Supplementary Information (SI) for ChemComm.

Supporting Information

Photocatalytic coupling of silyl enol ethers with oxime phosphonates for streamlined *β*-ketophosphonates synthesis

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1 General Information

^{1}H, ^{13}C ^{31}P and ^{19}F NMR spectra were recorded on a Bruker AVANCE III 400 MHz Superconducting Fourier or 500 MHz Superconducting Fourier, and were internally referenced to residual protio solvent signal (note: $CDCl_3$ referenced at δ 7.26 ppm for ^{1}H NMR and δ 77.16 ppm for ^{13}C NMR, respectively). Data for ^{1}H NMR are reported as follows: chemical shift (ppm), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constant (Hz). Data for ^{13}C NMR are reported in terms of chemical shift and no special nomenclature is used for equivalent carbons. High-resolution mass spectrometry data were recorded on an Thermo Scientific Q Exactive instrument using direct injection of samples in dichloromethane into the electrospray source (ESI) with positive or negative ionization.

All reactions were carried out under an inert atmosphere of nitrogen in oven dried or flame dried glassware with magnetic stirring, unless otherwise noted. Commercially available reagents including dry solvents were used without additional purification, unless otherwise indicated. Reactions were monitored by thin-layer chromatography (TLC) and carried out on 0.25 mm coated commercial silica gel plates using UV light as visualizing agent. Flash chromatography was performed on silica gel (Silicycle, SiliaFlash P60, 200-300 mesh).

2 Experimental Procedures and Spectral Data

2.1 Preparation of Substrates

2.1.1 Preparation of Silyl Enol Ethers

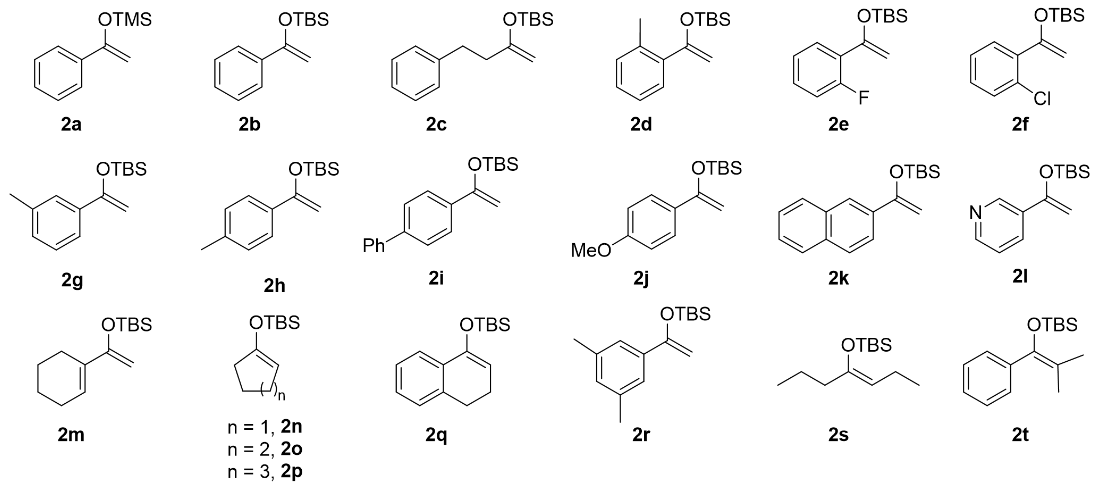


Figure S1. Silyl Enol Ethers

The compounds **2a** was commercially available. The compounds (**2b**[1], **2c**[2], **2d**[1], **2e**[3], **2f**[3], **2g**[1], **2h**[1], **2i**[1], **2j**[1], **2k**[3], **2l**[3], **2m**[4], **2n**[5], **2o**[5], **2p**[5], **2q**[5], **2r**[6], **2s**[7], **2t**[7]) were prepared according to known procedures in the literature.

2.1.2 Synthesis of Oxime Phosphonates

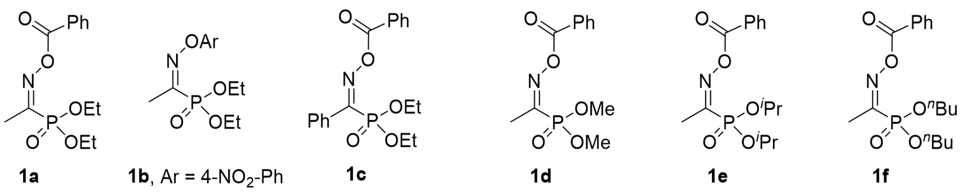
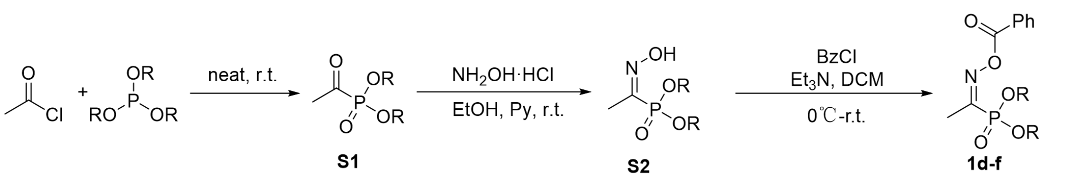


Figure S2. Oxime Ester Phosphonates

The following compounds (**1a-c**) are known compounds and all the spectral data are consistent with reported literature[8]. The compounds (**1d-f**) were prepared according to known procedures in the literature (see the General Procedure below for details)

General Procedure: Synthesis of Oxime Phosphonates[8]

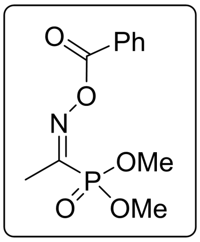


Scheme S1: Synthesis of Oxime Phosphonates

To an oven-dried round-bottom flask fitted with a magnetic stir-bar, acetyl chloride (11.0 mmol, 1.1 equiv.) was added and cooled to 0 °C. Trialkyl phosphite (10.0 mmol, 1.0 equiv.) was added dropwise and the resulting reaction mixture was stirred at 25 °C. After 12 h, the reaction mixture was concentrated under vacuo and the crude product was purified by vacuum distillation. To a solution of the above obtained ketophosphonate **S1** (10.0 mmol, 1.0 equiv.) in EtOH (40 mL) was added hydroxylamine hydrochloride (11.0 mmol, 1.1 equiv.) and pyridine (12.0 mmol, 1.2 equiv.). The mixture was stirred at room temperature overnight. The reaction was quenched with 1N HCl (aq.), and extracted with ethyl acetate, washed with water and brine. The organic phase was collected and the solvent was removed under vacuum to give the crude product **S2**, which was used for the next step without further purification. The above obtained **S2** was dissolved in dichloromethane (40 mL). Then triethylamine (1.2 equiv.) and benzoyl chloride (1.05 equiv) were added to the mixture at 0 °C. The reaction mixture was allowed warm to room temperature and stirred overnight. The crude product was purified by chromatography to give the oxime phosphonates **1d-f**.

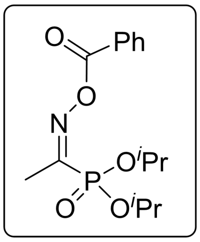
Characterization of Products

Dimethyl-(1-((benzoyloxy)imino)ethyl)phosphonate (1d)



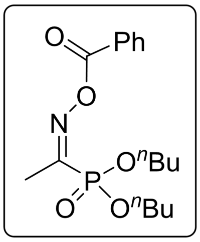
1d: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/3) to afford **1d** (0.894 g, 30 % yield) as a colorless liquid.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 3.86 (d, *J* = 11.2 Hz, 6H), 2.27 (d, *J* = 10.8 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.7, 159.6 (d, *J* = 200.9 Hz), 133.8, 129.7, 128.7, 128.2 (d, *J* = 2.3 Hz), 54.2 (d, *J* = 6.5 Hz), 14.2 (d, *J* = 14.7 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 9.4; HRMS (ESI) *m/z* calcd for $C_{11}H_{14}NO_5PNa$ $(M+Na)^+$ 294.05018, found 294.04904.

Diisopropyl-(1-((benzoyloxy)imino)ethyl)phosphonate (1e)



1e: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/3) to afford **1e** (1.70 g, 52% yield) as a colorless liquid.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.1 Hz, 2H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 4.96–4.77 (m, 2H), 2.30 (d, *J* = 10.6 Hz,3H), 1.43 (d, *J* = 6.2 Hz, 6H), 1.40 (d, *J* = 6.2 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.1 (d, *J* = 2.0 Hz), 160.8 (d, *J* = 201.8 Hz), 133.8, 129.8, 128.8, 128.7 (d, *J* = 2.8 Hz), 73.4 (d, *J* = 6.6 Hz), 24.2 (d, *J* = 3.7 Hz), 23.8 (d, *J* = 5.3 Hz), 14.3 (d, *J* = 14.5 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 5.3; HRMS (ESI) *m/z* calcd for $C_{15}H_{22}NO_5PNa$ $(M+Na)^+$ 350.11278, found 350.11172.

Dibutyl-(1-((benzoyloxy)imino)ethyl)phosphonate (1f)



1f: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/3) to afford **1f** (2.410 g, 68% yield) as a colorless liquid.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.1 Hz, 2H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 4.36–4.10 (m, 4H), 2.31 (d, *J* = 10.7 Hz, 3H), 1.81–1.59 (m, 4H), 1.55–1.36 (m, 4H), 0.94 (t, *J* = 7.4 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.0 (d, *J* = 1.7 Hz), 160.3 (d, *J* = 200.5 Hz), 133.8, 129.9, 128.8, 128.6 (d, *J* = 2.8 Hz), 67.9 (d, *J* = 6.7 Hz), 32.5 (d, *J* = 6.3 Hz), 18.8, 14.4 (d, *J* = 14.6 Hz), 13.7; **^{31}P NMR** (160 MHz, $CDCl_3$) δ 7.2; HRMS (ESI) *m/z* calcd for $C_{17}H_{26}NO_5PNa$ $(M+Na)^+$ 378.14408, found 378.14325.

2.2 Photocatalytic Coupling of Silyl Enol Ethers with Oxime Phosphonates

2.2.1 Reaction Optimization

To an 10-mL Schlenk tube equipped with magnetic stir bar were sequentially added oxime phosphonate **1**, enol silyl ether **2**, photocatalyst (1 mol%) and solvent. Then the resulting mixture was degassed via “freeze, pump, thraw” operation. The Schlenk tube was irradiated at a distance of approx 5 cm from the light source for 24 h (Kessil LED, 40 W). The internal temperature was maintained at approximately 29 °C by an electric fan placed approximately 10 cm above the Schlenk tube. The reaction set up apparatus is shown in Figure S3. After 24 h, the yield was determined by ^{1}H NMR using 1, 3, 5-trimethoxybenzene as an internal standard. The results were summarized in **Table S1-4**.

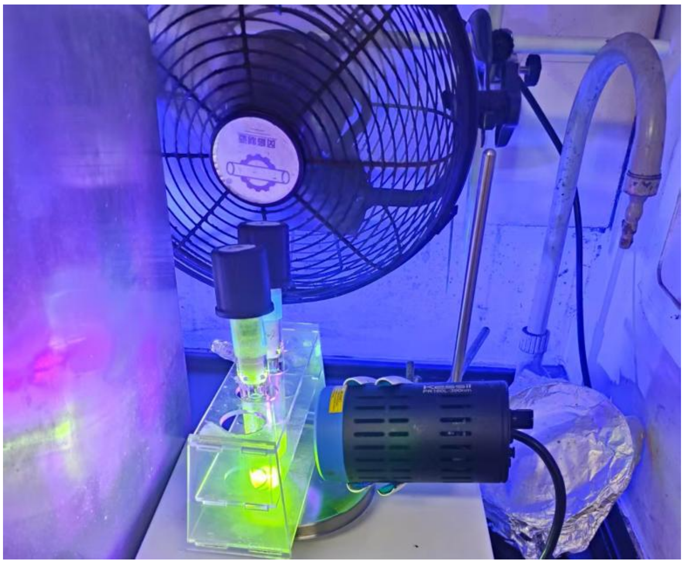
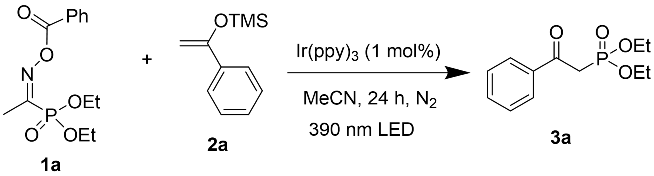


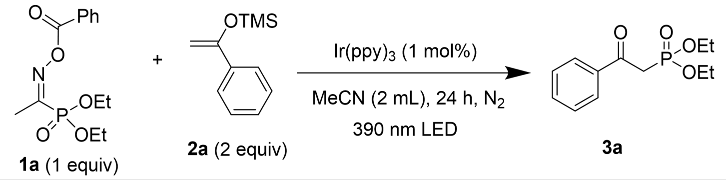
Figure S3: Reaction Setup Diagram

Table S1: Screening of Reactant Ratios, Solvent Types and Concentrations



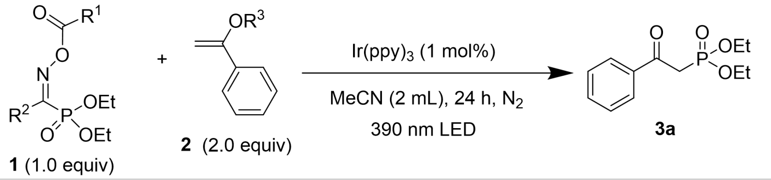
Entry	**1a** (equiv)	**2a** (equiv)	Solvent	Yield of **3a** (%)
1	1	2	MeCN (2 mL)	83
2	1	3	MeCN (2 mL)	81
3	2	1	MeCN (2 mL)	60
4	1	2	MeCN (1 mL)	57
5	1	2	MeCN (3 mL)	83
6	1	2	DCM (2 mL)	30
7	1	2	DCE (2 mL)	0
8	1	2	DMF (2 mL)	0
9	1	2	DMSO (2 mL)	57

Table S2: Screening of Photocatalysts and Light Sources



Entry	Photocatalyst	Wavelength of light	Yield of **3a** (%)
1	$Ir(ppy)_3$	390 nm	83
2	$Ir(ppy)_3$	456 nm	40
3	$Ir[dF(CF_3)ppy]_2(dtbbpyPF_6)$	456 nm	28
4	$Ir[dF(CF_3)ppy]_2(dtbbpyPF_6)$	390 nm	0
5	3DPA2FBN	456 nm	0
6	4CzIPN	456 nm	0
7	$Mes\text{-}Acr^+BF_4^-$	456 nm	0

Table S3: Screening of Oxime Phosphonates and Silyl Enol Ethers



Entry	Oxime phosphonate	Silyl enol ether	Yield of **3a** (%)
1	**1a**	**2a**	83
2	**1b**	**2a**	50
3	**1c**	**2a**	80
4	**1a**	**2b**	88
5	**1d**	**2b**	0
6	**1e**	**2b**	0

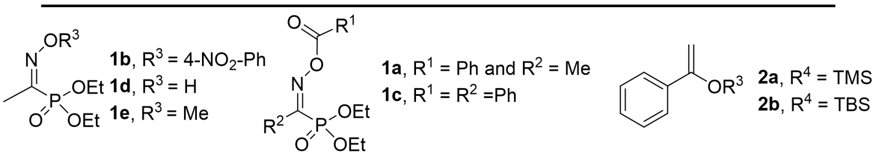
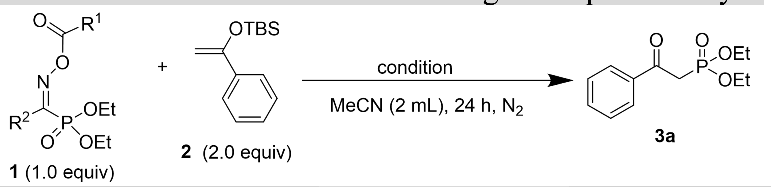
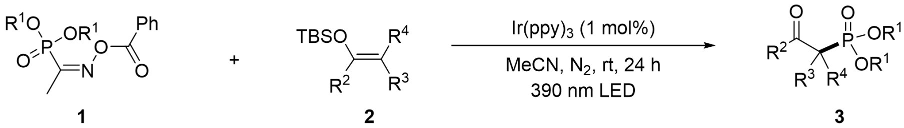


Table S4: Thermal reaction without light and photocatalyst.



Entry	Temperature (ºC)	light and photocatalyst.	Yield of **3a** (%)
1	29	390nm LED, $Ir(ppy)_3$	88
2	50	none	0
3	80	none	0

General Procedure for Substrate Scope

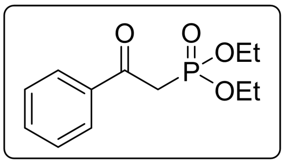


Scheme S2: General Procedure for Substrate Scope

To an 10-mL Schlenk tube equipped with magnetic stir bar were sequentially added oxime ester phosphonate (0.2 mmol, 1.0 equiv), silyl enol ether (0.4 mmol, 2.0 equiv), $Ir(ppy)_3$ (1 mol%) and MeCN (2 mL). Then the resulting mixture was degassed via “freeze, pump, thraw” operation. The Schlenk tube was irradiated at a distance of approx. 5 cm from the light source for 24 h (Kessil LED, 40 W). The internal temperature was maintained at approximately 29 ^{o}C by an electric fan placed approximately 10 cm above the Schlenk tube. After 24 h, The mixture was concentrated under reduced pressure and purified by flash column chromatography to afford the **3a-3u**.

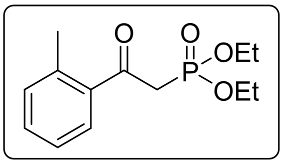
2.2.2 Characterization of Products

Diethyl (2-oxo-2-phenylethyl)phosphonate (3a)



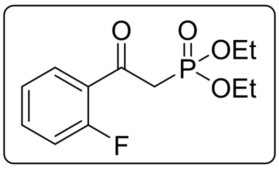
3a: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3a** (41.4 mg, 81% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.3 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 4.16–4.08 (m, 4H), 3.62 (d, *J* = 22.7 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.1 (d, *J* = 6.6 Hz), 136.6, 133.8, 129.2, 128.7, 62.8 (d, *J* = 6.4 Hz), 38.6 (d, *J* = 130.1 Hz), 16.4 (d, *J* = 6.2 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 19.9.
The spectroscopic data are consistent with those reported in the literature[9].

Diethyl (2-oxo-2-(*o*-tolyl)ethyl)phosphonate (3b)



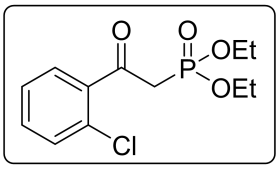
3b: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3b** (47.5 mg, 88% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.31–7.22 (m, 2H), 4.15–4.07 (m, *J* = 7.2 Hz, 4H), 3.59 (d, *J* = 22.5 Hz, 2H), 2.51 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.2 (d, *J* = 6.6 Hz), 139.1, 137.4 (d, *J* = 1.9 Hz), 132.1, 132.1, 129.7, 125.8, 62.7 (d, *J* = 6.3 Hz), 41.2 (d, *J* = 129.4 Hz), 21.5, 16.4 (d, *J* = 6.4 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.2.
The spectroscopic data are consistent with those reported in the literature[9].

Diethyl (2-(2-fluorophenyl)-2-oxoethyl)phosphonate (3c)



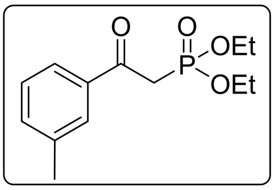
3c: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3c** (45.7 mg, 83% yield) as a yellow oil.; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85 (t, J = 7.7 Hz, 1H), 7.57–7.50 (m, 1H), 7.25–7.21 (m, 1H), 7.13 (dd, *J* = 12.5, 8.3 Hz, 1H), 4.15–4.09 (m, 4H), 3.71 (d, *J* = 22.2 Hz, 2H), 1.25 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 190.1 (dd, *J* = 7.3, 2.8 Hz), 161.9 (d, *J* = 255.1 Hz), 135.3 (d, *J* = 9.2 Hz), 131.2 (d, *J* = 2.0 Hz), 125.7 (dd, *J* = 11.5, 2.3 Hz), 124.7 (d, *J* = 3.4 Hz), 116.8 (d, *J* = 23.9 Hz), 62.7 (d, *J* = 6.4 Hz), 42.6 (dd, *J* = 130.3, 7.8 Hz), 16.3 (d, *J* = 6.5 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 19.8; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -109.89.
The spectroscopic data are consistent with those reported in the literature[9].

Diethyl (2-(2-chlorophenyl)-2-oxoethyl)phosphonate (3d)



3d: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3d** (40.6 mg, 70% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59–7.52 (m, 1H), 7.43 –7.35 (m, 2H), 7.35–7.29 (m, 1H), 4.22–3.99 (m, 4H), 3.69 (d, *J* = 22.3 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.5 (d, *J* = 6.7 Hz), 138.8, 132.4, 131.2, 130.6, 130.1, 127.1, 62.8 (d, *J* = 6.5 Hz), 42.3 (d, *J* = 128.6 Hz), 16.4 (d, *J* = 6.5 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 19.1.
The spectroscopic data are consistent with those reported in the literature[10].

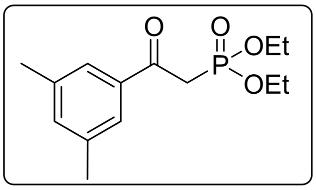
Diethyl (2-oxo-2-(*m*-tolyl)ethyl)phosphonate (3e)



3e: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3e** (44.2 mg, 82% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77–7.79 (m, 2H), 7.30–7.48 (m, 2H), 4.15–4.08 (m, 4H), 3.60 (d, *J* = 22.7 Hz, 2H), 2.39 (s, 3H), 1.26 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.2 (d, *J* = 6.6 Hz), 138.5, 136.7 (d, *J* = 2.1 Hz), 134.5, 129.5, 128.6, 126.4, 62.7 (d, *J* = 6.6 Hz), 38.4 (d, *J* = 130.0 Hz), 21.4, 16.3 (d, *J* = 6.3 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.1.

The spectroscopic data are consistent with those reported in the literature[9].

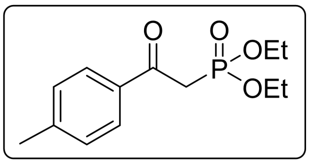
Diethyl (2-(3,5-dimethylphenyl)-2-oxoethyl)phosphonate (3f)



3f: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3f** (48.9 mg, 86% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59 (s, 2H), 7.20 (s, 1H), 4.30–3.94 (m, 4H), 3.58 (d, *J* = 22.7 Hz, 2H), 2.35 (s, 6H), 1.26 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.4 (d, *J* = 6.7 Hz), 138.3, 136.7 (d, *J* = 2.2 Hz), 135.4, 126.9, 62.7 (d, *J* = 6.5 Hz), 38.5 (d, *J* = 130.3 Hz), 21.3, 16.3 (d, *J* = 6.4 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.2.

The spectroscopic data are consistent with those reported in the literature[11].

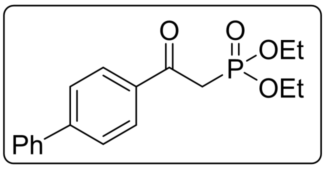
Diethyl (2-oxo-2-(*p*-tolyl)ethyl)phosphonate (3g)



3g: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3g** (42.6 mg, 79% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 4.29–3.89 (m, 4H), 3.57 (d, *J* = 22.7 Hz, 2H), 2.38 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.6 (d, *J* = 6.6 Hz), 144.7, 134.2 (d, *J* = 2.1 Hz), 129.4, 129.3, 62.7 (d, *J* = 6.5 Hz), 38.4 (d, *J* = 130.0 Hz), 21.8, 16.3 (d, *J* = 6.3 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.2.

The spectroscopic data are consistent with those reported in the literature[9].

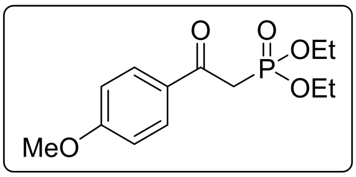
Diethyl (2-([1,1'-biphenyl]-4-yl)-2-oxoethyl)phosphonate (3h)



3h: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3h** (48.5 mg, 73% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.3 Hz, 2H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 7.5 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 1H), 4.24–4.08 (m, 4H), 3.66 (d, *J* = 22.8 Hz, 2H), 1.43–1.16 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.6 (d, *J* = 6.6 Hz), 146.4, 139.8, 135.3 (d, *J* = 2.1 Hz), 129.8, 129.1, 128.5, 127.4, 127.3, 62.8 (d, *J* = 6.5 Hz), 38.7 (d, *J* = 129.8 Hz), 16.4 (d, *J* = 6.4 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.0.

The spectroscopic data are consistent with those reported in the literature[11].

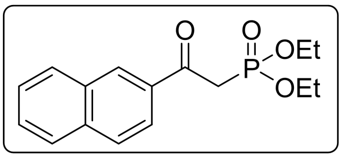
Diethyl (2-(4-methoxyphenyl)-2-oxoethyl)phosphonate (3i)



3i: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3i** (40.6 mg, 71% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 9.0 Hz, 2H), 6.92 (d, *J* = 9.0 Hz, 2H), 4.14-4.07 (m, 4H), 3.85 (s, 3H), 3.55 (d, *J* = 22.7 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 6H);

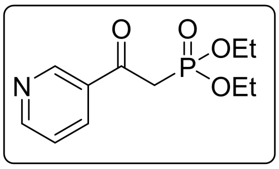
^{13}C NMR (100 MHz, $CDCl_3$) δ 190.4 (d, *J* = 6.4 Hz), 164.1, 131.6, 129.7, 113.9, 62.7 (d, *J* = 6.6 Hz), 55.6, 38.3 (d, *J* = 129.8 Hz), 16.4 (d, *J* = 6.3 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.4.
The spectroscopic data are consistent with those reported in the literature[9].

Diethyl (2-(naphthalen-2-yl)-2-oxoethyl)phosphonate (3j)



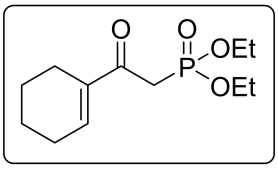
3j: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3j** (33 mg, 54% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 8.06 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.89 (t, *J* = 8.3 Hz, 2H), 7.66–7.50 (m, 2H), 4.25–4.05 (m, 4H), 3.76 (d, *J* = 22.7 Hz, 2H), 1.28 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.0 (d, *J* = 6.5 Hz), 135.9, 134.0 (d, *J* = 1.8 Hz), 132.5, 131.6, 129.9, 129.0, 128.6, 127.9, 127.0, 124.3, 62.9 (d, *J* = 6.6 Hz), 38.8 (d, *J* = 129.9 Hz), 16.4 (d, *J* = 6.4 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 20.0.
The spectroscopic data are consistent with those reported in the literature[12].

Diethyl (2-oxo-2-(pyridin-3-yl)ethyl)phosphonate (3k)



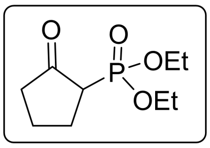
3k: The crude residue was purified by column chromatography on silica gel (DCM/MeOH = 15/1) to afford **3k** (19.5 mg, 38% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.79 (d, *J* = 3.6 Hz, 1H), 8.30 (d, *J* = 8.0 Hz, 1H), 7.44 (dd, *J* = 8.0, 4.8 Hz, 1H), 4.30–3.96 (m, 4H), 3.63 (d, *J* = 22.9 Hz, 2H), 1.28 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.0 (d, *J* = 7.0 Hz), 153.9, 150.5, 136.6, 131.9, 123.7, 63.0 (d, *J* = 6.5 Hz), 39.0 (d, *J* = 129.3 Hz), 16.4 (d, *J* = 6.5 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 18.9.
The spectroscopic data are consistent with those reported in the literature[12].

Diethyl (2-(cyclohex-1-en-1-yl)-2-oxoethyl)phosphonate (3l)



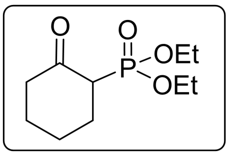
3l: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3l** (36.2 mg, 70% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.11–6.85 (m, 1H), 4.27–3.98 (m, 4H), 3.30 (d, *J* = 24.0 Hz, 2H), 2.27–2.22 (m, 4H), 1.78–1.51 (m, 4H), 1.29 (t, *J* = 6.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.7 (d, *J* = 6.2 Hz), 143.8, 139.4 (d, *J* = 1.8 Hz), 62.6 (d, *J* = 6.5 Hz), 37.1 (d, *J* = 130.9 Hz), 26.4, 23.2, 21.9, 21.5, 16.4 (d, *J* = 6.3 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 21.1.
The spectroscopic data are consistent with those reported in the literature[13].

Diethyl (2-oxocyclopentyl)phosphonate (3m)



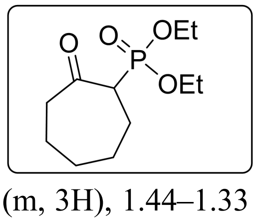
3m: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3m** (31.2 mg, 71% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.34 –4.00 (m, 4H), 2.70 (dt, *J* = 26.0, 8.4 Hz, 1H), 2.42–2.20 (m, 4H), 2.19–2.05 (m, 1H), 1.94–1.81 (m, 1H), 1.30 (t, *J* = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 212.2, 62.7 (d, *J* = 6.7 Hz), 62.3 (d, *J* = 6.7 Hz), 47.0 (d, *J* = 137.7 Hz), 39.0 (d, *J* = 3.7 Hz), 25.5 (d, *J* = 3.3 Hz), 21.7 (d, *J* = 9.0 Hz), 16.4 (d, *J* = 6.1 Hz), 16.3 (d, *J* = 6.1 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 22.9.
The spectroscopic data are consistent with those reported in the literature[14].

Diethyl (2-oxocyclohexyl)phosphonate (3n)



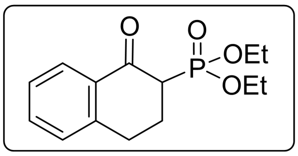
3n: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3n** (35.6 mg, 76% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.71 (s, 1H, enol form), 4.35–3.81 (m, 4H), 2.93 (dt, *J* = 23.5, 5.9 Hz, 1H), 2.67–2.53 (m, 1H), 2.40–1.54 (m, 7H), 1.31–1.26 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 206.4, 62.5 (d, *J* = 6.9 Hz), 62.4 (d, *J* = 6.7 Hz), 50.4 (d, *J* = 133.2 Hz), 41.8 (d, *J* = 2.5 Hz), 28.1 (d, *J* = 5.2 Hz), 26.7, 22.8 (d, *J* = 6.2 Hz), 16.5 (d, *J* = 6.1 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 23.5.
The spectroscopic data are consistent with those reported in the literature[14].

Diethyl (2-oxocycloheptyl)phosphonate (3o)



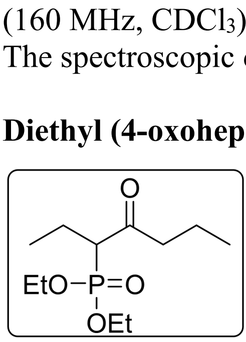
3o: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3o** (34.7 mg, 70% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.23–3.93 (m, 4H), 2.99 (ddd, *J* = 25.2, 12.1, 4.9 Hz, 1H), 2.76 (td, *J* = 12.1, 2.9 Hz, 1H), 2.45–2.40 (m, 1H), 2.23–2.15 (m, 1H), 2.03–1.98 (m, 1H), 1.95–1.79 (m, 3H), 1.44–1.33 (m, 2H), 1.29 (t, *J* = 7.0 Hz, 3H), 1.28 (t, *J* = 7.0 Hz, 3H), 1.23–1.08 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 212.3 (d, *J* = 4.6 Hz), 62.8 (d, *J* = 6.6 Hz), 62.4 (d, *J* = 6.7 Hz), 47.1 (d, *J* = 137.9 Hz), 39.1, 39.0, 25.6, 25.6, 21.8 (d, *J* = 8.9 Hz), 16.4 (d, *J* = 6.0 Hz), 16.4 (d, *J* = 6.0 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 22.9.
The spectroscopic data are consistent with those reported in the literature[14].

Diethyl (1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl)phosphonate (3p)



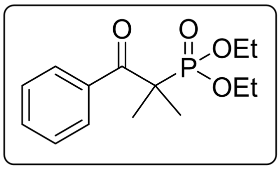
3p: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3p** (78% yield, NMR yield with 1,3,5-trimethoxybenzene as internal standard) as a yellow oil. Since it was not possible to obtain a highly pure sample through column chromatography, only the NMR yield of compound **3p** is provided in Scheme 3 in the main text.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.5 Hz, 1H), 7.48 (t, *J* = 8.1 Hz, 1H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 4.34–3.74 (m, 4H), 3.41–3.09 (m, 2H), 2.92 (dt, *J* = 16.8, 5.6 Hz, 1H), 2.63–2.35 (m, 2H), 1.4 (t, *J* = 7.0 Hz, 3H), 1.21 (t, *J* = 7.1 Hz, 3H); 13C NMR (100 MHz, CDCl3) δ 192.7 (*d*, J = 5.2 Hz), 144.0, 134.0, 132.3 (*d*, J = 2.9 Hz), 128.9, 127.8, 126.9, 62.8 (*d*, J = 6.6 Hz), 62.6 (*d*, J = 6.6 Hz), 47.4 (*d*, J = 133.0 Hz), 27.6 (*d*, J = 7.4 Hz), 24.6 (*d*, J = 4.4 Hz), 16.4 (*t*, J = 6.2 Hz);**^{31}P NMR** (160 MHz, $CDCl_3$) δ 23.2.
The spectroscopic data are consistent with those reported in the literature[15].

Diethyl (4-oxoheptan-3-yl)phosphonate (3q)



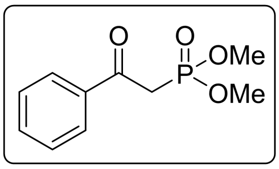
3q: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3q** (42.1 mg, 84% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.25–3.90 (m, 4H), 3.14–2.96 (m, 1H), 2.72-2.64 (m, 1H), 2.48-2.40 (m, 1H), 2.03-1.96 (m, 1H), 1.84-1.77 (m, 1H), 1.63-1.53 (m, 2H), 1.36–1.21 (m, 6H), 0.94–0.79 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 206.1 (d, *J* = 4.4 Hz), 62.7 (d, *J* = 6.8 Hz), 62.5 (d, *J* = 6.9 Hz), 54.6 (d, *J* = 125.2 Hz), 46.3, 20.2 (d, *J* = 5.3 Hz), 16.9, 16.4 (d, *J* = 5.9 Hz), 16.4 (d, *J* = 6.1 Hz), 13.7, 13.2 (d, *J* = 16.0 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 22.6; HRMS (ESI) *m/z* calcd for $C_{11}H_{24}O_4P$ $(M+H)^+$ 251.14067, found 251.14043.

Diethyl (2-methyl-1-oxo-1-phenylpropan-2-yl)phosphonate (3r)



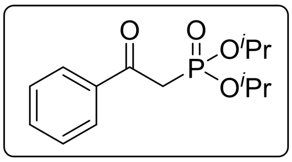
3r: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3r** (43.0 mg, 76% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (d, J = 7.1 Hz, 2H), 7.44 (d, J = 7.3 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 4.16–4.09 (m, 4H), 1.57 (d, J = 16.6 Hz, 6H), 1.29 (t, J = 7.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 203.0, 138.6, 131.5, 128.7, 128.0, 63.0 (d, J = 7.2 Hz), 50.3 (d, J = 131.0 Hz), 23.0 (d, J = 5.0 Hz), 16.5 (d, J = 5.9 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 27.3.
The spectroscopic data are consistent with those reported in the literature[7].

Dimethyl (2-oxo-2-phenylethyl)phosphonate (3s)



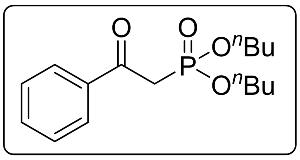
3s: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3s** (33.3 mg, 73% yield) as a yellow oil.; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.98 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 3.76 (d, J = 11.2 Hz, 6H), 3.62 (d, J = 22.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.8 (d, J = 6.7 Hz), 136.4 (d, J = 2.5 Hz), 133.9, 129.0, 128.8, 53.2 (d, J = 6.5 Hz), 37.5 (d, J = 131.4 Hz); **^{31}P NMR** (160 MHz, $CDCl_3$) δ 22.8.
The spectroscopic data are consistent with those reported in the literature[9].

Diisopropyl (2-oxo-2-phenylethyl)phosphonate (3t)



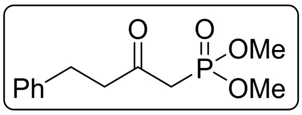
3t: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3t** (31.8 mg, 56% yield) as a yellow oil.; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.01 (d, J = 8.3 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 5.00–4.23 (m, 2H), 3.58 (d, J = 22.9 Hz, 2H), 1.27 (d, J = 6.1 Hz, 6H), 1.26 (d, J = 6.1 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.2 (d, J = 6.7 Hz), 136.8, 133.6, 129.2, 128.6, 71.6 (d, J = 6.7 Hz), 39.8 (d, J = 130.4 Hz), 24.1 (d, J = 3.9 Hz), 23.9 (d, J = 5.2 Hz); **^{31}P NMR** (200 MHz, $CDCl_3$) δ 18.4.
The spectroscopic data are consistent with those reported in the literature[9].

