

**Reinvestigation of the Substitutions Reaction of Stereogenic Phosphoryl Compounds:
Stereochemistry, Mechanism and Applications**

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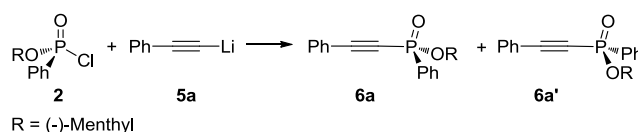
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Part 1. The substitution of P-Cl bond with alkynyl metallic reagents.

Typical procedure for the reaction of **2** with **5a**:

The powder of **2** (0.046 g, 0.147 mmol) was added portionwise within 1 minute to the solution of **3a** that was prepared in situ from addition of *n*BuLi (1.6 M solution in hexane, 0.092 ml, 0.147 mmol) to the solution of phenylethyne (0.016 ml, 0.147 mmol) in ether (1 ml) at 0 °C. Then the mixture was stirred for about 3 h while the temperature elevated to room temperature gradually. Saturated ammonium chloride solution was added, and the mixture was extracted with dichloromethane for three times. The combined organic layer was dried over sodium sulfate, concentrated in vacuo. The residue was used for analysis with NMR. The yields and diastereomeric ratio were estimated by ³¹P{¹H}-NMR spectroscopy based on **2**.

Table S1. Reaction of **2 with **5a** under various conditions**



Entry	Temperature/Time	Yield % (6a/6a') (2/2') ^a
1	0 °C to rt/3 h	>99 (89:11) ^b
2	0 °C to rt/4 h	80 (99: 1)
3	0 °C/1 h	96 (68:32) ^c
4	rt/4 h	95 (53:47)
5	−80 °C/1 h; to rt/16 h	96 (45:55) ^{c,d}
6	−20 °C/5 min; to rt/5 h	>99 (70:30) ^{c, d}
7	−20 °C/1 h; to rt/16 h	91 (40:60) ^d
8	−20 °C/9 h; rt/1 h	99 (42: 58)
9	−20 °C/9 h; rt/16 h	99 (40: 60)
10	−20 °C/9 h	97 (51:49) ^e
11	−45 °C/8 h	99 (>99:1)
12	−15 °C/2.5 h; rt/5 min	99 (51:49) ^f
13	0 °C/20 min; rt /2.5 h	37 (69:31) (68:32) ^{c, g}
14	−15 °C /3 h; rt/5 min	63 (80:20) (81:19) ^g
15	−15 °C /3 h; rt/16 h	64 (83:17) (50:50) ^g
16	−20 °C /3 h; rt/5 min	61 (62:38) (91:9) ^g

^a In a typical procedure, the powder of **2** was added portionwise within 1 minute to the solution of **5a**. The yields and dr were estimated by the peaks' integrations on ³¹P and proton NMR spectroscopy. The data in second parentheses was the ratio of unconsumed **2/2'** (if applicable). ^b **5a** was added to the solution of **2** (0.147 M in ether). ^c Solid of **2** added in one portion. ^d The mixture was warmed from an ice-water bath to rt. ^e The mixture was quenched with acetic acid at −20 °C. ^f Both two reactants were used in 0.0735 M solution of ether. ^g The ratio of **5a/2** was 1:2.

A diastereomeric mixture of (*S*_P)-**2**/ (*R*_P)-**2'** reacted with phenylethynyl lithium **5a**, affording two stereoisomers of *O*-menthyl phenyl (phenylethynyl) phosphinate **6a** and **6a'** whose ³¹P-NMR signals were observed at 7.78 (s) and 9.37 ppm (s), respectively. The yield and dr (ratio of **6a/6a'**) for the reaction of optically pure (*S*_P)-**2** with **5a** under various conditions were examined (Table S-1). When **2** was slowly added to **5a** at 0 °C, **6a** was formed with greater than 99:1 dr. The

reverse addition resulted in poor dr (entries 1–3), which indicated the dr was sensitive to the stoichiometry of the two starting materials. In entries 1 to 11, a temperature dependence of the dr was observed. When the reaction was carried out at either rt or $-20\text{ }^{\circ}\text{C}$ (entries 4 to 10), poor dr was obtained. When **2** and **5a** were mixed at $-80\text{ }^{\circ}\text{C}$, then gradually warmed to rt, **6a/6a'** were still formed with poor dr (entry 5), in contrast to the normal thinking that better selectivity is obtained at low temperature, as seen in Imamoto's reaction at $-78\text{ }^{\circ}\text{C}$. However, when **2** and **5a** were stirred at $-45\text{ }^{\circ}\text{C}$ for enough time (8 h), **6a** was formed in near quantitative conversion and $>99 : 1$ dr (entry 11).

After **2** and **5a** were mixed at $-80\text{ }^{\circ}\text{C}$ in ether, the solution was monitored with ^{31}P -NMR spectral. At beginning stage at rt, only the peak of **6a** was observed. The peaks of unconsumed **2**, its epimer **2'**, **6a'** and other *P*-containing species were not detected. The peak of **6a'** started to emerge after five minutes, and gradually increased with prolonged time, until 59:41 dr after an hour (Figure S-1).

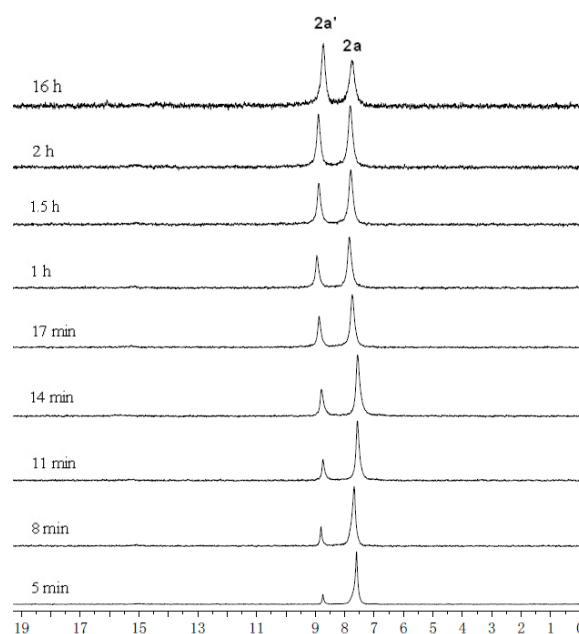
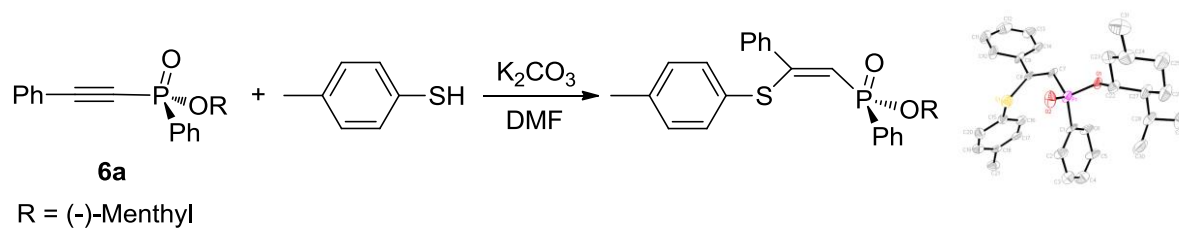


Figure S1. Relationship between the formation of **6a/6a'** and the reaction time (in ether).

In dilute solution, **6a'** was not dominantly formed (entry 12). The results indicated a dilute solution, which was developed after formation of **6a**, was not favour for the formation of **6a'**. **6a'** was not produced from the reported chloride-exchange or epimerization of **1**, as proved by no epimerization of **1** was observed in the LiCl-containing solution.

The reaction of 4-methylbenzenethiol with **6a** under alkali condition to afford menthyl (*Z*)-2-(*p*-tolylthio)-2-phenylvinyl (phenyl)phosphinate, whose S_{P} configuration was confirmed by X-ray diffraction (Scheme S-1). Because the addition didn't involve in phosphorus atom, the phosphorus atom of **6a** has the same R_{P} configuration. On the basis of the above observations, two routes to form **6a** and **6a'** were proposed as *P*-inversed normal $S_{\text{N}}2$ substitution (route A, to form **6a**) and *P*-retained Berry pseudorotation (BPR, route B, to form **6a'**), respectively.



Scheme S1. The conversion of 6a and the confirmation of its structure.

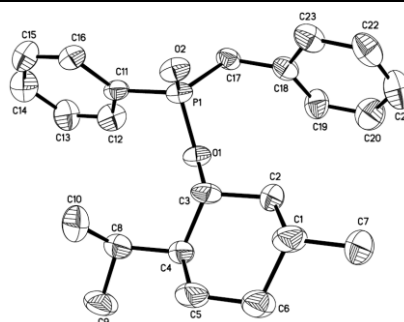
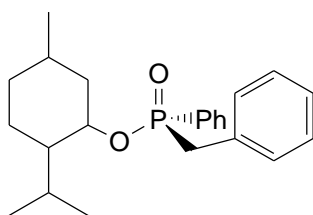
Part 2. The comparison of ^{31}P NMR spectroscopy for **12** and **12'**

Table S2. The comparison of ^{31}P -NMR spectrum for the R_P/S_P -**12/12'**.

entry	R/major product	(S_P)- 12	(R_P)- 12'
		^{31}P -NMR spectrum (ppm):	^{31}P -NMR spectrum (ppm):
1	Me (12a)	40.74 (s, 90 %)	40.05 (s, 10 %).
2	Et (12b)	45.19 (s, 98 %)	44.20 (s, 2 %).
3	<i>n</i> -Bu (12c)	43.97 (s, 98 %)	43.05 (s, 2%).
4	<i>i</i> -Pr (12d)	47.70.	
5	<i>cyclo</i> -Hex (12e)	44.96.	
6	<i>s</i> -Bu (12f)	47.17 and 46.79 (two single peaks, 88.5%)	45.86 and 45.62 (two single peaks, 11.5%)
7	<i>t</i> -Bu (12g)	48.98.	
8	Bn (12h)	38.41.	
9	allyl (12i)	39.02 (s, 98 %)	38.47 (s, 2 %)
10	<i>p</i> -BrC ₆ H ₄ (12j)	(R_P) 28.96.	
11	<i>p</i> -ClC ₆ H ₄ (12k)	(R_P) 28.84	
12	12k/12k' (from 2/2')	(R_P) 29.000	(S_P) 28.955
13	<i>o</i> -MeC ₆ H ₄ (12l)	(R_P) 25.67	
14	12m	(R_P) 28.84	
15	Me (12a')		40.41
16	Et (12b')		44.20.
17	<i>n</i> -Bu (12c')		42.99.
18	<i>i</i> -Pr (12d')	47.61 (s, 3%)	δ 46.41 (s, 97%),
19	<i>s</i> -Bu (12f')	47.32 and 46.94 (two single peaks, 11%)	45.98 and 45.75 (two single peaks, 89%).
20	<i>t</i> -Bu (12g')	48.99 (s, 13%)	48.19 (s, 87%),
21	Bn (12h')		37.63
22	allyl (12i')	39.08 (s, 4%)	38.53 (s, 96%).
23	<i>p</i> -BrC ₆ H ₄ (12j')	(R_P) 29.075 (s, 12%)	(S_P) 29.036 (s, 88%).
34	<i>o</i> -MeC ₆ H ₄ (12l')		(S_P) 29.02.
25	<i>p</i> -MeC ₆ H ₄ (12n')		(S_P) 29.30.

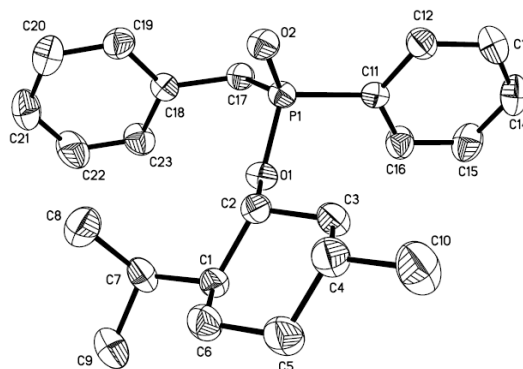
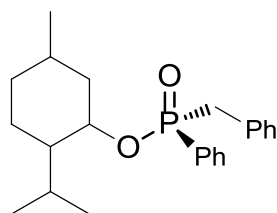
Part 3. Crystallography information.

Table S3. Crystallography data of (*S_P*)-*O*-Menthyl benzylphenylphosphinate (12h) (ORTEP drawing of 12h with thermal ellipsoids at the 30% probability)



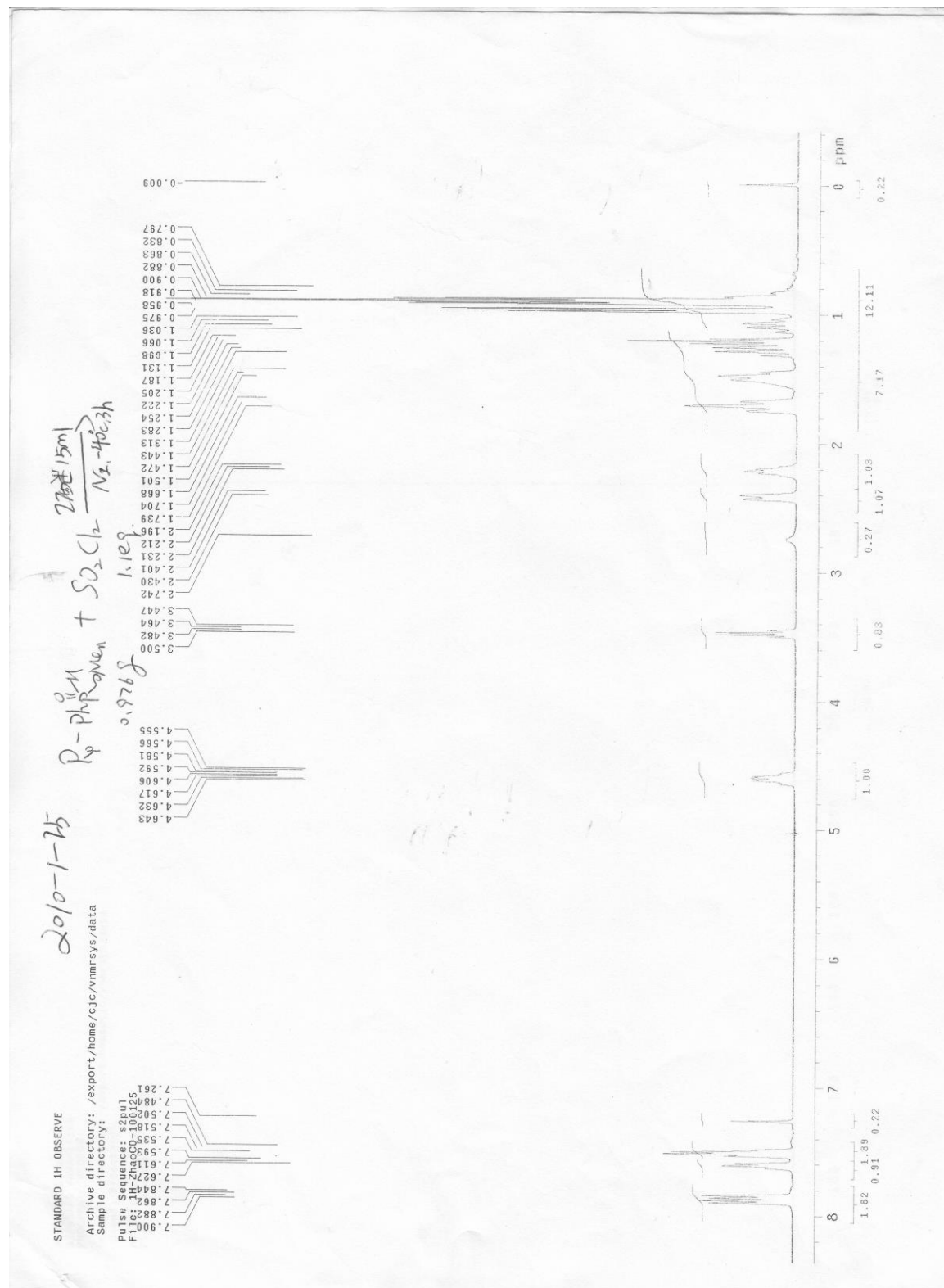
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Space group	P2 (1)2 (1)2 (1)
Formula weight	370.45
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b, Å	17.5633 (19)
c, Å	21.791 (2)
α, deg	90
β, deg	90
γ, deg	90
V, Å ³	2192.2 (4)
Z	4
T, K	298 (2)
λ, Å	0.71073
ρ, g cm ⁻³	1.122
R _{int}	0.1094
R1 [I N 2σ (I)]	0.0522
R1 (all data)	0.1436
wR2 [I N 2σ (I)]	0.1184
wR2 (all data)	0.1655
Flack	0.0 (2)
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Table S4. Crystallography data of (*R_P*)-*O*-Menthyl benzylphenylphosphinate (12h') (ORTEP drawing of 12h' with thermal ellipsoids at the 30% probability)



