Supporting Information

Efficient electrosynthesis of phosphinic amides via oxidative cross-coupling between N−H/P−H

Yukang Wang,[a] Peng Qian,[a] Ji-Hu Su,[b] Yanan Li,[a] Meixiang Bi,[a] Zhenggen Zha*[a] and Zhiyong Wang*[a]

[a] *Hefei National Laboratory for Physical Sciences at Microscale, CAS Key Laboratory of Soft Matter Chemistry and Department of Chemistry & Collaborative Innovation Center of Suzhou Nano Science and Technology, University of Science and Technology of China, Hefei, Anhui 230026, P. R. China*

[b] *CAS Key Laboratory of Microscale Magnetic Resonance, Department of Modern Physics, University of Science and Technology of China, Hefei, Anhui 230026, P. R. China*

Fax: (+86)551-3603185; E-mail: zwang3@ustc.edu.cn, zgzha@ustc.edu.cn

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Experimental Section

General Information

^{1}H NMR, ^{13}C NMR and ^{31}P NMR were recorded on a Bruker-400MHz Spectrometer (^{1}H NMR: 400MHz, ^{13}C NMR: 100MHz, ^{31}P NMR: 162MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz, respectively. HRMS (ESI) were recorded on a Waters™ Q-TOF Premier. Commercially available compounds were used without further purification. Solvents were purified according to the standard procedures unless otherwise noted.

Instruments

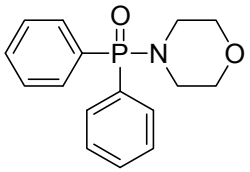
The instrument for electrolysis is dual display potentiostat (CJS-292) (made in China). The anode electrode and cathode electrode all are Pt (1.5×1.5 cm^2).

Experimental procedure

A mixture of **1a** derivatives (0.3mmol) and **2** (0.9mmol), KI (1mmol) and EtOH (10mL) was added to an undivided cell. The cell was equipped with platinum electrodes (1.5×1.5 cm^2) as both the anode and cathode. The reaction mixture was stirred and electrolyzed at a constant current of 30 mA under room temperature for corresponding time. When the reaction was finished, the solvent was removed with a rotary evaporator. The residue was purified by column chromatography on silica gel to afford the desired product.

Characterization data for the products

morpholinodiphenylphosphine oxide (3aa)

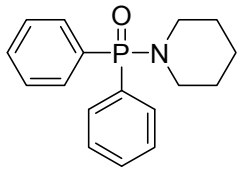


3aa

The compound **3aa** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 86% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.90–7.86 (m, 4H), 7.53–7.44 (m, 6H), 3.71 (t, *J* = 4.5 Hz, 4H), 3.08 (m, 4H); **^{13}C NMR** (100 MHz,

$CDCl_3$, ppm): δ = 131.3 (d, *J* = 9.1 Hz), 131.0 (d, *J* = 2.7 Hz), 129.7 (d, *J* = 128.5 Hz), 127.7 (d, *J* = 12.4 Hz), 66.2 (d, *J* = 6.7 Hz), 43.9; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.17. HRMS (ESI) calcd for $C_{16}H_{19}NO_2P$ $[M + H]^+$ 288.1153, found 288.1152.

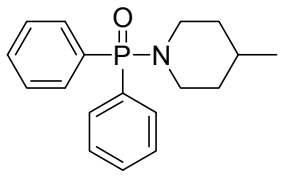
diphenyl(piperidin-1-yl)phosphine oxide (3ab)



3ab

The compound **3ab** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 75% yield; mp = 112-114 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.90–7.85 (m, 4H), 7.50–7.42 (m, 6H), 3.04-3.00 (m, 4H), 1.63-1.61 (m, 2H), 1.58-1.57 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 131.3 (d, *J* = 9.7 Hz), 130.9 (d, *J* = 128.7 Hz), 130.6 (d, *J* = 2.7 Hz), 127.5 (d, *J* = 12.3 Hz), 44.7, 25.2 (d, *J* = 6.8 Hz), 23.5; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 28.99. HRMS (ESI) calcd for $C_{17}H_{21}NOP$ $[M + H]^+$ 286.1361, found 286.1361.

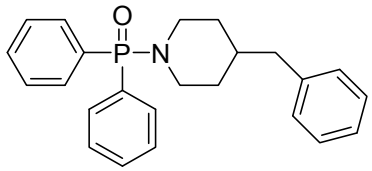
(4-methylpiperidin-1-yl)diphenylphosphine oxide (3ac)



3ac

The compound **3ac** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 84% yield; mp = 131-132 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.81–7.76 (m, 4H), 7.40–7.32 (m, 6H), 3.20-3.15 (m, 2H), 2.72-2.66 (m, 2H), 1.51-1.39 (m, 3H), 1.17-1.07 (m, 2H), 0.86 (d, *J* = 6.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 132.3 (d, *J* = 128.7 Hz), 132.1 (d, *J* = 8.9 Hz), 131.4 (d, *J* = 2.5 Hz), 128.3 (d, *J* = 12.2 Hz), 45.0, 34.4 (d, *J* = 7.2 Hz), 30.8, 21.9; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 28.98. HRMS (ESI) calcd for $C_{18}H_{23}NOP$ $[M + H]^+$ 300.1517, found 300.1517.

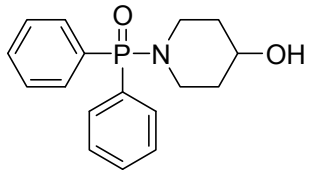
(4-benzylpiperidin-1-yl)diphenylphosphine oxide (3ad)



3ad

The compound **3ad** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 86% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.89–7.84 (m, 4H), 7.49–7.43 (m, 6H), 7.27–7.24 (m, 2H), 7.19–7.11 (m, 3H), 3.27 (s, 2H), 2.77-2.70 (m, 2H), 2.57 (d, *J* = 7.2 Hz, 2H), 1.72-1.66 (m, 1H), 1.62-1.59 (m, 2H), 1.32-1.29 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 140.0, 132.1 (d, *J* = 9.1 Hz), 131.7 (d, *J* = 128.8 Hz), 131.6 (d, *J* = 2.6 Hz), 129.0, 128.5 (d, *J* = 12.3 Hz), 128.1, 125.8, 45.1, 43.3, 38.1, 32.5 (d, *J* = 7.0 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.27. HRMS (ESI) calcd for $C_{24}H_{27}NOP$ $[M + H]^+$ 376.1830, found 376.1826.

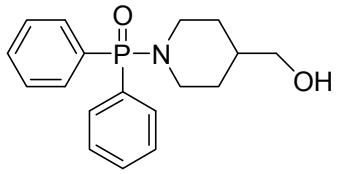
(4-hydroxypiperidin-1-yl)diphenylphosphine oxide (3ae)



3ae

The compound **3ae** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate / methanol = 20:1) to give the product as a yellow oil: 83% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.87–7.82 (m, 4H), 7.50–7.43 (m, 6H), 3.83-3.79 (m, 1H), 3.48 (s, 1H), 3.31-3.28 (m, 2H), 2.87-2.80 (m, 2H), 1.91-1.88 (m, 2H), 1.62-1.53 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 132.1 (d, *J* = 9.1 Hz), 131.7 (d, *J* = 2.4 Hz), 131.3 (d, *J* = 129.4 Hz), 128.6 (d, *J* = 12.4 Hz), 67.2, 42.8, 34.8 (d, *J* = 6.5 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.77. HRMS (ESI) calcd for $C_{17}H_{21}NO_2P$ $[M + H]^+$ 302.1310, found 302.1309.

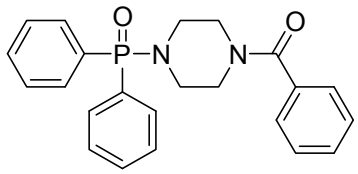
(4-(hydroxymethyl)piperidin-1-yl)diphenylphosphine oxide (3af)



3af

The compound **3af** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate / methanol = 20:1) to give the product as a yellow oil: 79% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.88–7.83 (m, 4H), 7.50–7.44 (m, 6H), 3.53 (d, *J* = 5.5 Hz, 2H), 3.31-3.30 (m, 2H), 2.84-2.76 (m, 2H), 2.59 (s, 1H), 1.71-1.69 (m, 3H), 1.35-1.30 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 131.2 (d, *J* = 9.2 Hz), 130.7 (d, *J* = 2.6 Hz), 130.0, 127.6 (d, *J* = 12.4 Hz), 60.4, 44.0, 37.7, 28.2 (d, *J* = 6.7 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.71. HRMS (ESI) calcd for $C_{18}H_{23}NO_2P$ $[M + H]^+$ 316.1466, found 316.1459.

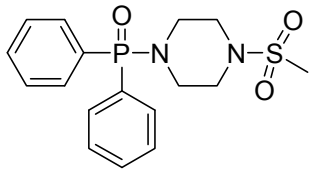
(4-(diphenylphosphoryl)piperazin-1-yl)(phenyl)methanone (3ag)



3ag

The compound **3ag** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate / methanol = 20:1) to give the product as a yellow oil: 64% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.89–7.84 (m, 4H), 7.54–7.47 (m, 6H), 7.37 (s, 5H), 3.81-3.46 (m, 4H), 3.15-3.06 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 169.5, 134.4, 131.3 (d, *J* = 9.2 Hz), 131.1 (d, *J* = 2.4 Hz), 129.7 (d, *J* = 128.1 Hz), 128.8, 127.8 (d, *J* = 12.4 Hz), 127.5, 125.9, 44.1; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 30.00. HRMS (ESI) calcd for $C_{23}H_{24}N_2O_2P$ $[M + H]^+$ 319.1575, found 319.1571.

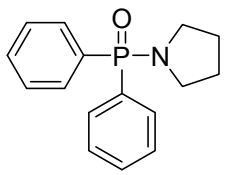
(4-(methylsulfonyl)piperazin-1-yl)diphenylphosphine oxide (3ah)



3ah

The compound **3ah** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate / methanol = 20:1) to give the product as a yellow solid: 54% yield; mp = 176-178 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.88–7.84 (m, 4H), 7.56–7.48 (m, 6H), 3.25-3.22 (m, 8H), 2.80 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 132.19 (d, *J* = 9.1 Hz), 132.18 (d, *J* = 2.7 Hz), 130.5 (d, *J* = 128.7 Hz), 128.8 (d, *J* = 12.5 Hz), 46.2 (d, *J* = 4.3 Hz), 44.9, 34.3; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 30.23. HRMS (ESI) calcd for $C_{17}H_{22}N_2O_3PS$ $[M + H]^+$ 365.1079, found 365.1082.

diphenyl(pyrrolidin-1-yl)phosphine oxide (3ai)

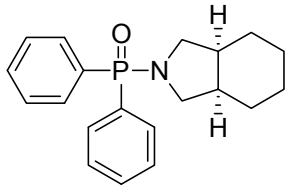


3ai

The compound **3ai** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 91% yield; mp = 92-93 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.91–7.87 (m, 4H), 7.49–7.42 (m, 6H), 3.14-3.10 (m, 4H), 1.88 (t, *J* = 6.3 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 131.6 (d, *J* = 129.1 Hz), 131.2 (d, *J* = 9.3 Hz),

130.6 (d, J = 2.5 Hz), 127.5 (d, J = 12.3 Hz), 45.9 (d, J = 2.0 Hz), 25.6 (d, J = 6.7 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 25.40. HRMS (ESI) calcd for $C_{16}H_{19}NOP$ $[M + H]^+$ 272.1204, found 272.1206.

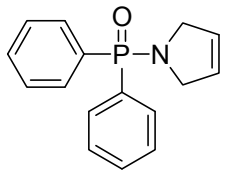
((*3aR,7aS*)-octahydro-2H-isoindol-2-yl)diphenylphosphine oxide (3aj)



3aj

The compound **3aj** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 88% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.92–7.88 (m, 4H), 7.49–7.44 (m, 6H), 3.19-3.14 (m, 2H), 2.99-2.94 (m, 2H), 2.23 (s, 2H), 1.61-1.49 (m, 6H), 1.32-1.31 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 131.6 (d, J = 129.5 Hz), 131.2 (d, J = 9.0 Hz), 130.6 (d, J = 2.5 Hz), 127.5 (d, J = 12.3 Hz), 49.6 (d, J = 1.1 Hz), 37.5 (d, J = 6.2 Hz), 24.6, 21.8; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 25.08. HRMS (ESI) calcd for $C_{20}H_{25}NOP$ $[M + H]^+$ 326.1674, found 326.1674.

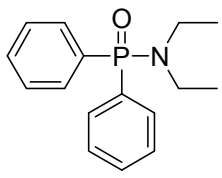
(2,5-dihydro-1H-pyrrol-1-yl)diphenylphosphine oxide (3ak)



3ak

The compound **3ak** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 80% yield; mp = 118-120 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.93–7.88 (m, 4H), 7.52–7.44 (m, 6H), 5.77 (s, 2H), 3.99 (d, J = 5.8 Hz, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 132.2 (d, J = 9.4 Hz), 132.1 (d, J = 128.7 Hz), 131.7 (d, J = 2.5 Hz), 128.6 (d, J = 12.4 Hz), 126.5 (d, J = 7.3 Hz), 54.2 (d, J = 3.5 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 25.06. HRMS (ESI) calcd for $C_{16}H_{17}NOP$ $[M + H]^+$ 270.1048, found 270.1048.

N,N-diethyl-P,P-diphenylphosphinic amide (3al)

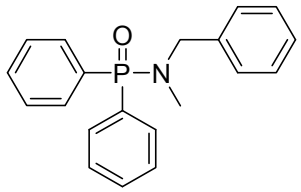


3al

The compound **3al** was prepared according to the general working procedure (4 h)

and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 57% yield; mp = 123-125 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.88–7.84 (m, 4H), 7.50–7.42 (m, 6H), 3.07 (dt, *J* = 17.9, 7.0 Hz, 4H), 1.10 (t, *J* = 7.0 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 132.5 (d, *J* = 127.8 Hz), 132.2 (d, *J* = 9.1 Hz), 131.5 (d, *J* = 2.7 Hz), 128.3 (d, *J* = 12.3 Hz), 39.2 (d, *J* = 3.4 Hz), 14.0 (d, *J* = 3.9 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 30.56. HRMS (ESI) calcd for $C_{16}H_{21}NOP$ $[M + H]^+$ 274.1361, found 274.1360.

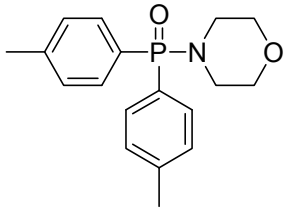
N-benzyl-N-methyl-P,P-diphenylphosphinic amide (3am)



3am

The compound **3am** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 77% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.96–7.91 (m, 4H), 7.51–7.42 (m, 6H), 7.37–7.32 (m, 4H), 7.28–7.24 (m, 1H), 4.13 (d, *J* = 7.7 Hz, 2H), 2.54 (d, *J* = 11.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 137.4 (d, *J* = 7.1 Hz), 132.2 (d, *J* = 9.2 Hz), 131.7 (d, *J* = 2.7 Hz), 131.6 (d, *J* = 128.0 Hz), 128.6, 128.4 (d, *J* = 4.5 Hz), 127.9, 127.2, 52.8(d, *J* = 3.0 Hz), 33.6 (d, *J* = 2.6 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 31.66. HRMS (ESI) calcd for $C_{20}H_{21}NOP$ $[M + H]^+$ 322.1361, found 322.1359.

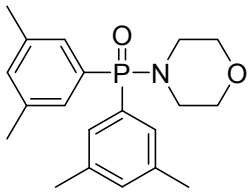
morpholinodi-*p*-tolylphosphine oxide (3ba)



3ba

The compound **3ba** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 77% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.77–7.72 (m, 4H), 7.27–7.26 (m, 4H), 3.70 (s, 4H), 3.06 (s, 4H), 2.37 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 142.3 (d, *J* = 2.6 Hz), 132.3 (d, *J* = 9.4 Hz), 129.4 (d, *J* = 12.7 Hz), 127.6 (d, *J* = 131.0 Hz), 67.2 (d, *J* = 6.7 Hz), 44.9, 21.5; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.89. HRMS (ESI) calcd for $C_{18}H_{23}NO_2P$ $[M + H]^+$ 316.1466, found 316.1464.

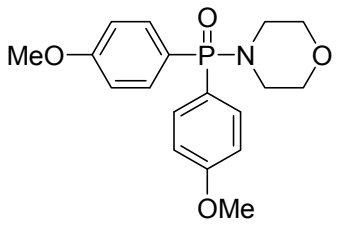
bis(3,5-dimethylphenyl)(morpholino)phosphine oxide (3ca)



3ca

The compound **3ca** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 86% yield; mp = 122-124 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.49 (s, 2H), 7.46 (s, 2H), 7.12 (s, 2H), 3.71 (t, *J* = 4.5 Hz, 4H), 3.09-3.05 (m, 4H), 2.34 (s, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 138.3 (d, *J* = 12.9 Hz), 133.6 (d, *J* = 2.7 Hz), 130.5 (d, *J* = 126.9 Hz), 129.9 (d, *J* = 8.9 Hz), 67.2 (d, *J* = 6.6 Hz), 45.0, 21.3; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 30.15. HRMS (ESI) calcd for $C_{20}H_{27}NO_2P$ $[M + H]^+$ 344.1779, found 344.1780.

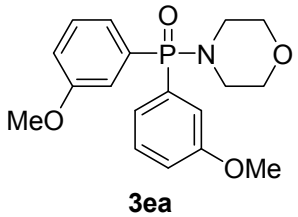
bis(4-methoxyphenyl)(morpholino)phosphine oxide (3da)



3da

The compound **3da** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 76% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.80–7.75 (m, 4H), 6.98–6.96 (m, 4H), 3.83 (s, 6H), 3.69 (t, *J* = 4.4 Hz, 4H), 3.07-3.03 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 162.4 (d, *J* = 2.8 Hz), 134.0 (d, *J* = 10.4 Hz), 120.0 (d, *J* = 135.9 Hz), 114.2 (d, *J* = 13.3 Hz), 67.1 (d, *J* = 6.8 Hz), 55.2, 44.8; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.75. HRMS (ESI) calcd for $C_{18}H_{23}NO_4P$ $[M + H]^+$ 348.1365, found 348.1364.

bis(3-methoxyphenyl)(morpholino)phosphine oxide (3ea)

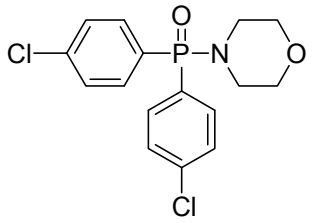


3ea

The compound **3ea** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 72% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.45–7.37 (m, 6H), 7.05–7.03 (m, 2H), 3.84 (s, 6H), 3.71 (t, *J* = 4.4 Hz, 4H), 3.10-3.06 (m, 4H); **^{13}C**

NMR (100 MHz, $CDCl_3$, ppm): δ = 158.7 (d, *J* = 15.5 Hz), 131.1 (d, *J* = 127.9 Hz), 128.9 (d, *J* = 14.6 Hz), 123.3 (d, *J* = 8.8 Hz), 117.1 (d, *J* = 2.7 Hz), 116.3 (d, *J* = 10.4 Hz), 66.2 (d, *J* = 6.8 Hz), 54.4, 44.0; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.31. HRMS (ESI) calcd for $C_{18}H_{23}NO_4P$ $[M + H]^+$ 348.1365, found 348.1363.

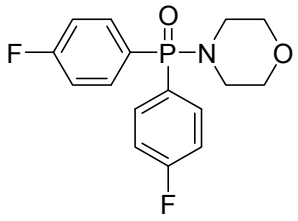
bis(4-chlorophenyl)(morpholino)phosphine oxide (3fa)



3fa

The compound **3fa** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 75% yield; mp = 139-141 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.82-7.77 (m, 4H), 7.47-7.45 (m, 4H), 3.71 (s, 4H), 3.06 (s, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 138.9 (d, *J* = 3.3 Hz), 133.8 (d, *J* = 9.9 Hz), 129.2 (d, *J* = 13.0 Hz), 129.0 (d, *J* = 131.1 Hz), 67.1 (d, *J* = 6.5 Hz), 45.0; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 27.22. HRMS (ESI) calcd for $C_{16}H_{17}Cl_2NO_2P$ $[M + H]^+$ 356.0374, found 356.0377.

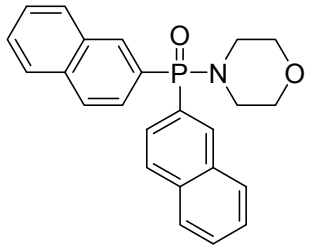
bis(4-fluorophenyl)(morpholino)phosphine oxide (3ga)



3ga

The compound **3ga** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 78% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.90–7.84 (m, 4H), 7.19–7.15 (m, 4H), 3.71 (t, *J* = 4.2 Hz, 4H), 3.08-3.04 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 165.2 (dd, *J* = 252.4 ($C_{C,F}$), 3.36 Hz), 134.8 (dd, *J* = 10.0, 8.9 Hz), 126.6 (dd, *J* = 132.8, 3.37 ($C_{C,F}$) Hz), 116.2 (dd, *J* = 21.2, 13.6 Hz), 67.1 (d, *J* = 6.6 Hz), 45.0; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 27.37. HRMS (ESI) calcd for $C_{16}H_{17}F_2NO_2P$ $[M + H]^+$ 324.0965, found 324.0965.

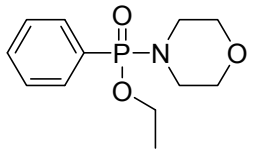
morpholinodi(naphthalen-2-yl)phosphine oxide (3ha)



3ha

The compound **3ha** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 85% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 8.59 (d, *J* = 13.9 Hz, 2H), 7.95–7.84 (m, 8H), 7.59–7.52 (m, 4H), 3.77 (s, 4H), 3.17 (s, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 135.1 (d, *J* = 9.2 Hz), 134.7 (d, *J* = 2.1 Hz), 132.6 (d, *J* = 13.8 Hz), 128.9, 128.6, 128.5, 128.3, 127.3 (d, *J* = 78.7 Hz), 127.2, 126.6 (d, *J* = 9.3 Hz), 67.3(d, *J* = 6.7 Hz), 45.1; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.29. HRMS (ESI) calcd for $C_{24}H_{23}NO_2P$ $[M + H]^+$ 388.1466, found 388.1467.

