Boron Lewis Acid-Catalyzed Hydrophosphinylation of *N*-Heteroaryl-Substituted Alkenes with Secondary Phosphine Oxides

Jimin Han, Jongwon Kim, Jaehoo Lee, Younghun Kim, and Sarah Yunmi Lee*

Department of Chemistry, Yonsei University, 50 Yonsei-ro, Seodaemun-gu, Seoul 03722, Korea
sarahyunmi@yonsei.ac.kr

Supporting Information

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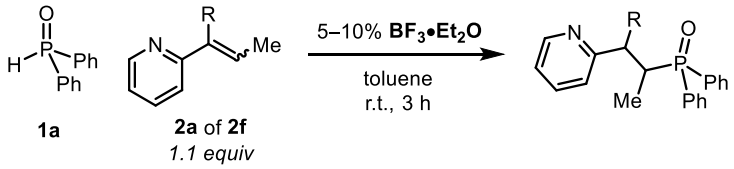
I. Development of Boron-Catalyzed Hydrophosphinylations of Alkenes

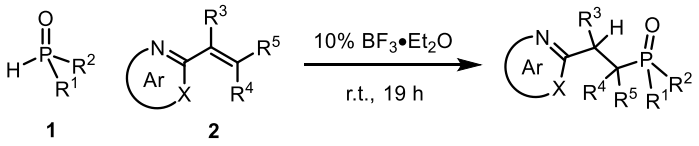
Table S1. *Effect of catalyst loadings and reaction concentrations.*[a,b]



entry	alkene	catalyst	solvent	yield (%)[c]
1	H	5%	0.5 M	90
2	H	5%	1 M	99
3	Me	10%	0.5 M	66
4	Me	10%	1 M	84

[a] Reaction conditions: Secondary phosphine oxide (0.05 mmol), alkene (1.2 equiv), and catalyst (5—10%) in toluene at r.t. for 3 h. [b] 2-(prop-1-en-1-yl)pyridine (*E*:*Z* ratio of 5.0:1.0) and (*E*)-2-(but-2-en-2-yl)pyridine were used. [c] Determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

Table S2. *Effect of solvents for the reactions with challenging substrates.*[a,b]

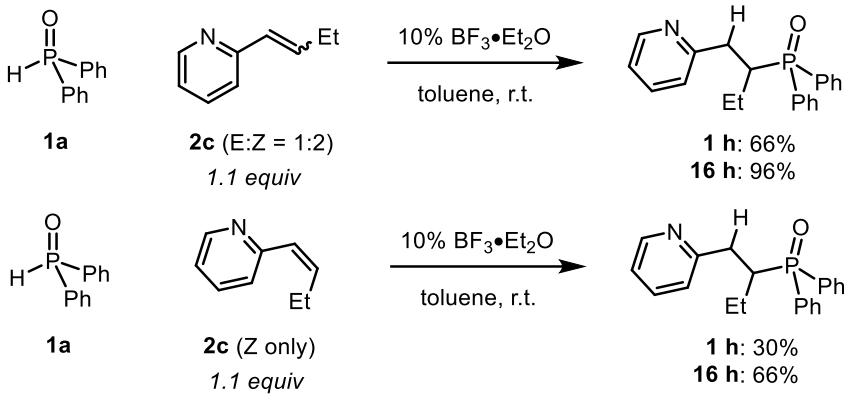


entry	phosphine oxide	alkene	solvent	yield (%)[c]
1	R^1, R^2 =		toluene	33
2			DCM	30
3	R^1, R^2 =		tolunene	53
4			DCM	40
5	R^1, R^2 =		toluene	41
6			DCM	59
7	R^1, R^2 = Me, Ph		toluene	18
8			DCM	11

[a] Reaction conditions: Secondary phosphine oxide (0.05 mmol), alkene (1.0 equiv), and catalyst (10%) in toluene (0.5 M) at r.t. for 19 h. [b] 1-Methyl-2-(prop-1-en-1-yl)-1H-indole (*E*:*Z* ratio of 1.2:1.0) was used. Racemic mixture of methyl(phenyl)phosphine oxide was used. [c] Determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

: Our boron-catalyzed hydrophosphinylation generally proceeded well in either toluene or dichloromethane as a reaction solvent; when the phosphine oxide containing *p*-methoxyphenyl group was employed, the reaction in dichloromethane resulted in higher yield of the product compared to that in toluene, due to the low solubility of the phosphine oxide in toluene.

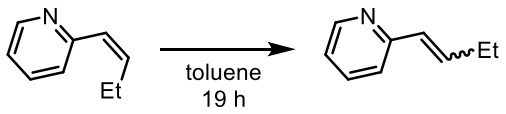
Scheme S1. *Effect of alkene geometry on the reaction rate.*[a,b]



[a] Reaction conditions: Secondary phosphine oxide (0.05 mmol), alkene (1.1 equiv), and catalyst (10%) in toluene (0.5 M) at r.t. for 19 h. [b] The yield of the product was determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

: The reaction of alkene **2c** (*E*:*Z* ratio 1:2) with **1a** proceeded faster than that of alkene **2c** (*Z* only).

Table S3. *The Z-to-E Isomerization of (Z)-2-(but-1-en-1-yl)pyridine in the presence of a borane.*[a]

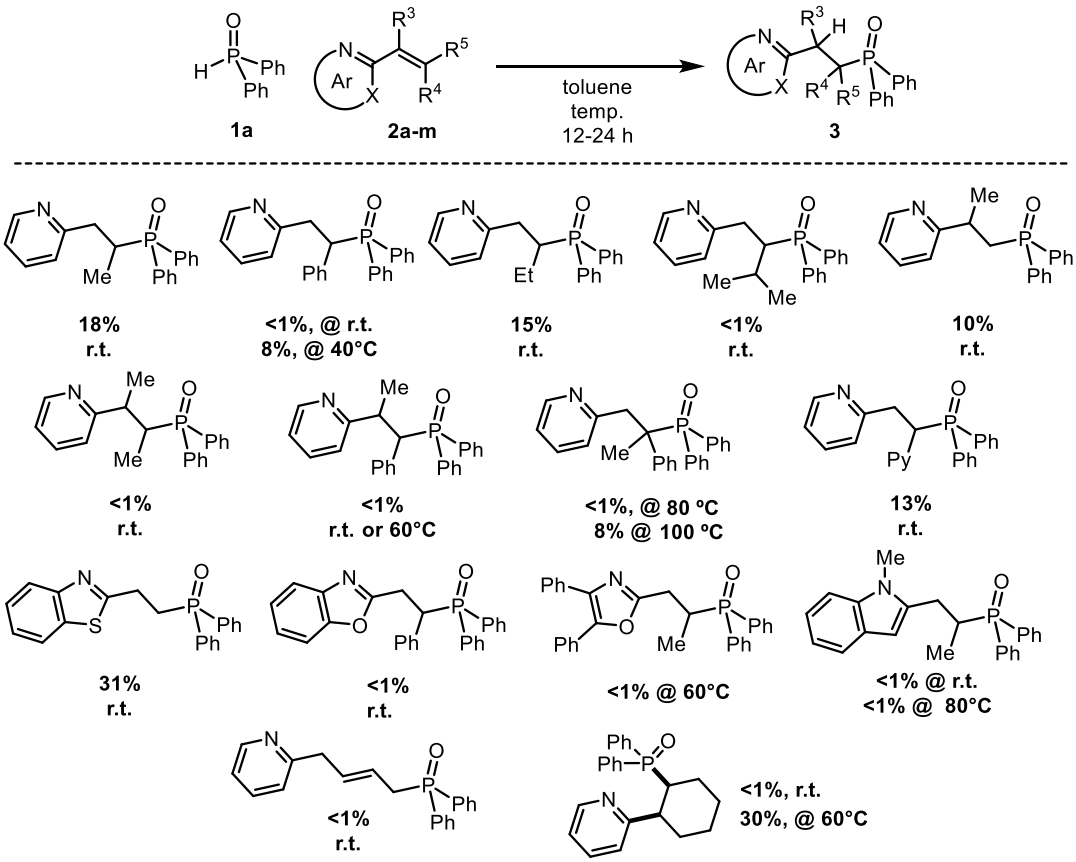


entry	catalyst	temperature	*E*:*Z*[b]
1	–	r.t.	< 1: 20
2	–	80 °C	< 1: 20
3	10% BF_3•Et_2O	r.t.	1:4
4	10% BF_3•Et_2O	80 °C	> 20:1
5	10% $B(C_6F_5)_3$	r.t.	1:6
6	10% $B(C_6F_5)_3$	80 °C	10:1

[a] Reaction conditions: alkene (1.0 equiv), and catalyst (10%) in toluene (0.5 M) at r.t. for 19 h. [b] The E:Z ratio was determined by ^{1}H NMR spectroscopic analysis.

: We observed the isomerization of alkene **2c** in the presence of a borane catalyst at room temperature or 80 °C.

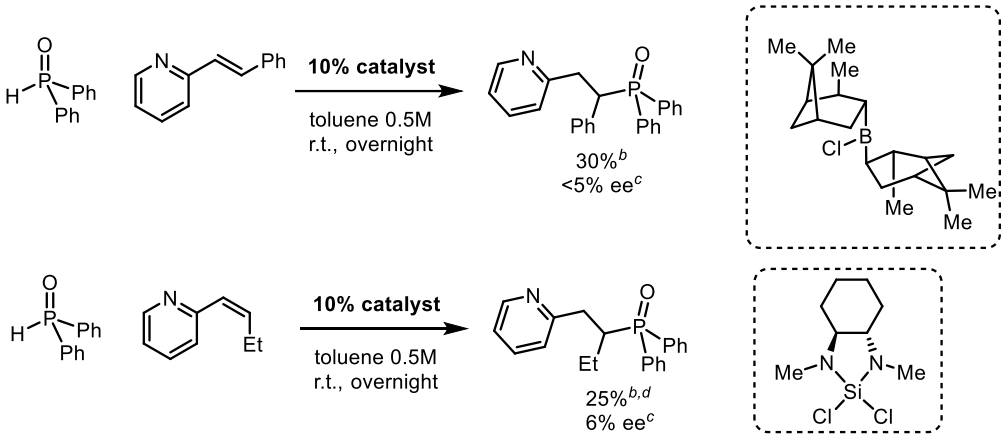
***Scheme S2**. Reactions with no catalyst: background reactions for few substrates.*[a,b]



[a] Reaction conditions: **1a** (0.05 mmol), alkene (1.1 equiv) in toluene (0.5–1 M) at the given temperature for 12–24 h. [b] The yield of the product was determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

: Few alkene substrates reacted slowly with SPO **1a** in the absence of a catalyst.

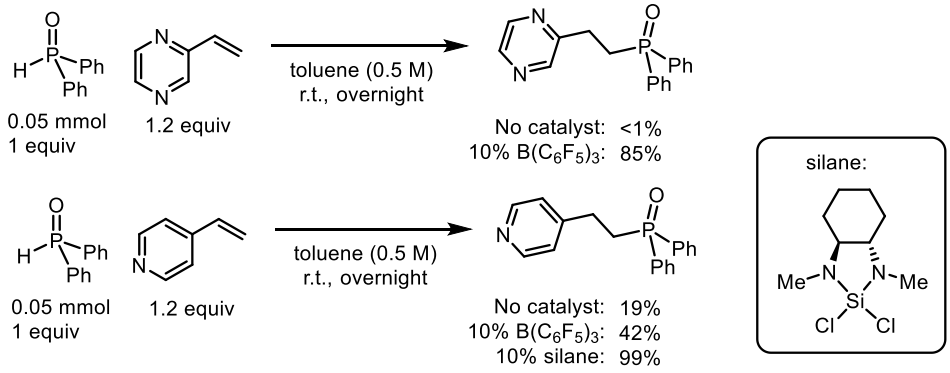
***Scheme S3**. Reactions with chiral catalysts.*[a,b]



[a] Reaction conditions: Secondary phosphine oxide (0.05 mmol), alkene (1.0 equiv), and catalyst (10%) in toluene (0.5 M). [b] Determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard. [c] Determined by AD column, IPA 10 – 20%. [d] In the absence of the catalyst: <5% yield.

: Our brief investigations to develop catalytic asymmetric hydrophosphinylations were not successful.

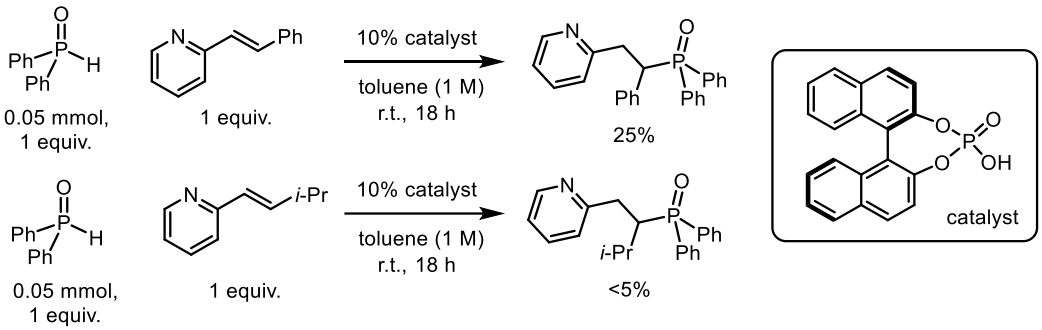
***Scheme S4**. Reactions of alkene substrates that are not included in the main paper.*[a,b]



[a] Reaction conditions: Secondary phosphine oxide (0.05 mmol), alkene (1.2 equiv), and catalyst (10%) in toluene (0.5 M). [b] Determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

: Since our hydrophosphinylation focused on the use of multisubstituted alkenes, we did not include these substrates in the paper, however, alkenes bearing a 4-pyridyl substituent and a pyrazyl substituent.

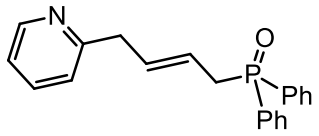
***Scheme S5**. Hydrophosphinylations catalyzed by a Brønsted acid.*[a,b]



[a] Reaction conditions: Secondary phosphine oxide (0.05 mmol), alkene (1 equiv), and catalyst (10%) in toluene (1 M). [b] Determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

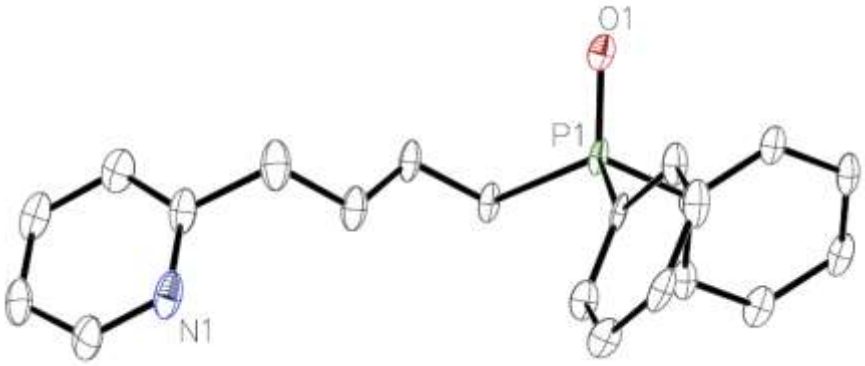
: Brønsted acids as a catalyst did not effectively promote the hydrophosphinylation.

II. X-ray Crystallographic Data for 3ao



Diphenyl[4-(pyridin-2-yl)but-2-en-1-yl]phosphine oxide (eq 1, 3ao).

Note: The structure of product **3ao** was determined by X-ray crystallography**.** Crystals suitable for X-ray crystallography were grown from the saturated solution of $CHCl_3$/hexanes at -20 °C.



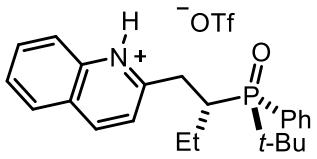
The diffraction data from yellow crystals of **3ao** (0.075 × 0.035 × 0.020 mm^3) mounted on a MiTeGen MicroMount© were collected at 100 on a ADSC Quantum 210 CCD diffractometer with synchrotron radiation (0.8000 Å) at Supramolecular Crystallography 2D, Pohang Accelerator Laboratory (PAL), Pohang, Korea. The ADSC Q210 ADX program[1] was used for data collection (detector distance is 66 mm, omega scan; $\Delta\omega = 3^o$, exposure time is 2 sec/frame for **3ao** and HKL3000sm (Ver. 703r)[2] was used for cell refinement, reduction and absorption correction. The crystal structures of **3ao** was solved by the direct method with SHELX-XT (Ver. 2014/5)[3] and refined by full-matrix least-squares calculations with the SHELX-XL (Ver. 2016/4)[4] program package.

Table S4. Crystal data and structure refinement for 3ao.

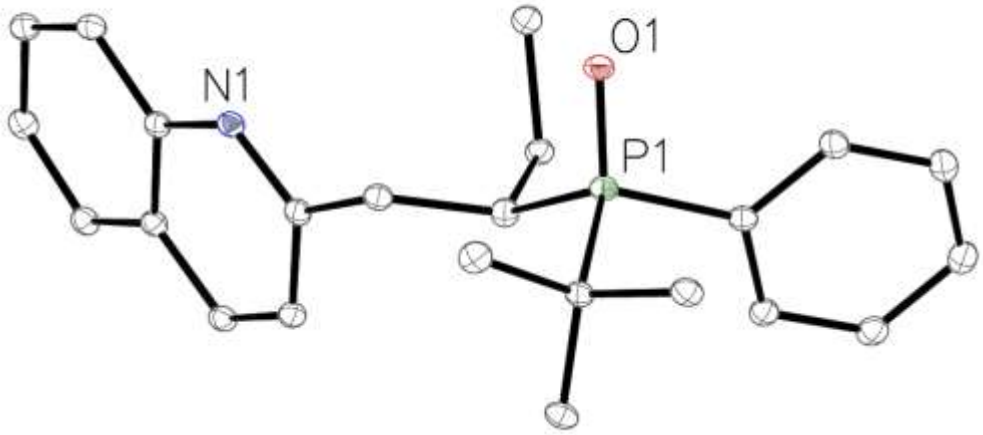
Identification code	3ao
Empirical formula	$C_{21}H_{20}NOP$
Formula weight	333.35
Temperature/K	100
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	5.8300(12)
b/Å	24.776(5)
c/Å	12.198(2)
α/°	90
β/°	101.64(3)
γ/°	90
Volume/Å^3	1725.7(6)
Z	4
ϱ_{calc}g/cm^3	1.283
μ/mm^{-1}	0.227
F(000)	704.0
Crystal size/mm^3	0.075 × 0.035 × 0.02
Radiation	synchrotron (λ = 0.800)
2Θ range for data collection/°	3.7 to 55.994
Index ranges	$-6 \le h \le 6$, $-29 \le k \le 29$, $-14 \le l \le 14$
Reflections collected	10533
Independent reflections	2841 [R_{int} = 0.0494, R_{sigma} = 0.0335]
Data/restraints/parameters	2841/6/217
Goodness-of-fit on F^2	1.072
Final R indexes [I>=2σ (I)]	R_1 = 0.0378, wR_2 = 0.1019
Final R indexes [all data]	R_1 = 0.0412, wR_2 = 0.1044
Largest diff. peak/hole / e Å^{-3}	0.57/-0.34

III. X-ray Crystallographic Data for 3kp·HOTf

Note: The structure of product **3kp** was determined by X-ray crystallography of the corresponding quinolinium triflate**.** Crystals suitable for X-ray diffraction were grown from dichloromethane/ether at room temperature.



^{1}H NMR (400 MHz, $CDCl_3$) δ 8.91 (d, *J* = 8.5 Hz, 1H), 8.37 (d, *J* = 8.5 Hz, 1H), 8.29 (d, *J* = 8.5 Hz, 1H), 8.20 (d, *J* = 8.3 Hz, 1H), 8.09 (t, *J* = 7.7 Hz, 1H), 7.89 (dd, *J* = 17.0, 9.0 Hz, 3H), 7.71 – 7.56 (m, 3H), 4.25 – 3.97 (m, 2H), 3.27 – 3.12 (m, 1H), 1.84 (s, 2H), 1.76 – 1.61 (m, 1H), 1.34 (d, *J* = 15.1 Hz, 9H), 0.95 (t, *J* = 7.3 Hz, 3H). **^{31}P NMR** (162 MHz, $CDCl_3$) δ 60.75 (s).



(Only one enantiomer is shown.)

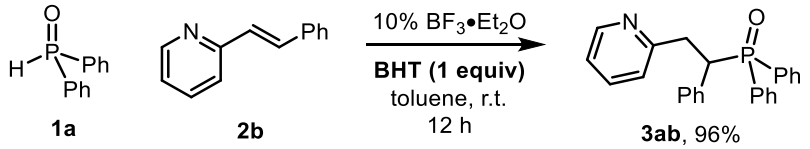
The diffraction data from yellow crystals of **LYM1** (0.09 × 0.09 × 0.04 mm^3) mounted on a MiTeGen MicroMount© were collected at 100 on a ADSC Quantum 210 CCD diffractometer with synchrotron radiation (0.7000 Å) at Supramolecular Crystallography 2D, Pohang Accelerator Laboratory (PAL), Pohang, Korea. The ADSC Q210 ADX program[1] was used for data collection (detector distance is 66 mm, omega scan; Δω = 3º, exposure time is 1 sec/frame for **LYM1** and HKL3000sm (Ver. 703r)[2] was used for cell refinement, reduction and absorption correction. The crystal structures of **LYM1** was solved by the direct method with SHELX-XT (Ver. 2014/5)[3] and refined by full-matrix least-squares calculations with the SHELX-XL (Ver. 2016/4)[4] program package.

Table S5. Crystal data and structure refinement for LYM1.

Identification code	LYM1
Empirical formula	$C_{24.01}H_{29.03}F_3NO_4PS$
Formula weight	515.73
Temperature/K	100
Crystal system	triclinic
Space group	P-1
a/Å	8.5600(17)
b/Å	12.297(3)
c/Å	13.486(3)
α/°	69.28(3)
β/°	77.10(3)
γ/°	75.74(3)
Volume/Å^3	1272.3(5)
Z	2
ρ_{calc}g/cm^3	1.346
μ/mm^{-1}	0.233
F(000)	540.0
Crystal size/mm^3	0.09 × 0.09 × 0.04
Radiation	synchrotron (λ = 0.700)
2Θ range for data collection/°	3.216 to 55.994
Index ranges	-11 ≤ h ≤ 11, -16 ≤ k ≤ 16, -18 ≤ l ≤ 17
Reflections collected	11356
Independent reflections	5826 [R_{int} = 0.0420, R_{sigma} = 0.0825]
Data/restraints/parameters	5826/0/390
Goodness-of-fit on F^2	0.854
Final R indexes [I>=2σ (I)]	R_1 = 0.0579, wR_2 = 0.1445
Final R indexes [all data]	R_1 = 0.1276, wR_2 = 0.1703
Largest diff. peak/hole / e Å^{-3}	0.31/-0.43

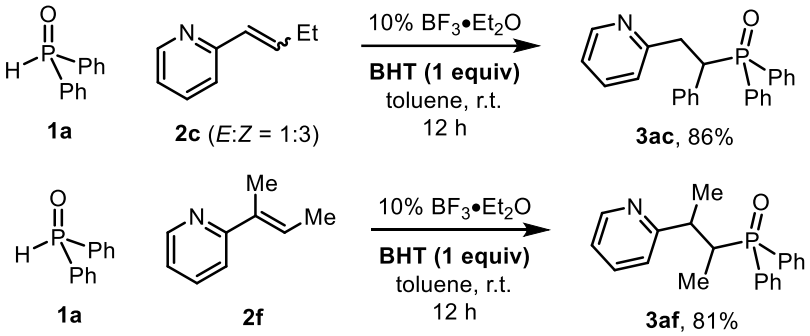
IV. Reactivity and Mechanistic Studies

Scheme 2a of the main paper. *Reaction in the presence of BHT.*

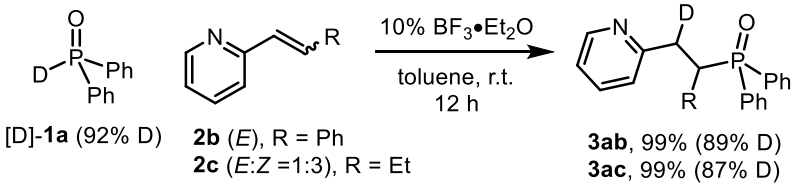


In a nitrogen-filled glovebox, diphenylphosphine oxide (10.1 mg, 0.050 mmol, 1 equiv.), (*E*)-2-styrylpyridine (7.30 mg, 0.050 mmol, 1 equiv.), 2,6-di-*tert*-butyl-4-methylphenol [128-37-0] (11.0 mg, 0.050 mmol) with 10% BF_3•Et_2O (0.005 mmol) and toluene (1 M) were combined in a 4-mL vial equipped with a stir bar. The resulting mixture was stirred at room temperature for 12 h. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The yield of the product was determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

**Additional information: Reactions of other alkenes in the presence of BHT.*



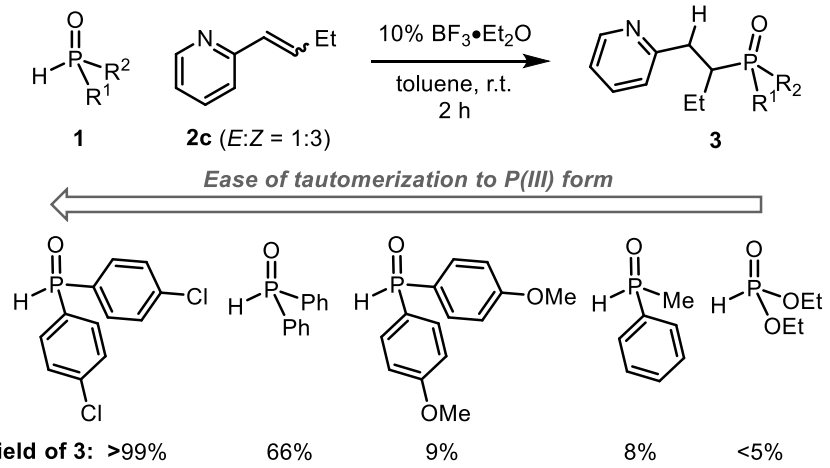
Scheme 2b of the main paper. *Reaction with deuterated diphenylphosphine oxide***.**



In a nitrogen-filled glovebox, deuterated diphenylphosphine oxide (0.05 mmol, 1.0 equiv.), corresponding alkene (1.1 equiv.), 20% BF_3•Et_2O (0.010 mmol) and toluene (1 M) were combined in a 4-mL vial equipped with a stir bar. The resulting mixture was stirred at room temperature for 12 h. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The yield of the product and deuterium incorporation was determined by ^{1}H NMR spectroscopic analysis.

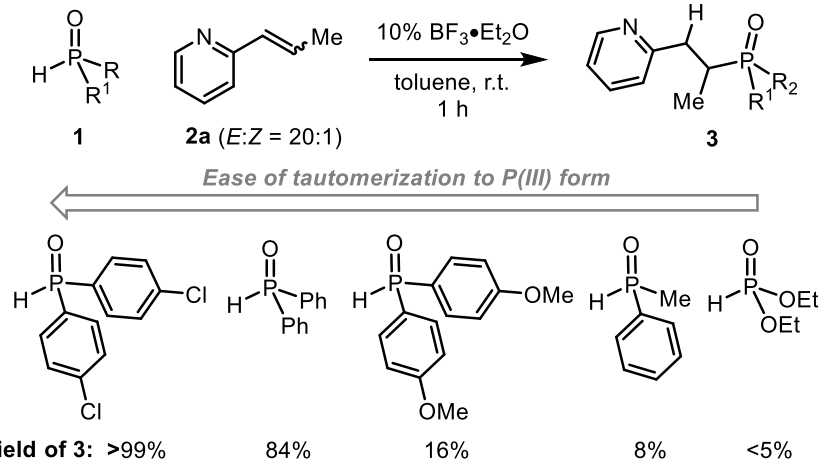
Note:* *Deuterated diphenylphosphine oxide was prepared according the following procedure.* To an oven dried 20-mL vial was added diphenylphosphine oxide [4559-70-0] (0.6 mmol) and a stir-bar. The solid was then dissolved in d_4-MeOH (1.2 mL) to obtain a 0.5 M solution. The resulting solution was stirred at room temperature for 12 hours. The clear solution was concentrated *in vacuo*. ^{1}H NMR spectrum showed 92% D incorporation. **^{1}H NMR (400 MHz, MeOD) δ 8.73 (s, 0.08H), 7.81 – 7.73 (m, 2H), 7.72 – 7.65 (m, 1H), 7.64 –7.55 (m, 2H).

Scheme 2c of the main paper. *Reactivity trend of various phosphine oxides toward additions to an alkene.*



In a nitrogen-filled glovebox, secondary phosphine oxide (10.1 mg, 0.050 mmol), 2-(but-1-en-1-yl)pyridine [71532-20-2] (*E*:*Z* = 1.0:3.0; 9.96 mg, 0.055 mmol), BF_3•Et_2O (0.0050 mmol) and toluene (50 μL) were combined in a 4-mL vial equipped with a stir bar. The resulting mixture was stirred at room temperature for 2 h. The mixture was then quenched with 5 μL IPA, and the solvent was evaporated under reduced pressure. The yield of the product was determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

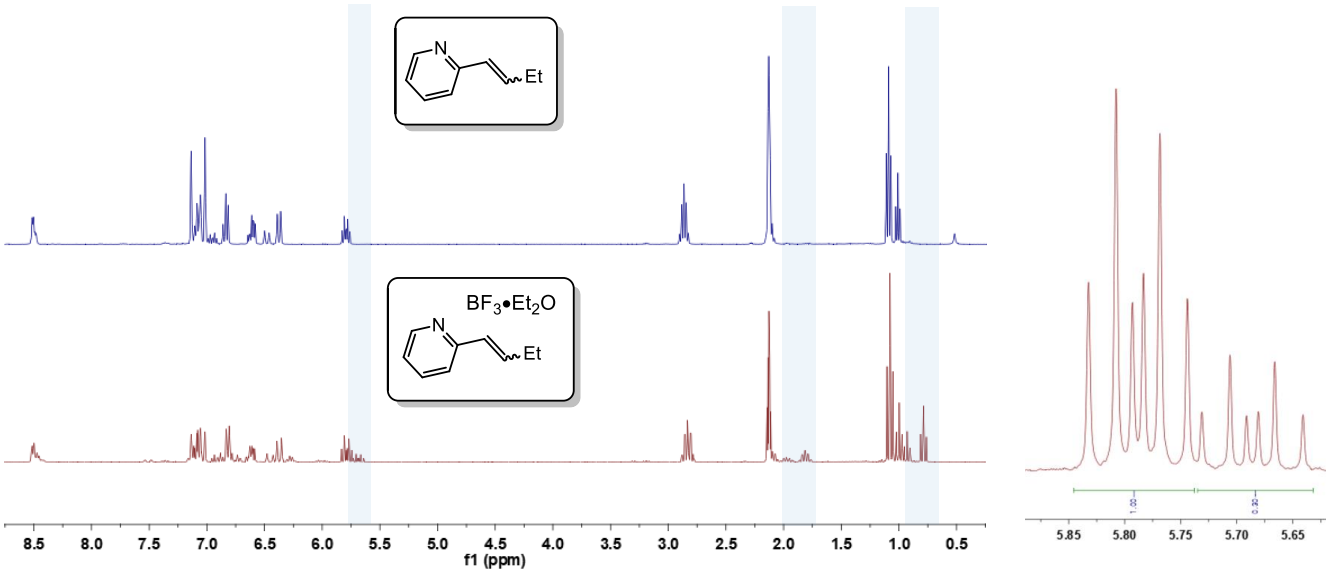
Additional information: The same reactivity trend was observed for the reaction with alkene **2a.*



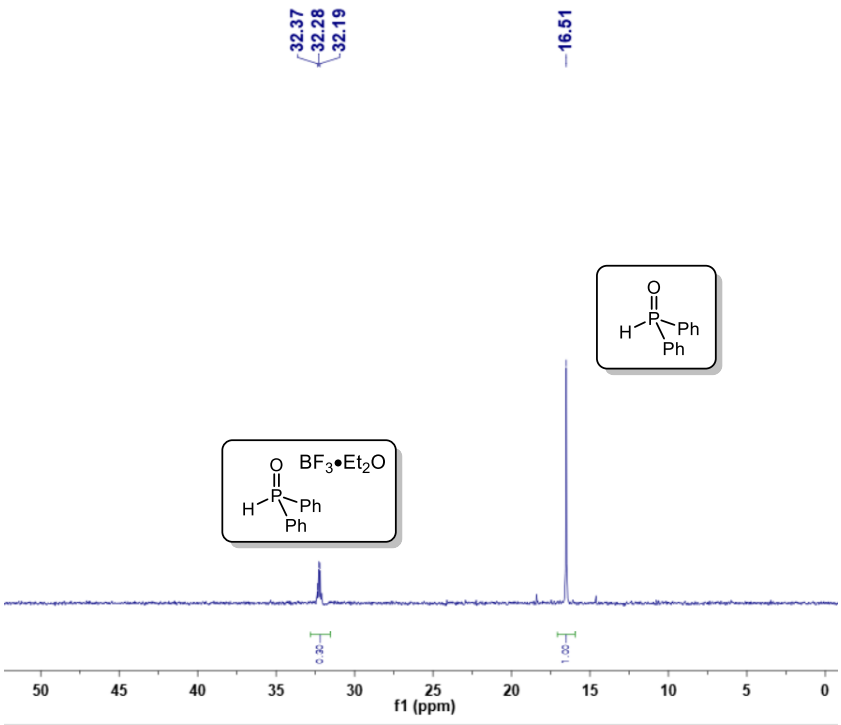
<NMR Spectroscopic studies>

1. **Monitoring of the catalytic reaction between SPO 1a and alkene 2c by ^{1}H and ^{31}P NMR spectroscopy.** *30% of BF_3•Et_2O was used to facilitate the NMR spectroscopic analysis.*

1) Interaction between BF_3•Et_2O (30%) and alkene **2c** at r.t. in toluene-d_8. (^{1}H NMR spectra)

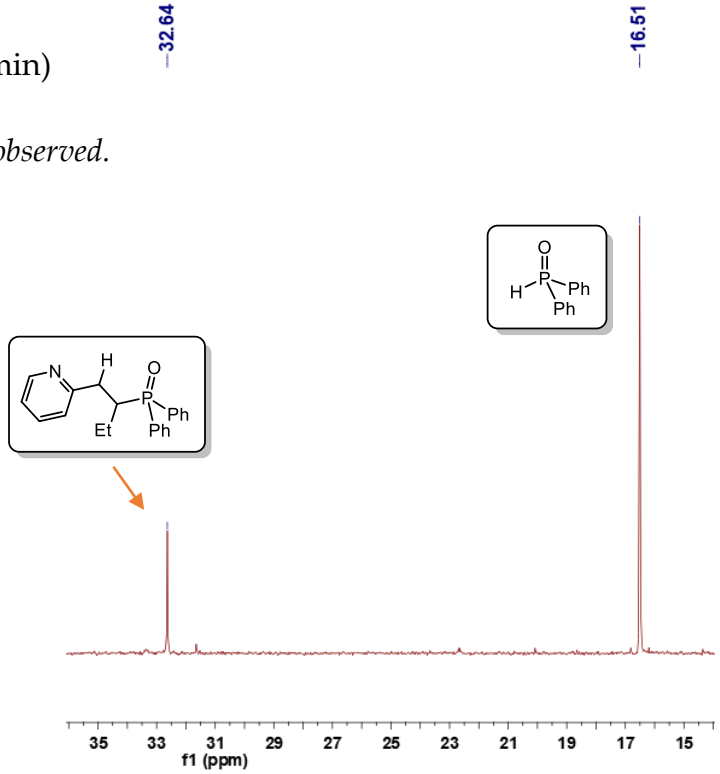


2) Interaction between BF_3•Et_2O (30%) and diphenylphosphine oxide **1a** at r.t. in toluene-d_8. (^{31}P NMR spectra)



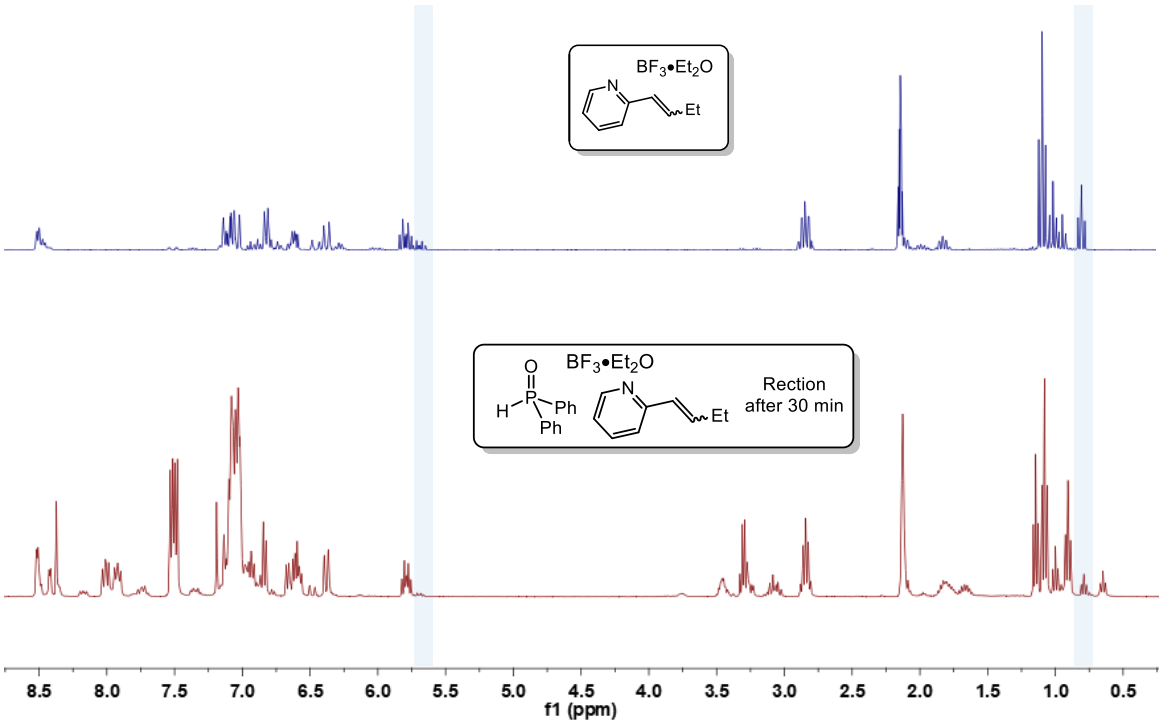
3) ^{1}H and ^{31}P NMR spectra of the reaction between diphenylphosphine oxide **1a** and alkene **2c** in the presence of $BF_3 \bullet Et_2O$ (30%) at r.t. in toluene-*d*$_8$, after ~30 min.

