Electronic Supplementary Material (ESI) for Chemical Communications.

Visible-light-induced ligand-free $RuCl_3$ catalyzed C-H phosphorylation in water

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1. General Methods

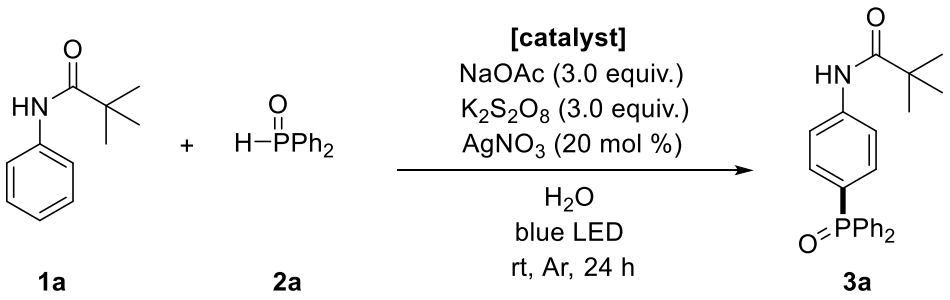
General Procedures. Unless otherwise noted, reactions were performed under argon atmosphere. Solvent was freshly distilled/degassed prior to use unless otherwise noted. Analytical TLC was performed with silica gel GF254 plates. For column chromatography, a 200-300 mesh silica gel was employed. Organic solutions were concentrated under reduced pressure using a rotary evaporator. Room temperature (rt) is 18-25°C.

Materials. Substrate **2y**-**2ac**[1] were synthesized according to previously described methods. Commercial reagents were purchased from Adamas, Ark, Aladdin, or TCI and used as received with the following exceptions. 1,4-Dioxane and toluene were dried with CaH_2 and freshly distilled, DCE was dried with P_2O_5 and freshly distilled. Other commercially available reagents and solvents were used without further purification.

Instrumentation. Deuterated solvents were purchased from Cambridge Isotope Laboratories. ^{1}H NMR spectra were recorded on Bruker AVANCE III 400 and INOVA instruments with 400 frequencies, and ^{13}C NMR spectra were recorded on Bruker AVANCE III 400 with 101 MHz frequencies. ^{31}P NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer with a ^{31}P operating frequency of 162 MHz. ^{19}F NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer with a ^{19}F operating frequency of 376 MHz. Chemical shifts (δ) were reported in ppm relative to the residual solvent signal (CDCl3 $\delta = 7.26$ for ^{1}H NMR and $\delta = 77.0$ for ^{13}C NMR). Chemical shifts (ppm) were recorded with tetramethyl silane (TMS) as the internal reference standard. Multiplicities are given as s (singlet), d (doublet), t (triplet), dd (doublet of doublets), td (triplet of doublets) or m (multiplet). HRMS was obtained using a Q-TOF instrument equipped with an ESI source. Data collection for crystal structure was performed at room temperature using Mo Kα radiation on a Bruker APEXII diffractometer.

2. Optimization Studies

Table S1. Screening of catalyst[a]

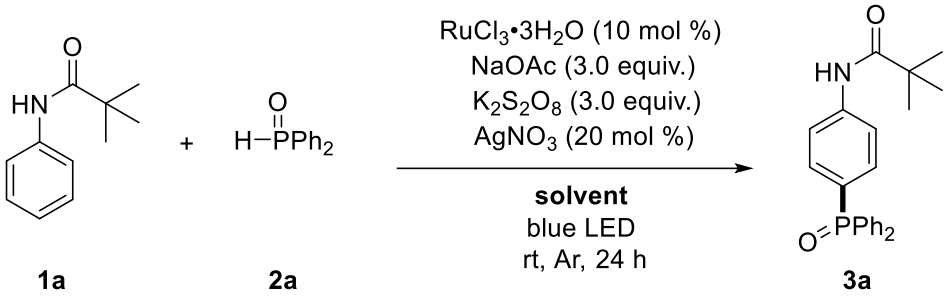


1a **2a** **3a**

entry	[catalyst]	yield (%)[b]
1	$[RuCl_2(p\text{-cymene})]_2$	14
2	$[Ru(O_2CMes)_2(p\text{-cymene})]$	<5
3	$Ru(pph_3)_3Cl_2$	13
4	$Ru_3(CO)_{12}$	<5
5	**$RuCl_3 \cdot 3H_2O$**	**58**
6	$AlCl_3$	14
7	$FeCl_3$	10
8	$FeCl_2$	16
9	$CoCl_2$	13
10	$BF_3 \cdot Et_2O$	12
11	$Zn(OTf)_2$	7
12	$Sc(OTf)_3$	12
13	$Bi(OTf)_3$	15
14	$Cu(OTf)_2$	8
15	$Ru(bpy)_3Cl_2$	0
16	RuI_3	23

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.4 mmol), **catalyst (10 mol %)**, NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O (1.0 mL), 24 h, Ar, blue LED at room temperature (18-25 ℃); [b] Yield of isolated products.

Table S2. Screening of solvent[a]



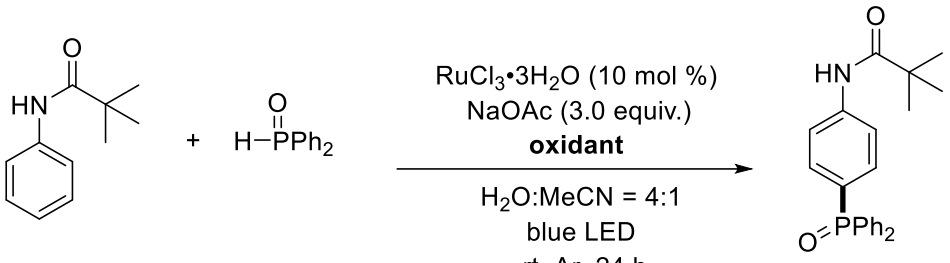
1a **2a** **3a**

entry	solvent	yield (%)[b]
1	H_2O	58
2	DCE	0
3	PhMe	0
4	dioxane	0

5	EtOH	0
6	TFE	0
7	MeCN	0
8	H_2O:MeCN = 1:1	61
9	H_2O:MeCN = 8:1	71
10	**H_2O:MeCN = 4:1**	**76**
11	H_2O:MeCN = 2:1	64
12	EtOH:MeCN = 4:1	0

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.4 mmol), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), **solvent (1.0 mL)**, 24 h, Ar, blue LED at room temperature (18-25 ℃); [b] Yield of isolated products.

Table S3. Screening of oxidant[a]

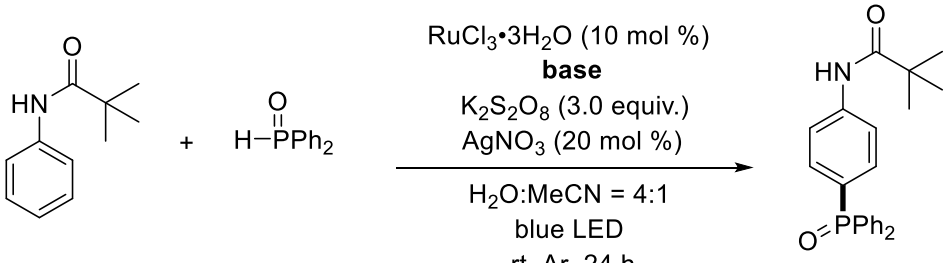


1a **2a** **3a**

entry	oxidant	yield (%)[b]
1	**$K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %)**	**76**
2	$K_2S_2O_8$ (3.0 equiv.)	23
3	$AgNO_3$ (20 mol %)	0
4	$K_2S_2O_8$ (3.0 equiv.), AgOAc (20 mol %)	65
5	TBHP, $AgNO_3$ (20 mol %)	<5
6	$Mn(OAc)_3 \cdot 2H_2O$, $AgNO_3$ (20 mol %)	15

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.4 mmol), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), **oxidant (3.0 equiv.)**, H_2O:MeCN = 4:1 (0.8 mL:0.2 mL), 24 h, Ar, blue LED at room temperature (18-25 ℃); [b] Yield of isolated products.

Table S4. Screening of base[a]



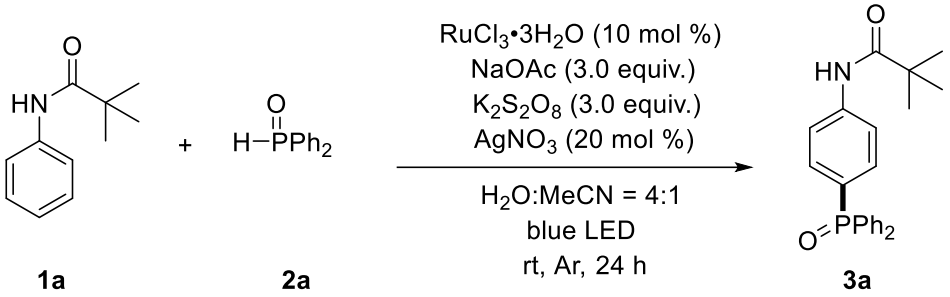
1a **2a** **3a**

entry	base	yield (%)[b]
1	Na_2CO_3	34
2	**NaOAc**	**76**
3	MesCOOK	54
4	CsF	43

5	PivOK	49

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.4 mmol), $RuCl_3 \cdot 3H_2O$ (10 mol %), **base (3.0 equiv.)**, $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O:MeCN = 4:1 (0.8 mL:0.2 mL), 24 h, Ar, blue LED at room temperature (18-25 ℃); [b] Yield of isolated products.

Table S5. Black experiments[a]



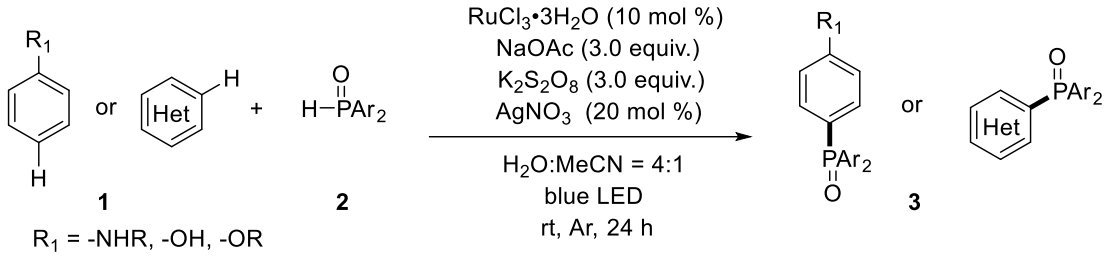
1a **2a** **3a**

entry	deviation from standard conditions	yield (%)[b]
1	no $RuCl_3 \cdot 3H_2O$	<5
2	In the dark	<5

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.4 mmol), $RuCl_3 \cdot 3H_2O$ (10 mol %), base (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O:MeCN = 4:1 (0.8 mL:0.2 mL), 24 h, Ar, blue LED at room temperature (18-25 ℃); [b] Yield of isolated products.

3. General Procedure

a) C-H phosphorylation of arenes and heteroarenes

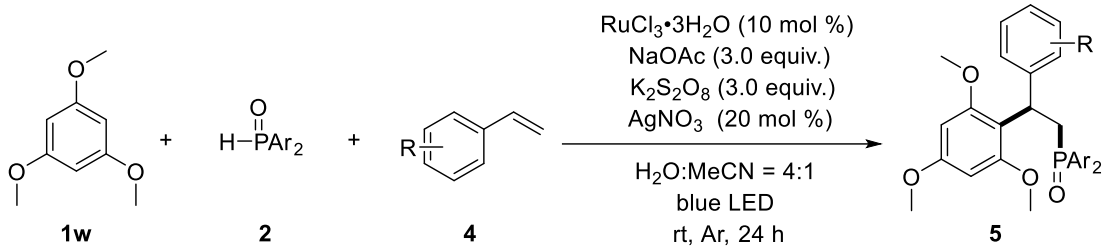


1 **2** **3**

R_1 = -NHR, -OH, -OR

In a 20 mL tube, a mixture of **1** (0.2 mmol, 1.0 equiv.), **2** (0.8 mmol, 4.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %) were added and charged with argon more than three times. H_2O (1.6 mL) was injected into the tube first, then MeCN (0.4 mL) was injected into the tube (the sequence of solvent injection is very important). The resulting black suspension was stirred vigorously at room temperature for 10 minutes before being irradiated in reactor with cooling device using a 440 (± 15) nm LED (25 W) for 24 hours. After the reaction was completed, the solvents were evaporated under reduced pressure, and the residue was purified with chromatography column on silica gel or preparative TLC (PTLC) (DCM/MeOH = 100:1 - 20:1).

b) Three-component series phosphination and aromatization reaction of olefins

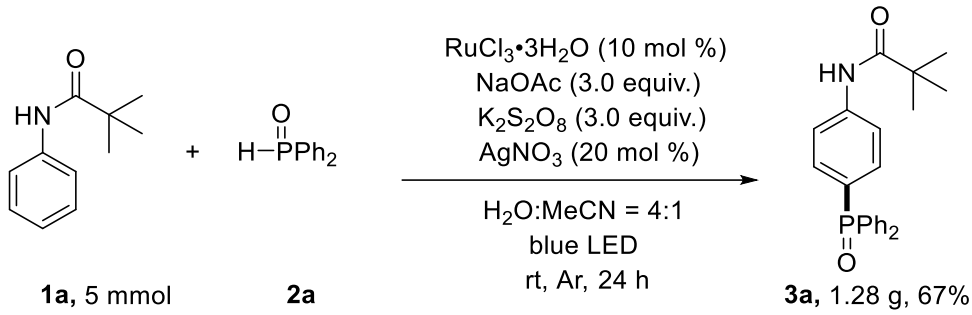


1w **2** **4** **5**

In a 20 mL tube, a mixture of **1w** (0.2 mmol, 1.0 equiv.), **2** (0.8 mmol, 4.0 equiv.), **4** (0.8 mmol, 4.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %) were added and charged with argon more than three times. H_2O (1.6 mL) was injected into the tube first, then MeCN (0.4 mL) was injected into the tube (the sequence of solvent injection is very important). The resulting black suspension was stirred vigorously at room temperature for 10 minutes before being irradiated in reactor with cooling device using a 440 (± 15) nm LED (25 W) for 24 hours. After the reaction was completed, the solvents were evaporated under reduced pressure, and the residue was purified with chromatography column on silica gel or preparative TLC (PTLC) (DCM/MeOH = 100:1 - 20:1).

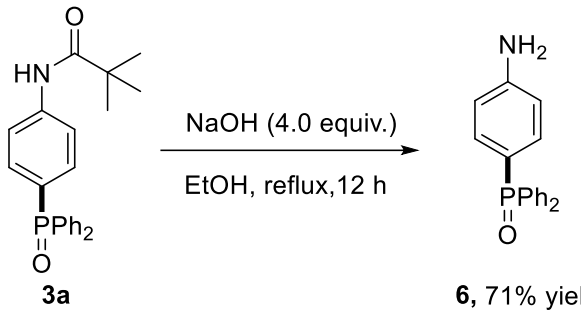
4. Derivatization of phosphonylated products

a) Gram-scale synthesis



In a 50 mL flask, a mixture of **1a** (5 mmol, 1.0 equiv.), **2a** (20 mmol, 4.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %) were added and charged with argon more than three times. H_2O (20.0 mL) was injected into the tube first, then MeCN (5.0 mL) was injected into the tube (the sequence of solvent injection is very important). The resulting black suspension was stirred vigorously at room temperature for 10 minutes before being irradiated in reactor with cooling device using a 440 (± 15) nm LED (25 W) for **48** hours. After the reaction was completed, the solvents were evaporated under reduced pressure, and the residue was purified with chromatography column on silica gel or preparative TLC (PTLC) (DCM/MeOH = 100:1).

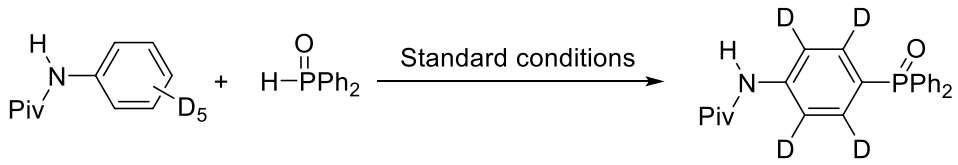
b) Deprotection of N-(4-(oxophosphanyl)phenyl)pivalamide[2]



In a 20 mL tube, **3a** (0.2 mmol) and NaOH (4.0 equiv.) were added in EtOH (2.0 mL) under reflux for 12 hours. After evaporation of solvents under reduced pressure, the residue was purified by column chromatography (DCM/MeOH) on silica gel to give the product **6** as white solid (41.5 mg, 71%).

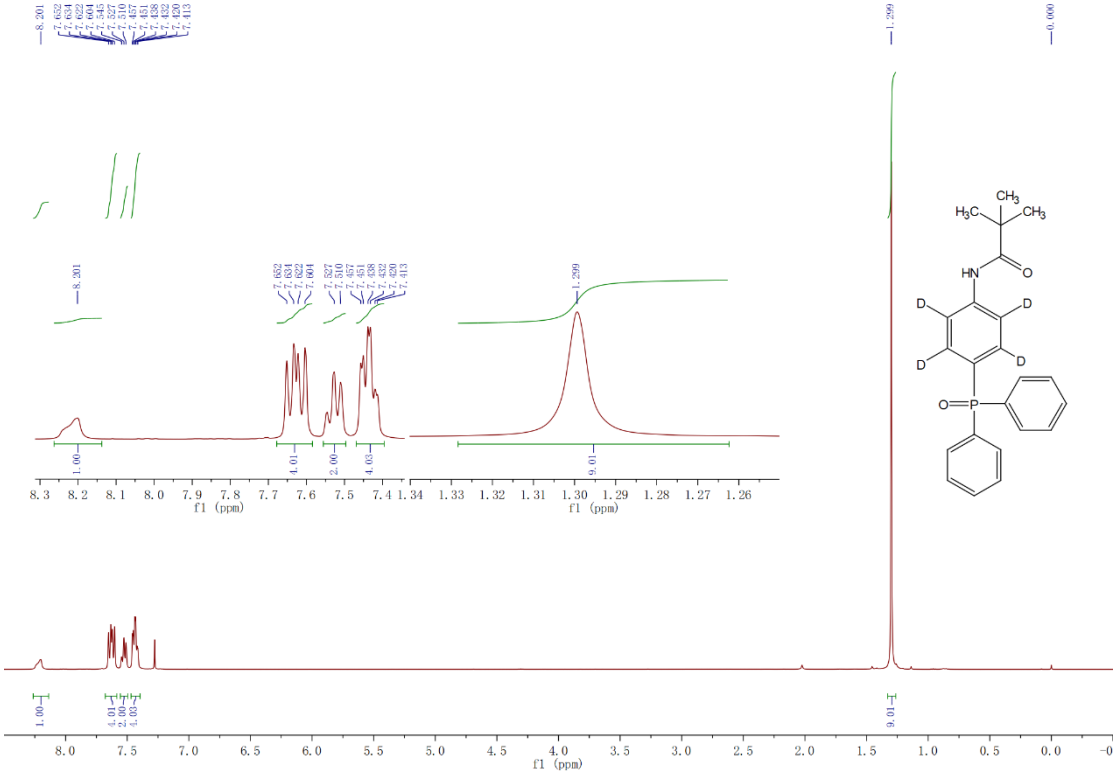
5. Mechanistic Studies

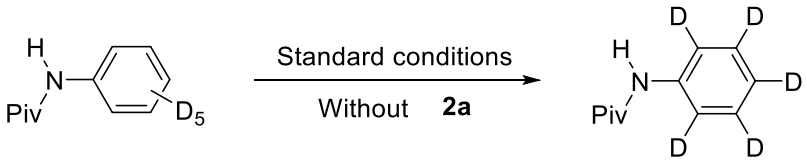
a) Isotopic labeling studies



1a-[D_5] **2a** **3a-[D_4], 73%**

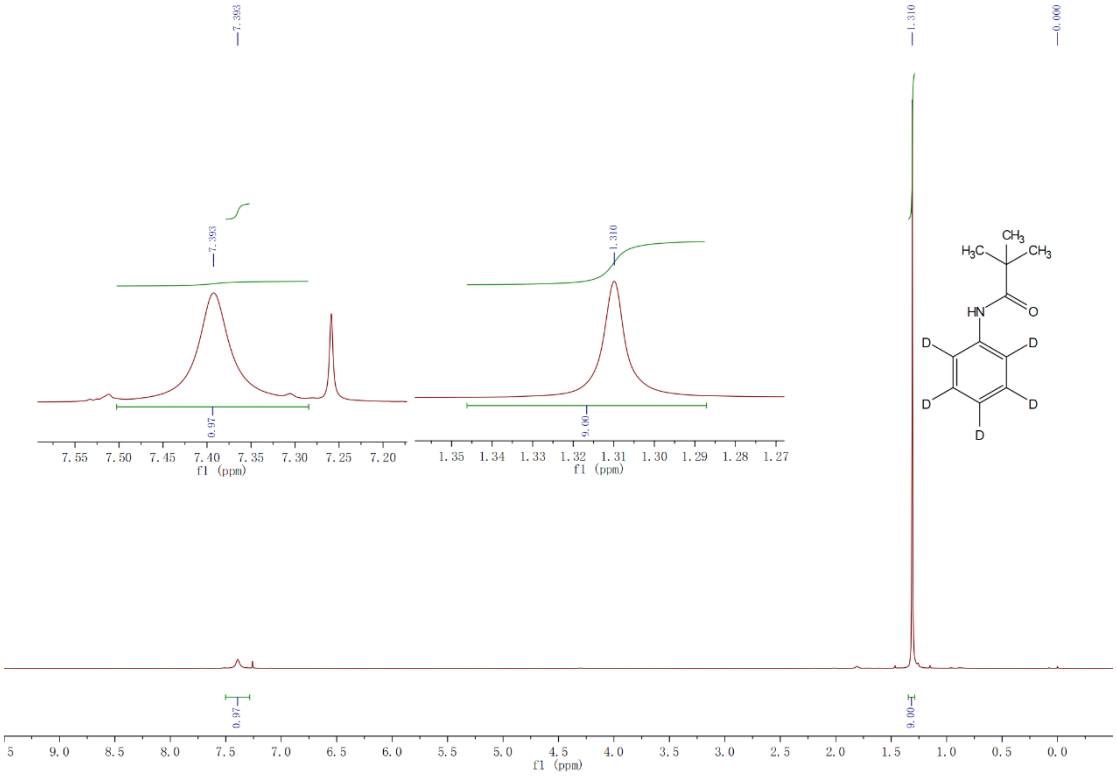
The general procedure was followed, the reaction was run on **1a-[D_5]** (0.2 mmol, 1.0 equiv.), **2a** (0.8 mmol, 4.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O:MeCN = 4:1 (1.6 mL:0.4 mL). The residue was purified by column chromatography (DCM/MeOH) on silica gel to give a 73% yield of **3a-[D_4]**, which was analyzed by ^1H NMR spectroscopy.

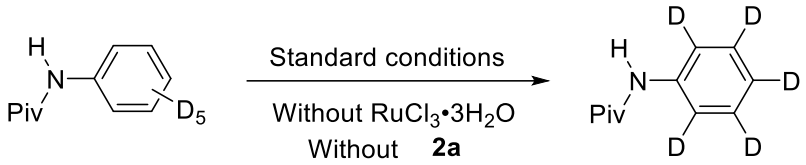




1a-[D_5] **1a-[D_5], 92%**

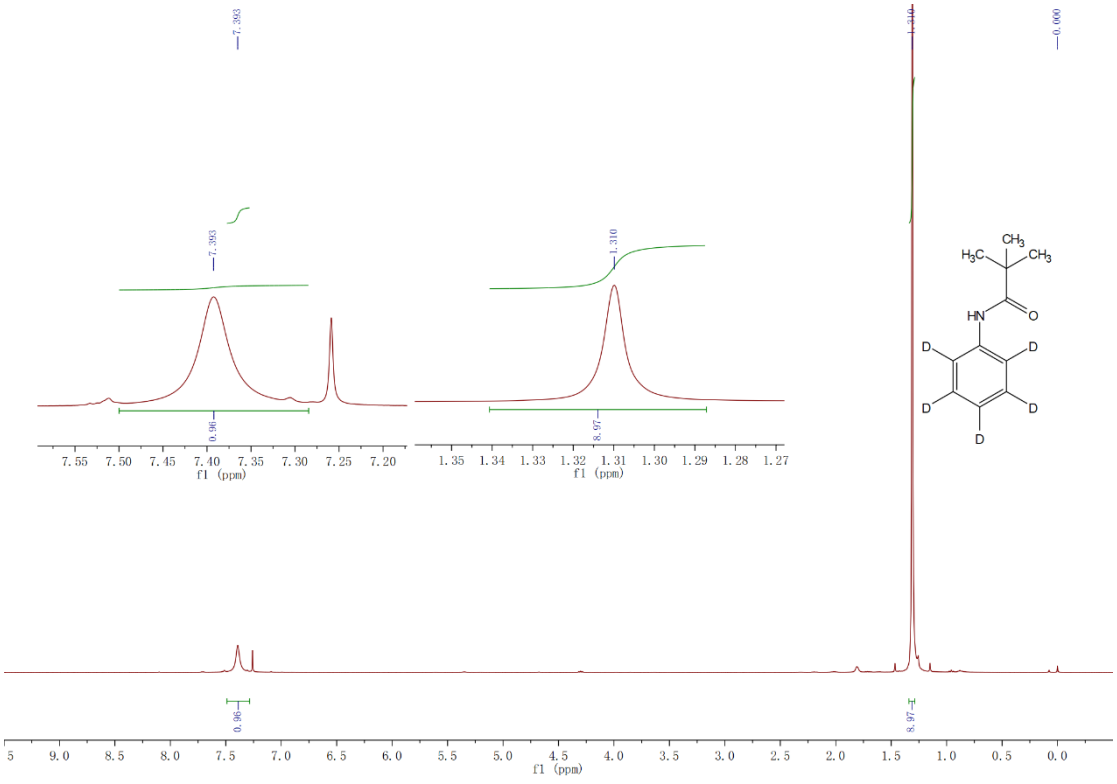
The general procedure was followed, the reaction was run on **1a-[D_5]** (0.2 mmol, 1.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O:MeCN = 4:1 (1.6 mL:0.4 mL). The residue was purified by column chromatography (DCM/MeOH) on silica gel to give a 92% yield of **1a-[D_5]**, which was analyzed by ^1H NMR spectroscopy.



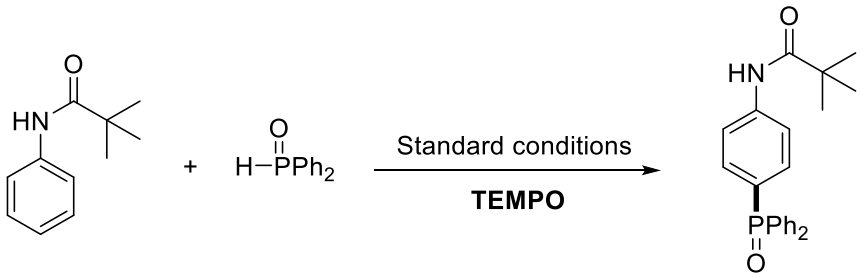


1a-[D_5] **1a-[D_5], 93%**

The general procedure was followed, the reaction was run on **1a-[D_5]** (0.2 mmol, 1.0 equiv.), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O:MeCN = 4:1 (1.6 mL:0.4 mL). The residue was purified by column chromatography (DCM/MeOH) on silica gel to give a 93% yield of **1a-[D_5]**, which was analyzed by ^1H NMR spectroscopy.

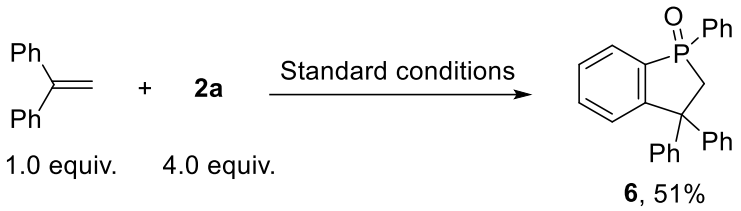


b) Radical trapping experiments



1a **2a** **3a,** 0%

The general procedure was followed, the reaction was run on **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.8 mmol, 4.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), **TEMPO** (3.0 equiv.), H_2O:MeCN = 4:1 (1.6 mL:0.4 mL). After the reaction was completed, the TLC and GC-MS were employed to observe the result, no desired product had been observed.



6, 51%

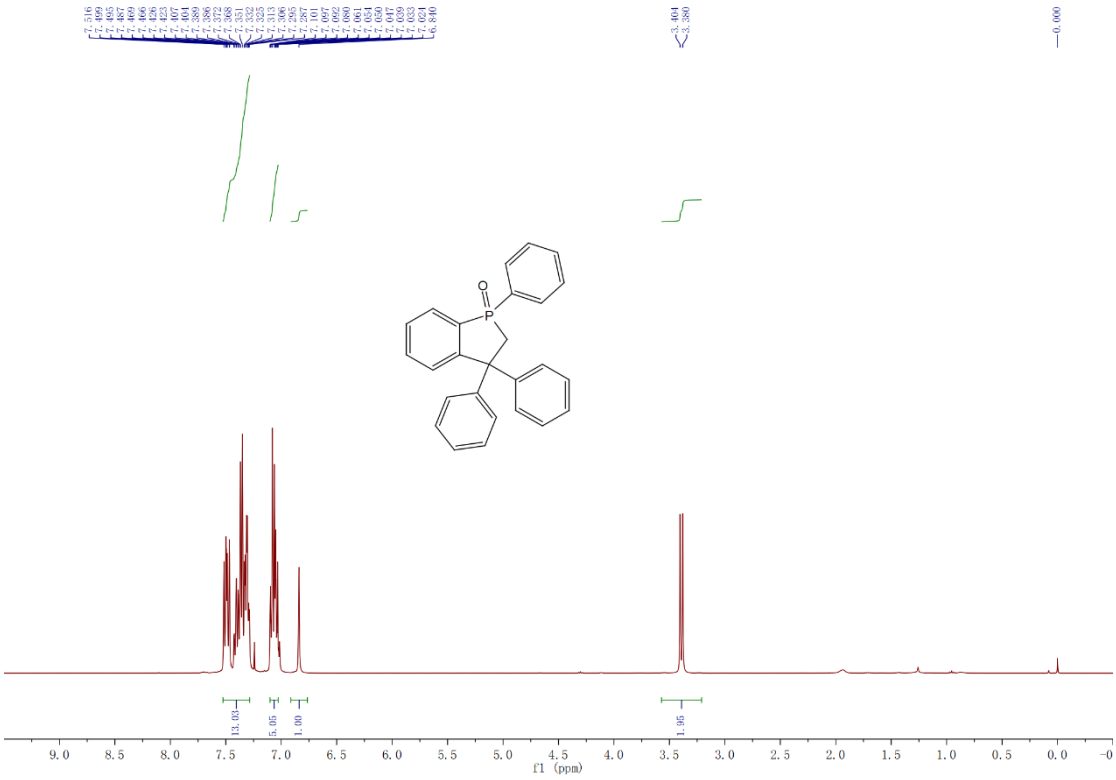
The general procedure was followed, the reaction was run on 1, 2-diphenyl ethylene (0.2 mmol, 1.0 equiv.), **2a** (0.8 mmol, 4.0 equiv.), $RuCl_3 \cdot 3H_2O$ (10 mol %), NaOAc (3.0 equiv.), $K_2S_2O_8$ (3.0 equiv.), $AgNO_3$ (20 mol %), H_2O:MeCN = 4:1 (1.6 mL:0.4 mL). The residue was purified by flash silica gel column chromatography (DCM/MeOH) to give the corresponding product **6** as White solid (38.7 mg, 51%), which was analyzed by ^1H NMR and ^{31}P NMR spectroscopy.

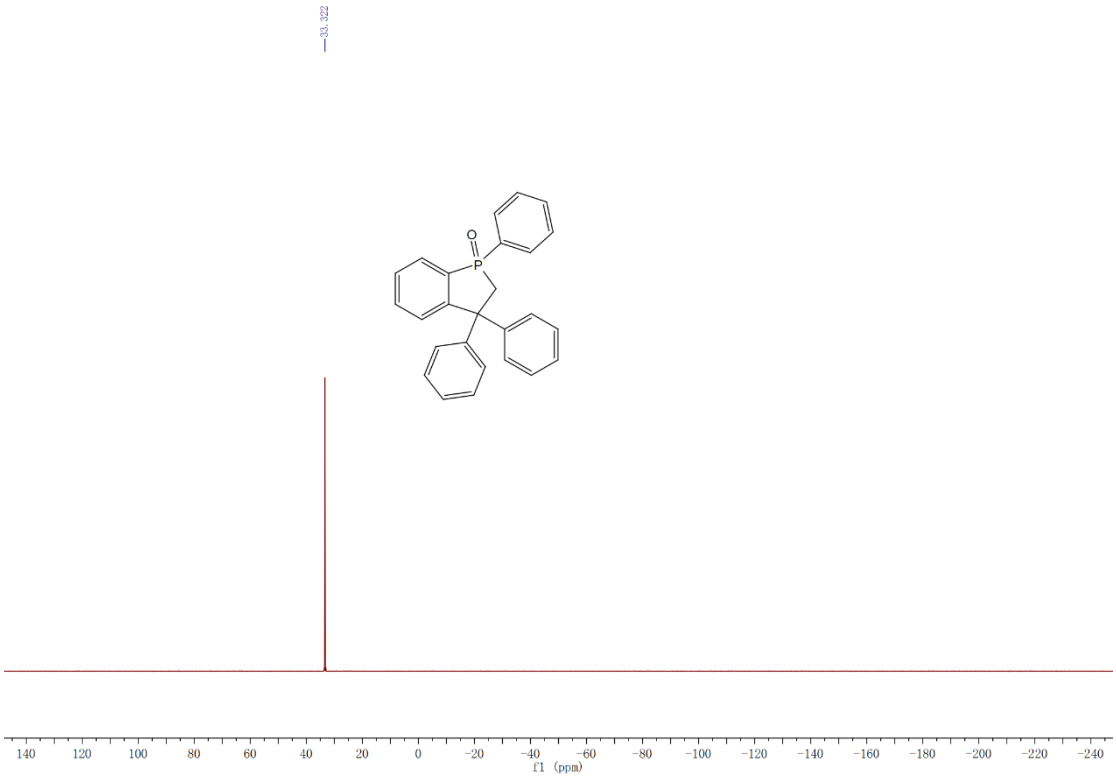
1,3,3-triphenyl-2,3-dihydrophosphindole 1-oxide (6)

^1H NMR (400 MHz, Chloroform-*d*) δ 7.5 – 7.3 (m, 13H), 7.1 – 7.0 (m, 5H), 6.8 (s, 1H), 3.4 (d, J = 9.6 Hz, 2H).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 33.3.

HRMS (ESI) Calcd for $C_{26}H_{21}OP$ $[M+H]^+$: 381.1403, found: 381.1400.




33.322
O
P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

6. Identification of the photocatalyst

a) The UV-vis absorption spectra

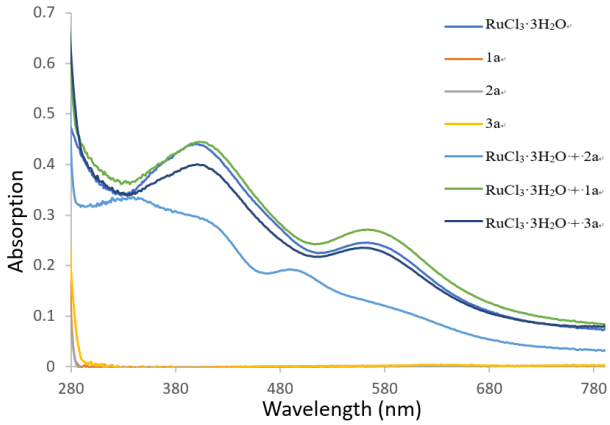


Figure S1. The UV-vis absorption spectra.

(1). $RuCl_3 \cdot 3H_2O$ (10 mol %, 0.01 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

(2). **1a** (1.0 equiv., 0.1 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

(3). **2a** (4.0 equiv., 0.1 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

(4). **3a** (10 mol %, 0.01 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

(5). $RuCl_3 \cdot 3H_2O$ (10 mol %, 0.01 mmol) + **2a** (4.0 equiv., 0.1 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

(6). $RuCl_3 \cdot 3H_2O$ (10 mol %, 0.01 mmol) + **1a** (1.0 equiv., 0.1 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

(7). $RuCl_3 \cdot 3H_2O$ (10 mol %, 0.01 mmol) + **3a** (10 mol %, 0.01 mmol) in 5.0 mL H_2O:MeCN = 4:1 (4.0 mL:1.0 mL).

Noted: on the basis of the above concentration, taking out 200 uL and dilute to the cuvette containing 3.0 mL solvent (H_2O:MeCN = 4:1) respectively.

b) On/off light experiments.

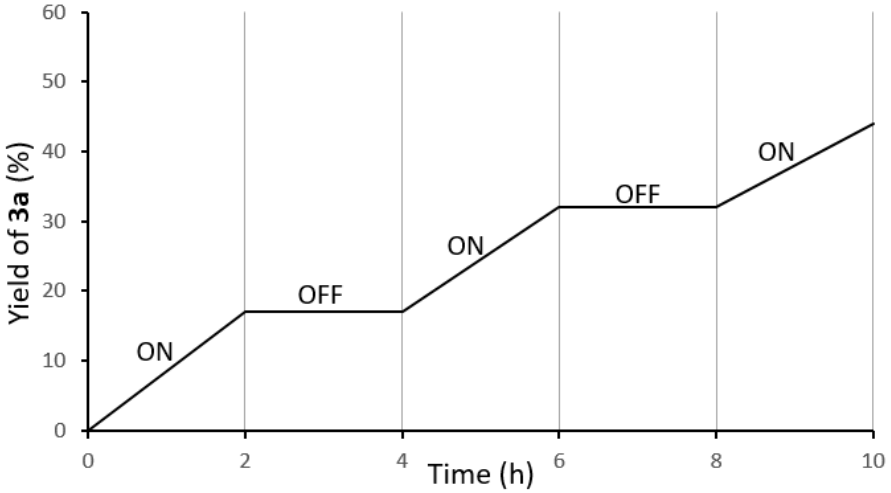


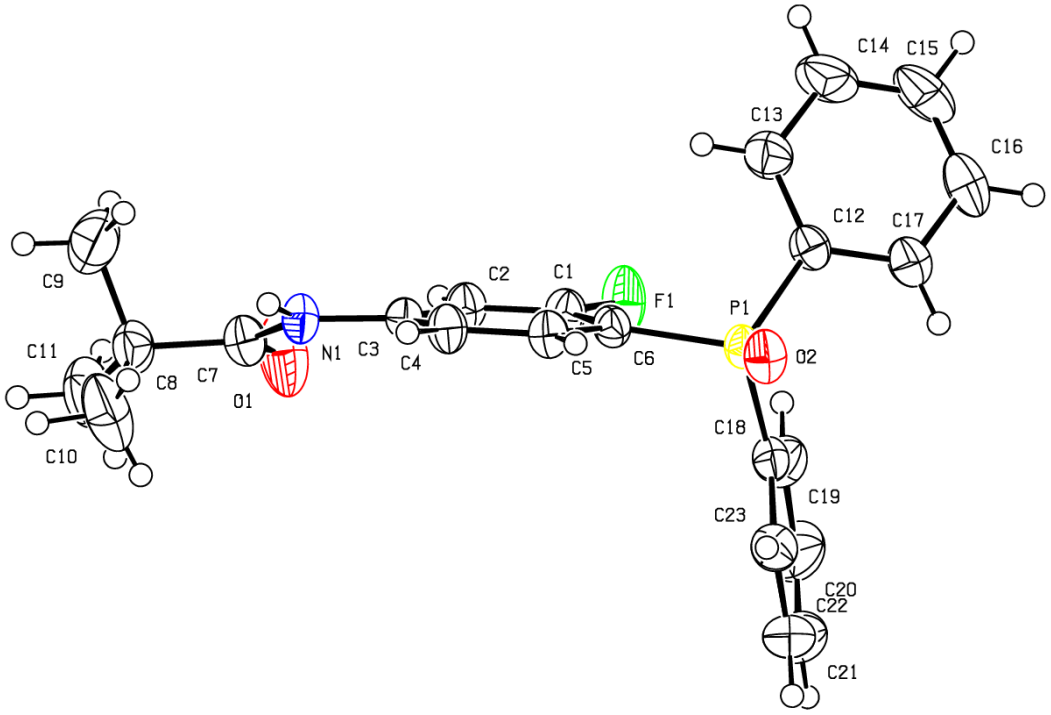
Figure S2. Light ON/OFF experiment for the synthesis of **3a**.

The reaction was set up following the general procedure on a 0.1 mmol scale by using five 20 mL tubes (**No.1**, **No.2**, **No.3**, **No.4** and **No.5**). Under alternating periods of irradiation (blue LEDs) and darkness (wrapped in tinfoil), proceed as follows. Then the separation yield is obtained by column chromatography (DCM/MeOH) on silica gel.

Time / Number	0~2 h	2~4 h	4~6 h	6~8 h	8~10 h
No.1	ON				
No.2	ON	OFF			
No.3	ON	OFF	ON		
No.4	ON	OFF	ON	OFF	
No.5	ON	OFF	ON	OFF	ON

7. X-ray Crystallographic Data of 3j

Crystals were grown from a mixture of ethyl acetate and n-hexane. The ellipsoid contour percent probability level is 30% in the caption of the thermalellipsoid plot. (CCDC: 1973724)



Bond precision:	C-C = 0.0040 A		Wavelength=1.54184
Cell:	a=13.7106 (3)	b=10.0817 (2)	c=19.9799 (4)
	alpha=90	beta=91.4380 (18)	gamma=90
Temperature:	236 K		

	Calculated	Reported
Volume	2760.88 (10)	2760.85 (10)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C23 H23 F N O2 P, C H Cl3 [+ solvent]	C H Cl3, C23 H23 F N O2 P
Sum formula	C24 H24 Cl3 F N O2 P [+ solvent]	C24 H24 Cl3 F N O2 P
Mr	514.76	514.76
Dx,g cm-3	1.238	1.238
Z	4	4
Mu (mm-1)	3.774	3.775
F000	1064.0	1064.0
F000’	1072.05	

h,k,lmax	16,12,23	16,12,23
Nref	4876	4874
Tmin,Tmax	0.668,0.739	0.368,1.000
Tmin’	0.561	

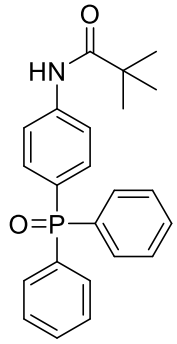
Correction method= # Reported T Limits: Tmin=0.368 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.000 Theta(max)= 66.594

R(reflections)= 0.0487(3974) wR2(reflections)= 0.1399(4874)

S = 1.046 Npar= 292

8. Characterization Data



N-(4-(diphenylphosphoryl)phenyl)pivalamide (**3a)**

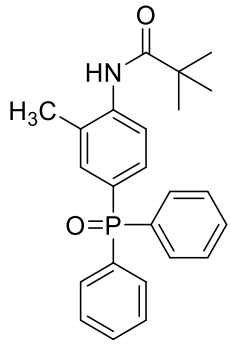
57.4 mg, yield: 76%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.4 (s, 1H), 7.8 – 7.7 (m, 2H), 7.7 – 7.6 (m, 4H), 7.6 – 7.5 (m, 4H), 7.5 – 7.4 (m, 4H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.4, 142.0 (d, *J* = 2.7 Hz), 132.8 (d, *J* = 10.7 Hz), δ 132.3 (d, *J* = 104.8 Hz). 131.9 (d, *J* = 10.1 Hz), 128.4 (d, *J* = 12.2 Hz), 126.2 (d, *J* = 108.5 Hz), 119.7 (d, *J* = 12.5 Hz), 39.7, 27.4.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.4.

HRMS (ESI) Calcd for $C_{23}H_{24}NO_2P$ $[M+H]^+$: 378.1545, found: 378.1541.



N-(4-(diphenylphosphoryl)-2-methylphenyl)pivalamide (3b)

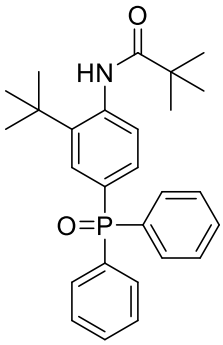
46.2 mg, yield: 59%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.1 (s, 1H), 7.7 – 7.6 (m, 6H), 7.5 (t, *J* = 7.3 Hz, 2H), 7.5 – 7.4 (m, 4H), 7.4 – 7.3 (m, 1H), 2.3 (s, 3H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 176.7, 139.5, 133.9 (d, *J* = 9.9 Hz), 132.5 (d, *J* = 102.9 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 2.7 Hz), 130.9 (d, *J* = 11.1 Hz), 128.4 (d, *J* = 12.2 Hz), 127.4 (d, *J* = 107.1 Hz), 121.5 (d, *J* = 12.9 Hz), 121.4 (d, *J* = 12.6 Hz), 39.9, 27.6, 17.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.0.

HRMS (ESI) Calcd for $C_{24}H_{26}NO_2P$ $[M+H]^+$: 392.1774, found: 392.1771.



N-(2-(tert-butyl)-4-(diphenylphosphoryl)phenyl)pivalamide (3c)

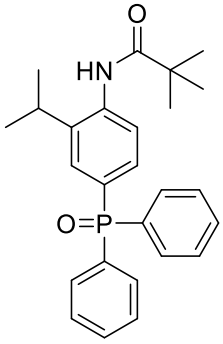
33.8 mg, yield: 39%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.0 (dd, *J* = 8.3, 3.2 Hz, 1H), 7.8 (dd, *J* = 13.0, 1.4 Hz, 1H), 7.7 – 7.6 (m, 5H), 7.6 – 7.5 (m, 2H), 7.5 – 7.4 (m, 4H), 7.4 – 7.3 (m, 1H), 1.4 (s, 9H), 1.4 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 176.2, 140.5 (d, *J* = 11.4 Hz), 139.3 (d, *J* = 3.1 Hz), 132.7 (d, *J* = 104.2 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 2.7 Hz), 130.7 (d, *J* = 11.3 Hz), 130.6 (d, *J* = 10.9 Hz), 128.4 (d, *J* = 12.1 Hz), 128.1 (d, *J* = 107.0 Hz), 125.3 (d, *J* = 13.1 Hz), 39.8, 34.6, 30.3, 27.6.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.4.

HRMS (ESI) Calcd for $C_{27}H_{32}NO_2P$ $[M+H]^+$: 3434.2243, found: 434.2241.



N-(4-(diphenylphosphoryl)-2-isopropylphenyl)pivalamide (3d)

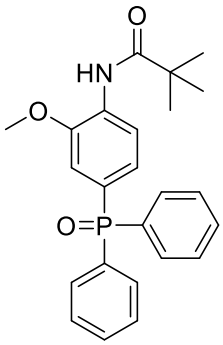
24.3 mg, yield: 29%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.1 – 8.1 (m, 1H), 7.8 (dd, *J* = 12.5, 1.4 Hz, 1H), 7.7 – 7.5 (m, 7H), 7.5 – 7.4 (m, 4H), 7.3 – 7.3 (m, 1H), 3.0 (h, *J* = 6.9 Hz, 1H), 1.3 (s, 9H), 1.3 (s, 3H), 1.2 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 176.6, 138.1 (d, *J* = 3.2 Hz), 138.1 (d, *J* = 11.7 Hz), 132.7 (d, *J* = 104.2 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 2.7 Hz), 130.6 (d, *J* = 11.4 Hz), 129.5 (d, *J* = 10.1 Hz), 128.4 (d, *J* = 12.1 Hz), 127.8 (d, *J* = 107.1 Hz), 122.2 (d, *J* = 13.0 Hz), 40.0, 28.3, 27.6, 22.3.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.4.

HRMS (ESI) Calcd for $C_{26}H_{30}NO_2P$ $[M+H]^+$: 420.2087, found: 420.2084.



N-(4-(diphenylphosphoryl)-2-methoxyphenyl)pivalamide (3e)

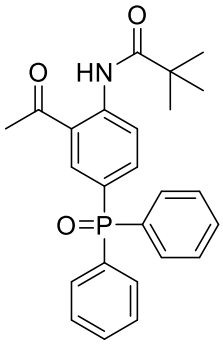
71.7 mg, yield: 88%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.5 (dd, *J* = 8.2, 3.6 Hz, 1H), 8.3 (s, 1H), 7.7 – 7.6 (m, 4H), 7.5 (t, *J* = 7.4 Hz, 2H), 7.5 – 7.4 (m, 5H), 7.0 – 6.9 (m, 1H), 3.9 (s, 3H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 176.8, 147.9 (d, *J* = 14.4 Hz), 132.5 (d, *J* = 106.5 Hz), 131.9 (d, *J* = 10.0 Hz), 131.8 (d, *J* = 2.8 Hz), 131.2 (d, *J* = 2.9 Hz), 128.4 (d, *J* = 12.2 Hz), 126.2 (d, *J* = 107.6 Hz), 125.6 (d, *J* = 11.1 Hz), 118.5 (d, *J* = 14.4 Hz), 112.7 (d, *J* = 10.4 Hz), 56.1, 40.1, 27.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.6.

HRMS (ESI) Calcd for $C_{24}H_{26}NO_3P$ $[M+H]^+$: 408.1723, found: 408.1722.



N-(4-(diphenylphosphoryl)-2-methoxyphenyl)pivalamide (3f)

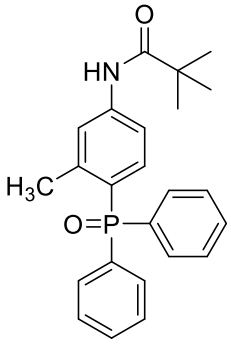
21.8 mg, yield: 26%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 11.8 (s, 1H), 9.0 (dd, *J* = 13.4, 1.2 Hz, 1H), 8.0 (dd, *J* = 8.1, 2.9 Hz, 1H), 7.8 – 7.7 (m, 5H), 7.6 – 7.5 (m, 2H), 7.5 – 7.5 (m, 4H), 2.7 (s, 3H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 202.7, 178.2, 140.8 (d, *J* = 15.3 Hz), 139.7 (d, *J* = 99.7 Hz), 132.2 (d, *J* = 2.8 Hz), 132.0 (d, *J* = 10.0 Hz), 131.9, 131.5 (d, *J* = 87.2 Hz), 131.5 (d, *J* = 12.7 Hz), 128.6 (d, *J* = 12.3 Hz), 125.3 (d, *J* = 8.3 Hz), 124.0 (d, *J* = 14.0 Hz), 40.3, 28.8, 27.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 27.5.

HRMS (ESI) Calcd for $C_{25}H_{26}NO_3P$ $[M+H]^+$: 420.1723, found: 420.1720.



N-(4-(diphenylphosphoryl)-3-methylphenyl)pivalamide (3g)

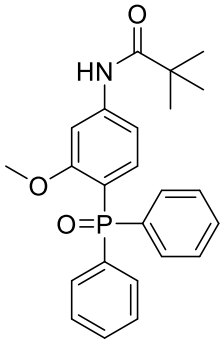
49.3 mg, yield: 63%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.0 (s, 1H), 7.7 – 7.4 (m, 12H), 6.9 (dd, *J* = 13.5, 8.4 Hz, 1H), 2.3 (s, 3H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.3, 144.2 (d, *J* = 8.9 Hz), 141.8 (d, *J* = 2.9 Hz), 134.5 (d, *J* = 13.7 Hz), 132.7 (d, *J* = 104.0 Hz), 131.8 (d, *J* = 9.8 Hz), 131.7 (d, *J* = 3.1 Hz), 128.5 (d, *J* = 12.1 Hz), 125.3 (d, *J* = 107.1 Hz), 122.7 (d, *J* = 10.8 Hz), 116.2 (d, *J* = 13.3 Hz), 39.7, 27.4, 21.6.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 31.4.

HRMS (ESI) Calcd for $C_{24}H_{26}NO_2P$ $[M+H]^+$: 392.1774, found: 392.1773.



N-(4-(diphenylphosphoryl)-3-methoxyphenyl)pivalamide (3h)

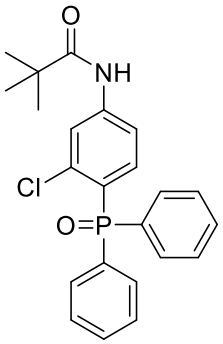
57.8 mg, yield: 71%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.5 (s, 1H), 7.8 – 7.8 (m, 1H), 7.7 – 7.5 (m, 7H), 7.4 – 7.3 (m, 4H), 7.0 (s, 1H), 3.5 (s, 3H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.7, 161.6 (d, *J* = 3.9 Hz), 144.5, 134.8 (d, *J* = 7.5 Hz), 133.1 (d, *J* = 107.9 Hz), 131.7 (d, *J* = 10.4 Hz), 131.4 (d, *J* = 2.7 Hz), 128.0 (d, *J* = 12.5 Hz), 114.0 (d, *J* = 110.4 Hz), 111.7 (d, *J* = 11.8 Hz), 103.2 (d, *J* = 6.7 Hz), 55.1, 39.9, 27.4.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 27.7.

HRMS (ESI) Calcd for $C_{24}H_{26}NO_3P$ $[M+H]^+$: 408.1723, found: 408.1720.



N-(3-chloro-4-(diphenylphosphoryl)phenyl)pivalamide (3i)

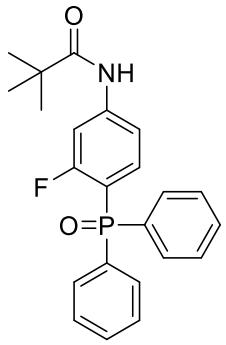
27.2 mg, yield: 33%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.3 (s, 1H), 7.9 – 7.9 (m, 1H), 7.7 (dd, *J* = 12.4, 8.1 Hz, 4H), 7.6 – 7.3 (m, 8H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.5, 143.2 (d, *J* = 2.4 Hz), 138.3 (d, *J* = 4.4 Hz), 136.0 (d, *J* = 9.8 Hz), 132.0 (d, *J* = 3.6 Hz), 131.9 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 108.3 Hz), 128.5 (d, *J* = 12.5 Hz), 124.9 (d, *J* = 108.6 Hz), 122.1 (d, *J* = 7.0 Hz), 117.6 (d, *J* = 11.3 Hz), 39.9, 27.4.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 28.5.

HRMS (ESI) Calcd for $C_{23}H_{23}ClNO_2P$ $[M+H]^+$: 412.1228, found: 412.1229.



N-(4-(diphenylphosphoryl)-3-fluorophenyl)pivalamide (3j)

37.2 mg, yield: 47%. White solid.

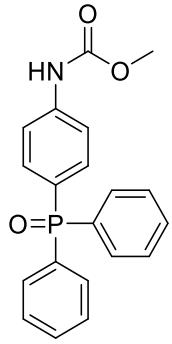
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.3 (s, 1H), 7.8 – 7.8 (m, 1H), 7.7 (dd, *J* = 12.7, 7.3 Hz, 5H), 7.6 – 7.5 (m, 2H), 7.4 (td, *J* = 7.5, 3.0 Hz, 4H), 7.3 – 7.3 (m, 1H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.5, 164.6 (d, *J* = 2.6 Hz), 163.4 (d, J_{C-F} = 248.5 Hz), 163.3 (d, J_{C-F} = 248.3 Hz), 162.1 (d, *J* = 2.6 Hz), 144.7 (d, *J* = 11.6 Hz), 132.1 (d, J_{C-P} = 108.7 Hz), 132.1 (d, *J* = 2.7 Hz), 131.7 (d, *J* = 1.6 Hz), 131.5 (d, *J* = 1.4 Hz), 128.5 (d, *J* = 12.7 Hz), 115.4 (d, *J* = 2.6 Hz), 115.3 (d, *J* = 2.5 Hz), 114.3 (d, J_{C-F} = 19.3 Hz), 113.8 (d, J_{C-P} = 105.8 Hz), 113.6 (d, J_{C-P} = 105.2 Hz), 113.2 (d, J_{C-F} = 18.7 Hz), 107.8 (d, *J* = 5.7 Hz), 107.7 (d, J_{C-F} = 28.4 Hz), 107.6 (d, J_{C-F} = 28.5 Hz), 107.5 (d, *J* = 5.8 Hz), 39.9, 27.3.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 24.8.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -97.5.

HRMS (ESI) Calcd for $C_{23}H_{23}FNO_2P$ $[M+H]^+$: 396.1523, found: 396.1521.



methyl (4-(diphenylphosphoryl)phenyl)carbamate (3k)

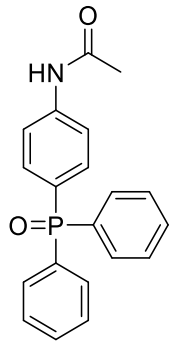
52.7 mg, yield: 75%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.8 (s, 1H), 7.7 – 7.6 (m, 6H), 7.5 – 7.4 (m, 8H), 3.6 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.6, 142.3 (d, *J* = 3.0 Hz), 132.9 (d, *J* = 10.8 Hz), 132.1 (d, *J* = 104.9 Hz), 132.1 (d, *J* = 2.7 Hz), 131.9 (d, *J* = 10.0 Hz), 128.6 (d, *J* = 12.2 Hz), 125.9 (d, *J* = 108.8 Hz), 119.5 (d, *J* = 12.4 Hz), 24.4.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.7.

HRMS (ESI) Calcd for $C_{20}H_{18}NO_3P$ $[M+H]^+$: 352.1097, found: 352.1095.



N-(4-(diphenylphosphoryl)phenyl)acetamide (3l)

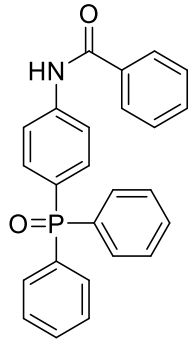
55.0 mg, yield: 82%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.5 (s, 1H), 7.7 – 7.6 (m, 6H), 7.6 – 7.5 (m, 2H), 7.5 – 7.4 (m, 6H), 2.1 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.1, 142.4, 133.0 (d, *J* = 10.8 Hz), 132.4 (d, *J* = 106.1 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 2.7 Hz), 128.4 (d, *J* = 13.1 Hz), 125.0 (d, *J* = 109.7 Hz), 118.1 (d, *J* = 12.7 Hz), 52.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.0.

HRMS (ESI) Calcd for $C_{20}H_{18}NO_2P$ $[M+H]^+$: 336.1148, found: 336.1146.



N-(4-(diphenylphosphoryl)phenyl)benzamide (3m)

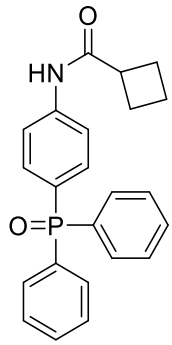
56.4 mg, yield: 71%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.1 – 9.9 (m, 1H), 8.0 (d, *J* = 7.3 Hz, 2H), 7.9 – 7.8 (m, 2H), 7.6 – 7.4 (m, 13H), 7.3 – 7.3 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.7, 142.5 (d, *J* = 3.9 Hz), 134.6, 132.7 (d, *J* = 4.7 Hz), 132.4 (d, *J* = 85.7 Hz), 131.9 (d, *J* = 10.1 Hz), 131.7 (d, *J* = 9.5 Hz), 128.5 (d, *J* = 12.2 Hz), 128.3, 127.7, 126.2 (d, *J* = 107.2 Hz), 120.3 (d, *J* = 12.5 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.8.

HRMS (ESI) Calcd for $C_{25}H_{20}NO_2P$ $[M+H]^+$: 398.1304, found: 398.1303.



N-(4-(diphenylphosphoryl)phenyl)cyclobutanecarboxamide (3n)

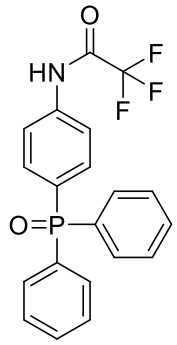
59.9 mg, yield: 80%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.4 (s, 1H), 7.8 (d, *J* = 7.1 Hz, 2H), 7.6 (dd, *J* = 12.0, 7.2 Hz, 4H), 7.6 – 7.4 (m, 8H), 3.2 (p, *J* = 8.4 Hz, 1H), 2.4 – 2.3 (m, 2H), 2.1 – 2.0 (m, 2H), 1.9 – 1.7 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 142.6 (d, *J* = 3.0 Hz), 132.8 (d, *J* = 10.8 Hz), 132.2 (d, *J* = 104.7 Hz), 132.0 (d, *J* = 2.7 Hz), 131.9 (d, *J* = 10.0 Hz), 128.5 (d, *J* = 12.1 Hz), 125.4 (d, *J* = 109.2 Hz), 119.4 (d, *J* = 12.6 Hz), 40.5, 25.1, 18.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.9.

