Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary Information

Copper-catalyzed aerobic oxidative coupling of ketones with P(O)-H compounds leading to β-ketophosphine oxides

Qiang Fu[a,b], Dong Yi[a,b], Zhijie Zhang[a,b], Wu Liang[a,b], Suyuan Chen[a,b], Lu Yang[a,b]

Qiang Zhang [a,]*, Jianxin Ji[a], Wei Wei[c,]*

[a] *Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, China;*

[b] *University of the Chinese Academy of Sciences, Beijing 100049, China;*

[c] *School of Chemistry and Chemical Engineering, Qufu Normal University, Qufu 273165, Shandong, China.*

* Corresponding author: weiweiqfnu@163.com (W.Wei); zhangqiang@cib.ac.cn (Q. Zhang)

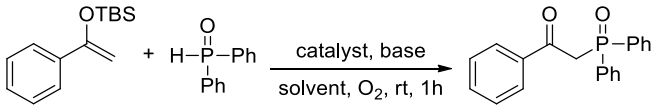
Contents

1. General information

All solvents were distilled before used by standard methods. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR were recorded in $CDCl_3$ on bruker advance Ⅲ 400 M NMR with TMS as internal standard at room temperature and chemical shifts were quoted in parts per million (ppm) downfield from tetramethylsilane. HRMS and LC-MS analyses were obtained on Bruker microTOF Q Ⅱ mass spectrometer and Waters UPLC-Xevo TQ MS (PDA Detector) / Quattro Premier XE triquadrupole mass spectrometer by ESI method respectively. Flash column chromatography was carried out on silica gel (100-200 mesh). IR spectra were obtained with a PerkinElmer Spectrum One FTIR Spectrometer. Melting points were obtained with a X - 6 melting point apparatus. Commercial reagents from TCI (Shanghai) Development Co., Ltd., Alfa Aesar and Sigma Aldrich were without further purification.

2. Screening with different parameters

Table S1 Optimization of reaction conditions[a]



1a' **2a** **3aa**

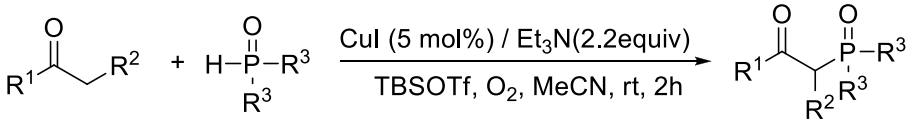
Entry	Catalyst	Base	Solvent	Yield(%)[b]
1	—	—	THF	0
2	—	TEA	THF	0
3	$AgNO_3$	TEA	THF	0
4	$FeCl_3$	TEA	THF	0
5	$InCl_3$	TEA	THF	0
6	$Pd(OAc)_2$	TEA	THF	0
7	$Mn(OAc)_2$	TEA	THF	0
8	$CuCl_2$	TEA	THF	67
9	CuCl	TEA	THF	57
10	$CuBr_2$	TEA	THF	84
11	CuBr	TEA	THF	79
12	CuI	TEA	THF	85
13	CuCN	TEA	THF	70
14	$Cu(CH_3CN)_4PF_6$	TEA	THF	42
15	$Cu(OAc)_2 \cdot H_2O$	TEA	THF	64
16	$CuSO_4$	TEA	THF	48
17	CuI	—	THF	0
18	CuI	DIPEA	THF	58
19	CuI	DBU	THF	39
20	CuI	Pyridine	THF	13
21	CuI	Na_2CO_3	THF	19
22	CuI	TEA	CH_2Cl_2	81
23	CuI	TEA	$CHCl_3$	43

24	CuI	TEA	DCE	71
25	CuI	TEA	Dioxane	79
26	CuI	TEA	MTBE	61
27	**CuI**	**TEA**	**MeCN**	**87**
28	CuI	TEA	MeCN	81[c]
29	CuI	TEA	MeCN	80[d]
30	CuI	TEA	MeCN	0[e]

[a] Reaction conditions: **1a** (0.5 mmol), **2a** (1 mmol), base (0.5 mmol), catalyst (5 mol%), solvent (5 mL), O_2 (balloon), 1h. TEA: Et_3N; DIPEA: Ethyldiisopropylamine; DBU: 1,8-diazabicyclo[5.4.0]undec-7-ene; DCE: 1,2-dichloroethane; MTBE: methyl tert-butyl ether. [b] Yields of isolated products based on **1a**. [c] CuI (2 mol%). [d] Air balloon, 8h. [e] Under N_2.

3. Typical experimental procedure

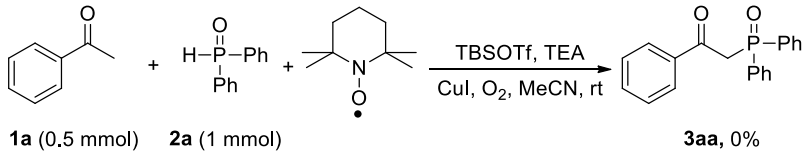
General method for oxidative coupling of ketones with P(O)-H compounds



Ketone **1** (0.5 mmol), Et_3N (1.1 mmol), TBSOTf (0.6 mmol), P(O)-H compound **2** (1.0 mmol) and CuI (5 mg, 5 mol%) were added to MeCN (5 mL) in a dried flask, the reaction mixture was allowed to stir under dioxygen balloon at room temperature for 2h. After completion of the reaction, the reaction mixture was concentrated in vaccum. The residue was purified by flash chromatography on silica gel (petroleum ether / ethyl acetate, or dichloromethane / methanol) to afford the corresponding product **3**.

4. Preliminary mechanistic studies

(1) Radical trapping experiment



Acetophenone **1a** (0.5 mmol), Et_3N (1.1 mmol), TBSOTf (0.6 mmol), diphenylphosphine oxide **2a** (1.0 mmol), TEMPO (1 mmol) and CuI (5mg, 5 mol%) were added to a dried flask with MeCN (5 mL), then the reaction mixture was stirred under dioxygen at room temperature for 3h. $Ph_2P(O)$-TEMPO was detected by LC-MS and none of the desired product **3aa** was detected in the reaction mixture.

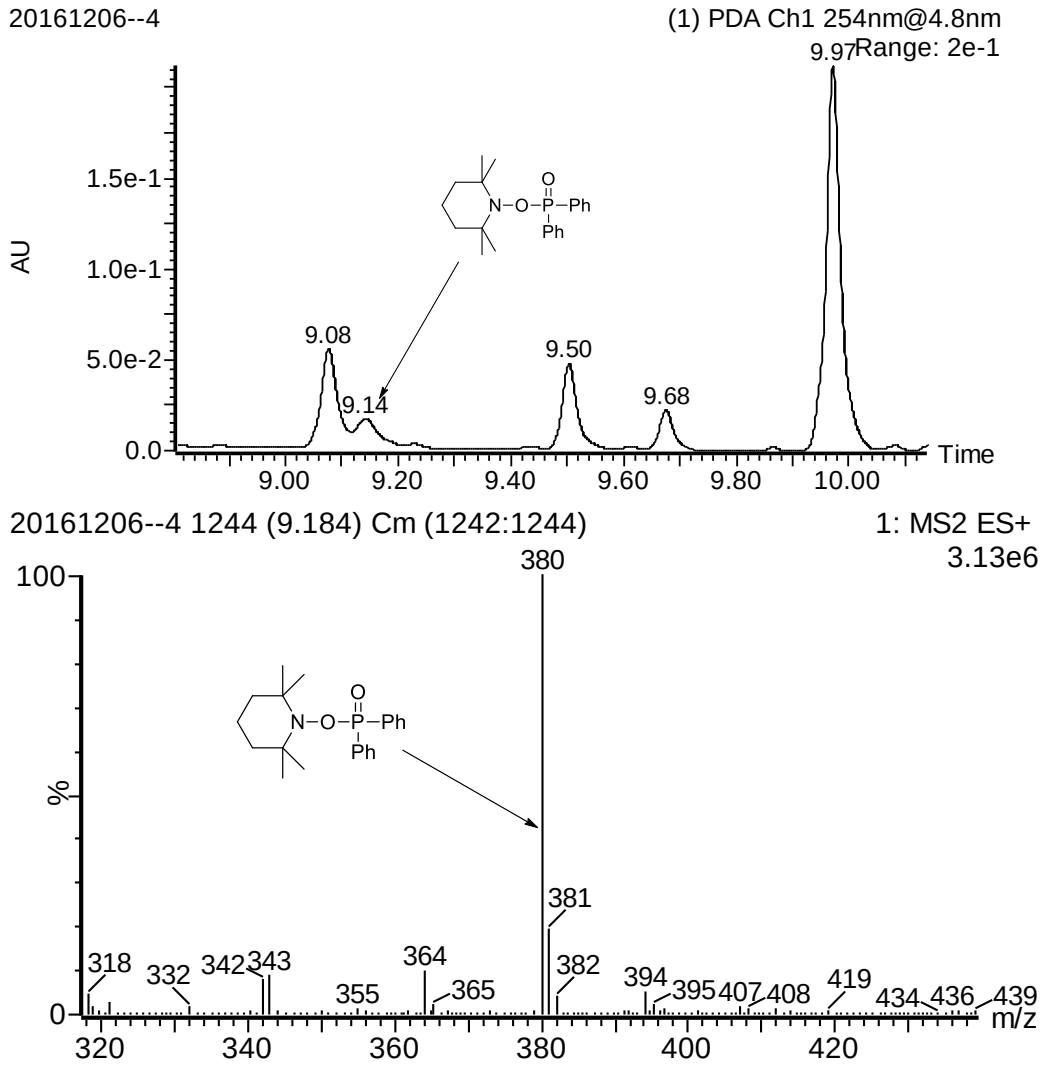
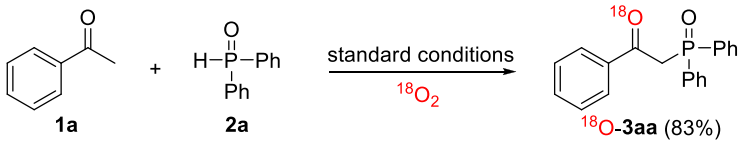


Figure S1. Copies of LC-MS spectra

(2) $^{18}O_2$-labeling experiment using $^{18}O_2$



Acetophenone (**1a**) (0.5 mmol), Et_3N (1.1 mmol), TBSOTf (0.6 mmol), diphenylphosphine oxide (1.0 mmol) and CuI (5mg, 5 mol%) were added to a dried flask with MeCN (5 mL), the reaction mixture was allowed to stir under ^{18}O-labeled dioxygen (balloon) at room temperature for 2h. After completion of the reaction, the reaction mixture was concentrated in vaccum. The residue was purified by flash

chromatography on silica gel (petroleum ether / ethyl acetate = 1:1) to afford the β-ketophosphine oxide product ^{18}O-**3aa** in 83% yield. Moreover, the side-product TBSOH was detected by HRMS.

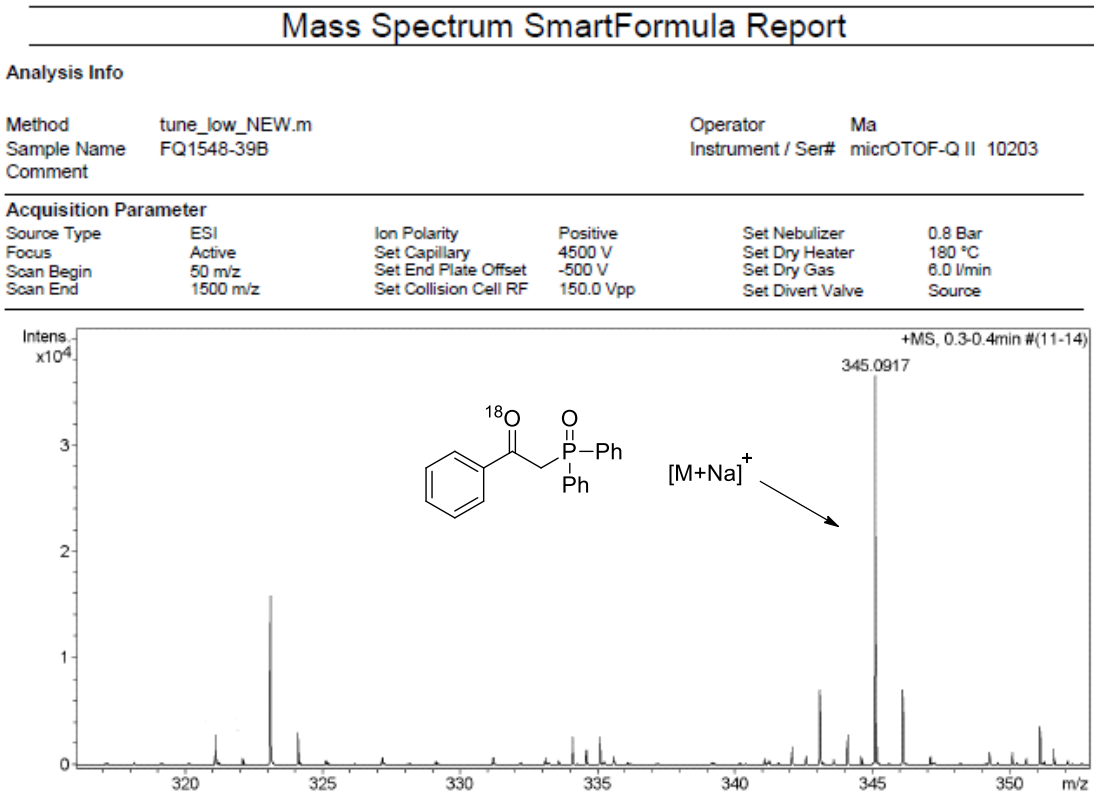


Figure S2.Copy of HRMS spectra of product ^{18}O-3aa

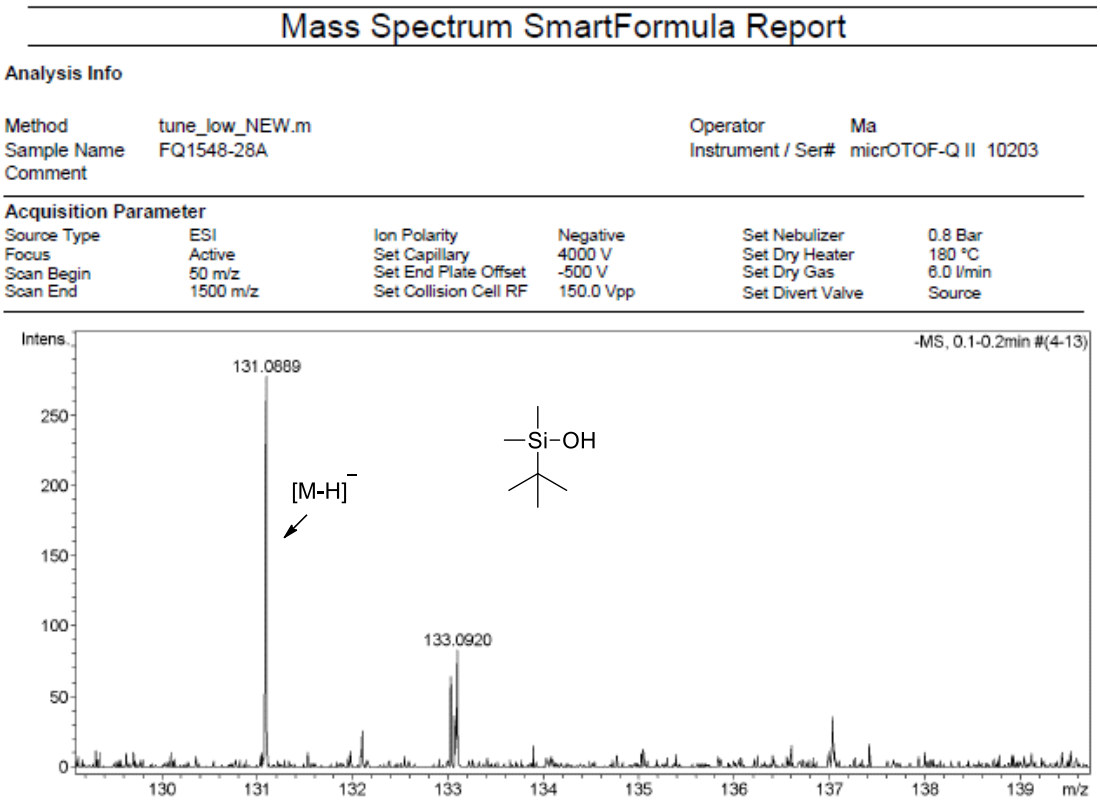
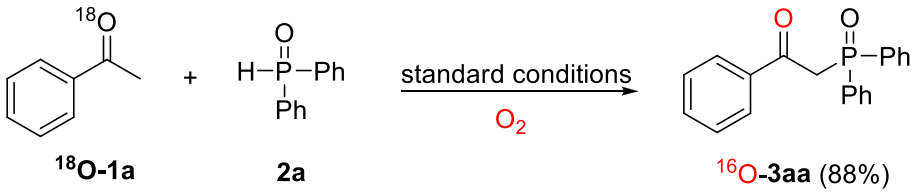


Figure S3. Copy of HRMS spectra of side-product TBSOH

(3) Isotope-labeling experiment using ^{18}O-labled acetophenone



^{18}O-labled acetophenone (prepared according the reference[1]) (^{18}O-**1a**) (0.5 mmol), Et_3N (1.1 mmol), TBSOTf (0.6 mmol), diphenylphosphine oxide **2a** (1.0 mmol) and CuI (5mg, 5 mol%) were added to a dried flask with MeCN (5 mL), the reaction mixture was allowed to stir under dioxygen (balloon) at room temperature for 2h. After completion of the reaction, the reaction mixture was concentrated in vaccum. The residue was purified by flash chromatography on silica gel (petroleum ether / ethyl acetate =1:1) to afford the β-ketophosphine oxide product ^{16}O-**3aa** in 88% yield.

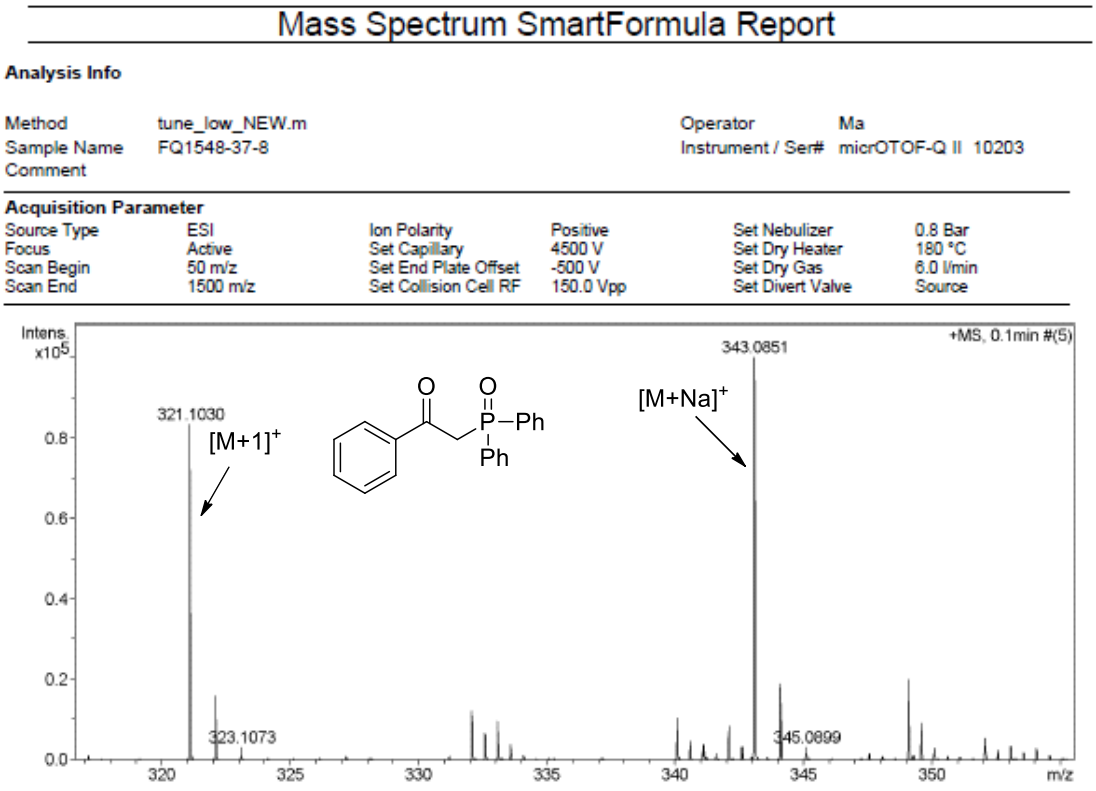
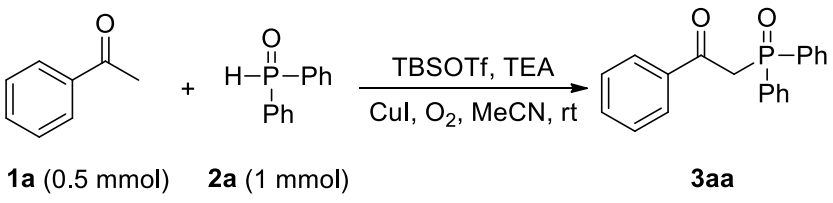


Figure S4. Copy of HRMS spectra of the product ^{16}O-**3aa**

(4) LC-MS analysis of the reaction mixture of model reaction

The model reaction was conducted at room temperature for 6 minutes. Then, the reaction mixture (1 mL) was taken to centrifugal tube and was analyzed by LC-MS using the following method: HPLC: UPLC: ACQUITY BEH C18 column, 2.1×100 mm, 1.7 μm; PDA detector: 254 nm; 25 °C. flow rate: 0.3 mL/min; The mobile phase consisted of A (water(0.1% formic acid)) and B (acetonitrile), gradient elution: B(acetonitrile): 5%-95%; run time: 10 min. MS: Capillary +2.5 kV; Cone 30 V; Source temperature 150 °C; Desolvation temperature 500 °C; Cone gas flow OFF; Desolvation gas flow 500 L/h. Scan 100-700 m/z.

Copies of LC-MS spectra



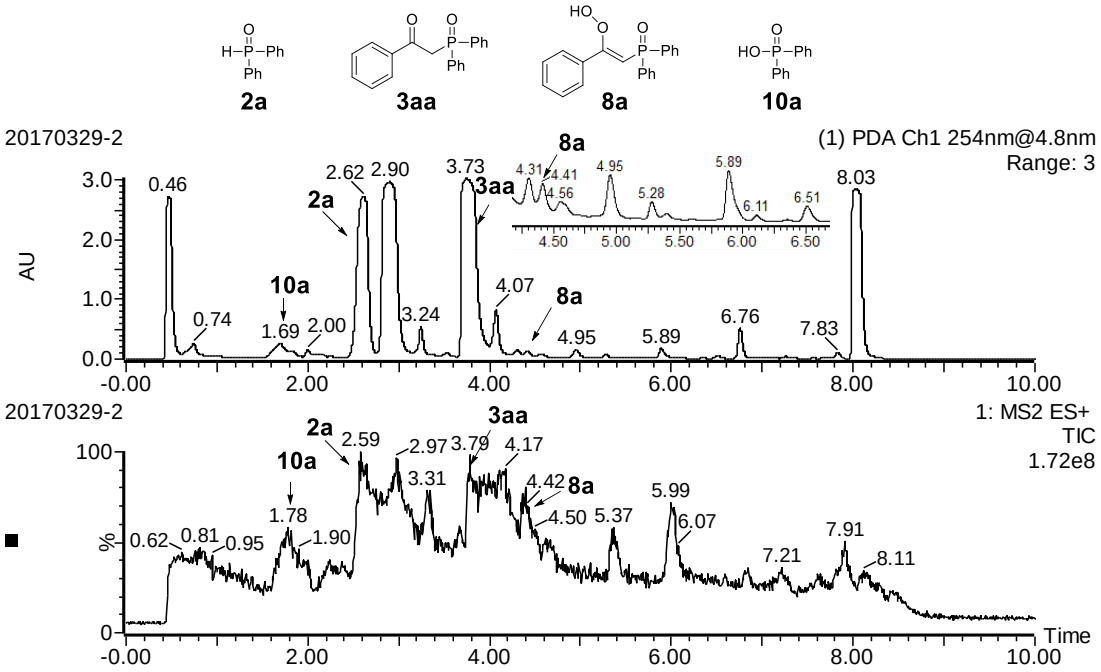


Figure S5. The analysis of LC-MS spectra for the reaction

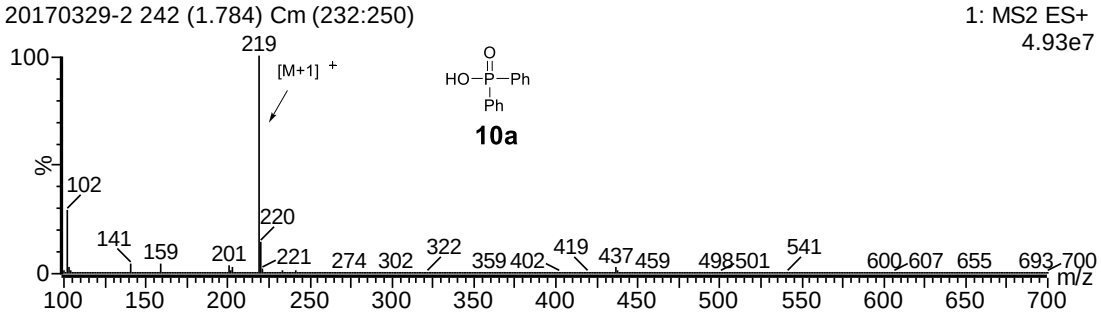


Figure S6. The MS spectrum corresponding to the peak in LC spectrum at the retention time of 1.69 min.

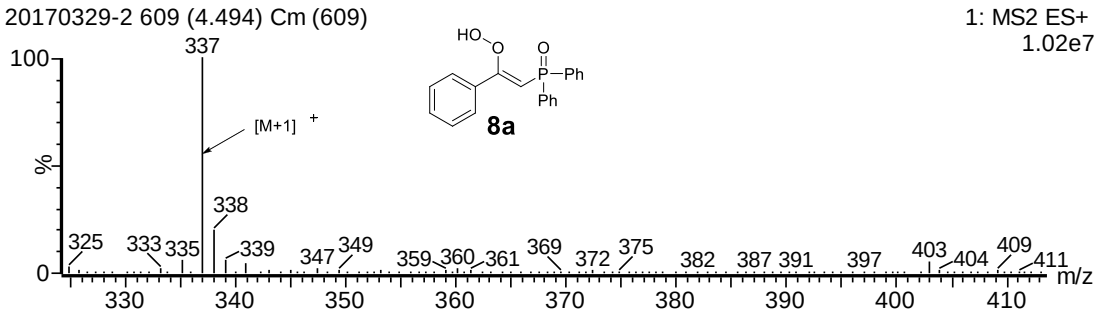


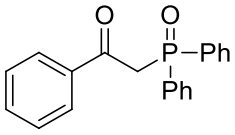
Figure S7. The MS spectrum corresponding to the peak in LC spectrum at the retention time of 4.41 min.

Reference

1. X. Creary and P. A. Inocencio, *J. Am. Chem. Soc.*, 1986, **108**, 5979-5983.

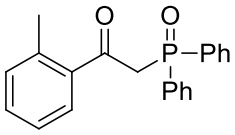
5. Characterization data of products

2-(diphenylphosphoryl)-1-phenylethanone (3aa)



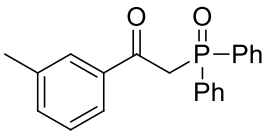
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.96 (m, 2H), 7.85 – 7.77 (m, 4H), 7.56 – 7.49 (m, 3H), 7.49 – 7.36 (m, 6H), 4.15 (d, *J* = 15.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.84 (d, *J* = 5.6 Hz), 136.96, 133.63, 132.19 (d, *J* = 2.8 Hz), 131.97 (d, *J* = 103.3 Hz), 131.12 (d, *J* = 9.8 Hz), 129.26, 128.66 (d, *J* = 12.3 Hz), 128.56, 43.27 (d, *J* = 58.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.92. HRMS (ESI) calcd for $C_{20}H_{17}NaO_2P$ $[M+Na]^+$: 343.0858; found: 343.0860.

2-(diphenylphosphoryl)-1-(o-tolyl)ethanone (3ba)



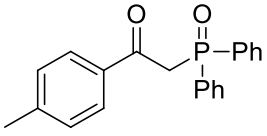
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 7.7, 0.8 Hz, 1H), 7.85 – 7.76 (m, 4H), 7.57 – 7.50 (m, 2H), 7.50 – 7.42 (m, 4H), 7.35 (td, *J* = 7.5, 1.2 Hz, 1H), 7.25 (t, *J* = 7.4 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 4.15 (d, *J* = 15.1 Hz, 2H), 2.32 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.71 (d, *J* = 5.6 Hz), 139.01, 137.42, 132.14 (d, *J* = 2.8 Hz), 132.09 (d, *J* = 103.2 Hz), 132.00, 131.86, 131.11 (d, *J* = 9.8 Hz), 130.34, 128.65 (d, *J* = 12.3 Hz), 125.80, 45.61 (d, *J* = 58.7 Hz), 21.31. ^{31}P NMR(162 MHz, $CDCl_3$) δ 27.31. HRMS (ESI) calcd for $C_{21}H_{19}NaO_2P$ $[M+Na]^+$: 357.1015; found: 357.1019.

2-(diphenylphosphoryl)-1-(m-tolyl)ethanone (3ca)



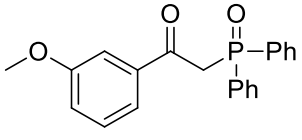
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.76 (m, 5H), 7.73 (s, 1H), 7.55 – 7.41 (m, 6H), 7.37 – 7.24 (m, 2H), 4.14 (d, *J* = 15.4 Hz, 2H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.99 (d, *J* = 5.6 Hz), 138.28, 137.00, 134.41, 132.14 (d, *J* = 2.8 Hz), 132.05 (d, *J* = 103.3 Hz), 131.14 (d, *J* = 9.8 Hz), 129.55, 128.61 (d, *J* = 12.3 Hz), 128.44, 126.59, 43.21 (d, *J* = 58.4 Hz), 21.32. ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.98. HRMS (ESI) calcd for $C_{21}H_{19}NaO_2P$ $[M+Na]^+$: 357.1015; found: 357.1008.

2-(diphenylphosphoryl)-1-(p-tolyl)ethanone (3da)



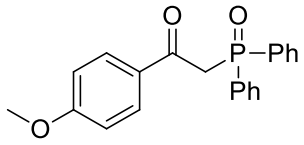
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.85 – 7.75 (m, 4H), 7.56 – 7.38 (m, 6H), 7.20 (d, *J* = 8.1 Hz, 2H), 4.12 (d, *J* = 15.3 Hz, 2H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.34 (d, *J* = 5.6 Hz), 144.62, 134.55, 132.13 (d, *J* = 2.8 Hz), 132.06 (d, *J* = 103.2 Hz), 131.12 (d, *J* = 9.8 Hz), 129.43, 129.25, 128.62 (d, *J* = 12.3 Hz), 43.15 (d, *J* = 58.3 Hz), 21.72. ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.01. HRMS (ESI) calcd for $C_{21}H_{19}NaO_2P$ $[M+Na]^+$: 357.1015; found: 357.1011.

2-(diphenylphosphoryl)-1-(3-methoxyphenyl)ethanone (3ea)



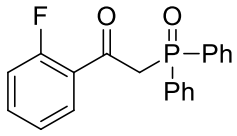
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.75 (m, 4H), 7.58 (d, *J* = 7.8 Hz, 1H), 7.53 – 7.39 (m, 7H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.06 (dd, *J* = 8.0, 2.3 Hz, 1H), 4.13 (d, J = 15.2 Hz, 2H), 3.77 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.67 (d, *J* = 5.7 Hz), 159.66, 138.29, 132.19 (d, *J* = 2.8 Hz), 131.95 (d, *J* = 103.3 Hz), 131.11 (d, *J* = 9.8 Hz), 129.58, 128.66 (d, *J* = 12.3 Hz), 122.22, 120.54, 112.75, 55.42, 43.30 (d, *J* = 58.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.02. HRMS (ESI) calcd for $C_{21}H_{20}O_3P$ $[M+H]^+$: 351.1145; found: 351.1151.

2-(diphenylphosphoryl)-1-(4-methoxyphenyl)ethanone (3fa)



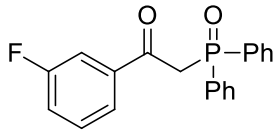
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.95 (m, 2H), 7.86 – 7.75 (m, 4H), 7.57 – 7.42 (m, 6H), 6.89 (dd, *J* = 9.4, 2.3 Hz, 2H), 4.10 (d, *J* = 15.3 Hz, 2H), 3.84 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.06 (d, *J* = 5.5 Hz), 163.97, 132.13 (d, *J* = 2.8 Hz), 132.05 (d, *J* = 103.3 Hz), 131.80, 131.13 (d, *J* = 9.8 Hz), 130.12, 128.62 (d, *J* = 12.3 Hz), 113.72, 55.53, 43.14 (d, *J* = 58.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.12. HRMS (ESI) calcd for $C_{21}H_{19}NaO_3P$ $[M+Na]^+$: 373.0964; found : 373.0969.

2-(diphenylphosphoryl)-1-(2-fluorophenyl)ethanone (3ga)



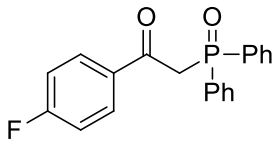
White solid; mp: 89.5-91.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.74 (m, 4H), 7.71 (m, 1H), 7.55 – 7.41 (m, 7H), 7.19 – 7.07 (m, 1H), 7.02 (dd, *J* = 11.1, 8.4 Hz, 1H), 4.25 (d, *J* = 14.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.84 (dd, *J* = 6.0, 2.8 Hz), 161.61 (d, *J* = 254.8 Hz), 135.18 (d, *J* = 9.3 Hz), 132.13 (d, *J* = 103.3 Hz), 132.13 (d, *J* = 2.8 Hz), 131.08 (d, *J* = 9.8 Hz), 130.95 (d, *J* = 1.7 Hz), 128.62 (d, *J* = 12.3 Hz), 126.09 (d, *J* = 11.5 Hz), 124.50 (d, *J* = 3.4 Hz), 116.61 (d, *J* = 23.7 Hz), 46.69 (dd, *J* = 59.3, 7.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.75. ^{19}F NMR (376 MHz, $CDCl_3$) δ -109.27 (d, *J* = 1.2 Hz). IR (cm^{-1}, KBr): 1676 (C=O), 1183 (P=O). HRMS (ESI) calcd for $C_{20}H_{17}FO_2P$ $[M+H]^+$: 339.0945; found: 339.0943.

2-(diphenylphosphoryl)-1-(3-fluorophenyl)ethanone (3ha)



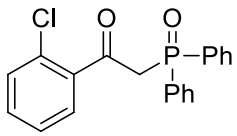
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.76 (m, 5H), 7.68 – 7.61 (m, 1H), 7.57 – 7.37 (m, 7H), 7.23 (m, 1H), 4.13 (d, *J* = 15.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.71 (dd, *J* = 5.7, 2.2 Hz), 162.63 (d, *J* = 248.0 Hz), 138.99 (d, *J* = 6.4 Hz), 132.31 (d, *J* = 2.8 Hz), 131.71 (d, *J* = 103.6 Hz), 131.07 (d, *J* = 9.8 Hz), 130.26 (d, *J* = 7.6 Hz), 128.71 (d, *J* = 12.4 Hz), 125.37 (d, *J* = 3.0 Hz), 120.66 (d, *J* = 21.5 Hz), 115.64 (d, *J* = 22.6 Hz), 43.53 (d, *J* = 57.2 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.75. ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.71. HRMS (ESI) calcd for $C_{20}H_{16}NaFO_2P$ $[M+Na]^+$: 361.0764; found: 361.0750.

2-(diphenylphosphoryl)-1-(4-fluorophenyl)ethanone (3ia)



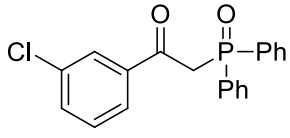
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 8.03 (m, 2H), 7.86 – 7.77 (m, 4H), 7.58 – 7.44 (m, 6H), 7.09 (m, 2H), 4.12 (d, *J* = 15.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.24 (d, *J* = 5.6 Hz), 166.09 (d, *J* = 256.1 Hz), 133.43 (d, *J* = 2.8 Hz), 132.29 (d, *J* = 2.7 Hz), 132.19 (d, *J* = 9.6 Hz), 131.77 (d, *J* = 101.3 Hz), 131.08 (d, *J* = 9.8 Hz), 128.71 (d, *J* = 12.4 Hz), 115.69 (d, *J* = 22.0 Hz), 43.57 (d, *J* = 56.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.74. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.07. HRMS (ESI) calcd for $C_{20}H_{16}NaFO_2P$ $[M+Na]^+$: 361.0764; found: 361.0762.

1-(2-chlorophenyl)-2-(diphenylphosphoryl)ethanone (3ja)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.72 (m, 4H), 7.57 – 7.40 (m, 7H), 7.36 – 7.27 (m, 2H), 7.26 – 7.18 (m, 1H), 4.25 (d, *J* = 14.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.96 (d, *J* = 5.9 Hz), 138.96, 132.24, 132.21 (d, *J* = 2.9 Hz), 131.85 (d, *J* = 103.2 Hz), 131.04 (d, *J* = 9.9 Hz), 130.91, 130.28, 130.03, 128.68 (d, *J* = 12.4 Hz), 127.04, 46.66 (d, *J* = 57.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.89. HRMS (ESI) calcd for $C_{20}H_{16}ClNaO_2P$ $[M+Na]^+$: 377.0469; found: 377.0459.

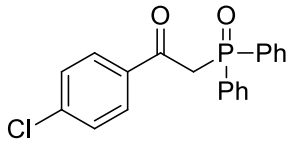
1-(3-chlorophenyl)-2-(diphenylphosphoryl)ethanone (3ka)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.86 (m, 2H), 7.85 – 7.72 (m, 4H), 7.58 – 7.40 (m, 7H), 7.35 (t, *J* = 7.8 Hz, 1H), 4.12 (d, *J* = 15.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.71 (d, *J* = 5.7 Hz),

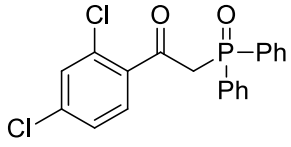
138.44, 134.82, 133.48, 132.32 (d, *J* = 2.8 Hz), 131.70 (d, *J* = 103.5 Hz), 131.07 (d, *J* = 9.8 Hz), 129.91, 128.96, 128.72 (d, *J* = 12.3 Hz), 127.67, 43.49 (d, *J* = 57.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.63. HRMS (ESI) calcd for $C_{20}H_{16}ClNaO_2P$ $[M+Na]^+$: 377.0469; found: 377.0474.

1-(4-chlorophenyl)-2-(diphenylphosphoryl)ethanone (3la)



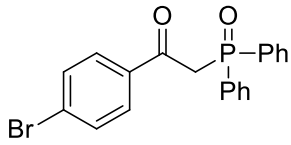
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.6 Hz, 2H), 7.87 – 7.73 (m, 4H), 7.60 – 7.43 (m, 6H), 7.39 (d, *J* = 8.6 Hz, 2H), 4.12 (d, *J* = 15.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.68 (d, *J* = 5.7 Hz), 140.24, 135.28, 132.32 (d, *J* = 2.8 Hz), 131.73 (d, *J* = 103.5 Hz), 131.07 (d, *J* = 9.8 Hz), 130.80, 128.86, 128.73 (d, *J* = 12.3 Hz), 43.57 (d, *J* = 56.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.71. HRMS (ESI) calcd for $C_{20}H_{16}O_2P$ $[M+Na]^+$:377.0453.

1-(2,4-dichlorophenyl)-2-(diphenylphosphoryl)ethanone (3ma)



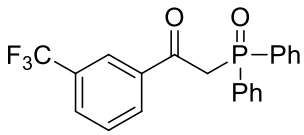
White solid; mp: 87.8-88.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.70 (m, 4H), 7.51 (m, 7H), 7.31 (d, *J* = 1.8 Hz, 1H), 7.22 (d, *J* = 8.1 Hz, 1H), 4.21 (d, *J* = 14.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.76 (d, *J* = 6.0 Hz), 137.92, 137.12, 132.31 (d, *J* = 2.6 Hz), 132.06, 131.66 (d, *J* = 103.8 Hz), 131.27, 130.99 (d, *J* = 9.8 Hz), 130.19, 128.75 (d, *J* = 12.3 Hz), 127.43, 46.74 (d, *J* = 56.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.95. IR (cm^{-1}, KBr): 1670 (C=O), 1186 (P=O). HRMS (ESI) calcd for $C_{20}H_{15}Cl_2NaO_2P$ $[M+Na]^+$: 411.0079; found: 411.0081.

1-(4-bromophenyl)-2-(diphenylphosphoryl)ethanone (3na)



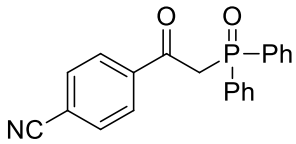
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.5 Hz, 2H), 7.80 (dd, *J* = 12.1, 7.4 Hz, 4H), 7.54 (m, 4H), 7.47 (m, 4H), 4.11 (d, *J* = 15.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.89 (d, *J* = 5.6 Hz), 135.67, 132.32 (d, *J* = 2.9 Hz), 131.86, 131.71 (d, *J* = 103.5 Hz), 131.06 (d, *J* = 9.8 Hz), 130.88, 129.13, 128.74 (d, *J* = 12.3 Hz), 43.55 (d, *J* = 56.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.68. HRMS (ESI) calcd for $C_{20}H_{16}BrNaO_2P$ $[M+Na]^+$: 420.9963; found : 420.9973.

