

Supplementary Information

TEMPO/PhI(OAc)₂ promoted the α -aminophosphinylation of alcohols with amines and H-phosphine oxides in the aqueous medium

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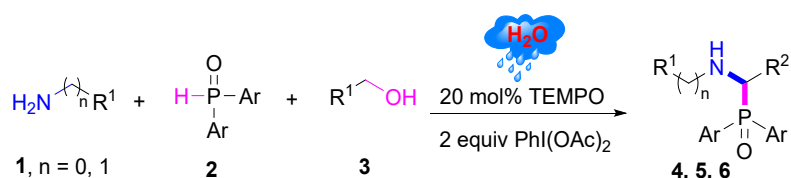
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1. General information

All commercially available reagent grade chemicals were purchased from BenTu Reagent, Acros, and Alfa Aesar Chemical Company and used as received without further purification unless otherwise stated. ^1H , ^{13}C , ^{31}P and ^{19}F NMR spectra were recorded in CDCl_3 on bruker advance 400 MHz NMR at room temperature, the chemical shifts (δ) were expressed in ppm and J values were given in Hz. The following abbreviations are used to indicate the multiplicity: singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublets (dd), doublet of triplets (dt), and multiplet (m). All first order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted were designated as multiplet (m). High resolution mass spectra (HRMS) were obtained on a Waters UPLC-Xevo TQMS (PDA Detector) / Quattro Premier XE triquadrupole mass spectrometer by ESI method. Flash column chromatography was conducted on silica gel(200-300 mesh).

2. Experimental Section

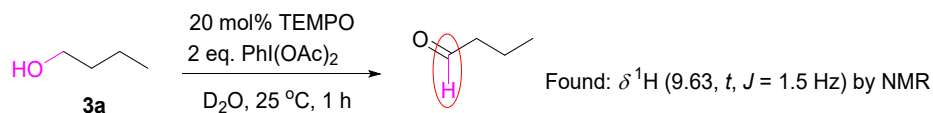
2.1 General procedure for the synthesis of compounds 4, 5, and 6



Alcohols (**3**, 0.5 mL), amines (**1**, 1 mmol), H-phosphine oxides (**2**, 2.5 mmol), $\text{PhI}(\text{OAc})_2$ (2 mmol, 2 equiv), TEMPO (0.2 mmol) and H_2O (4 mL) were dissolved in sealed tube and stirring at 25 °C for 5 h. After completion of reaction, the mixture was quenched with sodium bicarbonate (20 mL), extracted with ethyl acetate (3×10 mL). The combined organic layers were washed with saturated brine twice, and dried over anhydrous MgSO_4 . After filtration, the solvent was evaporated in vacuum. The crude product was purified by silica gel chromatography (DCM : methanol = 50:1) to give the desired compounds **4**, **5** and **6**.

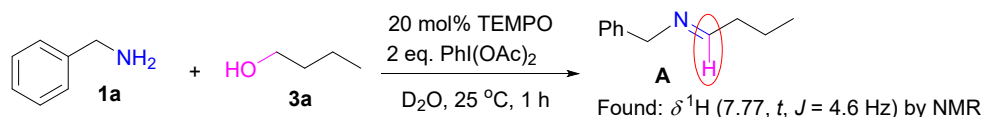
2.2 Control experiment

(a) the detection of *n*-butyl aldehyde



n-Butyl alcohol (**3a**, 0.5 mL), PhI(OAc)₂ (2 mmol, 2 equiv), TEMPO (0.2 mmol) and D₂O (4 mL) were dissolved in sealed tube and stirring at 25 °C for 1 h. The mixture was detected by NMR analysis in DMSO-*d*₆, and the result was recorded in Figure S1a.

(b) the detection of imine



n-Butyl alcohols (**3a**, 0.5 mL), benzylamine (**1a**, 1 mmol), PhI(OAc)₂ (2 mmol, 2 equiv), TEMPO (0.2 mmol) and D₂O (4 mL) were dissolved in sealed tube and stirring at 25 °C for 1 h. The mixture was detected by NMR analysis in DMSO-*d*₆, and the result was recorded in Figure S1b.

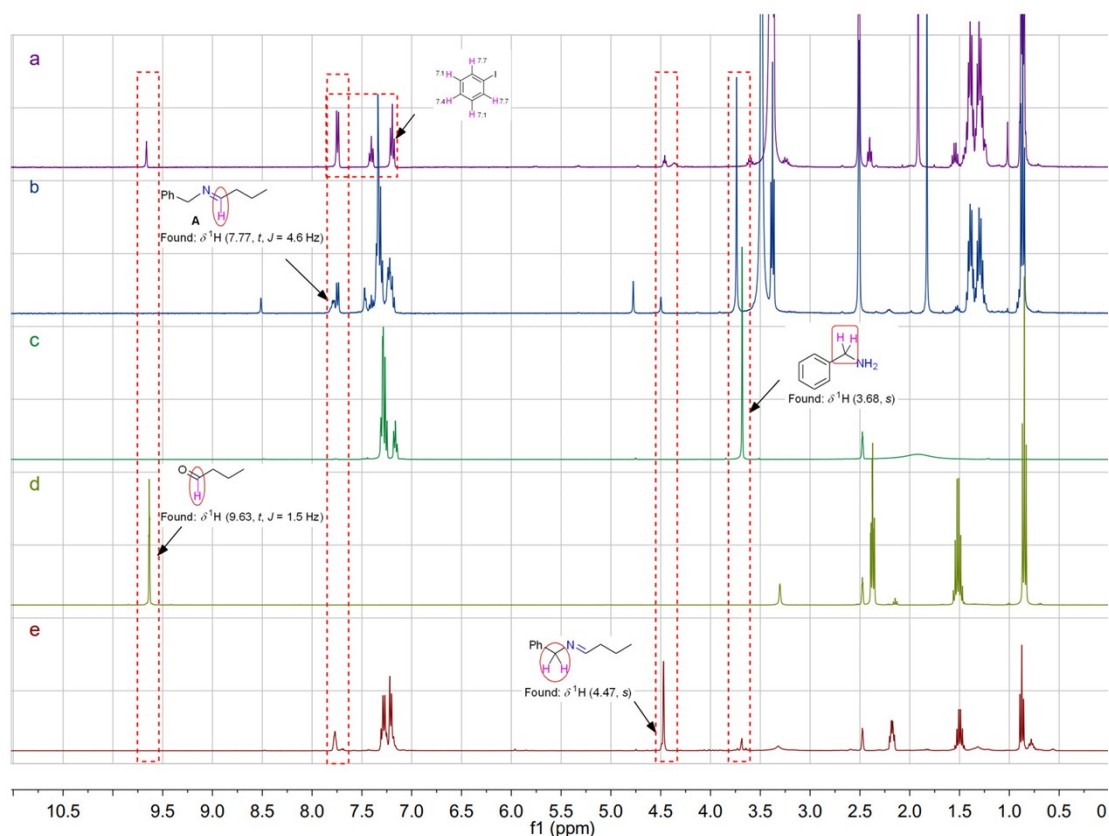


Figure S1 NMR analysis of control experiment. (a: the mixture of control experiment (a); b: the mixture of control experiment (b); c: standard substance benzylamine; d: standard substance *n*-butyl aldehyde; e: the mixture of reaction

between benzylamine and *n*-butyl aldehyde in absence of TEMPO and $\text{PhI}(\text{OAc})_2$
 under a solvent-free condition)

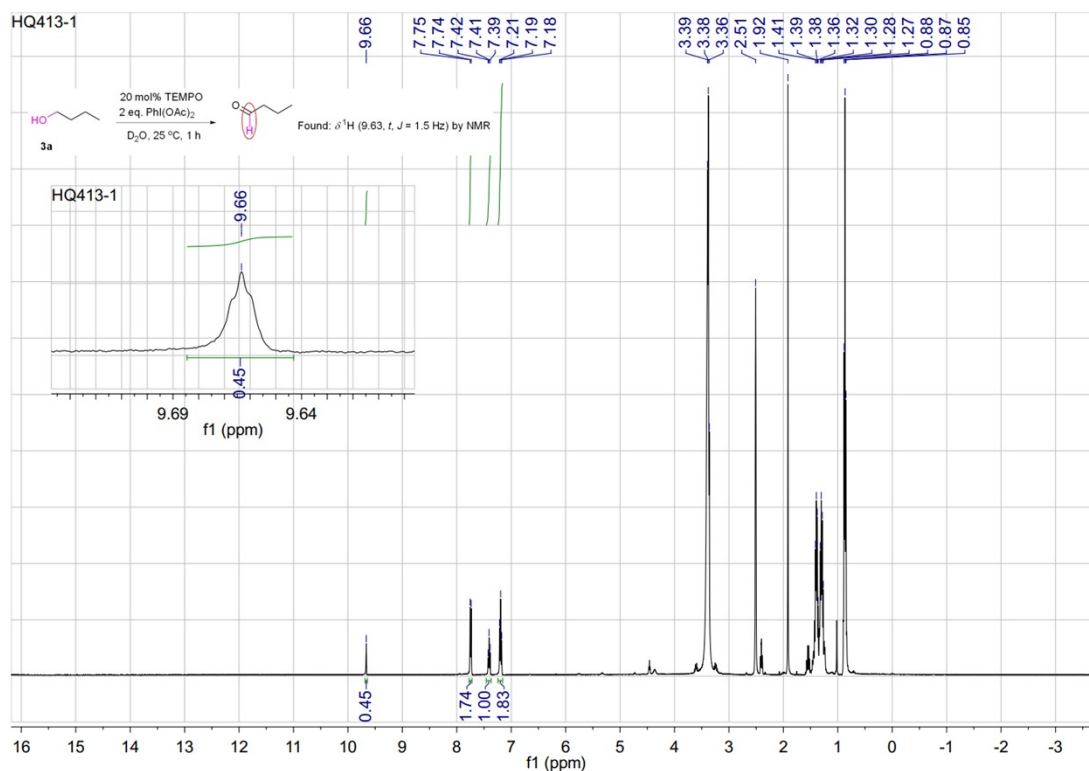


Figure S1a the mixture of control experiment (a)

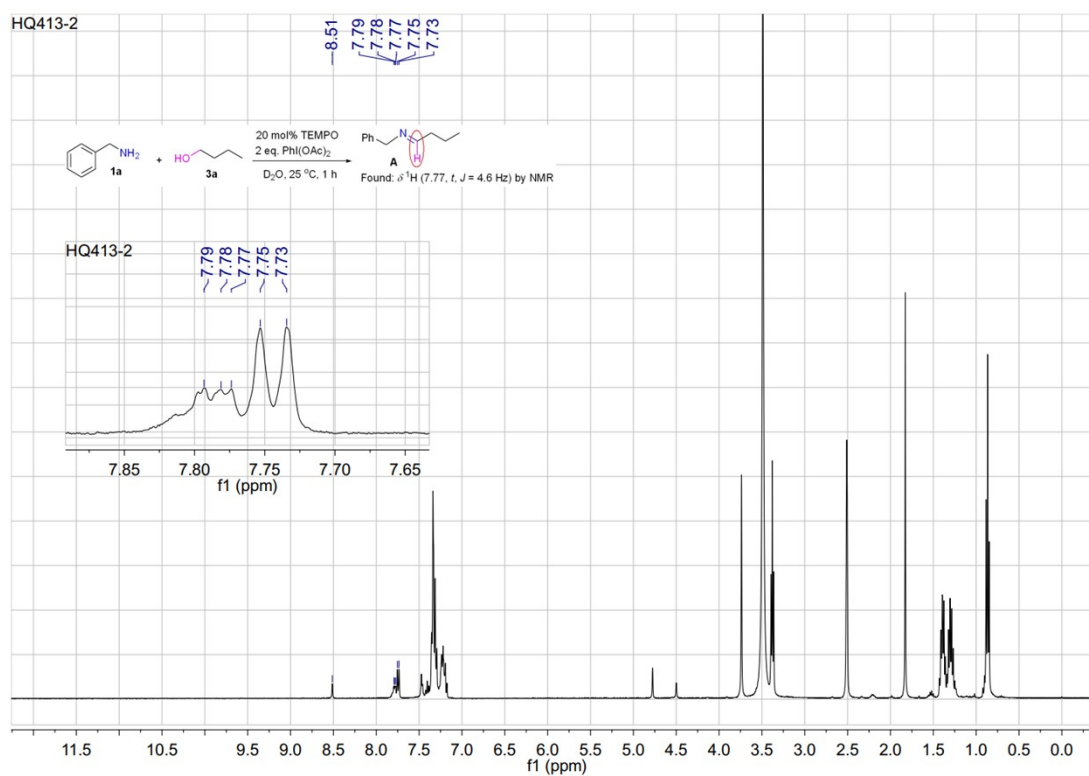


Figure S1b the mixture of control experiment (b)

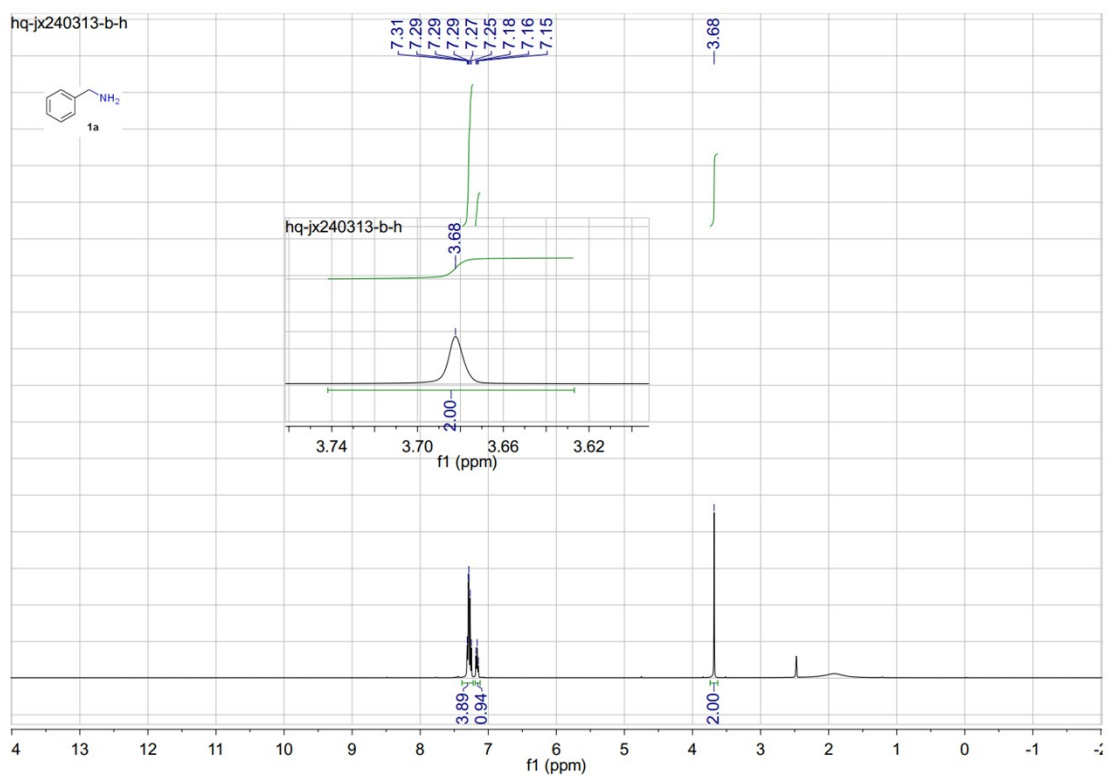


Figure S1c standard substance benzylamine

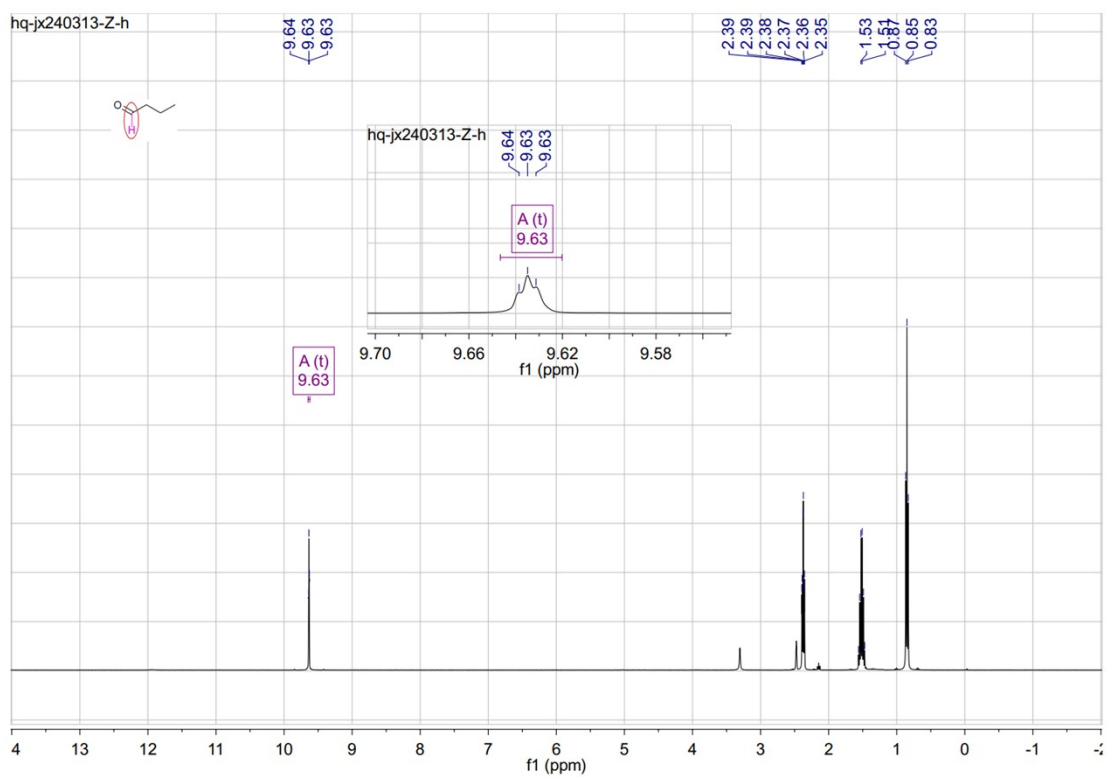


Figure S1d standard substance *n*-butyl aldehyde

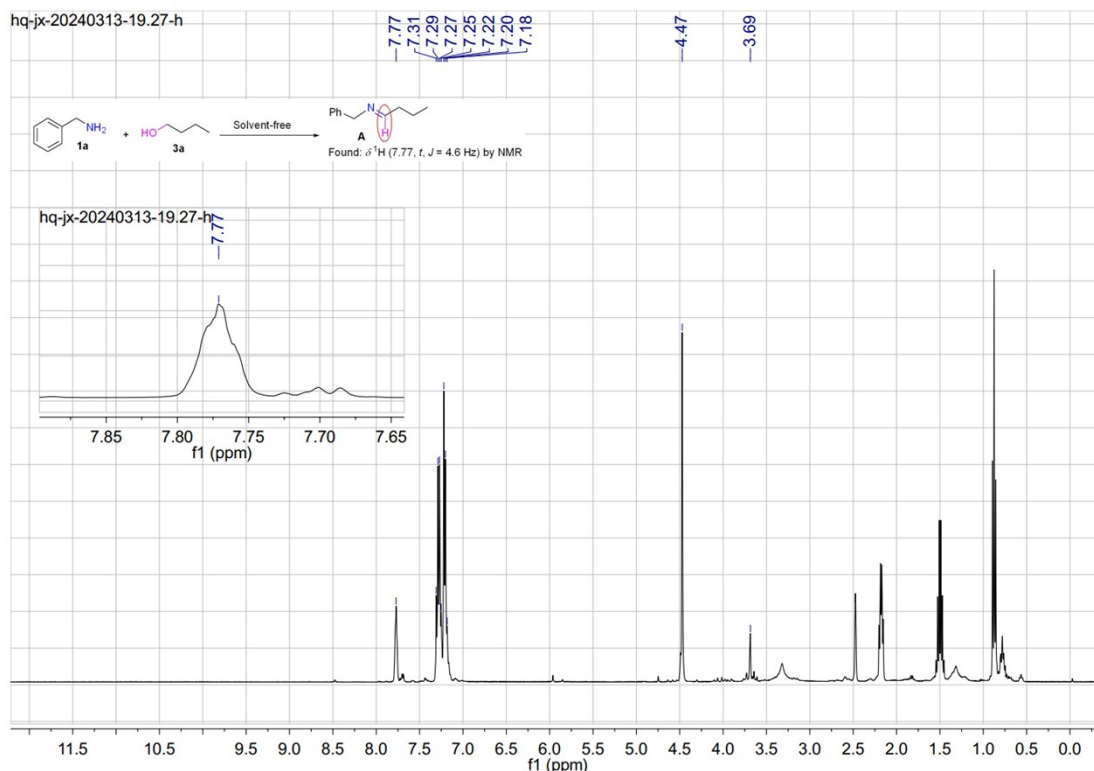
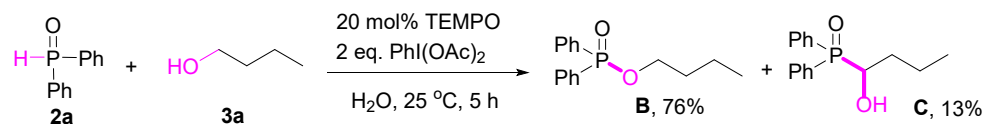


Figure S1e the mixture of reaction between benzylamine and *n*-butyl alcohol in absence of TEMPO and $\text{PhI}(\text{OAc})_2$ under a solvent-free condition.

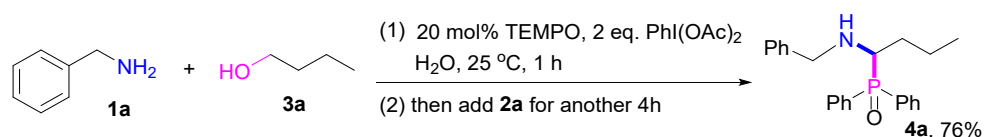
(c) the reaction of *n*-butyl alcohol and H-phosphine oxide



n-Butyl alcohols (**3a**, 0.5 mL), H-phosphine oxides (**2a**, 2.5 mmol), $\text{PhI}(\text{OAc})_2$ (2 mmol, 2 equiv), and H_2O (4 mL) were dissolved in sealed tube with TEMPO (0.2 mmol) and stirring at 25 °C for 5 h. After completion of reaction, the mixture was quenched with sodium bicarbonate (20 mL), extracted with ethyl acetate (3×10 mL). The combined organic layers were washed with saturated brine twice, and dried over anhydrous MgSO_4 . After filtration, the solvent was evaporated in vacuum. The crude product was purified by silica gel chromatography (PE : EA = 2:1) to give the desired compounds **B** and **C**. **Butyl diphenylphosphinate (B)**: ^1H NMR (400 MHz, CDCl_3) δ 7.80 (dd, $J = 12.0, 7.7$ Hz, 4H), 7.56 – 7.37 (m, 6H), 4.02 (q, $J = 6.6$ Hz, 2H), 1.74 – 1.65 (m, 2H), 1.48 – 1.37 (m, 2H), 0.90 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3)

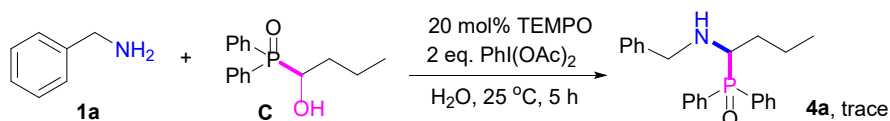
δ 132.23, 132.08 (d, $J = 2.8$ Hz), 131.66, 131.56, 130.87, 128.56, 128.43, 64.72 (d, $J = 6.1$ Hz), 32.56 (d, $J = 6.7$ Hz), 18.87, 13.66. ^{31}P NMR (162 MHz, CDCl_3) δ 31.08. HRMS: $[\text{M}+\text{Na}]^+$ m/z calcd for $\text{C}_{16}\text{H}_{19}\text{O}_2\text{PNa}$: 297.1015; found: 297.1011. **(1-hydroxybutyl)diphenylphosphine oxide (C)**: ^1H NMR (400 MHz, CDCl_3) δ 7.87 (dd, $J = 9.6, 8.2$ Hz, 2H), 7.78 (dd, $J = 9.7, 8.2$ Hz, 2H), 7.57 – 7.41 (m, 6H), 4.40 (d, $J = 9.6$ Hz, 1H), 3.56 (s, 1H), 1.75 – 1.53 (m, 3H), 1.45 – 1.31 (m, 1H), 0.85 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 132.00, 131.97, 131.95, 131.87, 131.49, 131.46, 131.40, 130.58 (d, $J = 11.0$ Hz), 129.68 (d, $J = 4.0$ Hz), 128.67, 128.55, 128.44, 70.33 (d, $J = 83.3$ Hz), 32.44 (d, $J = 2.8$ Hz), 19.21 (d, $J = 12.3$ Hz), 13.66. ^{31}P NMR (162 MHz, CDCl_3) δ 31.86. HRMS: $[\text{M}+\text{Na}]^+$ m/z calcd for $\text{C}_{16}\text{H}_{19}\text{O}_2\text{PNa}$: 297.1015; found: 297.1009.

(d) the three-component reaction was conducted in a stepwise manner



n-Butyl alcohols (**3a**, 0.5 mL), benzylamine (**1a**, 1 mmol), $\text{PhI}(\text{OAc})_2$ (2 mmol, 2 equiv), TEMPO (0.2 mmol) and H_2O (4 mL) were dissolved in sealed tube and stirring at $25\text{ }^\circ\text{C}$ for 1 h. Then, the mixture was added **2a** (2.5 mmol) for another 4 h. After completion of reaction, the mixture was quenched with sodium bicarbonate (20 mL), extracted with ethyl acetate (3×10 mL). The combined organic layers were washed with saturated brine twice, and dried over anhydrous MgSO_4 . After filtration, the solvent was evaporated in vacuum. The crude product was purified by silica gel chromatography (DCM : methanol = 50:1) to give the desired compound **4a**.

(e) the reaction between 1a and compound C

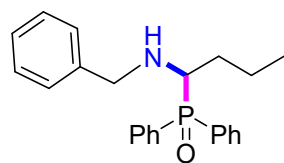


(1-hydroxybutyl)diphenylphosphine oxide (**C**, 0.1 mmol), benzylamine (**1a**, 0.12 mmol), $\text{PhI}(\text{OAc})_2$ (0.2 mmol, 2 equiv), TEMPO (0.02 mmol) and H_2O (0.4 mL) were dissolved in sealed tube and stirring at $25\text{ }^\circ\text{C}$ for 5 h. After completion of reaction, the

mixture was quenched with sodium bicarbonate (1 mL), extracted with ethyl acetate (3×3 mL). The combined organic layer was detected by TLC, and only a trace of **4a** was observed.

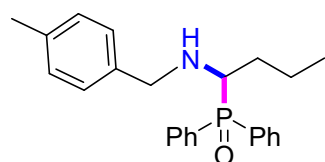
3. Characterization data of compounds 4, 5, 6

(1-(benzylamino)butyl)diphenylphosphine oxide (4a)



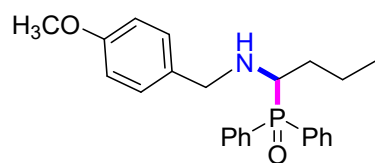
^1H NMR (400 MHz, CDCl_3) δ 8.05 – 7.95 (m, 2H), 7.92 – 7.84 (m, 2H), 7.60 – 7.45 (m, 6H), 7.33 – 7.26 (m, 3H), 7.16 – 7.09 (m, 2H), 3.65 (dd, $J = 50.5, 12.8$ Hz, 2H), 3.49 – 3.42 (m, 1H), 1.91 – 1.75 (m, 2H), 1.64 – 1.51 (m, 2H), 1.44 – 1.34 (m, 1H), 0.87 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.73, 133.23, 132.32, 131.85, 131.76, 131.63, 131.61, 131.58, 131.40, 131.35, 131.26, 128.57, 128.45 (d, $J = 2.4$ Hz), 128.30 (d, $J = 1.0$ Hz), 56.76 (d, $J = 82.0$ Hz), 52.92 (d, $J = 7.2$ Hz), 31.71 (d, $J = 3.6$ Hz), 19.85 (d, $J = 10.1$ Hz), 14.00. ^{31}P NMR (162 MHz, CDCl_3) δ 31.06. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NOP}$: 364.1825; found: 364.1823.

(1-((4-methylbenzyl)amino)butyl)diphenylphosphine oxide (4b)



^1H NMR (400 MHz, CDCl_3) δ 8.02 – 7.94 (m, 2H), 7.93 – 7.85 (m, 2H), 7.57 – 7.44 (m, 6H), 7.09 (d, $J = 7.8$ Hz, 2H), 7.01 (d, $J = 8.0$ Hz, 2H), 3.60 (dd, $J = 54.6, 12.6$ Hz, 2H), 3.44 (ddd, $J = 8.6, 6.2, 3.9$ Hz, 1H), 2.34 (s, 3H), 1.91 – 1.78 (m, 2H), 1.65 – 1.50 (m, 2H), 1.44 – 1.33 (m, 1H), 0.87 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 136.70 (d, $J = 4.3$ Hz), 133.27, 132.36, 132.28, 131.89, 131.81, 131.60 (t, $J = 2.5$ Hz), 131.38, 131.36 – 131.34 (m), 131.29, 128.98, 128.56, 128.44 (d, $J = 3.0$ Hz), 128.31, 128.26, 56.79 (d, $J = 82.4$ Hz), 52.71 (d, $J = 7.3$ Hz), 31.74 (d, $J = 3.5$ Hz), 21.10, 19.87 (d, $J = 10.2$ Hz), 14.02. ^{31}P NMR (162 MHz, CDCl_3) δ 31.04. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NOP}$: 378.1981; found: 378.1982.

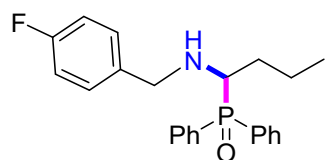
(1-((4-methoxybenzyl)amino)butyl)diphenylphosphine oxide (4c)



^1H NMR (400 MHz, CDCl_3) δ 8.01 – 7.92 (m, 2H), 7.90 – 7.83 (m, 2H), 7.52 – 7.43 (m, 6H), 7.02 (d, $J = 8.6$ Hz, 2H), 6.83 – 6.77 (m, 2H), 3.78 (s, 3H), 3.57 (dd, $J = 52.1, 12.6$ Hz, 2H), 3.43 (ddd, $J = 8.6, 6.0, 4.0$ Hz, 1H), 1.90 – 1.75 (m, 1H), 1.56 (tdt, $J = 9.6, 8.0, 4.8$ Hz, 2H), 1.45 – 1.31 (m, 1H), 0.85 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 158.73, 133.15, 132.61 (d, $J = 2.9$ Hz), 132.24,

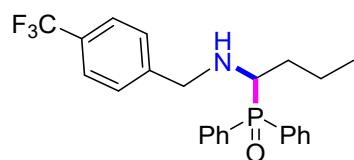
131.83, 131.81, 131.74, 131.66 – 131.58 (m), 131.32, 131.24, 130.77, 130.65, 129.49, 128.99, 128.86, 128.57, 128.44 (d, $J = 3.0$ Hz), 128.32, 113.67, 56.52 (d, $J = 82.3$ Hz), 55.26, 52.30 (d, $J = 7.3$ Hz), 31.71 (d, $J = 3.5$ Hz), 19.85 (d, $J = 10.1$ Hz), 14.02. ^{31}P NMR (162 MHz, CDCl_3) δ 31.27. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NO}_2\text{P}$: 394.1930; found: 394.1933.

(1-((4-fluorobenzyl)amino)butyl)diphenylphosphine oxide (4d)



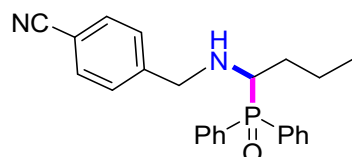
^1H NMR (400 MHz, CDCl_3) δ 7.98 – 7.90 (m, 2H), 7.89 – 7.82 (m, 2H), 7.59 – 7.44 (m, 6H), 7.09 – 7.01 (m, 2H), 6.97 – 6.90 (m, 2H), 3.63 (dd, $J = 55.8, 12.9$ Hz, 2H), 3.46 – 3.39 (m, 1H), 1.94 – 1.72 (m, 2H), 1.66 – 1.51 (m, 2H), 1.45 – 1.31 (m, 1H), 0.86 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.95 (d, $J = 244.9$ Hz), 135.46 (d, $J = 3.1$ Hz), 133.07, 132.45, 132.16, 131.71, 131.66, 131.63, 131.53, 131.27, 131.18, 129.85, 129.77, 128.60, 128.48 (d, $J = 1.4$ Hz), 128.37, 115.14, 114.93, 56.88, 56.07, 51.99 (d, $J = 7.2$ Hz), 31.66 (d, $J = 3.7$ Hz), 19.85 (d, $J = 10.1$ Hz), 13.98. ^{19}F NMR (376 MHz, CDCl_3) δ -115.72. ^{31}P NMR (162 MHz, CDCl_3) δ 31.21. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{26}\text{FNOP}$: 382.1731; found: 382.1729.

Diphenyl(1-((4-(trifluoromethyl)benzyl)amino)butyl)phosphine oxide (4e)



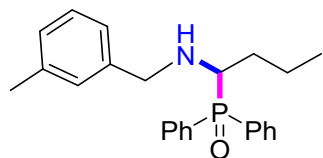
^1H NMR (400 MHz, CDCl_3) δ 7.96 – 7.88 (m, 2H), 7.88 – 7.80 (m, 2H), 7.57 – 7.45 (m, 8H), 7.20 (d, $J = 8.0$ Hz, 2H), 3.74 (dd, $J = 62.9, 13.5$ Hz, 2H), 3.43 (ddd, $J = 9.0, 5.3, 4.1$ Hz, 1H), 1.93 (s, 1H), 1.85 – 1.73 (m, 1H), 1.67 – 1.53 (m, 2H), 1.39 (ddd, $J = 13.9, 8.9, 4.1$ Hz, 1H), 0.87 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.90, 132.85, 132.44, 131.94, 131.75 (d, $J = 1.9$ Hz), 131.60, 131.52, 131.21, 131.12, 128.67, 128.56, 128.45, 128.42, 125.17 (q, $J = 3.8$ Hz), 56.56 (d, $J = 80.4$ Hz), 52.09 (d, $J = 7.2$ Hz), 31.62 (d, $J = 3.8$ Hz), 19.86 (d, $J = 10.0$ Hz), 13.97. ^{19}F NMR (376 MHz, CDCl_3) δ -62.42. ^{31}P NMR (162 MHz, CDCl_3) δ 31.39. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{26}\text{F}_3\text{NOP}$: 432.1699; found: 432.1701.

4-(((1-(diphenylphosphoryl)butyl)amino)methyl)benzonitrile (4f)



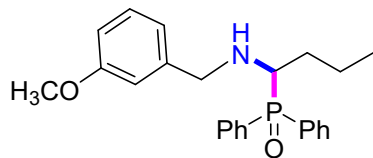
^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.86 (m, 2H), 7.86 – 7.79 (m, 2H), 7.56 – 7.43 (m, 8H), 7.18 (d, $J = 8.3$ Hz, 2H), 3.74 (dd, $J = 62.3, 14.0$ Hz, 2H), 3.42 (dt, $J = 8.8, 4.3$ Hz, 1H), 2.22 – 1.90 (m, 1H), 1.82 – 1.67 (m, 1H), 1.67 – 1.52 (m, 2H), 1.43 – 1.29 (m, 1H), 0.84 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 145.50, 132.64, 132.53, 132.03, 131.77 (t, $J = 3.0$ Hz), 131.60, 131.47, 131.39, 131.14, 131.05, 128.71, 128.59 (d, $J = 1.3$ Hz), 128.49, 118.89, 110.77, 56.58 (d, $J = 79.4$ Hz), 52.01 (d, $J = 6.8$ Hz), 31.55 (d, $J = 3.9$ Hz), 19.83 (d, $J = 10.1$ Hz), 13.94. ^{31}P NMR (162 MHz, CDCl_3) δ 31.51. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{26}\text{N}_2\text{OP}$: 389.1777; found: 389.1780.

(1-((3-methylbenzyl)amino)butyl)diphenylphosphine oxide (4g)



^1H NMR (400 MHz, CDCl_3) δ 8.02 – 7.93 (m, 2H), 7.93 – 7.84 (m, 2H), 7.58 – 7.45 (m, 6H), 7.17 (t, $J = 7.9$ Hz, 1H), 7.06 (d, $J = 7.6$ Hz, 1H), 6.92 (d, $J = 6.6$ Hz, 2H), 3.63 (dd, $J = 54.3, 12.7$ Hz, 2H), 3.46 (ddd, $J = 8.5, 6.1, 4.0$ Hz, 1H), 2.32 (s, 3H), 2.00 (s, 1H), 1.90 – 1.77 (m, 1H), 1.66 – 1.50 (m, 2H), 1.47 – 1.34 (m, 1H), 0.87 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.64, 137.89, 133.22, 132.30, 131.87, 131.78, 131.67 – 131.57 (m), 131.37, 131.28, 129.12, 128.58, 128.47, 128.44, 128.33, 128.19, 127.84, 125.33, 56.71 (d, $J = 82.0$ Hz), 52.88 (d, $J = 7.5$ Hz), 31.68 (d, $J = 3.5$ Hz), 21.37, 19.84 (d, $J = 10.1$ Hz), 14.02. ^{31}P NMR (162 MHz, CDCl_3) δ 31.24. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NOP}$: 378.1981; found: 378.1983.