HRMS (ESI) Calcd for $C_{23}H_{22}NO_2P$ $[M+H]^+$: 376.1461, found: 376.1460.



N-(4-(diphenylphosphoryl)phenyl)-2,2,2-trifluoroacetamide (3o)

17.9 mg, yield: 23%. White solid.

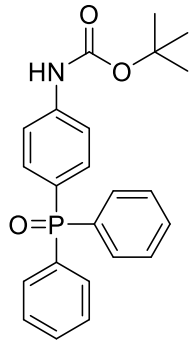
^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.4 (s, 1H), 7.8 (d, *J* = 6.8 Hz, 2H), 7.7 – 7.6 (m, 4H), 7.6 – 7.4 (m, 8H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 147.9 (d, *J* = 37.8 Hz), 132.9 (d, *J* = 3.1 Hz), 123.5 (d, *J* = 106.1 Hz), 125.2 (d, *J* = 10.9 Hz), 124.8 (d, *J* = 2.7 Hz), 124.0 (d, *J* = 10.2 Hz), 121.0 (d, *J* = 12.4 Hz), 120.3 (d, *J* = 107.9 Hz), 112.9 (d, *J* = 12.6 Hz), 108.2 (d, *J* = 287.5 Hz).

^{19}F NMR (376 MHz, Chloroform-*d*) δ -75.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.1.

HRMS (ESI) Calcd for $C_{20}H_{15}F_3NO_2P$ $[M+H]^+$: 390.0865, found: 390.0865.



tert-butyl (4-(diphenylphosphoryl)phenyl)carbamate (3p)

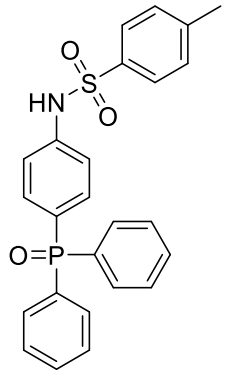
48.8 mg, yield: 62%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.8 (s, 1H), 7.7 – 7.6 (m, 4H), 7.6 – 7.5 (m, 6H), 7.5 – 7.4 (m, 4H), 1.5 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 152.6, 142.4 (d, *J* = 3.0 Hz), 133.0 (d, *J* = 10.9 Hz), 132.6 (d, *J* = 104.5 Hz), 132.0 (d, *J* = 10.0 Hz), 131.8 (d, *J* = 2.7 Hz), 128.4 (d, *J* = 12.1 Hz), 125.0 (d, *J* = 109.6 Hz), 117.9 (d, *J* = 12.6 Hz), 80.7, 28.2.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.3.

HRMS (ESI) Calcd for $C_{23}H_{24}NO_3P$ $[M+H]^+$: 394.1567, found: 394.1565.



N-(4-(diphenylphosphoryl)phenyl)-4-methylbenzenesulfonamide (3q)

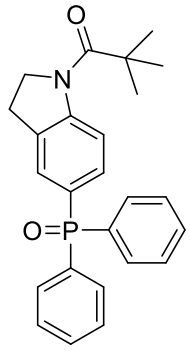
32.2 mg, yield: 36%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.6 (m, 6H), 7.5 – 7.5 (m, 2H), 7.5 – 7.4 (m, 6H), 7.2 (dd, *J* = 8.6, 2.1 Hz, 2H), 7.1 (d, *J* = 8.2 Hz, 2H), 2.3 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.7, 141.3 (d, *J* = 2.9 Hz), 136.5, 133.2 (d, *J* = 10.9 Hz), 132.0 (d, *J* = 105.0 Hz), 131.9 (d, *J* = 9.9 Hz), 131.9 (d, *J* = 3.9 Hz), 129.5, 128.4 (d, *J* = 12.2 Hz), 127.1, 126.4 (d, *J* = 108.2 Hz), 119.0 (d, *J* = 12.7 Hz), 21.4.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.6.

HRMS (ESI) Calcd for $C_{25}H_{22}NO_3PS$ $[M+H]^+$: 448.1131, found: 448.1129.



1-(5-(diphenylphosphoryl)indolin-1-yl)-2,2-dimethylpropan-1-one (3r)

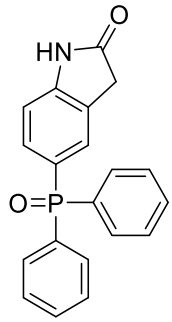
59.7 mg, yield: 74%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.3 (d, *J* = 8.3 Hz, 1H), 7.7 – 7.3 (m, 13H), 4.3 (t, *J* = 8.2 Hz, 2H), 3.1 (t, *J* = 8.1 Hz, 2H), 1.4 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.1, 148.0 (d, *J* = 2.9 Hz), 132.8 (d, *J* = 104.2 Hz), 132.1 (d, *J* = 11.4 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 2.7 Hz), 131.4 (d, *J* = 13.2 Hz), 128.4 (d, *J* = 12.1 Hz), 127.8 (d, *J* = 10.4 Hz), 126.6 (d, *J* = 107.7 Hz), 117.8 (d, *J* = 13.1 Hz), 49.7, 40.3, 28.9, 27.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.1.

HRMS (ESI) Calcd for $C_{25}H_{26}NO_2P$ $[M+H]^+$: 404.1774, found: 404.1772.



N-(4-(diphenylphosphoryl)phenyl)acetamide (3s)

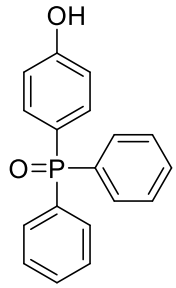
46.0 mg, yield: 69%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.2 (s, 1H), 7.7 – 7.6 (m, 4H), 7.6 – 7.4 (m, 8H), 6.9 (d, *J* = 7.8 Hz, 1H), 3.5 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.4, 146.9 (d, *J* = 2.3 Hz), 132.7 (d, *J* = 11.0 Hz), 132.3 (d, *J* = 104.8 Hz), 132.0 (d, *J* = 10.0 Hz), 132.0 (d, *J* = 2.7 Hz), 128.5 (d, *J* = 12.2 Hz), 127.7 (d, *J* = 11.5 Hz), 125.8 (d, *J* = 14.0 Hz), 124.8 (d, *J* = 109.0 Hz), 109.9 (d, *J* = 13.4 Hz), 35.9.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.3.

HRMS (ESI) Calcd for $C_{20}H_{16}NO_2P$ $[M+H]^+$: 334.0991, found: 334.0990.



(4-hydroxyphenyl)diphenylphosphine oxide (3t)

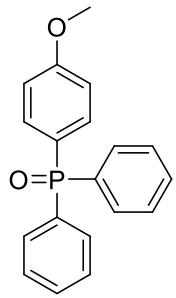
22.9 mg, yield: 39%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.7 (s, 1H), 7.6 (dd, *J* = 12.0, 7.4 Hz, 4H), 7.5 – 7.3 (m, 8H), 7.0 (d, *J* = 7.1 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.8 (d, *J* = 2.6 Hz), 133.9 (d, *J* = 11.7 Hz), 132.2 (d, *J* = 105.6 Hz), 132.0 (d, *J* = 10.2 Hz), 132.0 (d, *J* = 3.1 Hz), 128.5 (d, *J* = 12.2 Hz), 119.8 (d, *J* = 113.9 Hz), 116.3 (d, *J* = 13.5 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 32.0.

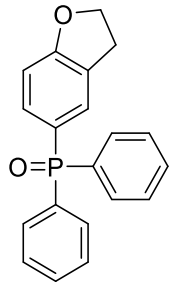
HRMS (ESI) Calcd for $C_{18}H_{15}O_2P$ $[M+H]^+$: 295.0882, found: 295.0881.



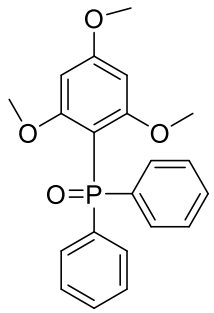
(4-methoxyphenyl)diphenylphosphine oxide (3u)

46.8 mg, yield: 76% (o + p). White solid.

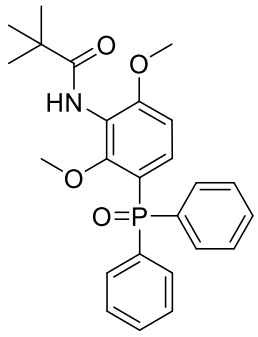
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.9 – 7.3 (m, 12H), 7.1 – 6.8 (m, 2H), 3.8 (s, 1H), 3.6 (s, 2H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.4 (d, *J* = 2.8 Hz), 160.8 (d, *J* = 3.3 Hz), 134.9 (d, *J* = 7.1 Hz), 134.2 (d, *J* = 2.0 Hz), 133.9 (d, *J* = 11.2 Hz), 133.1 (d, *J* = 107.4 Hz), 132.9 (d, *J* = 104.5 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 4.7 Hz), 131.7 (d, *J* = 10.3 Hz), 131.4 (d, *J* = 2.8 Hz), 128.4 (d, *J* = 12.1 Hz), 128.0 (d, *J* = 12.5 Hz), 123.4 (d, *J* = 110.4 Hz), 120.9 (d, *J* = 11.6 Hz), 120.1 (d, *J* = 103.6 Hz), 114.0 (d, *J* = 13.2 Hz), 111.3 (d, *J* = 6.5 Hz), 55.3, 55.2.
^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.2, 27.5.
HRMS (ESI) Calcd for $C_{19}H_{17}O_2P$ $[M+H]^+$: 309.1039, found: 309.1036.



(2,3-dihydrobenzofuran-5-yl)diphenylphosphine oxide (3v)
30.8 mg, yield: 48% (o + p). White solid.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.8 – 7.6 (m, 4H), 7.6 – 7.3 (m, 8H), 7.0 – 6.8 (m, 1H), 4.7 – 4.4 (m, 2H), 3.2 (q, *J* = 8.8 Hz, 2H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.3 (d, *J* = 2.7 Hz), 161.5 (d, *J* = 3.1 Hz), 133.2 (d, *J* = 12.0 Hz), 133.0 (d, *J* = 104.4 Hz), 132.6 (d, *J* = 106.7 Hz), 132.0 (d, *J* = 9.9 Hz), 131.8 (d, *J* = 10.5 Hz), 131.7 (d, *J* = 2.7 Hz), 131.6 (d, *J* = 2.8 Hz), 129.1 (d, *J* = 2.4 Hz), 128.8 (d, *J* = 11.2 Hz), 128.3 (d, *J* = 12.1 Hz), 128.1 (d, *J* = 12.5 Hz), 127.9 (d, *J* = 14.2 Hz), 123.3 (d, *J* = 110.1 Hz), 120.3 (d, *J* = 10.8 Hz), 112.8 (d, *J* = 104.7 Hz), 109.4 (d, *J* = 14.2 Hz).
^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.6, 26.3.
HRMS (ESI) Calcd for $C_{20}H_{17}O_2P$ $[M+H]^+$: 321.1039, found: 321.1037.



diphenyl(2,4,6-trimethoxyphenyl)phosphine oxide (3w)
72.9 mg, yield: 99%. White solid.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.8 – 7.7 (m, 4H), 7.4 – 7.3 (m, 6H), 6.0 (t, *J* = 3.5 Hz, 2H), 3.8 (d, *J* = 4.9 Hz, 3H), 3.3 (d, *J* = 4.5 Hz, 6H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 165.3, 164.4, 137.0 (d, *J* = 109.8 Hz), 130.5 (d, *J* = 9.8 Hz), 130.1 (d, *J* = 2.7 Hz), 127.6 (d, *J* = 12.5 Hz), 100.8 (d, *J* = 110.8 Hz), 91.1 (d, *J* = 6.5 Hz), 55.3, 55.2.
^{31}P NMR (162 MHz, Chloroform-*d*) δ 21.4.
HRMS (ESI) Calcd for $C_{21}H_{21}O_4P$ $[M+H]^+$: 369.1250, found: 369.1248.



N-(3-(diphenylphosphoryl)-2,6-dimethoxyphenyl)pivalamide (3x)

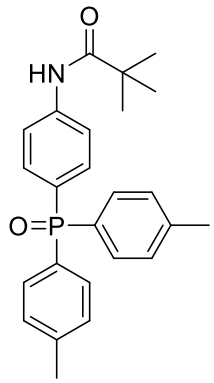
35.7 mg, yield: 41%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.3 (d, *J* = 14.1 Hz, 1H), 7.7 – 7.7 (m, 4H), 7.7 (s, 1H), 7.5 – 7.4 (m, 6H), 6.5 (d, *J* = 4.9 Hz, 1H), 3.9 (s, 3H), 3.5 (s, 3H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 176.0, 158.6 (d, *J* = 3.5 Hz), 153.8 (d, *J* = 2.6 Hz), 133.4 (d, *J* = 107.7 Hz), 131.7 (d, *J* = 10.2 Hz), 131.2 (d, *J* = 2.8 Hz), 128.0 (d, *J* = 12.4 Hz), 127.2 (d, *J* = 10.5 Hz), 120.9 (d, *J* = 15.0 Hz), 111.5 (d, *J* = 109.5 Hz), 95.4 (d, *J* = 8.1 Hz), 56.0, 55.8, 39.6, 27.6.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 27.0.

HRMS (ESI) Calcd for $C_{25}H_{28}NO_4P$ $[M+H]^+$: 438.1829, found: 438.1826.



N-(4-(di-p-tolylphosphoryl)phenyl)pivalamide (3y)

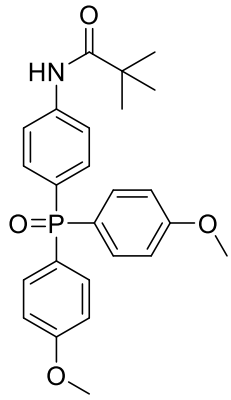
59.2 mg, yield: 73%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.3 (s, 1H), 7.7 (d, *J* = 8.3 Hz, 2H), 7.5 (dd, *J* = 11.6, 8.1 Hz, 6H), 7.2 (d, *J* = 6.7 Hz, 4H), 2.4 (s, 6H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.3, 142.2 (d, *J* = 2.7 Hz), 141.8 (d, *J* = 2.6 Hz), 132.8 (d, *J* = 10.7 Hz), 131.9 (d, *J* = 10.3 Hz), 129.4 (d, *J* = 107.0 Hz), 129.1 (d, *J* = 12.5 Hz), 127.0 (d, *J* = 108.8 Hz), 119.7 (d, *J* = 12.4 Hz), 39.7, 27.4, 21.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.2.

HRMS (ESI) Calcd for $C_{25}H_{28}NO_2P$ $[M+H]^+$: 406.1930, found: 406.1929.



N-(4-(bis(4-methoxyphenyl)phosphoryl)phenyl)pivalamide (3z)

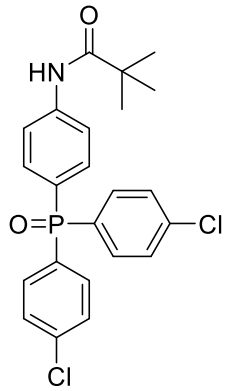
41.9 mg, yield: 48%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.0 (s, 1H), 7.7 (dd, *J* = 8.6, 2.2 Hz, 2H), 7.5 (dd, *J* = 11.4, 8.7 Hz, 6H), 6.9 (dd, *J* = 8.7, 2.0 Hz, 4H), 3.8 (s, 6H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.2, 162.3 (d, *J* = 2.8 Hz), 141.5 (d, *J* = 2.9 Hz), 133.8 (d, *J* = 11.3 Hz), 132.9 (d, *J* = 10.7 Hz), 127.7 (d, *J* = 108.7 Hz), 124.0 (d, *J* = 111.2 Hz), 119.5 (d, *J* = 12.4 Hz), 114.0 (d, *J* = 13.2 Hz), 55.3, 39.7, 27.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 28.7.

HRMS (ESI) Calcd for $C_{25}H_{28}NO_4P$ $[M+H]^+$: 438.1829, found: 438.1825.



N-(4-(bis(4-chlorophenyl)phosphoryl)phenyl)pivalamide (3aa)

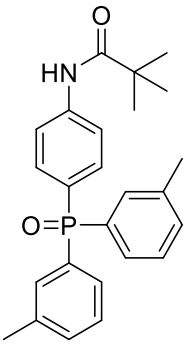
16.0 mg, yield: 18%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 (dd, *J* = 8.6, 2.4 Hz, 2H), 7.6 (s, 1H), 7.6 (dd, *J* = 11.5, 8.3 Hz, 6H), 7.4 (dd, *J* = 8.4, 2.2 Hz, 4H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.0, 141.9 (d, *J* = 2.9 Hz), 138.9 (d, *J* = 3.3 Hz), 133.3 (d, *J* = 10.9 Hz), 133.0 (d, *J* = 10.8 Hz), 130.7 (d, *J* = 106.2 Hz), 129.0 (d, *J* = 12.8 Hz), 126.1 (d, *J* = 110.1 Hz), 119.6 (d, *J* = 12.7 Hz), 39.8, 27.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 27.5.

HRMS (ESI) Calcd for $C_{23}H_{22}Cl_2NO_2P$ $[M+H]^+$: 446.0838, found: 446.0837.



N-(4-(di-m-tolylphosphoryl)phenyl)pivalamide (3ab)

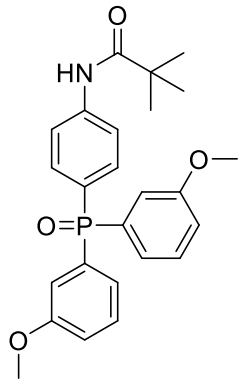
52.7 mg, yield: 65%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.2 (s, 1H), 7.7 (d, *J* = 8.3 Hz, 2H), 7.5 (d, *J* = 12.0 Hz, 4H), 7.4 – 7.3 (m, 6H), 2.3 (s, 6H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.3, 141.8, 138.3 (d, *J* = 12.0 Hz), 132.9 (d, *J* = 10.7 Hz), 132.6 (d, *J* = 2.7 Hz), 132.3 (d, *J* = 105.3 Hz), 132.3 (d, *J* = 9.6 Hz), 129.1 (d, *J* = 10.3 Hz), 128.2 (d, *J* = 12.9 Hz), 126.9 (d, *J* = 95.3 Hz), 119.6 (d, *J* = 12.5 Hz), 39.7, 27.4, 21.3.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.3.

HRMS (ESI) Calcd for $C_{25}H_{28}NO_2P$ $[M+H]^+$: 406.1930, found: 406.1928.



N-(4-(bis(3-methoxyphenyl)phosphoryl)phenyl)pivalamide (3ac)

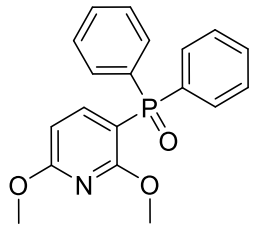
40.2 mg, yield: 46%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.8 (s, 1H), 7.7 (dd, *J* = 8.6, 2.3 Hz, 2H), 7.6 (dd, *J* = 11.5, 8.6 Hz, 2H), 7.3 (td, *J* = 7.9, 3.8 Hz, 2H), 7.3 – 7.2 (m, 2H), 7.1 – 7.0 (m, 4H), 3.8 (s, 6H), 1.3 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.1, 159.5 (d, *J* = 14.9 Hz), 141.7 (d, *J* = 3.0 Hz), 133.7 (d, *J* = 103.9 Hz), 133.0 (d, *J* = 10.7 Hz), 129.6 (d, *J* = 14.5 Hz), 126.8 (d, *J* = 108.3 Hz), 124.3 (d, *J* = 10.1 Hz), 119.4 (d, *J* = 12.6 Hz), 118.2 (d, *J* = 2.6 Hz), 116.6 (d, *J* = 10.8 Hz), 55.4, 39.8, 27.5.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.3.

HRMS (ESI) Calcd for $C_{25}H_{28}NO_4P$ $[M+H]^+$: 438.1829, found: 438.1826.



(2,6-dimethoxypyridin-3-yl)diphenylphosphine oxide (3ad)

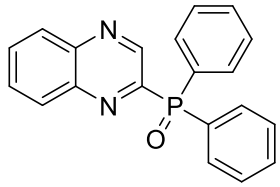
46.1 mg, yield: 68%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.9 (dd, *J* = 12.0, 8.2 Hz, 1H), 7.7 – 7.6 (m, 4H), 7.5 – 7.4 (m, 6H), 6.4 (d, *J* = 8.2 Hz, 1H), 3.9 (s, 3H), 3.8 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.4 (d, *J* = 1.5 Hz), 163.6 (d, *J* = 6.2 Hz), 146.6 (d, *J* = 8.2 Hz), 132.8 (d, *J* = 108.6 Hz), 131.7 (d, *J* = 10.3 Hz), 131.5 (d, *J* = 2.8 Hz), 128.1 (d, *J* = 12.5 Hz), 104.2 (d, *J* = 111.0 Hz), 102.0 (d, *J* = 9.4 Hz), 53.8, 53.3.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 25.9.

HRMS (ESI) Calcd for $C_{19}H_{18}NO_3P$ $[M+H]^+$: 340.1097, found: 340.1095.



diphenyl(quinoxalin-2-yl)phosphine oxide (3ae)

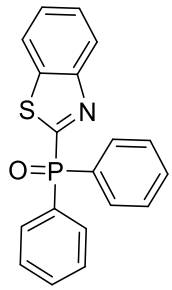
45.6 mg, yield: 69%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.7 (s, 1H), 8.2 – 8.1 (m, 2H), 8.0 – 7.9 (m, 4H), 7.8 (dt, *J* = 15.6, 7.0 Hz, 2H), 7.6 – 7.5 (m, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 152.2 (d, *J* = 124.1 Hz), 146.4 (d, *J* = 22.1 Hz), 142.6 (d, *J* = 2.3 Hz), 142.1 (d, *J* = 17.1 Hz), 132.2 (d, *J* = 2.8 Hz), 132.1 (d, *J* = 9.6 Hz), 131.3 (d, *J* = 123.7 Hz), 130.9，130.1, 129.6 (d, *J* = 1.8 Hz), 128.5 (d, *J* = 12.3 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 20.4.

HRMS (ESI) Calcd for $C_{20}H_{15}N_2OP$ $[M+H]^+$: 331.0995, found: 331.0994.



benzo[d]thiazol-2-yldiphenylphosphine oxide (3af)

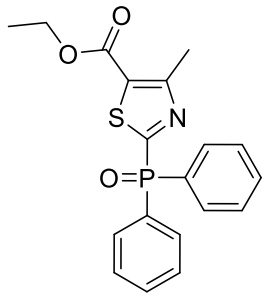
45.6 mg, yield: 68%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.2 (d, *J* = 7.9 Hz, 1H), 8.1 – 7.9 (m, 5H), 7.6 – 7.5 (m, 8H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.7 (d, *J* = 126.9 Hz), 155.3 (d, *J* = 21.6 Hz), 136.8, 132.6 (d, *J* = 2.9 Hz), 131.9 (d, *J* = 10.2 Hz), 130.9 (d, *J* = 109.0 Hz), 128.6 (d, *J* = 12.8 Hz), 126.6 (d, *J* = 5.4 Hz), 124.7, 122.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 20.1.

HRMS (ESI) Calcd for $C_{19}H_{14}NOPS$ $[M+H]^+$: 336.0606, found: 336.0606.



ethyl 2-(diphenylphosphoryl)-4-methylthiazole-5-carboxylate (3ag)

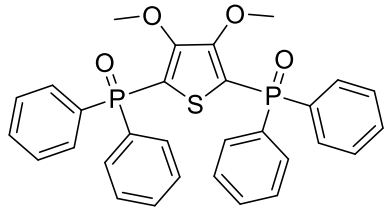
54.9 mg, yield: 74%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.9 (dd, *J* = 12.6, 7.6 Hz, 4H), 7.6 – 7.5 (m, 6H), 4.3 (q, *J* = 7.1 Hz, 2H), 2.8 (s, 3H), 1.4 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.5 (d, *J* = 125.7 Hz), 162.9 (d, *J* = 19.0 Hz), 161.6, 132.6 (d, *J* = 2.9 Hz), 131.7 (d, *J* = 10.3 Hz), 130.7 (d, *J* = 109.4 Hz), 128.6 (d, *J* = 12.8 Hz), 127.3, 61.6, 17.5, 14.1.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 18.7.

HRMS (ESI) Calcd for $C_{19}H_{18}NO_3PS$ $[M+H]^+$: 372.0818, found: 372.0815.



(3,4-dimethoxythiophene-2,5-diyl)bis(diphenylphosphine oxide) (3ah)

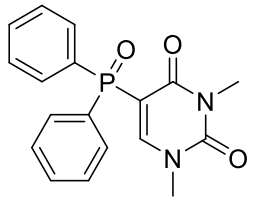
105.6 mg, yield: 97%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.8 – 7.7 (m, 8H), 7.6 – 7.4 (m, 12H), 3.5 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.8 (d, *J* = 15.9 Hz), 132.3, 131.6 (d, *J* = 11.2 Hz), 131.5 (d, *J* = 111.5 Hz), 128.4 (d, *J* = 13.3 Hz), 123.9 (d, *J* = 104.8 Hz), 60.2.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 20.5.

HRMS (ESI) Calcd for $C_{30}H_{26}O_4P_2S$ $[M+H]^+$: 545.1100, found: 545.1098.



5-(diphenylphosphoryl)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3ai)

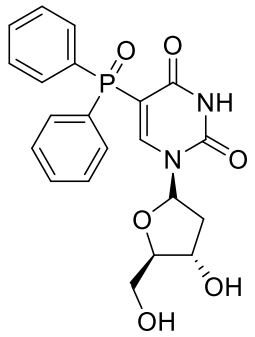
57.8 mg, yield: 85%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.3 (d, *J* = 10.7 Hz, 1H), 7.9 – 7.8 (m, 4H), 7.6 – 7.5 (m, 2H), 7.5 (td, *J* = 7.5, 3.0 Hz, 4H), 3.5 (s, 3H), 3.3 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.4 (d, *J* = 10.4 Hz), 152.0 (d, *J* = 11.3 Hz), 151.4, 132.2 (d, *J* = 2.9 Hz), 131.7 (d, *J* = 10.9 Hz), 131.3 (d, *J* = 111.7 Hz), 128.3 (d, *J* = 13.0 Hz), 104.0 (d, *J* = 114.6 Hz), 37.6, 27.9.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 23.7.

HRMS (ESI) Calcd for $C_{18}H_{17}N_2O_3P$ $[M+H]^+$: 341.1050, found: 341.1046.



5-(diphenylphosphoryl)-1-((2S,4S,5R)-4-hydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl)pyrimidine-2,4(1H,3H)-dione (3aj)

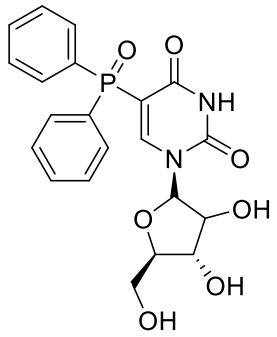
59.1 mg, yield: 69%. White solid.

^{1}H NMR (400 MHz, Methanol-d_4) δ 8.4 (d, J = 11.6 Hz, 1H), 7.9 – 7.7 (m, 4H), 7.6 – 7.5 (m, 6H), 6.2 (t, J = 6.5 Hz, 1H), 4.3 (dt, J = 6.3, 3.2 Hz, 1H), 4.0 (q, J = 3.7 Hz, 1H), 3.7 – 3.5 (m, 2H), 2.4 – 2.2 (m, 2H), 1.3 (s, 1H).

^{13}C NMR (101 MHz, Methanol-d_4) δ 164.8 (d, J = 11.4 Hz), 153.0, 151.7 (d, J = 14.3 Hz), 134.7 (d, J = 1.6 Hz), 133.9 (d, J = 10.8 Hz), 133.1 (d, J = 112.1 Hz), 130.7 (d, J = 12.9 Hz), 105.9 (d, J = 120.0 Hz), 90.5, 89.0, 73.5, 63.9, 42.8.

^{31}P NMR (162 MHz, Methanol-d_4) δ 27.9.

HRMS (ESI) Calcd for $C_{21}H_{21}N_2O_6P$ $[M+H]^+$: 429.1210, found: 429.1208.



1-((2S,4S,5R)-3,4-dihydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl)-5-(diphenylphosphoryl)pyrimidine-2,4(1H,3H)-dione (3k)

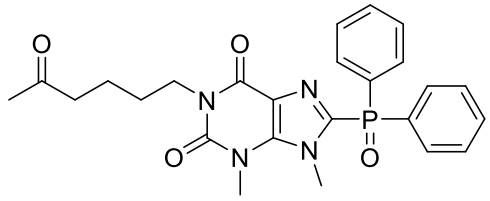
35.5 mg, yield: 40%. White solid.

^{1}H NMR (400 MHz, Methanol-d_4) δ 8.0 (d, J = 11.8 Hz, 1H), 7.7 – 7.6 (m, 4H), 7.5 – 7.4 (m, 6H), 6.1 (d, J = 3.6 Hz, 1H), 4.0 (dd, J = 3.6, 1.9 Hz, 1H), 3.9 (t, J = 2.2 Hz, 1H), 3.8 (td, J = 5.3, 2.5 Hz, 1H), 3.5 – 3.4 (m, 2H).

^{13}C NMR (101 MHz, Methanol-d_4) δ 164.6 (d, J = 10.1 Hz), 153.5 (d, J = 15.3 Hz), 152.7, 134.7 (d, J = 2.8 Hz), 133.9 (d, J = 10.7 Hz), 132.9 (d, J = 112.5 Hz), 130.8 (d, J = 12.5 Hz), 104.6 (d, J = 121.1 Hz), 89.4, 88.4, 79.2, 77.6, 63.8.

^{31}P NMR (162 MHz, Methanol-d_4) δ 27.9.

HRMS (ESI) Calcd for $C_{21}H_{21}N_2O_7P$ $[M+H]^+$: 445.1159, found: 445.1159.



8-(diphenylphosphoryl)-3,9-dimethyl-1-(5-oxohexyl)-3,9-dihydro-1H-purine-2,6-dione (3l)

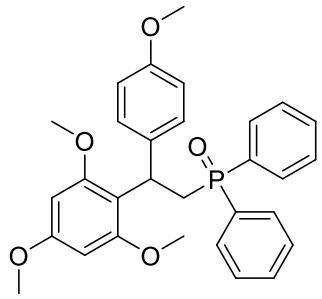
43.1 mg, yield: 45%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.9 – 7.8 (m, 4H), 7.6 (td, *J* = 7.3, 1.5 Hz, 2H), 7.5 – 7.5 (m, 4H), 4.3 (d, *J* = 0.9 Hz, 3H), 4.0 (t, *J* = 6.7 Hz, 2H), 3.5 (s, 3H), 2.5 (t, *J* = 6.9 Hz, 2H), 2.1 (s, 3H), 1.7 – 1.6 (m, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.7, 155.2, 151.2, 147.8 (d, *J* = 17.9 Hz), 145.0 (d, *J* = 135.6 Hz), 132.7 (d, *J* = 2.9 Hz), 131.8 (d, *J* = 10.4 Hz), 130.7 (d, *J* = 111.9 Hz), 128.6 (d, *J* = 13.1 Hz), 110.4 (d, *J* = 4.3 Hz), 43.1, 40.9, 34.3, 29.9, 29.8, 27.3, 20.9.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 19.2.

HRMS (ESI) Calcd for $C_{25}H_{27}N_4O_4P$ $[M+H]^+$: 479.1843, found: 479.1840.



(2-(4-methoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5a)

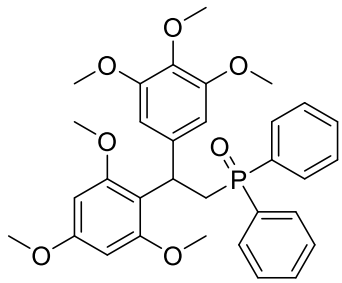
42.2 mg, yield: 42%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.7 (m, 2H), 7.5 – 7.3 (m, 7H), 7.3 – 7.2 (m, 3H), 6.8 – 6.6 (m, 2H), 5.8 (s, 2H), 5.3 – 5.2 (m, 1H), 3.7 (s, 3H), 3.7 (s, 3H), 3.6 (s, 6H), 3.6 – 3.5 (m, 1H), 3.0 (td, *J* = 14.7, 4.7 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.8, 158.6, 157.4, 137.0 (d, *J* = 11.6 Hz), 134.7 (d, *J* = 97.5 Hz), 132.9 (d, *J* = 97.7 Hz), 131.1 (d, *J* = 2.9 Hz), 130.7, 130.6, 130.6 (d, *J* = 3.2 Hz), 130.5, 128.6, 128.3 (d, *J* = 11.4 Hz), 127.6 (d, *J* = 11.7 Hz), 113.1, 90.8, 55.4, 55.1, 55.1, 33.3 (d, *J* = 71.3 Hz), 31.9 (d, *J* = 2.5 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.5.

HRMS (ESI) Calcd for $C_{30}H_{31}O_5P$ $[M+H]^+$: 503.1982, found: 503.1982.



diphenyl(2-(2,4,6-trimethoxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethyl)phosphine oxide (5b)

53.9 mg, yield: 48%. White solid.

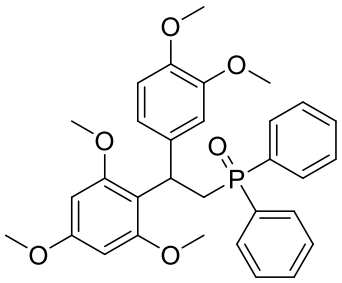
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.6 (m, 2H), 7.5 – 7.3 (m, 6H), 7.3 – 7.2 (m, 2H), 6.6 (s, 2H),

5.9 (s, 2H), 5.3 – 5.2 (m, 1H), 3.8 – 3.7 (m, 18H), 3.6 – 3.4 (m, 1H), 3.1 – 3.0 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.9, 158.6, 152.4, 140.4 (d, *J* = 10.6 Hz), 135.8, 134.1 (d, *J* = 97.6 Hz), 132.8 (d, *J* = 98.2 Hz), 131.2 (d, *J* = 2.6 Hz), 130.8 (d, *J* = 2.8 Hz), 130.6 (d, *J* = 19.1 Hz), 130.6, 128.2 (d, *J* = 11.4 Hz), 127.7 (d, *J* = 11.7 Hz), 111.3 (d, *J* = 4.8 Hz), 105.0, 90.8, 60.6, 55.8, 55.4, 55.1, 34.0 (d, *J* = 70.7 Hz), 33.3 (d, *J* = 2.5 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 31.0.

HRMS (ESI) Calcd for $C_{32}H_{35}O_7P$ $[M+H]^+$: 563.2193, found: 563.2194.



(2-(3,4-dimethoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5c)

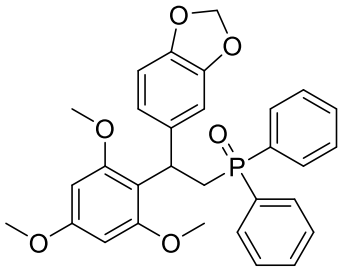
49.9 mg, yield: 47%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.6 (m, 2H), 7.5 – 7.3 (m, 6H), 7.3 – 7.2 (m, 2H), 6.9 (d, *J* = 1.9 Hz, 1H), 6.9 (dd, *J* = 8.3, 1.8 Hz, 1H), 6.7 (d, *J* = 8.3 Hz, 1H), 5.8 (s, 2H), 5.4 – 5.2 (m, 1H), 3.8 – 3.6 (m, 15H), 3.6 – 3.5 (m, 1H), 3.0 (td, *J* = 14.6, 5.0 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.8, 158.5, 148.1, 146.8, 137.4 (d, *J* = 11.2 Hz), 134.6 (d, *J* = 97.2 Hz), 132.9 (d, *J* = 97.7 Hz), 131.1 (d, *J* = 2.6 Hz), 130.6 (d, *J* = 9.2 Hz), 130.5 (d, *J* = 9.0 Hz), 128.2 (d, *J* = 11.4 Hz), 127.6 (d, *J* = 11.7 Hz), 119.6, 111.7 (d, *J* = 4.4 Hz), 111.5, 110.5, 90.8, 55.7, 55.6, 55.4, 55.1, 33.7 (d, *J* = 70.9 Hz), 32.4 (d, *J* = 2.5 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.5.

HRMS (ESI) Calcd for $C_{31}H_{33}O_6P$ $[M+H]^+$: 533.2088, found: 533.2086.



(2-(benzo[d][1,3]dioxol-5-yl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5d)

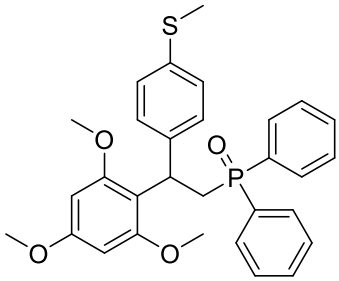
41.3 mg, yield: 40%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.6 (m, 2H), 7.5 – 7.3 (m, 6H), 7.2 (dd, *J* = 7.2, 2.8 Hz, 2H), 6.9 – 6.8 (m, 2H), 6.6 (d, *J* = 8.1 Hz, 1H), 5.8 (s, 4H), 5.3 – 5.2 (m, 1H), 3.7 (d, *J* = 21.2 Hz, 9H), 3.5 (ddd, *J* = 15.0, 9.8, 8.0 Hz, 1H), 3.0 (td, *J* = 14.6, 5.1 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.8, 158.5, 146.9, 145.2, 138.8 (d, *J* = 11.3 Hz), 134.4 (d, *J* = 97.3 Hz), 132.9 (d, *J* = 97.9 Hz), 131.1 (d, *J* = 2.6 Hz), 130.6 (d, *J* = 9.9 Hz), 130.6 (d, *J* = 5.5 Hz), 130.5, 128.3 (d, *J* = 11.4 Hz), 127.7 (d, *J* = 11.7 Hz), 120.7, 111.7 (d, *J* = 4.4 Hz), 108.5, 107.5, 100.5, 90.7, 55.4, 55.1, 33.3 (d, *J* = 71.2 Hz), 32.5 (d, *J* = 2.5 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.5.

HRMS (ESI) Calcd for $C_{30}H_{29}O_6P$ $[M+H]^+$: 517.1775, found: 517.1774.



(2-(4-(methylthio)phenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5h)

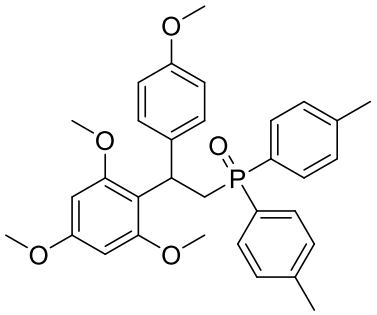
47.0 mg, yield: 43%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.6 (m, 2H), 7.5 – 7.3 (m, 6H), 7.3 – 7.2 (m, 4H), 7.1 (d, *J* = 8.4 Hz, 2H), 5.8 (s, 2H), 5.3 – 5.2 (m, 1H), 3.7 (d, *J* = 29.5 Hz, 9H), 3.6 – 3.5 (m, 1H), 3.0 (td, *J* = 14.8, 4.8 Hz, 1H), 2.4 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.9, 158.6, 142.1, 141.9, 134.7, 134.7 (d, *J* = 97.4 Hz), 132.8 (d, *J* = 97.7 Hz), 131.2 (d, *J* = 2.7 Hz), 130.7, 130.6, 130.6 (d, *J* = 2.4 Hz), 130.5 (d, *J* = 8.9 Hz), 128.3 (d, *J* = 11.4 Hz), 128.2, 127.7 (d, *J* = 11.6 Hz), 126.5, 111.5 (d, *J* = 4.2 Hz), 90.7, 55.4, 55.1, 32.9 (d, *J* = 71.5 Hz), 32.1 (d, *J* = 2.5 Hz), 16.2.

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.3.

HRMS (ESI) Calcd for $C_{32}H_{35}O_4PS$ $[M+Na]^+$: 569.1886, found: 569.1870.



(2-(4-methoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)di-p-tolylphosphine oxide (5i)

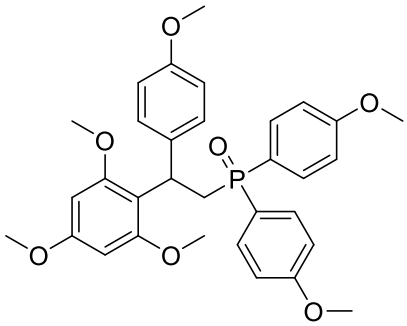
40.3 mg, yield: 38%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.6 (dd, *J* = 10.9, 8.1 Hz, 2H), 7.4 (dd, *J* = 11.2, 8.0 Hz, 2H), 7.2 (dd, *J* = 21.5, 7.4 Hz, 4H), 7.0 (d, *J* = 6.1 Hz, 2H), 6.7 (d, *J* = 8.7 Hz, 2H), 5.8 (s, 2H), 5.2 (td, *J* = 12.0, 11.3, 4.6 Hz, 1H), 3.7 – 3.6 (m, 12H), 3.5 – 3.4 (m, 1H), 3.0 (td, *J* = 14.8, 4.7 Hz, 1H), 2.3 (d, *J* = 16.0 Hz, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.7, 158.5, 157.3, 141.3 (d, *J* = 2.9 Hz), 140.7 (d, *J* = 2.9 Hz), 137.1 (d, *J* = 11.6 Hz), 131.6 (d, *J* = 99.8 Hz), 130.7 (d, *J* = 9.6 Hz), 130.5 (d, *J* = 9.4 Hz), 129.8 (d, *J* = 100.3 Hz), 128.9 (d, *J* = 11.7 Hz), 128.6, 128.3 (d, *J* = 12.0 Hz), 113.0, 90.7, 55.4, 55.1 (d, *J* = 3.2 Hz), 33.4 (d, *J* = 71.7 Hz), 31.9 (d, *J* = 2.4 Hz), 21.4 (d, *J* = 6.0 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.8.

HRMS (ESI) Calcd for $C_{32}H_{35}O_5P$ $[M+H]^+$: 531.2295, found: 531.2294.



bis(4-methoxyphenyl)(2-(4-methoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)phosphine oxide (5j)

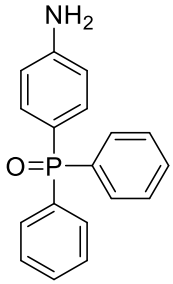
38.3 mg, yield: 34%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.6 (dd, *J* = 10.6, 8.7 Hz, 2H), 7.4 (dd, *J* = 10.9, 8.7 Hz, 2H), 7.2 (d, *J* = 8.6 Hz, 2H), 6.9 (dd, *J* = 8.7, 2.0 Hz, 2H), 6.7 – 6.7 (m, 4H), 5.8 (s, 2H), 5.2 (td, *J* = 11.3, 10.4, 4.6 Hz, 1H), 3.8 – 3.6 (m, 18H), 3.5 – 3.4 (m, 1H), 2.9 (td, *J* = 14.9, 4.7 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.7 (d, *J* = 2.8 Hz), 161.4 (d, *J* = 2.9 Hz), 159.6, 158.4, 157.3, 137.2 (d, *J* = 11.6 Hz), 132.5 (d, *J* = 10.6 Hz), 132.3 (d, *J* = 10.4 Hz), 128.6, 126.2 (d, *J* = 103.5 Hz), 124.2 (d, *J* = 103.9 Hz), 113.7 (d, *J* = 12.4 Hz), 113.1 (d, *J* = 12.6 Hz), 113.0, 112.1 (d, *J* = 4.0 Hz), 90.7, 55.4, 55.2, 55.1, 55.1, 55.0, 33.6 (d, *J* = 72.3 Hz), 31.9 (d, *J* = 2.4 Hz).

^{31}P NMR (162 MHz, Chloroform-*d*) δ 30.6.

HRMS (ESI) Calcd for $C_{32}H_{35}O_7P$ $[M+H]^+$: 563.2193, found: 563.2191.



(4-aminophenyl)diphenylphosphine oxide (6)

41.6 mg, yield: 71%. White solid.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.7 – 7.6 (m, 4H), 7.5 – 7.3 (m, 8H), 6.7 (dd, *J* = 8.6, 2.3 Hz, 2H), 4.2 (s, 2H).

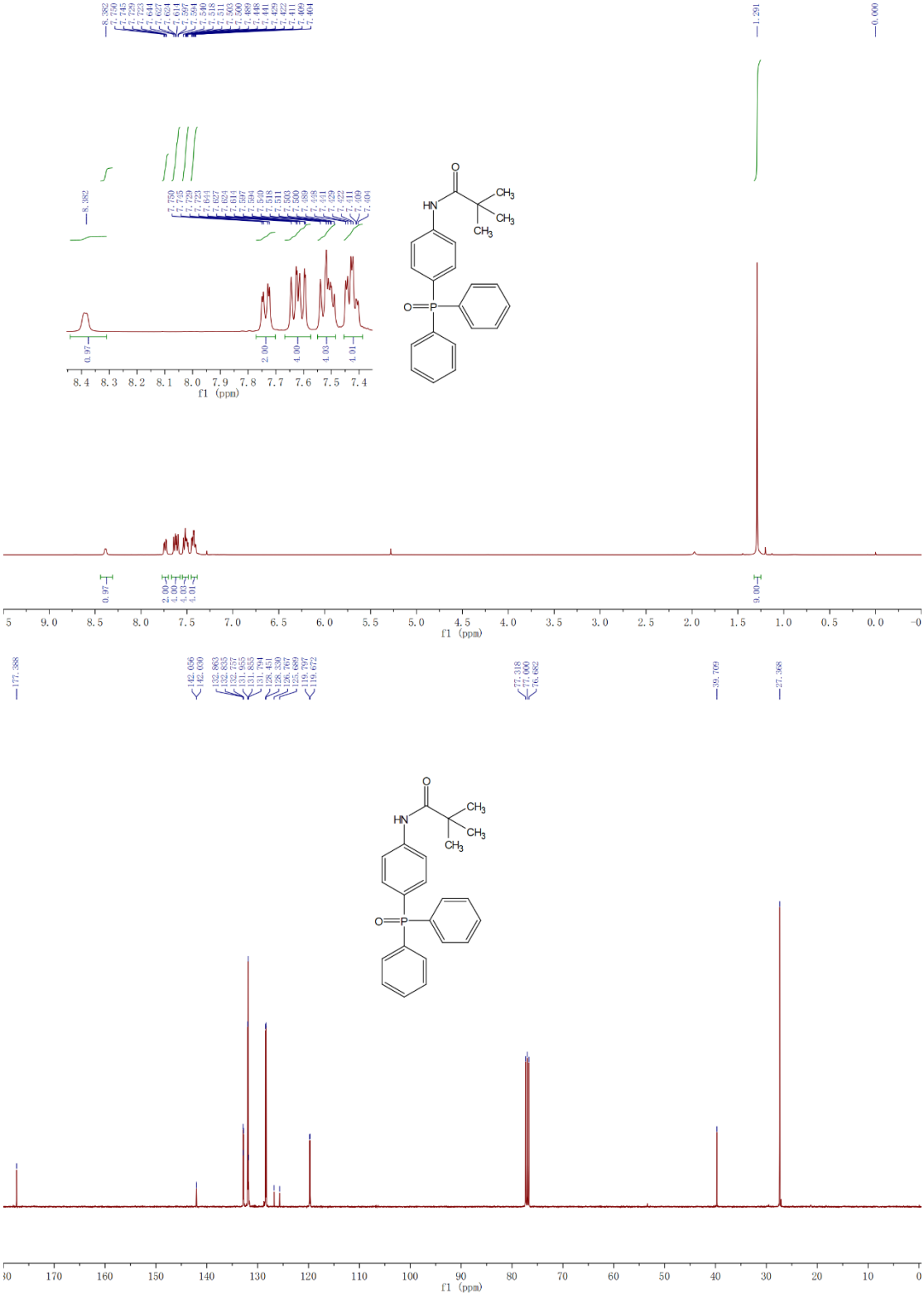
^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.0 (d, *J* = 2.7 Hz), 133.7 (d, *J* = 11.3 Hz), 133.3 (d, *J* = 104.2 Hz), 132.0 (d, *J* = 9.9 Hz), 131.5 (d, *J* = 2.7 Hz), 128.3 (d, *J* = 12.0 Hz), 119.4 (d, *J* = 113.7 Hz), 114.2 (d, *J* = 13.2 Hz).

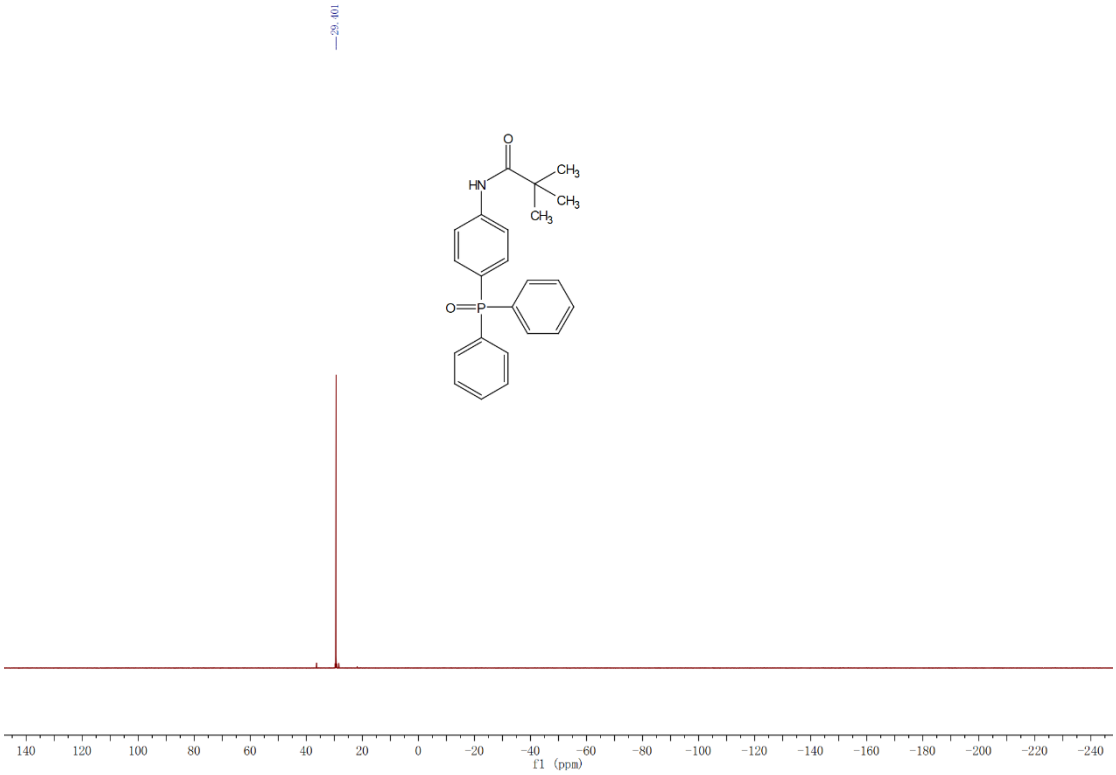
^{31}P NMR (162 MHz, Chloroform-*d*) δ 29.7.

HRMS (ESI) Calcd for $C_{18}H_{16}NOP$ $[M+H]^+$: 294.1042, found: 294.1041.

