

Supporting Information

Ph₂PI As A Reduction/Phosphination Reagent: Providing An Easy Access of Phosphine Oxides

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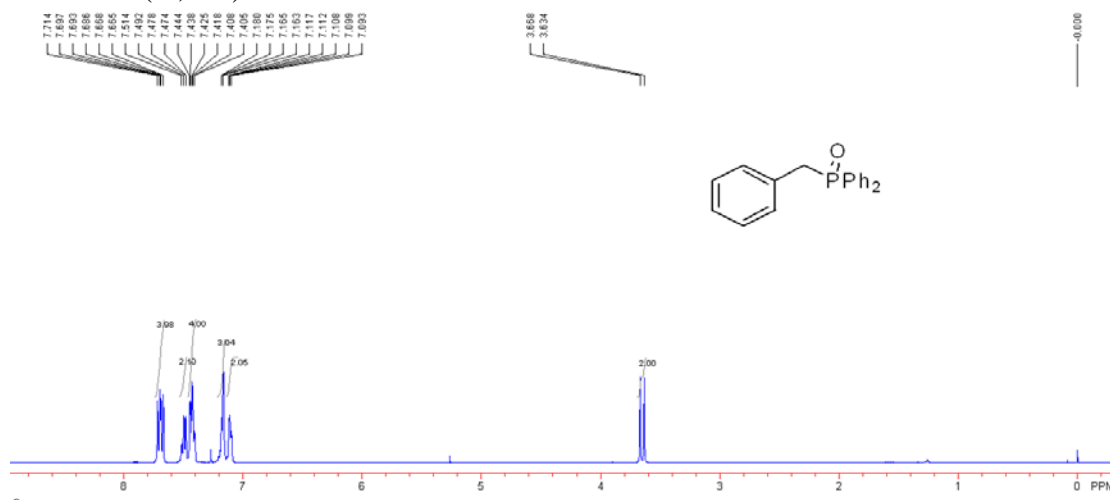
General remarks. Dichloromethane was freshly distilled from calcium hydride; CH₃CN was distilled from calcium hydride (CaH₂) under argon (Ar) atmosphere. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker AM-300 or AM-400 spectrometers. Flash column chromatography was performed using 300-400 mesh silica gel. For thin-layer chromatography (TLC), silica gel plates (Huanghai GF₂₅₄) were used. Mass spectra were recorded by EI and ESI, and HRMS were measured on a HP-5989 instrument.

General procedure for the reaction of aldehyde with Ph₂PI

To a stirred mixture of Ph₂PCl (4.5 mmol), NaI (4.5 mmol) and anhydrous CH₃CN (5 mL) was added aldehyde **2** (1.0 mmol) at room temperature under argon atmosphere. The reaction mixture was stirred at 80 °C in oil bath for 24 h. When the reaction temperature down to room temperature, 30% H₂O₂ aqueous (0.5 ml) was slowly added. After 10 minutes, sat. aqueous Na₂S₂O₃ was added to the reaction mixture. The organic layer was extracted with dichloromethane, washed with brine, dried over MgSO₄, and concentrated under reduced pressure. The residue was purified by chromatography on silica gel to obtain the corresponding phosphine oxide.

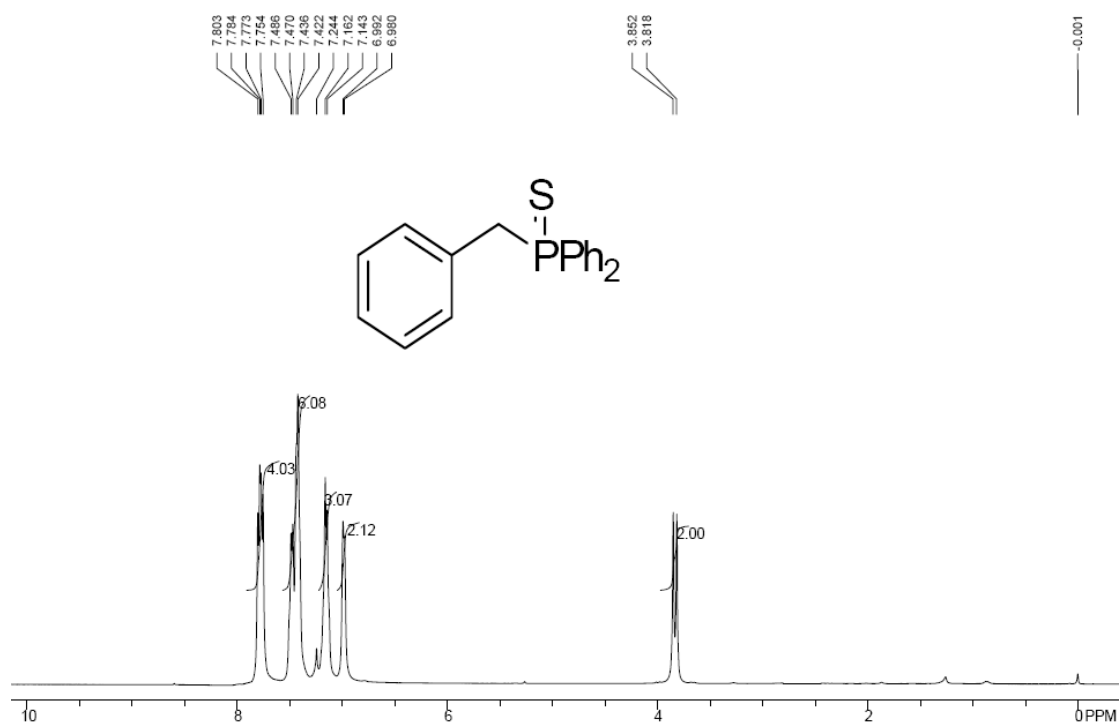
Benzyl diphenylphosphine oxide **3aa** ^[1]

White solid; ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.65 (d, *J* = 13.6 Hz, 2H), 7.09-7.12 (m, 2H), 7.16-7.18 (m, 3H), 7.41-7.44 (m, 4H), 7.47-7.51 (m, 2H), 7.67-7.71 (m, 4H).



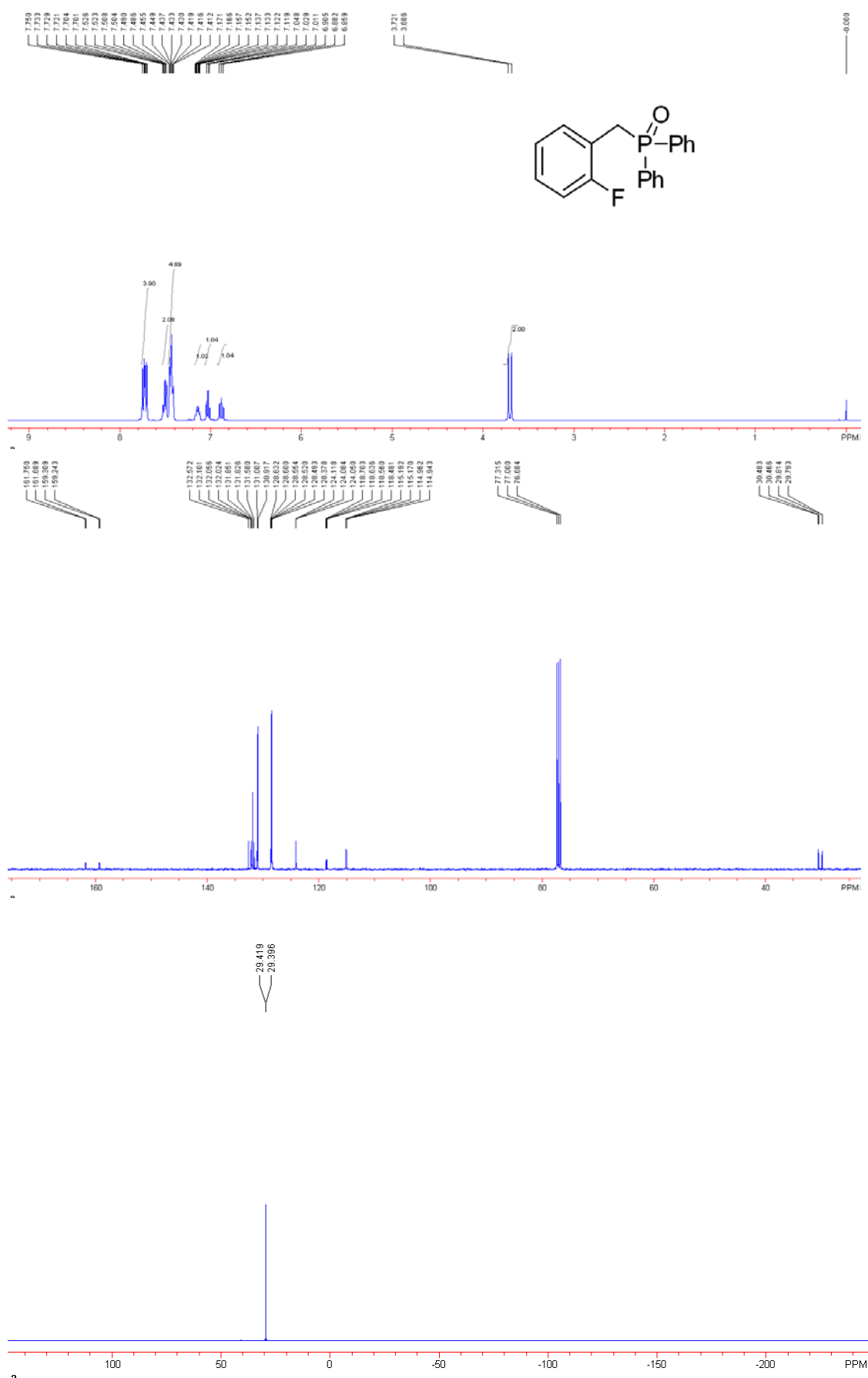
Benzyl diphenylphosphine sulfoxide **4a** ^[2]

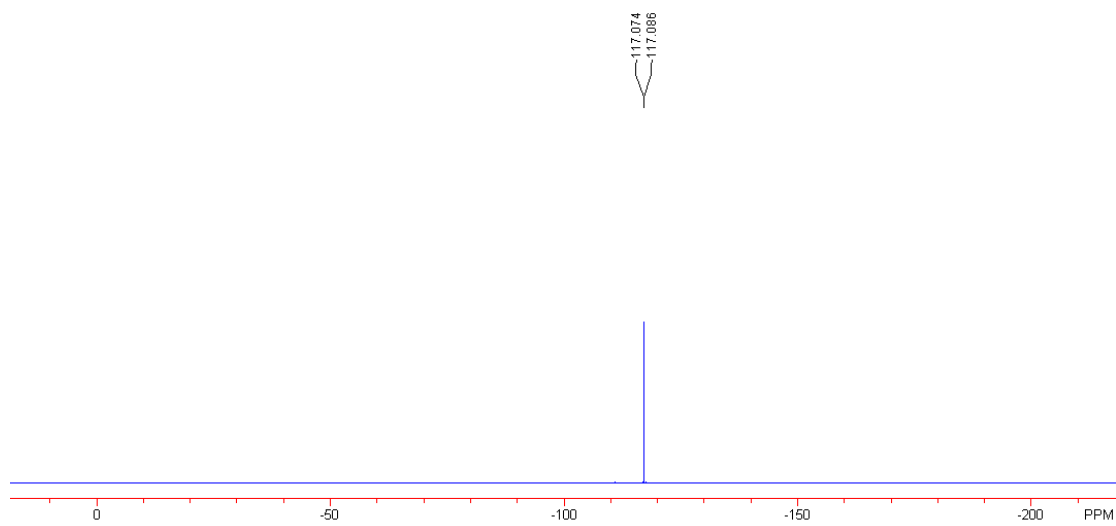
White solid, ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.84 (d, *J* = 13.6 Hz, 2H), 6.99 (d, *J* = 4.8 Hz, 2H), 7.14-7.16 (m, 3H), 7.42-7.49 (m, 6H), 7.75-7.80 (m, 4H).



2-Fluorobenzyl diphenylphosphine oxide **3ab**

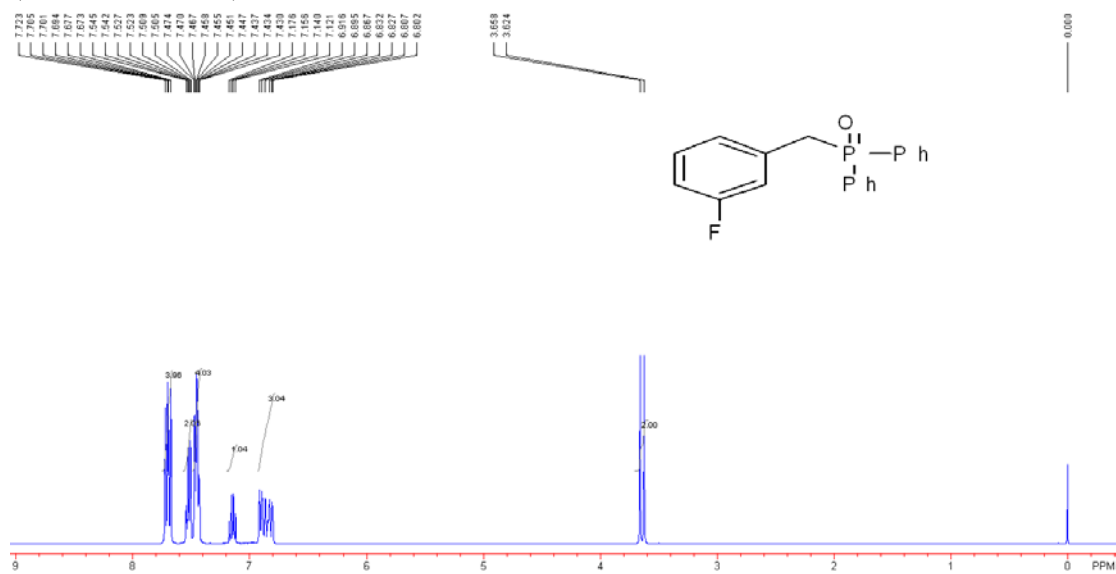
Light yellow solid, Mp 150.0-150.2 °C, ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.70 (d, *J* = 14.0 Hz, 2H), 6.88 (t, *J* = 9.2 Hz, 1H), 7.03 (t, *J* = 7.2 Hz, 1H), 7.12-7.17 (m, 1H), 7.41-7.46 (m, 5H) 7.49-7.53 (m, 2H), 7.70-7.75 (m, 4H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 29.79, 29.81, 30.47, 30.48, 114.94, 114.96, 115.17, 115.19, 118.5, 118.6, 118.64, 118.7, 124.05, 124.08, 124.1, 128.4, 128.49, 128.52, 128.55, 128.6, 128.63, 130.9, 131.0, 131.6, 131.8, 131.9, 132.02, 132.06, 132.1, 132.6, 159.2, 159.3, 161.7, 161.8. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 29.40, 29.42. ¹⁹F NMR (470 MHz, CDCl₃, TMS) δ -117.09, -117.07; MS (ESI) *m/z* (%): 311.1 (M + H, 100); HRMS (Micromass LCT) Calcd for C₁₉H₁₇OFP: 311.1001, Found: 311.1000.

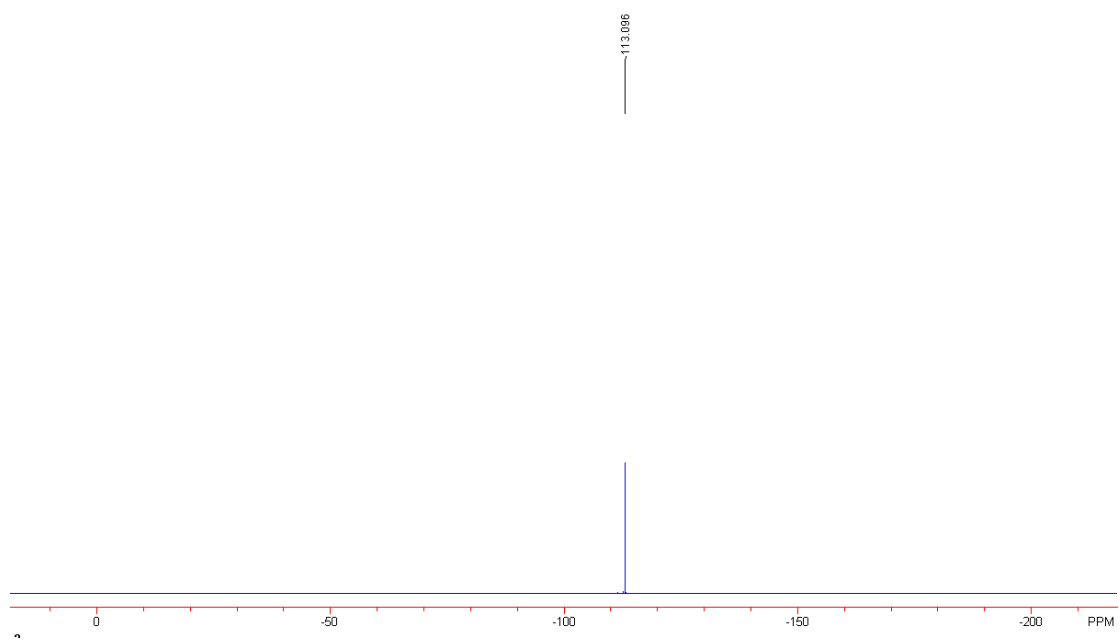
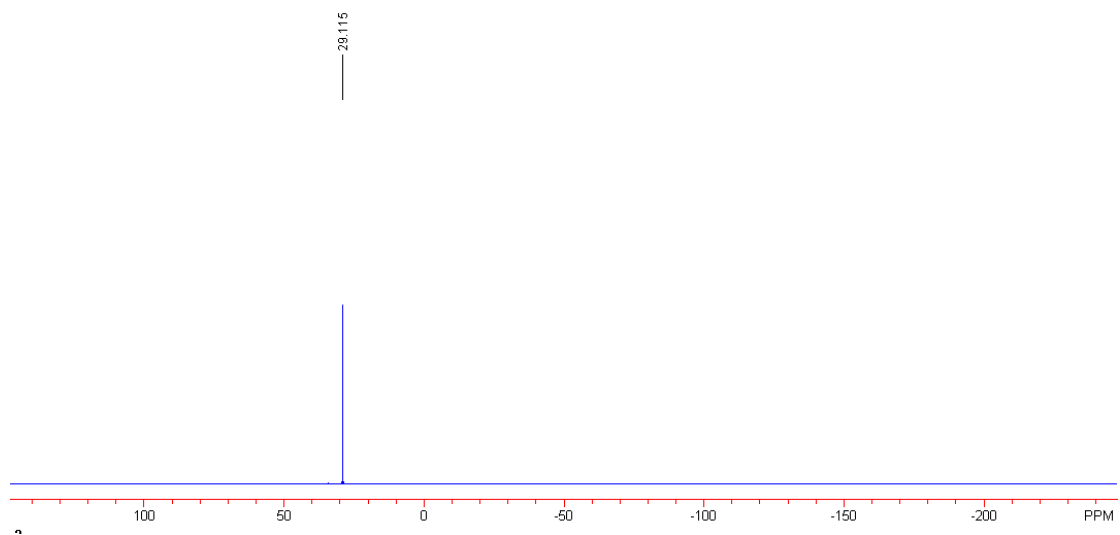
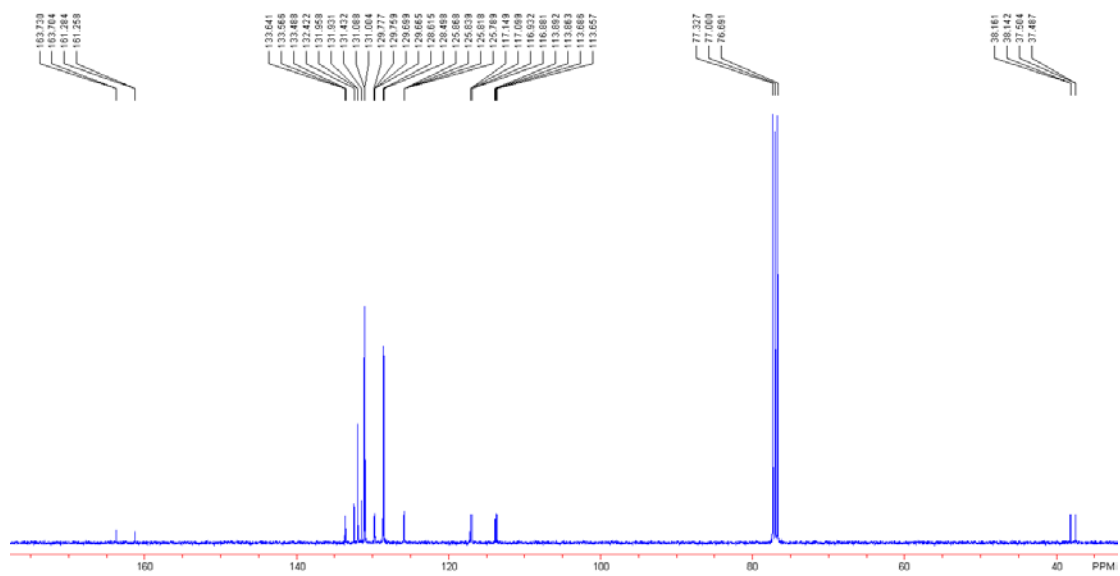




