Supporting Information for

A highly efficient one-pot strategy to β-ketophosphonates: sliver/copper-catalyzed direct oxyphosphorylation of alkynes with *H*-phosphonates and oxygen in the air

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1. ^{31}P NMR stacks diagram for Scheme 2.

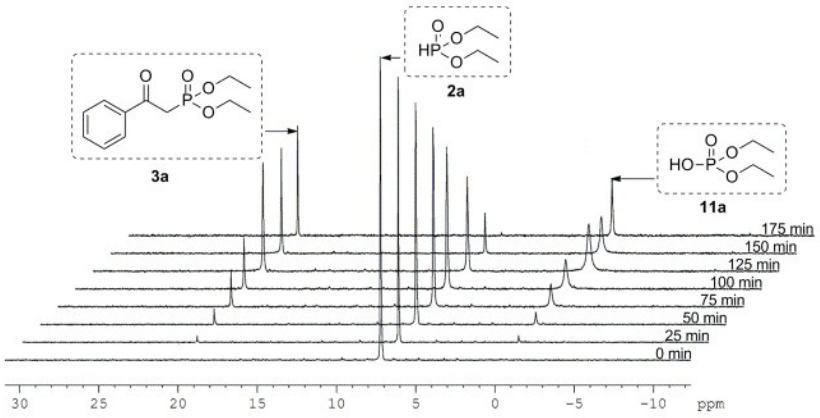


Fig. S1 ^{31}P NMR stacks diagram for Scheme 2. Reaction conditions: **1a** (0.3 mmol), **2a** (0.45 mmol), $AgNO_3$ (5.0 mol%), $CuSO_4$•$5H_2O$ (10.0 mol%), $K_2S_2O_8$ (4 equiv), CH_2Cl_2-H_2O (v/v = 1/1) (5.0 mL) at room temperature for 3 h. The whole process was monitored by ^{31}P NMR every 25 min (comp-**2a**, 7.1 ppm; comp-**3a**, 20.6 ppm; comp-**11a**, -0.8 ppm).

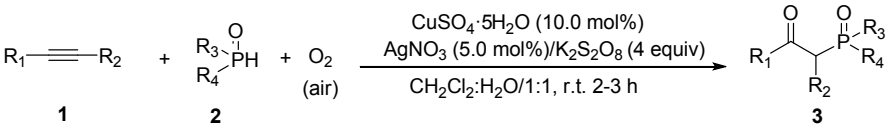
^{31}P NMR traced the progress of the reactions. Besides the main product **3a**, the ^{31}P NMR stacks diagram shown in Fig. S1 proved the formation of dialkyl phosphate **11a**. Besides the main product **3a**, the ^{31}P NMR stacks diagram shown in Fig. S1 proved the formation of dialkyl phosphate **11a**. In addition, ^{31}P NMR stacks diagram didn't offer support for the formation of alkynlphosphates in the reaction process. ^{31}P NMR signal from alkynylphosphate should appear at around -5 ppm (*J. Am. Chem. Soc.* 2009, **131**, 7956). But no trace of such a signal was detected by ^{31}P NMR. Zhao' group has reported that copper-catalyzed aerbic oxidative coupling of terminal alkynes with H-phosphonates can lead to the formation of alknylphosphonates in high yield (*J. Am. Chem. Soc.* 2009, **131**, 7956). However, with the same materials, alknylphosphonates were not detected under our different reaction conditions, implying that the target products, β-ketophosphonates, were not possibly formed via the hydration of alkynylphophonates in our cases. It is quite understandable, because we have already known that this one-pot reaction proceeds via a radical chain mechanism. The mechanism proposed in Scheme 2 does not support the formation of alkynylphosphate in the reaction process. The last strong support for our proposed mechaniem is from the formation of product **3aa**, which was synthesized starting from 1-phenyl-1-propyne, an internal aromatic alkyne. It is clear that it is only terminal alkynes that can react with *H*-

phosphonates via an aerobic oxidative coupling reaction to generate alkynylphosphonates (*J. Am. Chem. Soc.* 2009, **131**, 7956). Here, the formation of **3aa** once again offers support for the radical mechanism shown in Scheme 2 and excludes the possibility of formation of β-ketophosphonates via a hydration of alkynylphosphate.

2. General information

All commercial reagents and solvents were used without further purification. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ with tetramethylsilane (TMS) as the internal standard, and ^{31}P NMR spectra were obtained in $CDCl_3$ with H_3PO_4 as the internal standard. High resolution mass spectra (HRMS) were performed on a Q-TOF mass spectrometer. Column chromatography was carried out on columns of silica gel (200-300 mesh).

3. Experimental procedures for the synthesis of β-Ketophosphonates



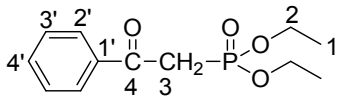
1 **2** **3**

R_1 = aryl, alkyl, phenolic; R_2 = H, Me; R_3 = aryl, aryloxy, alkoxy; R_4 = aryl, aryloxy, alkoxy

Alkynes **1** (0.3 mmol), *H*-phosphonates **2** (0.45 mmol), $AgNO_3$ (0.015 mmol), $CuSO_4 \cdot 5H_2O$ (0.03 mmol), and $K_2S_2O_8$ (4 equiv) were dissolved in round-bottomed flask and stirring at room temperature for 3 h in an air atmosphere. The reaction mixture was quenched with water (5.0 mL), extracted with ethyl acetate (3×5.0 mL). The combined organic layers were washed with brine (15.0 mL) and dried over anhydrous Na_2SO_4. After filtration, the solvent was evaporated in vacuo. The crude product was purified by silica gel chromatography (petroleum ether: ethyl acetate =1:1) to give the desired product.

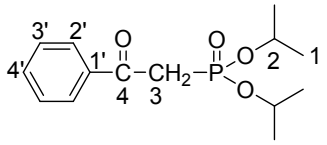
4. Characterization data for products

The Characterization data of 3a [1, 2]:



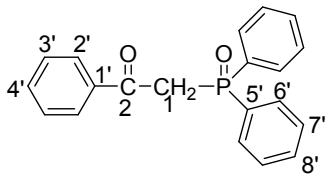
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.99 (d, 2H, 2'-H, J = 7.6 Hz), 7.56 (t, 1H, 4'-H, J = 7.2 Hz), 7.45 (t, 2H, 3'-H, J = 7.6 Hz), 4.15-4.08 (m, 4H, 2-H), 3.64 (d, 2H, 3-H, J = 22.8 Hz), 1.25 (t, 6H, 1-H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 192.0 (d, C-4, $J_{P\text{-}C}$ = 6.5 Hz), 136.5 (C-1'), 133.6 (C-4'), 129.0 (C-2'), 128.6 (C-3'), 62.7 (d, C-2, $J_{P\text{-}C}$ = 6.4 Hz), 38.4 (d, C-3, $J_{P\text{-}C}$ 129.3 Hz), 16.194 (d, C-1, $J_{P\text{-}C}$ = 6.2 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 21.3; HRMS: calcd for $C_{12}H_{17}O_4P$ $[M+H]^+$ 257.0937, found 257.0939.

The Characterization data of 3b [1, 2]:



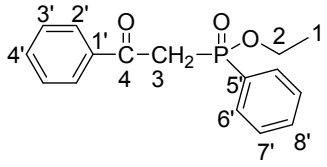
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 8.04 (d, 2H, 2'-H, J = 7.2 Hz), 7.59 (t, 1H, 4'-H, J = 7.6 Hz), 7.49 (t, 2H, 3'-H, J = 8.0 Hz), 4.80-4.69 (m, 2H, 2-H), 3.61 (d, 2H, 3-H, J = 22.8), 1.29 (dd, 12H, 1-H, J = 3.2 Hz, 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 192.1 (d, C-4, $J_{P\text{-}C}$ = 6.6 Hz), 136.7 (C-1'), 133.5 (C-4'), 129.1 (C-2'), 128.5 (C-3'), 71.5 (d, C-2, $J_{P\text{-}C}$ = 6.6 Hz), 39.7 (d, C-3, $J_{P\text{-}C}$ = 129.5 Hz), 23.8 (dd, C-1, $J_{P\text{-}C}$ = 5.1 Hz, 21.1Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 17.7; HRMS: calcd for $C_{12}H_{17}O_4P$ $[M+Na]^+$ 307.1070, found 307.1069.

The Characterization data of 3c [1, 3]:



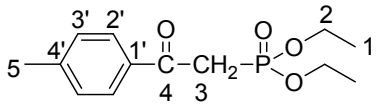
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.95 (d, 2H, 2'-H, J = 7.6 Hz), 7.84-7.79 (m, 4H, 6'-H), 7.52-7.46 (m, 3H, 3', 4'-H), 7.42-7.35 (m, 6H, 7', 8'-H), 4.25 (d, 2H, 2-H, J = 15.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 193.1 (d, C-2, $J_{P\text{-}C}$ = 5.1 Hz), 136.9 (C-1'), 133.6 (C-4'), 132.2 (d, C-8', $J_{P\text{-}C}$ = 2.7 Hz), 131.7 (d, C-5', $J_{P\text{-}C}$ = 103.1 Hz), 131.2 (d, C-6', $J_{P\text{-}C}$ = 9.9 Hz), 129.2 (C-2'), 128.6 (d, C-7', $J_{P\text{-}C}$ = 12.4 Hz), 128.5 (C-3'), 42.9 (d, C-1, $J_{P\text{-}C}$ = 52.4Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 28.0; HRMS: calcd for $C_{20}H_{17}O_2P$ $[M+H]^+$ 321.1039, found 321.1040

The Characterization data of 3d [1, 2]:



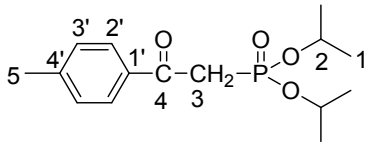
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.96 (dd, 2H, 2'-H, J = 0.8, 1.2 Hz), 7.81-7.76 (m, 2H, 6'-H), 7.57-7.52 (m, 2H, 3'-H), 7.48-7.41 (m, 4H, 4', 7', 8'-H), 4.17-3.90 (m, 2H, 3-H), 3.80 (dd, 2H, 2-H, J_{P-H}= 4.4Hz, 18.8Hz), 1.26 (t, 3H, 1-H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 192.2 (d, C-4, J_{P-C} = 5.5 Hz), 136.8 (C-1'), 133.5 (C-4'), 132.7 (d, C-8', J_{P-C} = 2.8 Hz), 131.8 (d, C-6', J_{P-C} = 10.2 Hz), 130.1 (d, C-5', J_{P-C} = 132.0 Hz) 129.1 (C-2'), 128.6 (d, C-7', J_{P-C} = 13.2 Hz) 128.5 (d, C-3'), 61.5 (d, C-2, J_{P-C} = 6.3 Hz), 43.0 (d, C-3, J_{P-C} = 85.8 Hz), 16.3 (d, C-1, J_{P-C} = 6.5 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 34.4; HRMS: calcd for $C_{16}H_{17}O_3P$ $[M+Na]^+$ 311.0808, found 311.0811

The Characterization data of 3e[4, 5, 11]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.92 (d, 2H, 3'-H, J = 8.0 Hz), 7.28 (d, 2H, 2'-H, J = 8.0 Hz), 4.17-4.10 (m, 4H, 2-H), 3.61 (d, 2H, 3-H, J = 22.8 Hz), 2.42 (s, 3H, 5-H), 1.29 (t, 6H, 1-H, J = 6.8Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.5 (d, C-4, J_{P-C} = 3.2Hz), 144.6 (C-4'), 134.1 (C-1'), 129.3 (C-3'), 129.2 (C-2'), 62.6 (d, C-2, J_{P-C} = 6.5 Hz), 38.4 (d, C-3, J_{P-C} = 129.2 Hz), 21.7 (C-5), 16.2 (d, C-1, J_{P-C} = 6.2 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 20.0; HRMS: calcd for $C_{13}H_{19}O_4P$ $[M+H]^+$ 271.1094, found 271.1095.

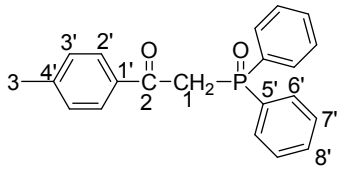
The Characterization data of 3f [2]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.91 (d, 2H, 3'-H, J = 8.4 Hz), 7.26 (d, 2H, 2'-H, J = 8.0 Hz), 4.75-4.67 (m, 2H, 2-H), 3.55 (d, 2H, 3-H, J = 23.2 Hz), 2.40 (s, 3H, 5-H), 1.27 (dd, 12H, J = 4.0 Hz, 6.4Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.6 (d, C-4, J_{P-C} = 6.7 Hz), 144.5 (C-4'), 134.3 (C-1'), 129.3 (C-3'), 129.2 (C-2'), 71.4 (d, C-2, J_{P-C} = 6.7 Hz), 39.6 (d, C-3, J_{P-C} = 129.4

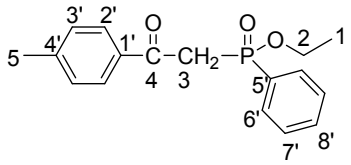
Hz), 23.8 (dd, C-1, $J_{P\text{-}C}$ = 5.2 Hz, 21.3 Hz), 21.6 (C-5); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 17.9; HRMS: calcd for $C_{15}H_{23}O_4P$ $[M+Na]^+$ 321.1226, found 321.1228.

The Characterization data of 3g [6]:



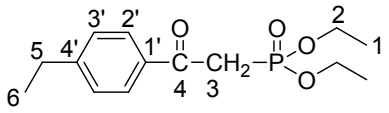
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.85-7.80 (m, 6H, 7', 8'-H), 7.45 (t, 2H, 3'-H, J = 7.2 Hz), 7.36-7.32 (m, 4H, 6'-H), 7.12 (d, 2H, 2'-H, J = 8.0 Hz), 4.33 (d, 2H, 1-H, J = 15.6 Hz), 2.33 (s, 3H, 3-H); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 193. (C-2), 144.7 (C-4'), 134.3 (C-1'), 132.148 (d, C-8', $J_{P\text{-}C}$ = 2.6 Hz), 131.4 (d, C-6', $J_{P\text{-}C}$ = 10.1 Hz), 131.3 (C-2', C-5', $J_{P\text{-}C}$ = 104.1 Hz), 129.4 (C-3'), 129.2 (C-2'), 128.6 (d, C-7', $J_{P\text{-}C}$ = 12.4 Hz), 42.9 (d, C-1, $J_{P\text{-}C}$ = 60.1 Hz), 21.6 (C-3); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 29.4; HRMS: calcd for $C_{21}H_{19}O_2P$ $[M+H]^+$ 335.1195, found 335.1197

The Characterization data of 3h:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.85 (d, 2H, 6'-H, J = 8.4Hz),7.80-7.75 (m, 2H, 7'-H), 7.55-7.51 (m, 1H, 8'-H), 7.46-7.42 (m, 2H, 3'-H), 7.21 (d, 2H, J = 8.0 Hz), 4.12-3.88 (m, 2H, 2-H), 3.76 (dd, 2H, 3-H, J = 4.4 Hz, 18.4 Hz), 1.25 (t, 3H, 1-H, J = 6.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.7 (d, C-4, $J_{P\text{-}C}$ = 5.5Hz), 144.4 (C-4'), 134. 4 (C-1'), 132.6 (d, C-8', $J_{P\text{-}C}$= 2.8 Hz), 131.8 (d, C-6', $J_{P\text{-}C}$ = 10.1 Hz), 130.2 (d, C-5', $J_{P\text{-}C}$ = 131.9 Hz,) 129.2 (C-3'), 129.2 (C-2'), 128.6 (d, C-7', $J_{P\text{-}C}$ = 13.2 Hz), 61.4 (d, C-2, $J_{P\text{-}C}$ = 6.2 Hz), 42.9 (d, C-3, $J_{P\text{-}C}$ = 86.0 Hz), 21.7 (C-5), 16.3 (d, C-1, $J_{P\text{-}C}$ = 6.5 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 34.6; HRMS: calcd for $C_{17}H_{19}O_3P$ $[M+Na]^+$ 325.0964, found 325.0966

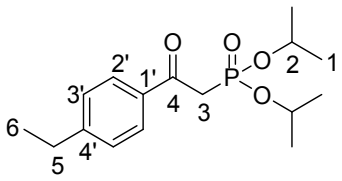
The Characterization data of 3i:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.95 (d, 2H, 3'-H, J = 8.4 Hz), 7.31 (d, 2H, 2'-H, J =

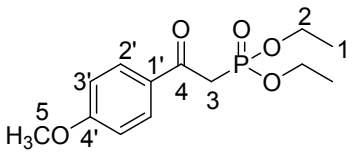
8.4 Hz), 4.18-4.11 (m, 4H, 2-H), 3.62 (d, 2H, 3-H, J = 22.8 Hz), 2.75-2.69 (q, 2H, 5-H), 1.31-1.25 (m, 9H, 1, 6-H, overlap); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.5 (C-4, $J_{P\text{-}C}$ = 6.5 Hz), 150.8 (C-4'), 134.3 (C-1'), 129.3 (C-2'), 128.1 (C-3'), 62.6 (d, C-2, $J_{P\text{-}C}$ = 6.5Hz), 38.4 (d, C-3, $J_{P\text{-}C}$ = 129.1 Hz), 28.9 (C-5), 16.3 (d, C-1, $J_{P\text{-}C}$ = 6.4 Hz), 15.1 (C-6); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 20.3; HRMS: calcd for $C_{14}H_{21}O_4P$ $[M+H]^+$ 285.1250, found 285.1251.

The Characterization data of 3j:



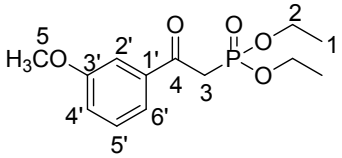
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.89 (d, 2H, 3'-H, J = 8.4 Hz), 7.23 (d, 2H, 2'-H, J = 8.4Hz), 4.73-4.61 (m, 2H, 2-H), 3.55-3.49 (d, 2H, 3-H, J = 22.8), 2.68-2.62 (q, 2H, 5-H), 1.24-1.18 (m, 15H, 1,6-H); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.6 (d, C-4, $J_{P\text{-}C}$ = 6.5 Hz), 150.5 (C-4'), 134.4 (C-1'), 129.3 (C-2'), 127.9 (C-3'), 71.3 (d, C-2, $J_{P\text{-}C}$ = 6.6 Hz), 39.6 (d, C-3, $J_{P\text{-}C}$ = 129.5 Hz), 28.9 (C-5), 23.8 (dd, C-1, $J_{P\text{-}C}$ = 3.7, 5.1Hz), 15.1 (C-6); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 18.3; HRMS: calcd for $C_{16}H_{25}O_4P$ $[M+Na]^+$ 335.1383, found 335.1384.

The Characterization data of 3k [2, 4]:



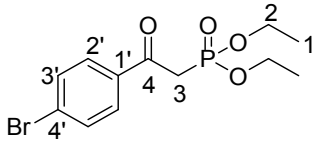
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.98 (d, 2H, 2'-H, J = 8.8 Hz), 6.93 (d, 2H, 3'-H, J = 8.8 Hz), 4.15-4.08 (m, 4H, 2-H), 3.854 (s, 3H, 5-H), 3.57 (d, 2H, 3-H, J = 22.4 Hz), 1.27 (t, 6H, 1-H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 190.26 (d, C-4, $J_{P\text{-}C}$ = 6.3), 163.9 (C-4'), 131.5 (C-2'), 129. 6 (C-1'), 62.6 (d, C-2, $J_{P\text{-}C}$ = 7.5Hz), 55.5 (C-5), 38.2 (d, C-3, $J_{P\text{-}C}$ = 28.9 Hz), 16.2 (d, C-1, $J_{P\text{-}C}$ = 6.2 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 20.4; HRMS: calcd for $C_{13}H_{19}O_5P$ $[M+Na]^+$ 309.0862, found 309.0862.

The Characterization data of 3l [7]:



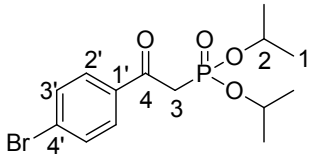
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.53 (d, 1H, 6'-H, J = 2.4 Hz), 7.52 (s, 1H, 2'-H), 7.38 (t, 1H, 5'-H, J = 7.6 Hz), 7.15-7.12 (q, 1H, 4'-H), 4.17-4.10 (m, 4H, 2-H), 3.85 (s, 3H, 5-H), 3.62 (d, 2H, 3-H, J = 22.4 Hz), 1.28 (t, 6H, 1-H, J = 6.8Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.8 (d, C-4, $J_{P\text{-}C}$ = 6.6Hz), 159.8 (C-3'), 137.8 (d, C-1', $J_{P\text{-}C}$ = 2.1 Hz), 129.6 (C-2'), 121.9 (C-5'), 120.4 (C-6'), 112.8 (C-4'), 62.6 (d, C-2, $J_{P\text{-}C}$ = 6.5Hz), 55.4 (C-5), 38.5 (d, C-3, J = 129.4 Hz), 16.2 (d, C-1, J = 6.3 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 20.0; HRMS: calcd for $C_{13}H_{19}O_5P$ $[M+H]^+$ 287.1043, found 287.1043.

The Characterization data of 3m [7]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.90 (d, 2H, 2'-H, J = 8.4 Hz), 7.639 (d, 2H, 4'-H, J = 8.8 Hz), 4.19-4.12 (m, 4H, 2-H), 3.61 (d, 2H, 3-H, J = 22.8 Hz), 1.30 (t, 6H, 1-H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 190.9 (d, C-4, $J_{P\text{-}C}$ = 6.7 Hz), 135.2 (C-1'), 131.9 (C-3'), 130.6 (C-2'), 129.1 (C-4'), 62.8 (d, C-2, $J_{P\text{-}C}$ = 6.5 Hz), 38.6 (d, C-3, $J_{P\text{-}C}$ = 128.4 Hz), 16.3 (d, C-1, $J_{P\text{-}C}$ = 6.2 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 20.3; HRMS: calcd for $C_{12}H_{16}BrO_4P$ $[M+H]^+$ 335.0042, found 335.0041.

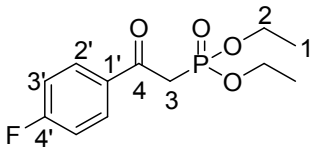
The Characterization data of 3n [2]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.85 (d, 2H, 2'-H, J = 8.4Hz), 7.56 (d, 2H, 3'-H, J = 8.8 Hz), 4.73-4.62 (m, 2H, 2-H), 3.51 (d, 2H, 3-H, J = 23.3 Hz), 1.23 (dd, 12H, 1-H, J = 2.4 Hz, 6.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 191.0 (d C-4, $J_{P\text{-}C}$ = 6.6 Hz), 135.3 (C-1'), 131.8 (C-3'), 130.7 (C-2'), 128.8 (C-4'), 71.6 (d, C-2, $J_{P\text{-}C}$ = 6.7 Hz), 39.9 (d, C-3, $J_{P\text{-}C}$ = 128.7 Hz), 23.8 (dd,

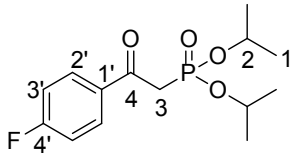
C-1, $J_{P\text{-}C}$ = 5.2 Hz, 18.9 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 17.2; HRMS: calcd for $C_{14}H_{20}BrO_4P$ $[M+H]^+$ 385.0175, found 385.0175.

The Characterization data of 3o [7]:



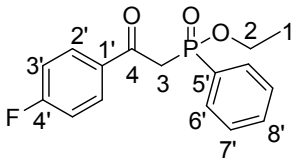
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 8.05-8.02 (q, 2H, 2'-H), 8.04 (d, 1H, 2'-H, J = 5.6 Hz), 8.02 (d, 1H, 2'-H, J = 5.2 Hz), 7.14-7.10 (t, 2H, 3'-H), 7.12 (t, C-3', J = 8.8 Hz) 4.15-4.07 (m, 4H, 2-H), 3.58 (d, 2H, 3-H, J = 22.8 Hz), 1.26 (t, 6H, 1-H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 190.3 (d, C-4, $J_{P\text{-}C}$ = 6.4Hz), 166.1 (d, C-4', $J_{F\text{-}C}$ = 254.6 Hz), 132.9 (C-1'), 131.8 (d, C-2', $J_{F\text{-}C}$ = 9.5 Hz), 115.7 (d, C-3', $J_{F\text{-}C}$ = 21.9 Hz), 62.7 (d, C-2, $J_{P\text{-}C}$ = 6.5 Hz), 38. 6 (d, C-3, $J_{P\text{-}C}$ = 128.7 Hz), 16.2 (d, C-1, $J_{P\text{-}C}$ = 6.4Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 19.6; HRMS: calcd for $C_{12}H_{16}FO_4P$ $[M+H]^+$ 275.0843, found 275.0847.

The Characterization data of 3p [2]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 8.04 (d, 1H, 2'-H, J = 5.6Hz), 8.02 (d, 1H, 2'-H, J = 5.2 Hz), 7.11 (t, 2H, 3'-H, J = 8.8 Hz), 4.74-4.63 (m, 2H, 2-H), 3.53 (d, 2H, 5-H, J = 22.8 Hz), 1.24 (dd, 12H, 1-H, J = 2.8Hz, 6.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 190.5 (d, C-4, $J_{P\text{-}C}$ = 6.5 Hz), 167.5 (d, C-4', $J_{F\text{-}C}$ = 254.1 Hz), 133.0 (C-1'), 131.9 (d, C-2', $J_{F\text{-}C}$ = 9.4 Hz),115.6 (d, C-3', $J_{F\text{-}C}$ = 21.7 Hz), 71.5 (d, C-2, $J_{P\text{-}C}$ = 6.6 Hz), 39.8 (d, C-3, $J_{P\text{-}C}$ = 128.9 Hz), 23.8 (dd, C-1, $J_{P\text{-}C}$ = 4.0, 5.1 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 16.3; HRMS: calcd for $C_{14}H_{20}FO_4P$ $[M+Na]^+$ 325.0975, found 325.0979.

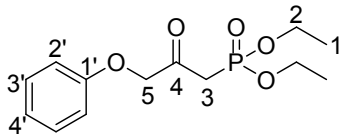
The Characterization data of 3q:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 8.03-7.99 (m, 2H, 2'-H), 7.82-7.77 (m, 2H, 6'-H),

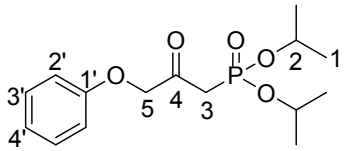
7.58-7.54 (m, 1H, 8'-H), 7.48-7.44 (m, 2H, 7'-H), 7.10 (t, 2H, J = 8.4 Hz), 4.19-3.91 (m, 2H, 2-H), 3.81 (d, 2H, 3-H, J = 4.6 Hz), 1.26 (t, 3H, 1-H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 190.7 (d, C-4, $J_{P\text{-}C}$ = 5.4 Hz), 166.0 (d, C-4', $J_{F\text{-}C}$ = 254.4 Hz), 133.2 (C-1'), 132.8 (d, C-8', $J_{P\text{-}C}$ = 2.8 Hz), 131.9 (d, C-2', $J_{P\text{-}C}$ = 3.8 Hz), 131.9 (d, C-2', $J_{P\text{-}C}$ = 15.9 Hz), 129.8 (d, C-5', $J_{P\text{-}C}$ = 132.7 Hz), 128.7 (d, C-7', $J_{P\text{-}C}$ = 13.2 Hz), 115.7 (d, C-3', $J_{P\text{-}C}$= 21.8 Hz), 61.7 (d, C-2, $J_{P\text{-}C}$= 6.2 Hz), 43.1 (d, C-3, $J_{P\text{-}C}$= 85.6 Hz), 16.3 (d, C-1, $J_{P\text{-}C}$= 6.6 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 34.4; HRMS: calcd for $C_{16}H_{16}FO_3P$ $[M+H]^+$ 307.0894, found 307.0895.

The Characterization data of 3r [8]:



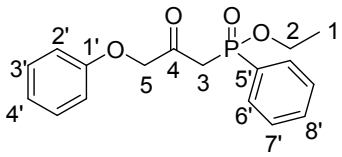
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.34-7.28 (m, 2H, 3'-H), 7.02 (t, 1H, 4'-H, J = 7.2 Hz), 6.94-6.92 (m, 2H, 2'-H), 6.94 (dd, 2H, 2'-H, J = 0.8 Hz, J = 7.6 Hz), 4.74 (s, 2H, 5-H), 4.22-4.15 (m, 4H, 2-H), 3.30 (d, 2H, 3-H, J = 22.8 Hz), 1.34 (t, 6H, 1-H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 198.7 (d, C-4, $J_{P\text{-}C}$ = 6.6Hz), 157.5 (C-1'), 129.7 (C-3'), 121.9 (C-4'), 114.6 (C-2'), 72.7 (C-5), 62. 9 (d, C-2, $J_{P\text{-}C}$ = 6.3Hz), 38.6 (d, C-3, $J_{P\text{-}C}$ = 127.0 Hz), 16.3 (d, C-1, $J_{P\text{-}C}$ = 6.3 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 21.344; HRMS: calcd for $C_{13}H_{19}O_5P$ $[M+H]^+$ 287.1043, found 287.1044.

The Characterization data of 3s:



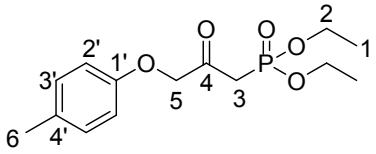
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7. 29-7.25 (m, 2H, 3'-H), 6.97 (t, 1H, 4'-H, J = 7.2 Hz), 6.9 (d, 2H, 2'-H, J = 8.0 Hz), 4.77-4.69 (m, 4H, 2,5-H), 3.20 (d, 2H, 3-H, J = 22.8 Hz), 1.31 (d, 12H, 1-H, J = 6.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 198.3 (d, C-4, $J_{P\text{-}C}$ = 6.5Hz), 157.6 (C-1'), 129.6 (C-3'), 121.7 (C-4'), 114.6 (C-2'), 72.6 (C-5), 71.8 (d, C-2, $J_{P\text{-}C}$ = 6.6 Hz), 39.9 (d, C-3, $J_{P\text{-}C}$ = 127.4 Hz), 23.9 (dd, C-1, $J_{P\text{-}C}$ = 4.9 Hz, 15.7 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 16.7; HRMS: calcd for $C_{15}H_{23}O_5P$ $[M+H]^+$ 315.1356, found 315.1353.

The Characterization data of 3t:



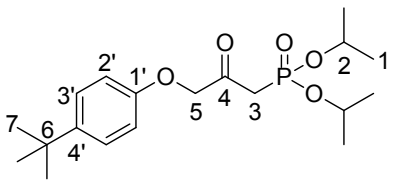
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.77-7.72 (m, 2H, 6'-H), 7.52-7.48 (m, 1H, 8'-H), 7.44-7.39 (m, 2H, 7'-H), 7.22-7.180 (m, 2H, 3'-H), 6.91 (t, 2H, 4'-H, J = 7.2 Hz), 6.81 (d, 2H, 2'-H, J = 8.0 Hz), 4.66 (d, 2H, 5-H, J = 4.8 Hz), 4.13-3.86 (m, 2H, 2-H), 3.37 (d, 2H, 3-H, J = 18.0 Hz), 1.24 (t, 3H, 1-H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 198.5 (C-4), 157.5 (C-1'), 132.9 (d, C-8', $J_{P\text{-}C}$= 2.8 Hz), 131.6 (d, C-6', $J_{P\text{-}C}$ = 10.4 Hz), 129.8 (d, C-5', $J_{P\text{-}C}$ = 132.8 Hz), 129.5 (C-3'), 128.8 (d, C-7', $J_{P\text{-}C}$ = 13.2 Hz), 121.6 (C-4'), 114.6 (C-2'), 73.1 (C-5), 61.7 (d, C-2, $J_{P\text{-}C}$ = 6.1Hz) 42.9 (d, C-3, $J_{P\text{-}C}$ = 84.3 Hz), 16.3 (d, C-5, $J_{P\text{-}C}$ = 6.5 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 33.4; HRMS: calcd for $C_{17}H_{19}O_4P$ $[M+H]^+$ 341.0913, found 341.0922.

The Characterization data of 3u [10]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.10 (d, 2H, 4'-H, J= 8.4 Hz), 6.82 (d, 2H, 2'-H, J = 8.8 Hz), 4.69 (s, 2H, 5-H), 4.21-4.14 (m, 4H, 2-H), 3.28 (d, 2H, 3-H, J = 22.8 Hz), 2.30 (s, 3H, 6-H), 1.34 (t, 6H, 1-H, J = 6.8Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 198.9 (d, C-4, $J_{P\text{-}C}$ = 6.5Hz), 155.4 (C-1'), 131.2 (C-4'), 130.1 (C-3'), 114.4 (C-2'), 72.9 (C-5), 62.8 (d, C-2, $J_{P\text{-}C}$ = 6.3 Hz), 38.6 (d, C-3, $J_{P\text{-}C}$ = 127.0 Hz), 20.5 (C-6), 16.3 (d, C-1, $J_{P\text{-}C}$ = 6.3 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 18.9; HRMS: calcd for $C_{14}H_{21}O_5P$ $[M+H]^+$ 301.1199, found 301.1200.

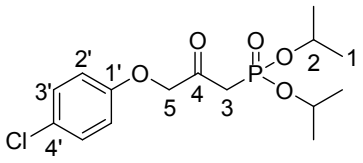
The Characterization data of 3v:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.30 (d, 2H, 3'-H, J = 8.8 Hz), 6.85 (d, 2H, 2'-H, J = 8.8 Hz), 4.82-4.74 (m, 2H, 2-H), 4.72 (s, 2H, 5-H), 3.22 (d, 2H, 3-H, J = 22.8 Hz), 1.34 (d, 12H, 1-H, J = 6.0 Hz), 1.30 (s, 9H, 7-H); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 198.7 (d, C-4, $J_{P\text{-}C}$ = 6.5 Hz),

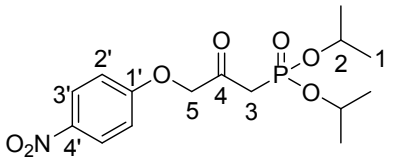
155.4 (C-1'), 144.5 (C-4'), 126.4 (C-3'), 114.1 (C-2'), 72.9 (C-5), 71.7 (d, C-2, J_{P-C} = 6.6 Hz), 39.9 (d, C-3, J_{P-C} = 127.5 Hz), 34.1 (C-6), 31.7 (C-7), 23.9 (dd, C-1, J_{P-C} = 5.0 Hz, 16.2 Hz); ^{1}P NMR ($CDCl_3$, 400 MHz) δ: 16.6; HRMS: calcd for $C_{19}H_{31}O_5P$ $[M+H]^+$ 371.1982, found 371.1983.

The Characterization data of 3w:



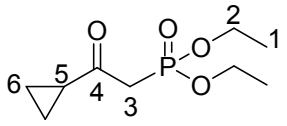
Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.24 (d, 2H, 3'-H, J = 9.2 Hz), 6.86 (d, 2H, 2'-H, J = 8.8 Hz), 4.81-4.71 (m, 4H, 2,5-H), 3.19 (d, 2H, 3-H, J = 22.8 Hz), 1.34 (dd, 12H. 1-H. J = 1.6, 6.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 197.6 (d, C-4, J_{P-C} = 6.5 Hz), 156.3 (C-1'), 129. 5 (C-3'), 126.6 (C-4'), 115.9 (C-2'), 72.8 (C-5), 71.9 (d, C-2, J_{P-C} = 6.7 Hz), 40.1 (d, C-3, J_{P-C} = 127.1 Hz), 23.9 (dd, C-1, J_{P-C} = 5.0, 14.5 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 16.3; HRMS: calcd for $C_{15}H_{22}ClO_5P$ $[M+H]^+$ 349.0966, found 349.0974.

The Characterization data of 3x:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 8.221 (d, 2H, 3'-H, J = 9.2 Hz), 7.04 (d, 2H, 2'-H, J = 9.2 Hz), 4.96 (s, 2H, 5-H), 4.84-4.73 (m, 2H, 2-H), 3.21 (d, 2H, 3-H, J_{P-C}= 23.2 Hz), 1.37 (dd, 12H, 1-H, J = 4.0 Hz, 6.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 196.1 (d, C-4, J_{P-C} = 6.3 Hz), 162.5 (C-1'), 142.2 (C-4'), 125.9 (C-3'), 114.7 (C-2'), 72.7 (C-5), 72.2 (d, C-2, J_{P-C} = 6.7 Hz), 40.4 (d, C-3, J_{P-C} = 126.3 Hz), 23.9 (dd, C-1, J_{P-C} = 4.8 Hz, 12.7 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 16.0; HRMS: calcd for $C_{15}H_{22}NO_7P$ $[M+Na]^+$ 371.0786, found 371.0783.

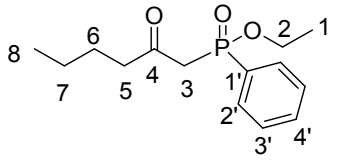
The Characterization data of 3y [9, 11]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 4.16-4.09 (m, 4H, 2-H), 3.17 (d, 2H, 3-H, J = 22.4 Hz), 2.19-2.13 (m, 1H, 5-H), 1.30 (t, 6H, 1-H, J = 7.2 Hz), 1.08-1.06 (m, 2H, 6-H), 0.96-0.91 (m, 2H,

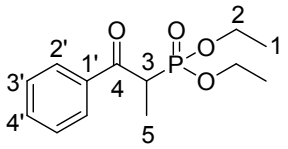
6-H); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 202. 1 (d, C-4, $J_{P\text{-}C}$ = 5.7 Hz), 62.5 (d, C-2, $J_{P\text{-}C}$ = 6.4 Hz), 43.3 (d, C-3, $J_{P\text{-}C}$ = 126.9 Hz), 21.7 (C-5), 16.3 (C-1), 12.0 (C-6); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 20.1; HRMS: calcd for $C_9H_{17}O_4P$ $[M+H]^+$ 221.0937, found 221.0937.

The Characterization data of 3z [1]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 7.76 (dd, 2H, 2'-H, J = 7.6 Hz, J = 12.4 Hz), 7.55-7.51 (m, 1H, 4'-H), 7.47-7.42 (m, 2H, 3'-H), 4.15-3.87 (m, 2H, 2-H) 3.25 (d, 2H, 3-H, J = 18.4 Hz), 2.62-2.45 (m, 2H, 5-H), 1.50-1.42 (m, 2H, 6-H), 1.29-1.17 (m, 5H, 7, 1-H), 0.82 (t, 3H, 8-H, J = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz); δ: 202.5 (C-4), 132.8 (d, C-4', $J_{P\text{-}C}$ = 2.8 Hz), 131.7 (d, C-2', $J_{P\text{-}C}$ = 10.3 Hz), 129.9 (d, C-1', $J_{P\text{-}C}$ = 131.7 Hz), 128.7 (d, C-3', $J_{P\text{-}C}$ = 13.1 Hz), 61.5 (d, C-2, $J_{P\text{-}C}$ = 6.1Hz) 46.5 (d, C-3, $J_{P\text{-}C}$ = 85.1 Hz), 44.5 (C-5), 25.4 (C-6), 21.9 (C-7), 16.3 (d, C-1, $J_{P\text{-}C}$ = 6.6 Hz), 13.8 (C-8); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 34.6; HRMS: calcd for $C_{14}H_{21}O_3P$ $[M+H]^+$ 269.1301, found 269.1303.

The Characterization data of 3aa [2]:



Yellow oil, ^{1}H NMR ($CDCl_3$, 400 MHz,) δ: 8.02 (d, 2H, 2'-H, J = 7.2 Hz), 7.59 (t, 1H, 4'-H, J = 7.6 Hz), 7.49 (t, 2H, 3'-H, J = 7.6 Hz), 4.26-4.06 (m, 5H, 2, 3-H), 1.56 (dd, 3H, 5-H, J = 5.6 Hz, 18.0 Hz), 1.30 (t, 6H, 1-H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$,100 MHz); δ: 196.5 (d, C-4, $J_{P\text{-}C}$ = 5.0 Hz), 136.9 (C-1'), 133.3 (C-4'), 128.8 (C-2'), 128.5 (C-3'), 62.7 (dd, C-2, J = 6.8Hz, $J_{P\text{-}C}$ = 10.3Hz), 41.3 (d, C-3, $J_{P\text{-}C}$ = 129.5 Hz), 16.3 (dd, C-1, $J_{P\text{-}C}$ = 6.0 Hz, 14.5Hz), 12.2 (d, C-5, $J_{P\text{-}C}$ = 6.6 Hz); ^{31}P NMR ($CDCl_3$, 400 MHz) δ: 23.4; HRMS: calcd for $C_{13}H_{19}O_4P$ $[M+H]^+$ 271.1094, found 271.1099.