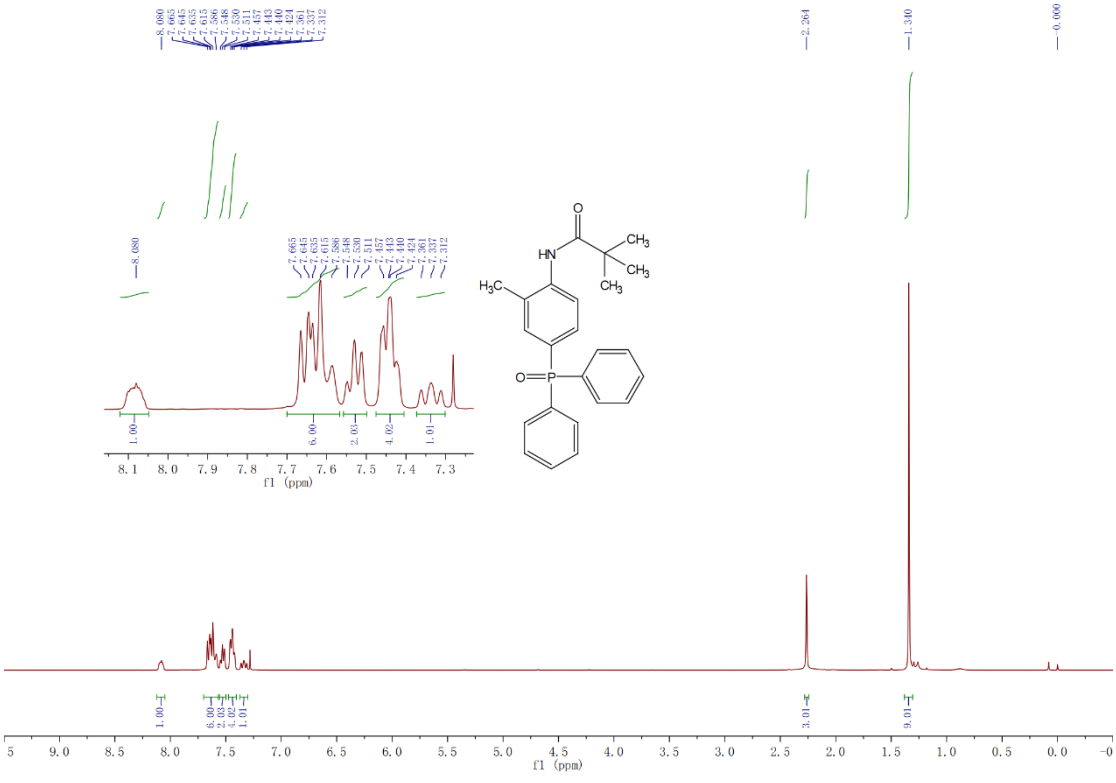
9. NMR Spectroscopic Data

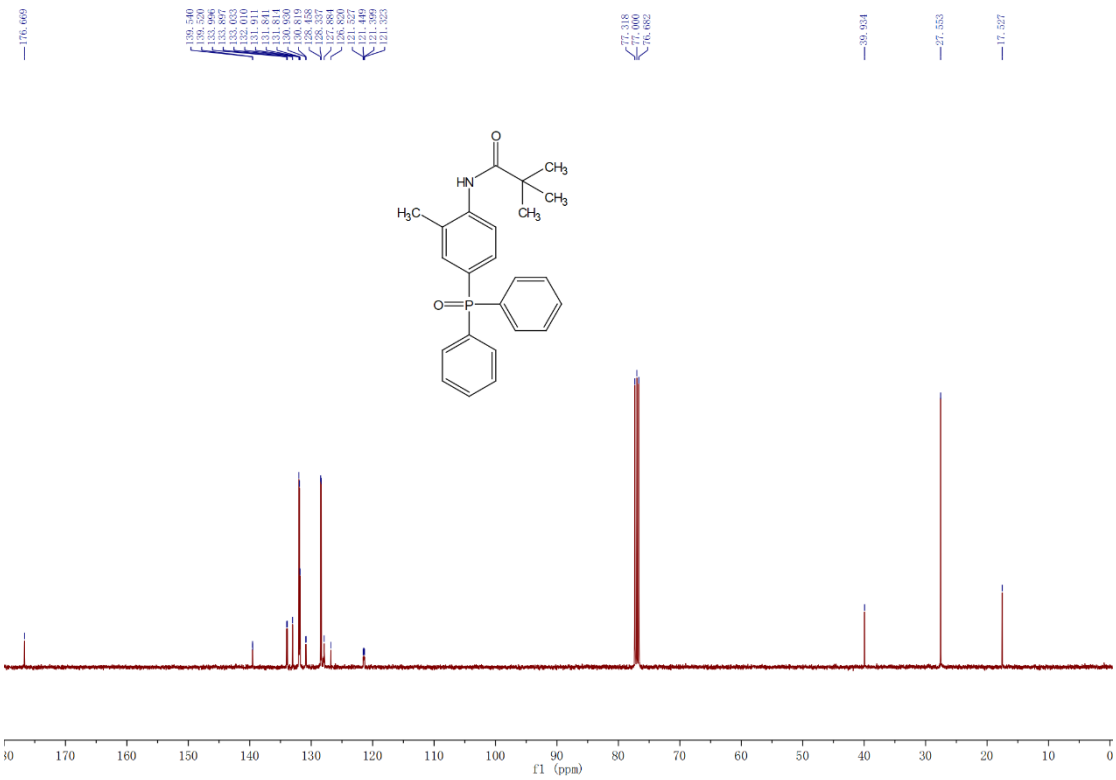
N-(4-(diphenylphosphoryl)phenyl)pivalamide (3a)

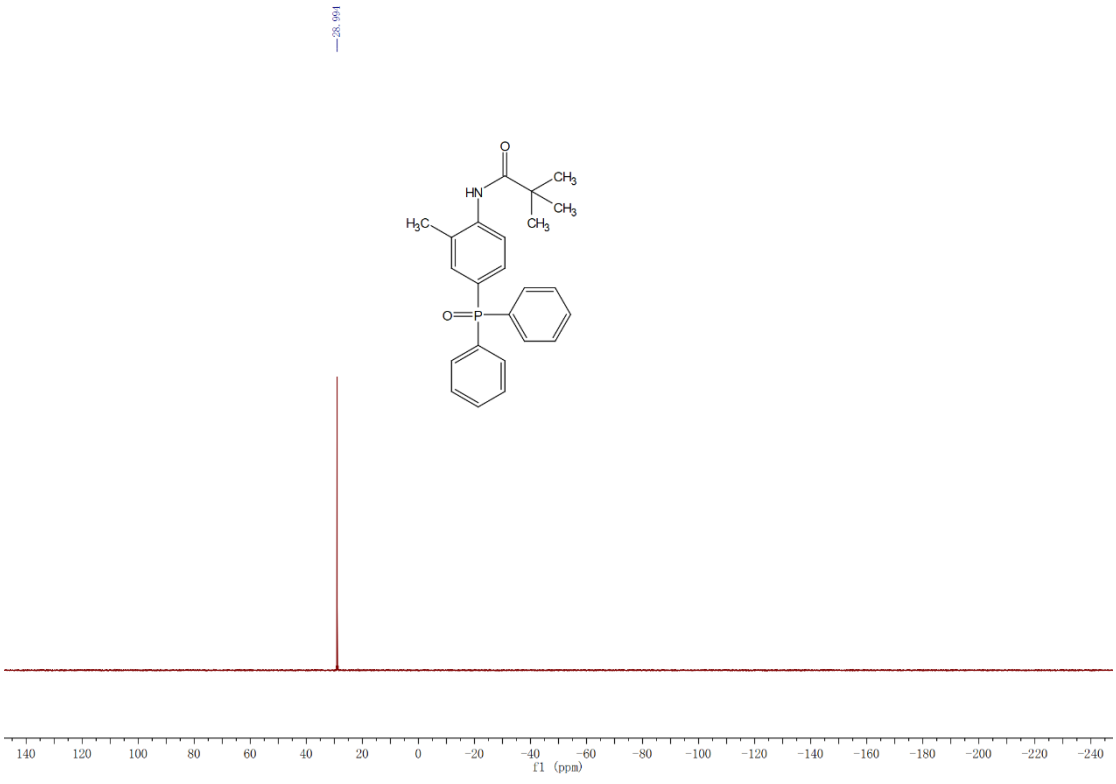




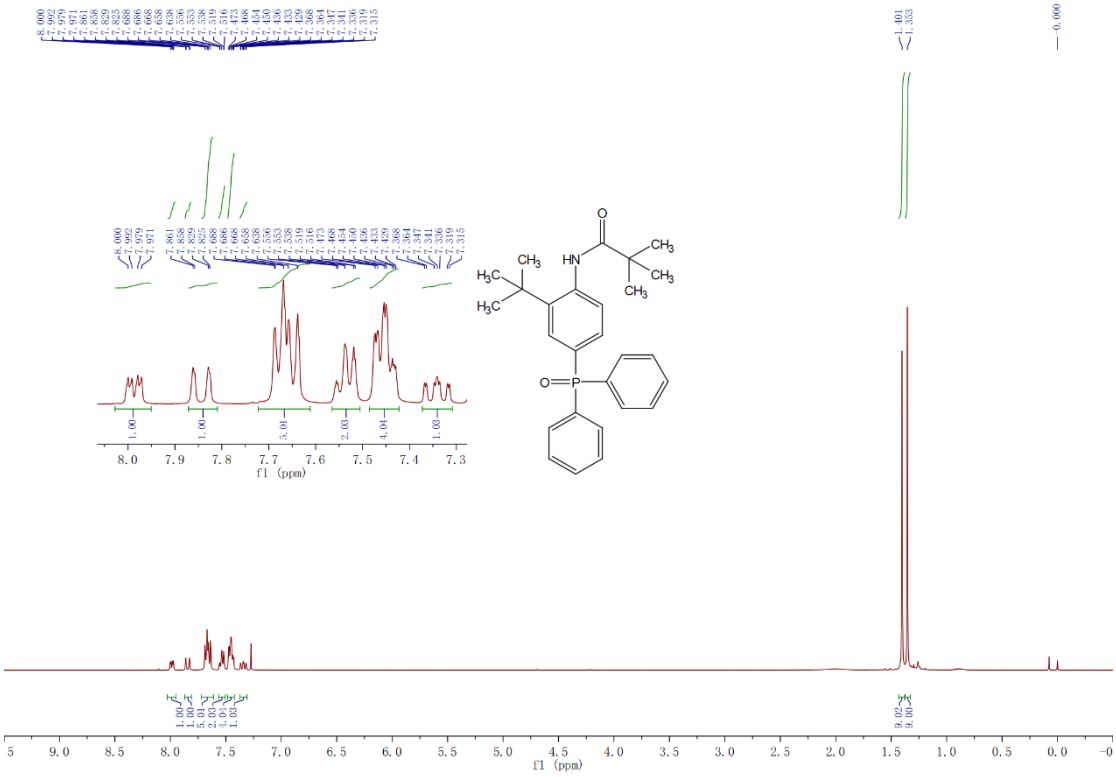
N-(4-(diphenylphosphoryl)-2-methylphenyl)pivalamide (3b)

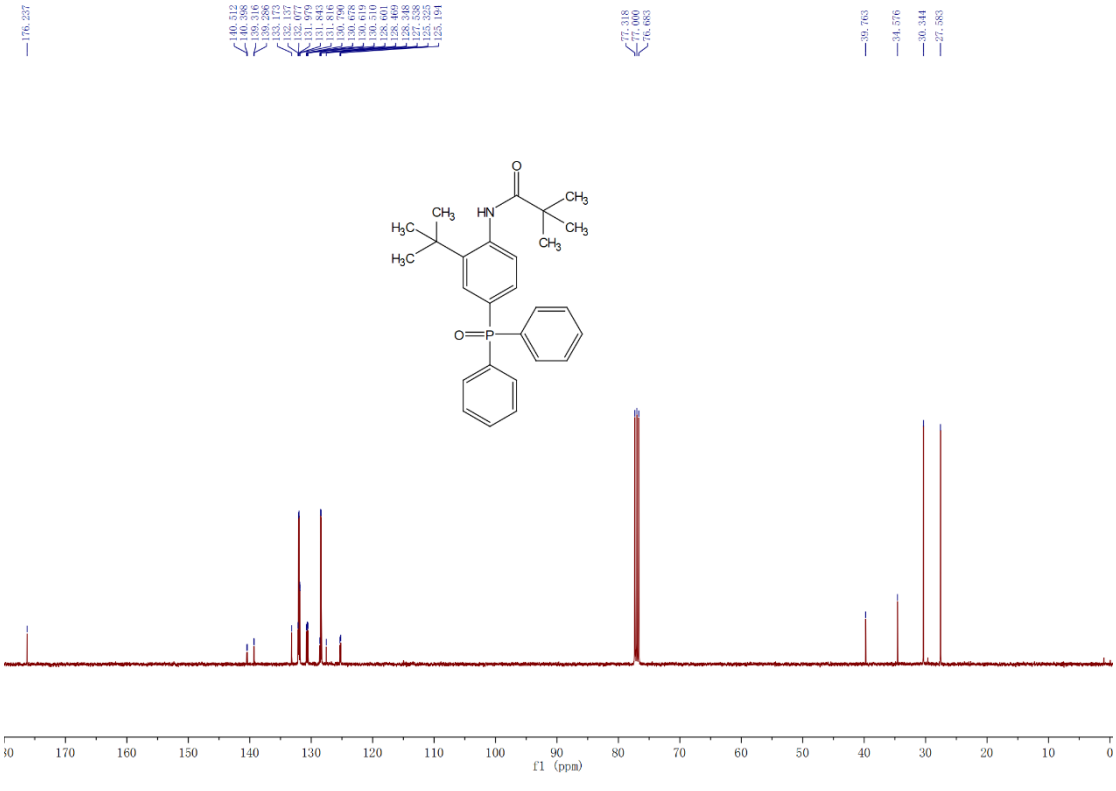


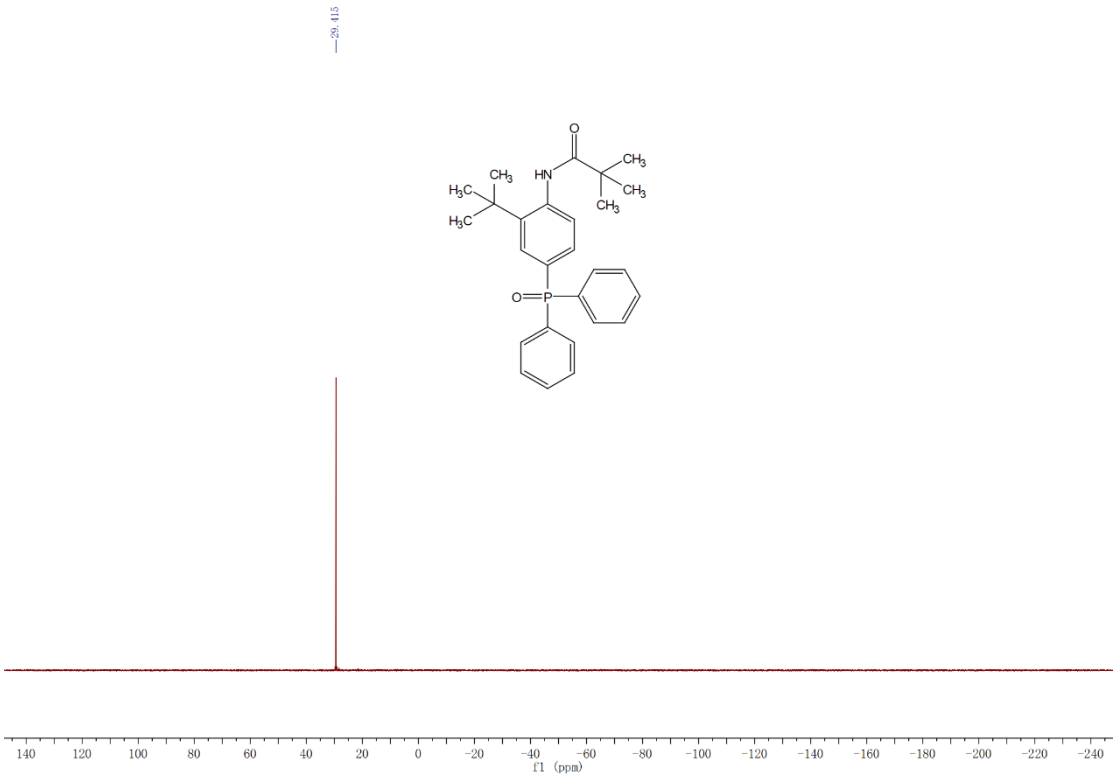

176.669
139.540
139.320
133.946
133.897
133.033
132.610
132.411
131.841
131.814
130.950
130.819
130.496
128.337
127.884
126.820
121.557
121.449
121.399
121.323
77.318
77.000
76.682
39.934
27.553
17.527
O
HN
CH3
CH3
CH3
H3C
O=P
f1 (ppm)


28.994
O
HN
CH3
CH3
CH3
H3C
O=P
f1 (ppm)

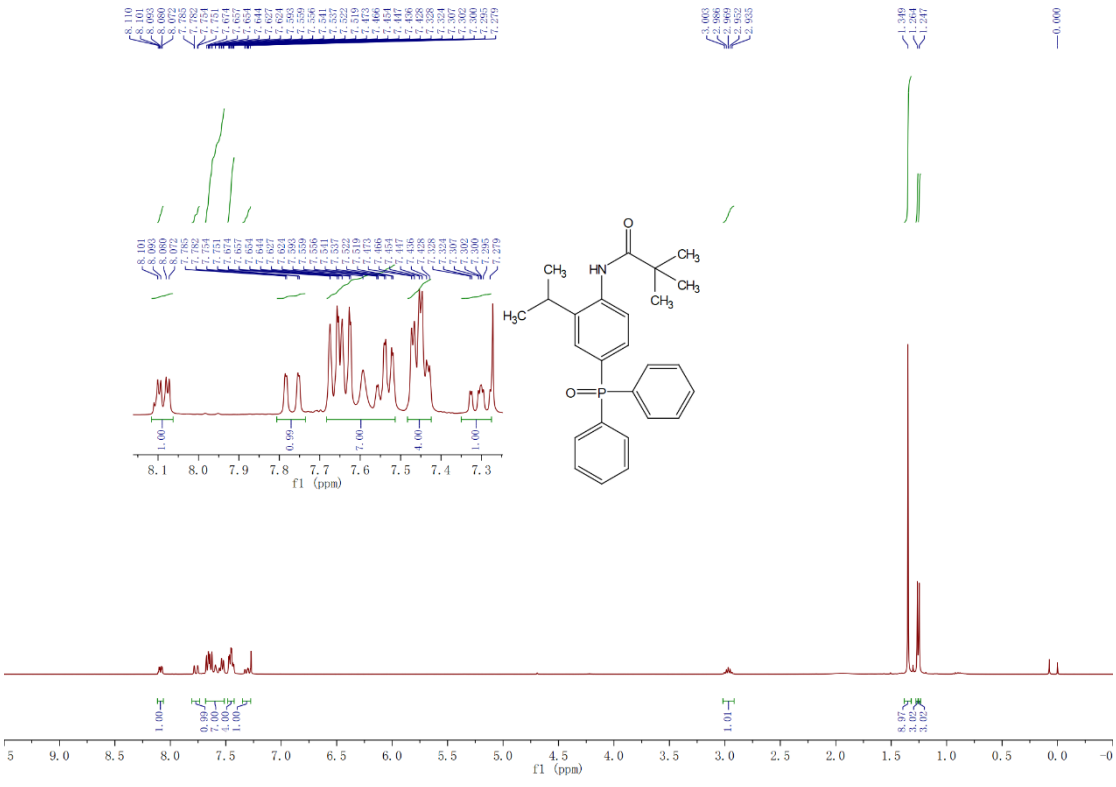
N-(2-(tert-butyl)-4-(diphenylphosphoryl)phenyl)pivalamide (3c)

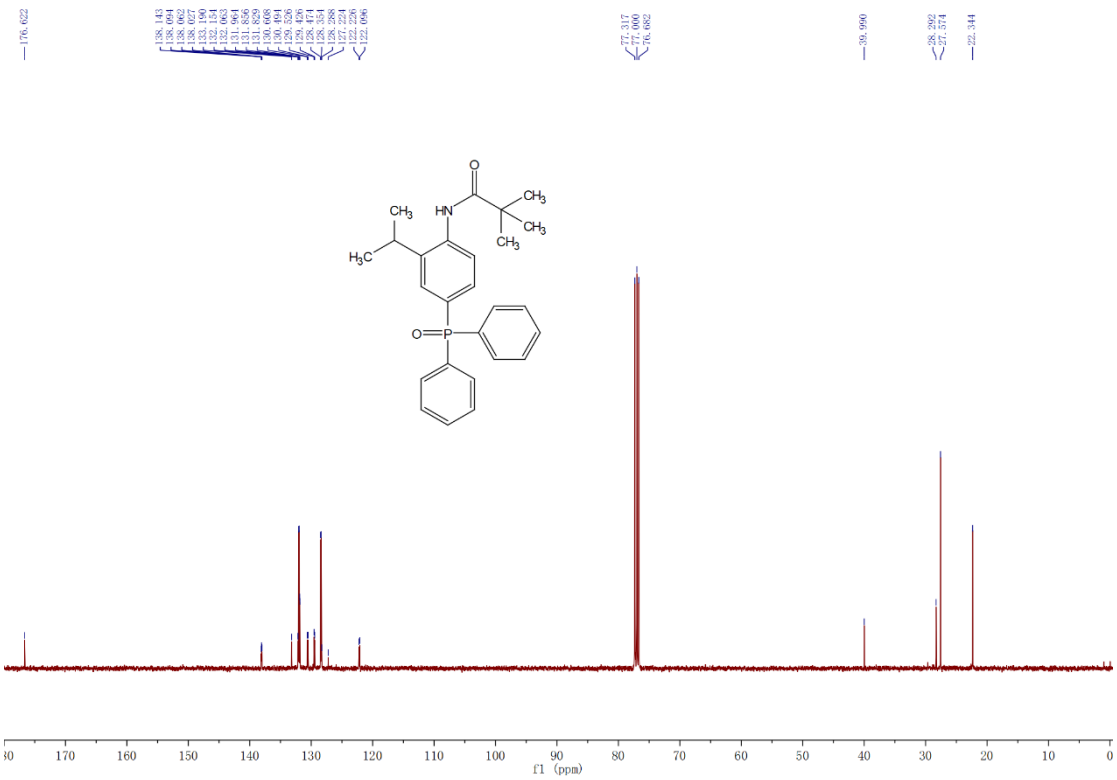


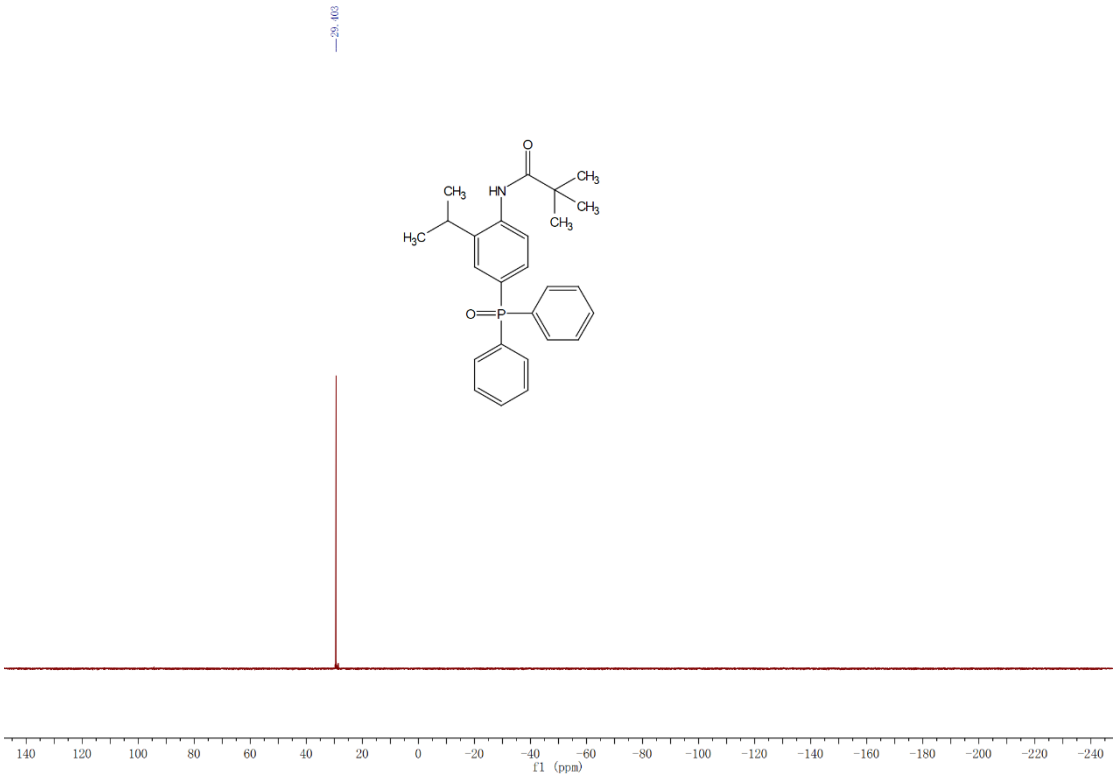




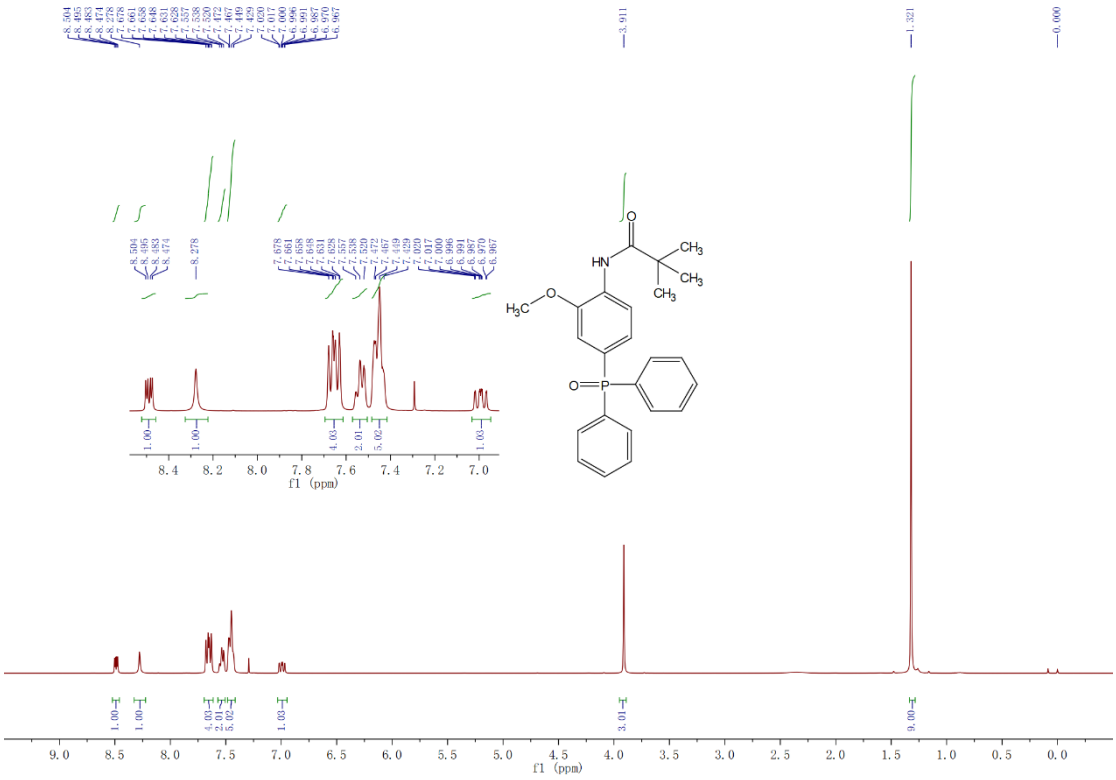
N-(4-(diphenylphosphoryl)-2-isopropylphenyl)pivalamide (3d)

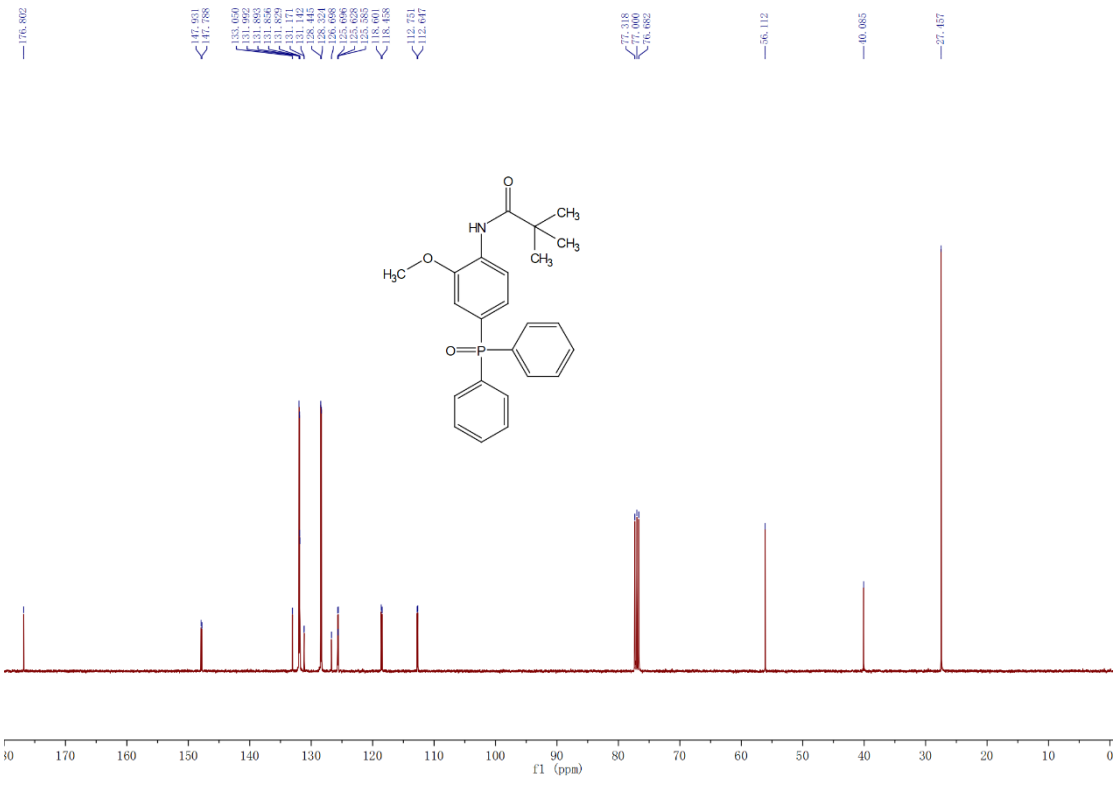


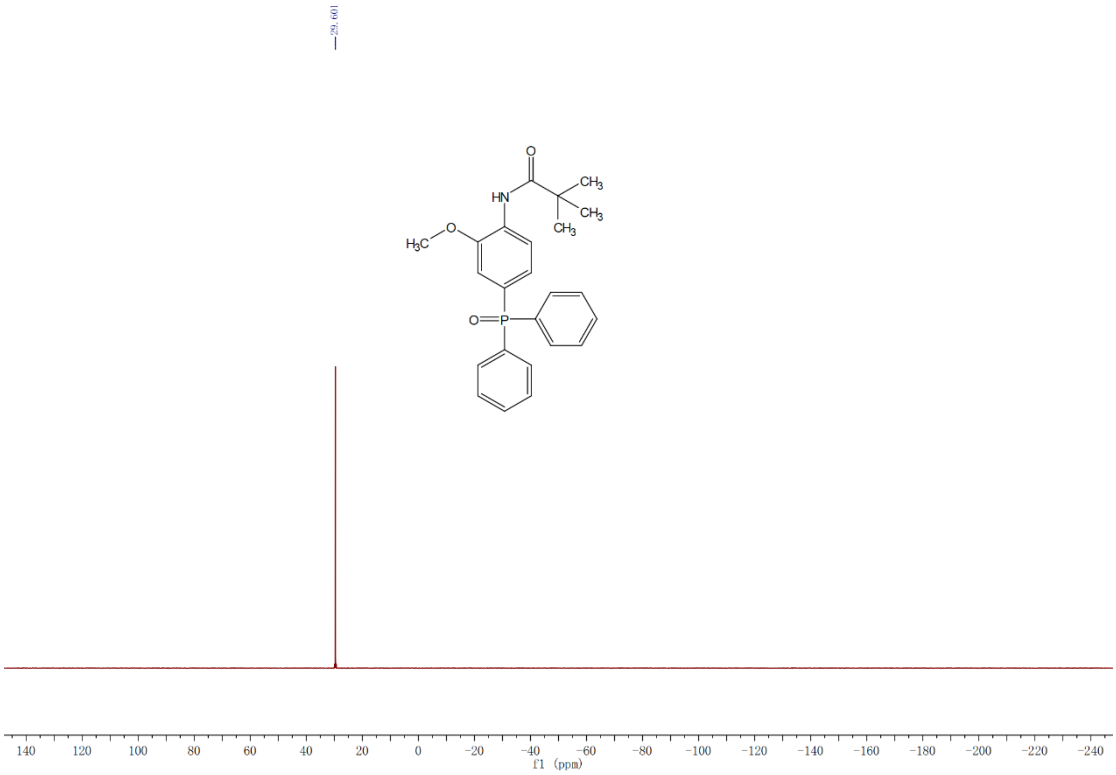

176.622
138.143
138.064
138.062
138.027
133.190
132.151
132.136
131.964
131.856
131.829
130.646
130.494
129.526
129.426
128.471
128.354
128.288
127.224
122.226
122.096
77.317
77.000
76.682
39.990
28.292
27.374
22.344
O
HN
CH3
CH3
CH3
CH3
H3C
O=P
f1 (ppm)


29.403
O
HN
CH3
CH3
CH3
CH3
H3C
O=P
f1 (ppm)

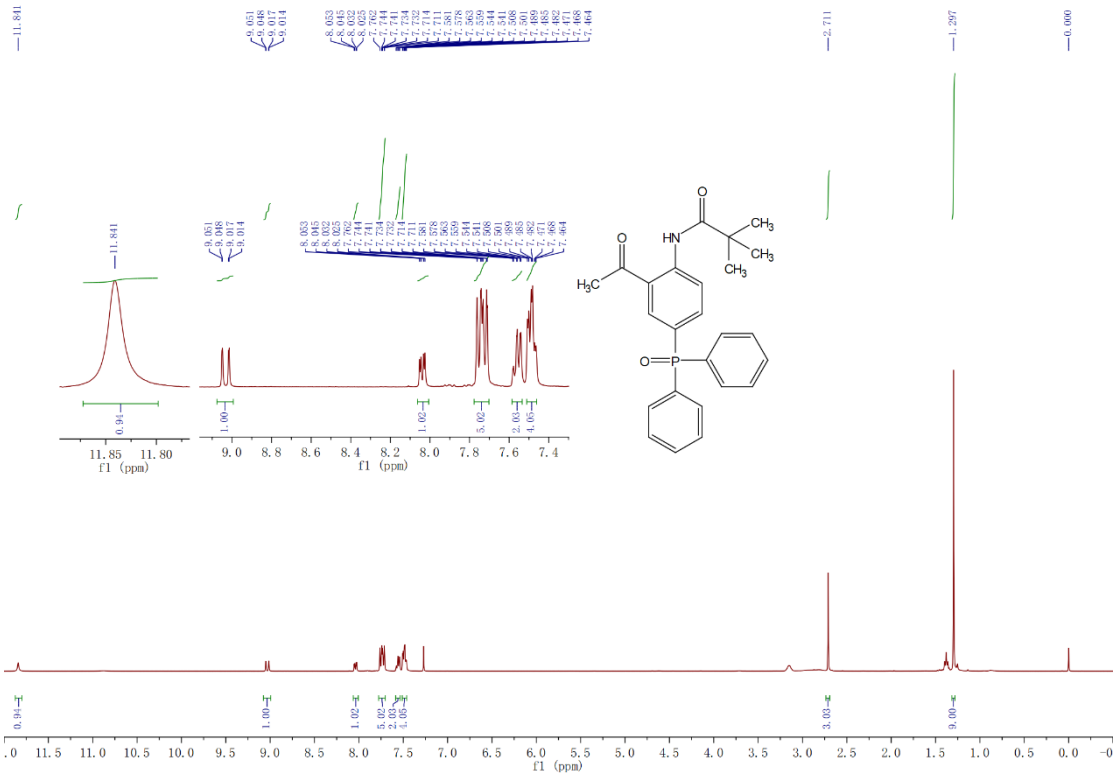
N-(4-(diphenylphosphoryl)-2-methoxyphenyl)pivalamide (3e)

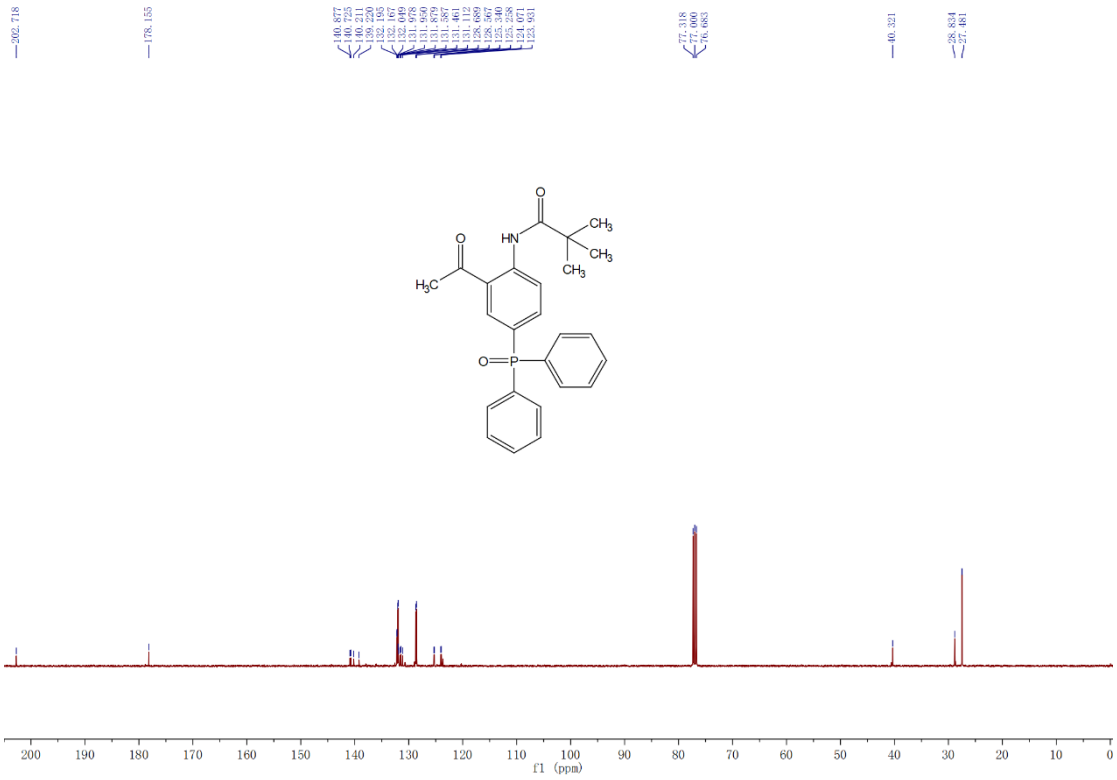


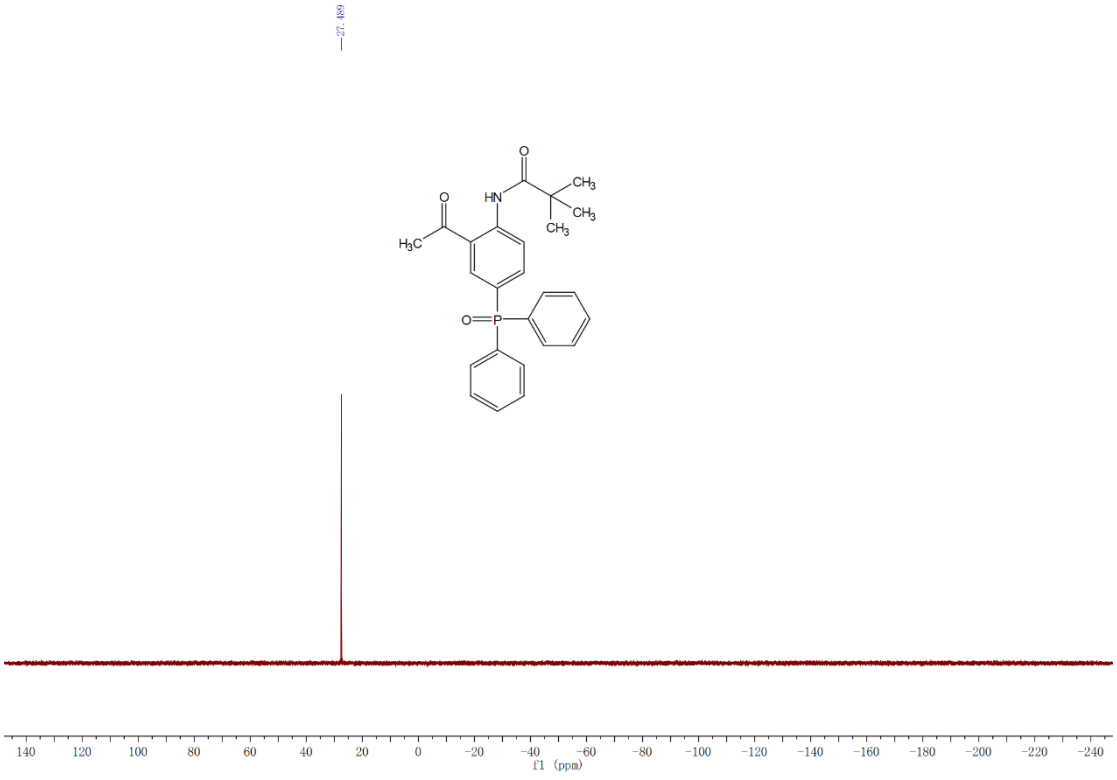




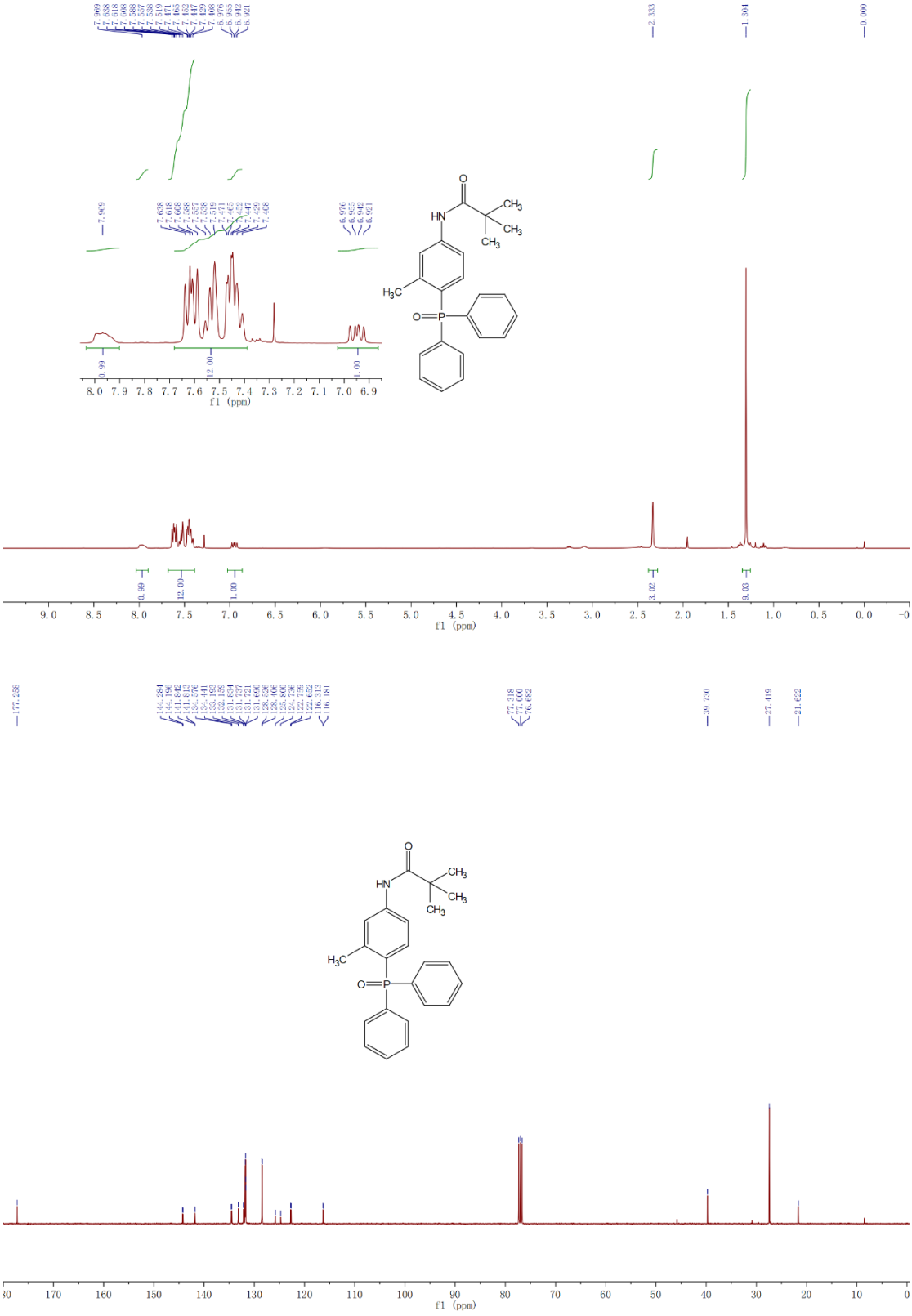
N-(4-(diphenylphosphoryl)-2-methoxyphenyl)pivalamide (3f)

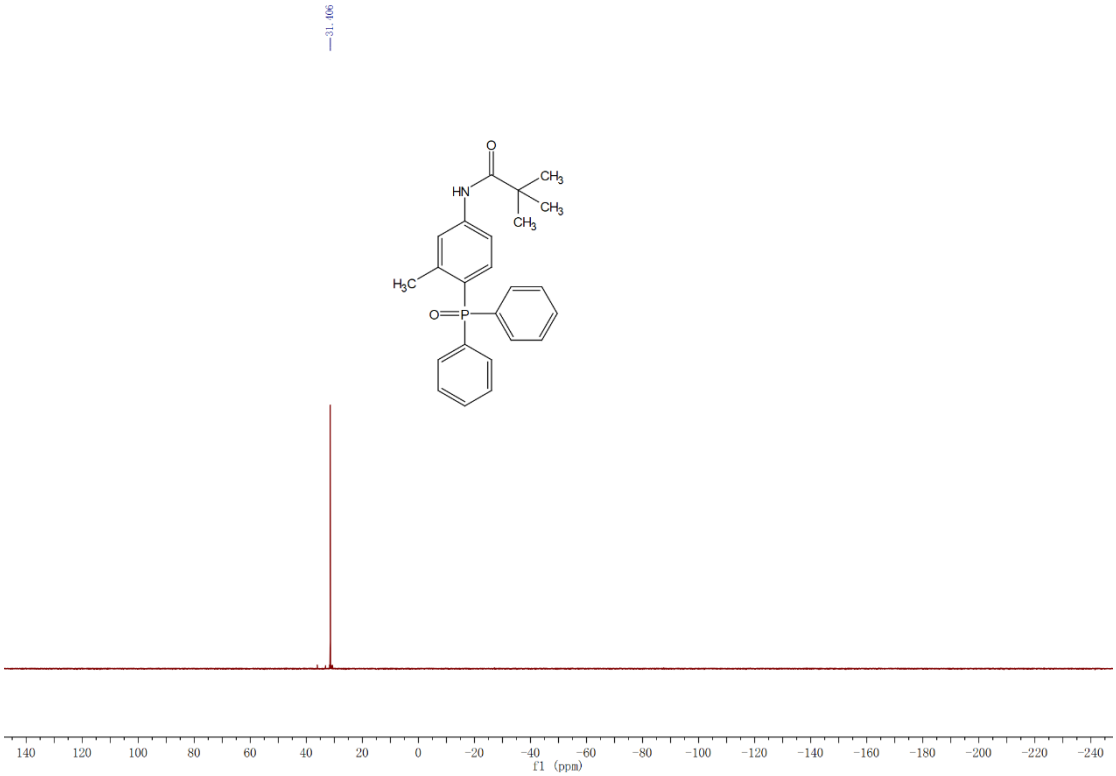



202.718
178.155
140.877
140.725
140.211
139.220
132.145
132.107
132.049
131.978
131.950
131.859
131.387
131.461
131.112
128.699
128.567
125.340
125.258
124.971
124.931
77.318
77.000
76.683
40.321
28.834
27.481
O
HN
CH3
CH3
CH3
O
H3C
O=P
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

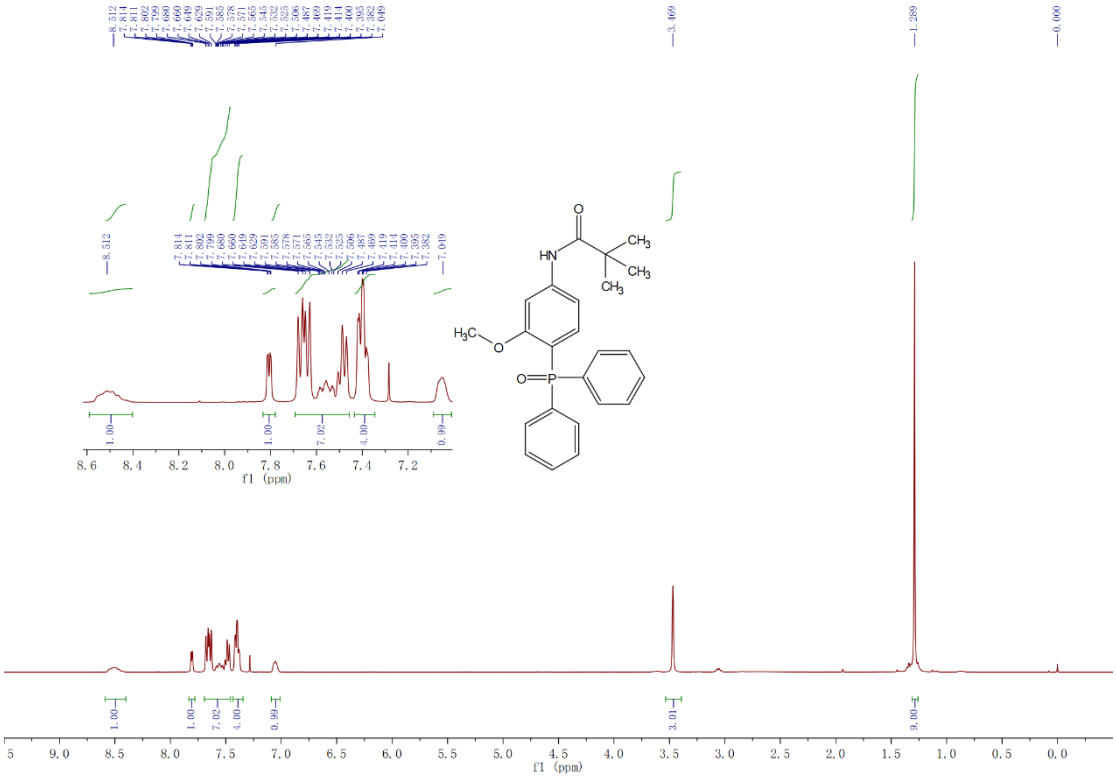

27.489
O
HN
CH3
CH3
CH3
O
H3C
O=P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

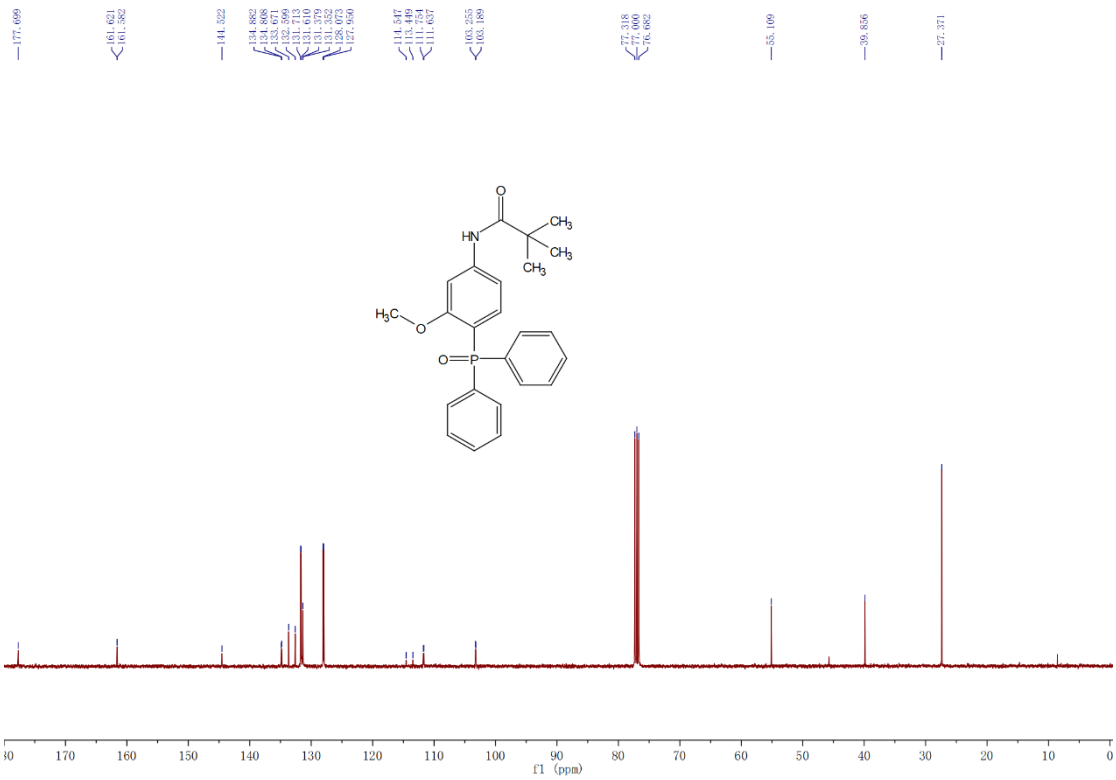
N-(4-(diphenylphosphoryl)-3-methylphenyl)pivalamide (3g)

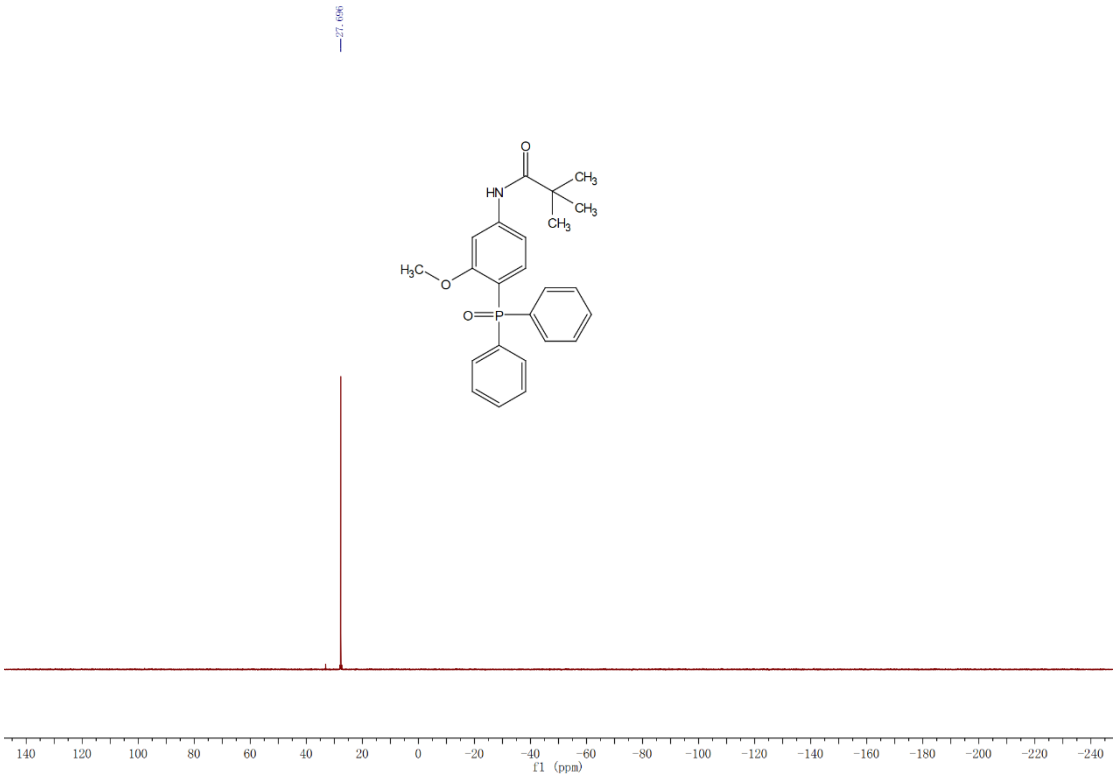




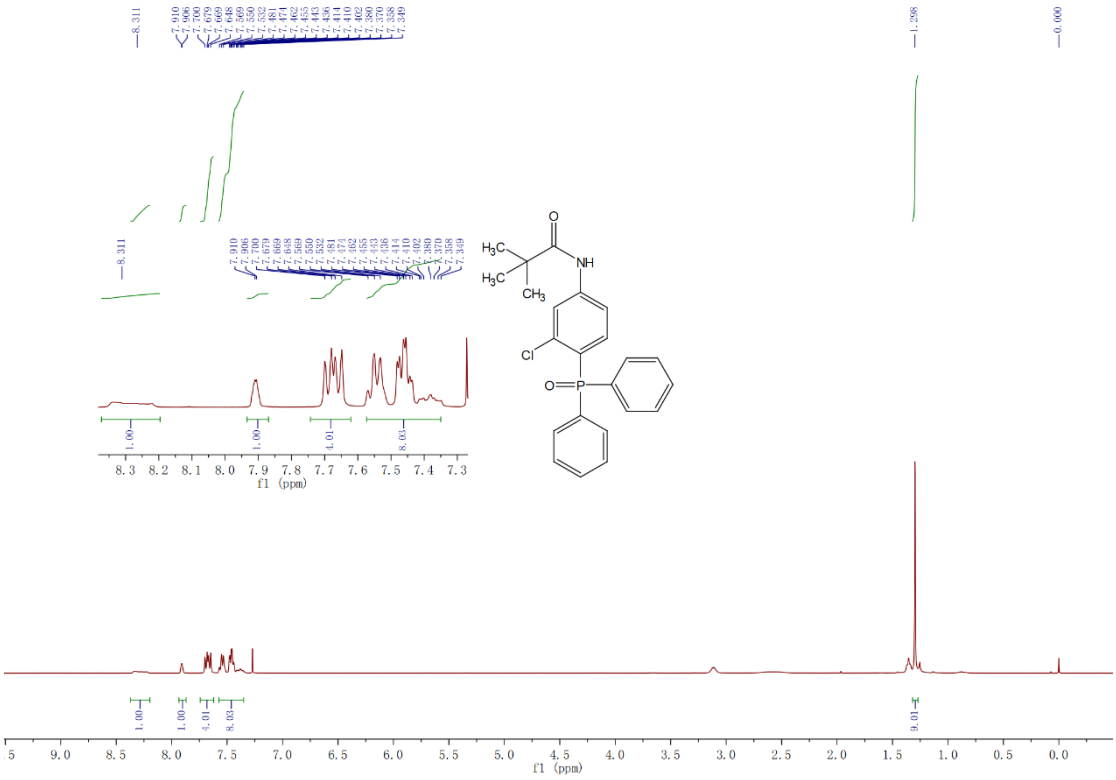
N-(4-(diphenylphosphoryl)-3-methoxyphenyl)pivalamide (3h)

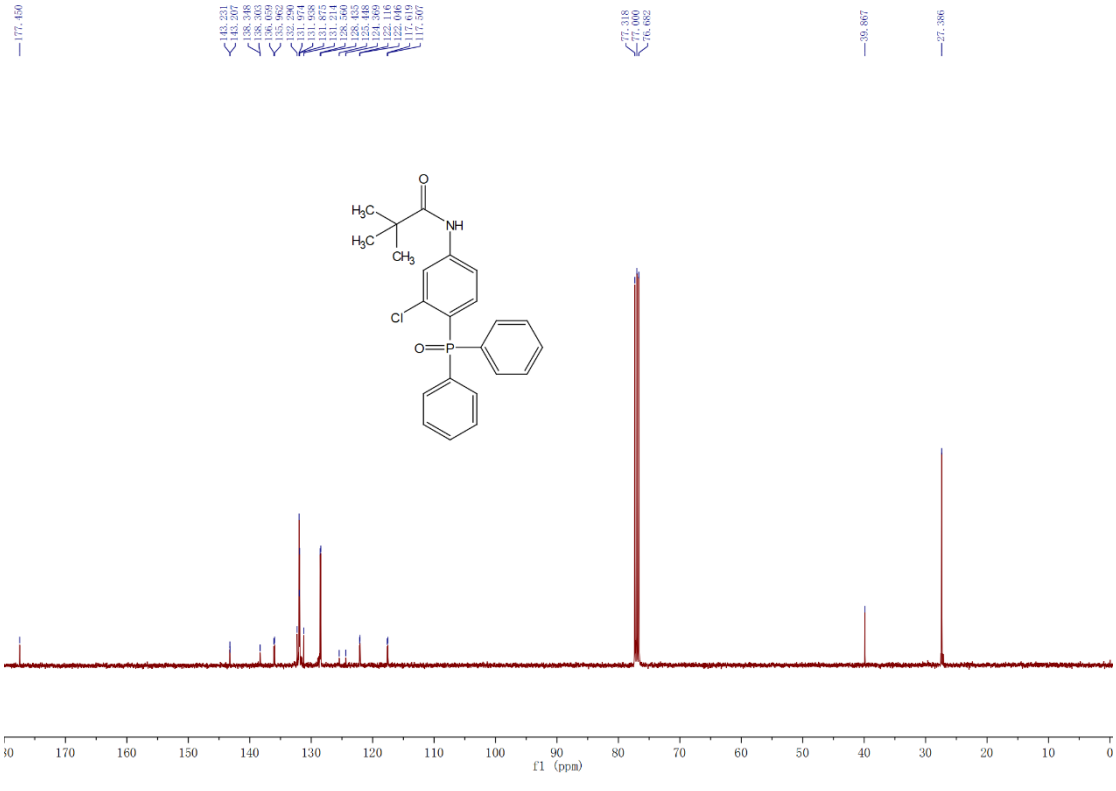


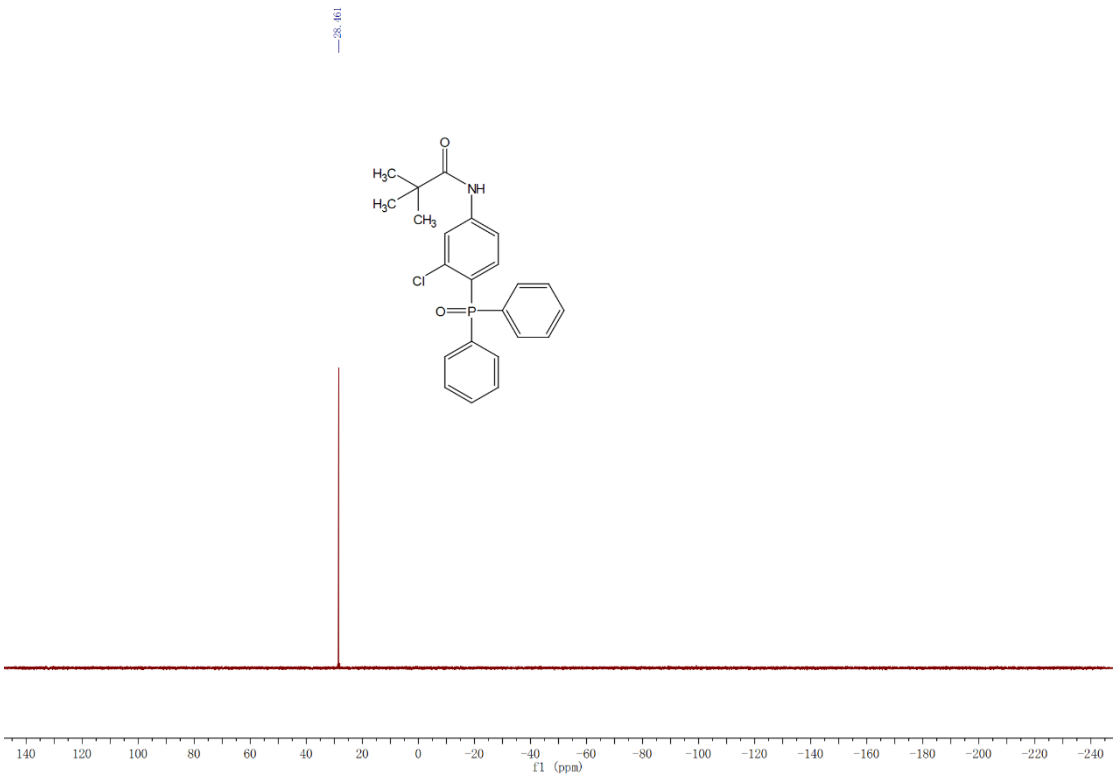

177.699
161.621
161.382
144.322
134.882
134.808
133.671
132.399
131.713
131.610
131.379
131.352
128.673
127.980
114.547
113.449
111.754
111.637
103.255
103.189
77.318
77.000
76.682
55.109
39.856
27.371
HN
O
CH3
CH3
CH3
H3C
O
O=P
f1 (ppm)


27.696
HN
O
CH3
CH3
CH3
H3C
O
O=P
f1 (ppm)

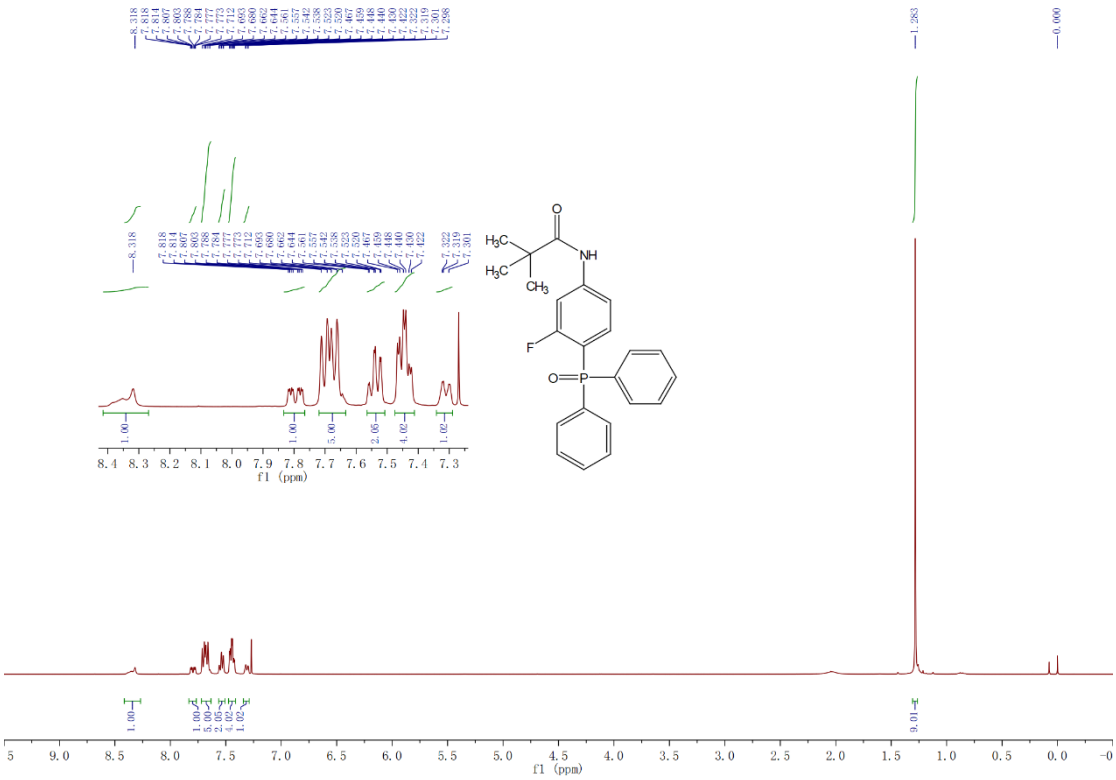
N-(3-chloro-4-(diphenylphosphoryl)phenyl)pivalamide (3i)