(^{31}P NMR spectra of
the crude mixture after 30 min)



<5% of borane-SPO form was observed.

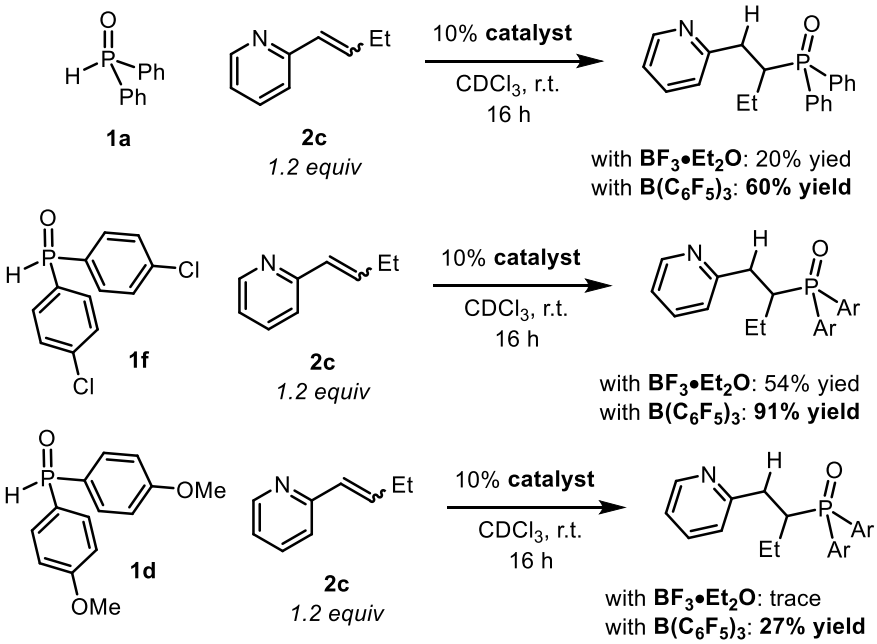
(^{1}H NMR spectra)



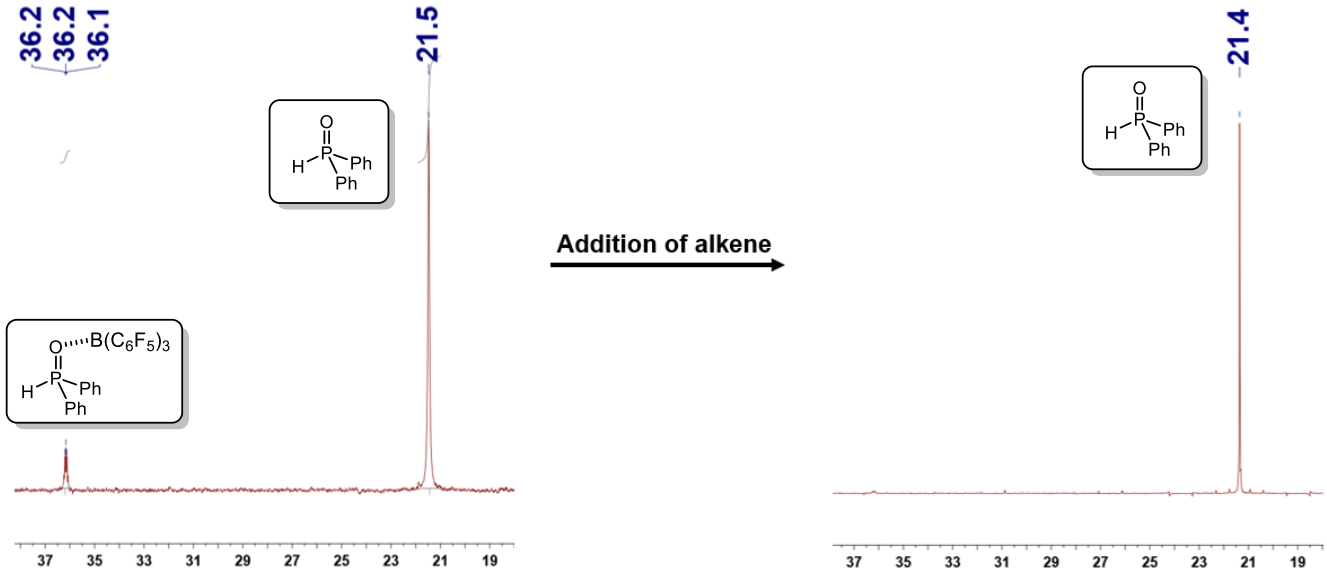
: Interaction between borane and alkene was observed during the catalytic reaction.

2. **Monitoring of the catalytic reaction between SPOs 1a, 1d, 1f and alkene 2c by ^{1}H and ^{31}P NMR spectroscopy.** *30% of $B(C_6F_5)_3$ was used to facilitate the NMR spectroscopic analysis.*

Notes:* a) Due to the low solubility of SPO **1d in toluene, we performed these reactions in $CDCl_3$; b) The hydrophosphinylation in $CDCl_3$ proceeded smoothly to produce the corresponding products (see below); c) In $CDCl_3$, reactions with $B(C_6F_5)_3$ led to the formation of products in higher yields compared to that with $BF_3 \bullet Et_2O$.



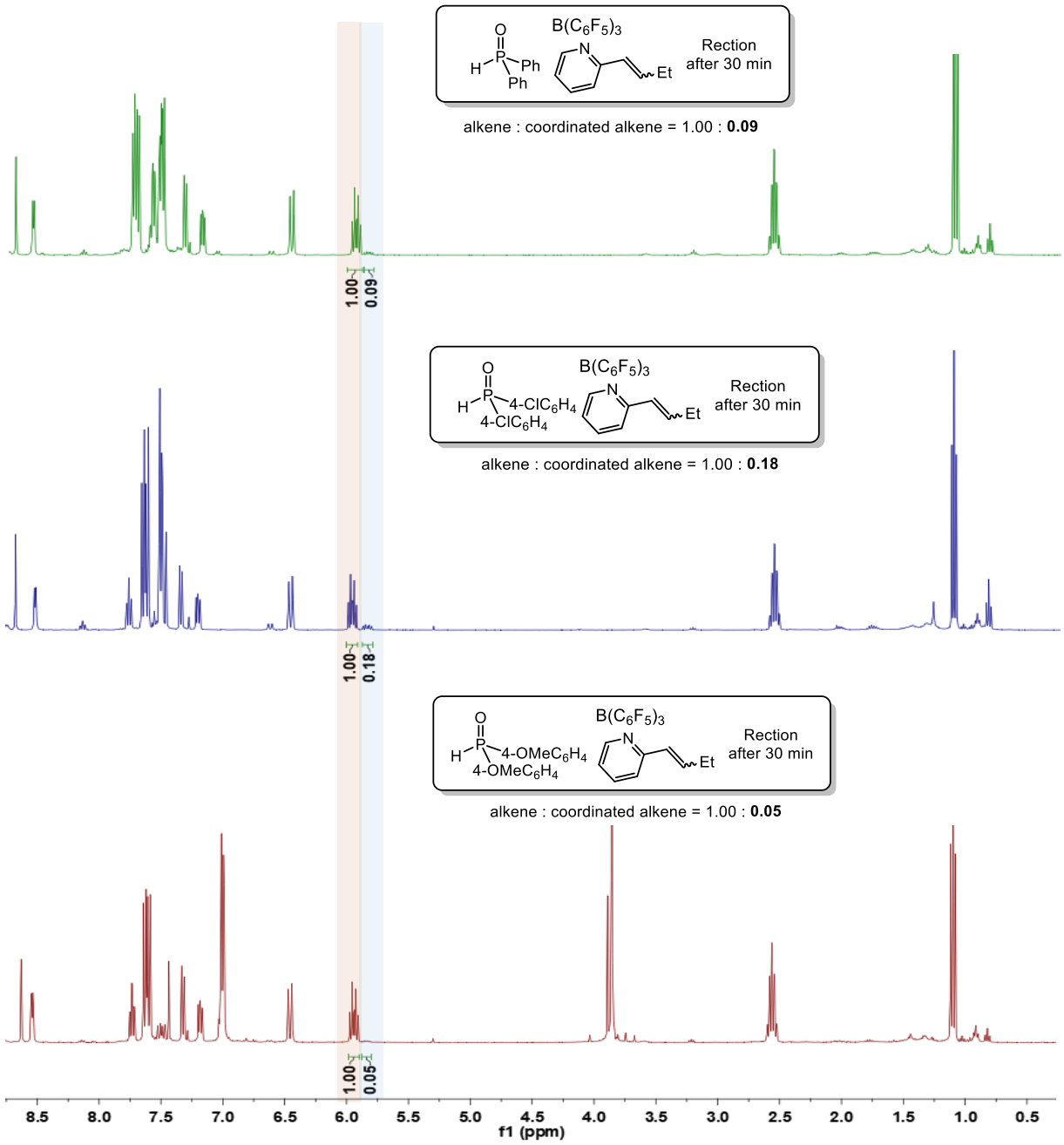
1) Interaction between $B(C_6F_5)_3$ (30%), SPO **1a**, and alkene at r.t. in $CDCl_3$. (^{31}P NMR spectra)



: The ^{31}P NMR signal (B-P interaction) disappeared upon the addition of alkene.

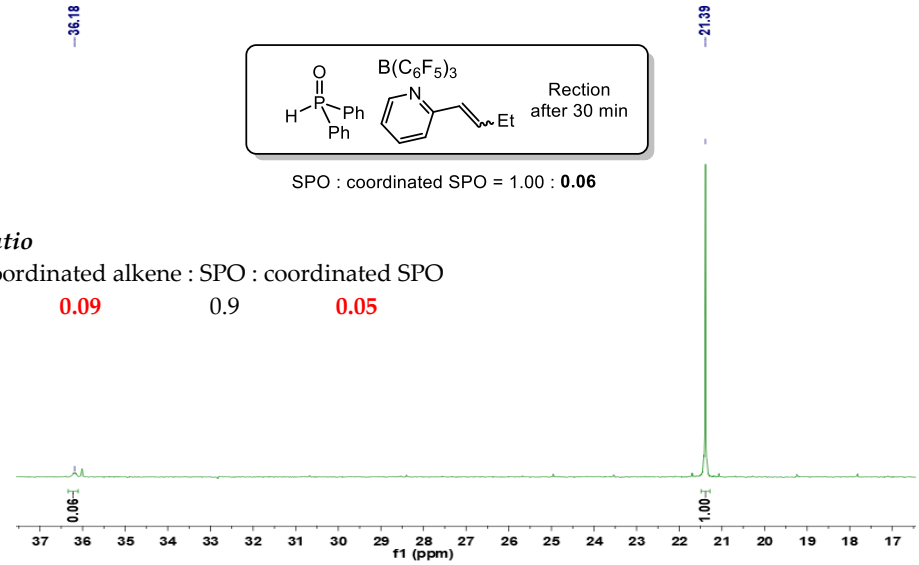
2) ^{1}H and ^{31}P NMR spectra of the reaction between SPO **1a, 1d, 1f** and alkene **2c** in the presence of $B(C_6F_5)_3$ (30%) at r.t. in $CDCl_3$, after ~30 min.

(^{1}H NMR spectra of the crude mixture)



: The higher percentage of borane-alkene species was observed when the SPO contained electron-withdrawing groups (1f > 1a> 1d).

(^{31}P NMR spectra of the crude mixture)

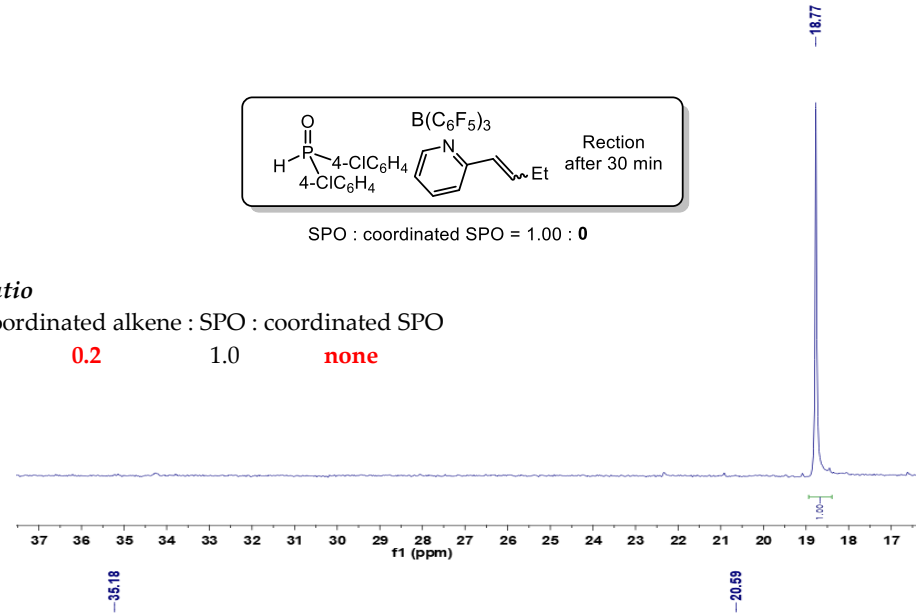


SPO : coordinated SPO = 1.00 : **0.06**

Overall ratio

alkene : coordinated alkene : SPO : coordinated SPO

1 **0.09** 0.9 **0.05**

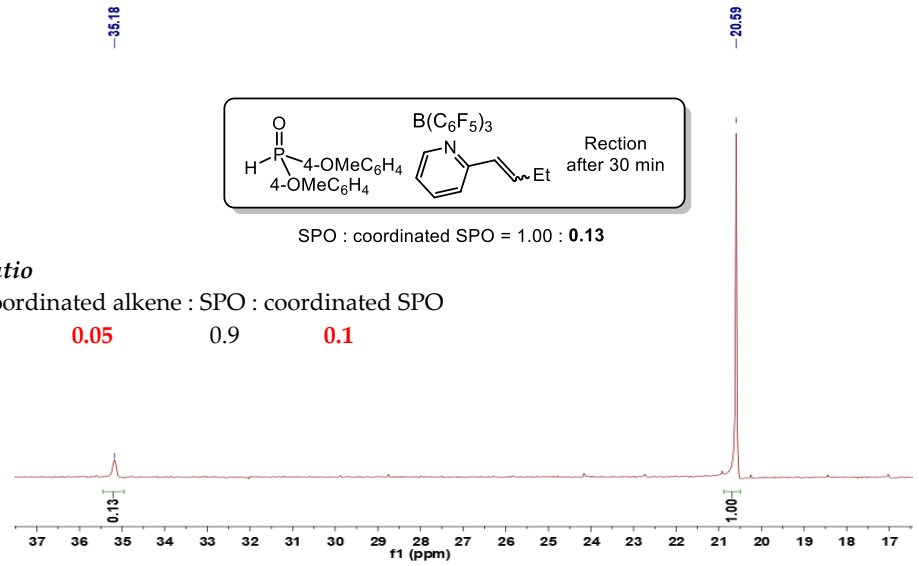


SPO : coordinated SPO = 1.00 : **0**

Overall ratio

alkene : coordinated alkene : SPO : coordinated SPO

1 **0.2** 1.0 **none**



SPO : coordinated SPO = 1.00 : **0.13**

Overall ratio

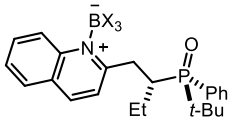
alkene : coordinated alkene : SPO : coordinated SPO

1 **0.05** 0.9 **0.1**

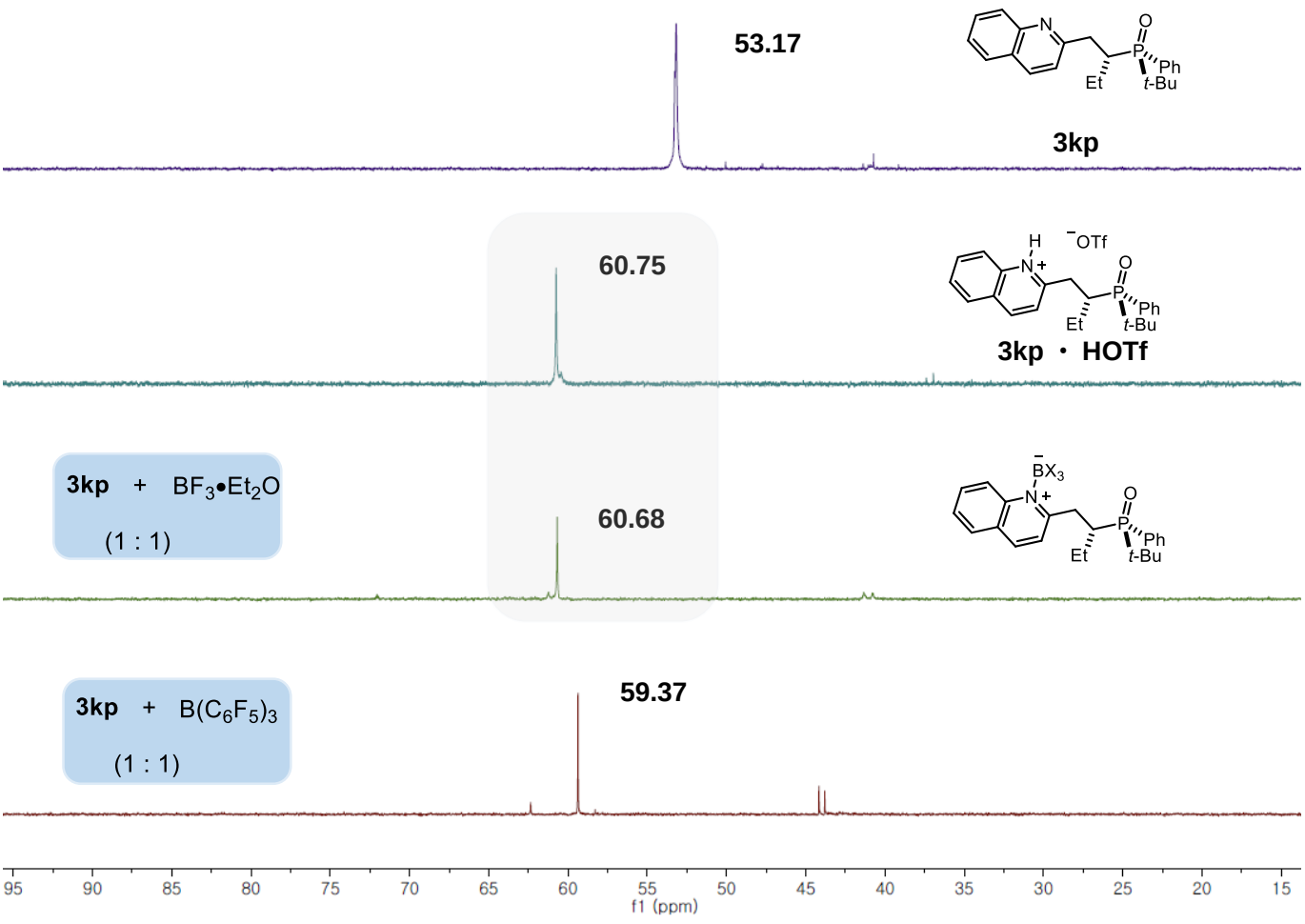
3. **(Scheme 2d)**

- **Monitoring the formation of the acid-base pair C between a borane and product 3kp by NMR spectroscopy: in comparison with the spectra of the quinolinium triflate of 3kp.** *Note*: the structure of **3kp**·HOTf was confirmed by X-ray crystallography.

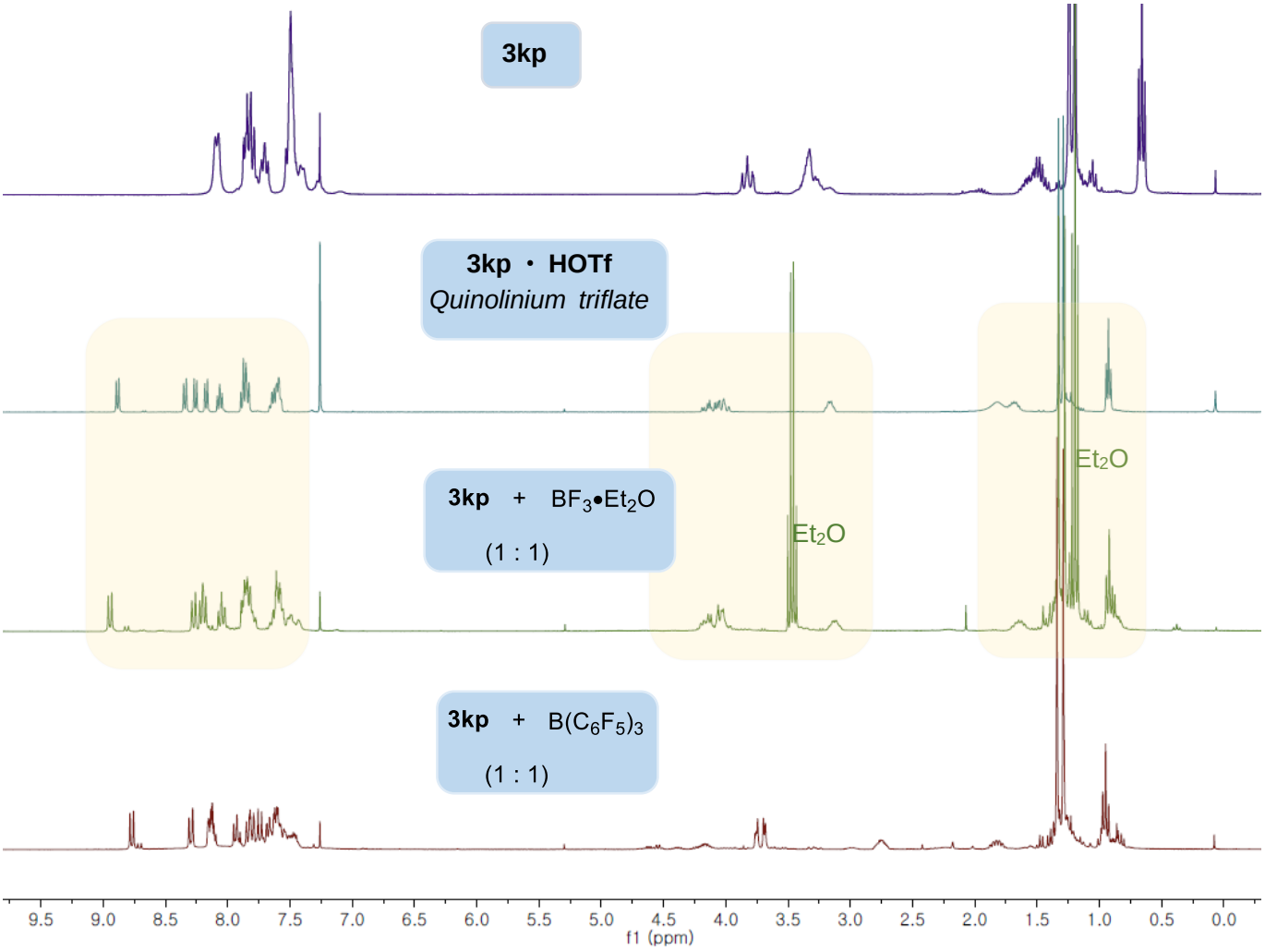
: Based on the following ^{31}P and ^{1}H spectra, we concluded that the <u>major</u> species formed by <u>the reaction between BF_3•Et_2O and product 3</u> was the the B–N acid-base pair.



1) ^{31}P NMR spectra of the mixture of **3kp** and borane (1:1) at r.t. in $CDCl_3$ (after ~30 min)



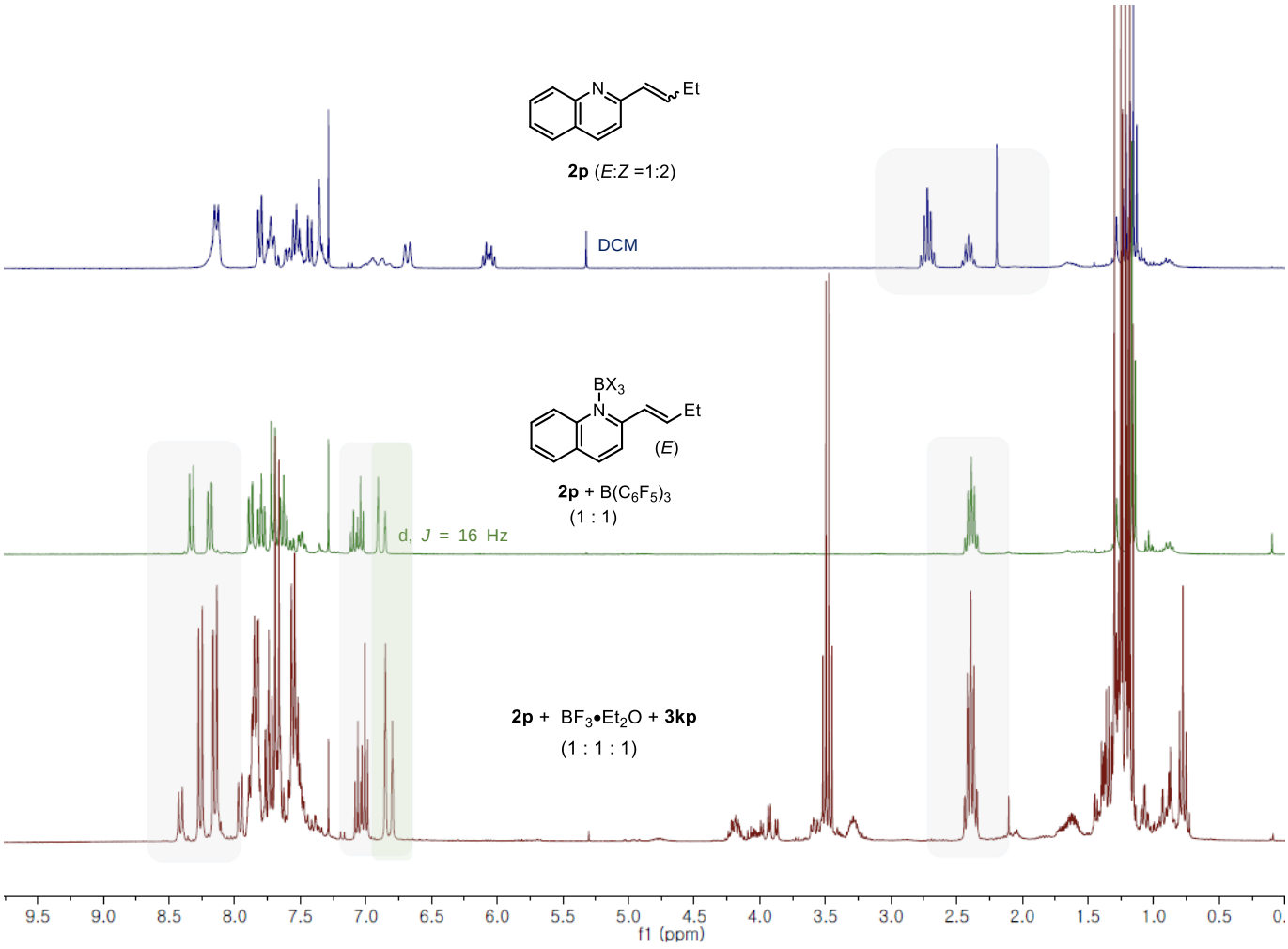
2) ^{1}H NMR spectra of the mixture of **3kp** and borane (1:1) at r.t. in $CDCl_3$ (after ~30 min)



- **Monitoring the generation of alkene-borane intermediate upon the addition of alkene 2p to the mixture of a borane and product 3kp by NMR spectroscopy.**

: Based on the following ^{1}H spectra, we concluded that the major species in the mixture of alkene 2p, a borane, and product 3kp was the activated form of alkene 2p.

(Note: In the presence of a borane catalyst, the E/Z mixture of alkene 2p transformed into (E)-alkene 2p.)



4. *(Additional information on the mechanism)*

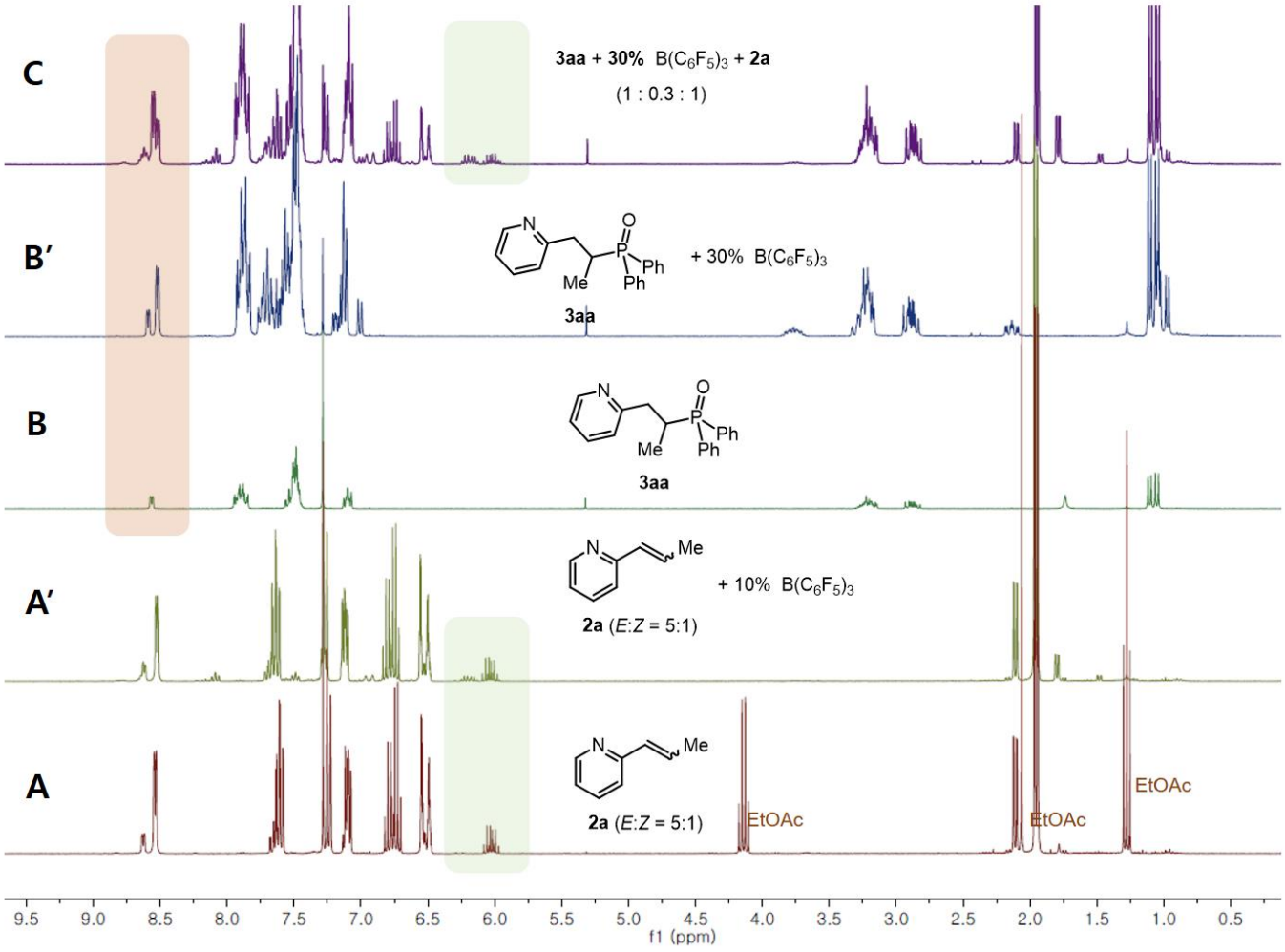
Monitoring the interaction among $B(C_6F_5)_3$, alkene 2a, and reaction product 3aa by ^1H and ^{31}P NMR spectroscopy.