3-Fluorobenzyl diphenylphosphine oxide **3ac**

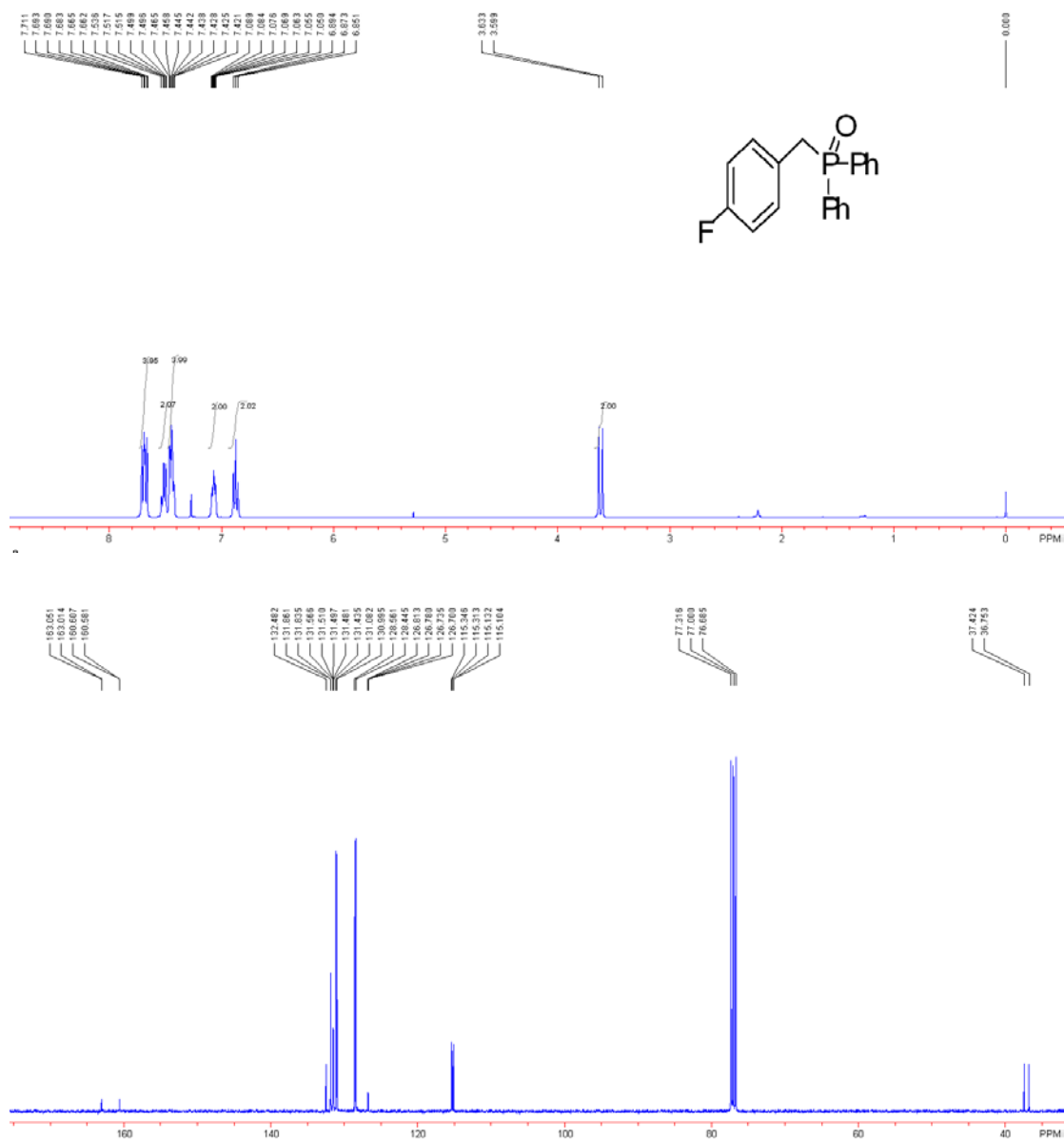
Light yellow solid, Mp 179.5-180.3 °C ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.64 (d, $J = 13.6$ Hz, 2H), 6.80-6.92 (m, 3H), 7.15 (dd, $J = 8.0, 14.4$ Hz, 1H), 7.43-7.47 (m, 4H), 7.51-7.55 (m, 2H), 7.67-7.72 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 37.49, 37.50, 38.1, 38.2, 113.66, 113.69, 113.86, 113.89, 116.88, 116.93, 117.10, 117.15, 125.79, 125.82, 125.84, 125.87, 128.5, 128.6, 129.67, 129.70, 129.76, 129.78, 131.0, 131.09, 131.4, 131.93, 131.96, 132.4, 133.5, 133.57, 133.64, 161.26, 161.28, 163.70, 163.73. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 29.1. ^{19}F NMR (470 MHz, CDCl_3 , TMS) δ -113.1. MS (ESI) m/z (%): 311.1 (M + H, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{19}\text{H}_{17}\text{OFP}$: 311.1001, Found: 311.0999.

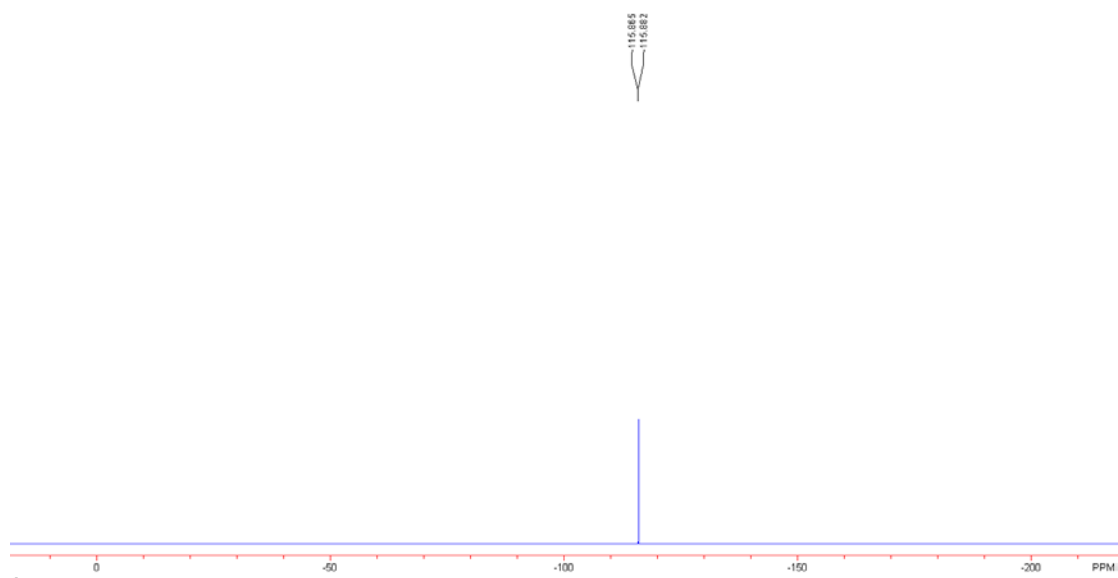
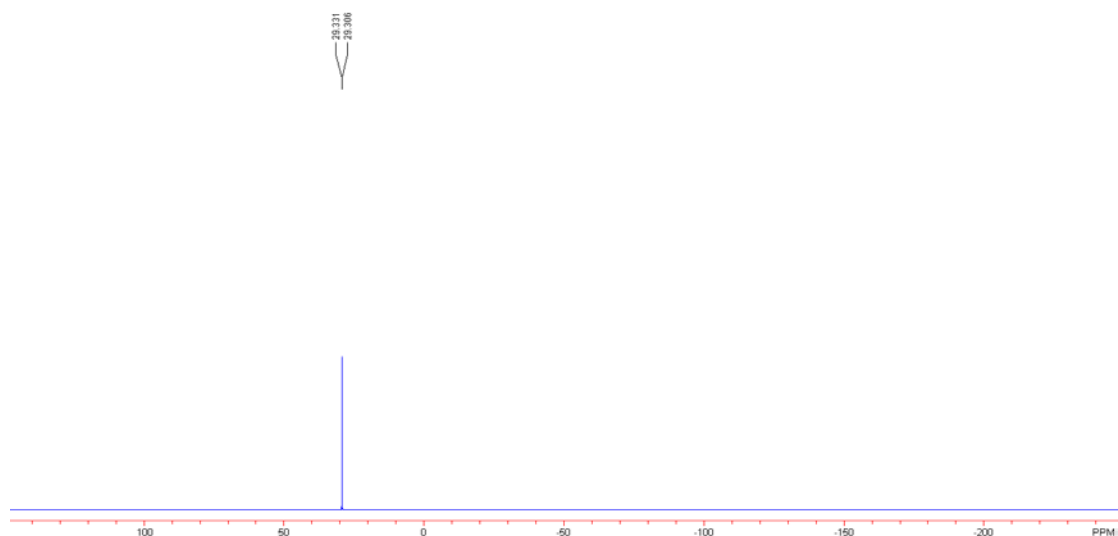




4-Fluorobenzyl diphenylphosphine oxide **3ad**

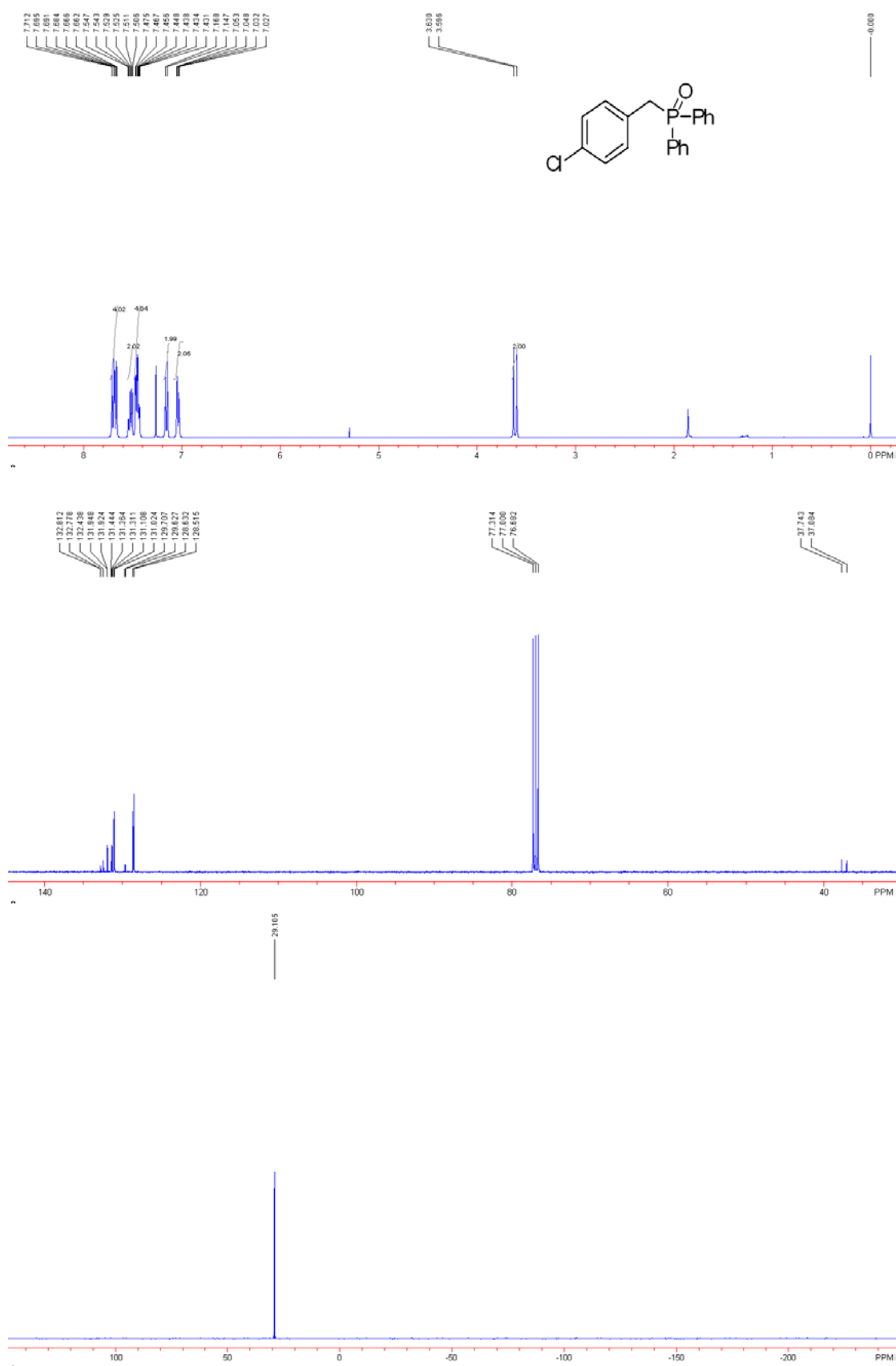
Light yellow solid, Mp 212.3-212.5 °C ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.62 (d, $J = 13.6$ Hz, 2H), 6.87 (t, $J = 8.4$ Hz, 2H), 7.05-7.09 (m, 2H), 7.42-7.47 (m, 4H), 7.50-7.54 (m, 2H), 7.66-7.7 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 36.8, 37.4, 115.10, 115.13, 115.31, 115.35, 126.70, 126.74, 126.78, 126.81, 128.4, 128.6, 131.0, 131.1, 131.4, 131.48, 131.50, 131.51, 131.6, 131.84, 131.86, 132.5, 160.6, 160.61, 163.01, 163.05. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 29.31, 29.33. ^{19}F NMR (470 MHz, CDCl_3 , TMS) δ -115.88, -115.87. MS (ESI) m/z (%): 311.1 (M + H, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{19}\text{H}_{17}\text{OFP}$: 311.1001, Found: 311.1005.





