Supporting Information

Rhodium-Catalyzed enantioselective transfer hydrogenation of *α*-chloro *β*-ketophosphonates via dynamic kinetic resolution

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I. General remark

All reactions and manipulations were performed in an argon-filled glovebox or using standard Schlenk techniques. Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. Column Chromatography was performed with silica gel Merck 60 (200-300 mesh). Anhydrous solvents. Anhydrous solvents were purchased from J&K Chemical and degassed by bubbling argon over a period of 30 min prior to use. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded on a Bruker Avance Neo 400 MHz or 600 MHz spectrometer with $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in ppm, upfield to TMS (0.00 ppm) and relative to $CDCl_3$ (7.26 ppm) for ^{1}H NMR or $CDCl_3$ (77.0 ppm) for ^{13}C NMR. HRMS(ESI) were recorded on Thermo-Fisher Orbitrap Fusion instrument. Optical rotations were determined using a 1 mL cell with a 1 dm path length on a Rudolph Autopol I polarimeter at 589 nm. HPLC analyses were performed using an Agilent 1260 Series instrument.

II. Condition optimization of dynamic kinetic resolution

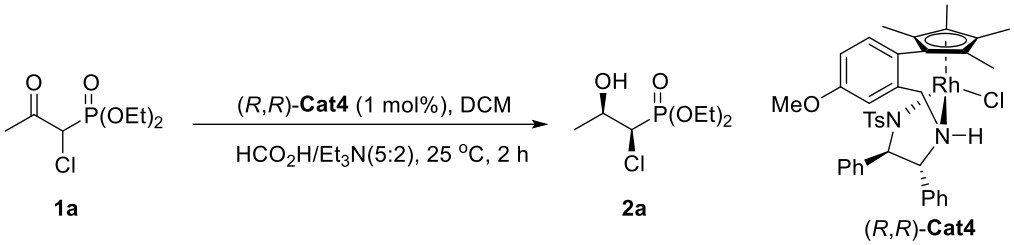
General procedure:

To a 2.5 mL vial was added the model substrates (0.1 mmol), catalyst (0.001 mmol), Et_3N/HCOOH mixture and solvent (1 mL) under argon atmosphere. The mixture was stirred for 2 hours at room temperature. After the reaction finished, the reaction mixture was passed through a short column of silica gel to remove the metal complex. The product was concentrated *in vacuo* then 0.4 mL pyridine and 0.2 mmol benzoyl chloride was added. The mixture was allowed to stir at room temperature for 4 hours. After the reaction finished, 4 M HCl solution was add, the mixture was quenched with EtOAc and concentrated *in vacuo*.[1] The product was analyzed by ^{1}H NMR to determine the conversion. The ee values were determined by HPLC on a chiral stationary phase.

Optimization of catalysts:

Four catalysts applied on transfer hydrogenation were optimized for the model substrate with general procedure. In all groups substrate had completely converted, while **Cat4** gave the highest dr and ee result. Therefore, **Cat4** will be applied on further optimization. After determined the catalyst with highest activity and enantioselectivity, the effect of solvent on this reaction was tested. The results showed that solvent affect few on this reaction, and DCM gave better performance than other groups. The concentration of reaction and hydrogen donor were further studied. The results show that the concentration and the ratio of hydrogen donor also had no effect on both reactivity and selectivity. **Cat4** were synthesized according to the literature.[2]

Table S1. Optimization of reaction concentration:

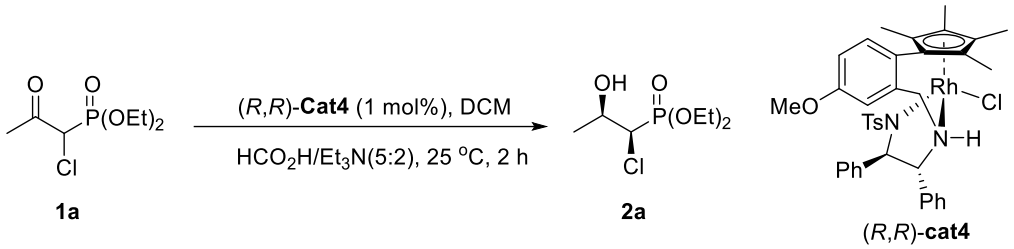


1a **2a** (*R*,*R*)-**Cat4**

entry[a]	concentration (mol/L)	conv. (%)[b]	dr[c]	ee[d]
1	0.1	>99	>99:1	98%
2	0.16	>99	>99:1	98%
3	0.25	>99	>99:1	98%
4	0.5	>99	>99:1	98%

[a]Reaction conditions: **1a** (0.1 mmol), HCOOH/Et_3N (5:2) (0.05 mL), catalyst. (1 mol%), DCM (0.2-1 mL), 25 °C, 2 h. [b]NMR yield. [b]The reaction mixture was analyzed by ^1H. [c]Determined by NMR [d]Determined by chiral HPLC analysis.

Table S2. Optimization of hydrogen donor:

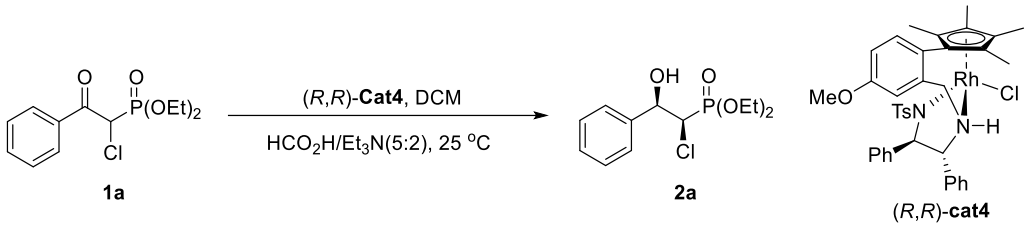


1a **2a** (*R*,*R*)-**cat4**

entry[a]	hydrogen donor	conv. (%)[b]	dr[c]	ee [d]
1	HCOOH/Et_3N (5:2)	>99	>99:1	98%
2	HCOOH/Et_3N (1:1)	>99	>99:1	98%
3	HCOOH/Et_3N (2:1)	>99	>99:1	98%
4	HCOOH/DBU (2:1)	>99	>99:1	95%

[a]Reaction conditions: **1a** (0.1 mmol), hydrogen donor (0.05 mL), catalyst. (1 mol%), DCM (0.2 mL), 25 °C, 2 h. [b]NMR yield. [b]The reaction mixture was analyzed by ^1H. [c]Determined by NMR [d]Determined by chiral HPLC analysis.

Table S3. TON Study of DKR-ATH of 1h:



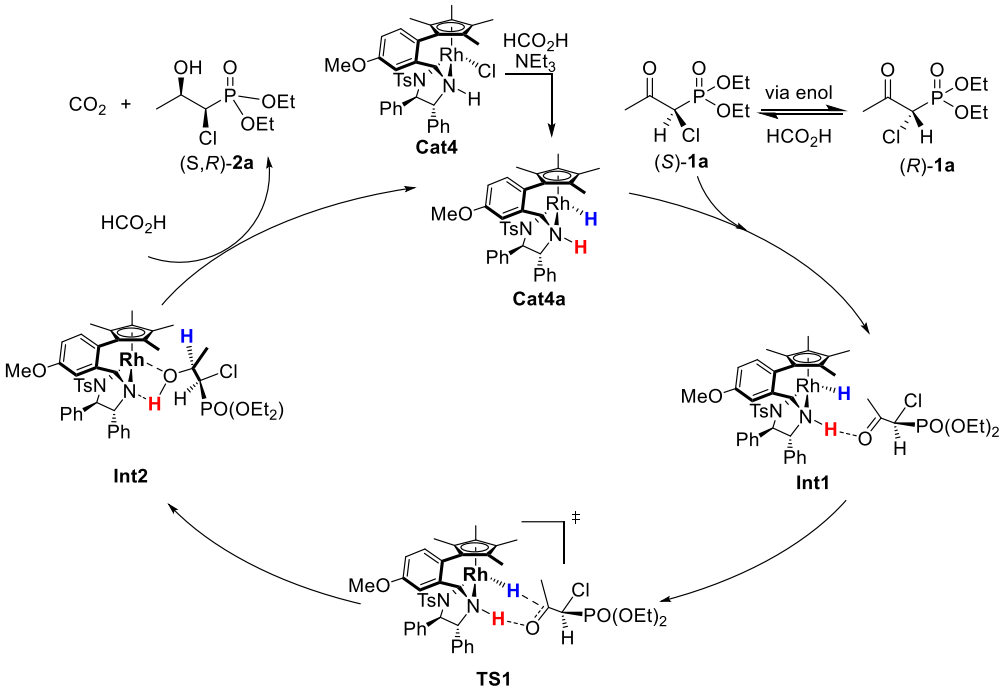
entry[a]	S/C	time (h)	conv. (%)[b]	dr[c]	ee (*cis*)[d]
1	200	2	>99	>99:1	>99%
2	500	5	>99	>99:1	>99%
3	1000	12	>99	>99:1	>99%
4	2000	36	>99	>99:1	>99%

The reactions were carried out with different catalyst/substrate (0.2 mmol) ratio in 0.2 mL of solvent, HCO_2H/Et_3N azeotropic mixture (100 μL) at 25 °C.

Gram-Scale DKR-ATH Procedure:

In a nitrogen filled glovebox, to a 50 mL Schleck tube charged with a magnetic stirring bar were added successively substrate **1a** (5 mmol, 1.15 g), formic acid/trimethylamine azeotropic mixture (5:2, 0.4 mL), the catalyst (**cat4**, 7.6 mg, 0.01 mmol) and the solvent (10 mL). The mixture was then stirred at room temperature for 14 h. After the reaction finished, the reaction solution was concentrated and the residue was passed through a short column of silica gel (eluent: EA:PE = 3:1) to remove the metal complex. Compounds **2a** was obtained as a colorless oil (1.04 g, 91% yield, 97% ee, >99:1 dr).

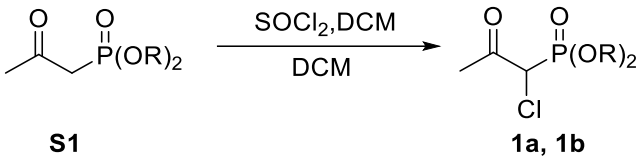
III. Proposed Catalytic Circle



Based on our previous investigations, a plausible catalytic circle was proposed. Firstly, **Cat4** was transformed to the reactive species **Cat4a** in the presence of HCO_2H and NEt_3. The two enantiomers of **1a**, (*S*)-**1a** and (*R*)-**1a**, are in equilibrium with each other. (*S*)-**1a** matches with the catalyst **Cat4a** better and it coordinate with **Cat4a** to form **Int1**. Intramolecular hydrogen transfer of **Int1** generated **Int2** via **TS1**. The desired product (*S*,*R*)-**2a** was formed by reaction of **Int2** with HCO_2H, and the reactive catalyst **Cat4a** was also regenerated in this process.

IV. Preparation of substrates

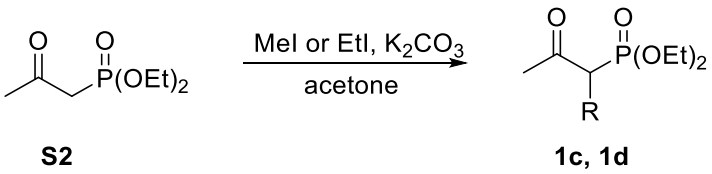
Procedure A: Preparation of substrate 1a, 1b.



Scheme 1. Synthetic route of compound **1a, 1b**

To a solution of phosphonate **S1** (3 mmol) in anhydrous CH_2Cl_2 (5 mL) under Ar atmosphere at 0 °C was added $SOCl_2$ (3 mmol) dropwise. After stirring for 1 hour, water was added to quench the reaction. The resulting mixture was extracted with CH_2Cl_2 and concentrated *in vacuo*. The residue was purified by flash chromatography (PE:EA 3:1) to afford product. Compound **1a, 1b** was obtained with medium to high yield (80%).

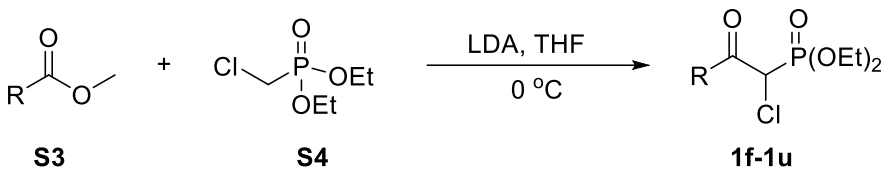
Procedure B: Preparation of substrate 1c, 1d.



Scheme 2. Synthetic route of compound **1c, 1d**

To a solution of phosphonate **S2** (2 mmol) and K_2CO_3 (3 mmol) in acetone (8 mL) was added methyl iodide/ethyl iodide (3 mmol) dropwise, and the resulting mixture was stirred at room temperature for 24 hours. The reaction was quenched with saturated NH_4Cl and extracted with EtOAc. The combined organic phases were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by chromatography to afford product. Compound was obtained with medium yield (50-60%).

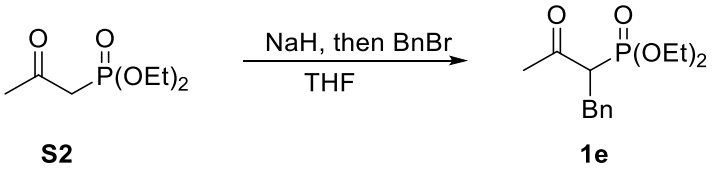
Procedure C: Preparation of substrate 1f-1u.



Scheme 3. Synthetic route of compound **1f-1u**

To a solution of ester **S3** (2.2 mmol) and phosphonate **S4** (2 mmol) in anhydrous THF (2.5 mL) under Ar atmosphere at 0 °C was added LDA (4.4 mmol) dropwise. After stirring for 0.5-hour, 4 M HCl solution was added to quench the reaction. The resulting mixture was extracted with EtOAc and concentrated *in vacuo*. The residue was purified by flash chromatography (PE:EA from 10:1 to 3:1) to afford product. Compound **1f-1u** was obtained in 60-85% yield.

Procedure D: Preparation of substrate 1e.

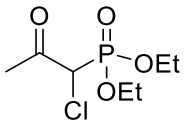


Scheme 4. Synthetic route of compound **1e**

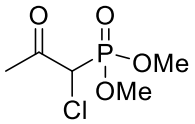
The substrate **S3** (5.0 mmol, 1.0 eq) was added to a round-bottom flask under an argon atmosphere, followed by the addition of 10 mL of THF as the solvent. The mixture was cooled to 0°C using an ice-water bath and NaH (6.0 mmol, 1.2 eq, 60% dispersion in mineral oil) was added portion-wise. The reaction mixture was stirred at 0°C for 30 minutes until gas evolution ceased. Next, benzyl bromide (6.0 mmol, 1.2 eq) was added dropwise. The mixture was stirred at 0°C for 30 minutes before allowing it to warm gradually to room temperature. Stirring was continued overnight. The reaction was quenched with a saturated NH_4Cl solution, extracted with EA and washed with water and brine. The organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography

to afford product (65% yield).

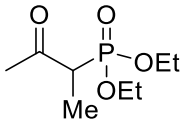
Characterization Data of 1



Diethyl (1-chloro-2-methyl-2-oxoethyl)phosphonate (1a):[3] colorless liquid, 552 mg, 80% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.48 (dd, *J* = 17.5, 1.0 Hz, 1H), 4.30 – 4.16 (m, 4H), 2.45 (d, *J* = 1.6 Hz, 3H), 1.36 (tdd, *J* = 7.2, 3.5, 1.5 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 197.67, 64.61 (dd, *J* = 14.4, 6.9 Hz), 57.46 (d, *J* = 138.7 Hz), 27.77, 16.31 (d, *J* = 5.9 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.47. HRMS Calcd. For C7H14ClNaO4P+ [M+Na+]: 251.0210. Found: 251.0213.

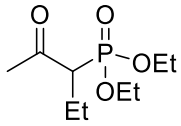


Dimethyl (1-chloro-2-methyl-2-oxoethyl)phosphonate (1b):[3] colorless liquid, 485 mg, 80% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.54 (d, *J* = 17.6 Hz, 1H), 3.90 (ddd, *J* = 11.1, 2.6, 1.1 Hz, 6H), 2.47 (d, *J* = 2.5 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 196.54, 55.83 (d, *J* = 139.9 Hz), 53.88 (dd, *J* = 22.1, 6.9 Hz), 26.81. **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 14.93. HRMS Calcd. For C5H10ClNaO4P+ [M+Na+]: 222.9897. Found: 222.9897.

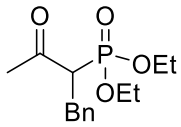


Diethyl (1-methyl-2-methyl-2-oxoethyl)phosphonate (1c):[3] colorless liquid, 311 mg, 57% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.28 – 4.02 (m, 4H), 3.21 (dq, *J* = 25.6, 7.1 Hz, 1H), 2.34 (s, 3H), 1.43 – 1.28 (m, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 203.91 (d, *J* = 3.9 Hz), 62.61 (dd, *J* = 10.5, 6.9 Hz), 47.50 (d, *J* = 126.7 Hz), 30.40,

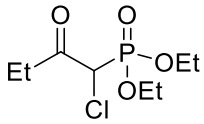
16.36 (d, *J* = 4.3 Hz), 10.89 (d, *J* = 6.4 Hz).**^{31}P NMR** (162 MHz, Chloroform-*d*) δ23.42. HRMS Calcd. For C8H17NaO4P+ [M+Na+]: 231.0757. Found: 231.0754.



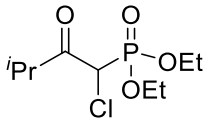
Diethyl (1-ethyl-2-methyl-2-oxoethyl)phosphonate (1d): colorless liquid, 1.22 g, 55% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 4.23 – 3.98 (m, 4H), 3.05 (ddt, *J* = 24.3, 10.6, 2.7 Hz, 1H), 2.31 (t, *J* = 1.7 Hz, 3H), 2.02 (ddtd, *J* = 16.6, 13.5, 7.4, 3.2 Hz, 1H), 1.86 (dtt, *J* = 14.5, 7.5, 3.6 Hz, 1H), 1.32 (td, *J* = 7.1, 1.8 Hz, 6H), 1.05 – 0.83 (m, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 203.93 (d, *J* = 4.1 Hz), 62.54 (dd, *J* = 16.7, 6.8 Hz), 55.44 (d, *J* = 124.9 Hz), 31.19, 20.02 (d, *J* = 5.1 Hz), 16.36 (dd, *J* = 6.0, 2.6 Hz), 13.12 (d, *J* = 15.8 Hz). **^{31}P NMR** (243 MHz, Chloroform-*d*) δ 22.32. HRMS Calcd. For C9H19NaO4P+ [M+Na+]: 245.0913. Found: 245.0911.



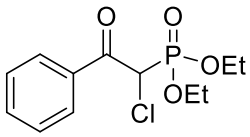
Diethyl (1-benzyl-2-methyl-2-oxoethyl)phosphonate (1e): colorless liquid, 1.64 g, 58% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.29 – 7.24 (m, 3H), 7.22 – 7.17 (m, 1H), 7.15 (d, *J* = 7.5 Hz, 2H), 4.16 (dddd, *J* = 13.9, 11.0, 6.8, 4.3 Hz, 4H), 3.53 (ddd, *J* = 24.2, 11.2, 3.4 Hz, 1H), 3.31 (ddd, *J* = 14.3, 11.2, 7.7 Hz, 1H), 3.10 (ddd, *J* = 14.2, 10.7, 3.3 Hz, 1H), 2.17 (d, *J* = 1.2 Hz, 3H), 1.35 (td, *J* = 7.1, 3.2 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 203.09 (d, *J* = 4.5 Hz), 138.99 (d, *J* = 15.9 Hz), 128.63, 128.54, 126.65, 62.90 (d, *J* = 6.8 Hz), 62.67 (d, *J* = 6.9 Hz), 55.14 (d, *J* = 123.5 Hz), 32.03, 16.40 (d, *J* = 6.0 Hz). **^{31}P NMR** (243 MHz, Chloroform-*d*) δ 21.34. HRMS Calcd. For C14H21NaO4P+ [M+Na+]: 307.1070. Found: 307.1071.



Diethyl (1-chloro-2-ethyl-2-oxoethyl)phosphonate (1f): colorless liquid, 786 mg, 65% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.49 (d, *J* = 17.4 Hz, 1H), 4.22 – 4.10 (m, 4H), 2.96 – 2.52 (m, 2H), 1.29 (tt, *J* = 6.8, 3.3 Hz, 6H), 1.03 (td, *J* = 7.1, 3.3 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 200.44, 64.55 (d, *J* = 6.9 Hz), 64.40 (d, *J* = 6.9 Hz), 57.37, 55.99, 33.73, 16.21 (d, *J* = 6.0 Hz), 7.61. **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.68. HRMS Calcd. For C8H16ClNaO4P+ [M+Na+]: 265.0367. Found: 265.0364.

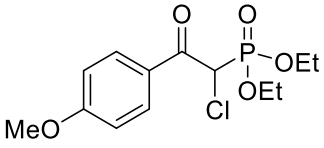


Diethyl (1-chloro-2-(*i*-propyl)-2-oxoethyl)phosphonate (1g): colorless liquid, 780 mg, 61% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.62 (dt, *J* = 17.5, 2.6 Hz, 1H), 4.16 (dddd, *J* = 14.5, 12.4, 8.2, 6.0 Hz, 4H), 3.14 (dqt, *J* = 13.5, 6.7, 3.2 Hz, 1H), 1.27 (qt, *J* = 6.5, 3.0 Hz, 6H), 1.07 (dt, *J* = 6.5, 3.2 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 203.59, 64.46 (t, *J* = 7.1 Hz), 56.01, 54.63, 38.89, 18.95, 18.25, 16.22 (d, *J* = 6.0 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.63. HRMS Calcd. For C9H18ClNaO4P+ [M+Na+]: 279.0523. Found: 279.0523.

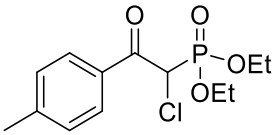


Diethyl (1-chloro-2-phenyl-2-oxoethyl)phosphonate (1h):[3] yellow solid, 657 mg, 75% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.99 (dt, *J* = 7.9, 3.4 Hz, 2H), 7.59 (dt, *J* = 8.1, 4.0 Hz, 1H), 7.47 (tt, *J* = 7.7, 3.3 Hz, 2H), 5.46 (dd, *J* = 16.4, 2.8 Hz, 1H), 4.21 (ddtt, *J* = 25.1, 10.7, 7.0, 3.4 Hz, 4H), 1.26 (ddq, *J* = 28.7, 7.1, 3.5 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 189.61, 134.85, 134.15, 129.35, 128.69, 64.62 (d, *J* = 7.1 Hz), 64.52 (d, *J* = 6.9 Hz), 53.41, 51.98, 16.23 (dd, *J* = 9.7, 6.0 Hz). **^{31}P NMR** (162

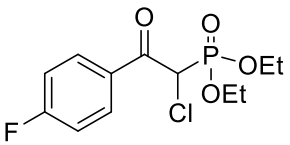
MHz, Chloroform-*d*) δ 12.60. HRMS Calcd. For C12H16ClNaO4P+ [M+Na+]: 313.0367. Found: 313.0370.



Diethyl (1-chloro-2-(4-methoxyphenyl)-2-oxoethyl)phosphonate (1i):[3] yellow solid, 753 mg, 78% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.03 – 7.82 (m, 2H), 6.88 (dd, *J* = 8.6, 6.4 Hz, 2H), 5.39 (d, *J* = 16.0 Hz, 1H), 4.15 (dtd, *J* = 14.3, 7.2, 3.7 Hz, 4H), 3.80 (q, *J* = 5.6, 4.9 Hz, 3H), 1.22 (dq, *J* = 20.8, 7.1 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 187.73, 164.37, 131.88 (d, *J* = 2.0 Hz), 127.56, 113.87, 66.20 – 62.62 (m), 55.55, 53.16, 51.73, 16.23 (t, *J* = 5.2 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 13.00. HRMS Calcd. For C13H18ClNaO5P+ [M+Na+]: 343.0473. Found: 343.0474.

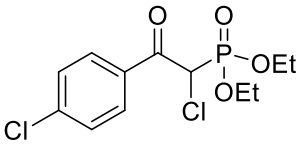


Diethyl (1-chloro-2-(4-methylphenyl)-2-oxoethyl)phosphonate (1j):[3] yellow solid, 762 mg, 83% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.04 – 7.78 (m, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.44 (d, *J* = 16.1 Hz, 1H), 4.24 (ddtd, *J* = 26.0, 11.8, 7.2, 3.2 Hz, 4H), 2.43 (s, 3H), 1.30 (dt, *J* = 26.6, 7.1 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 189.09, 145.35, 132.29, 129.47 (d, *J* = 19.7 Hz), 64.54 (dd, *J* = 10.9, 6.9 Hz), 52.54 (d, *J* = 143.9 Hz), 21.78, 16.27 (dd, *J* = 11.9, 6.1 Hz).**^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.85. HRMS Calcd. For C13H18ClNaO4P+ [M+Na+]: 327.0523. Found: 327.0522.

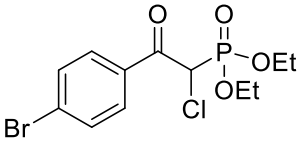


Diethyl (1-chloro-2-(4-florophenyl)-2-oxoethyl)phosphonate (1k):[3] yellow solid, 716 mg, 76.8% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.08 (ddt, *J* = 9.8, 4.9, 2.6

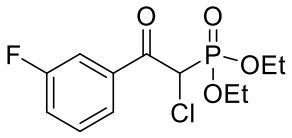
Hz, 2H), 7.16 (ddp, *J* = 7.9, 5.2, 2.8 Hz, 2H), 5.44 – 5.34 (m, 1H), 4.23 (dtdd, *J* = 13.3, 10.6, 5.4, 2.7 Hz, 4H), 1.30 (ddddt, *J* = 21.6, 9.3, 7.4, 4.5, 2.6 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 188.10, 132.37, 132.28, 116.03, 115.81, 64.72, 64.65, 53.59, 52.16, 16.24 (d, *J* = 6.1 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.44. Calcd. For C12H15ClFNaO4P+ [M+Na+]: 331.0273. Found: 331.0278.



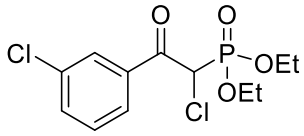
Diethyl (1-chloro-2-(4-chlorophenyl)-2-oxoethyl)phosphonate (1l):[3] yellow solid, 834 mg, 85.2% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.98 (ddd, *J* = 9.3, 4.7, 2.4 Hz, 2H), 7.46 (ddd, *J* = 8.0, 5.0, 2.7 Hz, 2H), 5.40 (dd, *J* = 16.6, 1.7 Hz, 1H), 4.24 (dd, *J* = 14.8, 12.6, 10.3, 7.5, 4.9, 2.7 Hz, 4H), 1.35 – 1.24 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 188.53, 140.78, 133.02, 130.86, 129.01, 64.69 (d, *J* = 6.9 Hz), 53.65, 52.22, 16.26. **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.30. HRMS Calcd. For C12H15Cl2NaO4P+ [M+Na+]: 346.9977. Found: 346.9981.



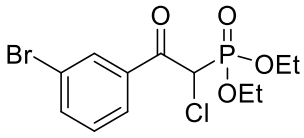
Diethyl (1-chloro-2-(4-bromophenyl)-2-oxoethyl)phosphonate (1m):[3] yellow solid, 688 mg, 62% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.87 (dd, *J* = 8.7, 2.6 Hz, 2H), 7.60 (dd, *J* = 8.6, 3.2 Hz, 2H), 5.38 (d, *J* = 16.6 Hz, 1H), 4.21 (dtq, *J* = 25.7, 11.1, 4.2, 3.6 Hz, 4H), 1.27 (dtd, *J* = 32.3, 7.1, 3.0 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 188.76 (d, *J* = 2.0 Hz), 133.45 (d, *J* = 2.0 Hz), 132.00, 130.90, 129.59, 64.69 (d, *J* = 6.8 Hz), 53.41, 52.46, 16.25 (dd, *J* = 7.7, 6.0 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.27. HRMS Calcd. For C12H15BrClNaO4P+ [M+Na+]: 390.9472. Found: 390.9471.



Diethyl (1-chloro-2-(3-florophenyl)-2-oxoethyl)phosphonate (1n):[3] yellow liquid, 725 mg, 78% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.81 (dd, *J* = 8.3, 4.5 Hz, 1H), 7.74 – 7.66 (m, 1H), 7.55 – 7.40 (m, 1H), 7.31 (tt, *J* = 8.1, 3.7 Hz, 1H), 5.45 – 5.34 (m, 1H), 4.29 – 4.17 (m, 4H), 1.34 – 1.21 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 188.60, 130.37 (d, *J* = 7.6 Hz), 125.23 (d, *J* = 3.1 Hz), 121.22 (d, *J* = 21.6 Hz), 116.32, 64.73, 53.60, 52.18, 16.24. **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.16 (d, *J* = 2.8 Hz). HRMS Calcd. For C12H15ClFNaO4P+ [M+Na+]: 331.0273. Found: 331.0275.

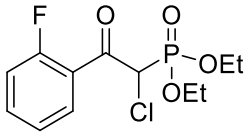


Diethyl (1-chloro-2-(3-chlorophenyl)-2-oxoethyl)phosphonate (1o):[3] yellow liquid, 706 mg, 72% yield. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.99 (t, *J* = 1.9 Hz, 1H), 7.96 – 7.87 (m, 1H), 7.63 – 7.57 (m, 1H), 7.44 (t, *J* = 7.9 Hz, 1H), 5.38 (d, *J* = 16.6 Hz, 1H), 4.50 – 4.01 (m, 4H), 1.48 – 1.07 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 188.59, 136.32, 135.06, 134.03, 129.97, 129.37, 127.54, 64.75 (dd, *J* = 10.1, 6.9 Hz), 53.68, 52.26, 16.21 (d, *J* = 6.3 Hz). **^{31}P NMR** (162 MHz, Chloroform-d) δ 12.08. HRMS Calcd. For C12H15Cl2NaO4P+ [M+Na+]: 346.9977. Found: 346.9976.

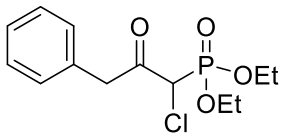


Diethyl (1-chloro-2-(3-bromophenyl)-2-oxoethyl)phosphonate (1p):[3] colorless liquid, 779 mg, 70% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.14 (q, *J* = 2.1 Hz, 1H), 7.96 (d, *J* = 7.9 Hz, 1H), 7.74 (dd, *J* = 7.9, 2.4 Hz, 1H), 7.38 (td, *J* = 8.0, 2.2 Hz, 1H), 5.38 (dt, *J* = 16.6, 1.7 Hz, 1H), 4.24 (tdd, *J* = 14.5, 7.2, 2.4 Hz, 4H), 1.30 (dtd, *J* = 25.7, 7.2, 2.2 Hz, 7H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 188.47 (d, *J* = 2.0 Hz),

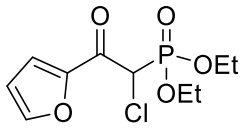
137.79 – 135.44 (m), 132.27, 130.21, 127.98, 122.94, 64.75 (dd, *J* = 12.3, 6.9 Hz), 52.97 (d, *J* = 143.5 Hz), 16.26 (dd, *J* = 8.1, 6.1 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.07. HRMS Calcd. For C12H15BrClNaO4P+ [M+Na+]: 390.9472. Found: 390.9471.



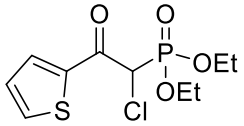
Diethyl (1-chloro-2-(2-florophenyl)-2-oxoethyl)phosphonate (1q):[3] yellow liquid, 762 mg, 82.1% yield **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.85 (tt, *J* = 7.5, 2.1 Hz, 1H), 7.57 (dddt, *J* = 8.5, 6.9, 3.2, 1.6 Hz, 1H), 7.31 – 7.22 (m, 1H), 7.21 – 7.11 (m, 1H), 5.67 – 5.57 (m, 1H), 4.30 – 4.08 (m, 4H), 1.24 (dtd, *J* = 33.3, 7.1, 2.7 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 187.48, 162.61, 160.08, 135.62 (d, *J* = 9.3 Hz), 131.40 (d, *J* = 2.0 Hz), 124.82 (d, *J* = 3.3 Hz), 116.69, 116.45, 64.61 (d, *J* = 6.8 Hz), 64.37 (d, *J* = 6.8 Hz), 57.57 (d, *J* = 10.3 Hz), 56.16 (d, *J* = 10.1 Hz), 16.15 (dd, *J* = 14.3, 6.1 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.13. HRMS Calcd. For C12H15ClFNaO4P+ [M+Na+]: 331.0273. Found: 331.0271.



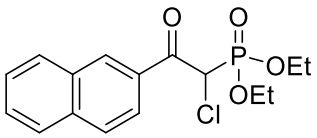
Diethyl (1-chloro-2-benzyl-2-oxoethyl)phosphonate (1r):[3] colorless liquid, 597 mg, 65% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.40 – 7.14 (m, 7H), 4.64 (d, *J* = 17.4 Hz, 1H), 4.35 – 4.20 (m, 4H), 4.20 – 4.03 (m, 2H), 1.37 (td, *J* = 7.1, 1.6 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 197.26, 132.97, 129.69, 128.76, 64.70 (dd, *J* = 16.0, 6.9 Hz), 56.42 (d, *J* = 138.4 Hz), 47.03, 16.34 (d, *J* = 5.9 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.27. HRMS Calcd. For C13H18ClNaO4P+ [M+Na+]: 327.0523. Found: 327.0524.



Dimethyl (1-chloro-2-(furan-2-yl)-2-oxoethyl)phosphonate (1s):[3] yellow solid, 744 mg, 88% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 1.7 Hz, 1H), 7.43 (d, *J* = 3.6 Hz, 1H), 6.62 (dd, *J* = 3.6, 1.7 Hz, 1H), 5.34 (dd, *J* = 16.4, 1.5 Hz, 1H), 4.35 – 4.11 (m, 4H), 1.31 (dt, *J* = 26.7, 7.1 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 177.78 (d, *J* = 2.4 Hz), 150.60 (d, *J* = 2.1 Hz), 147.67, 120.28, 113.16, 64.64 (dd, *J* = 29.2, 6.9 Hz), 52.71, 51.77, 16.28 (dd, *J* = 11.6, 6.1 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.20. HRMS Calcd. For C10H14ClNaO5P+ [M+Na+]: 305.0316. Found: 303.0160.



Dimethyl (1-chloro-2-oxo-2-(thiophen-2-yl)ethyl)phosphonate (1t):[3] yellow solid , 760 mg, 85% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.95 (dd, *J* = 4.0, 1.1 Hz, 1H), 7.76 (dd, *J* = 4.9, 1.1 Hz, 1H), 7.17 (dd, *J* = 5.0, 3.9 Hz, 1H), 5.22 (d, *J* = 16.1 Hz, 1H), 4.41 – 4.09 (m, 4H), 1.32 (dtd, *J* = 19.1, 7.0, 0.8 Hz, 7H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 182.01, 141.28 (d, *J* = 2.3 Hz), 136.04, 134.88, 128.48, 64.72 (t, *J* = 6.6 Hz), 53.95, 53.00, 17.10 – 15.34 (m). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.22. HRMS Calcd. For C10H14ClNaO4PS+ [M+Na+]: 318.9931. Found: 318.9936.

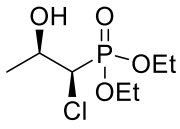


Diethyl (1-chloro-2-naphthyl-2-oxoethyl)phosphonate (1u):[3] white solid, 851 mg, 83% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.05 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.99 (dd, *J* = 8.3, 1.3 Hz, 1H), 7.95 – 7.85 (m, 2H), 7.64 (ddd, *J* = 8.2, 6.9, 1.4 Hz, 1H), 7.57 (ddd, *J* = 8.1, 6.9, 1.3 Hz, 1H), 5.62 (d, *J* = 16.2 Hz, 1H), 4.38 – 4.11 (m, 4H), 1.29 (dtd,

J = 36.7, 7.0, 0.8 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 189.49, 135.97, 132.25, 132.10, 131.72, 129.90, 129.24, 128.66, 127.82, 127.10, 124.43, 64.72 (d, *J* = 7.0 Hz), 64.56 (d, *J* = 6.9 Hz), 53.46, 52.03, 16.29 (dd, *J* = 9.7, 6.0 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.80. HRMS Calcd. For C16H18ClNaO4P+ [M+Na+]: 363.0523. Found: 363.0525.

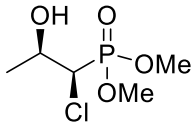
V. Procedure for dynamic kinetic resolution reaction

To a 2.5 mL vial was added the substrates (0.1 mmol), catalyst (0.001 mmol), Et_3N/HCOOH (5:2) mixture (0.05 mL) and anhydrous DCM (0.2 mL) under argon atmosphere. The mixture was stirred for 2 hours at room temperature. After the reaction finished, the reaction mixture was passed through a short column of silica gel to remove the metal complex. The product was analyzed by ^{1}H NMR to determine the conversion. The ee values and diastereo-isomer ratio were determined by HPLC on a chiral stationary phase. For hydrogenation product **2a-2f**, derivatization was conducted before determination. 0.4 mL pyridine and 0.2 mmol benzoyl chloride was added. The mixture was allowed to stir at room temperature for 4 hours. After the reaction finished, 4 M HCl solution was add, the mixture was quenched with EtOAc and concentrated *in vacuo*.[1] The product was analyzed by ^{1}H NMR to determine the conversion. The ee values and diastereo-isomer ratio were determined by HPLC or UPLC on a chiral stationary phase. The configuration was determined by optical rotation and previous literature.[4]



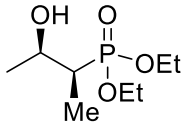
***Cis*-(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(methyl)ethyl)phosphonate (2a)[4]**

93% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 4.35 (tt, *J* = 8.7, 6.3 Hz, 1H), 4.31 – 4.15 (m, 4H), 3.85 (dd, *J* = 12.5, 2.4 Hz, 1H), 1.42 – 1.32 (m, 9H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 66.19 (d, *J* = 1.9 Hz), 63.93 (dd, *J* = 205.9, 7.0 Hz), 57.17 (d, *J* = 155.6 Hz), 20.12 (d, *J* = 11.0 Hz), 16.41 (dd, *J* = 9.3, 5.9 Hz). **^{31}P NMR** (243 MHz, Chloroform-*d*) δ 19.15. $[\alpha]^{20}_D$ = +52.2 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral OD-H column, 230 nm, 25 ^{o}C, ^{n}Hexane: ^{i}PrOH = 90:10; flow 1.0 mL/min; t_1 (minor) = 5.56 min, t_2 (minor) = 5.85 min, t_3 (minor) = 7.99 min, t_4 (major) = 10.53 min. HRMS Calcd. For C7H16ClNaO4P+ [M+Na+]: 253.0367 Found: 253.0366.



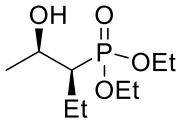
***Cis* -(1*S*, 2*R*)-dimethyl (1-chloro-2-hydroxy-2-(methyl)ethyl)phosphonate (2b)[4]**

91% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.45 – 4.26 (m, 1H), 3.92 (d, *J* = 10.7 Hz, 3H), 3.90 (dd, *J* = 12.5, 2.5 Hz, 1H), 3.84 (d, *J* = 10.8 Hz, 3H), 3.04 (d, *J* = 5.1 Hz, 1H), 1.35 (dd, *J* = 6.3, 1.4 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 65.90 (d, *J* = 4.7 Hz), 64.29 – 57.28 (m), 37.42 (d, *J* = 137.0 Hz), 20.23 (d, *J* = 13.8 Hz), 17.85 – 13.74 (m), 7.15 (d, *J* = 3.9 Hz).**^{31}P NMR** (162 MHz, Chloroform-*d*) δ33.52. $[\alpha]^{20}_{D}$ = +48.4 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral OD-H column, 220 nm, 25 °C, *n*Hexane: *i*PrOH = 90:10; flow 1.0 mL/min; t_1 (minor) = 7.75 min, t_2 (major) = 8.82 min, t_3 (minor) = 11.82 min, t_4 (minor) = 16.93 min. HRMS Calcd. For C5H12ClNaO4P+ [M+Na+]: 225.0054. Found: 225.0052.



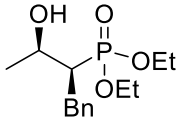
***cis*-(1*S*,2*R*)-Diethyl (1-methyl-2-hydroxy-2-(methyl)ethyl)phosphonate (2c)[5]**

93% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.22 – 4.10 (m, 4H), 3.21 (d, *J* = 4.0 Hz, 1H), 2.08 – 1.88 (m, 1H), 1.36 (t, *J* = 7.1 Hz, 6H), 1.27 – 1.16 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 65.90 (d, *J* = 4.7 Hz), 61.97 (d, *J* = 5.3 Hz), 37.42 (d, *J* = 137.1 Hz), 20.22 (d, *J* = 13.8 Hz), 16.44, 7.15 (d, *J* = 3.9 Hz).**^{31}P NMR** (243 MHz, Chloroform-*d*) δ 33.52. $[\alpha]^{20}_{D}$ = +33.6 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral OD-3 column, 230 nm, 25 °C, *n*Hexane: *i*PrOH = 95:5; flow 1.0 mL/min; t_1 (minor) = 10.74 min, t_2 (major) = 11.87 min, t_3 (minor) = 12.79 min, t_4 (minor) = 13.98 min. HRMS Calcd. For C8H19NaO4P+ [M+Na+]: 233.0913. Found: 233.0917



***Cis*-(1*S*, 2*R*)-diethyl (1-ethyl-2-hydroxy-2-(methyl)ethyl)phosphonate (2d).**

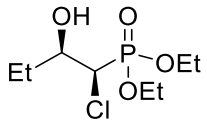
90% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 4.15 – 4.07 (m, 4H), 3.63 (d, *J* = 7.4 Hz, 1H), 1.88 (dtd, *J* = 19.7, 6.6, 3.2 Hz, 1H), 1.69 (tt, *J* = 14.8, 7.4 Hz, 1H), 1.61 – 1.47 (m, 1H), 1.32 (td, *J* = 7.1, 1.9 Hz, 7H), 1.27 (d, *J* = 6.5 Hz, 3H), 1.03 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 66.54 (d, *J* = 4.5 Hz), 61.85 (d, *J* = 6.5 Hz), 61.54 (d, *J* = 7.1 Hz), 45.72, 44.84, 20.07, 18.39 (d, *J* = 3.9 Hz), 16.45, 13.48 (d, *J* = 7.7 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 32.95. $[\alpha]^{20}_{D}$ = +40.1 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **UPLC** on Chiral IC-U column, 230 nm, 25 °C, *n*Hexane: *i*PrOH = 95:5 in 0-30 min, then *n*Hexane: *i*PrOH = 50:50 in 30-60 min; flow 0.5 mL/min; t_1 (minor) = 16.40 min, t_2 (minor) = 19.52 min, t_3 (minor) = 20.94 min, t_4 (major) = 34.94 min. HRMS Calcd. For C9H21NaO4P+ [M+Na+]: 247.1070. Found: 247.1072.



***Cis*-(1*S*, 2*R*)-diethyl (1-benzyl-2-hydroxy-2-(methyl)ethyl)phosphonate (2e).**

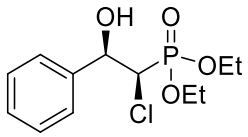
95% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.28 (t, *J* = 7.6 Hz, 2H), 7.24 – 7.18 (m, 3H), 4.21 – 3.92 (m, 4H), 3.62 (d, *J* = 7.9 Hz, 1H), 3.01 (td, *J* = 13.8, 6.2 Hz, 1H), 2.79 (td, *J* = 15.5, 8.0 Hz, 1H), 2.48 – 2.28 (m, 1H), 1.34 – 1.30 (m, 6H), 1.26 (t, *J* = 5.2 Hz, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 128.76, 128.49, 126.43, 66.25 (d, *J* = 4.7 Hz), 62.02 (d, *J* = 6.6 Hz), 61.71 (d, *J* = 7.2 Hz), 45.69, 44.80, 31.18 (d, *J* = 3.3 Hz), 20.10 (d, *J* = 9.1 Hz), 16.38 (dd, *J* = 8.7, 6.0 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 31.67. $[\alpha]^{20}_{D}$ = +48.5 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **UPLC** on Chiral IC-U column, 230 nm, 25 °C, *n*Hexane: *i*PrOH = 95:5 in 0-30 min, then *n*Hexane: *i*PrOH = 50:50 in 30-60 min; flow 0.5 mL/min;

t_1 (minor) = 18.87 min, t_2 (minor) = 20.33 min, t_3 (minor) = 23.44 min, t_4 (major) = 35.66 min. HRMS Calcd. For C14H23NaO4P+ [M+Na+]: 309.1226. Found: 309.1226.



***Cis*-(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(ethyl)ethyl)phosphonate (2f)**

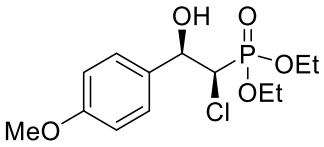
94% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 4.36 – 4.14 (m, 4H), 4.05 (dq, *J* = 12.6, 6.8, 5.8 Hz, 1H), 3.94 (dd, *J* = 13.1, 2.0 Hz, 1H), 3.11 (d, *J* = 4.6 Hz, 1H), 1.83 – 1.69 (m, 2H), 1.69 – 1.55 (m, 1H), 1.37 (t, *J* = 7.1 Hz, 7H), 0.96 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 71.24 (d, *J* = 2.5 Hz), 64.64 (d, *J* = 6.9 Hz), 63.26 (d, *J* = 7.1 Hz), 56.16, 54.60, 26.92 (d, *J* = 11.3 Hz), 16.42 (t, *J* = 6.0 Hz), 9.97. **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 19.66. $[\alpha]^{20}_D$ = +26.2 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 220 nm, 25 ºC, ^{n}Hexane: ^{i}PrOH = 95:5; flow 1.0 mL/min; t_1 (major) = 41.03 min, t_2 (minor) = 45.49 min, t_3 (minor) = 47.72 min, t_4 (minor) = 85.46 min. HRMS Calcd. For C8H18ClNaO4P+ [M+Na+]: 267.0523. Found: 267.0520.



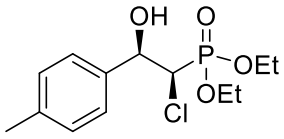
***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(phenyl)ethyl)phosphonate (2h)[3]**

95% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 7.2 Hz, 2H), 7.37 (dd, *J* = 8.4, 6.8 Hz, 2H), 7.33 – 7.29 (m, 1H), 5.37 (dd, *J* = 5.4, 2.6 Hz, 1H), 4.41 – 4.16 (m, 4H), 4.10 (dd, *J* = 12.6, 2.6 Hz, 1H), 3.69 (s, 1H), 1.35 (td, *J* = 7.1, 3.1 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 139.31 (d, *J* = 12.4 Hz), 128.27, 128.16, 126.36, 71.55, 64.12 (dd, *J* = 233.5, 6.9 Hz), 57.65 (d, *J* = 153.4 Hz), 16.39 (dd, *J* = 8.4, 5.8 Hz). **^{31}P NMR** (243 MHz, Chloroform-*d*) δ 18.85. $[\alpha]^{20}_D$ = +62.6 (c 0.5, $CHCl_3$). The

enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, ^{n}Hexane: ^{i}PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 10.96 min, t_2 (major) = 14.14 min, t_3 (minor) = 17.75 min, t_4 (minor) = 66.95 min. C12H18ClNaO4P+ [M+Na+]: 315.0523. Found: 315.0523.

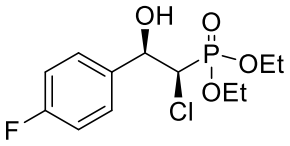


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(4-methoxyphenyl)ethyl)phosphonate (2i).**[3] 92% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.34 (dd, *J* = 8.6, 2.1 Hz, 2H), 6.89 (dd, *J* = 8.6, 2.0 Hz, 2H), 5.31 (dq, *J* = 7.5, 4.9, 3.6 Hz, 1H), 4.37 – 4.10 (m, 4H), 4.05 (dt, *J* = 12.3, 2.3 Hz, 1H), 3.84 – 3.76 (m, 3H), 3.65 (s, 1H), 1.34 (td, *J* = 7.0, 1.7 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 159.47, 131.44 (d, *J* = 11.9 Hz), 127.62, 113.64, 71.29, 64.83 (d, *J* = 7.0 Hz), 63.30 (d, *J* = 7.1 Hz), 58.63, 57.11, 55.29, 16.39 (t, *J* = 6.2 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.88. $[\alpha]^{20}_{D}$ = +35.5 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, ^{n}Hexane: ^{i}PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 18.12 min, t_2 (minor) = 28.67 min, t_3 (major) = 31.10 min, t_4 (minor) = 98.48 min. HRMS Calcd. For C13H20ClNaO5P+ [M+Na+]: 345.0629. Found: 345.0632.

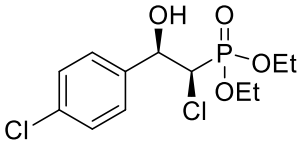


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(4-methylphenyl)ethyl)phosphonate (2j).**[3] 94% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.30 (d, *J* = 7.7 Hz, 2H), 7.17 (d, *J* = 7.5 Hz, 2H), 5.34 (dd, *J* = 5.3, 2.6 Hz, 1H), 4.38 – 4.13 (m, 4H), 4.08 (dd, *J* = 12.6, 2.6 Hz, 1H), 2.34 (d, *J* = 4.4 Hz, 3H), 1.35 (t, *J* = 7.0 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 137.87, 136.40 (d, *J* = 12.1 Hz), 128.95, 126.23, 71.38, 64.86 (d, *J* = 6.9 Hz), 63.37 (d, *J* = 7.1 Hz), 58.57, 57.05, 21.17, 16.39 (t, *J* = 6.2 Hz). **^{31}P**

NMR (162 MHz, Chloroform-*d*) δ 18.94. $[\alpha]^{20}_D$ = +23.3 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 14.52 min, t_2 (minor) = 21.55 min, t_3 (major) = 24.80 min, t_4 (minor) = 68.94 min. HRMS Calcd. For C13H20ClNaO4P+ [M+Na+]: 329.0680. Found: 329.0680.

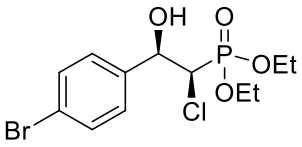


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(4-florophenyl)ethyl)phosphonate (2k).**[3] 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.45 – 7.34 (m, 2H), 7.05 (t, *J* = 8.7 Hz, 2H), 5.37 – 5.29 (m, 1H), 4.36 – 4.12 (m, 4H), 4.03 (dd, *J* = 12.7, 2.6 Hz, 1H), 3.86 (s, 1H), 1.35 (t, *J* = 7.1 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 128.16 (d, *J* = 8.2 Hz), 115.13 (d, *J* = 21.4 Hz), 71.06, 65.01 (d, *J* = 7.4 Hz), 58.34, 56.82, 16.38. ^{31}P NMR (162 MHz, Chloroform-*d*) δ 18.68. $[\alpha]^{20}_D$ = +34.9 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 7.69 min, t_2 (major) = 9.28 min, t_3 (minor) = 11.58 min, t_4 (minor) = 53.34 min. HRMS Calcd. For C12H17ClFNaO4P+ [M+Na+]: 333.0429. Found: 333.0429.

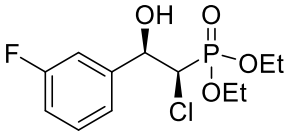


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(4-chlorophenyl)ethyl)phosphonate (2l).**[3] 93% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.39 – 7.31 (m, 4H), 5.33 (dt, *J* = 5.2, 2.5 Hz, 1H), 4.38 – 4.15 (m, 4H), 4.03 (dd, *J* = 12.8, 2.5 Hz, 1H), 3.88 (d, *J* = 3.2 Hz, 1H), 1.35 (td, *J* = 7.1, 4.7 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 137.85 (d, *J* = 12.5 Hz), 133.93, 128.42, 127.81, 71.01, 64.25 (dd, *J* = 251.7, 7.1 Hz), 57.35 (d, *J* = 153.7 Hz), 16.39 (dd, *J* = 8.7, 5.9 Hz). **^{31}P NMR** (243 MHz) δ 18.09. $[\alpha]^{20}_D$ = +53.3

(c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, ^{n}Hexane: ^{i}PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 7.35 min, t_2 (major) = 8.50 min, t_3 (minor) = 11.80 min, t_4 (minor) = 41.70 min. HRMS Calcd. For C12H17Cl2NaO4P+ [M+Na+]: 349.0134. Found: 349.0130.

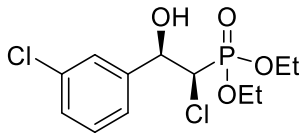


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(4-bromophenyl)ethyl)phosphonate (2m).**[3] 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 5.38 – 5.23 (m, 1H), 4.47 – 4.11 (m, 4H), 4.03 (dt, *J* = 12.8, 1.8 Hz, 1H), 3.90 (d, *J* = 3.3 Hz, 1H), 1.47 – 1.20 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 138.34, 131.37, 128.15, 122.08, 71.06, 65.10 (d, *J* = 7.0 Hz), 63.43 (d, *J* = 7.0 Hz), 58.03, 56.51, 29.70, 16.39. **^{31}P NMR** (162 MHz, Chloroform-d) δ 18.61. $[\alpha]^{20}_D$ = +43.7 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio was determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, ^{n}Hexane: ^{i}PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 7.72 min, t_2 (major) = 8.94 min, t_3 (minor) = 11.40 min, t_4 (minor) = 41.70 min. HRMS Calcd. For C12H17BrClNaO4P+ [M+Na+]: 392.9629. Found: 392.9627.

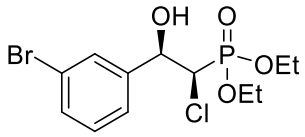


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(3-florophenyl)ethyl)phosphonate (2n).**[3] 97% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.33 (td, *J* = 8.0, 5.8 Hz, 1H), 7.17 (td, *J* = 8.9, 8.0, 1.7 Hz, 2H), 7.03 – 6.98 (m, 1H), 5.36 (dt, *J* = 5.5, 2.8 Hz, 1H), 4.39 – 4.18 (m, 4H), 4.07 (dd, *J* = 12.9, 2.4 Hz, 1H), 3.84 (d, *J* = 3.3 Hz, 1H), 1.41 – 1.32 (m, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 162.80 (d, *J* = 246.0 Hz), 141.98 (dd, *J* = 12.6, 7.1 Hz), 129.76 (d, *J* = 8.2 Hz), 121.80 (d, *J* = 3.1 Hz), 115.00 (d, *J* = 21.2 Hz), 113.70 (d, *J* = 22.6 Hz), 70.93, 64.27 (dd, *J* = 249.7, 7.1 Hz), 57.23 (d, *J* =

154.1 Hz), 16.40 (t, *J* = 6.4 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 12.16 (d, *J* = 2.8 Hz). $[\alpha]^{20}_D$ = +44.6 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio was determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 6.81 min, t_2 (major) = 7.77 min, t_3 (minor) = 9.89 min, t_4 (minor) = 36.84 min. HRMS Calcd. For C12H17ClFNaO4P+ [M+Na+]: 333.0429. Found: 333.0433.

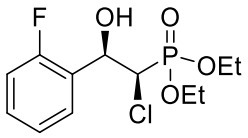


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(3-chlorophenyl)ethyl)phosphonate (2o)**.[3] 96% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.45 (d, *J* = 1.4 Hz, 1H), 7.36 – 7.28 (m, 3H), 5.35 (dd, *J* = 5.4, 2.4 Hz, 1H), 4.43 – 4.18 (m, 4H), 4.08 (dd, *J* = 12.9, 2.4 Hz, 1H), 3.97 (s, 1H), 1.47 – 1.34 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 141.47 (d, *J* = 12.5 Hz), 134.27, 129.52, 128.26, 126.68, 124.53, 70.98, 65.09 (d, *J* = 7.2 Hz), 63.44 (d, *J* = 7.2 Hz), 57.99, 56.46, 19.81 – 10.58 (m). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.38. ^{31}P NMR (243 MHz, Chloroform-*d*) δ 18.66. $[\alpha]^{20}_D$ = +24.3 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio was determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 7.79 min, t_2 (major) = 8.24 min, t_3 (minor) = 11.43 min, t_4 (minor) = 49.27 min. HRMS Calcd. For C12H17Cl2NaO4P+ [M+Na+]: 349.0134. Found: 349.0136.

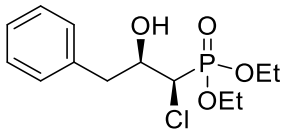


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(3-bromophenyl)ethyl)phosphonate (2p)**.[3] 97% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.61 (d, *J* = 2.0 Hz, 1H), 7.52 – 7.42 (m, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.30 – 7.21 (m, 1H), 5.40 – 5.31 (m, 1H), 4.38 – 4.17 (m, 4H), 4.07 (dd, *J* = 12.9, 2.4 Hz, 1H), 3.96 (d, *J* = 3.2 Hz, 1H), 1.38 (td, *J* = 7.1, 4.3 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 141.70 (d, *J* = 12.6 Hz), 131.19, 129.68 (d, *J* = 25.7 Hz), 128.26, 126.37, 125.04, 122.41, 70.93, 64.26 (dd, *J* = 166.4,

7.0 Hz), 57.22 (d, *J* = 153.9 Hz), 16.40. **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.58. $[\alpha]^{20}_{D}$ = +62.3 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio was determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 90:10; flow 1.0 mL/min; t_1 (minor) = 15.54 min, t_2 (major) = 16.60 min, t_3 (minor) = 25.17 min, t_4 (minor) = 119.94 min. HRMS Calcd. For C12H17BrClNaO4P+ [M+Na+]: 392.9629. Found: 392.9629.

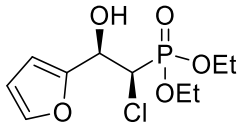


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(2-florophenyl)ethyl)phosphonate (2q).**[3] 93% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.68 (td, *J* = 7.6, 1.9 Hz, 1H), 7.58 (td, *J* = 7.5, 1.8 Hz, 4H), 7.40 – 7.27 (m, 6H), 7.21 (ddd, *J* = 8.9, 6.1, 1.2 Hz, 5H), 7.05 (dtd, *J* = 10.8, 7.9, 1.2 Hz, 5H), 5.69 (s, 1H), 5.36 (ddd, *J* = 17.1, 7.5, 5.1 Hz, 4H), 4.63 (d, *J* = 5.2 Hz, 4H), 4.44 – 4.19 (m, 17H), 4.19 – 3.90 (m, 9H), 1.41 (tt, *J* = 7.2, 4.6 Hz, 17H), 1.22 (t, *J* = 7.1 Hz, 12H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 129.99, 128.37, 124.30 (d, *J* = 3.4 Hz), 115.24 (d, *J* = 21.9 Hz), 69.31, 64.15 (dd, *J* = 153.1, 7.0 Hz), 52.38, 16.26 (dd, *J* = 35.4, 5.9 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.97, 18.91. $[\alpha]^{20}_{D}$ = +27.6 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio was determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 8.03 min, t_2 (minor) = 9.76 min, t_3 (minor) = 11.34 min, t_4 (major) = 18.83 min. HRMS Calcd. For C12H17ClFNaO4P+ [M+Na+]: 333.0429. Found: 333.0430.

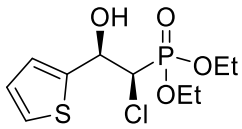


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-benzylethyl)phosphonate (2r).** 91% yield.[3] **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.68 – 7.12 (m, 5H), 4.41 (dtt, *J* = 9.7, 5.8, 1.7 Hz, 1H), 4.34 – 4.08 (m, 4H), 3.83 (dd, *J* = 13.1, 1.6 Hz, 1H), 3.38 (d, *J* = 3.9 Hz,

1H), 3.07 (ddd, *J* = 13.5, 6.4, 2.7 Hz, 1H), 2.95 (dd, *J* = 13.5, 7.9 Hz, 1H), 1.35 (dt, *J* = 9.6, 7.1 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 136.82, 129.32, 128.77, 126.92, 71.05 (d, *J* = 2.2 Hz), 64.02 (dd, *J* = 139.0, 7.0 Hz), 54.39 (d, *J* = 157.3 Hz), 39.90 (d, *J* = 12.0 Hz), 16.37 (dd, *J* = 8.5, 5.8 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 19.64. $[\alpha]^{20}_{D}$ = +57.1 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 12.23 min, t_2 (minor) = 15.58 min, t_3 (major) = 16.9 min, t_4 (minor) =39.37 min. HRMS Calcd. For C13H20ClNaO4P+ [M+Na+]: 329.0680. Found: 329.0678.

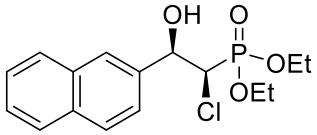


***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(furan-2-yl)ethyl)phosphonate (2s).** 94% yield.[3] **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.37 (d, *J* = 1.8 Hz, 1H), 6.44 (d, *J* = 3.3 Hz, 1H), 6.36 (dd, *J* = 3.4, 1.8 Hz, 1H), 5.35 (dd, *J* = 6.1, 2.8 Hz, 1H), 4.32 (dd, *J* = 12.5, 2.8 Hz, 1H), 4.30 – 4.13 (m, 4H), 1.35 (t, *J* = 7.1 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 151.90 (d, *J* = 13.1 Hz), 142.14, 110.53, 108.09, 67.28, 64.75 (d, *J* = 6.7 Hz), 63.48 (d, *J* = 7.2 Hz), 55.67, 54.13, 16.38 (t, *J* = 5.8 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.09. $[\alpha]^{20}_{D}$ = +53.8 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio was determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, *n*Hexane: *i*PrOH = 80:20; flow 1.0 mL/min; t_1 (major) = 16.75 min, t_2 (minor) = 19.85 min, t_3 (minor) = 25.08 min, t_4 (minor) = 77.52 min. HRMS Calcd. For C10H16ClNaO5P+ [M+Na+]: 305.0316. Found: 305.0319.