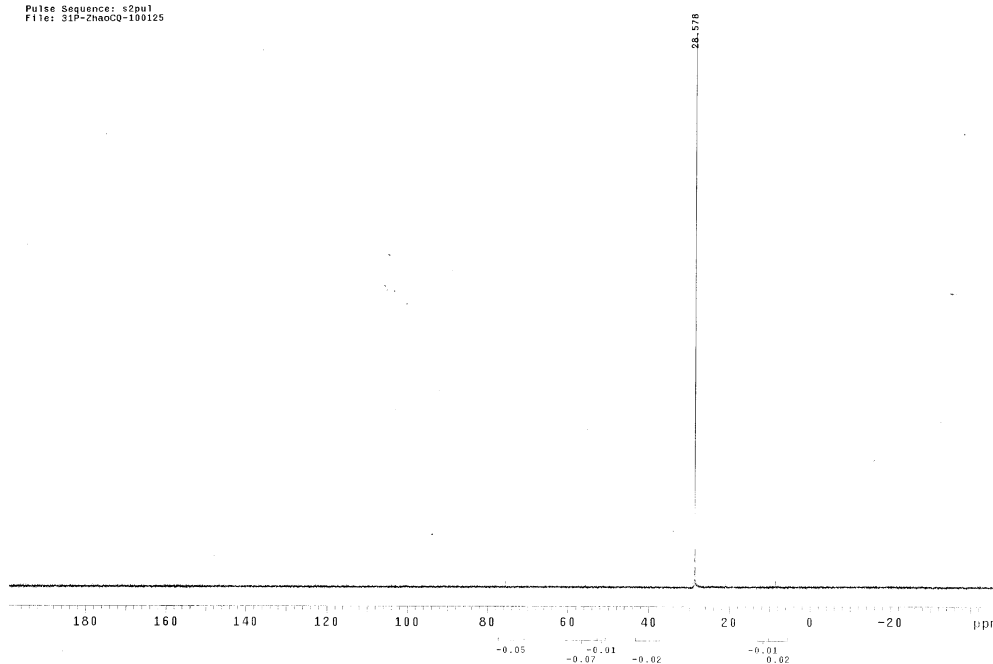
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Crystal system	Monoclinic
Space group	P2 (1)
Formula weight	370.45
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b, Å	5.7970 (7)
c, Å	15.4190 (19)
α, deg	90
β, deg	100.7270 (10)
γ, deg	90
V, Å ³	1095.8 (2)
Z	2
T, K	298 (2)
λ, Å	0.71073
ρ, g cm ⁻³	1.123
R _{int}	0.0846
R1 [I N 2σ (I)]	0.0658
R1 (all data)	0.0877
wR2 [I N 2σ (I)]	0.1567
wR2 (all data)	0.1697
Flack	-0.17 (16)
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Part 4. Selected ^1H , ^{31}P and ^{13}C NMR spectroscopy
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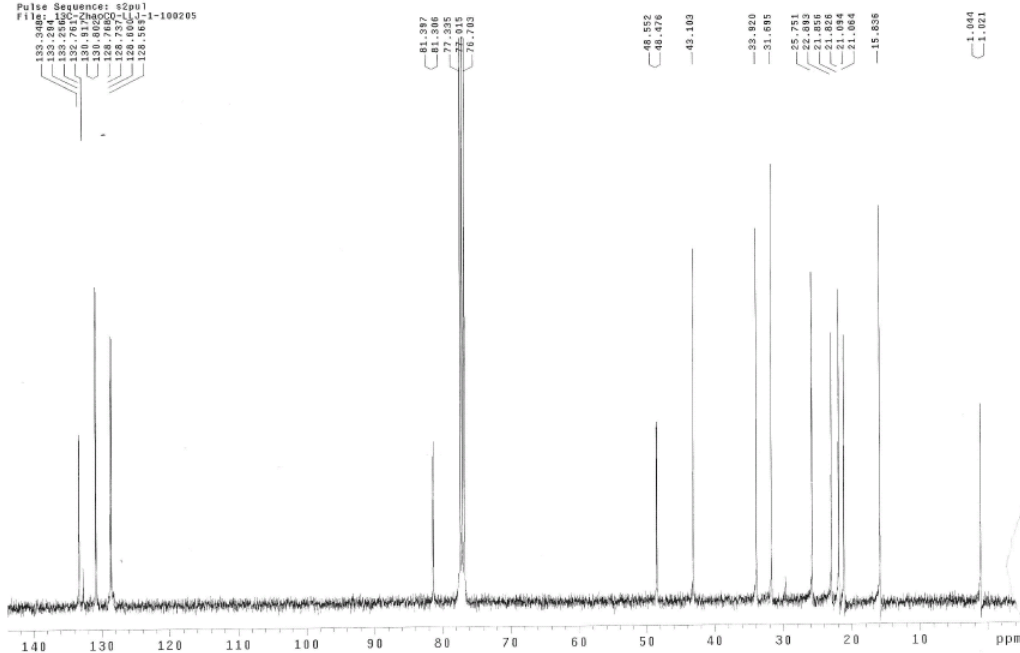
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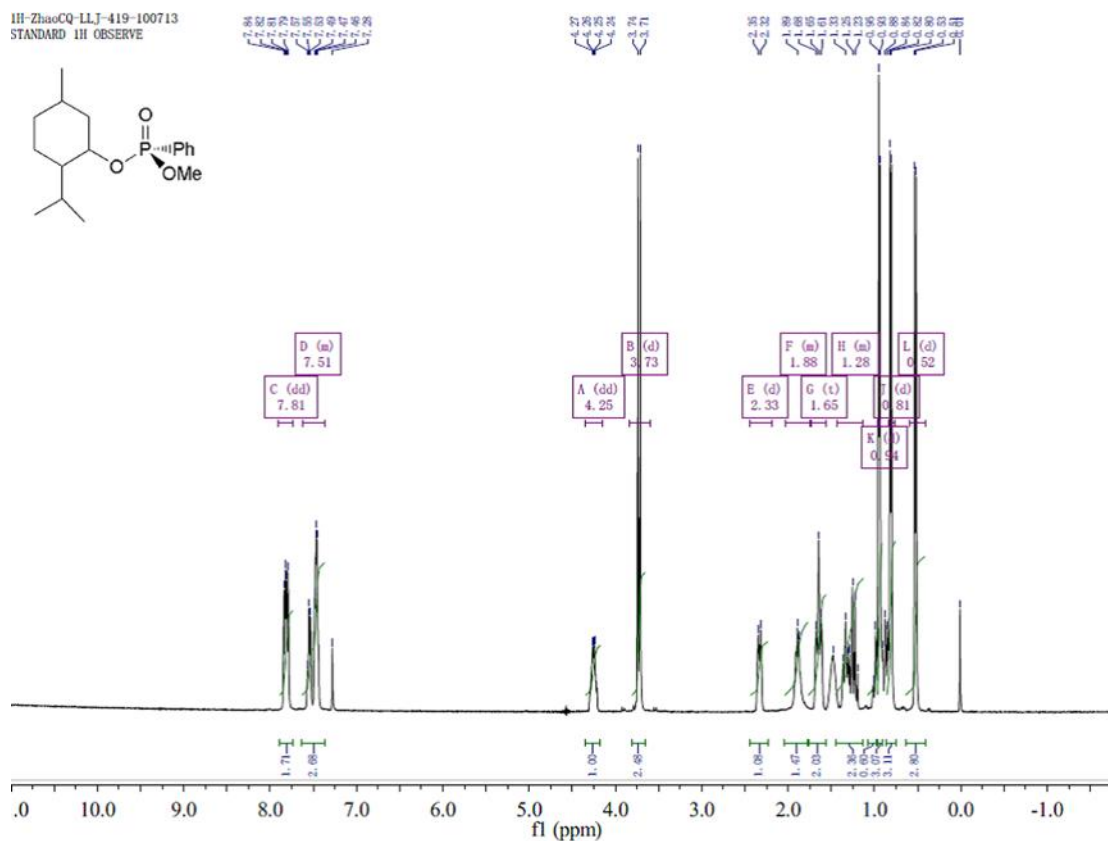
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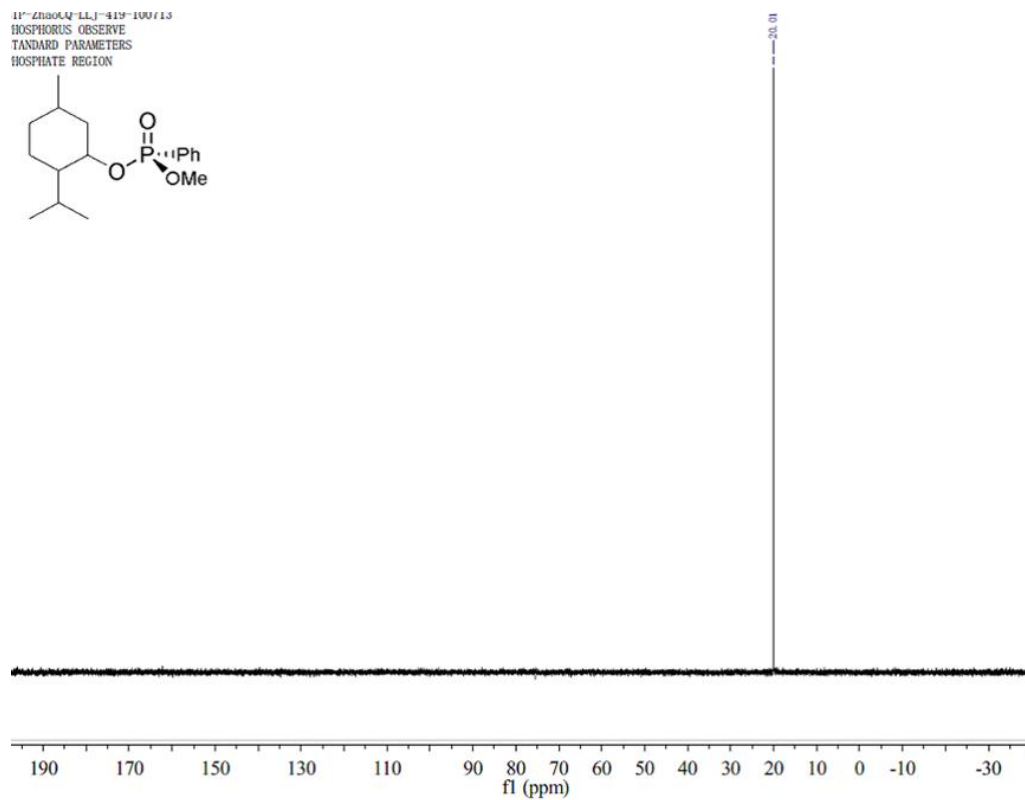
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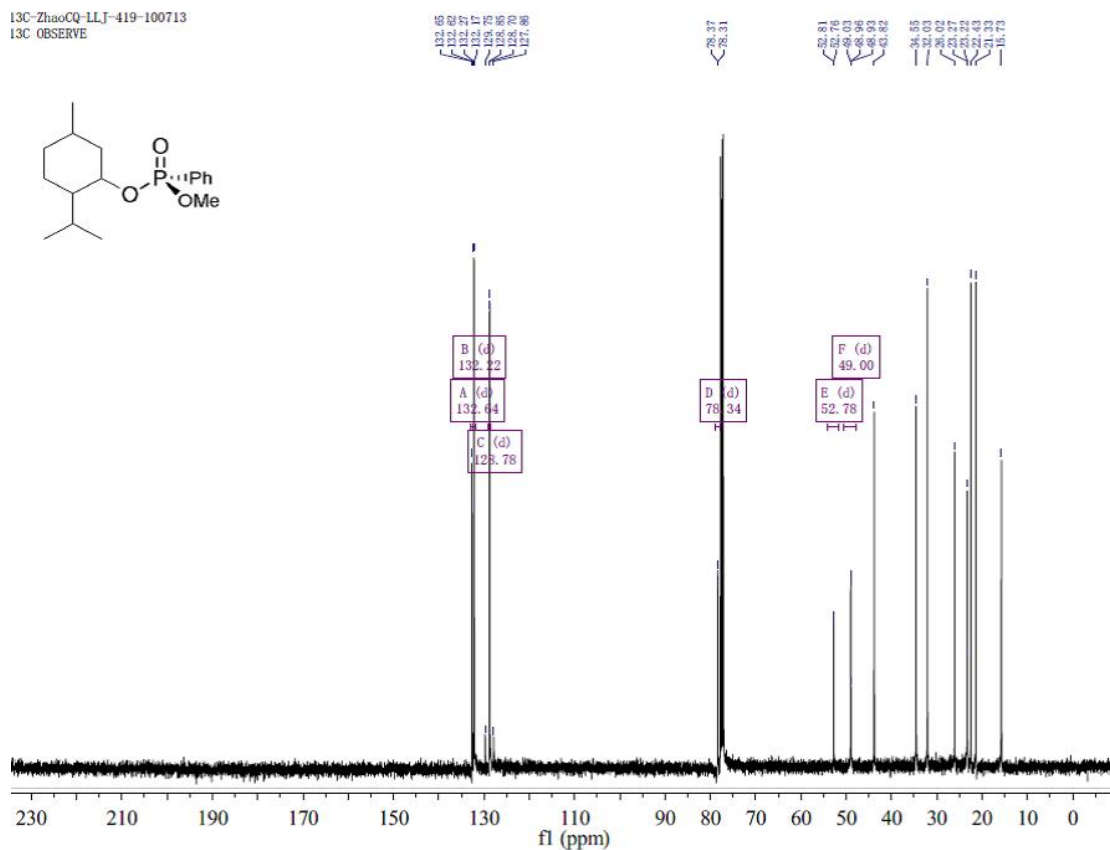
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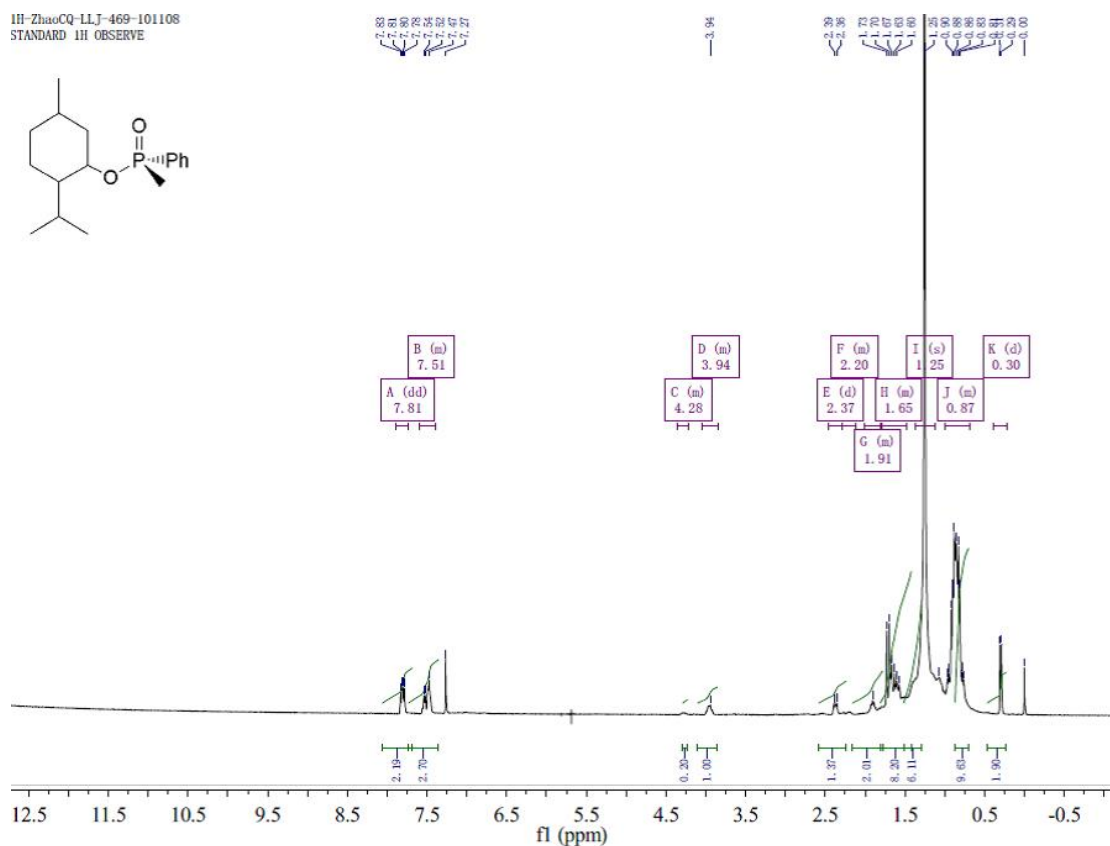
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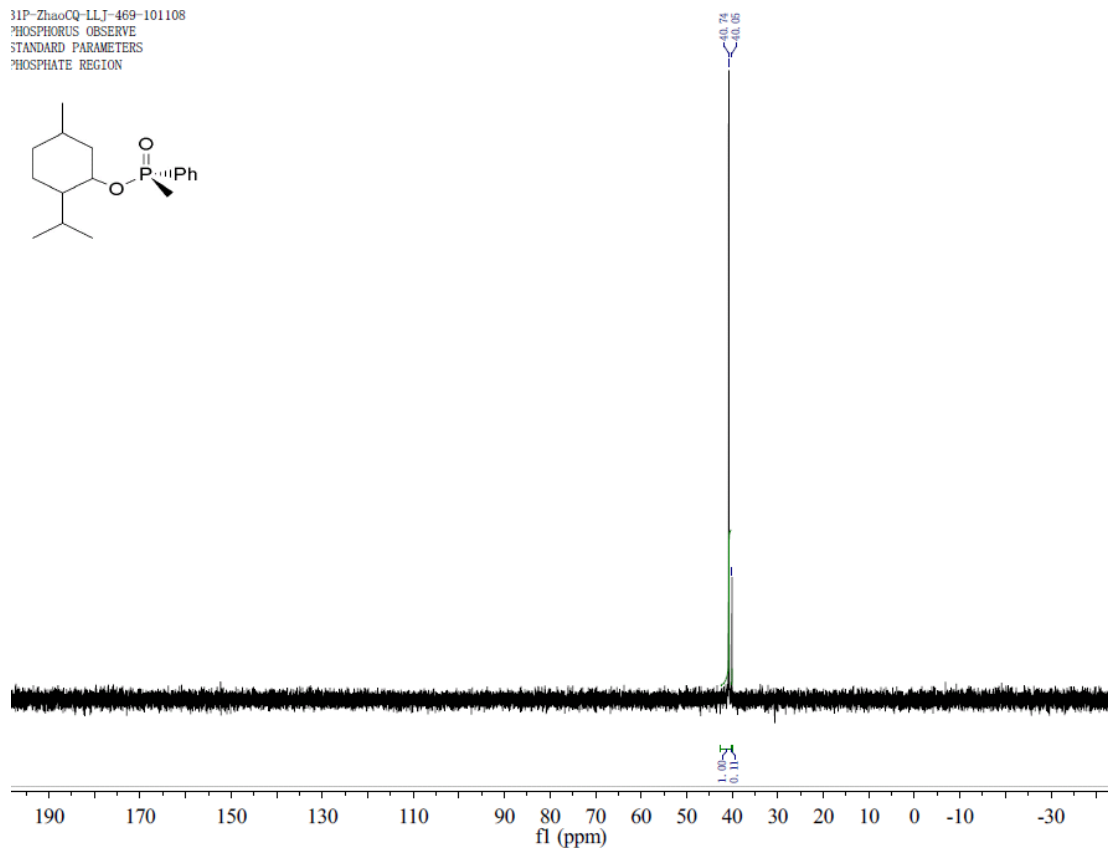
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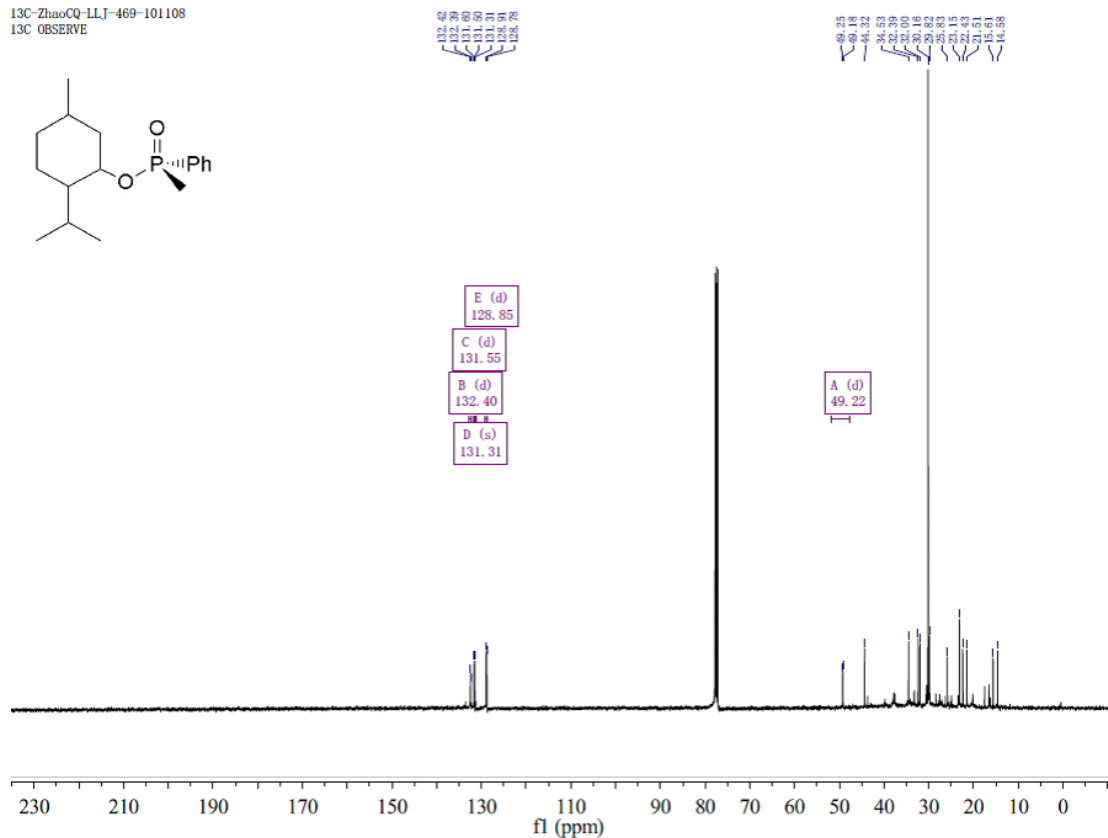
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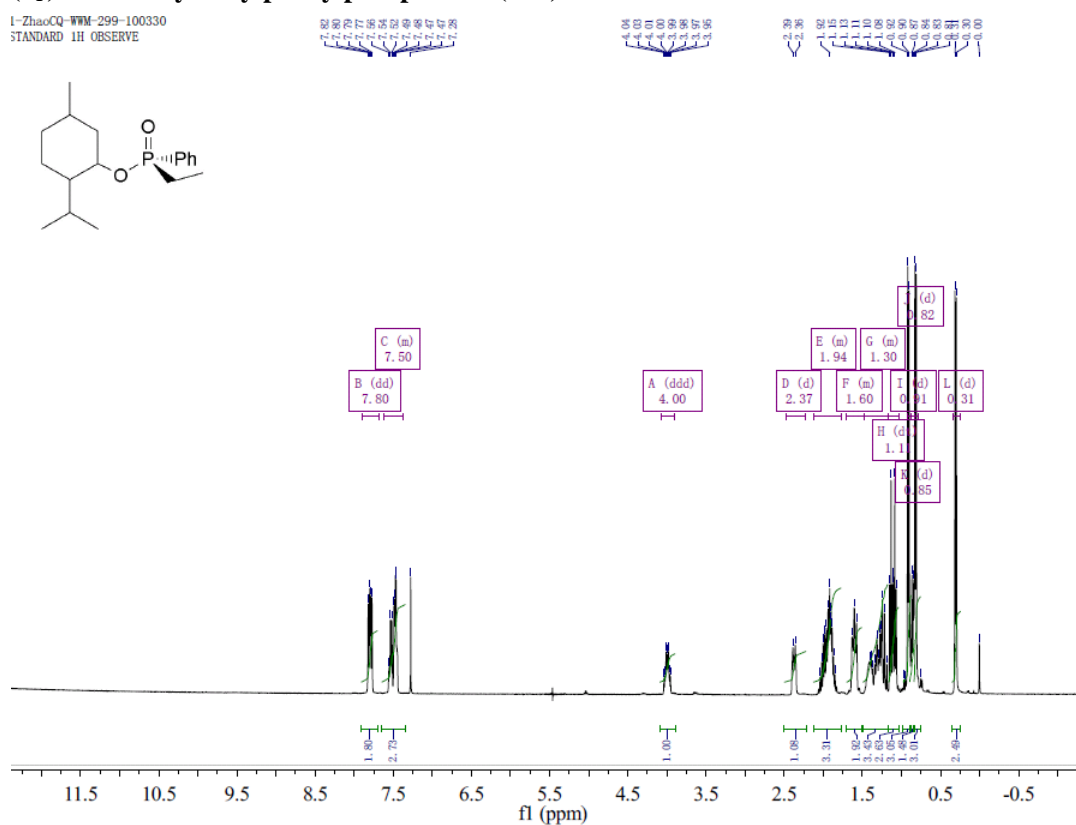
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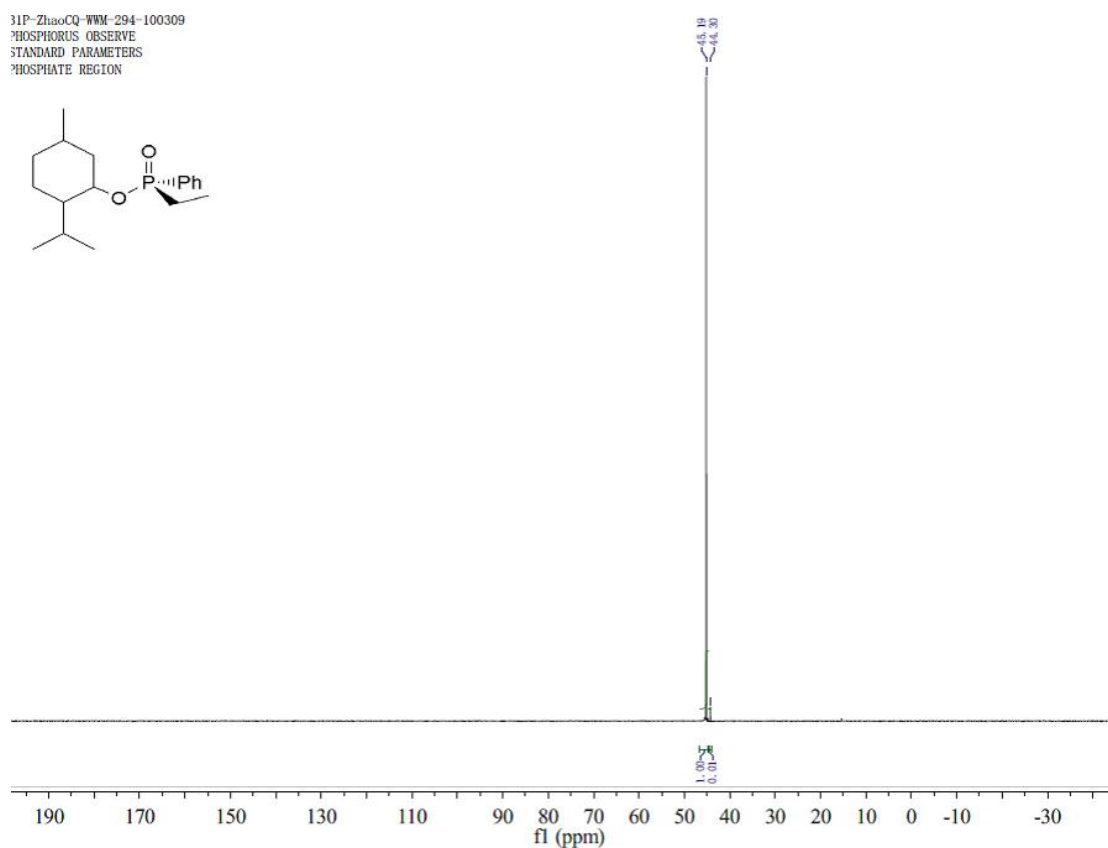
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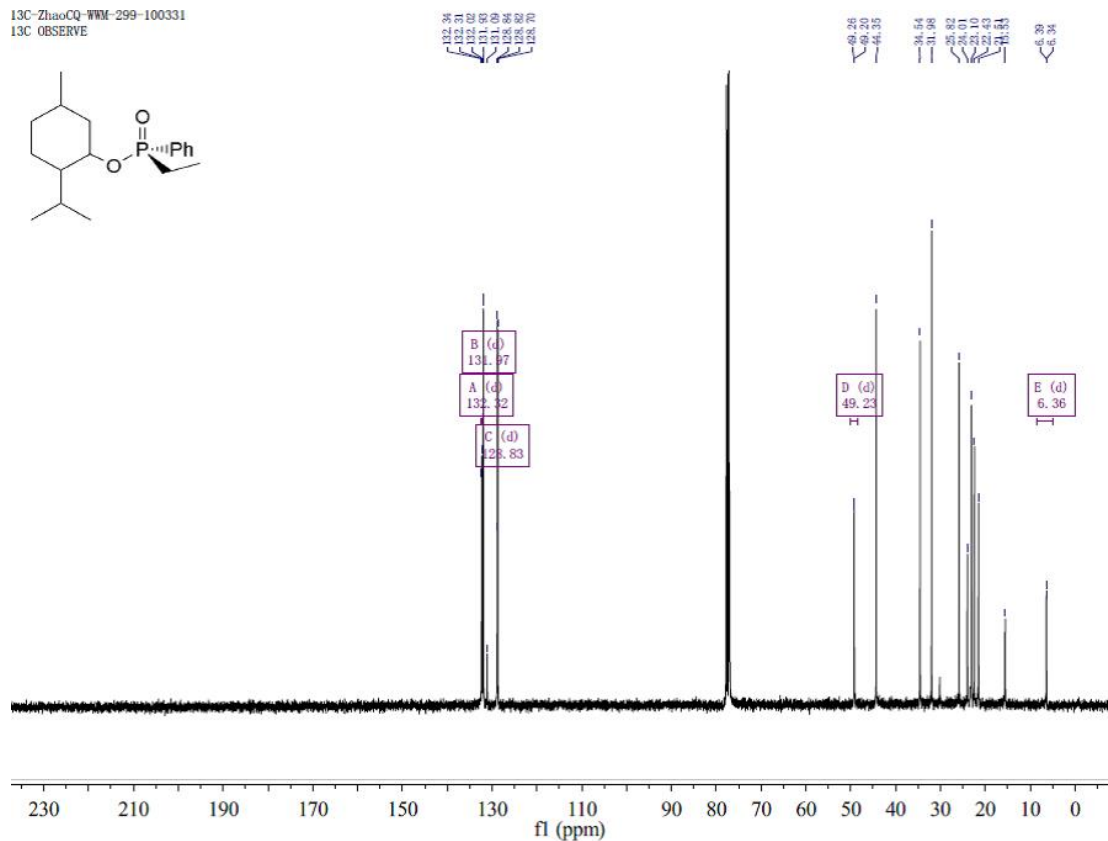
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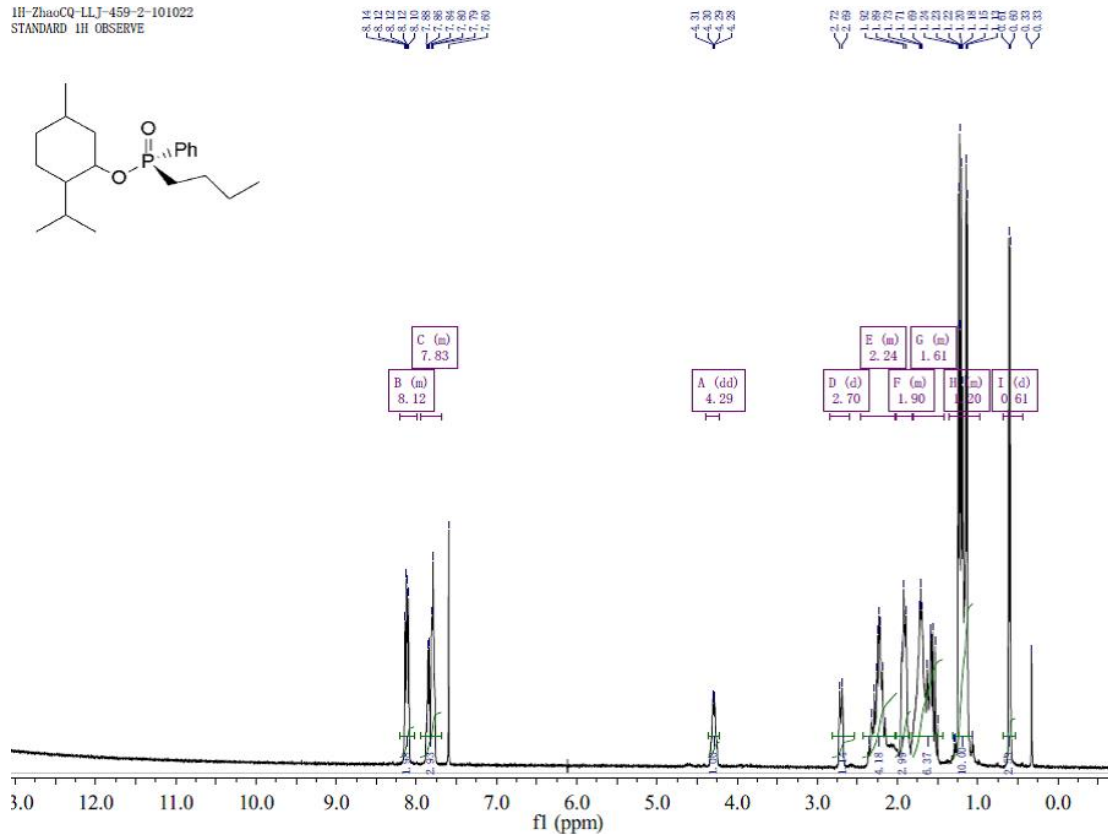
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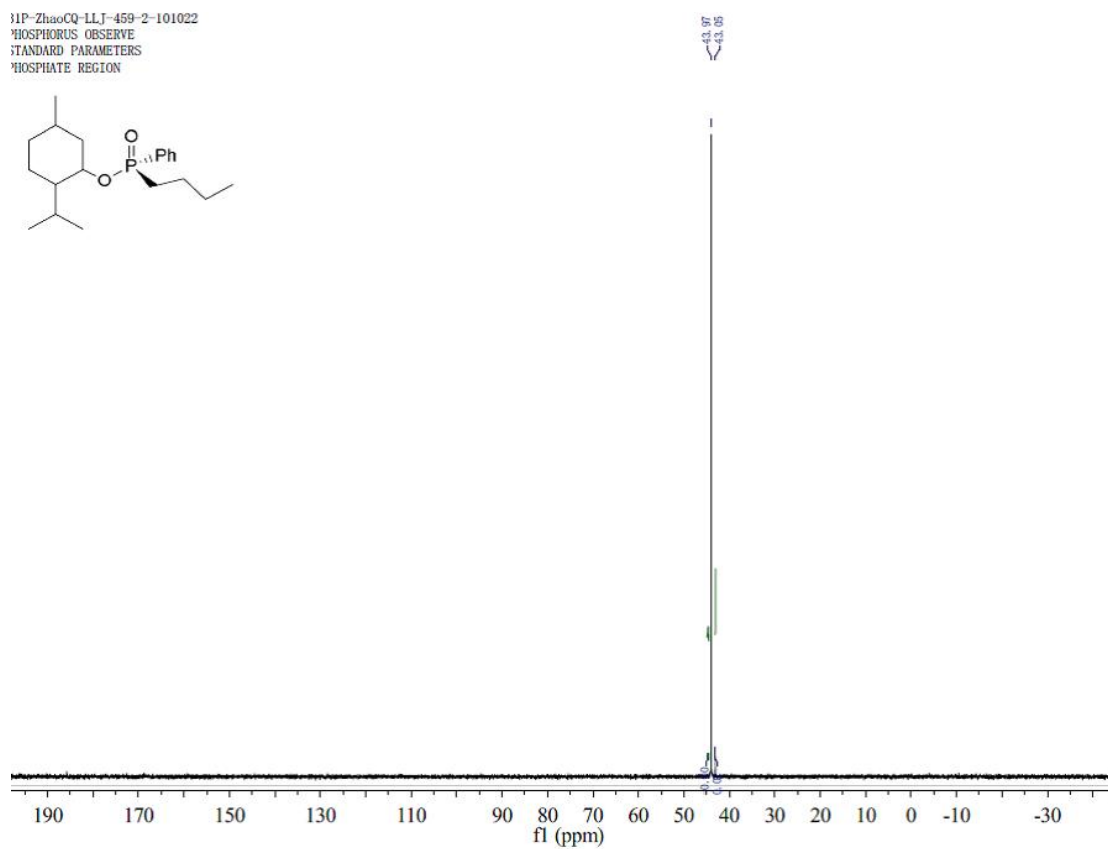
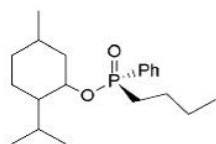
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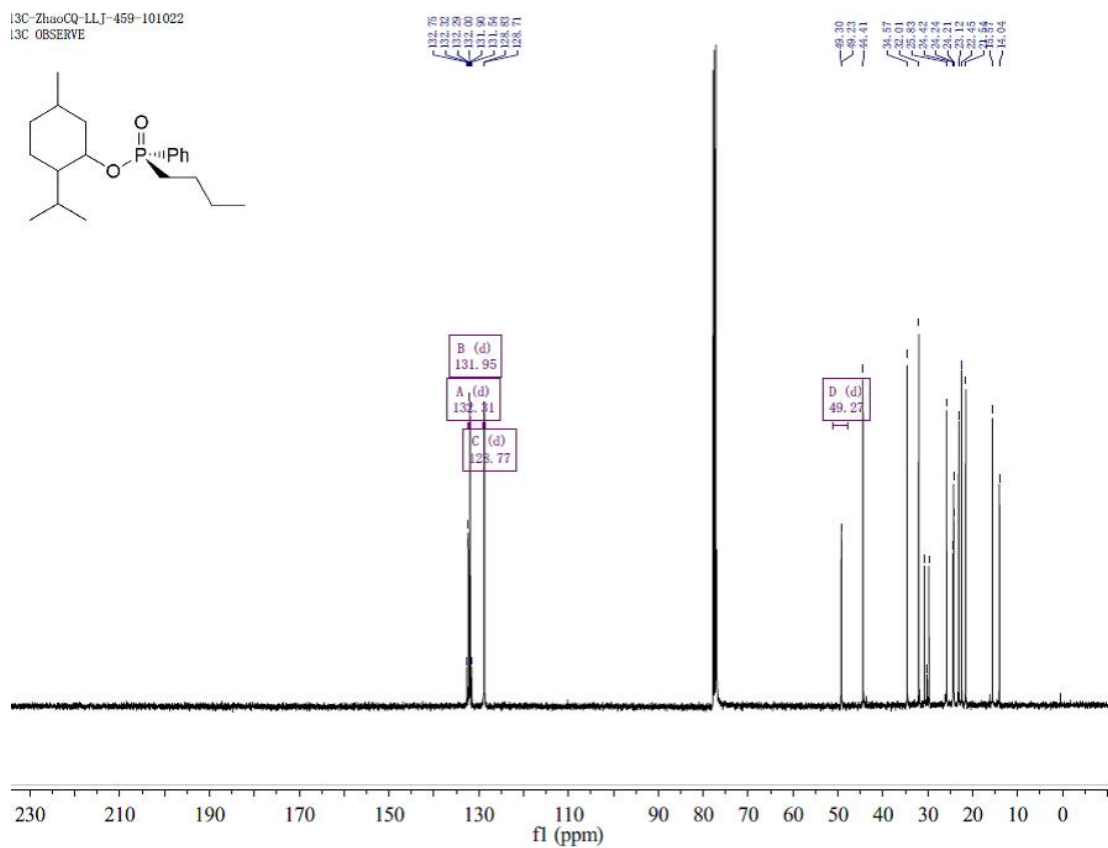
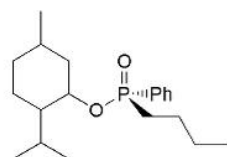
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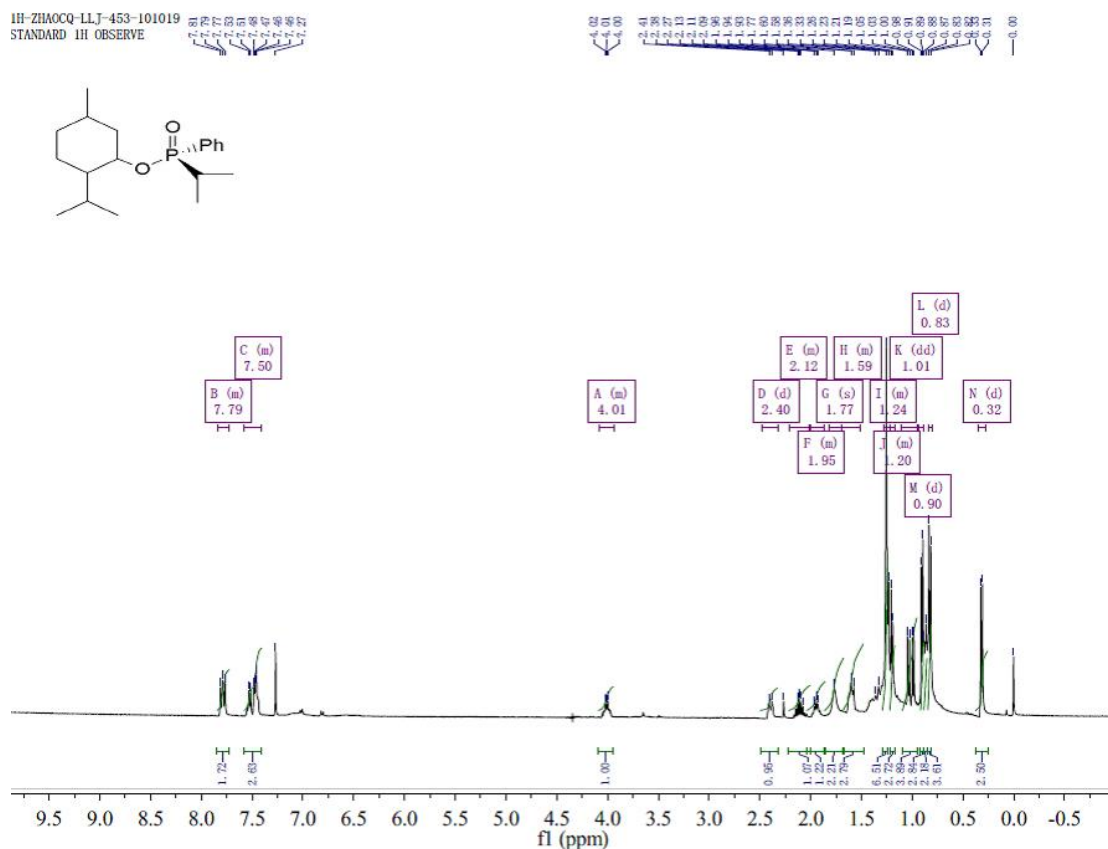
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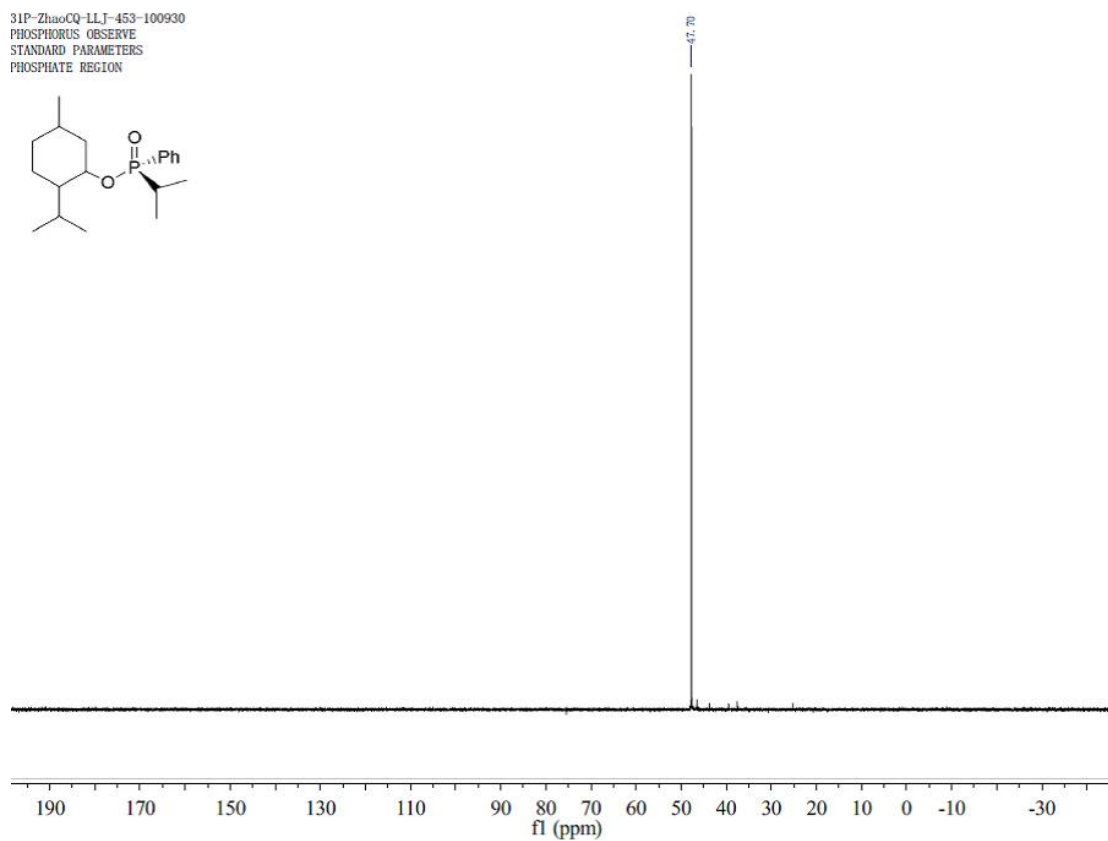
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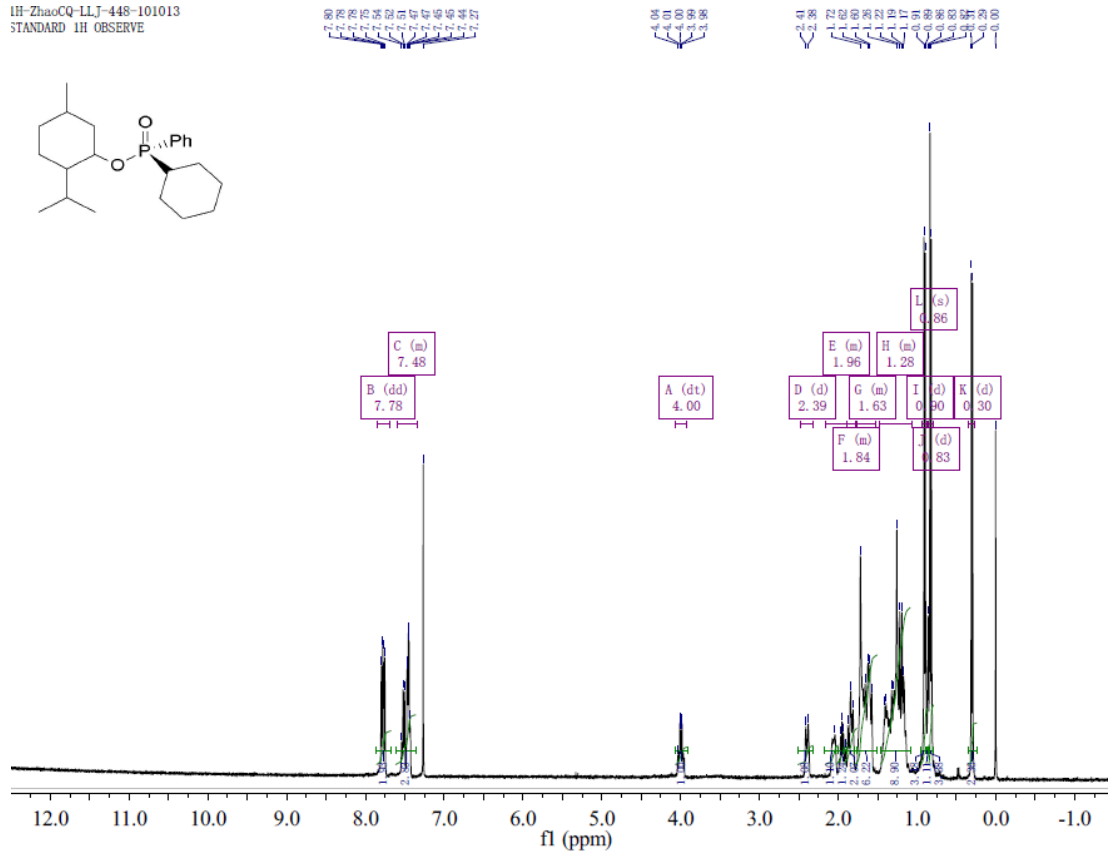