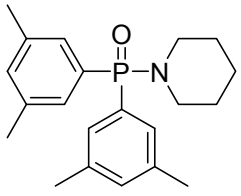
ethyl morpholino(phenyl)phosphinate (3ia)



3ia

The compound **3ia** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 88% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.76–7.71 (m, 2H), 7.54–7.45 (m, 3H), 4.22–4.09 (m, 2H), 3.63 (s, 4H), 3.11-3.10 (m, 4H), 1.39 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 131.7 (d, *J* = 2.9 Hz), 131.1 (d, *J* = 9.4 Hz), 129.7 (d, *J* = 172.5 Hz), 128.3 (d, *J* = 14.0 Hz), 66.8 (d, *J* = 5.7 Hz), 60.5 (d, *J* = 5.9 Hz), 40.9, 16.2 (d, *J* = 6.5 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 21.04. HRMS (ESI) calcd for $C_{12}H_{19}NO_3P$ $[M + H]^+$ 256.1103, found 256.1103.

bis(3,5-dimethylphenyl)(piperidin-1-yl)phosphine oxide (3cb)

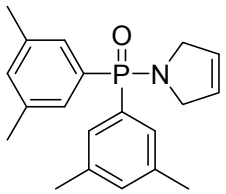


3cb

The compound **3cb** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 84% yield; mp = 145-146 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.50 (s, 2H), 7.47 (s, 2H), 7.09 (s, 2H), 3.02-2.98 (m, 4H), 2.33 (s, 12H), 1.63-1.56 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 137.0 (d, *J* = 12.8 Hz), 132.2 (d, *J* = 2.8

Hz), 130.8 (d, *J* = 127.3 Hz), 128.8 (d, *J* = 8.9 Hz), 44.7, 25.2 (d, *J* = 6.8 Hz), 23.6, 20.3; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 29.89. HRMS (ESI) calcd for $C_{21}H_{29}NOP$ $[M + H]^+$ 342.1987, found 342.1985.

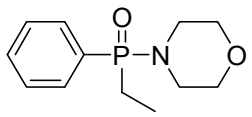
(2,5-dihydro-1H-pyrrol-1-yl)bis(3,5-dimethylphenyl)phosphine oxide (3ck)



3ck

The compound **3ck** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow solid: 86% yield; mp = 132-134 °C; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.53 (s, 2H), 7.50 (s, 2H), 7.11 (s, 2H), 5.76 (s, 2H), 3.98 (d, *J* = 6.0 Hz, 4H), 2.34 (s, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 137.2 (d, *J* = 13.0 Hz), 132.4 (d, *J* = 2.8 Hz), 131.0 (d, *J* = 127.6 Hz), 128.8 (d, *J* = 9.3 Hz), 125.5 (d, *J* = 7.2 Hz), 53.2 (d, *J* = 3.4 Hz), 20.3; **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 25.86. HRMS (ESI) calcd for $C_{20}H_{25}NOP$ $[M + H]^+$ 326.1674, found 326.1672.

ethyl(morpholino)(phenyl)phosphine oxide (3ja)

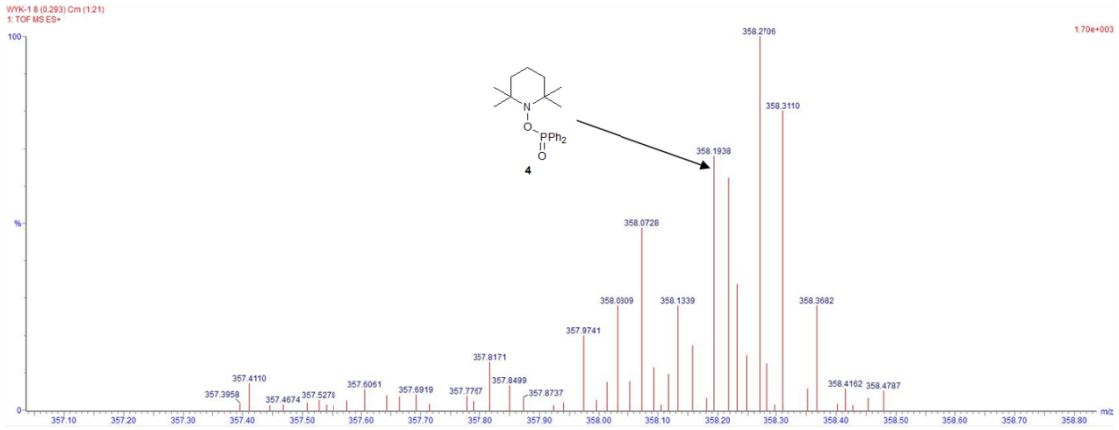


3ja

The compound **3ja** was prepared according to the general working procedure (4 h) and purified by column chromatography (ethyl acetate) to give the product as a yellow oil: 67% yield; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ = 7.78-7.73 (m, 2H), 7.56-7.47 (m, 3H), 3.69 (t, *J* = 4.6 Hz, 4H), 3.12-3.01 (m, 4H), 2.09-1.96 (m, 2H), 1.14-1.06 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ = 130.9 (d, *J* = 2.5 Hz), 130.7 (d, *J* = 9.0 Hz), 129.5 (d, *J* = 118.9 Hz), 127.6 (d, *J* = 11.8 Hz), 66.1 (d, *J* = 6.8 Hz), 43.3, 19.0 (d, *J* = 91.7 Hz), 5.1 (d, *J* = 4.4 Hz); **^{31}P NMR** (162 MHz, $CDCl_3$, ppm) δ = 40.04. HRMS (ESI) calcd for $C_{12}H_{19}NO_2P$ $[M + H]^+$ 240.1153, found 240.1156.

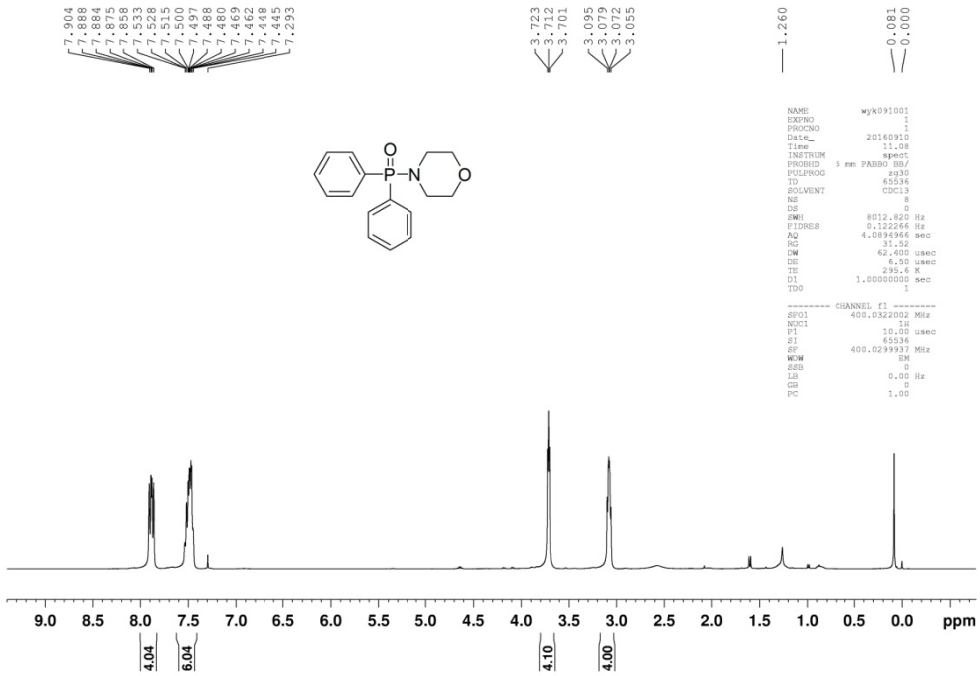
ESI-HRMS analysis for the TEMPO−P(O)Ph$_2$ adduct 4

The adduct **4** was detected by ESI-HRMS measurement of the crude reaction mixture. HRMS (ESI) calcd for $C_{21}H_{29}NOP$ $[M + H]^+$ 358.1936, found 358.1938.