2-(diphenylphosphoryl)-1-(3-(trifluoromethyl)phenyl)ethanone (3oa)



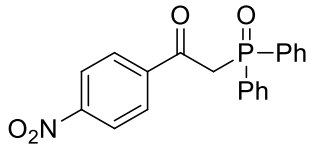
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 7.9 Hz, 1H), 8.18 (s, 1H), 7.87 – 7.75 (m, 5H), 7.62 – 7.45 (m, 7H), 4.18 (d, *J* = 15.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.74 (d, *J* = 5.7 Hz), 137.40 , 132.80 , 132.38 (d, *J* = 2.8 Hz), 131.61 (d, *J* = 102.1 Hz), 131.07 (q, *J* = 33.0 Hz), 131.06 (d, *J* = 9.8 Hz), 129.89 (q, *J* = 3.5 Hz), 129.29, 128.75 (d, *J* = 12.4 Hz), 125.77 (q, *J* = 3.8 Hz), 123.57 (q, *J* = 272.7 Hz), 43.63 (d, *J* = 56.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.65. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.79. HRMS (ESI) calcd for $C_{21}H_{16}F_3NaO_2P$ $[M+H]^+$: 389.0913; found: 389.0921.

4-(2-(diphenylphosphoryl)acetyl)benzonitrile (3pa)



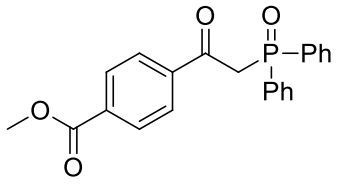
^{1}H NMR (400 MHz, CDCl3) δ 8.15 (d, J = 8.1 Hz, 2H), 7.80 (m, 4H), 7.74 (d, J = 8.1 Hz, 2H), 7.61 – 7.54 (m, 2H), 7.54 – 7.43 (m, 4H), 4.16 (d, J = 15.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.81 (d, J = 5.2 Hz), 139.77, 132.51 (d, J = 2.4 Hz), 132.37, 131.40 (d, J = 104.1 Hz), 131.02 (d, J = 9.9 Hz), 129.78, 128.83 (d, J = 12.4 Hz), 117.94, 116.70, 44.06 (d, J = 55.2 Hz). ^{31}P NMR (162 MHz, CDCl3) δ 26.62. HRMS (ESI) calcd for $C_{21}H_{16}NNaO_2P$ $[M+Na]^+$: 368.0811; found : 368.0813.

2-(diphenylphosphoryl)-1-(4-nitrophenyl)ethanone (3qa)



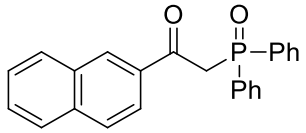
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (m, 4H), 7.86 – 7.77 (m, 4H), 7.62 – 7.55 (m, 2H), 7.54 – 7.45 (m, 4H), 4.20 (d, *J* = 15.0 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.69 (d, *J* = 5.5 Hz), 150.43 , 141.19, 132.54 (d, *J* = 2.4 Hz), 131.30 (d, *J* = 103.9 Hz), 131.02 (d, *J* = 9.8 Hz), 130.43, 128.85 (d, *J* = 12.4 Hz), 123.68 , 44.21 (d, *J* = 55.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.75. HRMS (ESI) calcd for $C_{20}H_{16}NNaO_4P$ $[M+H]^+$: 366.0890; found: 366.0905.

methyl 4-(2-(diphenylphosphoryl)acetyl)benzoate (3ra)



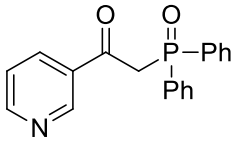
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 7.99 (m, 4H), 7.84 – 7.74 (m, 4H), 7.55 – 7.49 (m, 2H), 7.45 (m, 4H), 4.17 (d, *J* = 15.2 Hz, 2H), 3.91 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.52 (d, *J* = 5.7 Hz), 166.12, 140.06, 134.12, 132.33 (d, *J* = 2.8 Hz), 131.70 (d, *J* = 103.5 Hz), 131.06 (d, *J* = 9.8 Hz), 129.70, 129.16, 128.73 (d, J = 12.4 Hz), 52.51, 43.66 (d, J = 57.0 Hz). ^{31}P NMR (162 MHz, CDCl3) δ 26.71. HRMS (ESI) calcd for $C_{16}H_{17}NaO_3P$ $[M+Na]^+$: 311.0808; found: 311.0822.

2-(diphenylphosphoryl)-1-(naphthalen-2-yl)ethanone (3sa)



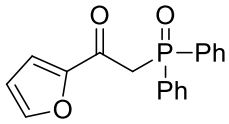
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 8.02 – 7.93 (m, 2H), 7.91 – 7.80 (m, 6H), 7.63 – 7.42 (m, 8H), 4.29 (d, *J* = 15.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.69 (d, *J* = 5.6 Hz), 135.72, 134.29, 132.33, 132.22 (d, *J* = 2.8 Hz), 131.99, 131.93 (d, *J* = 103.4 Hz), 131.16 (d, *J* = 9.8 Hz), 129.99, 128.90, 128.68 (d, *J* = 12.3 Hz), 128.38, 127.68, 126.79, 124.12, 43.33 (d, *J* = 58.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.31. HRMS (ESI) calcd for $C_{24}H_{19}NaO_2P$ $[M+Na]^+$: 393.1015; found: 393.1005.

2-(diphenylphosphoryl)-1-(pyridin-3-yl)ethanone (3ta)



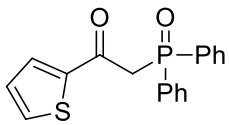
White solid; mp: 125.3-128.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.11 (s, 1H), 8.70 (d, *J* = 3.8 Hz, 1H), 8.30 (m, 1H), 7.83 – 7.73 (m, 4H), 7.55 – 7.39 (m, 6H), 7.34 (m, 1H), 4.14 (d, *J* = 15.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.98 (d, *J* = 5.8 Hz), 153.70, 150.39, 136.69, 132.39 (d, *J* = 2.9 Hz), 132.21, 131.56 (d, *J* = 102.5 Hz), 131.00 (d, *J* = 9.9 Hz), 128.76 (d, *J* = 12.4 Hz), 123.41, 43.65 (d, *J* = 56.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.62. IR (cm^{-1}, KBr):1682 (C=O), 1181 (P=O). HRMS (ESI) calcd for $C_{19}H_{17}NO_2P$ $[M+H]^+$: 322.0991; found: 322.0998.

2-(diphenylphosphoryl)-1-(furan-2-yl)ethanone (3ua)



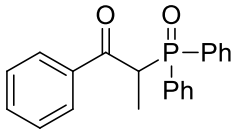
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.75 (m, 4H), 7.56 – 7.41 (m, 7H), 7.28 (d, *J* = 3.6 Hz, 1H), 6.47 (dd, *J* = 3.6, 1.7 Hz, 1H), 4.01 (d, *J* = 15.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.50 (d, *J* = 5.4 Hz), 152.49, 147.29, 132.16 (d, *J* = 2.8 Hz), 131.84 (d, *J* = 103.4 Hz), 131.07 (d, *J* = 9.9 Hz), 128.59 (d, *J* = 12.3 Hz), 119.64, 112.77, 43.04 (d, *J* = 57.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.92. HRMS (ESI) calcd for $C_{18}H_{15}NaO_3P$ $[M+Na]^+$: 333.0651; found: 333.0642.

2-(diphenylphosphoryl)-1-(thiophen-2-yl)ethanone (3va)



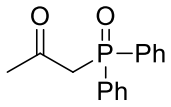
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 3.8, 0.9 Hz, 1H), 7.87 – 7.76 (m, 4H), 7.64 (dd, *J* = 4.9, 0.9 Hz, 1H), 7.59 – 7.52 (m, 2H), 7.52 – 7.43 (m, 4H), 7.10 (dd, *J* = 4.8, 4.0 Hz, 1H), 4.07 (d, *J* = 15.3 Hz, 2H). ^{13}C NMR (101 MHz, CDCl3) δ 184.99 (d, J = 5.3 Hz), 144.44, 135.29, 135.09, 132.26 (d, J = 2.8 Hz), 131.73 (d, J = 103.7 Hz), 131.15 (d, J = 9.9 Hz), 128.67 (d, J = 12.4 Hz), 128.51, 44.28 (d, J = 57.3 Hz). ^{31}P NMR (162 MHz, CDCl3) δ 26.76. HRMS (ESI) calcd for $C_{18}H_{15}NaO_2PS[M+Na]^+$: 349.0423; found: 349.0409.

2-(diphenylphosphoryl)-1-phenylpropan-1-one (3wa)



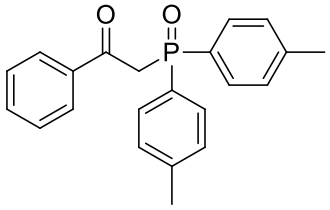
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.87 (m, 2H), 7.87 – 7.82 (m, 2H), 7.80 – 7.73 (m, 2H), 7.53 – 7.31 (m, 9H), 4.62 (dq, *J* = 14.3, 7.1 Hz, 1H), 1.58 (dd, *J* = 16.1, 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.12 (d, *J* = 2.9 Hz), 137.03 (d, *J* = 1.3 Hz), 133.18, 132.08 (d, *J* = 2.9 Hz), 132.02 (d, *J* = 2.8 Hz), 131.81 (d, *J* = 9.3 Hz), 131.50 (d, *J* = 9.1 Hz), 131.04 (d, *J* = 101.8 Hz), 130.60 (d, *J* = 99.9 Hz), 128.62 (d, *J* = 11.2 Hz), 128.45, 128.44, 45.68 (d, *J* = 59.7 Hz), 12.98 (d, *J* = 3.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.15. HRMS (ESI) calcd for $C_{21}H_{19}NaO_2P$ $[M+Na]^+$: 357.1015; found: 357.1016.

1-(diphenylphosphoryl)propan-2-one (3xa)



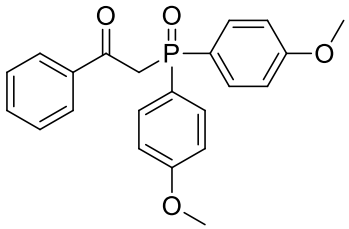
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.69 (m, 4H), 7.57 – 7.51 (m, 2H), 7.51 – 7.44 (m, 4H), 3.60 (d, *J* = 14.8 Hz, 2H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.98 (d, *J* = 5.2 Hz), 132.32 (d, *J* = 2.8 Hz), 131.82 (d, *J* = 103.4 Hz), 130.85 (d, *J* = 9.9 Hz), 128.81 (d, *J* = 12.3 Hz), 47.94 (d, *J* = 56.4 Hz), 32.71. ^{31}P NMR (162 MHz, $CDCl_3$) δ 26.33. HRMS (ESI) calcd for $C_{15}H_{15}NaO_2P$ $[M+H]^+$: 259.0882; found: 259.0881.

2-(di-p-tolylphosphoryl)-1-phenylethanone (3ab)



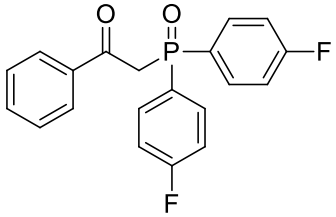
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.96 (m, 2H), 7.68 (dd, *J* = 11.9, 8.1 Hz, 4H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.25 (dd, *J* = 8.0, 2.5 Hz, 4H), 4.11 (d, *J* = 15.3 Hz, 2H), 2.37 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.04 (d, *J* = 5.6 Hz), 142.60 (d, *J* = 2.9 Hz), 137.04, 133.50, 131.10 (d, *J* = 10.2 Hz), 129.35 (d, *J* = 12.6 Hz), 129.30, 128.87 (d, *J* = 107.7 Hz), 128.49, 43.56 (d, *J* = 57.7 Hz), 21.63. ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.28. HRMS (ESI) calcd for $C_{22}H_{21}NaO_2P$ $[M+Na]^+$:371.1171; found: 371.1159.

2-(bis(4-methoxyphenyl)phosphoryl)-1-phenylethanone (3ac)



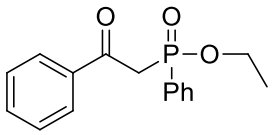
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.94 (m, 2H), 7.76 – 7.65 (m, 4H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 6.94 (dd, *J* = 8.9, 2.3 Hz, 4H), 4.09 (d, *J* = 15.5 Hz, 2H), 3.81 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.24 (d, *J* = 5.5 Hz), 162.51 (d, *J* = 2.9 Hz), 137.03, 133.50, 133.00 (d, *J* = 11.2 Hz), 129.26, 128.50, 123.33 (d, *J* = 110.3 Hz), 114.13 (d, *J* = 13.4 Hz), 55.34, 43.83 (d, *J* = 58.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.07. HRMS (ESI) calcd for $C_{22}H_{21}NaO_4P$ $[M+Na]^+$: 403.1070; found: 403.1070.

2-(bis(4-fluorophenyl)phosphoryl)-1-phenylethanone (3ad)



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.92 (m, 2H), 7.87 – 7.74 (m, 4H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.21 – 7.09 (m, 4H), 4.14 (d, *J* = 15.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.71 (d, *J* = 5.6 Hz), 165.18 (dd, *J* = 254.3, 3.4 Hz), 136.75, 133.87, 133.71 (dd, *J* = 11.4, 8.9 Hz), 129.19, 128.65, 127.71 (dd, *J* = 106.9, 3.4 Hz), 116.15 (dd, *J* = 21.5, 13.6 Hz), 43.30 (d, *J* = 59.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 25.89. ^{19}F NMR (376 MHz, $CDCl_3$) δ -105.77 (d, *J* = 1.5 Hz). HRMS (ESI) calcd for $C_{20}H_{15}F_2NaO_2P$ $[M+Na]^+$: 379.0670; found: 379.0679.

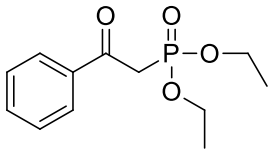
ethyl (2-oxo-2-phenylethyl)(phenyl)phosphinate (3ae)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.5 Hz, 2H), 7.79 (dd, *J* = 12.4, 7.1 Hz, 2H), 7.55 (dd, *J* = 11.7, 4.3 Hz, 2H), 7.46 (m, 4H), 4.20 –

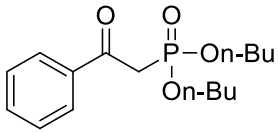
3.89 (m, 2H), 3.87 – 3.73 (m, 2H), 1.26 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.20 (d, *J* = 5.5 Hz), 136.79, 133.53, 132.73 (d, *J* = 2.9 Hz), 131.85 (d, *J* = 10.3 Hz), 130.08 (d, *J* = 132.9 Hz), 129.09, 128.63 (d, *J* = 13.3 Hz), 128.52, 61.47 (d, *J* = 6.2 Hz), 43.02 (d, *J* = 86.4 Hz), 16.32 (d, *J* = 6.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 34.39. HRMS (ESI) calcd for $C_{16}H_{17}NaO_3P$ $[M+Na]^+$: 311.0808; found: 311.0822.

diethyl (2-oxo-2-phenylethyl)phosphonate (3af)



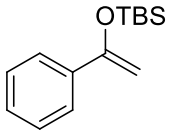
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 7.96 (m, 2H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 4.15 (p, *J* = 7.2 Hz, 4H), 3.65 (d, *J* = 22.7 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 192.00 (d, *J* = 6.6 Hz), 136.53 (d, *J* = 1.8 Hz), 133.70, 129.07, 128.63, 62.67 (d, *J* = 6.5 Hz), 38.49 (d, *J* = 129.9 Hz), 16.26 (d, *J* = 6.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.91. HRMS (ESI) calcd for $C_{12}H_{17}NaO_4P$ $[M+Na]^+$: 279.0757; found: 279.0770.

dibutyl (2-oxo-2-phenylethyl)phosphonate (3ag)



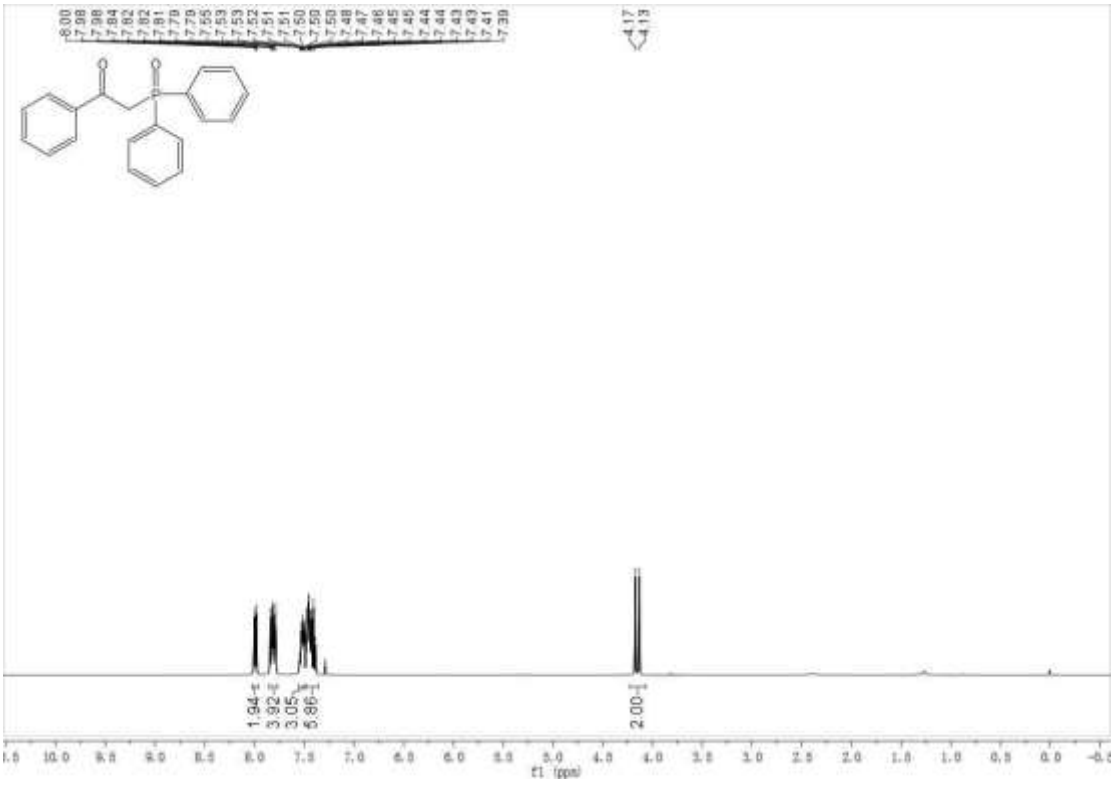
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 7.97 (m, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 4.13 – 4.01 (m, 4H), 3.64 (d, *J* = 22.8 Hz, 2H), 1.64 – 1.54 (m, 4H), 1.33 (dq, *J* = 14.7, 7.4 Hz, 4H), 0.89 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.93 (d, *J* = 6.7 Hz), 136.53 (d, *J* = 1.8 Hz), 133.62, 66.32 (d, *J* = 6.8 Hz), 38.33 (d, *J* = 129.5 Hz), 32.36 (d, *J* = 6.4 Hz), 18.62, 13.56. ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.86. HRMS (ESI) calcd for $C_{16}H_{25}NaO_4P^+$ $[M+Na]^+$: 335.1383; found: 335.1391.

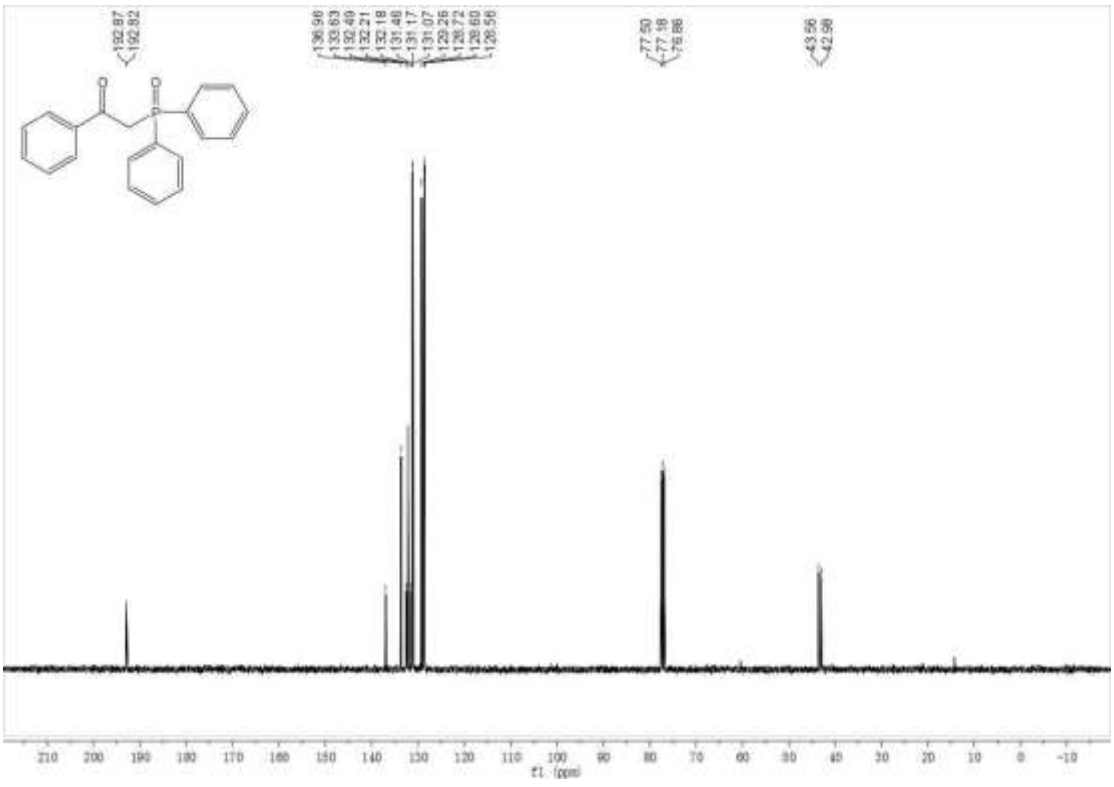
tert-butyldimethyl((1-phenylvinyl)oxy)silane(1a')

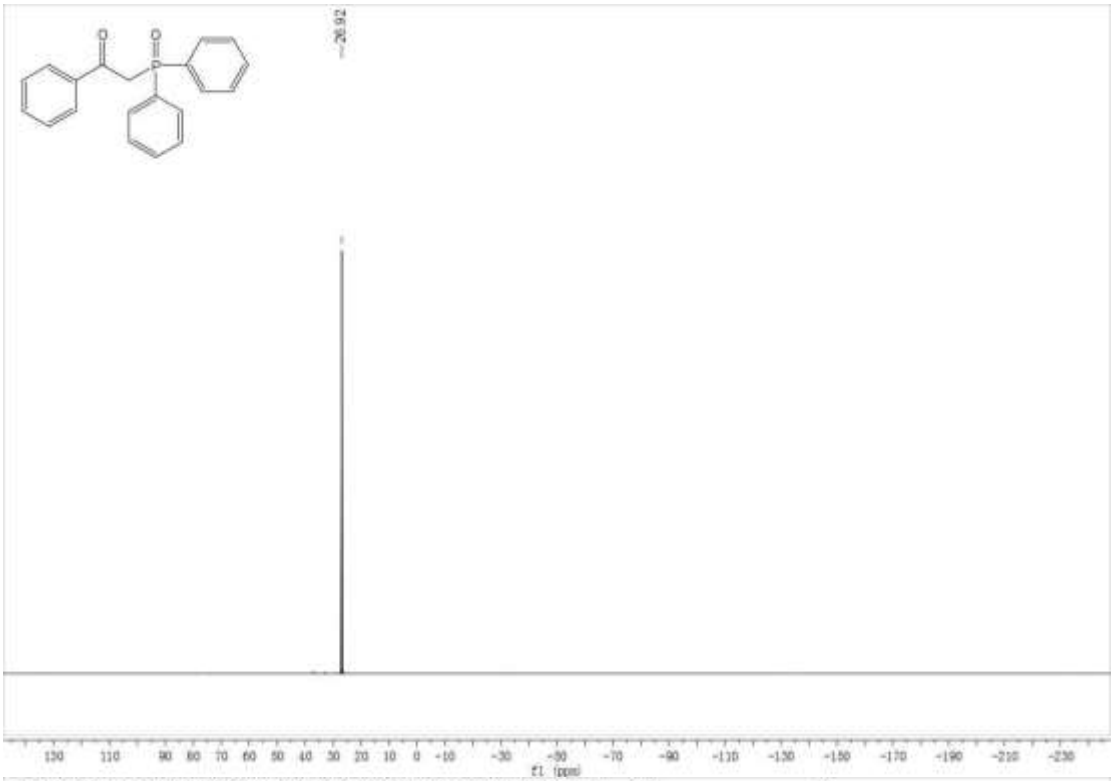


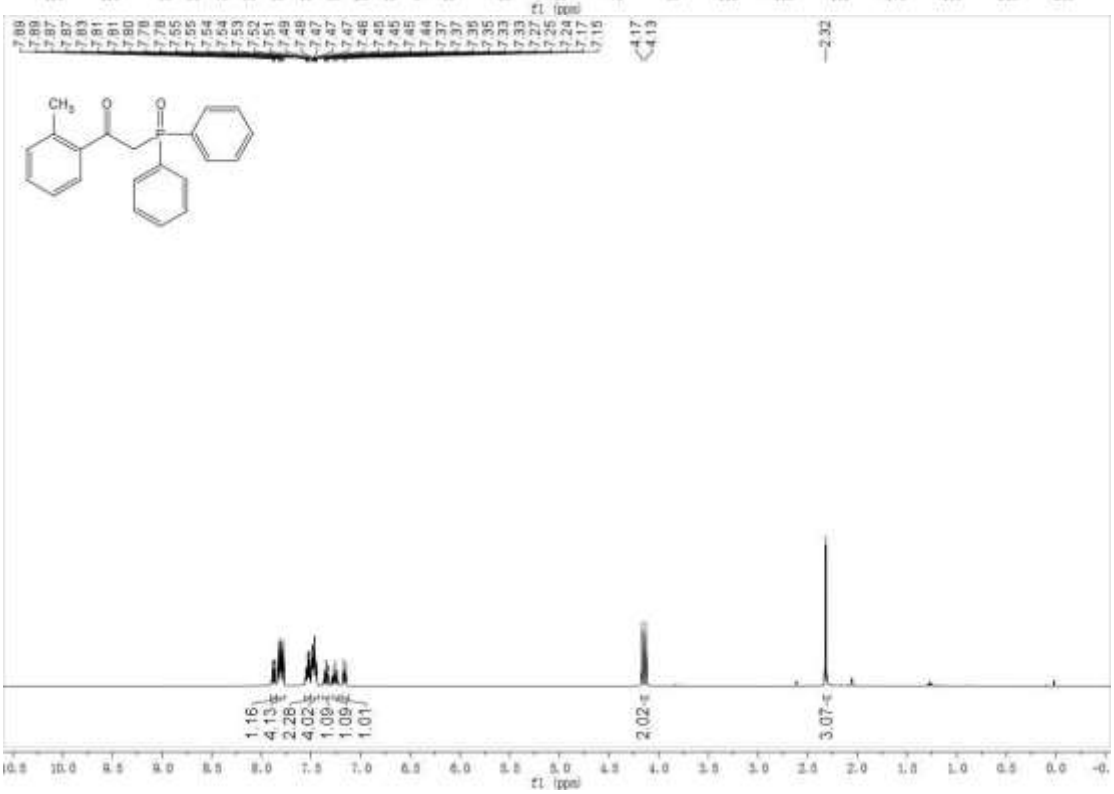
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (m, 2H), 7.41 – 7.30 (m, 3H), 4.94 (d, *J* = 1.7 Hz, 1H), 4.47 (d, *J* = 1.7 Hz, 1H), 1.06 (s, 9H), 0.26 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.95, 137.78, 128.17, 128.07, 125.28, 90.91, 25.89, 18.37, -4.59.

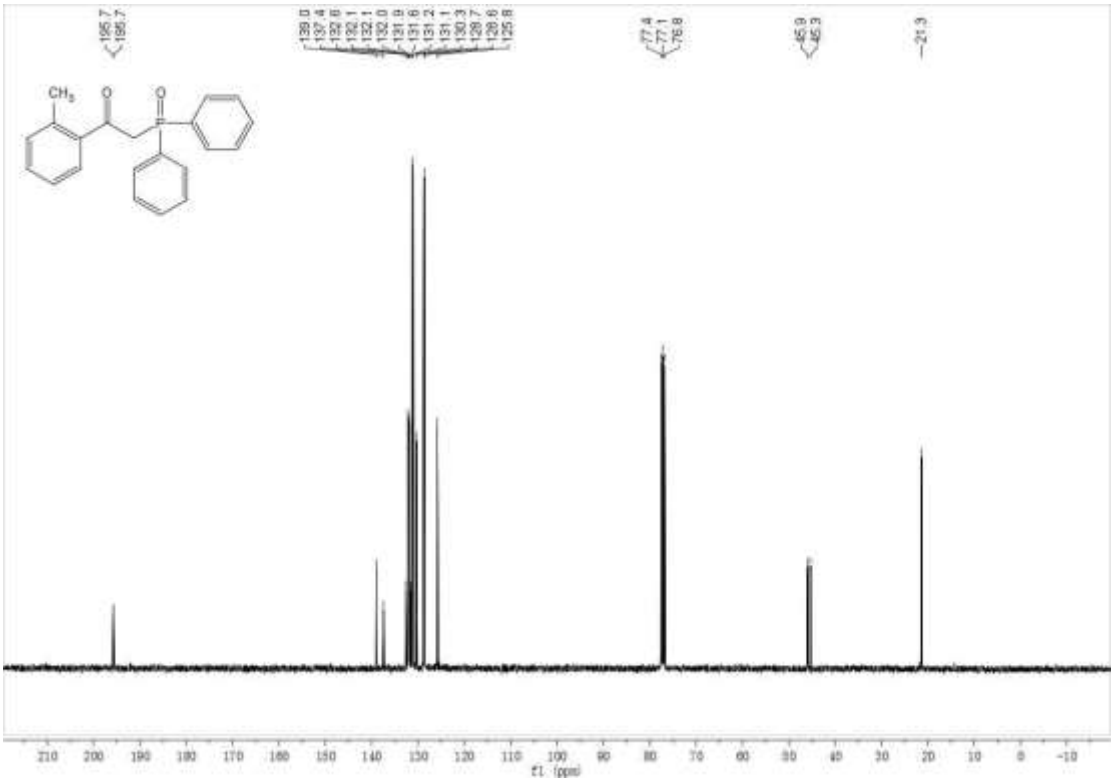
6. **Copies of NMR spectra for products**

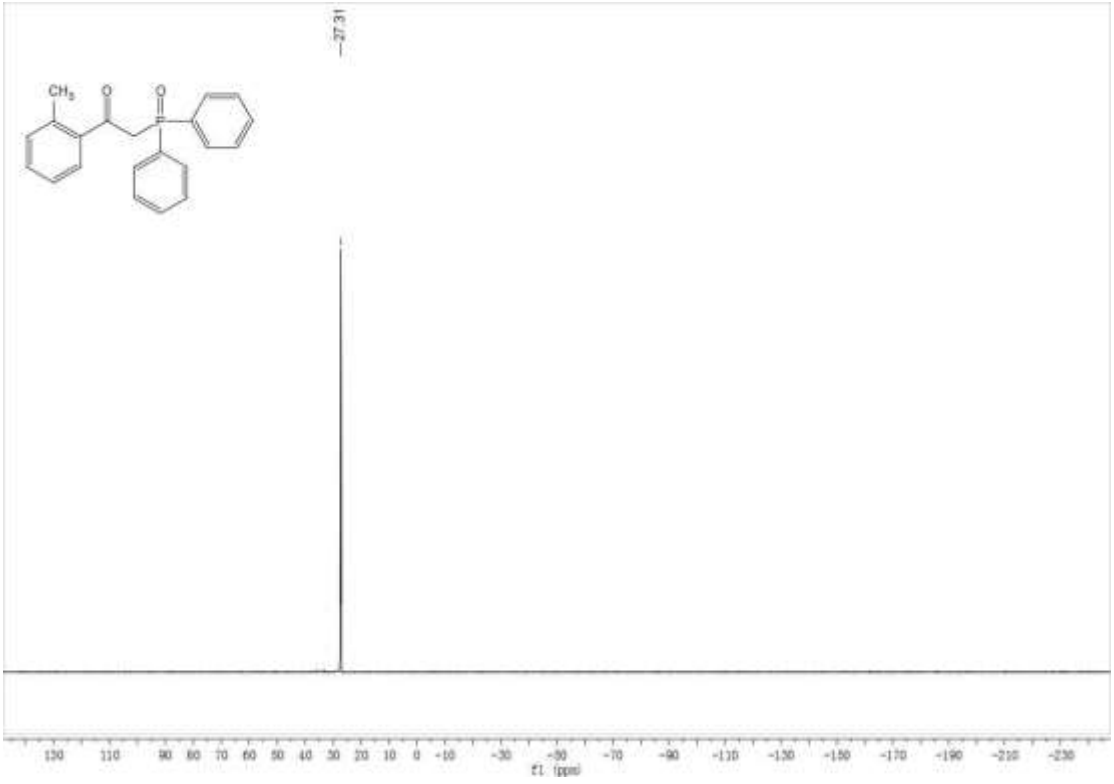


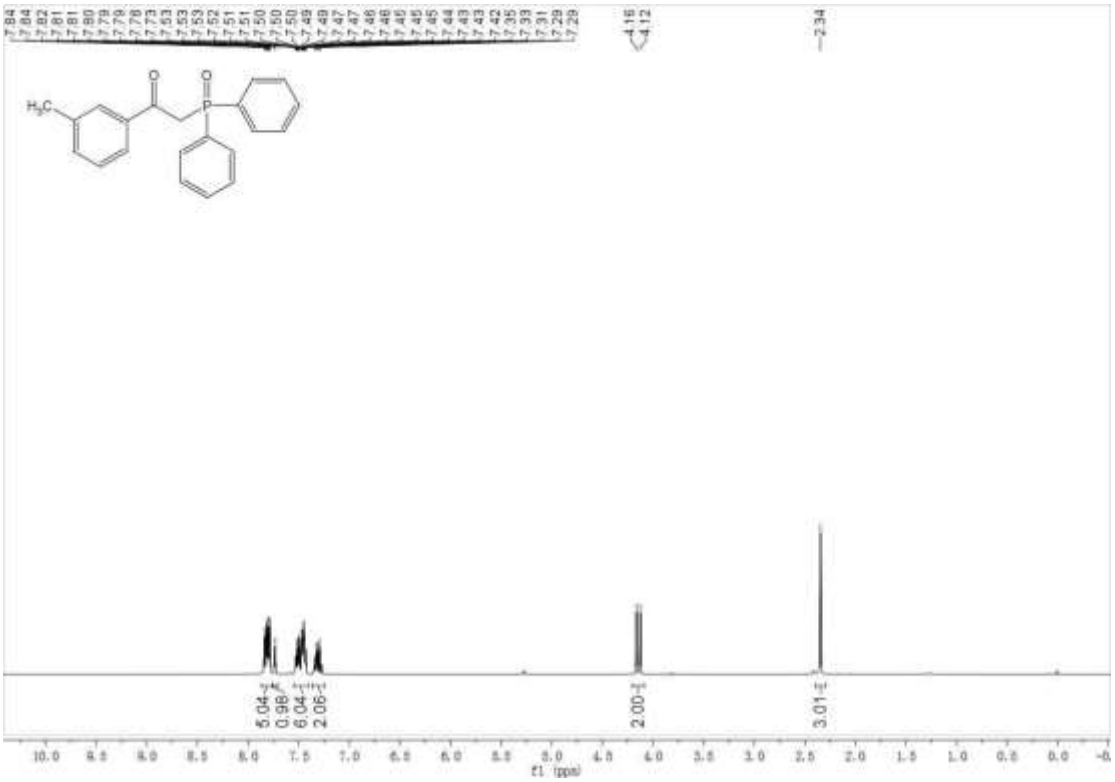


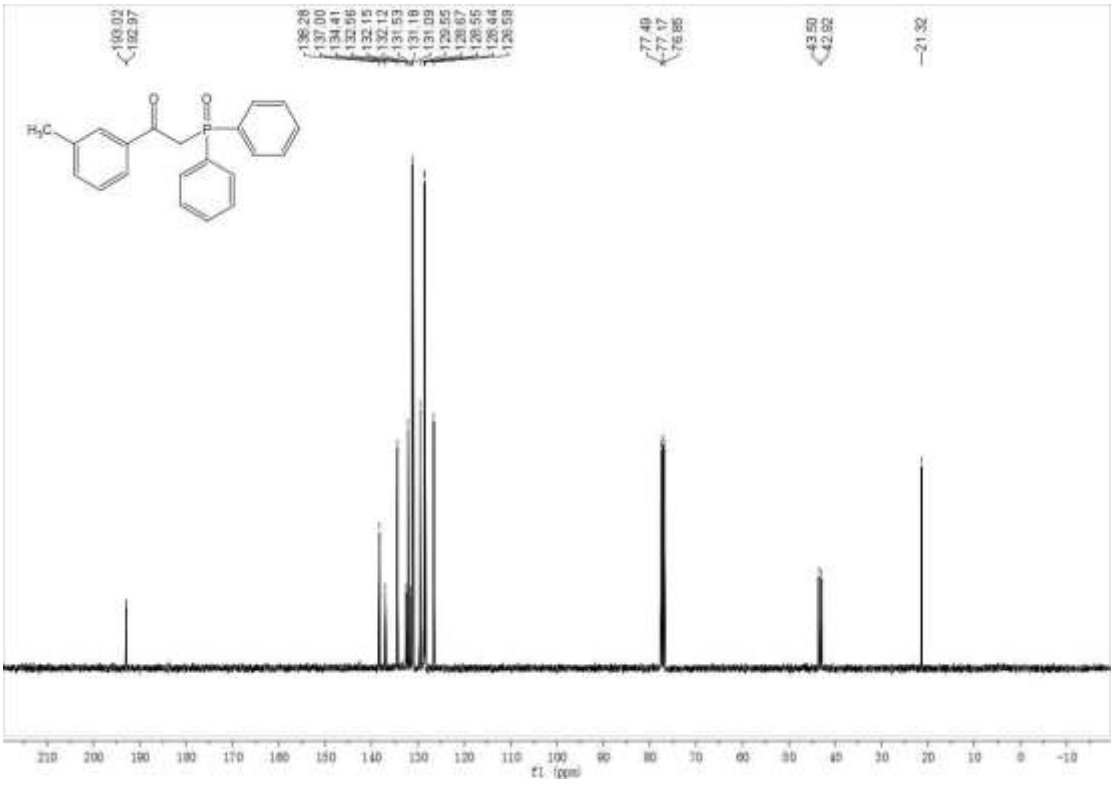

26.92
f1 (ppm)

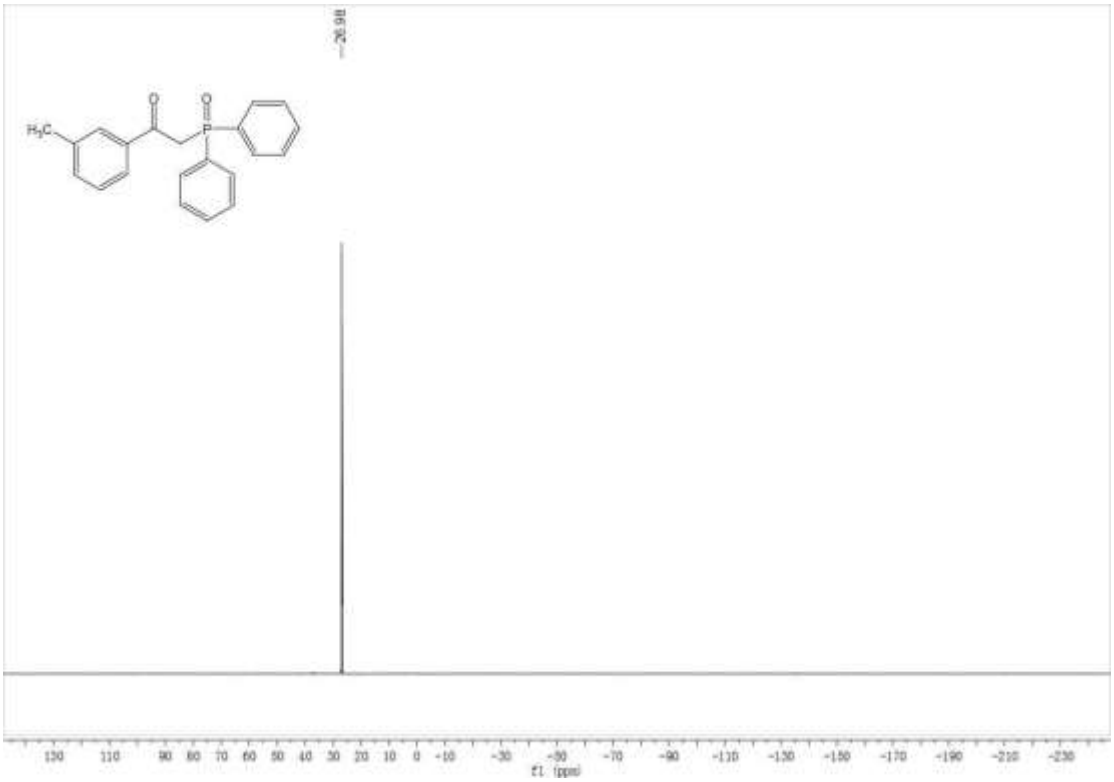

CH3
4.17
4.13
2.32
1.16
4.13
2.28
4.02
1.09
1.09
1.01
2.02
3.07
f1 (ppm)