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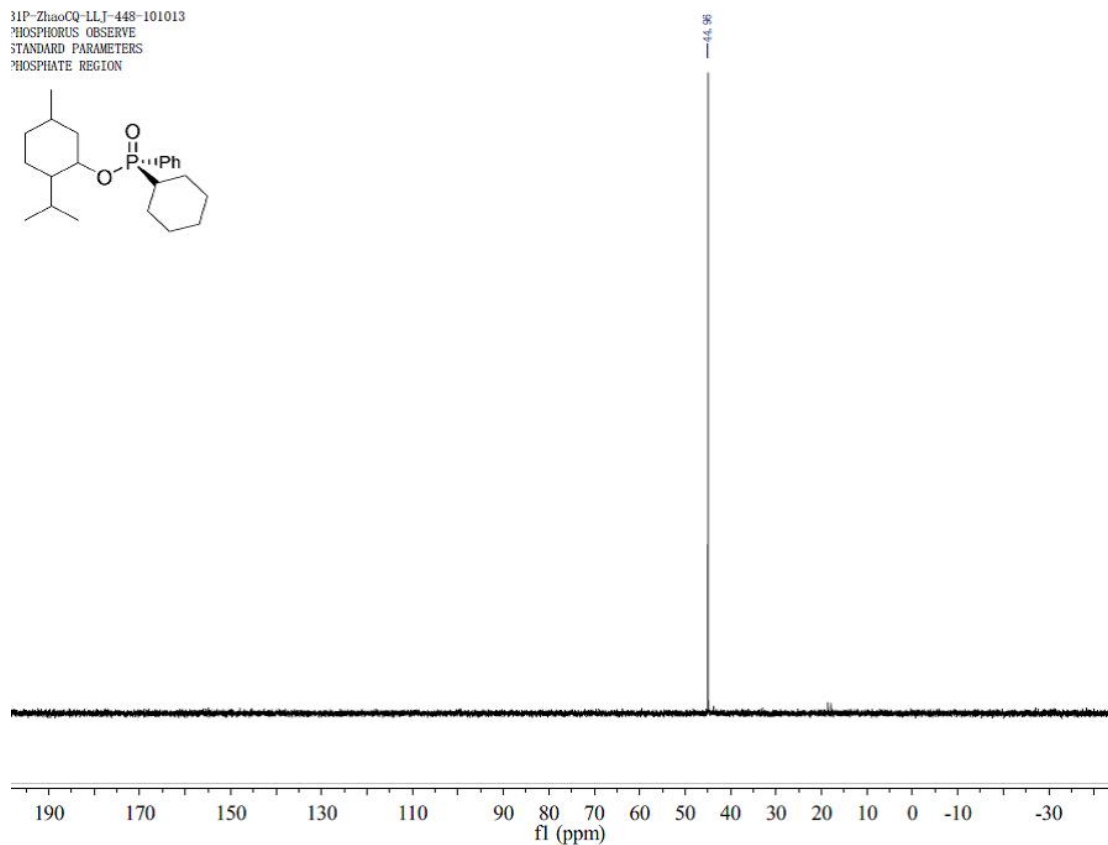
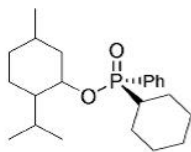


