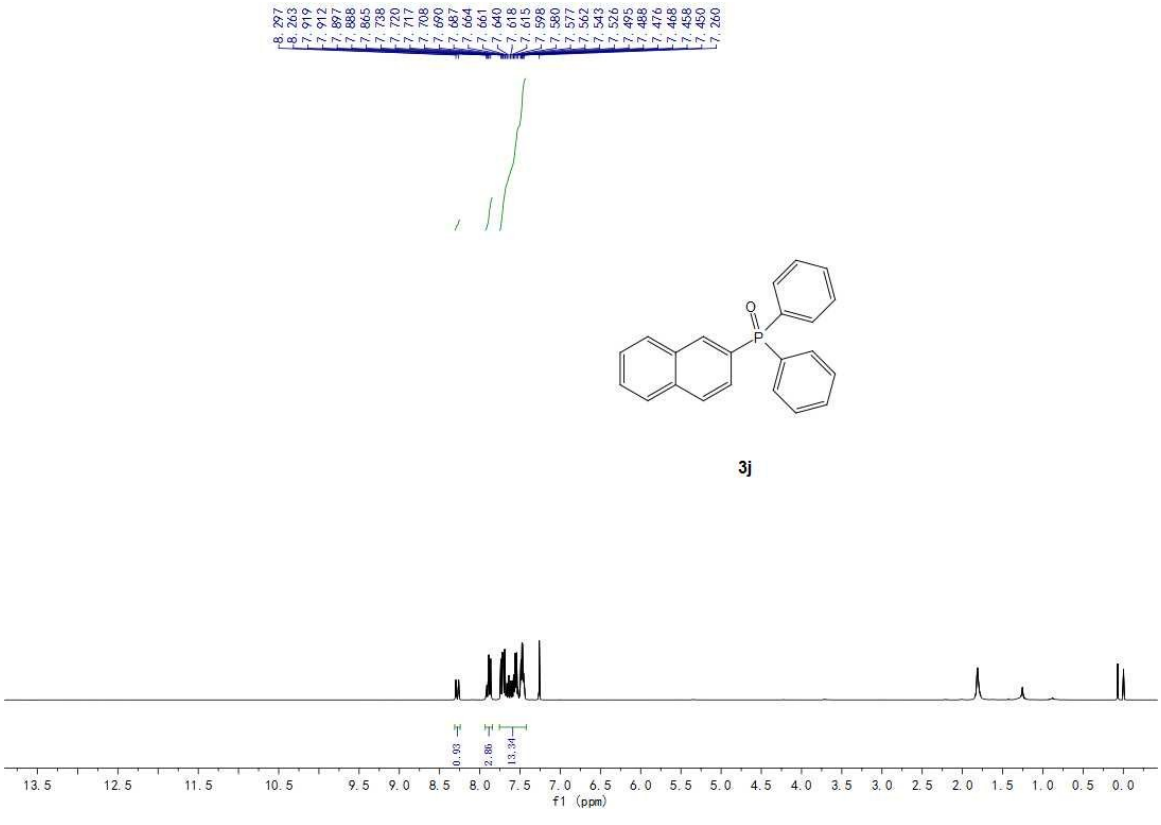

32.575
130
110
90
80
70
60
50
40
30
20
10
0
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
f1 (ppm)

P
O

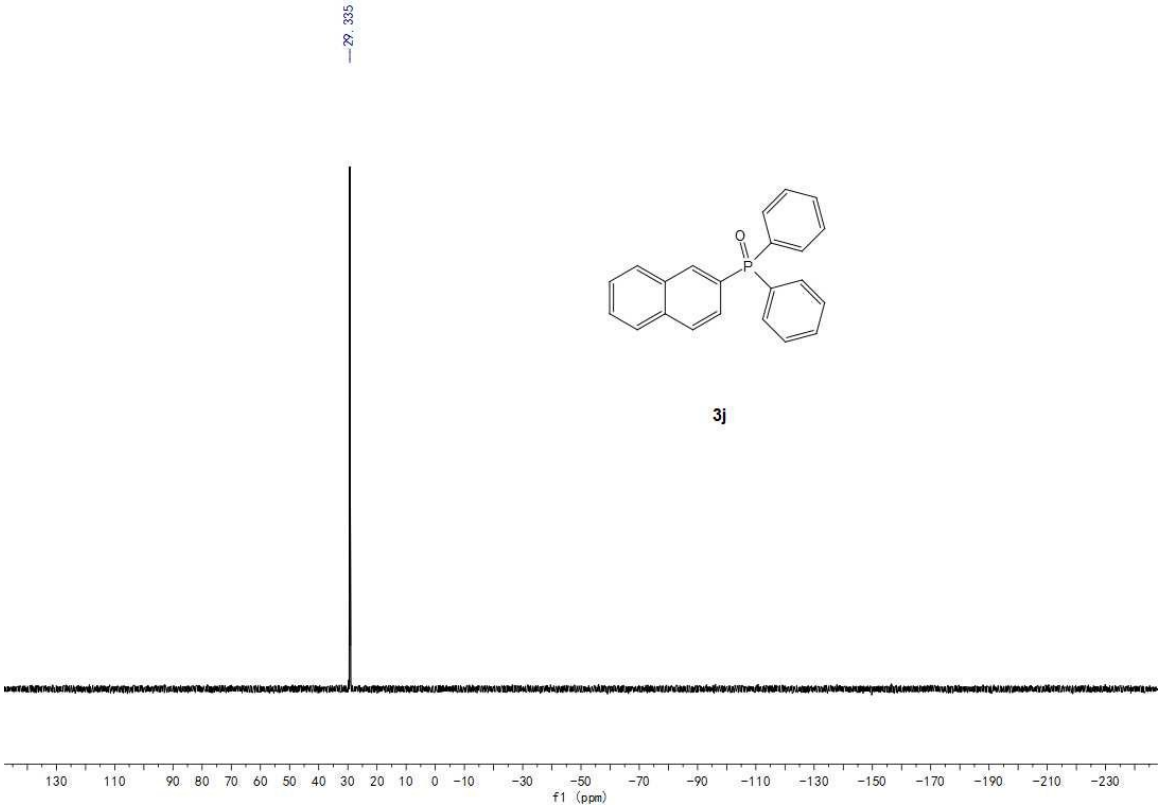
3i

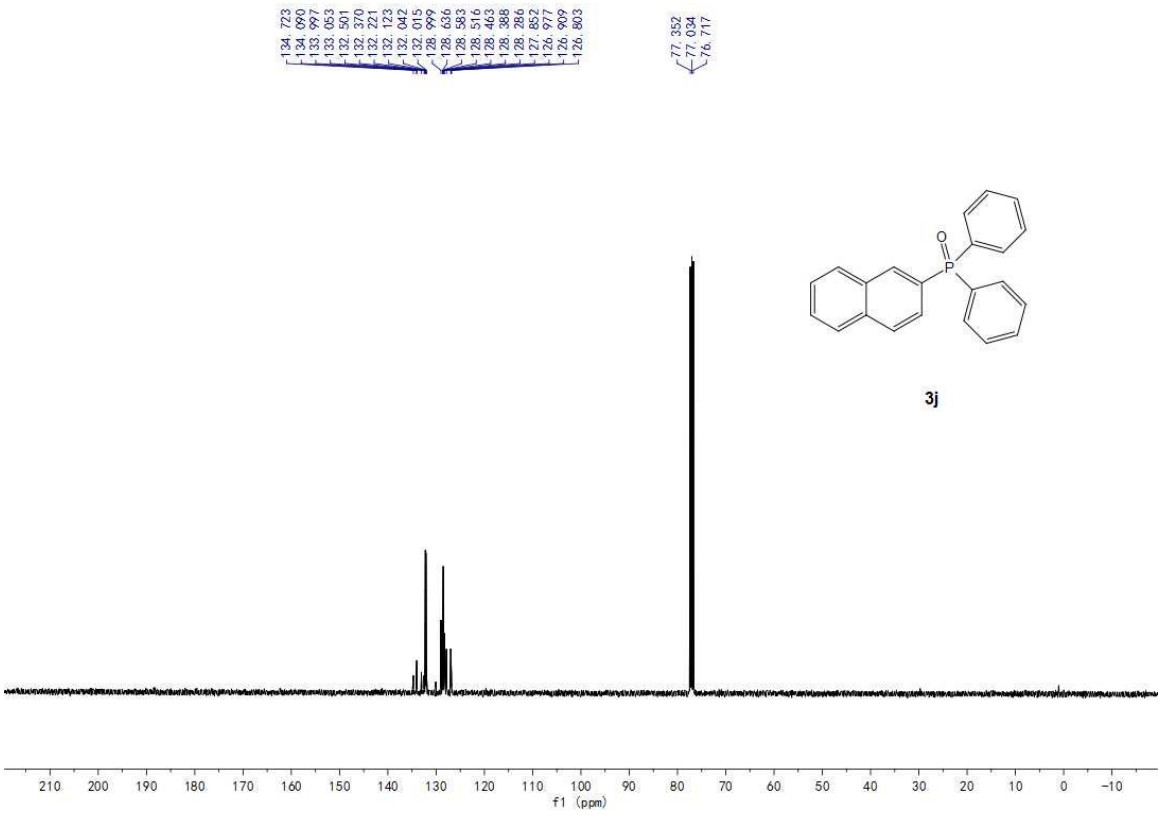

133.942
133.858
133.751
133.671
133.340
133.314
133.244
132.140
132.042
131.946
131.921
128.786
128.675
128.554
128.321
127.619
127.562
127.380
126.520
124.231
124.089
77.356
77.038
76.720
P
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3i

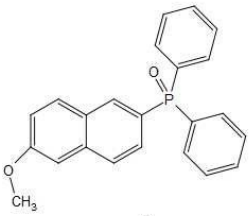

8.297
8.263
7.919
7.912
7.897
7.888
7.865
7.738
7.720
7.717
7.708
7.690
7.687
7.664
7.661
7.640
7.618
7.615
7.598
7.580
7.577
7.562
7.543
7.526
7.495
7.488
7.476
7.468
7.458
7.450
7.260
O
P
0.93
2.86
13.34
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3j

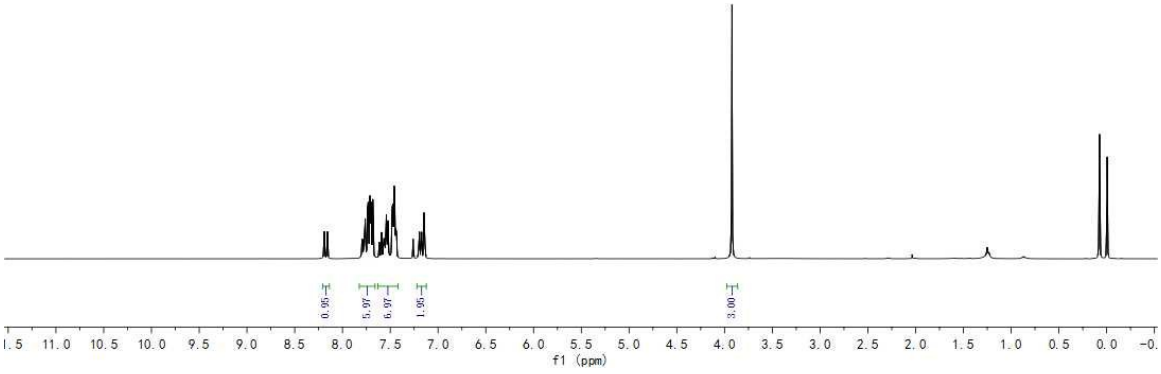

29.335
O
P
3j
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)


134.723
134.090
133.997
133.053
132.501
132.370
132.221
132.123
132.042
132.015
128.999
128.636
128.583
128.516
128.463
128.388
128.286
127.852
126.977
126.909
126.803
77.352
77.034
76.717
O
P
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

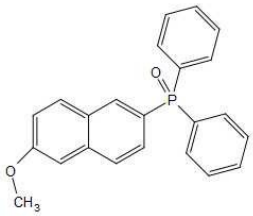

8.191
8.157
7.789
7.775
7.768
7.761
7.738
7.731
7.712
7.701
7.682
7.591
7.565
7.541
7.524
7.479
7.473
7.460
7.454
7.442
7.436
7.260
7.199
7.194
7.177
7.171
7.146
3.922


O
P
O
CH3

3k

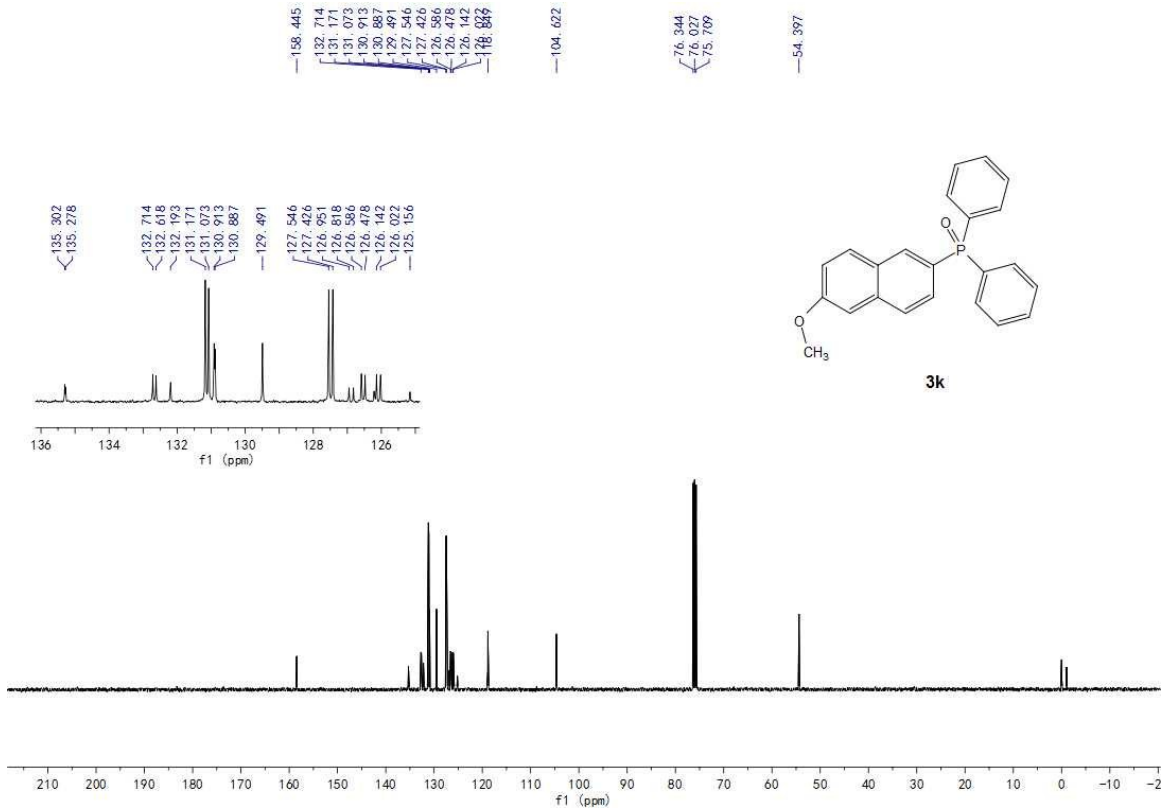

0.95
5.97
6.97
1.95
3.00
f1 (ppm)