***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(thiophen-2-yl)ethyl)phosphonate**

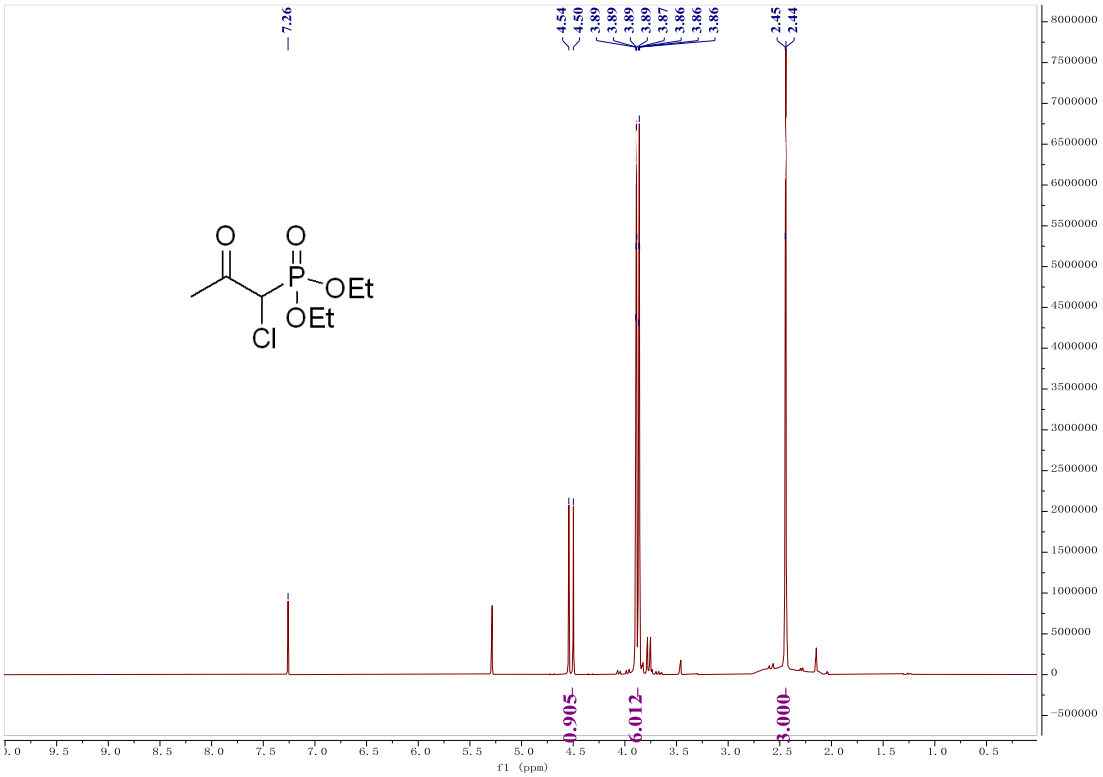
(2t).[3] 92% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.29 (d, *J* = 5.0 Hz, 1H), 7.07 (d, *J* = 3.6 Hz, 1H), 7.02 – 6.96 (m, 1H), 5.60 (dd, *J* = 6.6, 2.5 Hz, 1H), 4.35 – 4.18 (m, 4H), 4.18 – 4.13 (m, 2H), 3.91 (s, 1H), 1.35 (td, *J* = 7.1, 4.1 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 143.00 (d, *J* = 14.2 Hz), 126.58, 125.39, 124.75, 68.88, 64.88 (d, *J* = 6.9 Hz), 63.46 (d, *J* = 7.1 Hz), 58.10, 56.58, 16.38 (t, *J* = 5.7 Hz). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.14. $[\alpha]^{20}_{D}$ = +59.3 (c 0.5, $CHCl_3$). The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, ^{n}Hexane: ^{i}PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 13.85 min, t_2 (major) = 16.57 min, t_3 (minor) = 26.35 min, t_4 (minor) = 90.66 min. HRMS Calcd. For C10H16ClNaO4PS+ [M+Na+]: 321.0088. Found: 321.0090.



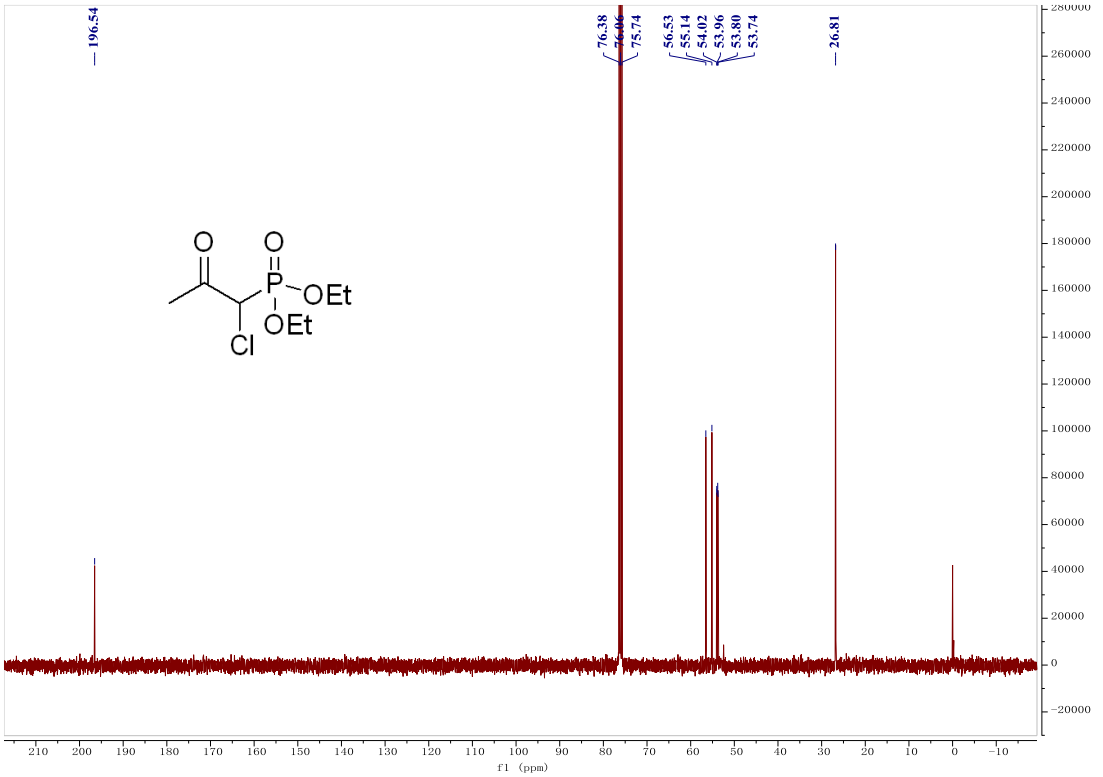
***Cis* -(1*S*, 2*R*)-diethyl (1-chloro-2-hydroxy-2-(naphthalen-2-yl)ethyl)phosphonate (2u).**[3] 95% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.99 – 7.95 (m, 1H), 7.92 – 7.81 (m, 3H), 7.57 – 7.44 (m, 3H), 5.57 (dt, *J* = 5.2, 2.5 Hz, 1H), 4.38 – 4.18 (m, 4H), 3.97 (d, *J* = 3.4 Hz, 1H), 1.43 – 1.32 (m, 6H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 136.75 (d, J = 12.3 Hz), 133.13 (d, J = 7.5 Hz), 128.18, 128.02, 127.68, 126.26, 126.16, 125.71, 123.91, 71.64, 64.96 (d, J = 6.7 Hz), 63.38 (d, J = 7.1 Hz), 58.35, 56.82, 17.68 – 14.08 (m). **^{31}P NMR** (162 MHz, Chloroform-*d*) δ 18.14. $[\alpha]^{20}_{D}$ = +38.9 (c 0.5, $CHCl_3$) The enantiomeric excess and diastereo-isomer ratio were determined by **HPLC** on Chiral IC column, 210 nm, 25 °C, ^{n}Hexane: ^{i}PrOH = 80:20; flow 1.0 mL/min; t_1 (minor) = 12.57 min, t_2 (major) = 15.44 min, t_3 (minor) = 24.92 min, t_4 (minor) = 67.12 min. HRMS Calcd. For C16H20ClNaO4P+ [M+Na+]: 365.0680. Found: 365.0679.

VI. NMR Spectra

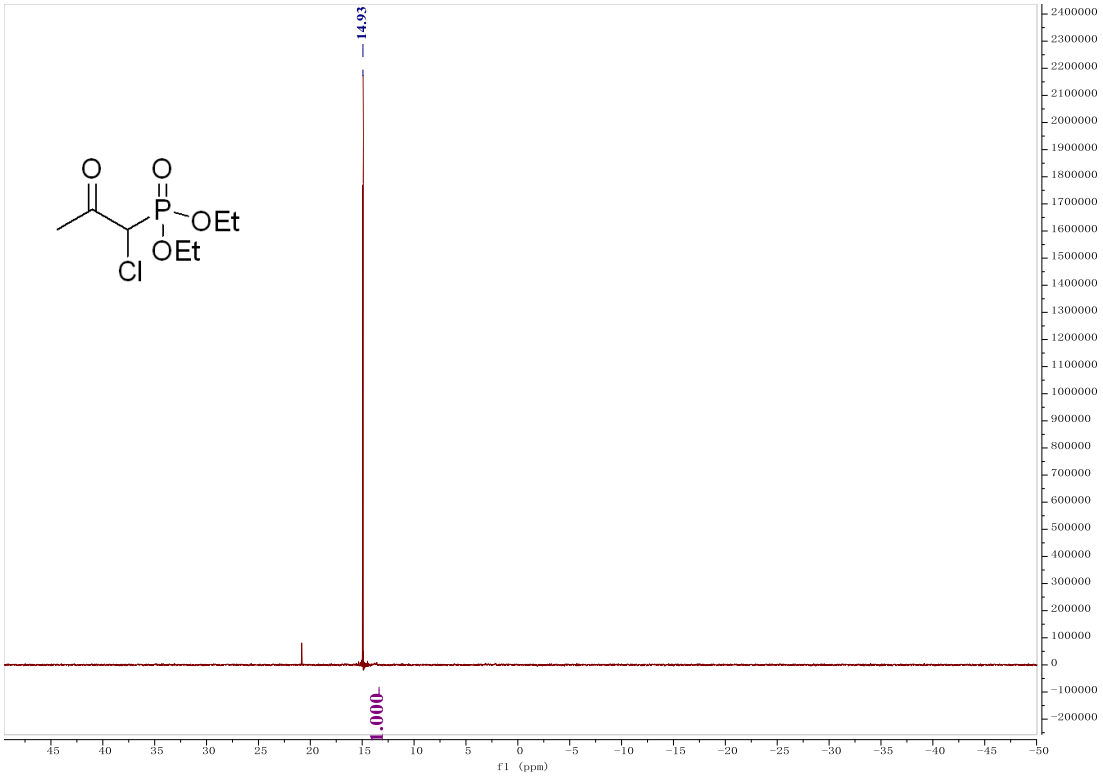
^{1}H NMR of **1a** (400 MHz, Chloroform-*d*)



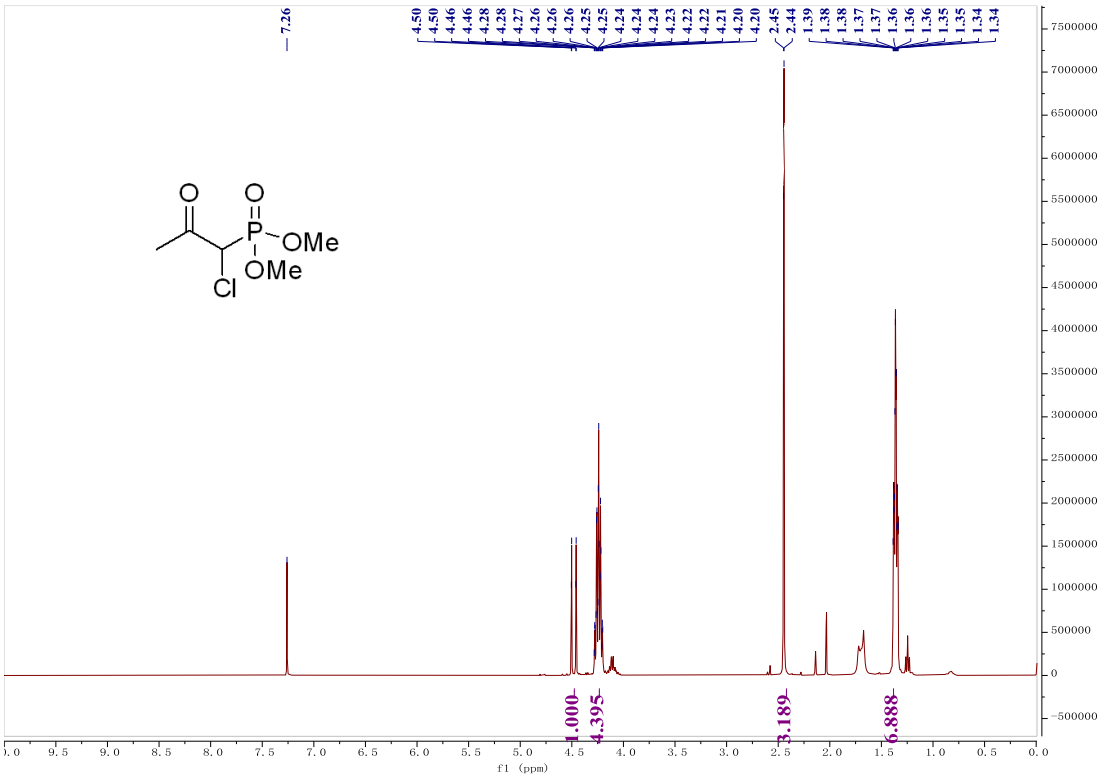
^{13}C NMR of **1a** (101 MHz, Chloroform-*d*)



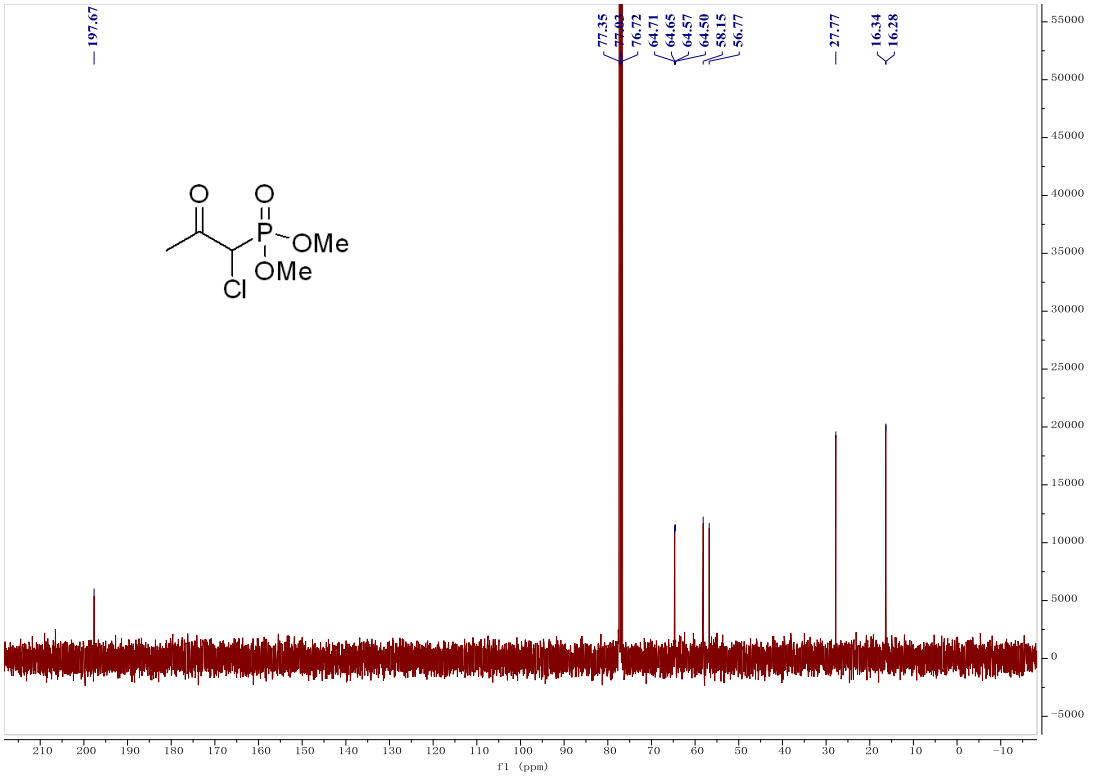
^{31}P NMR of **1a**, (162 MHz, Chloroform-*d*)



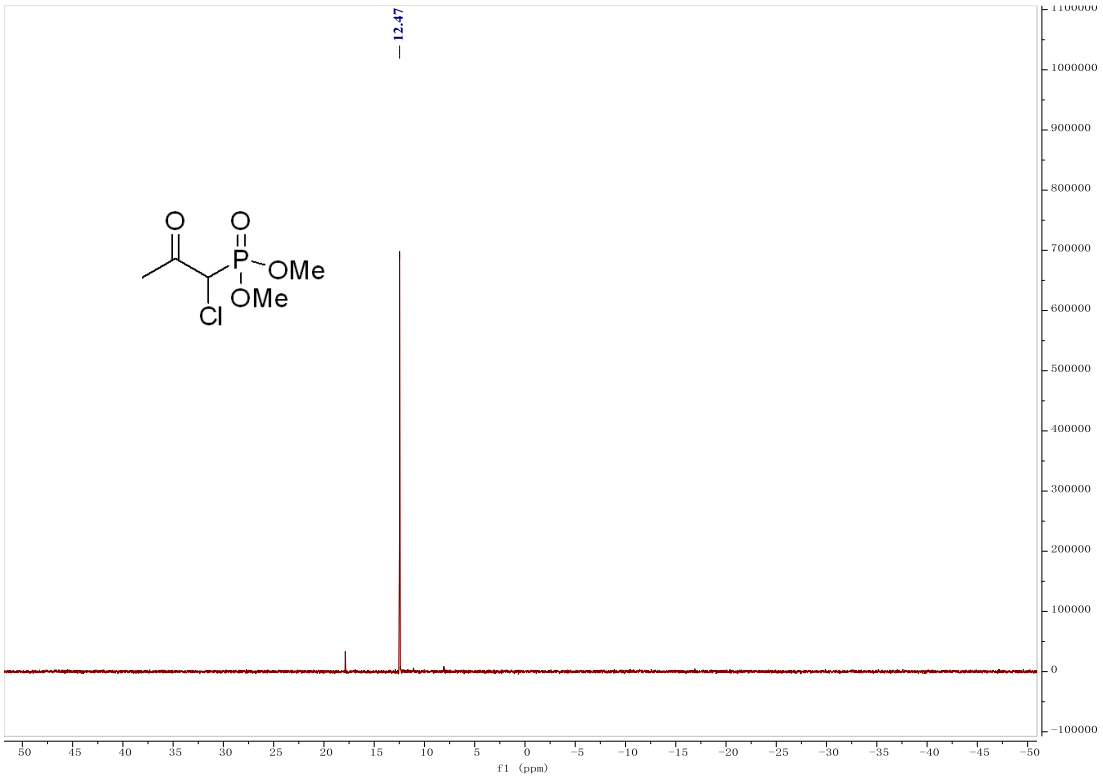
^{1}H NMR of **1b**, (400 MHz, Chloroform-*d*)



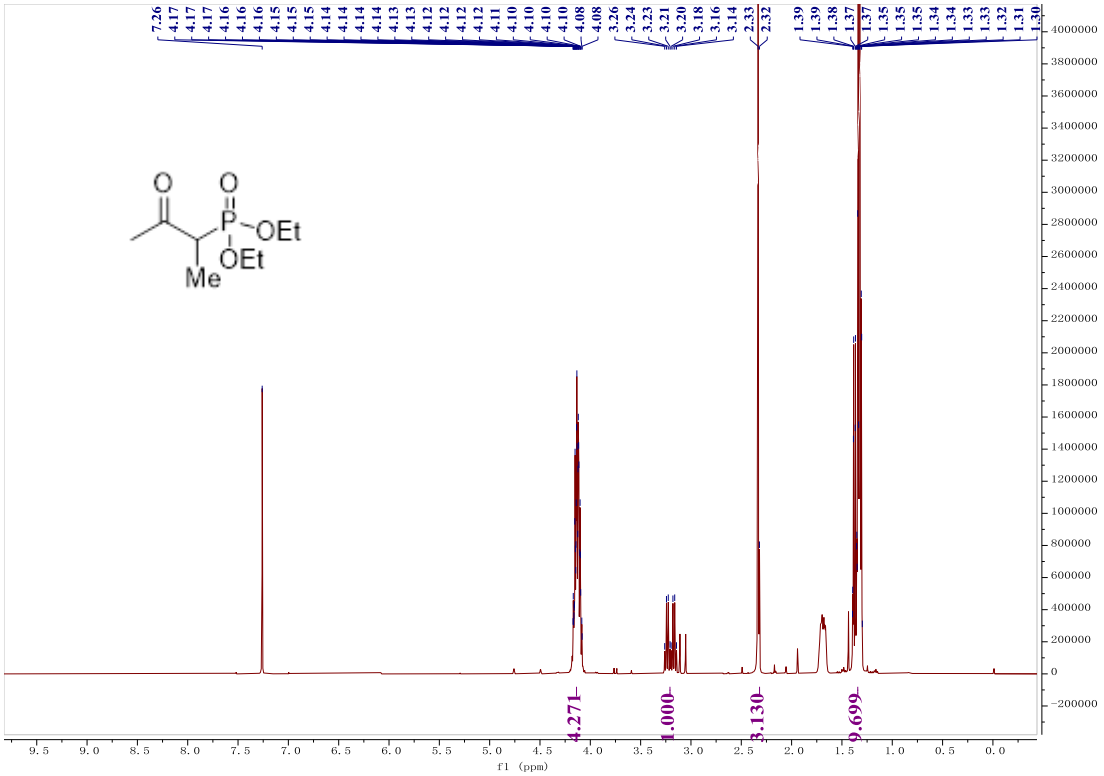
^{13}C NMR of **1b**, (101 MHz, Chloroform-*d*)



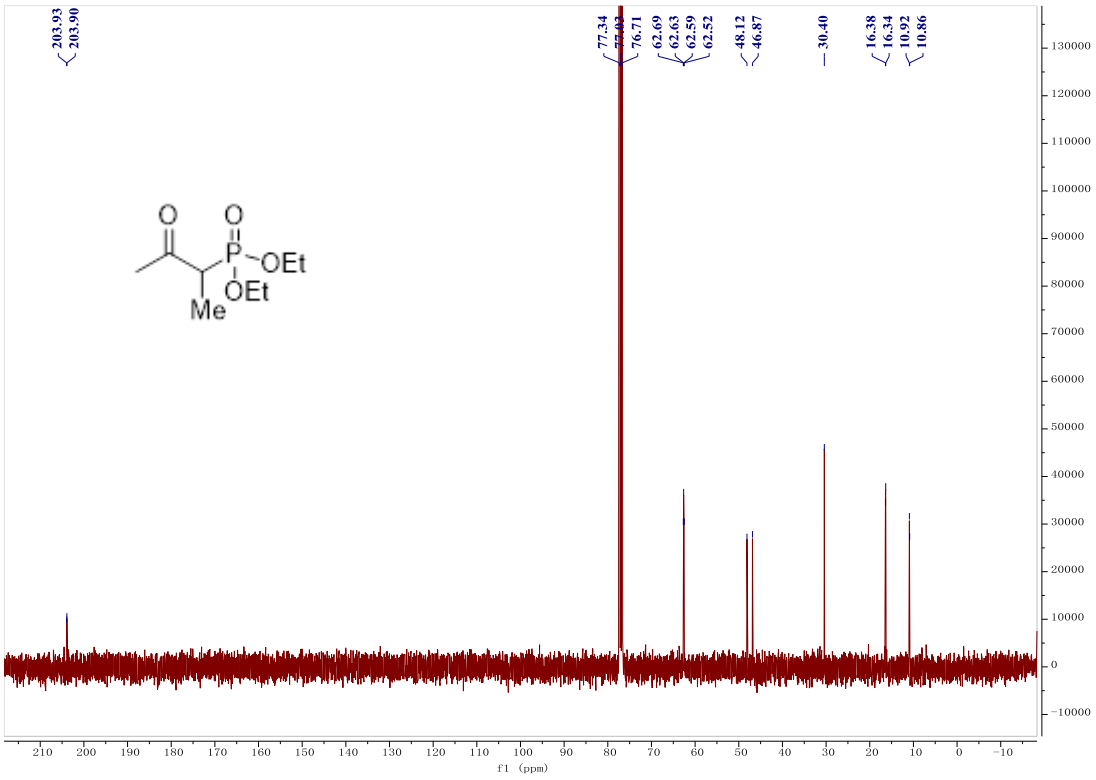
^{31}P NMR of **1b**, (162 MHz, Chloroform-*d*)



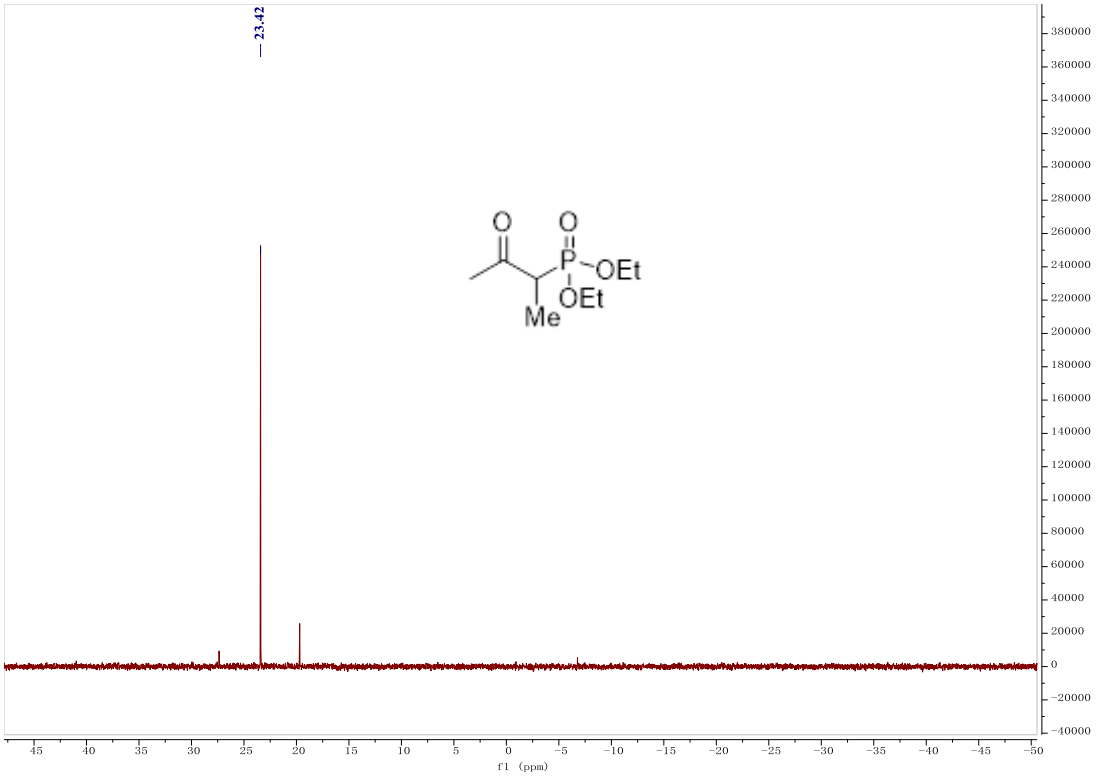
^{1}H NMR of 1**c**, (400 MHz, Chloroform-*d*)



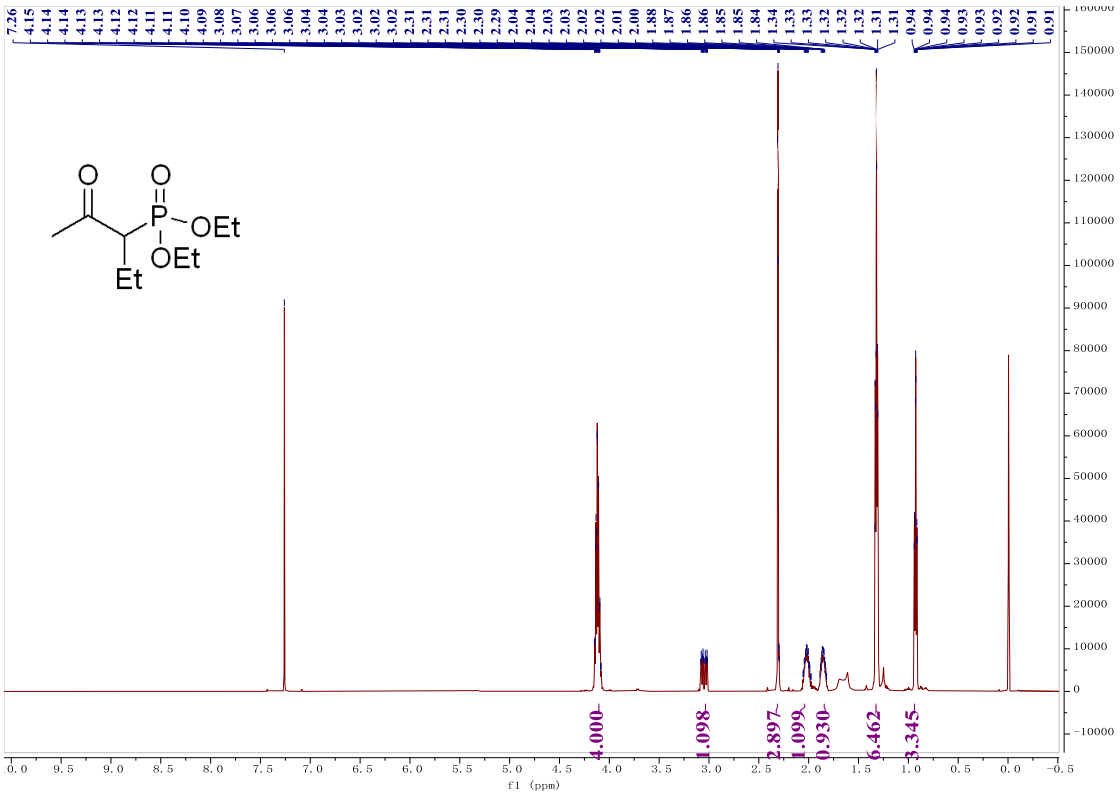
^{13}C NMR of 1**c**, (101 MHz, Chloroform-*d*)



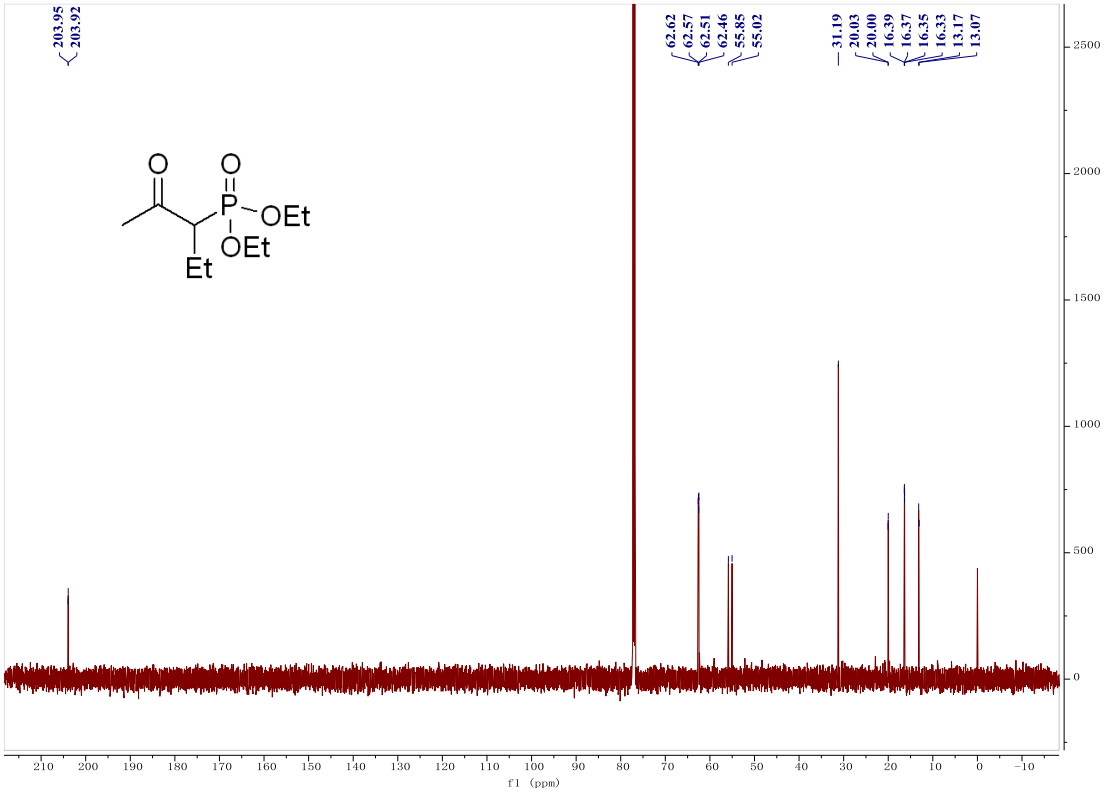
^{31}P NMR of 1**c**, (162 MHz, Chloroform-*d*)



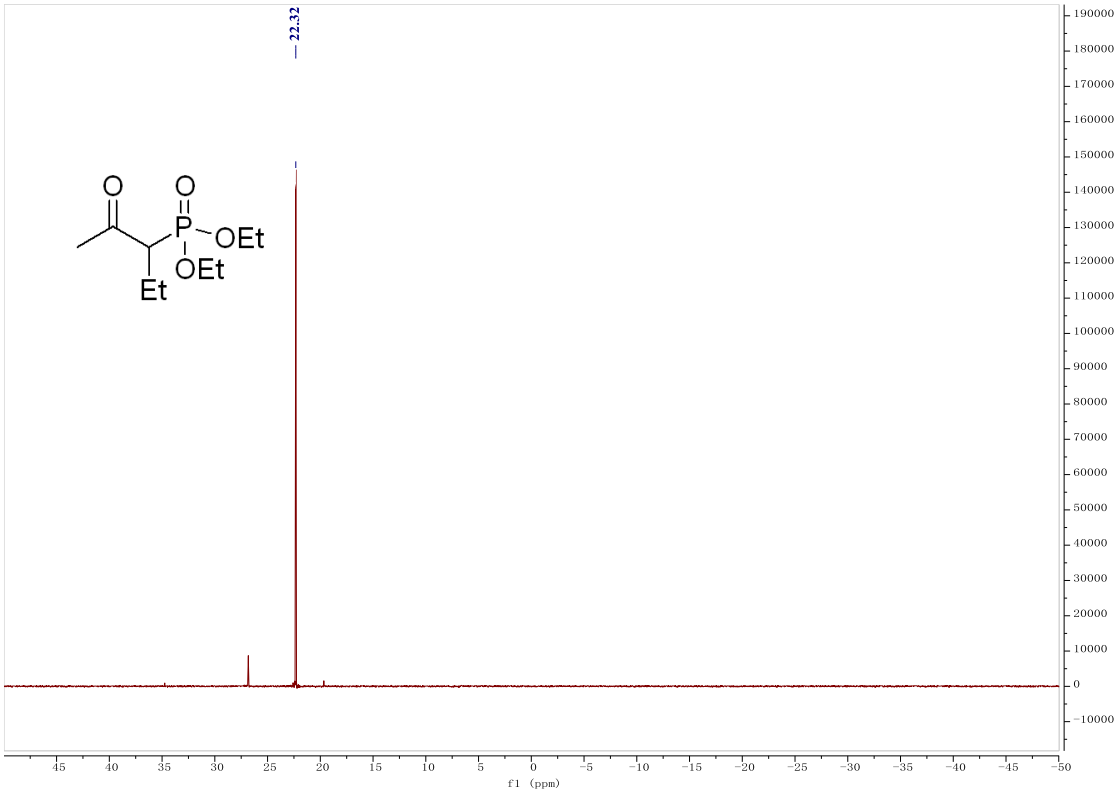
^{1}H NMR of **1d**, (400 MHz, Chloroform-*d*)



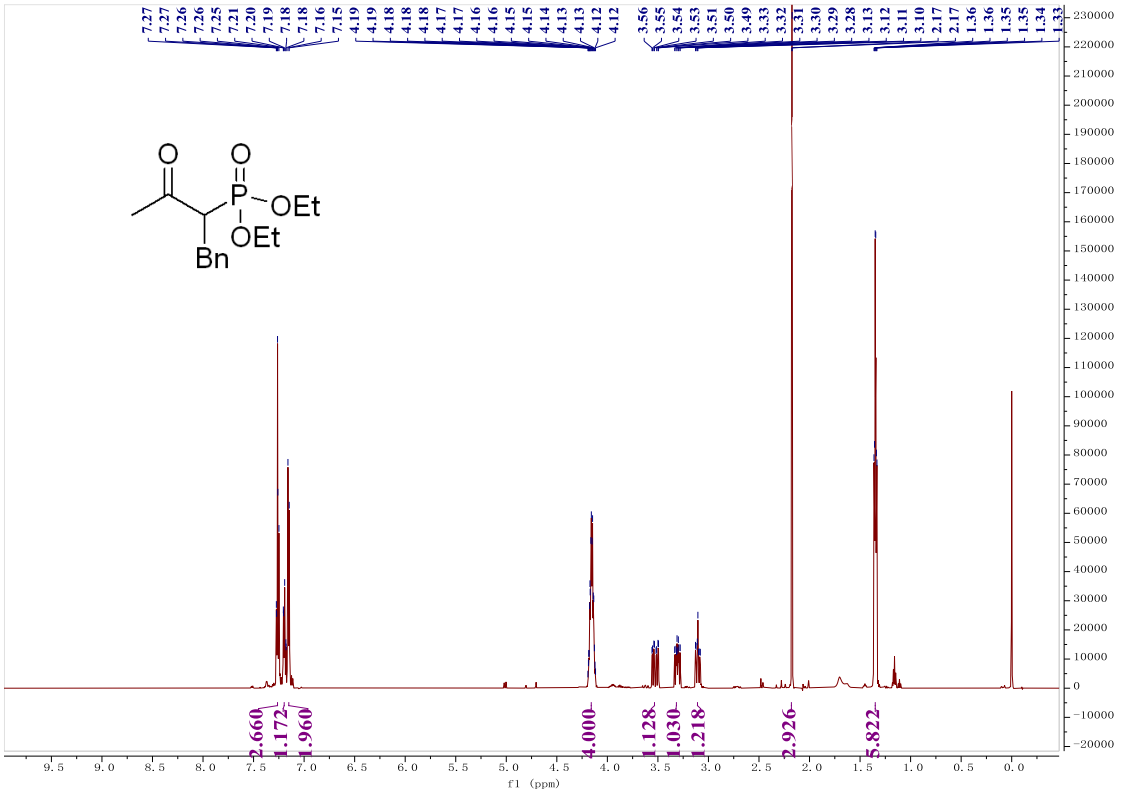
^{13}C NMR of **1d**, (101 MHz, Chloroform-*d*)



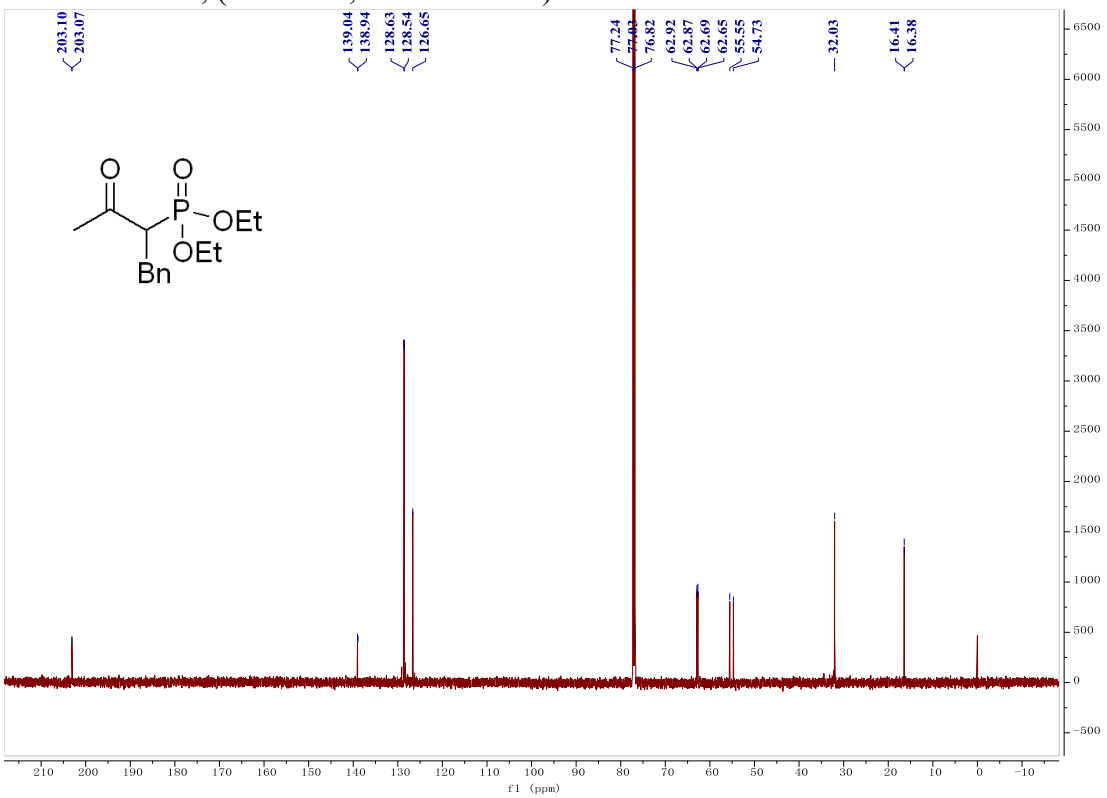
^{31}P NMR of **1d**, (162 MHz, Chloroform-*d*)



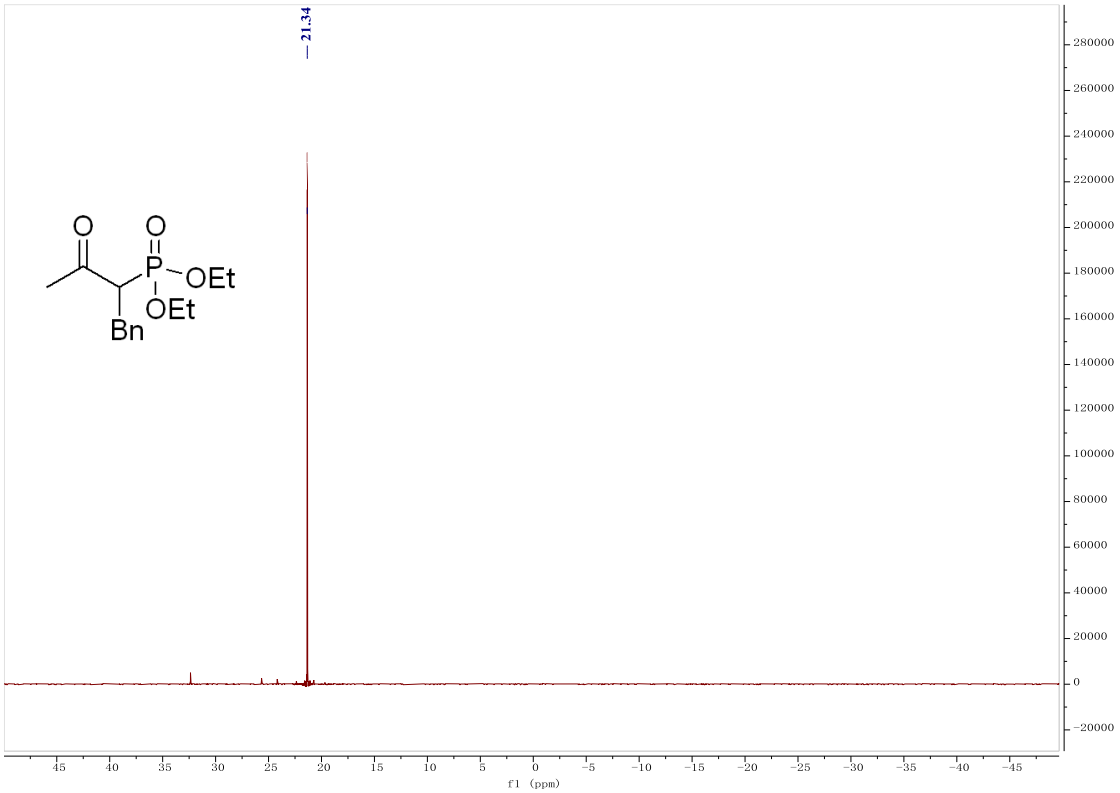
^{1}H NMR of **1e**, (400 MHz, Chloroform-*d*)



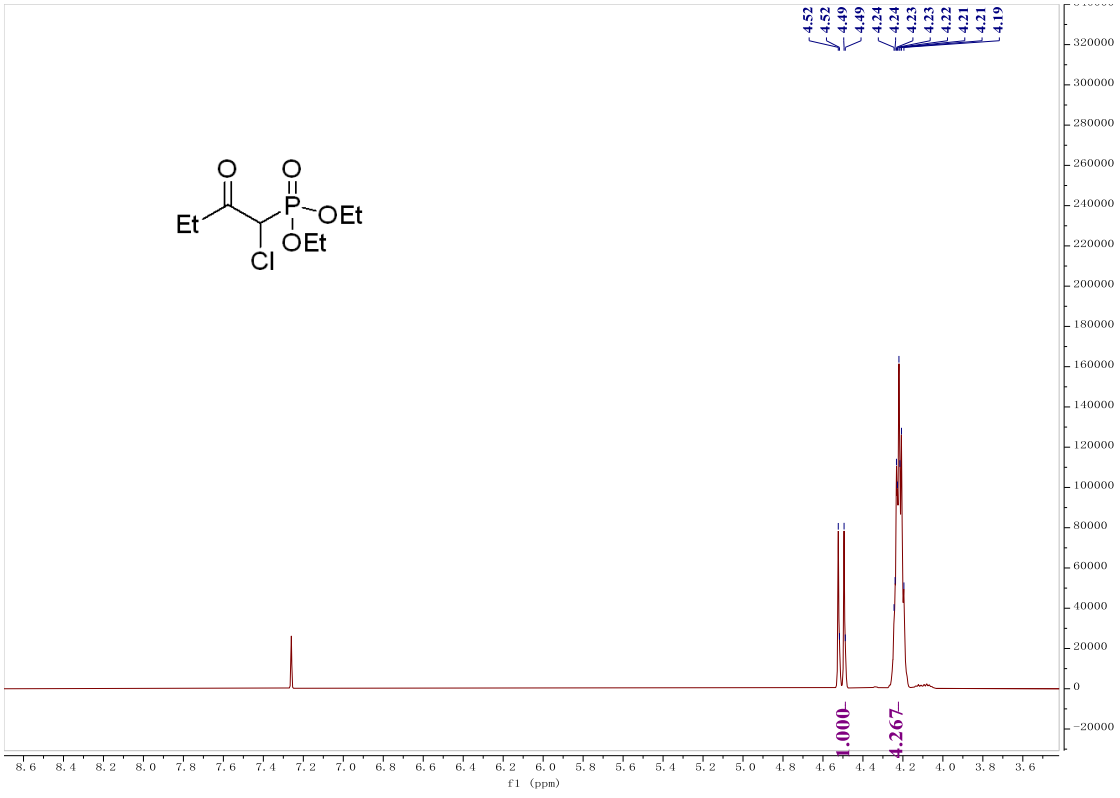
^{13}C NMR of **1e**, (101 MHz, Chloroform-*d*)



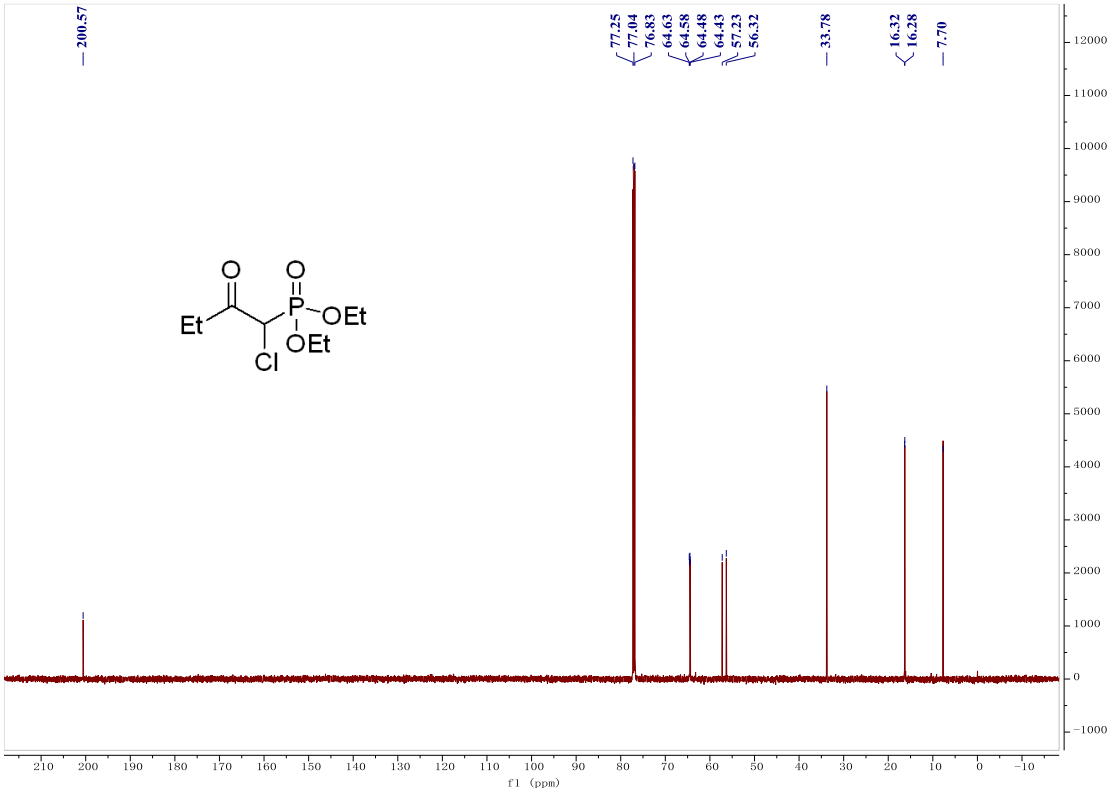
^{31}P NMR of **1e**, (162 MHz, Chloroform-*d*)



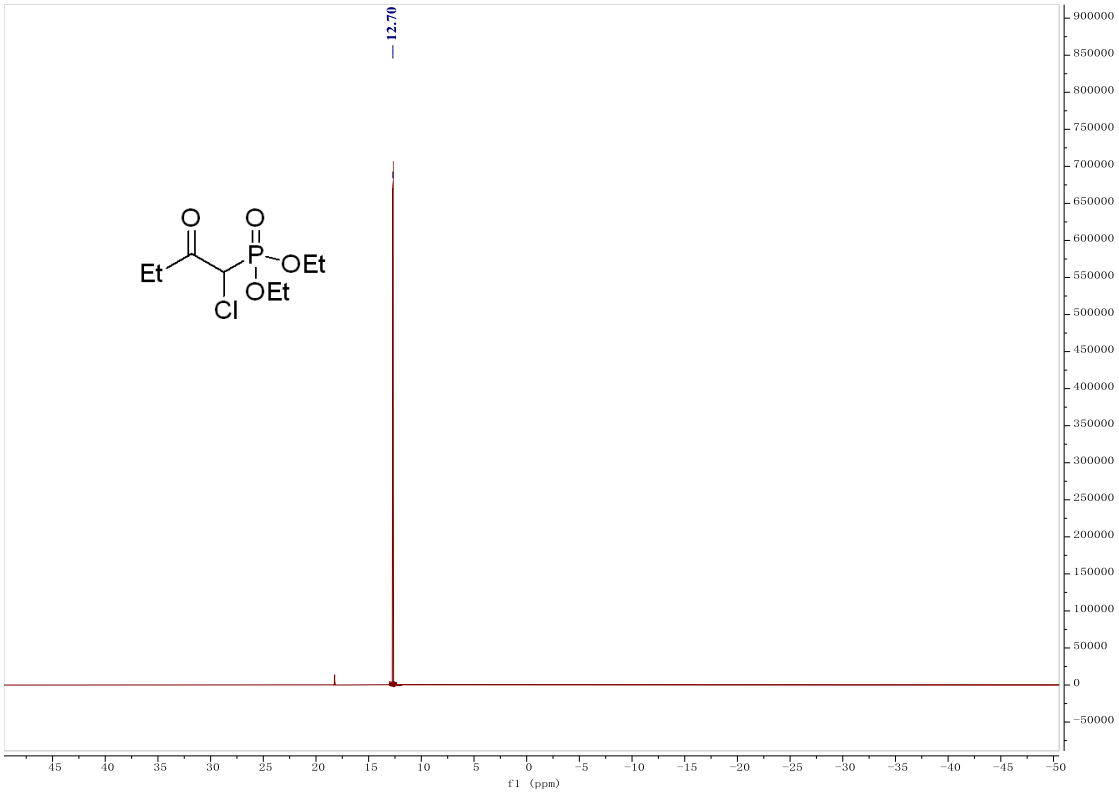
^{1}H NMR of **1f**, (400 MHz, Chloroform-*d*)



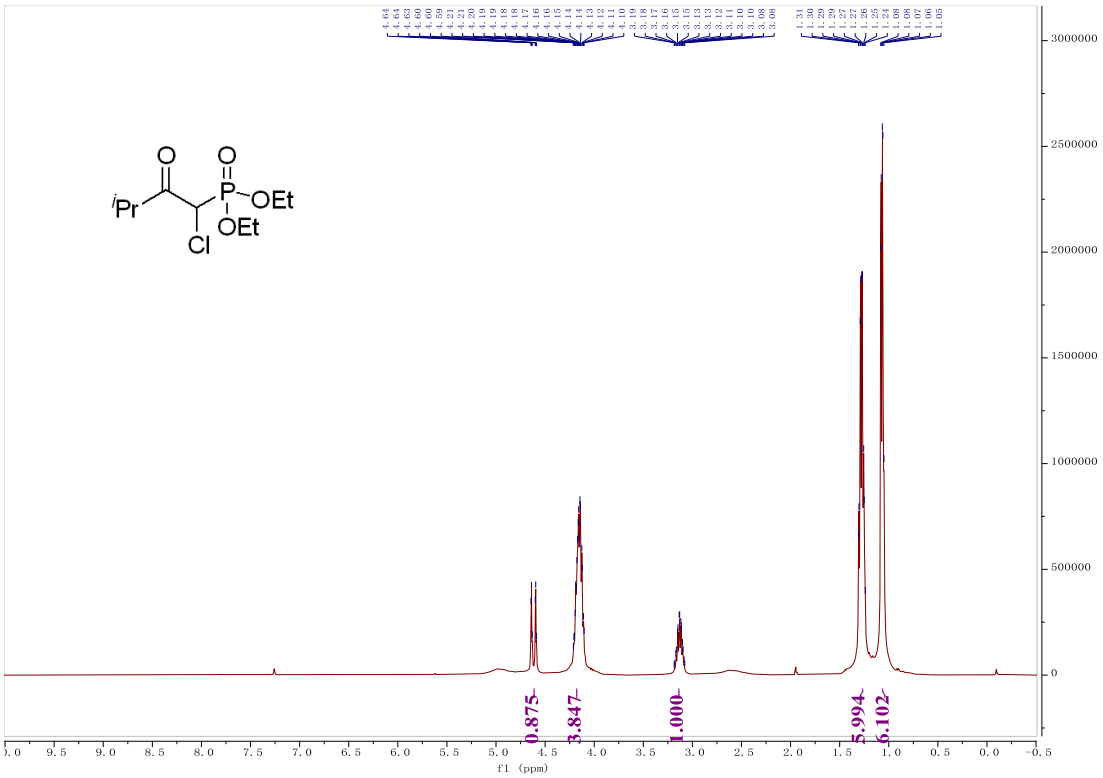
^{13}C NMR of **1f**, (101 MHz, Chloroform-*d*)



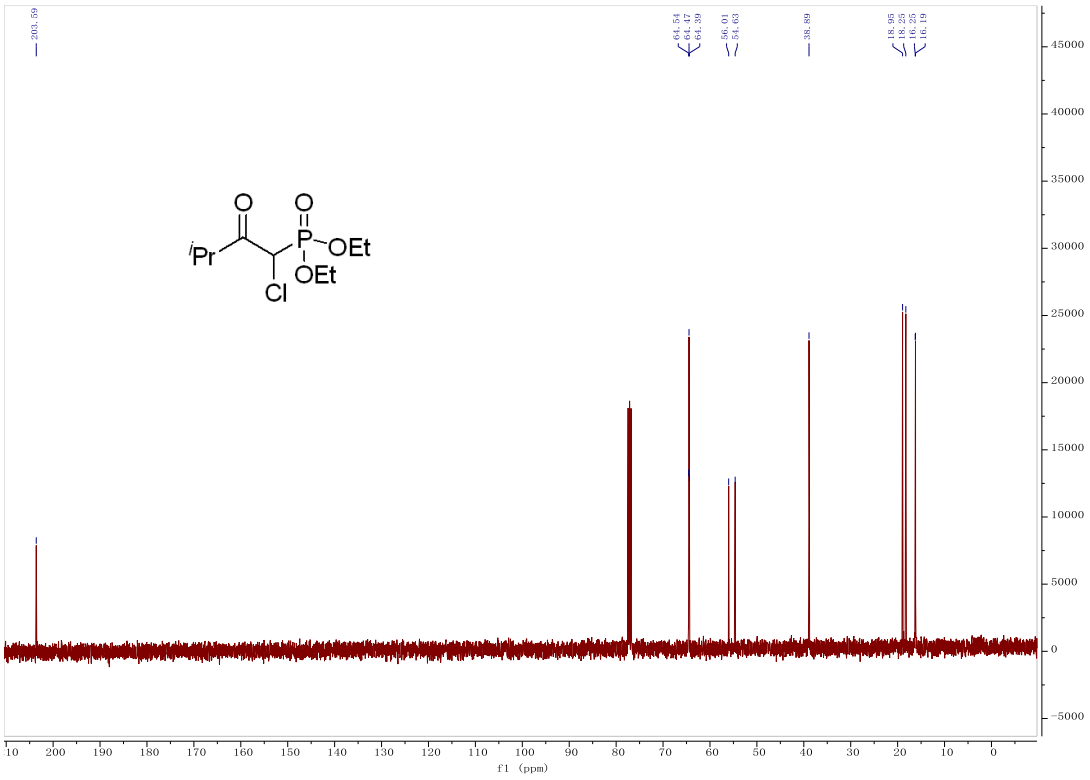
^{31}P NMR of **1f**, (162 MHz, Chloroform-*d*)