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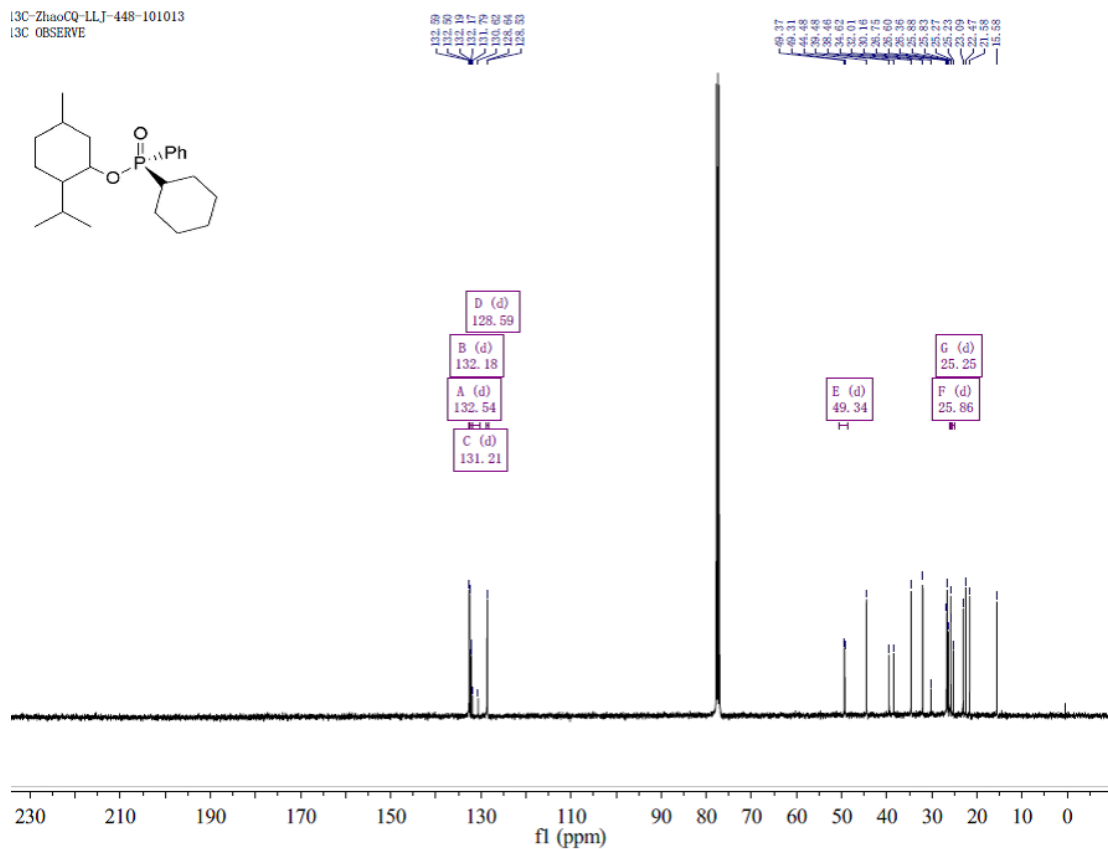
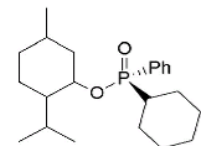
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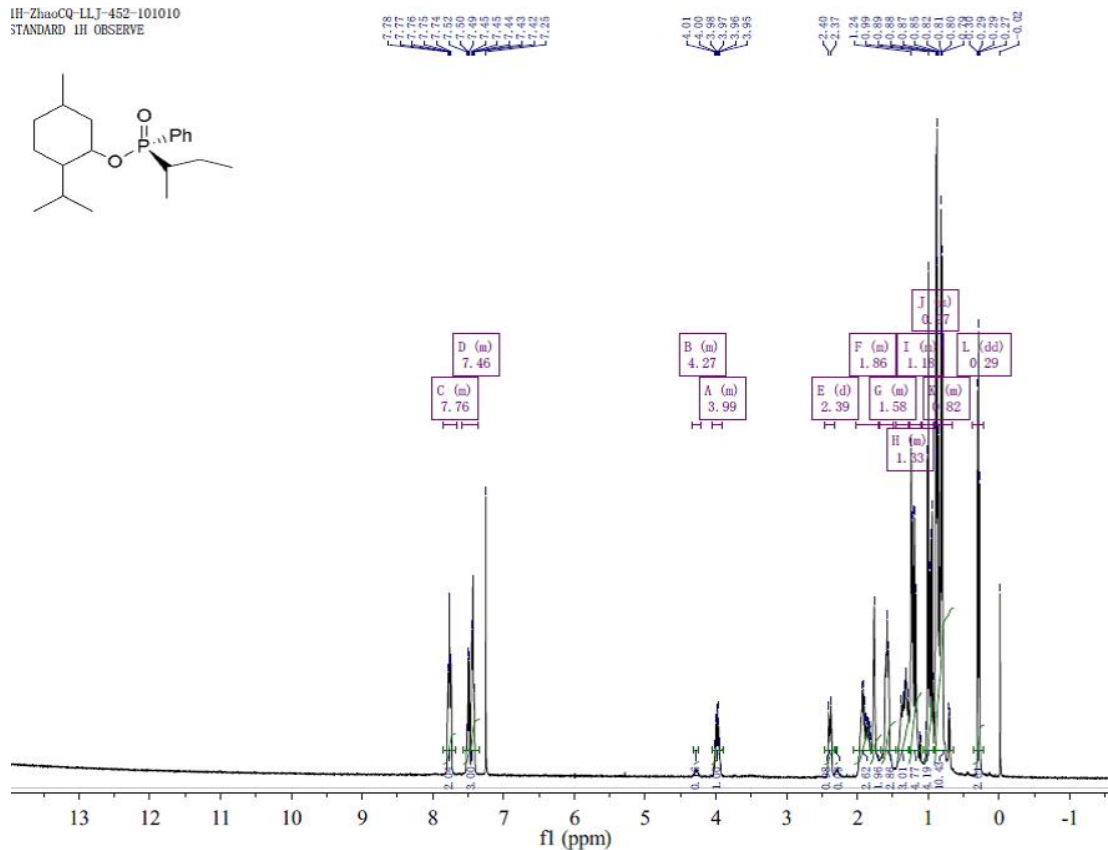
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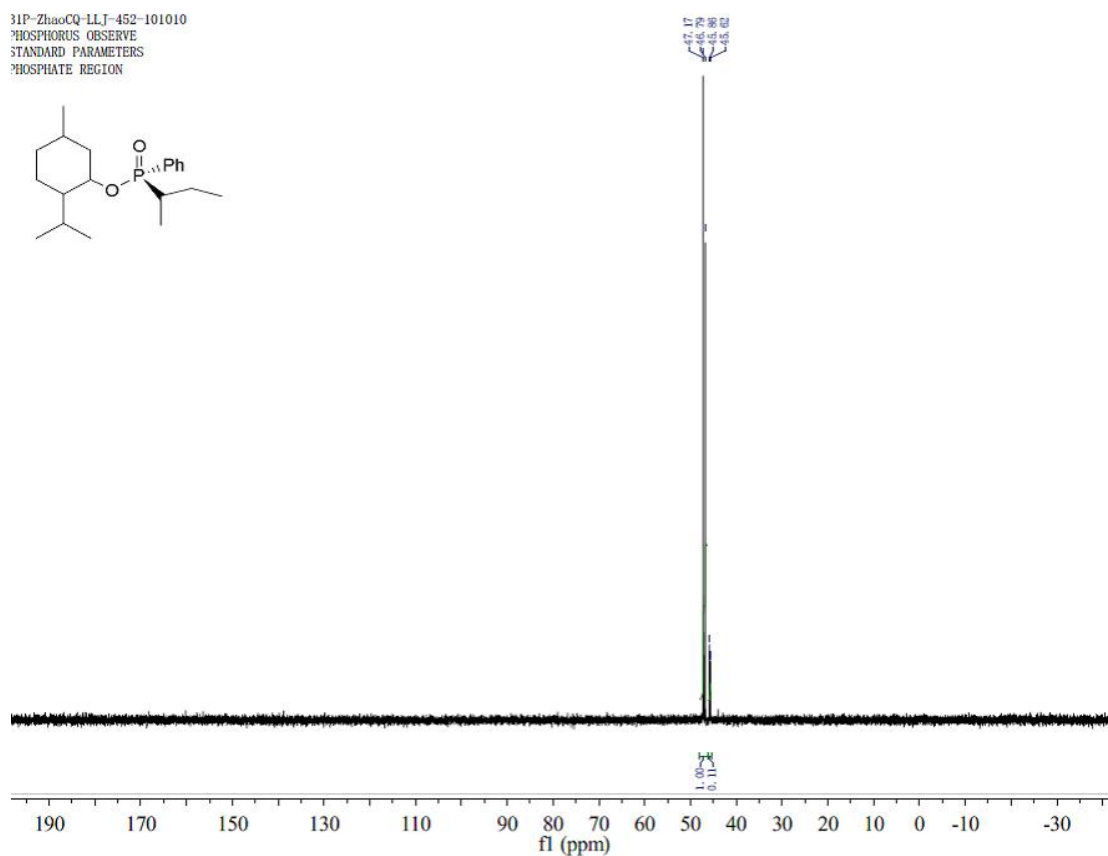
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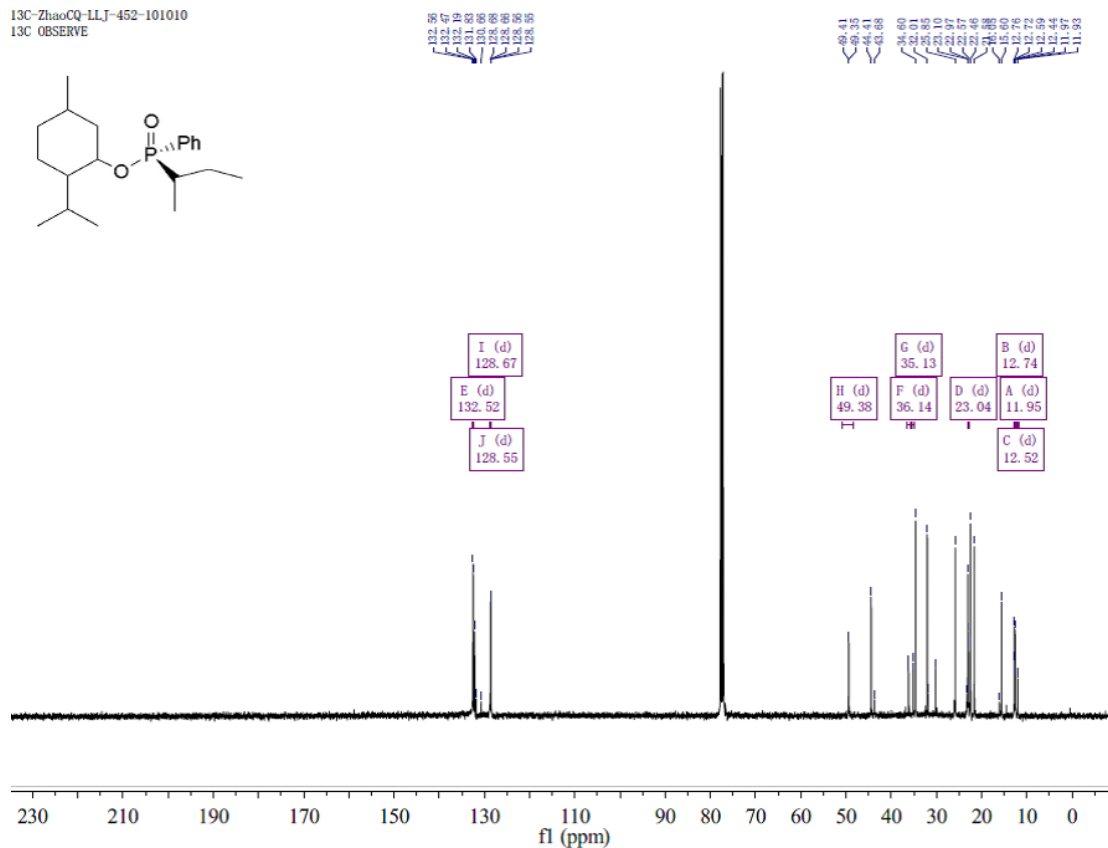
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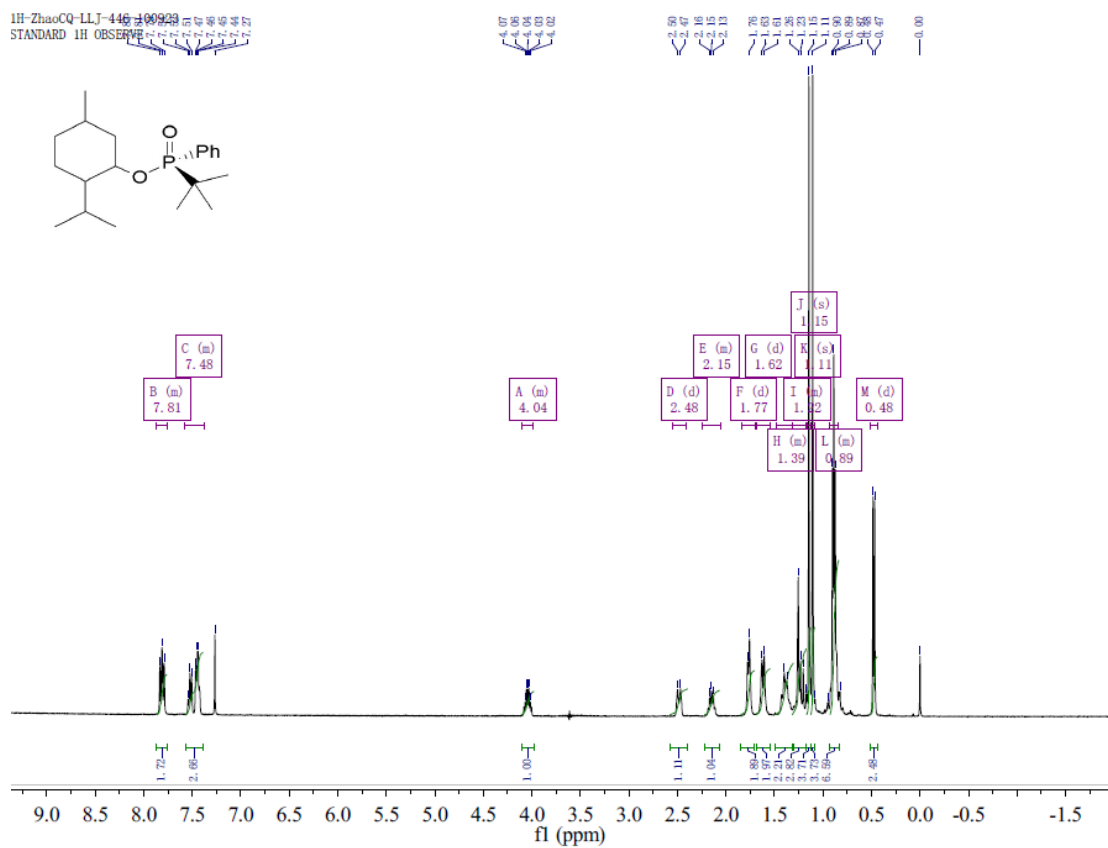
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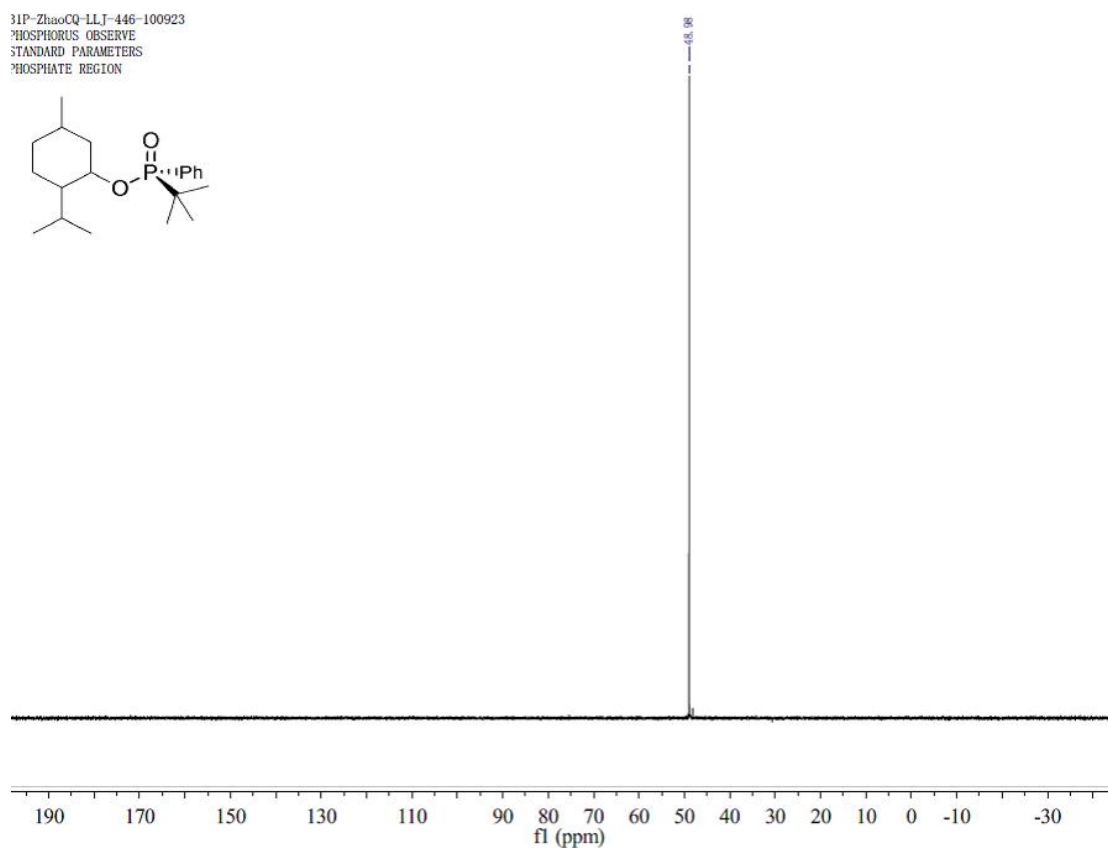
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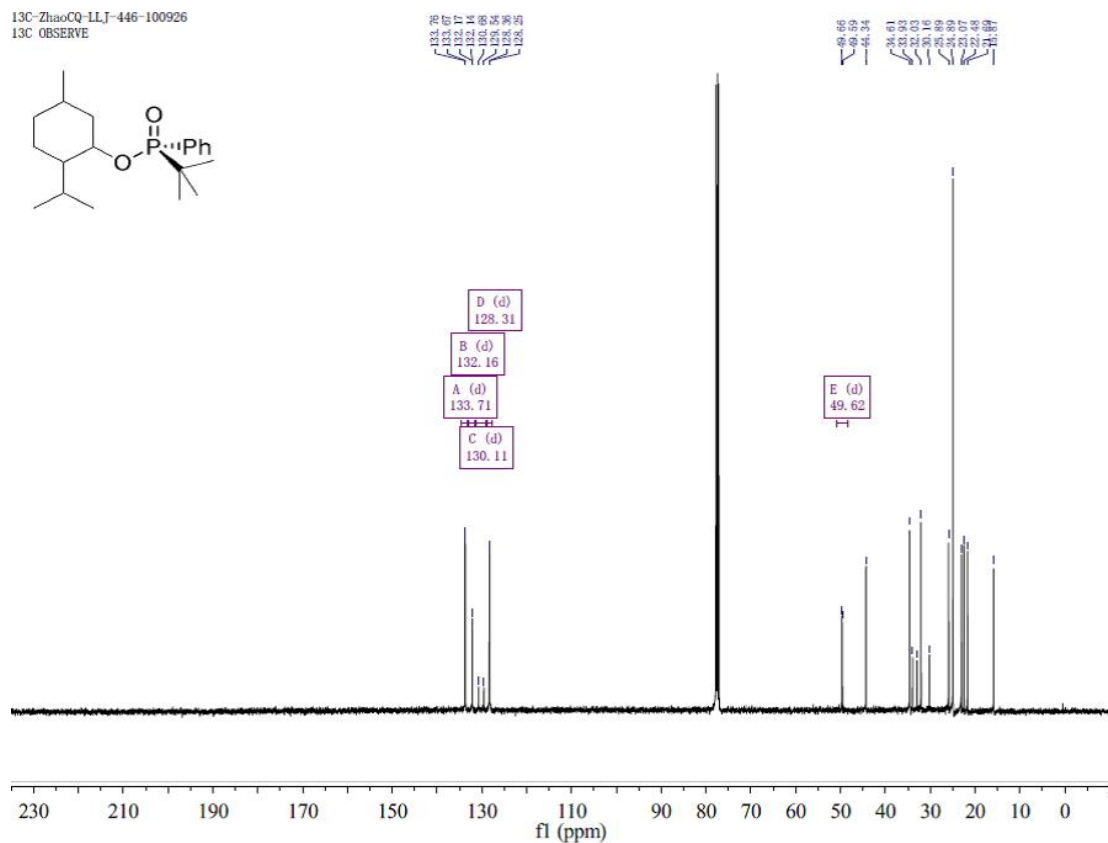
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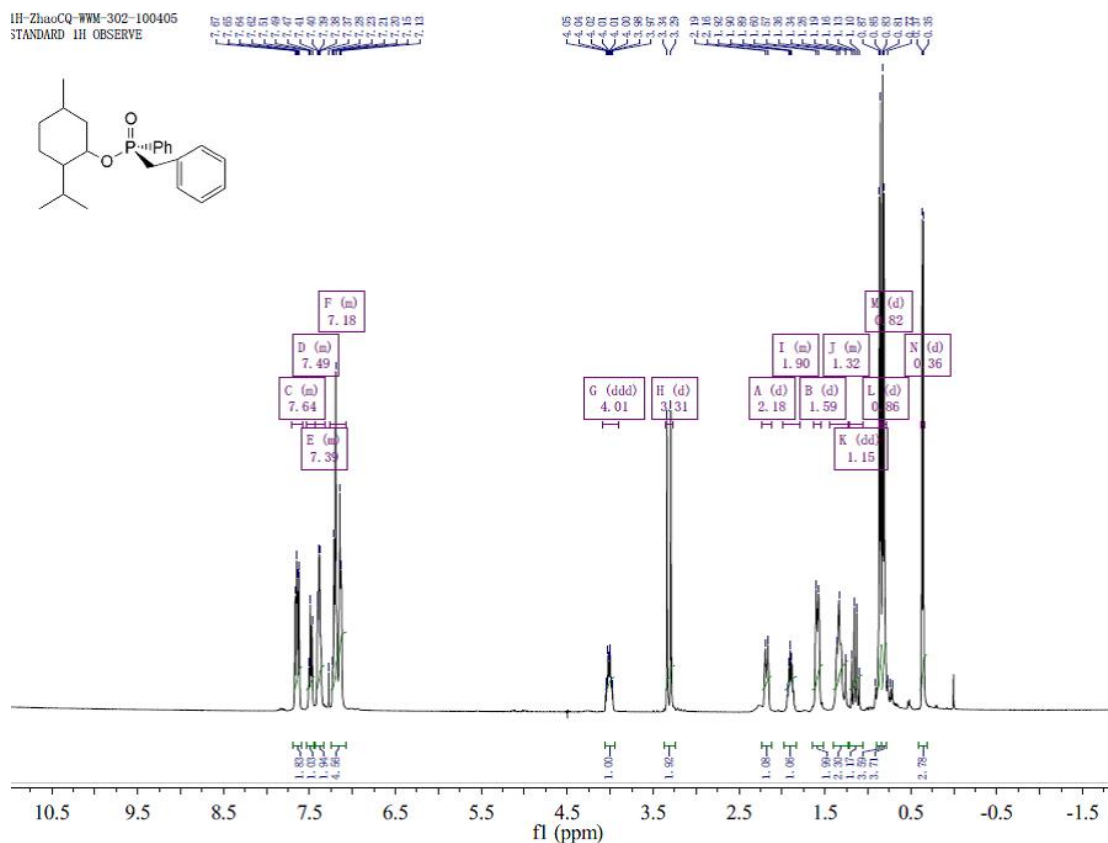
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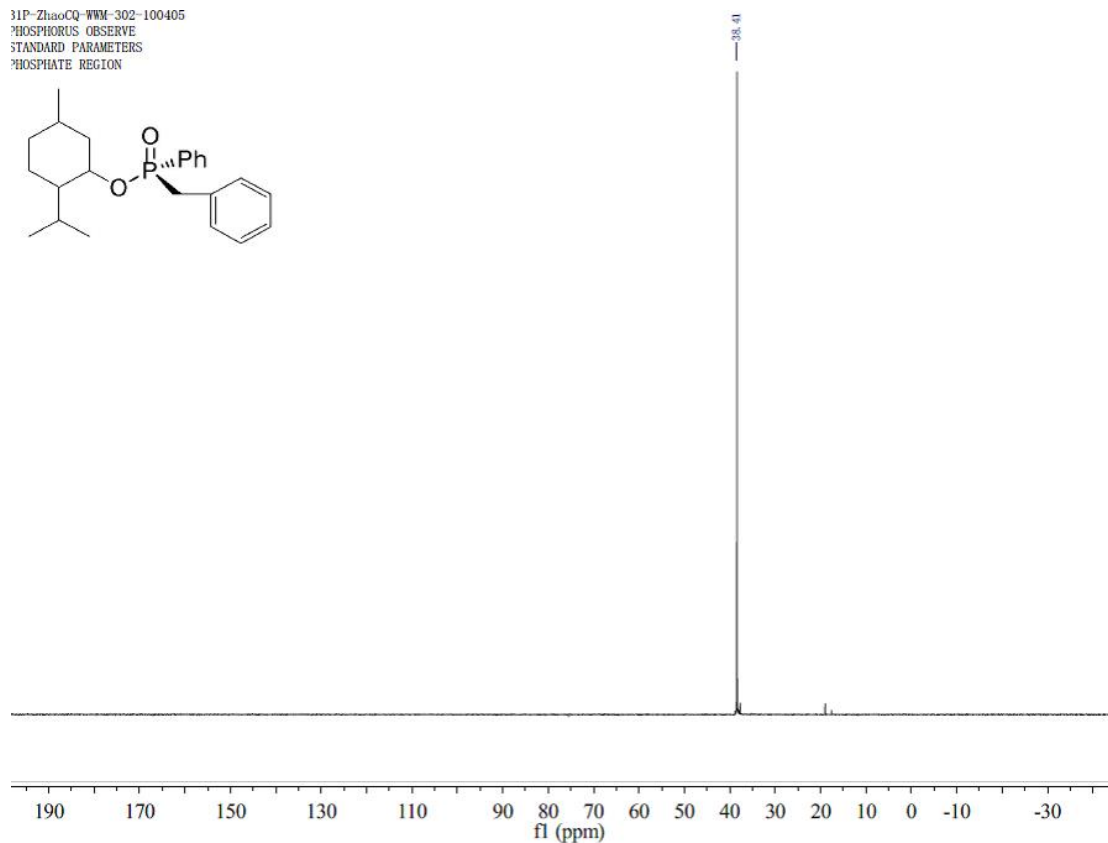
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1H-ZhaoCQ-WWM-302-100405
STANDARD 1H OBSERVE



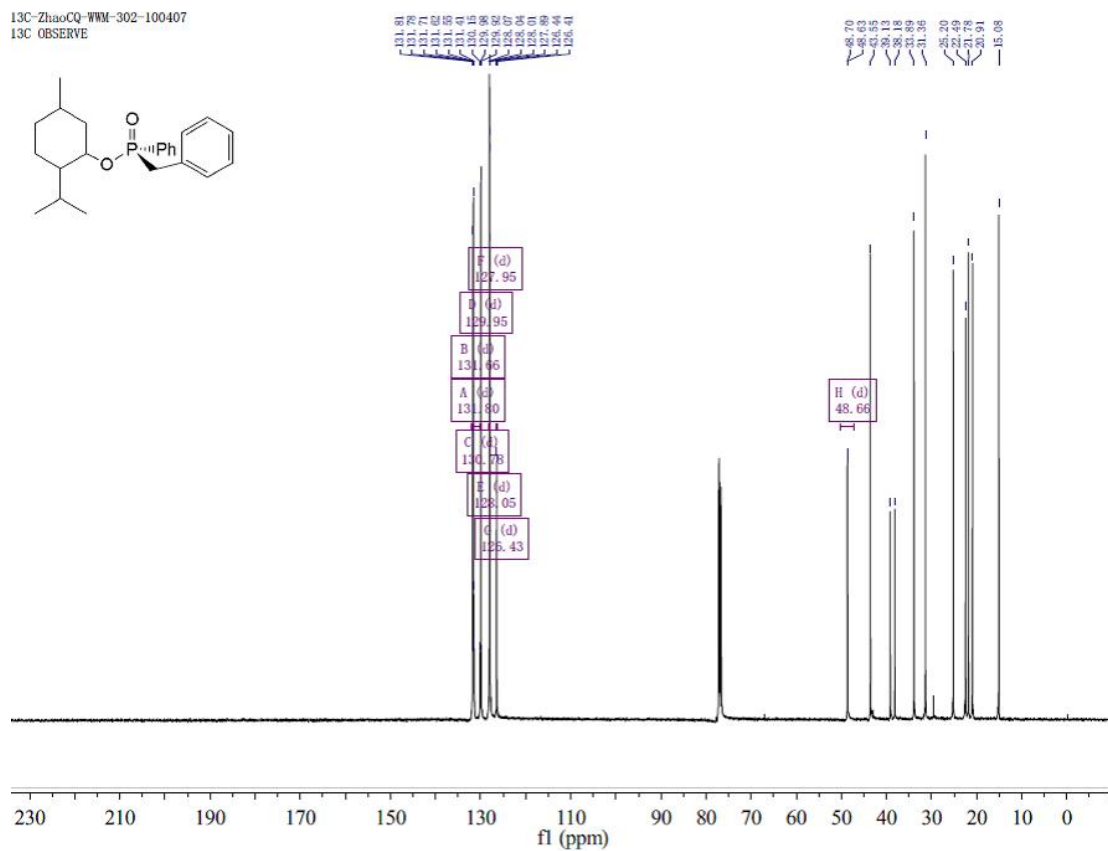
(S_P)-O-Menthyl benzylphenylphosphinate (12h)

31P-ZhaoCQ-WWM-302-100405
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION



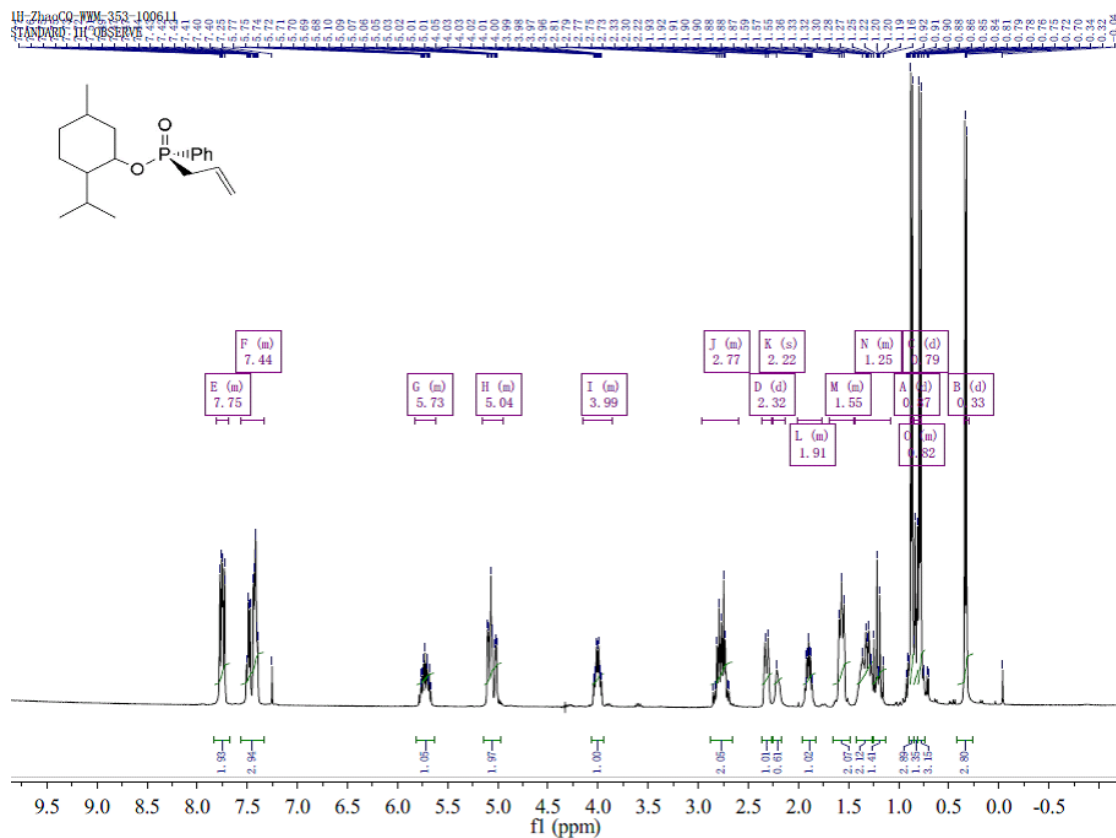
(*S_P*)-*O*-Menthyl benzylphenylphosphinate (12h)