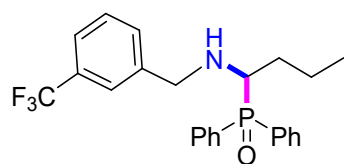
(1-((3-methoxybenzyl)amino)butyl)diphenylphosphine oxide (4h)



^1H NMR (400 MHz, CDCl_3) δ 8.01 – 7.92 (m, 2H), 7.91 – 7.84 (m, 2H), 7.58 – 7.44 (m, 6H), 7.18 (t, $J = 7.8$ Hz, 1H), 6.82 – 6.76 (m, 1H), 6.70 (t, $J = 5.3$ Hz, 2H), 3.75 (s, 3H), 3.63 (dd, $J = 50.8, 12.9$ Hz, 2H), 3.46 (ddd, $J = 8.6, 5.9, 4.0$ Hz, 1H), 1.82 (dddd, $J = 12.0, 10.0, 7.3, 3.9$ Hz, 1H), 1.58 (qdd, $J = 13.9, 8.1, 4.4$ Hz, 2H), 1.46 – 1.34 (m, 1H), 0.86 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3)

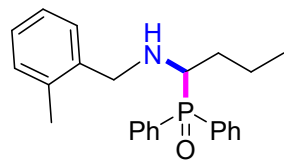
δ 159.63, 141.38, 133.22, 132.33 (d, $J = 3.1$ Hz), 131.82, 131.73, 131.63 (t, $J = 2.3$ Hz), 131.43, 131.32, 131.24, 129.26, 128.59, 128.47 (d, $J = 2.4$ Hz), 128.34, 120.59, 113.71, 112.75, 56.61 (d, $J = 81.9$ Hz), 55.15, 52.80 (d, $J = 7.3$ Hz), 31.68 (d, $J = 3.6$ Hz), 19.86 (d, $J = 10.1$ Hz), 14.03. ^{31}P NMR (162 MHz, CDCl_3) δ 31.13. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NO}_2\text{P}$: 394.1930; found: 394.1932.

Diphenyl(1-((3-(trifluoromethyl)benzyl)amino)butyl)phosphine oxide (4i)



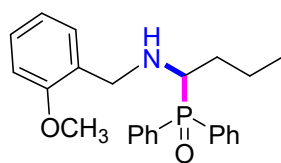
^1H NMR (400 MHz, CDCl_3) δ 7.97 – 7.88 (m, 2H), 7.87 – 7.79 (m, 2H), 7.54 – 7.40 (m, 7H), 7.33 (d, $J = 10.7$ Hz, 2H), 7.27 (t, $J = 7.4$ Hz, 1H), 3.71 (dd, $J = 62.1, 13.4$ Hz, 2H), 3.44 (dt, $J = 8.7, 4.4$ Hz, 1H), 2.43 (s, 1H), 1.83 – 1.68 (m, 1H), 1.66 – 1.52 (m, 2H), 1.45 – 1.29 (m, 1H), 0.83 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.79, 132.74, 132.32, 131.84, 131.80 – 131.70 (m), 131.53, 131.44, 131.42 – 131.37 (m), 131.15, 131.06, 130.66, 130.34, 128.66, 128.55, 128.45, 125.49, 124.95 (q, $J = 3.6$ Hz), 123.88 (d, $J = 3.7$ Hz), 122.79, 56.43 (d, $J = 80.1$ Hz), 52.07 (d, $J = 6.8$ Hz), 31.55 (d, $J = 3.7$ Hz), 19.78 (d, $J = 10.1$ Hz), 13.90. ^{19}F NMR (376 MHz, CDCl_3) δ -62.51. ^{31}P NMR (162 MHz, CDCl_3) δ 31.67. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{26}\text{F}_3\text{NOP}$: 432.1699; found: 432.1698.

(1-((2-methylbenzyl)amino)butyl)diphenylphosphine oxide (4j)



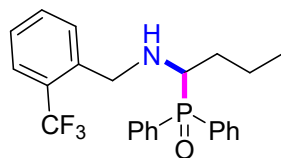
^1H NMR (400 MHz, CDCl_3) δ 7.99 – 7.91 (m, 2H), 7.88 – 7.81 (m, 2H), 7.58 – 7.44 (m, 6H), 7.20 – 7.07 (m, 4H), 3.65 (dd, $J = 63.2, 12.5$ Hz, 2H), 3.51 (ddd, $J = 8.5, 6.2, 4.1$ Hz, 1H), 2.10 (s, 3H), 1.93 – 1.78 (m, 2H), 1.60 (dddd, $J = 9.8, 7.2, 6.1, 3.8$ Hz, 2H), 1.47 – 1.39 (m, 1H), 0.89 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 137.59, 136.80, 133.31, 132.40, 132.26, 131.81, 131.72, 131.66 – 131.56 (m), 131.34, 131.28, 131.20, 130.30, 128.95, 128.59, 128.47 (d, $J = 3.0$ Hz), 128.34, 127.26, 125.78, 56.85 (d, $J = 81.9$ Hz), 50.82 (d, $J = 7.6$ Hz), 31.66 (d, $J = 3.7$ Hz), 19.90 (d, $J = 9.9$ Hz), 18.73, 14.04. ^{31}P NMR (162 MHz, CDCl_3) δ 31.48. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NOP}$: 378.1981; found: 378.1985.

(1-((2-methoxybenzyl)amino)butyl)diphenylphosphine oxide (4k)



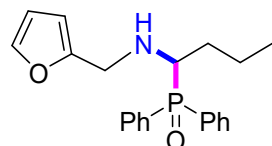
^1H NMR (400 MHz, CDCl_3) δ 8.10 – 8.01 (m, 2H), 7.96 – 7.88 (m, 2H), 7.57 – 7.43 (m, 6H), 7.28 – 7.20 (m, 1H), 6.99 (dd, $J = 7.3, 1.5$ Hz, 1H), 6.91 – 6.80 (m, 2H), 3.76 (s, 3H), 3.54 (q, $J = 13.0$ Hz, 2H), 3.40 (ddd, $J = 9.8, 6.2, 3.9$ Hz, 1H), 1.96 – 1.78 (m, 2H), 1.56 – 1.43 (m, 2H), 1.35 – 1.21 (m, 1H), 0.81 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.61, 133.46, 132.55, 131.92, 131.63, 131.54, 131.50 (d, $J = 2.5$ Hz), 130.99, 130.11, 128.46, 128.34, 128.26, 128.15, 127.78, 120.34, 110.19, 57.47 (d, $J = 86.6$ Hz), 55.06, 48.81 (d, $J = 7.3$ Hz), 31.77 (d, $J = 2.4$ Hz), 19.51 (d, $J = 10.6$ Hz), 13.95. ^{31}P NMR (162 MHz, CDCl_3) δ 30.25. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NO}_2\text{P}$: 394.1930; found: 394.1935.

Diphenyl(1-(2-(trifluoromethyl)benzyl)amino)butylphosphine oxide (4l)



^1H NMR (400 MHz, CDCl_3) δ 8.01 – 7.92 (m, 2H), 7.90 – 7.82 (m, 2H), 7.57 – 7.44 (m, 7H), 7.39 (t, $J = 7.4$ Hz, 1H), 7.29 (t, $J = 7.2$ Hz, 2H), 3.79 (dd, $J = 83.4, 14.0$ Hz, 2H), 3.50 (ddd, $J = 9.1, 5.5, 4.0$ Hz, 1H), 2.39 (s, 1H), 1.88 – 1.75 (m, 1H), 1.66 – 1.48 (m, 2H), 1.46 – 1.29 (m, 1H), 0.84 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 138.45, 132.87, 132.03, 131.96, 131.82, 131.75, 131.73, 131.67, 131.27, 131.18, 131.10, 130.58, 128.66, 128.55, 128.50, 128.39, 128.09, 127.79, 126.96, 125.62 (dd, $J = 11.9, 6.1$ Hz), 122.99, 57.57 (d, $J = 82.5$ Hz), 49.06 (d, $J = 7.5$ Hz), 31.74 (d, $J = 3.5$ Hz), 19.66 (d, $J = 10.1$ Hz), 13.93. ^{19}F NMR (376 MHz, CDCl_3) δ -59.42. ^{31}P NMR (162 MHz, CDCl_3) δ 31.55. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{26}\text{F}_3\text{NOP}$: 432.1699; found: 432.1698.

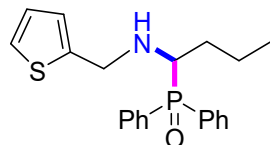
(1-((furan-2-ylmethyl)amino)butyl)diphenylphosphine oxide (4m)



^1H NMR (400 MHz, CDCl_3) δ 8.04 – 7.95 (m, 2H), 7.94 – 7.84 (m, 2H), 7.56 – 7.44 (m, 6H), 7.34 (d, $J = 1.1$ Hz, 1H), 6.29 (dd, $J = 3.0, 1.9$ Hz, 1H), 6.05 (d, $J = 3.0$ Hz, 1H), 3.55 (dd, $J = 49.8, 14.5$ Hz, 2H), 3.46 – 3.37 (m, 1H), 2.01 – 1.75 (m, 2H), 1.55 – 1.39 (m, 2H), 1.27 (dd, $J = 16.1, 6.5$ Hz, 1H), 0.81 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 153.15, 141.91, 133.02, 132.10, 131.94, 131.86, 131.76 – 131.59 (m), 131.46, 131.38, 130.99, 128.56, 128.43 (d, $J = 3.5$ Hz), 128.31,

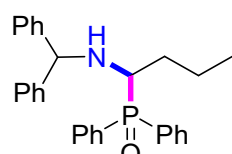
110.13, 107.54, 56.28 (d, $J = 84.4$ Hz), 45.12 (d, $J = 7.0$ Hz), 31.78 (d, $J = 2.8$ Hz), 19.60 (d, $J = 10.4$ Hz), 13.92. ^{31}P NMR (162 MHz, CDCl_3) δ 30.41. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{21}\text{H}_{25}\text{NO}_2\text{P}$: 354.1617; found: 354.1620.

Diphenyl(1-((thiophen-2-ylmethyl)amino)butyl)phosphine oxide (4n)



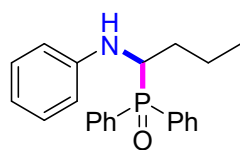
^1H NMR (400 MHz, CDCl_3) δ 8.05 – 7.97 (m, 2H), 7.95 – 7.88 (m, 2H), 7.57 – 7.45 (m, 6H), 7.21 (dd, $J = 5.1, 1.1$ Hz, 1H), 6.92 (dd, $J = 5.1, 3.5$ Hz, 1H), 6.79 – 6.75 (m, 1H), 3.84 (dd, $J = 69.2, 13.7$ Hz, 2H), 3.50 (ddd, $J = 9.5, 6.0, 3.8$ Hz, 1H), 1.92 – 1.77 (m, 2H), 1.66 – 1.47 (m, 2H), 1.44 – 1.34 (m, 1H), 0.87 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.47, 133.06, 132.13 (d, $J = 2.9$ Hz), 131.90, 131.82, 131.68 (dd, $J = 4.6, 2.8$ Hz), 131.37, 131.29, 131.19, 128.61, 128.49 (d, $J = 2.6$ Hz), 128.37, 126.46, 125.03, 124.70, 56.65 (d, $J = 81.9$ Hz), 47.52 (d, $J = 7.2$ Hz), 31.75 (d, $J = 3.4$ Hz), 19.85 (d, $J = 10.2$ Hz), 13.99. ^{31}P NMR (162 MHz, CDCl_3) δ 30.47. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{21}\text{H}_{25}\text{NOPS}$: 370.1389; found: 370.1392.

(1-(benzhydrylamino)butyl)diphenylphosphine oxide (4o)



^1H NMR (400 MHz, CDCl_3) δ 7.95 – 7.87 (m, 2H), 7.80 – 7.73 (m, 2H), 7.64 – 7.41 (m, 7H), 7.30 – 7.21 (m, 6H), 7.21 – 7.16 (m, 3H), 7.09 (dd, $J = 6.7, 2.9$ Hz, 2H), 4.73 (s, 1H), 3.43 (dt, $J = 9.5, 4.7$ Hz, 1H), 1.89 (s, 2H), 1.84 – 1.69 (m, 1H), 1.65 – 1.52 (m, 2H), 1.37 – 1.29 (m, 1H), 0.78 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.25, 143.00, 133.28, 132.91, 132.36, 132.01, 131.61, 131.52, 131.16, 131.07, 128.72, 128.60, 128.50, 128.43, 128.39, 128.35, 127.51, 127.41, 127.24, 127.11, 64.65 (d, $J = 7.6$ Hz), 53.59 (d, $J = 78.0$ Hz), 31.87 (d, $J = 3.8$ Hz), 19.45 (d, $J = 7.8$ Hz), 14.16. ^{31}P NMR (162 MHz, CDCl_3) δ 31.71. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{29}\text{H}_{31}\text{NOP}$: 440.2138; found: 440.2142.

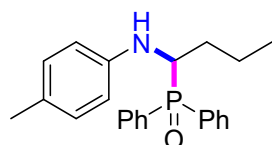
Diphenyl(1-(phenylamino)butyl)phosphine oxide (5a)



^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.86 (m, 2H), 7.80 (ddd, $J = 10.9, 8.2, 1.2$ Hz, 2H), 7.59 – 7.47 (m, 3H), 7.47 – 7.41 (m, 1H), 7.41 – 7.33 (m, 2H), 7.09 (t, $J = 7.9$ Hz, 2H), 6.67 (t, $J = 7.3$ Hz, 1H), 6.55 (d, $J = 7.8$ Hz, 2H), 4.33 (ddd, $J =$

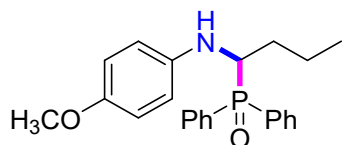
= 13.5, 10.6, 3.7 Hz, 1H), 4.03 (s, 1H), 1.97 – 1.81 (m, 1H), 1.75 – 1.48 (m, 2H), 1.38 – 1.29 (m, 1H), 0.83 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.24 (d, $J = 6.7$ Hz), 131.99 (d, $J = 2.7$ Hz), 131.81 (d, $J = 2.7$ Hz), 131.48, 131.40, 131.31, 131.22, 129.23, 128.79, 128.68, 128.47, 128.36, 117.91, 113.22, 52.57 (d, $J = 80.3$ Hz), 32.57 (d, $J = 4.1$ Hz), 19.65 (d, $J = 10.1$ Hz), 13.98. ^{31}P NMR (162 MHz, CDCl_3) δ 31.63. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{22}\text{H}_{25}\text{NOP}$: 350.1668; found: 350.1670.

Diphenyl(1-(*p*-tolylamino)butyl)phosphine oxide (5b)



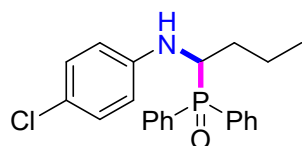
^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.86 (m, 2H), 7.85 – 7.77 (m, 2H), 7.57 – 7.44 (m, 4H), 7.42 – 7.35 (m, 2H), 6.91 (d, $J = 8.2$ Hz, 2H), 6.47 (d, $J = 8.4$ Hz, 2H), 4.40 – 4.20 (m, 1H), 3.86 (s, 1H), 2.22 (s, 3H), 1.94 – 1.83 (m, 1H), 1.69 – 1.47 (m, 2H), 1.38 – 1.29 (m, 1H), 0.82 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 144.91 (d, $J = 7.5$ Hz), 131.94 (d, $J = 2.7$ Hz), 131.76 (d, $J = 2.7$ Hz), 131.55, 131.46, 131.35, 131.26, 129.74, 128.76, 128.65, 128.47, 128.36, 127.14, 113.36, 52.92 (d, $J = 81.3$ Hz), 32.68 (d, $J = 4.2$ Hz), 20.35, 19.68 (d, $J = 10.0$ Hz), 13.99. ^{31}P NMR (162 MHz, CDCl_3) δ 31.63. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NOP}$: 364.1825; found: 364.1830.

(1-((4-methoxyphenyl)amino)butyl)diphenylphosphine oxide (5c)



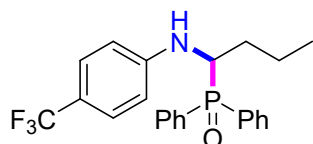
^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.85 (m, 2H), 7.83 – 7.77 (m, 2H), 7.56 – 7.42 (m, 4H), 7.40 – 7.33 (m, 2H), 6.71 – 6.65 (m, 2H), 6.53 – 6.46 (m, 2H), 4.21 (s, 1H), 3.85 – 3.75 (m, 1H), 3.72 (s, 3H), 1.94 – 1.79 (m, 1H), 1.69 – 1.48 (m, 2H), 1.37 – 1.29 (m, 1H), 0.82 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 152.31, 141.44 (d, $J = 7.2$ Hz), 131.96 (d, $J = 2.8$ Hz), 131.78 (d, $J = 2.7$ Hz), 131.53, 131.44, 131.31, 131.22, 128.78, 128.66, 128.45, 128.33, 114.81, 114.69, 55.73, 53.94 (d, $J = 80.7$ Hz), 32.63 (d, $J = 4.3$ Hz), 19.69 (d, $J = 10.2$ Hz), 13.97. ^{31}P NMR (162 MHz, CDCl_3) δ 31.75. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_2\text{P}$: 380.1774; found: 380.1776.

(1-((4-chlorophenyl)amino)butyl)diphenylphosphine oxide (5d)



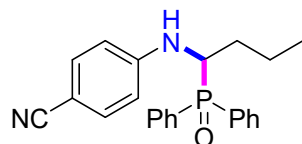
^1H NMR (400 MHz, CDCl_3) δ 7.91 – 7.84 (m, 2H), 7.80 – 7.73 (m, 2H), 7.58 – 7.47 (m, 3H), 7.46 – 7.39 (m, 1H), 7.38 – 7.31 (m, 2H), 7.03 – 6.96 (m, 2H), 6.50 – 6.42 (m, 2H), 4.38 – 4.19 (m, 2H), 1.88 – 1.76 (m, 1H), 1.74 – 1.61 (m, 1H), 1.60 – 1.46 (m, 1H), 1.37 – 1.29 (m, 1H), 0.81 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 146.03 (d, $J = 6.1$ Hz), 132.13 (d, $J = 2.7$ Hz), 131.92 (d, $J = 2.7$ Hz), 131.61 (d, $J = 4.9$ Hz), 131.32, 131.23, 131.22, 131.13, 130.67, 128.98, 128.87, 128.76, 128.53, 128.42, 122.14, 114.17, 52.86 (d, $J = 79.7$ Hz), 32.36 (d, $J = 4.2$ Hz), 19.62 (d, $J = 10.4$ Hz), 13.92. ^{31}P NMR (162 MHz, CDCl_3) δ 31.90. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{22}\text{H}_{24}\text{ClNOP}$: 384.1279; found: 384.1283.

Diphenyl(1-((4-(trifluoromethyl)phenyl)amino)butyl)phosphine oxide (5e)



^1H NMR (400 MHz, CDCl_3) δ 7.92 – 7.85 (m, 2H), 7.79 – 7.72 (m, 2H), 7.61 – 7.51 (m, 3H), 7.45 – 7.39 (m, 1H), 7.38 – 7.26 (m, 4H), 6.56 (d, $J = 8.6$ Hz, 2H), 4.78 (d, $J = 8.2$ Hz, 1H), 4.34 (tdd, $J = 9.8, 5.6, 3.9$ Hz, 1H), 1.87 – 1.67 (m, 2H), 1.64 – 1.48 (m, 1H), 1.39 – 1.31 (m, 1H), 0.84 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 150.05 (d, $J = 5.5$ Hz), 132.25 (d, $J = 2.7$ Hz), 132.04 (d, $J = 2.7$ Hz), 131.23 (d, $J = 2.5$ Hz), 131.14 (d, $J = 2.3$ Hz), 128.96, 128.84, 128.59, 128.48, 126.64 – 126.44 (m), 119.13 (q, $J = 32.7$ Hz), 112.18, 52.17 (d, $J = 79.0$ Hz), 32.18 (d, $J = 3.9$ Hz), 19.58 (d, $J = 10.4$ Hz), 13.87. ^{31}P NMR (162 MHz, CDCl_3) δ 31.91. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{24}\text{F}_3\text{NOP}$: 418.1542; found: 418.1544.

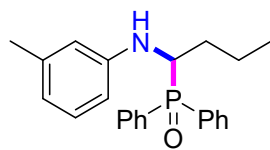
4-((1-(diphenylphosphoryl)butyl)amino)benzonitrile (5f)



^1H NMR (400 MHz, CDCl_3) δ 7.92 – 7.84 (m, 2H), 7.76 – 7.68 (m, 2H), 7.62 – 7.52 (m, 3H), 7.46 – 7.40 (m, 1H), 7.36 – 7.26 (m, 4H), 6.53 (d, $J = 8.8$ Hz, 2H), 5.31 (d, $J = 8.7$ Hz, 1H), 4.32 (ddd, $J = 15.1, 10.4, 4.9$ Hz, 1H), 1.77 (dt, $J = 14.9, 7.3$ Hz, 2H), 1.55 (dt, $J = 20.9, 7.2$ Hz, 1H), 1.40 – 1.29 (m, 1H), 0.84 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 150.98 (d, $J = 4.6$ Hz), 133.56, 132.38 (d, $J = 2.7$ Hz), 132.13, 131.15, 131.07 (d, $J = 1.9$ Hz), 129.04, 128.93, 128.62, 128.51, 120.22, 112.43, 99.03, 52.01 (d, $J = 78.4$ Hz), 31.91 (d, $J = 3.7$ Hz), 19.55 (d, $J = 10.6$ Hz),

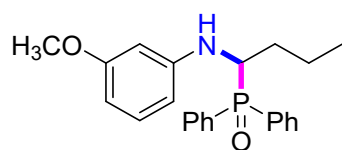
13.82. ^{31}P NMR (162 MHz, CDCl_3) δ 31.64. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{24}\text{N}_2\text{OP}$: 375.1621; found: 375.1625.

Diphenyl(1-(*m*-tolylamino)butyl)phosphine oxide (5g)



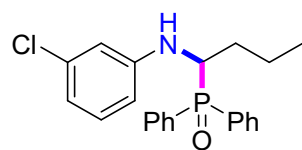
^1H NMR (400 MHz, CDCl_3) δ 7.95 – 7.87 (m, 2H), 7.85 – 7.77 (m, 2H), 7.57 – 7.47 (m, 3H), 7.46 – 7.41 (m, 1H), 7.40 – 7.32 (m, 2H), 6.98 (t, $J = 7.7$ Hz, 1H), 6.49 (d, $J = 7.4$ Hz, 1H), 6.41 – 6.30 (m, 2H), 4.32 (dt, $J = 9.4, 6.9$ Hz, 1H), 4.01 (d, $J = 8.6$ Hz, 1H), 2.21 (s, 3H), 1.96 – 1.83 (m, 1H), 1.69 – 1.60 (m, 1H), 1.59 – 1.48 (m, 1H), 1.37 – 1.28 (m, 1H), 0.82 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.22 (d, $J = 6.8$ Hz), 138.95, 131.98 (d, $J = 2.6$ Hz), 131.80 (d, $J = 2.5$ Hz), 131.51, 131.42, 131.32, 131.23, 129.05, 128.78, 128.67, 128.45, 128.34, 118.86, 114.16, 110.24, 52.58 (d, $J = 80.8$ Hz), 32.56 (d, $J = 4.1$ Hz), 21.56, 19.63 (d, $J = 10.1$ Hz), 14.00. ^{31}P NMR (162 MHz, CDCl_3) δ 31.74. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NOP}$: 364.1825; found: 364.1829.

(1-((3-methoxyphenyl)amino)butyl)diphenylphosphine oxide (5h)



^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.85 (m, 2H), 7.84 – 7.75 (m, 2H), 7.60 – 7.41 (m, 4H), 7.40 – 7.34 (m, 2H), 7.00 (t, $J = 8.1$ Hz, 1H), 6.21 (ddd, $J = 20.3, 8.1, 2.1$ Hz, 2H), 6.10 (t, $J = 2.2$ Hz, 1H), 4.38 – 4.18 (m, 2H), 3.72 (s, 3H), 1.92 – 1.78 (m, 1H), 1.75 – 1.49 (m, 2H), 1.40 – 1.30 (m, 1H), 0.83 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 160.70, 148.73 (d, $J = 6.7$ Hz), 132.05 (d, $J = 2.7$ Hz), 131.86 (d, $J = 2.7$ Hz), 131.43, 131.34, 131.28, 131.19, 130.89, 130.75, 129.96, 128.83, 128.72, 128.50, 128.39, 106.28, 102.94, 99.31, 55.07, 52.49 (d, $J = 80.2$ Hz), 32.49 (d, $J = 4.2$ Hz), 19.61 (d, $J = 10.2$ Hz), 13.99. ^{31}P NMR (162 MHz, CDCl_3) δ 32.25. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_2\text{P}$: 380.1774; found: 380.1775.

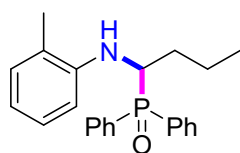
(1-((3-chlorophenyl)amino)butyl)diphenylphosphine oxide (5i)



^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.85 (m, 2H), 7.82 – 7.73 (m, 2H), 7.59 – 7.49 (m, 3H), 7.47 – 7.41 (m, 1H), 7.40 – 7.33 (m, 2H), 6.96 (t, $J = 8.0$ Hz, 1H), 6.62 – 6.56 (m, 1H), 6.49 (s, 1H), 6.41 (dd, $J = 8.2, 2.0$ Hz, 1H), 4.35 (s, 1H),

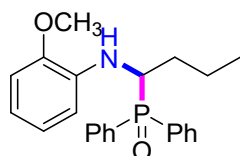
4.30 – 4.21 (m, 1H), 1.82 (tdd, $J = 13.9, 6.4, 3.6$ Hz, 1H), 1.76 – 1.63 (m, 1H), 1.61 – 1.49 (m, 1H), 1.39 – 1.30 (m, 1H), 0.84 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 148.52 (d, $J = 5.6$ Hz), 134.87, 132.15 (d, $J = 2.7$ Hz), 131.97 (d, $J = 2.7$ Hz), 131.32, 131.23, 131.14, 130.13, 128.89, 128.78, 128.53, 128.42, 117.63, 112.68, 111.49, 52.53 (d, $J = 79.3$ Hz), 32.24 (d, $J = 4.0$ Hz), 19.58 (d, $J = 10.3$ Hz), 13.93. ^{31}P NMR (162 MHz, CDCl_3) δ 31.67. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{22}\text{H}_{24}\text{ClNOP}$: 384.1279; found: 384.1280.

Diphenyl(1-(*o*-tolylamino)butyl)phosphine oxide (5j)



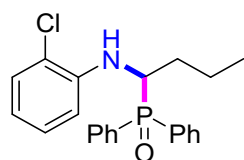
^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.85 (m, 2H), 7.84 – 7.74 (m, 2H), 7.60 – 7.42 (m, 4H), 7.39 – 7.32 (m, 2H), 7.08 – 6.93 (m, 2H), 6.61 (dd, $J = 12.1, 7.7$ Hz, 2H), 4.46 – 4.35 (m, 1H), 3.94 – 3.80 (m, 1H), 2.03 (s, 3H), 1.97 – 1.88 (m, 1H), 1.76 – 1.63 (m, 1H), 1.54 (tdd, $J = 12.0, 7.4, 4.7$ Hz, 1H), 1.41 – 1.31 (m, 1H), 0.84 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 145.05 (d, $J = 6.4$ Hz), 131.98 (d, $J = 2.5$ Hz), 131.83 (d, $J = 2.8$ Hz), 131.44, 131.35, 131.25, 131.16, 130.39, 128.80, 128.69, 128.39, 128.28, 126.92, 122.40, 117.44, 110.27, 52.37 (d, $J = 79.4$ Hz), 32.66 (d, $J = 4.0$ Hz), 19.64 (d, $J = 9.7$ Hz), 17.49, 14.04. ^{31}P NMR (162 MHz, CDCl_3) δ 31.94. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NOP}$: 364.1825; found: 364.1826.

(1-((2-methoxyphenyl)amino)butyl)diphenylphosphine oxide (5k)



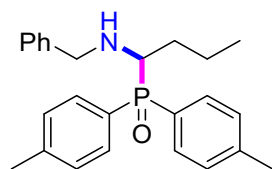
^1H NMR (400 MHz, CDCl_3) δ 7.95 – 7.80 (m, 4H), 7.58 – 7.42 (m, 4H), 7.41 – 7.34 (m, 2H), 6.79 – 6.67 (m, 2H), 6.62 (td, $J = 7.7, 1.4$ Hz, 1H), 6.52 (d, $J = 7.2$ Hz, 1H), 4.53 (dd, $J = 10.5, 3.2$ Hz, 1H), 4.30 (ddd, $J = 18.6, 10.3, 3.4$ Hz, 1H), 3.77 (s, 3H), 2.02 – 1.88 (m, 1H), 1.71 – 1.46 (m, 2H), 1.37 – 1.28 (m, 1H), 0.84 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 146.89, 137.21 (d, $J = 6.5$ Hz), 131.86 (d, $J = 2.6$ Hz), 131.73, 131.69, 131.65, 131.37, 131.29, 128.69, 128.58, 128.26, 128.15, 120.98, 117.02, 110.33, 109.80, 55.46, 52.94 (d, $J = 81.1$ Hz), 32.73 (d, $J = 4.1$ Hz), 19.60 (d, $J = 10.5$ Hz), 13.95. ^{31}P NMR (162 MHz, CDCl_3) δ 30.86. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{27}\text{NO}_2\text{P}$: 380.1774; found: 380.1778.

(1-((2-chlorophenyl)amino)butyl)diphenylphosphine oxide (5l)



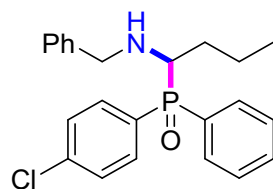
^1H NMR (400 MHz, CDCl_3) δ 7.96 – 7.81 (m, 4H), 7.59 – 7.45 (m, 4H), 7.44 – 7.37 (m, 2H), 7.18 (dd, $J = 7.8, 1.3$ Hz, 1H), 7.03 – 6.97 (m, 1H), 6.63 – 6.54 (m, 2H), 4.52 (dd, $J = 10.3, 2.6$ Hz, 1H), 4.34 (ddd, $J = 18.7, 10.2, 3.5$ Hz, 1H), 2.06 – 1.94 (m, 1H), 1.73 – 1.60 (m, 1H), 1.59 – 1.46 (m, 1H), 1.41 – 1.29 (m, 1H), 0.86 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.00 (d, $J = 6.0$ Hz), 132.09 (d, $J = 2.7$ Hz), 132.00 (d, $J = 2.7$ Hz), 131.73, 131.64, 131.37, 131.28, 130.98, 130.77, 130.03, 129.35, 128.80, 128.69, 128.50, 128.39, 127.59, 119.62, 117.90, 111.71, 53.08 (d, $J = 80.4$ Hz), 32.60 (d, $J = 3.8$ Hz), 19.60 (d, $J = 10.4$ Hz), 13.91. ^{31}P NMR (162 MHz, CDCl_3) δ 30.39. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{22}\text{H}_{24}\text{ClNOP}$: 384.1279; found: 384.1281.

(1-(benzylamino)butyl)di-p-tolylphosphine oxide (6a)



^1H NMR (400 MHz, CDCl_3) δ 7.83 (dd, $J = 10.3, 8.1$ Hz, 2H), 7.74 (dd, $J = 10.4, 8.1$ Hz, 2H), 7.32 – 7.25 (m, 7H), 7.17 – 7.11 (m, 2H), 3.65 (dd, $J = 42.3, 12.8$ Hz, 2H), 3.41 (ddd, $J = 9.5, 6.0, 3.9$ Hz, 1H), 2.41 (d, $J = 6.1$ Hz, 6H), 2.09 – 1.98 (m, 1H), 1.81 (ddt, $J = 13.8, 8.3, 6.0$ Hz, 1H), 1.64 – 1.48 (m, 2H), 1.44 – 1.32 (m, 1H), 0.85 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 141.96 (t, $J = 3.0$ Hz), 139.87, 131.80, 131.72, 131.32, 131.24, 129.91, 129.32, 129.20, 129.08, 129.03, 128.98, 128.69, 128.33, 128.27, 128.08, 127.06, 56.73 (d, $J = 81.8$ Hz), 52.88 (d, $J = 7.1$ Hz), 31.68 (d, $J = 3.5$ Hz), 21.59, 19.86 (d, $J = 10.2$ Hz), 14.02. ^{31}P NMR (162 MHz, CDCl_3) δ 31.89. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{25}\text{H}_{31}\text{NOP}$: 392.2138; found: 392.2136.

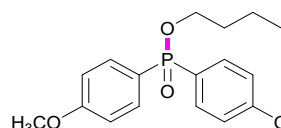
(1-(benzylamino)butyl)bis(4-chlorophenyl)phosphine oxide (6b)



^1H NMR (400 MHz, CDCl_3) δ 7.87 (dd, $J = 10.0, 8.5$ Hz, 2H), 7.79 (dd, $J = 10.2, 8.4$ Hz, 2H), 7.46 (dd, $J = 8.4, 2.1$ Hz, 4H), 7.29 (q, $J = 5.5$ Hz, 3H), 7.15 – 7.09 (m, 2H), 3.66 (dd, $J = 59.1, 12.8$ Hz, 2H), 3.40 (td, $J = 7.9, 4.1$ Hz, 1H), 2.01 (s, 1H), 1.93 – 1.79 (m, 1H), 1.63 – 1.47 (m, 2H), 1.37 (ddd, $J = 14.4, 11.3, 7.3$

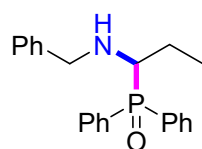
Hz, 1H), 0.88 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.24, 138.48 (dd, $J = 5.8, 3.3$ Hz), 133.28, 133.19, 132.75, 132.65, 131.34, 130.41, 130.17, 129.23, 129.03, 128.89 (d, $J = 4.7$ Hz), 128.75, 128.45, 128.30, 127.35, 56.80 (d, $J = 85.7$ Hz), 53.16 (d, $J = 8.5$ Hz), 31.73 (d, $J = 3.5$ Hz), 19.81 (d, $J = 10.0$ Hz), 14.03. ^{31}P NMR (162 MHz, CDCl_3) δ 29.90. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{23}\text{H}_{25}\text{Cl}_2\text{NOP}$: 432.1045; found: 432.1048.

butyl bis(4-methoxyphenyl)phosphinate (D)



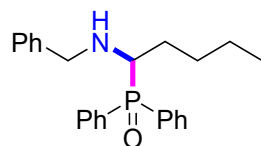
^1H NMR (400 MHz, CDCl_3) δ 7.74 – 7.65 (m, 4H), 6.95 – 6.87 (m, 4H), 3.95 (q, $J = 6.6$ Hz, 2H), 3.81 (s, 6H), 1.70 – 1.61 (m, 2H), 1.44 – 1.34 (m, 2H), 0.88 (t, $J = 7.4$ Hz, 3H). HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{18}\text{H}_{24}\text{O}_4\text{P}$: 335.1407; found: 335.1410.

(1-(benzylamino)propyl)diphenylphosphine oxide (6e)



^1H NMR (400 MHz, CDCl_3) δ 8.00 – 7.92 (m, 2H), 7.91 – 7.83 (m, 2H), 7.56 – 7.44 (m, 6H), 7.27 (dt, $J = 14.6, 4.9$ Hz, 3H), 7.17 – 7.09 (m, 2H), 3.68 (dd, $J = 53.5, 12.9$ Hz, 2H), 3.39 (ddd, $J = 8.1, 6.3, 4.3$ Hz, 1H), 2.11 (s, 1H), 2.01 – 1.86 (m, 1H), 1.62 (td, $J = 15.3, 7.8$ Hz, 1H), 1.02 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.66, 133.17, 132.31 (d, $J = 9.7$ Hz), 131.78, 131.70, 131.66 – 131.58 (m), 131.43, 131.30, 131.22, 128.58, 128.47, 128.44, 128.33, 128.30, 127.12, 57.98 (d, $J = 82.0$ Hz), 52.67 (d, $J = 8.0$ Hz), 22.37 (d, $J = 3.8$ Hz), 11.14 (d, $J = 9.7$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 31.12 (d, $J = 2.2$ Hz). HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{22}\text{H}_{25}\text{NOP}$: 350.1668; found: 350.1665.