1) Interaction among $B(C_6F_5)_3$ (30%), alkene **2a**, and product **3aa** at r.t. in $CDCl_3$. (^1H NMR spectra)

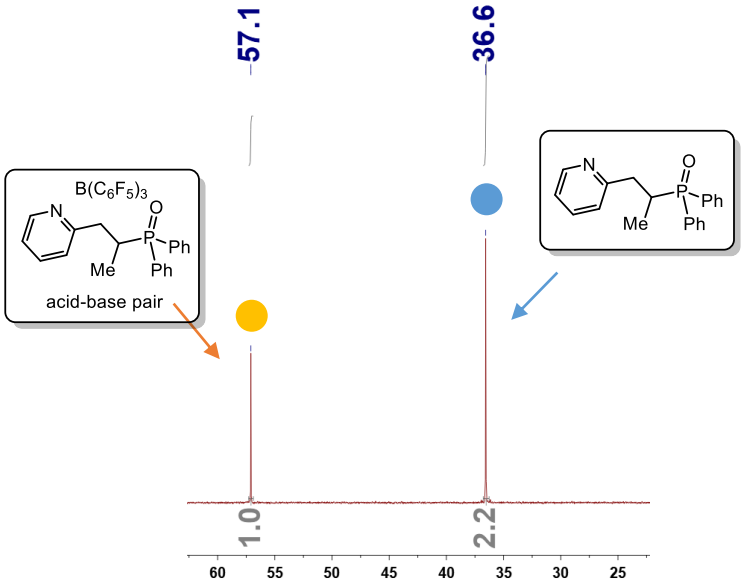
Note: <Spectrum C> Alkene 2a was added to the mixture of product 3aa and borane (30%).

: It displayed both interactions of borane-alkene and borane-product

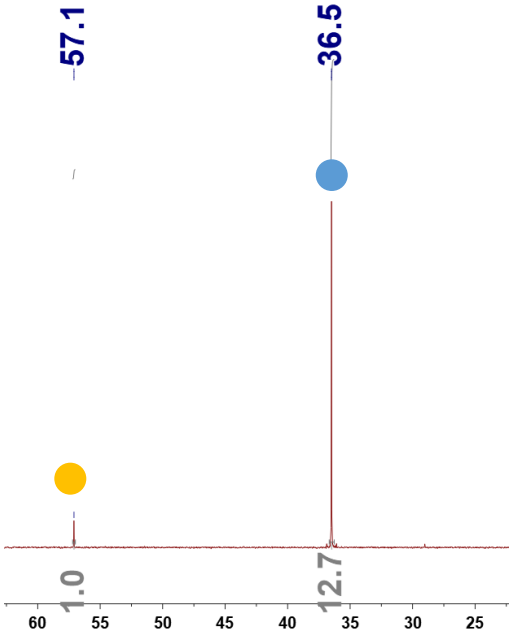
(see also ^{31}P spectra on the next page).



2) Interaction between $B(C_6F_5)_3$ (30%) and product **3aa** at r.t. in $CDCl_3$. (^{31}P NMR spectra)

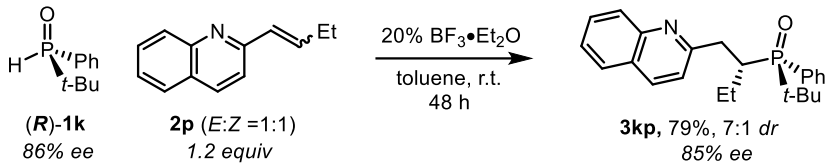


3) Interaction among $B(C_6F_5)_3$ (30%), product **3aa**, and alkene **2a** at r.t. in $CDCl_3$. (^{31}P NMR spectra). *Addition of alkene **2a** (1 equiv) to the mixture of $B(C_6F_5)_3$ (30%) and product **3aa** (1 equiv)*



: The interaction between product and borane considerably diminished upon adding alkene.

V. HPLC Spectra of 3kp (eq 2)



(*R*)-**1k**
86% ee

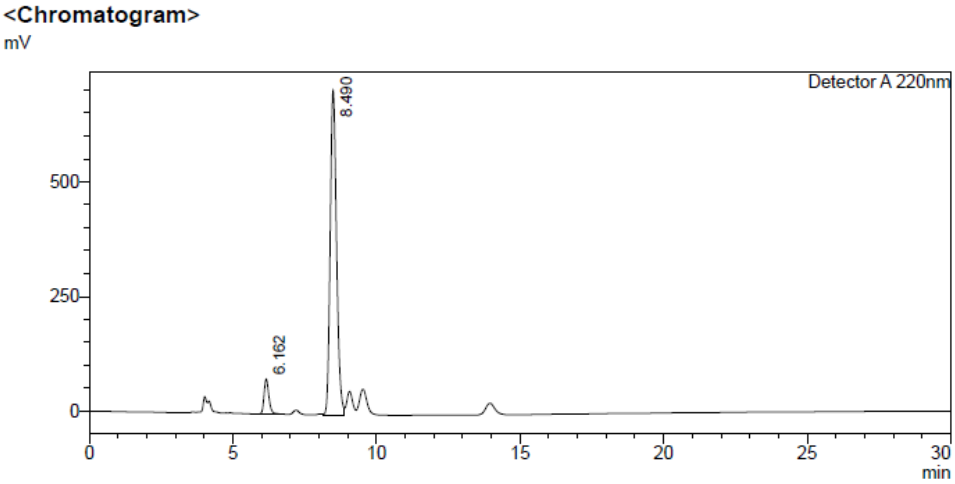
2p (*E*:*Z* =1:1)
1.2 equiv

3kp, 79%, 7:1 *dr*
85% ee

(*R*)-*tert*-Butyl(phenyl)((*R*)-1-(quinolin-2-yl)butan-2-yl)phosphine oxide (eq 2, 3kp).

The *ee* of the product was determined by HPLC (Daicel CHIRALCEL AD-H column; solvent system: 10.0% *i*-PrOH in hexanes; 1.0 mL/min; retention times: 6.16 min (minor), 8.49 min (major)).

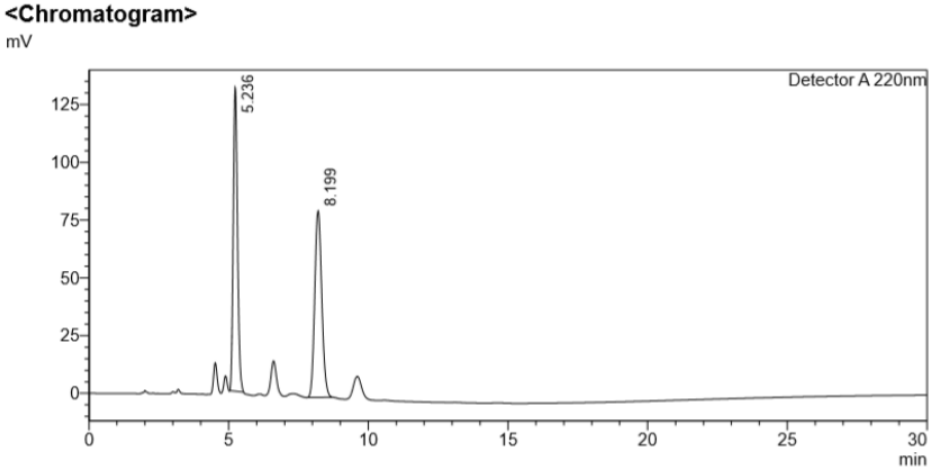
<HPLC trace of 85% ee of **3kp**>



<Peak Table>
Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	6.162	896029	76439	7.572			
2	8.490	10937012	707454	92.428			
Total		11833041	783893				

<HPLC trace of racemic **3kp**>



<Peak Table>
Detector A 220nm

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	5.236	1390356	131357	50.156			
2	8.199	1381695	80751	49.844			
Total		2772051	212108				

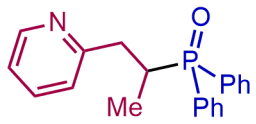
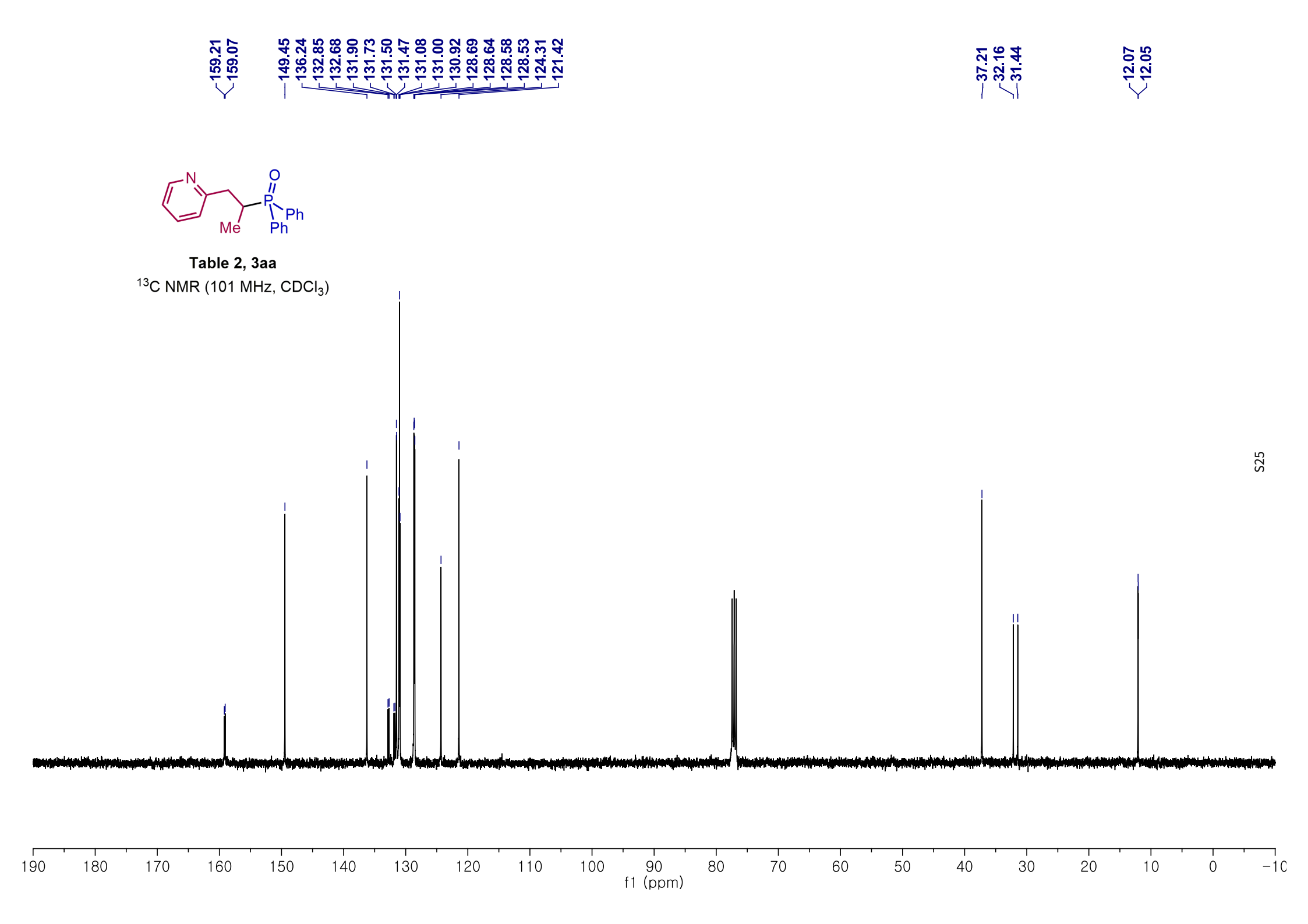


Table 2, 3aa

^{1}H NMR (400 MHz, $CDCl_3$)


159.21
159.07
149.45
136.24
132.85
132.68
131.90
131.73
131.50
131.47
131.08
131.00
130.92
128.69
128.64
128.58
128.53
124.31
121.42
37.21
32.16
31.44
12.07
12.05
N
O
P
Ph
Ph
Me
Table 2, 3aa
13C NMR (101 MHz, CDCl3)
S25
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

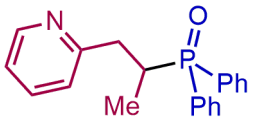
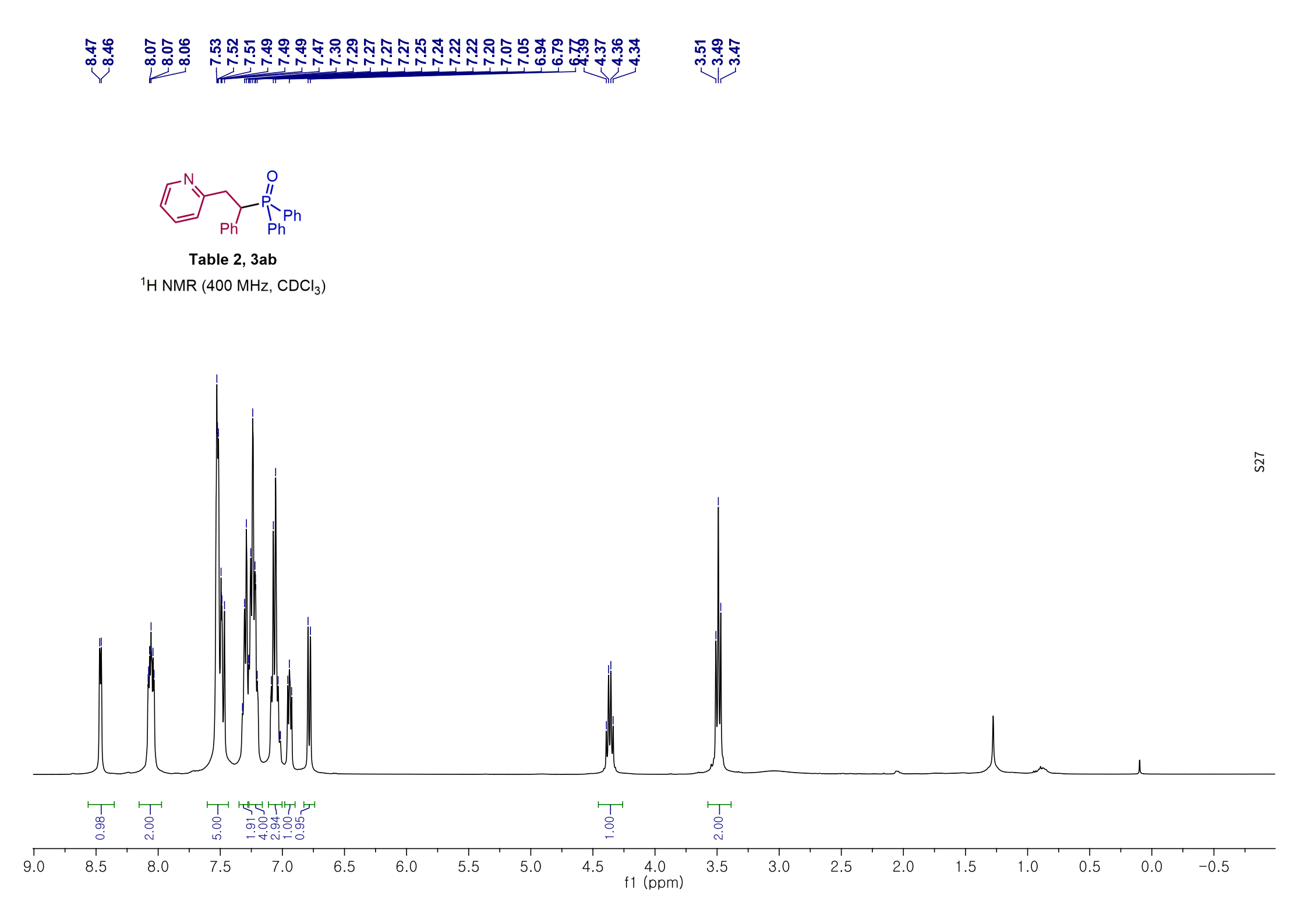
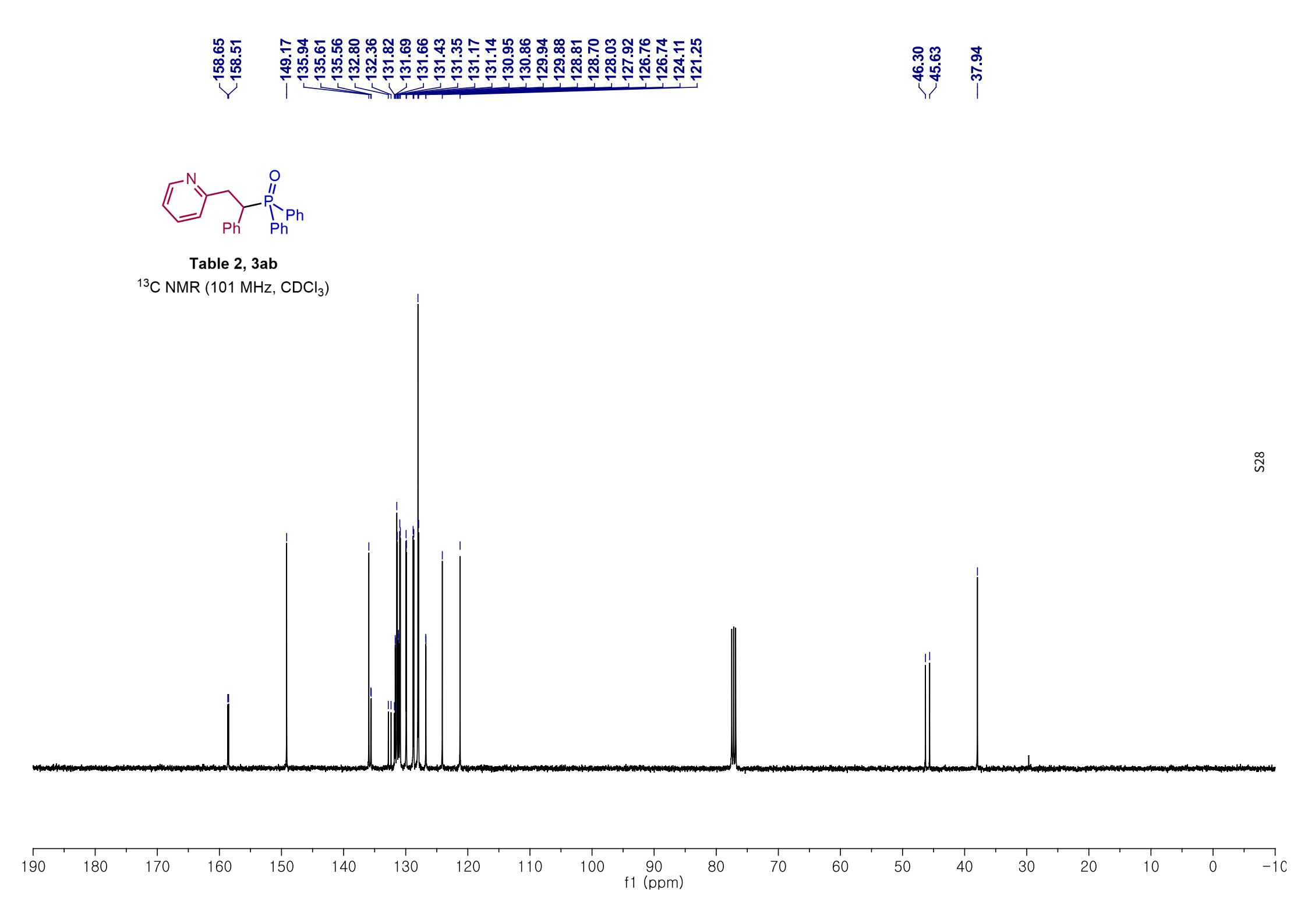


Table 2, 3aa

^{31}P NMR (162 MHz, $CDCl_3$)


8.47
8.46
8.07
8.07
8.06
7.53
7.52
7.51
7.49
7.49
7.49
7.47
7.30
7.29
7.27
7.27
7.27
7.25
7.24
7.22
7.22
7.20
7.07
7.05
6.94
6.79
6.77
4.39
4.37
4.36
4.34
3.51
3.49
3.47
N
O
P
Ph
Ph
Ph
Ph
Table 2, 3ab
1H NMR (400 MHz, CDCl3)
S27
0.98
2.00
5.00
1.91
4.00
2.94
1.00
0.95
1.00
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


158.65
158.51
149.17
135.94
135.61
135.56
132.80
132.36
131.82
131.69
131.66
131.43
131.35
131.17
131.14
130.95
130.86
129.94
129.88
128.81
128.70
128.03
127.92
126.76
126.74
124.11
121.25
46.30
45.63
37.94
N
O
P
Ph
Ph
Ph
Table 2, 3ab
13C NMR (101 MHz, CDCl3)
S28
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

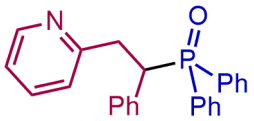


Table 2, 3ab

^{31}P NMR (162 MHz, $CDCl_3$)

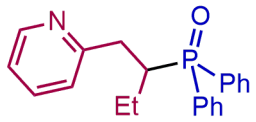


Table 2, 3ac

^{1}H NMR (300 MHz, $CDCl_3$)

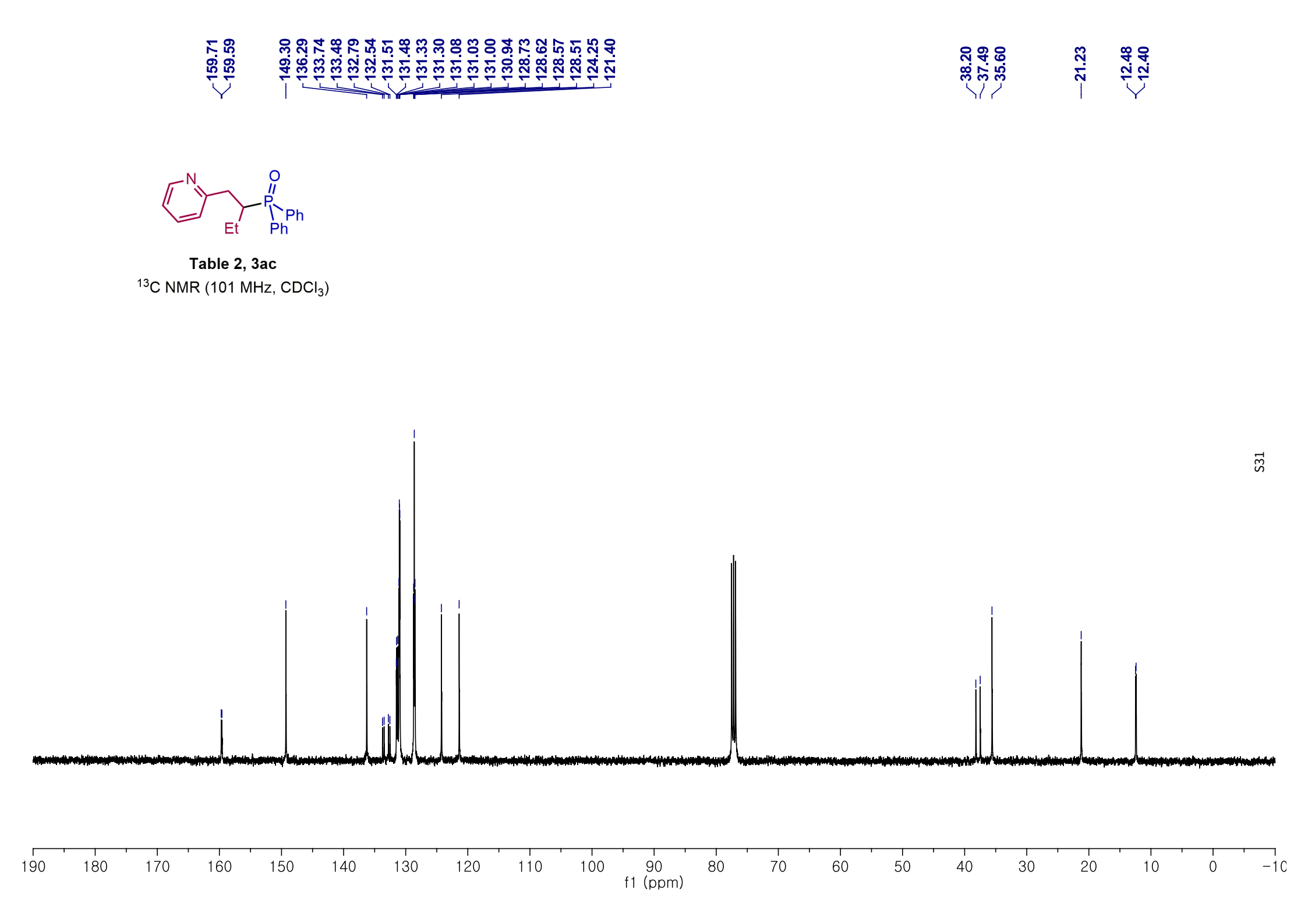


Table 2, 3ac

^{13}C NMR (101 MHz, $CDCl_3$)

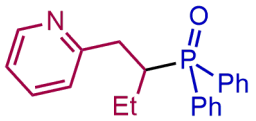


Table 2, 3ac

^{31}P NMR (162 MHz, $CDCl_3$)

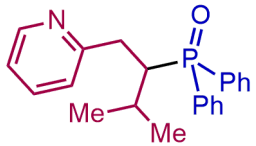
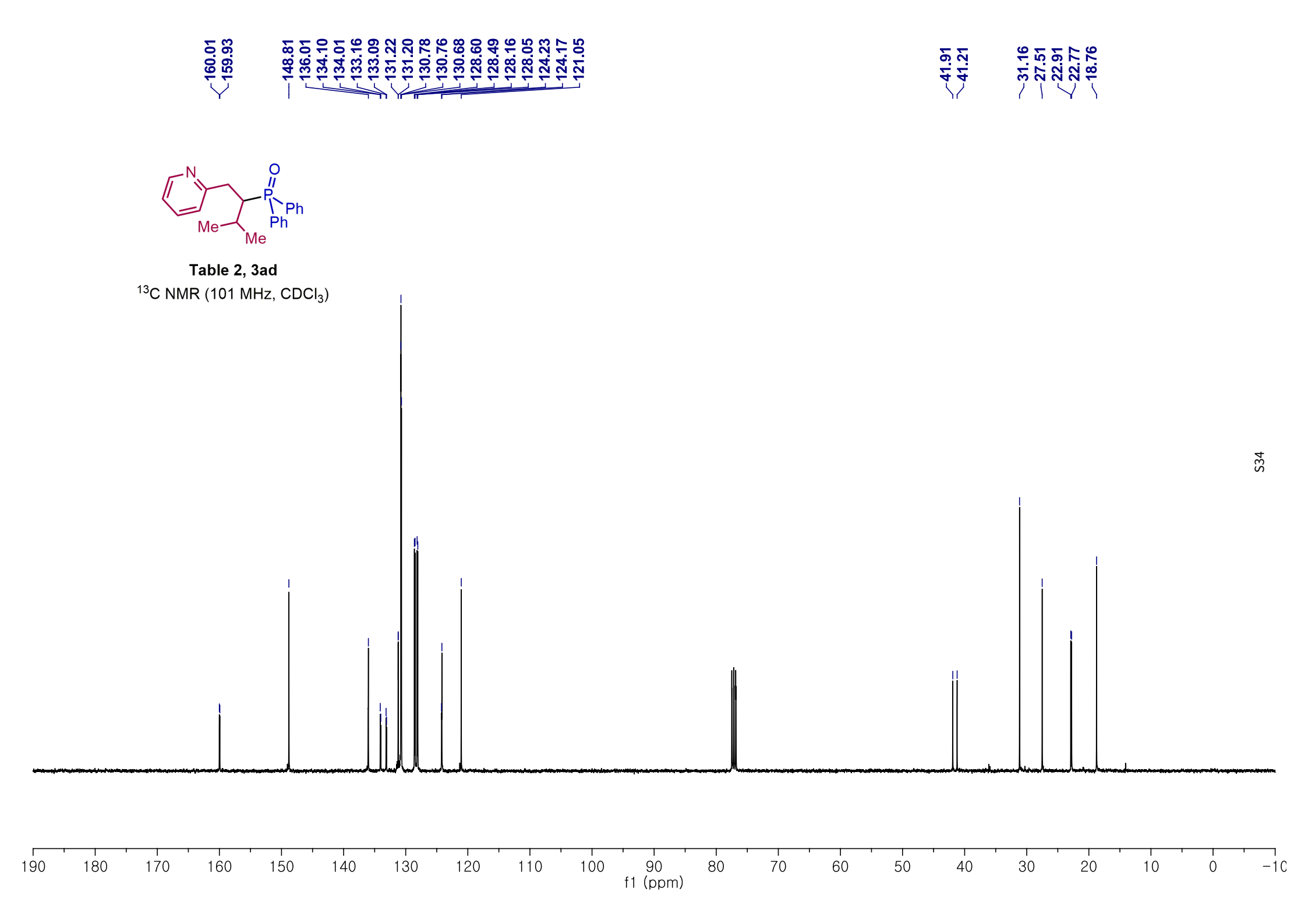


Table 2, 3ad

^{1}H NMR (400 MHz, $CDCl_3$)


160.01
159.93
148.81
136.01
134.10
134.01
133.16
133.09
131.22
131.20
130.78
130.76
130.68
128.60
128.49
128.16
128.05
124.23
124.17
121.05
41.91
41.21
31.16
27.51
22.91
22.77
18.76
N
O
P
Ph
Ph
Me
Me
Table 2, 3ad
13C NMR (101 MHz, CDCl3)
S34
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

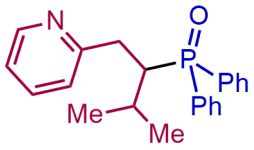
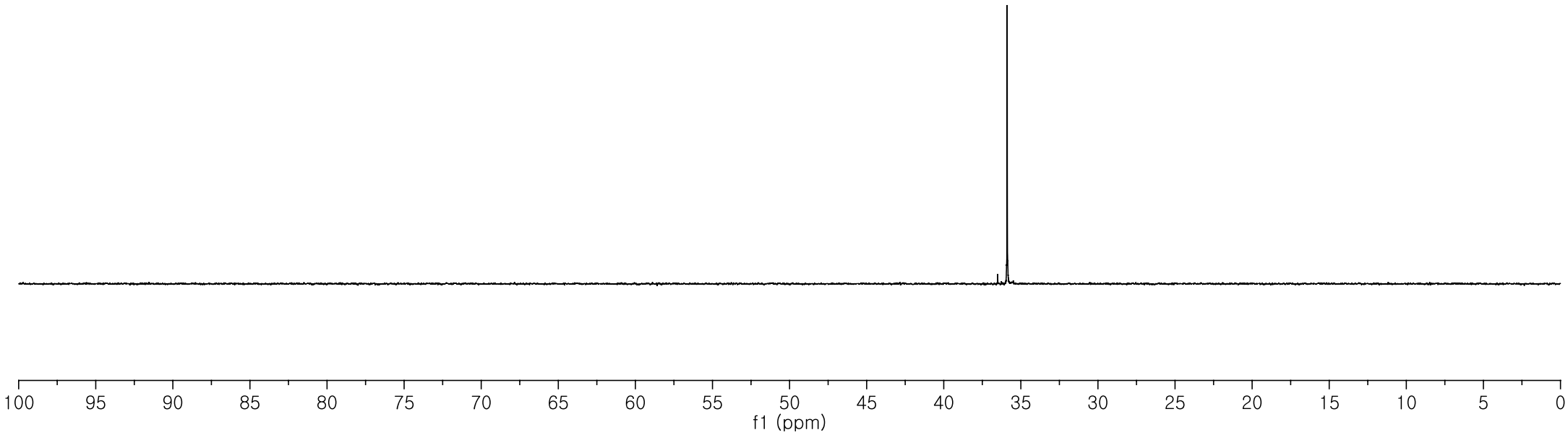


Table 2, 3ad

^{31}P NMR (162 MHz, $CDCl_3$)



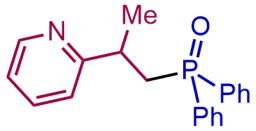


Table 2, 3ae

^{1}H NMR (400 MHz, $CDCl_3$)

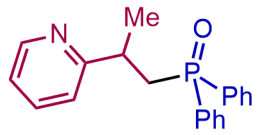


Table 2, 3ae

^{13}C NMR (101 MHz, $CDCl_3$)

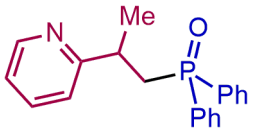


Table 2, 3ae

^{31}P NMR (162 MHz, $CDCl_3$)

S38

100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0

f1 (ppm)