References:

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5. ^{1}H NMR, ^{13}C NMR，and HRMS (ESI) copies of products

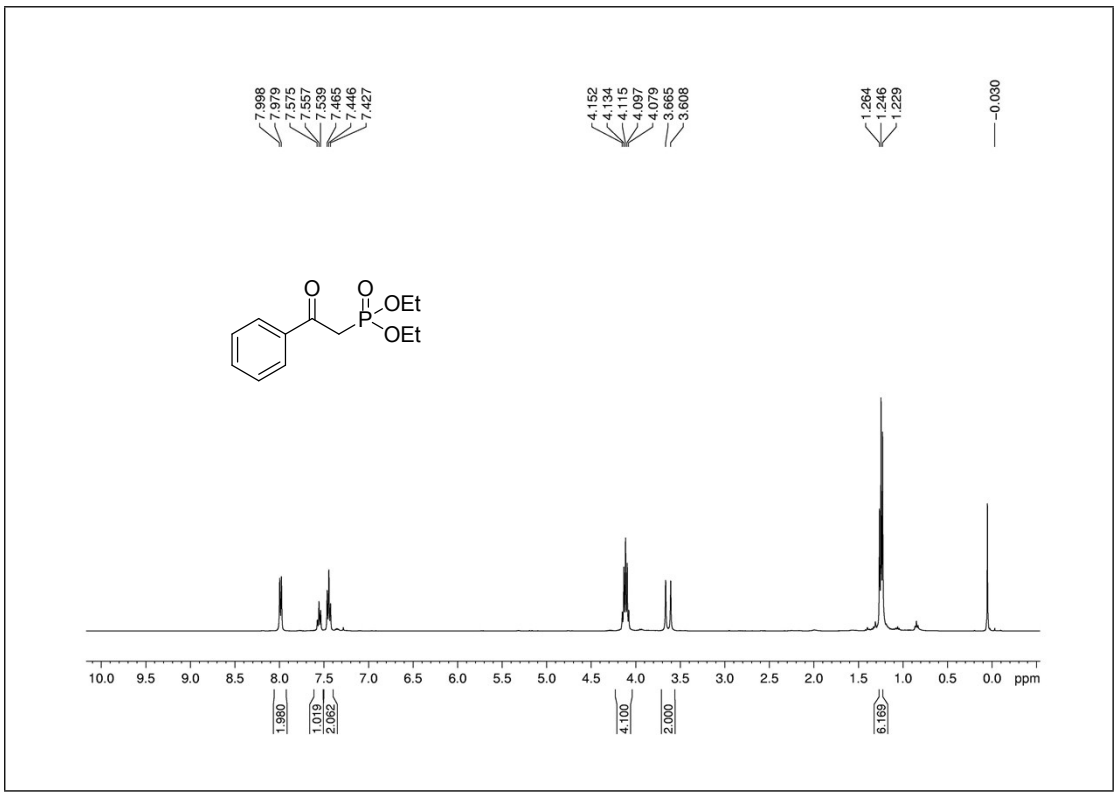

Fig. 1 ^{1}H NMR spectrum of compound **3a**

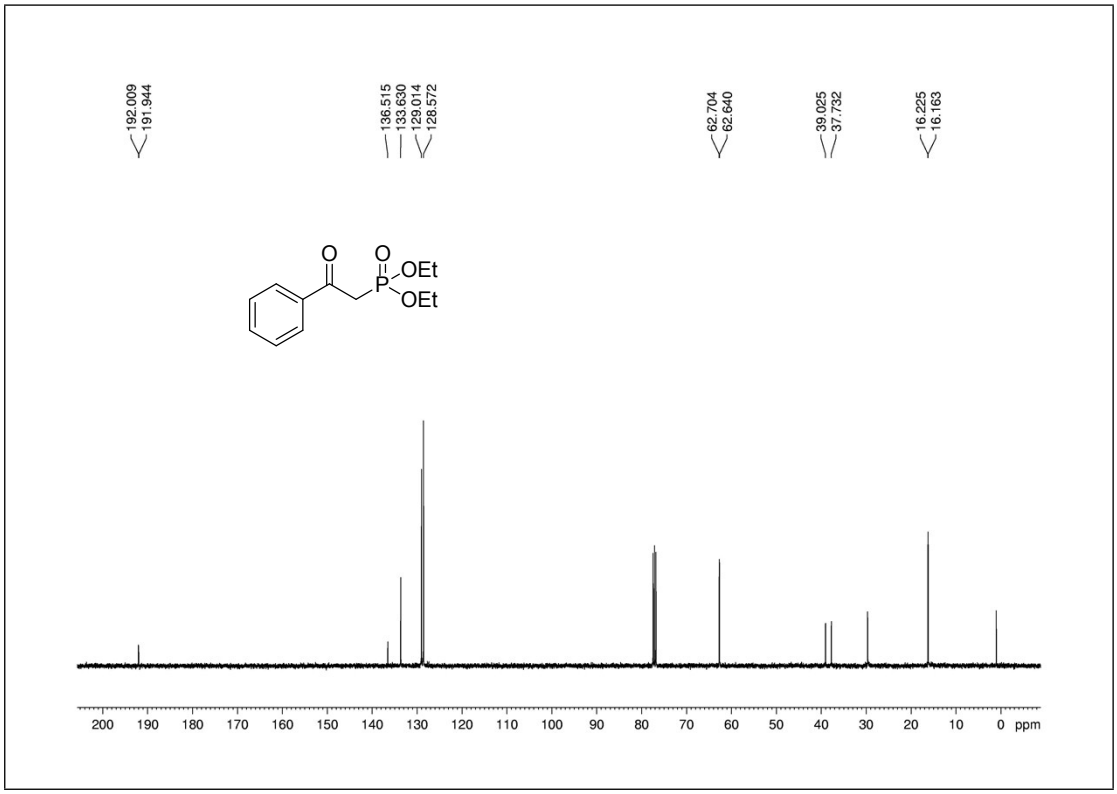

Fig. 2 ^{13}C NMR spectrum of compound **3a**

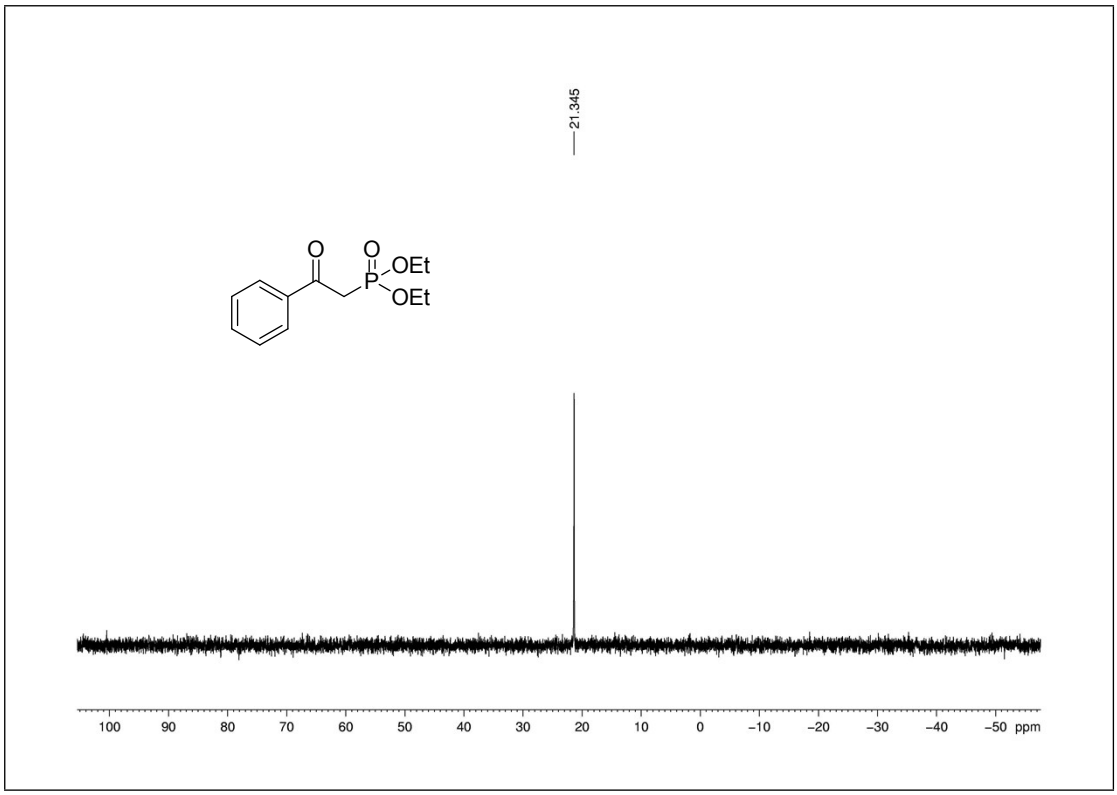

Fig. 3 ^{31}P NMR spectrum of compound **3a**

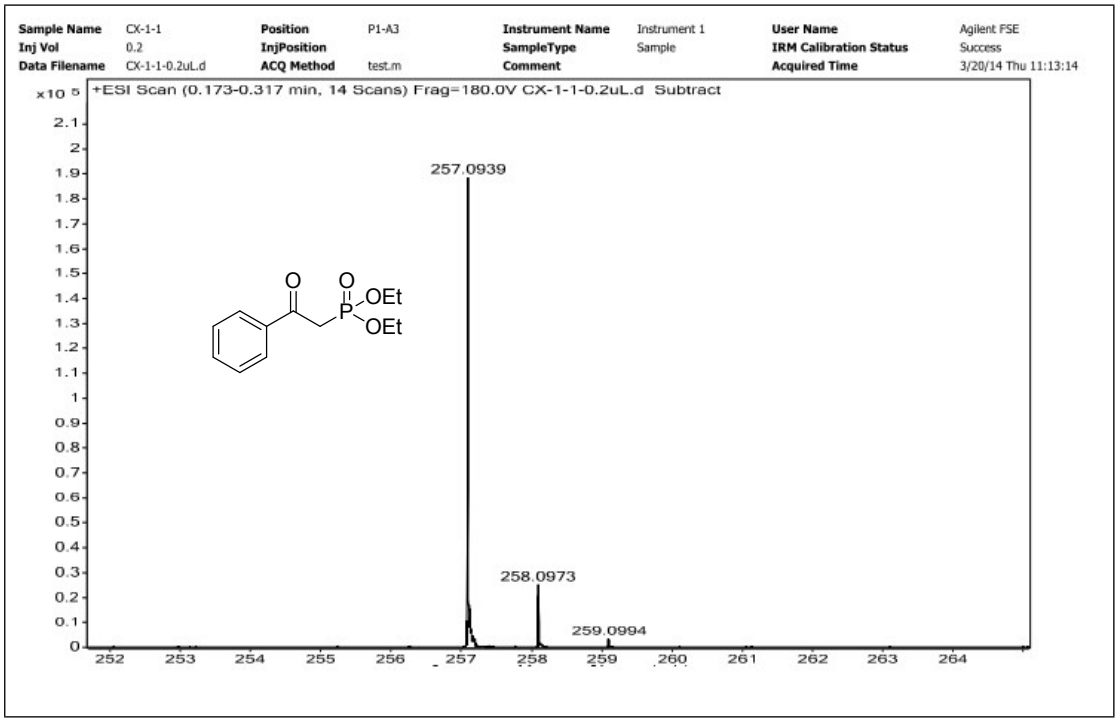

Fig. 4 HRMS spectrum of compound **3a**

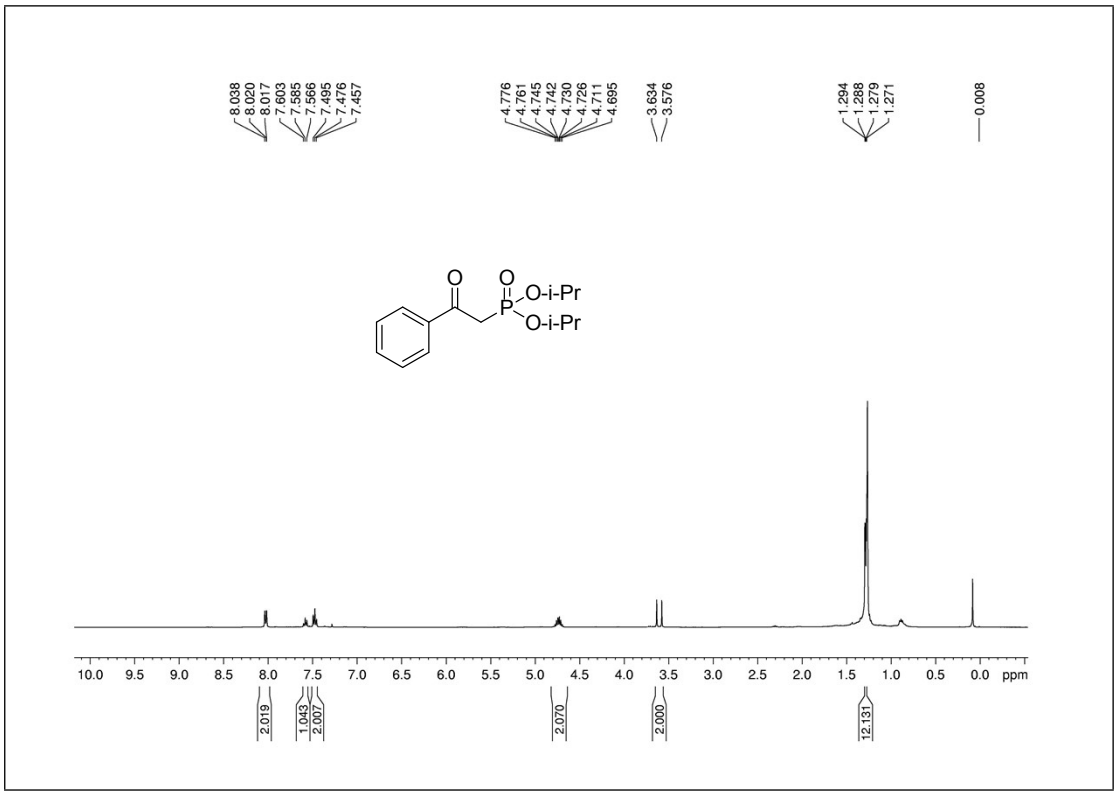

Fig. 5 ^{1}H NMR spectrum of compound **3b**

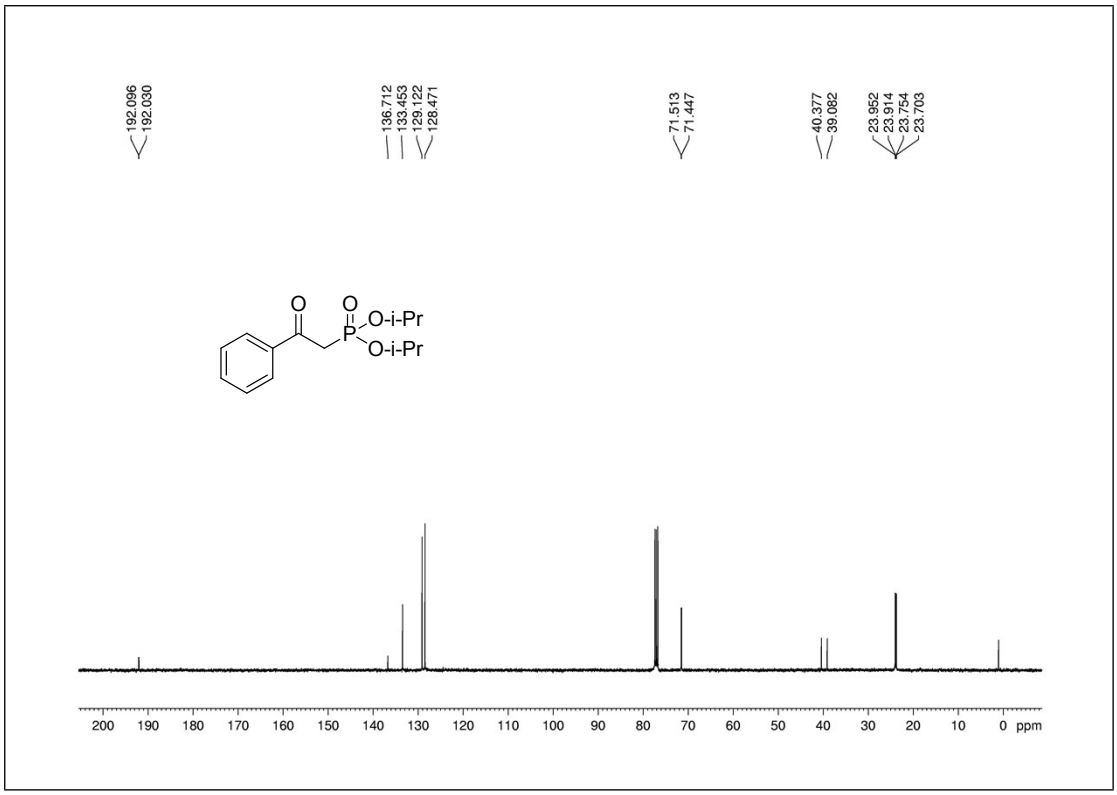

Fig. 6 ^{13}C NMR spectrum of compound **3b**

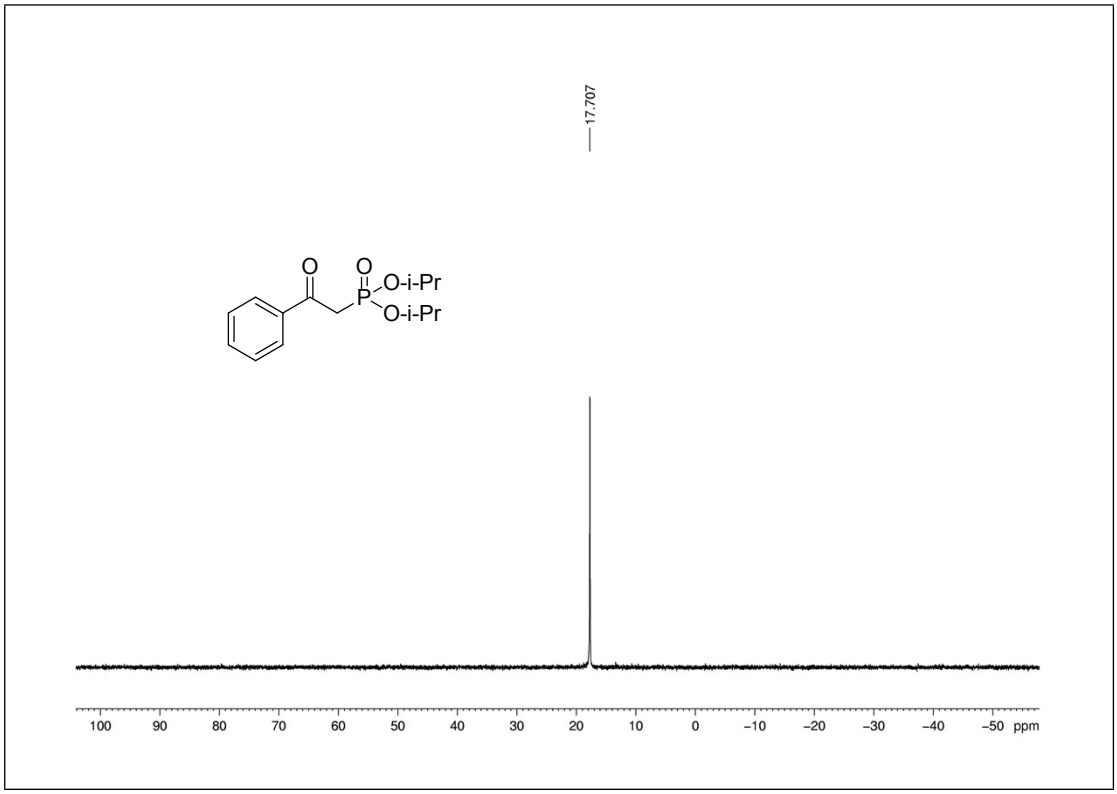

Fig. 7 ^{31}P NMR spectrum of compound **3b**

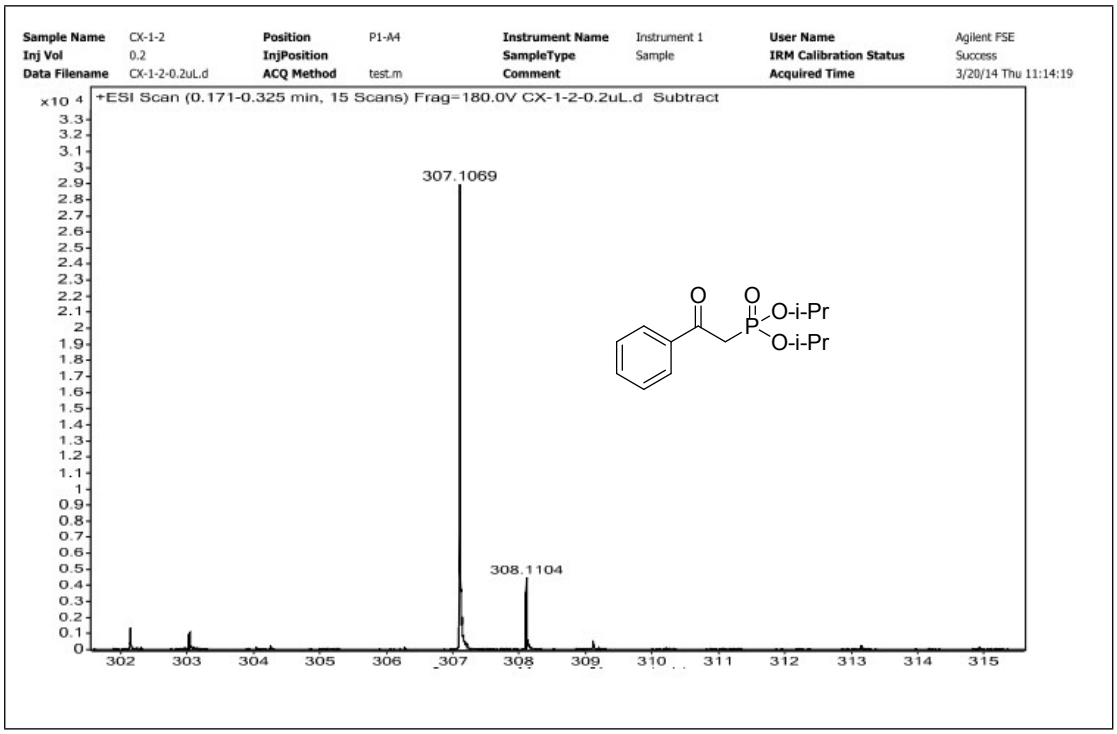

Fig. 8 HRMS spectrum of compound **3b**

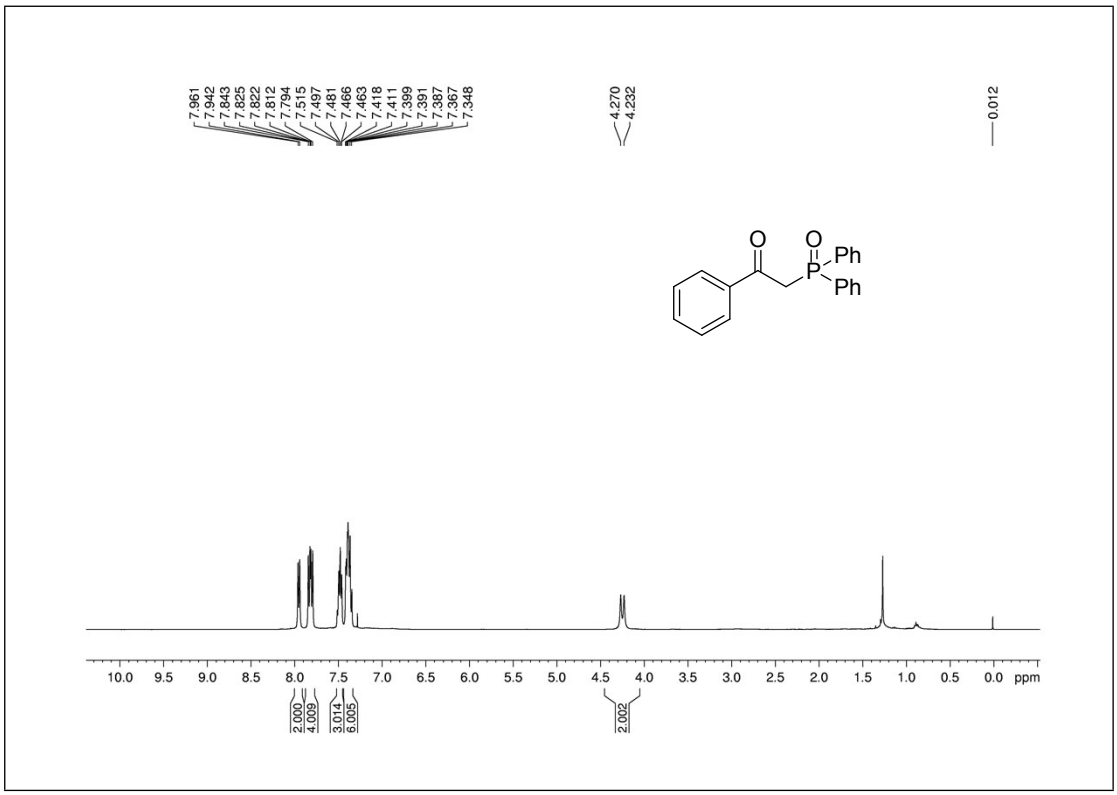

Fig. 9 ^{1}H NMR spectrum of compound **3c**

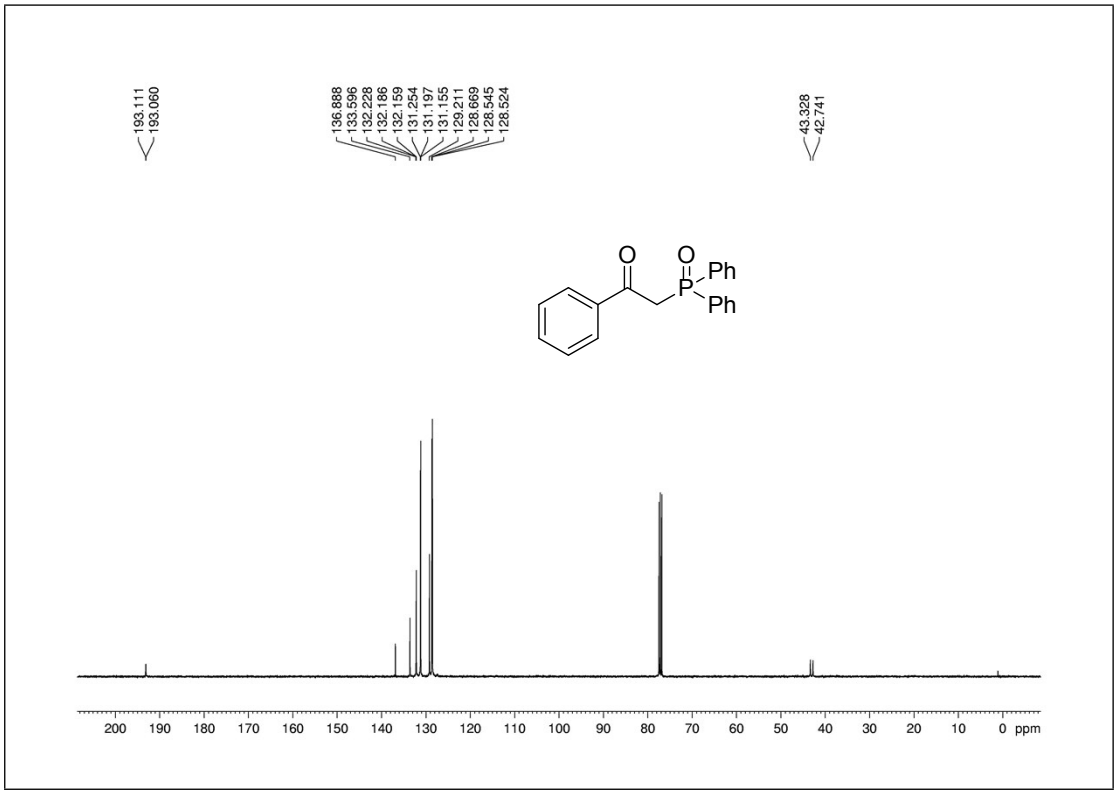

Fig. 10 ^{13}C NMR spectrum of compound **3c**

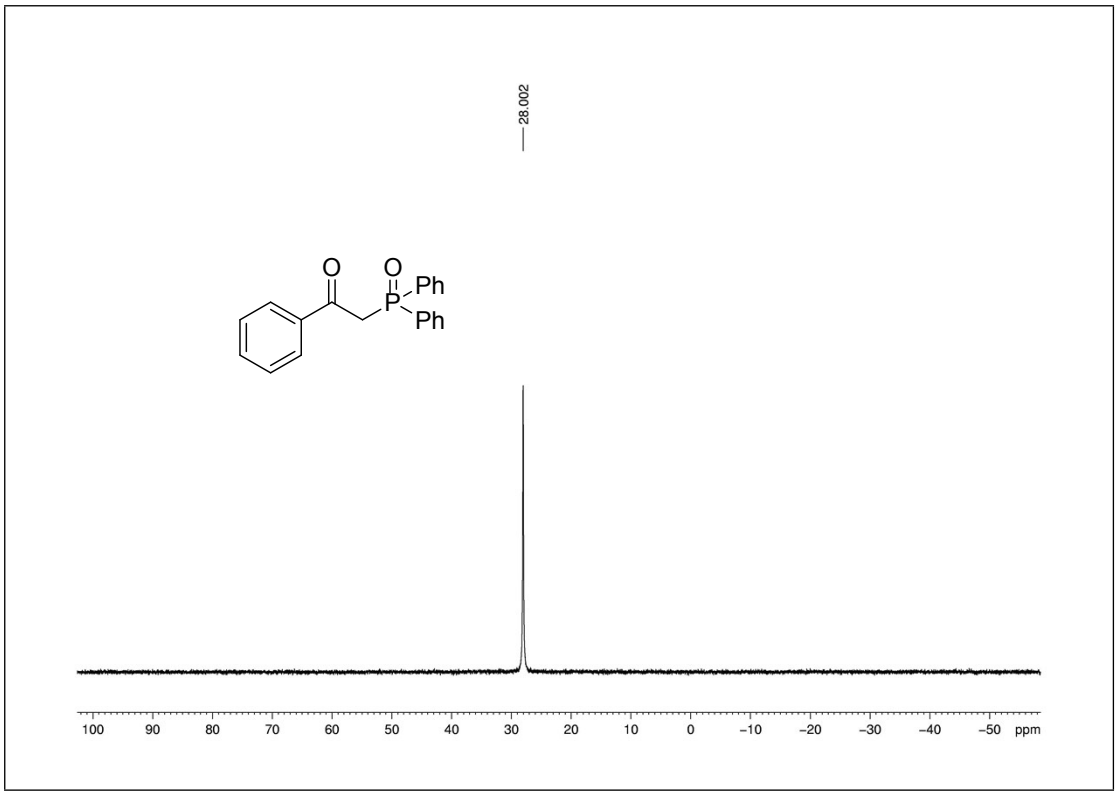

Fig. 11 ^{31}P NMR spectrum of compound **3c**

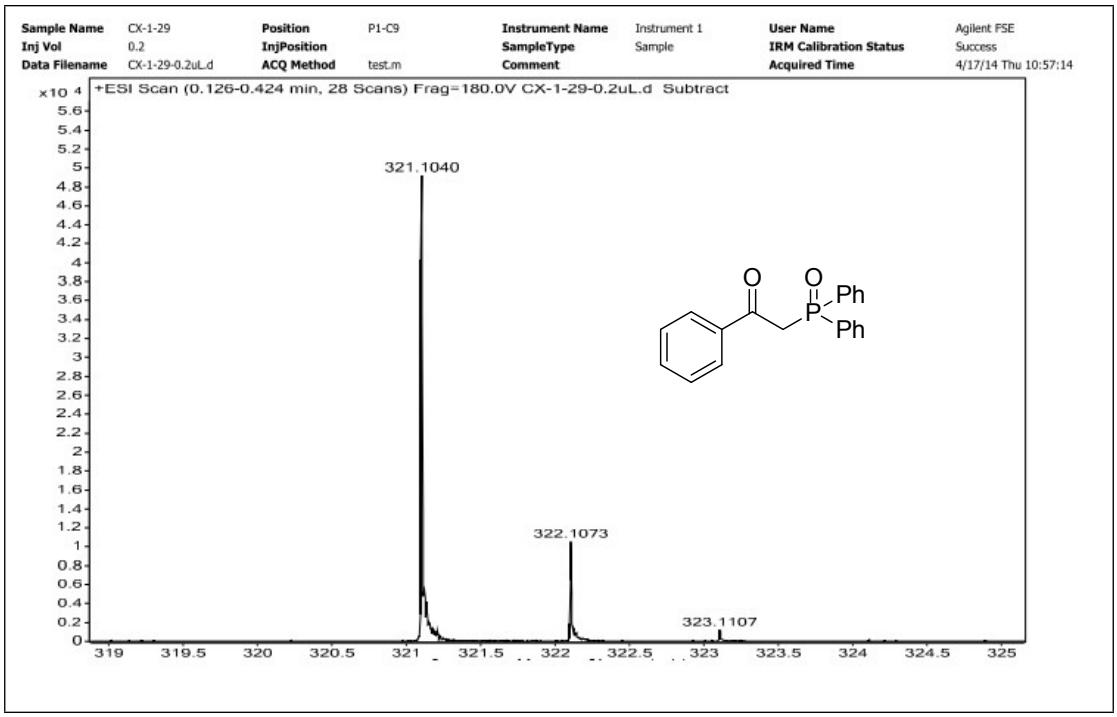

Fig. 12 HRMS spectrum of compound **3c**

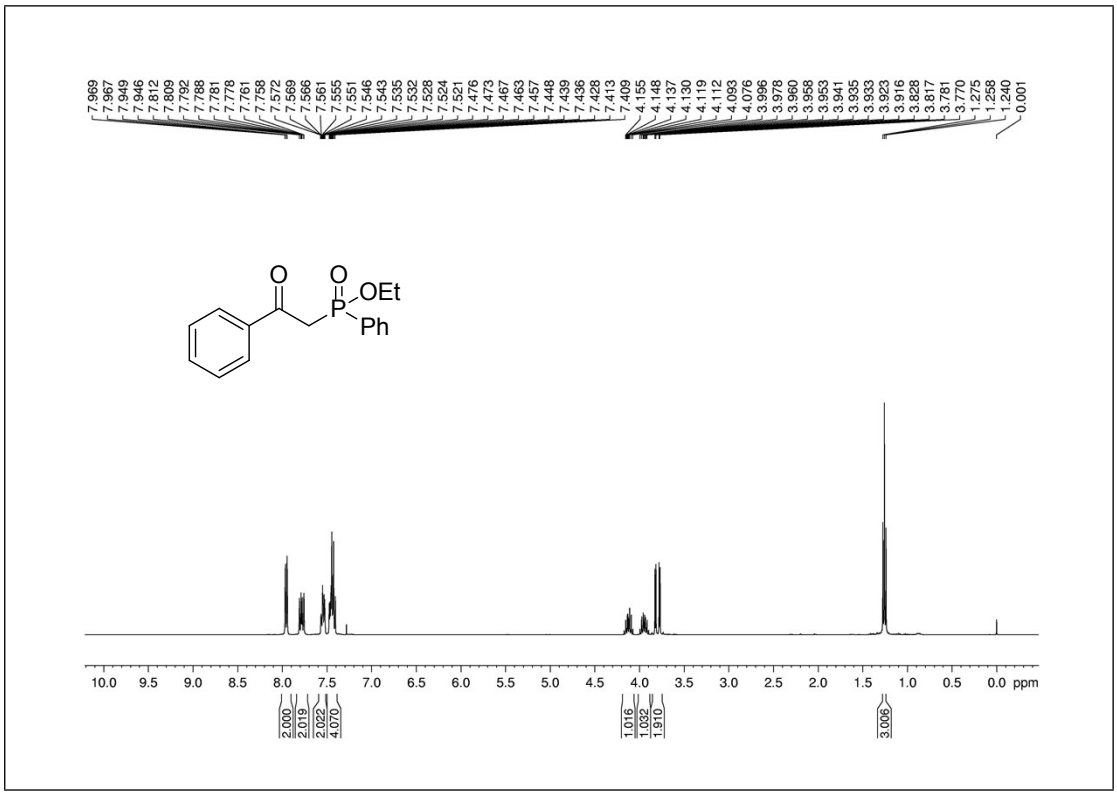

Fig. 13 ^{1}H NMR spectrum of compound **3d**

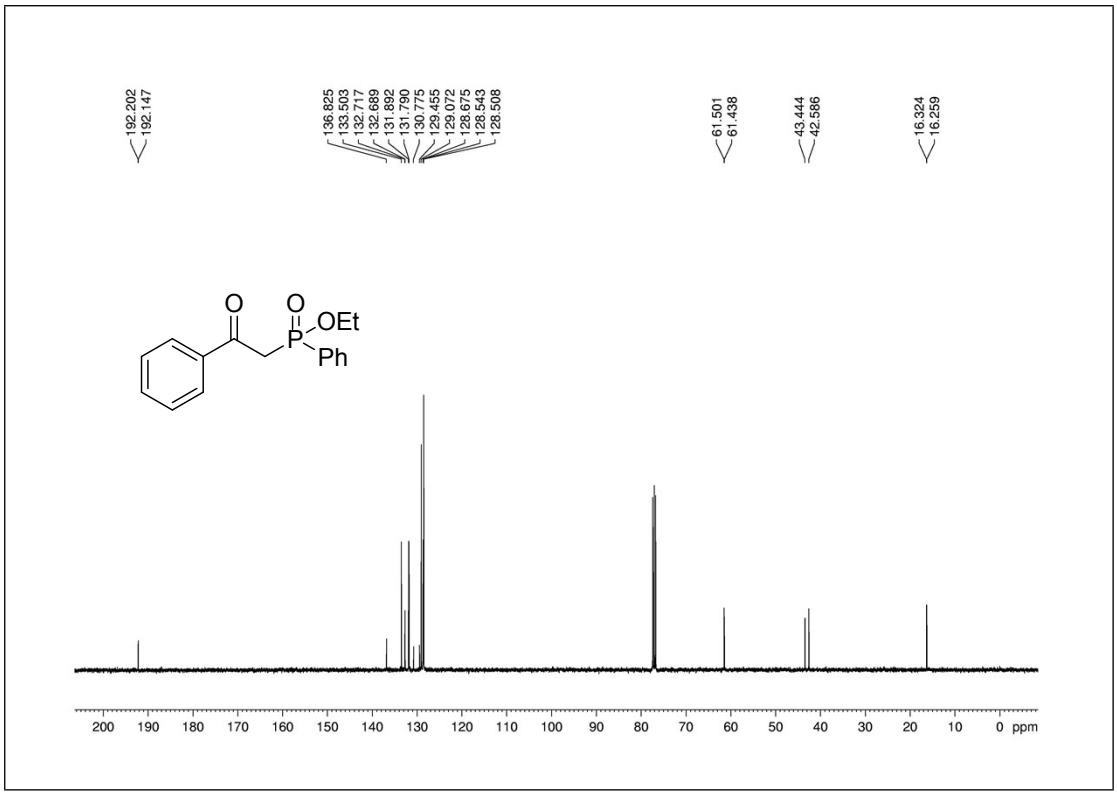

Fig. 14 ^{13}C NMR spectrum of compound **3d**