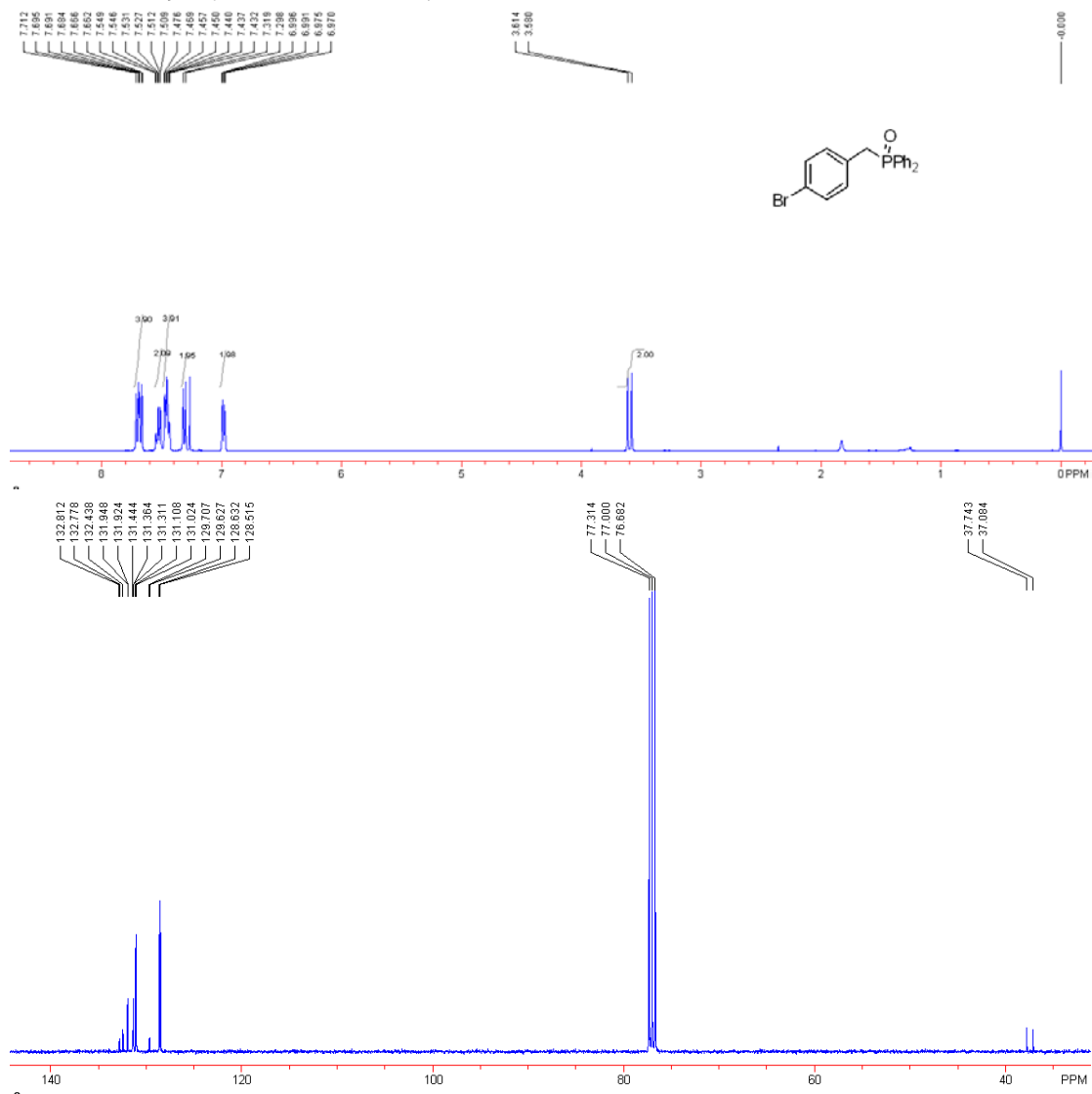
4-Chlorobenzyl diphenylphosphine oxide **3ae** ^[3]

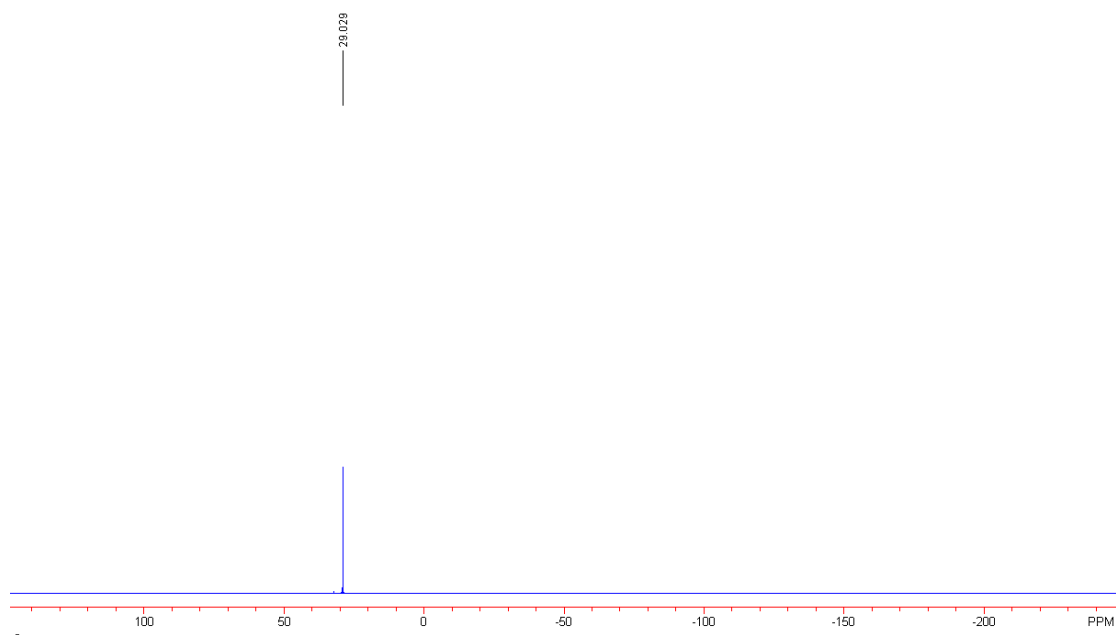
White solid, Mp 240.3-241.0 °C, ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.61 (d, *J* = 13.6 Hz, 2H), 7.04 (dd, *J* = 2.0, 8.4 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.43-7.48 (m, 4H), 7.51-7.55 (m, 2H), 7.67-7.71 (m, 4H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 37.1, 37.7, 128.5, 128.6, 129.6, 129.7, 131.0, 131.1, 131.31, 131.36, 131.44, 131.92, 131.95, 132.4, 132.77, 132.81. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 29.1. MS (ESI) *m/z* (%): 327.1 (M + H, 100); HRMS (Micromass LCT) Calcd for C₁₉H₁₇ClOP: 327.0706, Found: 327.0706.



4-Bromobenzyl diphenylphosphine oxide **3af** ^[1]

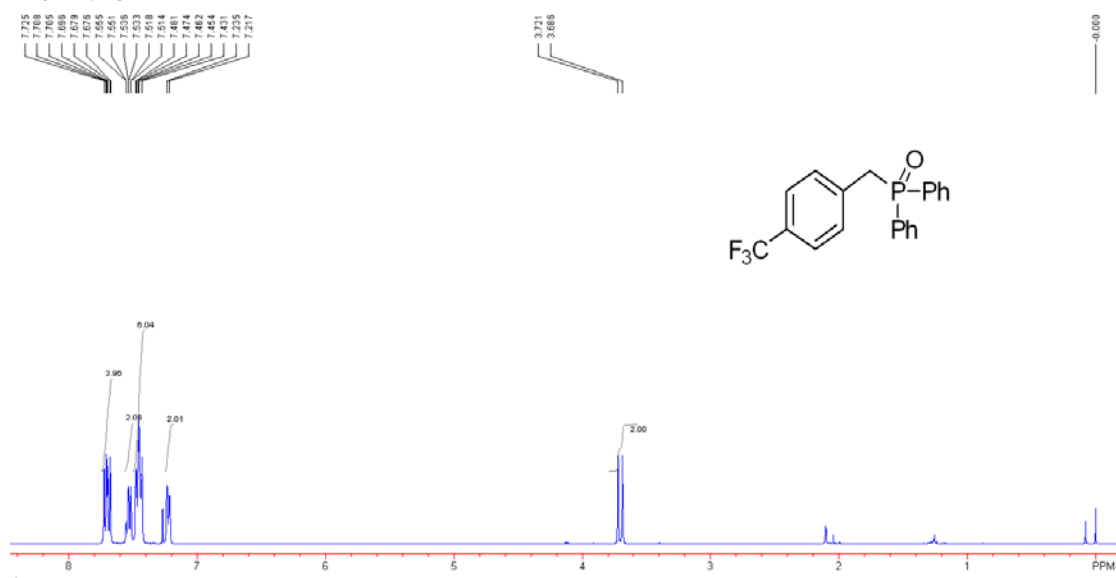
Light yellow solid, Mp 239.8-240.5 °C, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.60 (d, $J = 13.6$ Hz, 2H), 6.98 (dd, $J = 2.0, 8.4$ Hz, 2H), 7.31 (d, $J = 8.4$ Hz, 2H), 7.43-7.48 (m, 4H), 7.51-7.55 (m, 2H), 7.66-7.71 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 37.1, 37.7, 128.5, 128.6, 129.6, 129.7, 131.0, 131.1, 131.31, 131.36, 131.44, 131.92, 131.95, 132.4, 132.78, 132.81. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 29.0. MS (ESI) m/z (%): 371.0 (M + H, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{19}\text{H}_{17}\text{BrOP}$: 371.0200, Found: 371.0204.

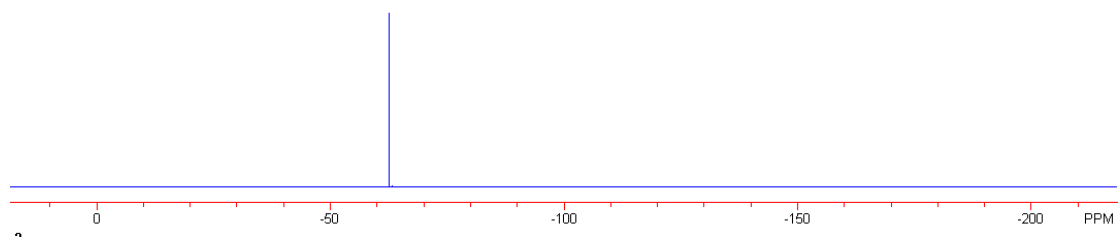
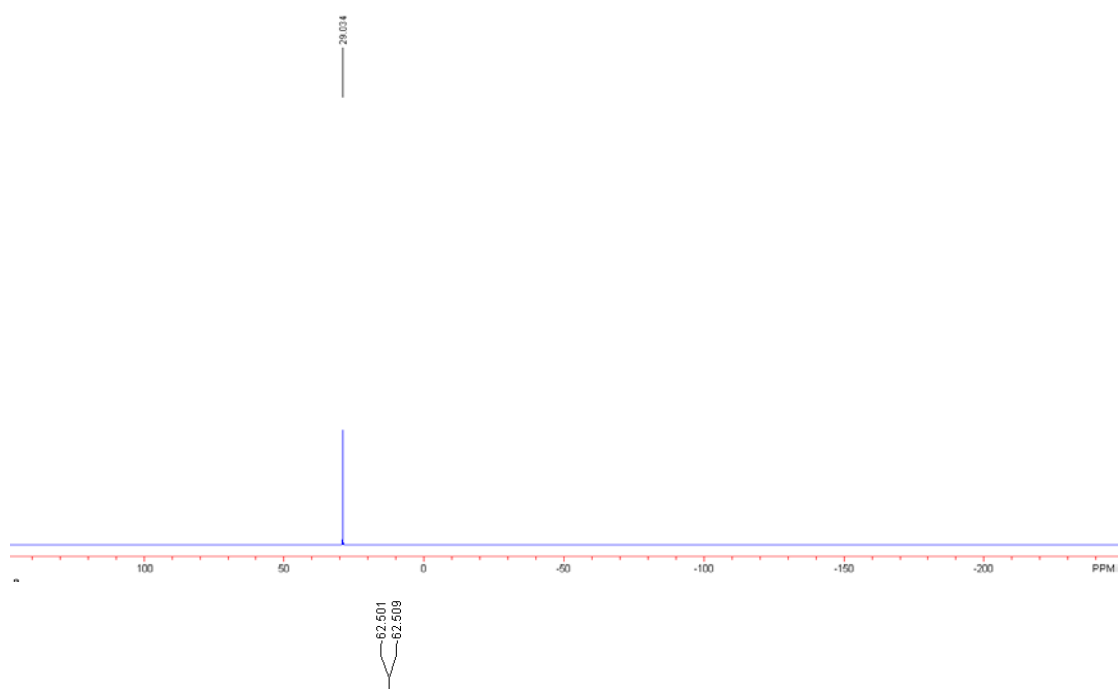
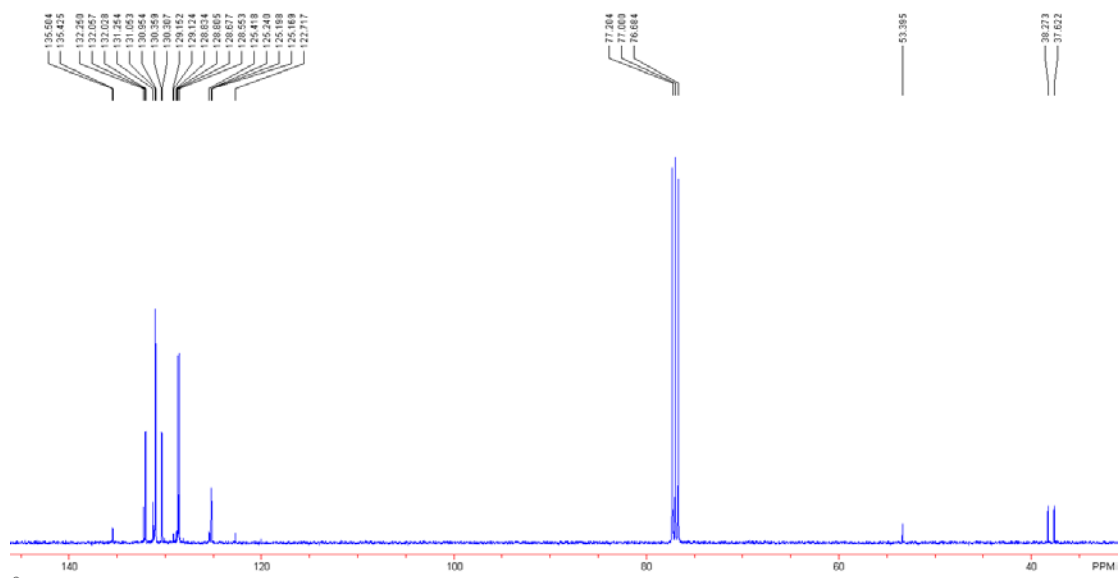




4-(Trifluoromethyl)benzyl diphenylphosphine oxide **3ag**

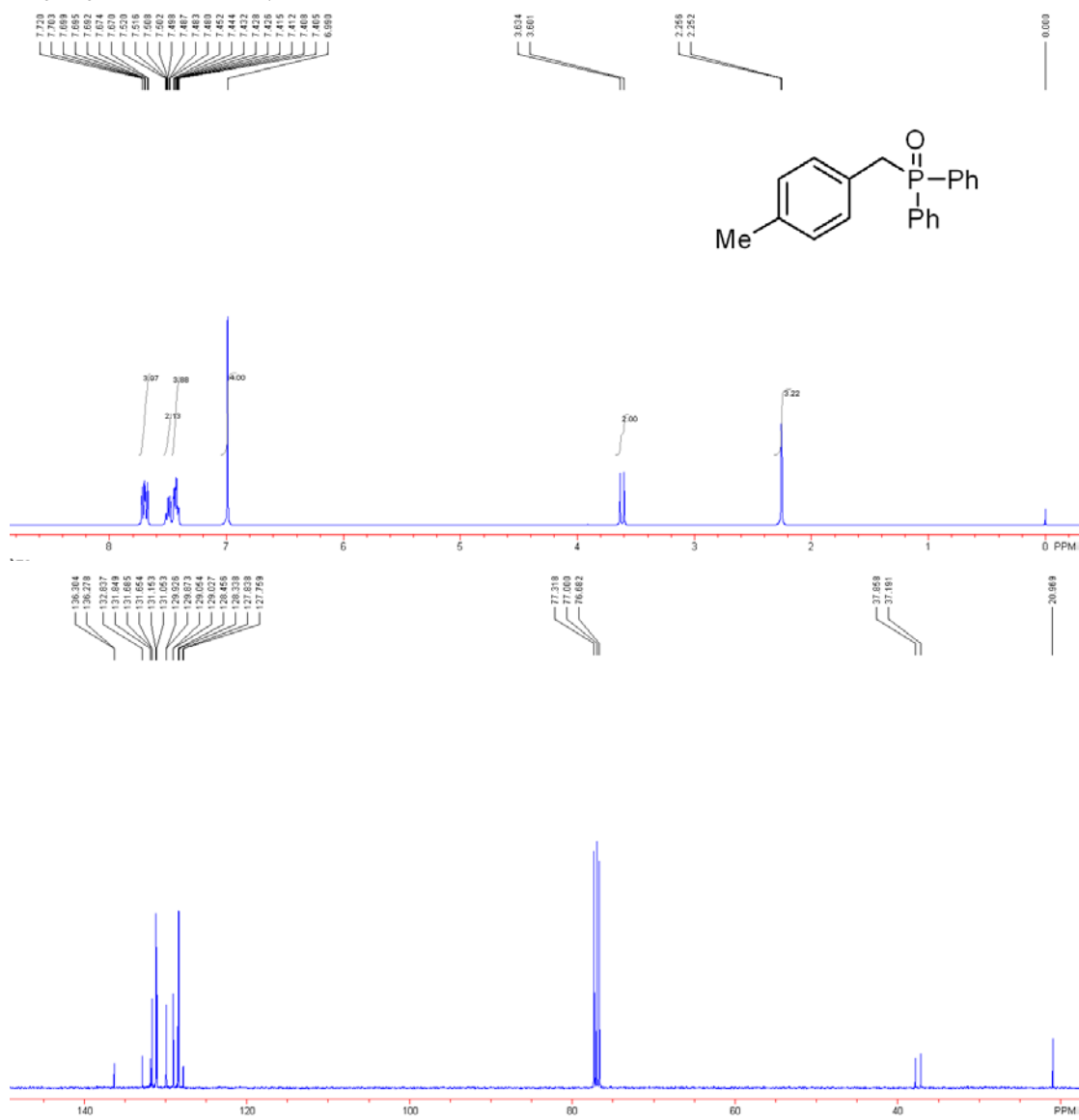
White solid, Mp 212.5-213.0 °C. $^1\text{H NMR}$ (400 MHz, CDCl_3 , TMS) δ 3.70 (d, $J = 14.0$ Hz, 2H), 7.23 (d, $J = 7.2$ Hz, 2H), 7.43-7.48 (m, 6H), 7.51-7.56 (m, 2H), 7.68-7.73 (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3 , TMS) δ 37.6, 38.3, 53.4, 122.7, 125.17, 125.20, 125.24, 125.4, 128.6, 128.7, 128.81, 128.83, 129.12, 129.15, 130.3, 130.4, 131.0, 131.1, 131.3, 132.03, 132.06, 132.3, 135.4, 135.5. $^{31}\text{P NMR}$ (160 MHz, CDCl_3 , TMS) δ 29.0. $^{19}\text{F NMR}$ (470 MHz, CDCl_3 , TMS) δ -62.509, -62.501. MS (ESI) m/z (%): 361.1 (M + H, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{19}\text{H}_{17}\text{F}_3\text{OP}$: 361.0969, Found: 361.0967