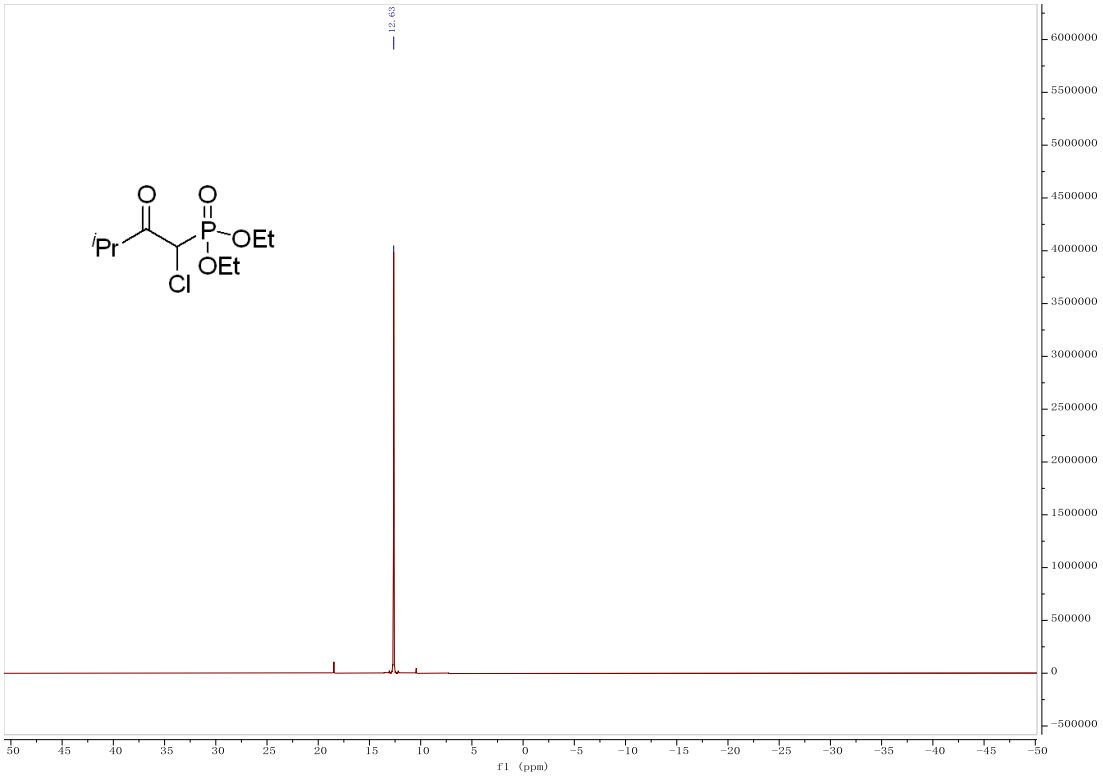
^{1}H NMR of **1g**



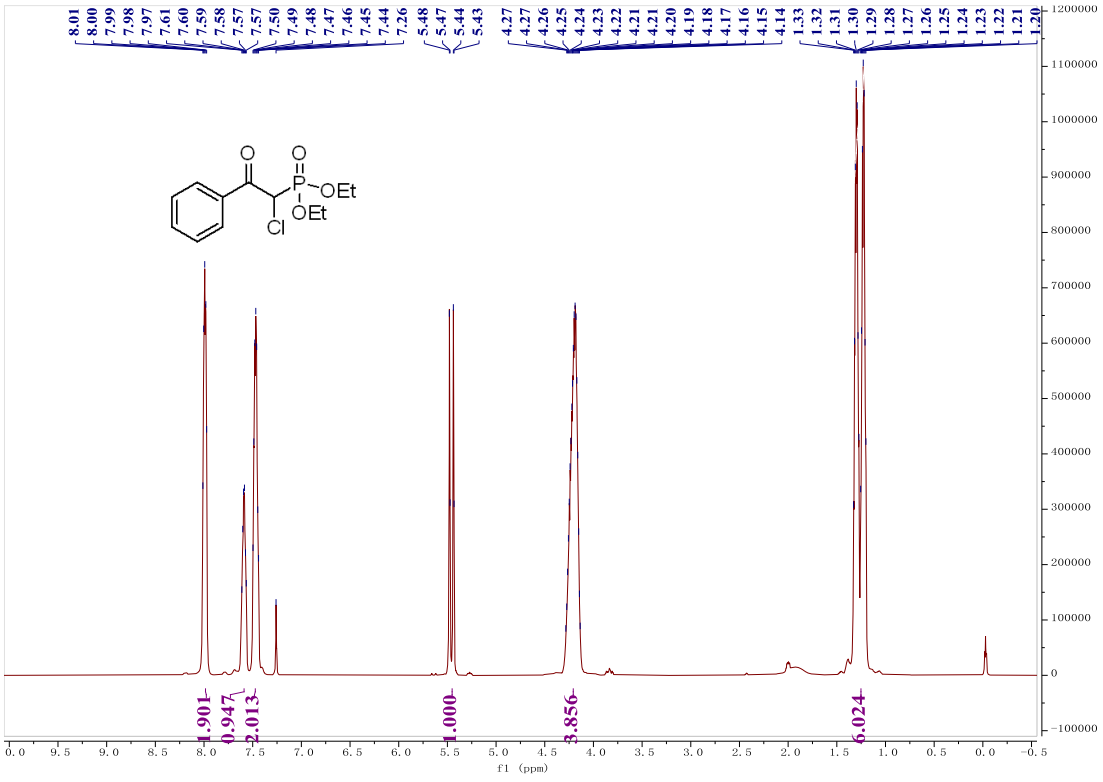
^{13}C NMR of **1g**



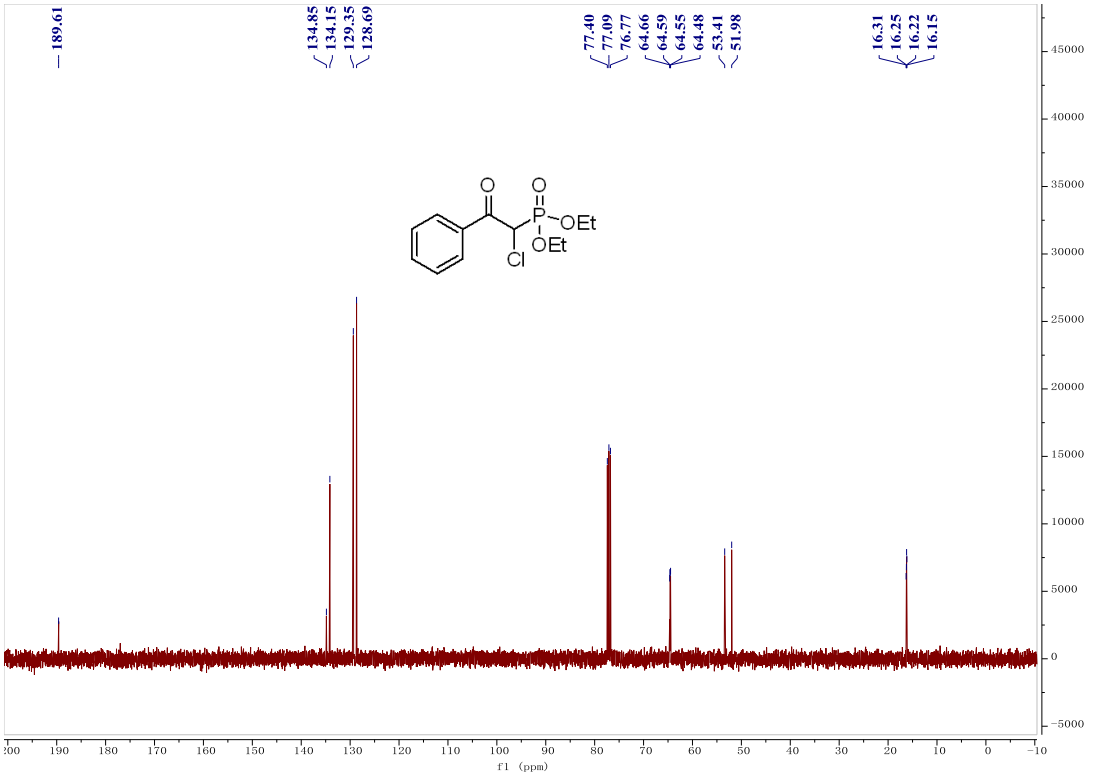
^{31}P NMR of **1g**



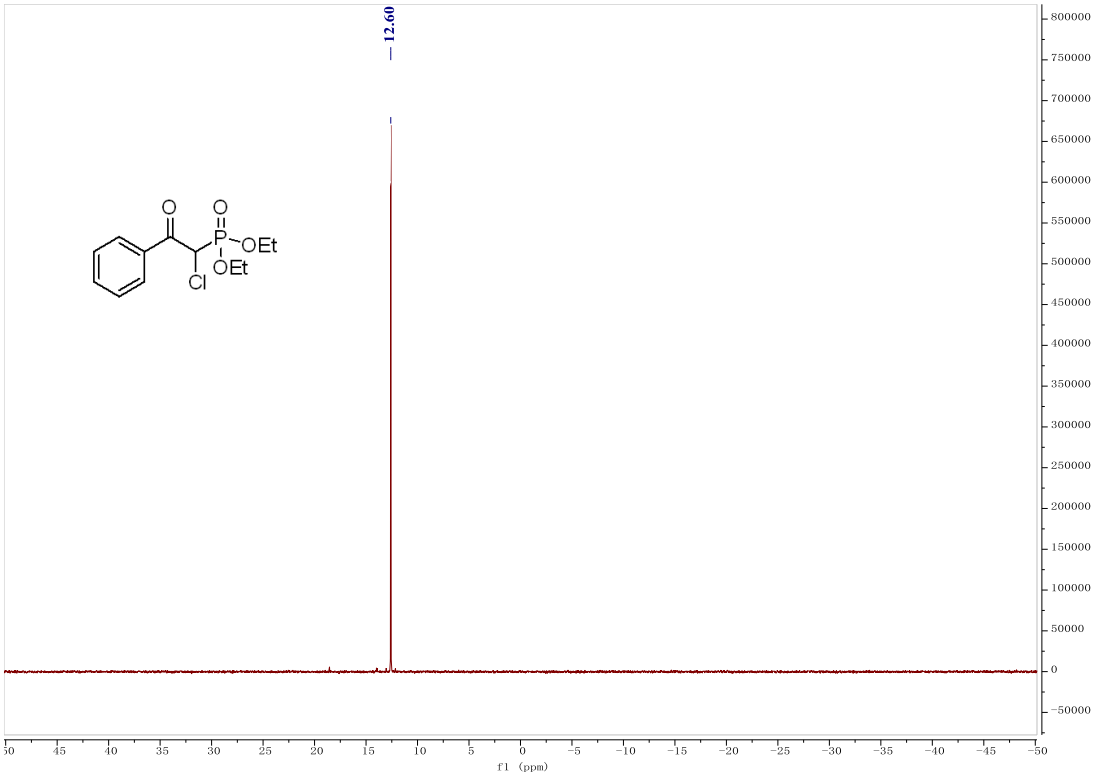
^{1}H NMR of **1h**, (400 MHz, Chloroform-*d*)



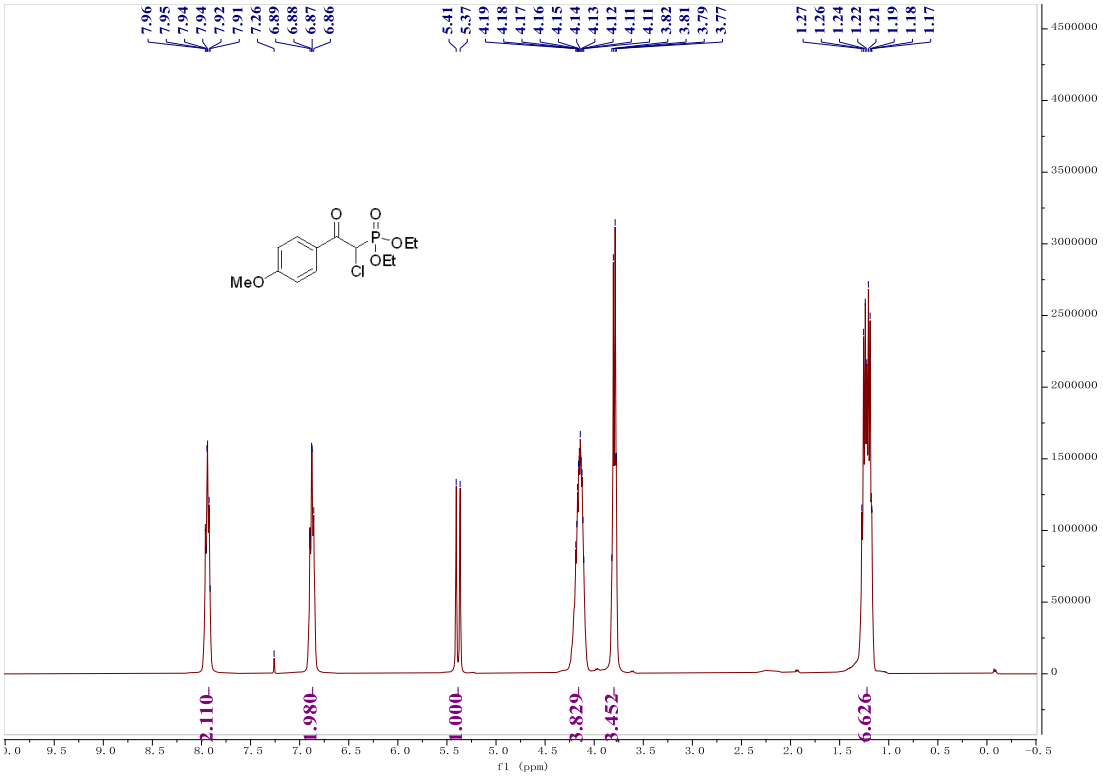
^{13}C NMR of **1h**, (101 MHz, Chloroform-*d*)



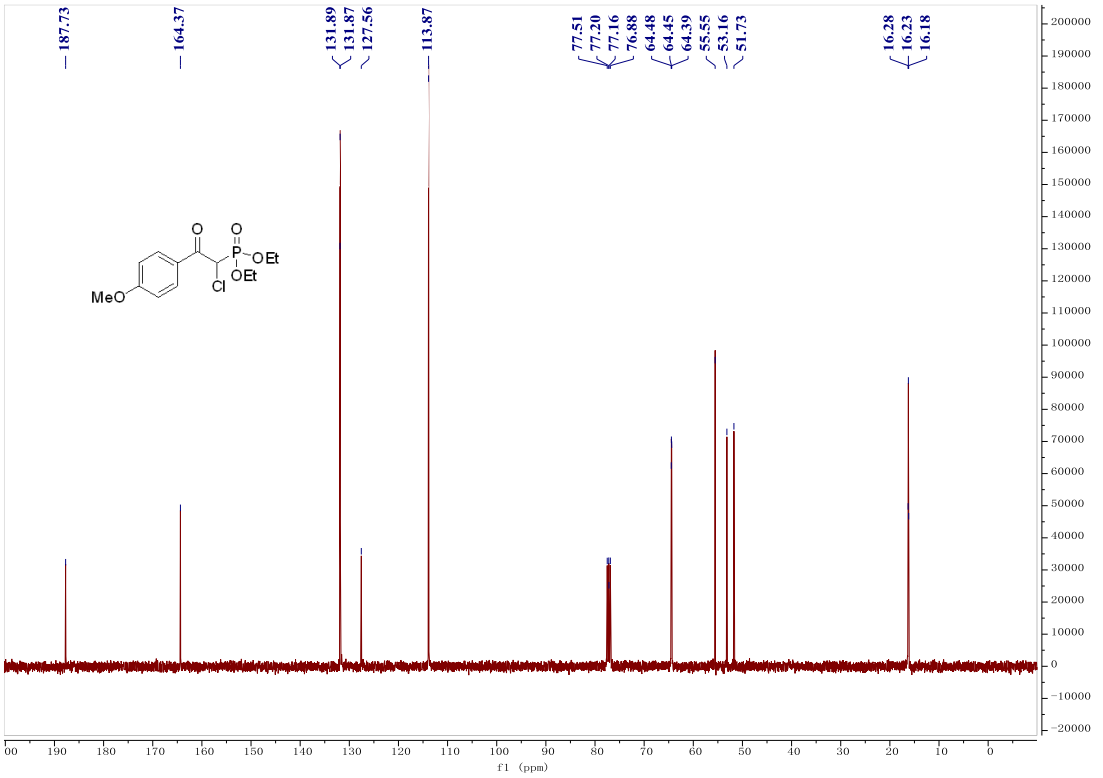
^{31}P NMR of **1h**, (162 MHz, Chloroform-*d*)



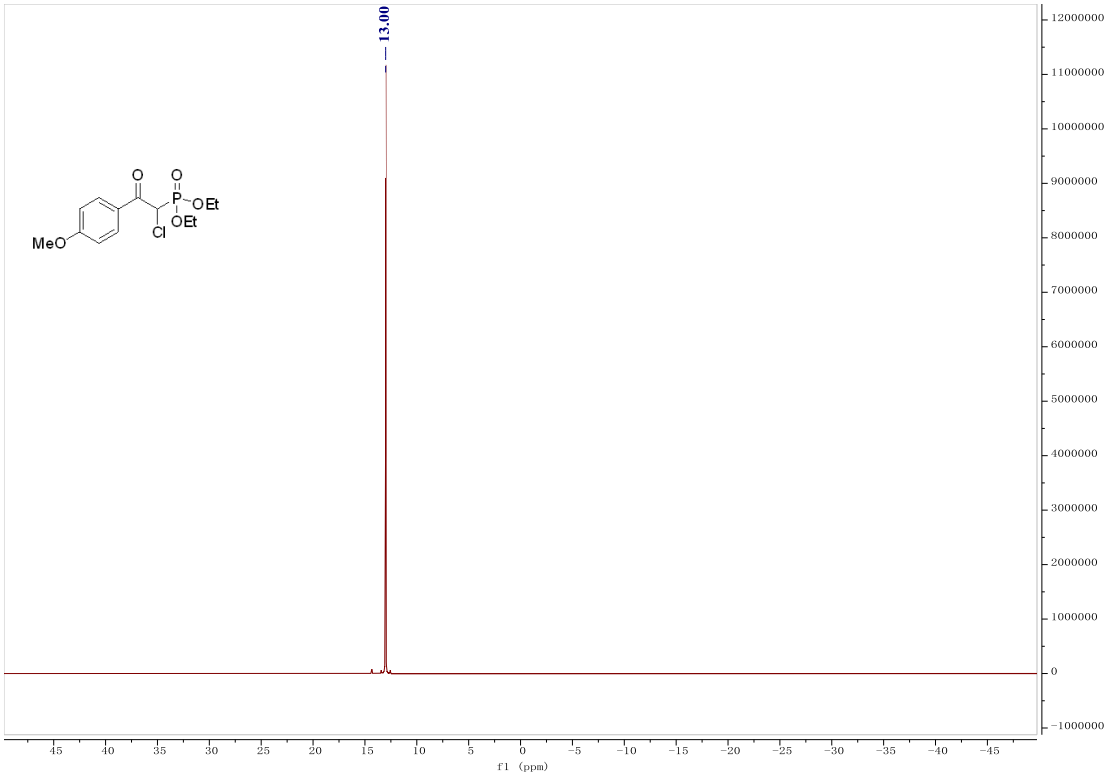
^{1}H NMR of **1i**, (400 MHz, Chloroform-*d*)



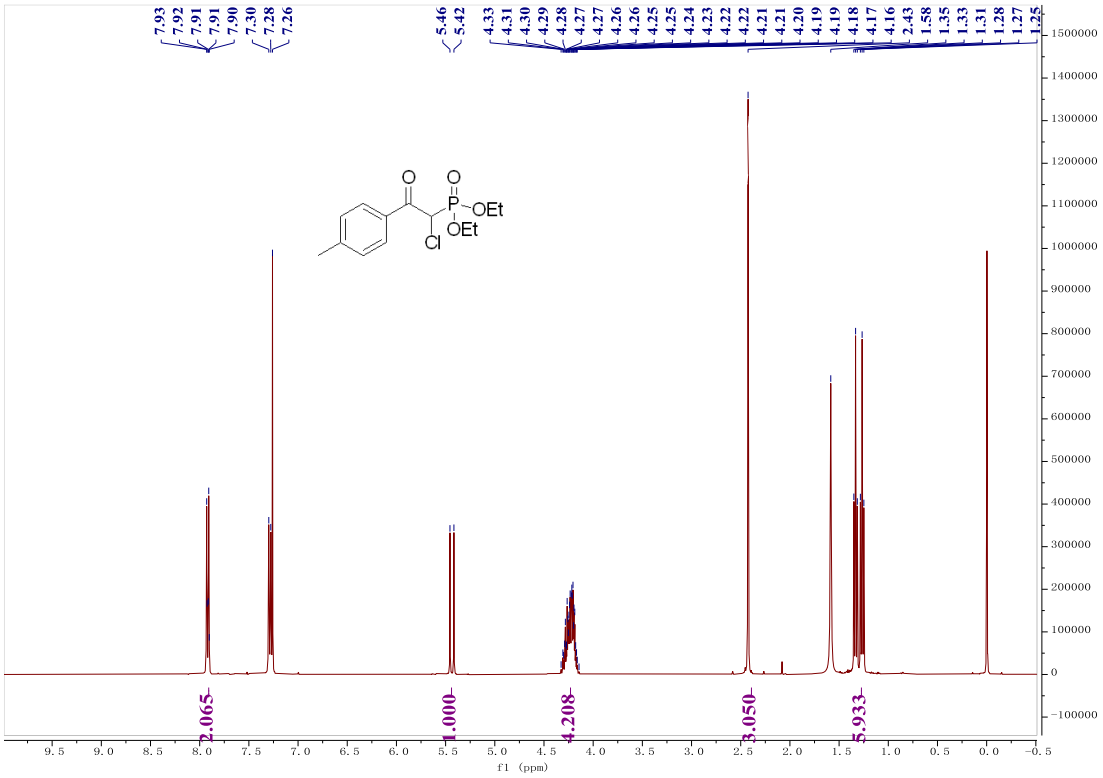
^{13}C NMR of **1i**, (101 MHz, Chloroform-*d*)



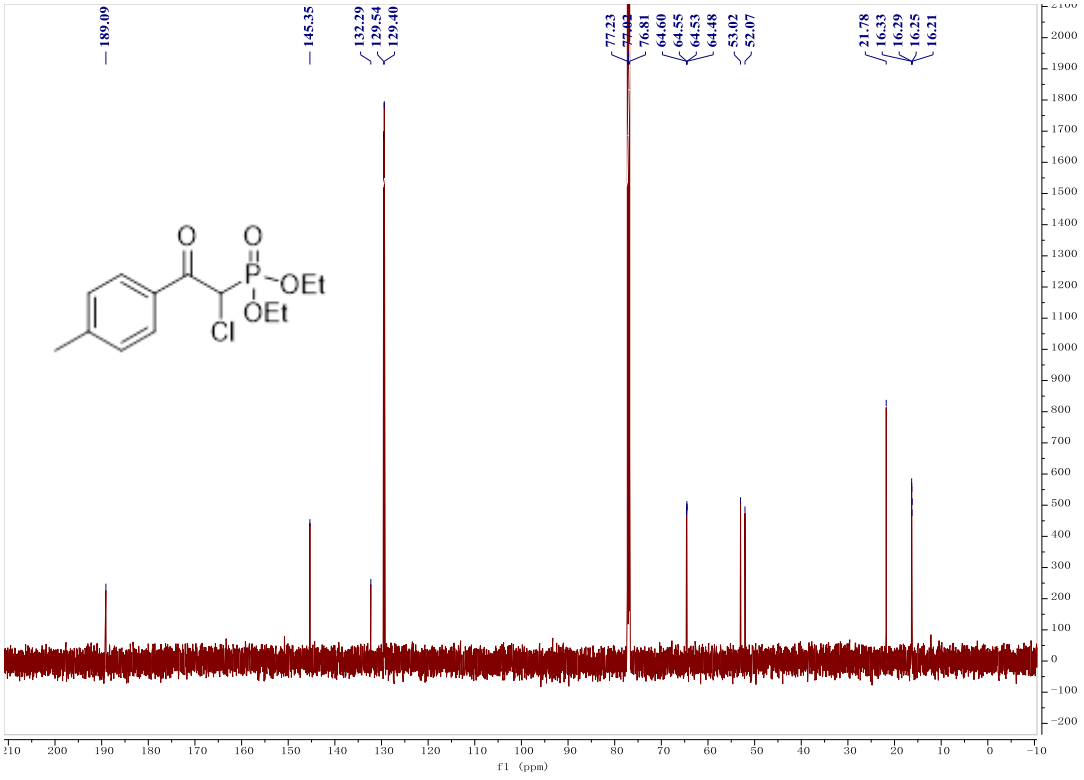
^{31}P NMR of **1i**, (162 MHz, Chloroform-*d*)



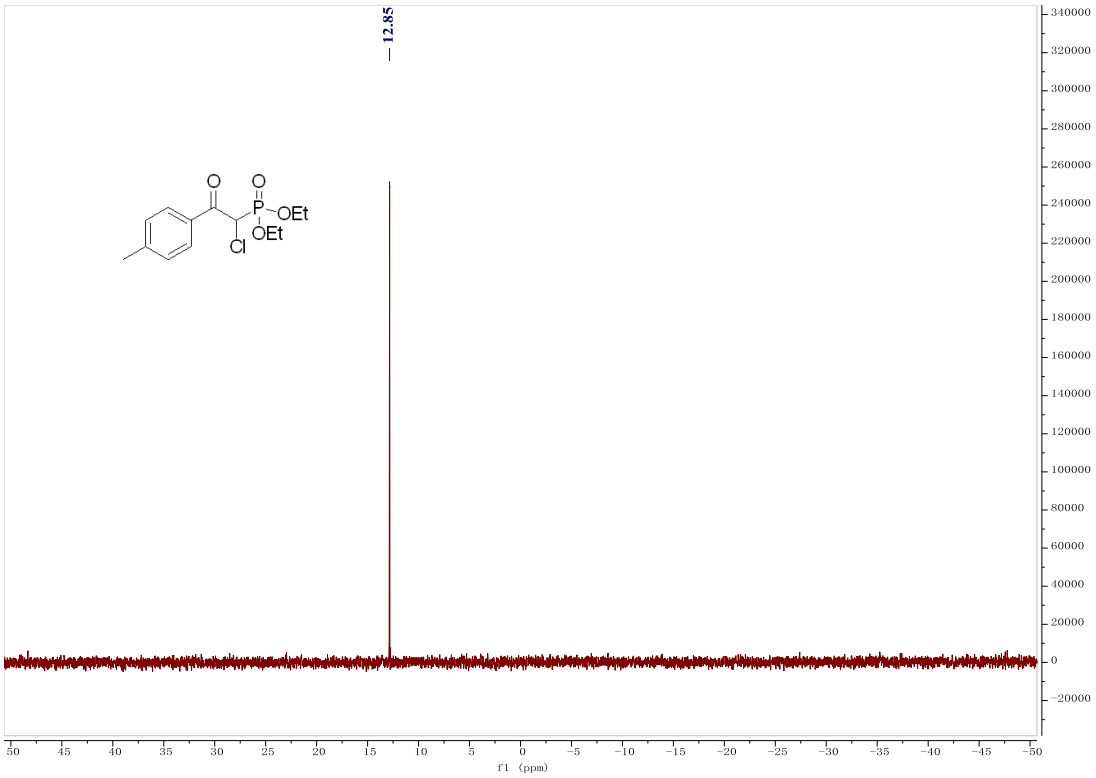
^{1}H NMR of **1j**, (400 MHz, Chloroform-*d*)



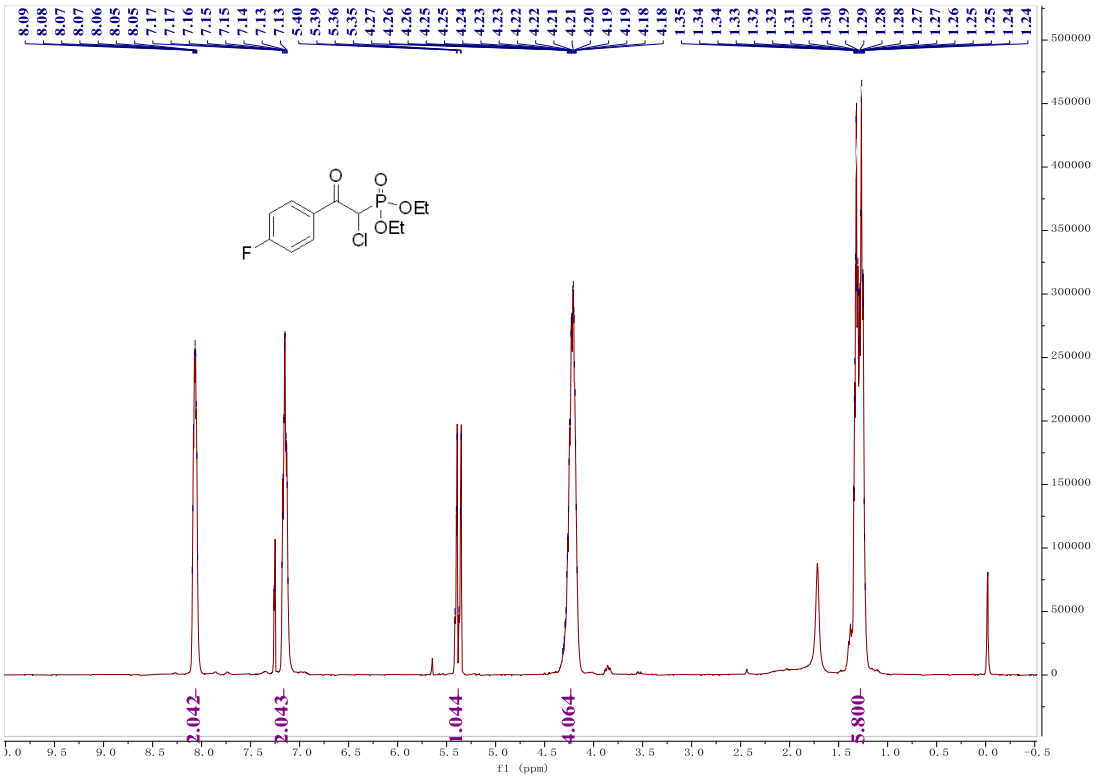
^{13}C NMR of **1j**, (101 MHz, Chloroform-*d*)



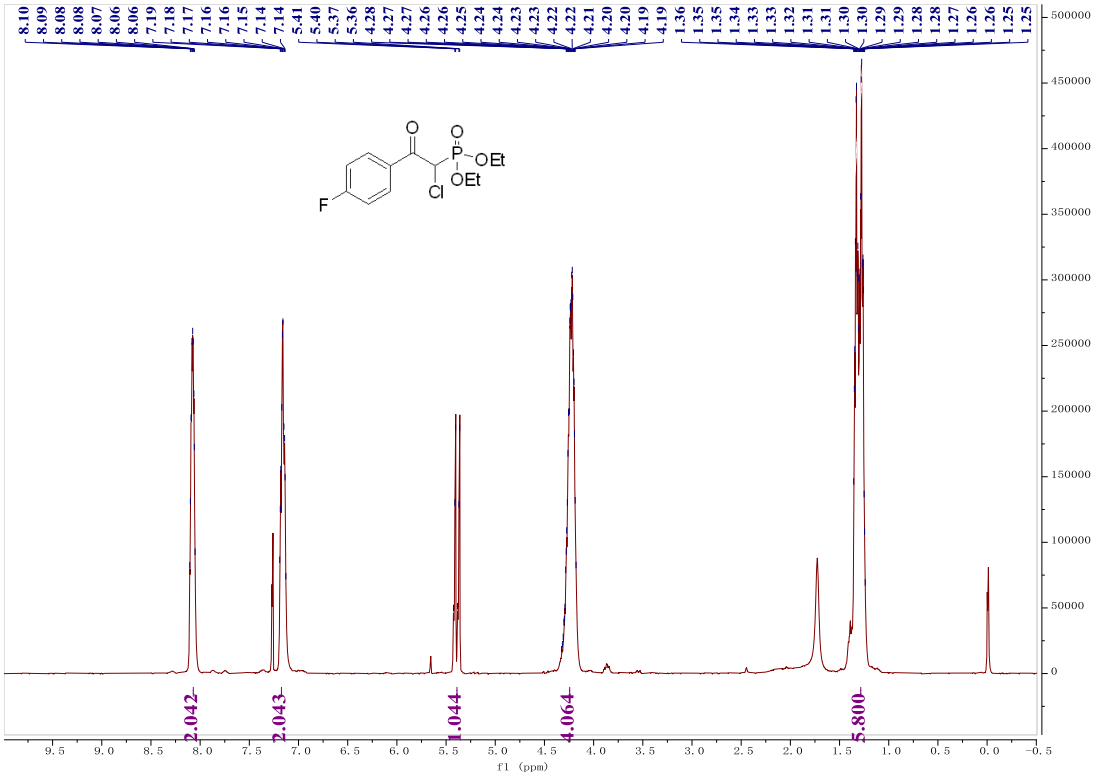
^{31}P NMR of **1j**, (162 MHz, Chloroform-*d*)



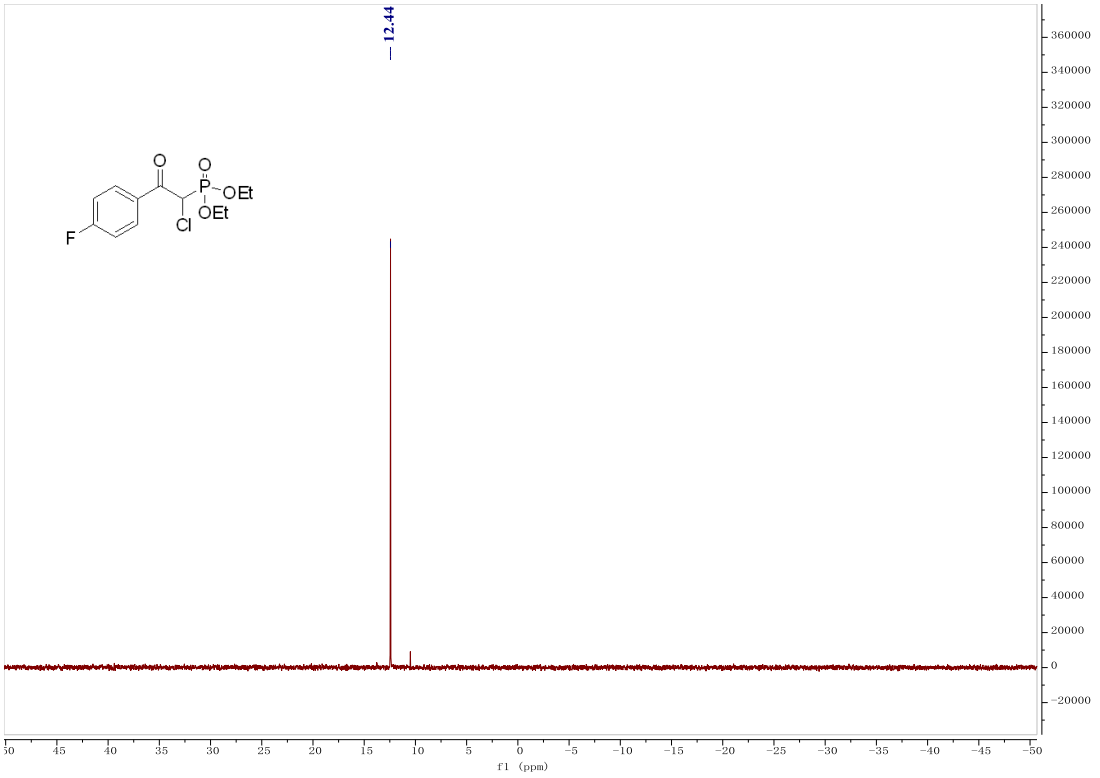
^{1}H NMR of **1k**, (400 MHz, Chloroform-*d*)



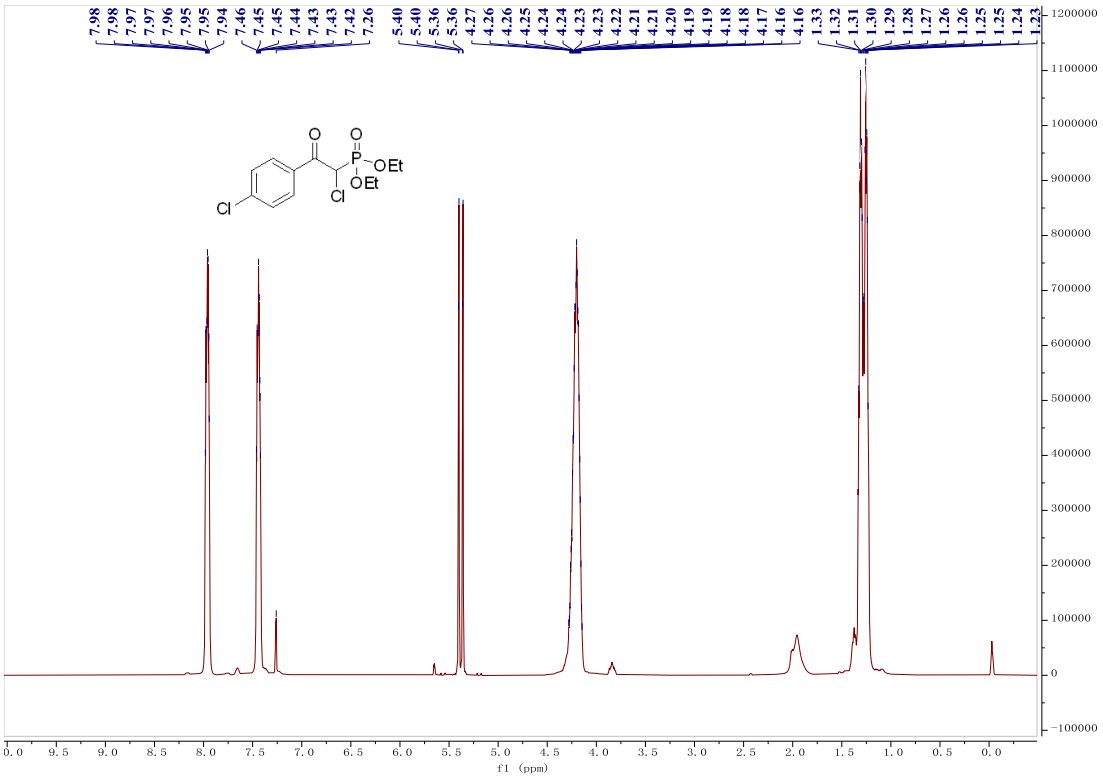
^{13}C NMR of **1k**, (101 MHz, Chloroform-*d*)



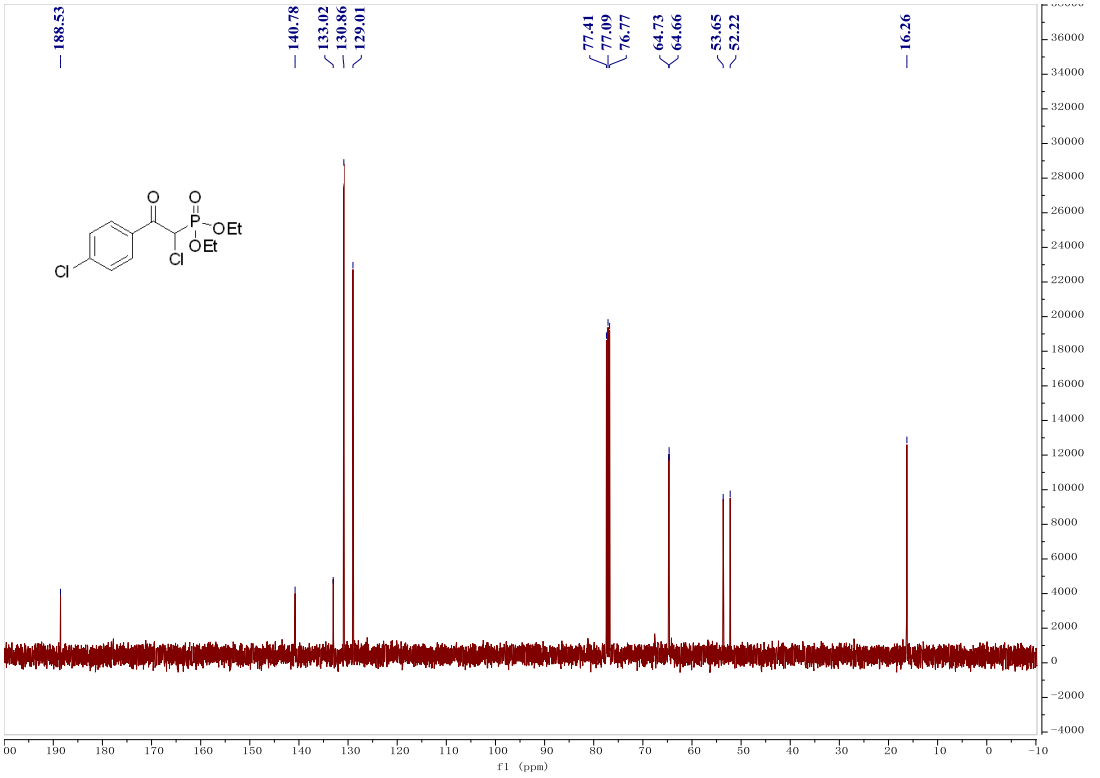
^{31}P NMR of **1k**, (162 MHz, Chloroform-*d*)



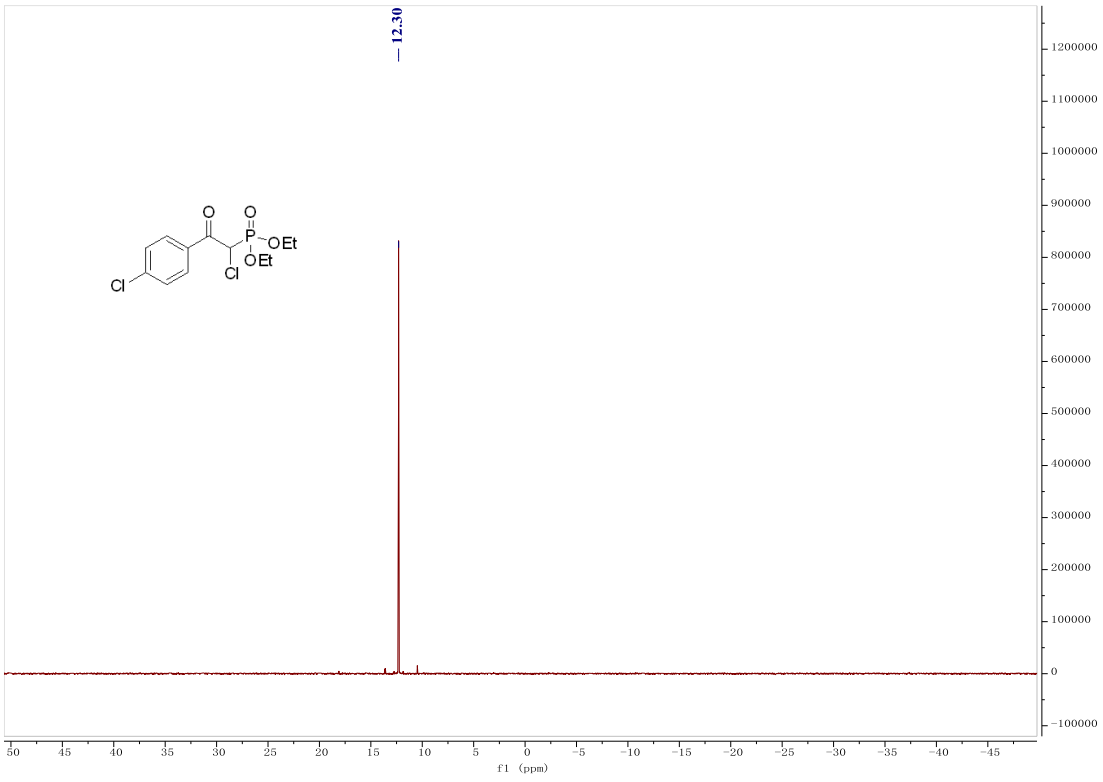
^{1}H NMR of **1l**, (400 MHz, Chloroform-*d*)



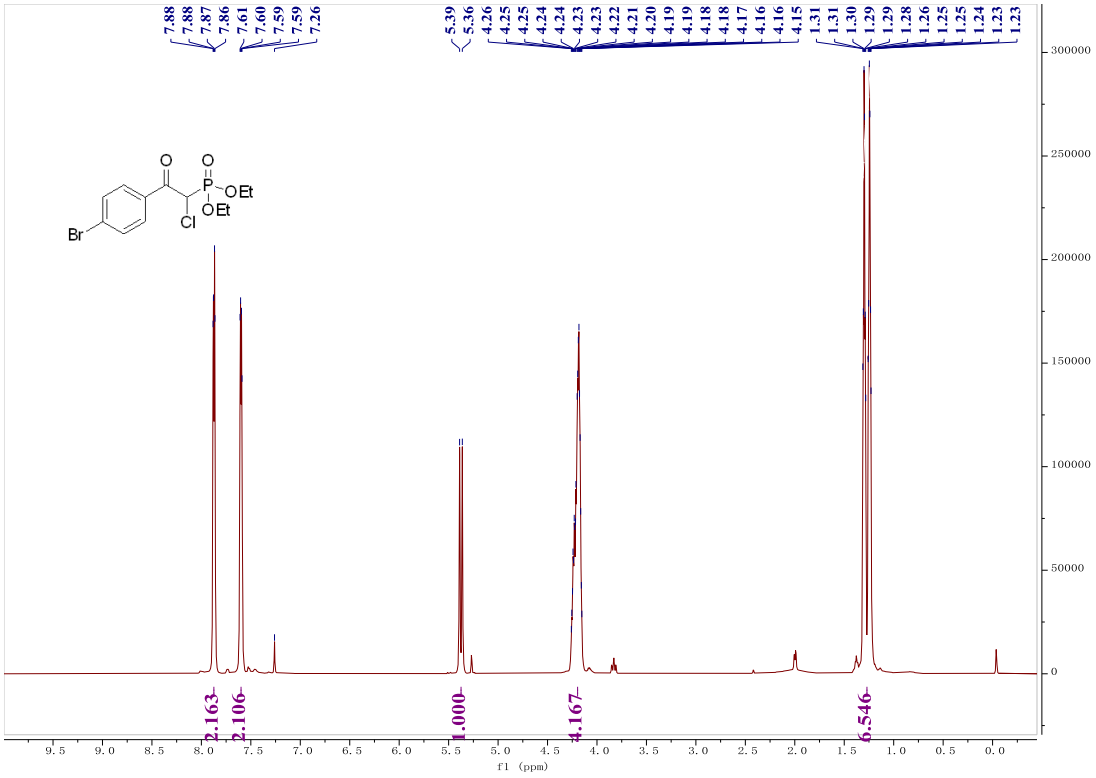
^{13}C NMR of **1l**, (101 MHz, Chloroform-*d*)



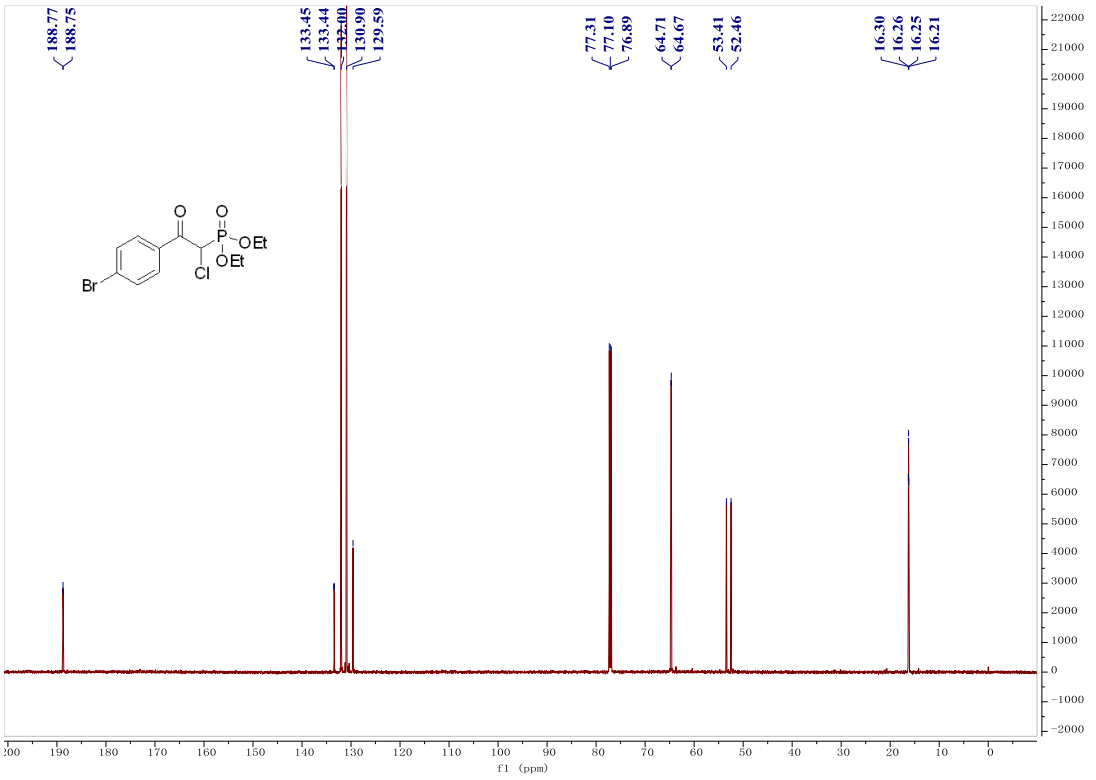
^{31}P NMR of **1l**, (162 MHz, Chloroform-*d*)



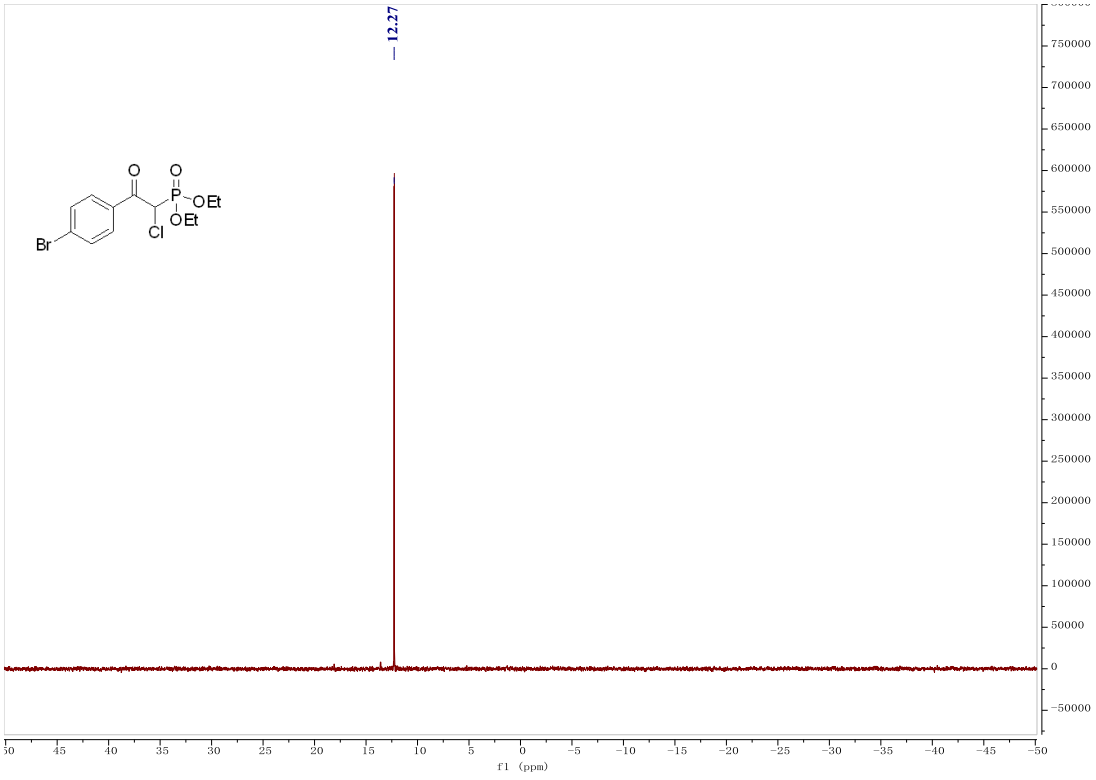
^{1}H NMR of **1m**, (400 MHz, Chloroform-*d*)



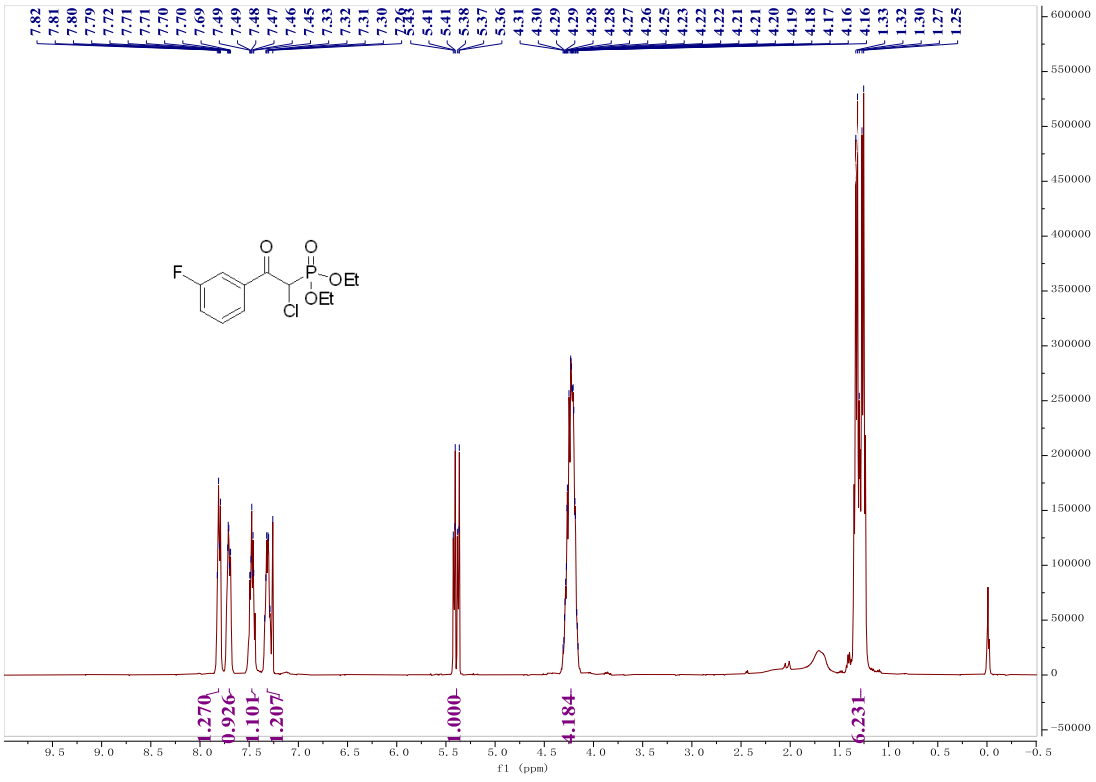
^{13}C NMR of **1m**, (101 MHz, Chloroform-*d*)



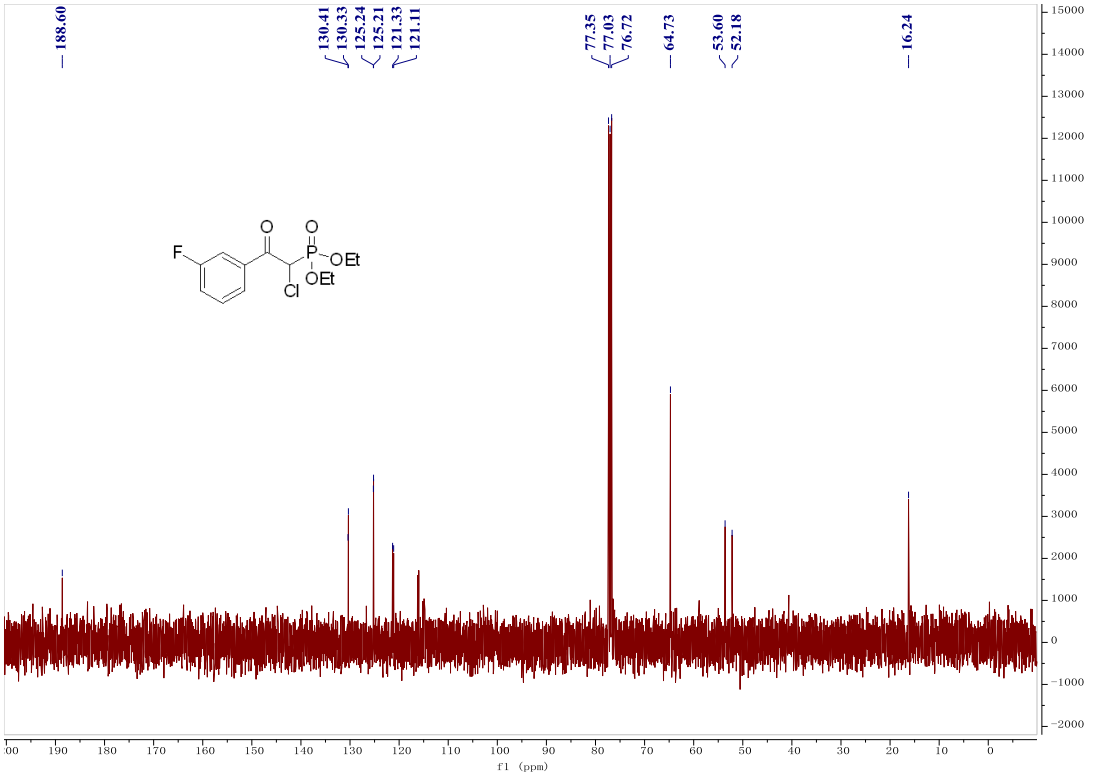
^{31}P NMR of **1m**, (162 MHz, Chloroform-*d*)



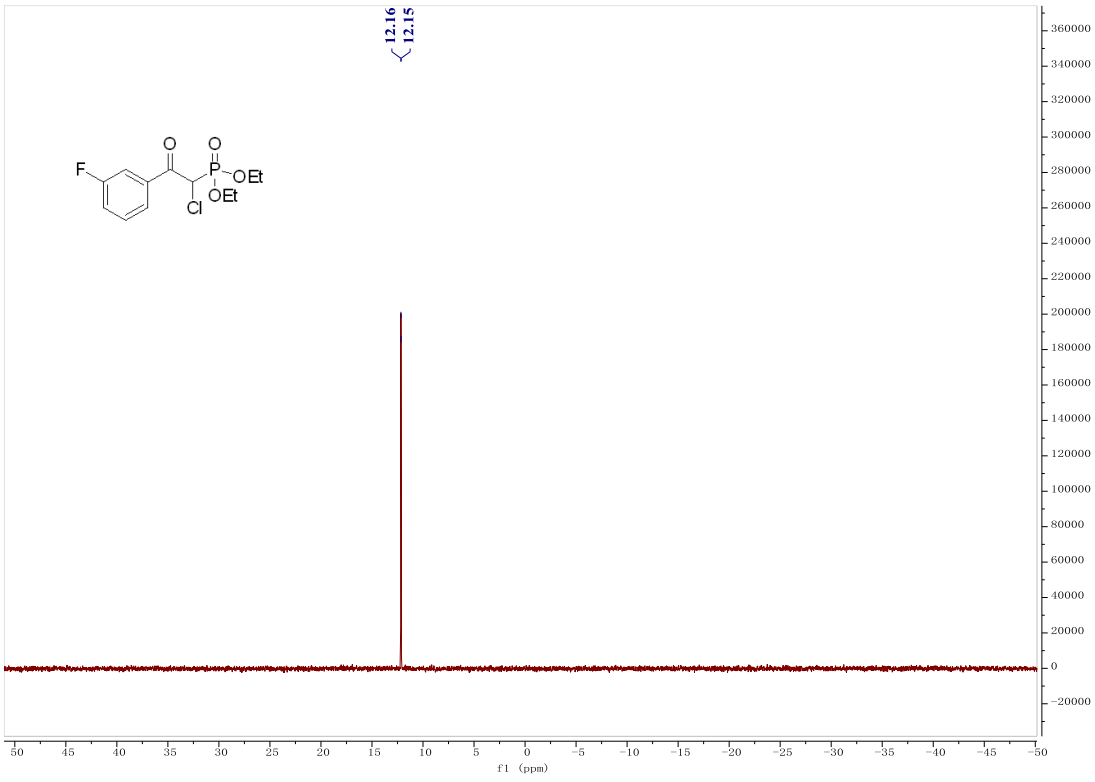
^{1}H NMR of **1n**, (400 MHz, Chloroform-*d*)



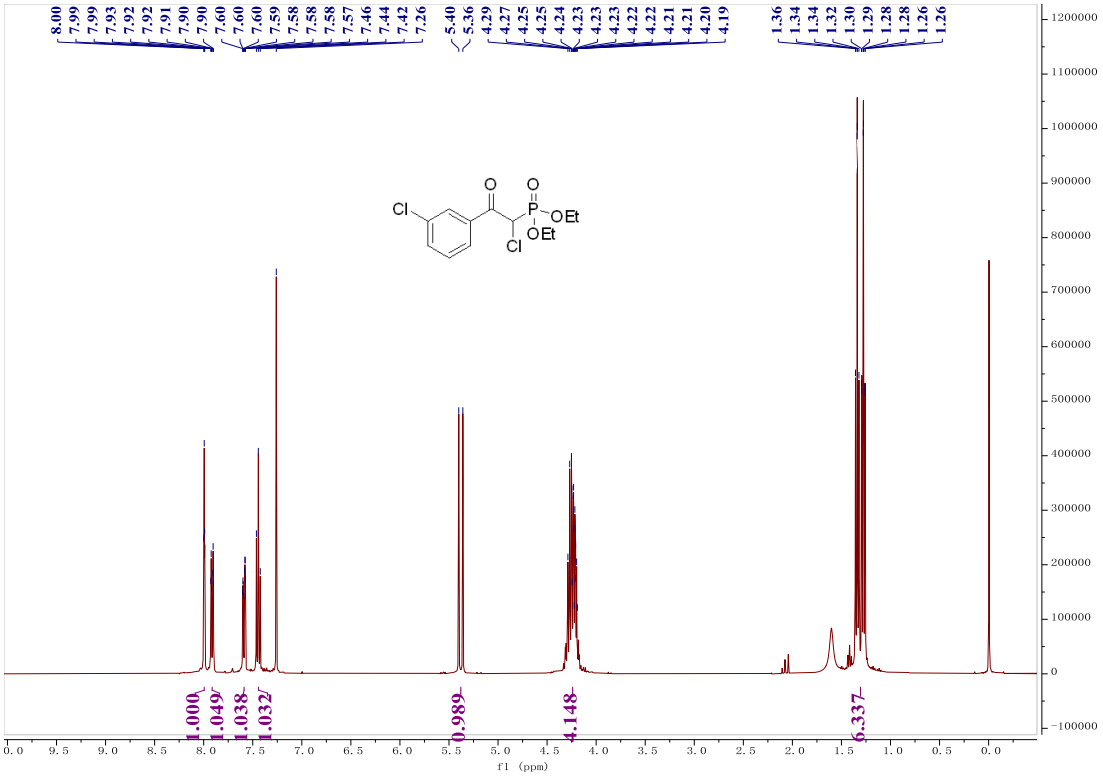
^{13}C NMR of **1n**, (101 MHz, Chloroform-*d*)