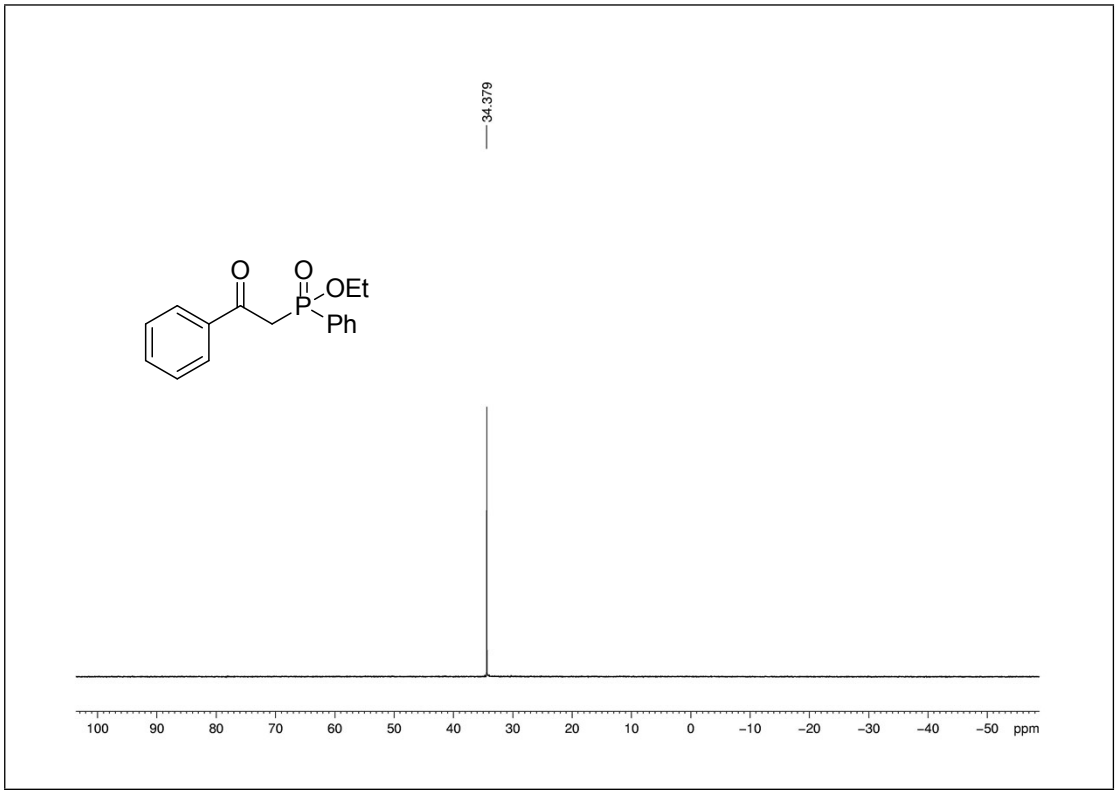

Fig. 15 ^{31}P NMR spectrum of compound **3d**

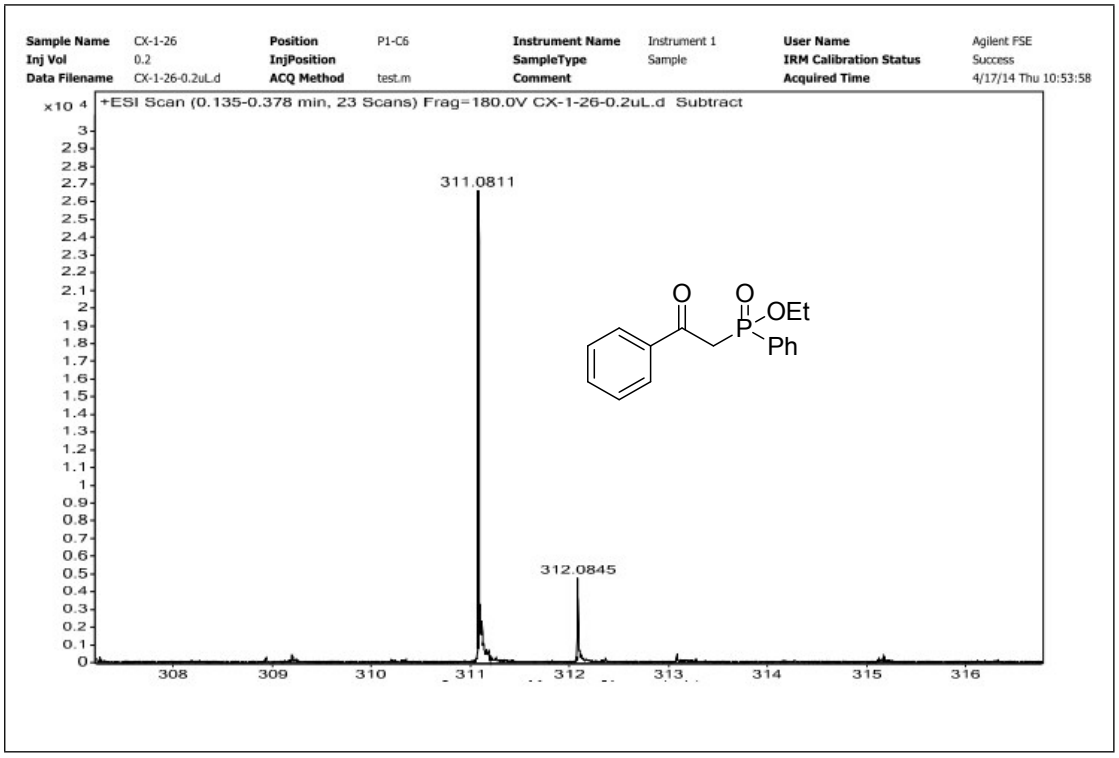

Fig. 16 HRMS spectrum of compound **3d**

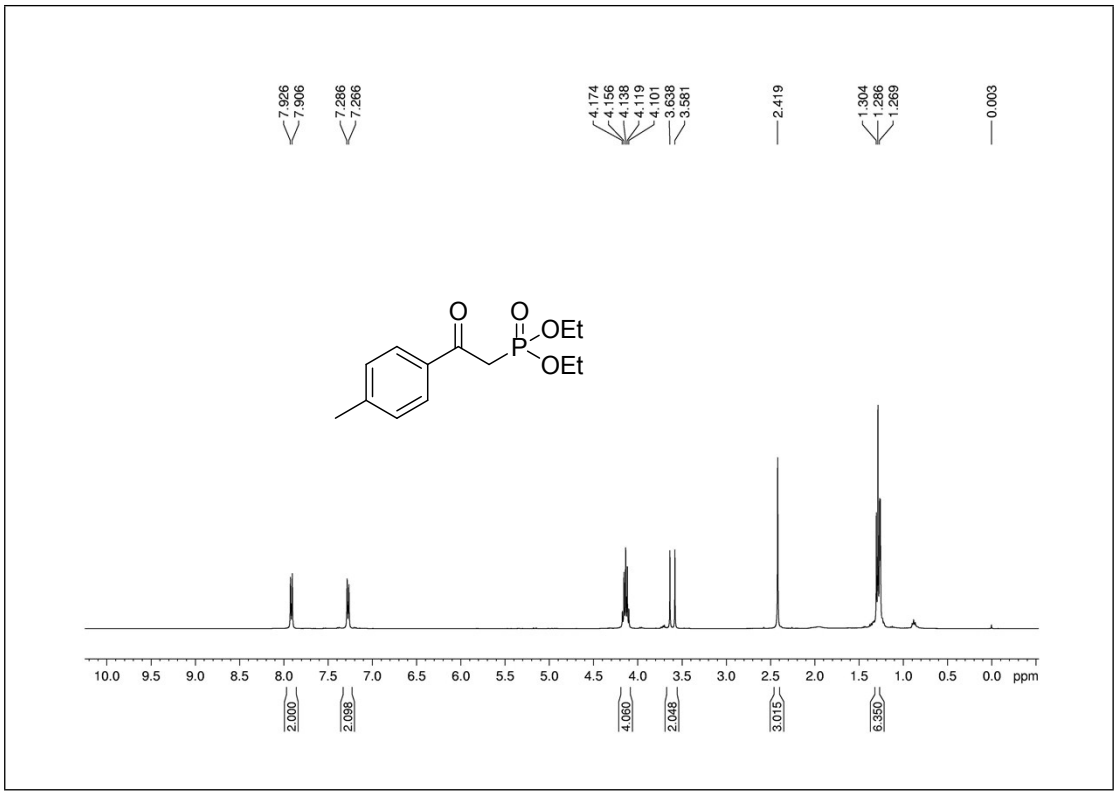

Fig. 17 ^{1}H NMR spectrum of compound **3e**

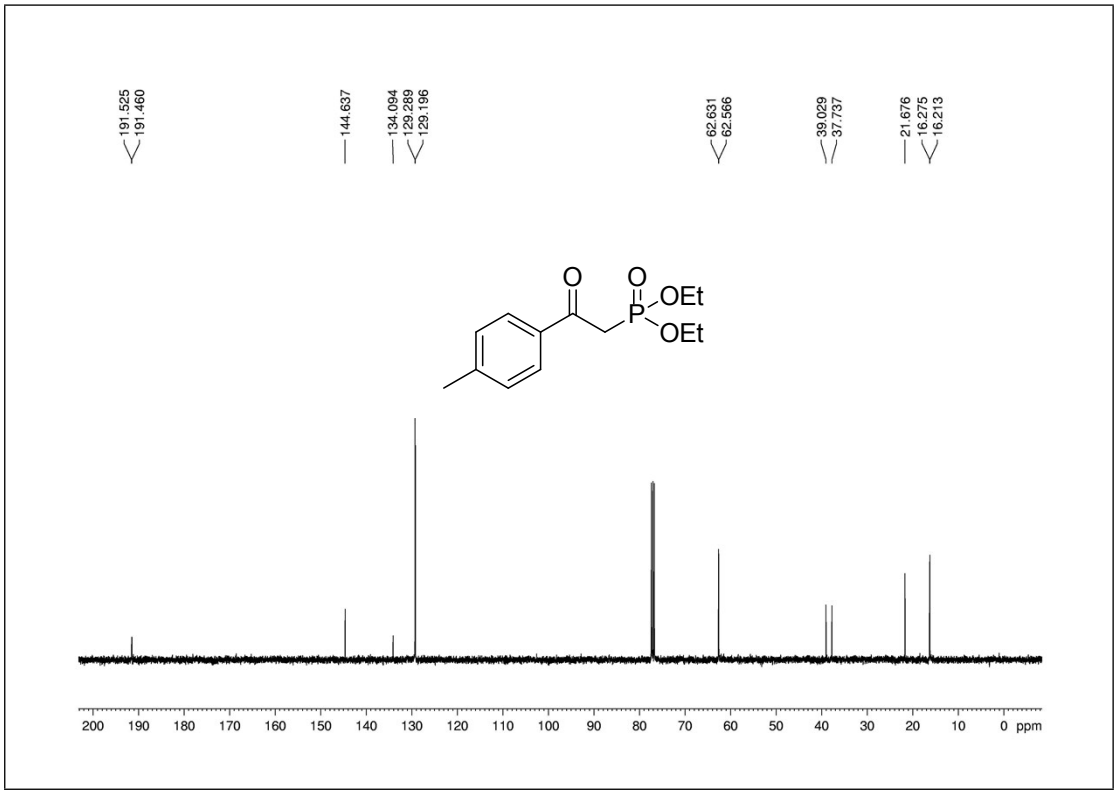

Fig. 18 ^{13}C NMR spectrum of compound **3e**

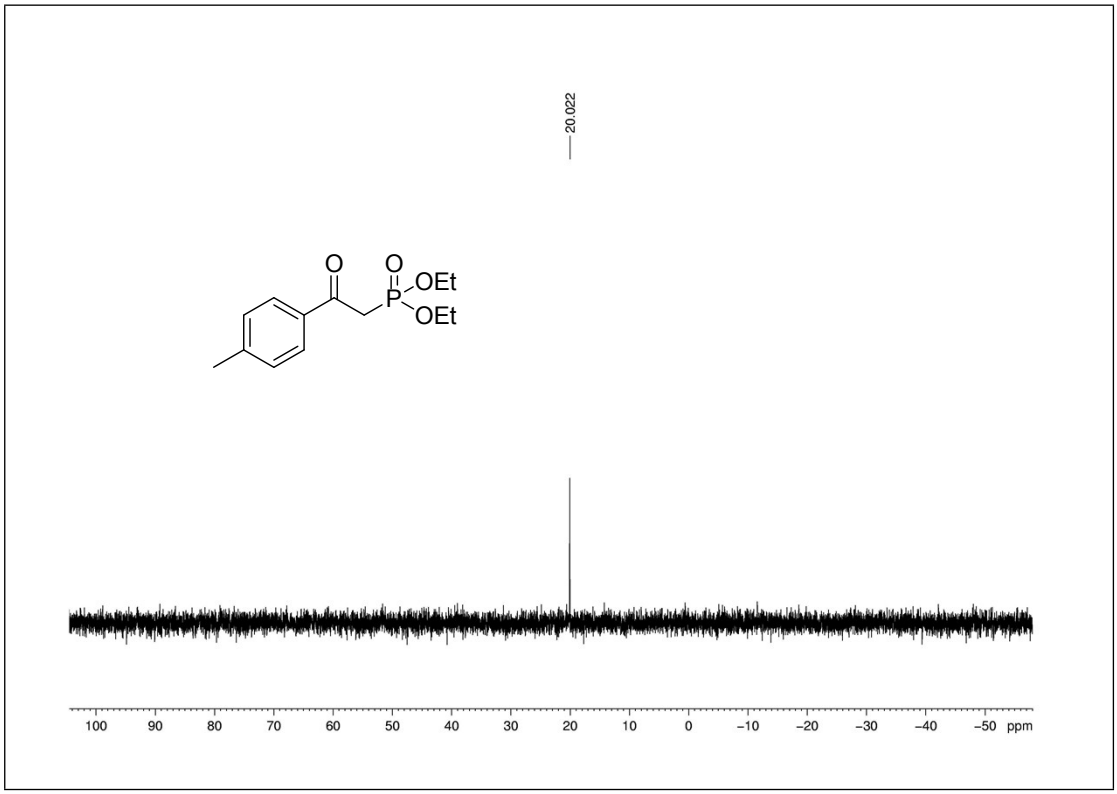

Fig. 19 ^{31}P NMR spectrum of compound **3e**

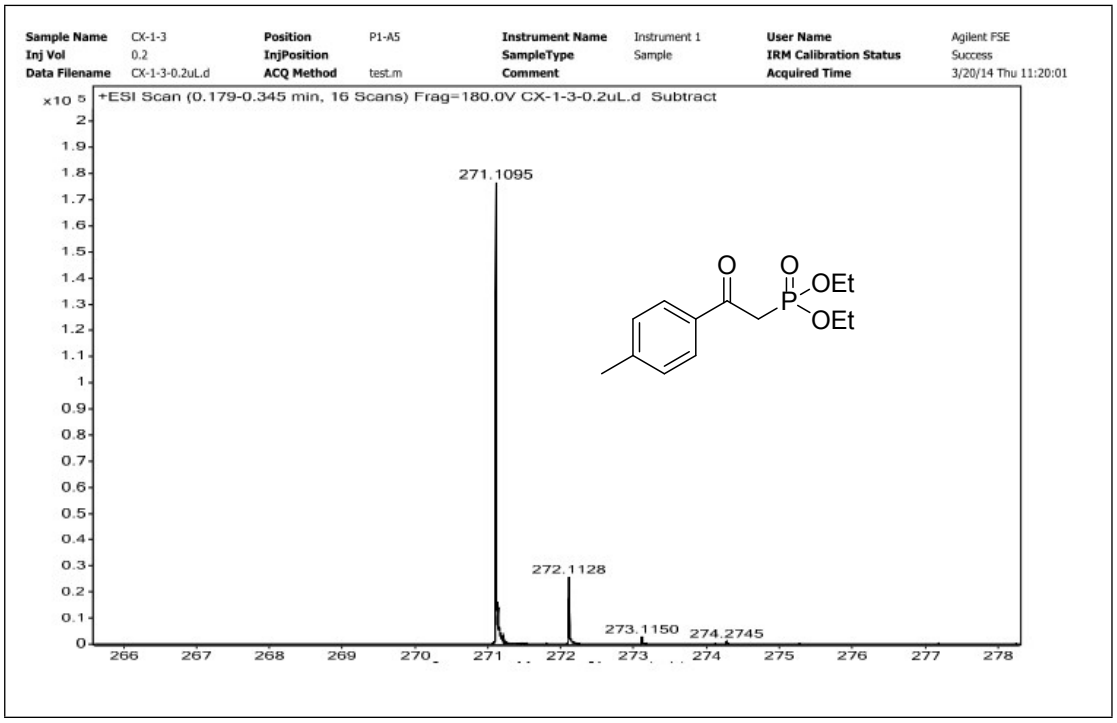

Fig. 20 HRMS spectrum of compound **3e**

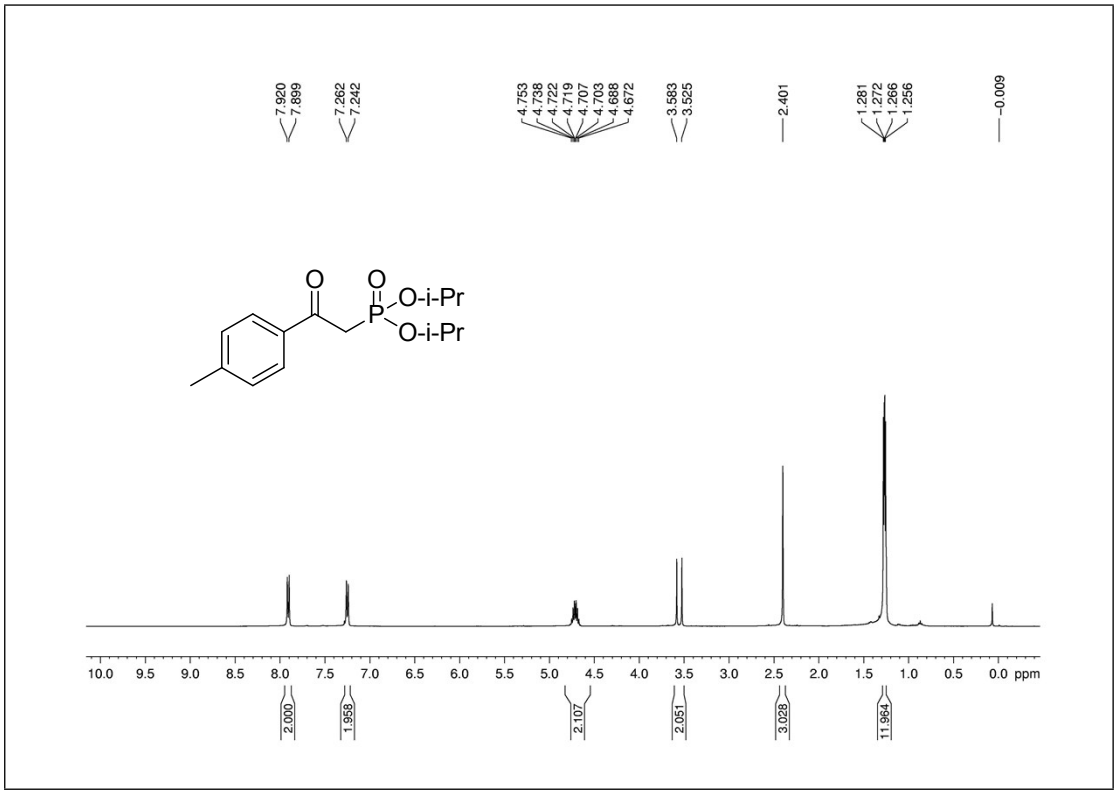

Fig. 21 ^{1}H NMR spectrum of compound **3f**

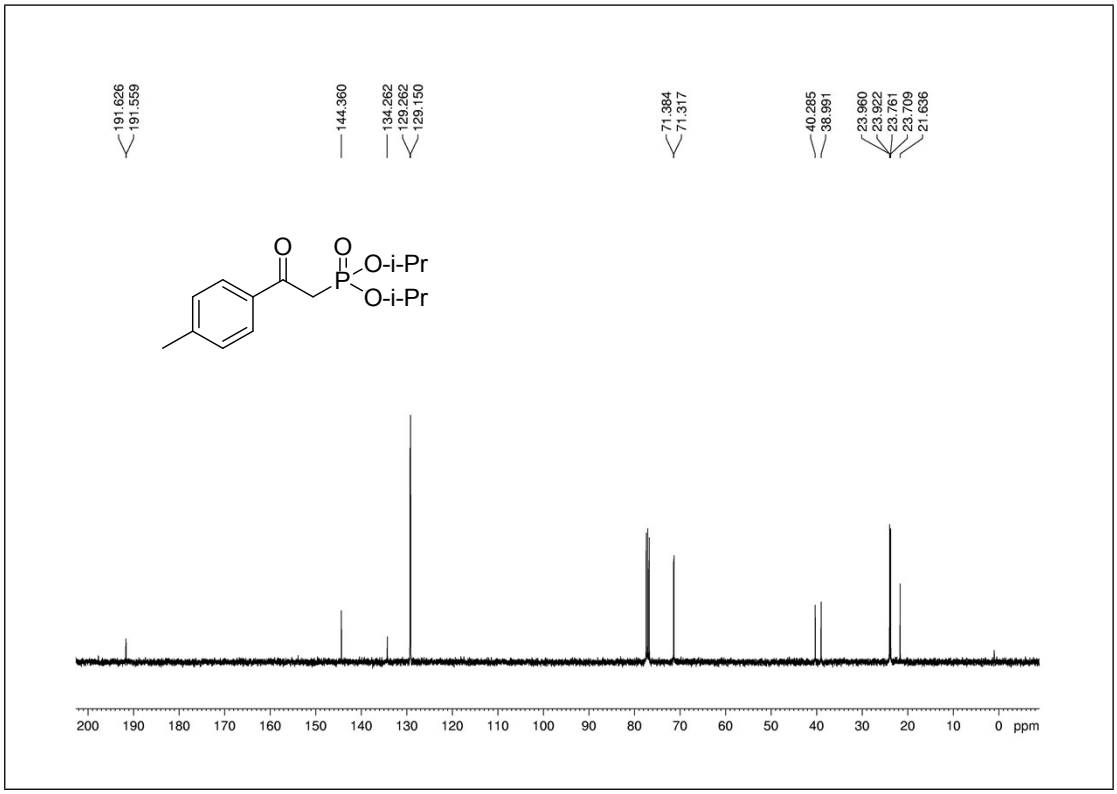

Fig. 22 ^{13}C NMR spectrum of compound **3f**

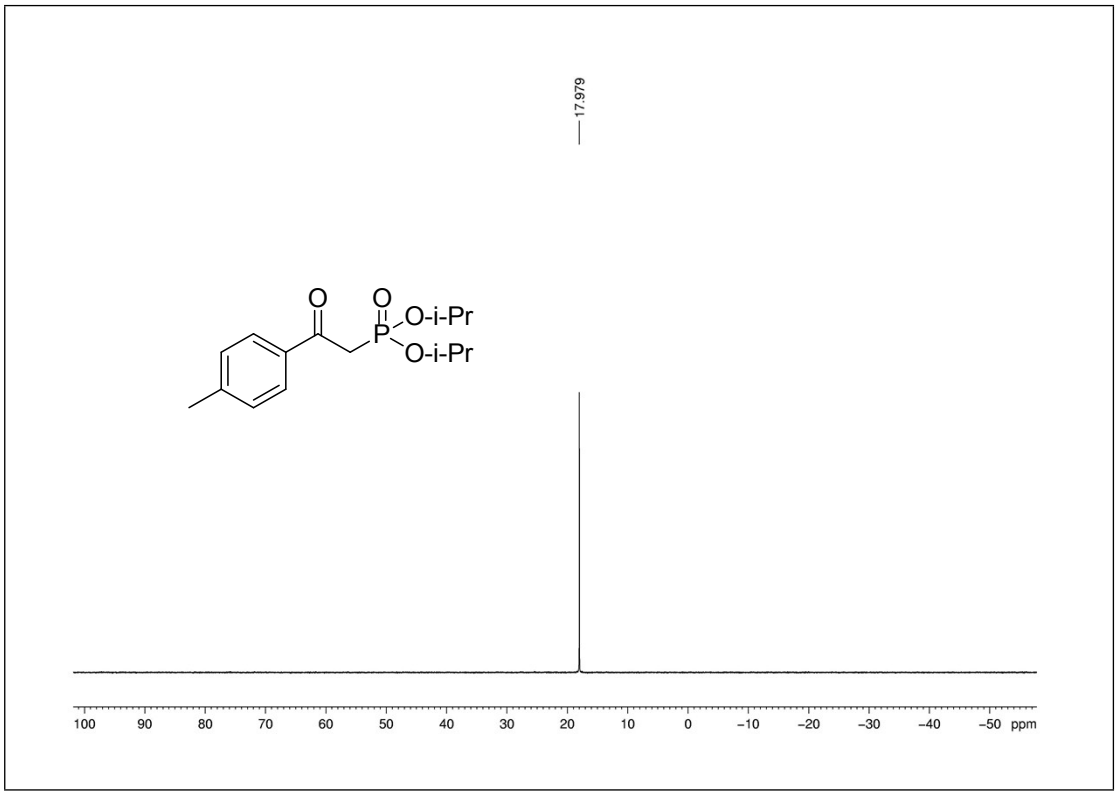

Fig. 23 ^{31}P NMR spectrum of compound **3f**

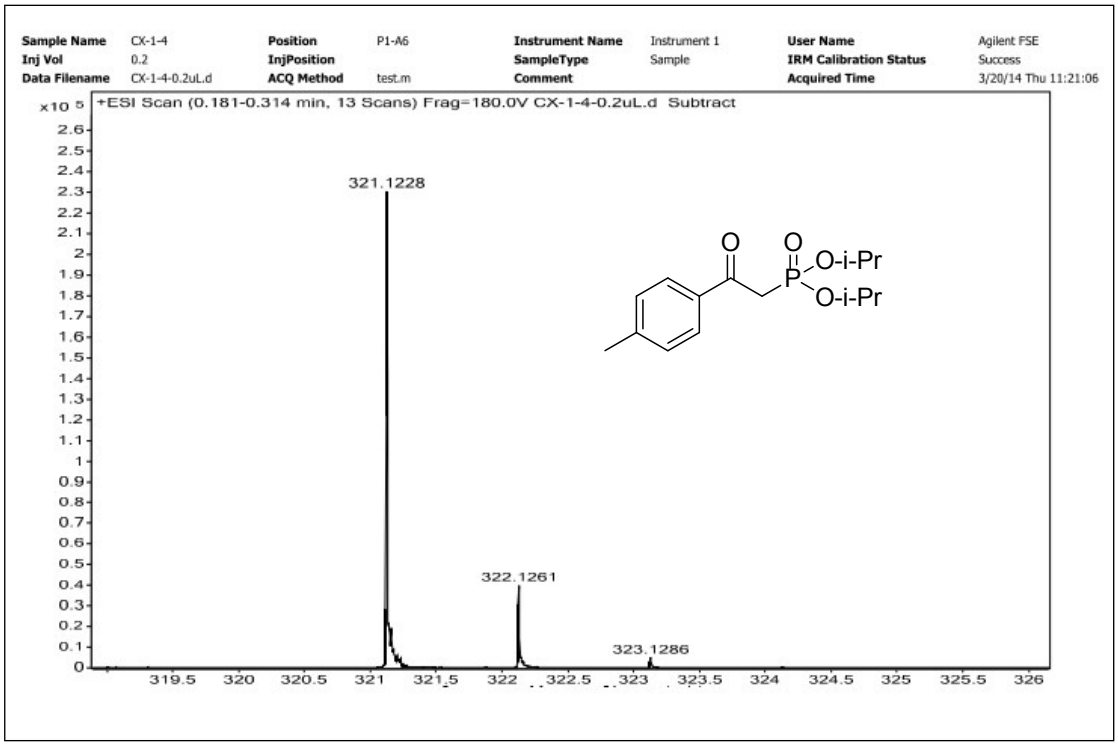

Fig. 24 HRMS spectrum of compound **3f**

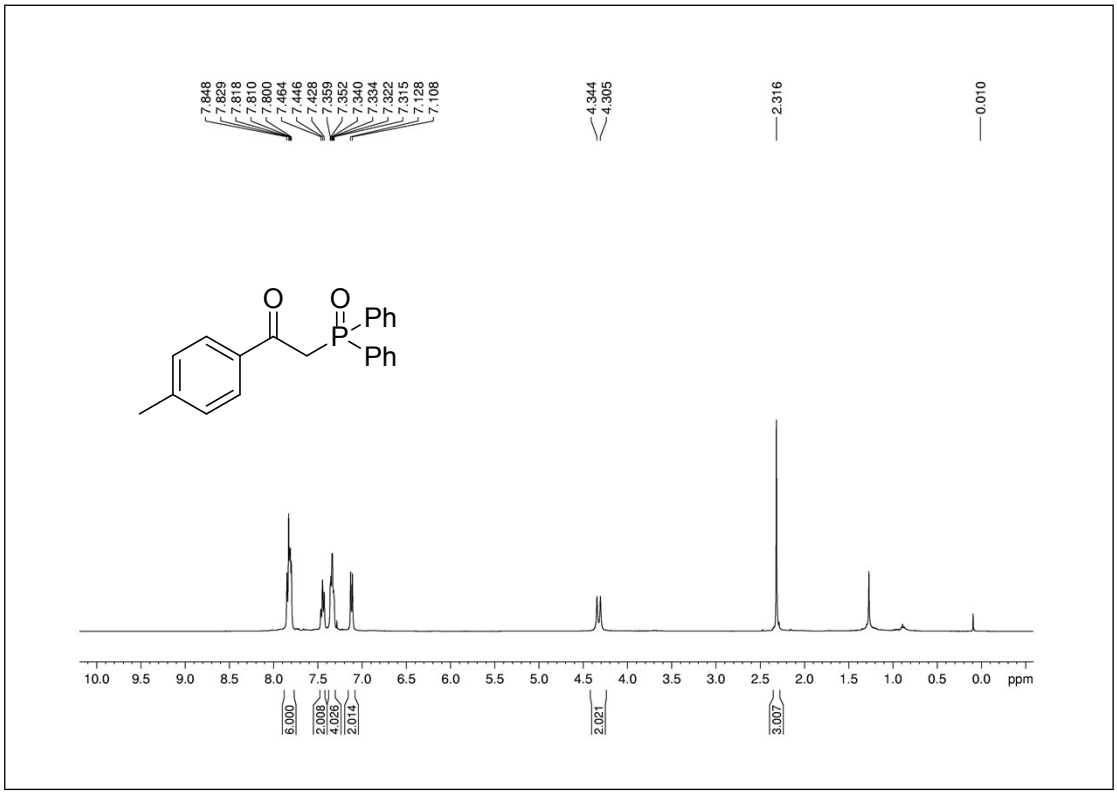

Fig. 25 ^{1}H NMR spectrum of compound **3g**

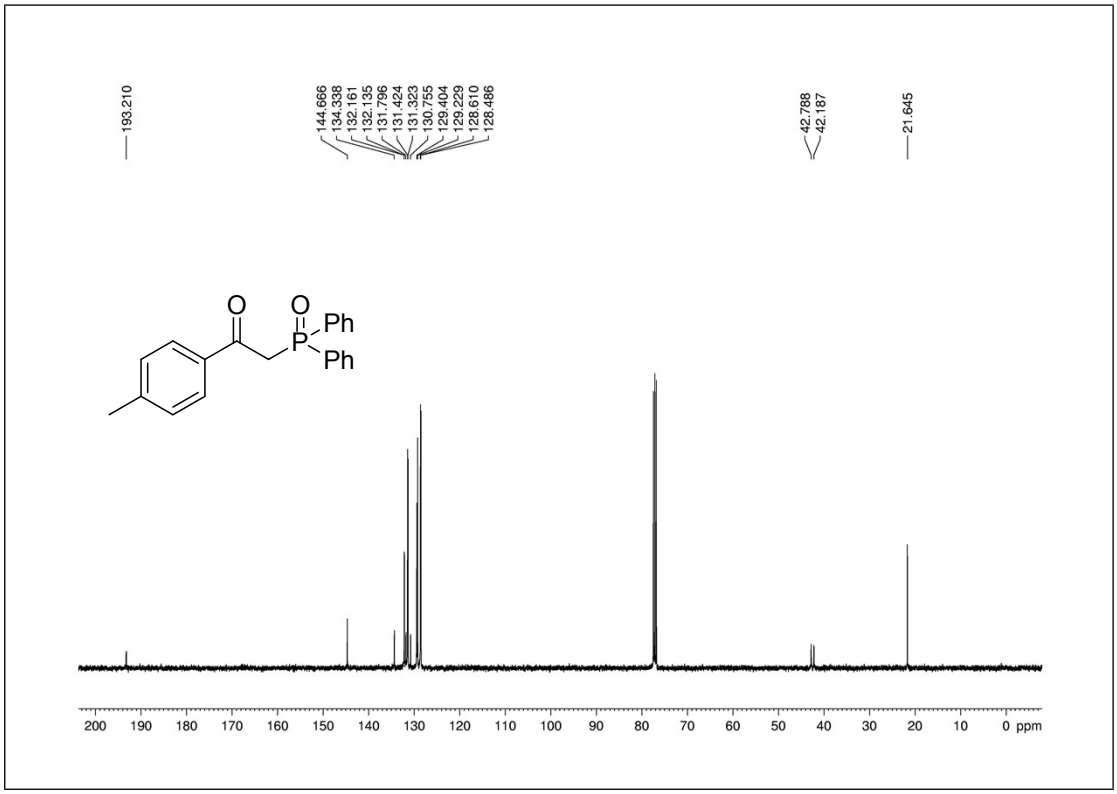

Fig. 26 ^{13}C NMR spectrum of compound **3g**

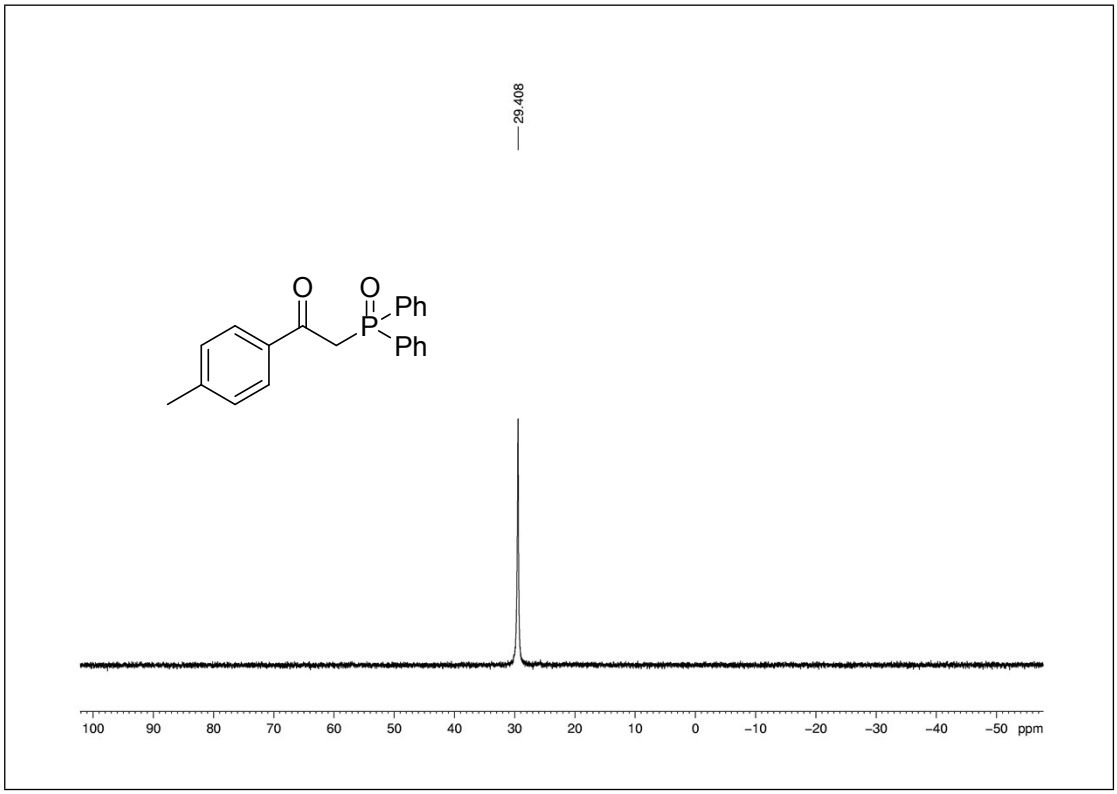

Fig. 27 ^{31}P NMR spectrum of compound **3g**

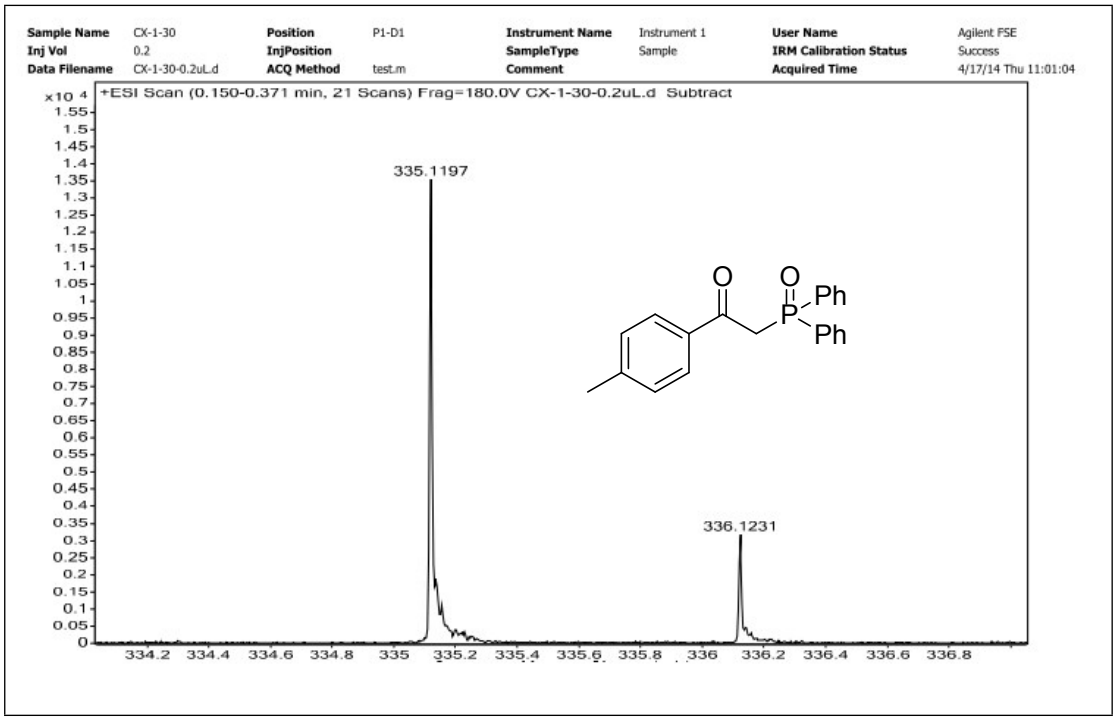

Fig. 28 HRMS spectrum of compound **3g**

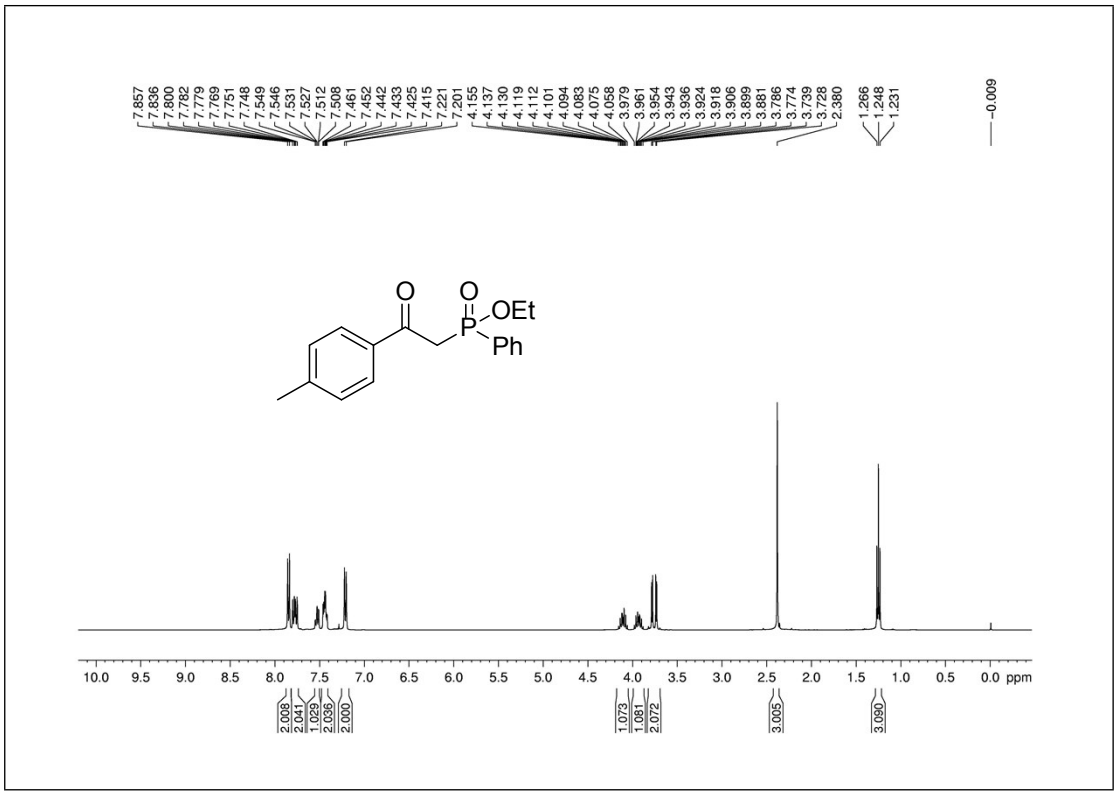

Fig. 29 ^{1}H NMR spectrum of compound **3h**

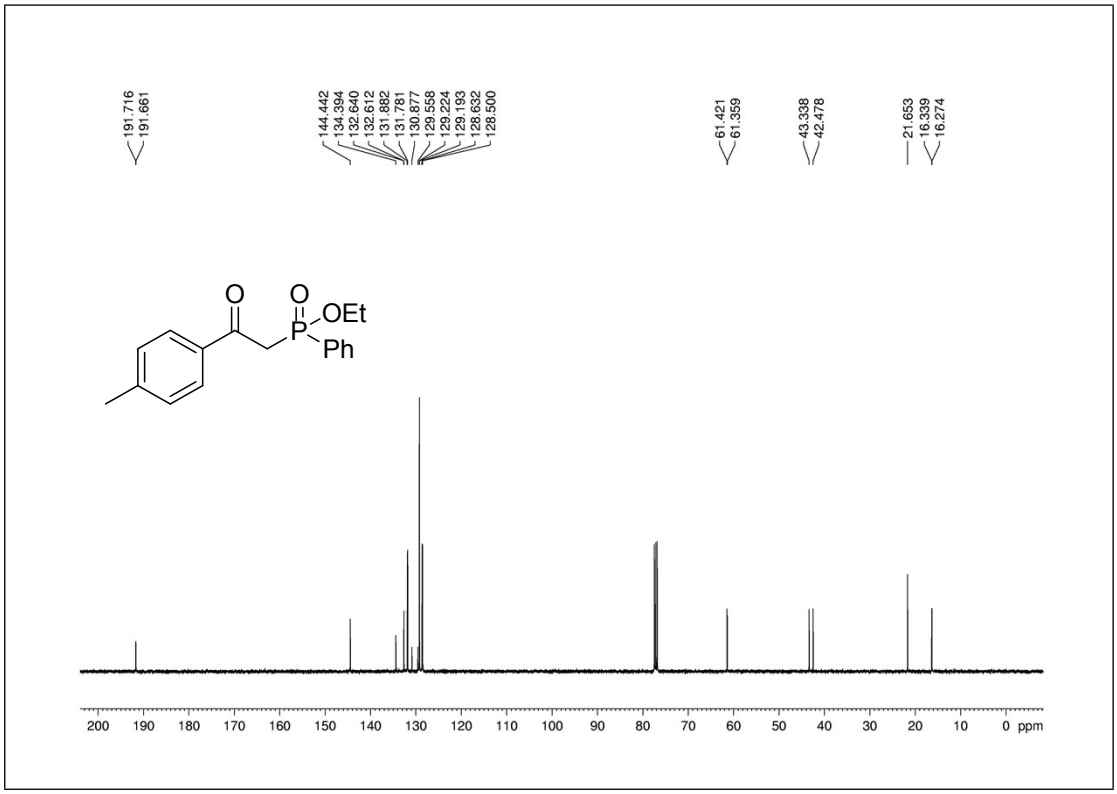