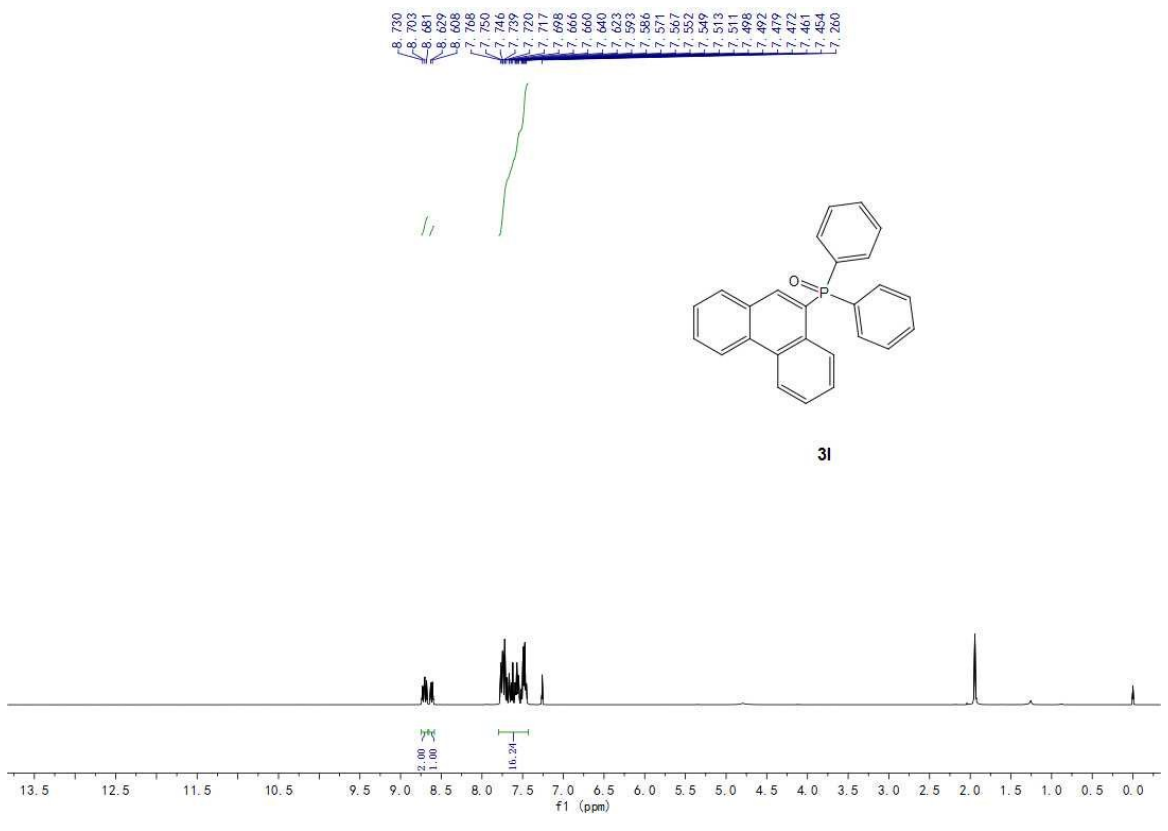
29.595

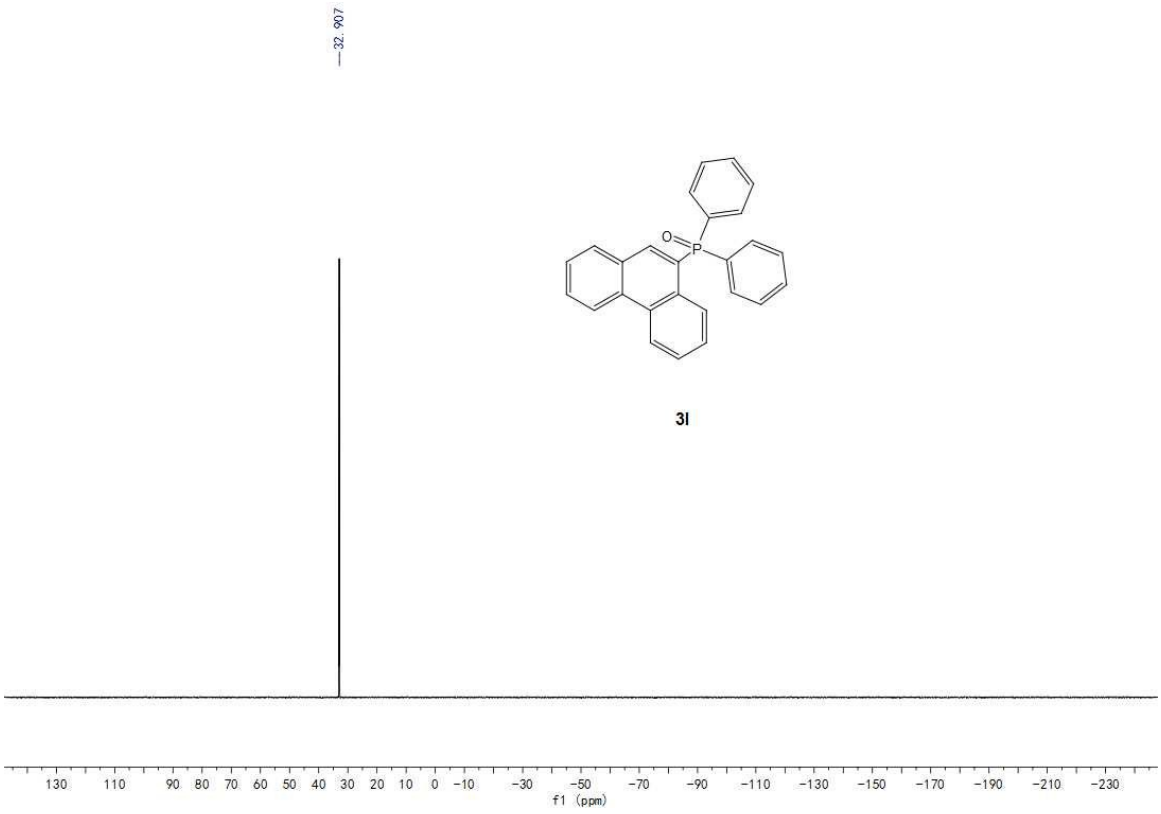

O
P
O
CH3

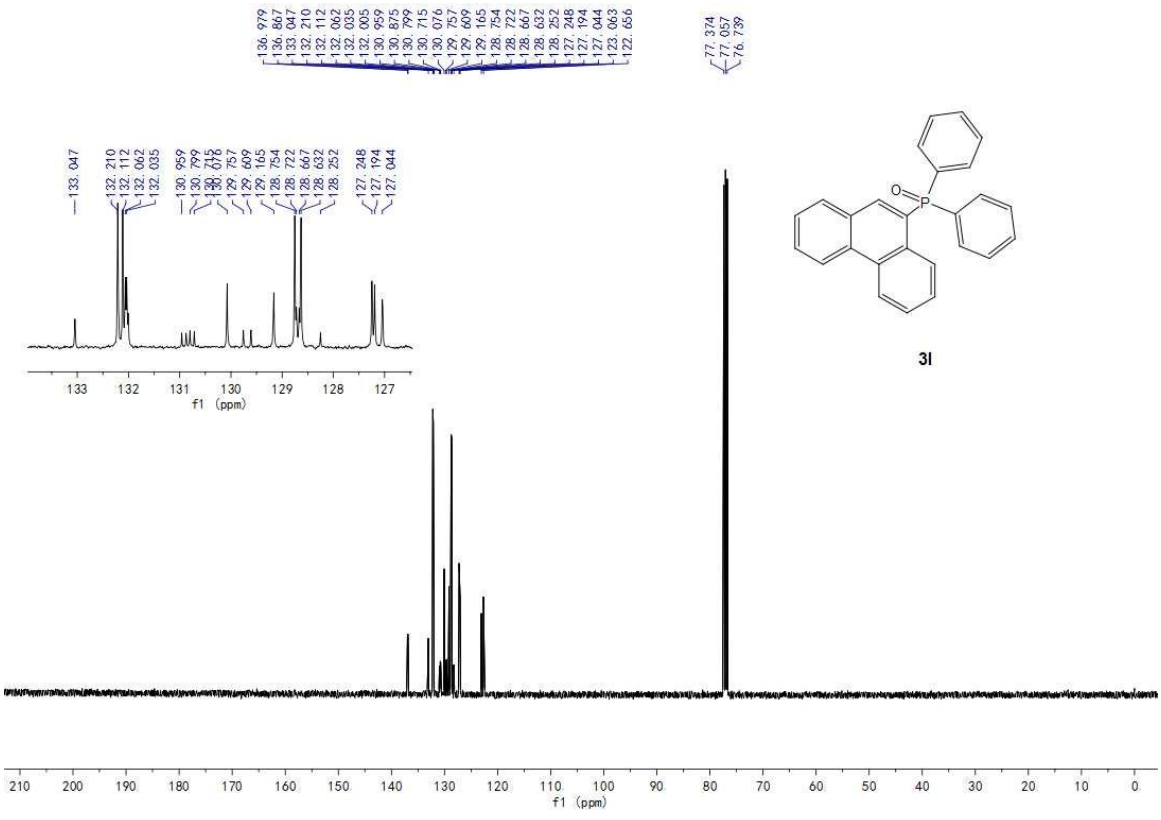
3k

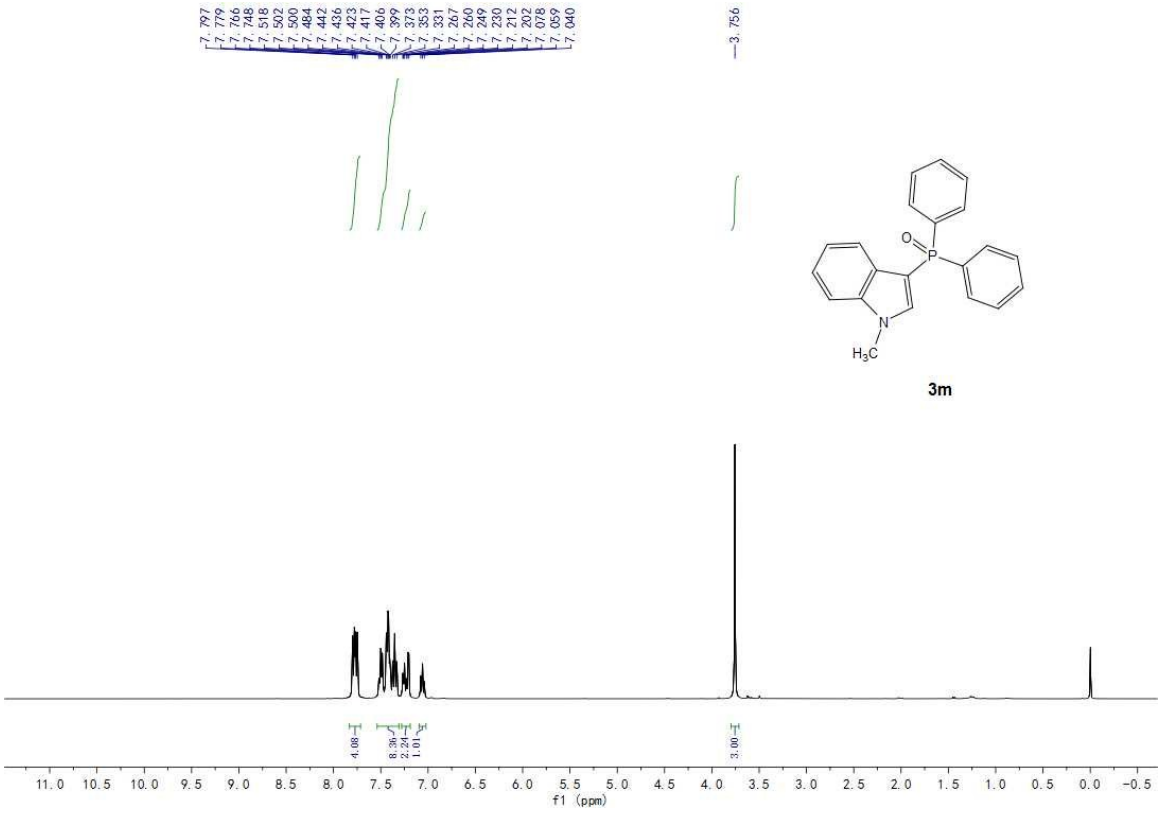
f1 (ppm)

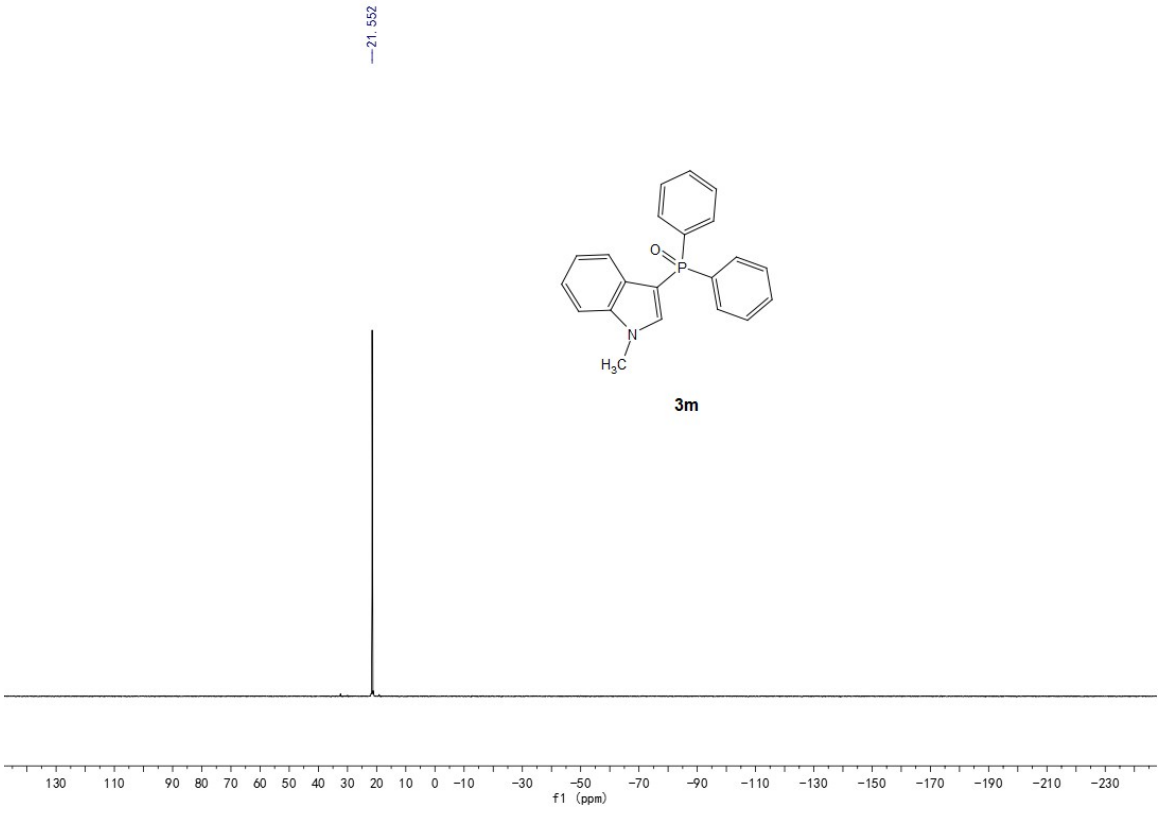

158.445
132.714
131.171
131.073
130.913
130.887
129.491
127.546
127.426
126.586
126.478
126.142
104.622
76.344
76.027
75.709
54.397
135.302
135.278
132.714
132.618
132.193
131.171
131.073
130.913
130.887
129.491
127.546
127.426
126.951
126.818
126.586
126.478
126.142
126.022
125.156
136
134
132
130
128
126
f1 (ppm)
O
P
O
CH3
3k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−2
f1 (ppm)