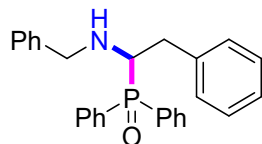
(1-(benzylamino)pentyl)diphenylphosphine oxide (6f)



^1H NMR (400 MHz, CDCl_3) δ 8.02 – 7.92 (m, 2H), 7.92 – 7.84 (m, 2H), 7.54 – 7.42 (m, 6H), 7.31 – 7.22 (m, 3H), 7.16 – 7.08 (m, 2H), 3.64 (dd, $J = 48.0, 12.8$ Hz, 2H), 3.48 – 3.38 (m, 1H), 1.86 (ddt, $J = 11.8, 10.3, 4.0$ Hz, 1H), 1.66 – 1.47 (m, 3H), 1.39 – 1.20 (m, 3H), 0.82 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.71, 133.21, 132.35 (d, $J = 8.4$ Hz), 131.95 – 131.10 (m), 128.47 (dt, $J = 14.5, 8.7$

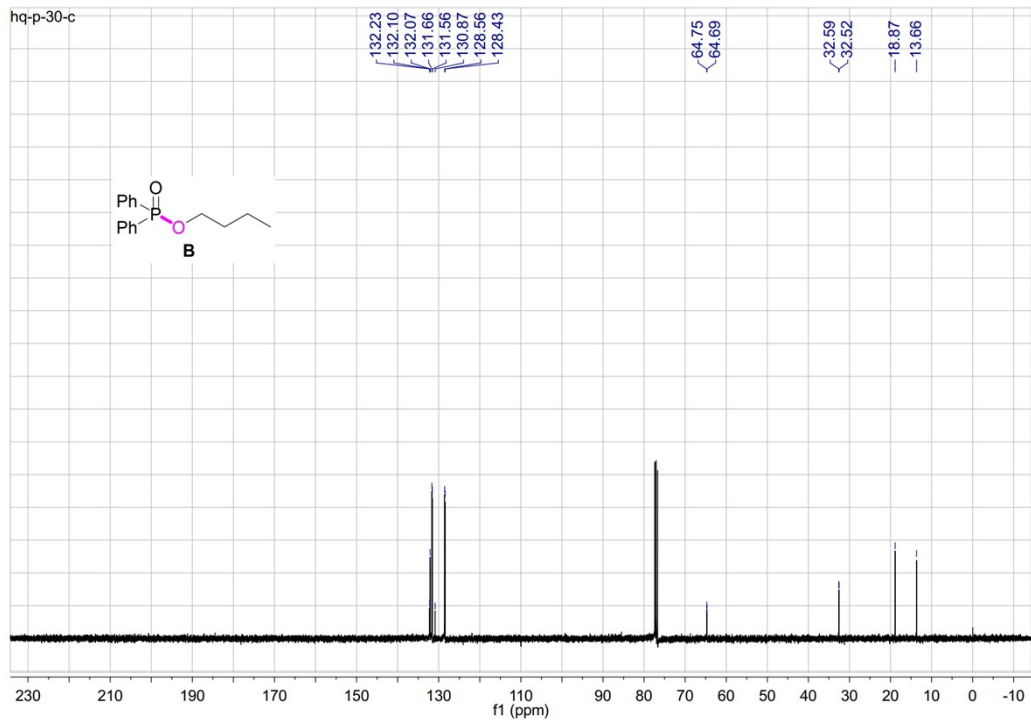
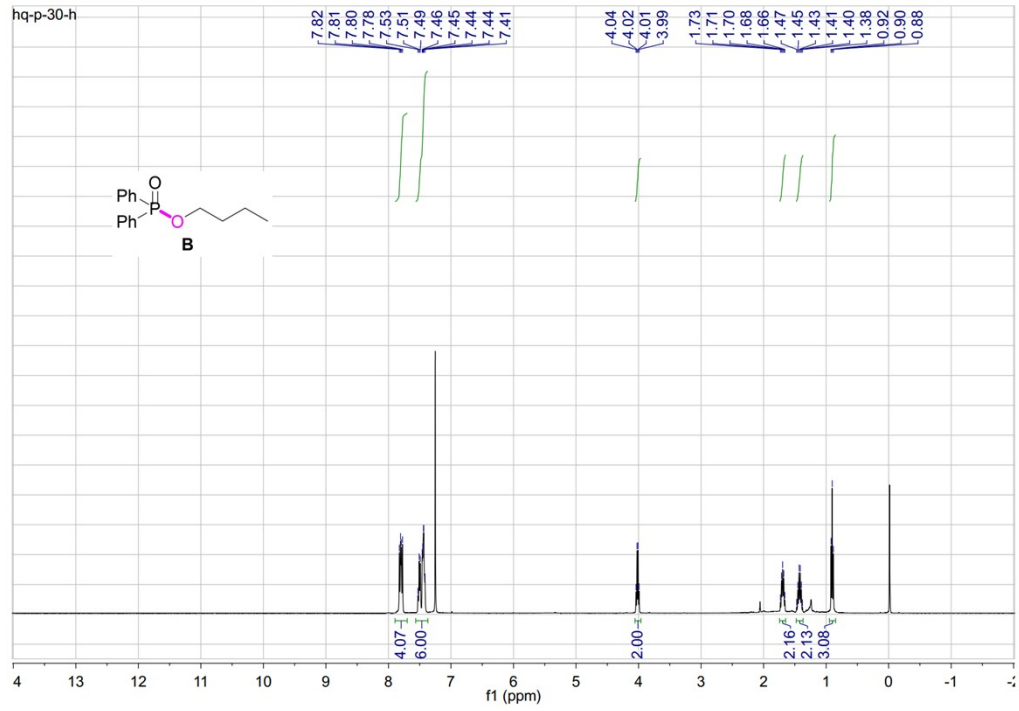
Hz), 127.11, 57.20, 56.39, 52.83 (d, $J = 7.3$ Hz), 29.18 (d, $J = 3.6$ Hz), 28.70 (d, $J = 9.6$ Hz), 22.58, 13.91. ^{31}P NMR (162 MHz, CDCl_3) δ 31.06. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{24}\text{H}_{29}\text{NOP}$: 378.1981; found: 378.1985.

(1-(benzylamino)-2-phenylethyl)diphenylphosphine oxide (6h)



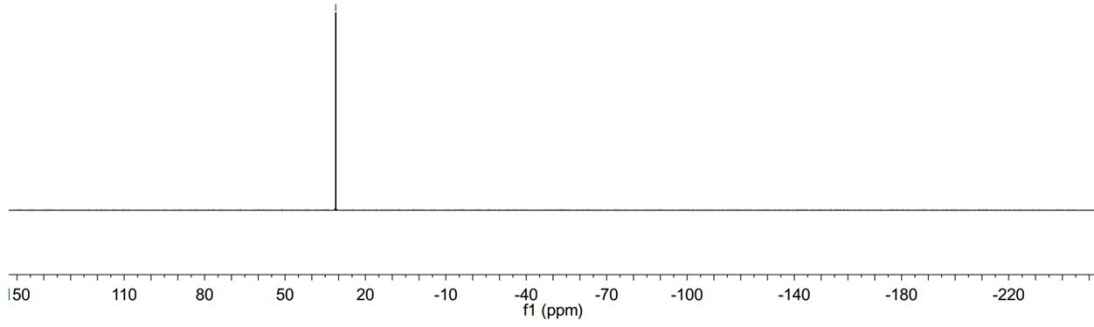
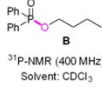
^1H NMR (400 MHz, CDCl_3) δ 8.06 – 7.98 (m, 2H), 7.90 – 7.87 (m, 2H), 7.53 – 7.44 (m, 6H), 7.31-7.25 (m, 5H), 7.20 – 7.14 (m, 5H), 4.46 (d, $J = 6.0$ Hz, 1H), 3.74 – 3.69 (m, 1H), 3.37 – 3.20 (m, 3H), 2.74 (ddd, $J = 14.2, 10.6, 8.0$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.21, 138.41 (d, $J = 12.9$ Hz), 131.99, 131.91, 131.82 (d, $J = 2.5$ Hz), 131.39, 131.31, 129.29, 128.71, 128.63, 128.59, 128.50, 128.39, 128.32, 128.26, 128.20, 127.75, 127.01, 126.67, 59.16 (d, $J = 84.4$ Hz), 53.32 (d, $J = 6.8$ Hz), 36.08 (d, $J = 4.6$ Hz). ^{31}P NMR (162 MHz, CDCl_3) δ 30.58. HRMS: $[\text{M}+\text{H}]^+$ m/z calcd for $\text{C}_{27}\text{H}_{27}\text{NOP}$: 412.1825; found: 412.1830.

4. NMR spectra for products 4, 5, 6

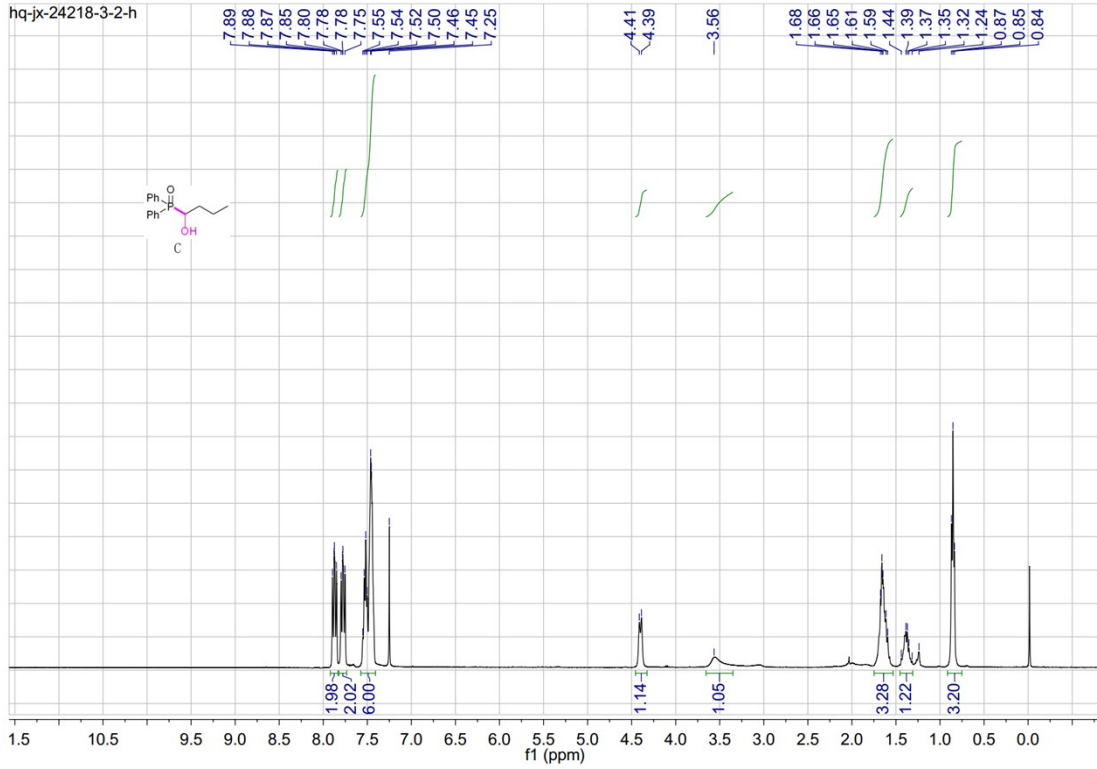


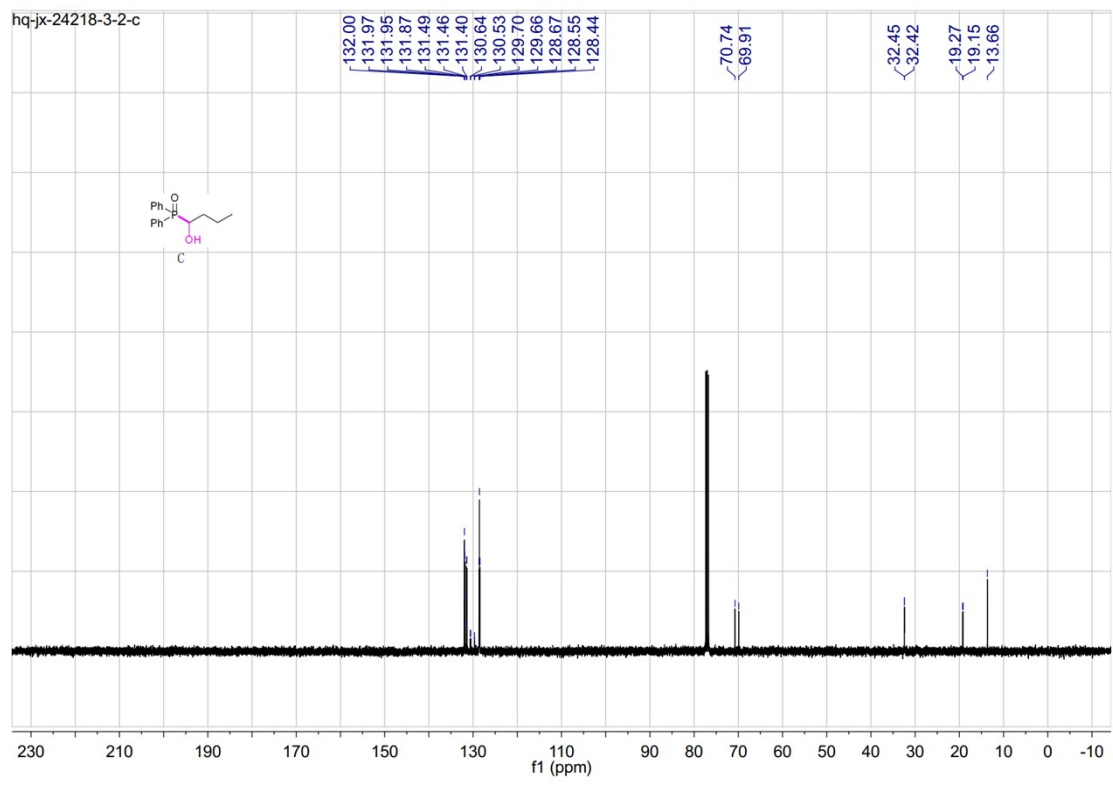
Mar18-2024 HQ-LY01

-31.08



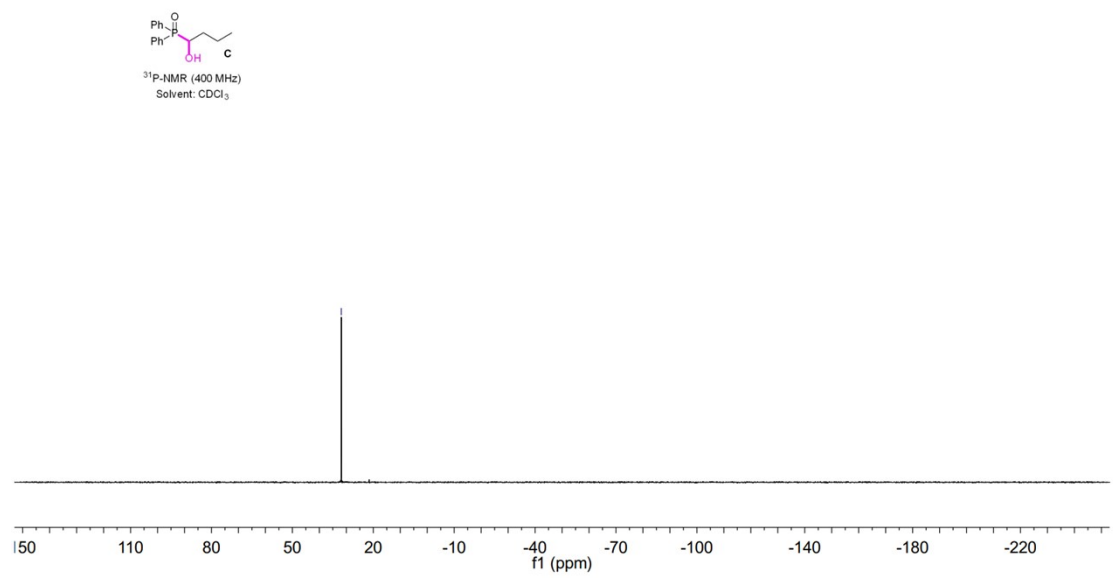
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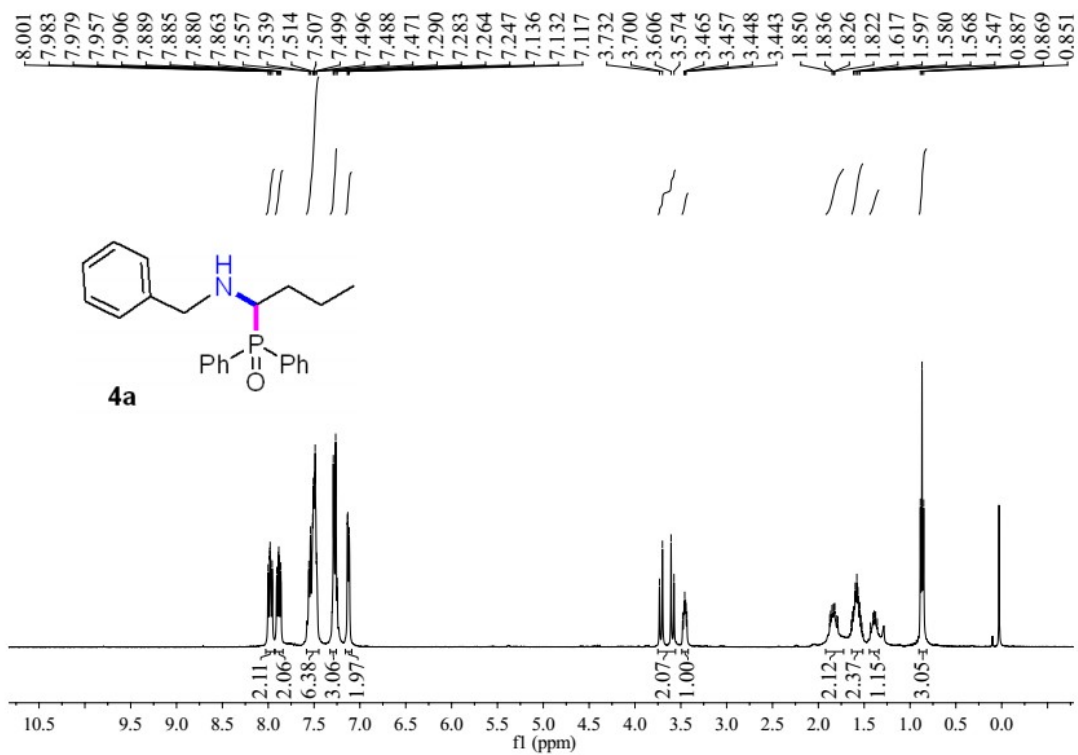
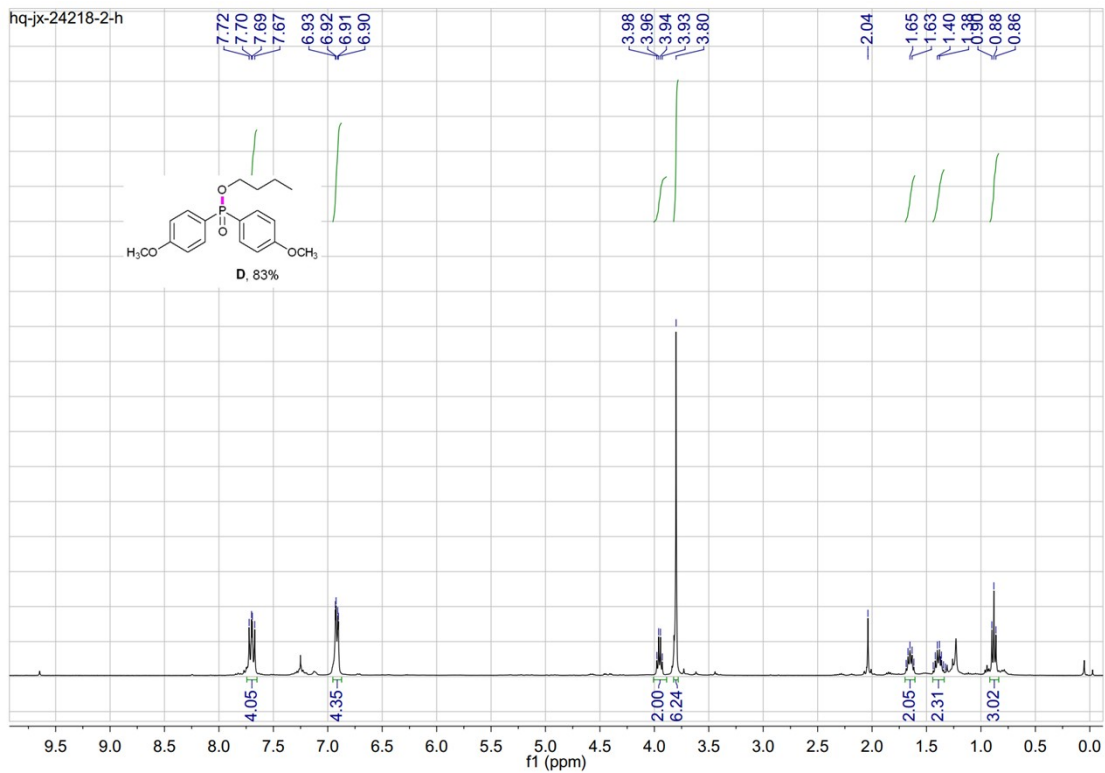


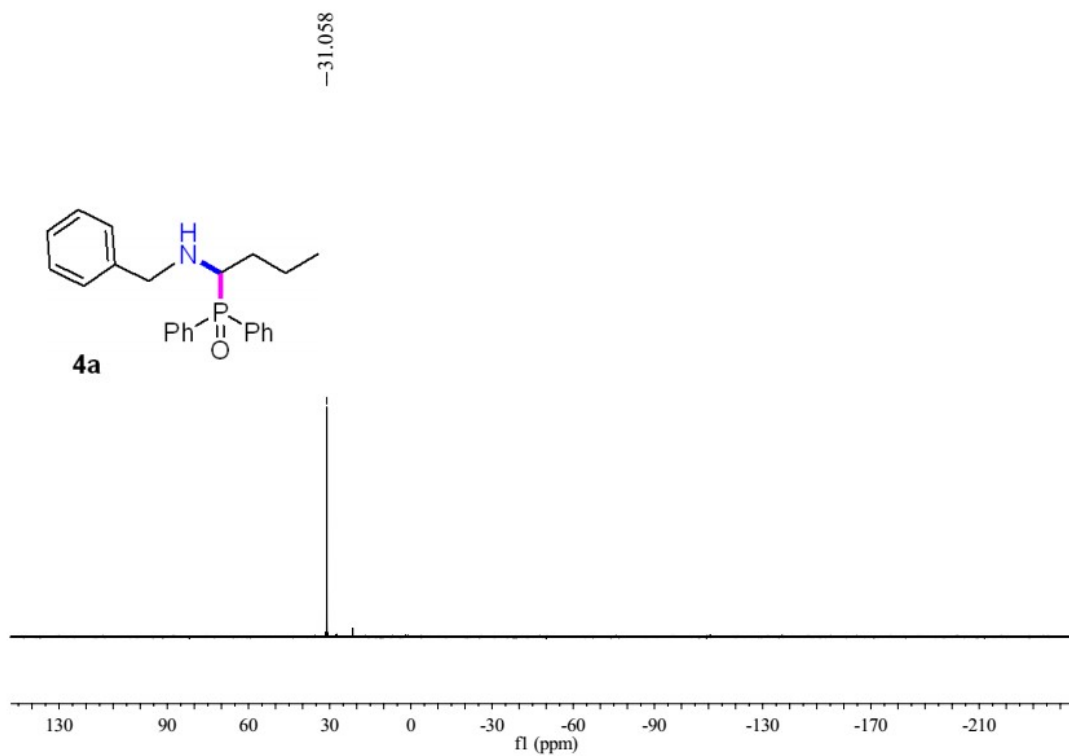
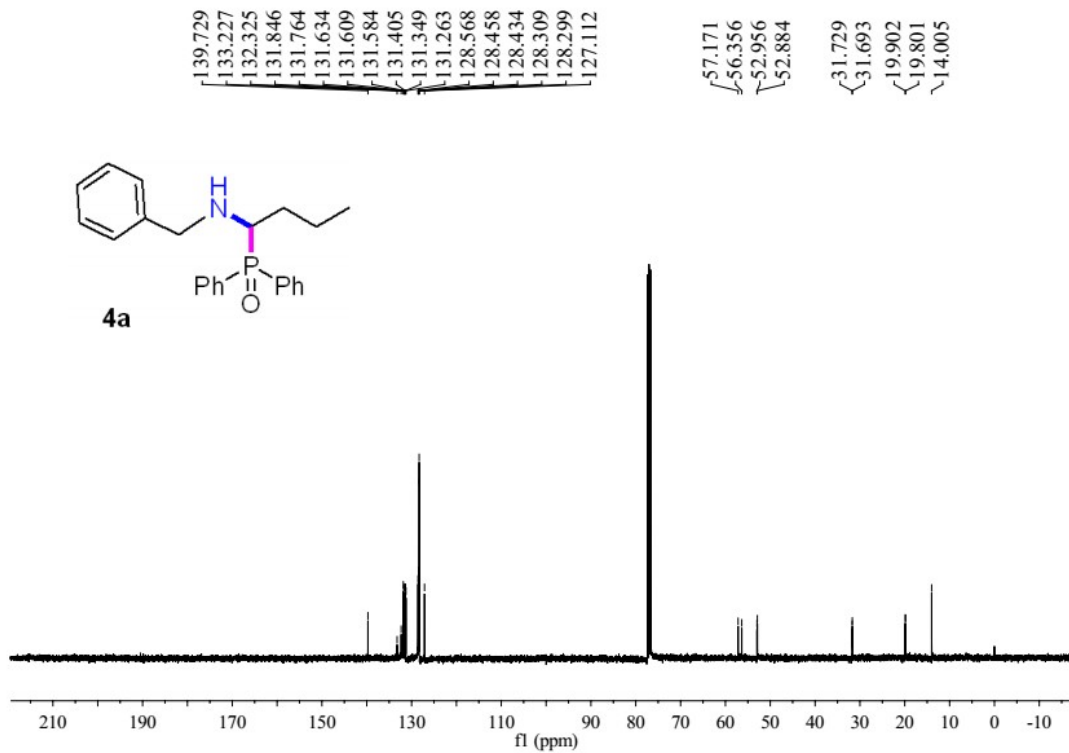


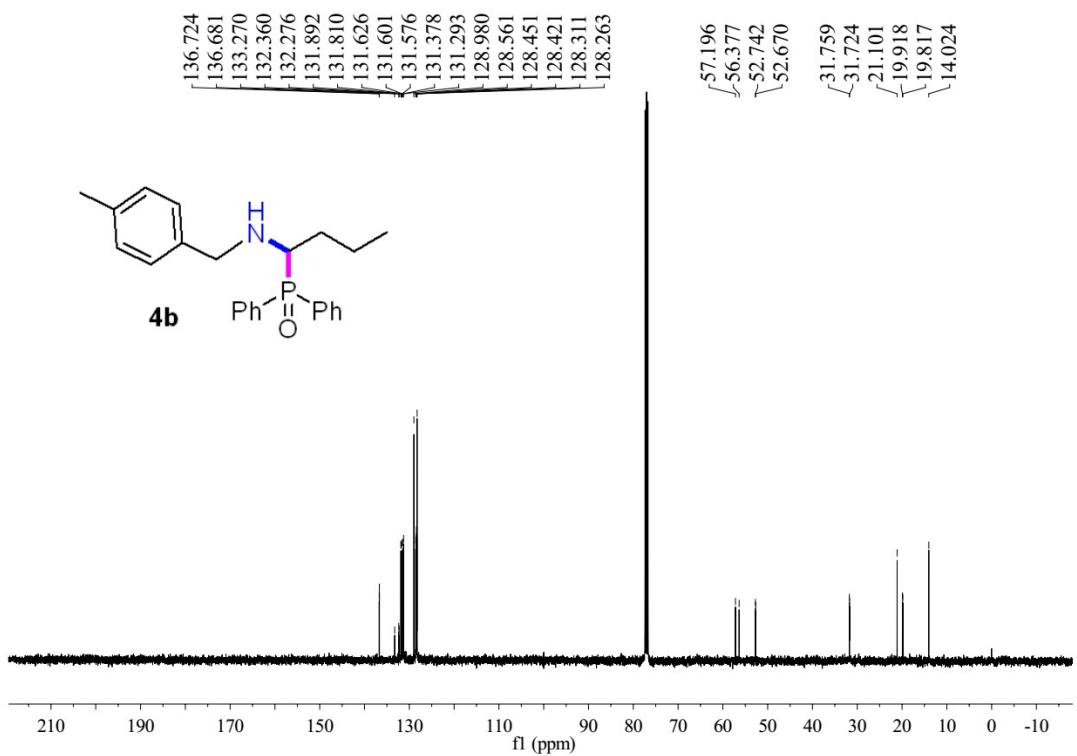
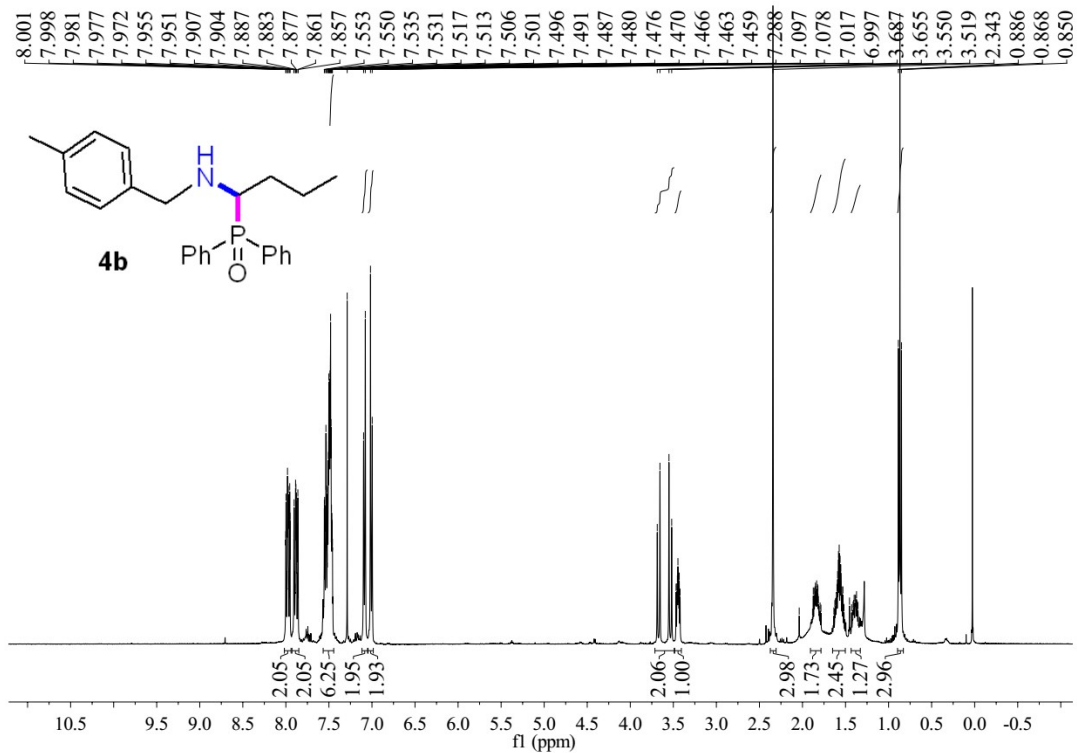
Mar18-2024 HQ-LY-P

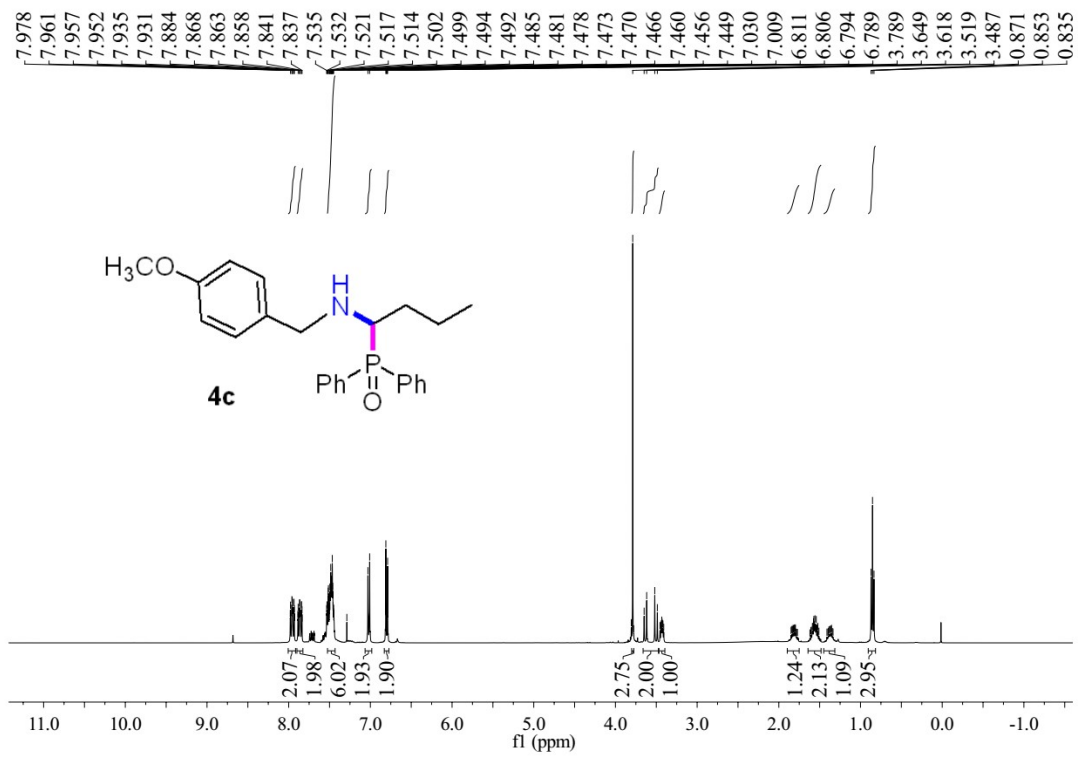
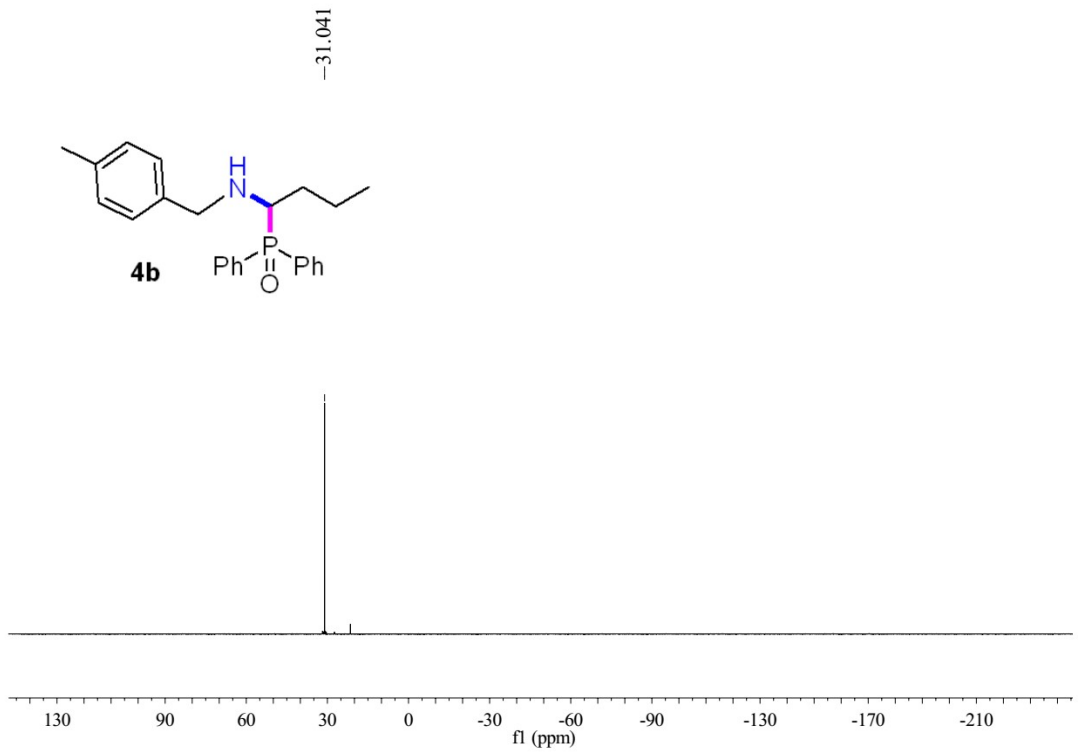
—31.86







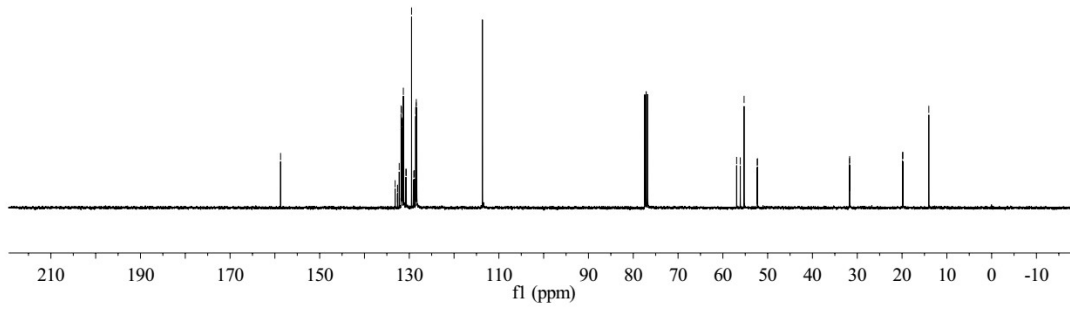
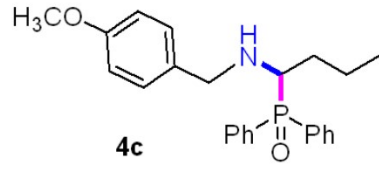