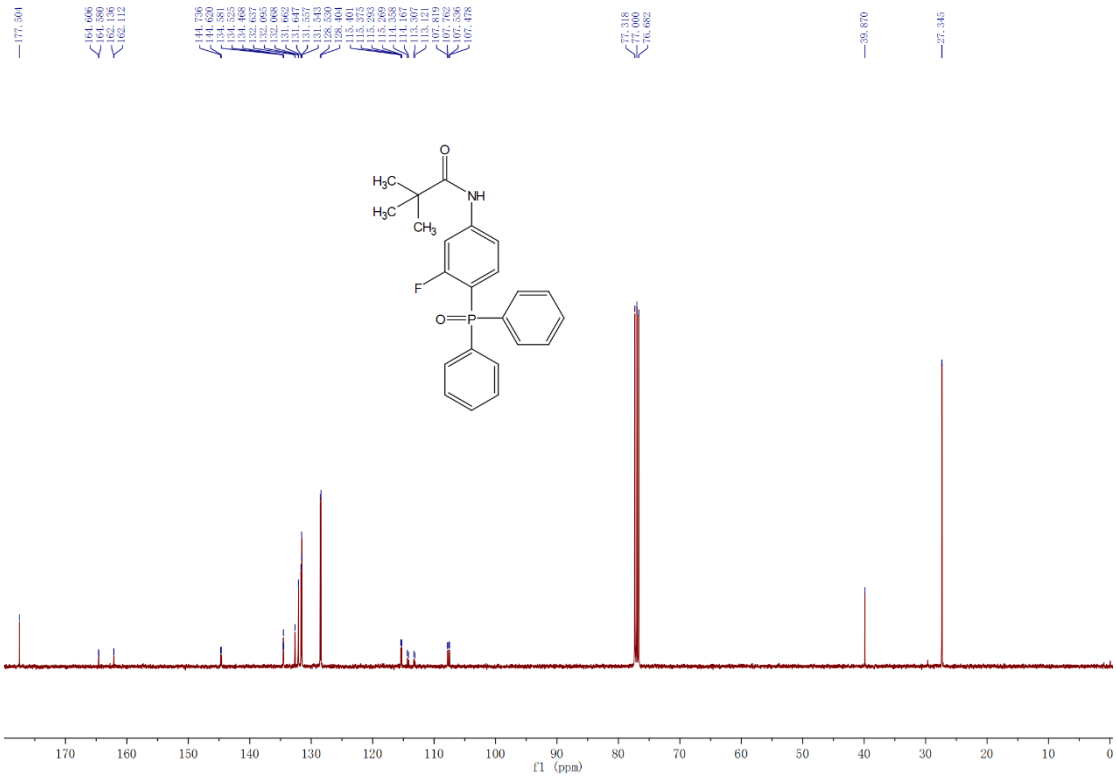


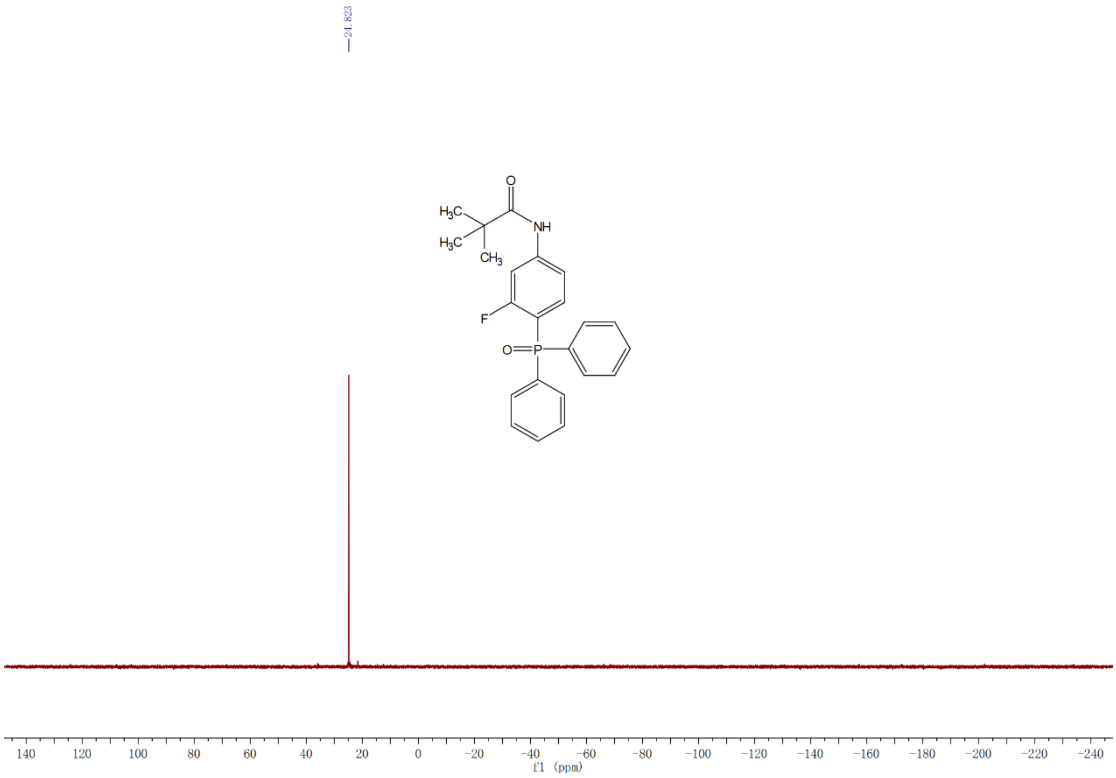


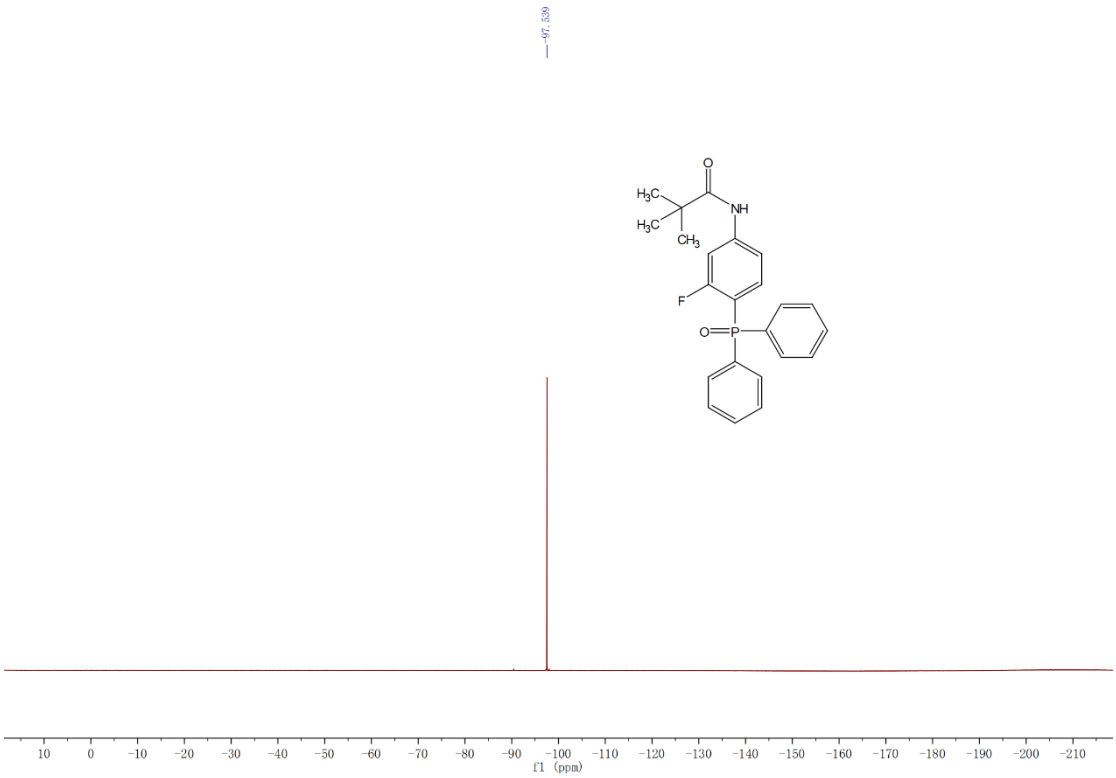


N-(4-(diphenylphosphoryl)-3-fluorophenyl)pivalamide (3j)

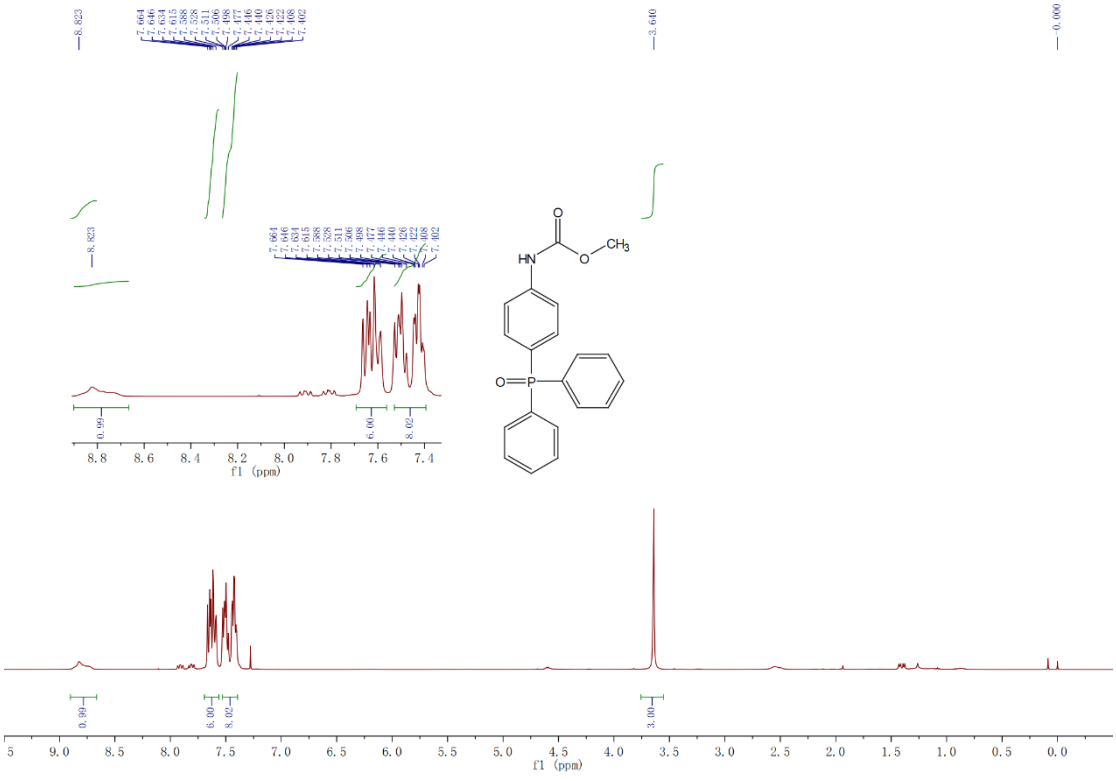


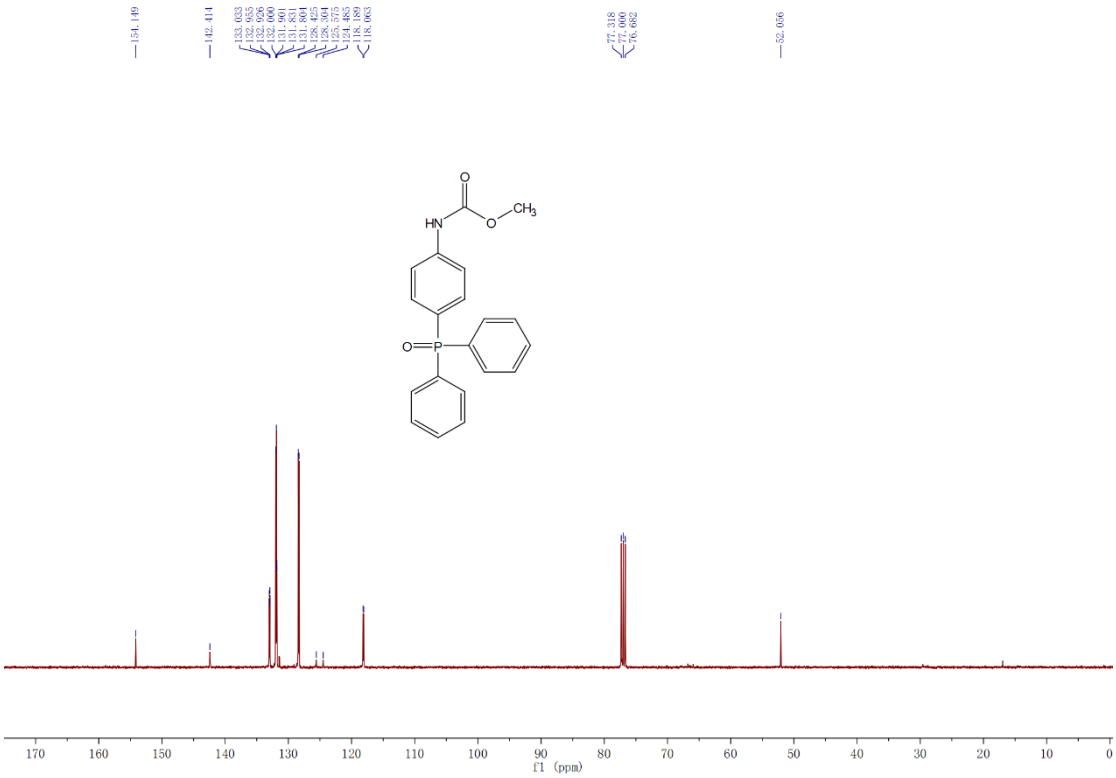

177.504
164.606
164.388
162.136
162.112
144.736
141.620
134.581
134.525
134.468
132.657
132.095
132.068
131.662
131.447
131.557
131.543
128.530
128.464
128.401
115.411
115.375
115.269
115.098
114.167
113.307
113.121
107.762
107.762
107.536
107.478
77.318
77.000
76.682
39.870
27.345
O
H3C
H3C
CH3
NH
F
O=P
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

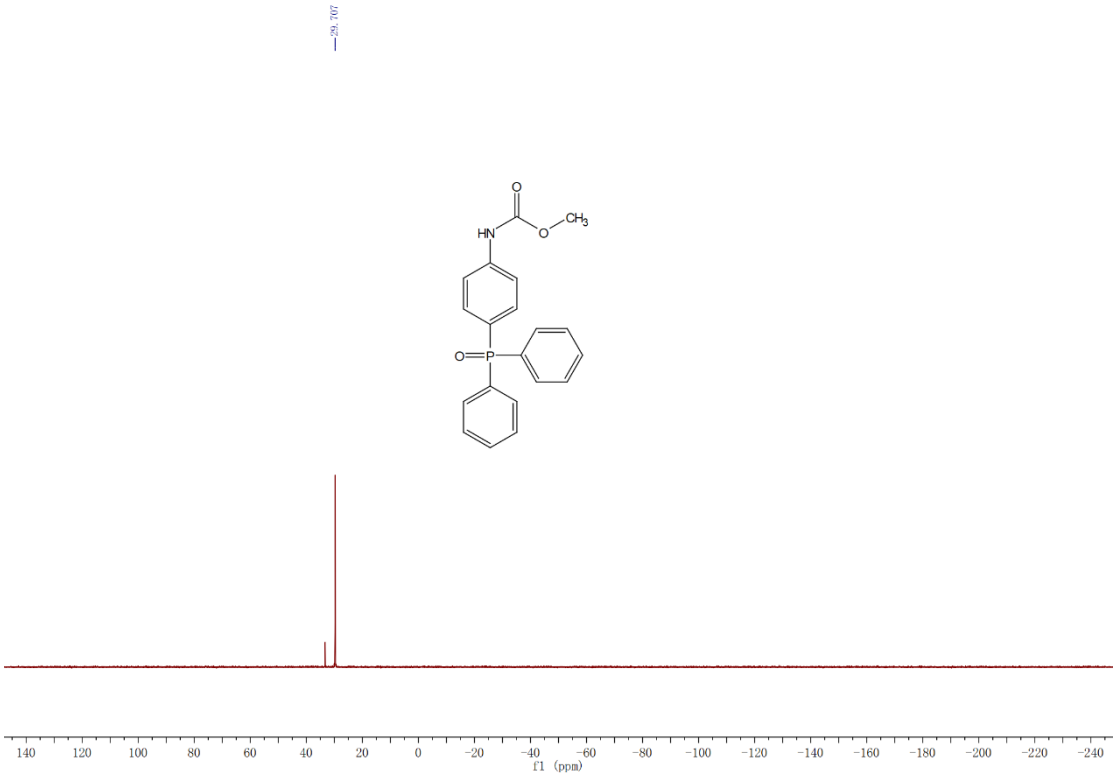

24.823
O
H3C
H3C
CH3
NH
F
O=P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)



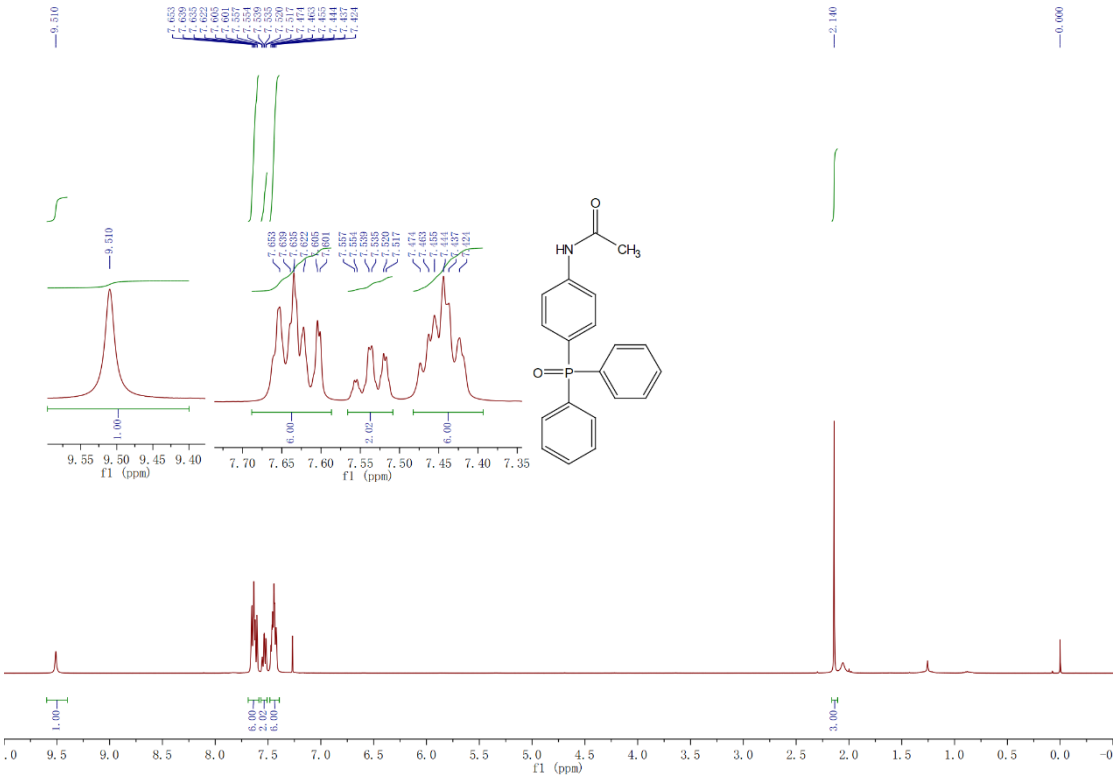
methyl (4-(diphenylphosphoryl)phenyl)carbamate (3k)

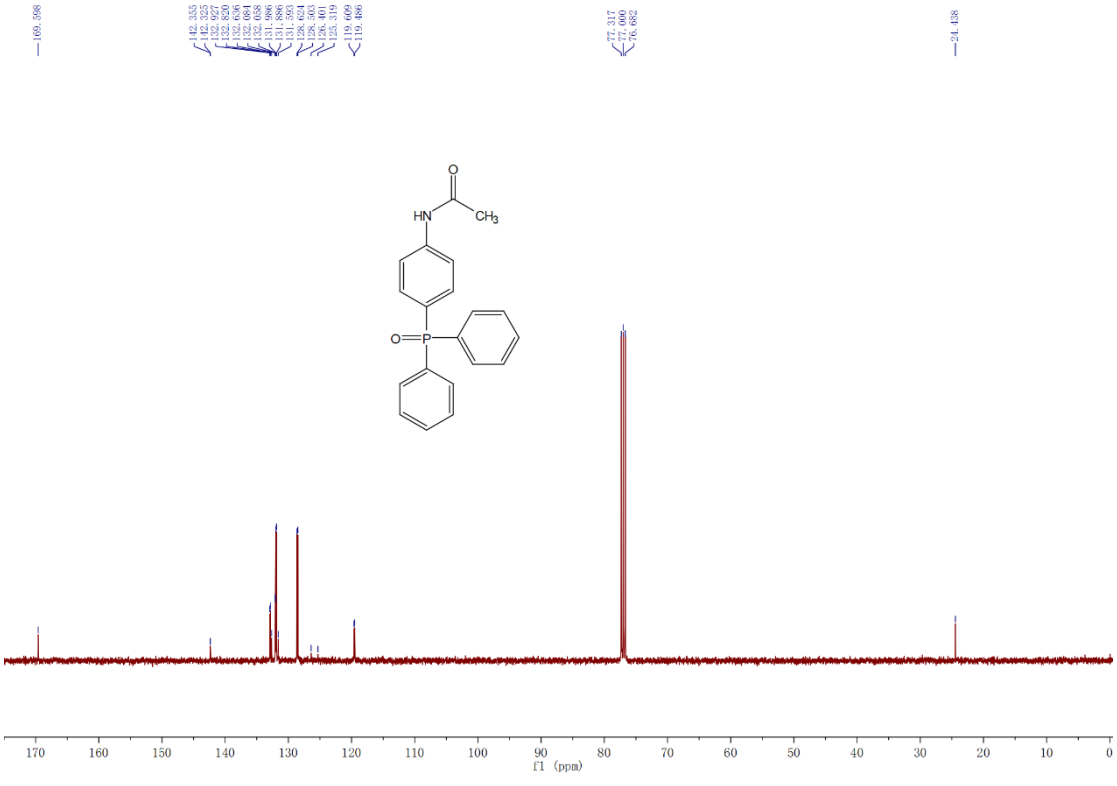


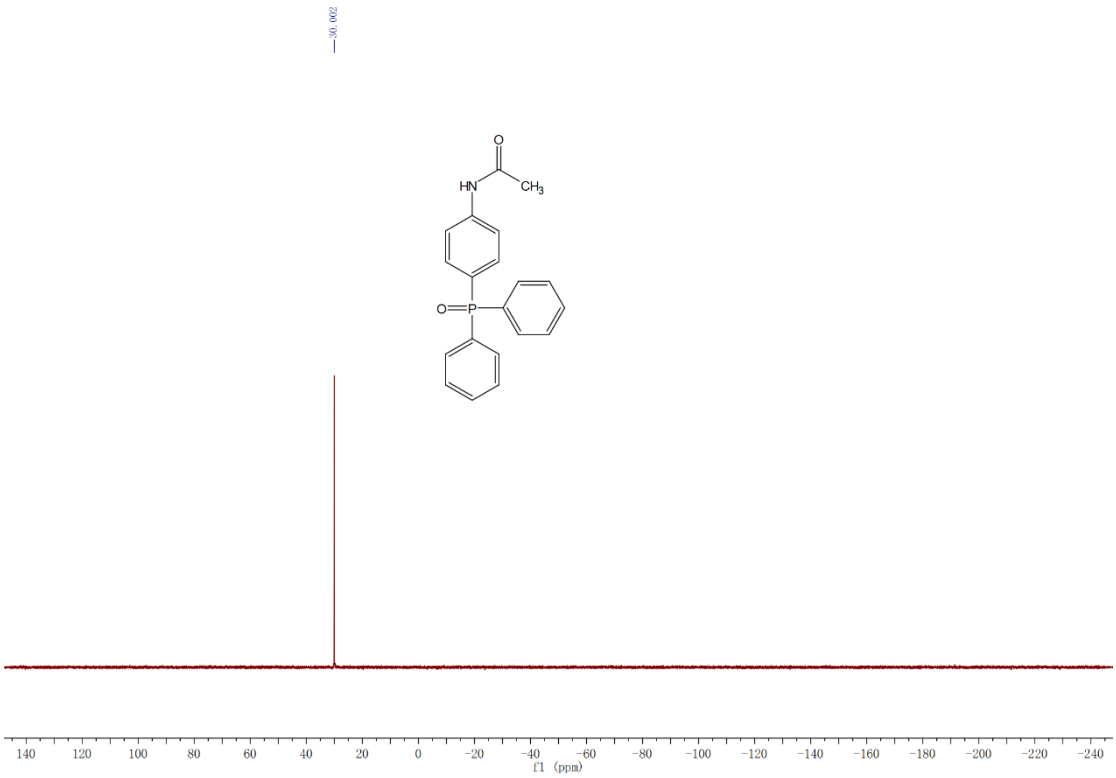

154.149
142.414
133.033
132.955
132.926
132.000
131.991
131.831
131.804
128.425
128.394
125.575
124.485
118.189
118.063
77.318
77.000
76.682
52.056
O
HN
O
CH3
O=P
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


29.707
O
HN
O
CH3
O=P
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240
f1 (ppm)

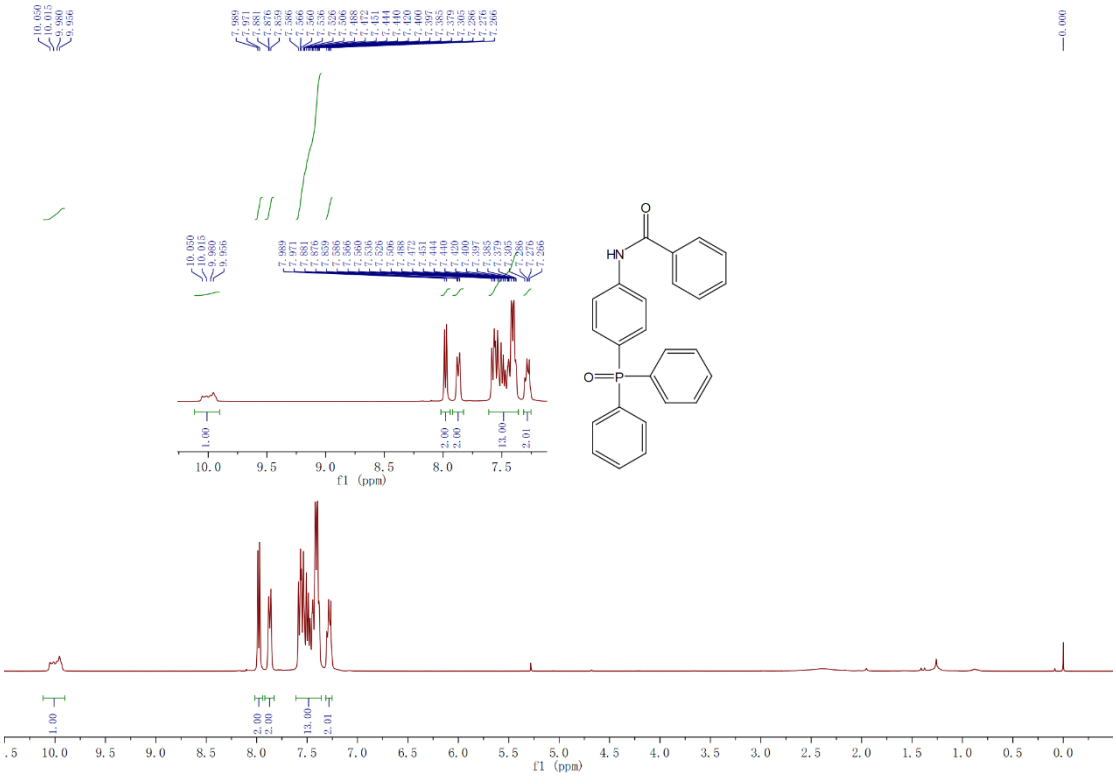
N-(4-(diphenylphosphoryl)phenyl)acetamide (3l)

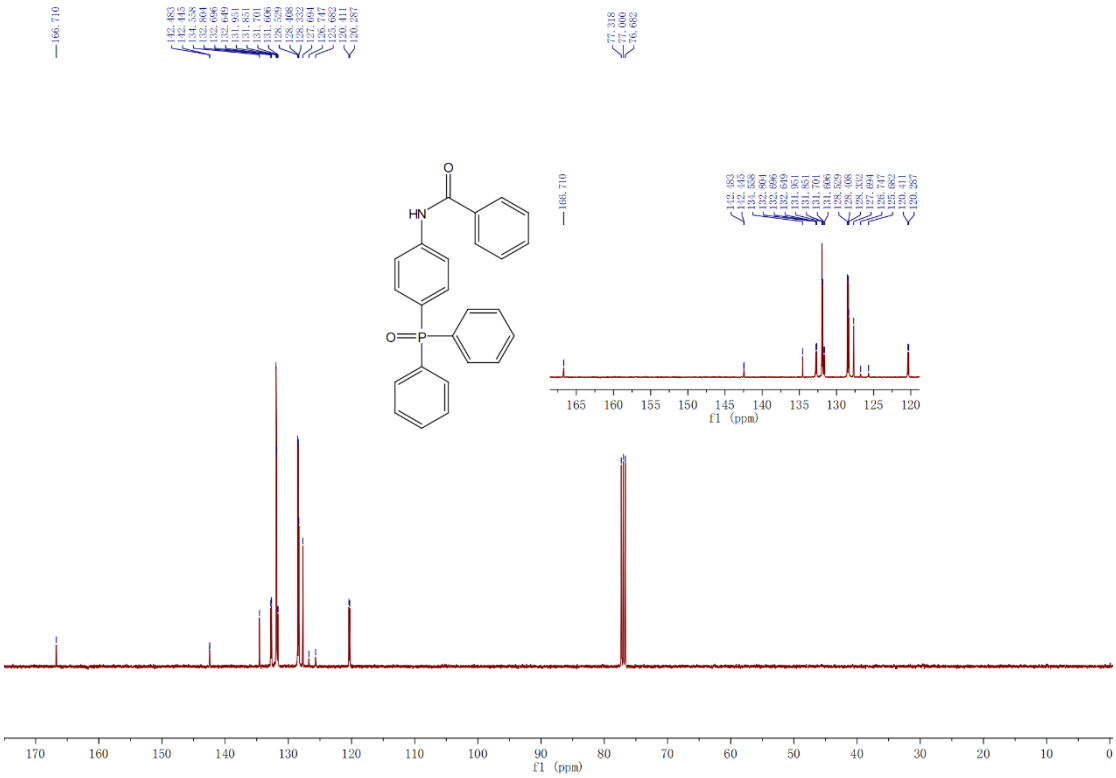


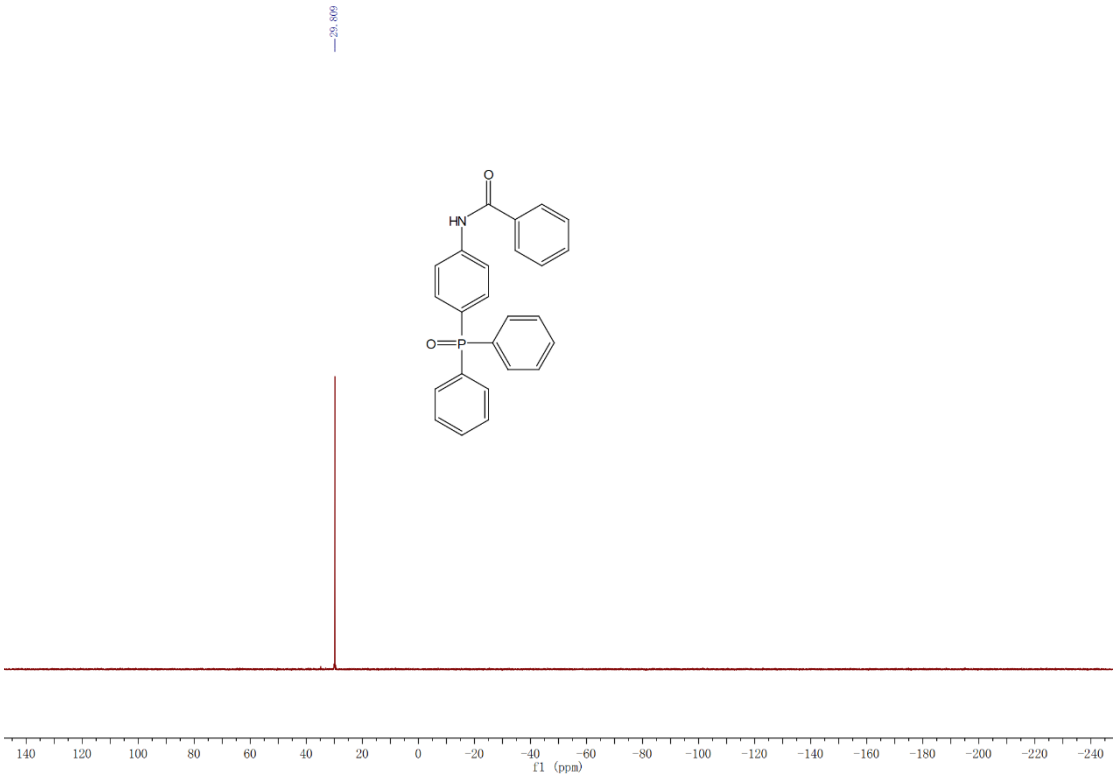




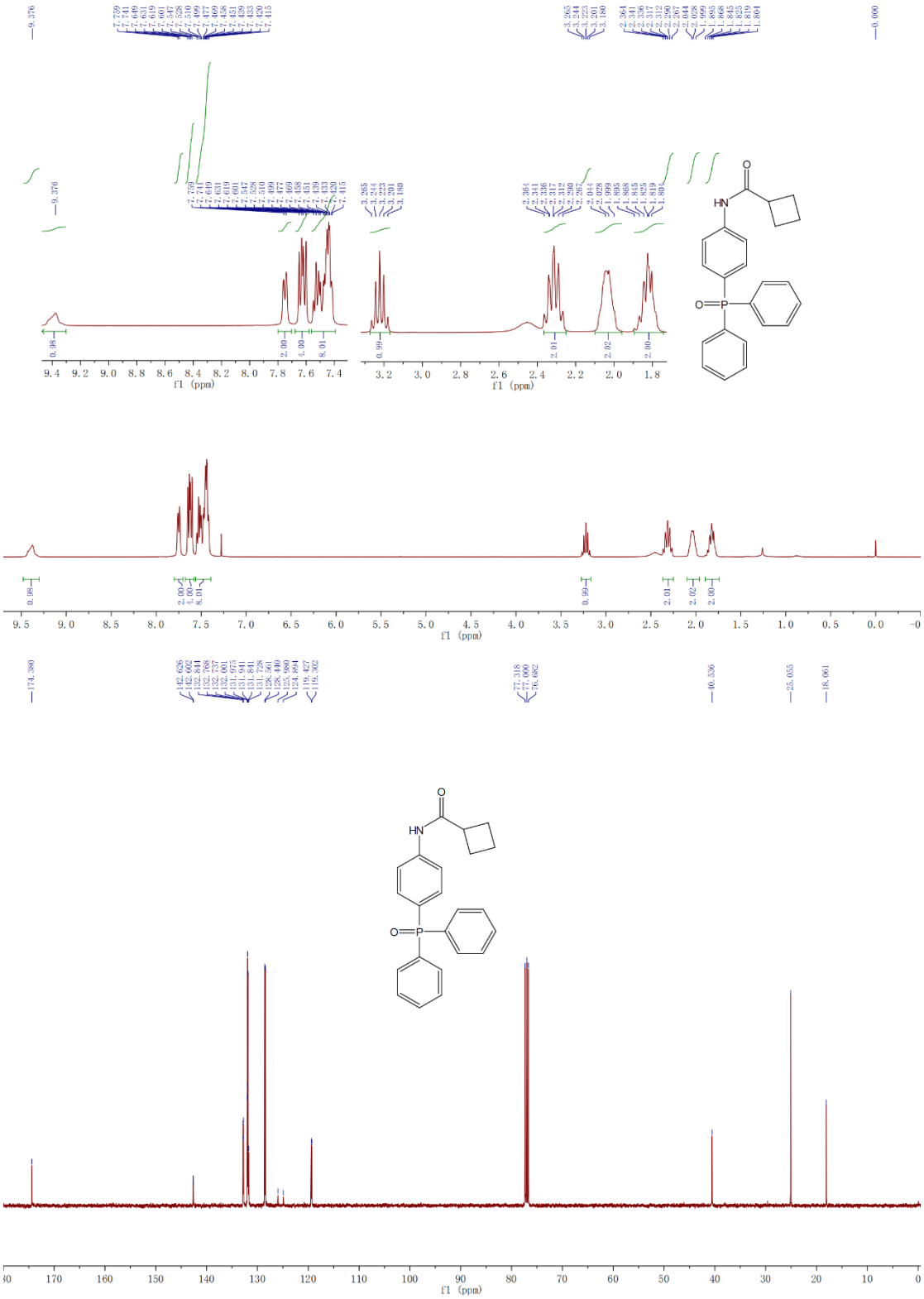
N-(4-(diphenylphosphoryl)phenyl)benzamide (3m)

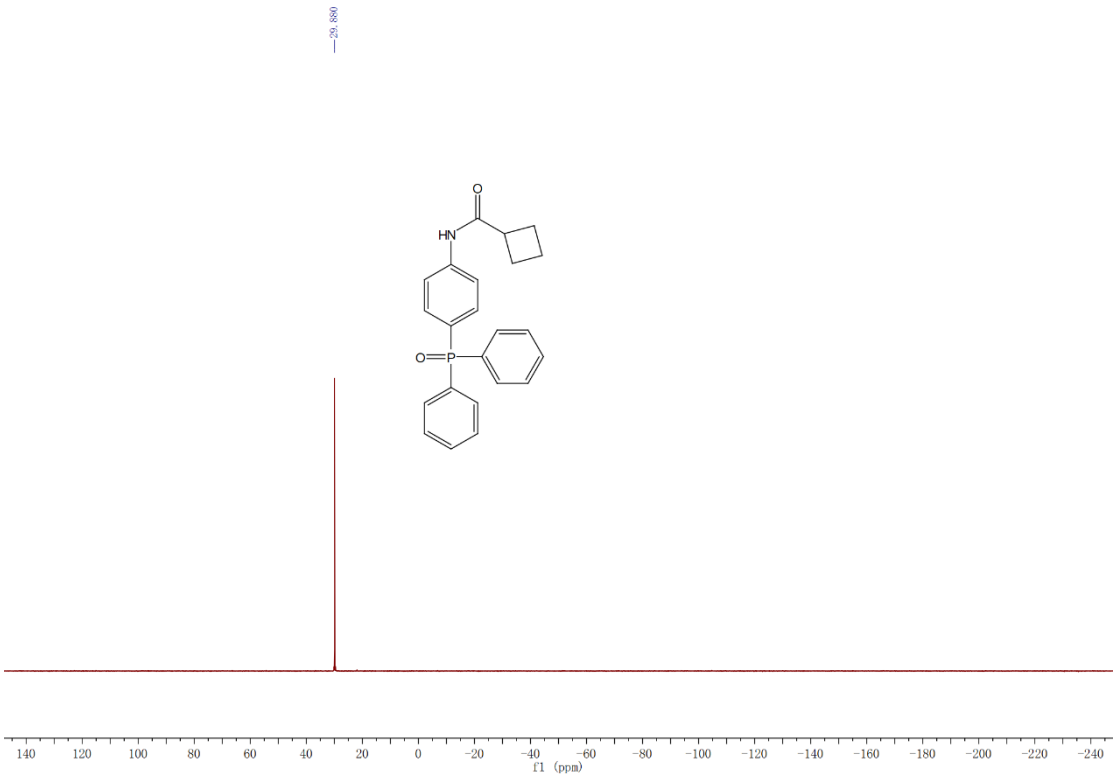



166.710
142.483
142.445
134.558
132.804
132.696
132.649
131.951
131.851
131.701
131.696
128.529
128.408
128.332
127.644
125.747
125.682
120.411
120.287
77.318
77.000
76.682
O
HN
O=P
f1 (ppm)

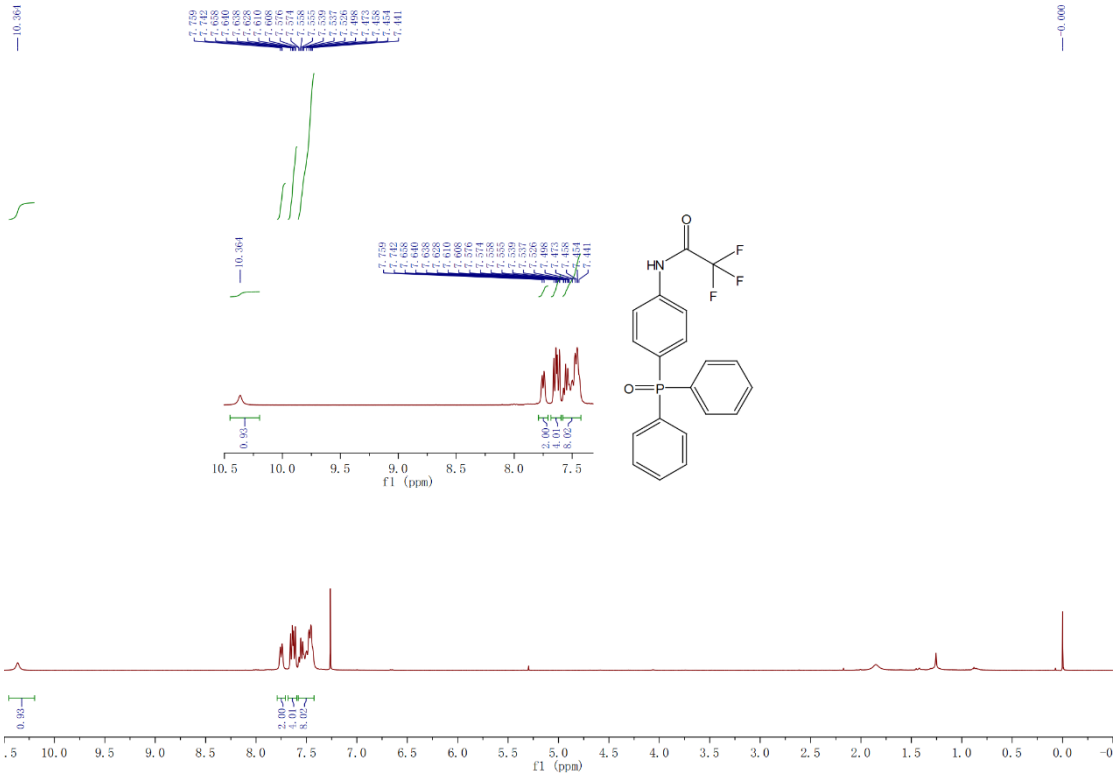

29.809
O
HN
O=P
f1 (ppm)

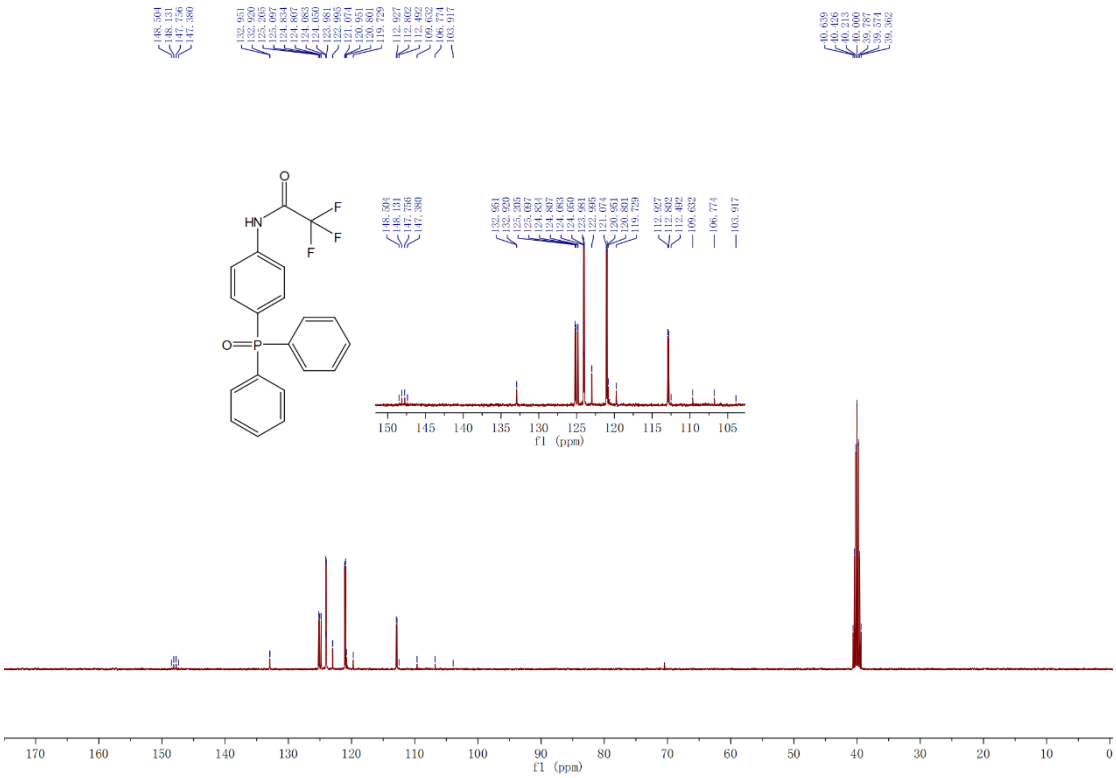
N-(4-(diphenylphosphoryl)phenyl)cyclobutanecarboxamide (3n)

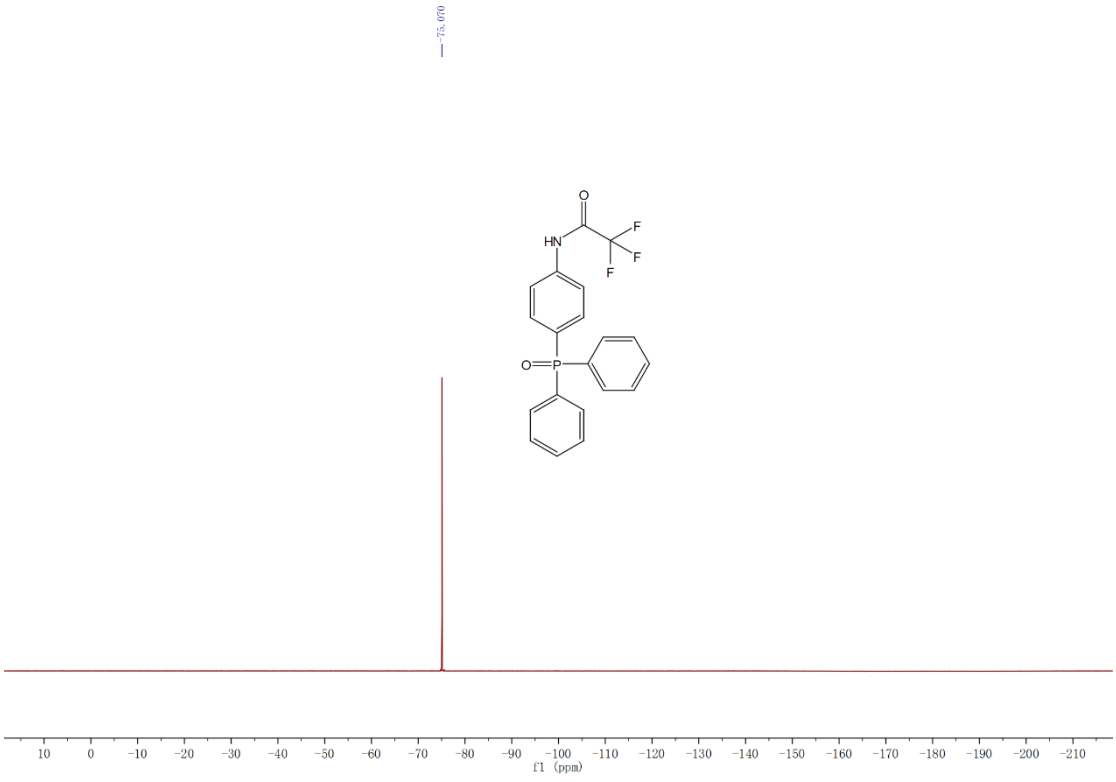


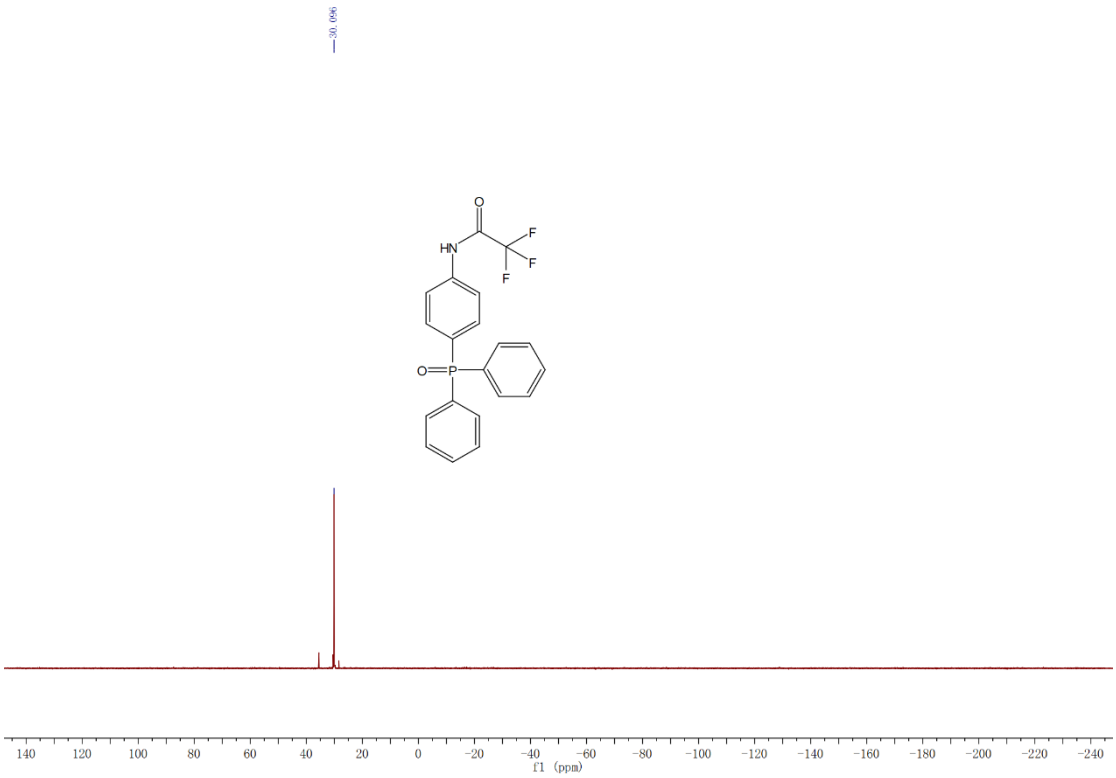


N-(4-(diphenylphosphoryl)phenyl)-2,2,2-trifluoroacetamide (3o)

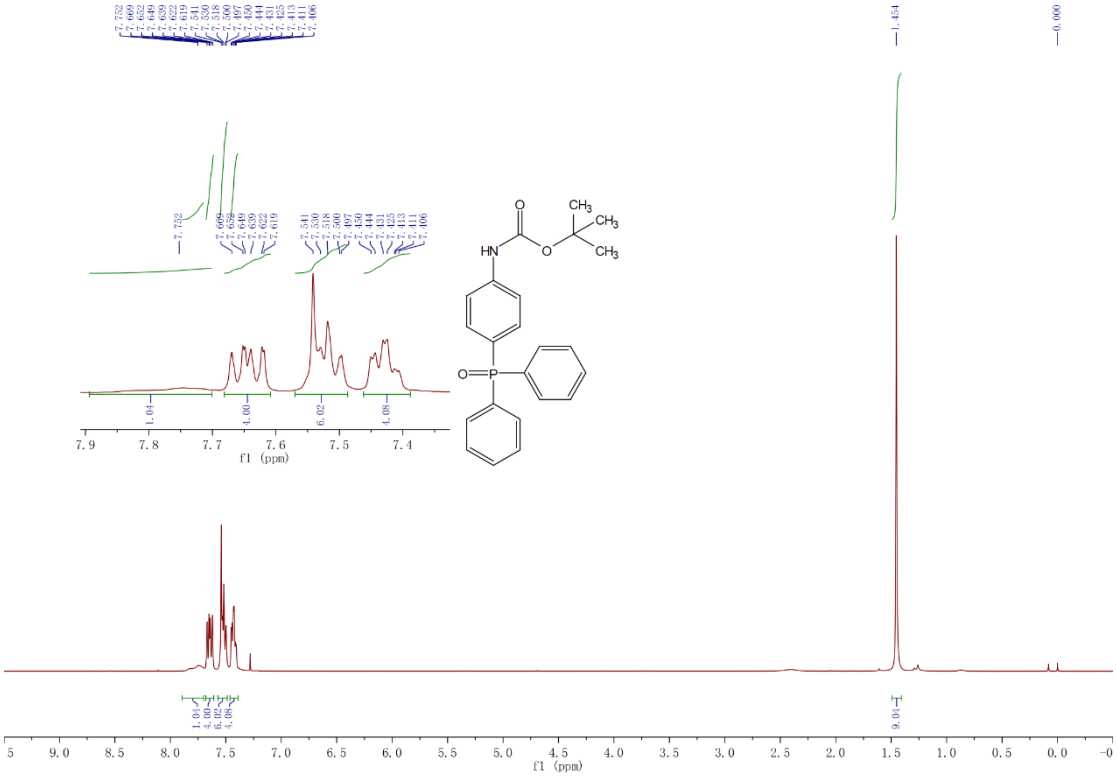


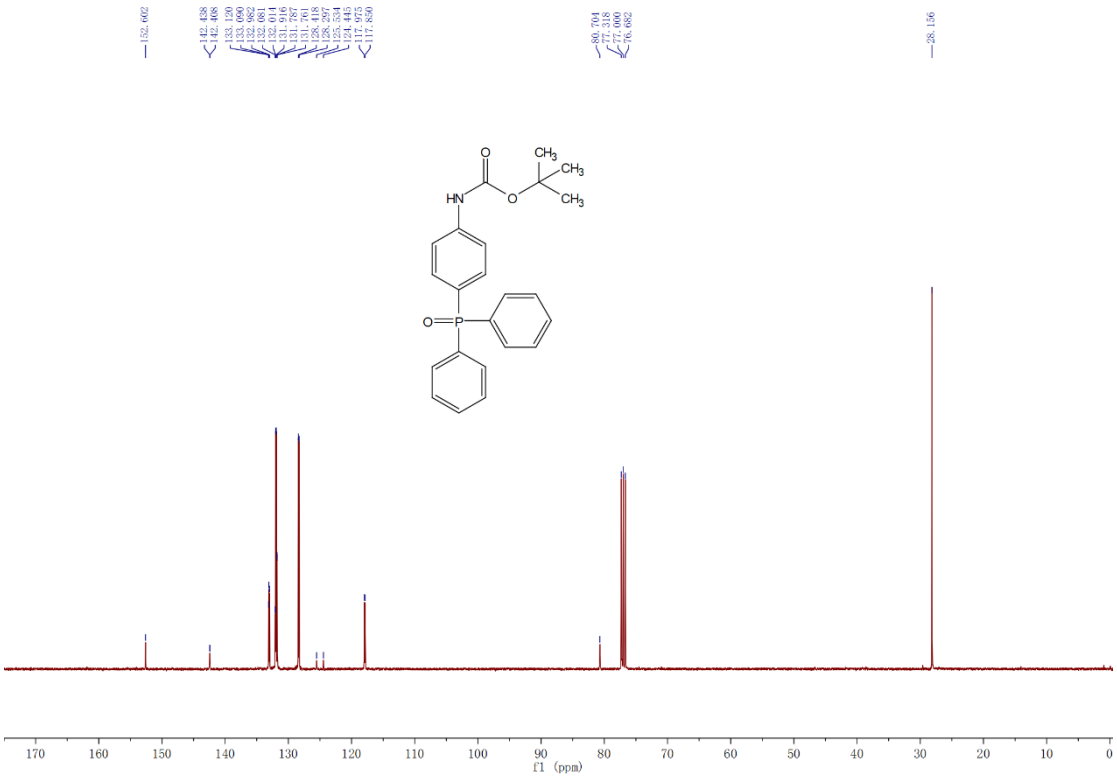

148.504
148.131
147.756
147.380
132.951
132.950
125.205
125.097
124.854
124.807
124.683
124.650
123.981
122.985
121.074
120.951
120.891
119.729
112.927
112.802
112.492
109.632
106.774
103.917
40.639
40.426
40.213
40.000
39.787
39.574
39.362
HN
O
F
F
F
O
P
150 145 140 135 130 125 120 115 110 105
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

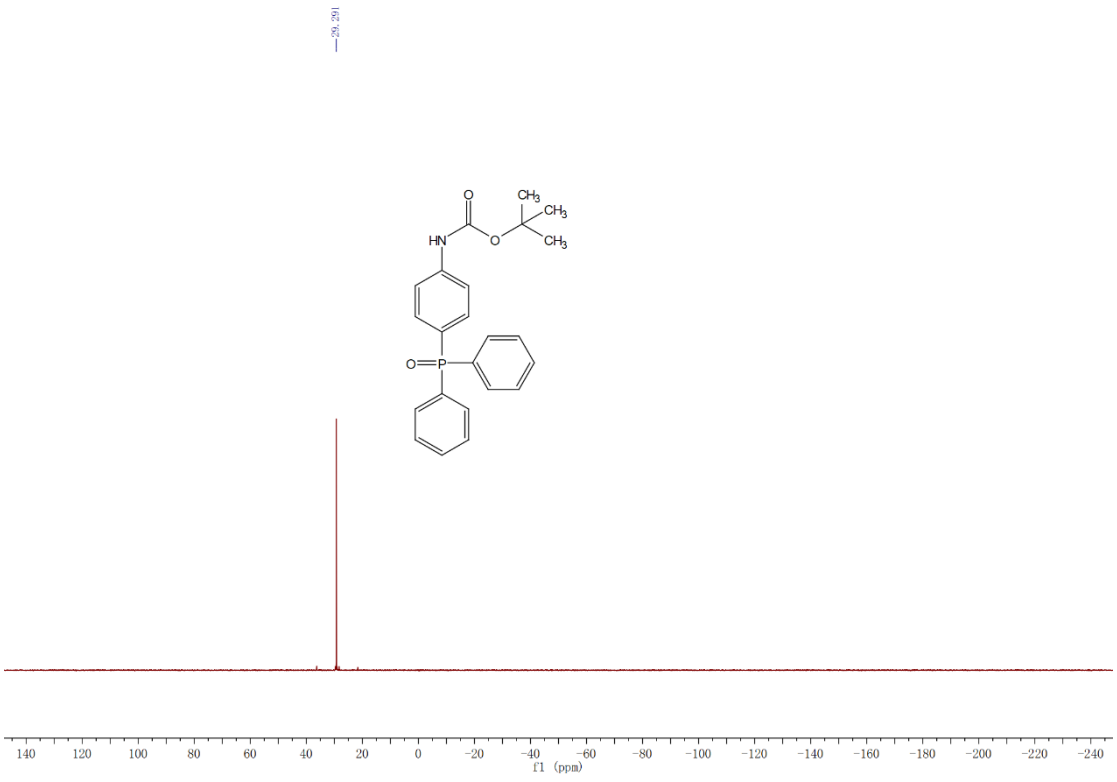

-75.070
HN
O
F
F
F
O
P
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)