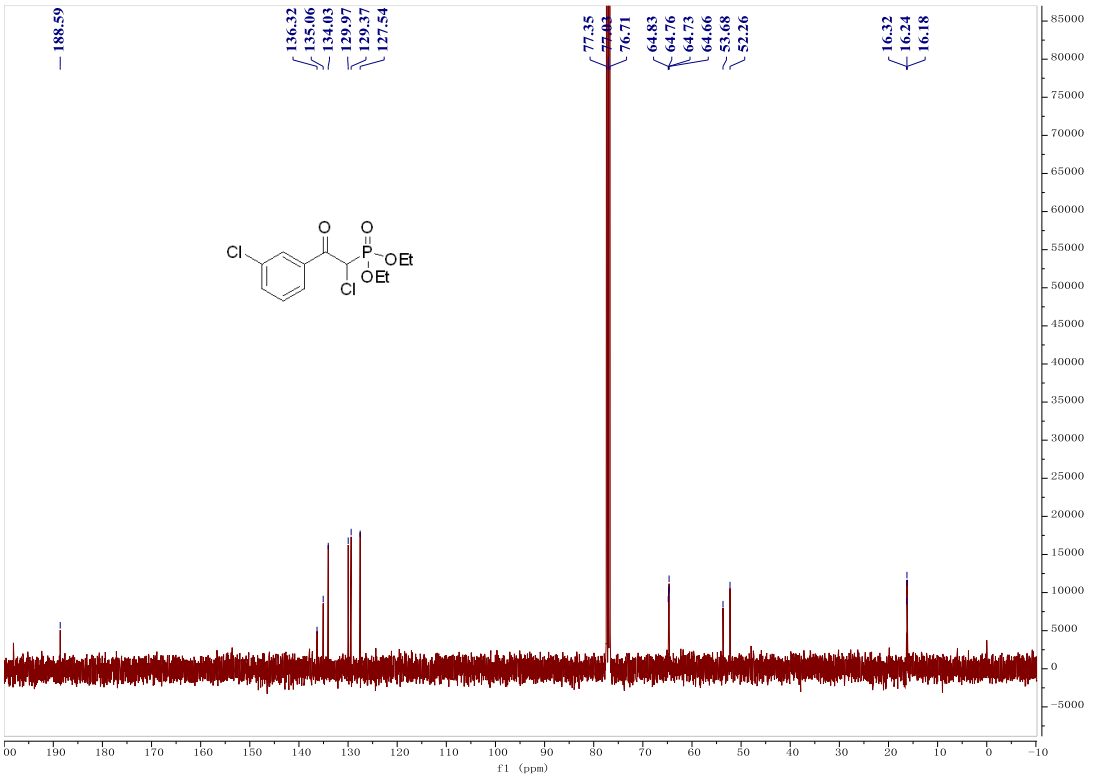
^{31}P NMR of **1n**, (162 MHz, Chloroform-*d*)



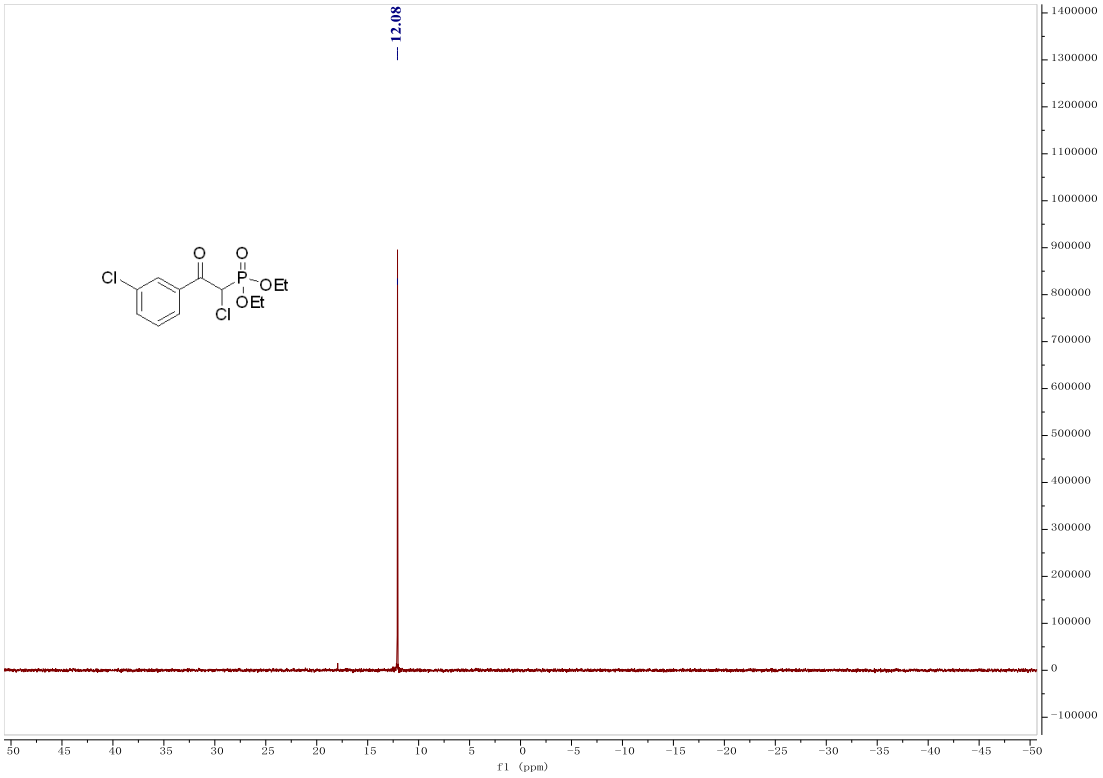
^{1}H NMR of **1o**, (400 MHz, Chloroform-*d*)



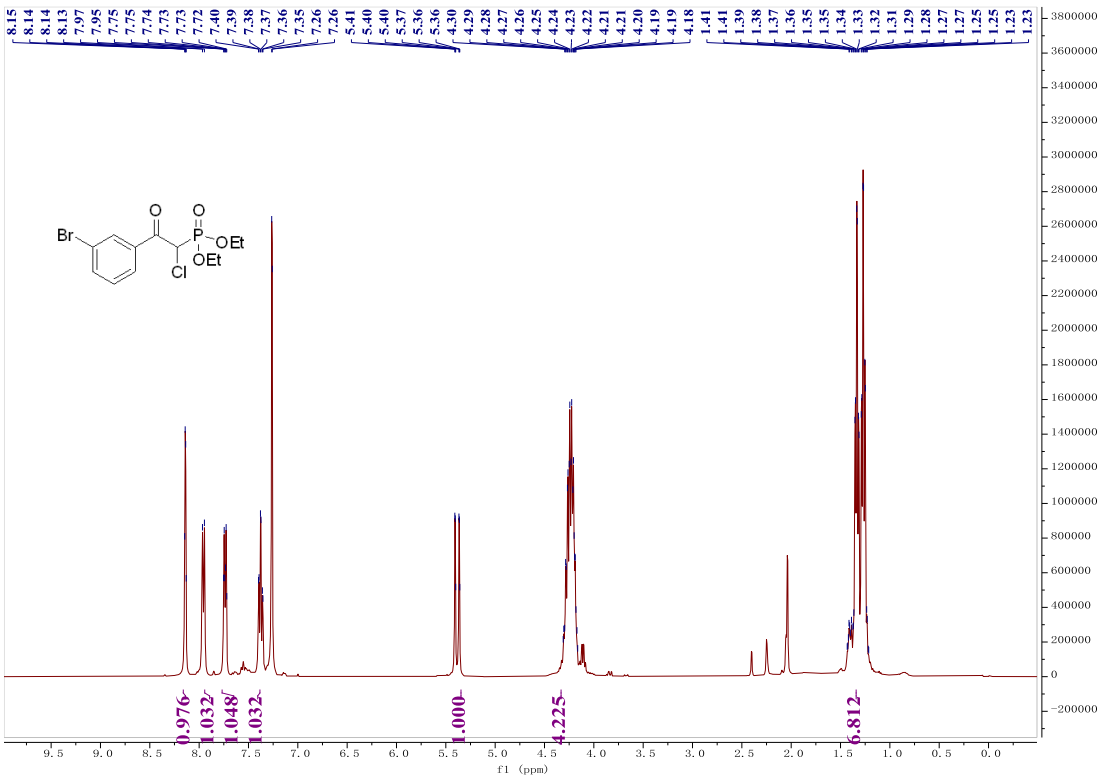
^{13}C NMR of **1o**, (101 MHz, Chloroform-*d*)



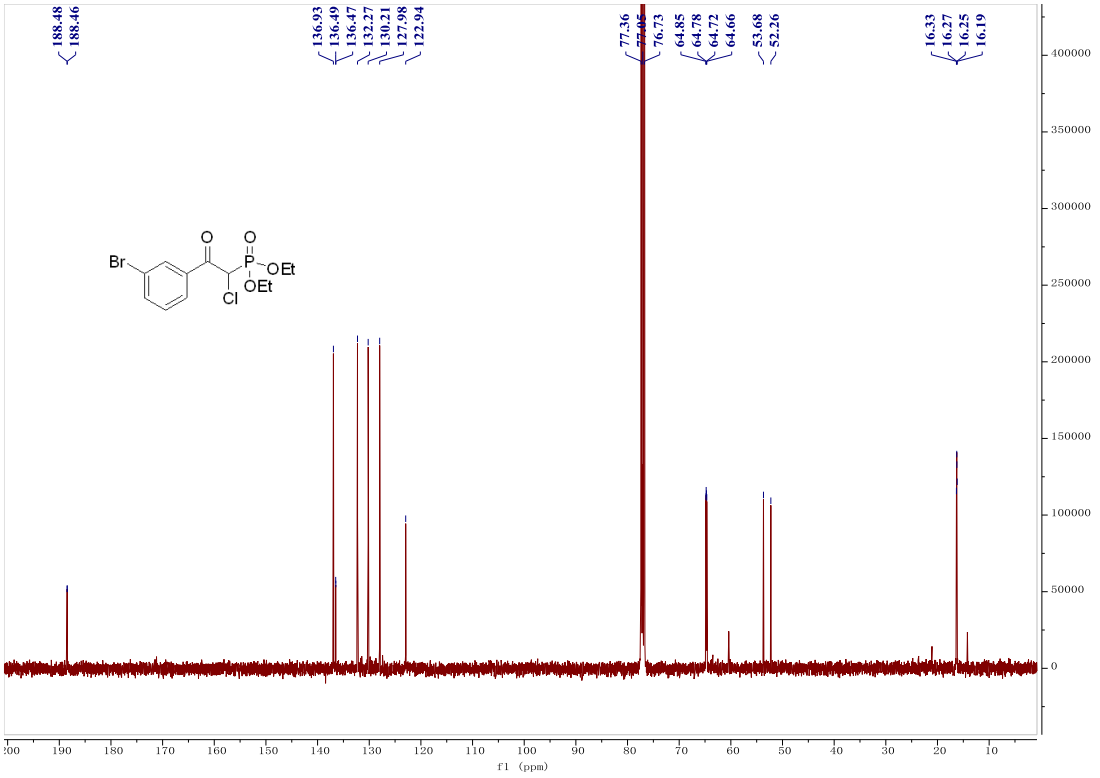
^{31}P NMR of **1o**, (162 MHz, Chloroform-*d*)



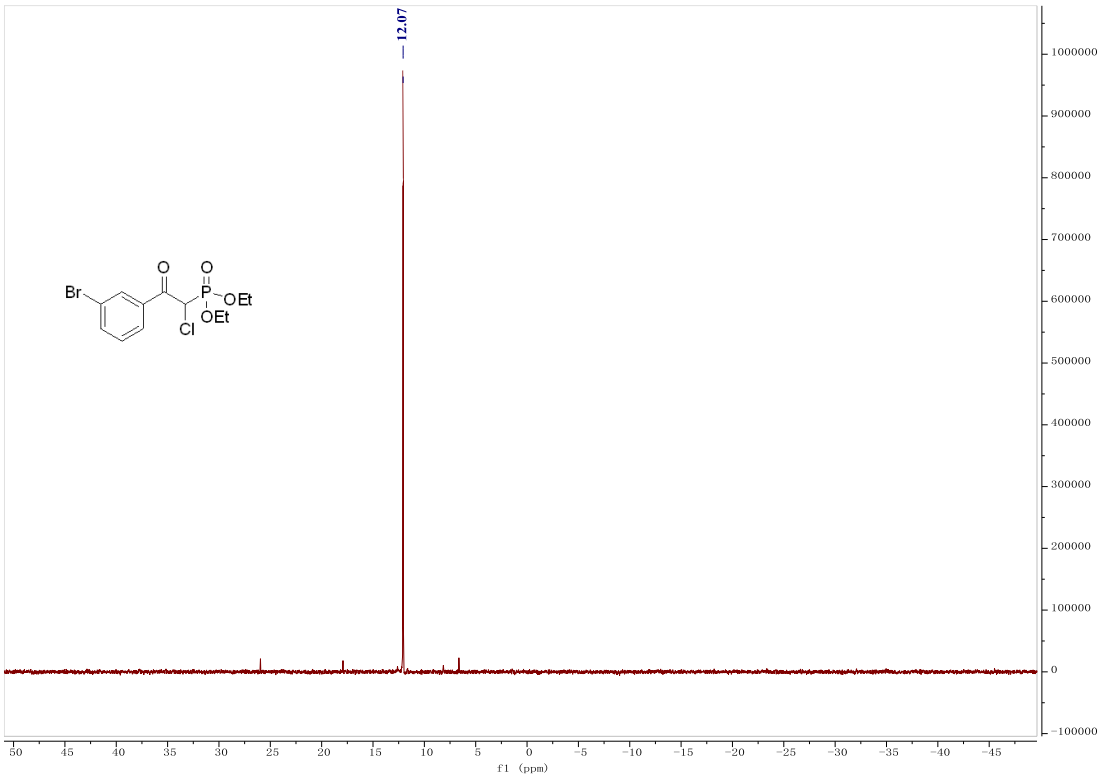
^{1}H NMR of **1p**, (400 MHz, Chloroform-*d*)



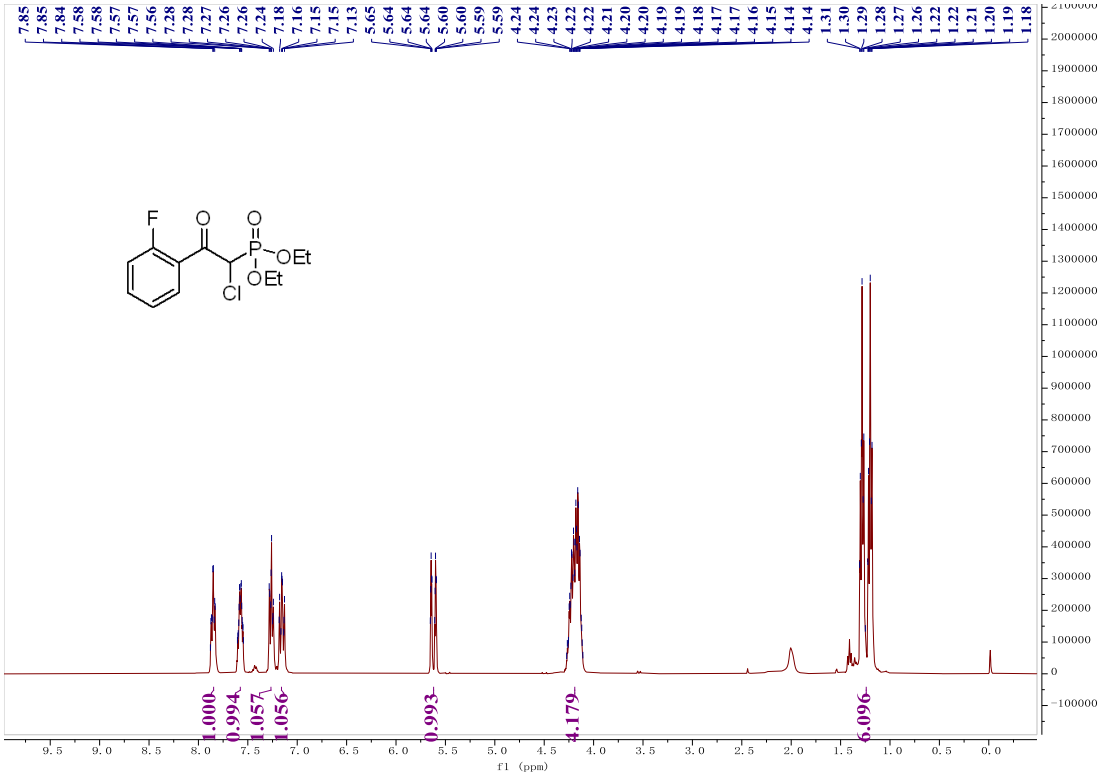
^{13}C NMR of **1p**, (101 MHz, Chloroform-*d*)



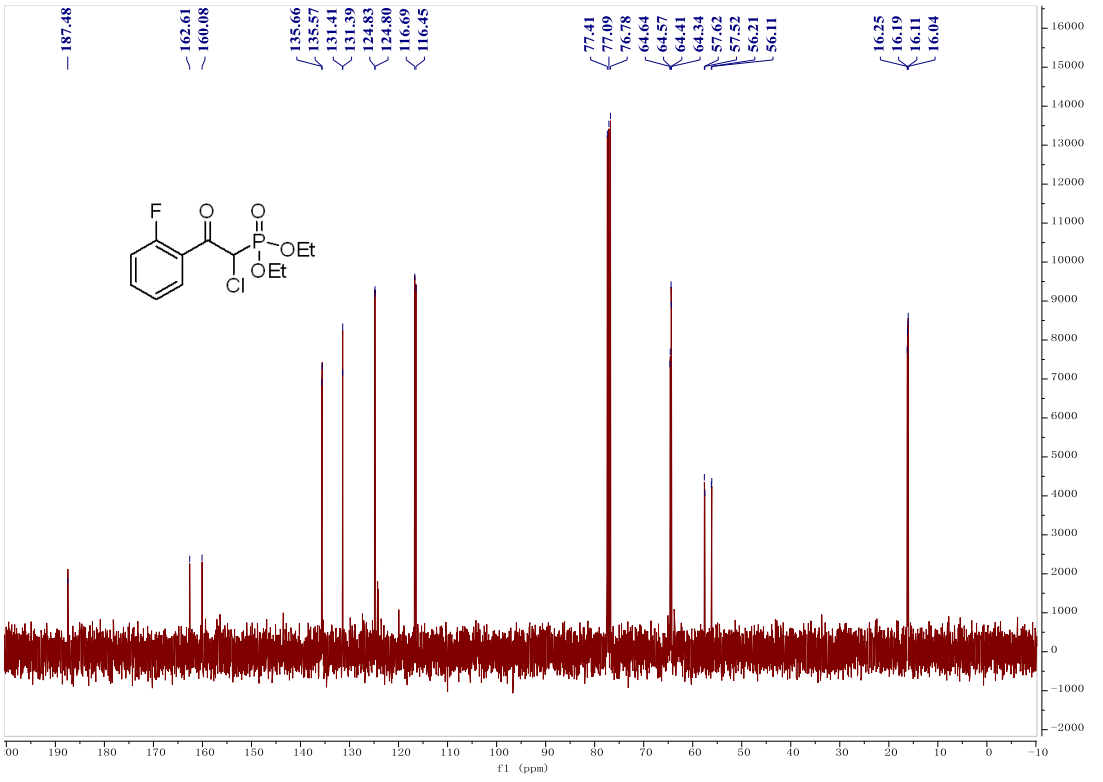
^{31}P NMR of **1p**, (162 MHz, Chloroform-*d*)



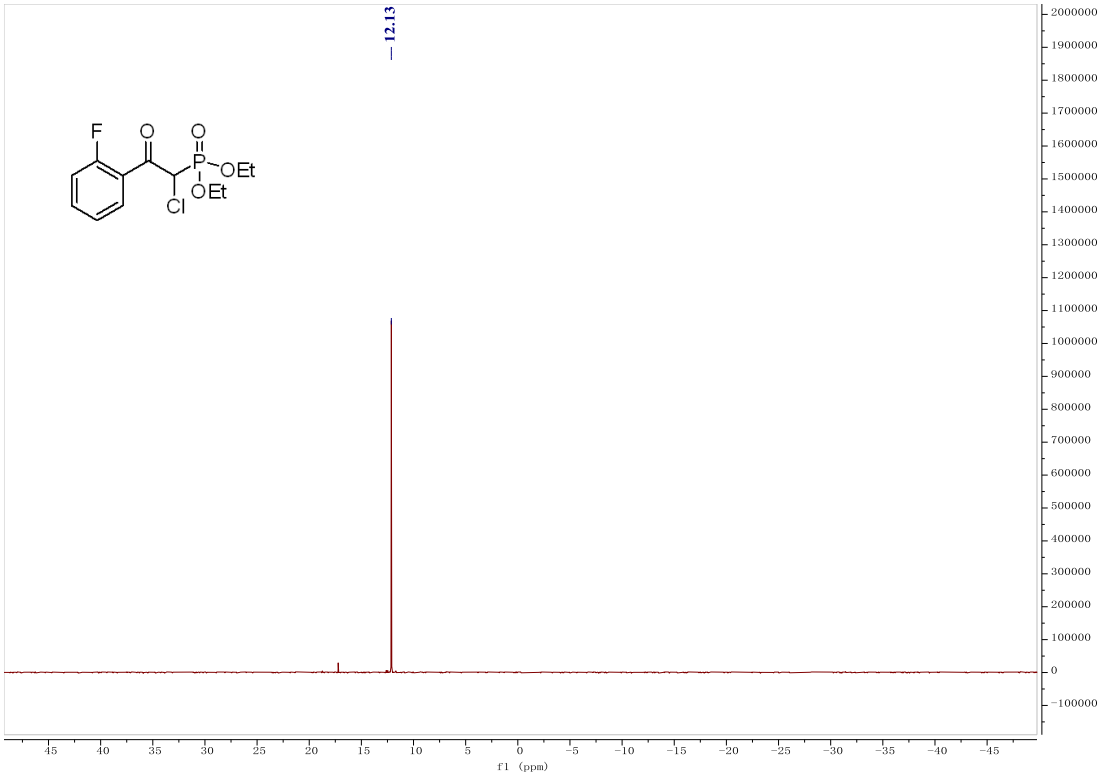
^{1}H NMR of **1q**, (400 MHz, Chloroform-*d*)



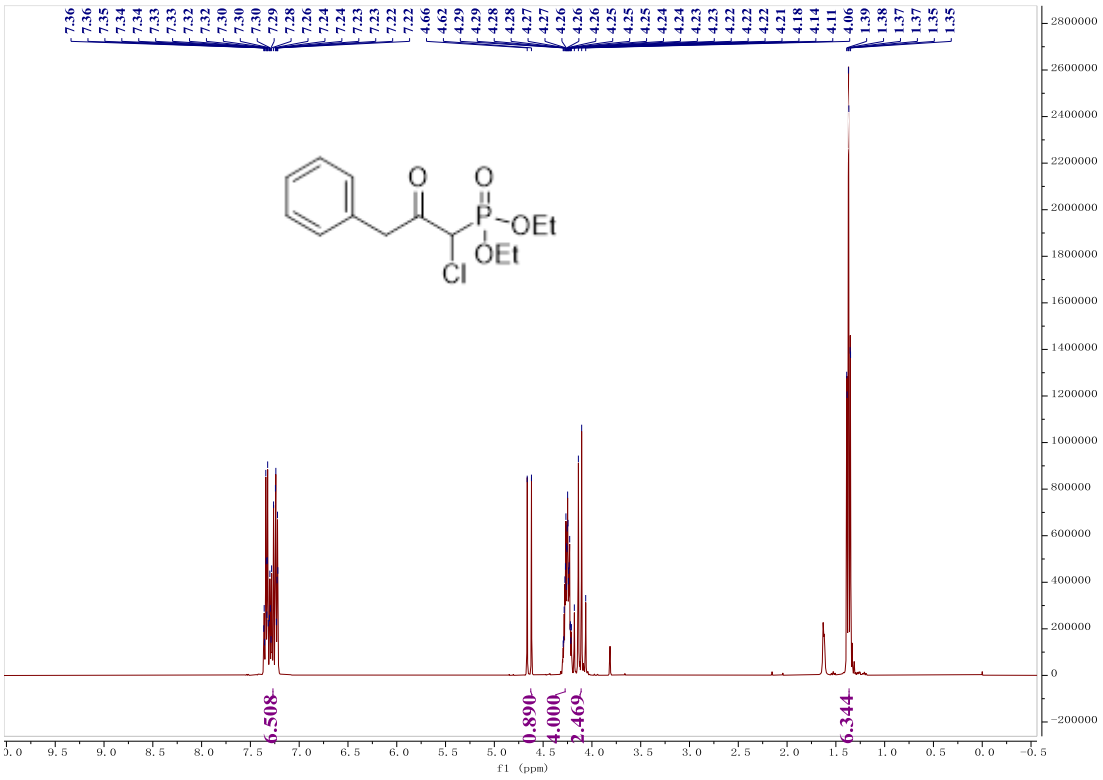
^{13}C NMR of **1q**, (101 MHz, Chloroform-*d*)



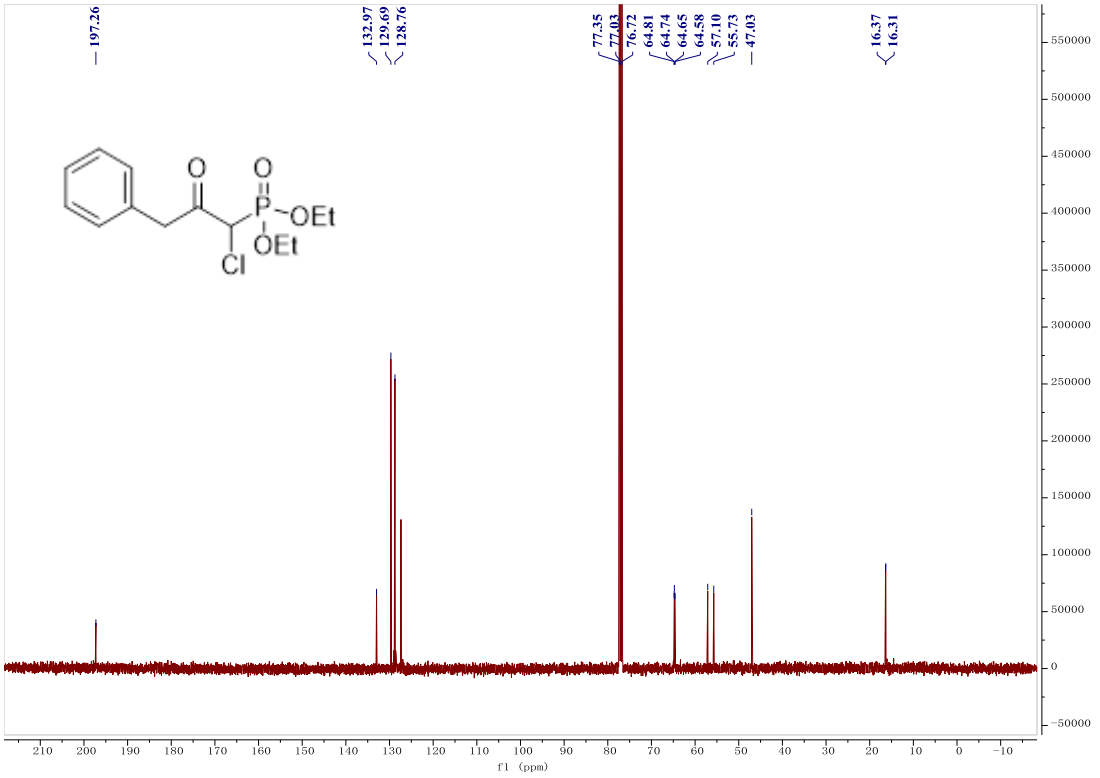
^{31}P NMR of **1q**, (162 MHz, Chloroform-*d*)



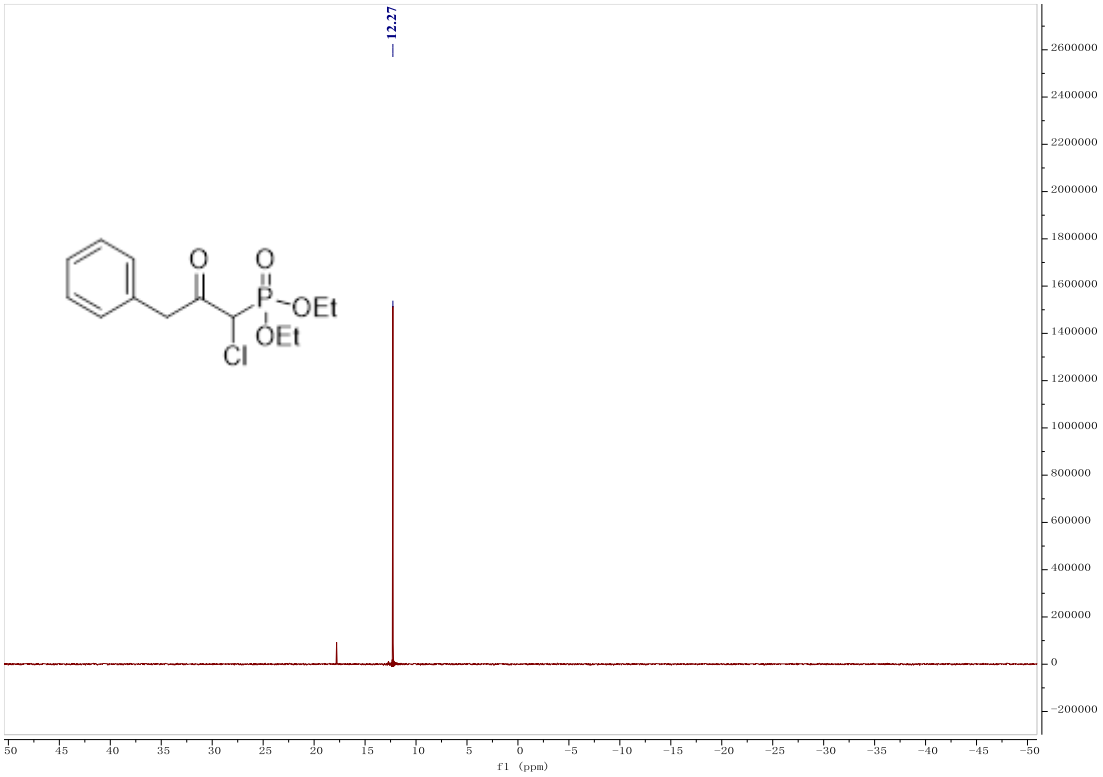
^{1}H NMR of **1r**, (400 MHz, Chloroform-*d*)



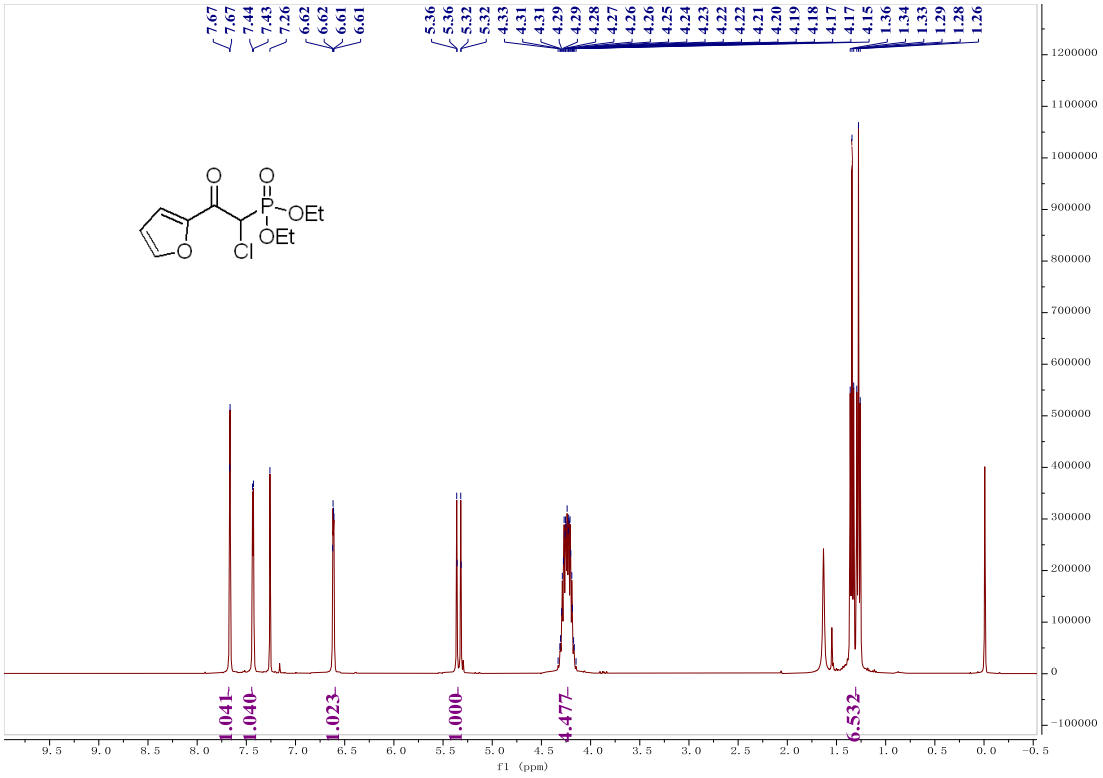
^{13}C NMR of **1r**, (101 MHz, Chloroform-*d*)



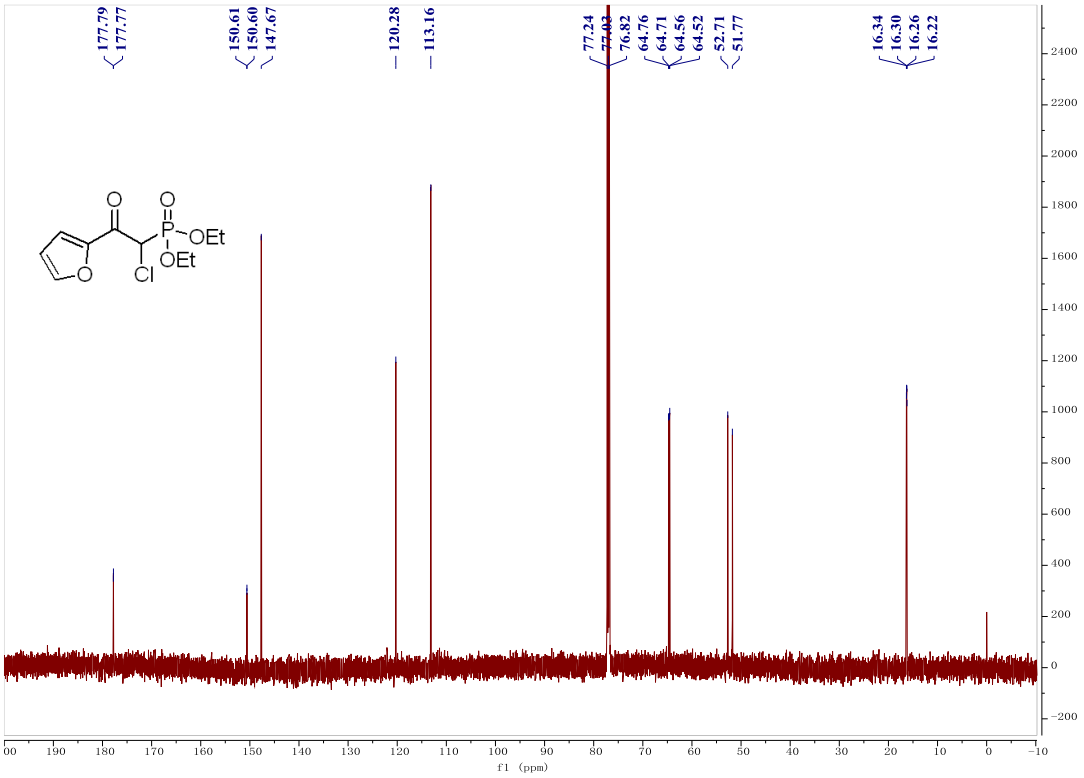
^{31}P NMR of **1r**, (162 MHz, Chloroform-*d*)



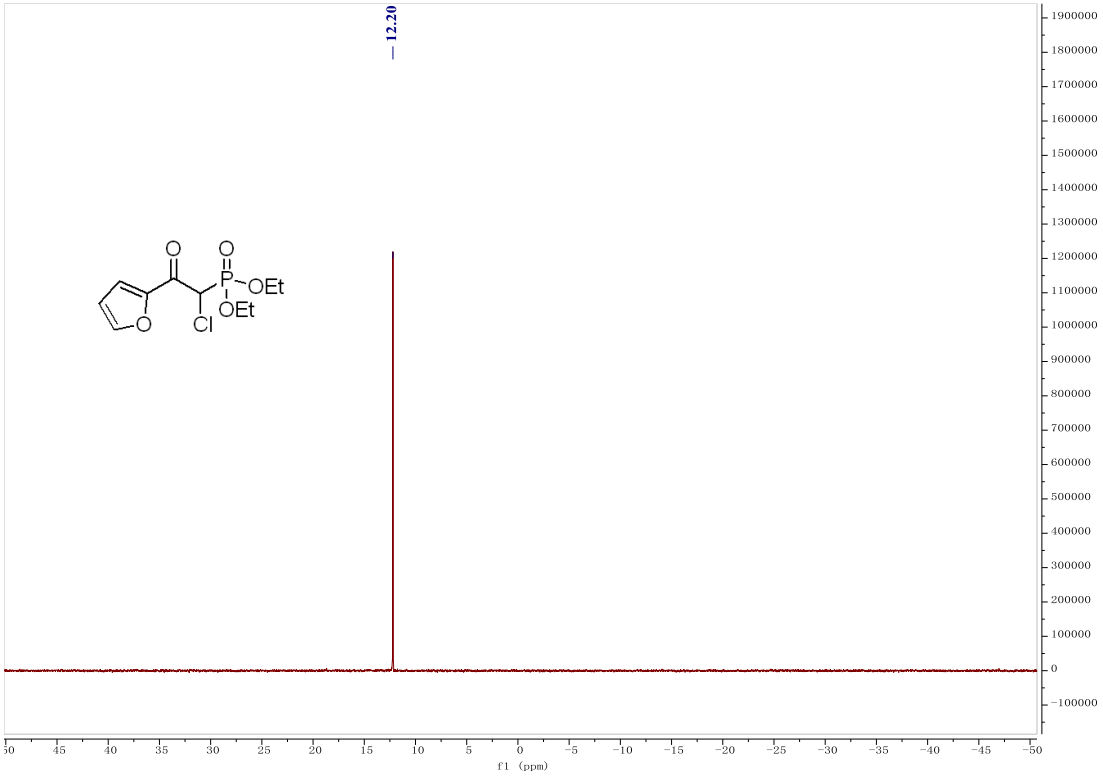
^{1}H NMR of **1s**, (400 MHz, Chloroform-*d*)



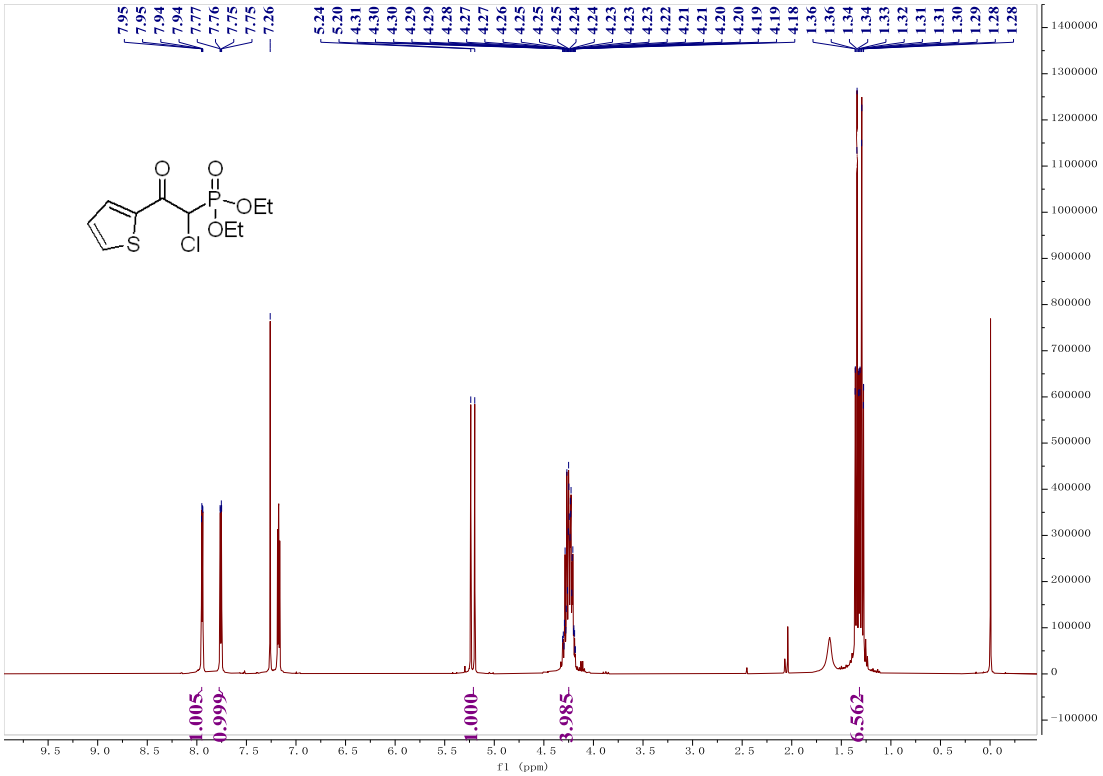
^{13}C NMR of **1s**, (101 MHz, Chloroform-*d*)



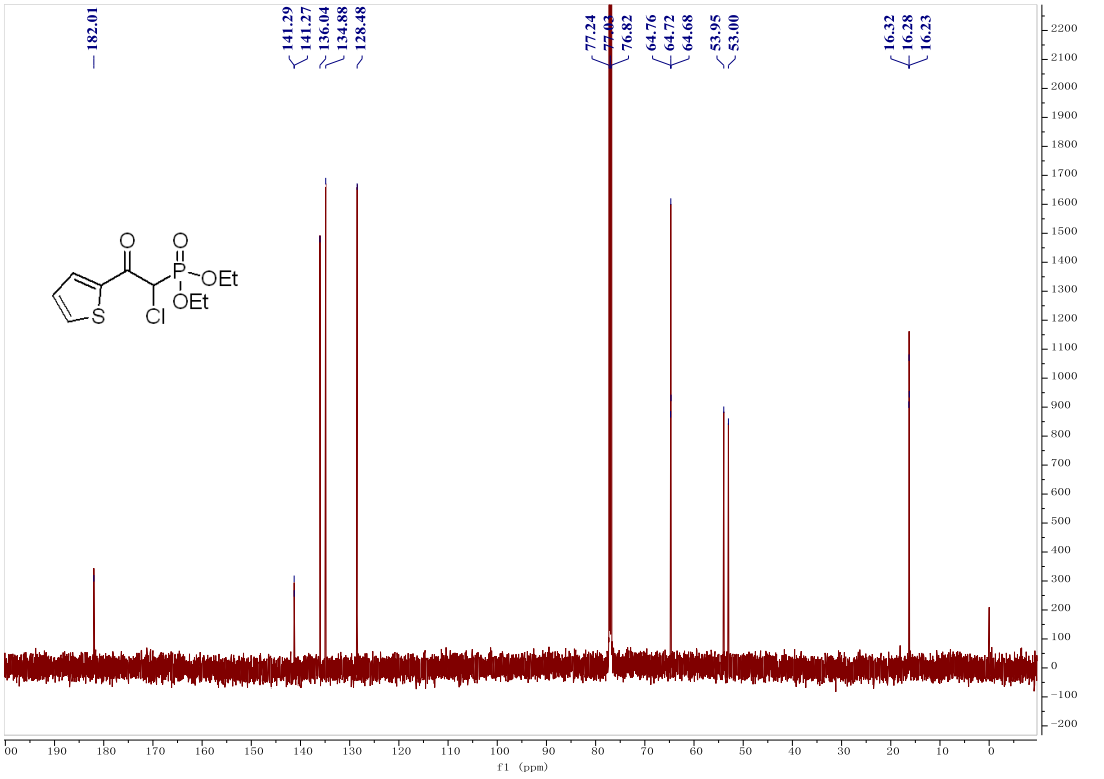
^{31}P NMR of **1s**, (162 MHz, Chloroform-*d*)



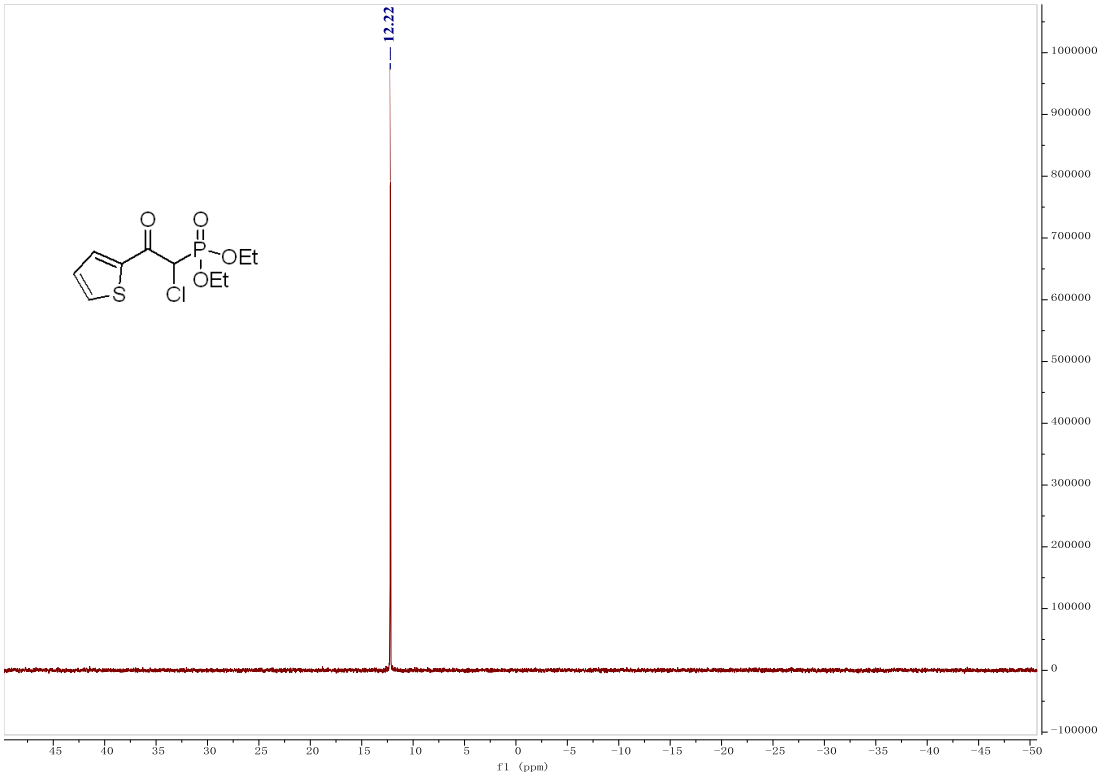
^{1}H NMR of **1t**, (400 MHz, Chloroform-*d*)



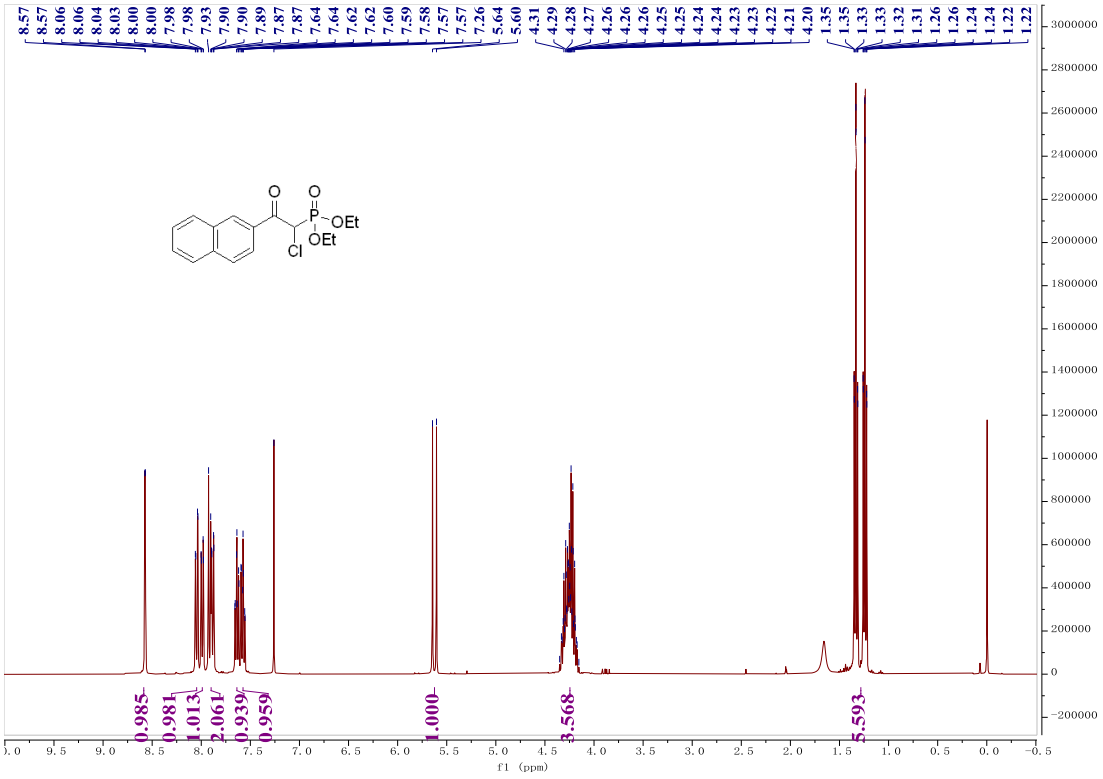
^{13}C NMR of **1t**, (101 MHz, Chloroform-*d*)



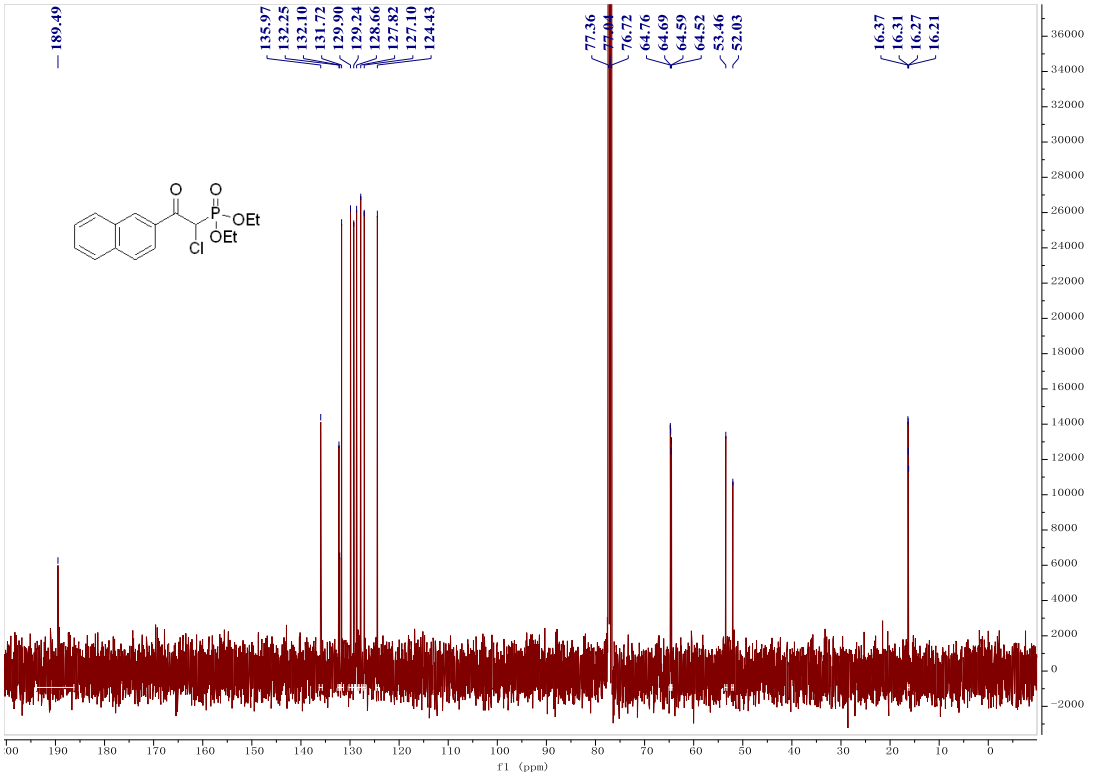
^{31}P NMR of **1t**, (162 MHz, Chloroform-*d*)



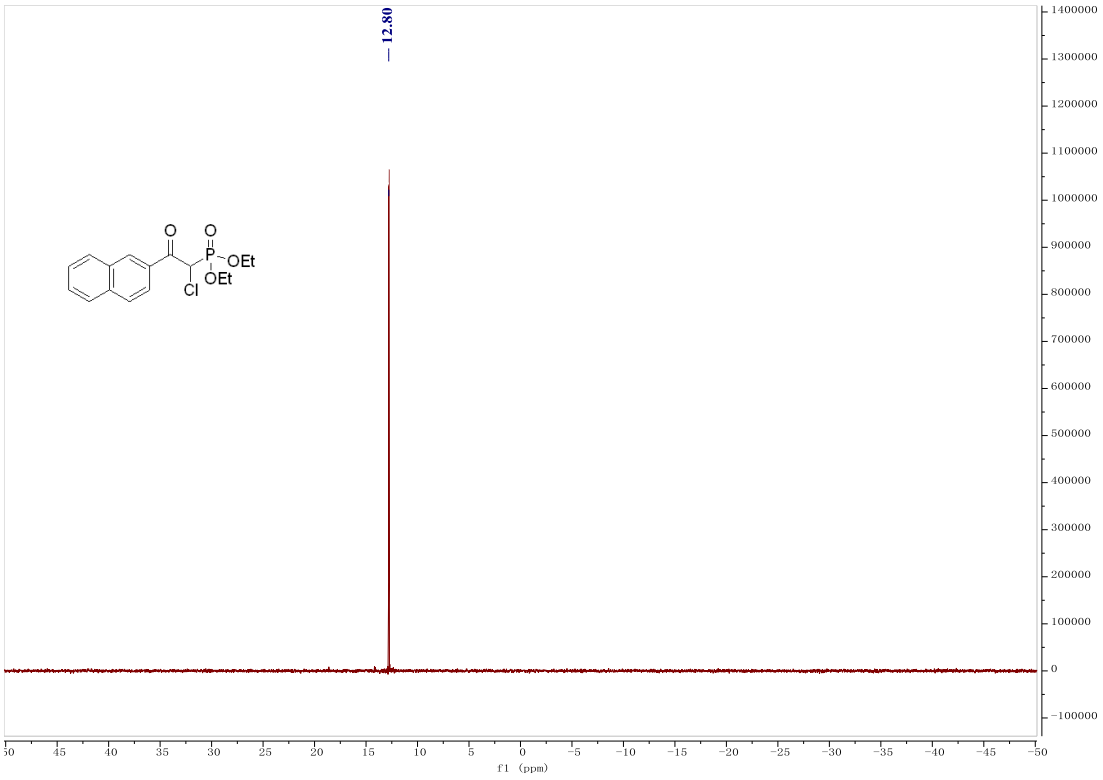
^{1}H NMR of **1u**, (400 MHz, Chloroform-*d*)



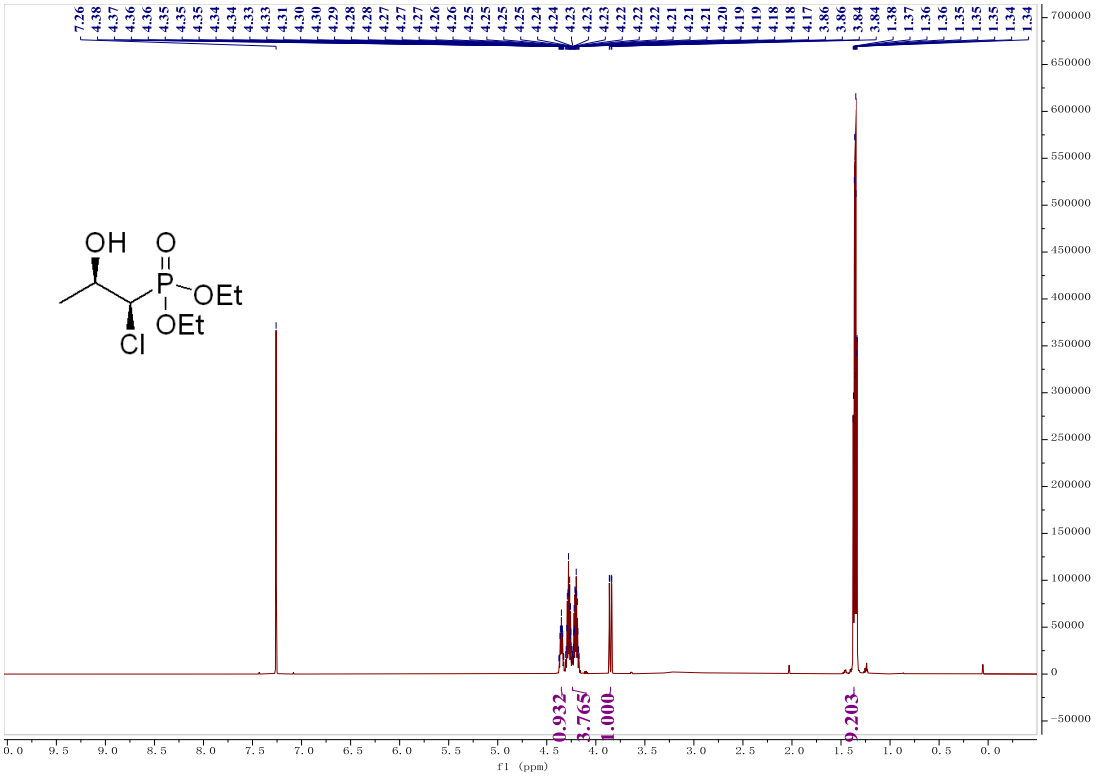
^{13}C NMR of **1u**, (101 MHz, Chloroform-*d*)



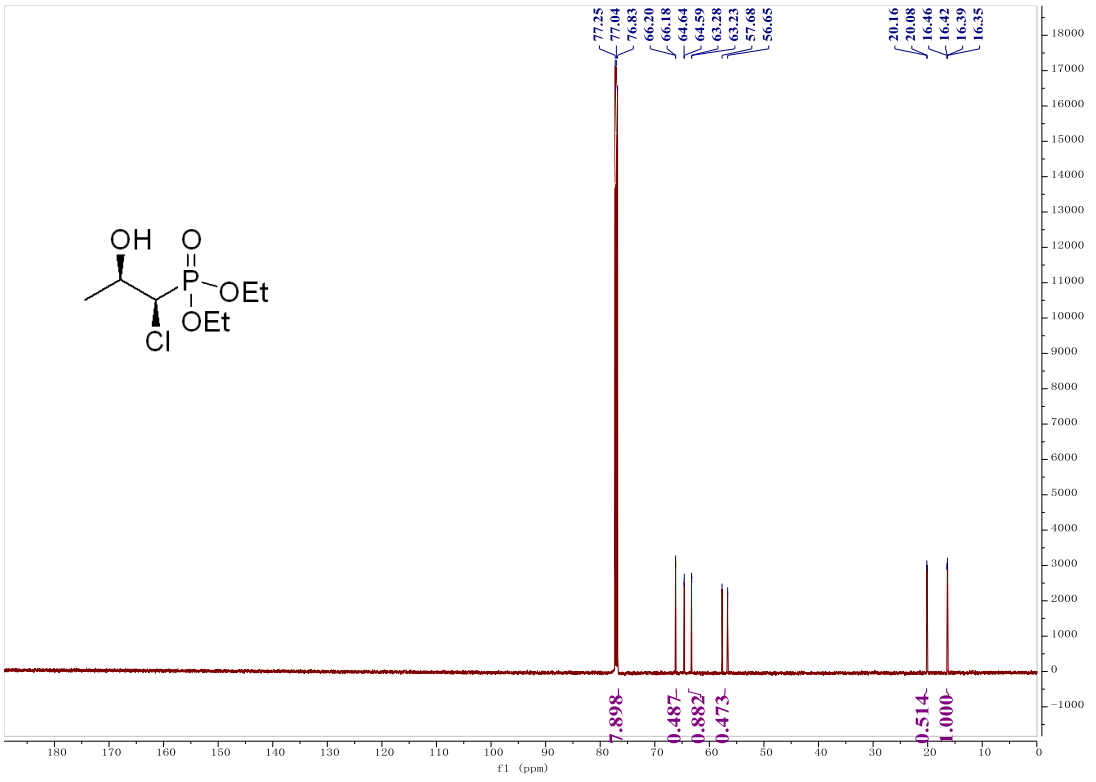
^{31}P NMR of **1u**, (162 MHz, Chloroform-*d*)