13C-ZhaoCQ-WM-302-100407
13C OBSERVE



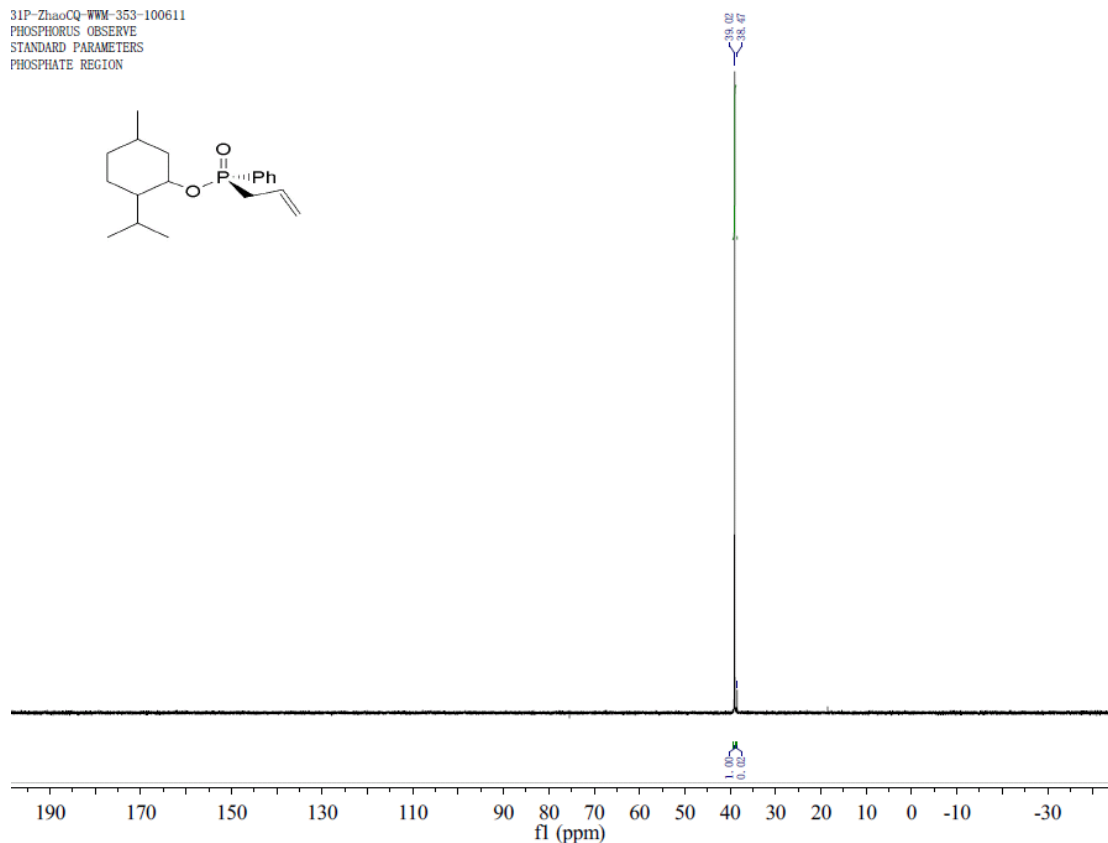
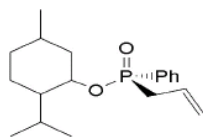
(*S_P*)-*O*-Menthyl allylphenylphosphinate (12i)

1H-ZhaoCQ-WM-353-100611
STANDARD 1H OBSERVE



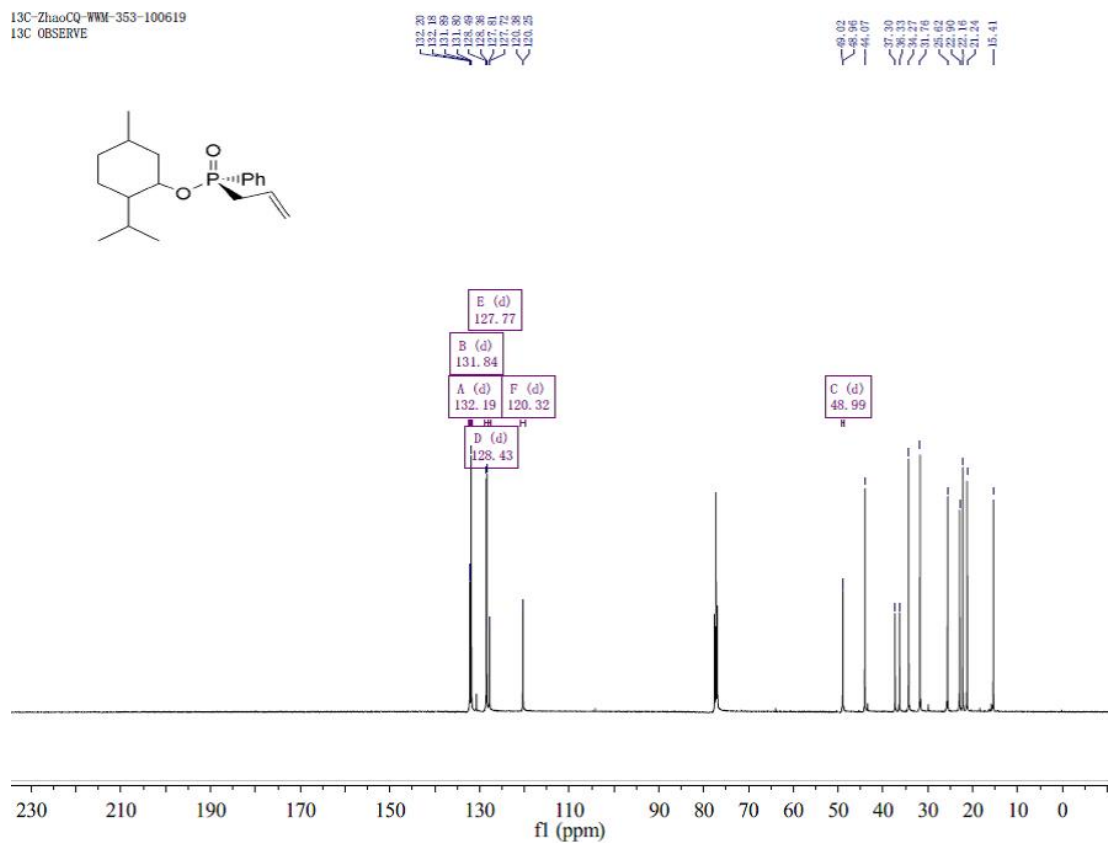
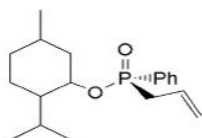
(*S_P*)-*O*-Menthyl allylphenylphosphinate (12i)

31P-ZhaoCQ-WWM-353-100611
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

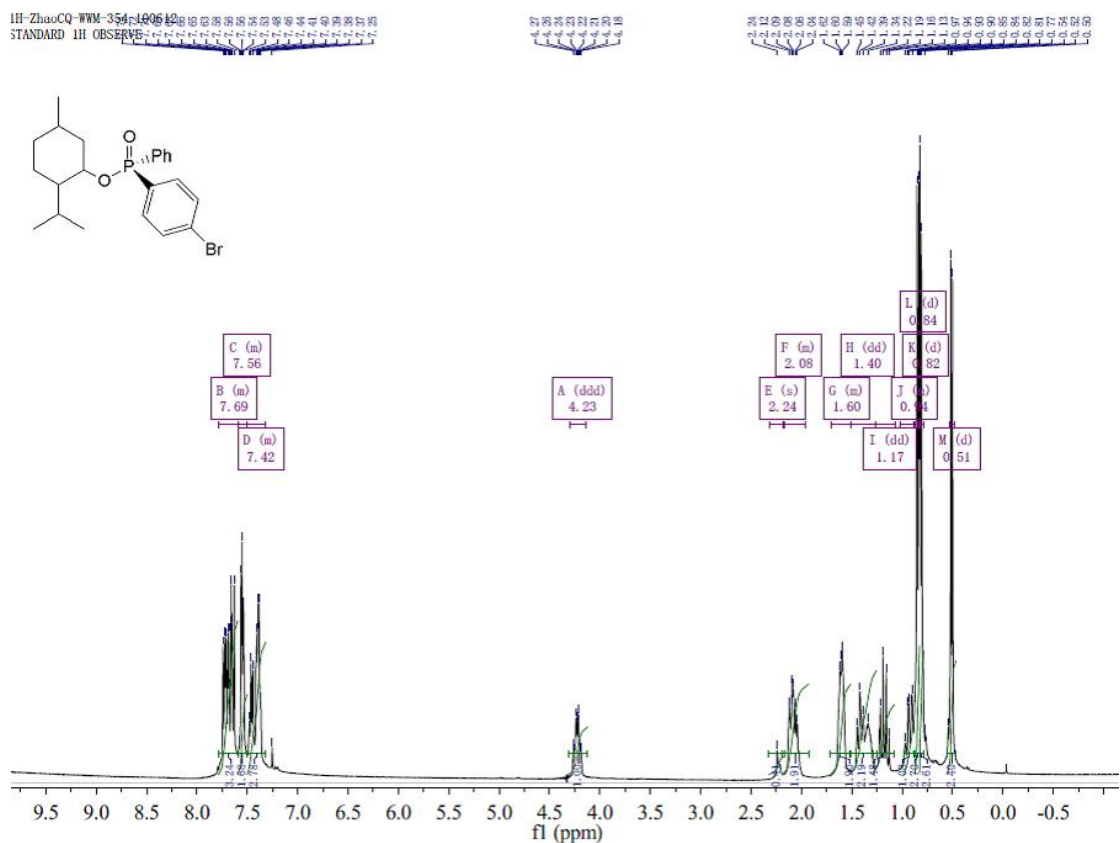


(*S_P*)-*O*-Menthyl allylphenylphosphinate (12i)

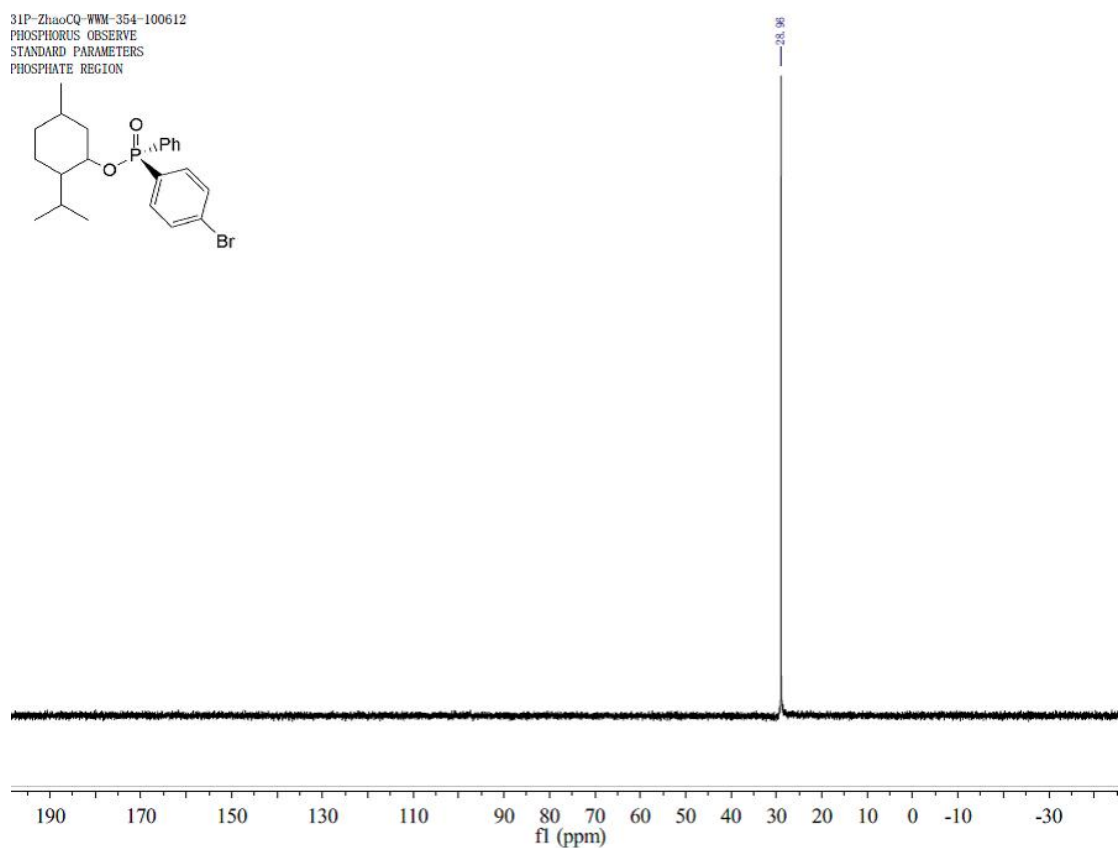
13C-ZhaoCQ-WWM-353-100619
13C OBSERVE



(*R_P*)-*O*-Menthyl *p*-bromophenylphenylphosphinate (12j)

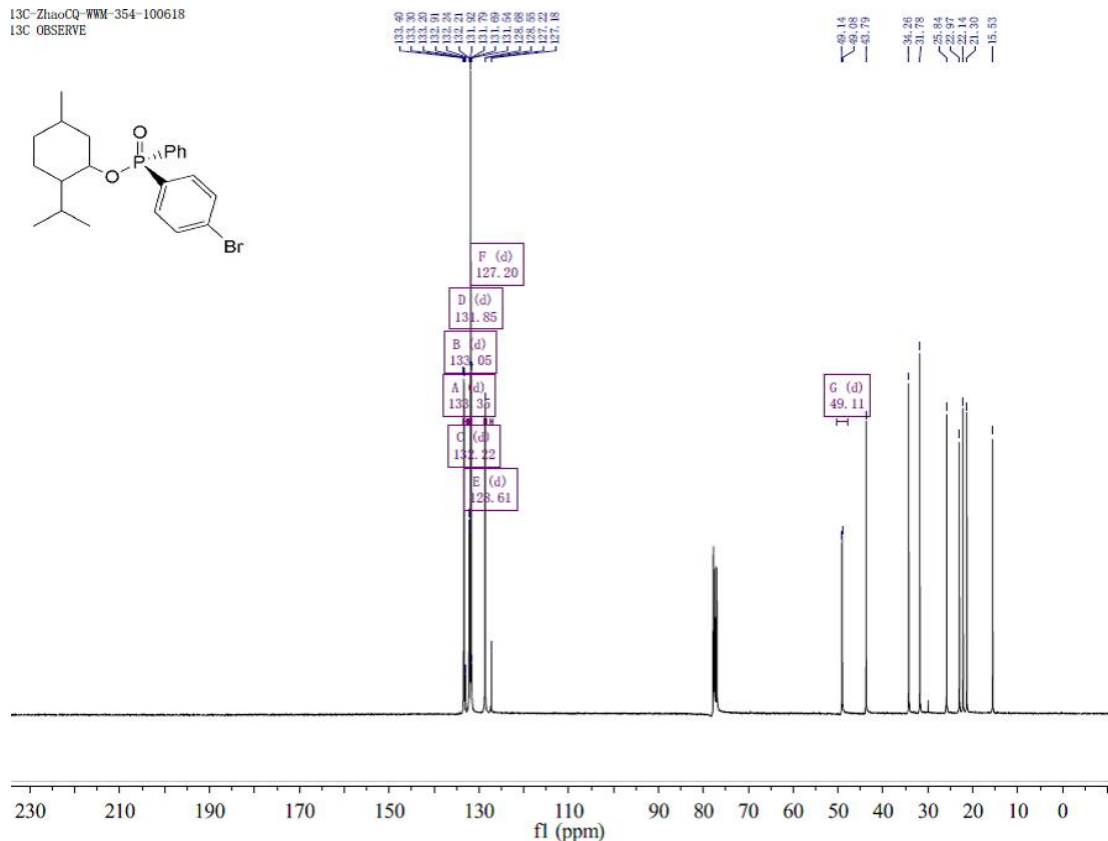


(*R_P*)-*O*-Menthyl *p*-bromophenylphenylphosphinate (12j)



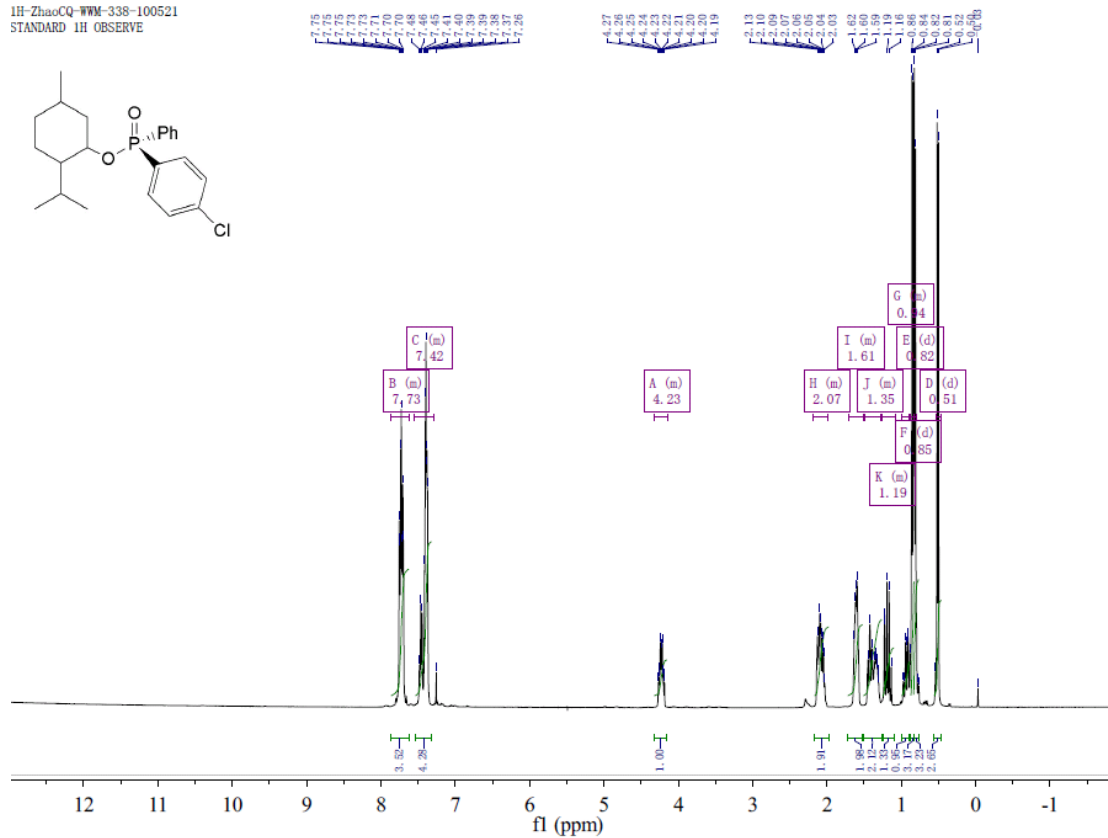
(*R*_P)-*O*-Menthyl *p*-bromophenylphenylphosphinate (12j)

13C-ZhaoCQ-WWM-354-100618
13C OBSERVE

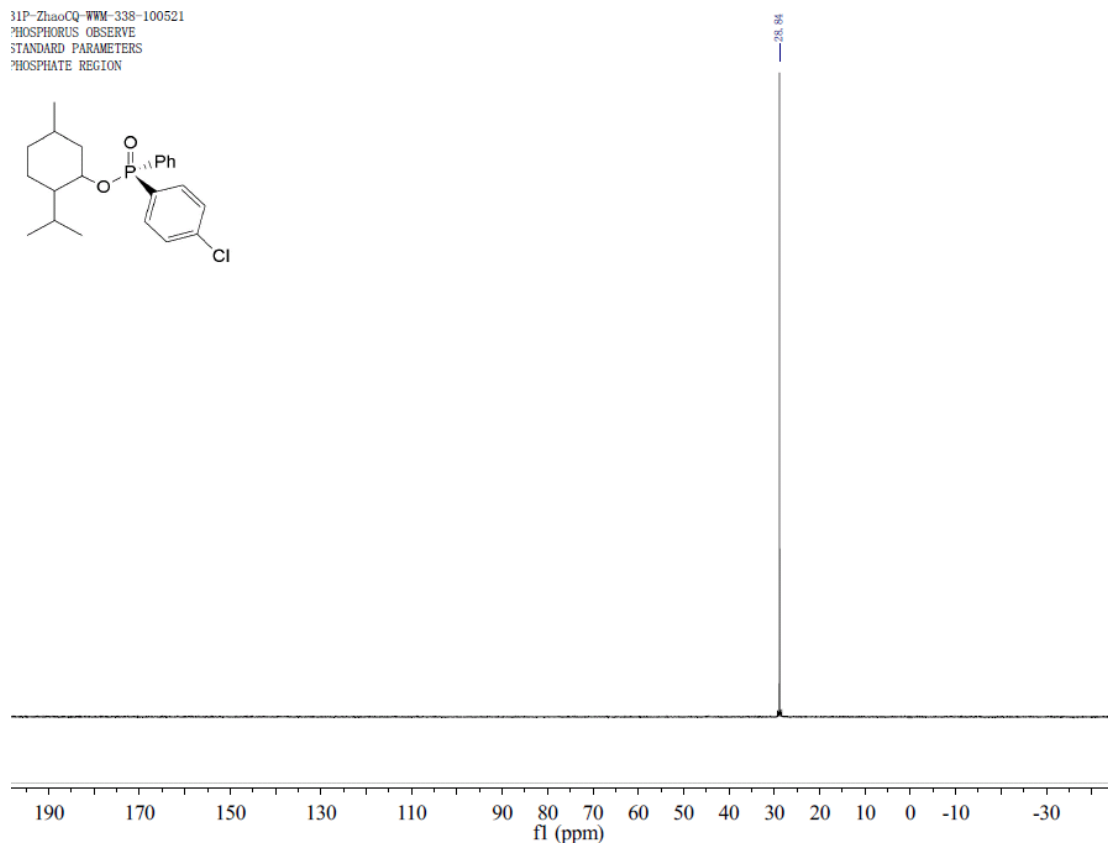
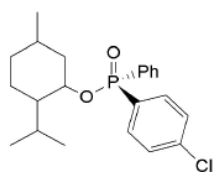


(*R*_P)-*O*-Menthyl *p*-chlorophenylphenylphosphinate (12k)

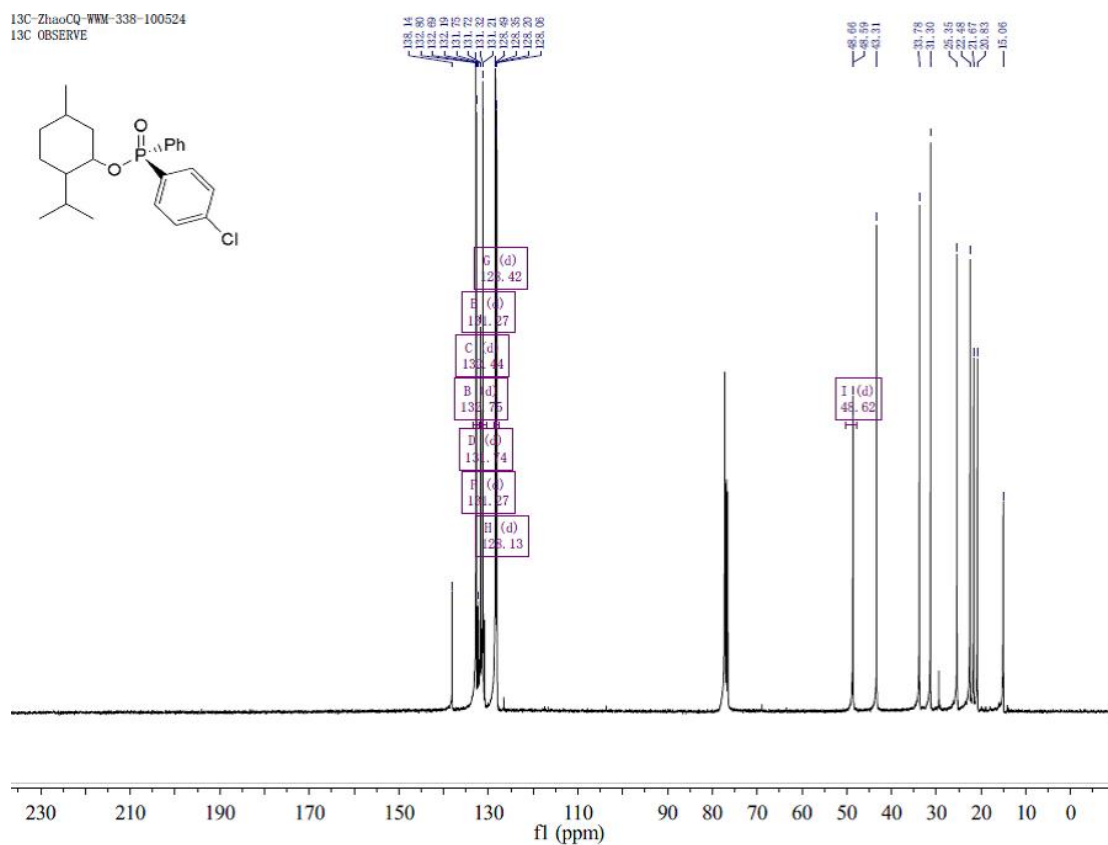
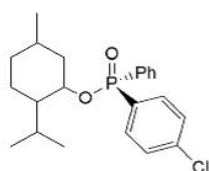
1H-ZhaoCQ-WWM-338-100521
STANDARD 1H OBSERVE