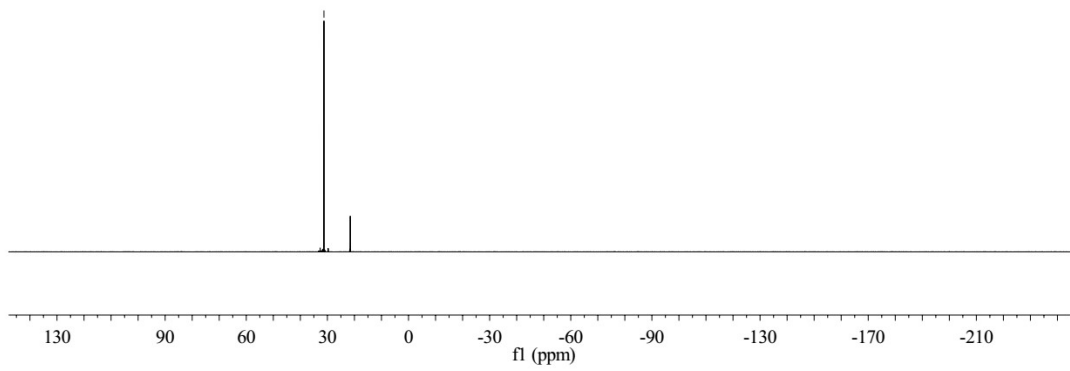
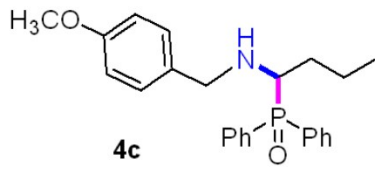
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1.09
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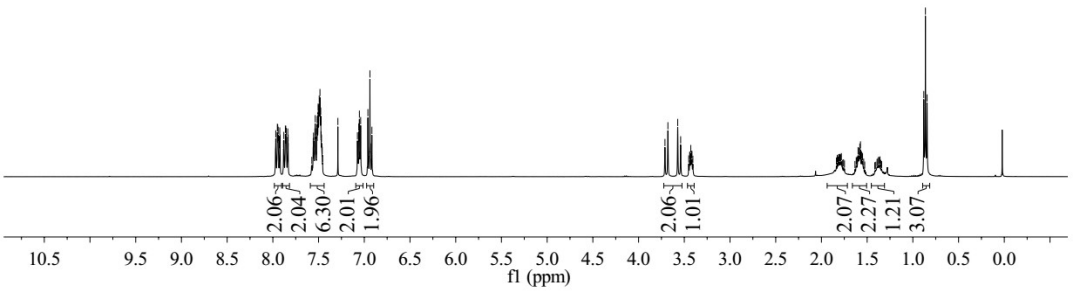
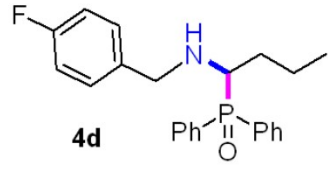
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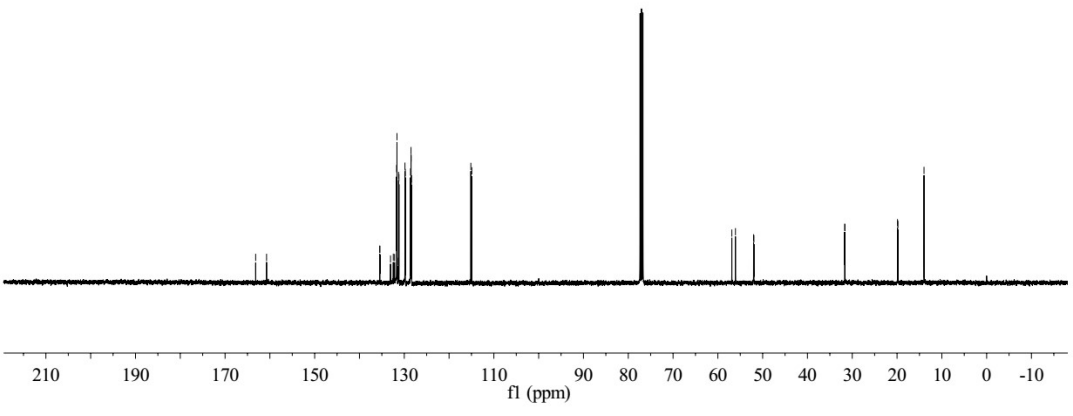
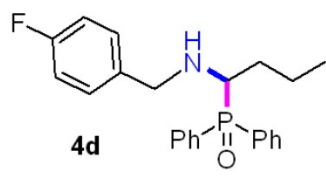
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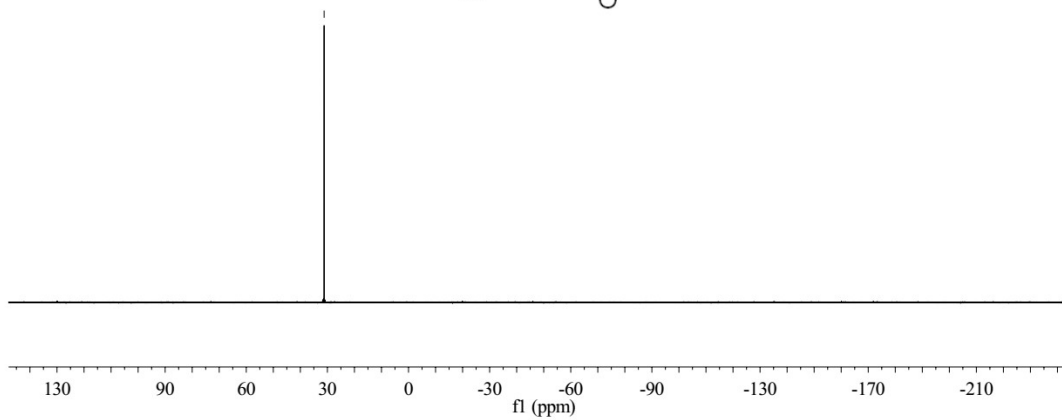
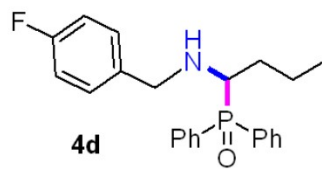
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7.504
7.502
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7.472
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7.061
7.053
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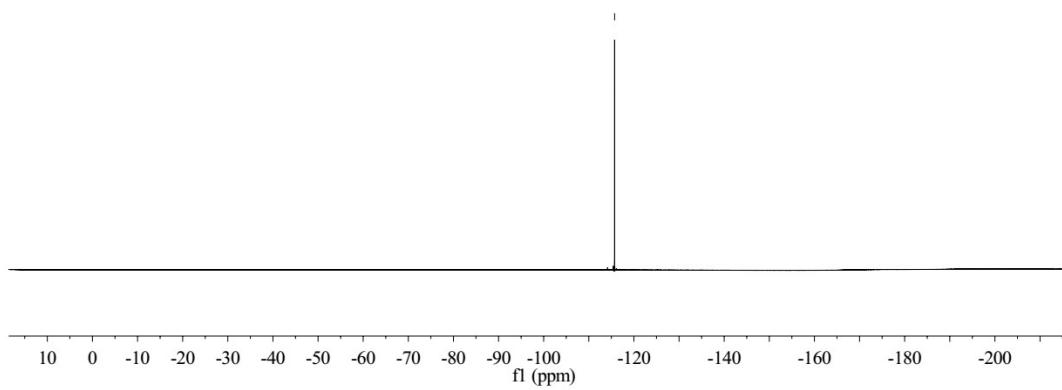
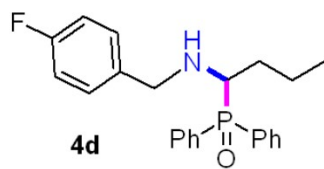
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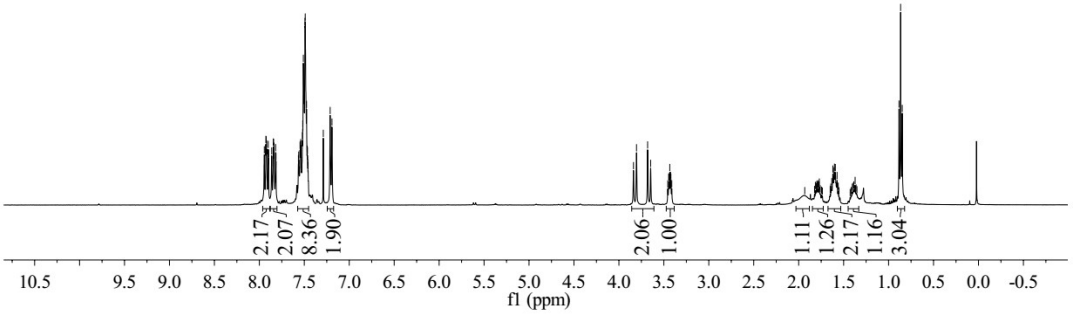
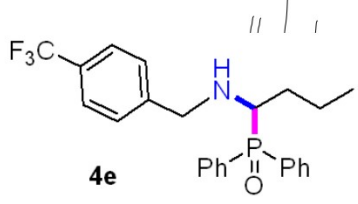
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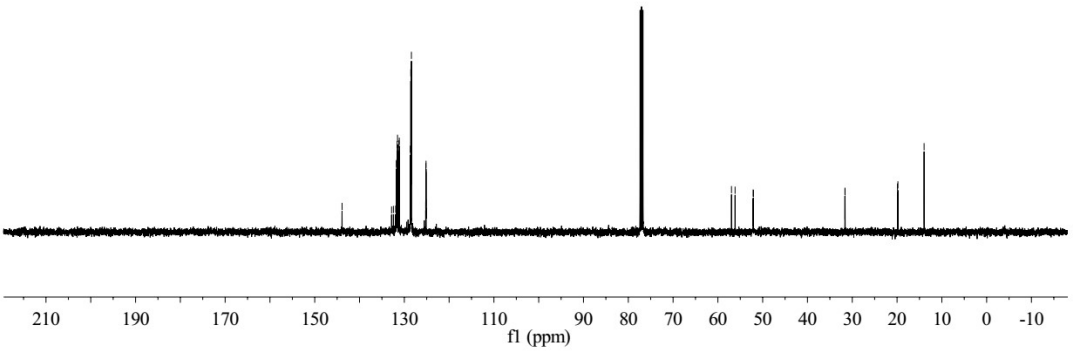
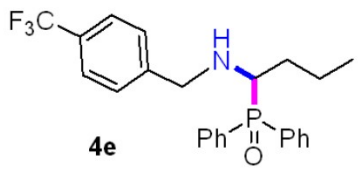
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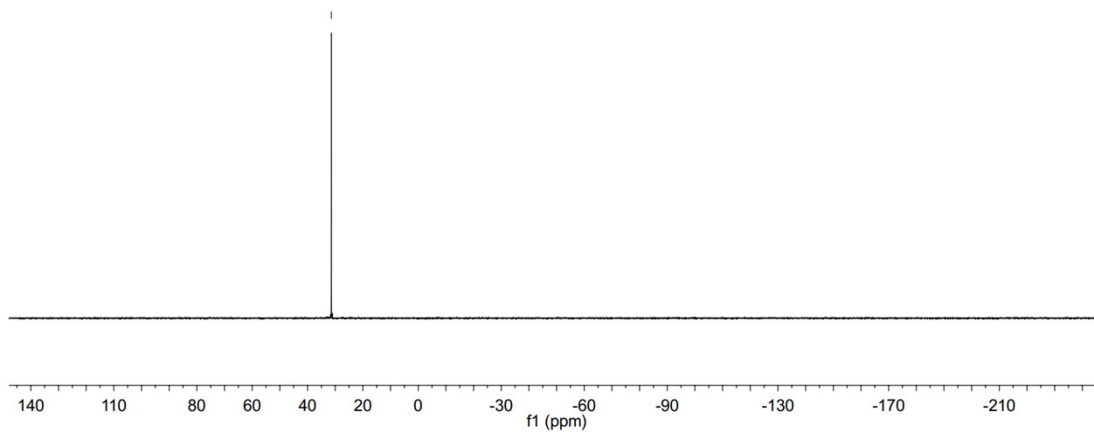
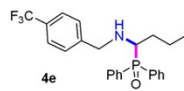
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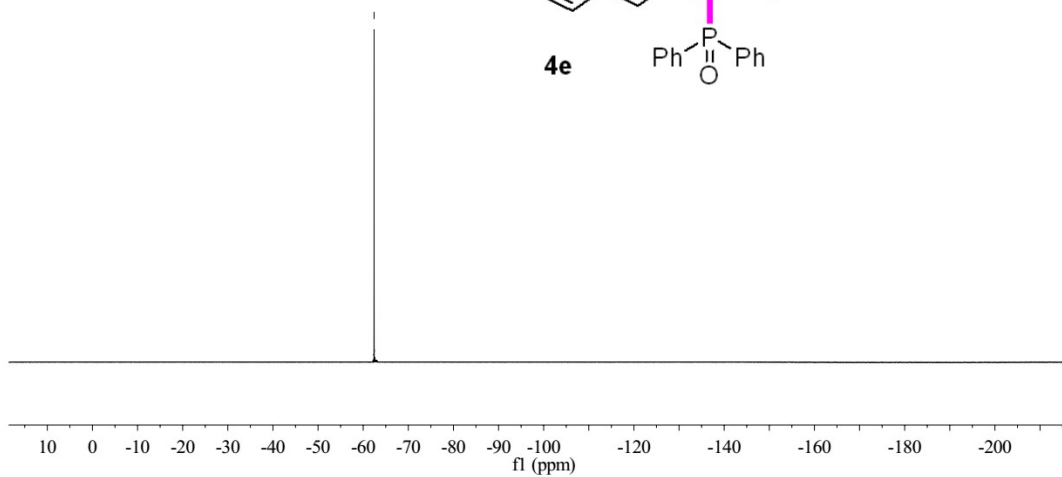
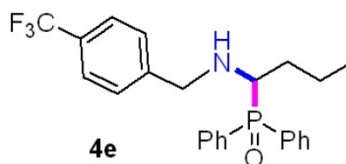
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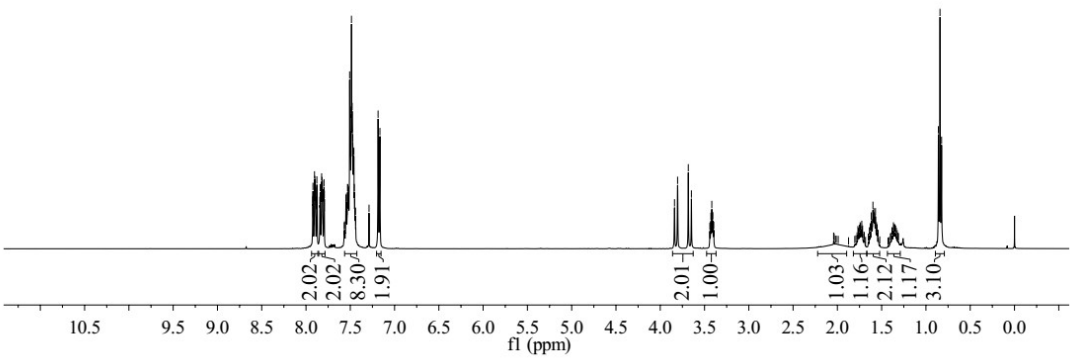
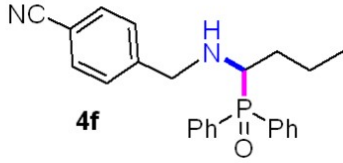
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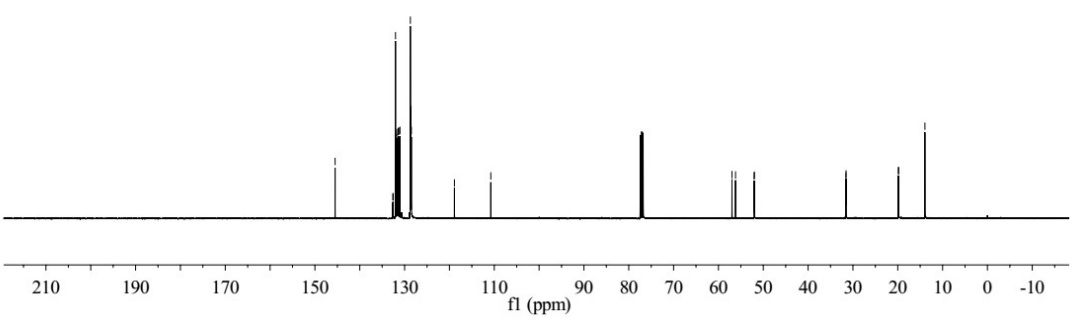
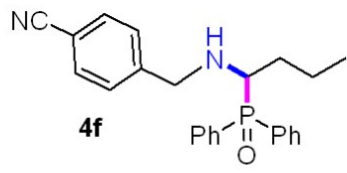
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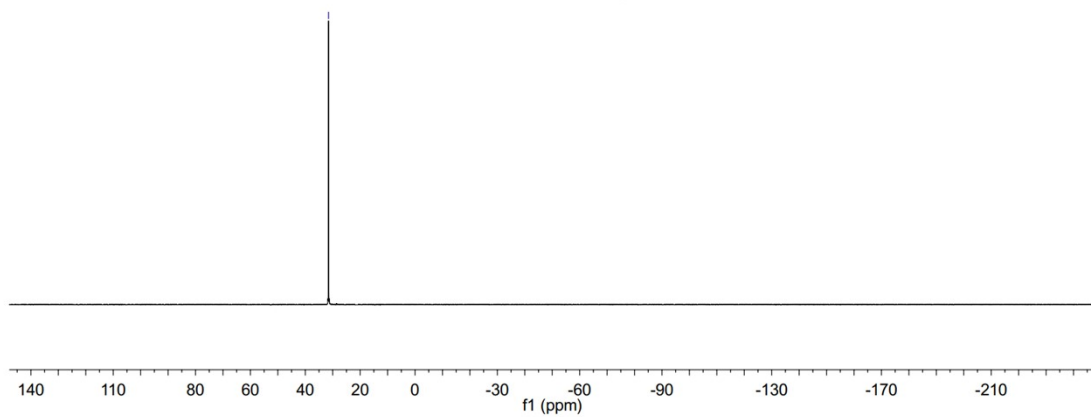
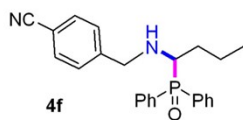
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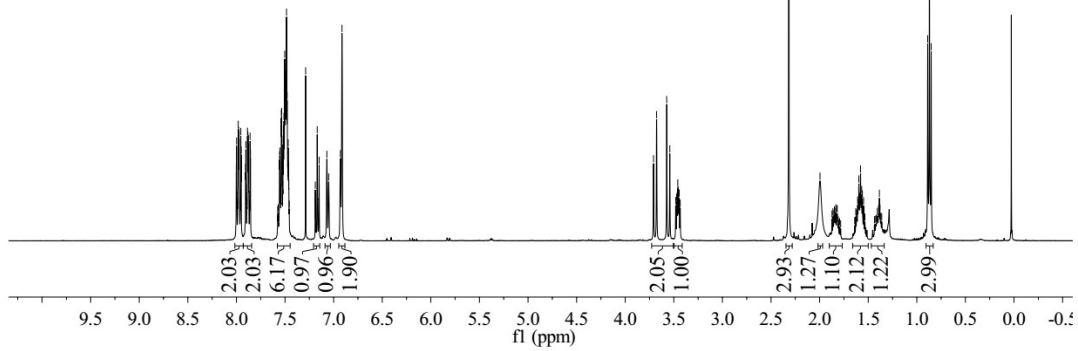
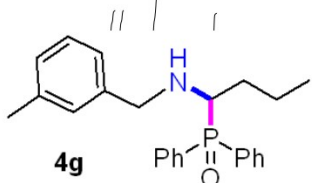
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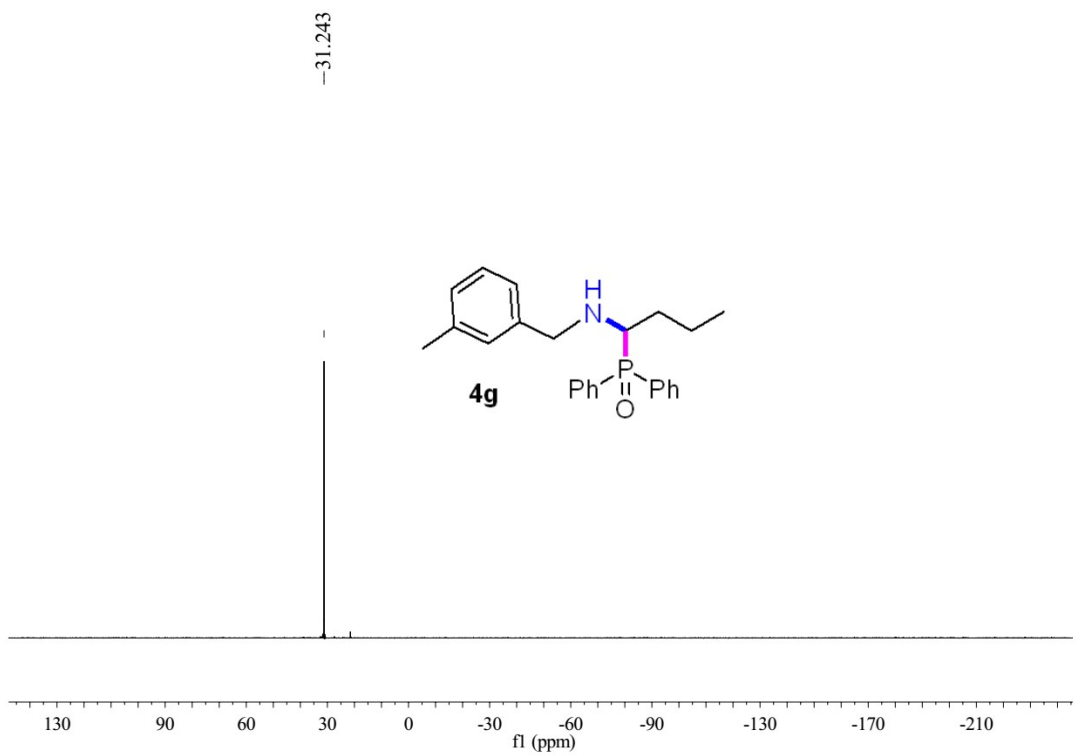
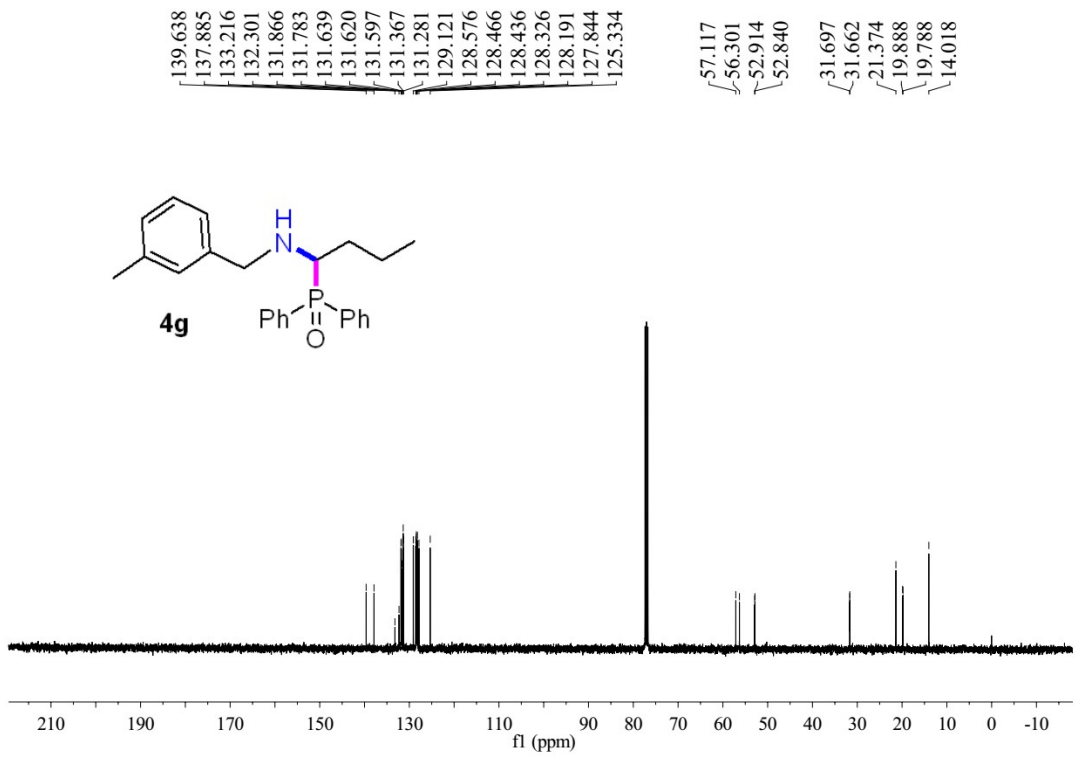


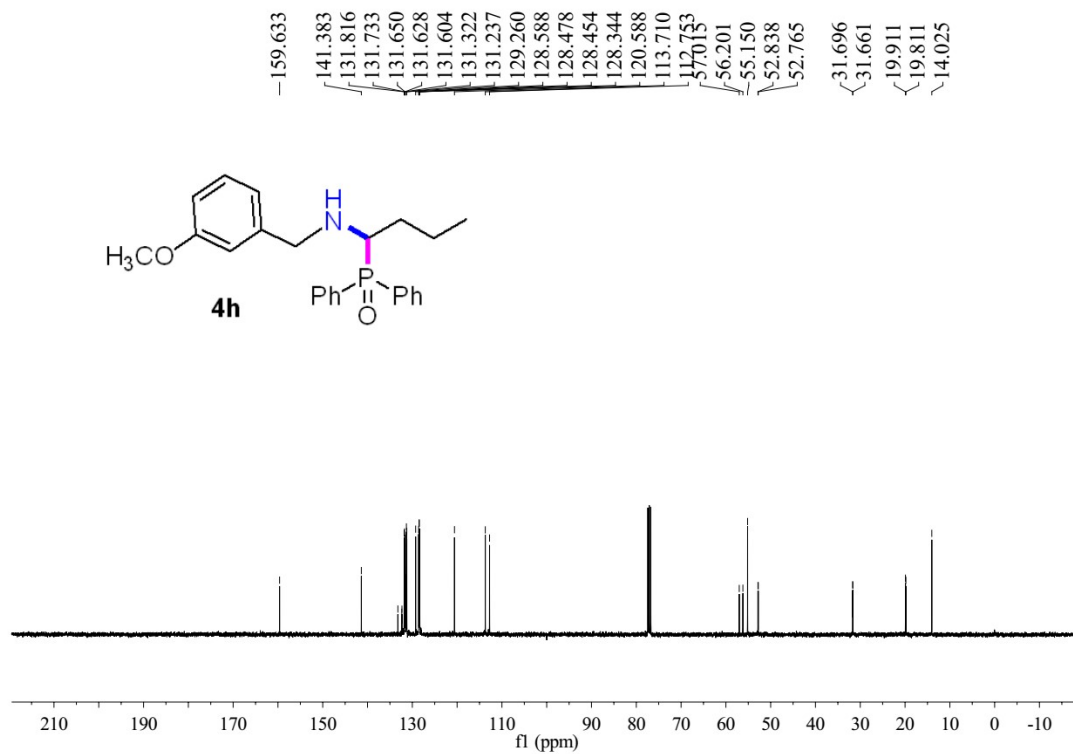
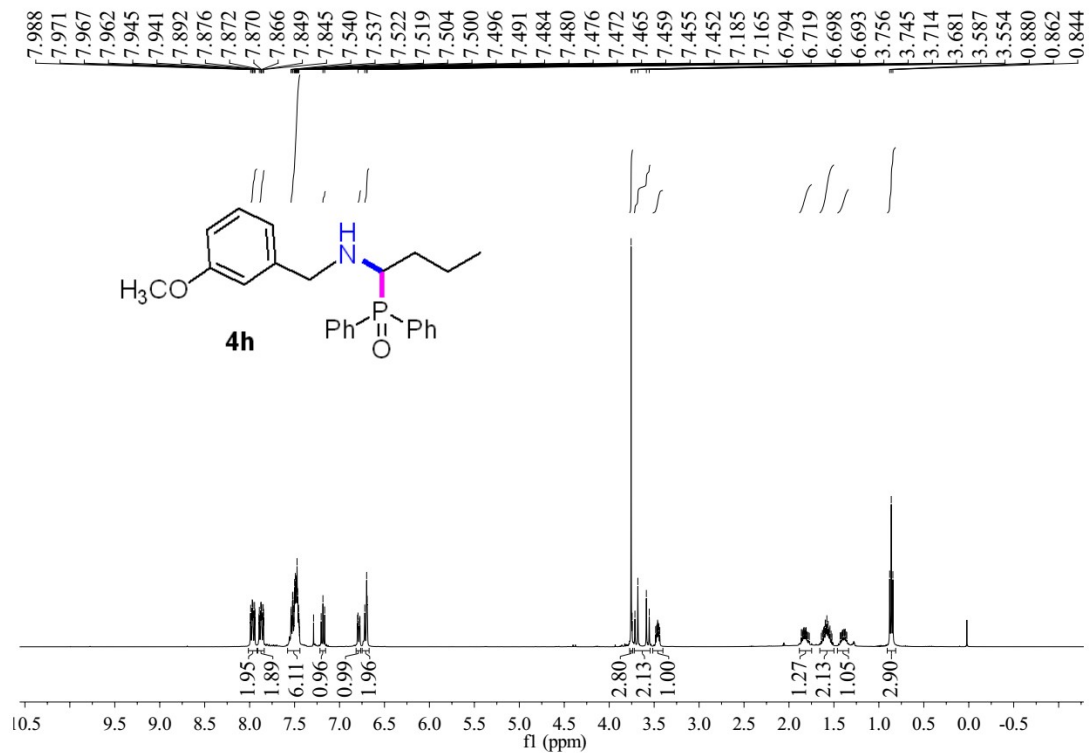
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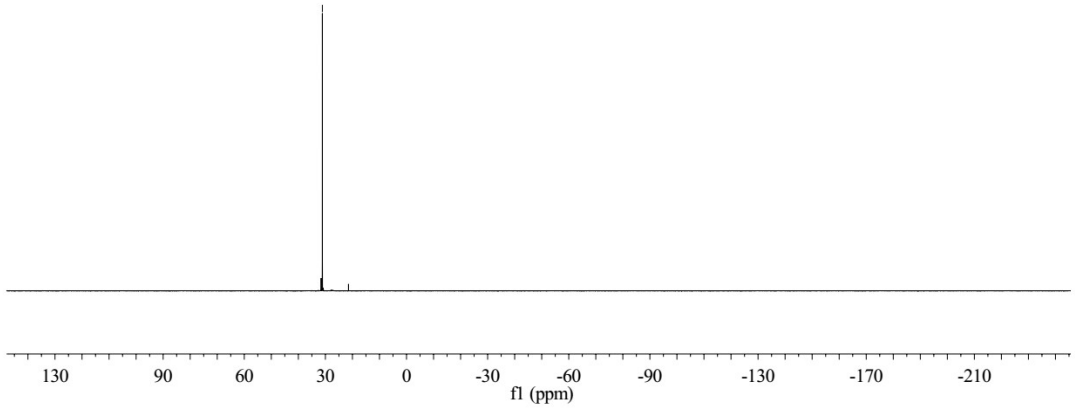
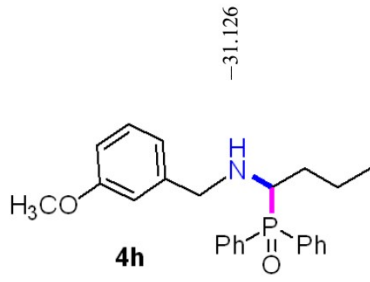


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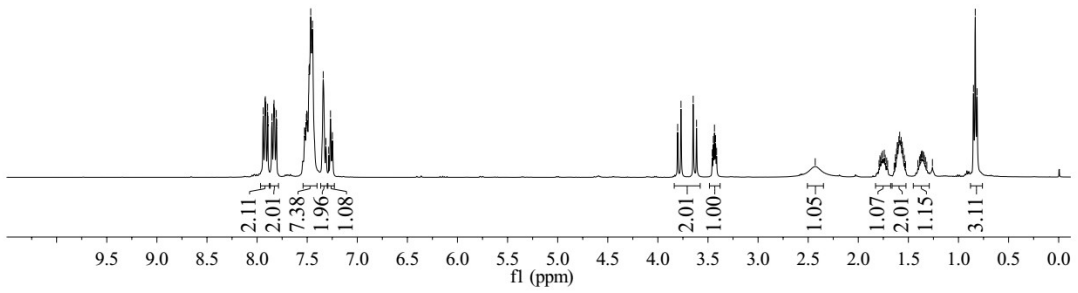
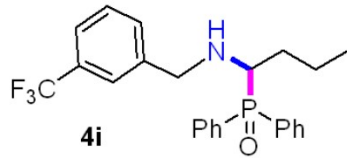








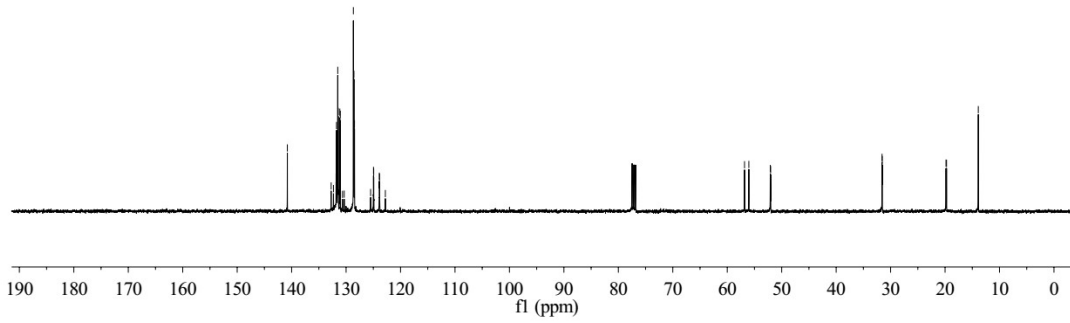
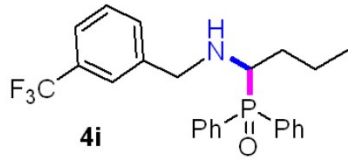
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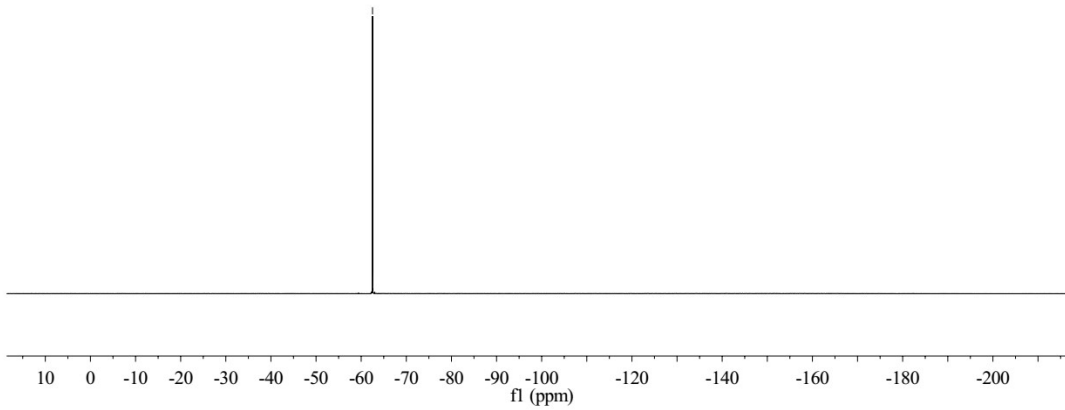
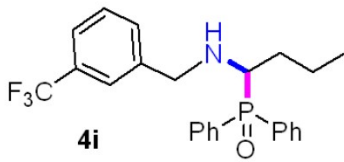
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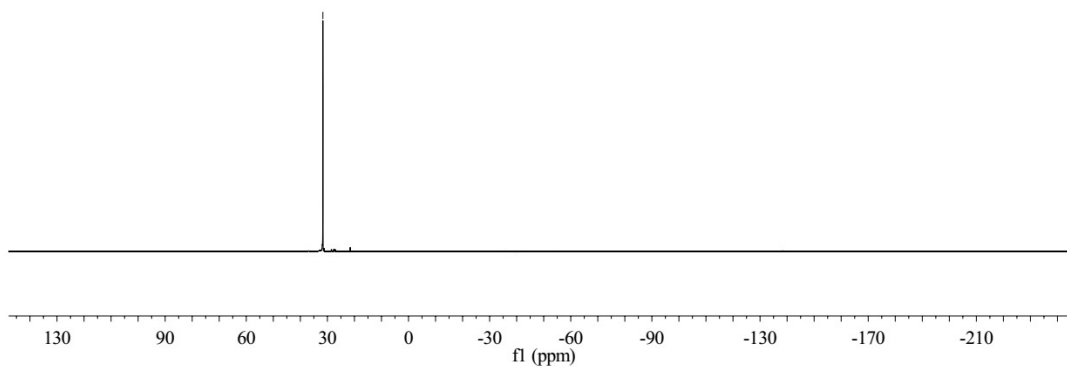
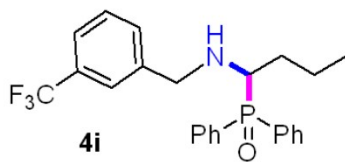
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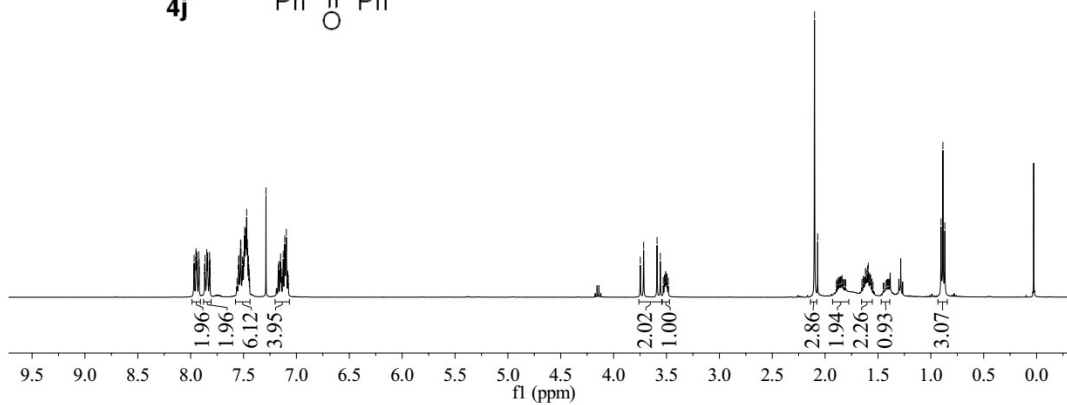
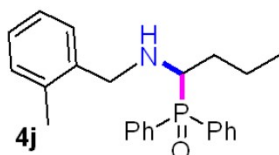
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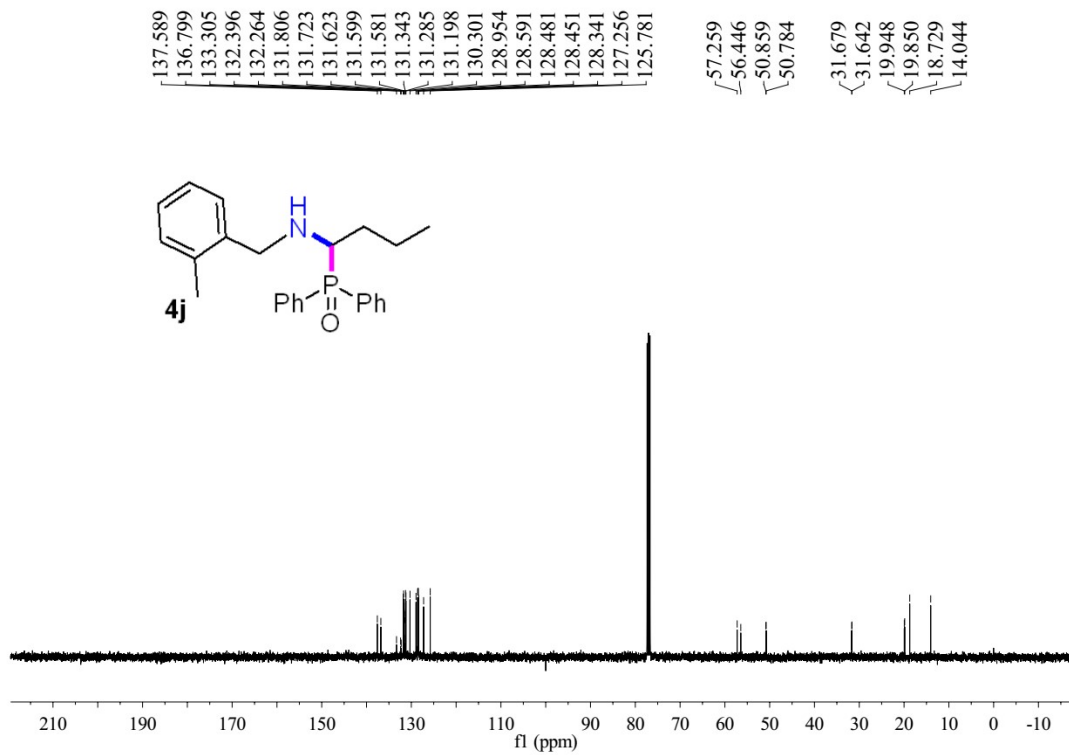


