

Supporting Information

Synthesis of Enol Phosphates Directly from Ketones via a Modified
One-Pot Perkow Reaction

Huichuang Guo, Yulong Zhang, Zhenya Li, Peichao Zhao, Ning Li,
Enxue Shi*

State Key Laboratory of NBC Protection for Civilian, Beijing 102205, P. R. China

E-mail: exshi@sina.com

Contents

1. General information.....	3
2. General procedure for the synthesis of 3.....	3
3. Characterization data of compounds 3aa - 3df.....	3
4. Copies of ^1H , ^{13}C , and ^{31}P NMR spectra.....	16

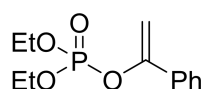
1. General information

All reactions were performed in oven-dried glassware with magnetic stirring. Unless otherwise stated, all commercially available reagents were used without further purification. ^1H , ^{13}C and ^{31}P NMR spectra were recorded at ambient temperature on Bruker 600 instruments. All spectra were referenced to CDCl_3 (^1H δ 7.26 ppm and ^{13}C NMR δ 77.00 ppm). Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, qd = quartet of doublets, m = multiplet), coupling constants (Hz) and integration. High-resolution mass spectra (HRMS) were obtained on an Agilent 6545 Q-TOF HPLC and mass spectrometry.

2. General procedure for the synthesis of 3

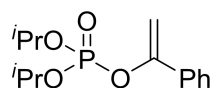
A mixture of ketone **4** (1.1 eq) and HTIB (1 eq) were stirred at 60 °C for about 1h and then added 4Å molecular sieve and phosphite **2** (1 eq) in DCM at room temperature, and further react about 2h. After the reaction was finished, column chromatography (n-hexane: ethyl acetate = 1:1) separation gives the target product **3**.

3. Characterization data of compounds 3aa - 3df



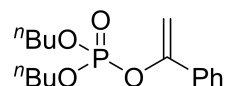
Diethyl (1-phenylvinyl) phosphate (3aa)

Oily liquid, yield 95%, 243 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.59 - 7.58 (m, 2H), 7.41 - 7.27 (m, 3H), 5.29 - 5.28 (m, 1H), 5.23 - 5.22 (m, 1H), 4.36 - 4.02 (m, 4H), 1.34 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.29 (d, $J = 7.8$ Hz), 134.29 (d, $J = 6.9$ Hz), 129.03, 128.34, 125.17, 97.23 (d, $J = 3.6$ Hz), 64.48 (d, $J = 6.0$ Hz), 16.06 (d, $J = 6.8$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.34. HRMS (m/z) calcd for $\text{C}_{12}\text{H}_{17}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 257.0937, found 257.0936.



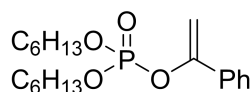
Diisopropyl (1-phenylvinyl) phosphate (3ab)

Oily liquid, yield 90%, 256 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.67 - 7.52 (m, 2H), 7.38 - 7.32 (m, 3H), 5.27 - 5.26 (m, 1H), 5.25 - 5.23 (m, 1H), 4.78 - 4.71 (m, 2H), 1.36 (d, $J = 6.2$ Hz, 6H), 1.32 (d, $J = 6.2$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.22 (d, $J = 8.0$ Hz), 134.38 (d, $J = 7.3$ Hz), 128.71, 128.09, 125.00, 96.74 (d, $J = 3.5$ Hz), 73.11 (d, $J = 6.1$ Hz), 23.46 (d, $J = 6.1$ Hz), 23.32 (d, $J = 6.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -8.07. HRMS (m/z) calcd for $\text{C}_{14}\text{H}_{21}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 285.1250, found 285.1252.



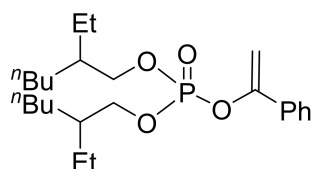
Dibutyl (1-phenylvinyl) phosphate (3ac)

Oily liquid, yield 93%, 290 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.62 - 7.54 (m, 2H), 7.40 - 7.27 (m, 3H), 5.28 - 5.28 (m, 1H), 5.23 - 5.22 (m, 1H), 4.19 - 4.00 (m, 4H), 1.73 - 1.56 (m, 4H), 1.46 - 1.30 (m, 4H), 0.91 (t, $J = 7.4$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.32 (d, $J = 7.9$ Hz), 134.34 (d, $J = 6.8$ Hz), 129.01, 128.32, 125.18, 97.23 (d, $J = 3.6$ Hz), 68.17 (d, $J = 7.1$ Hz), 32.19 (d, $J = 7.0$ Hz), 18.60, 13.51. ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.10. HRMS (m/z) calcd for $\text{C}_{16}\text{H}_{25}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 313.1563, found 313.1563.



Dihexyl (1-phenylvinyl) phosphate (3ad)

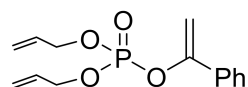
Oily liquid, yield 92%, 339 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.62 - 7.55 (m, 2H), 7.38 - 7.29 (m, 3H), 5.29 - 5.28 (m, 1H), 5.23 - 5.22 (m, 1H), 4.15 - 4.09 (m, 4H), 1.69 - 1.65 (m, 4H), 1.36 - 1.33 (m, 4H), 1.29 - 1.25 (m, 8H), 0.87 (t, $J = 7.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.31 (d, $J = 7.8$ Hz), 134.33 (d, $J = 6.9$ Hz), 129.01, 128.32, 125.18, 97.25 (d, $J = 3.6$ Hz), 68.52 (d, $J = 6.3$ Hz), 31.25, 30.17 (d, $J = 6.9$ Hz), 25.05, 22.48, 13.94. ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.14. HRMS (m/z) calcd for $\text{C}_{20}\text{H}_{33}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 369.2189, found 369.2188.



Bis(2-ethylhexyl) (1-phenylvinyl) phosphate (3ae)

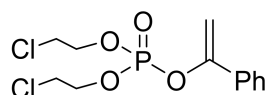
Oily liquid, yield 92%, 390 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.64 - 7.54 (m, 2H), 7.39 - 7.30 (m, 3H), 5.28 - 5.28 (m, 1H), 5.23 - 5.22 (m, 1H), 4.09 - 3.98 (m, 4H), 1.56 (dt, $J = 12.0, 5.9$ Hz, 2H), 1.39 - 1.32 (m, 6H), 1.30 - 1.24 (m, 10H), 0.89 - 0.84

(m, 12H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.34 (d, $J = 7.9$ Hz), 134.37 (d, $J = 6.8$ Hz), 128.99, 128.31, 125.19, 97.26 (d, $J = 3.5$ Hz), 70.41 (d, $J = 6.6$ Hz), 65.30, 41.97, 40.04, 29.84, 28.83, 23.21, 22.92, 14.00, 10.88. ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.87. HRMS (m/z) calcd for $\text{C}_{24}\text{H}_{41}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 425.2815, found 425.2819.



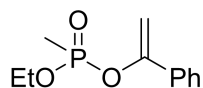
Diallyl (1-phenylvinyl) phosphate (3af)

Oily liquid, yield 93%, 260 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.62 - 7.52 (m, 2H), 7.39 - 7.31 (m, 3H), 5.93 (m, 2H), 5.37 (q, $J = 1.5$ Hz, 1H), 5.35 (q, $J = 1.5$ Hz, 1H), 5.30 (t, $J = 2.8$ Hz, 1H), 5.26 (d, $J = 1.2$ Hz, 1H), 5.24 (t, $J = 2.5$ Hz, 2H), 4.65 - 4.61 (m, 4H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.24 (d, $J = 7.9$ Hz), 134.10 (d, $J = 6.8$ Hz), 132.07 (d, $J = 7.0$ Hz), 129.12, 128.37, 125.21, 118.51, 97.59 (d, $J = 3.2$ Hz), 68.77 (d, $J = 5.6$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.23. HRMS (m/z) calcd for $\text{C}_{14}\text{H}_{17}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 281.0937, found 281.0935.



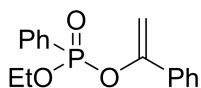
Bis(2-chloroethyl) (1-phenylvinyl) phosphate (3ag)

Oily liquid, yield 91%, 295 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.64 - 7.48 (m, 2H), 7.46 - 7.31 (m, 3H), 5.34 - 5.33 (m, 1H), 5.27 - 5.26 (m, 1H), 4.37 (m, 4H), 3.70 (m, 4H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.15 (d, $J = 7.9$ Hz), 133.74 (d, $J = 6.5$ Hz), 129.31, 128.47, 125.18, 98.12 (d, $J = 3.8$ Hz), 67.73 (d, $J = 5.5$ Hz), 42.17 (d, $J = 7.8$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.96. HRMS (m/z) calcd for $\text{C}_{12}\text{H}_{15}\text{Cl}_2\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 325.0158, found 325.0159.



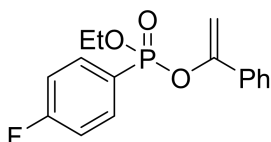
Ethyl (1-phenylvinyl) methylphosphonate (3ah)

Oily liquid, yield 86%, 195 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.62 - 7.50 (m, 2H), 7.39 - 7.30 (m, 3H), 5.29 - 5.28 (m, 1H), 5.22 - 5.21 (m, 1H), 4.30 - 4.18 (m, 1H), 4.18 - 4.07 (m, 1H), 1.62 (d, $J = 17.6$ Hz, 3H), 1.31 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.15 (d, $J = 8.9$ Hz), 134.54 (d, $J = 5.2$ Hz), 129.05, 128.38, 125.16, 97.65 (d, $J = 4.3$ Hz), 62.33 (d, $J = 6.7$ Hz), 16.28 (d, $J = 6.3$ Hz), 11.69, 10.73. ^{31}P NMR (243 MHz, Chloroform-*d*) δ 27.16. HRMS (m/z) calcd for $\text{C}_{11}\text{H}_{15}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$ 227.0832, found 227.0832.



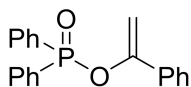
Ethyl (1-phenylvinyl) phenylphosphonate (3ai)

Oily liquid, yield 97%, 279 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.94 - 7.81 (m, 2H), 7.58 - 7.53 (m, 3H), 7.48 - 7.45 (m, 2H), 7.34 - 7.30 (m, 3H), 5.23 - 5.22 (m, 1H), 5.19 - 5.18 (m, 1H), 4.29 - 4.19 (m, 2H), 1.35 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.10 (d, $J = 8.2$ Hz), 134.58 (d, $J = 5.7$ Hz), 132.65 (d, $J = 3.0$ Hz), 131.83 (d, $J = 10.2$ Hz), 128.91, 128.50, 128.40, 128.27, 127.14, 125.18, 97.65 (d, $J = 4.2$ Hz), 62.89 (d, $J = 5.9$ Hz), 16.29 (d, $J = 6.4$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ 15.28. HRMS (m/z) calcd for $\text{C}_{16}\text{H}_{17}\text{O}_3\text{P}$ [$\text{M}+\text{H}$] $^+$ 289.0988, found 289.0989.



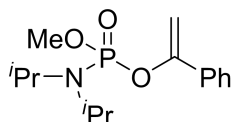
Ethyl (1-phenylvinyl) (4-fluorophenyl)phosphonate (3aj)

Oily liquid, yield 95%, 290 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.96 - 7.81 (m, 2H), 7.57 - 7.46 (m, 2H), 7.37 - 7.29 (m, 3H), 7.16 - 7.12 (m, 2H), 5.23 - 5.22 (m, 1H), 5.19 - 5.14 (m, 1H), 4.29 - 4.21 (m, 2H), 1.35 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 165.47 (dd, $J = 254.2, 4.0$ Hz), 152.09 (d, $J = 8.2$ Hz), 134.48 (dd, $J = 11.7, 8.9$ Hz), 134.43, 129.00, 128.32, 125.16, 123.81 (dd, $J = 196.3, 4.0$ Hz), 115.87 (dd, $J = 21.5, 16.7$ Hz), 97.81 (d, $J = 4.3$ Hz), 63.03 (d, $J = 5.9$ Hz), 16.29 (d, $J = 6.3$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ 14.23. HRMS (m/z) calcd for $\text{C}_{16}\text{H}_{16}\text{FO}_3\text{P}$ [$\text{M}+\text{H}$] $^+$ 307.0894, found 307.0892.



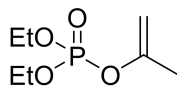
1-phenylvinyl diphenylphosphinate (3ak)

White solid, yield 94%, 301 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.92 - 7.85 (m, 4H), 7.58 (dd, $J = 7.7, 1.8$ Hz, 2H), 7.55 - 7.51 (m, 2H), 7.45 (td, $J = 7.6, 3.7$ Hz, 4H), 7.38 - 7.31 (m, 3H), 5.29 - 5.21 (m, 1H), 5.20 - 5.05 (m, 1H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.33 (d, $J = 8.8$ Hz), 134.91 (d, $J = 5.7$ Hz), 132.35 (d, $J = 2.8$ Hz), 131.63 (d, $J = 10.4$ Hz), 131.51, 130.59, 128.92, 128.60, 128.51, 128.36, 125.11, 97.75 (d, $J = 5.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ 30.02. HRMS (m/z) calcd for $\text{C}_{20}\text{H}_{17}\text{O}_2\text{P}$ [$\text{M}+\text{H}$] $^+$ 321.1039, found 321.1041.



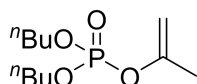
Methyl (1-phenylvinyl) diisopropylphosphoramidate (3al)

Oily liquid, yield 83%, 247 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.64 - 7.52 (m, 2H), 7.39 - 7.28 (m, 3H), 5.36 - 5.30 (m, 1H), 5.23 - 5.22 (m, 1H), 3.76 (d, $J = 11.5$ Hz, 3H), 3.56 - 3.38 (m, 2H), 1.27 (d, $J = 6.8$ Hz, 6H), 1.23 (d, $J = 6.8$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.39 (d, $J = 7.7$ Hz), 135.33 (d, $J = 7.4$ Hz), 128.65, 128.26, 125.08, 96.34 (d, $J = 3.7$ Hz), 52.88 (d, $J = 5.8$ Hz), 46.27 (d, $J = 5.1$ Hz), 22.51 (d, $J = 35.2$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ 4.47. HRMS (m/z) calcd for $\text{C}_{15}\text{H}_{24}\text{NO}_3\text{P}$ $[\text{M}+\text{H}]^+$ 298.1567, found 298.1566.



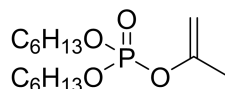
Diethyl prop-1-en-2-yl phosphate (3ba)

Oily liquid, yield 86%, 167 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 4.75 - 4.74 (m, 1H), 4.48 - 4.47 (m, 1H), 4.17 - 4.12 (m, 4H), 1.93 (s, 3H), 1.34 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.11 (d, $J = 8.5$ Hz), 97.98 (d, $J = 4.9$ Hz), 64.16 (d, $J = 6.0$ Hz), 20.68 (d, $J = 5.0$ Hz), 16.02 (d, $J = 6.8$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.78. HRMS (m/z) calcd for $\text{C}_7\text{H}_{15}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 195.0781, found 195.0786.



Dibutyl prop-1-en-2-yl phosphate (3bb)

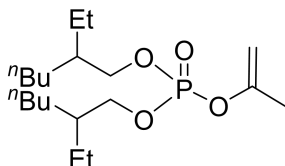
Oily liquid, yield 84%, 210 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 4.75 - 4.75 (m, 1H), 4.48 - 4.48 (m, 1H), 4.08 (q, $J = 6.7$ Hz, 4H), 1.93 (s, 3H), 1.71 - 1.59 (m, 4H), 1.42 - 1.32 (m, 4H), 0.93 (t, $J = 7.4$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.14 (d, $J = 8.5$ Hz), 97.96 (d, $J = 4.9$ Hz), 67.89 (d, $J = 6.3$ Hz), 32.19 (d, $J = 6.9$ Hz), 20.70 (d, $J = 5.1$ Hz), 18.62, 13.52. ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.56. HRMS (m/z) calcd for $\text{C}_{11}\text{H}_{23}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 251.1407, found 251.1409.



Dihexyl prop-1-en-2-yl phosphate (3bc)

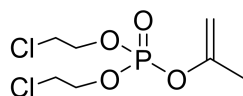
Oily liquid, yield 85%, 260 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 4.75 - 4.75 (m, 1H), 4.48 - 4.48 (m, 1H), 4.09 - 4.05 (m, 4H), 1.93 (s, 3H), 1.70 - 1.65 (m, 4H), 1.40 -

1.35 (m, 4H), 1.31 - 1.26 (m, 8H), 0.88 (t, $J = 6.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.14 (d, $J = 8.4$ Hz), 97.95 (d, $J = 4.9$ Hz), 68.21 (d, $J = 6.3$ Hz), 31.26, 30.16 (d, $J = 7.0$ Hz), 25.06, 22.48, 20.71 (d, $J = 5.1$ Hz), 13.94. ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.56. HRMS (m/z) calcd for $\text{C}_{15}\text{H}_{31}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 307.2033, found 307.2033.



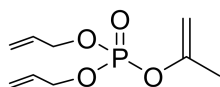
Bis(2-ethylhexyl) prop-1-en-2-yl phosphate (3bd)

Oily liquid, yield 81%, 293 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 4.75 - 4.75 (m, 1H), 4.48 - 4.48 (m, 1H), 4.01 - 3.97 (m, 4H), 1.94 (s, 3H), 1.58 - 1.56 (m, 2H), 1.44 - 1.33 (m, 6H), 1.32 - 1.26 (m, 10H), 0.92 - 0.86 (m, 12H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.16 (d, $J = 8.5$ Hz), 97.90 (d, $J = 4.8$ Hz), 70.13 (d, $J = 6.6$ Hz), 40.06 (d, $J = 7.5$ Hz), 29.85, 28.83, 23.07 (d, $J = 43.4$ Hz), 20.72 (d, $J = 5.2$ Hz), 14.00, 10.88. ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.33. HRMS (m/z) calcd for $\text{C}_{19}\text{H}_{39}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 363.2659, found 363.2660.



Bis(2-chloroethyl) prop-1-en-2-yl phosphate (3be)

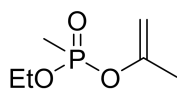
Oily liquid, yield 81%, 213 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 4.81 - 4.80 (m, 1H), 4.55 - 4.54 (m, 1H), 4.35 - 4.30 (m, 4H), 3.78 - 3.64 (m, 4H), 1.96 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 151.92 (d, $J = 8.7$ Hz), 98.87 (d, $J = 5.0$ Hz), 67.52 (d, $J = 5.6$ Hz), 42.21 (d, $J = 7.8$ Hz), 20.56 (d, $J = 4.9$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -7.46. HRMS (m/z) calcd for $\text{C}_7\text{H}_{13}\text{Cl}_2\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 263.0001, found 263.0004.



Diallyl prop-1-en-2-yl phosphate (3bf)

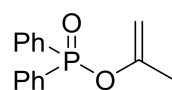
Oily liquid, yield 82%, 179 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 5.97 - 5.91 (m, 2H), 5.37 (dd, $J = 17.1, 1.4$ Hz, 2H), 5.25 (dd, $J = 10.4, 1.1$ Hz, 2H), 4.78 - 4.77 (m, 1H), 4.61 - 4.55 (m, 4H), 4.52 - 4.49 (m, 1H), 1.94 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.06 (d, $J = 8.5$ Hz), 132.20 (d, $J = 7.1$ Hz), 118.36, 98.35 (d, $J = 4.9$ Hz), 68.49 (d, $J = 5.7$ Hz), 20.68 (d, $J = 5.0$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.65. HRMS (m/z) calcd for $\text{C}_9\text{H}_{15}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 219.0781, found

219.0786.



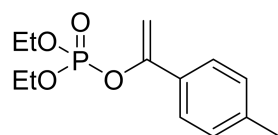
Ethyl prop-1-en-2-yl methylphosphonate (3bg)

Oily liquid, yield 76%, 125 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 4.73 - 4.72 (m, 1H), 4.54 - 4.41 (m, 1H), 4.24 - 3.98 (m, 2H), 1.93 (s, 3H), 1.55 (d, $J = 17.6$ Hz, 3H), 1.33 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.16 (d, $J = 9.3$ Hz), 98.11 (d, $J = 5.0$ Hz), 61.99 (d, $J = 6.5$ Hz), 21.26 (d, $J = 3.9$ Hz), 16.29 (d, $J = 7.6$ Hz), 11.74, 10.78. ^{31}P NMR (243 MHz, Chloroform-*d*) δ 26.38. HRMS (m/z) calcd for $\text{C}_6\text{H}_{13}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$ 165.0675, found 165.0675.



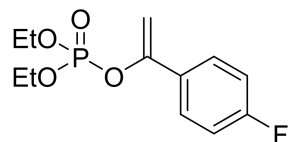
Prop-1-en-2-yl diphenylphosphinate (3bh)

White solid, yield 84%, 217 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.92 - 7.75 (m, 4H), 7.55 - 7.49 (m, 2H), 7.48 - 7.43 (m, 4H), 4.73 - 4.72 (m, 1H), 4.50 - 4.25 (m, 1H), 1.93 (d, $J = 1.0$ Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.18 (d, $J = 9.3$ Hz), 132.22 (d, $J = 2.8$ Hz), 131.62 (d, $J = 10.2$ Hz), 131.41 (d, $J = 138.3$ Hz), 128.46 (d, $J = 13.4$ Hz), 98.87 (d, $J = 5.5$ Hz), 21.69 (d, $J = 4.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ 28.49. HRMS (m/z) calcd for $\text{C}_{15}\text{H}_{15}\text{O}_2\text{P}$ $[\text{M}+\text{H}]^+$ 259.0882, found 259.0884.



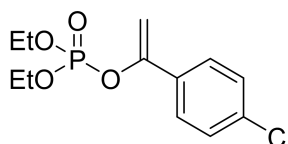
Diethyl (1-(p-tolyl)vinyl) phosphate (3ca)

Oily liquid, yield 84%, 227 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.48 (d, $J = 8.2$ Hz, 2H), 7.16 (d, $J = 8.0$ Hz, 2H), 5.24 - 5.23 (m, 1H), 5.17 - 5.17 (m, 1H), 4.27 - 4.12 (m, 4H), 2.35 (s, 3H), 1.34 (td, $J = 7.1, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.38 (d, $J = 7.8$ Hz), 139.07, 131.53 (d, $J = 6.8$ Hz), 129.03, 125.10, 96.35 (d, $J = 3.7$ Hz), 64.45 (d, $J = 6.0$ Hz), 21.20, 16.07 (d, $J = 6.8$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.32. HRMS (m/z) calcd for $\text{C}_{13}\text{H}_{19}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 271.1094, found 271.1099.



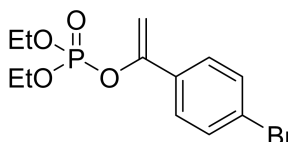
Diethyl (1-(4-fluorophenyl)vinyl) phosphate (3cb)

Oily liquid, yield 83%, 228 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.62 - 7.48 (m, 2H), 7.07 - 7.00 (m, 2H), 5.22 - 5.21 (m, 1H), 5.21 - 5.20 (m, 1H), 4.25 - 4.16 (m, 4H), 1.35 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 163.19 (d, $J = 249.0$ Hz), 151.47 (d, $J = 7.7$ Hz), 130.54 (dd, $J = 7.0, 3.3$ Hz), 127.16 (d, $J = 8.3$ Hz), 115.36 (d, $J = 21.9$ Hz), 97.12 (dd, $J = 3.6, 1.5$ Hz), 64.56 (d, $J = 6.0$ Hz), 16.08 (d, $J = 6.8$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.31. HRMS (m/z) calcd for $\text{C}_{12}\text{H}_{16}\text{FO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 275.0843, found 275.0845.



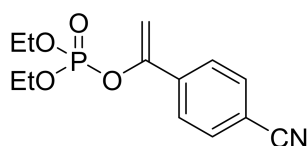
1-(4-chlorophenyl)vinyl diethyl phosphate (3cc)

Oily liquid, yield 89%, 258 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.57 - 7.45 (m, 2H), 7.39 - 7.28 (m, 2H), 5.27 - 5.26 (m, 1H), 5.25 - 5.23 (m, 1H), 4.27 - 4.13 (m, 4H), 1.35 (td, $J = 7.1, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 151.34 (d, $J = 7.7$ Hz), 134.99, 132.82 (d, $J = 7.0$ Hz), 128.58, 126.52, 97.78 (d, $J = 3.6$ Hz), 64.59 (d, $J = 6.0$ Hz), 16.08 (d, $J = 6.7$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.30. HRMS (m/z) calcd for $\text{C}_{12}\text{H}_{16}\text{ClO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 291.0547, found 291.0548.



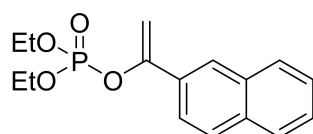
1-(4-bromophenyl)vinyl diethyl phosphate (3cd)

Oily liquid, yield 88%, 294 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.50 - 7.47 (m, 2H), 7.46 - 7.43 (m, 2H), 5.29 - 5.28 (m, 1H), 5.25 - 5.24 (m, 1H), 4.24 - 4.16 (m, 4H), 1.35 (td, $J = 7.1, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 151.39 (d, $J = 7.6$ Hz), 133.28 (d, $J = 6.9$ Hz), 131.55, 126.78, 123.24, 97.88 (d, $J = 3.6$ Hz), 64.60 (d, $J = 6.0$ Hz), 16.08 (d, $J = 6.7$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.30. HRMS (m/z) calcd for $\text{C}_{12}\text{H}_{16}\text{BrO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 335.0042, found 335.0042.



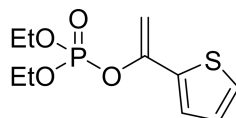
1-(4-cyanophenyl)vinyl diethyl phosphate (3ce)

Oily liquid, yield 96%, 270 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.70 - 7.67 (m, 2H), 7.66 - 7.63 (m, 2H), 5.43 - 5.42 (m, 1H), 5.40 - 5.39 (m, 1H), 4.26 - 4.17 (m, 4H), 1.35 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 150.59 (d, $J = 7.5$ Hz), 138.50 (d, $J = 7.0$ Hz), 132.23, 125.70, 118.42, 112.55, 100.44, 64.74 (d, $J = 6.0$ Hz), 16.08 (d, $J = 6.7$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.27. HRMS (m/z) calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 282.0890, found 282.0886.



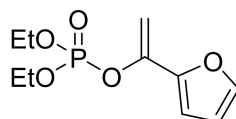
Diethyl (1-(naphthalen-2-yl)vinyl) phosphate (3cf)

Oily liquid, yield 91%, 279 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 8.09 - 8.05 (m, 1H), 7.88 - 7.85 (m, 1H), 7.82 - 7.80 (m, 2H), 7.67 (dd, $J = 8.7, 1.8$ Hz, 1H), 7.52 - 7.47 (m, 2H), 5.44 - 5.43 (m, 1H), 5.34 - 5.33 (m, 1H), 4.29 - 4.20 (m, 4H), 1.37 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.29 (d, $J = 7.8$ Hz), 133.45, 132.97, 131.46 (d, $J = 6.8$ Hz), 128.55, 128.10, 127.56, 126.66, 126.47, 124.56, 122.74, 97.80 (d, $J = 3.5$ Hz), 64.54 (d, $J = 6.0$ Hz), 16.10 (d, $J = 6.7$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.18. HRMS (m/z) calcd for $\text{C}_{16}\text{H}_{19}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 307.1094, found 307.1098.



Diethyl (1-(thiophen-2-yl)vinyl) phosphate (3cg)

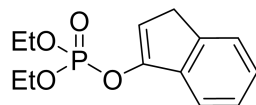
Oily liquid, yield 83%, 218 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.27 - 7.23 (m, 2H), 6.99 (dd, $J = 5.0, 3.7$ Hz, 1H), 5.17 - 5.16 (m, 1H), 5.12 - 5.11 (m, 1H), 4.26 - 4.18 (m, 4H), 1.36 (td, $J = 7.1, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 147.45 (d, $J = 7.1$ Hz), 138.37 (d, $J = 8.2$ Hz), 127.45, 125.95, 125.08, 96.10 (d, $J = 3.5$ Hz), 64.68 (d, $J = 6.1$ Hz), 16.07 (d, $J = 6.7$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.68. HRMS (m/z) calcd for $\text{C}_{10}\text{H}_{15}\text{O}_4\text{PS}$ $[\text{M}+\text{H}]^+$ 263.0501, found 263.0506.



Diethyl (1-(furan-2-yl)vinyl) phosphate (3ch)

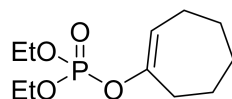
Oily liquid, yield 81%, 199 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.38 - 7.38 (m, 1H), 6.52 (d, $J = 3.3$ Hz, 1H), 6.40 (dd, $J = 3.4, 1.8$ Hz, 1H), 5.26 - 5.25 (m, 1H), 5.11

- 5.10 (m, 1H), 4.25 - 4.18 (m, 4H), 1.36 (td, $J = 7.1, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 148.78 (d, $J = 9.5$ Hz), 144.09 (d, $J = 6.7$ Hz), 143.17, 111.32, 108.09, 95.64 (d, $J = 3.8$ Hz), 64.63 (d, $J = 6.1$ Hz), 16.07 (d, $J = 6.7$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.20. HRMS (m/z) calcd for $\text{C}_{10}\text{H}_{15}\text{O}_5\text{P}$ $[\text{M}+\text{H}]^+$ 247.0730, found 247.0731.



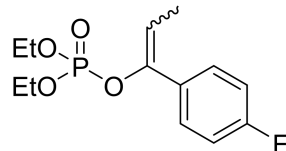
Diethyl (1H-inden-3-yl) phosphate (3ci)

Oily liquid, yield 65%, 174 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 7.45 - 7.42 (m, 2H), 7.34 - 7.31 (m, 1H), 7.28 - 7.26 (m, 1H), 6.11 (q, $J = 2.1$ Hz, 1H), 4.29 - 4.22 (m, 4H), 3.38 - 3.38 (m, 2H), 1.38 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 148.88 (d, $J = 7.5$ Hz), 142.04, 138.87 (d, $J = 8.2$ Hz), 126.36, 125.87, 124.06, 118.04, 111.52 (d, $J = 3.3$ Hz), 64.71 (d, $J = 6.0$ Hz), 34.48, 16.13 (d, $J = 6.6$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.77. HRMS (m/z) calcd for $\text{C}_{13}\text{H}_{17}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 269.0937, found 269.0937.



Cyclohept-1-en-1-yl diethyl phosphate (3cj)

Oily liquid, yield 57%, 142 mg; ^1H NMR (600 MHz, Chloroform-*d*) δ 5.62 (td, $J = 6.5, 2.6$ Hz, 1H), 4.13 (p, $J = 7.2$ Hz, 4H), 2.42 - 2.38 (m, 2H), 2.08 - 2.04 (m, 2H), 1.70 - 1.67 (m, 2H), 1.65 - 1.61 (m, 2H), 1.59 - 1.56 (m, 2H), 1.34 (td, $J = 7.1, 0.9$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 152.16 (d, $J = 9.5$ Hz), 115.36 (d, $J = 5.5$ Hz), 63.99 (d, $J = 6.1$ Hz), 33.34 (d, $J = 3.6$ Hz), 30.57, 26.93, 25.04, 24.80 (d, $J = 1.1$ Hz), 16.11 (d, $J = 6.8$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.99. HRMS (m/z) calcd for $\text{C}_{11}\text{H}_{21}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 249.1250, found 249.1251.



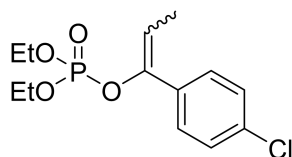
Diethyl (1-(4-fluorophenyl)prop-1-en-1-yl) phosphate (3da)

Oily liquid, yield 71%, 205 mg; HRMS (m/z) calcd for $\text{C}_{13}\text{H}_{18}\text{FO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 289.1000, found 289.1003.

(**Configuration 1**): ^1H NMR (600 MHz, Chloroform-*d*) δ 7.49 - 7.46 (m, 2H), 7.08 - 7.04 (m, 2H), 5.74 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.15 - 4.09 (m, 4H), 1.87 (dd, $J = 7.1, 3.1$ Hz, 3H), 1.29 - 1.23 (m, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 163.36 (d, $J =$

8.8 Hz), 145.91 (d, $J = 8.8$ Hz), 131.96 (dd, $J = 3.2, 1.6$ Hz), 130.48 (d, $J = 8.2$ Hz), 115.16 (d, $J = 1.8$ Hz), 112.04 (d, $J = 6.6$ Hz), 64.29 (d, $J = 6.0$ Hz), 16.04 (d, $J = 2.1$ Hz), 12.91 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.59 .

(Configuration 2): ^1H NMR (600 MHz, Chloroform-*d*) δ 7.45 - 7.42 (m, 2H), 7.02 - 6.98 (m, 2H), 5.59 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.09 - 4.03 (m, 4H), 1.73 (dd, $J = 7.1, 3.1$ Hz, 3H), 1.29 - 1.23 (m, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.72 (d, $J = 8.8$ Hz), 145.42 (d, $J = 8.8$ Hz), 130.19 (dd, $J = 3.2, 1.6$ Hz), 127.18 (d, $J = 8.2$ Hz), 115.02 (d, $J = 1.8$ Hz), 111.79 (d, $J = 6.6$ Hz), 64.18 (d, $J = 6.0$ Hz), 15.99 (d, $J = 2.1$ Hz), 11.71 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.64 .

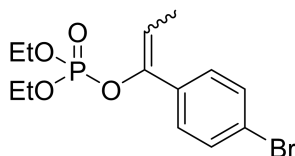


1-(4-chlorophenyl)prop-1-en-1-yl diethyl phosphate (3db)

Oily liquid, yield 72%, 219 mg; HRMS (m/z) calcd for $\text{C}_{13}\text{H}_{18}\text{ClO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 305.0704, found 305.0706.

(Configuration 1): ^1H NMR (600 MHz, Chloroform-*d*) δ 7.45 - 7.44 (m, 1H), 7.41 - 7.40 (m, 1H), 7.34 - 7.34 (m, 1H), 7.28 - 7.28 (m, 1H), 5.77 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.16 - 4.03 (m, 4H), 1.74 (dd, $J = 7.1, 3.1$ Hz, 3H), 1.28 (td, $J = 7.1, 1.1$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 145.27 (d, $J = 8.8$ Hz), 134.32 , 132.56 (d, $J = 4.3$ Hz), 129.88 , 128.32 , 112.35 (d, $J = 6.6$ Hz), 64.23 (d, $J = 6.1$ Hz), 16.01 (d, $J = 3.6$ Hz), 12.94 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.57 .

(Configuration 2): ^1H NMR (600 MHz, Chloroform-*d*) δ 7.43 - 7.42 (m, 1H), 7.39 - 7.38 (m, 1H), 7.36 - 7.35 (m, 1H), 7.30 - 7.29 (m, 1H), 5.66 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.16 - 4.03 (m, 4H), 1.88 (dd, $J = 7.1, 3.1$ Hz, 3H), 1.26 (td, $J = 7.1, 1.1$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 145.80 (d, $J = 8.8$ Hz), 134.23 (d, $J = 4.3$ Hz), 133.86 , 128.36 , 126.58 , 112.80 (d, $J = 6.6$ Hz), 64.34 (d, $J = 6.1$ Hz), 16.05 (d, $J = 3.6$ Hz), 11.79 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.61 .



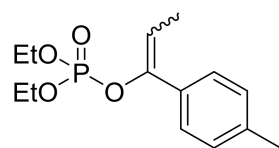
1-(4-bromophenyl)prop-1-en-1-yl diethyl phosphate (3dc)

Oily liquid, yield 75%, 261 mg; HRMS (m/z) calcd for $\text{C}_{13}\text{H}_{18}\text{BrO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 349.0199, found 349.0198.

(Configuration 1): ^1H NMR (600 MHz, Chloroform-*d*) δ 7.52 - 7.51 (m, 1H), 7.45 - 7.45 (m, 1H), 7.37 - 7.36 (m, 1H), 7.32 - 7.32 (m, 1H), 5.77 (qd, $J = 7.0, 2.7$ Hz, 1H),

4.17 - 4.03 (m, 4H), 1.74 (dd, $J = 7.1, 3.1$ Hz, 3H), 1.28 (td, $J = 7.1, 1.1$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform- d) δ 145.29 (d, $J = 8.8$ Hz), 133.02 (d, $J = 2.0$ Hz), 131.28, 130.14, 122.57, 112.42 (d, $J = 5.0$ Hz), 64.23 (d, $J = 6.0$ Hz), 16.02 (d, $J = 6.8$ Hz), 12.94 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform- d) δ -5.57.

(Configuration 2): ^1H NMR (600 MHz, Chloroform- d) δ 7.50 - 7.49 (m, 1H), 7.44 - 7.43 (m, 1H), 7.38 - 7.37 (m, 1H), 7.34 - 7.33 (m, 1H), 5.67 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.17 - 4.03 (m, 4H), 1.87 (dd, $J = 7.1, 3.1$ Hz, 3H), 1.26 (td, $J = 7.1, 1.1$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform- d) δ 145.84 (d, $J = 8.8$ Hz), 134.68 (d, $J = 2.0$ Hz), 131.31, 126.85, 122.04, 112.90 (d, $J = 5.0$ Hz), 64.35 (d, $J = 6.0$ Hz), 16.04 (d, $J = 6.8$ Hz), 11.80 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform- d) δ -5.61.

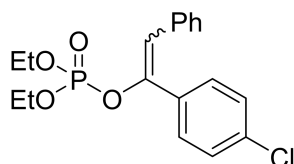


Diethyl (1-(p-tolyl)prop-1-en-1-yl) phosphate (3dd)

Oily liquid, yield 72%, 205 mg; HRMS (m/z) calcd for $\text{C}_{14}\text{H}_{21}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 285.1250, found 285.1252.

(Configuration 1): ^1H NMR (600 MHz, Chloroform- d) δ 7.39 (d, $J = 8.2$ Hz, 2H), 7.18 (d, $J = 7.9$ Hz, 2H), 5.72 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.15 - 4.08 (m, 4H), 2.36 (s, 3H), 1.87 (dd, $J = 7.0, 3.1$ Hz, 3H), 1.27 (td, $J = 6.5, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform- d) δ 146.86 (d, $J = 8.8$ Hz), 138.33, 132.94 (d, $J = 1.5$ Hz), 128.84, 128.42, 111.20 (d, $J = 6.5$ Hz), 64.20 (d, $J = 6.1$ Hz), 21.29, 16.03 (d, $J = 2.2$ Hz), 12.96 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform- d) δ -5.65.

(Configuration 2): ^1H NMR (600 MHz, Chloroform- d) δ 7.34 (d, $J = 8.2$ Hz, 2H), 7.12 (d, $J = 7.9$ Hz, 2H), 5.61 (qd, $J = 7.0, 2.7$ Hz, 1H), 4.07 - 4.01 (m, 4H), 2.33 (s, 3H), 1.75 (dd, $J = 7.0, 3.1$ Hz, 3H), 1.24 (td, $J = 6.5, 1.0$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform- d) δ 146.39 (d, $J = 8.8$ Hz), 137.89, 131.21 (d, $J = 1.5$ Hz), 128.71, 125.23, 111.10 (d, $J = 6.5$ Hz), 64.09 (d, $J = 6.1$ Hz), 21.14, 15.98 (d, $J = 2.2$ Hz), 11.70 (d, $J = 1.1$ Hz). ^{31}P NMR (243 MHz, Chloroform- d) δ -5.68.

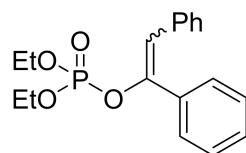


1-(4-chlorophenyl)-2-phenylvinyl diethyl phosphate (3de)

Oily liquid, yield 65%, 238 mg; HRMS (m/z) calcd for $\text{C}_{18}\text{H}_{20}\text{ClO}_4\text{P}$ $[\text{M}+\text{H}]^+$ 367.0860, found 367.0861.

(Configuration 1): ^1H NMR (600 MHz, Chloroform-*d*) δ 8.11 - 7.37 (m, 5H), 7.36 - 6.74(m, 4H), 6.40 (m, 1H), 4.03 - 3.97 (m, 2H), 3.92 - 3.84 (m, 2H), 1.14 (td, $J = 7.1$, 1.1 Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 145.43 (d, $J = 9.0$ Hz), 134.57 , 134.19 (d, $J = 1.6$ Hz), 131.47 , 129.26 , 128.76 , 128.49 , 128.41 , 127.23 , 116.35 (d, $J = 7.2$ Hz), 64.45 (d, $J = 6.0$ Hz), 15.89 (d, $J = 7.2$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.53 .

(Configuration 2): ^1H NMR (600 MHz, Chloroform-*d*) δ 8.11 - 7.37 (m, 5H), 7.36 - 6.74(m, 4H), 6.74 (d, $J = 2.6$ Hz, 1H), 4.20 - 4.13 (m, 4H), 1.33 (td, $J = 7.1$, 1.1 Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 146.04 (d, $J = 9.0$ Hz), 134.96 , 134.66 (d, $J = 1.6$ Hz), 130.57 , 128.98 , 128.57 , 128.33 , 127.68 , 127.48 , 117.66 (d, $J = 7.2$ Hz), 64.59 (d, $J = 6.0$ Hz), 16.08 (d, $J = 7.2$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -5.93 .



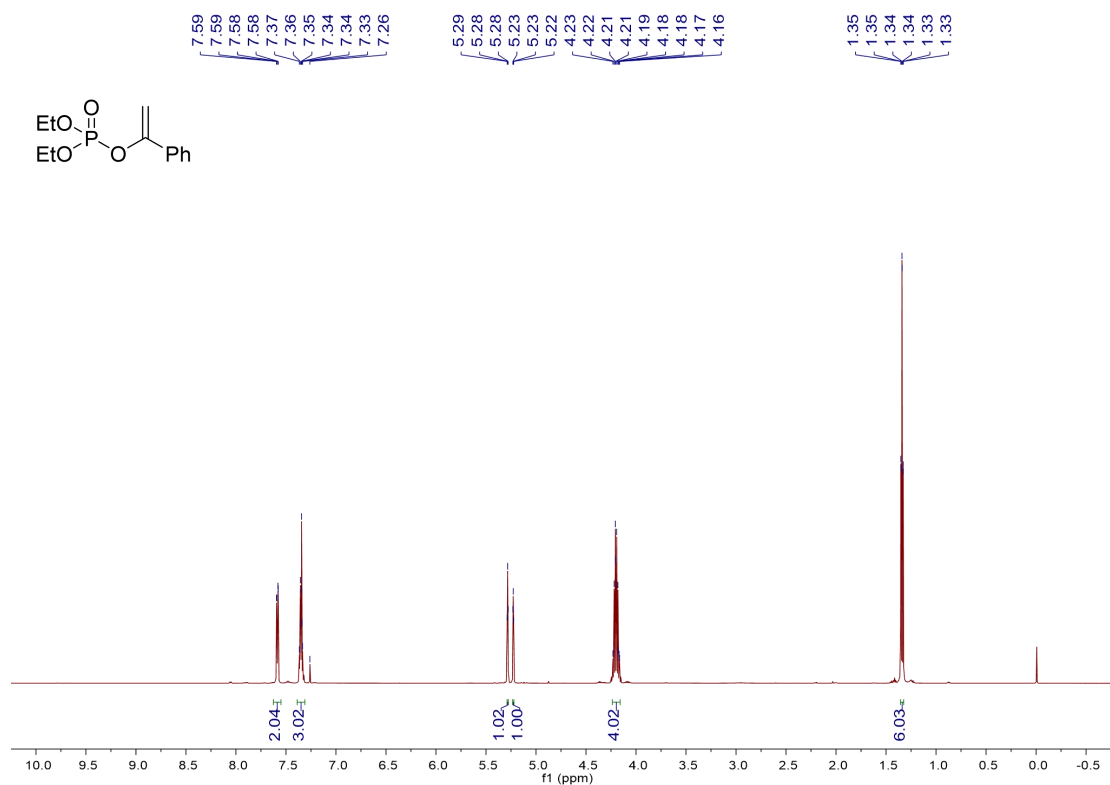
1,2-diphenylvinyl diethyl phosphate (3df)

Oily liquid, yield 66%, 219 mg; HRMS (m/z) calcd for $\text{C}_{18}\text{H}_{21}\text{O}_4\text{P}$ [$\text{M}+\text{H}$] $^+$ 333.1250, found 333.1251.