Fig. 30 ^{13}C NMR spectrum of compound **3h**

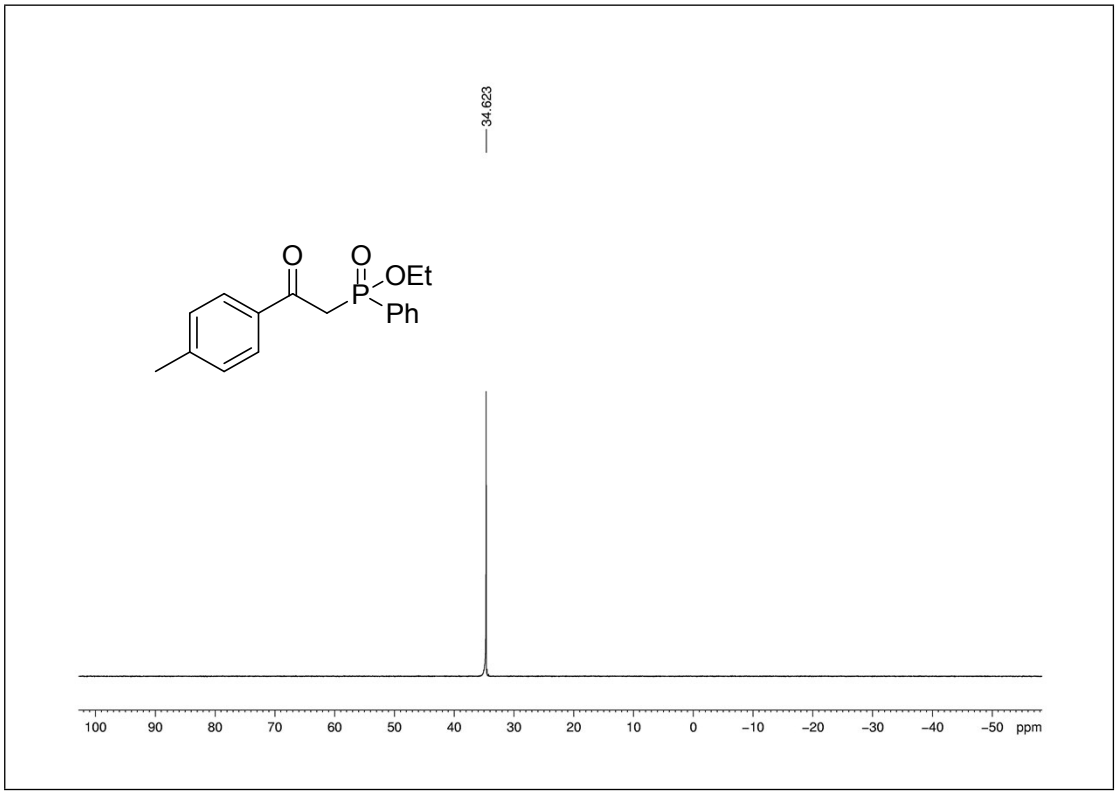

Fig. 31 ^{31}P NMR spectrum of compound **3h**

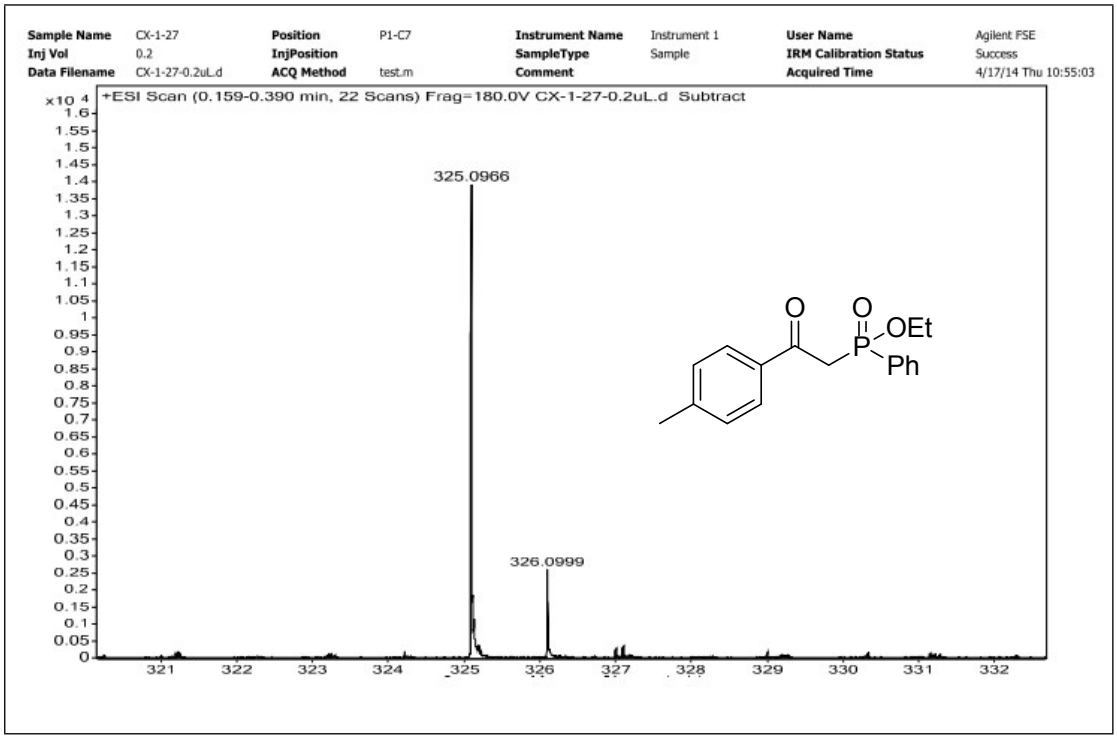

Fig. 32 HRMS spectrum of compound **3h**

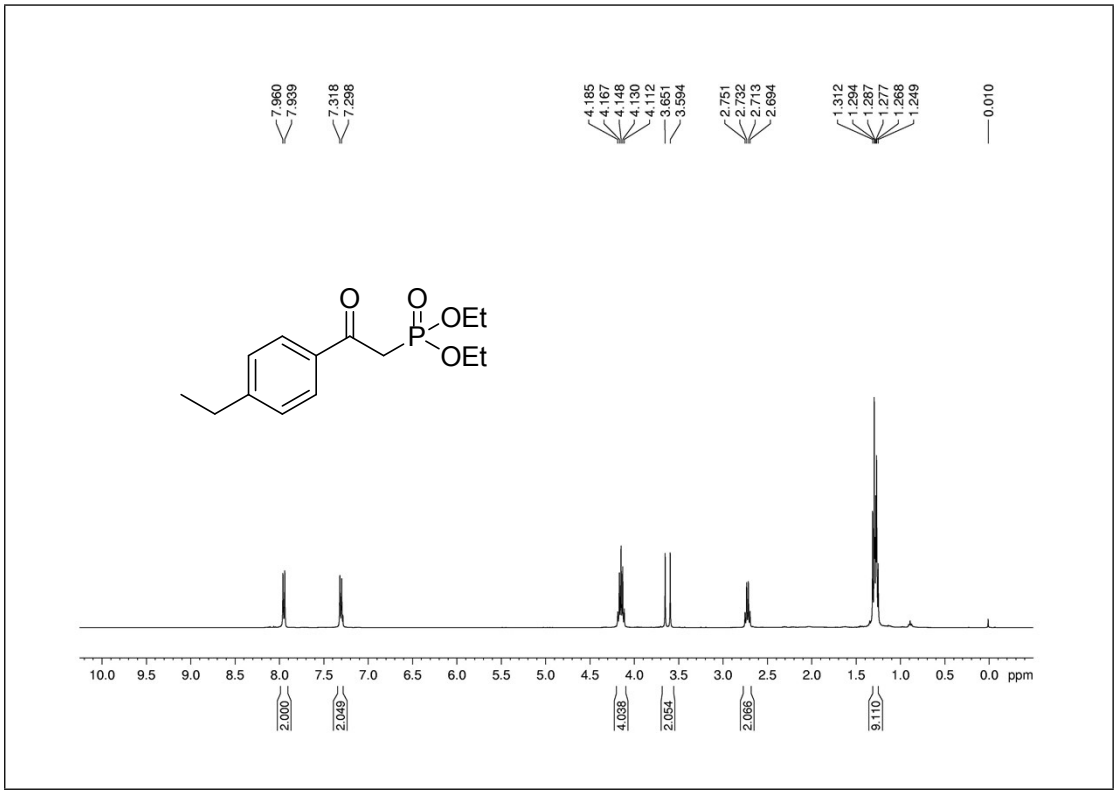

Fig. 33 [1]H NMR spectrum of compound **3i**

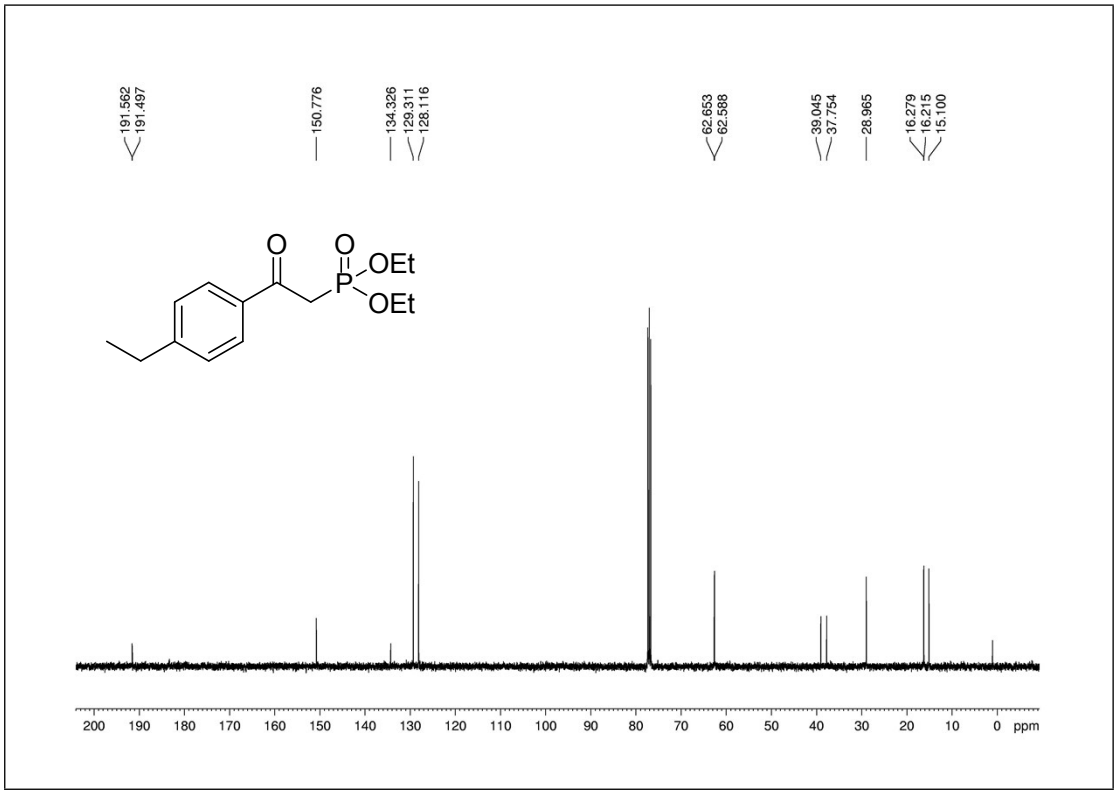

Fig. 34 ^{13}C NMR spectrum of compound **3i**

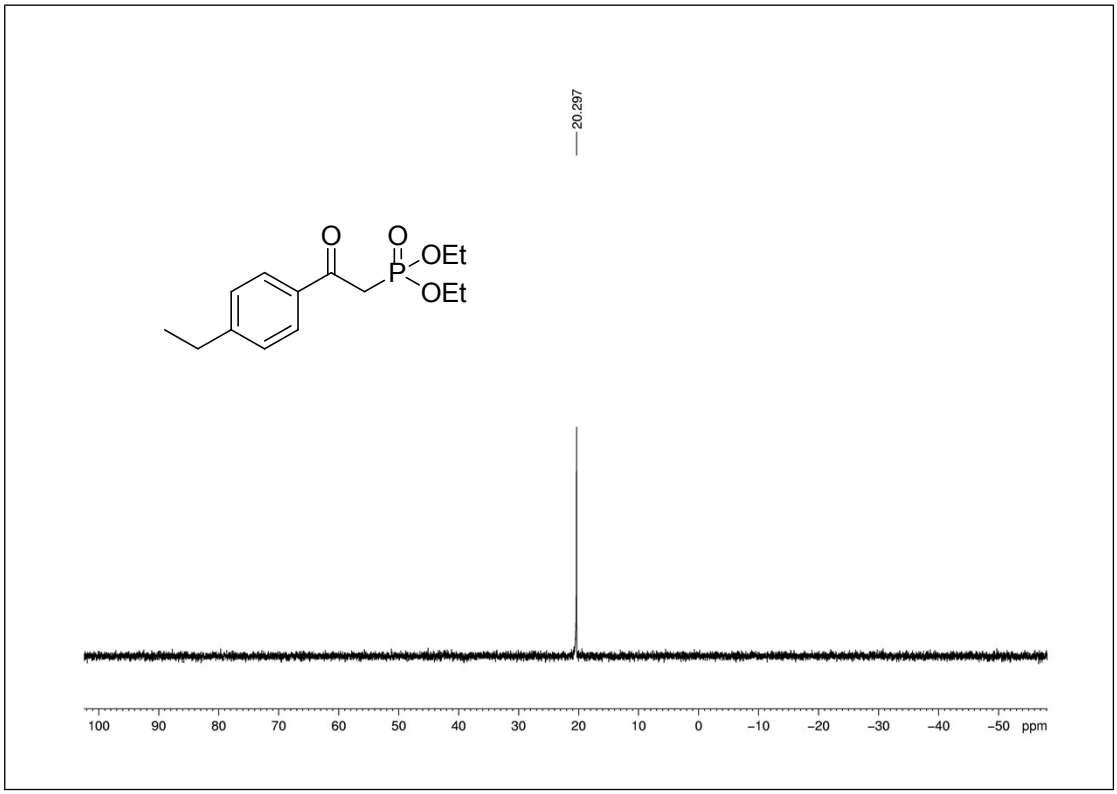

Fig. 35 ^{31}P NMR spectrum of compound **3i**

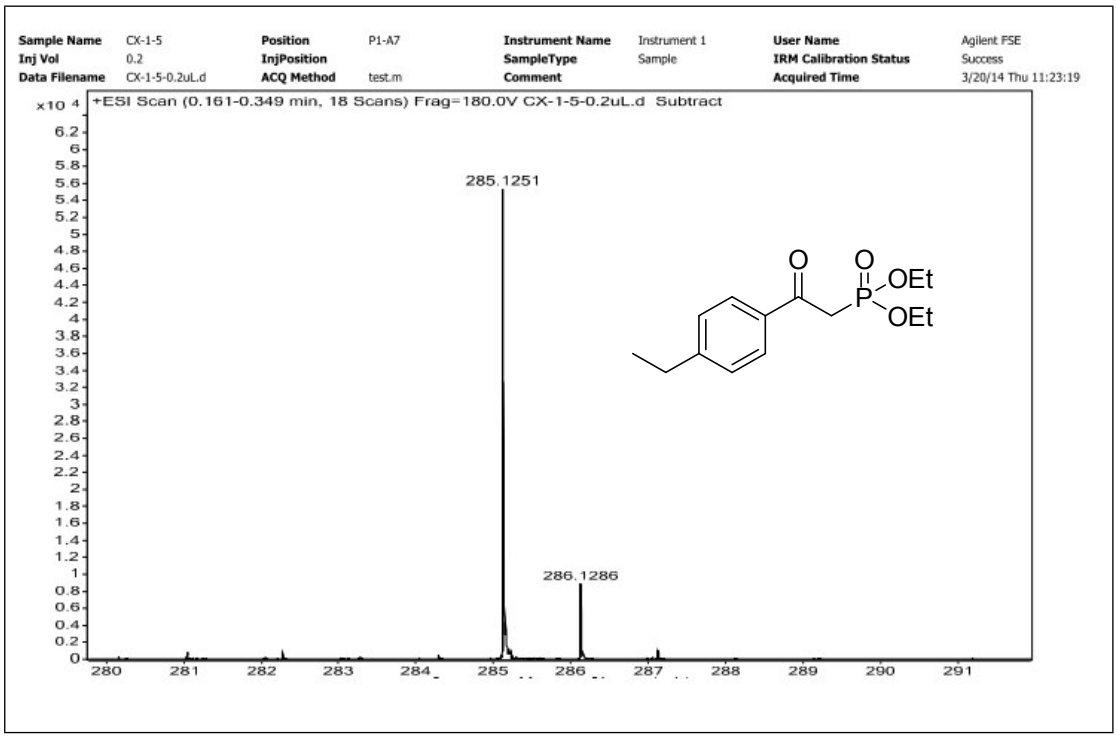

Fig. 36 HRMS spectrum of compound **3i**

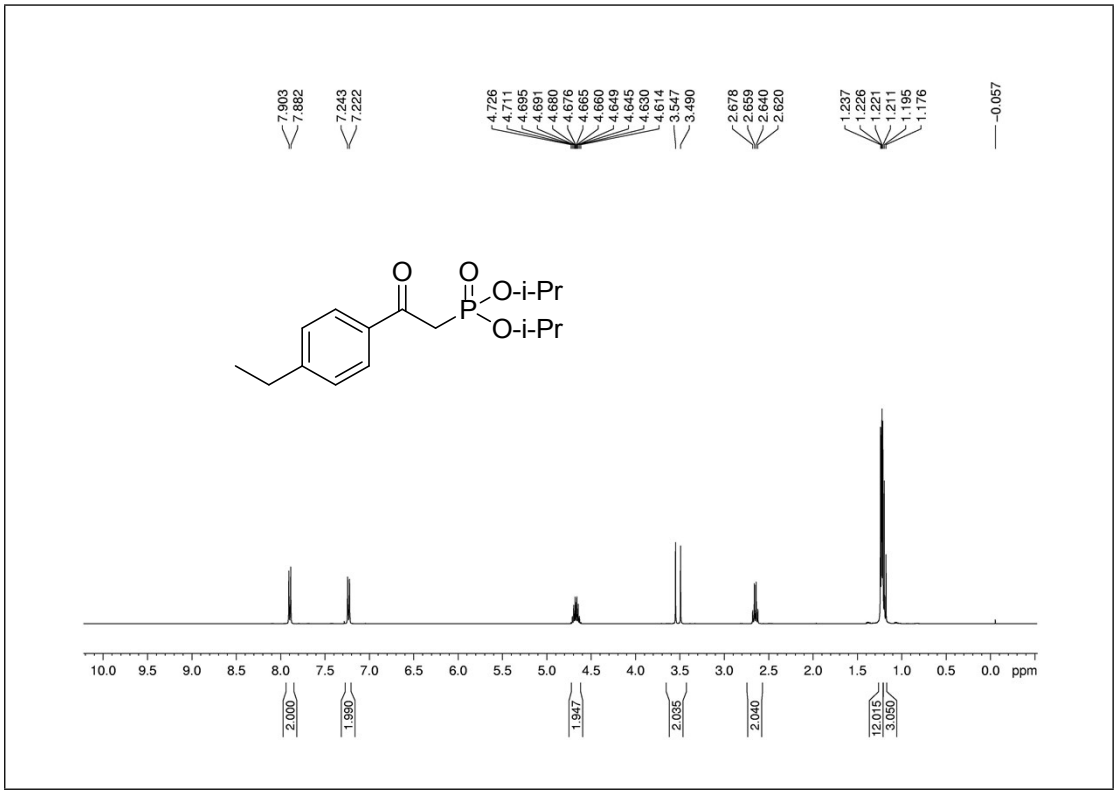

Fig. 37 ^{1}H NMR spectrum of compound **3j**

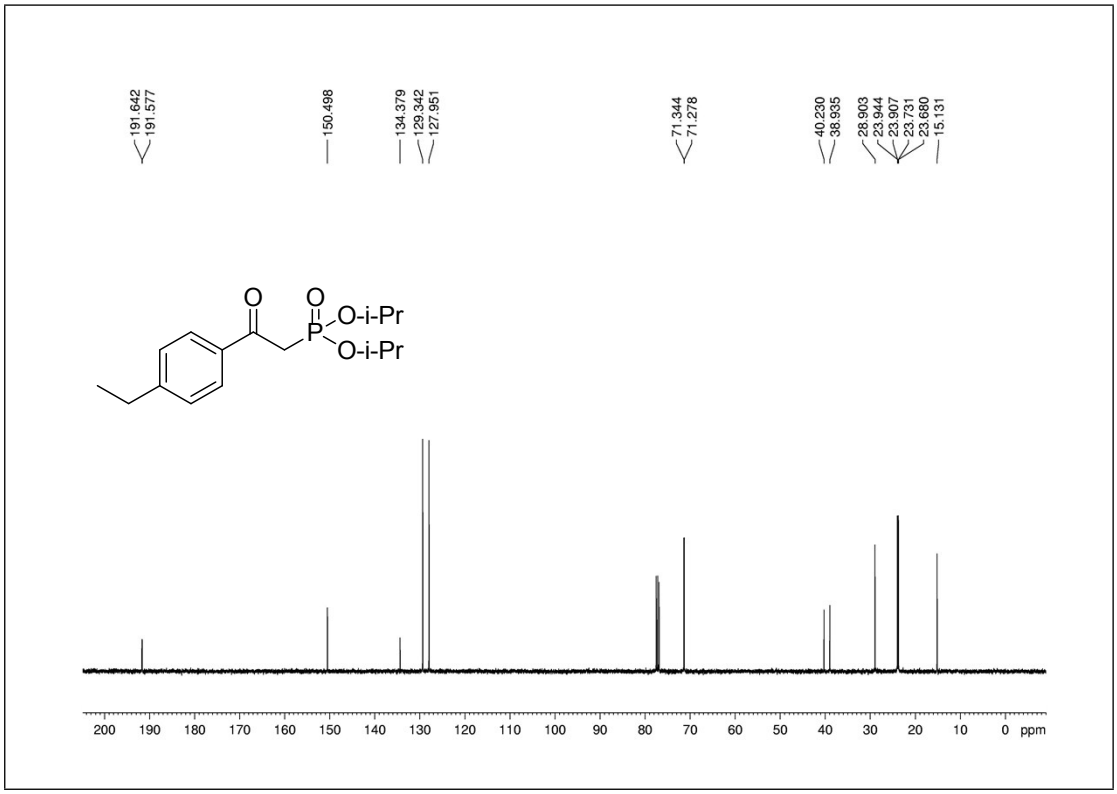

Fig. 38 ^{13}C NMR spectrum of compound **3j**

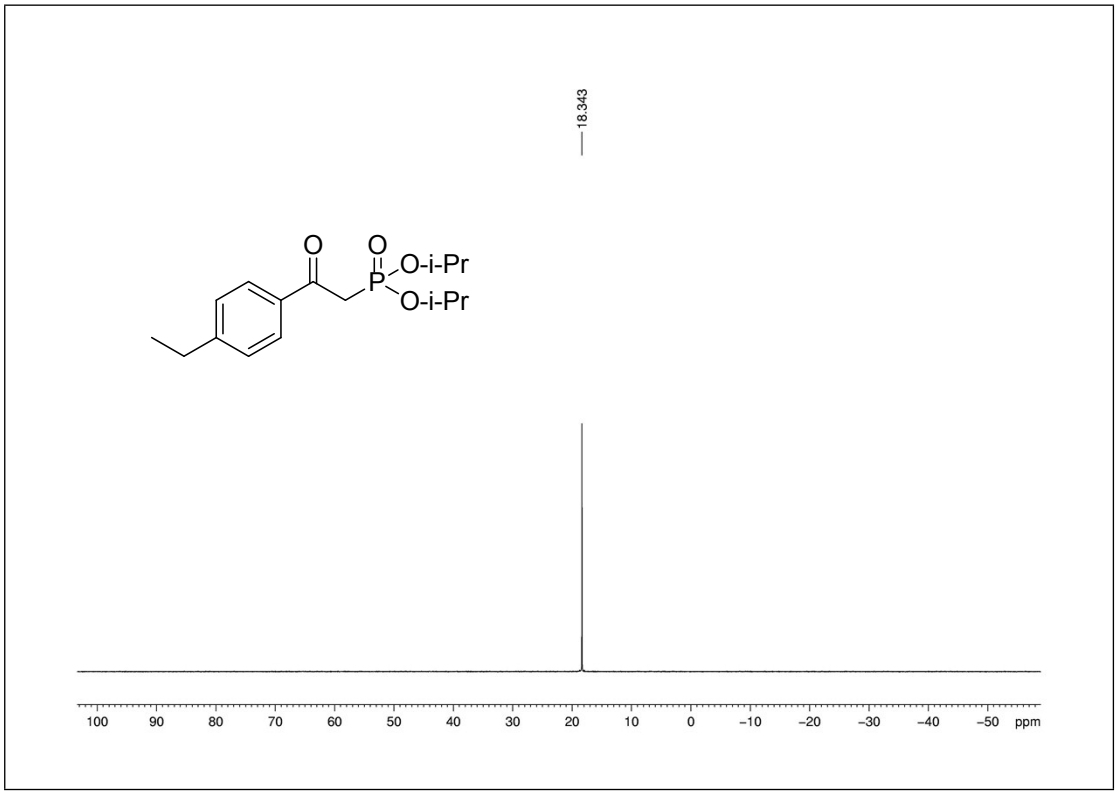

Fig. 39 ^{31}P NMR spectrum of compound **3j**

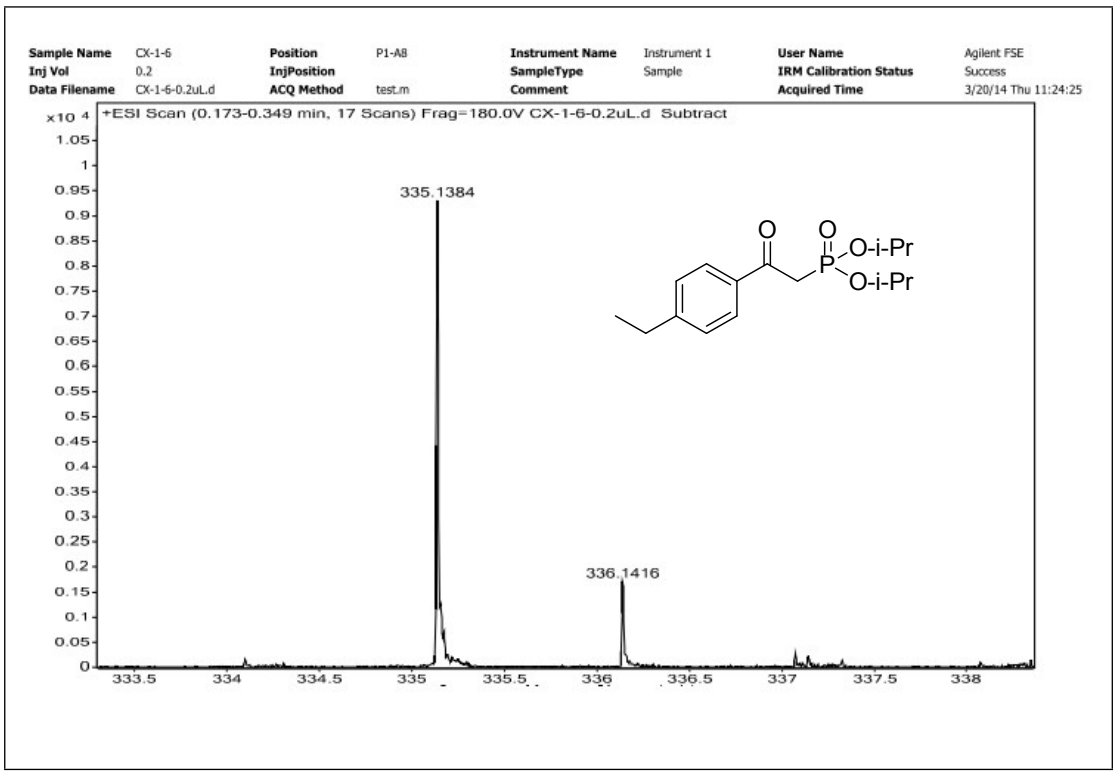

Fig. 40 HRMS spectrum of compound **3j**

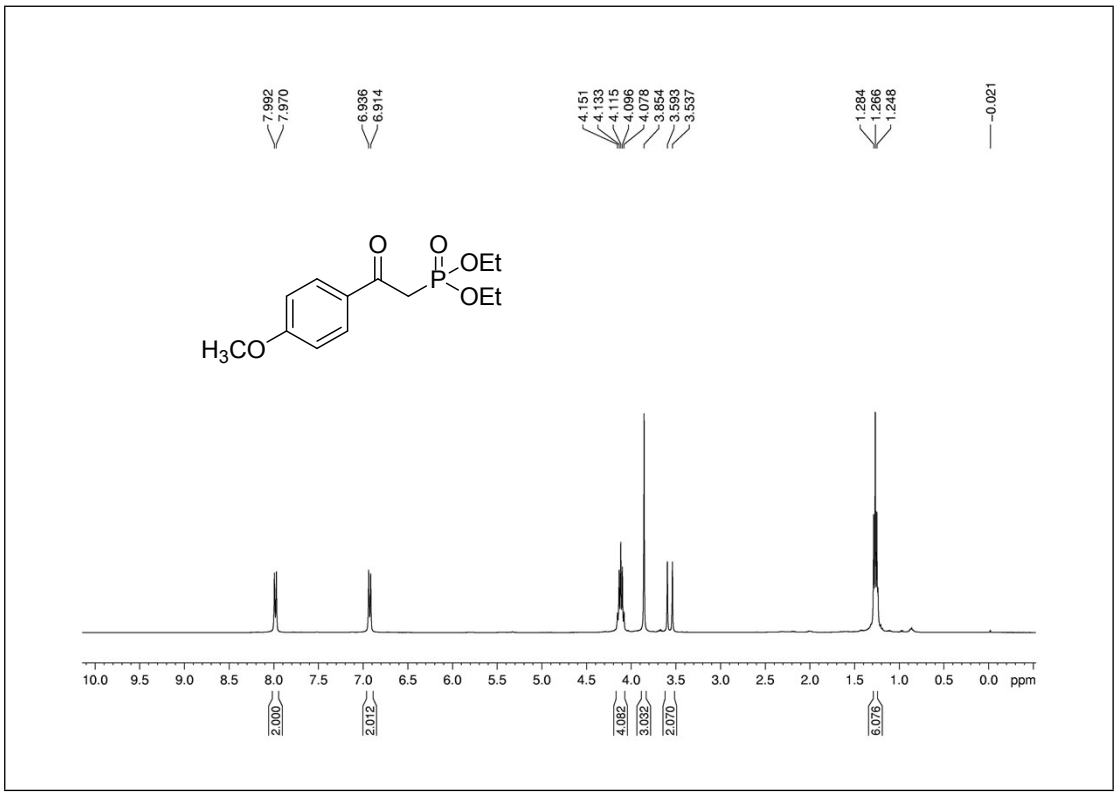

Fig.41 ^{1}H NMR spectrum of compound **3K**

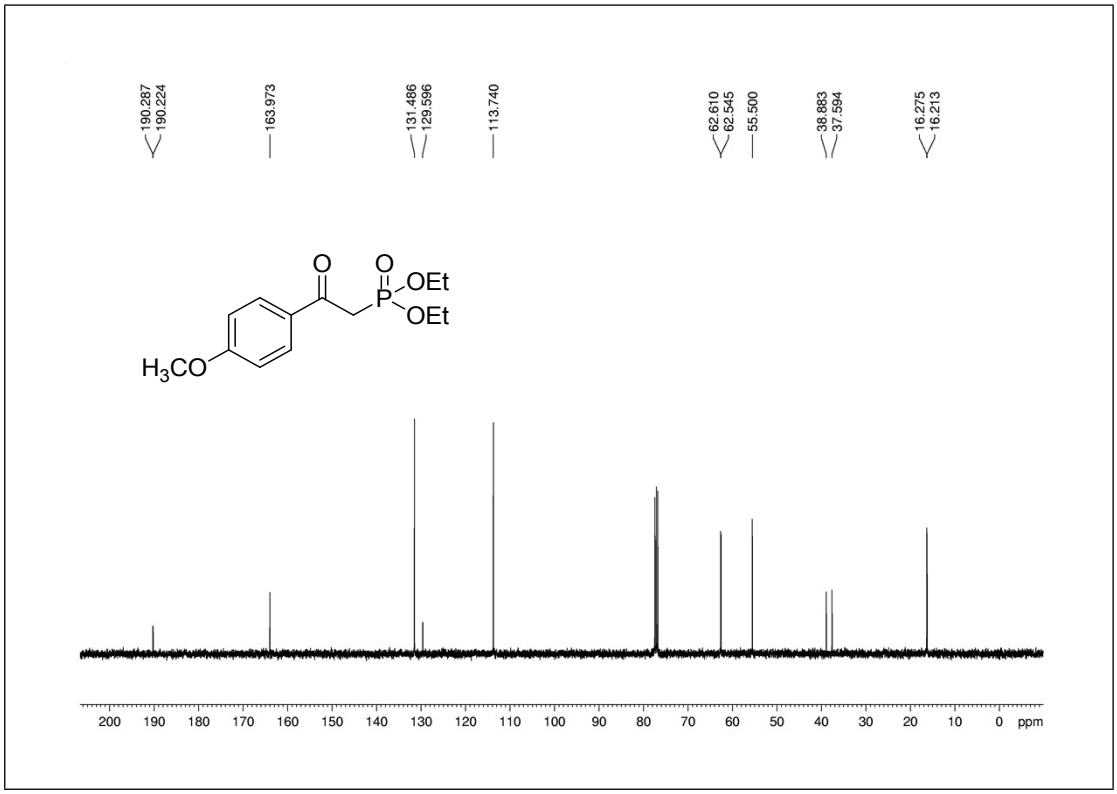

Fig. 42 ^{13}C NMR spectrum of compound **3K**

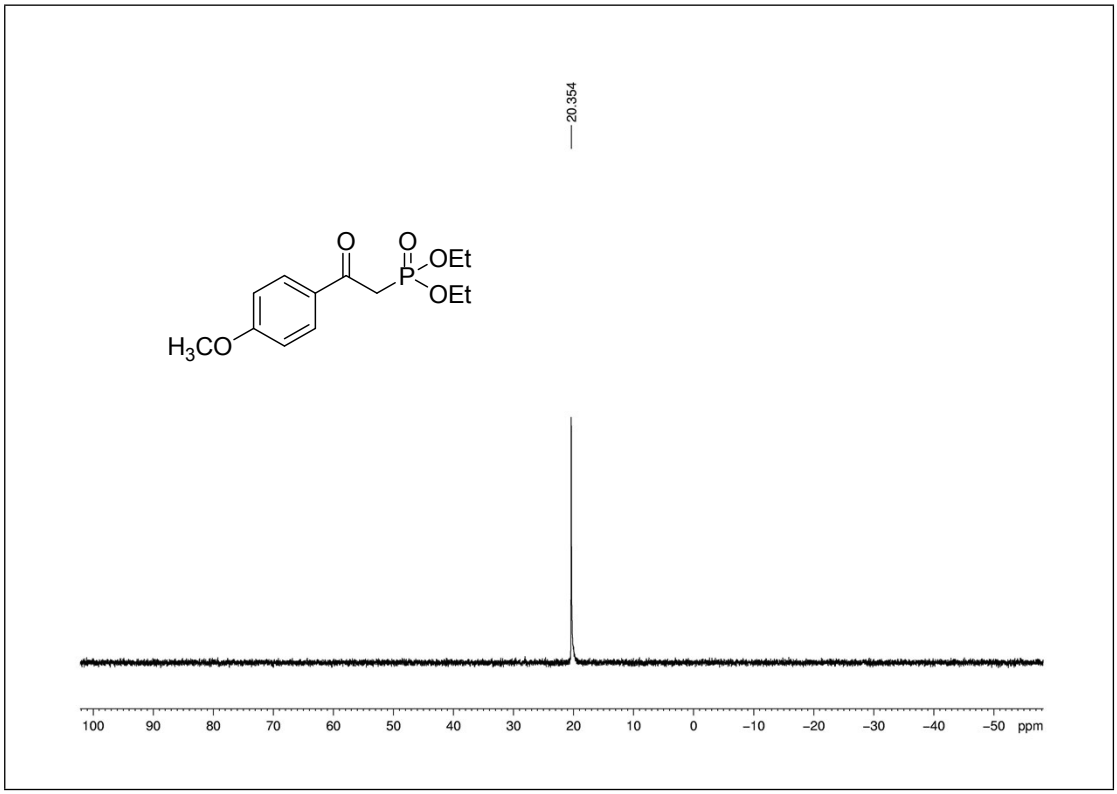

Fig. 43 ^{31}P NMR spectrum of compound **3K**

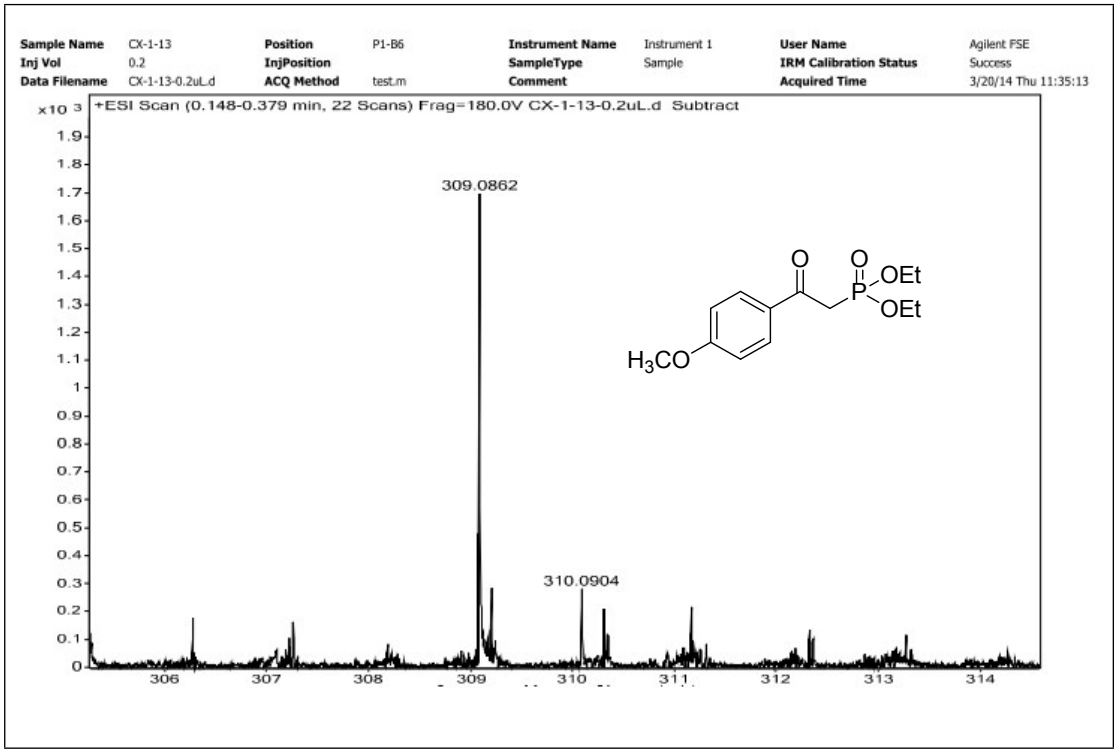

Fig. 44 HRMS spectrum of compound **3K**

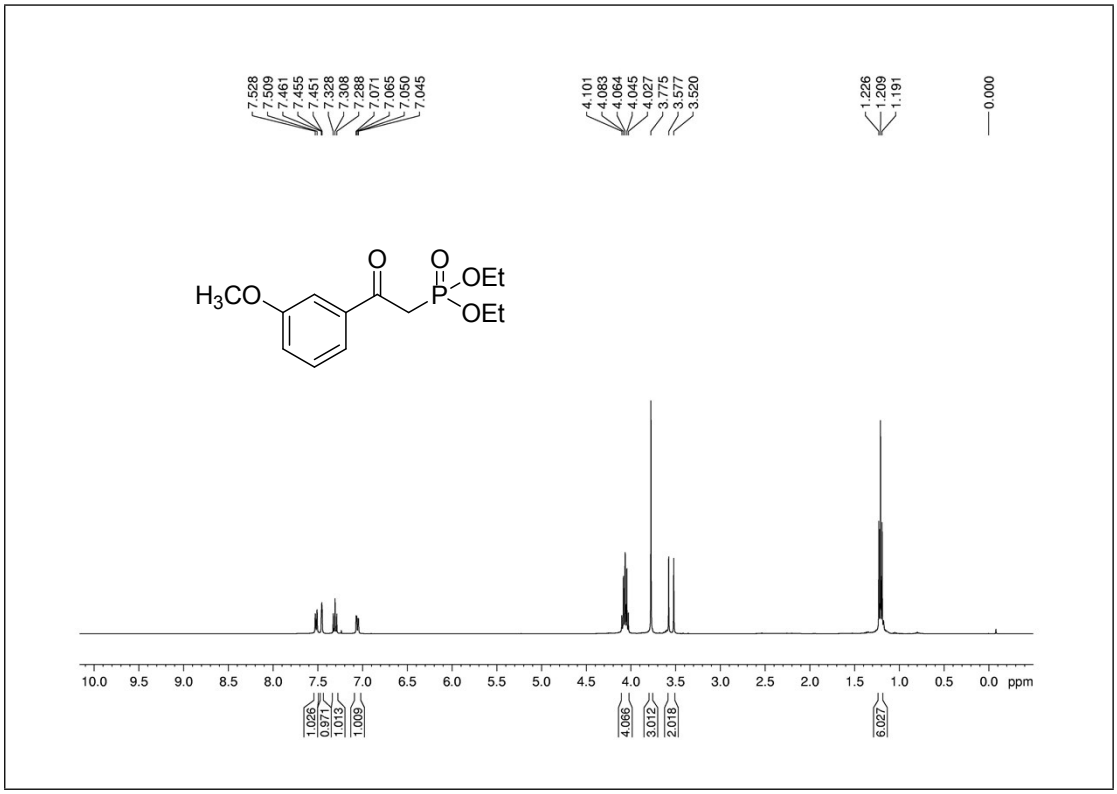

Fig.45 ^{1}H NMR spectrum of compound **3l**