31P-ZhaoCQ-WM-338-100521
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

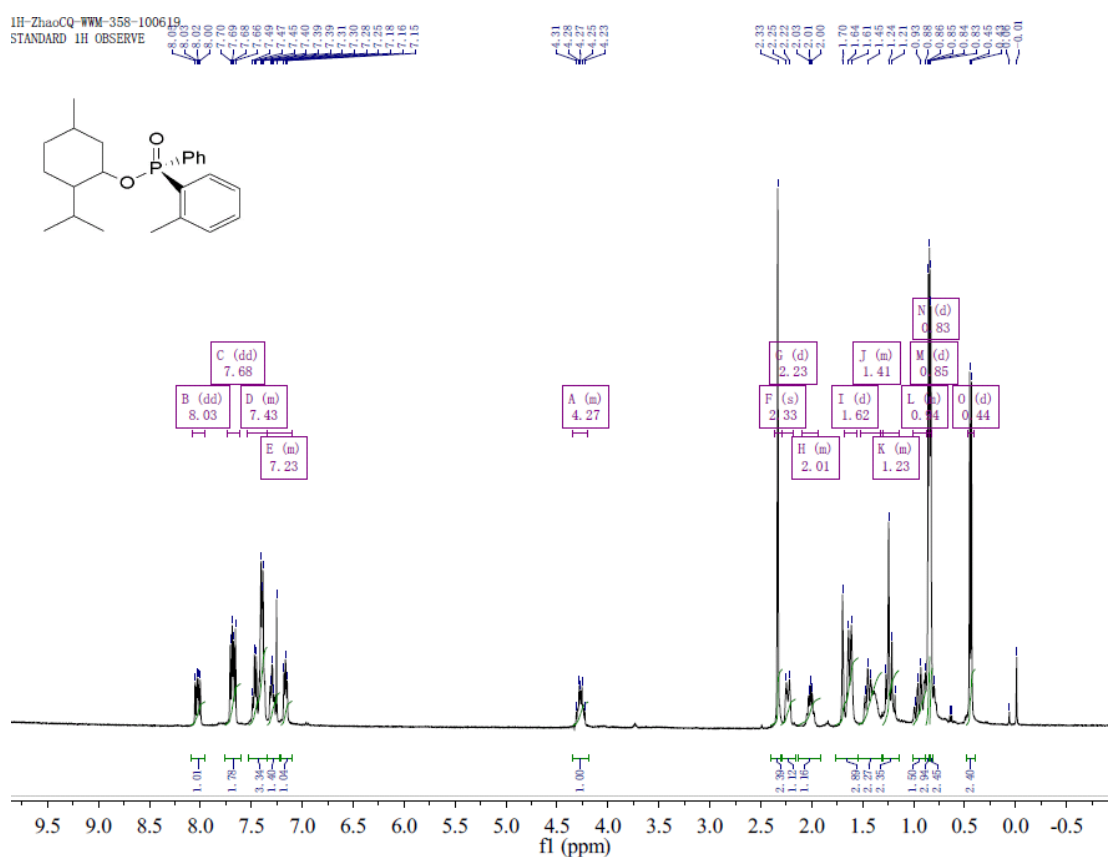


13C-ZhaoCQ-WWM-338-100524
13C OBSERVE



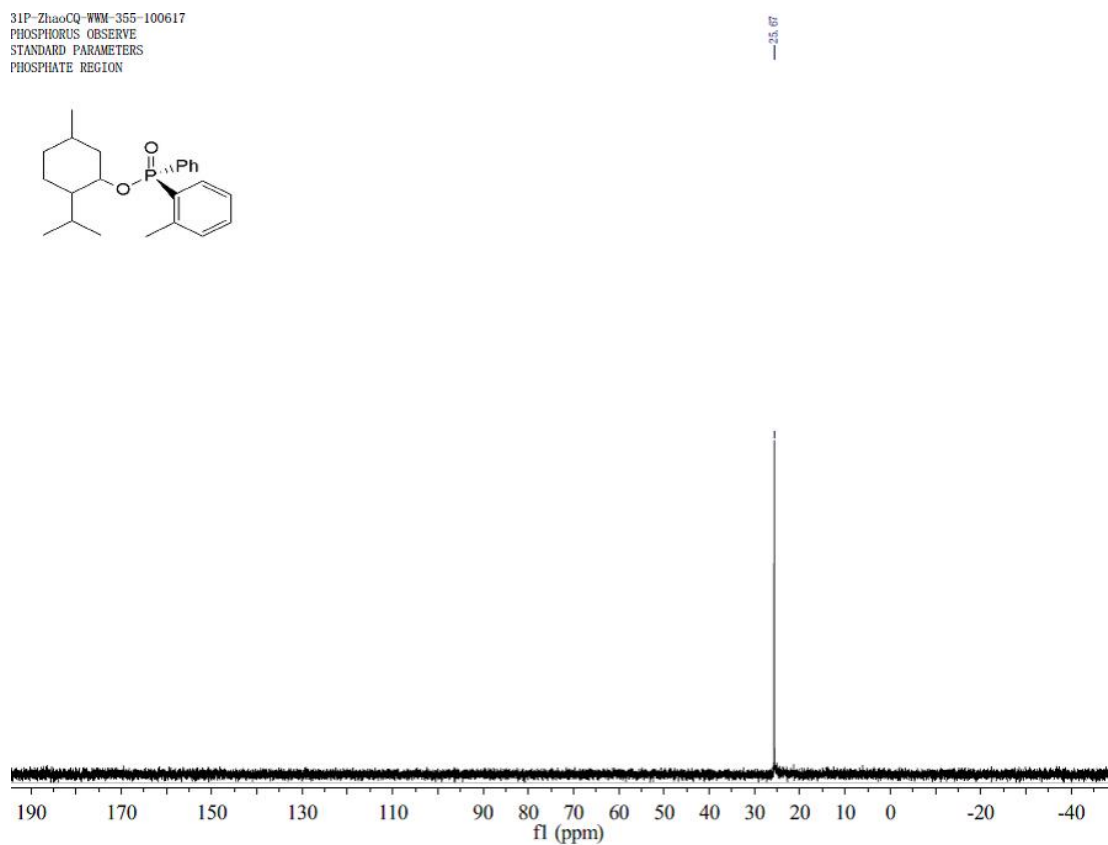
(*R_P*)-*O*-Menthyl *o*-methylphenylphenylphosphinate (12l)

1H-ZhaoCQ-WM-358-100619
STANDARD 1H OBSERVE

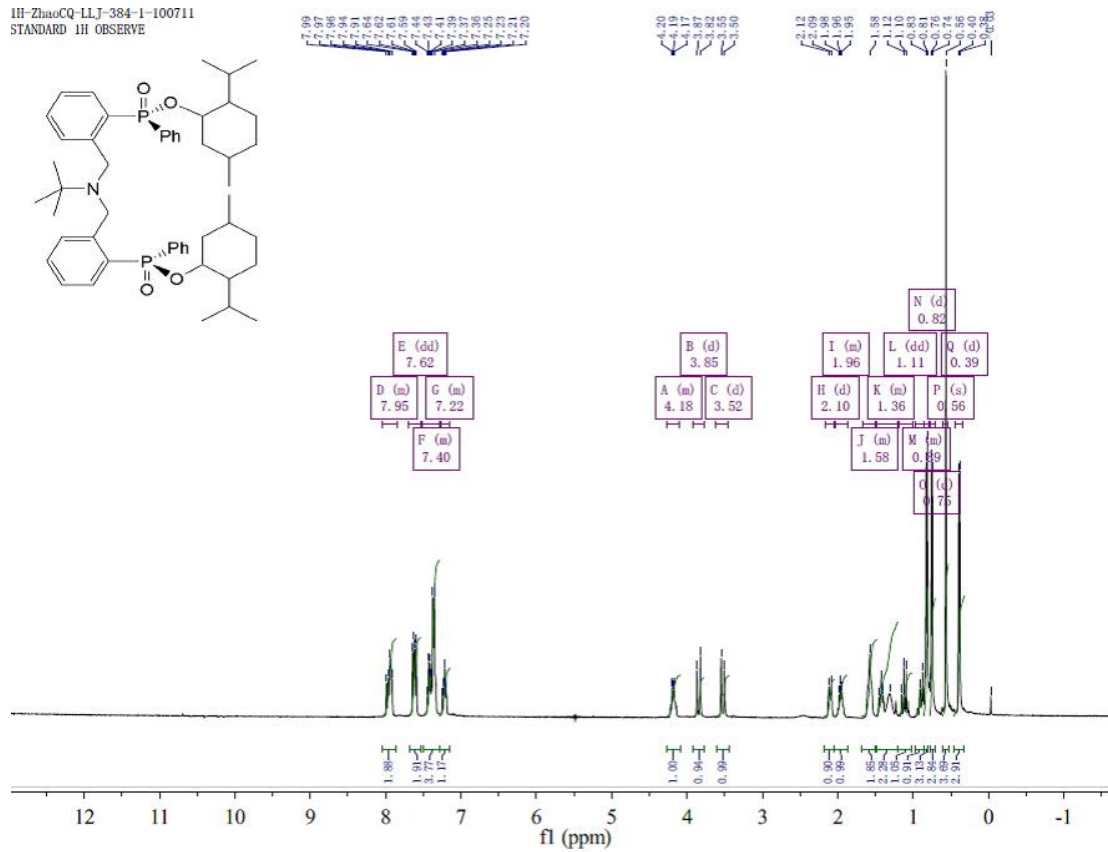


(*R_P*)-*O*-Menthyl *o*-methylphenylphenylphosphinate (12l)

31P-ZhaoCQ-WM-355-100617
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

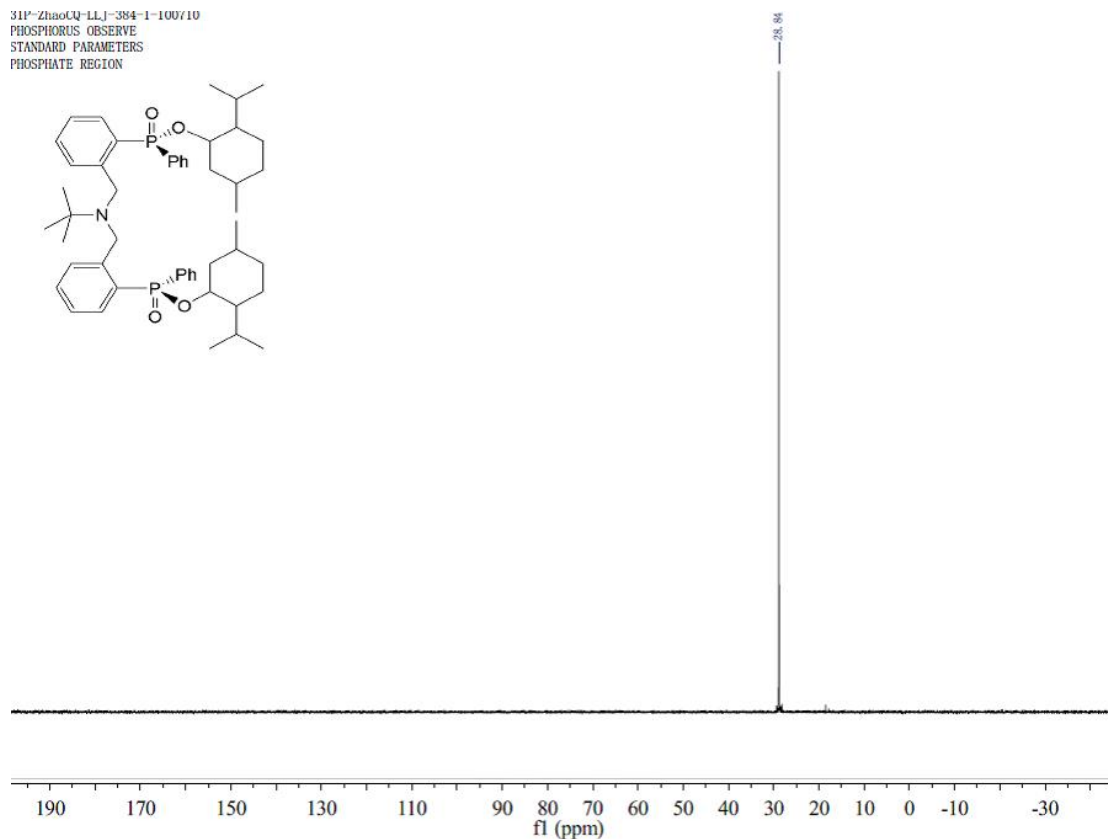


13C-ZhaoCQ-WWM-358-100621
13C OBSERVE

1H-ZhaoCQ-LLJ-384-1-100711
STANDARD 1H OBSERVE

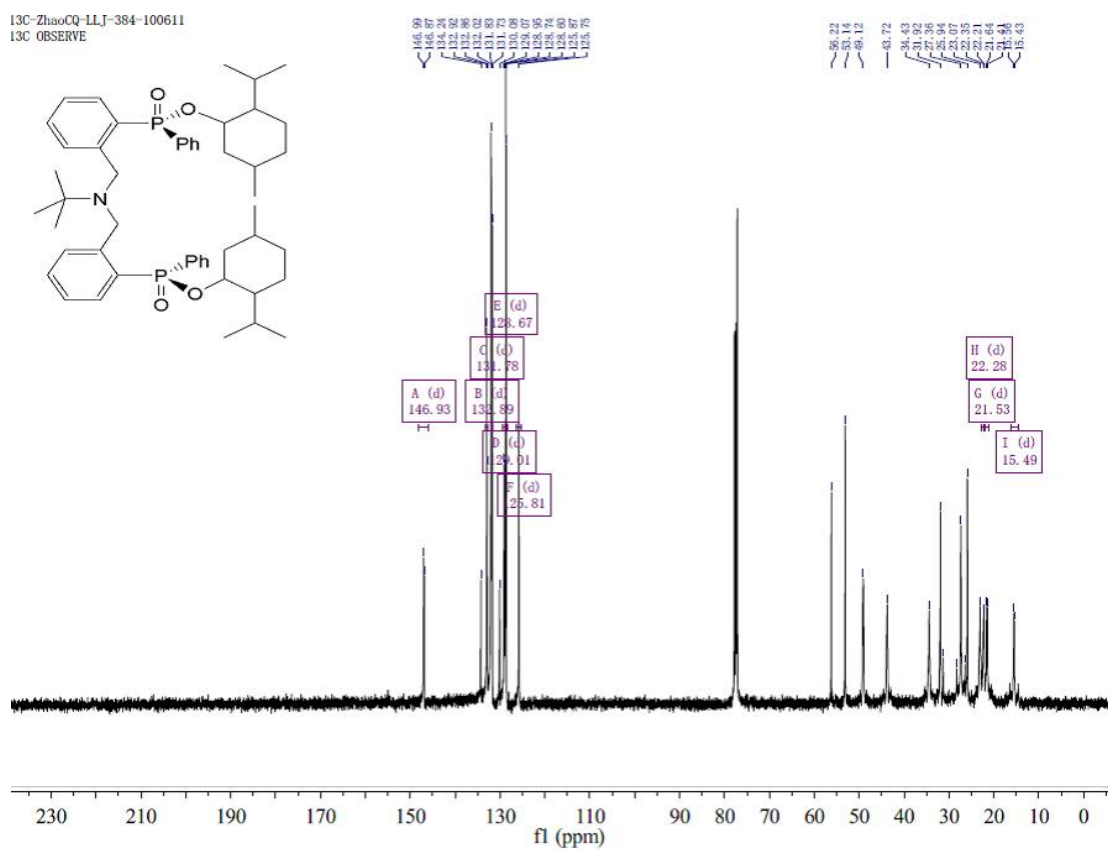
Bis-phenylphosphinate 12m

31P-ZhaoCQ-LLJ-384-1-100710
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION



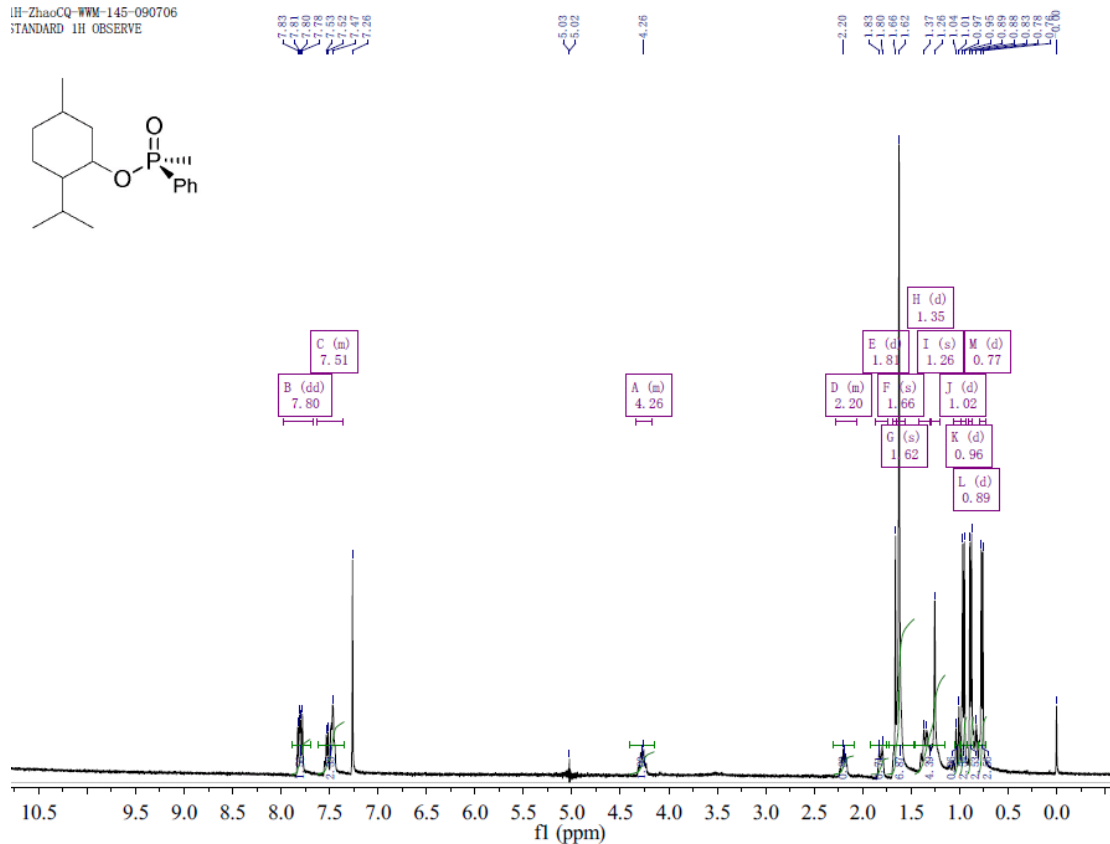
Bis-phenylphosphinate 12m

13C-ZhaoCQ-LLJ-384-100611
13C OBSERVE



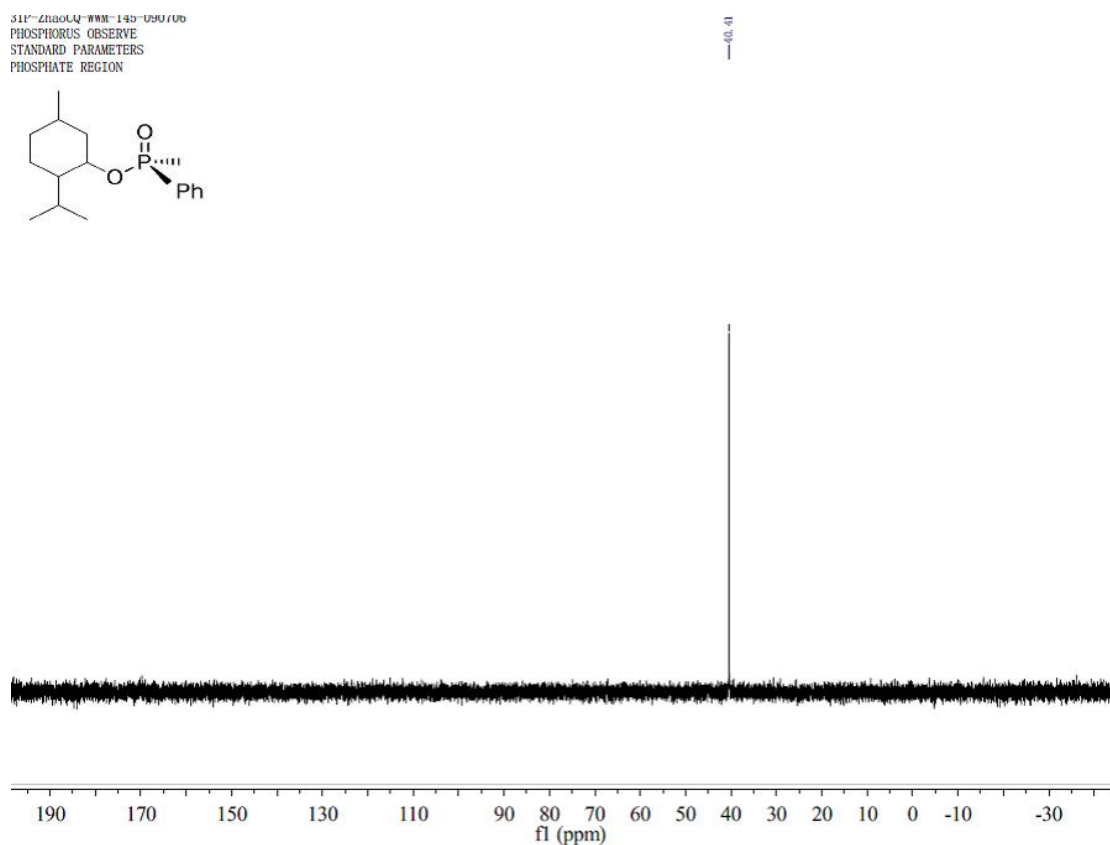
(*R*_P)-*O*-Menthyl methylphenylphosphinate (12a')

1H-ZhaoCQ-WMM-145-090706
STANDARD 1H OBSERVE



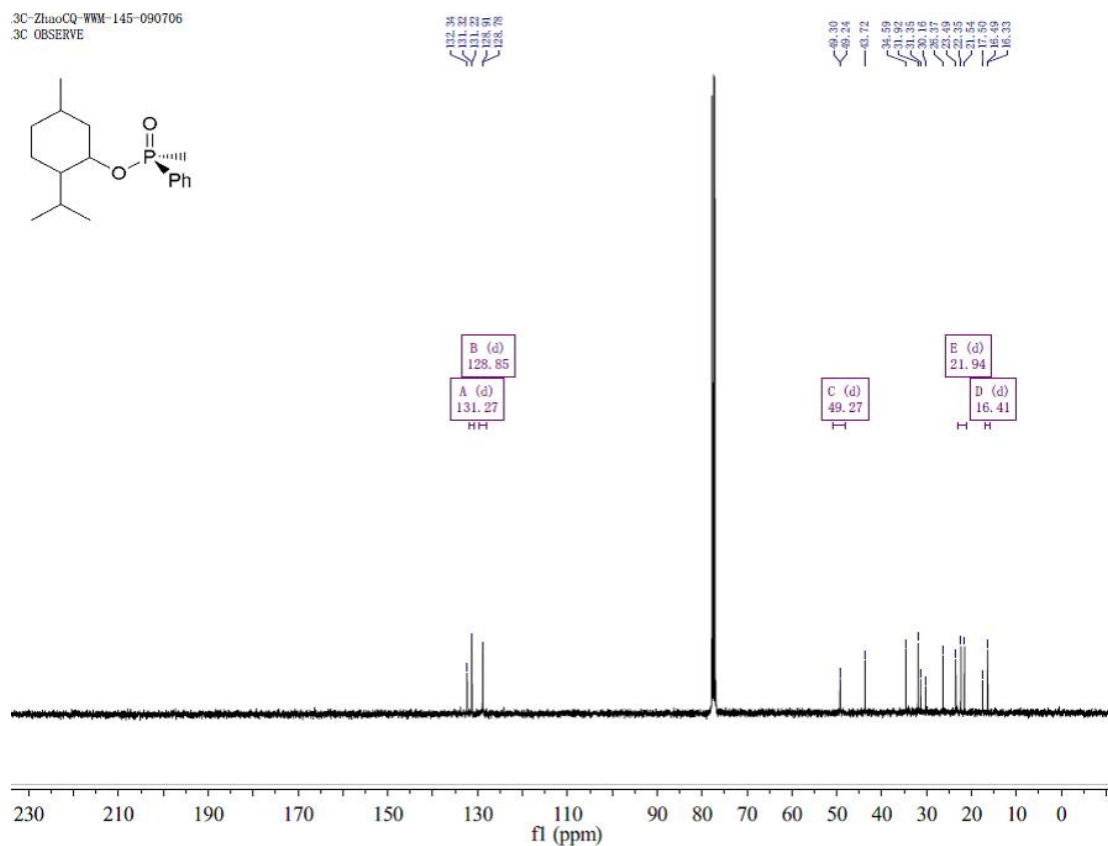
(*R*_P)-*O*-Menthyl methylphenylphosphinate (12a')

31P-ZhaoCQ-WMM-145-090706
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION