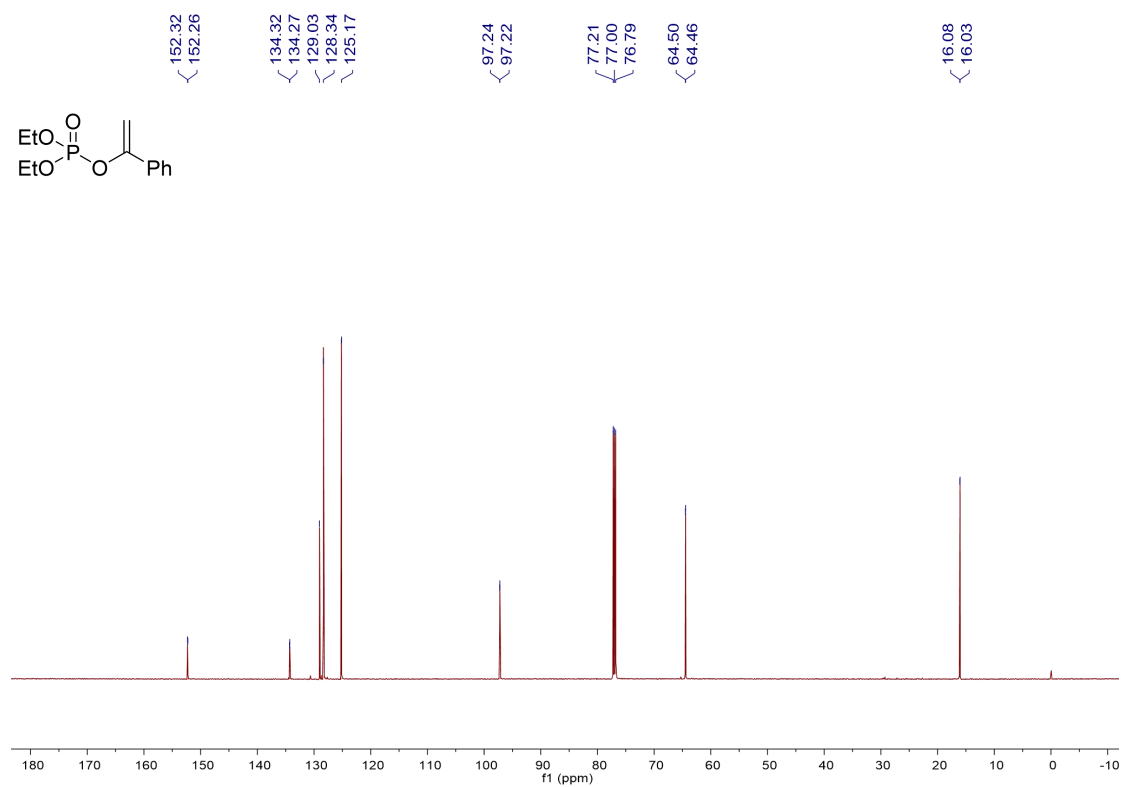
(Configuration 1): ^1H NMR (600 MHz, Chloroform-*d*) δ 8.14 - 7.62 (m, 5H), 7.47 - 7.28 (m, 5H), 6.41 (s, 1H), 4.01 - 3.98 (m, 2H), 3.89 - 3.82 (m, 2H), 1.11 (td, $J = 7.1$, 1.1 Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 146.55 (d, $J = 9.1$ Hz), 136.22 (d, $J = 1.0$ Hz), 134.22 (d, $J = 2.3$ Hz), 130.09 , 129.28, 129.10 , 128.73 , 128.38 , 127.48 , 115.98 (d, $J = 7.3$ Hz), 64.34 (d, $J = 6.0$ Hz), 15.88 (d, $J = 7.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.61 .

(Configuration 2): ^1H NMR (600 MHz, Chloroform-*d*) δ 7.47 - 7.28 (m, 5H), 7.17 - 7.04 (m, 5H), 6.73 (d, $J = 2.6$ Hz, 1H), 4.20 - 4.09 (m, 4H), 1.31 (td, $J = 7.1$, 1.1 Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 147.29 (d, $J = 9.1$ Hz), 134.54 (d, $J = 1.0$ Hz), 134.38 (d, $J = 2.3$ Hz), 129.24 , 129.03 , 128.31 , 128.29 , 128.18 , 126.99 , 117.10 (d, $J = 7.3$ Hz), 64.49 (d, $J = 6.0$ Hz), 16.08 (d, $J = 7.1$ Hz). ^{31}P NMR (243 MHz, Chloroform-*d*) δ -6.03 .

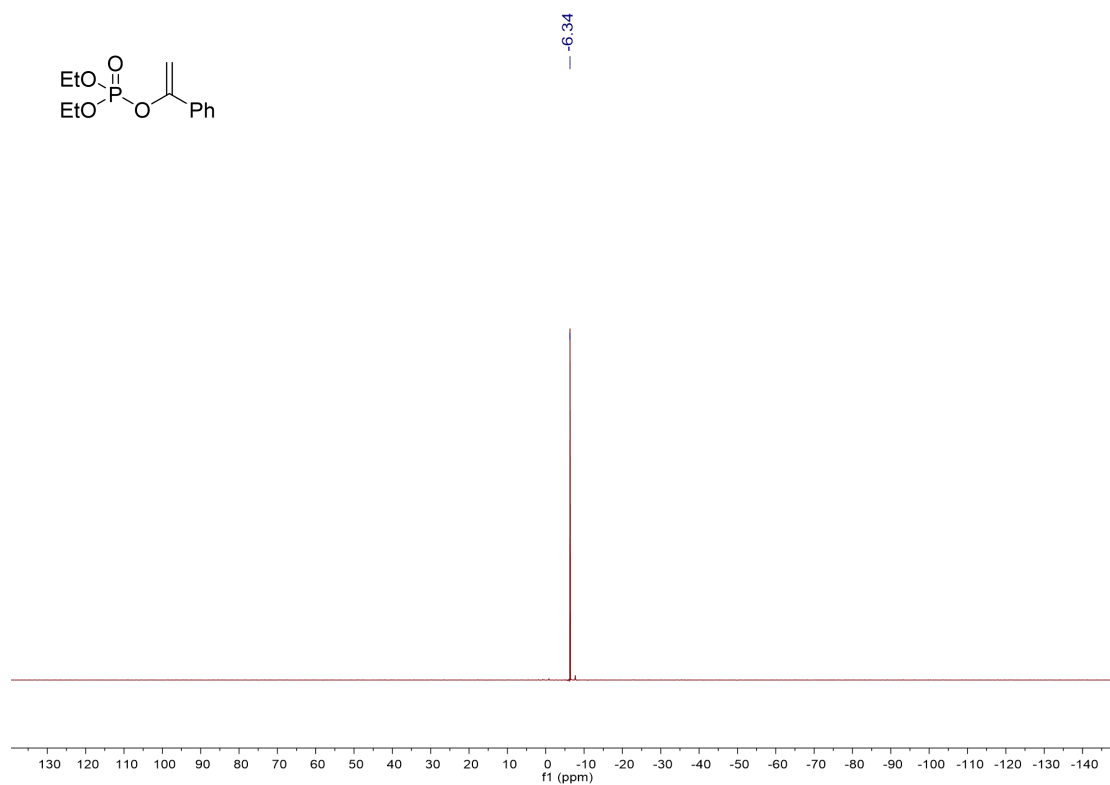
4. Copies of ^1H , ^{13}C , and ^{31}P NMR spectra