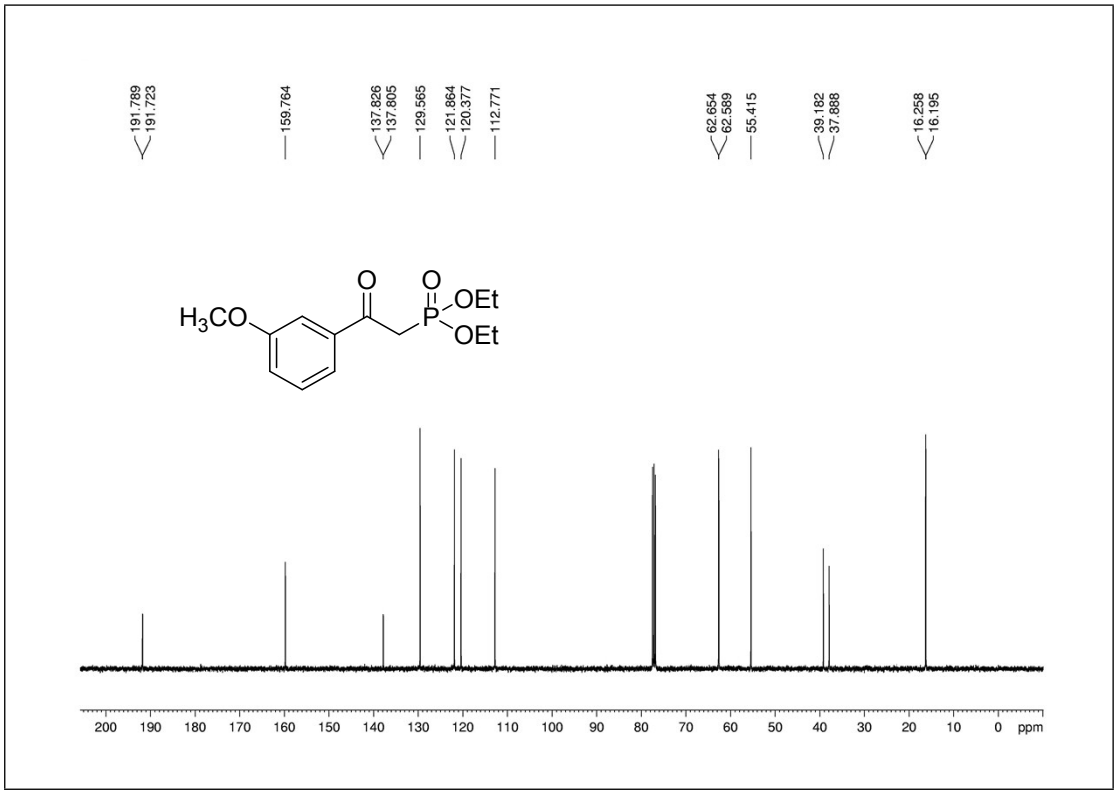

Fig. 46 ^{13}C NMR spectrum of compound **3l**

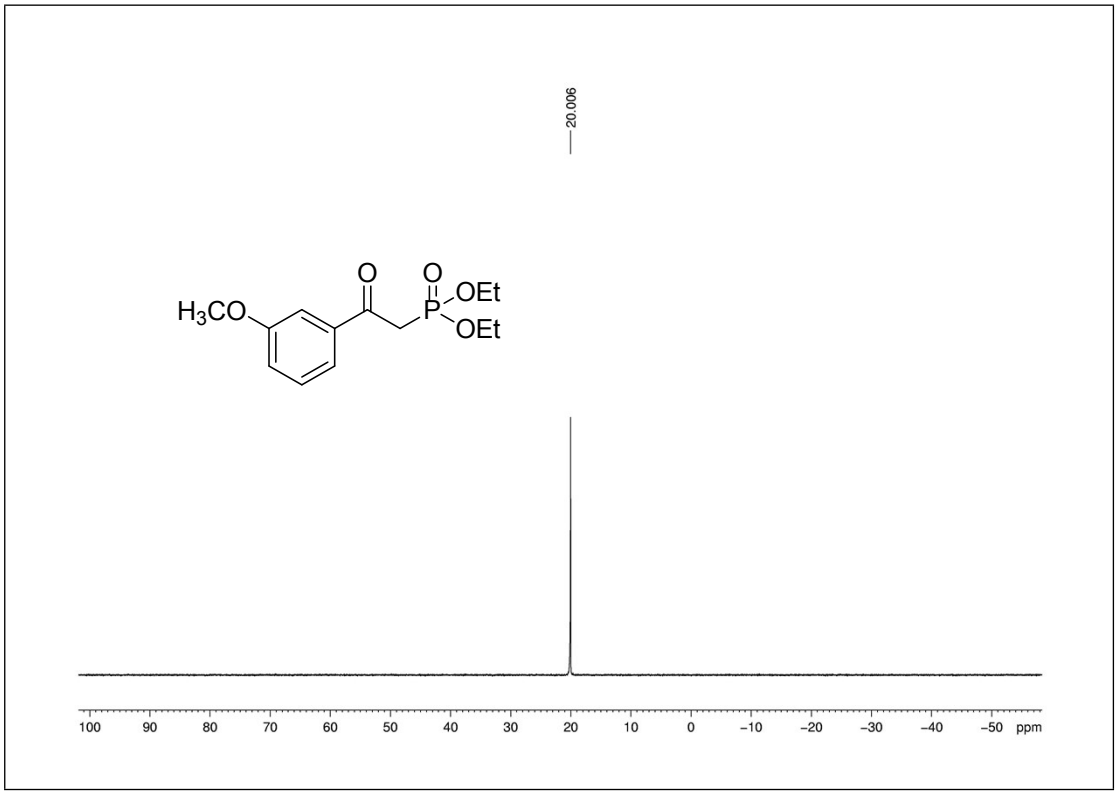

Fig. 47 ^{31}P NMR spectrum of compound **3l**

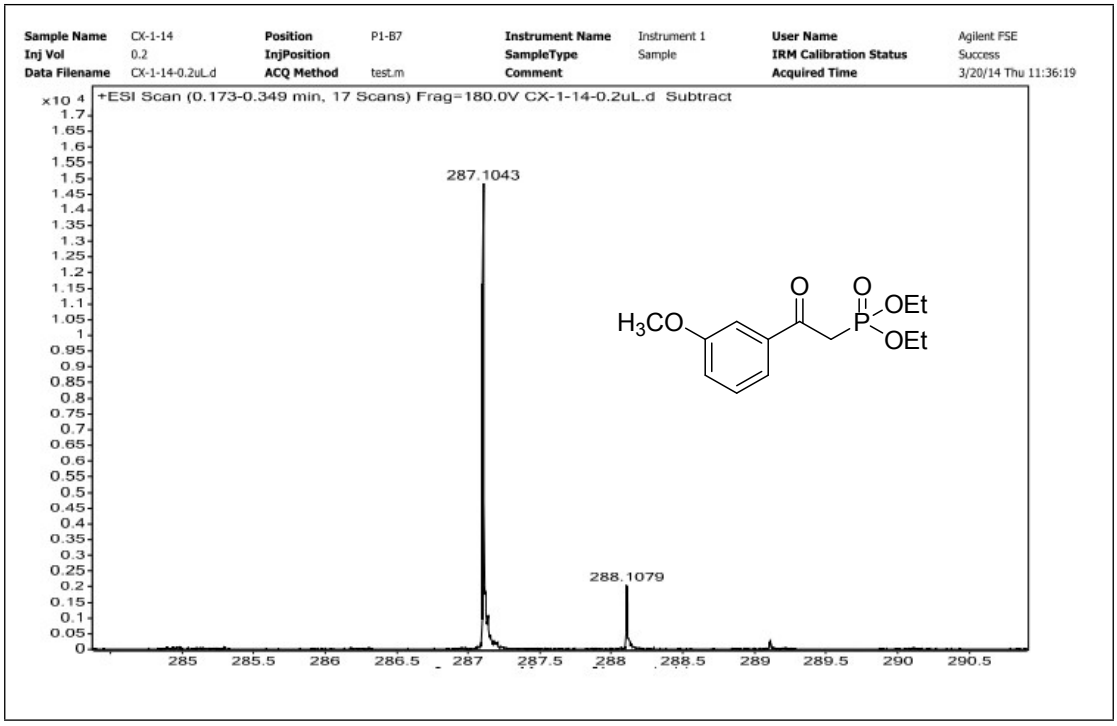

Fig. 48 HRMS spectrum of compound **3l**

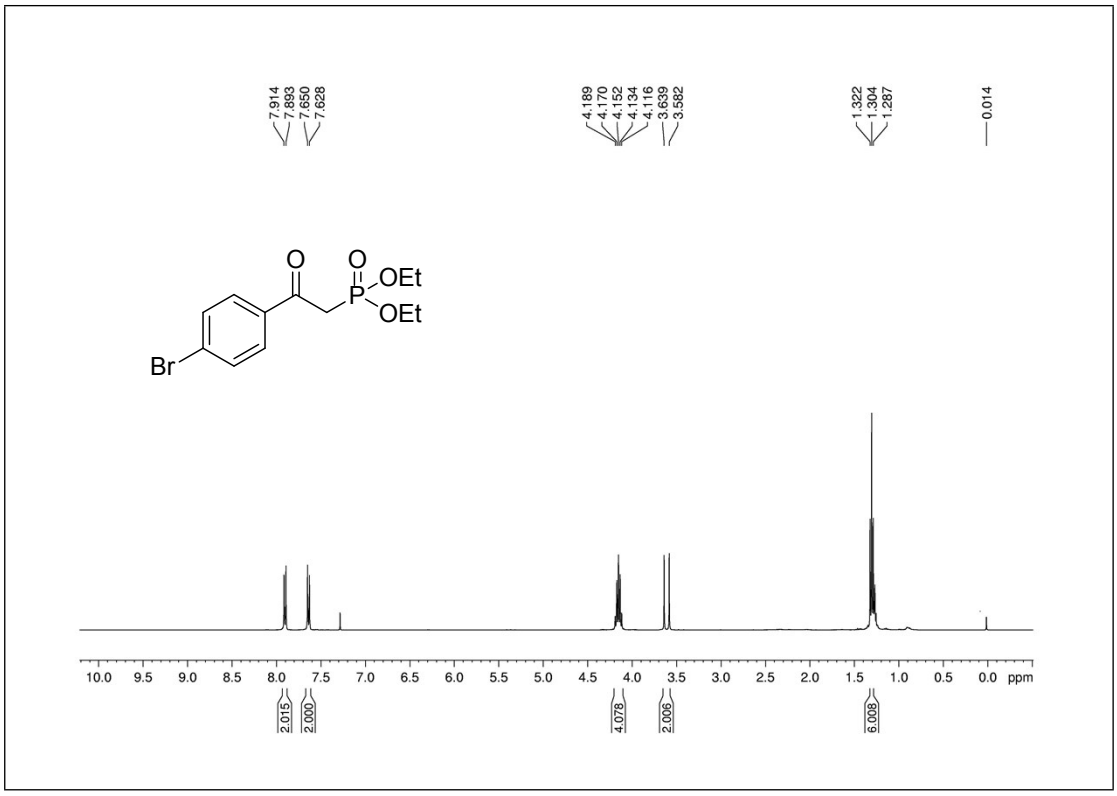

Fig.49 ^{1}H NMR spectrum of compound **3m**

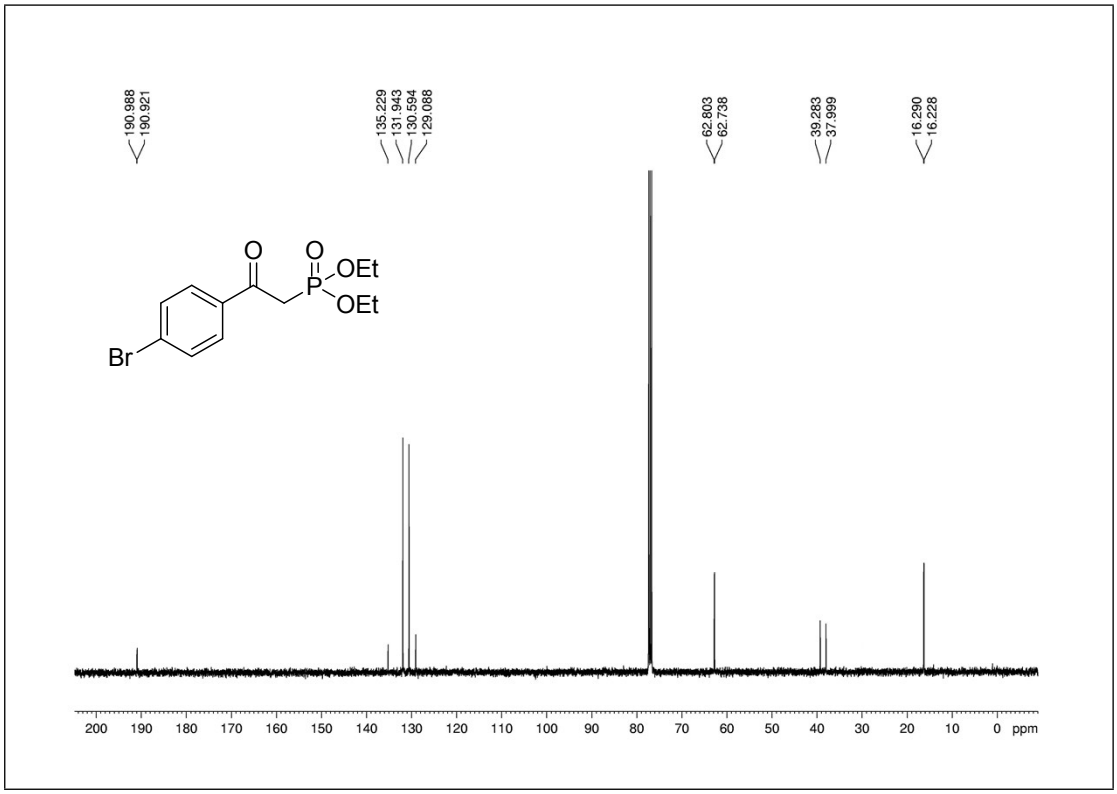

Fig. 50 ^{13}C NMR spectrum of compound **3m**

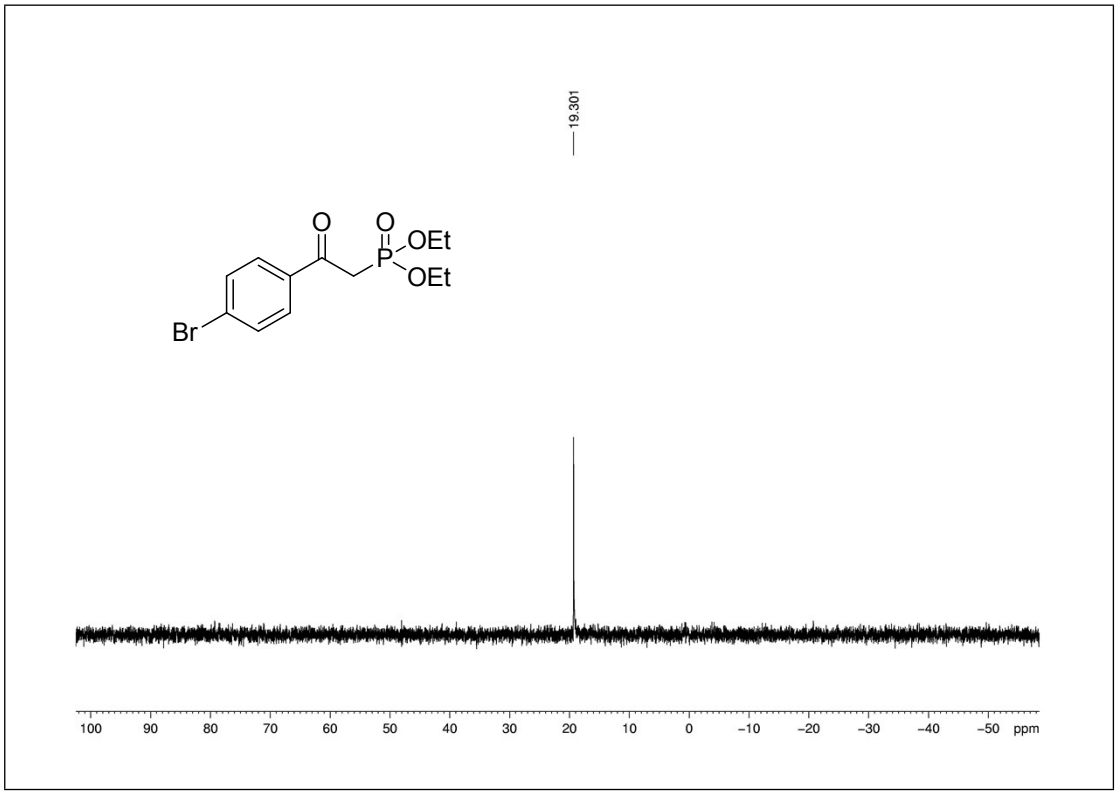

Fig. 51 ^{31}P NMR spectrum of compound **3m**

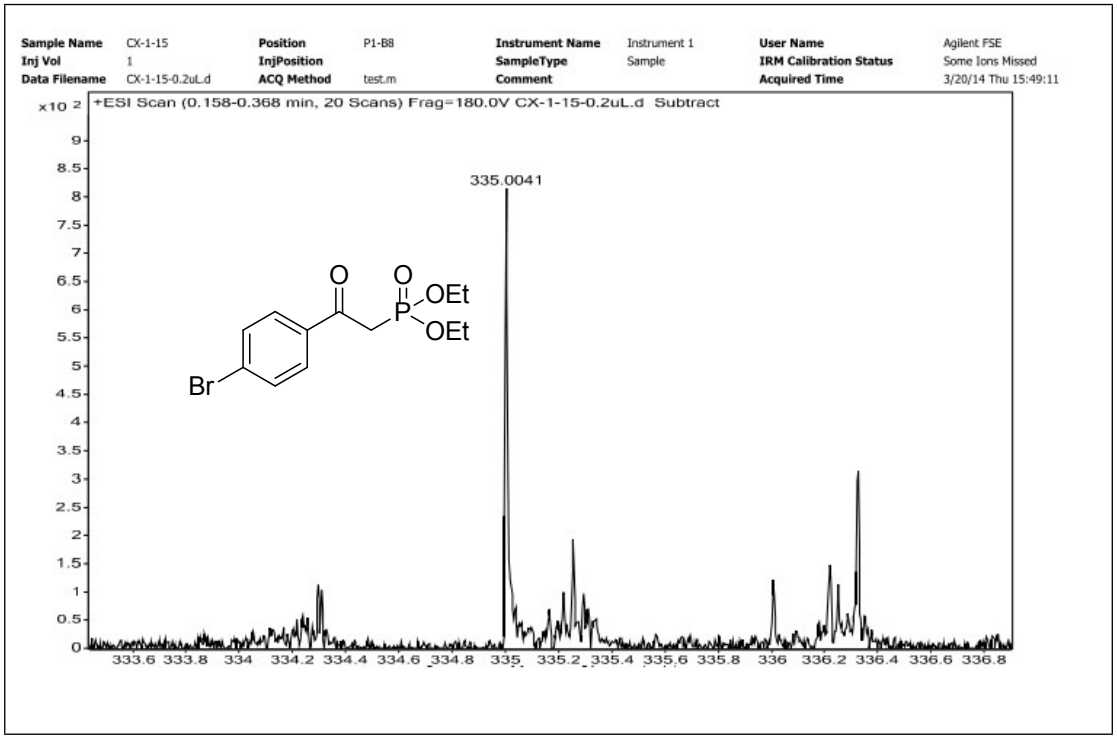

Fig. 52 HRMS spectrum of compound **3m**

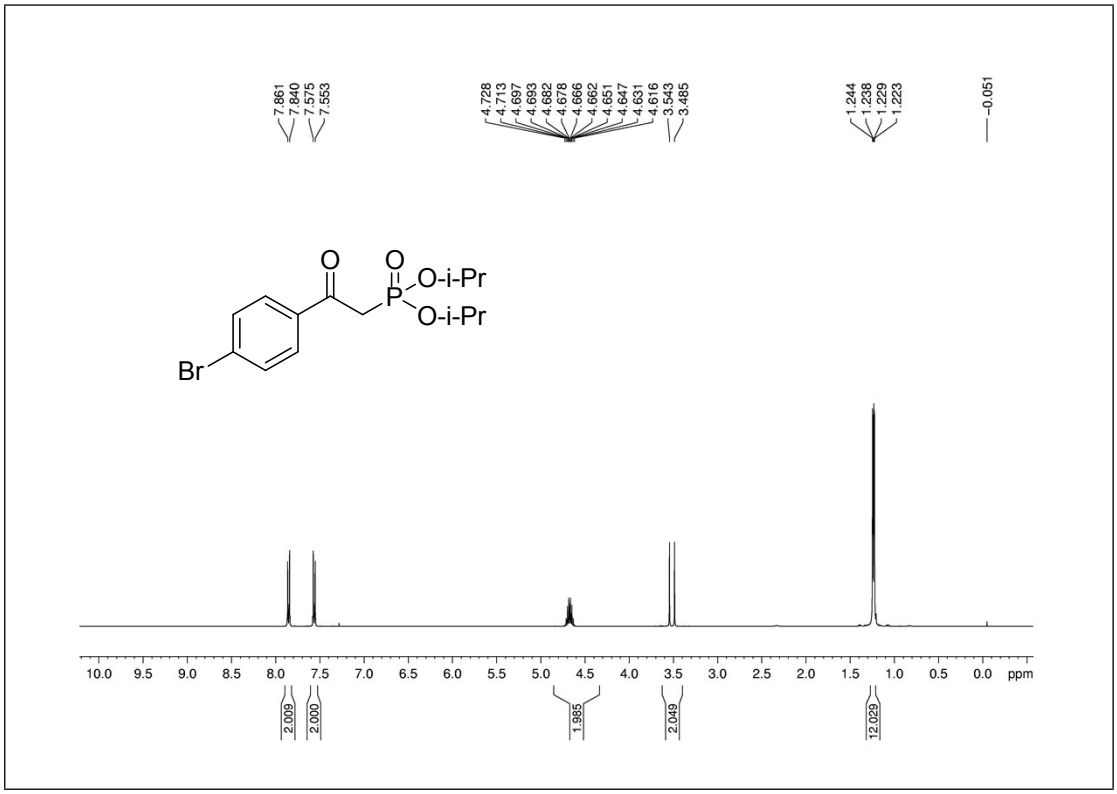

Fig.53 [1]H NMR spectrum of compound **3n**

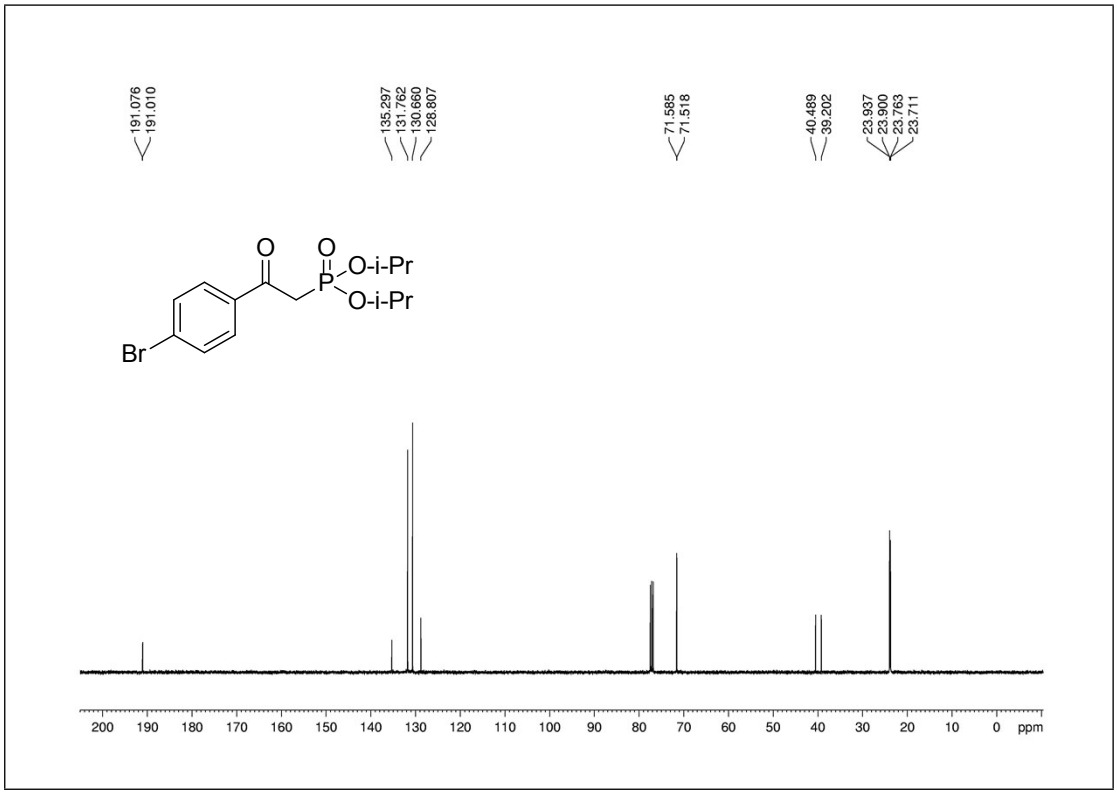

Fig. 54 ^{13}C NMR spectrum of compound **3n**

Fig. 55 ^{31}P NMR spectrum of compound **3n**

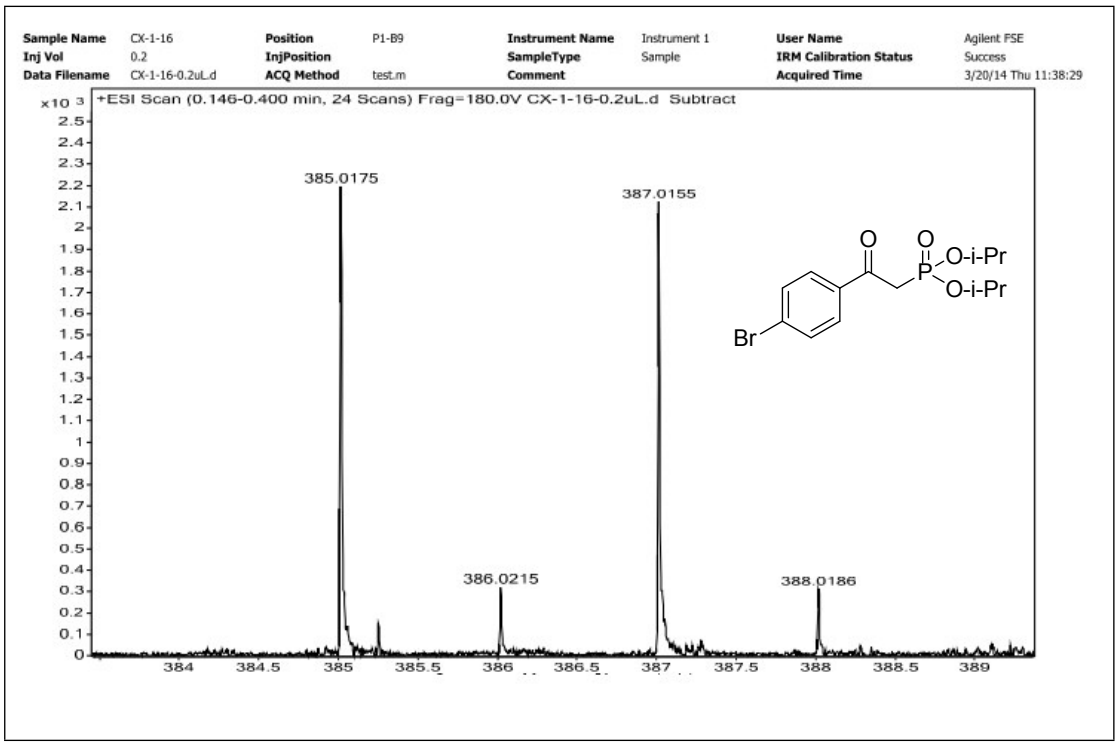

Fig. 56 HRMS spectrum of compound **3n**

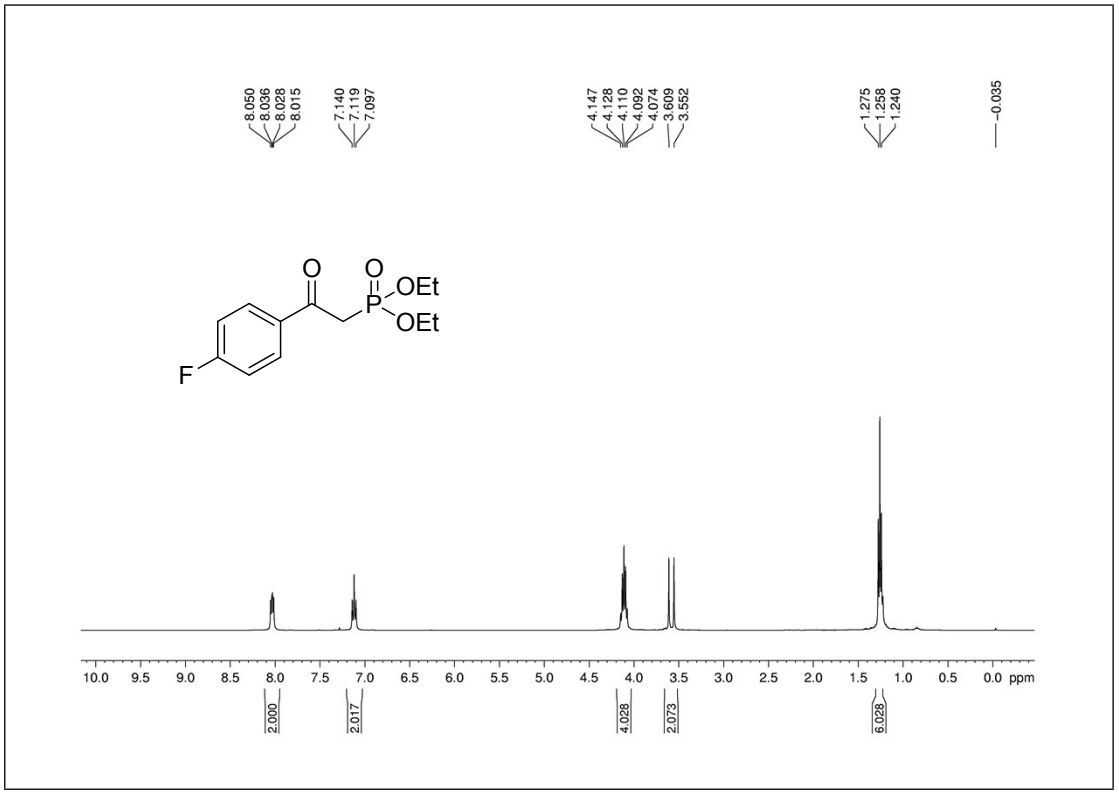

Fig.57 ^{1}H NMR spectrum of compound **3o**

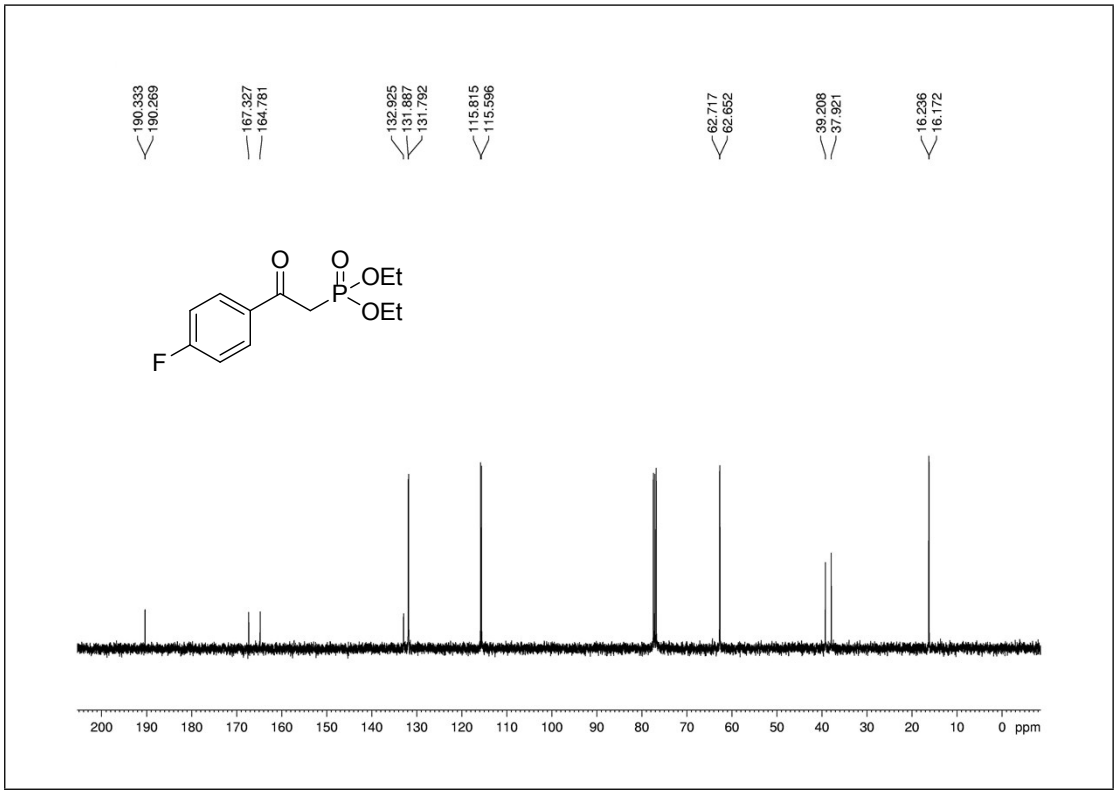

Fig. 58 ^{13}C NMR spectrum of compound **3o**

Fig. 59 ^{31}P NMR spectrum of compound **3o**

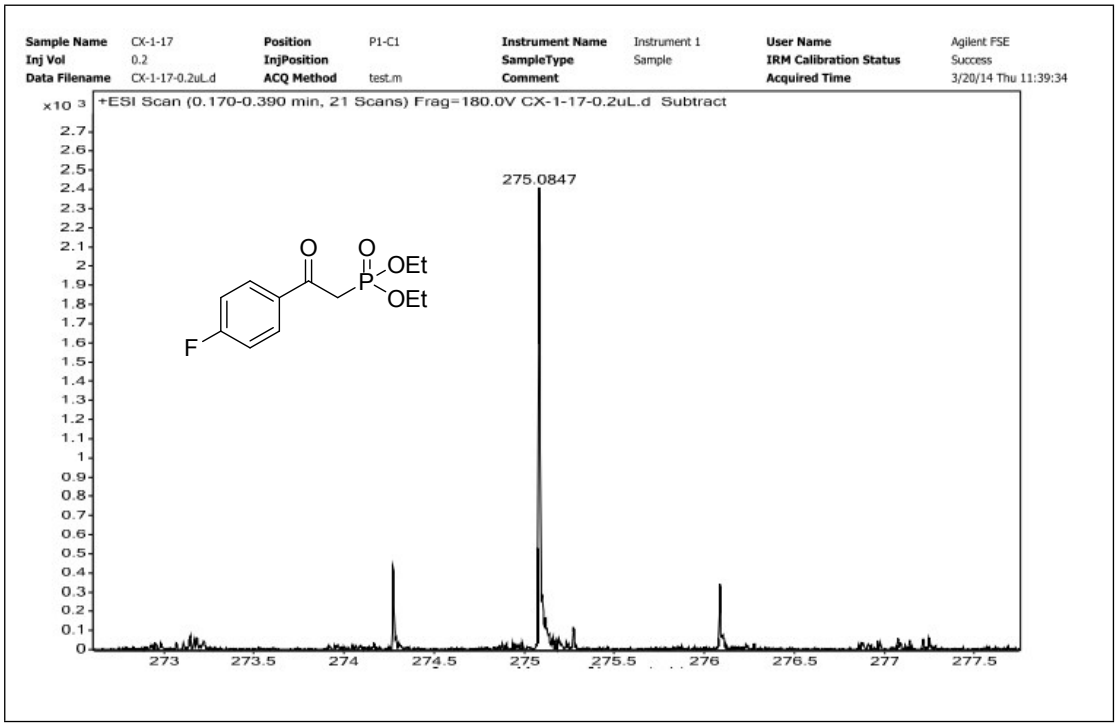

Fig. 60 HRMS spectrum of compound **3o**

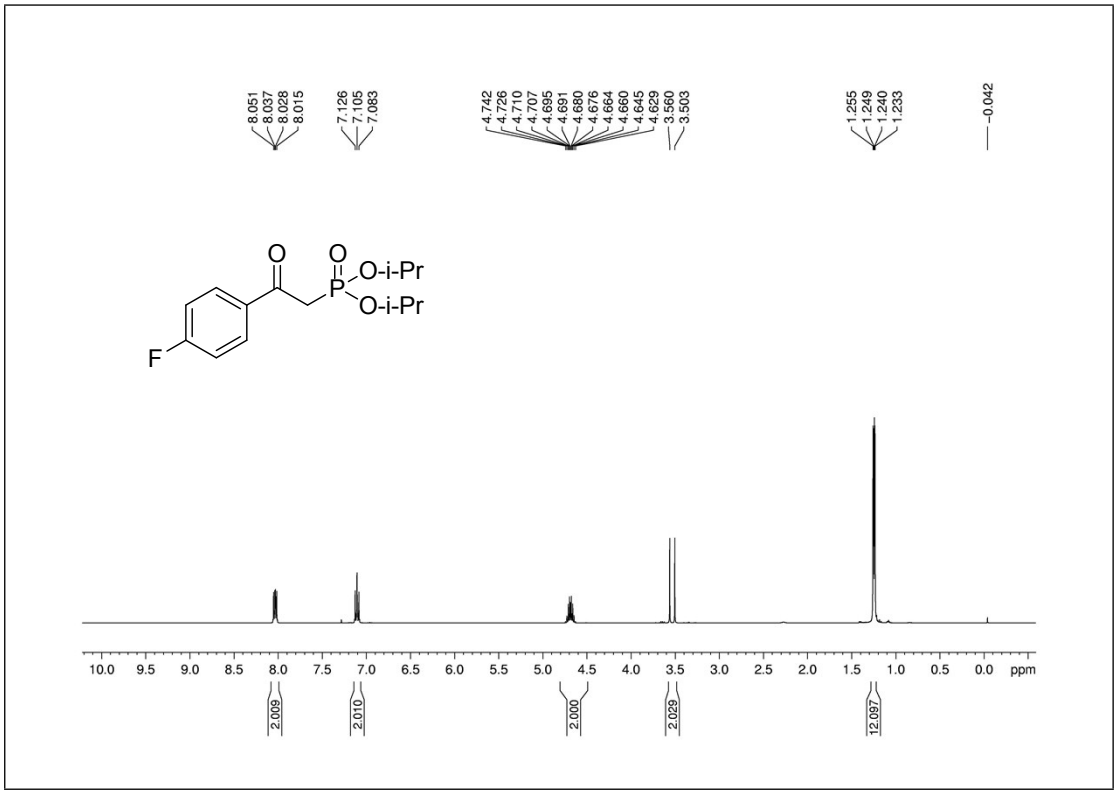

Fig.61 ^{1}H NMR spectrum of compound **3p**