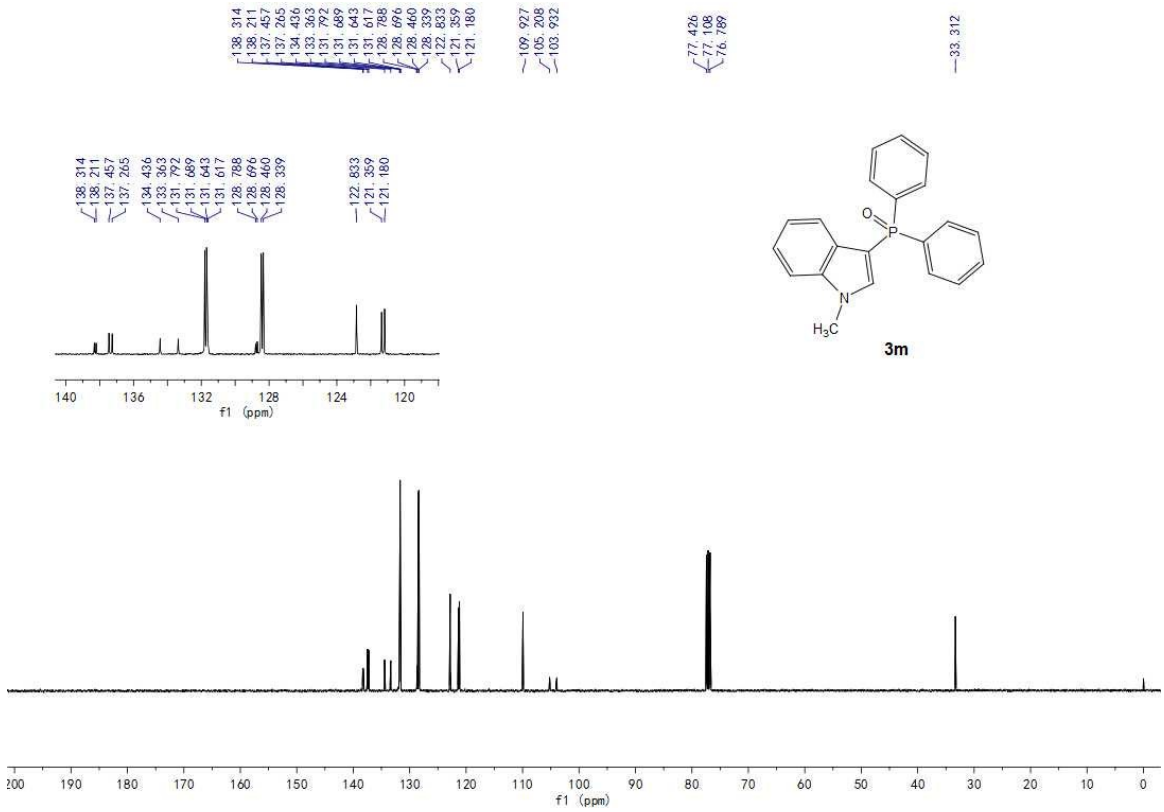

8.730
8.703
8.681
8.629
8.608
7.768
7.750
7.746
7.739
7.720
7.717
7.698
7.666
7.660
7.640
7.623
7.593
7.586
7.571
7.567
7.552
7.549
7.513
7.511
7.498
7.492
7.479
7.472
7.461
7.454
7.260
O
P
3l
2.00
1.00
10.39
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

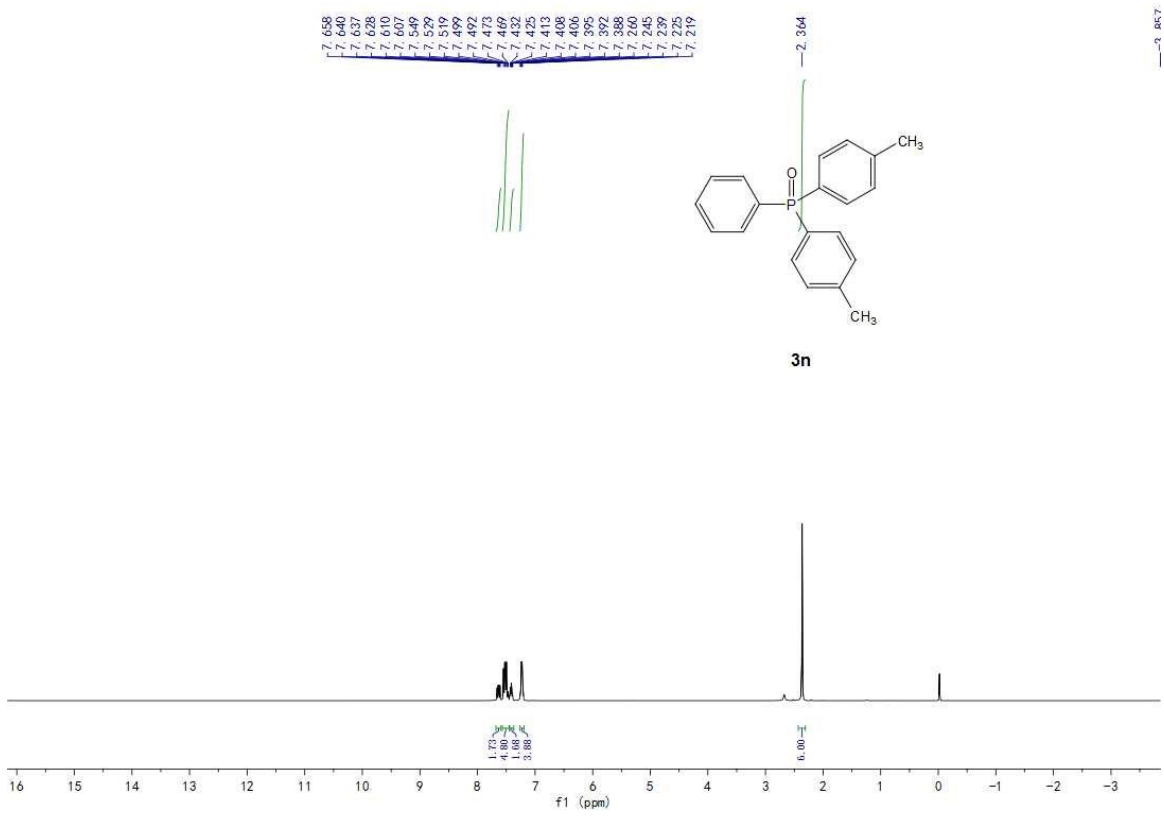

32.907
O
P
3l
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

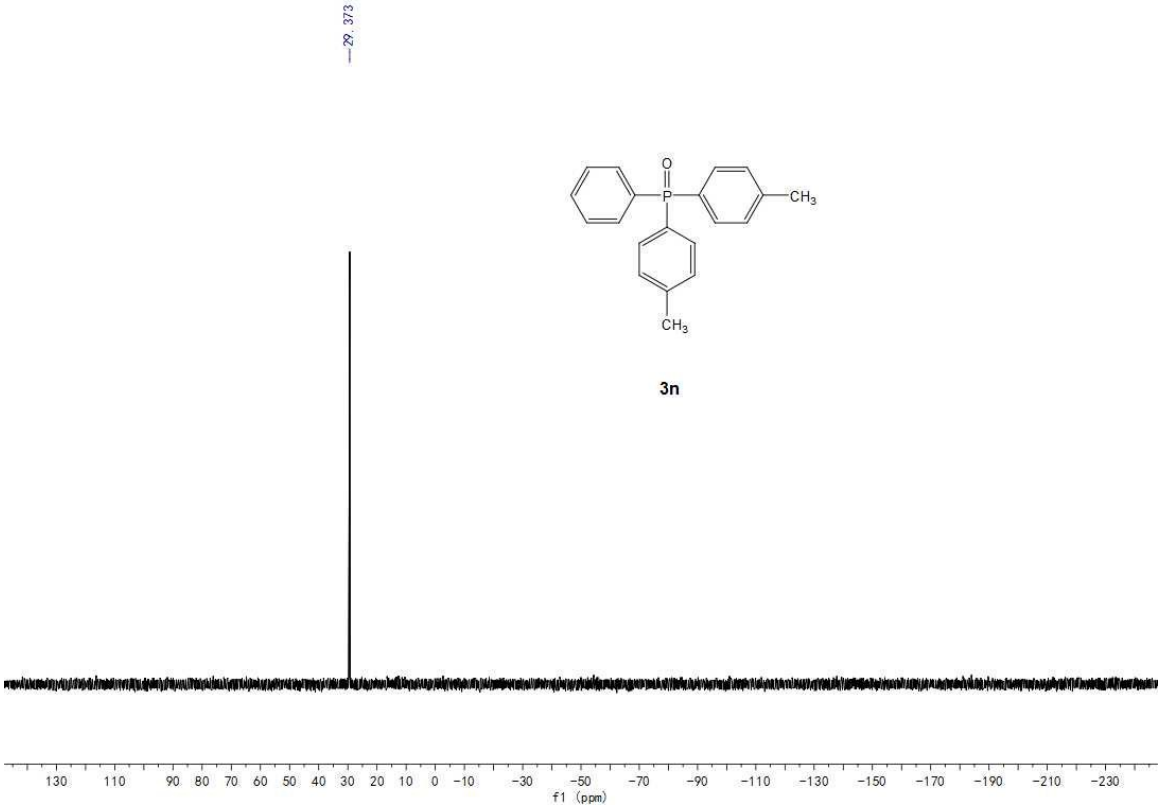

136.979
136.867
133.047
132.210
132.112
132.062
132.035
132.005
130.959
130.875
130.799
130.715
130.076
129.757
129.609
129.165
128.754
128.722
128.667
128.632
128.252
127.248
127.194
127.044
123.063
122.656
77.374
77.057
76.739
133.047
132.210
132.112
132.062
132.035
130.959
130.799
130.715
130.076
129.757
129.609
129.165
128.754
128.722
128.667
128.632
128.252
127.248
127.194
127.044
O
P
3l
133 132 131 130 129 128 127
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.797
7.779
7.766
7.748
7.518
7.502
7.500
7.484
7.442
7.436
7.423
7.417
7.406
7.399
7.373
7.353
7.331
7.267
7.260
7.249
7.230
7.212
7.202
7.078
7.059
7.040
3.756
O
P
N
H3C
3m
4.08
8.36
2.24
1.01
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

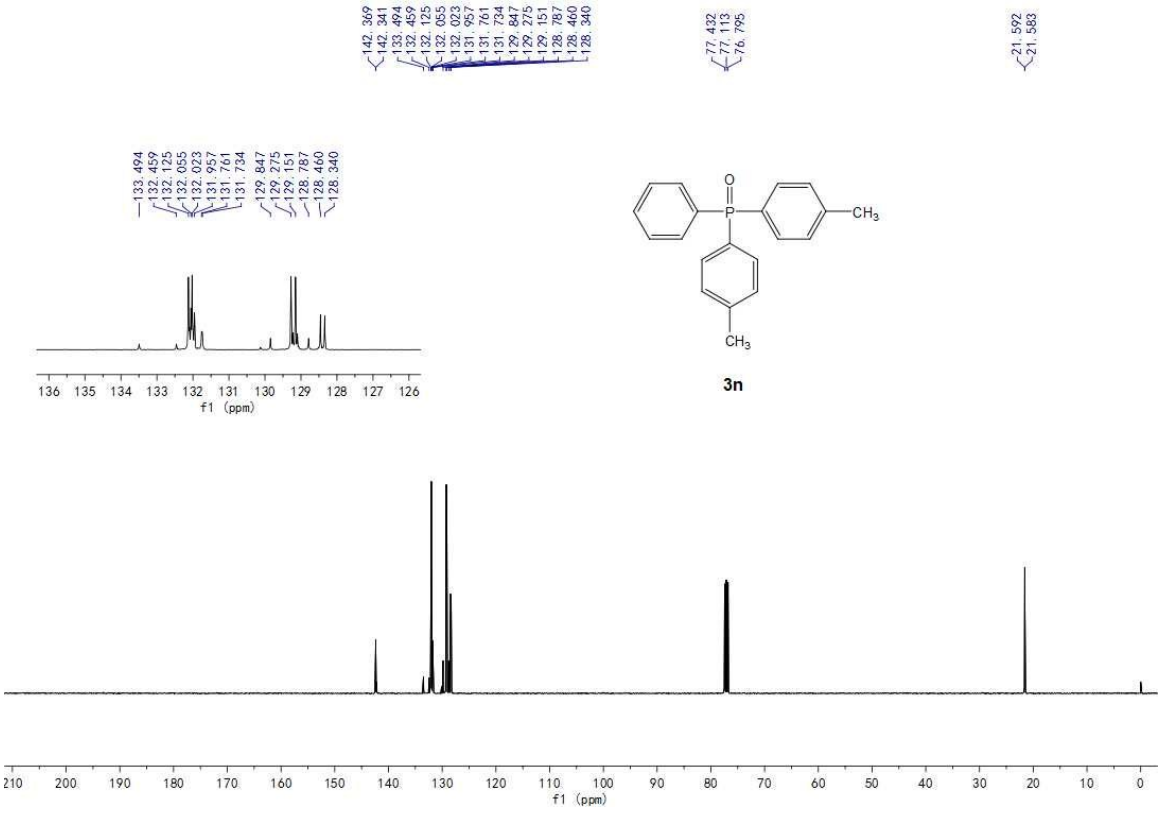

21.552
O
P
N
H3C
3m
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)


138.314
138.211
137.457
137.265
134.436
133.363
131.792
131.689
131.643
131.617
128.788
128.696
128.460
128.339
122.833
121.359
121.180
109.927
105.208
103.932
77.426
77.108
76.789
33.312
O
P
N
H3C
3m
f1 (ppm)


7.658
7.640
7.637
7.628
7.610
7.607
7.549
7.529
7.519
7.499
7.492
7.473
7.469
7.432
7.425
7.413
7.408
7.406
7.395
7.392
7.388
7.260
7.245
7.239
7.225
7.219
2.364
CH3
O
P
CH3
3n
1.73
4.80
1.68
3.88
6.00
f1 (ppm)

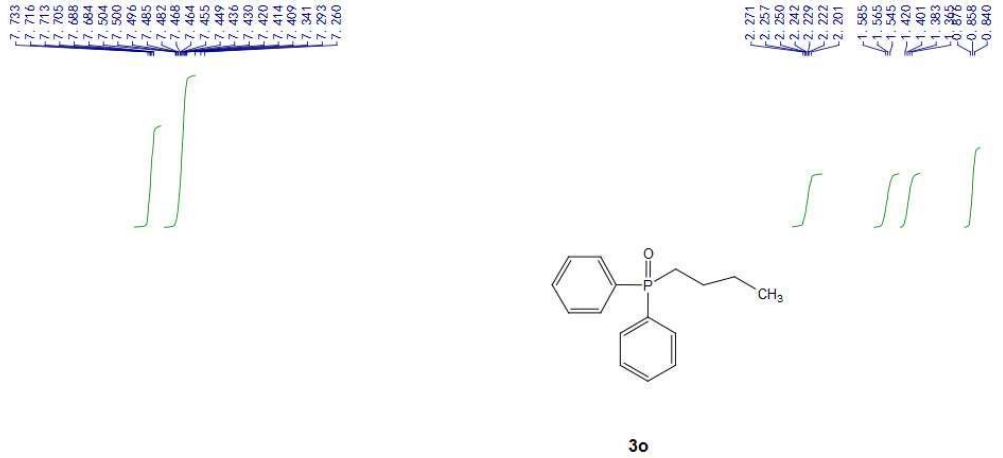

29.373
f1 (ppm)

3n

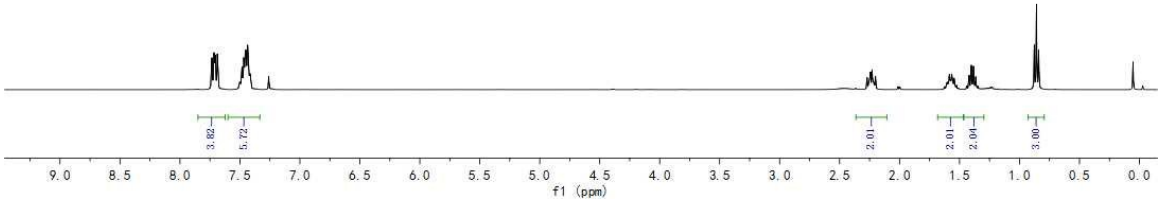

142.369
142.341
133.494
132.459
132.125
132.055
132.023
131.957
131.761
131.734
129.847
129.275
129.151
128.787
128.460
128.340
77.432
77.113
76.795
21.592
21.583
f1 (ppm)