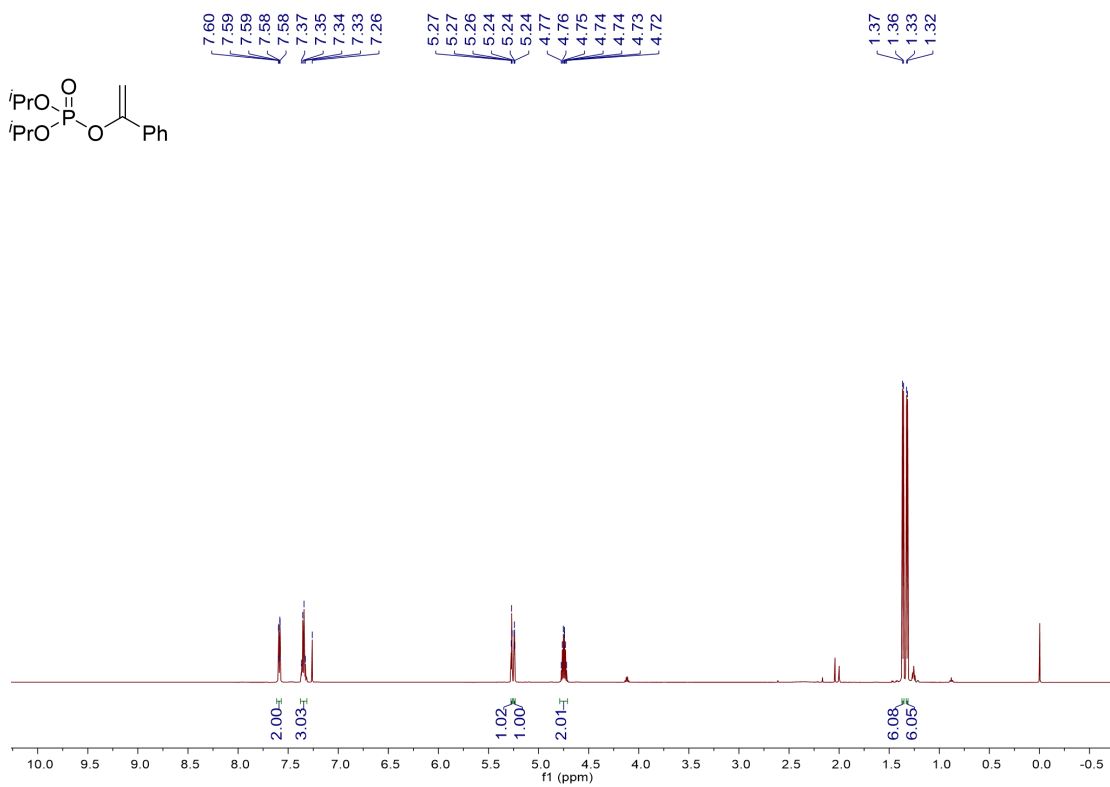
^1H NMR of compound **3aa**



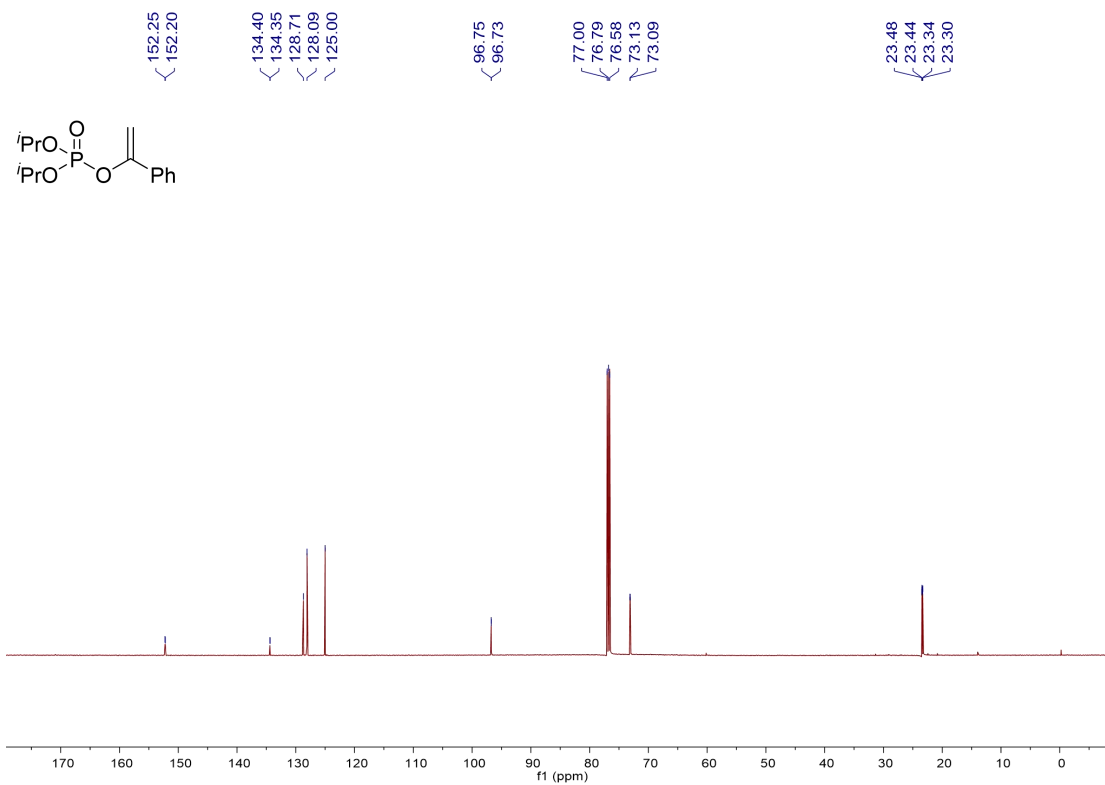
^{13}C NMR of compound **3aa**



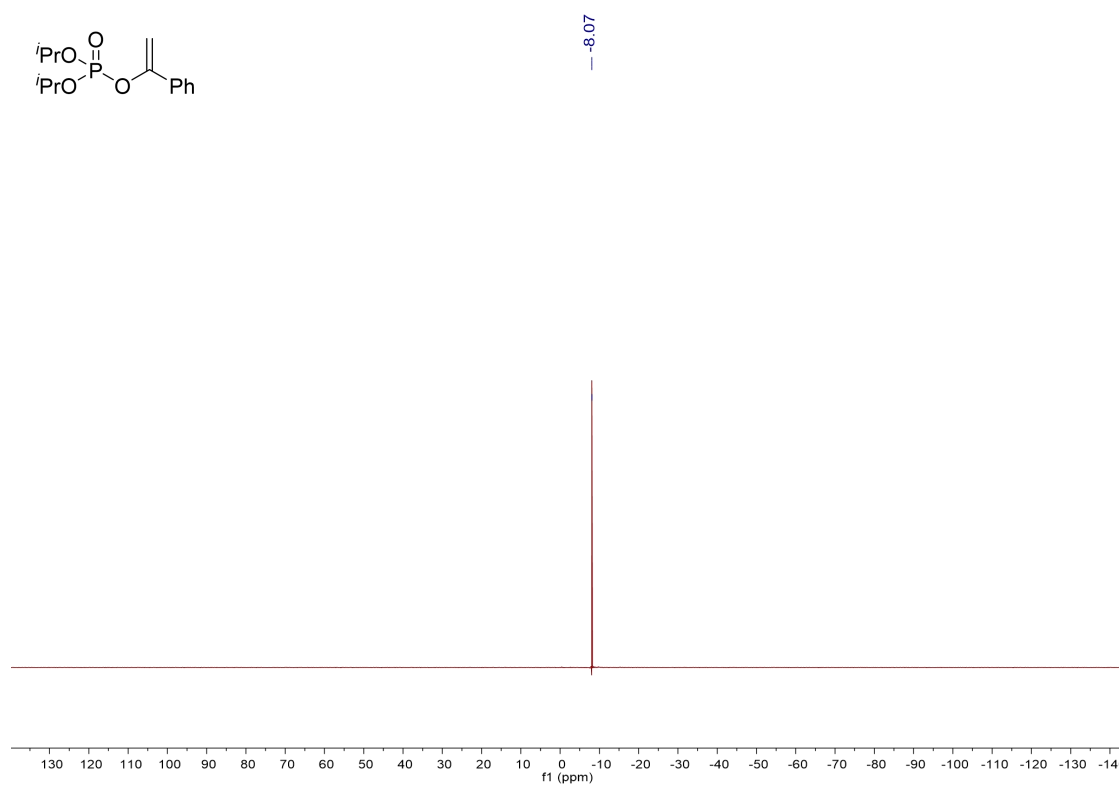
^{31}P NMR of compound **3aa**



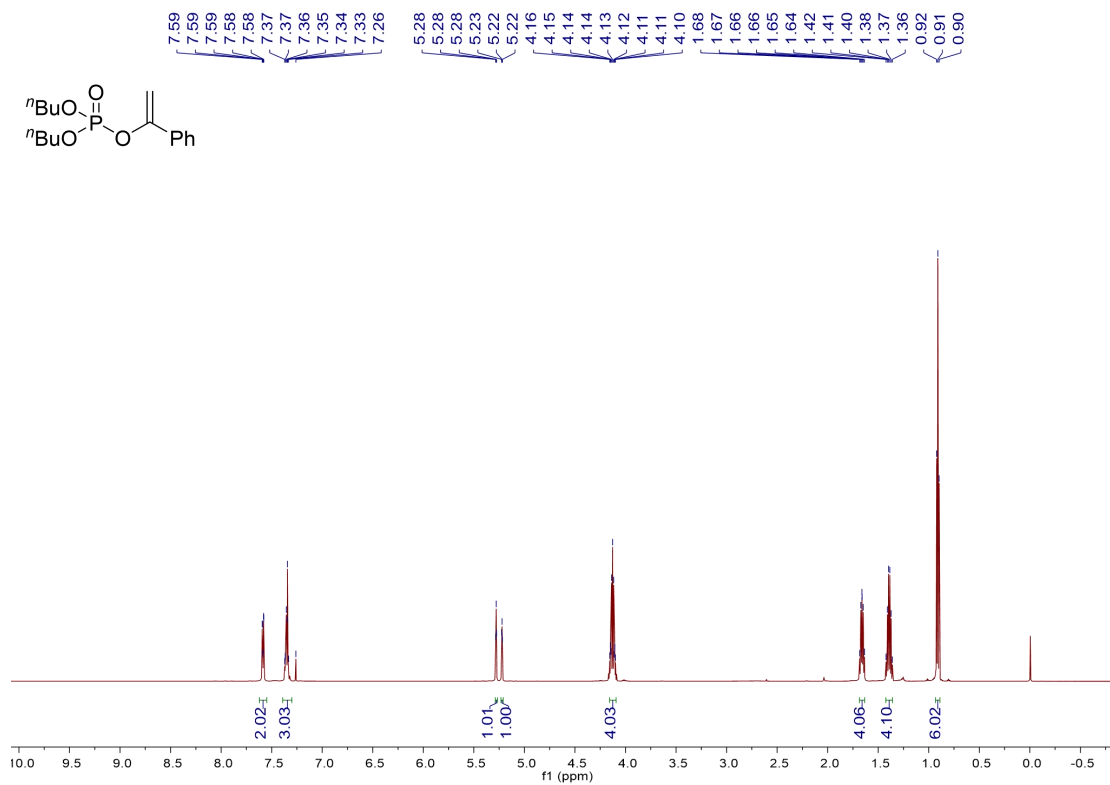
^1H NMR of compound **3ab**



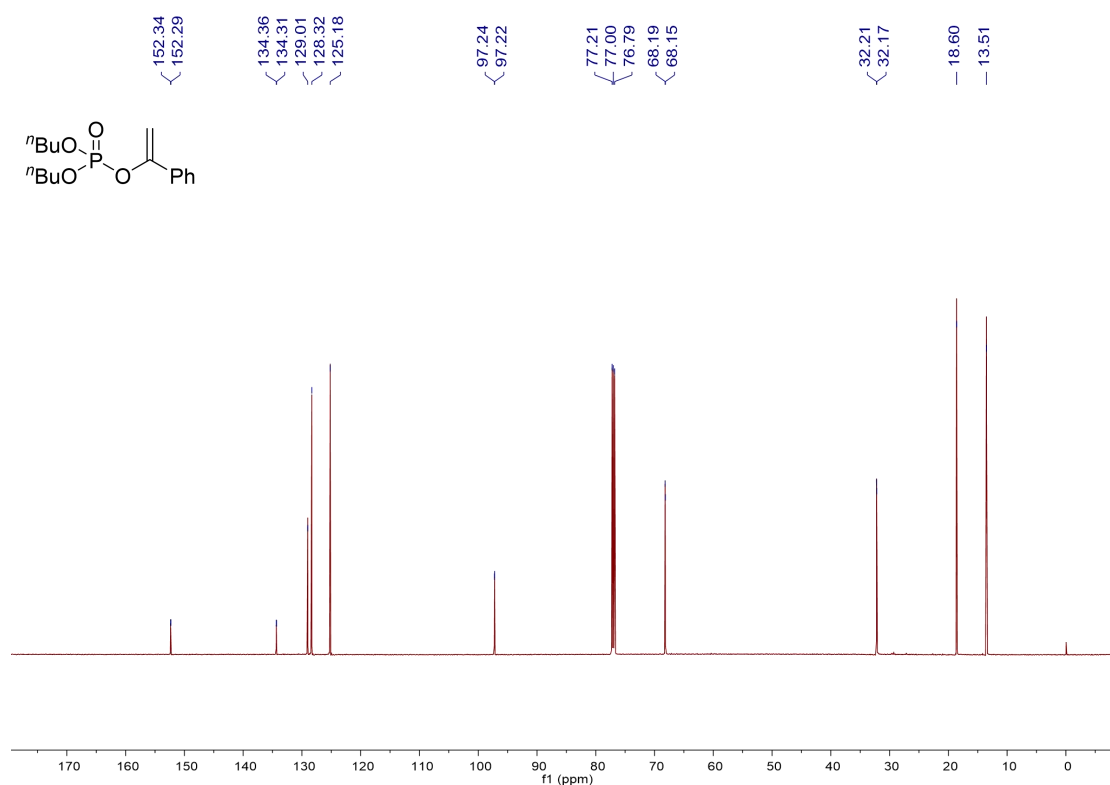
¹³C NMR of compound **3ab**



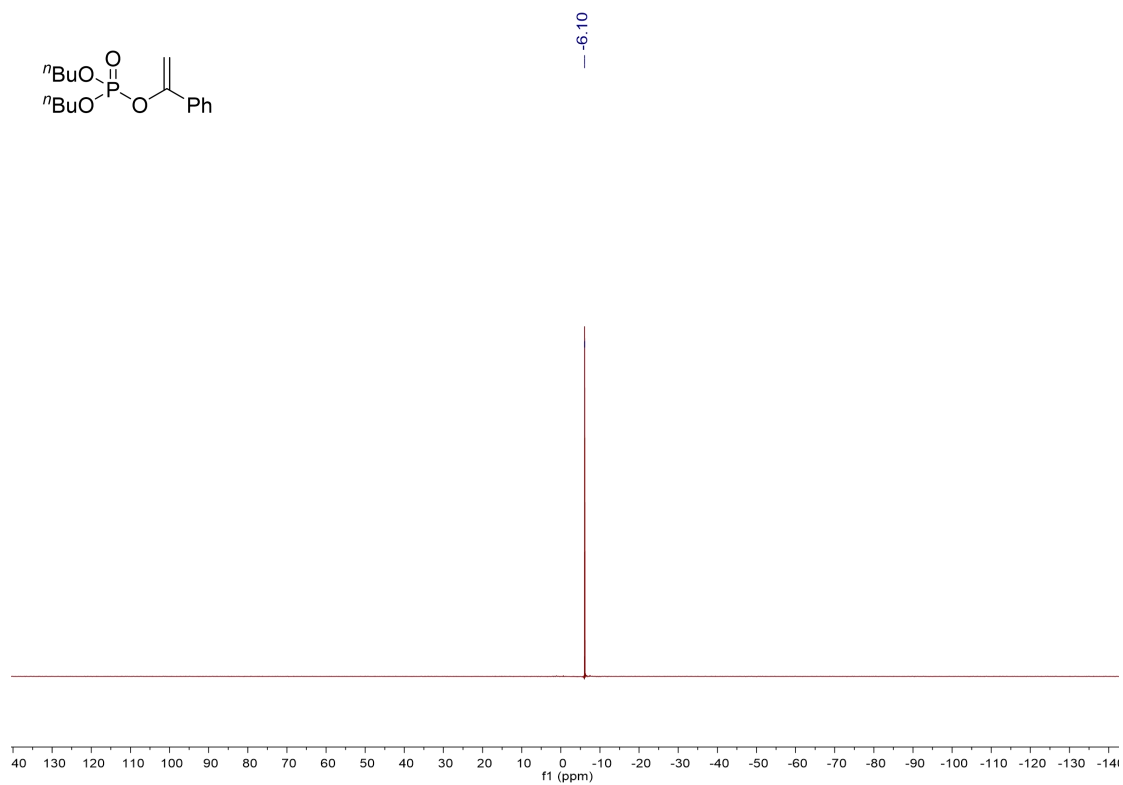
³¹P NMR of compound **3ab**



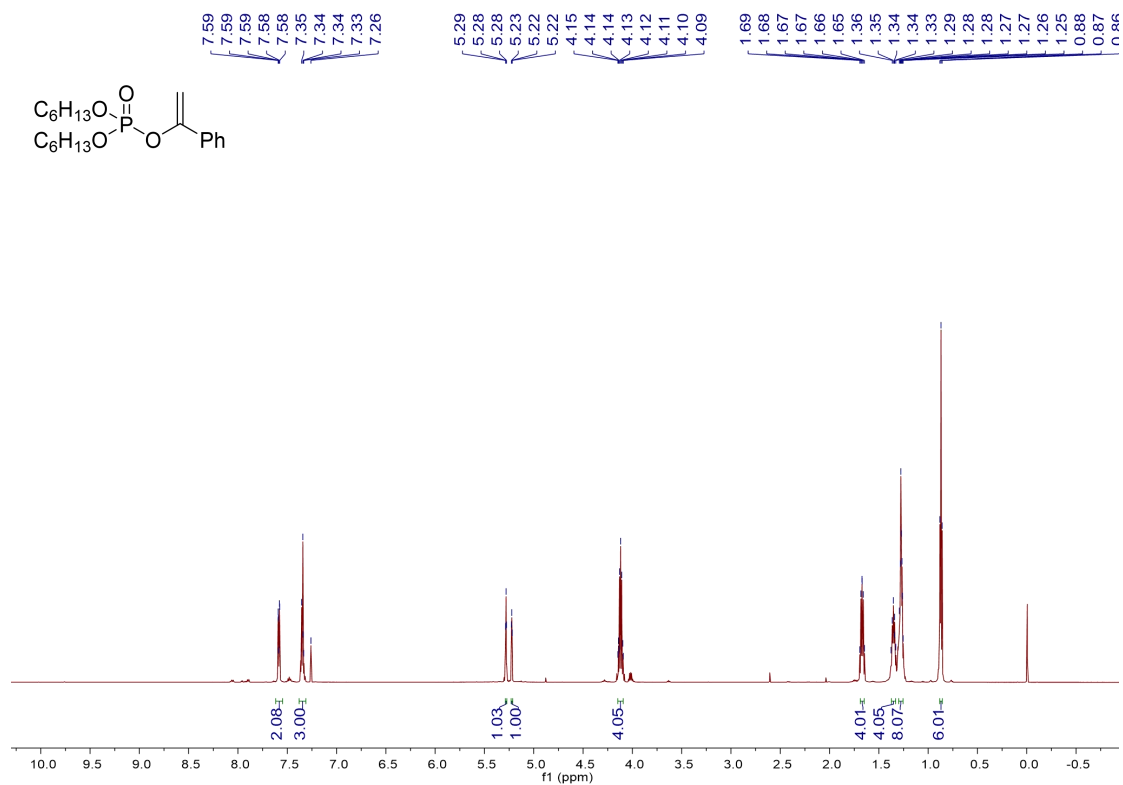
¹H NMR of compound **3ac**



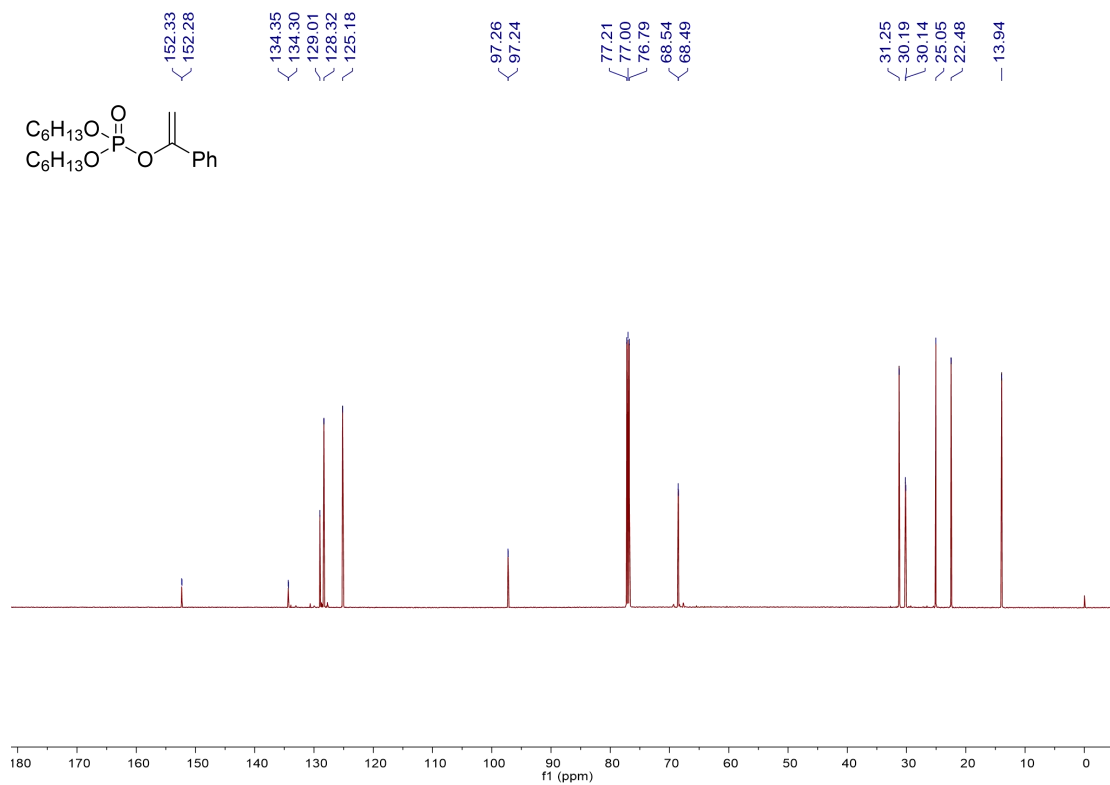
¹³C NMR of compound **3ac**



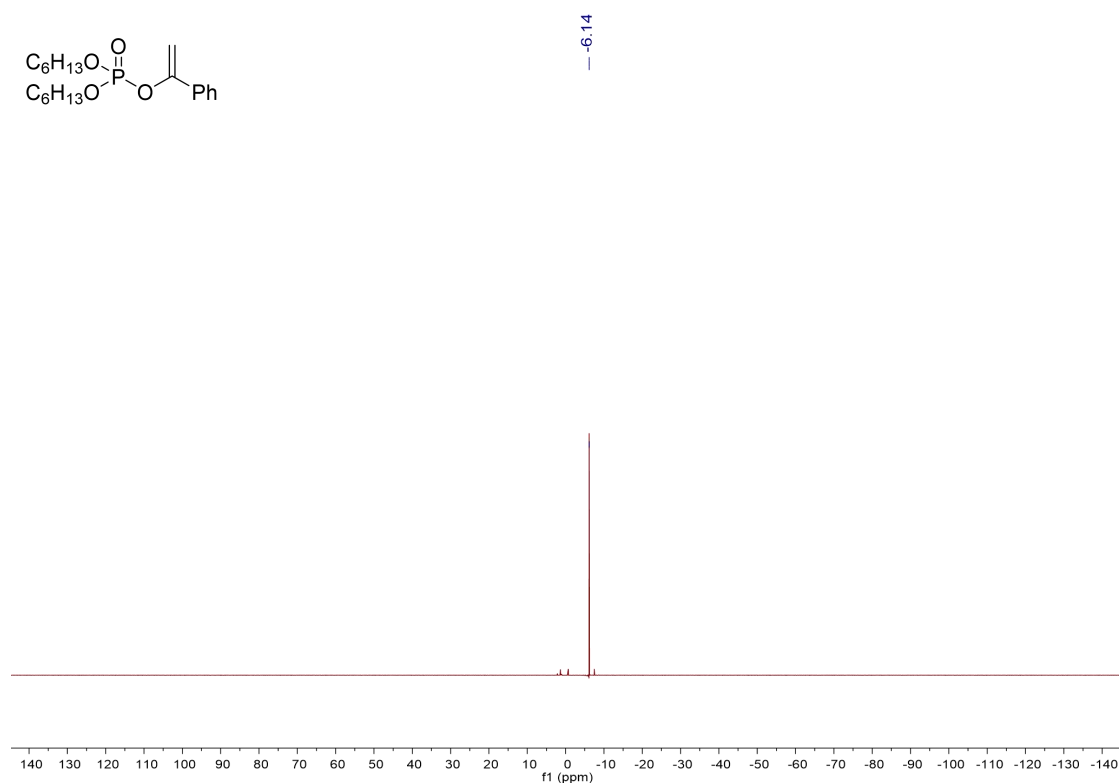
^{31}P NMR of compound **3ac**



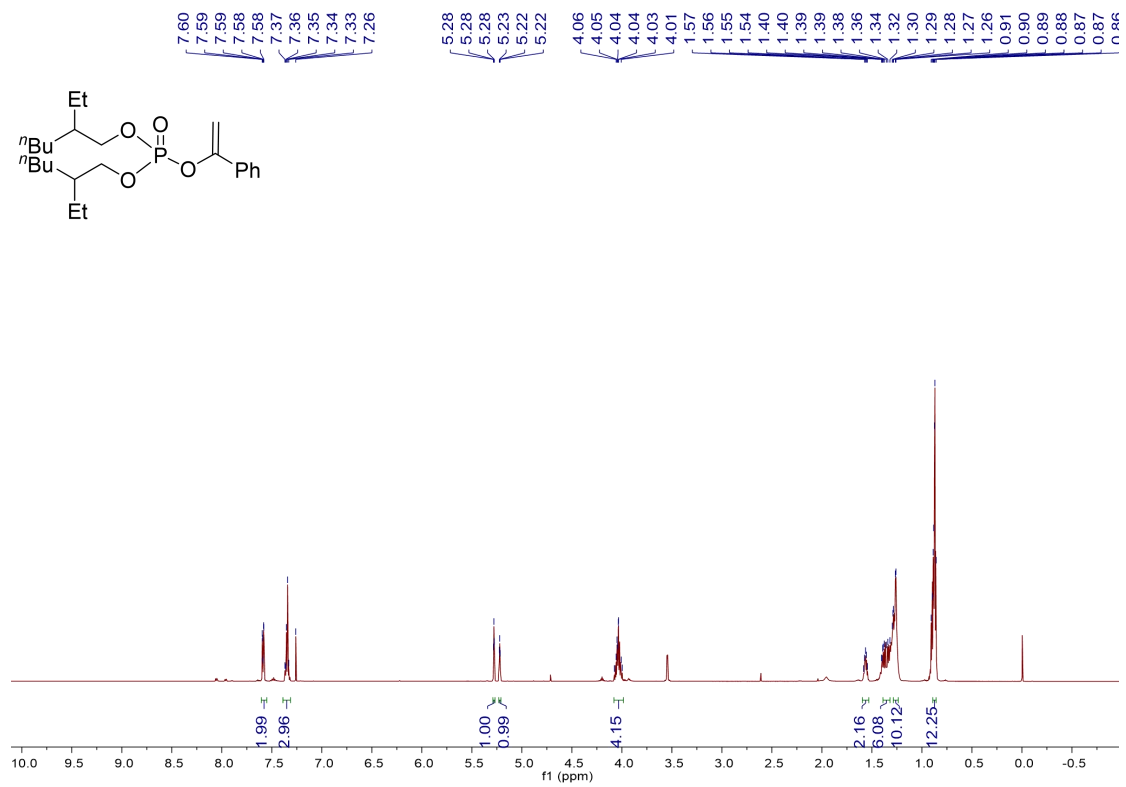
^1H NMR of compound **3ad**



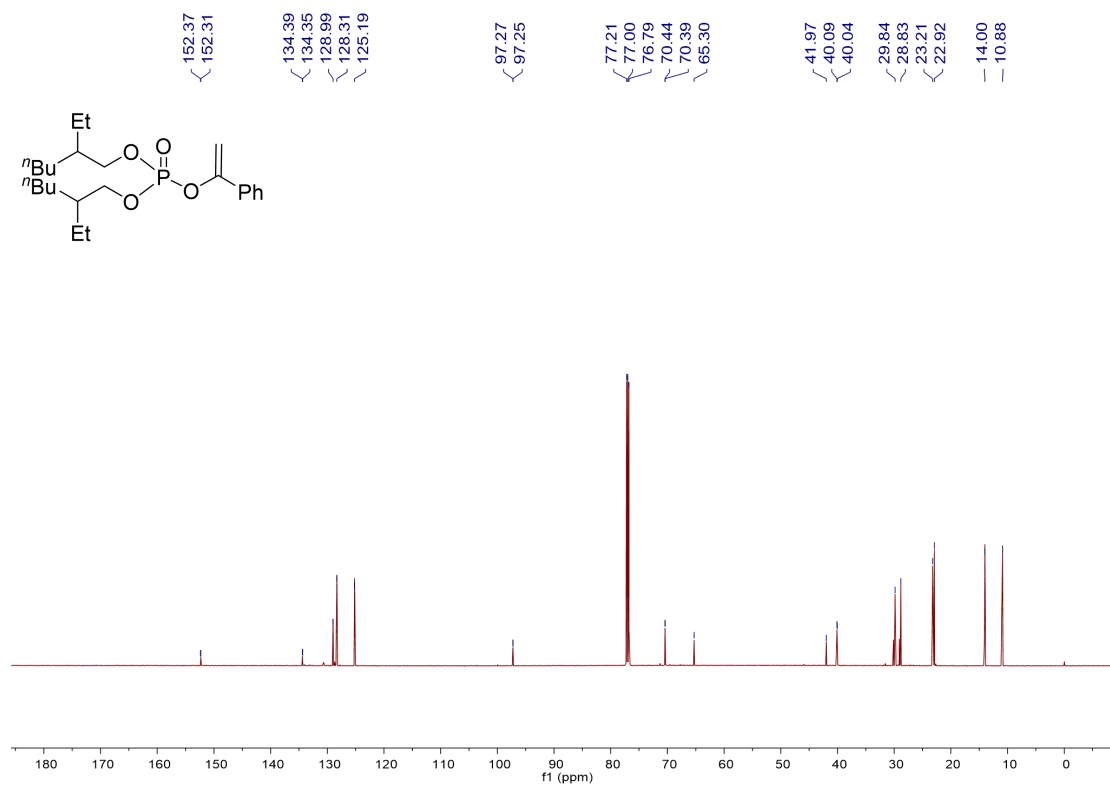
¹³C NMR of compound **3ad**



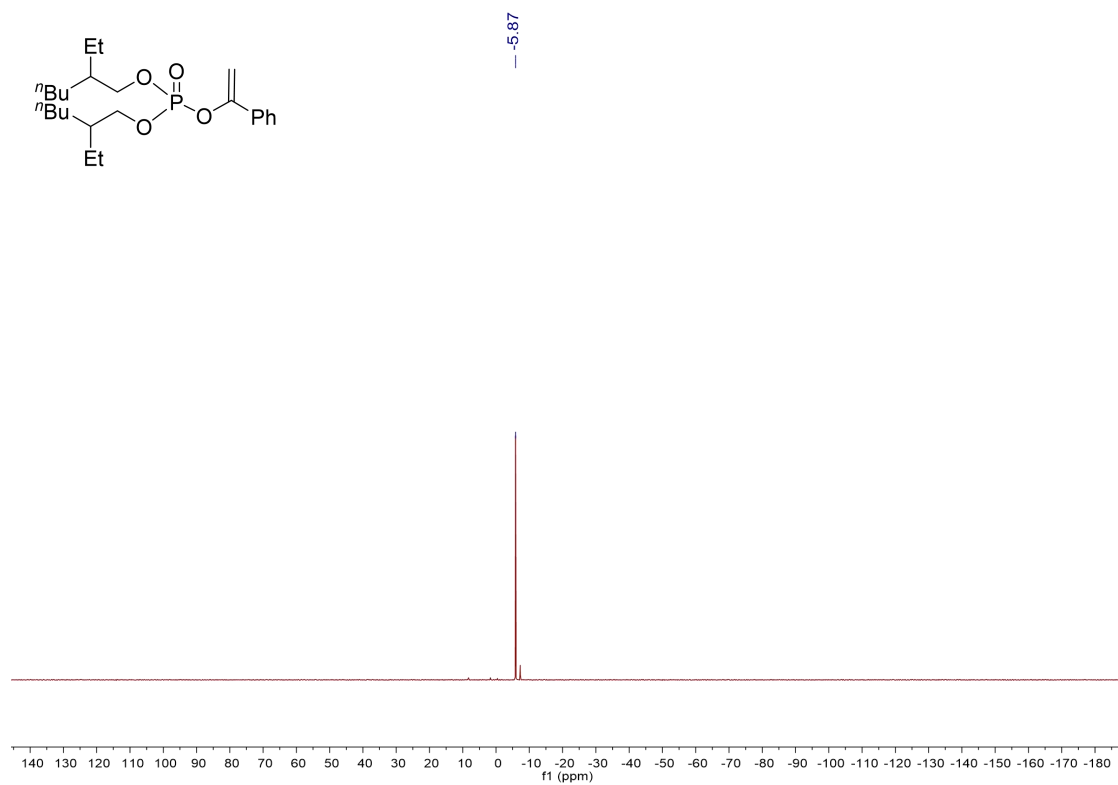
³¹P NMR of compound **3ad**



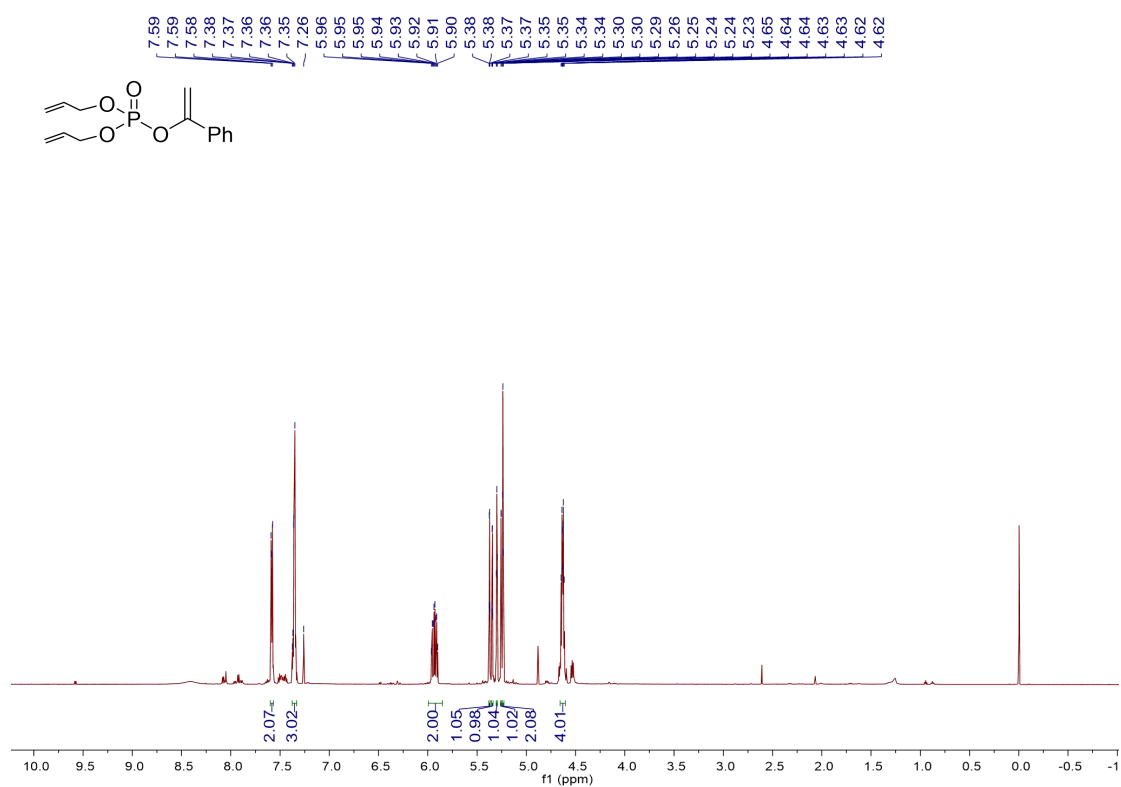
¹H NMR of compound **3ae**



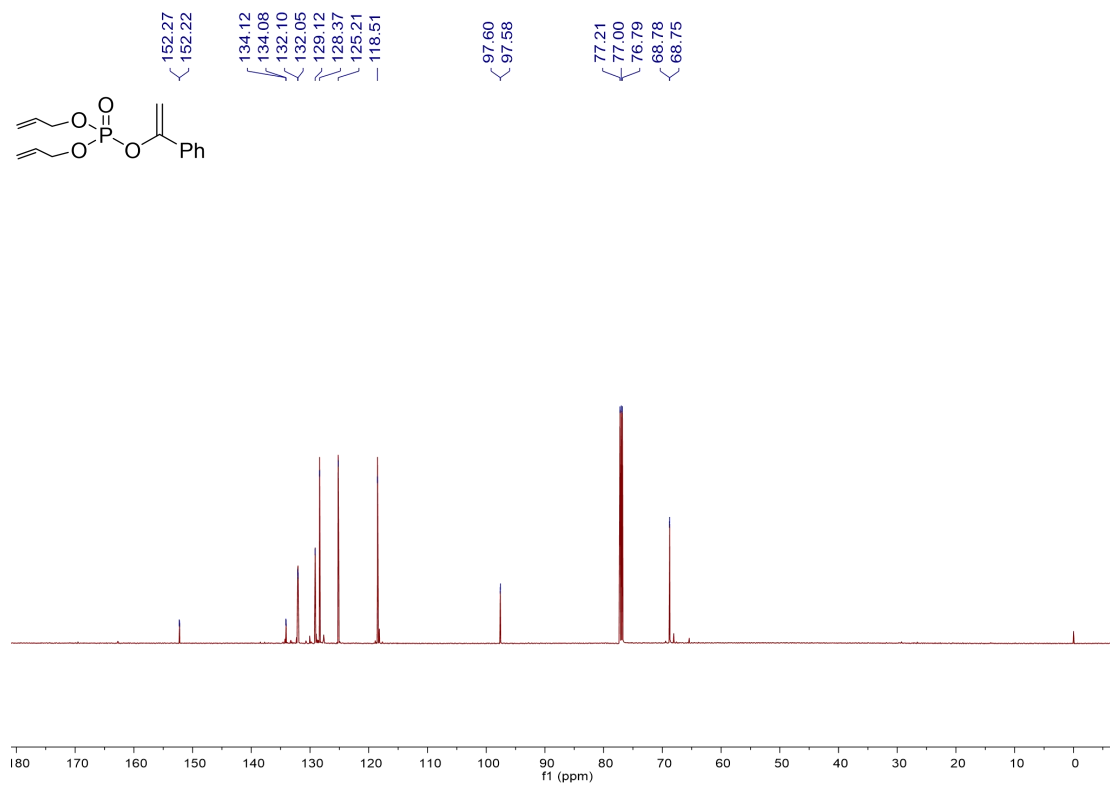
¹³C NMR of compound **3ae**



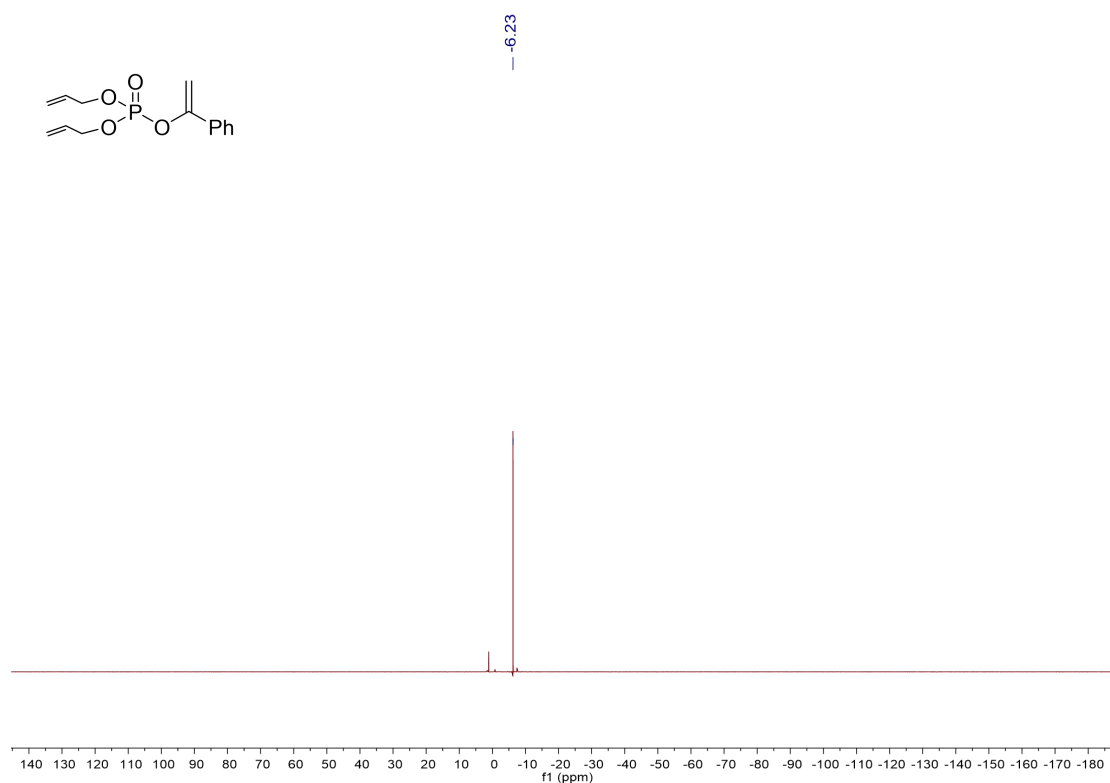
^{31}P NMR of compound 3ae



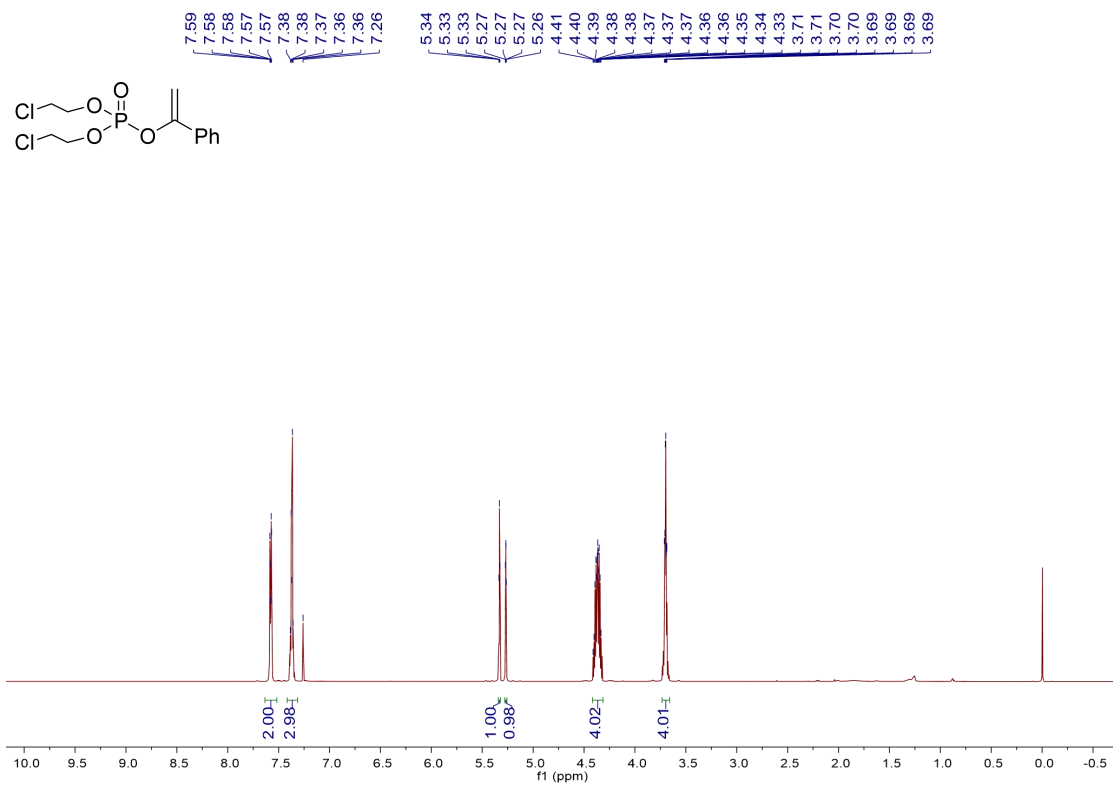
^1H NMR of compound 3af



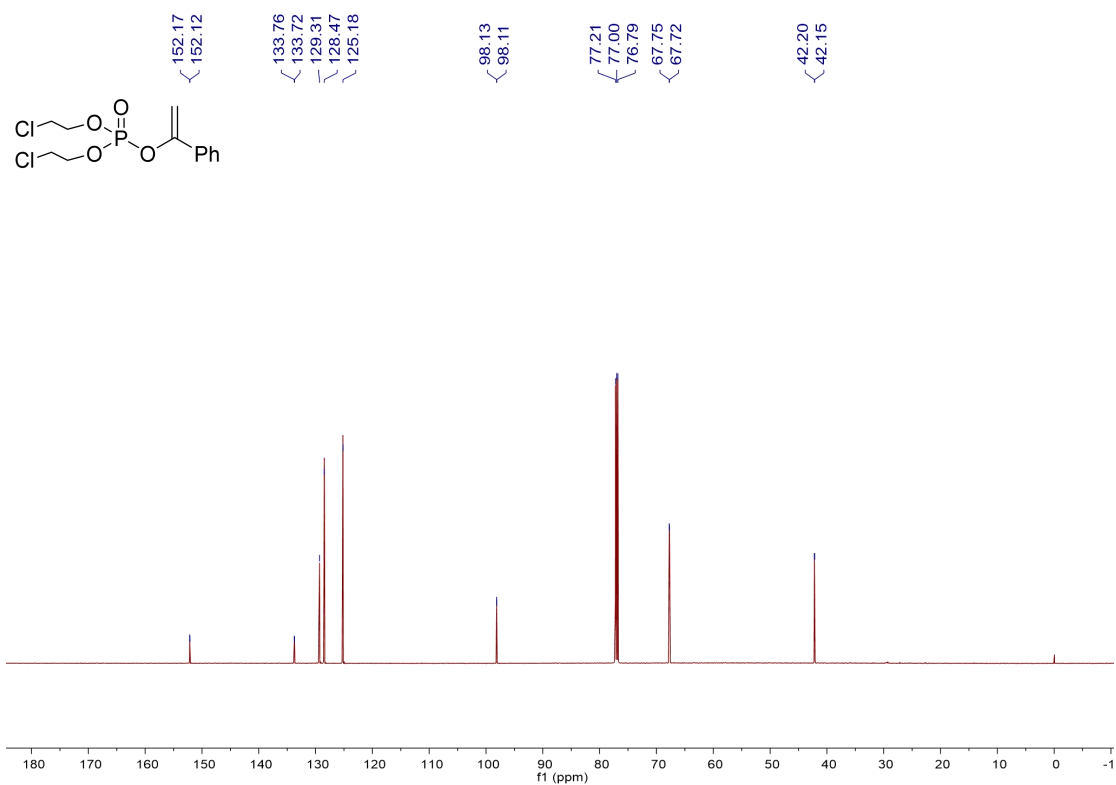
^{13}C NMR of compound **3af**



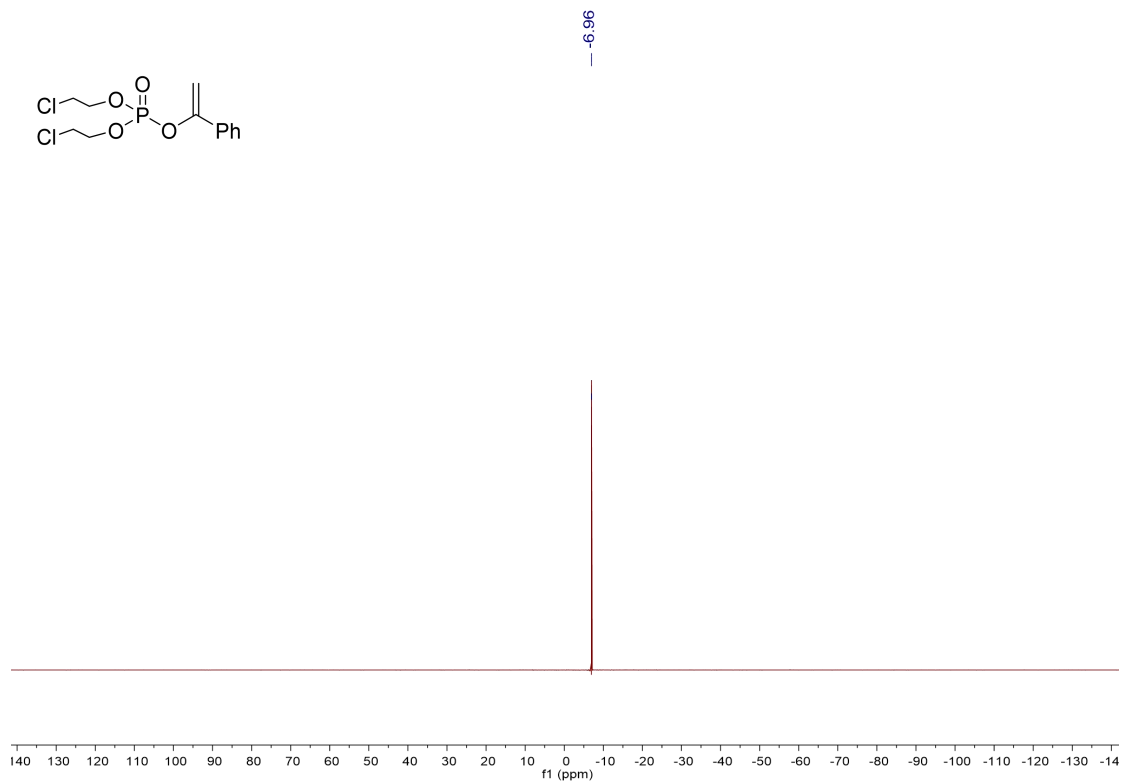