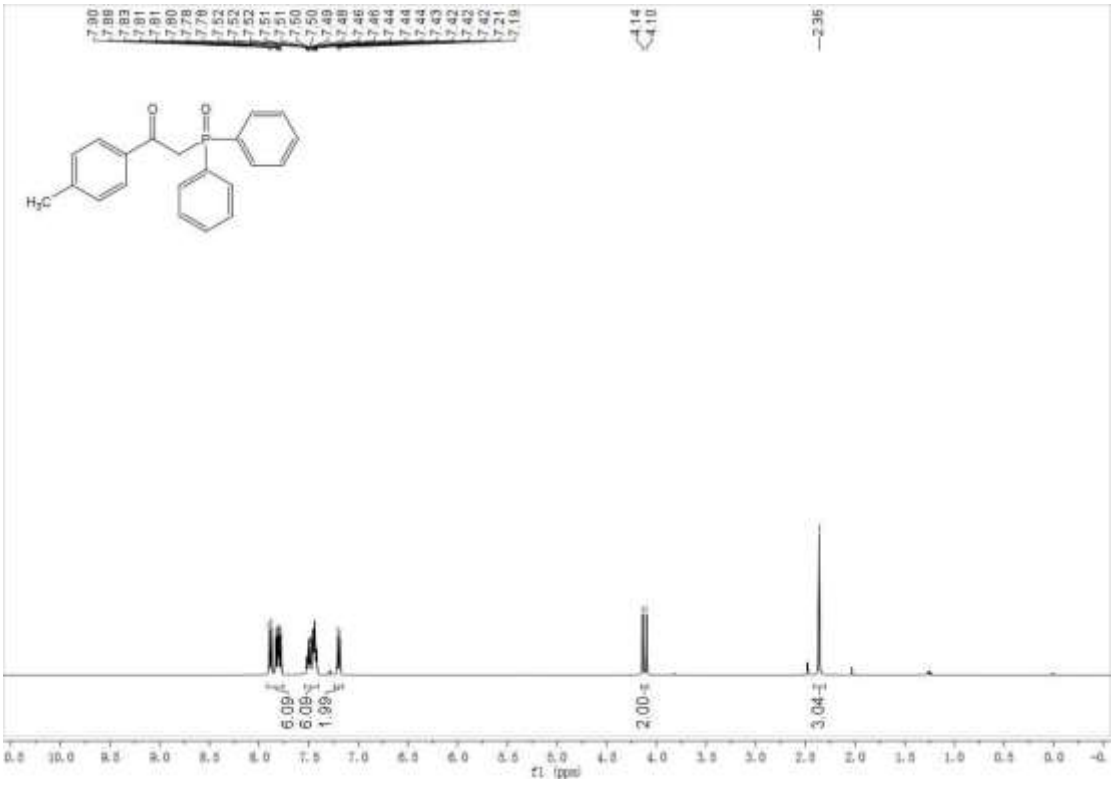

195.7
186.7
139.0
137.4
132.6
132.1
132.0
131.9
131.6
131.2
131.1
130.3
128.7
128.6
125.8
77.4
77.1
76.8
65.9
65.3
21.3
CH3
O
O
P
f1 (ppm)

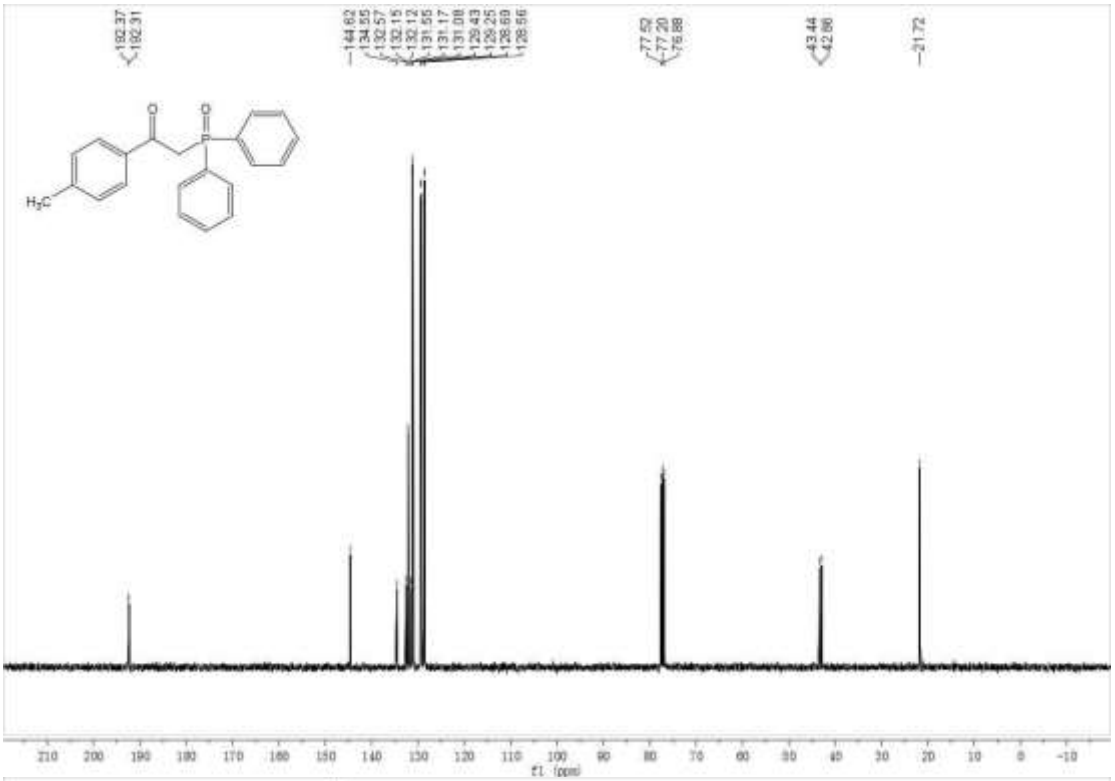

27.31
CH3
O
O
P
f1 (ppm)

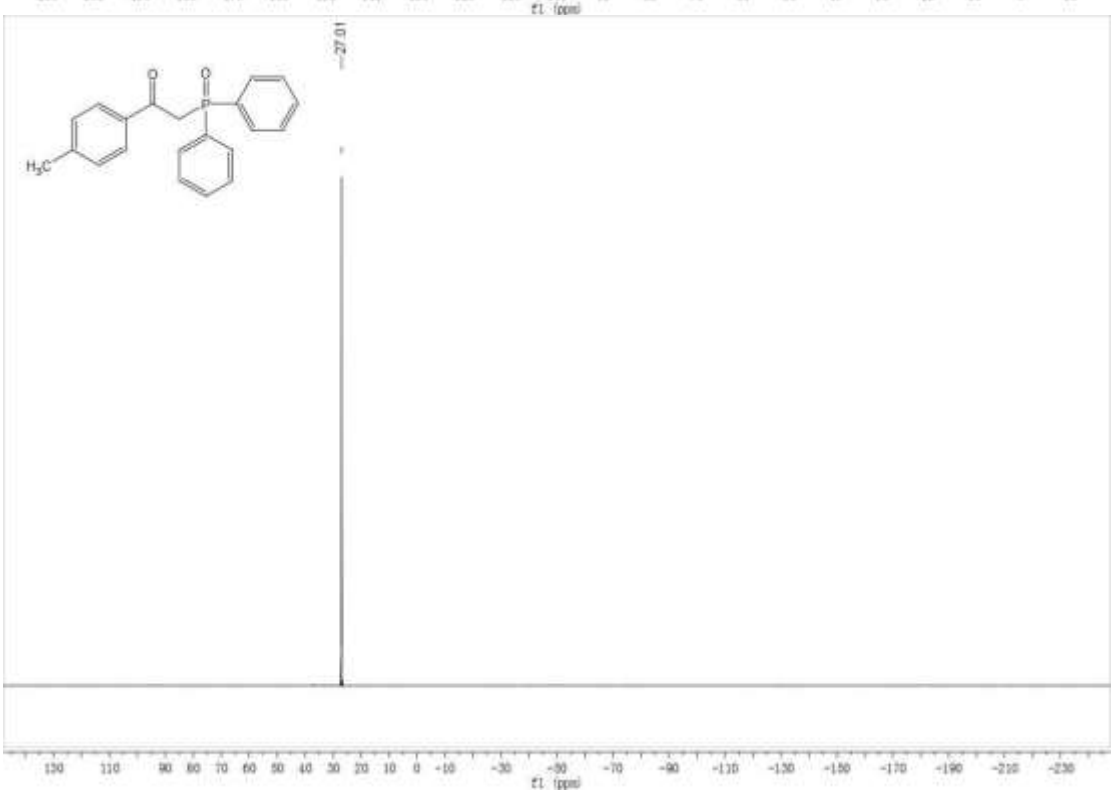


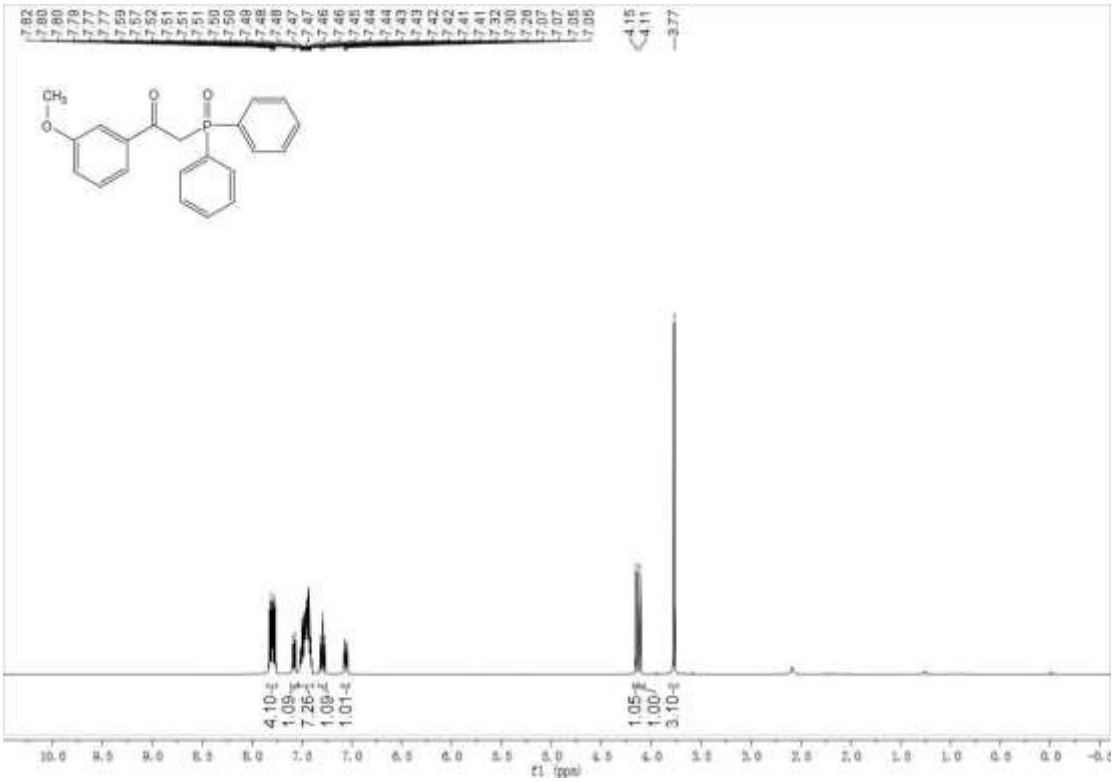


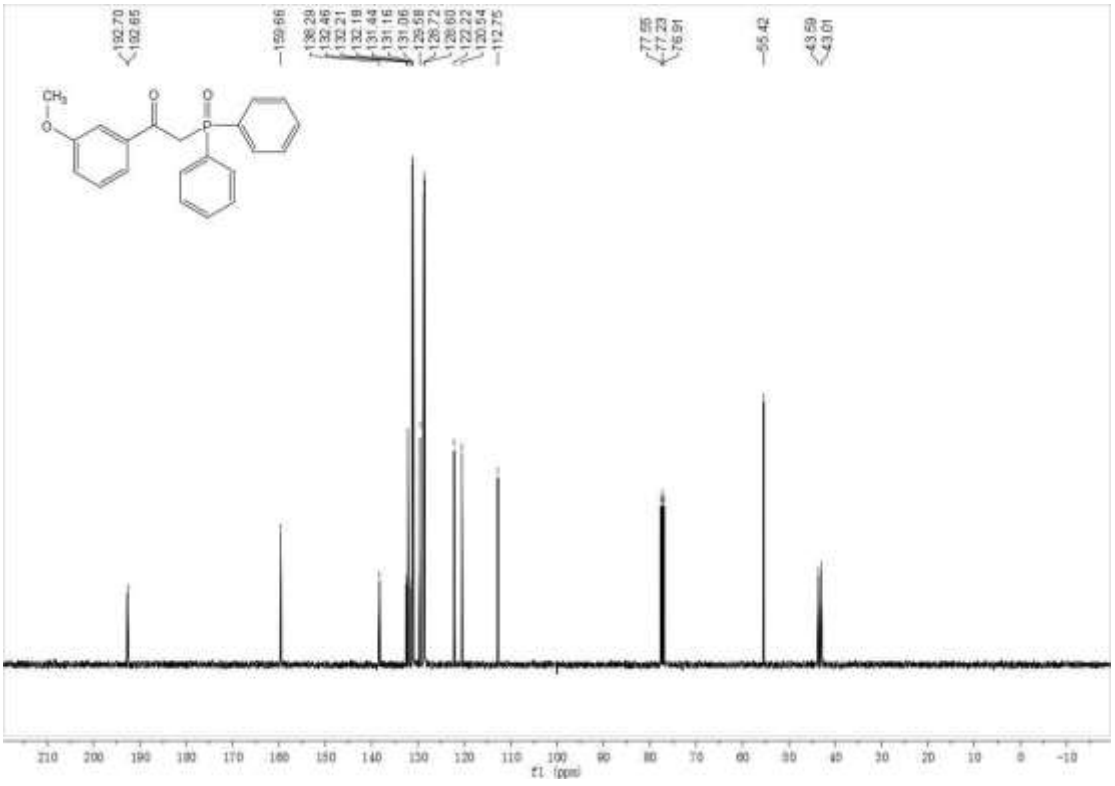

26.98
H3C
O
O
P
f1 (ppm)

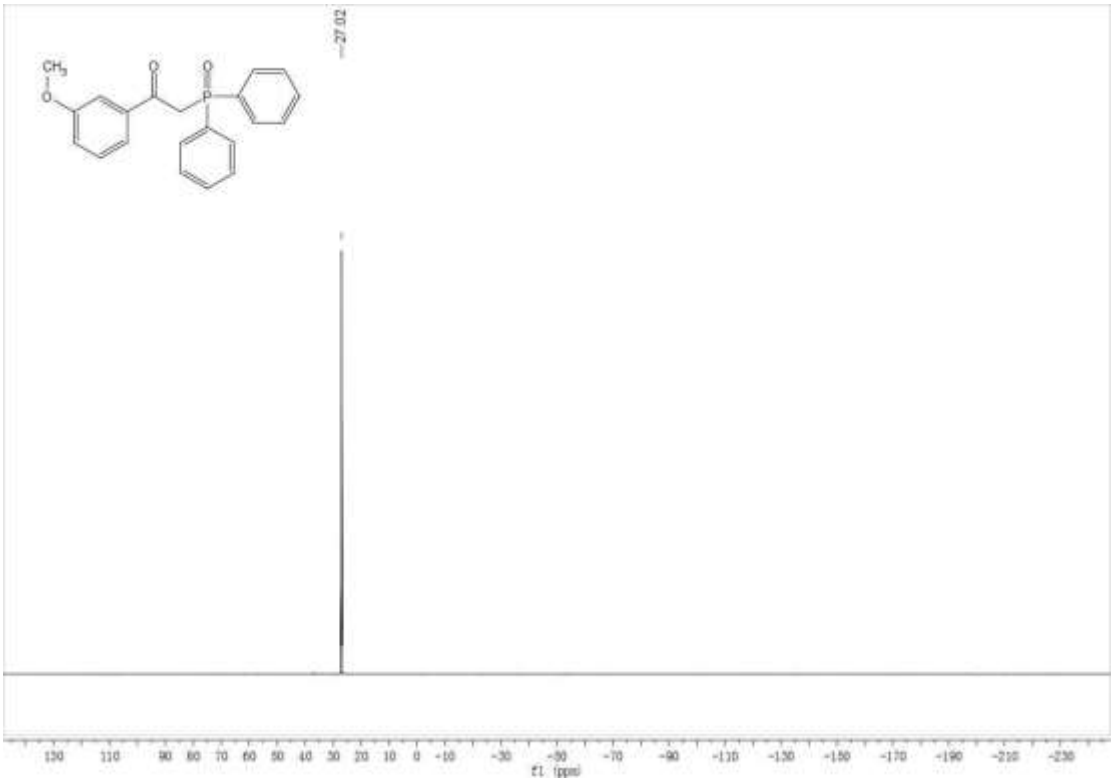

7.90
7.89
7.83
7.81
7.81
7.80
7.78
7.78
7.52
7.52
7.52
7.51
7.51
7.50
7.50
7.49
7.48
7.46
7.46
7.44
7.44
7.43
7.42
7.42
7.42
7.21
7.19
4.14
4.10
2.36
H3C
O
O
P
6.09
6.09
1.99
2.00
3.04
f1 (ppm)

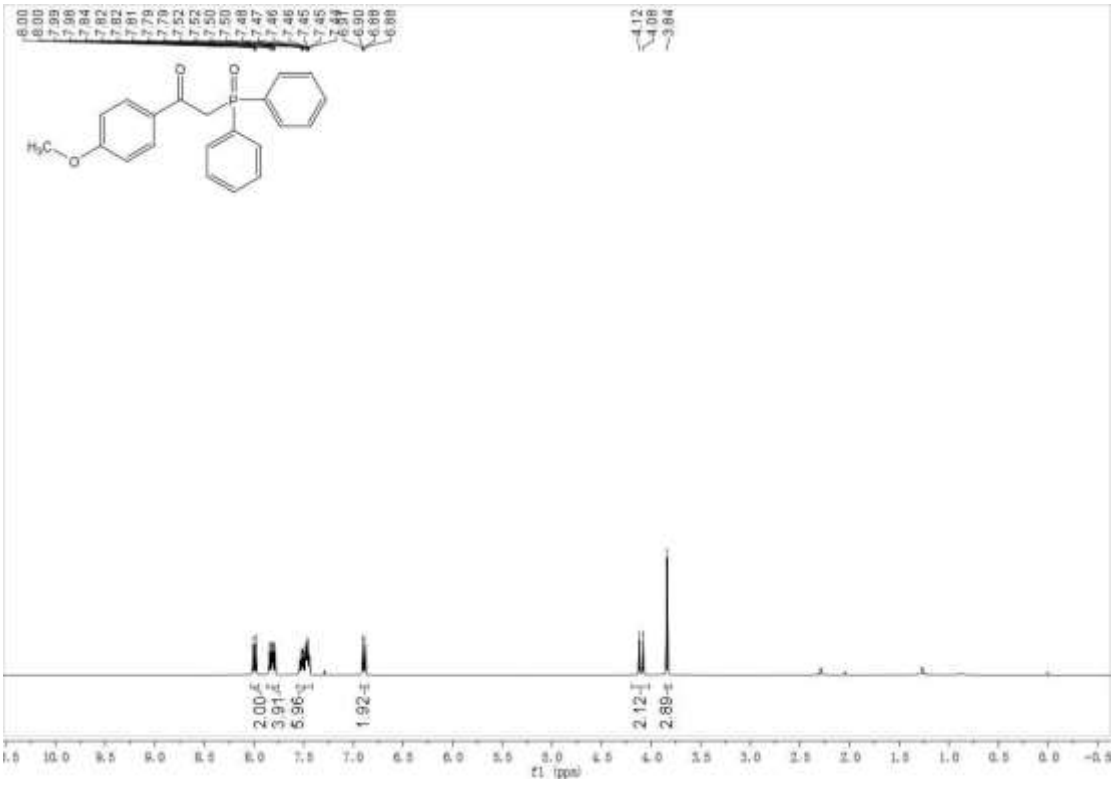

192.37
192.31
144.62
134.55
132.57
132.15
132.12
131.55
131.17
131.08
129.43
129.25
128.69
128.56
77.52
77.20
76.88
43.44
42.86
21.72
f1 (ppm)

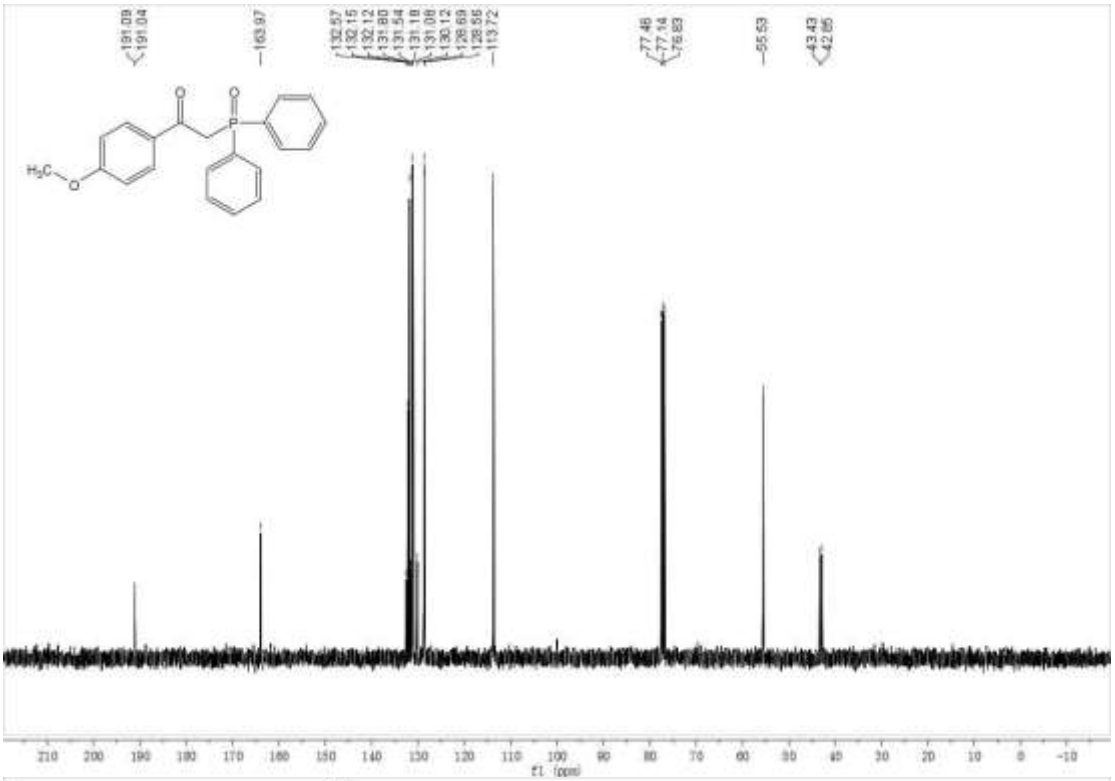

27.01
f1 (ppm)

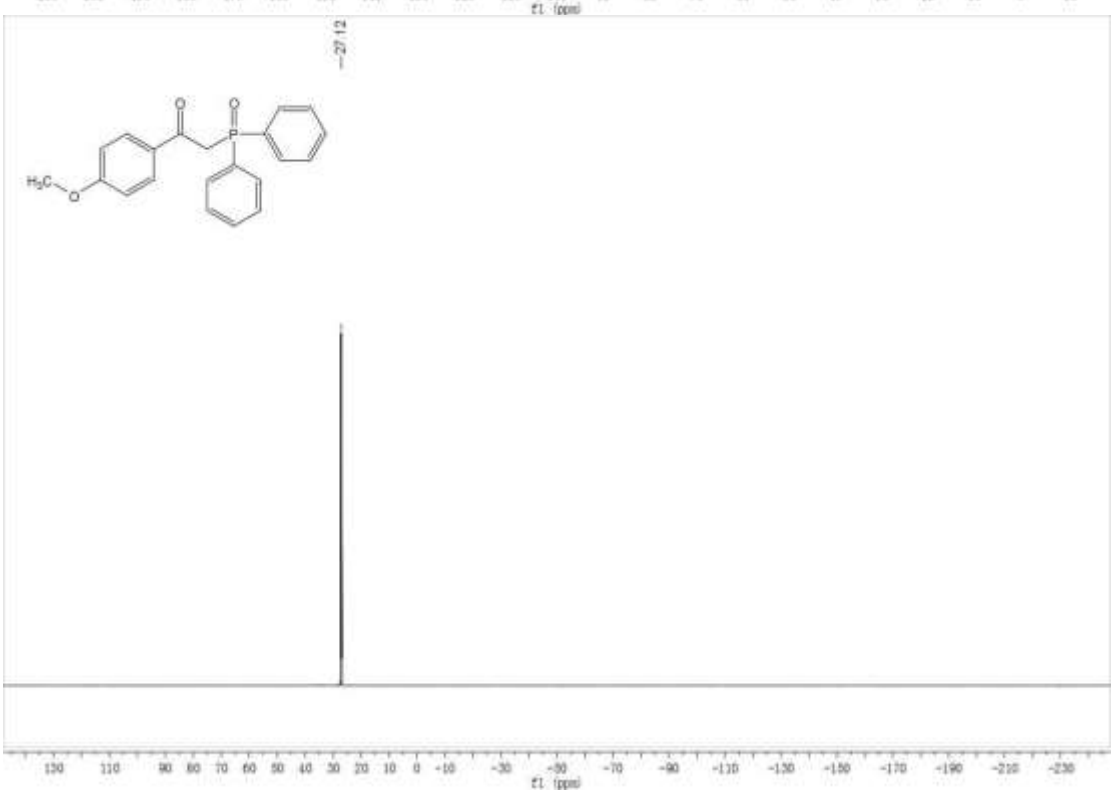


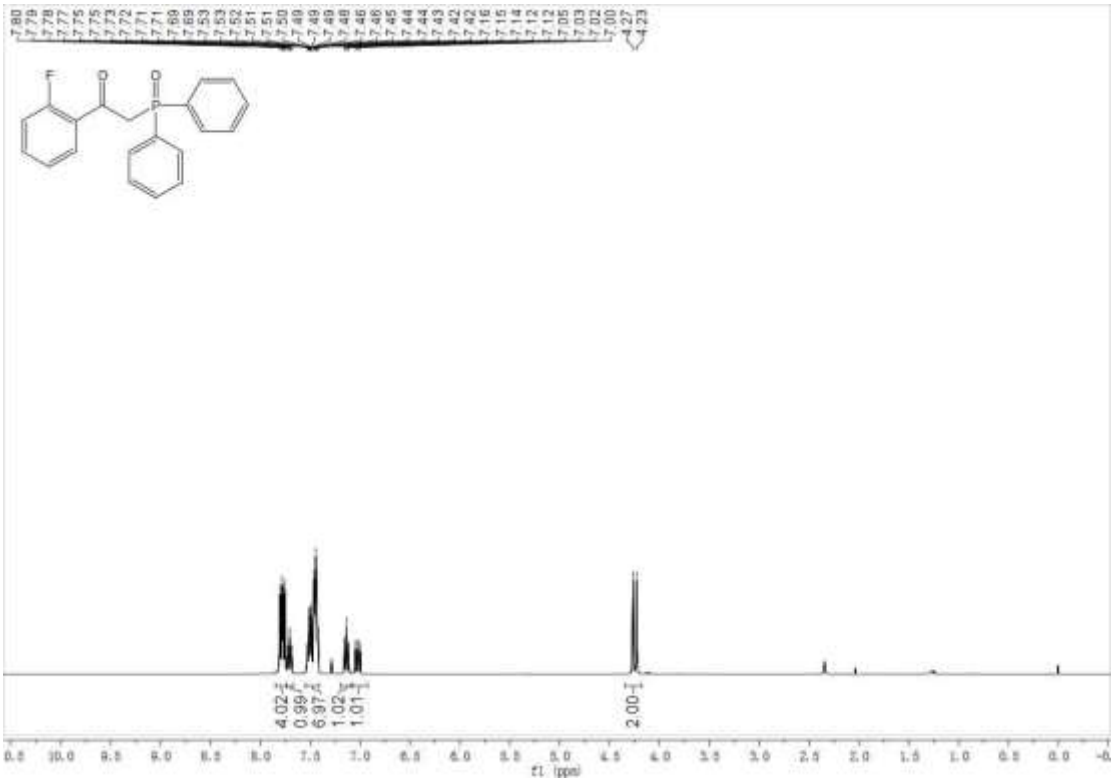


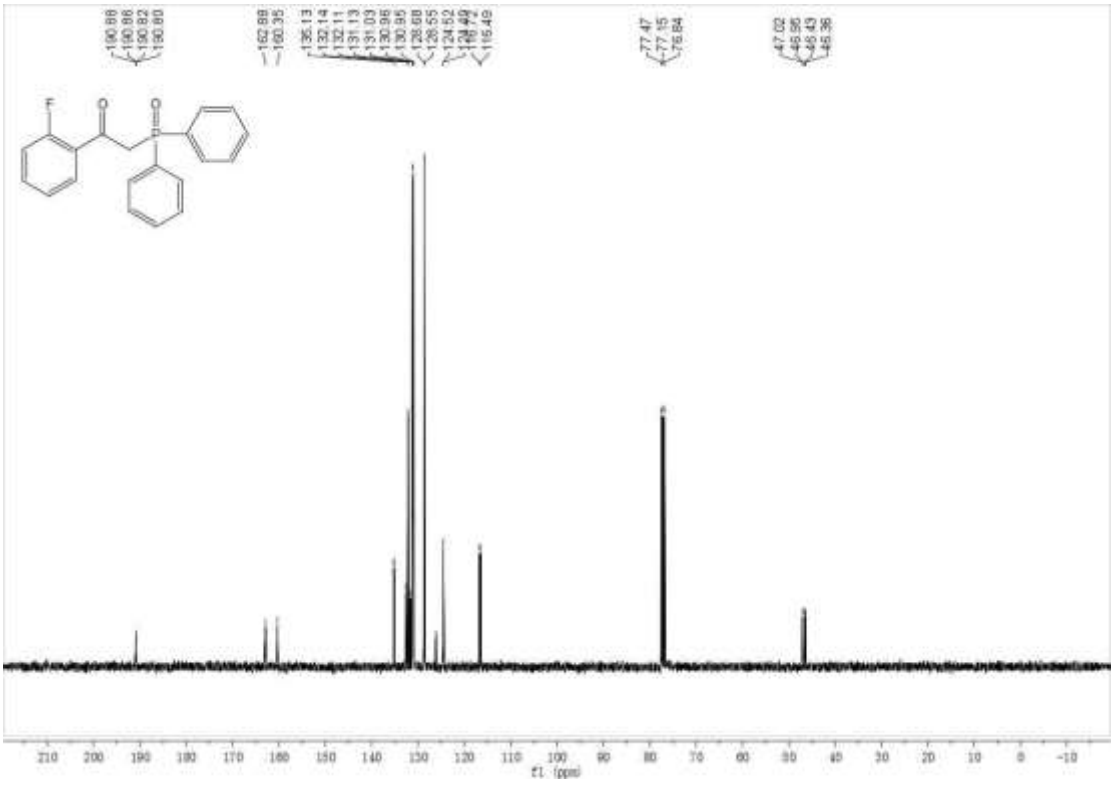

27.02
f1 (ppm)

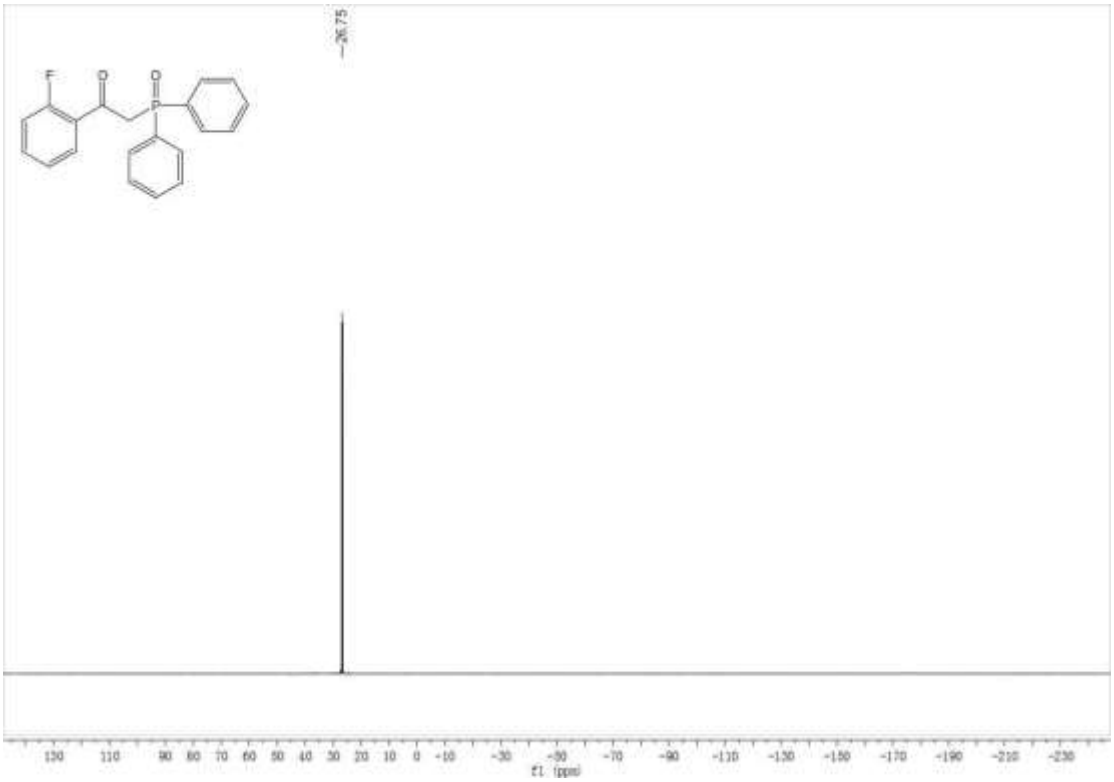

8.00
8.00
7.99
7.96
7.84
7.82
7.82
7.81
7.79
7.79
7.52
7.52
7.50
7.50
7.48
7.47
7.46
7.46
7.45
7.45
6.90
6.88
6.88
4.12
4.08
3.84
2.00
3.91
5.96
1.92
2.12
2.89
f1 (ppm)

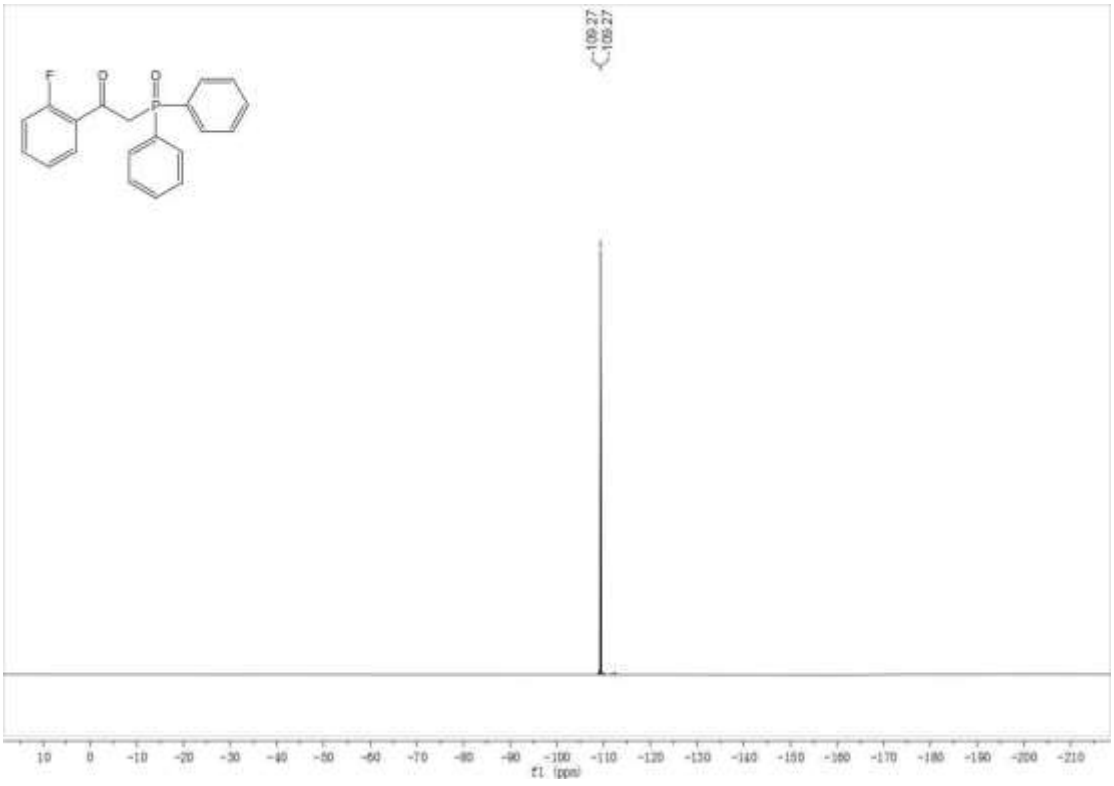

191.09
191.04
163.97
132.57
132.15
132.12
131.80
131.54
131.18
131.08
130.12
128.69
128.56
113.72
77.46
77.14
76.83
55.53
43.43
42.86
H3C
O
P
f1 (ppm)