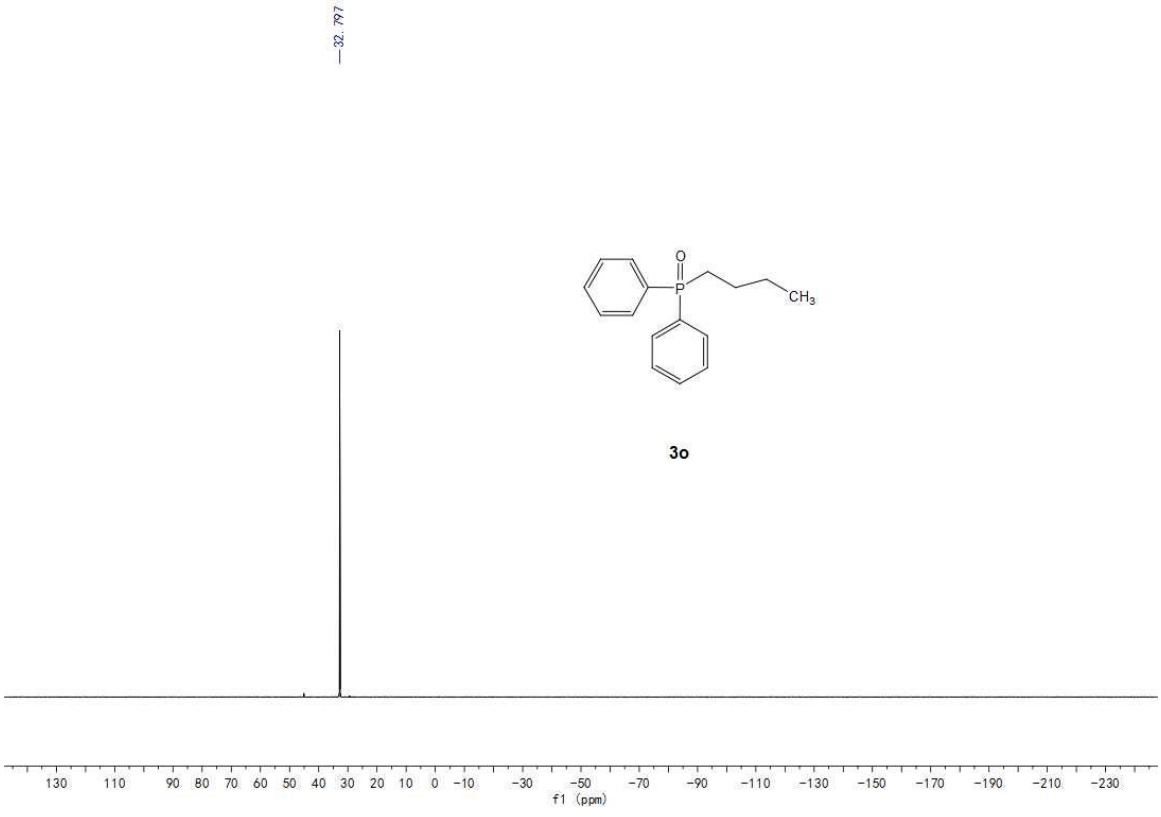
133.494
132.459
132.125
132.055
132.023
131.957
131.761
131.734
129.847
129.275
129.151
128.787
128.460
128.340
f1 (ppm)

3n

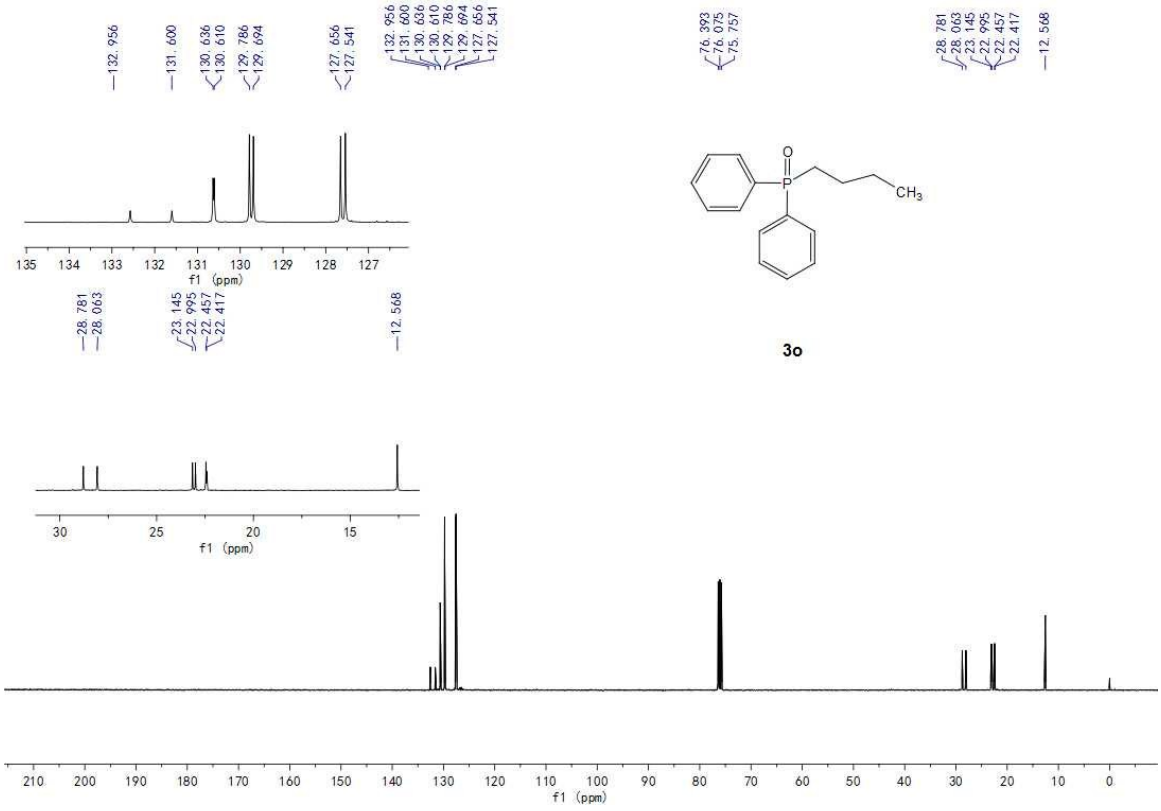

7.733
7.716
7.713
7.705
7.688
7.684
7.504
7.500
7.496
7.485
7.482
7.468
7.464
7.455
7.449
7.436
7.430
7.420
7.414
7.409
7.341
7.293
7.260
2.271
2.257
2.250
2.242
2.229
2.222
2.201
1.585
1.565
1.545
1.420
1.401
1.383
0.858
0.840
O
P
CH3

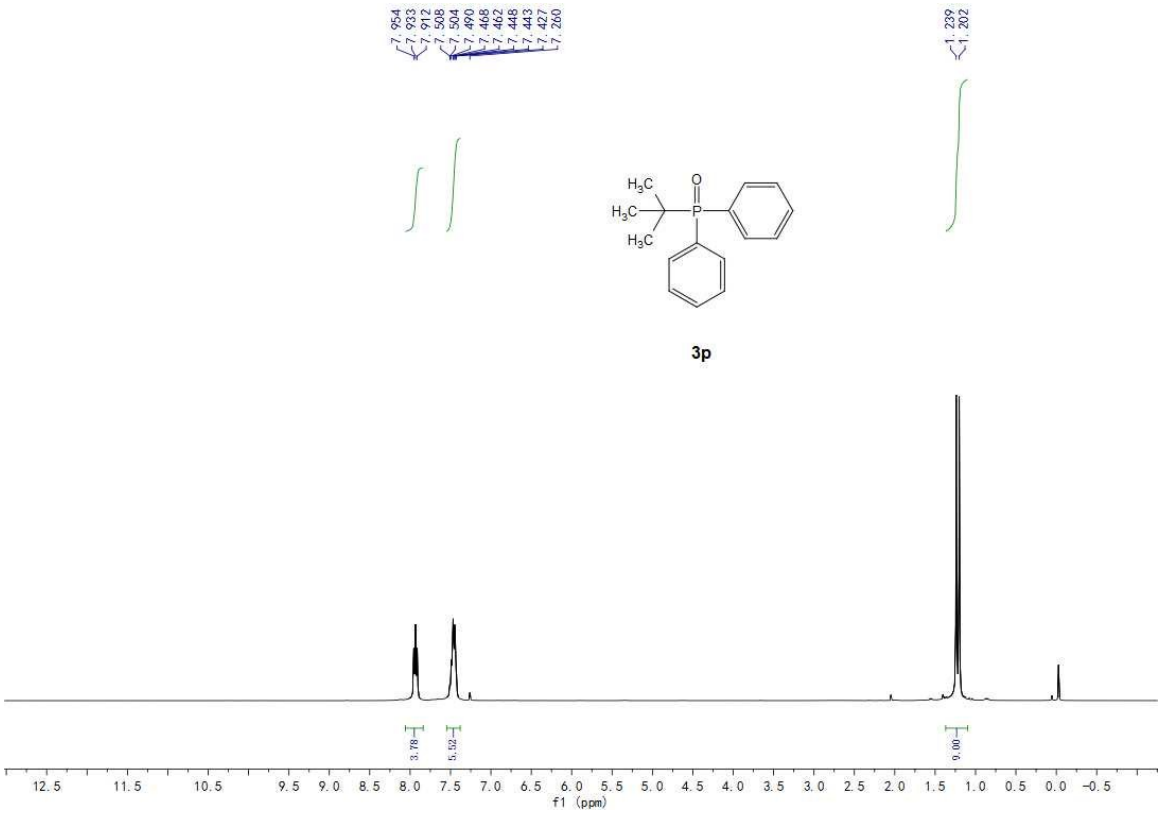
3o

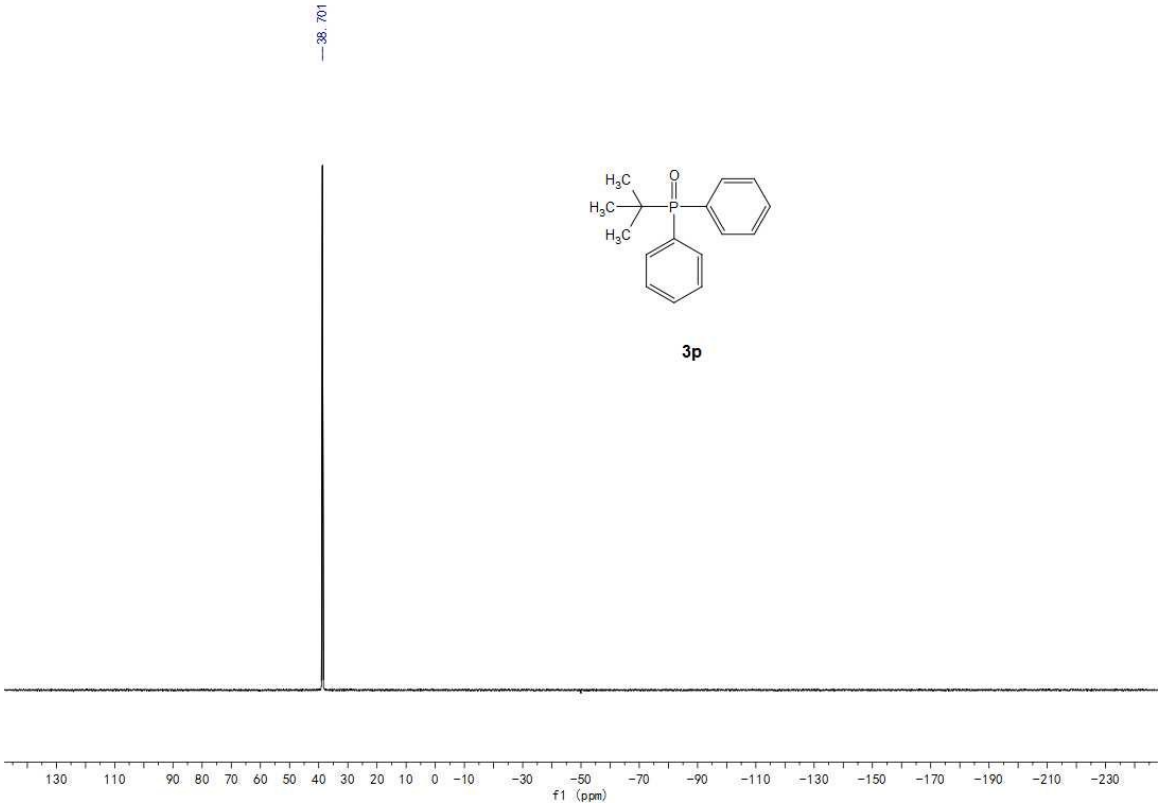

3.82
5.72
2.01
2.01
2.04
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

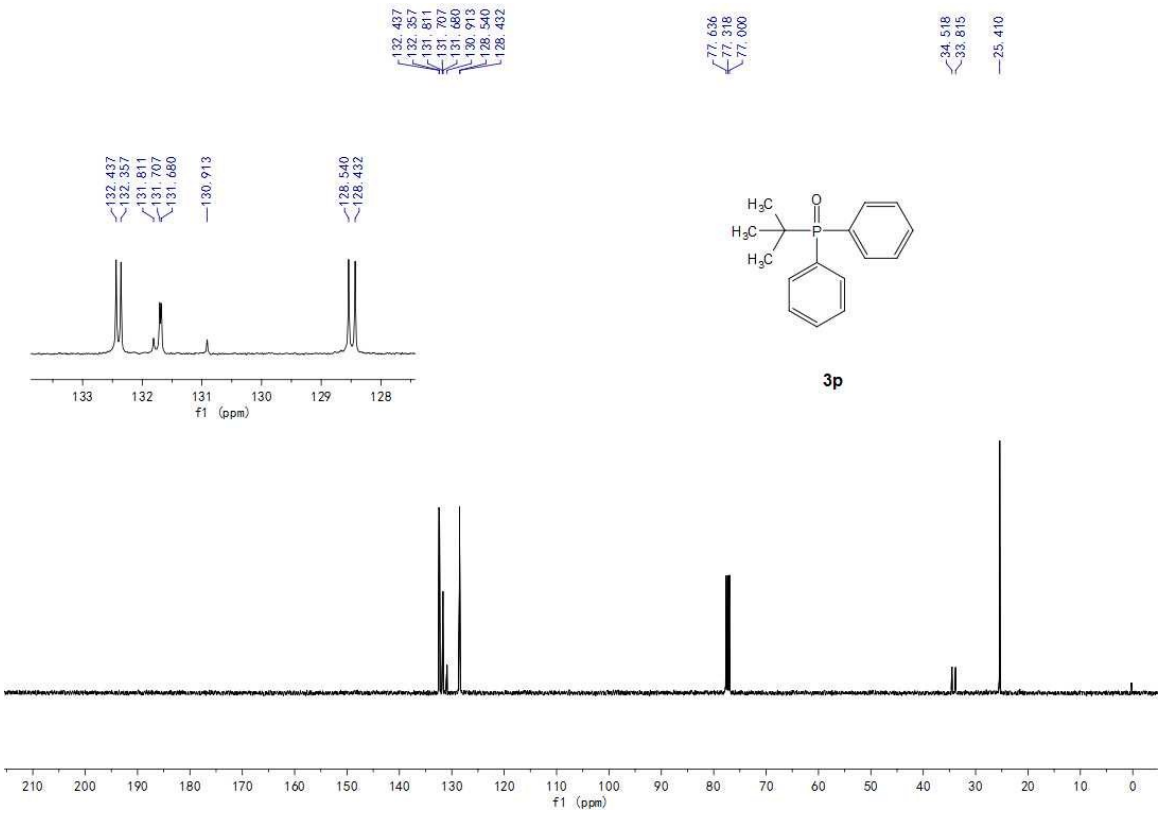

32.797
O
P
CH3
130
110
90
80
70
60
50
40
30
20
10
0
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
f1 (ppm)

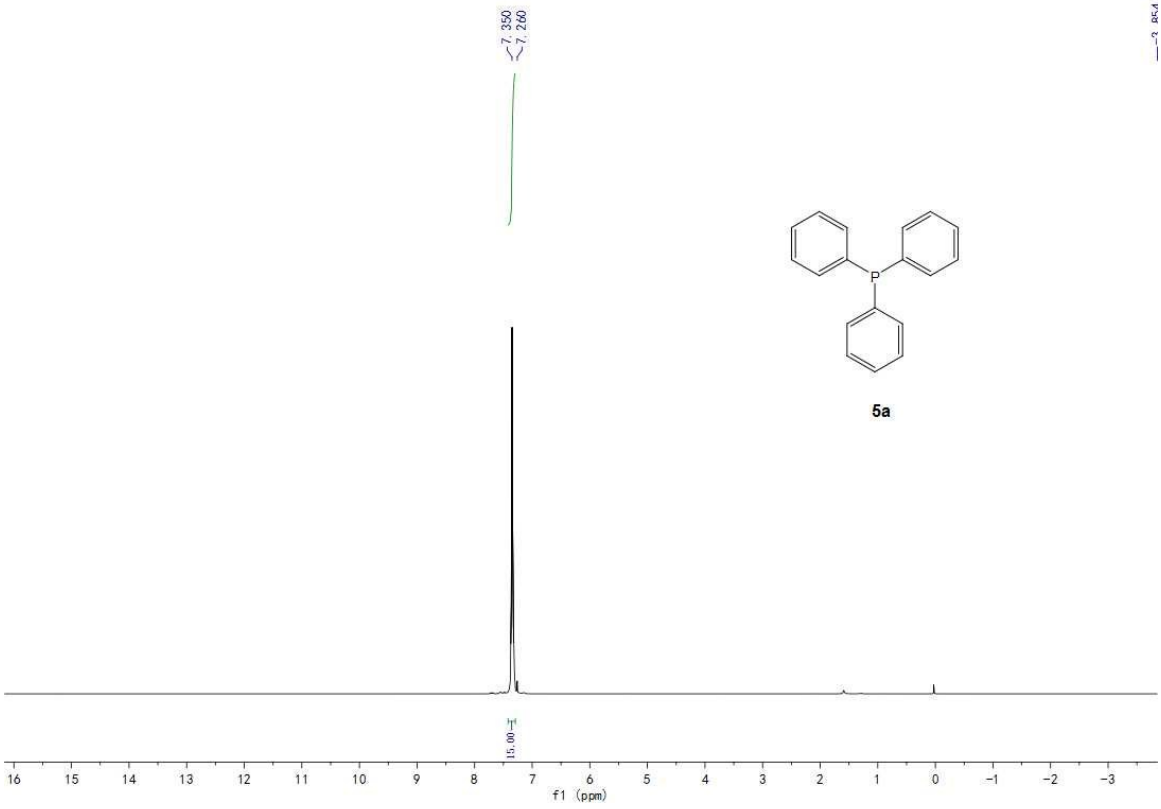
3o


132.956
131.600
130.636
130.610
129.786
129.694
127.656
127.541
76.393
76.075
75.757
28.781
28.063
23.145
22.995
22.457
22.417
12.568
O
P
CH3
3o
f1 (ppm)