^{31}P NMR of compound **3af**



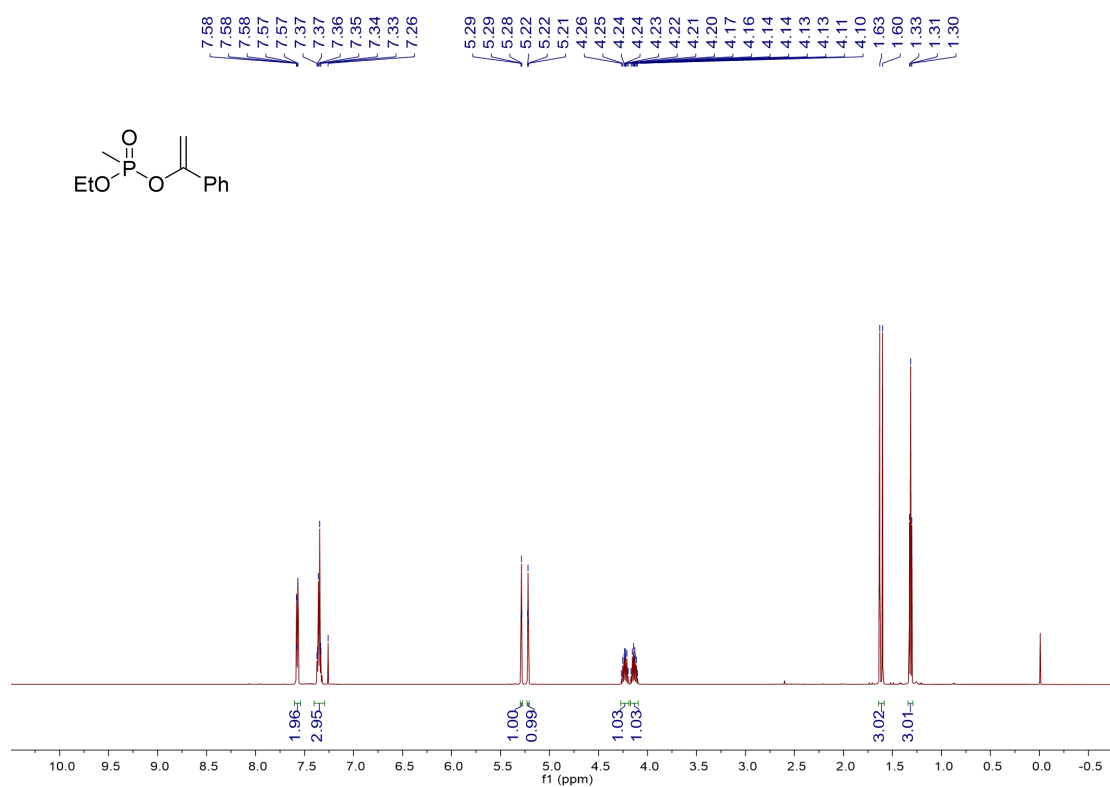
^1H NMR of compound **3ag**



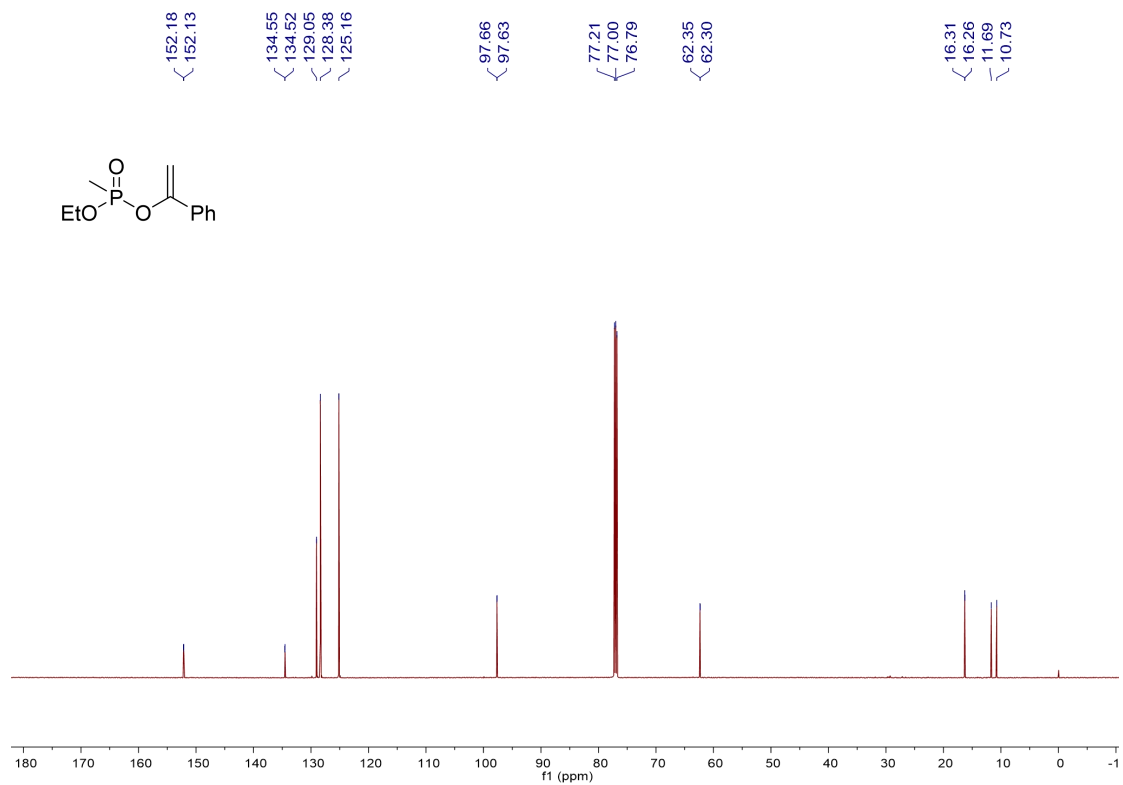
^{13}C NMR of compound **3ag**



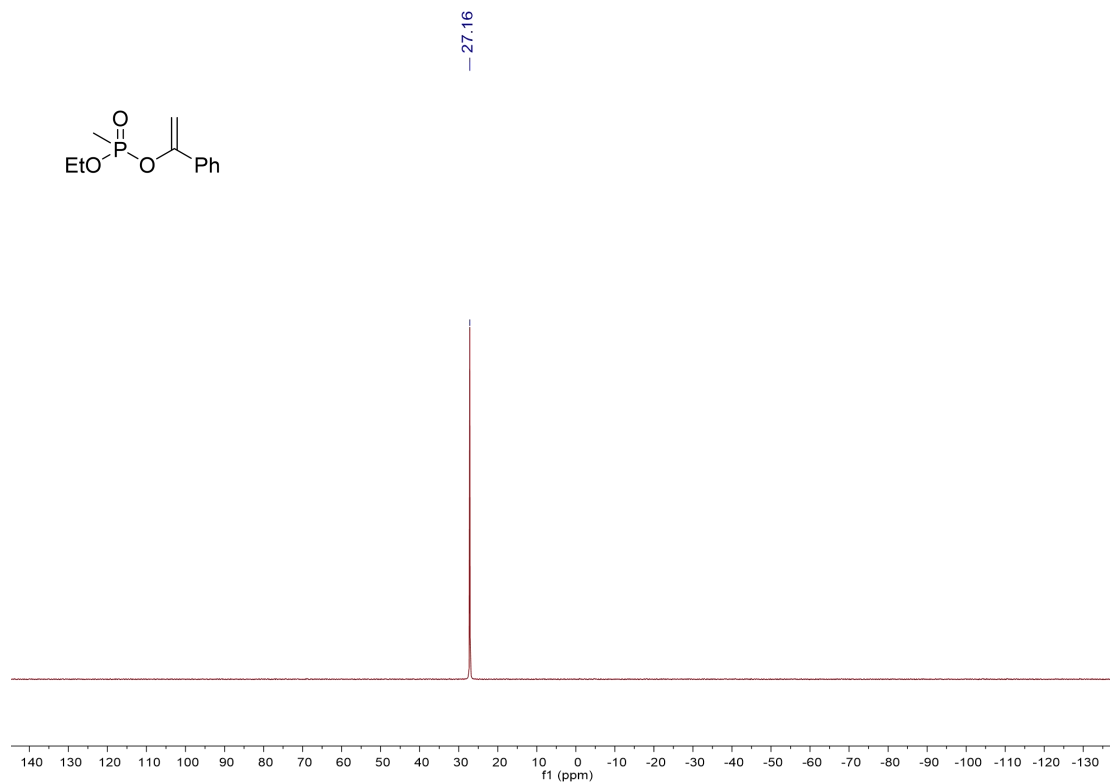
^{31}P NMR of compound **3ag**



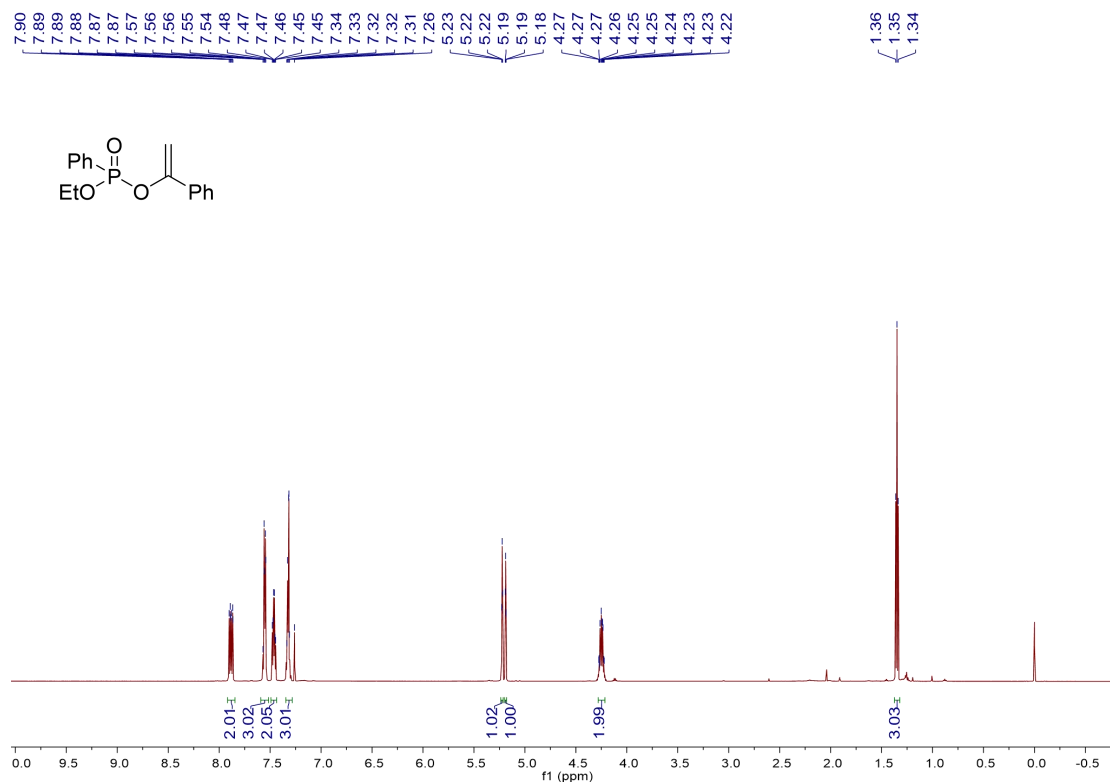
^1H NMR of compound **3ah**



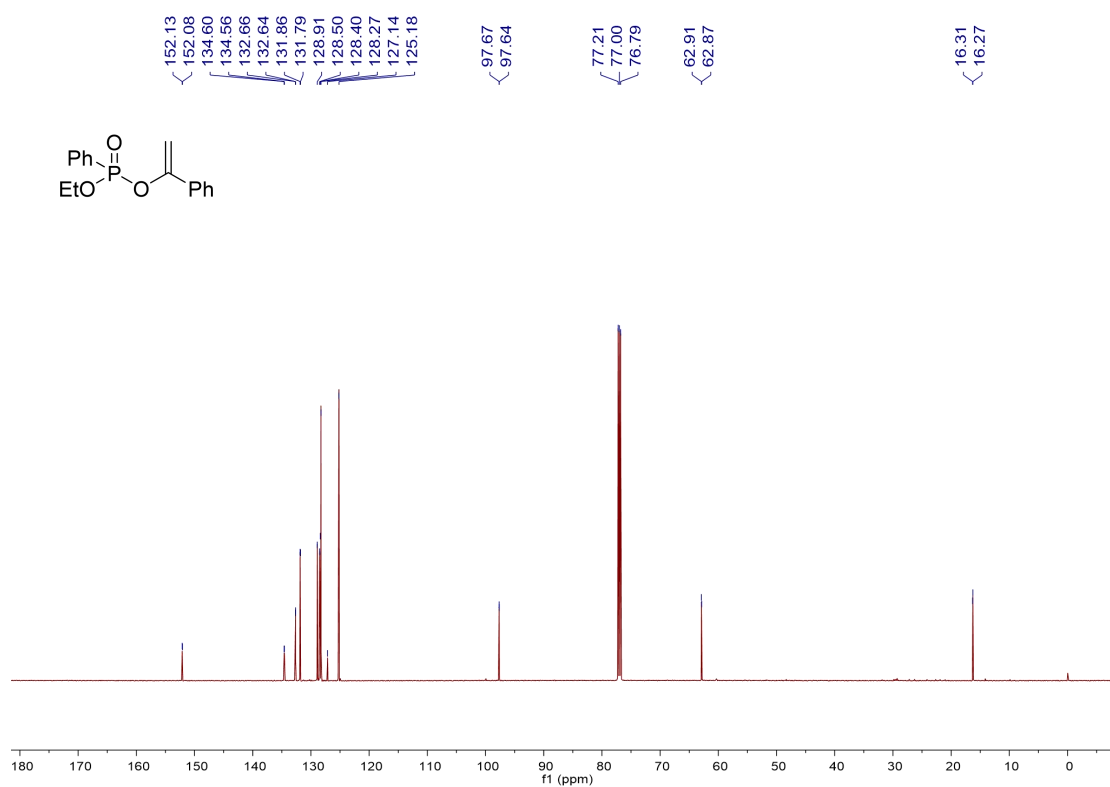
¹³C NMR of compound **3ah**



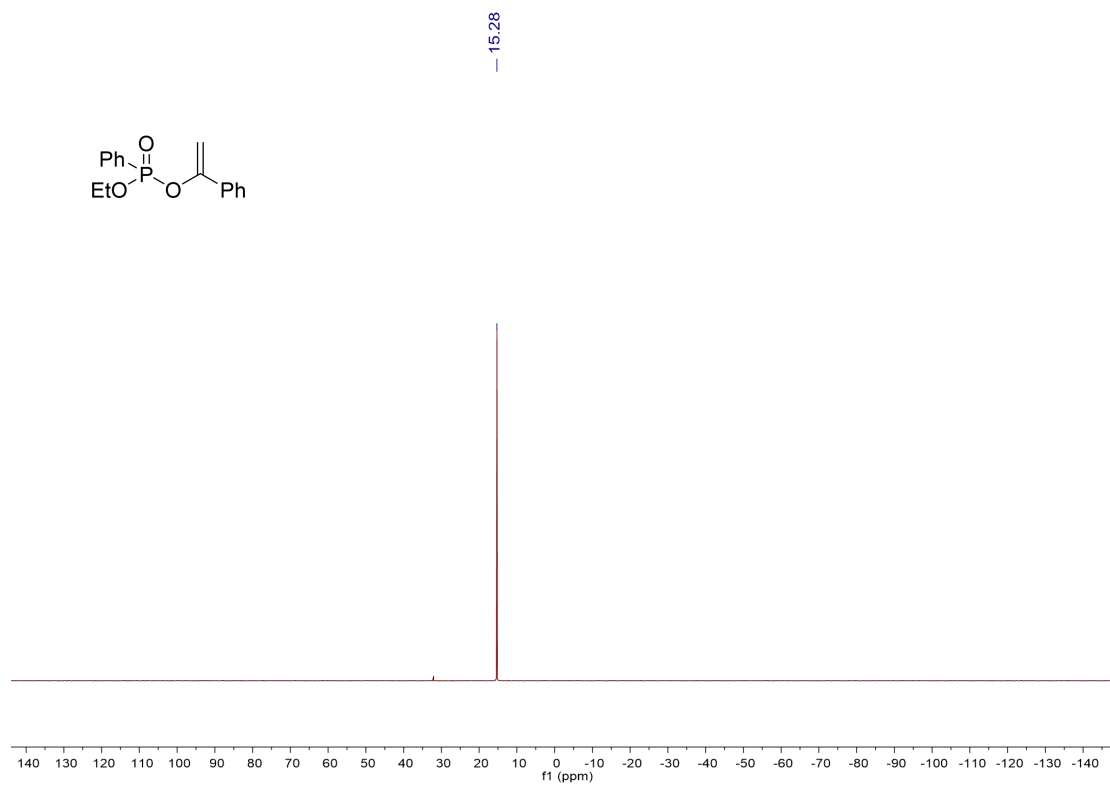
³¹P NMR of compound **3ah**



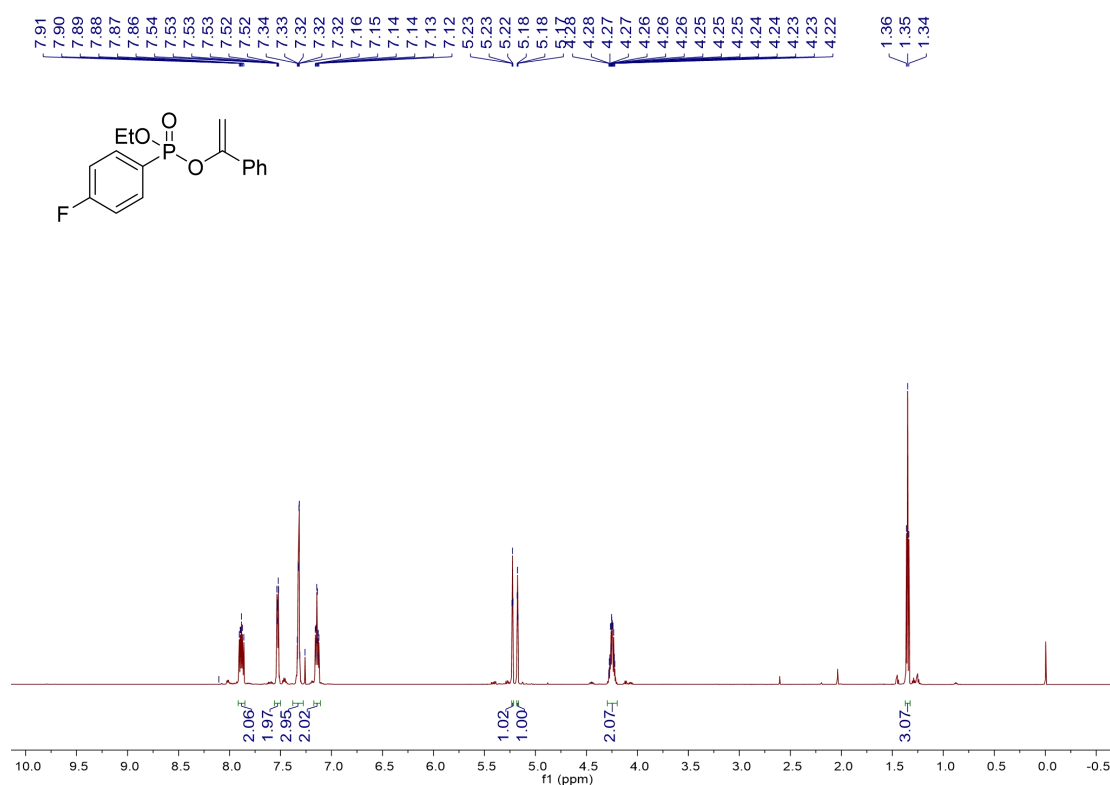
^1H NMR of compound **3ai**



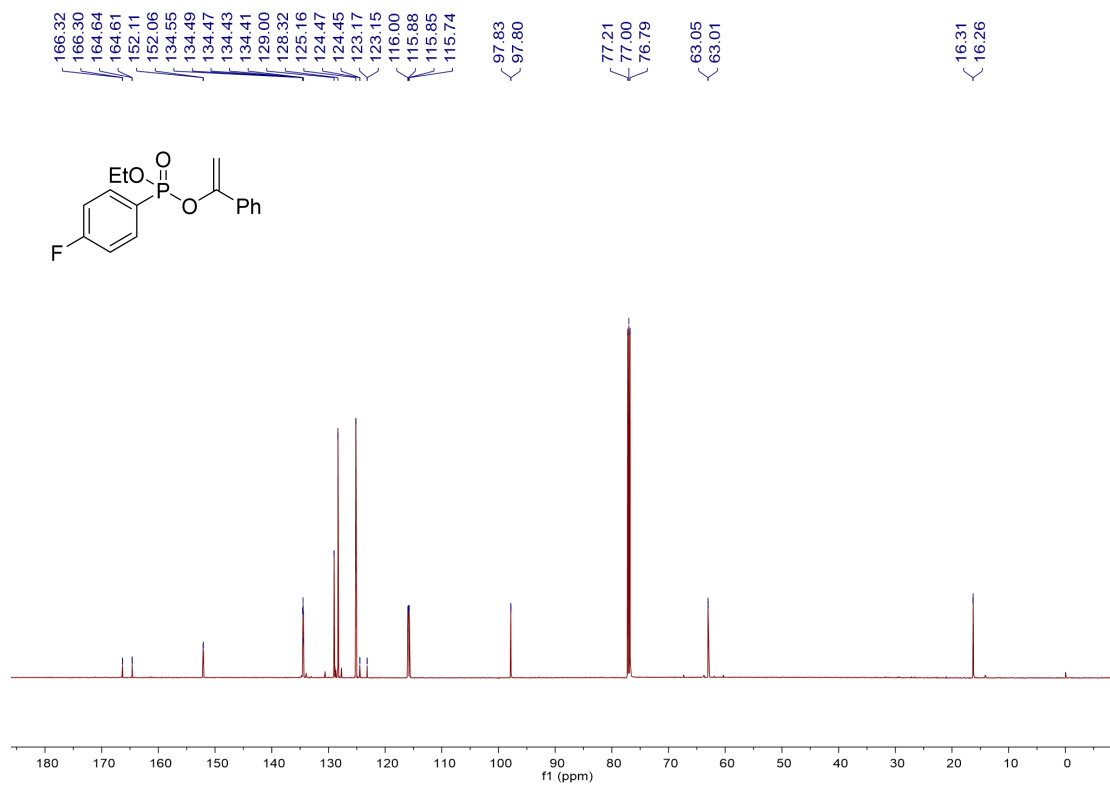
^{13}C NMR of compound **3ai**



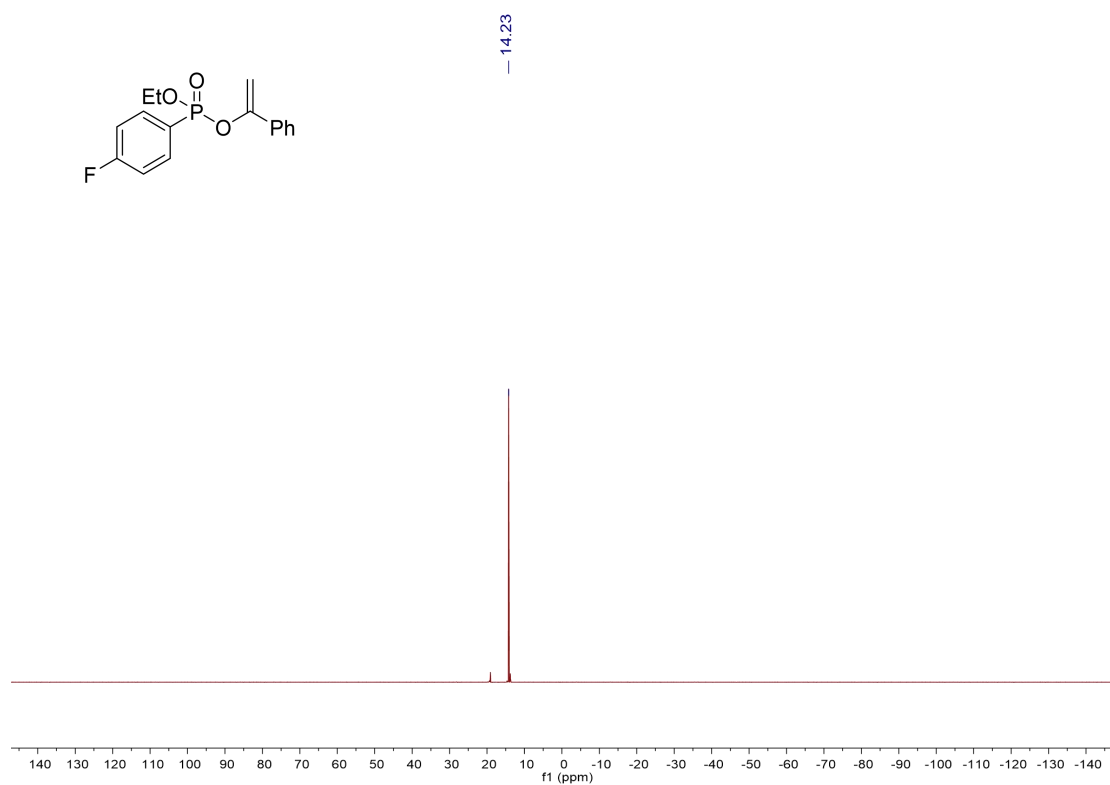
³¹P NMR of compound 3ai



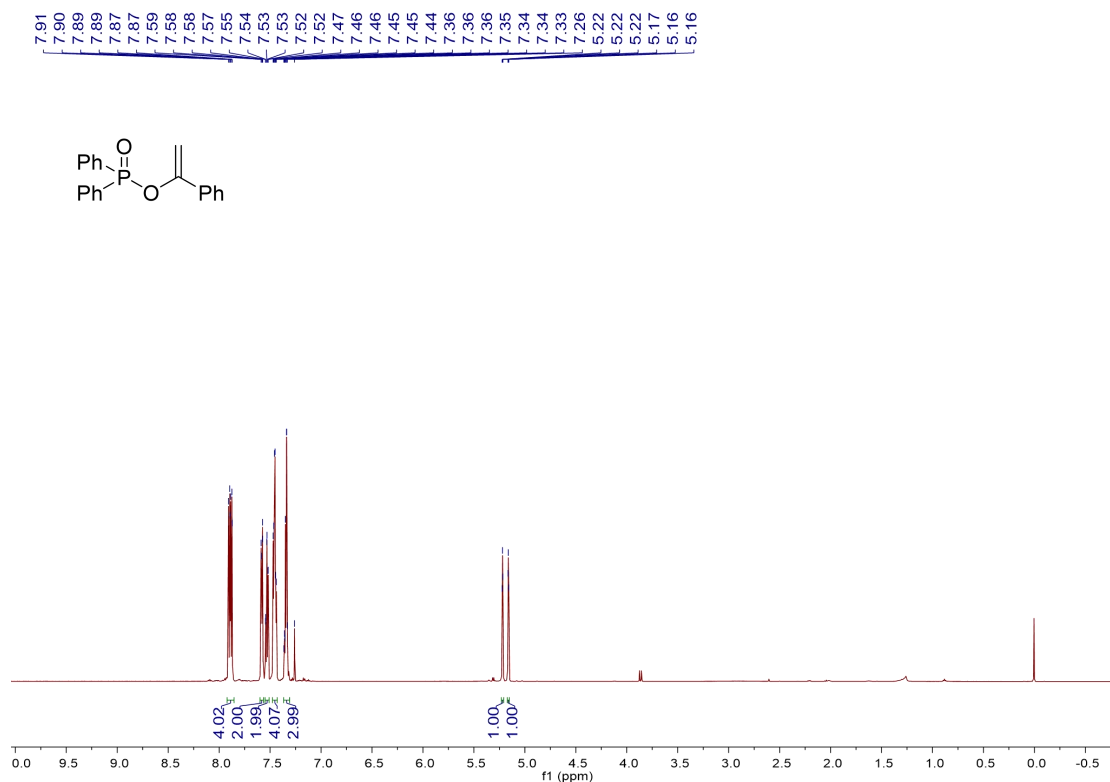
¹H NMR of compound 3aj



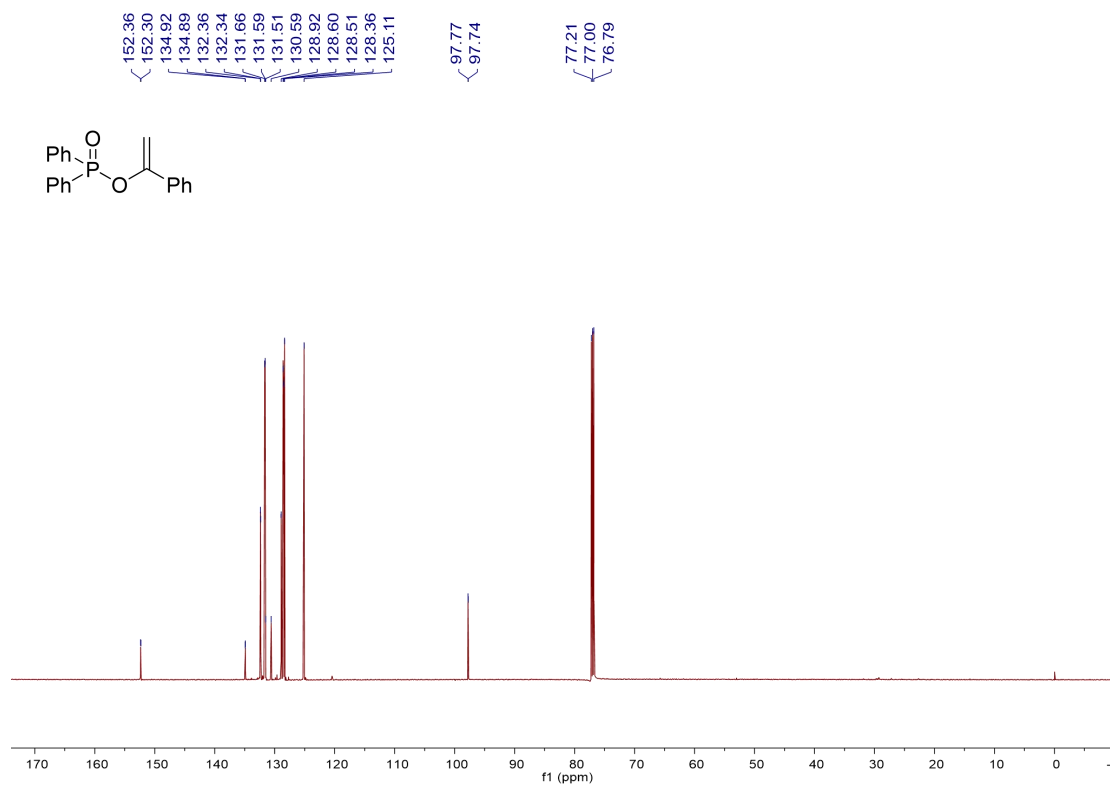
^{13}C NMR of compound 3aj



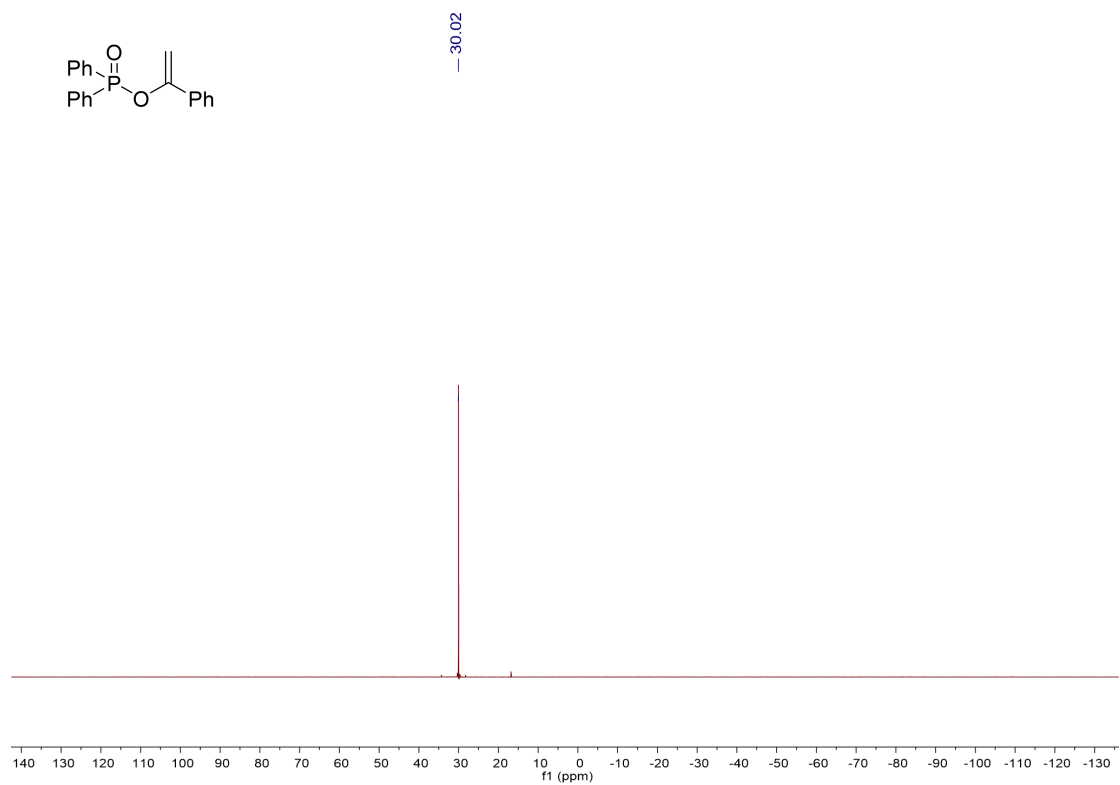
^{31}P NMR of compound 3aj



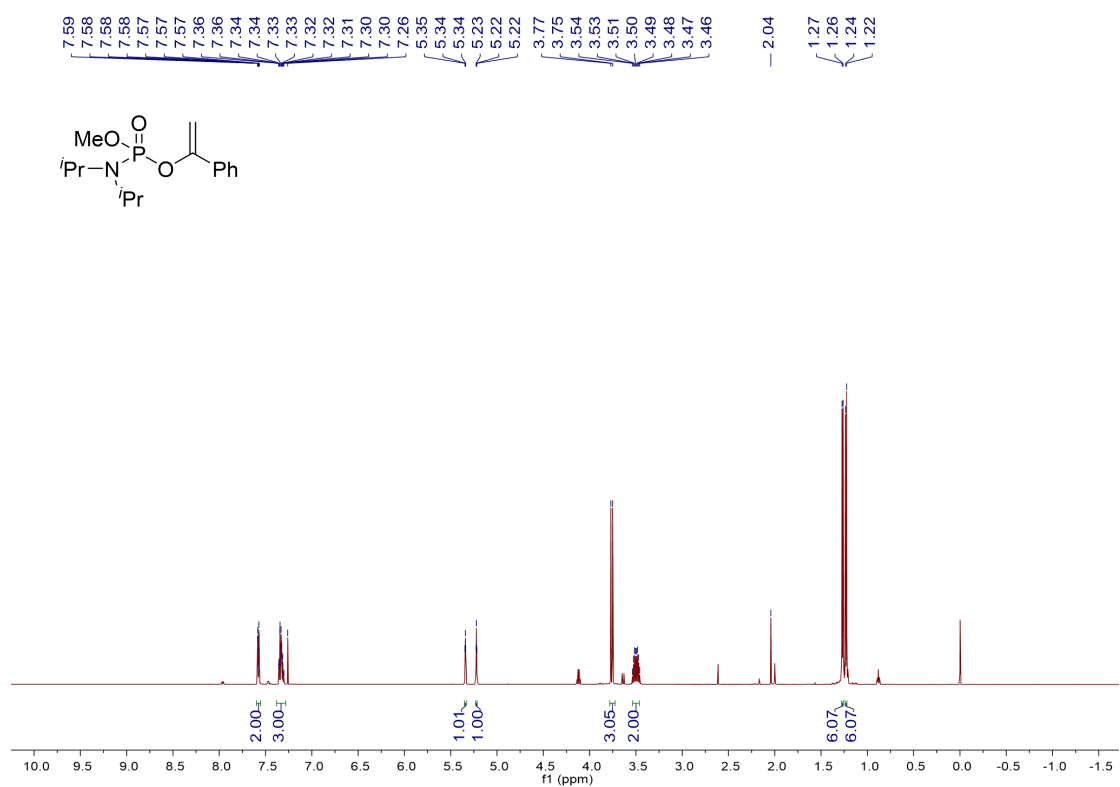
¹H NMR of compound **3ak**



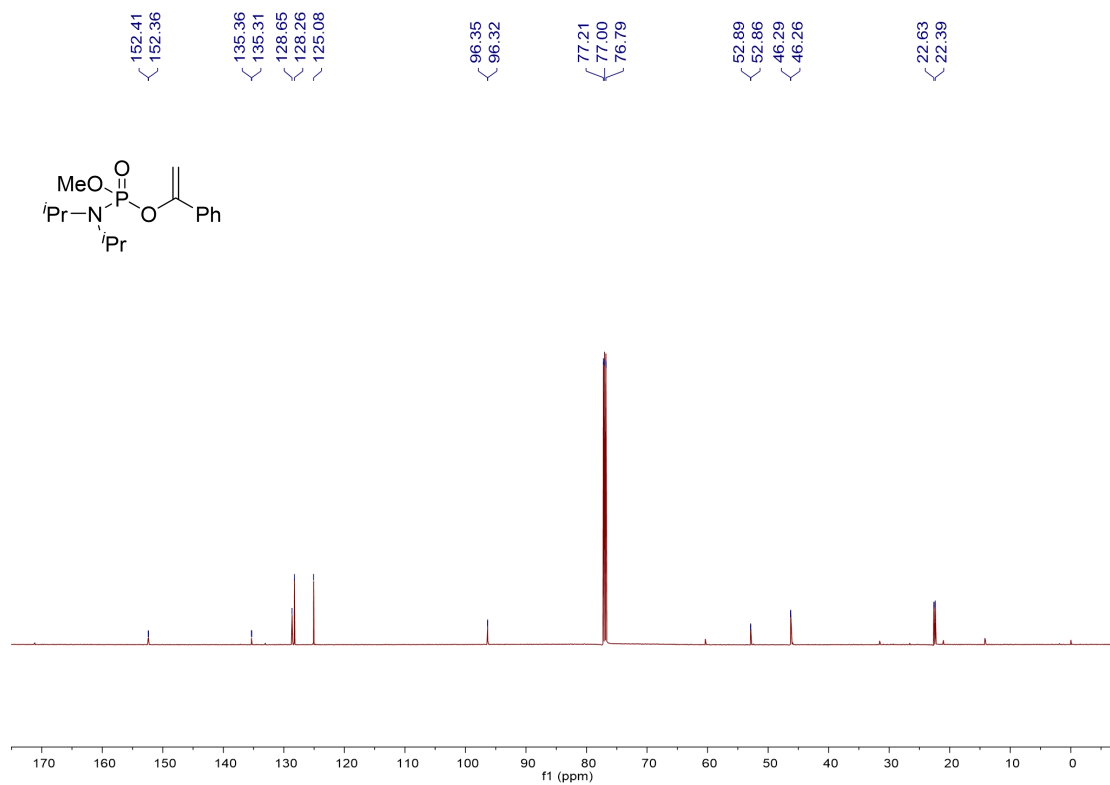
¹³C NMR of compound **3ak**



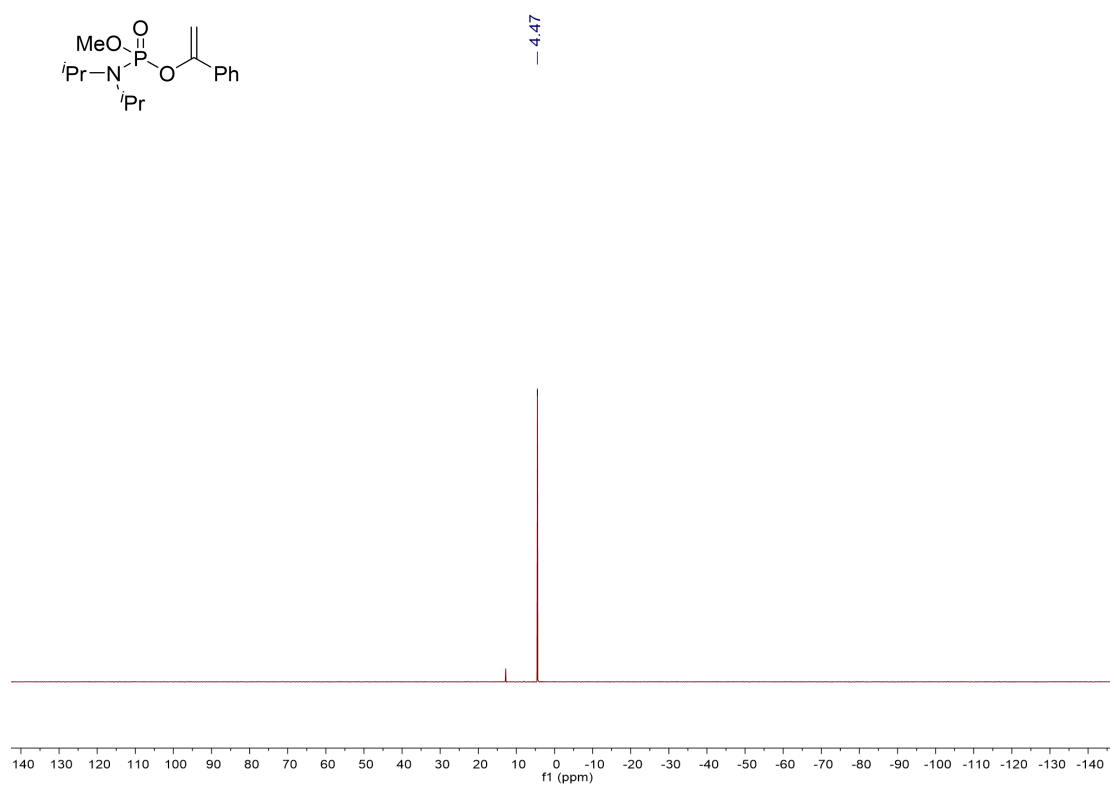
³¹P NMR of compound 3ak



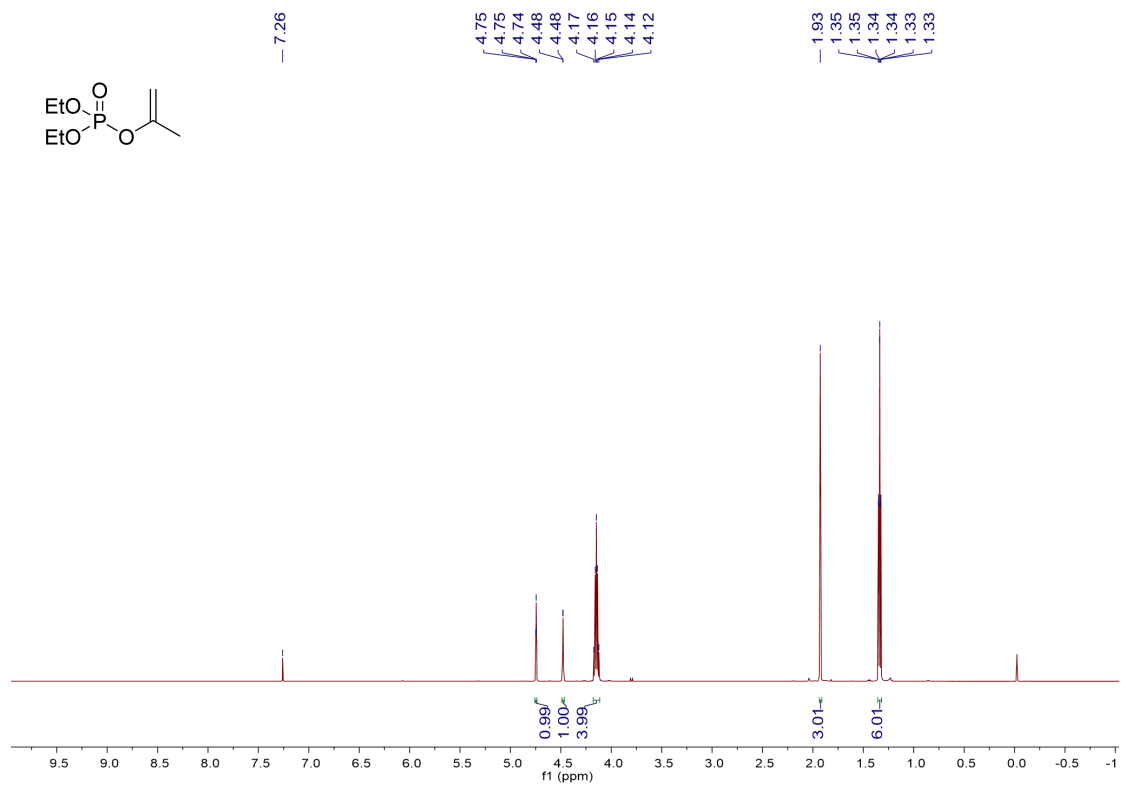
¹H NMR of compound 3al



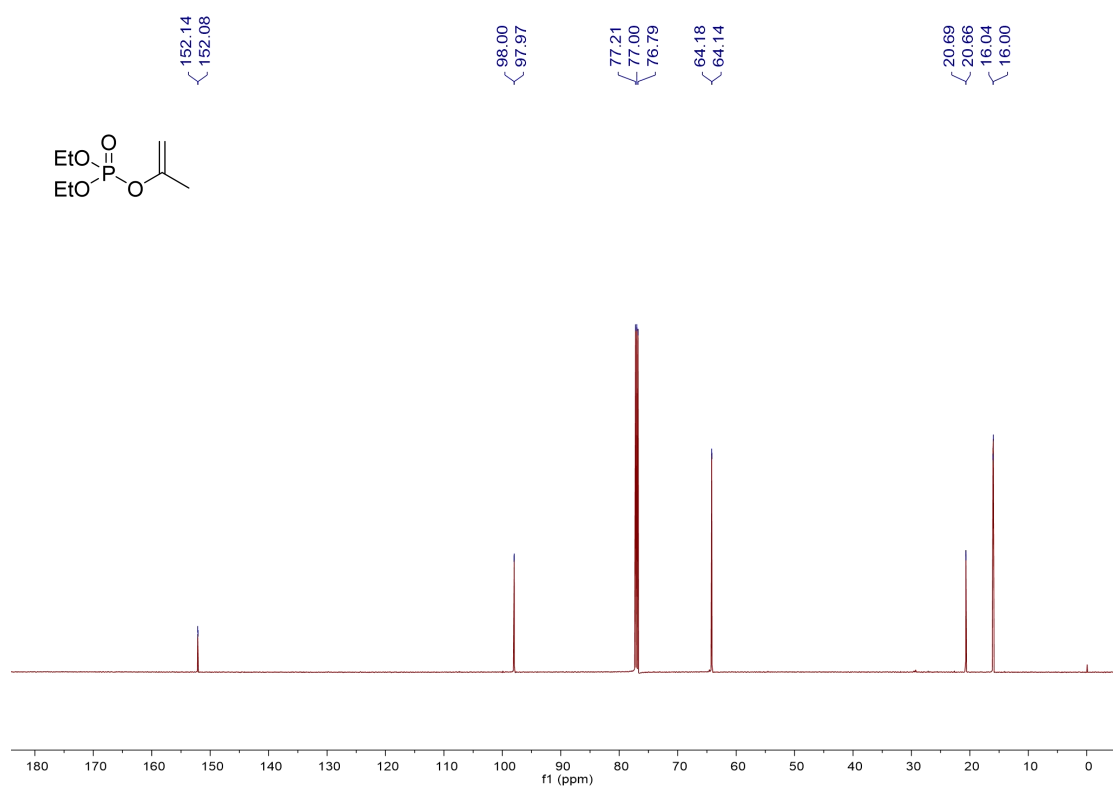
^{13}C NMR of compound **3al**