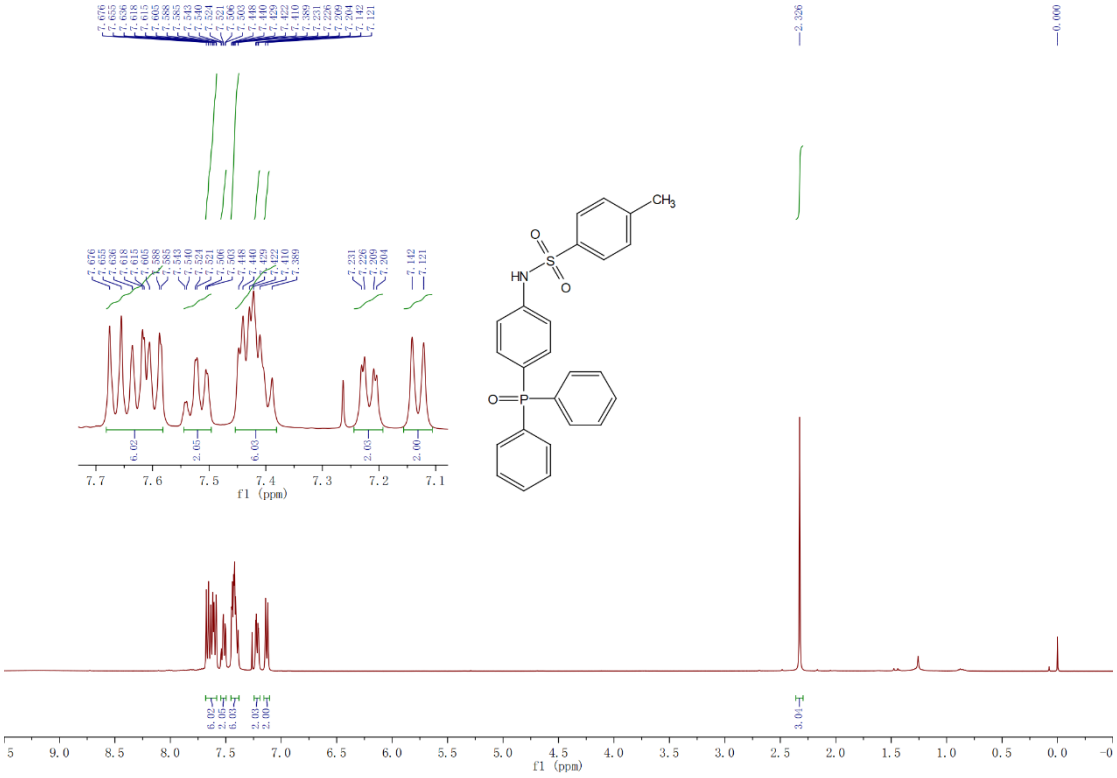
tert-butyl (4-(diphenylphosphoryl)phenyl)carbamate (3p)

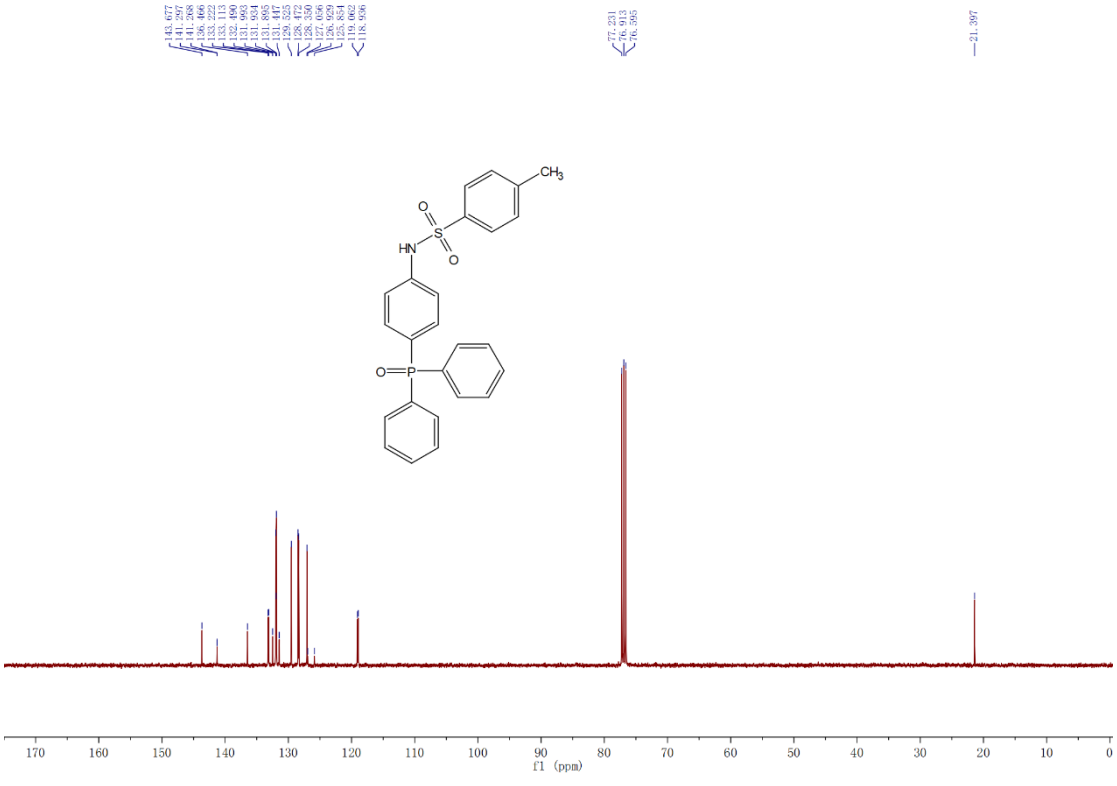


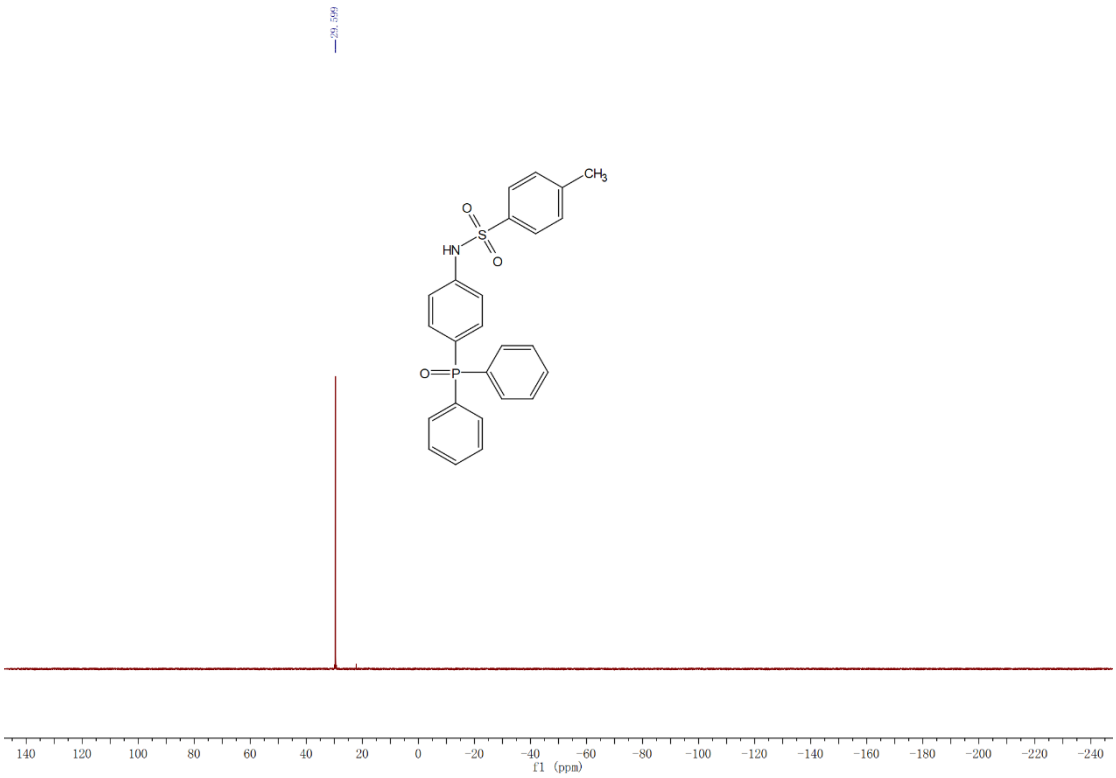

152.602
142.438
142.408
133.120
133.060
132.982
132.681
132.614
131.946
131.787
131.761
128.418
128.297
125.534
124.445
117.975
117.850
80.704
77.418
77.000
76.582
28.156
HN
O
CH3
CH3
CH3
O
O=P
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


29.291
HN
O
CH3
CH3
CH3
O
O=P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

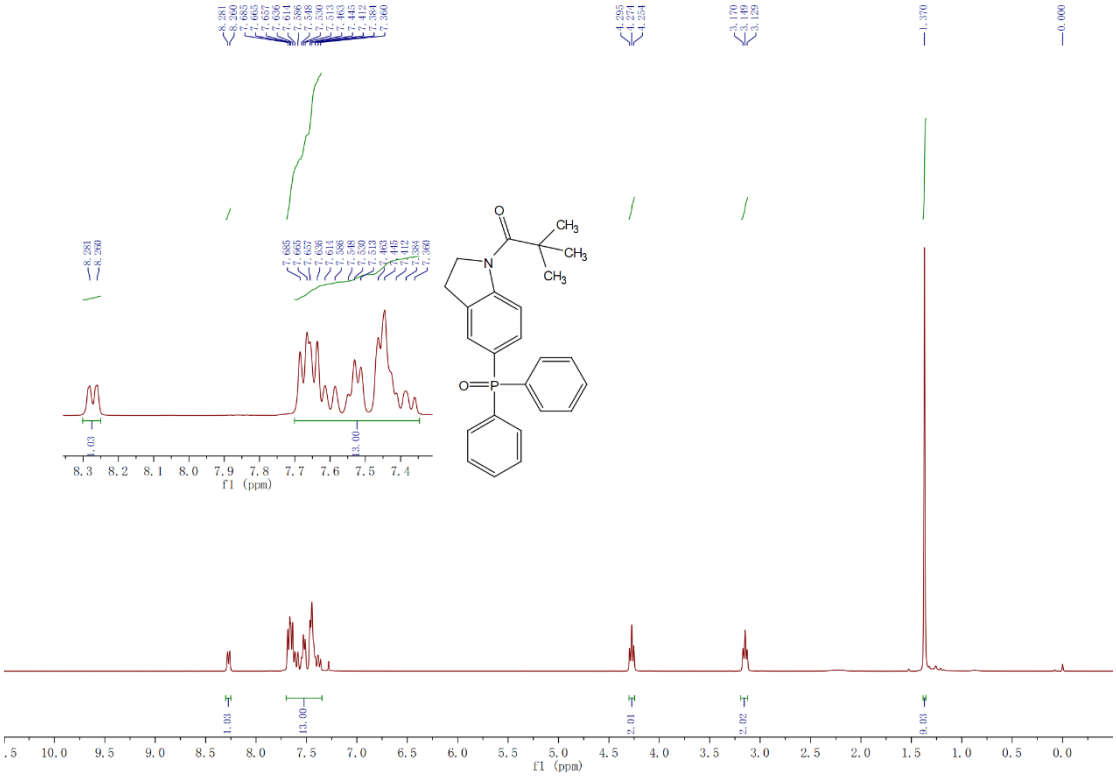
N-(4-(diphenylphosphoryl)phenyl)-4-methylbenzenesulfonamide (3q)

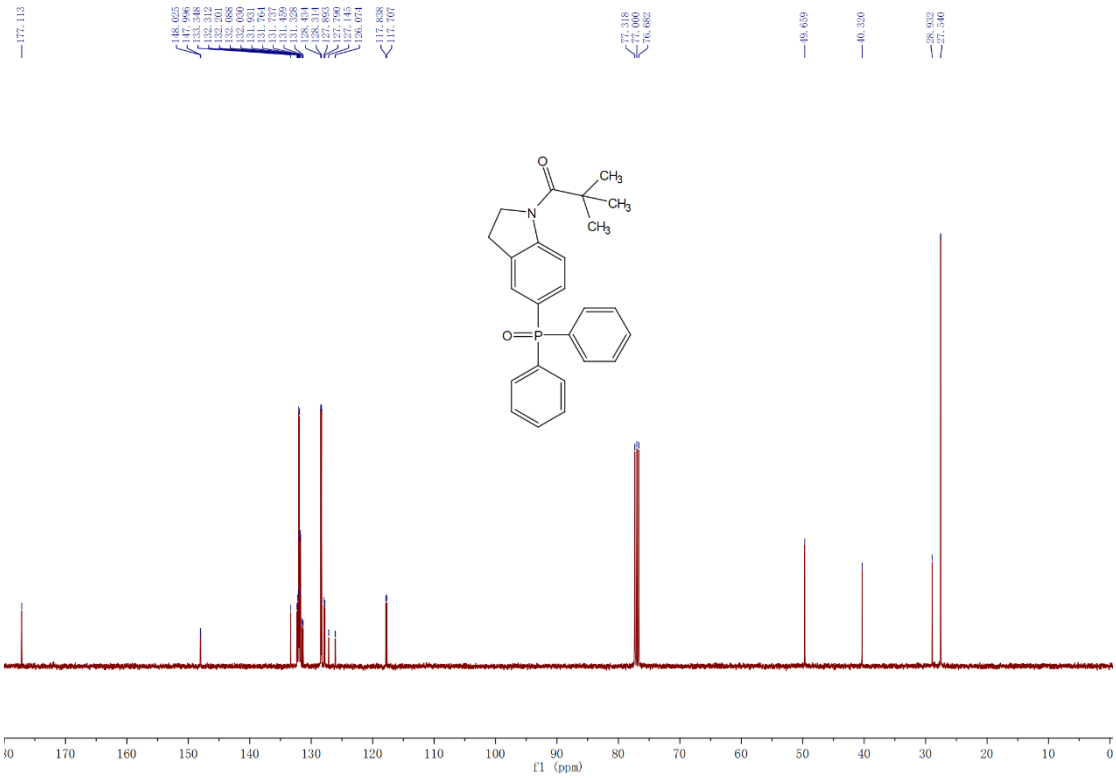


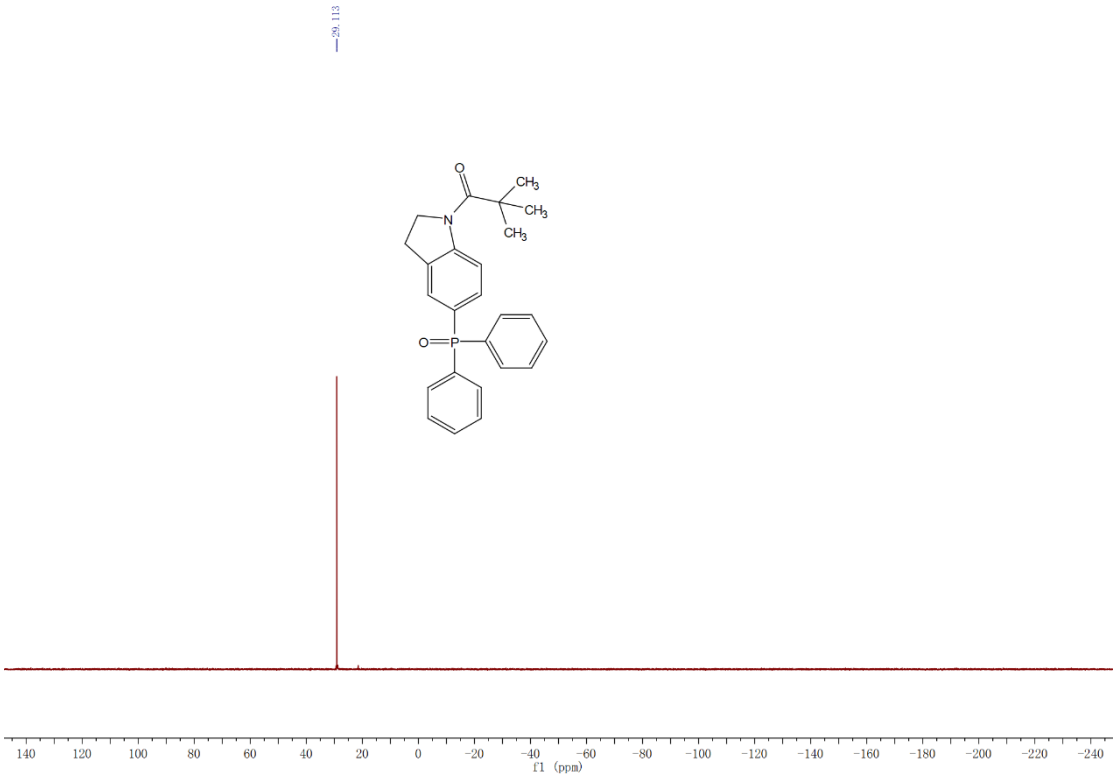




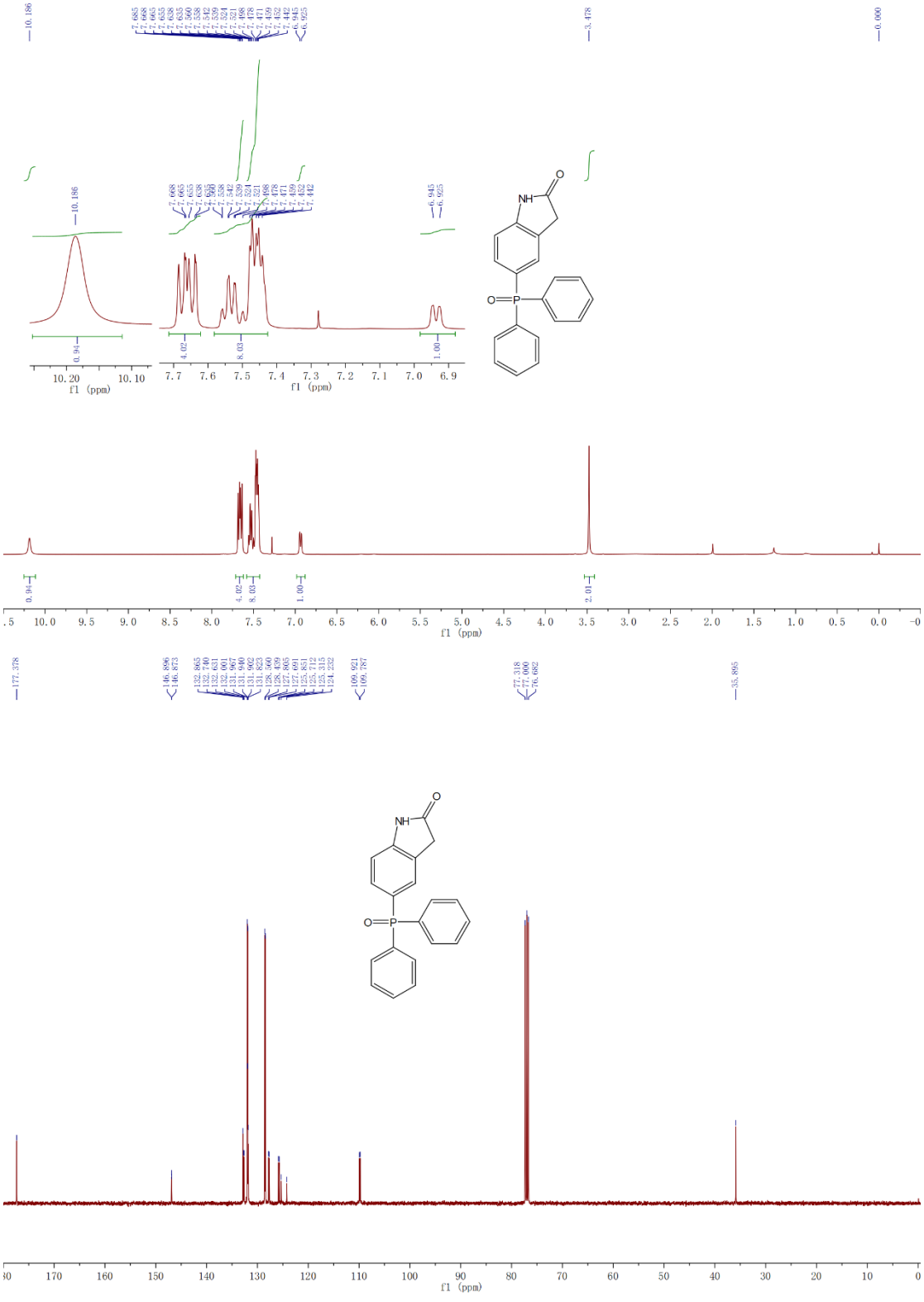
1-(5-(diphenylphosphoryl)indolin-1-yl)-2,2-dimethylpropan-1-one (3r)

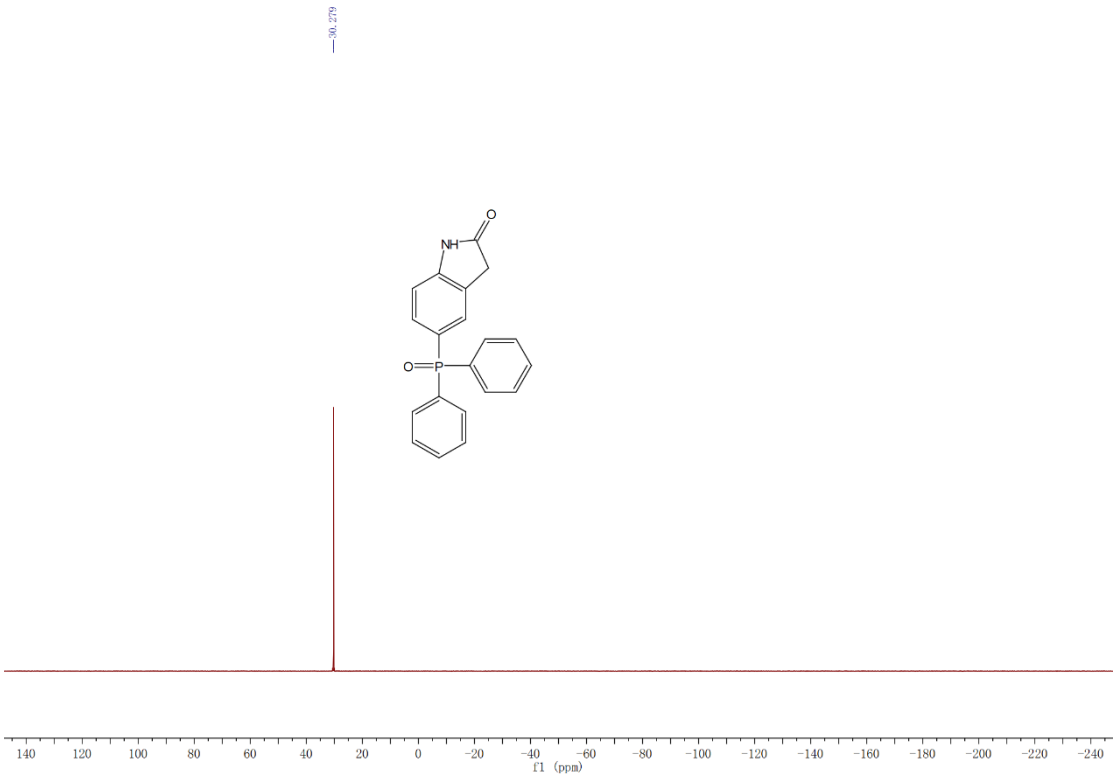



177.113
148.025
147.996
143.146
132.312
132.201
132.060
131.931
131.764
131.737
131.489
131.358
128.434
128.314
127.893
127.769
127.145
126.074
117.838
117.707
77.318
77.000
76.682
49.639
40.320
28.932
27.540
O
CH3
CH3
CH3
N
O=P
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

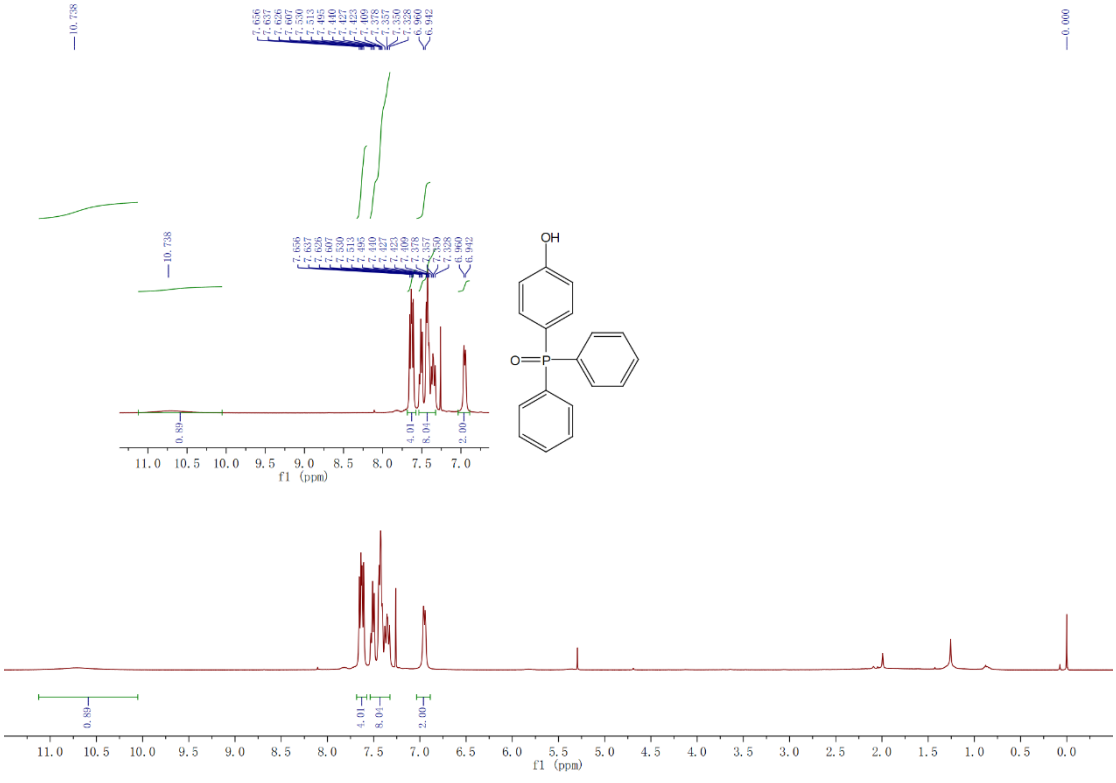

29.113
O
CH3
CH3
CH3
N
O=P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

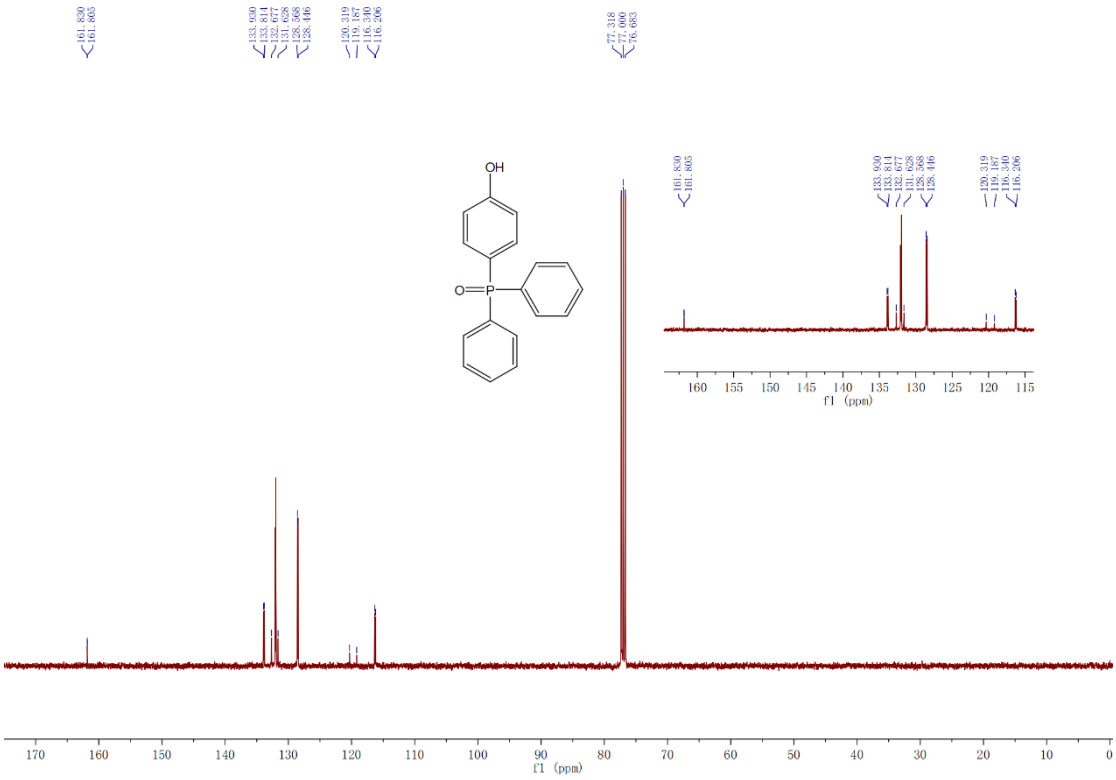
N-(4-(diphenylphosphoryl)phenyl)acetamide (3s)

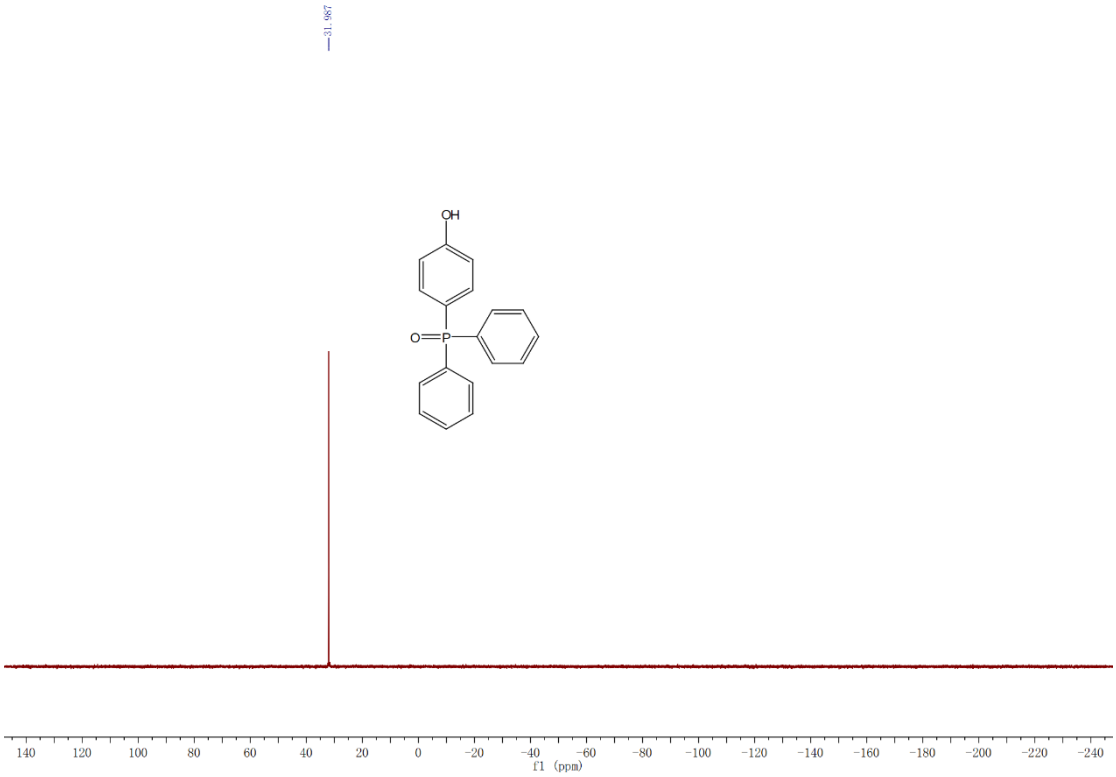




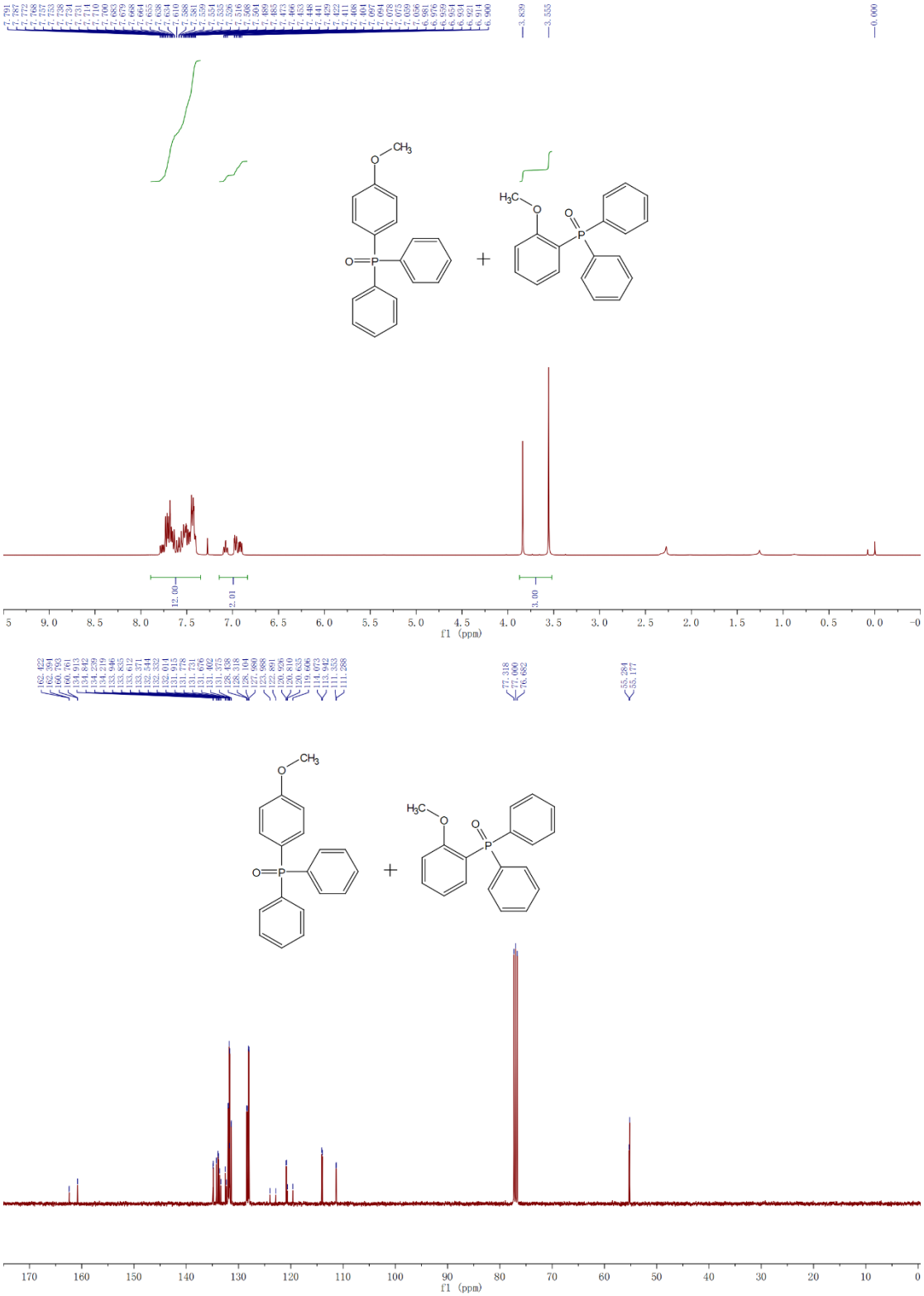
(4-hydroxyphenyl)diphenylphosphine oxide (3t)

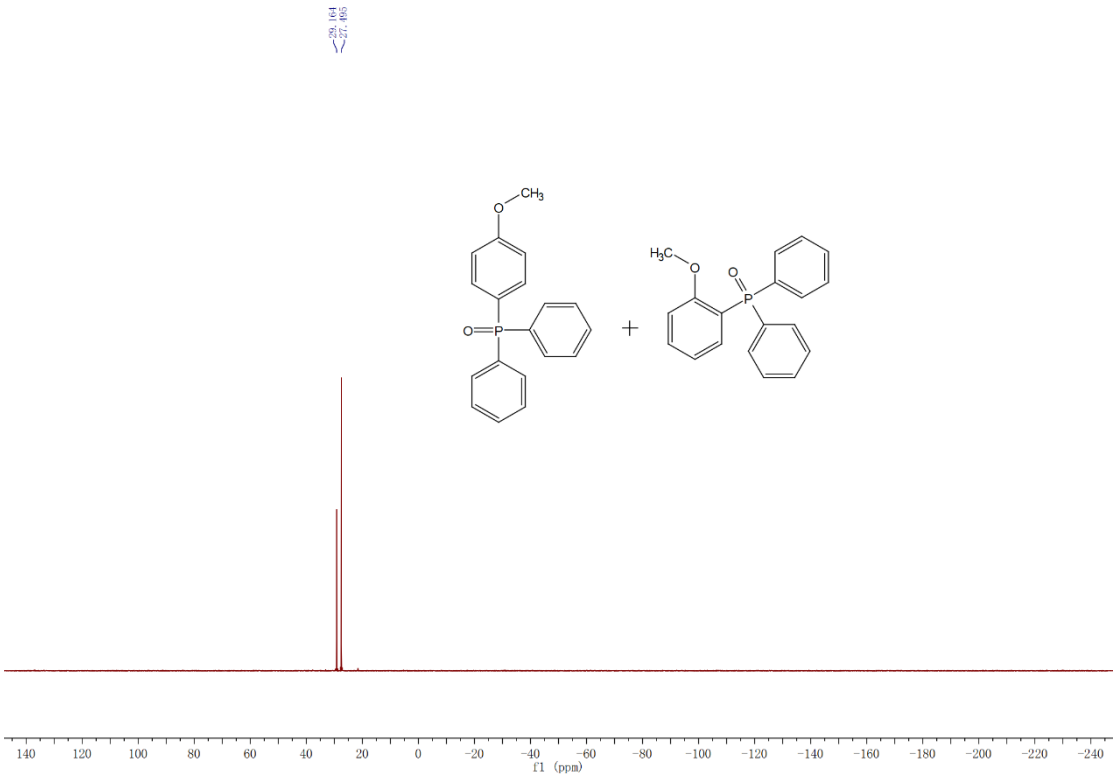



161.830
161.865
133.930
133.814
132.677
131.628
128.568
128.446
120.319
119.187
116.340
116.206
77.318
77.000
76.683
OH
O=P
f1 (ppm)

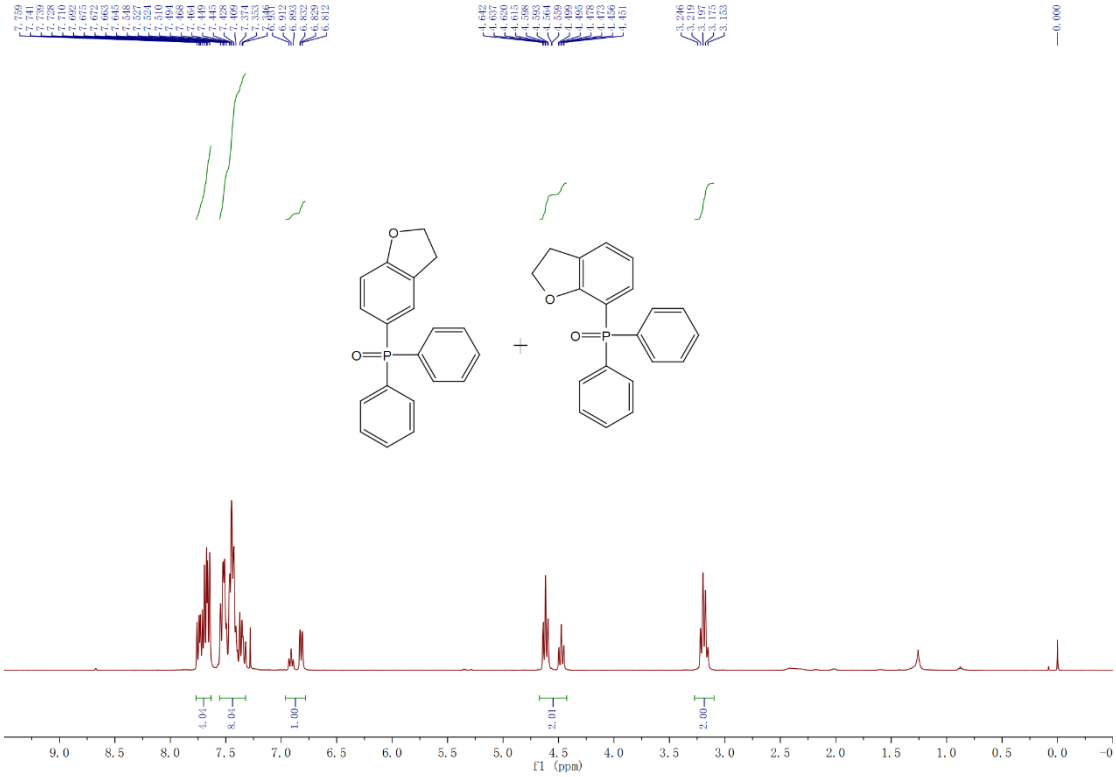

31.987
OH
O=P
f1 (ppm)

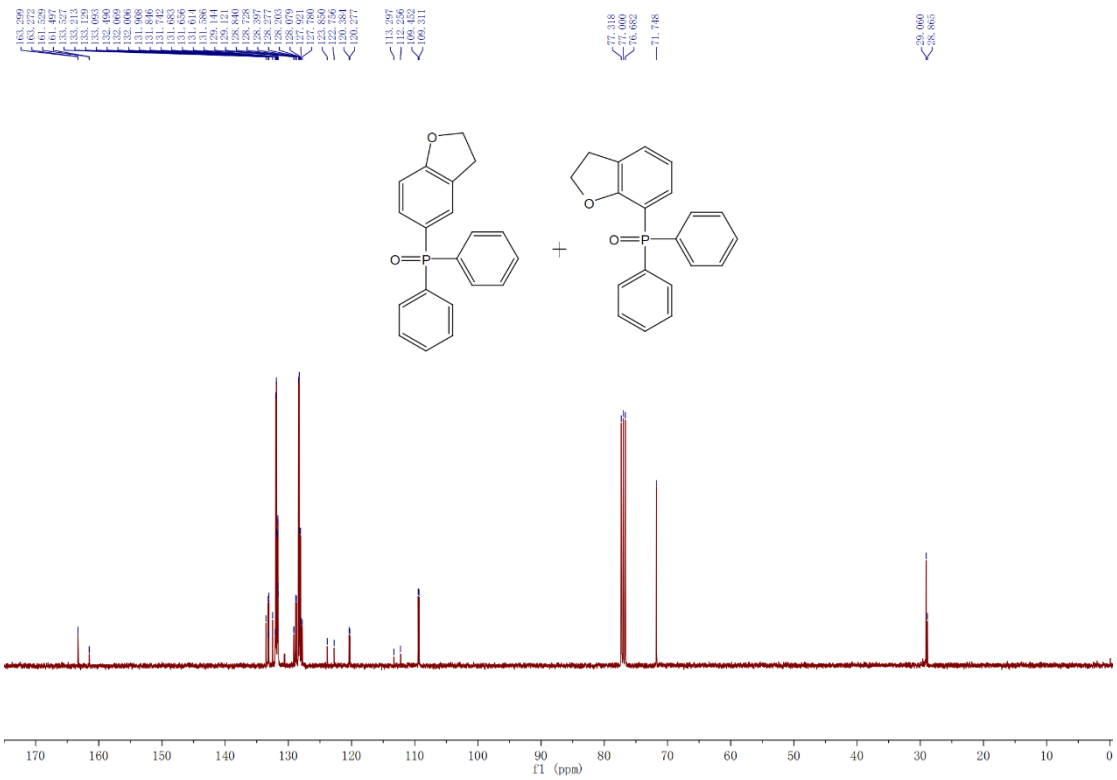
(4-methoxyphenyl)diphenylphosphine oxide (3u)

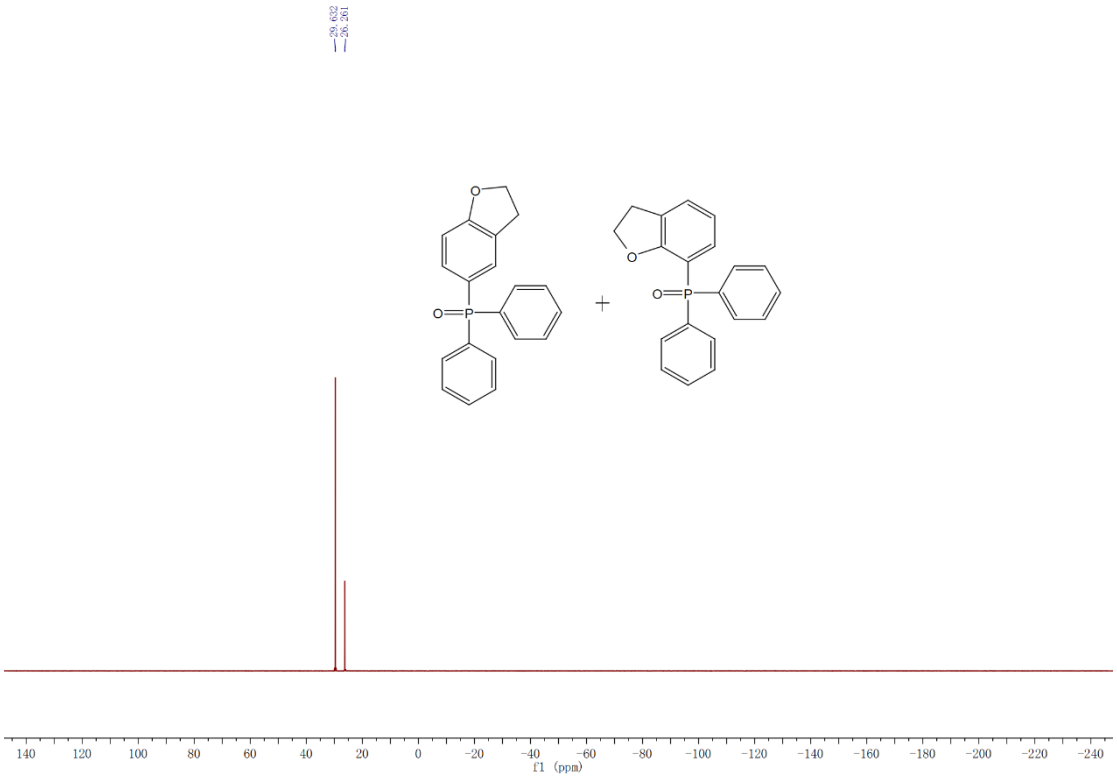




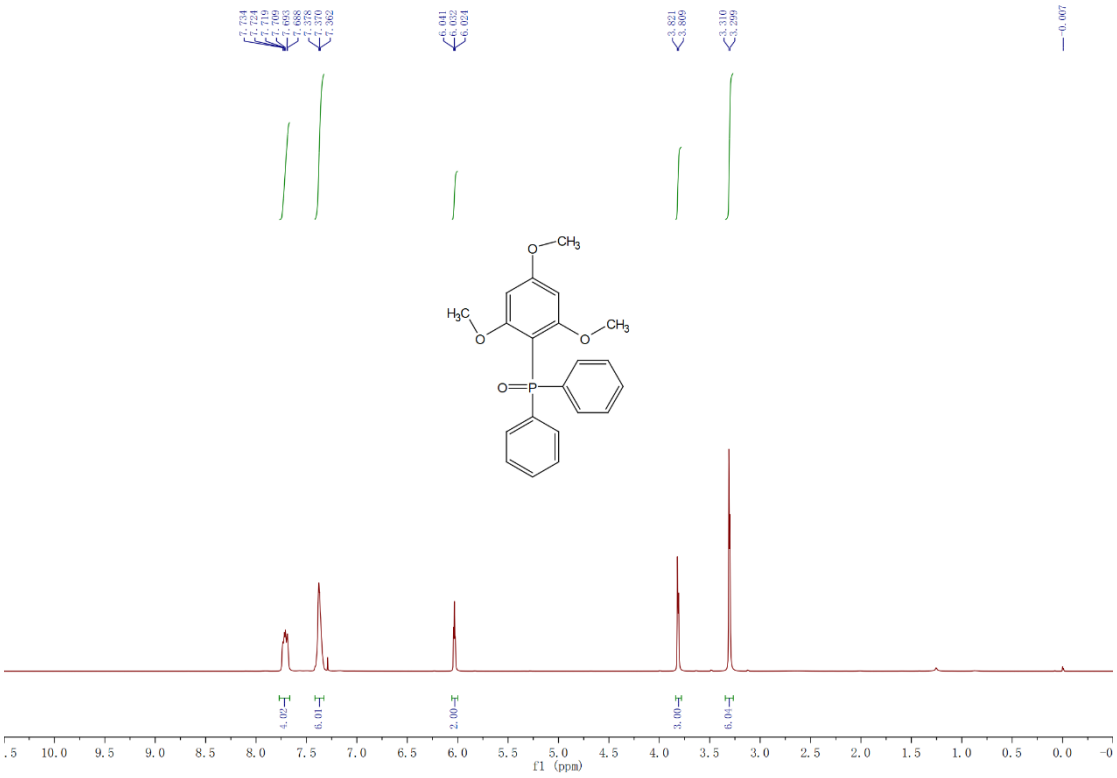
(2,3-dihydrobenzofuran-5-yl)diphenylphosphine oxide (3v)

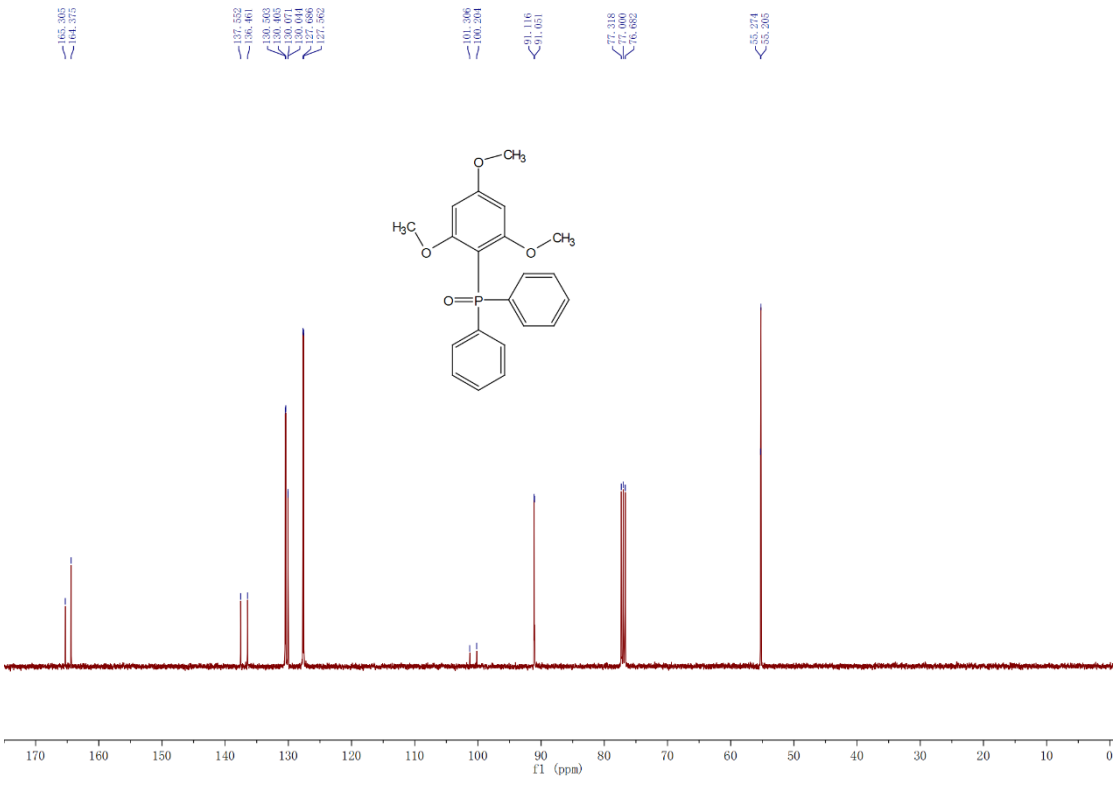


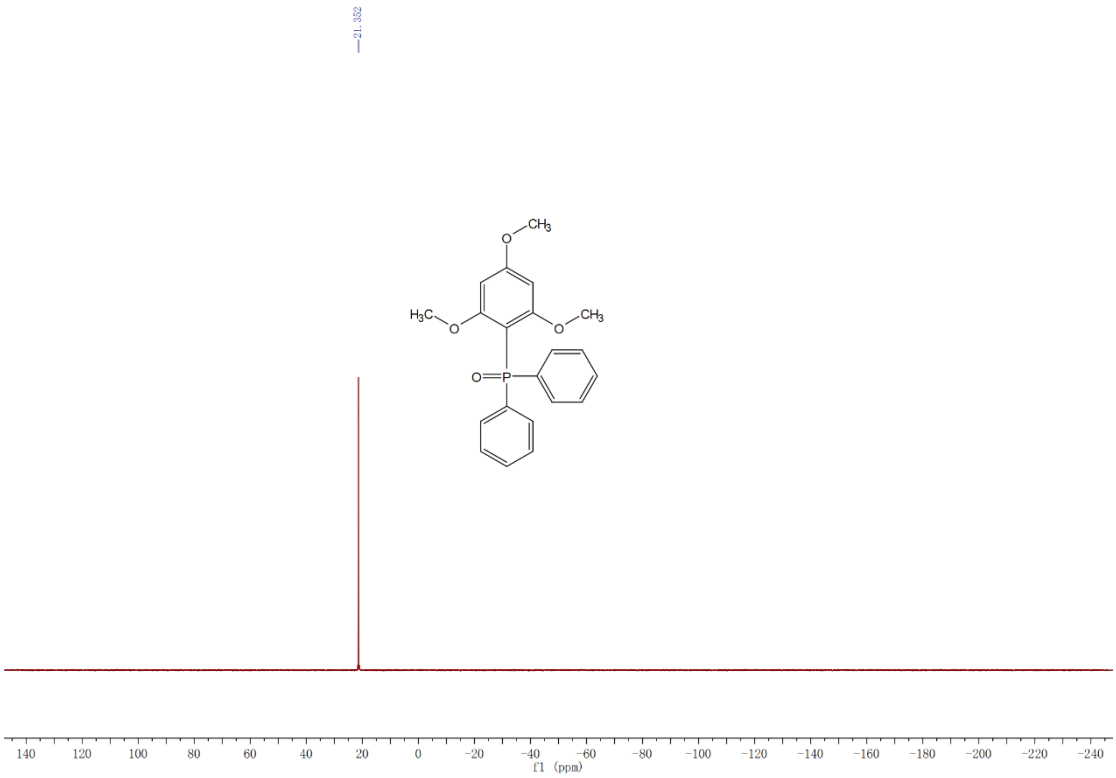

163.299
163.272
161.529
161.497
133.527
133.213
133.179
133.093
132.490
132.069
132.046
131.908
131.846
131.742
131.685
131.656
131.614
131.386
129.144
129.111
128.840
128.728
128.397
128.377
128.203
128.079
127.921
127.789
123.858
122.756
120.384
120.277
113.297
112.256
109.432
109.311
77.318
77.000
76.682
71.748
29.060
28.865
O
O=P
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


29.632
26.261
O
O=P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

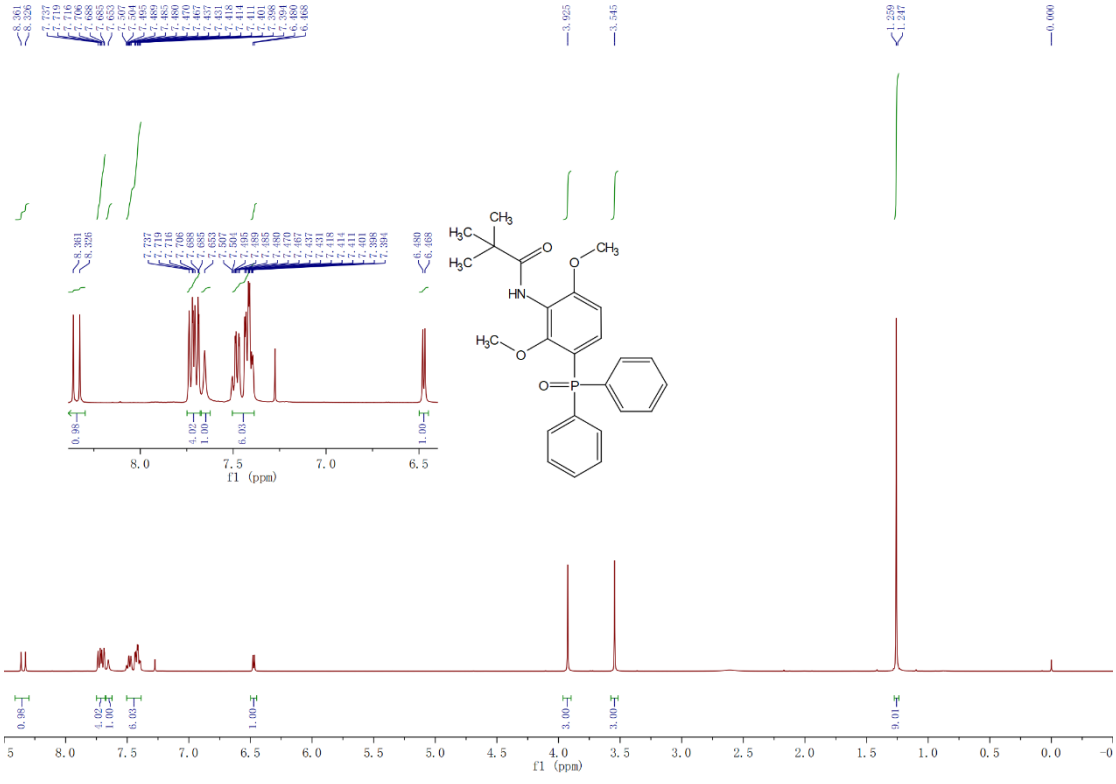
diphenyl(2,4,6-trimethoxyphenyl)phosphine oxide (3w)