Dibutyl (2-oxo-2-phenylethyl)phosphonate (3u)



3u: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3u** (49.2 mg, 79% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, J = 7.3 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.08-4.02 (m, 4H), 3.62 (d, J = 22.8 Hz, 2H), 1.61-1.54 (m, 4H), 1.35-1.26 (m, 4H), 0.87 (t, J = 7.4 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 192.0 (d, J = 6.7 Hz), 136.7 (d, J = 2.0 Hz), 133.7, 129.2, 128.7, 66.5 (d, J = 6.8 Hz), 38.4 (d, J = 129.6 Hz), 32.5 (d, J = 6.4 Hz), 18.7, 13.7; **^{31}P NMR** (160 MHz, $CDCl_3$) δ 19.9.
The spectroscopic data are consistent with those reported in the literature[9].

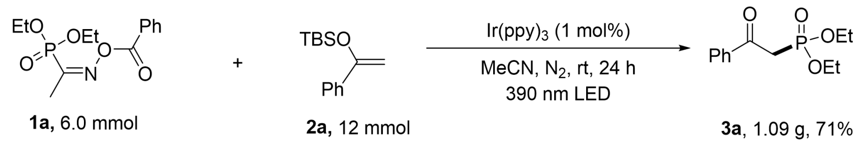
Dimethyl (2-oxo-4-phenylbutyl)phosphonate (3v)



3v: The crude residue was purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3v** (29.8 mg, 58% yield) as a yellow oil.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.25–7.21 (m, 2H), 7.19–7.12 (m, 3H), 3.71 (d, J = 11.2 Hz, 6H), 3.04 (d, J = 22.7 Hz,

2H), 2.97–2.83 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 201.0 (d, J = 6.1 Hz), 140.6, 128.6, 128.5, 126.3, 53.1 (d, J = 6.5 Hz), 45.6 (d, J = 0.9 Hz), 41.6 (d, J = 128.0 Hz), 29.5; **^{31}P NMR** (160 MHz, $CDCl_3$) δ 22.5.
The spectroscopic data are consistent with those reported in the literature[16].

2.2.3 Scale-up Reaction

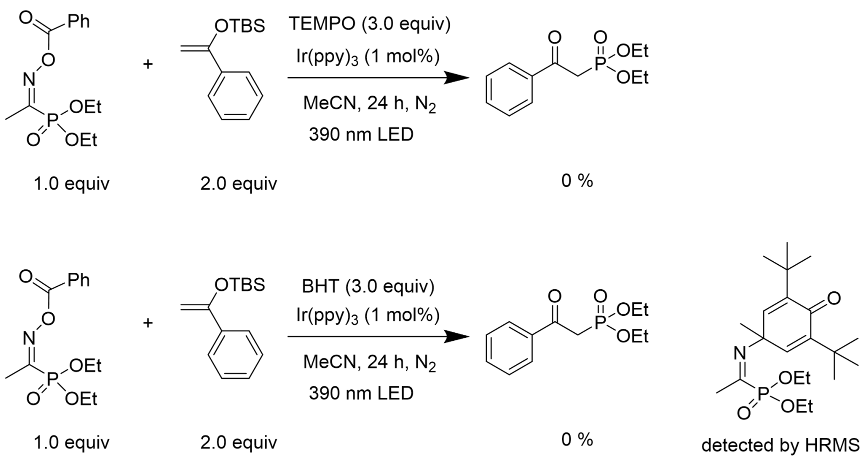


Scheme S3: Scale-up Reaction

To an 250-mL Schlenk round flask equipped with magnetic stir bar were sequentially added oxime ester phosphonate (6.0 mmol, 1.0 equiv), silyl enol ether (12.0 mmol, 2.0 equiv), Ir(ppy)$_3$ (1 mol%) and MeCN (60 mL). Then the resulting mixture was degassed via "freeze, pump, thraw" operation. The Schlenk round flask was irradiated at a distance of approx. 5 cm from the light source for 24 h (Kessil LED, 40 W). The internal temperature was maintained at approximately 29 °C by an electric fan placed approximately 10 cm above the Schlenk tube. After 24 h, The mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (EtOAc/PE = 1/4~1/1) to afford **3a** (1.09 g, 71% yield) as a yellow oil.

2.3 Mechanistic Experiments

2.3.1 Radical Trapping Experiment



Scheme S4: Radical Trapping Experiment

To an 10-mL Schlenk tube equipped with magnetic stir bar were sequentially added oxime ester phosphonate (0.2 mmol, 1.0 equiv), silyl enol ether (0.4 mmol, 2.0 equiv), TEMPO or BHT (0.3 mmol, 3.0 equiv), Ir(ppy)$_3$ (1 mol%) and MeCN (2 mL). Then the resulting mixture was degassed via "freeze, pump, thraw" operation. The Schlenk tube was irradiated at a distance of approx. 5 cm from the light source for 24 h (Kessil LED, 40 W). ^{1}H NMR analysis indicated that the yield of the corresponding β-ketophosphonate product was 0%, while HRMS confirmed the formation of the BHT radical adduct. The TEMPO radical adduct failed to be trapped successfully. The TEMPO radical adduct failed to be trapped successfully[17].

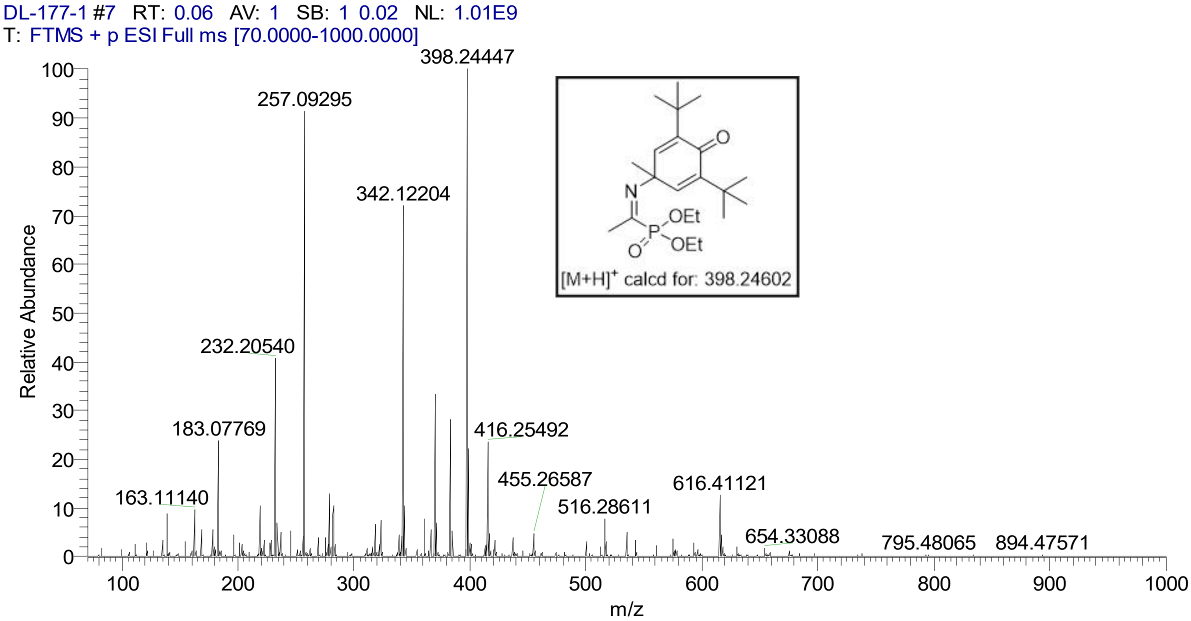


Figure S4: HRMS of the Reaction Solution from the Radical Trapping Experiment

2.3.2 Light On/Off Experiment

To an 10-mL Schlenk tube equipped with magnetic stir bar were sequentially added oxime ester phosphonate (0.2 mmol, 1.0 equiv), silyl enol ether (0.4 mmol, 2.0 equiv), Ir(ppy)$_3$ (1 mol%), 1,3,5-trimethoxybenzene (0.2 mmol, 1.0 equiv) and MeCN (2 mL). Then the resulting mixture was degassed via "freeze, pump, thraw" operation. The Schlenk tube was irradiated at a distance of approx. 5 cm from the light source (Kessil LED, 40 W). The resulting mixture was subjected to alternating intervals of irradiation with 390 nm light and dark at 30-minute intervals, with a sample taken at each interval for subsequent analysis. The reaction profile is shown below and the yield of product **3a** as a function of time was determined by 1H-NMR using 1,3,5-trimethoxybenzene as internal standard. These results indicated that continuous irradiation with light was essential for the progress of this reaction.

Table S5: Light On/Off Experiment

Time (min)	0	30	60	90	120	150	180
Yied of **3a** (%)	0	26	26	45	45	57	57

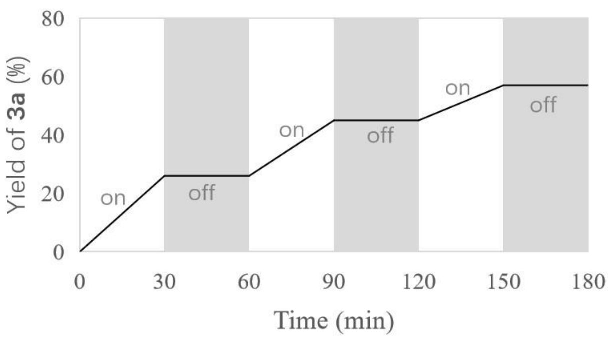


Figure S5: Light On/Off Experiment

2.3.3 Stern-Volmer Quenching Experiment

Stern-Volmer experiments were conducted on an OceanOptics™ QE pro, using a 0.2 mM solution of $Ir(ppy)_3$ in acetonitrile, quencher-A was a 1 M MeCN solution of oxime phosphonates **1a**, and quencher-B was a 1 M MeCN solution of silyl enol ether **2b**. A total of 11 sample sets were prepared in 3.0 mL quartz cuvettes fitted with polytetrafluoroethylene (PTFE) stoppers. For each cuvette, 3 mL of the $Ir(ppy)_3$ MeCN solution was first added, followed by the sequential addition of 0, 10, 30, 60, 120, and 200 μL of the two distinct quenchers, respectively. All samples were thoroughly degassed by bubbling with high-purity argon for 10 min to remove dissolved oxygen, and subsequently sealed with Parafilm to maintain an oxygen-free atmosphere. Excitation was conducted at 390 nm, and the emission luminescence was recorded at 530 nm .The results were summarized in Table S6 and Figure S6-S7. Linear regression of I_0/I against concentration is done in Origin. These experiments indicated that only **1a** will quench excited state of $Ir(ppy)_3$.

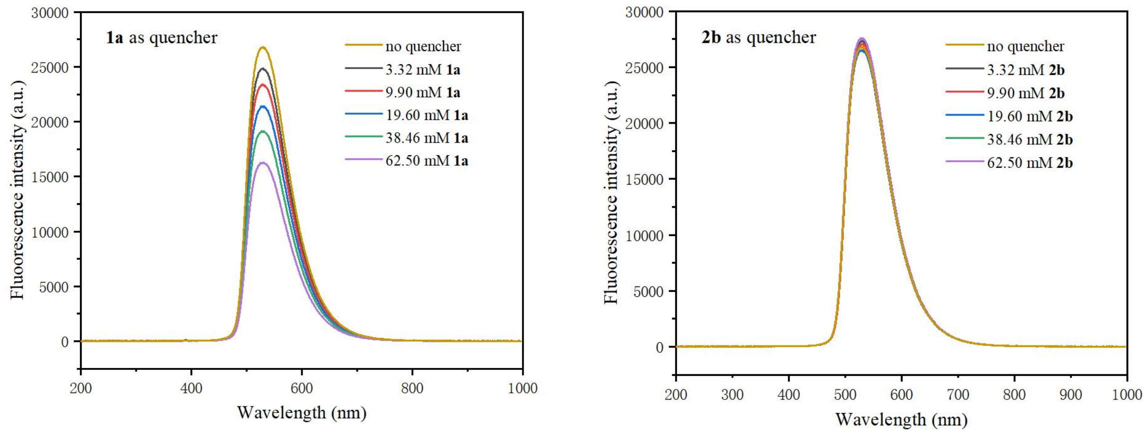


Figure S6: Stern-Volmer Quenching Experiment

Table S6: Stern-Volmer Quenching Experiment

(mM) / I_0/I	0	3.32	9.90	19.60	38.46	62.50
1a	1	1.08	1.15	1.25	1.40	1.65
2b	1	1.02	1.01	0.99	1	1.03

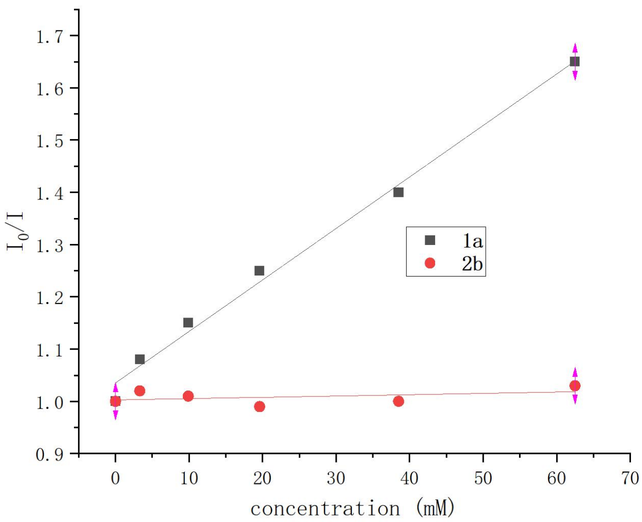
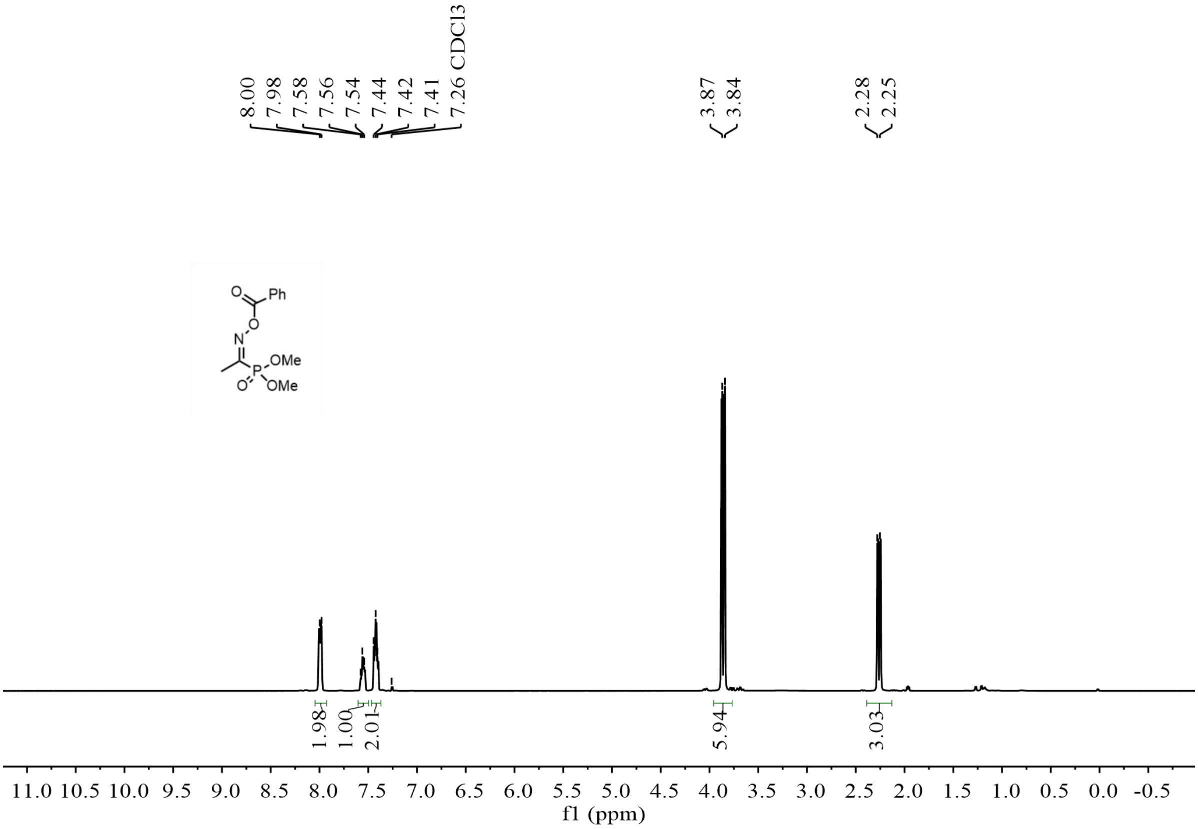
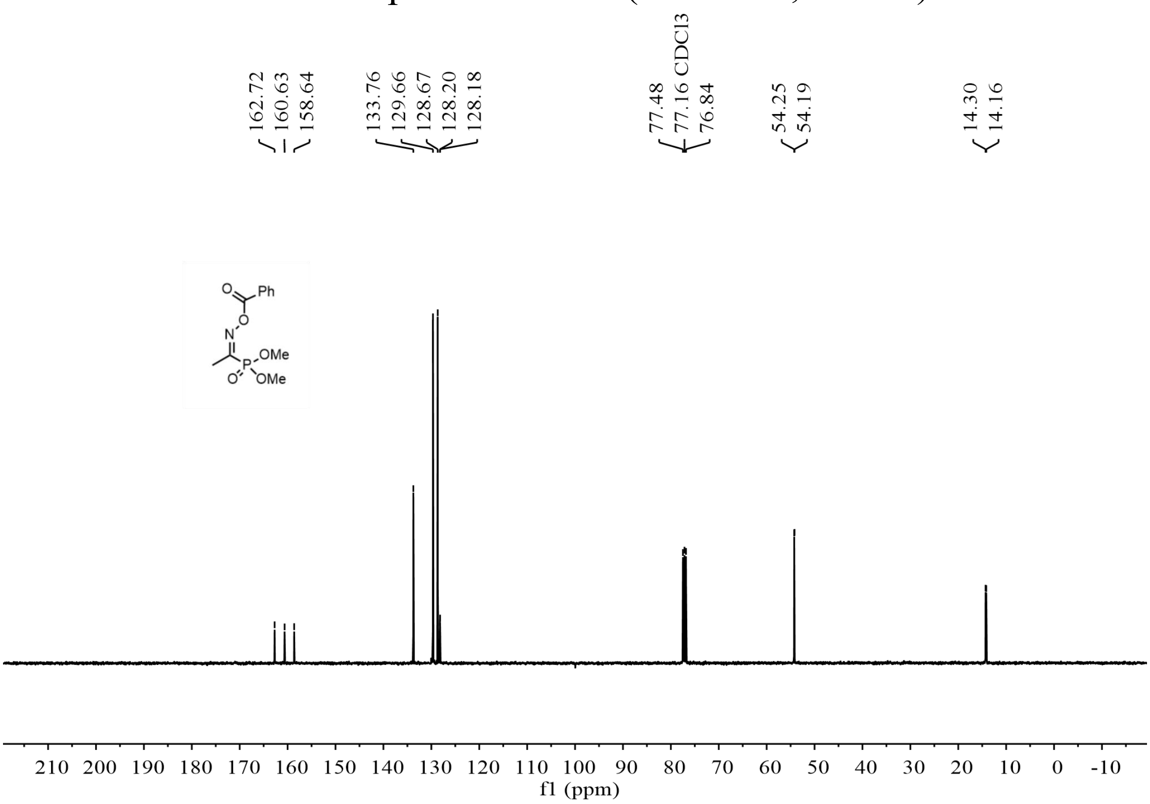


Figure S7: Stern-Volmer Quenching Experiment

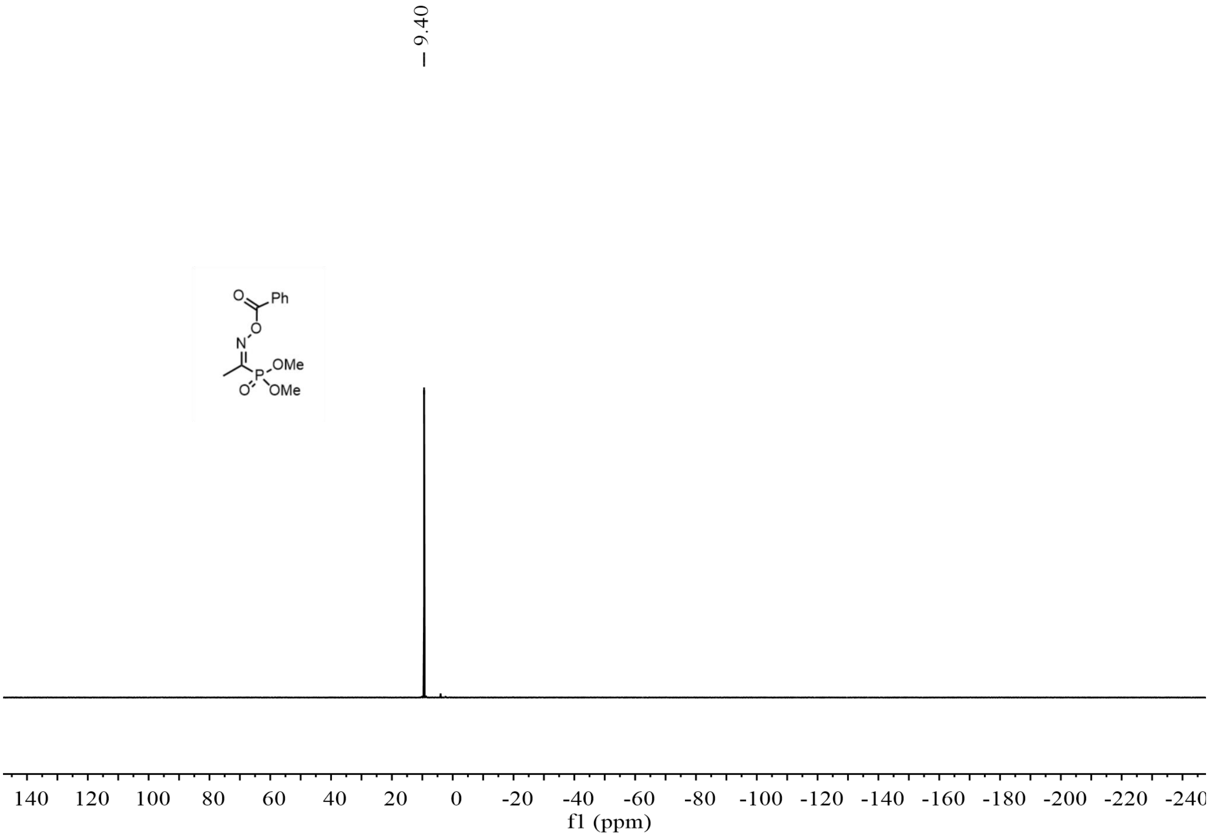
3 ¹H, ¹³C, ¹⁹F, and ³¹P NMR Spectra



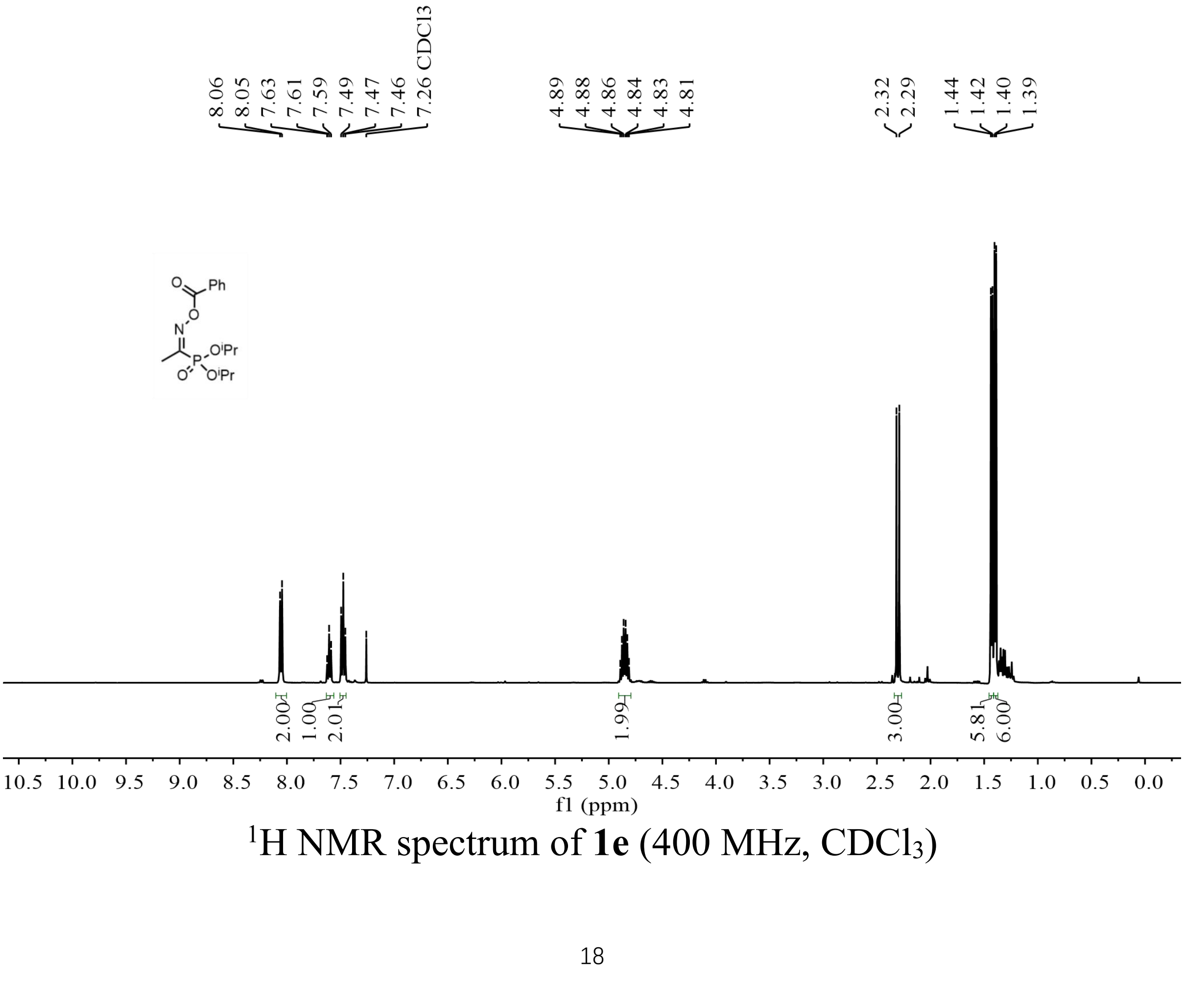
^{1}H NMR spectrum of **1d** (400 MHz, $CDCl_3$)



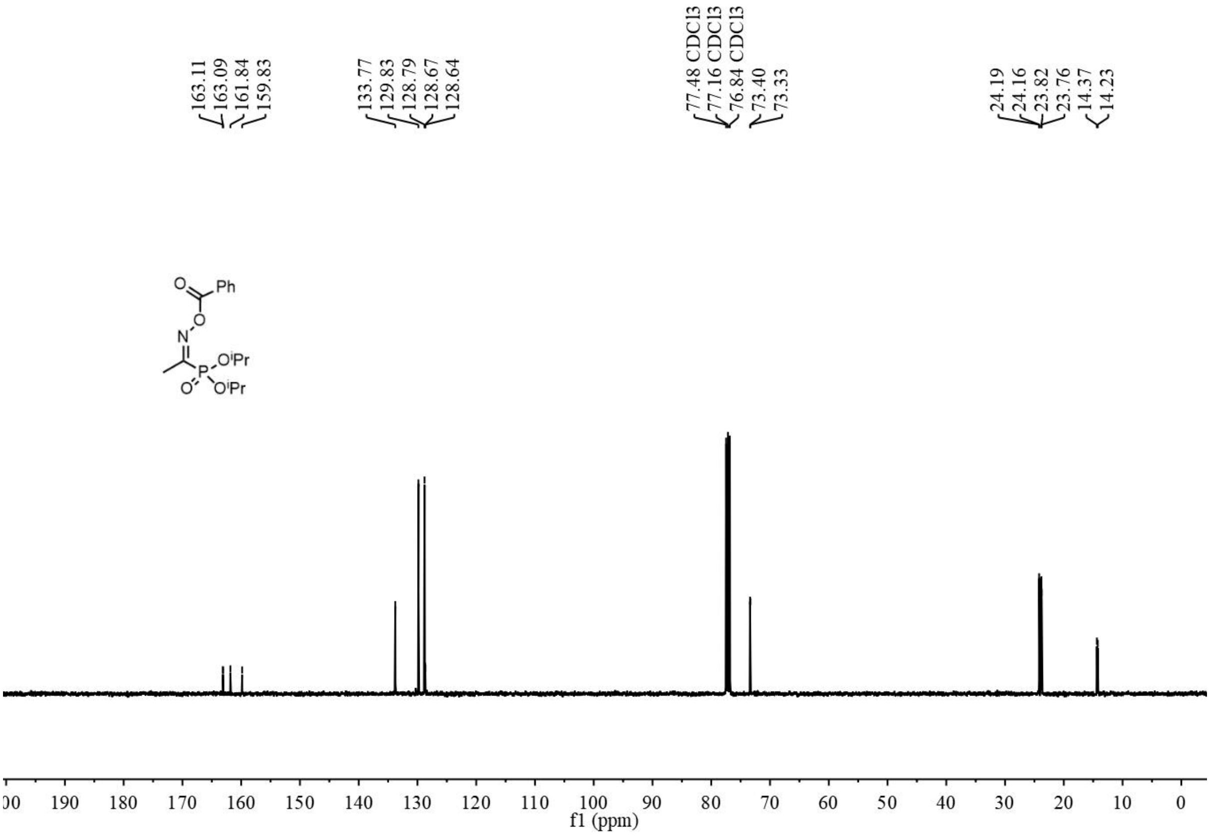
^{13}C NMR spectrum of **1d** (100 MHz, $CDCl_3$)



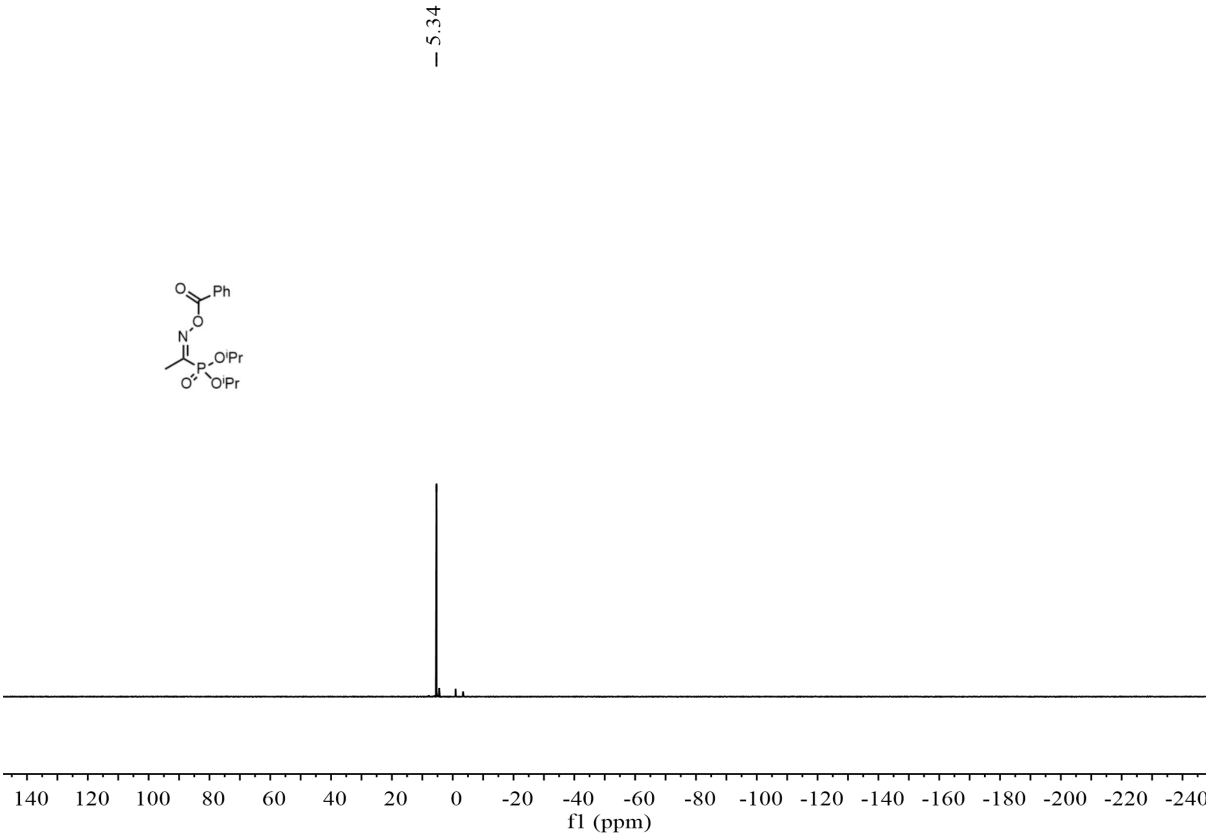
^{31}P NMR spectrum of **1d** (160 MHz, $CDCl_3$)



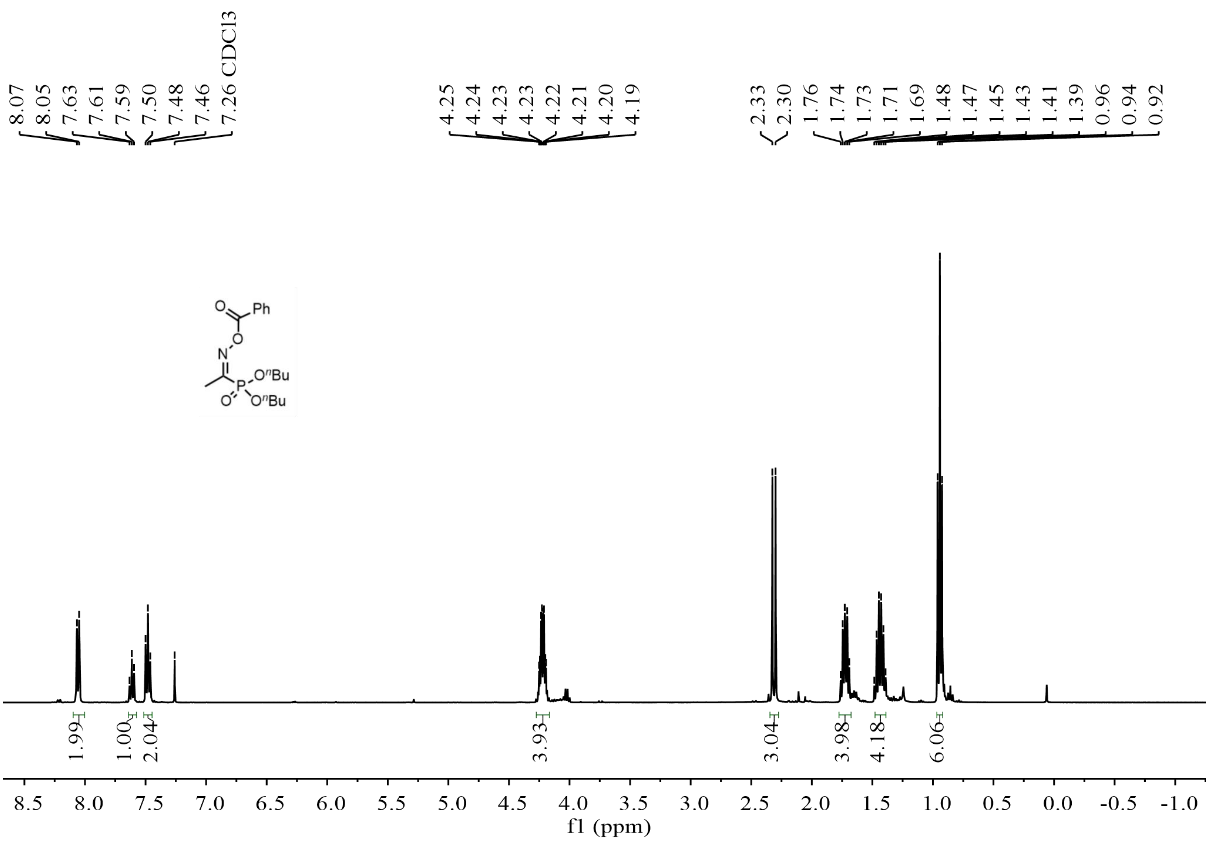
^{1}H NMR spectrum of **1e** (400 MHz, $CDCl_3$)