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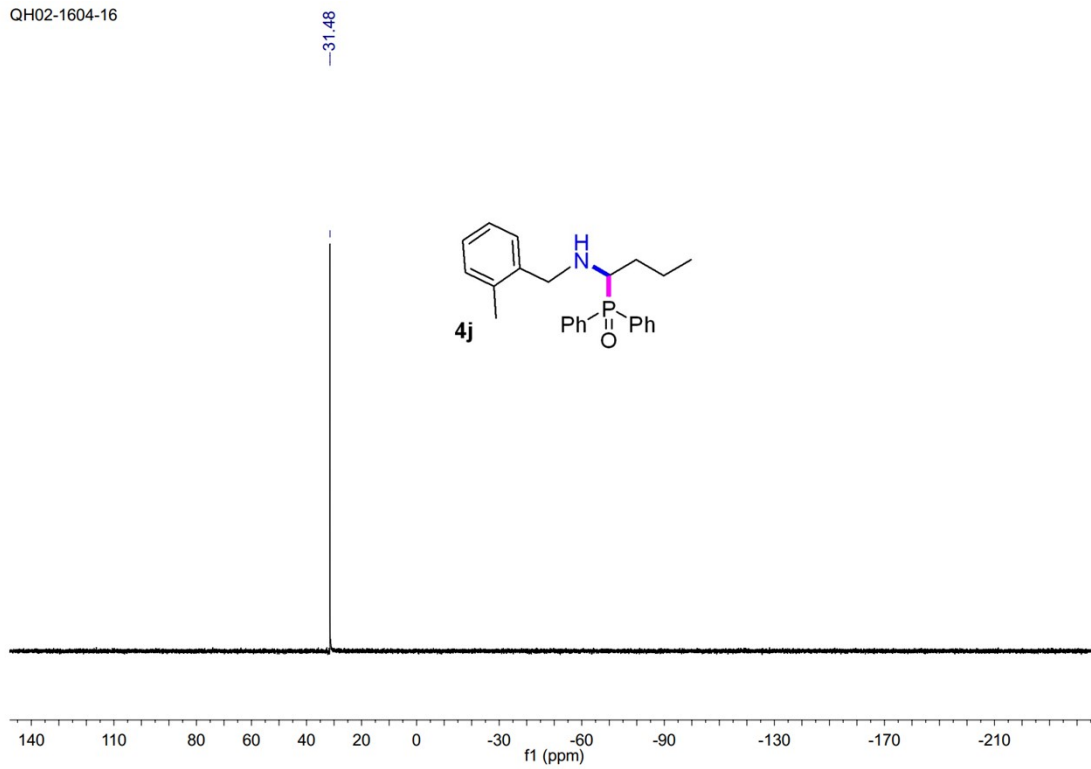


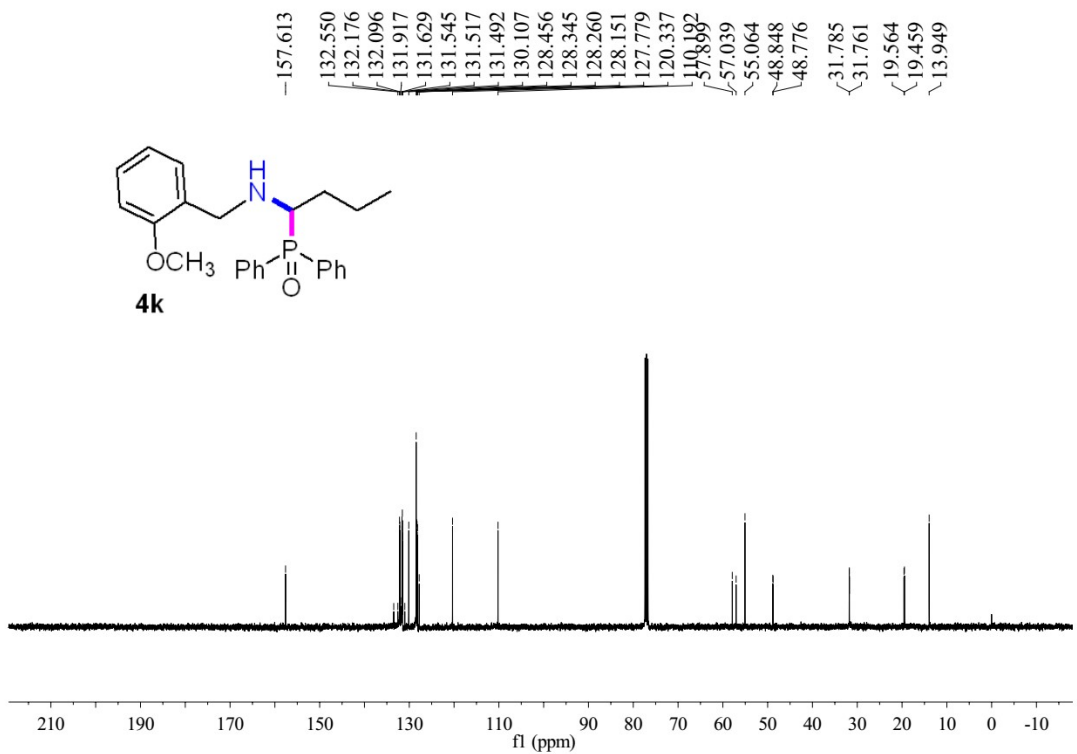
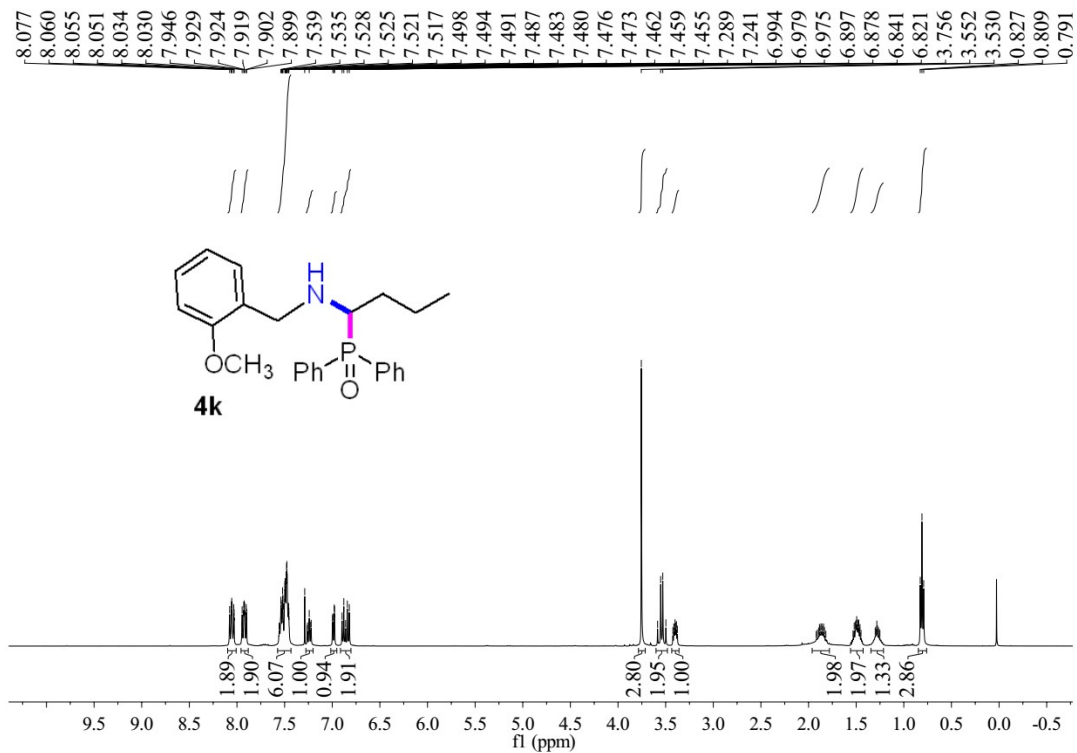
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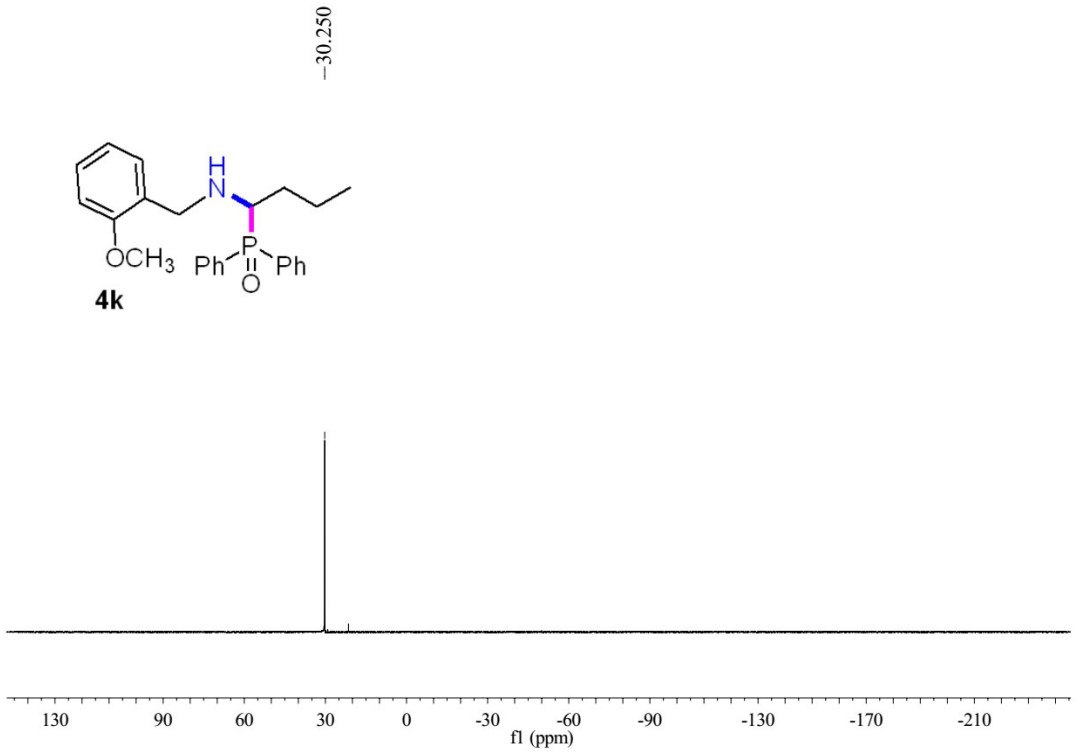
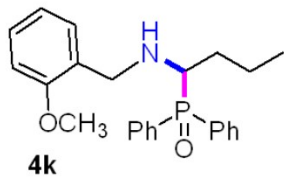




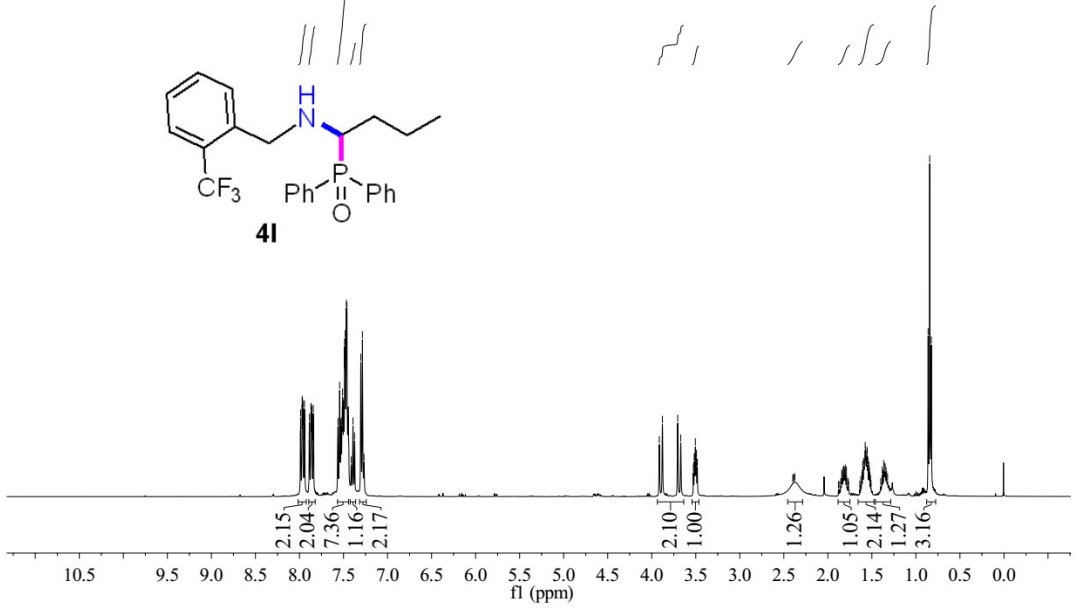
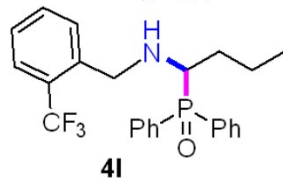
QH02-1604-16



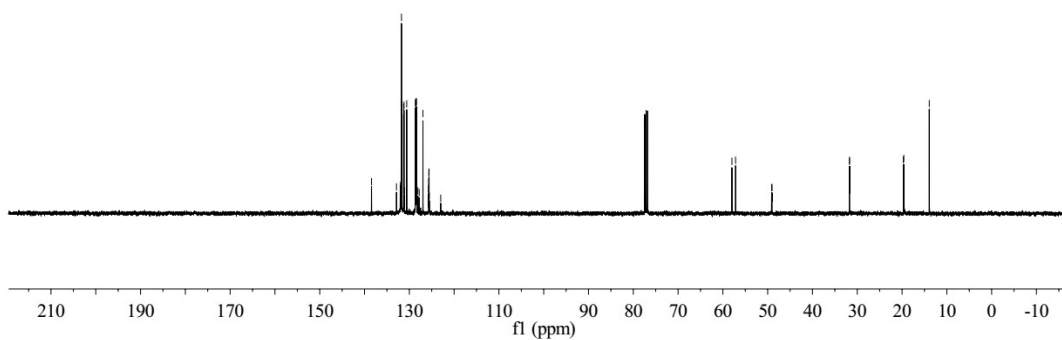
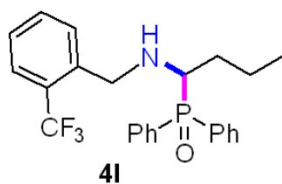




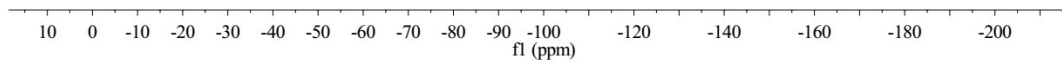
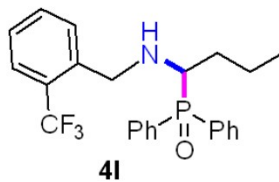
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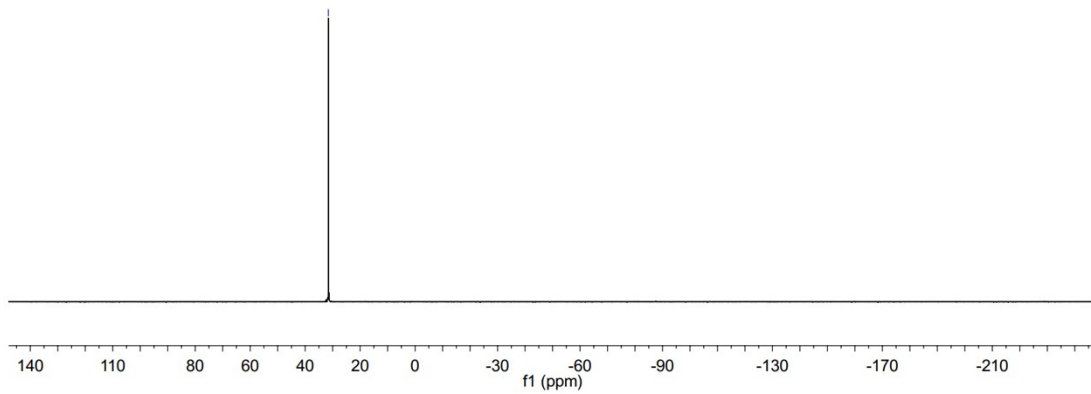
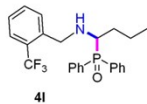


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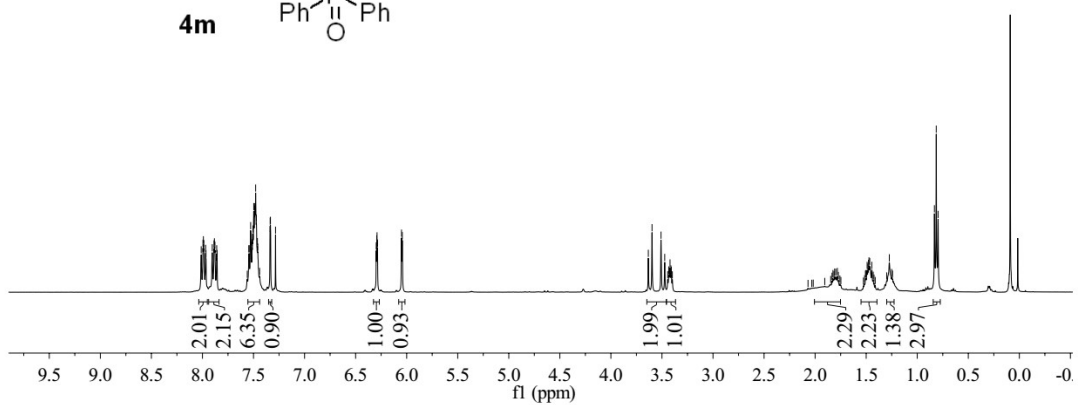
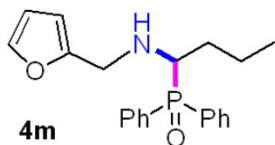


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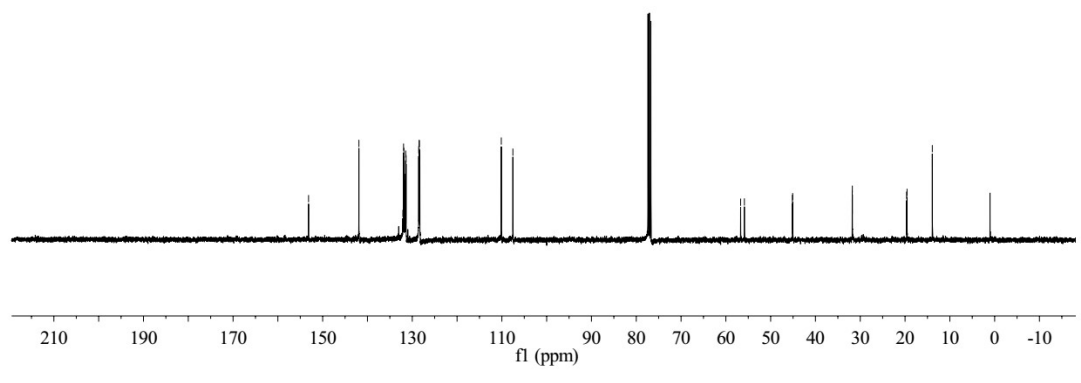
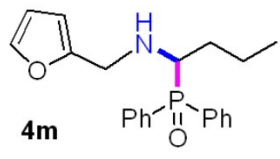


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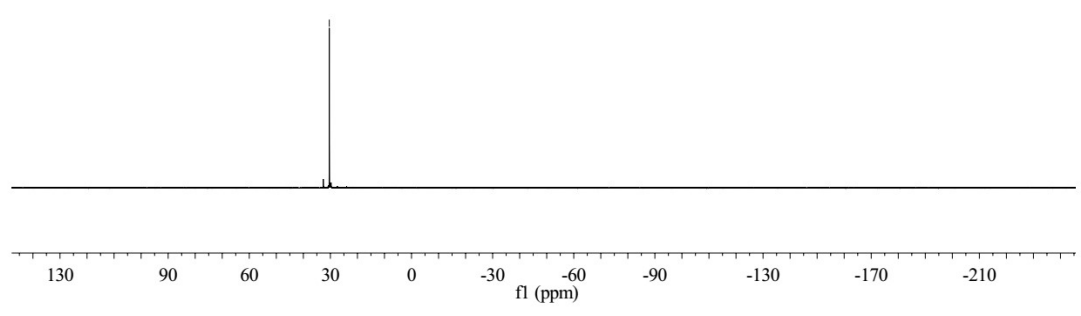
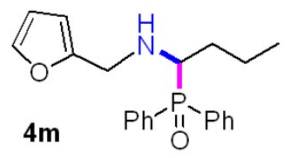


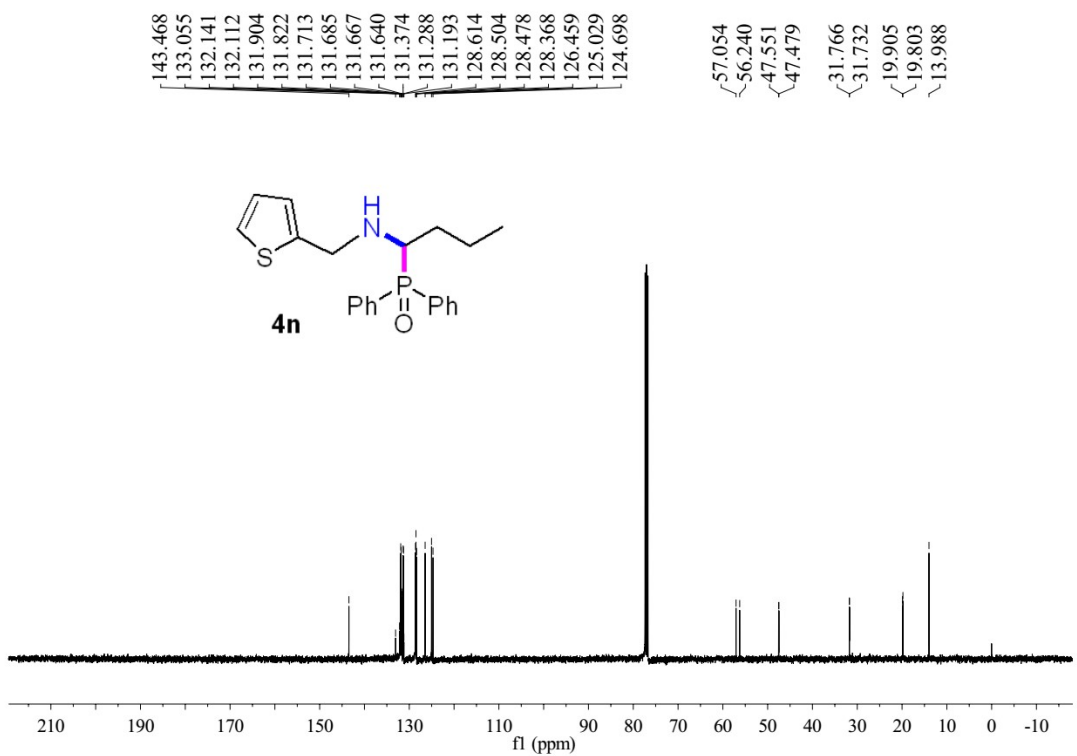
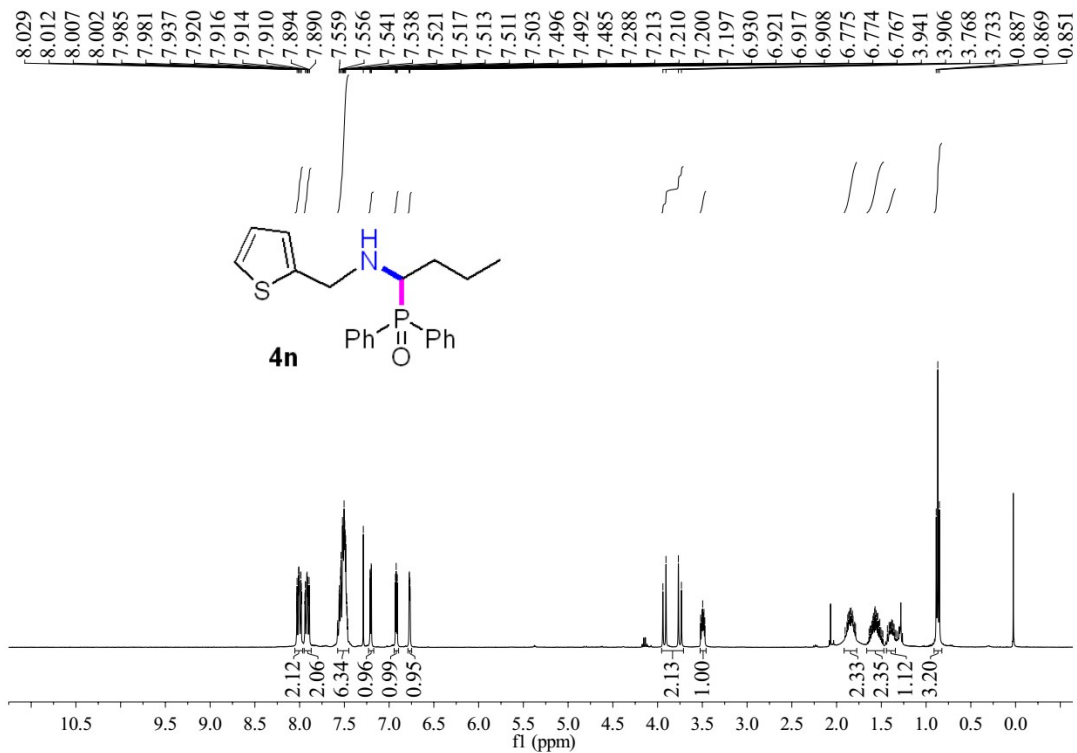
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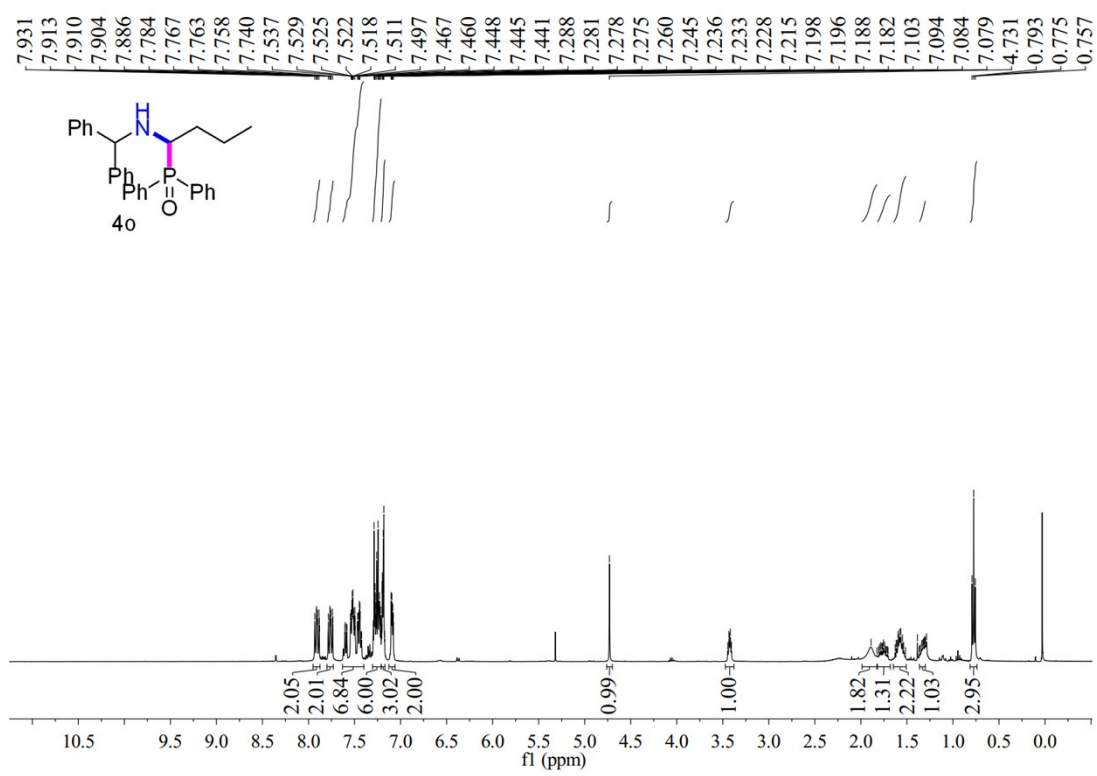
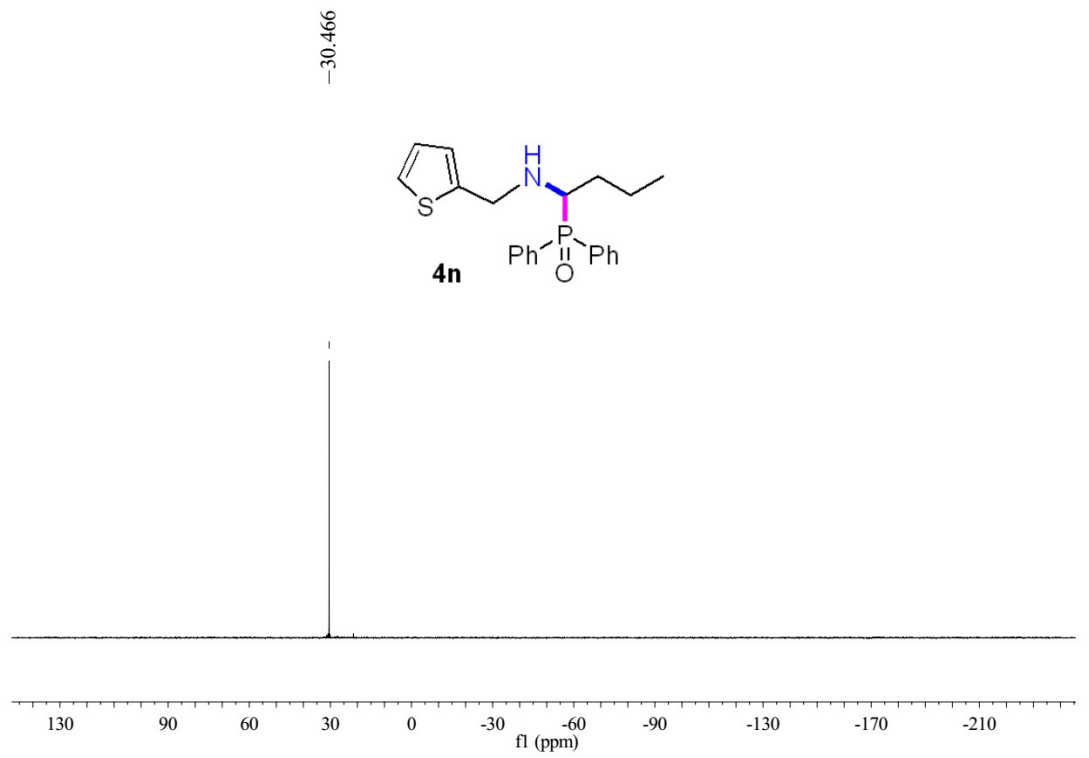
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19.553
13.921

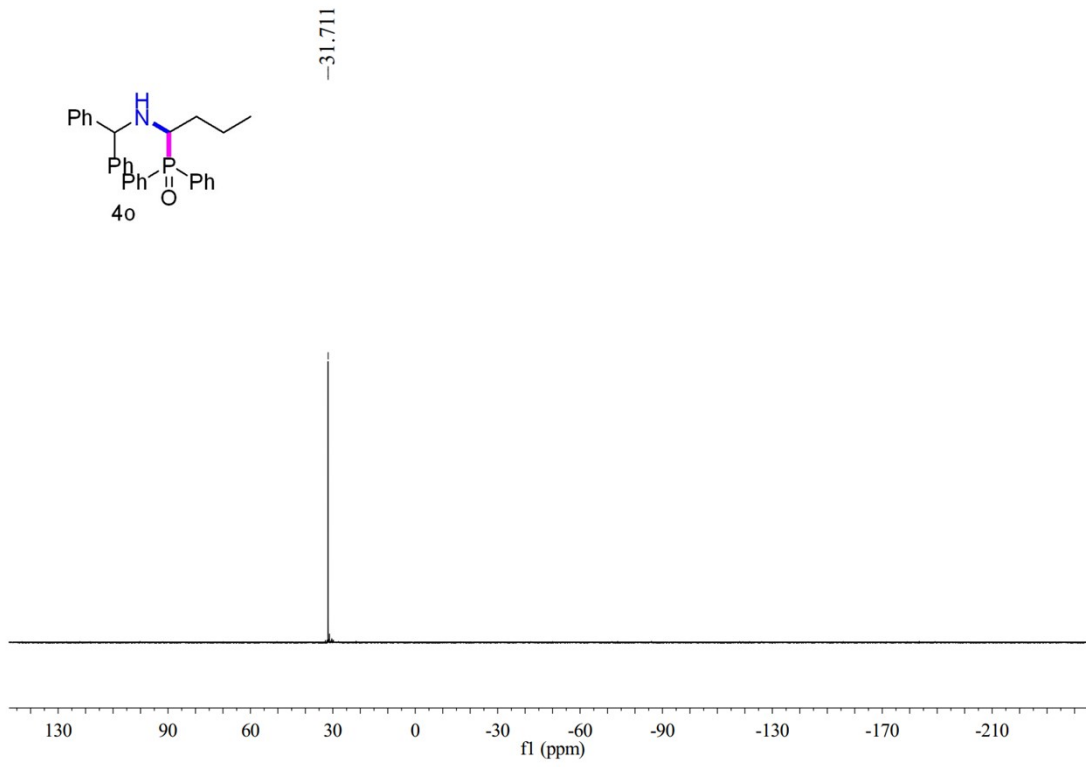
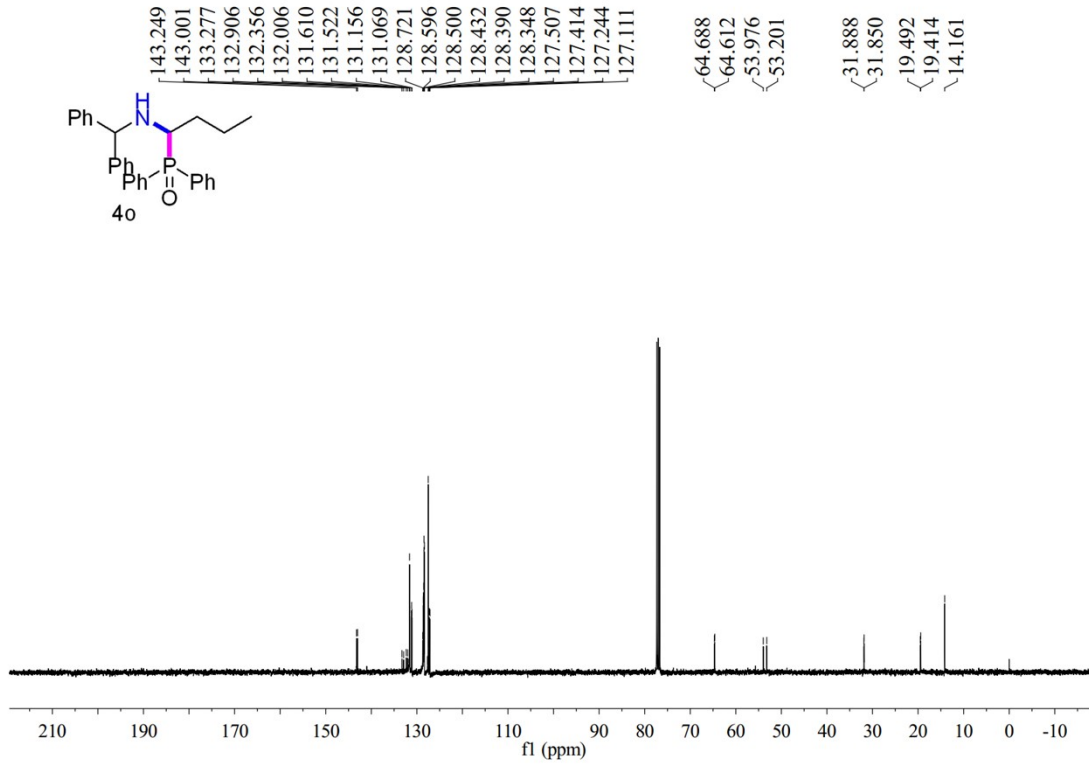