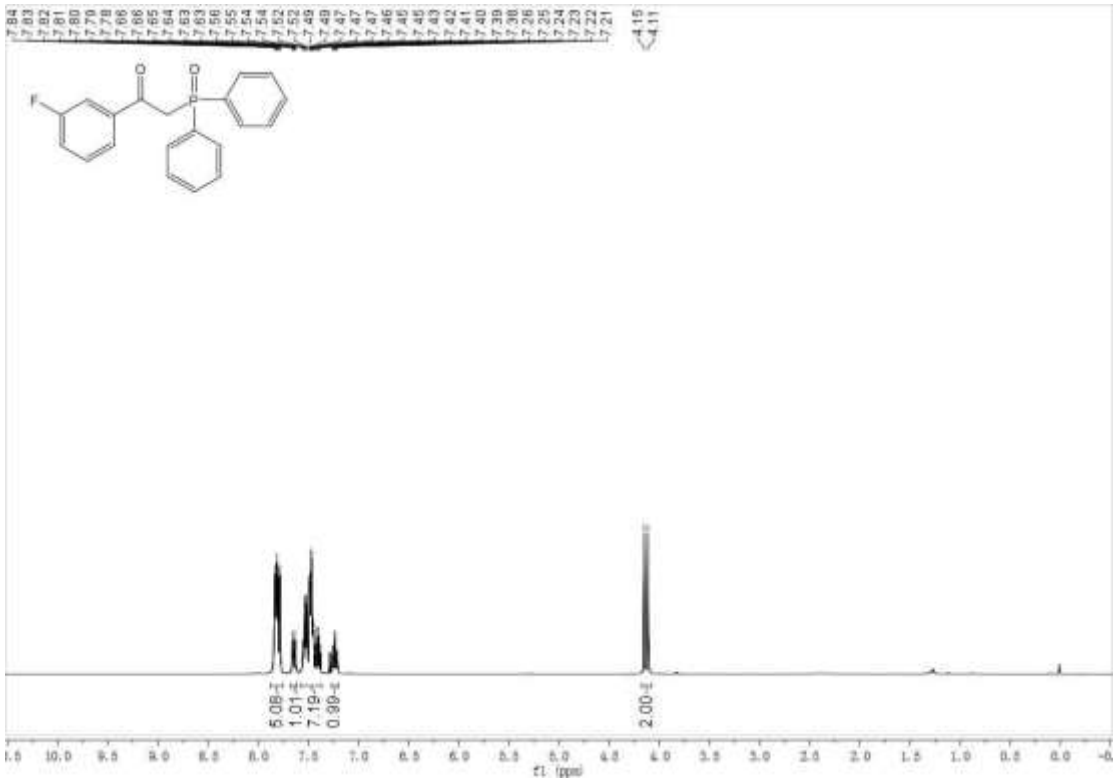

27.12
H3C
O
P
f1 (ppm)

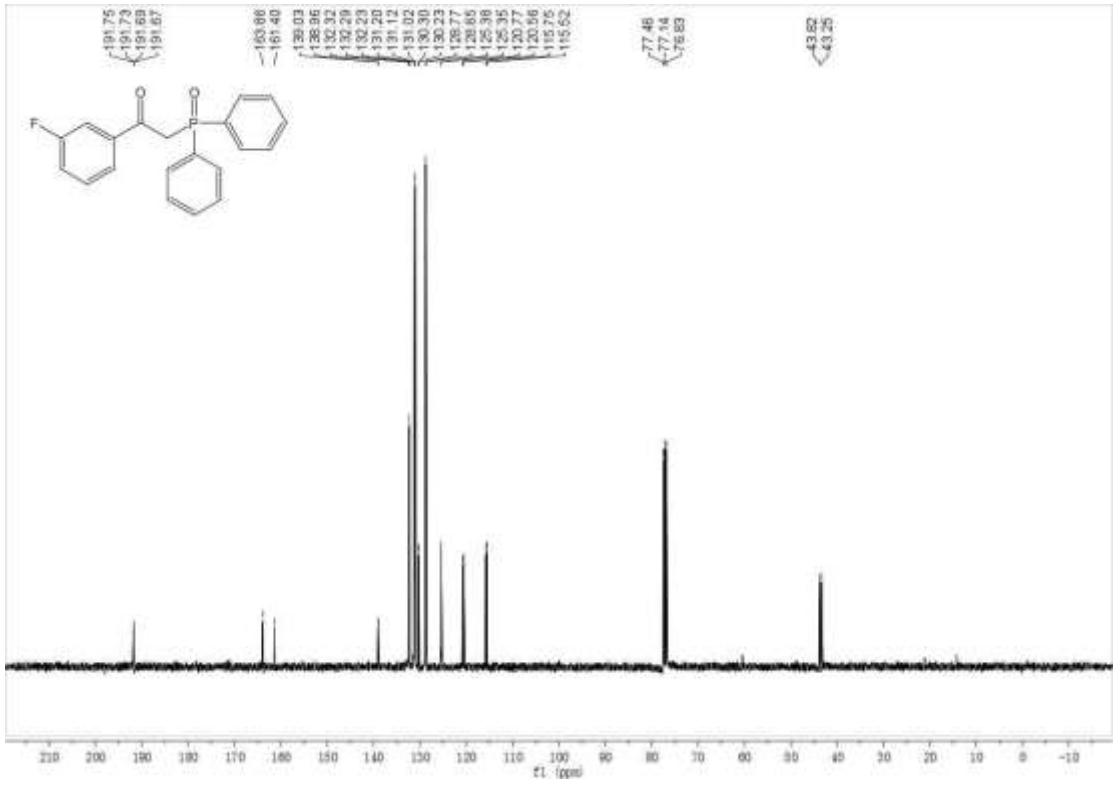


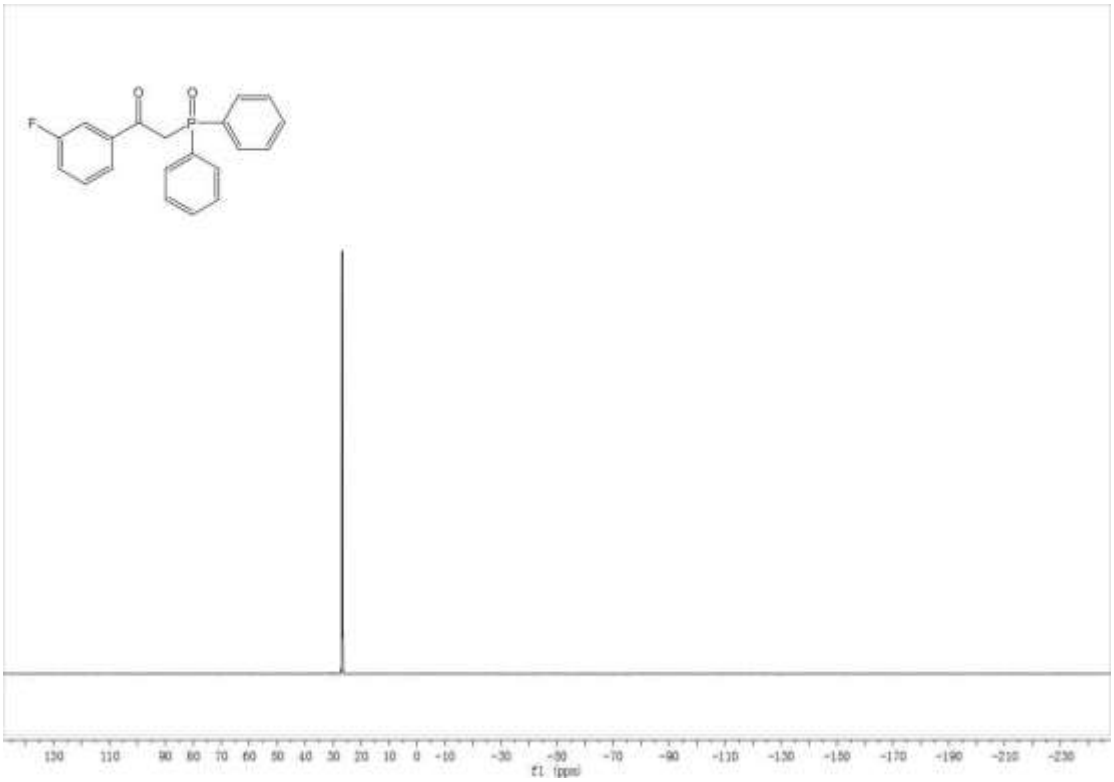


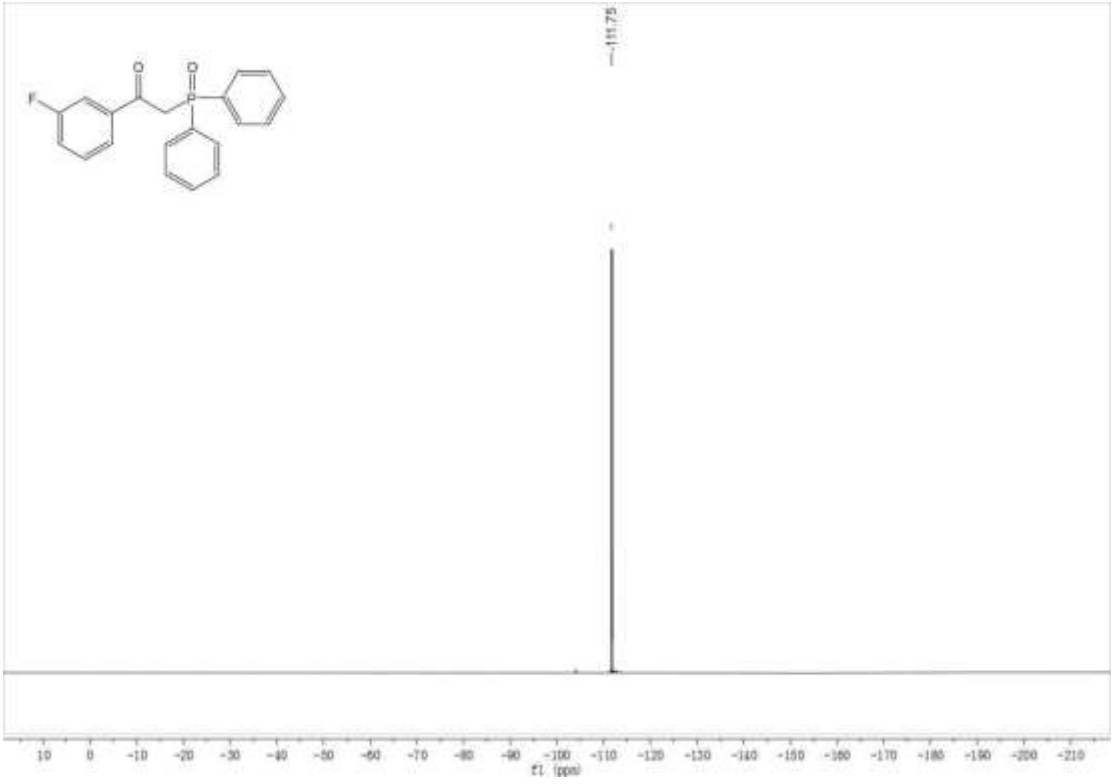

26.75
F
O
O
P
f1 (ppm)

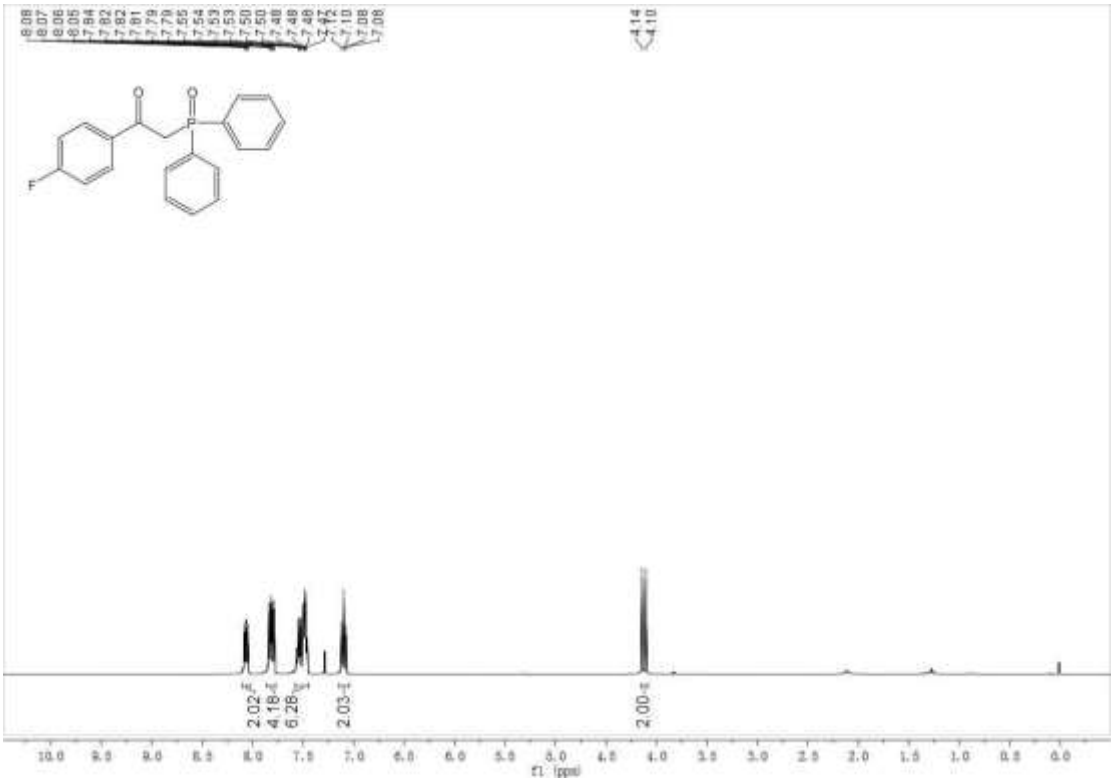

109.27
109.27
F
O
O
P
f1 (ppm)

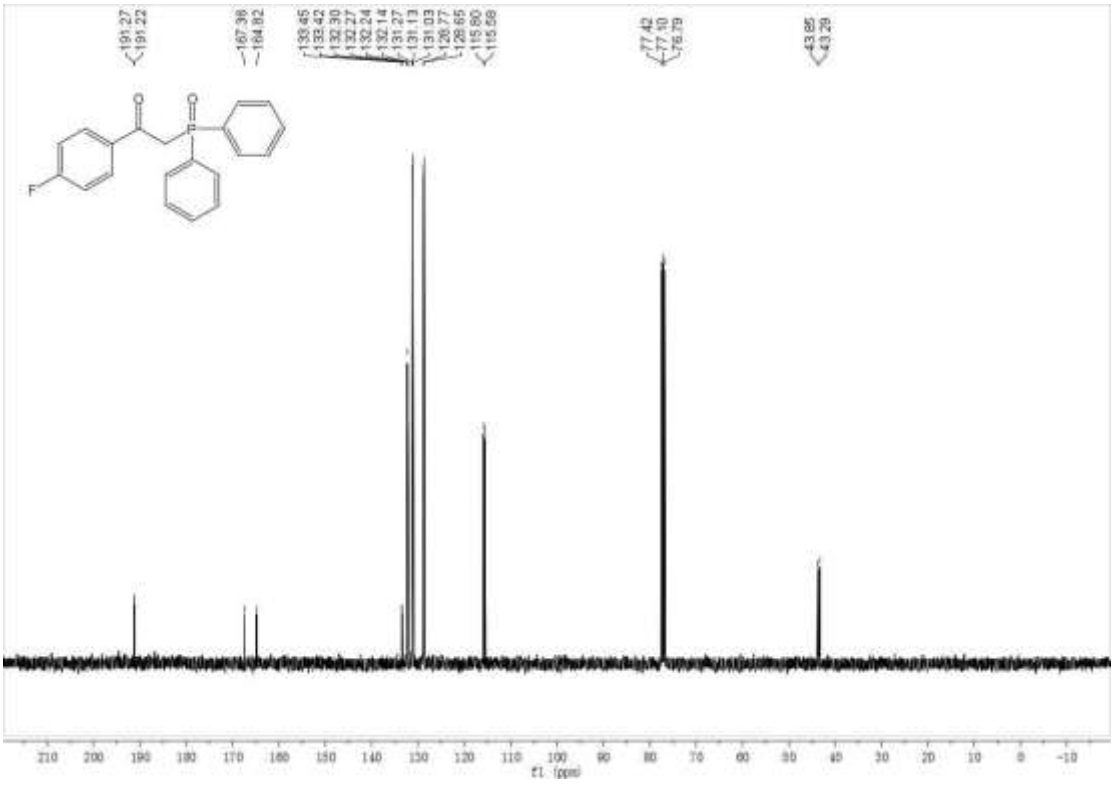


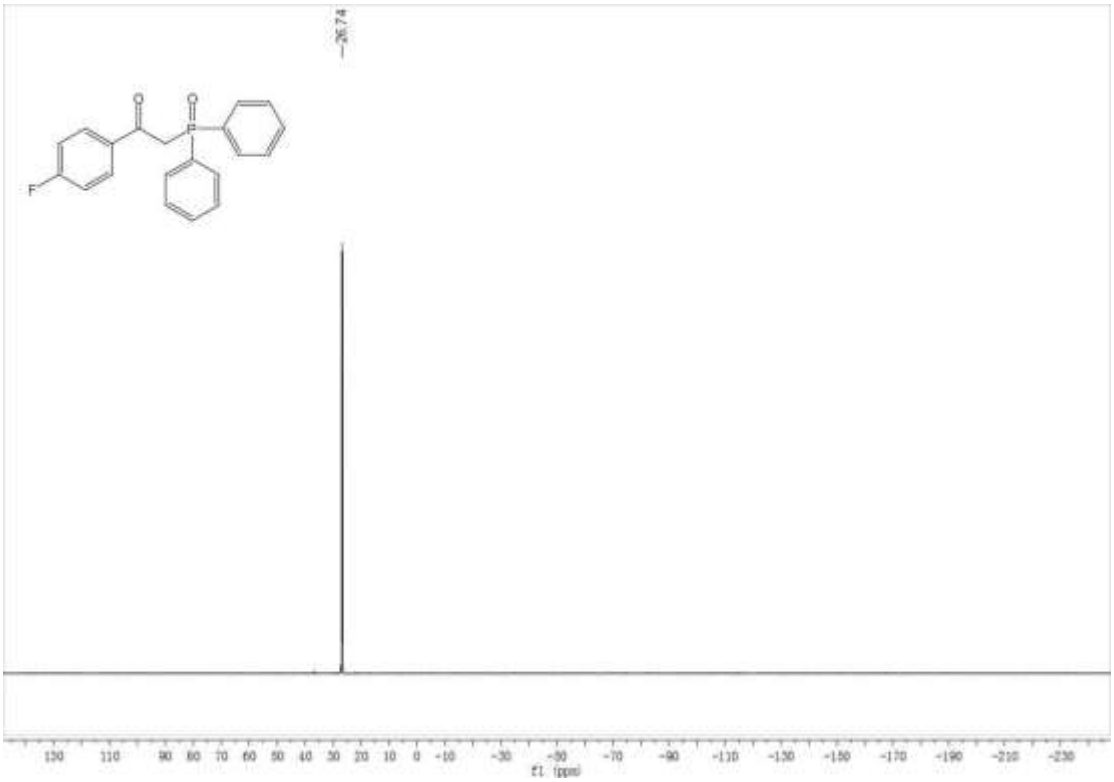


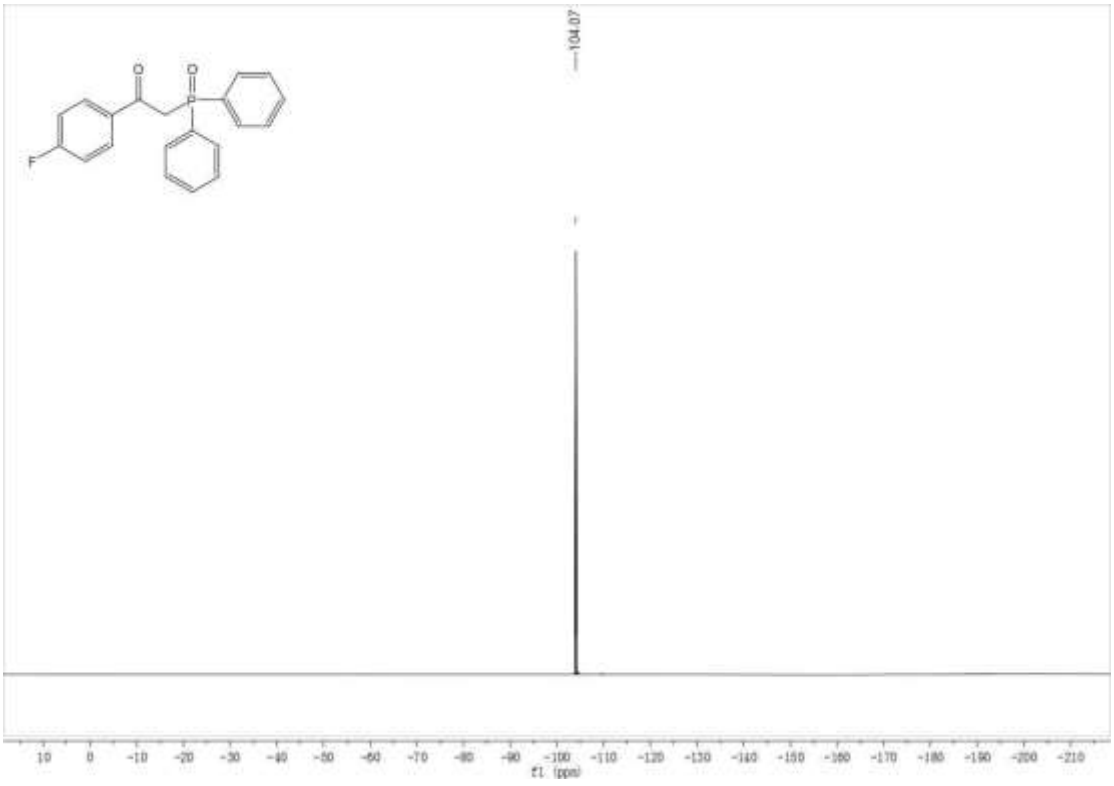

F
O
O
P
f1 (ppm)

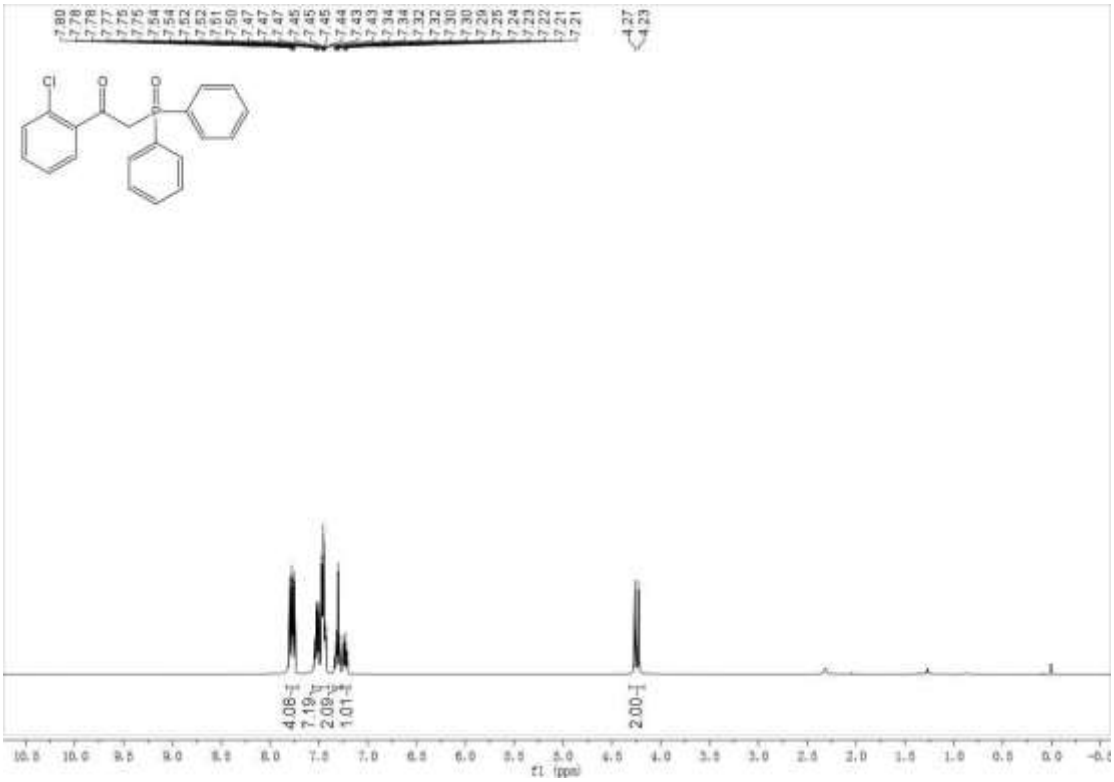

-111.75
F
O
O
P
f1 (ppm)

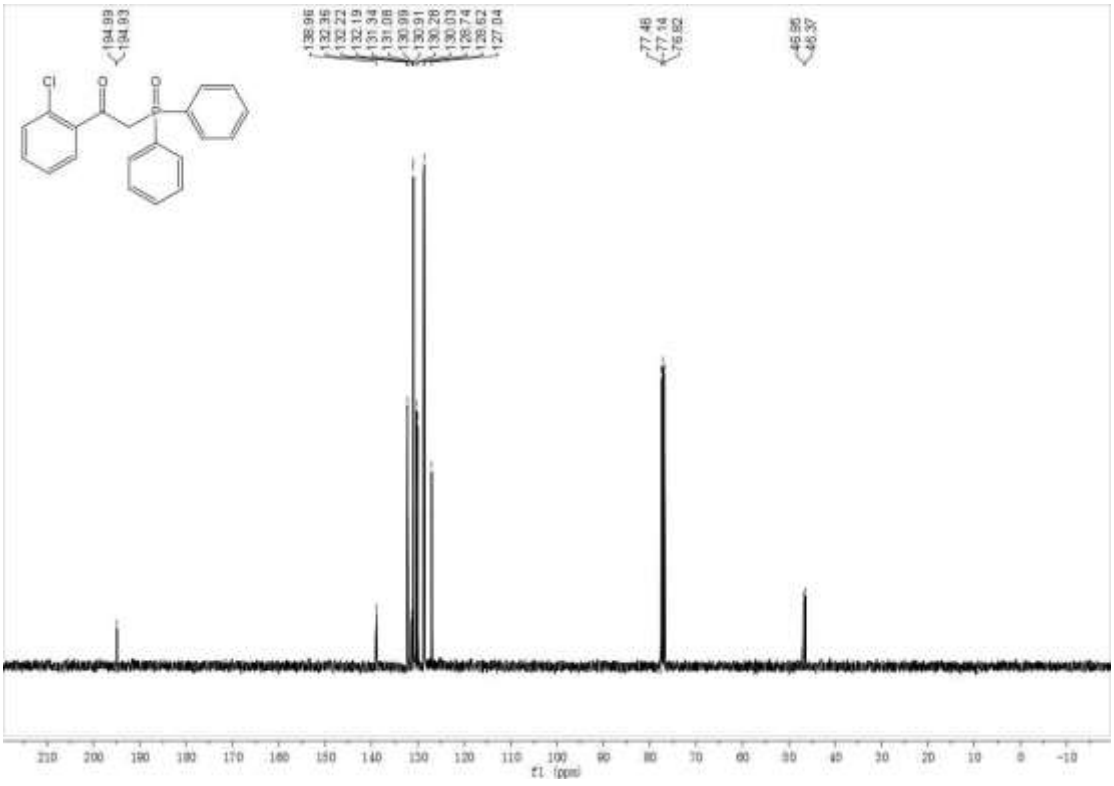


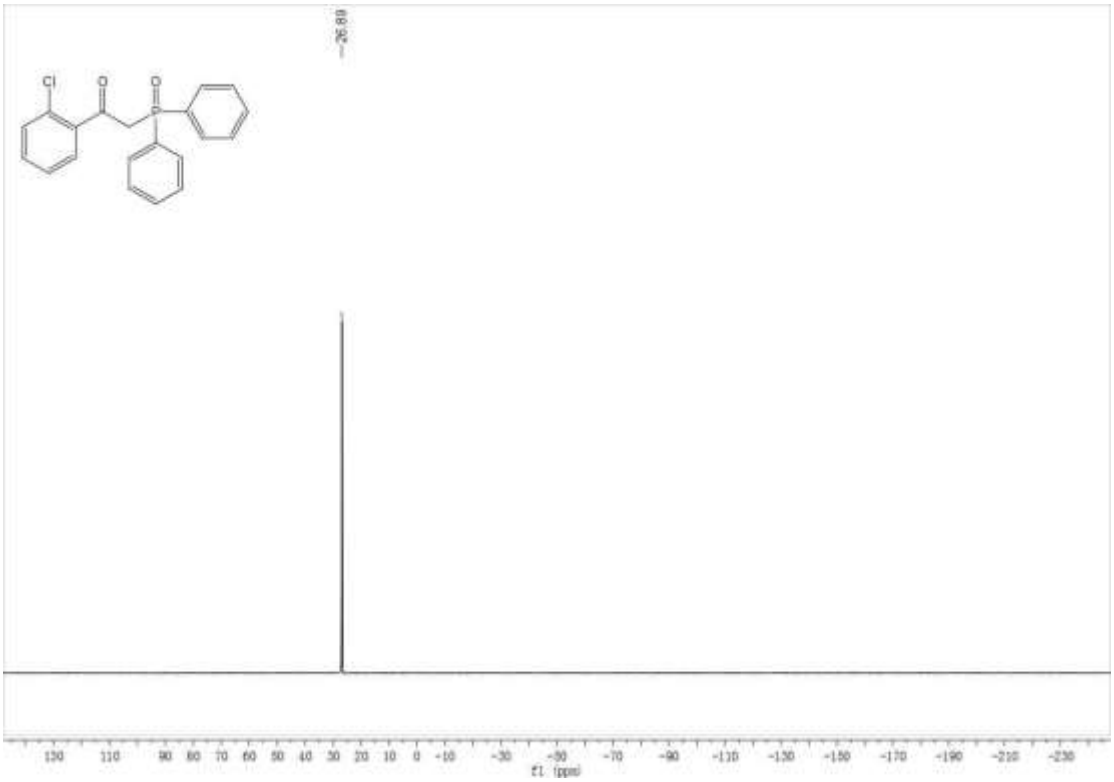


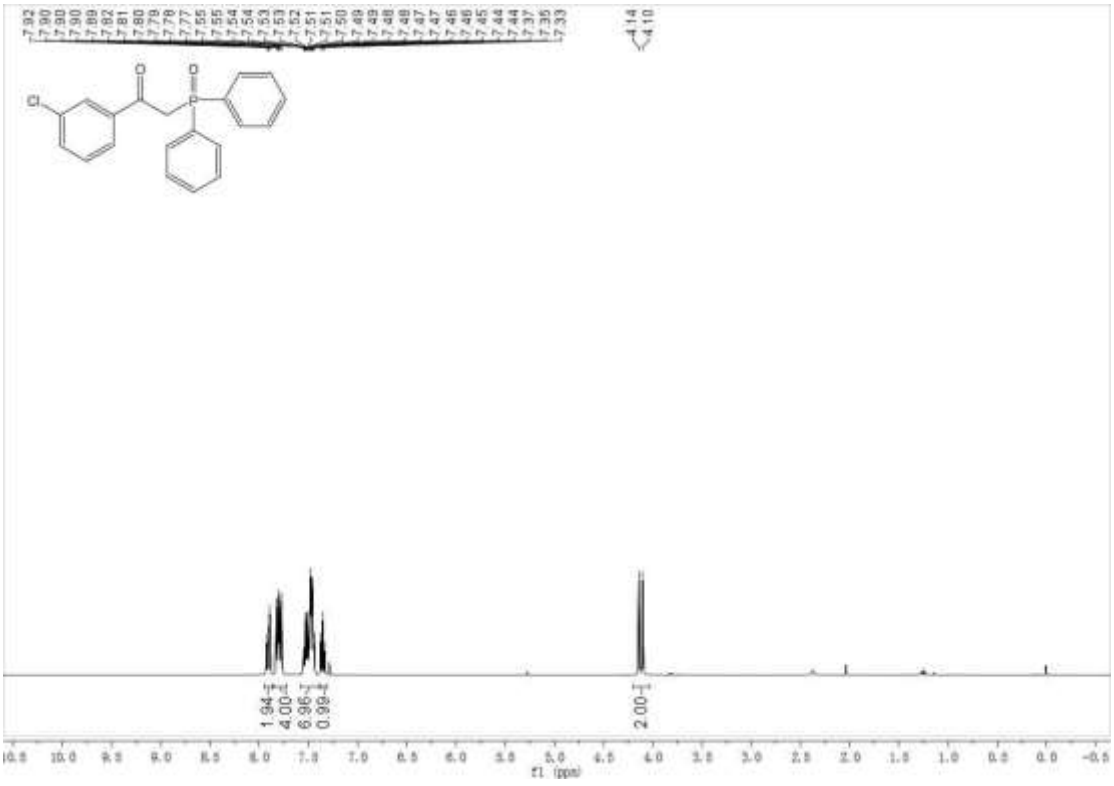

26.74
F
O
O
P
f1 (ppm)

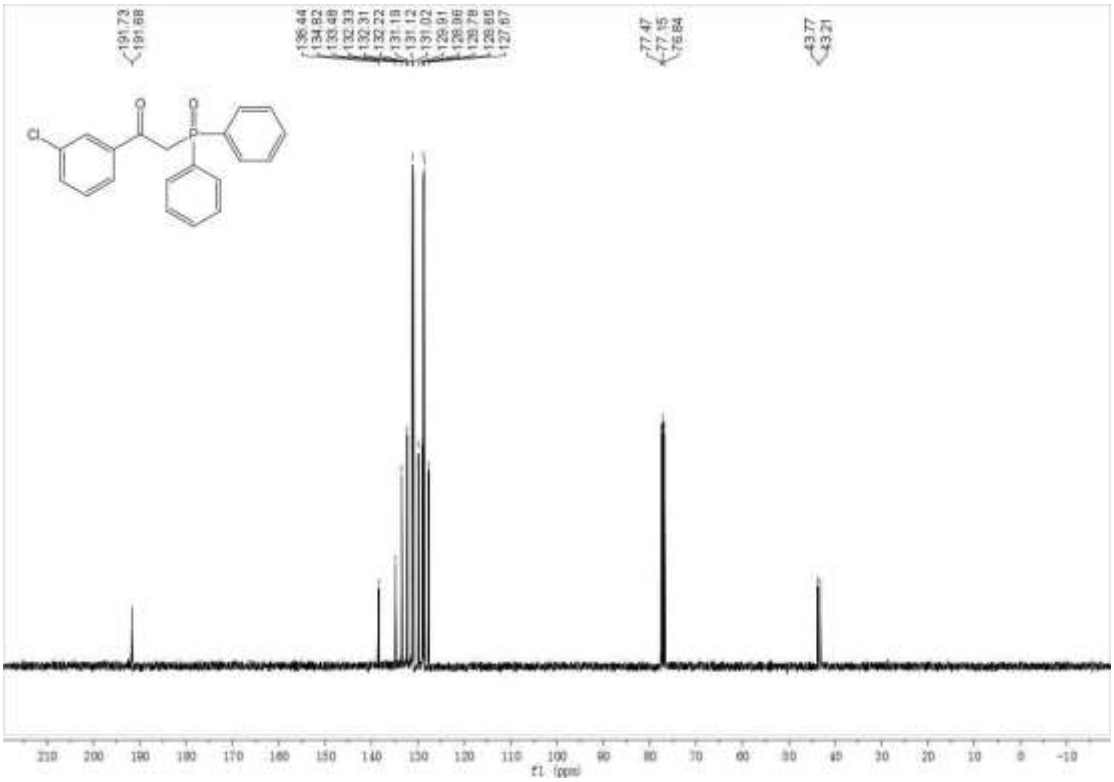

104.07
F
O
O
P
f1 (ppm)

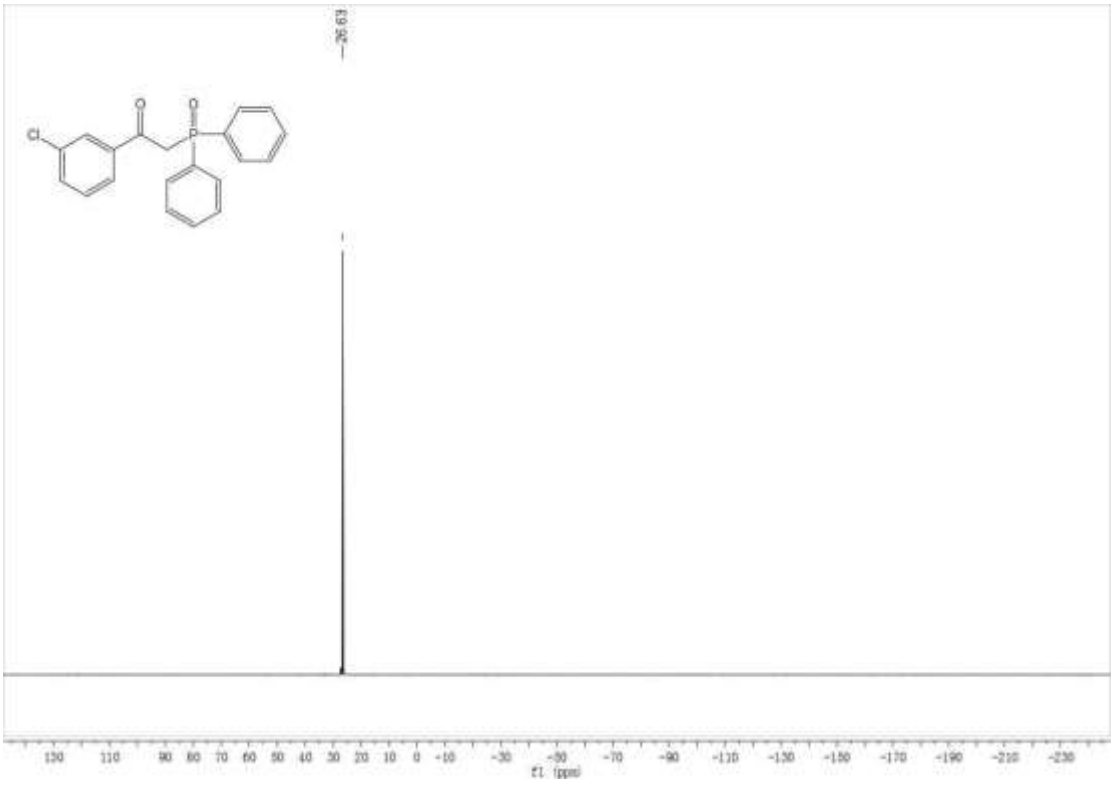


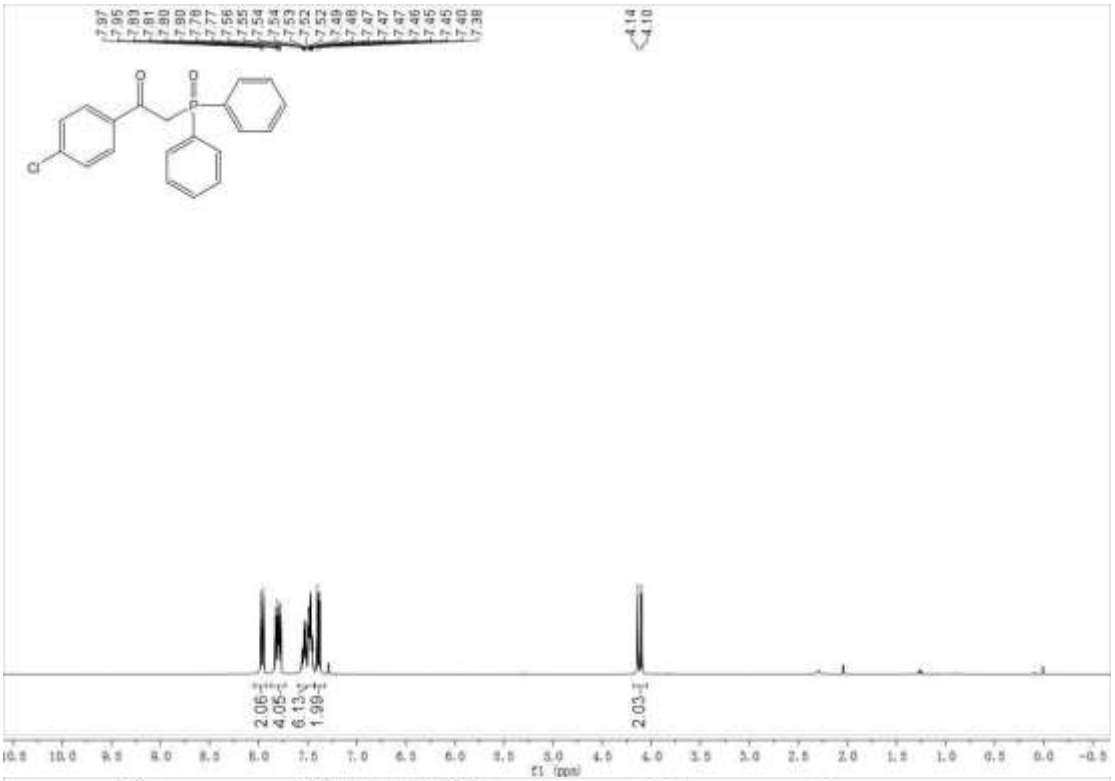


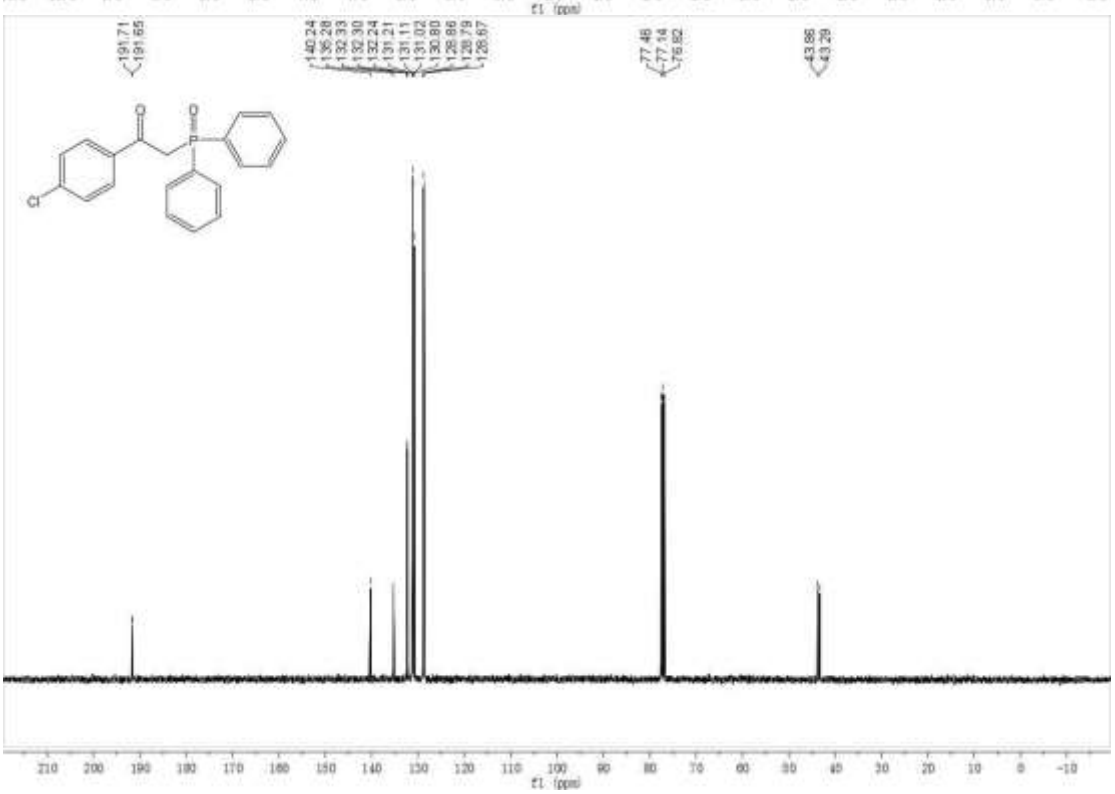

26.89
Cl
O
O
P
f1 (ppm)

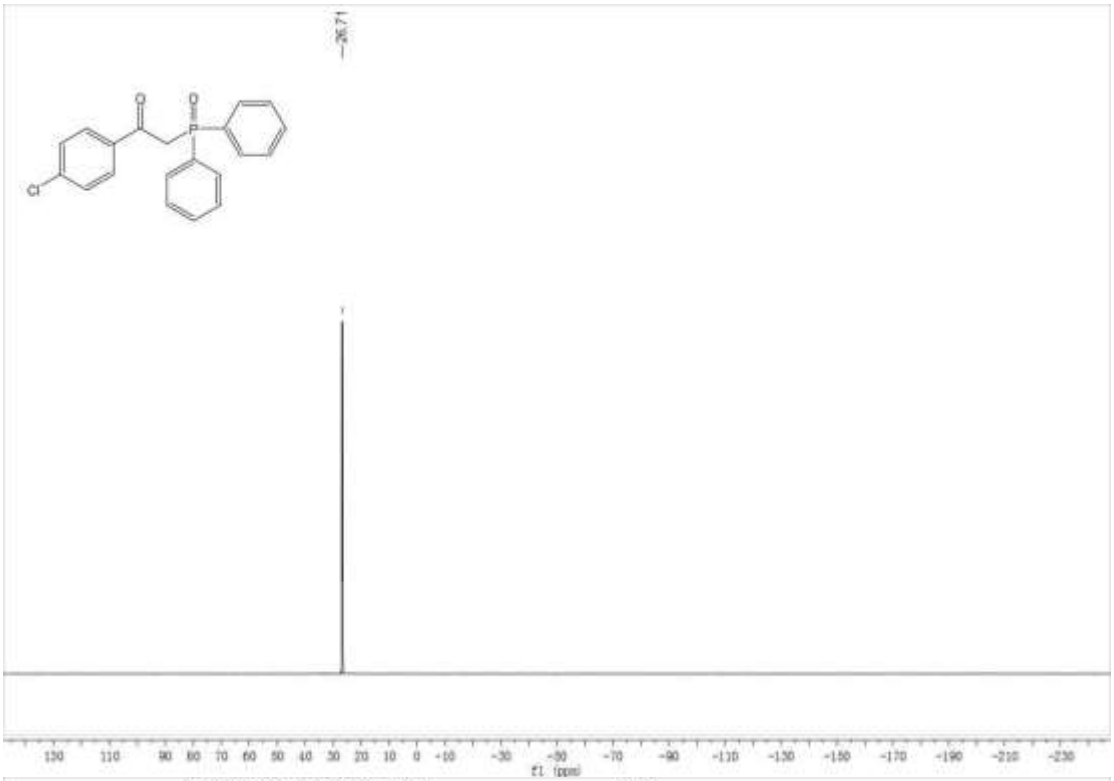

7.92
7.90
7.90
7.90
7.89
7.82
7.81
7.80
7.79
7.78
7.77
7.55
7.54
7.54
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.49
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.45
7.44
7.44
7.37
7.35
7.33
4.14
4.10
Cl
O
O
P
1.94
4.00
6.96
0.99
2.00
f1 (ppm)