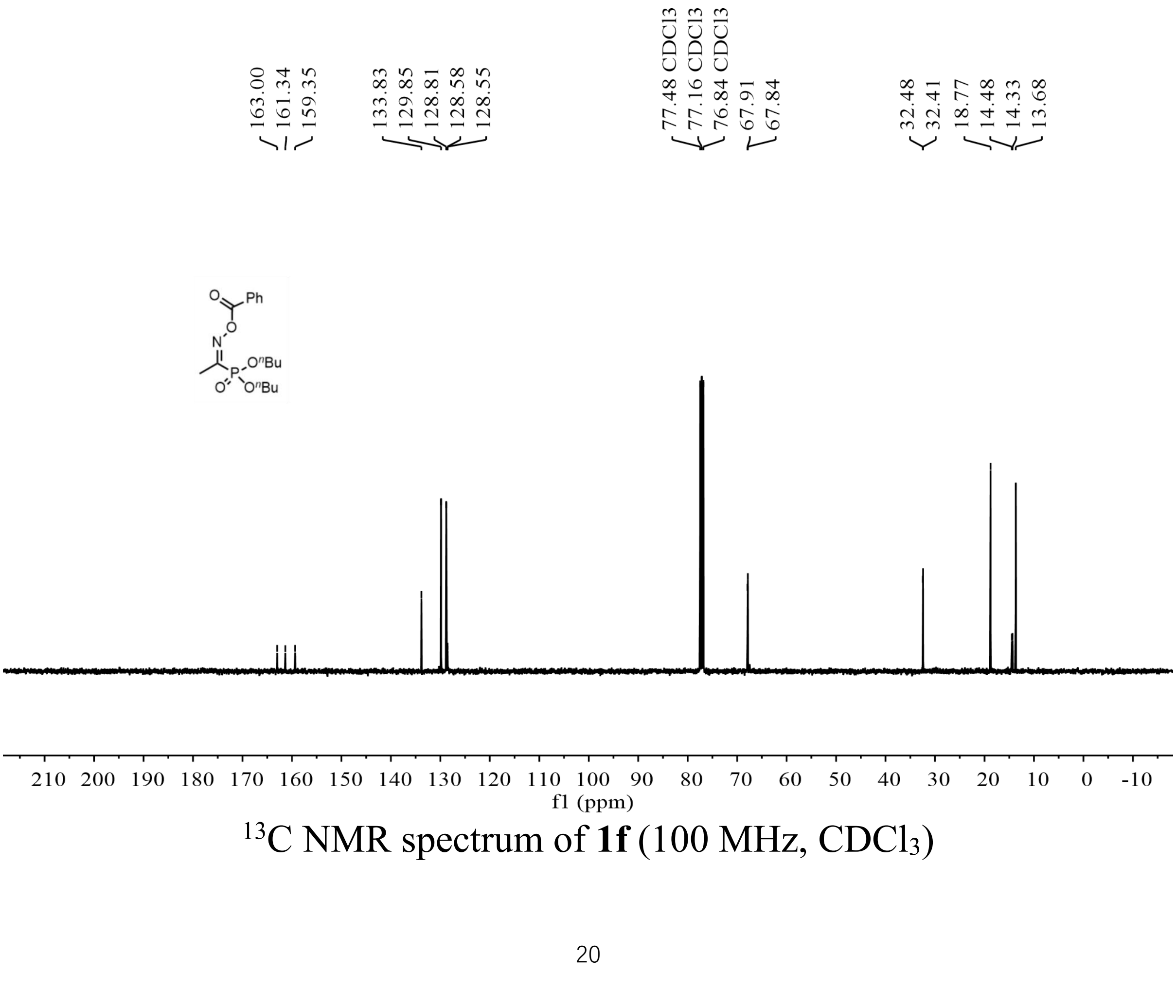
^{13}C NMR spectrum of **1e** (100 MHz, $CDCl_3$)



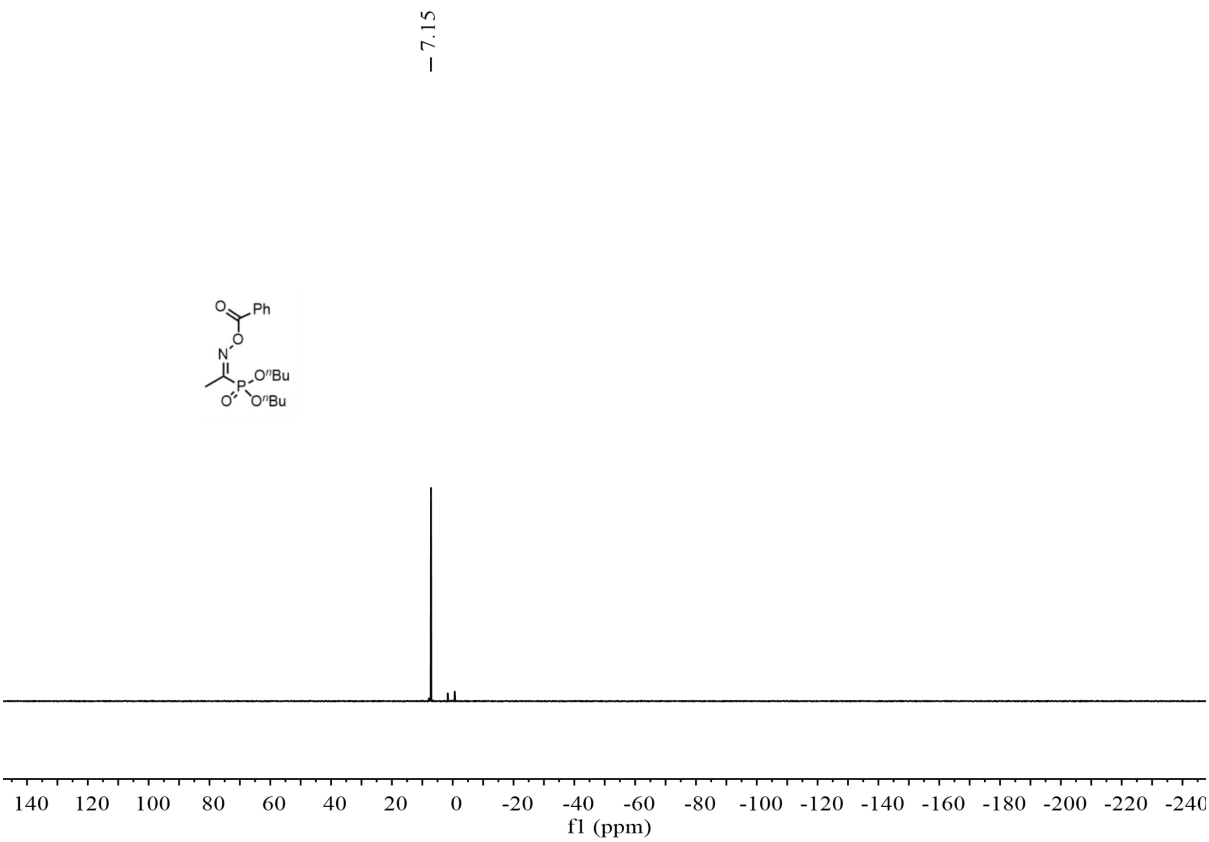
^{31}P NMR spectrum of **1e** (160 MHz, $CDCl_3$)



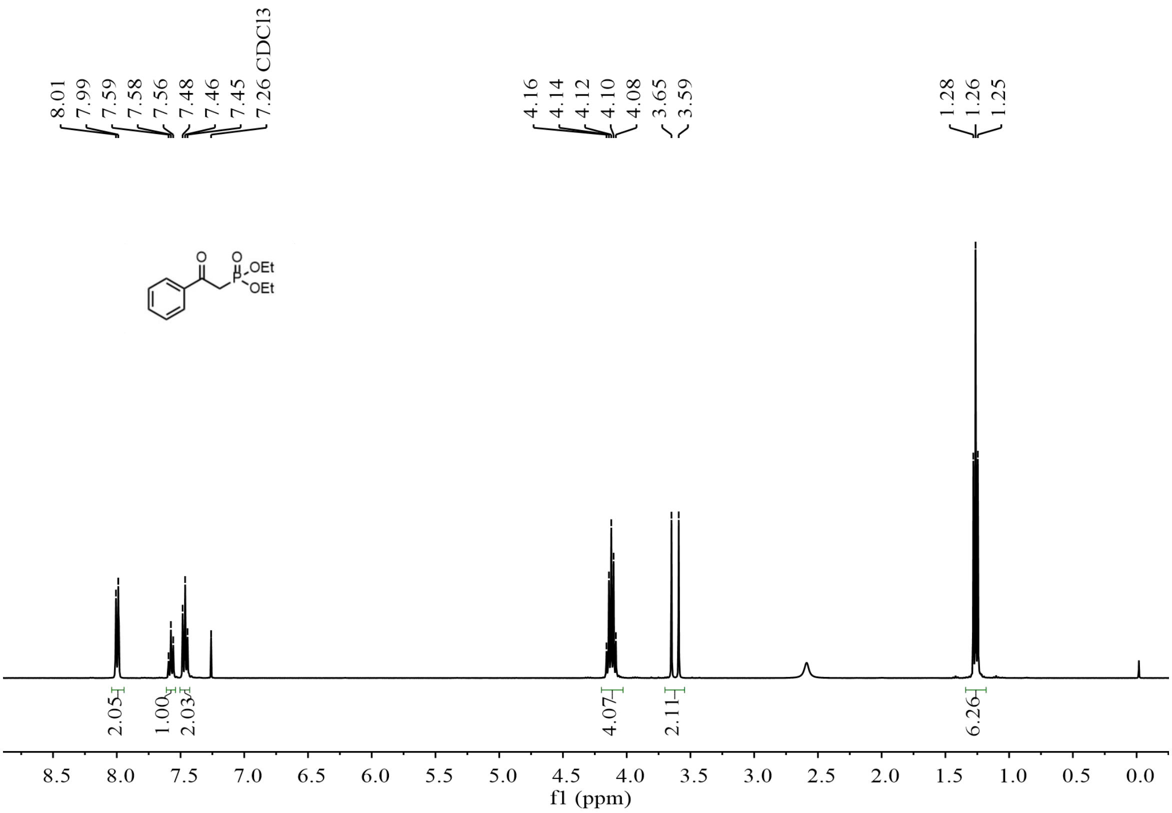
^{1}H NMR spectrum of **1f** (400 MHz, $CDCl_3$)



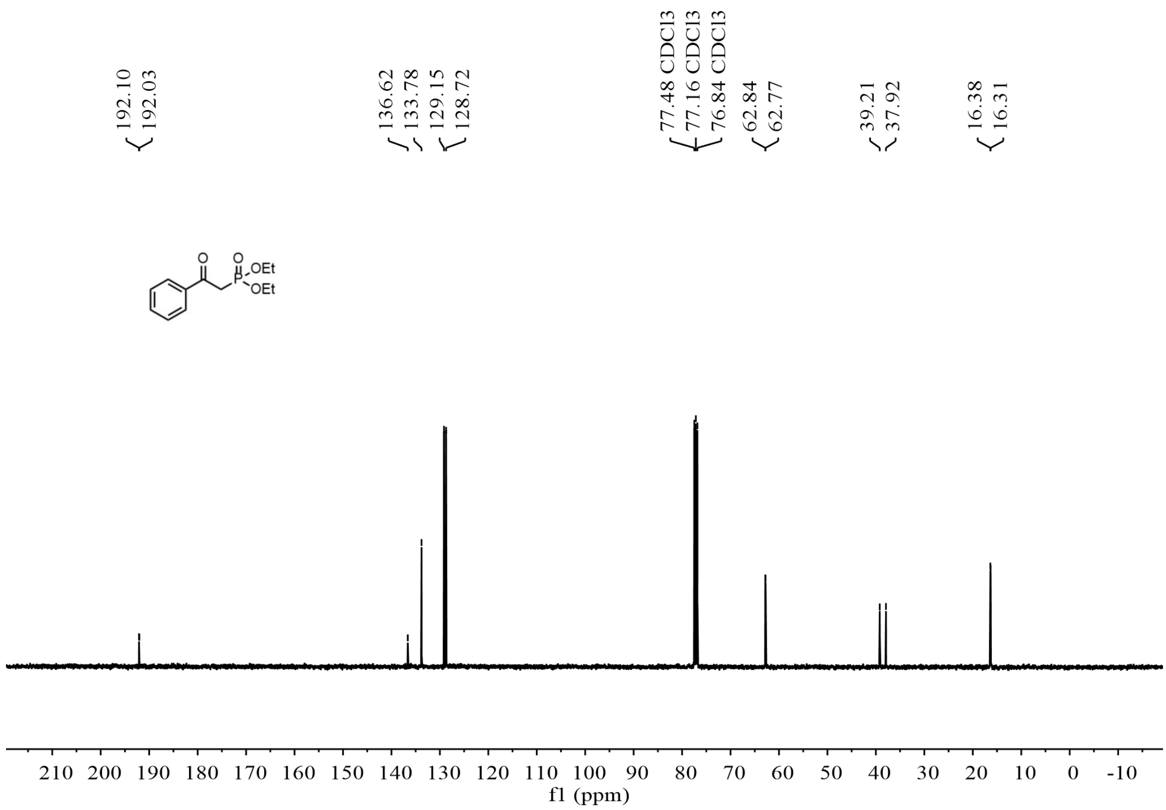
^{13}C NMR spectrum of **1f** (100 MHz, $CDCl_3$)



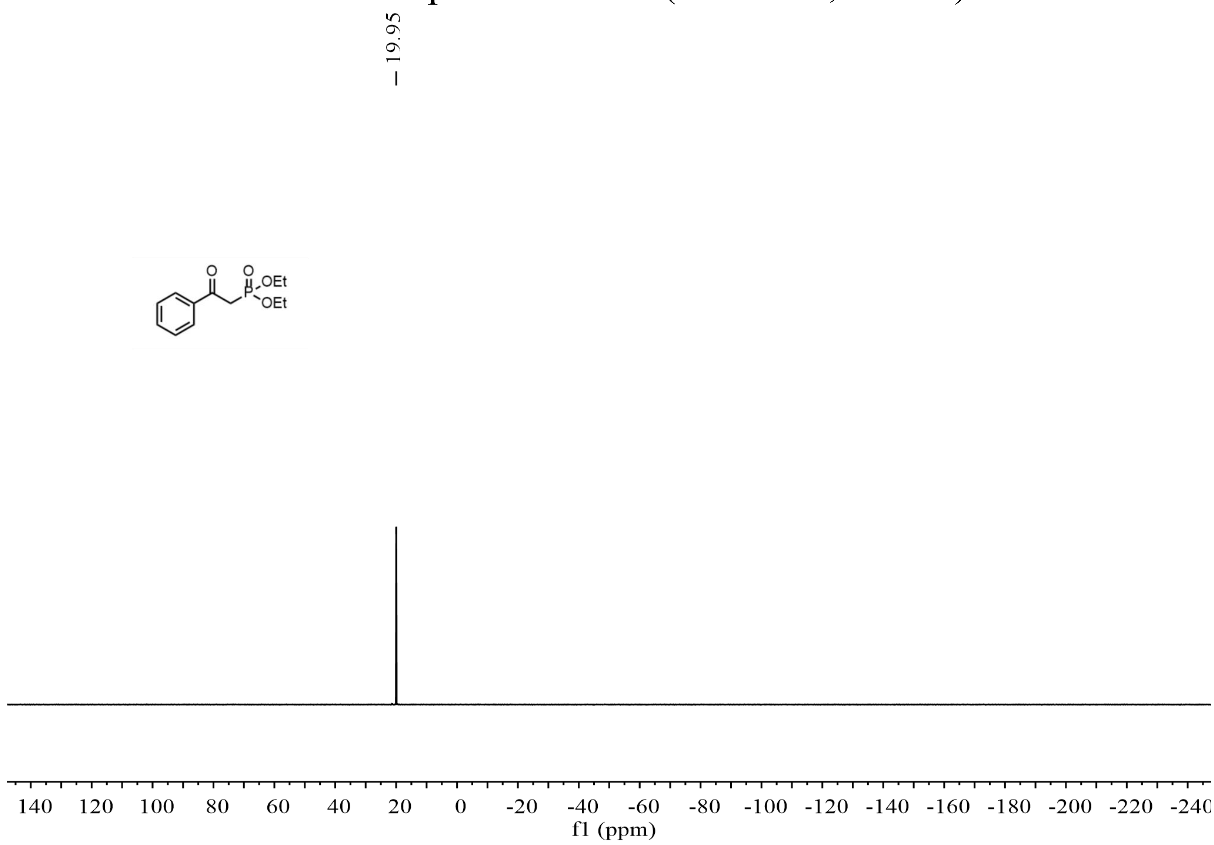
^{31}P NMR spectrum of **1f** (160 MHz, $CDCl_3$)



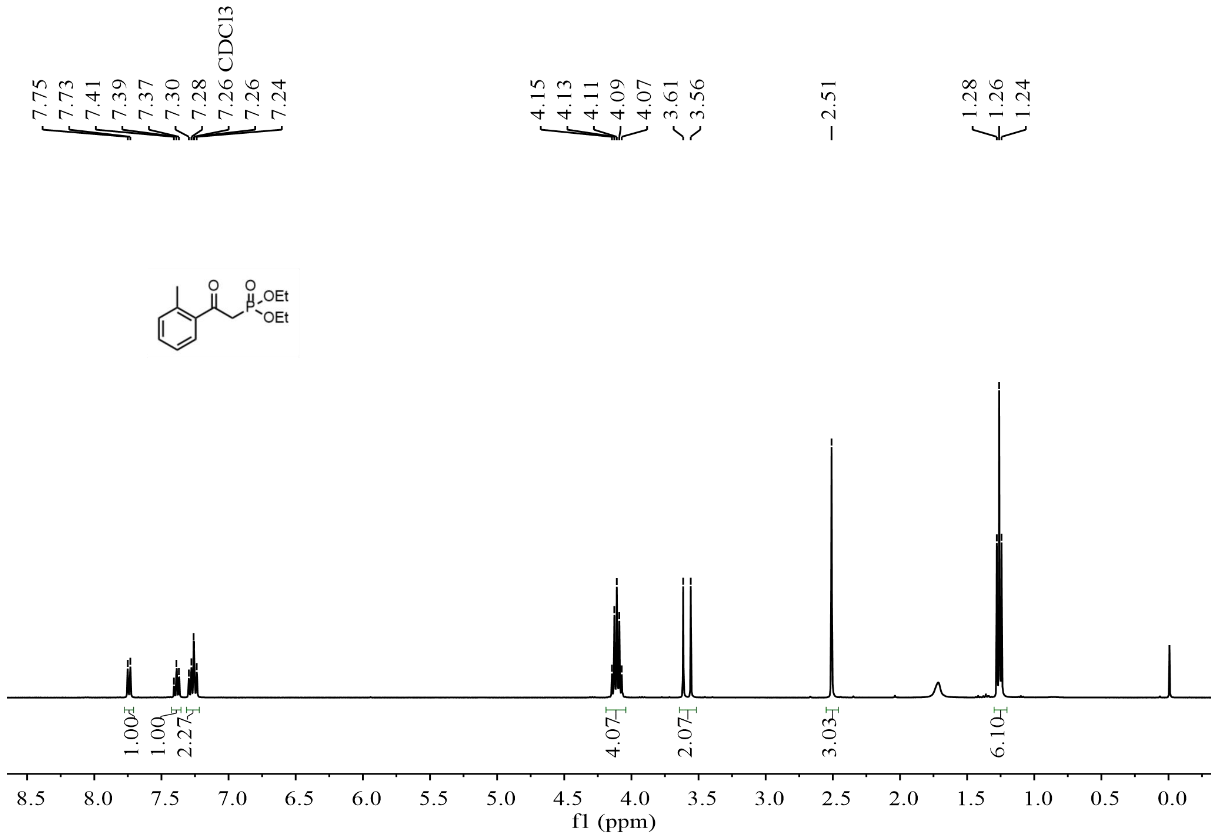
^{1}H NMR spectrum of **3a** (400 MHz, $CDCl_3$)



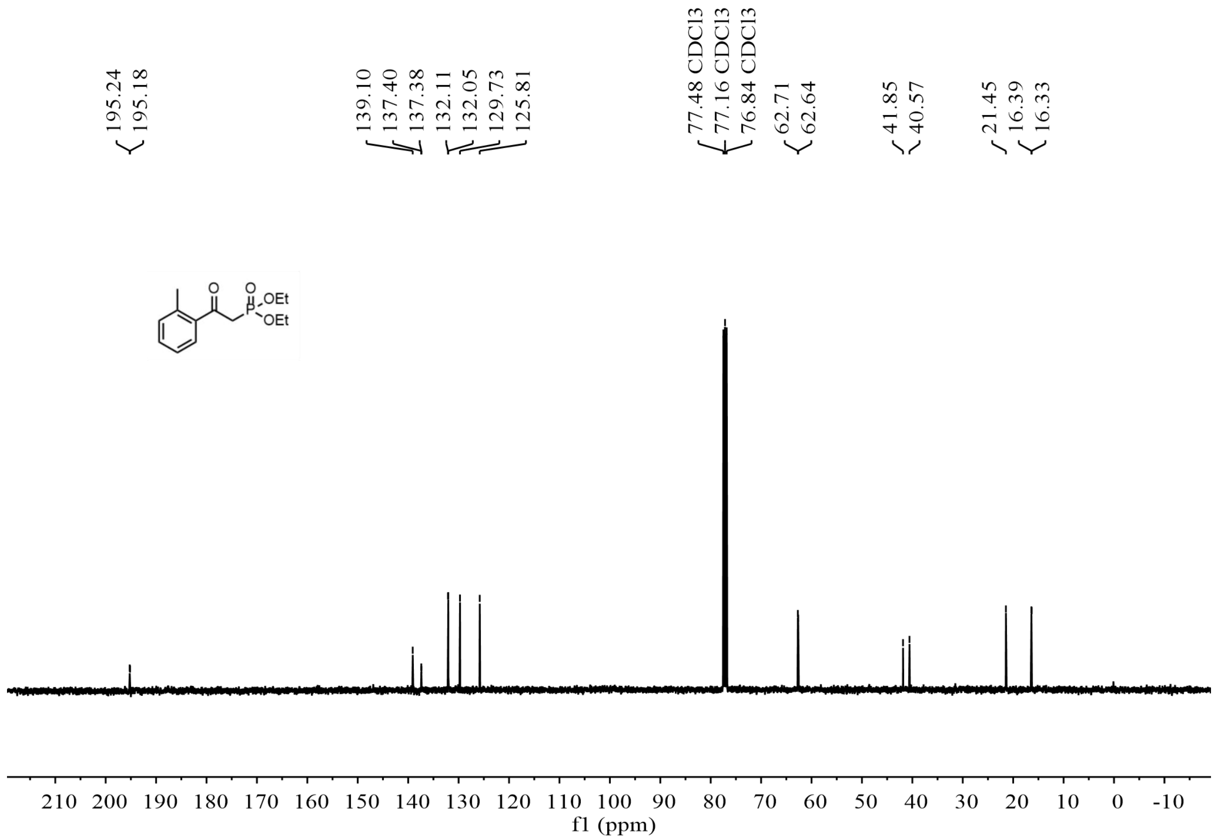
^{13}C NMR spectrum of **3a** (100 MHz, $CDCl_3$)



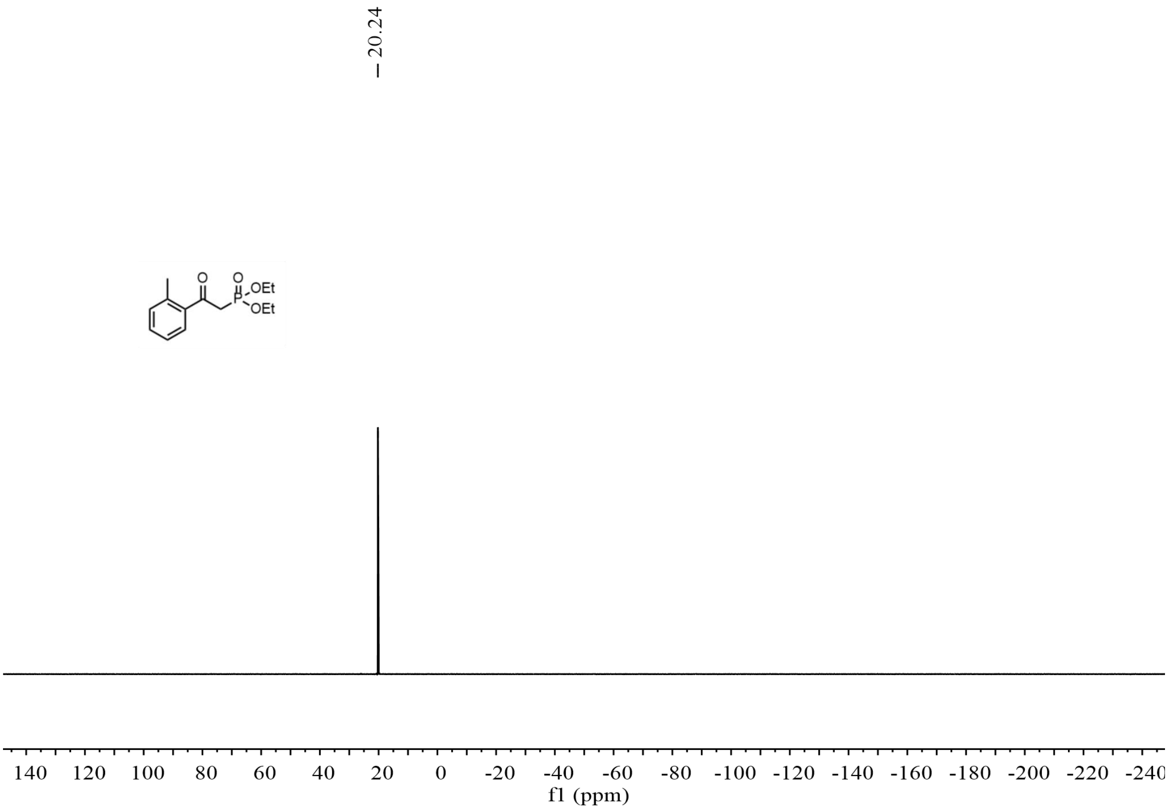
^{31}P NMR spectrum of **3a** (160 MHz, $CDCl_3$)



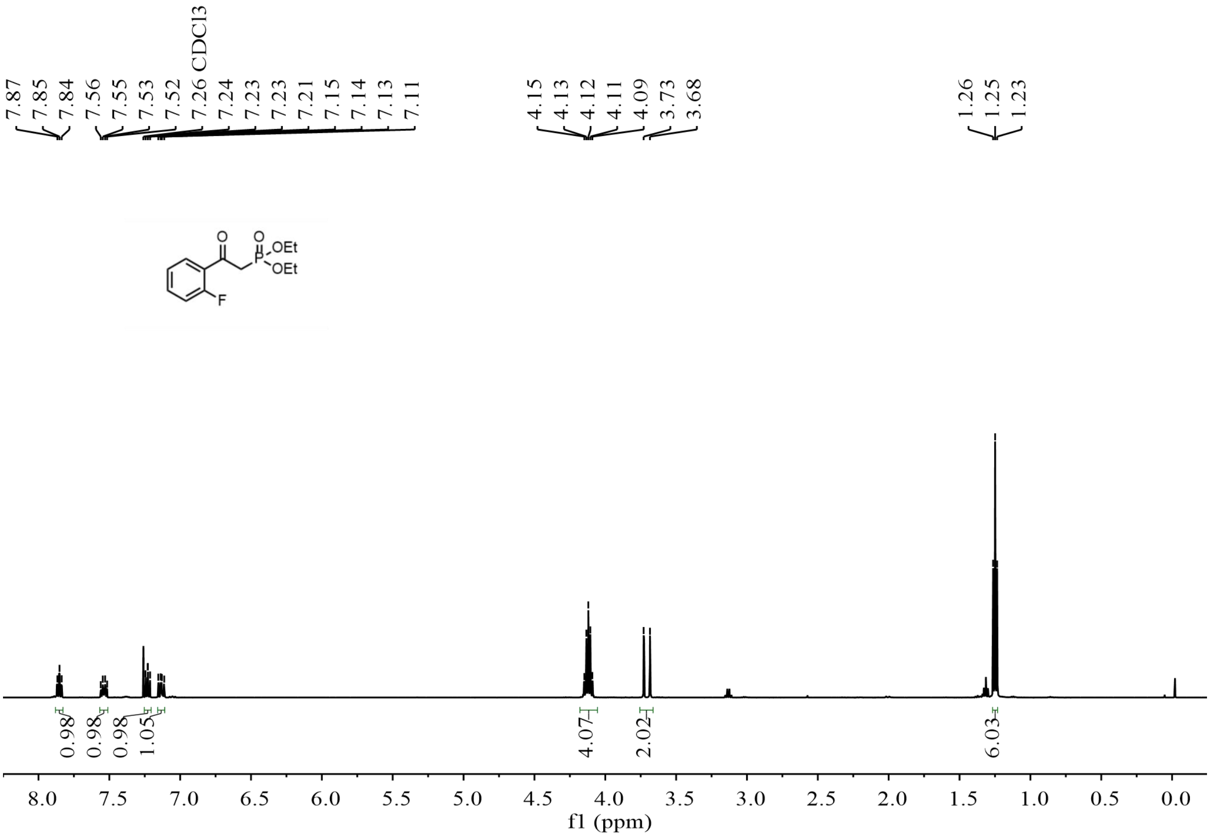
[1]H NMR spectrum of **3b** (400 MHz, $CDCl_3$)



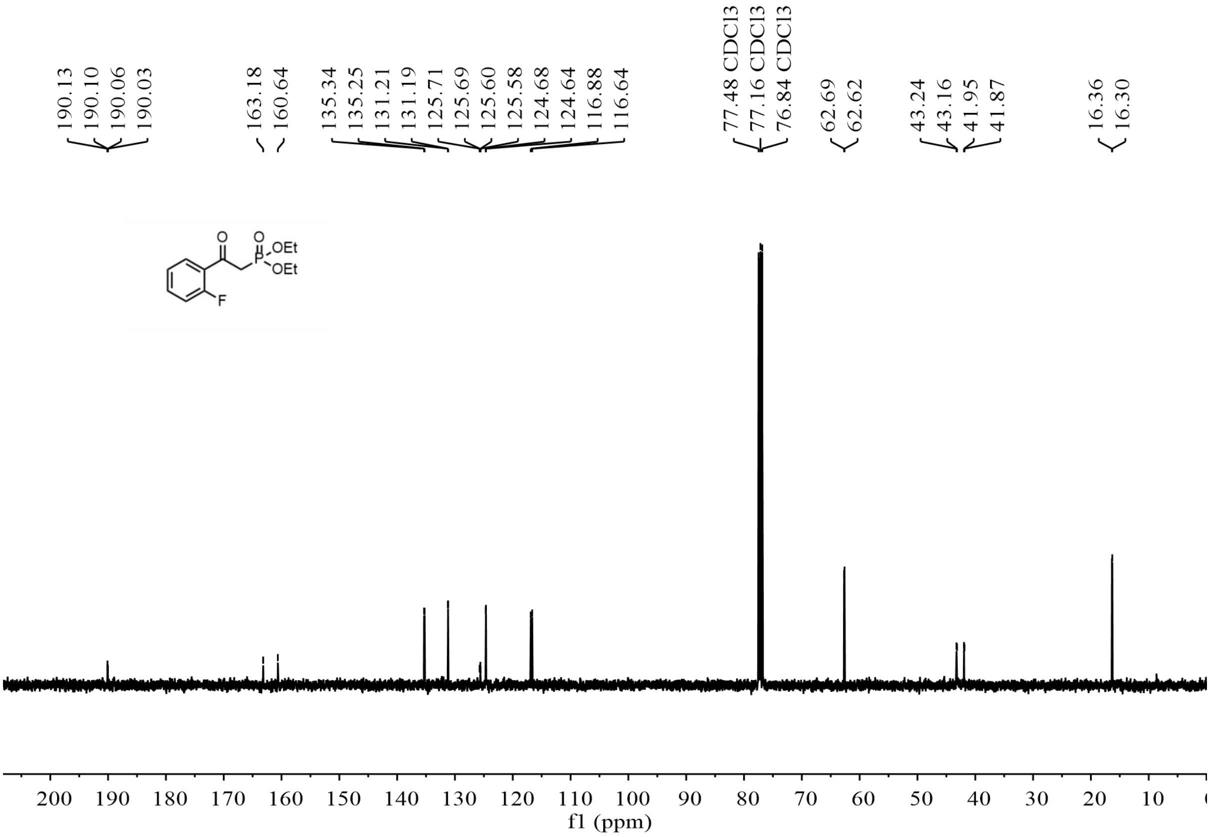
[13]C NMR spectrum of **3b** (100 MHz, $CDCl_3$)



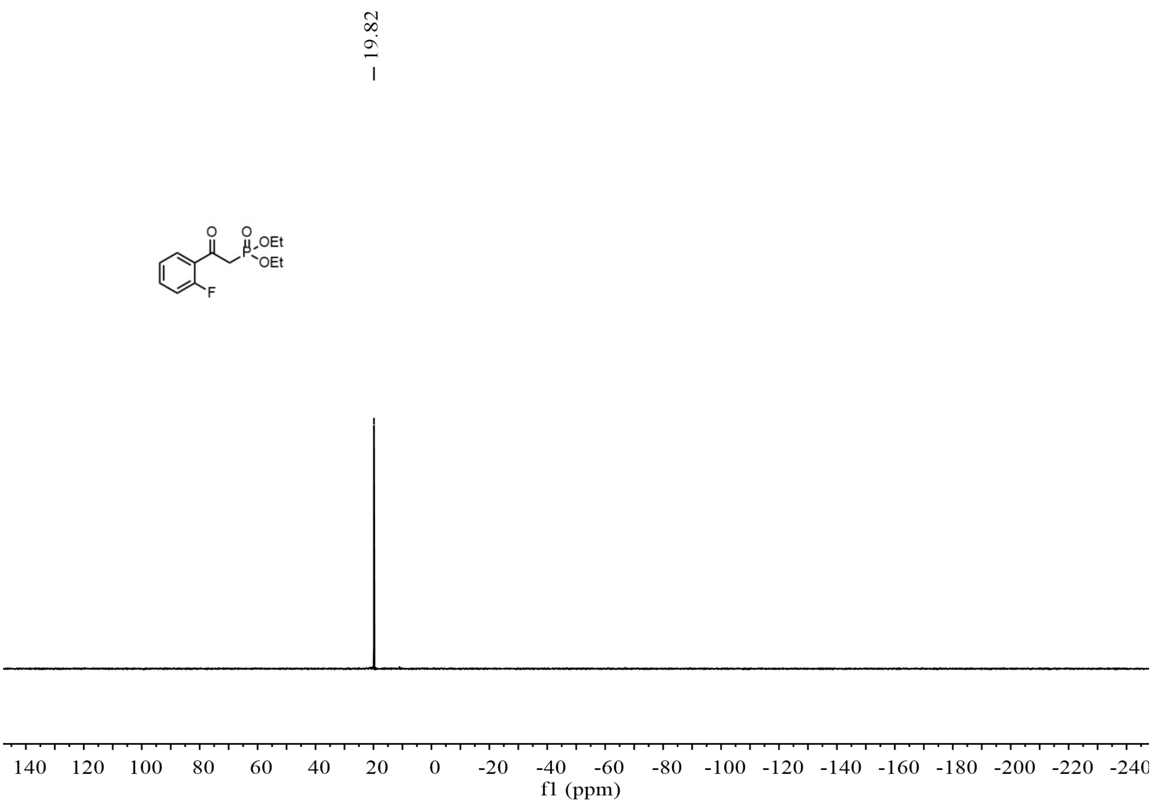
^{31}P NMR spectrum of **3b** (160 MHz, $CDCl_3$)



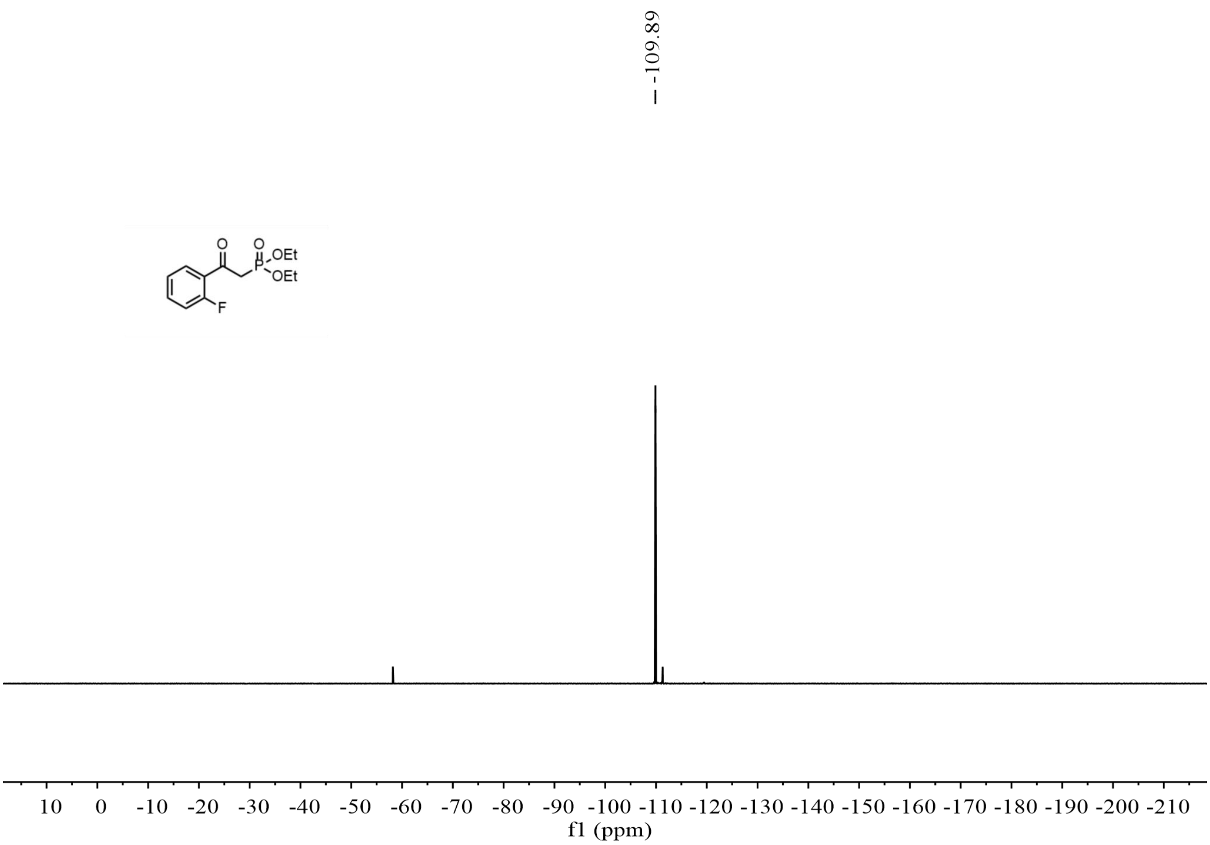
^{1}H NMR spectrum of **3c** (500 MHz, $CDCl_3$)