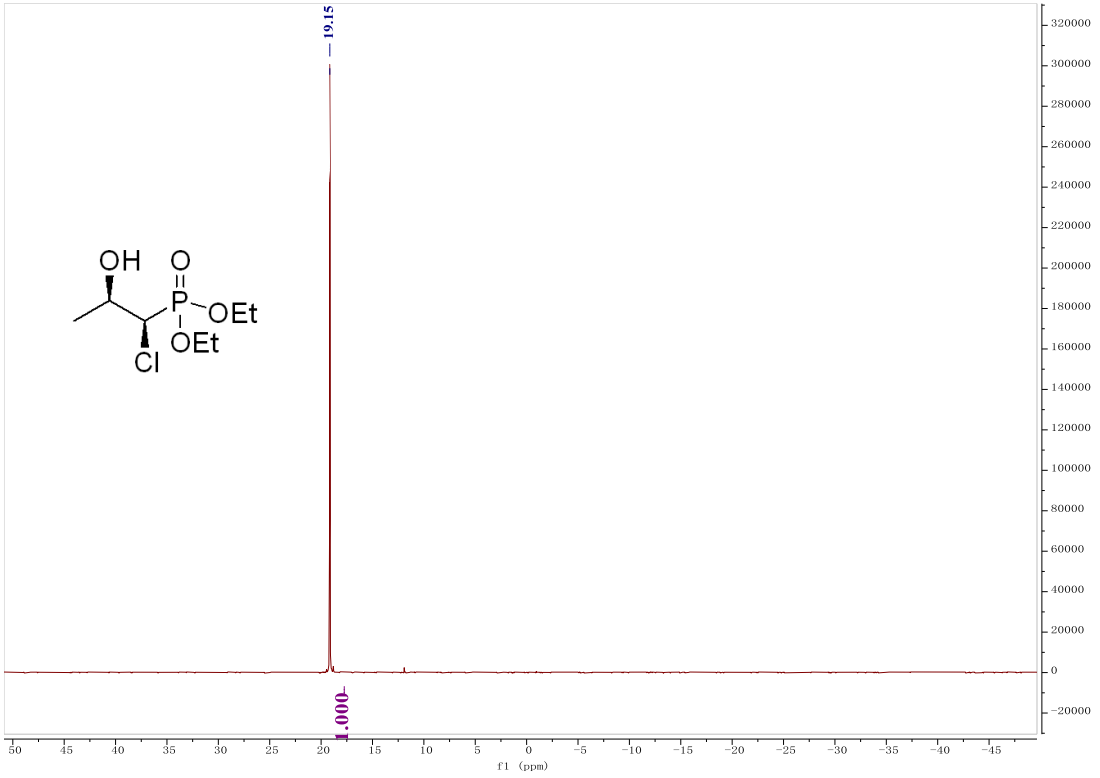
^{1}H NMR of **2a**, (400 MHz, Chloroform-*d*)



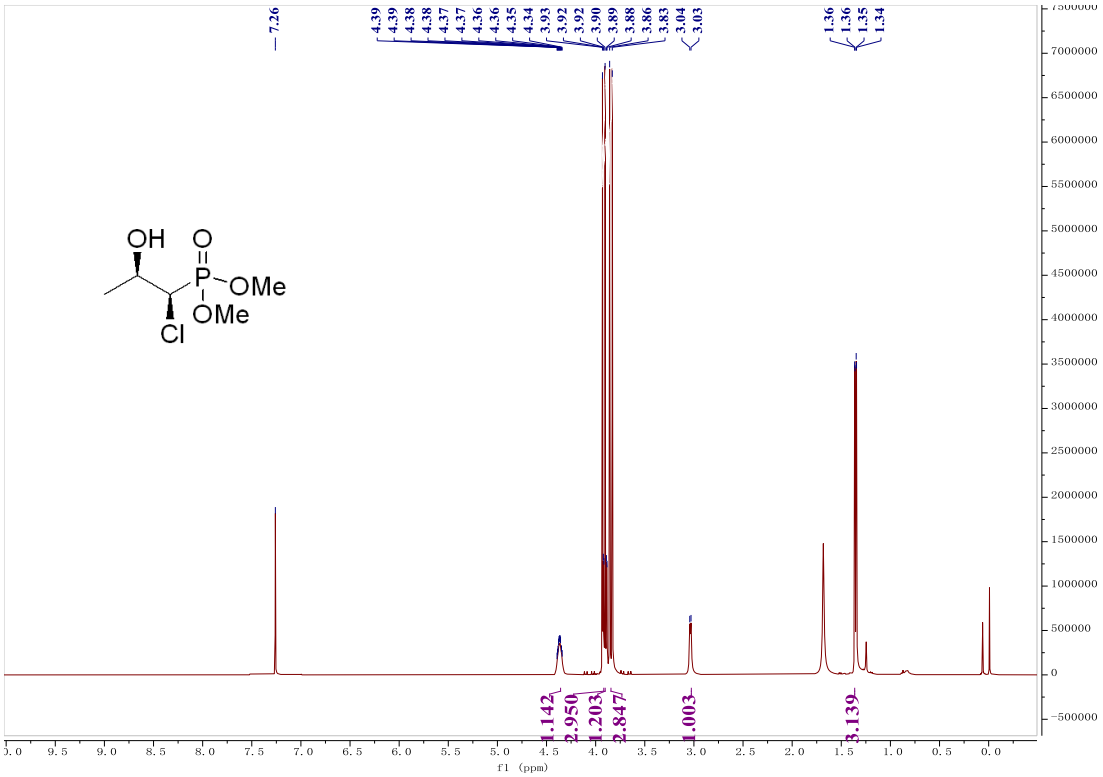
^{13}C NMR of **2a**, (101 MHz, Chloroform-*d*)



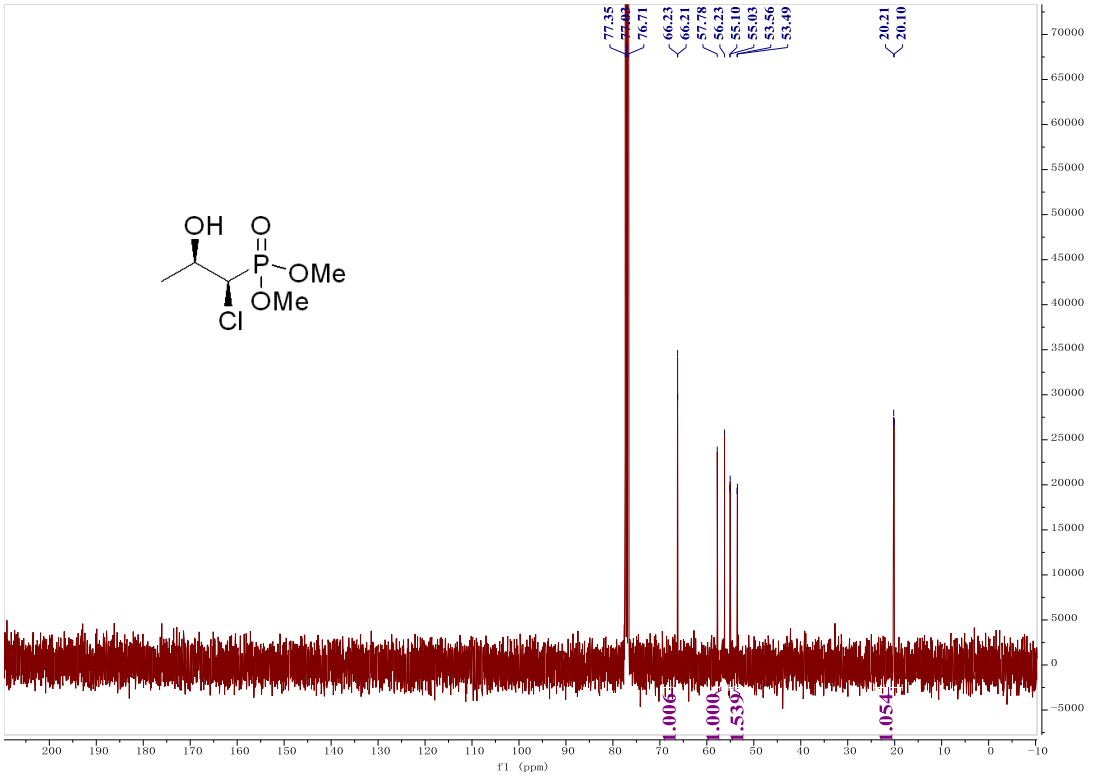
^{31}P NMR of **2a**, (162 MHz, Chloroform-*d*)



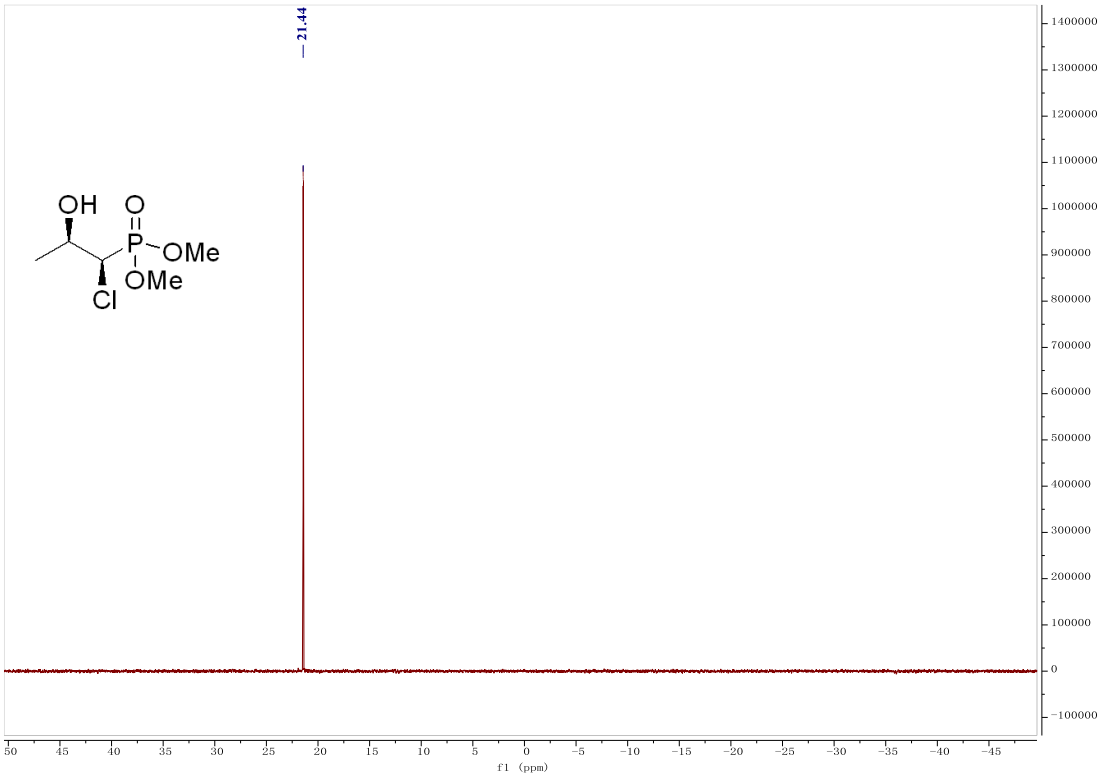
^{1}H NMR of **2b**, (400 MHz, Chloroform-*d*)



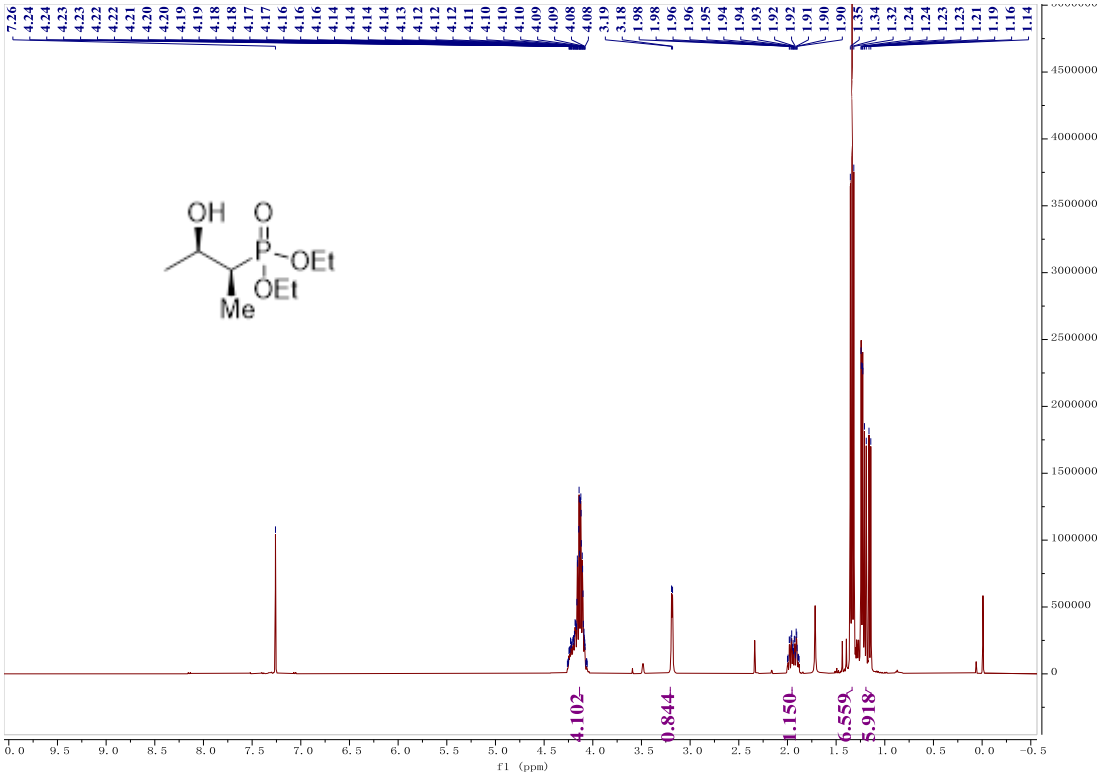
^{13}C NMR of **2b**, (101 MHz, Chloroform-*d*)



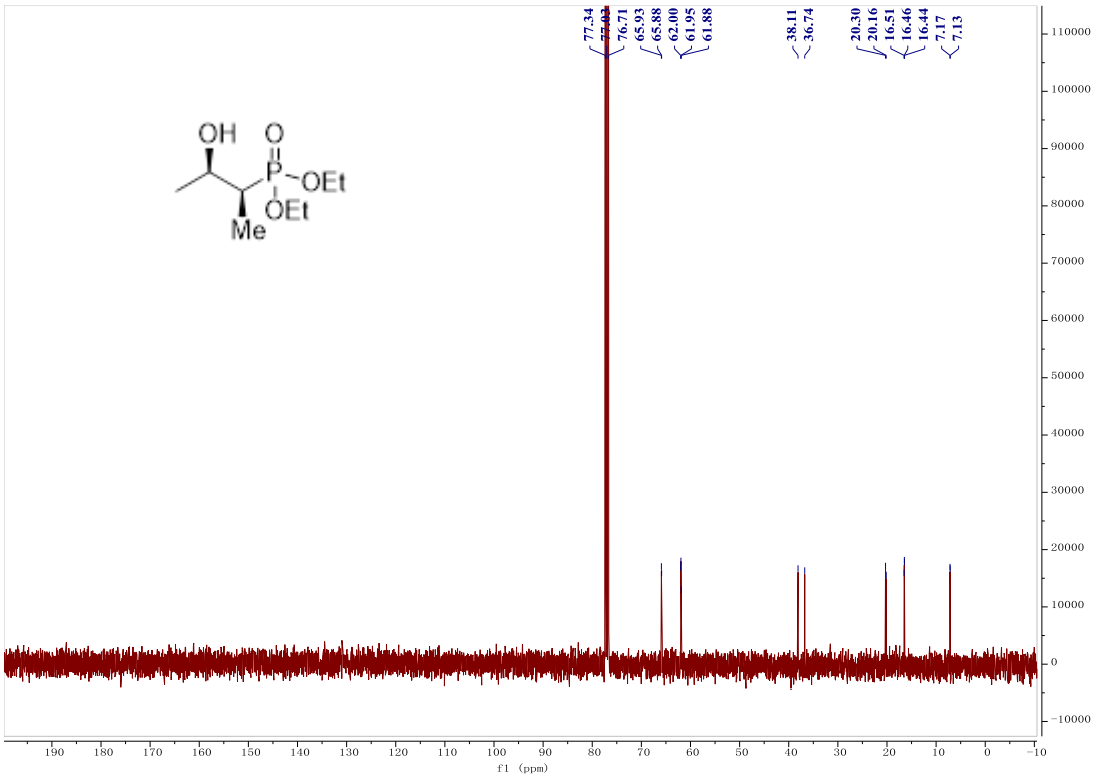
^{31}P NMR of **2b**, (162 MHz, Chloroform-*d*)



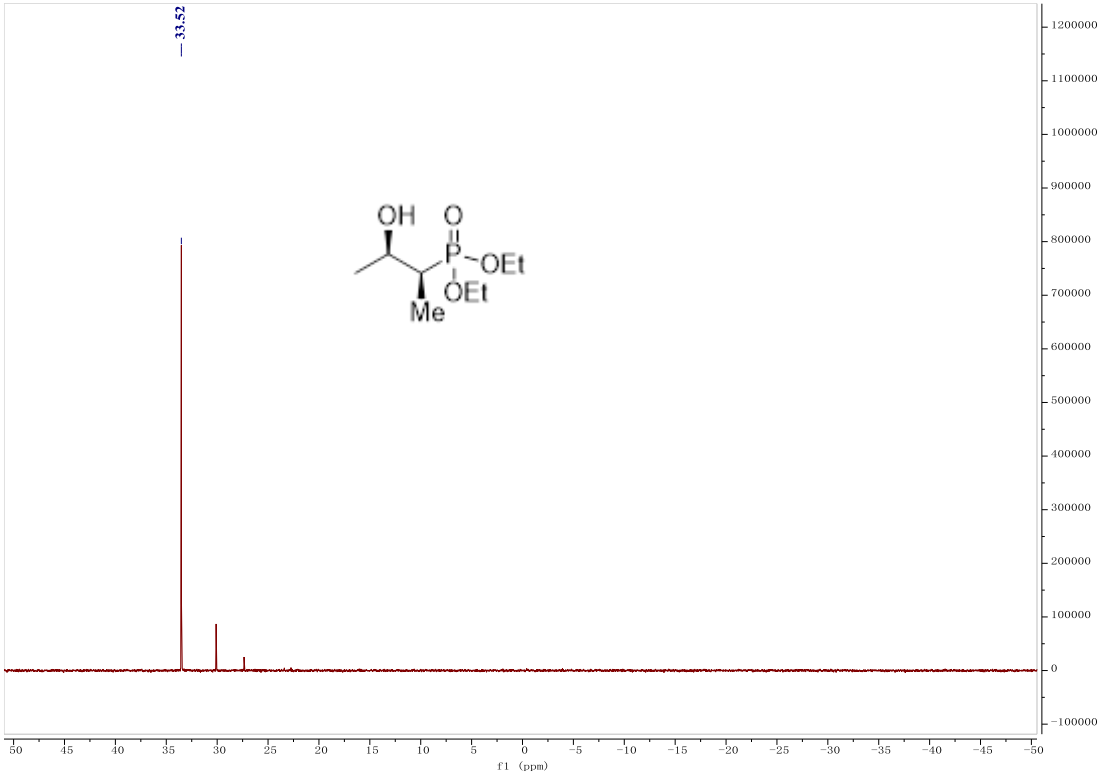
^{1}H NMR of **2c**, (400 MHz, Chloroform-*d*)



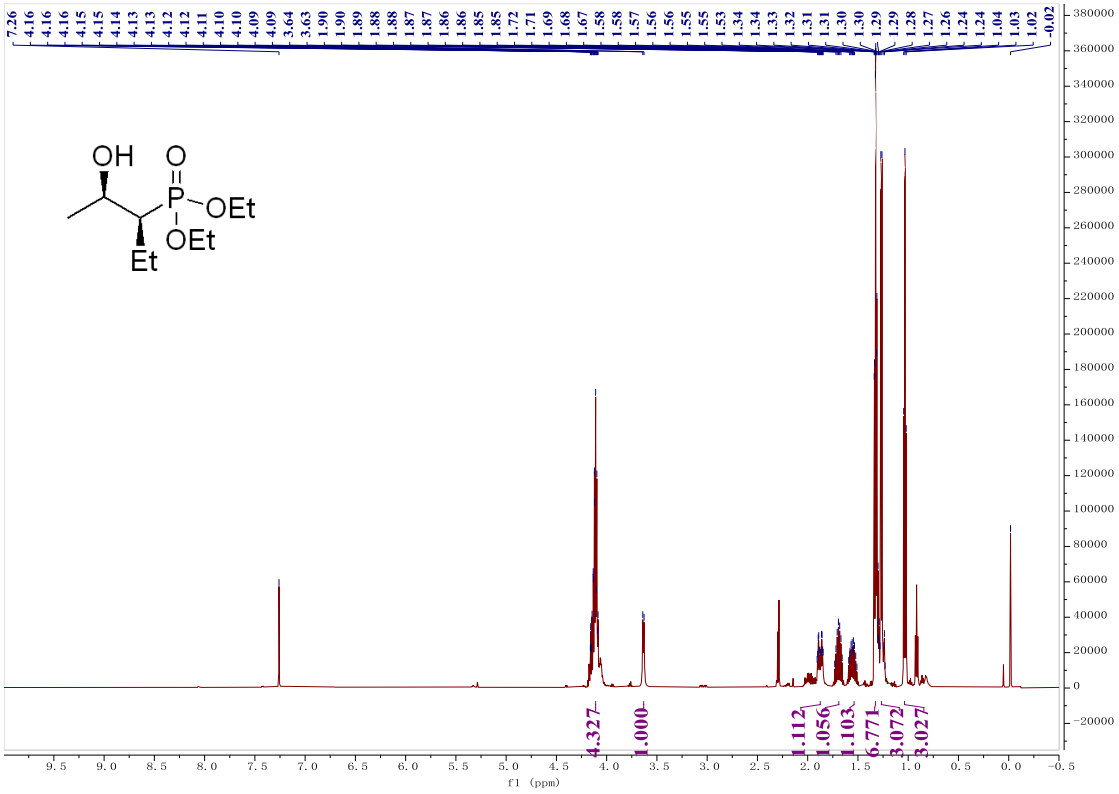
^{13}C NMR of **2c**, (101 MHz, Chloroform-*d*)



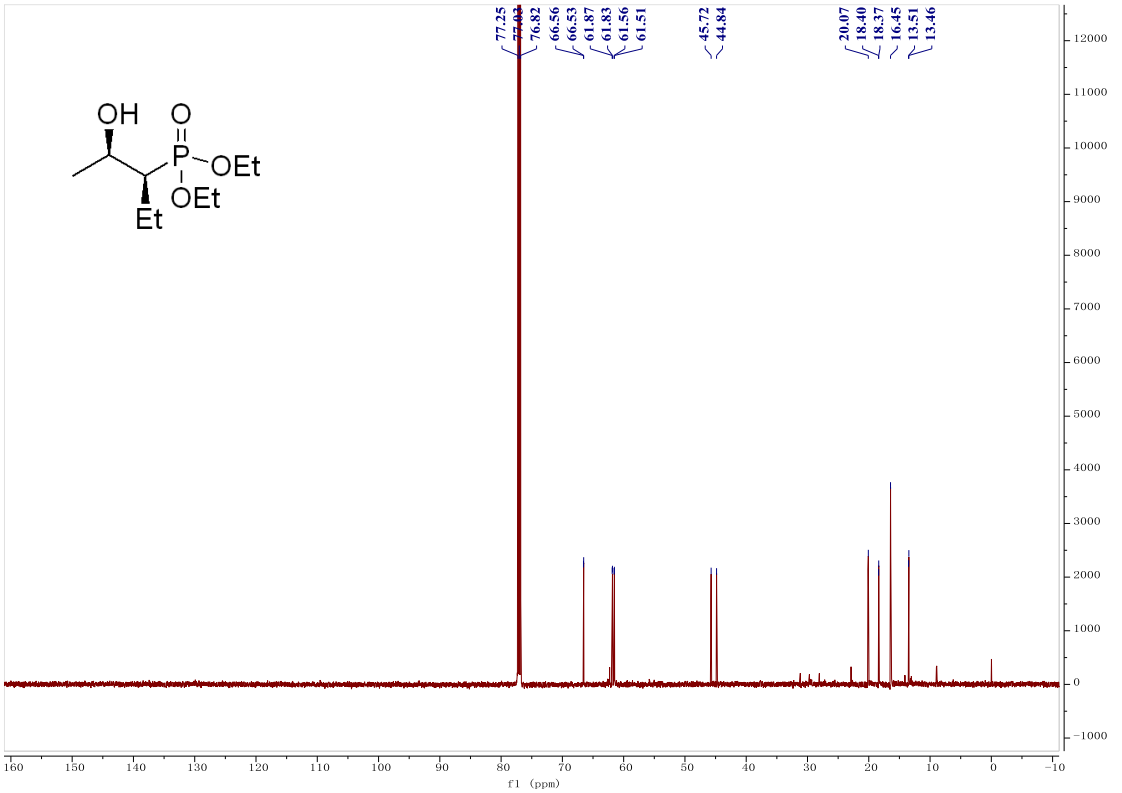
^{31}P NMR of **2c**, (162 MHz, Chloroform-*d*)



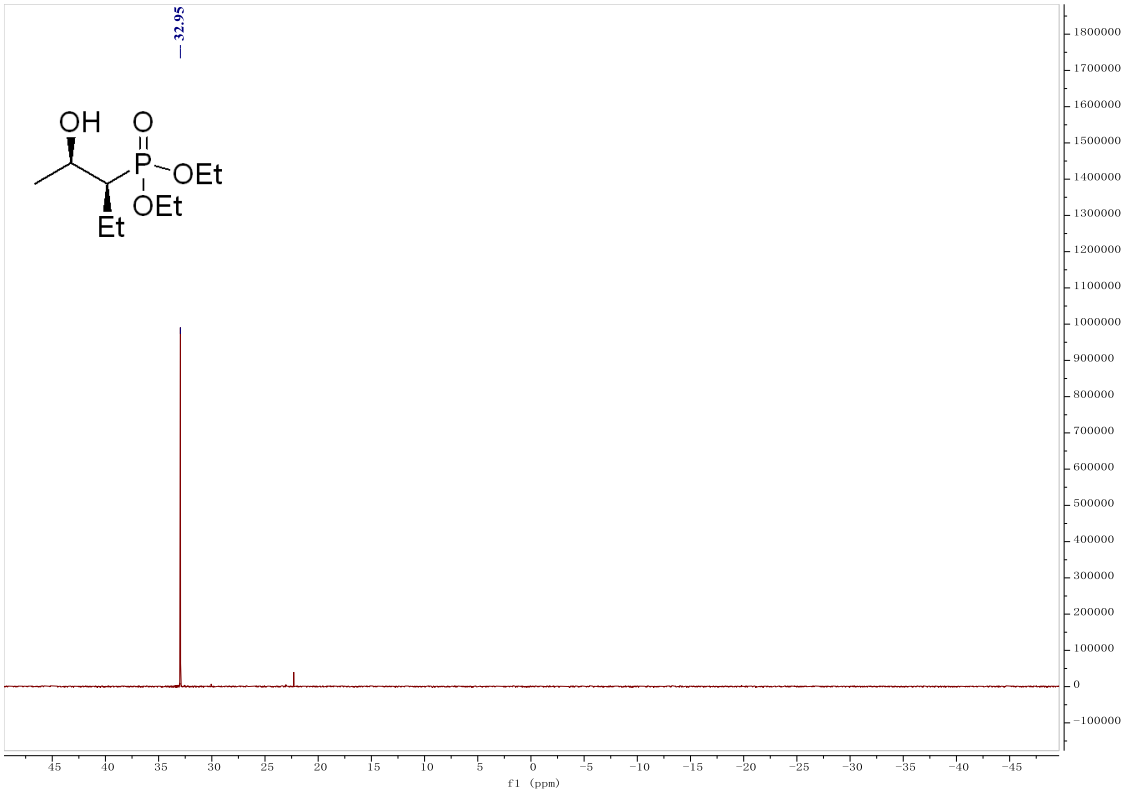
^{1}H NMR of 2**d**, (400 MHz, Chloroform-*d*)



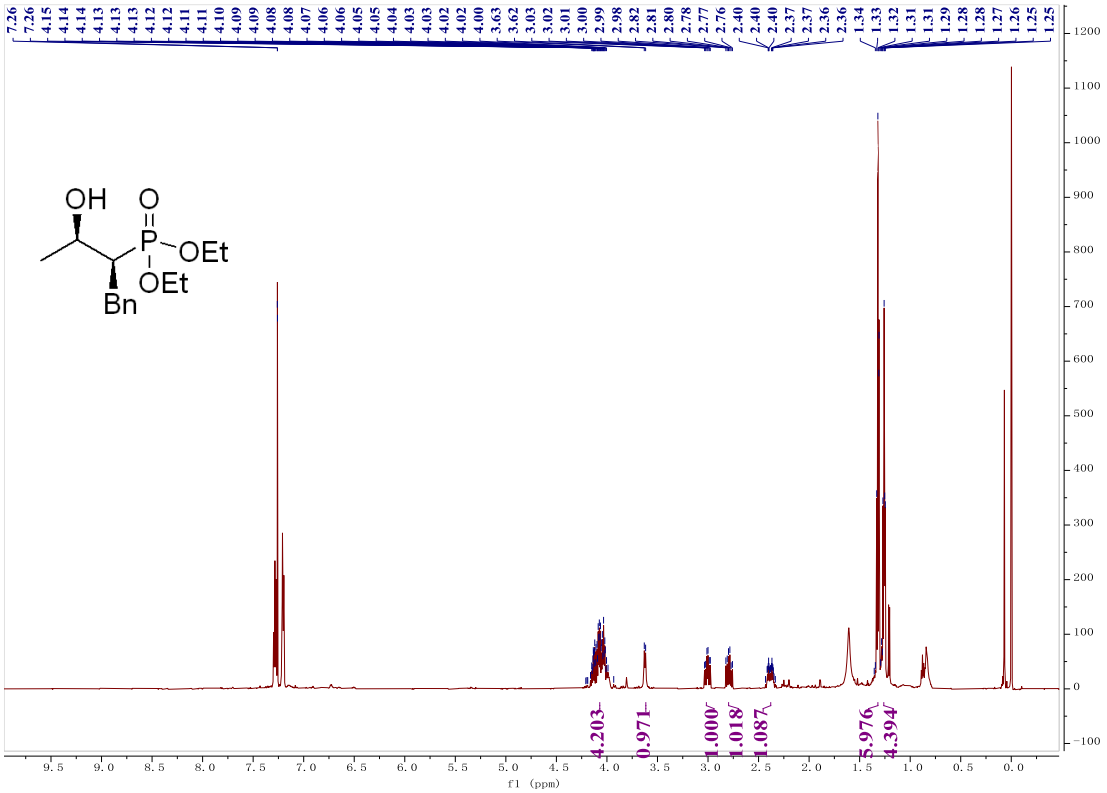
^{13}C NMR of **2d**, (101 MHz, Chloroform-*d*)



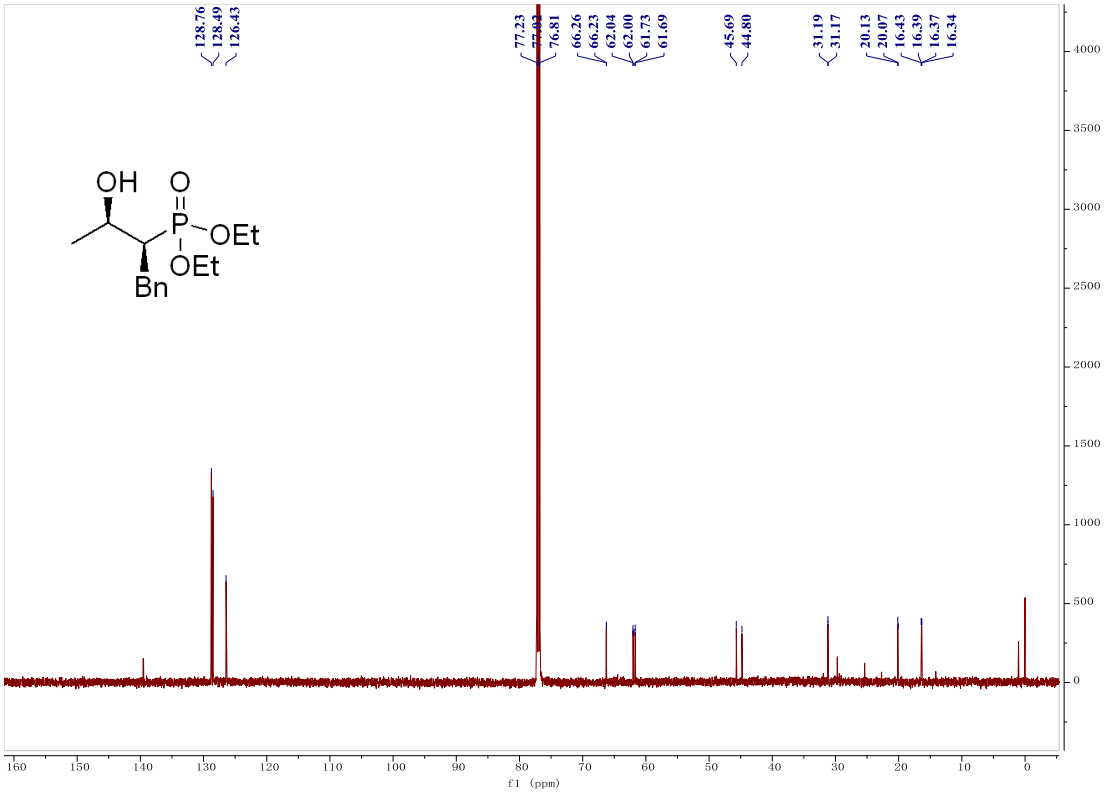
^{31}P NMR of **2d**, (162 MHz, Chloroform-*d*)



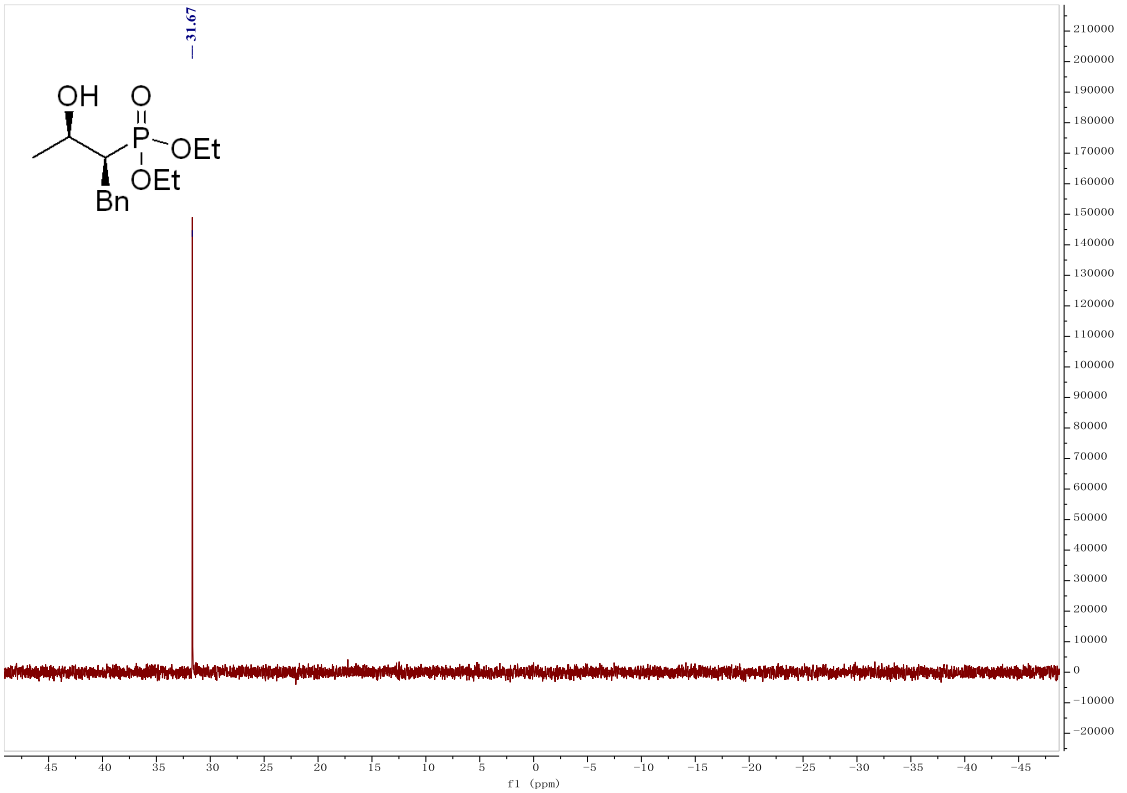
^{1}H NMR of **2e**, (400 MHz, Chloroform-*d*)



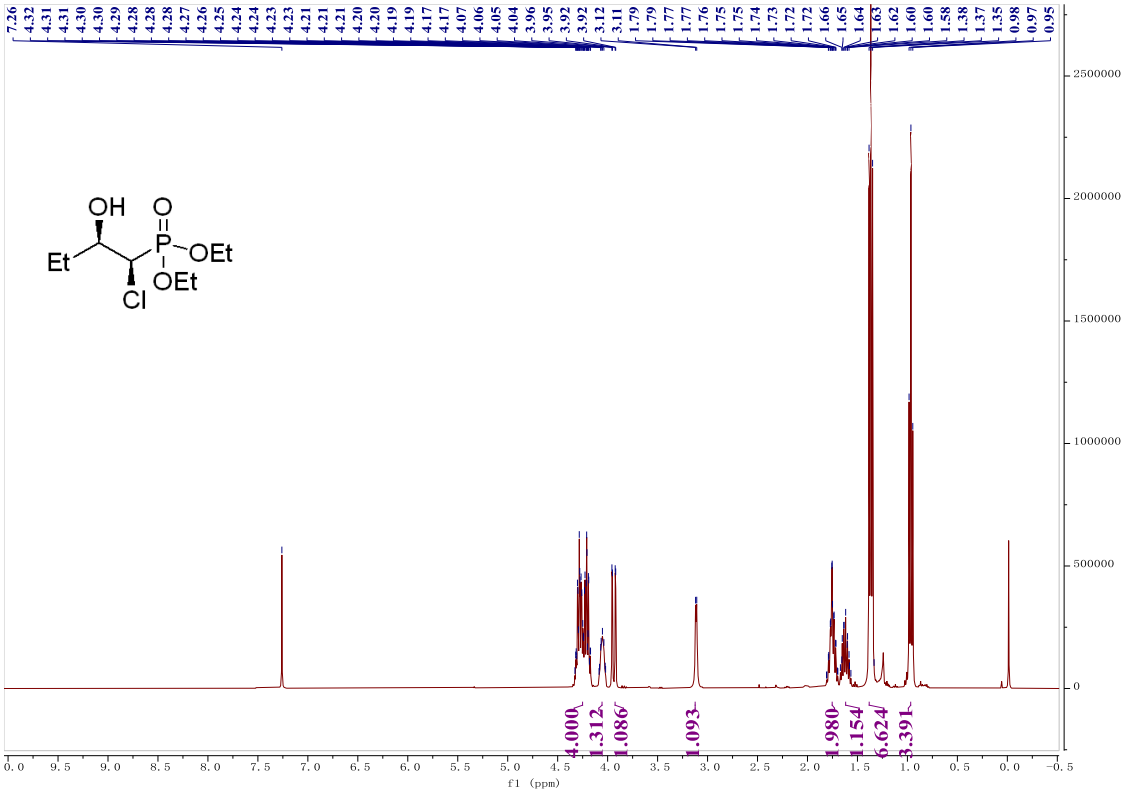
^{13}C NMR of **2e**, (101 MHz, Chloroform-*d*)



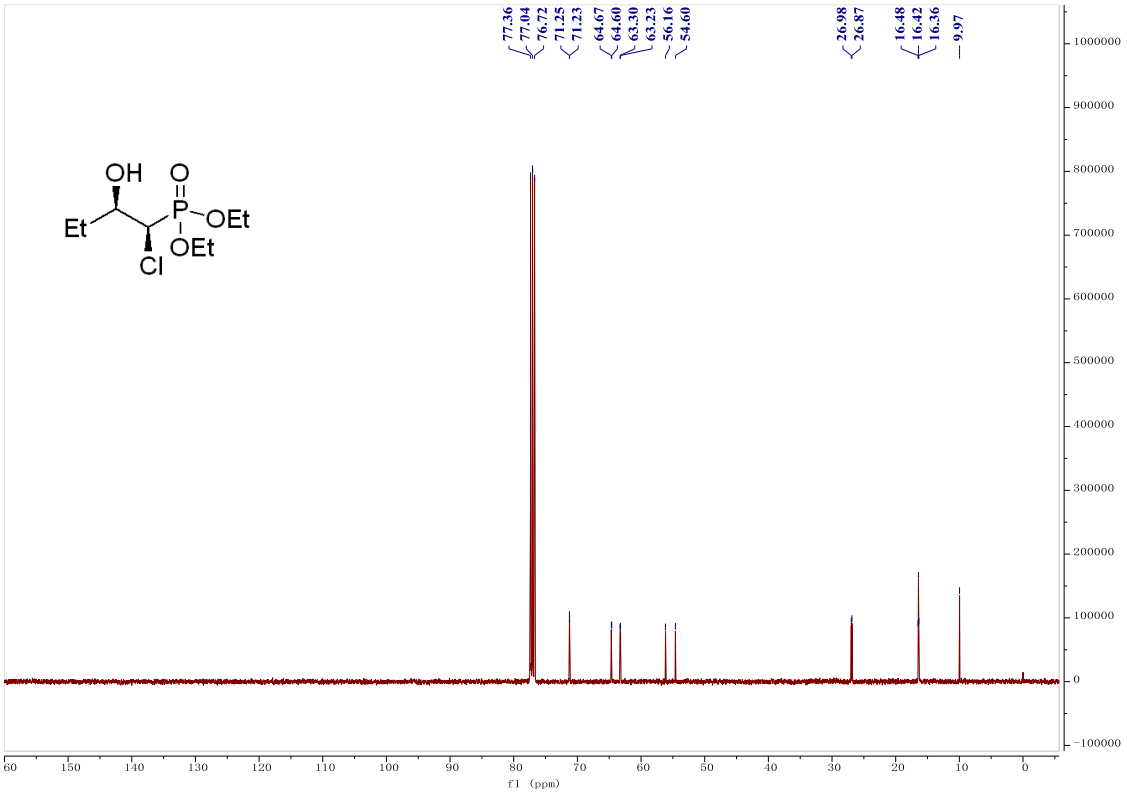
^{31}P NMR of **2e**, (162 MHz, Chloroform-*d*)



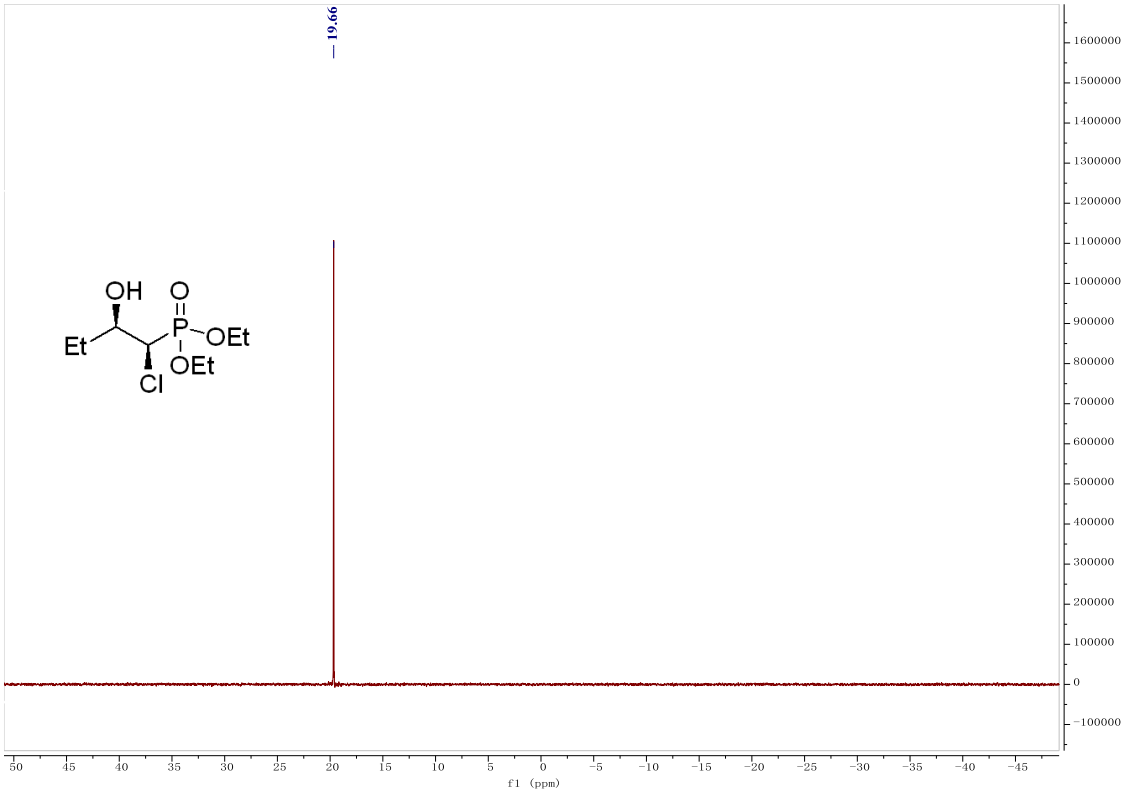
^{1}H NMR of **2f**, (400 MHz, Chloroform-*d*).



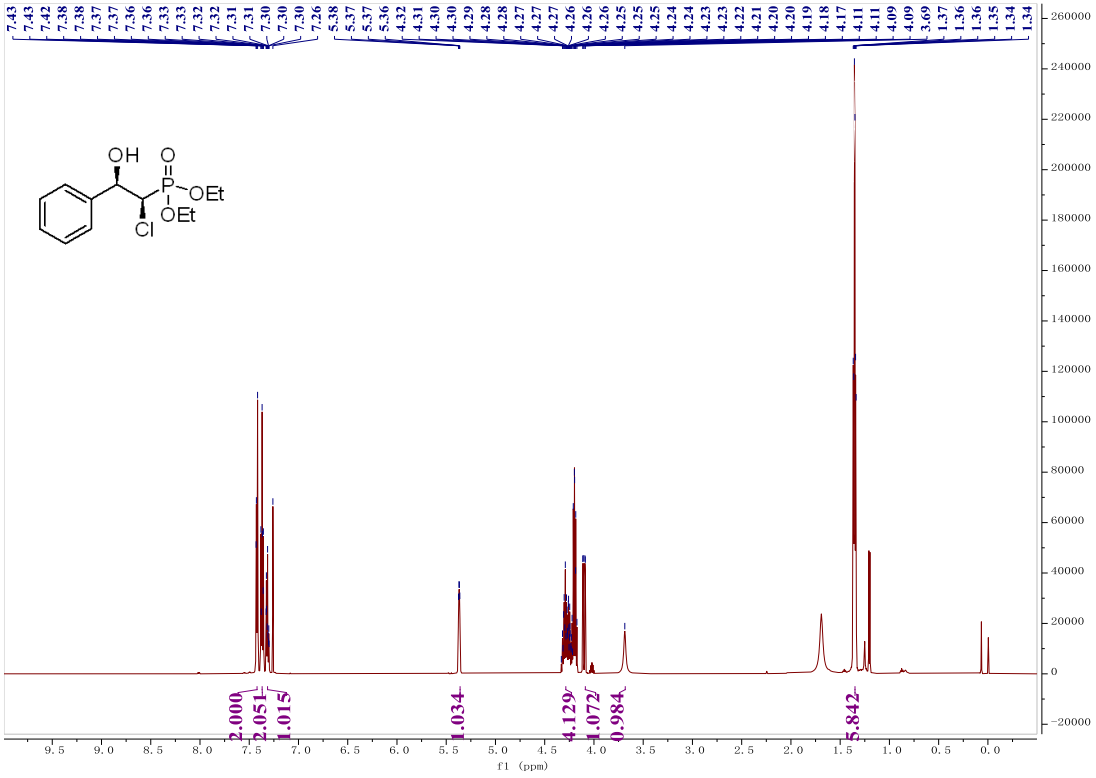
^{13}C NMR of **2f**, (101 MHz, Chloroform-*d*).



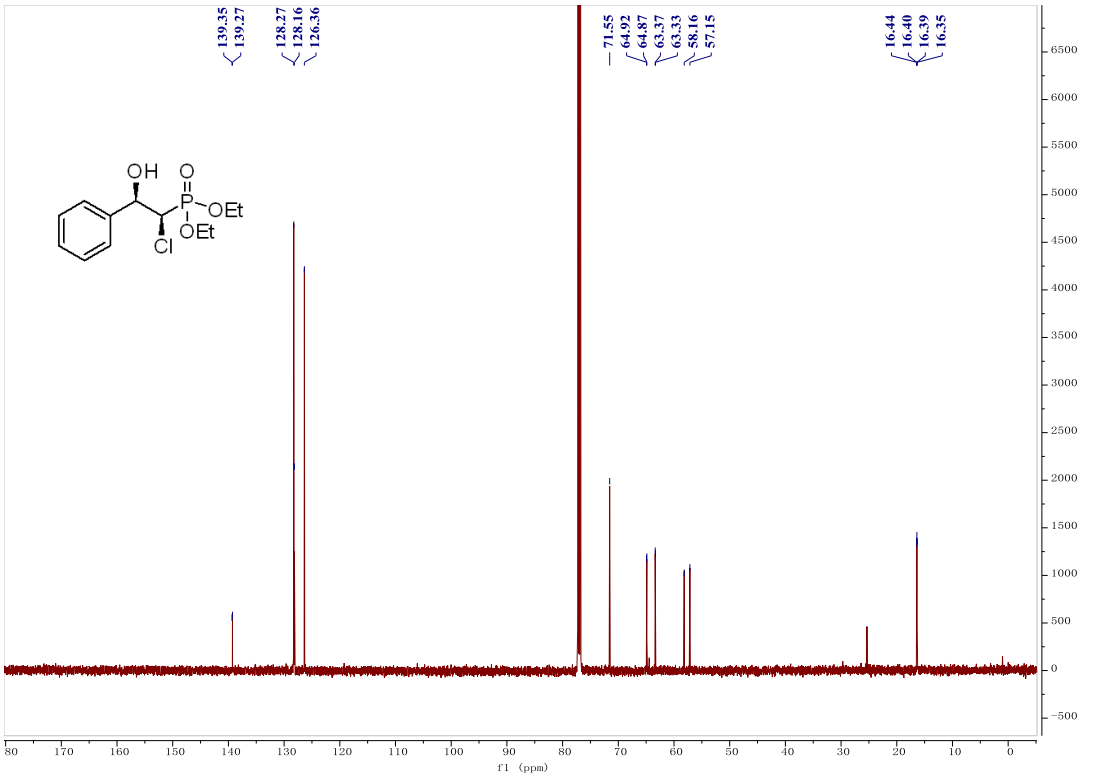
^{31}P NMR of **2f**, (162 MHz, Chloroform-*d*).



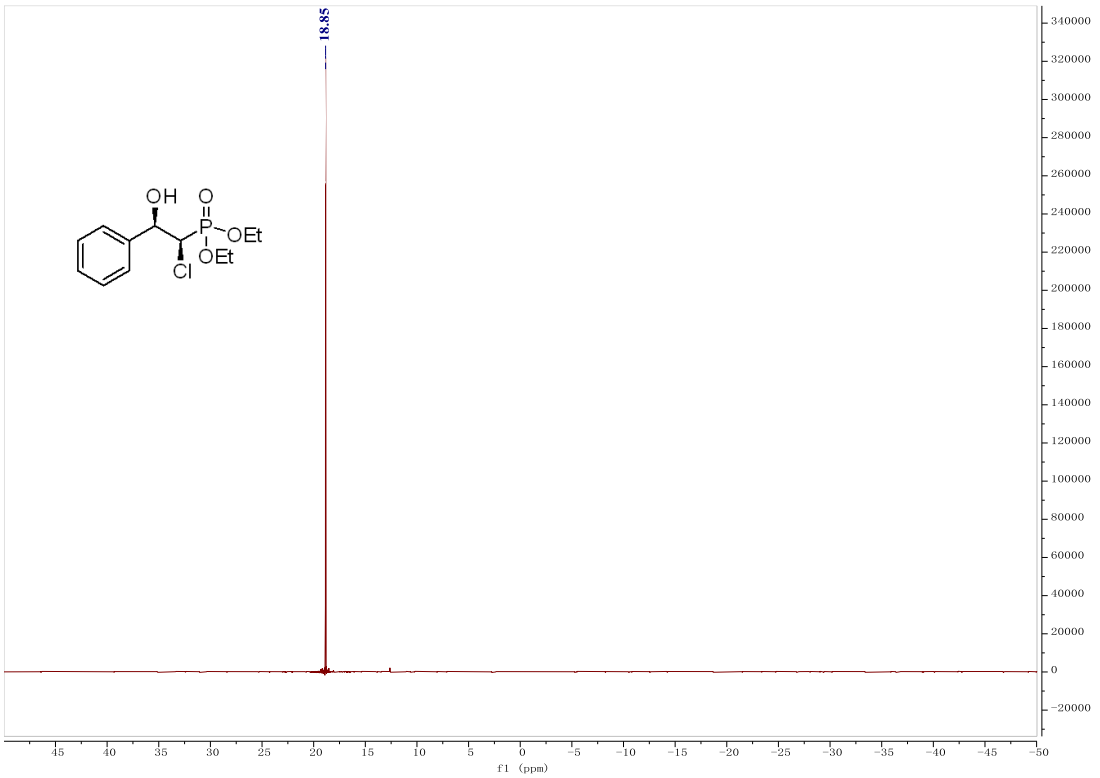
^{1}H NMR of **2h**, (400 MHz, Chloroform-*d*)



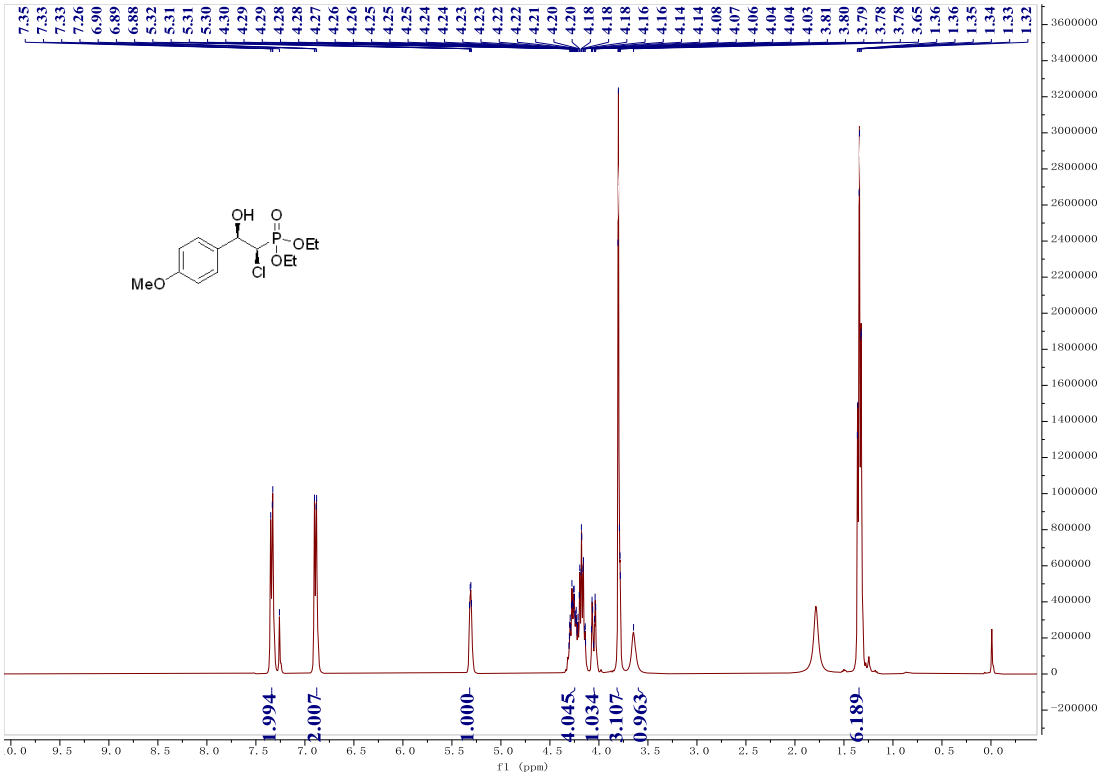
^{13}C NMR of **2h**, (101 MHz, Chloroform-*d*)



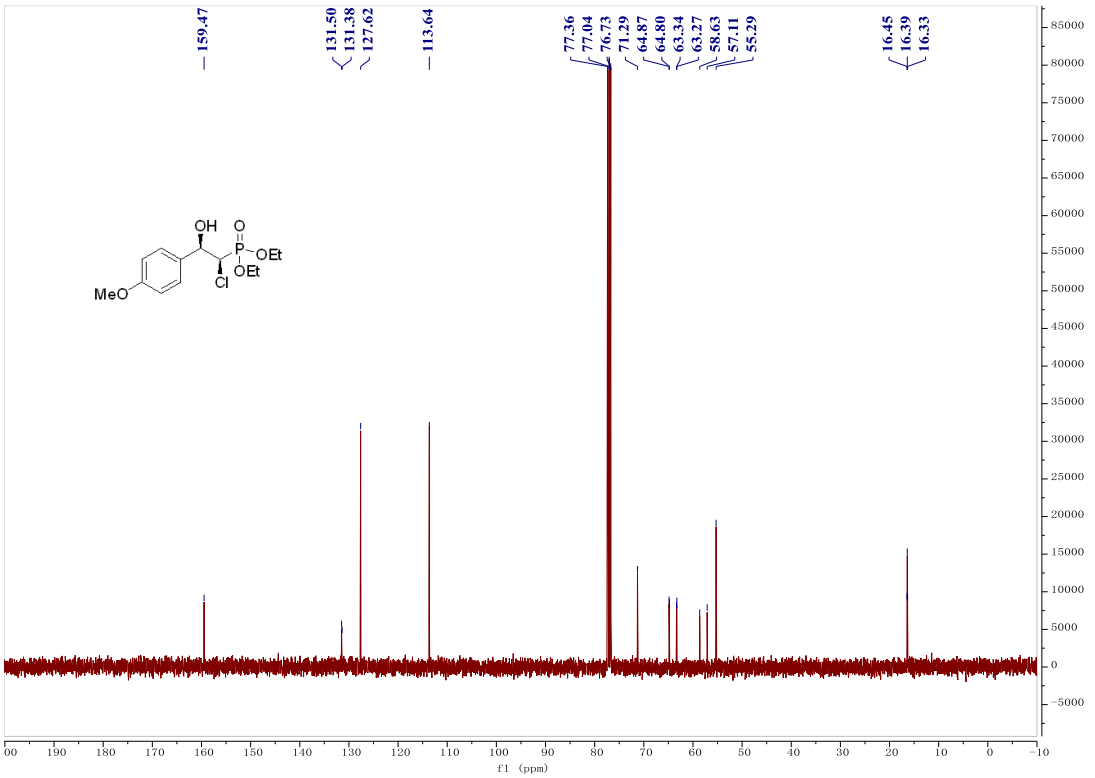
^{31}P NMR of **2h**, (162 MHz, Chloroform-*d*)



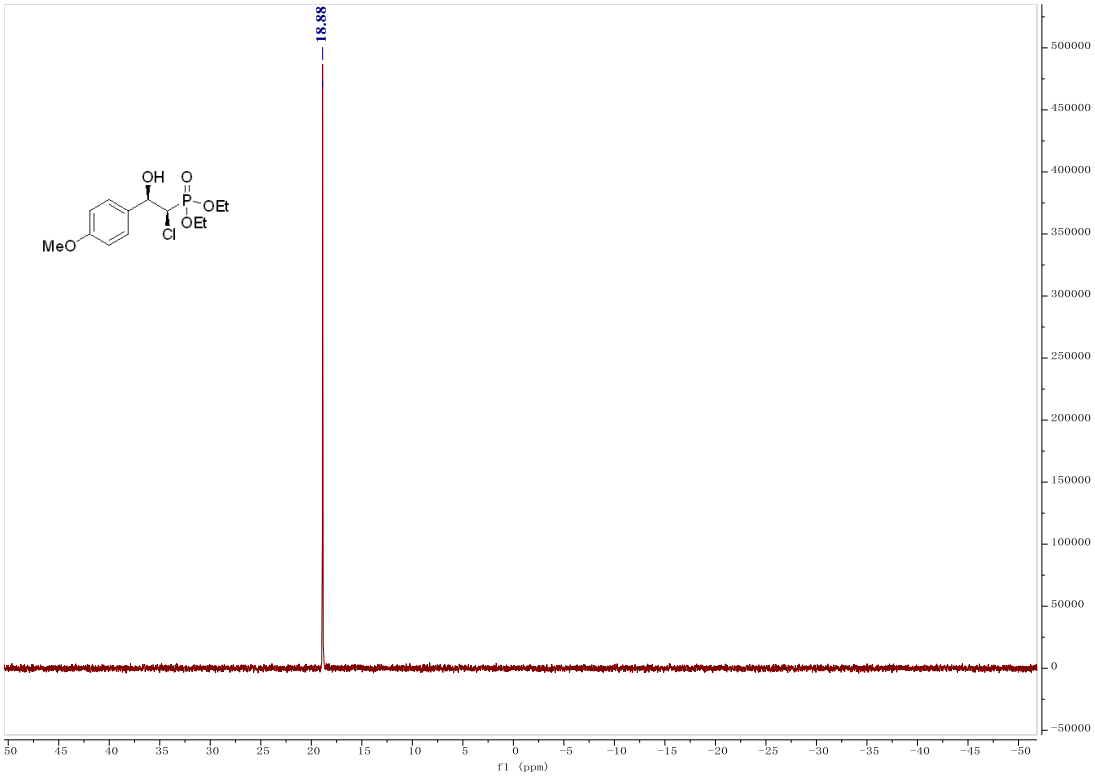
^{1}H NMR of **2i**, (400 MHz, Chloroform-*d*)