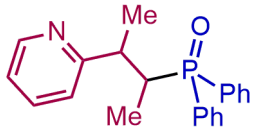
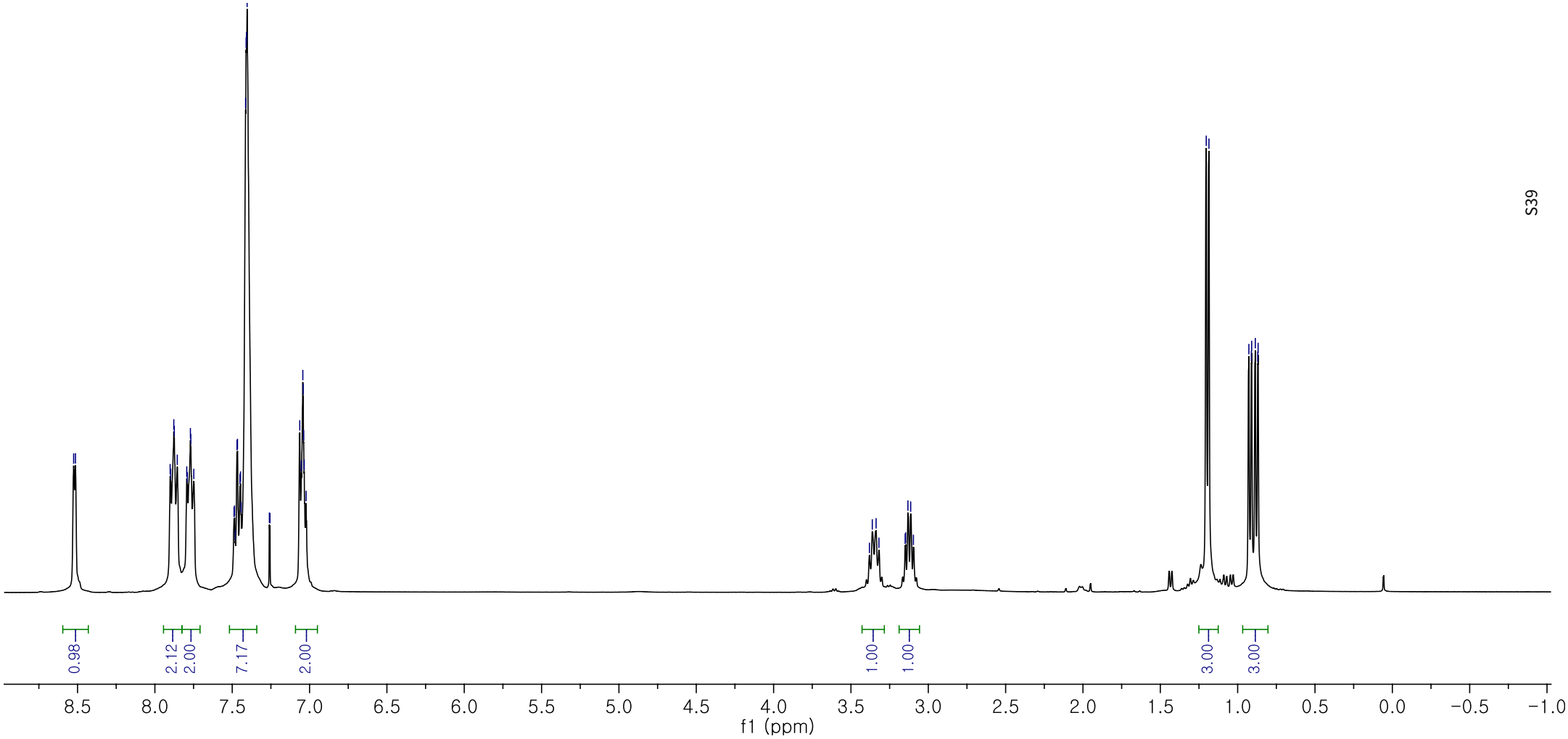


Table 2, 3af

^{1}H NMR (400 MHz, $CDCl_3$)



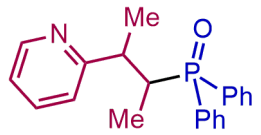
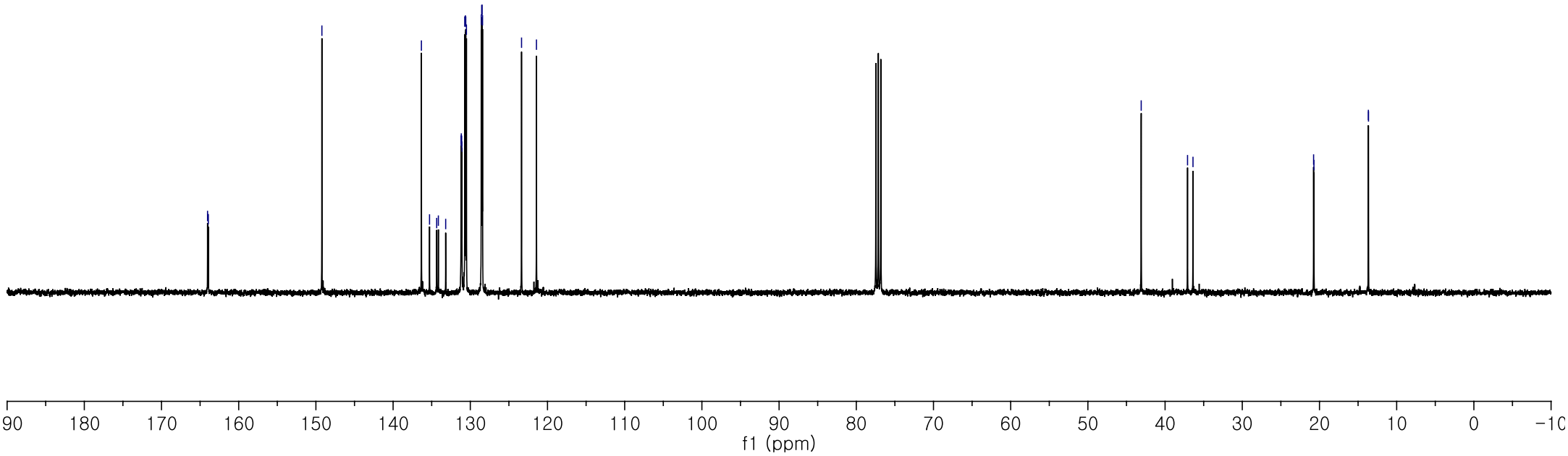


Table 2, 3af

^{13}C NMR (101 MHz, $CDCl_3$)



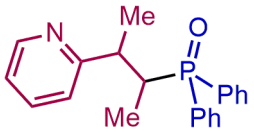
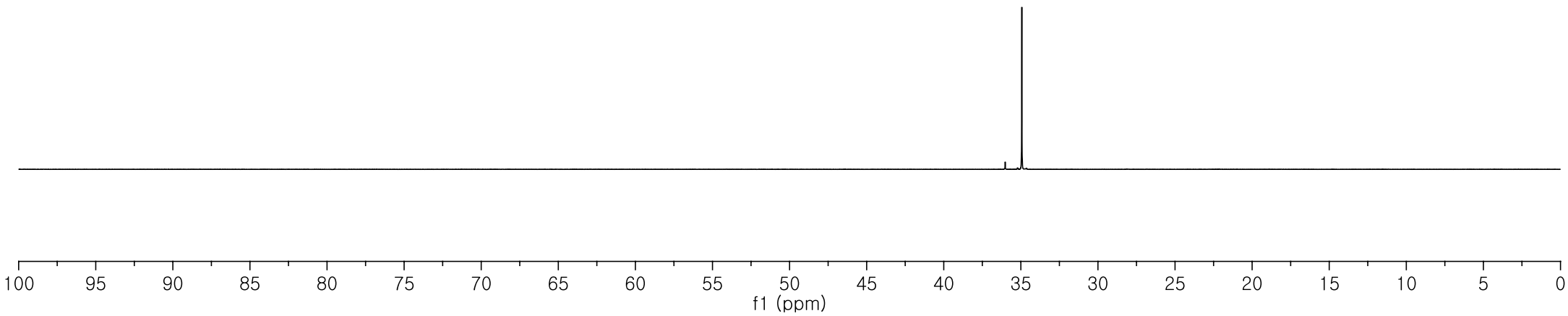
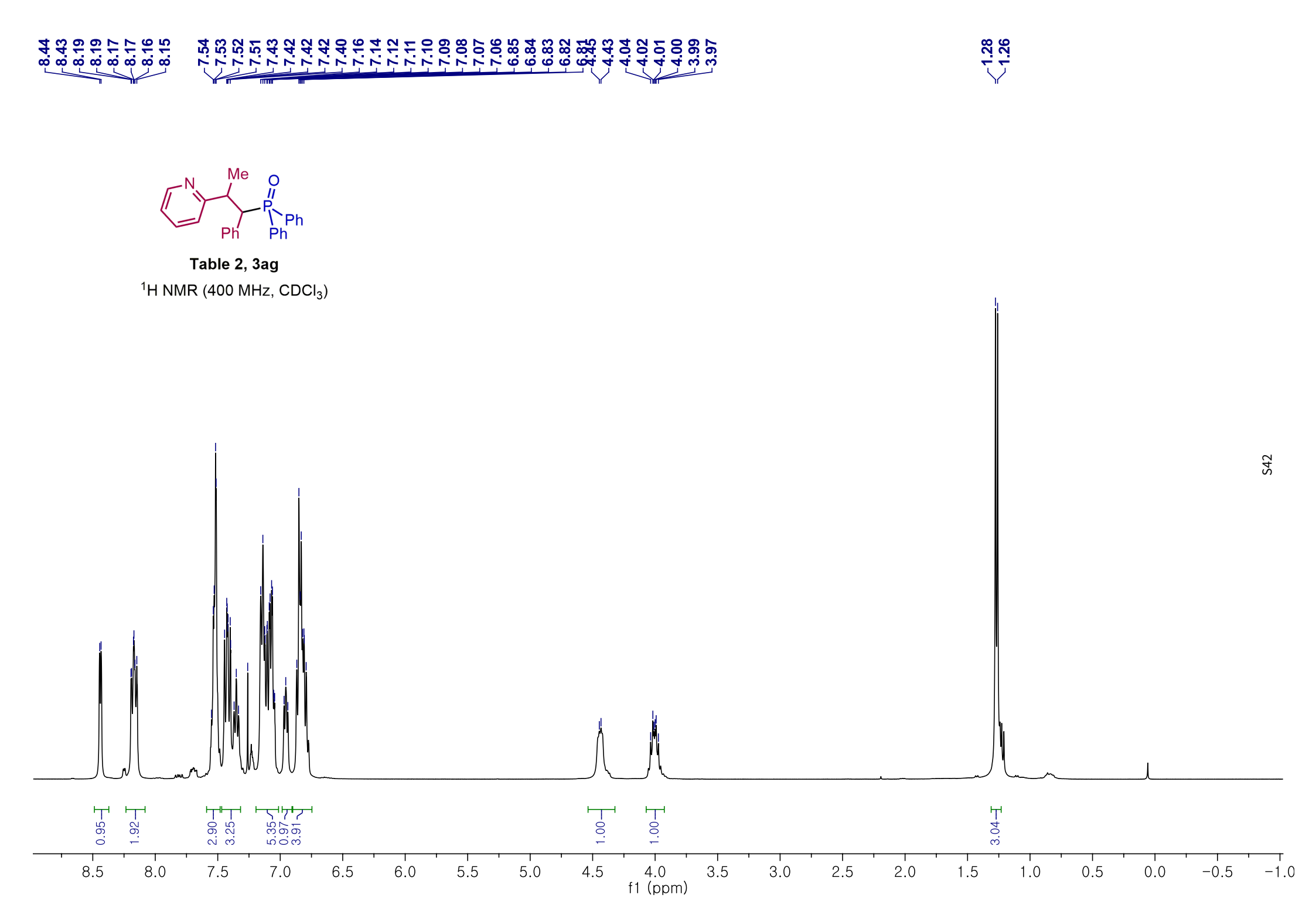
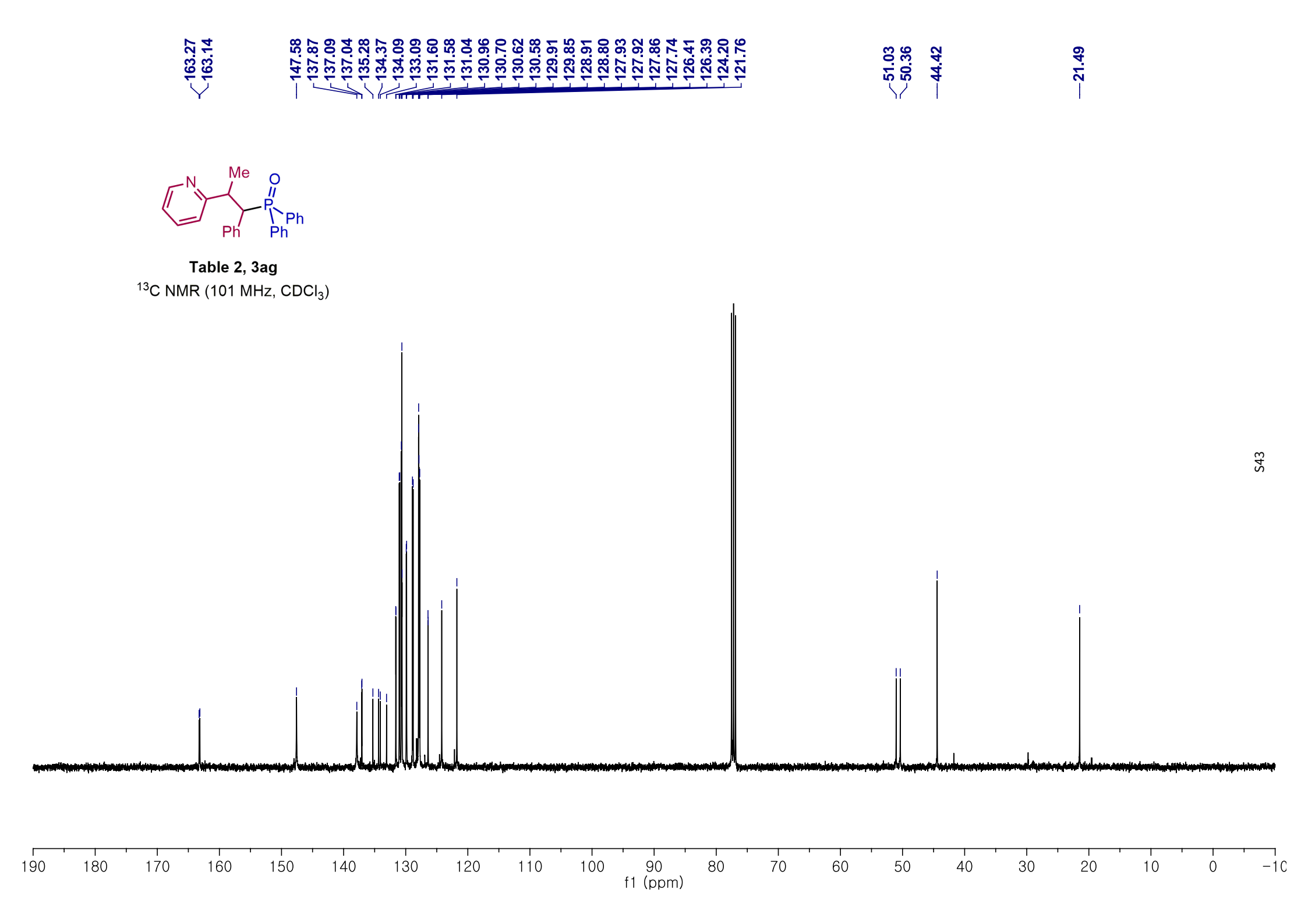


Table 2, 3af

^{31}P NMR (162 MHz, $CDCl_3$)




8.44
8.43
8.19
8.19
8.17
8.17
8.16
8.15
7.54
7.53
7.52
7.51
7.43
7.42
7.42
7.42
7.40
7.16
7.14
7.12
7.11
7.10
7.09
7.08
7.07
7.06
6.85
6.84
6.83
6.82
6.81
6.80
4.43
4.04
4.02
4.01
4.00
3.99
3.97
1.28
1.26
N
Me
O
P
Ph
Ph
Ph
Table 2, 3ag
1H NMR (400 MHz, CDCl3)
S42
0.95
1.92
2.90
3.25
5.35
0.97
3.91
1.00
1.00
3.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)


163.27
163.14
147.58
137.87
137.09
137.04
135.28
134.37
134.09
133.09
131.60
131.58
131.04
130.96
130.70
130.62
130.58
129.91
129.85
128.91
128.80
127.93
127.92
127.86
127.74
126.41
126.39
124.20
121.76
51.03
50.36
44.42
21.49
N
Me
O
P
Ph
Ph
Ph
Ph
Table 2, 3ag
13C NMR (101 MHz, CDCl3)
S43
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

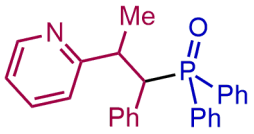
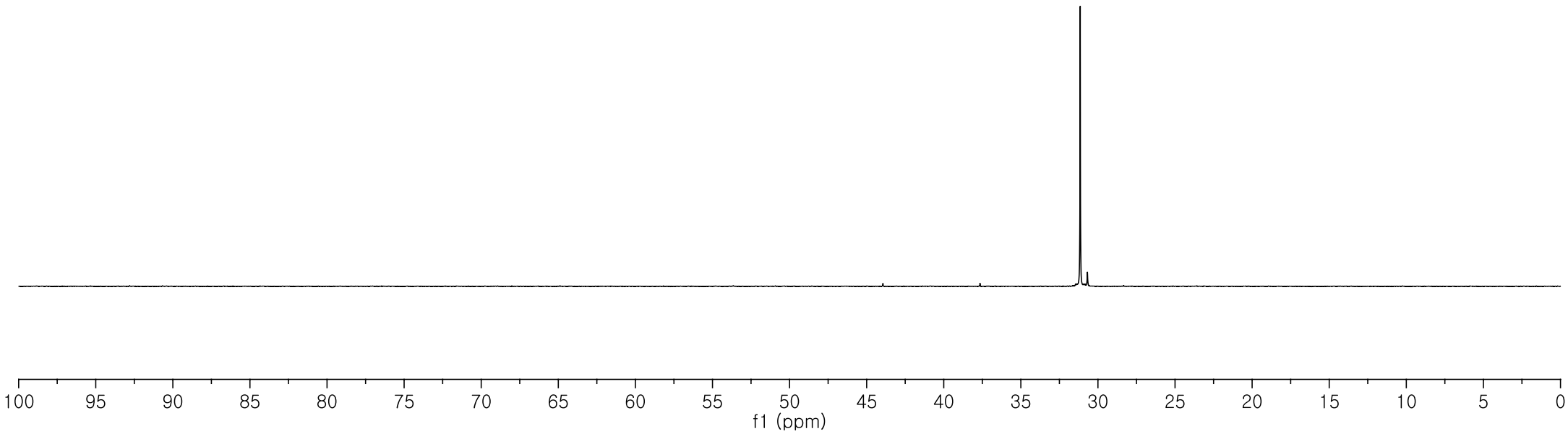
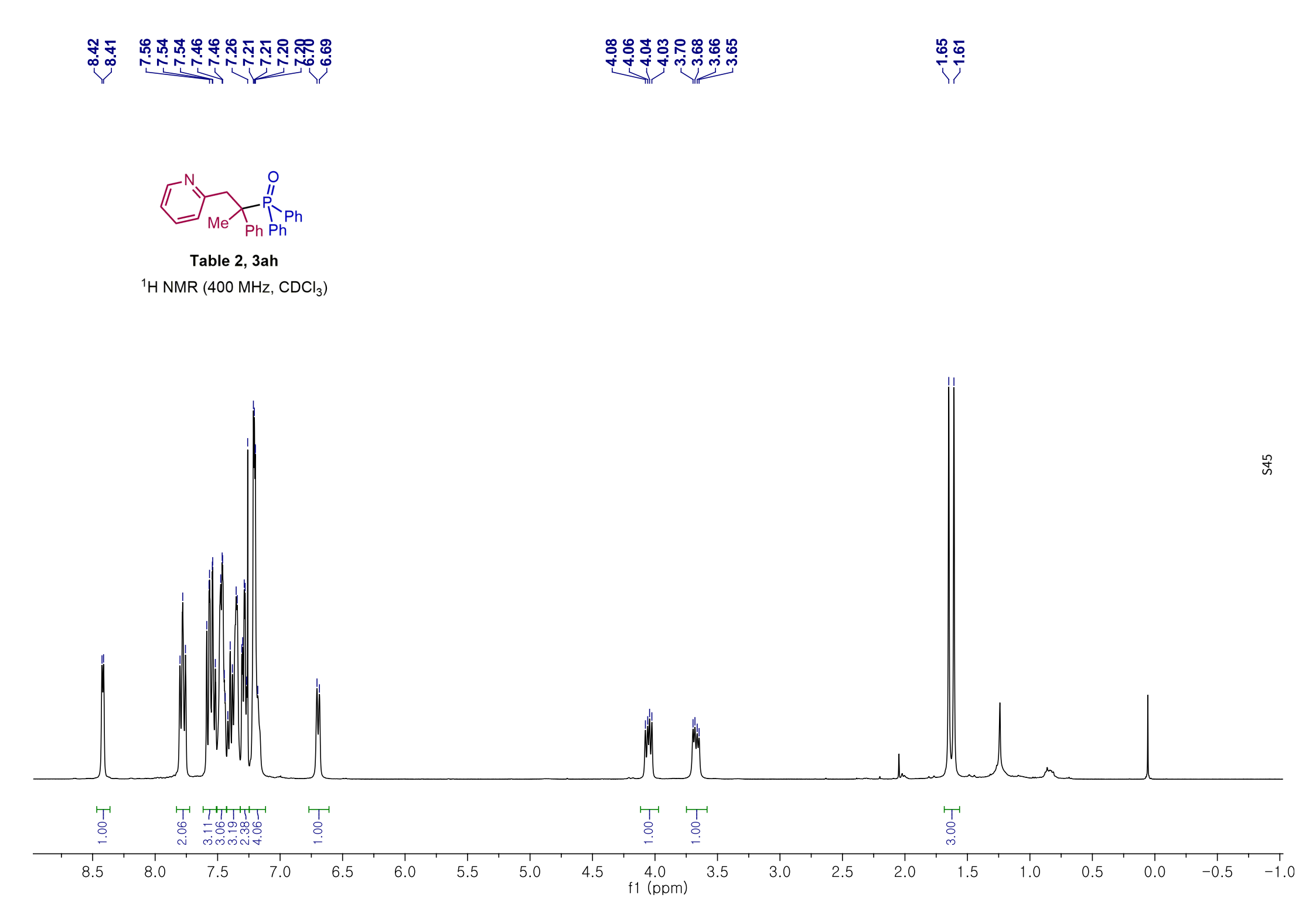
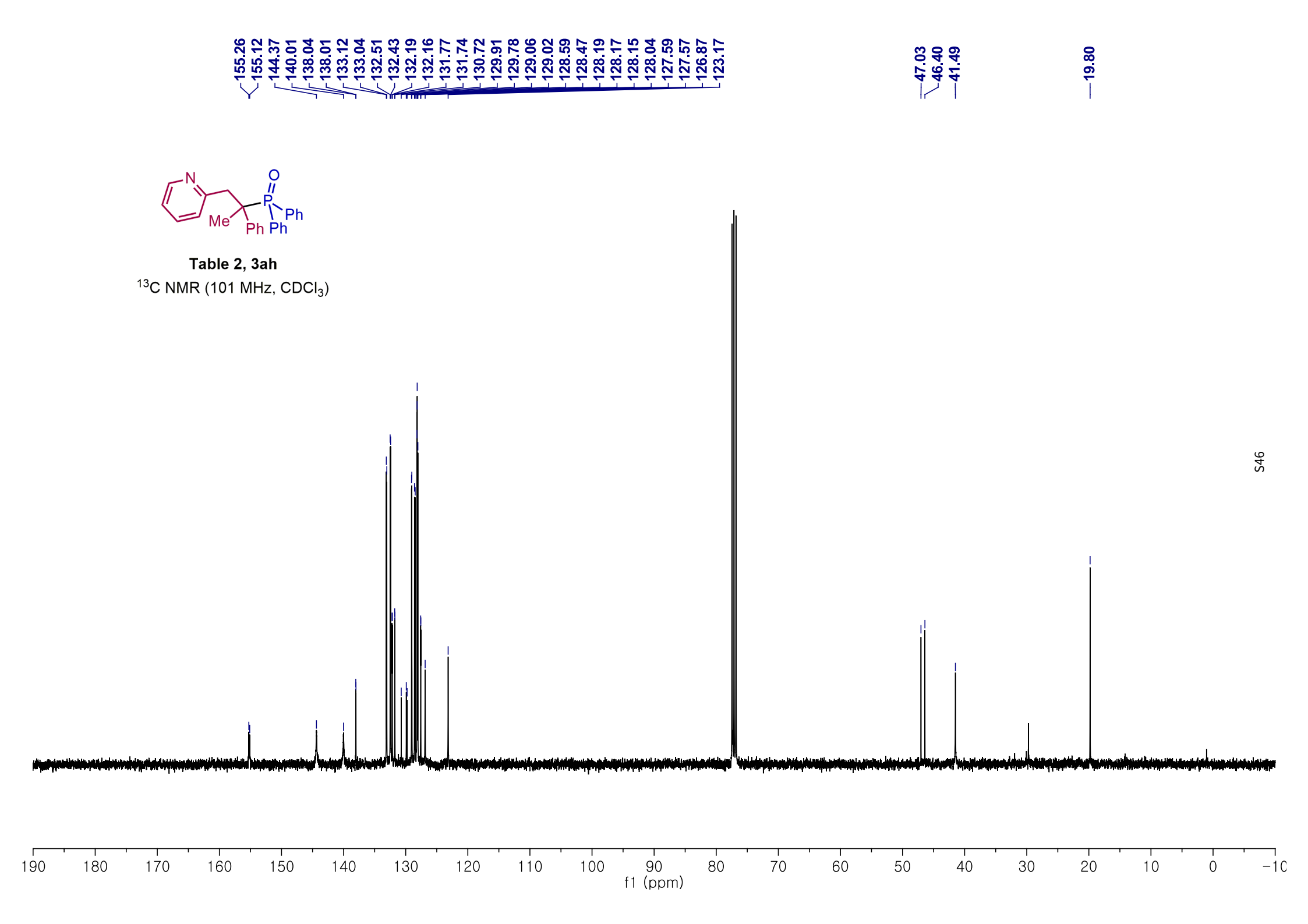


Table 2, 3ag

^{31}P NMR (162 MHz, $CDCl_3$)




8.42
8.41
7.56
7.54
7.54
7.46
7.46
7.26
7.21
7.21
7.20
7.20
6.70
6.69
4.08
4.06
4.04
4.03
3.70
3.68
3.66
3.65
1.65
1.61
N
O
P
Ph
Ph
Me
Ph
Table 2, 3ah
1H NMR (400 MHz, CDCl3)
S45
1.00
2.06
3.11
3.06
3.19
2.38
4.06
1.00
1.00
1.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)


155.26
155.12
144.37
140.01
138.04
138.01
133.12
133.04
132.51
132.43
132.19
132.16
131.77
131.74
130.72
129.91
129.78
129.06
129.02
128.59
128.47
128.19
128.17
128.15
128.04
127.59
127.57
126.87
123.17
47.03
46.40
41.49
19.80
N
O
P
Ph
Ph
Me
Ph
Table 2, 3ah
13C NMR (101 MHz, CDCl3)
S46
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

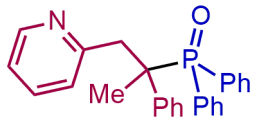
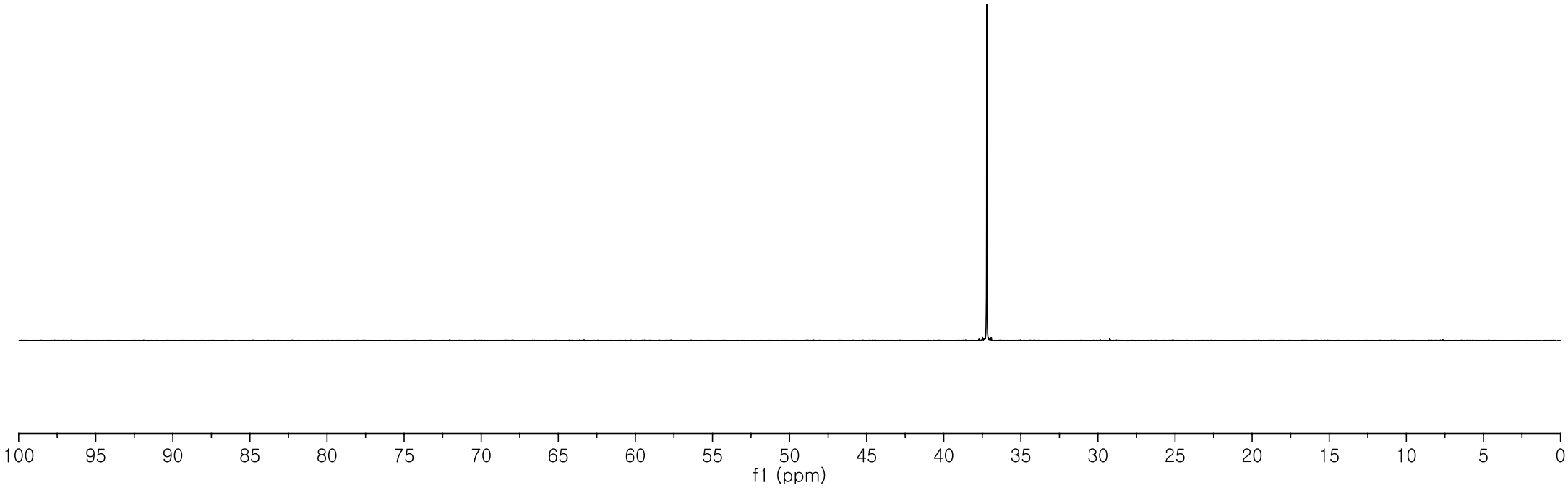


Table 2, 3ah

^{31}P NMR (162 MHz, $CDCl_3$)



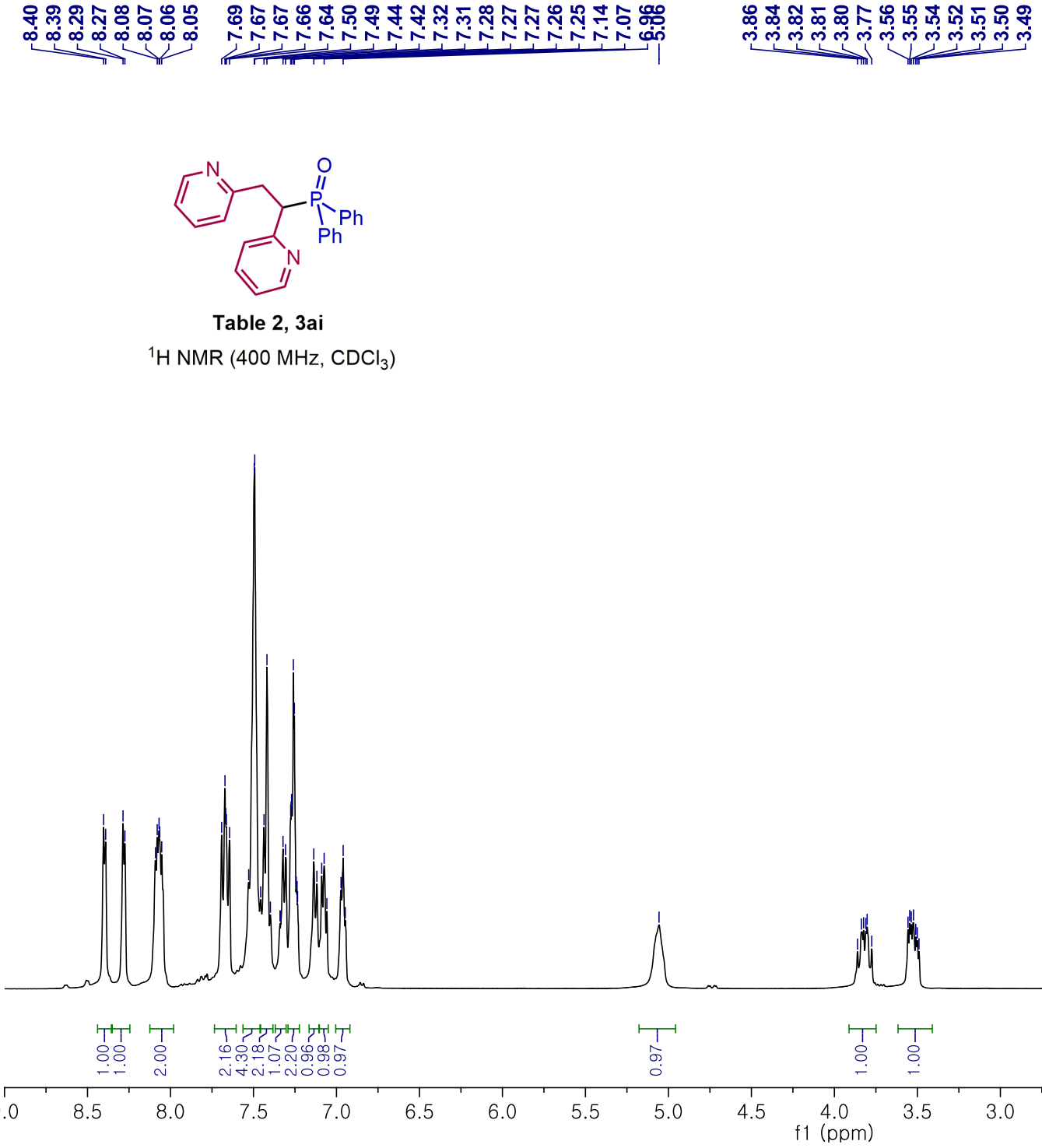


Table 2, 3ai

^{1}H NMR (400 MHz, $CDCl_3$)

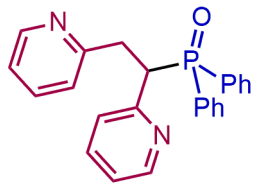


Table 2, 3ai

^{13}C NMR (101 MHz, $CDCl_3$)

157.49
157.36
155.36
155.31
148.18
147.16
138.15
136.84
131.95
131.92
131.87
131.55
131.53
131.46
131.38
131.21
131.12
130.93
130.91
128.91
128.79
128.26
128.14
125.11
125.07
125.01
122.06
122.03
122.01
48.11
47.48
35.83

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)