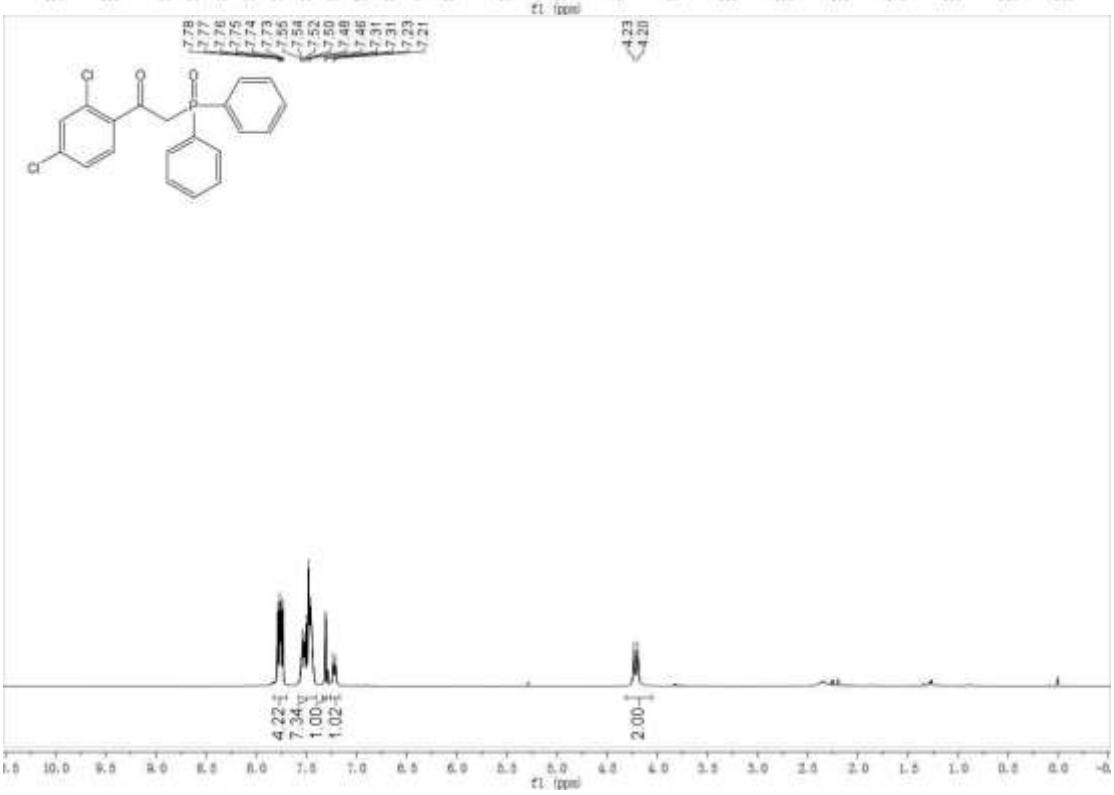

191.73
191.68
136.44
134.82
133.48
132.33
132.31
132.22
131.19
131.12
131.02
129.91
128.86
128.78
128.65
127.67
77.47
77.15
76.84
43.77
43.21
f1 (ppm)

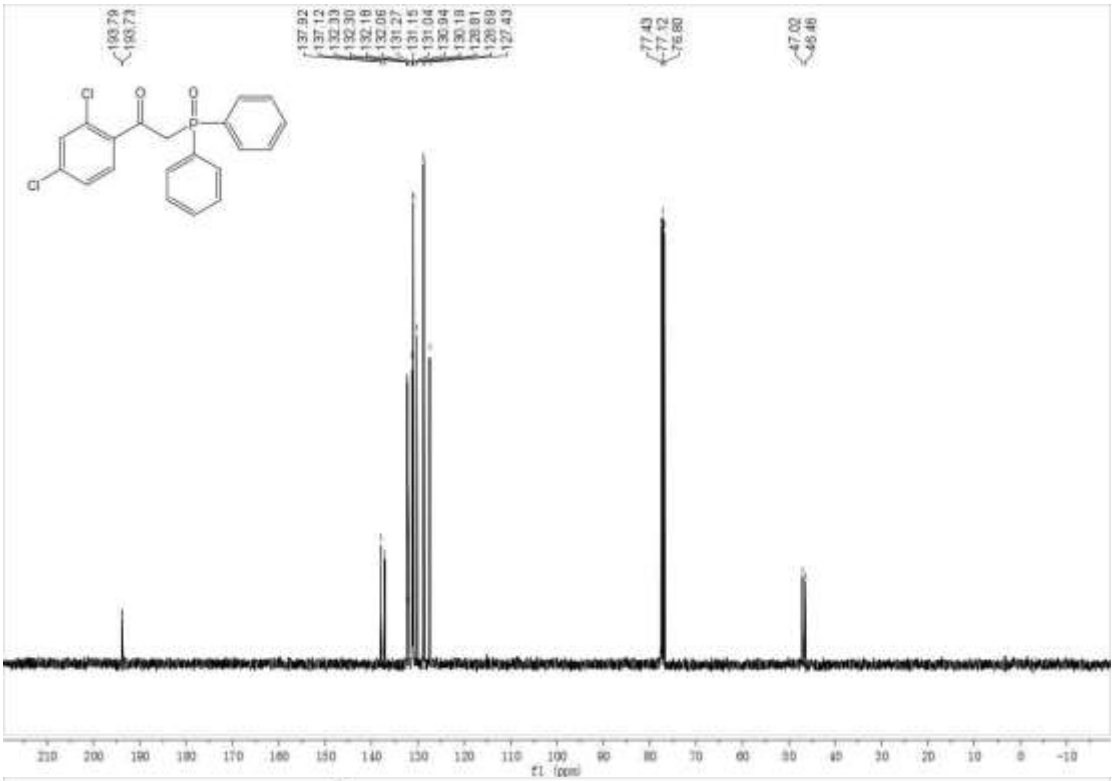

26.63
f1 (ppm)

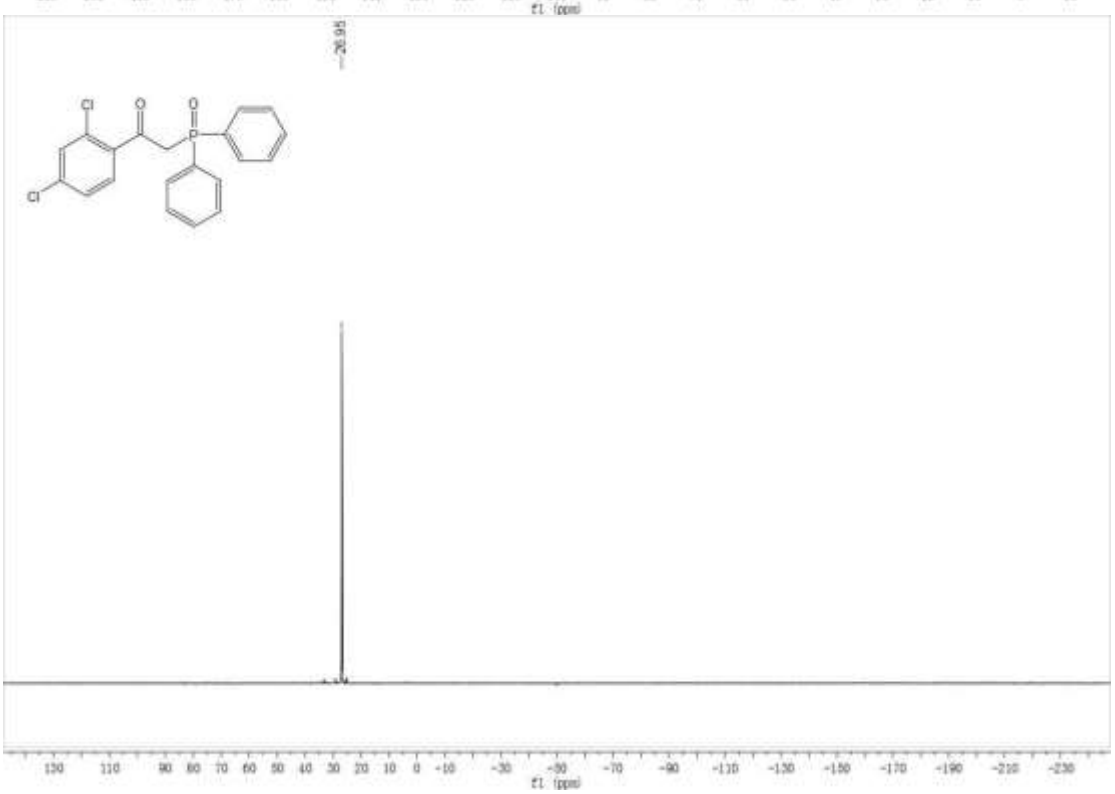

7.97
7.95
7.83
7.81
7.80
7.80
7.78
7.77
7.56
7.55
7.54
7.54
7.53
7.52
7.52
7.49
7.48
7.47
7.47
7.47
7.46
7.45
7.45
7.40
7.38
4.14
4.10
O
O
P
Cl
2.06
4.05
6.13
1.99
2.03
f1 (ppm)

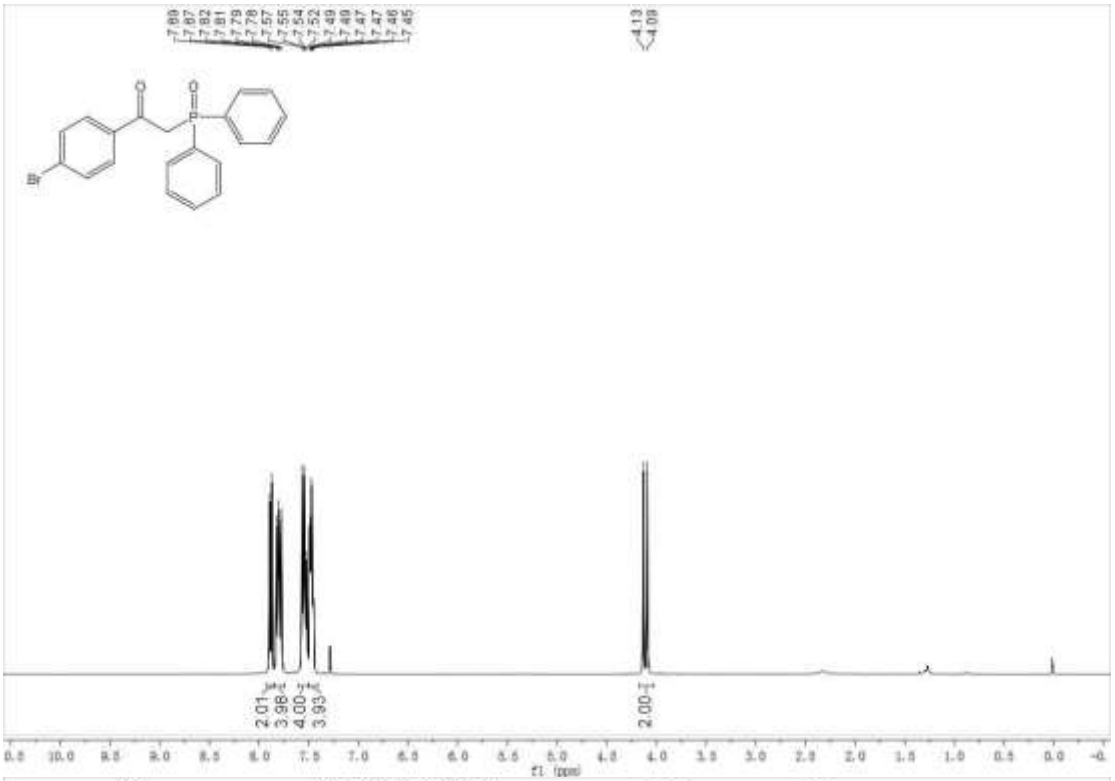

191.71
191.65
140.24
135.28
132.33
132.30
132.24
131.21
131.11
131.02
130.80
128.86
128.79
128.67
77.46
77.14
76.82
43.86
43.29
O
O
P
Cl
f1 (ppm)

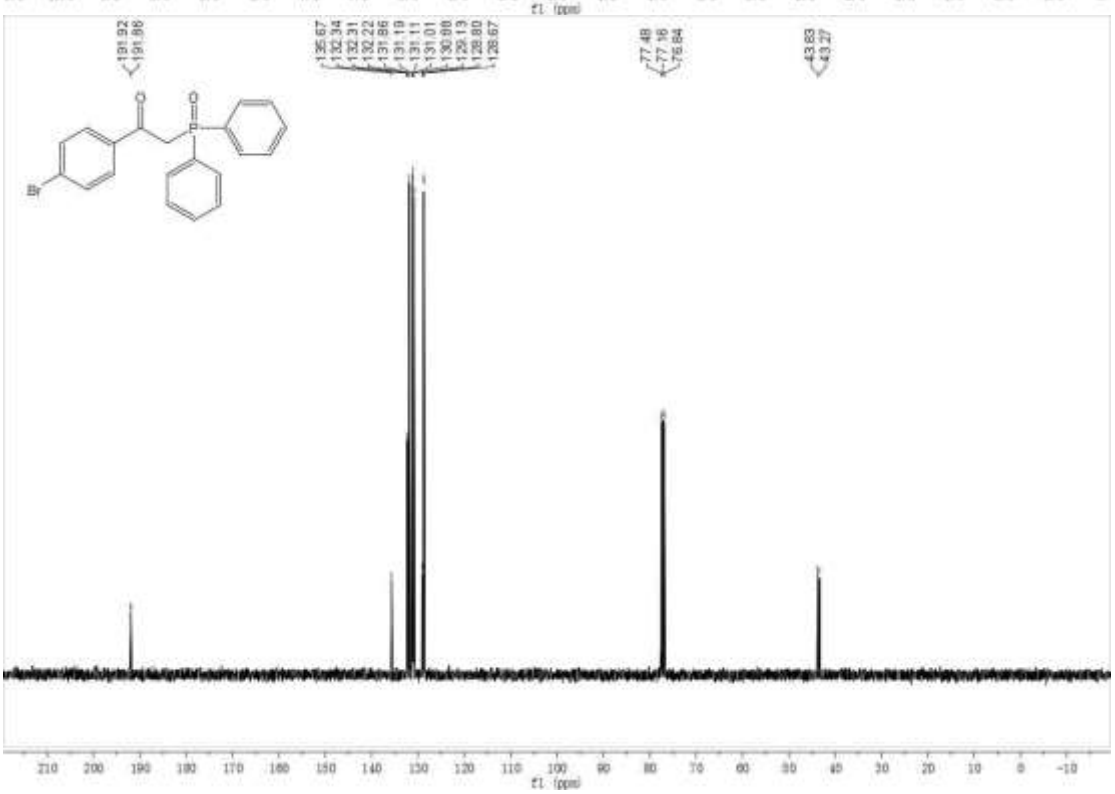

26.71
Cl
f1 (ppm)

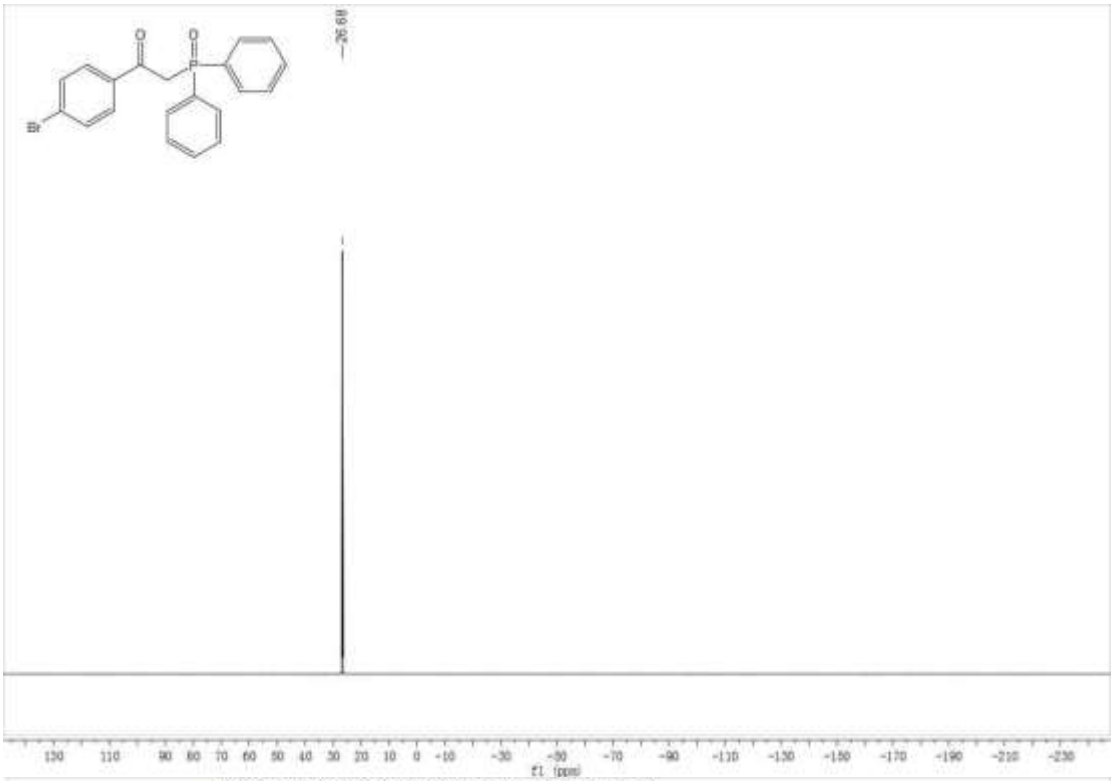

7.78
7.77
7.76
7.75
7.74
7.73
7.55
7.54
7.52
7.50
7.48
7.46
7.31
7.31
7.23
7.21
4.23
4.20
Cl
Cl
4.22
7.34
1.00
1.02
2.00
f1 (ppm)

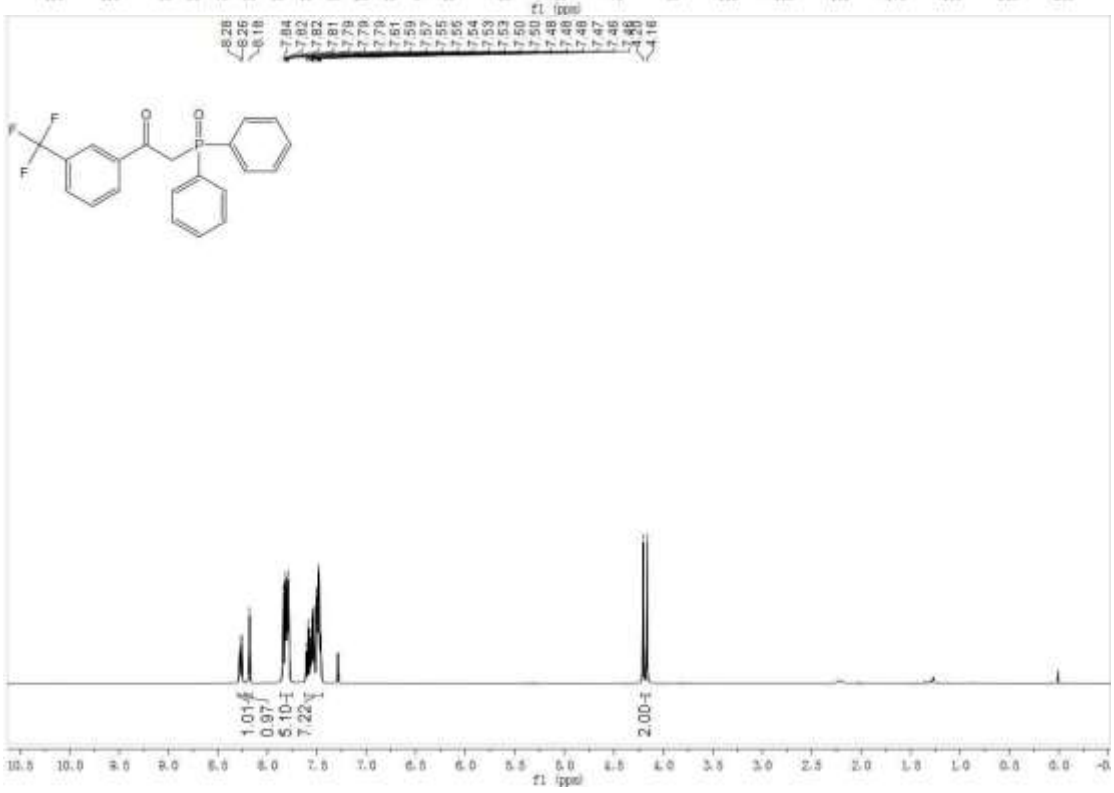

193.79
193.73
137.92
137.12
132.33
132.30
132.18
132.06
131.27
131.15
131.04
130.94
130.19
128.81
128.69
127.43
77.43
77.12
76.80
47.02
46.46
f1 (ppm)

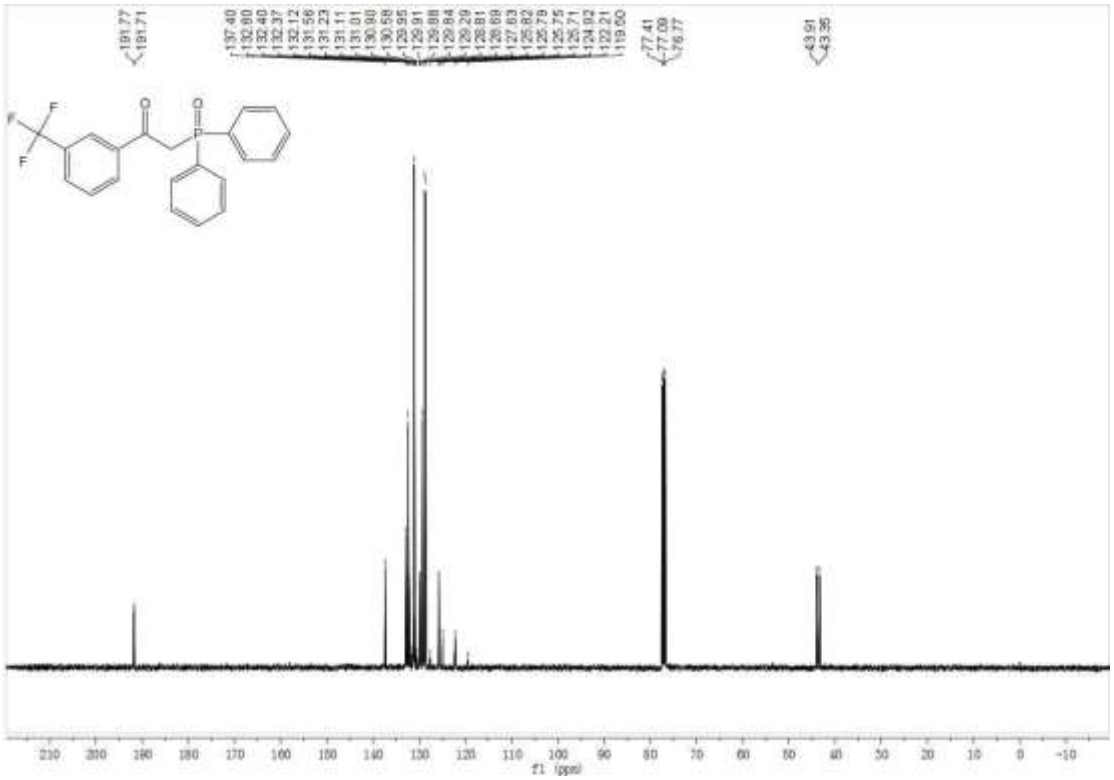

26.95
f1 (ppm)

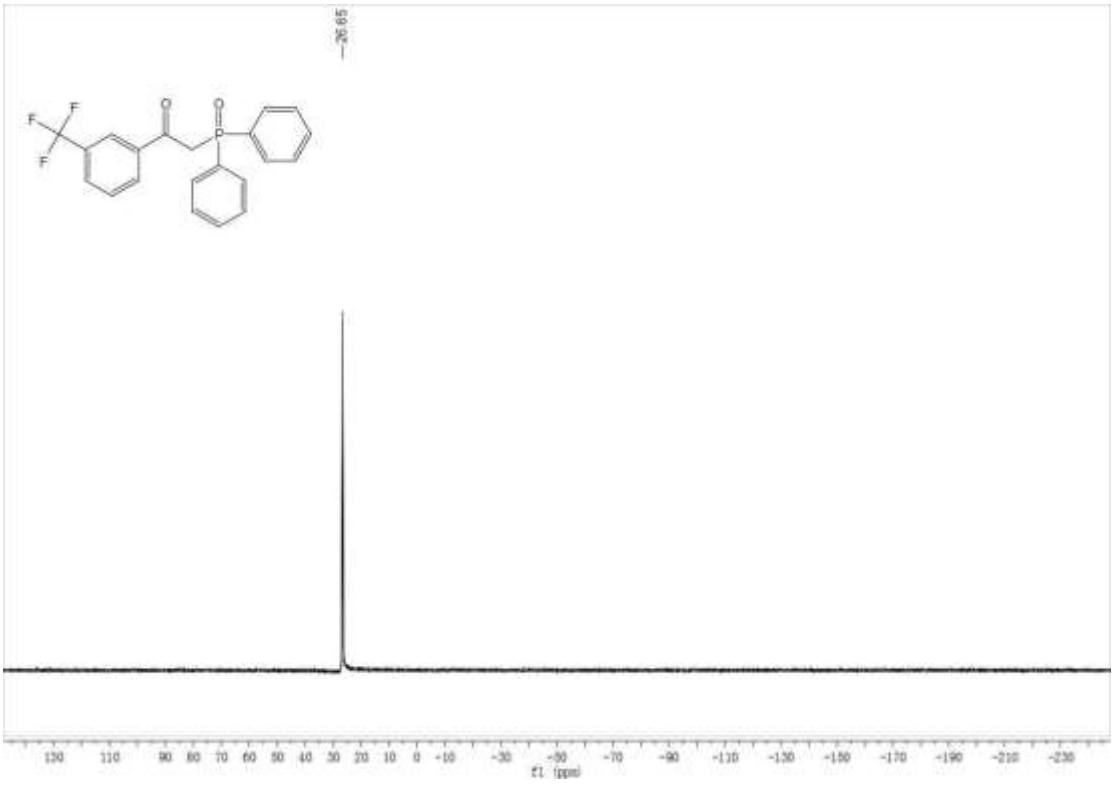

7.89
7.87
7.82
7.81
7.79
7.78
7.57
7.55
7.54
7.52
7.49
7.47
7.47
7.46
7.45
4.13
4.09
2.01
3.98
4.00
3.93
2.00
f1 (ppm)

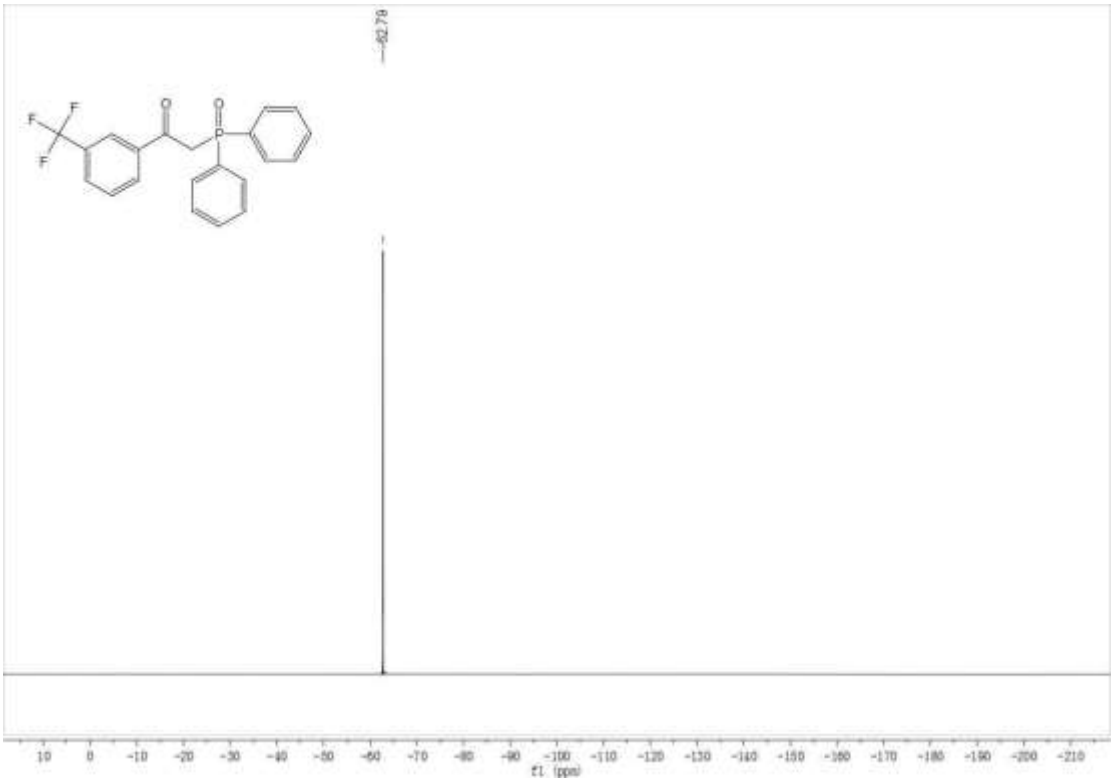

191.92
191.86
135.67
132.34
132.31
132.22
131.86
131.19
131.11
131.01
130.88
129.13
128.80
128.67
77.48
77.16
76.84
43.63
43.27
f1 (ppm)

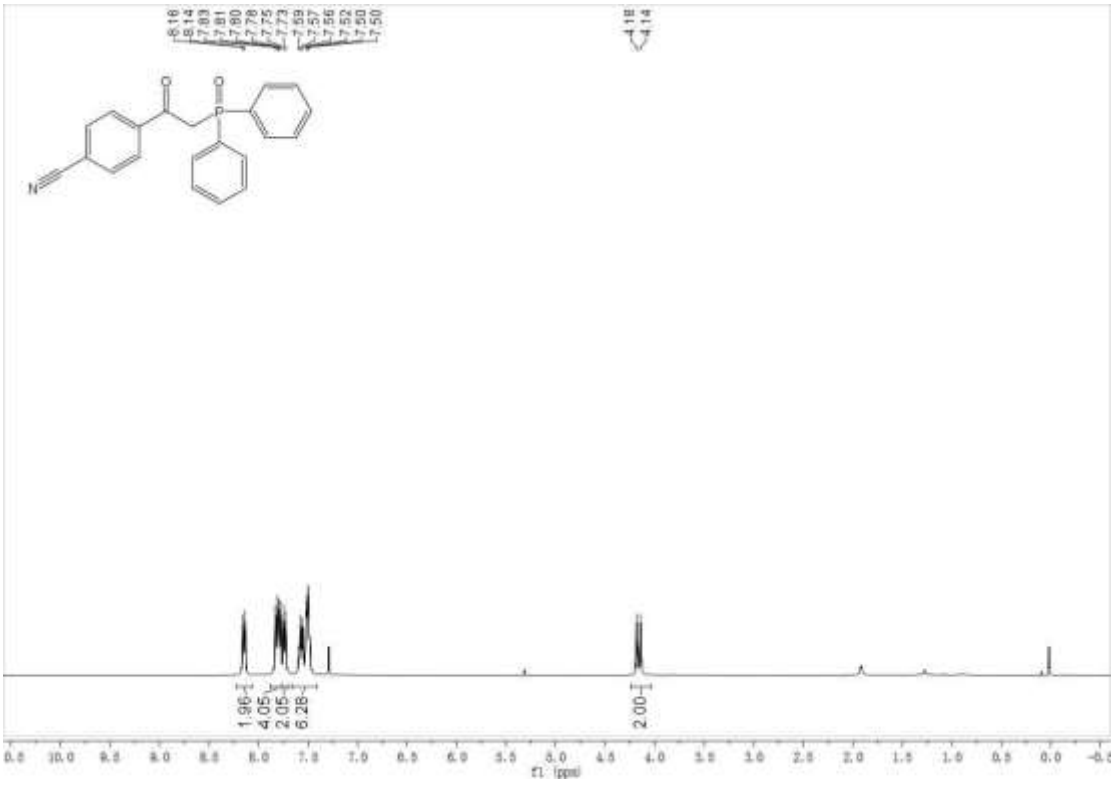

26.69
f1 (ppm)

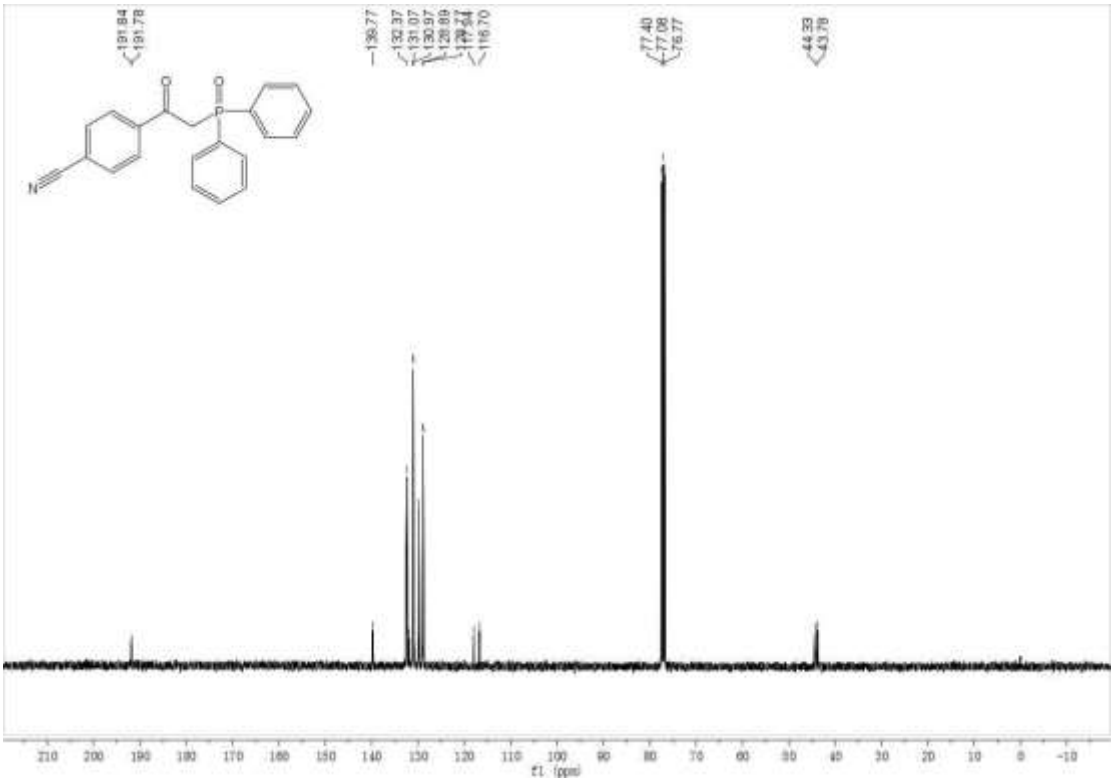

8.28
8.25
8.18
7.84
7.82
7.82
7.81
7.79
7.79
7.79
7.61
7.59
7.57
7.55
7.55
7.54
7.53
7.53
7.50
7.50
7.48
7.48
7.48
7.47
7.46
7.46
7.45
7.16
1.01
0.97
5.10
7.22
2.00
f1 (ppm)

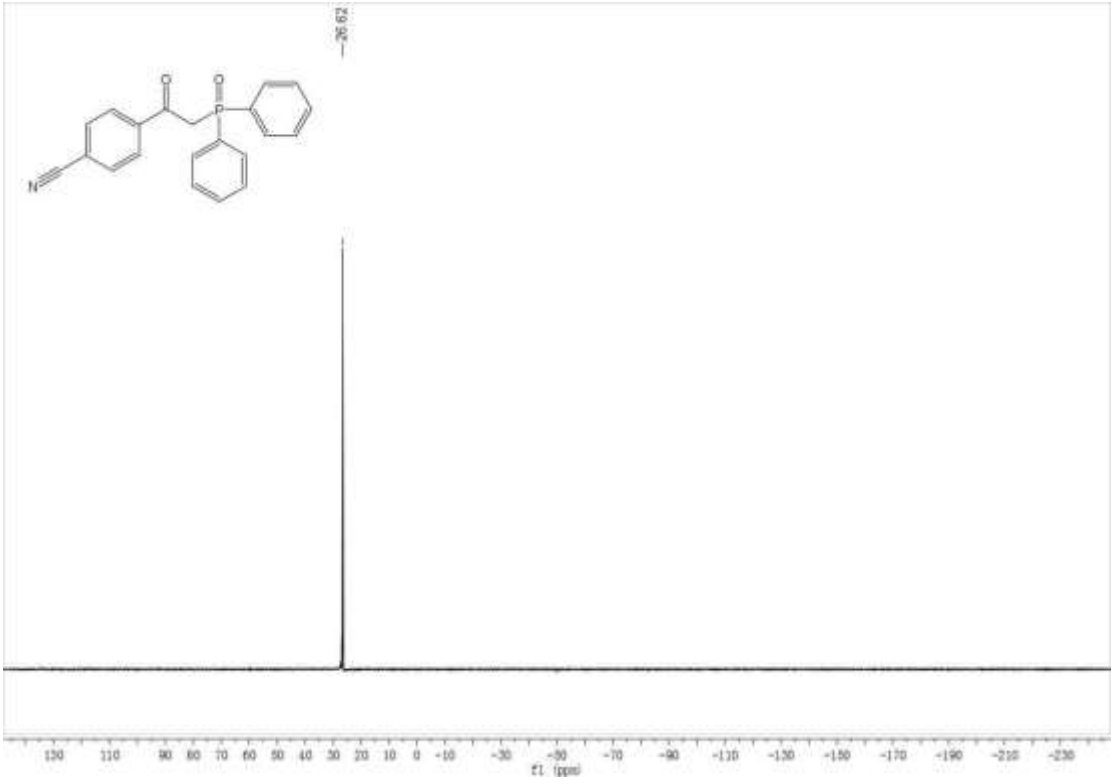

191.77
191.71
137.40
132.80
132.40
132.37
132.12
131.56
131.23
131.11
131.01
130.98
130.58
129.95
129.91
129.88
129.84
129.29
128.81
128.69
127.63
125.82
125.78
125.75
125.71
124.02
122.21
119.50
77.41
77.09
76.77
43.91
43.35
F
F
F
O
O
P
f1 (ppm)