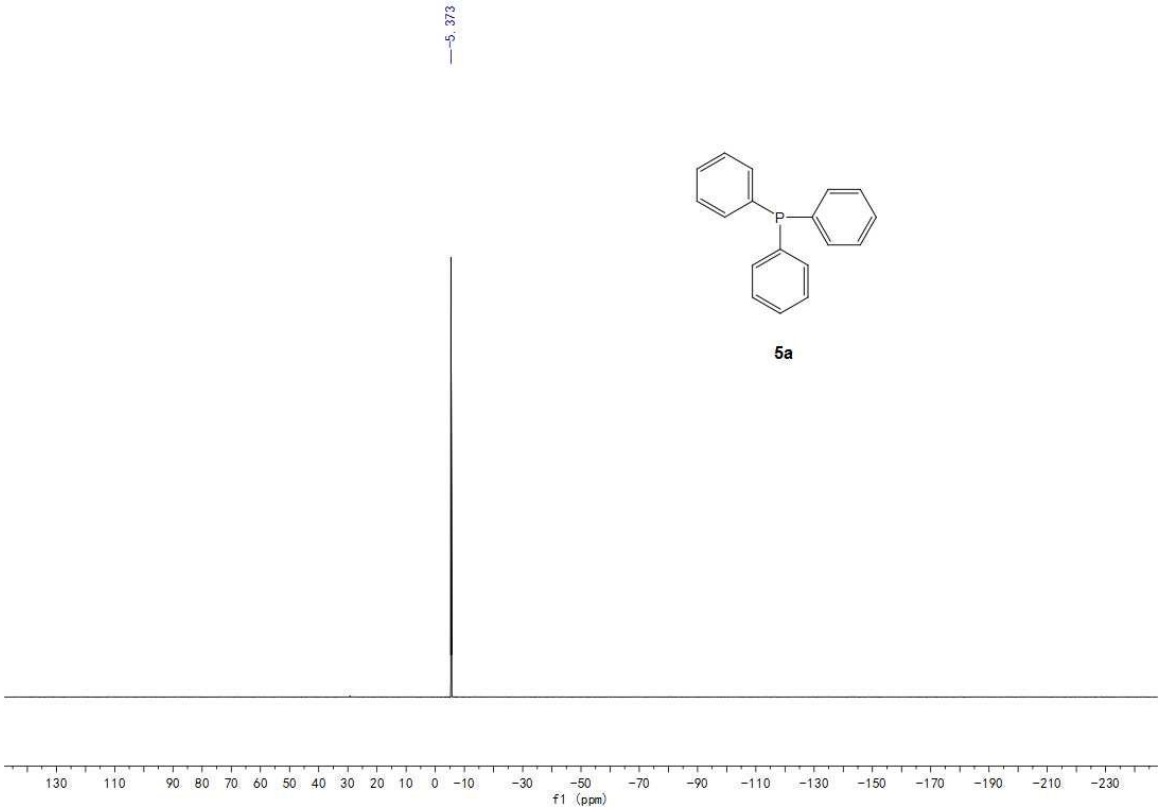

7.954
7.933
7.912
7.508
7.504
7.490
7.468
7.462
7.448
7.443
7.427
7.260
1.239
1.202
H3C
H3C
H3C
O
P
3p
3.78
5.52
9.00
f1 (ppm)


38.701
3p
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

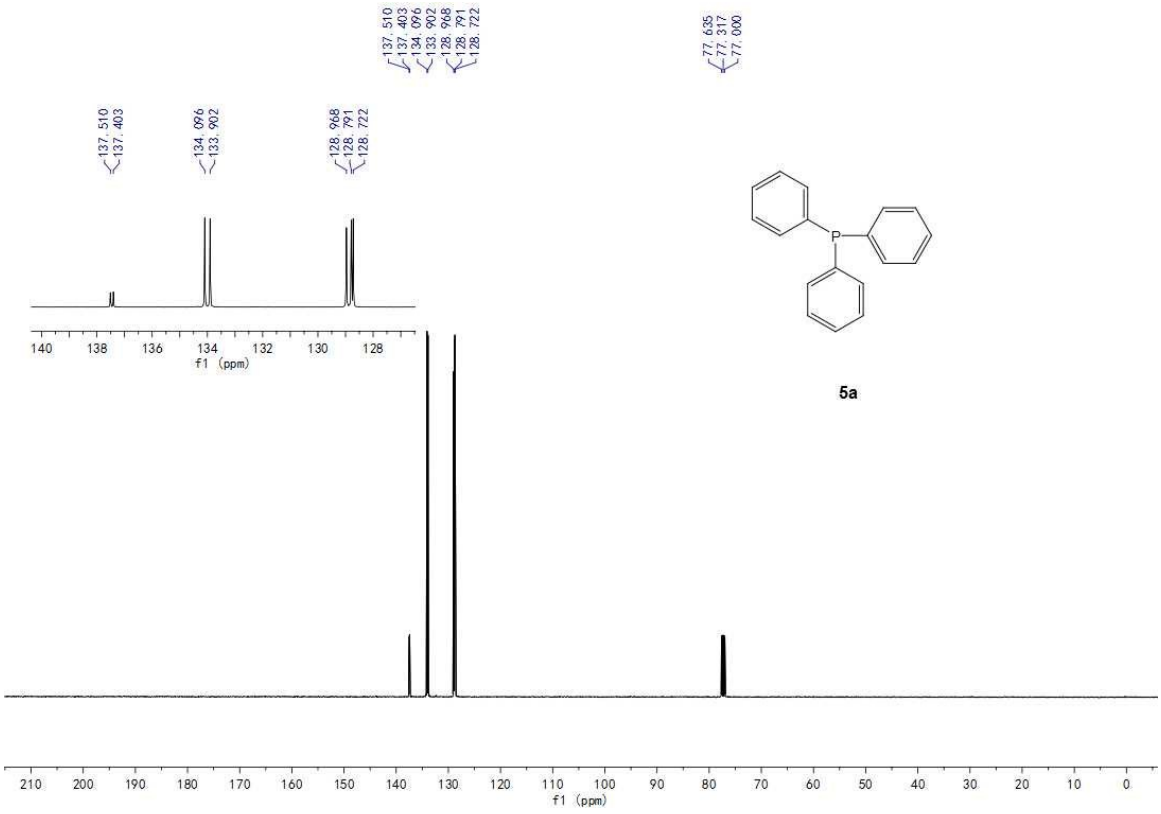

132.437
132.357
131.811
131.707
131.680
130.913
128.540
128.432
77.636
77.318
77.000
34.518
33.815
25.410
3p
133 132 131 130 129 128
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