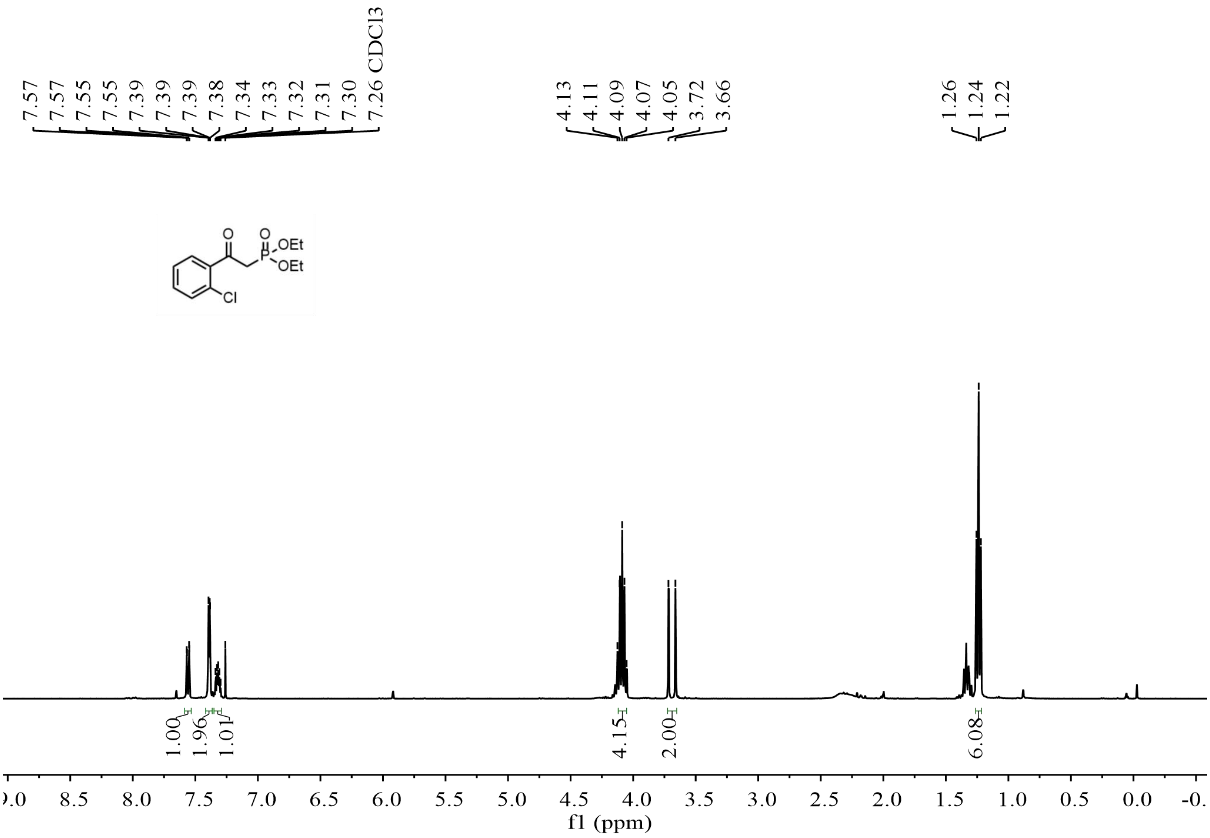
^{13}C NMR spectrum of **3c** (100 MHz, $CDCl_3$)



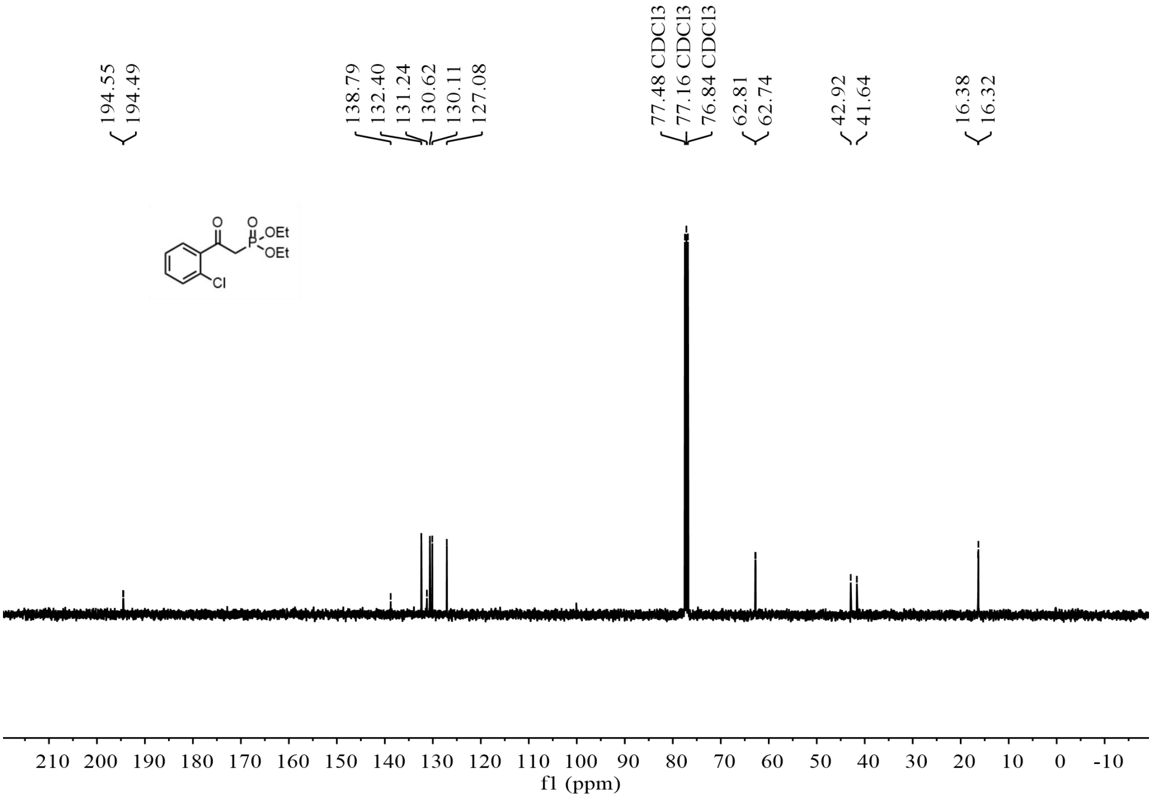
^{31}P NMR spectrum of **3c** (160 MHz, $CDCl_3$)



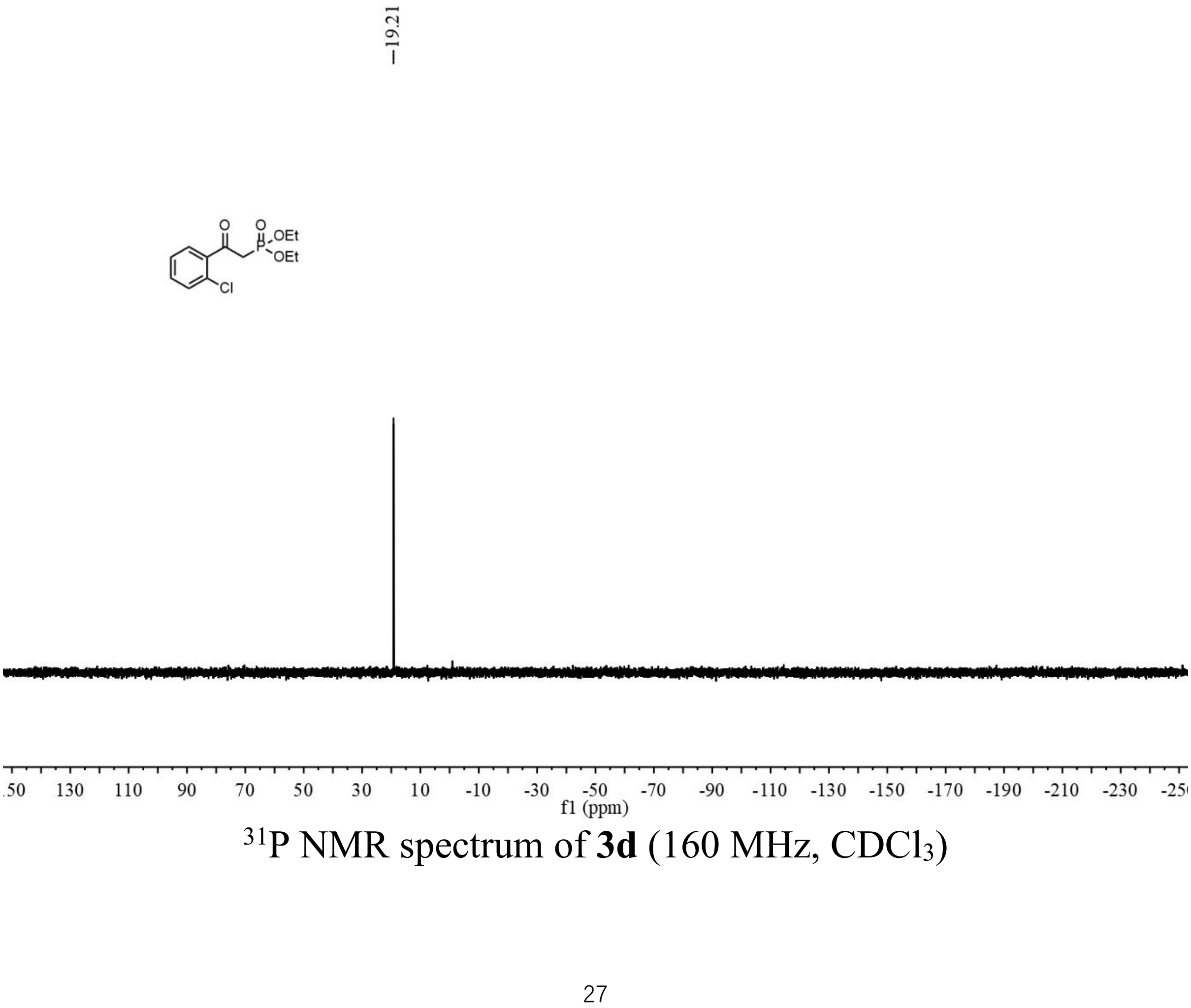
^{19}F NMR spectrum of **3c** (376 MHz, $CDCl_3$)



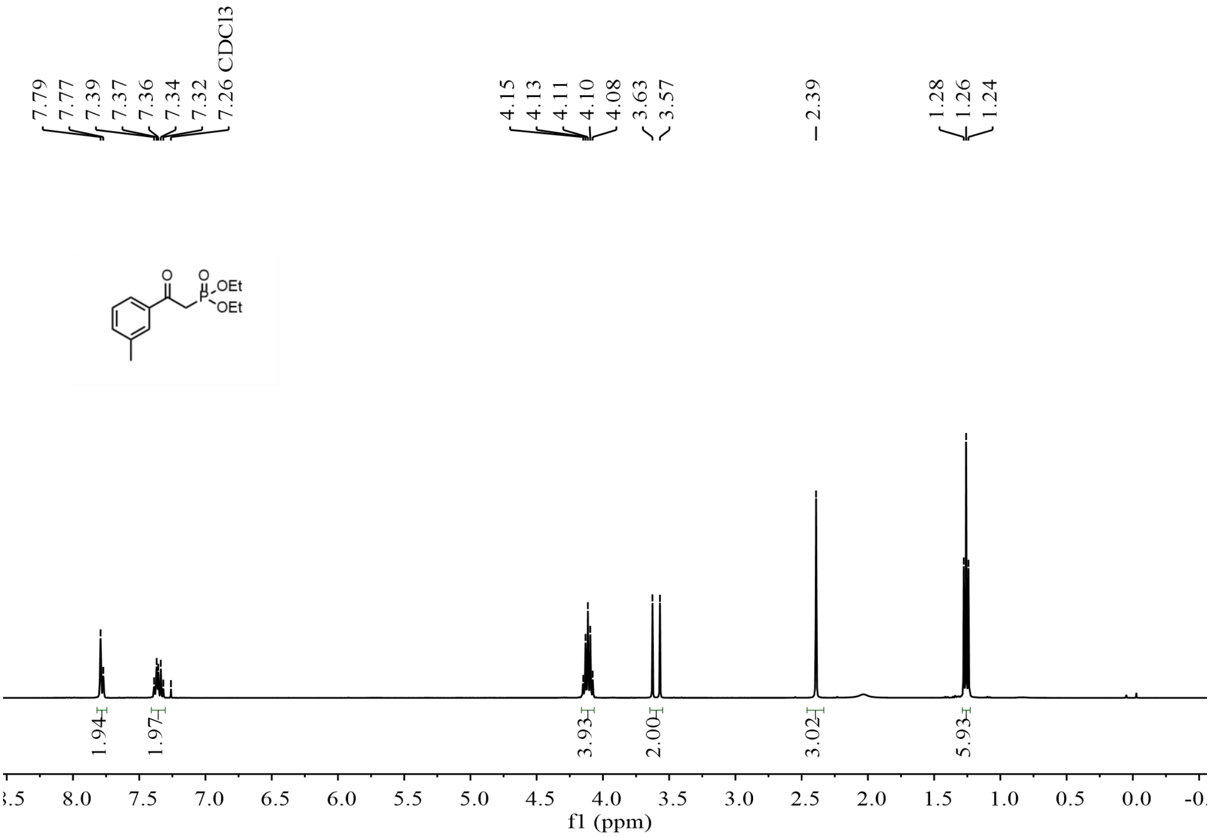
^{1}H NMR spectrum of **3d** (400 MHz, $CDCl_3$)



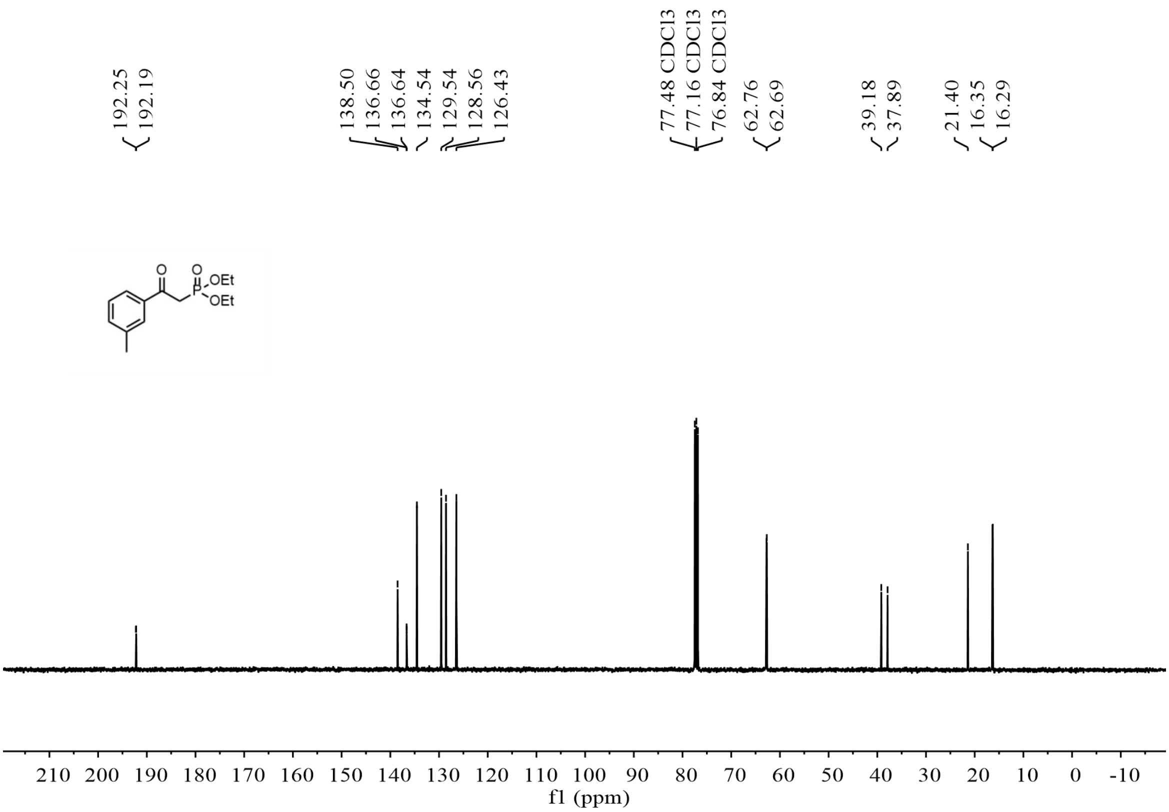
^{13}C NMR spectrum of **3d** (100 MHz, $CDCl_3$)



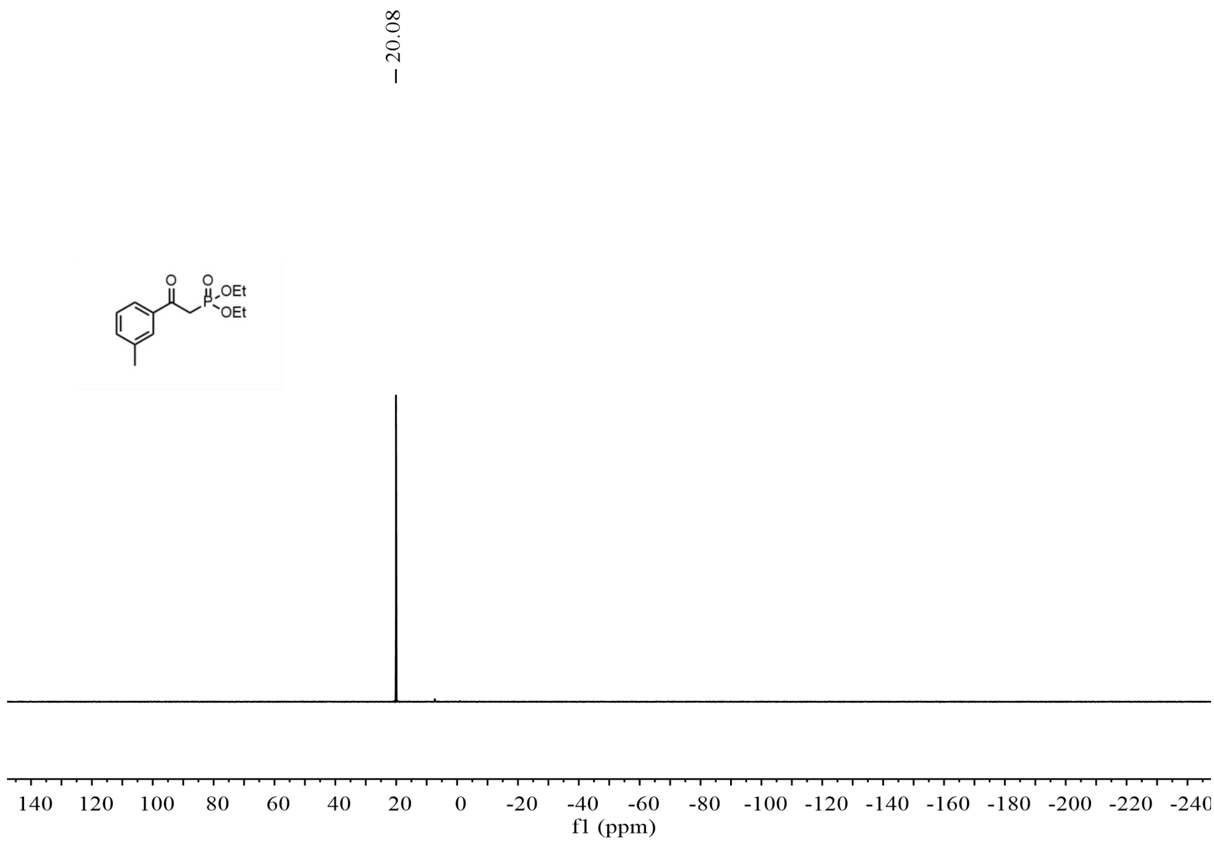
^{31}P NMR spectrum of **3d** (160 MHz, $CDCl_3$)



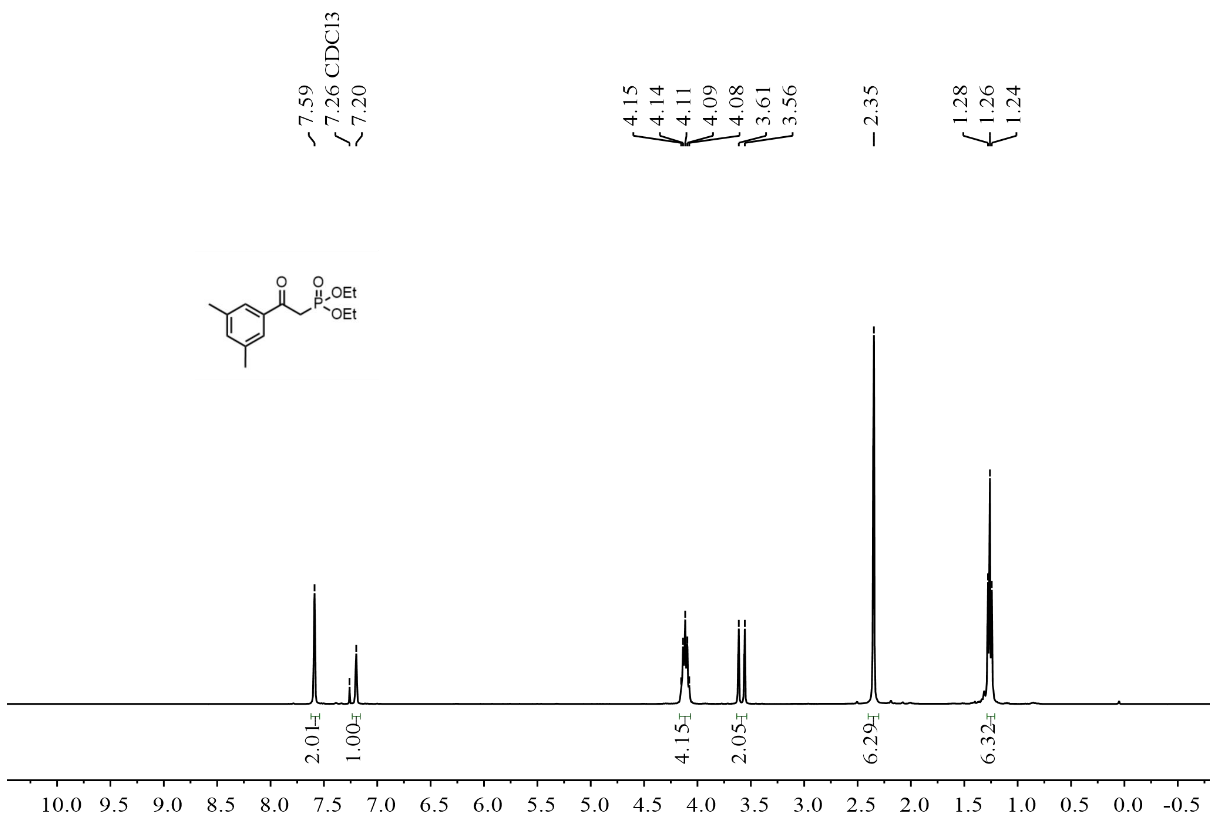
^{1}H NMR spectrum of **3e** (400 MHz, $CDCl_3$)



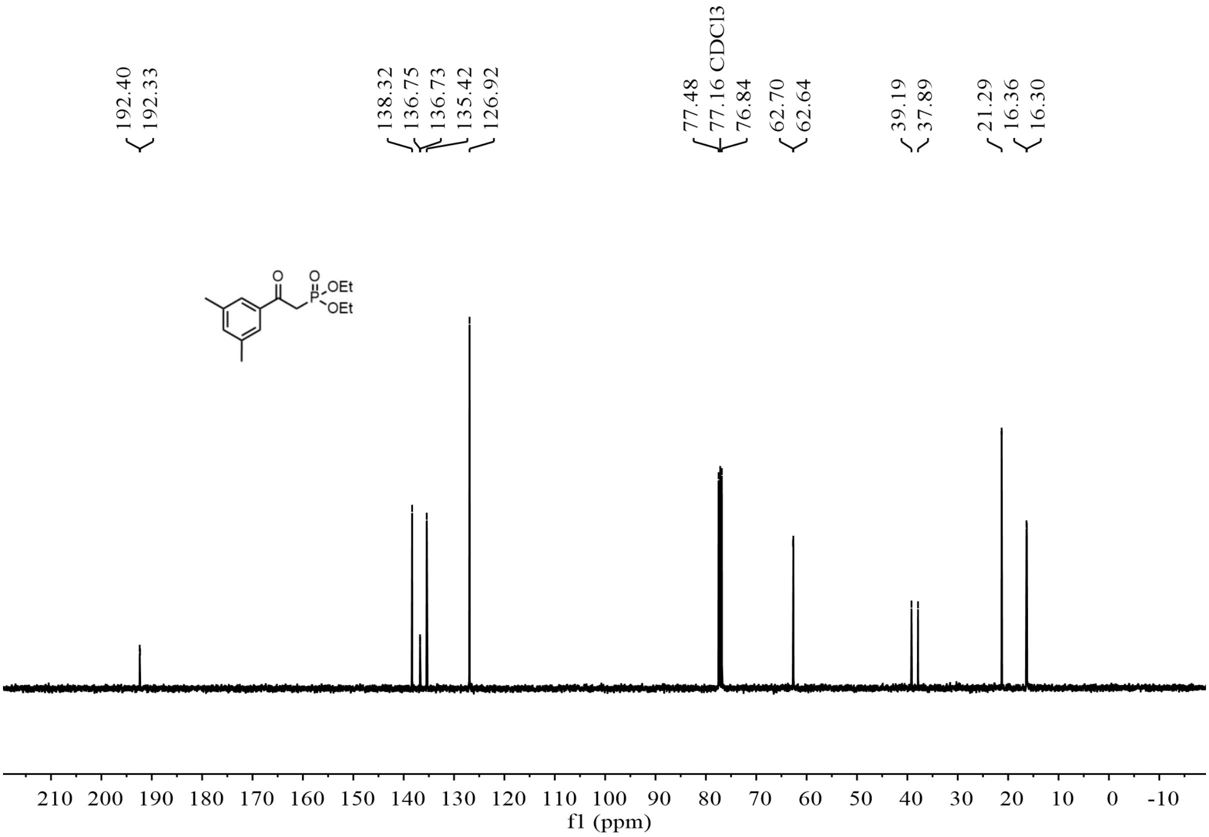
^{13}C NMR spectrum of **3e** (100 MHz, $CDCl_3$)



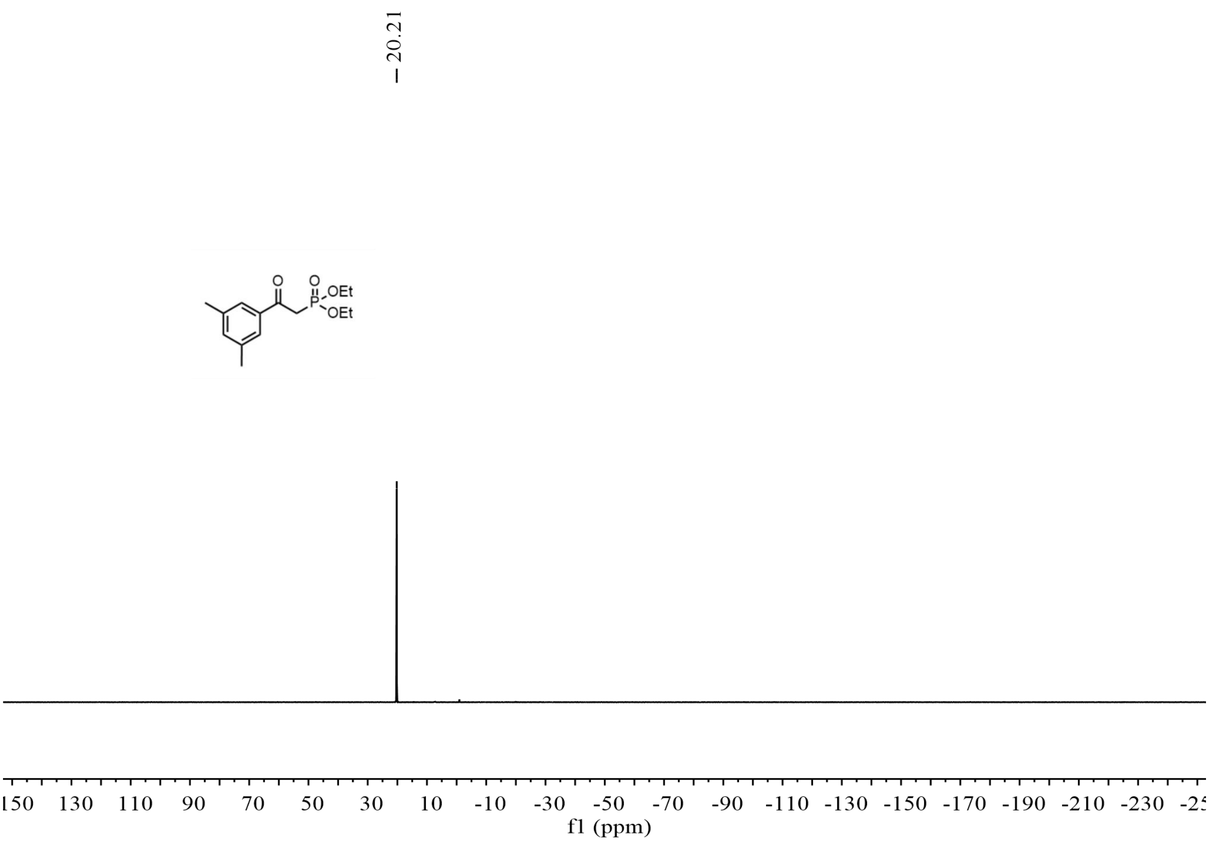
^{31}P NMR spectrum of **3e** (160 MHz, $CDCl_3$)



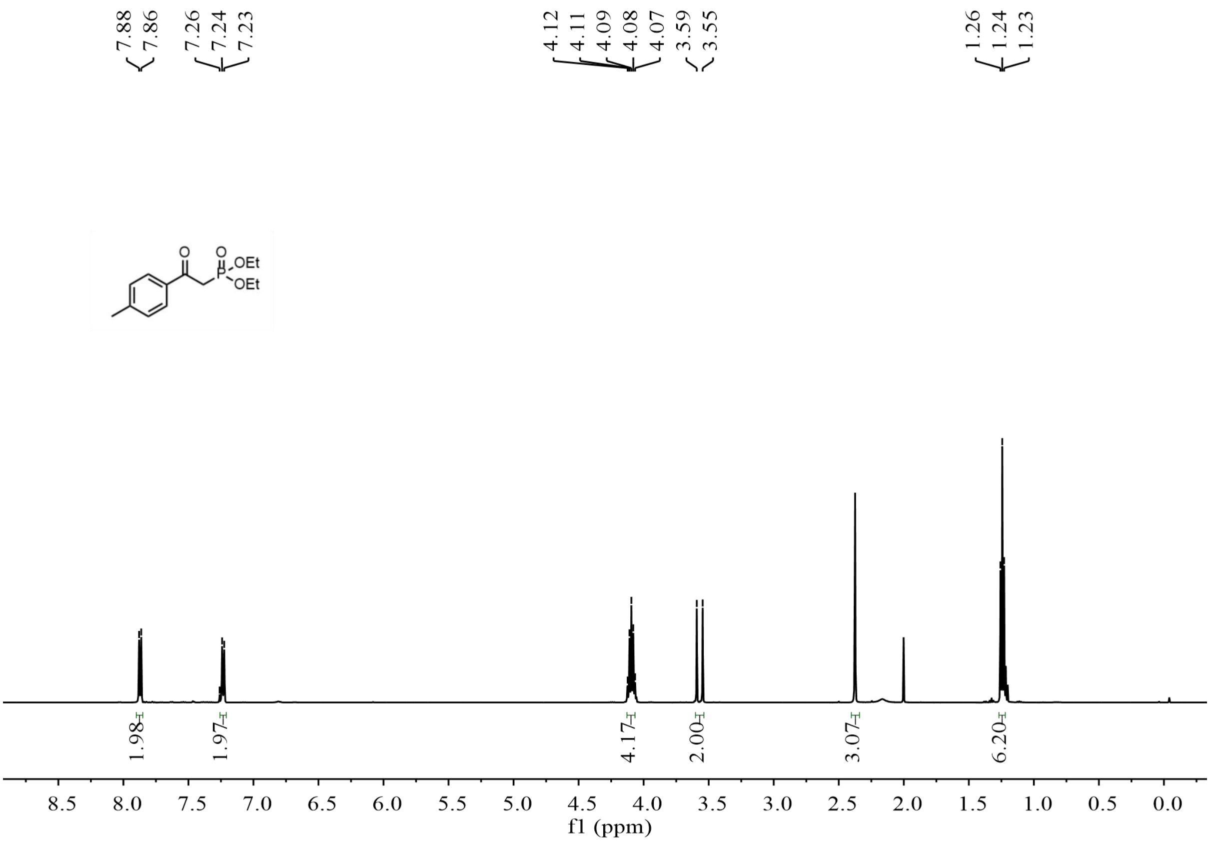
^{1}H NMR spectrum of **3f** (400 MHz, $CDCl_3$)



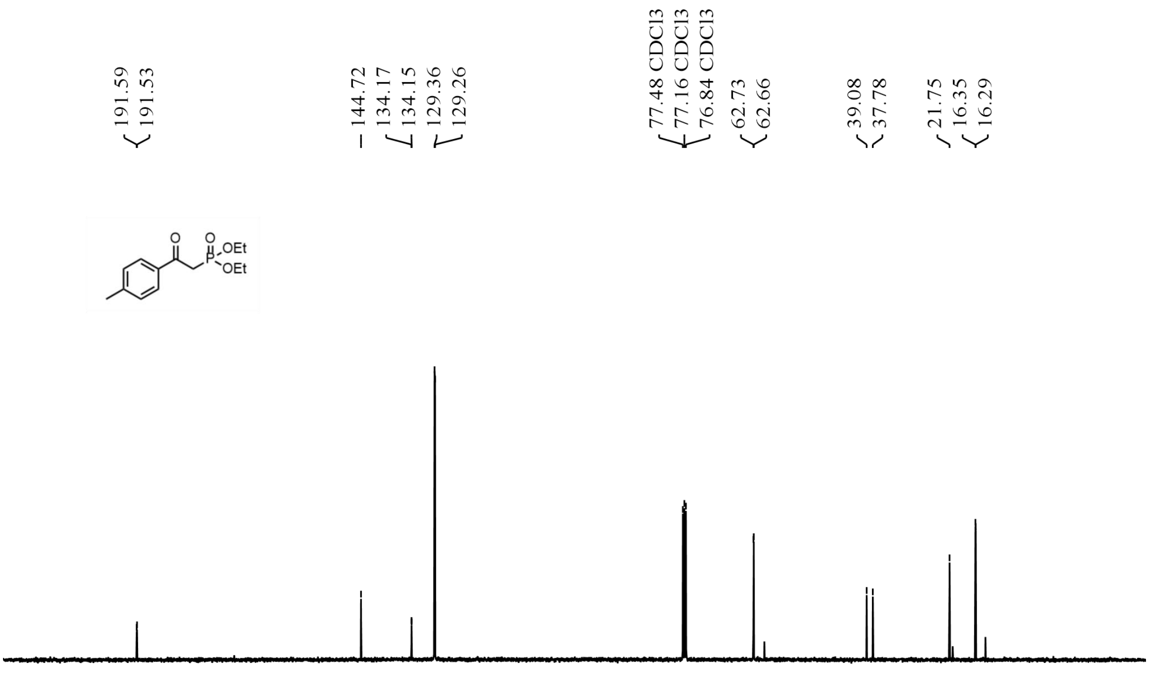
^{13}C NMR spectrum of **3f** (100 MHz, $CDCl_3$)