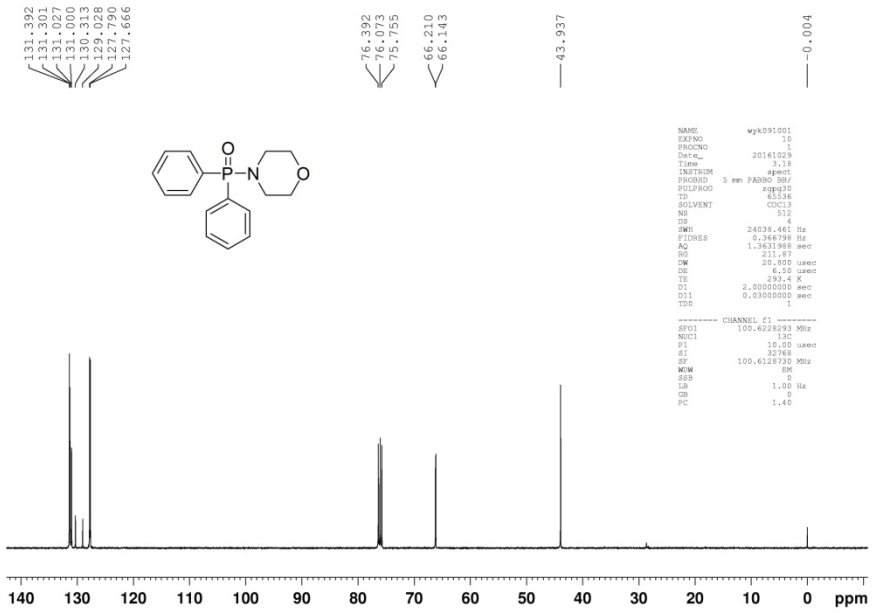


NMR spectra for the products

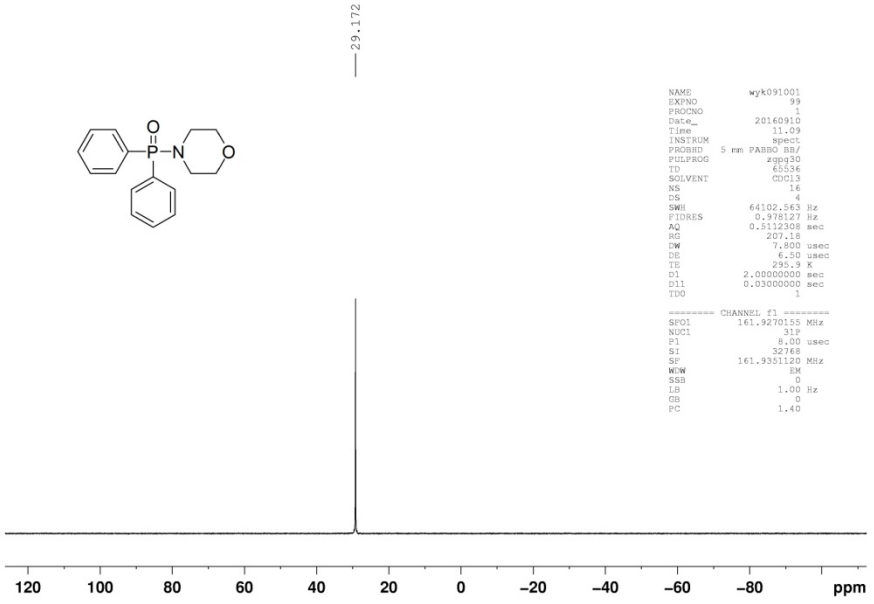
^{1}H NMR of **3aa**



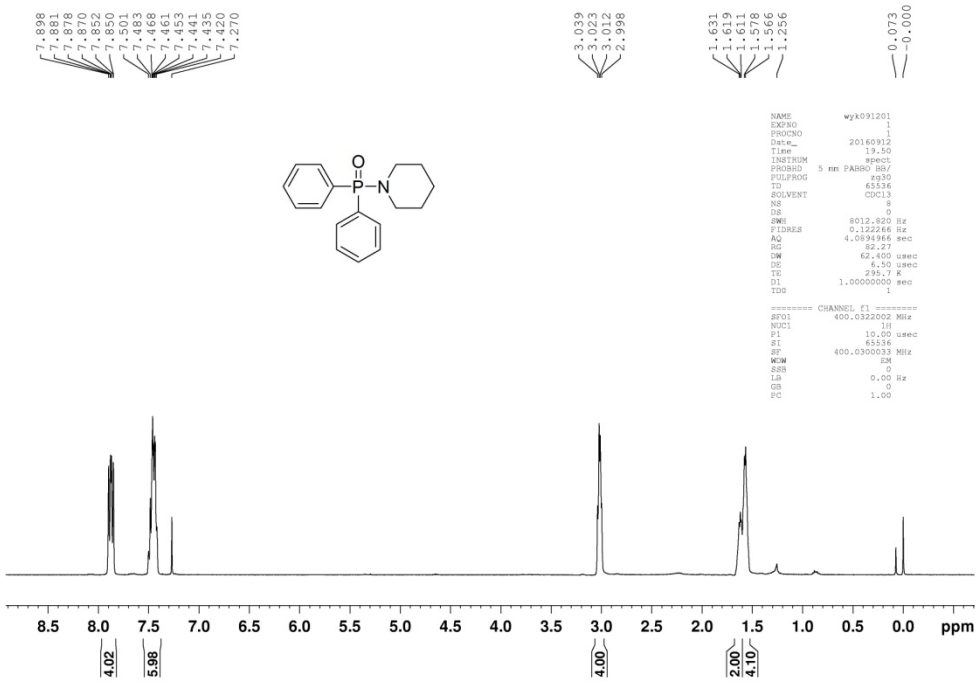
^{13}C NMR of **3aa**



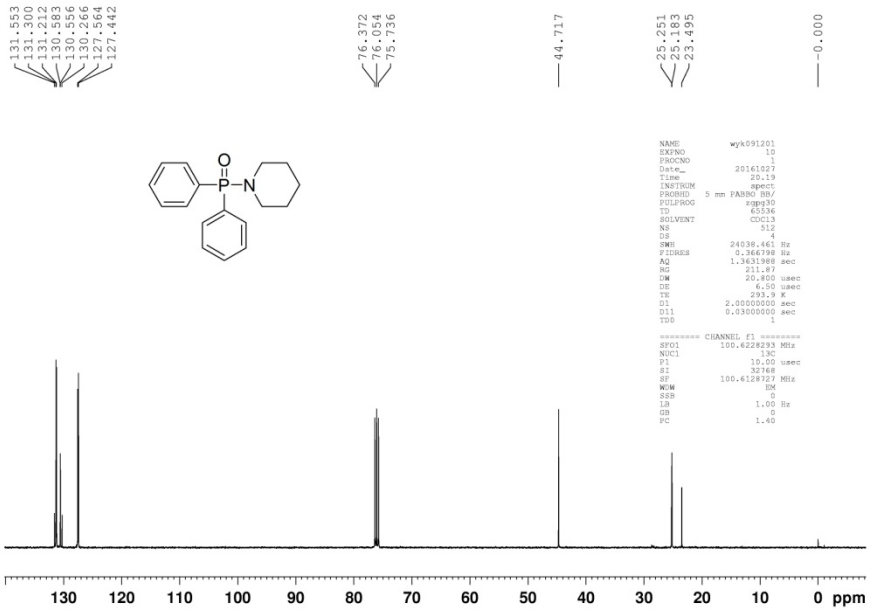
^{31}P NMR of **3aa**



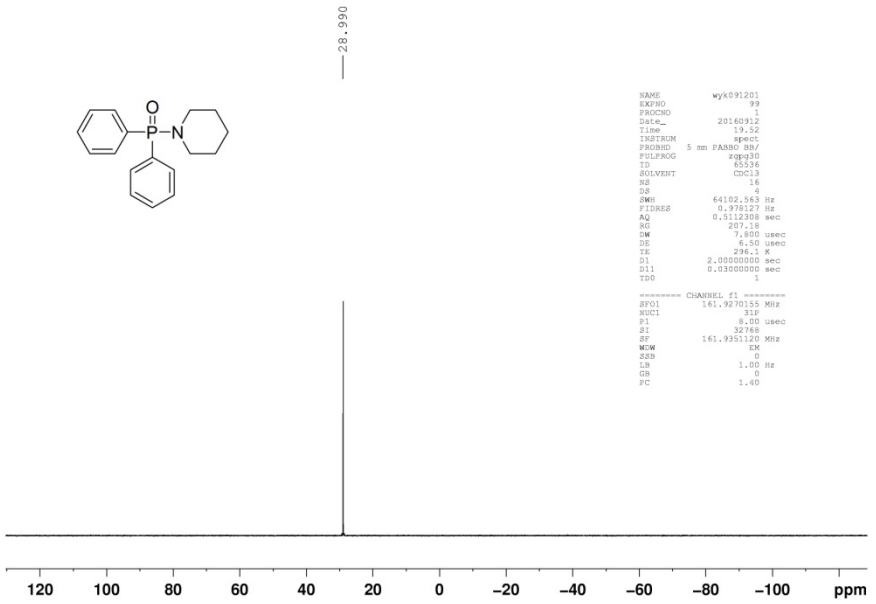
^{1}H NMR of **3ab**



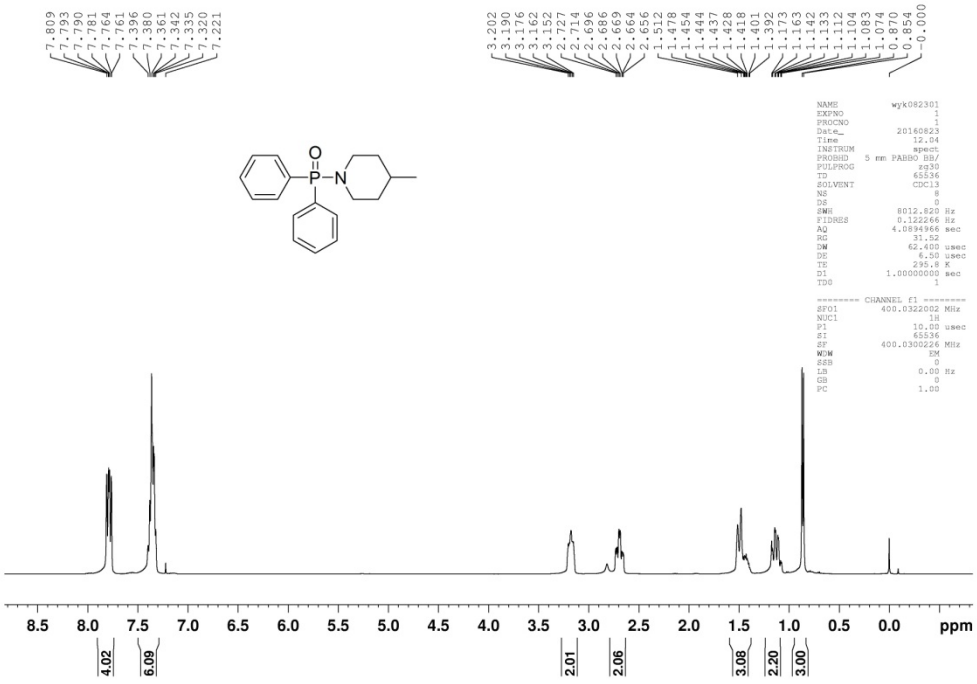
^{13}C NMR of **3ab**



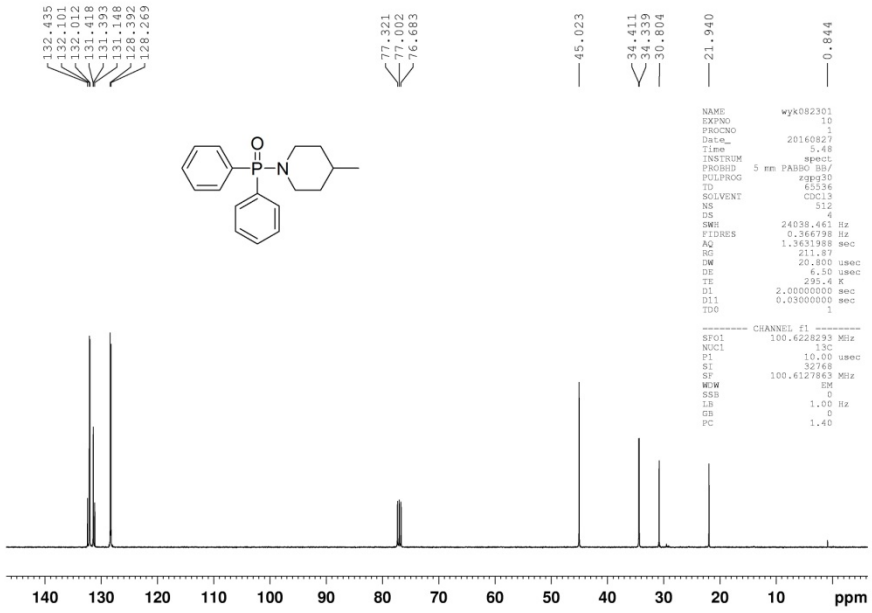
^{31}P NMR of **3ab**



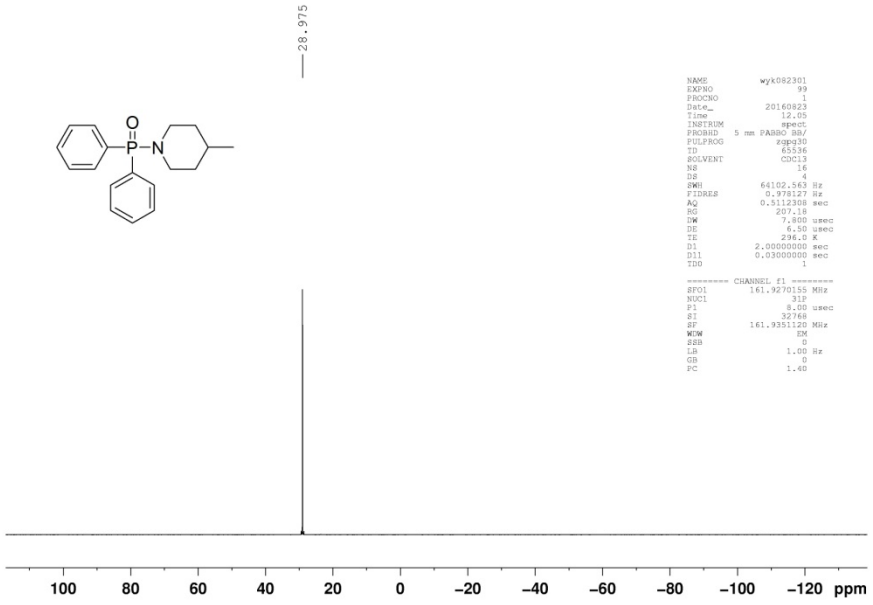
^{1}H NMR of **3ac**



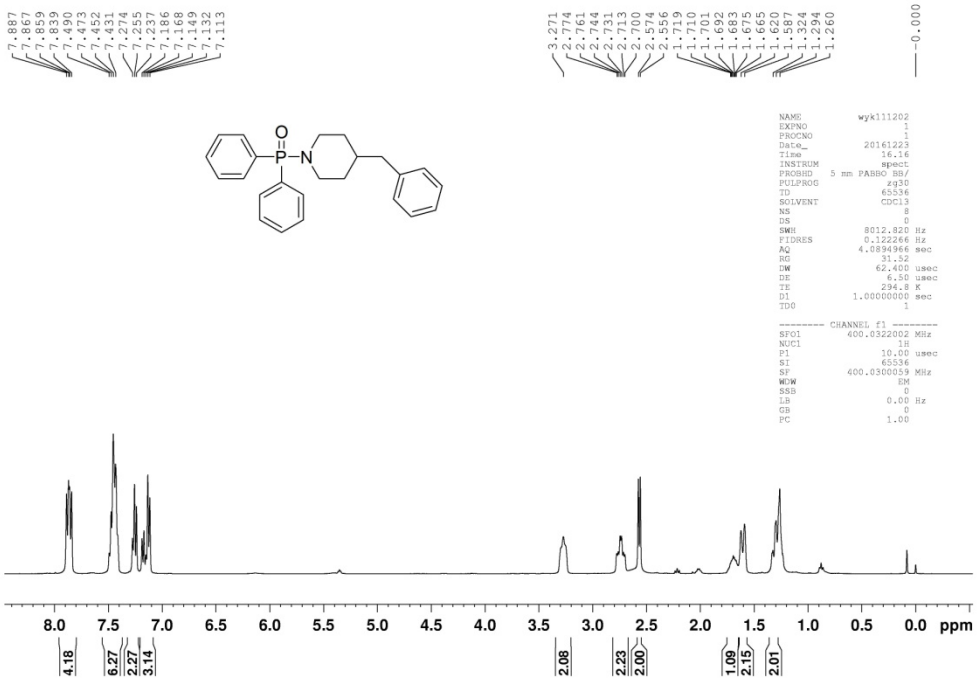
^{13}C NMR of **3ac**



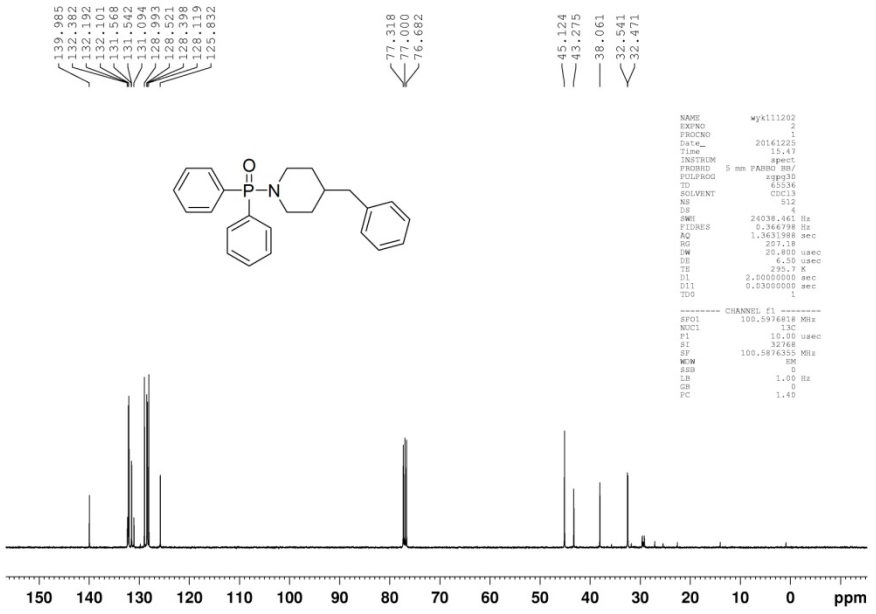
^{31}P NMR of **3ac**



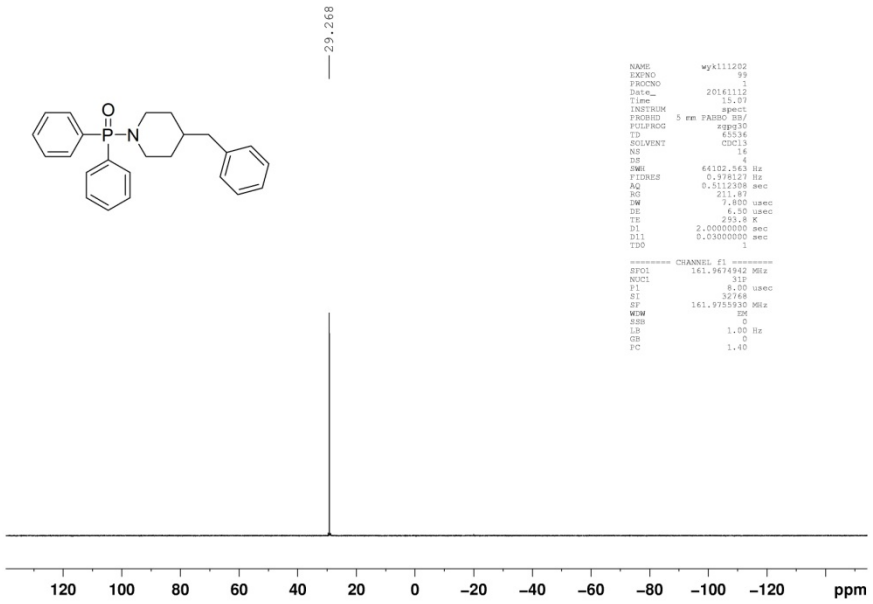
^{1}H NMR of **3ad**



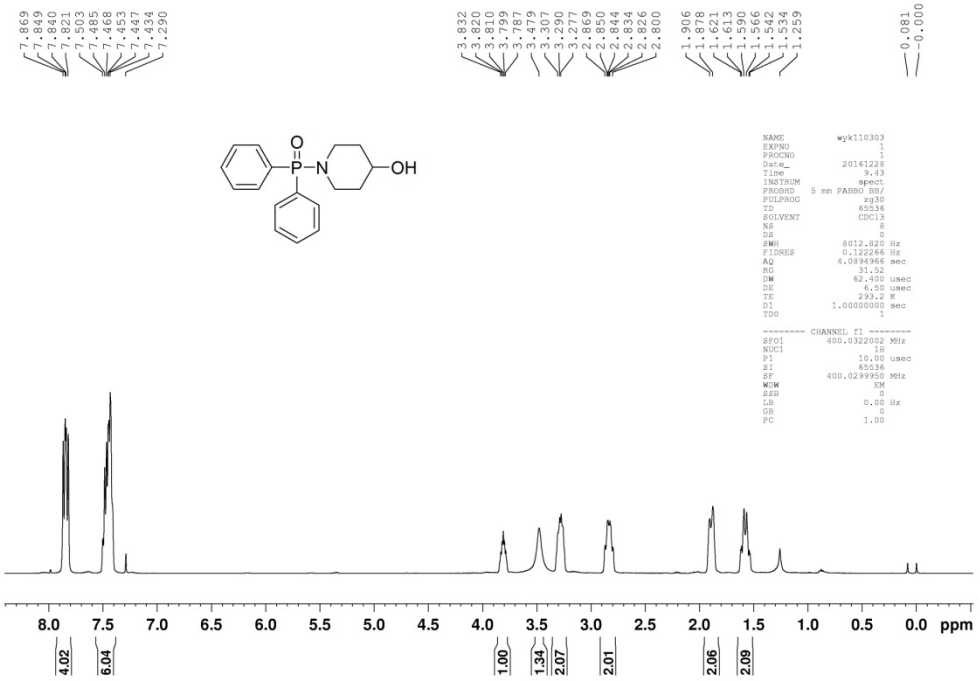
^{13}C NMR of **3ad**



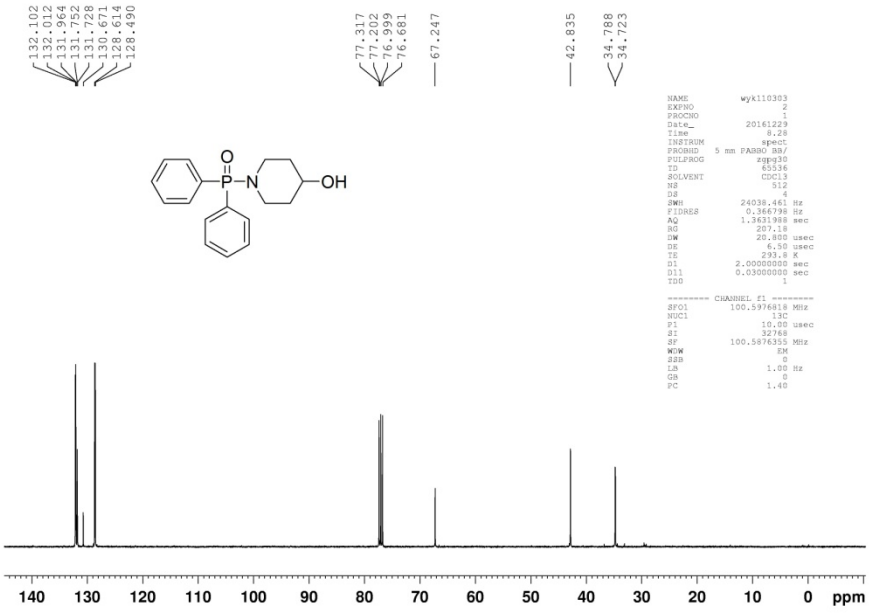
^{31}P NMR of **3ad**