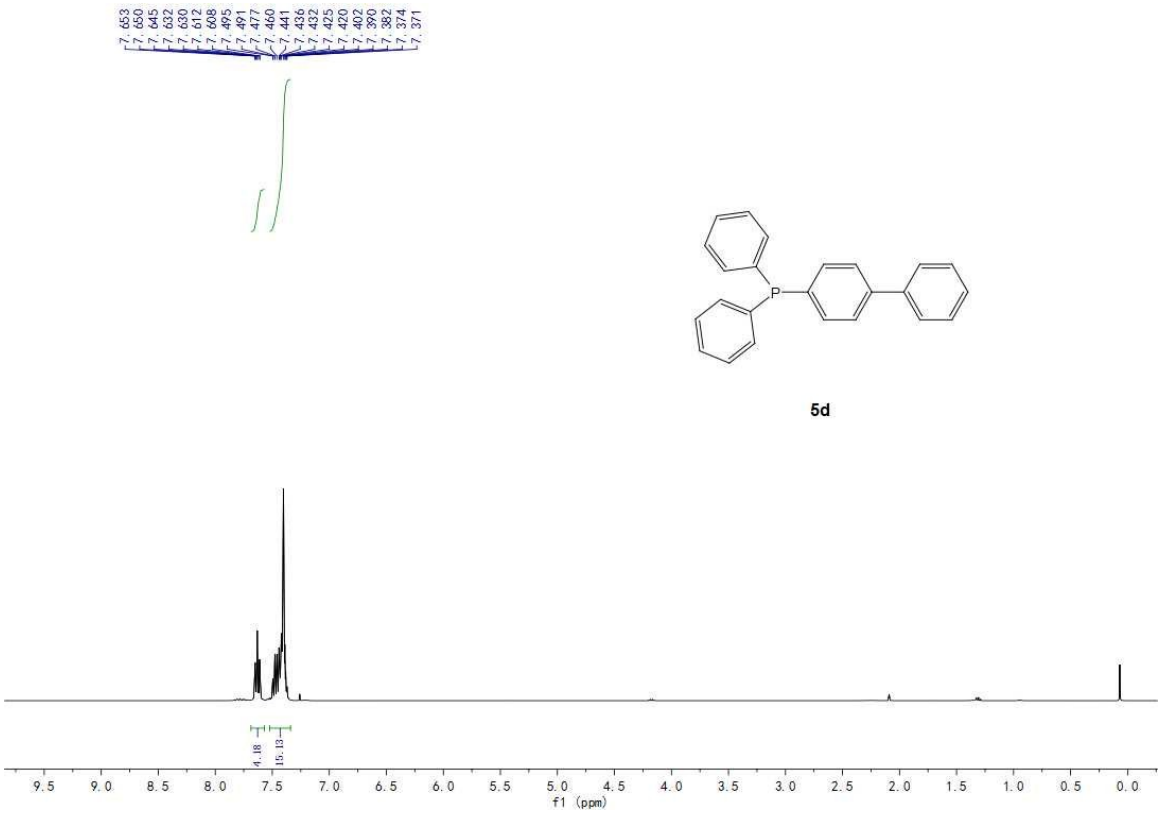

7.350
7.260
15.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

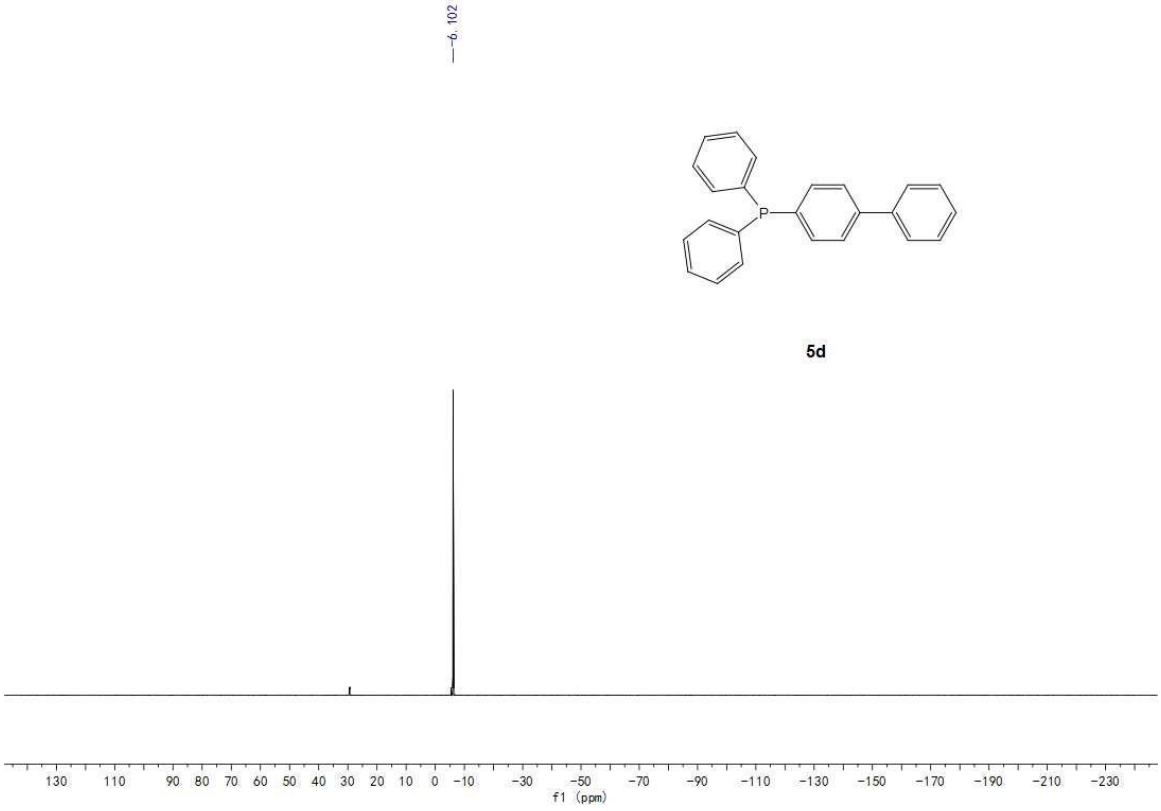
5a

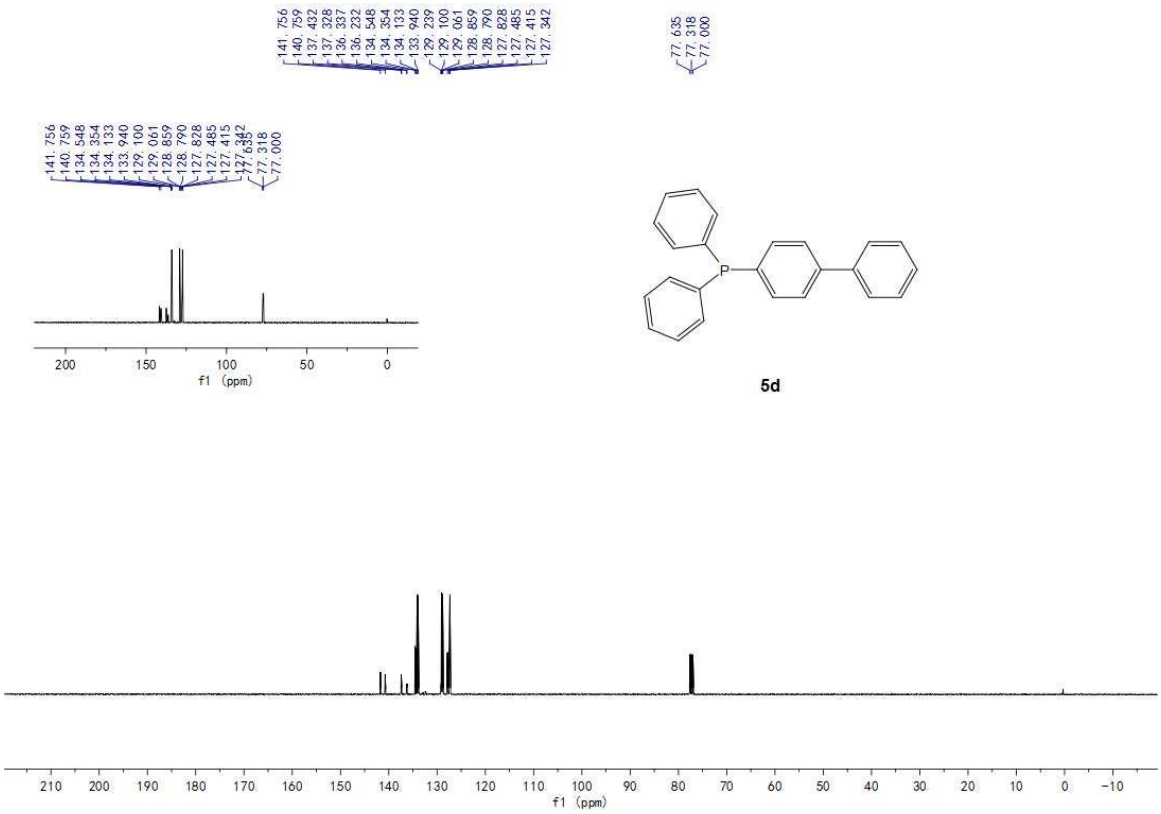

-5.373
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

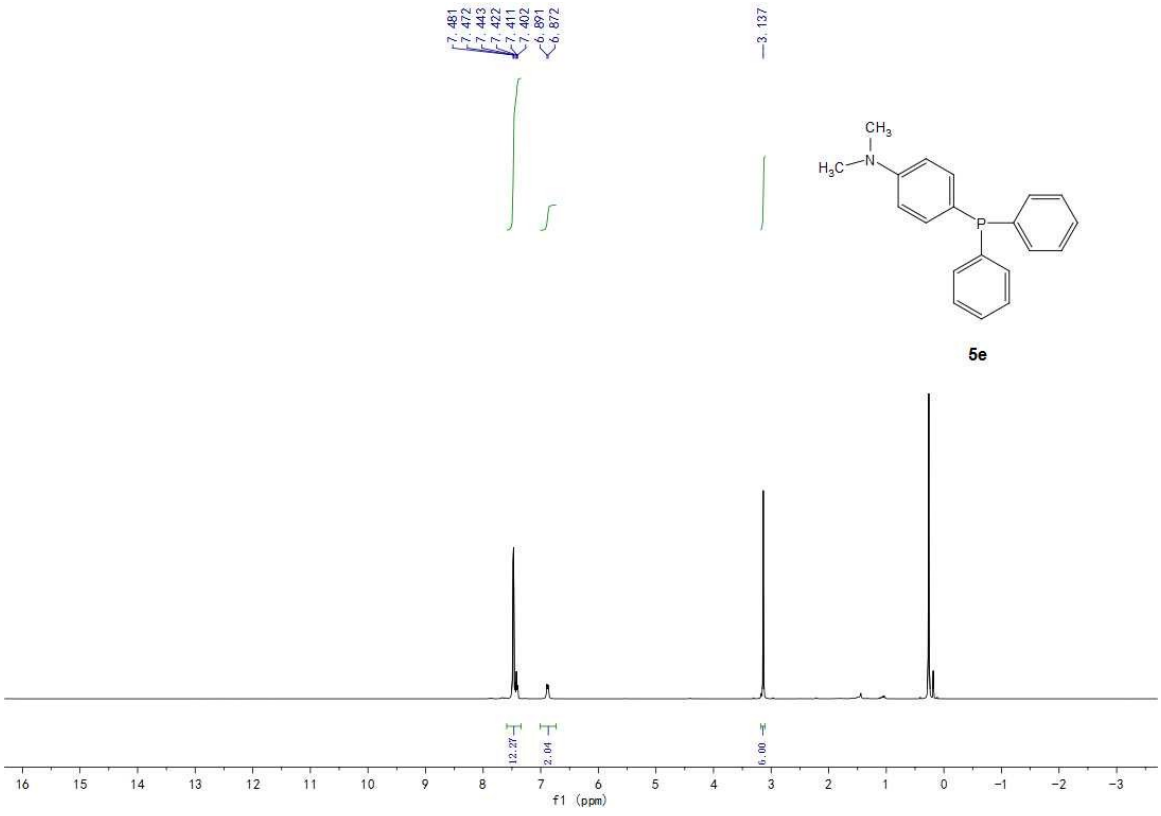
5a

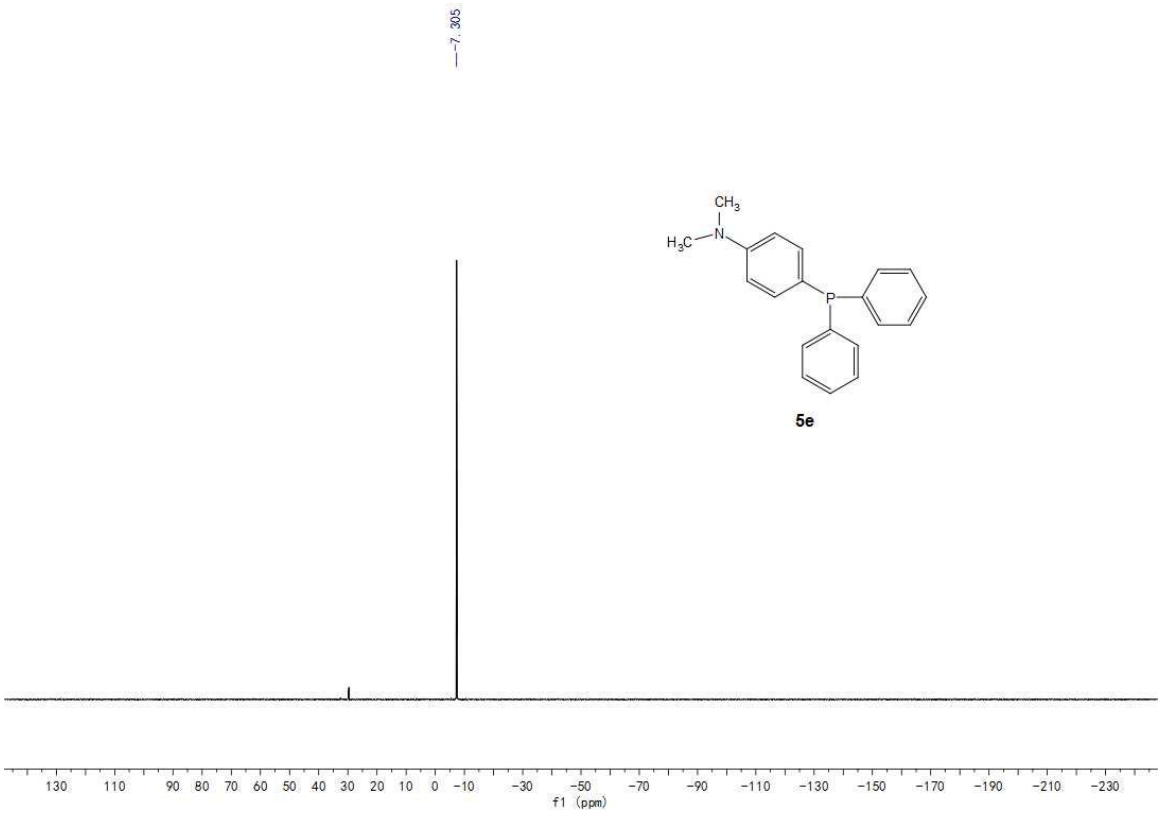

137.510
137.403
134.096
133.902
128.968
128.791
128.722
77.635
77.317
77.000
140 138 136 134 132 130 128
f1 (ppm)
P
5a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

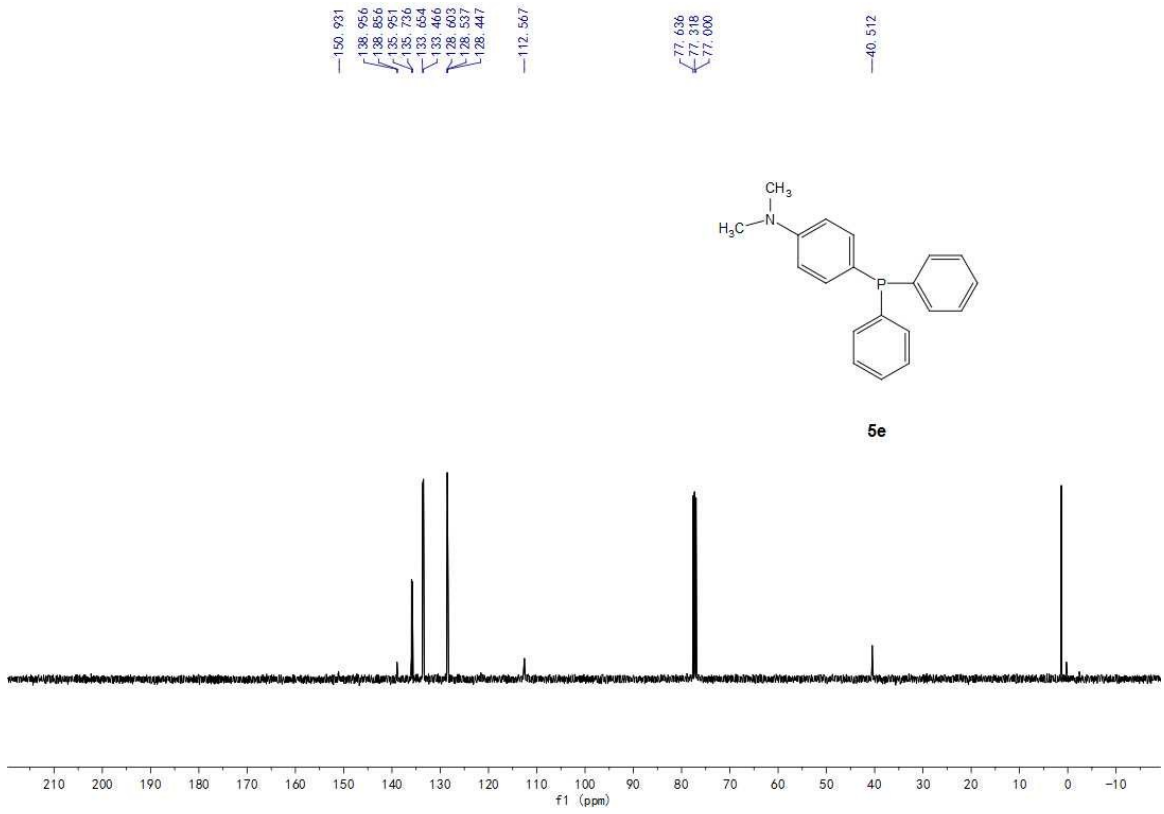

7.653
7.650
7.645
7.632
7.630
7.612
7.608
7.495
7.491
7.477
7.460
7.441
7.436
7.432
7.425
7.420
7.402
7.390
7.382
7.374
7.371
P
5d
4.18
15.13
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


-6.102
P
5d
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)


141.756
140.759
137.432
137.328
136.337
136.232
134.548
134.354
134.133
133.940
129.239
129.100
129.061
128.859
128.790
127.828
127.485
127.415
127.342
77.635
77.318
77.000
P
5d
200 150 100 50 0
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.481
7.472
7.443
7.422
7.411
7.402
6.891
6.872
3.137
CH3
H3C
N
P
5e
12.27
2.04
6.00
f1 (ppm)


7.305
CH3
H3C
N
P
5e
f1 (ppm)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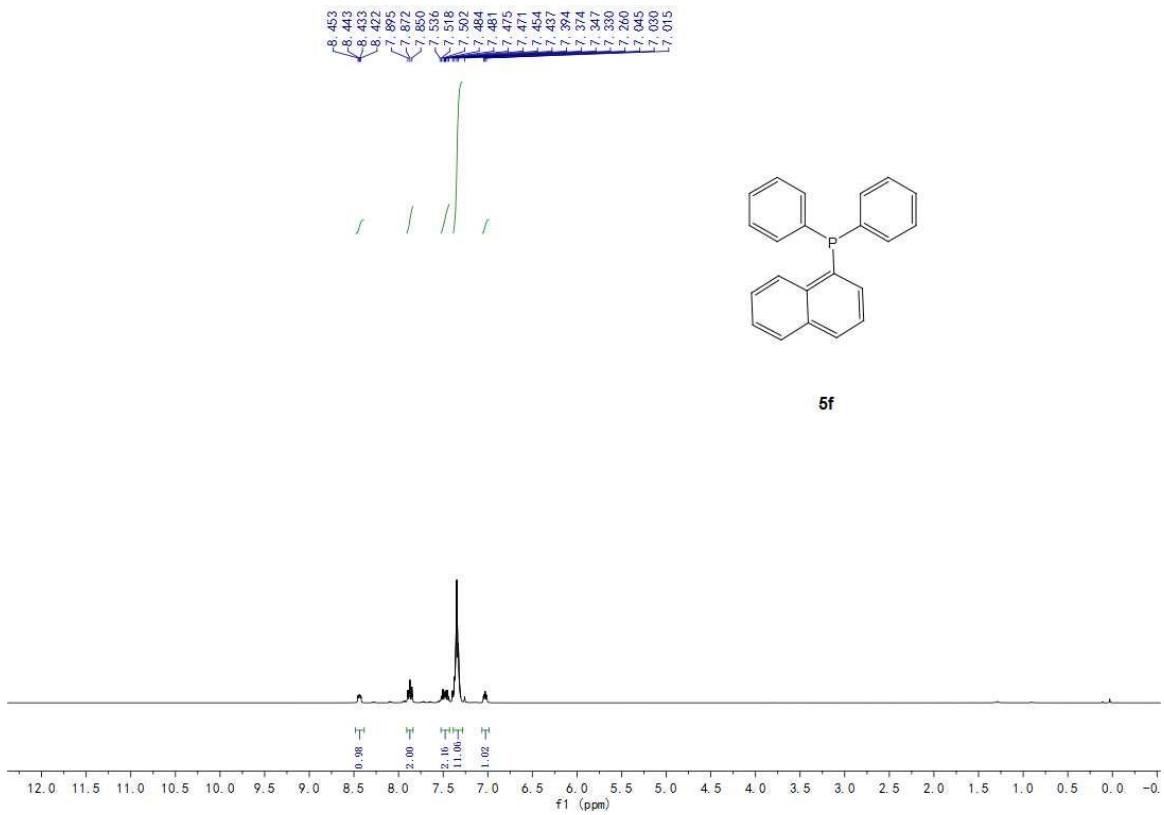
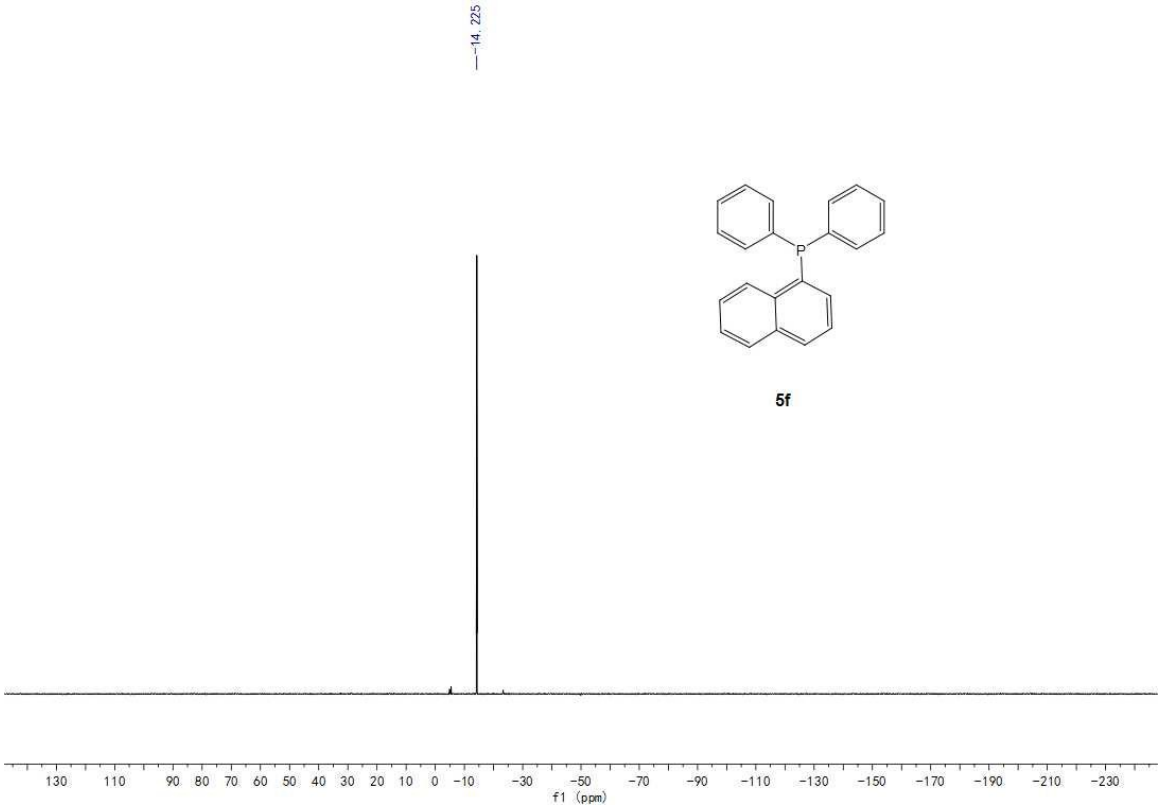
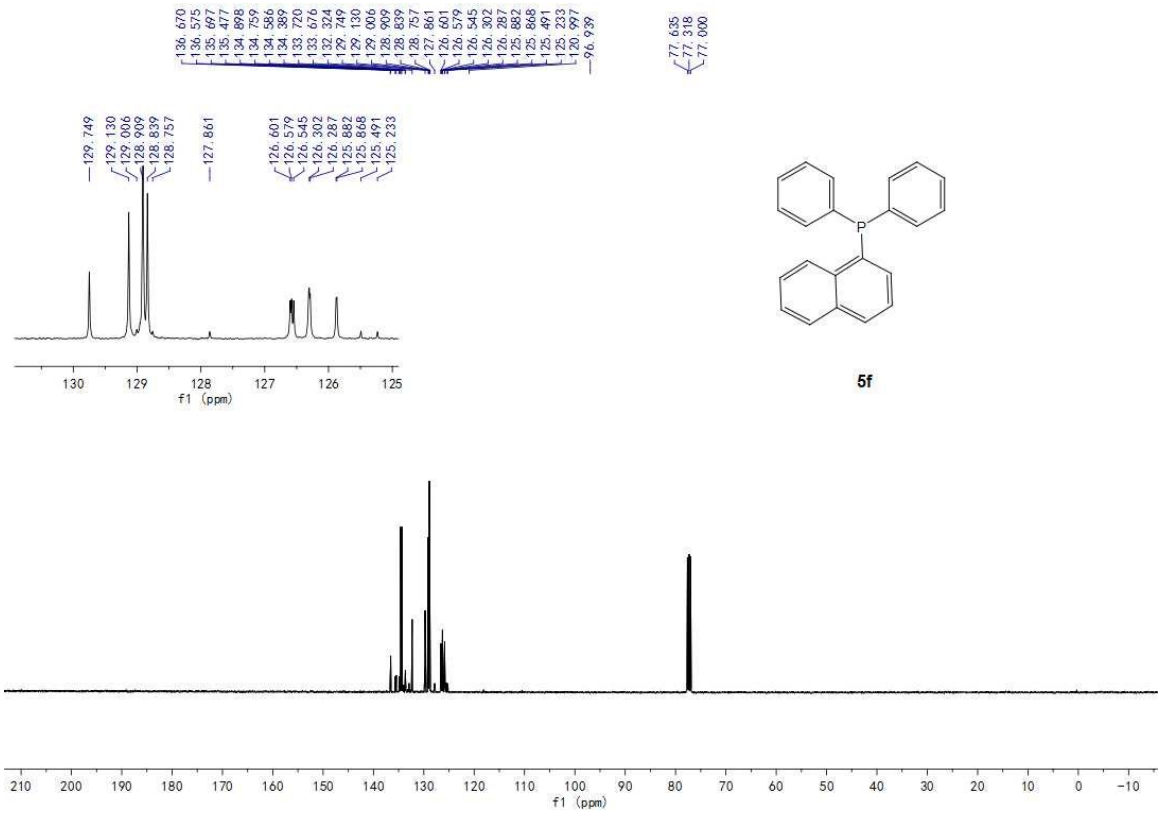
138.956
138.856
135.951
135.736
133.664
133.466
128.603
128.537
128.447
112.567
77.636
77.318
77.000
40.512
CH3
H3C
N
P
5e
f1 (ppm)