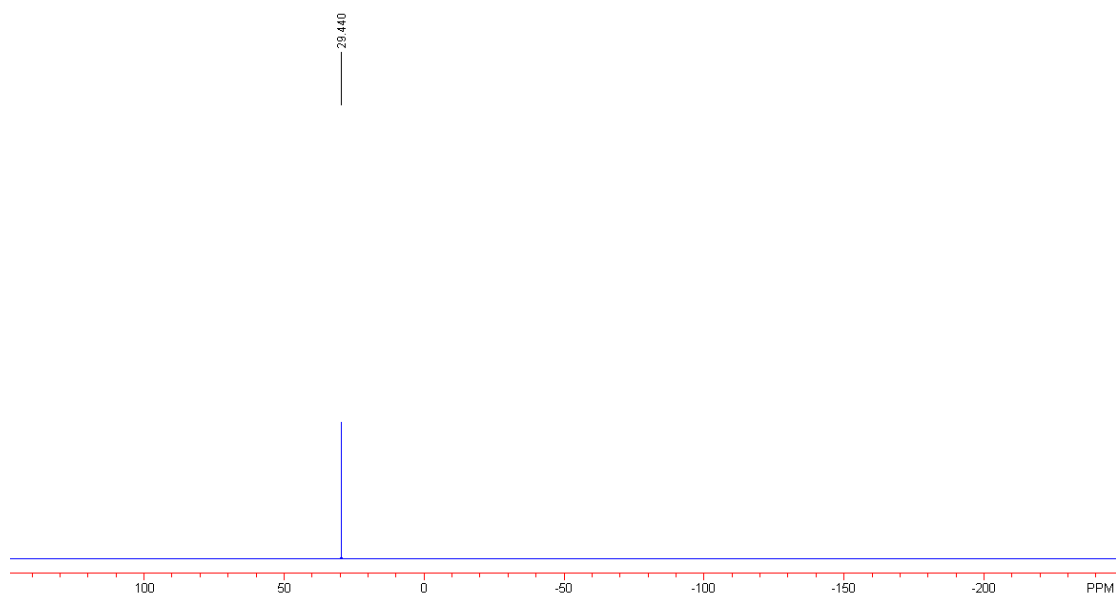




4-Methylbenzyl diphenylphosphine oxide **3ah** ^[3]

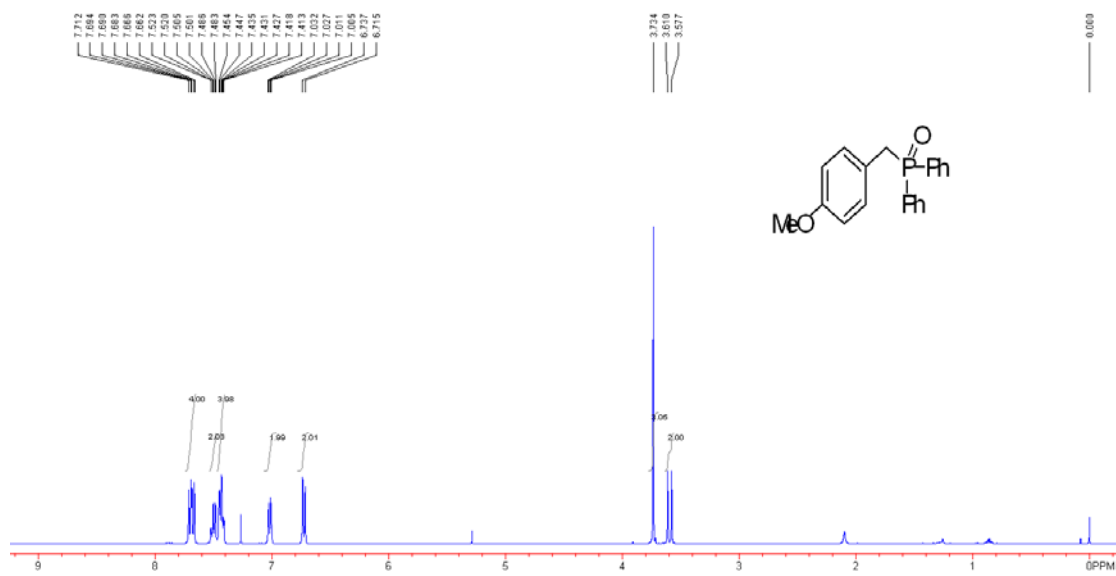
White solid, Mp 156.3-156.5 °C, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.25 (d, $J = 1.6$ Hz, 3H), 3.62 (d, $J = 13.2$ Hz, 2H), 6.99 (s, 4H), 7.41-7.45 (, 4H), 7.48-7.52 (m, 2H), 7.67-7.72 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 21.0, 37.2, 37.9, 127.76, 127.83, 128.3, 128.5, 129.0, 129.1, 129.87, 129.93, 131.1, 131.2, 131.65, 131.69, 131.8, 132.8, 136.28, 136.30. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 29.4. MS (ESI) m/z (%): 307.1 (M + H, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{20}\text{H}_{20}\text{OP}$: 307.1252, Found: 307.1256.

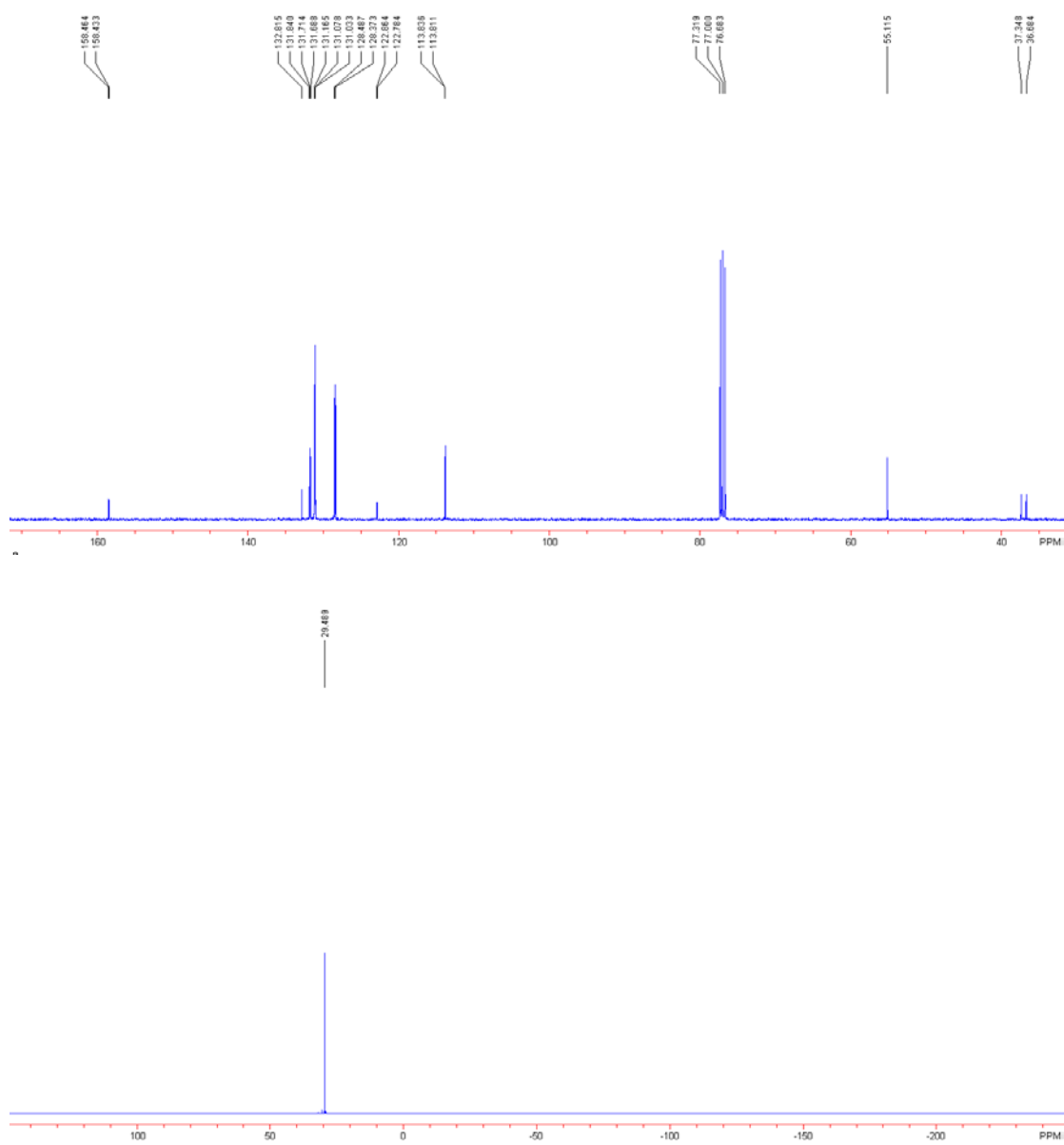




4-Methoxybenzyl diphenylphosphine oxide **3ai** ^[3]

White solid, Mp 222.4-223.2 °C, ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.59 (d, *J* = 13.2 Hz, 2H), 3.73 (s, 3H), 6.73 (d, *J* = 8.8 Hz, 2H), 7.02 (dd, *J* = 2.0, 8.4 Hz, 2H), 7.41-7.45 (m, 4H), 7.48-7.52 (m, 2H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 36.7, 37.3, 55.1, 113.81, 113.84, 122.8, 122.9, 128.4, 128.5, 131.0, 131.1, 131.2, 131.68, 131.71, 131.8, 132.8, 158.4, 158.5. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 29.5. MS (ESI) *m/z* (%): 323.1 (M + H, 100); HRMS (Micromass LCT) Calcd for C₂₀H₂₀O₂P: 323.1201, Found: 323.1198.

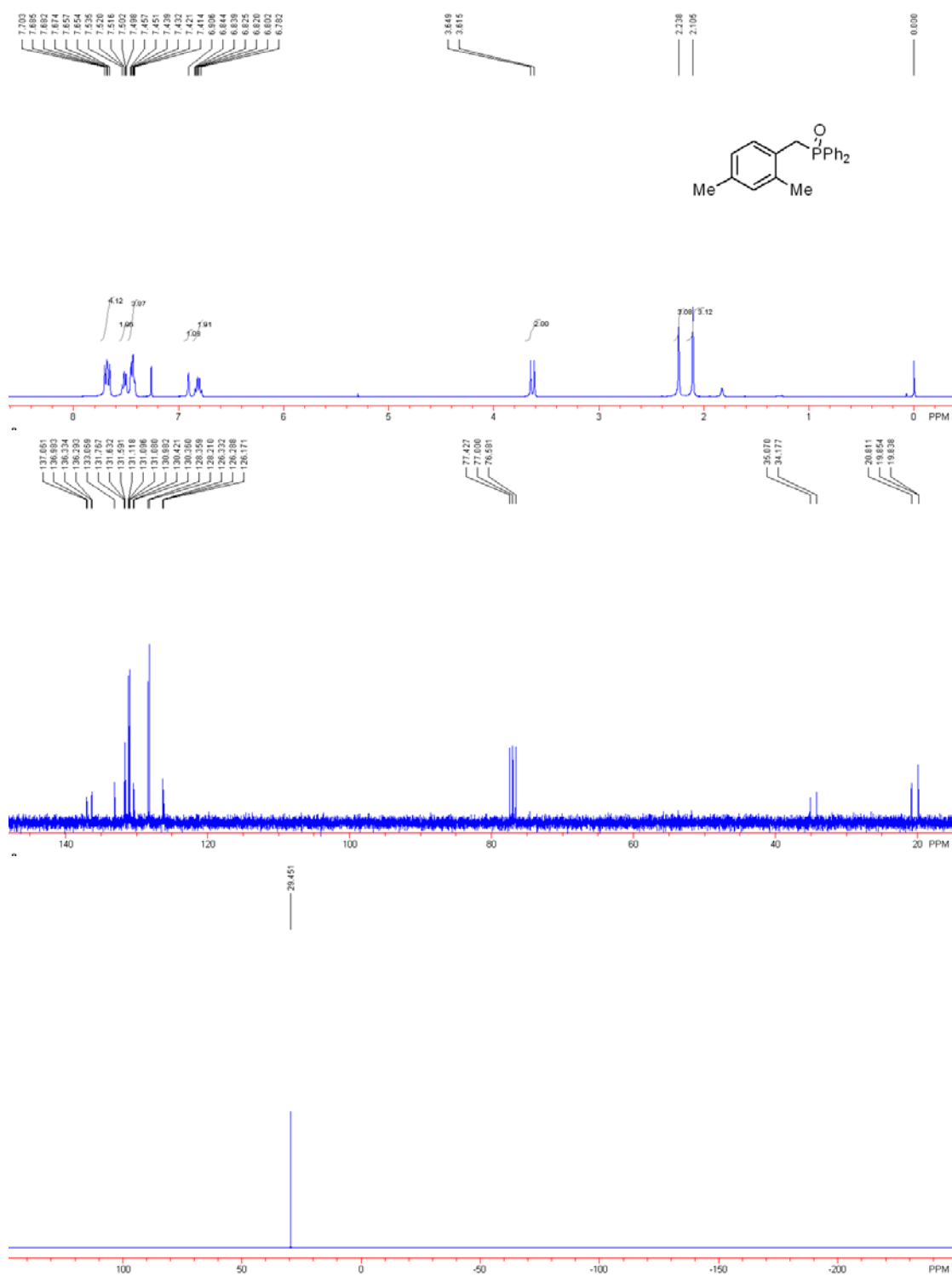




2,4-Dimethylbenzyl diphenylphosphine oxide **3aj**

White solid, Mp 156.3-156.5, ¹H NMR (400 MHz, CDCl₃, TMS)

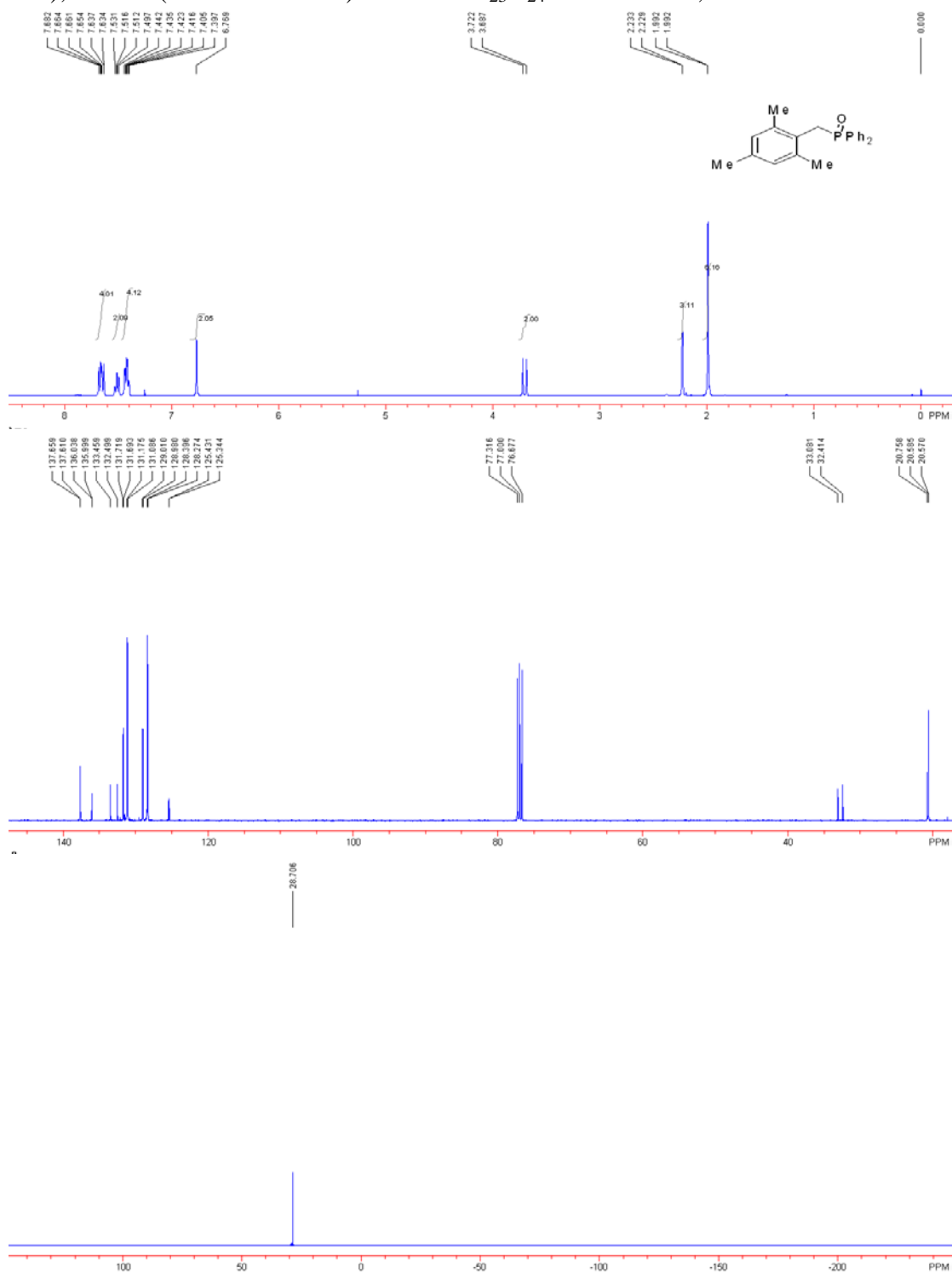
δ 2.11 (s, 3H), 2.24 (s, 3H), 3.63 (d, *J* = 13.6 Hz, 2H), 6.78-6.84 (m, 2H), 6.91 (s, 1H), 7.41-7.46 (m, 4H), 7.50-7.54 (m, 2H), 7.65-7.70 (m, 4H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 19.84, 19.85, 20.8, 34.2, 35.1, 126.2, 126.29, 126.33, 128.2, 128.4, 130.4, 130.98, 131.08, 131.10, 131.12, 131.59, 131.63, 131.8, 133.1, 136.29, 136.33, 137.0, 137.1. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 29.5. MS (ESI) *m/z* (%): 321.1 (M + H, 100); HRMS (Micromass LCT) Calcd for C₂₁H₂₂OP: 321.1408, Found: 321.1403.