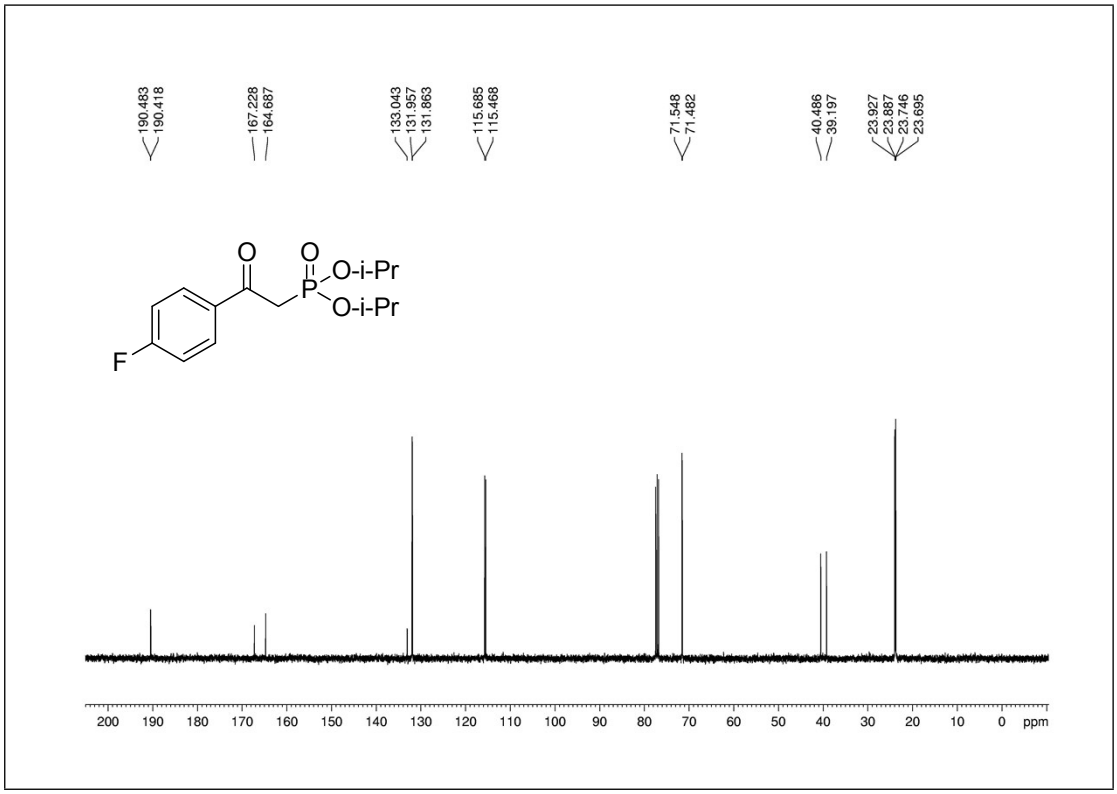

Fig. 62 ^{13}C NMR spectrum of compound **3p**

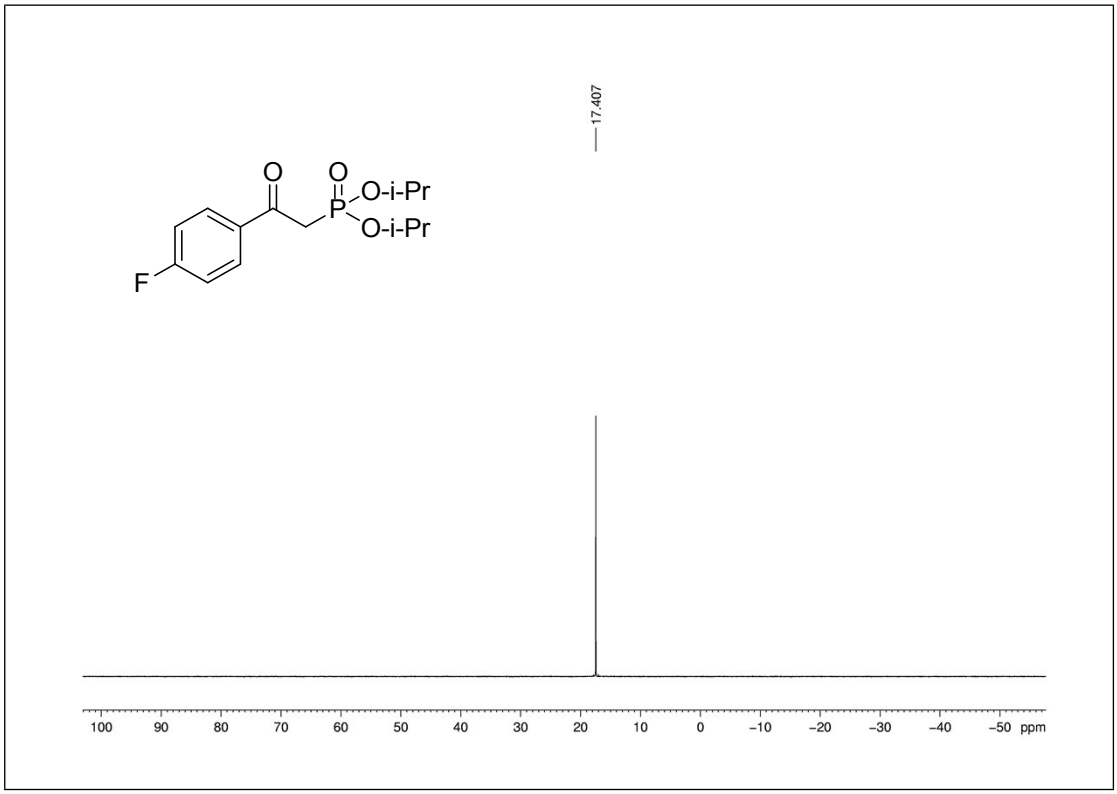

Fig. 63 ^{31}P NMR spectrum of compound **3p**

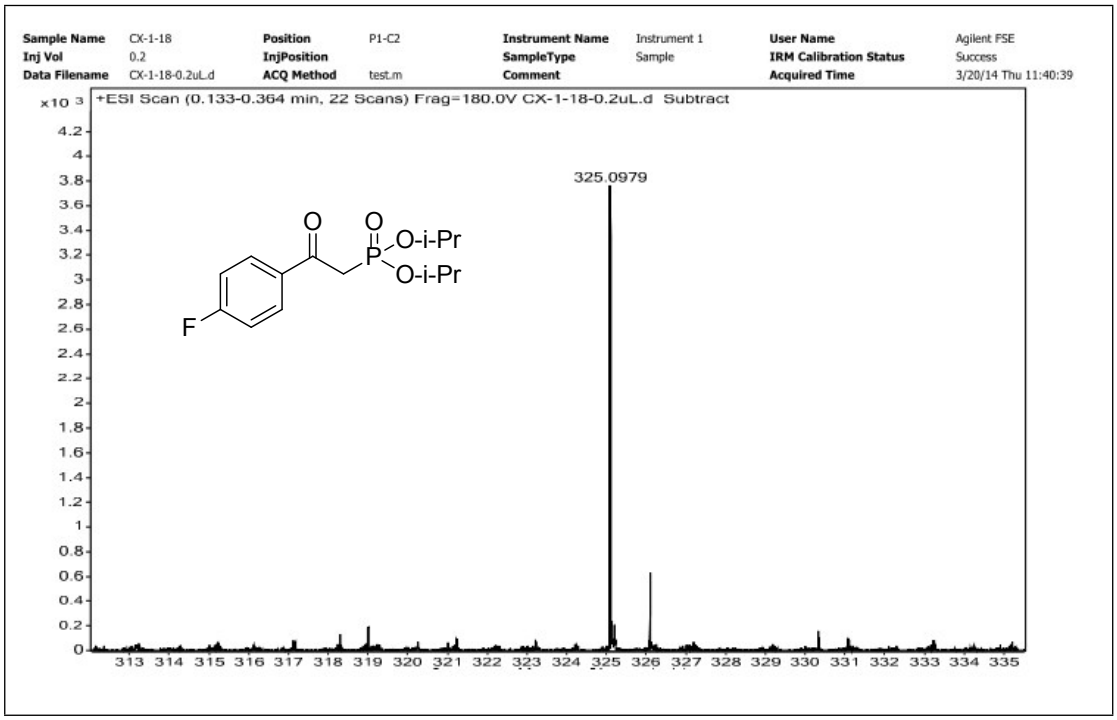

Fig. 64 HRMS spectrum of compound **3p**

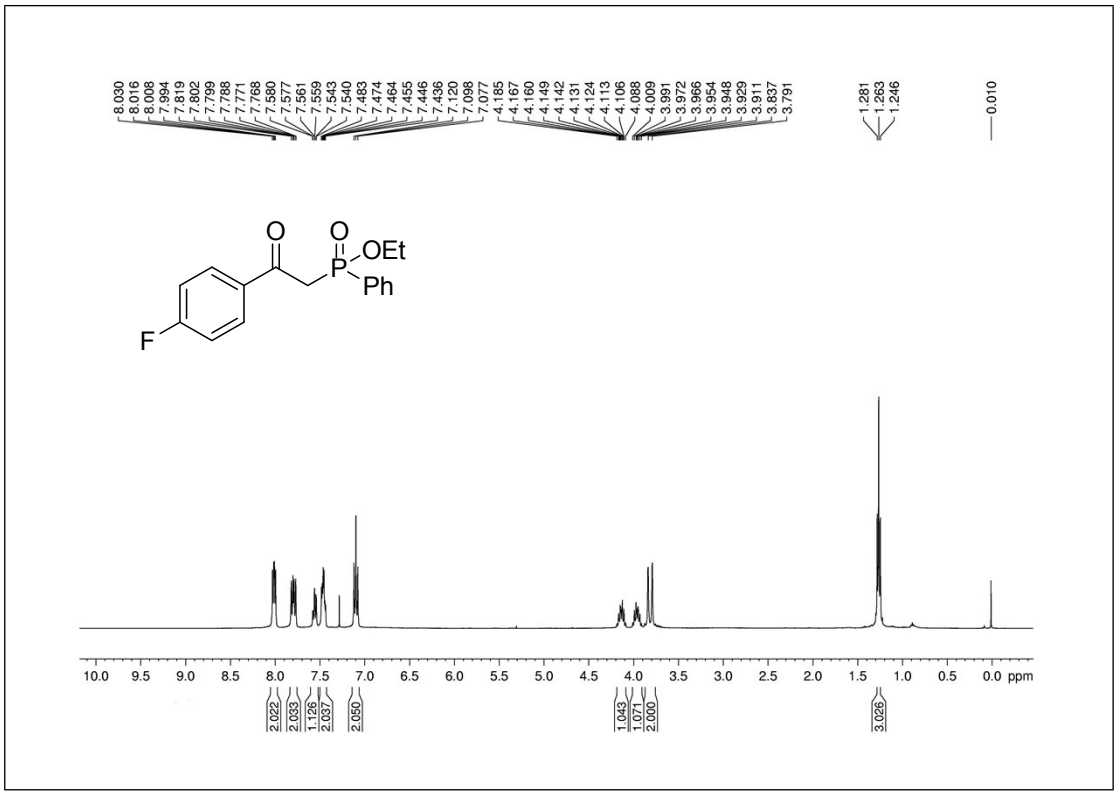

Fig.65 ^{1}H NMR spectrum of compound **3q**

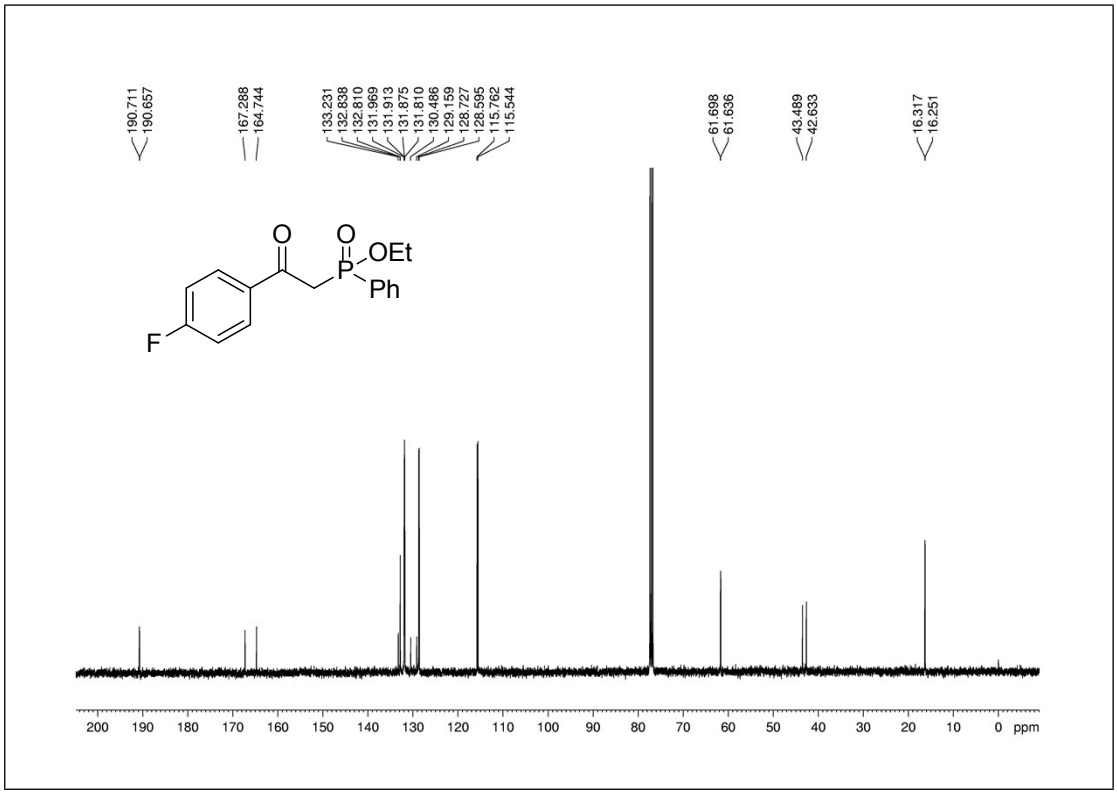

Fig. 66 ^{13}C NMR spectrum of compound **3q**

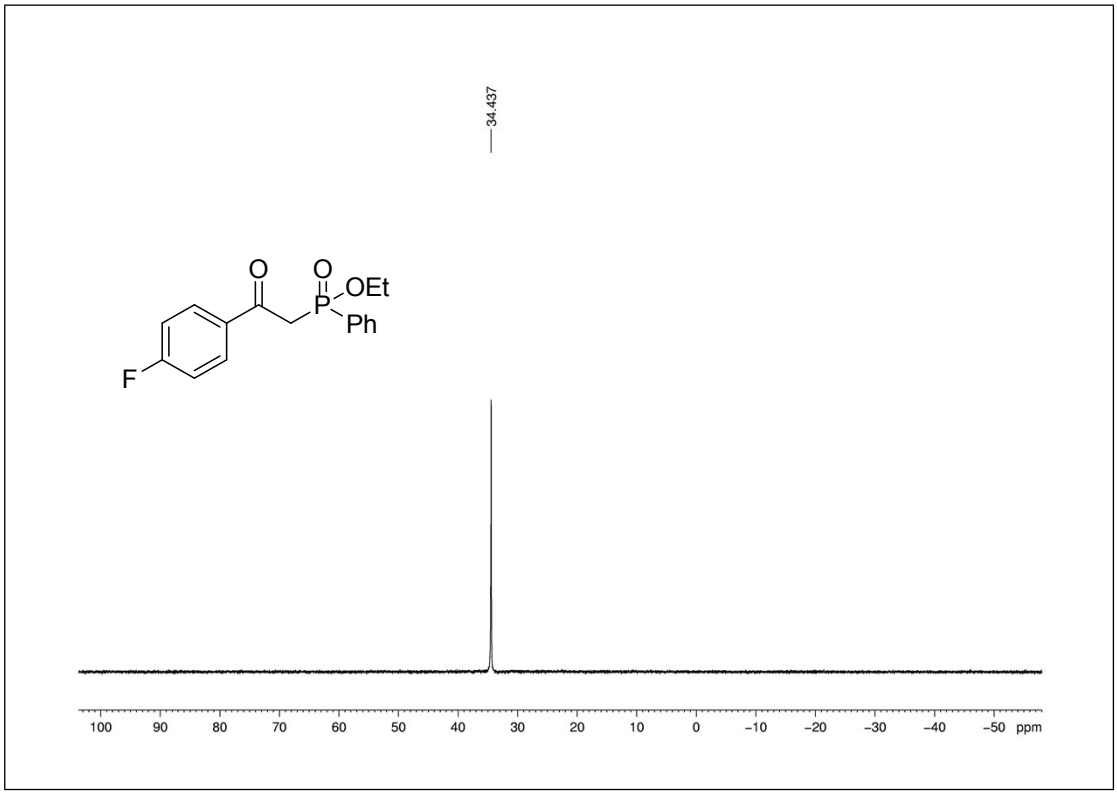

Fig. 67 ^{31}P NMR spectrum of compound **3q**

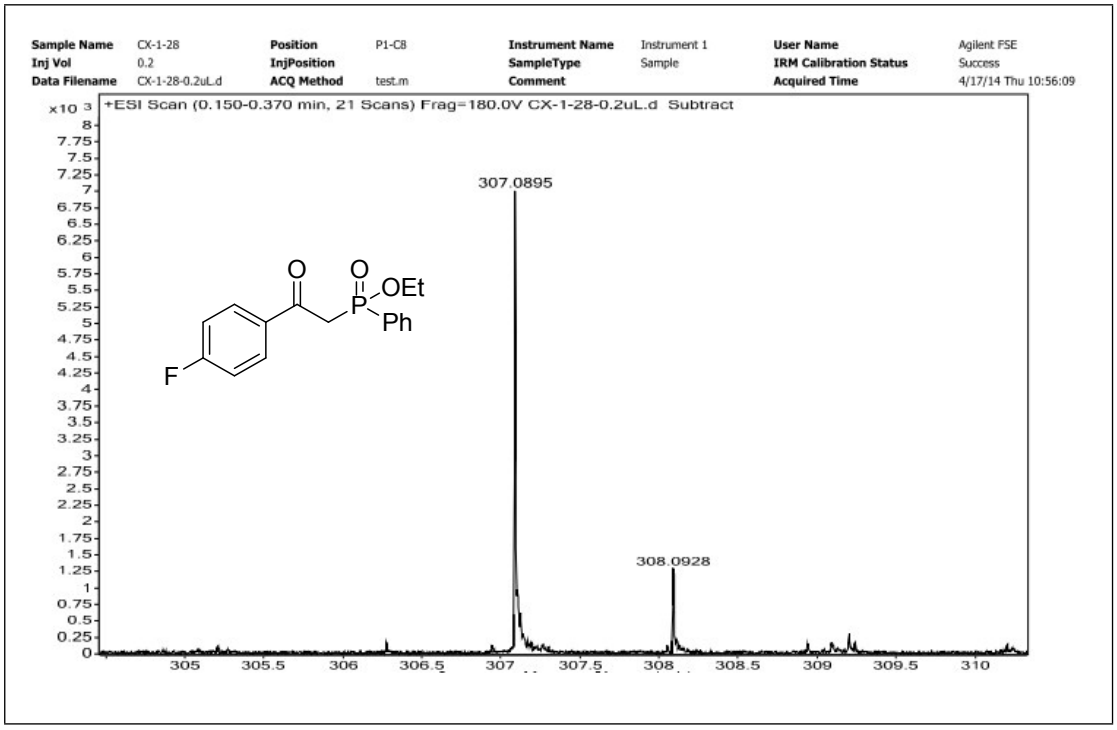

Fig. 68 HRMS spectrum of compound **3q**

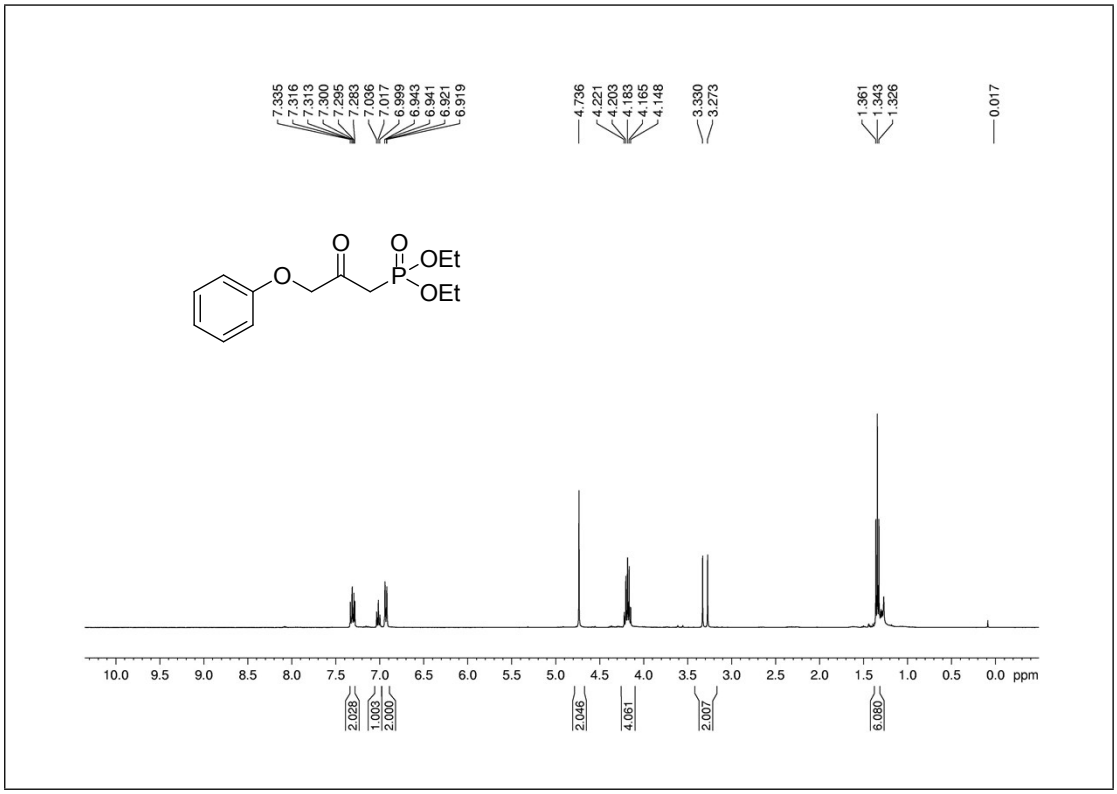

Fig.69 ^{1}H NMR spectrum of compound **3r**

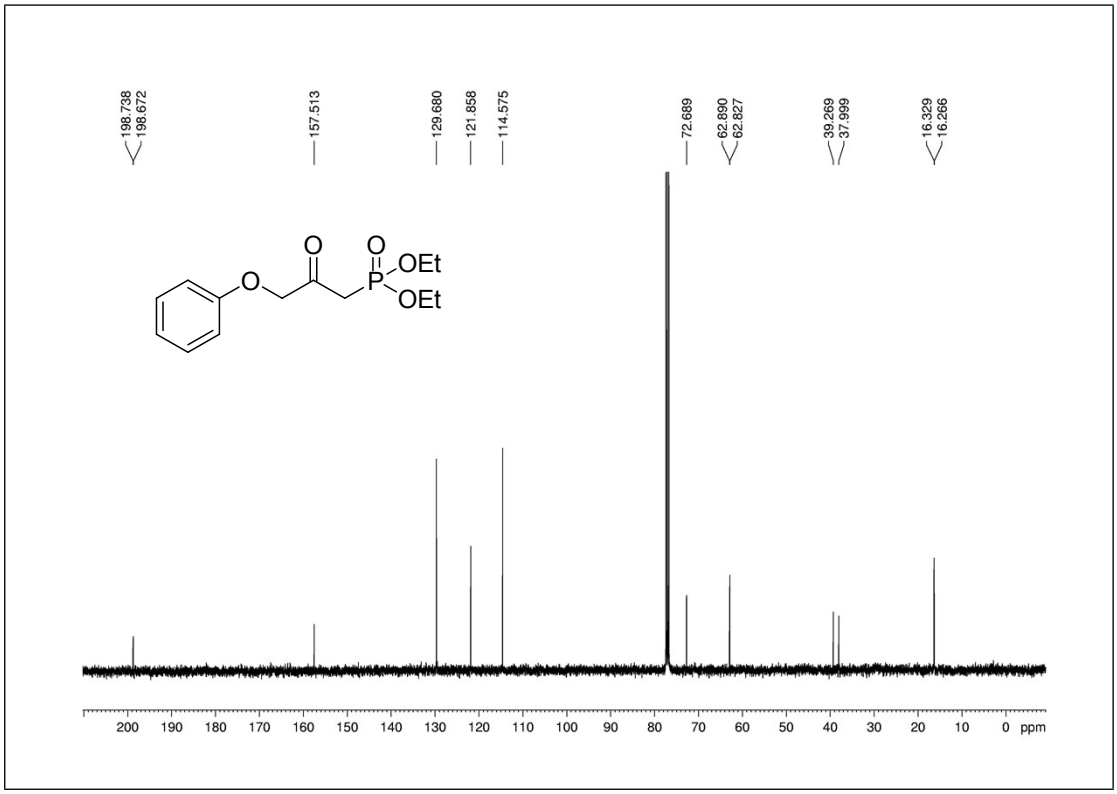

Fig. 70 ^{13}C NMR spectrum of compound **3r**

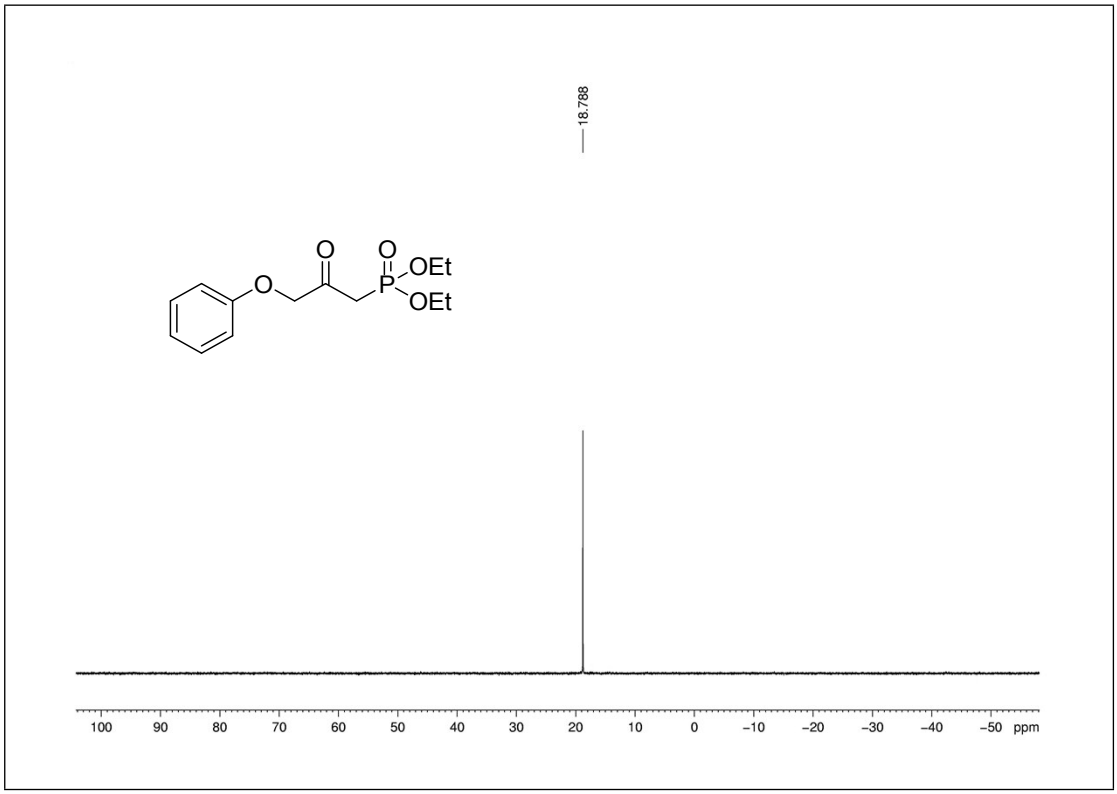

Fig. 71 ^{31}P NMR spectrum of compound **3r**

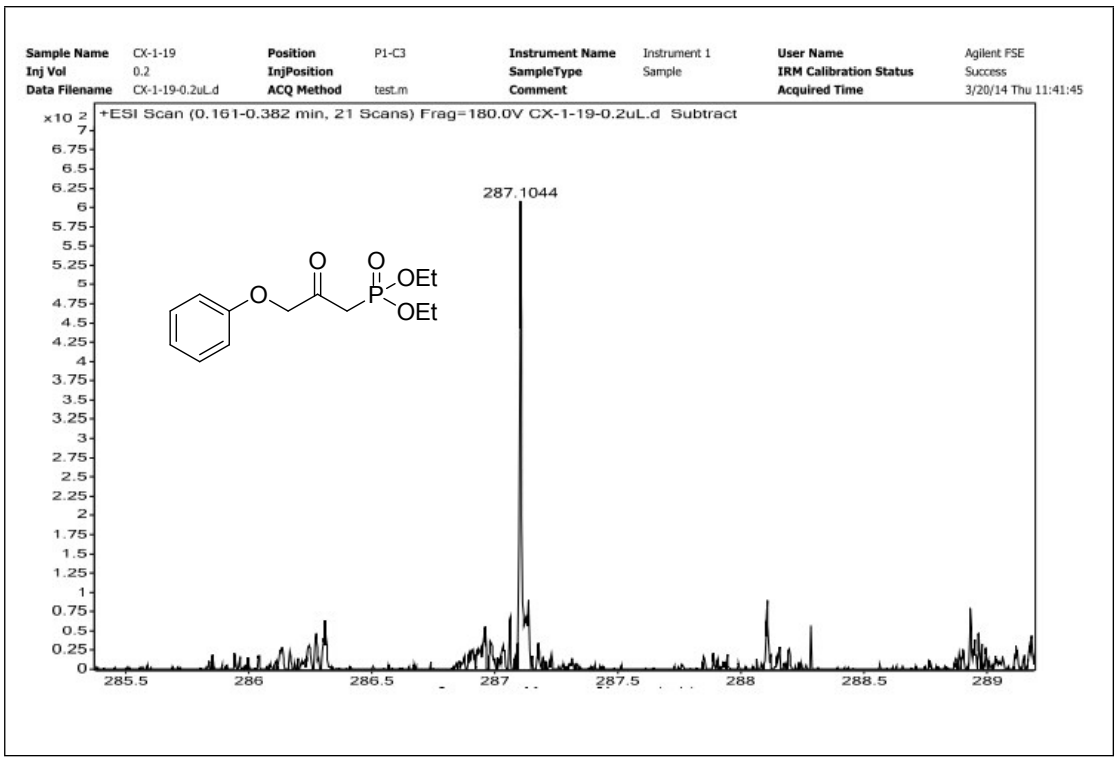

Fig. 72 HRMS spectrum of compound **3r**

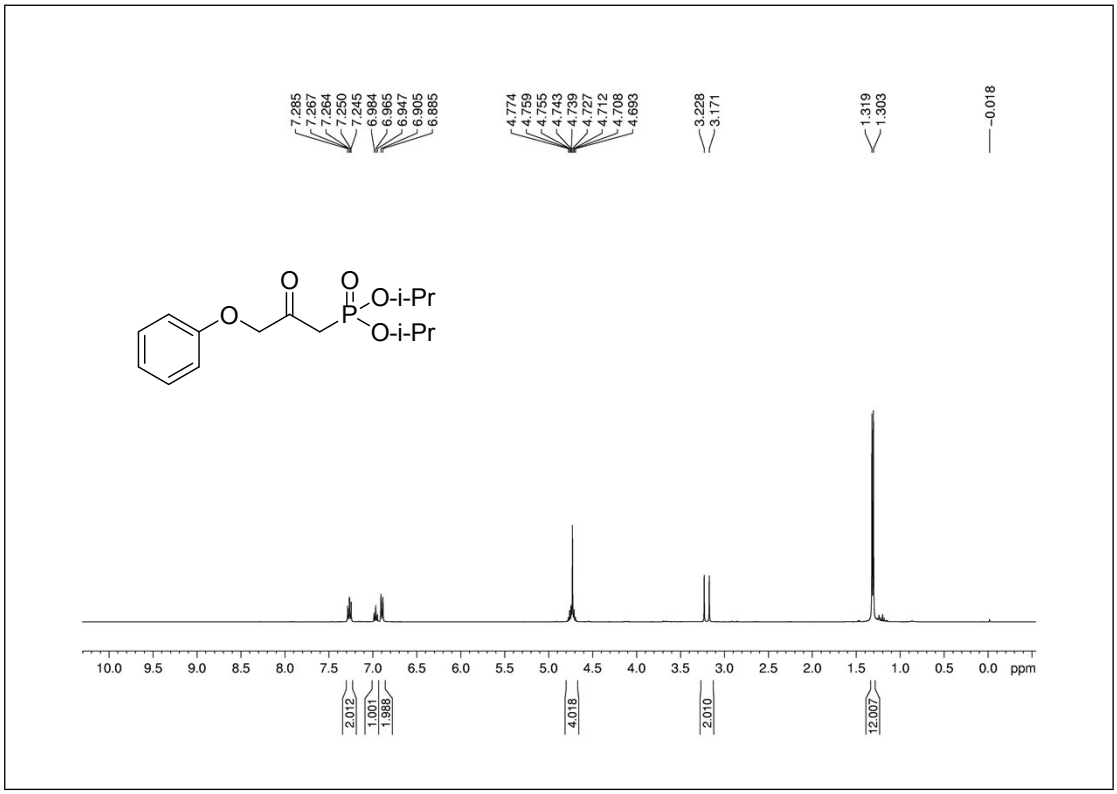

Fig.73 ^{1}H NMR spectrum of compound **3s**

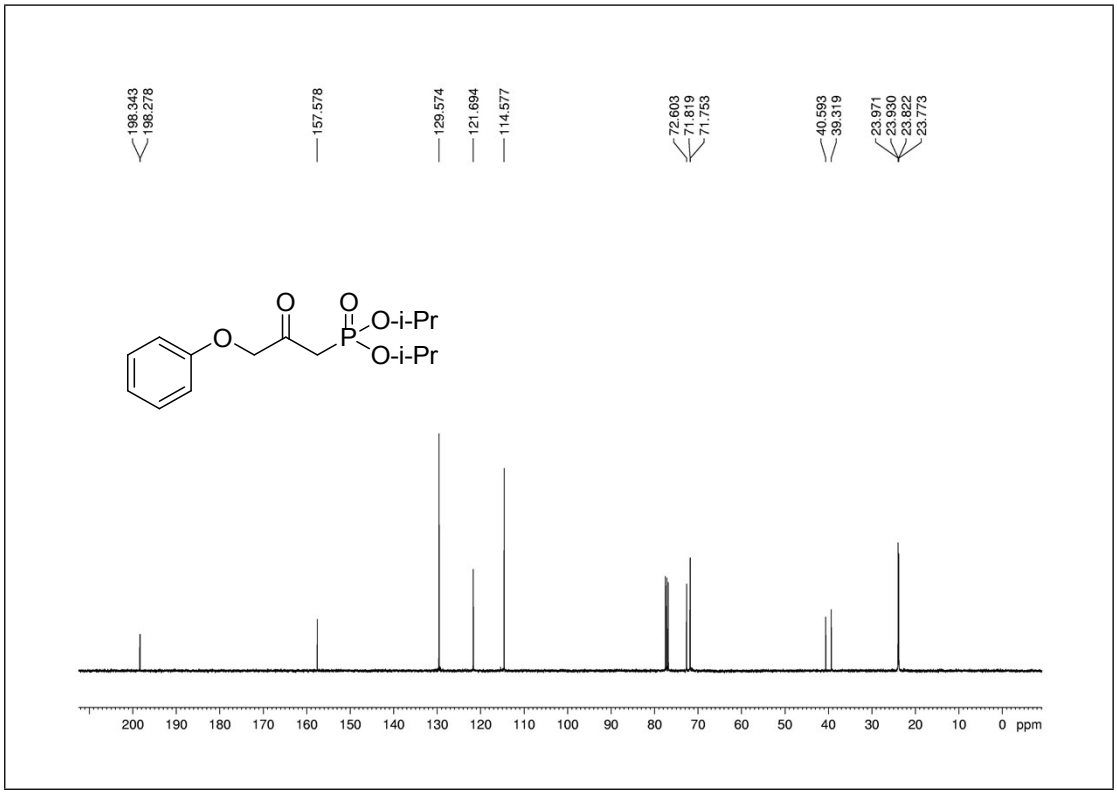

Fig. 74 ^{13}C NMR spectrum of compound **3s**

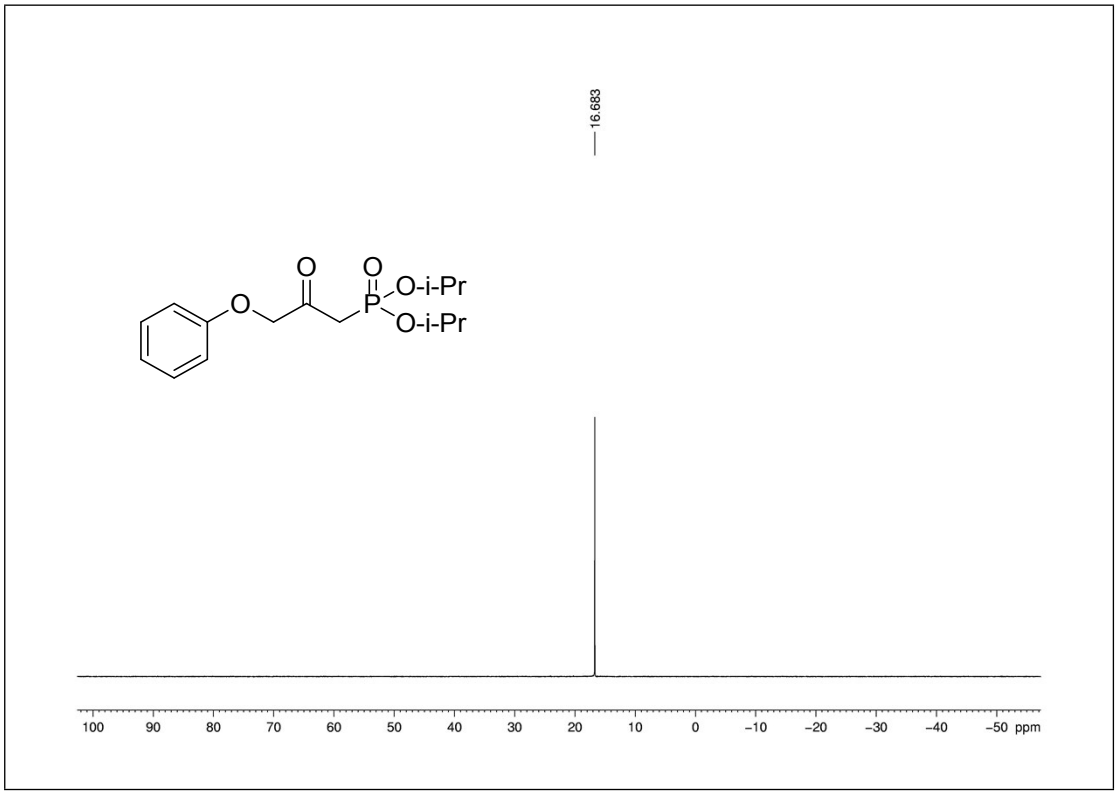

Fig. 75 ^{31}P NMR spectrum of compound **3s**

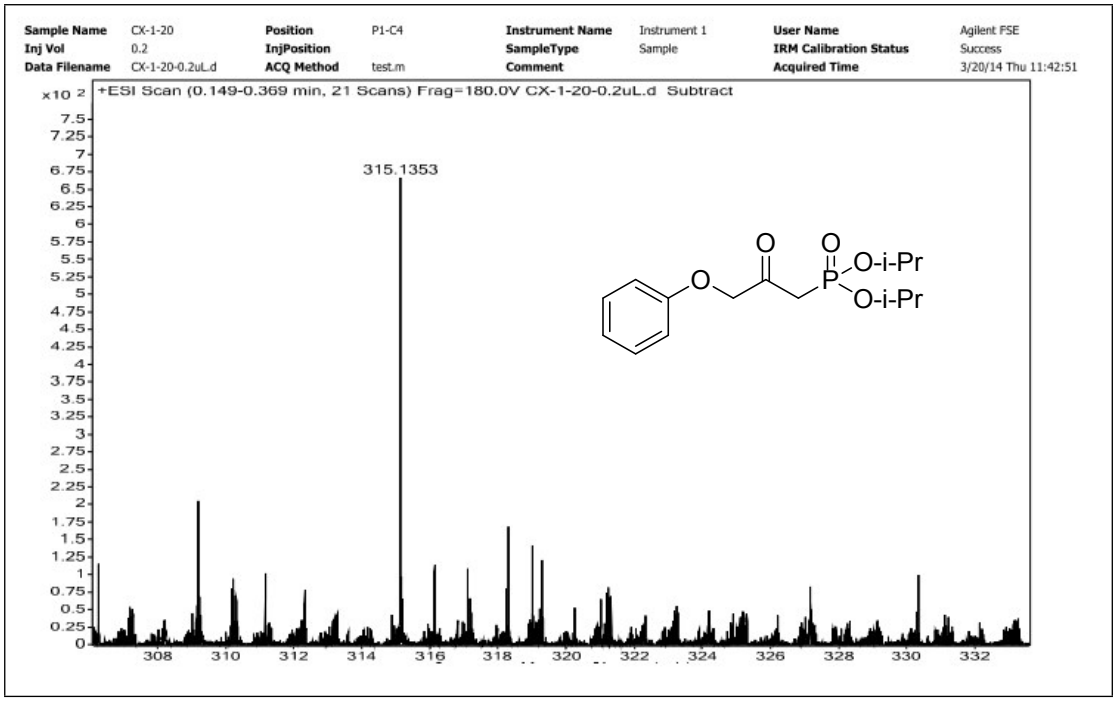

Fig. 76 HRMS spectrum of compound **3s**

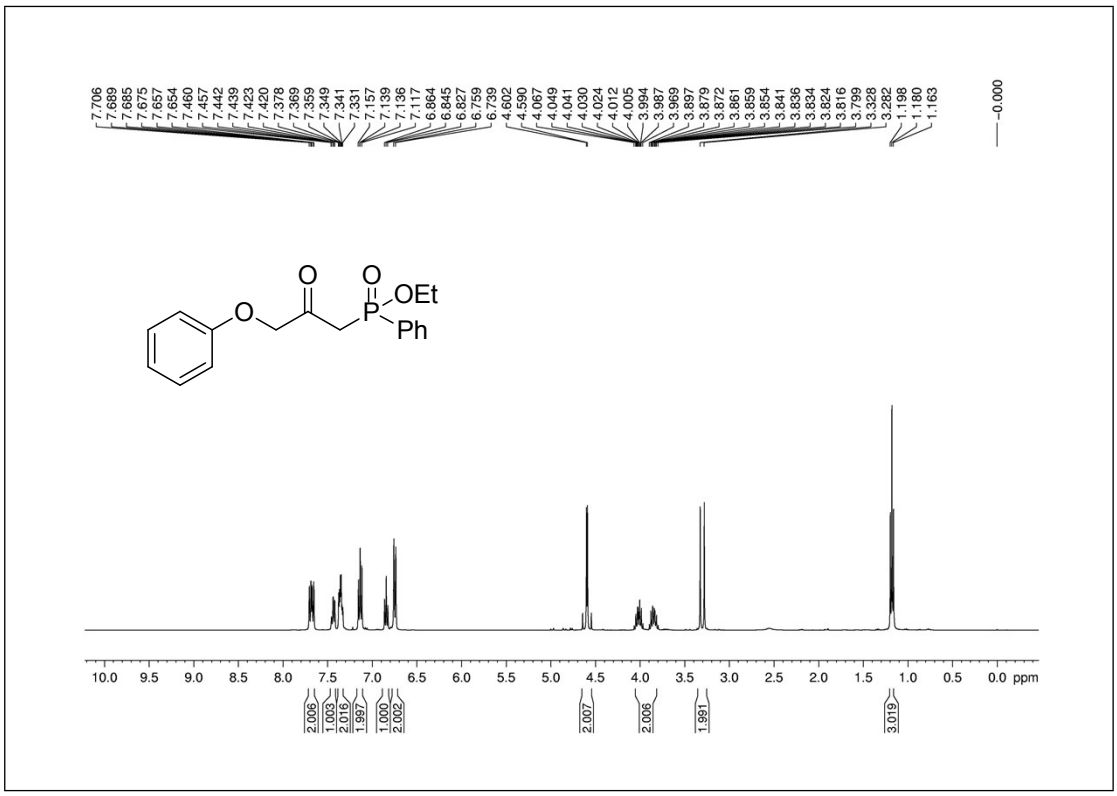