8.453
8.443
8.433
8.422
7.895
7.872
7.850
7.536
7.518
7.502
7.484
7.481
7.475
7.471
7.454
7.437
7.394
7.374
7.347
7.330
7.260
7.045
7.030
7.015
P
5f
0.98
2.00
2.16
11.00
1.02
f1 (ppm)


-14.225
5f
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)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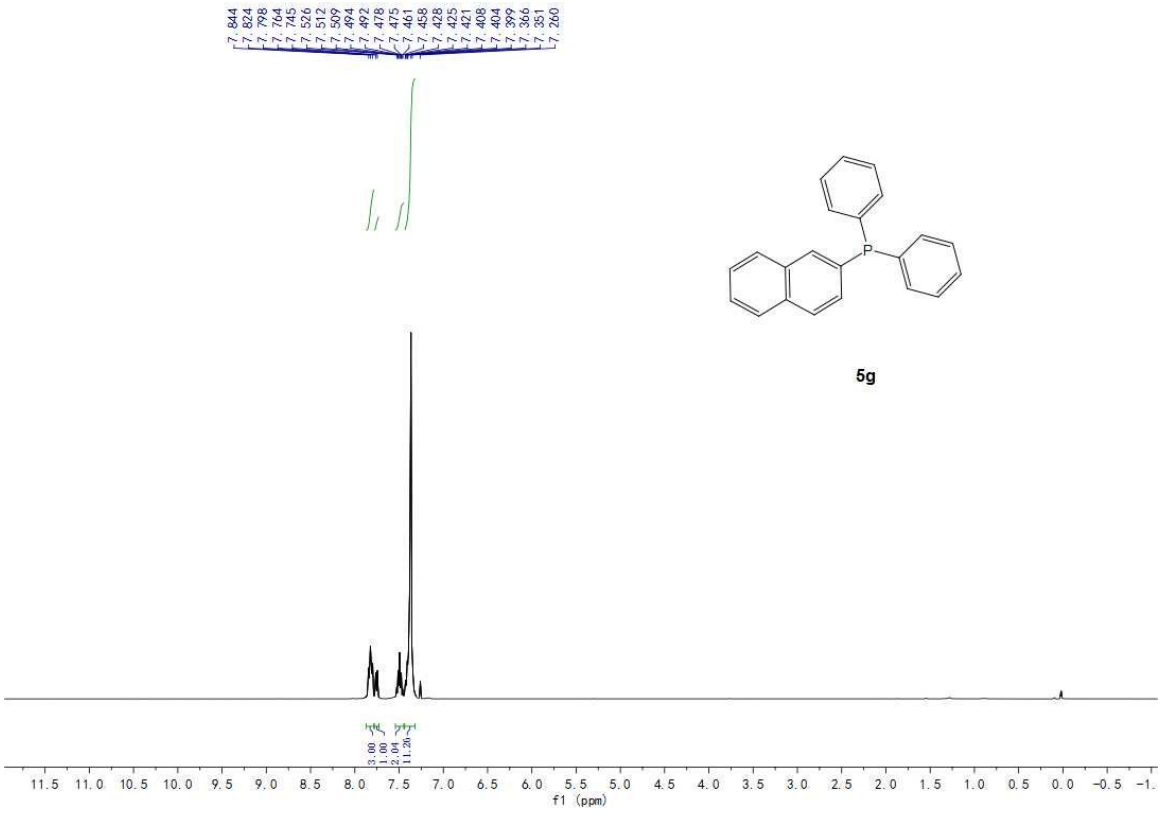
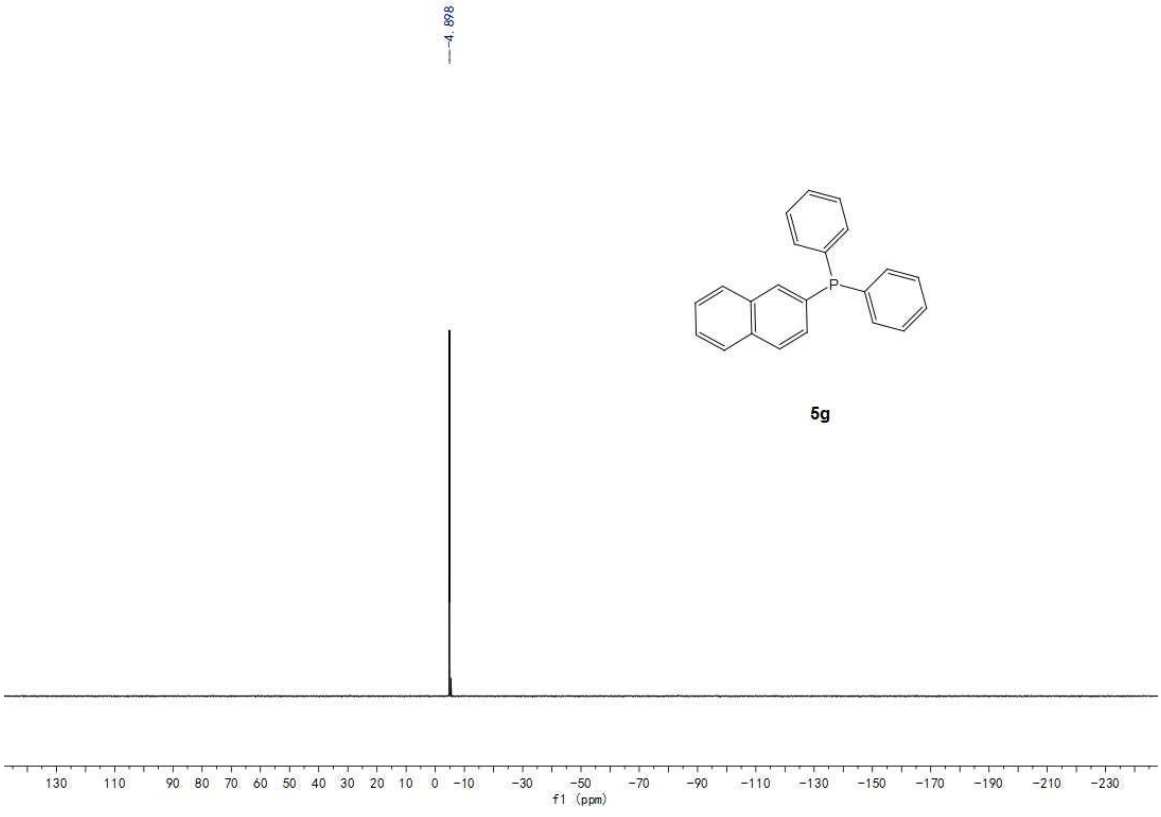
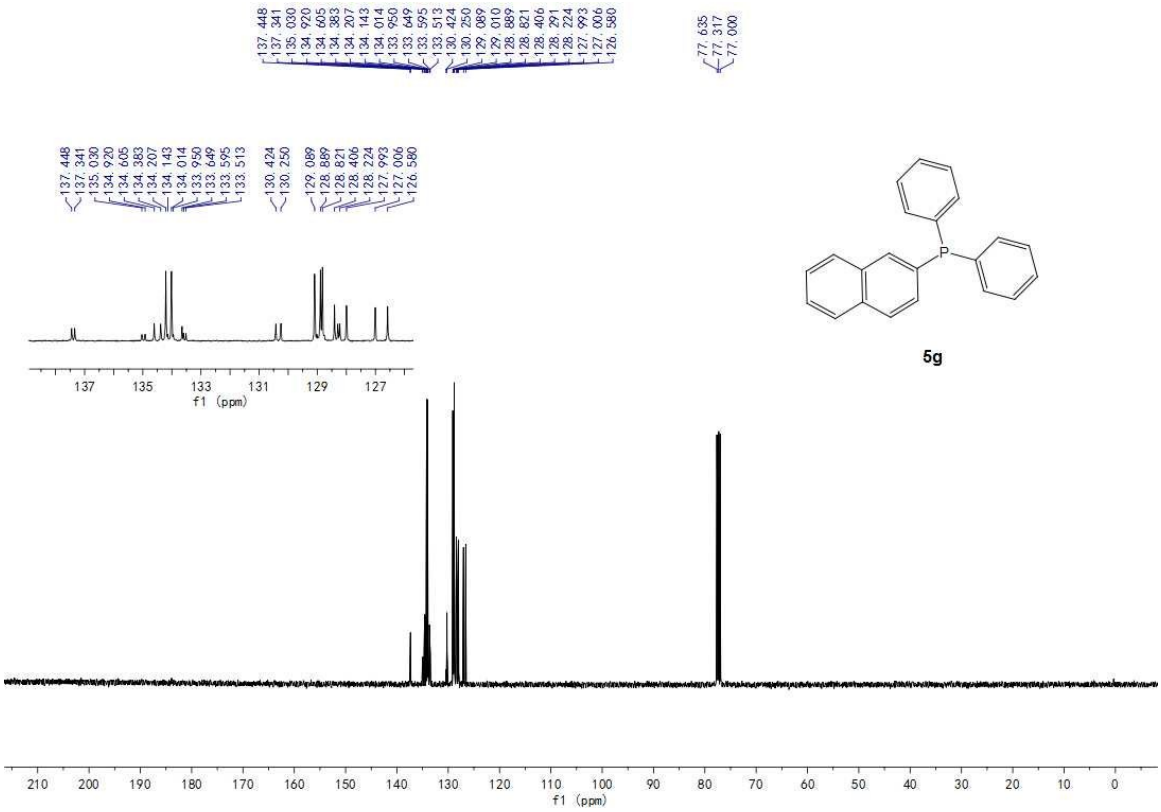
135.697
135.477
134.898
134.759
134.586
134.389
133.720
133.676
132.324
129.749
129.130
129.006
128.909
128.839
128.757
127.861
126.601
126.579
126.545
126.302
126.287
125.882
125.868
125.491
125.233
120.997
96.939
77.635
77.318
77.000
129.749
129.130
129.006
128.909
128.839
128.757
127.861
126.601
126.579
126.545
126.302
126.287
125.882
125.868
125.491
125.233
5f
130 129 128 127 126 125
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.844
7.824
7.798
7.764
7.745
7.526
7.512
7.509
7.494
7.492
7.478
7.475
7.461
7.458
7.428
7.425
7.421
7.408
7.404
7.399
7.366
7.351
7.260
3.00
1.00
2.04
11.26
f1 (ppm)

5g


-4.898
f1 (ppm)