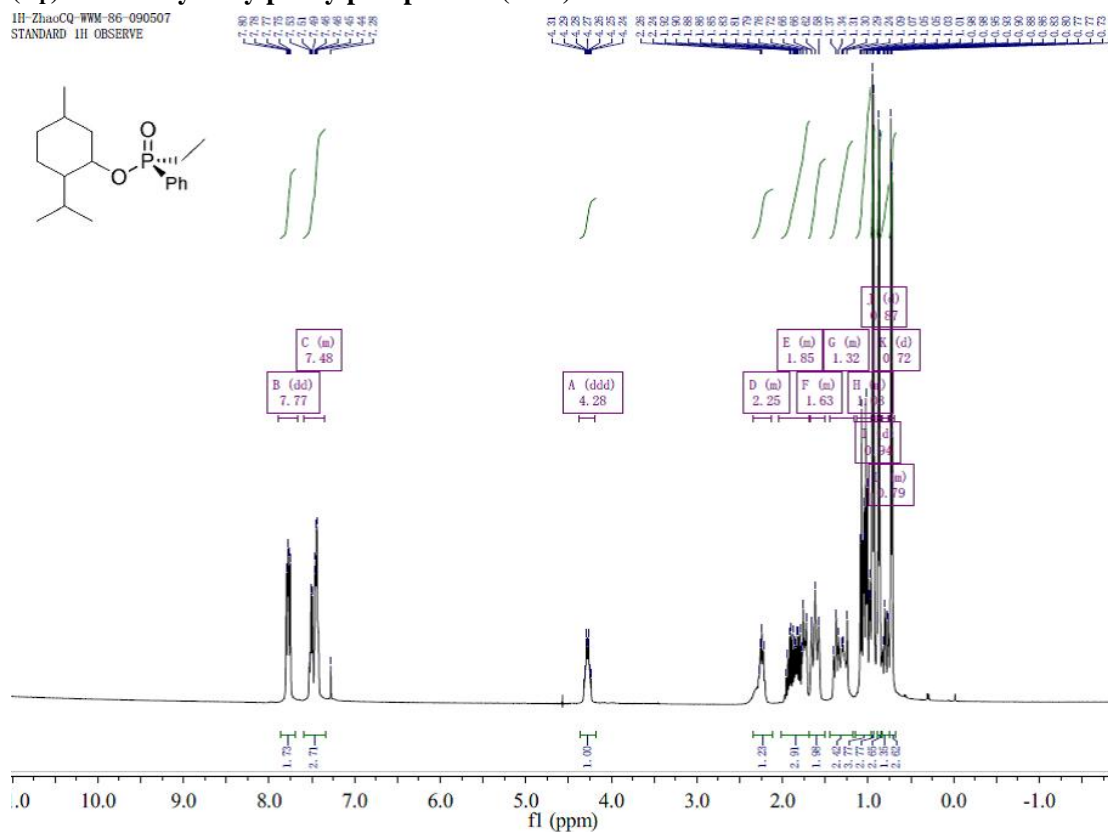
(*R*_P)-*O*-Menthyl methylphenylphosphinate (12a')

3C-ZhaoCQ-WM-145-090706
3C OBSERVE



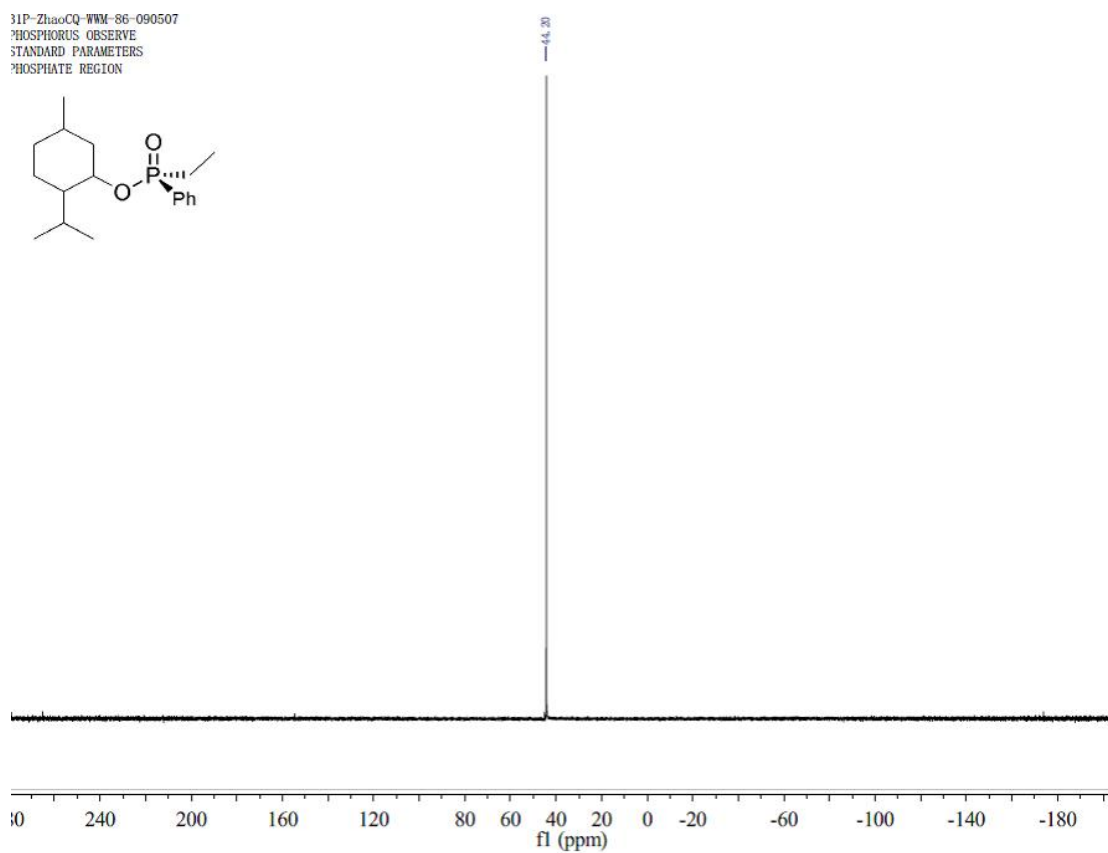
(*R*_P)-*O*-Menthyl ethylphenylphosphinate (12b')

1H-ZhaoCQ-WM-86-090507
STANDARD 1H OBSERVE



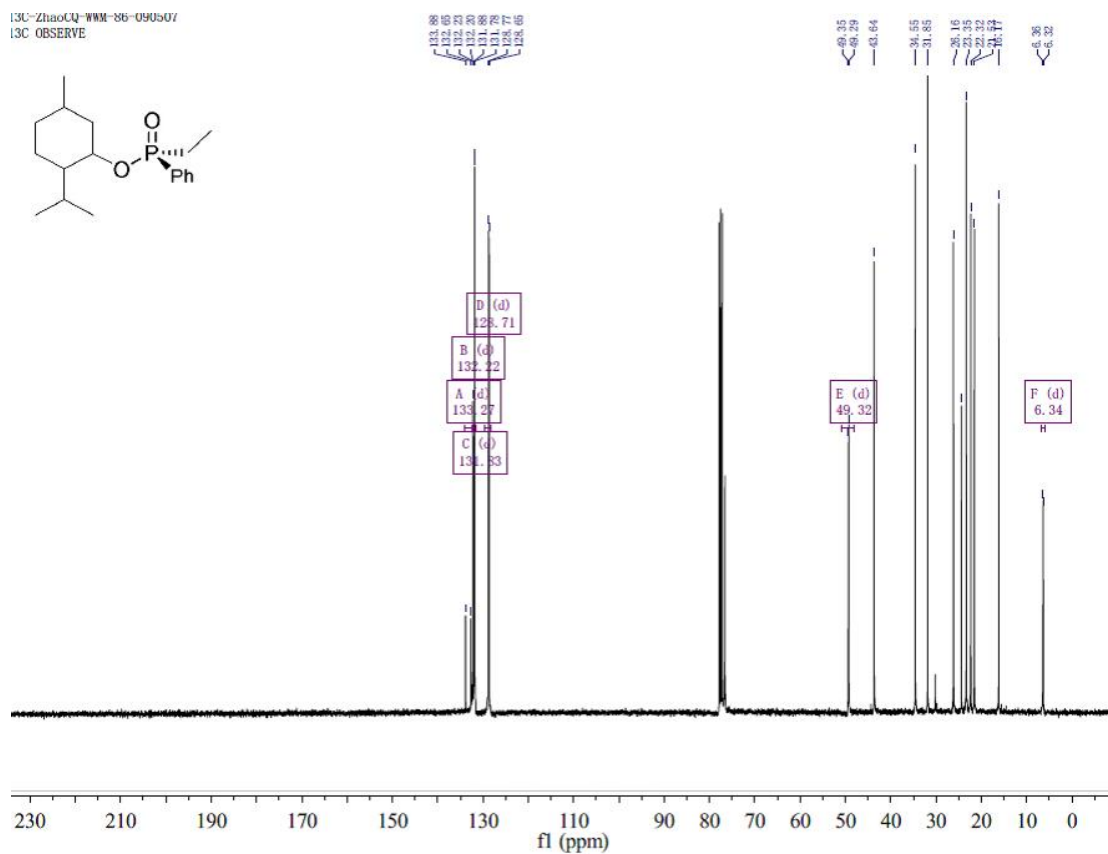
(*R*_P)-*O*-Menthyl ethylphenylphosphinate (12b')

31P-ZhaoCQ-WMM-86-090507
 PHOSPHORUS OBSERVE
 STANDARD PARAMETERS
 PHOSPHATE REGION



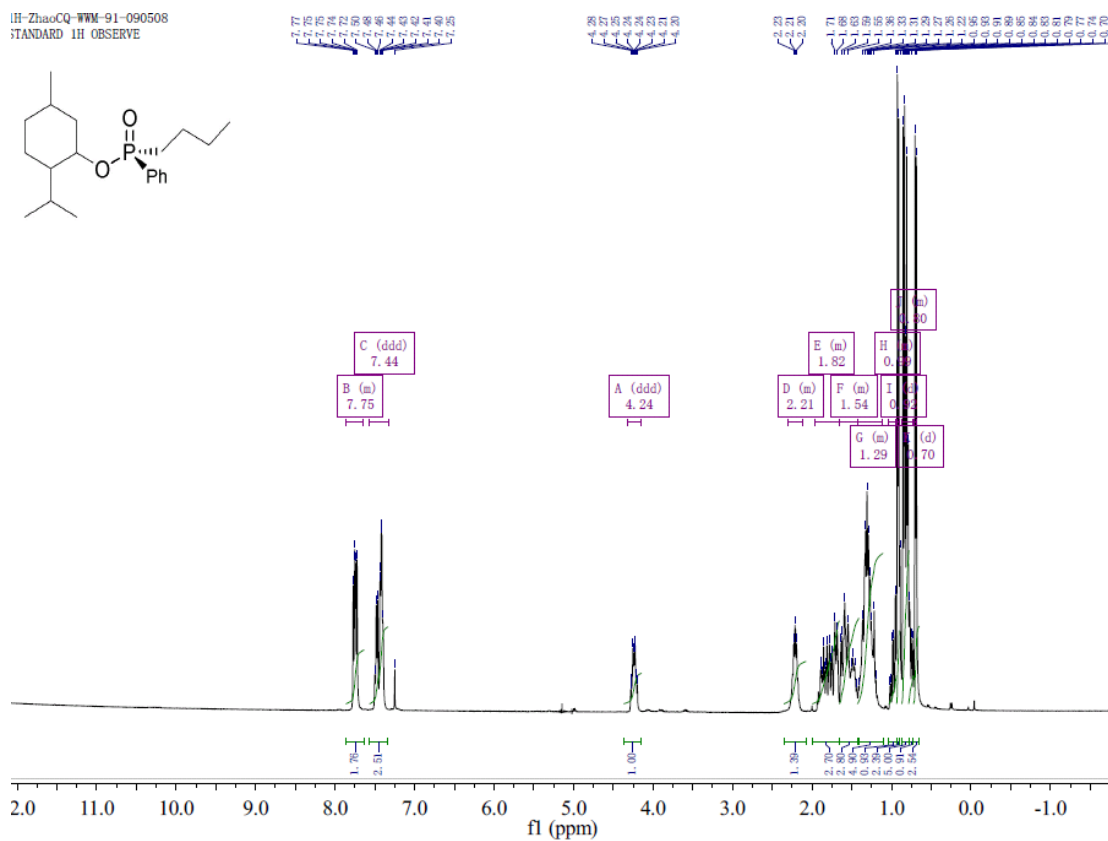
(*R*_P)-*O*-Menthyl ethylphenylphosphinate (12b')

13C-ZhaoCQ-WMM-86-090507
 13C OBSERVE



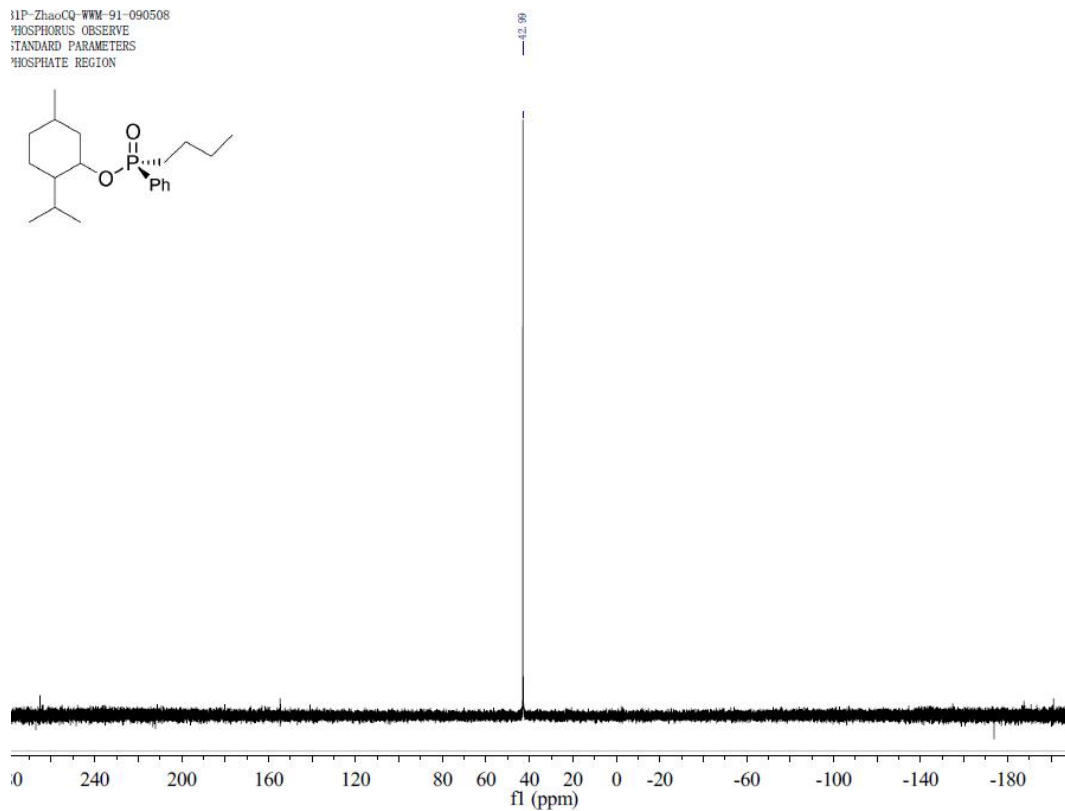
(*R*_P)-*O*-Menthyl *n*-butylphenylphosphinate (12c')

1H-ZhaoCQ-WM-91-090508
STANDARD 1H OBSERVE

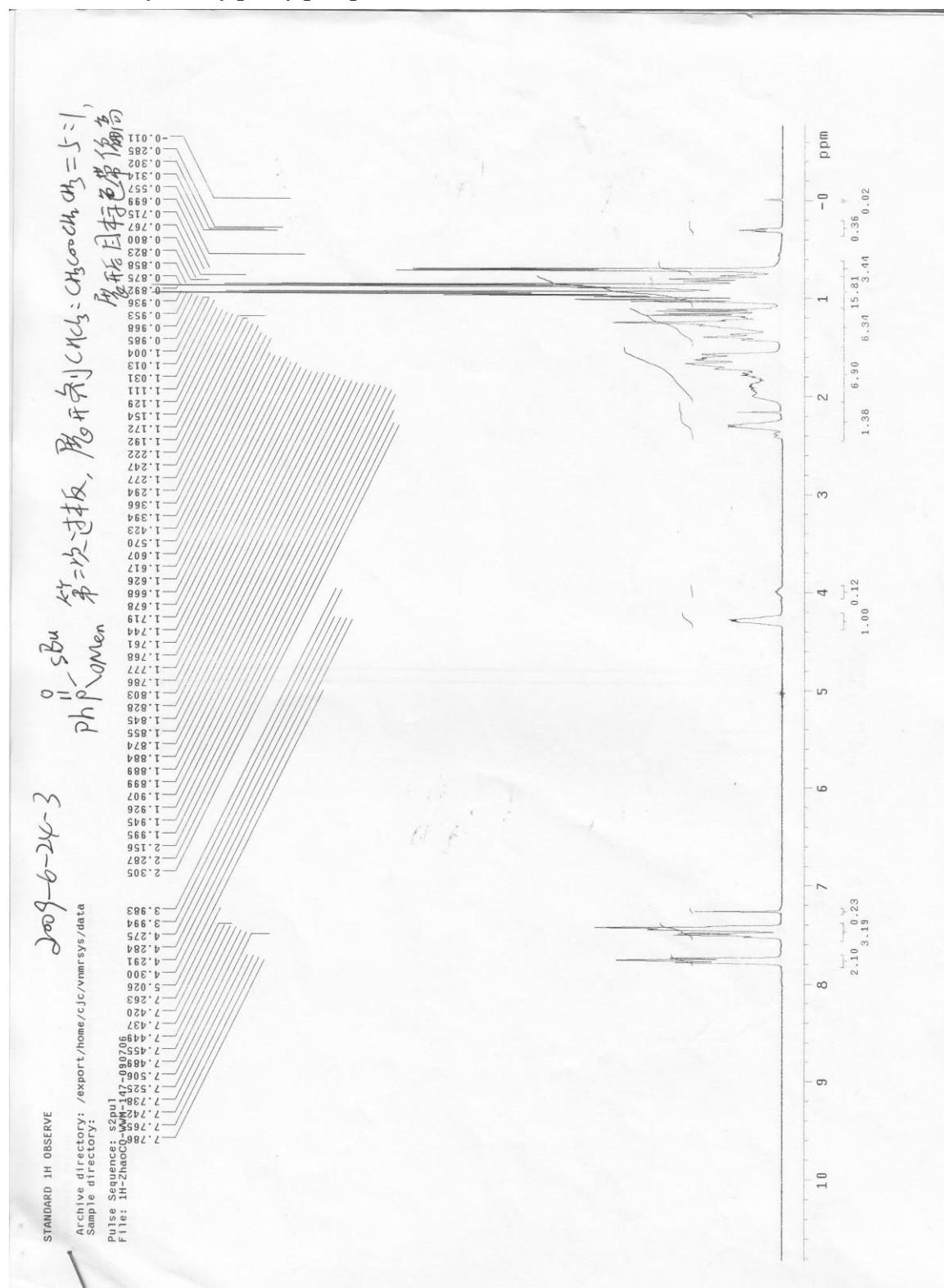


(*R*_P)-*O*-Menthyl *n*-butylphenylphosphinate (12c')

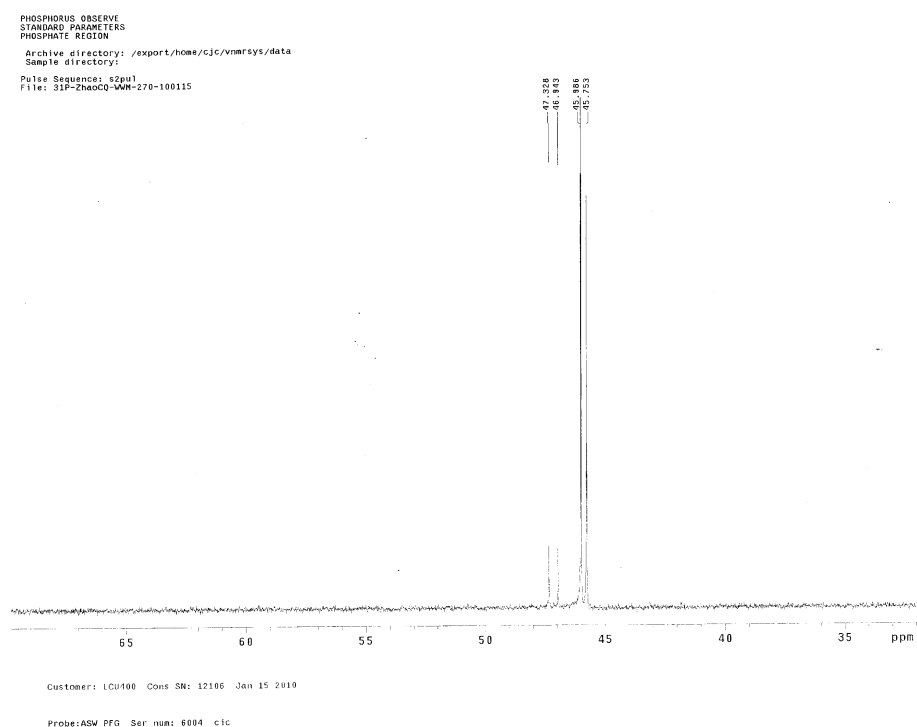
1P-ZhaoCQ-WM-91-090508
PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION



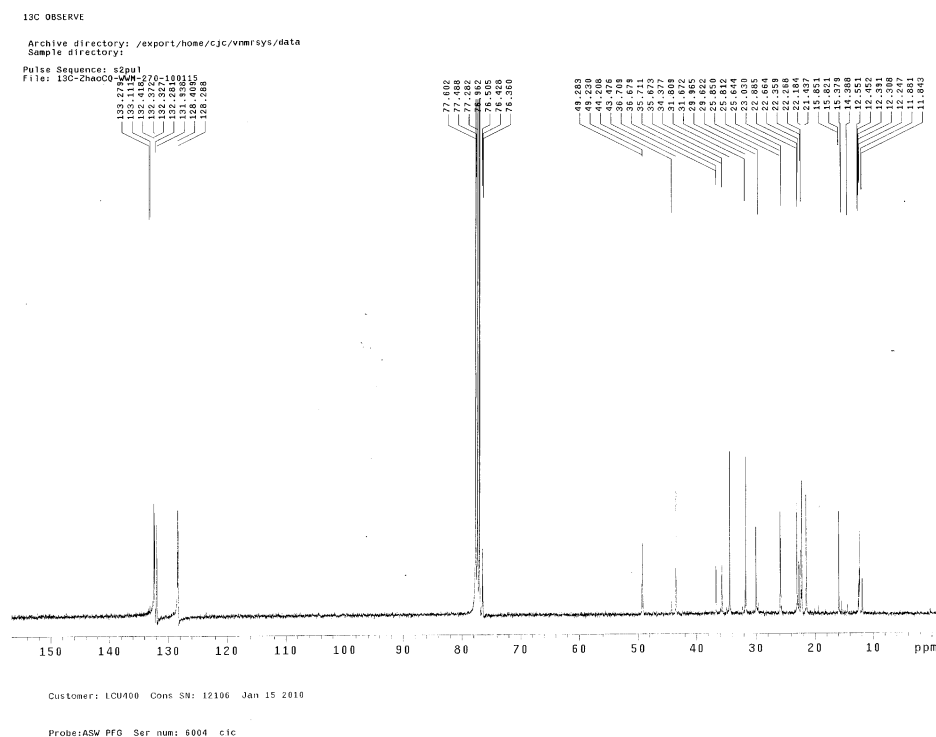
(R_P)-O-Menthyl *s*-butylphenylphosphinate (12f')



(*R*_P)-*O*-Menthyl *s*-butylphenylphosphinate (12f')



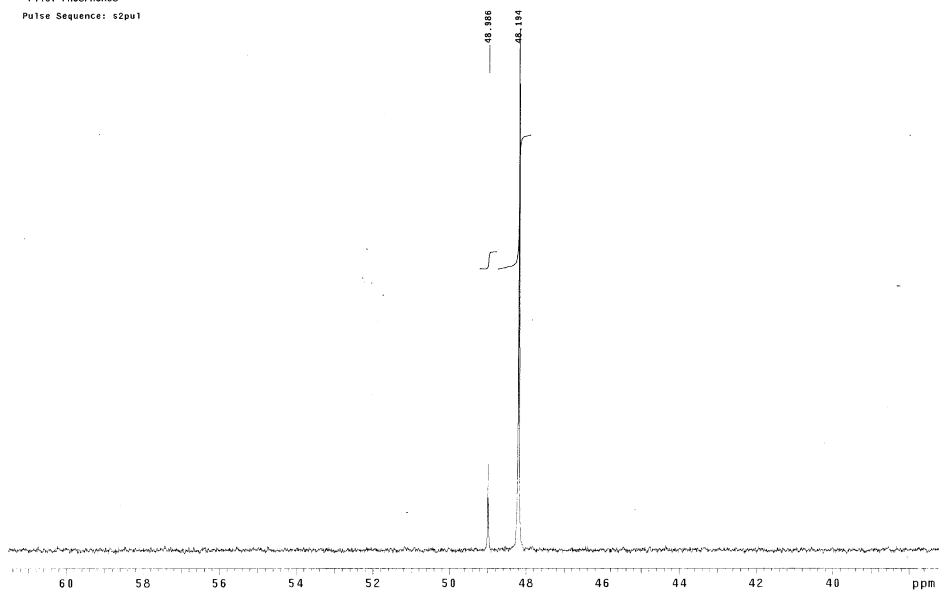
(*R*_P)-*O*-Menthyl *s*-butylphenylphosphinate (12f')



[illegible]

(S_P)-O-Menthyl *t*-butylphenylphosphinate (12g')

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION
Archive directory: /export/home/cjc/vmrsys/data
Sample directory:
File: PHOSPHORUS
Pulse Sequence: s2pu1