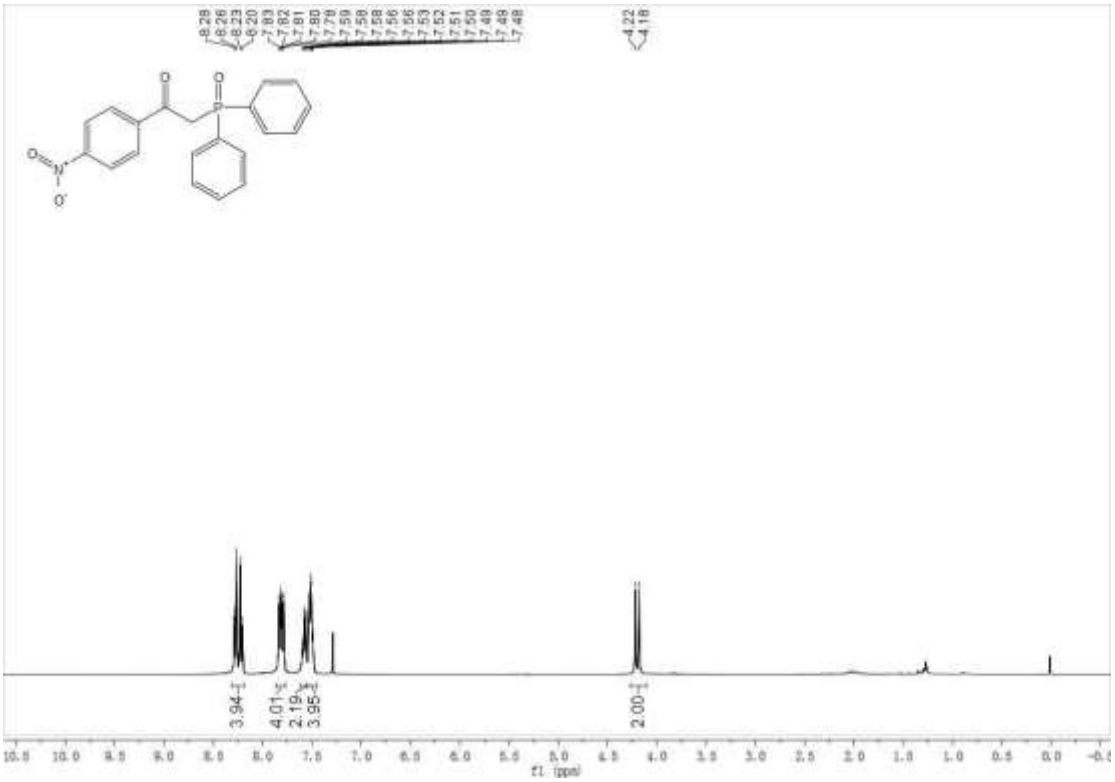

26.65
F
F
F
O
O
P
f1 (ppm)

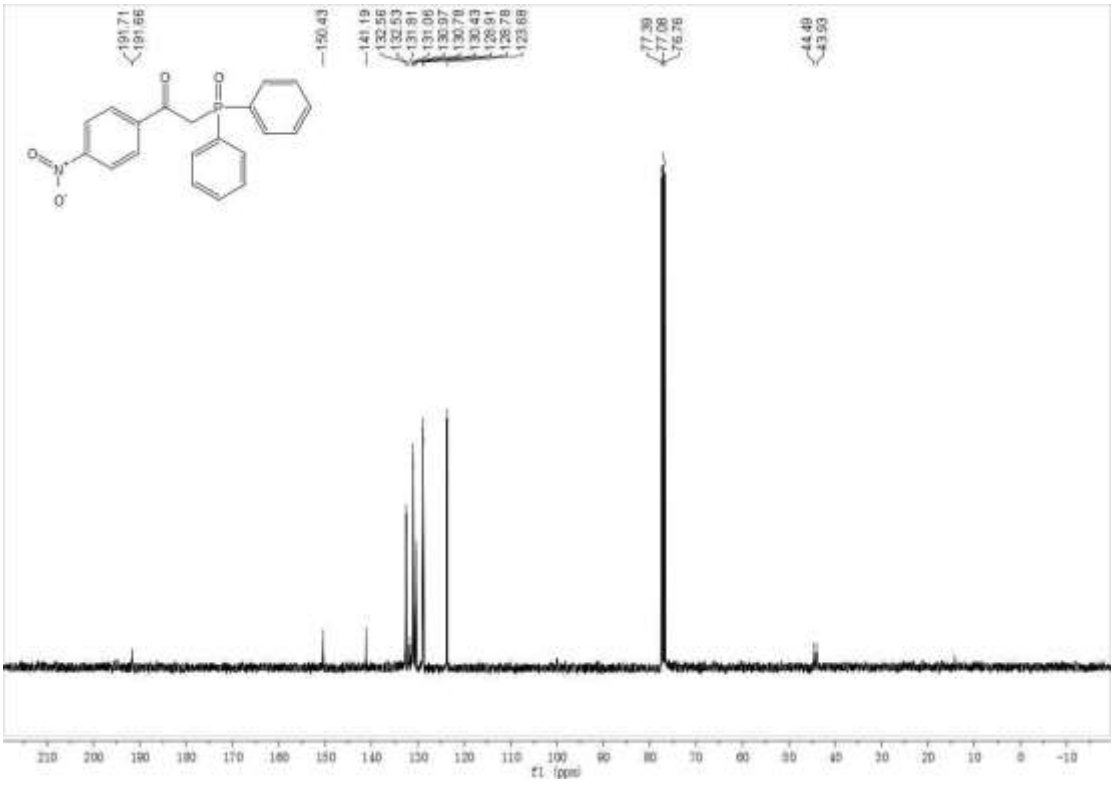

-62.79
f1 (ppm)

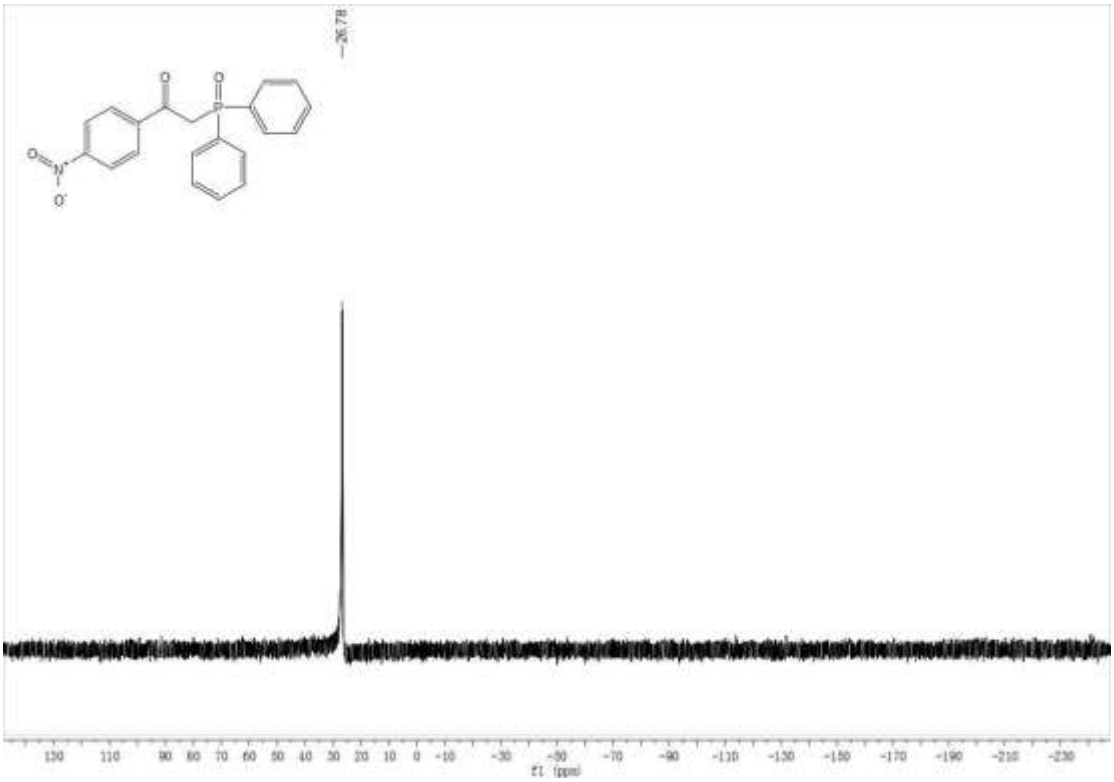

8.16
8.14
7.83
7.81
7.80
7.78
7.75
7.73
7.59
7.57
7.56
7.52
7.50
7.50
4.18
4.14
1.96
4.05
2.05
6.28
2.00
f1 (ppm)

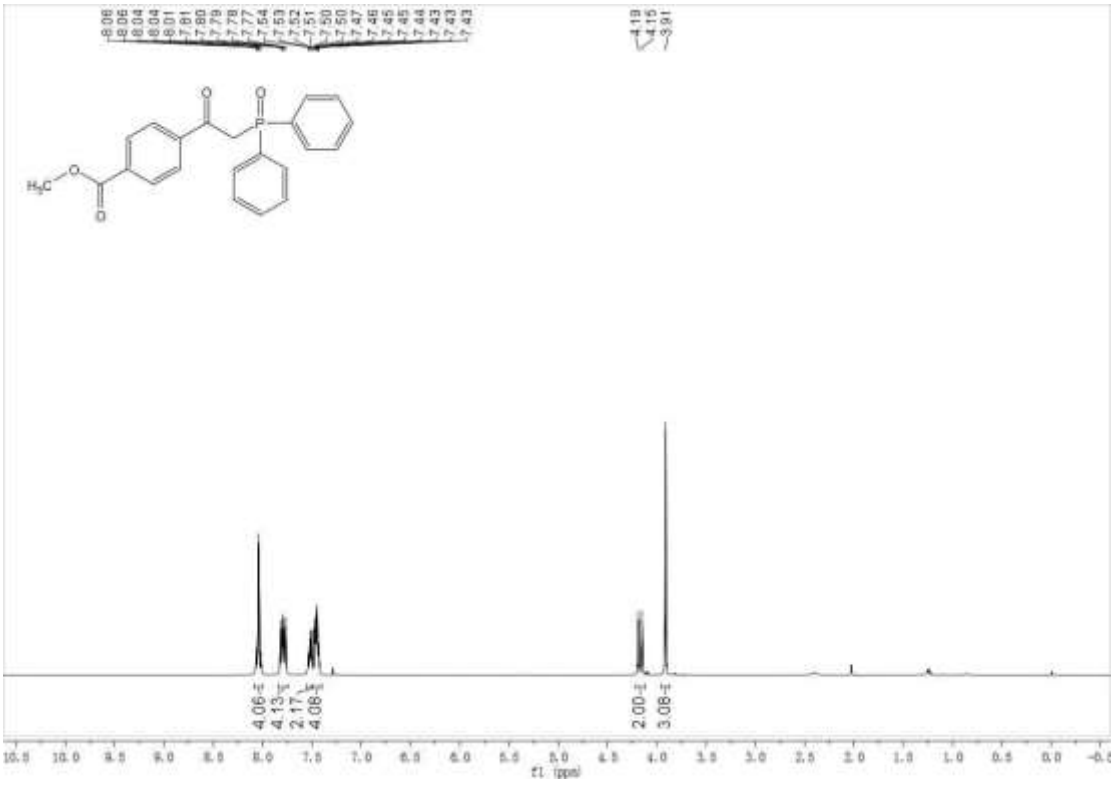

191.84
191.78
139.77
132.37
131.07
130.97
128.89
119.44
116.70
77.40
77.08
76.77
44.33
43.78
f1 (ppm)

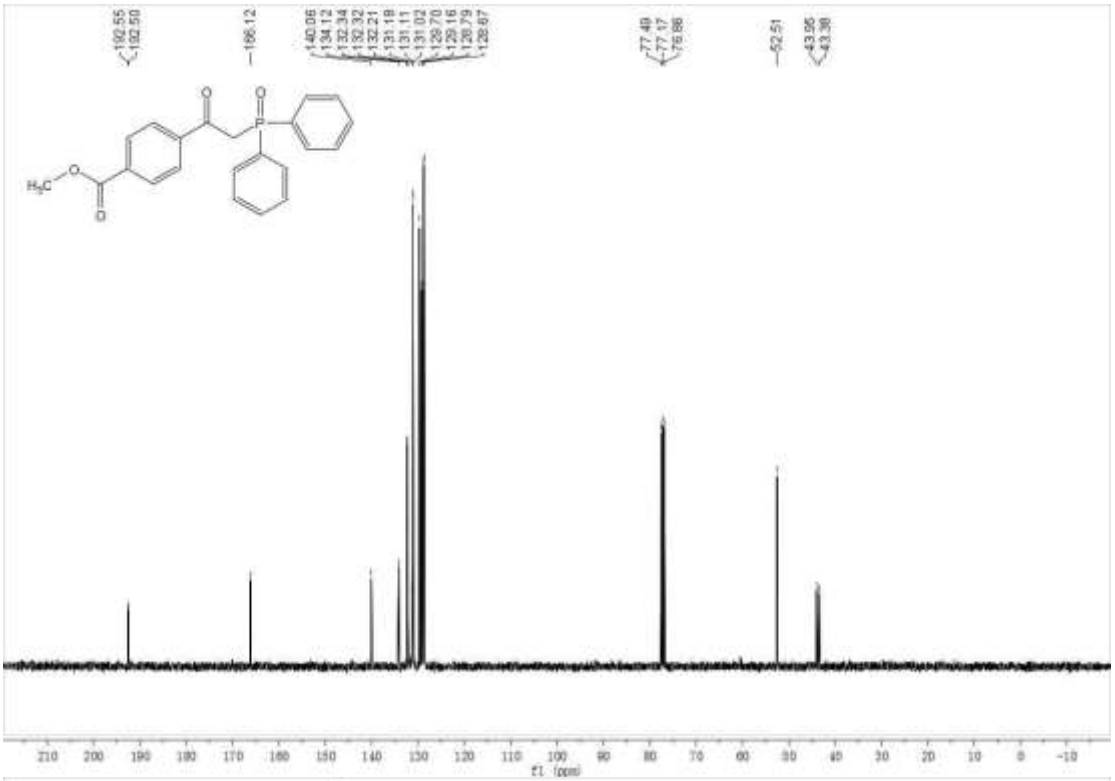

26.62
f1 (ppm)

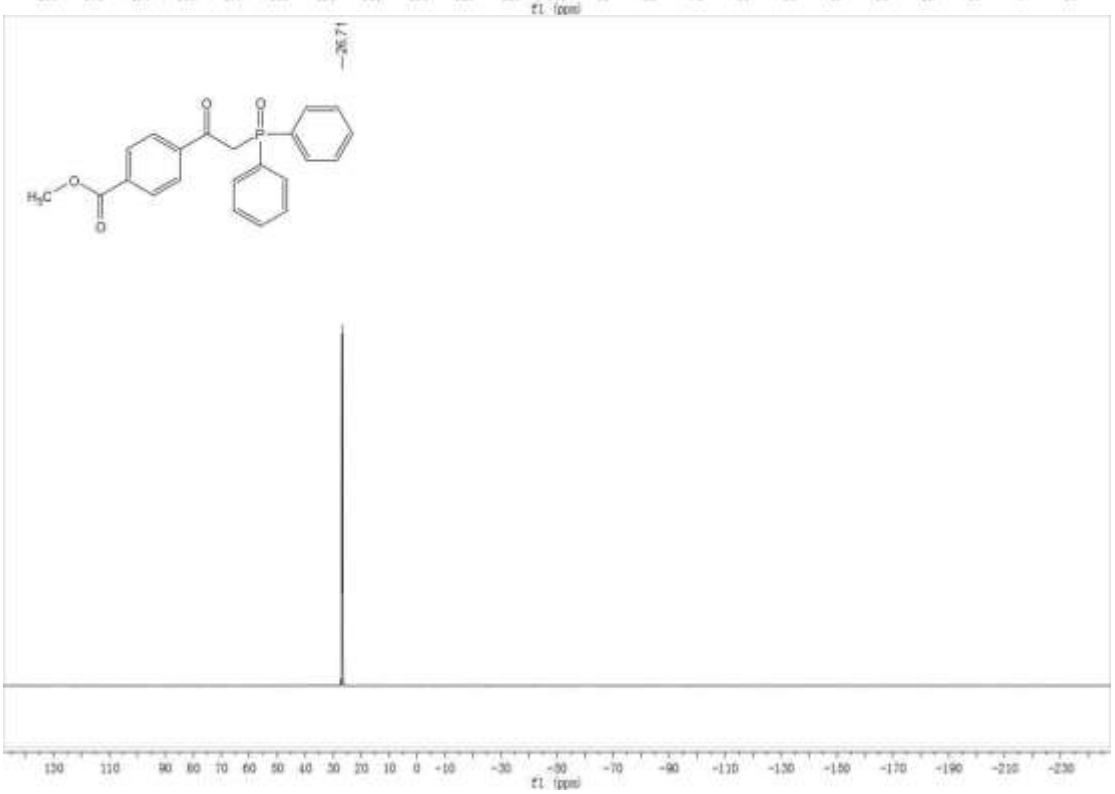

8.28
8.26
8.23
8.20
7.83
7.82
7.81
7.80
7.79
7.59
7.58
7.56
7.56
7.53
7.52
7.51
7.50
7.49
7.49
7.48
4.22
4.18
3.94
4.01
2.19
3.95
2.00
f1 (ppm)

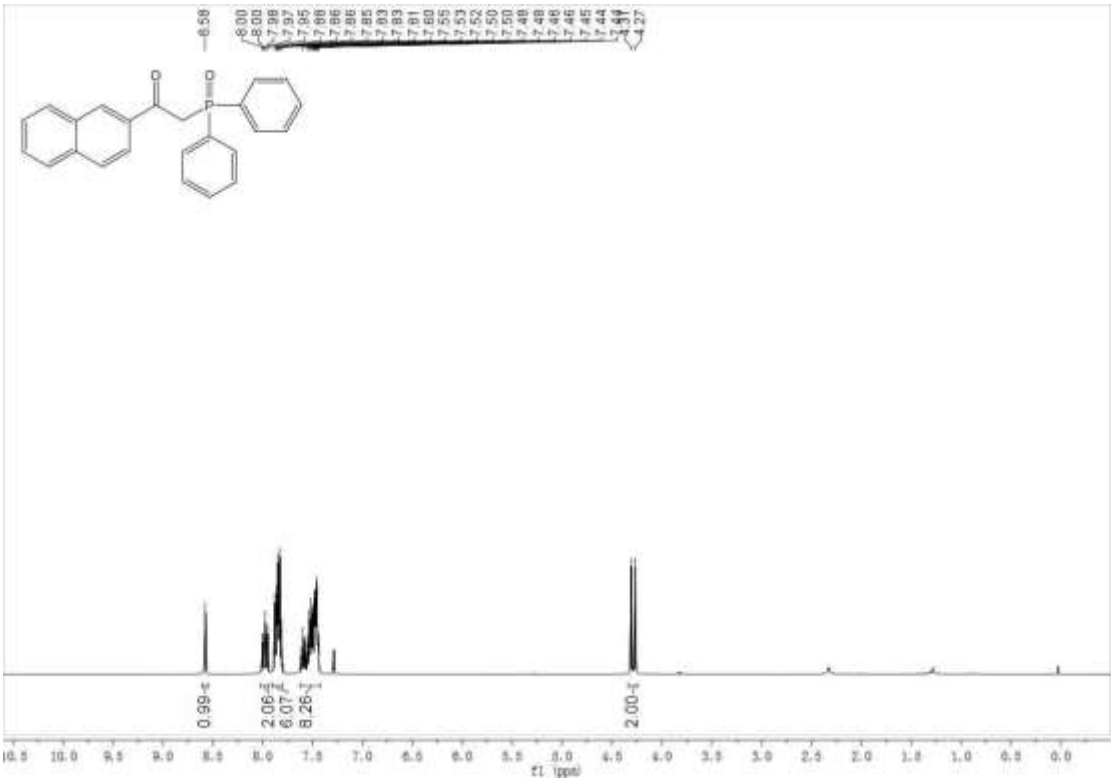

191.71
191.66
150.43
141.19
132.56
132.53
131.81
131.06
130.97
130.78
130.43
128.91
128.78
123.68
77.39
77.08
76.76
44.49
43.93
f1 (ppm)

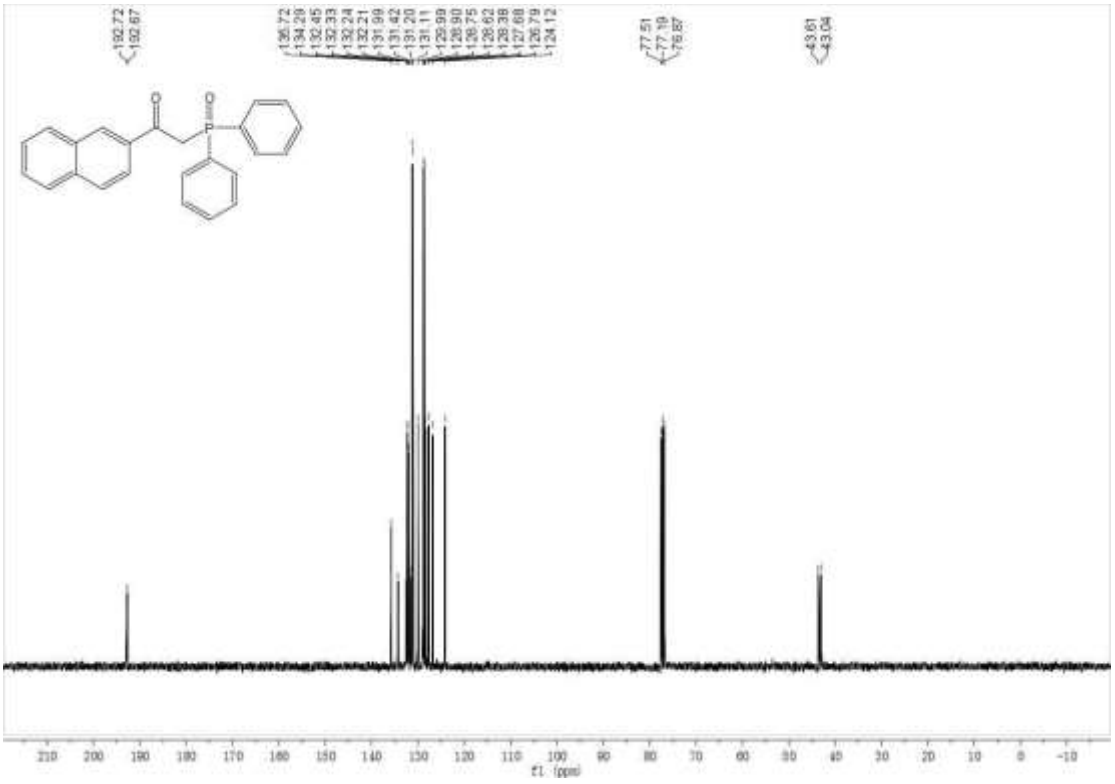

26.78
f1 (ppm)

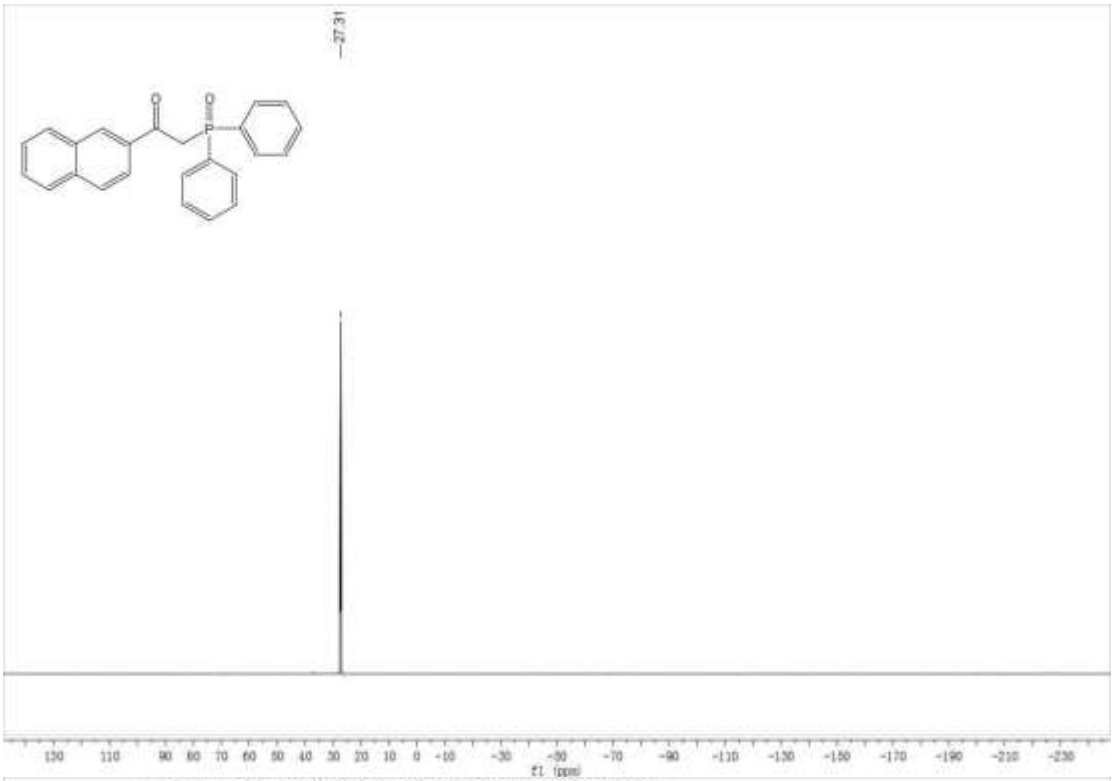

8.06
8.06
8.04
8.04
8.01
7.81
7.80
7.79
7.78
7.77
7.54
7.53
7.52
7.51
7.50
7.50
7.47
7.46
7.45
7.45
7.44
7.43
7.43
7.43
4.19
4.15
3.81
4.06
4.13
2.17
4.08
2.00
3.08
f1 (ppm)

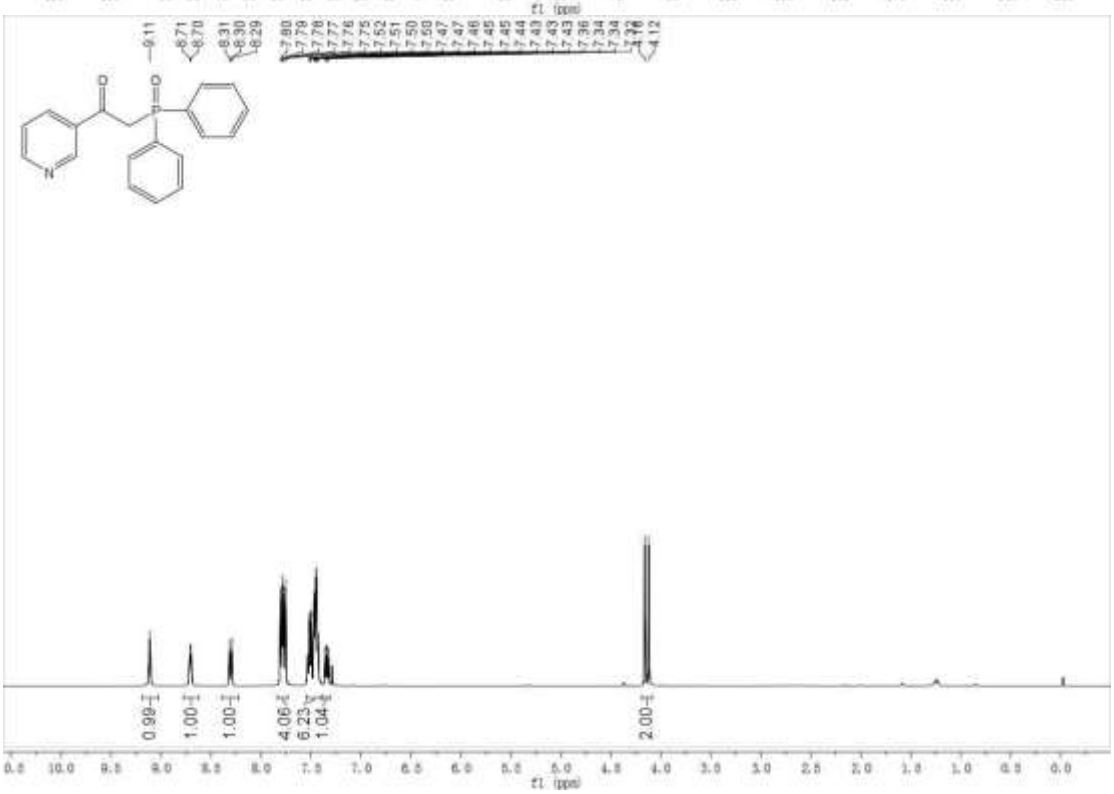

192.55
192.50
166.12
140.06
134.12
132.34
132.32
132.21
131.18
131.11
131.02
129.70
129.16
128.79
128.67
77.49
77.17
76.86
52.51
43.96
43.38
f1 (ppm)

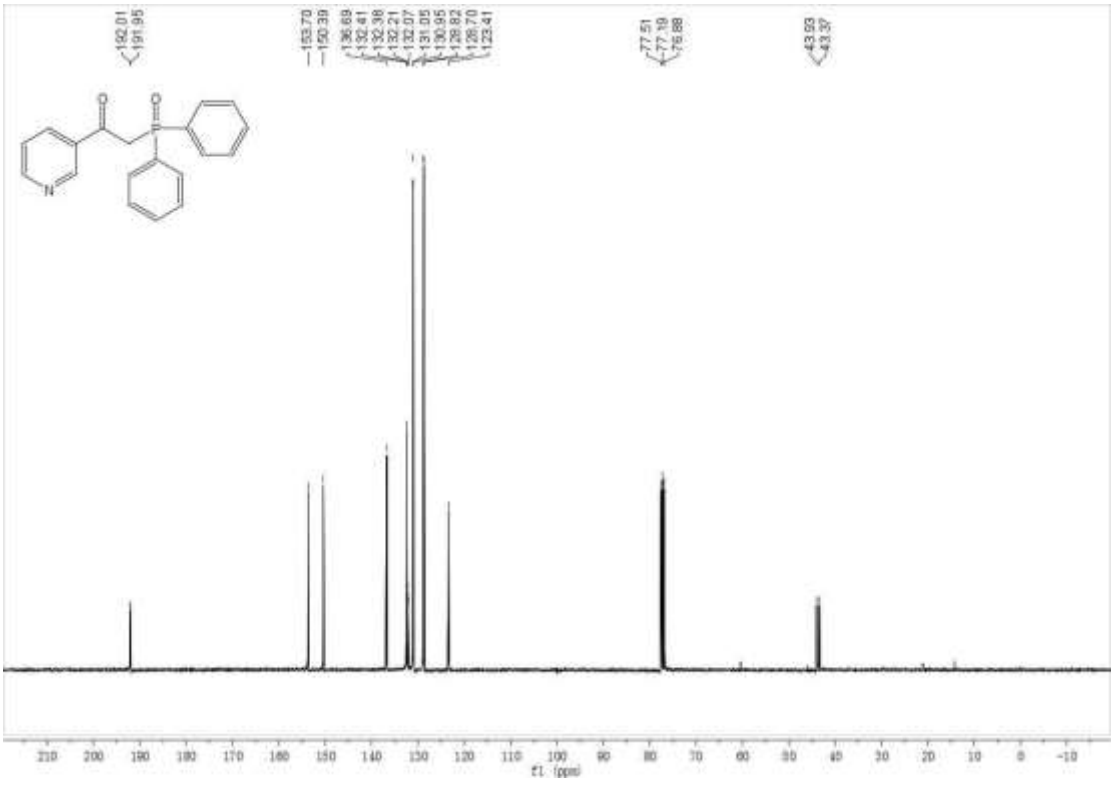

26.71
f1 (ppm)

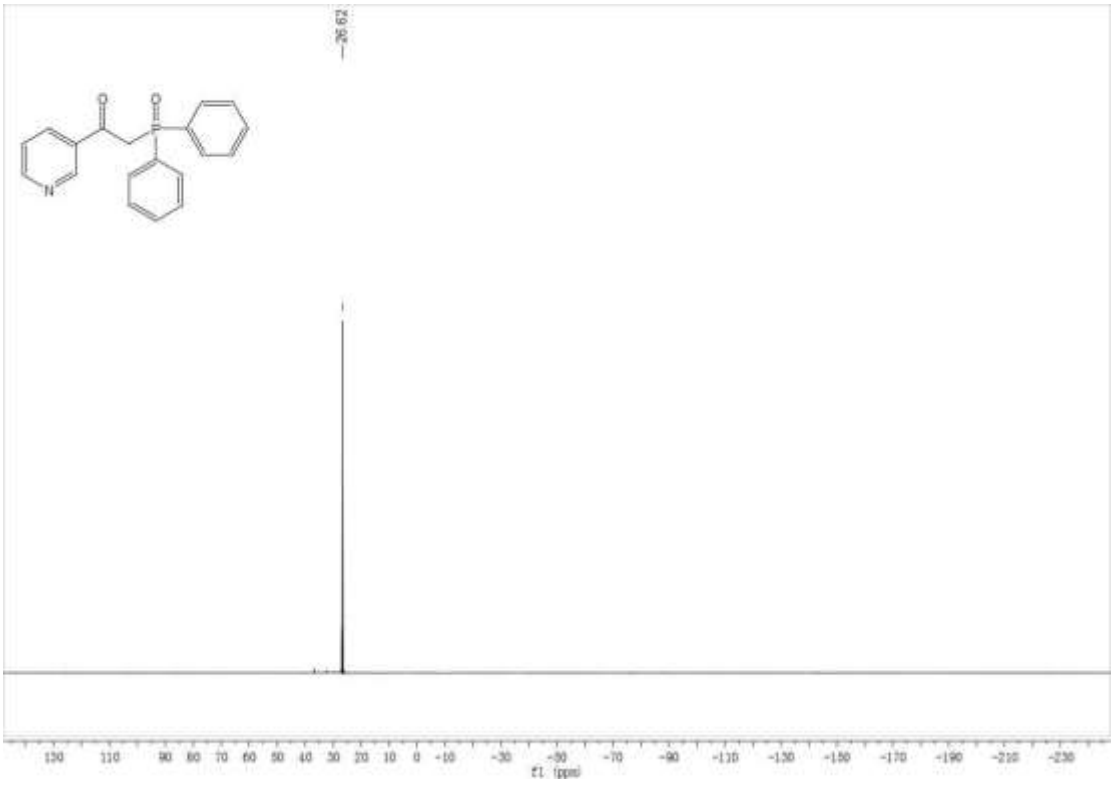


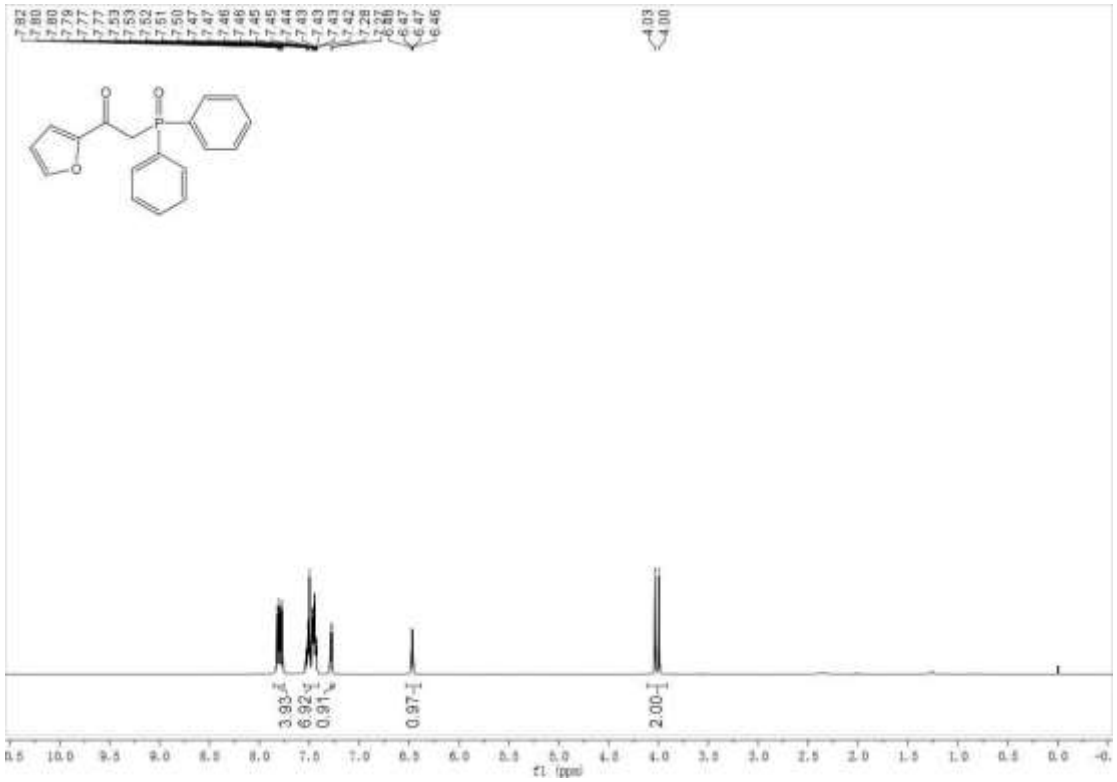


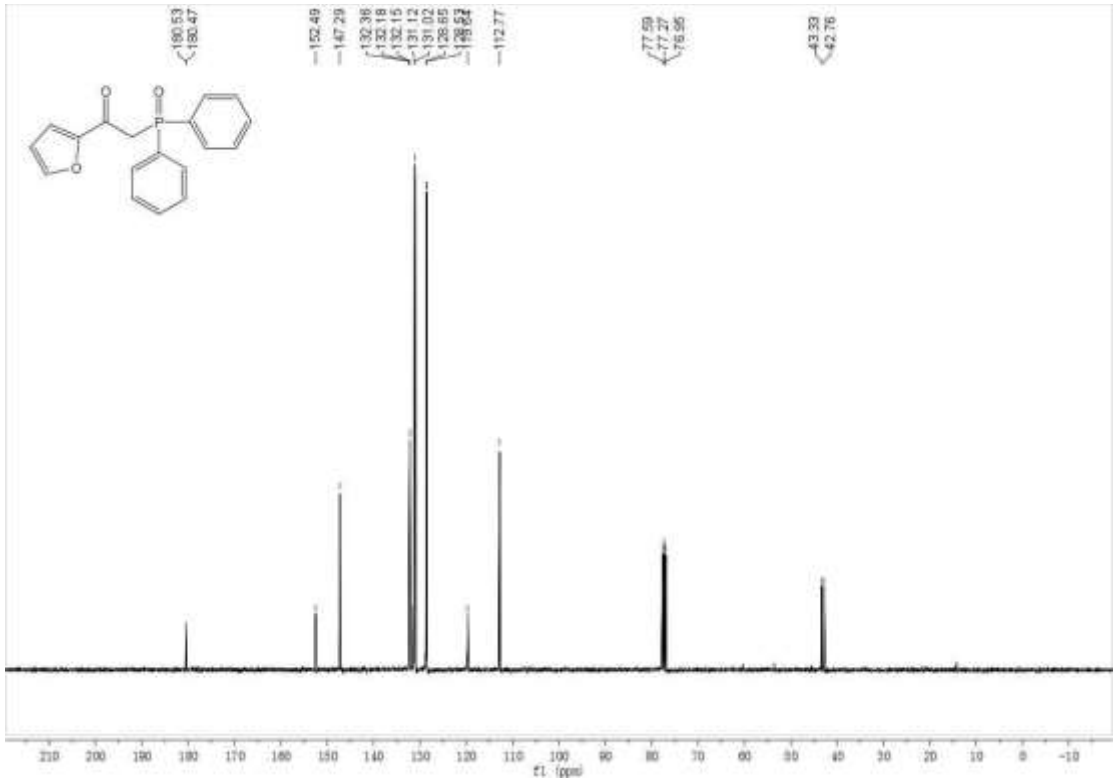

27.31
f1 (ppm)

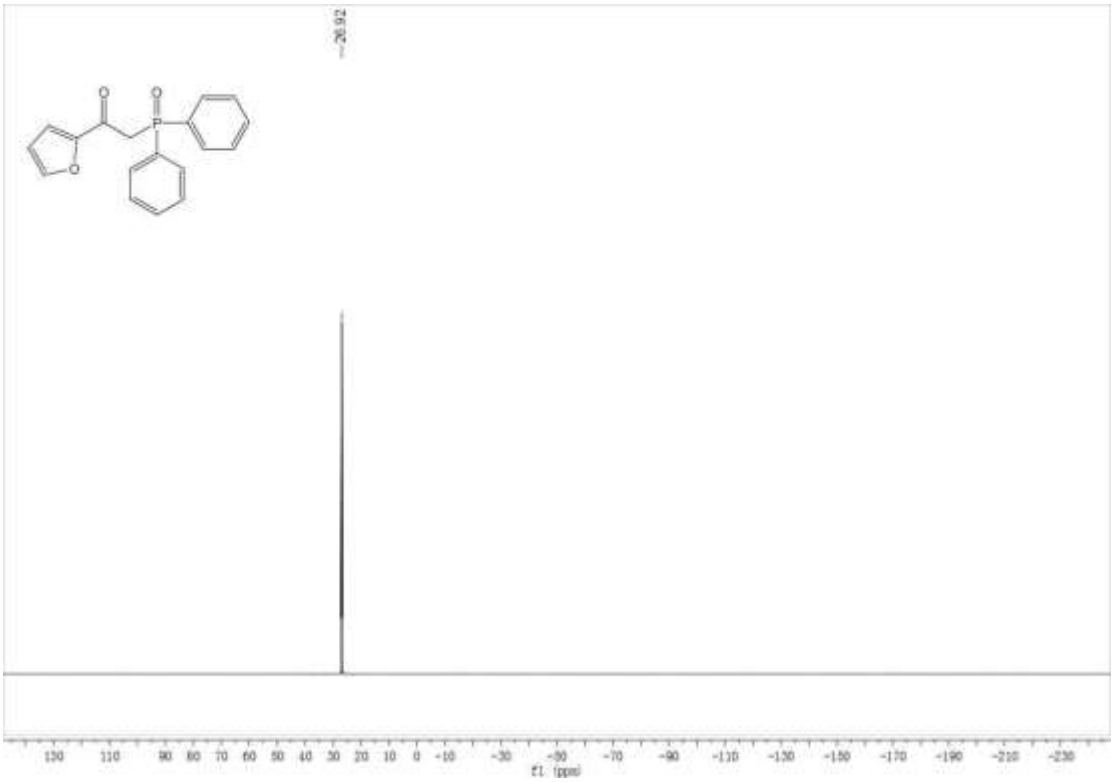

9.11
8.71
8.70
8.31
8.30
8.29
7.80
7.79
7.78
7.77
7.76
7.75
7.52
7.51
7.50
7.50
7.47
7.47
7.46
7.45
7.45
7.44
7.43
7.43
7.43
7.36
7.34
7.34
4.12
0.99
1.00
1.00
4.06
6.23
1.04
2.00
f1 (ppm)