2,4,6-Trimethylbenzyl diphenylphosphine oxide **3ak**

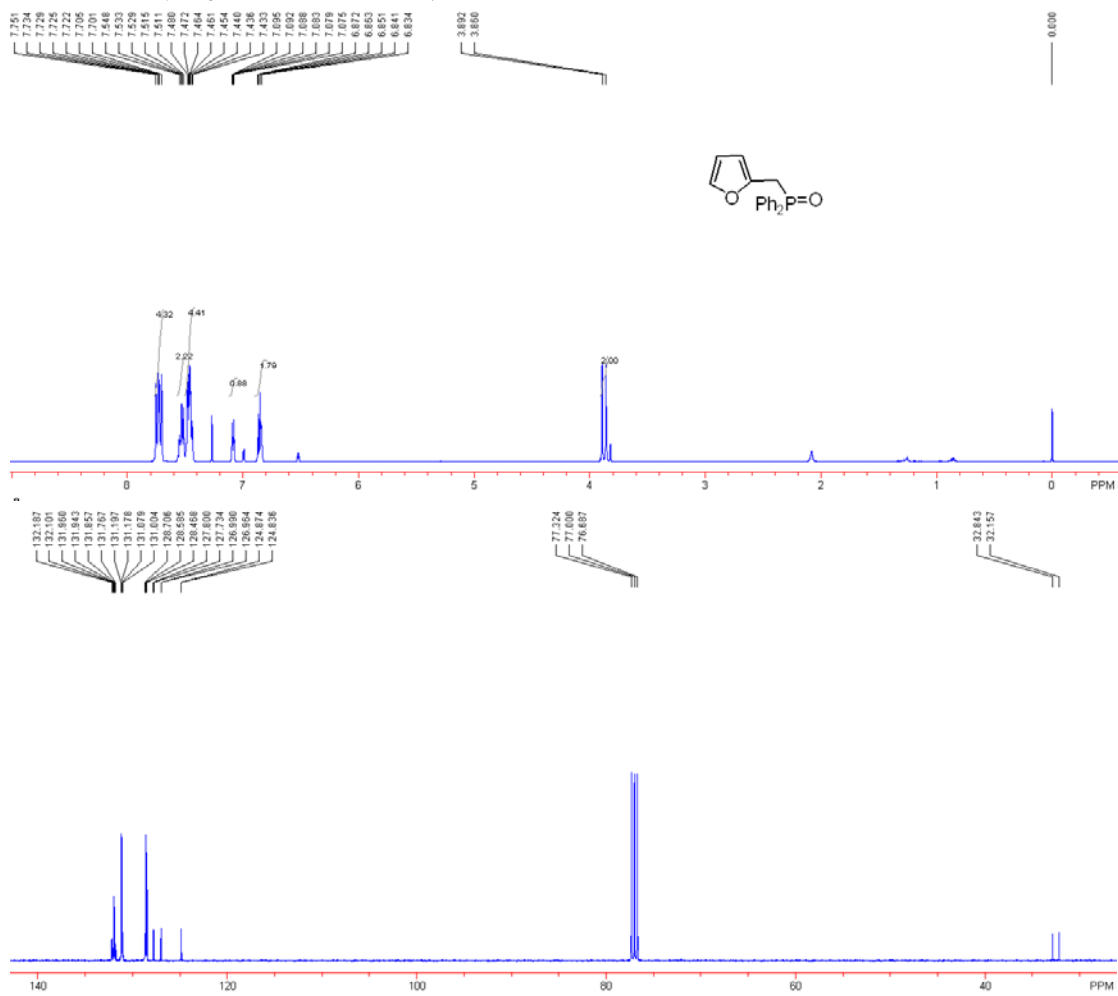
White solid, Mp 180.3-181.5 °C, ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.99 (s, 6H), 2.23 (d, *J* = 1.6 Hz, 3H), 3.70 (d, *J* = 14.0 Hz, 2H), 6.77 (s, 2H), 7.40-7.44 (m, 4H), 7.51 (dd, *J* = 6.0, 7.6 Hz, 2H), 7.64-7.68 (m, 4H). ¹³C NMR (100

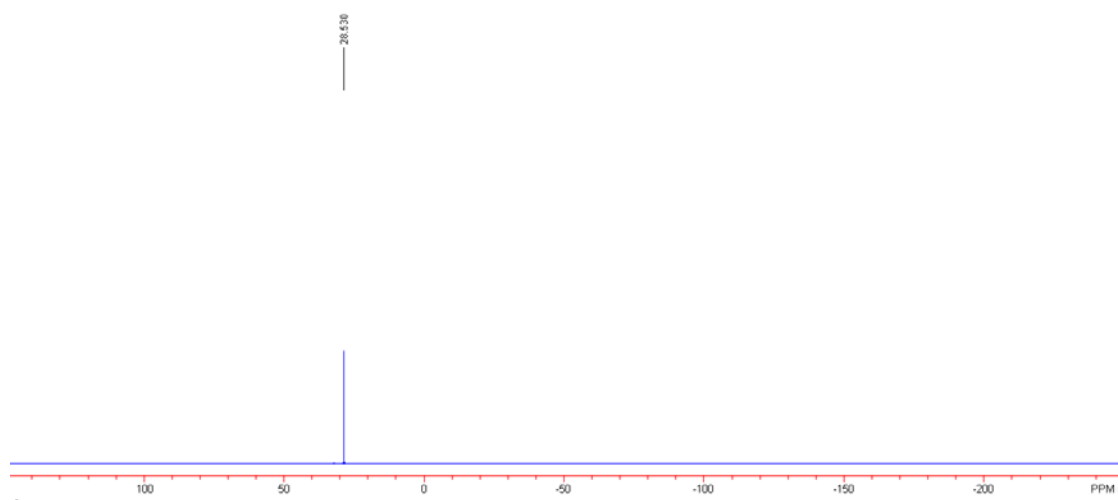
MHz, CDCl₃, TMS) δ 20.57, 20.59, 20.8, 32.4, 33.1, 125.3, 125.4, 128.3, 128.4, 128.98, 129.01, 131.1, 131.2, 131.69, 131.72, 132.5, 133.5, 136.00, 136.04, 137.6, 137.7. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 28.7. MS (ESI) *m/z* (%): 335.2 (M + H, 100); HRMS (Micromass LCT) Calcd for C₂₃H₂₄OP: 335.1565, Found: 335.1569.



(Furan-2-ylmethyl)diphenylphosphine oxide **3ai** ^[4]

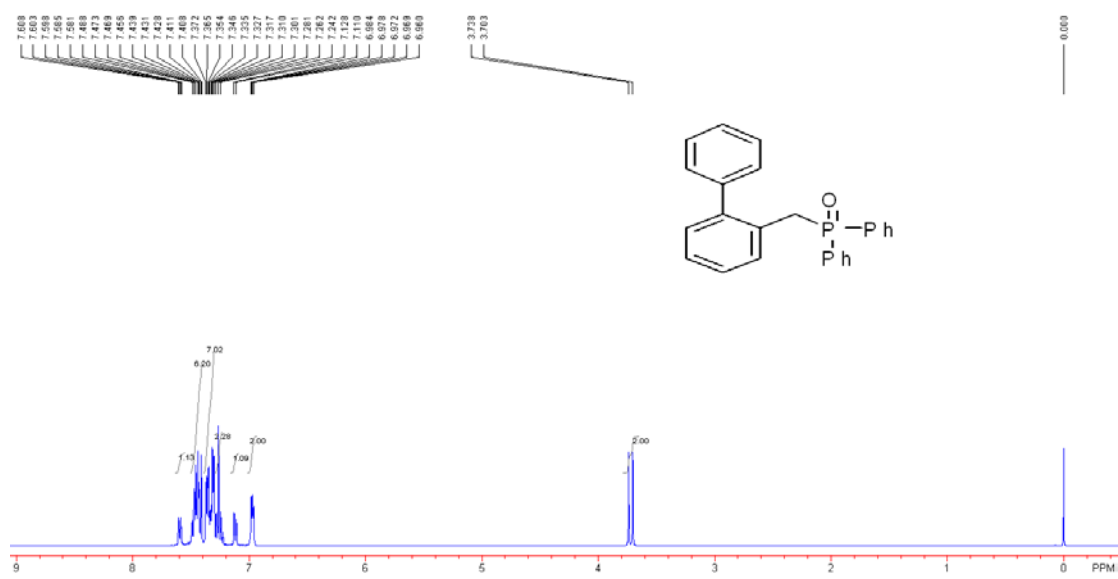
White solid, Mp 145.0-146.1 °C ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.88 (d, $J = 12.8$ Hz, 2H), 6.83-6.86 (m, 2H), 7.08-7.10 (m, 1H), 7.43-7.48 (m, 4H), 7.51-7.55 (m, 2H), 7.70-7.75 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 32.2, 32.8, 124.84, 124.87, 127.0, 127.7, 127.8, 128.5, 128.6, 128.7, 131.00, 131.08, 131.18, 131.20, 131.8, 131.90, 131.94, 131.96, 132.10, 132.19. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 28.5. MS (ESI) m/z (%): 335.2 (M + K, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{17}\text{H}_{15}\text{O}_2\text{PK}$: 321.0447, Found: 321.0443.

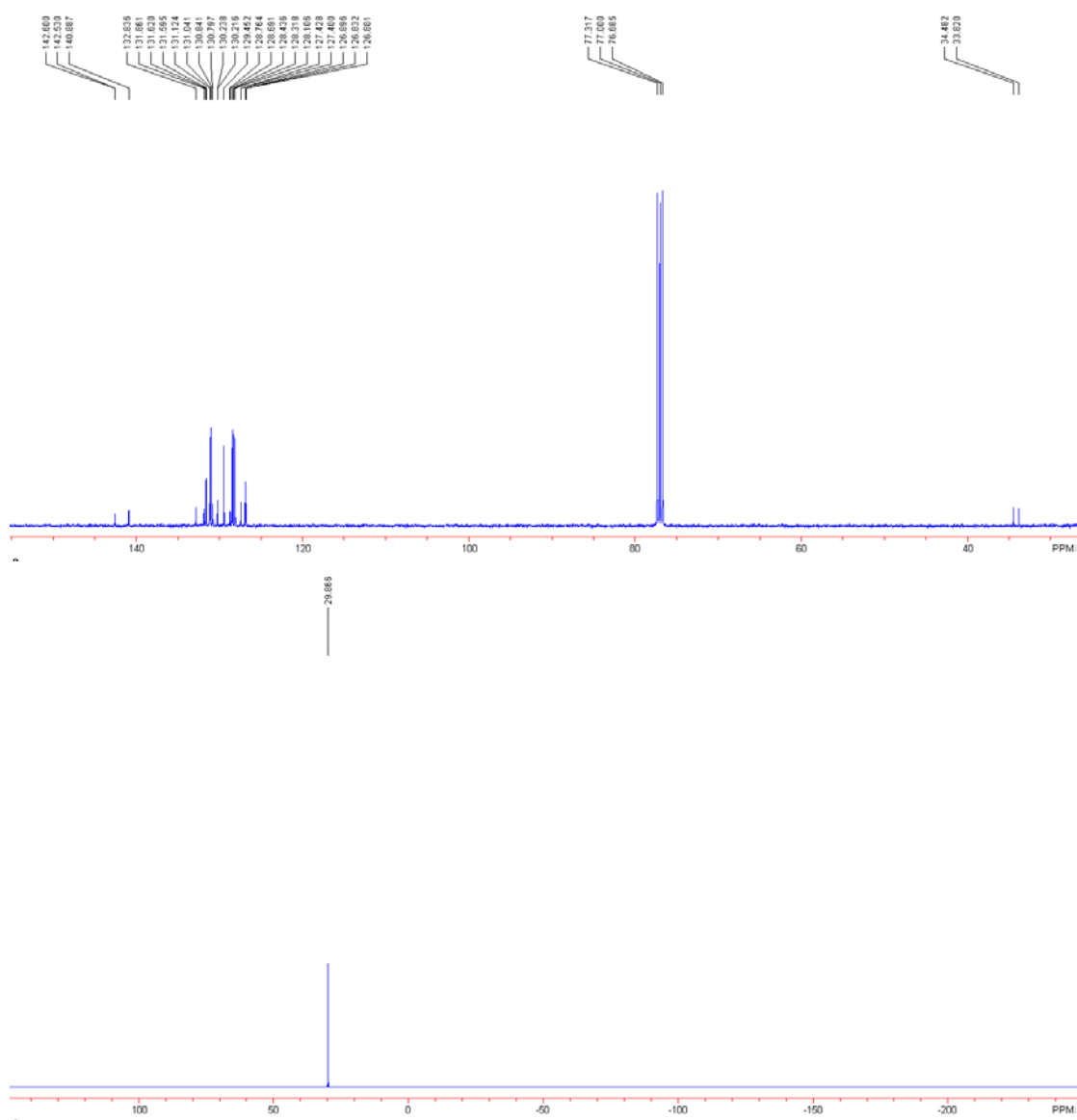




(Biphenyl-2-ylmethyl)diphenylphosphine oxide **3am**

White sticky solid, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.72 (d, $J = 14.0$ Hz, 2H), 6.96-6.98 (m, 2H), 7.12 (d, $J = 7.2$ Hz, 1H), 7.26-7.37 (m, 9H), 7.41-7.49 (m, 6H), 7.58-7.61 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 33.8, 34.5, 126.80, 126.83, 126.9, 127.40, 127.43, 128.1, 128.3, 128.4, 128.7, 128.8, 129.5, 130.22, 130.23, 130.80, 130.84, 131.0, 131.1, 131.60, 131.62, 131.9, 132.8, 140.9, 142.5, 142.6. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 29.9. MS (ESI) m/z (%): 391.1 (M + Na, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{23}\text{H}_{24}\text{OPNa}$: 391.1228, Found: 391.1227.