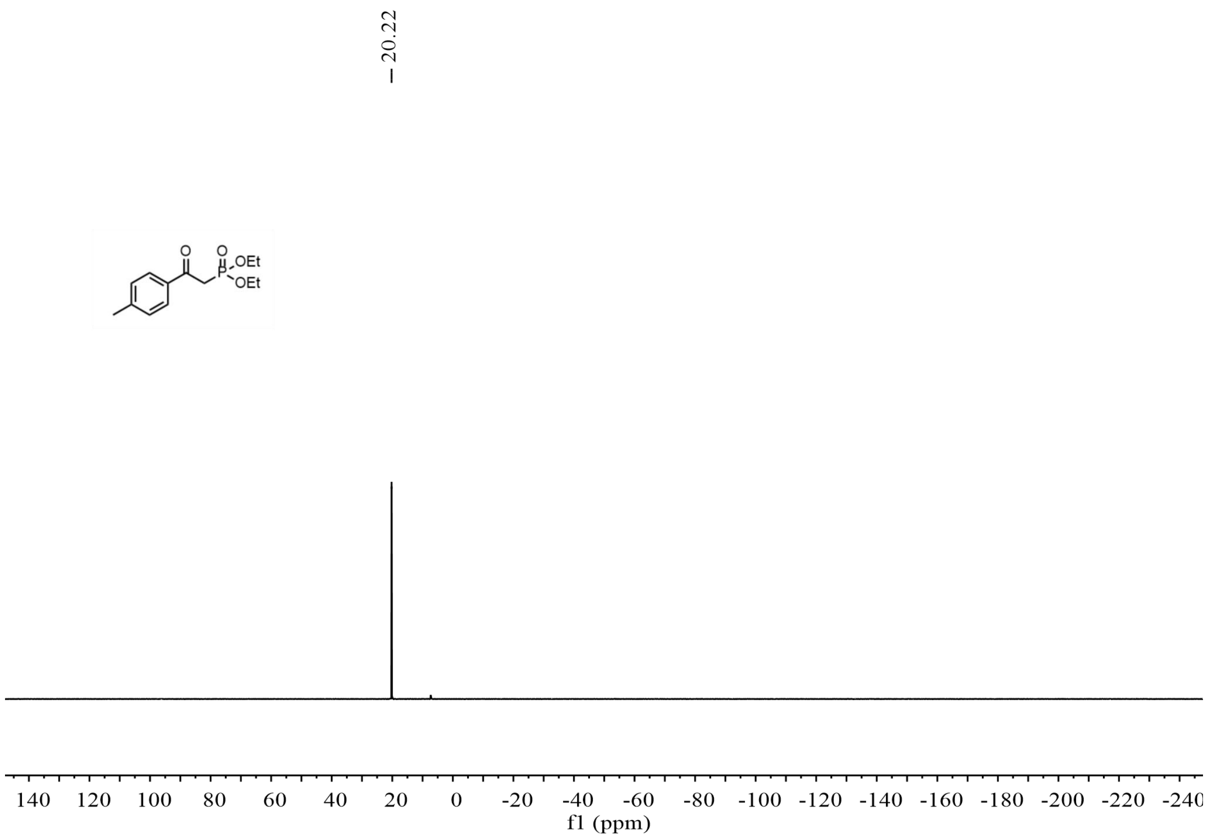
^{31}P NMR spectrum of **3f** (160 MHz, $CDCl_3$)



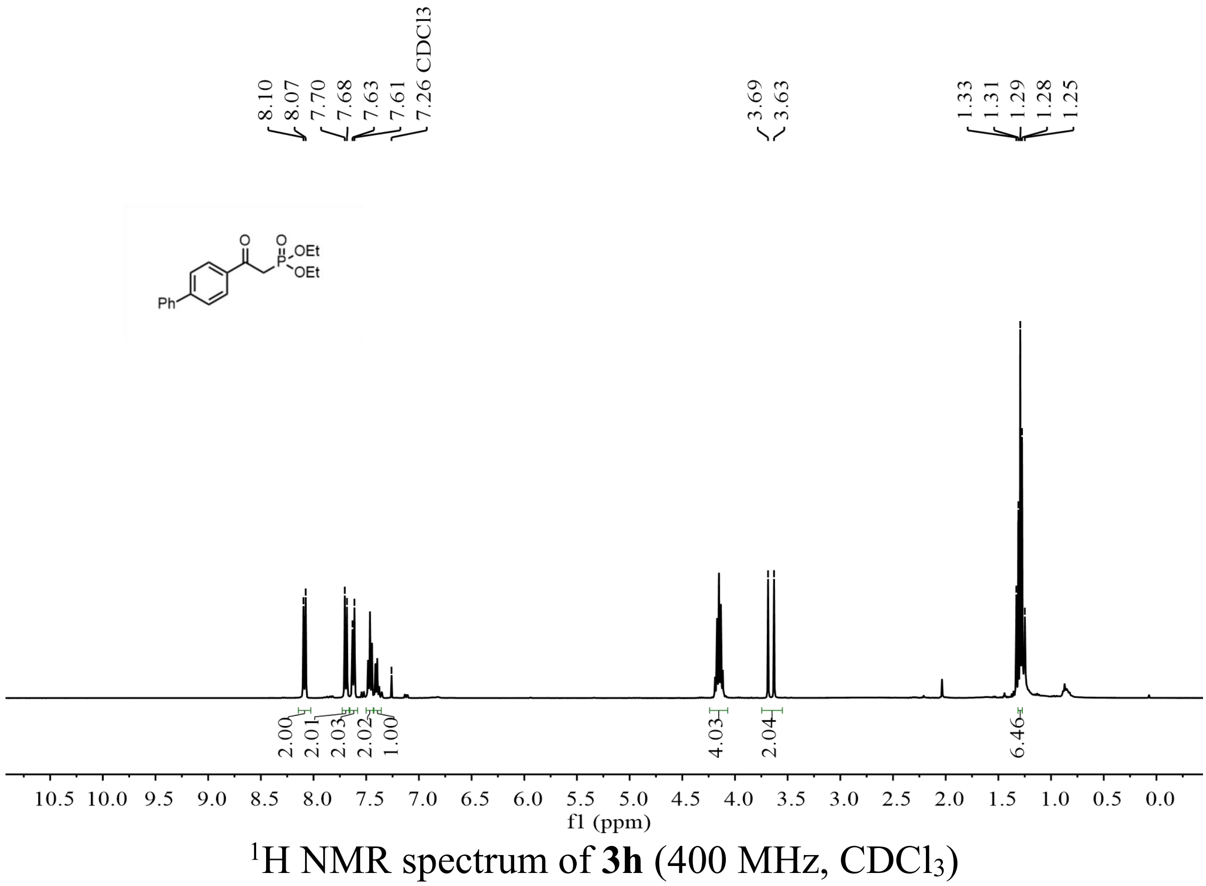
^{1}H NMR spectrum of **3g** (500 MHz, $CDCl_3$)



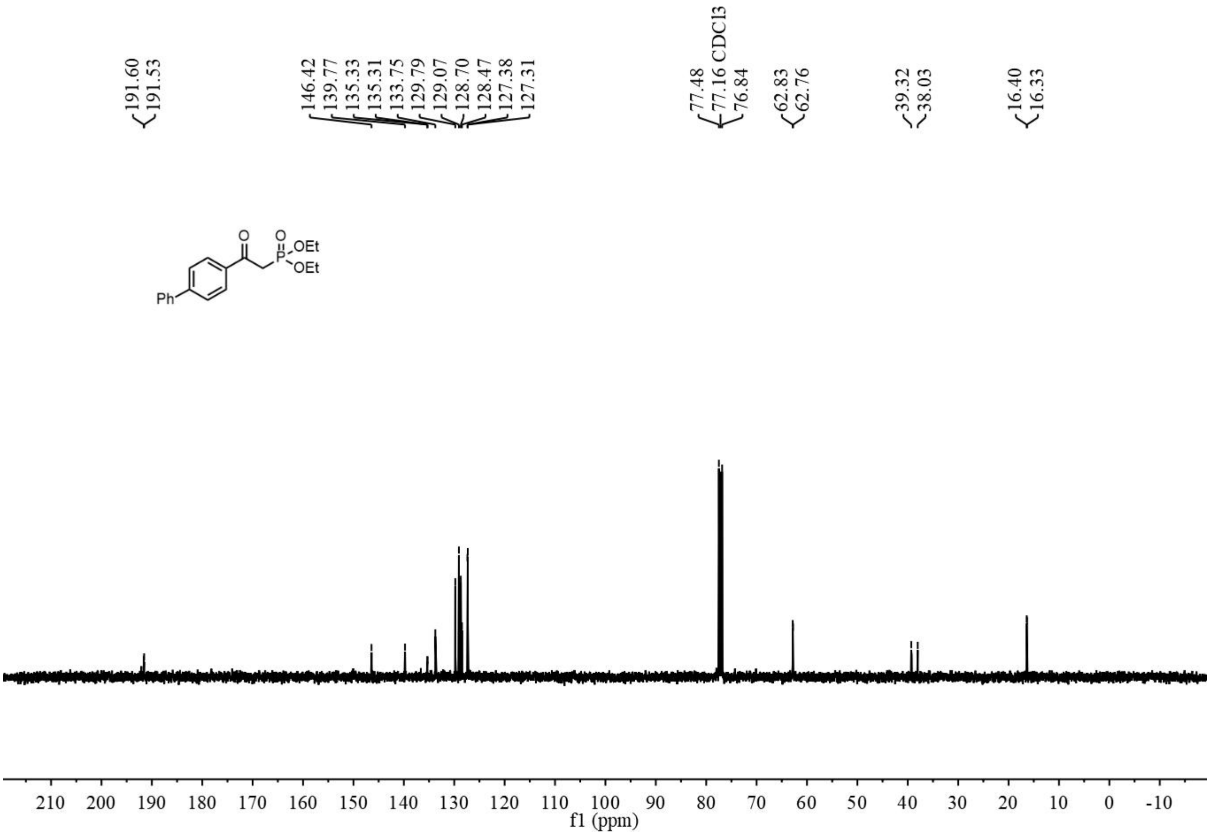
^{13}C NMR spectrum of **3g** (100 MHz, $CDCl_3$)



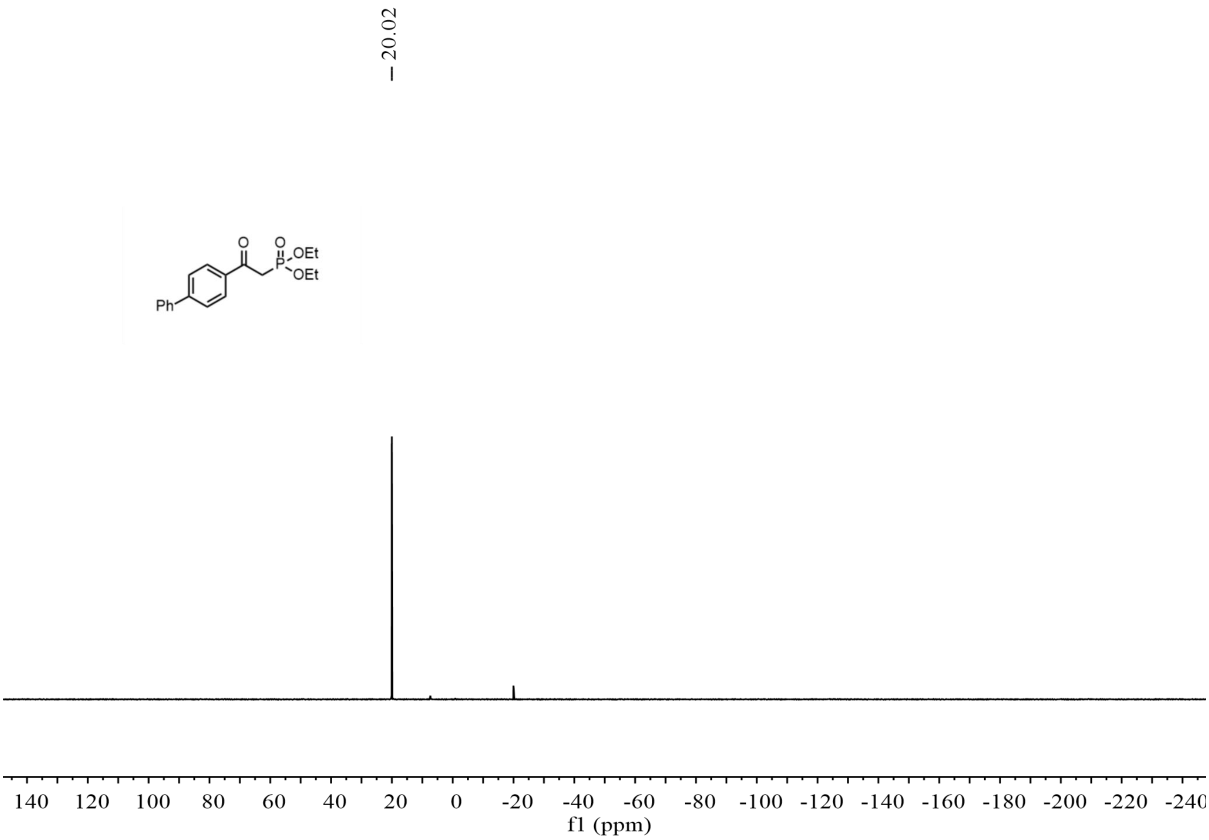
^{31}P NMR spectrum of **3g** (160 MHz, $CDCl_3$)



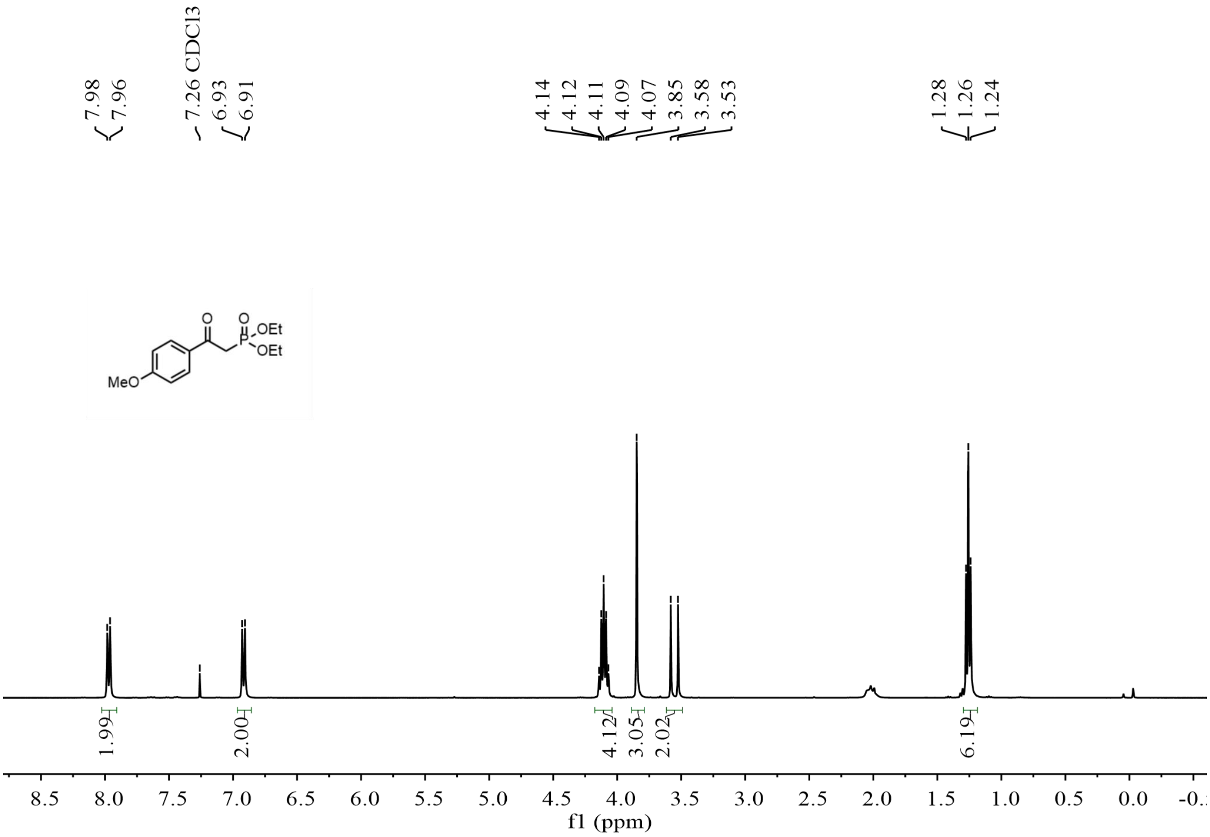
^{1}H NMR spectrum of **3h** (400 MHz, $CDCl_3$)



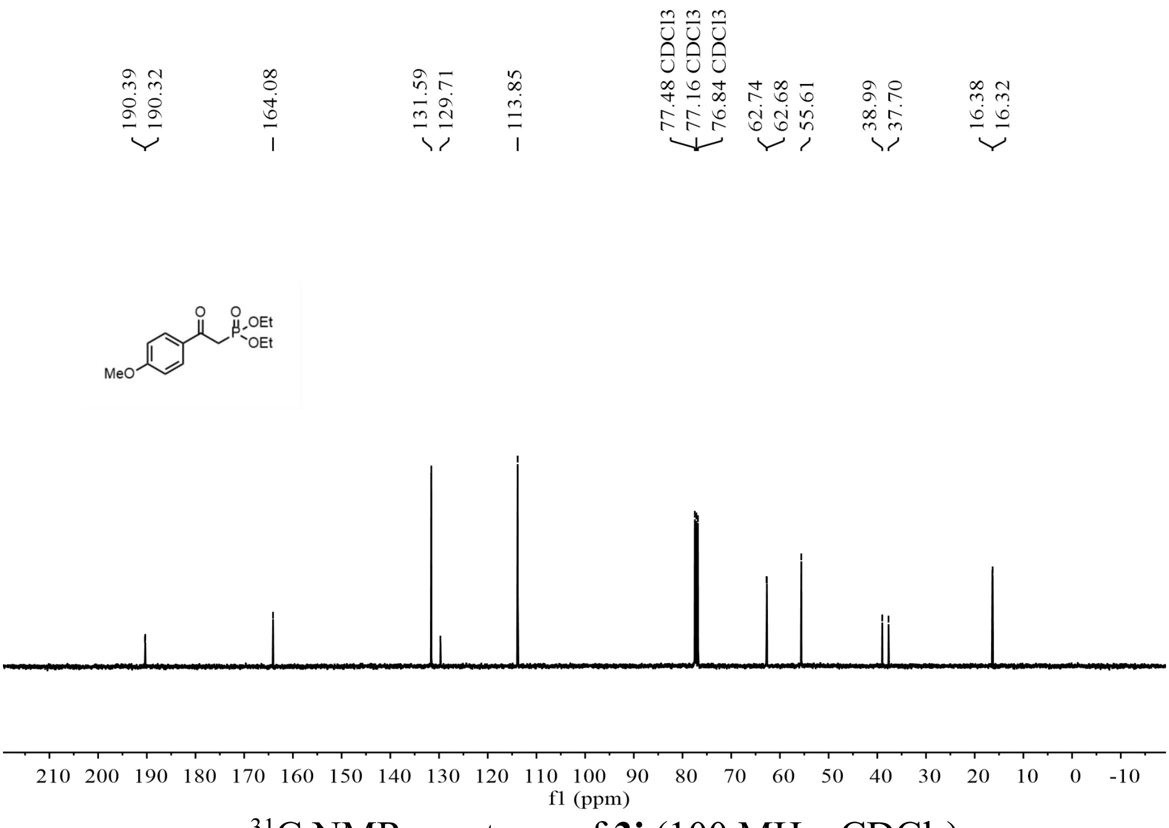
^{13}C NMR spectrum of **3h** (100 MHz, $CDCl_3$)



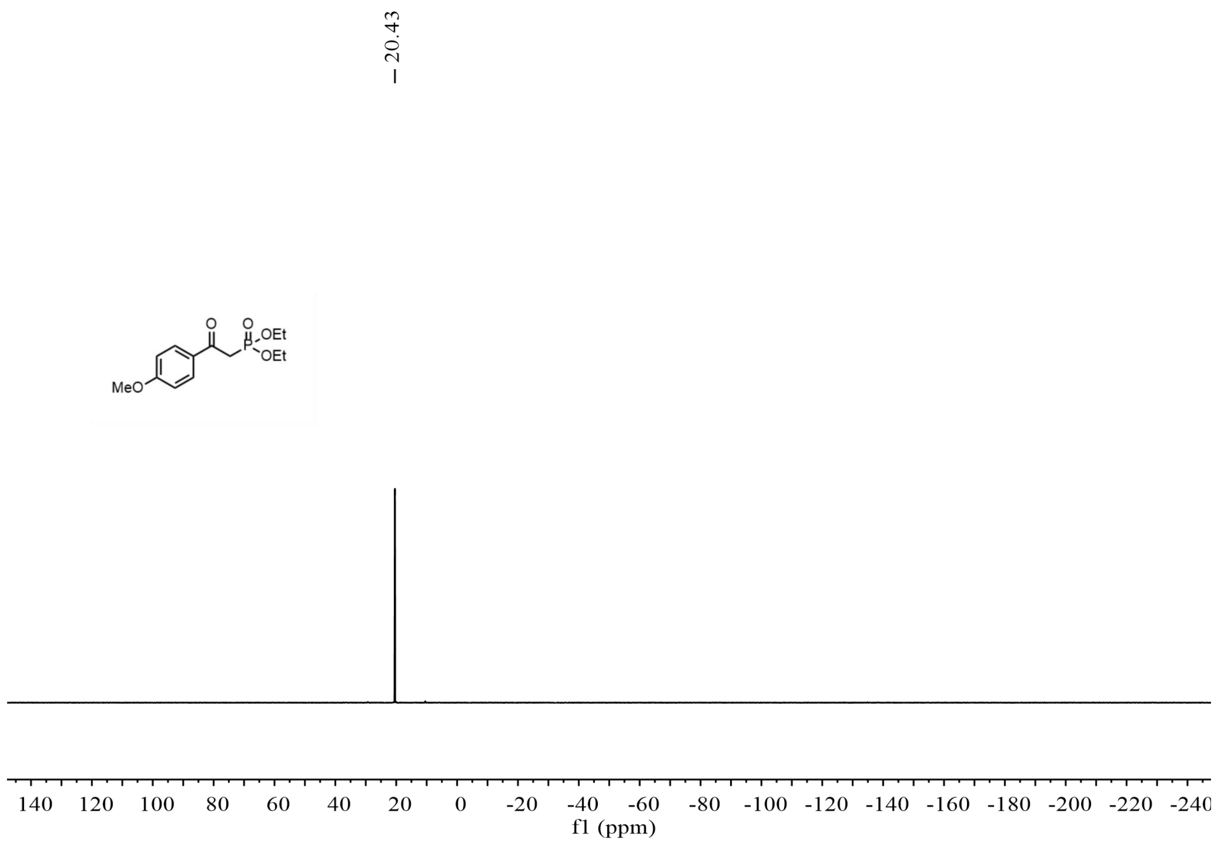
^{31}P NMR spectrum of **3h** (160 MHz, $CDCl_3$)



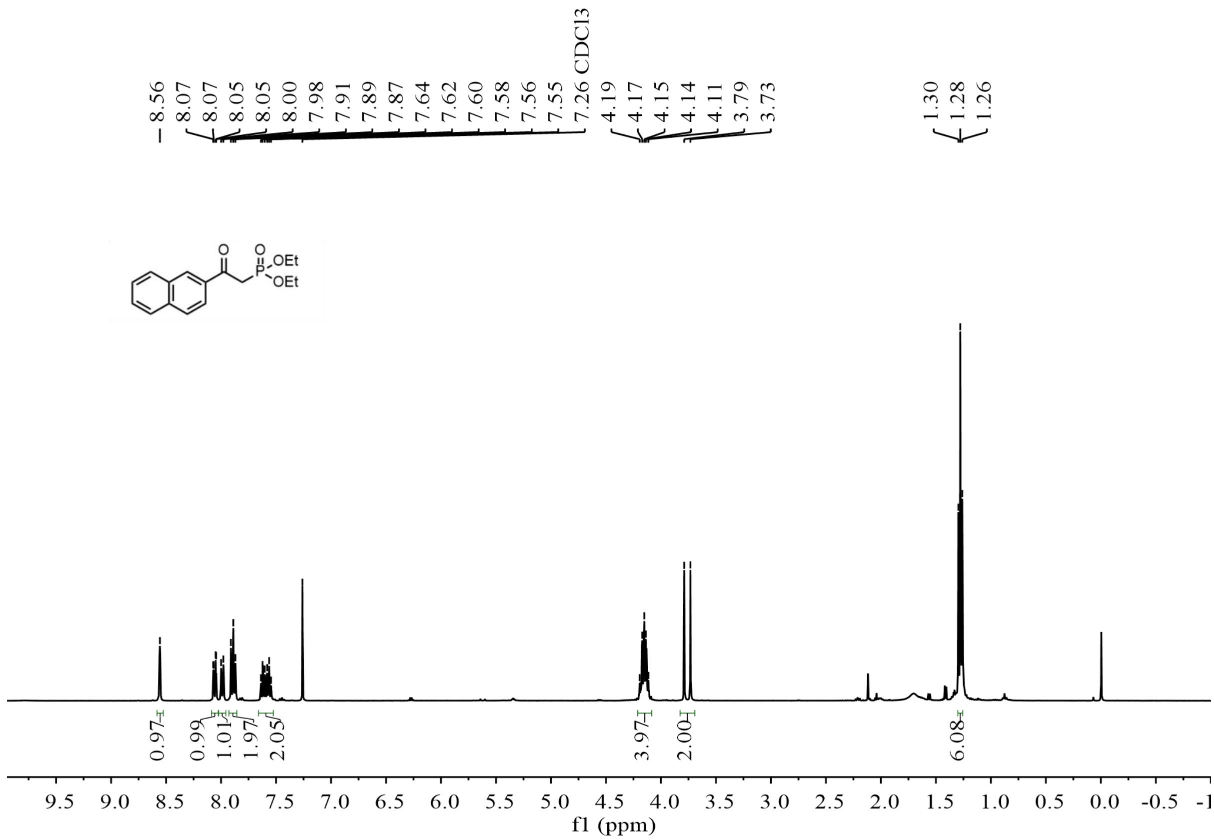
[1]H NMR spectrum of **3i** (400 MHz, $CDCl_3$)



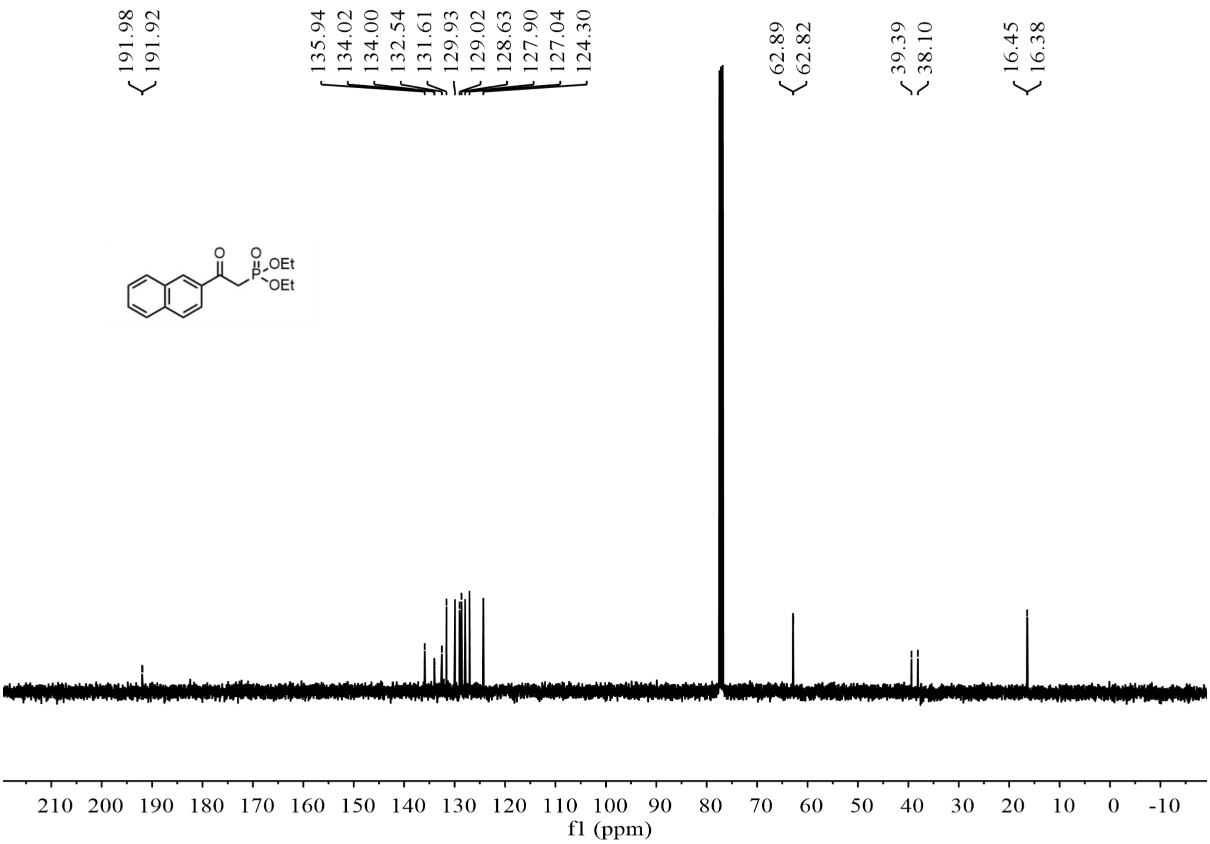
[31]C NMR spectrum of **3i** (100 MHz, $CDCl_3$)



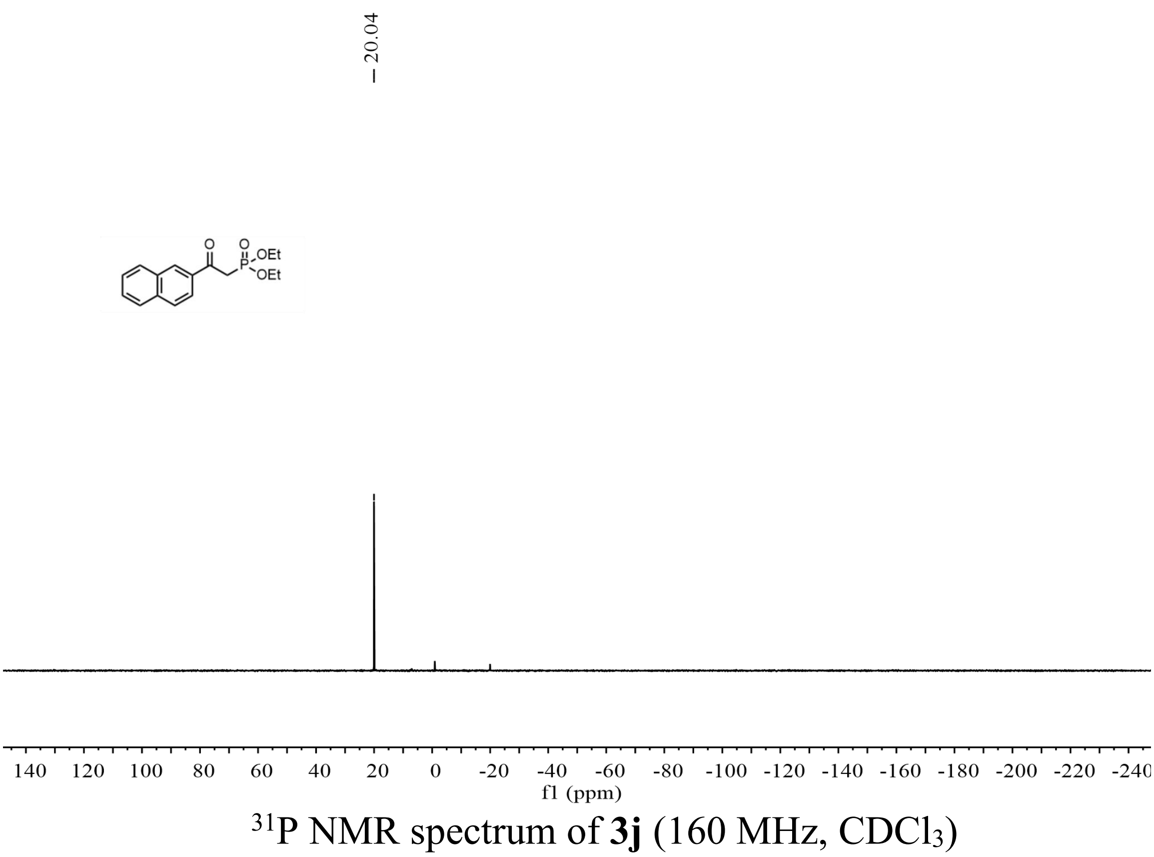
^{31}P NMR spectrum of **3i** (160 MHz, $CDCl_3$)



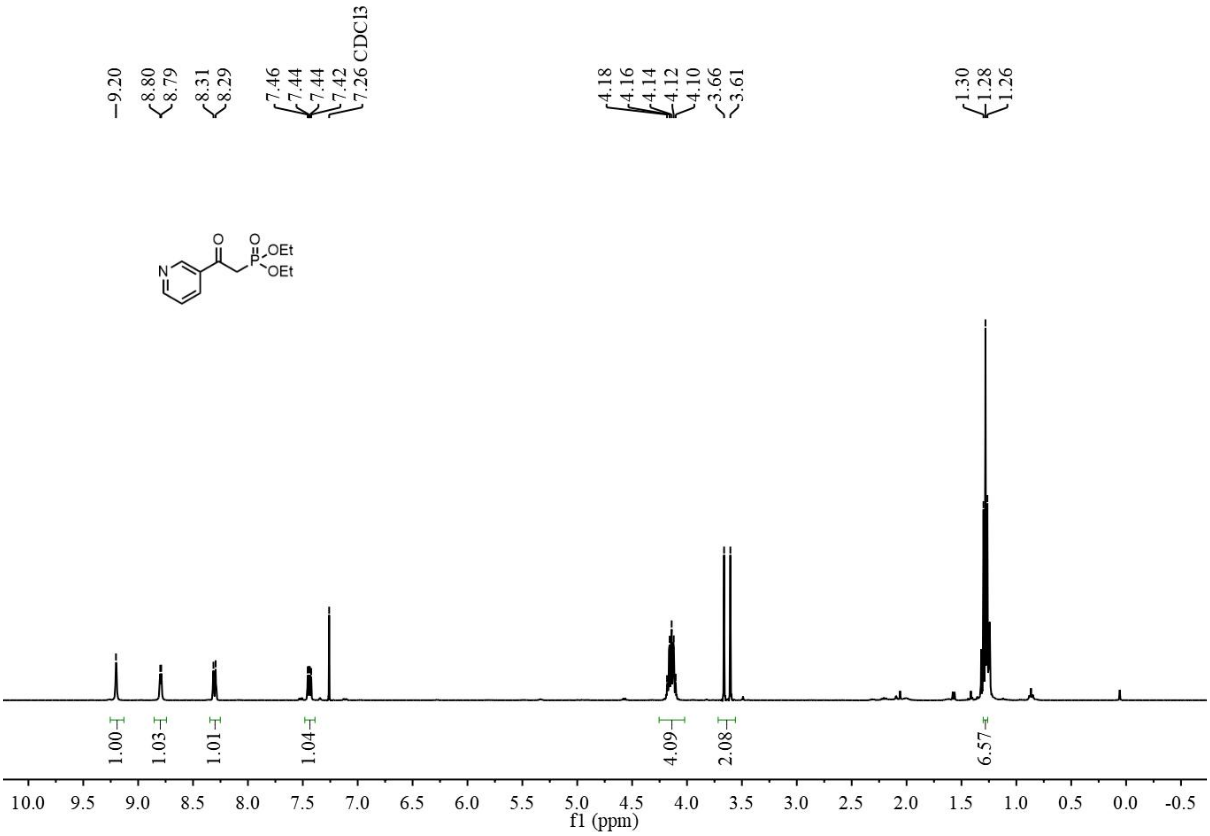
^{1}H NMR spectrum of **3j** (400 MHz, $CDCl_3$)



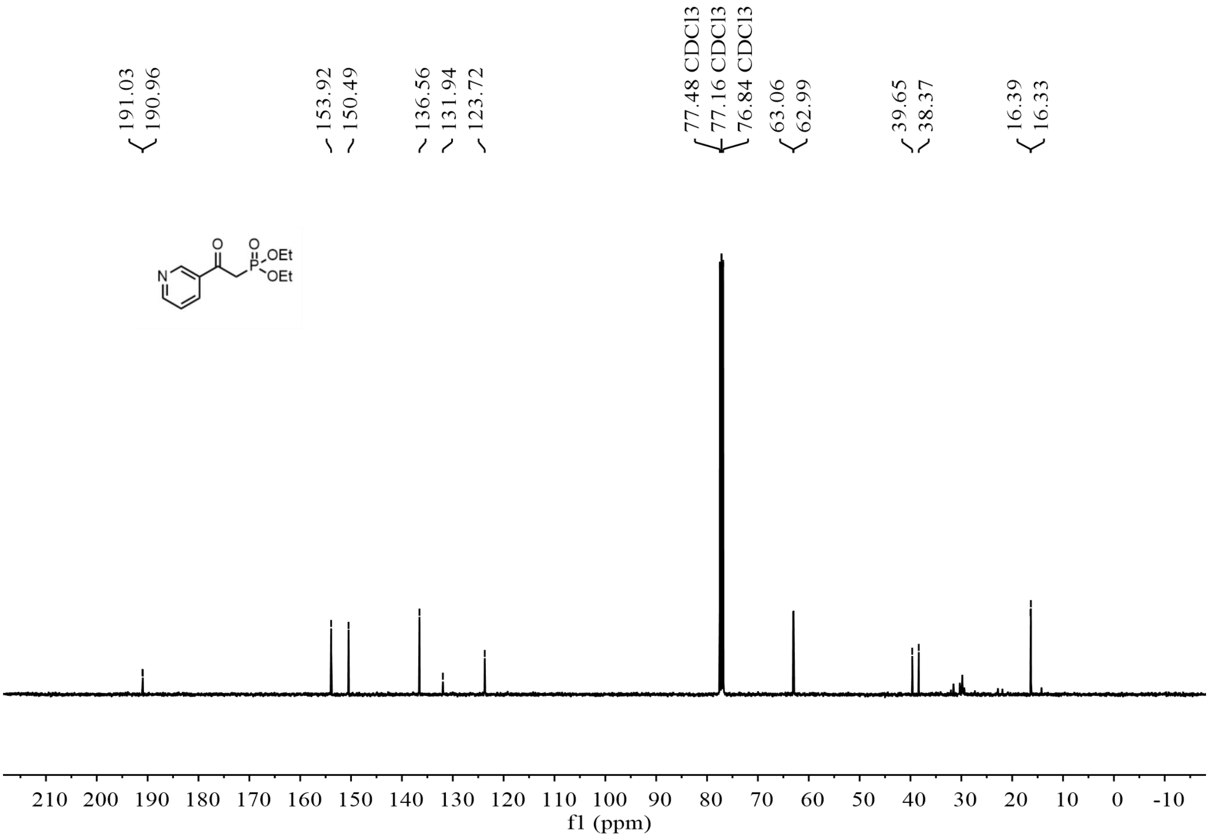
^{13}C NMR spectrum of **3j** (100 MHz, $CDCl_3$)