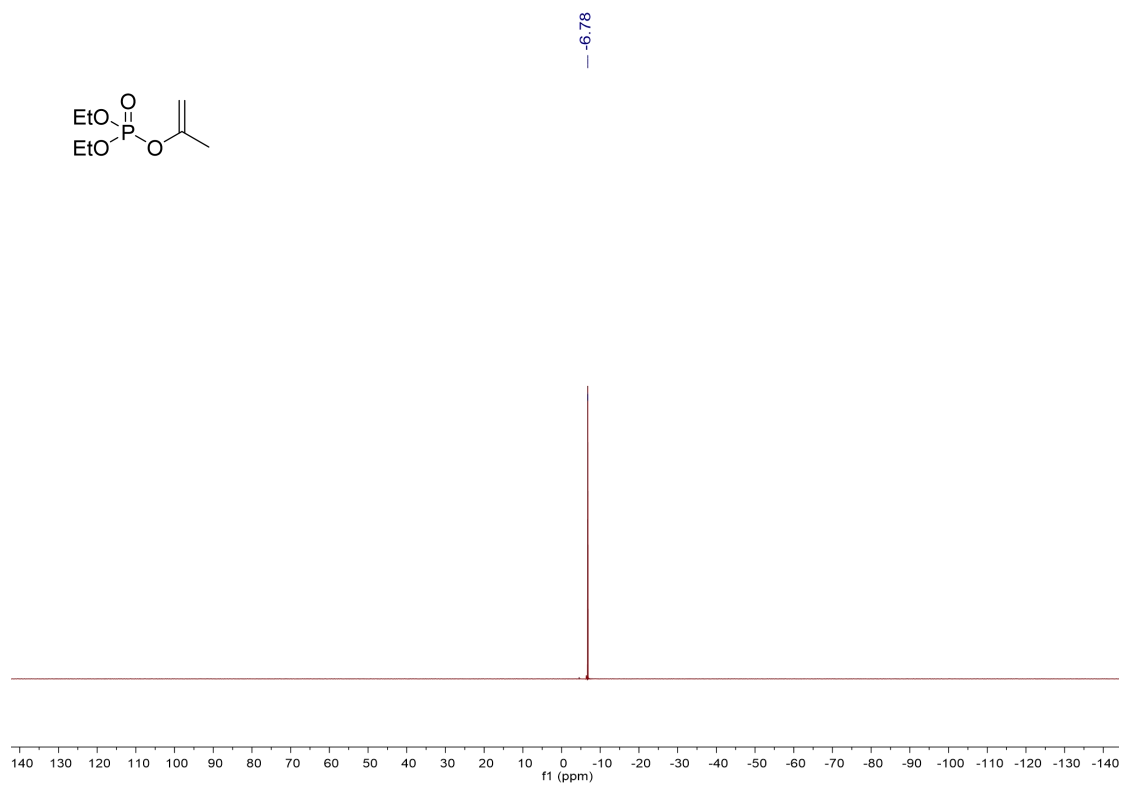
^{31}P NMR of compound **3al**



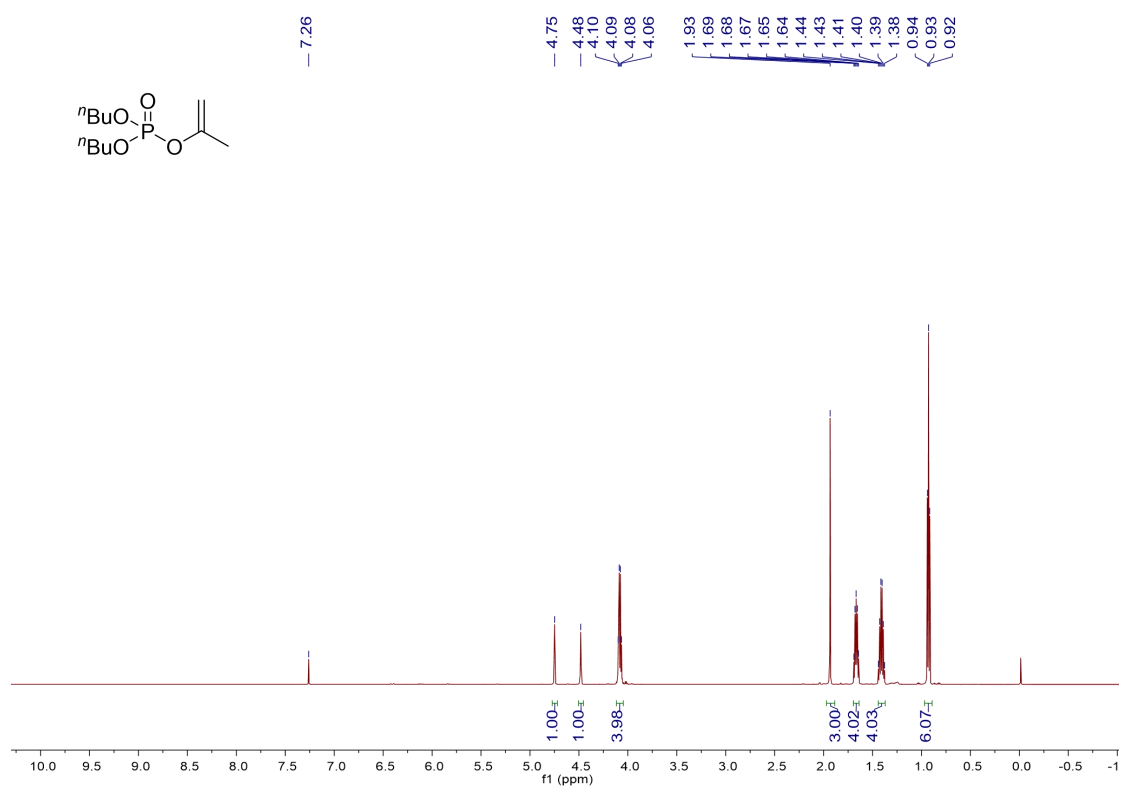
¹H NMR of compound **3ba**



¹³C NMR of compound **3ba**



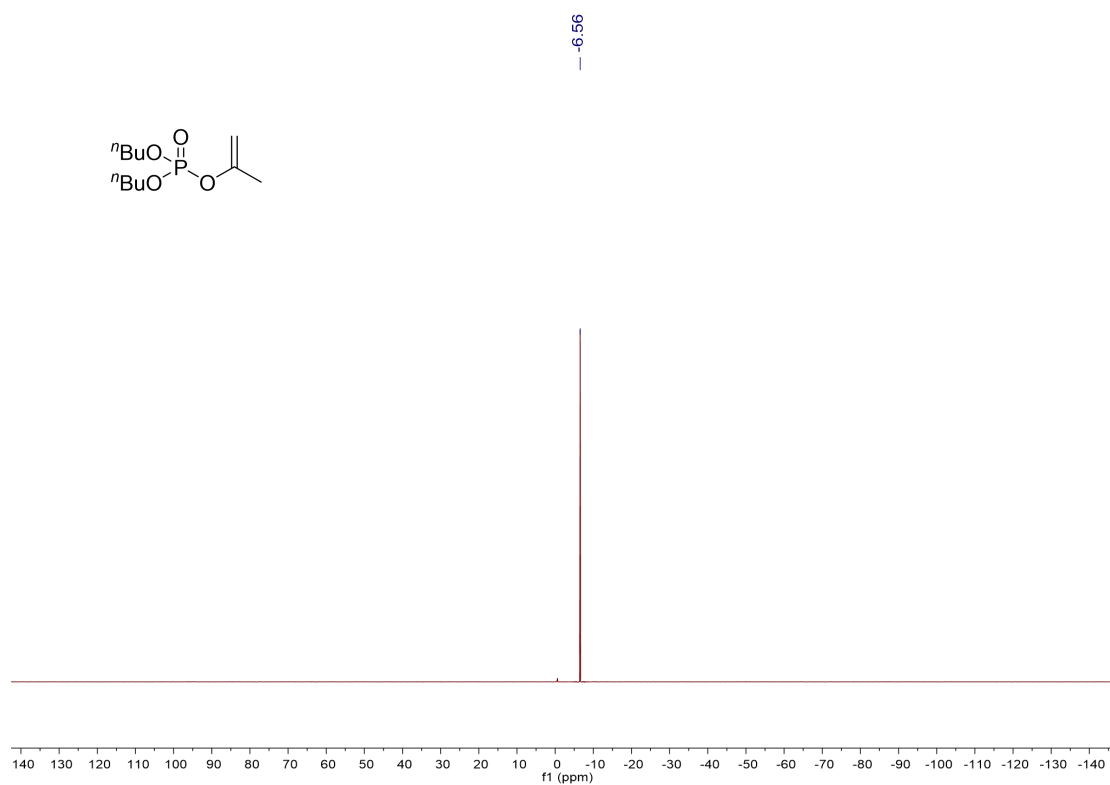
^{31}P NMR of compound **3ba**



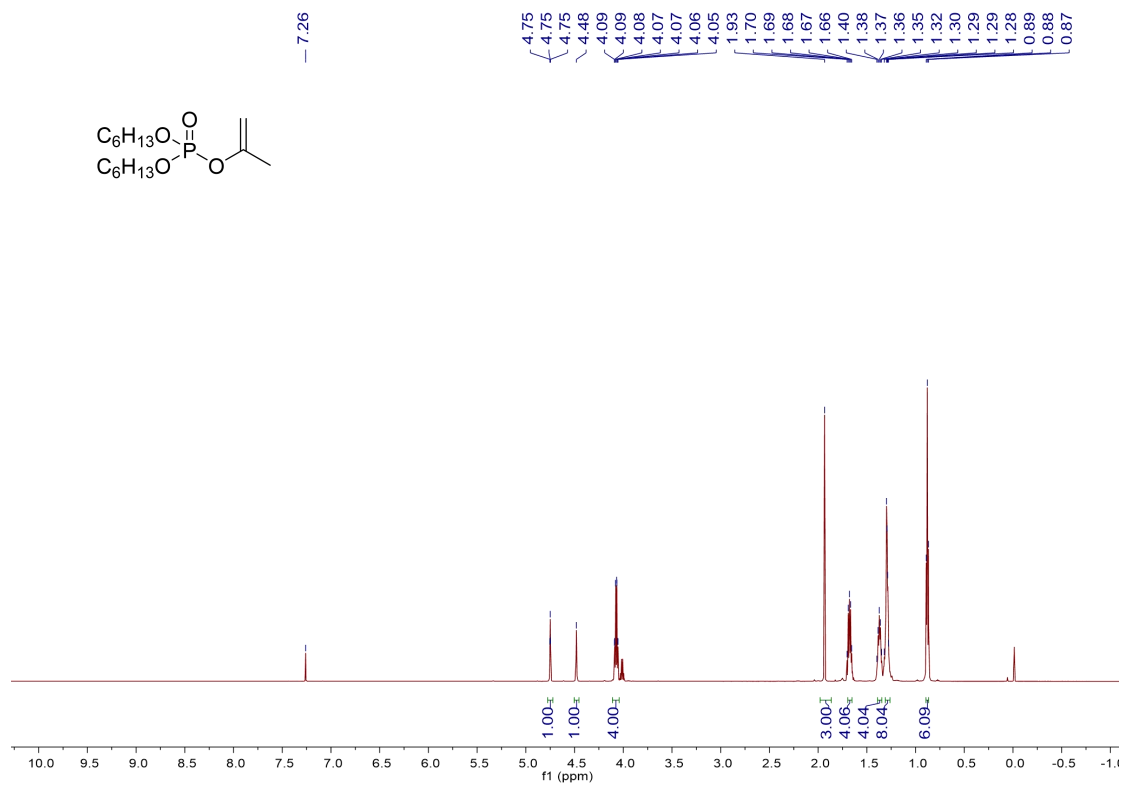
^1H NMR of compound **3bb**



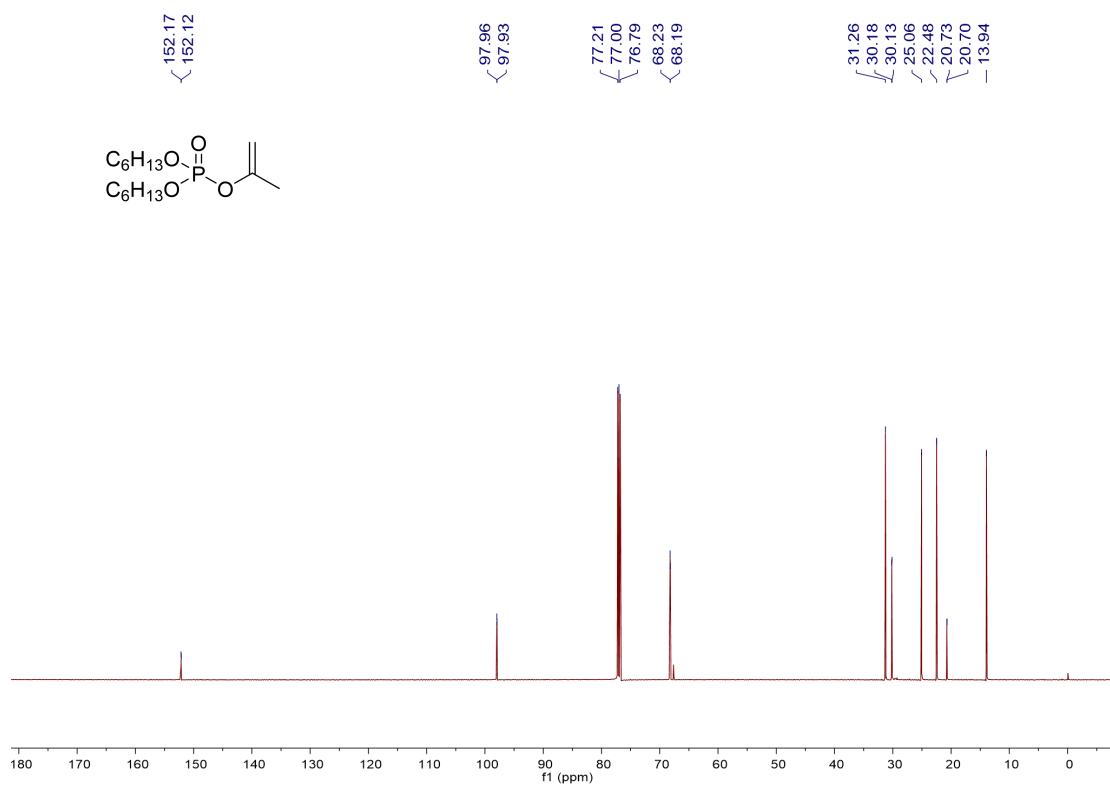
^{13}C NMR of compound **3bb**



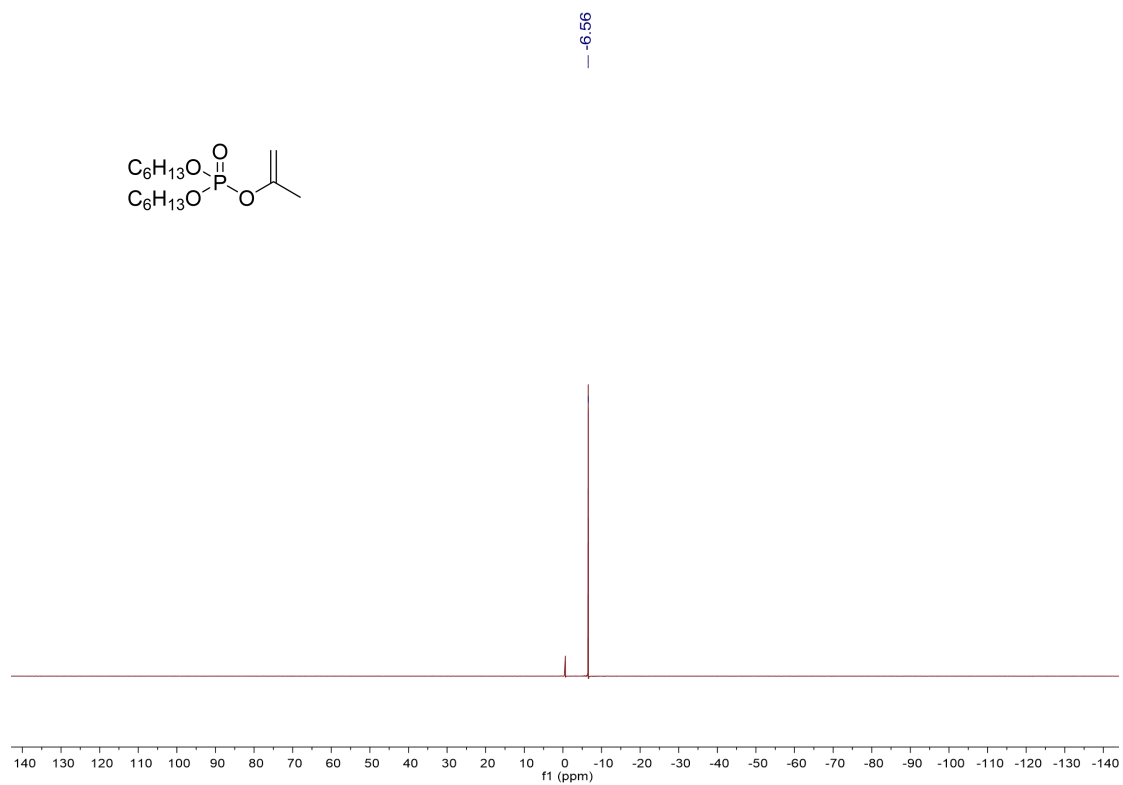
^{31}P NMR of compound **3bb**



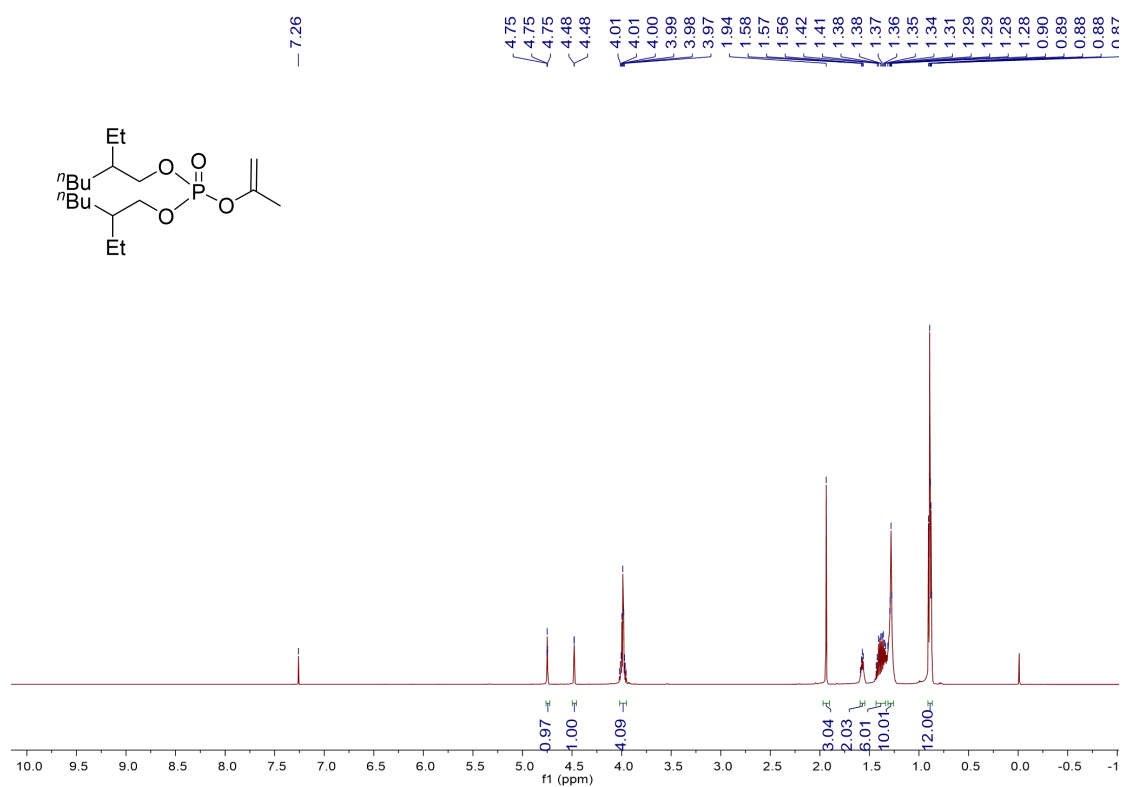
¹H NMR of compound 3bc



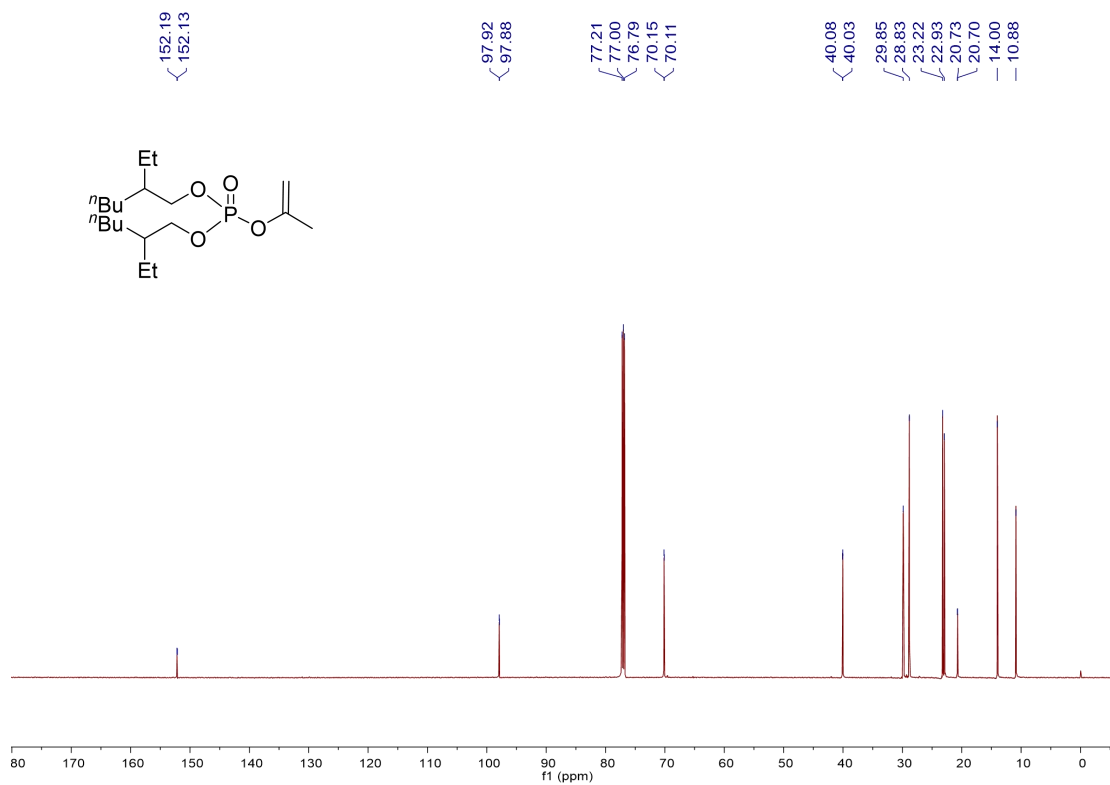
¹³C NMR of compound 3bc



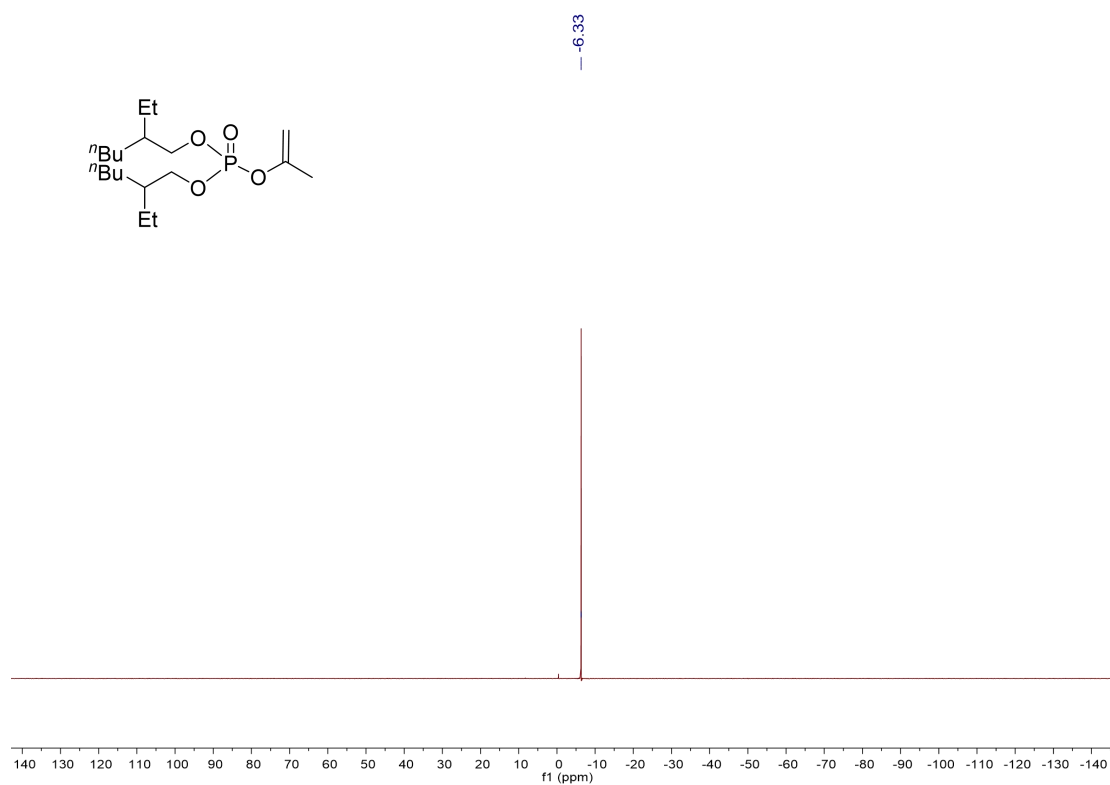
^{31}P NMR of compound **3bc**



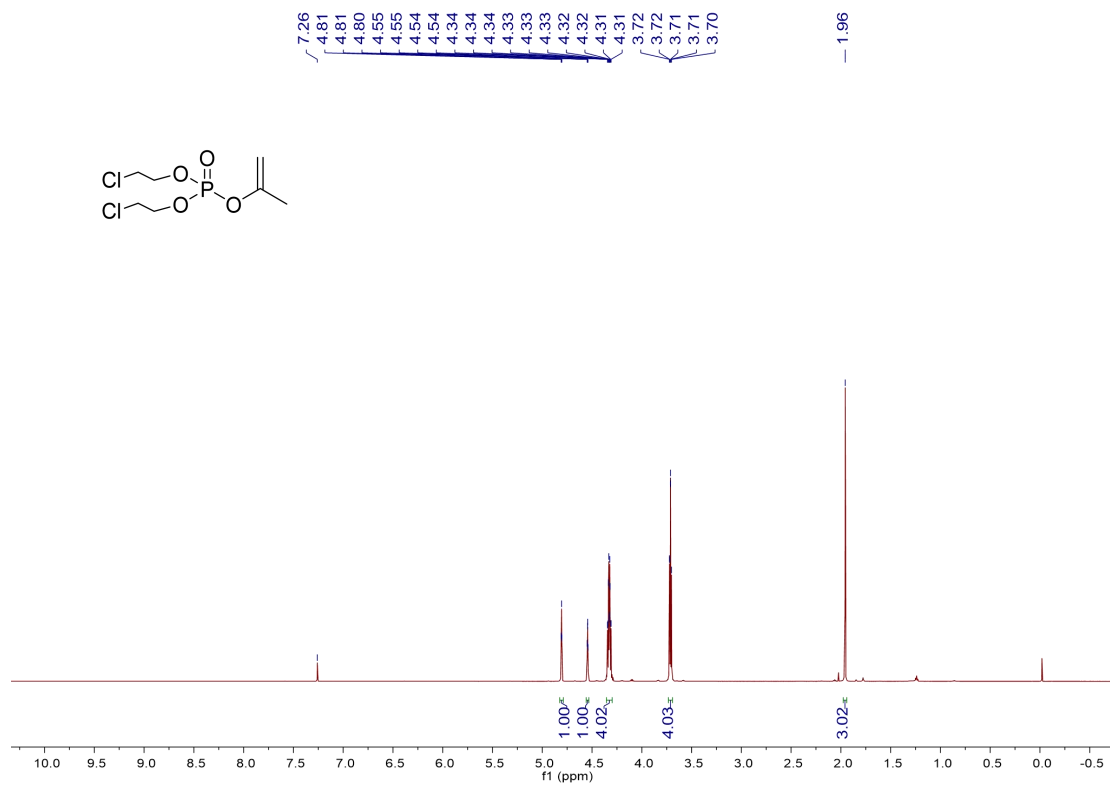
^1H NMR of compound **3bd**



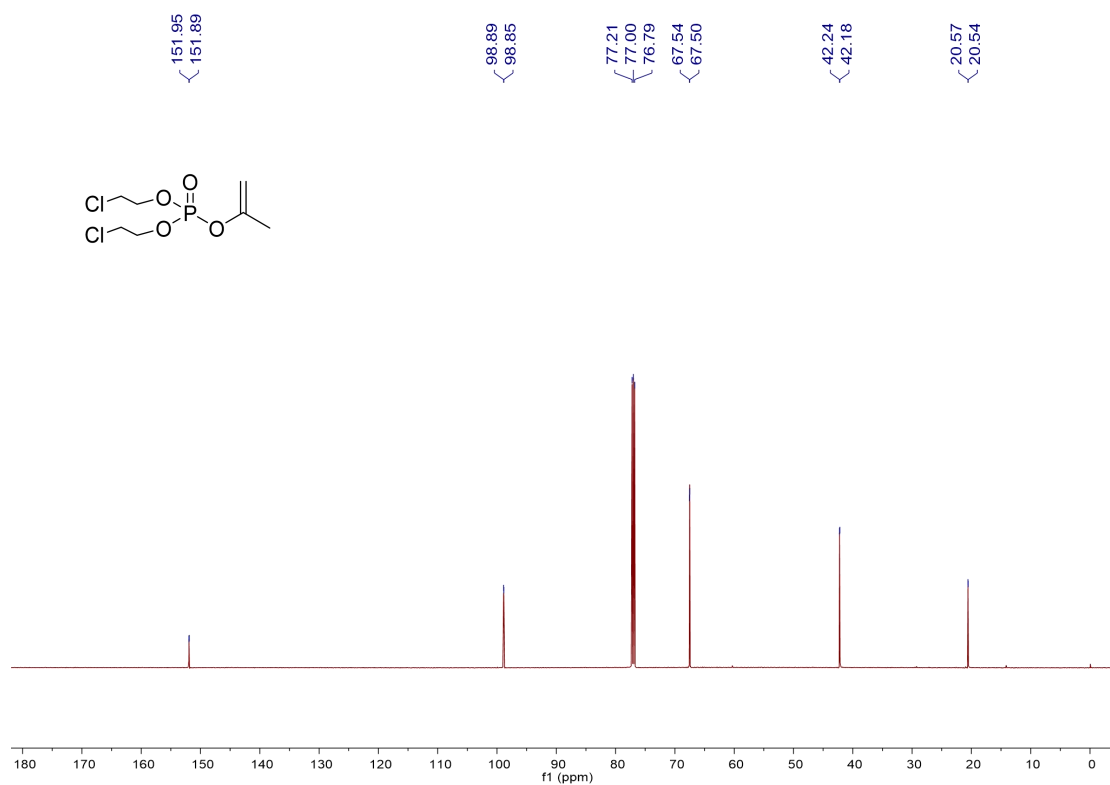
^{13}C NMR of compound **3bd**



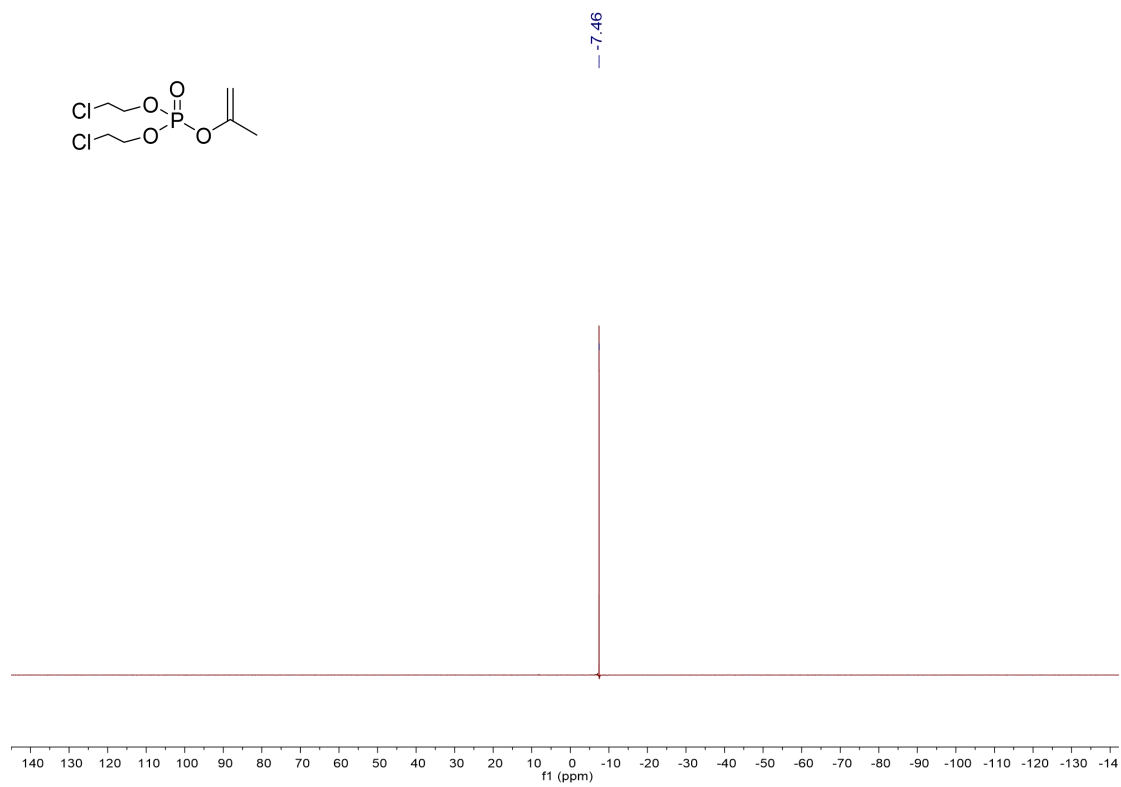
^{31}P NMR of compound **3bd**



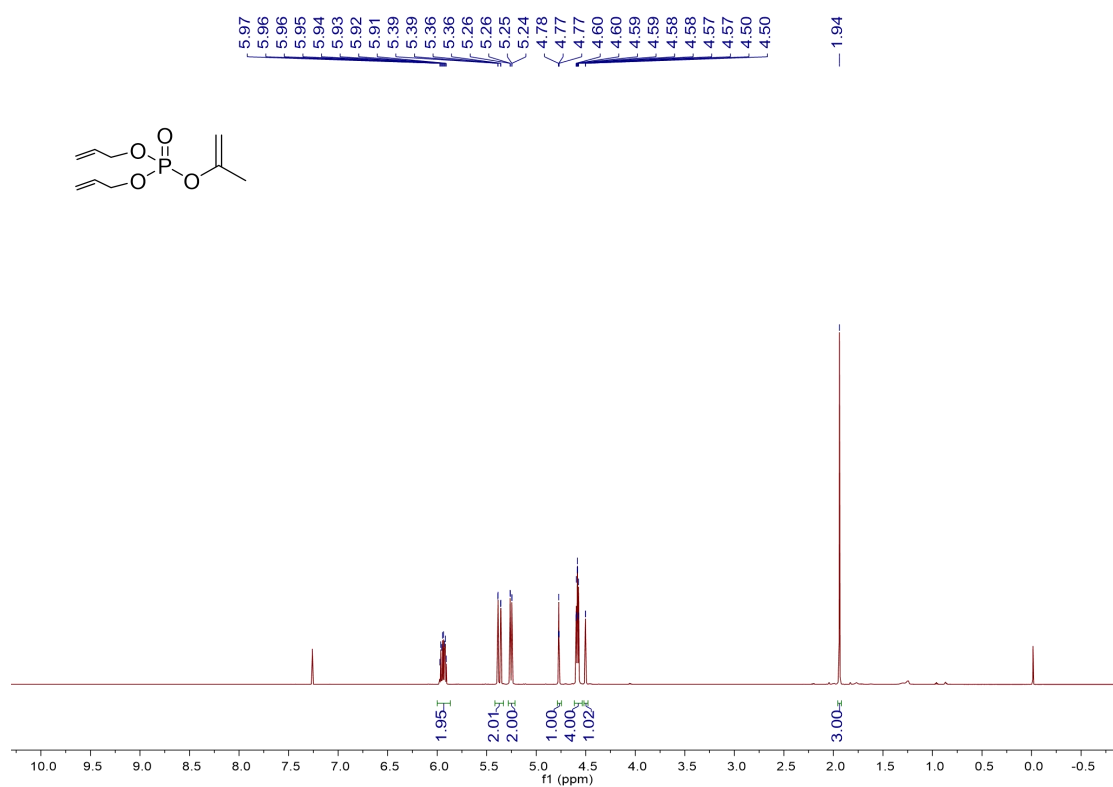
¹H NMR of compound **3be**



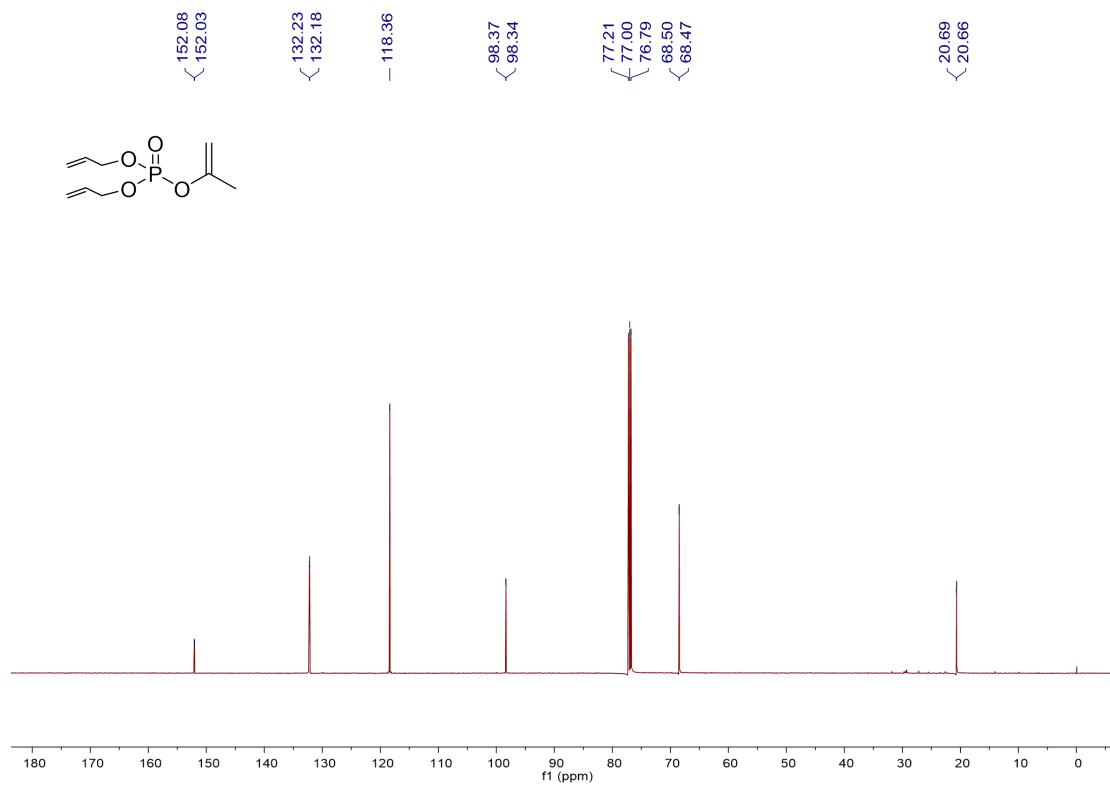
¹³C NMR of compound **3be**



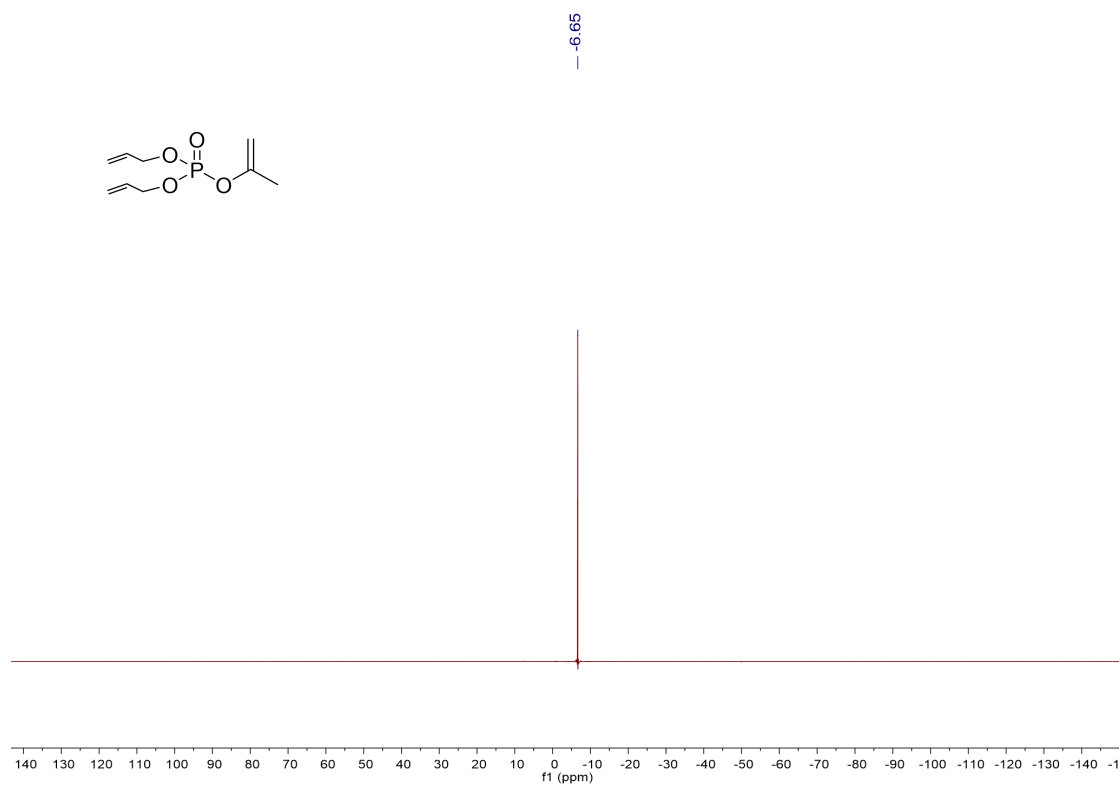
³¹P NMR of compound **3be**



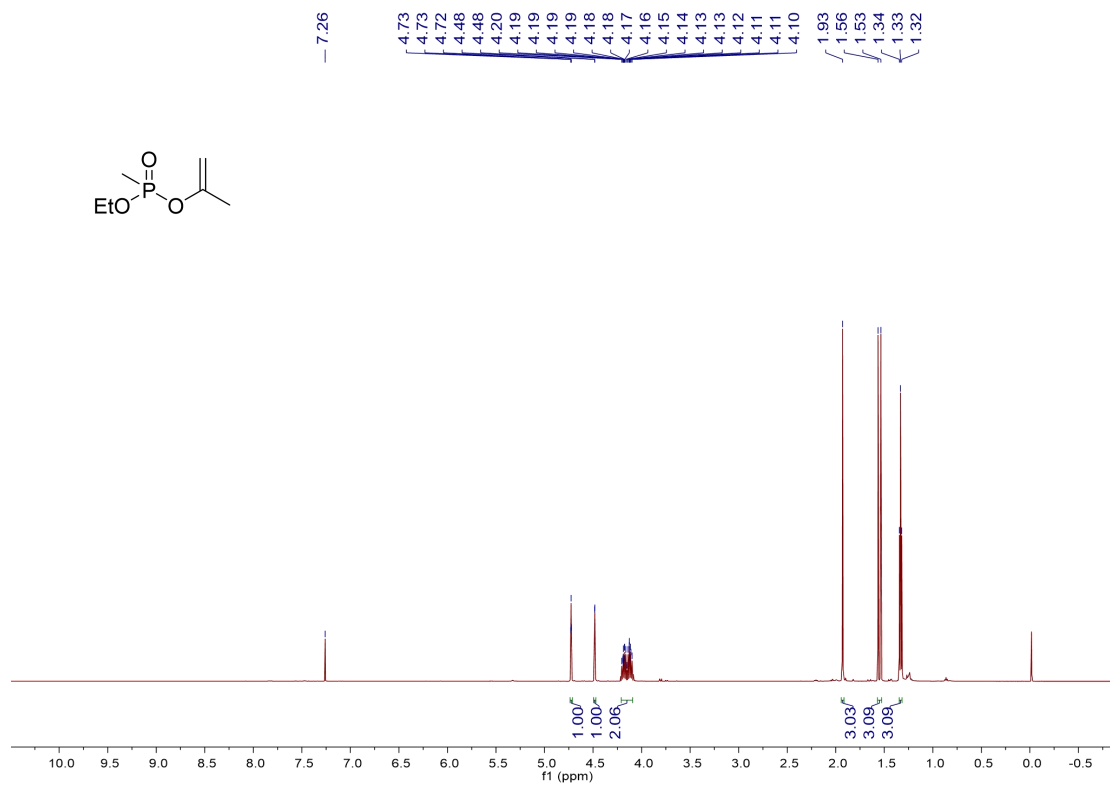
¹H NMR of compound **3bf**



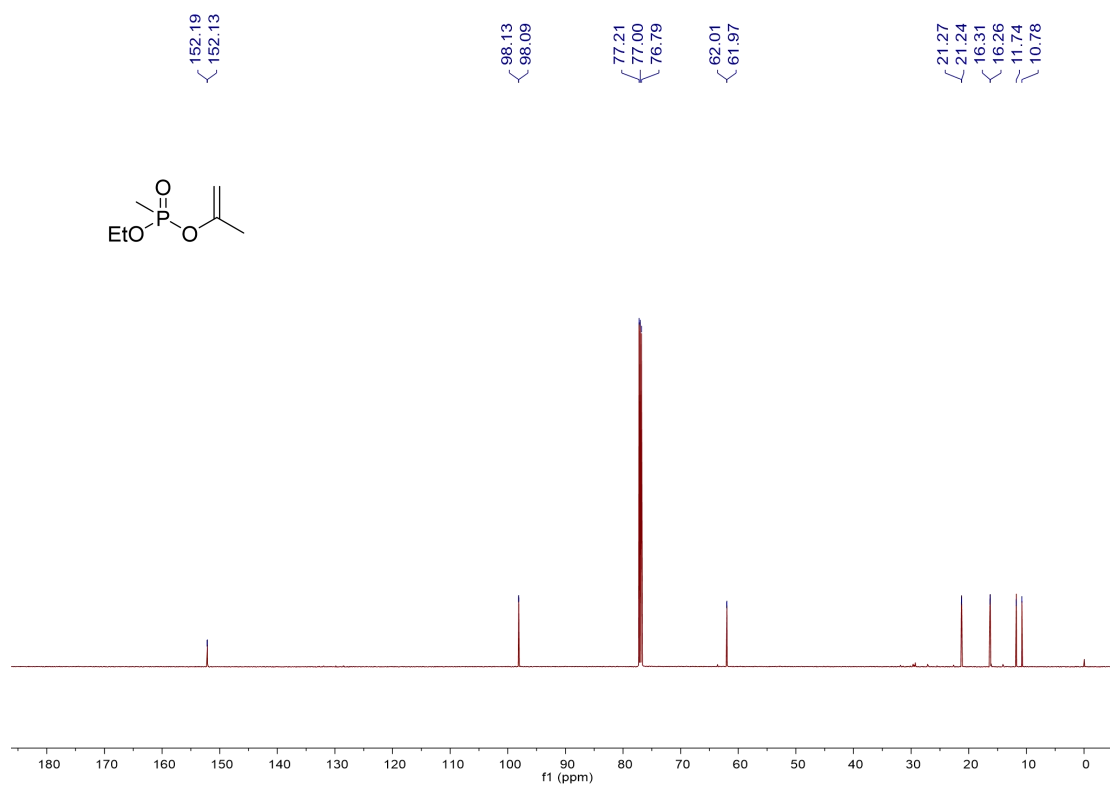
^{13}C NMR of compound **3bf**