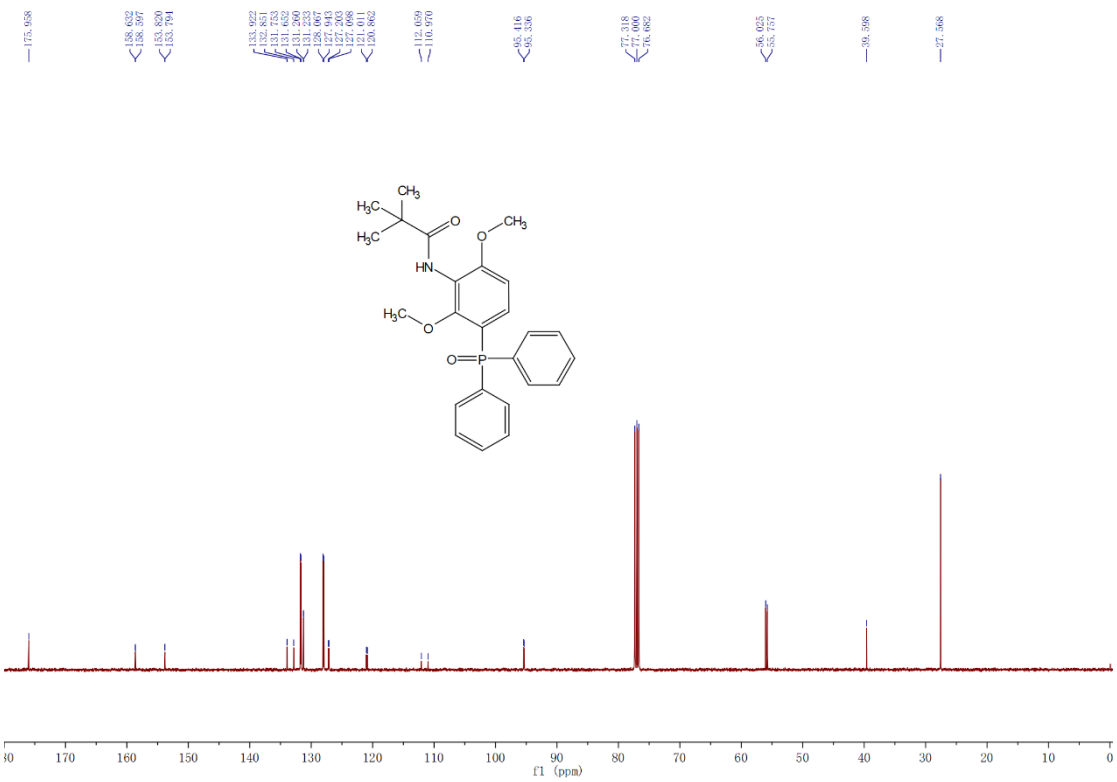


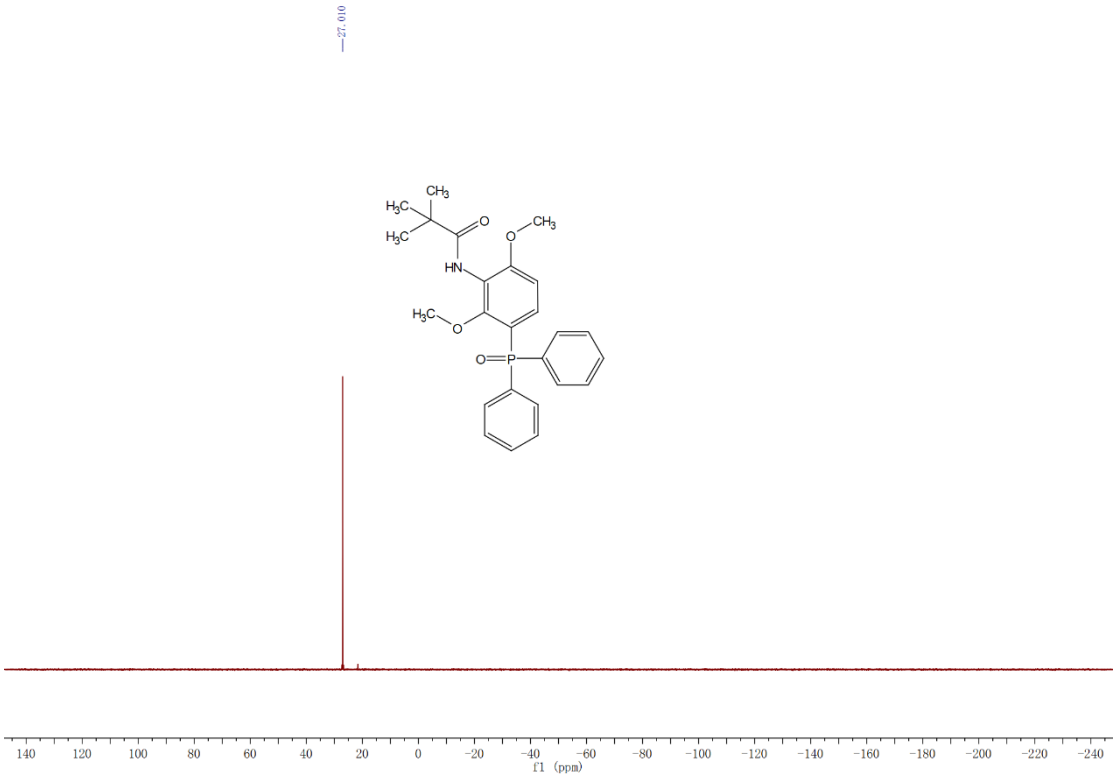




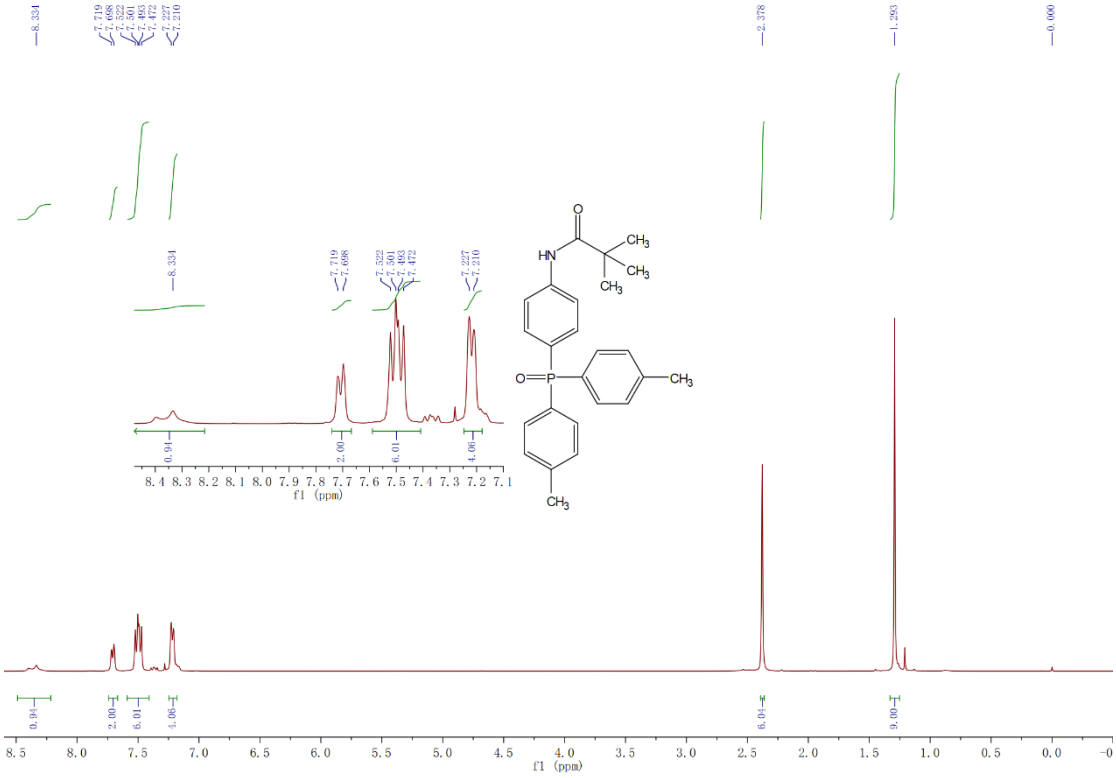
N-(3-(diphenylphosphoryl)-2,6-dimethoxyphenyl)pivalamide (3x)

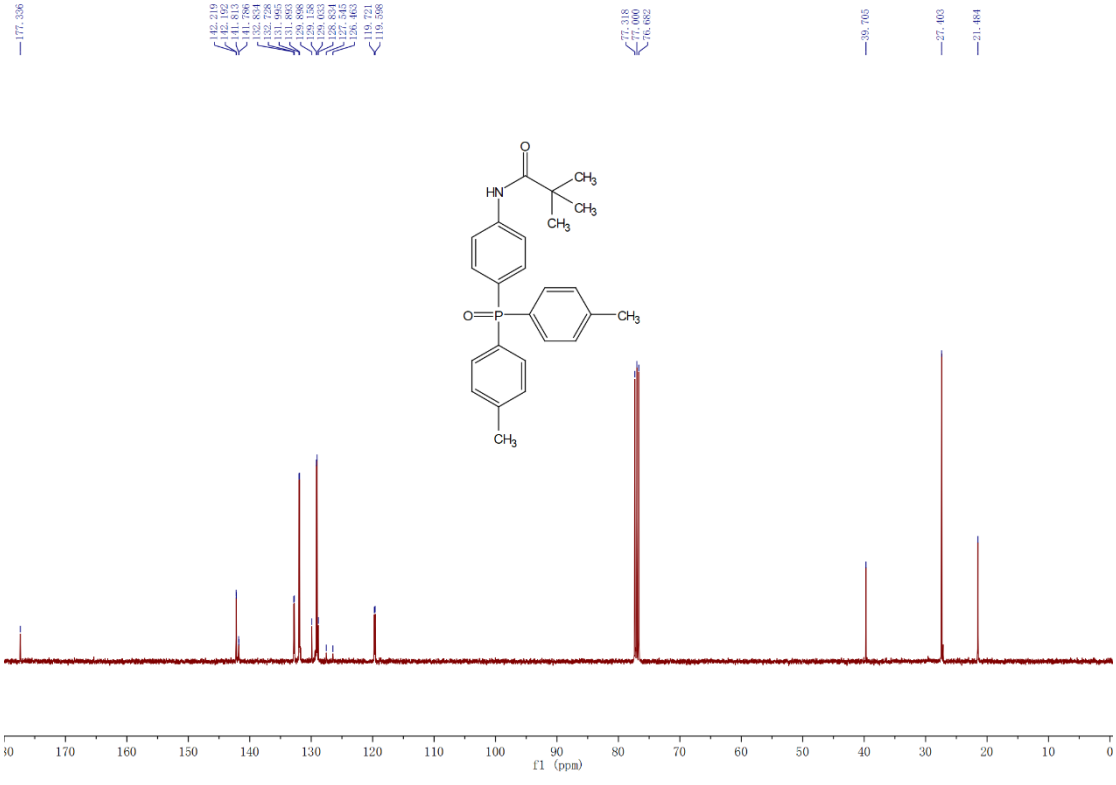


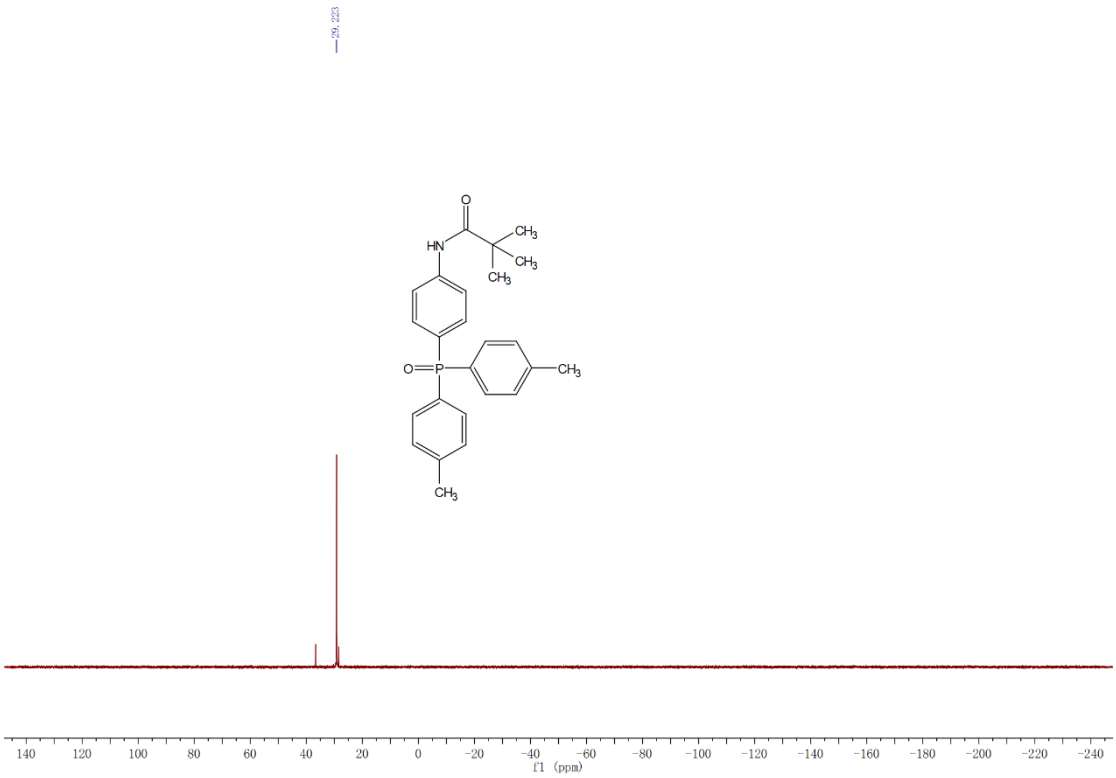

175.958
158.632
158.597
153.820
153.794
133.922
132.851
131.753
131.652
131.560
131.233
128.067
127.943
127.203
127.066
121.011
120.862
112.059
110.970
95.416
95.336
77.318
77.000
76.682
56.025
55.757
39.598
27.868
f1 (ppm)


27.010
f1 (ppm)

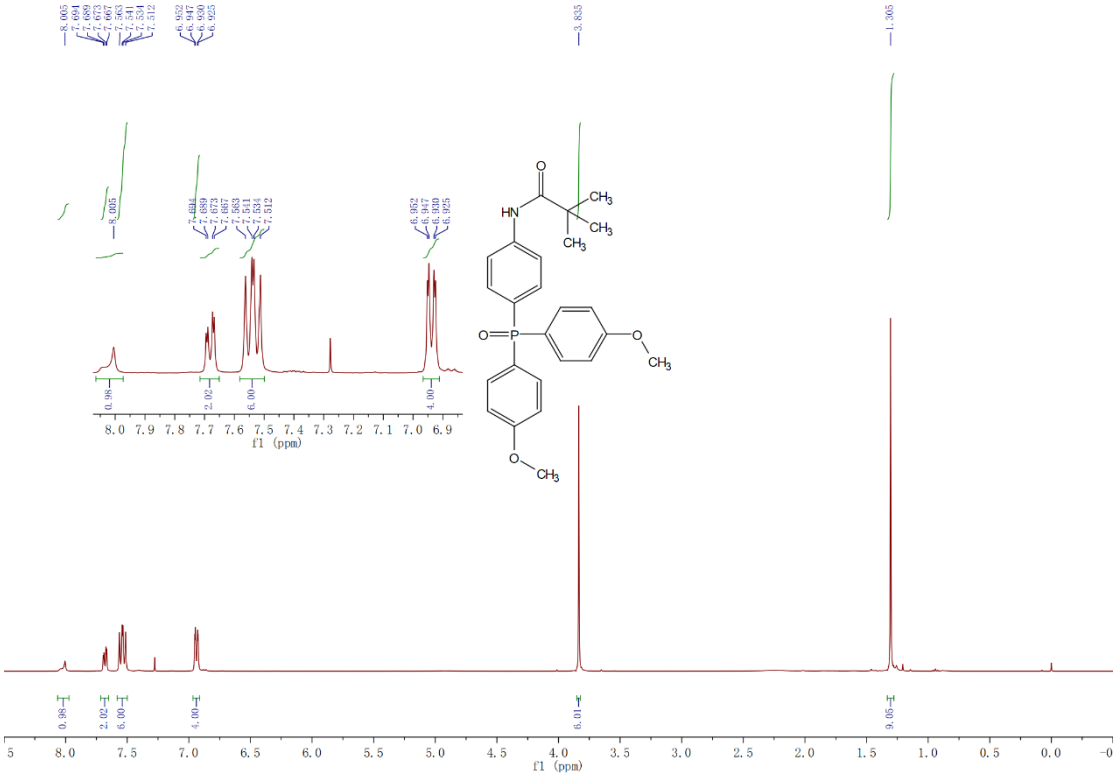
N-(4-(di-p-tolylphosphoryl)phenyl)pivalamide (3y)

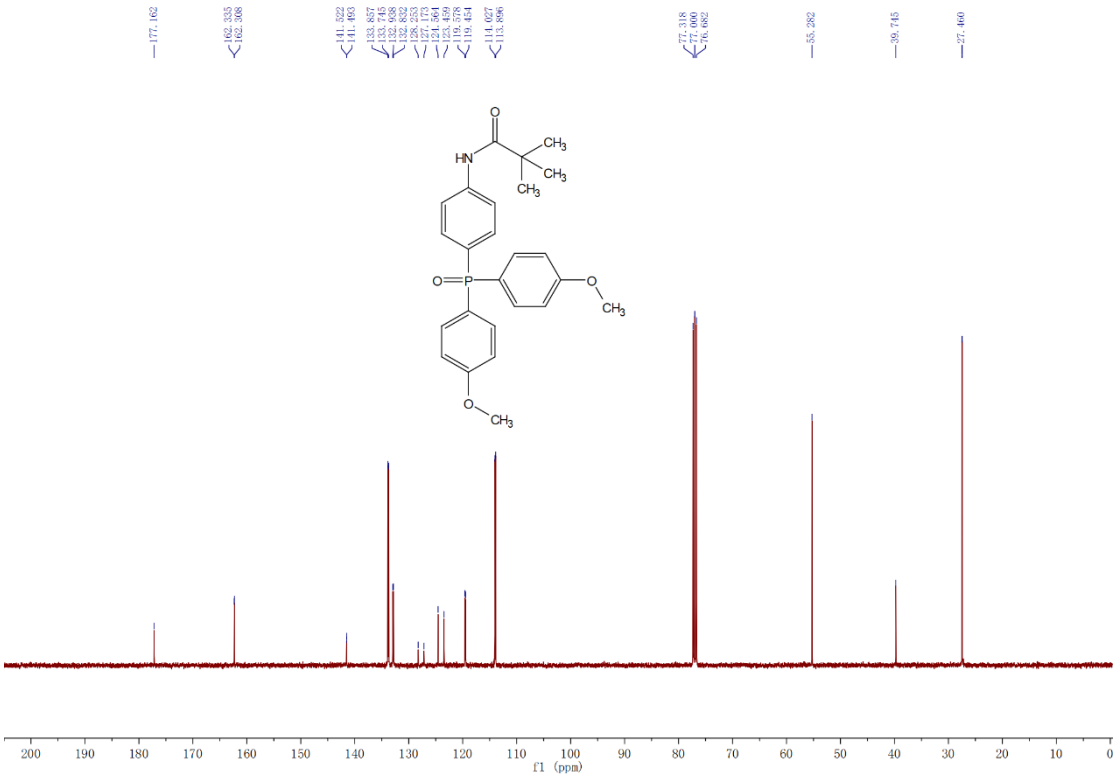


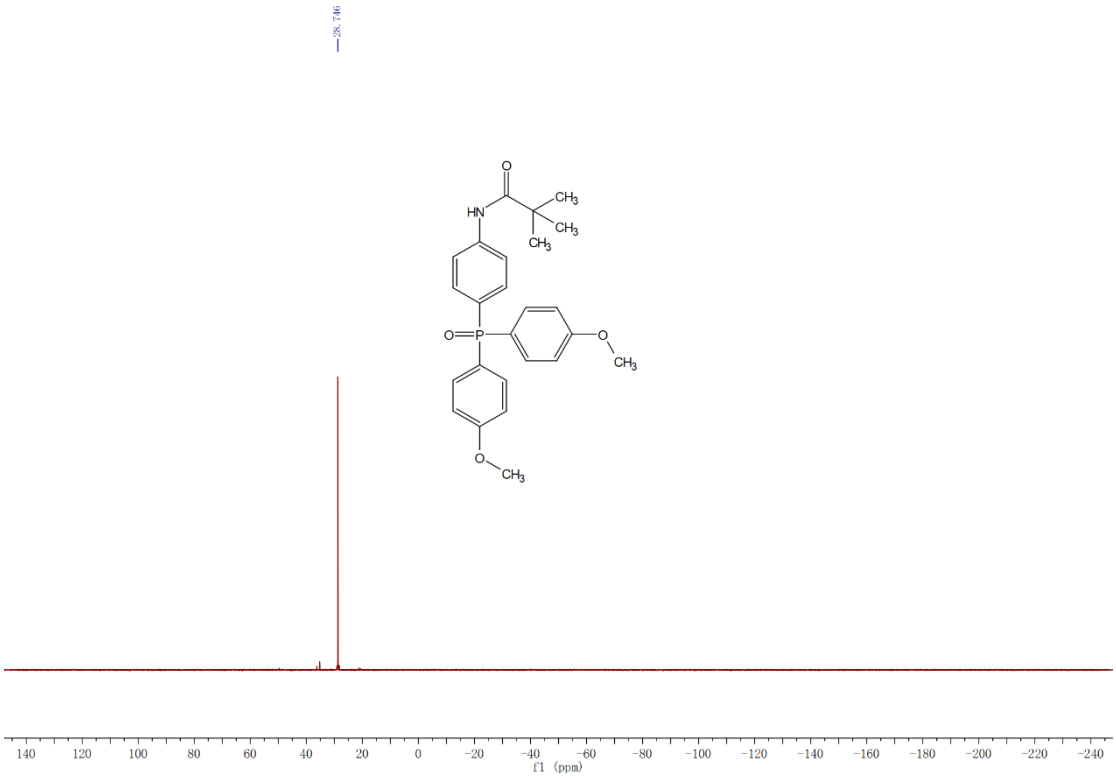




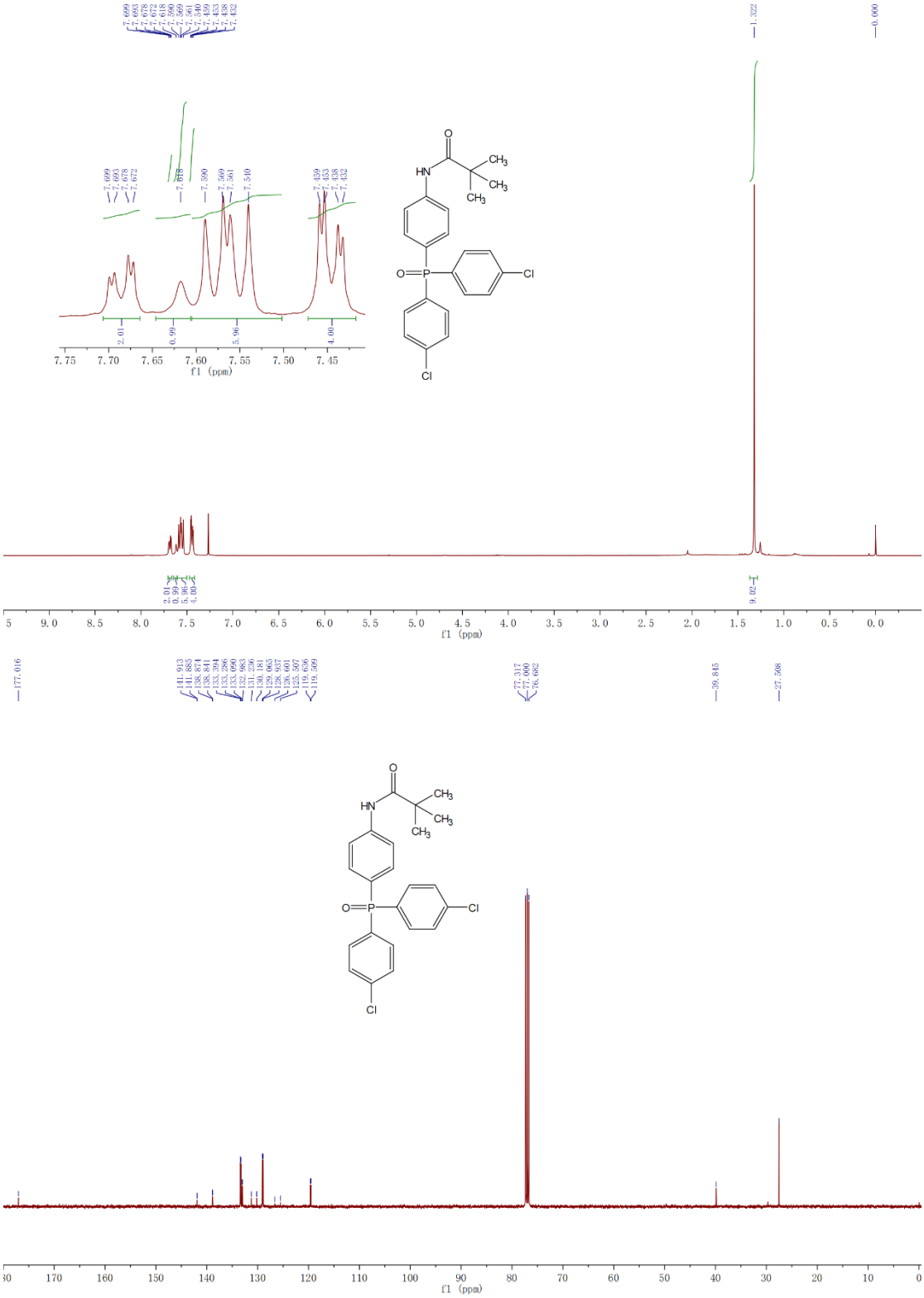
N-(4-(bis(4-methoxyphenyl)phosphoryl)phenyl)pivalamide (3z)

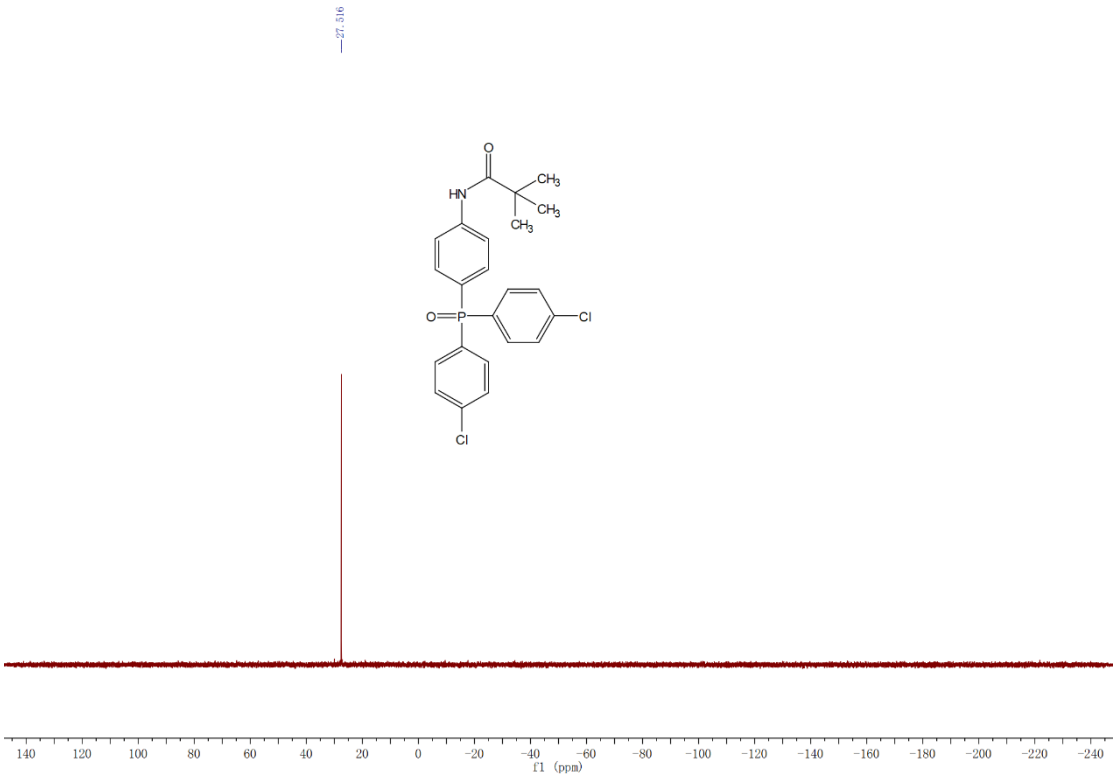



177.162
162.335
162.308
141.522
141.495
133.857
133.745
132.498
132.432
128.253
127.173
124.964
123.439
119.578
119.454
114.027
113.896
77.318
77.000
76.682
55.282
39.745
27.460
HN
O
CH3
CH3
CH3
O=P
O
CH3
O
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

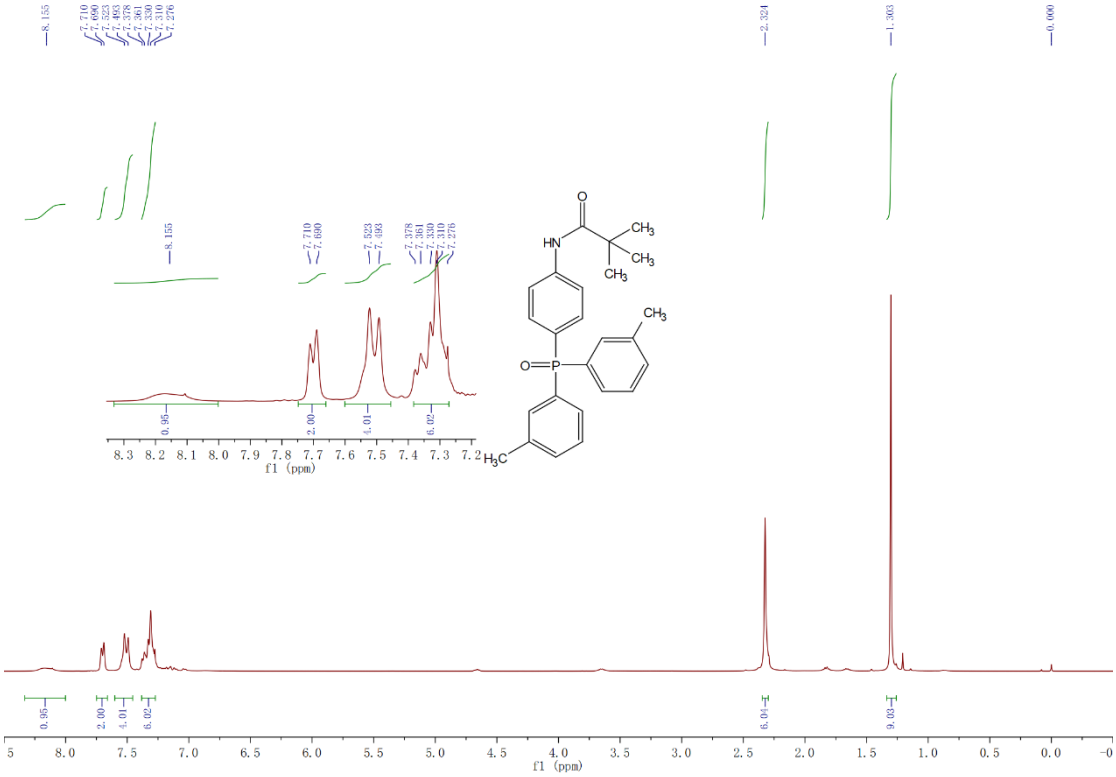

28.746
HN
O
CH3
CH3
CH3
O=P
O
CH3
O
CH3
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

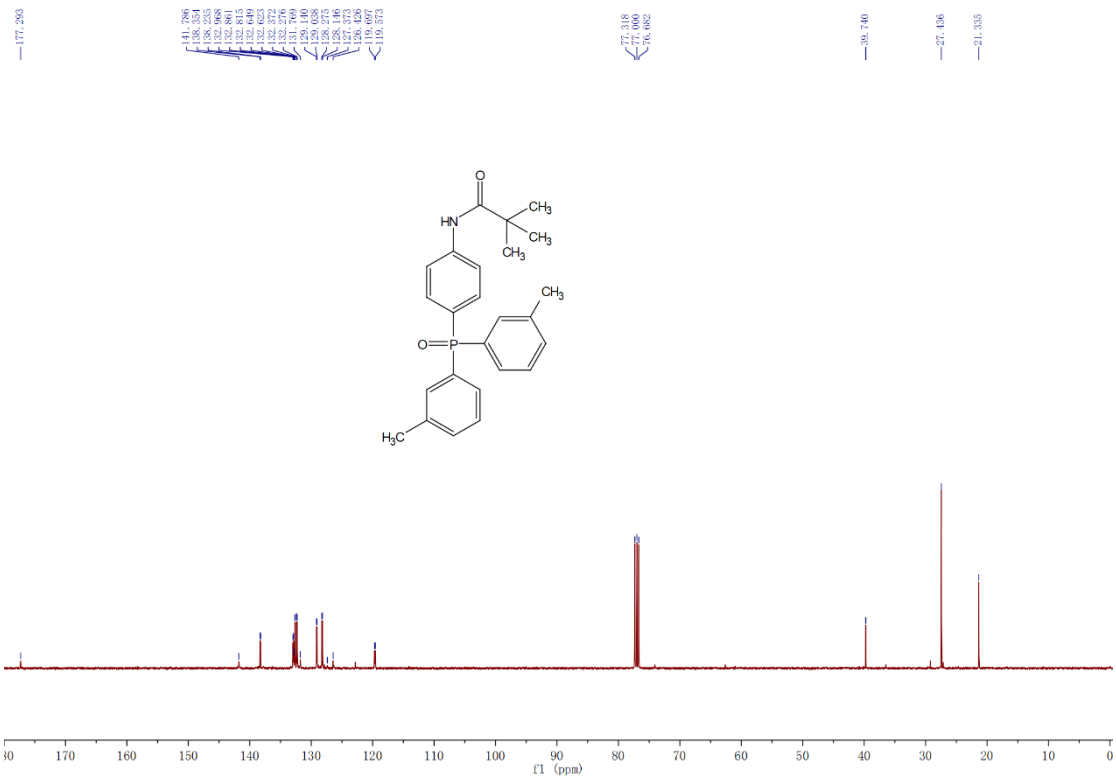
N-(4-(bis(4-chlorophenyl)phosphoryl)phenyl)pivalamide (3aa)

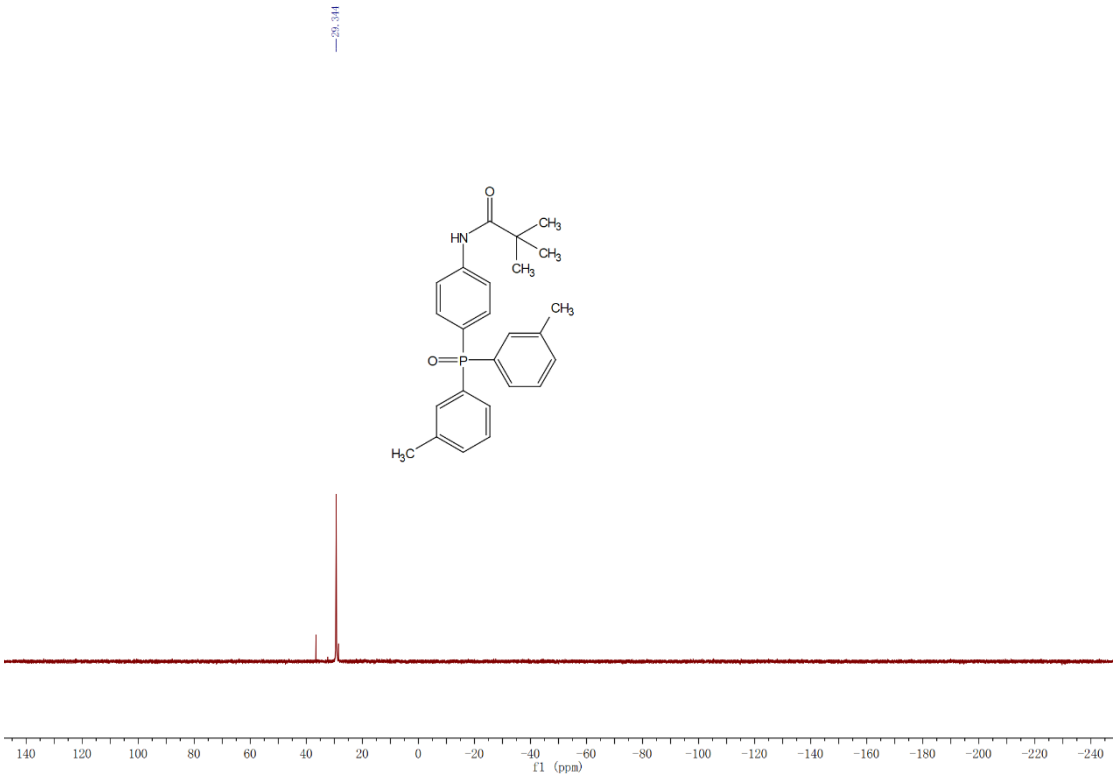




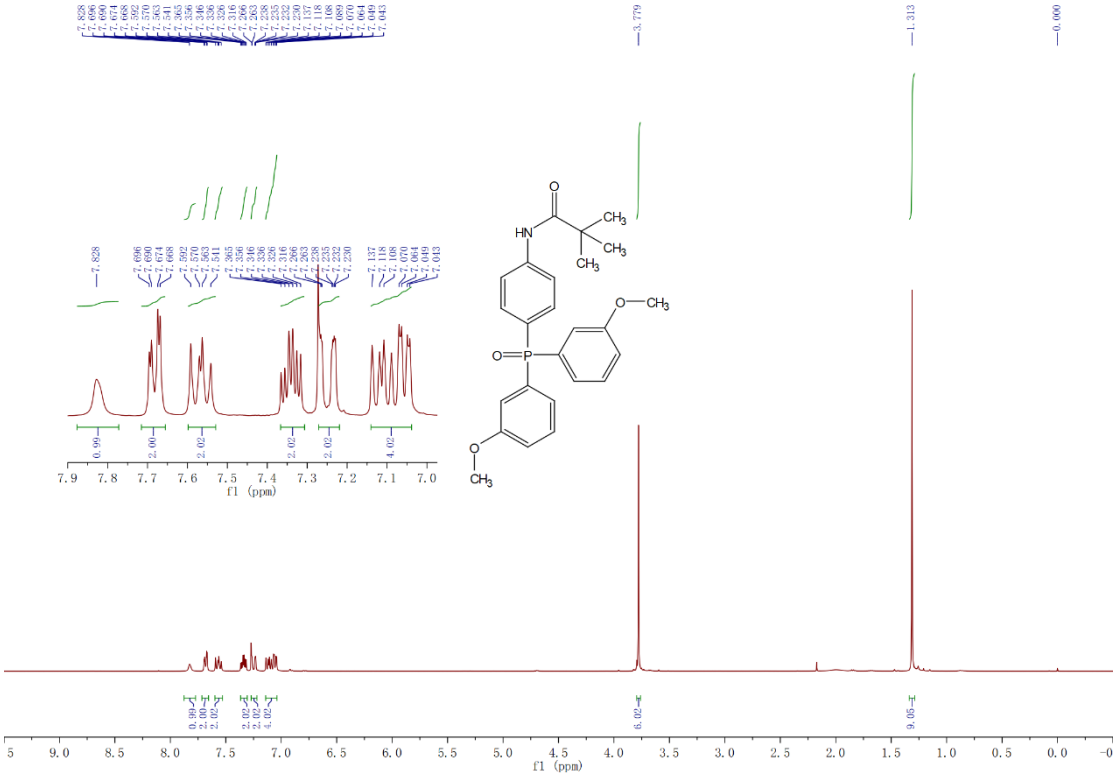
N-(4-(di-m-tolylphosphoryl)phenyl)pivalamide (3ab)

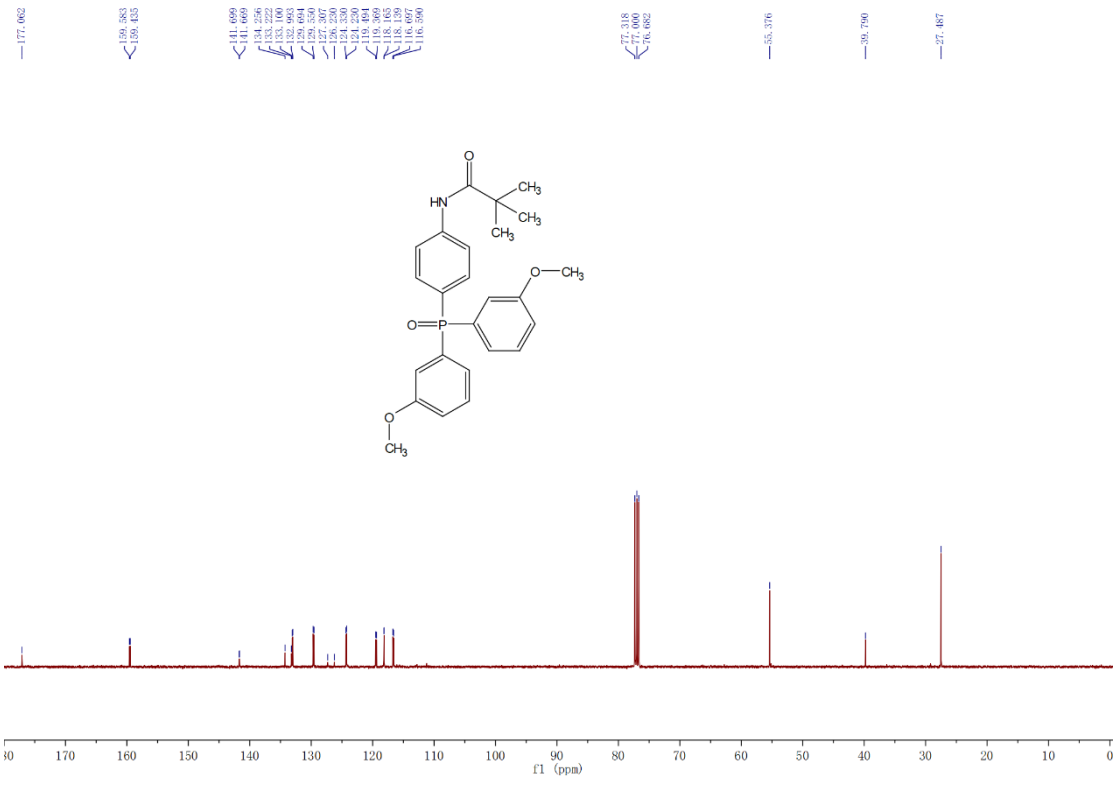


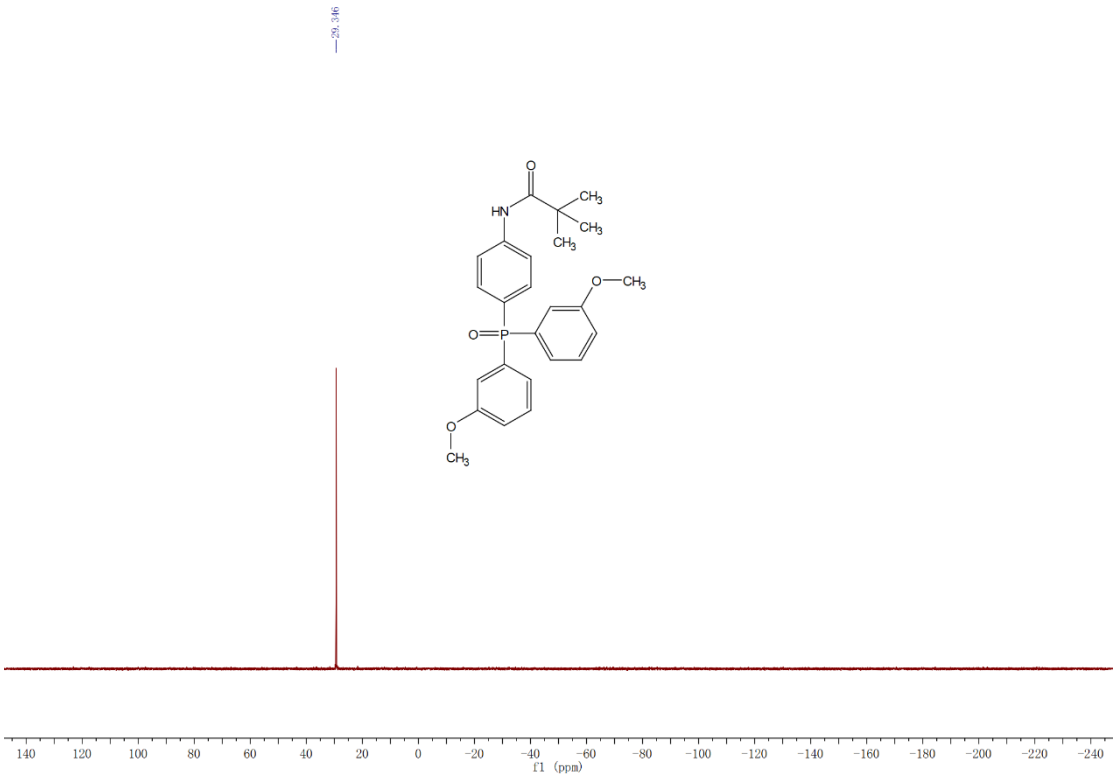

177.293
141.786
138.384
138.235
132.968
132.861
132.815
132.649
132.623
132.372
132.276
131.769
129.140
129.038
128.275
128.146
127.373
126.426
119.697
119.573
77.318
77.000
76.682
39.740
27.436
21.335
HN
O
CH3
CH3
CH3
CH3
O=P
H3C
f1 (ppm)


29.344
HN
O
CH3
CH3
CH3
CH3
O=P
H3C
f1 (ppm)

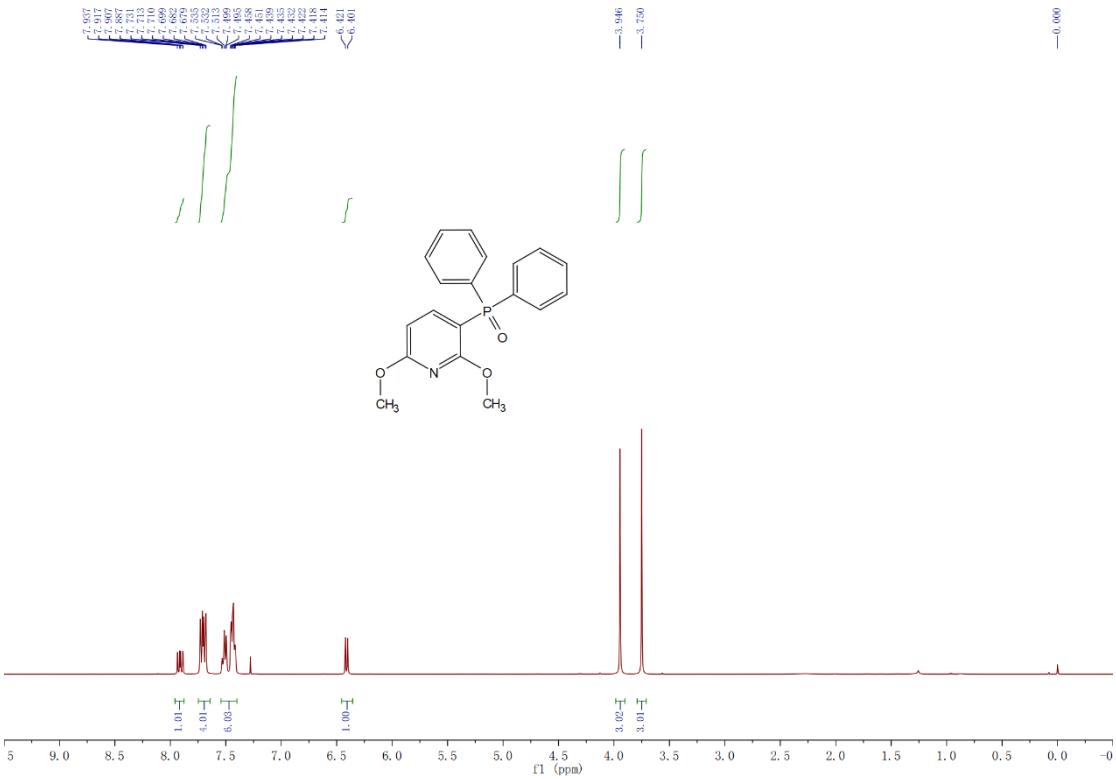
N-(4-(bis(3-methoxyphenyl)phosphoryl)phenyl)pivalamide (3ac)

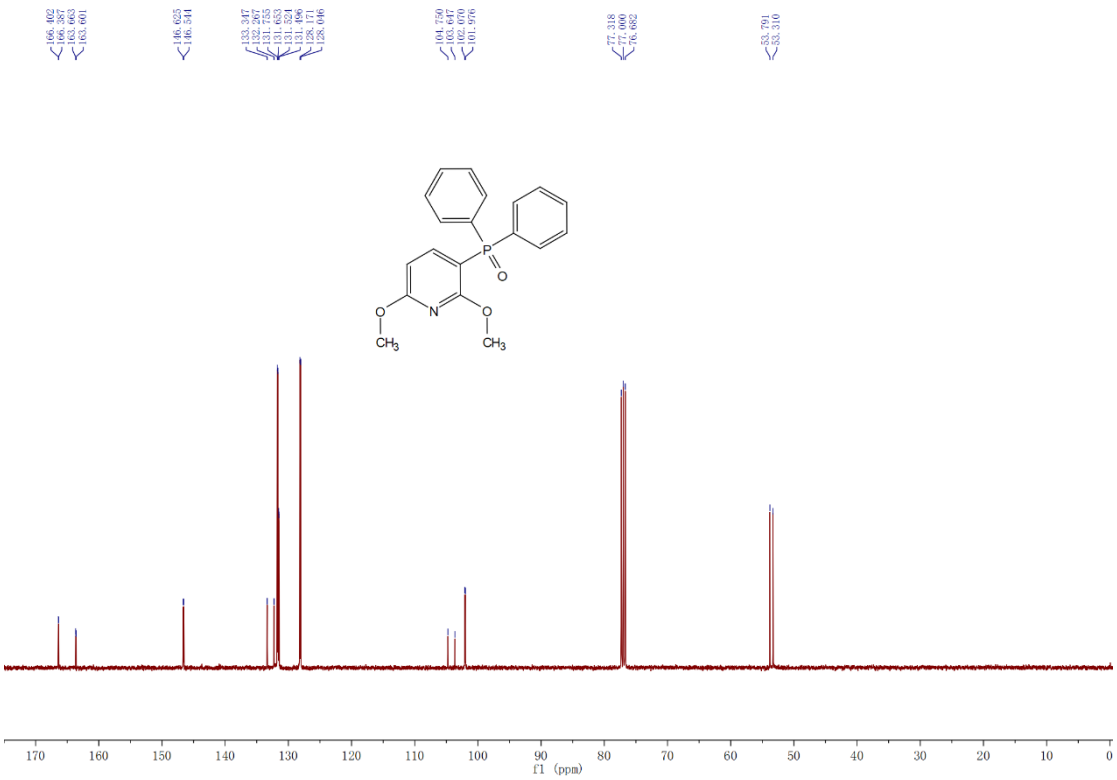


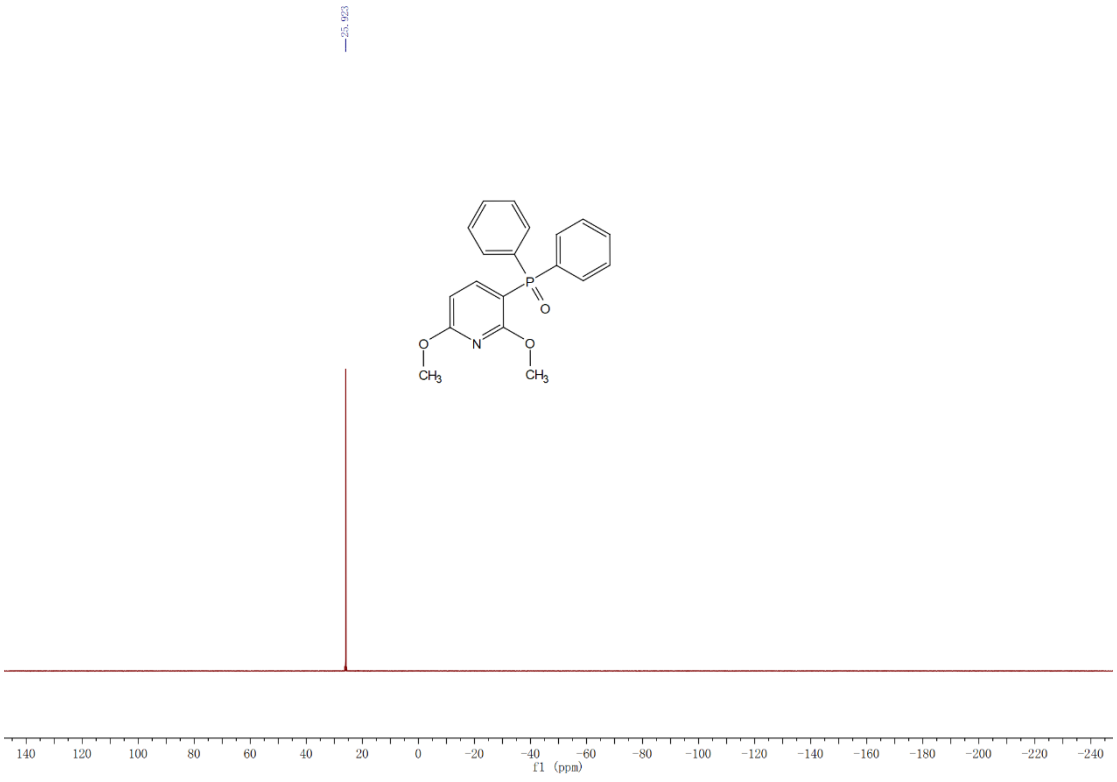




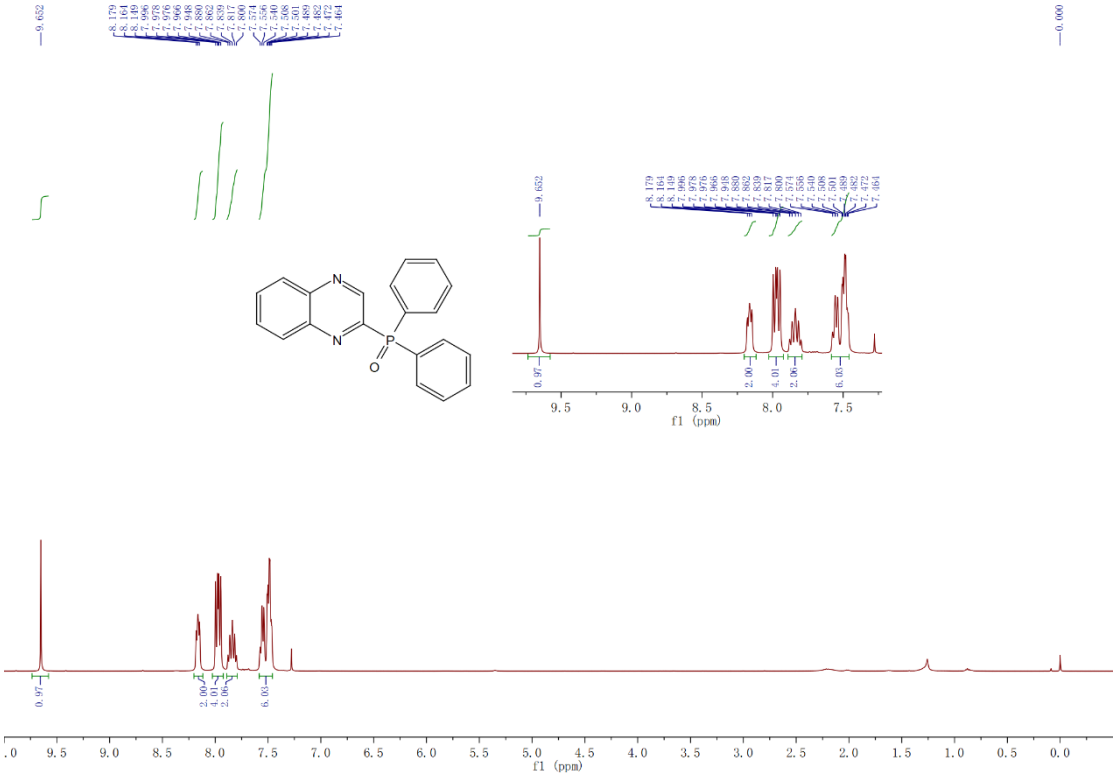
(2,6-dimethoxypyridin-3-yl)diphenylphosphine oxide (3ad)

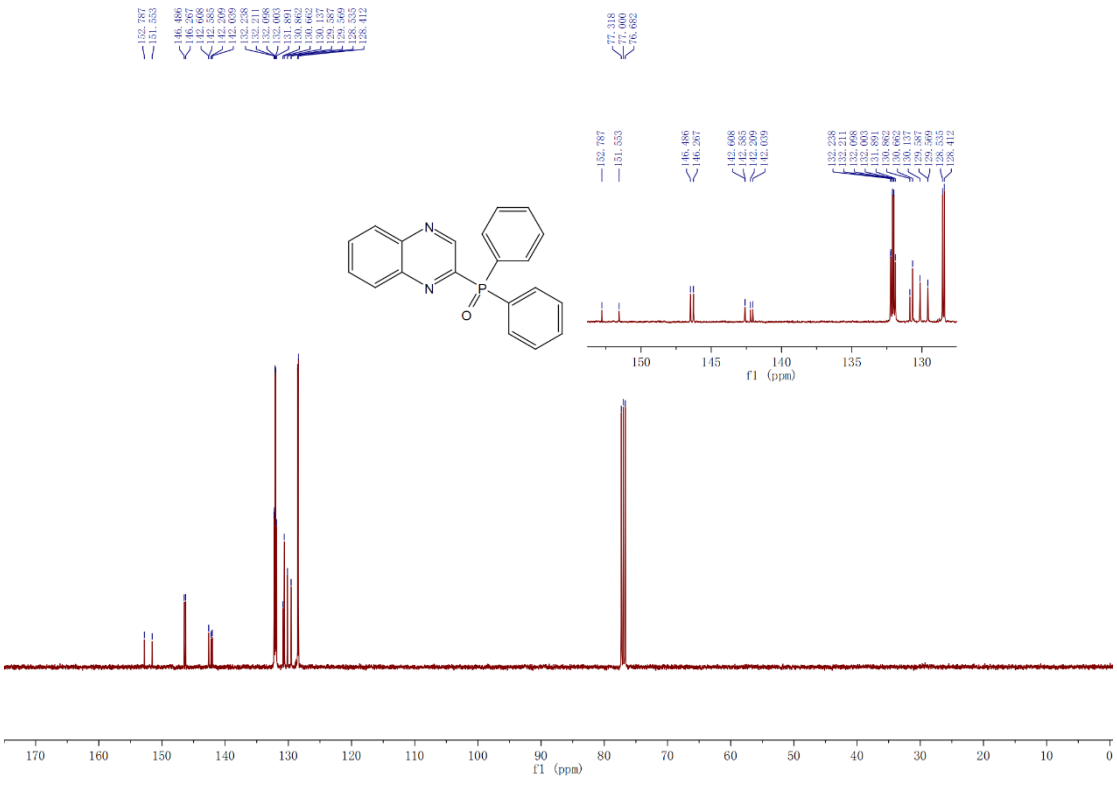


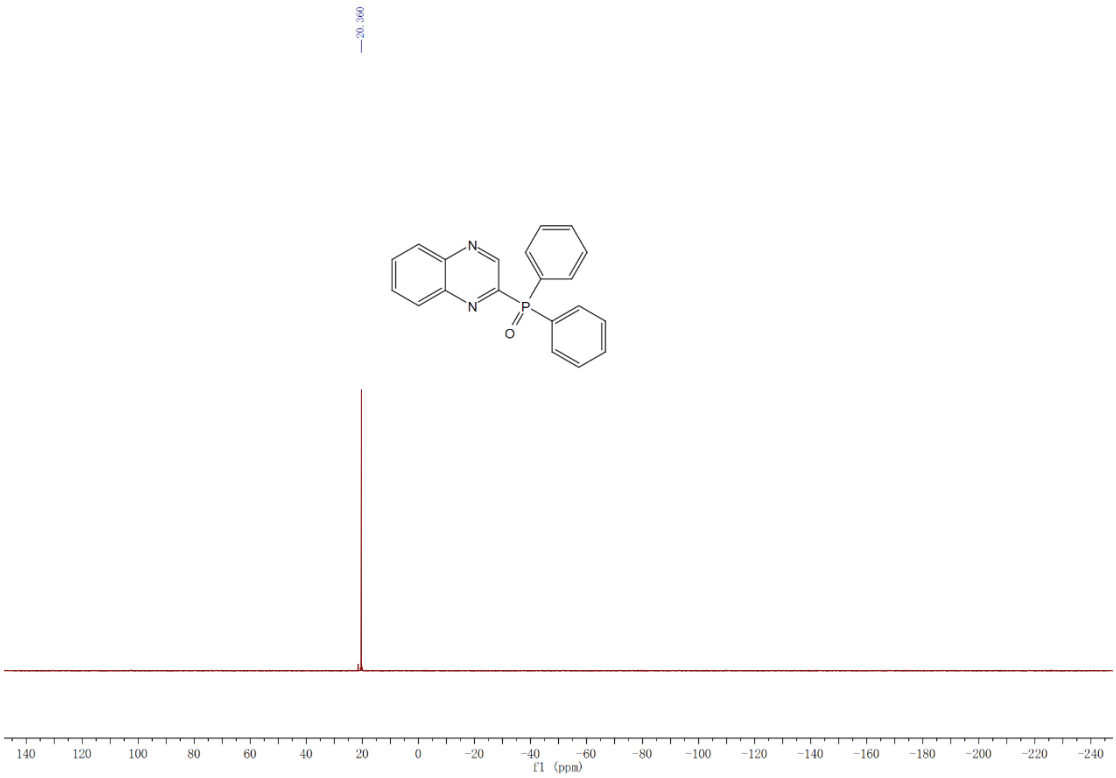

166.402
166.387
163.668
163.601
146.625
146.544
133.347
132.267
131.755
131.653
131.524
131.496
128.171
128.046
104.750
102.447
102.070
101.976
77.318
77.000
76.682
53.791
53.310
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