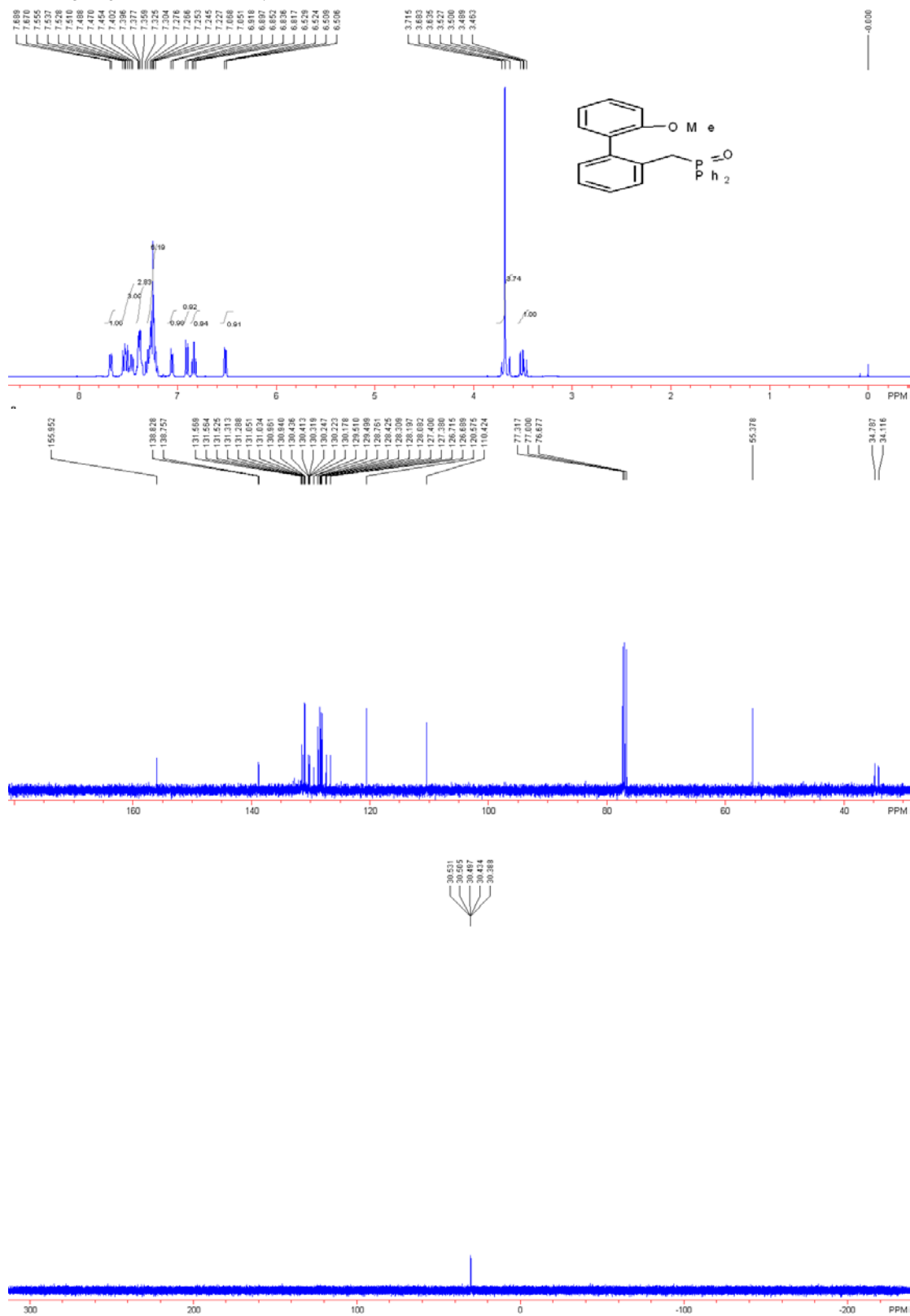




((2'-Methoxybiphenyl-2-yl)methyl)diphenylphosphine oxide 3an

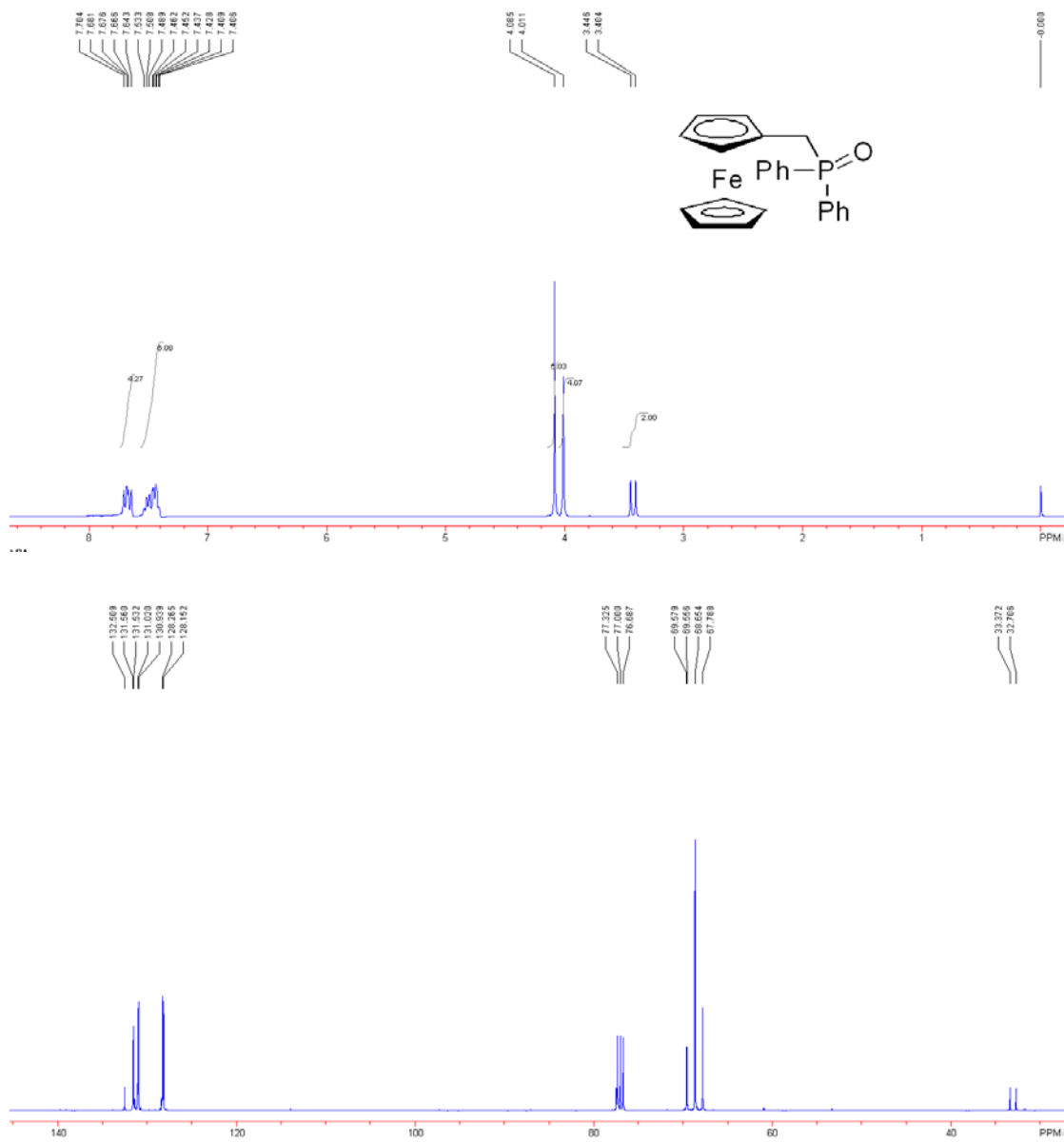
White sticky solid, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.49 (dd, $J = 10.4, 14.8$ Hz, 1H), 3.64-3.72 (m, 4H), 6.51 (dd, $J = 1.2, 7.2$ Hz, 1H), 6.84 (t, $J = 7.6$ Hz, 1H), 6.91 (d, $J = 8.4$ Hz, 1H), 7.06 (d, $J = 6.8$ Hz, 1H), 7.23-7.28 (m, 6H), 7.36-7.40 (m, 3H), 7.45-7.56 (m, 3H), 7.68 (d, $J = 7.6$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 34.1, 34.8, 55.4, 110.4, 120.6, 126.70, 126.72, 127.38, 127.40, 128.1, 128.2, 128.3, 128.4, 128.8, 129.50, 129.51, 130.18, 130.22, 130.25, 130.3, 130.41, 130.44, 130.94, 130.96, 131.03, 131.05, 131.29, 131.31, 131.53, 131.56, 131.57, 138.76, 138.83, 156.0. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 30.40, 30.43, 30.50, 30.51, 30.53. MS (ESI) m/z (%): 335.2 (M + H, 100); HRMS (Micromass LCT) Calcd

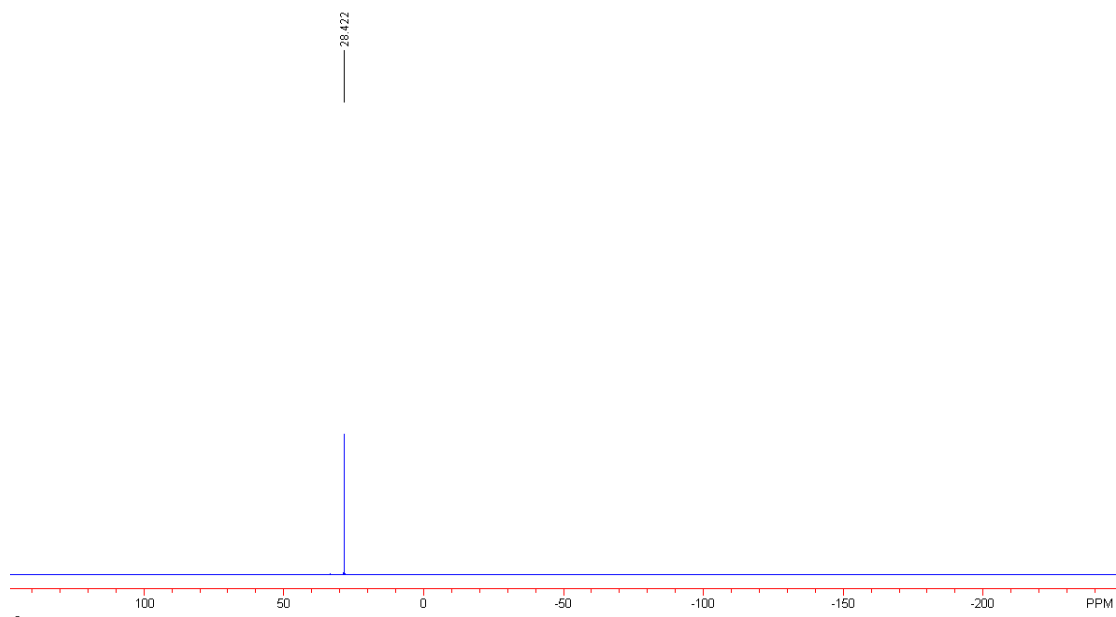
for $C_{23}H_{25}OP$: 399.1514, Found: 399.1517.



(Ferrocenylmethyl)diphenylphosphine oxide **3ao**

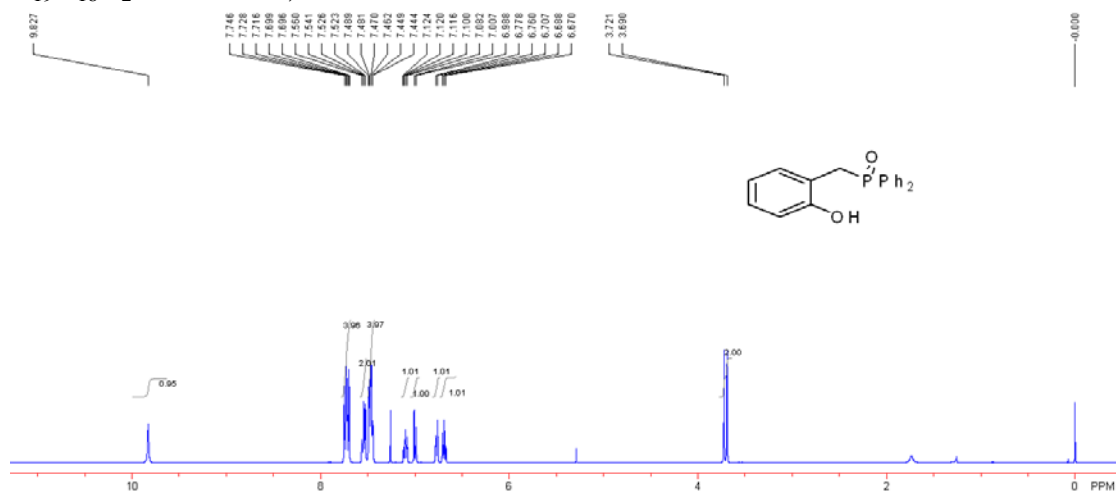
Yellow solid, Mp 201.5-202.1 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 3.43 (d, $J = 16.8$ Hz, 2H), 4.01 (s, 4H), 4.09 (s, 5H), 7.41-7.53 (m, 6H), 7.64-7.70 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 32.7, 33.4, 67.8, 68.7, 69.56, 69.58, 128.2, 128.3, 130.9, 131.0, 131.5, 131.6, 132.5. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 28.4. MS (ESI) m/z (%): 401.1 (M + H, 100); HRMS (Micromass LCT) Calcd for $\text{C}_{23}\text{H}_{24}\text{OPFe}$: 401.0758, Found: 401.0893.

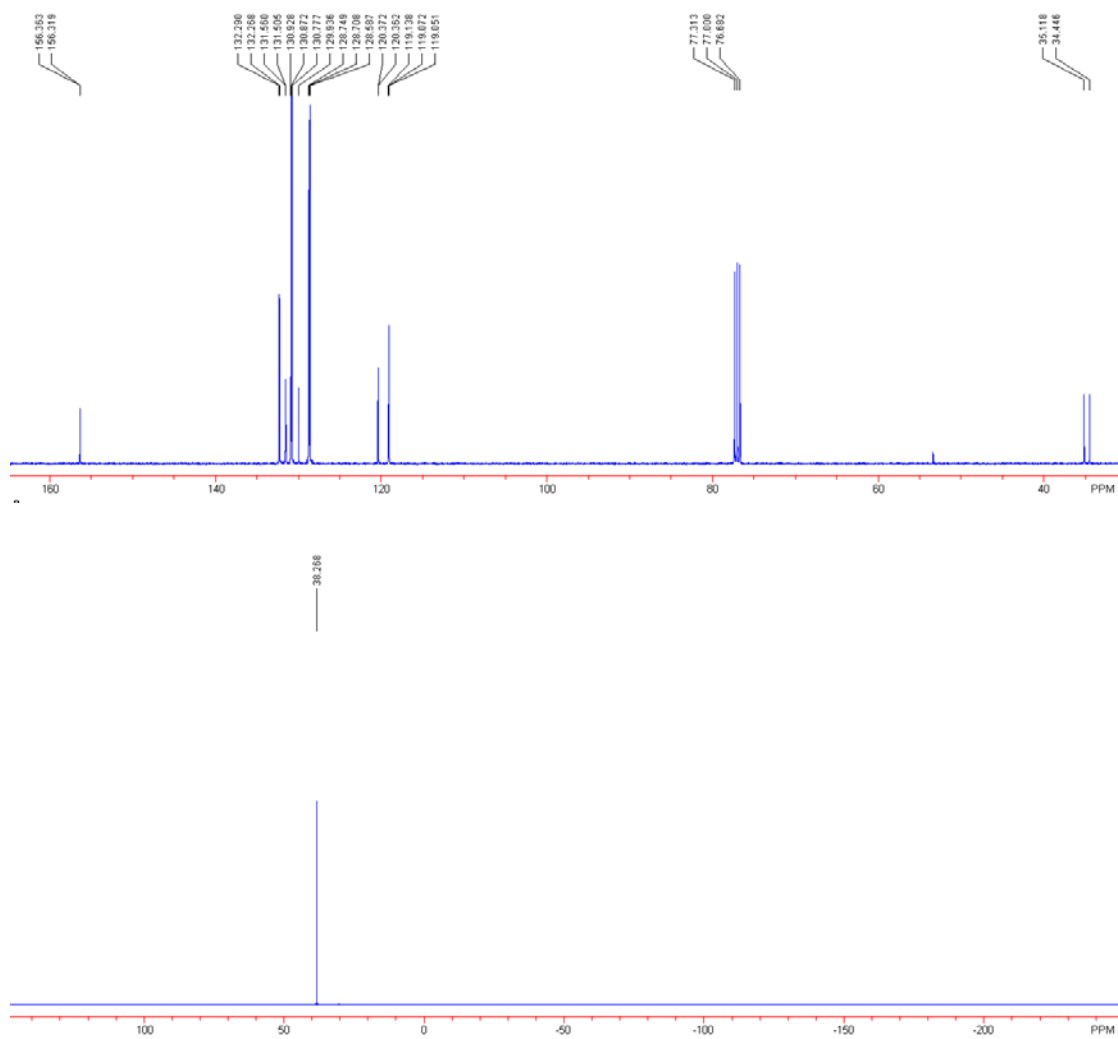




2-((Diphenylphosphino)methyl)phenol oxide **3ap** [5]

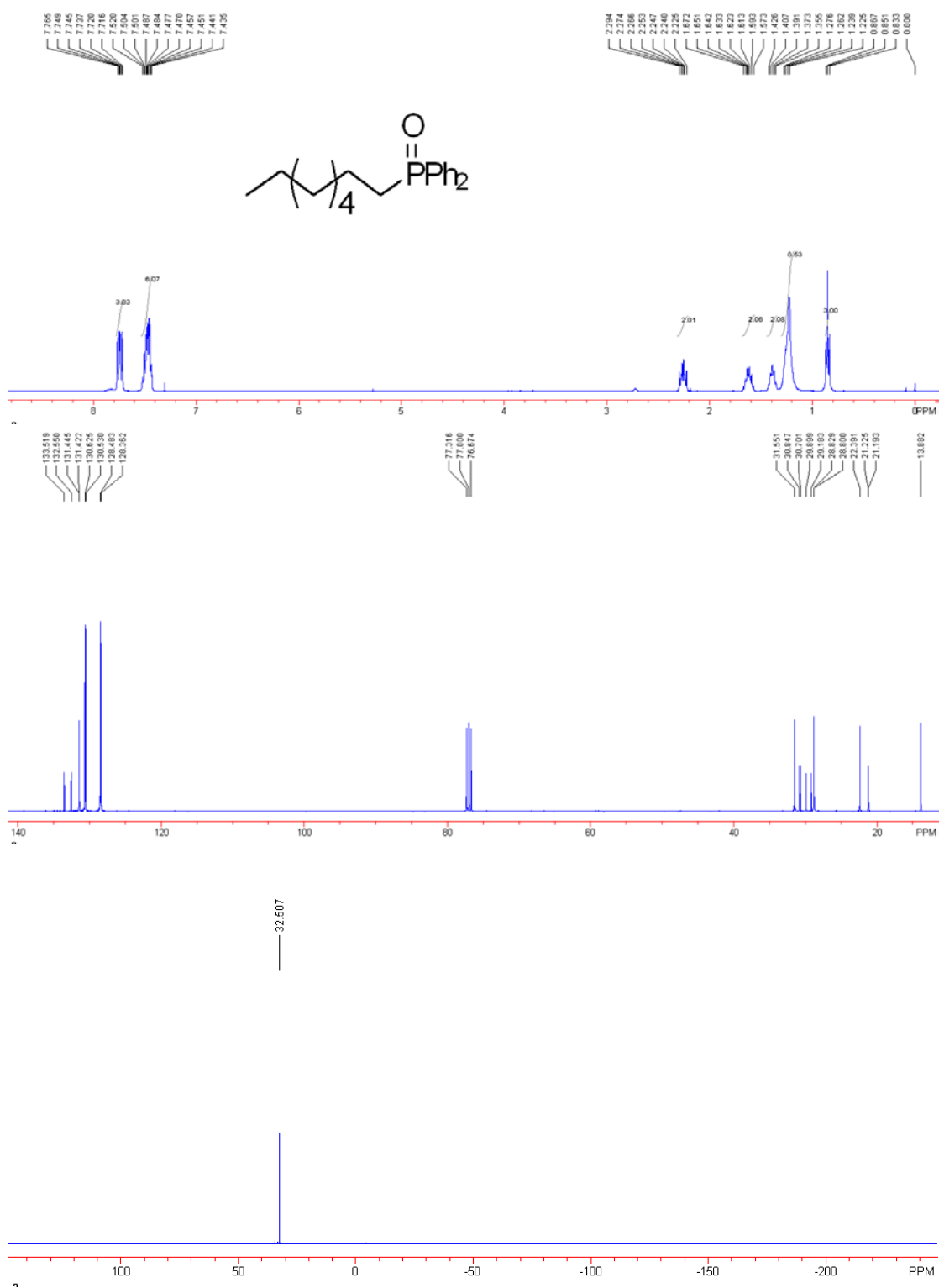
White solid, Mp 176.8-177.2 °C, ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.71 (d, *J* = 12.4 Hz, 2H), 6.69 (t, *J* = 7.2 Hz, 1H), 6.77 (t, *J* = 7.2 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 7.08-7.12 (m, 1H), 7.44-7.49 (m, 4H), 7.52-7.56 (m, 2H), 7.56-7.75 (m, 4H), 9.83 (s, 1H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 34.4, 35.1, 119.05, 119.07, 119.1, 120.36, 120.37, 128.6, 128.71, 128.75, 129.9, 130.8, 130.9, 131.51, 131.56, 132.27, 132.29, 156.32, 156.36. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 38.3. MS (ESI) *m/z* (%): 309.1 (M + H, 100); HRMS (Micromass LCT) Calcd for C₁₉H₁₈O₂P: 309.1044, Found: 309.1042.