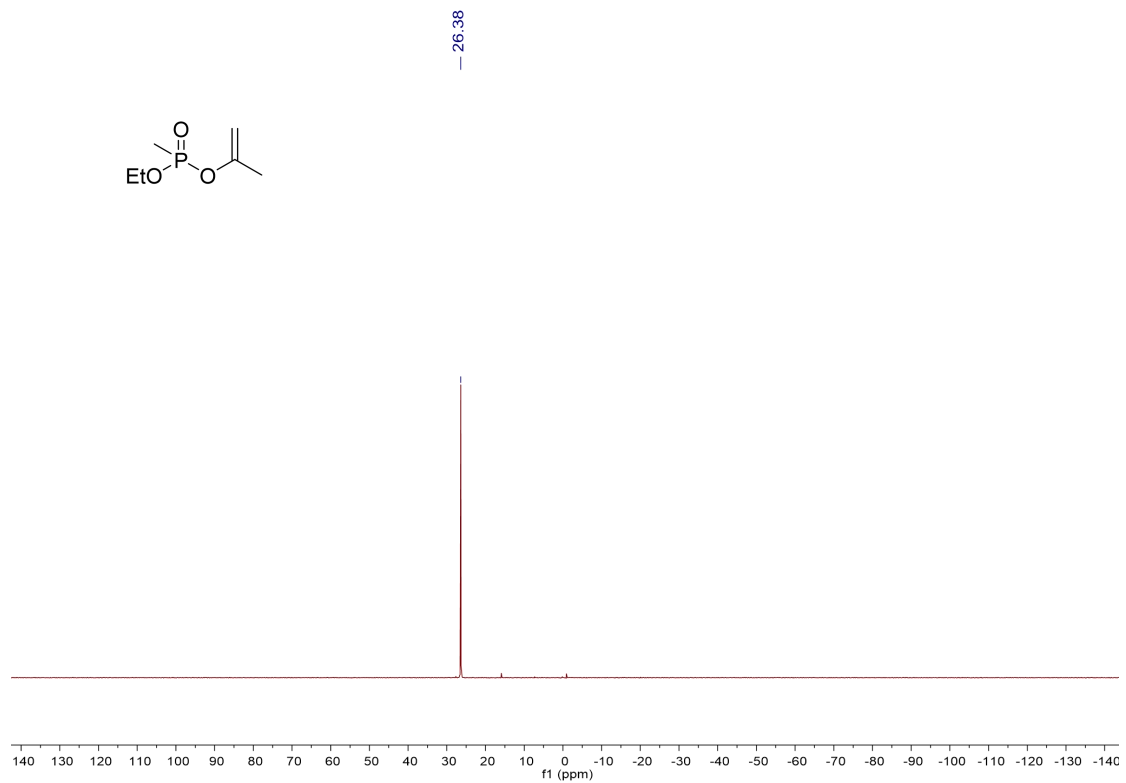
^{31}P NMR of compound **3bf**



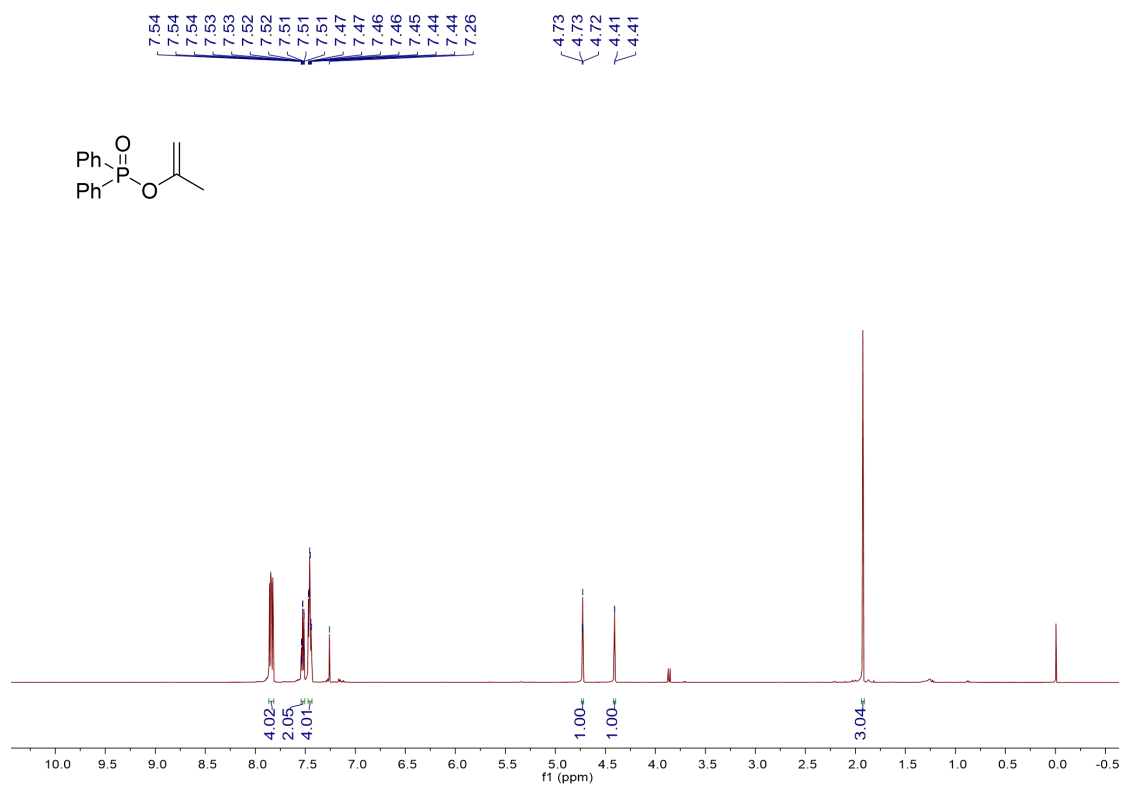
^1H NMR of compound **3bg**



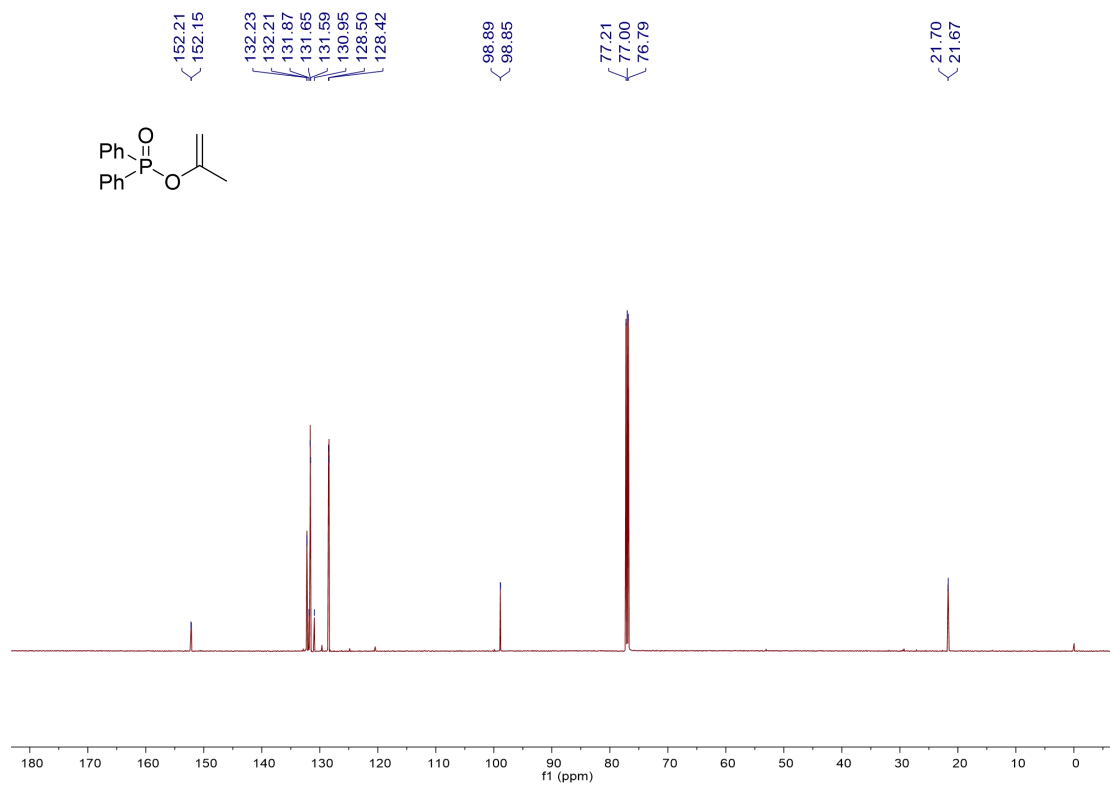
^{13}C NMR of compound **3bg**



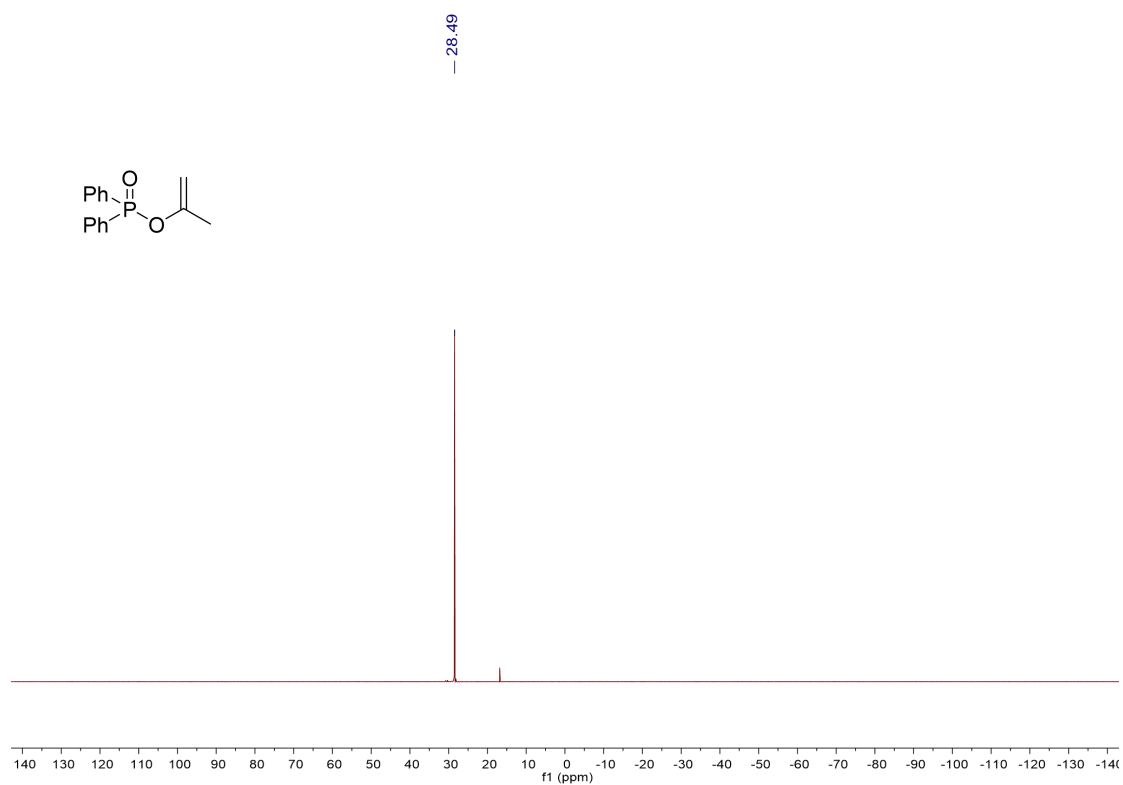
^{31}P NMR of compound **3bg**



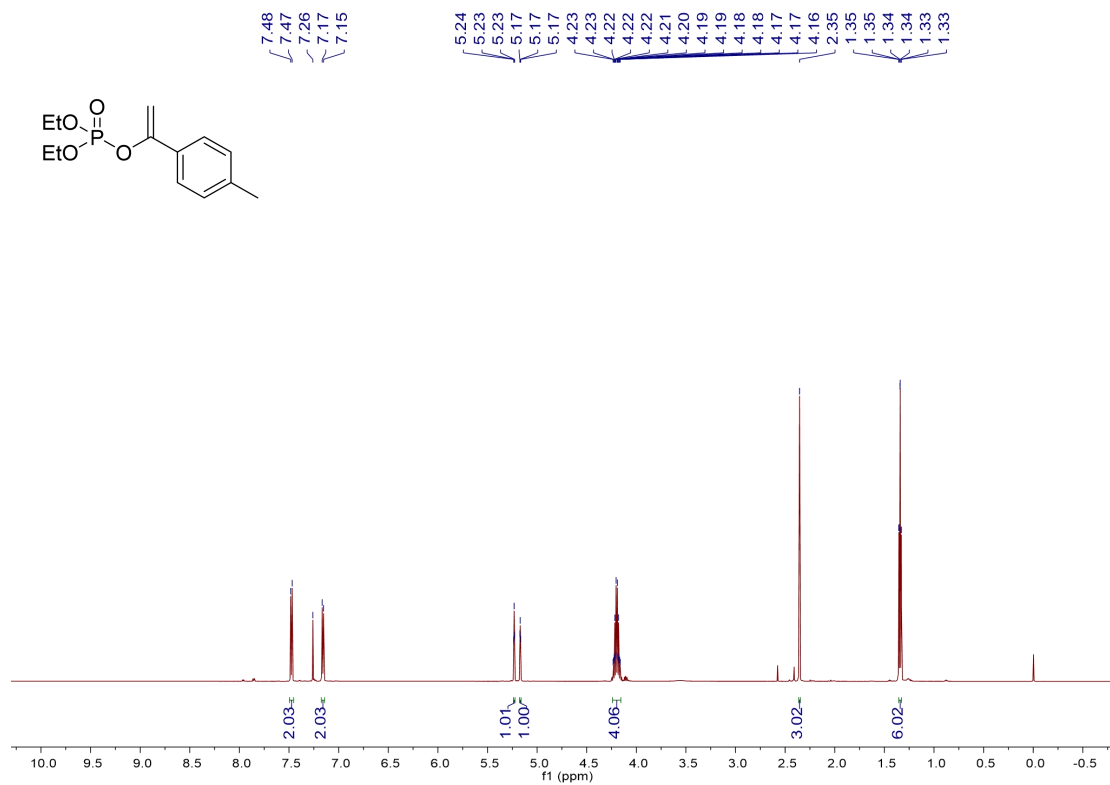
^1H NMR of compound **3bh**



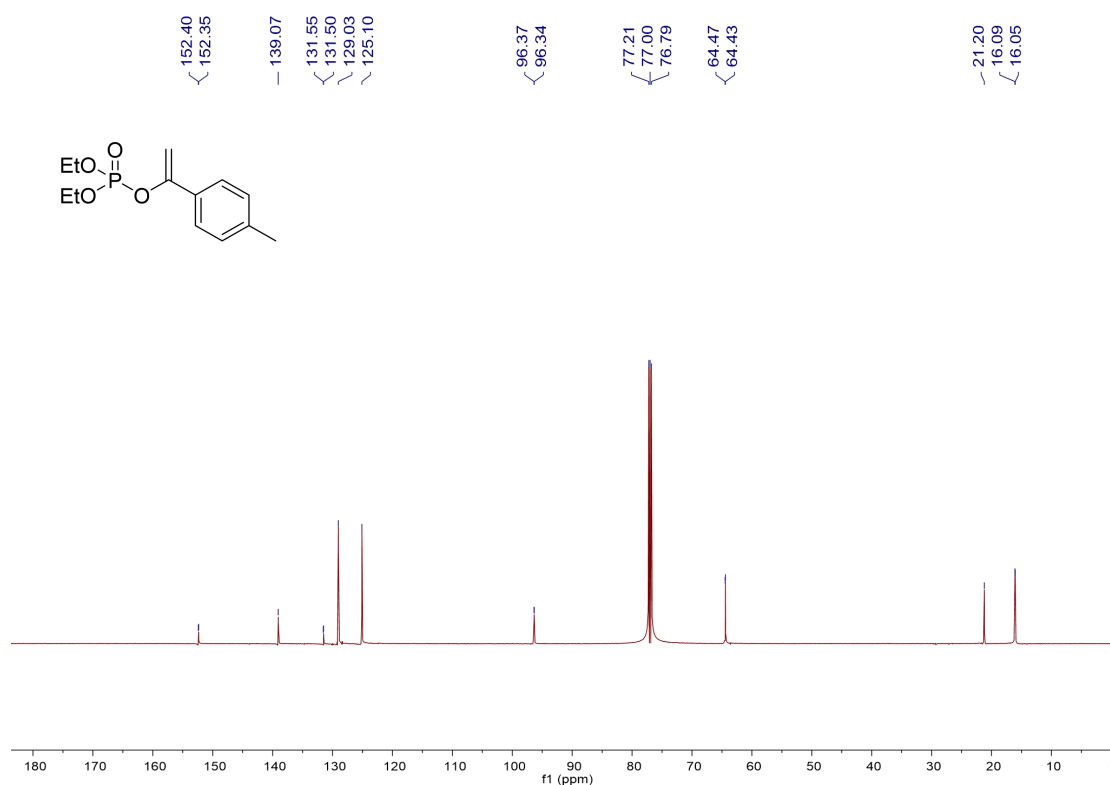
¹³C NMR of compound **3bh**



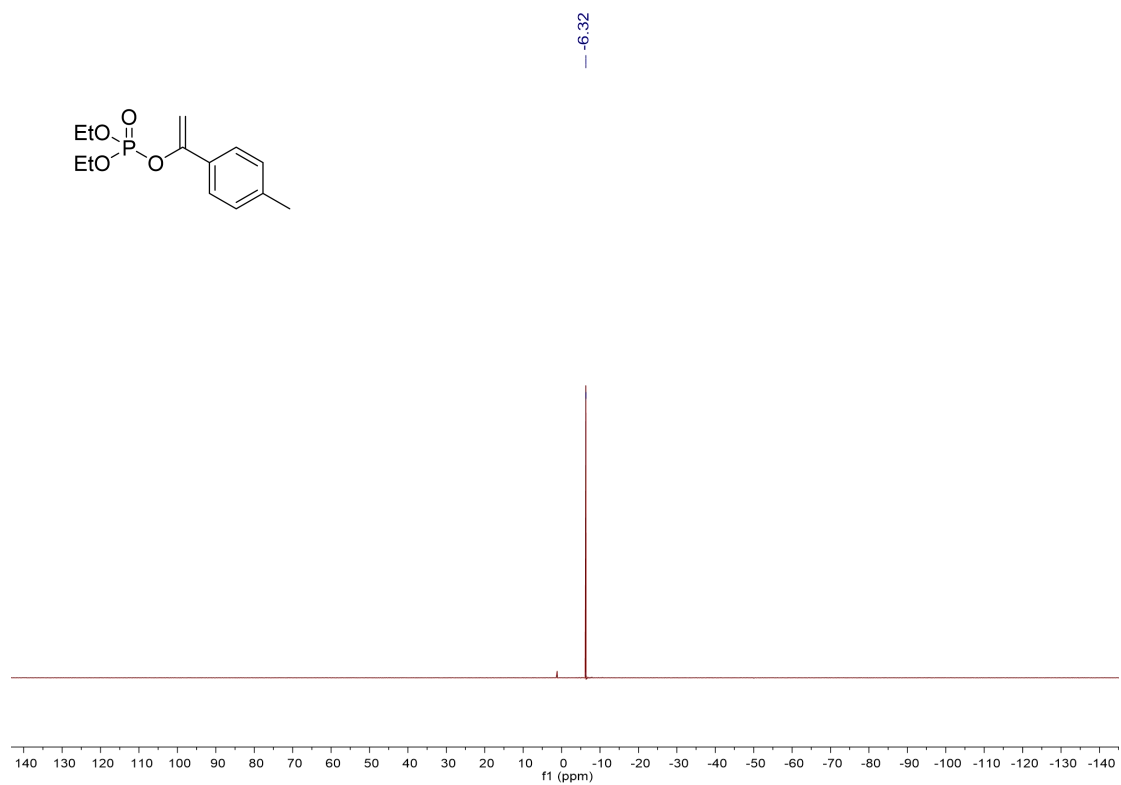
³¹P NMR of compound **3bh**



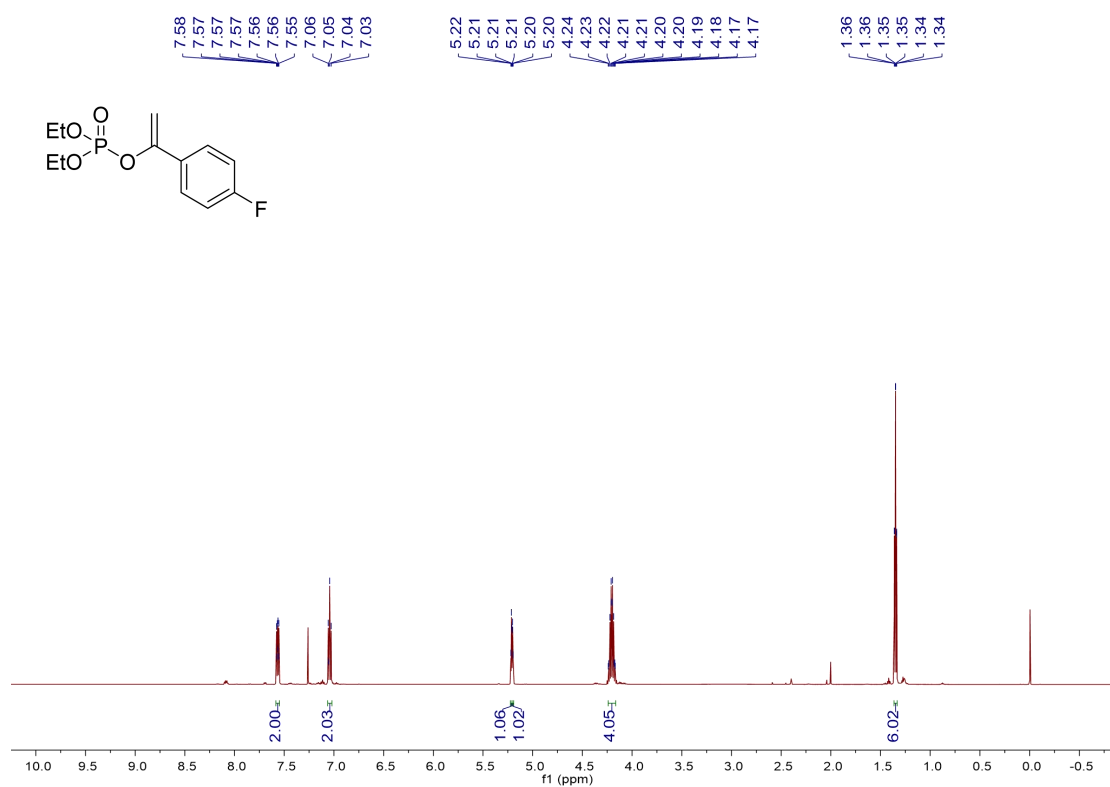
^1H NMR of compound **3ca**



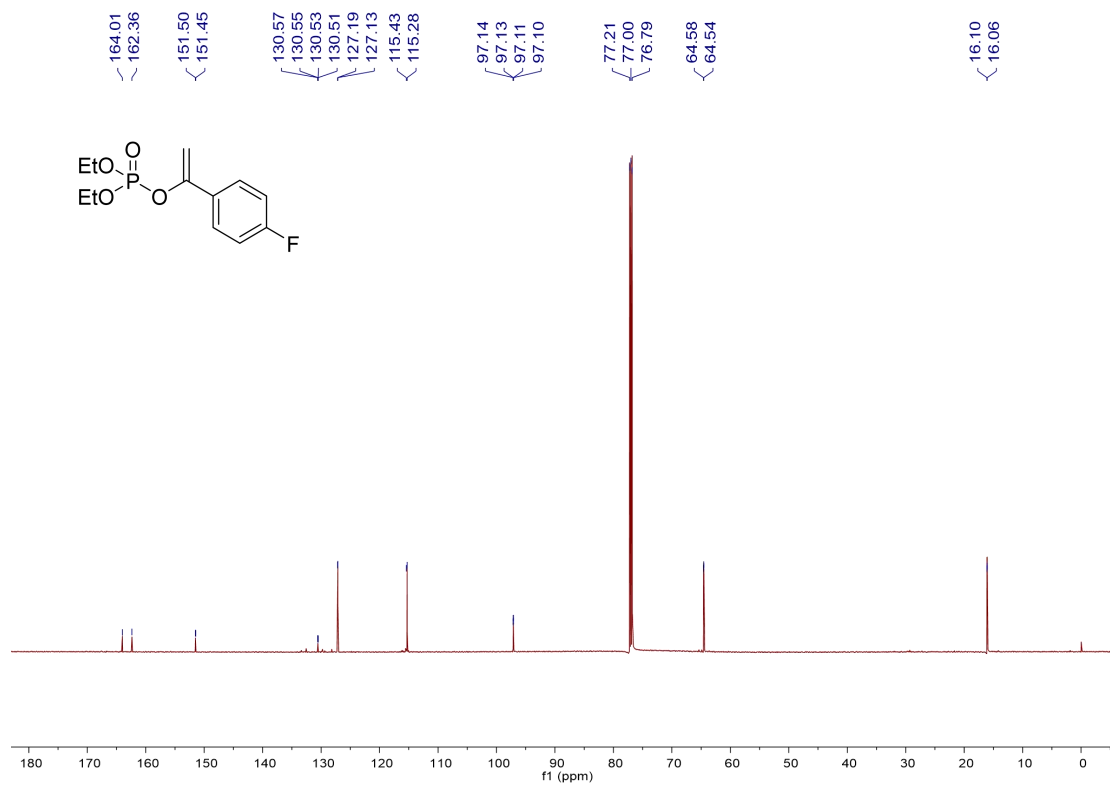
^{13}C NMR of compound **3ca**



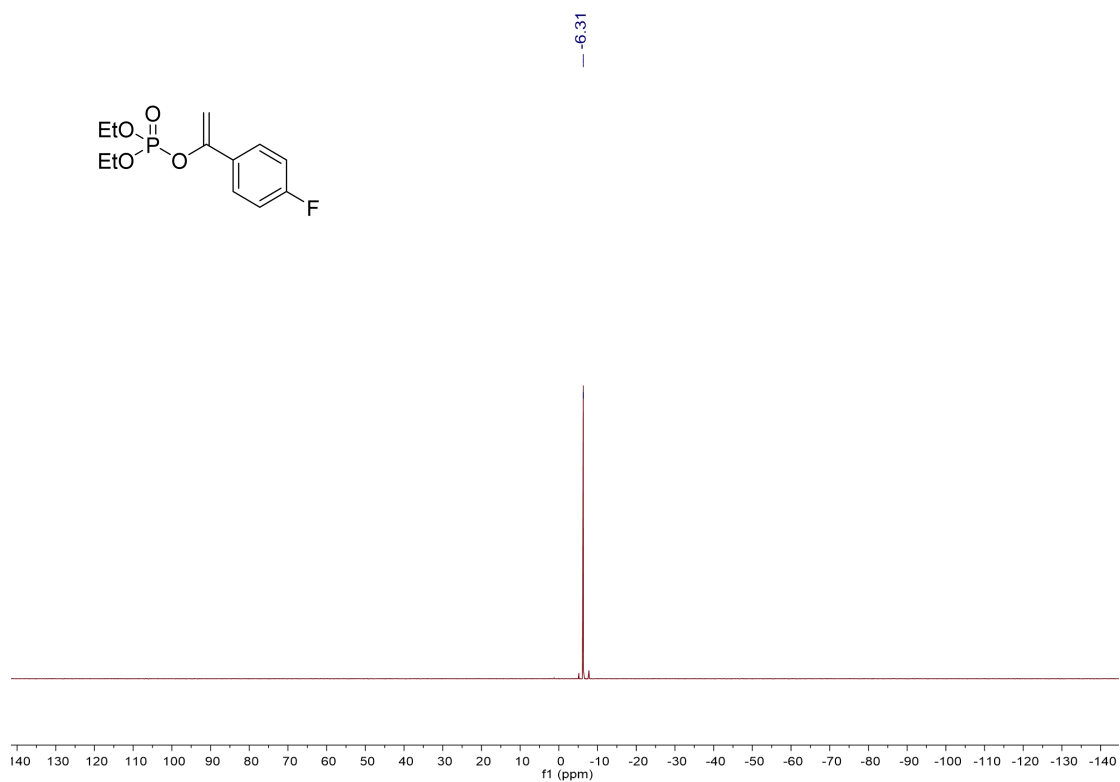
^{31}P NMR of compound 3ca



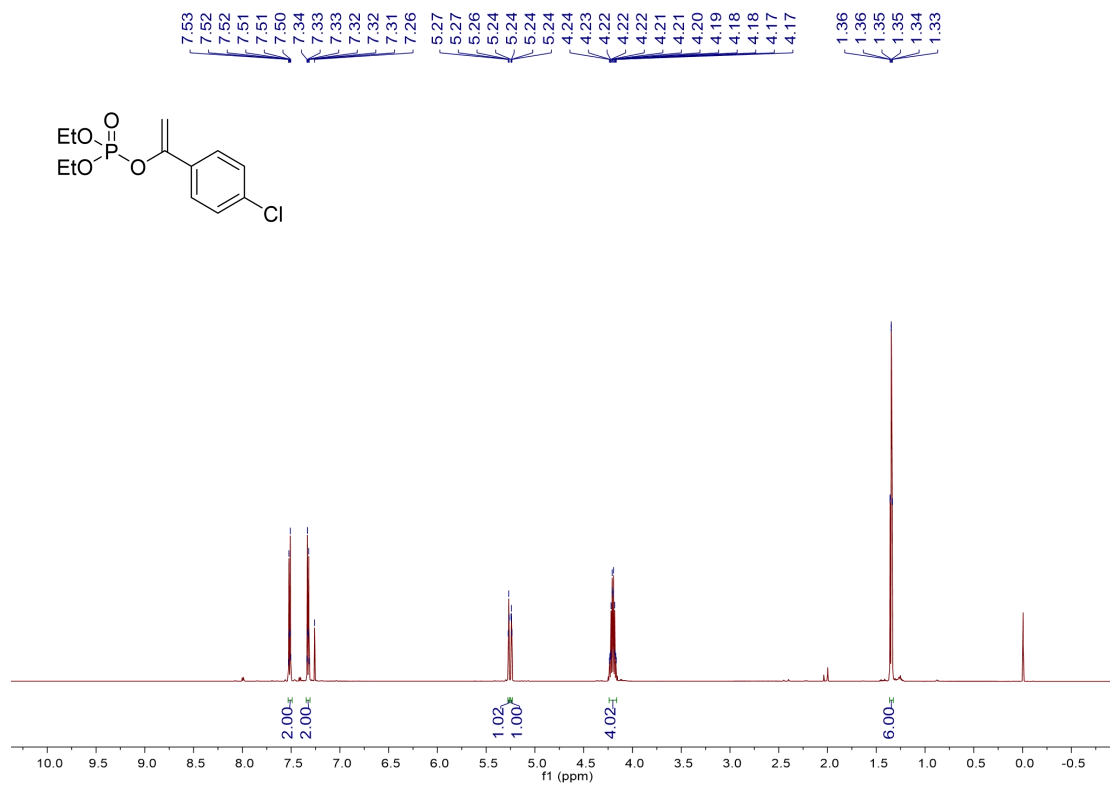
^1H NMR of compound 3cb



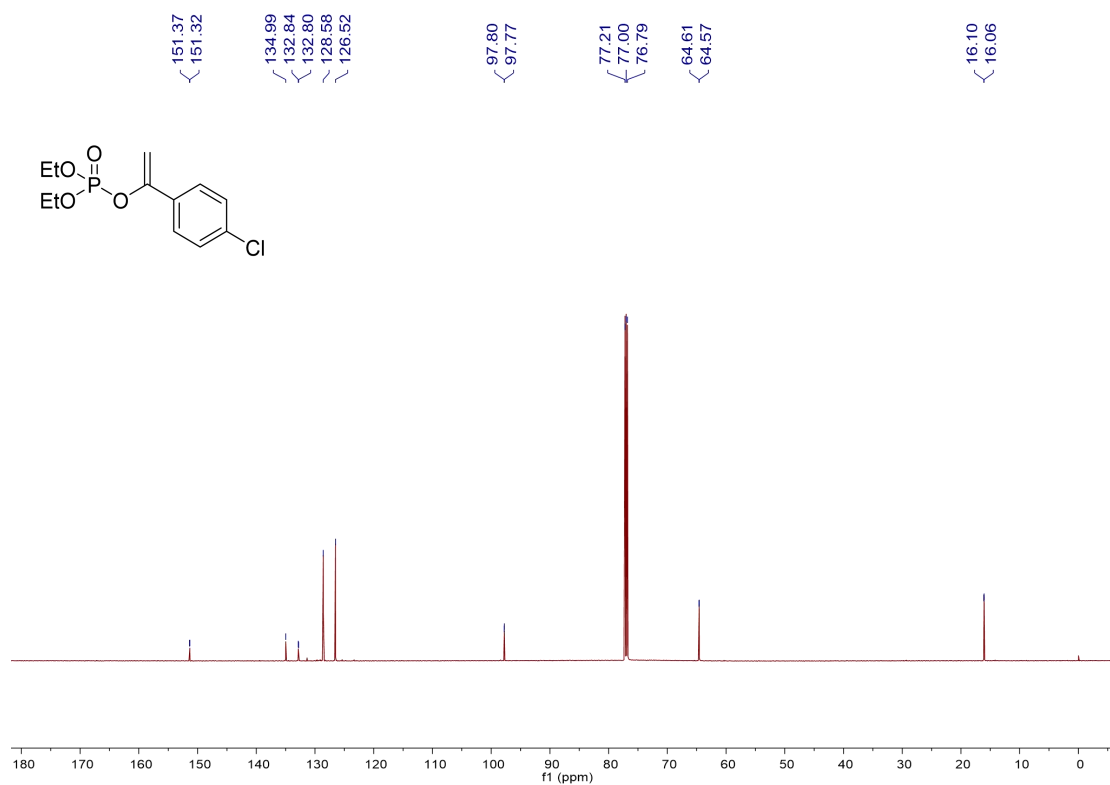
¹³C NMR of compound 3b



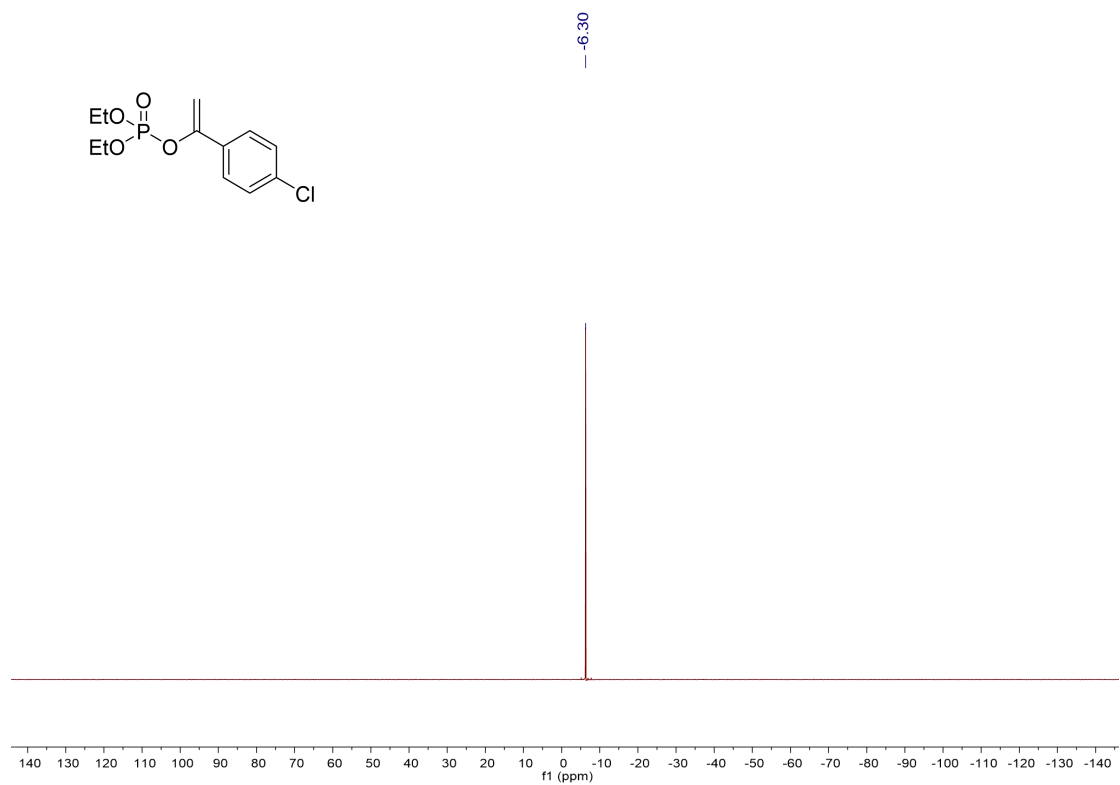
³¹P NMR of compound 3b



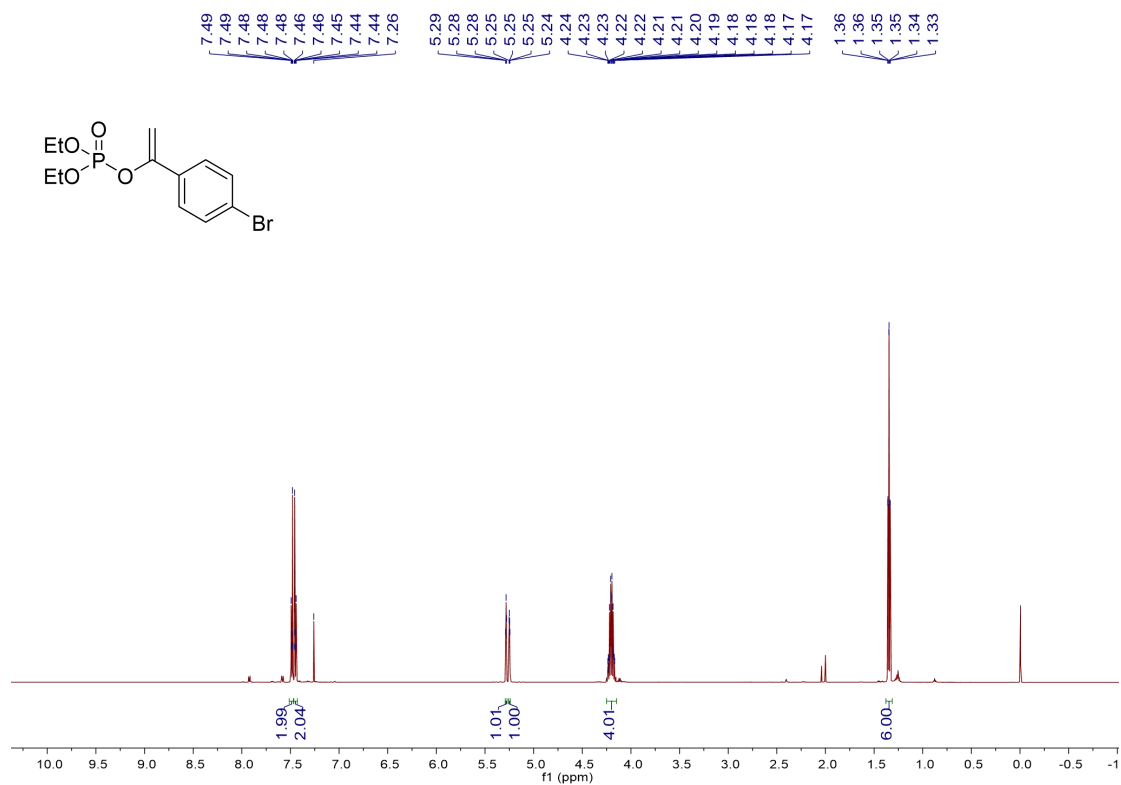
^1H NMR of compound **3cc**



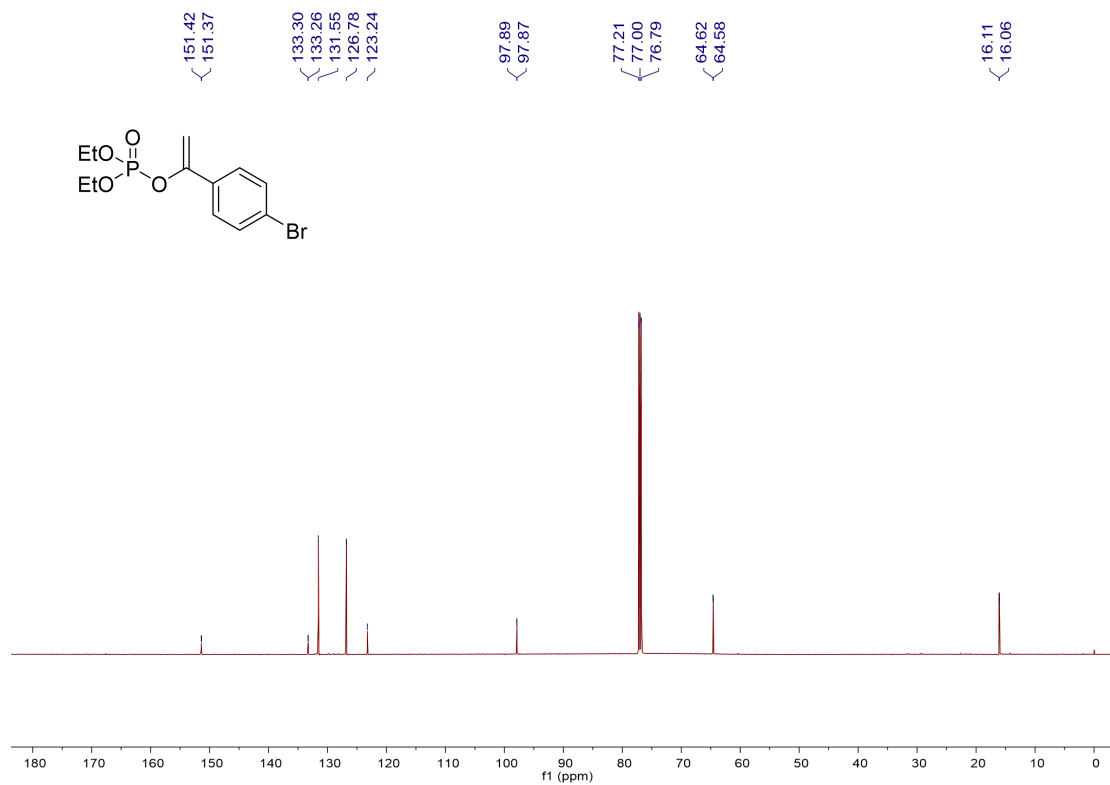
^{13}C NMR of compound **3cc**



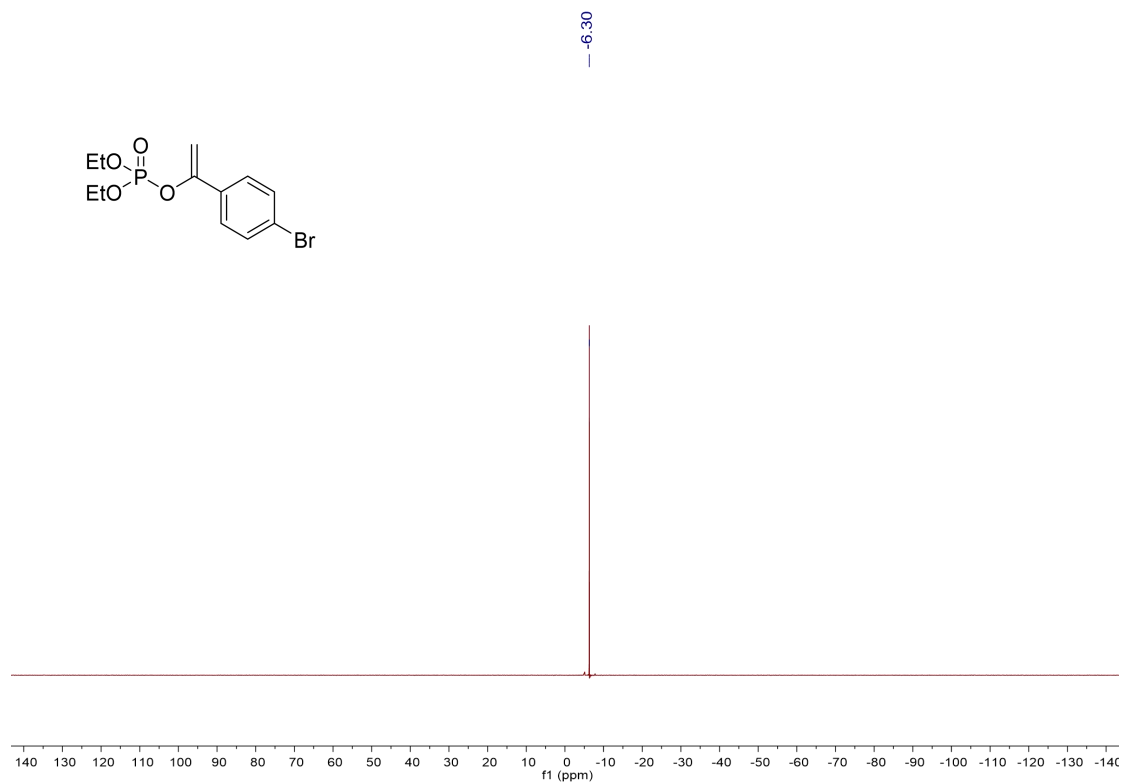
^{31}P NMR of compound 3cc



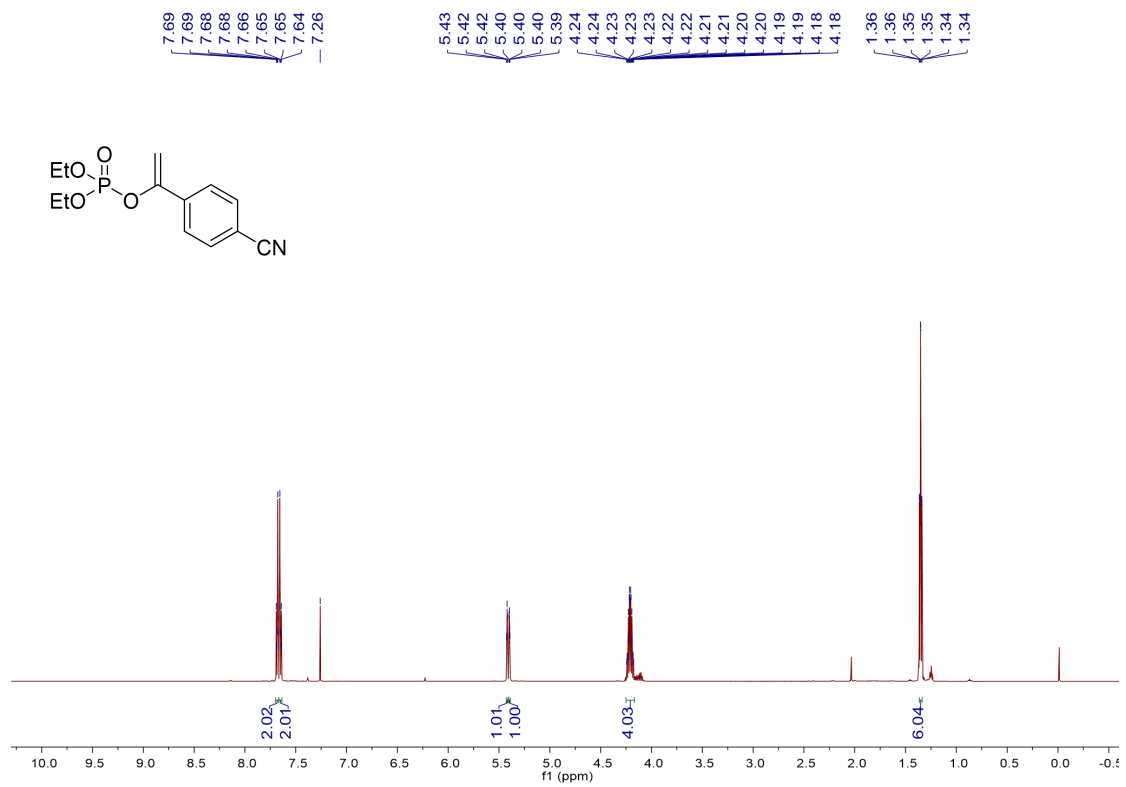
^1H NMR of compound 3cd



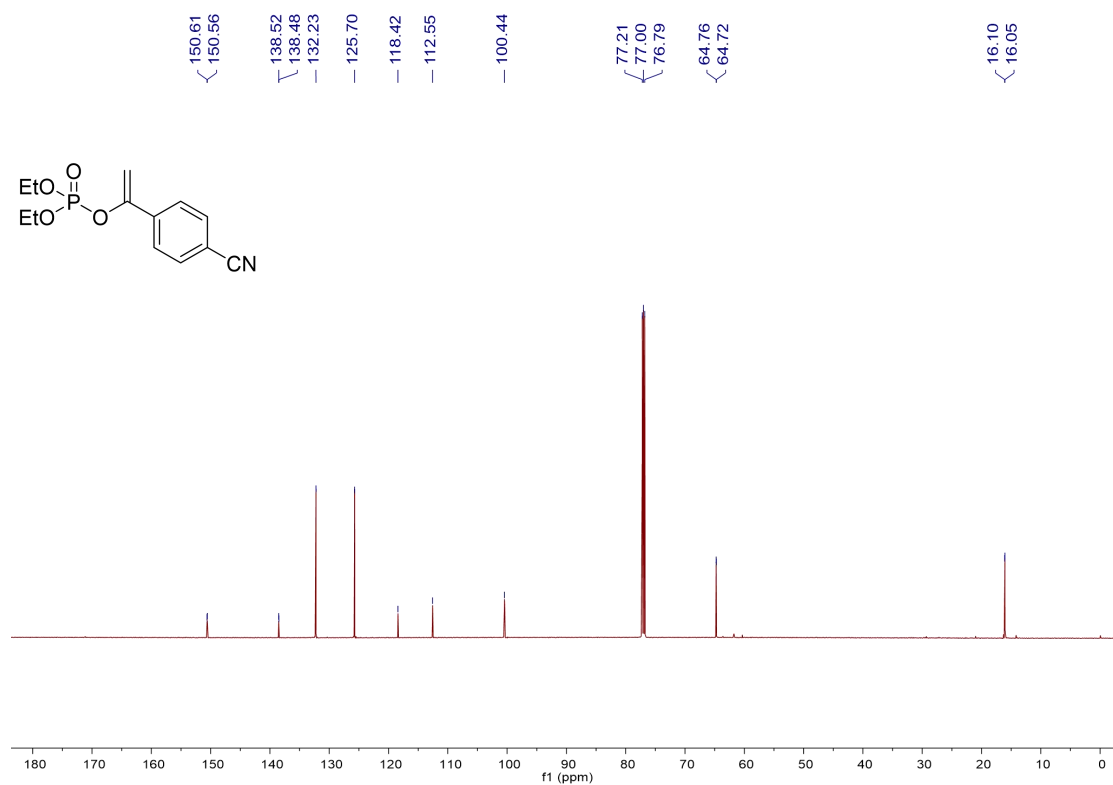