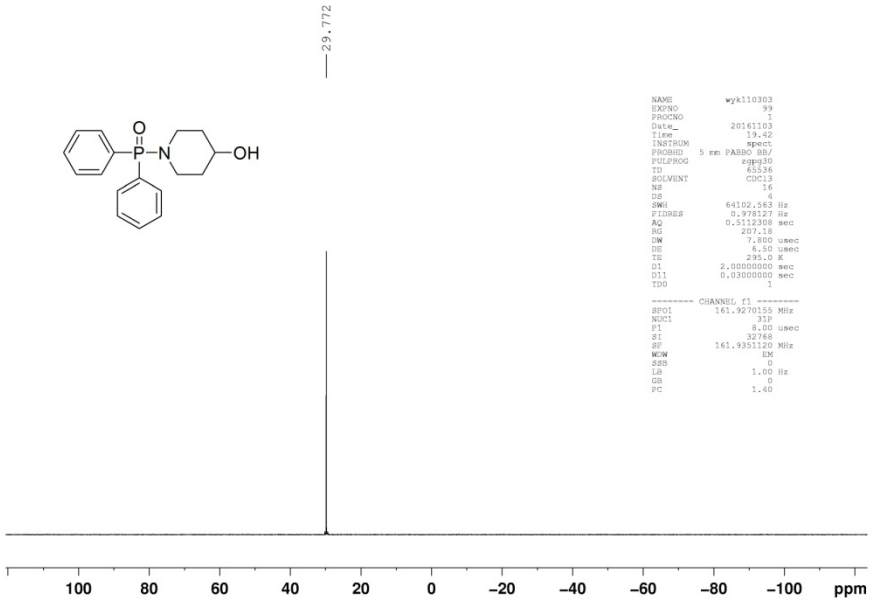
^{1}H NMR of **3ae**



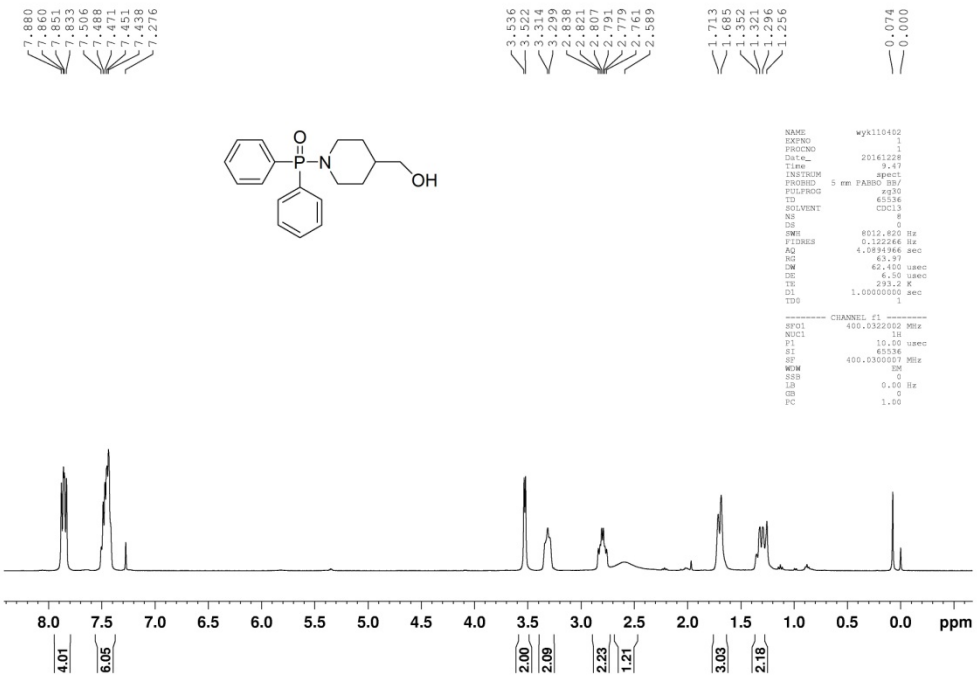
^{13}C NMR of **3ae**



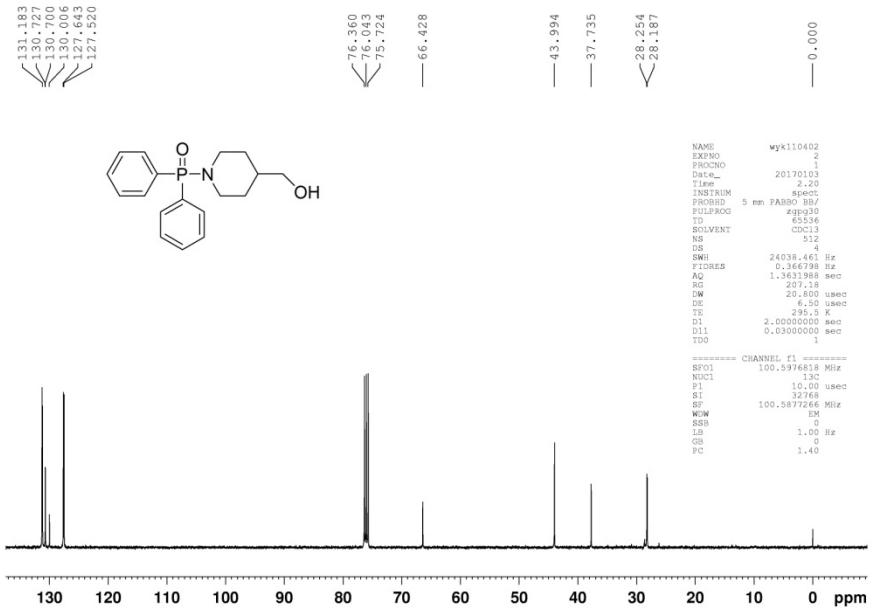
^{31}P NMR of **3ae**



^{1}H NMR of **3af**

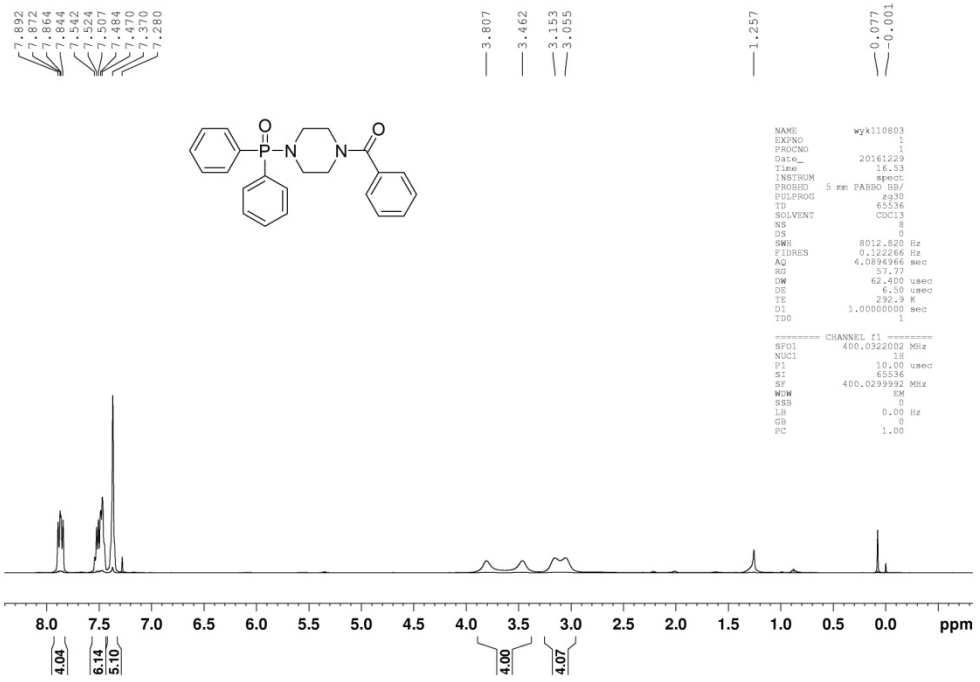


^{13}C NMR of **3af**

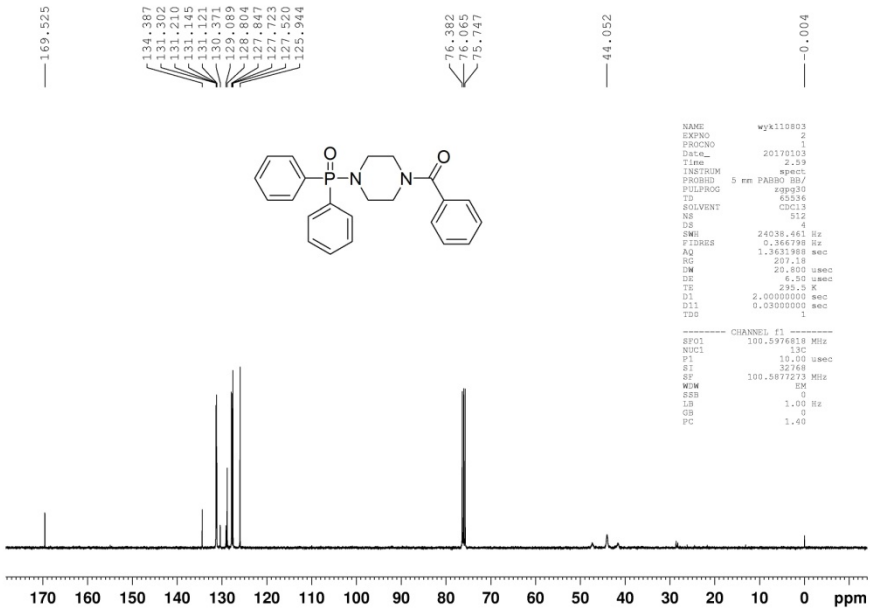


^{31}P NMR of **3af**

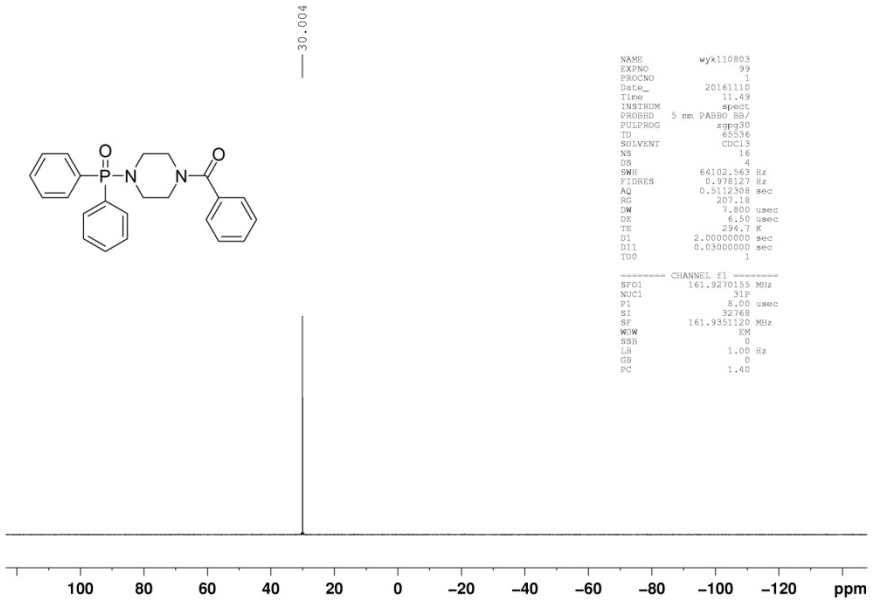
^{1}H NMR of **3ag**



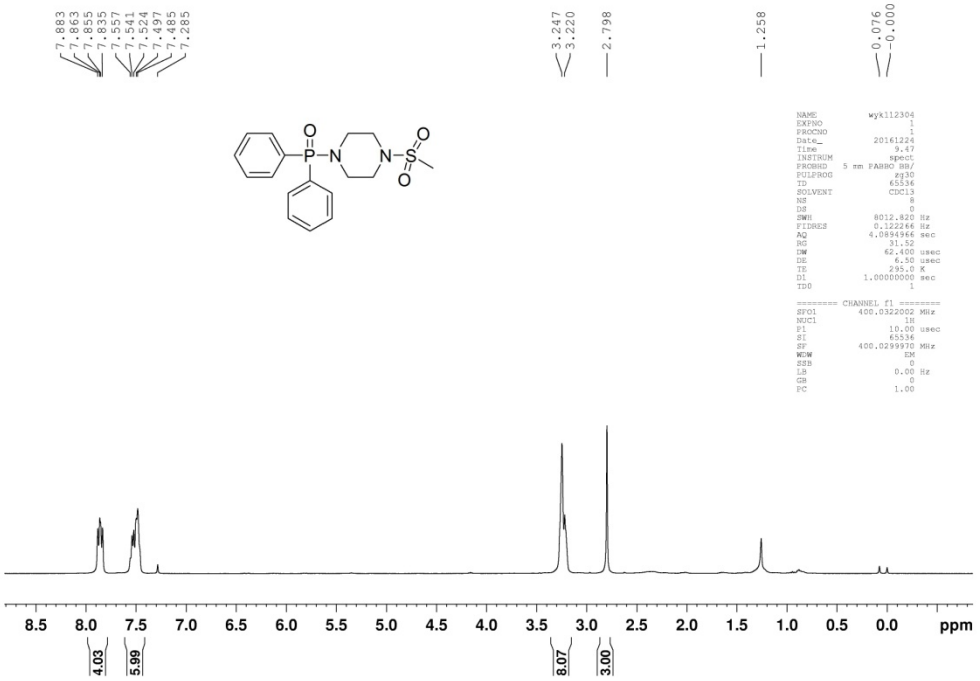
^{13}C NMR of **3ag**



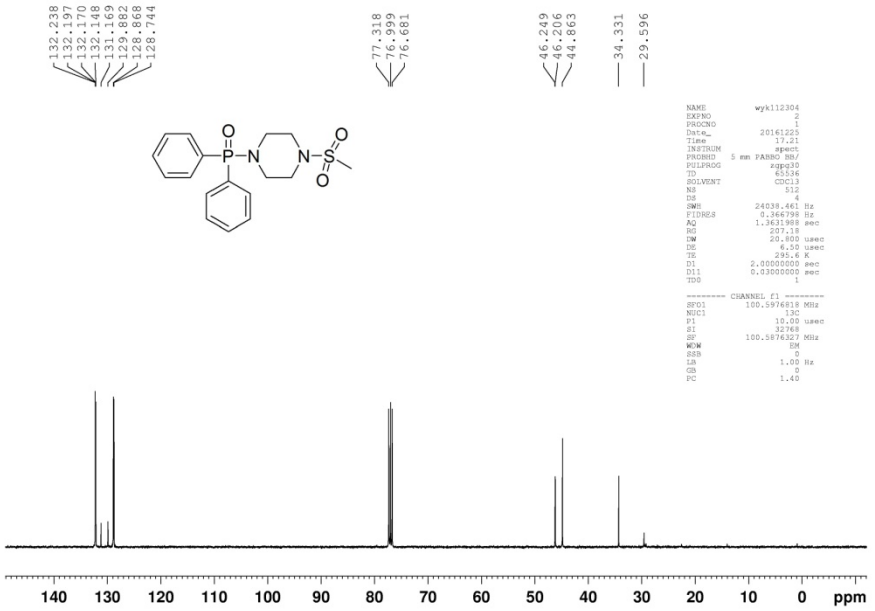
^{31}P NMR of **3ag**



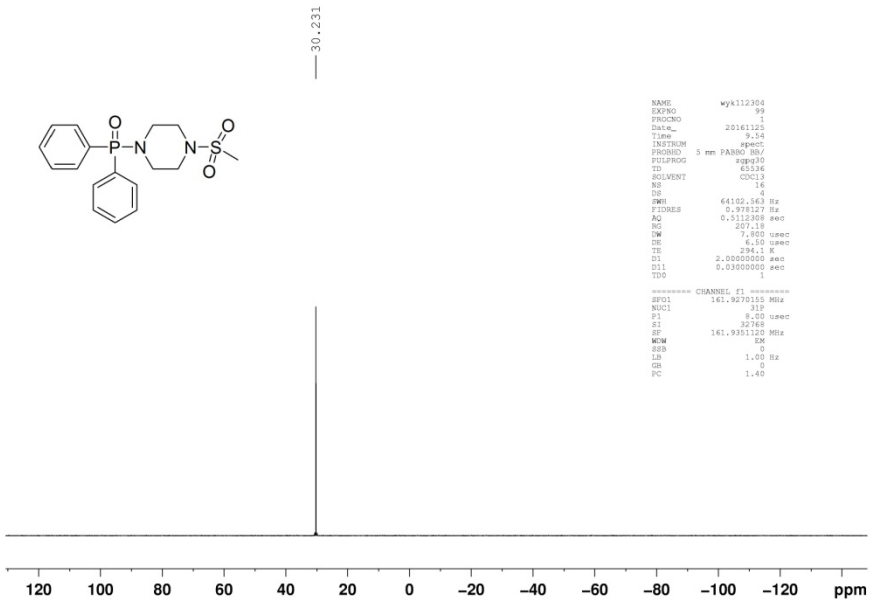
^{1}H NMR of **3ah**



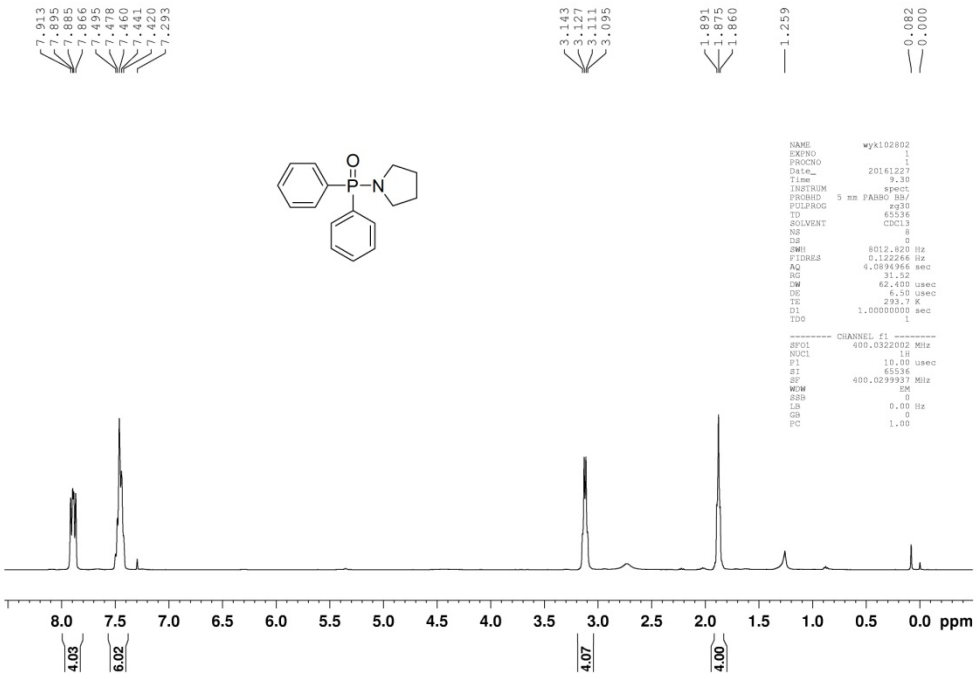
^{13}C NMR of **3ah**



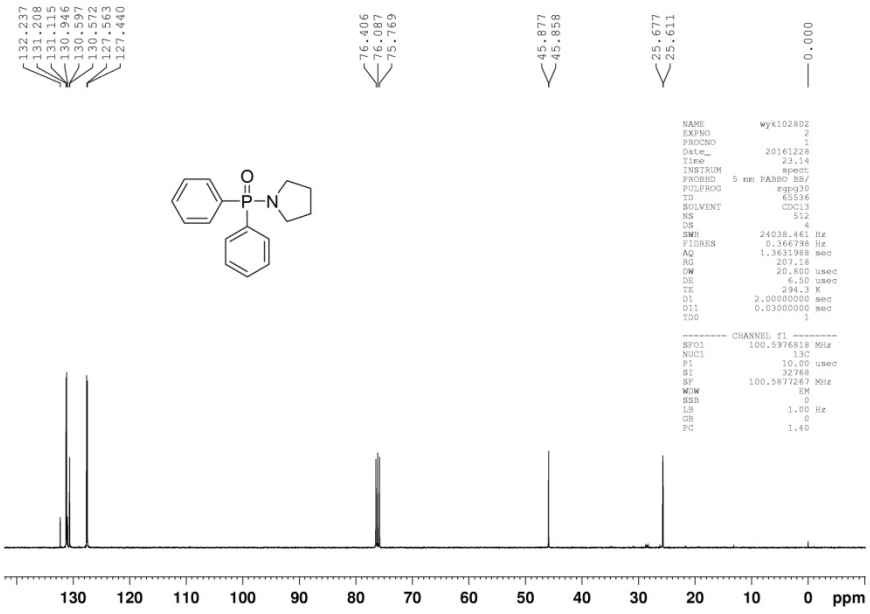
^{31}P NMR of **3ah**



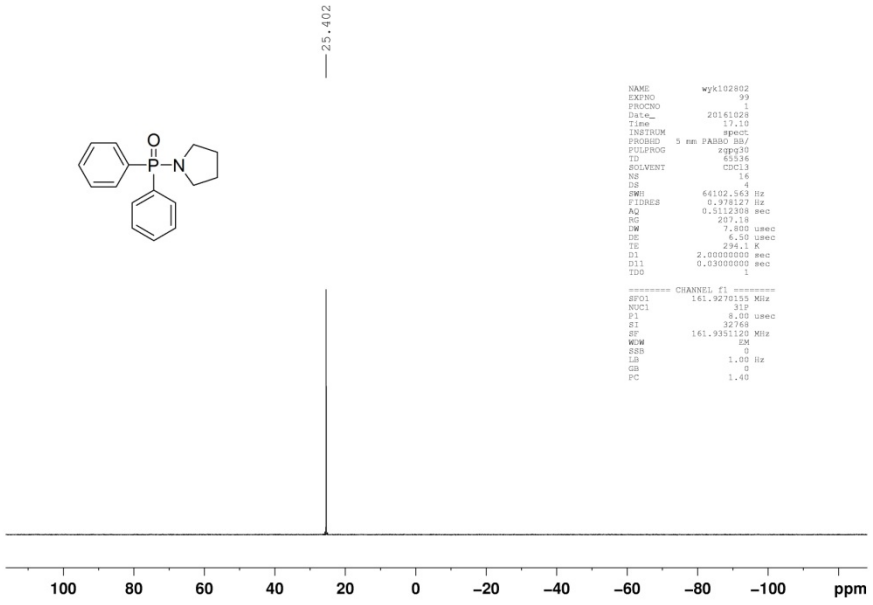
^{1}H NMR of **3ai**



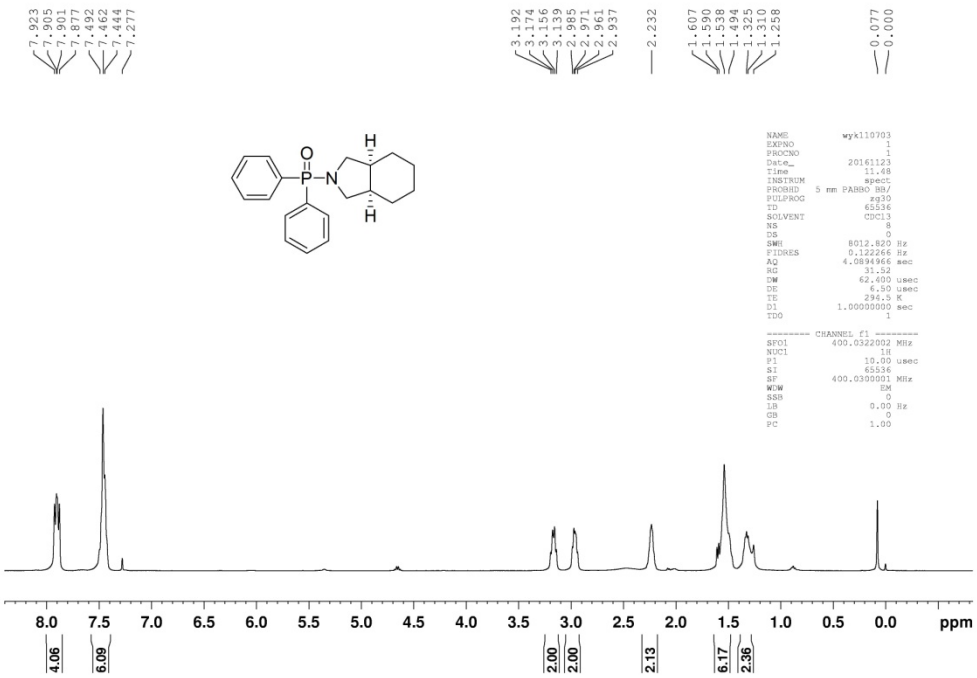