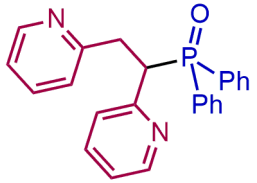
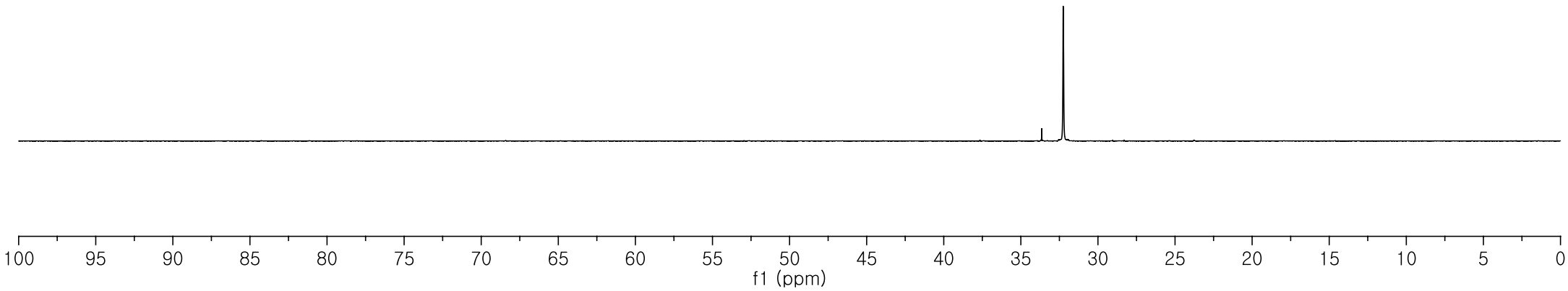


Table 2, 3ai

^{31}P NMR (162 MHz, $CDCl_3$)



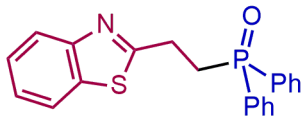
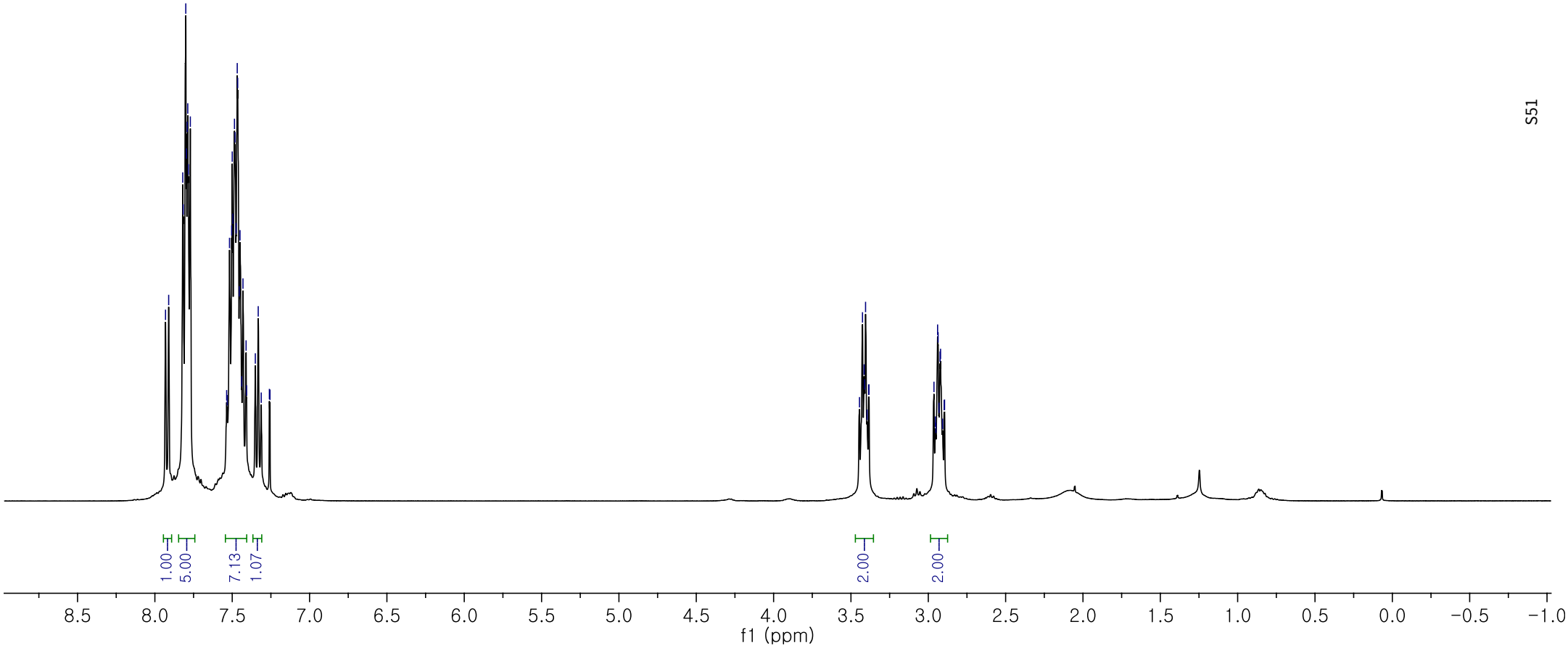
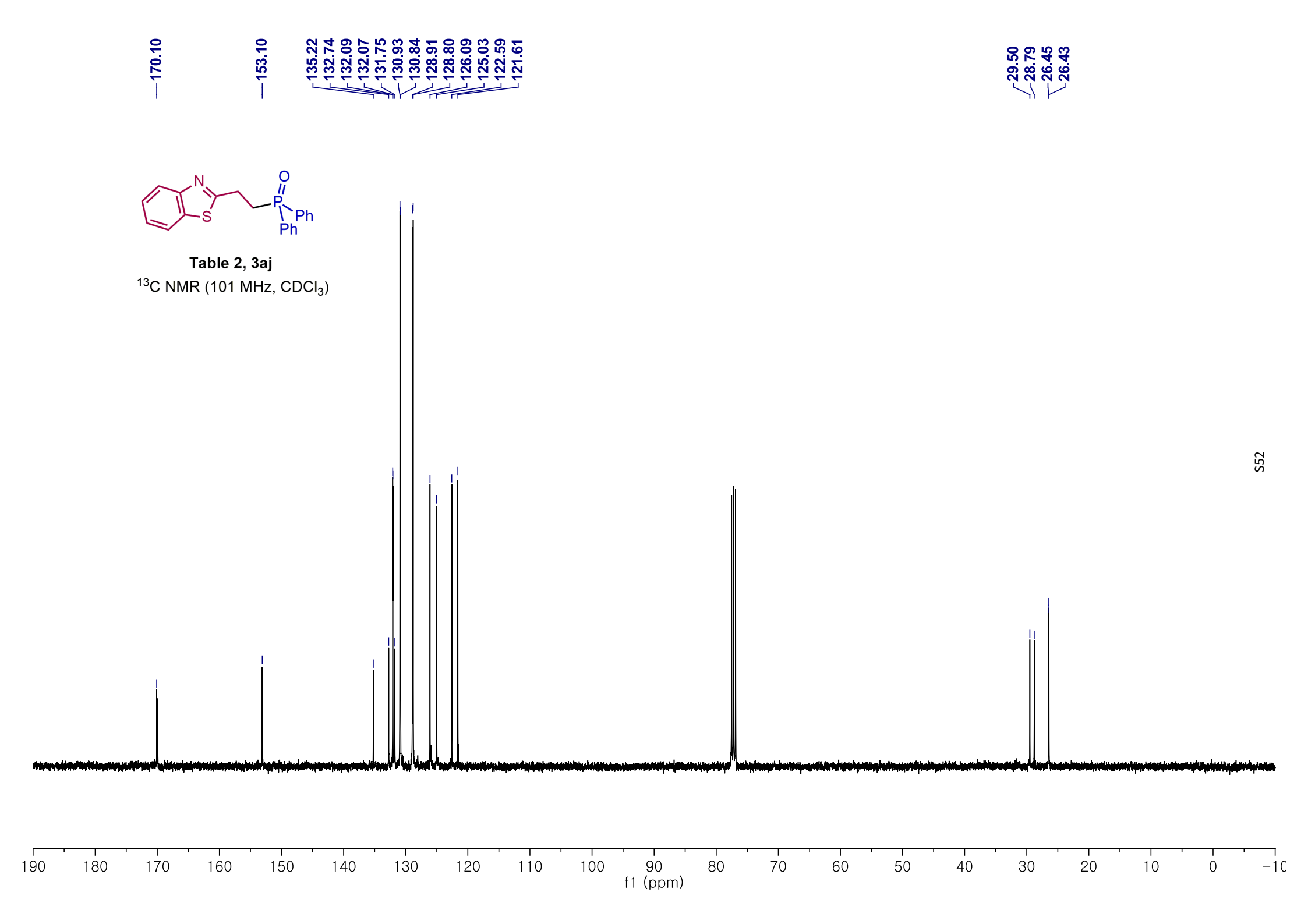


Table 2, 3aj

^{1}H NMR (400 MHz, $CDCl_3$)




170.10
153.10
135.22
132.74
132.09
132.07
131.75
130.93
130.84
128.91
128.80
126.09
125.03
122.59
121.61
29.50
28.79
26.45
26.43
N
S
O
P
Ph
Ph
Table 2, 3aj
13C NMR (101 MHz, CDCl3)
S52
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

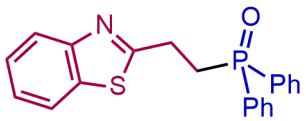
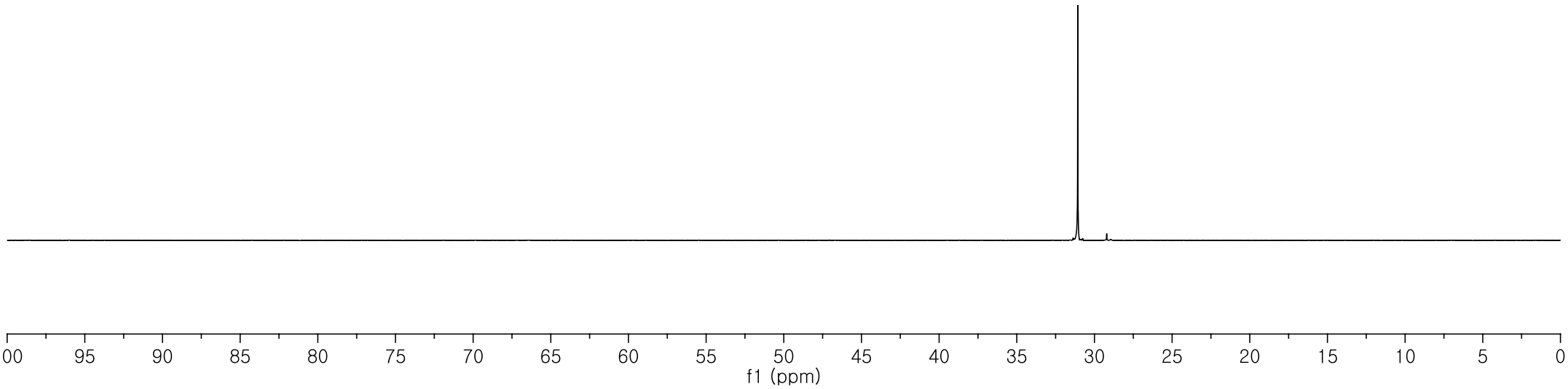


Table 2, 3aj

^{31}P NMR (162 MHz, $CDCl_3$)



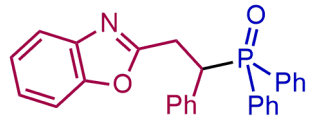
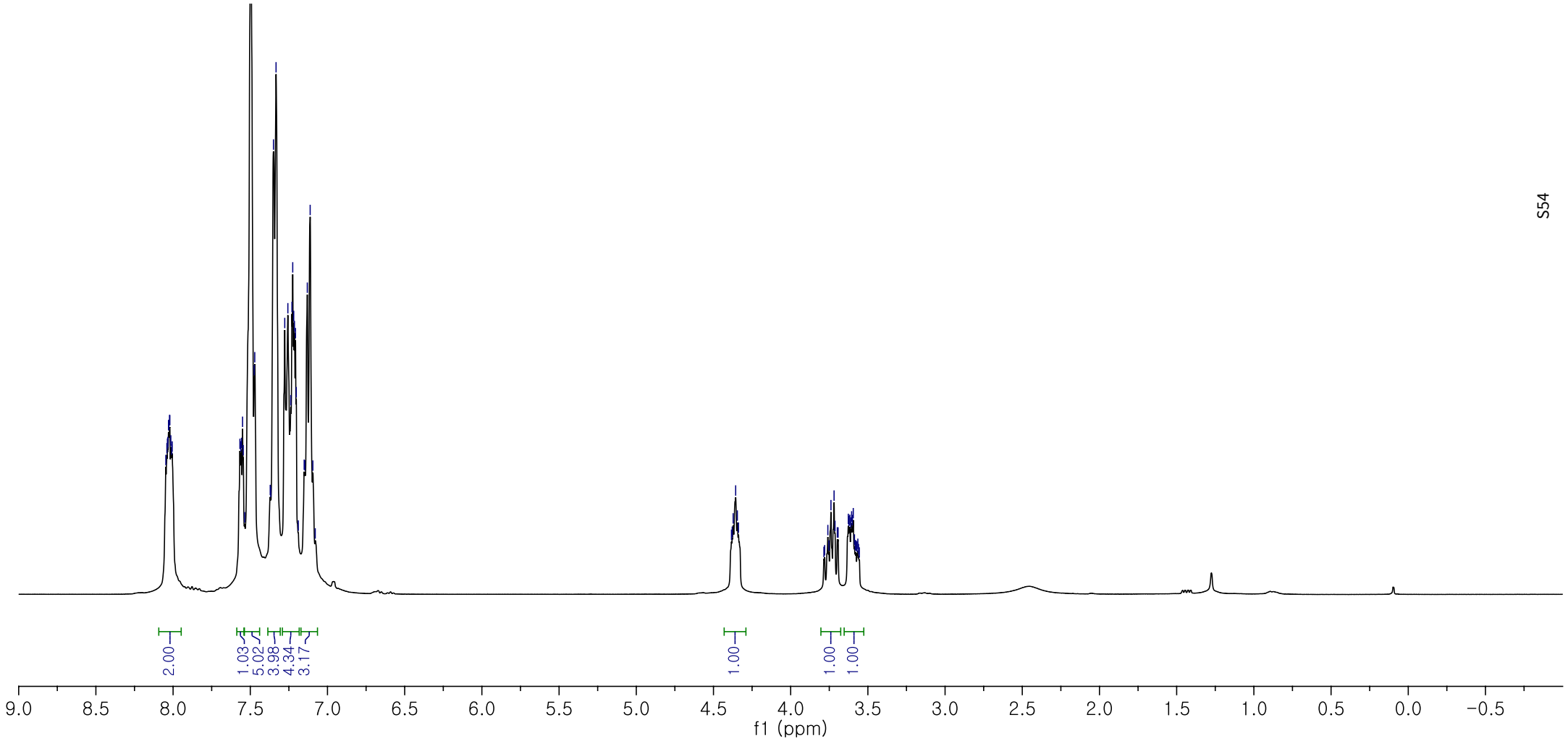


Table 2, 3ak

^{1}H NMR (400 MHz, $CDCl_3$)



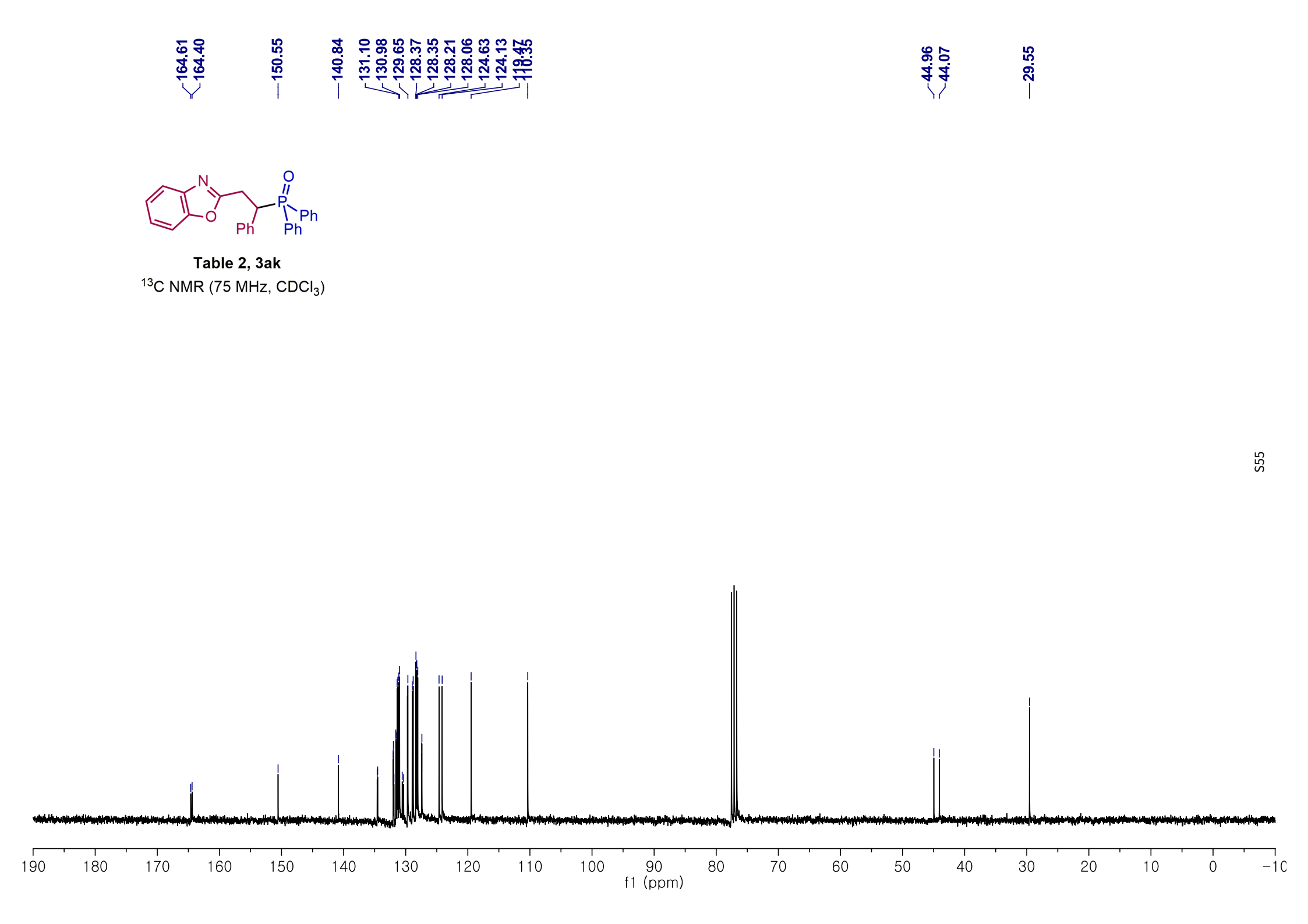


Table 2, 3ak

^{13}C NMR (75 MHz, $CDCl_3$)

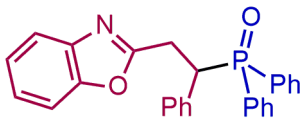
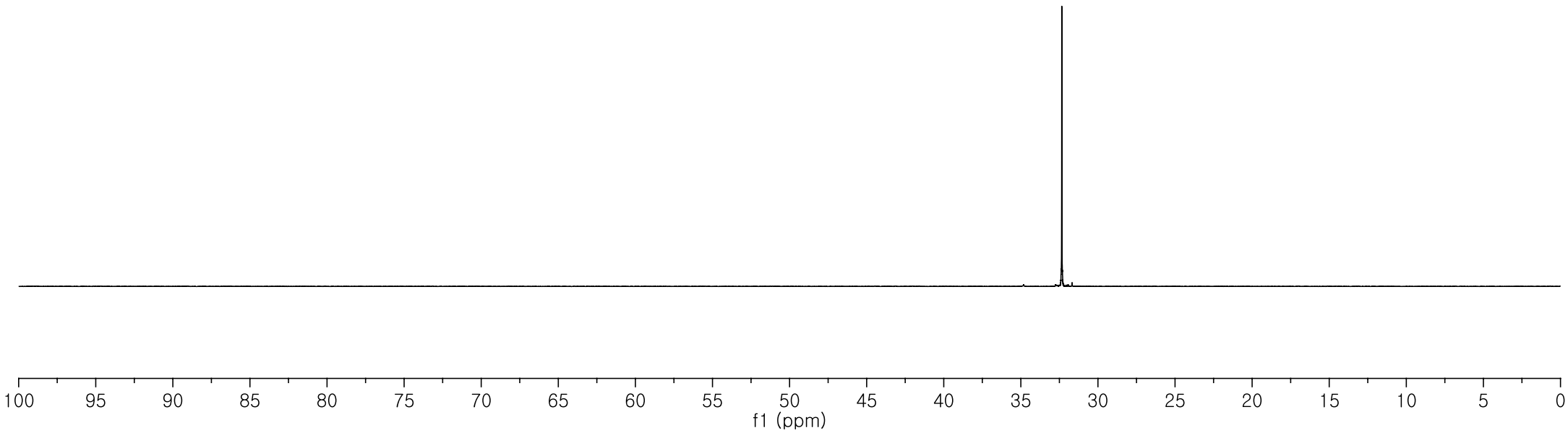


Table 2, 3ak

^{31}P NMR (122 MHz, $CDCl_3$)



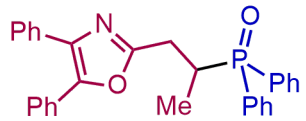
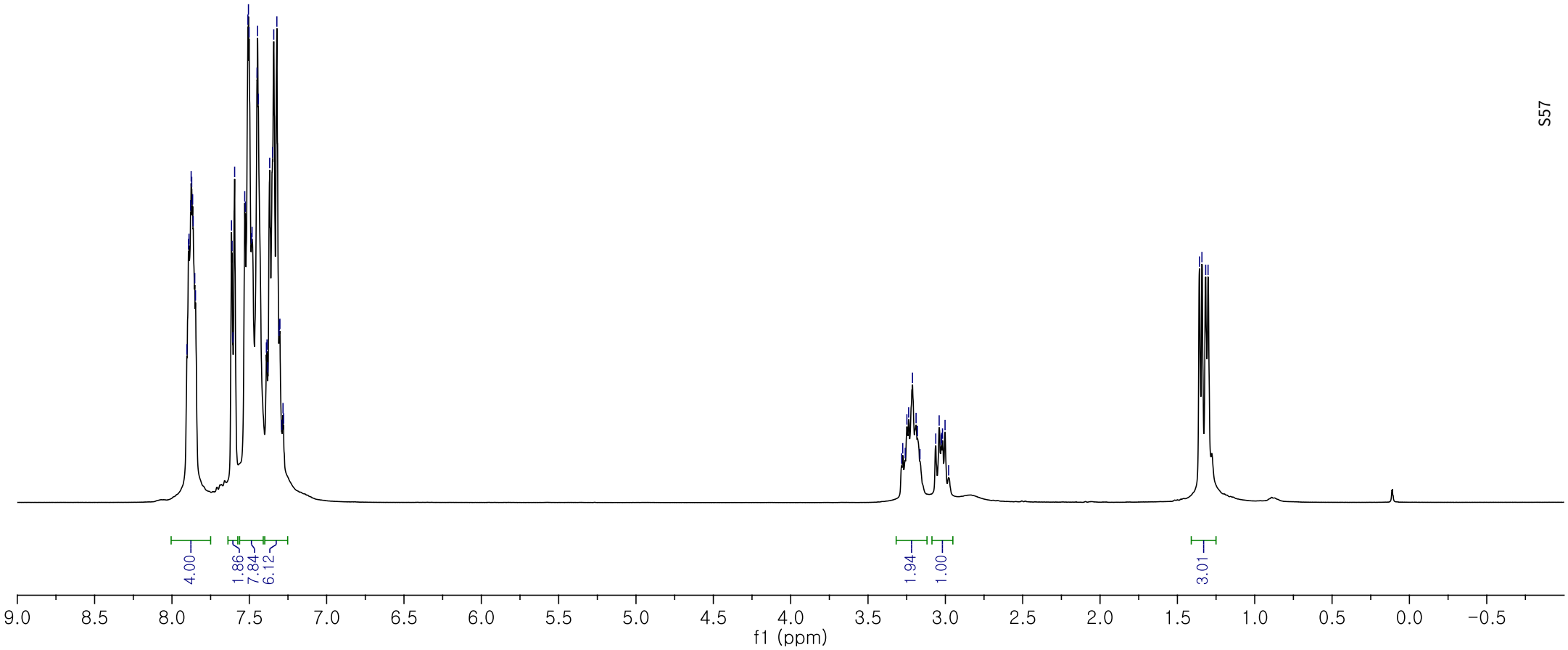


Table 2, 3al

^{1}H NMR (400 MHz, $CDCl_3$)



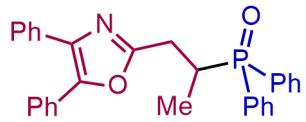


Table 2, 3al

^{13}C NMR (75 MHz, $CDCl_3$)

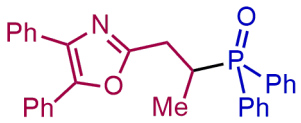
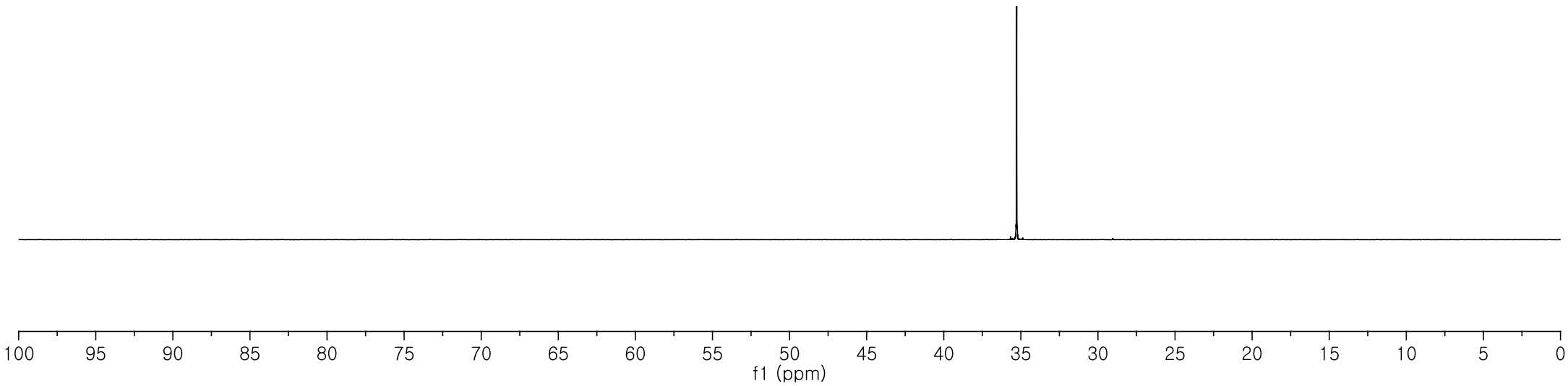


Table 2, 3al

^{31}P NMR (122 MHz, $CDCl_3$)



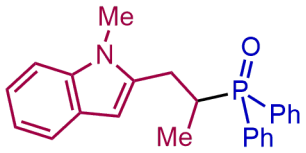


Table 2, 3am

^{1}H NMR (400 MHz, $CDCl_3$)

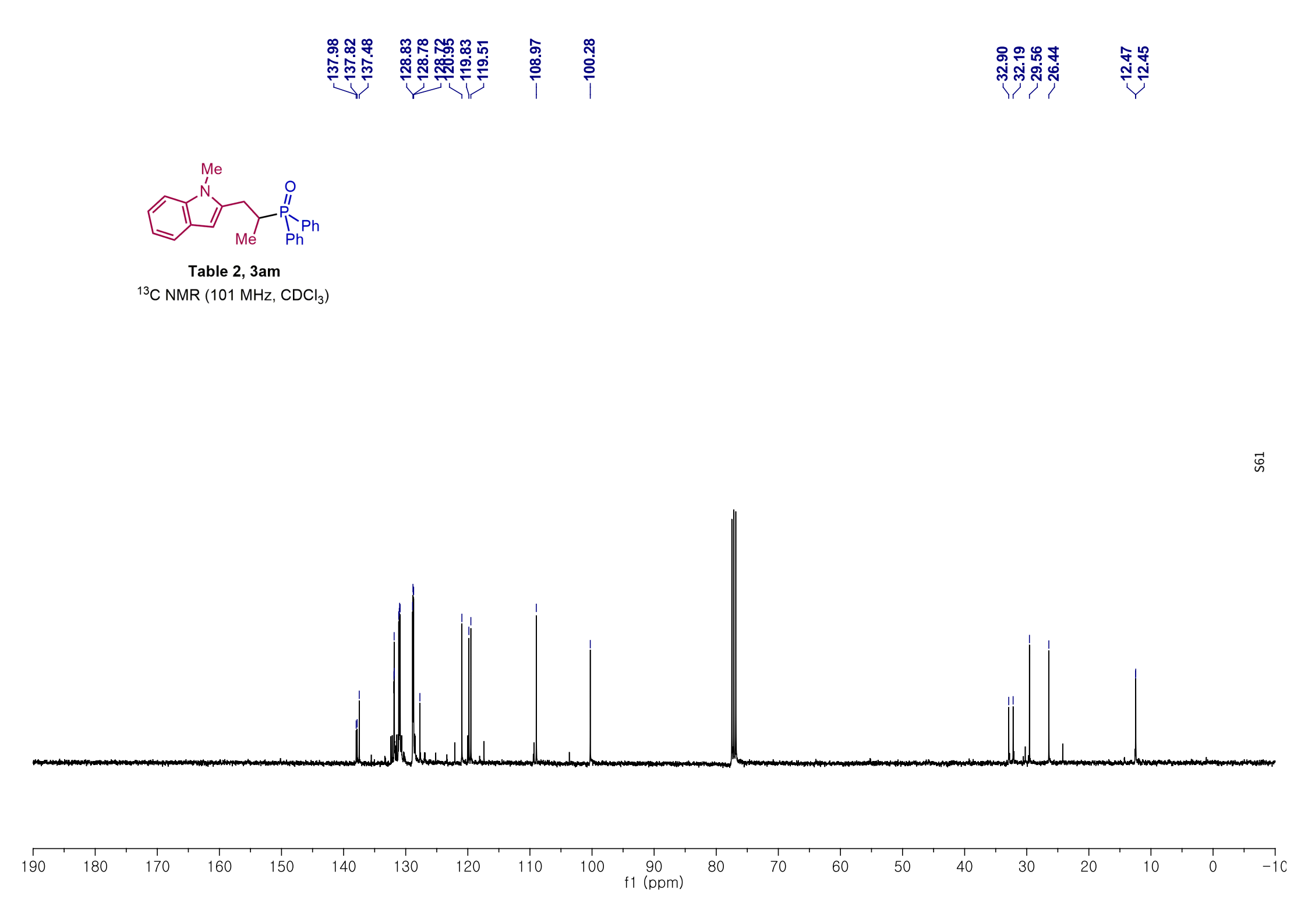


Table 2, 3am

^{13}C NMR (101 MHz, $CDCl_3$)

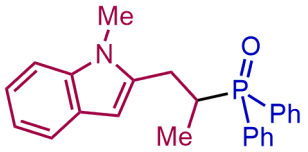
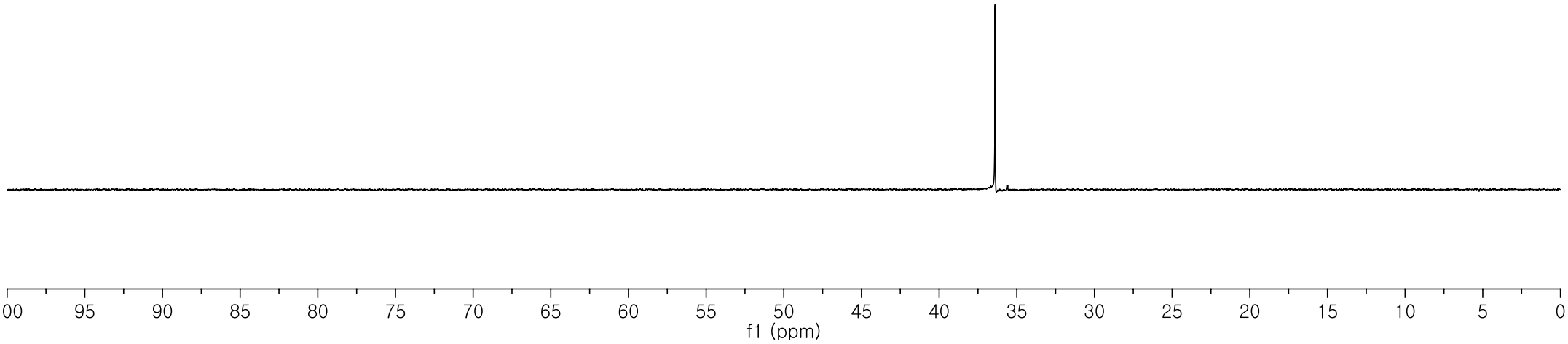


Table 2, 3am

^{31}P NMR (162 MHz, $CDCl_3$)



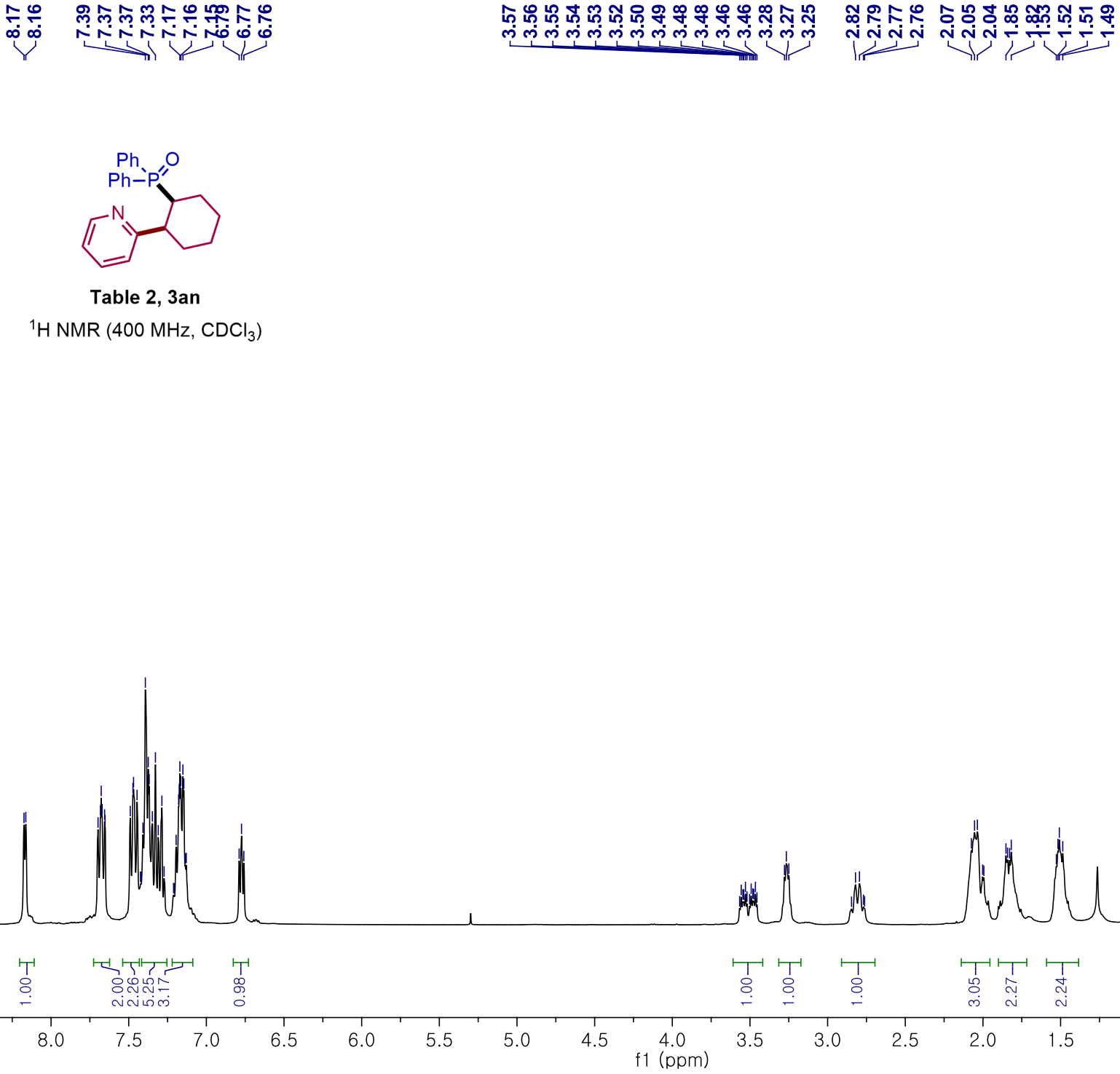


Table 2, 3an

^{1}H NMR (400 MHz, $CDCl_3$)