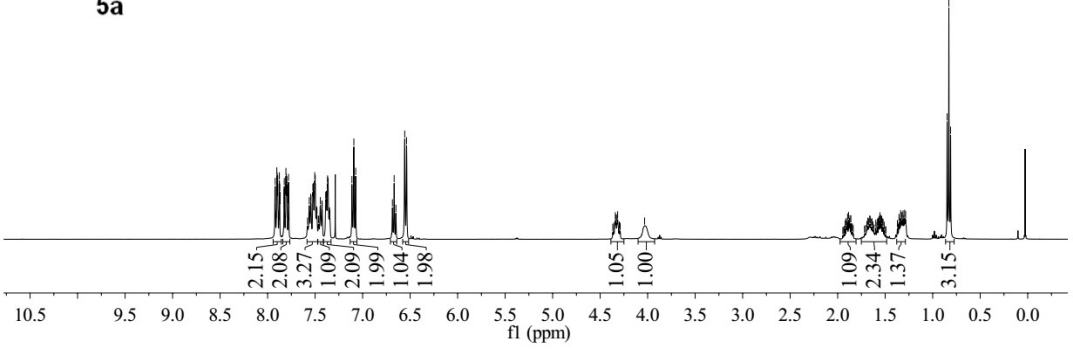
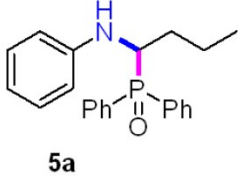
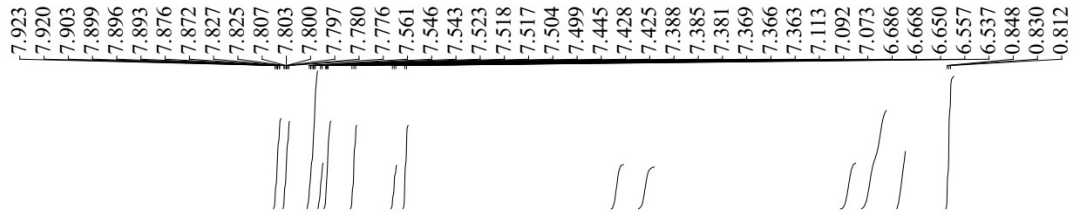
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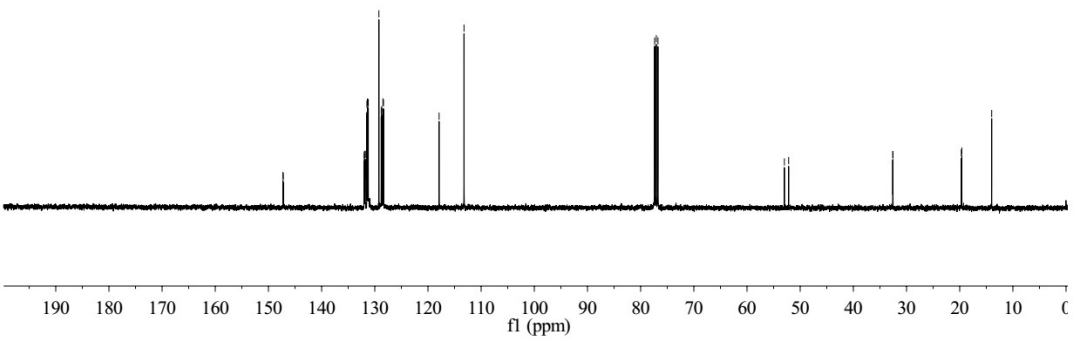
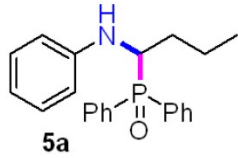


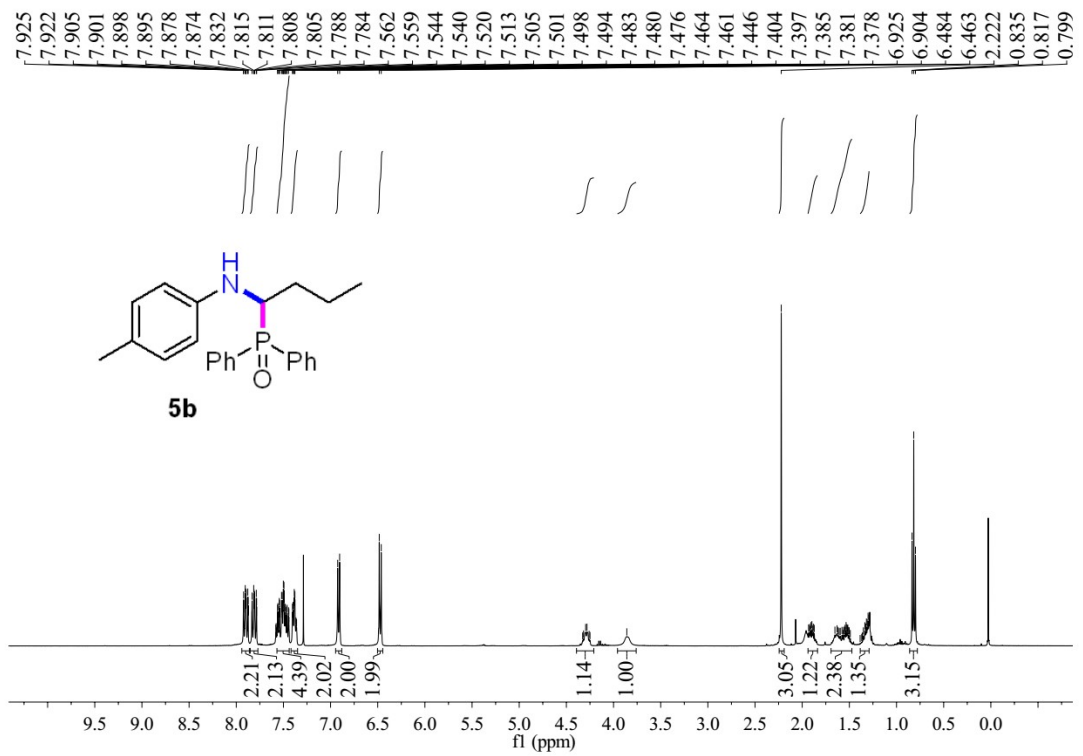
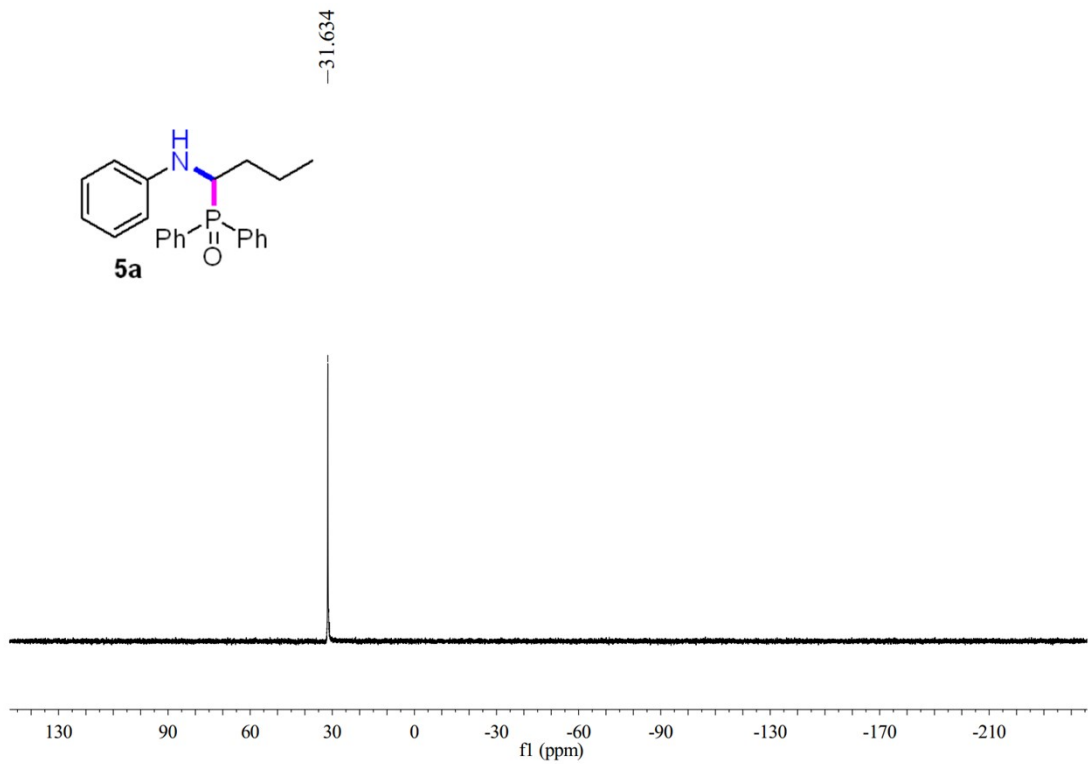


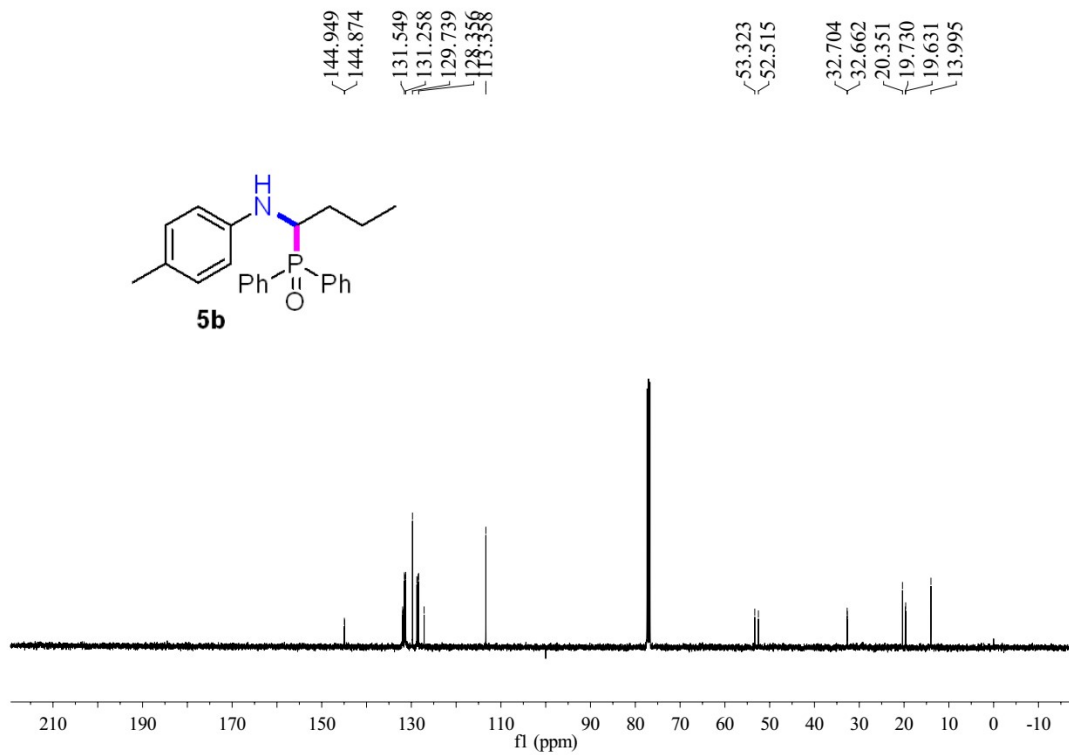




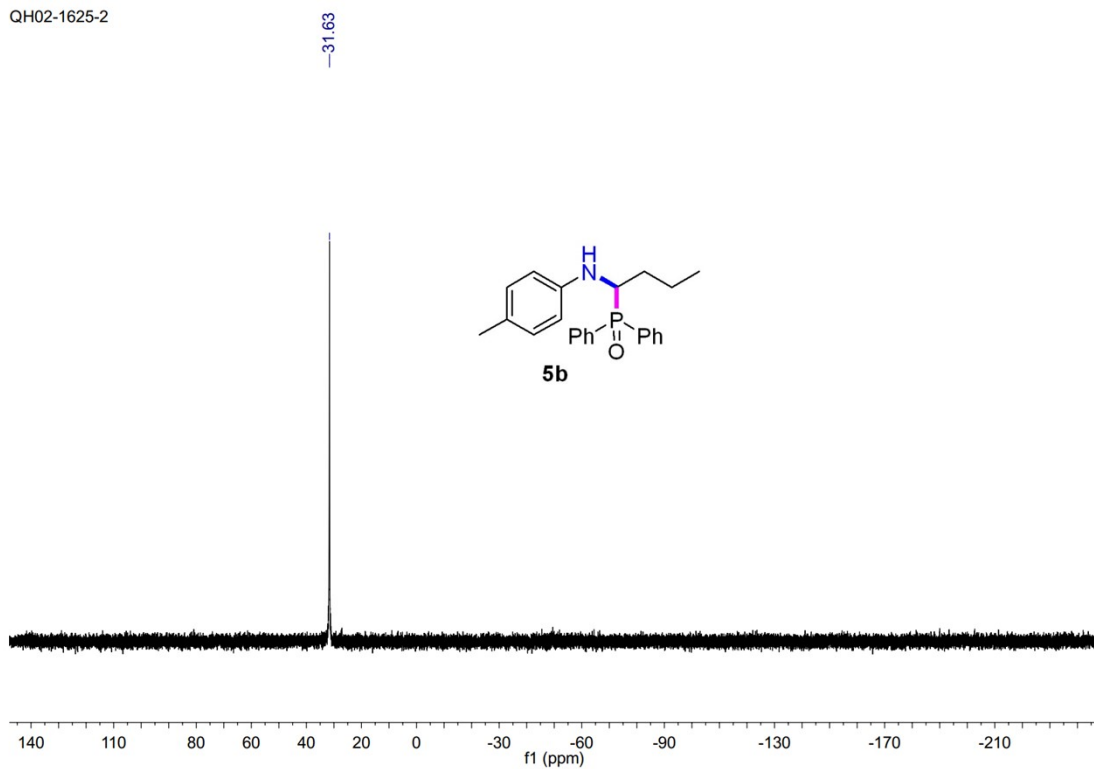
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- 147.208
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- 128.474
- 113.221
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- 52.969
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- 32.588
- 32.547
- 19.699
- 19.598
- 13.981

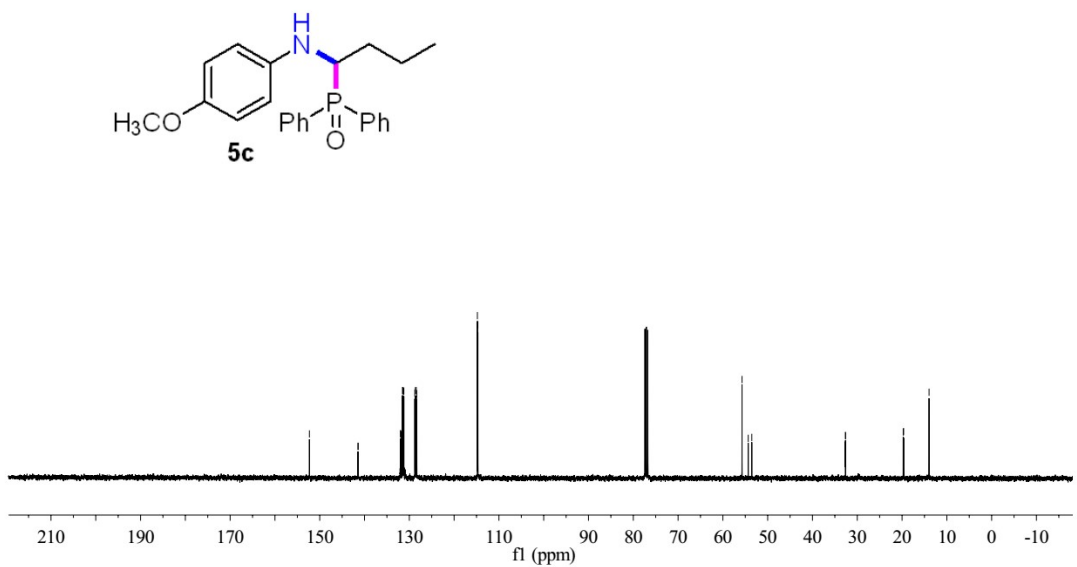
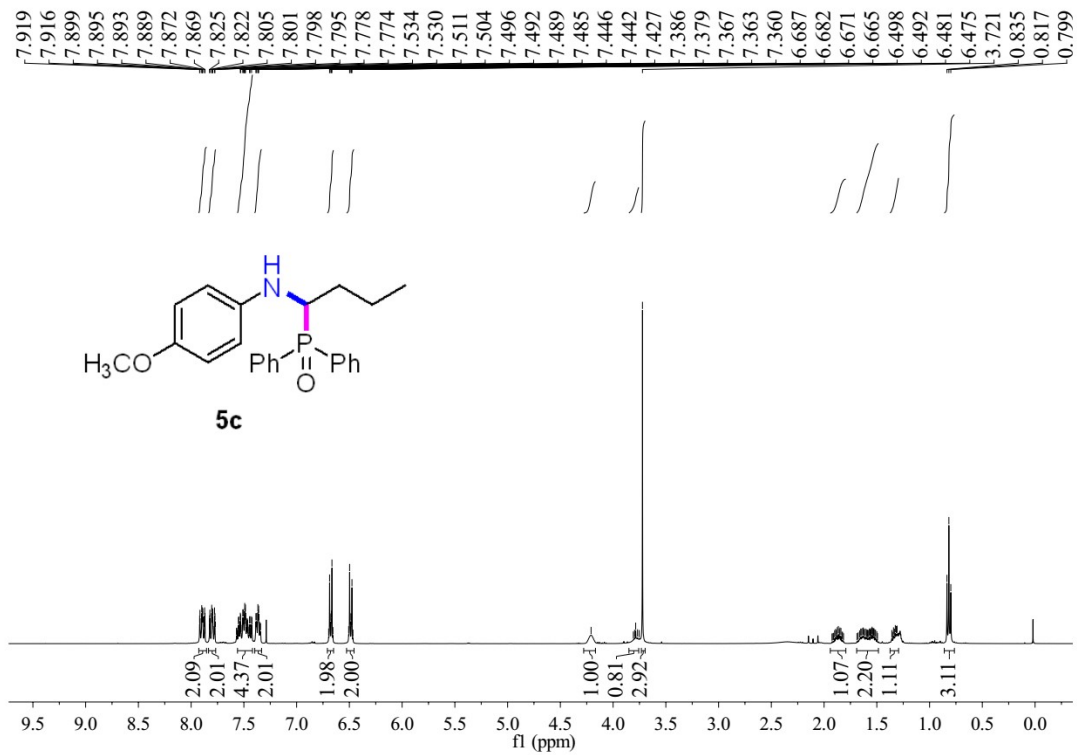


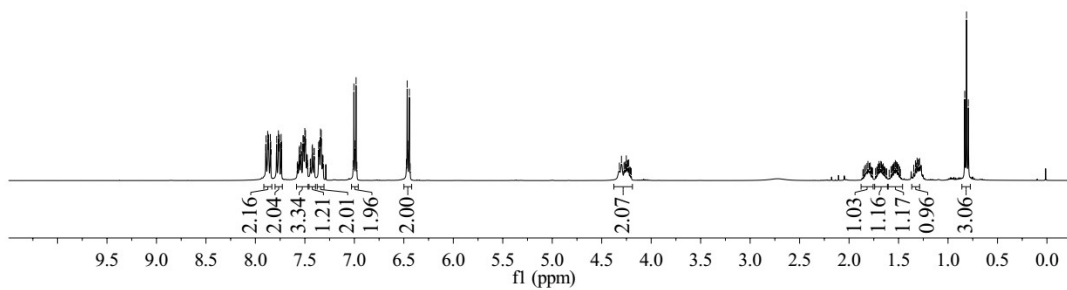
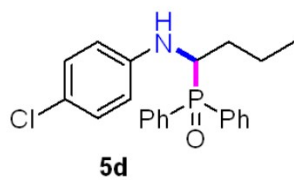
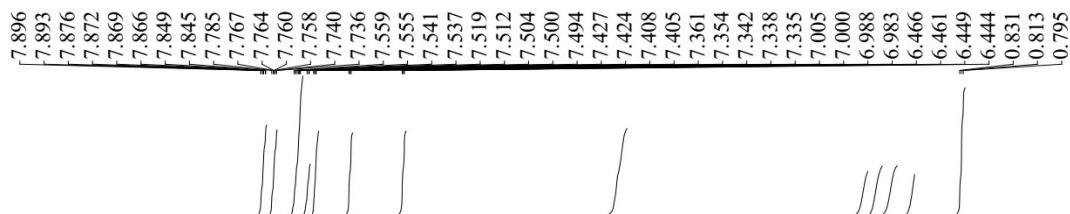
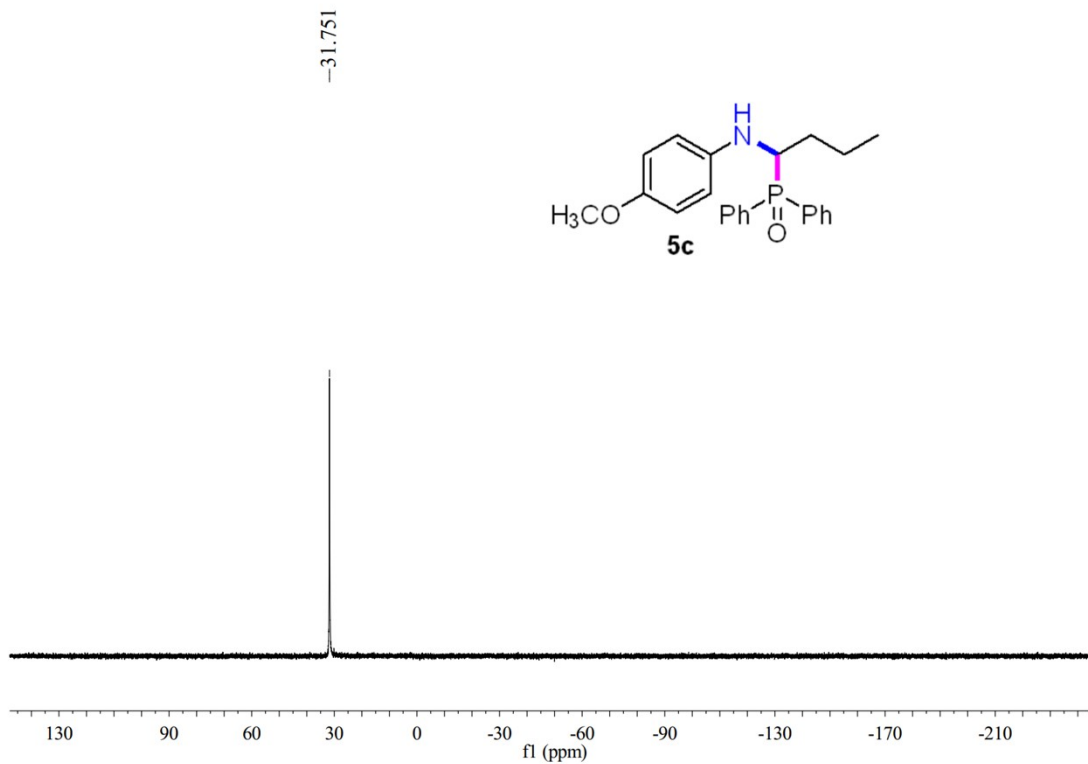
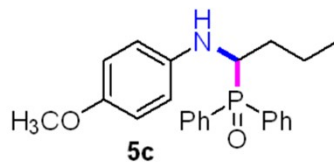


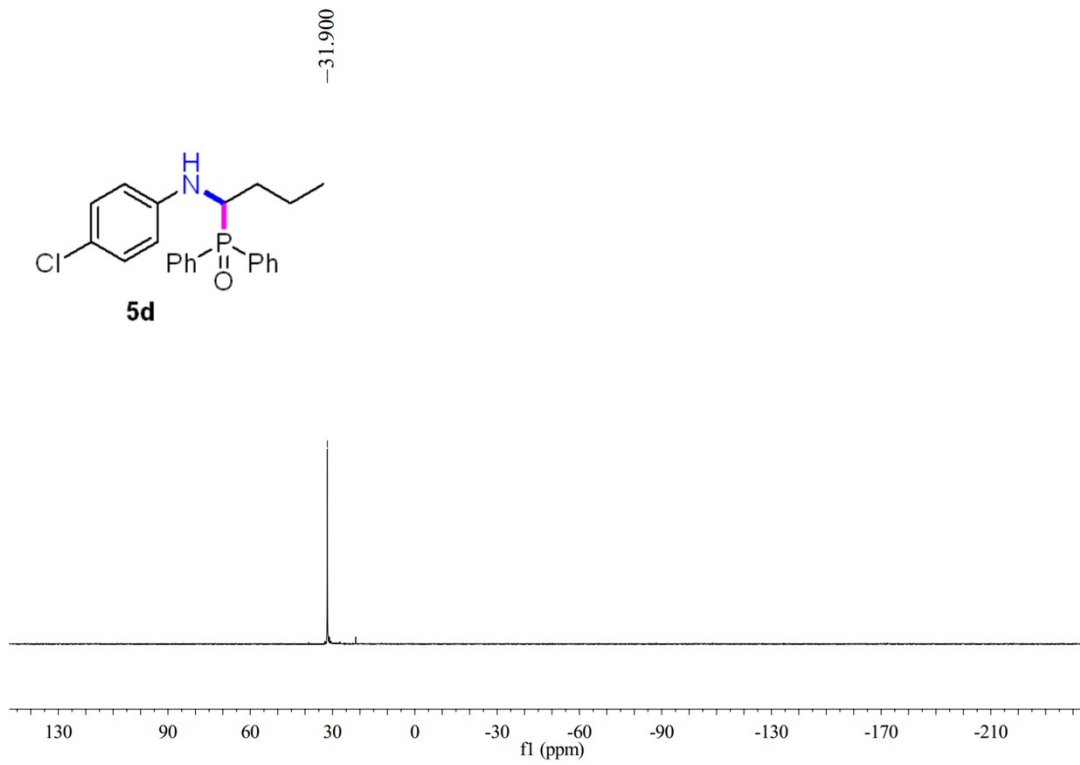
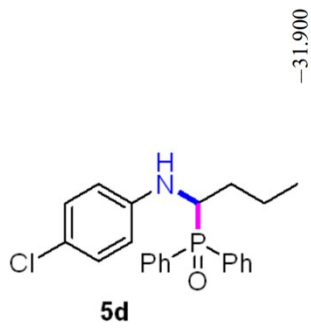
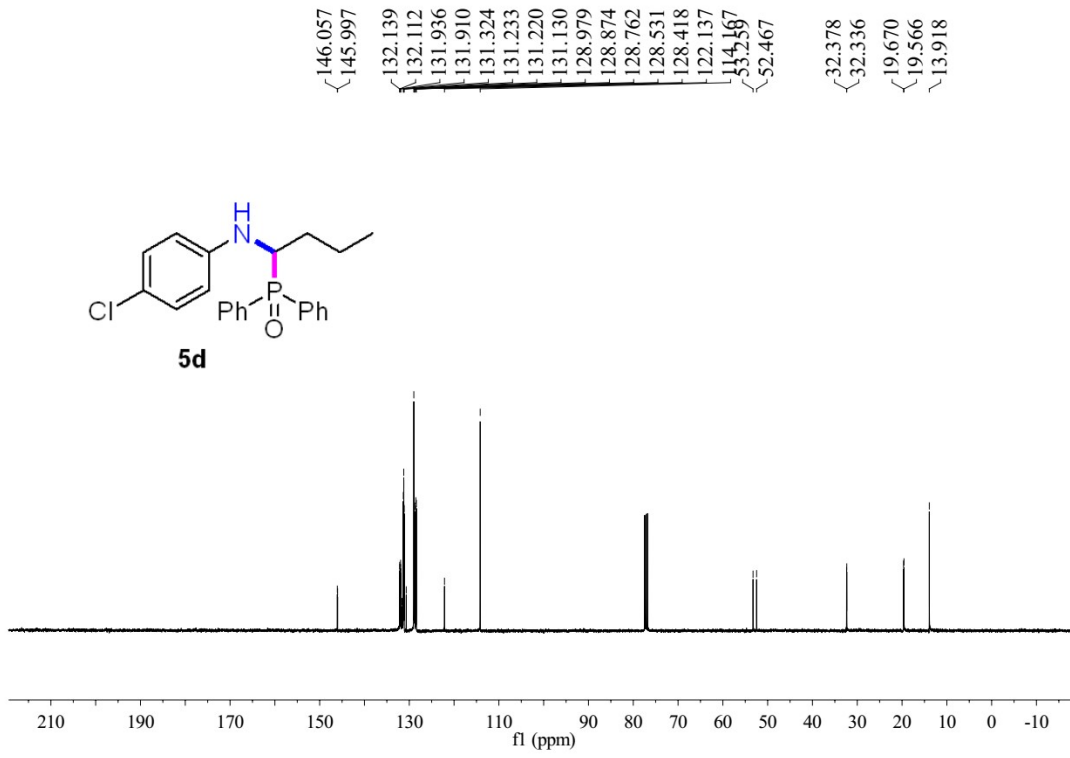
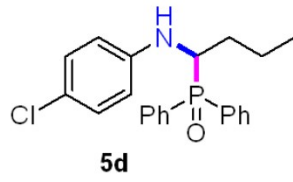


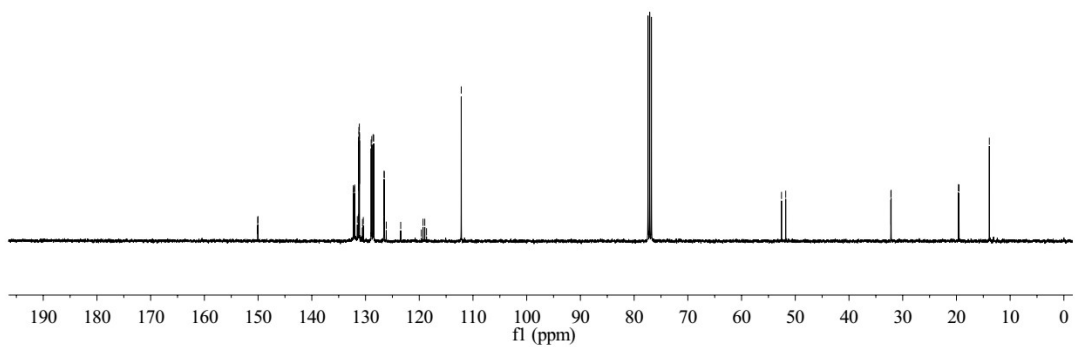
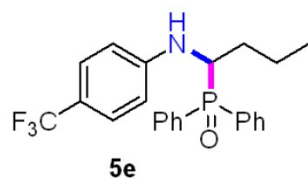
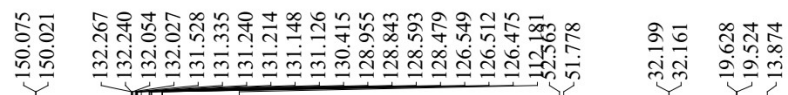
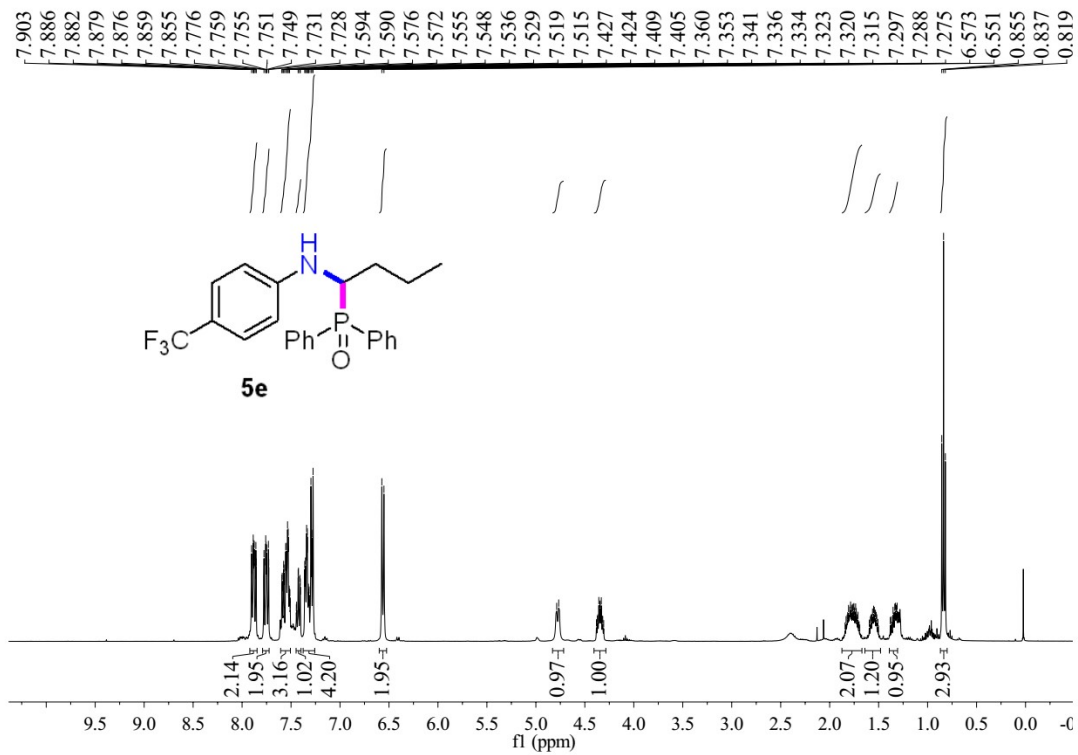
QH02-1625-2

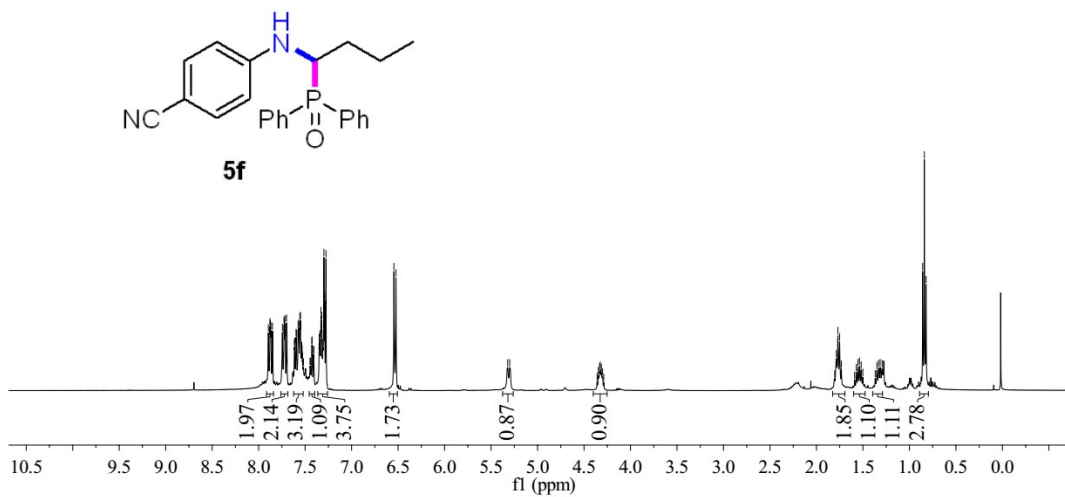
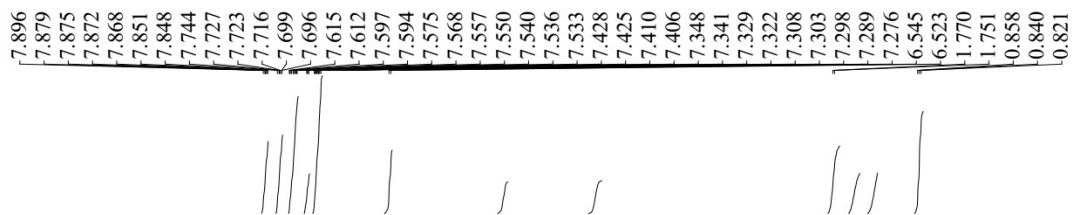
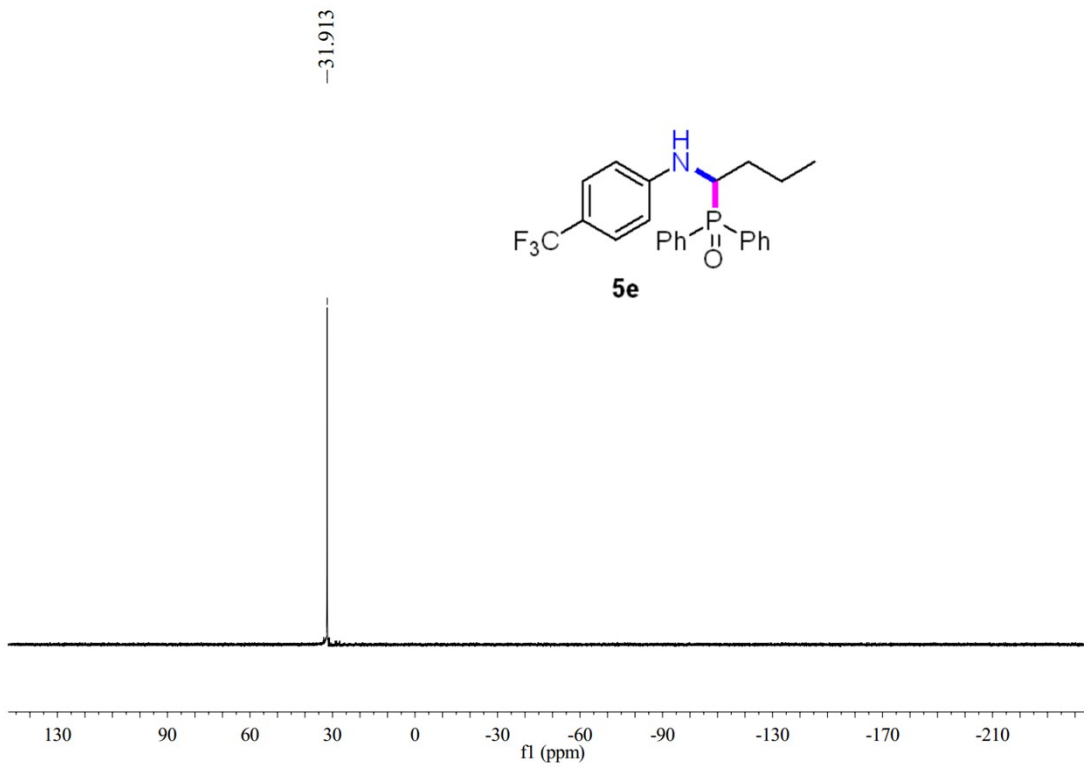