Fig.77 ^{1}H NMR spectrum of compound **3t**

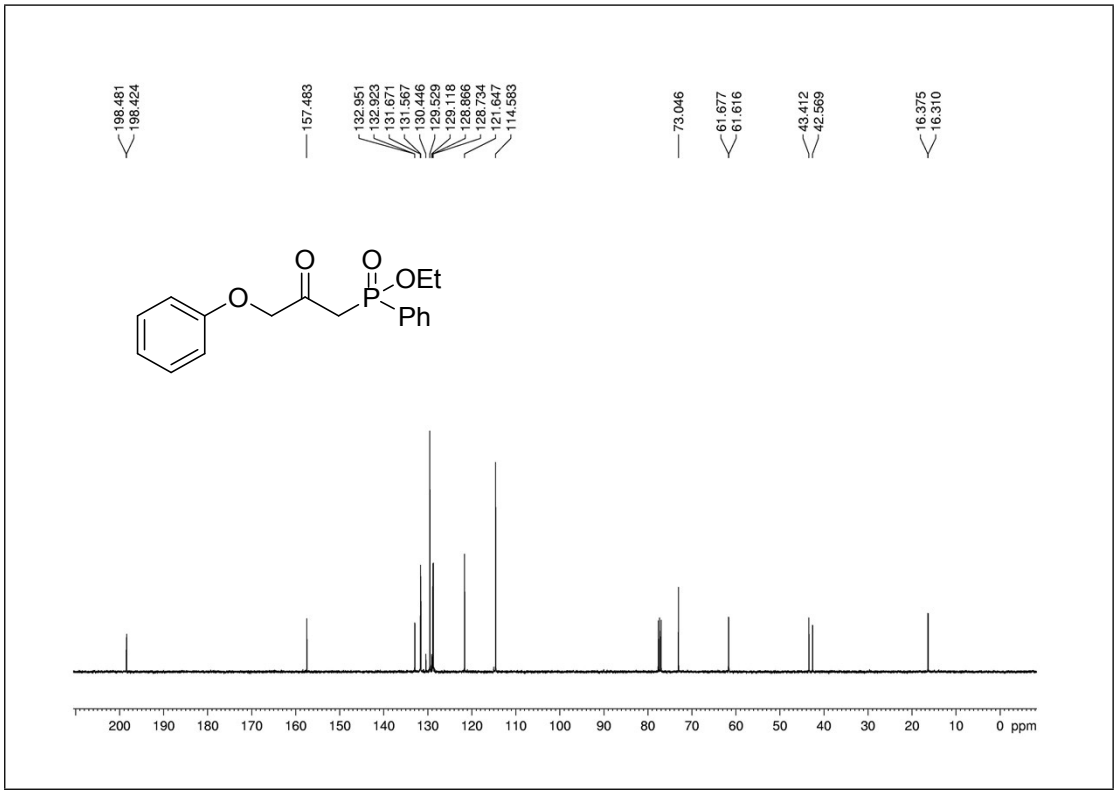

Fig. 78 ^{13}C NMR spectrum of compound **3t**

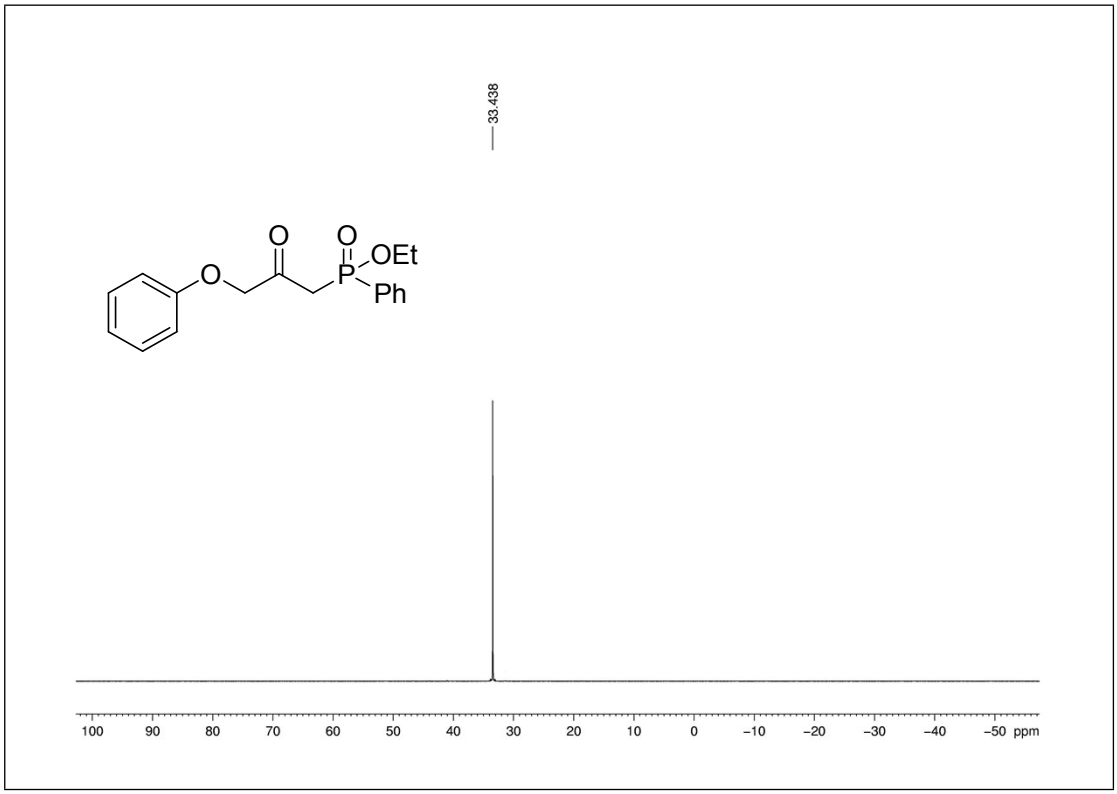

Fig. 79 ^{31}P NMR spectrum of compound **3t**

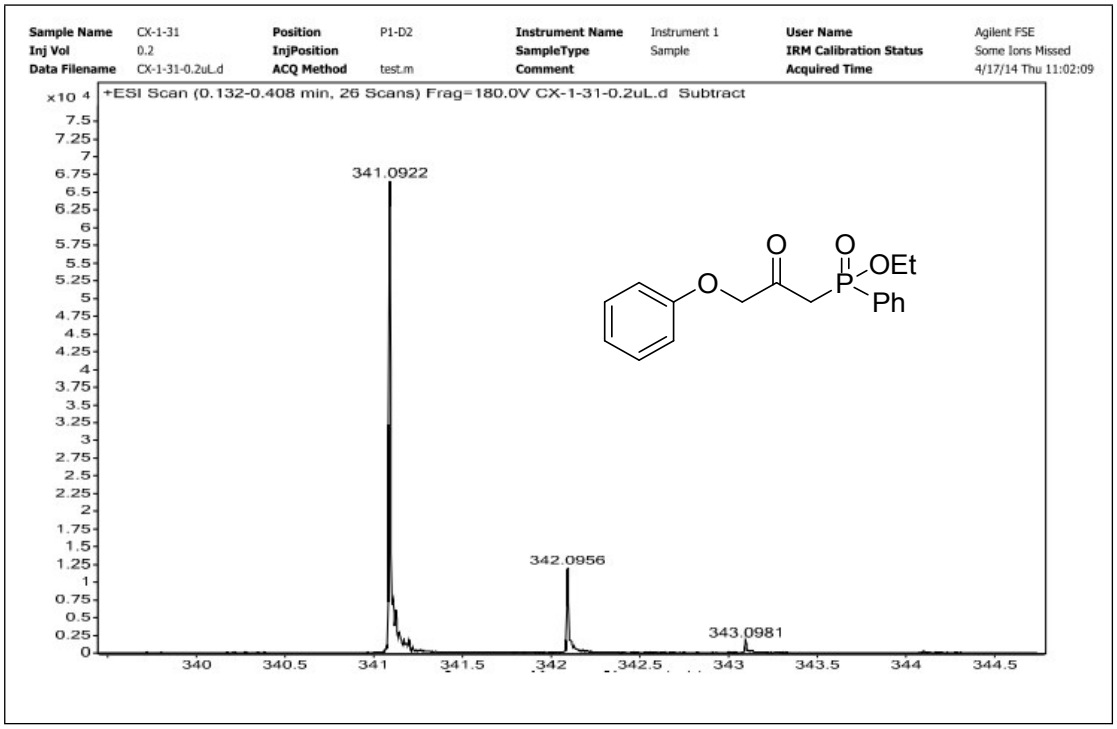

Fig. 80 HRMS spectrum of compound **3t**

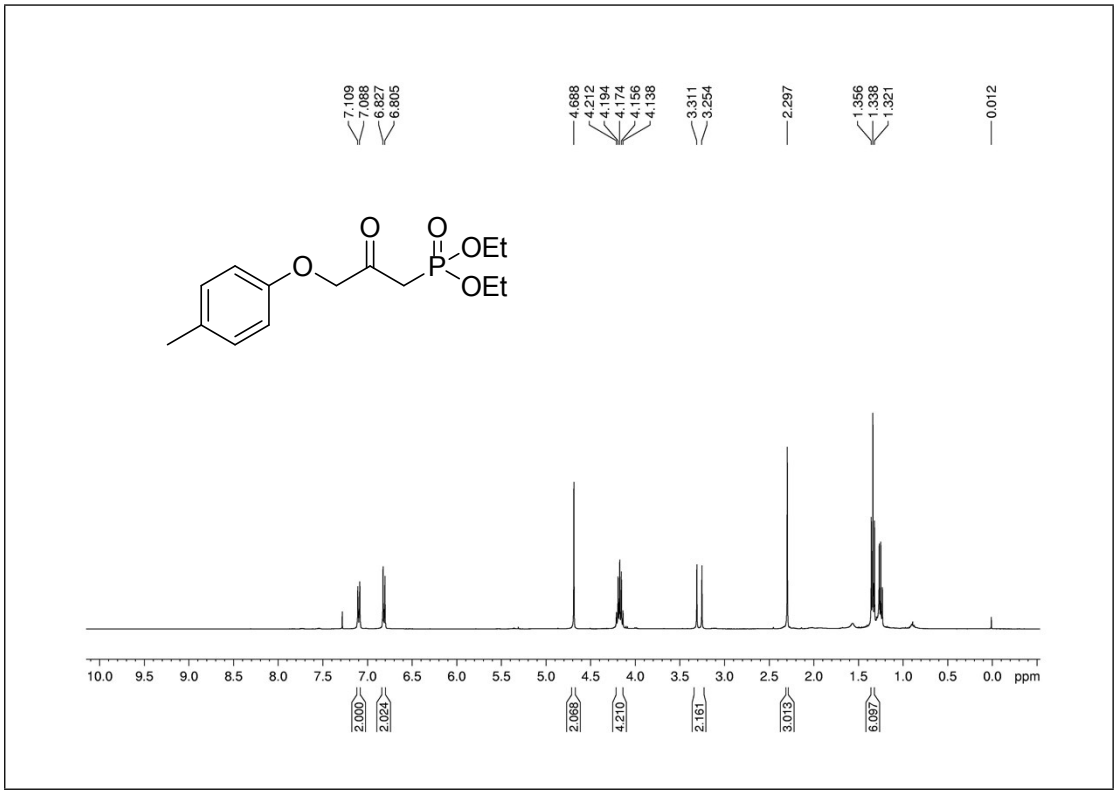

Fig.81 ^{1}H NMR spectrum of compound **3u**

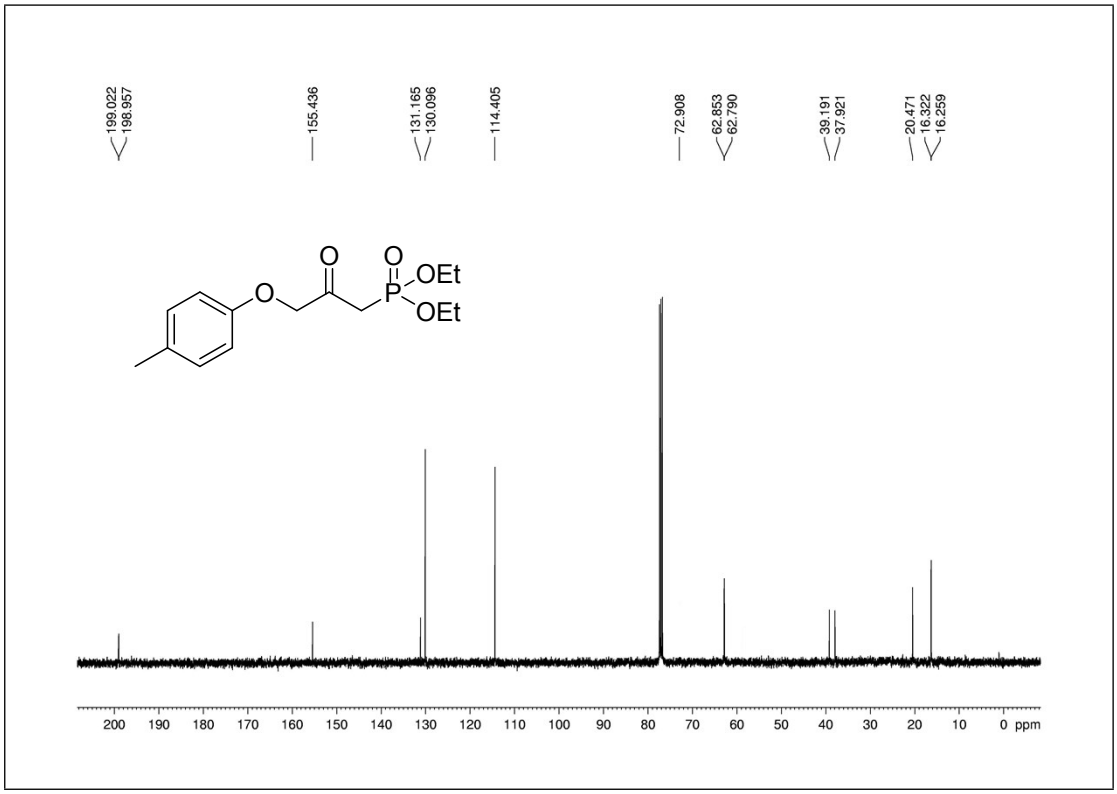

Fig. 82 ^{13}C NMR spectrum of compound **3u**

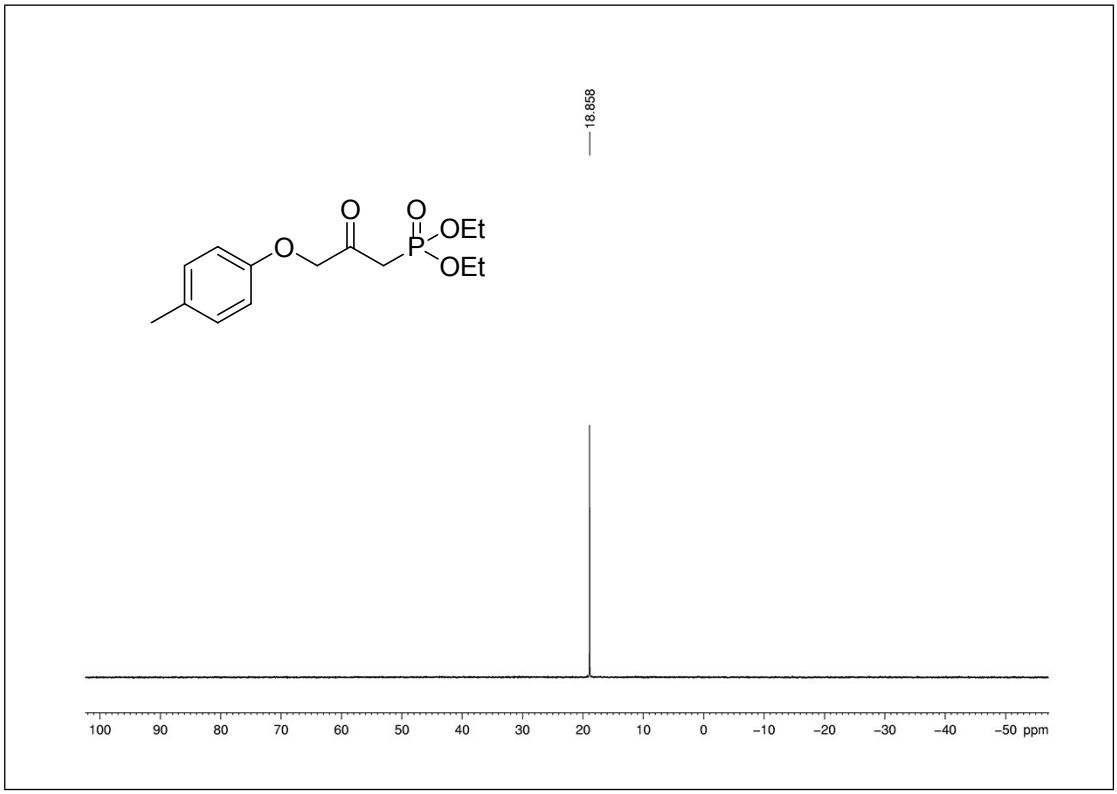

Fig.83 ^{31}P NMR spectrum of compound **3u**

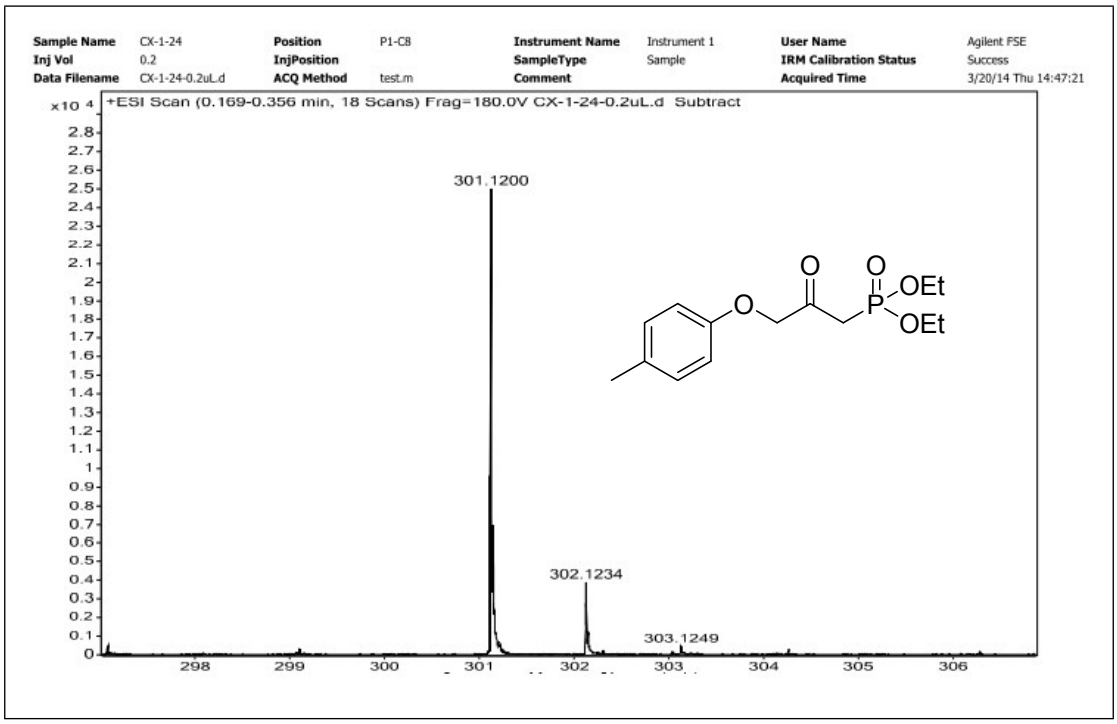

Fig. 84 HRMS spectrum of compound **3u**

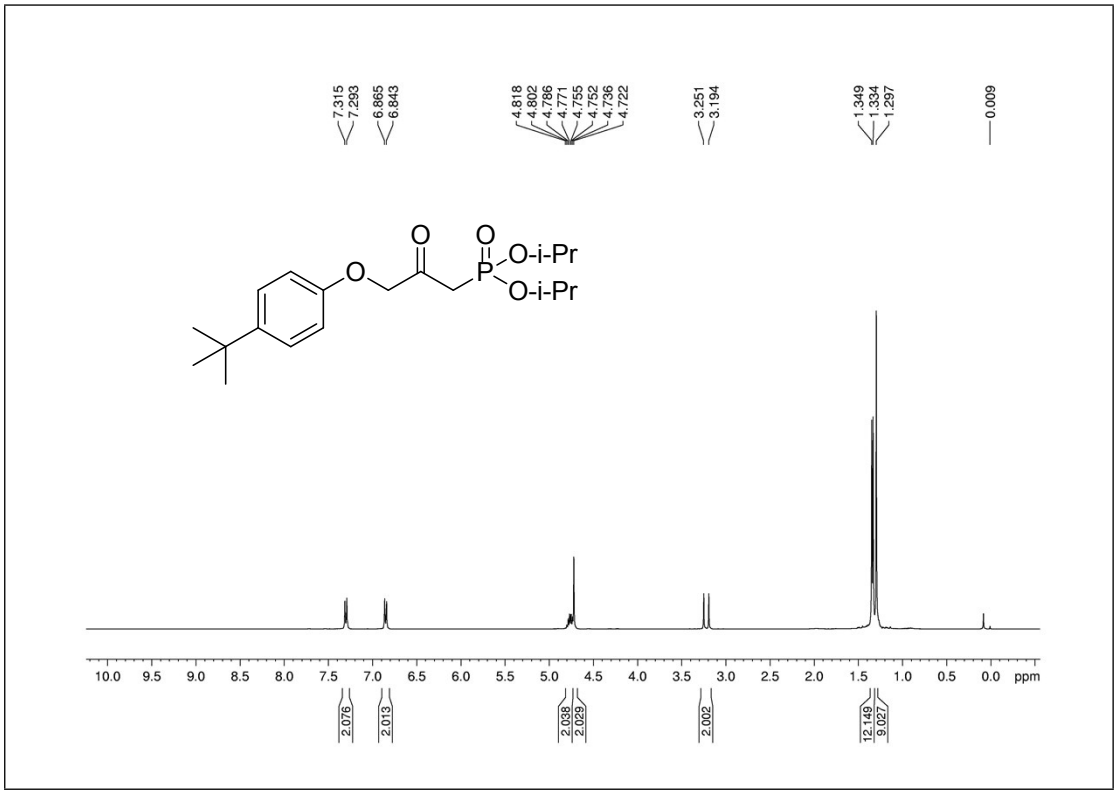

Fig.85 ^{1}H NMR spectrum of compound **3v**

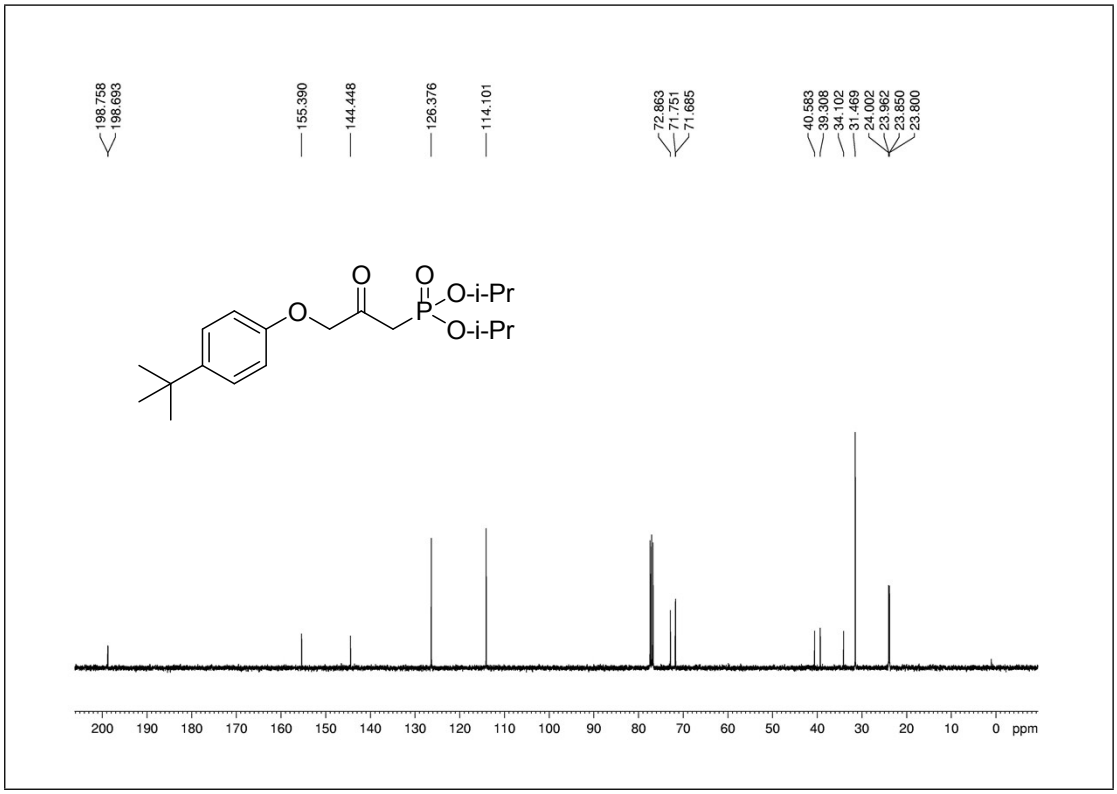

Fig. 86 ^{13}C NMR spectrum of compound **3v**

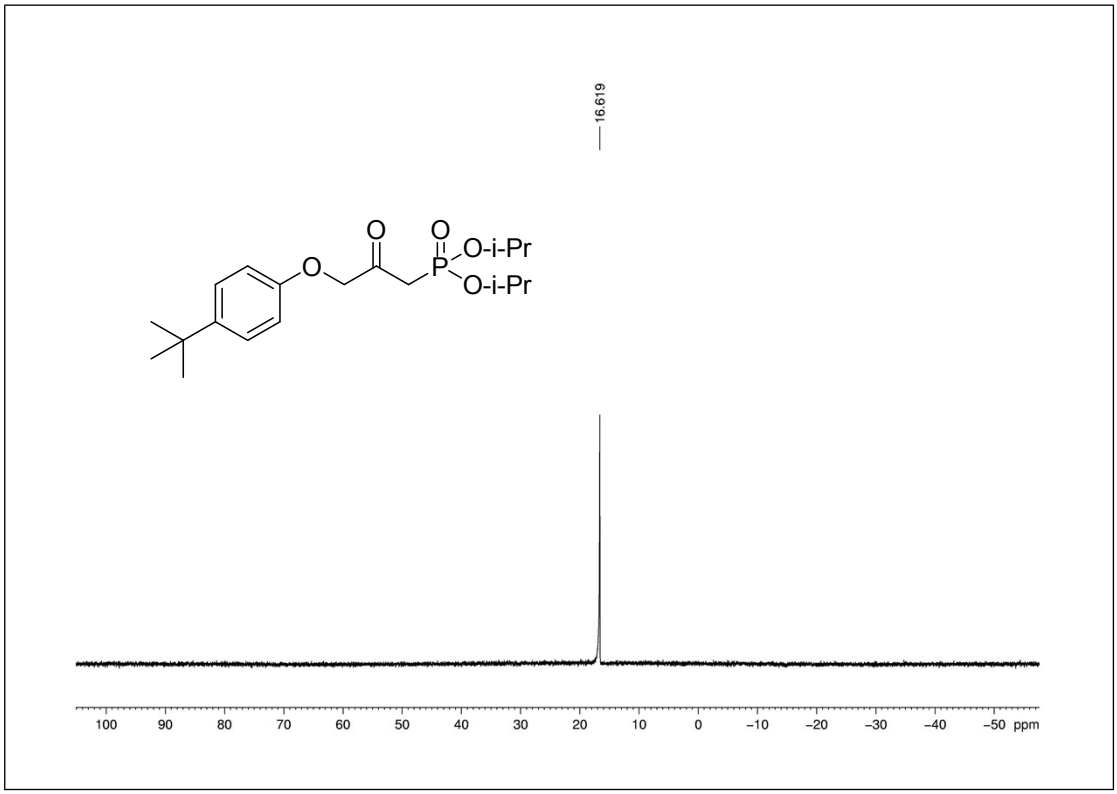

Fig.87 ^{31}P NMR spectrum of compound **3v**

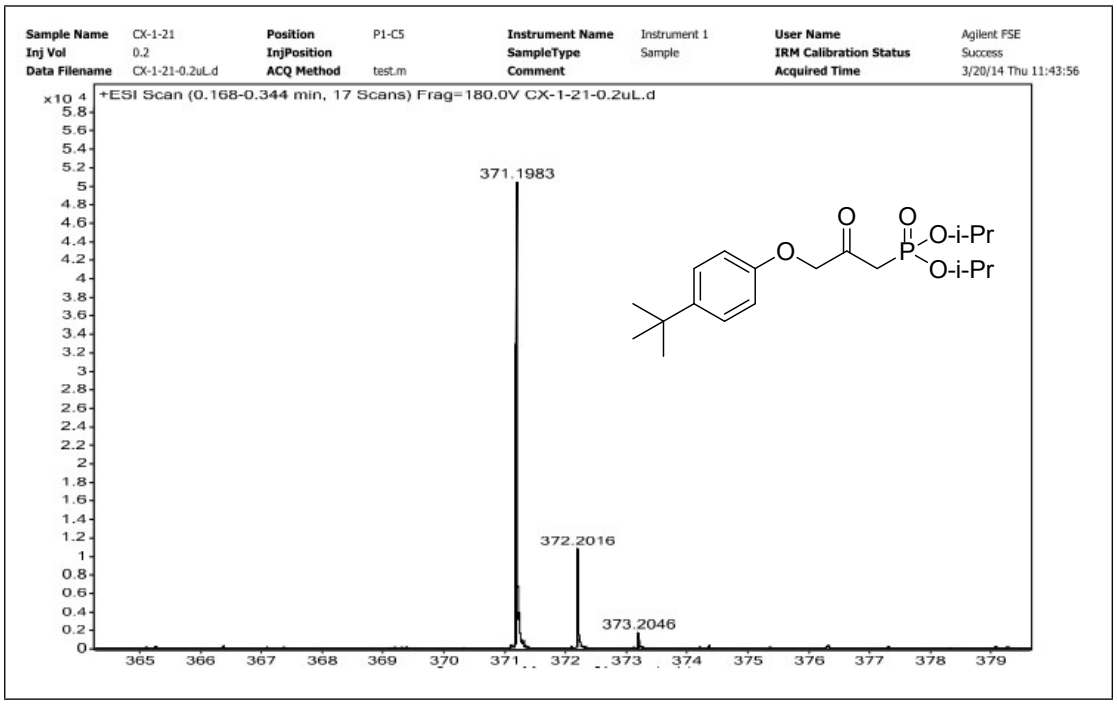

Fig. 88 HRMS spectrum of compound **3v**

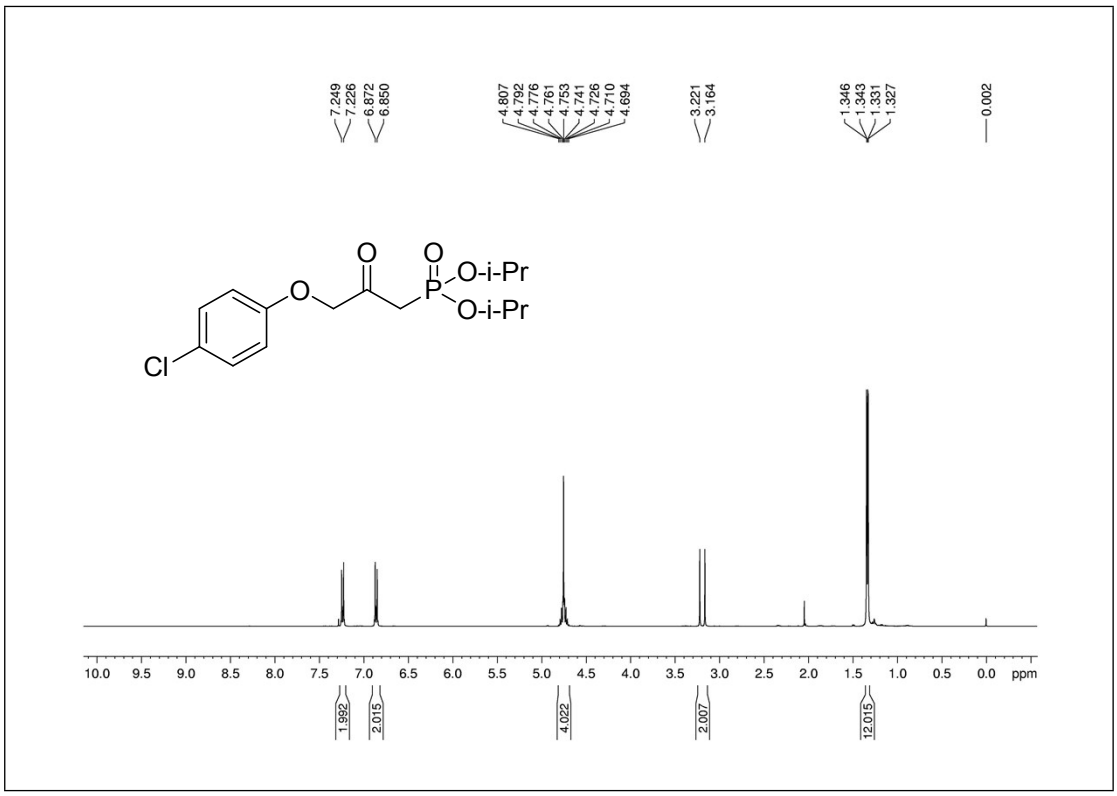

Fig.89 ^{1}H NMR spectrum of compound **3w**

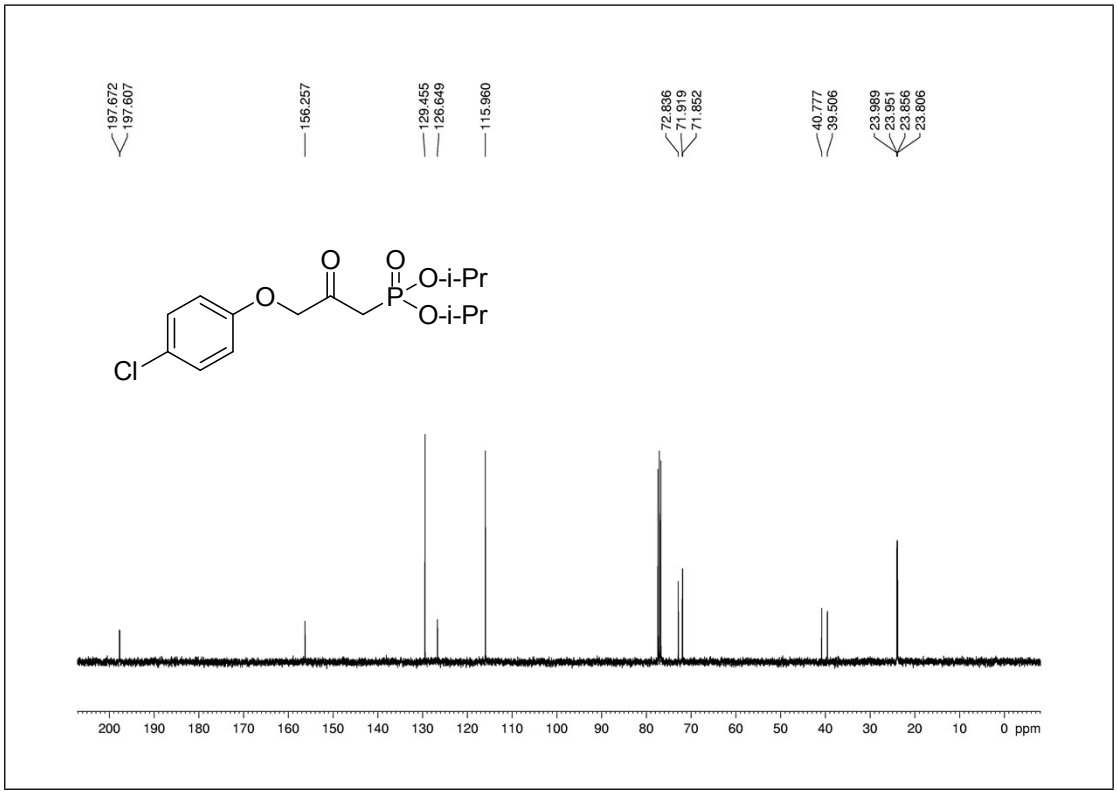

Fig.90 ^{13}C NMR spectrum of compound **3w**

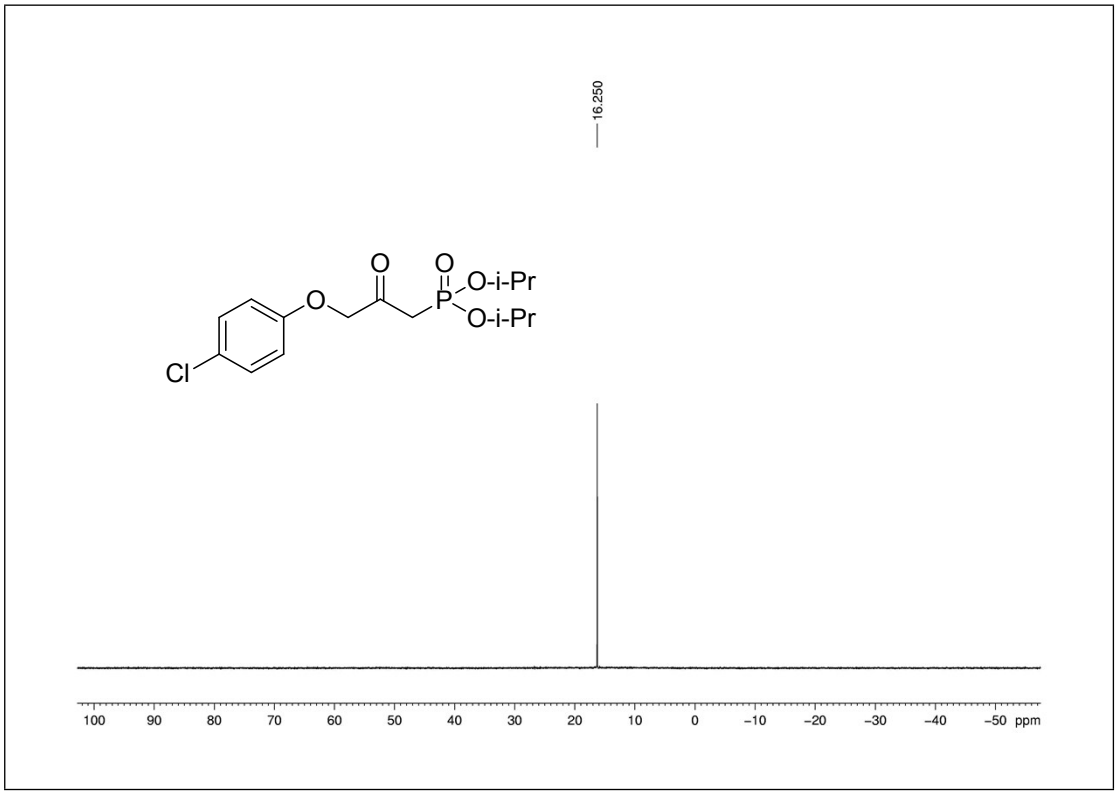

Fig.91 ^{31}P NMR spectrum of compound **3w**

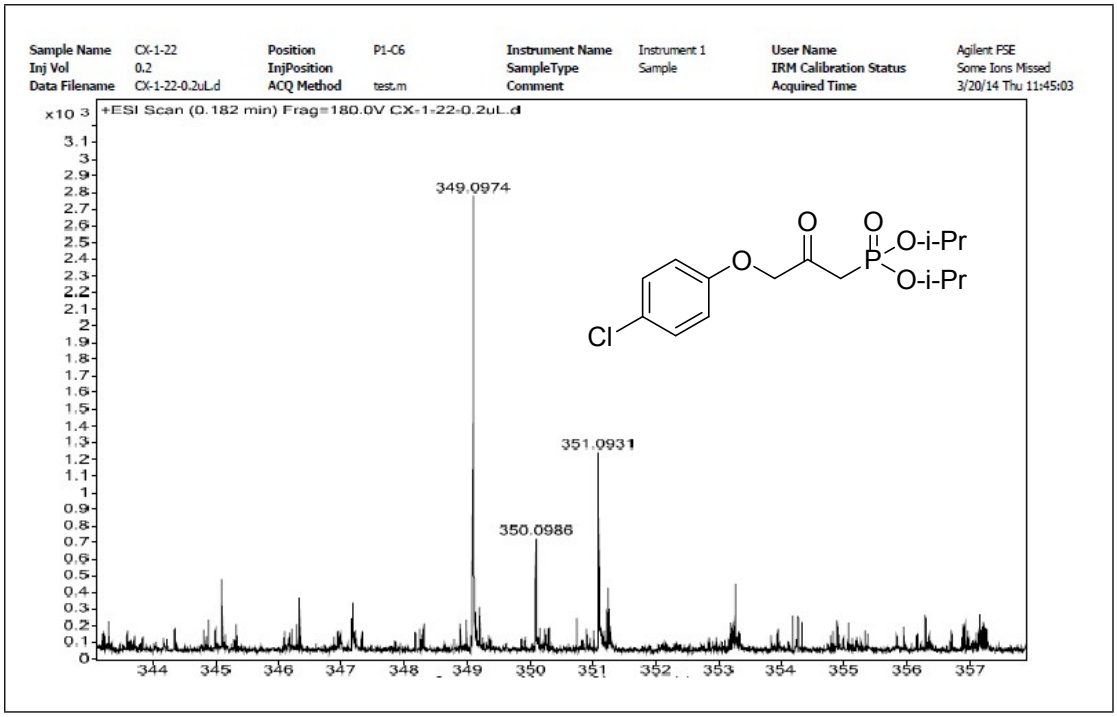

Fig. 92 HRMS spectrum of compound **3w**