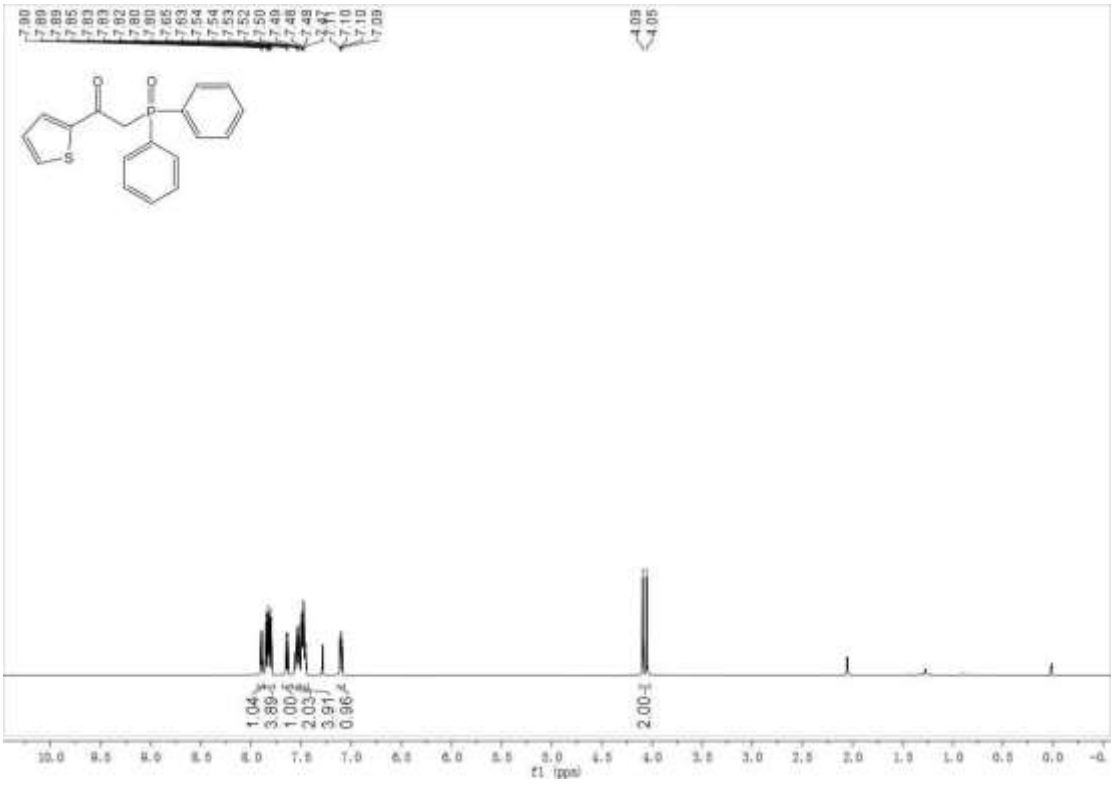

192.01
191.95
153.70
150.39
136.69
132.41
132.38
132.21
132.07
131.05
130.95
128.82
128.70
123.41
77.51
77.19
76.88
43.93
43.37
f1 (ppm)

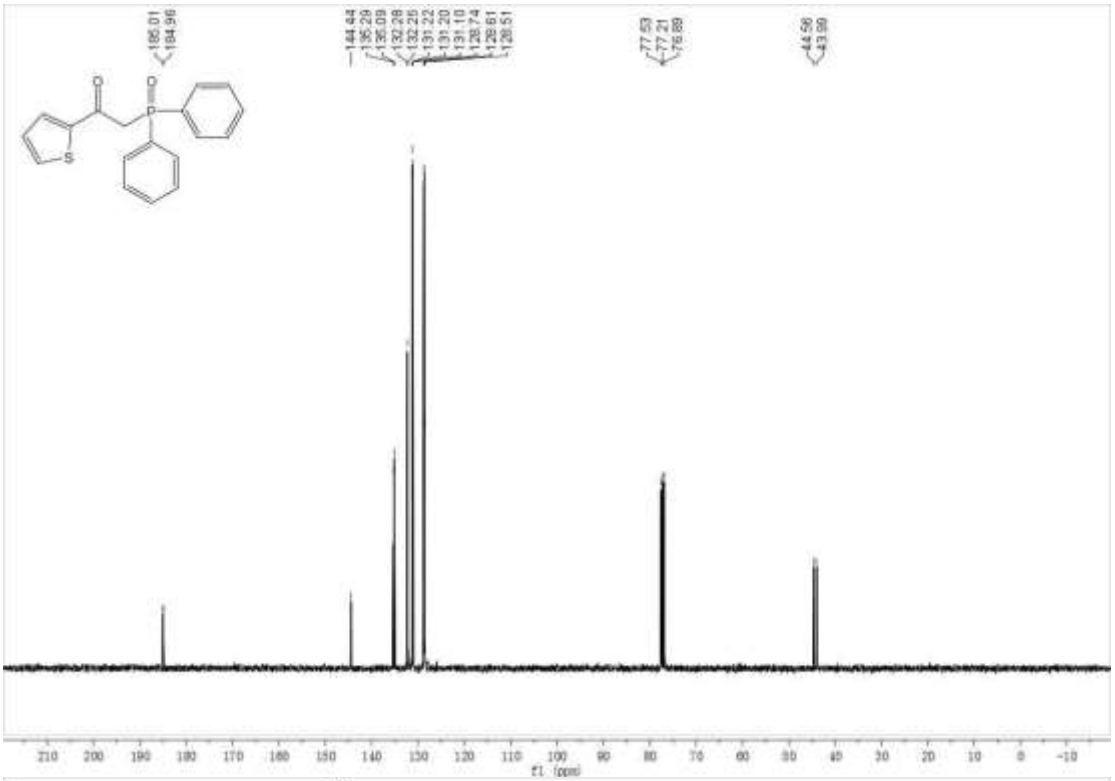

26.62
f1 (ppm)

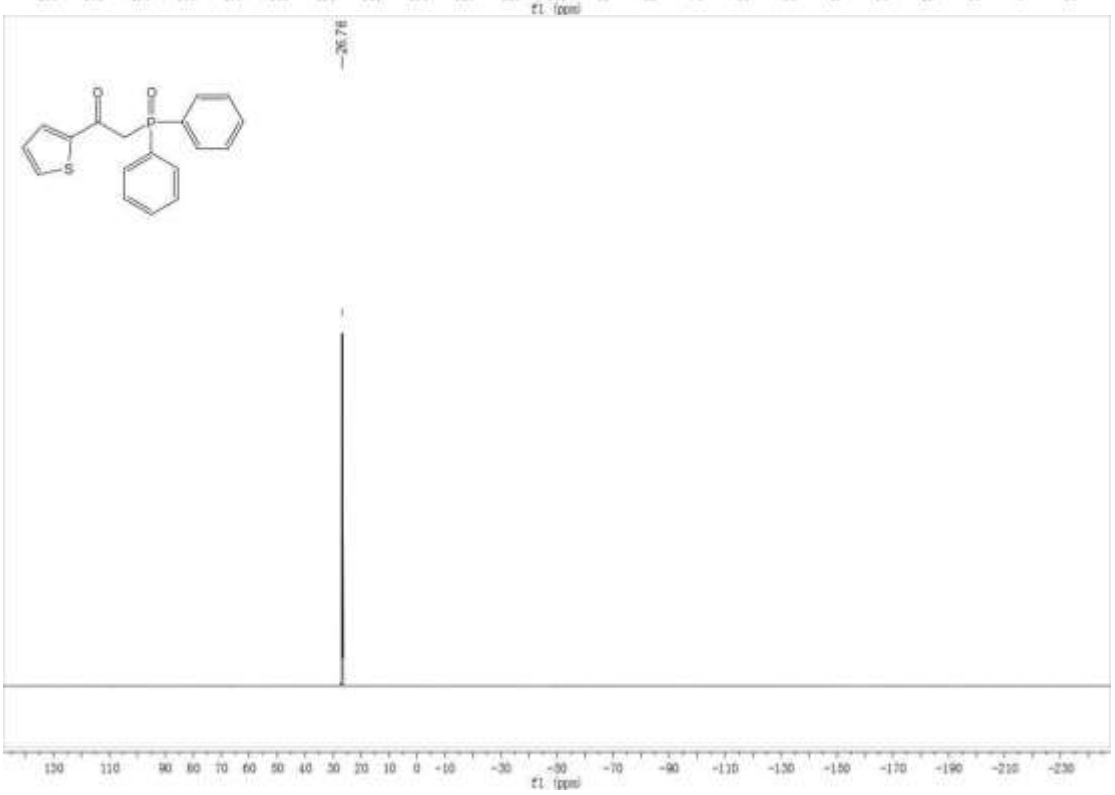


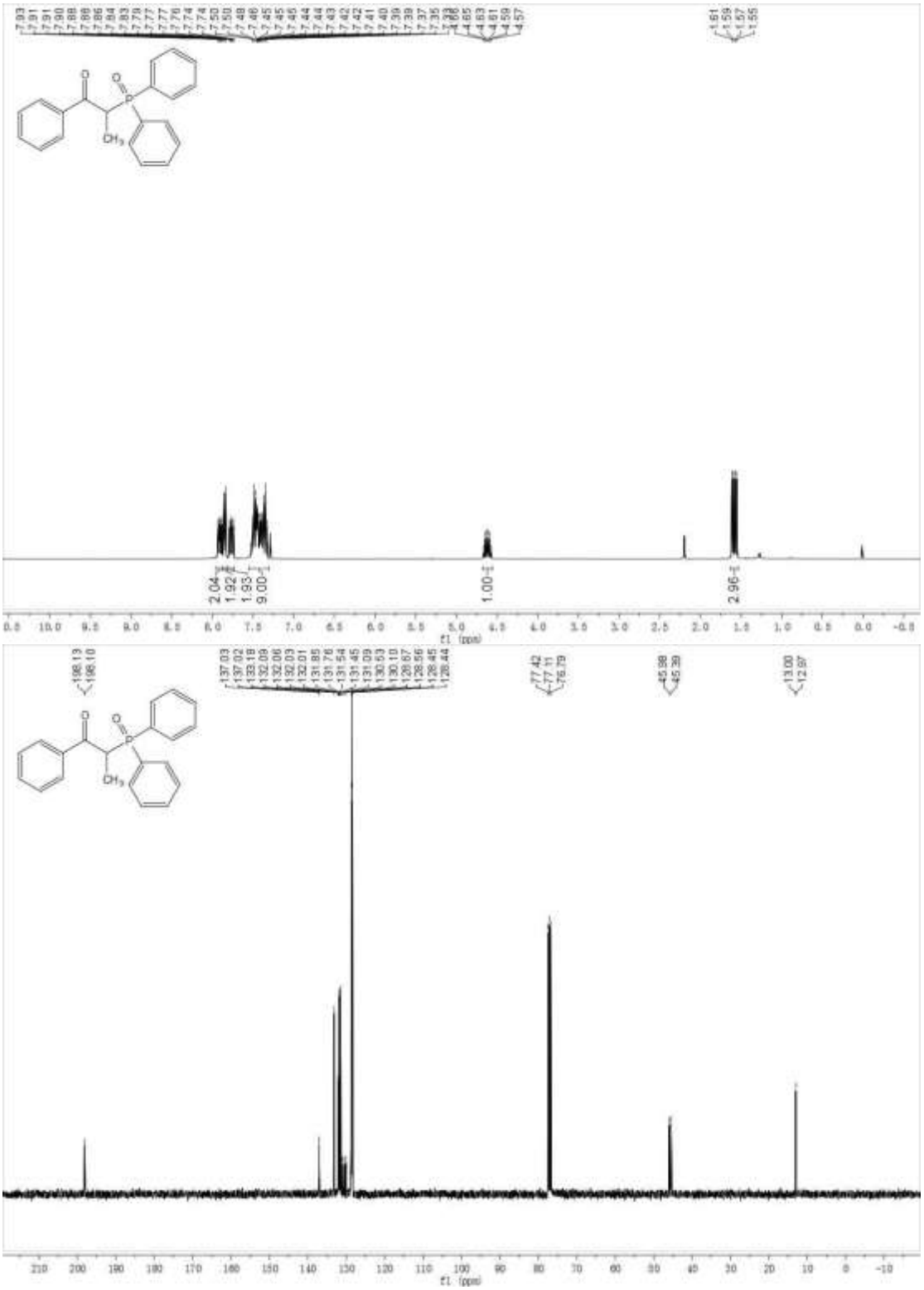


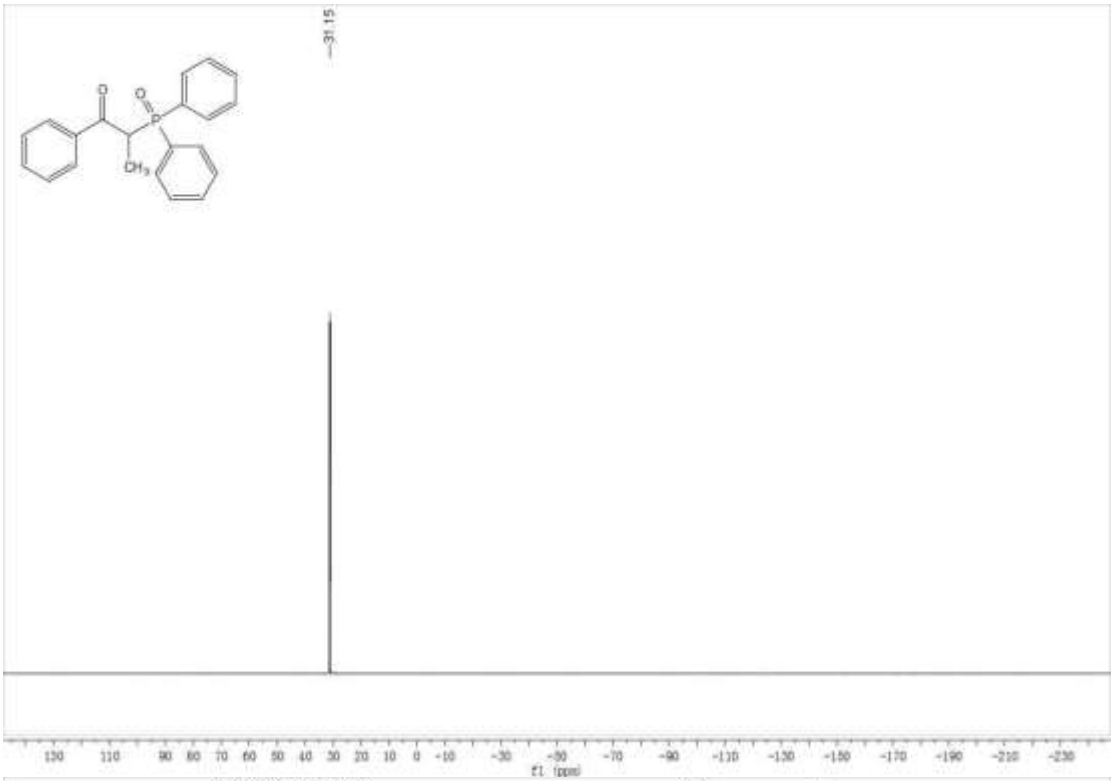

26.92
f1 (ppm)

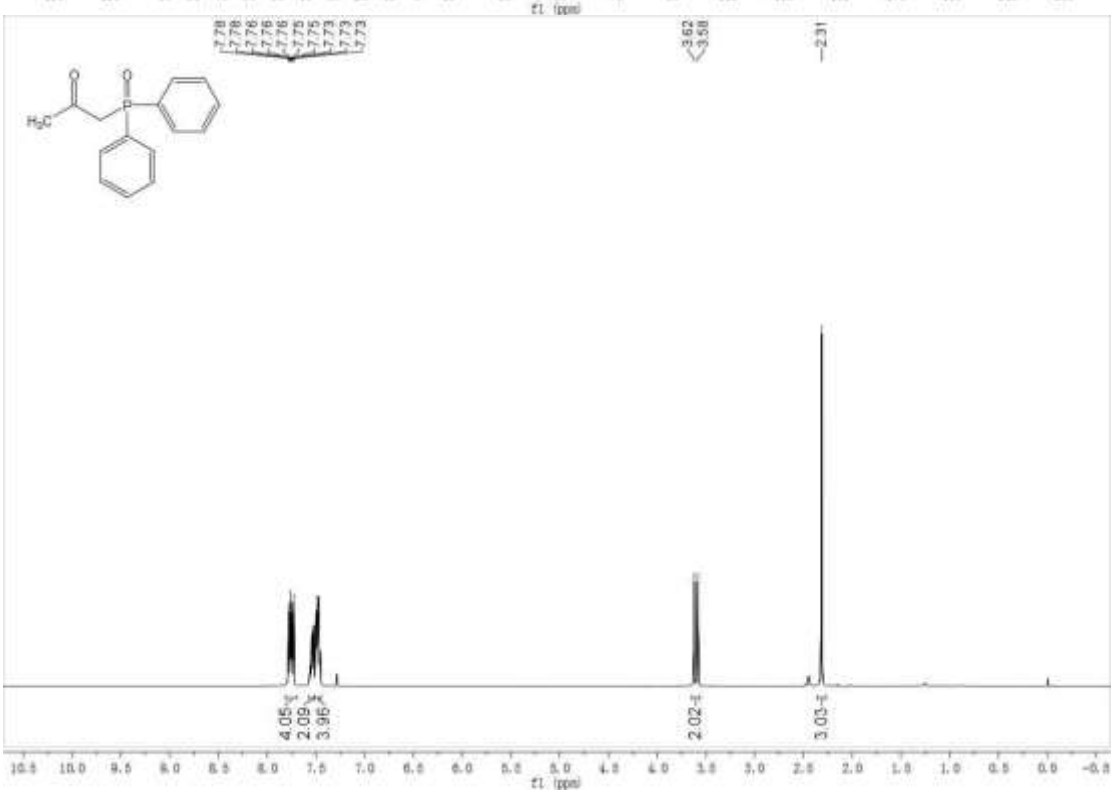

7.90
7.89
7.89
7.85
7.83
7.82
7.80
7.80
7.65
7.63
7.54
7.53
7.52
7.50
7.49
7.48
7.48
7.47
7.10
7.10
7.09
4.09
4.05
1.04
3.89
1.00
2.03
3.91
0.96
2.00
f1 (ppm)

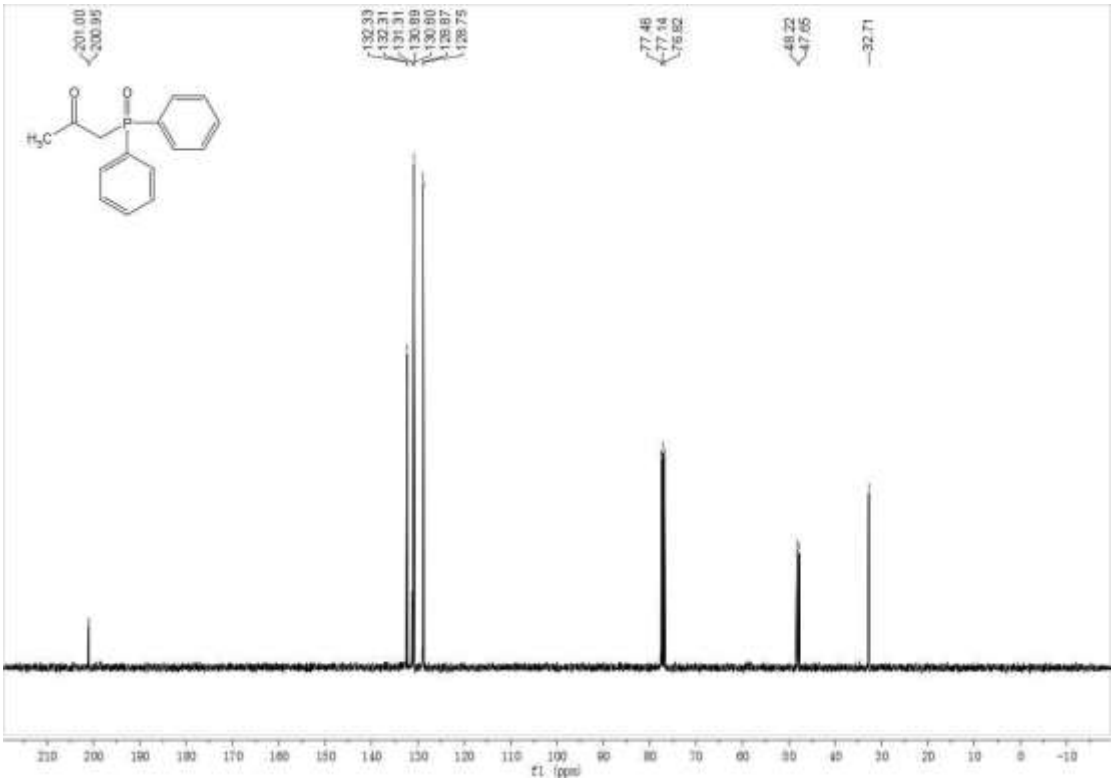

185.01
184.96
144.44
135.29
135.09
132.28
132.25
131.22
131.20
131.10
128.74
128.61
128.51
77.53
77.21
76.89
44.56
43.99
f1 (ppm)

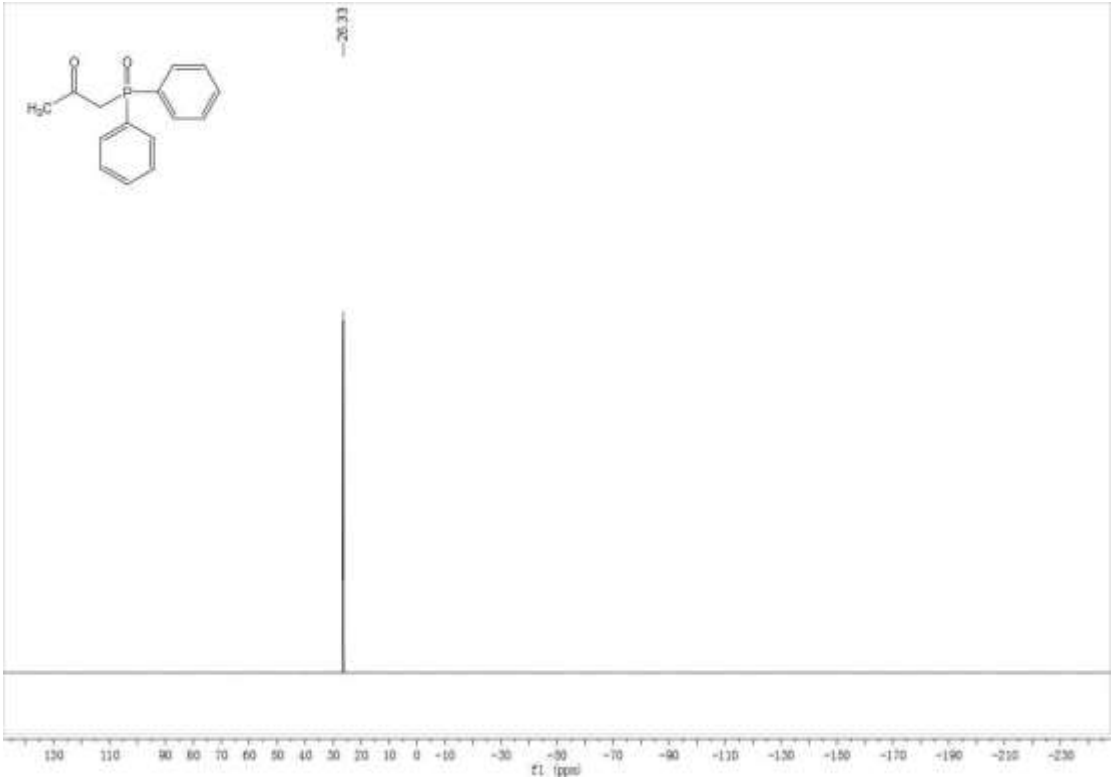

26.78
f1 (ppm)

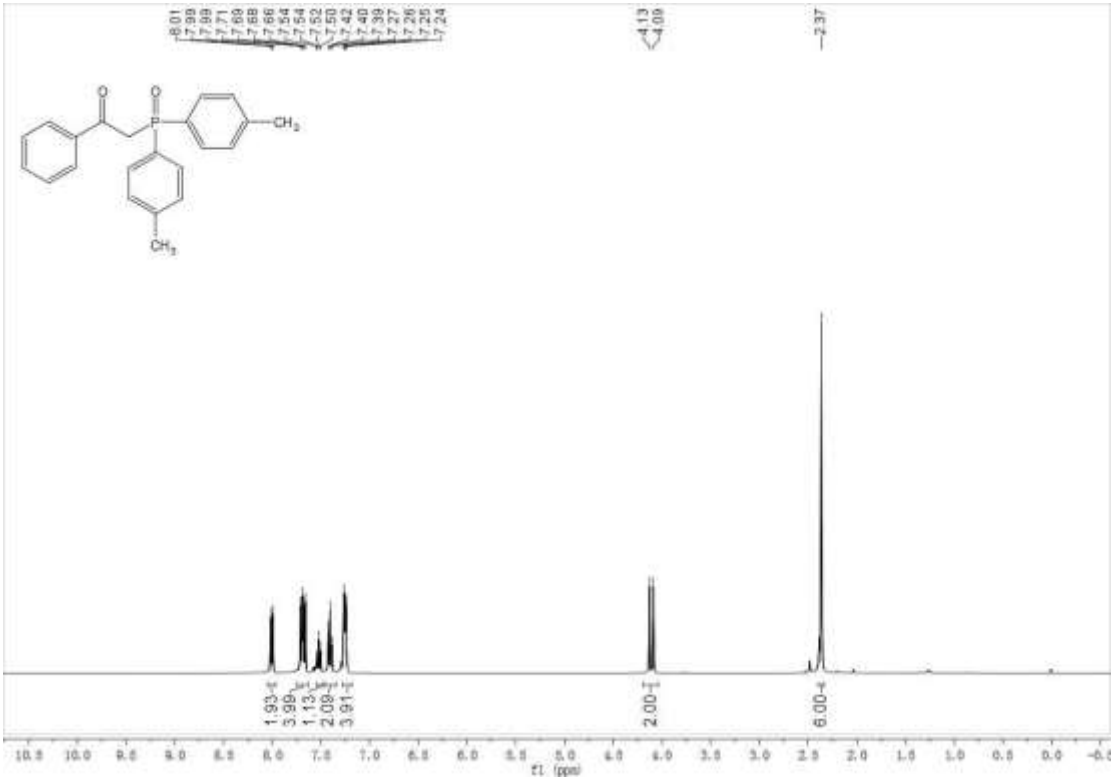


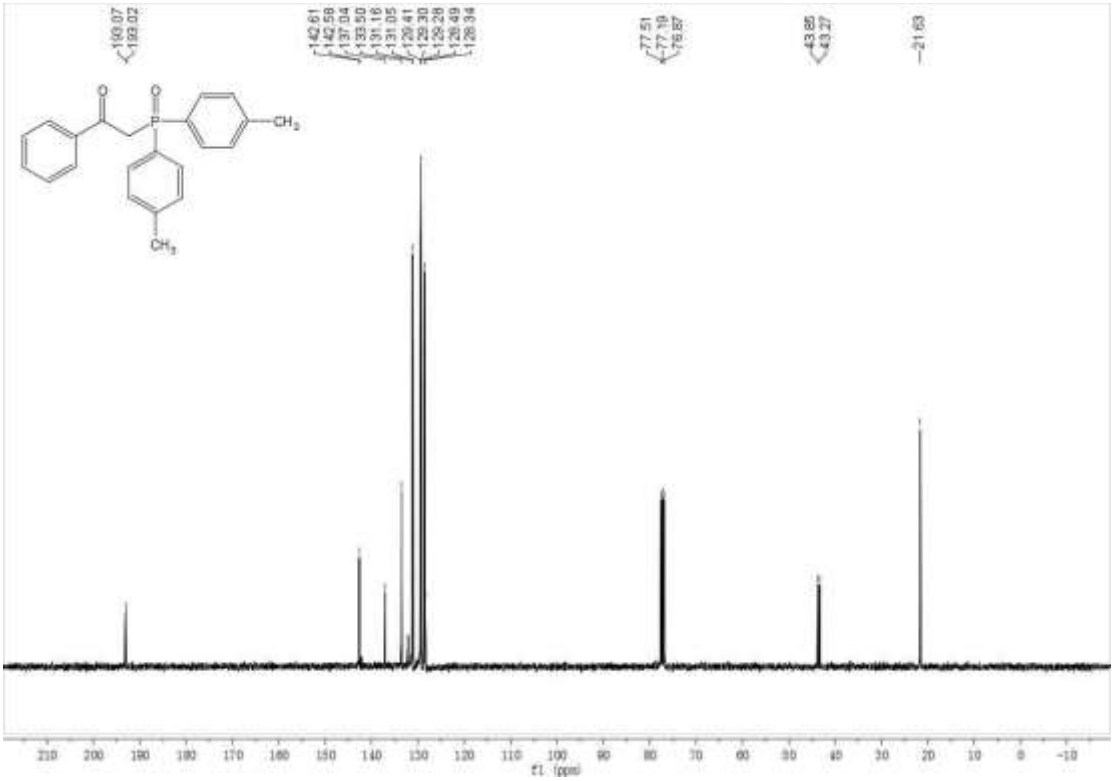

31.15
f1 (ppm)

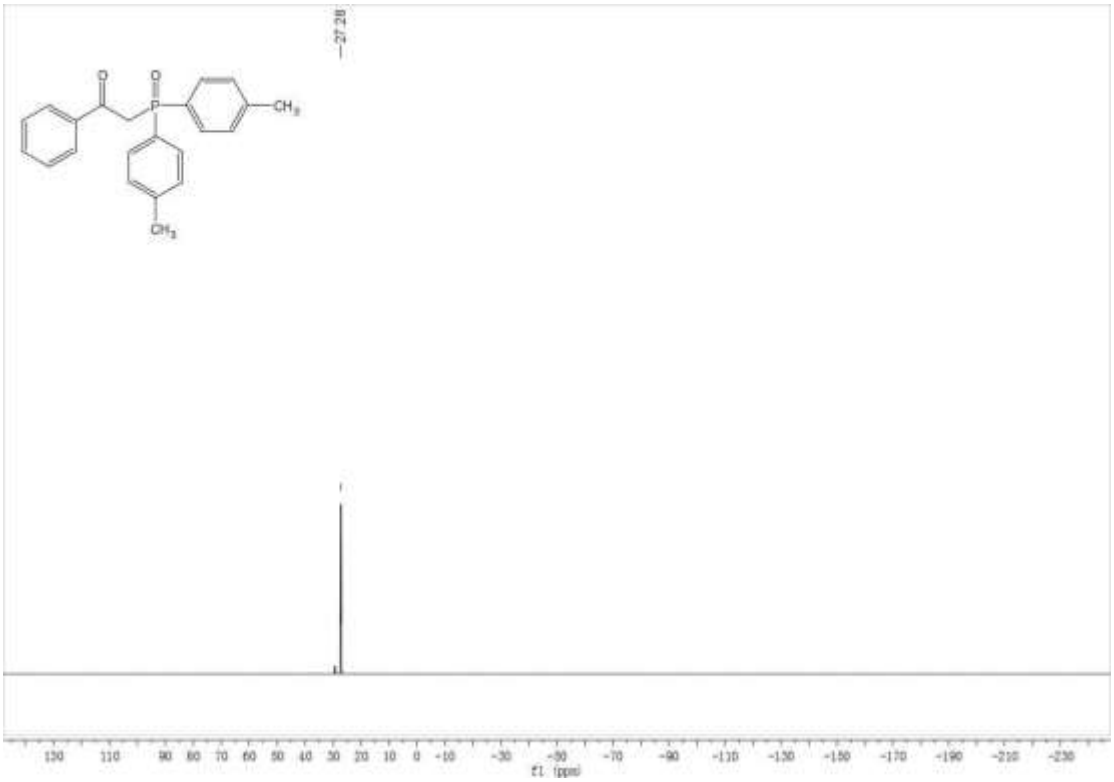

7.78
7.76
7.76
7.76
7.76
7.75
7.75
7.73
7.73
7.73
3.62
3.58
2.31
4.05
2.09
3.96
2.02
3.03
f1 (ppm)

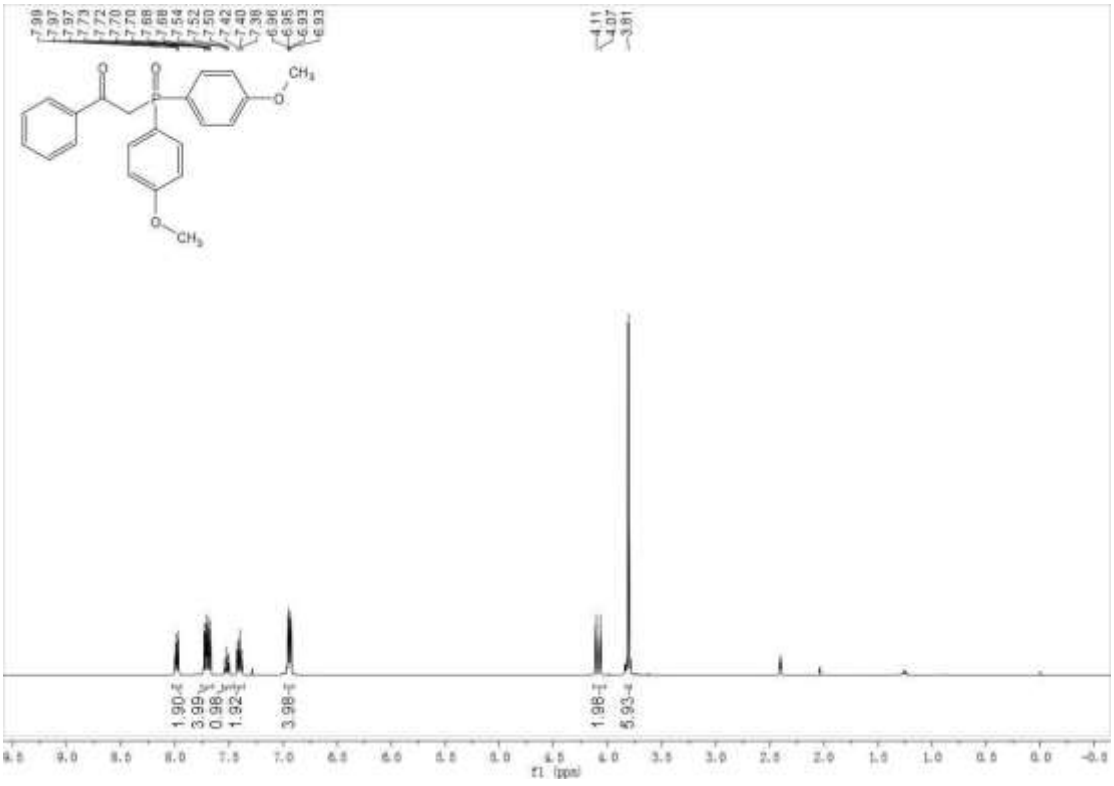

201.00
200.95
132.33
132.31
131.31
130.89
130.80
128.87
128.75
77.46
77.14
76.82
48.22
47.65
32.71
f1 (ppm)

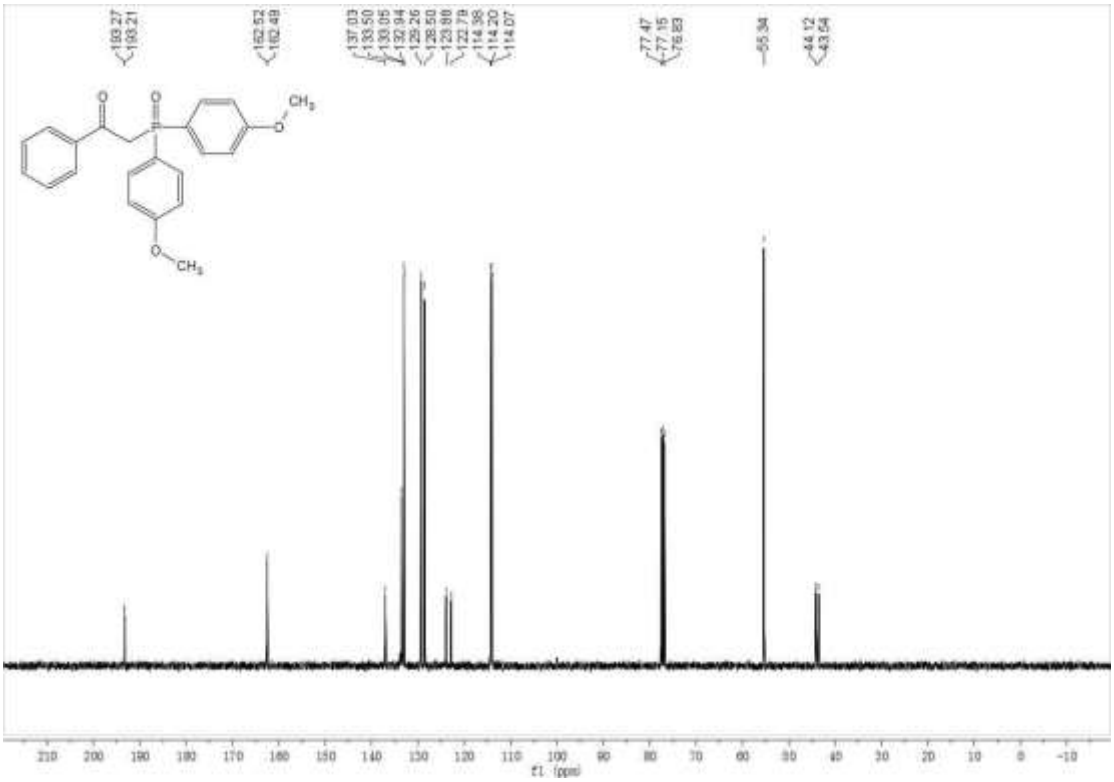

26.33
f1 (ppm)

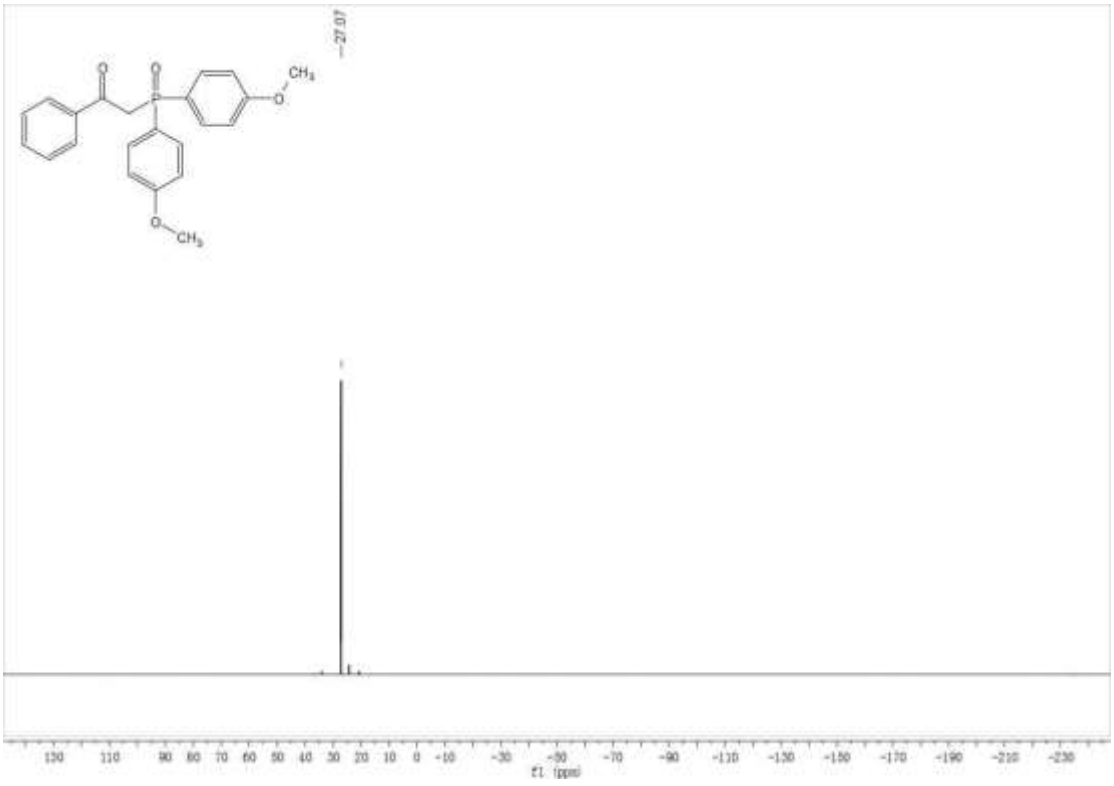

8.01
7.99
7.99
7.71
7.69
7.68
7.66
7.54
7.54
7.52
7.50
7.42
7.40
7.39
7.27
7.26
7.25
7.24
4.13
4.09
2.37
1.93
3.99
1.13
2.09
3.91
2.00
6.00
f1 (ppm)

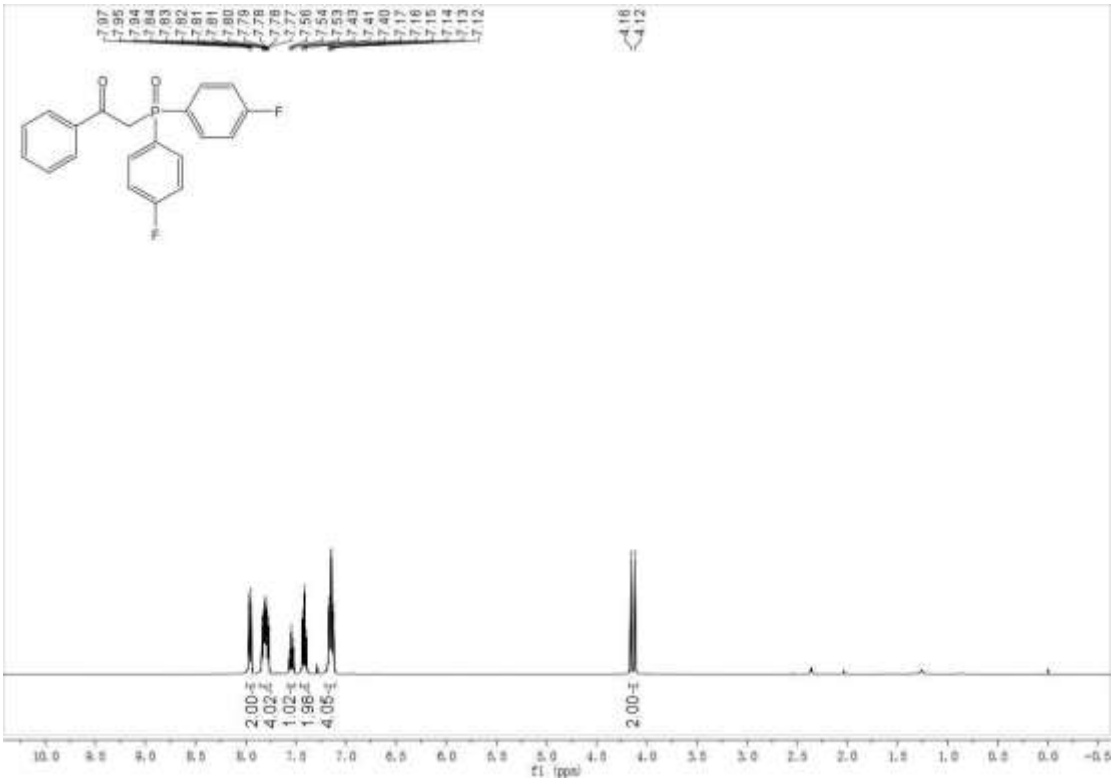

190.07
189.02
142.61
142.58
137.04
133.50
131.16
131.05
129.41
129.30
129.28
128.49
128.34
77.51
77.19
76.87
43.85
43.27
21.63
f1 (ppm)