^{13}C NMR of **3ai**



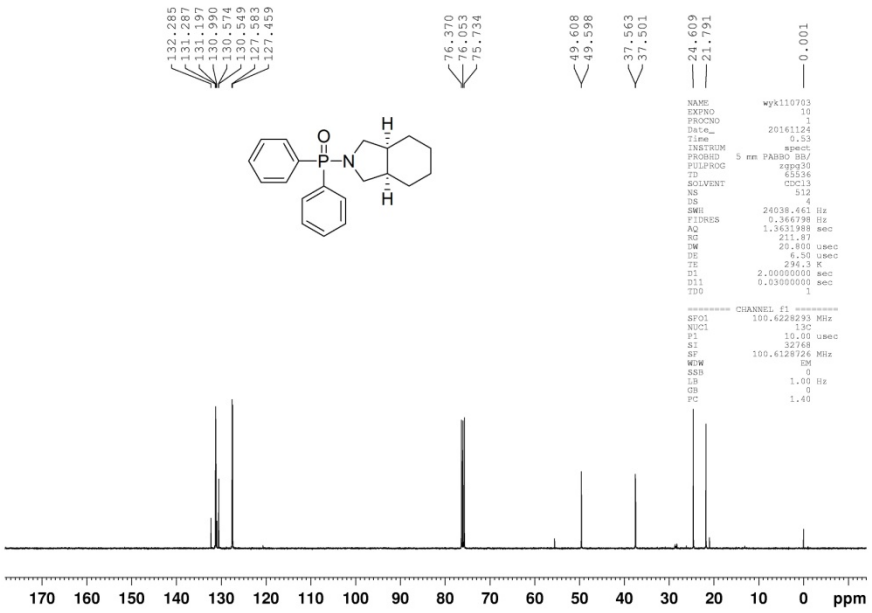
^{31}P NMR of **3ai**



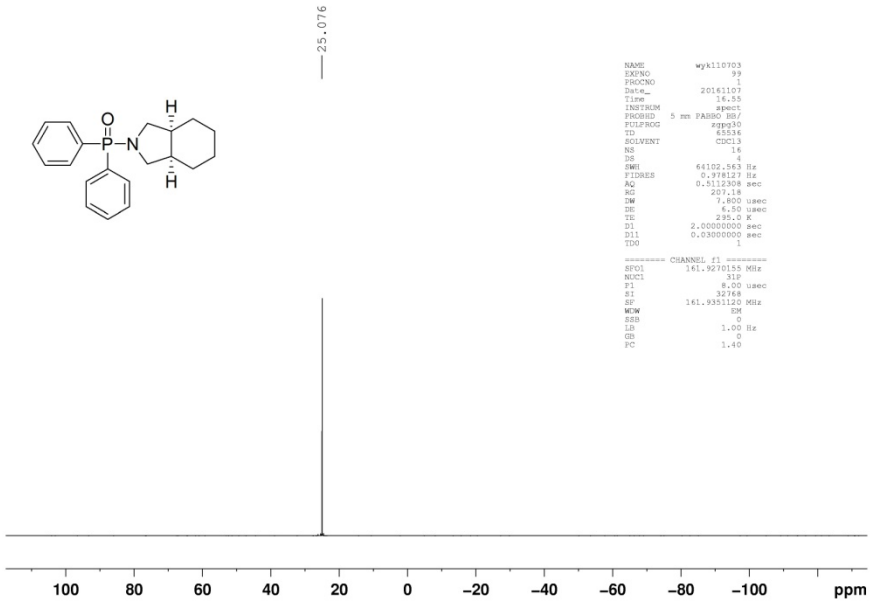
^{1}H NMR of **3aj**



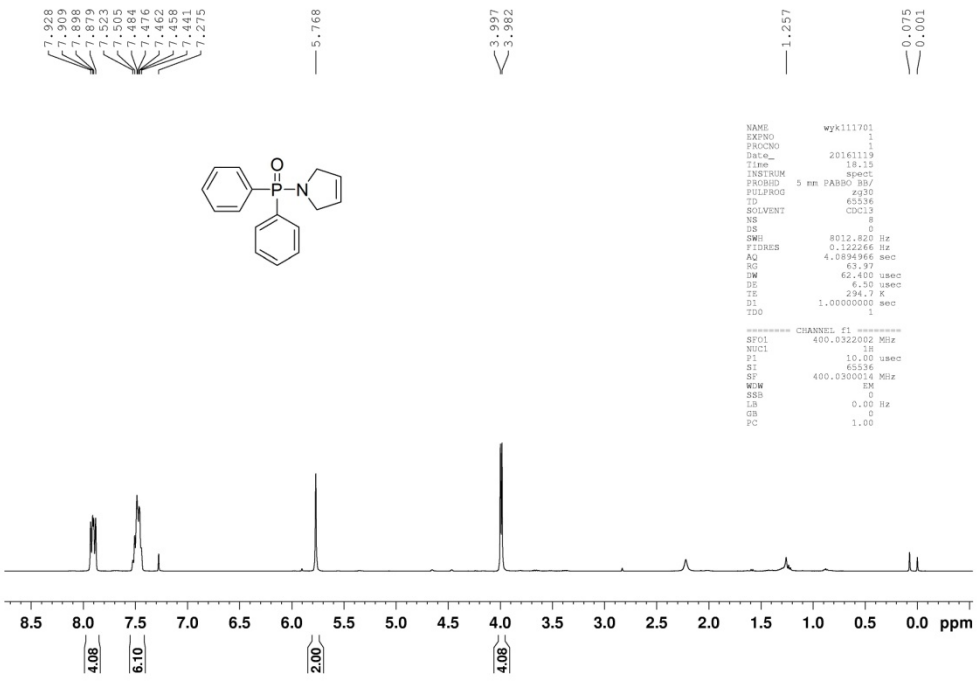
^{13}C NMR of **3aj**



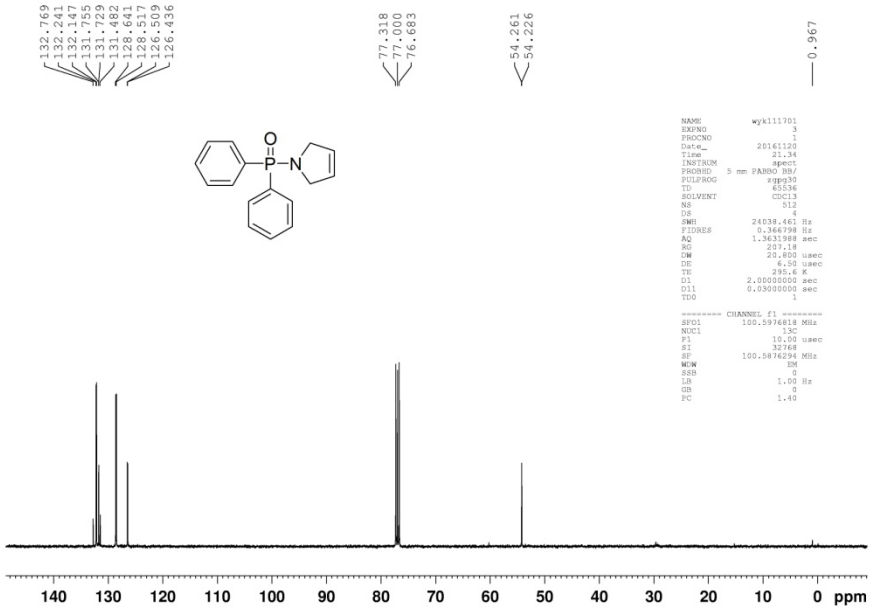
^{31}P NMR of **3aj**



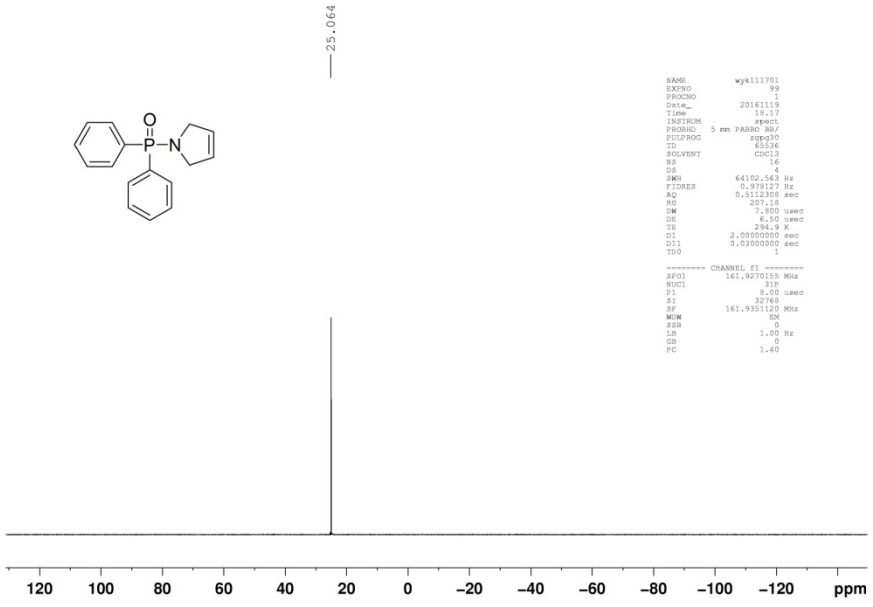
^{1}H NMR of **3ak**



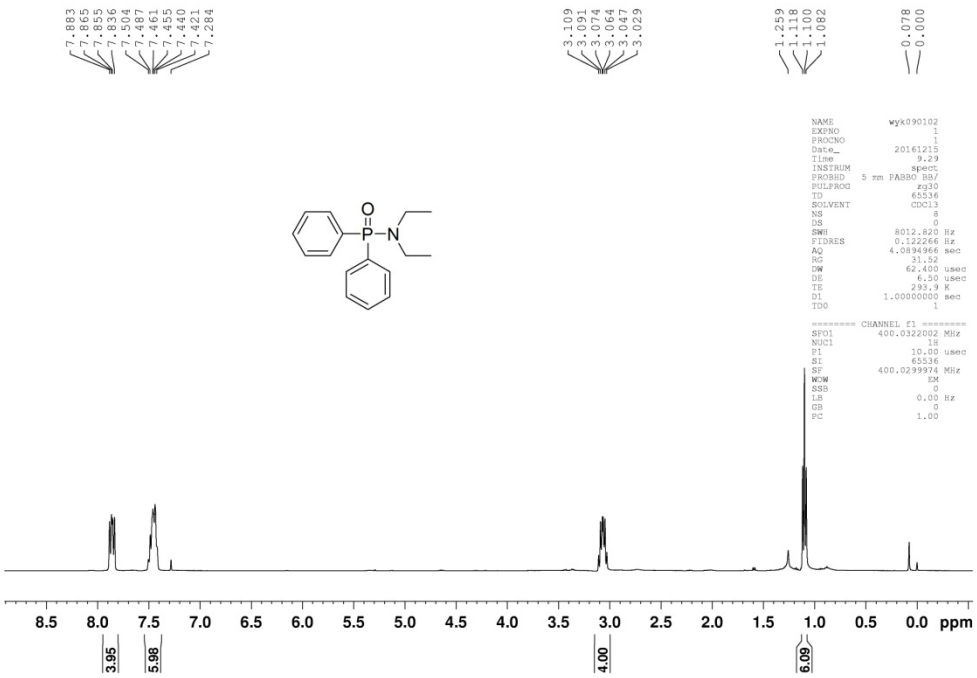
^{13}C NMR of **3ak**



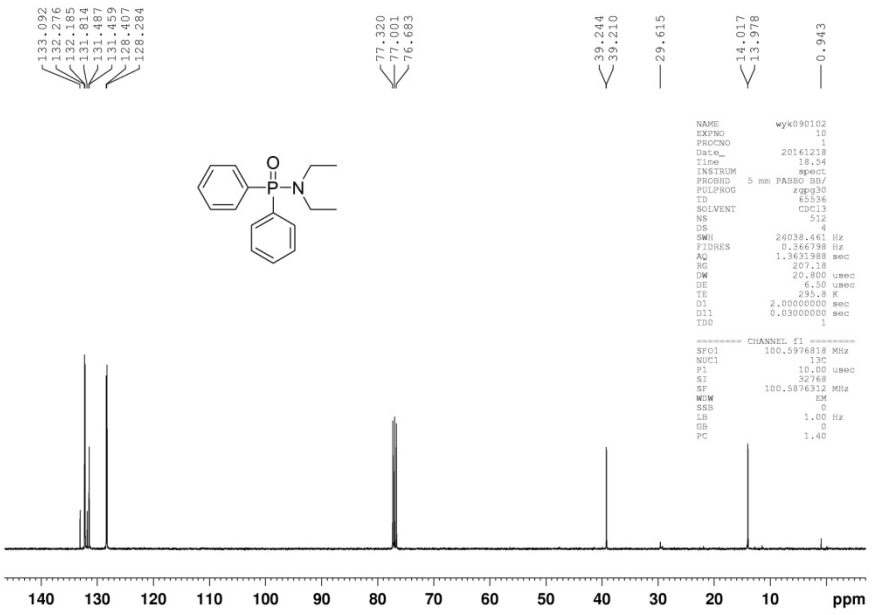
^{31}P NMR of **3ak**



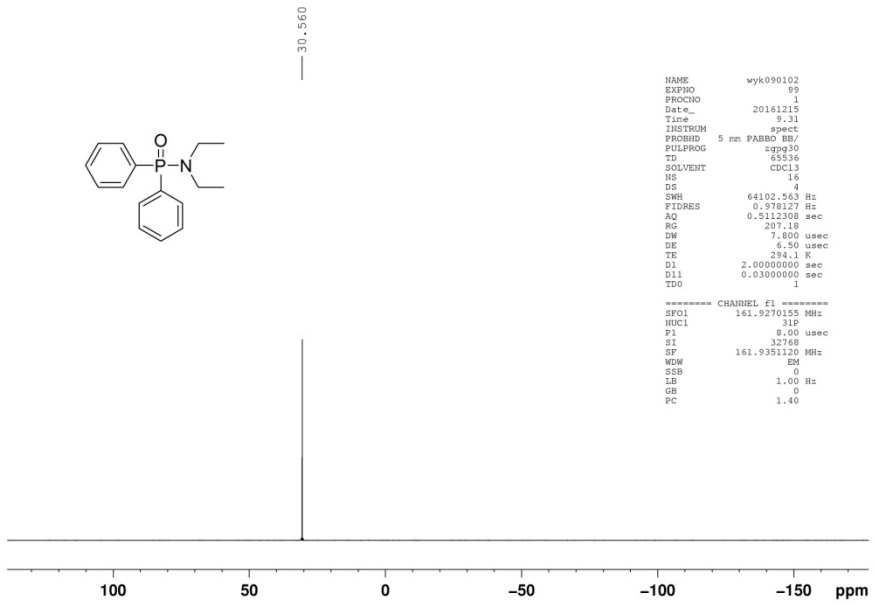
^{1}H NMR of **3al**



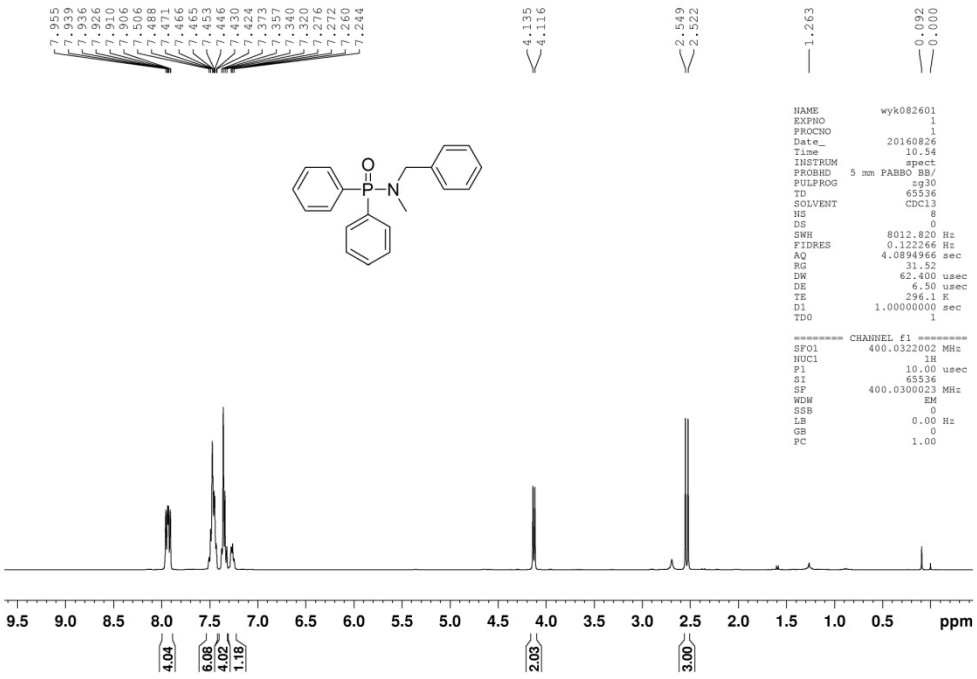
^{13}C NMR of **3al**



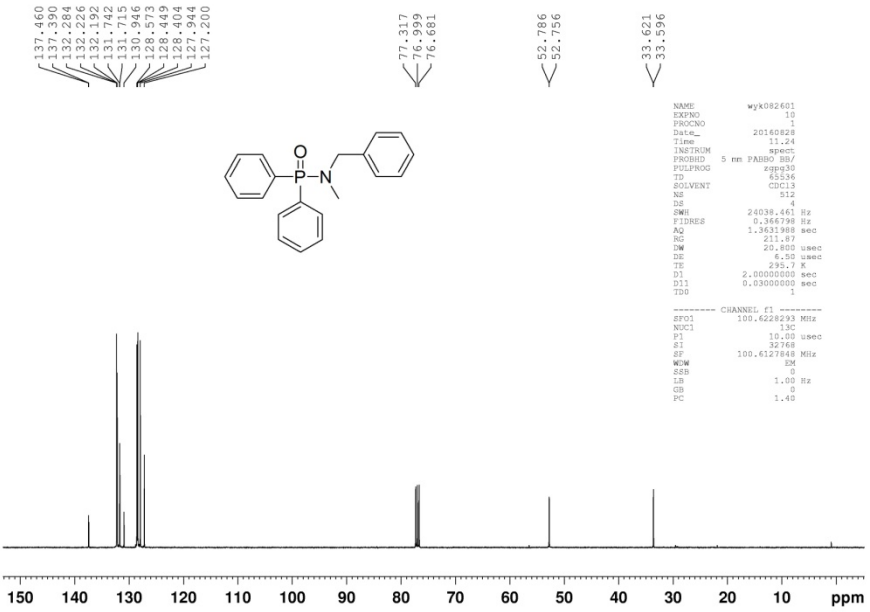
^{31}P NMR of **3al**



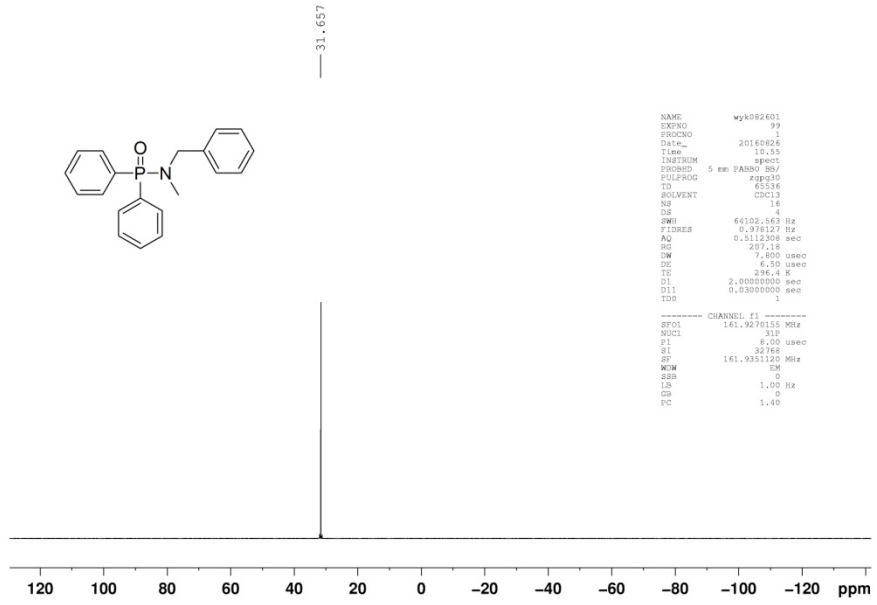
^{1}H NMR of **3am**



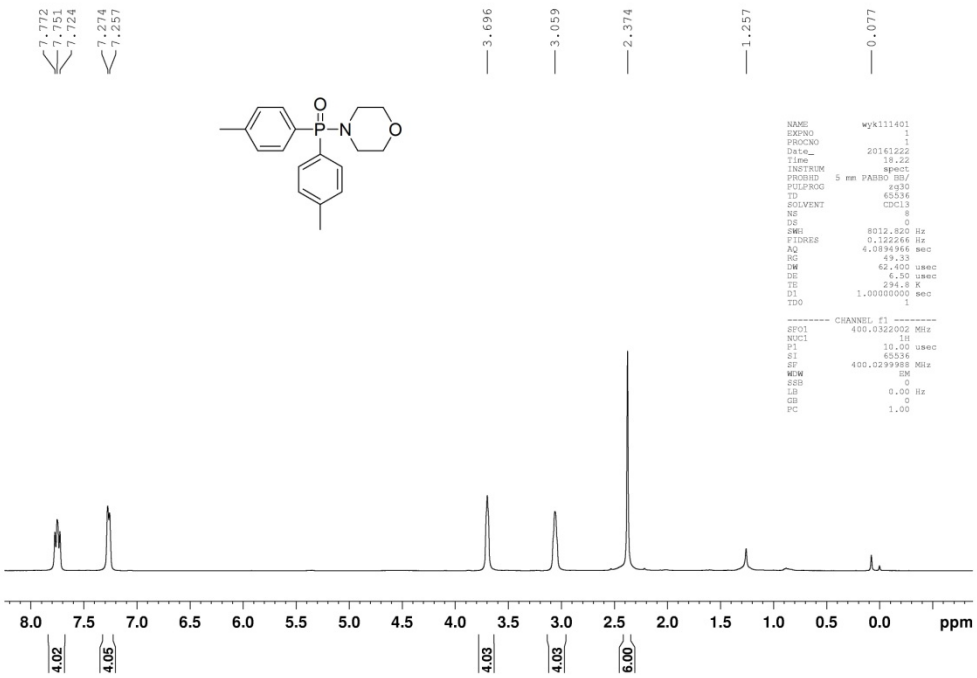
^{13}C NMR of **3am**



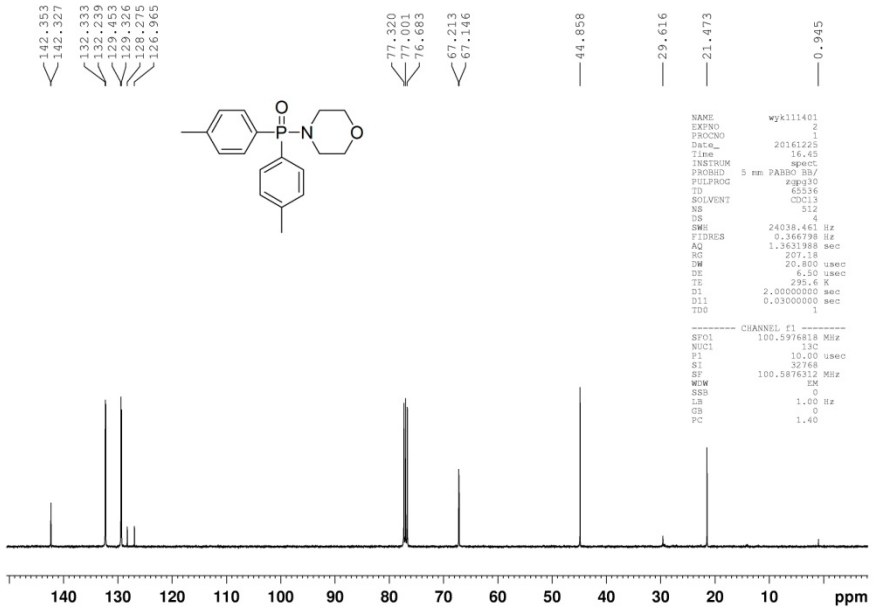