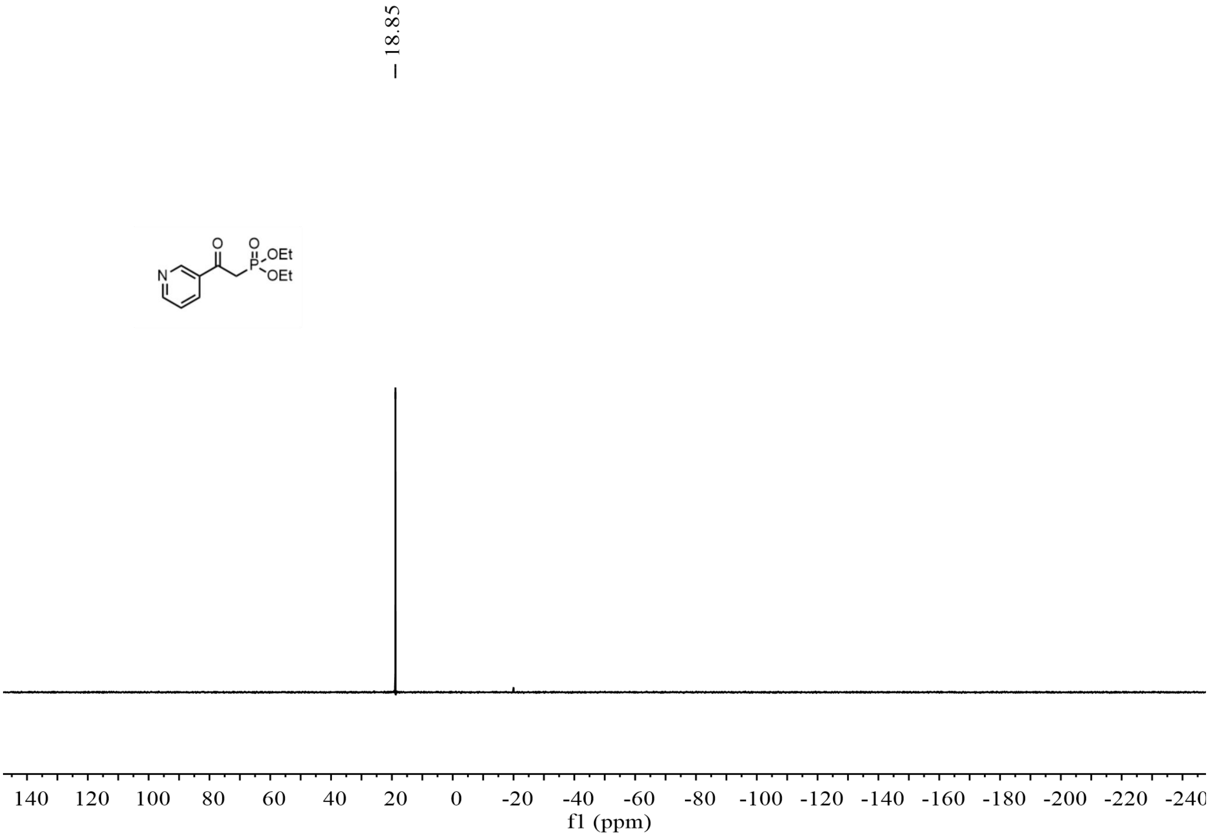
^{31}P NMR spectrum of **3j** (160 MHz, $CDCl_3$)



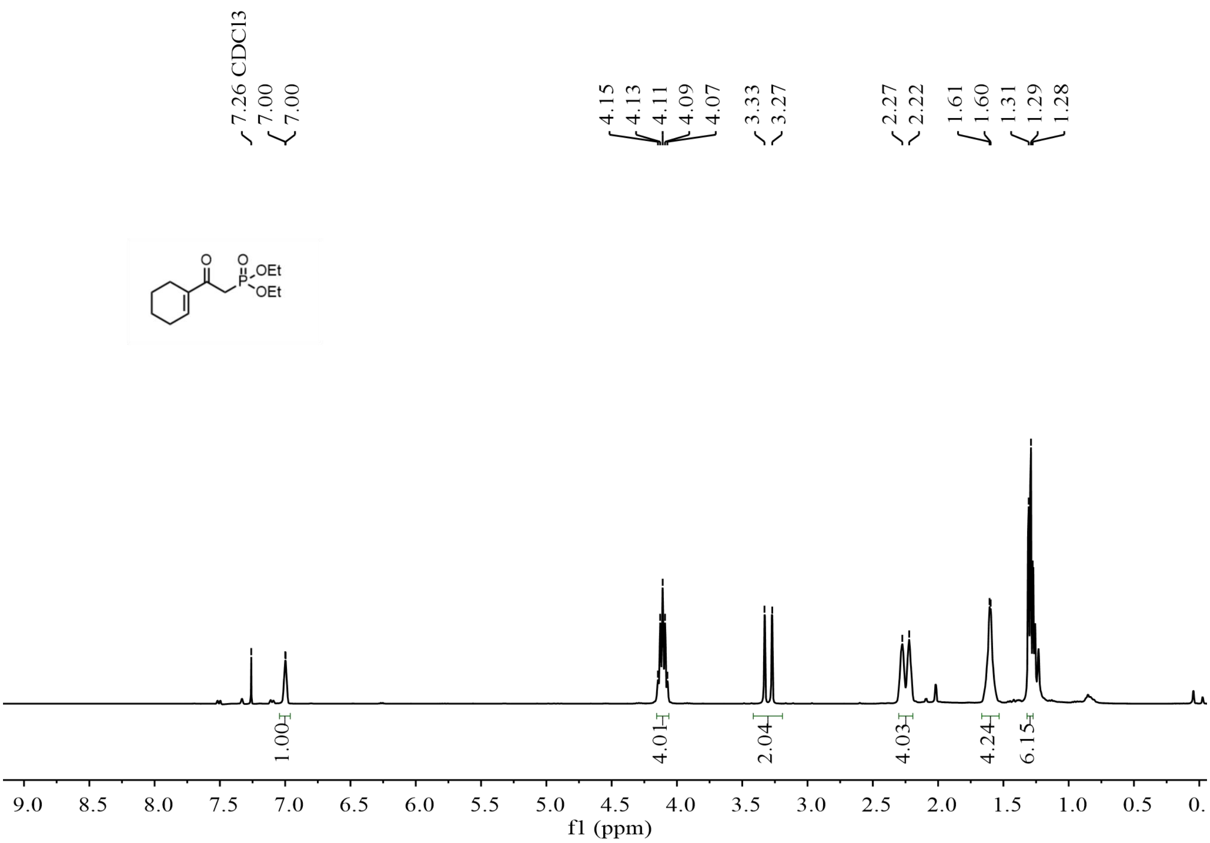
^{1}H NMR spectrum of **3k** (400 MHz, $CDCl_3$)



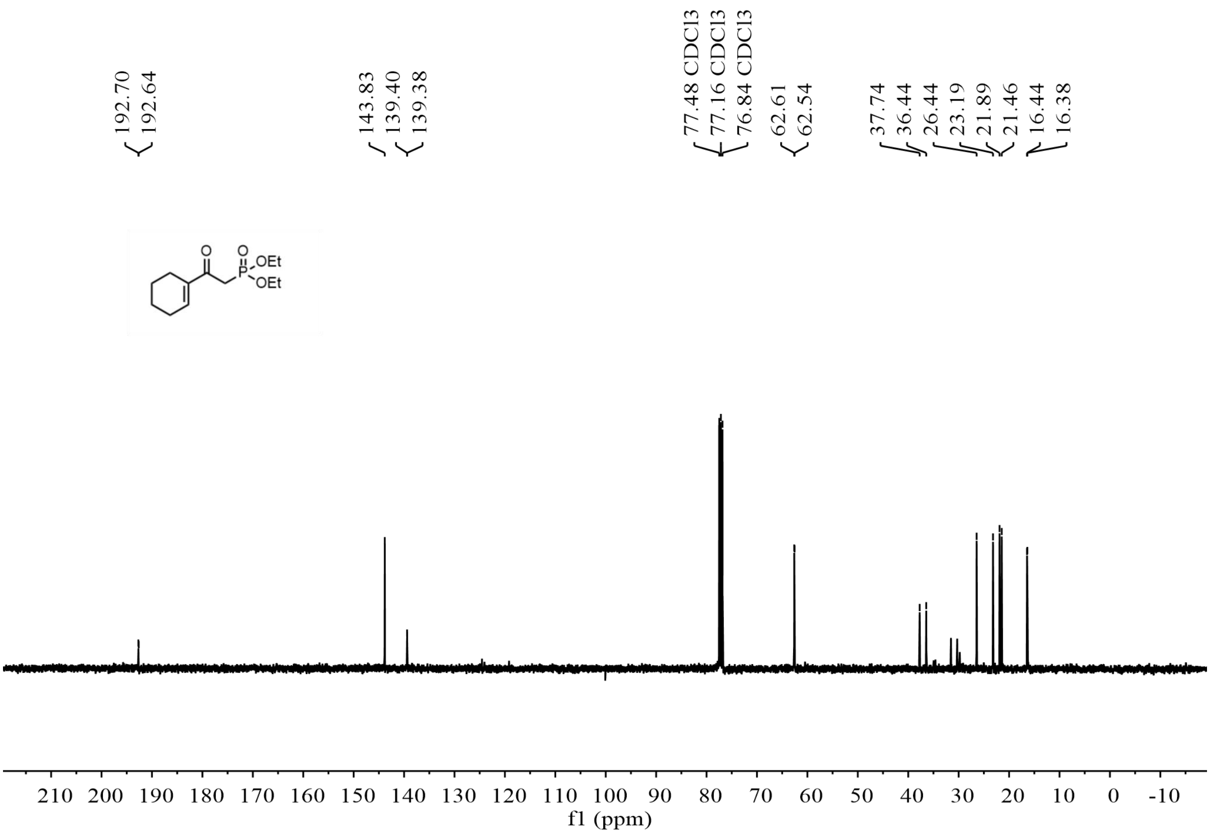
^{13}C NMR spectrum of **3k** (100 MHz, $CDCl_3$)



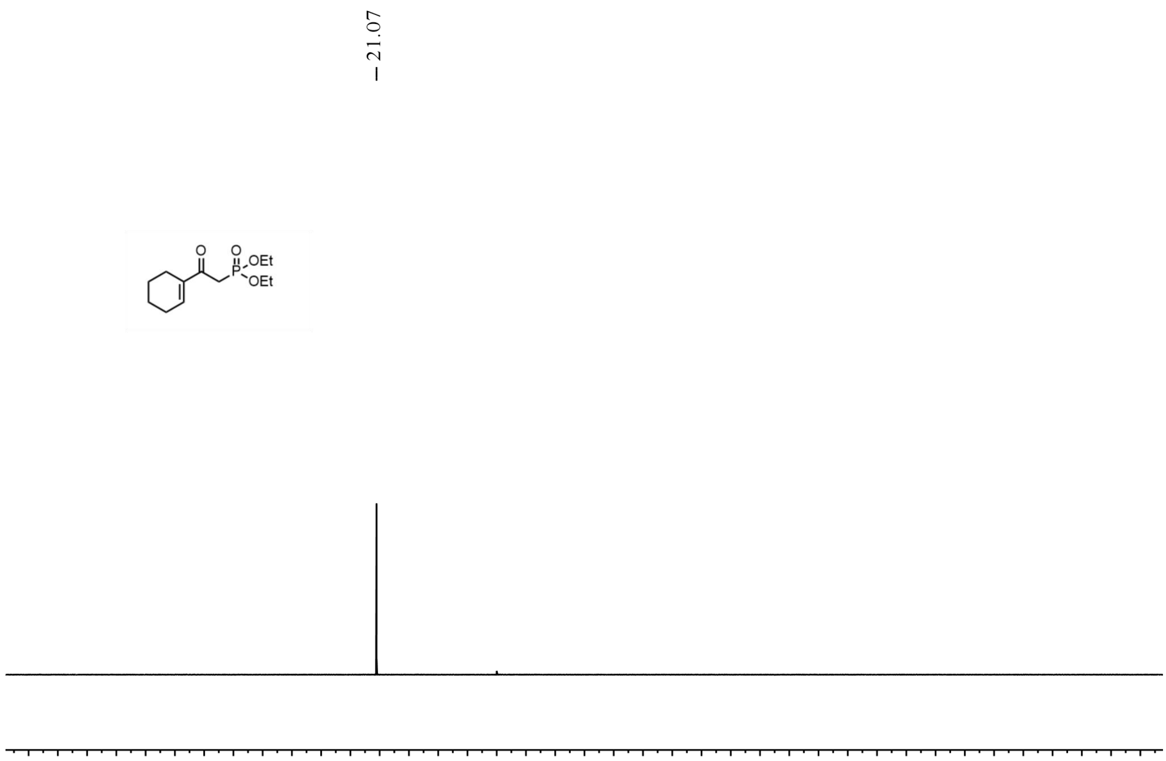
^{31}P NMR spectrum of **3k** (160 MHz, $CDCl_3$)



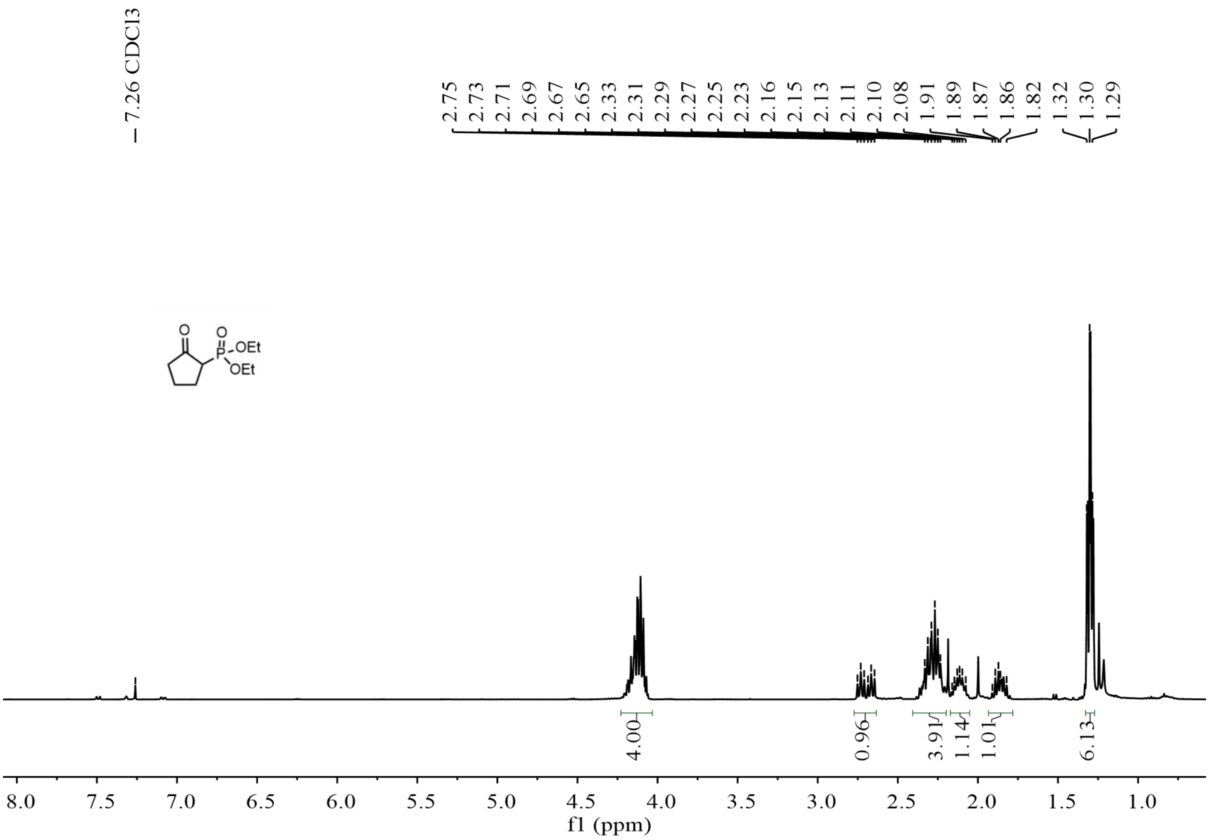
^{1}H NMR spectrum of **3l** (400 MHz, $CDCl_3$)



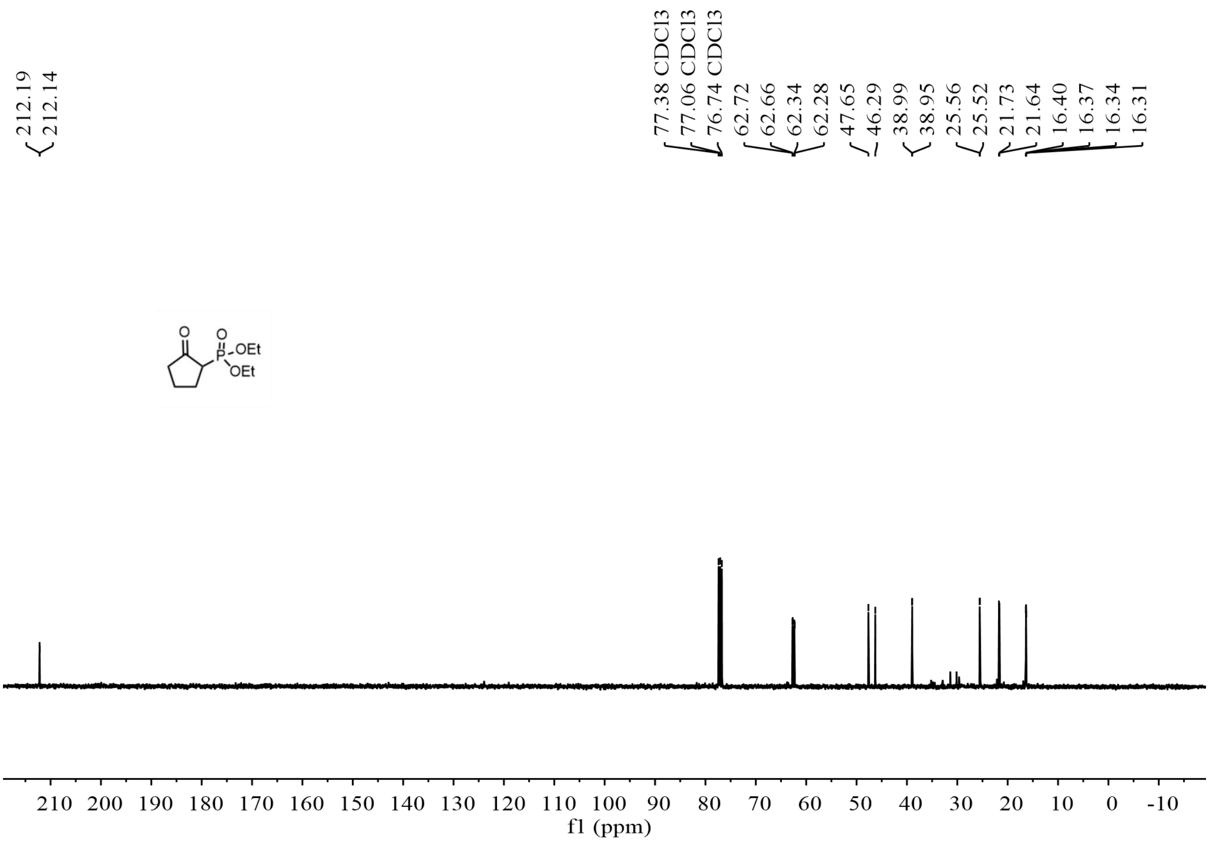
^{13}C NMR spectrum of **3l** (100 MHz, $CDCl_3$)



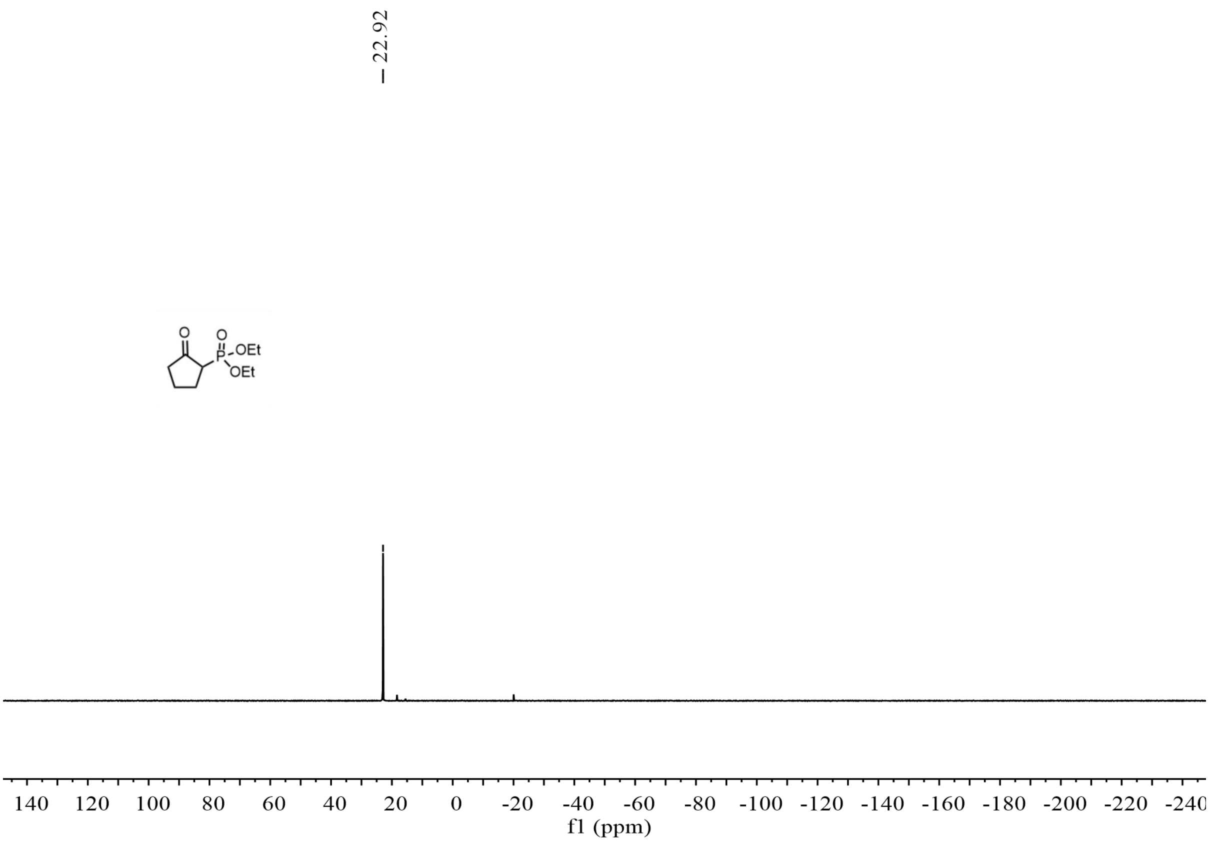
^{31}P NMR spectrum of **3l** (160 MHz, $CDCl_3$)



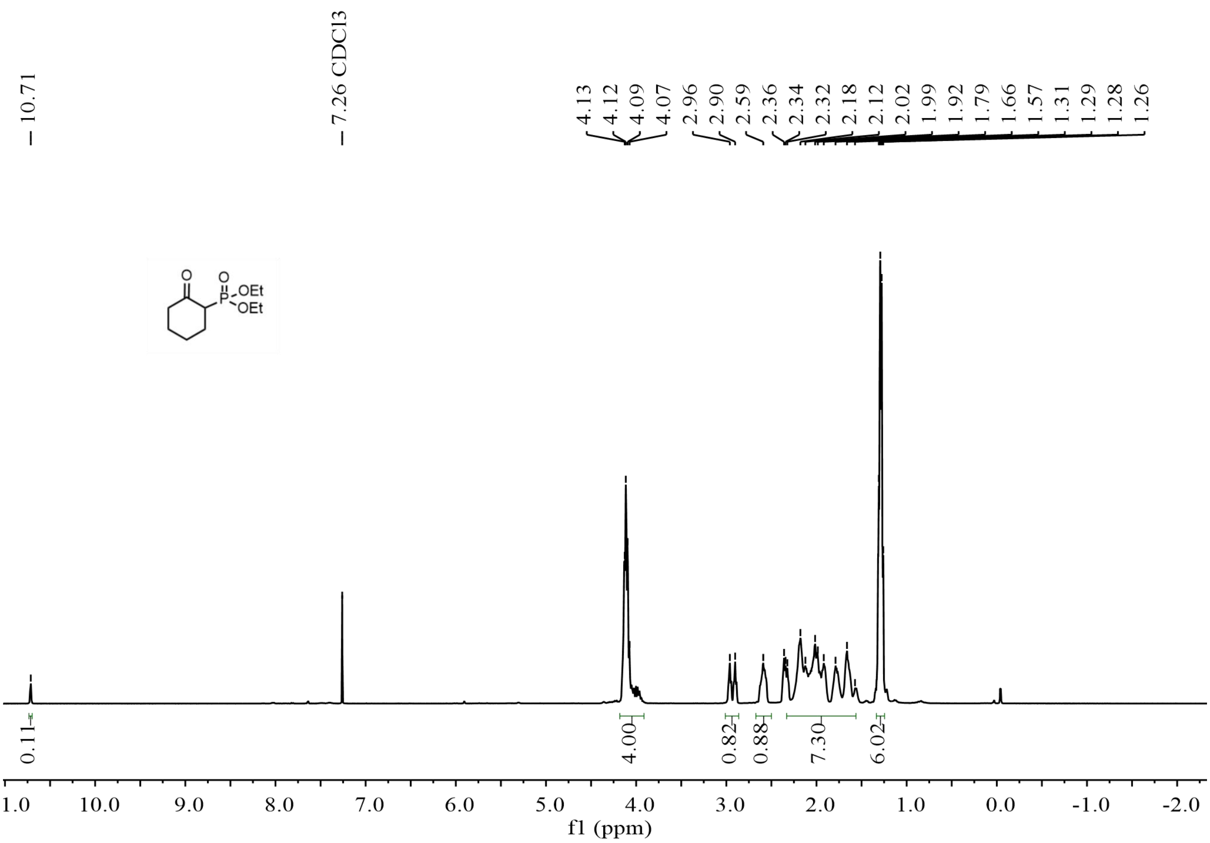
^{1}H NMR spectrum of **3m** (400 MHz, $CDCl_3$)



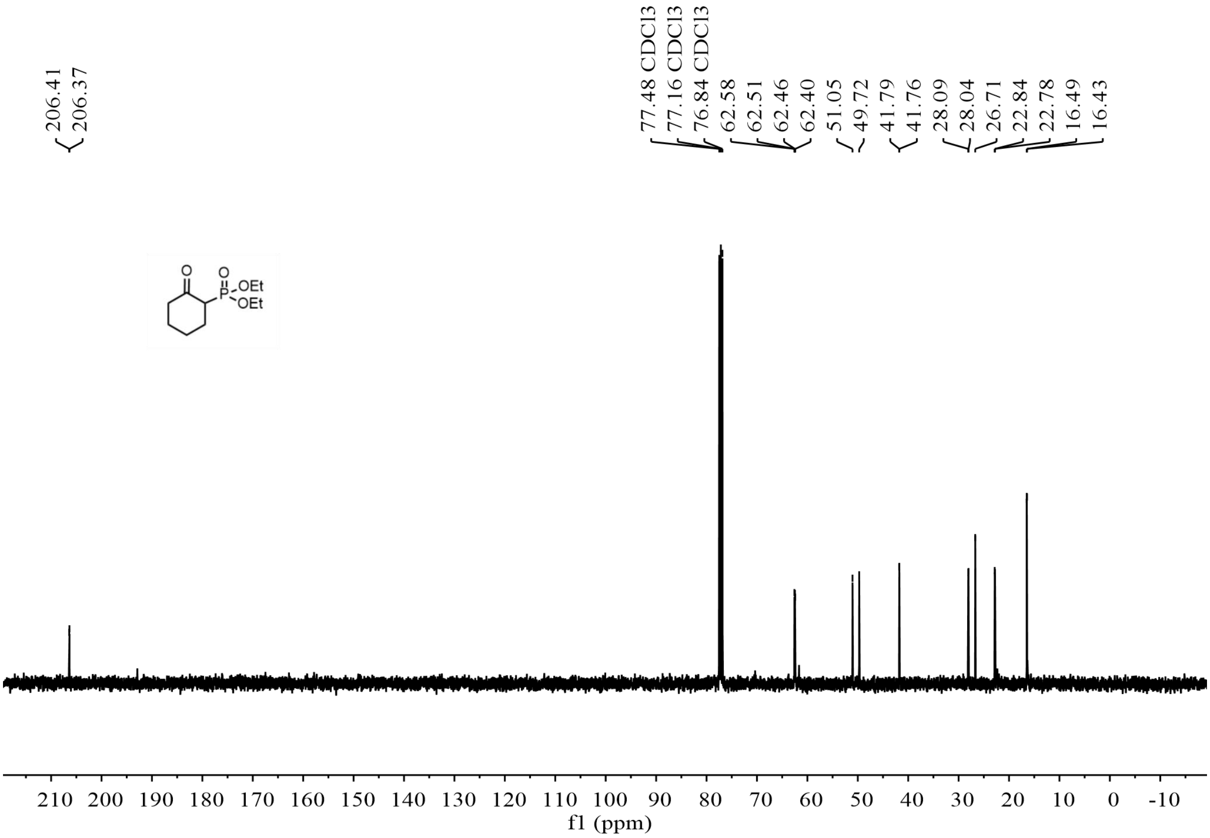
^{13}C NMR spectrum of **3m** (100 MHz, $CDCl_3$)



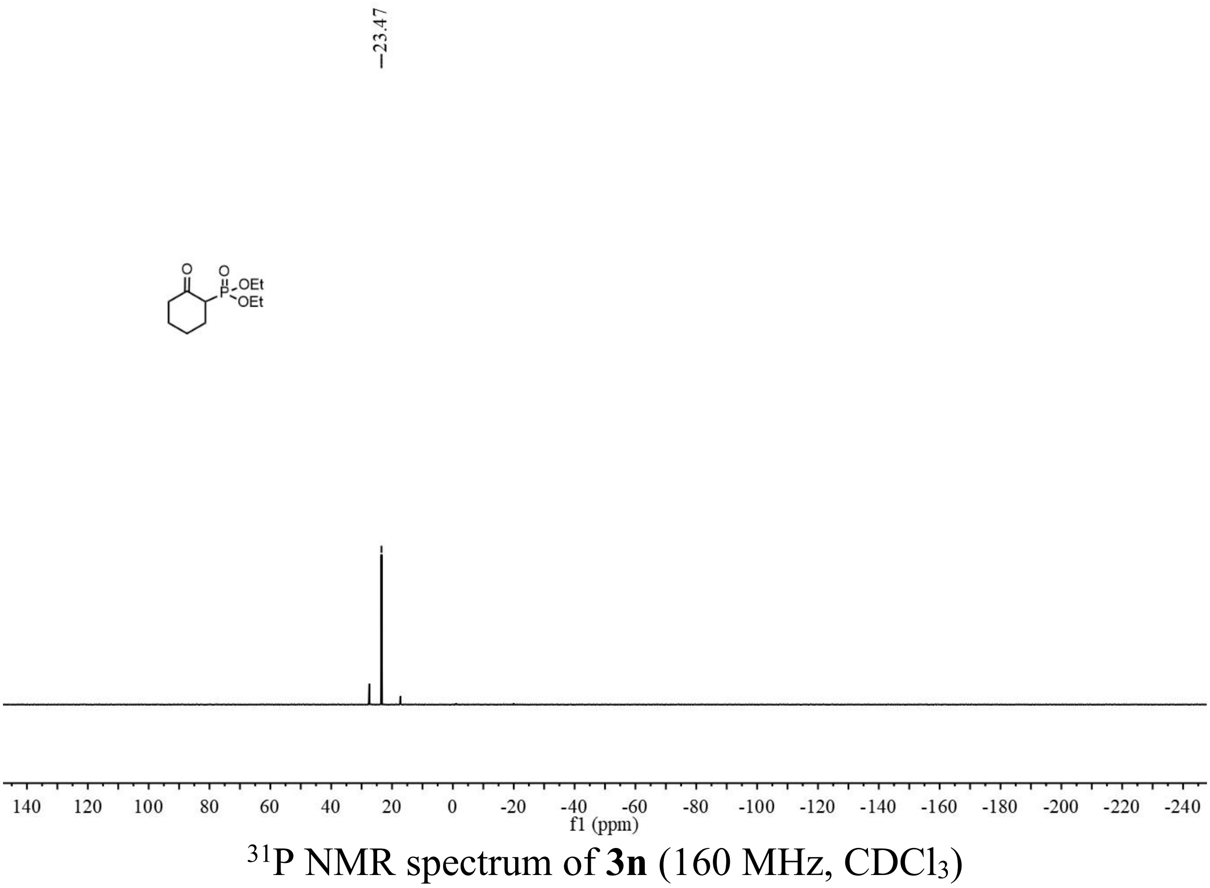
^{31}P NMR spectrum of **3m** (160 MHz, $CDCl_3$)



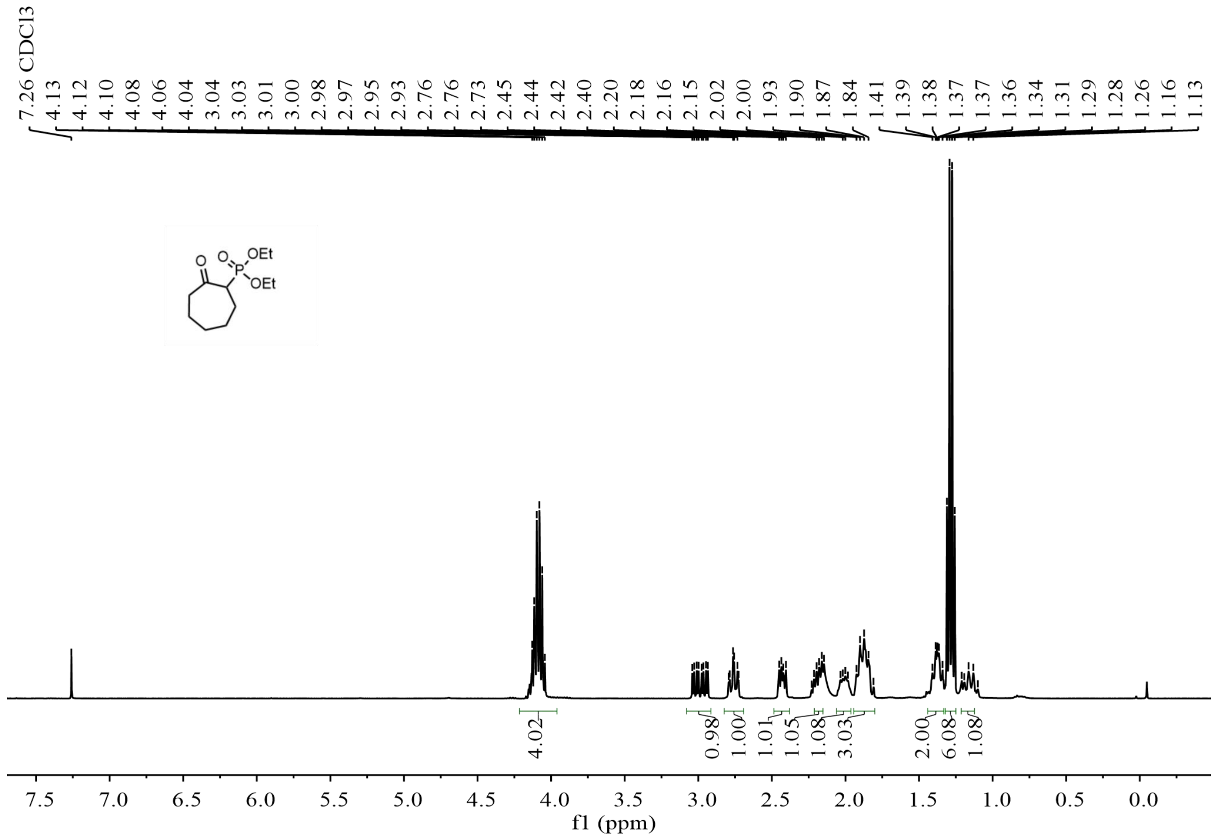
^{1}H NMR spectrum of **3n** (400 MHz, $CDCl_3$)