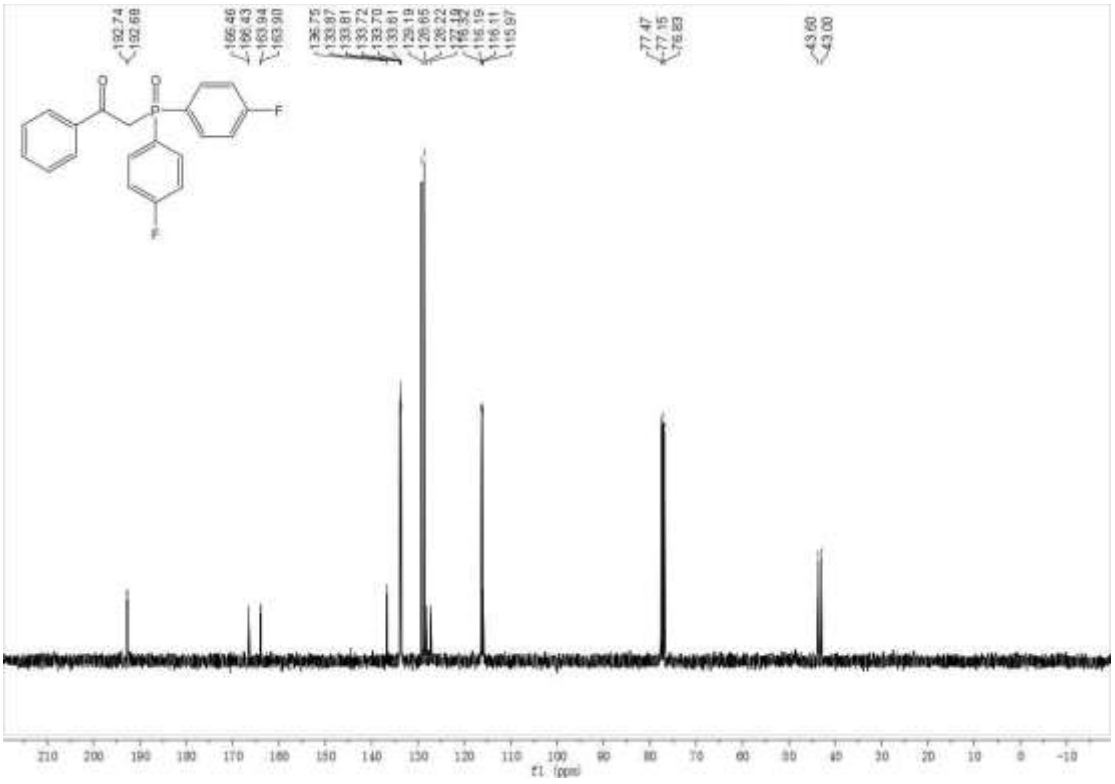

27.28
f1 (ppm)

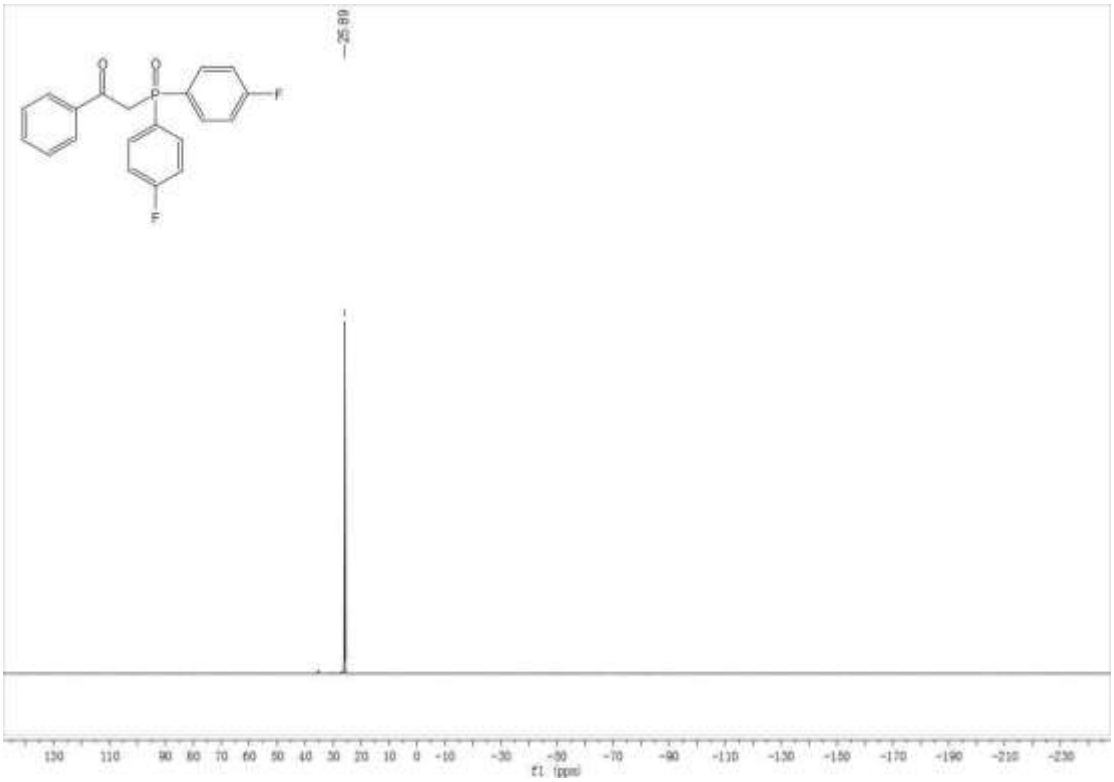

7.99
7.97
7.97
7.73
7.72
7.70
7.70
7.68
7.68
7.54
7.52
7.50
7.42
7.40
7.38
6.96
6.95
6.93
6.93
4.11
4.07
3.81
1.90
3.99
0.98
1.92
3.98
1.98
5.93
f1 (ppm)

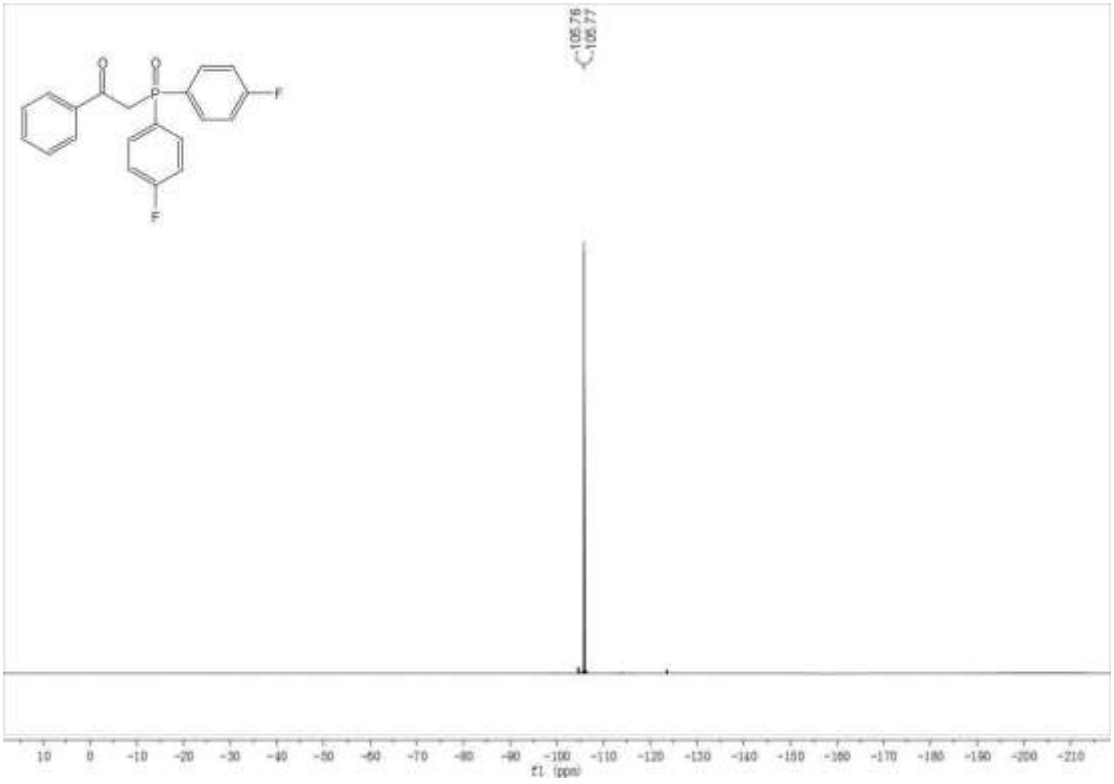

193.27
193.21
162.52
162.49
137.03
133.50
130.05
130.94
128.26
125.50
123.86
122.79
114.98
114.20
114.07
77.47
77.15
76.83
55.34
44.12
43.54
O
P
O
CH3
O
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

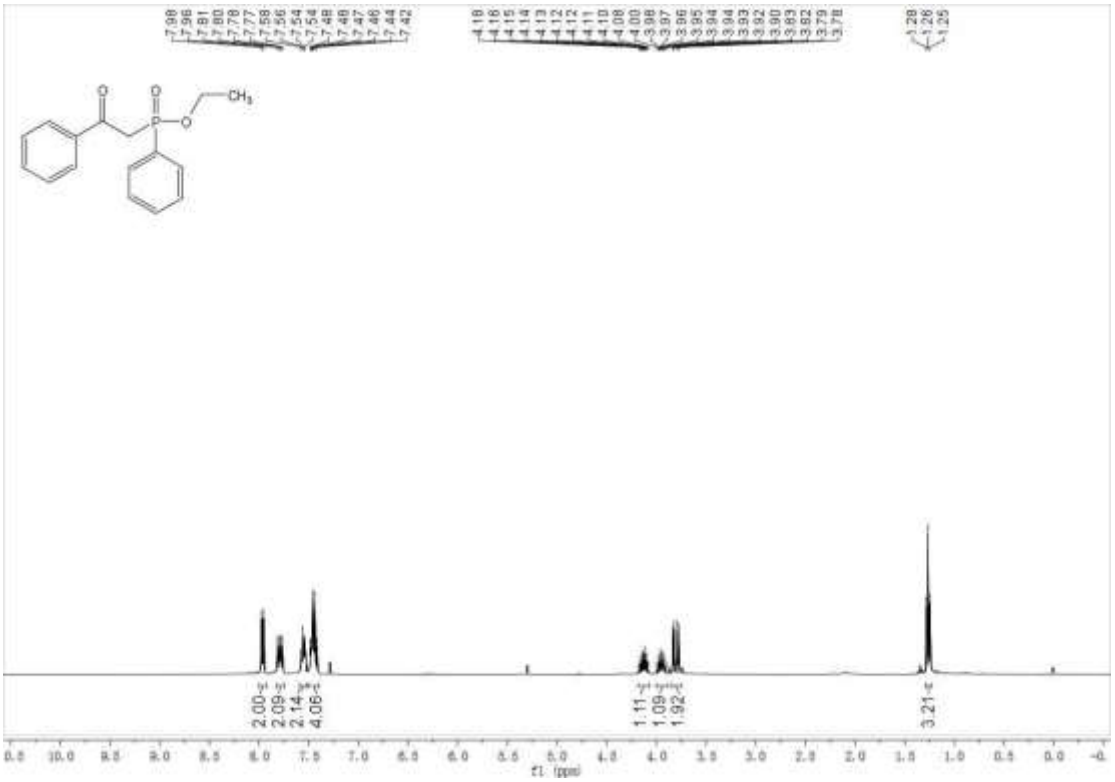

27.07
O
P
O
CH3
O
CH3
150 110 90 80 70 60 50 40 30 20 10 0 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

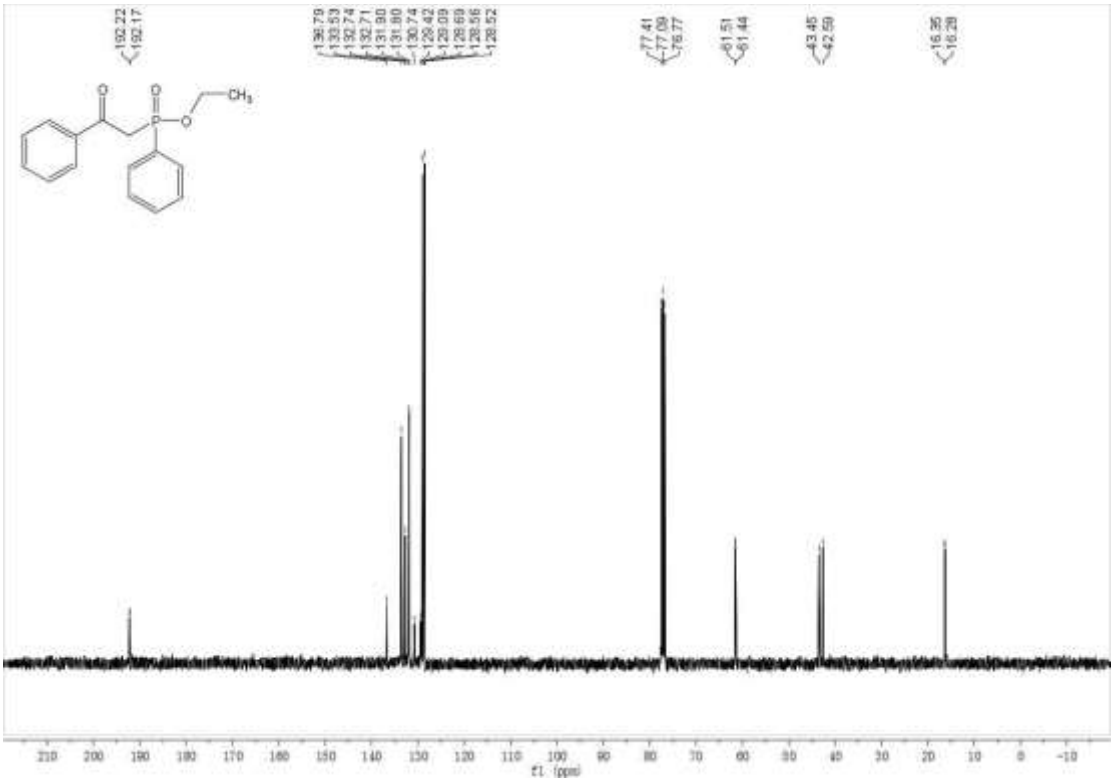

7.97
7.95
7.94
7.84
7.83
7.82
7.81
7.80
7.79
7.78
7.78
7.77
7.56
7.54
7.53
7.43
7.41
7.40
7.17
7.16
7.15
7.14
7.13
7.12
4.16
4.12
2.00
4.02
1.02
1.96
4.05
2.00
f1 (ppm)

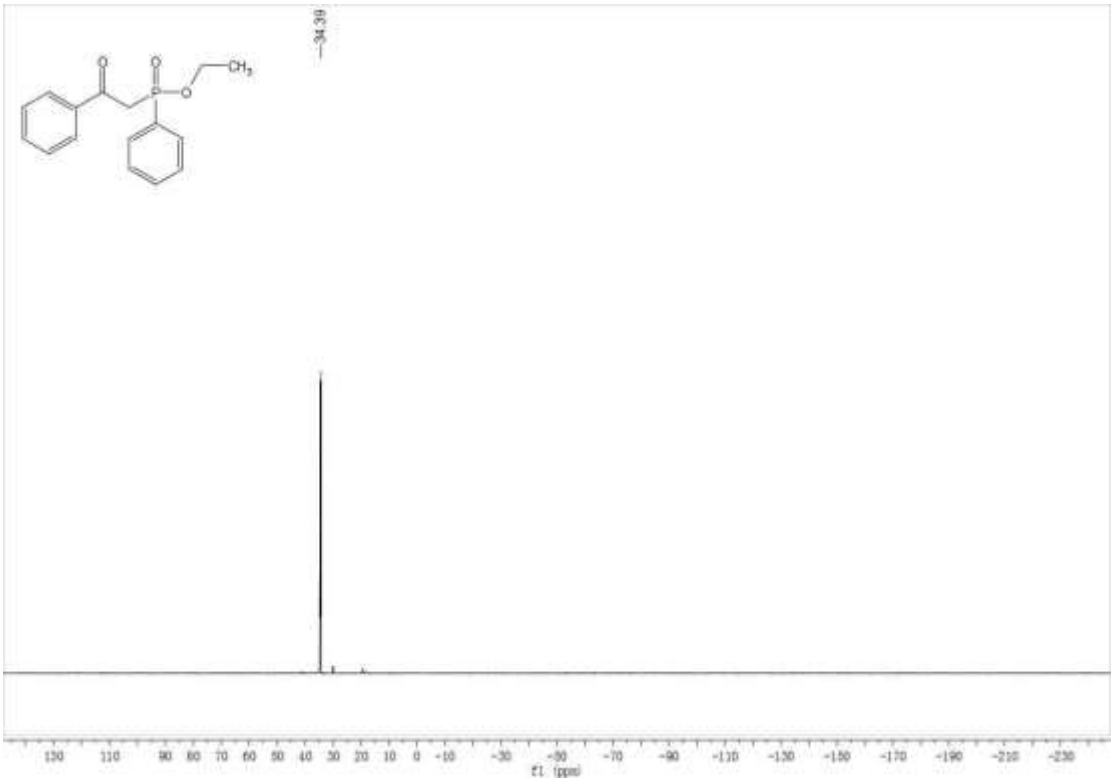

192.74
192.69
166.46
166.43
163.94
163.90
136.75
133.87
133.81
133.72
133.70
133.61
129.19
128.65
128.22
116.32
116.19
116.11
115.97
77.47
77.15
76.83
43.60
43.00
f1 (ppm)

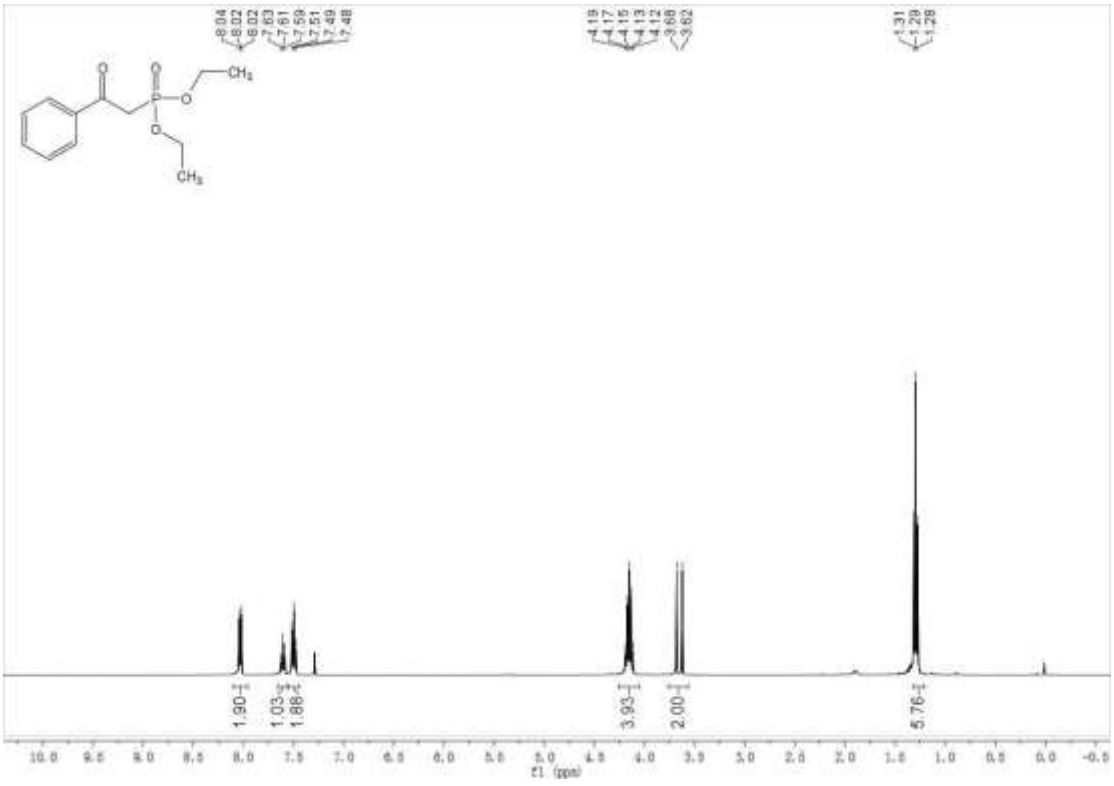

-25.89
f1 (ppm)

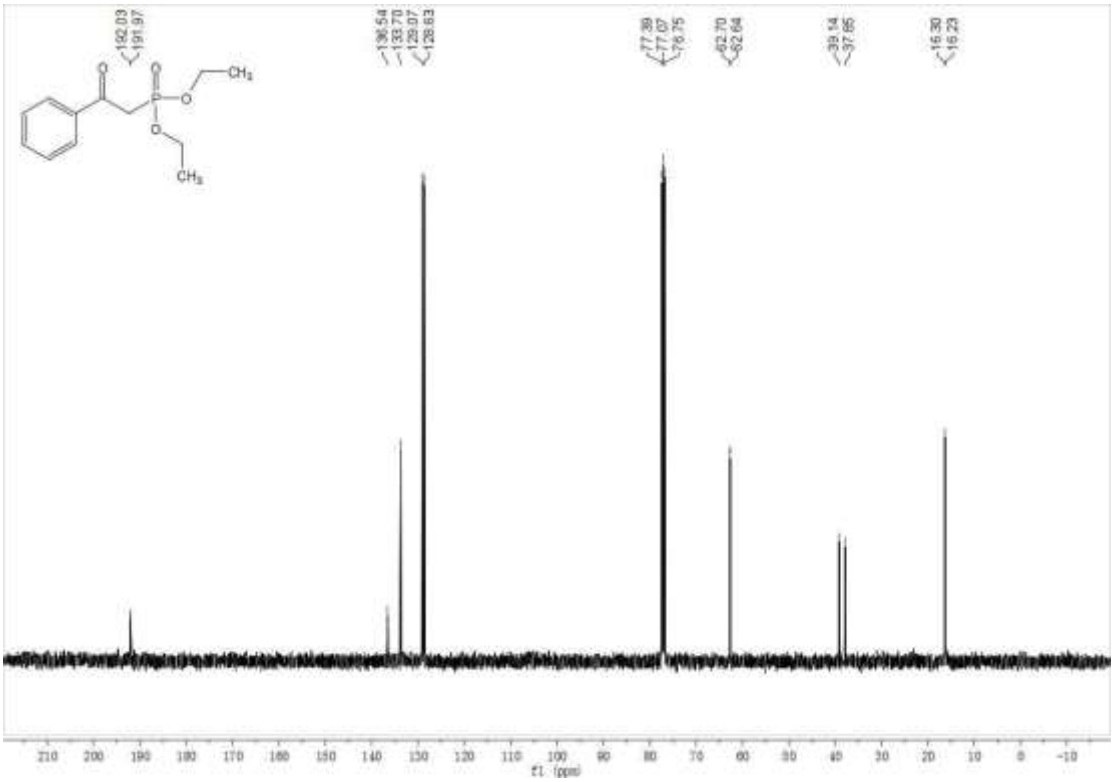

-105.76
-105.77
f1 (ppm)

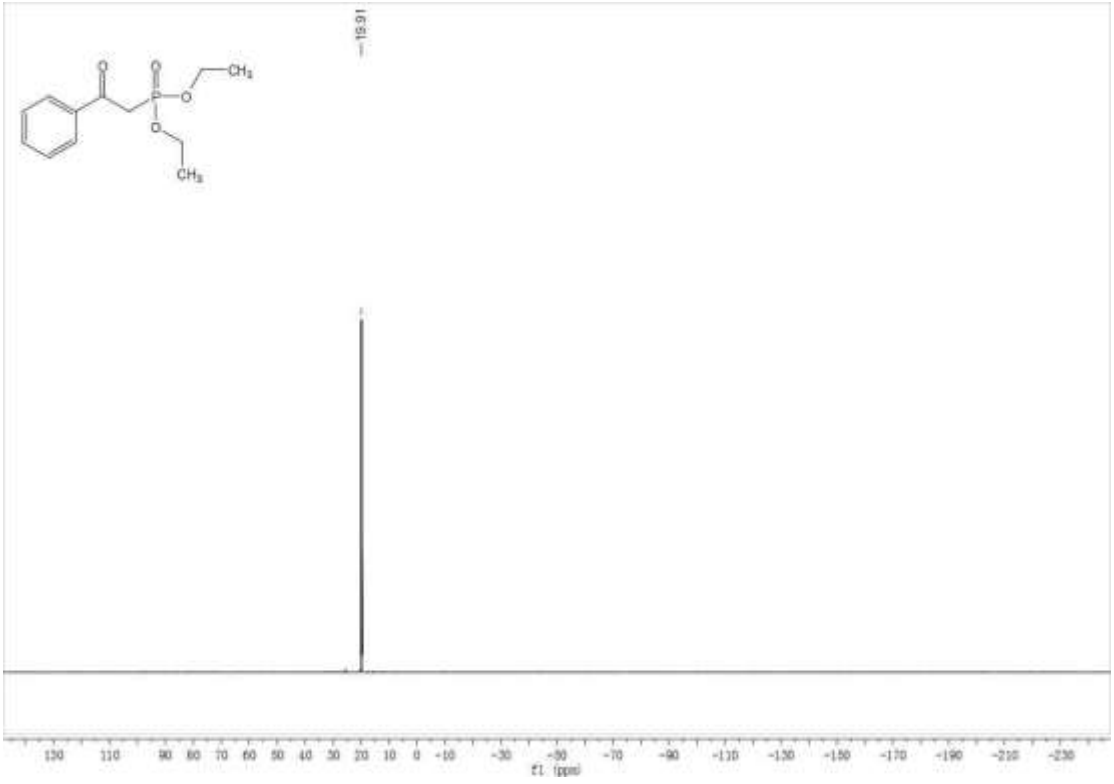


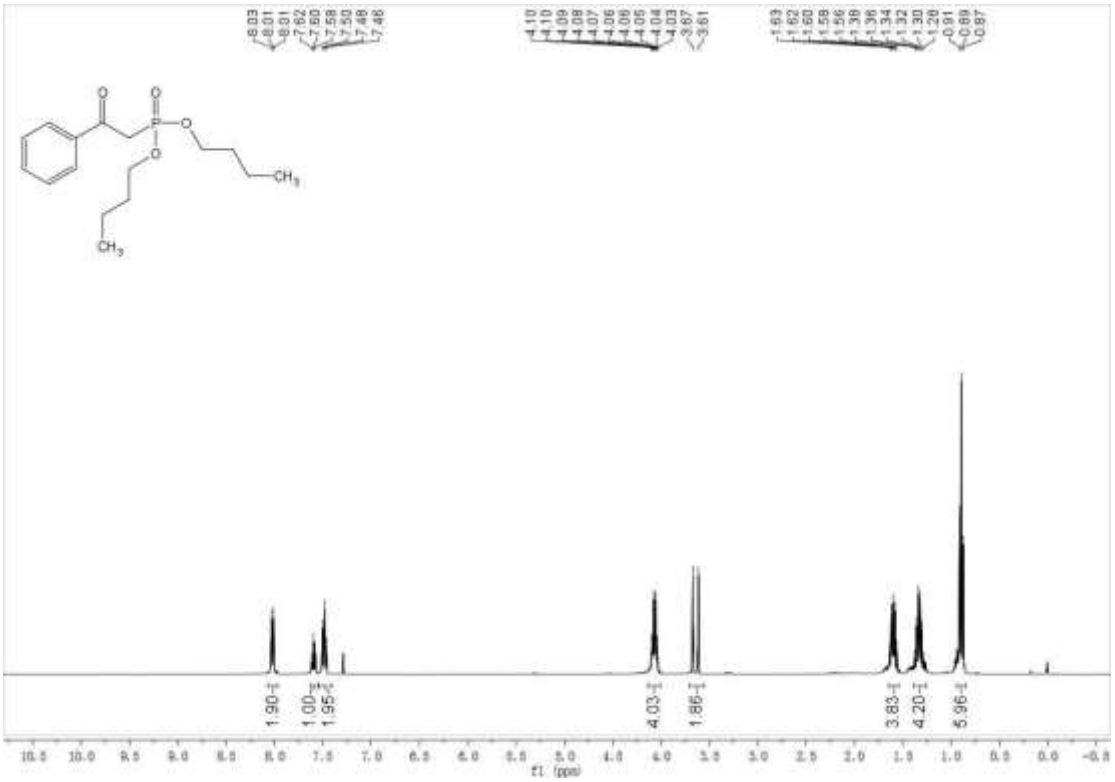


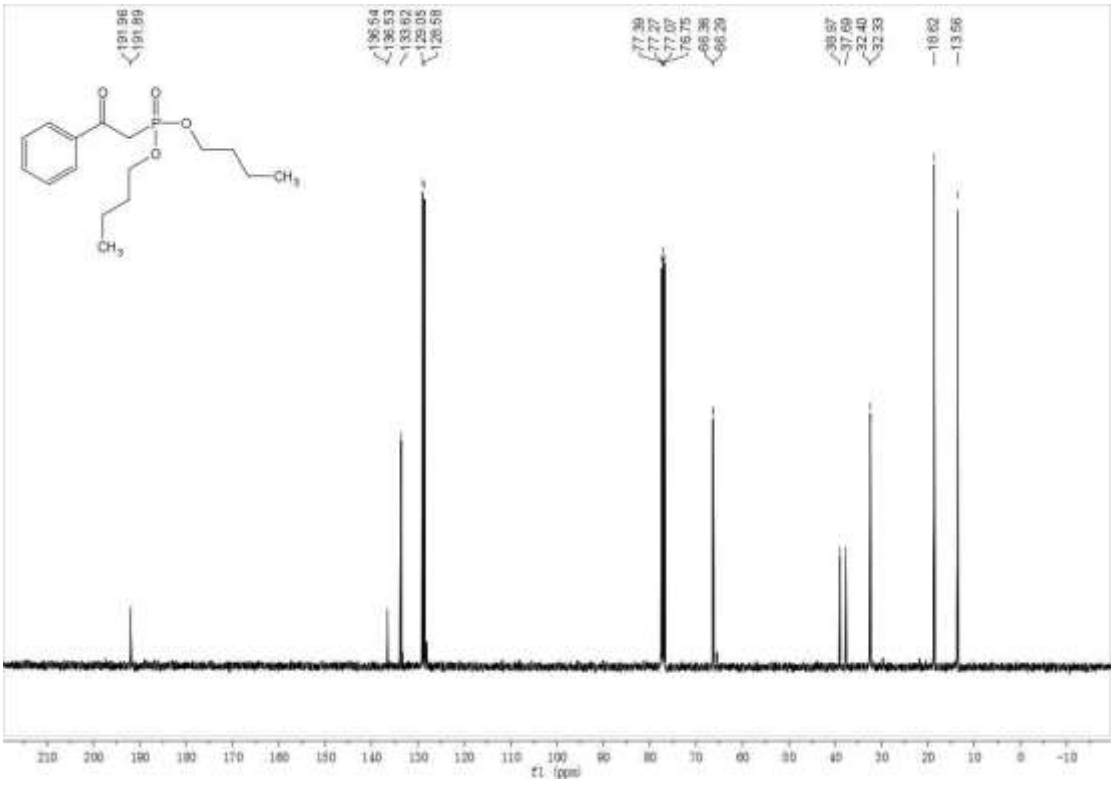

34.39
f1 (ppm)

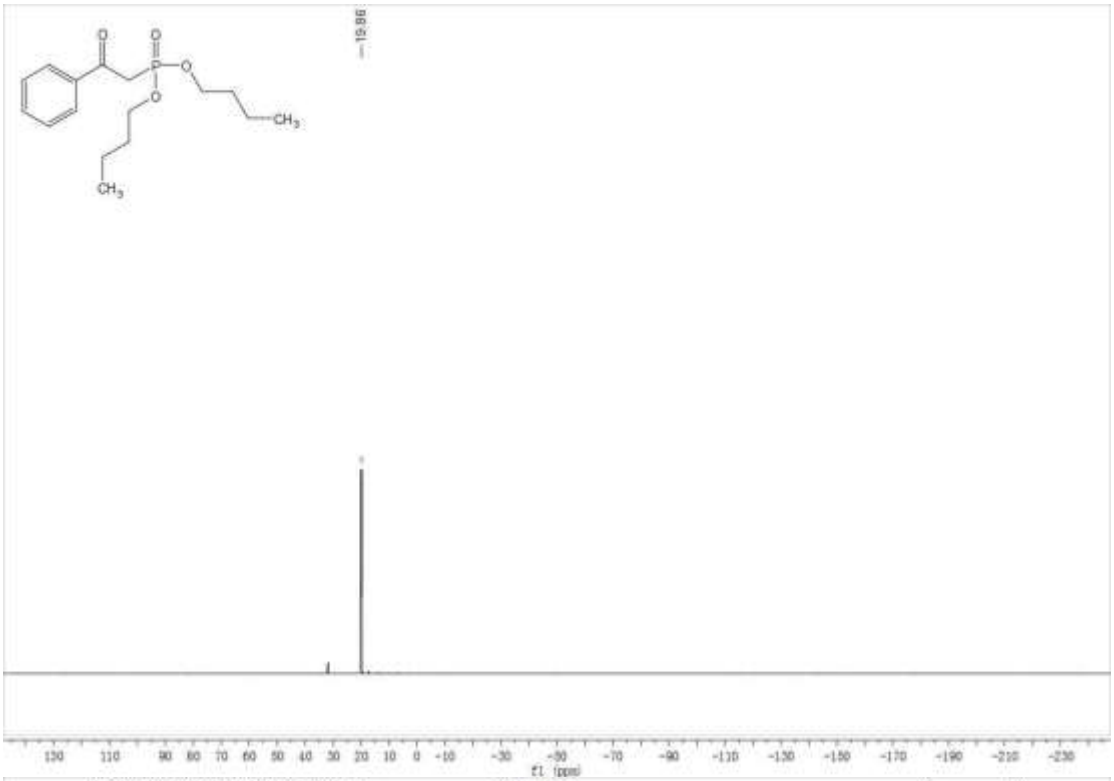

8.04
8.02
8.02
7.63
7.61
7.59
7.51
7.49
7.48
4.19
4.17
4.15
4.13
4.12
3.68
3.62
1.31
1.29
1.28
1.90
1.03
1.88
3.93
2.00
5.76
f1 (ppm)

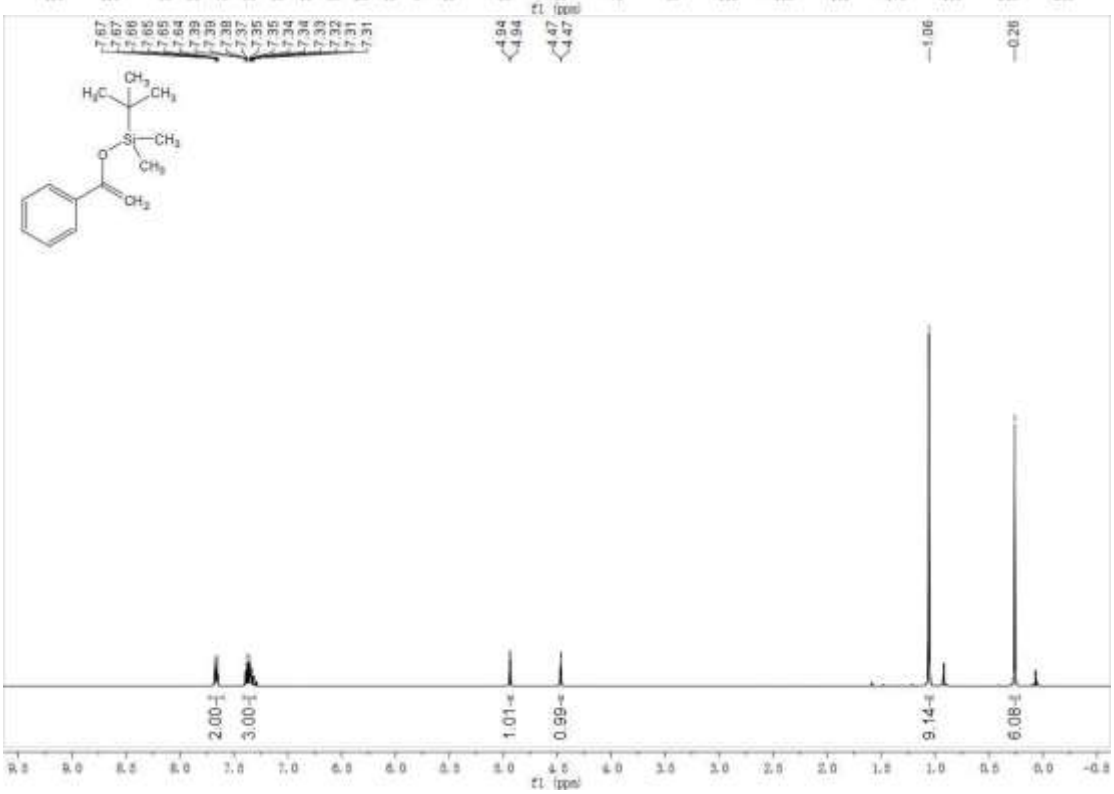

192.03
191.97
136.54
133.70
129.07
128.63
77.39
77.07
76.75
62.70
62.64
39.14
37.86
16.30
16.23
f1 (ppm)

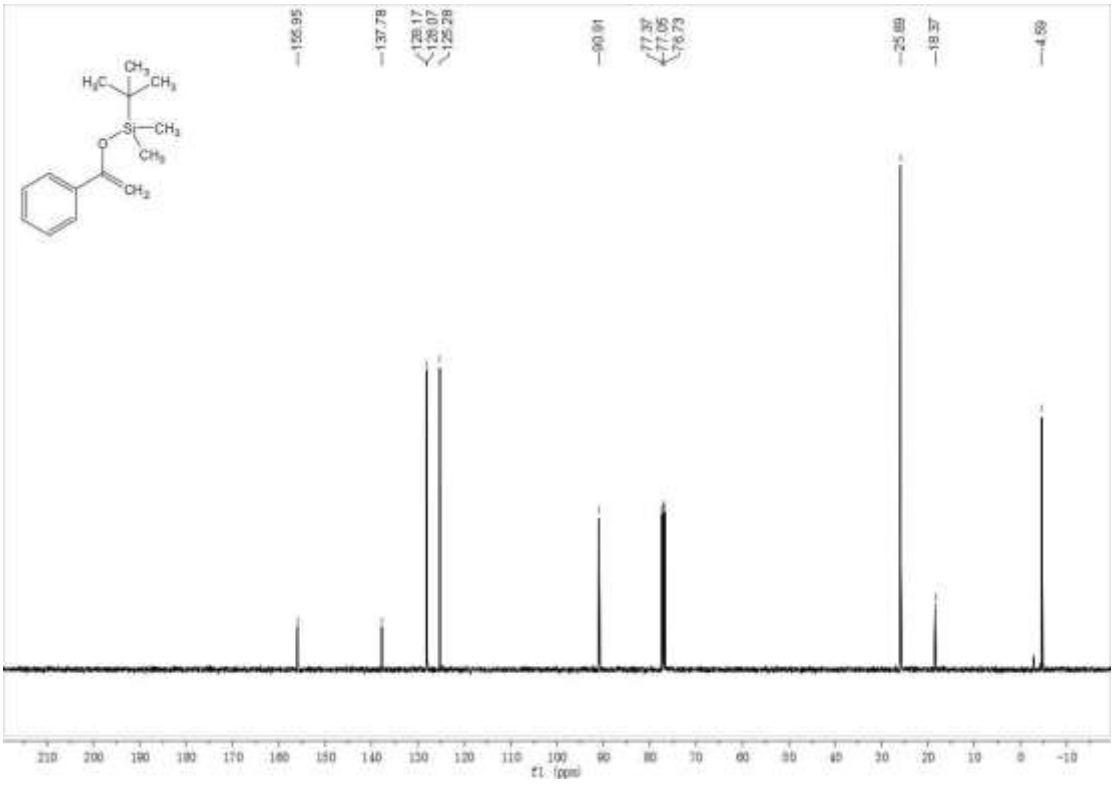

18.91
f1 (ppm)


8.03
8.01
8.01
7.62
7.60
7.58
7.58
7.48
7.46
4.10
4.10
4.09
4.09
4.07
4.06
4.06
4.05
4.04
4.03
3.67
3.61
1.63
1.62
1.60
1.58
1.56
1.39
1.36
1.34
1.32
1.30
1.28
0.91
0.89
0.87
1.90
1.00
1.95
4.03
1.86
3.83
4.20
5.96
f1 (ppm)


191.96
191.89
136.64
136.53
133.62
129.05
128.58
77.39
77.27
77.07
76.75
66.36
66.29
39.57
37.69
32.40
32.33
18.62
13.56
f1 (ppm)


19.86
f1 (ppm)


7.67
7.67
7.66
7.65
7.65
7.64
7.39
7.39
7.38
7.37
7.35
7.35
7.34
7.34
7.33
7.32
7.31
7.31
4.94
4.94
4.47
4.47
1.06
0.26
2.00
3.00
1.01
0.99
9.14
6.08
f1 (ppm)


156.95
137.78
128.17
126.07
125.28
90.91
77.37
77.05
76.73
25.89
18.37
-4.59
f1 (ppm)