^{31}P NMR of **3am**



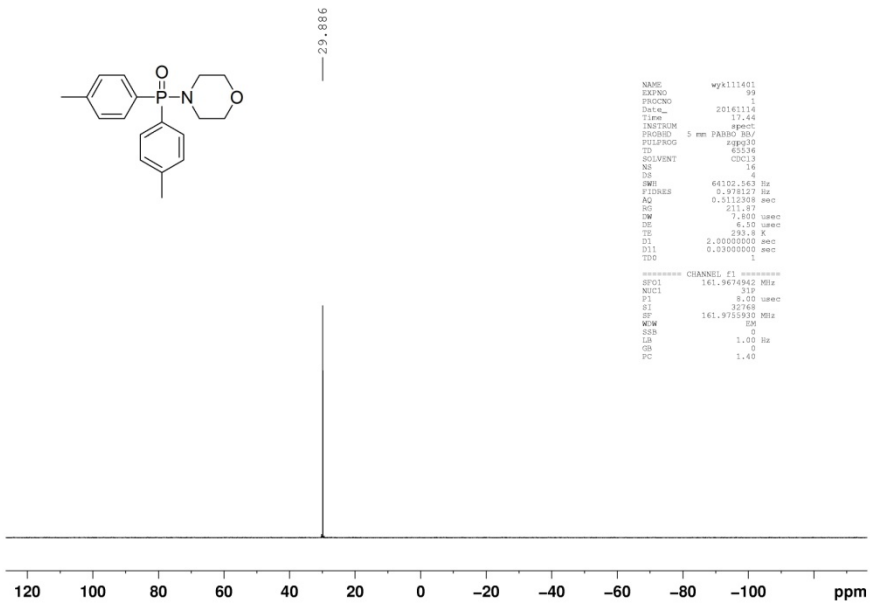
^{1}H NMR of **3ba**



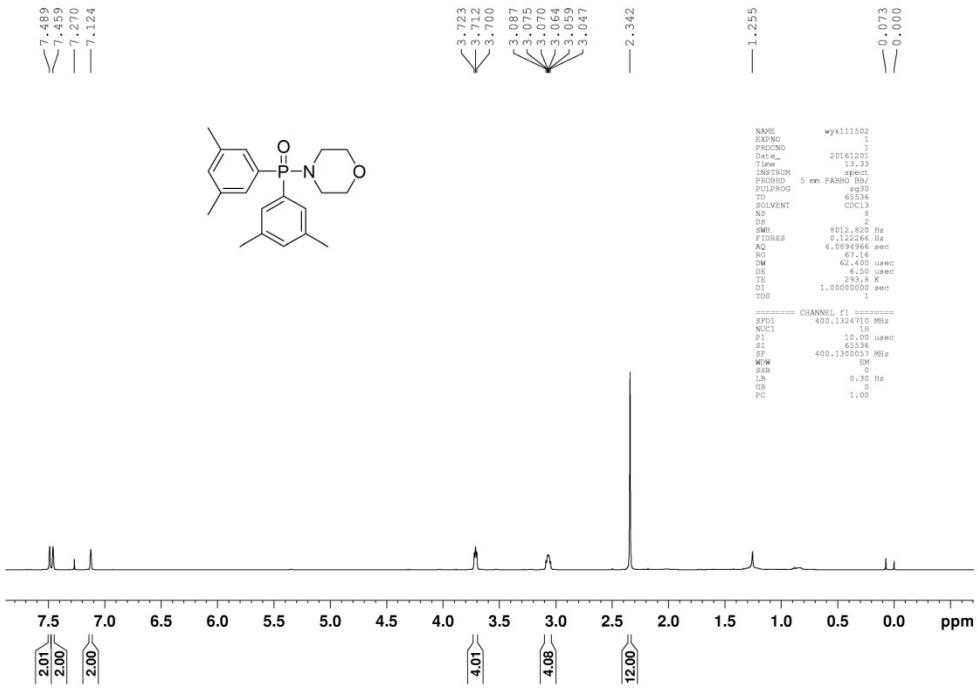
^{13}C NMR of **3ba**



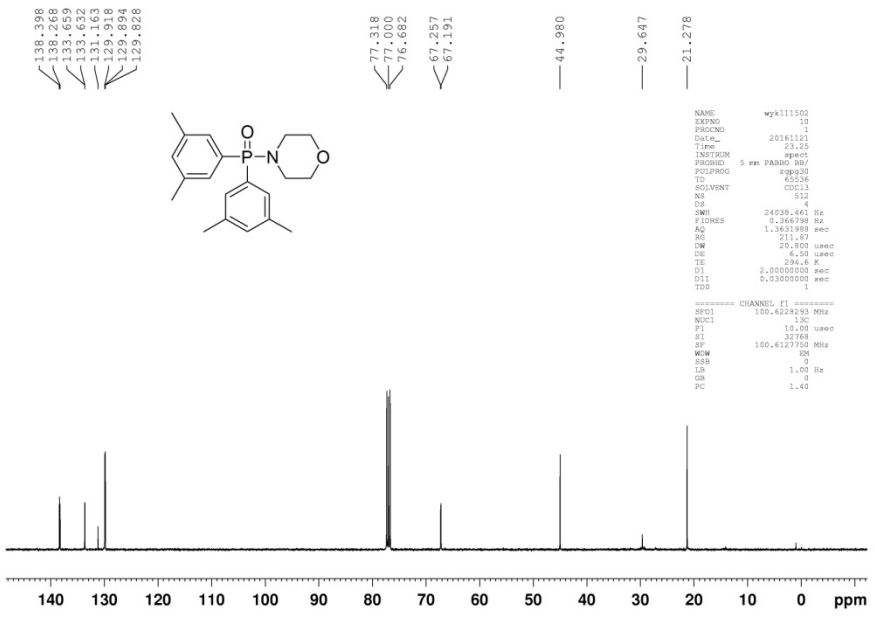
^{31}P NMR of **3ba**



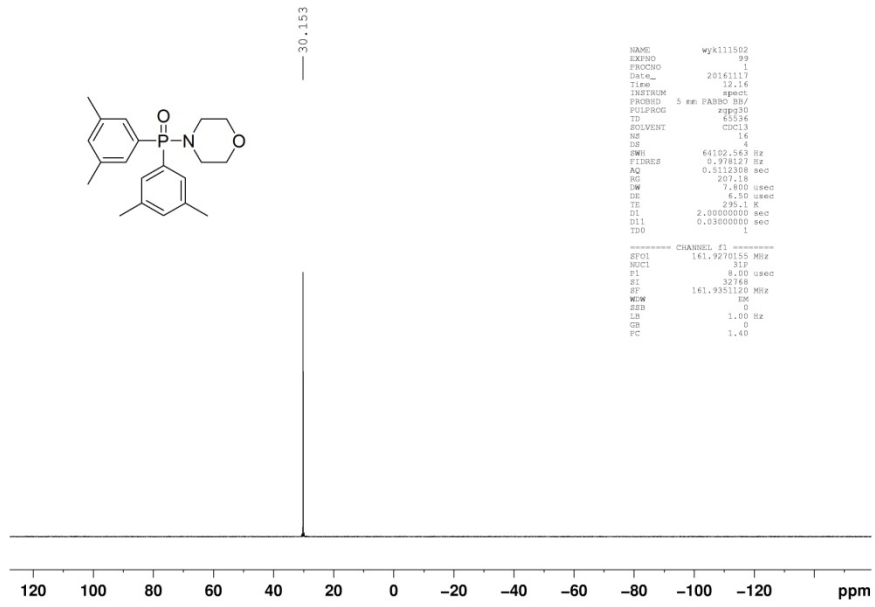
^{1}H NMR of **3ca**



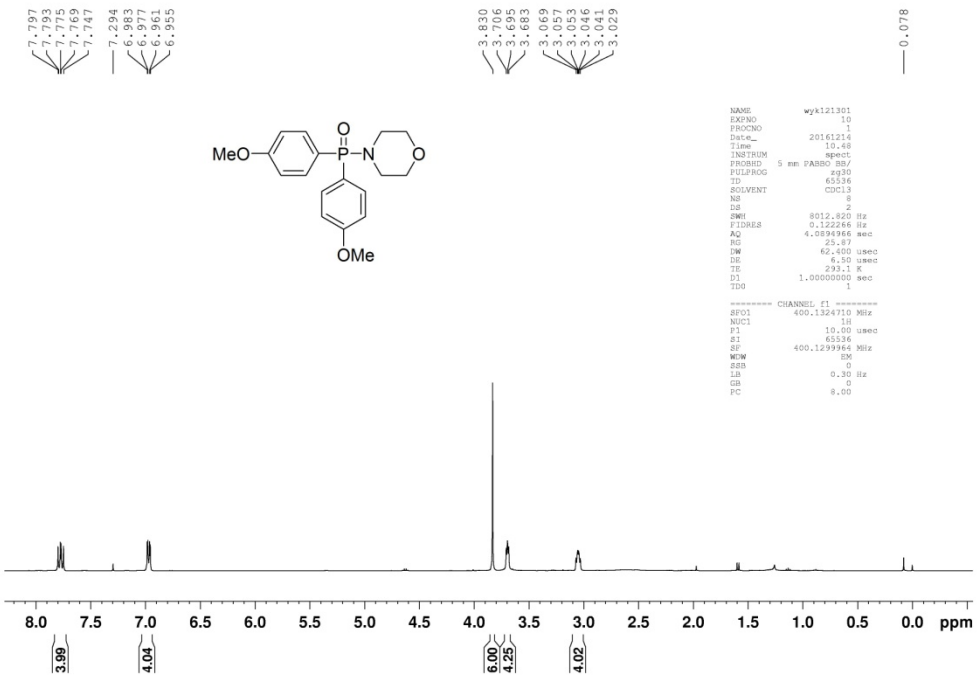
^{13}C NMR of **3ca**



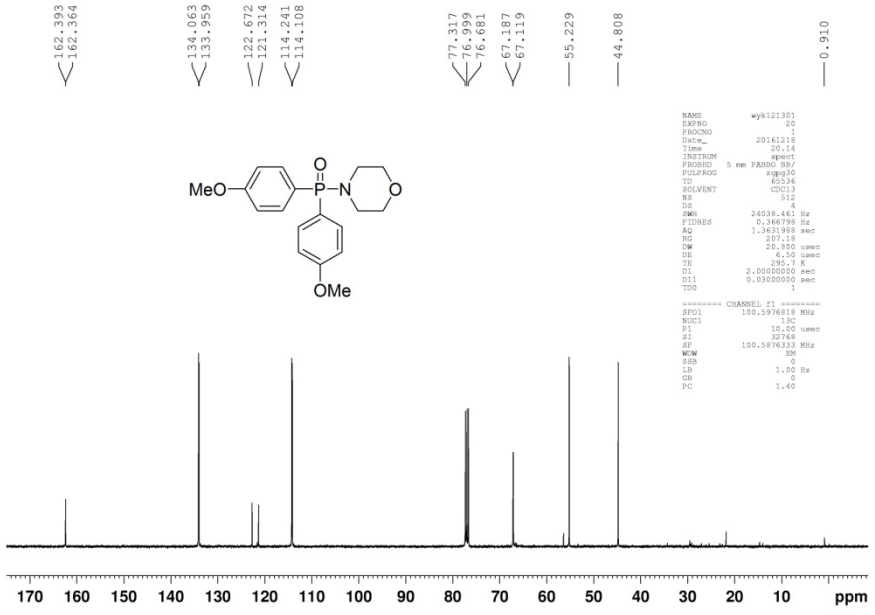
^{31}P NMR of **3ca**



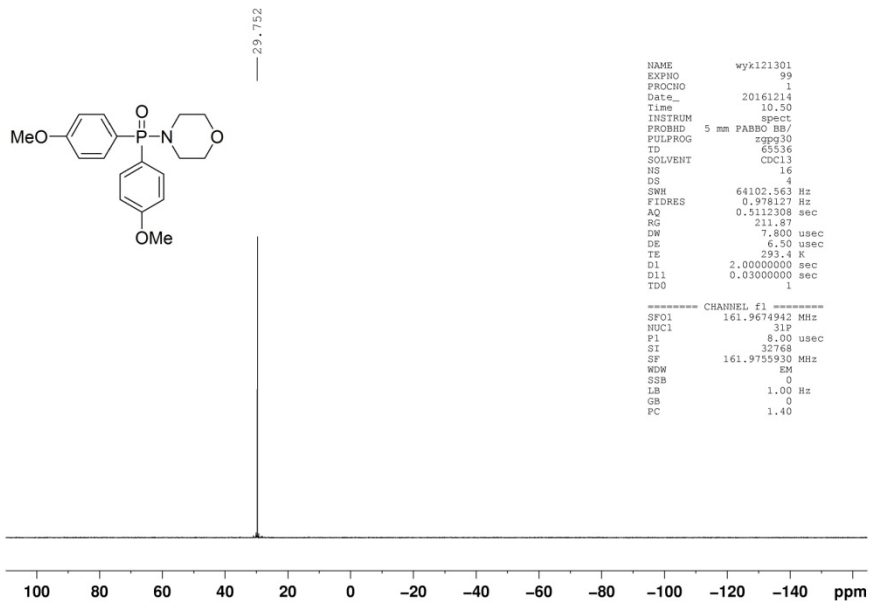
^{1}H NMR of **3da**



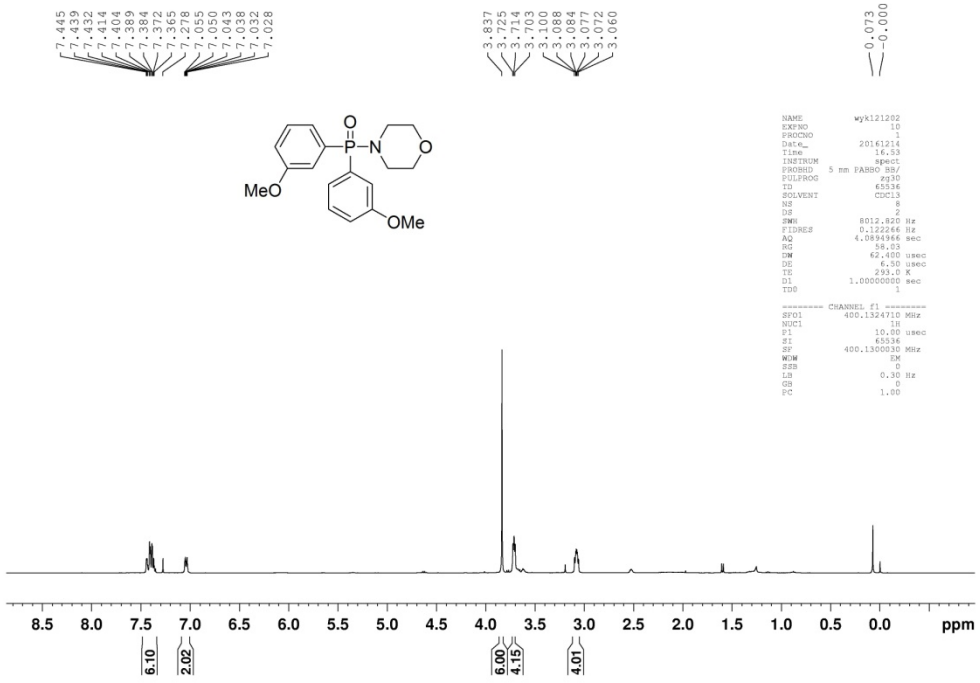
^{13}C NMR of **3da**



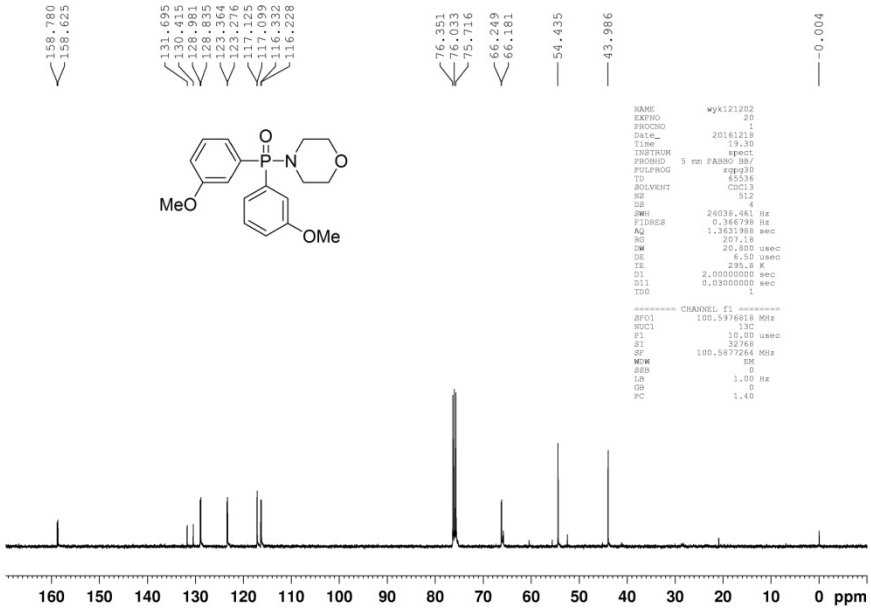
^{31}P NMR of **3da**



^{1}H NMR of **3ea**



^{13}C NMR of **3ea**



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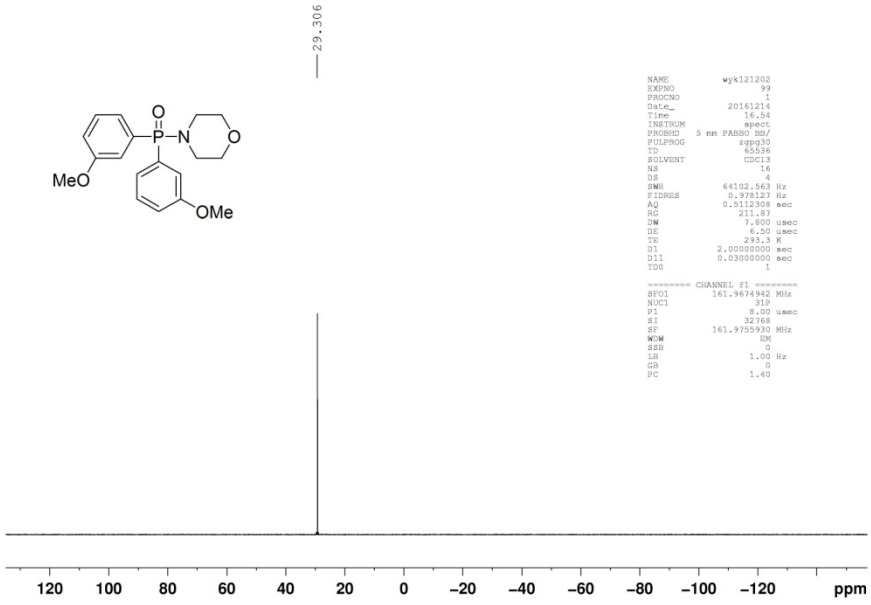
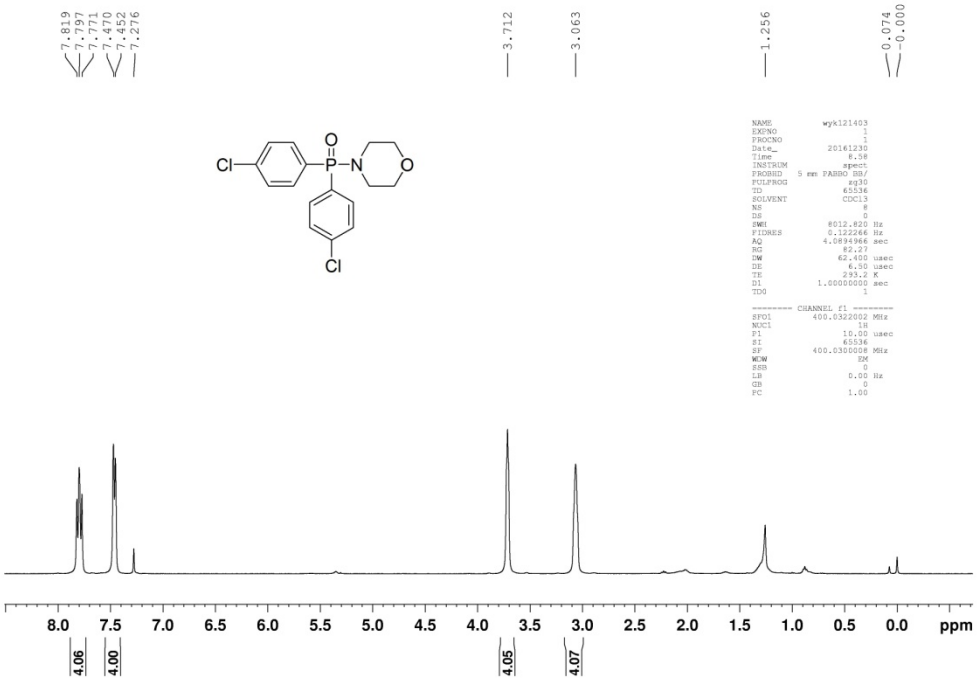
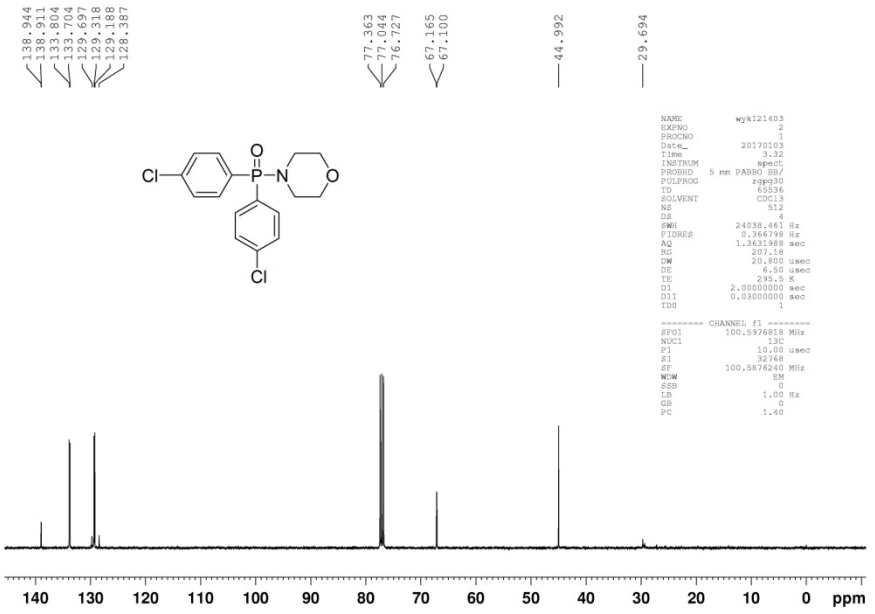