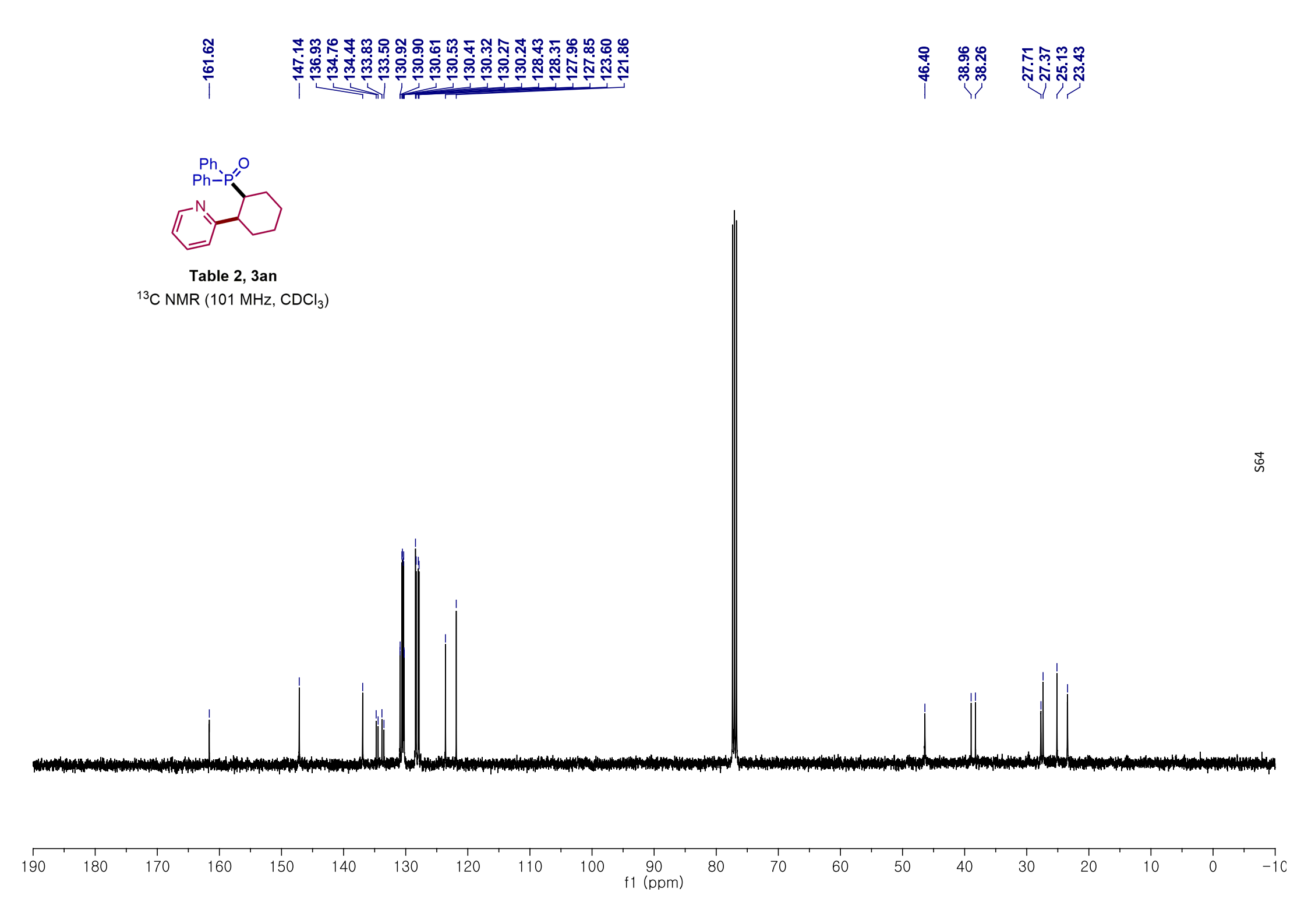


Table 2, 3an

^{13}C NMR (101 MHz, $CDCl_3$)

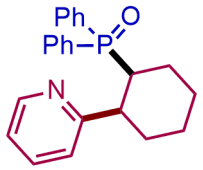
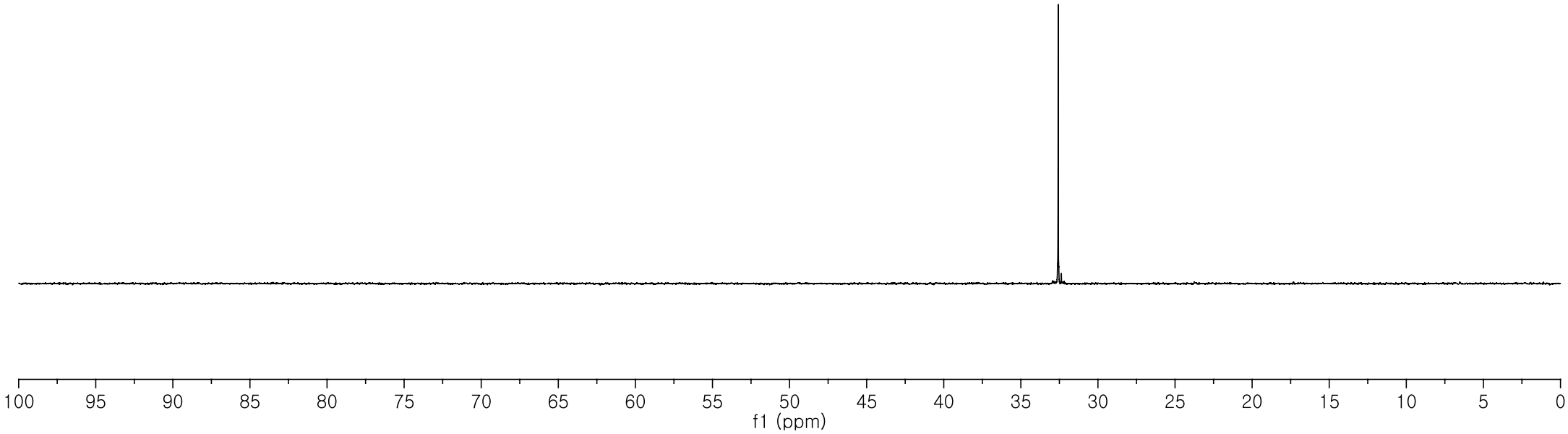
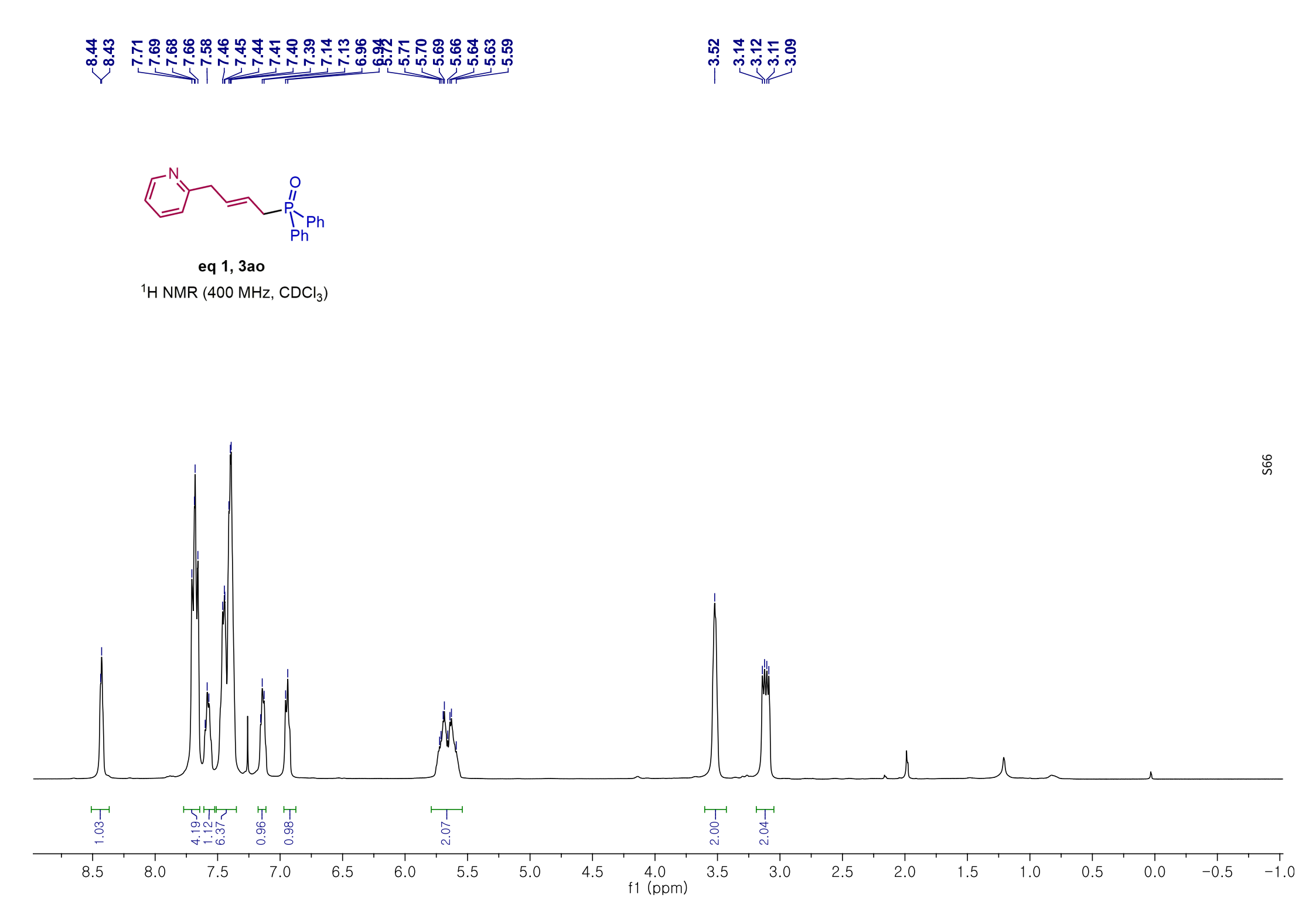


Table 2, 3an

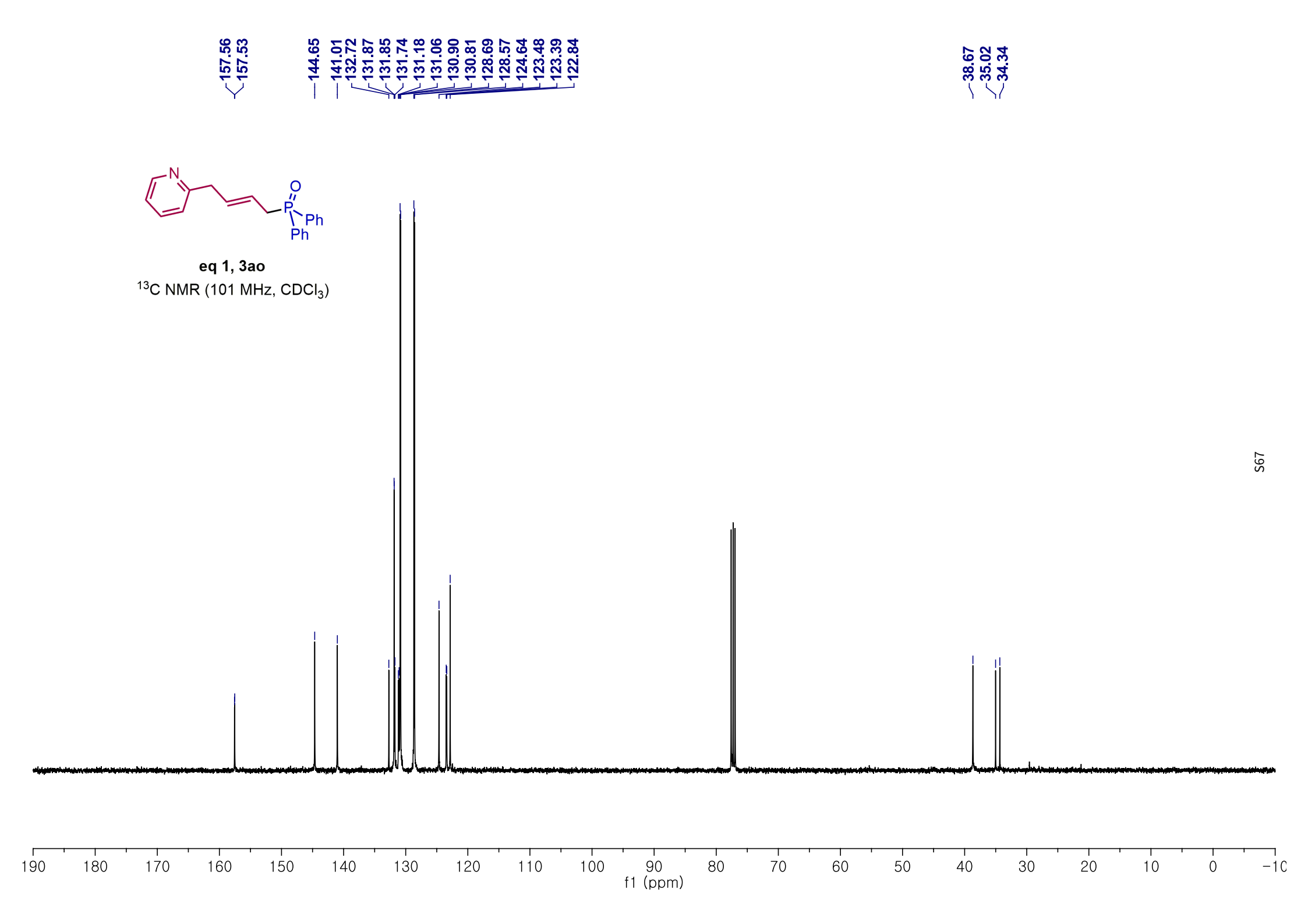
^{31}P NMR (162 MHz, $CDCl_3$)

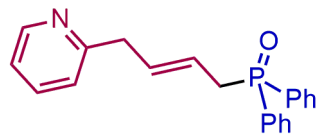




eq 1, 3ao

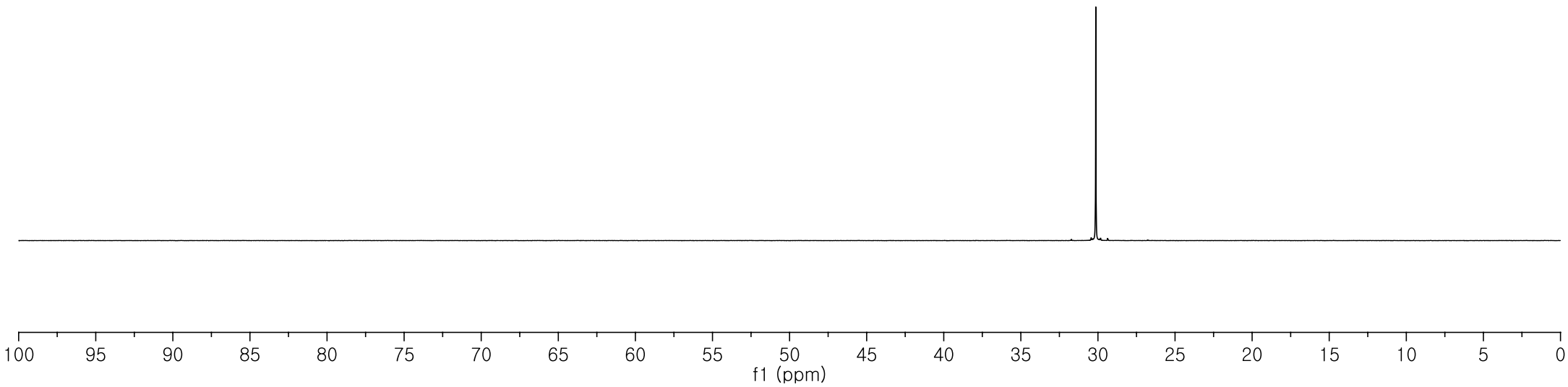
^{1}H NMR (400 MHz, $CDCl_3$)


157.56
157.53
144.65
141.01
132.72
131.87
131.85
131.74
131.18
131.06
130.90
130.81
128.69
128.57
124.64
123.48
123.39
122.84
38.67
35.02
34.34
N
O
P
Ph
Ph
eq 1, 3ao
13C NMR (101 MHz, CDCl3)
S67
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)



eq 1, 3ao

^{31}P NMR (162 MHz, $CDCl_3$)



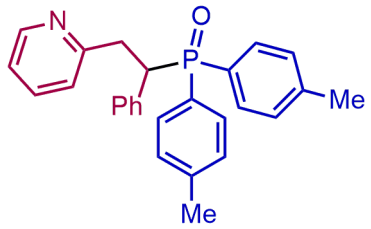


Table 3, 3bb

^{1}H NMR (400 MHz, $CDCl_3$)

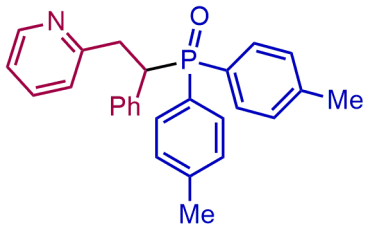


Table 3, 3bb

^{13}C NMR (101 MHz, $CDCl_3$)

158.93
158.79
149.16
142.01
141.98
141.41
135.89
135.85
135.79
131.45
131.36
131.00
130.91
130.00
129.94
129.70
129.55
129.44
129.22
128.80
128.69
128.26
128.01
127.99
126.66
126.64
124.13
121.14
46.54
45.87
38.02
21.57
21.44

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)

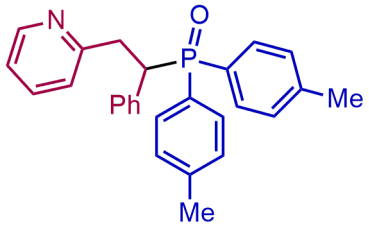
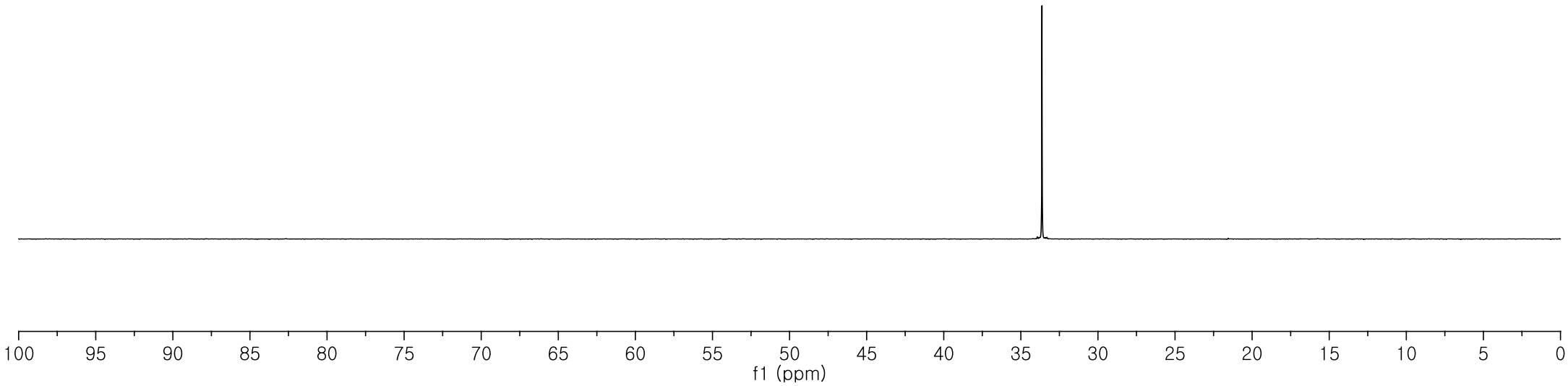


Table 3, 3bb

^{31}P NMR (162 MHz, $CDCl_3$)



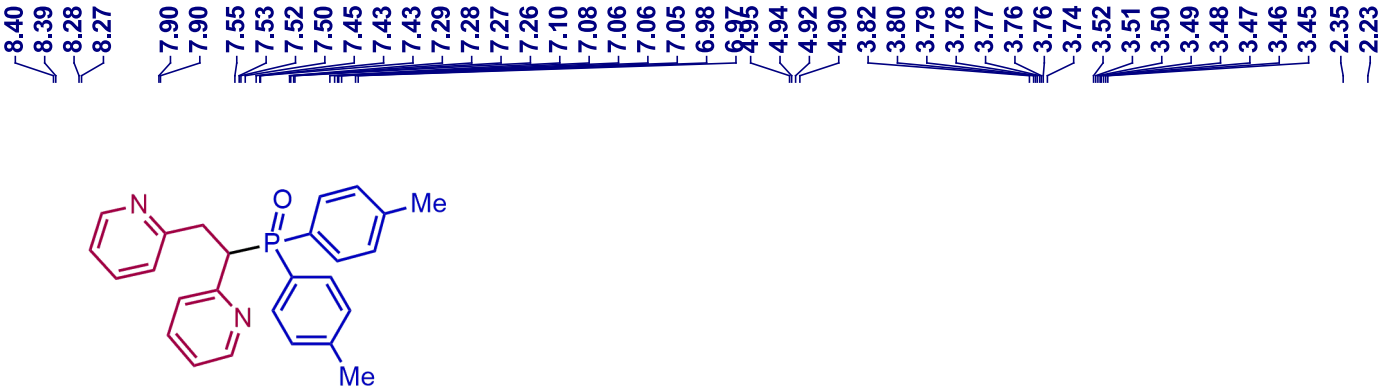


Table 3, 3bi

^{1}H NMR (400 MHz, $CDCl_3$)

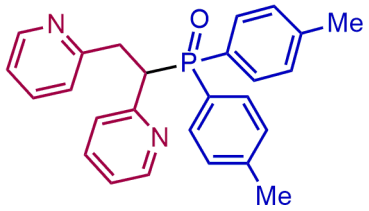


Table 3, 3bi

^{13}C NMR (101 MHz, $CDCl_3$)

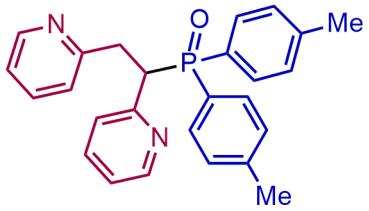
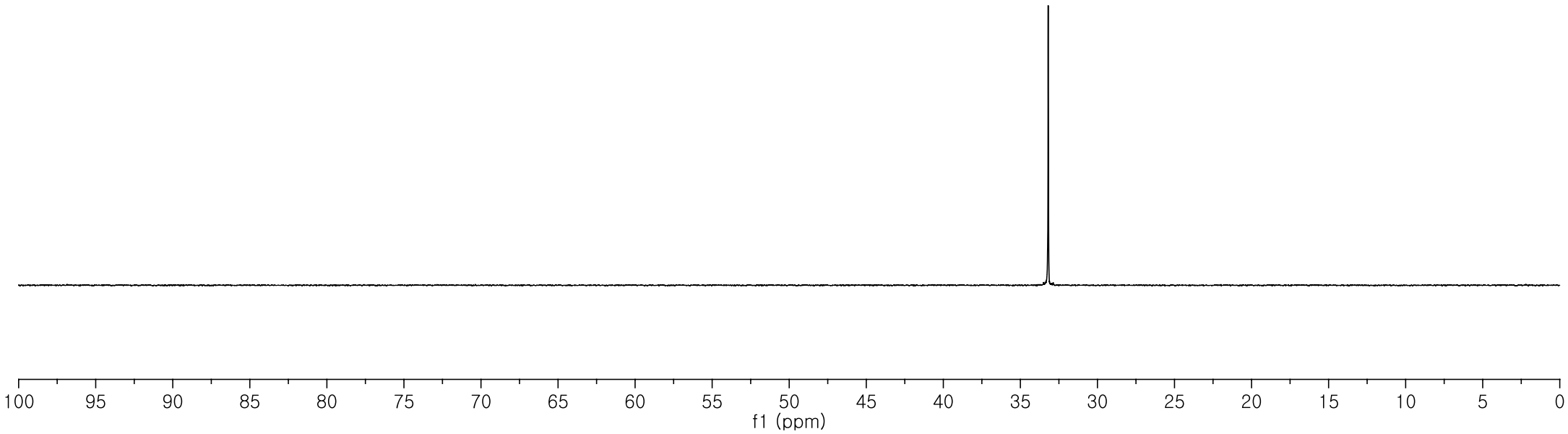
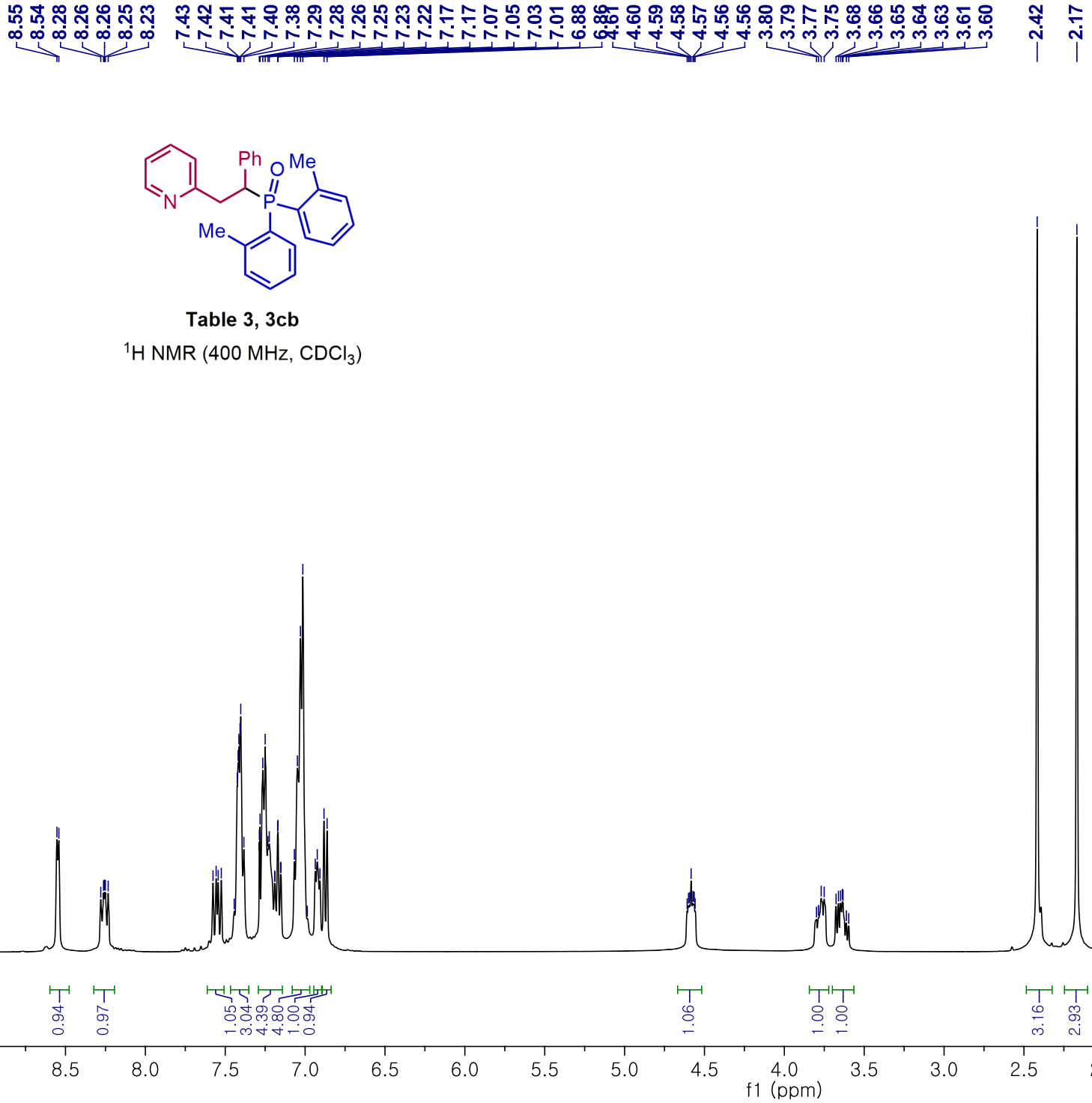


Table 3, 3bi

^{31}P NMR (162 MHz, $CDCl_3$)




8.55
8.54
8.28
8.26
8.26
8.25
8.23
7.43
7.42
7.41
7.41
7.40
7.38
7.29
7.28
7.26
7.25
7.23
7.22
7.17
7.17
7.07
7.05
7.03
7.01
6.88
6.86
4.61
4.60
4.59
4.58
4.57
4.56
4.56
3.80
3.79
3.77
3.75
3.68
3.66
3.65
3.64
3.63
3.61
3.60
2.42
2.17
Ph
O
Me
N
P
Me
Table 3, 3cb
1H NMR (400 MHz, CDCl3)
S75
0.94
0.97
1.05
3.04
4.39
4.80
1.00
0.94
1.06
1.00
1.00
3.16
2.93
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

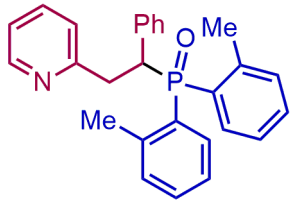


Table 3, 3cb

^{13}C NMR (101 MHz, $CDCl_3$)

158.58
158.45
148.62
142.98
142.90
141.72
141.64
136.74
135.93
135.88
132.61
132.50
132.32
132.22
131.93
131.82
131.66
131.59
131.25
131.13
131.02
130.68
130.07
129.82
129.77
127.98
126.76
125.74
125.63
125.03
124.91
124.51
121.59
44.90
44.23
38.25
21.41
21.37
21.05
21.02

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)

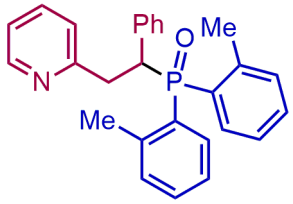
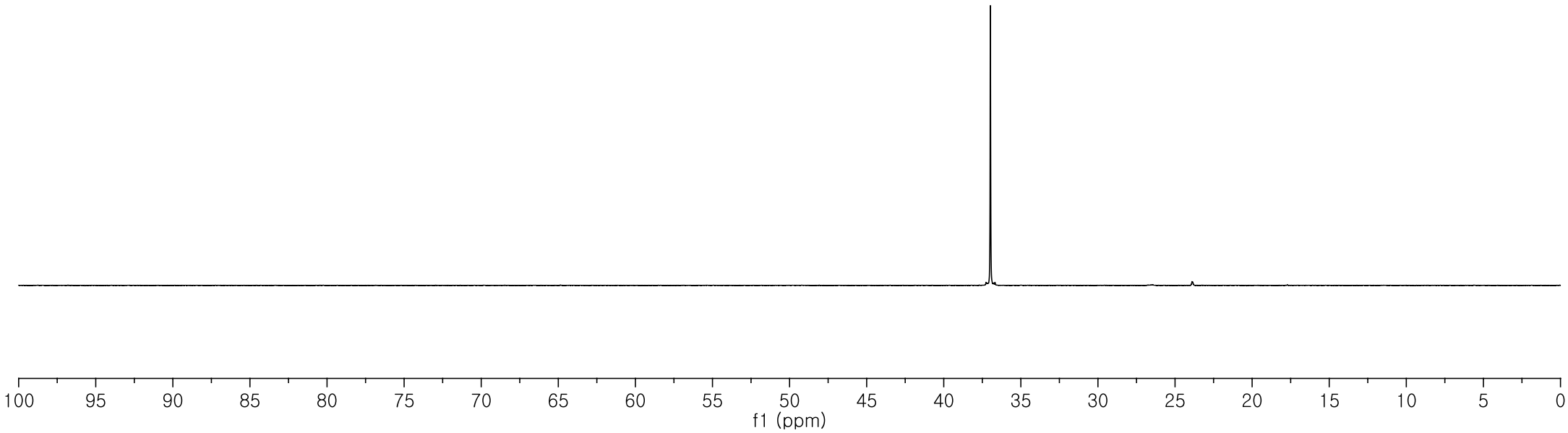


Table 3, 3cb

^{31}P NMR (162 MHz, $CDCl_3$)