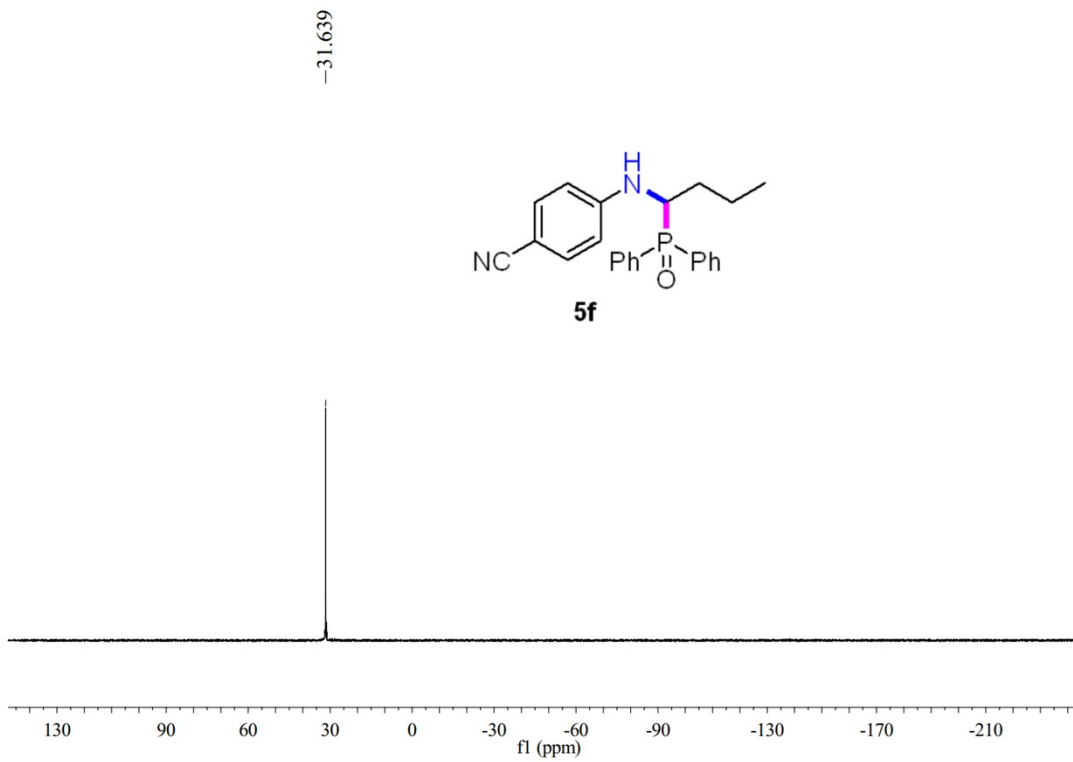
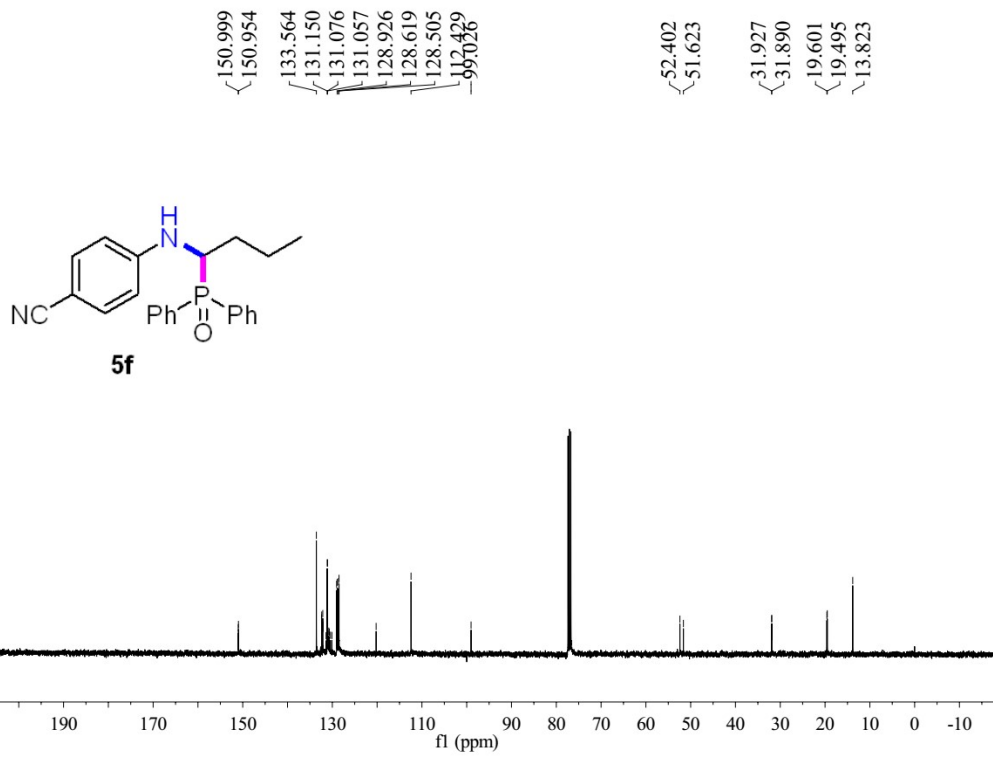


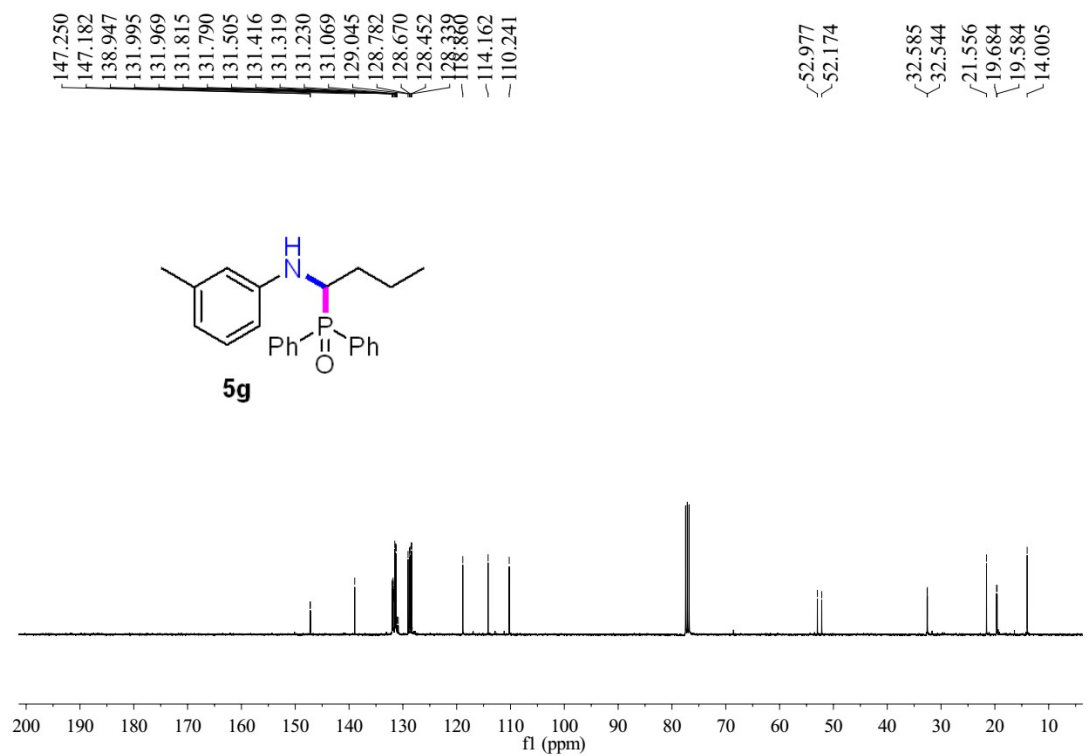
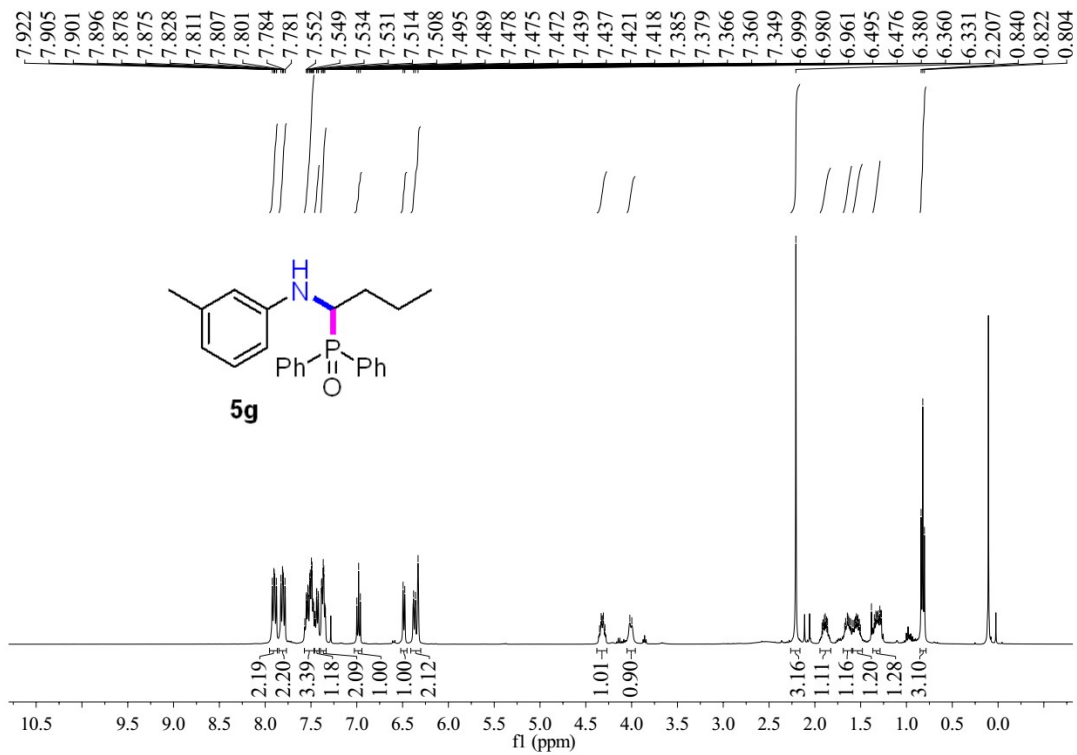




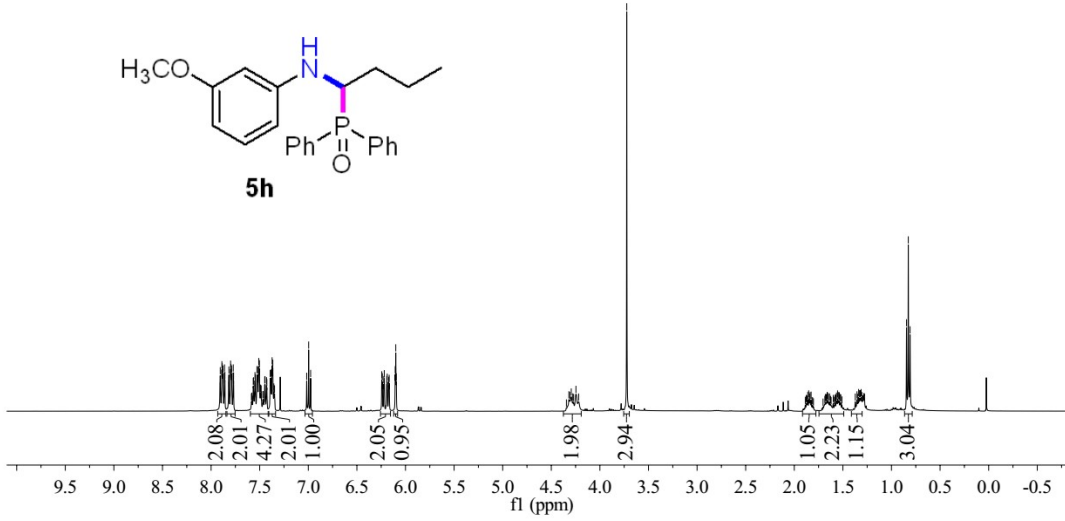
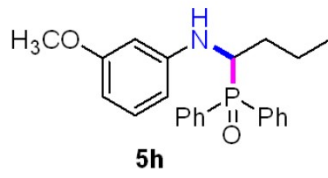
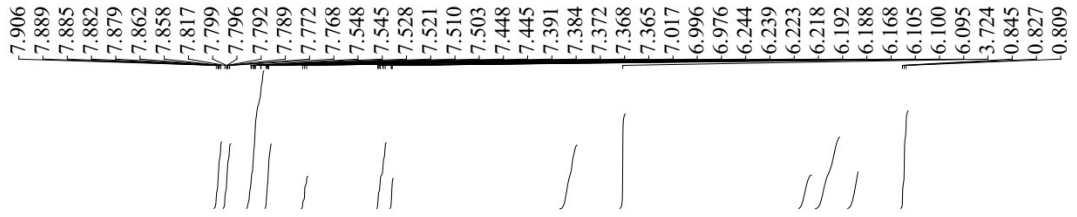
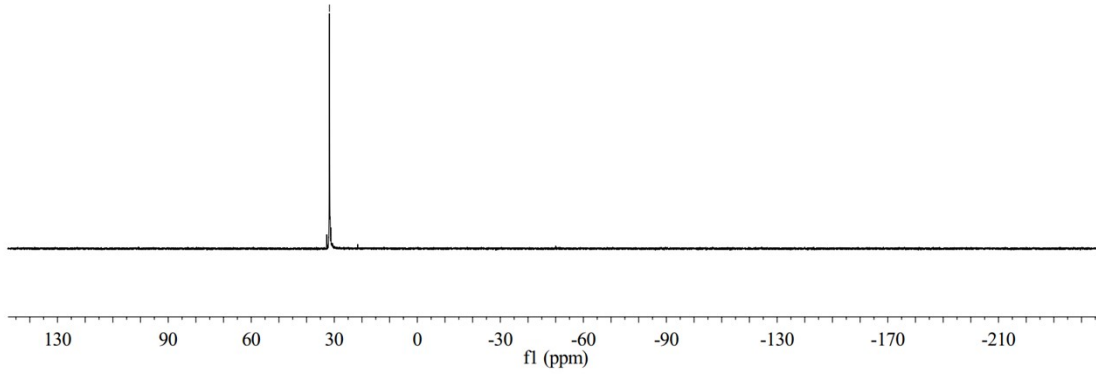
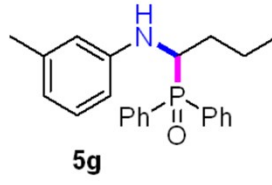


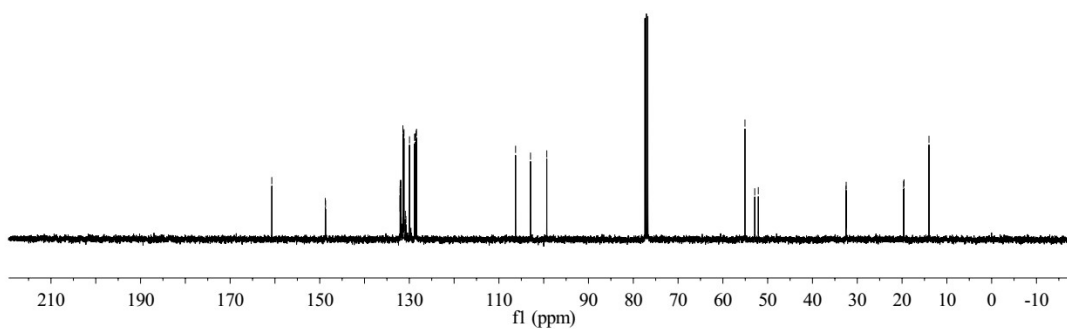
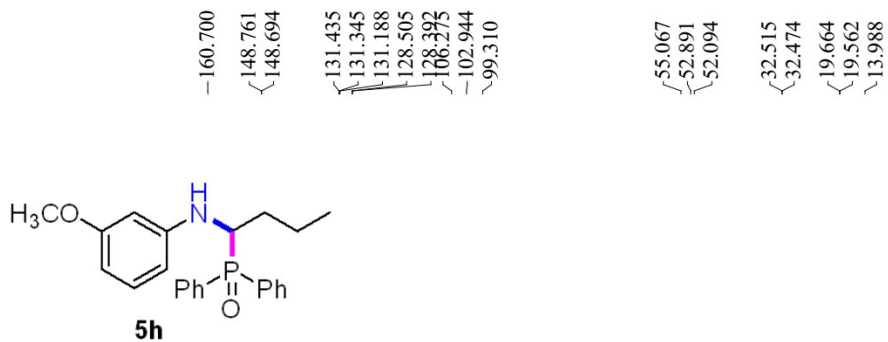




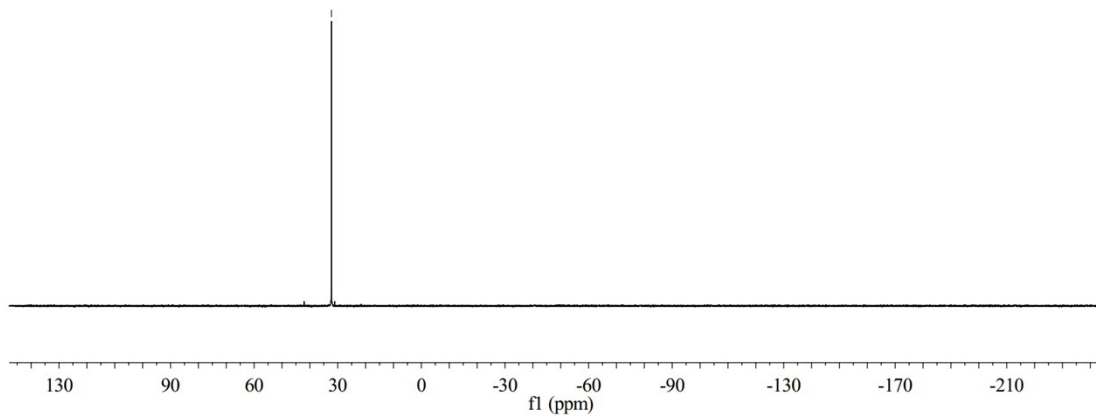
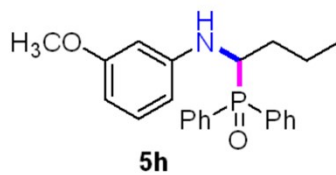


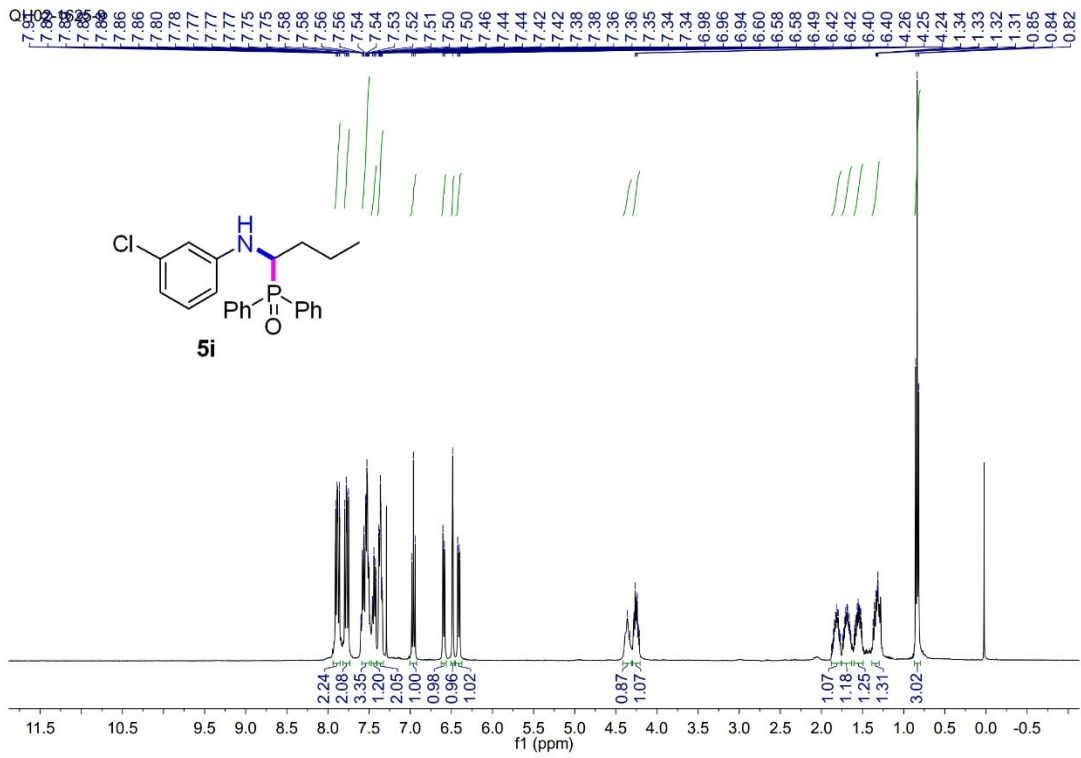
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$-\text{32.251}$





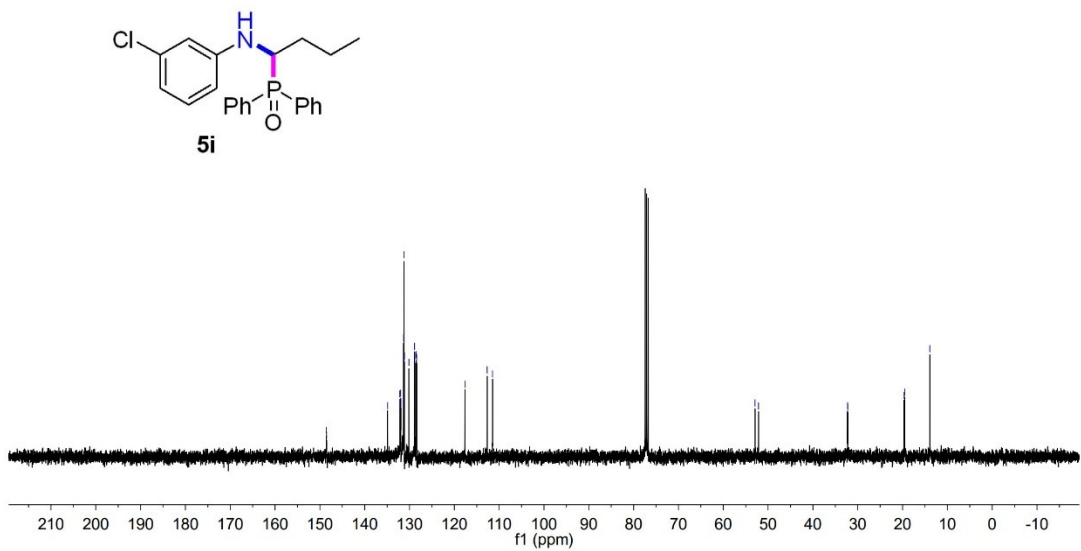
QH02-1625-9

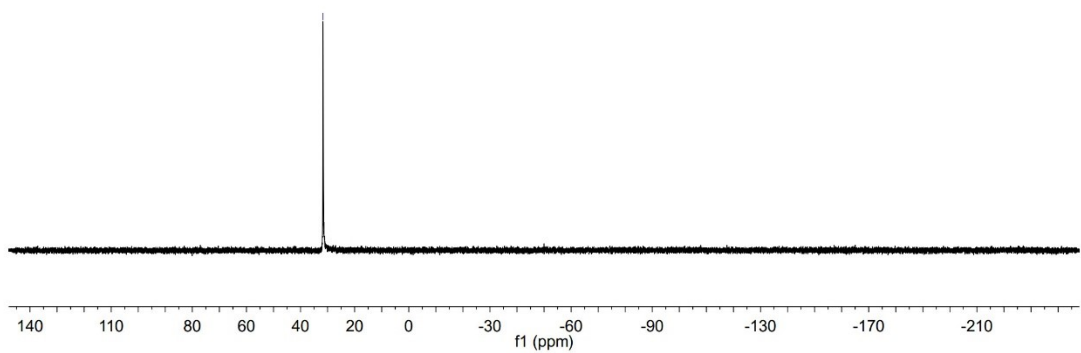
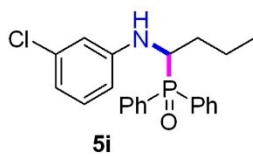
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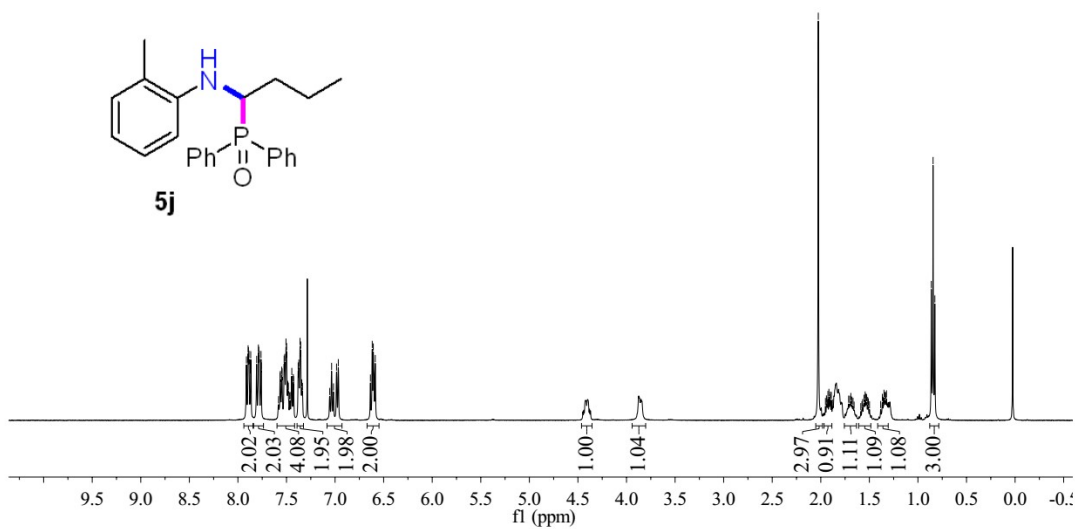
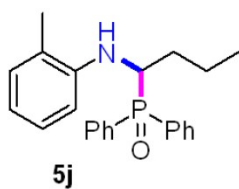
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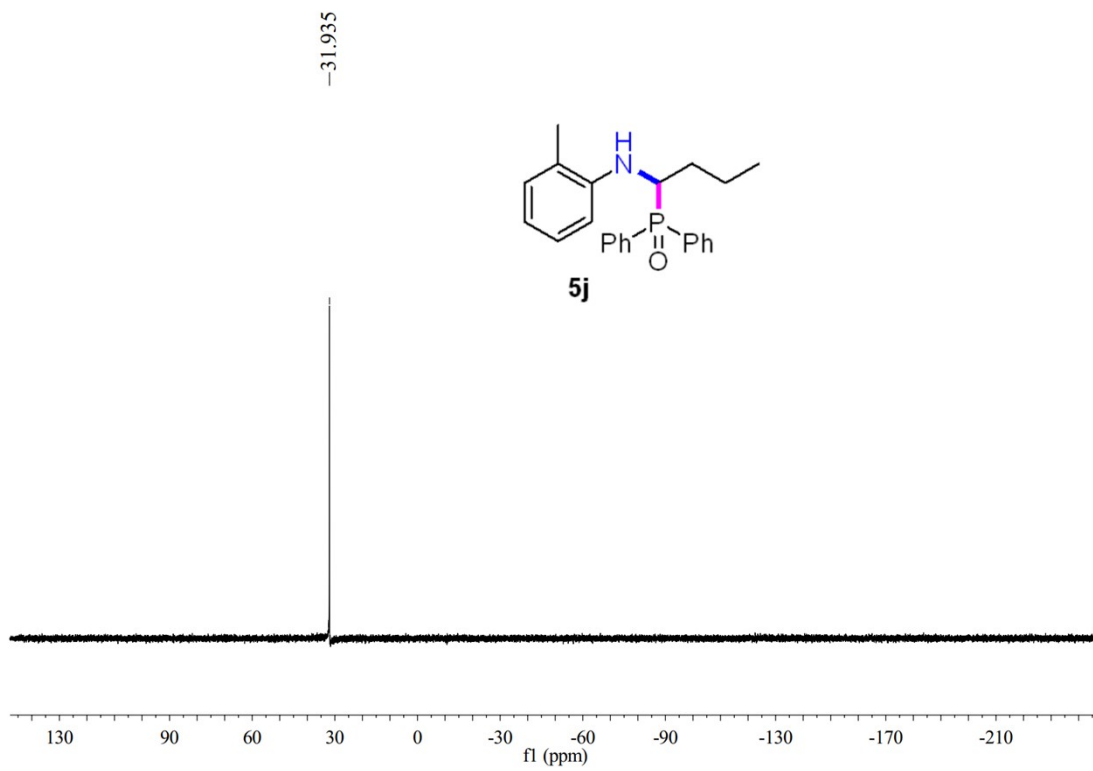
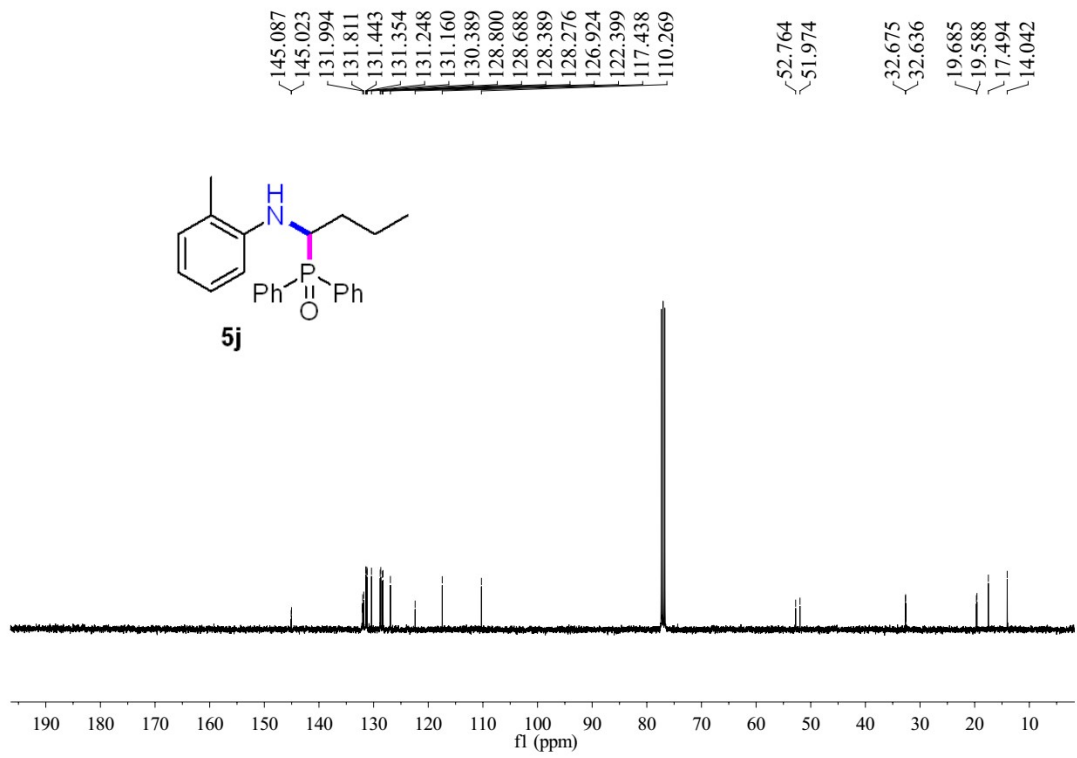
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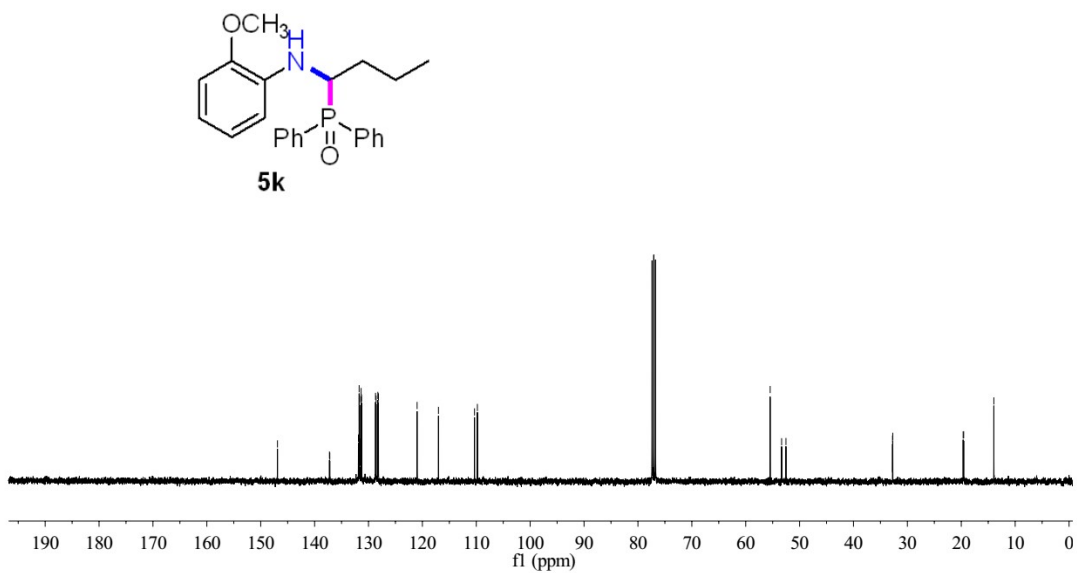
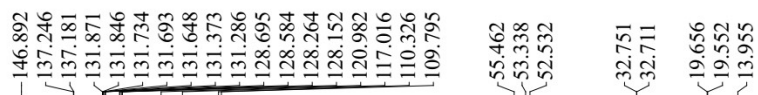
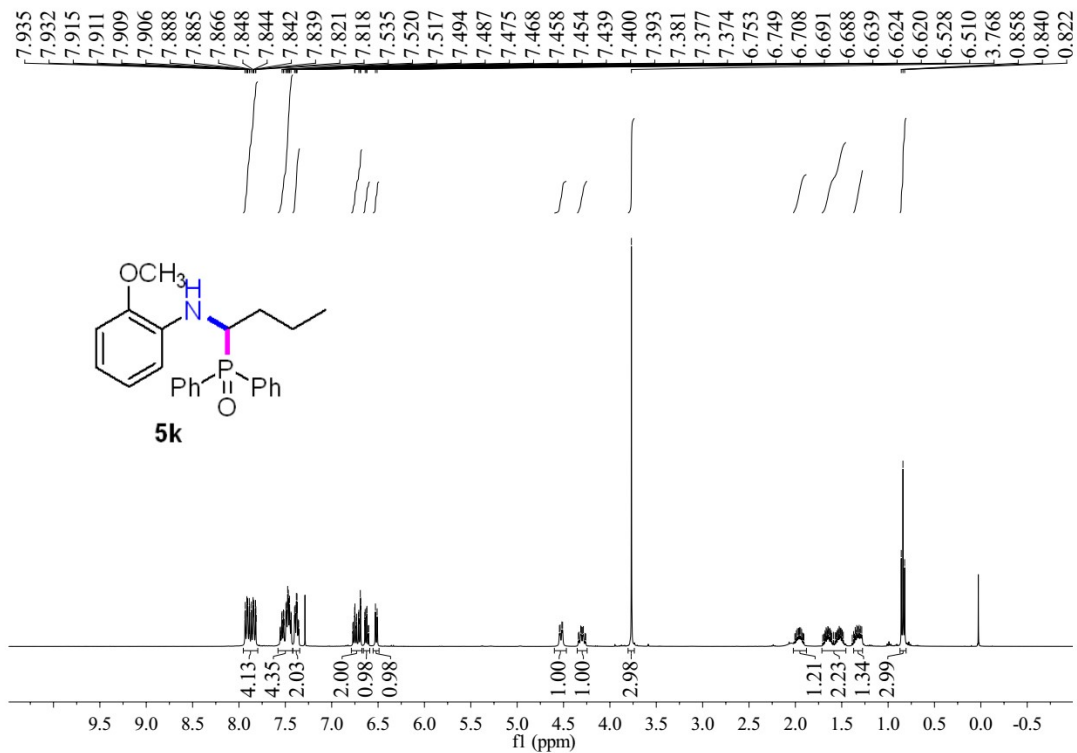