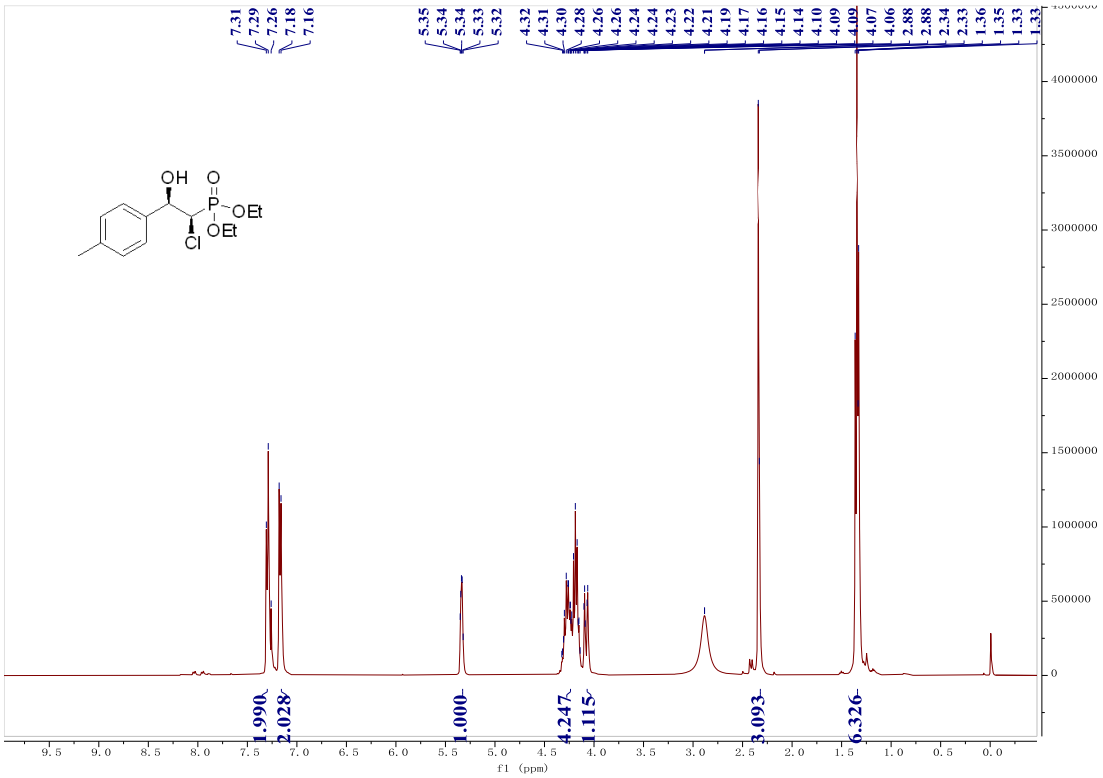
^{13}C NMR of **2i**, (101 MHz, Chloroform-*d*)



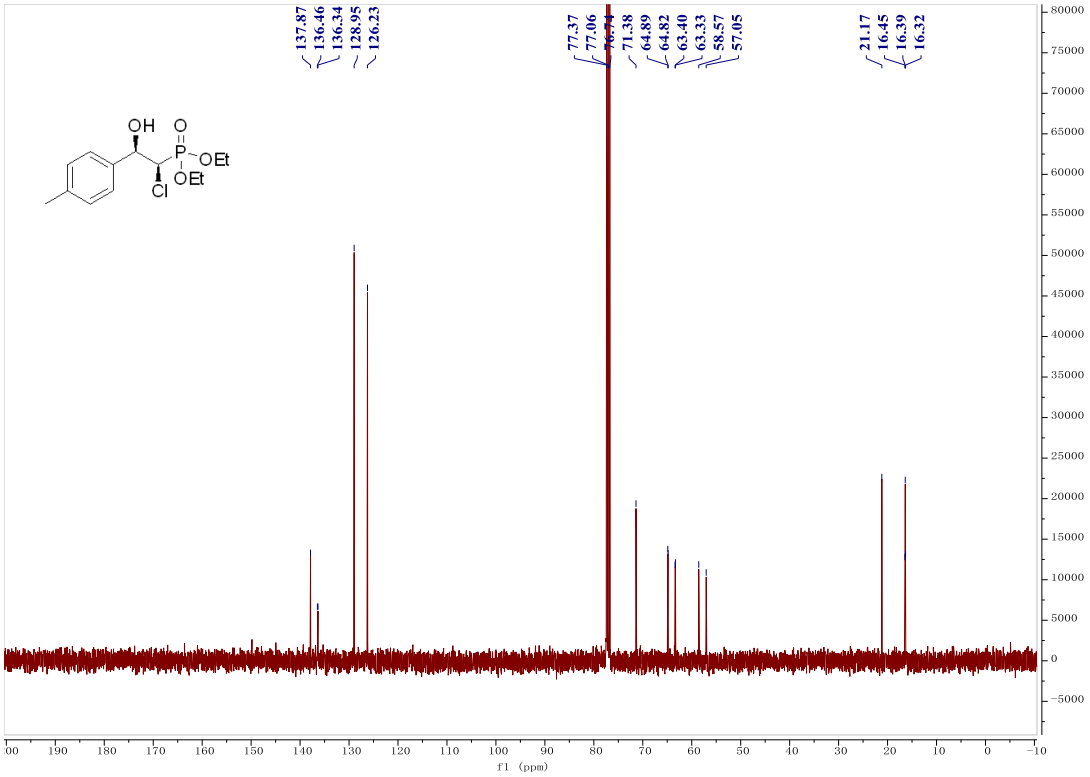
^{31}P NMR of **2i**, (162 MHz, Chloroform-*d*)



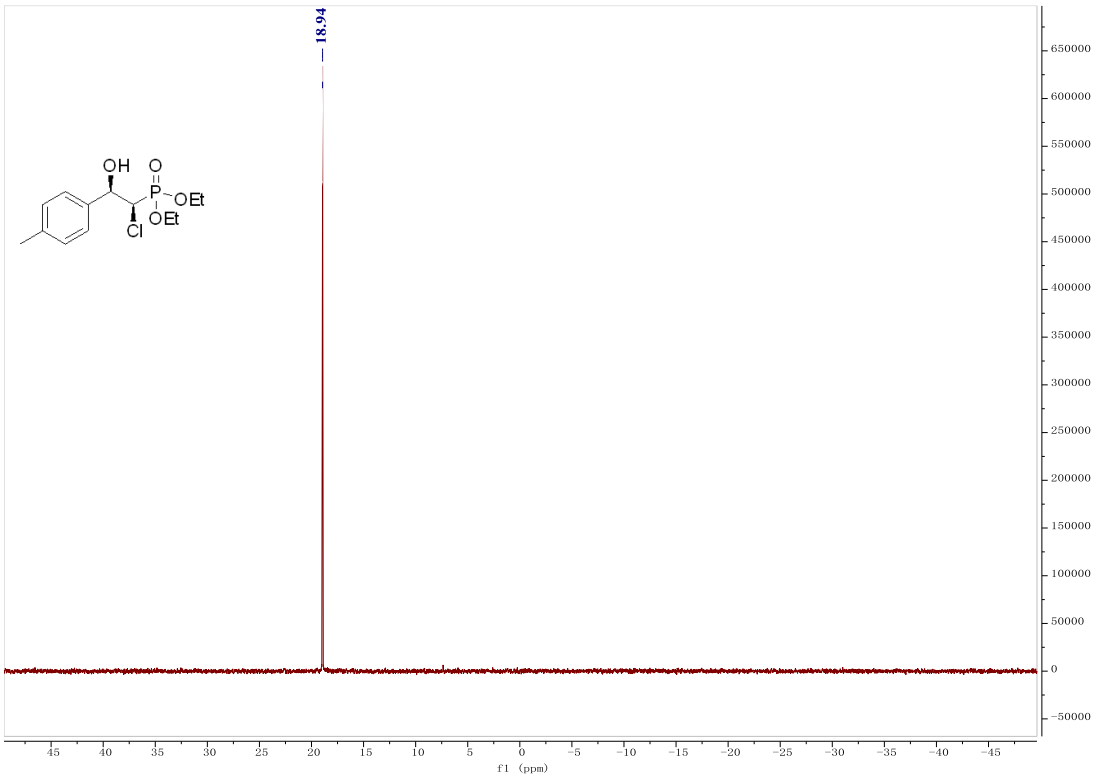
^{1}H NMR of **2j**, (400 MHz, Chloroform-*d*)



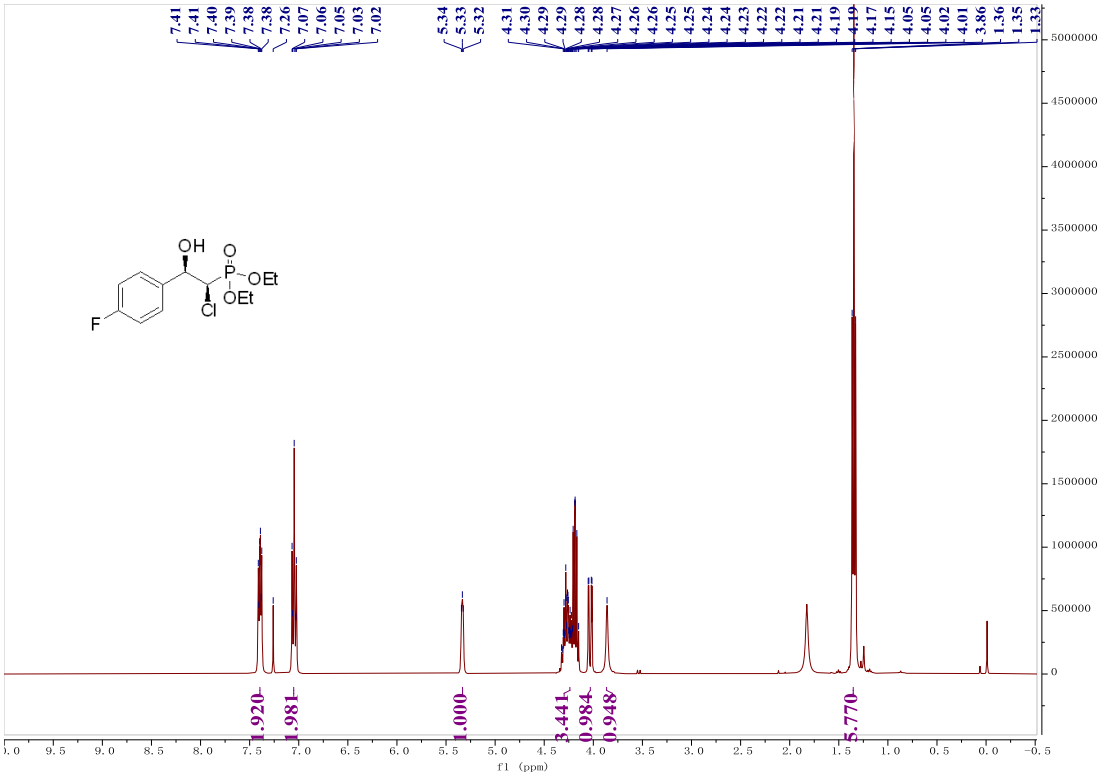
^{13}C NMR of **2j**, (101 MHz, Chloroform-*d*)



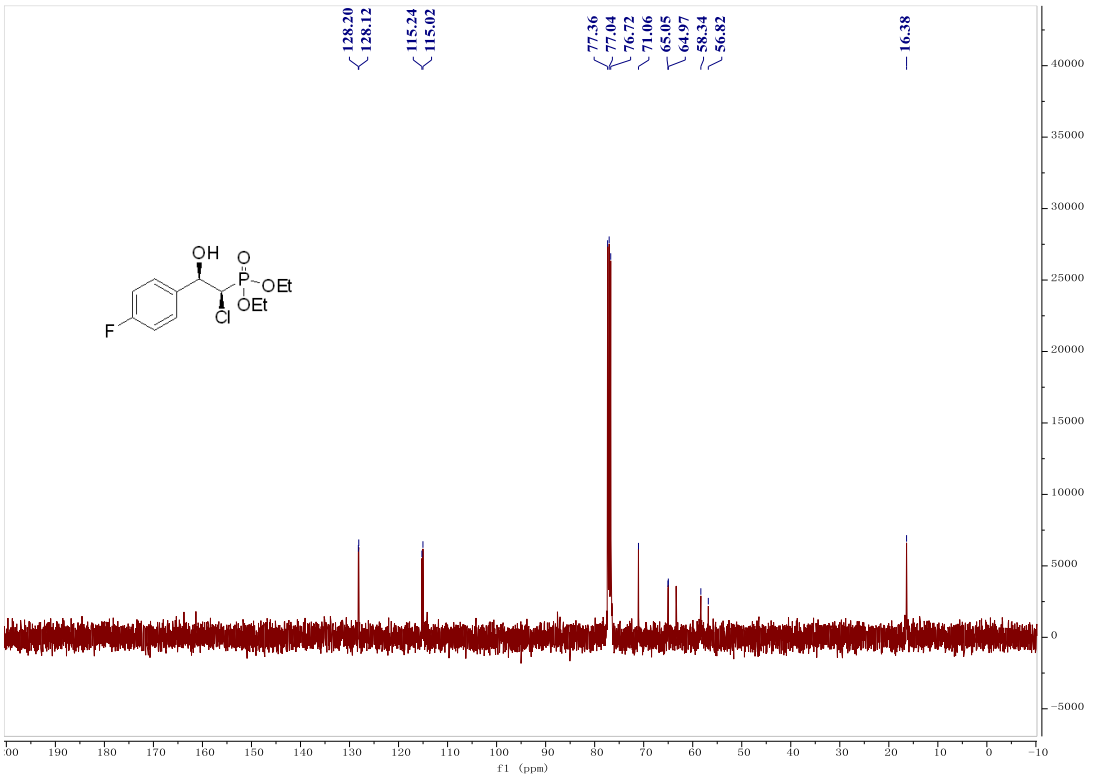
^{31}P NMR of **2j**, (162 MHz, Chloroform-*d*)



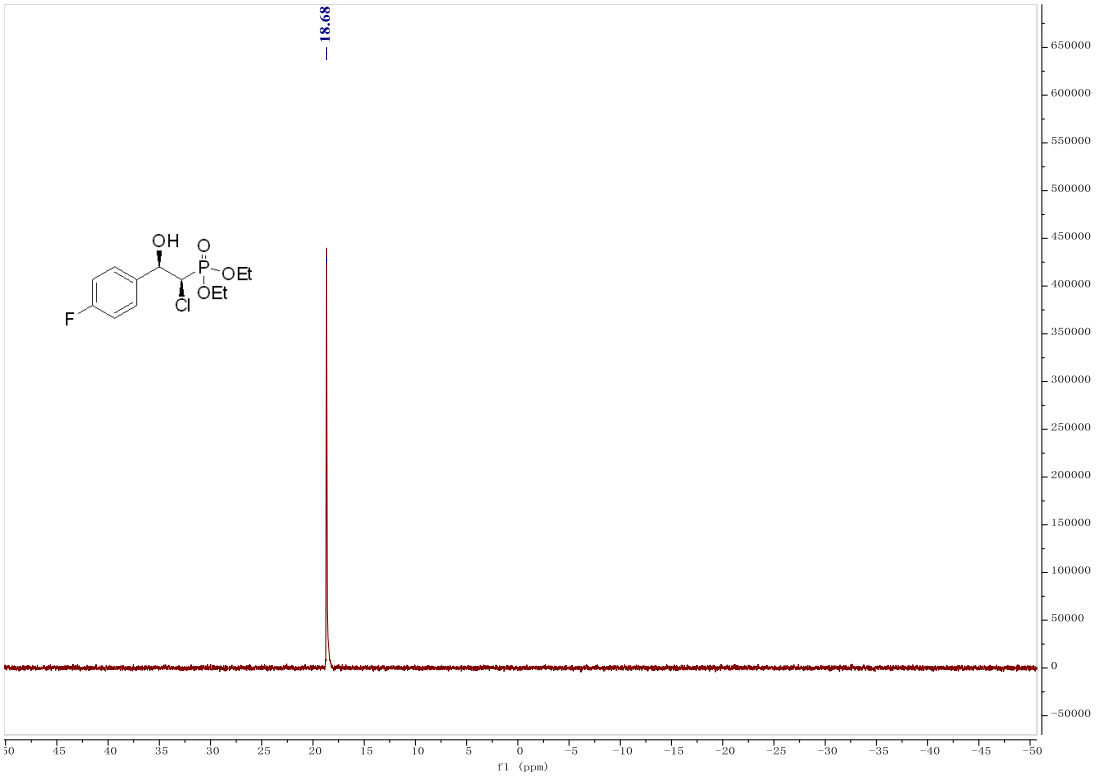
^{1}H NMR of **2k**, (400 MHz, Chloroform-*d*)



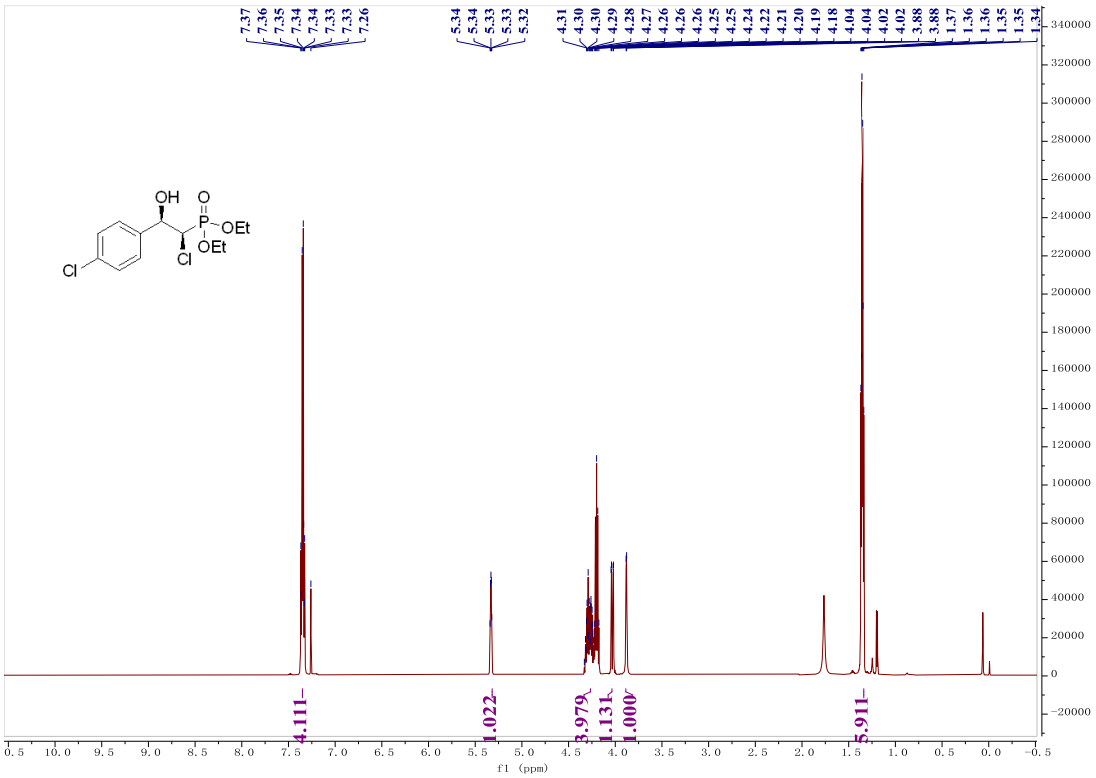
^{13}C NMR of **2k**, (101 MHz, Chloroform-*d*)



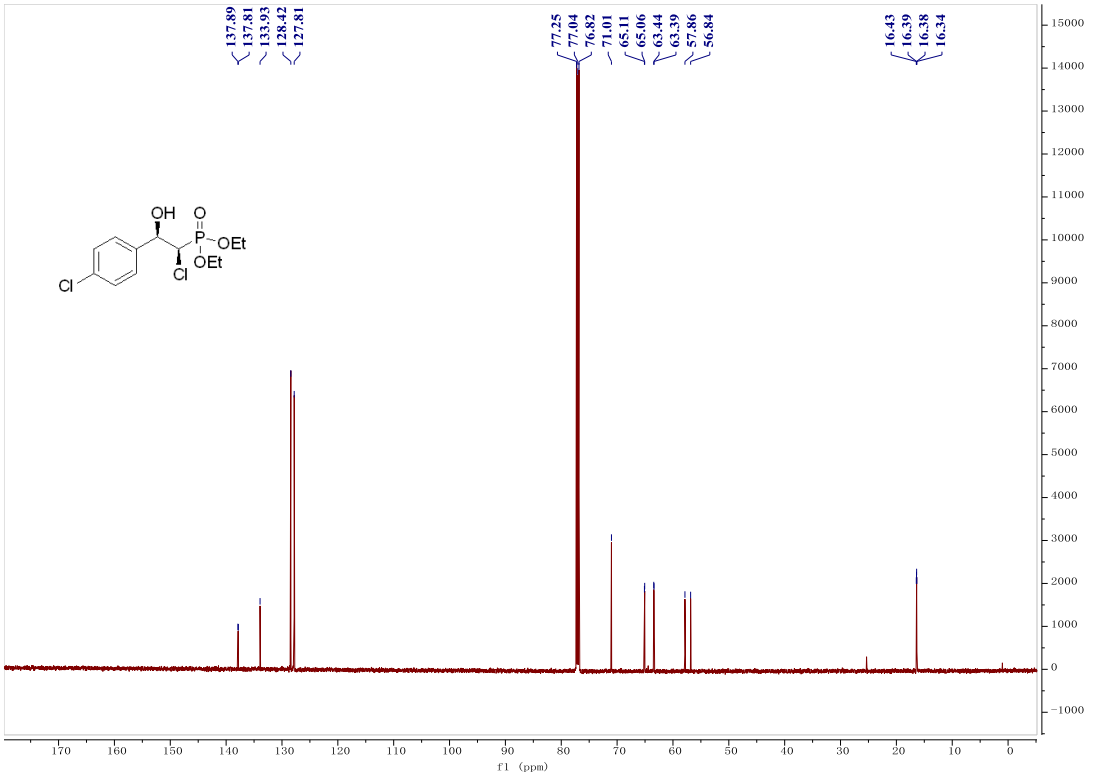
^{31}P NMR of **2k**, (162 MHz, Chloroform-*d*)



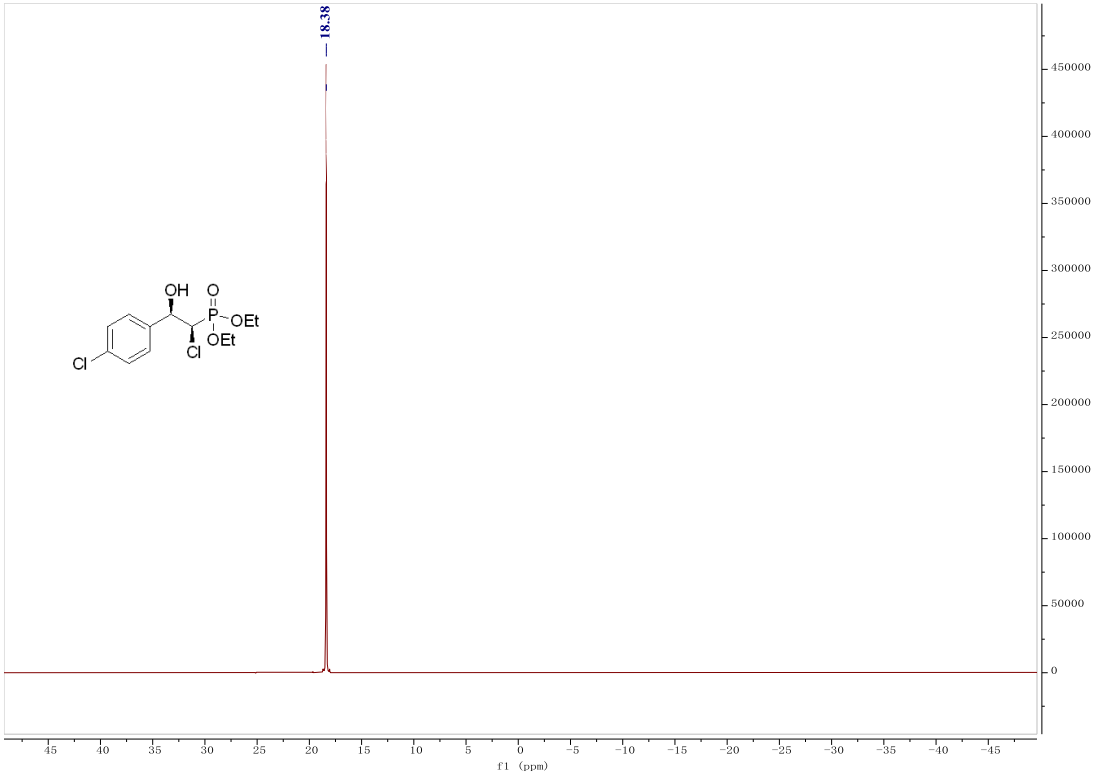
^{1}H NMR of **2l**, (400 MHz, Chloroform-*d*)



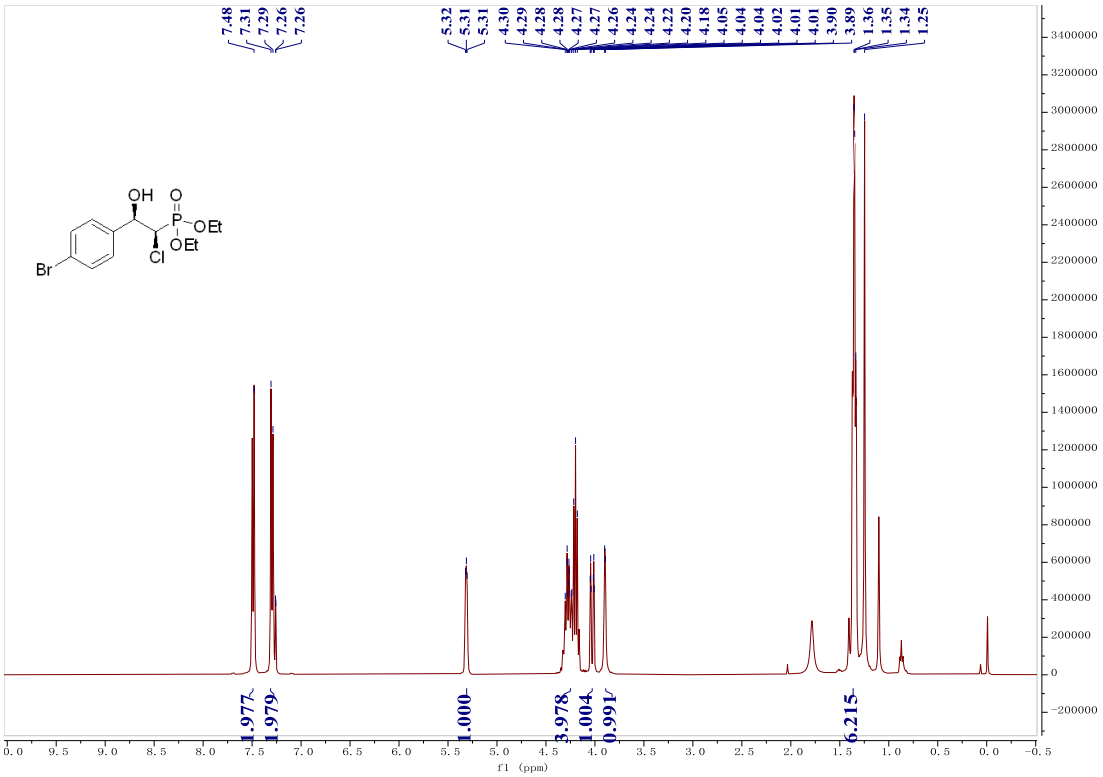
^{13}C NMR of **2l**, (101 MHz, Chloroform-*d*)



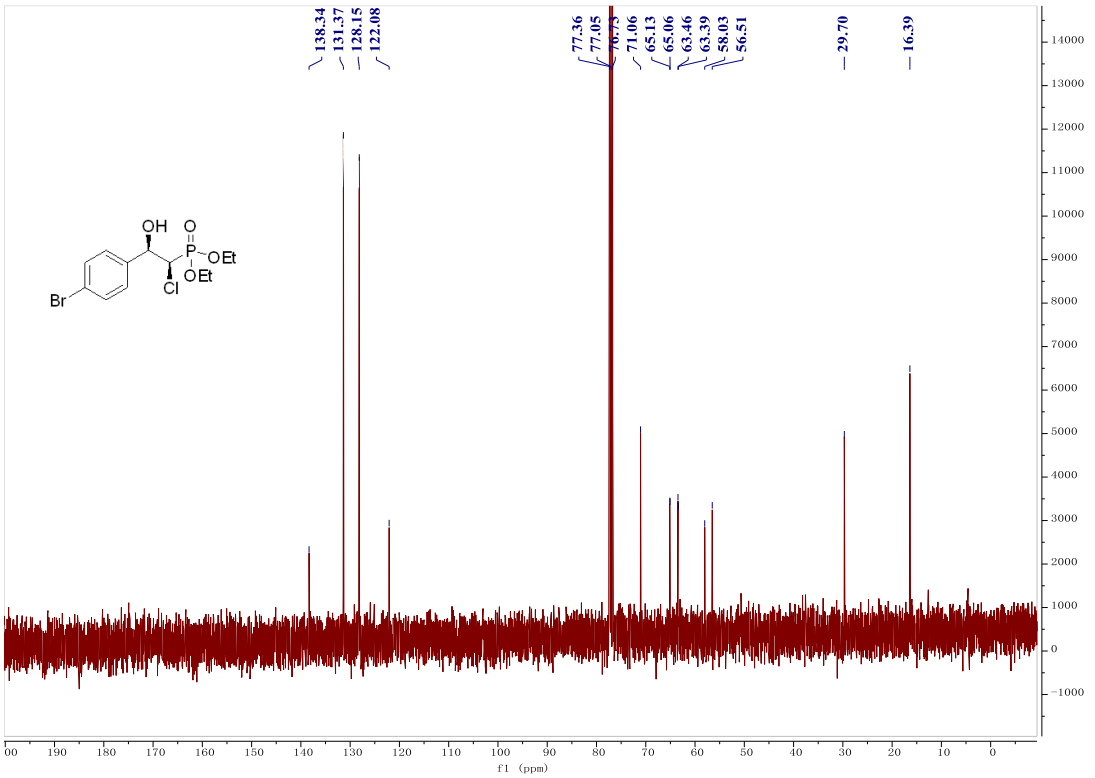
^{31}P NMR of **2l**, (162 MHz, Chloroform-*d*)



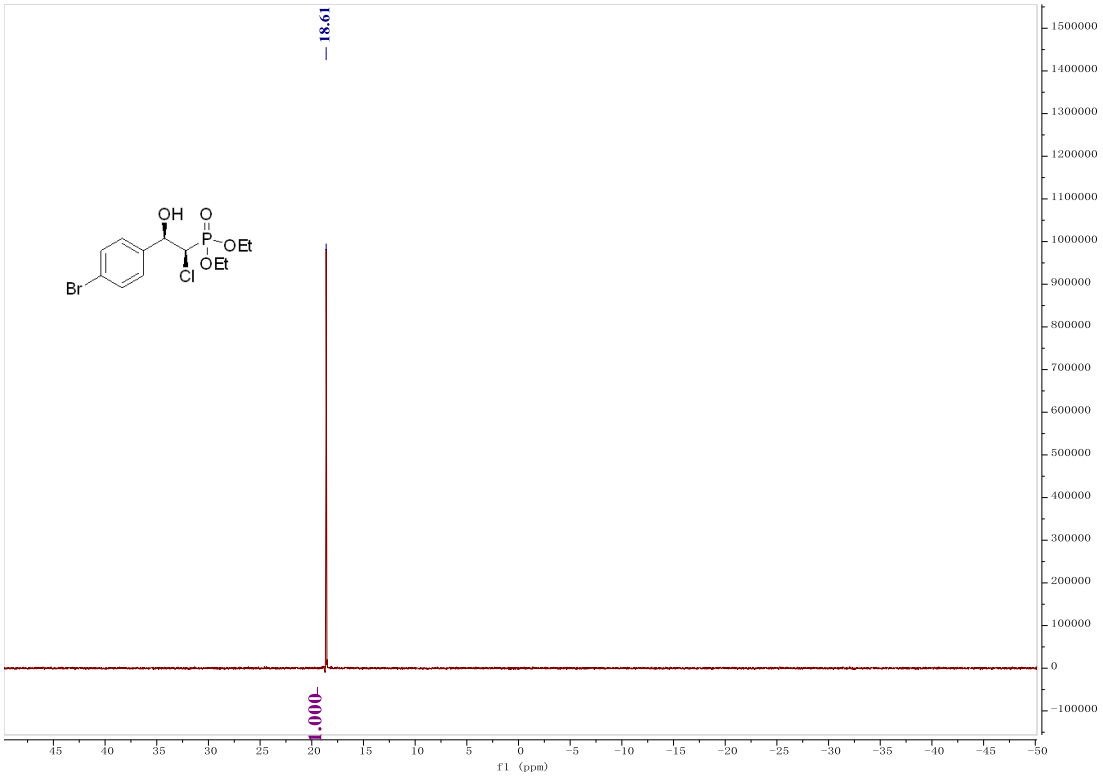
^{1}H NMR of **2m**, (400 MHz, Chloroform-*d*)



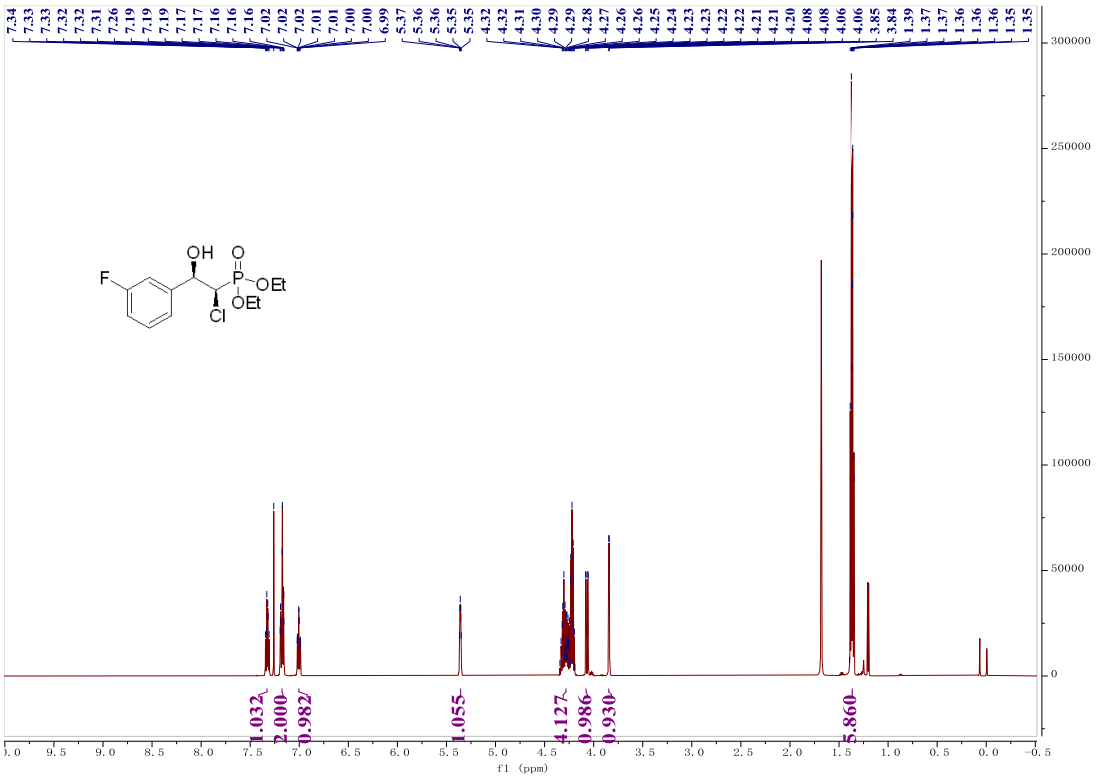
^{13}C NMR of **2m**, (101 MHz, Chloroform-*d*)



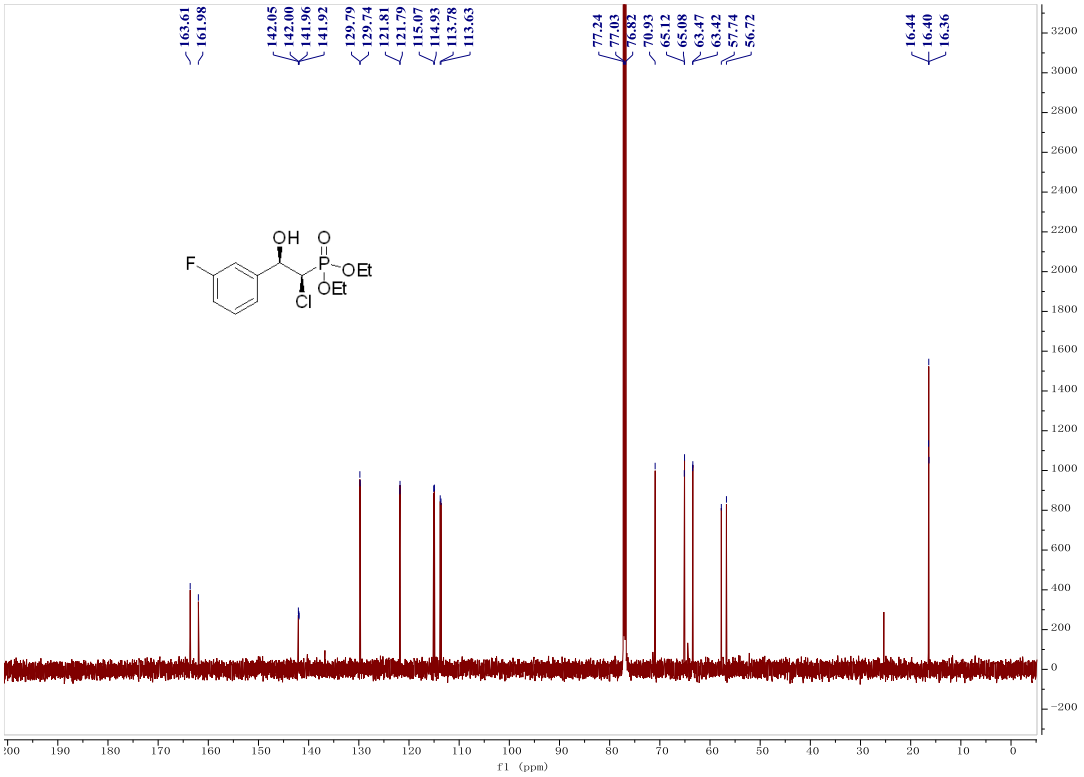
^{31}P NMR of **2m**, (162 MHz, Chloroform-*d*)



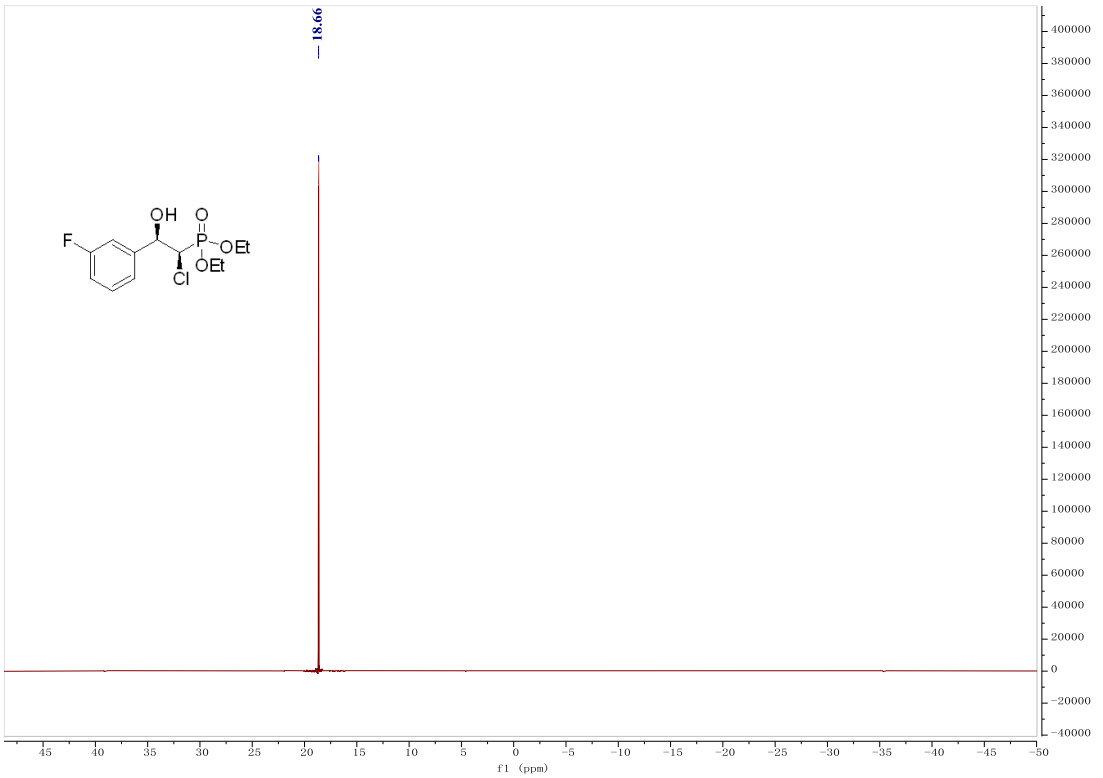
^{1}H NMR of **2n**, (400 MHz, Chloroform-*d*)



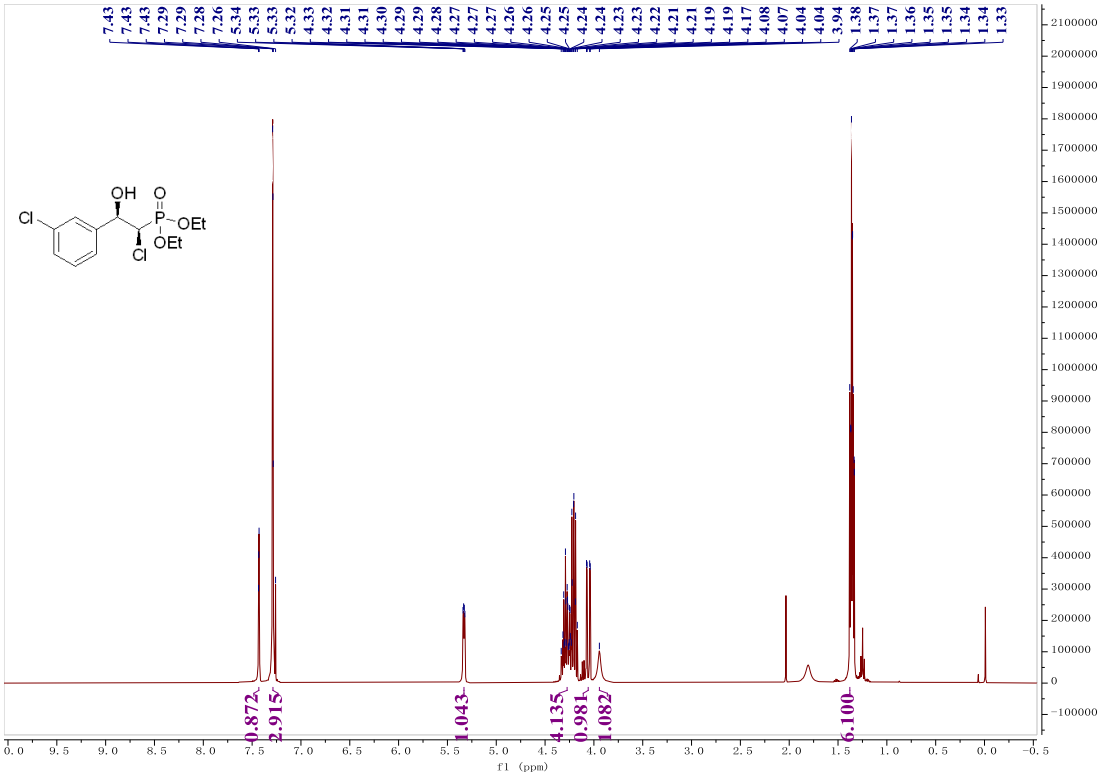
^{13}C NMR of **2n**, (101 MHz, Chloroform-*d*)



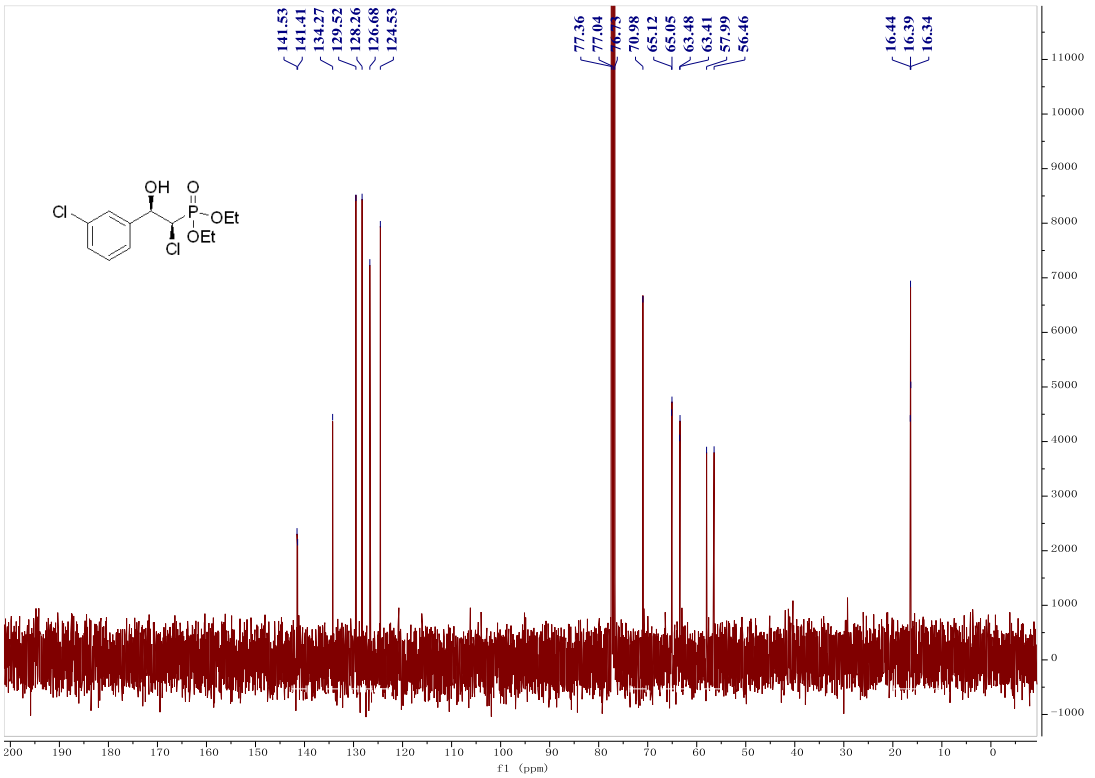
^{31}P NMR of **2n**, (162 MHz, Chloroform-*d*)



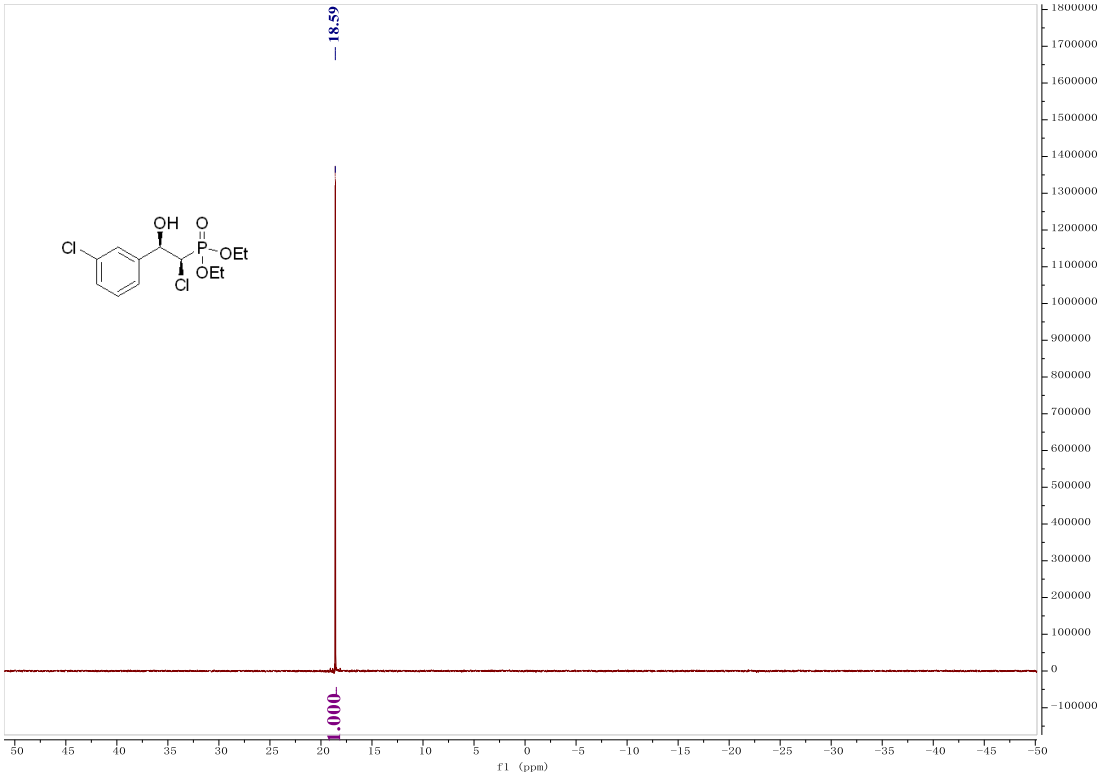
^{1}H NMR of **2o**, (400 MHz, Chloroform-*d*)



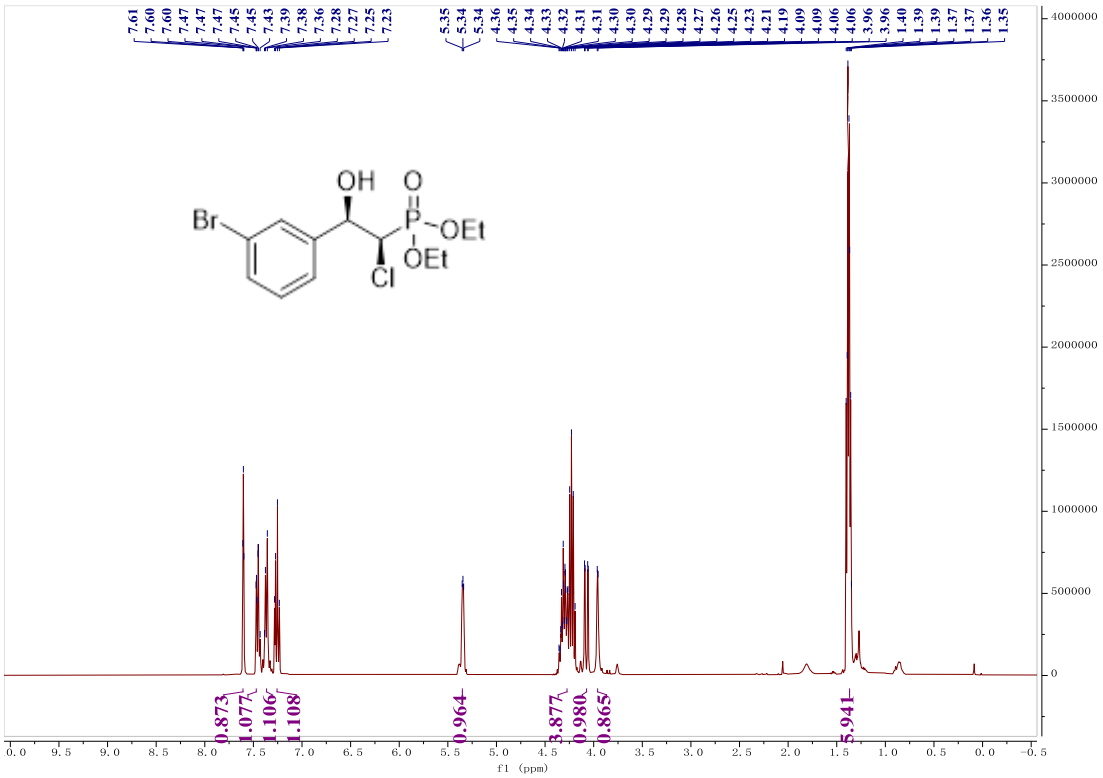
^{13}C NMR of **2o**, (101 MHz, Chloroform-*d*)



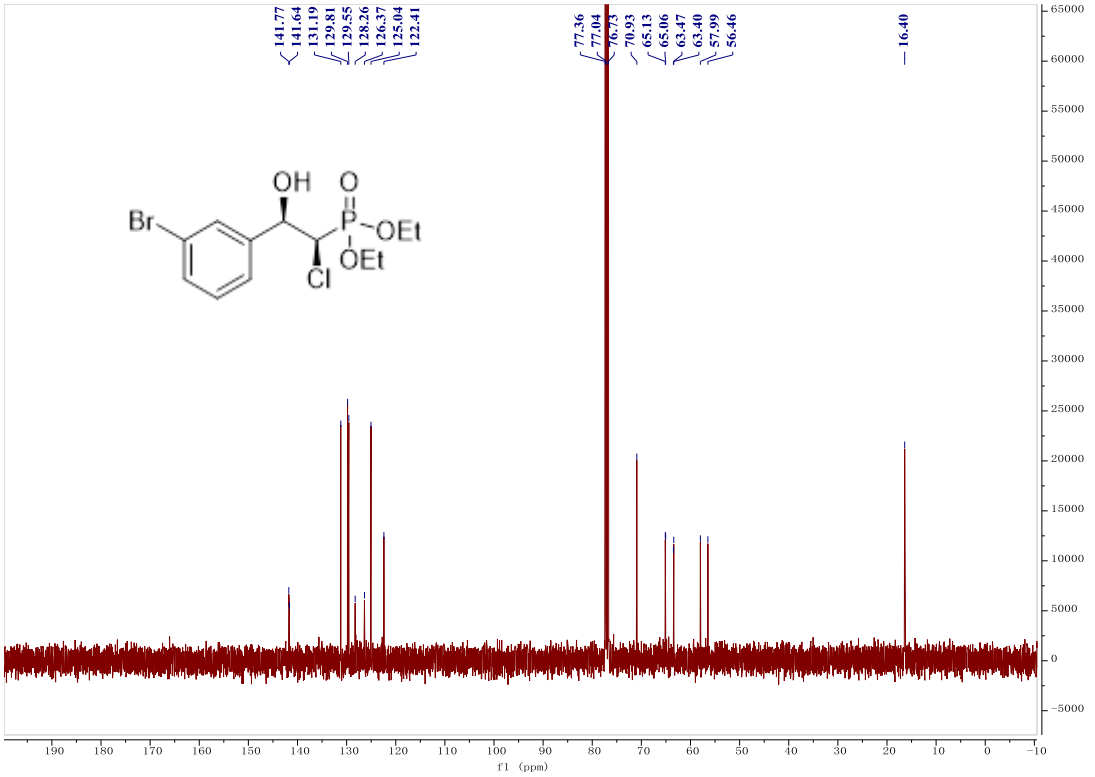
^{31}P NMR of **2o**, (162 MHz, Chloroform-*d*)



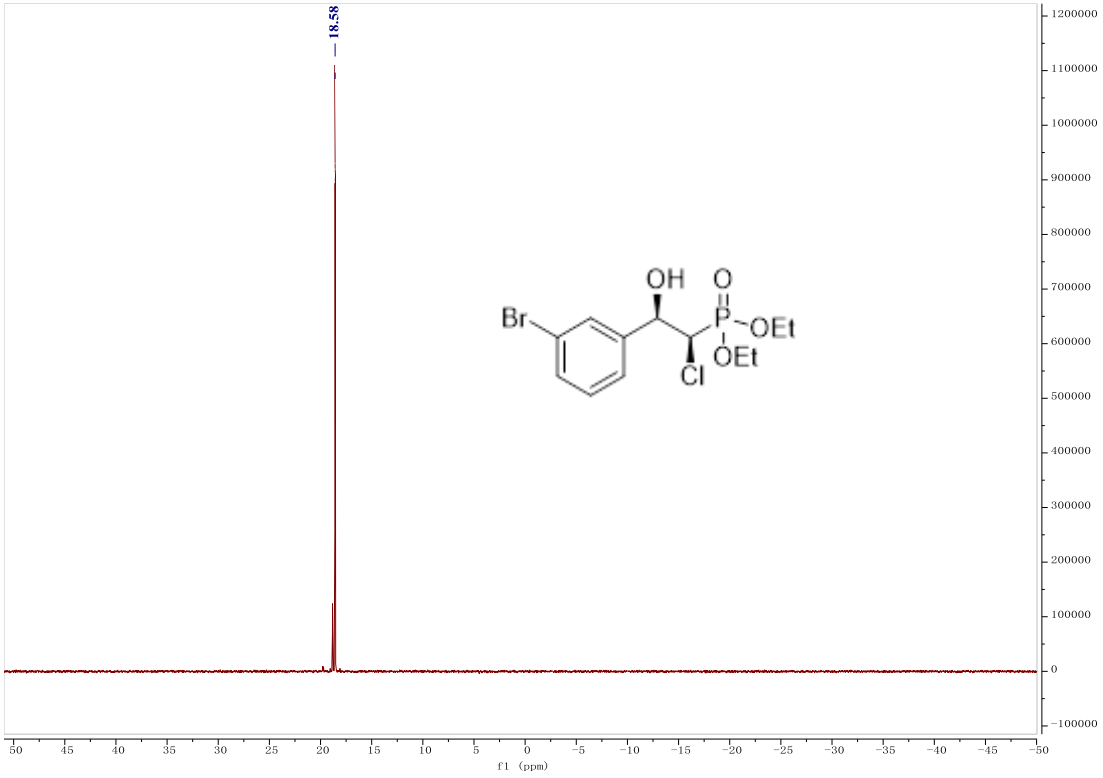
^{1}H NMR of **2p**, (400 MHz, Chloroform-*d*)



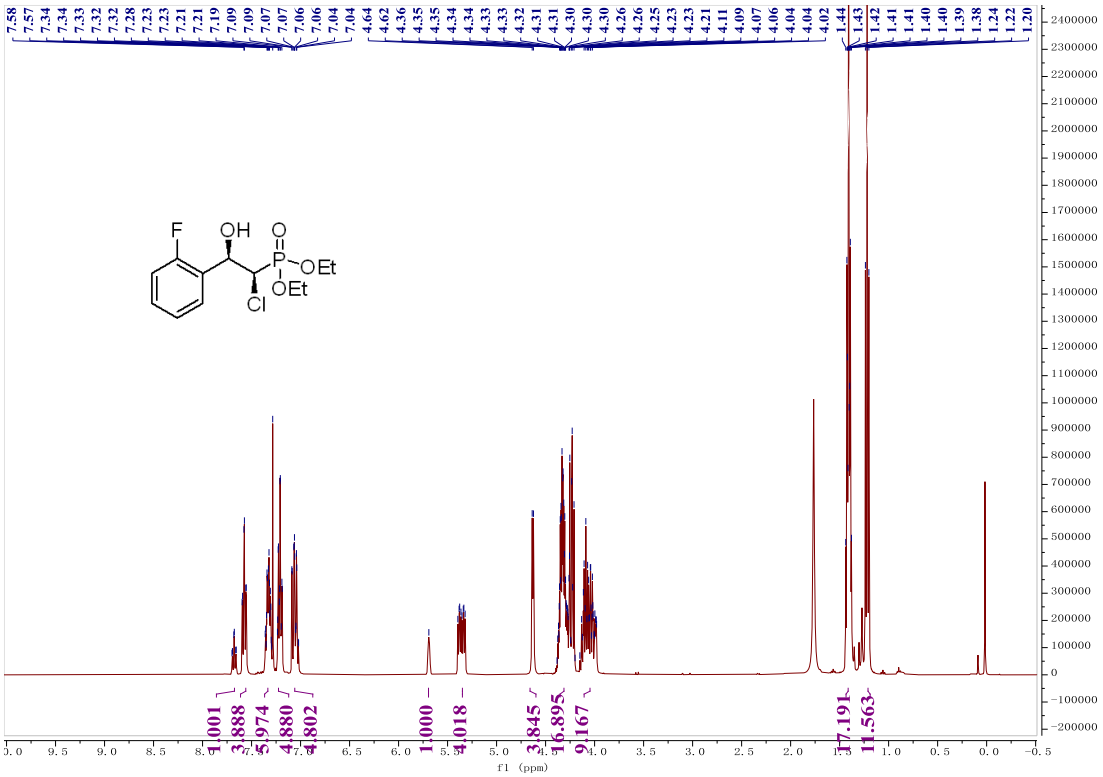
^{13}C NMR of **2p**, (101 MHz, Chloroform-*d*)



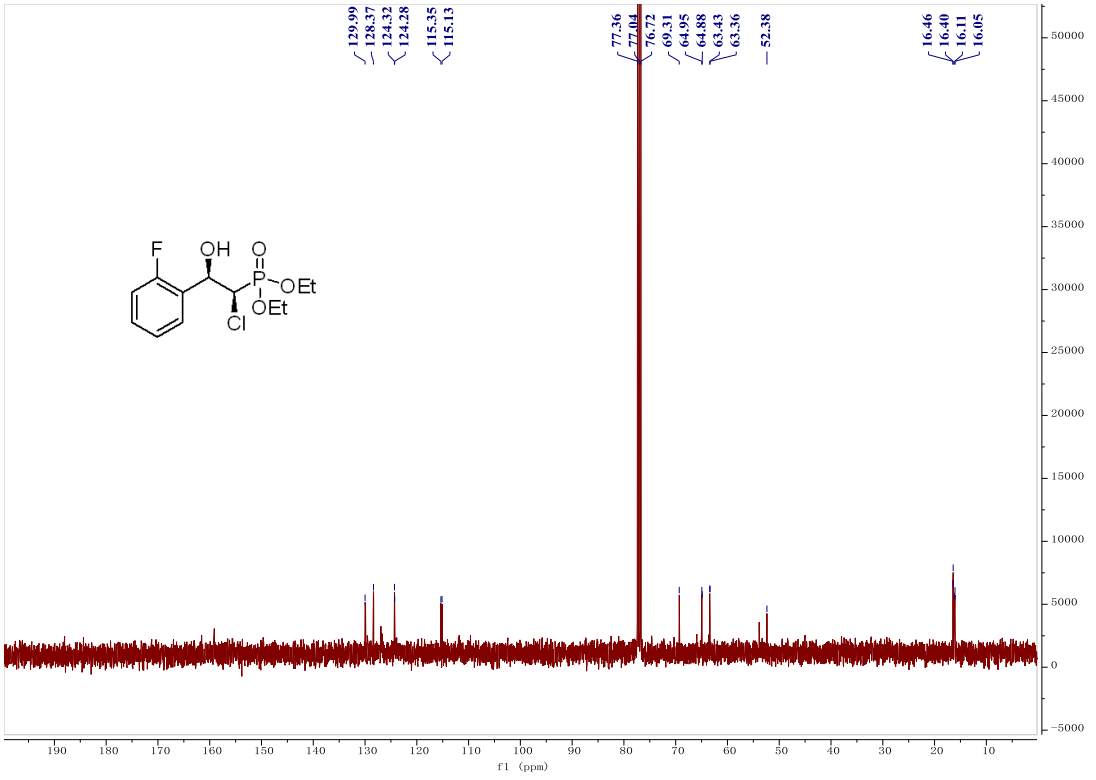
^{31}P NMR of **2p**, (162 MHz, Chloroform-*d*)