¹³C NMR of compound 3cd



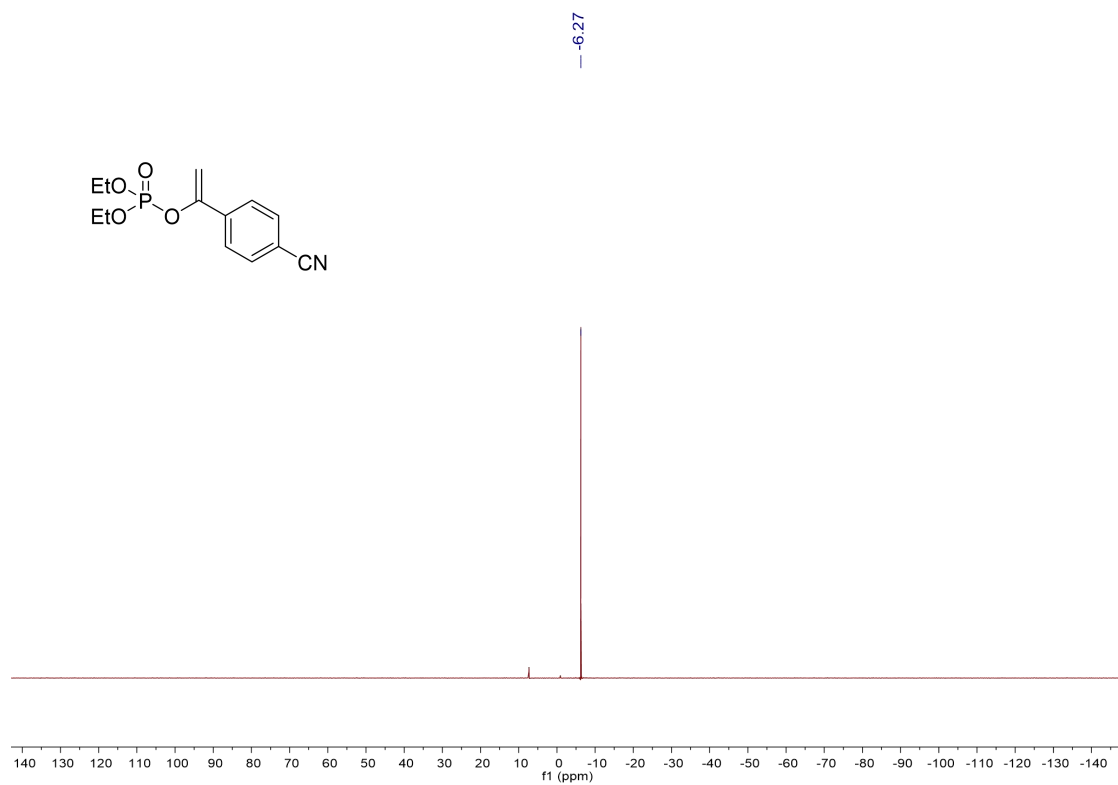
³¹P NMR of compound 3cd



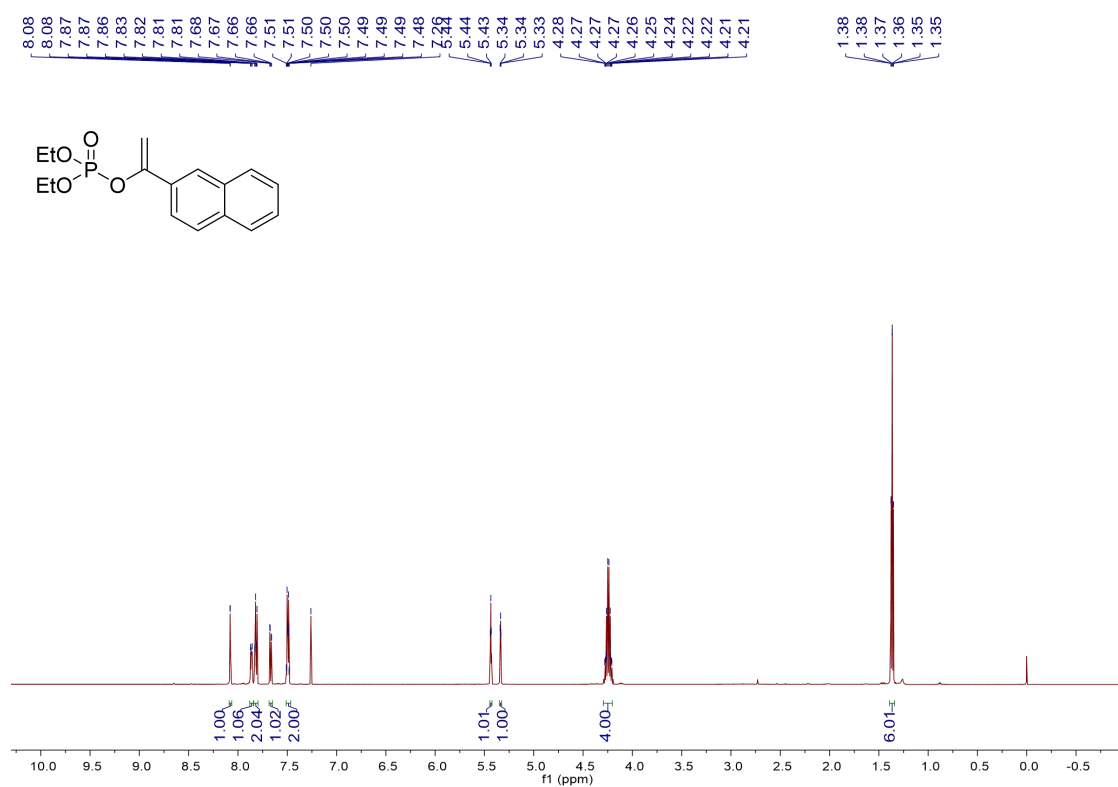
^1H NMR of compound **3ce**



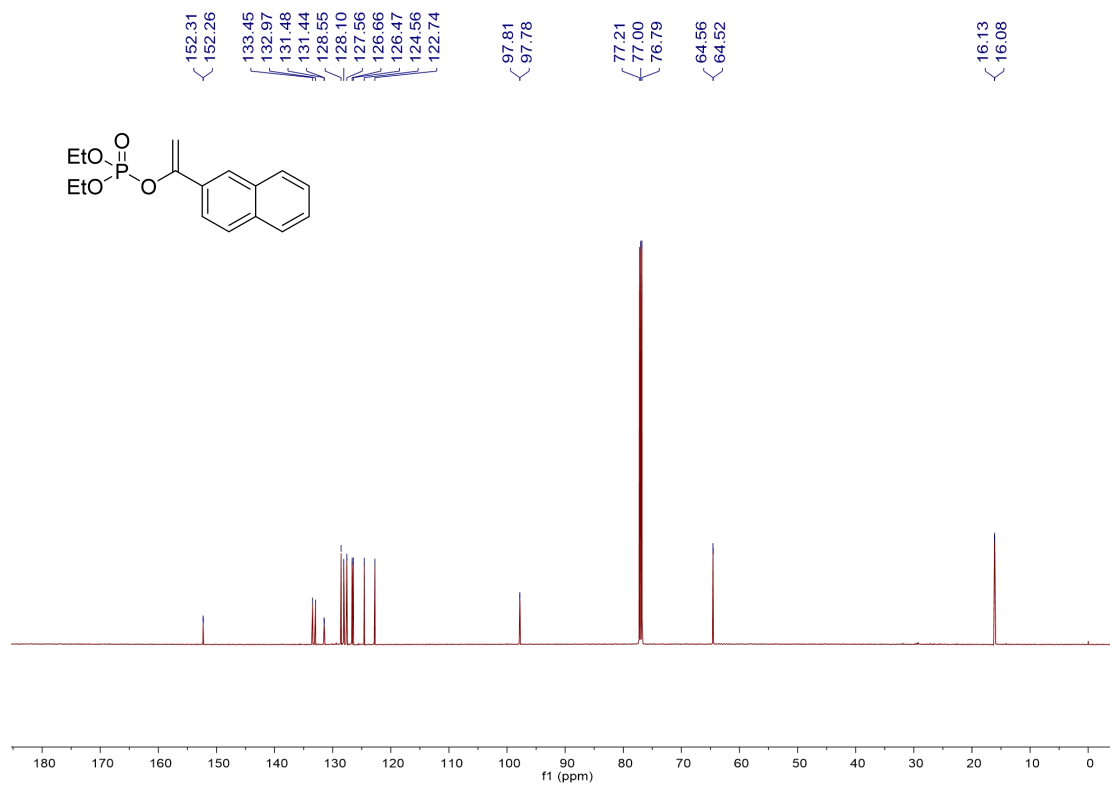
^{13}C NMR of compound **3ce**



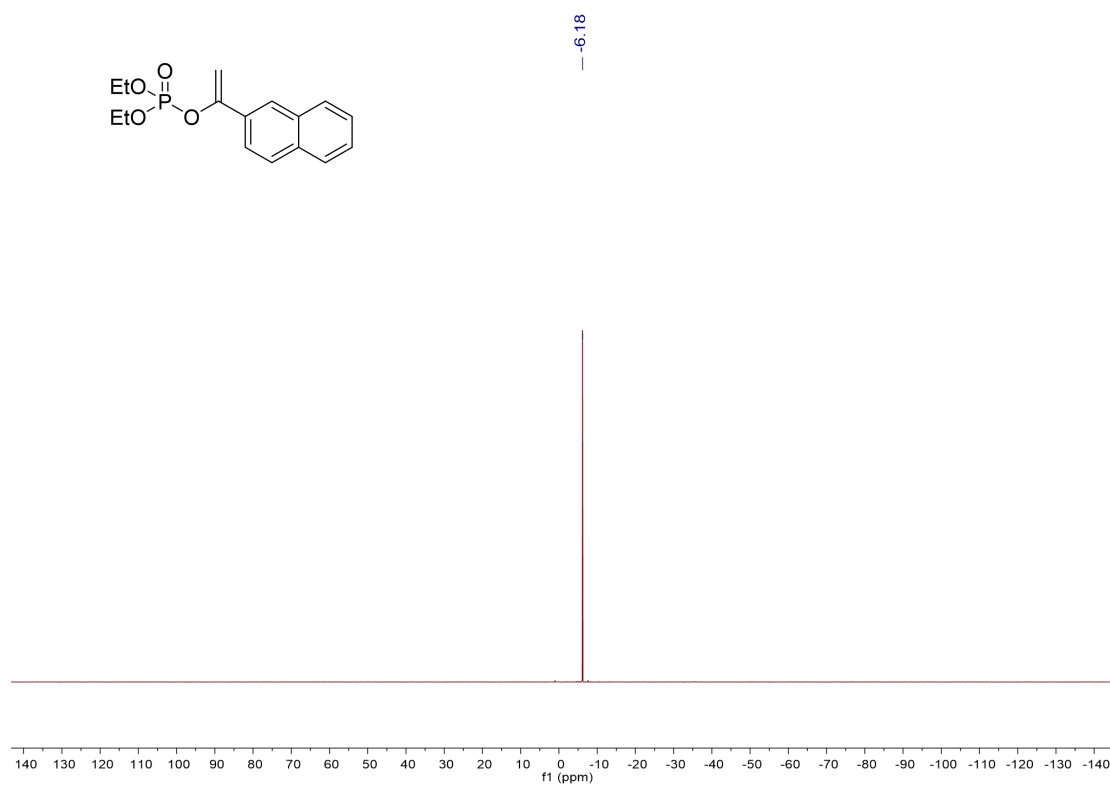
^{31}P NMR of compound 3ce



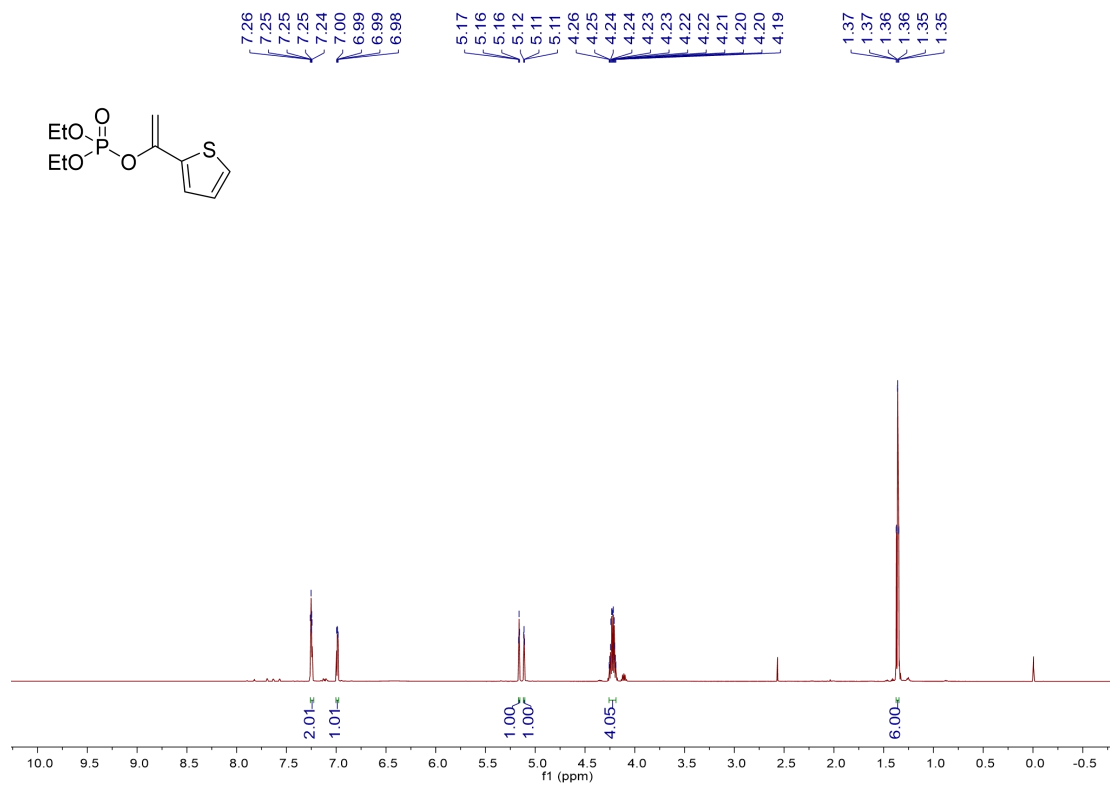
^1H NMR of compound 3cf



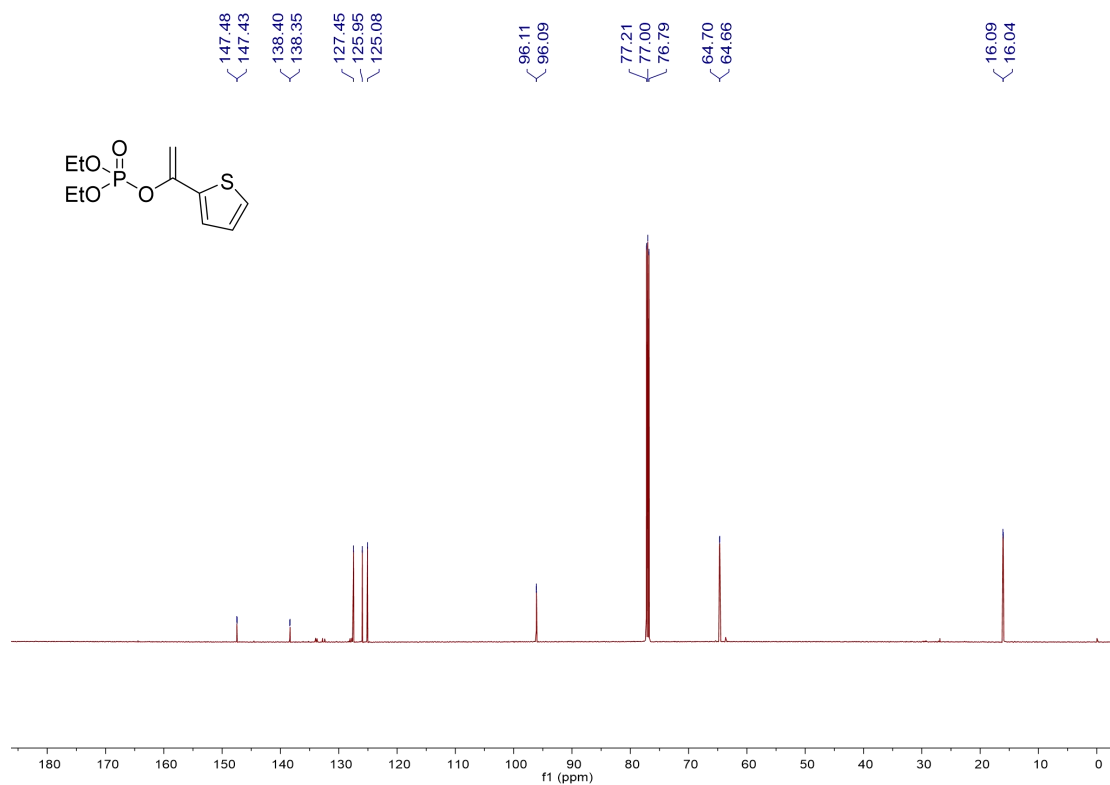
¹³C NMR of compound 3cf



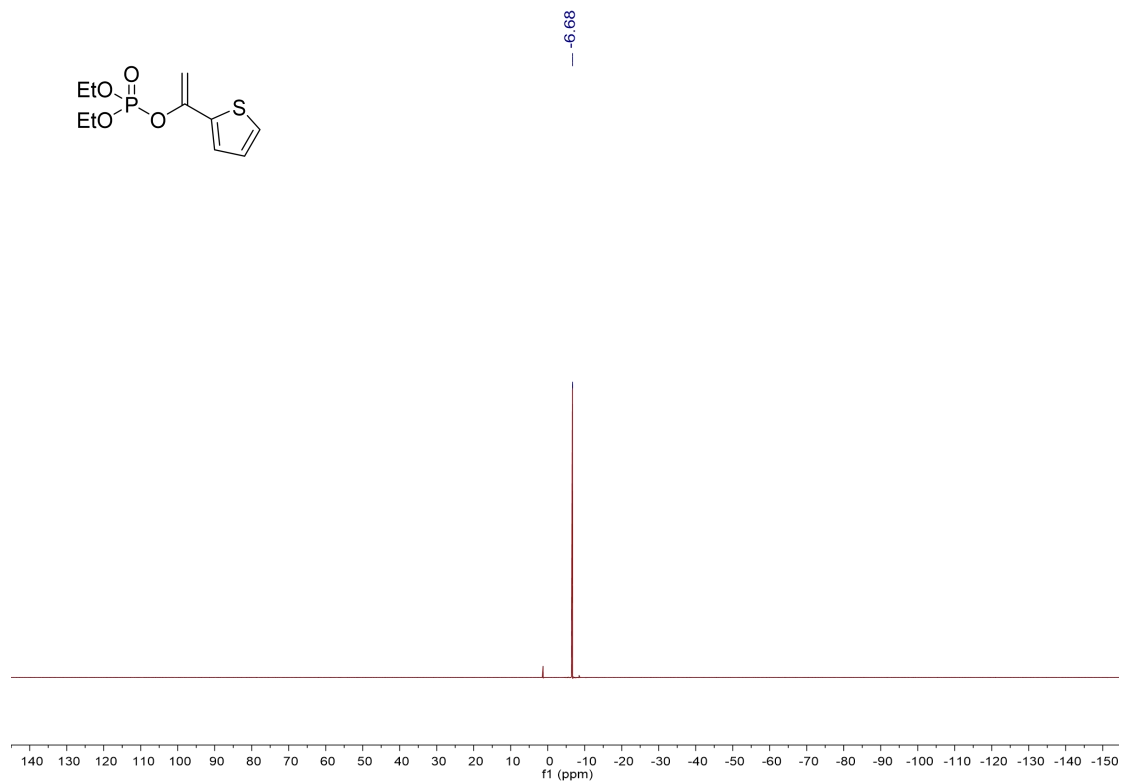
³¹P NMR of compound 3cf



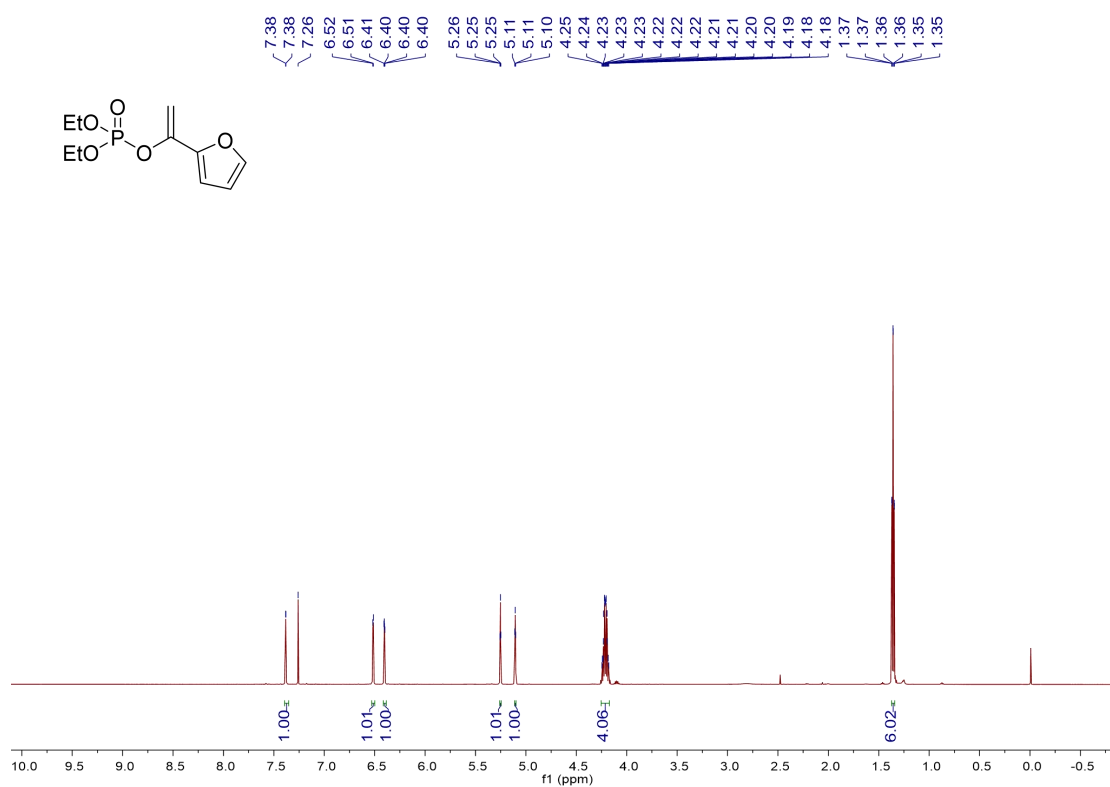
^1H NMR of compound **3cg**



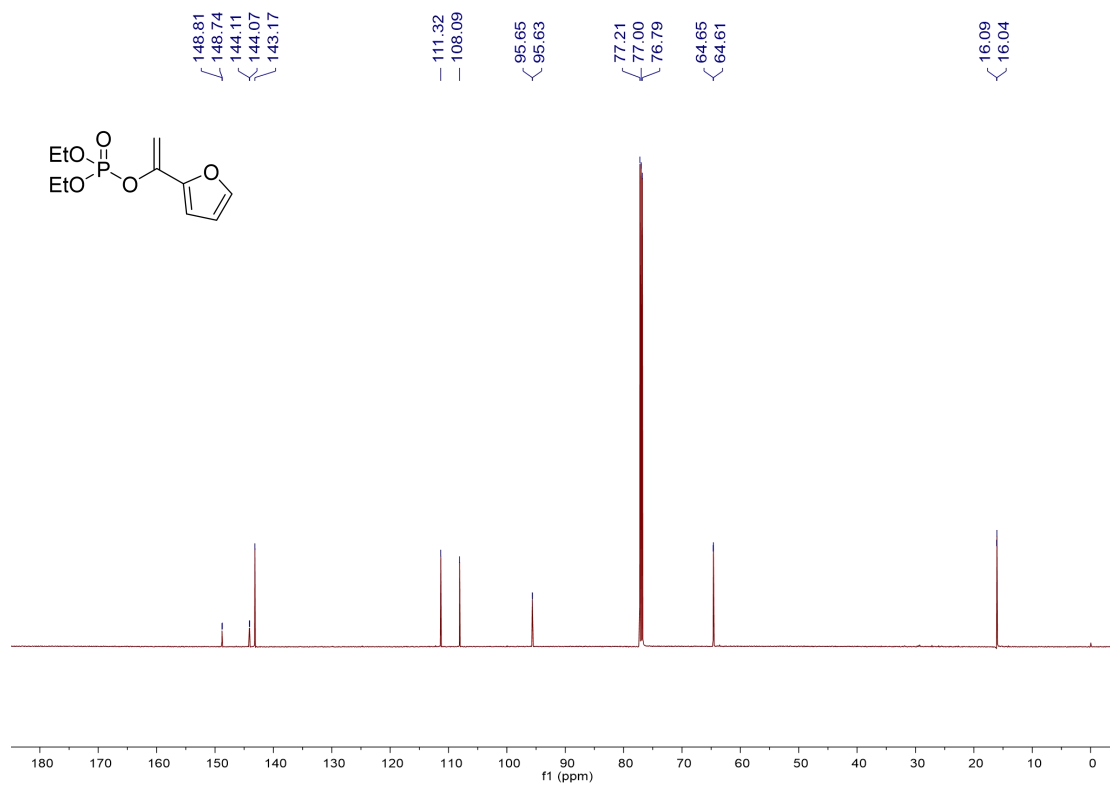
^{13}C NMR of compound **3cg**



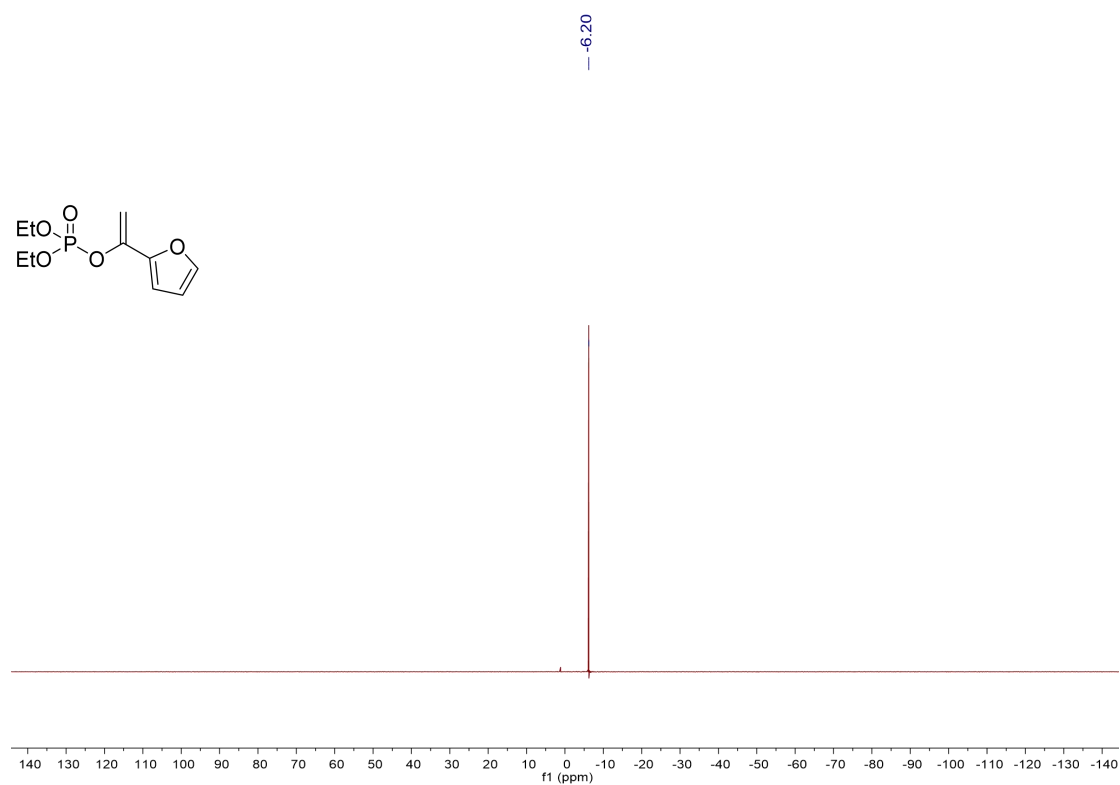
^{31}P NMR of compound **3cg**



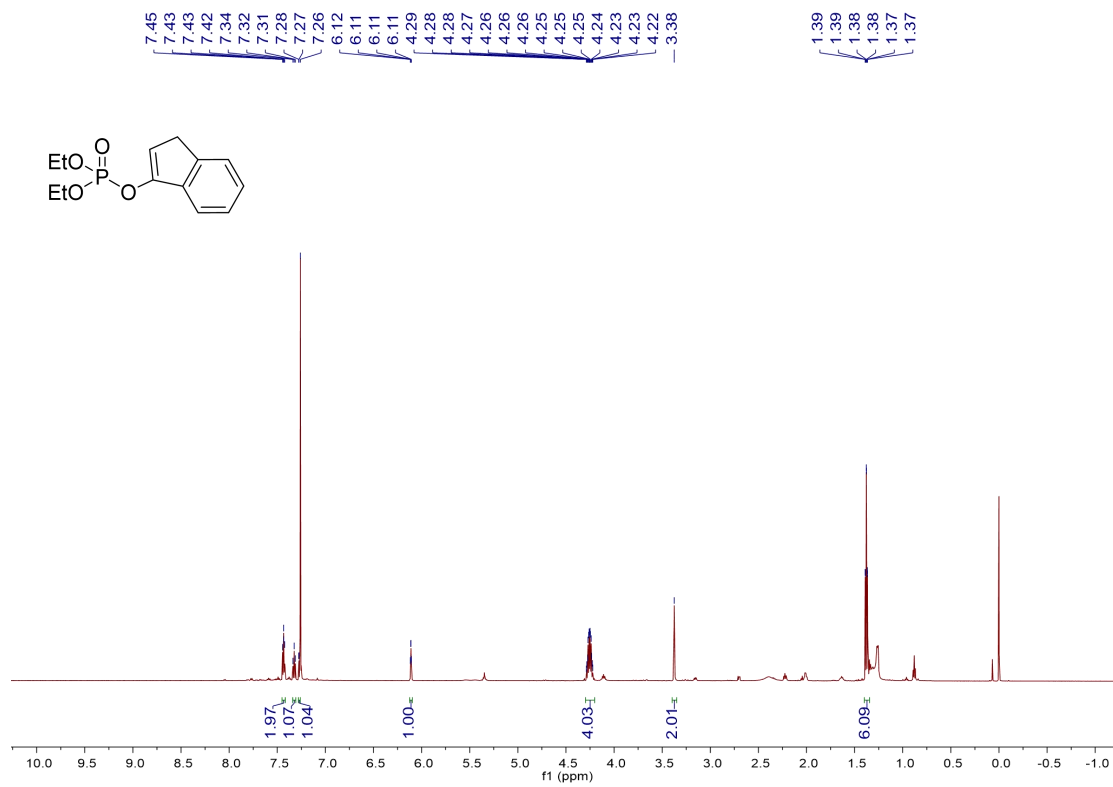
^1H NMR of compound **3ch**



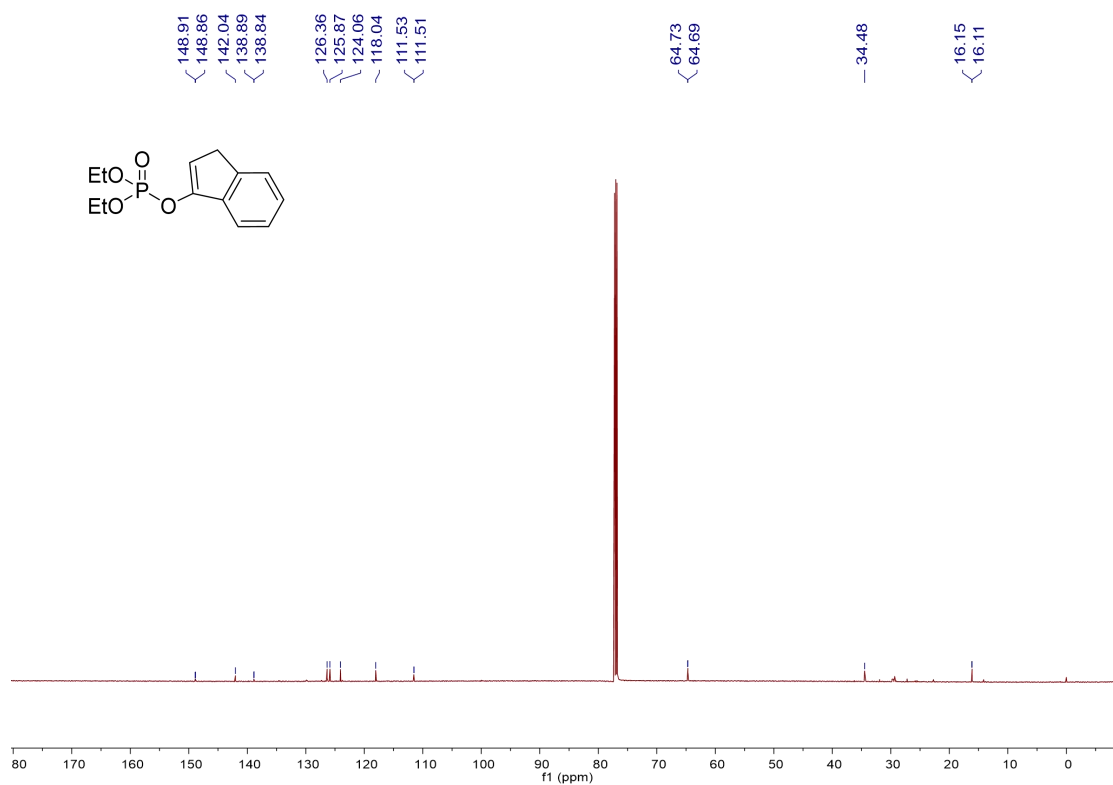
¹³C NMR of compound 3ch



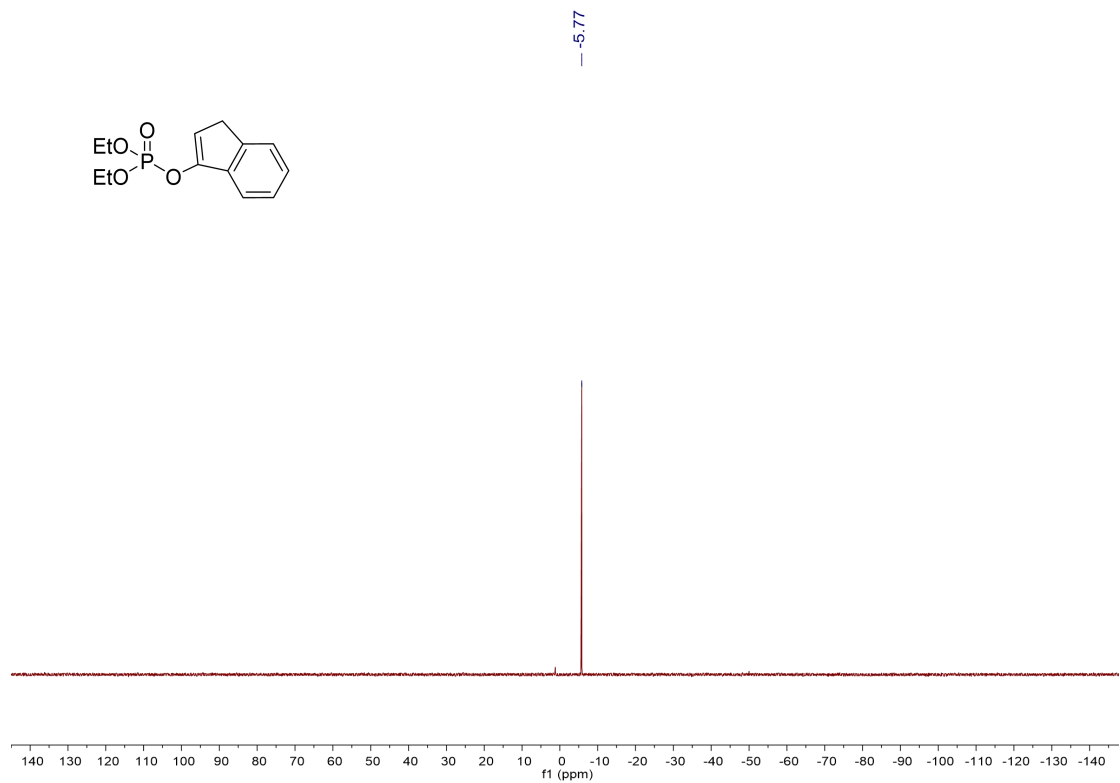
³¹P NMR of compound 3ch



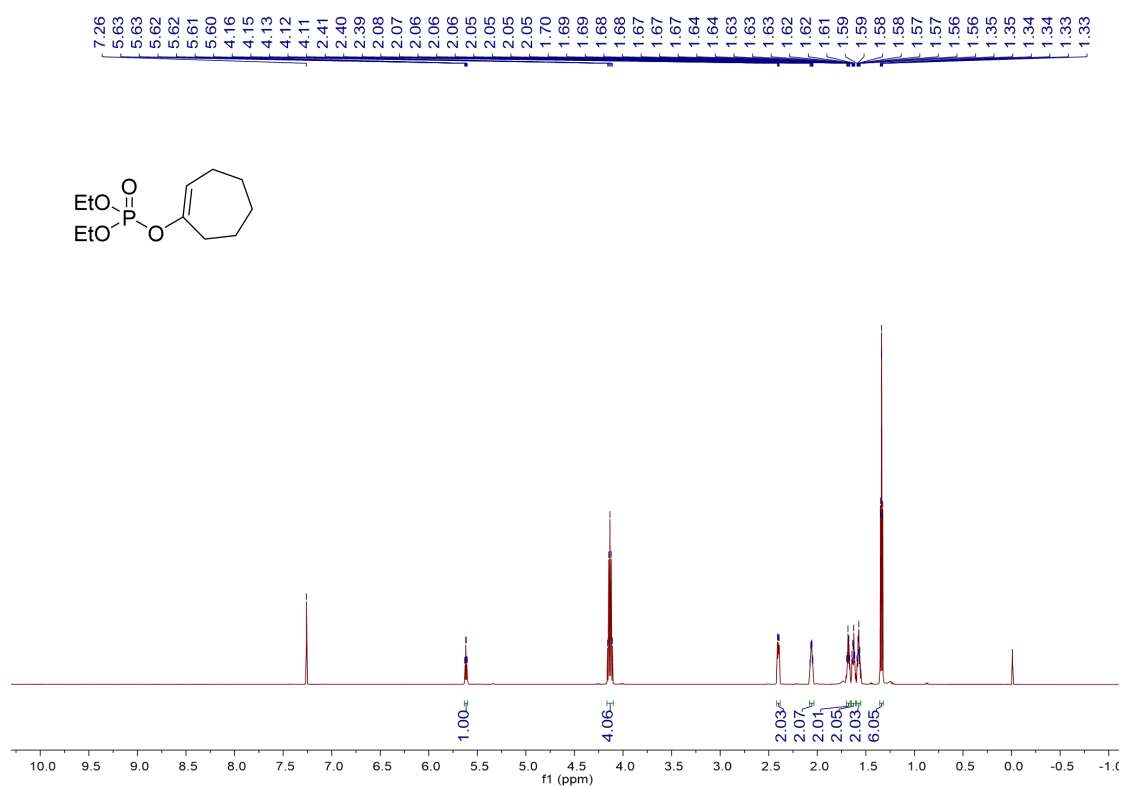
¹H NMR of compound 3ci



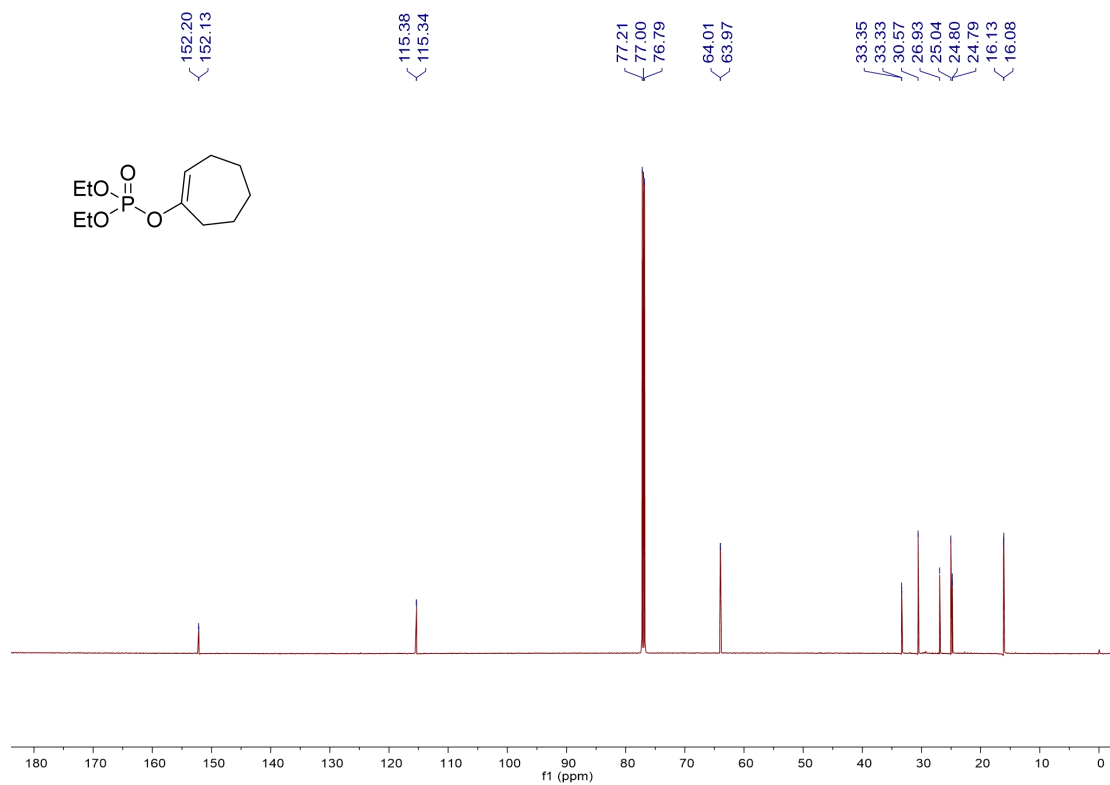
¹³C NMR of compound 3ci



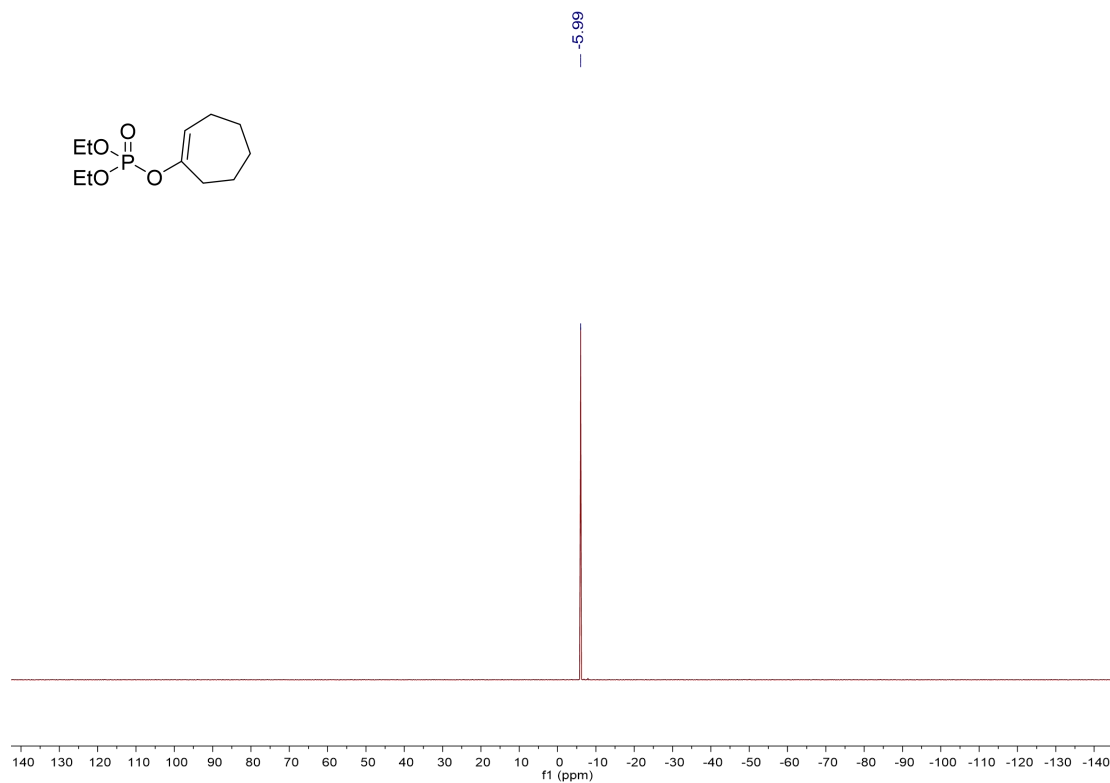
^{31}P NMR of compound 3ci



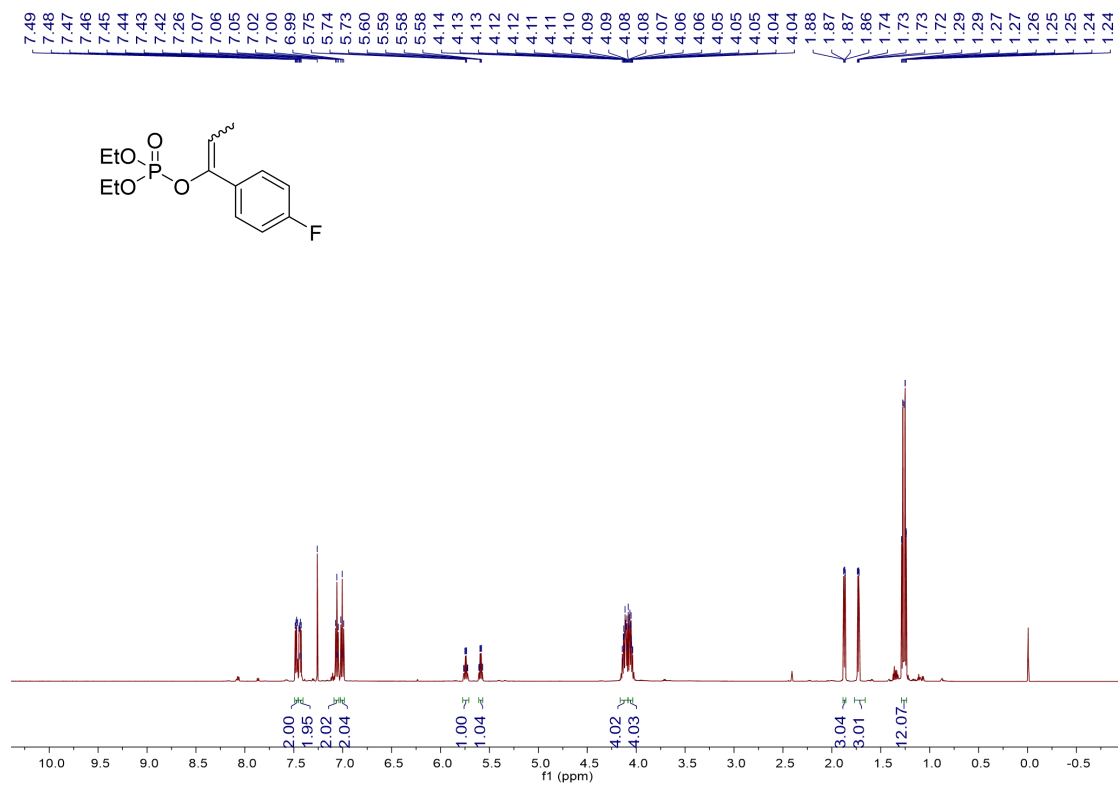
^1H NMR of compound 3cj



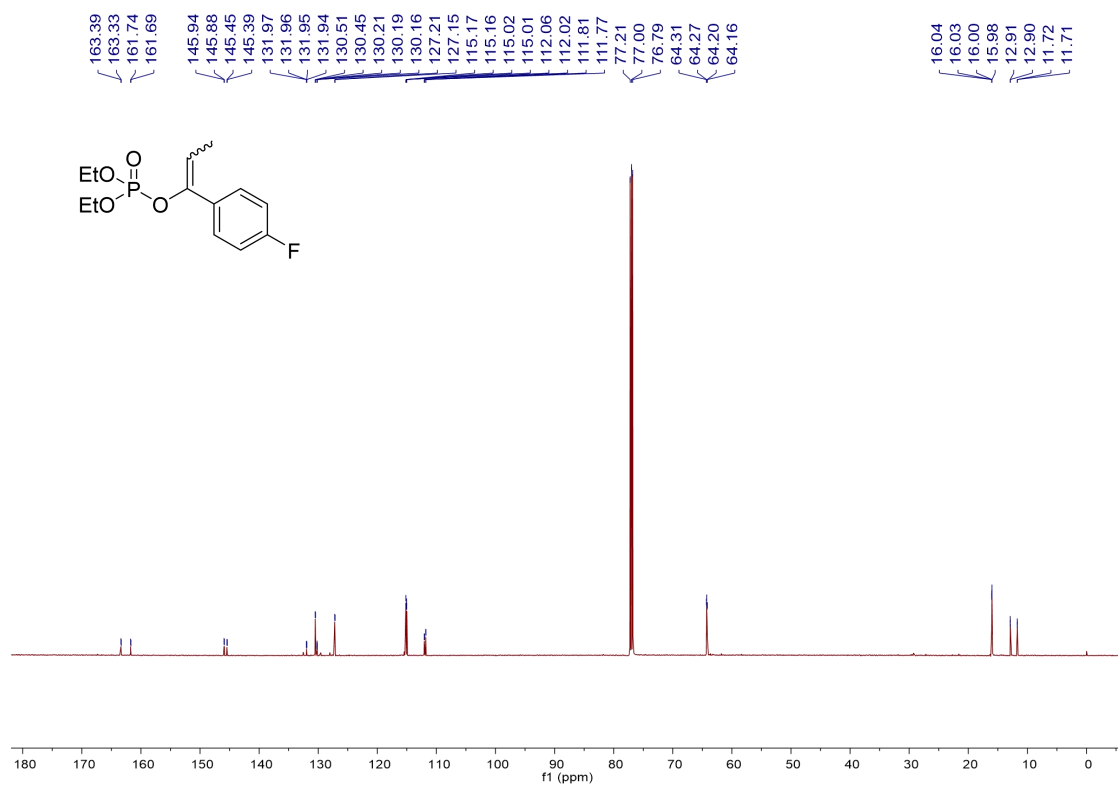