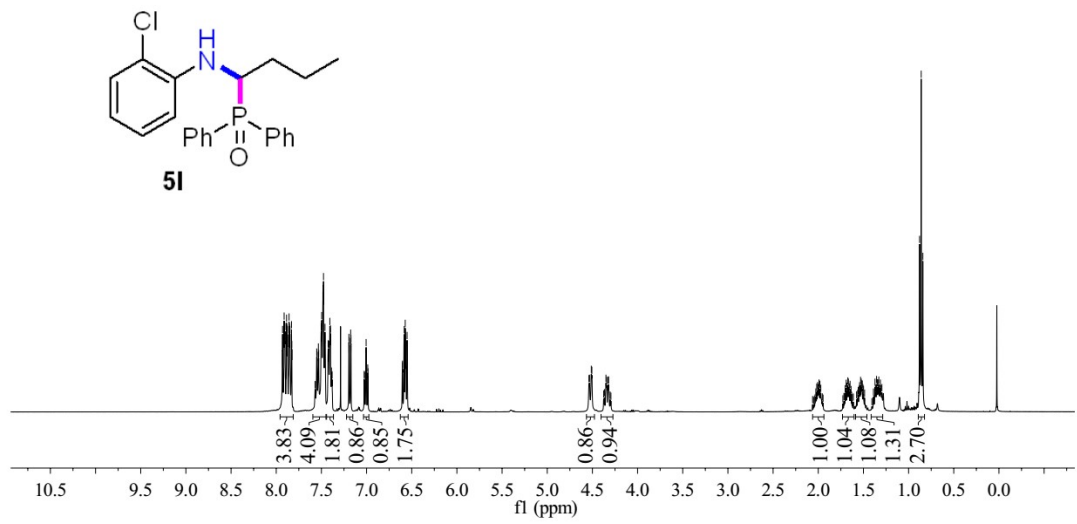
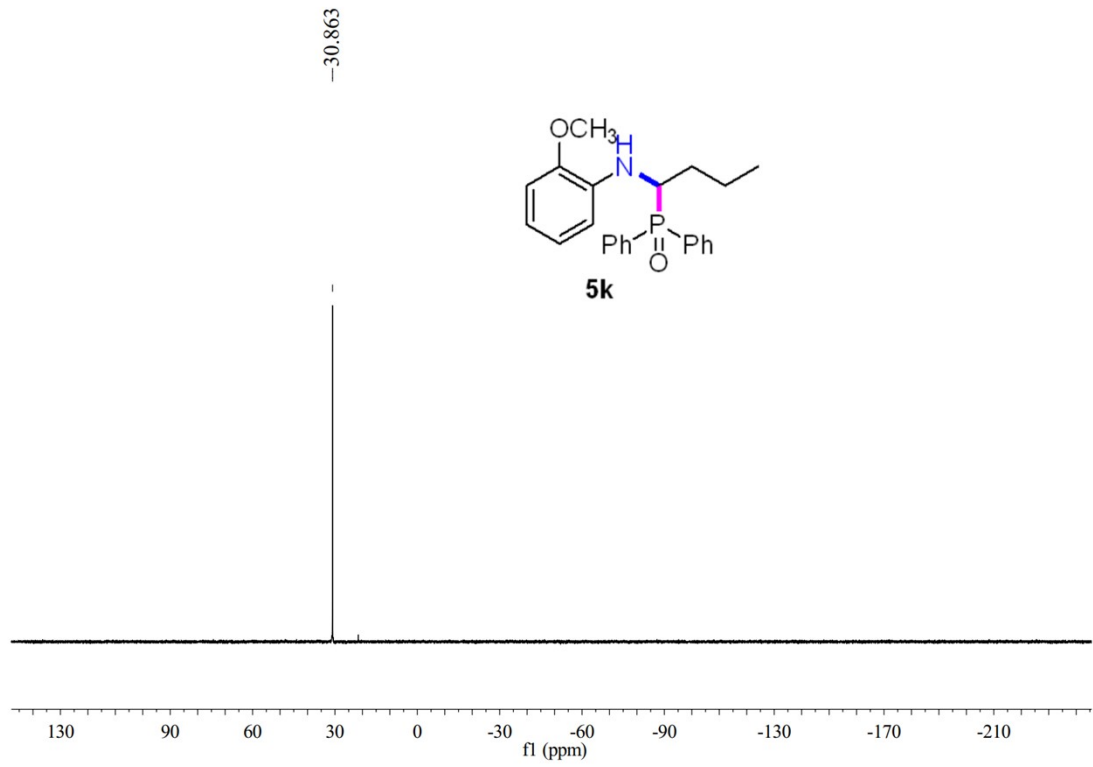


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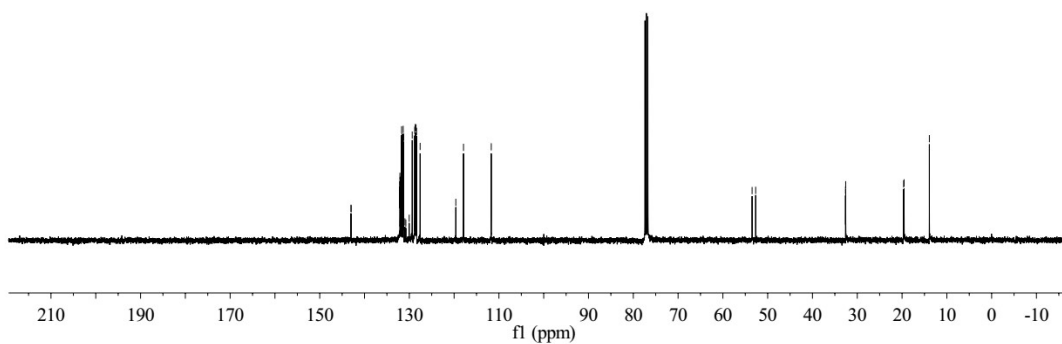
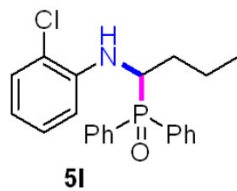




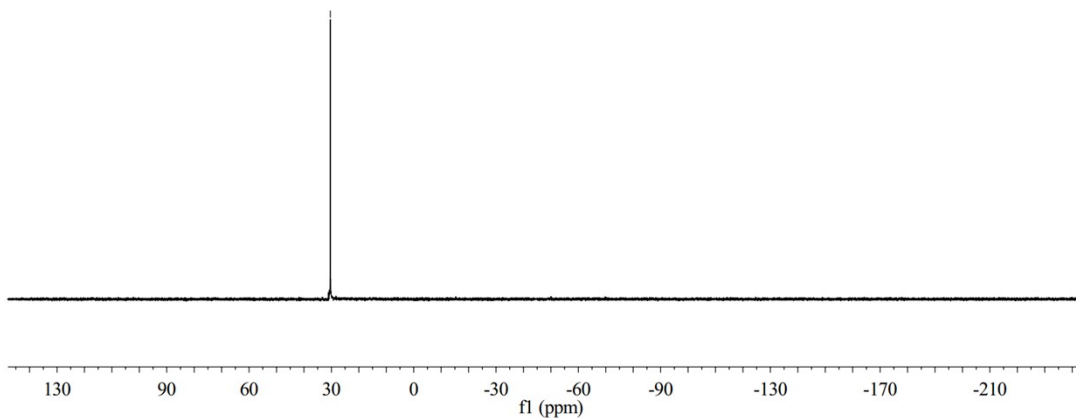
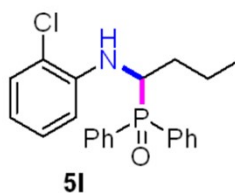


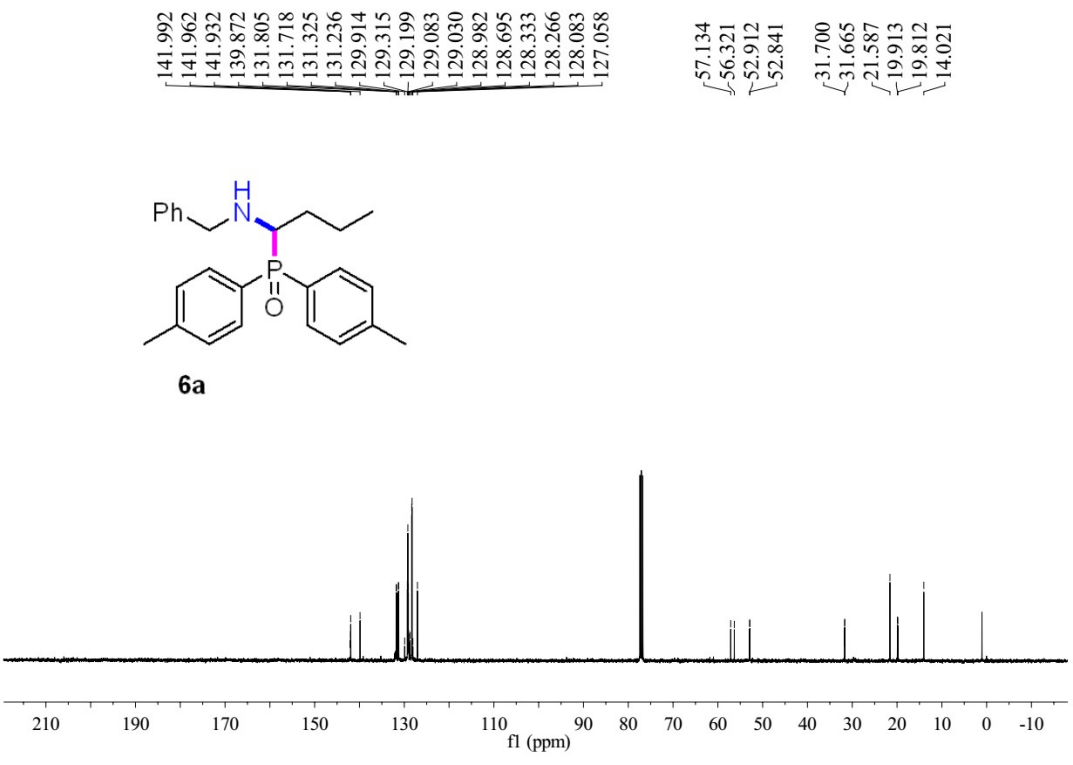
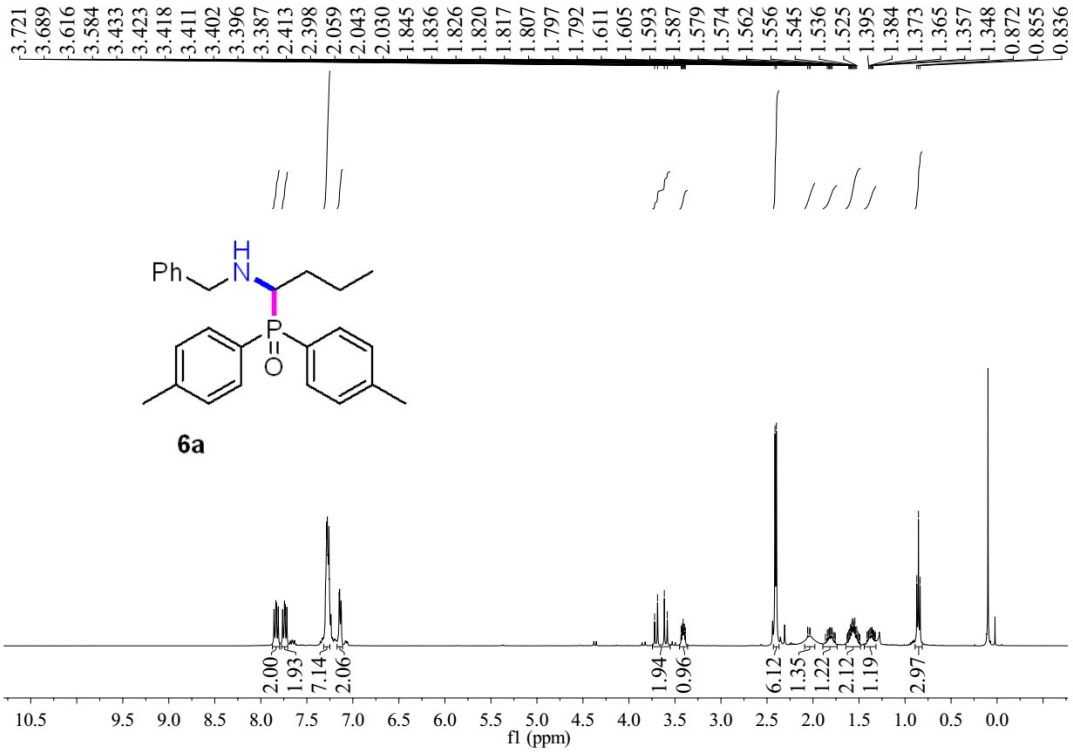


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 132.015
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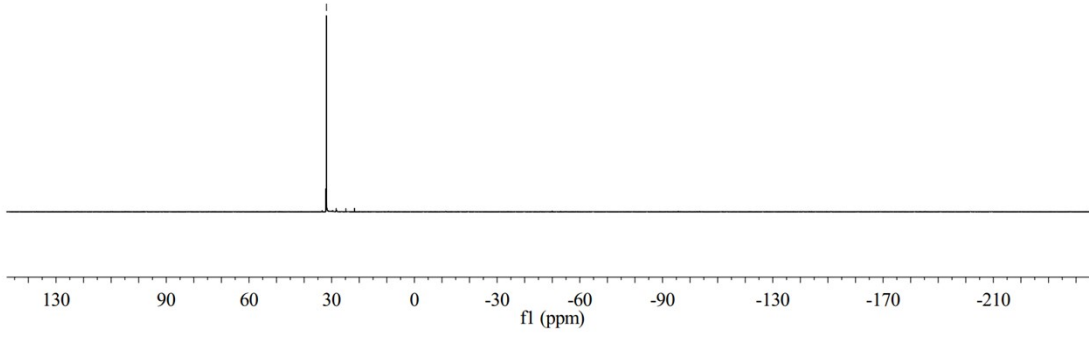
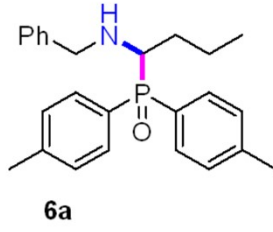


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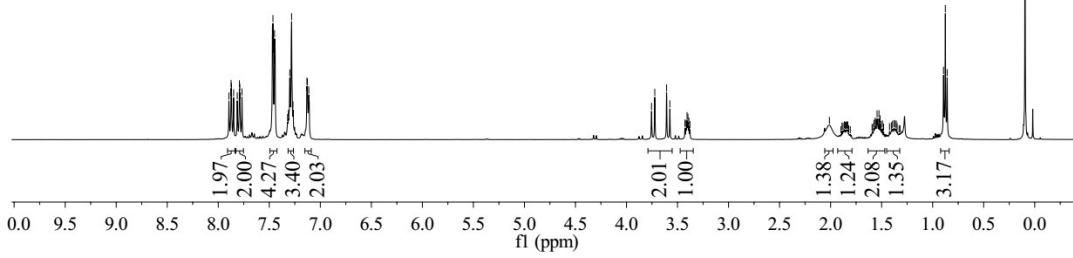
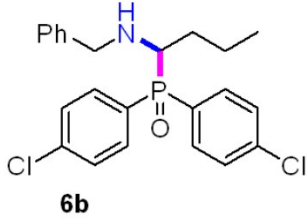




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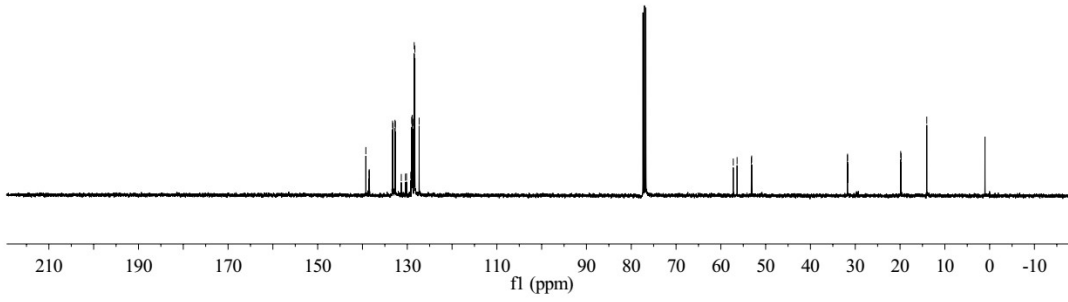
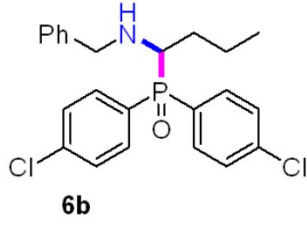
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7.127
7.112
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3.407
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3.388
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0.858



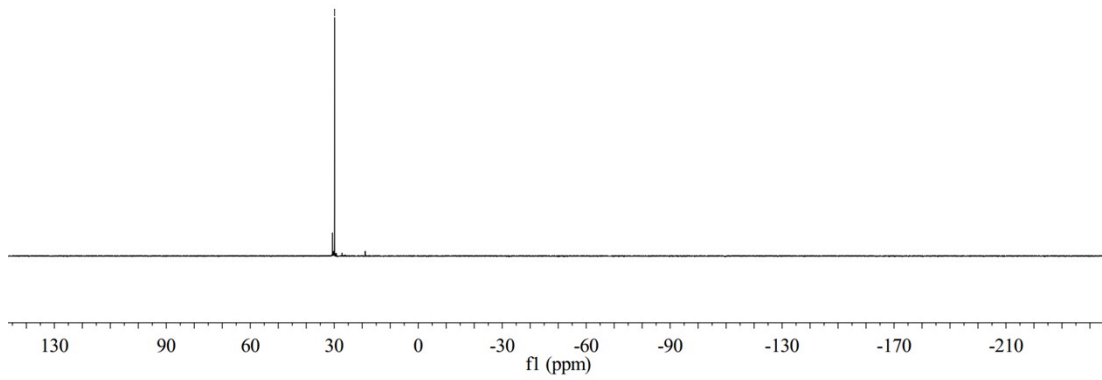
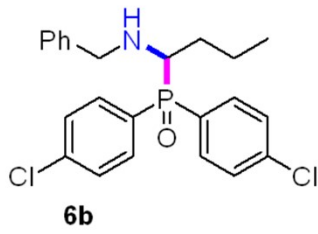
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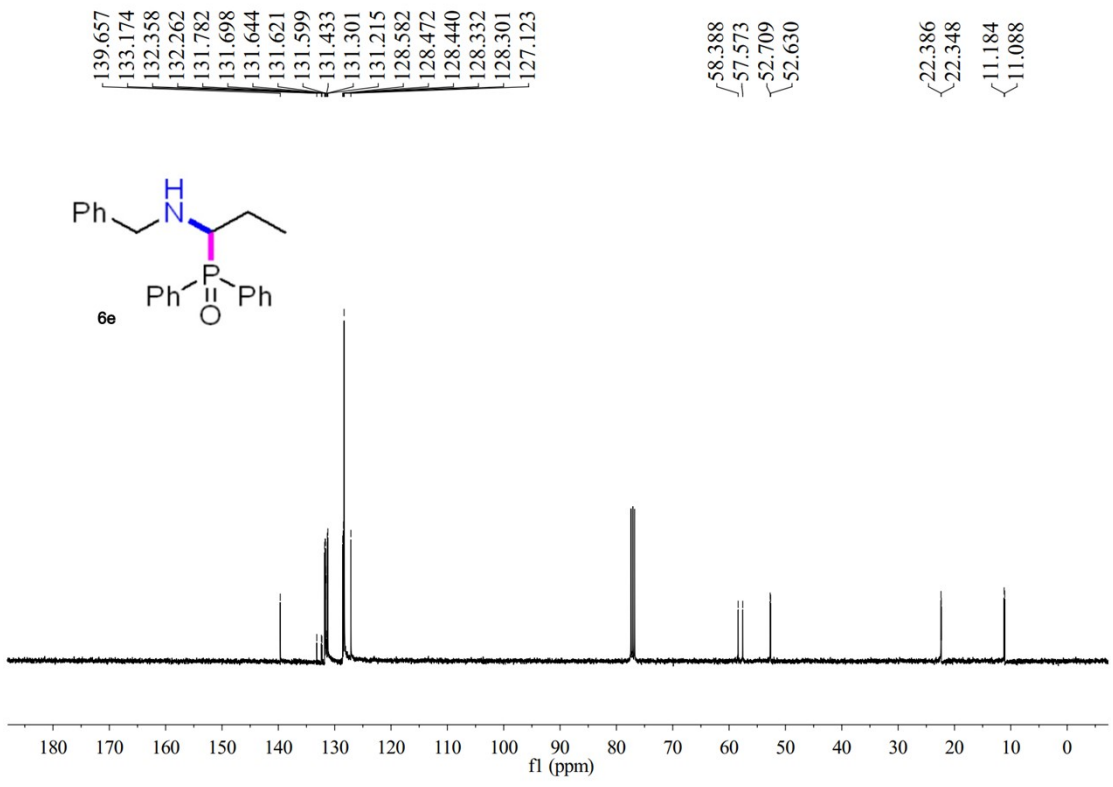
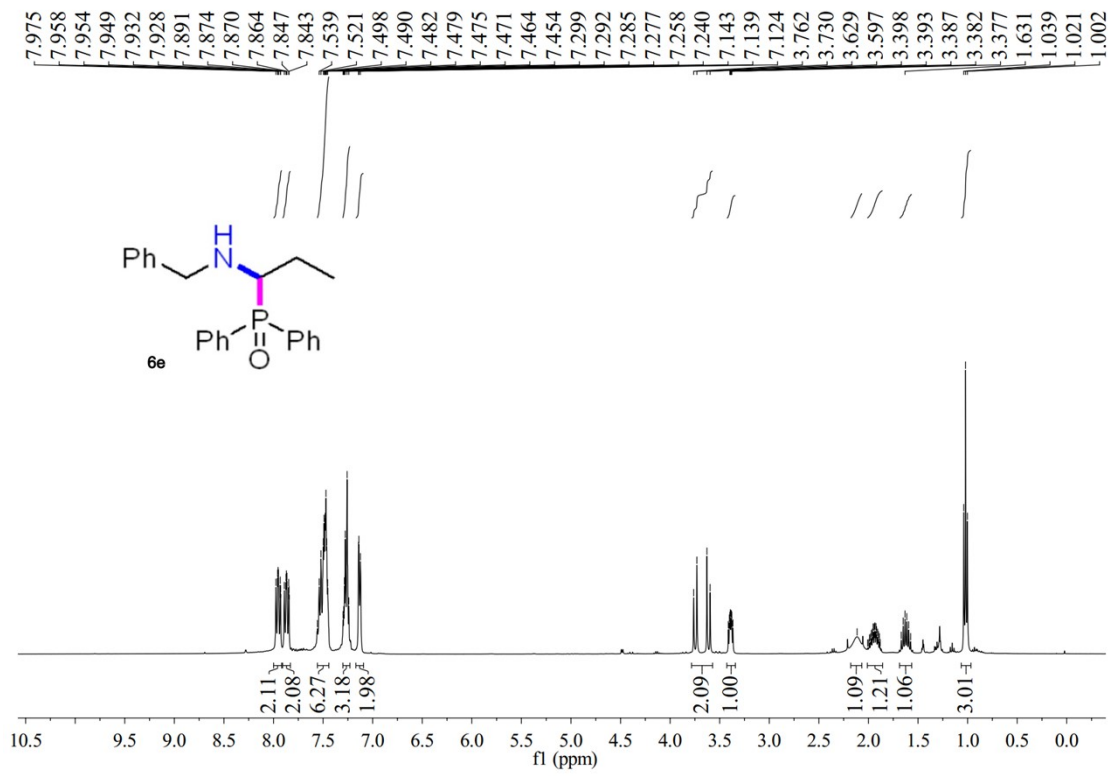
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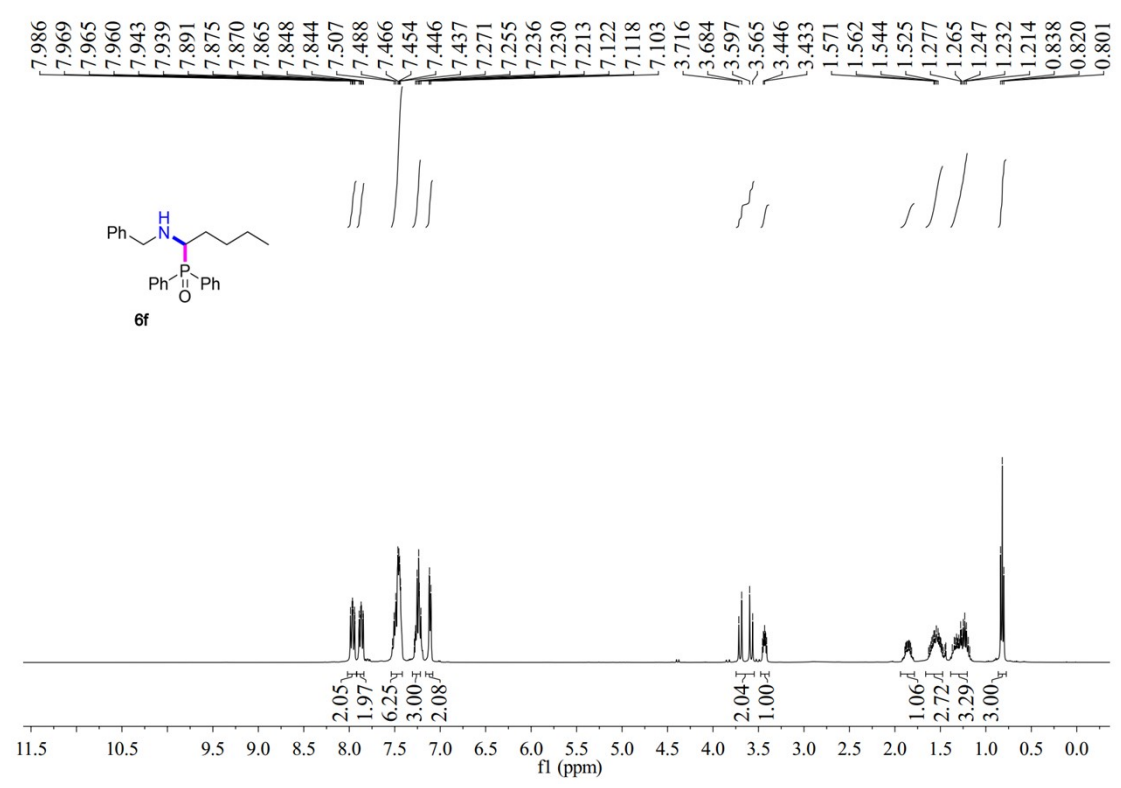
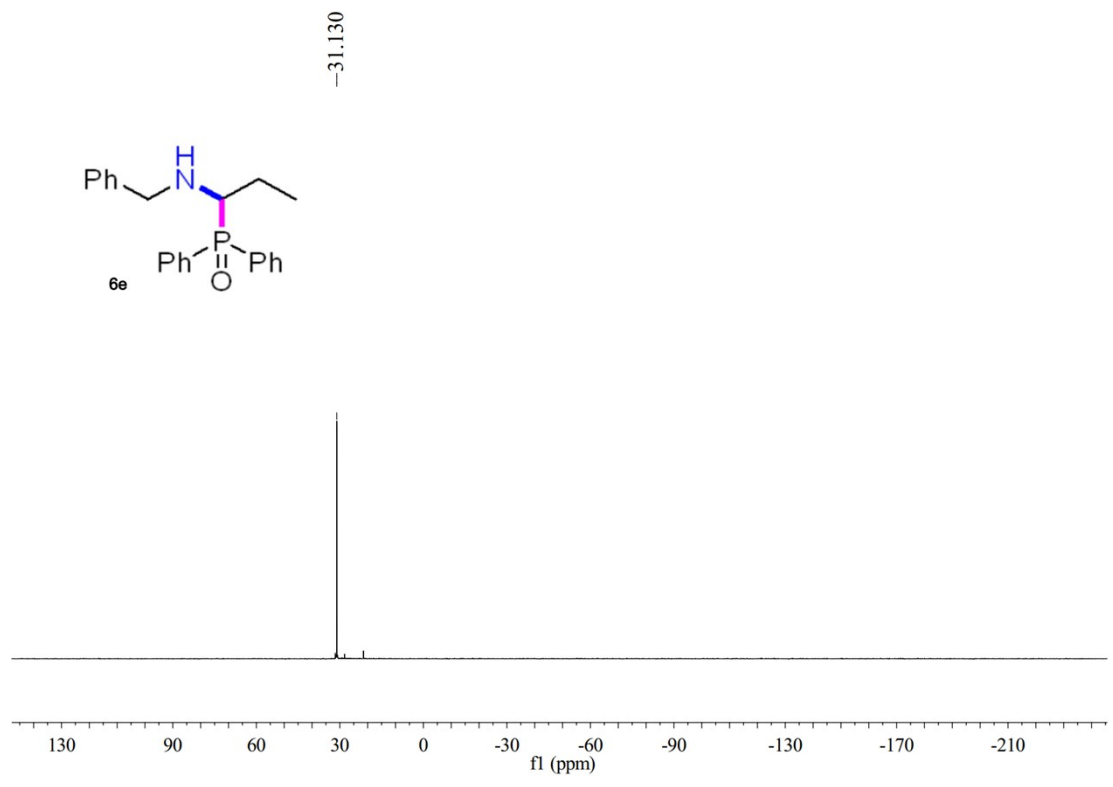
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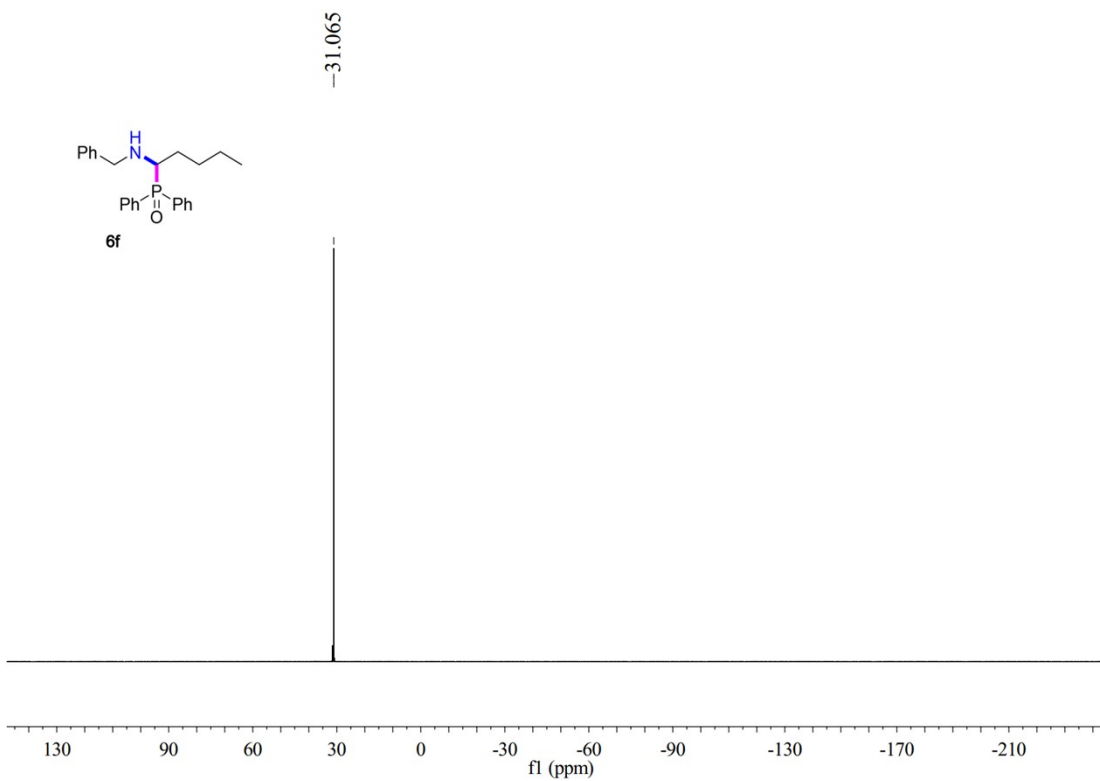
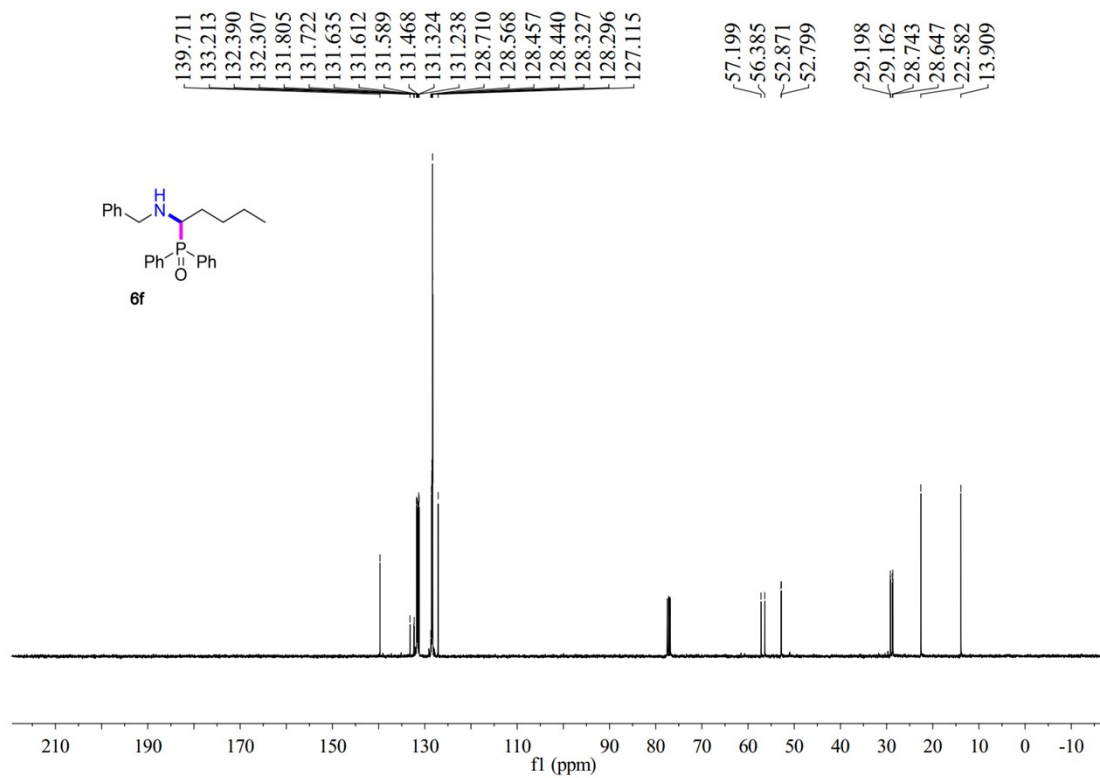


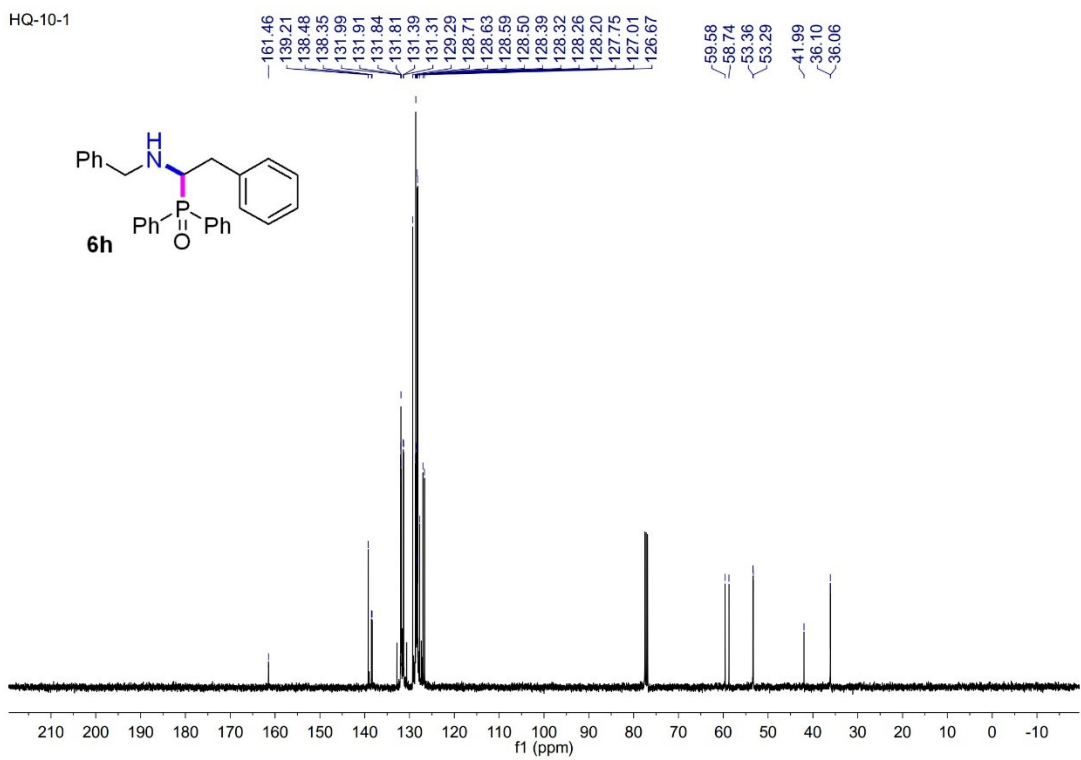
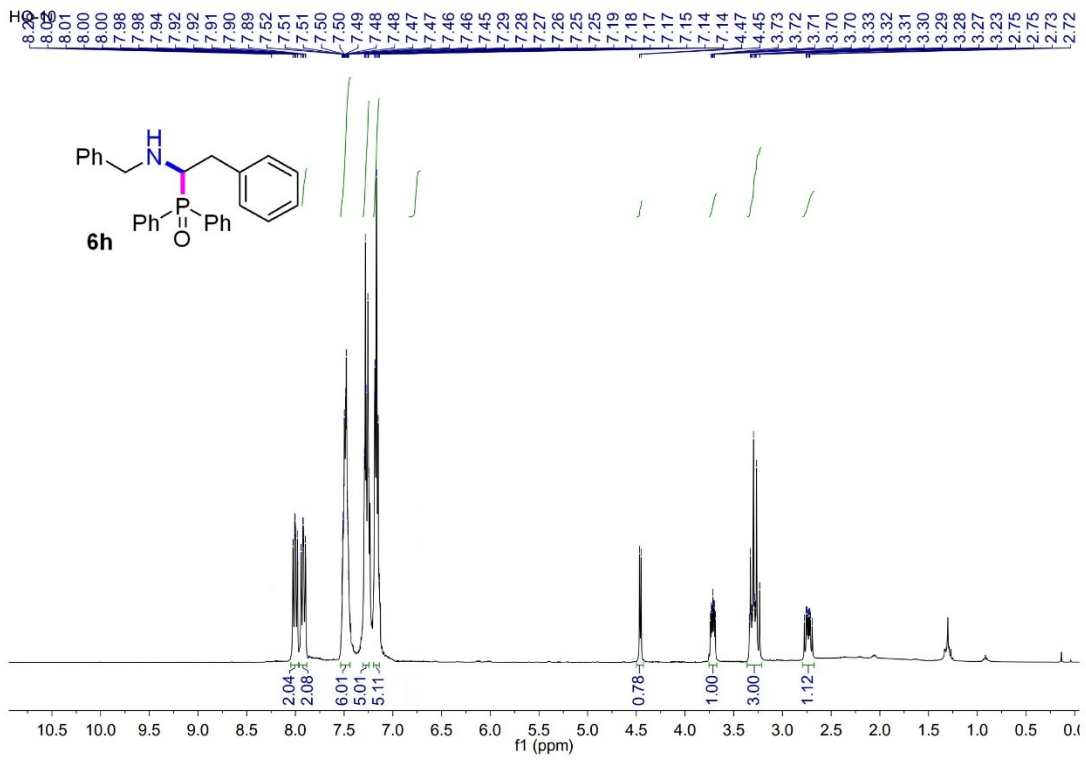
29.906











HQ-10-1

— 30.58