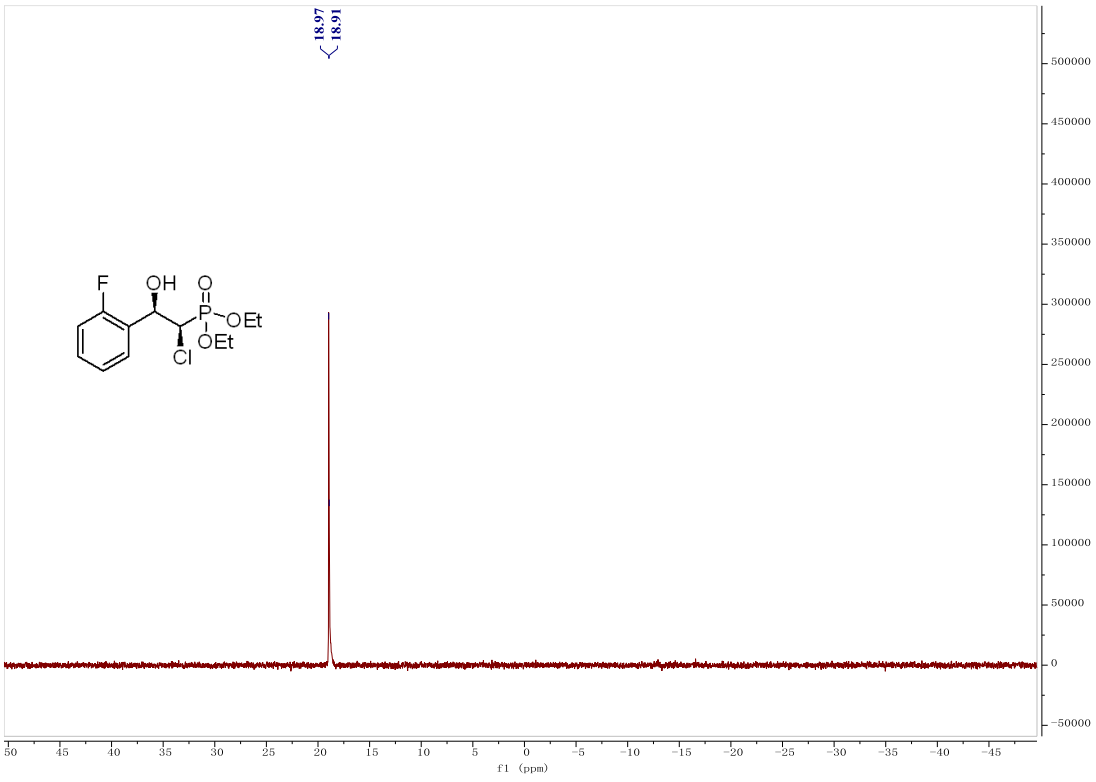
^{1}H NMR of **2q**, (400 MHz, Chloroform-*d*)



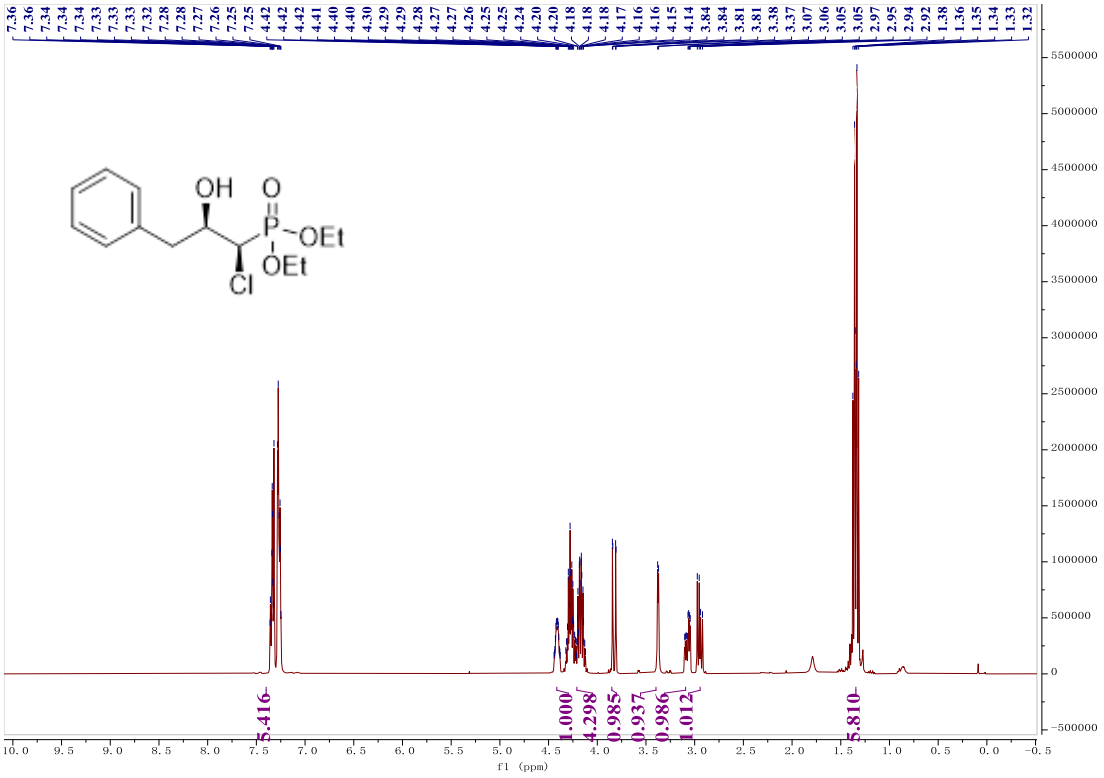
^{13}C NMR of **2q**, (101 MHz, Chloroform-*d*)



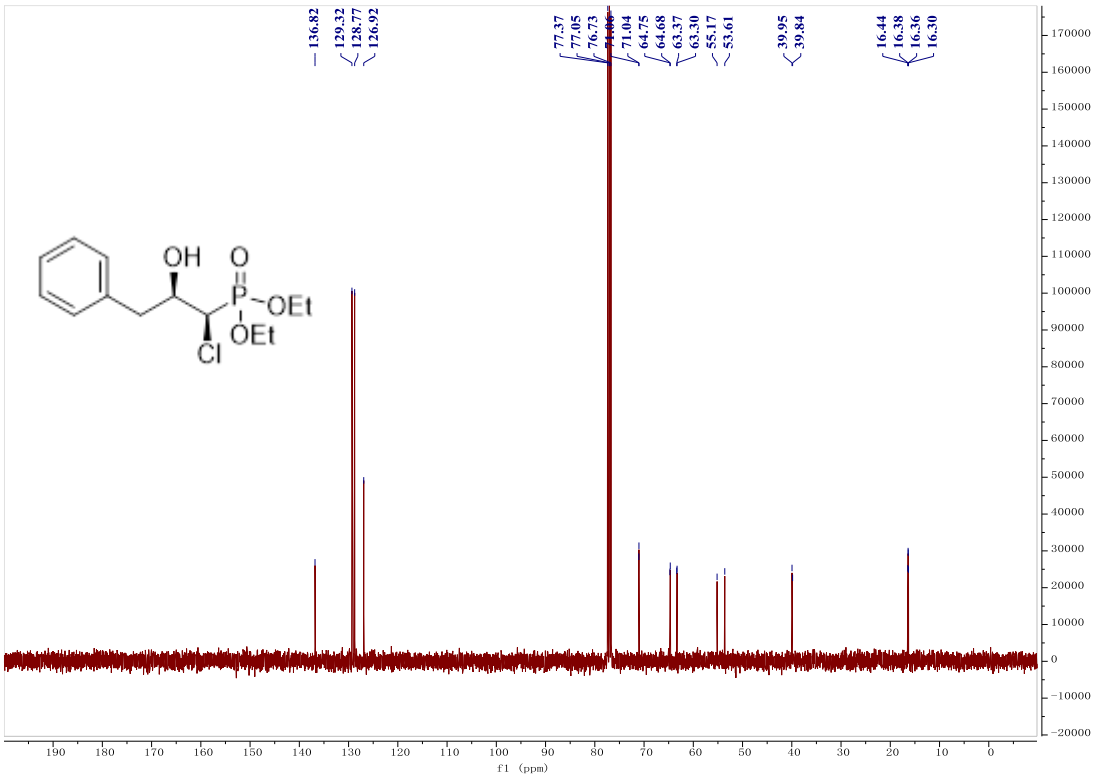
^{31}P NMR of **2q**, (162 MHz, Chloroform-*d*)



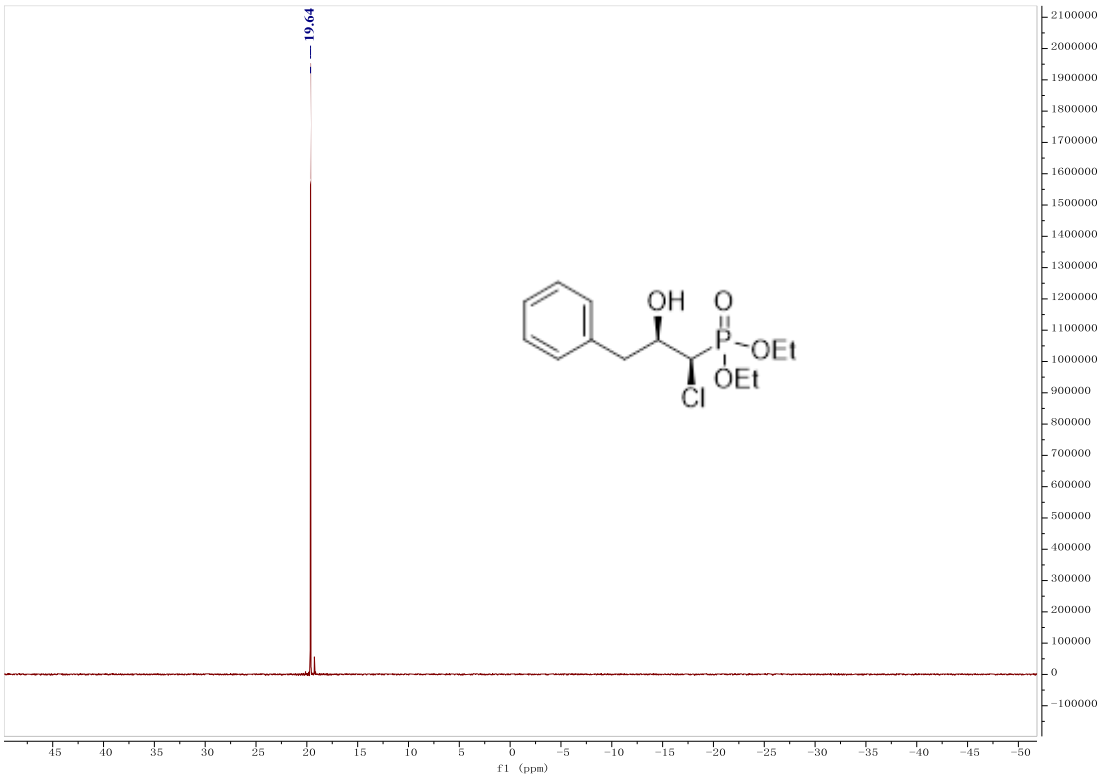
^{1}H NMR of **2r**, (400 MHz, Chloroform-*d*)



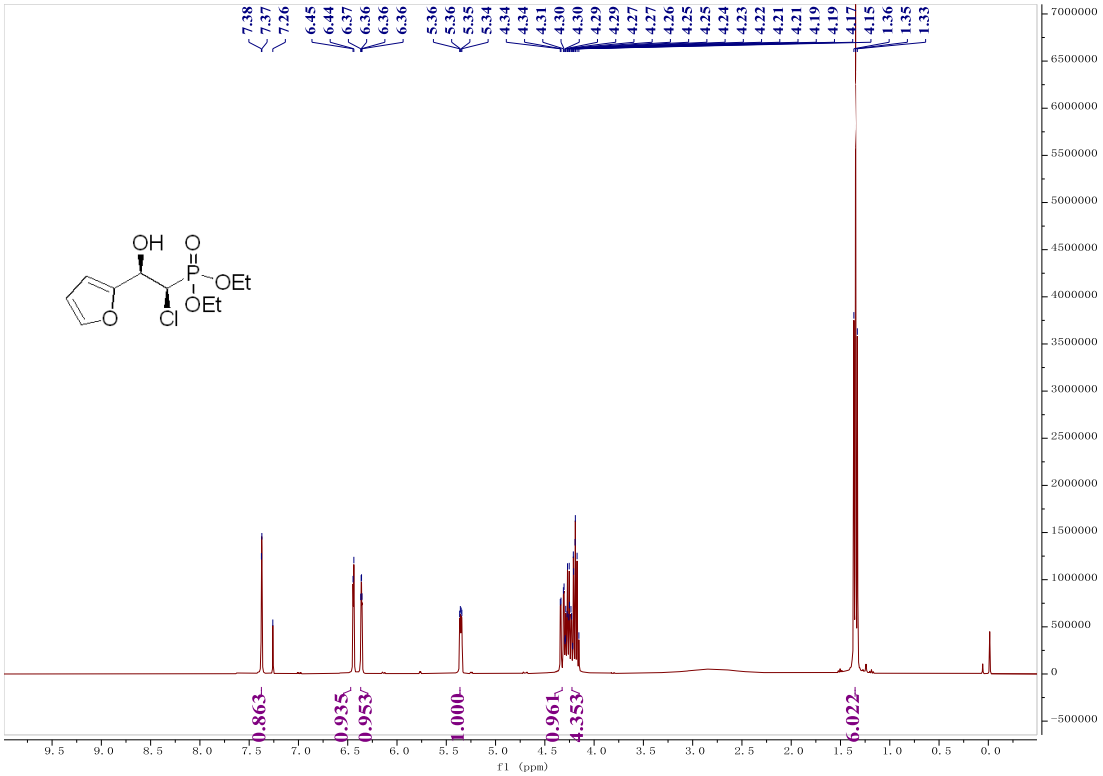
^{13}C NMR of **2r**, (101 MHz, Chloroform-*d*)



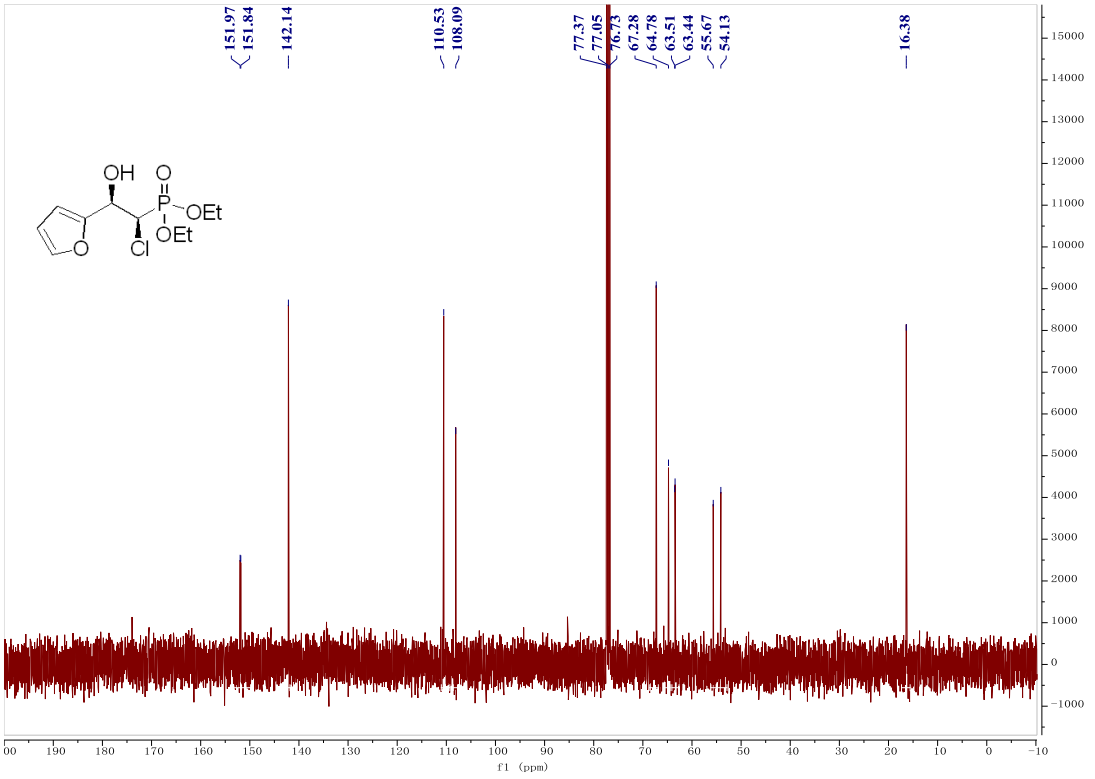
^{31}P NMR of **2r**, (162 MHz, Chloroform-*d*)



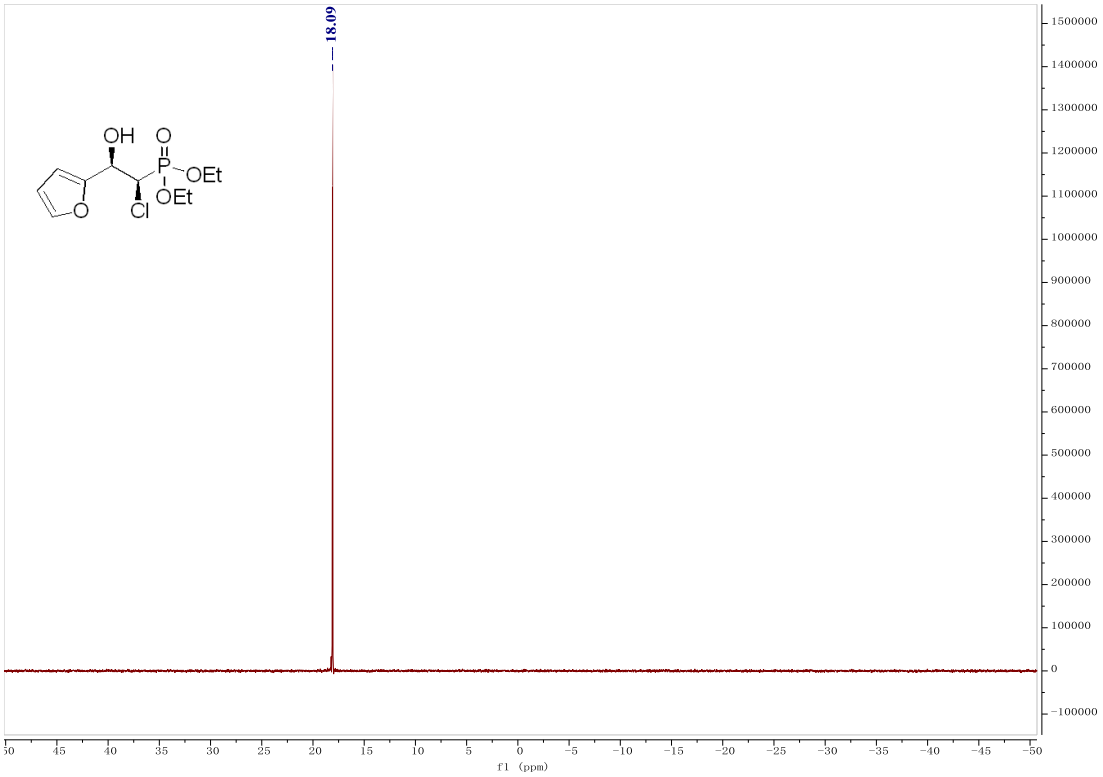
^{1}H NMR of **2s**, (400 MHz, Chloroform-*d*)



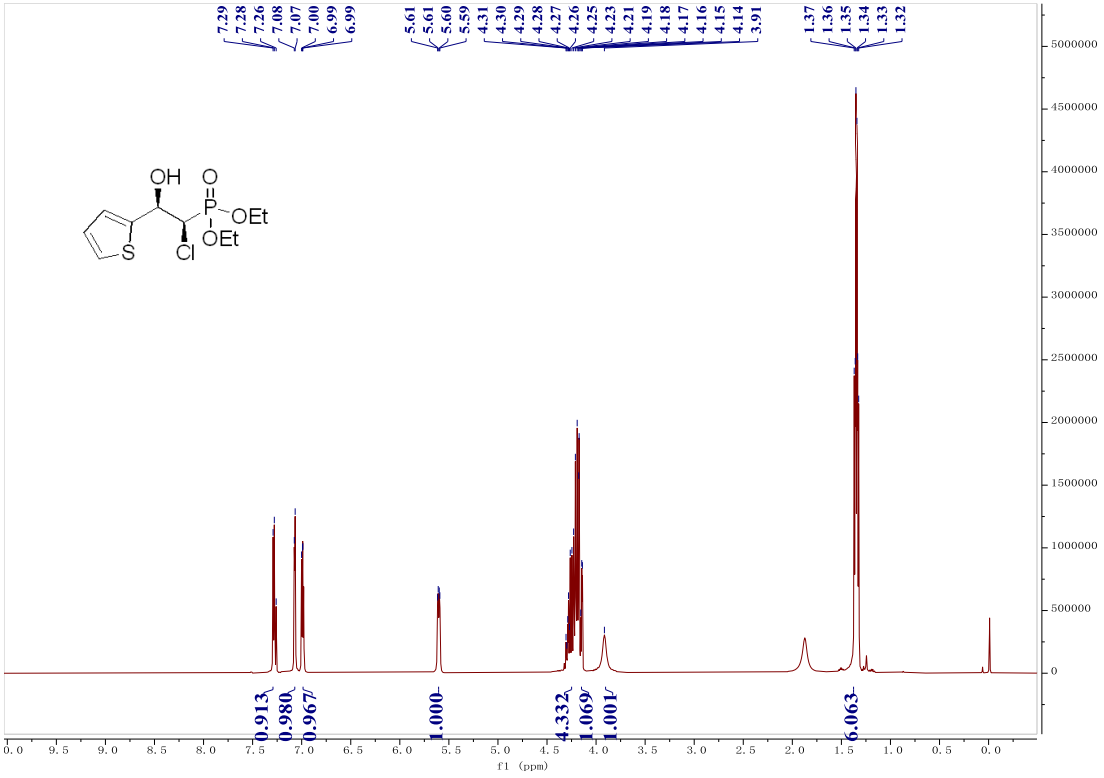
^{13}C NMR of **2s**, (101 MHz, Chloroform-*d*)



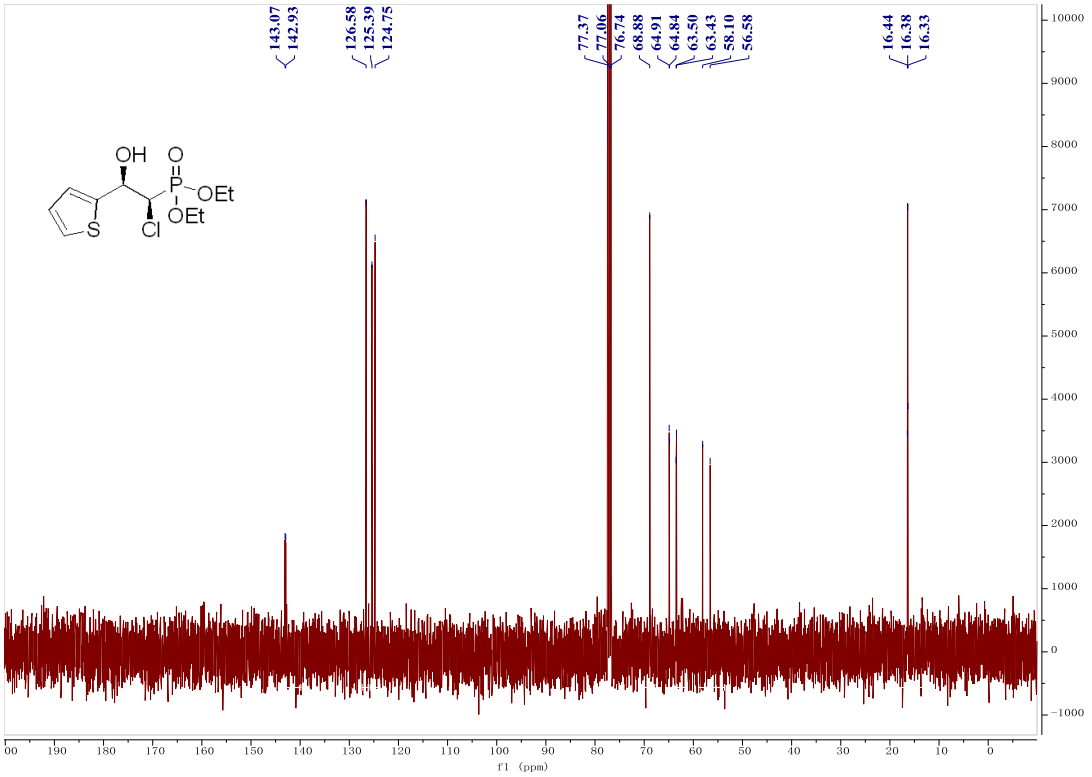
^{31}P NMR of **2s**, (162 MHz, Chloroform-*d*)



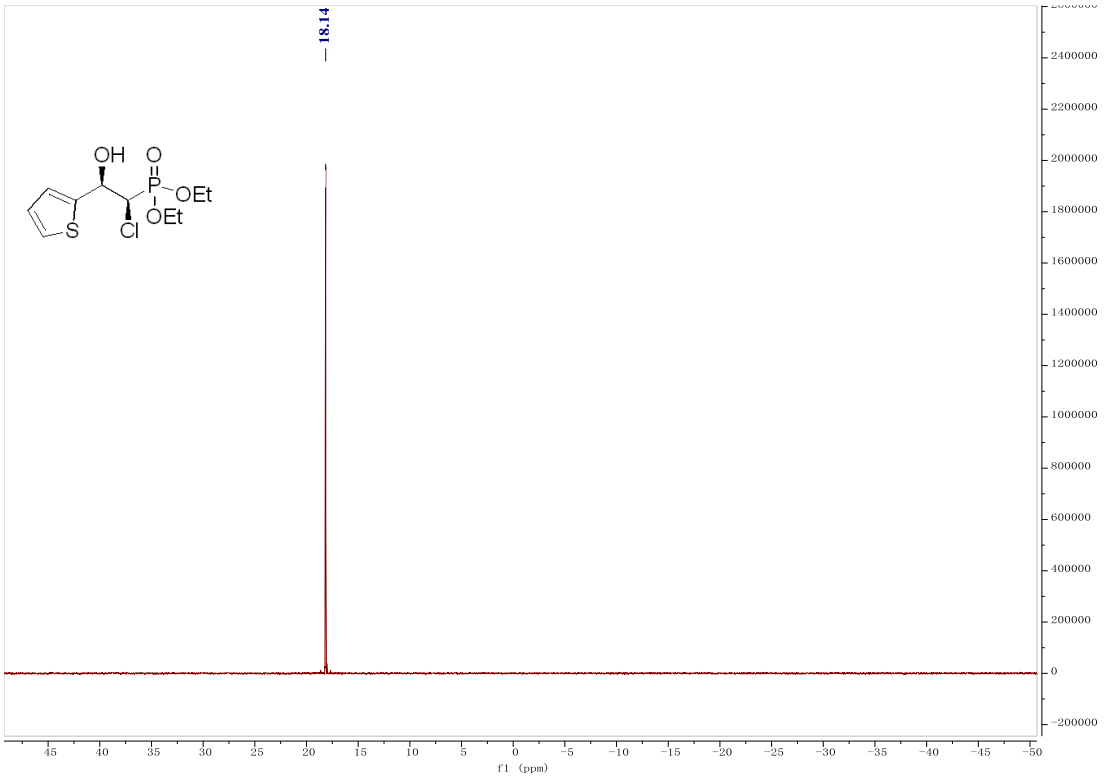
^{1}H NMR of **2t**, (400 MHz, Chloroform-*d*)



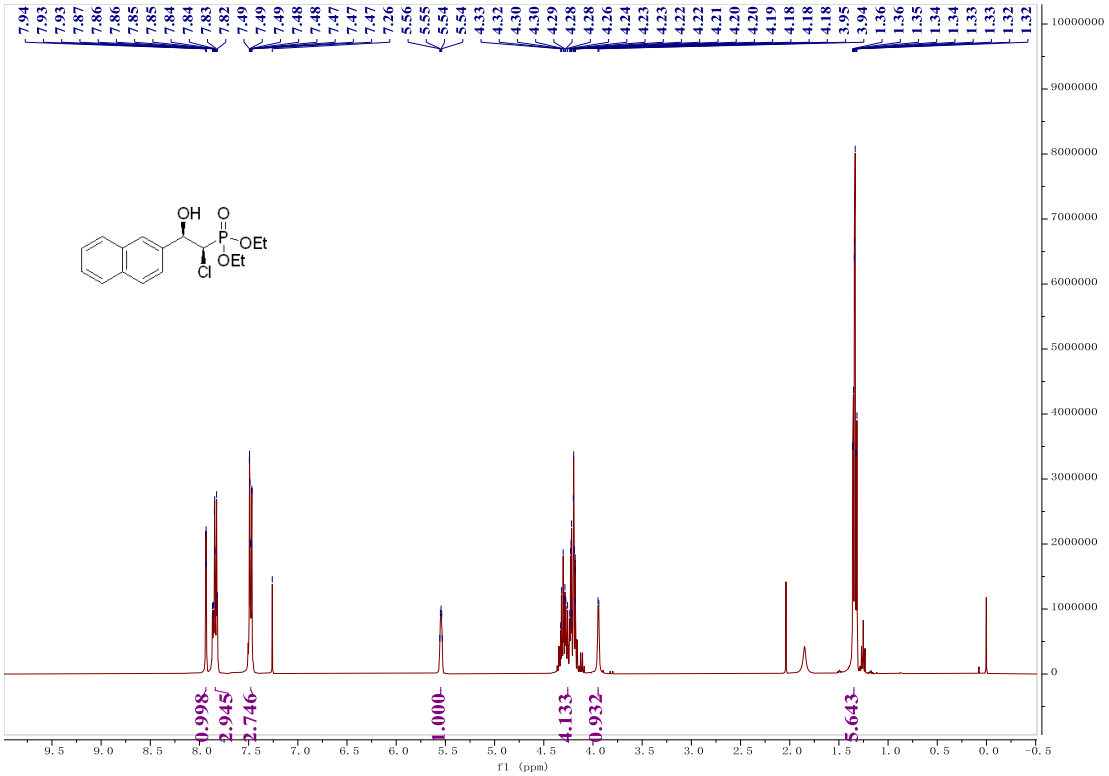
^{13}C NMR of **2t**, (101 MHz, Chloroform-*d*)



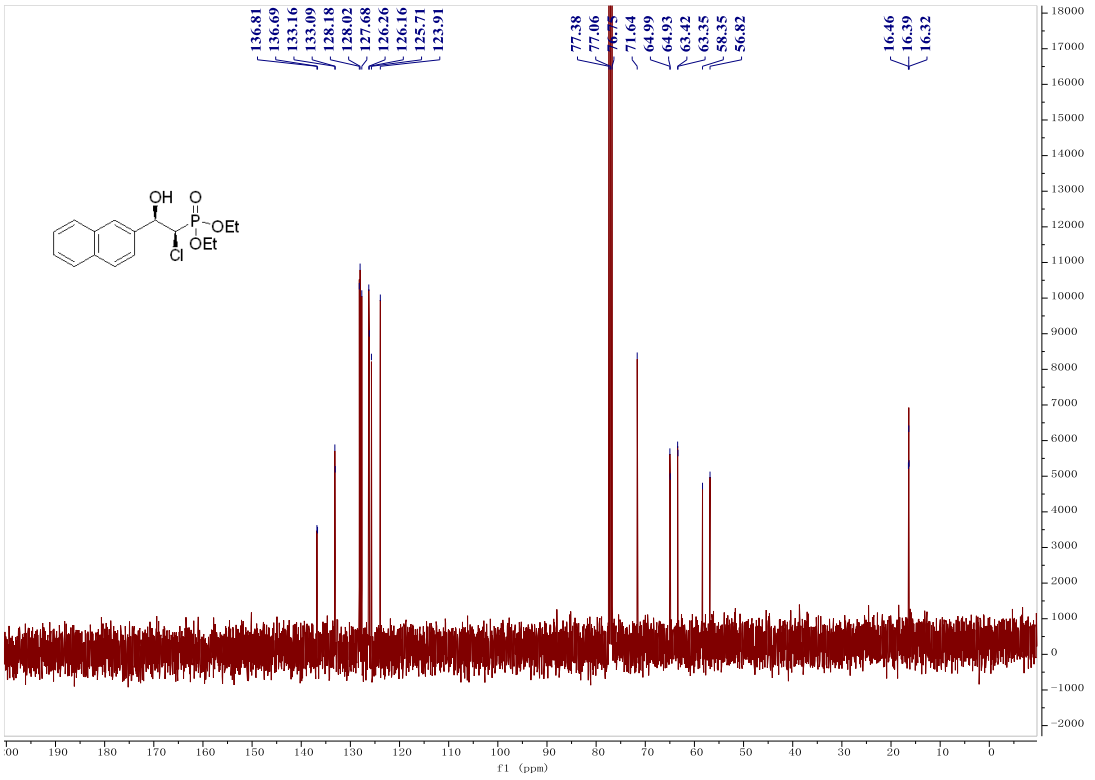
^{31}P NMR of **2t**, (162 MHz, Chloroform-*d*)



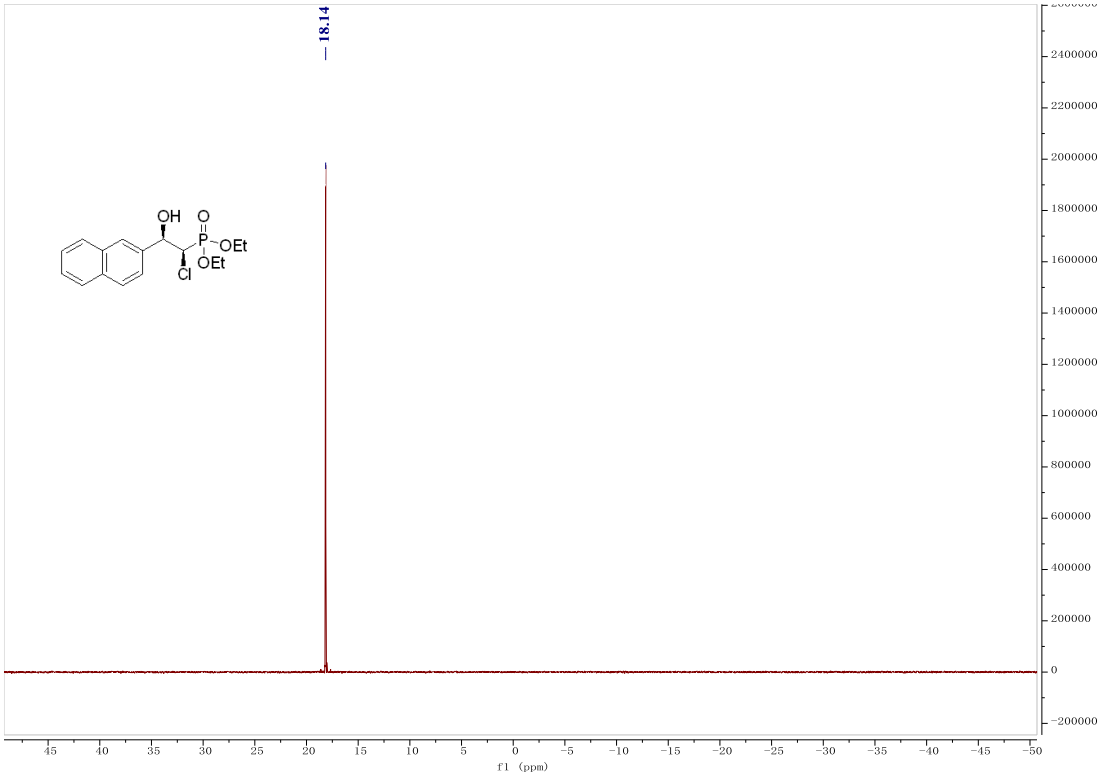
^{1}H NMR of **2u**, (400 MHz, Chloroform-*d*)



^{13}C NMR of **2u**, (101 MHz, Chloroform-*d*)

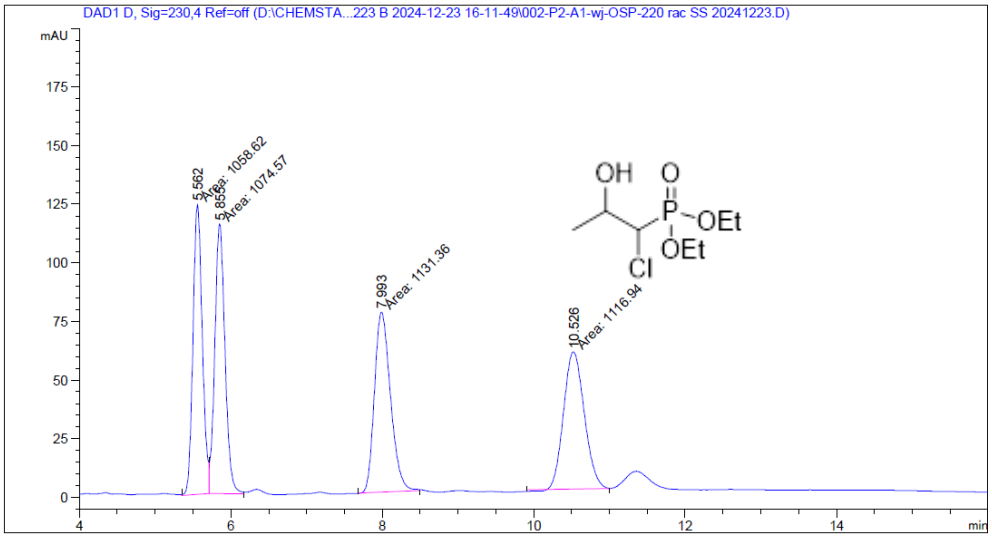


^{31}P NMR of **2u**, (162 MHz, Chloroform-*d*)



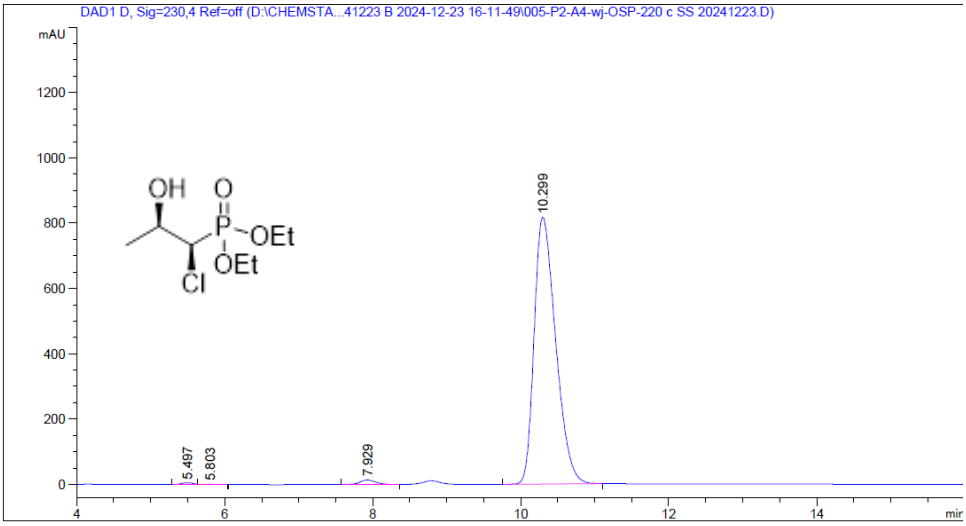
VII. HPLC Spectra

HPLC of **2a**



Signal 1: DAD1 D, Sig=230,4 Ref=off

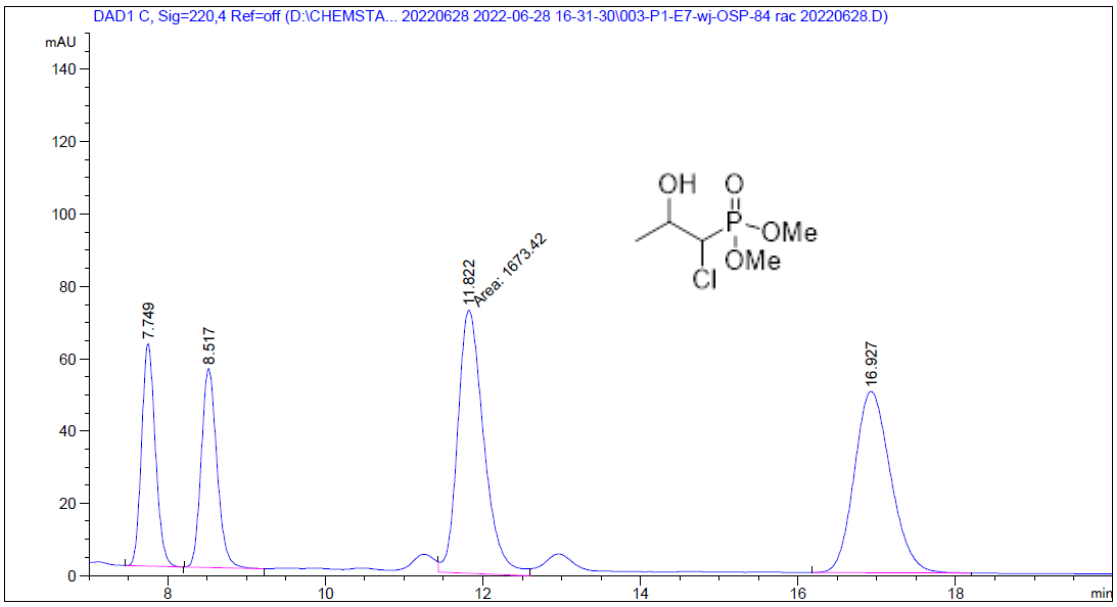
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.562	MM	0.1428	1058.62476	123.52968	24.1613
2	5.855	MM	0.1556	1074.57324	115.06432	24.5253
3	7.993	MM	0.2453	1131.35718	76.85720	25.8213
4	10.526	MM	0.3181	1116.94092	58.51867	25.4922
Totals :				4381.49609	373.96987	



Signal 1: DAD1 D, Sig=230,4 Ref=off

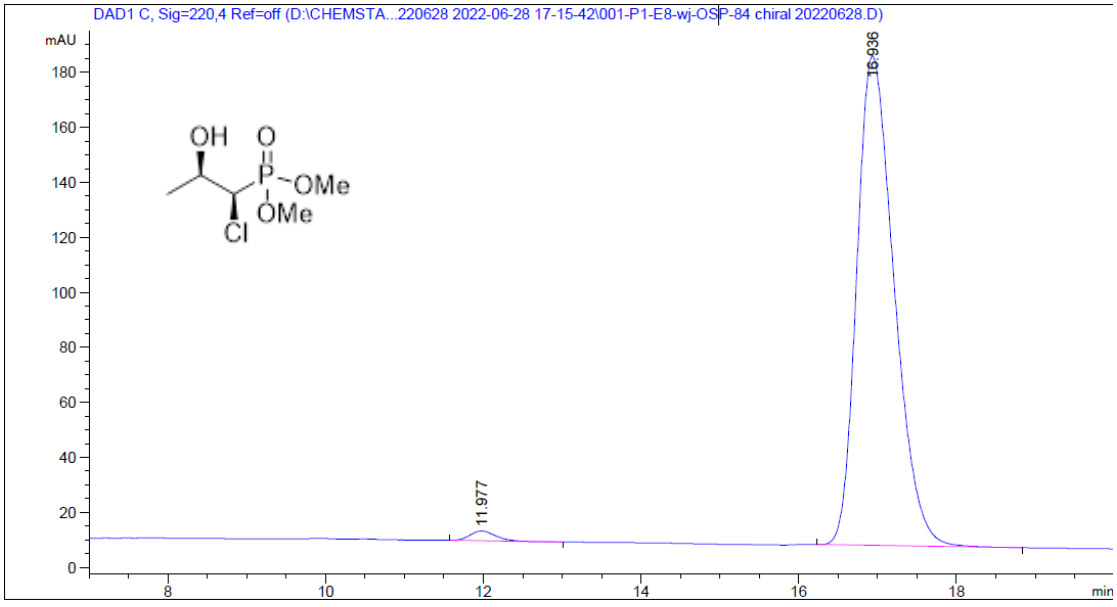
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.497	BV R	0.1672	60.20416	5.52373	0.3596
2	5.803	VB E	0.1379	9.47204	1.02458	0.0566
3	7.929	BB	0.2280	200.28401	13.58170	1.1963
4	10.299	BB	0.3113	1.64727e4	816.61066	98.3876
Totals :				1.67426e4	836.74067	

HPLC of **2b**



Signal 1: DAD1 C, Sig=220,4 Ref=off

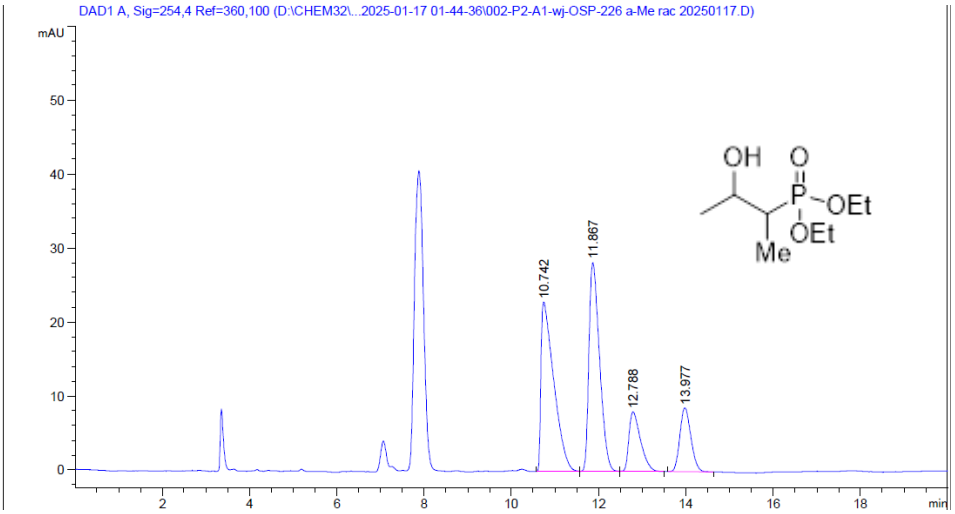
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.749	BB	0.1900	748.18353	61.47584	15.6170
2	8.517	BB	0.2137	763.02905	55.06368	15.9269
3	11.822	MM	0.3834	1673.41699	72.74020	34.9297
4	16.927	BB	0.4957	1606.18823	50.14058	33.5264



Signal 1: DAD1 C, Sig=220,4 Ref=off

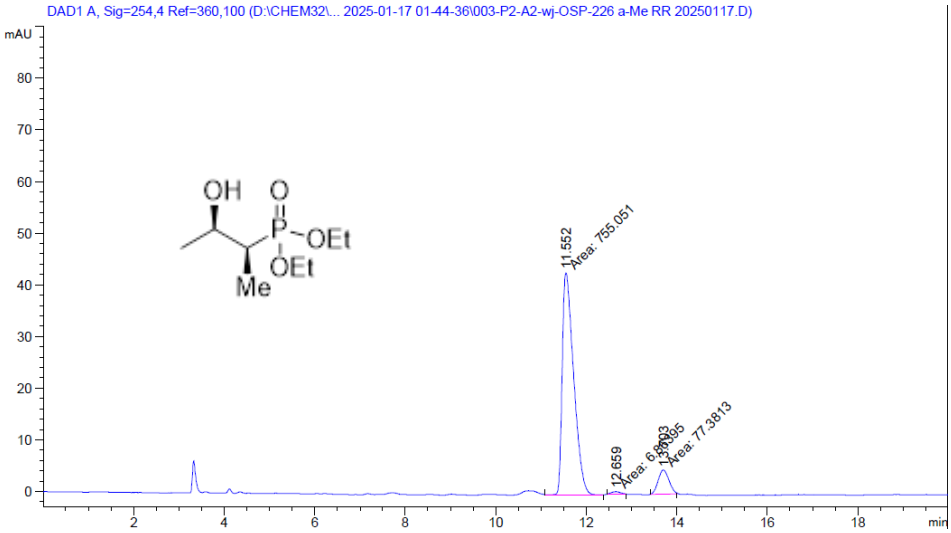
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.977	BB	0.3576	82.44858	3.56808	1.3922
2	16.936	BB	0.5090	5839.78955	177.87279	98.6078

HPLC of **2c**



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

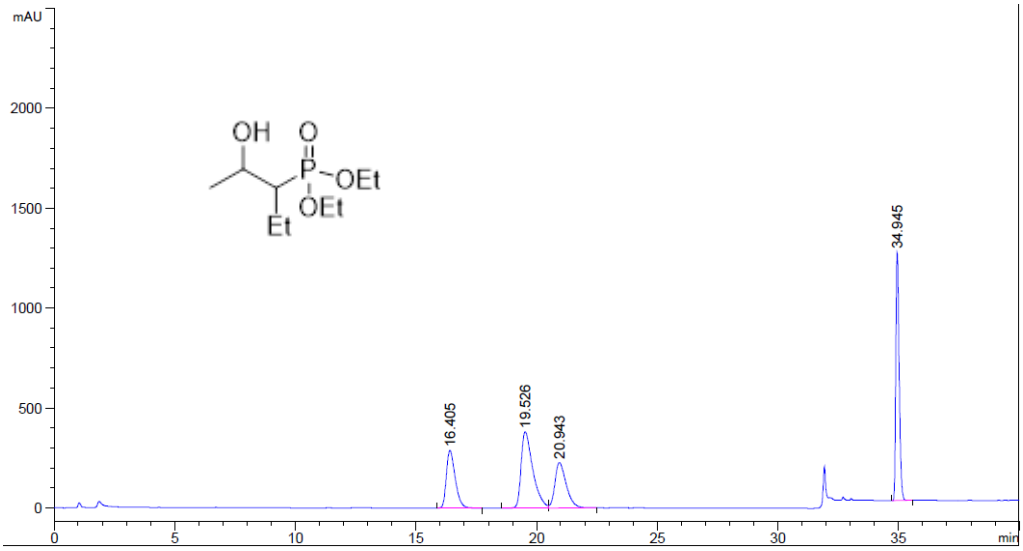
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.742	BB	0.2974	466.22635	22.90911	37.5036
2	11.867	BB	0.2606	475.59625	28.22087	38.2573
3	12.788	BB	0.2764	149.49303	8.05979	12.0253
4	13.977	BB	0.2733	151.83609	8.62937	12.2138
Totals :				1243.15172	67.81914	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

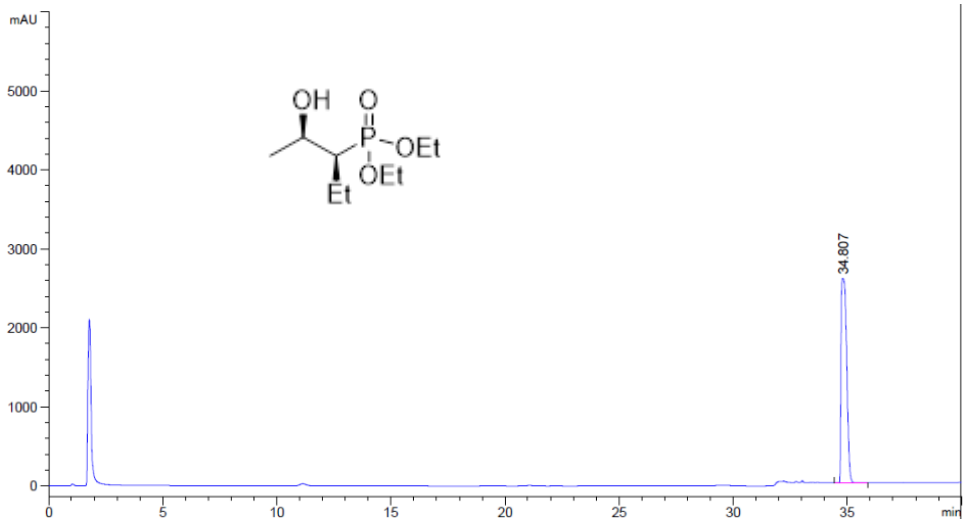
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.552	MM	0.2924	755.05121	43.03728	89.9624
2	12.659	MM	0.2343	6.86395	4.88330e-1	0.8178
3	13.703	MM	0.2766	77.38131	4.66254	9.2198
Totals :				839.29647	48.18815	

HPLC of **2d**



Signal 1: DAD1 D, Sig=230,4 Ref=360,100

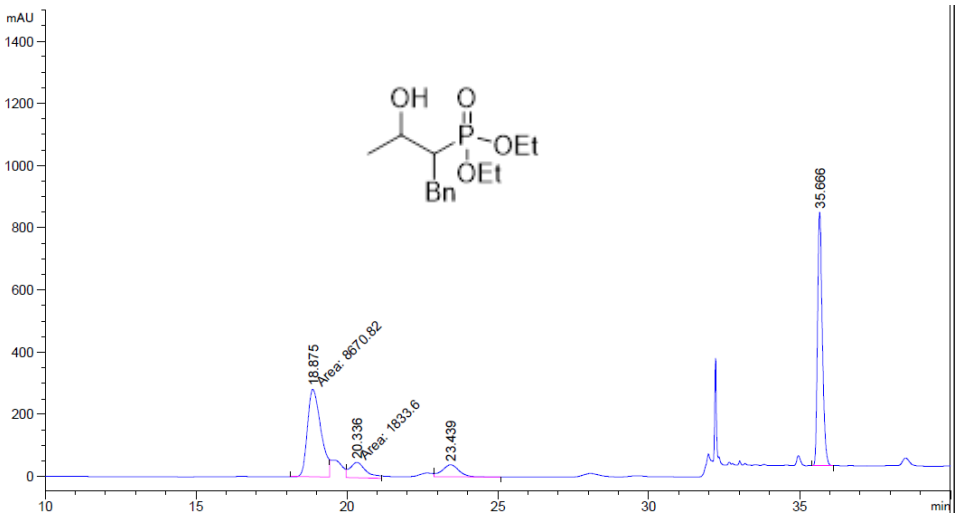
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.405	BB	0.3905	7337.29395	288.68762	18.5721
2	19.526	BV	0.5028	1.27798e4	381.49533	32.3481
3	20.943	VB	0.4940	7400.14014	227.21651	18.7311
4	34.945	BB	0.1508	1.19899e4	1239.63660	30.3487
Totals :				3.95072e4	2137.03606	



Signal 1: DAD1 D, Sig=230,4 Ref=360,100

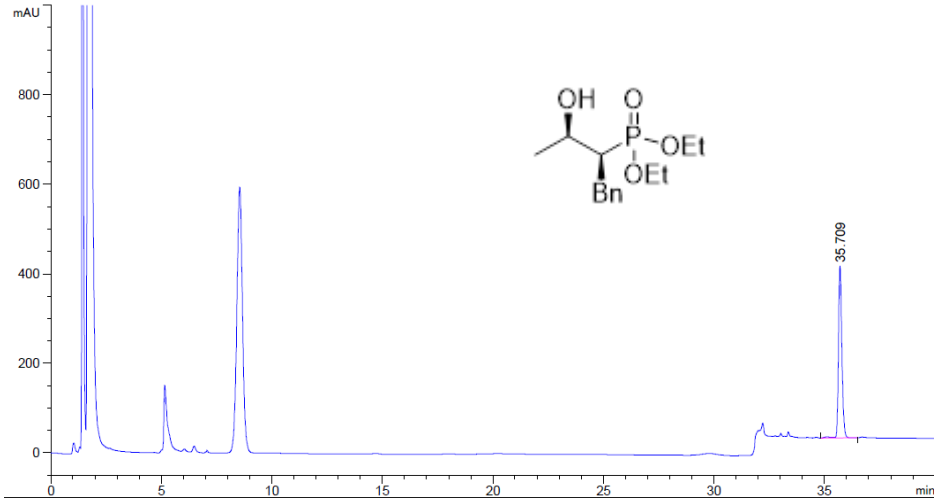
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	34.807	BV R	0.2775	4.42692e4	2587.62524	100.0000
Totals :				4.42692e4	2587.62524	

HPLC of **2e**



Signal 1: DAD1 D, Sig=230,4 Ref=360,100

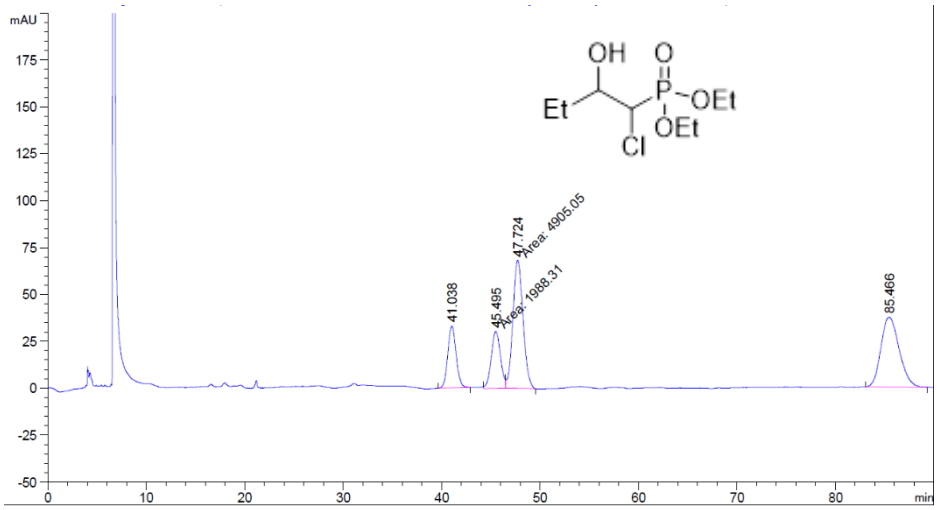
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.875	MF	0.5142	8670.82422	281.01865	41.1592
2	20.336	FM	0.6274	1833.60278	48.71229	8.7039
3	23.439	VB	0.5976	1567.27771	39.27284	7.4397
4	35.666	BB	0.1687	8994.82227	815.23395	42.6972
Totals :				2.10665e4	1184.23772	



Signal 1: DAD1 D, Sig=230,4 Ref=360,100

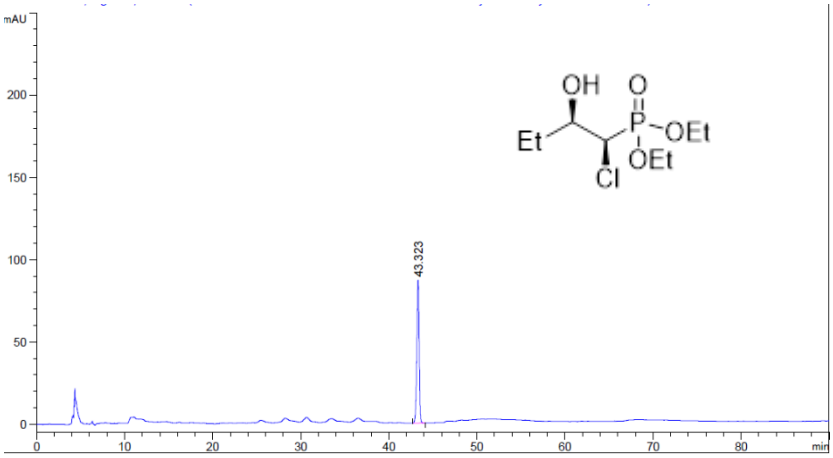
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.709	VB R	0.1646	4159.94141	384.13950	100.0000
Totals :				4159.94141	384.13950	