Octyldiphenylphosphine oxide **3aq** ^[6]

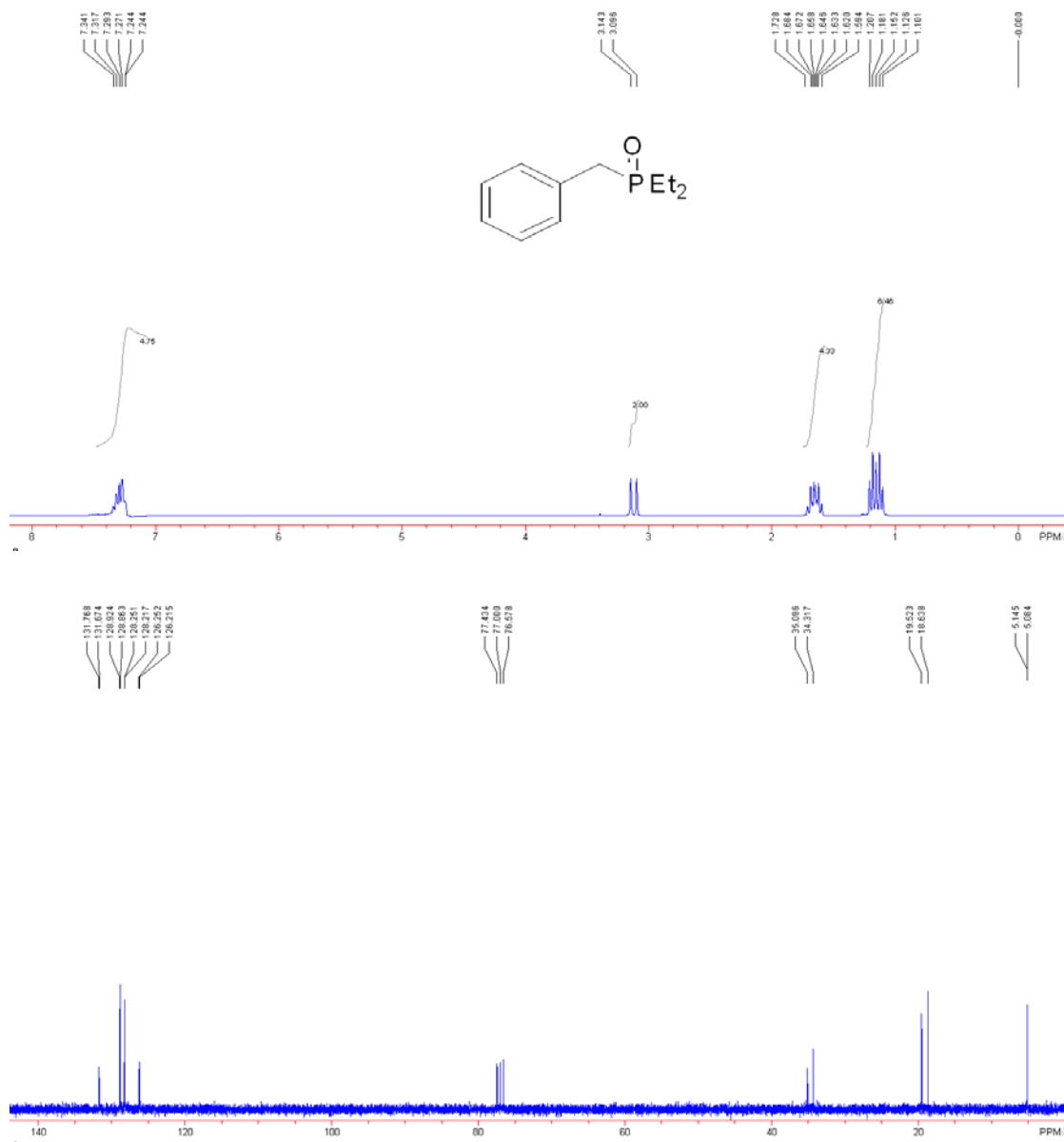
Colorless liquid, ¹H NMR (400 MHz, CDCl₃, TMS) δ 0.85 (t, *J* = 7.2 Hz, 3H), 1.23-1.28 (m, 8H), 1.36-1.43 (m, 2H), 1.57-1.67 (m, 2H), 2.23-2.29 (m, 2H), 7.44-7.52 (m, 6H), 7.72-7.77 (m, 4H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 13.9, 21.19, 21.23, 22.4, 28.80, 28.83, 29.2, 29.9, 30.7, 30.8, 31.6, 128.4, 128.5, 130.5, 130.6, 131.42, 131.45, 132.6, 133.5. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 32.5. MS (ESI) *m/z* (%): 315.2 (M + H, 100); HRMS (Micromass LCT) Calcd for C₂₀H₂₈OP: 315.1878, Found: 315.1884

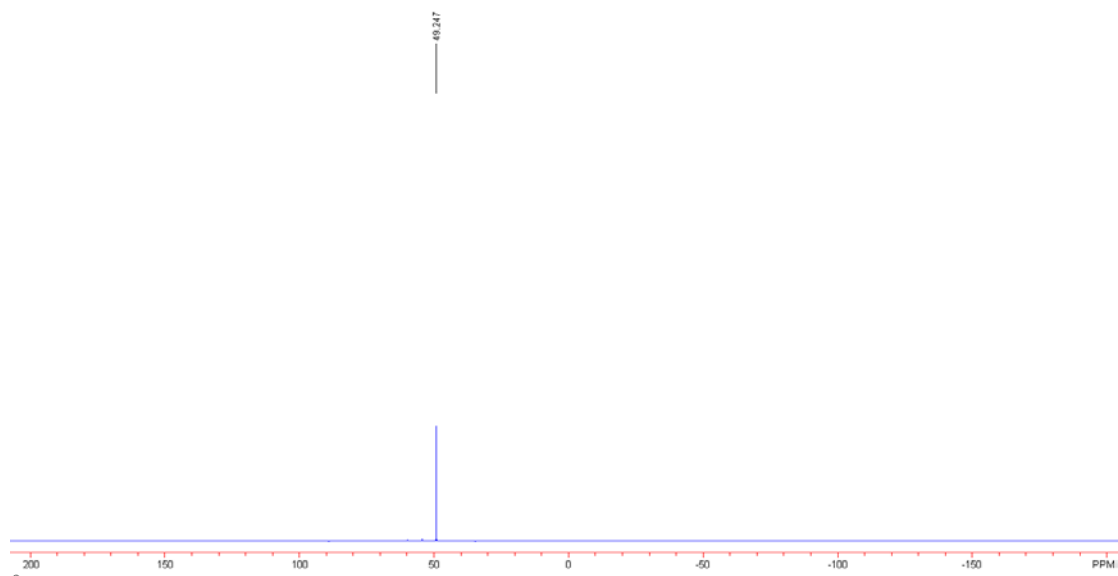


Benzyldiethylphosphine oxide **3ba** ^[5]

Colorless liquid, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.15 (dt, $J =$

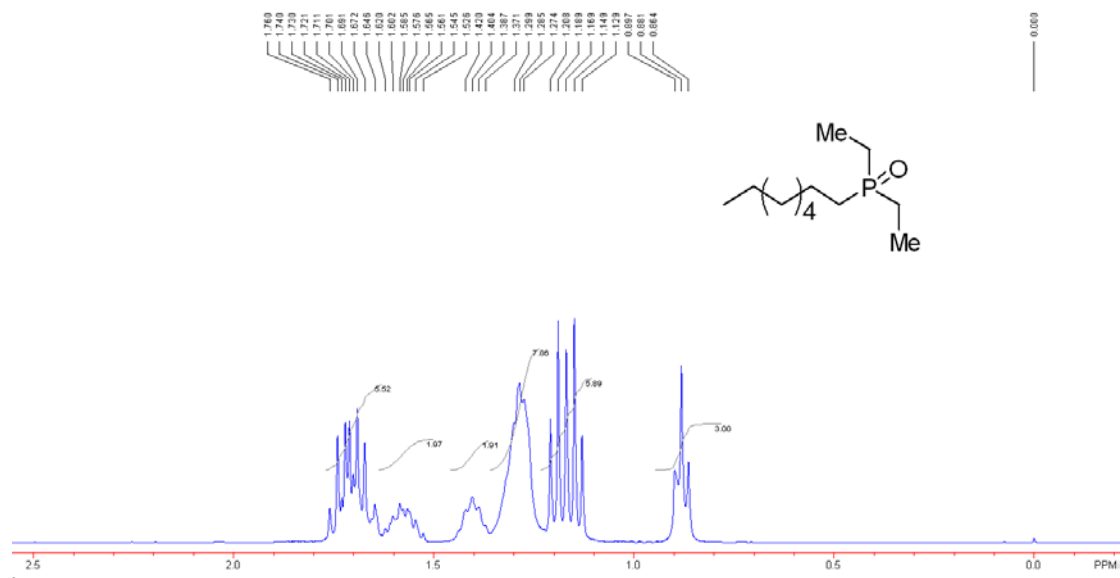
10.0, 22.0 Hz, 6H), 1.59-1.73 (m, 4H), 3.12 (d, $J = 14.8$ Hz, 2H), 7.24-7.34 (m, 5H).
 ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 5.08, 5.14, 18.6, 19.5, 34.3, 35.1, 126.2, 126.3, 128.2, 128.3, 128.86, 128.92, 131.7, 131.8. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 49.2.
MS (EI) m/z (%): 196.1 (M + H, 75), HRMS (Micromass LCT) Calcd for $\text{C}_{11}\text{H}_{17}\text{OP}$: 196.1017, Found: 196.1019.

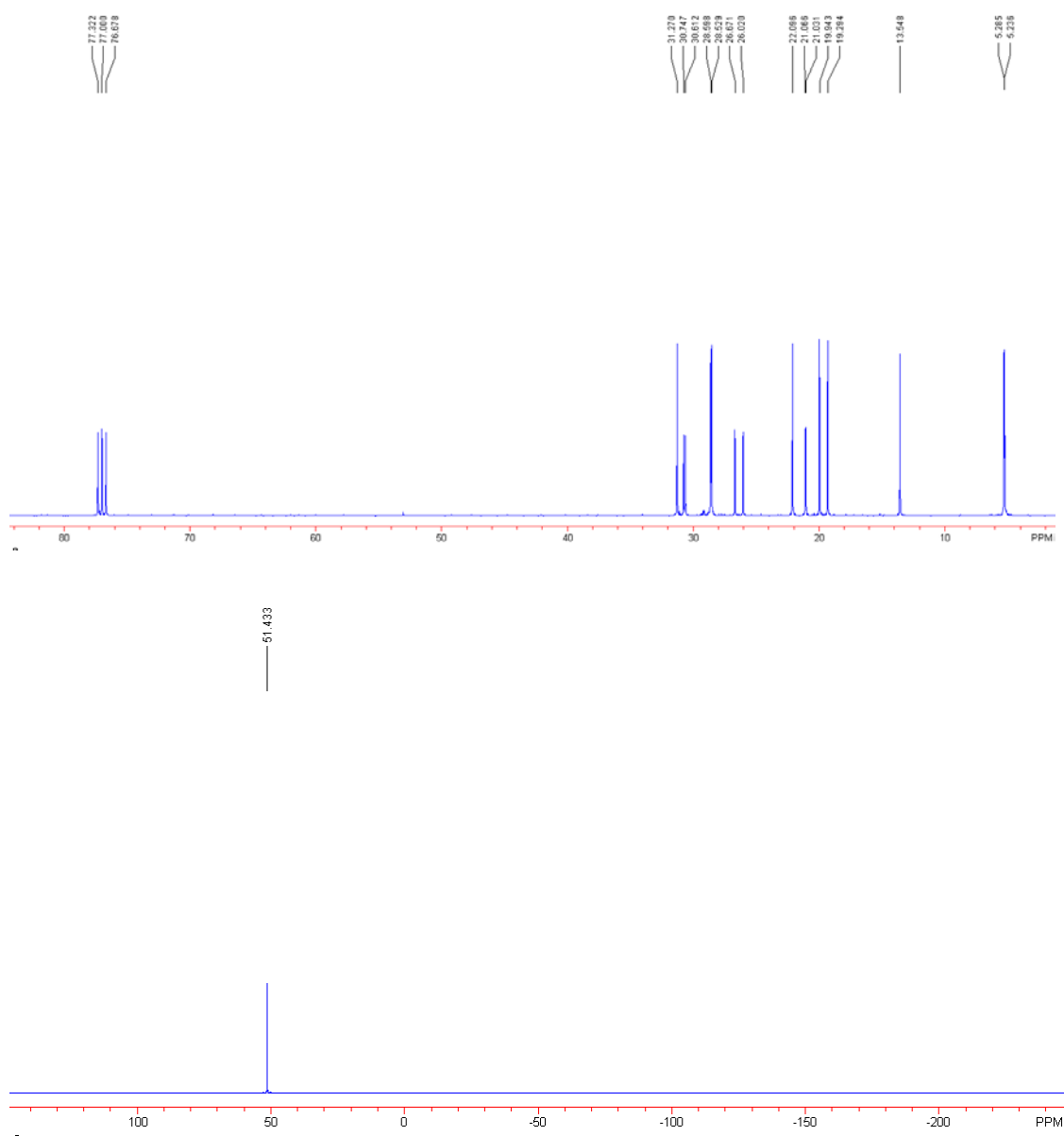




Diethyl(octyl)phosphine oxide **3bq**

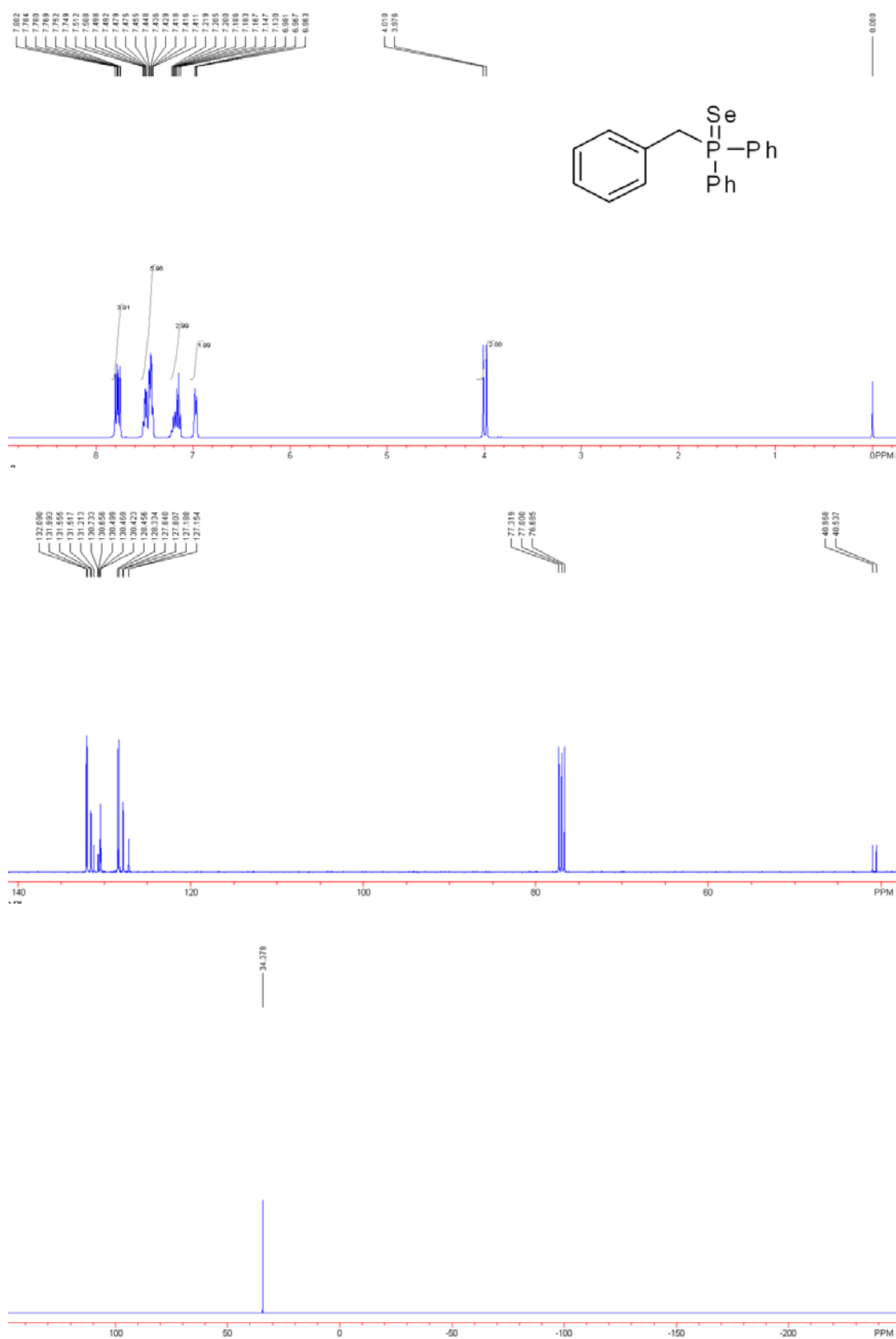
Colorless liquid, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 0.88 (t, $J = 6.8$ Hz, 3H), 1.17 (dt, $J = 7.6, 15.6$ Hz, 6H), 1.27-1.30 (m, 8H), 1.37-1.42 (m, 2H), 1.53-1.62 (m, 2H), 1.65-1.76 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 5.24, 5.29, 13.5, 19.3, 19.9, 21.0, 21.1, 26.0, 26.7, 28.5, 28.6, 30.6, 30.7, 31.3. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 51.4. MS (EI) m/z (%): 218.2 (M + H, 10), HRMS (Micromass LCT) Calcd for $\text{C}_{12}\text{H}_{27}\text{OP}$: 218.1800, Found: 218.1798.





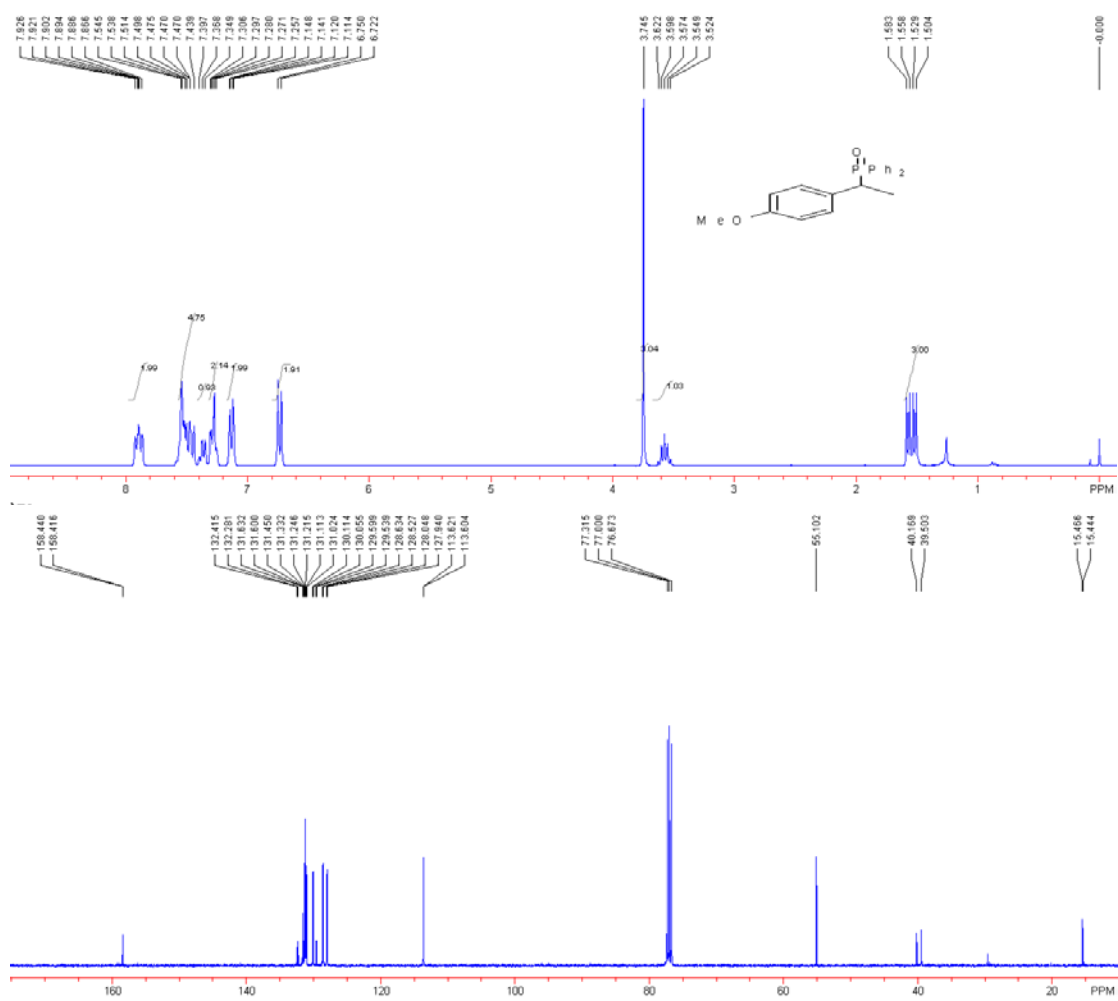
Benzyldiphenylphosphine selenide **4b** ^[7]

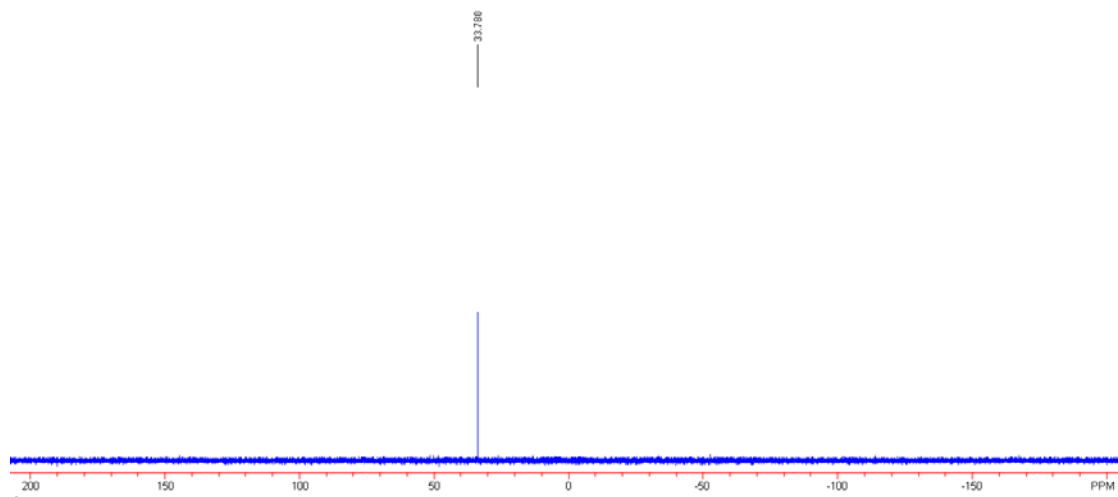
Yellow solid Mp 174.1-174.8 °C ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.99 (d, *J* = 13.6 Hz, 2H), 6.97 (d, *J* = 7.2 Hz, 2H), 7.13-7.21 (m, 2H), 7.41-7.51 (m, 6H), 7.75-7.80 (m, 4H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 40.5, 41.0, 127.15, 127.19, 127.81, 127.84, 128.3, 128.5, 130.42, 130.47, 130.5, 130.66, 130.73, 131.2, 131.5, 131.6, 132.0, 132.1. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 34.4. MS (EI) *m/z* (%): 356.0 (M + H, 60), HRMS (Micromass LCT) Calcd for C₁₉H₁₇PSe: 356.0233, Found: 356.0229.