Customer: LCU400 Cons SN: 12106 Jul 7 2009

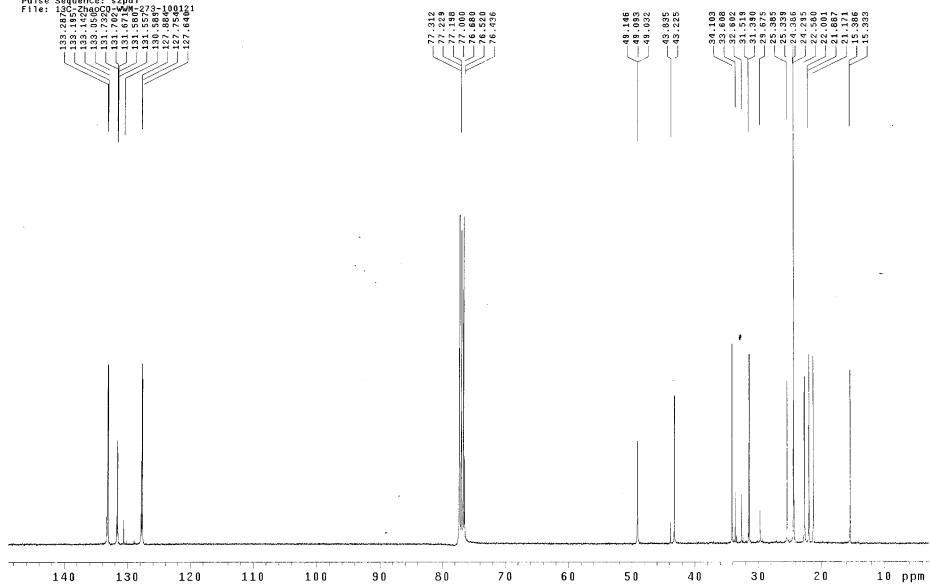
Probe:ASW PFG Ser num: 6004 c1c

(S_P)-O-Menthyl *t*-butylphenylphosphinate (12g')

13C OBSERVE

Archive directory: /export/home/cjc/vmrsys/data
Sample directory:

Pulse Sequence: s2pu1
File: 13C-2HUSO-WM-273-108121



2008-7-15-2

STANDARD 1H OBSERVE

Archive directory: /export/home/cjc/vnmrsys/data
Sample directory:

Pulse Sequence: szpul
File: 1h-20080715-168-030306

Ph⁰CH₂Ph
PhP₂OMe

二次重结晶, H.P. 基本干净

7.677
7.666
7.648
7.631
7.509
7.490
7.472
7.408
7.389
7.380
7.371
7.367
7.265
7.226
7.204
7.185
7.172
7.156
7.126
7.109
7.325
7.314
7.305
7.297
7.279
7.267
7.260
7.251
7.240
7.301
7.285
7.255
7.246
7.219
7.205
7.168
7.011
2.004
1.993
1.976
1.969
1.958
1.941
1.934
1.826
1.740
1.709
1.643
1.635
1.627
1.604
1.586
1.571
1.378
1.371
1.363
1.340
1.321
1.314
1.305
1.283
1.275
1.261
1.246
1.037
1.018
0.997
0.986
0.967
0.953
0.946
0.939
0.921
0.909
0.871
0.853
0.834
0.824
0.799
0.770
0.761
0.739
0.721
0.003

12 11 10 9 8 7 6 5 4 3 2 1 -1 -0

ppm

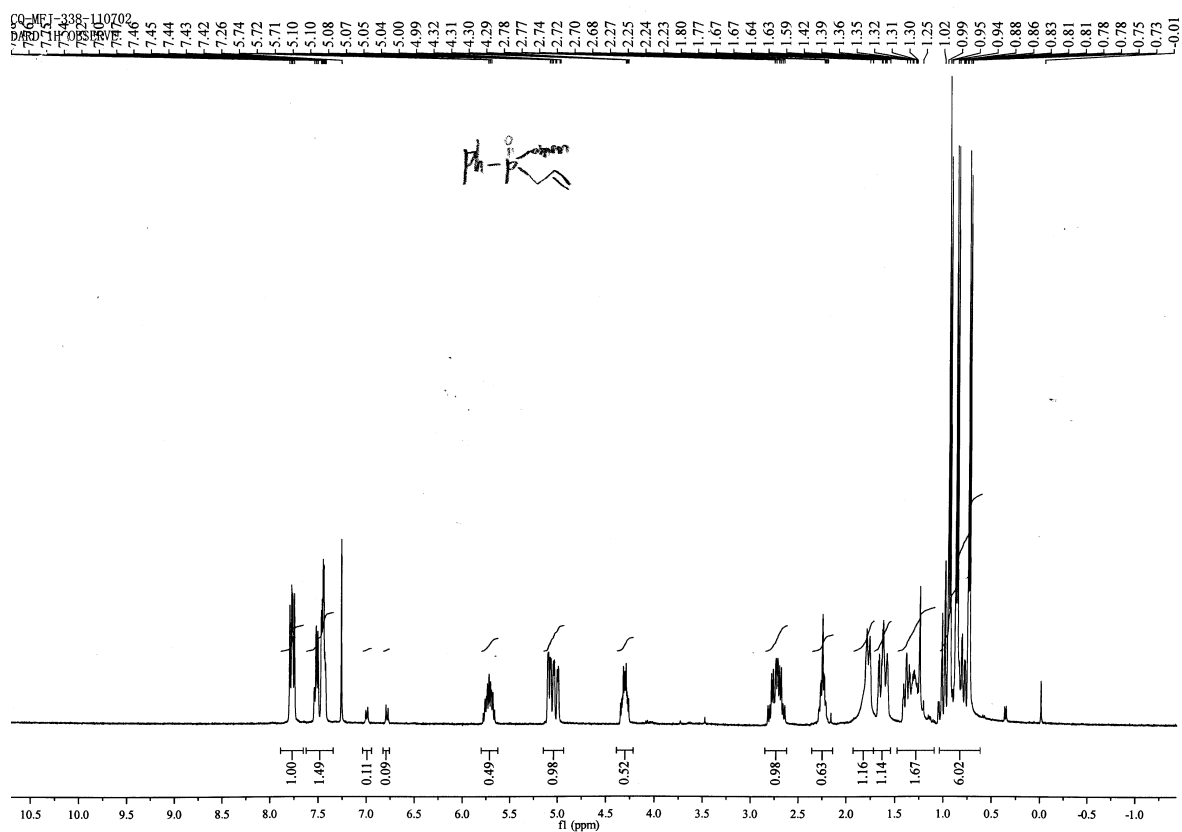
1.82, 2.184
0.38, 2.294

1.00

2.08

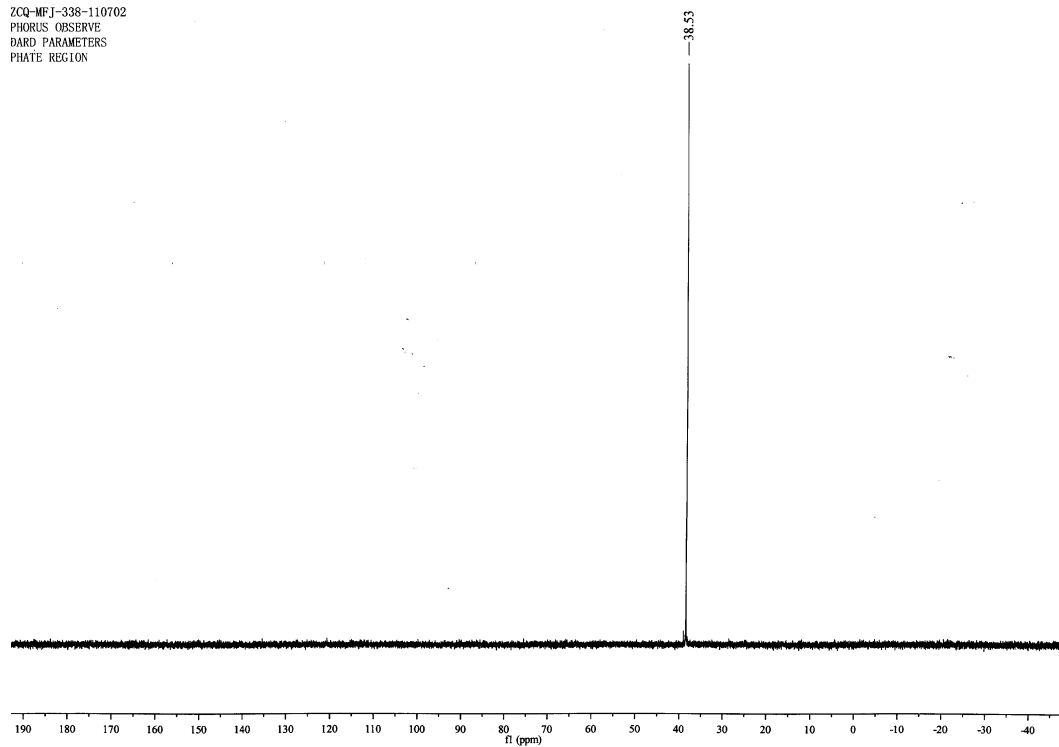
0.39, 2.15
4.61, 0.05
8.06

(*R*_P)-*O*-Menthyl allylphenylphosphinate (12i')



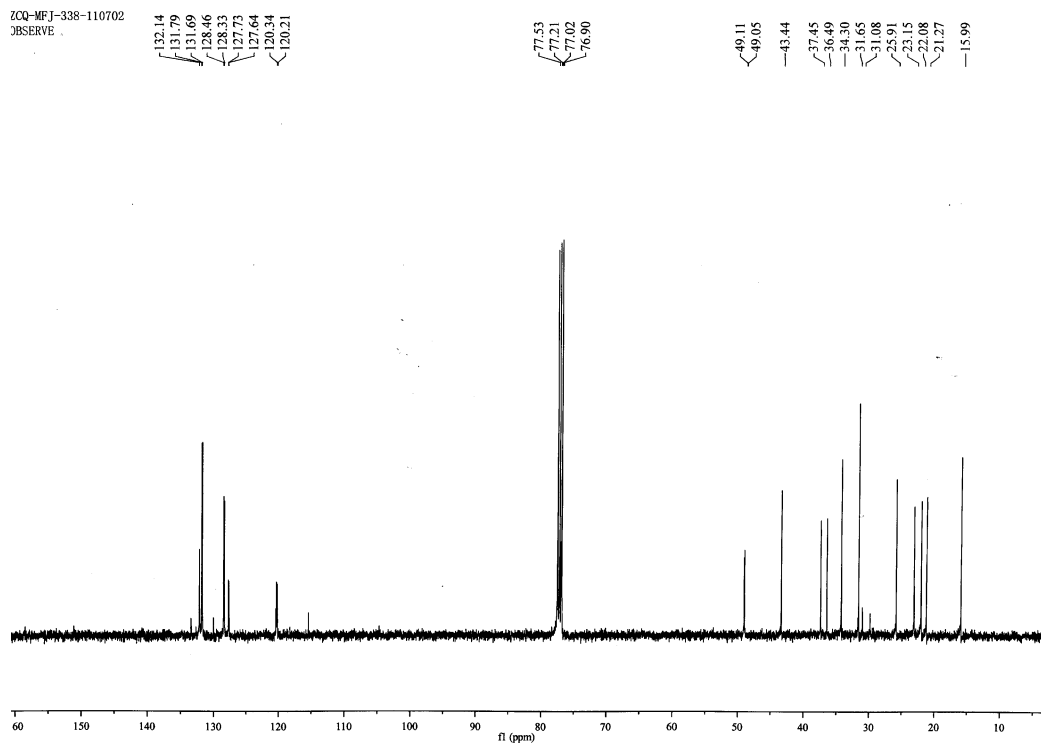
(*R*_P)-*O*-Menthyl allylphenylphosphinate (12i')

QC-MFJ-338-110702
PHORUS OBSERVE
HARD PARAMETERS
PHATE REGION

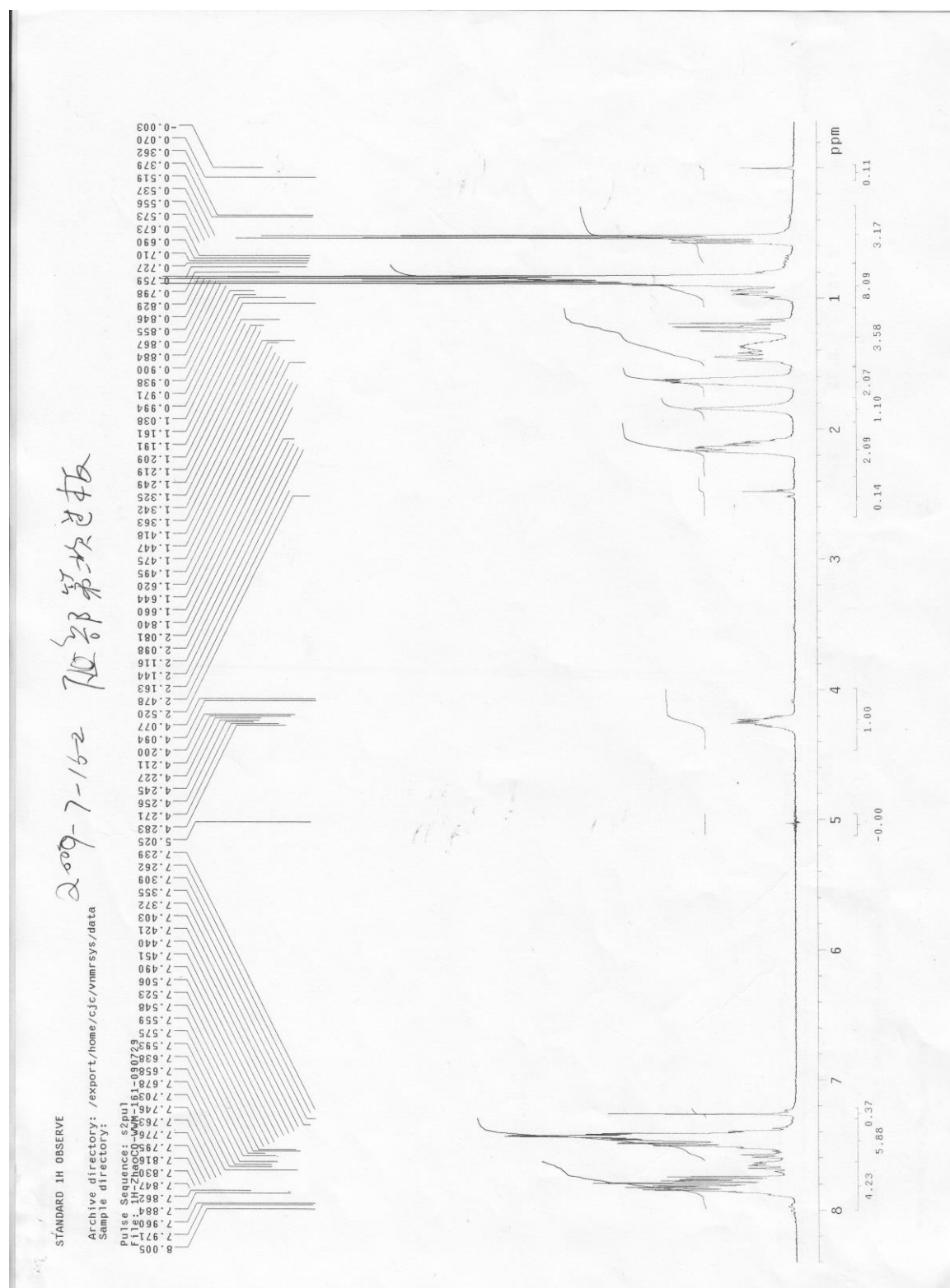


(*R*_P)-*O*-Menthyl allylphenylphosphinate (12i')

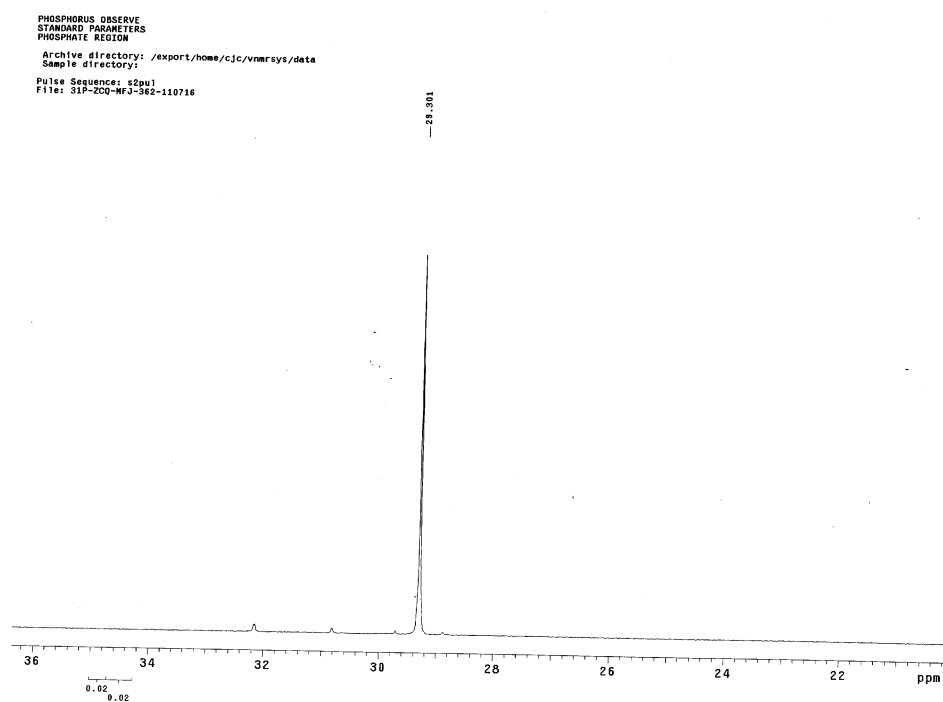
ZCQ-MFJ-338-110702
OBSERVE



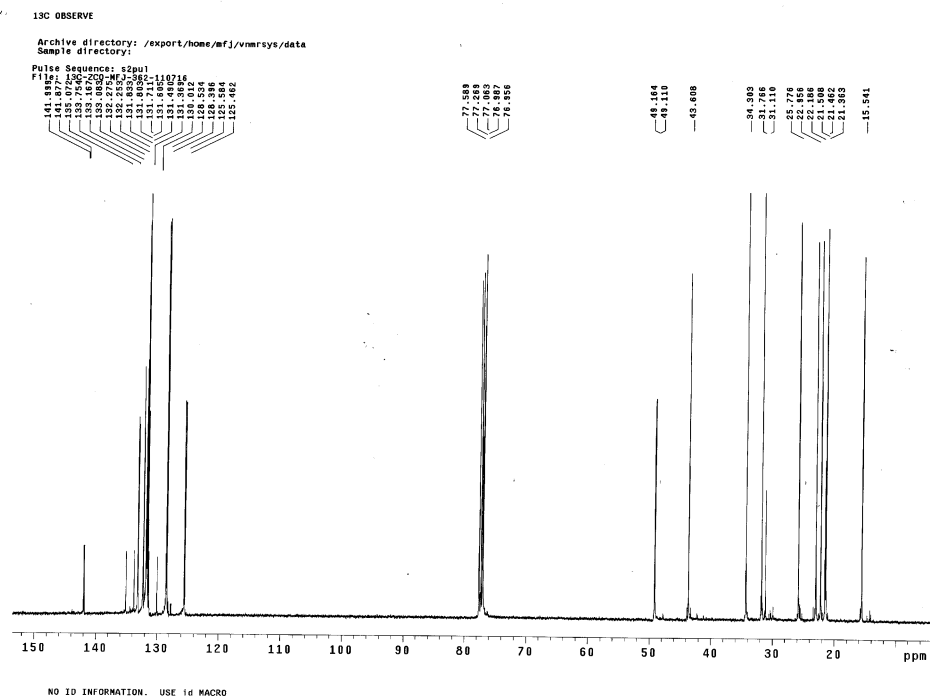
(S_P)-O-Menthyl *p*-bromophenylphenylphosphinate (12j')



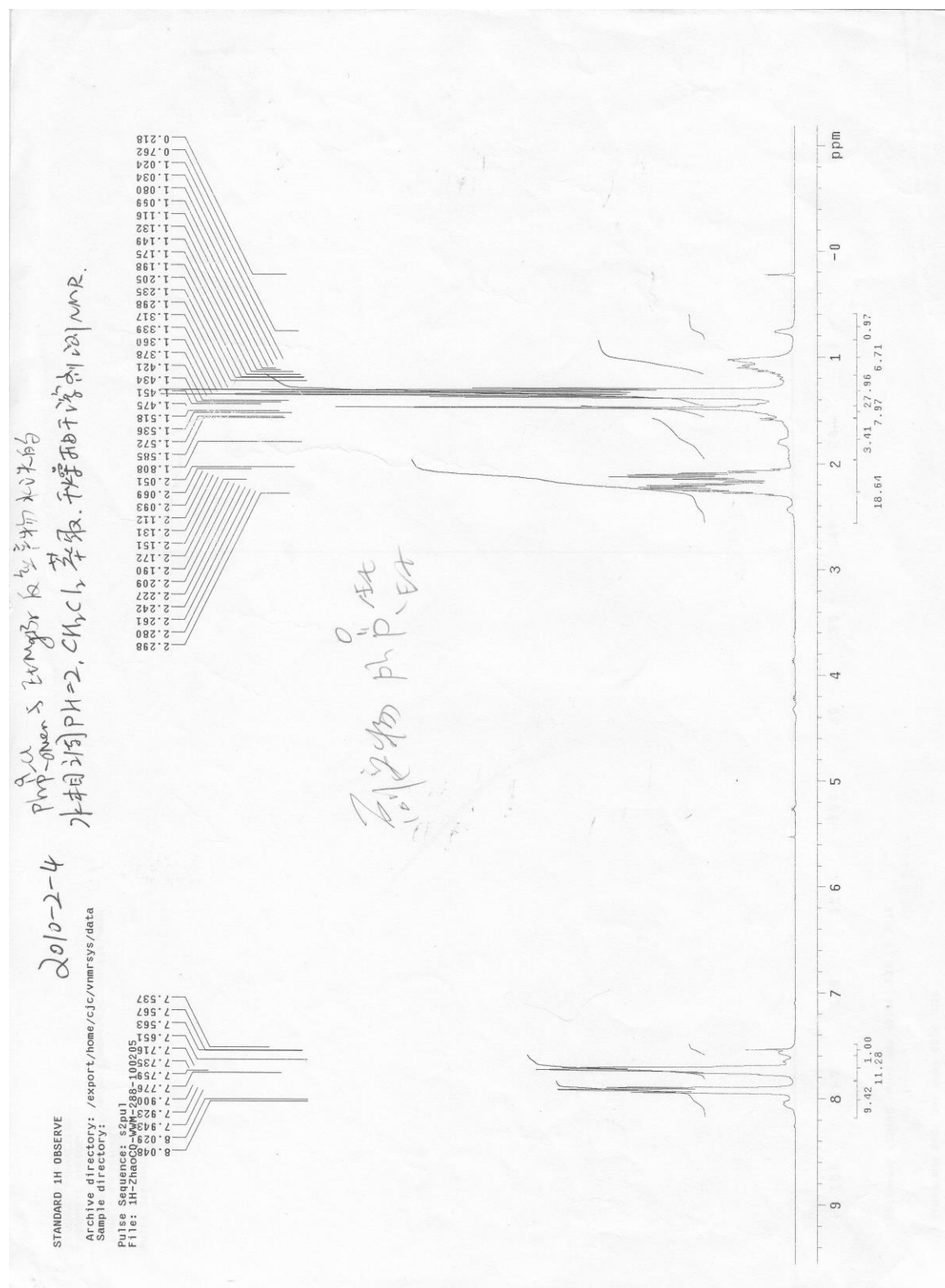
(S_P)-O-Menthyl *p*-methylphenylphenylphosphinate (12n')



(S_P)-O-Menthyl *p*-methylphenylphenylphosphinate (12n')

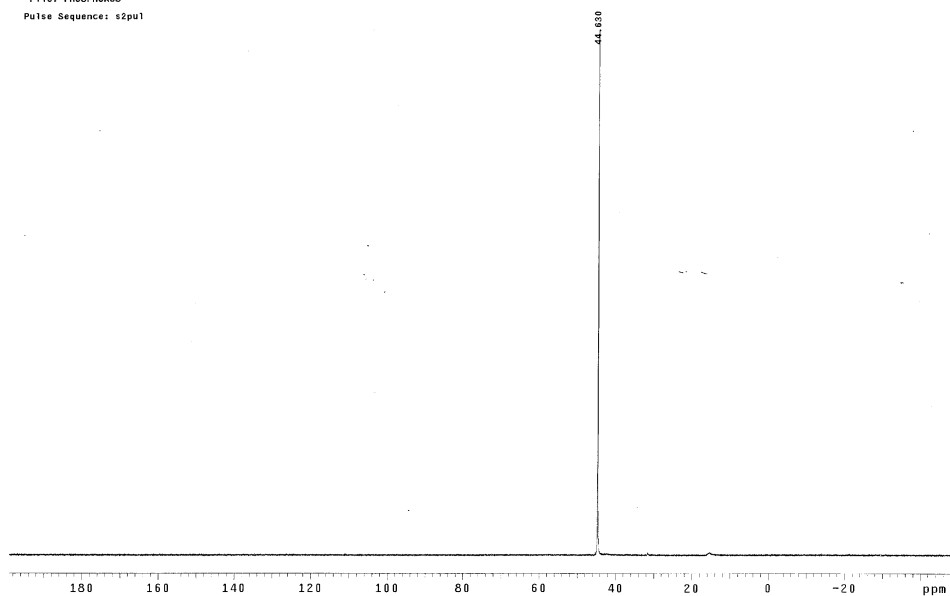


Diethyl phenylphosphine oxide 13b



Diethyl phenylphosphine oxide 13b

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION
Archive directory: /export/home/cjc/vnmrsys/data
Sample directory:
File: PHOSPHORUS
Pulse Sequence: s2pul



Customer: LCU400 Cons SN: 12186 Feb 5 2010

Probe:ASW PFG Ser num: 6094 c/c