^{13}C NMR of compound 3cj



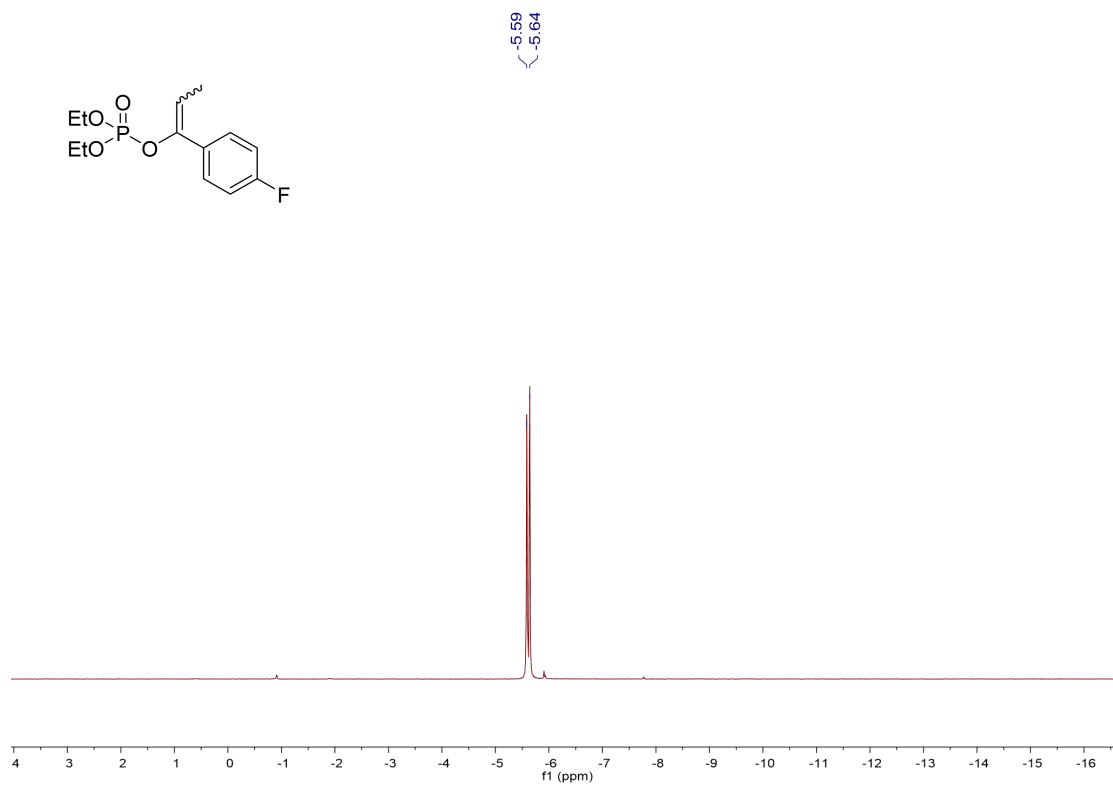
^{31}P NMR of compound 3cj



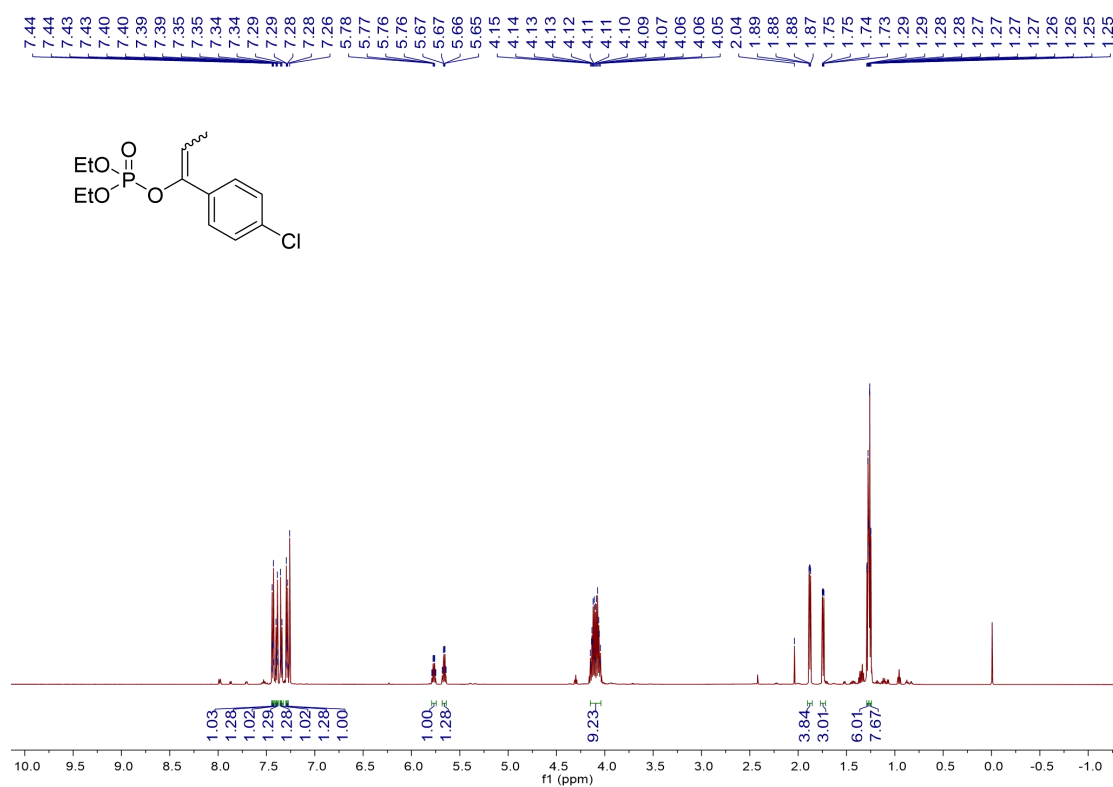
¹H NMR of compound 3da



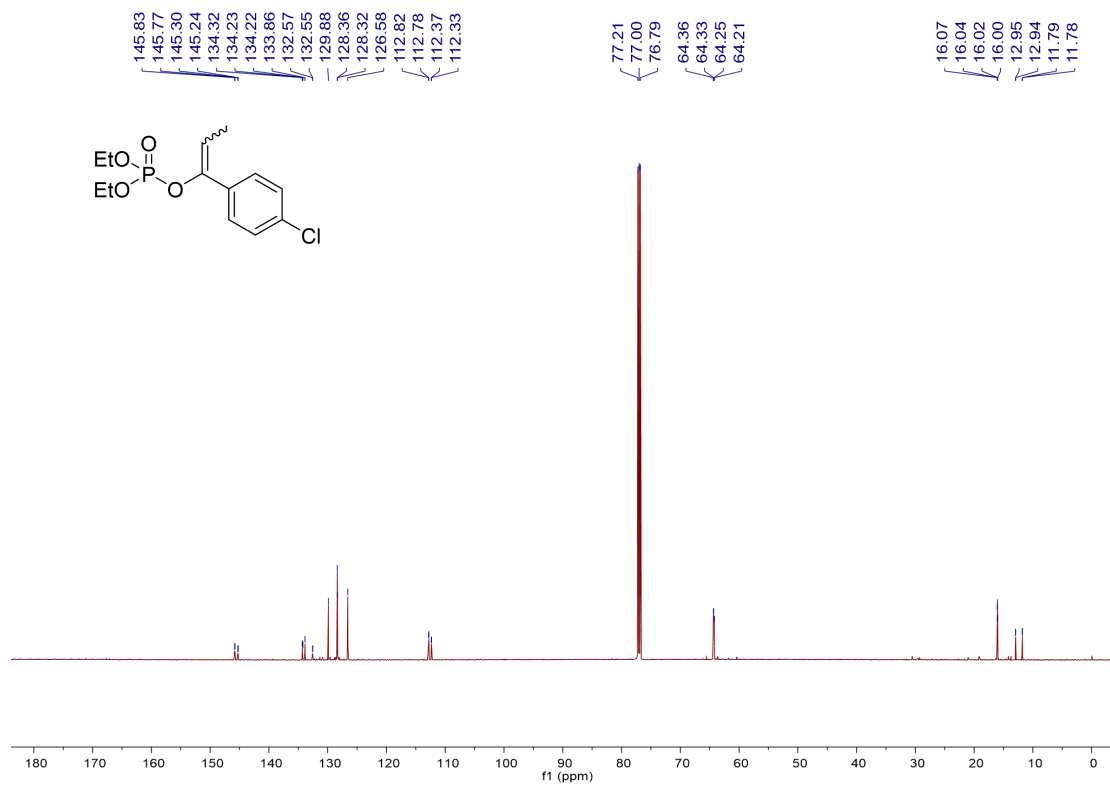
¹³C NMR of compound 3da



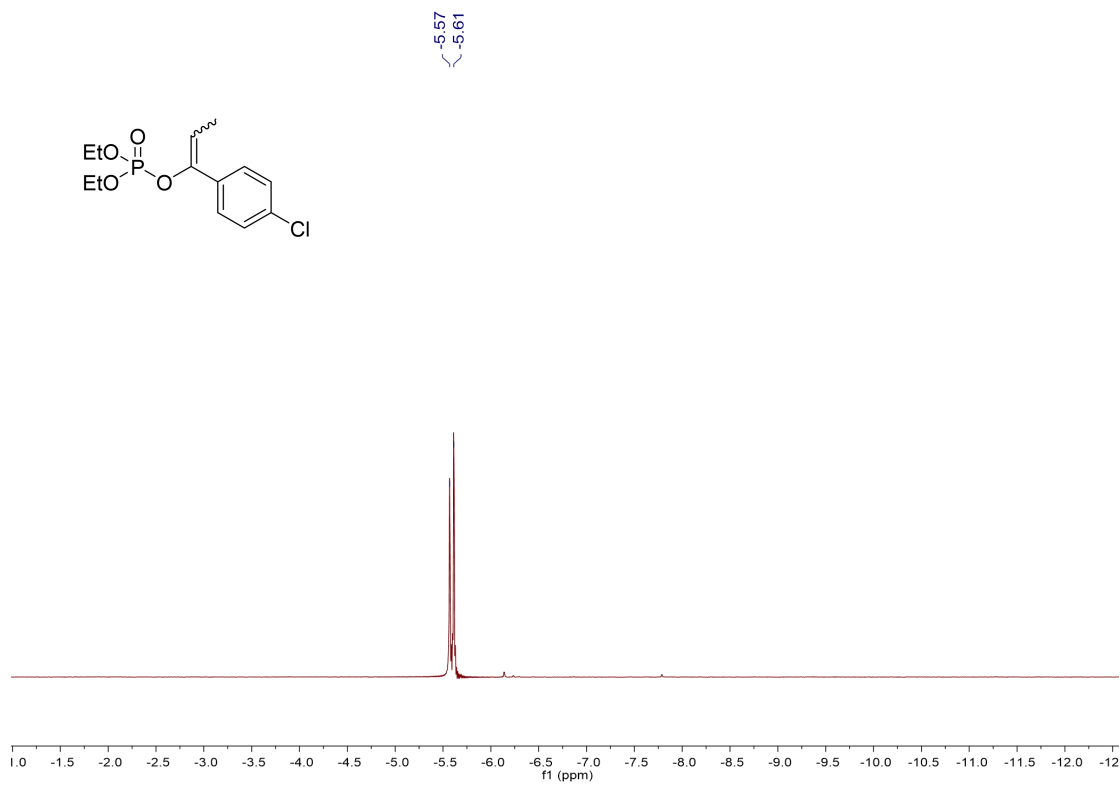
^{31}P NMR of compound 3da



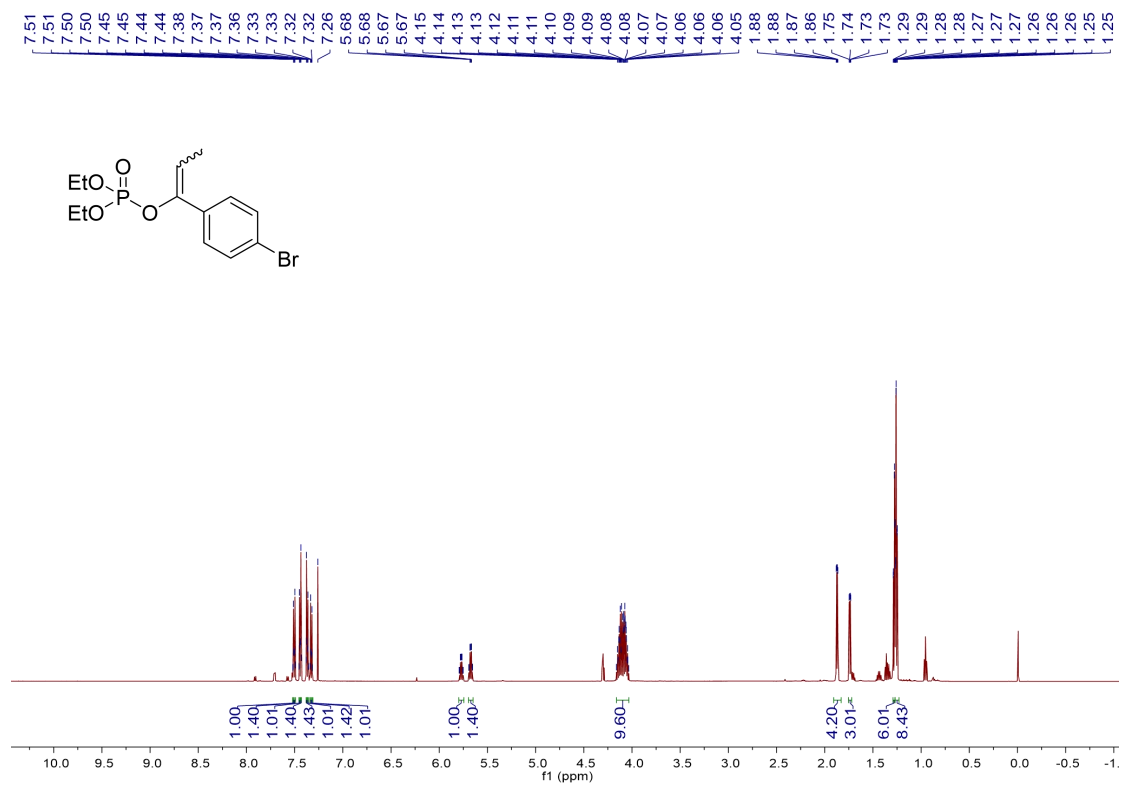
^1H NMR of compound 3db



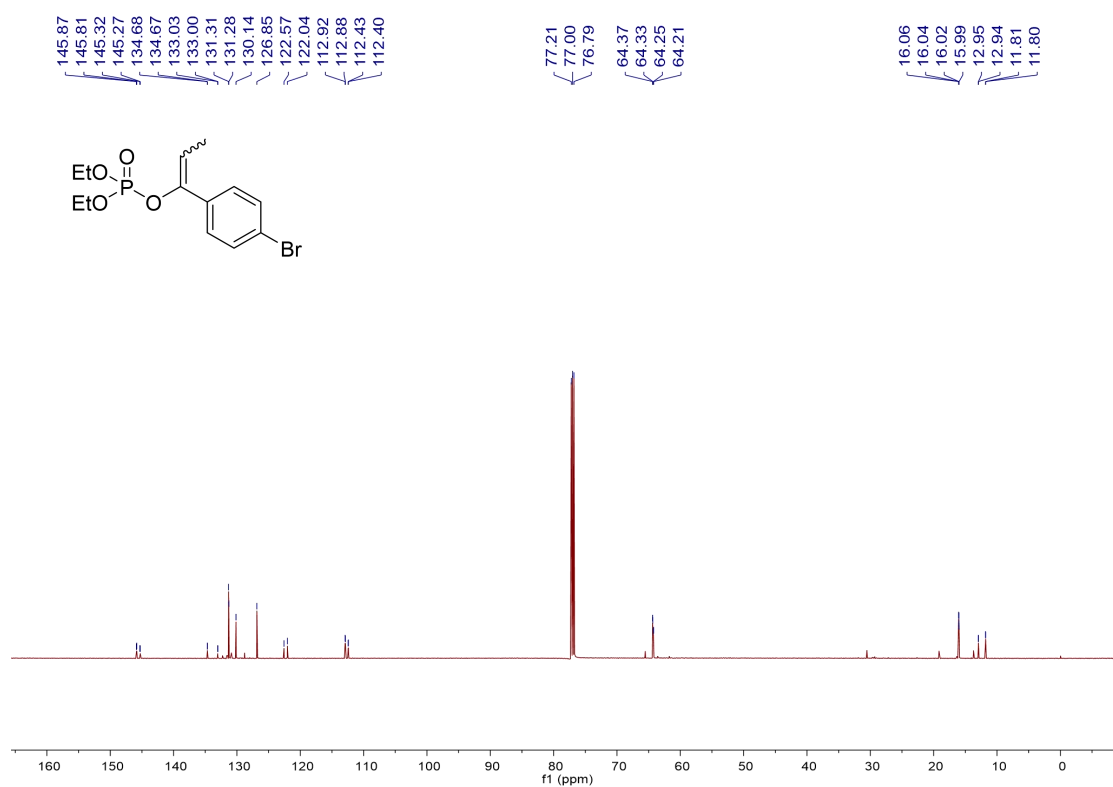
¹³C NMR of compound 3db



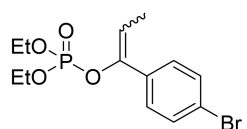
³¹P NMR of compound 3db

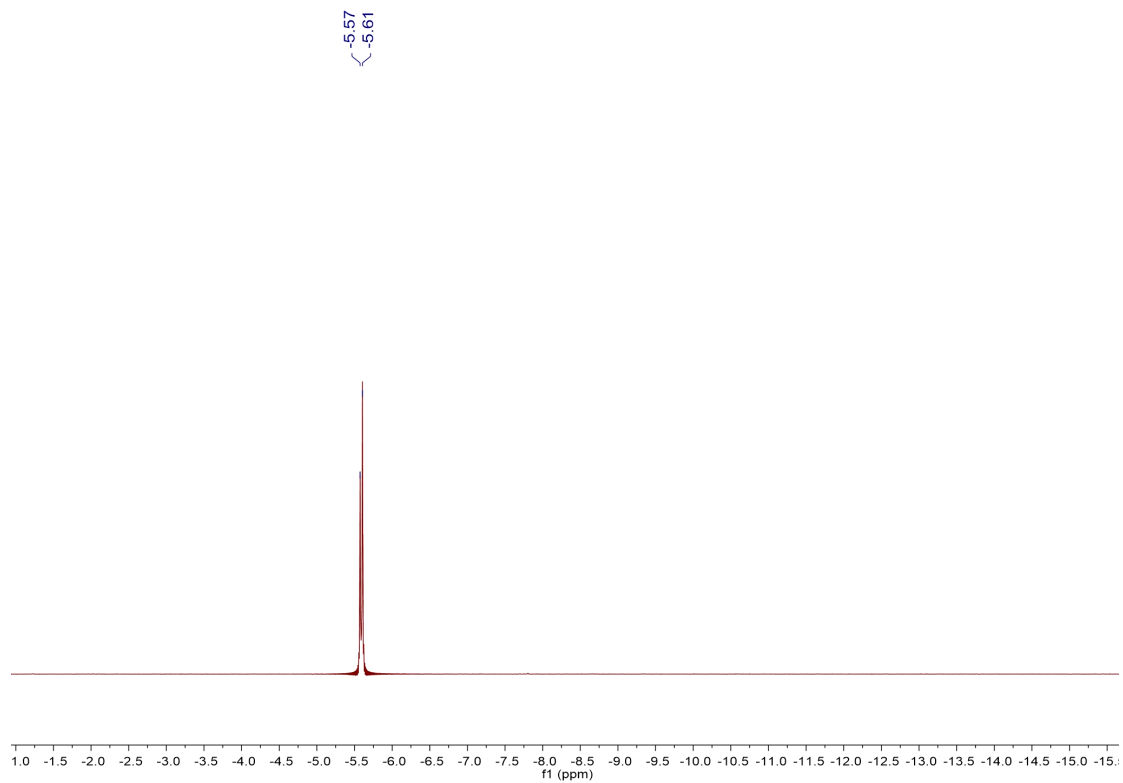


¹H NMR of compound **3dc**

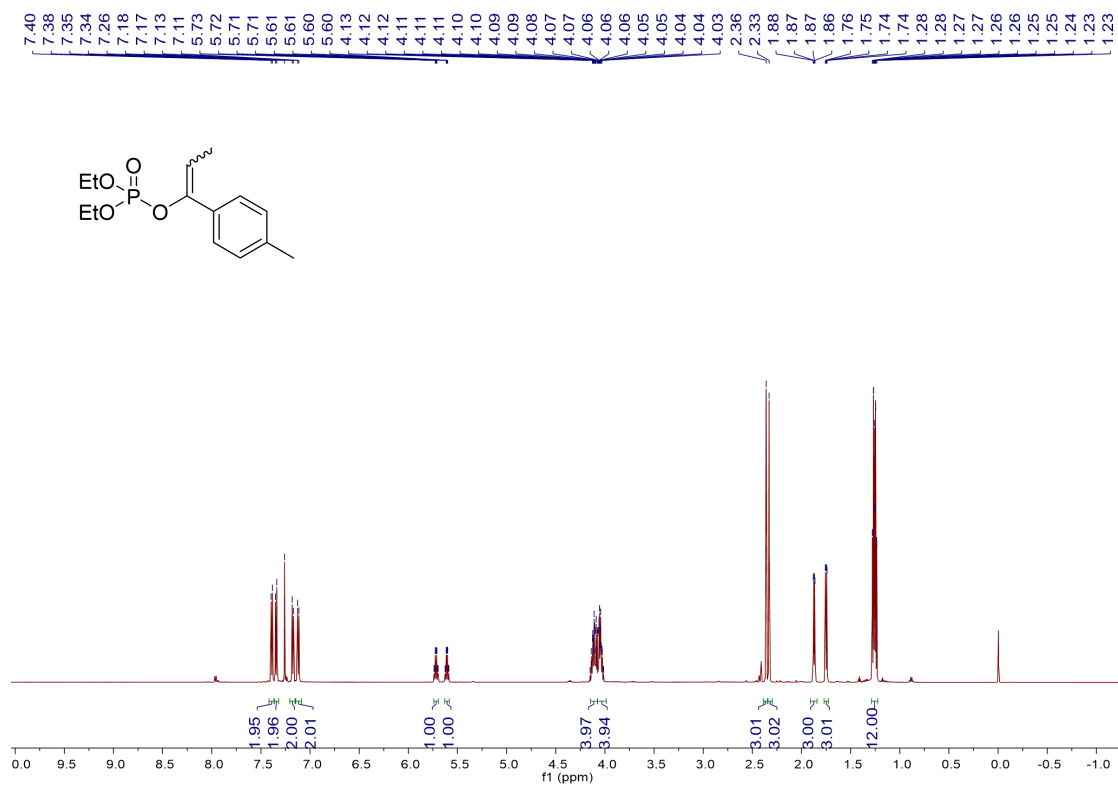


¹³C NMR of compound **3dc**

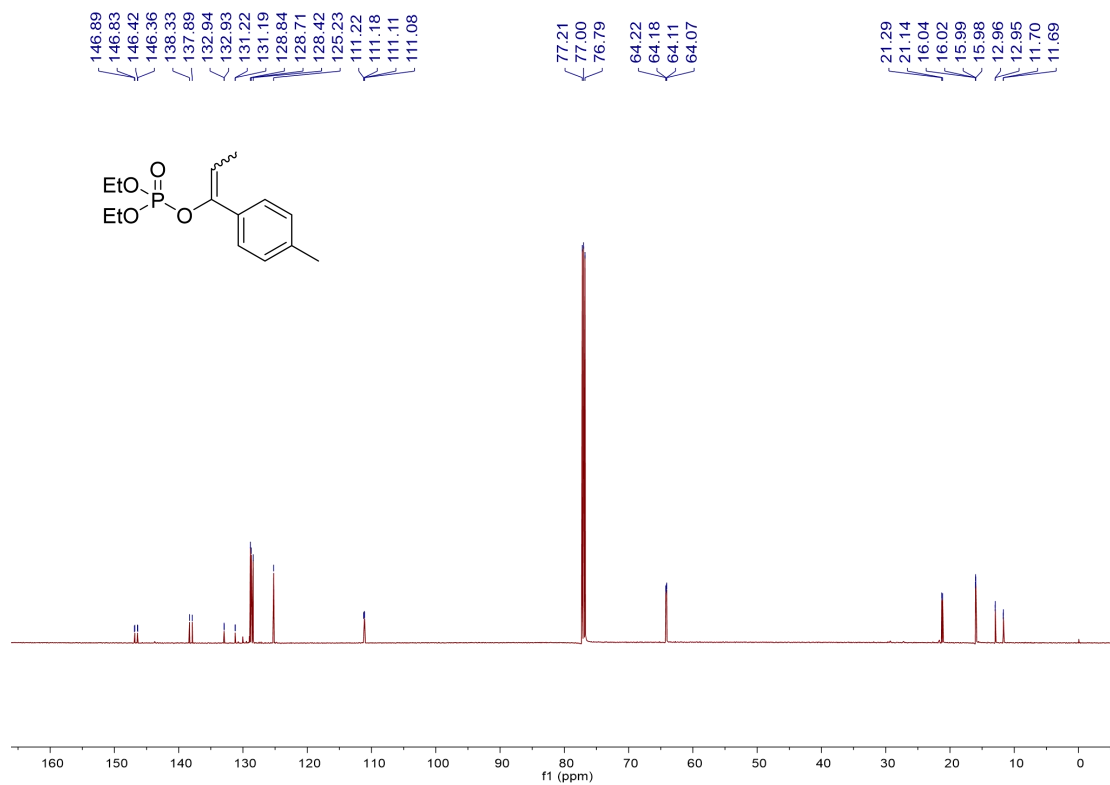




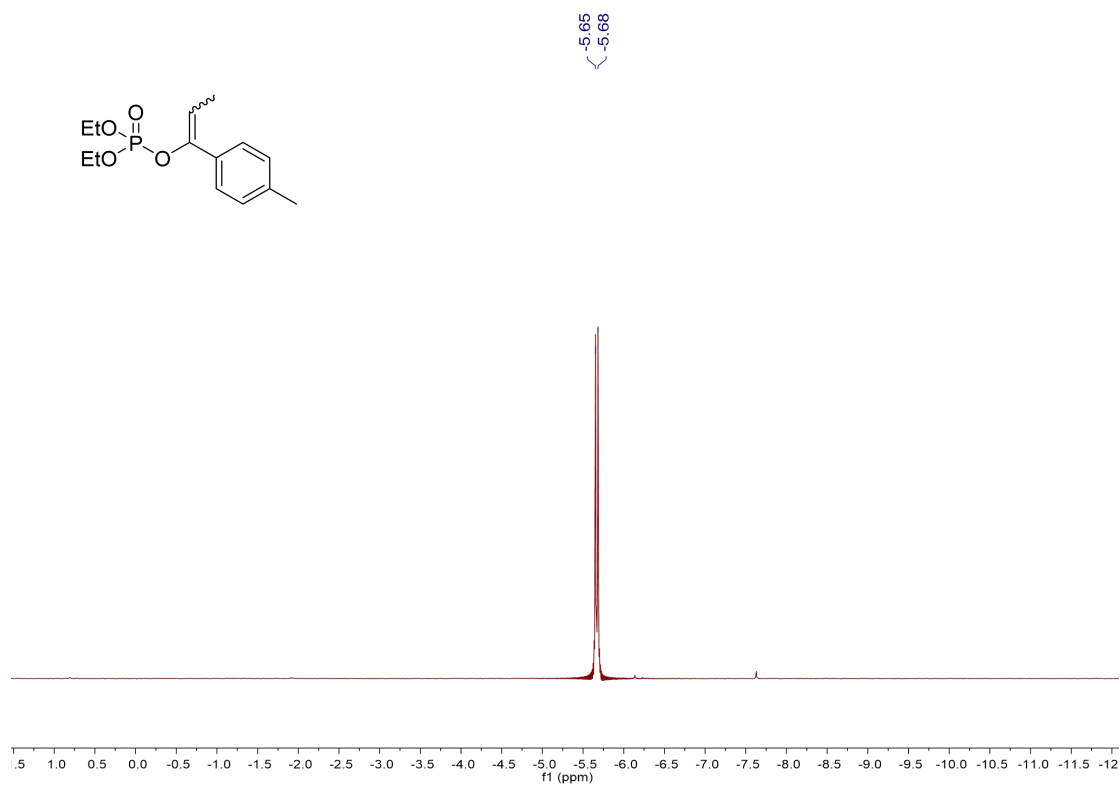
31P NMR of compound 3dc



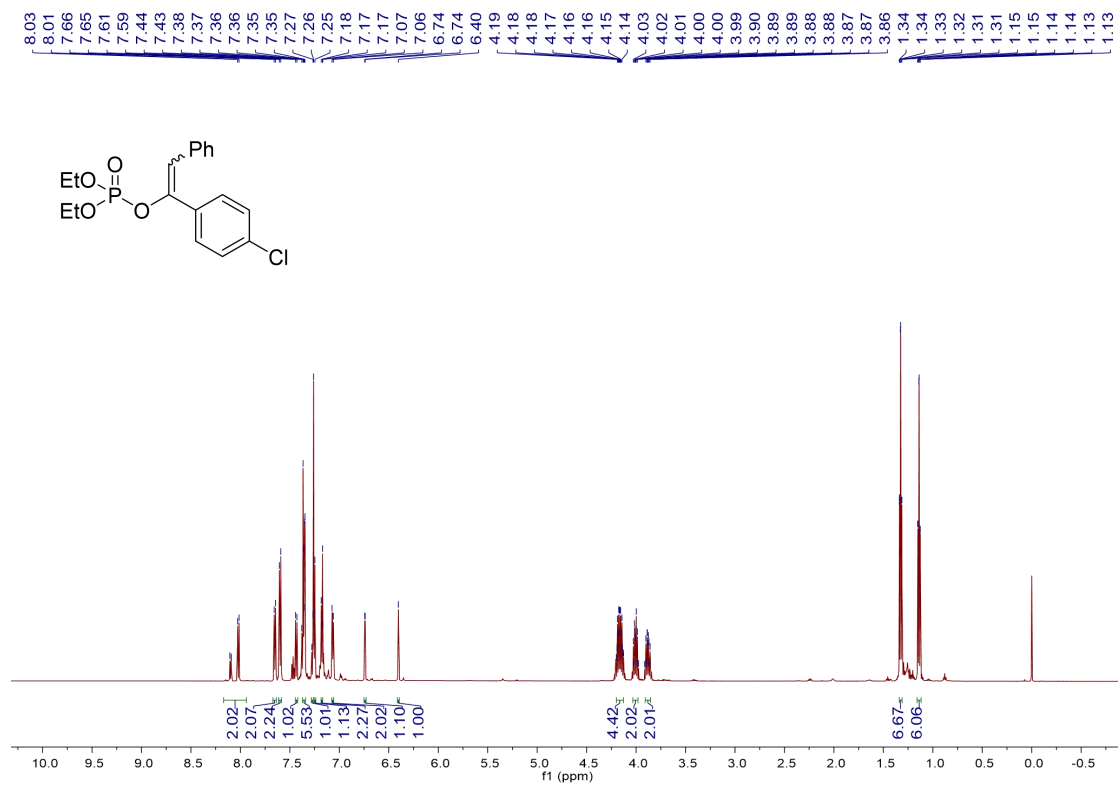
1H NMR of compound 3dd



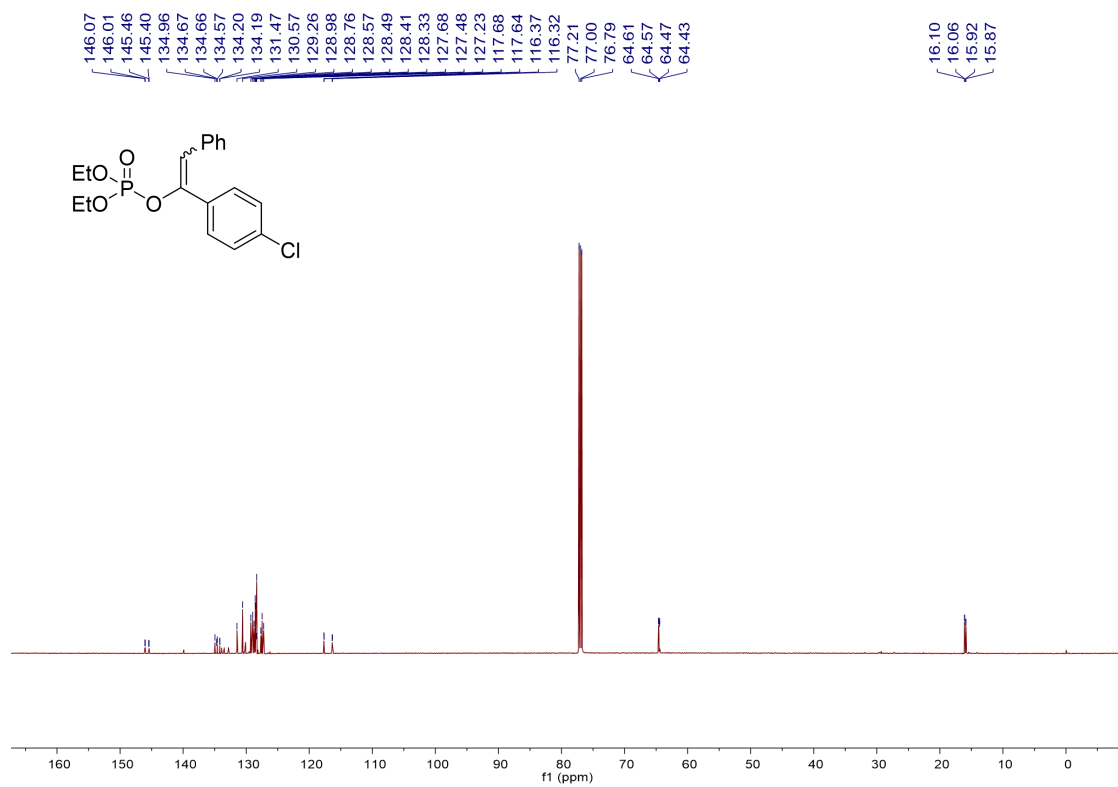
¹³C NMR of compound **3dd**



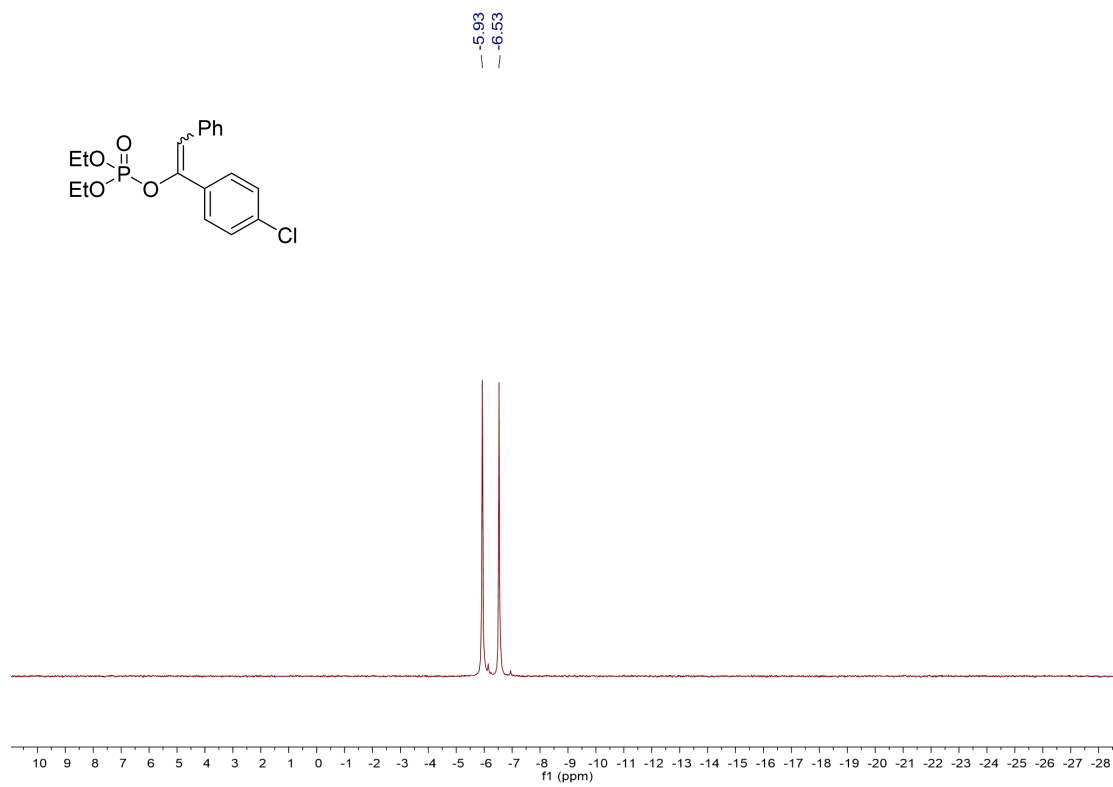
³¹P NMR of compound **3dd**



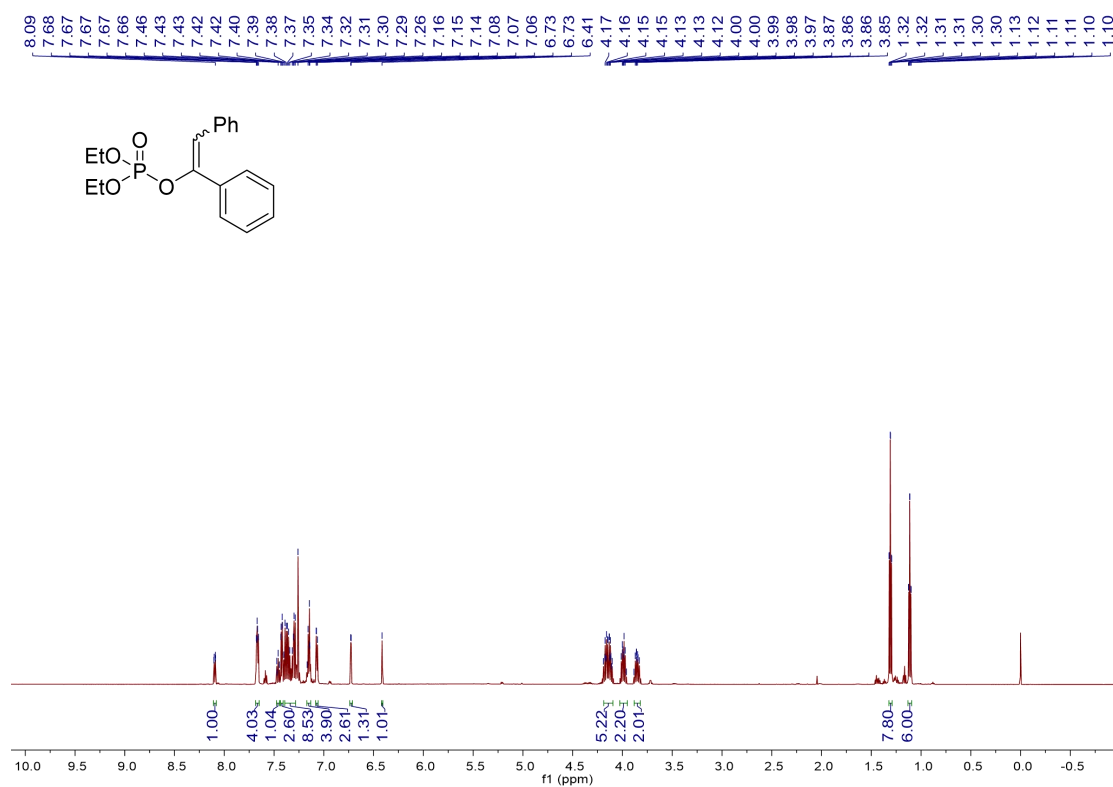
¹H NMR of compound 3de



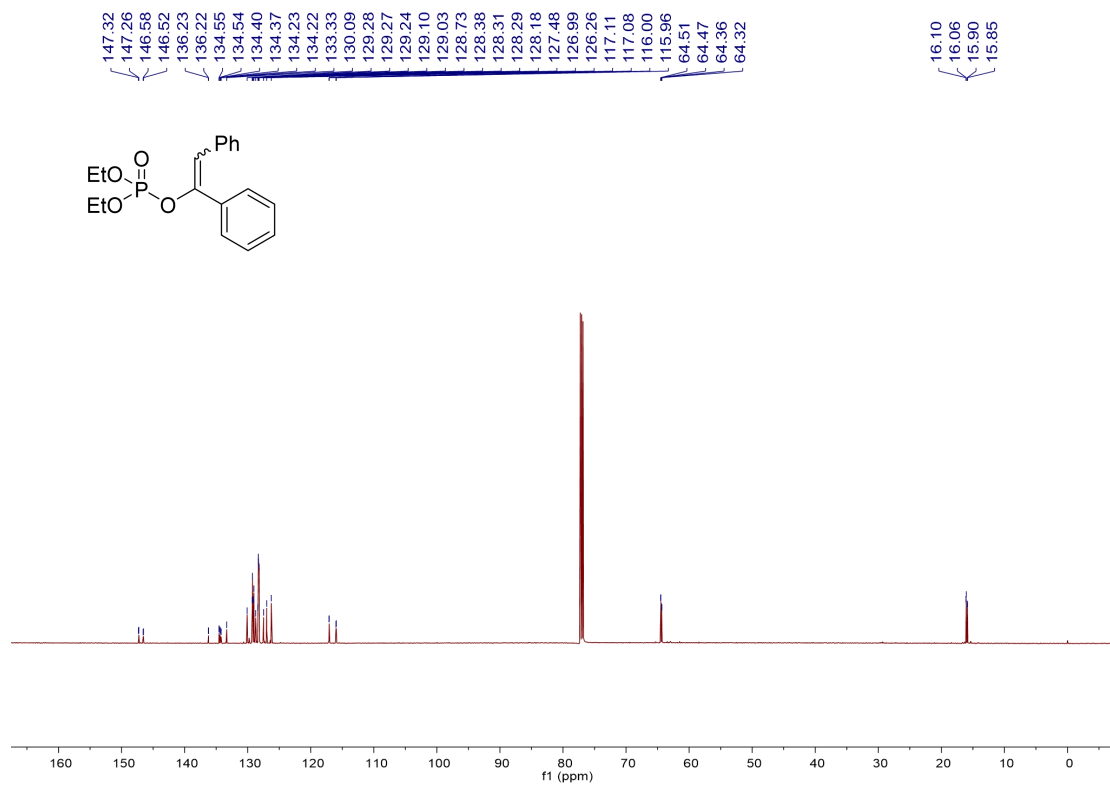
¹³C NMR of compound 3de



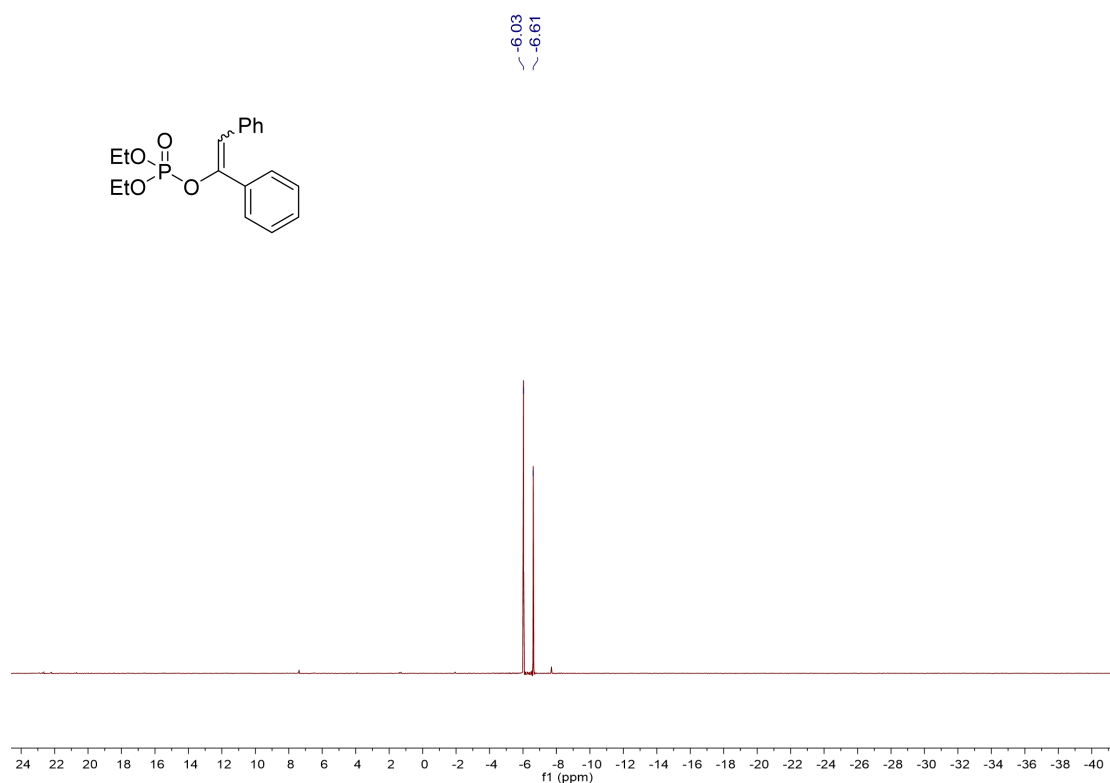
^{31}P NMR of compound 3de



^1H NMR of compound 3df



¹³C NMR of compound 3df



³¹P NMR of compound 3df