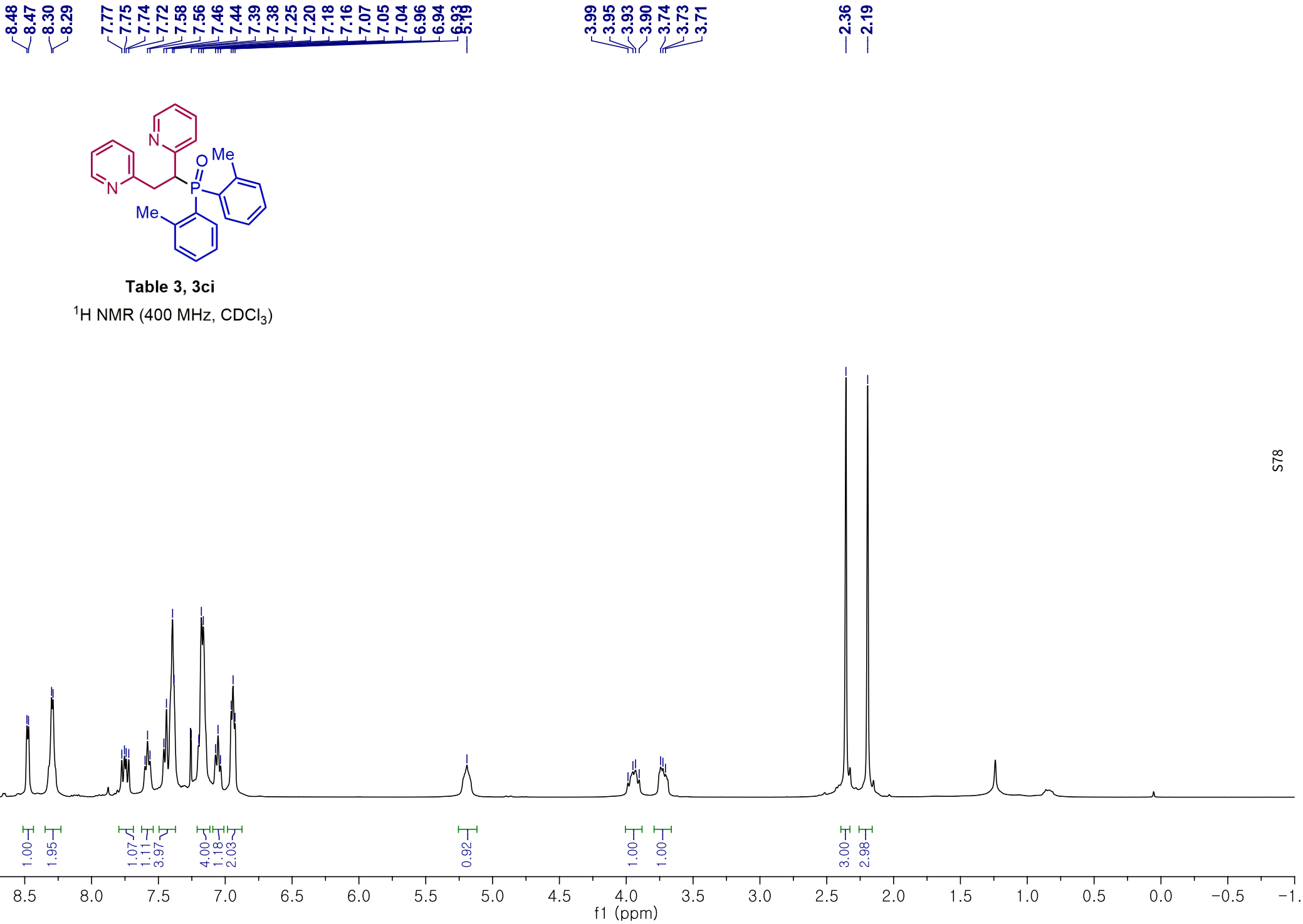


Table 3, 3ci

^{1}H NMR (400 MHz, $CDCl_3$)

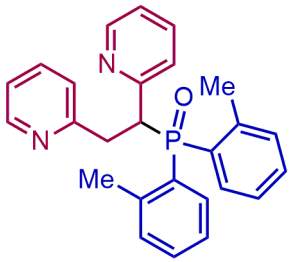


Table 3, 3ci

^{13}C NMR (101 MHz, $CDCl_3$)

157.41
157.28
155.78
148.19
146.78
142.53
142.45
142.02
141.93
138.63
136.66
132.74
132.63
132.39
132.28
132.22
132.11
131.91
131.45
131.41
131.30
130.83
130.55
129.89
129.59
125.97
125.85
125.30
125.26
125.13
122.24
121.99
46.77
46.14
36.39
21.39
21.35
21.12
21.08

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)

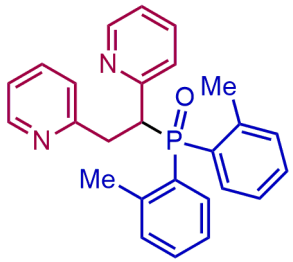
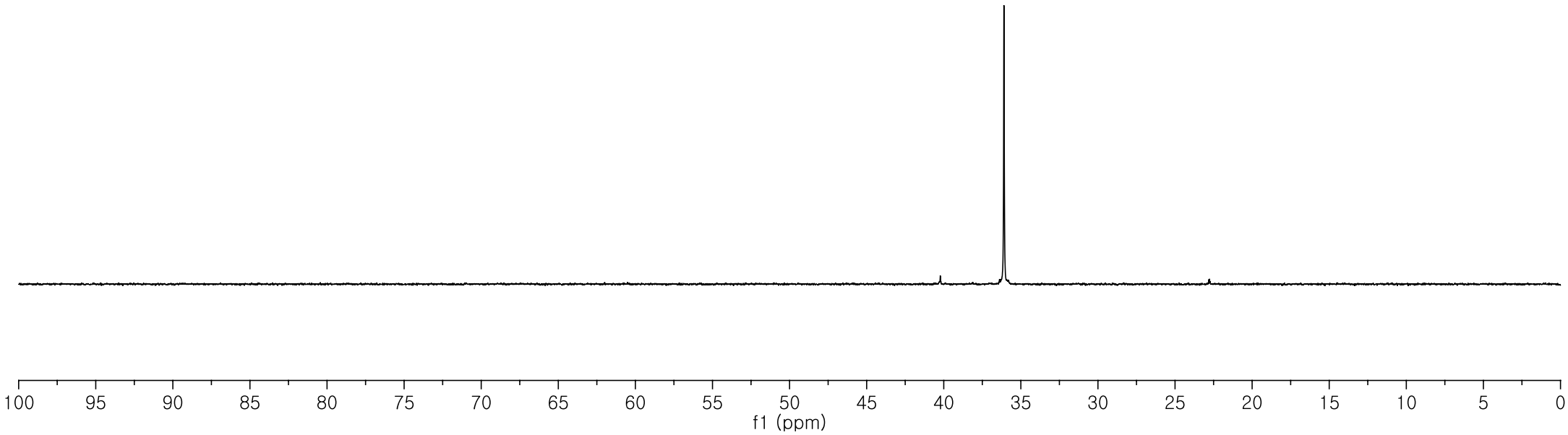


Table 3, 3ci

^{31}P NMR (162 MHz, $CDCl_3$)



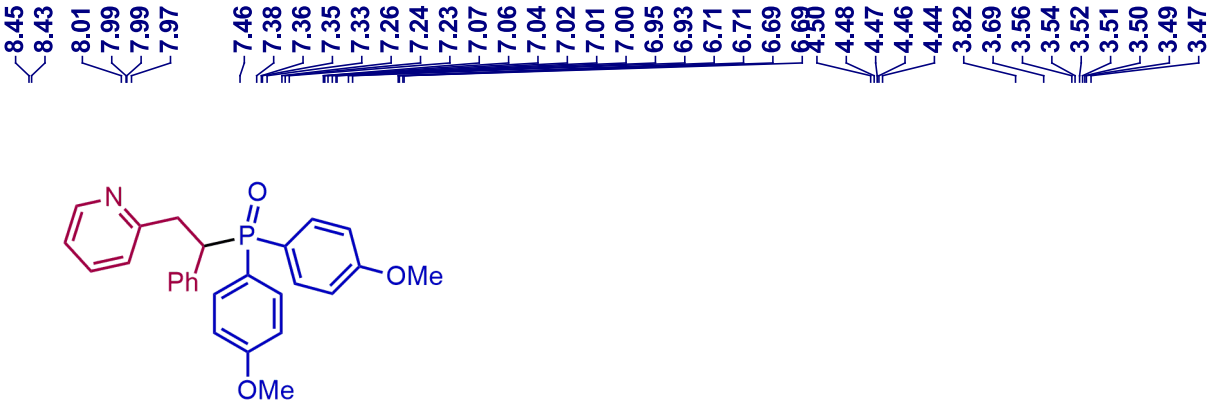


Table 3, 3db

^{1}H NMR (400 MHz, $CDCl_3$)

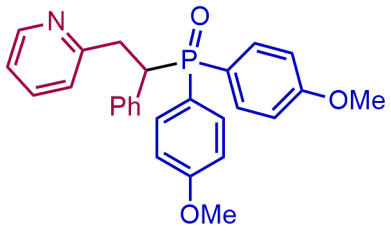


Table 3, 3db

^{13}C NMR (101 MHz, $CDCl_3$)

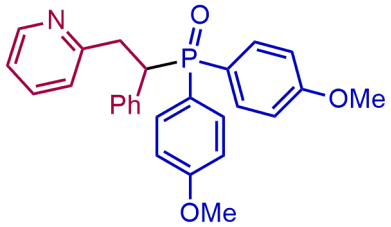
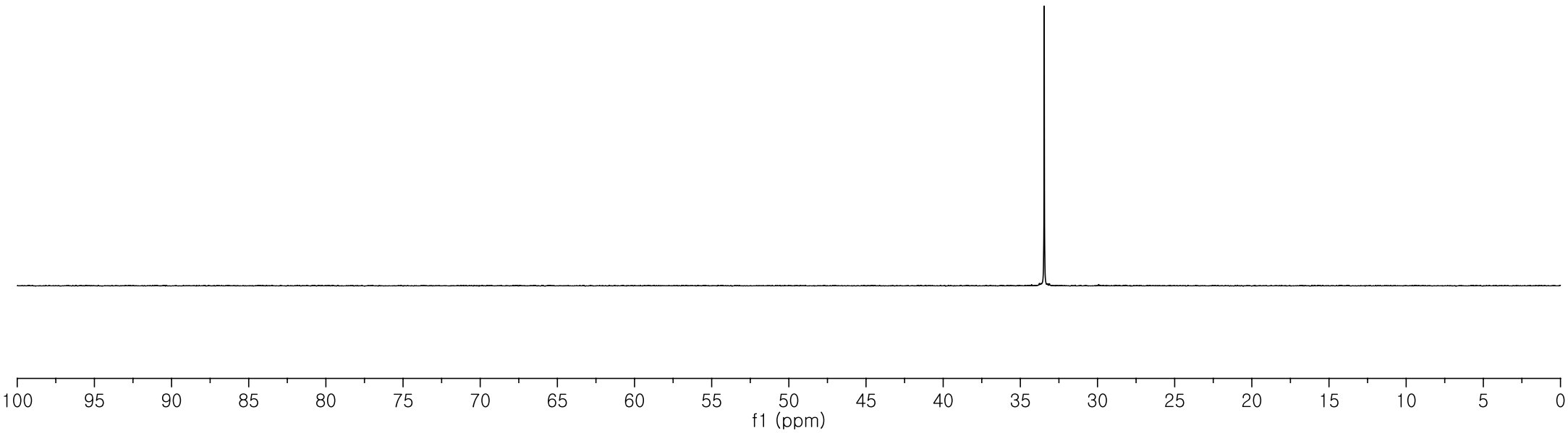


Table 3, 3db

^{31}P NMR (162 MHz, $CDCl_3$)



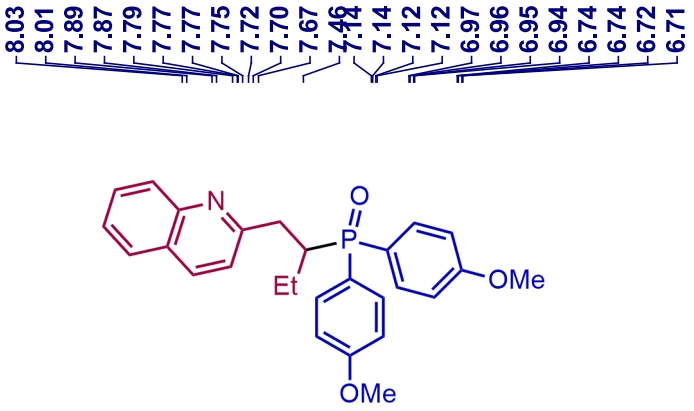


Table 3, 3dp

^{1}H NMR (400 MHz, $CDCl_3$)

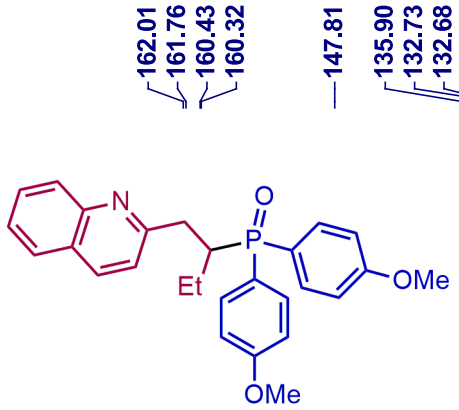


Table 3, 3dp

^{13}C NMR (101 MHz, $CDCl_3$)

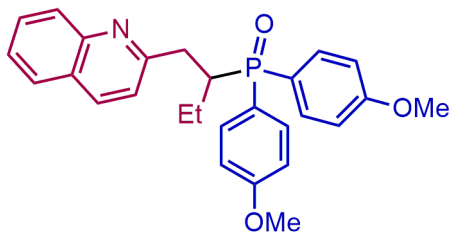
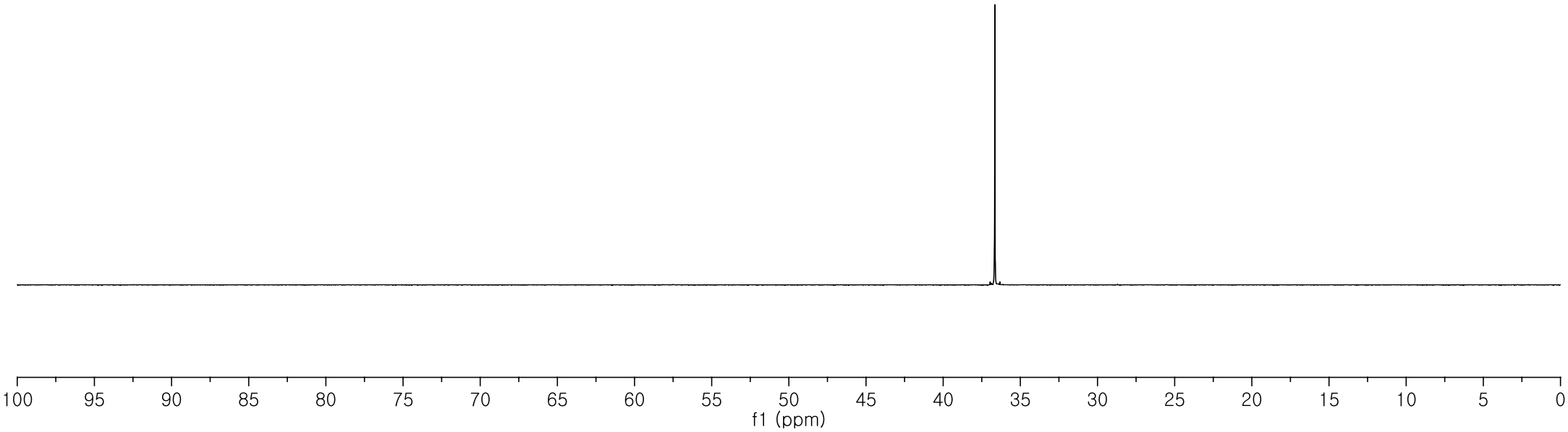


Table 3, 3dp

^{31}P NMR (162 MHz, $CDCl_3$)



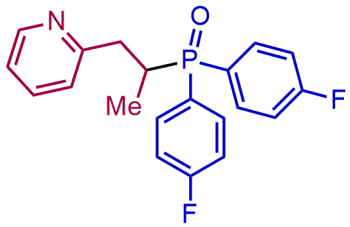


Table 3, 3ea

^{1}H NMR (400 MHz, $CDCl_3$)

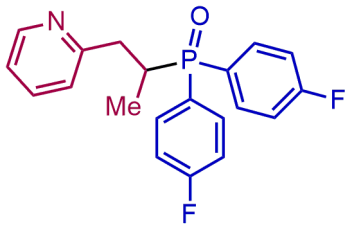


Table 3, 3ea

^{13}C NMR (101 MHz, $CDCl_3$)

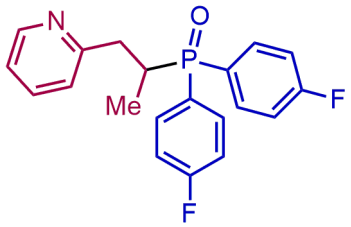
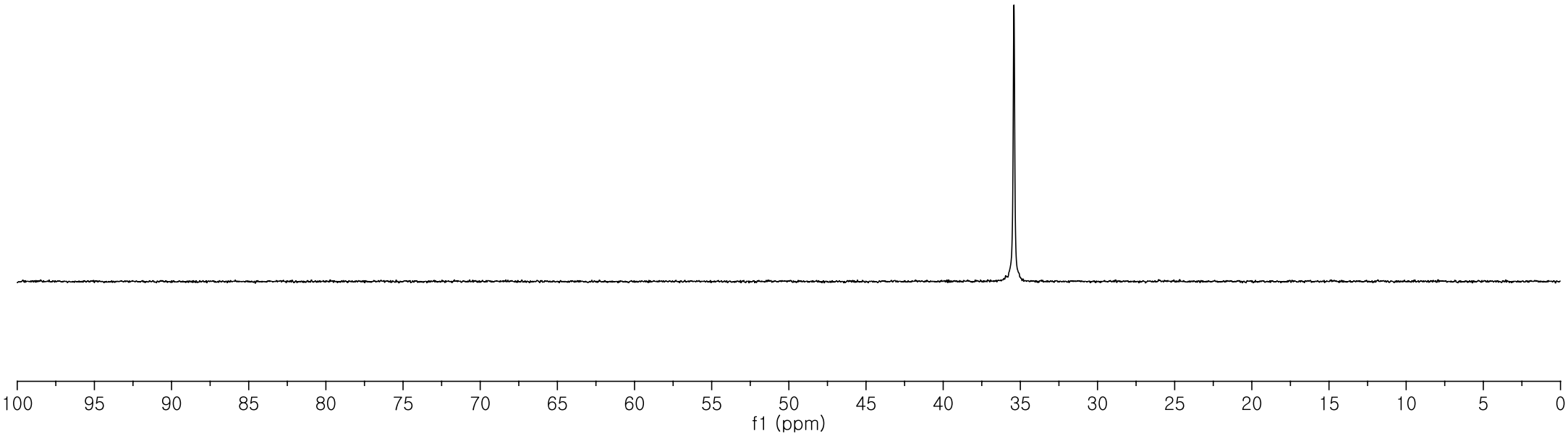
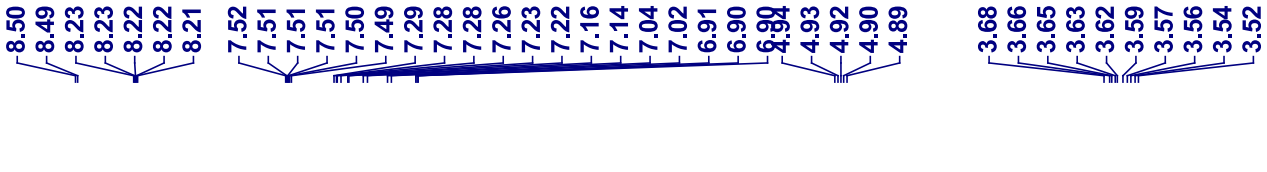


Table 3, 3ea

^{31}P NMR (162 MHz, $CDCl_3$)





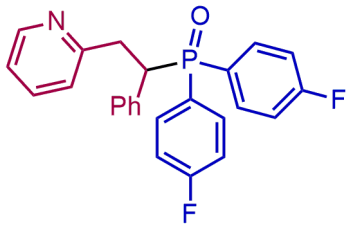
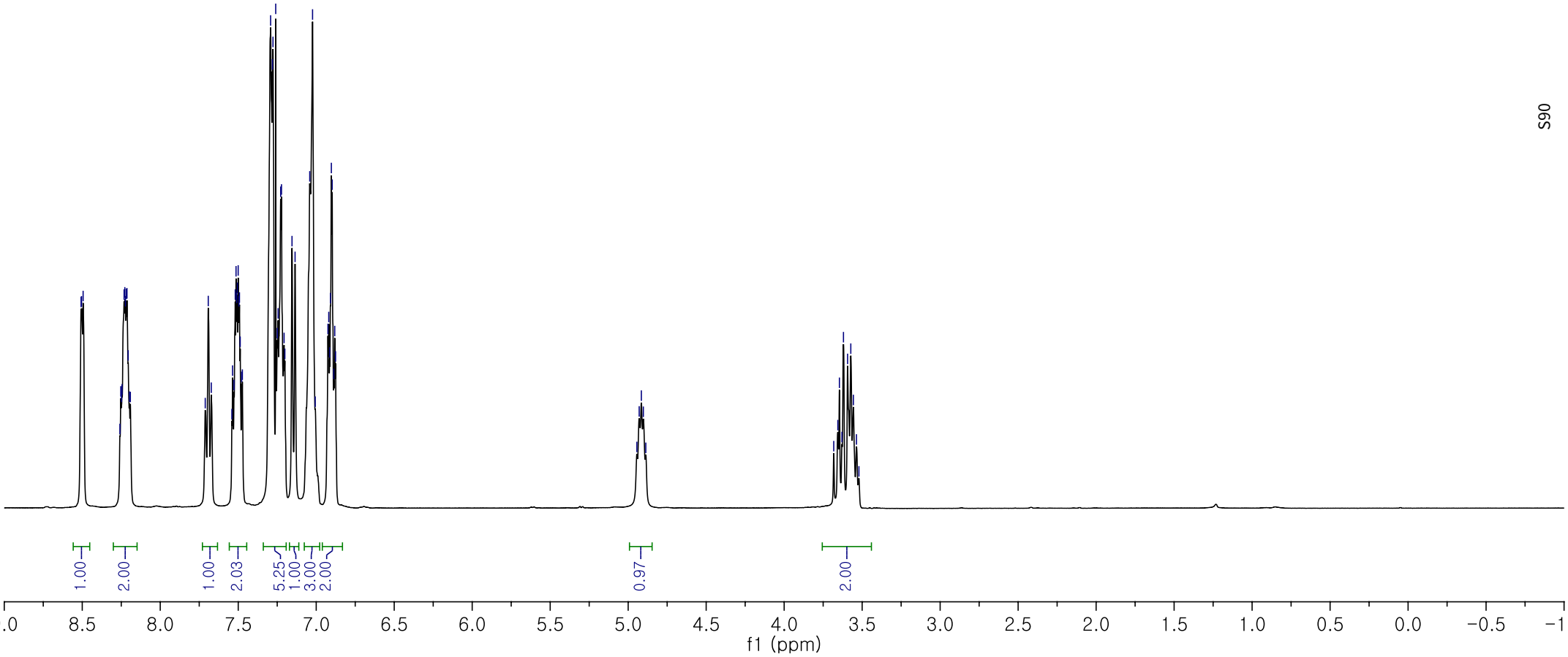
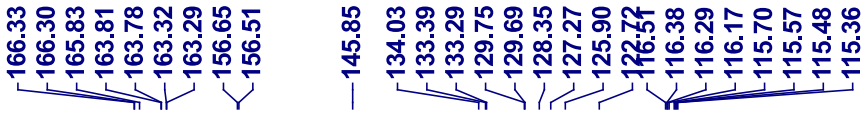


Table 3, 3eb

^{1}H NMR (400 MHz, $CDCl_3$)





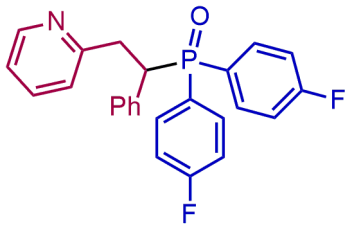
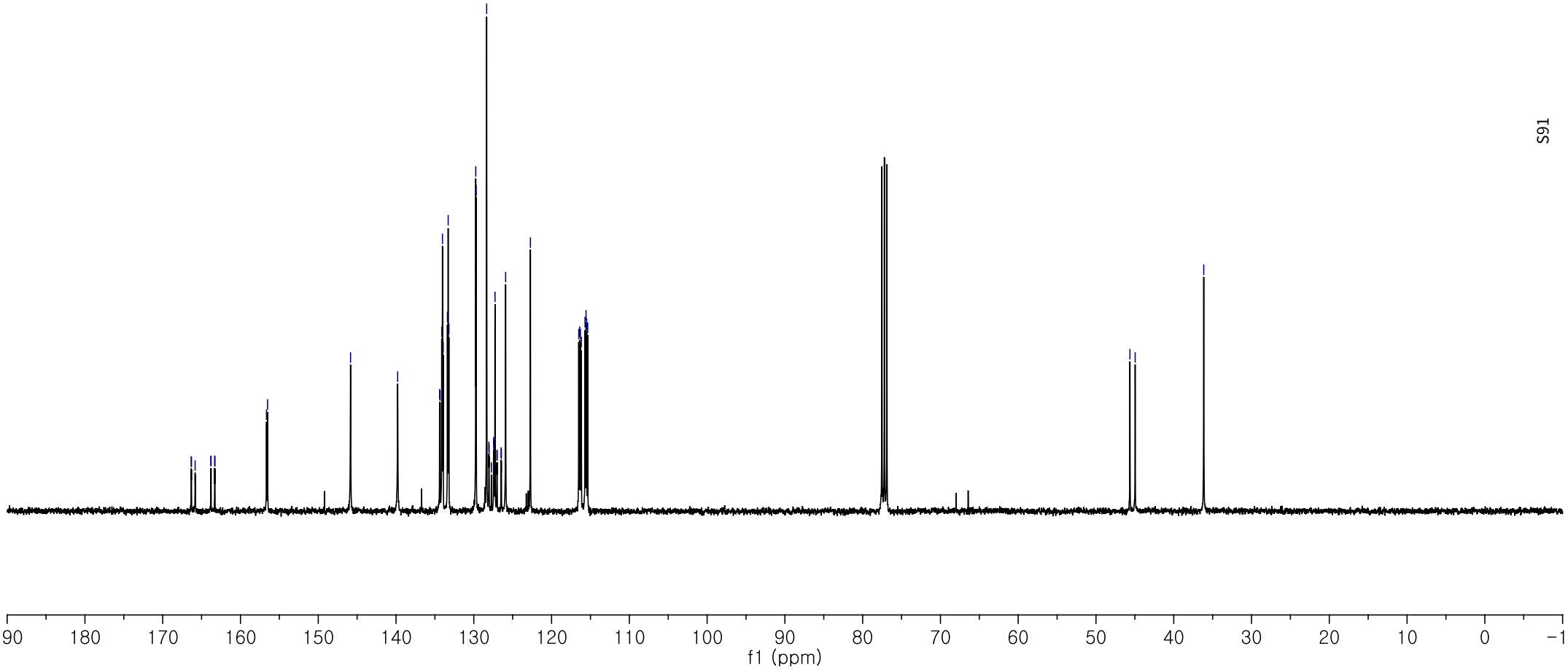


Table 3, 3eb

^{13}C NMR (101 MHz, $CDCl_3$)



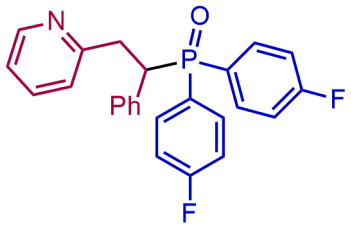
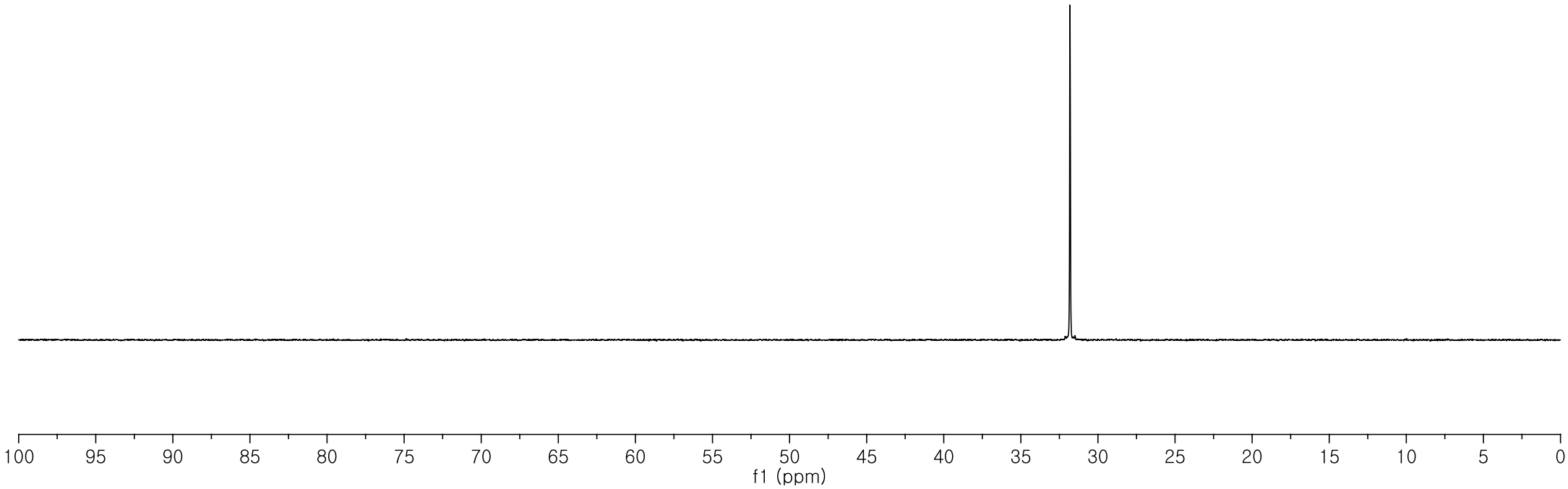


Table 3, 3eb

^{31}P NMR (162 MHz, $CDCl_3$)



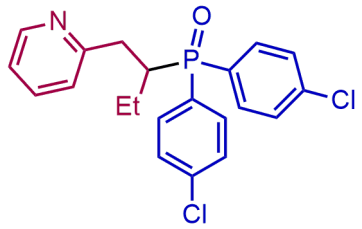
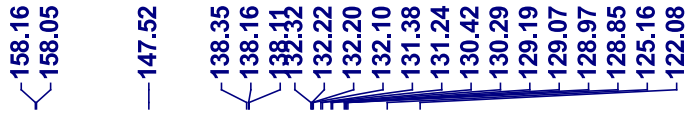


Table 3, 3fc

^{1}H NMR (400 MHz, $CDCl_3$)



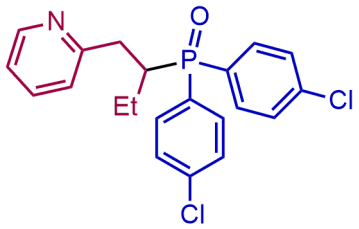
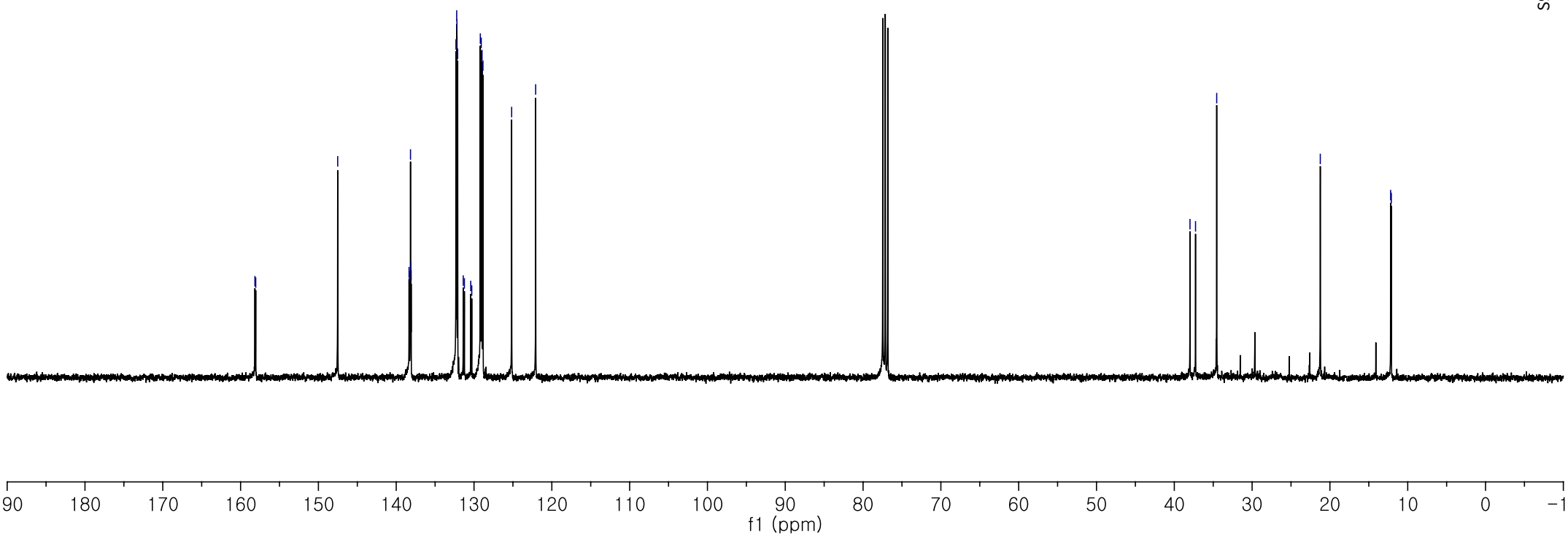


Table 3, 3fc

^{13}C NMR (101 MHz, $CDCl_3$)