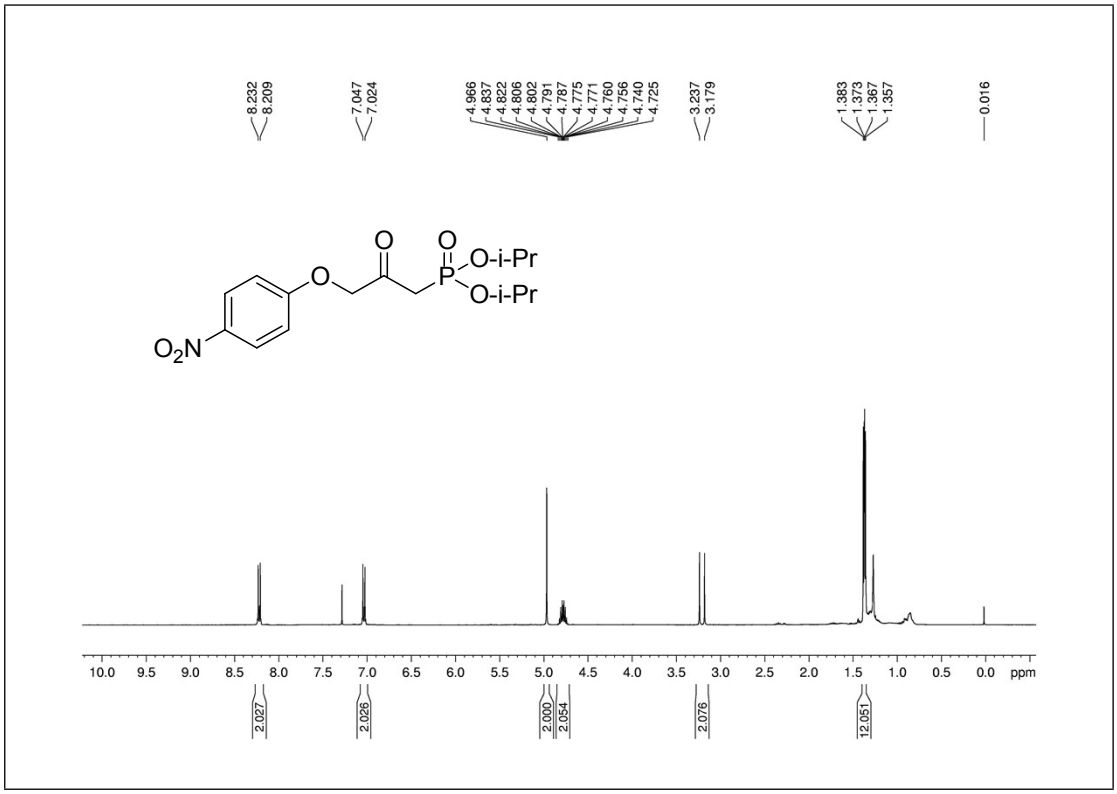

Fig.93 ^{1}H NMR spectrum of compound **3x**

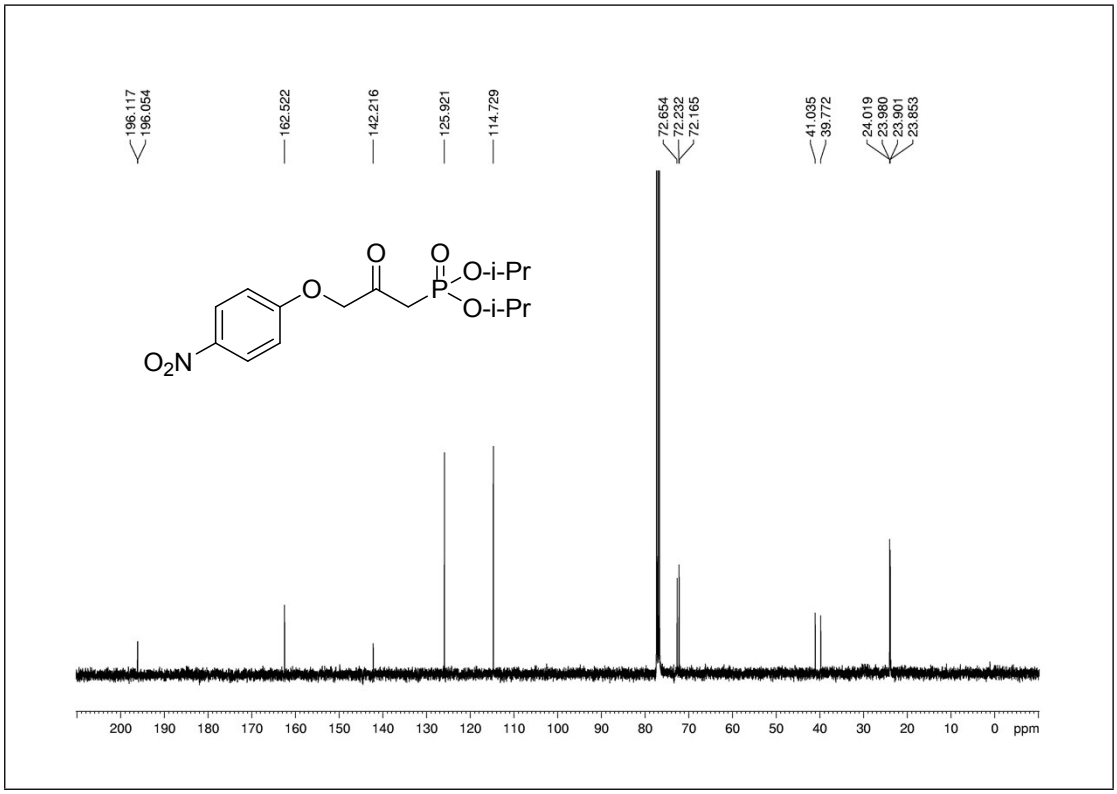

Fig.94 ^{13}C NMR spectrum of compound **3x**

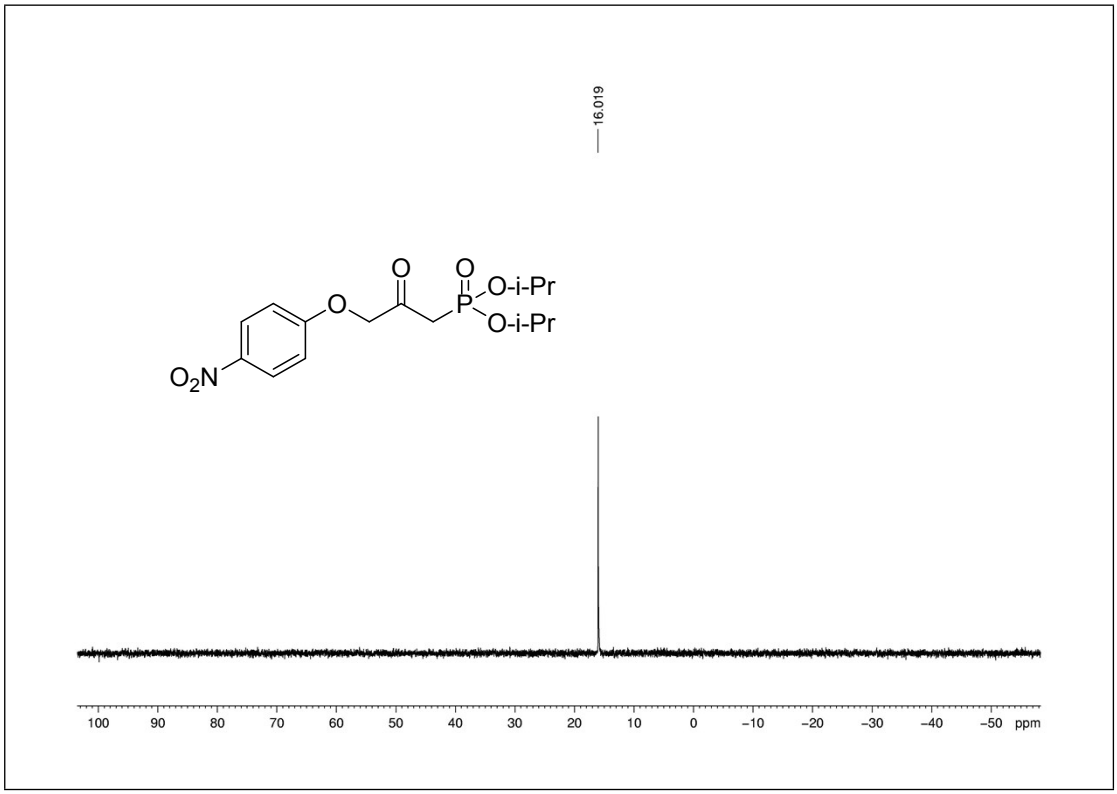

Fig.95 ^{31}P NMR spectrum of compound **3x**

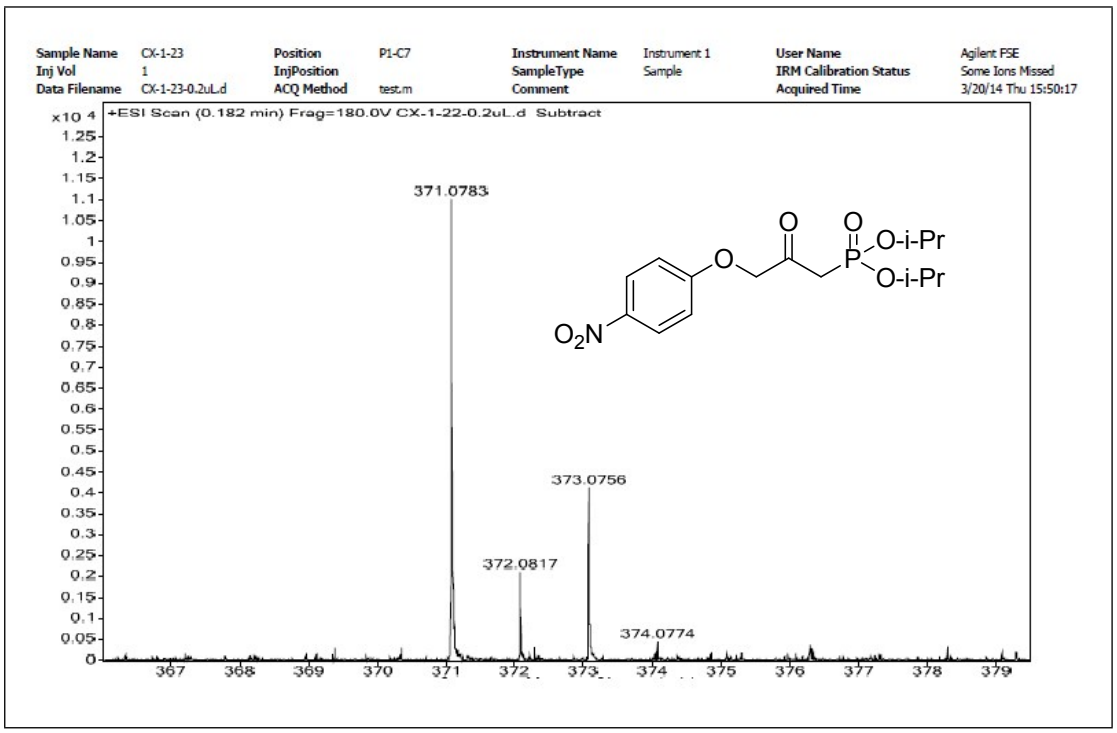

Fig. 96 HRMS spectrum of compound **3x**

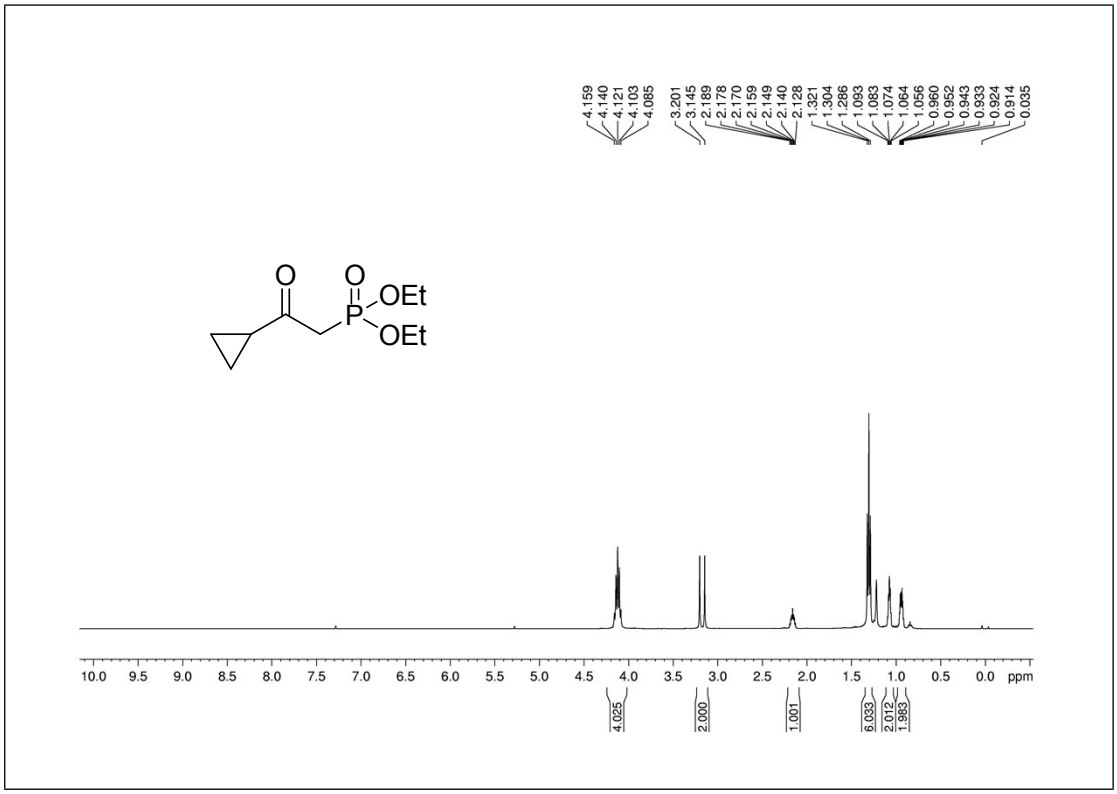

Fig.97 ^{1}H NMR spectrum of compound **3y**

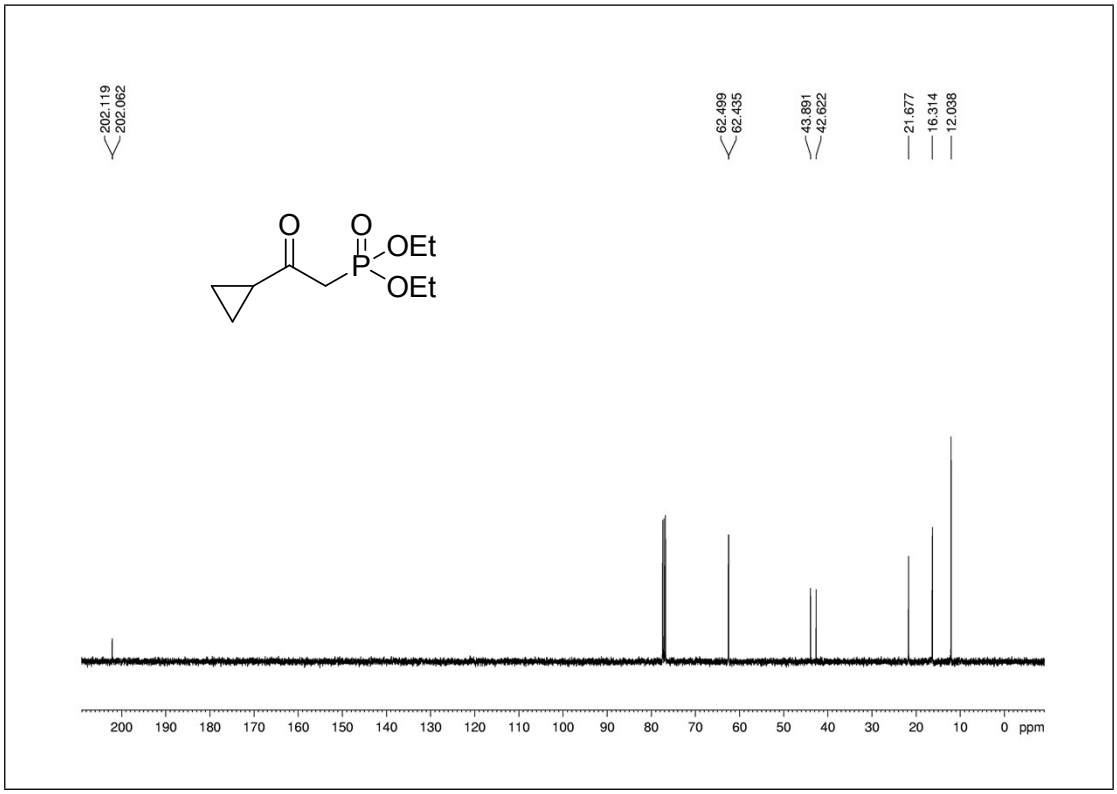

Fig.98 ^{13}C NMR spectrum of compound **3y**

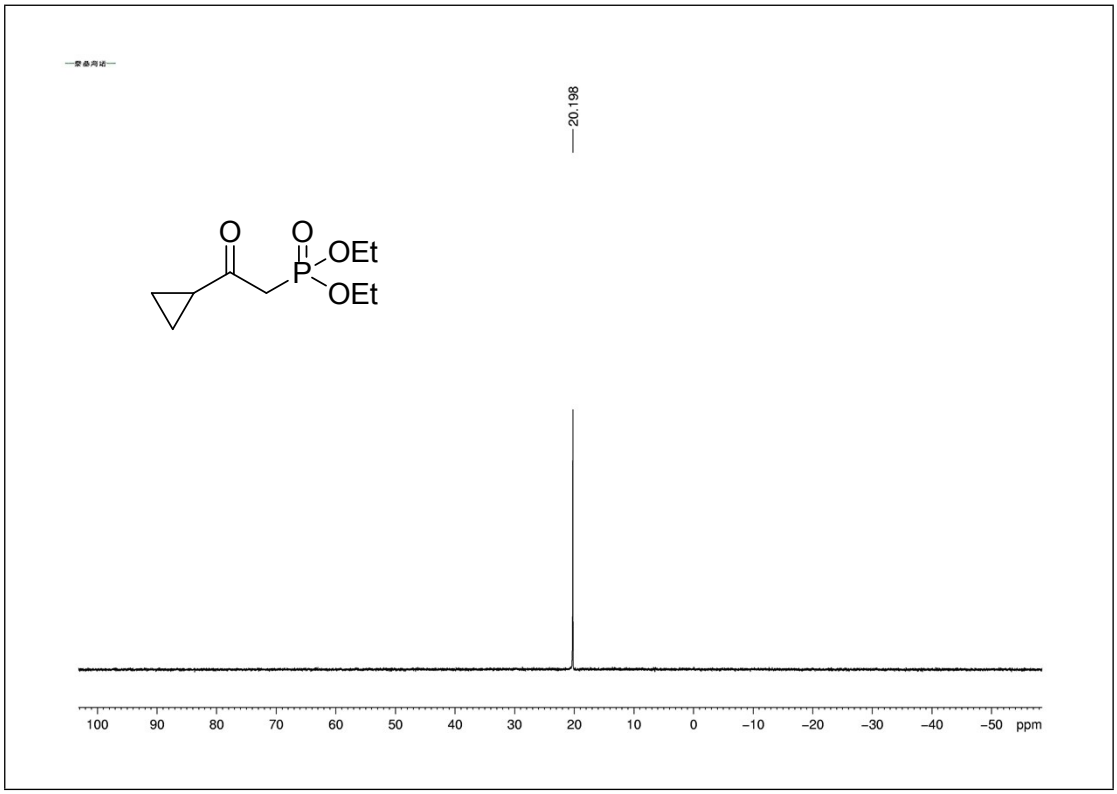

Fig.99 ^{31}P NMR spectrum of compound **3y**

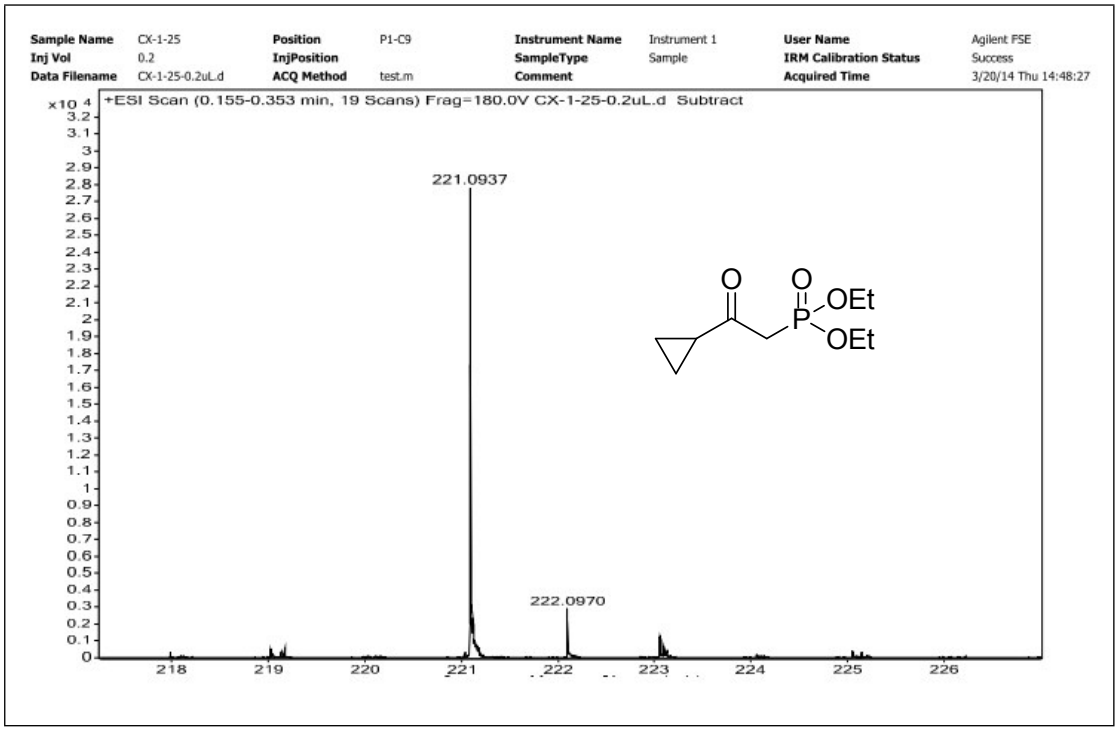

Fig. 100 HRMS spectrum of compound **3y**

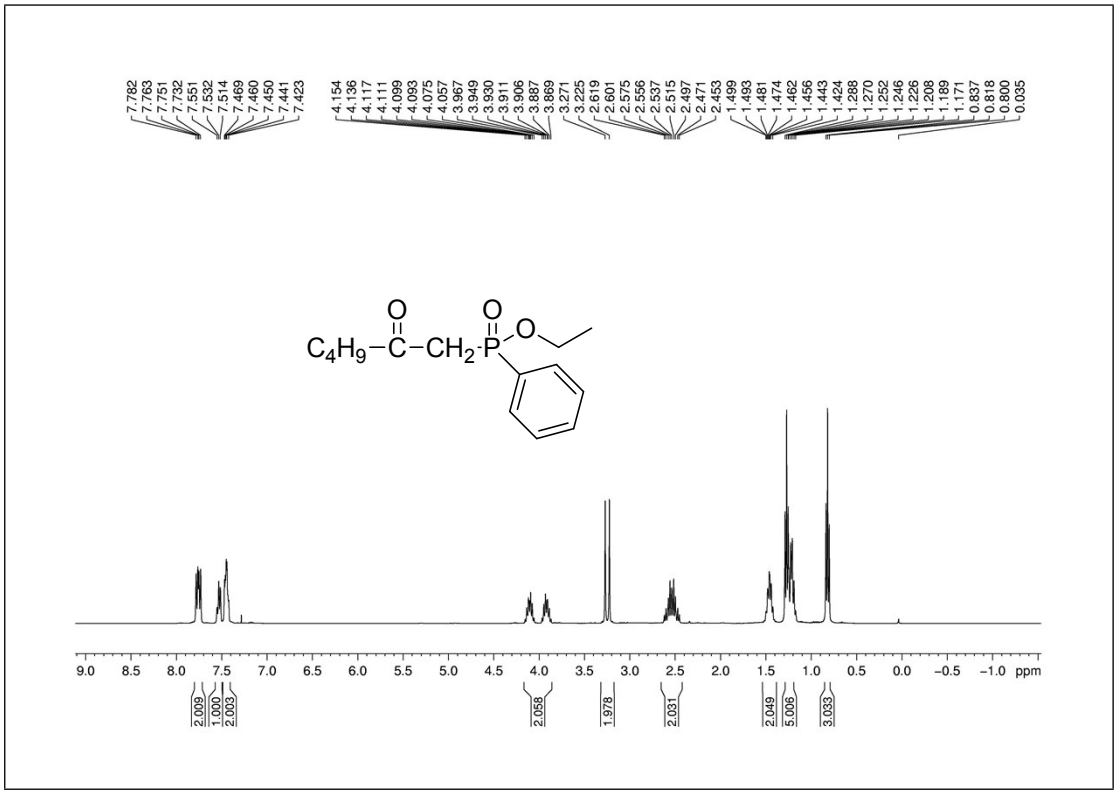

Fig.101 ^{1}H NMR spectrum of compound **3z**

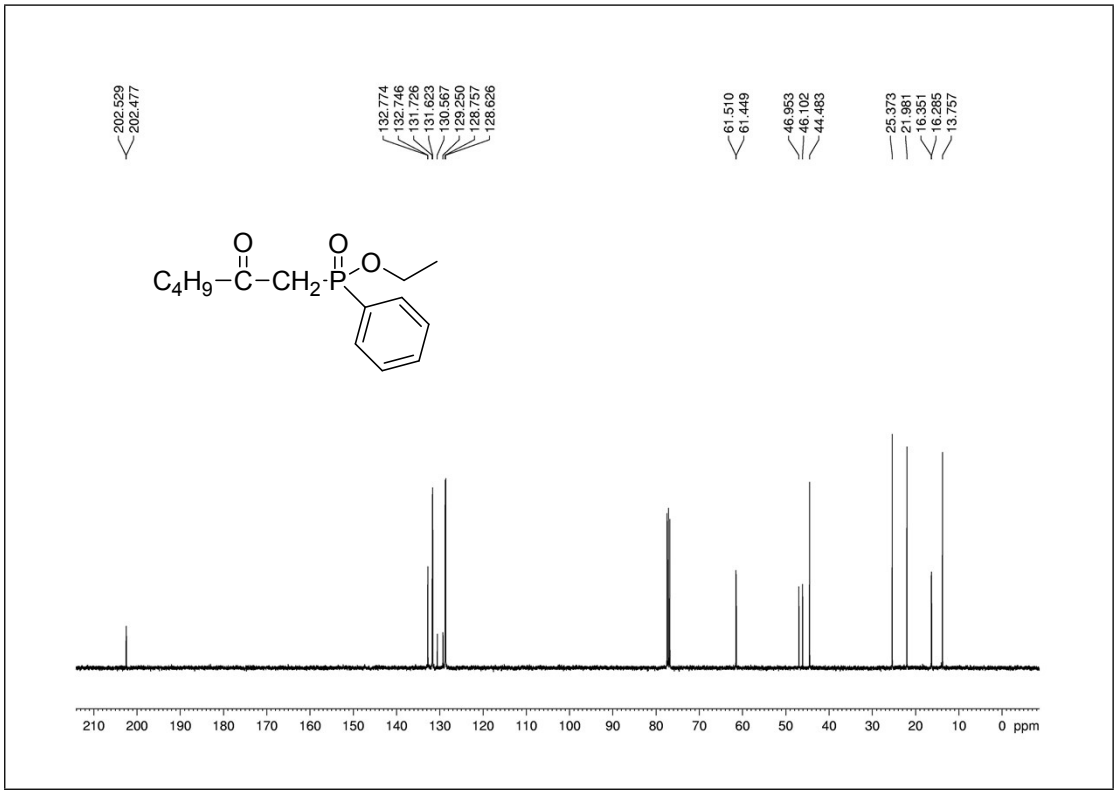

Fig.102 ^{13}C NMR spectrum of compound **3z**

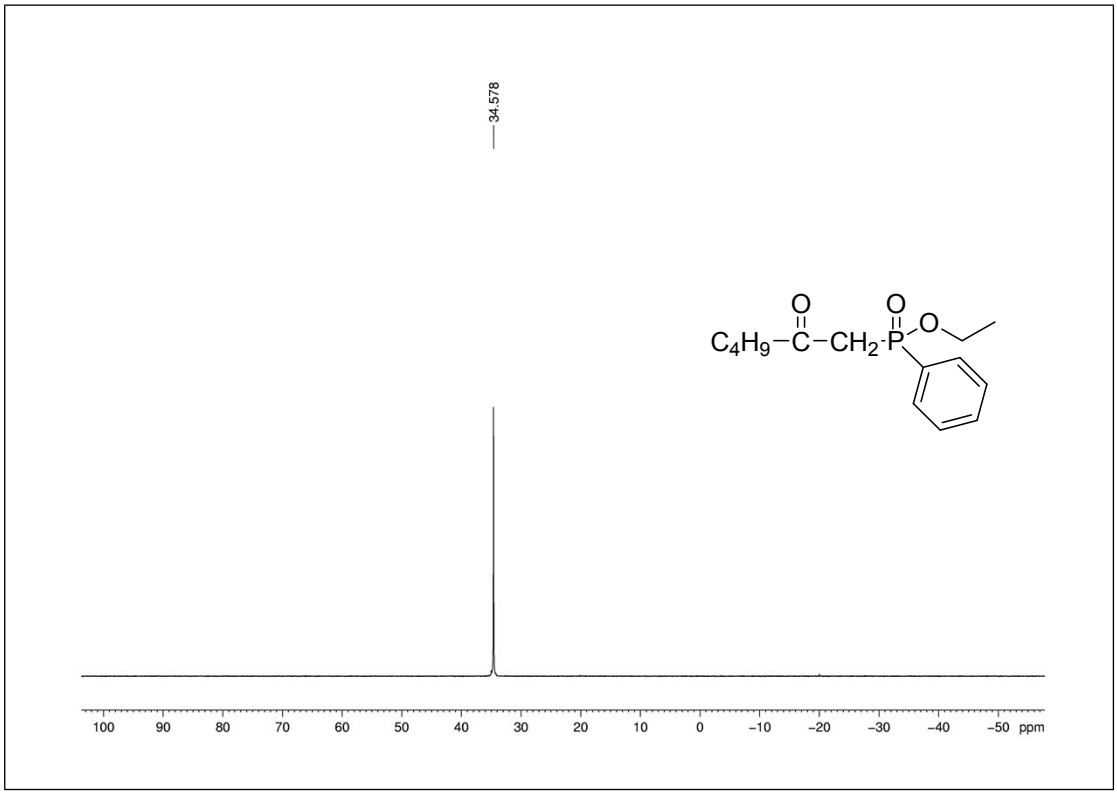

Fig.103 ^{31}P NMR spectrum of compound **3z**

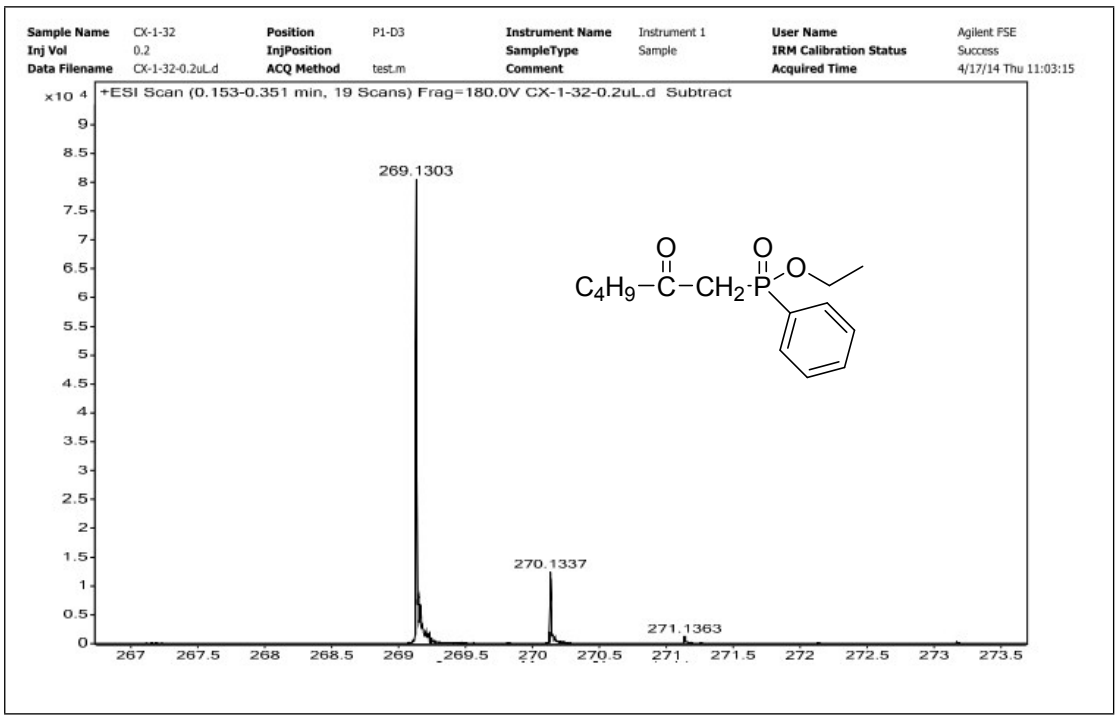

Fig. 104 HRMS spectrum of compound **3z**

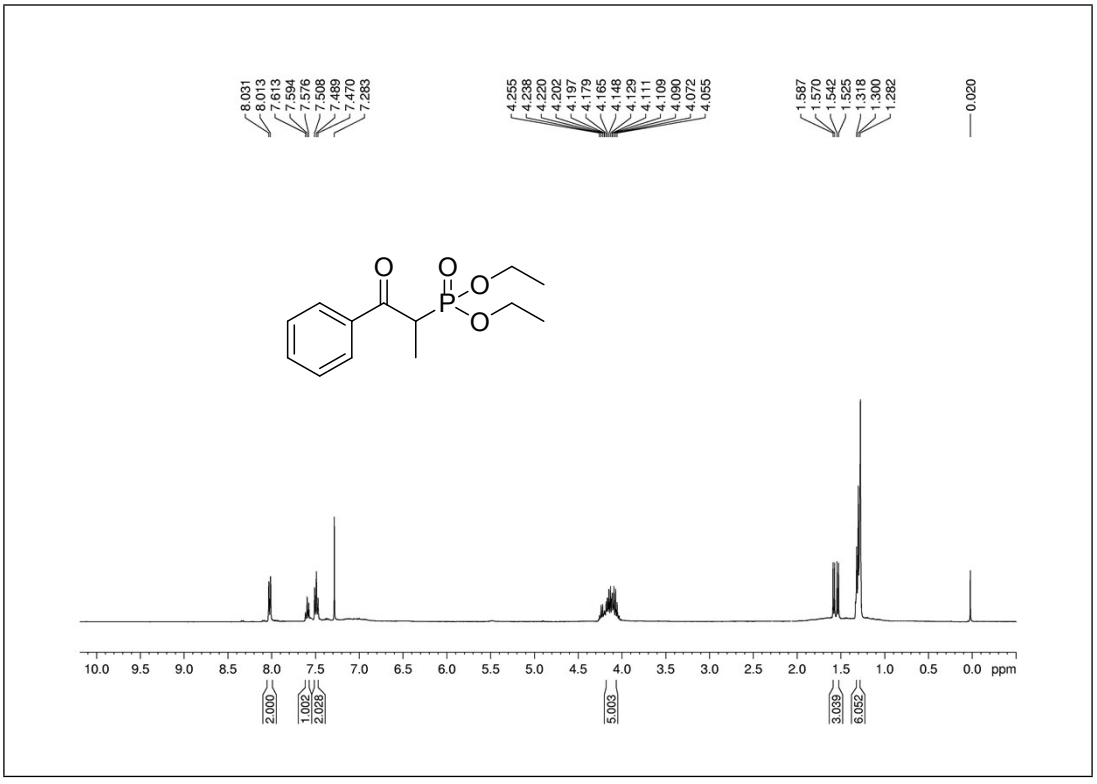

Fig.105 ^{1}H NMR spectrum of compound **3aa**

Fig.106 ^{13}C NMR spectrum of compound **3z**

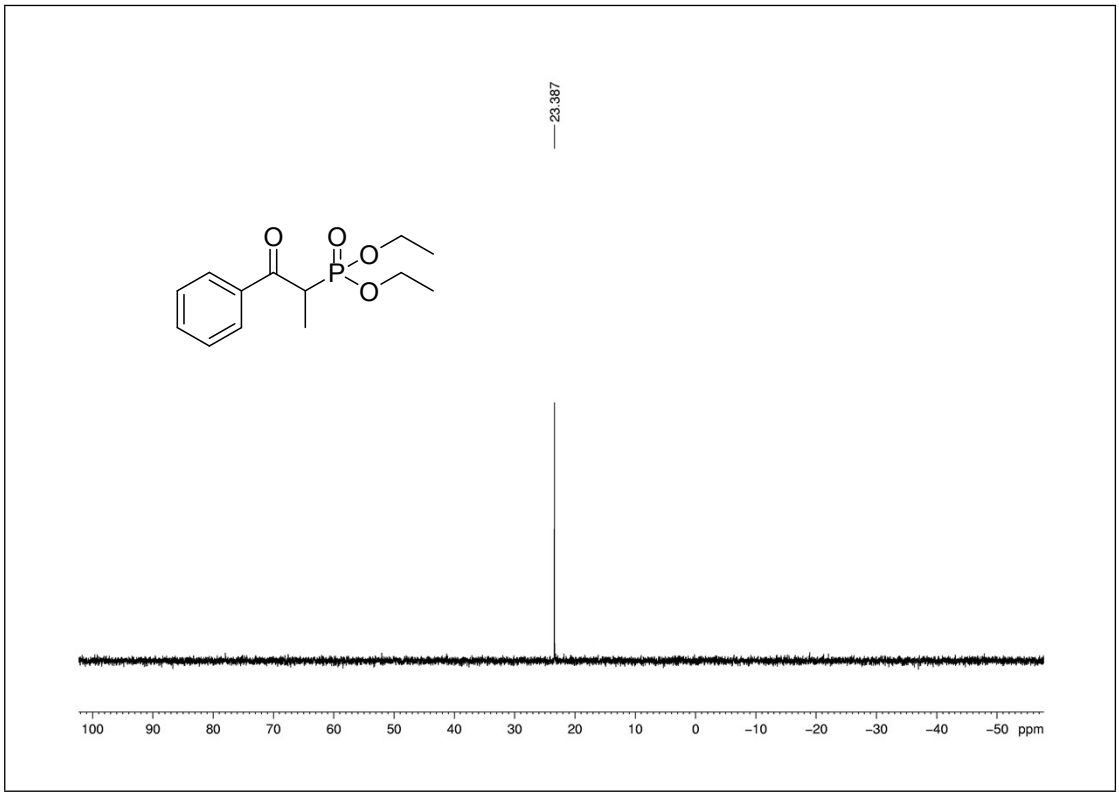

Fig.107 ^{31}P NMR spectrum of compound **3z**

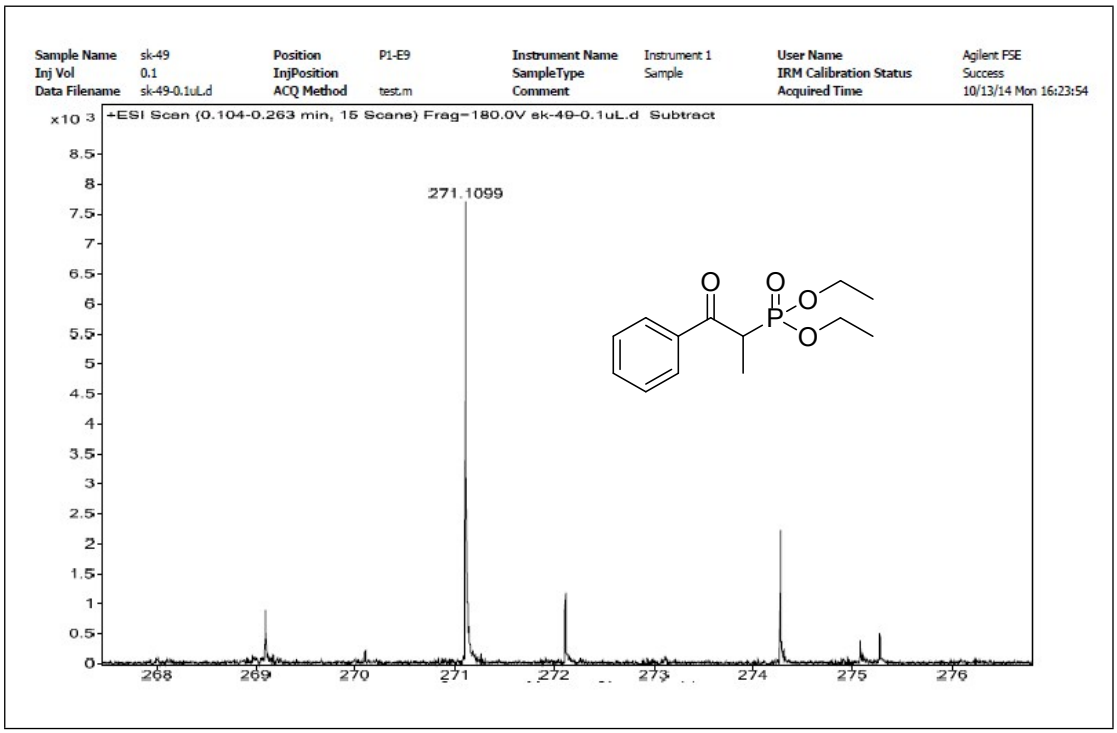

Fig. 108 HRMS spectrum of compound **3aa**