HPLC of **2f**



Signal 1: DAD1 C, Sig=220,4 Ref=off

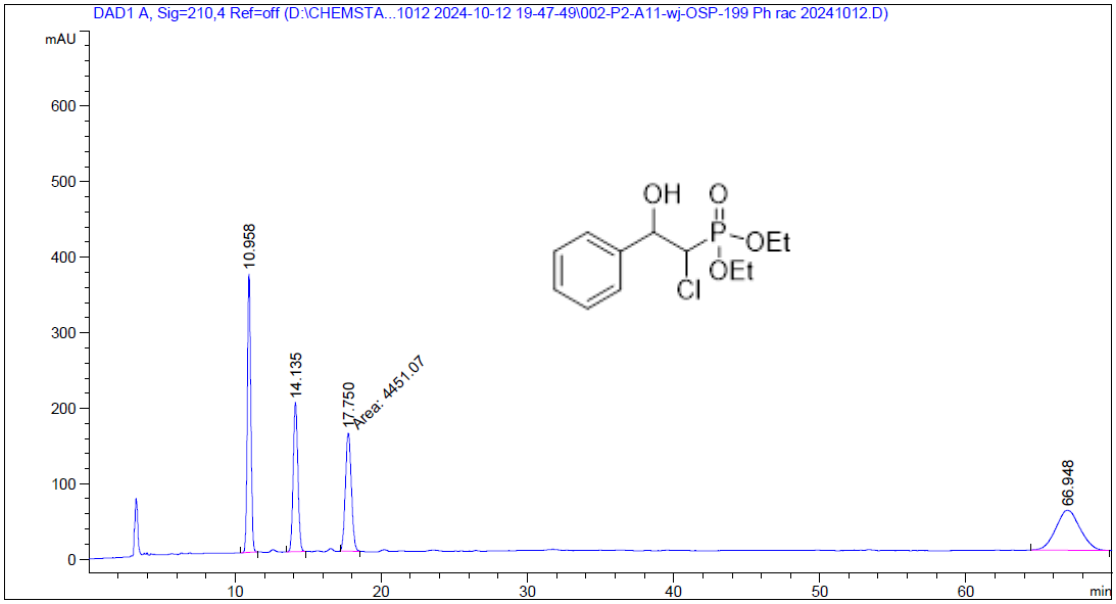
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	41.038	BB	0.8757	1940.01074	32.91411	14.2469
2	45.495	MM	1.0897	1988.30530	30.41005	14.6016
3	47.724	MM	1.1983	4905.05127	68.22302	36.0215
4	85.466	BB	1.5794	4783.66113	37.12123	35.1300
Totals :				1.36170e4	168.66841	



Signal 1: DAD1 C, Sig=220,4 Ref=off

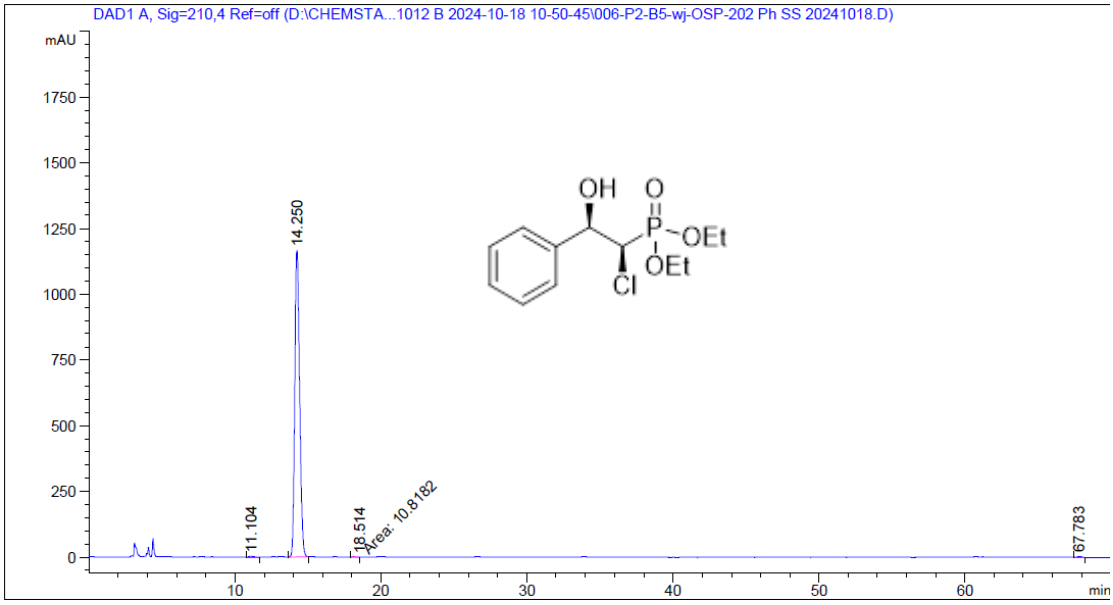
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	43.323	BB	0.2896	1620.49243	86.89072	100.0000
Totals :				1620.49243	86.89072	

HPLC of **2h**



Signal 1: DAD1 A, Sig=210,4 Ref=off

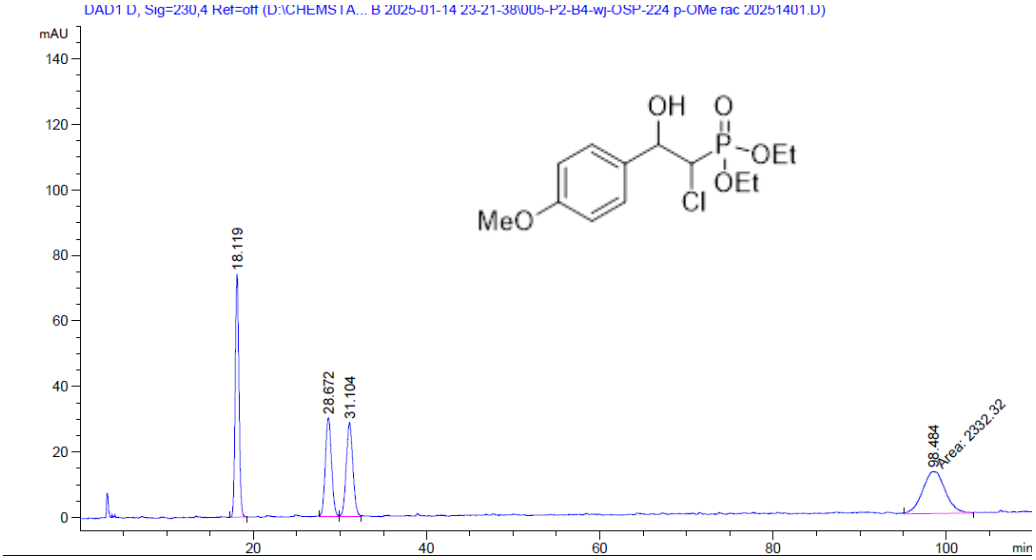
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.958	BB	0.2574	6121.45166	369.25934	29.0086
2	14.135	BB	0.3460	4410.79492	197.90979	20.9020
3	17.750	MM	0.4738	4451.07471	156.55952	21.0929
4	66.948	BB	1.3709	6118.89844	52.76560	28.9965



Signal 1: DAD1 A, Sig=210,4 Ref=off

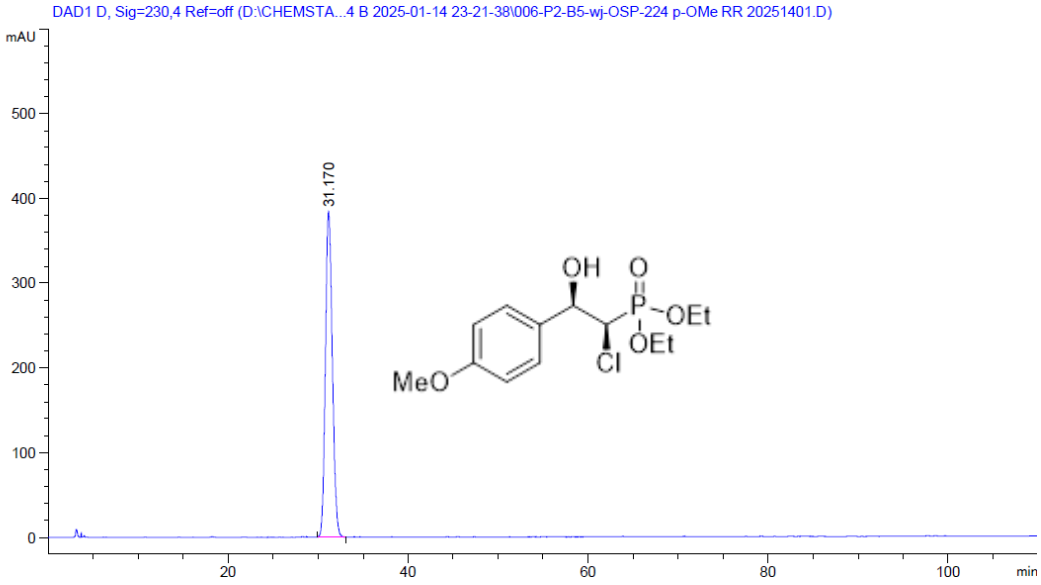
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.104	BB	0.2887	66.29499	3.32437	0.2460
2	14.250	BB	0.3601	2.68376e4	1167.83130	99.5706
3	18.514	MM	0.2953	10.81824	6.10533e-1	0.0401
4	67.783	BB	0.2873	38.63264	1.75203	0.1433

HPLC of **2i**



Signal 1: DAD1 D, Sig=230,4 Ref=off

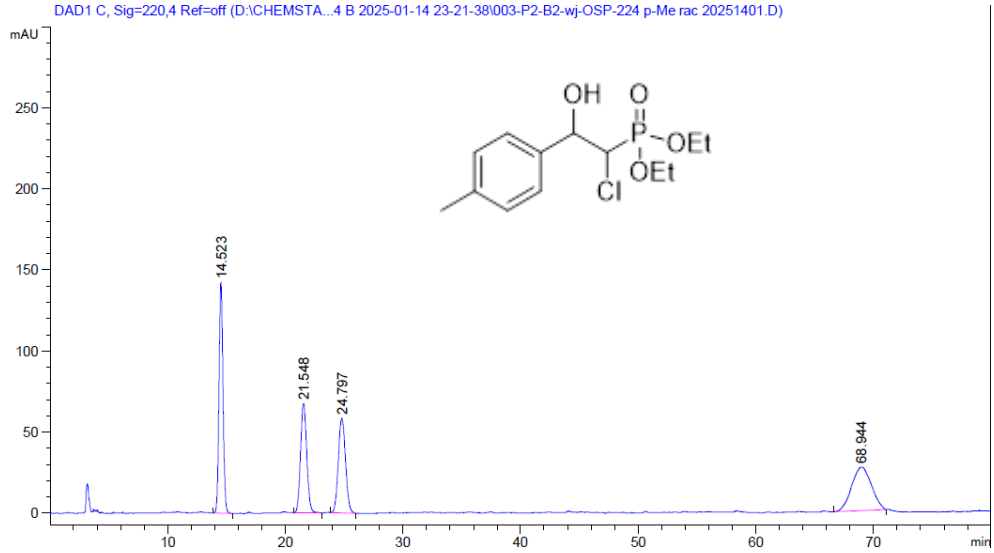
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.119	BB	0.4684	2228.76855	74.19719	29.5762
2	28.672	BB	0.7403	1476.28491	29.98834	19.5906
3	31.104	BB	0.7139	1498.30139	28.47536	19.8828
4	98.484	MM	3.0335	2332.31616	12.81435	30.9503
Totals :				7535.67102	145.47524	



Signal 1: DAD1 D, Sig=230,4 Ref=off

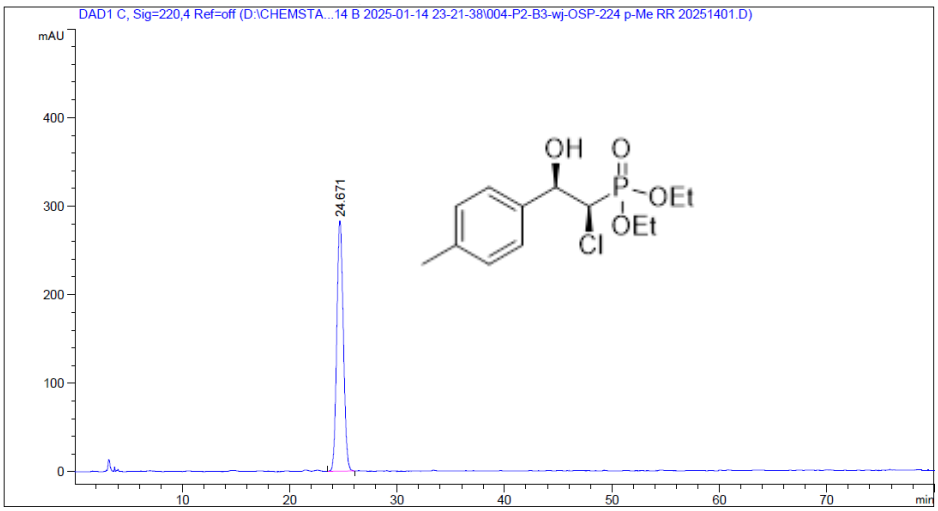
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	31.170	BB	0.8247	2.05980e4	384.66620	100.0000
Totals :				2.05980e4	384.66620	

HPLC of **2j**



Signal 1: DAD1 C, Sig=220,4 Ref=off

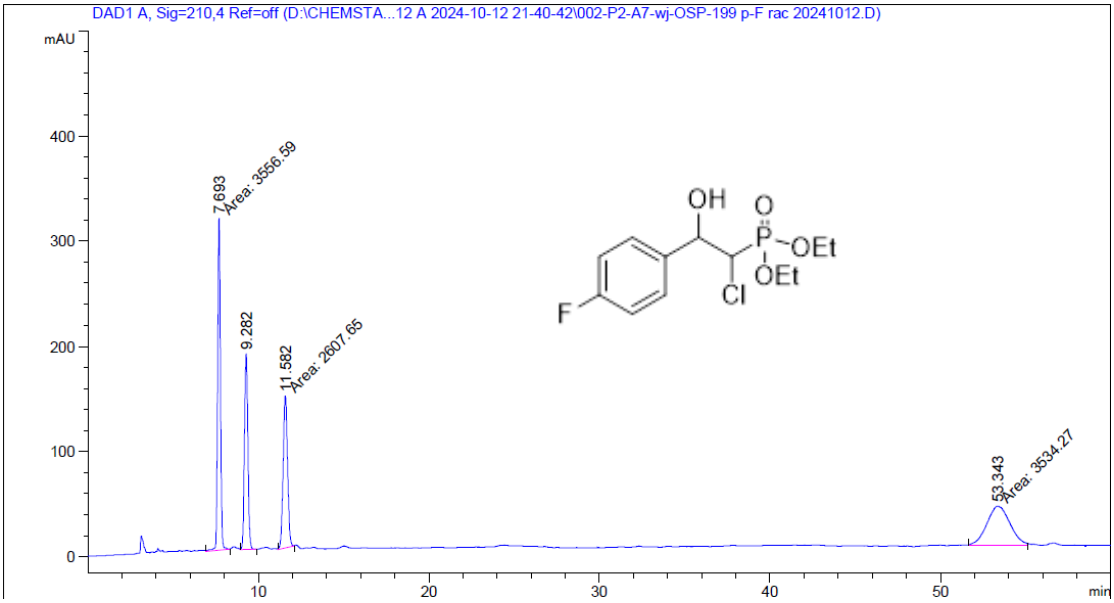
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.523	BB	0.3671	3353.52808	142.21466	29.2525
2	21.548	BB	0.5750	2478.36060	67.40285	21.6185
3	24.797	BB	0.6564	2484.12354	58.38495	21.6687
4	68.944	BB	1.3825	3148.07178	26.83766	27.4603
Totals :				1.14641e4	294.84012	



Signal 1: DAD1 C, Sig=220,4 Ref=off

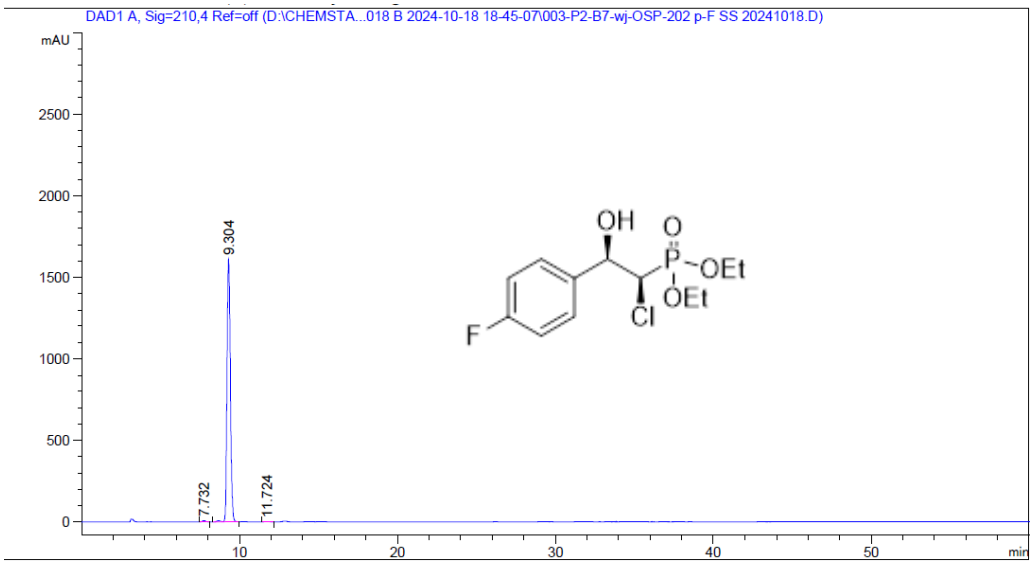
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.671	BB	0.6605	1.19441e4	282.91748	100.0000
Totals :				1.19441e4	282.91748	

HPLC of **2k**



Signal 1: DAD1 A, Sig=210,4 Ref=off

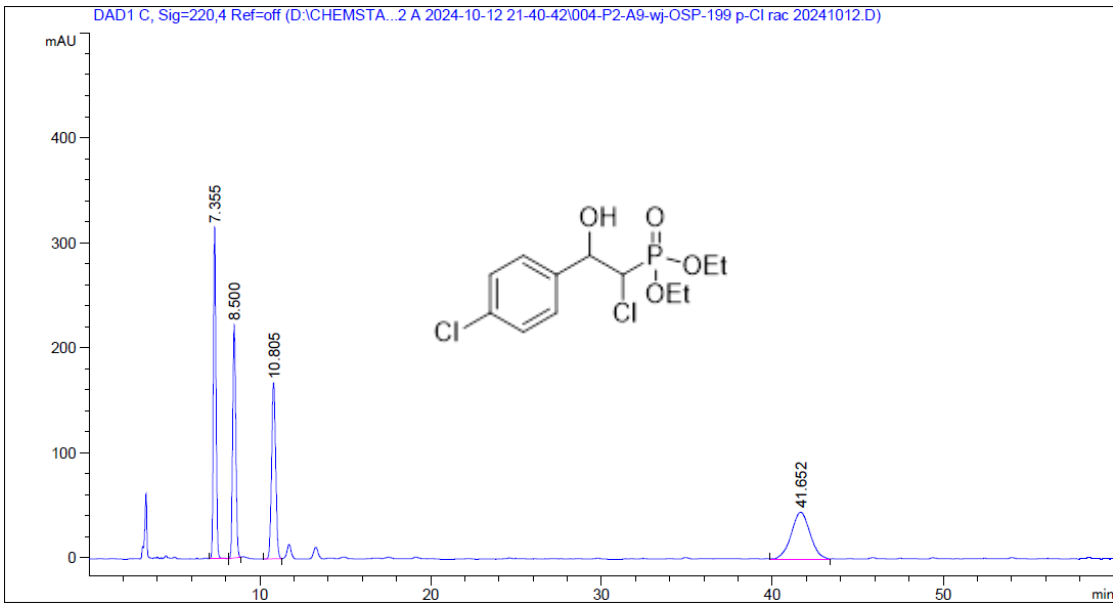
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.693	MM	0.1877	3556.59351	315.85071	28.9067
2	9.282	VB	0.2172	2605.16992	186.23610	21.1739
3	11.582	MM	0.3005	2607.65332	144.61369	21.1941
4	53.343	MM	1.5758	3534.27148	37.37983	28.7253
Totals :				1.23037e4	684.08033	



Signal 1: DAD1 A, Sig=210,4 Ref=off

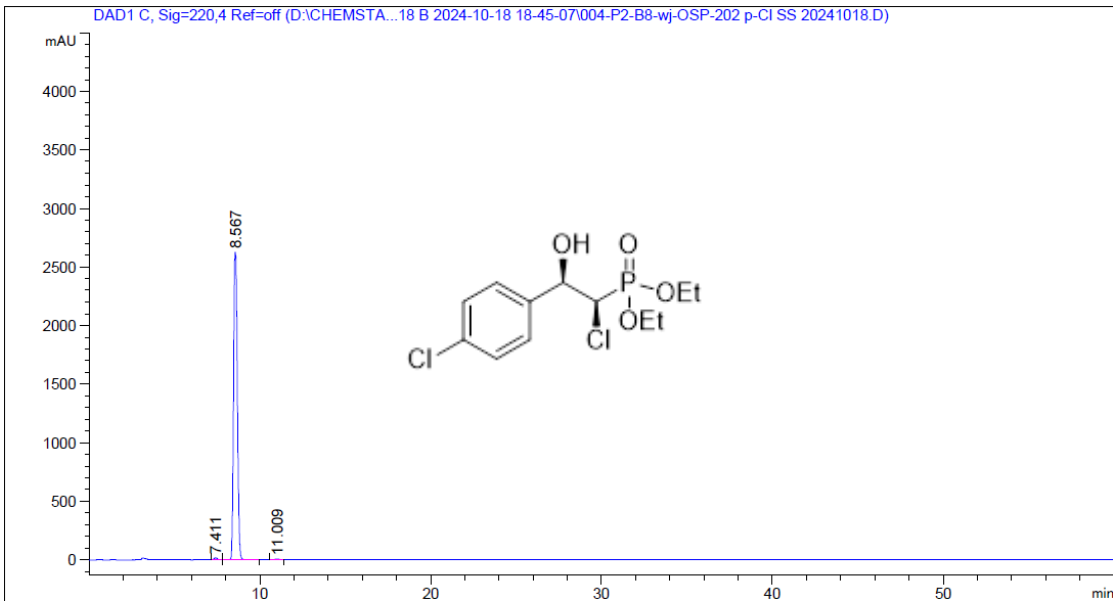
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.732	BB	0.2003	109.80225	8.19396	0.4569
2	9.304	VB R	0.2300	2.38639e4	1610.16992	99.2986
3	11.724	BB	0.2681	58.76505	3.20292	0.2445

HPLC of **2l**



Signal 1: DAD1 C, Sig=220,4 Ref=off

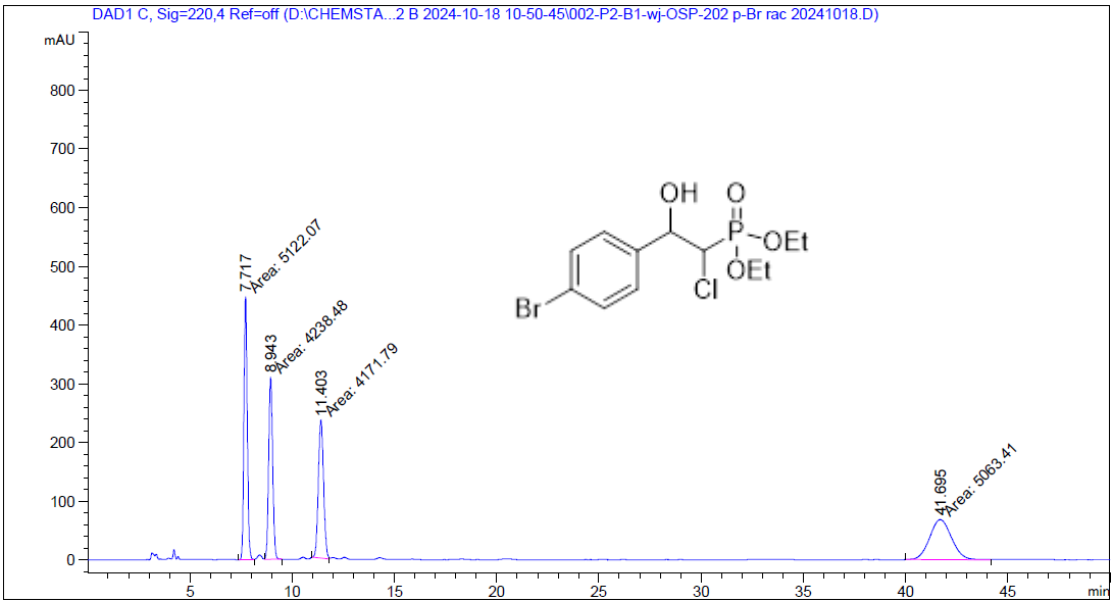
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.355	BB	0.1667	3373.72363	315.72449	27.2323
2	8.500	BB	0.1977	2813.71265	222.28502	22.7120
3	10.805	BB	0.2599	2815.56201	167.71826	22.7269
4	41.652	BB	1.1043	3385.67212	44.56732	27.3288



Signal 1: DAD1 C, Sig=220,4 Ref=off

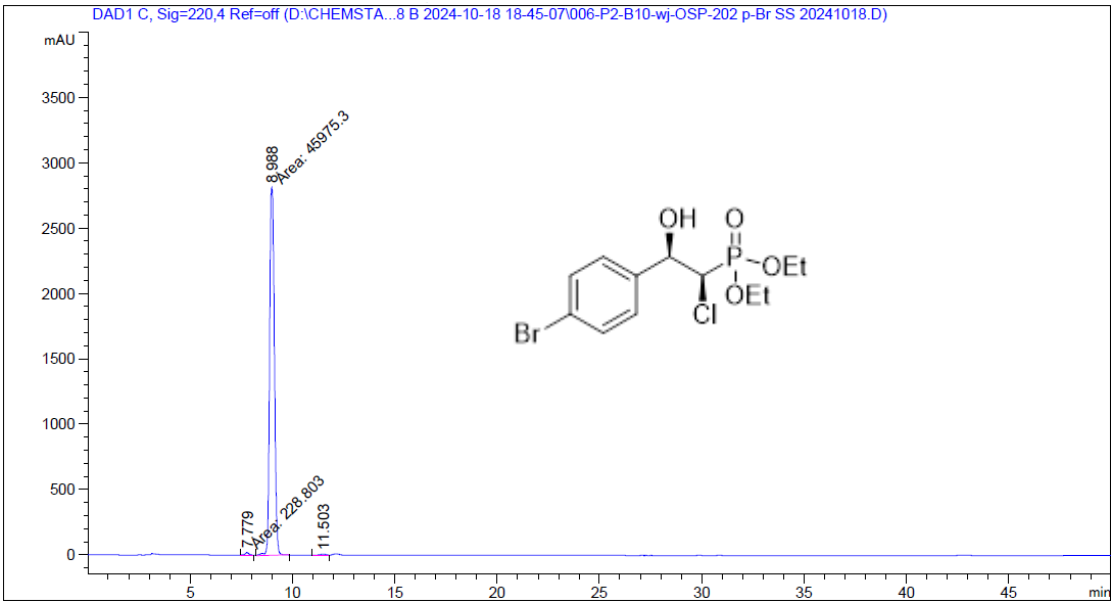
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.411	BB	0.1776	185.81328	16.22184	0.4550
2	8.567	VB R	0.2440	4.05396e4	2625.99731	99.2671
3	11.009	BB	0.2725	113.50641	6.47551	0.2779

HPLC of **2m**



Signal 1: DAD1 C, Sig=220,4 Ref=off

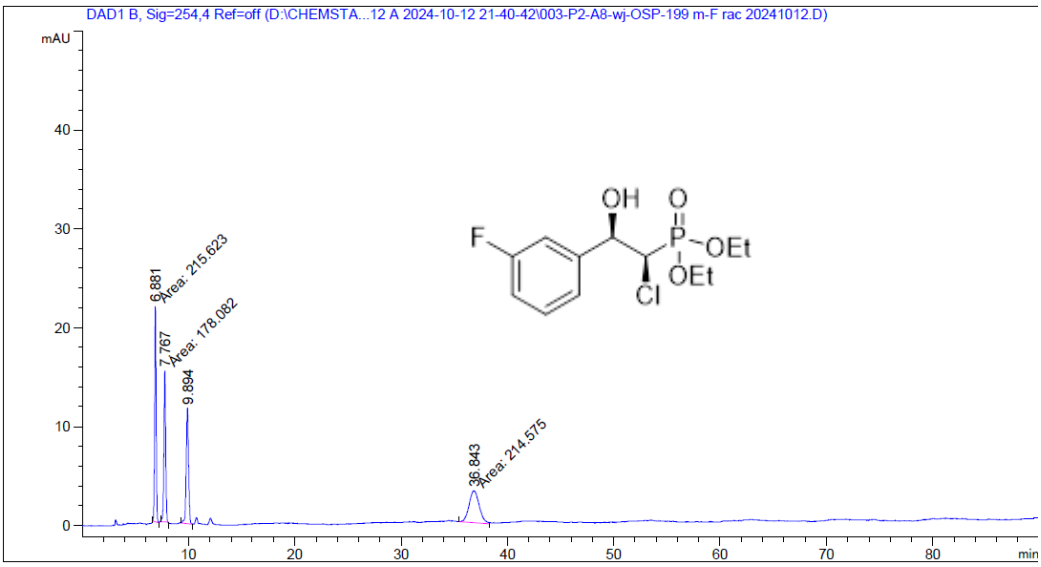
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.717	MM	0.1903	5122.07324	448.50299	27.5443
2	8.943	MM	0.2273	4238.48145	310.82501	22.7927
3	11.403	MM	0.2961	4171.78857	234.84311	22.4341
4	41.695	MM	1.2378	5063.40625	68.17602	27.2288



Signal 1: DAD1 C, Sig=220,4 Ref=off

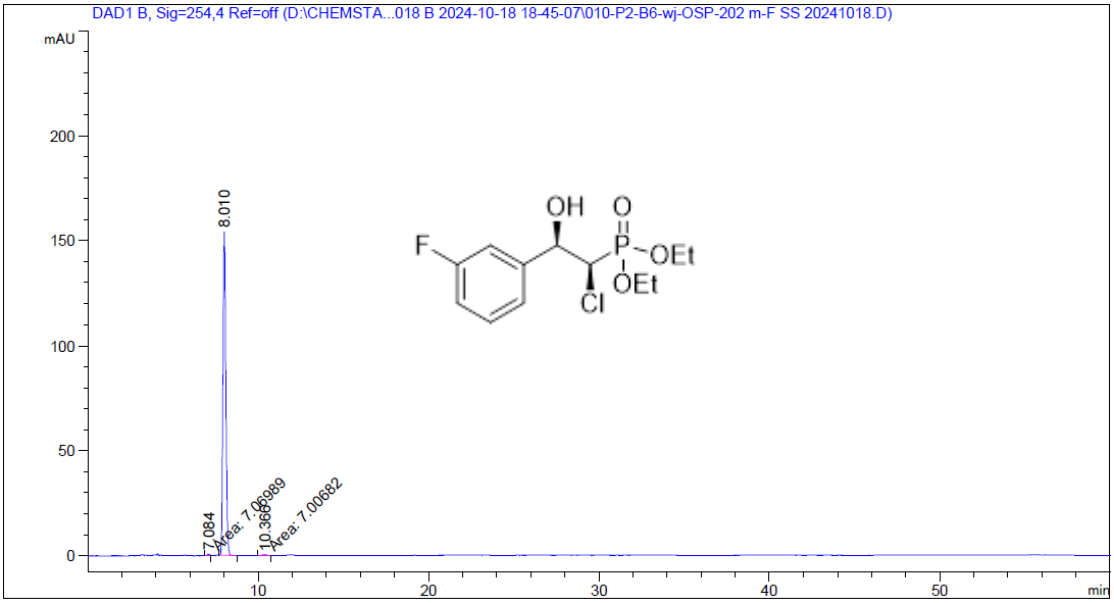
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.779	MM	0.2023	228.80267	18.84673	0.4938
2	8.988	MM	0.2722	4.59753e4	2814.85693	99.2218
3	11.503	BV	0.2994	131.78651	6.88247	0.2844

HPLC of **2n**



Signal 1: DAD1 B, Sig=254,4 Ref=off

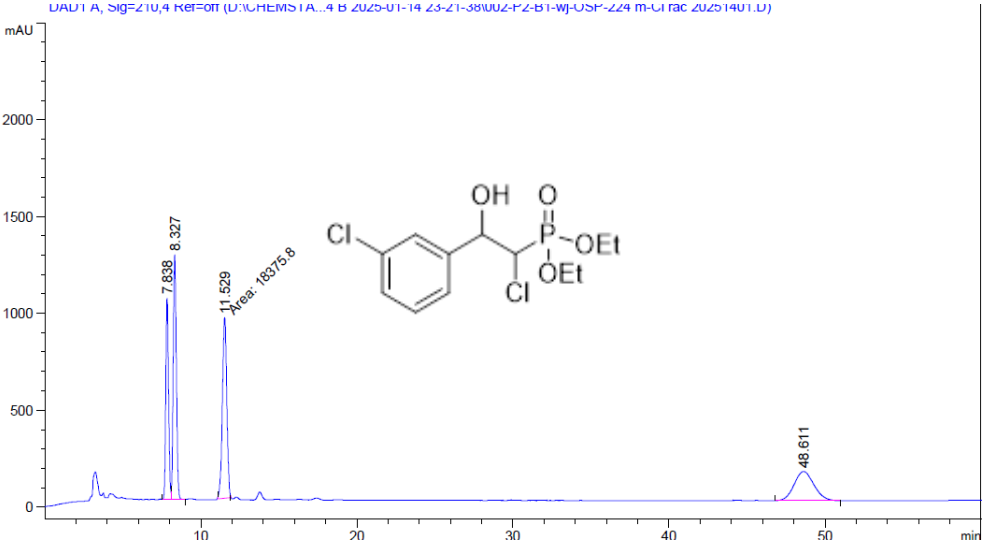
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.881	MM	0.1649	215.62331	21.79308	27.4120
2	7.767	MM	0.1935	178.08177	15.33899	22.6394
3	9.894	BB	0.2346	178.32146	11.64602	22.6699
4	36.843	MM	1.1061	214.57513	3.23332	27.2788



Signal 1: DAD1 B, Sig=254,4 Ref=off

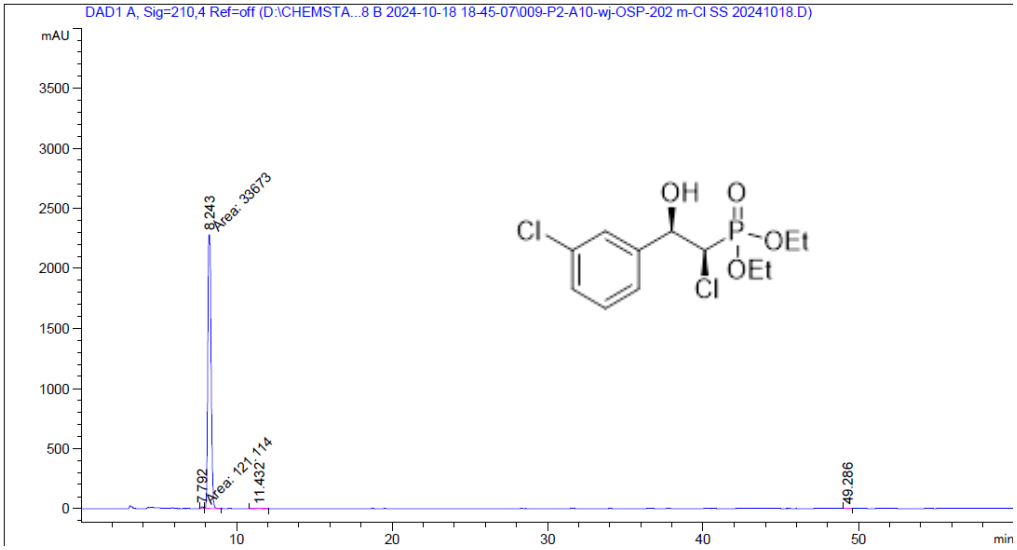
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.084	MM	0.1626	7.06989	7.24886e-1	0.3832
2	8.010	BB	0.1847	1831.11011	153.97067	99.2371
3	10.366	MM	0.2698	7.00682	4.32886e-1	0.3797

HPLC of **2o**



ignal 1: DAD1 A, Sig=210,4 Ref=off

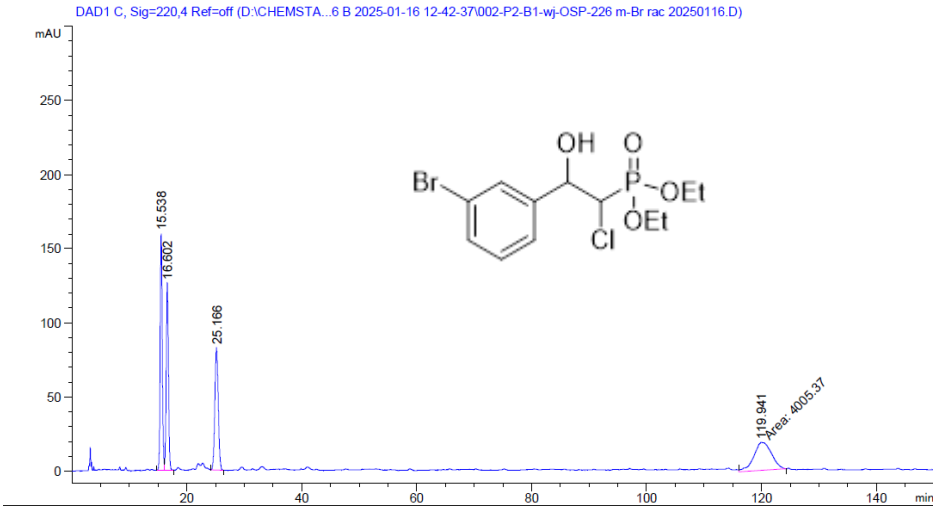
eak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.838	BV	0.1947	1.28837e4	1038.75354	20.7520
2	8.327	VB	0.2182	1.75836e4	1264.95471	28.3223
3	11.529	MM	0.3280	1.83758e4	933.74603	29.5981
4	48.611	BB	1.0473	1.32411e4	149.78676	21.3276
otals :				6.20842e4	3387.24104	



Signal 1: DAD1 A, Sig=210,4 Ref=off

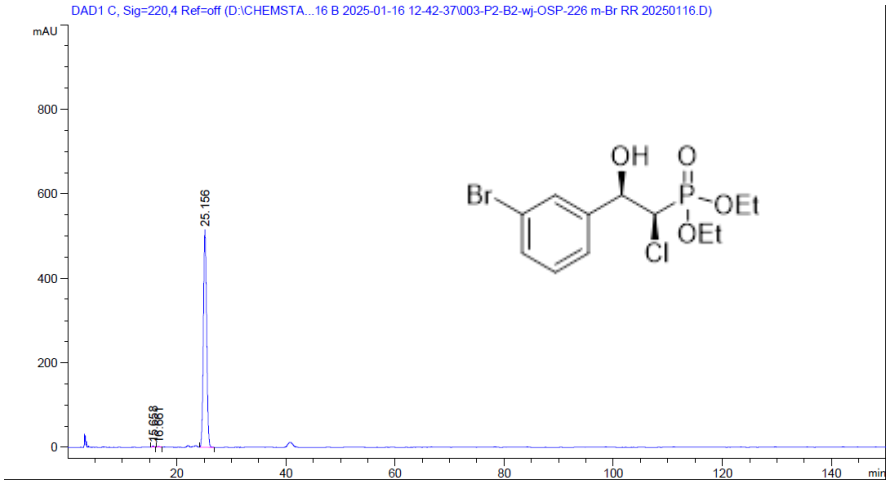
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.792	MM	0.1652	121.11361	12.22221	0.3568
2	8.243	MM	0.2459	3.36730e4	2282.16748	99.2013
3	11.432	BB	0.3757	123.61536	4.53943	0.3642
4	49.286	BB	0.2481	26.38588	1.61923	0.0777

HPLC of **2p**



Signal 1: DAD1 C, Sig=220,4 Ref=off

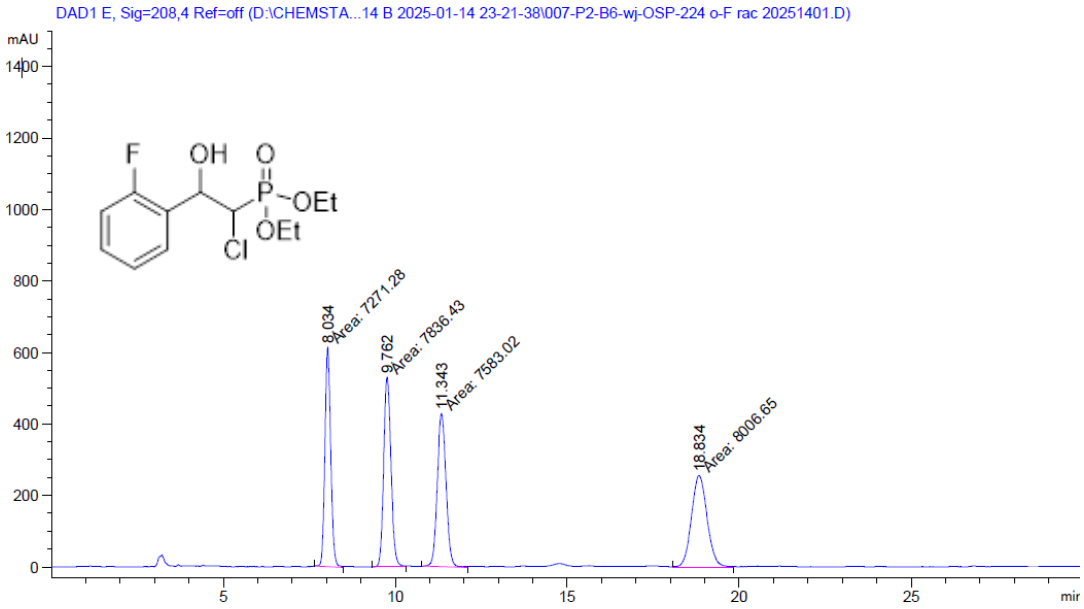
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.538	BV	0.3895	4015.62866	159.60670	27.0092
2	16.602	VB	0.4224	3431.22070	126.52686	23.0785
3	25.166	BB	0.6411	3415.42090	82.47607	22.9722
4	119.941	MM	3.5577	4005.36670	18.76361	26.9402
Totals :				1.48676e4	387.37325	



Signal 1: DAD1 C, Sig=220,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.658	BB	0.2834	60.63509	2.62562	0.2803
2	16.661	BB	0.3217	51.13678	1.93903	0.2364
3	25.156	BB	0.6557	2.15192e4	514.74353	99.4833
Totals :				2.16309e4	519.30818	

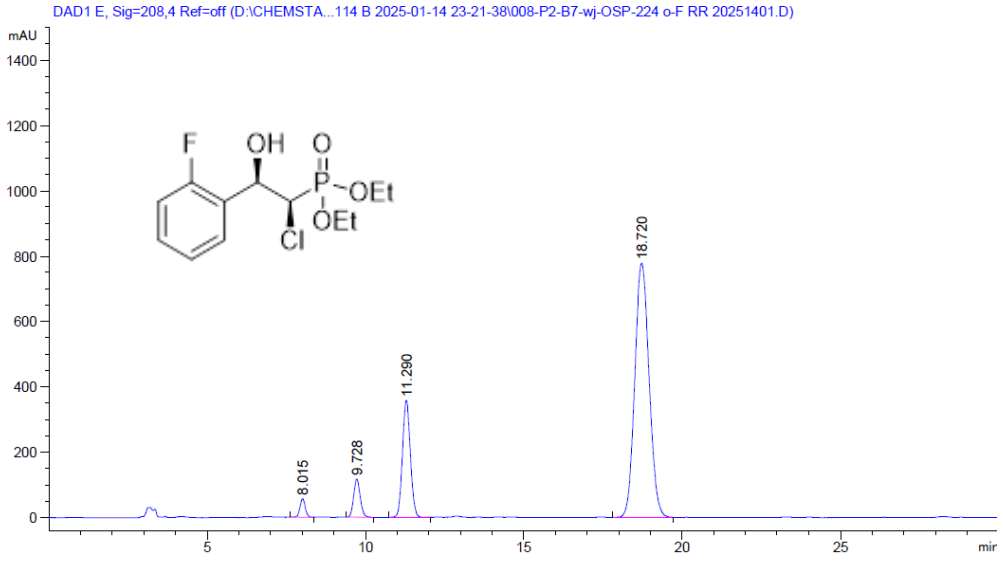
HPLC of **2q**



Signal 1: DAD1 E, Sig=208,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.034	MM	0.1974	7271.27881	613.95081	23.6870
2	9.762	MM	0.2461	7836.42676	530.76697	25.5280
3	11.343	MM	0.2950	7583.01611	428.47891	24.7025
4	18.834	MM	0.5228	8006.65283	255.24570	26.0825

Totals : 3.06974e4 1828.44238

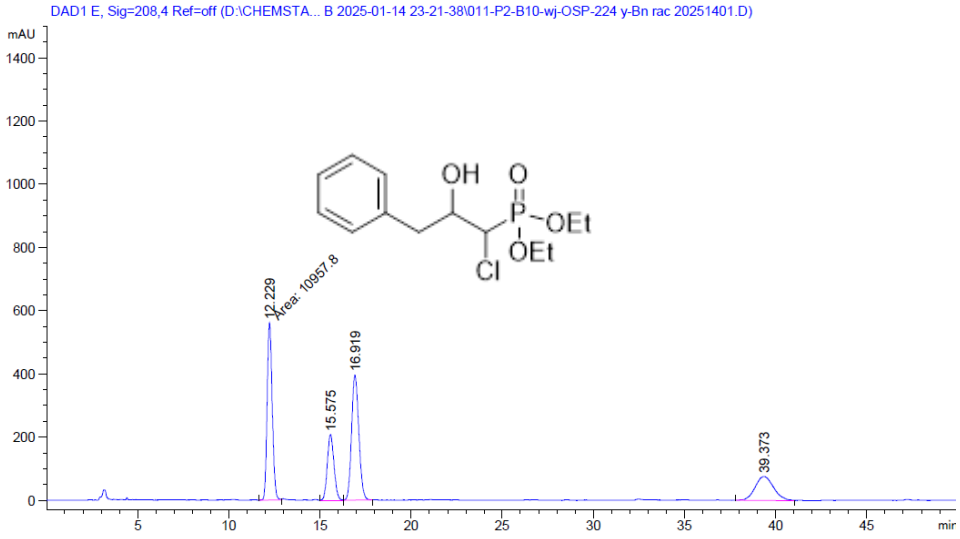


Signal 1: DAD1 E, Sig=208,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.015	BB	0.1832	658.49762	56.00063	1.9968
2	9.728	BB	0.2251	1702.91553	117.46472	5.1638
3	11.290	BV R	0.2704	6214.66309	358.24005	18.8450
4	18.720	BB	0.4921	2.44018e4	777.71918	73.9944

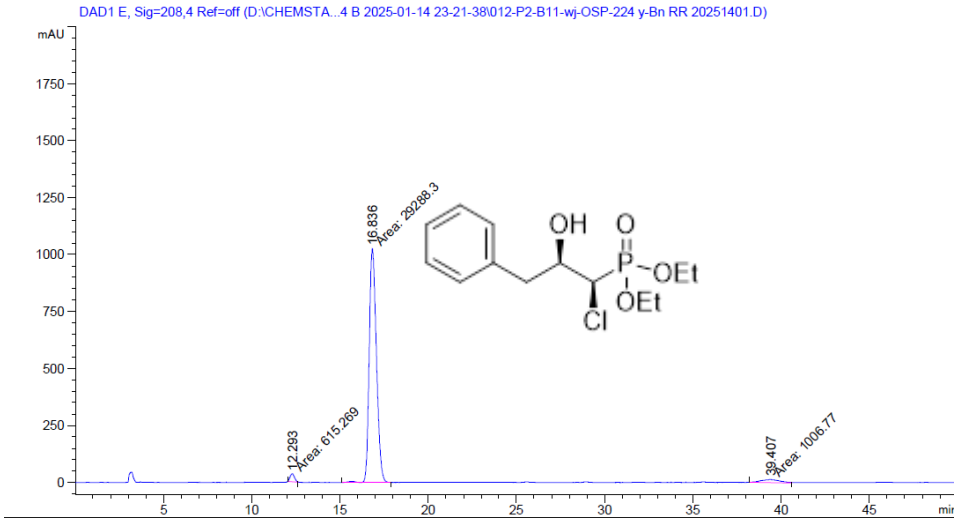
Totals : 3.29779e4 1309.42458

HPLC of **2r**



Signal 1: DAD1 E, Sig=208,4 Ref=off

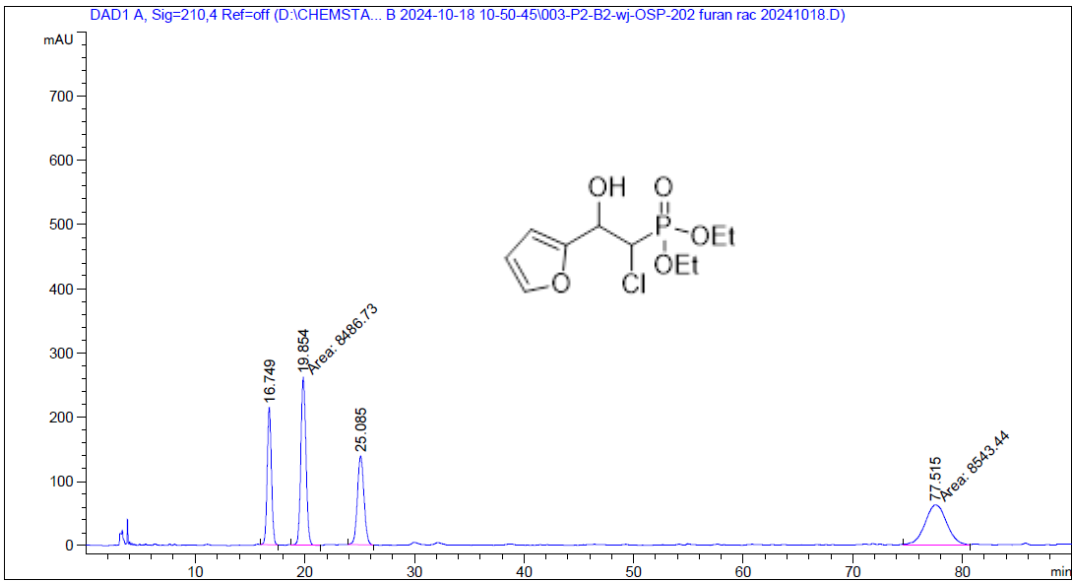
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.229	MM	0.3246	1.09578e4	562.60370	33.7204
2	15.575	BB	0.3958	5256.67969	207.32484	16.1764
3	16.919	BB	0.4265	1.09469e4	395.97037	33.6870
4	39.373	BB	0.8366	5334.60596	76.03169	16.4162
Totals :				3.24960e4	1241.93060	



Signal 1: DAD1 E, Sig=208,4 Ref=off

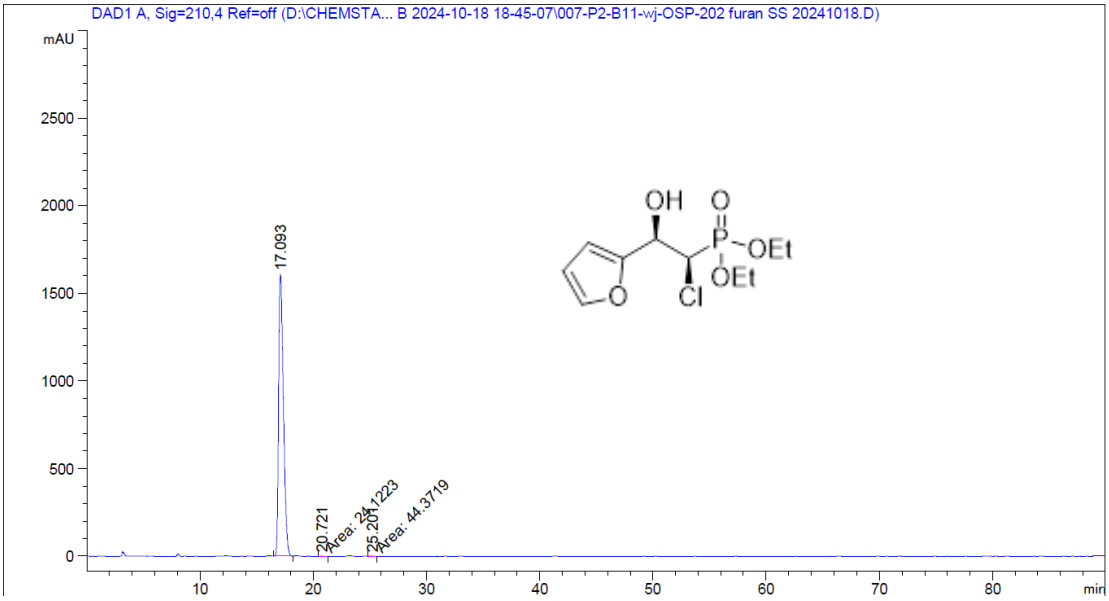
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.293	MM	0.2942	615.26904	34.85194	1.9905
2	16.836	MM	0.4759	2.92883e4	1025.72217	94.7524
3	39.407	MM	1.2564	1006.77142	13.35491	3.2571
Totals :				3.09103e4	1073.92903	

HPLC of **2s**



Signal 1: DAD1 C, Sig=220,4 Ref=off

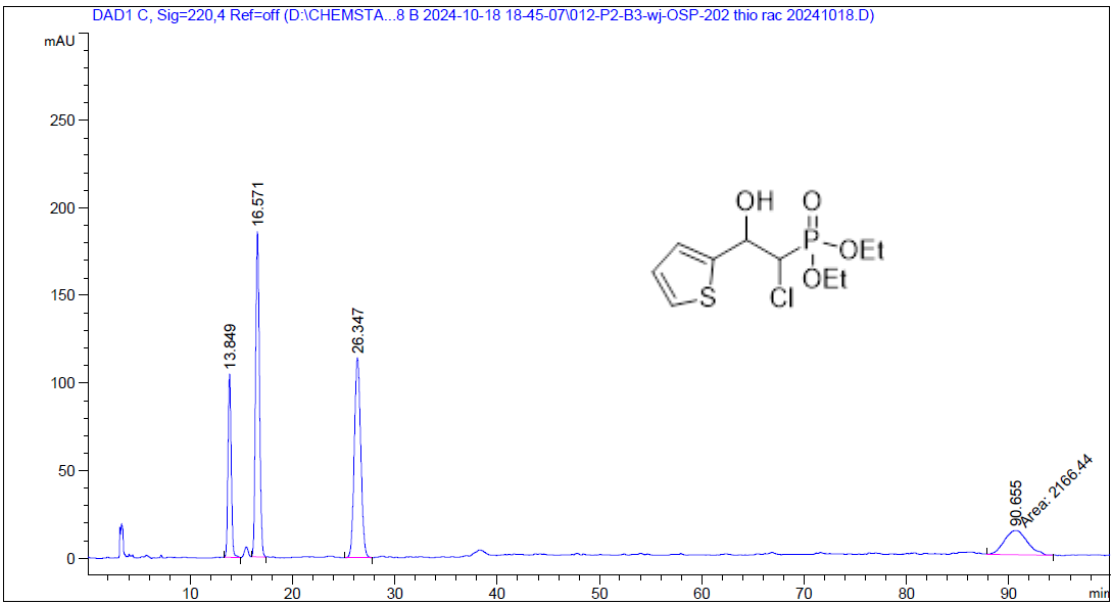
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.849	BB	0.3358	2279.43701	104.79841	15.8562
2	16.571	BB	0.4146	4947.95801	185.85635	34.4188
3	26.347	BB	0.6795	4981.89502	114.07495	34.6549
4	90.655	MM	2.5920	2166.43701	13.93054	15.0701
Totals :				1.43757e4	418.66025	



Signal 1: DAD1 A, Sig=210,4 Ref=off

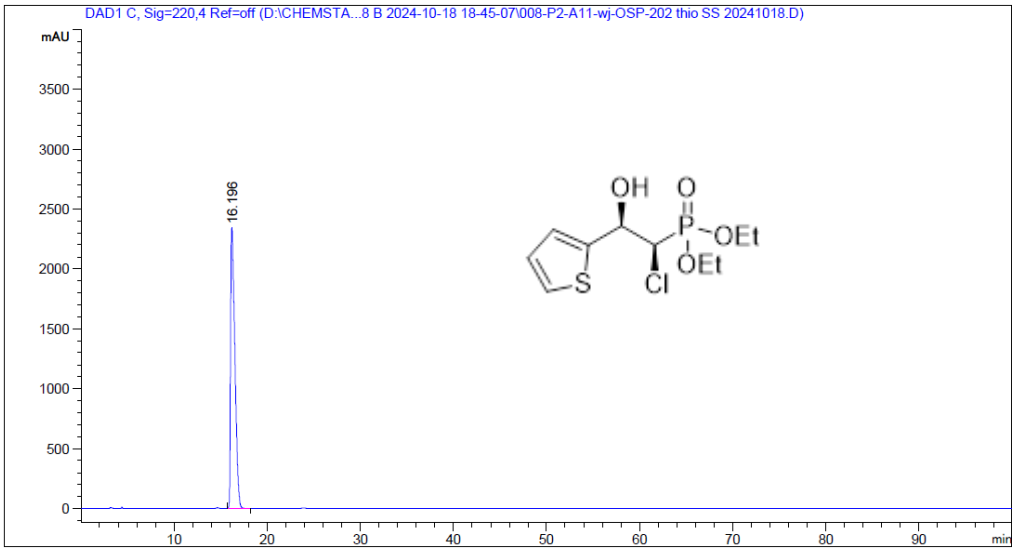
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.093	BB	0.4588	4.74440e4	1605.50659	99.8558
2	20.721	MM	0.3145	24.12230	1.27823	0.0508
3	25.201	MM	0.4545	44.37188	1.62699	0.0934

HPLC of **2t**



Signal 1: DAD1 C, Sig=220,4 Ref=off

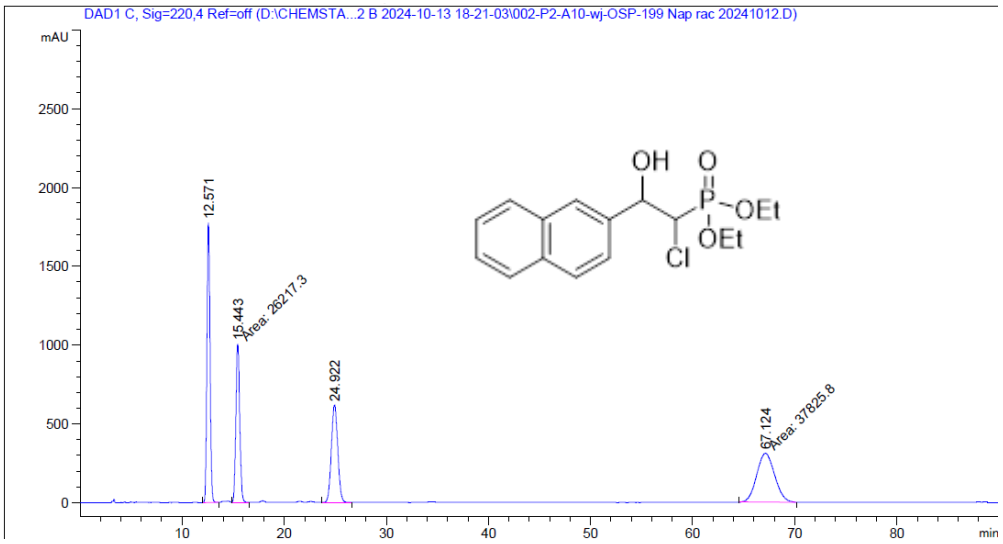
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.849	BB	0.3358	2279.43701	104.79841	15.8562
2	16.571	BB	0.4146	4947.95801	185.85635	34.4188
3	26.347	BB	0.6795	4981.89502	114.07495	34.6549
4	90.655	MM	2.5920	2166.43701	13.93054	15.0701
Totals :				1.43757e4	418.66025	



Signal 1: DAD1 C, Sig=220,4 Ref=off

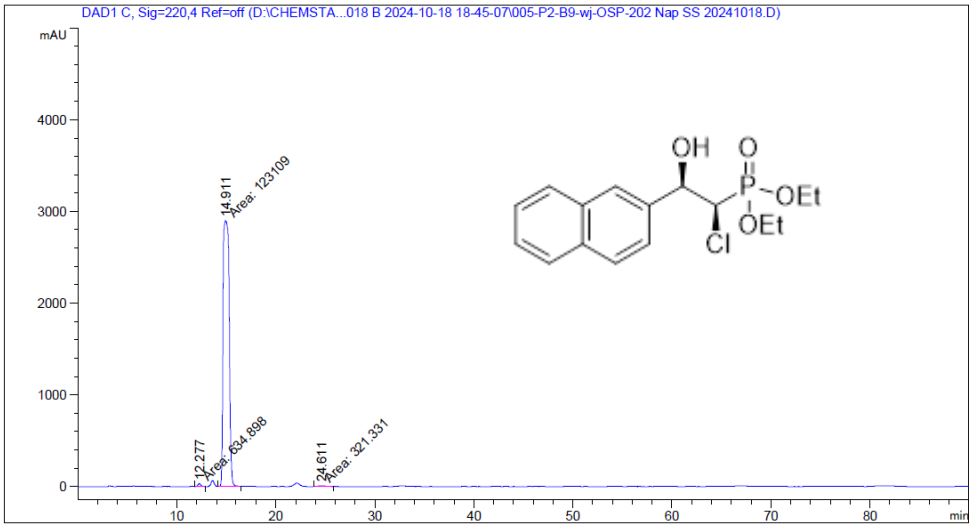
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.196	BB	0.4872	7.49999e4	2344.67505	100.0000

HPLC of **2u**



Signal 1: DAD1 C, Sig=220,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.571	BB	0.3284	3.71496e4	1773.29834	29.0127
2	15.443	MM	0.4350	2.62173e4	1004.42065	20.4749
3	24.922	BB	0.6807	2.68534e4	618.23450	20.9716
4	67.124	MM	2.0343	3.78258e4	309.90616	29.5408
Totals :				1.28046e5	3705.85965	



Signal 1: DAD1 C, Sig=220,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.277	MM	0.3331	634.89819	31.76896	0.5117
2	14.911	MM	0.7064	1.23109e5	2904.47266	99.2293
3	24.611	MM	0.6583	321.33051	8.13513	0.2590

VIII. Reference

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