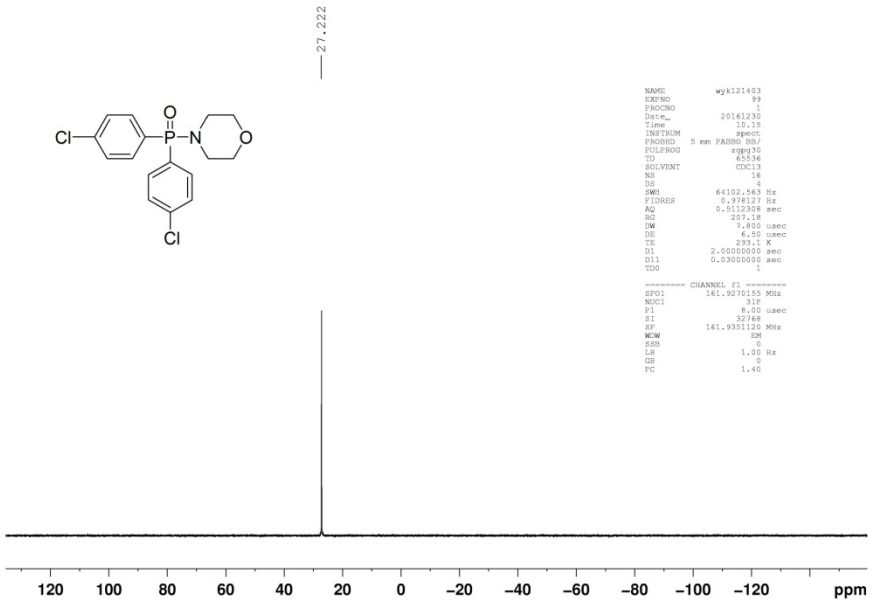
^{1}H NMR of **3fa**



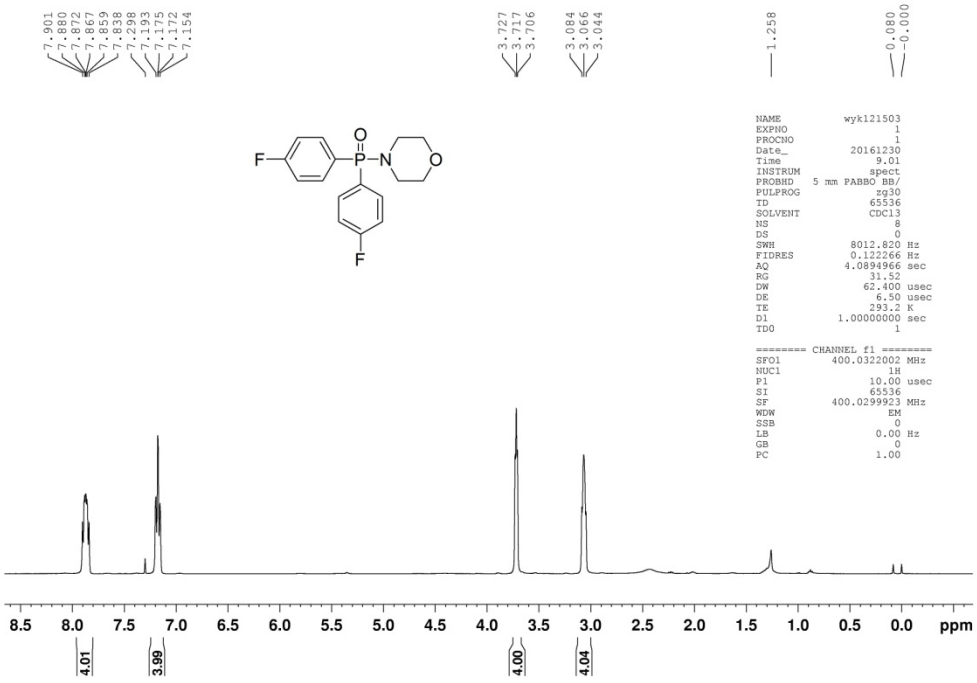
^{13}C NMR of **3fa**



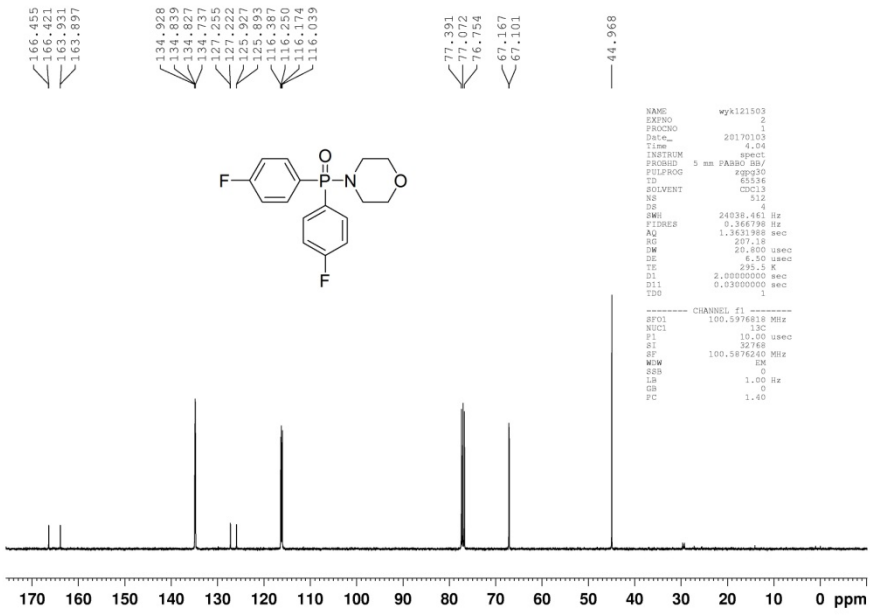
^{31}P NMR of **3fa**



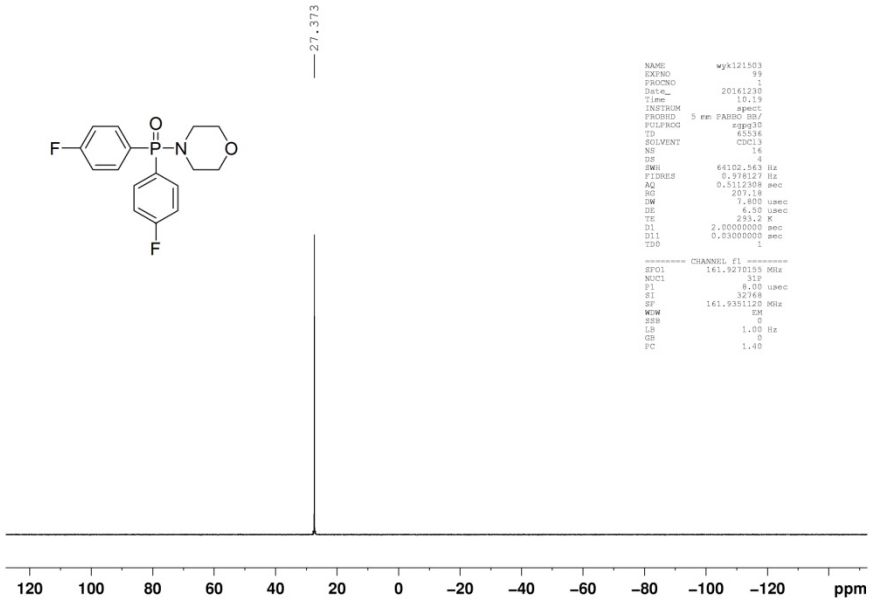
^{1}H NMR of **3ga**



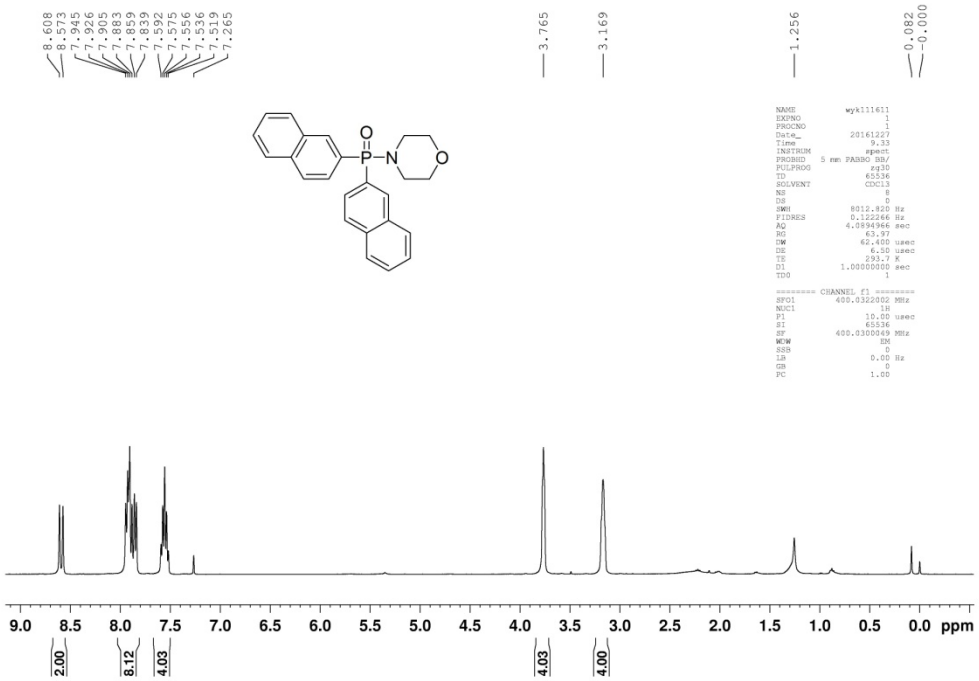
^{13}C NMR of **3ga**



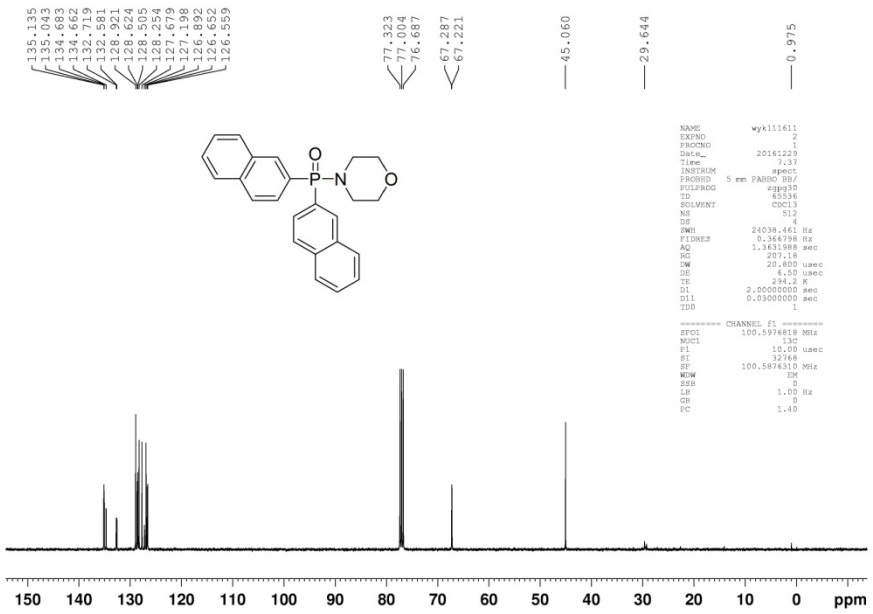
^{31}P NMR of **3ga**



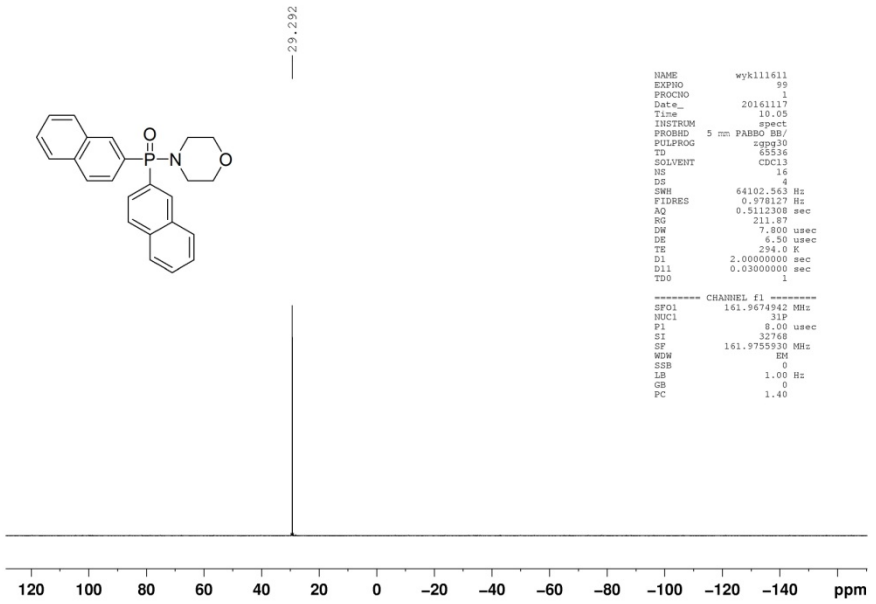
^{1}H NMR of **3ha**



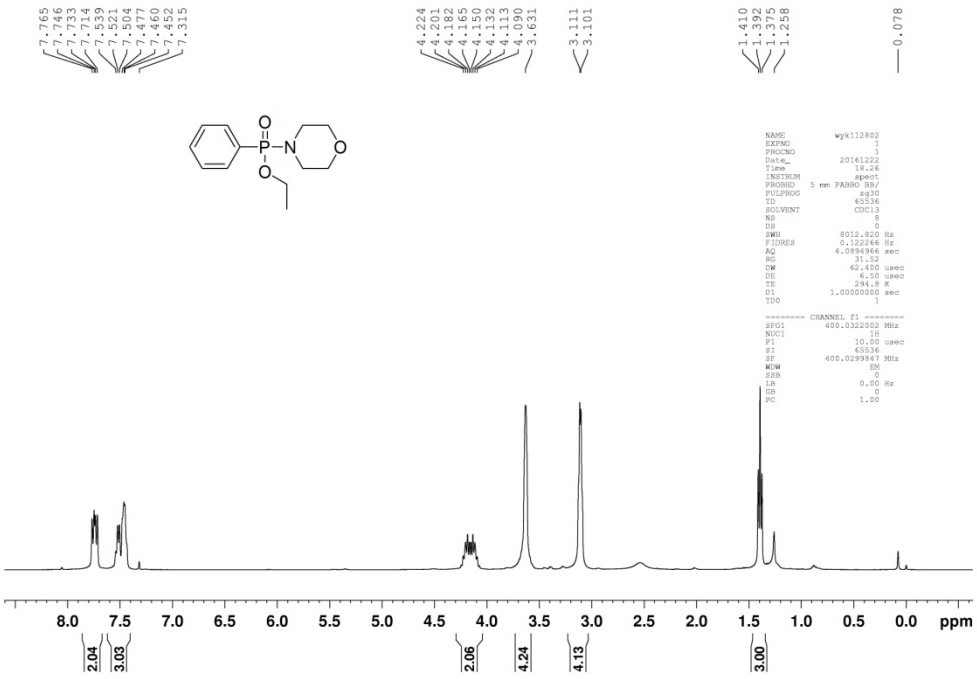
^{13}C NMR of **3ha**



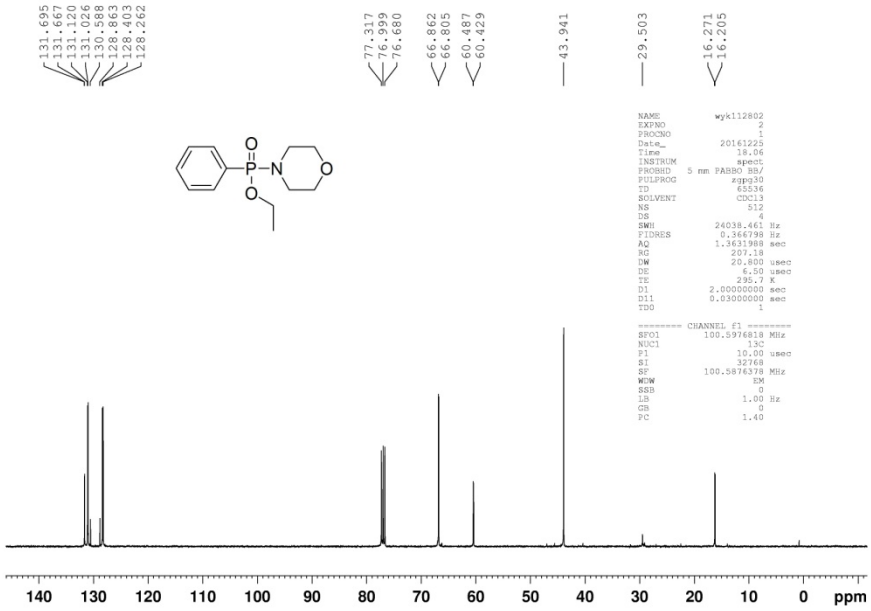
^{31}P NMR of **3ha**



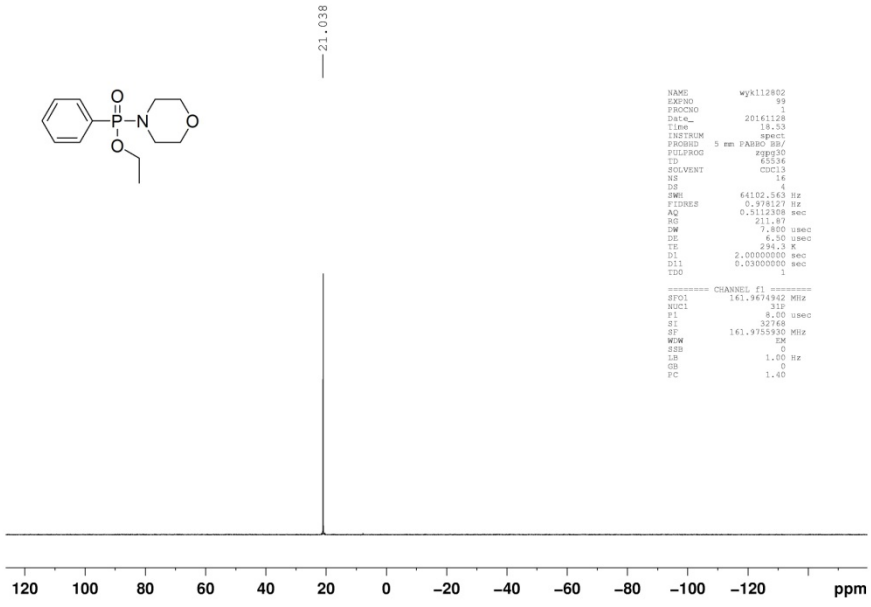
^{1}H NMR of **3ia**



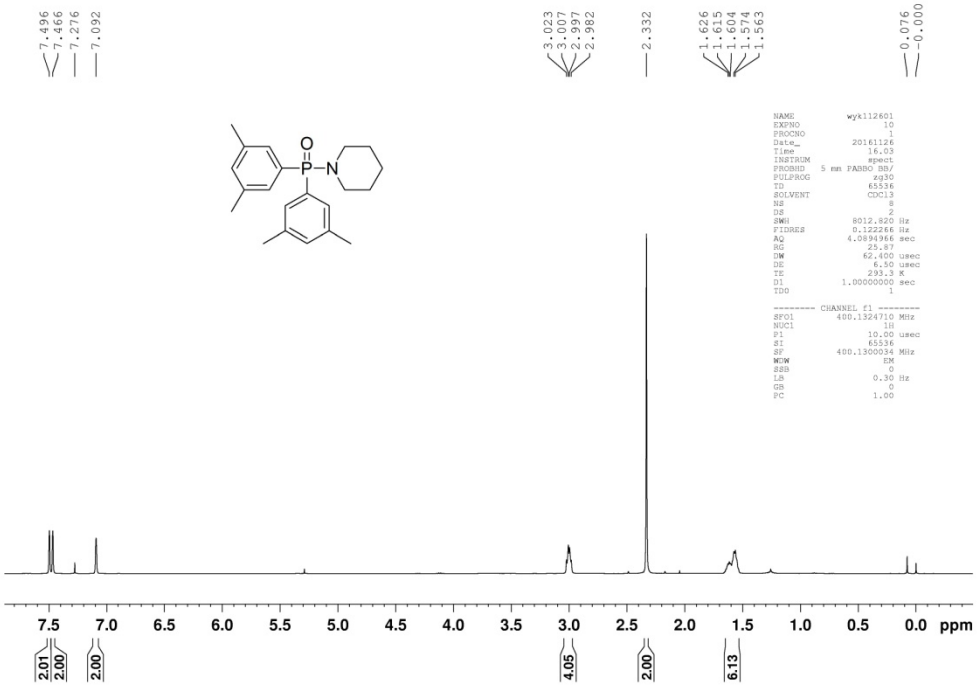
^{13}C NMR of **3ia**



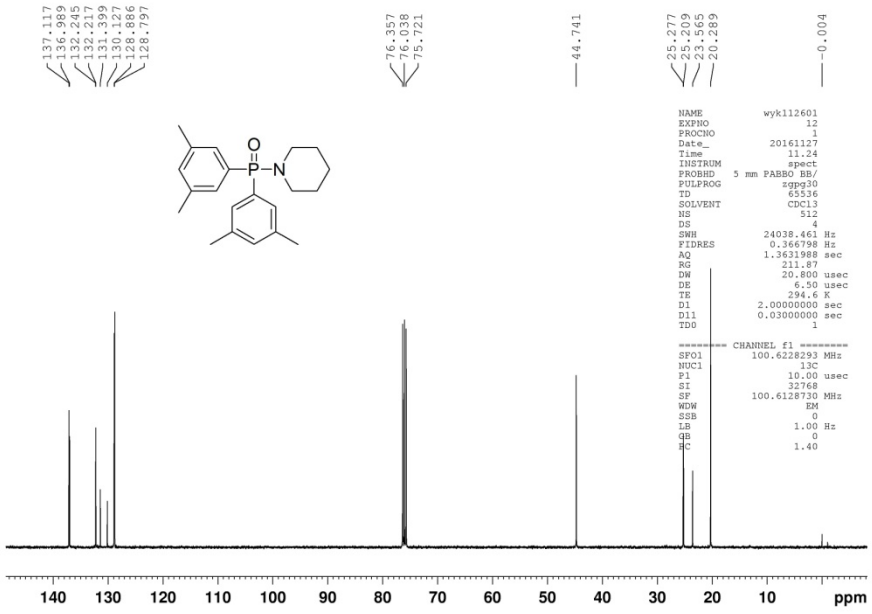
^{31}P NMR of **3ia**



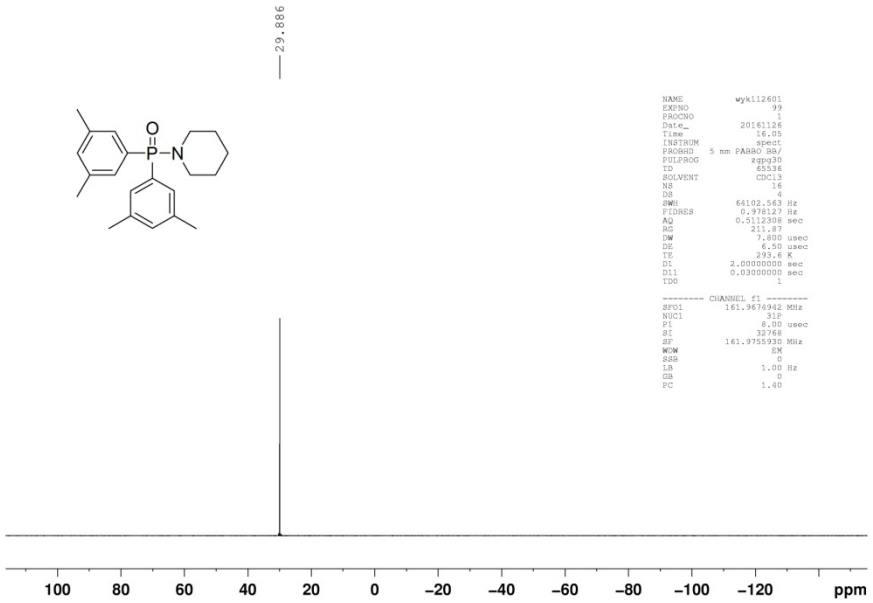
^{1}H NMR of **3cb**



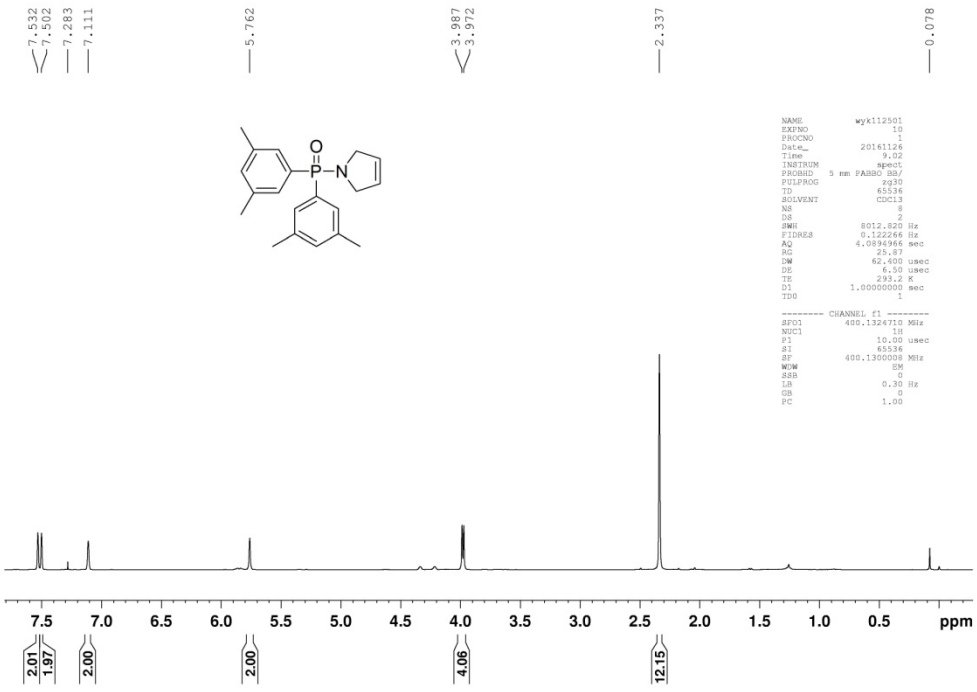
^{13}C NMR of **3cb**



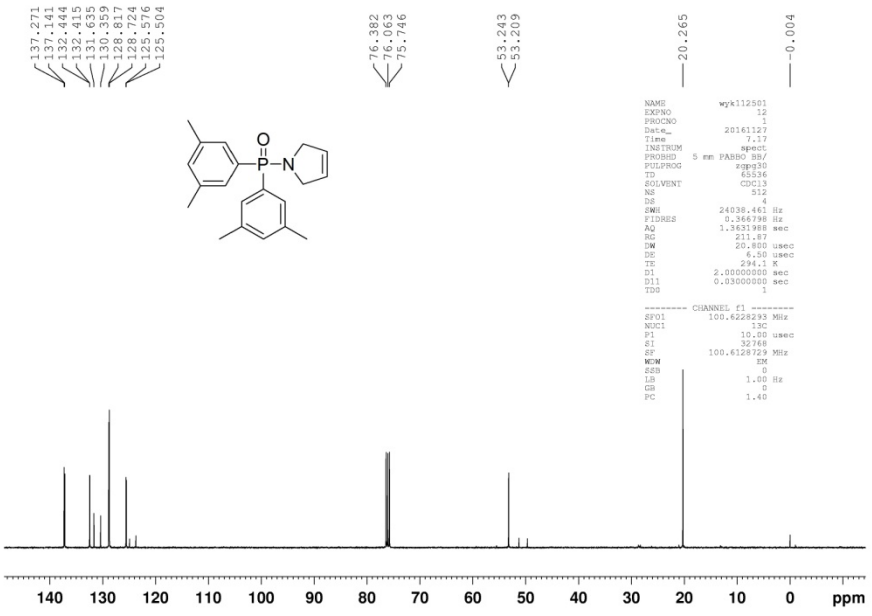
^{31}P NMR of **3cb**



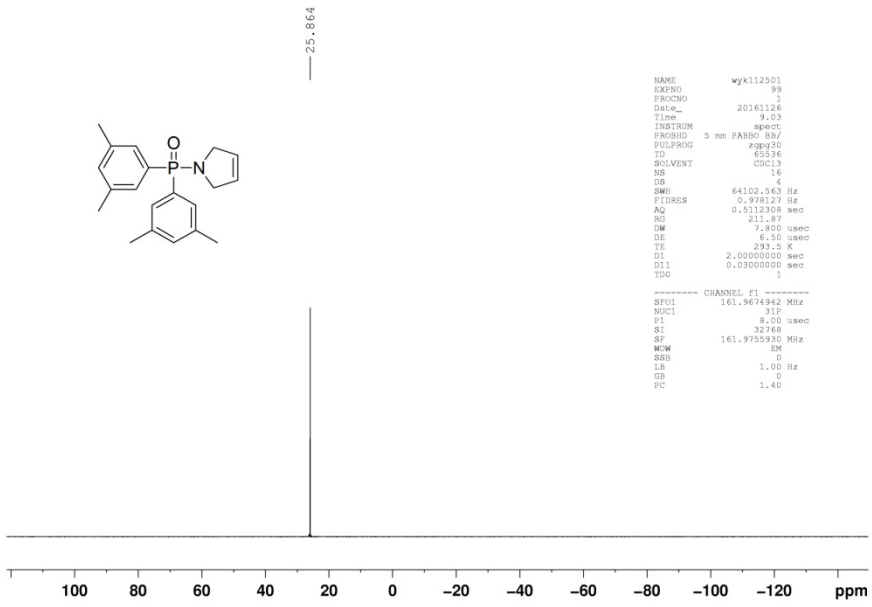
^{1}H NMR of **3ck**



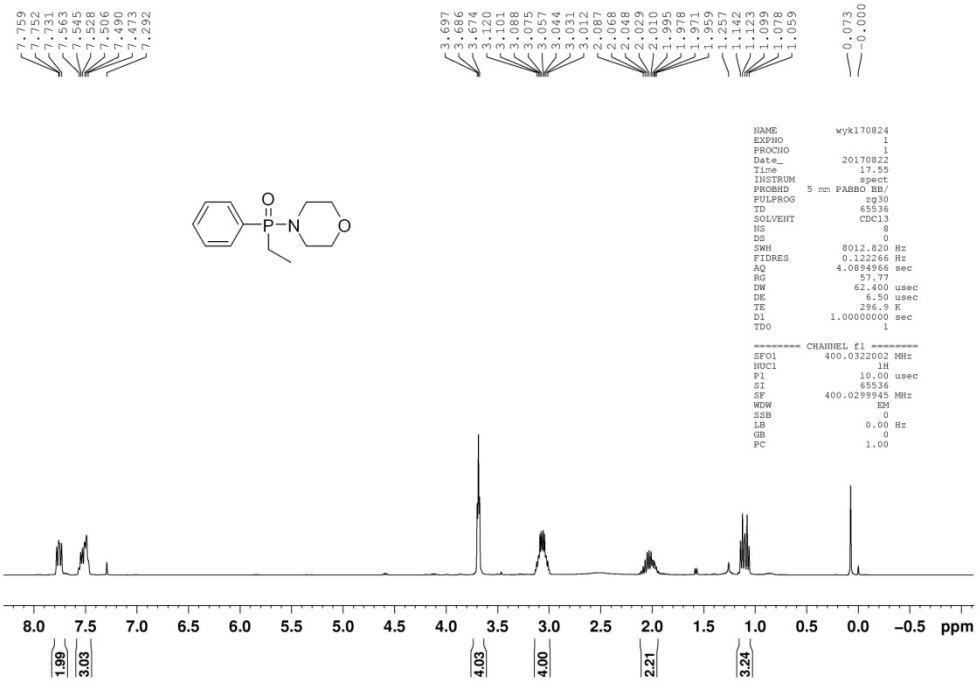
^{13}C NMR of **3ck**



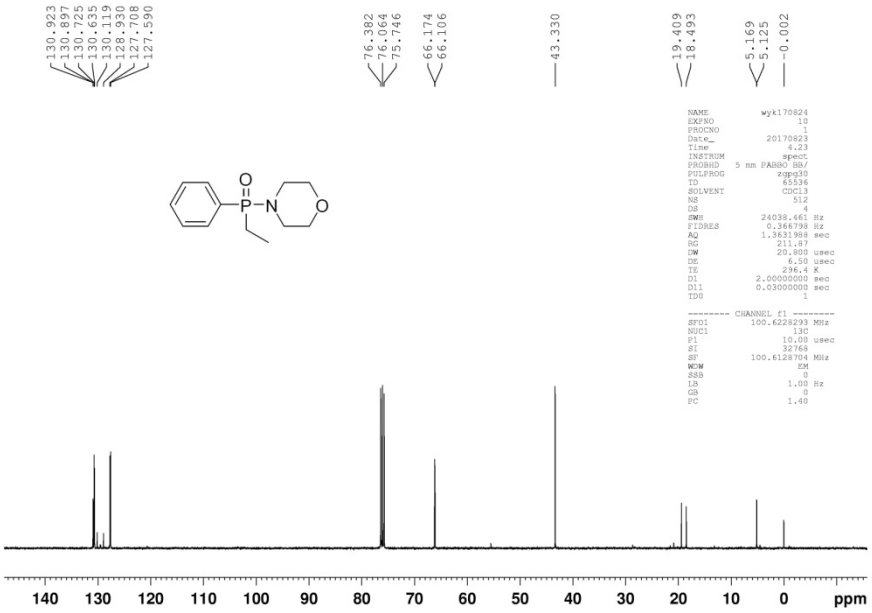
^{31}P NMR of **3ck**



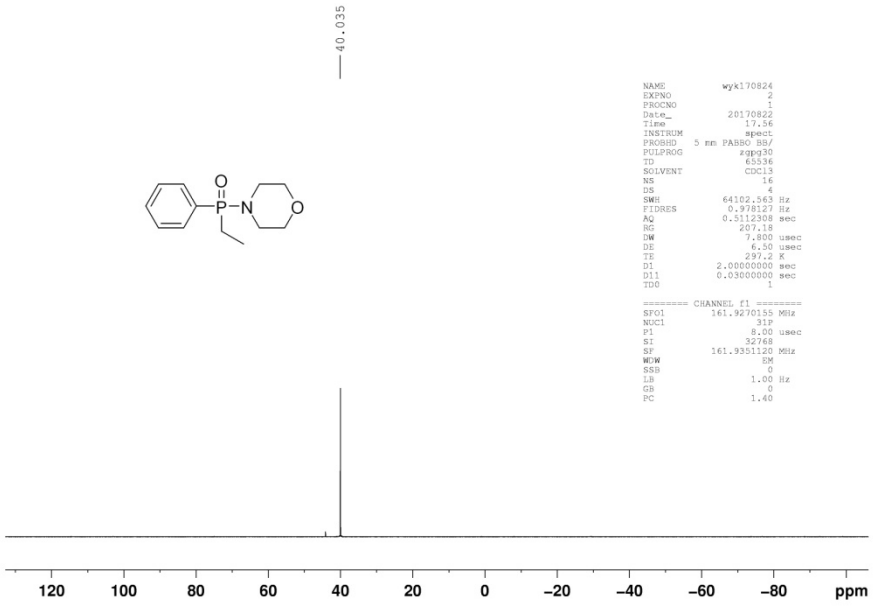
^{1}H NMR of **3ja**



^{13}C NMR of **3ja**

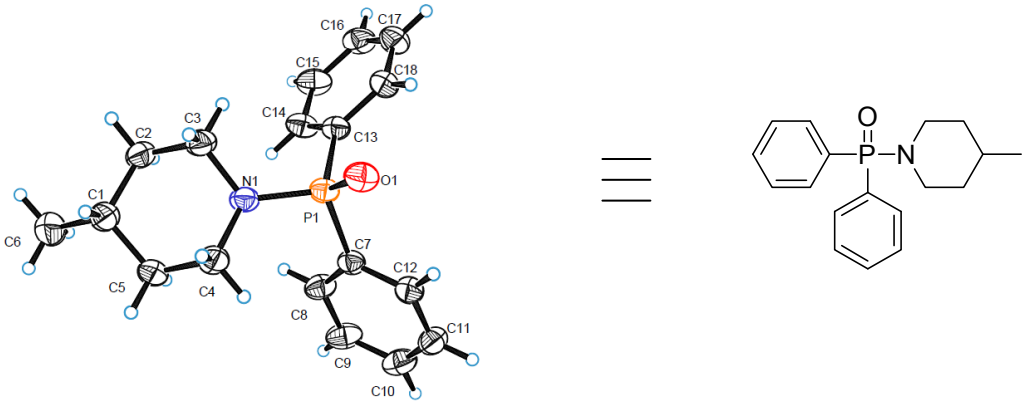


^{31}P NMR of **3ja**



Crystal structure data

A single crystal for X-ray analysis of **3ac** was obtained by recrystallation from ethyl acetate / petroleum ether.



CCDC-1511510

Table 1 Crystal data and structure refinement for 3ac.

Identification code	wyk161008
Empirical formula	$C_{18}H_{22}NOP$
Formula weight	299.33
Temperature/K	292(2)
Crystal system	triclinic
Space group	P-1

a/Å	8.8111(7)
b/Å	8.8406(6)
c/Å	11.0875(8)
α/°	71.938(6)
β/°	83.952(6)
γ/°	85.034(6)
Volume/$Å^3$	815.21(11)
Z	2
$\rho_{calc}g/cm^3$	1.219
μ/mm^{-1}	1.469
F(000)	320.0
Crystal size/mm^3	0.180 × 0.150 × 0.100
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.418 to 142.476
Index ranges	$-10 \leq h \leq 10$, $-9 \leq k \leq 10$, $-13 \leq l \leq 13$
Reflections collected	5167
Independent reflections	3048 [R_{int} = 0.0328, R_{sigma} = 0.0340]
Data/restraints/parameters	3048/0/191
Goodness-of-fit on F^2	1.082
Final R indexes [I>=2σ (I)]	R_1 = 0.0676, wR_2 = 0.1760
Final R indexes [all data]	R_1 = 0.0746, wR_2 = 0.1887
Largest diff. peak/hole / e $Å^{-3}$	0.58/-0.39

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for wyk161008. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
P1	2131.8(6)	6576.8(7)	7435.0(6)	48.8(2)
O1	1020(2)	7244(2)	8248.3(18)	62.2(5)
N1	2161(2)	7343(2)	5869.9(19)	52.2(5)
C13	4072(3)	6717(3)	7767(2)	50.5(5)
C7	1894(2)	4483(3)	7703(2)	49.2(5)
C12	996(3)	3678(3)	8779(2)	57.5(6)
C18	4302(3)	7081(3)	8869(2)	60.5(6)
C4	938(3)	7046(3)	5176(3)	60.4(6)
C5	1573(3)	7039(3)	3860(3)	61.7(7)
C14	5336(3)	6413(3)	7004(3)	62.9(6)
C8	2640(3)	3636(3)	6926(3)	60.5(6)
C3	2785(3)	8915(3)	5216(3)	61.6(6)
C2	3464(3)	8953(3)	3903(3)	64.4(7)
C11	863(3)	2042(3)	9076(3)	67.6(7)
C1	2318(3)	8586(3)	3104(3)	63.5(7)

C10	1598(3)	1222(3)	8309(3)	72.8(8)
C17	5772(4)	7151(4)	9183(3)	71.3(8)
C9	2476(3)	2017(4)	7217(3)	72.9(8)
C16	7012(3)	6851(4)	8423(3)	73.1(8)
C15	6807(3)	6488(4)	7326(3)	73.0(8)
C6	3066(5)	8489(5)	1827(3)	89.3(10)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 3ac. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
P1	40.9(4)	51.5(4)	58.3(4)	-22.0(3)	-5.6(2)	-4.4(2)
O1	51.4(9)	66.7(11)	75.3(12)	-32.6(9)	-1.7(8)	-4.1(8)
N1	48(1)	52.0(11)	59.4(11)	-17.5(9)	-9.6(8)	-9.9(8)
C13	46.9(12)	47.8(12)	60.1(13)	-18.5(10)	-10.3(10)	-5.4(9)
C7	41.0(11)	53.9(12)	55.0(12)	-17.3(10)	-9.3(9)	-5.2(9)
C12	54.4(13)	65.1(15)	55.2(13)	-18.4(11)	-8(1)	-10.2(11)
C18	60.1(14)	66.8(15)	58.5(14)	-20.9(12)	-12.7(11)	-8.0(12)
C4	43.1(12)	68.1(15)	67.8(15)	-13.4(12)	-12.1(10)	-9.0(11)
C5	56.3(14)	68.3(15)	64.1(14)	-18.9(12)	-17.3(11)	-13.0(12)
C14	49.9(13)	69.9(16)	80.5(17)	-38.3(13)	-8.7(12)	-5.0(11)
C8	48.5(13)	59.1(14)	77.3(16)	-27.0(12)	4.9(11)	-10.5(11)
C3	70.4(16)	45.5(12)	71.3(16)	-17.0(11)	-15.3(12)	-7.9(11)
C2	63.9(15)	57.6(14)	68.6(15)	-9.2(12)	-10.3(12)	-17.9(12)
C11	69.1(17)	60.3(15)	67.3(16)	-5.6(12)	-9.1(13)	-17.7(13)
C1	60.7(15)	60.6(14)	66.8(15)	-11.7(12)	-16.0(12)	-6.1(12)
C10	65.0(17)	51.1(14)	99(2)	-14.0(14)	-15.8(15)	-9.4(12)
C17	71.1(18)	79.4(19)	68.7(17)	-21.5(14)	-26.9(14)	-11.4(14)
C9	57.1(15)	60.4(15)	110(2)	-40.3(16)	1.4(15)	-5.2(12)
C16	56.7(16)	70.5(17)	95(2)	-19.4(15)	-31.1(15)	-7.6(13)
C15	44.5(13)	75.6(18)	107(2)	-39.2(17)	-7.8(13)	-1.9(12)
C6	97(2)	103(3)	67.6(18)	-21.0(17)	-4.2(17)	-25(2)

Table 4 Bond Lengths for 3ac.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
P1	O1	1.4712(19)	C4	C5	1.508(4)
P1	N1	1.654(2)	C5	C1	1.527(4)
P1	C13	1.809(2)	C14	C15	1.392(4)
P1	C7	1.809(2)	C8	C9	1.383(4)