(1-(4-Methoxyphenyl)ethyl)diphenylphosphine oxide **6a** [8]

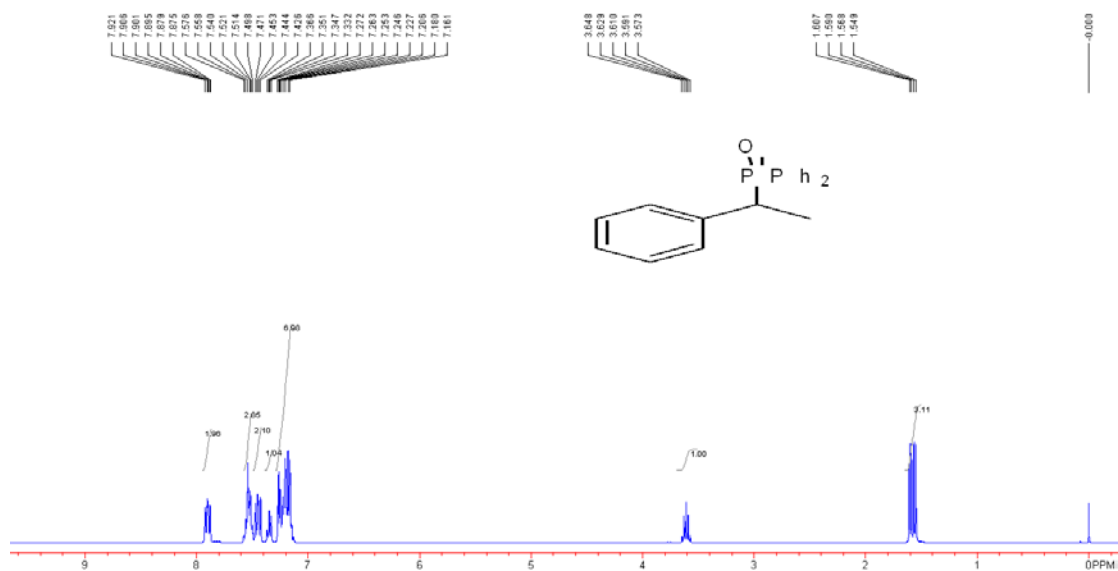
White solid, Mp 222.4-223.1 °C, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.54 (dd, $J = 10.0, 13.6$ Hz, 3H), 3.52-3.62 (m, 1H), 3.75 (s, 3H), 6.74 (d, $J = 11.2$ Hz, 2H), 7.13 (dd, $J = 2.4, 10.8$ Hz, 2H), 7.26-7.31 (m, 2H), 7.35-7.40 (m, 1H), 7.44-7.55 (m, 5H), 7.87-7.93 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 15.4, 15.5, 39.5, 40.2, 55.1, 113.60, 113.62, 127.9, 128.0, 128.5, 128.6, 129.5, 129.6, 130.06, 130.11, 131.0, 131.1, 131.22, 131.25, 131.3, 131.5, 131.60, 131.63, 132.3, 132.4, 158.42, 158.44. ^{31}P NMR (160 MHz, CDCl_3 , TMS) δ 33.8. MS (EI) m/z (%): 336.1 ($M + H$, 23), HRMS (Micromass LCT) Calcd for $\text{C}_{21}\text{H}_{21}\text{O}_2\text{P}$: 336.1279, Found: 336.1282.

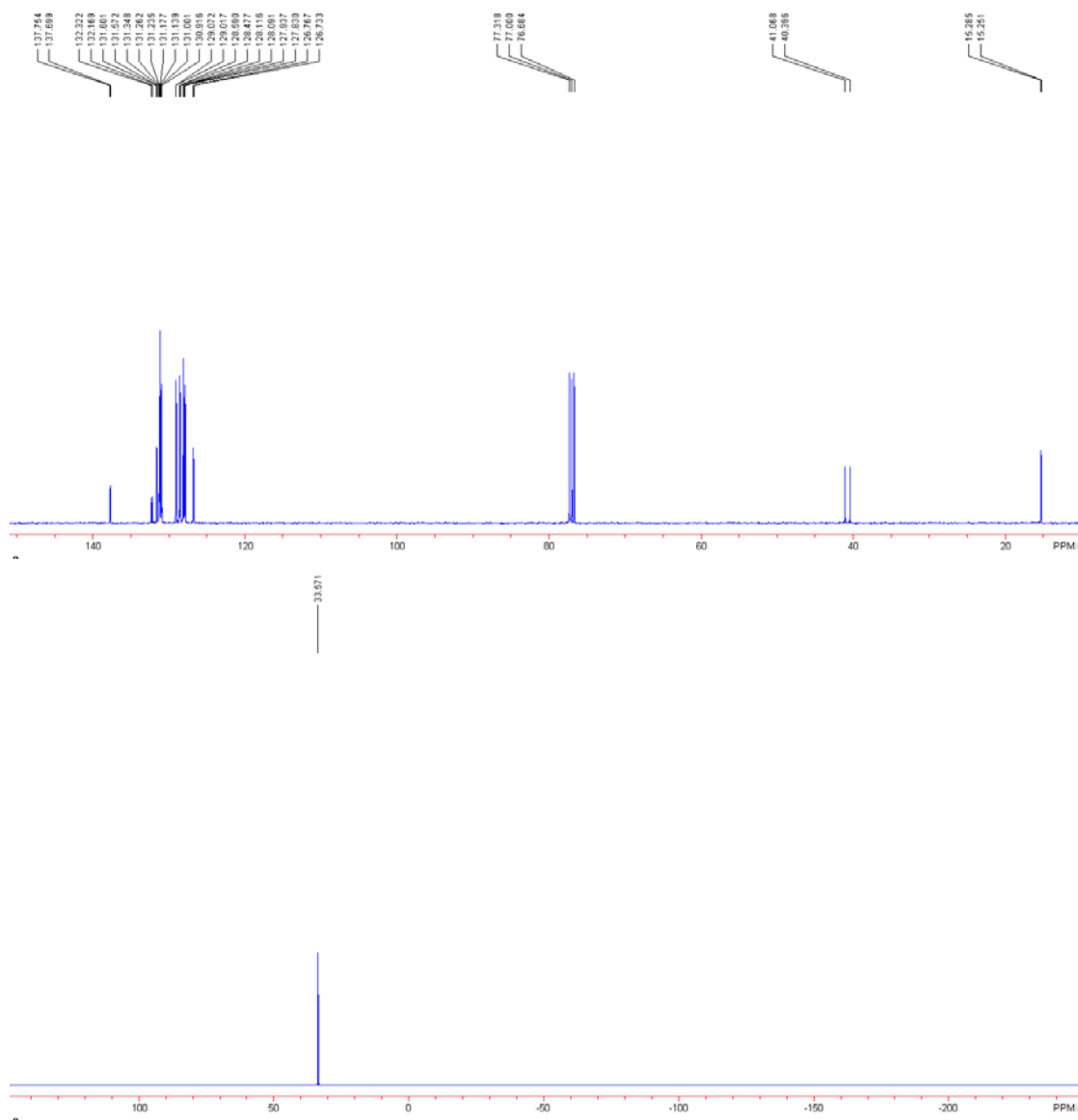




Diphenyl(1-phenylethyl)phosphine oxide **6b** [5]

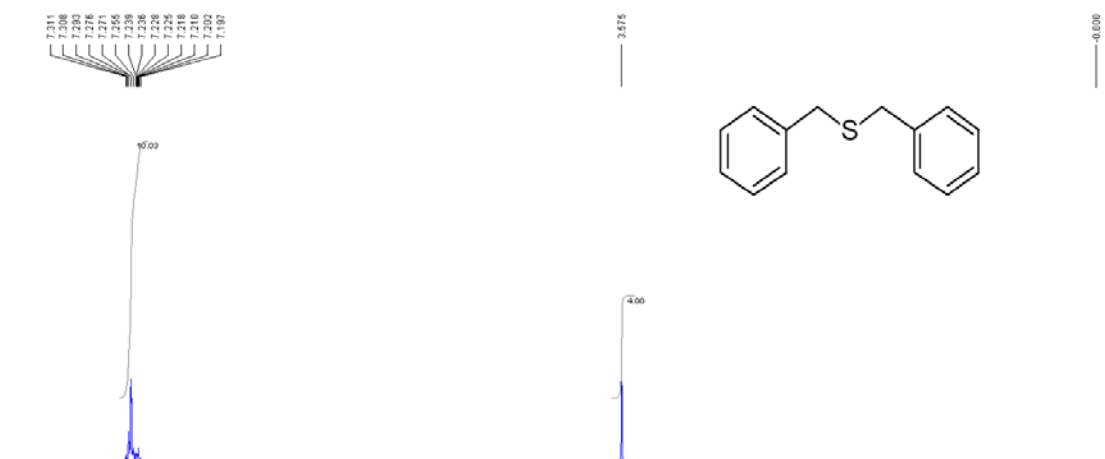
White solid, Mp 150.1-150.9 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.58 (dd, *J* = 7.6, 16.4 Hz, 3H), 3.61 (q, *J* = 7.6 Hz, 1H), 7.16-7.27 (m, 7H), 7.33-7.37 (m, 1H), 7.43-7.47 (m, 2H), 7.50-7.58 (m, 3H), 7.88-7.92 (m, 2H). ¹³C NMR (100 MHz, CDCl₃, TMS) δ 15.25, 15.29, 40.4, 41.1, 126.7, 126.8, 127.8, 127.9, 128.09, 128.12, 128.5, 128.6, 129.0, 129.1, 130.9, 131.0, 131.14, 131.18, 131.24, 131.26, 131.3, 131.57, 131.60, 132.2, 132.3, 137.7, 137.8. ³¹P NMR (160 MHz, CDCl₃, TMS) δ 33.6. MS (ESI) *m/z* (%): 307.1 (M + H, 100); HRMS (Micromass LCT) Calcd for C₂₀H₂₀OP: 307.1252, Found: 307.1252.



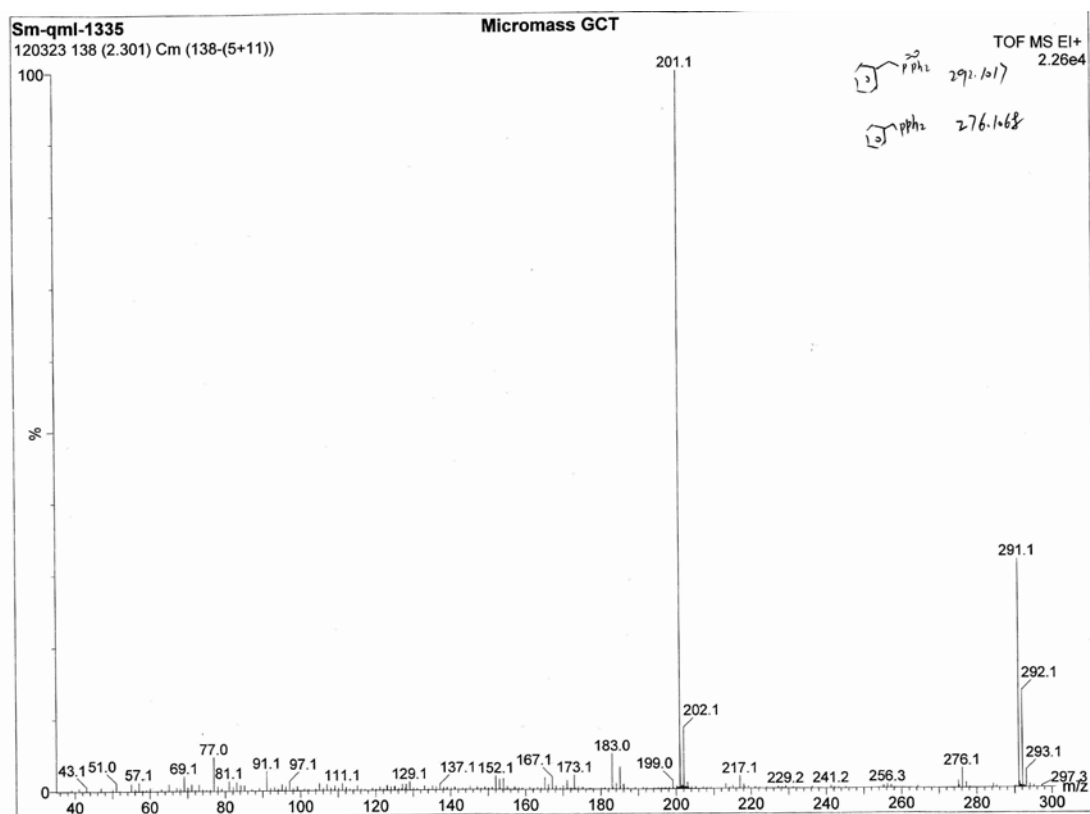


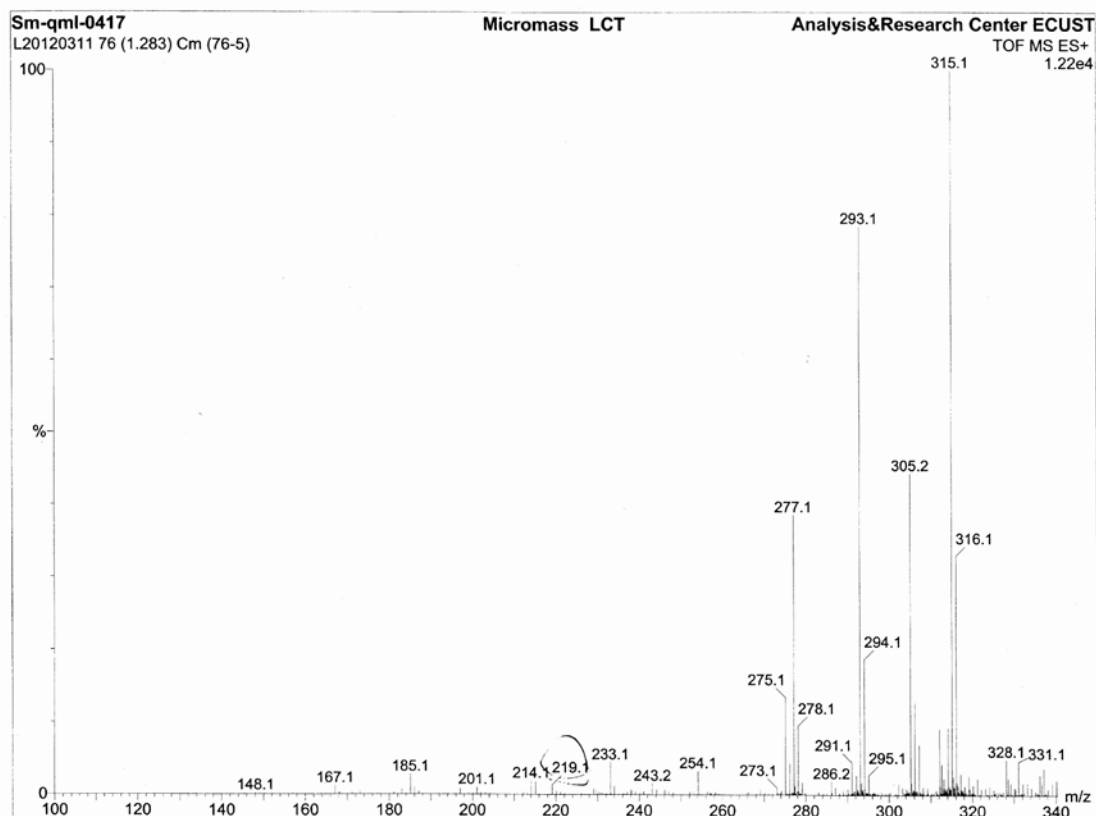
Dibenzylsulfane **8** ^[9]

Colorless liquid, ¹H NMR (400 MHz, CDCl₃, TMS) δ 3.58 (s, 4H), 7.20-7.31 (m, 10H).



Mass spectra for the reaction of benzaldehyde with Ph₂PI.





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