25.923
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

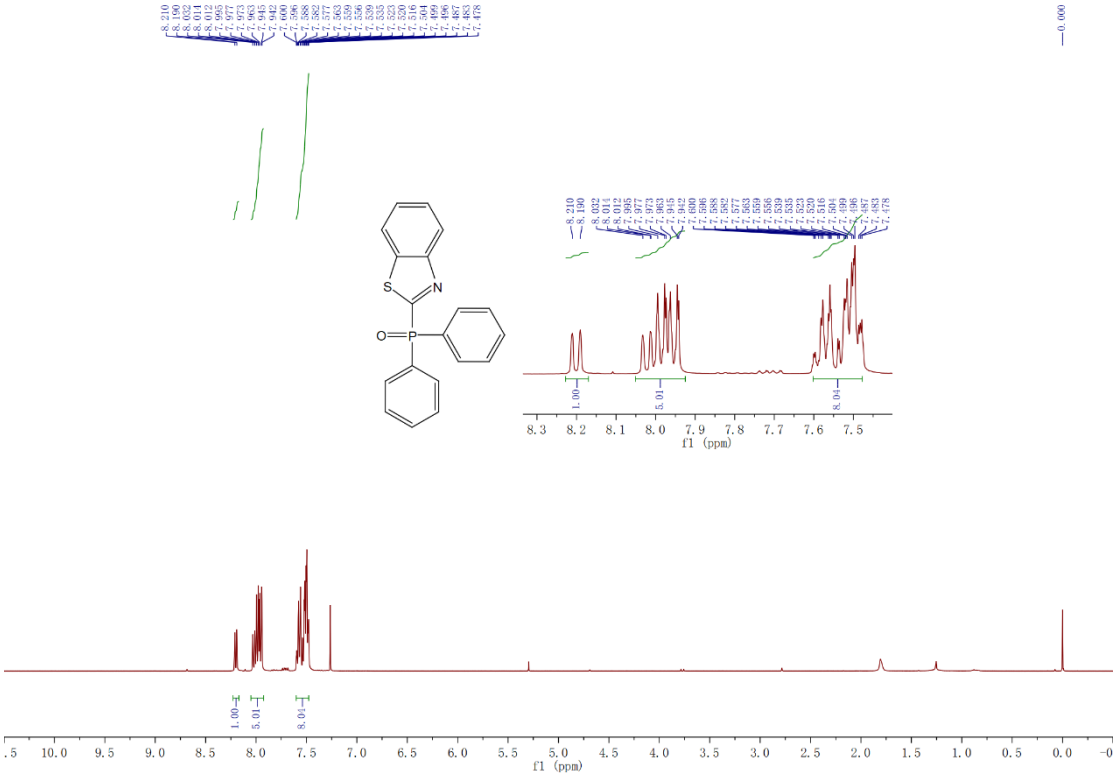
diphenyl(quinoxalin-2-yl)phosphine oxide (3ae)

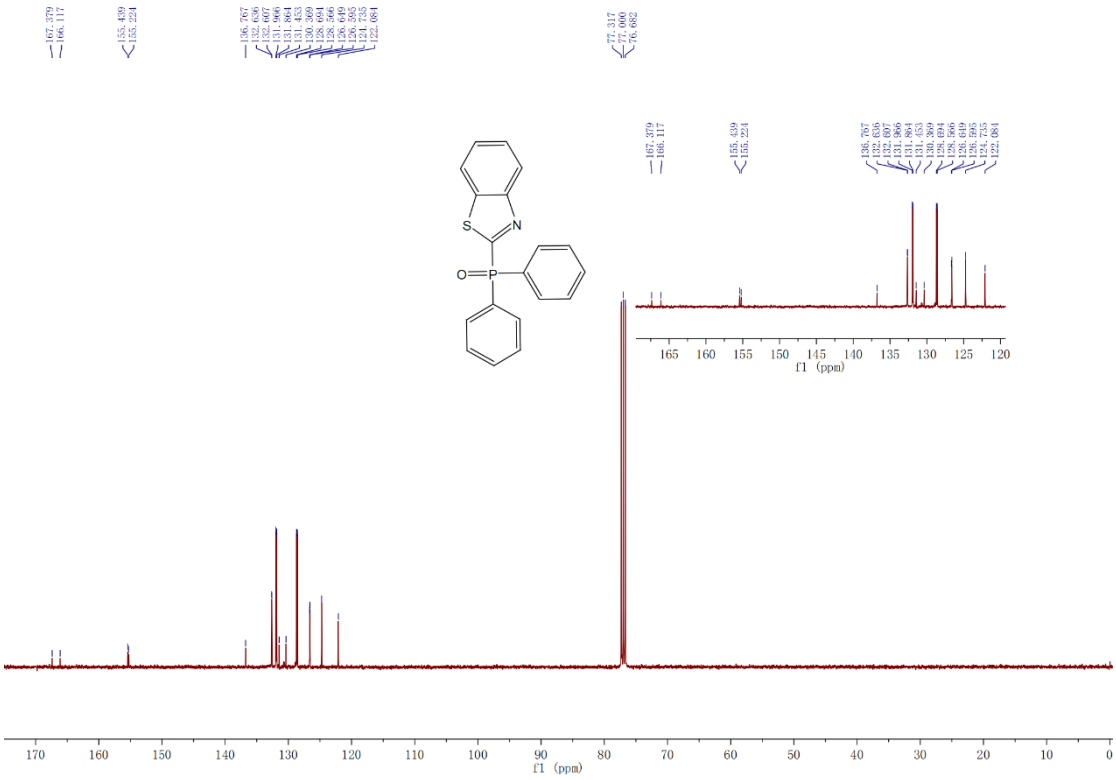


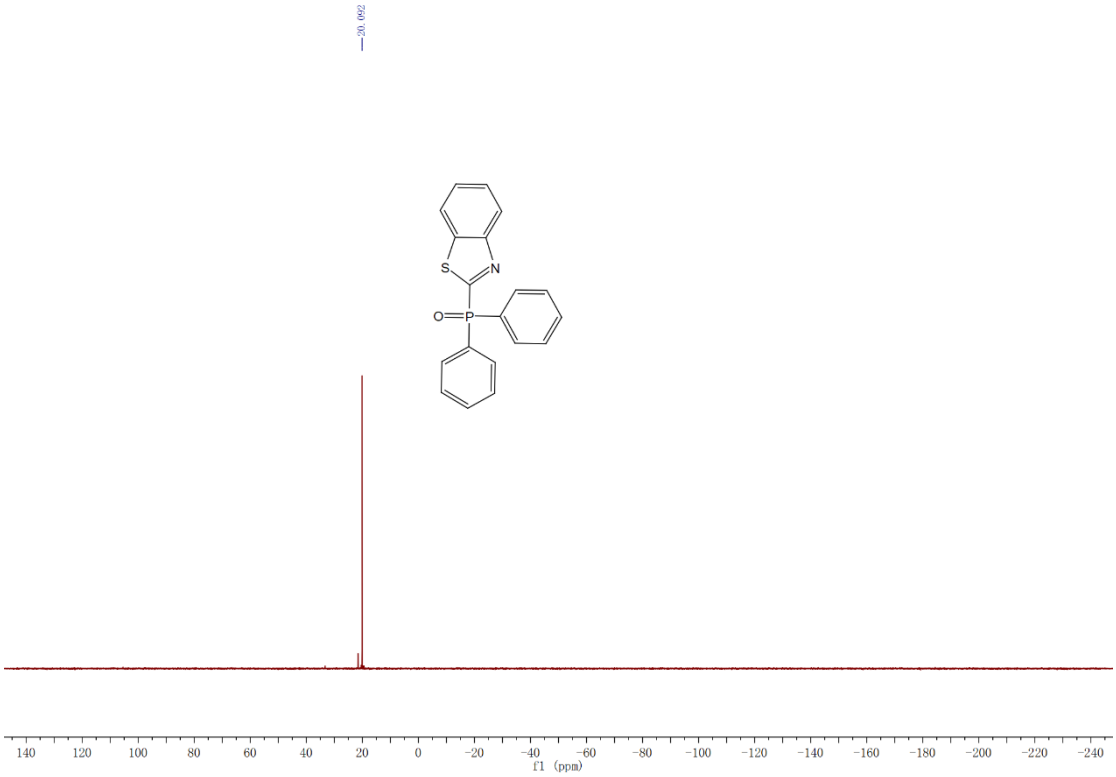




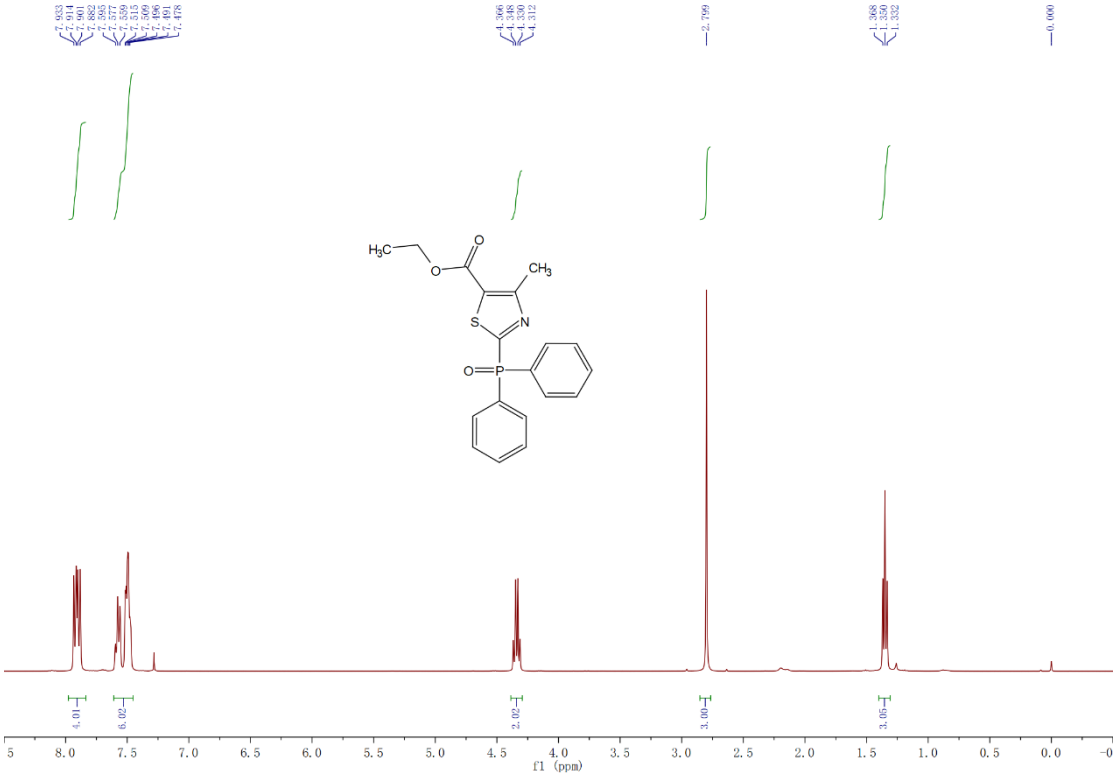
benzo[d]thiazol-2-yldiphenylphosphine oxide (3af)

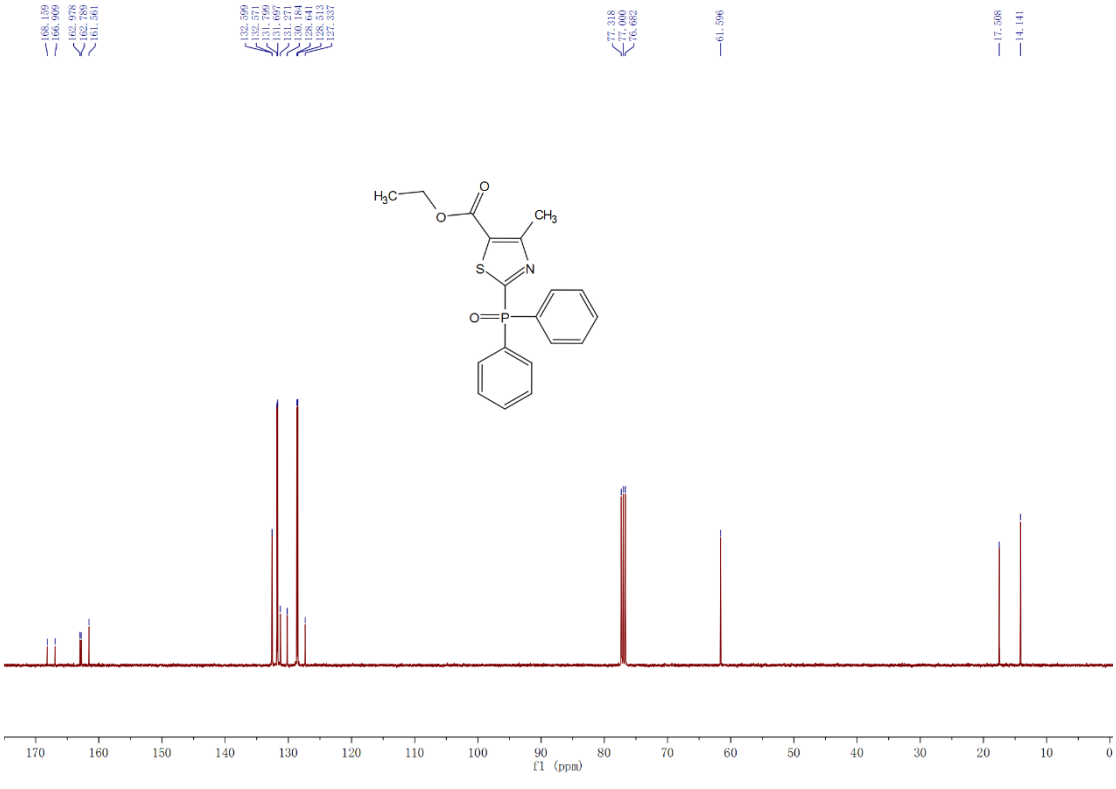


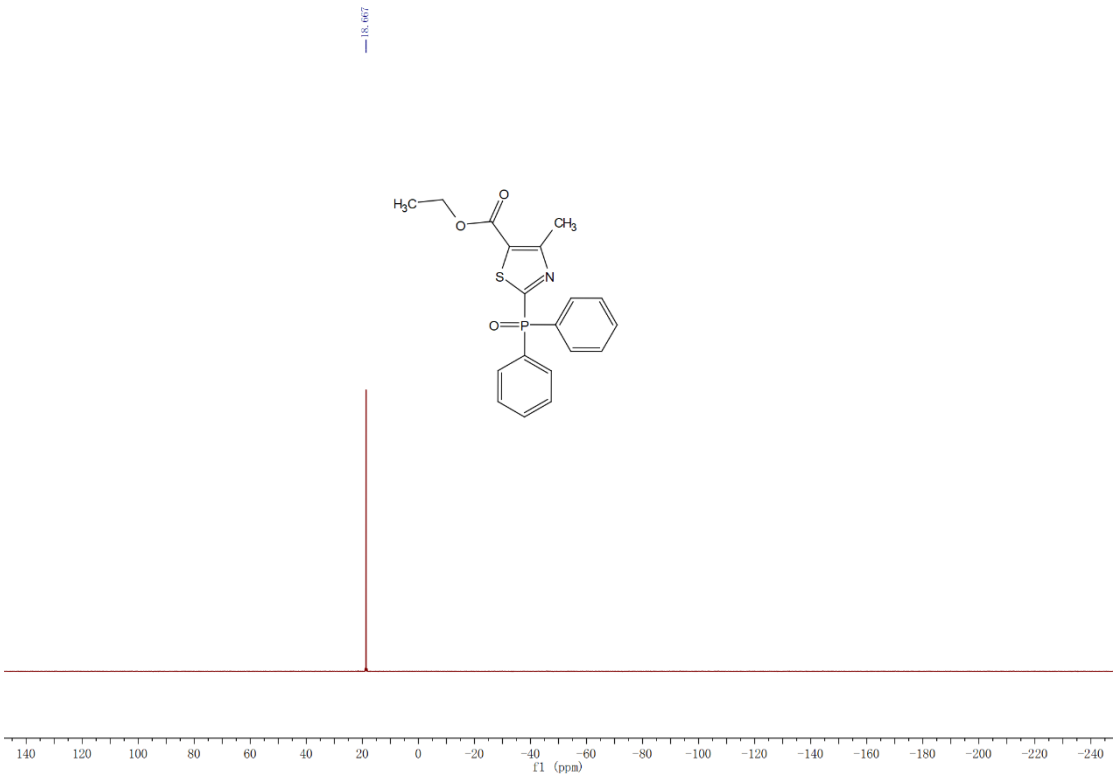

167.379
166.117
155.439
155.224
136.767
132.636
132.607
131.966
131.864
131.453
130.369
128.694
128.566
126.649
126.595
124.735
122.084
77.317
77.000
76.682
S
N
O=P
165 160 155 150 145 140 135 130 125 120
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


20.092
S
N
O=P
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240
f1 (ppm)

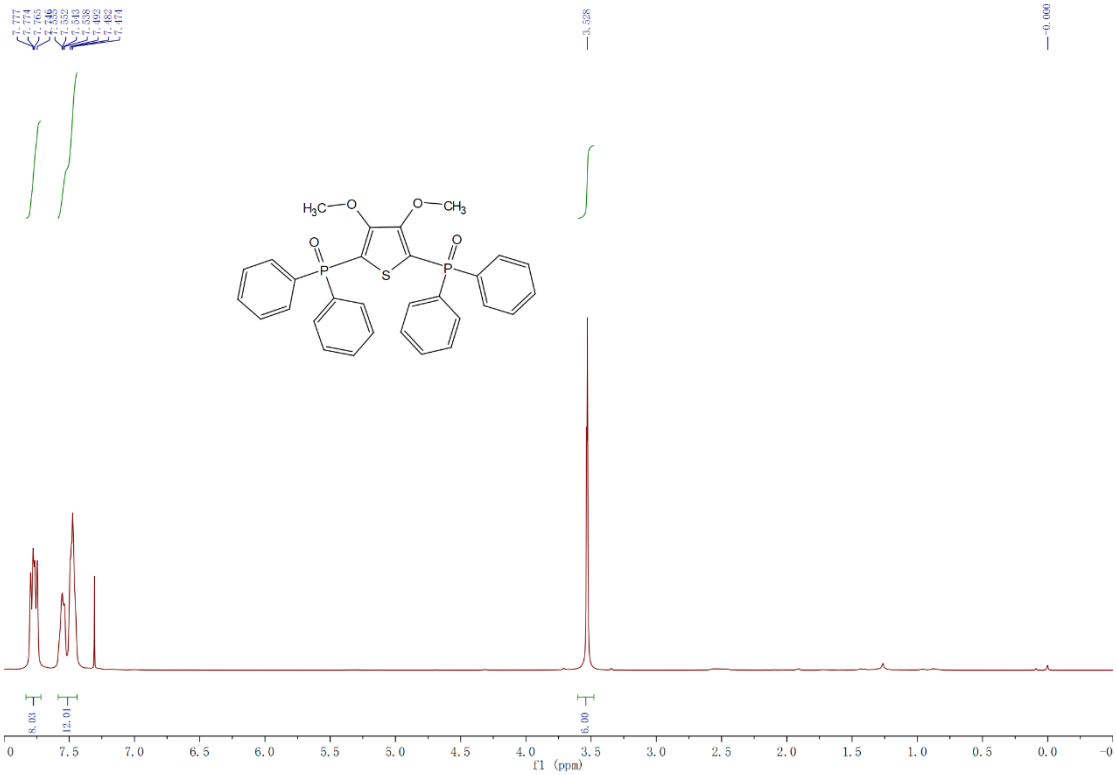
ethyl 2-(diphenylphosphoryl)-4-methylthiazole-5-carboxylate (3ag)

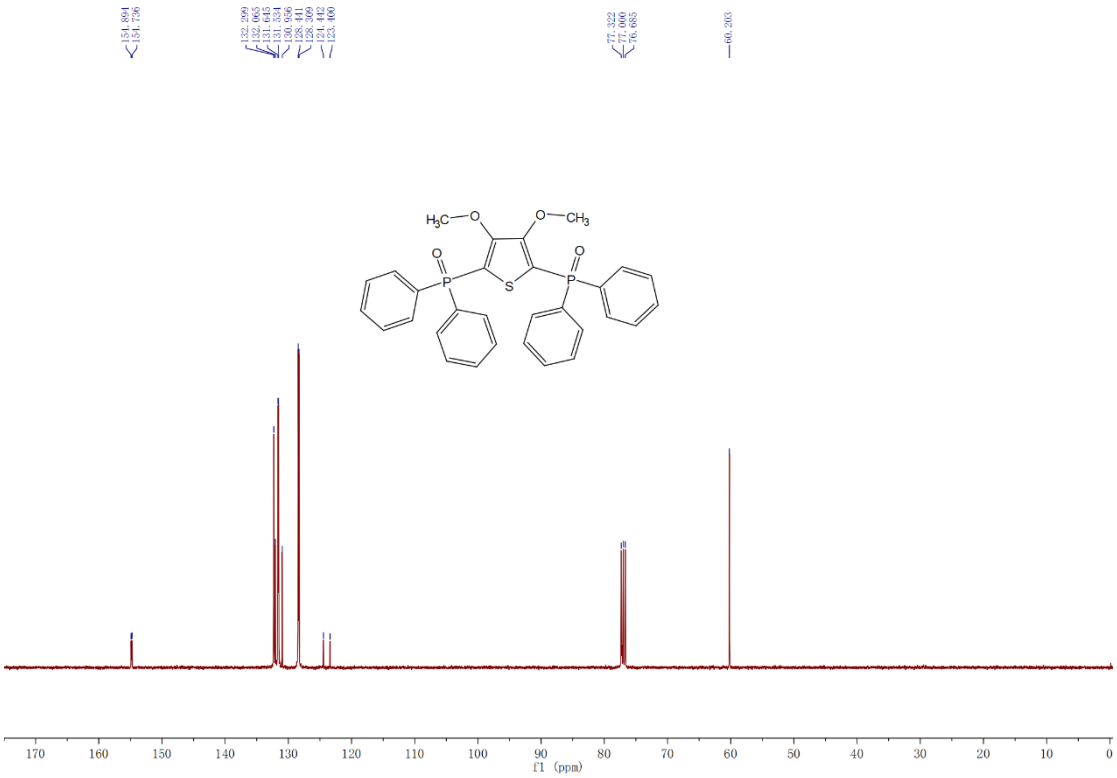


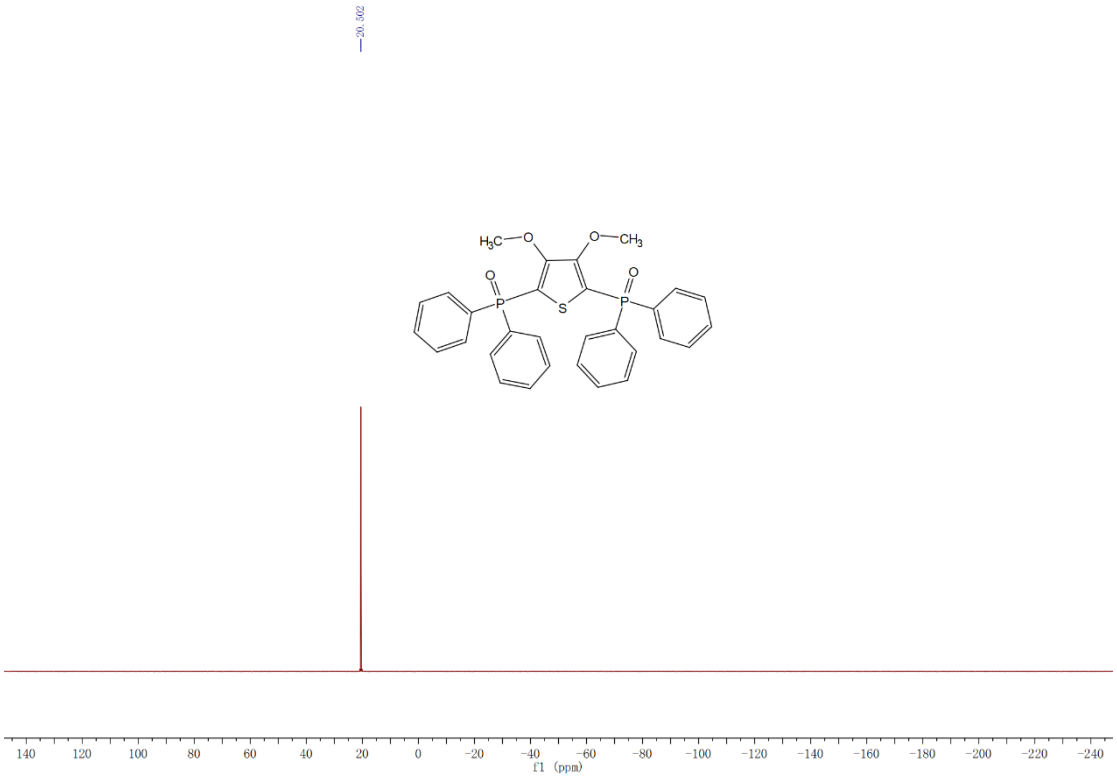




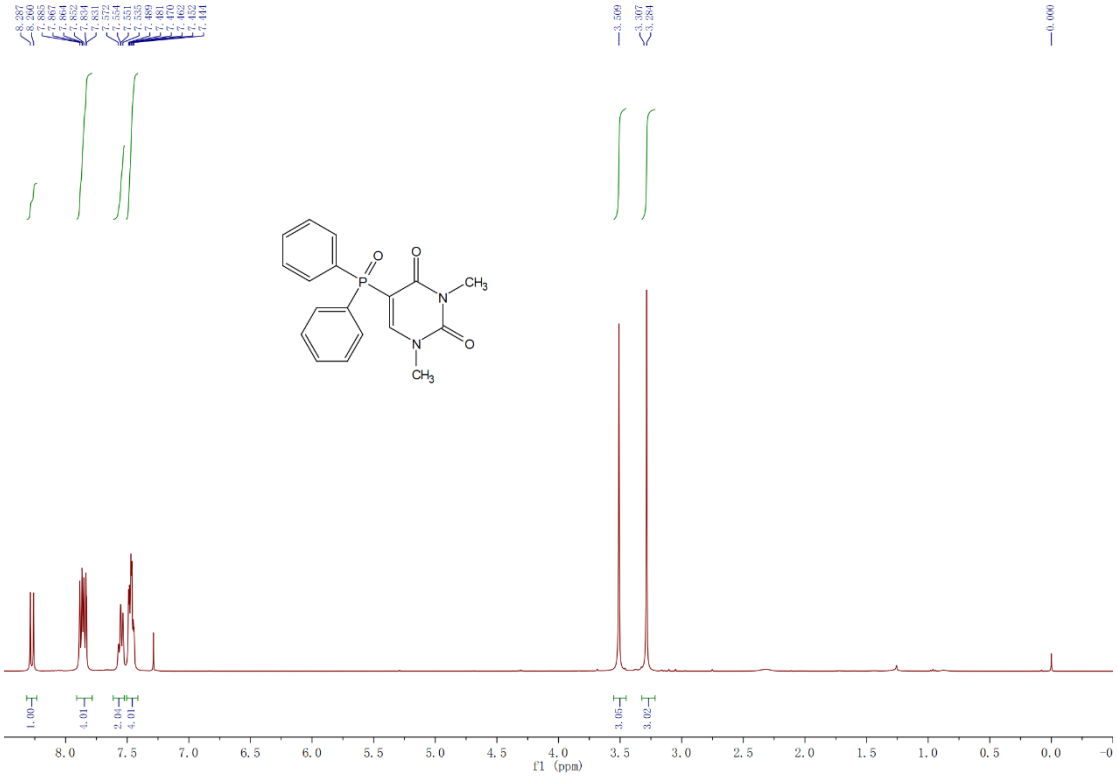
(3,4-dimethoxythiophene-2,5-diyl)bis(diphenylphosphine oxide) (3ah)

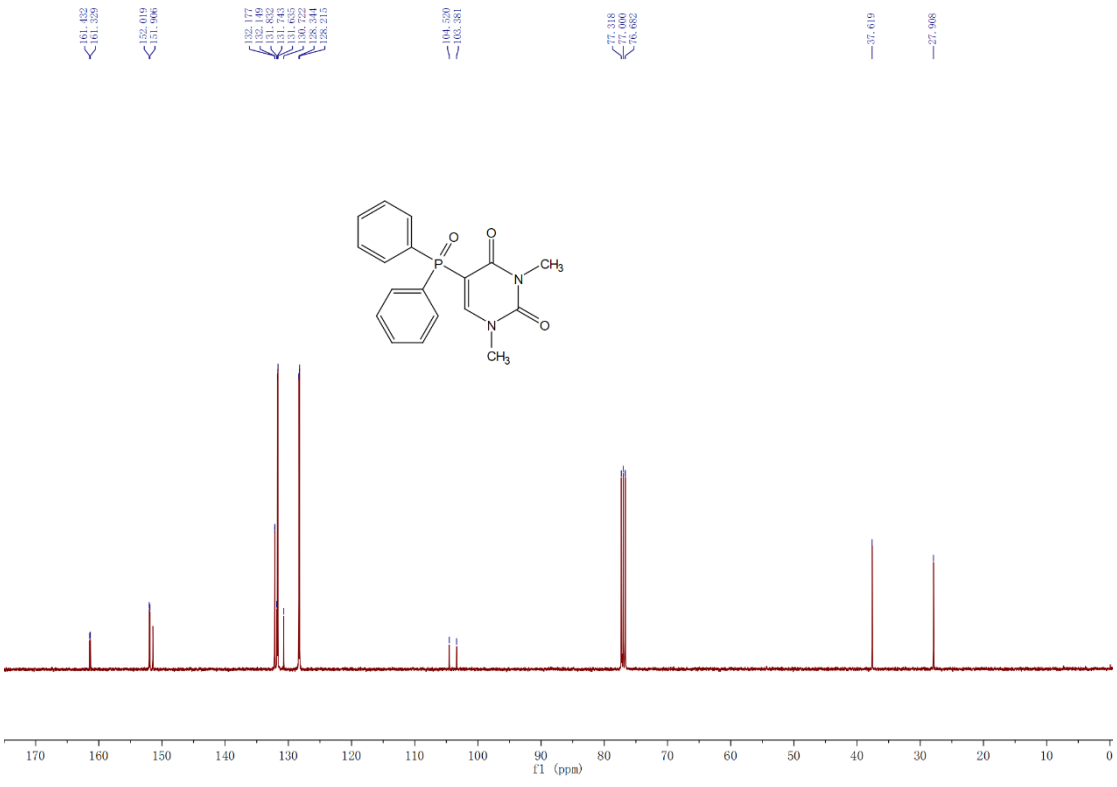


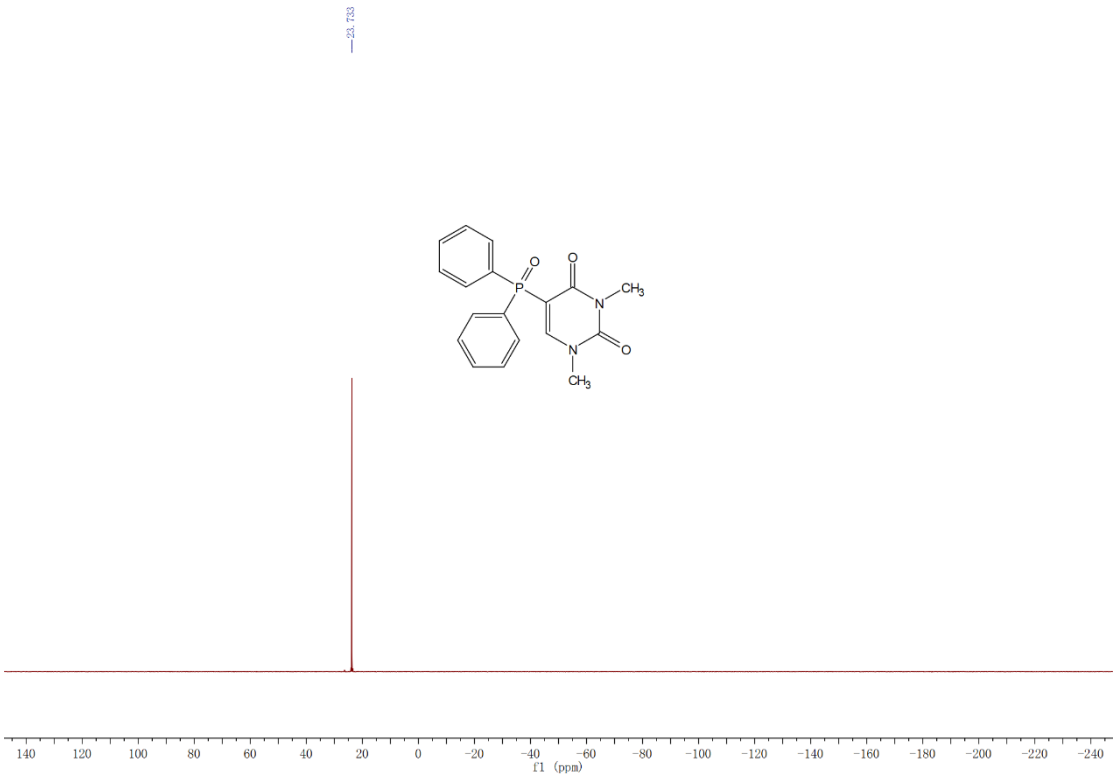

154.894
154.736
132.299
132.065
131.445
131.534
130.956
128.441
128.309
124.442
123.406
77.322
77.000
76.685
60.203
H3C—O
O—CH3
O
P
S
P
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


20.562
H3C—O
O—CH3
O
P
S
P
O
140
120
100
80
60
40
20
0
−20
−40
−60
−80
−100
−120
−140
−160
−180
−200
−220
−240
f1 (ppm)

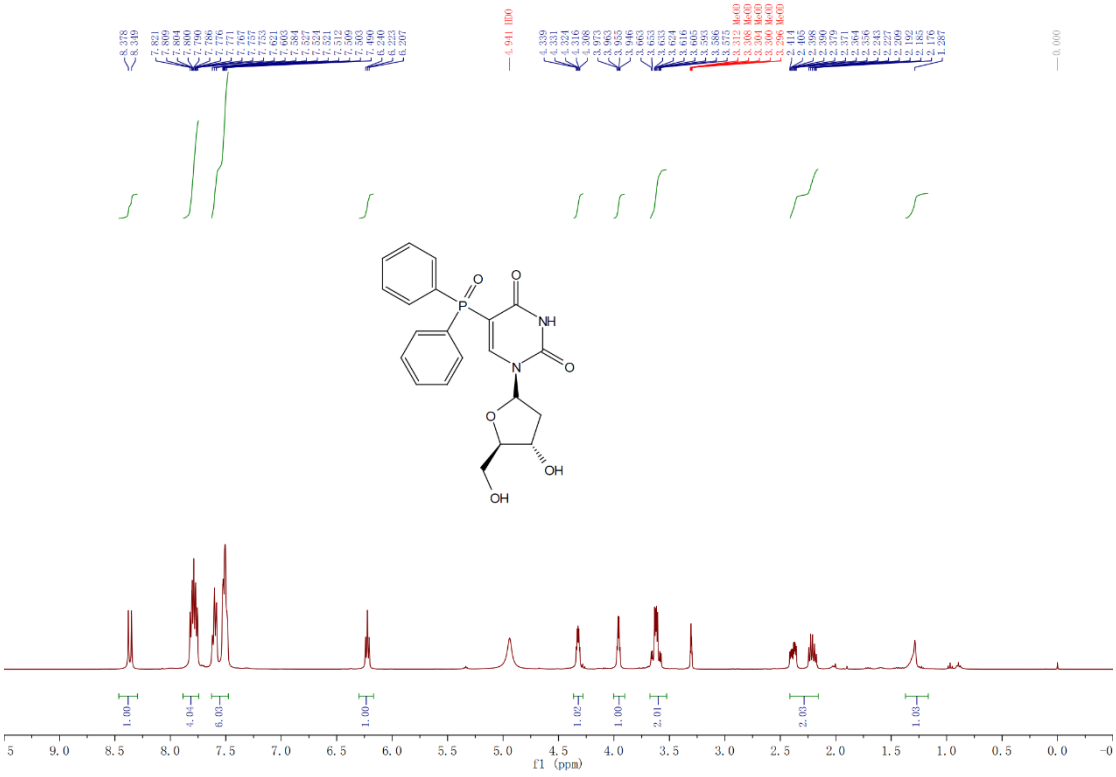
5-(diphenylphosphoryl)-1,3-dimethylpyrimidine-2,4(1H,3H)-dione (3ai)

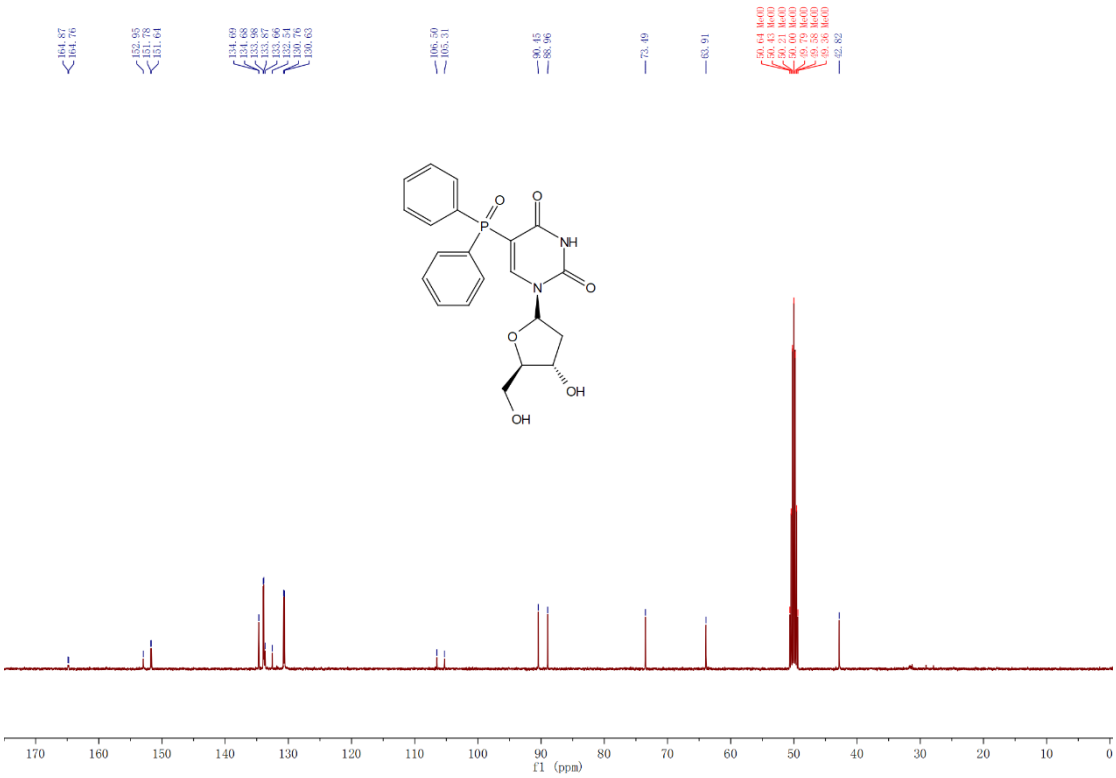


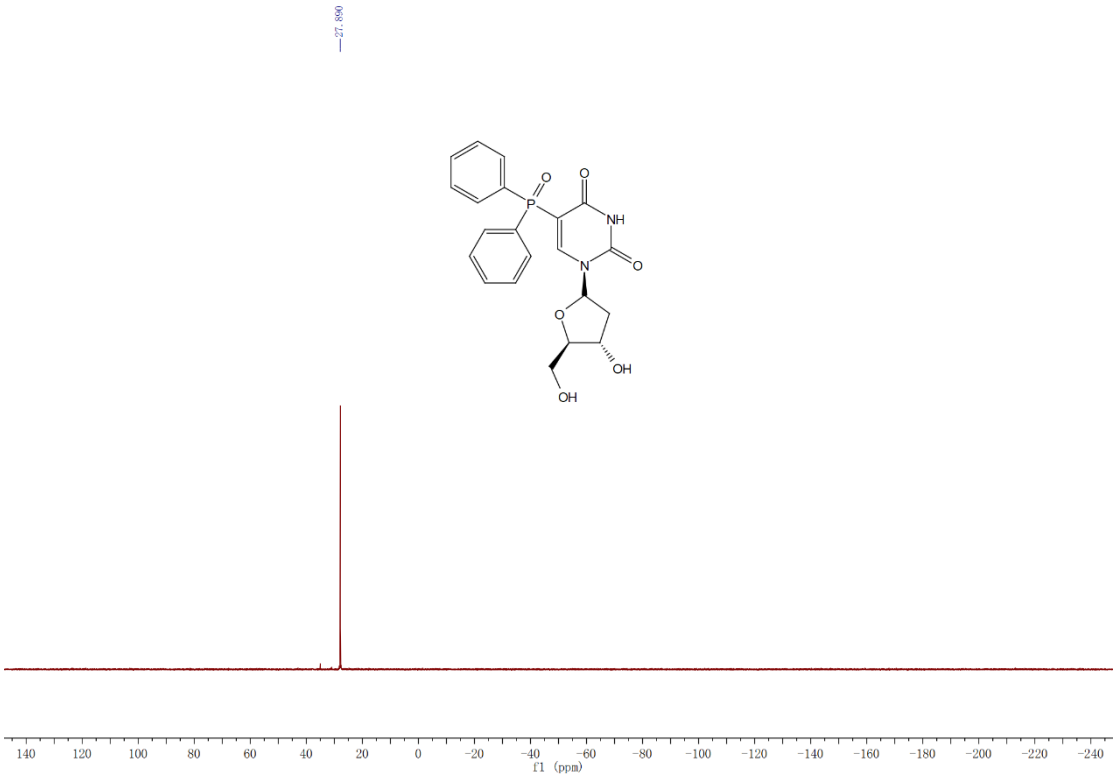




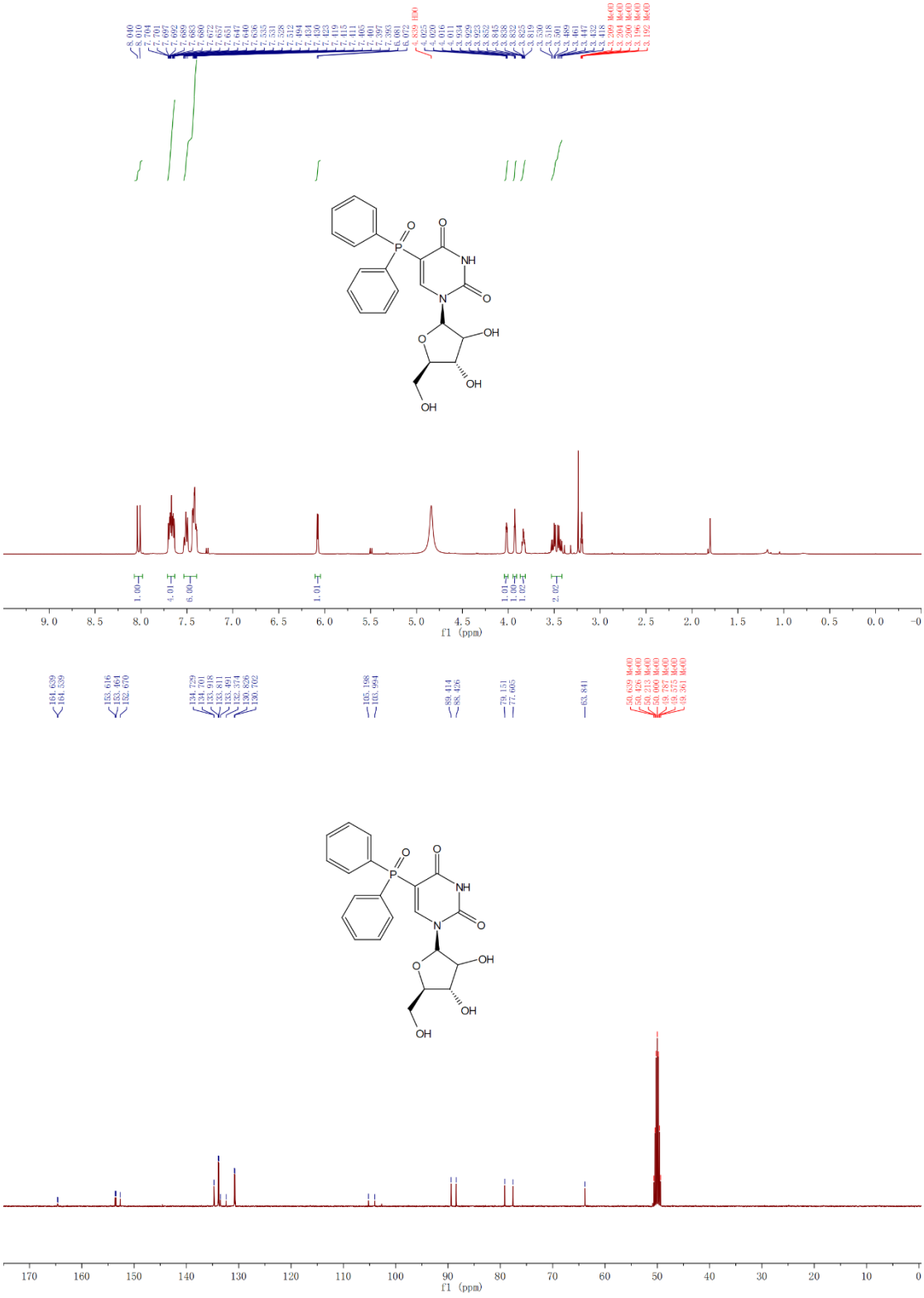
5-(diphenylphosphoryl)-1-((2S,4S,5R)-4-hydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl)pyrimidine-2,4(1H,3H)-dione (3aj)

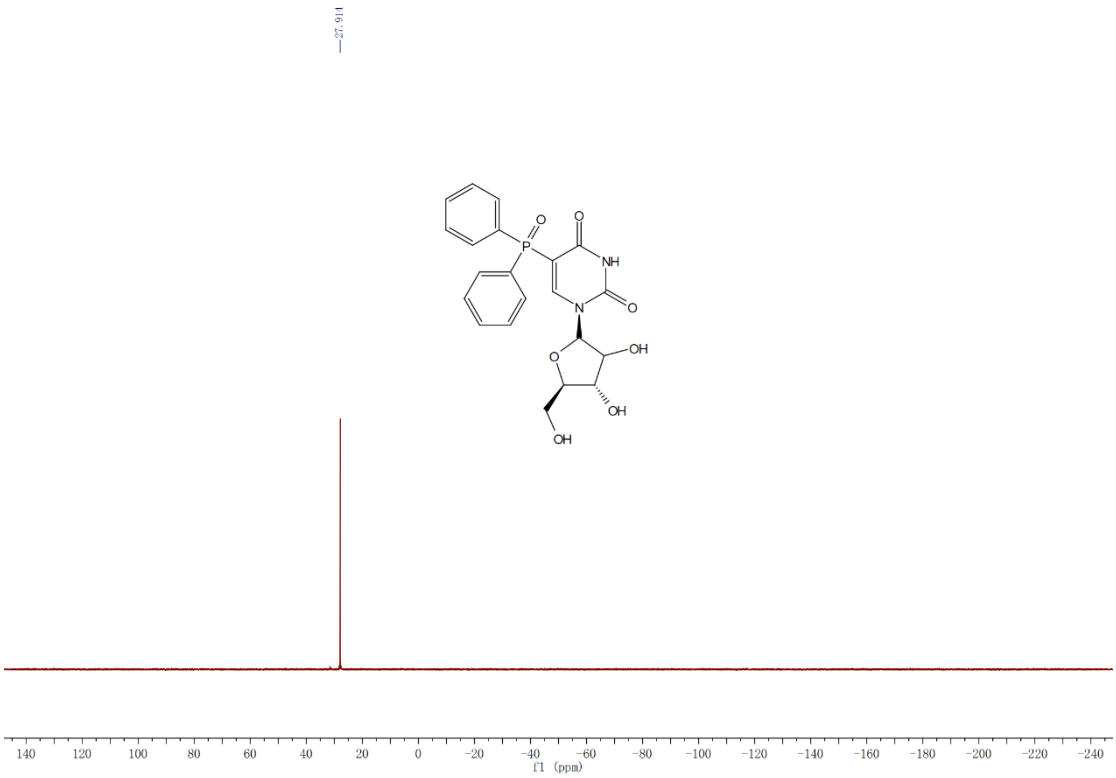



164.87
164.76
152.95
151.78
151.64
134.69
134.68
133.96
133.87
133.66
132.54
130.76
130.63
106.50
105.31
80.45
80.46
73.49
63.91
59.64
59.44
59.31
59.00
58.79
58.58
58.38
58.17
62.82
NH
O
P
N
OH
OH
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

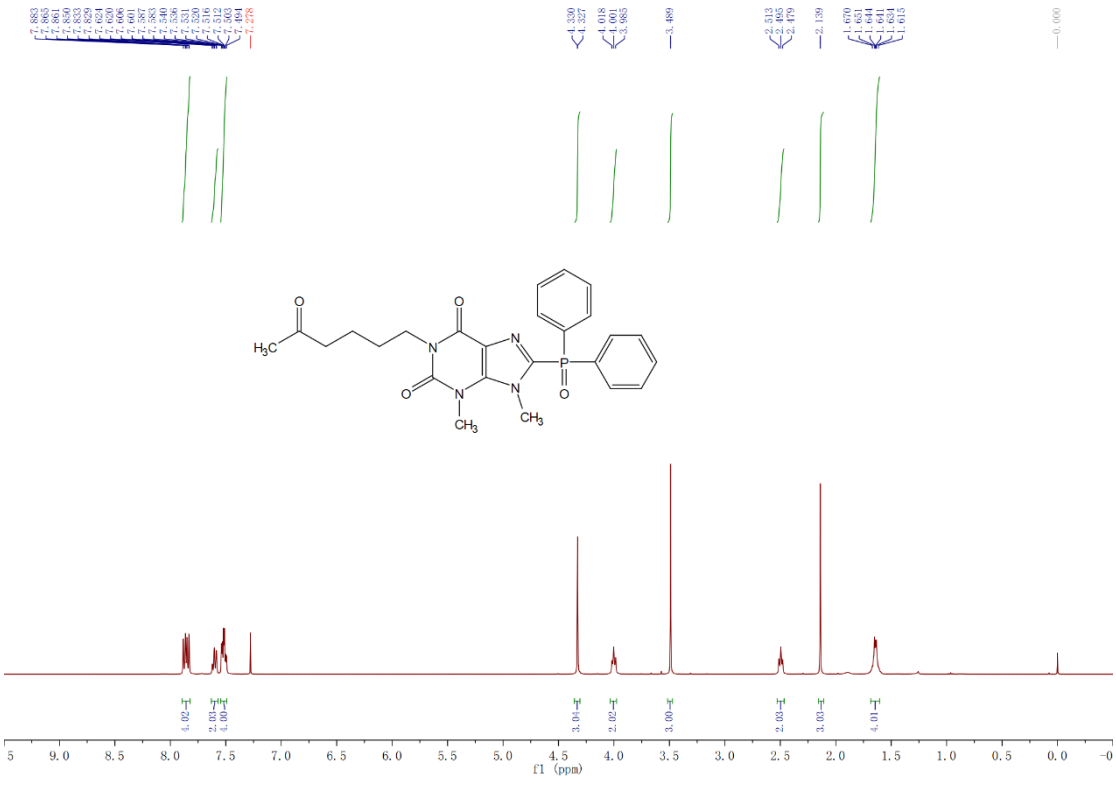

27.890
NH
O
P
N
OH
OH
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

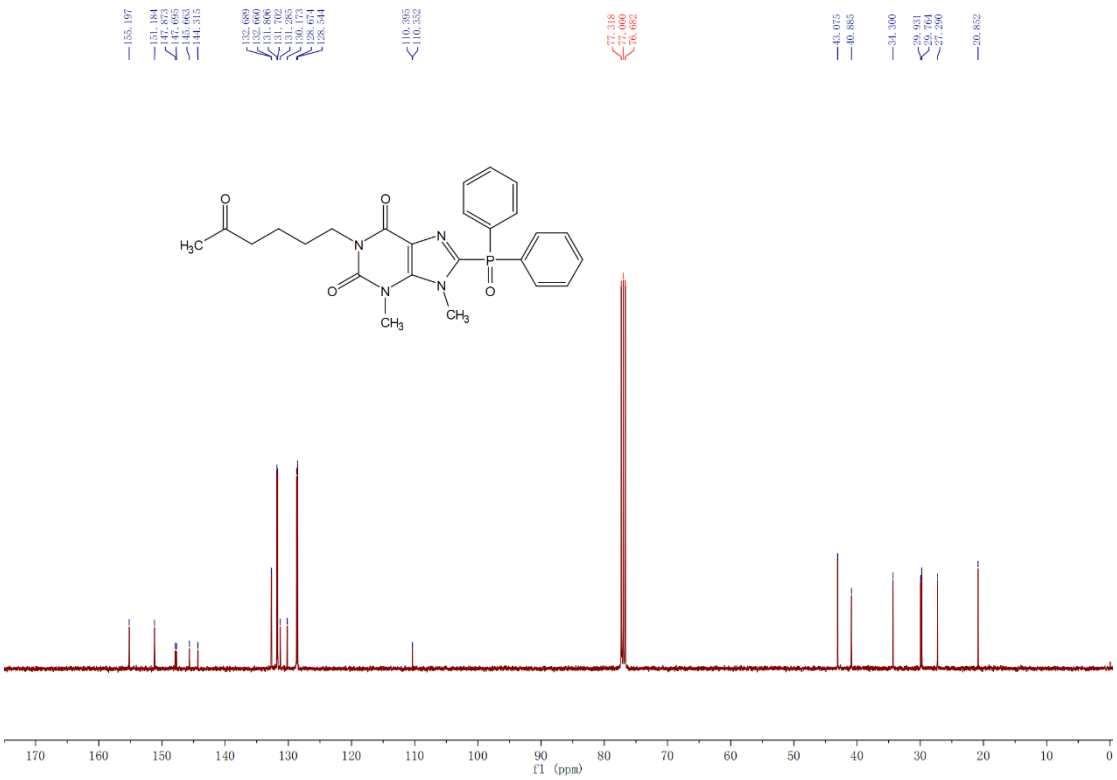
1-((2S,4S,5R)-3,4-dihydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl)-5-(diphenylphosphoryl)pyrimidine-2,4(1H,3H)-dione (3k)

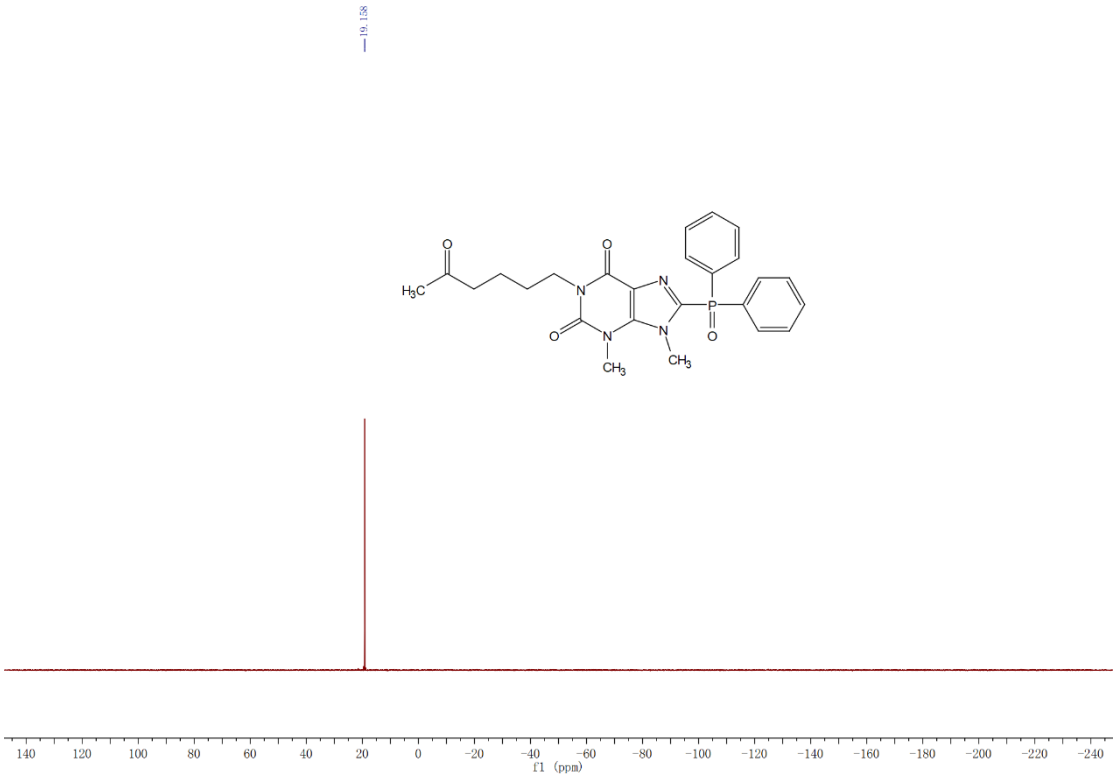




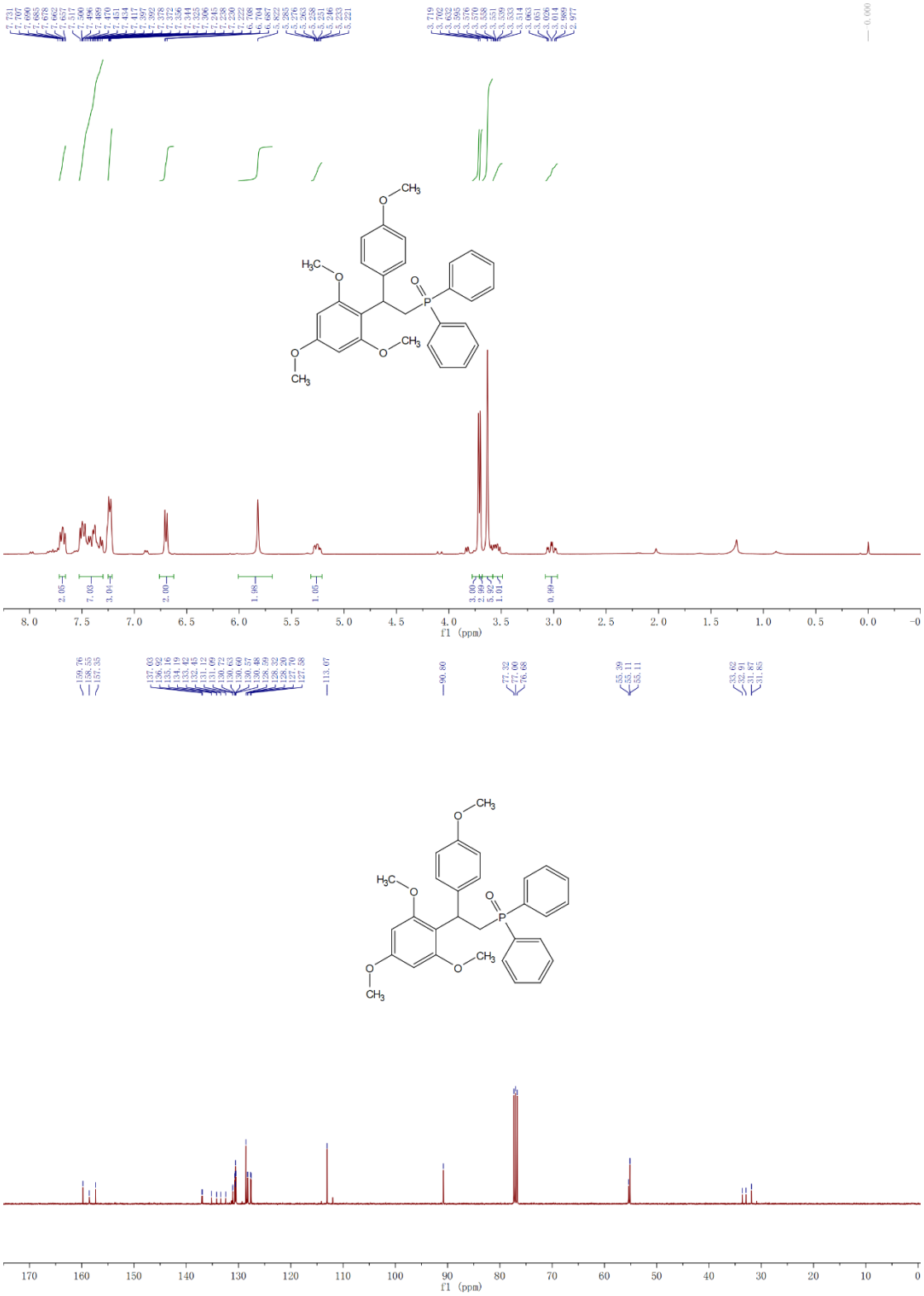
8-(diphenylphosphoryl)-3,9-dimethyl-1-(5-oxohexyl)-3,9-dihydro-1H-purine-2,6-dione (3l)