5g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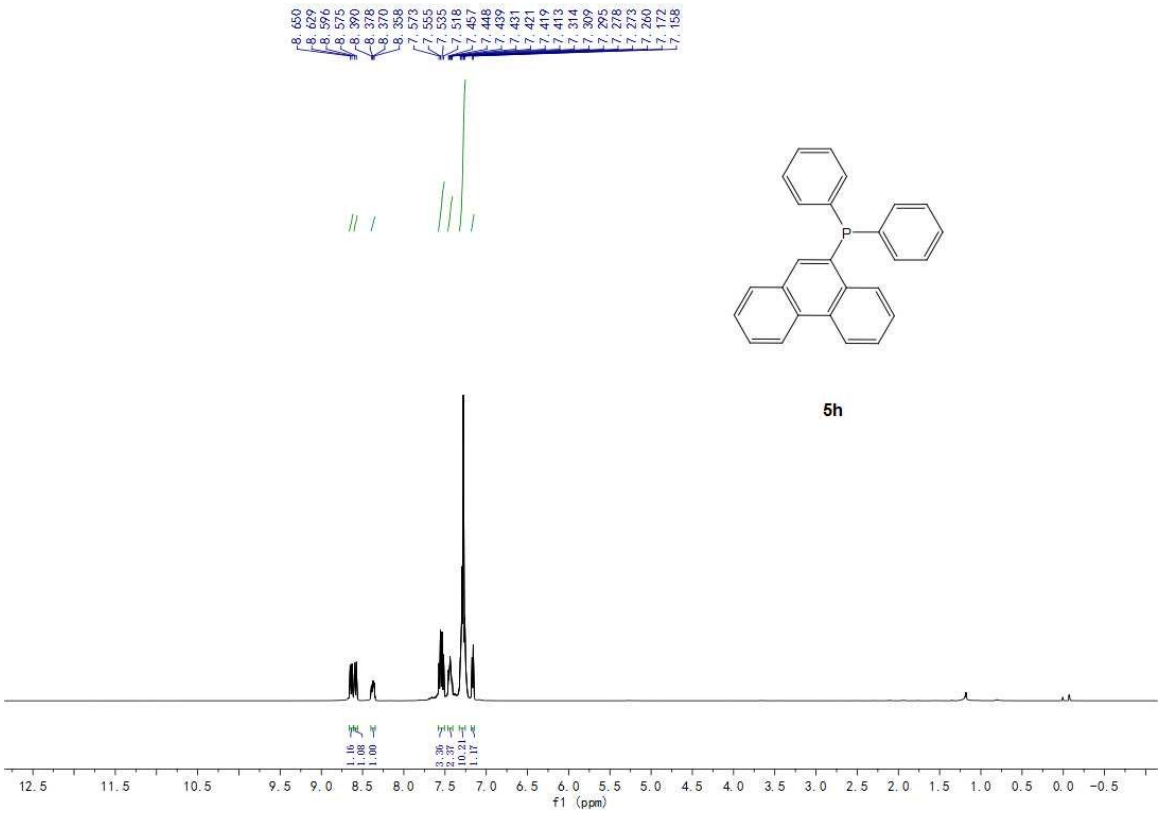
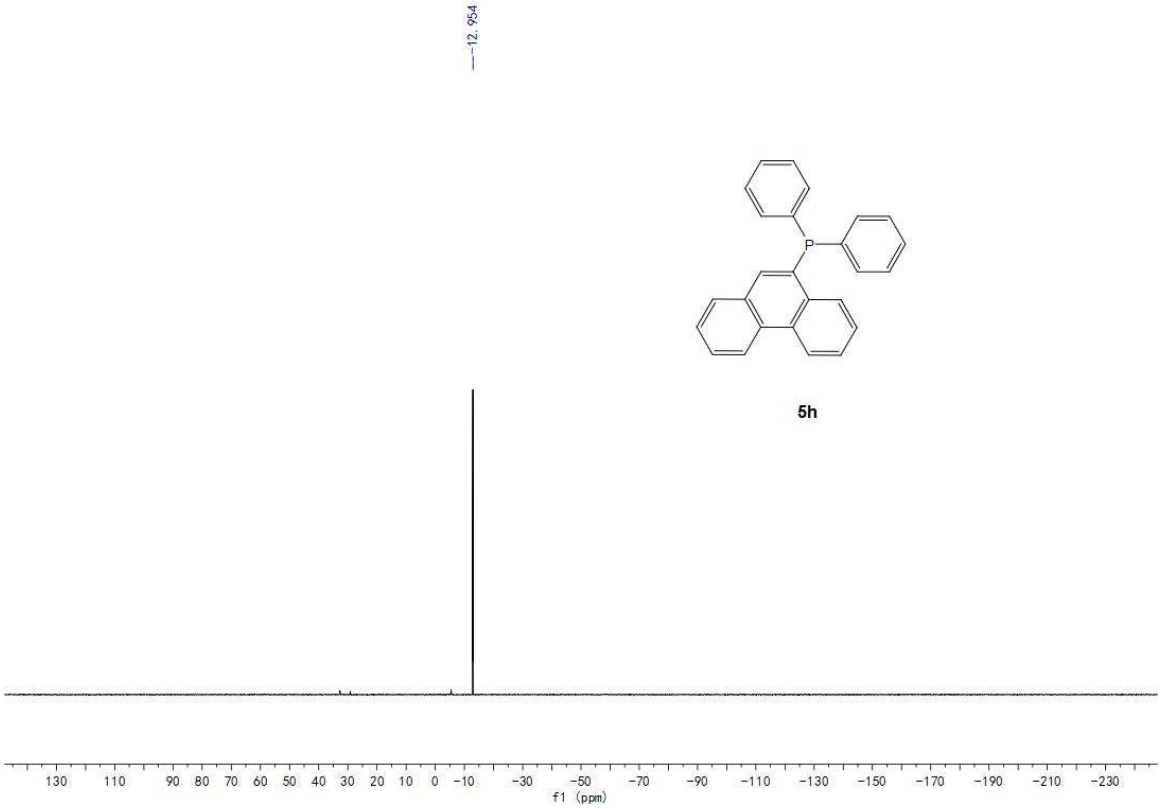
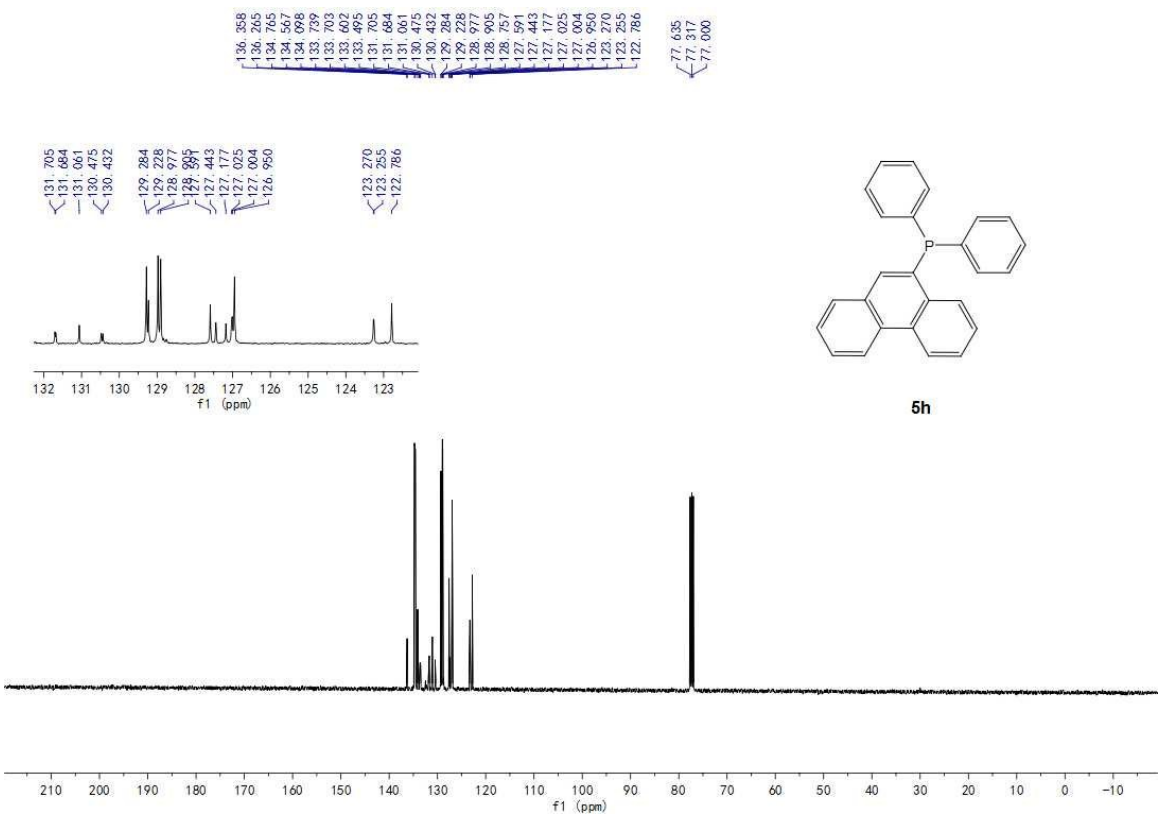
137.341
135.030
134.920
134.605
134.383
134.207
134.143
134.014
133.950
133.649
133.595
133.513
130.424
130.250
129.089
129.010
128.889
128.821
128.406
128.291
128.224
127.993
127.006
126.580
77.635
77.317
77.000
P
5g
f1 (ppm)


8.650
8.629
8.596
8.575
8.390
8.378
8.370
8.358
7.573
7.555
7.535
7.518
7.457
7.448
7.439
7.431
7.421
7.419
7.413
7.314
7.309
7.295
7.278
7.273
7.260
7.172
7.158
P
5h
f1 (ppm)


-12.954
P
5h
130 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)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133.739
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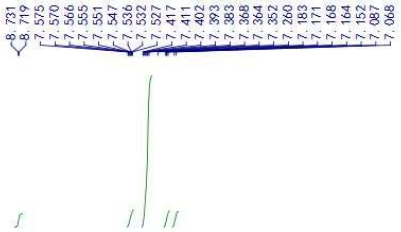
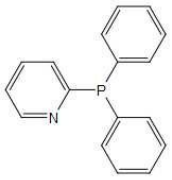
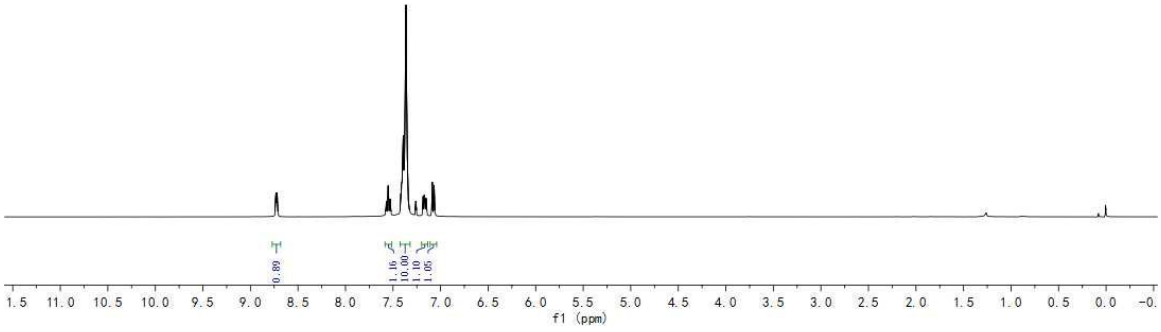
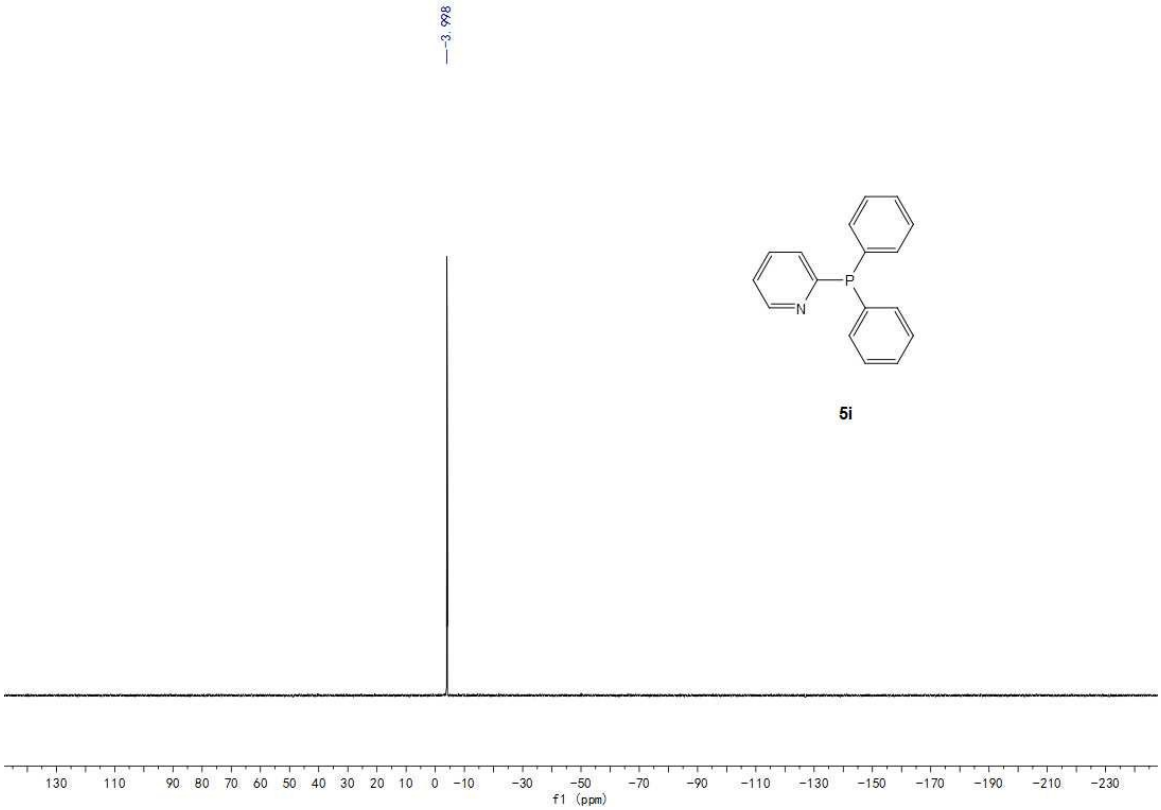
133.602
133.495
131.705
131.684
131.061
130.475
130.432
129.284
129.228
128.977
128.905
128.757
127.591
127.443
127.177
127.025
127.004
126.950
123.270
123.255
122.786
77.635
77.317
77.000
131.705
131.684
131.061
130.475
130.432
129.284
129.228
128.977
127.443
127.177
127.025
127.004
126.950
123.270
123.255
122.786
P
5h
132 131 130 129 128 127 126 125 124 123
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.731
8.719
7.575
7.570
7.566
7.555
7.551
7.547
7.536
7.532
7.527
7.417
7.411
7.402
7.393
7.383
7.368
7.364
7.352
7.260
7.183
7.171
7.168
7.164
7.152
7.087
7.068


P
N

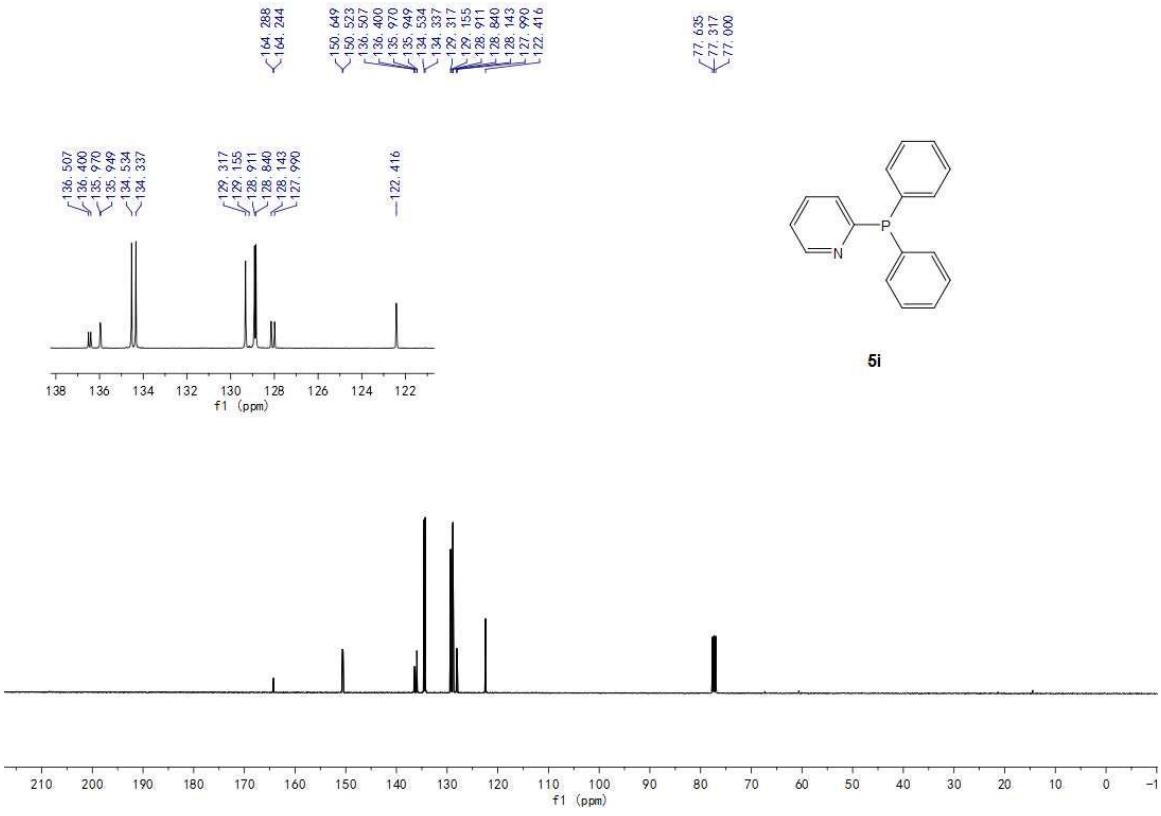
5i


0.89
1.16
1.00
1.10
1.05
f1 (ppm)


-3.998
f1 (ppm)

P
N

5i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136.400
135.970
135.949
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134.337
129.317
129.155
128.911
128.840
128.143
127.990
122.416
77.635
77.317
77.000
136.507
136.400
135.970
135.949
134.534
134.337
129.317
129.155
128.911
128.840
128.143
127.990
122.416
138 136 134 132 130 128 126 124 122
f1 (ppm)
N
P
5i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)