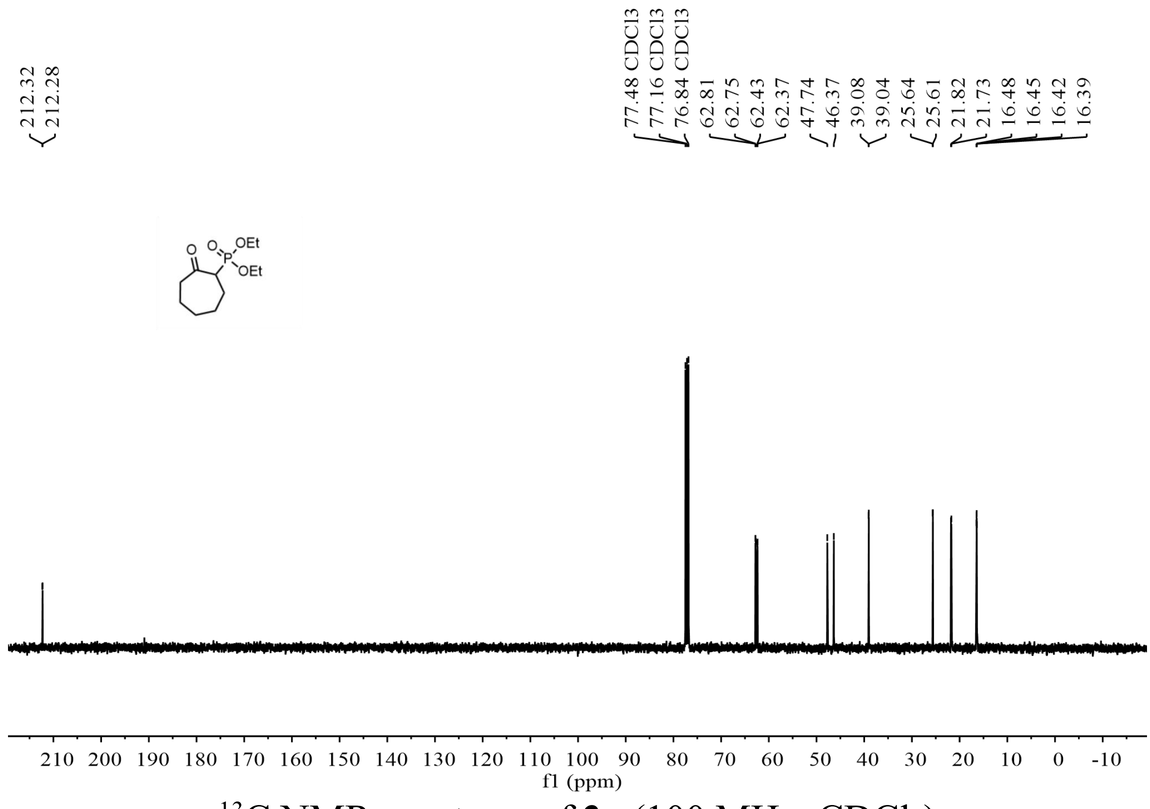
^{13}C NMR spectrum of **3n** (100 MHz, $CDCl_3$)



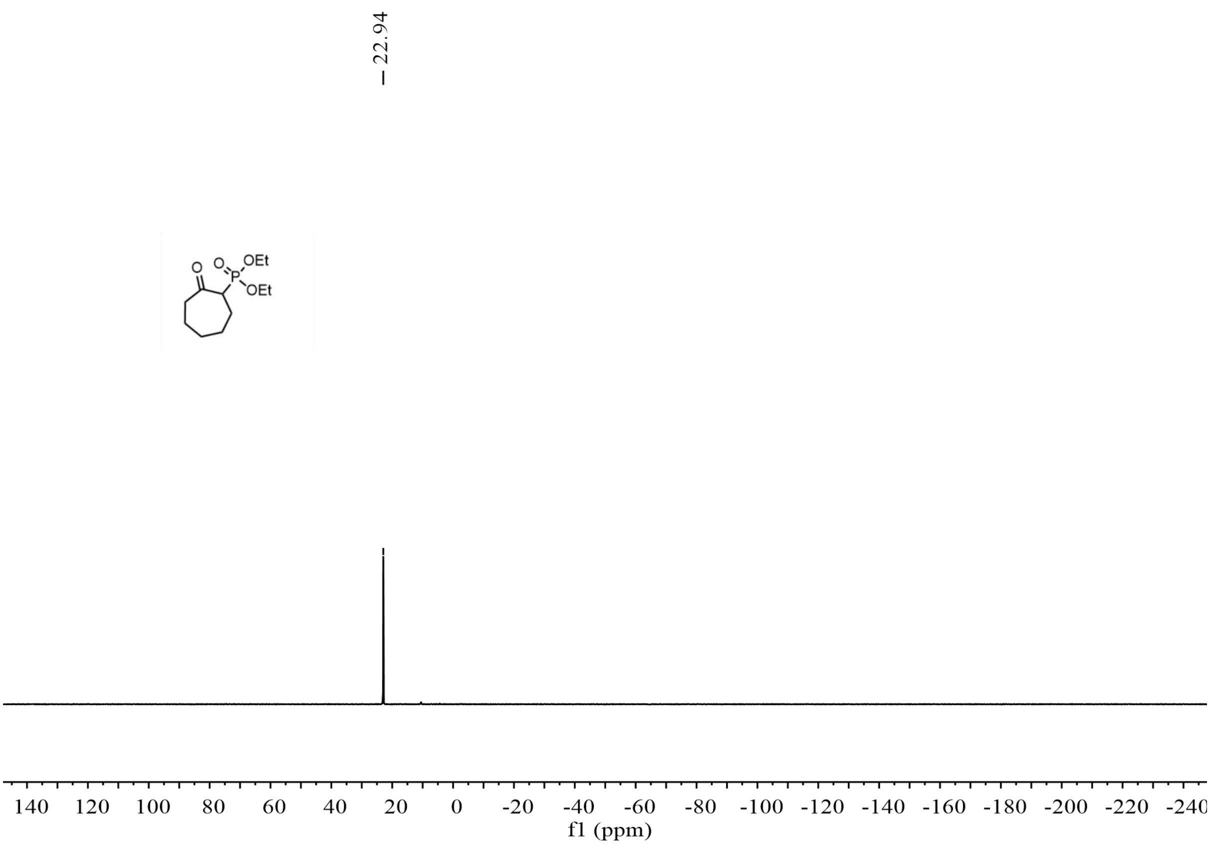
^{31}P NMR spectrum of **3n** (160 MHz, $CDCl_3$)



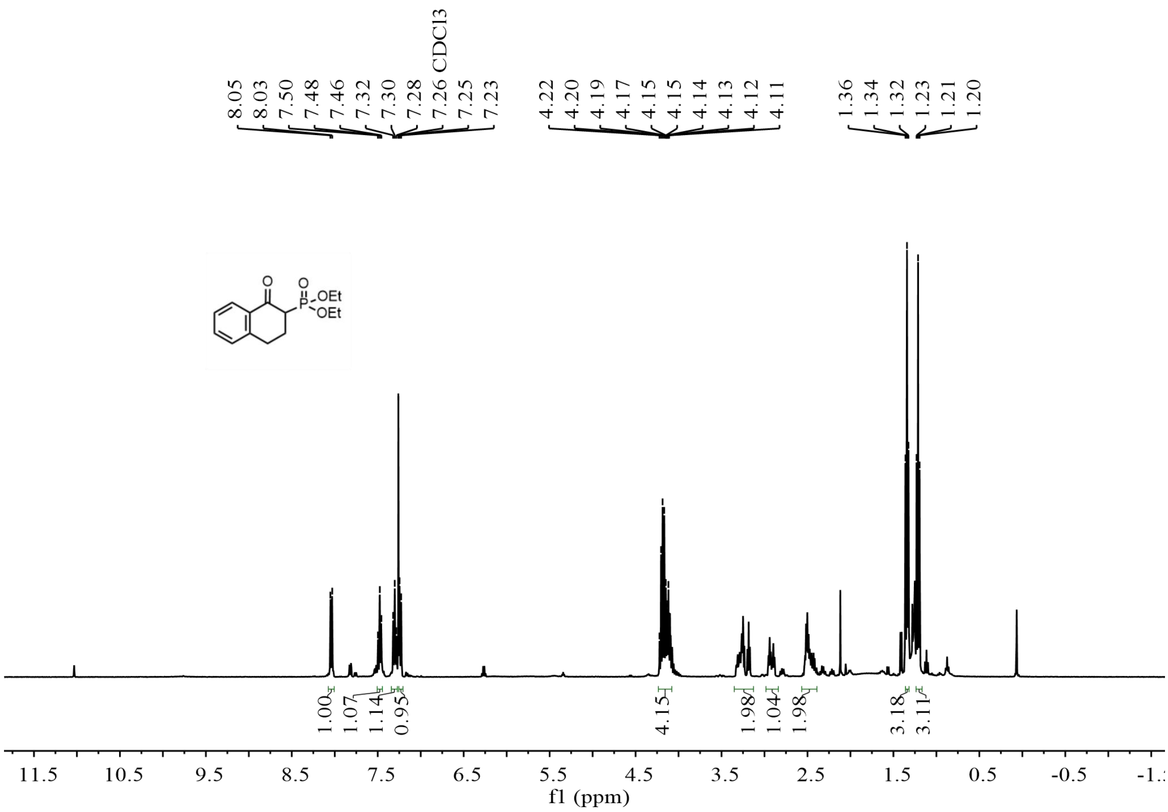
^{1}H NMR spectrum of **3o** (400 MHz, $CDCl_3$)



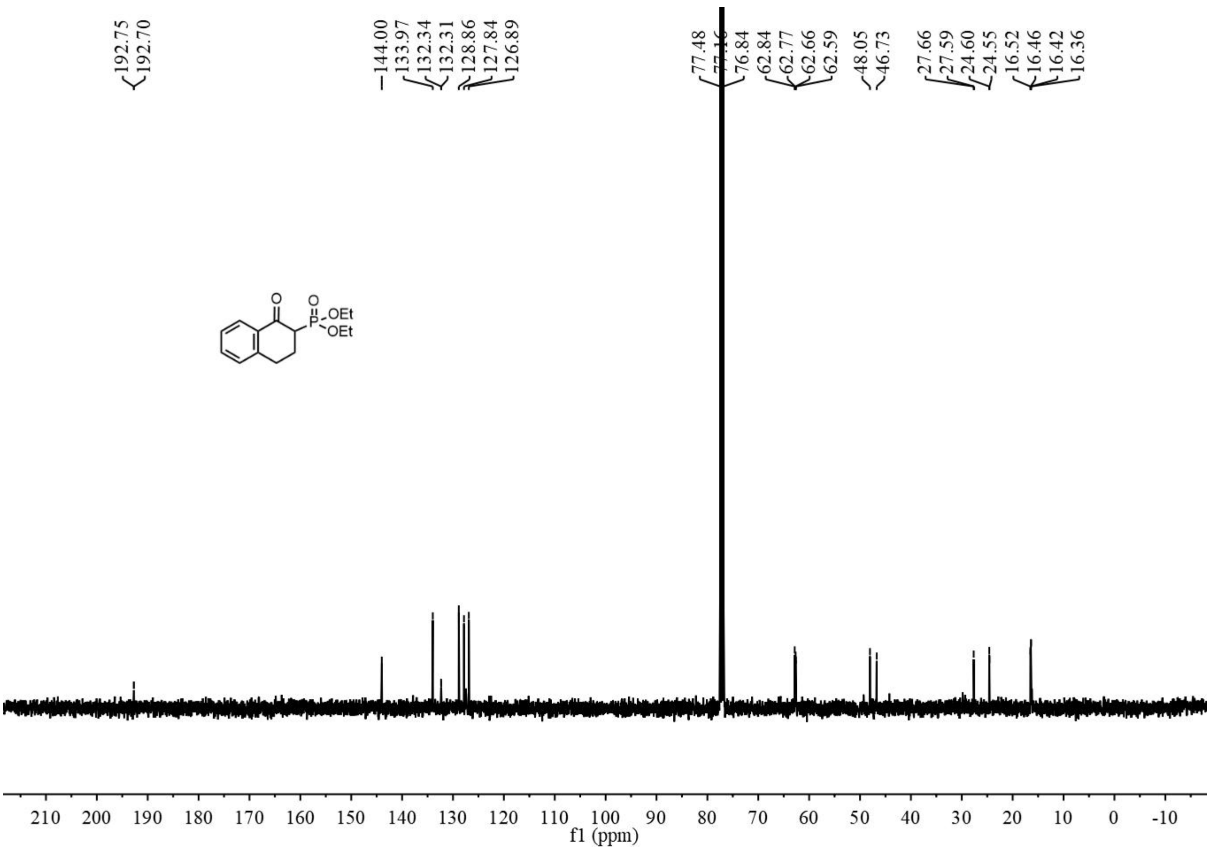
^{13}C NMR spectrum of **3o** (100 MHz, $CDCl_3$)



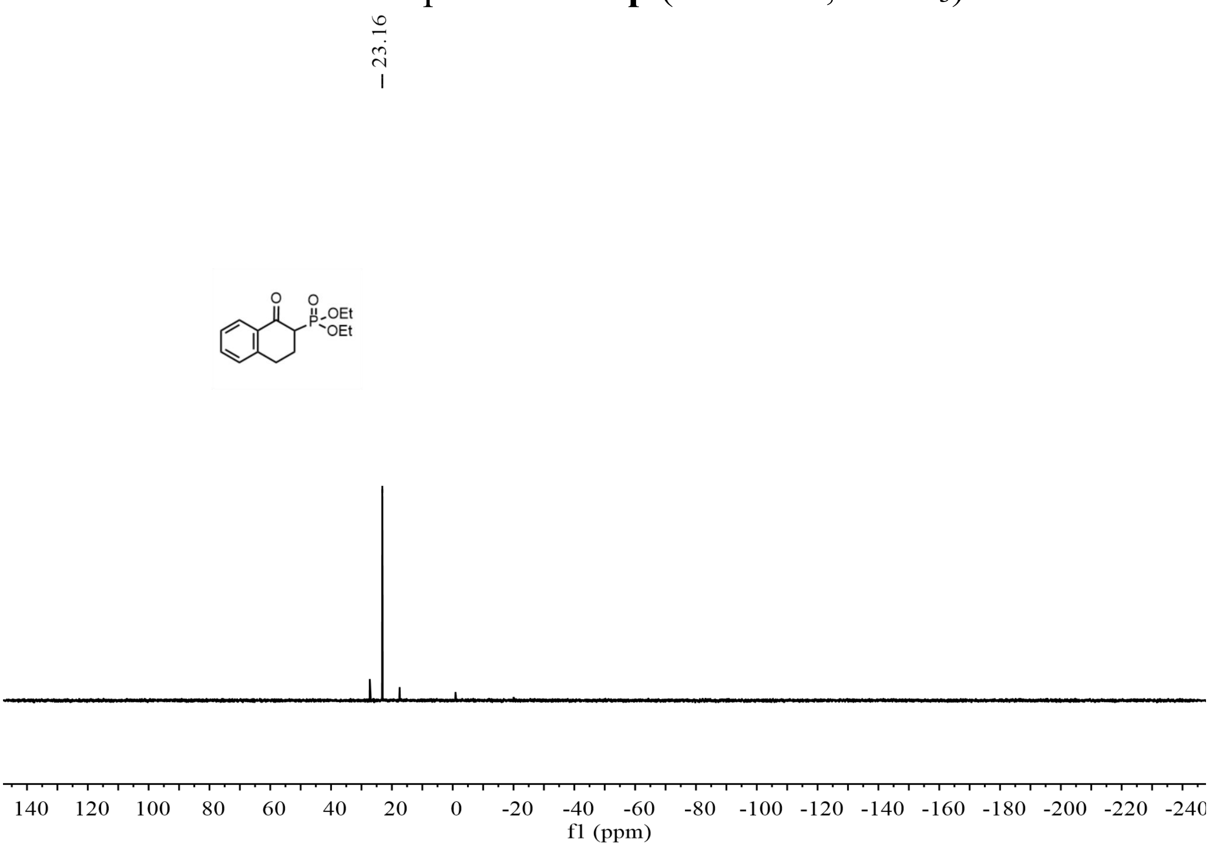
^{31}P NMR spectrum of **3o** (160 MHz, $CDCl_3$)



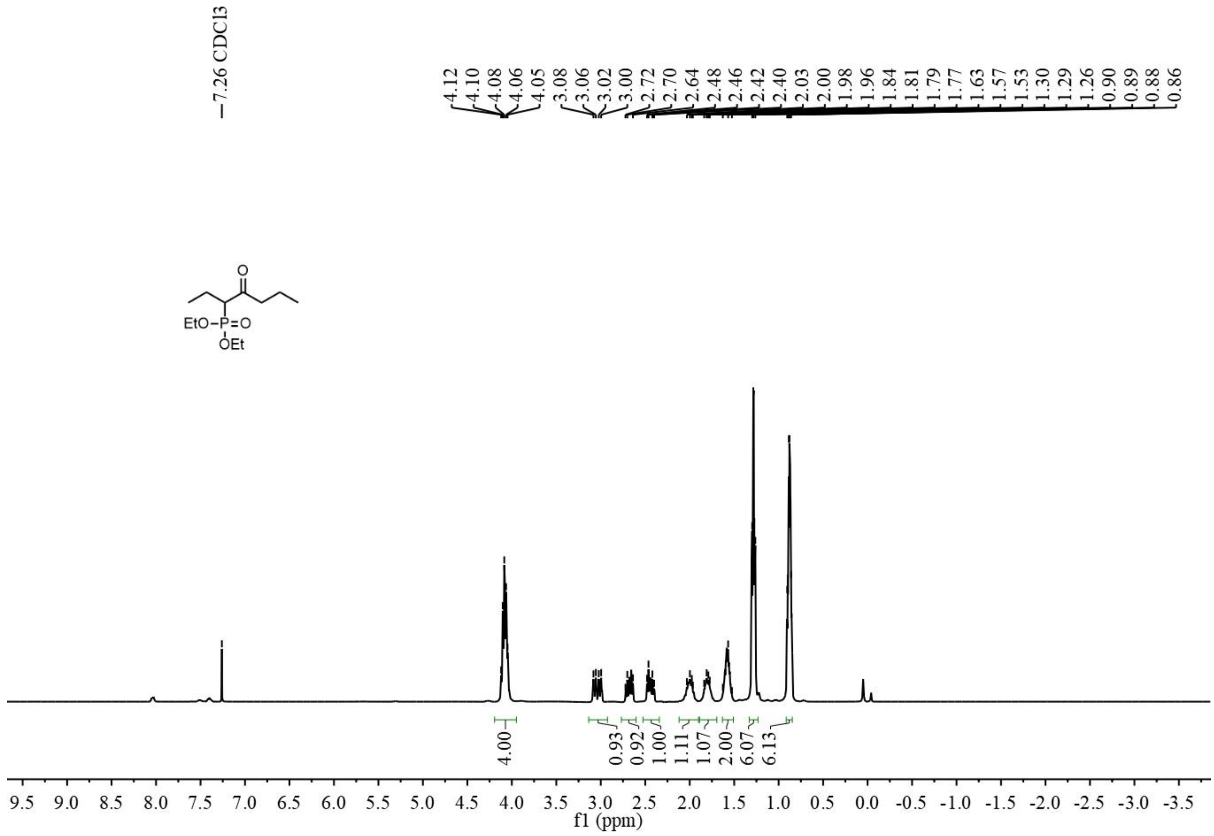
^{1}H NMR spectrum of **3p** (400 MHz, $CDCl_3$)



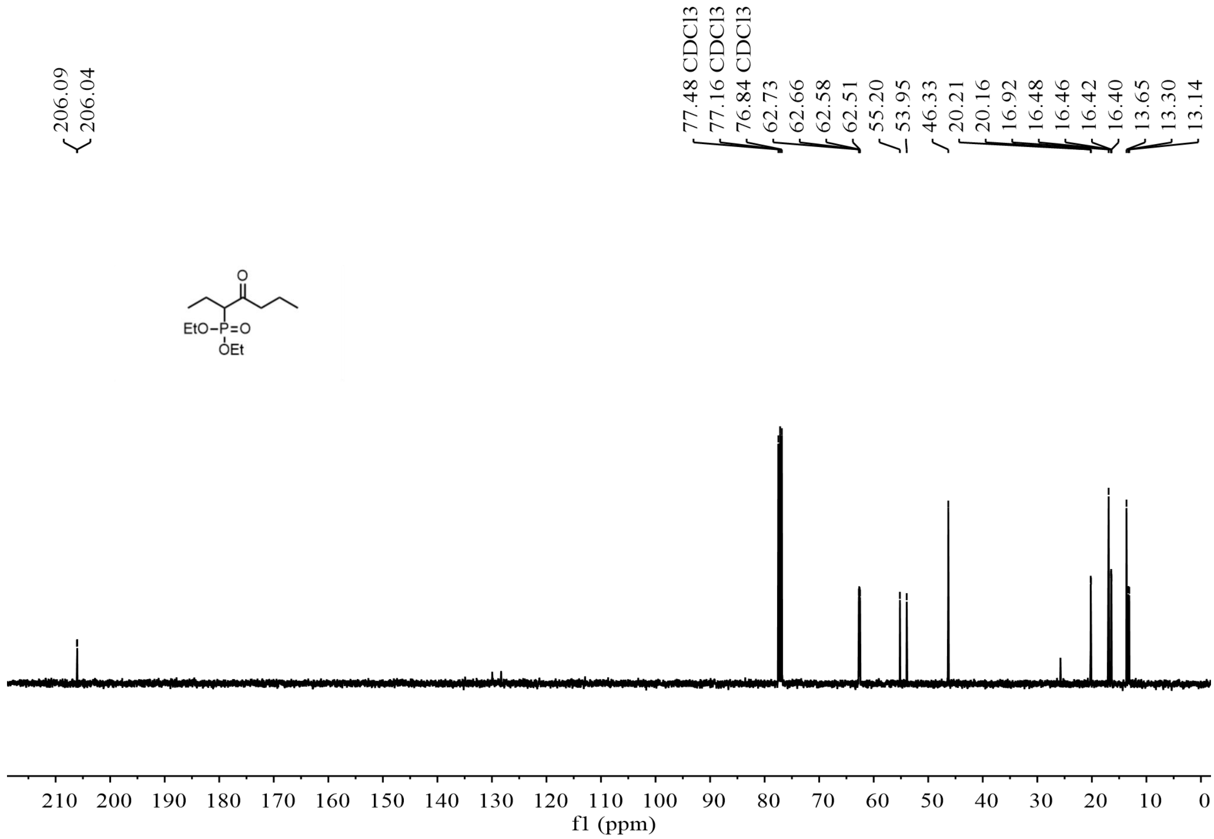
^{13}C NMR spectrum of **3p** (100 MHz, $CDCl_3$)



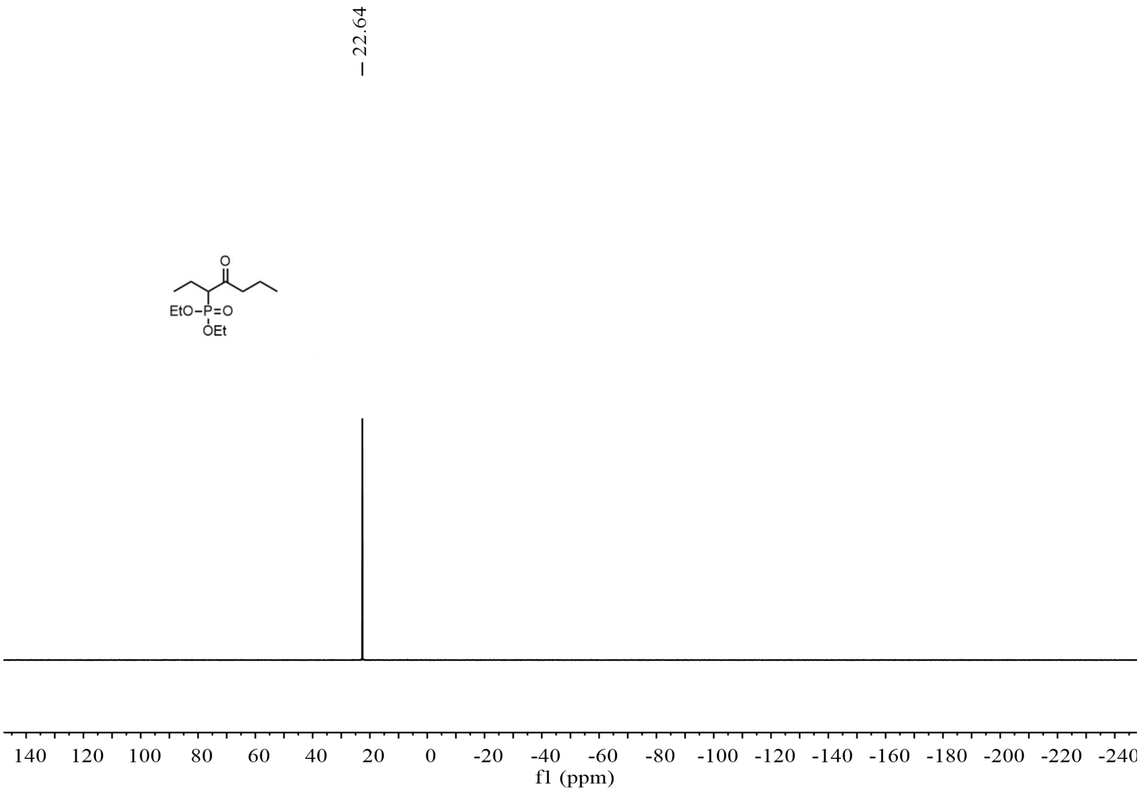
^{31}P NMR spectrum of **3p** (160 MHz, $CDCl_3$)



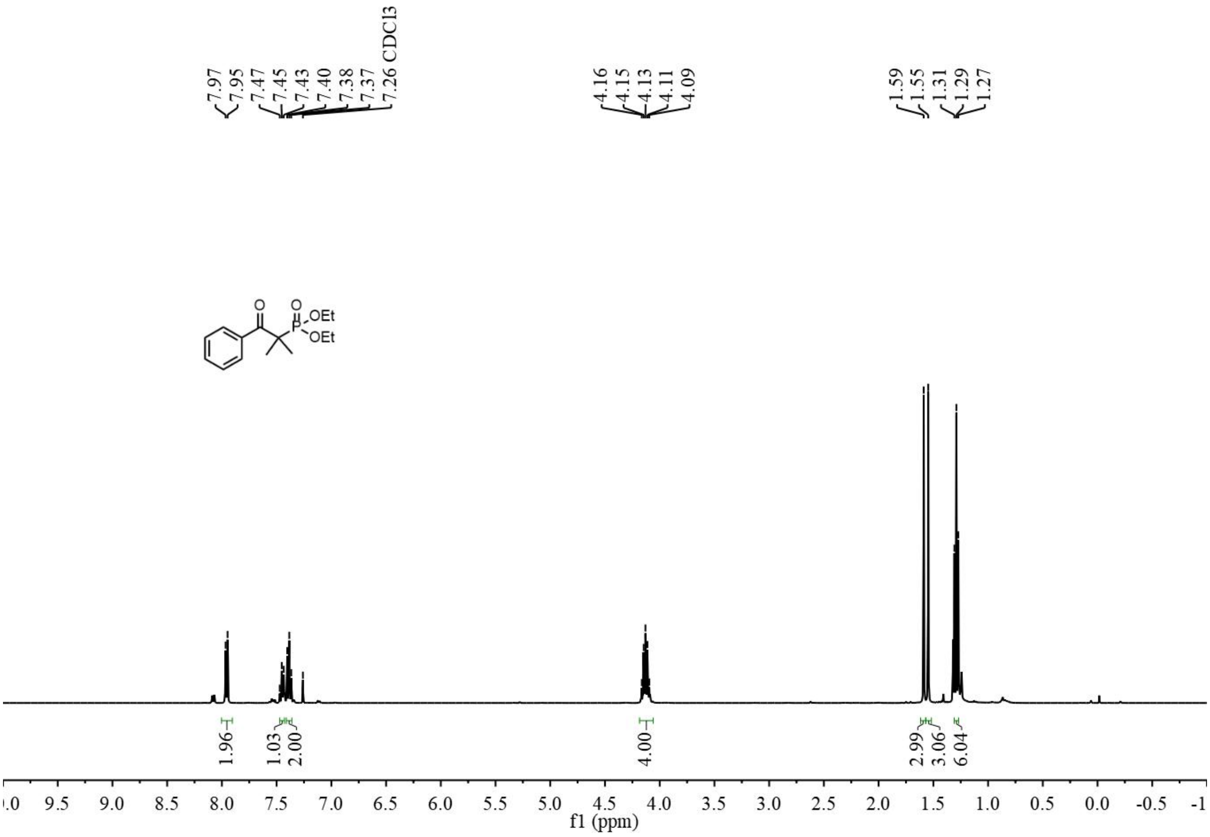
[1]H NMR spectrum of **3q** (400 MHz, $CDCl_3$)



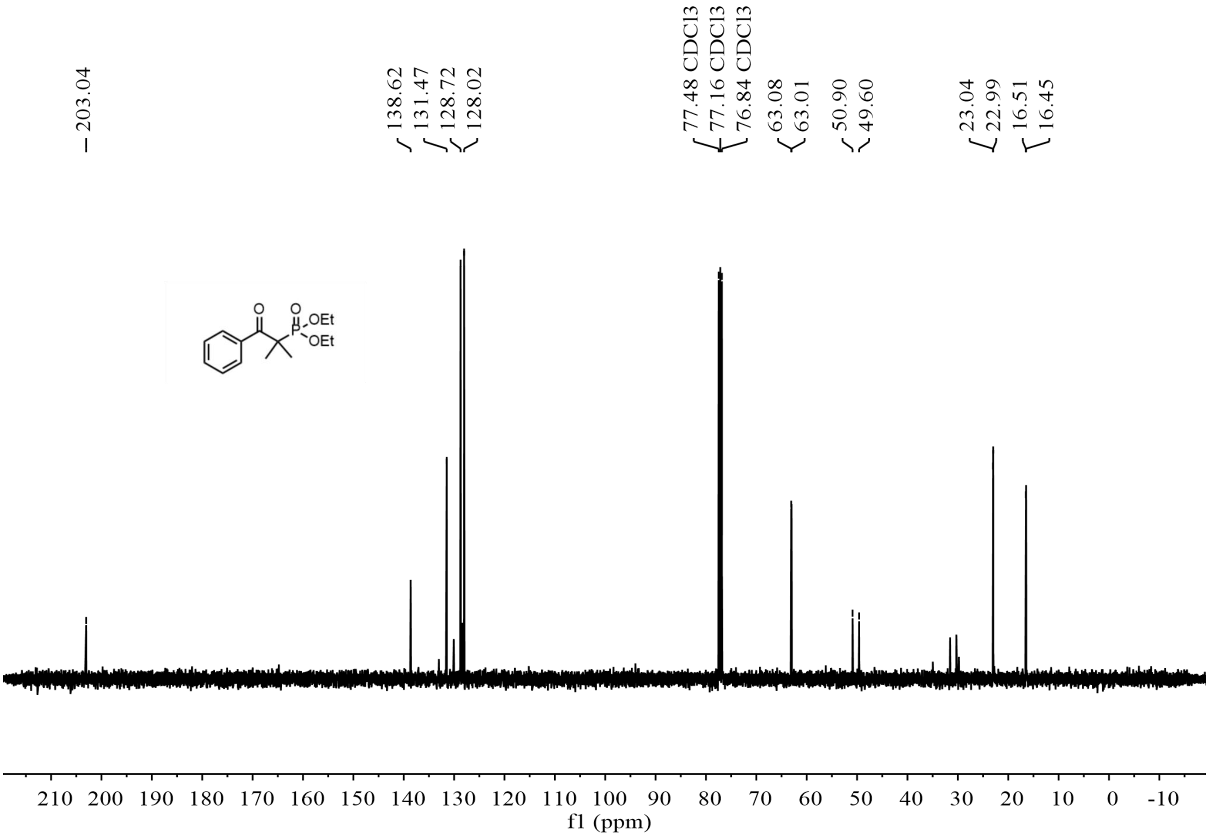
[13]C NMR spectrum of **3q** (100 MHz, $CDCl_3$)



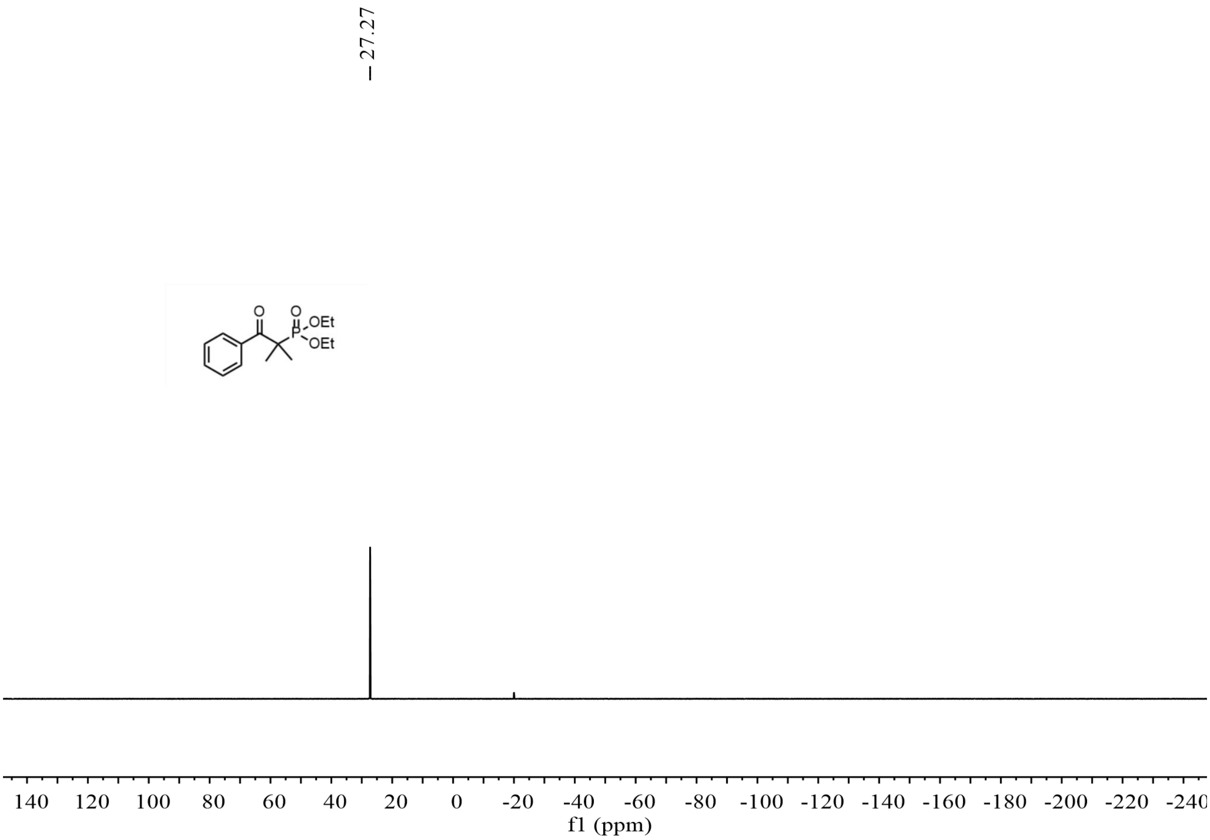
^{31}P NMR spectrum of **3q** (160 MHz, $CDCl_3$)



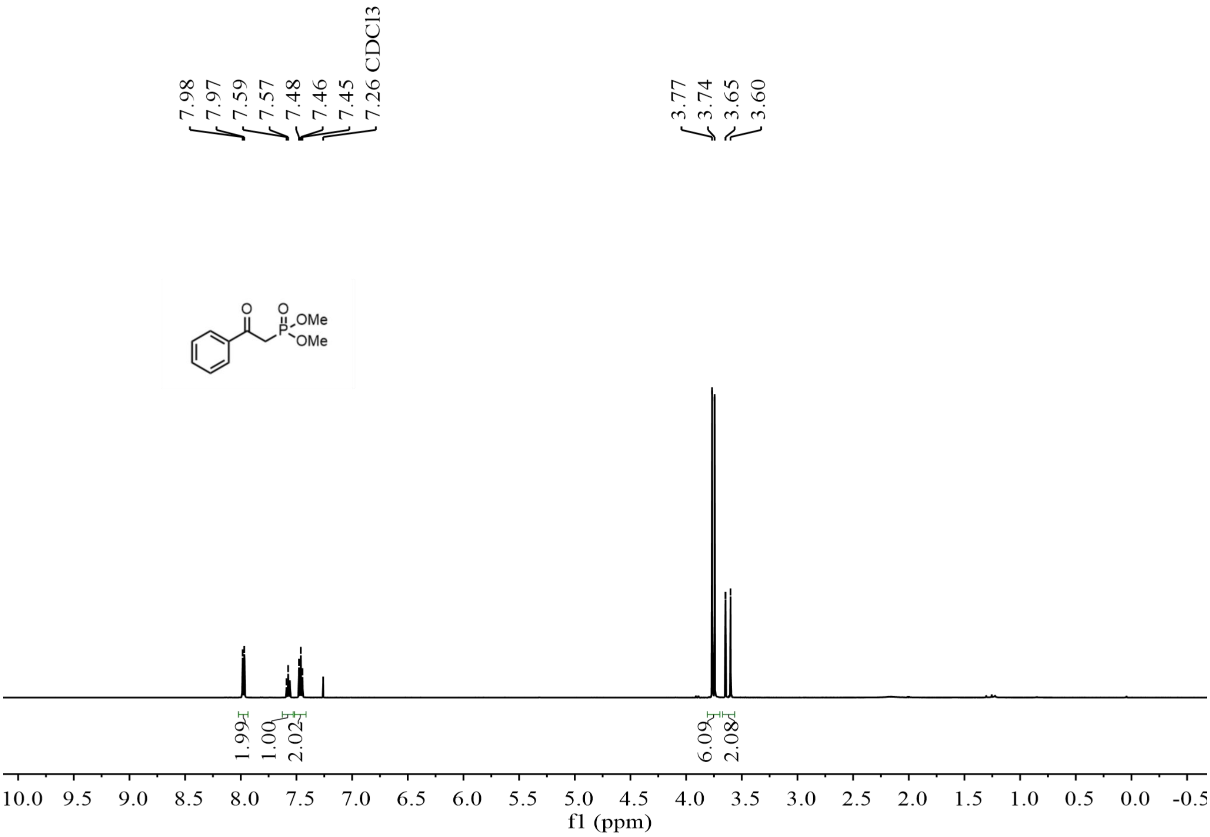
^{1}H NMR spectrum of **3r** (400 MHz, $CDCl_3$)