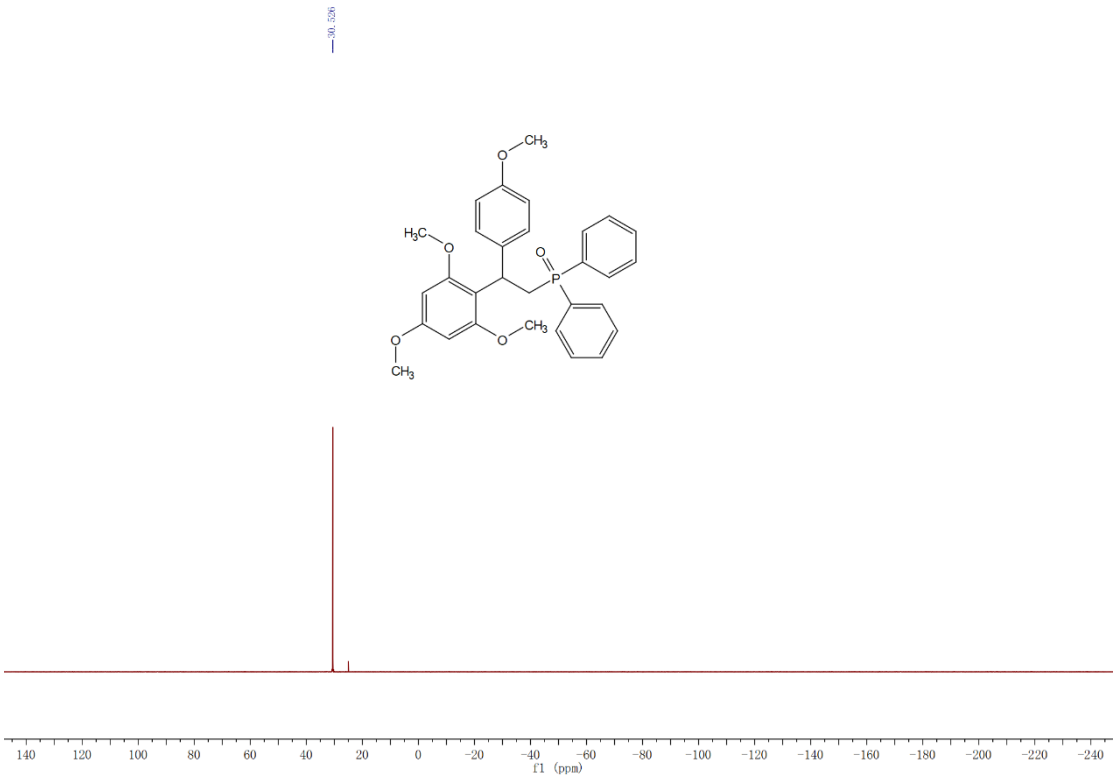



155.197
151.184
147.873
147.695
145.663
144.515
132.689
132.660
131.966
131.702
131.285
130.173
128.671
128.544
110.395
110.352
77.318
77.000
76.682
43.075
40.885
34.300
29.931
29.764
27.296
20.852
H3C
O
N
P
CH3
f1 (ppm)

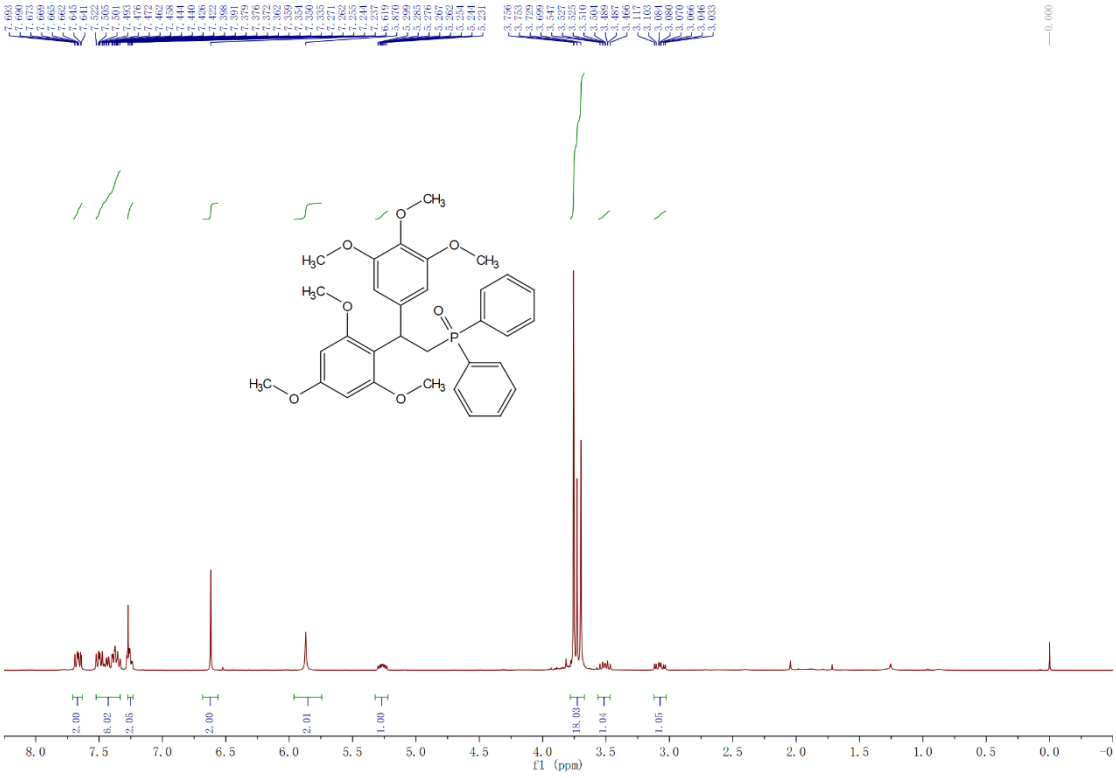

19.158
H3C
O
N
P
CH3
f1 (ppm)

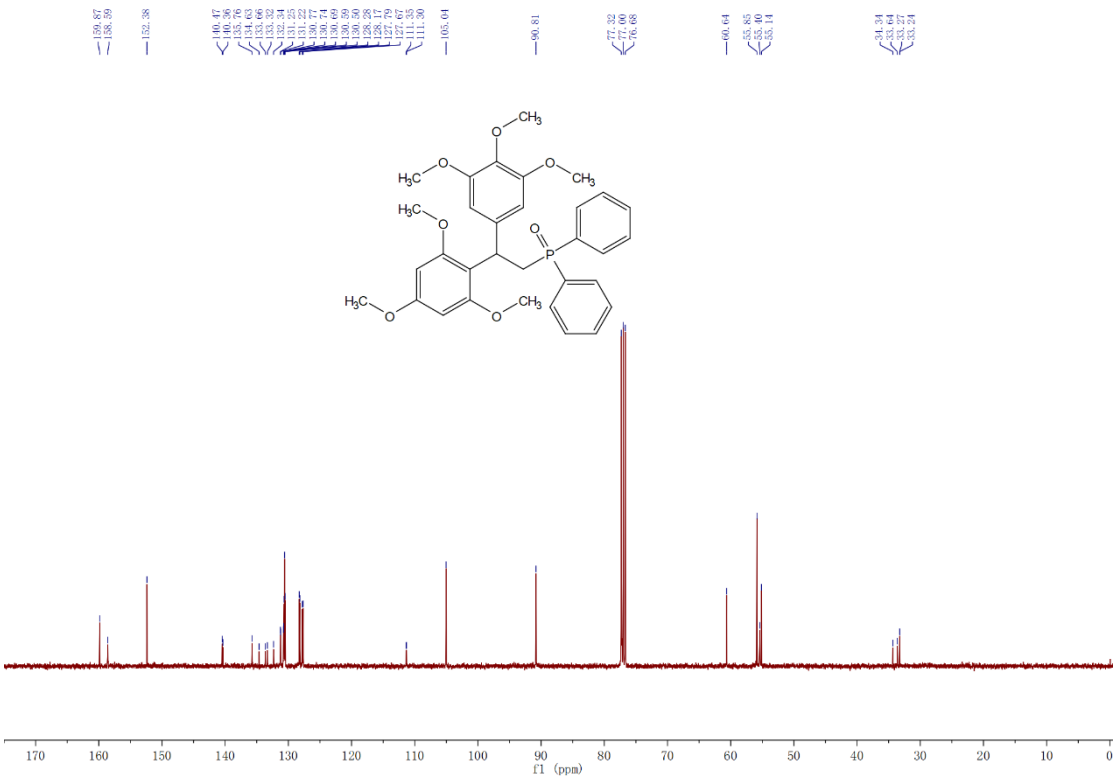
(2-(4-methoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5a)

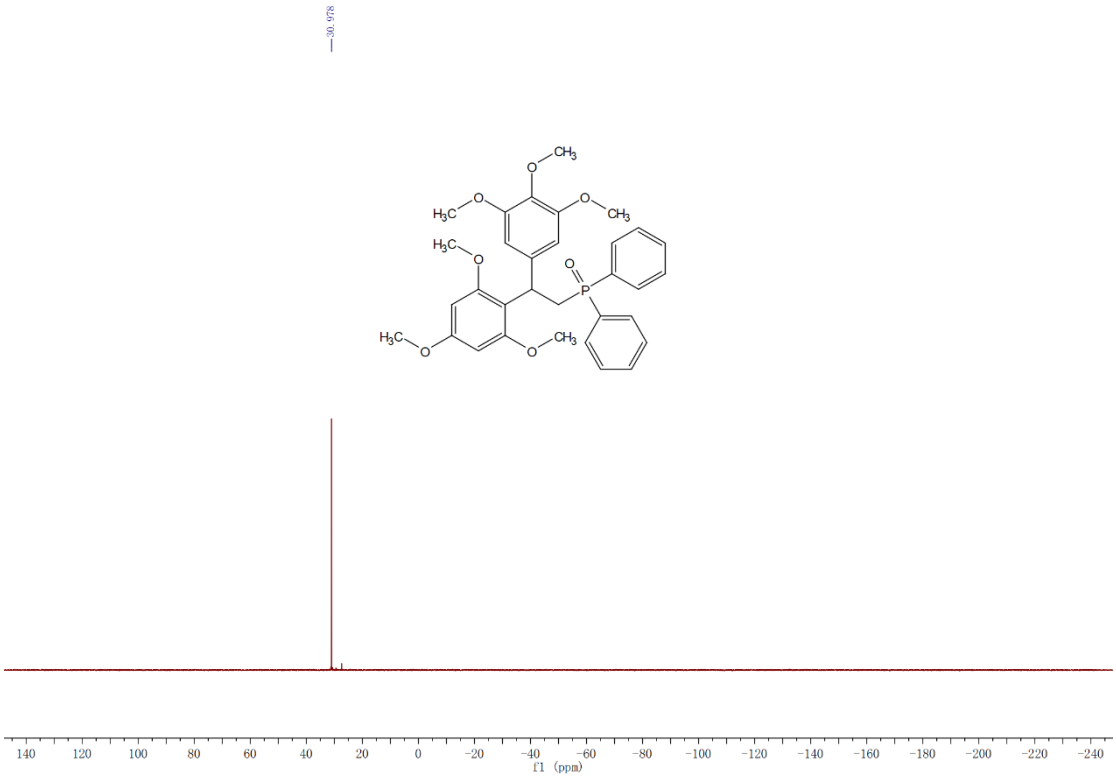




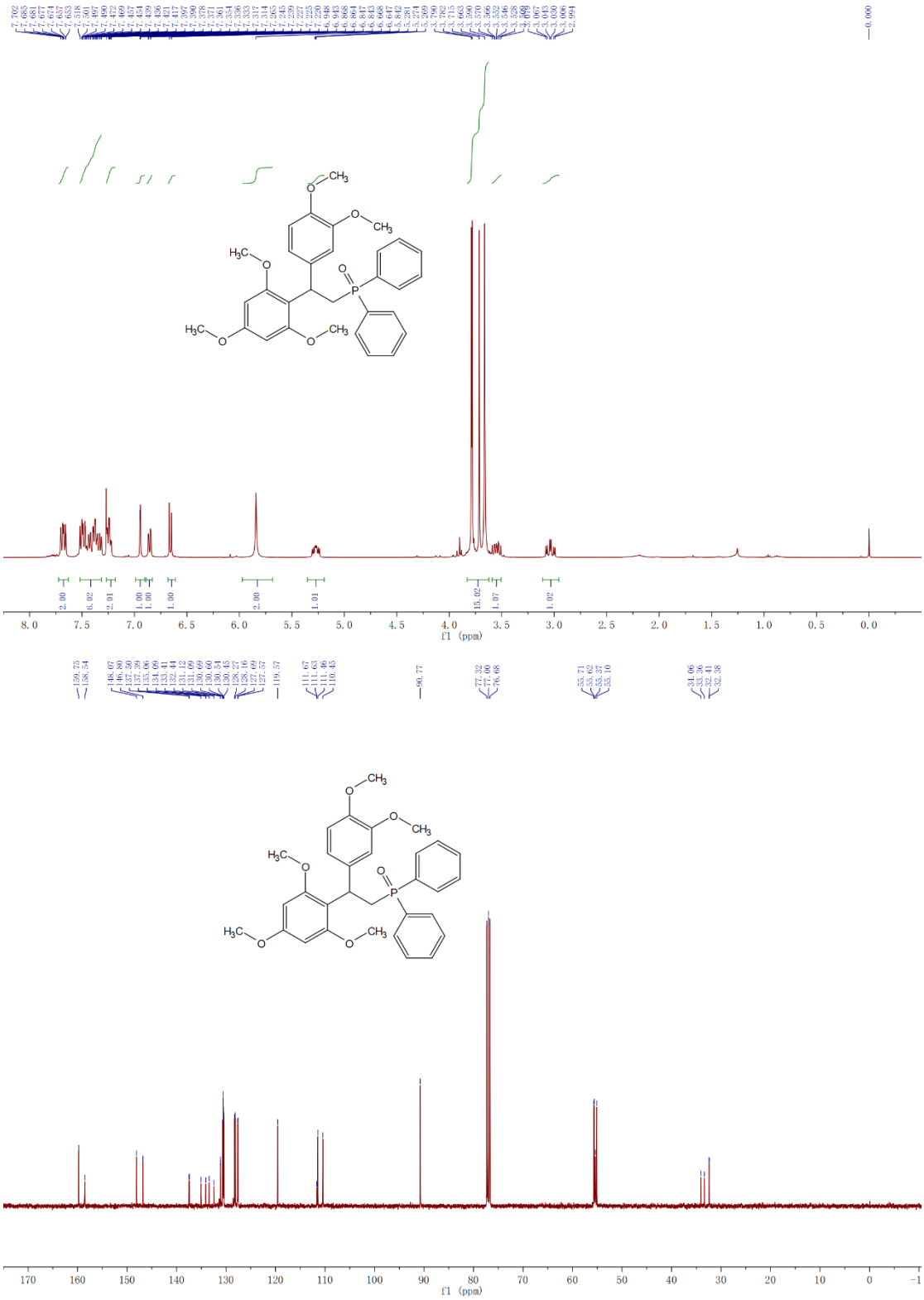
diphenyl(2-(2,4,6-trimethoxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethyl)phosphine oxide (5b)

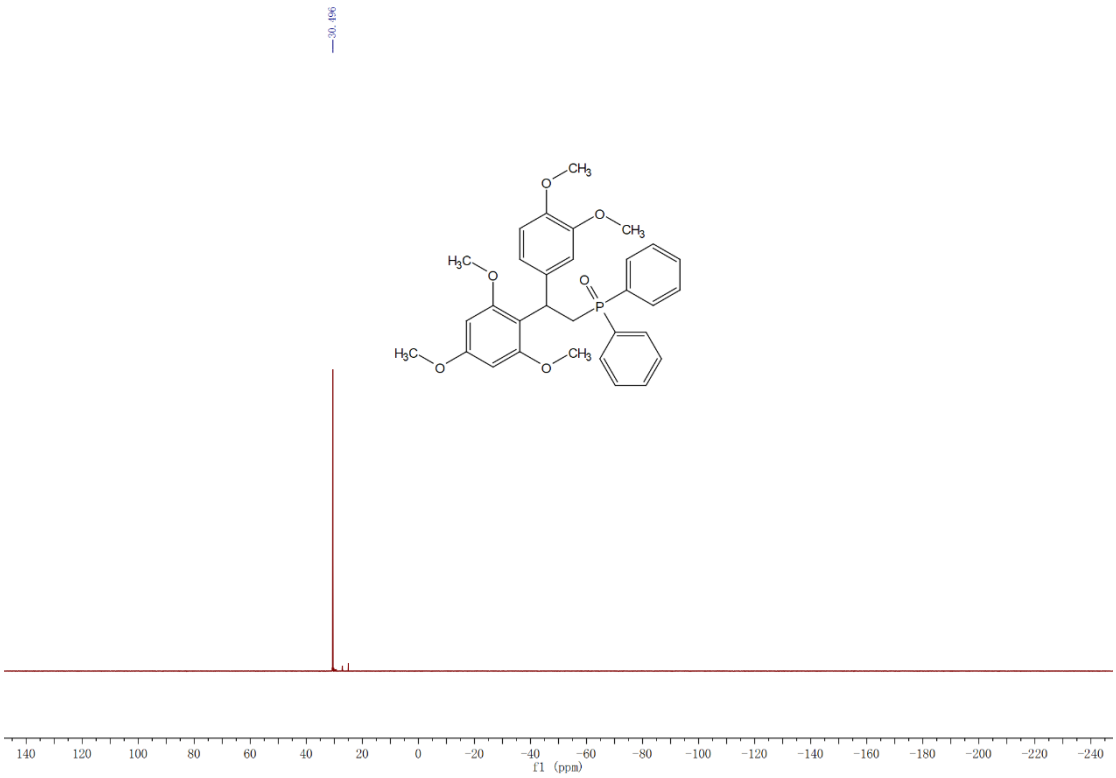



159.87
158.39
132.38
140.47
140.36
135.76
134.63
133.66
133.32
132.34
131.25
131.22
130.77
130.74
130.69
130.59
130.50
128.28
128.17
127.79
127.67
111.35
111.30
105.04
90.81
77.32
77.00
76.68
60.64
55.85
55.46
55.14
34.34
33.64
33.27
33.24
f1 (ppm)

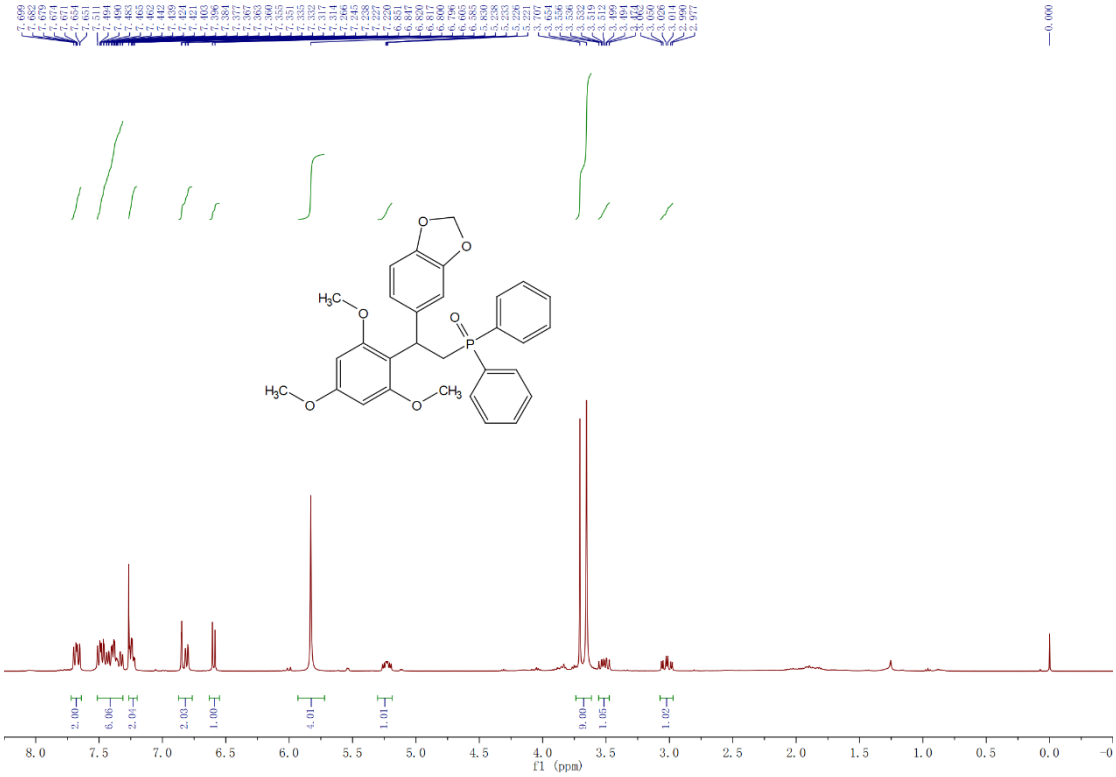

30.978
f1 (ppm)

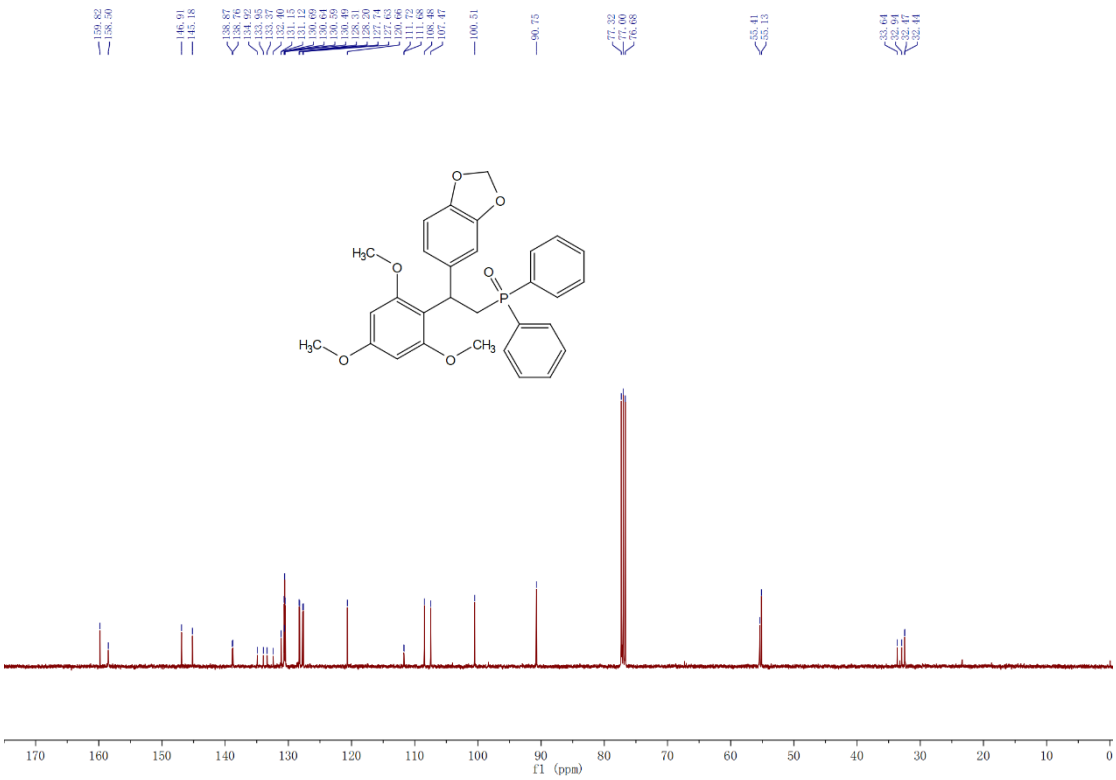
(2-(3,4-dimethoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5c)

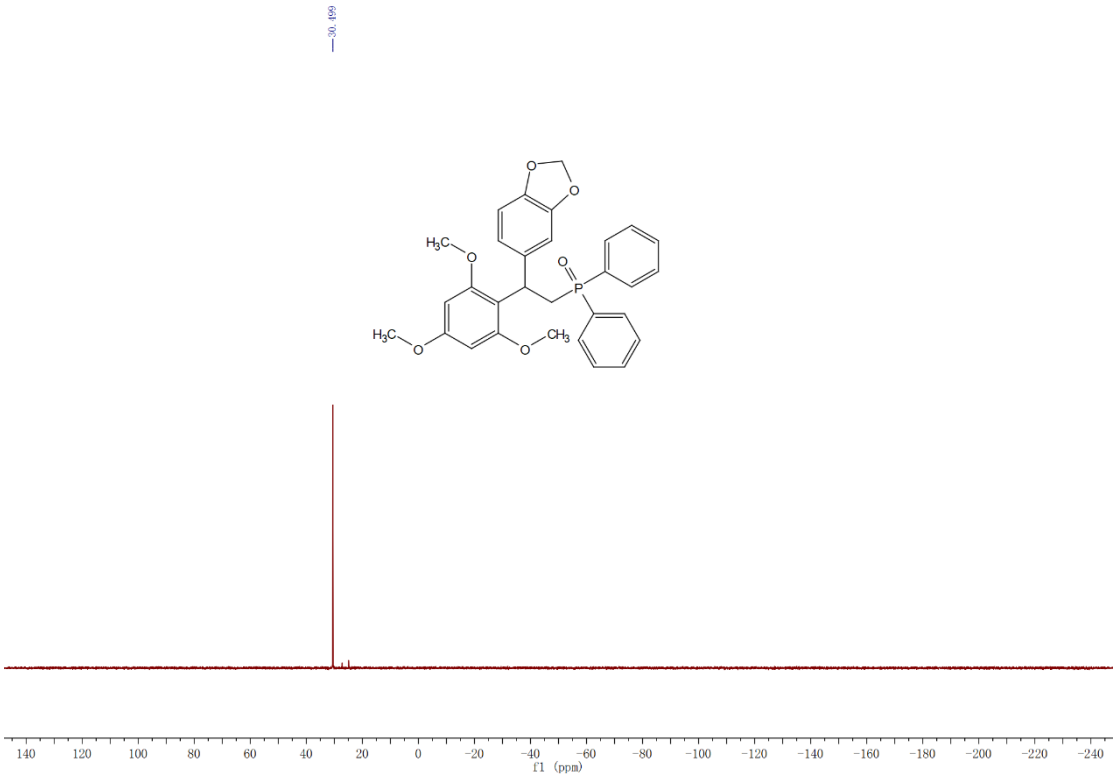




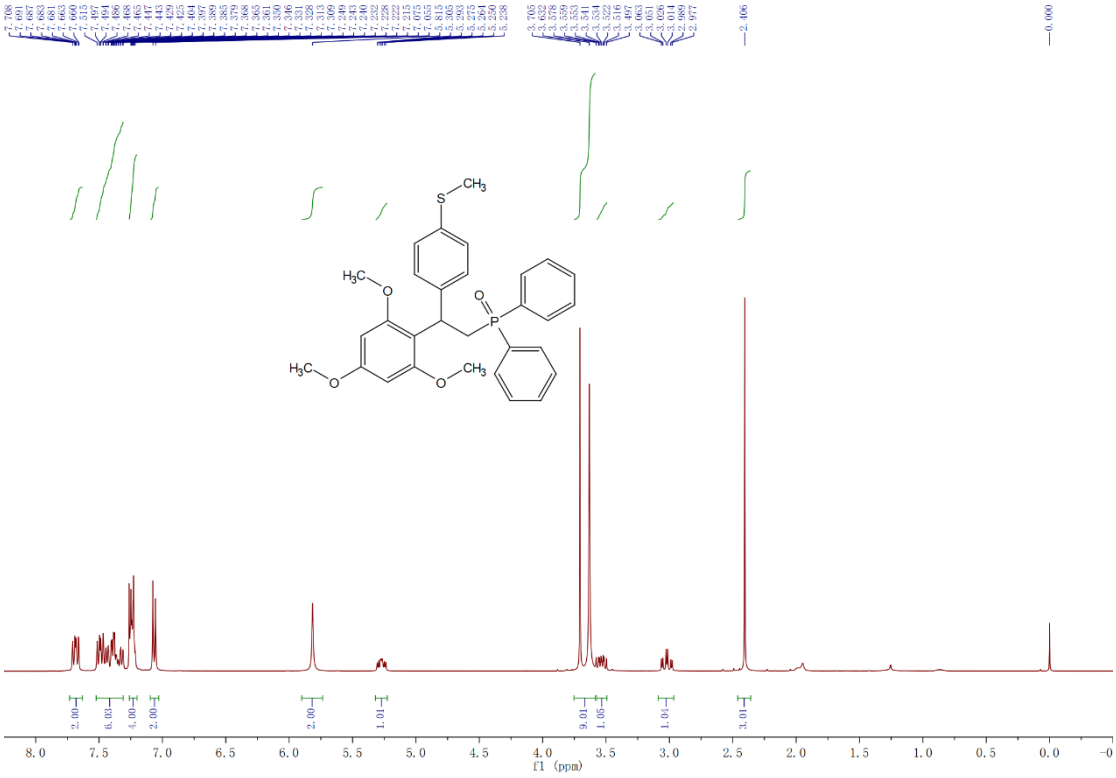
(2-(benzo[d][1,3]dioxol-5-yl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5d)

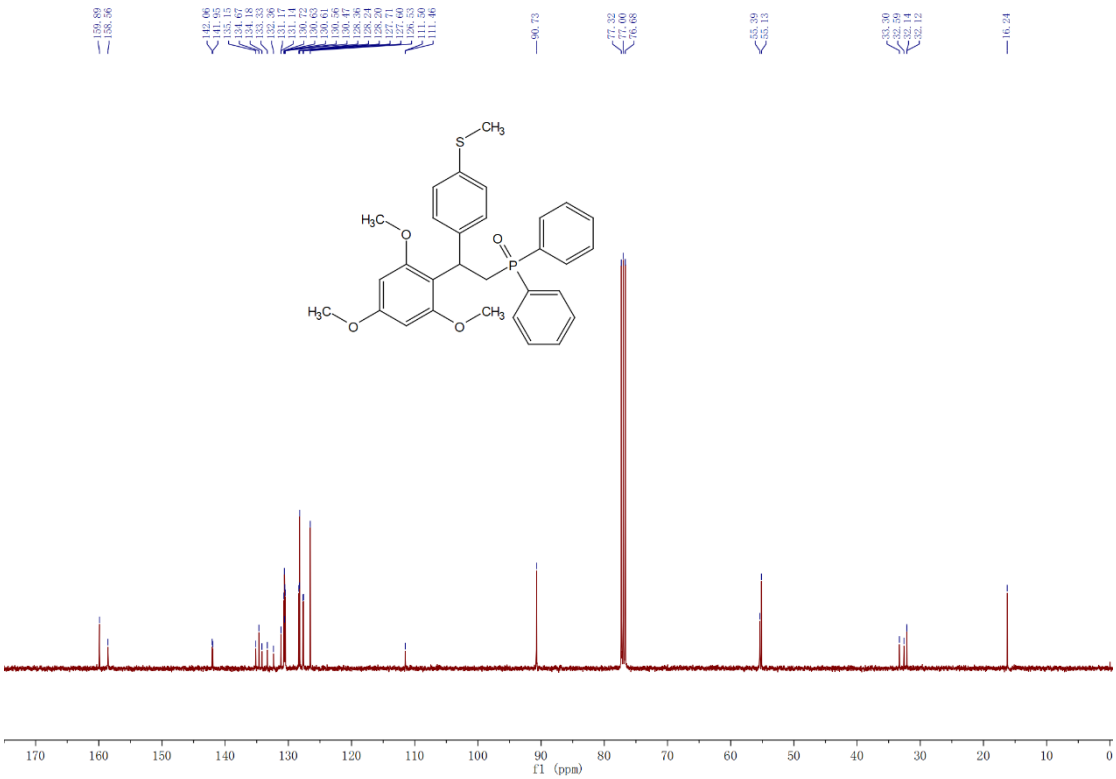


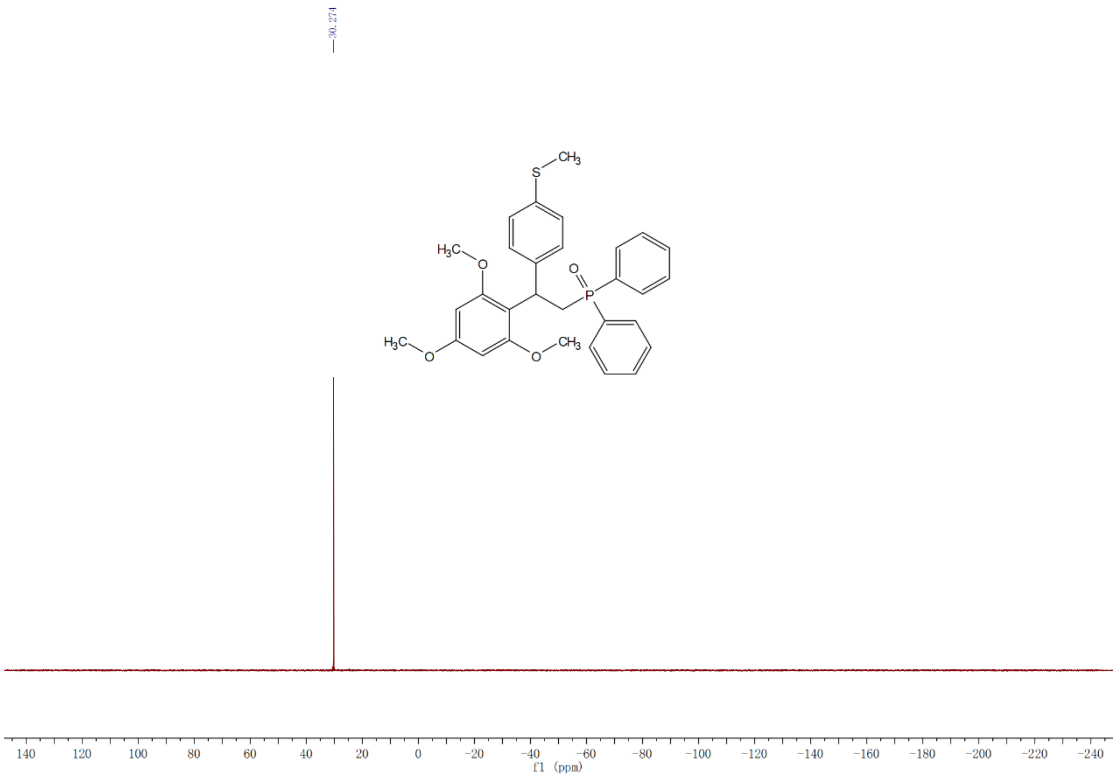

150.82
148.50
146.91
145.18
138.87
138.76
134.92
133.95
133.37
132.80
131.15
131.12
130.69
130.64
130.59
130.49
128.31
128.20
127.74
127.63
120.66
111.72
111.56
108.48
107.47
100.51
90.75
77.32
77.00
76.68
55.41
55.13
33.64
32.94
32.47
32.44
H3C
O
O
O
P
H3C
O
O
CH3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


30.699
H3C
O
O
O
P
H3C
O
O
CH3
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

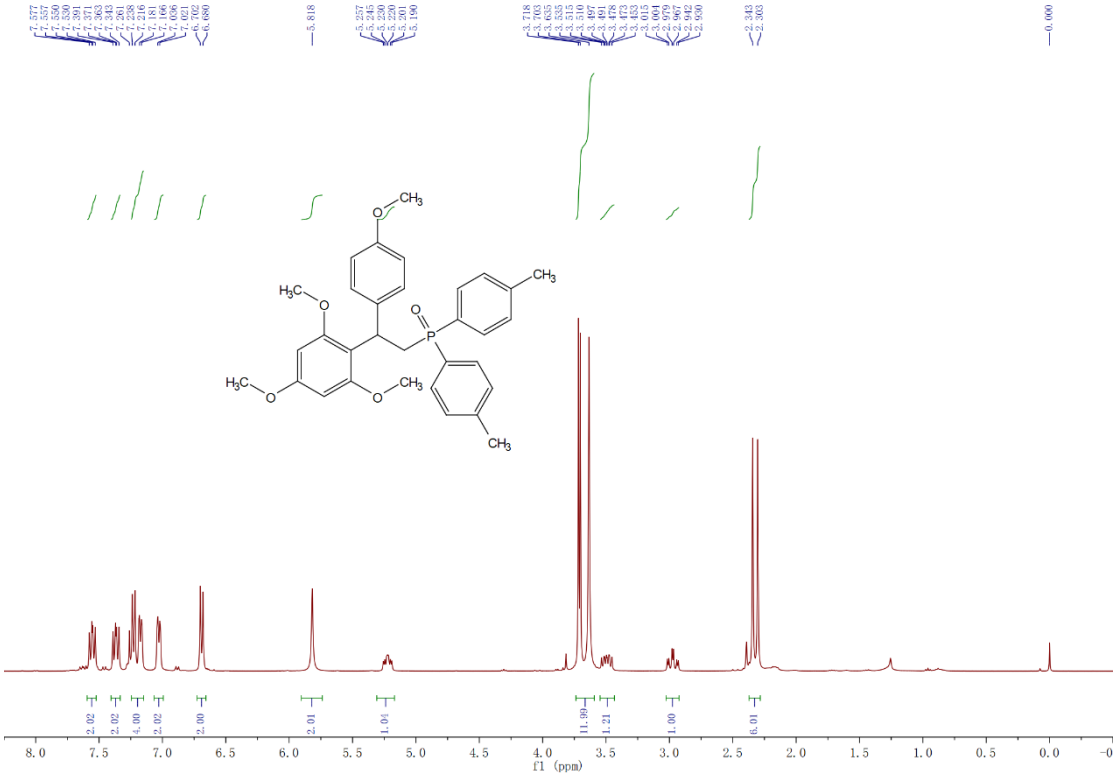
(2-(4-(methylthio)phenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)diphenylphosphine oxide (5h)

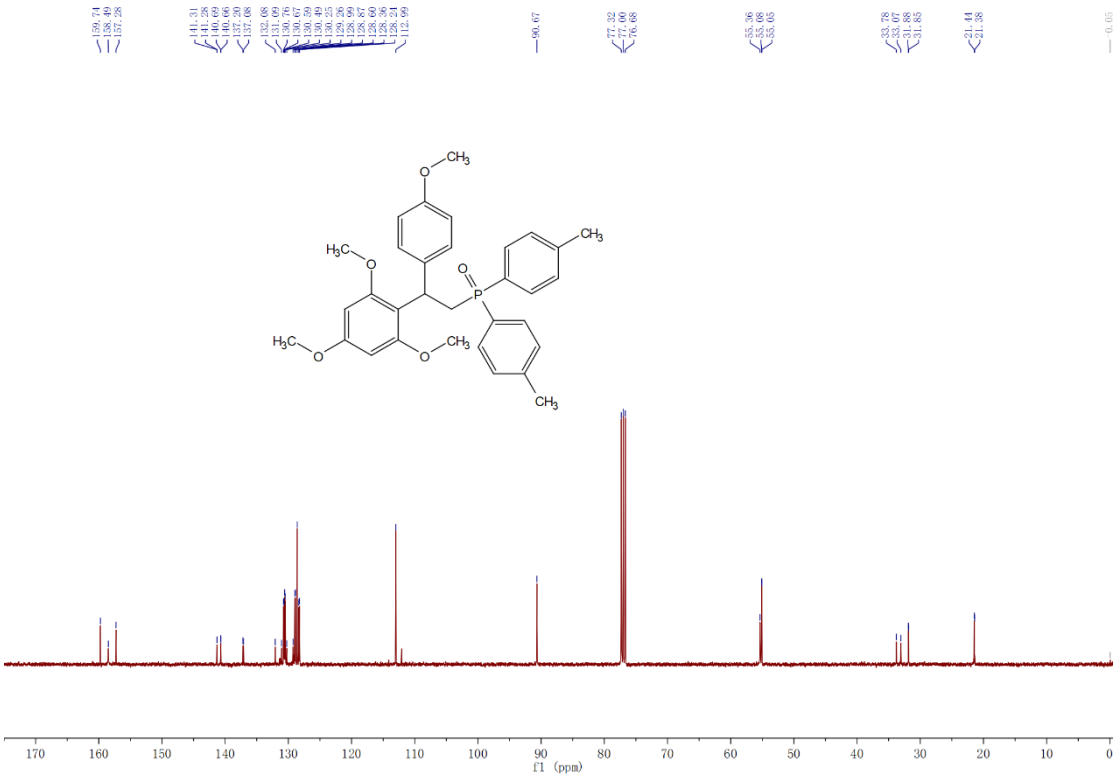


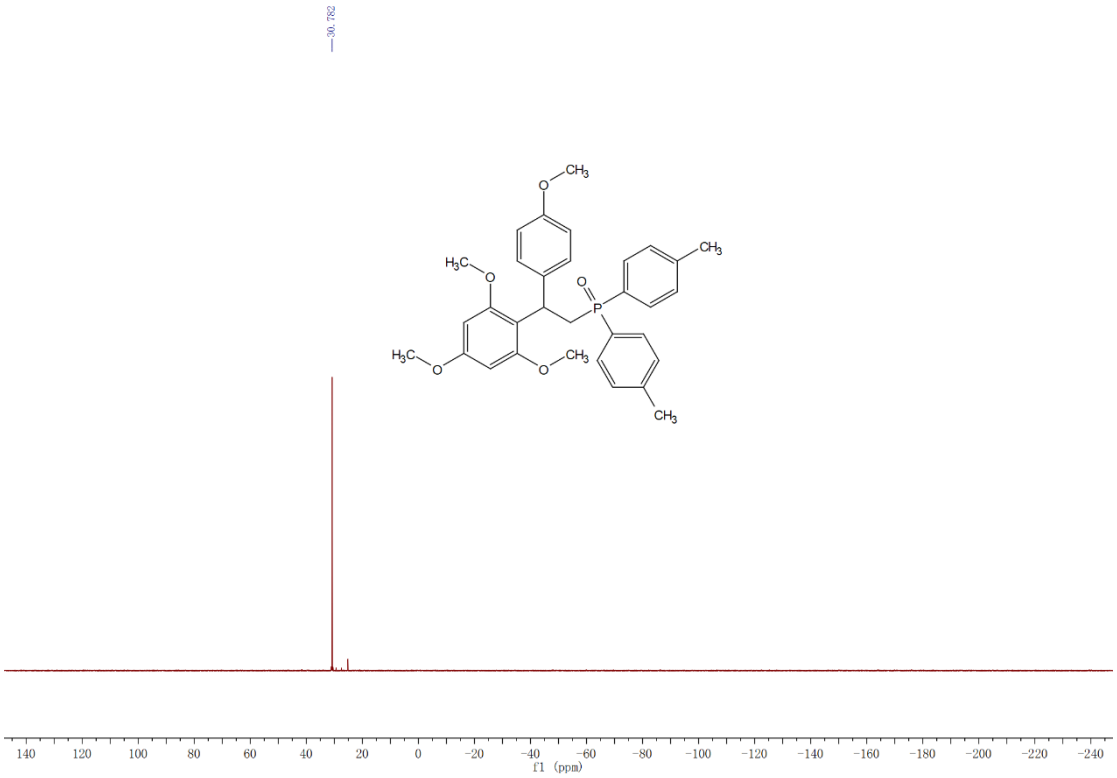




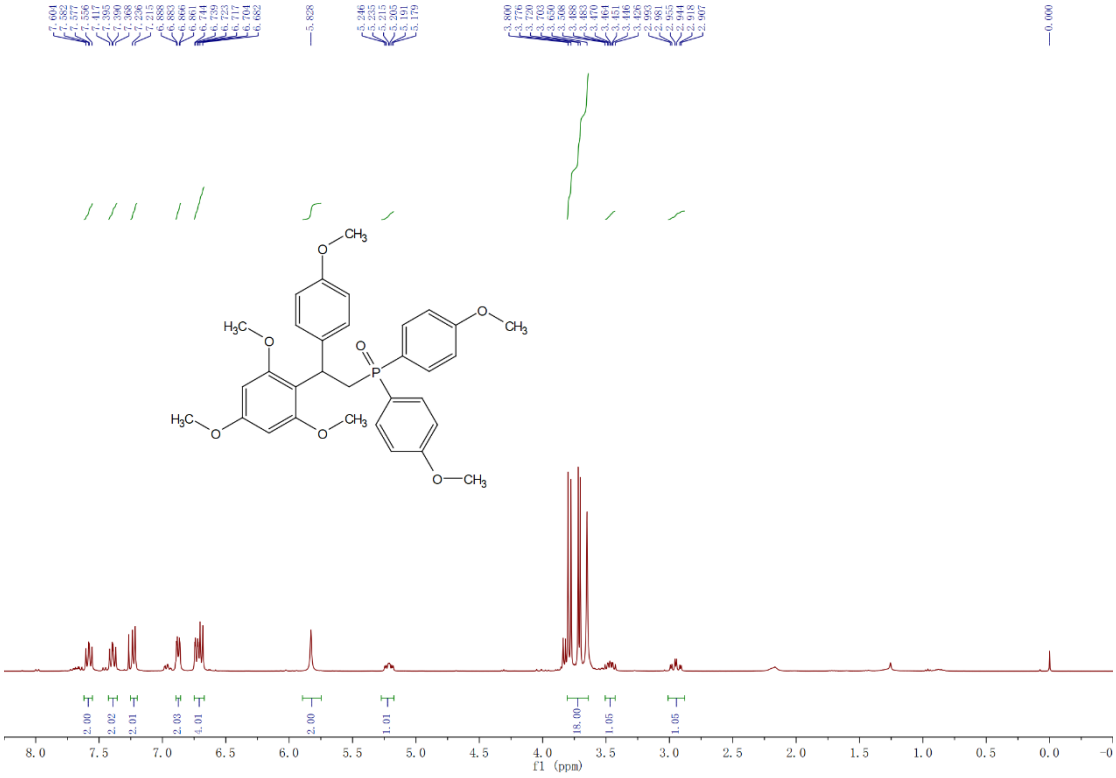
(2-(4-methoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)di-p-tolylphosphine oxide (5i)

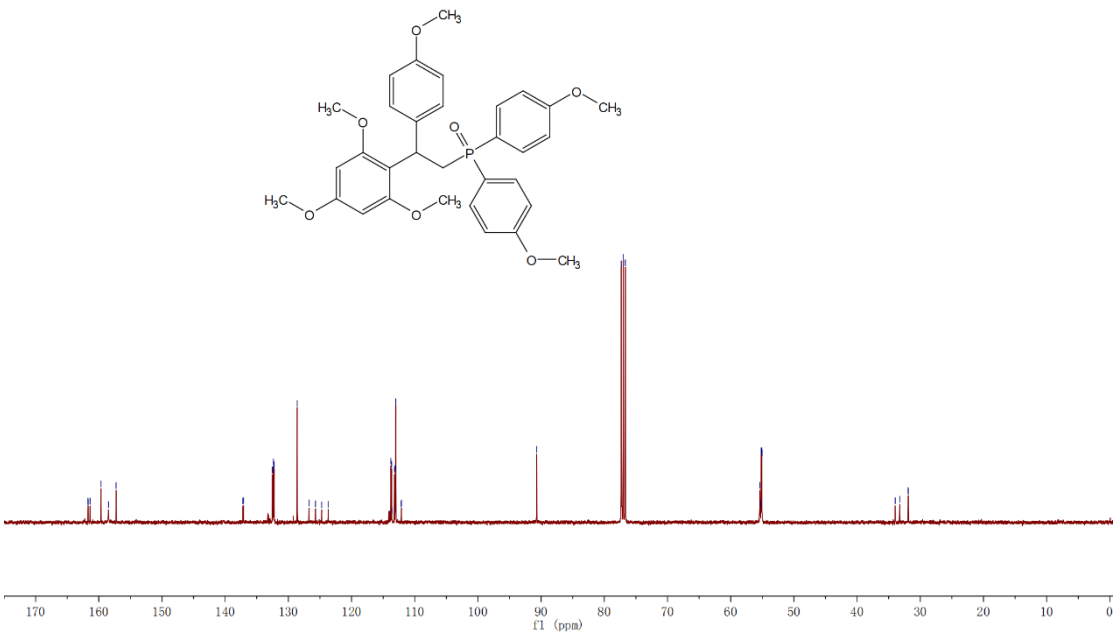


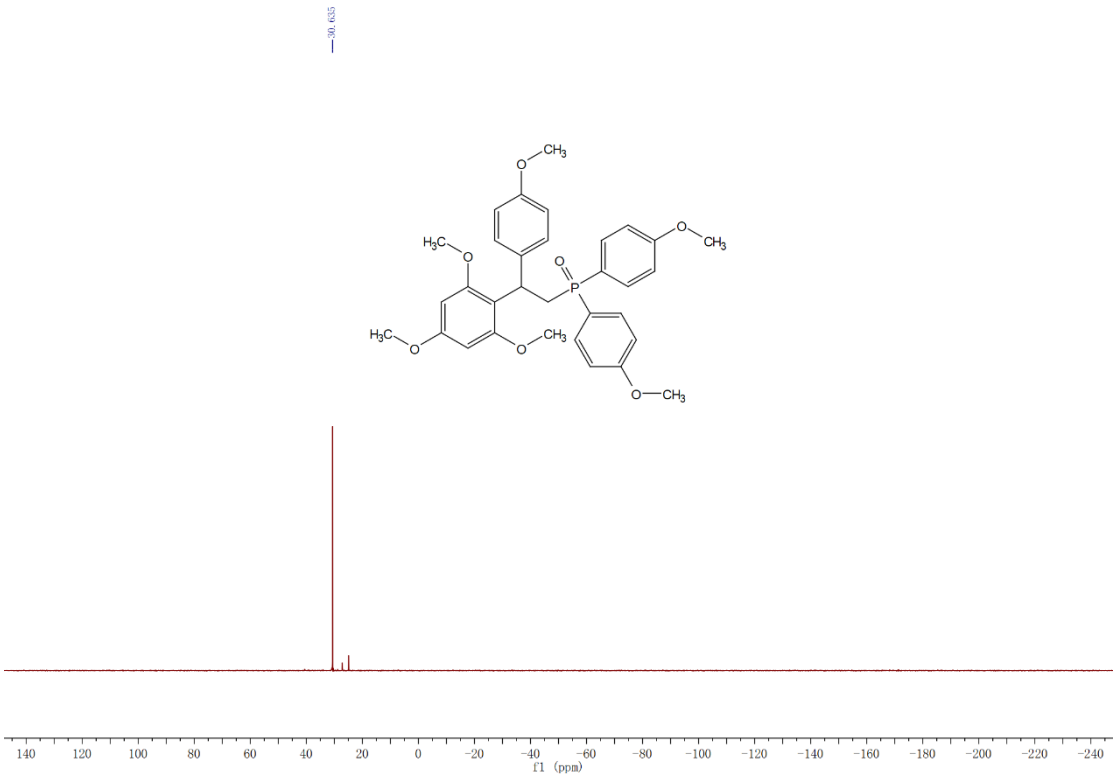

159.74
158.49
157.28
141.31
141.28
140.69
140.66
137.29
137.08
132.68
131.09
130.76
130.67
130.59
130.49
130.25
129.26
128.89
128.87
128.60
128.36
128.24
112.99
90.67
77.32
77.00
76.68
55.36
55.08
55.05
32.78
32.67
31.88
31.85
21.44
21.38
0.65
O
CH3
H3C
O
CH3
P
O
H3C
O
O
CH3
CH3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


30.782
O
CH3
H3C
O
CH3
P
O
H3C
O
O
CH3
CH3
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

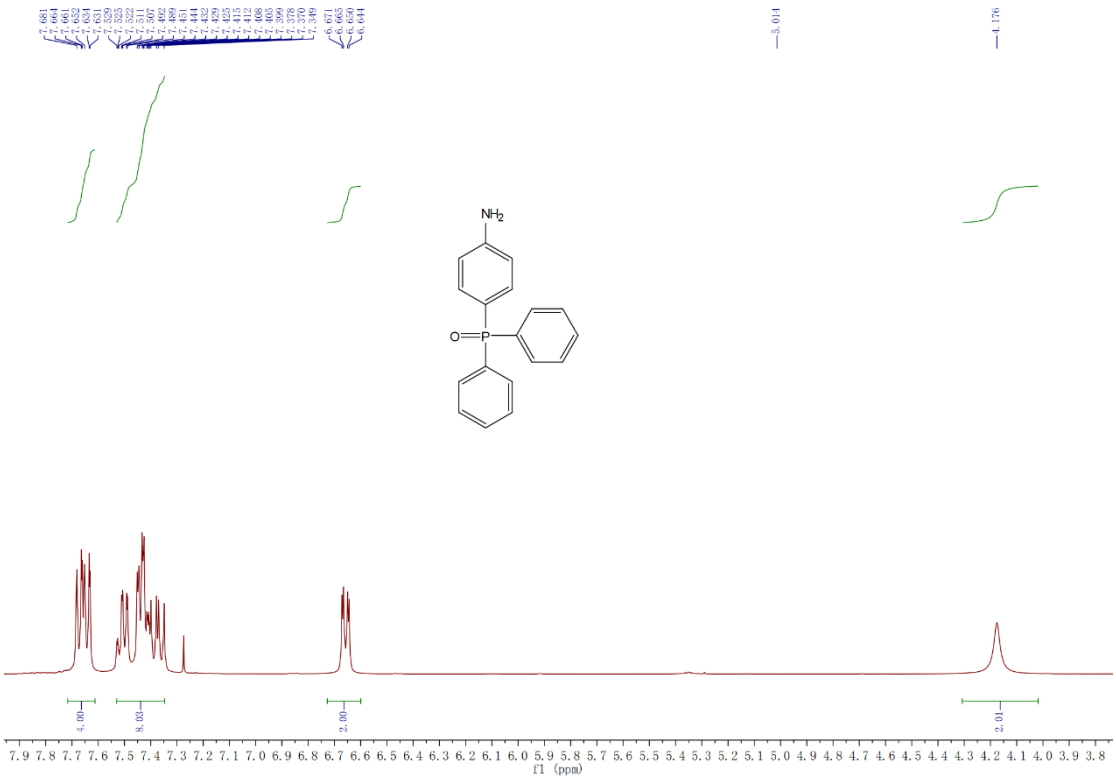
bis(4-methoxyphenyl)(2-(4-methoxyphenyl)-2-(2,4,6-trimethoxyphenyl)ethyl)phosphine oxide (5j)

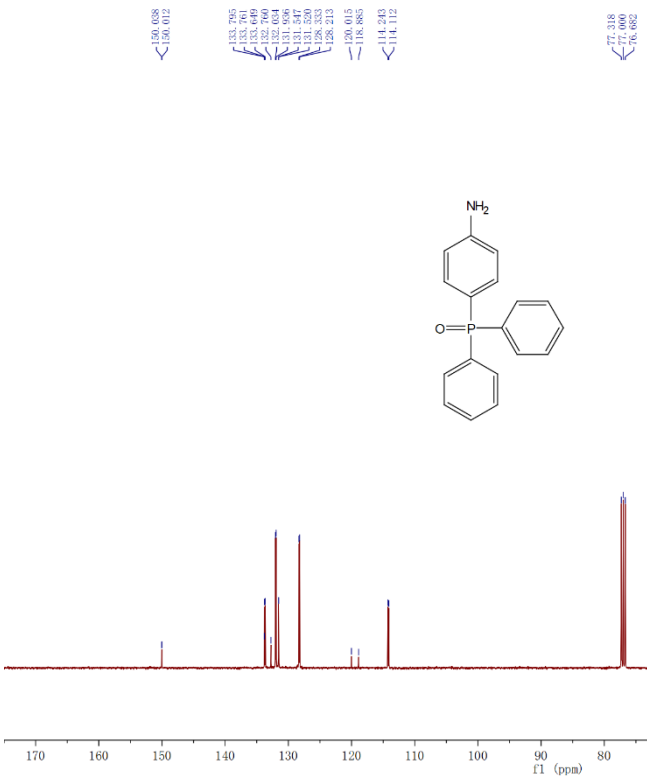


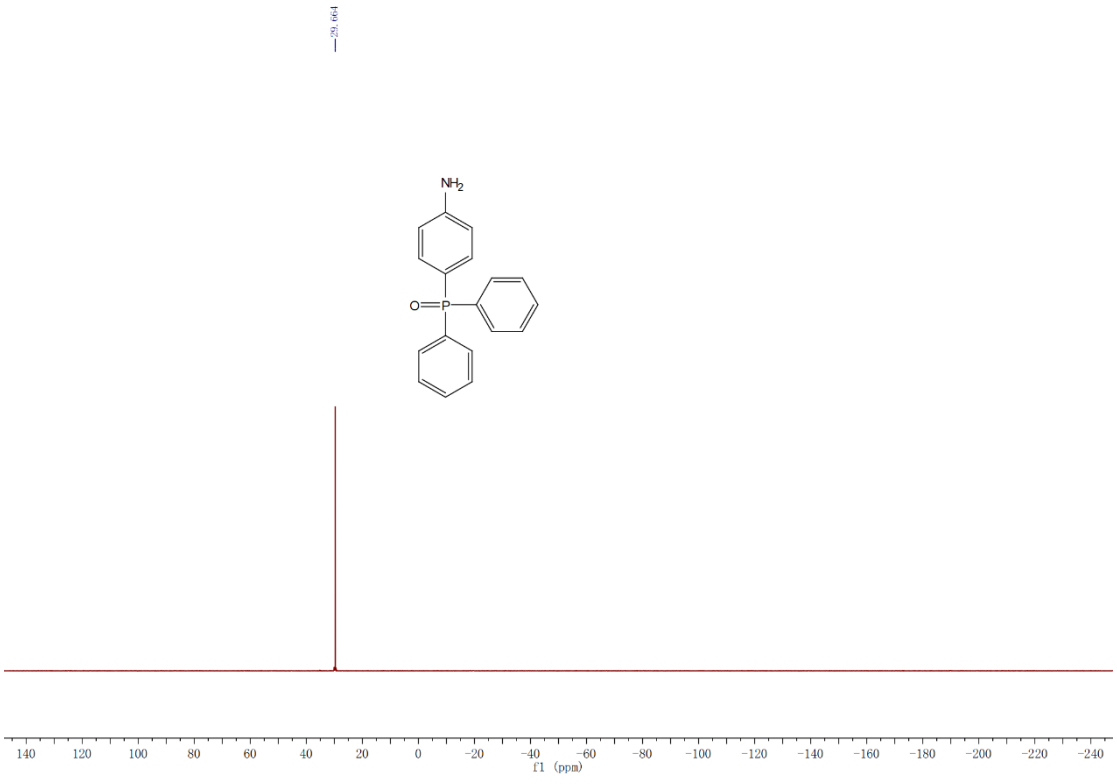




(4-aminophenyl)diphenylphosphine oxide (6)




150.038
150.012
133.795
133.761
133.449
132.749
132.634
131.956
131.547
131.350
128.333
128.213
120.015
118.885
114.243
114.112
77.318
77.000
76.682
NH2
O
P
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


29.664
NH2
O
P
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
f1 (ppm)

10. References

1. C. A. Busacca, J. C. Lorenz, N. Grinberg, N. Haddad, M. Hrapchak, B. Latli, H. Lee, P. Sabila, A. Saha, M. Sarvestani, S. Shen, R. Varsolona, X. Wei and C. H. Senanayake, *Org. Lett.*, 2005, **7**, 4277.
2. Kitson, S. L., Jones, S., Watters, W., Chen, F. & Madge, D, *J, Label Compd. Radiopharm., 2010,* **53**, 140.