N1	C4	1.474(3)	C3	C2	1.506(4)
N1	C3	1.478(3)	C2	C1	1.528(4)
C13	C14	1.385(4)	C11	C10	1.361(5)

C13	C18	1.394(3)	C1	C6	1.522(4)
C7	C12	1.389(3)	C10	C9	1.388(5)
C7	C8	1.390(4)	C17	C16	1.367(4)
C12	C11	1.392(4)	C16	C15	1.383(5)
C18	C17	1.387(4)			

Table 5 Bond Angles for 3ac.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	P1	N1	119.37(11)	C17	C18	C13	120.3(3)
O1	P1	C13	111.30(11)	N1	C4	C5	109.75(19)
N1	P1	C13	103.76(10)	C4	C5	C1	112.4(2)
O1	P1	C7	111.76(11)	C13	C14	C15	120.5(3)
N1	P1	C7	103.40(10)	C9	C8	C7	120.3(3)
C13	P1	C7	106.12(11)	N1	C3	C2	109.4(2)
C4	N1	C3	110.89(19)	C3	C2	C1	112.8(2)
C4	N1	P1	120.60(16)	C10	C11	C12	120.4(3)
C3	N1	P1	120.66(16)	C6	C1	C5	111.2(3)
C14	C13	C18	118.7(2)	C6	C1	C2	111.7(3)
C14	C13	P1	122.86(18)	C5	C1	C2	109.2(2)
C18	C13	P1	118.3(2)	C11	C10	C9	120.1(3)
C12	C7	C8	119.0(2)	C16	C17	C18	120.4(3)
C12	C7	P1	117.93(19)	C8	C9	C10	120.0(3)
C8	C7	P1	122.92(18)	C17	C16	C15	120.1(2)
C7	C12	C11	120.1(3)	C16	C15	C14	119.9(3)

Table 6 Torsion Angles for 3ac.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	P1	N1	C4	-72.9(2)	C3	N1	C4	C5	61.4(3)
C13	P1	N1	C4	162.6(2)	P1	N1	C4	C5	-149.27(19)
C7	P1	N1	C4	51.9(2)	N1	C4	C5	C1	-56.8(3)
O1	P1	N1	C3	73.4(2)	C18	C13	C14	C15	-0.7(4)
C13	P1	N1	C3	-51.1(2)	P1	C13	C14	C15	-177.9(2)
C7	P1	N1	C3	-161.73(19)	C12	C7	C8	C9	0.6(4)
O1	P1	C13	C14	-169.4(2)	P1	C7	C8	C9	176.6(2)
N1	P1	C13	C14	-39.8(2)	C4	N1	C3	C2	-61.3(3)
C7	P1	C13	C14	68.8(2)	P1	N1	C3	C2	149.45(19)
O1	P1	C13	C18	13.4(2)	N1	C3	C2	C1	56.8(3)
N1	P1	C13	C18	143.0(2)	C7	C12	C11	C10	-0.7(4)
C7	P1	C13	C18	-108.4(2)	C4	C5	C1	C6	175.1(2)
O1	P1	C7	C12	-14.0(2)	C4	C5	C1	C2	51.4(3)
N1	P1	C7	C12	-143.65(18)	C3	C2	C1	C6	-175.0(3)
C13	P1	C7	C12	107.48(19)	C3	C2	C1	C5	-51.6(3)

O1	P1	C7	C8	170.0(2)	C12	C11	C10	C9	-0.6(4)
N1	P1	C7	C8	40.3(2)	C13	C18	C17	C16	-0.6(4)
C13	P1	C7	C8	-68.5(2)	C7	C8	C9	C10	-1.9(4)
C8	C7	C12	C11	0.7(3)	C11	C10	C9	C8	1.9(5)
P1	C7	C12	C11	-175.50(19)	C18	C17	C16	C15	0.6(5)
C14	C13	C18	C17	0.6(4)	C17	C16	C15	C14	-0.7(5)
P1	C13	C18	C17	178.0(2)	C13	C14	C15	C16	0.8(5)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 3ac.

Atom	*x*	*y*	*z*	U(eq)
H12	481	4232	9302	69
H18	3466	7278	9396	73
H4A	122	7869	5114	73
H4B	518	6027	5635	73
H5A	752	6879	3400	74
H5B	2324	6153	3930	74
H14	5201	6156	6272	75
H8	3252	4159	6207	73
H3A	3565	9106	5699	74
H3B	1977	9747	5155	74
H2A	4328	8180	3978	77
H2B	3841	9999	3469	77
H11	269	1507	9803	81
H1	1518	9450	2943	76
H10	1513	128	8518	87
H17	5915	7405	9915	86
H9	2953	1461	6681	87
H16	7994	6890	8643	88
H15	7651	6295	6804	88
H6A	3871	7664	1965	134
H6B	3482	9491	1356	134
H6C	2316	8249	1354	134

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF

Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) shelxl

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary

Please wait while processing .... Interpreting this report

Structure factor report

Datablock: shelxl

Bond precision: C-C = 0.0041 A Wavelength=1.54184

Cell: a=8.8111(7) b=8.8406(6) c=11.0875(8)
alpha=71.938(6) beta=83.952(6) gamma=85.034(6)

Temperature: 292 K

	Calculated	Reported
Volume	815.21(11)	815.21(11)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C_{18} H_{22} N O P	?
Sum formula	C_{18} H_{22} N O P	C_{18} H_{22} N O P
Mr	299.34	299.33
Dx,g cm-3	1.219	1.219
Z	2	2
Mu (mm-1)	1.469	1.469
F000	320.0	320.0
F000'	321.36	
h,k,lmax	10,10,13	10,10,13
Nref	3162	3048
Tmin,Tmax	0.768,0.863	0.427,1.000
Tmin'	0.768	

Correction method= # Reported T Limits: Tmin=0.427 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.964 Theta(max)= 71.238

R(reflections)= 0.0676(2605) wR2(reflections)= 0.1887(3048)

S = 1.082 Npar= 191

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00412 Ang.
PLAT911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600 36 Report

---

Alert level G

PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.12 Report
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.006 Degree
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 79 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 9 Note

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
4 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
2 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PLAT340_shelxl
;
PROBLEM: Low Bond Precision on   C-C Bonds .....      0.00412 Ang.
RESPONSE: ...
;
_vrf_PLAT911_shelxl
;
PROBLEM: Missing # FCF Refl Between THmin & STh/L=  0.600          36 Report
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or

refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

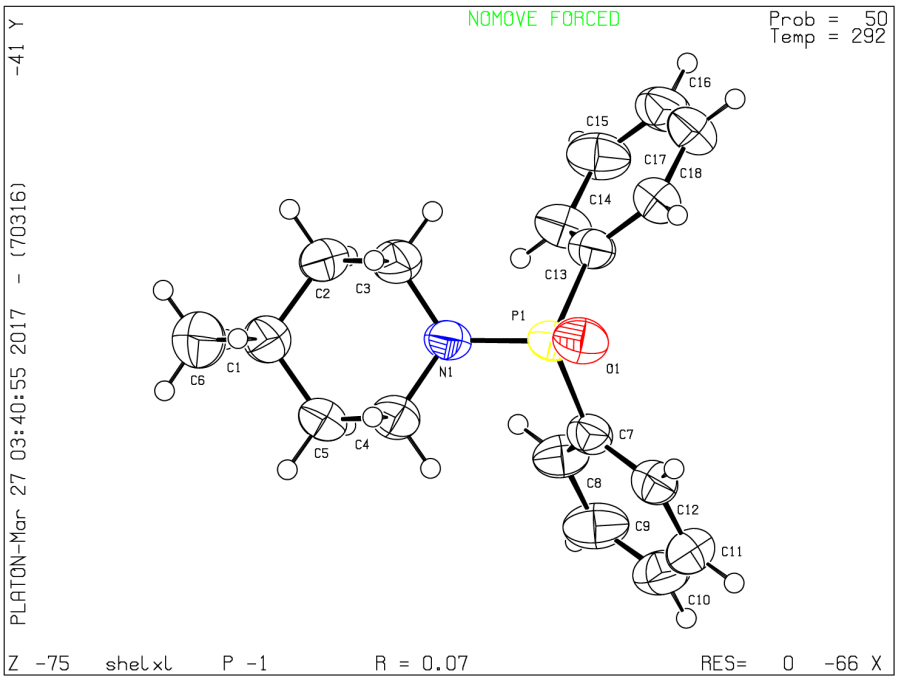
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*,*Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 26/02/2017; check.def file version of 21/02/2017

Datablock shelxl- ellipsoid plot



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