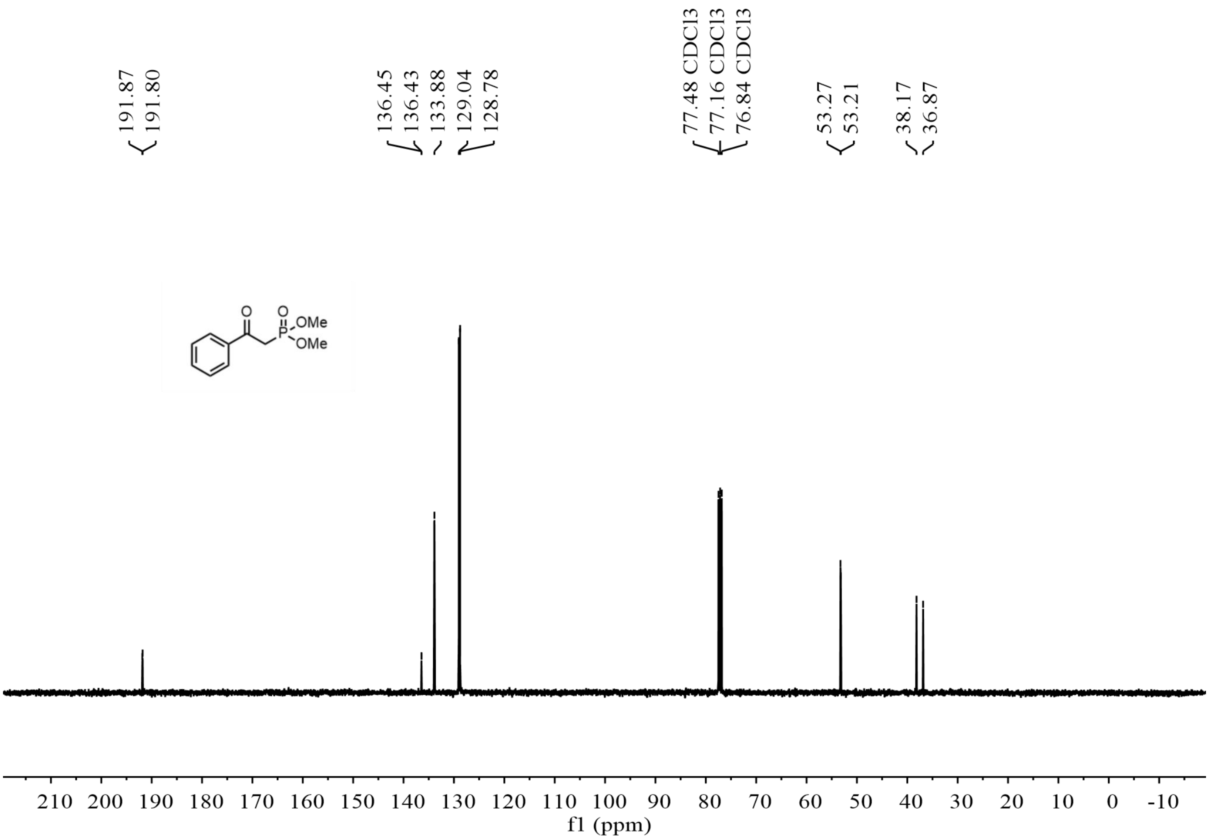
[13]C NMR spectrum of **3r** (100 MHz, $CDCl_3$)



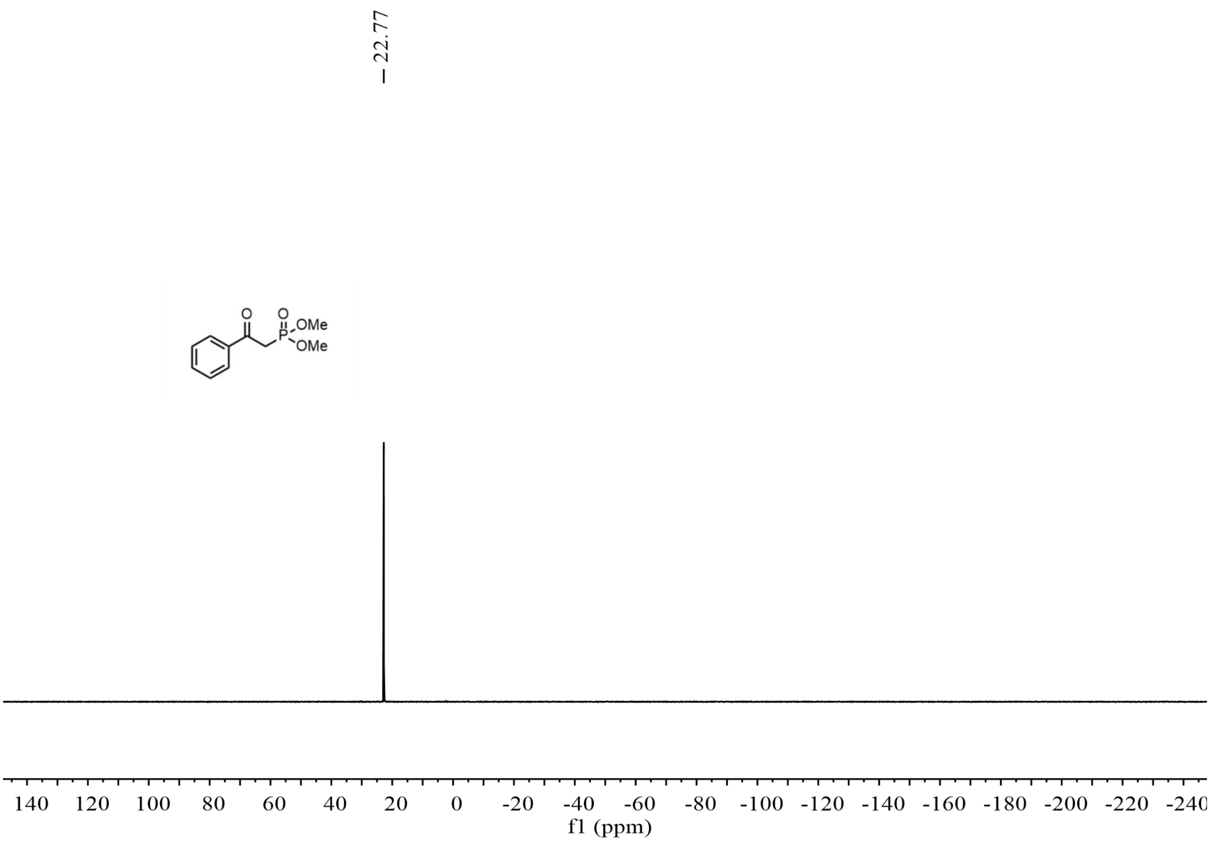
[31]P NMR spectrum of **3r** (160 MHz, $CDCl_3$)



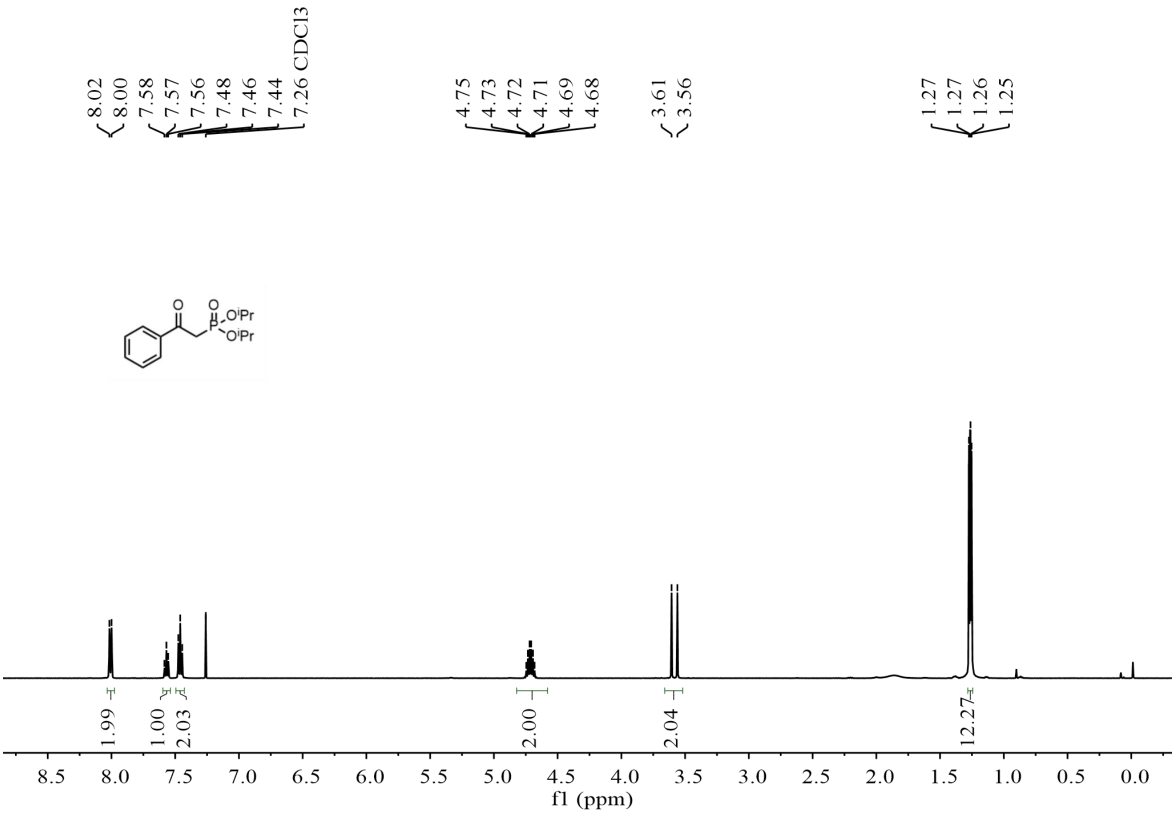
^{1}H NMR spectrum of **3s** (500 MHz, $CDCl_3$)



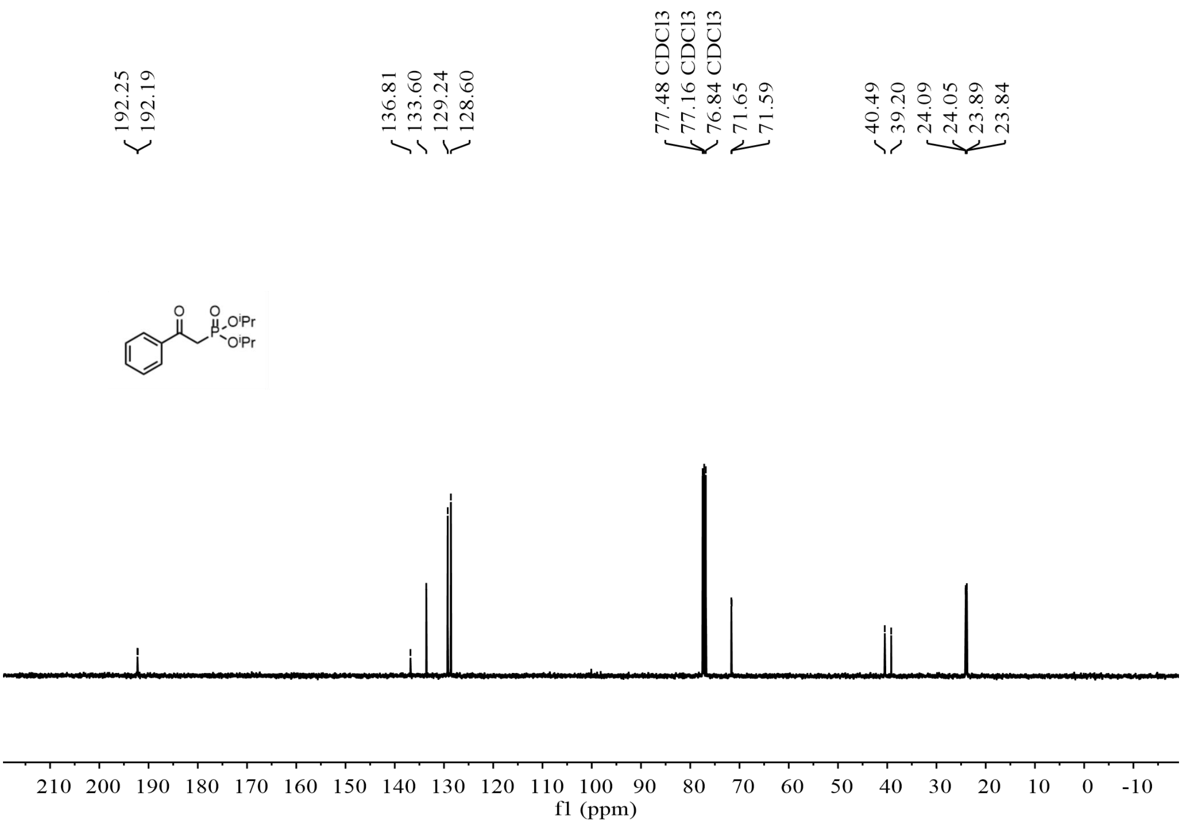
^{13}C NMR spectrum of **3s** (100 MHz, $CDCl_3$)



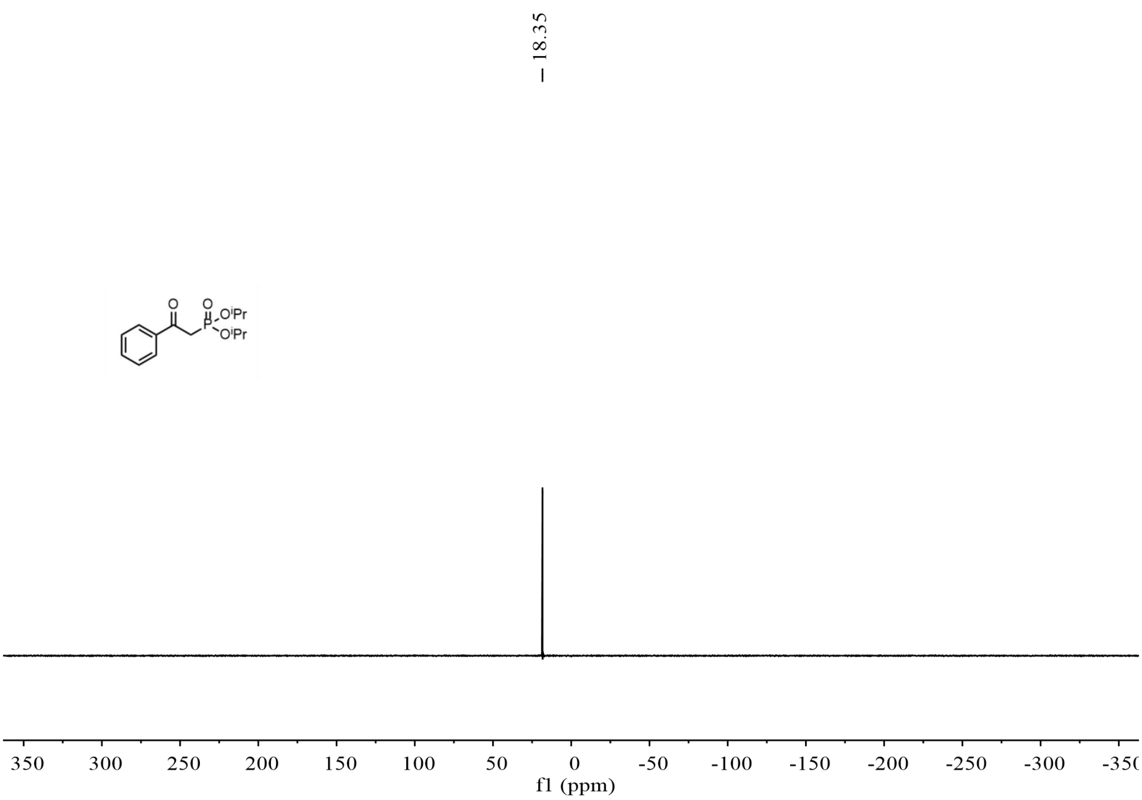
^{31}P NMR spectrum of **3s** (160 MHz, $CDCl_3$)



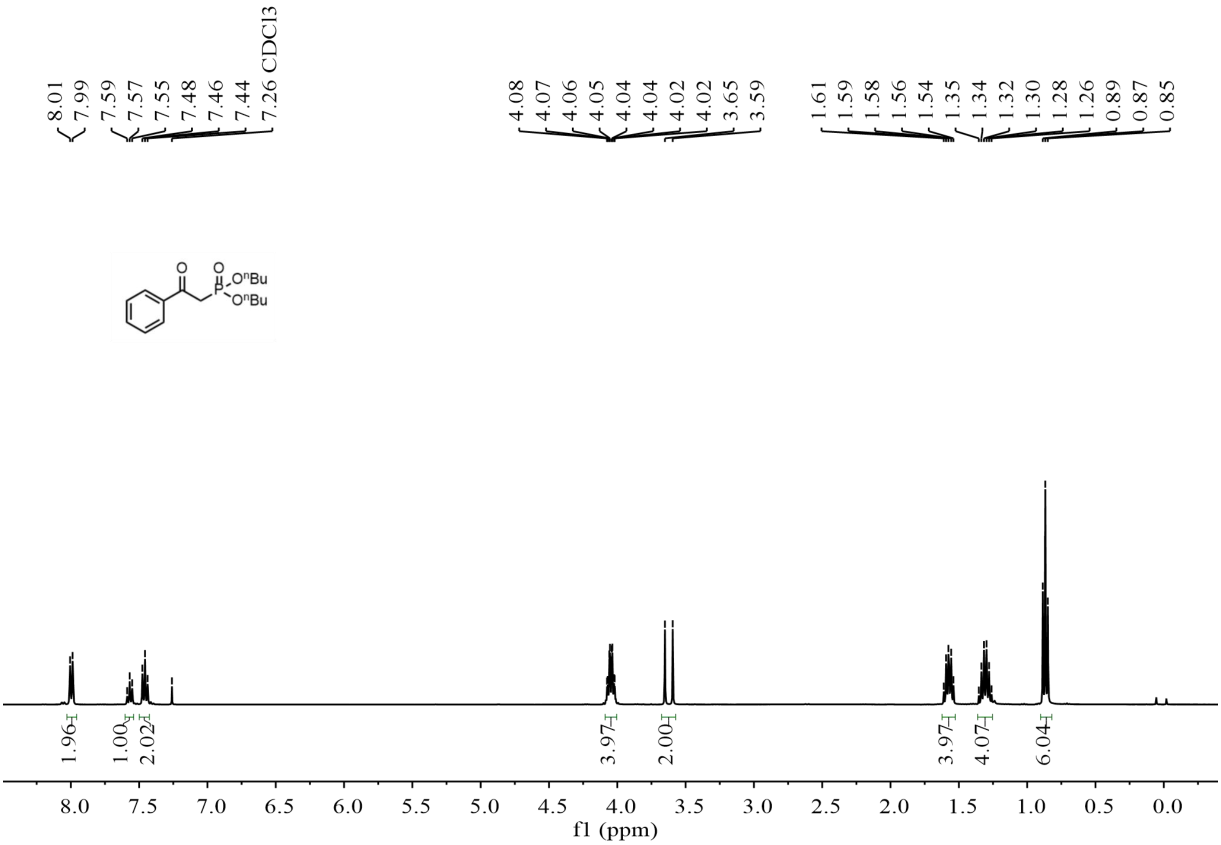
^{1}H NMR spectrum of **3t** (500 MHz, $CDCl_3$)



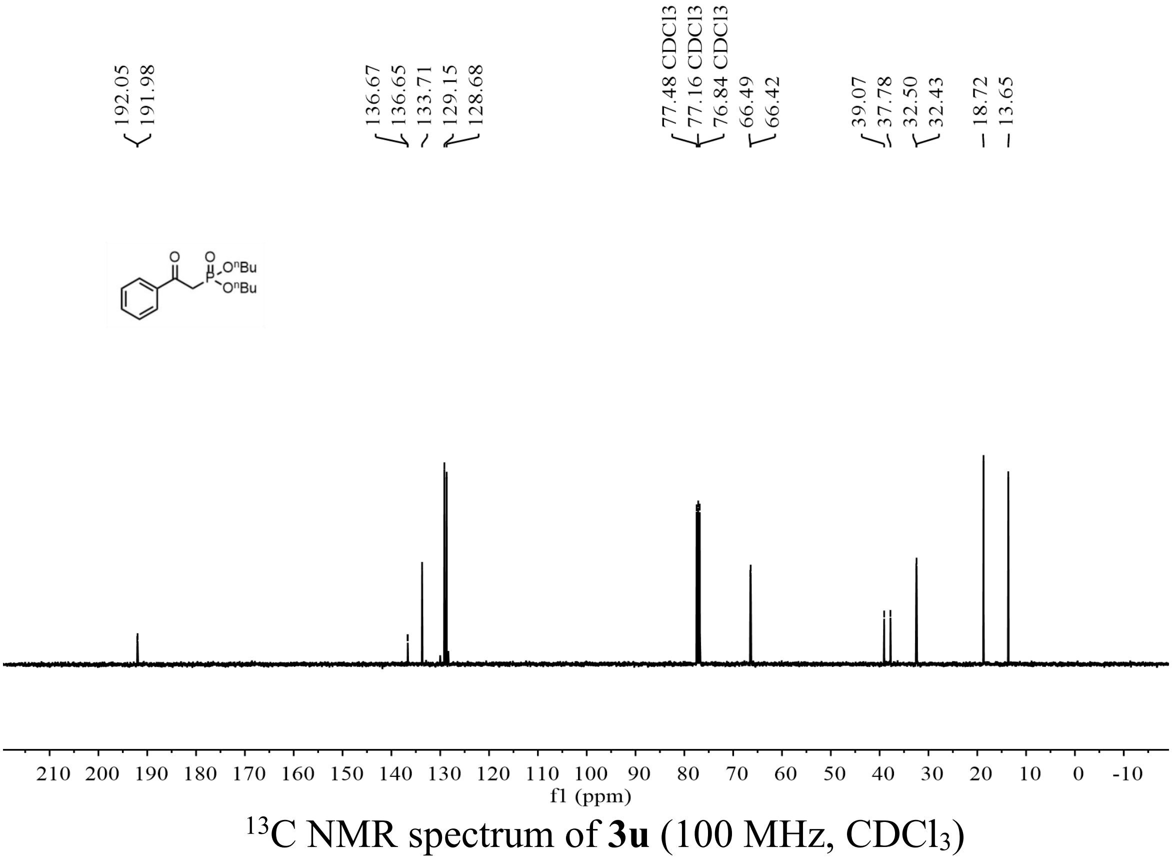
^{13}C NMR spectrum of **3t** (100 MHz, $CDCl_3$)



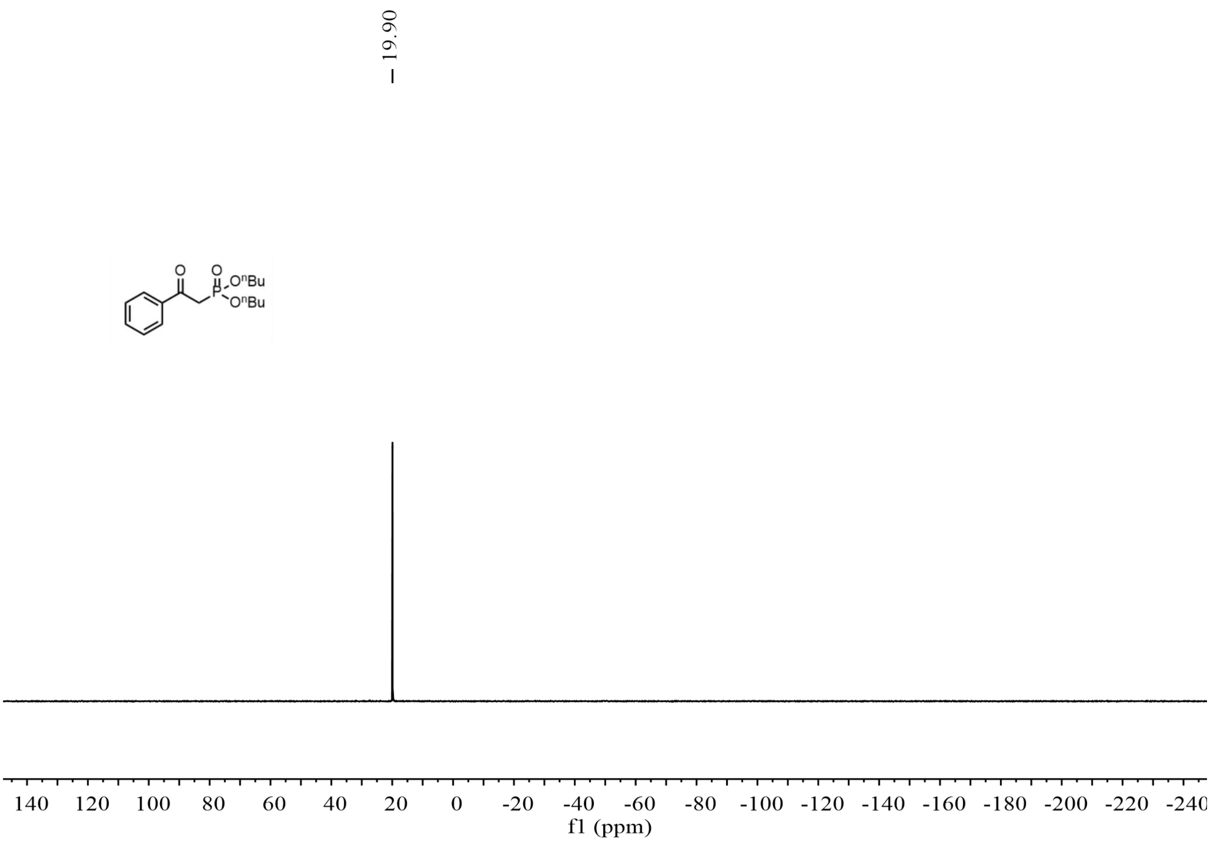
^{31}P NMR spectrum of **3t** (160 MHz, $CDCl_3$)



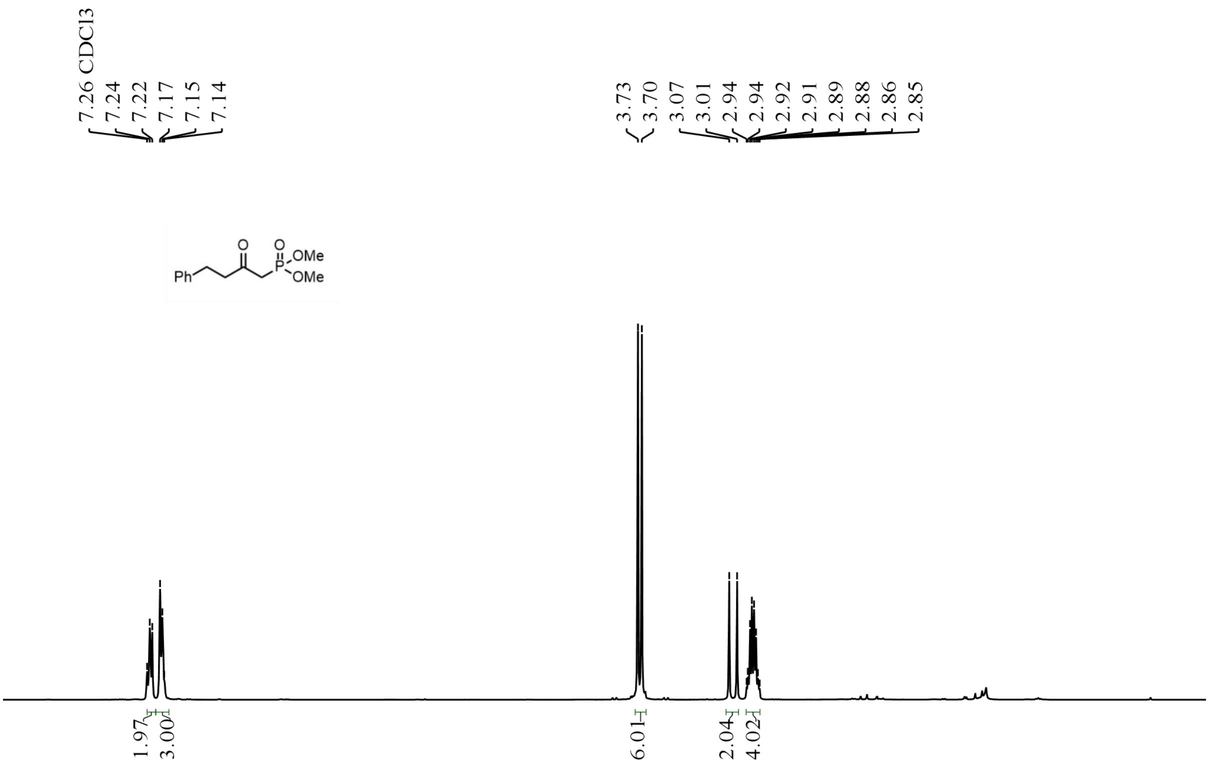
^{1}H NMR spectrum of **3u** (400 MHz, $CDCl_3$)



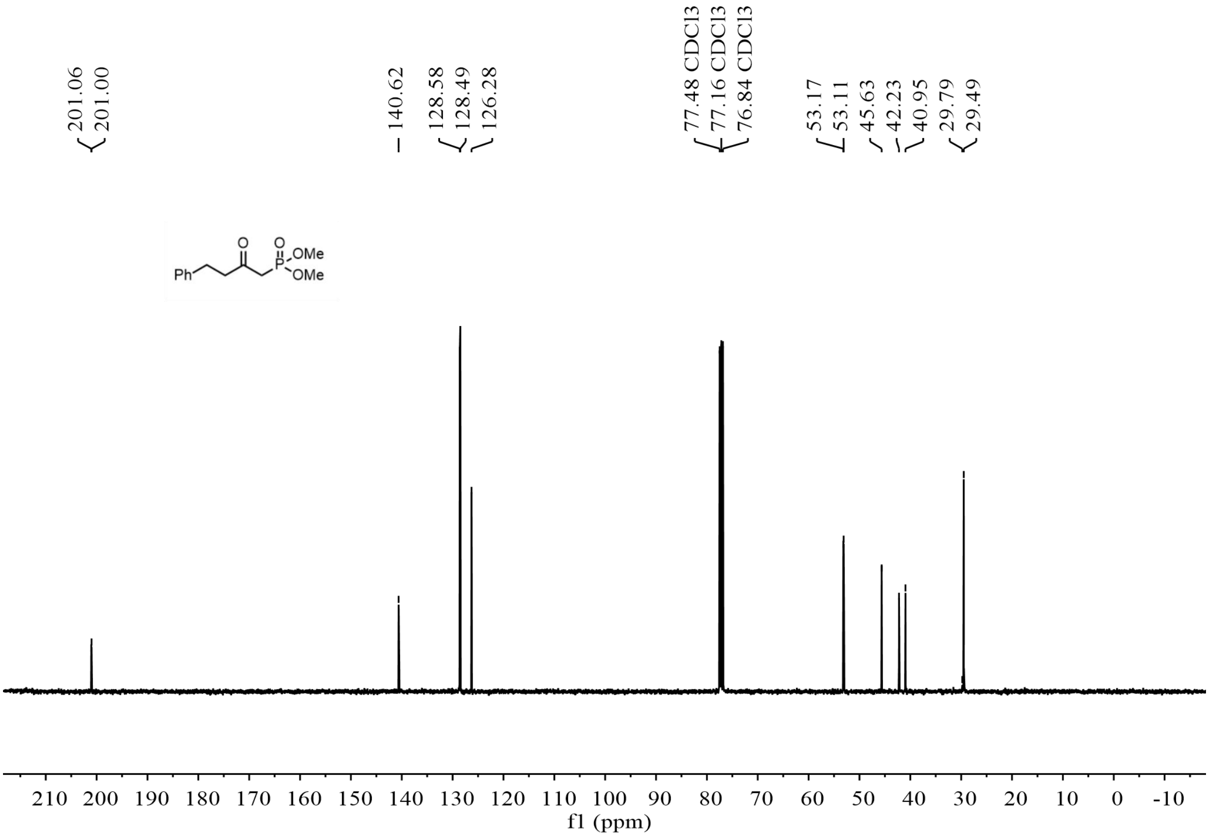
^{13}C NMR spectrum of **3u** (100 MHz, $CDCl_3$)



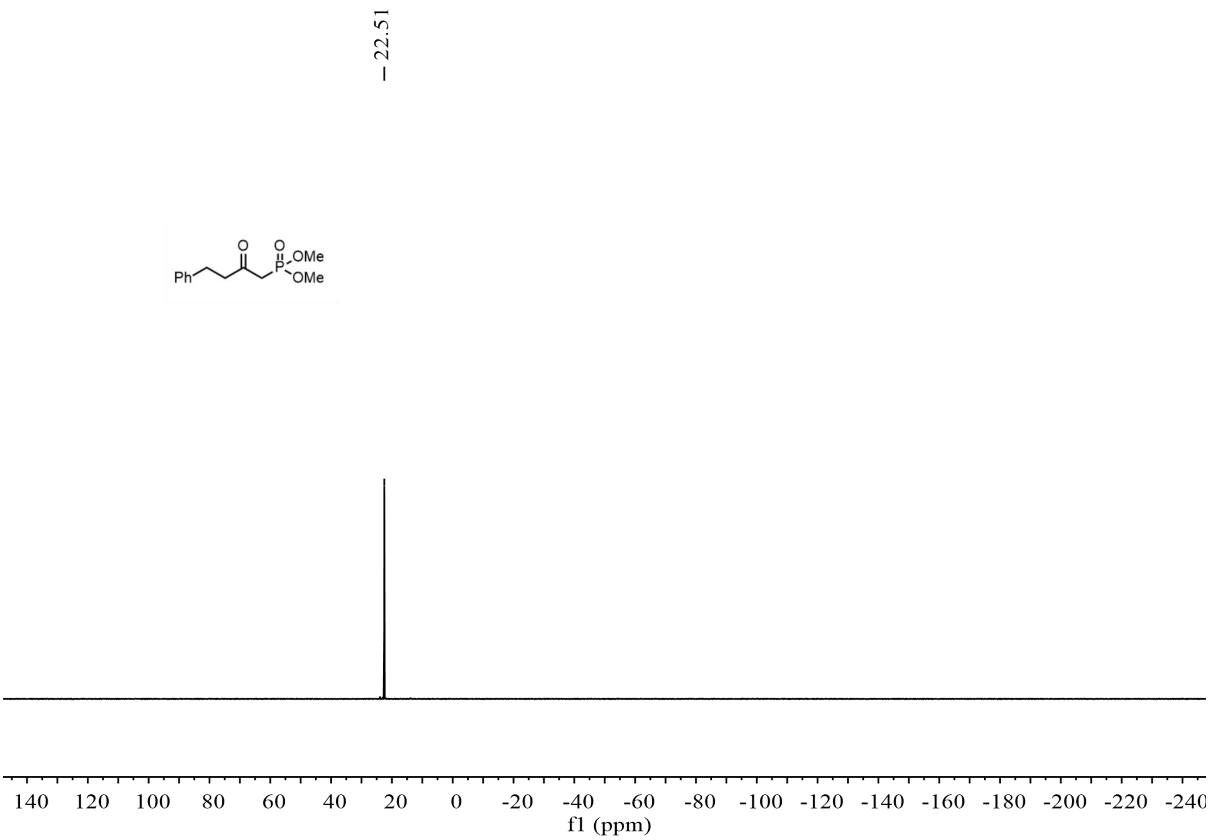
^{31}P NMR spectrum of **3u** (160 MHz, $CDCl_3$)



^{1}H NMR spectrum of **3v** (400 MHz, $CDCl_3$)



[13]C NMR spectrum of **3v** (100 MHz, $CDCl_3$)



[31]P NMR spectrum of **3v** (160 MHz, $CDCl_3$)

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