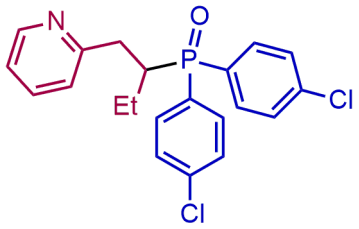
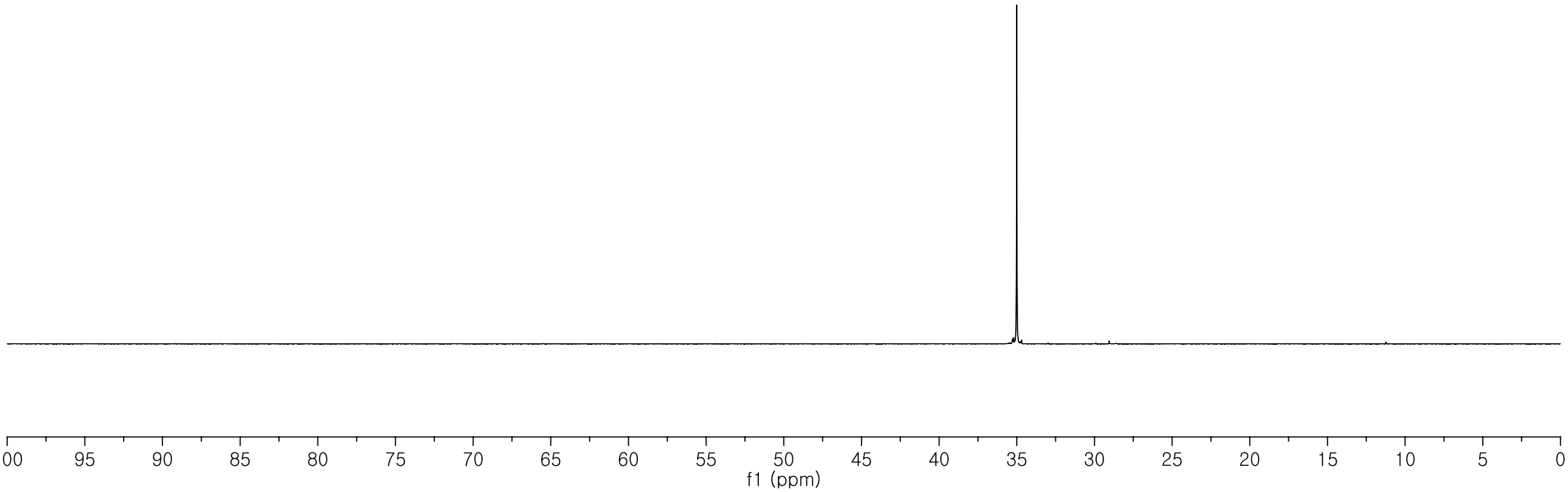


Table 3, 3fc

^{31}P NMR (162 MHz, $CDCl_3$)



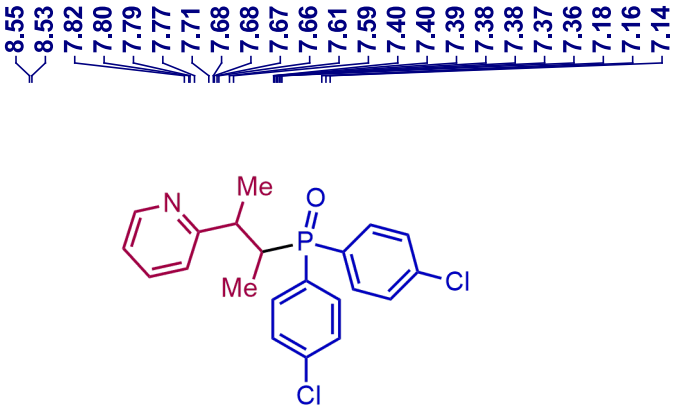


Table 3, 3ff

^{1}H NMR (400 MHz, $CDCl_3$)

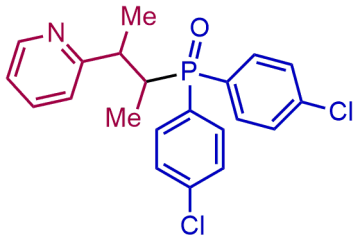


Table 3, 3ff

^{13}C NMR (101 MHz, $CDCl_3$)

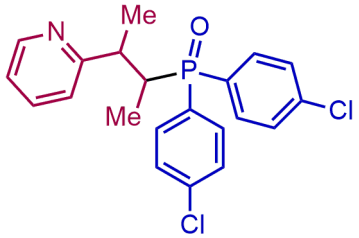
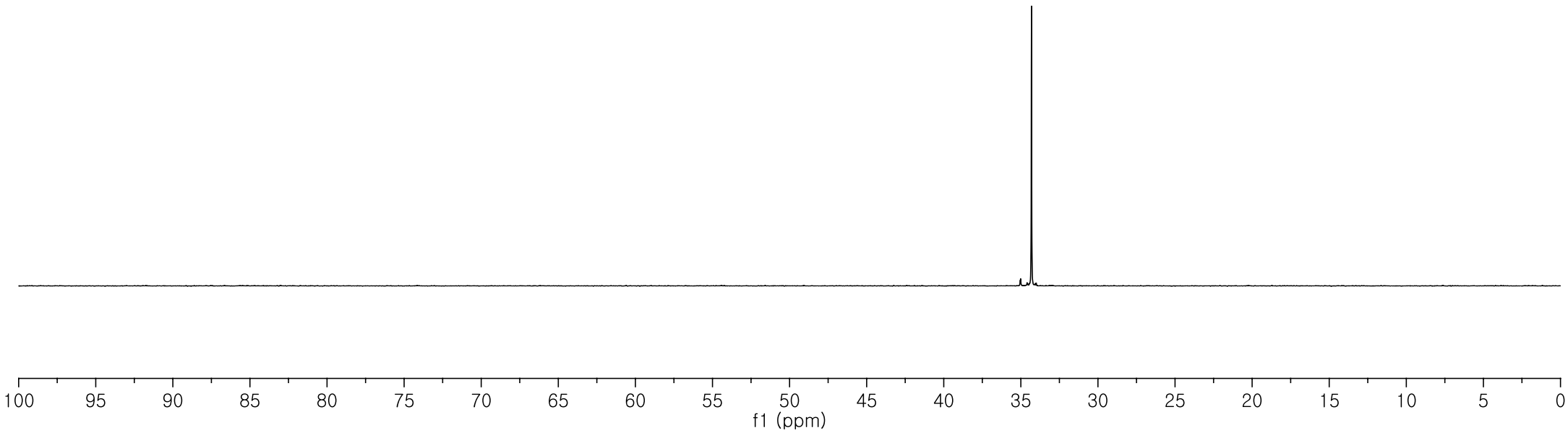


Table 3, 3ff

^{31}P NMR (162 MHz, $CDCl_3$)



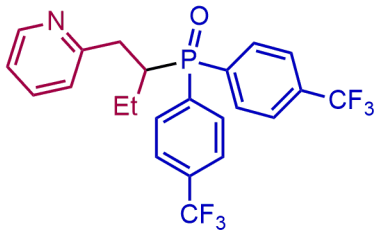


Table 3, 3gc

^{1}H NMR (400 MHz, $CDCl_3$)

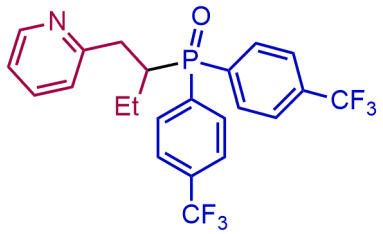


Table 3, 3gc

^{13}C NMR (101 MHz, $CDCl_3$)

157.72
157.62
147.10
139.00
137.24
137.14
136.31
136.23
131.62
131.53
131.45
131.36
125.92
125.89
125.81
125.77
125.67
125.61
125.57
125.50
125.46
122.63
122.19
77.51
77.19
76.87
37.94
37.23
34.37
21.49
12.22
12.13

S100

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)

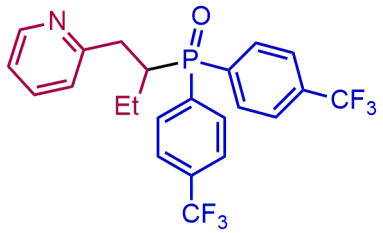
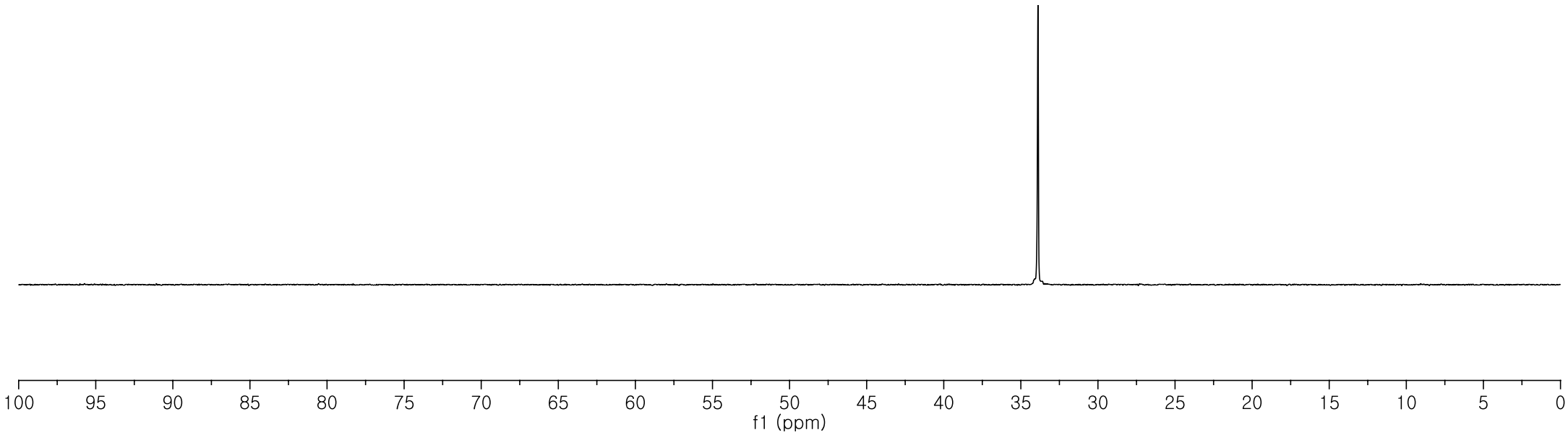


Table 3, 3gc

^{31}P NMR (162 MHz, $CDCl_3$)



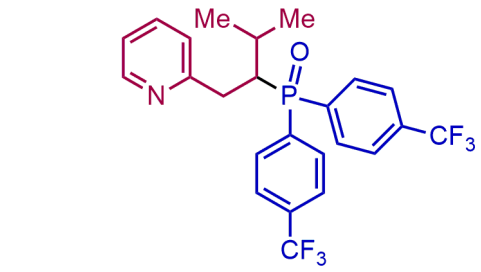


Table 3, 3gd

^{1}H NMR (400 MHz, $CDCl_3$)

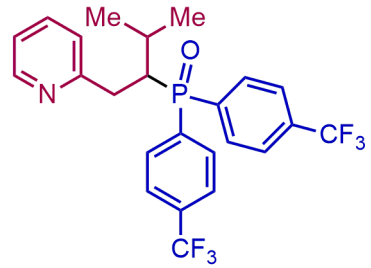


Table 3, 3gd

^{13}C NMR (101 MHz, $CDCl_3$)

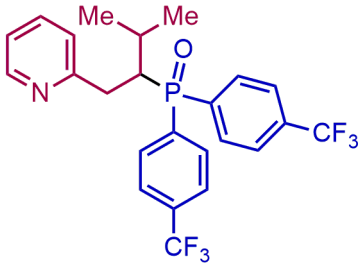
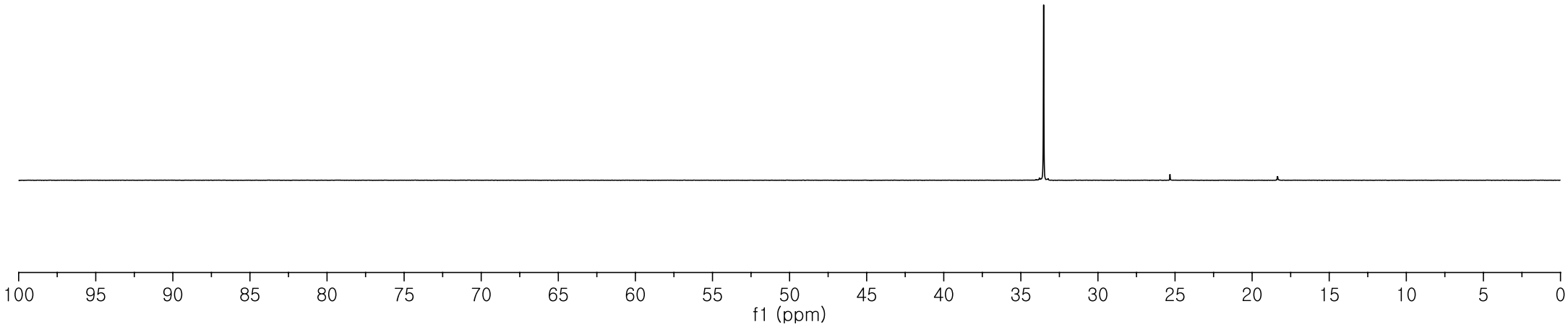


Table 3, 3gd

^{31}P NMR (162 MHz, $CDCl_3$)



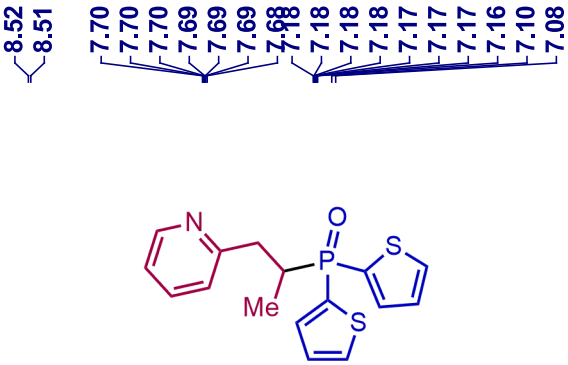


Table 3, 3ha

^{1}H NMR (400 MHz, $CDCl_3$)

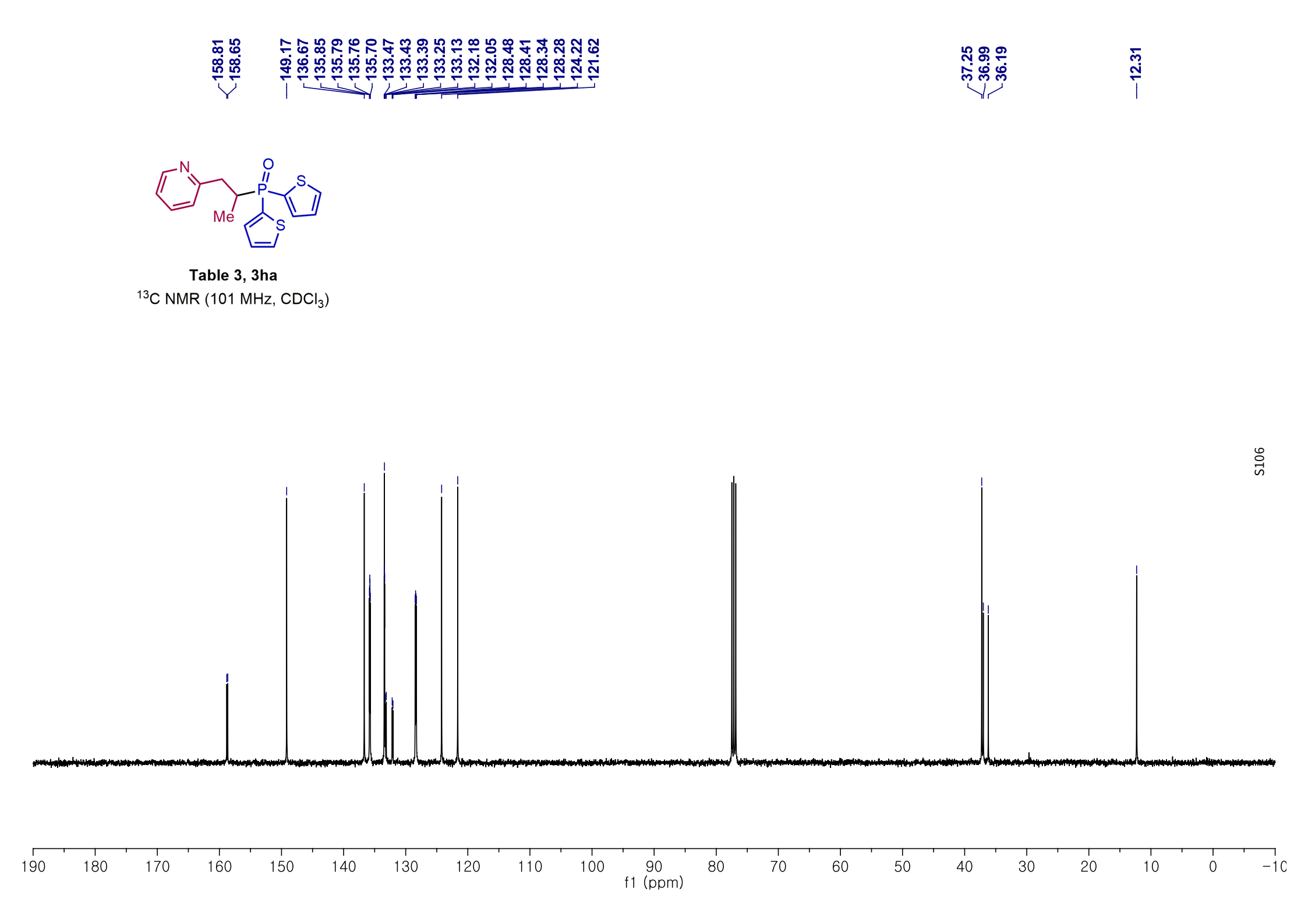


Table 3, 3ha

^{13}C NMR (101 MHz, $CDCl_3$)

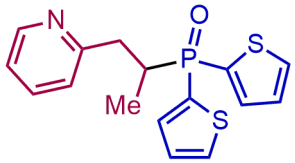
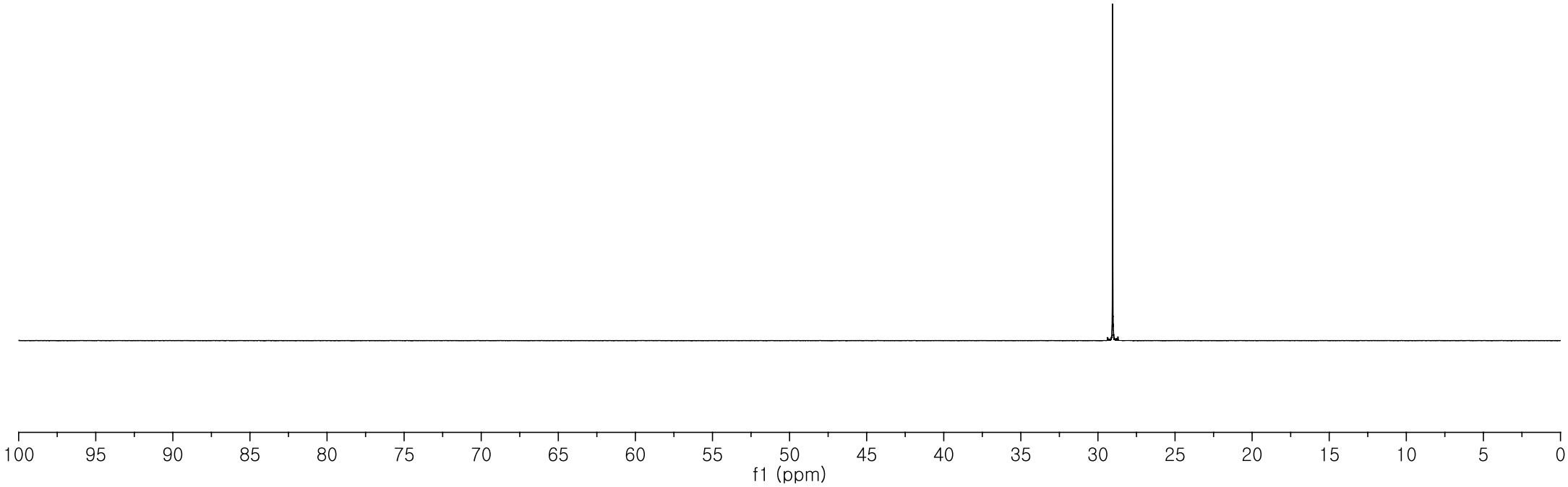
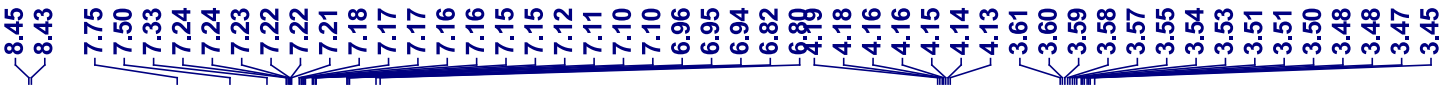


Table 3, 3ha

^{31}P NMR (162 MHz, $CDCl_3$)





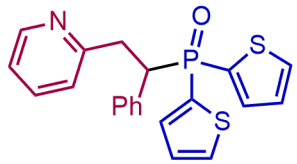
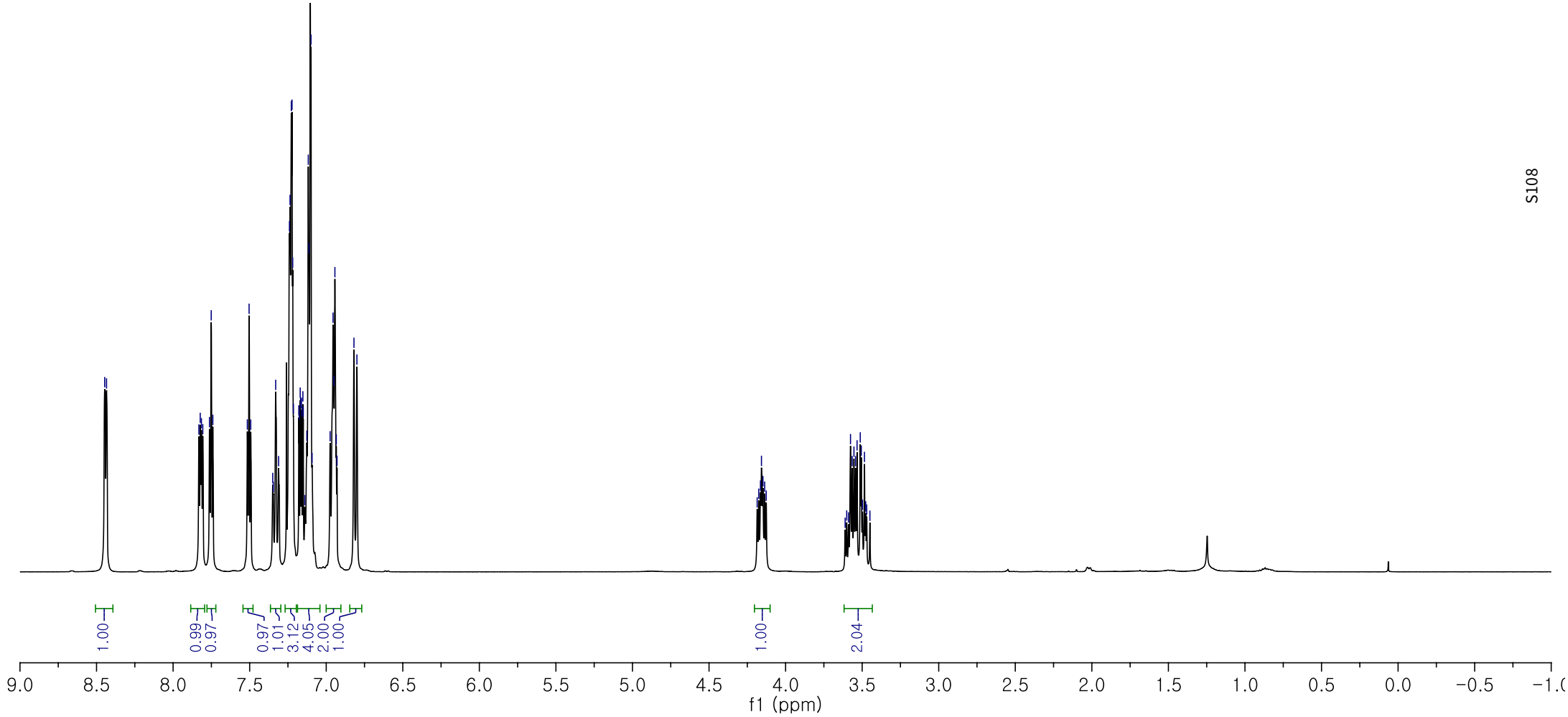
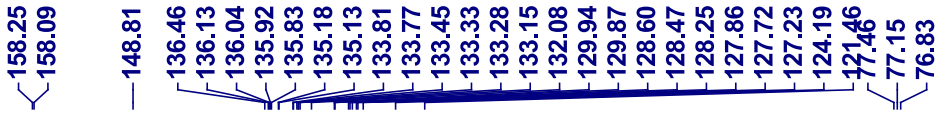


Table 3, 3hb

^{1}H NMR (400 MHz, $CDCl_3$)





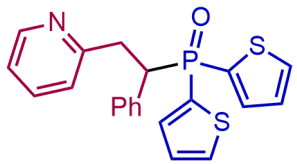
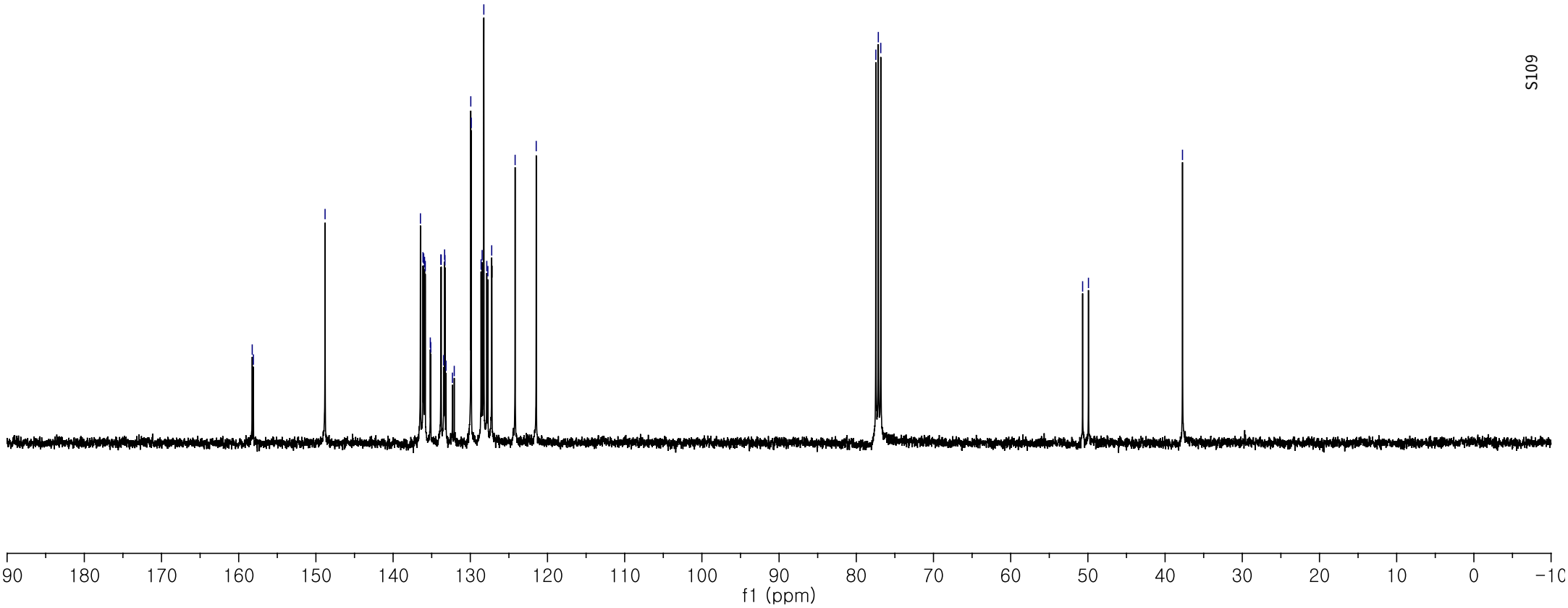


Table 3, 3hb

^{13}C NMR (101 MHz, $CDCl_3$)



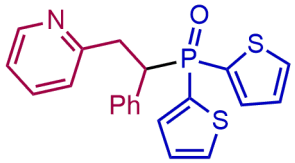
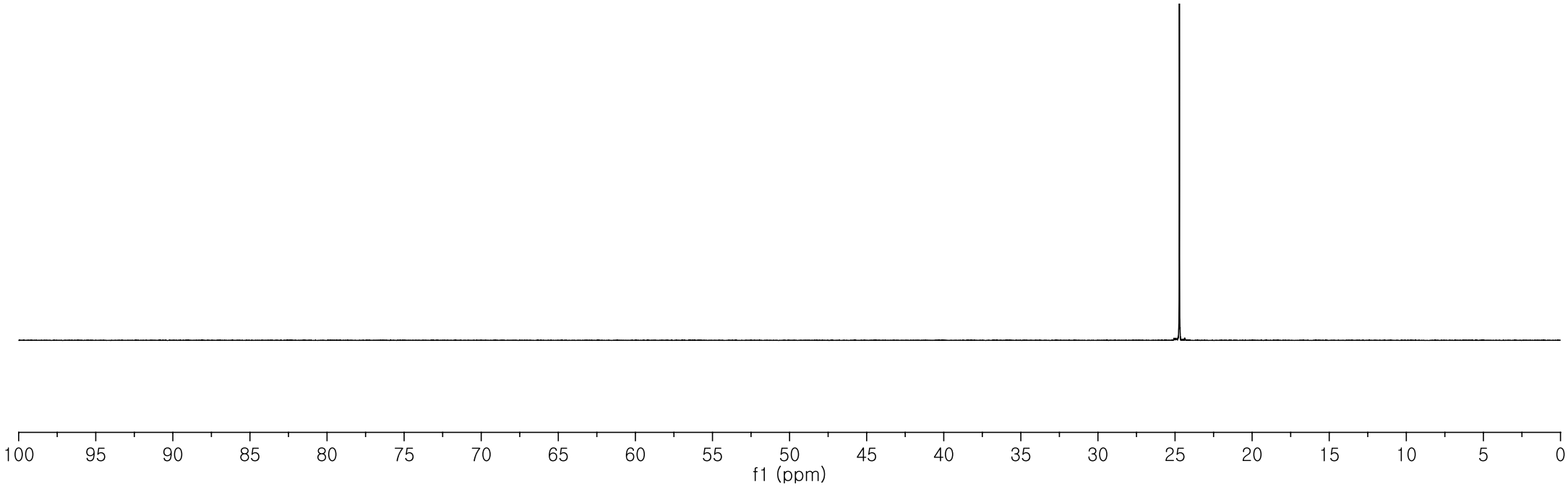


Table 3, 3hb

^{31}P NMR (162 MHz, $CDCl_3$)

S110



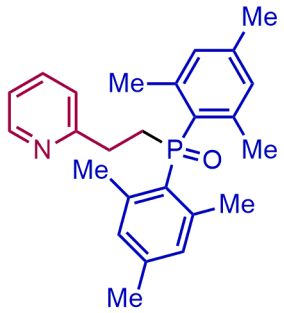


Table 3, 3i

^{1}H NMR (400 MHz, $CDCl_3$)

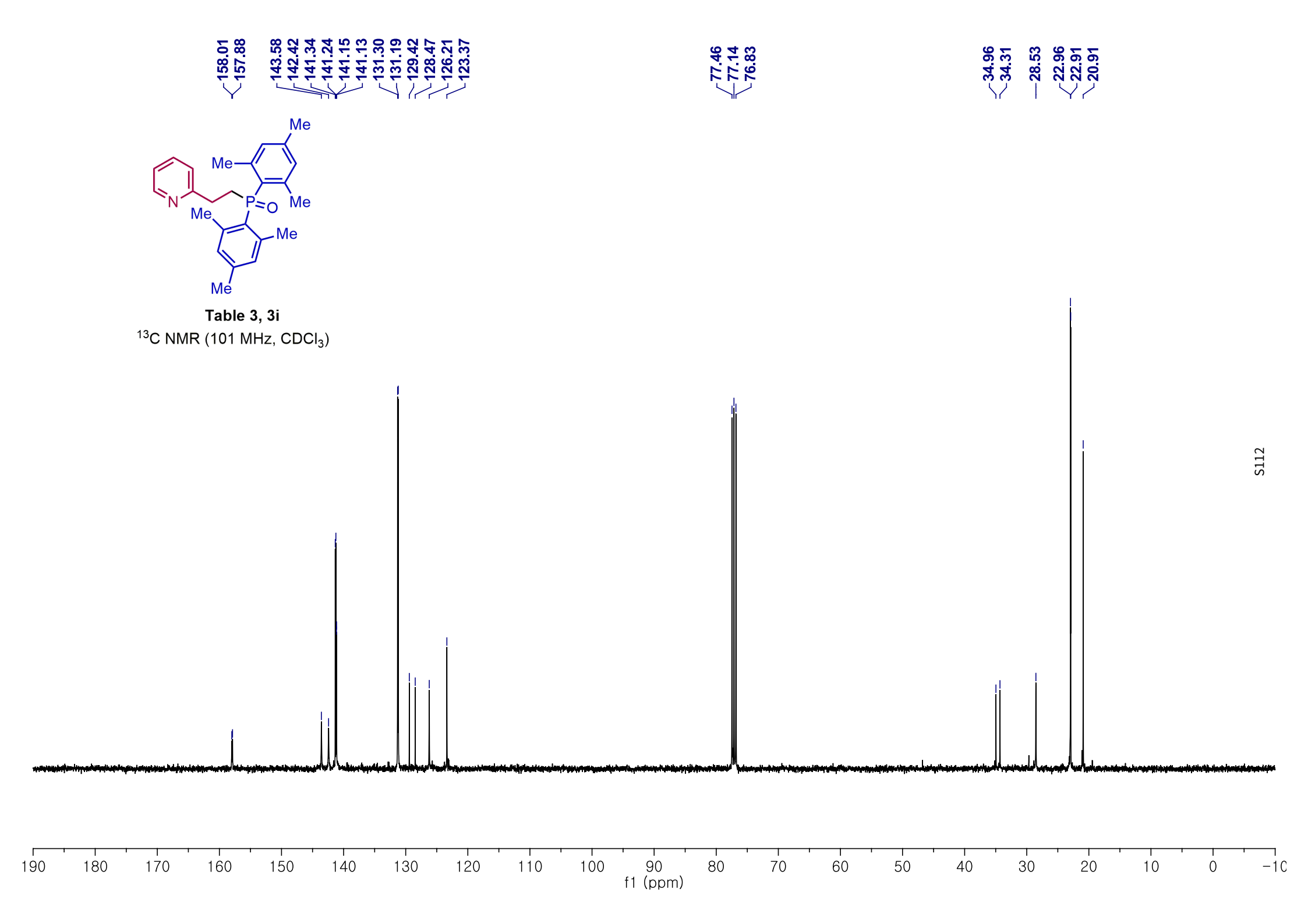


Table 3, 3i

^{13}C NMR (101 MHz, $CDCl_3$)

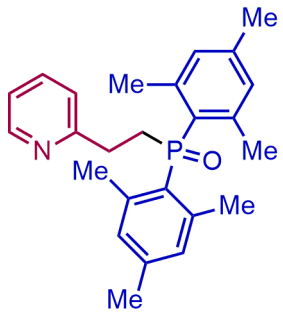
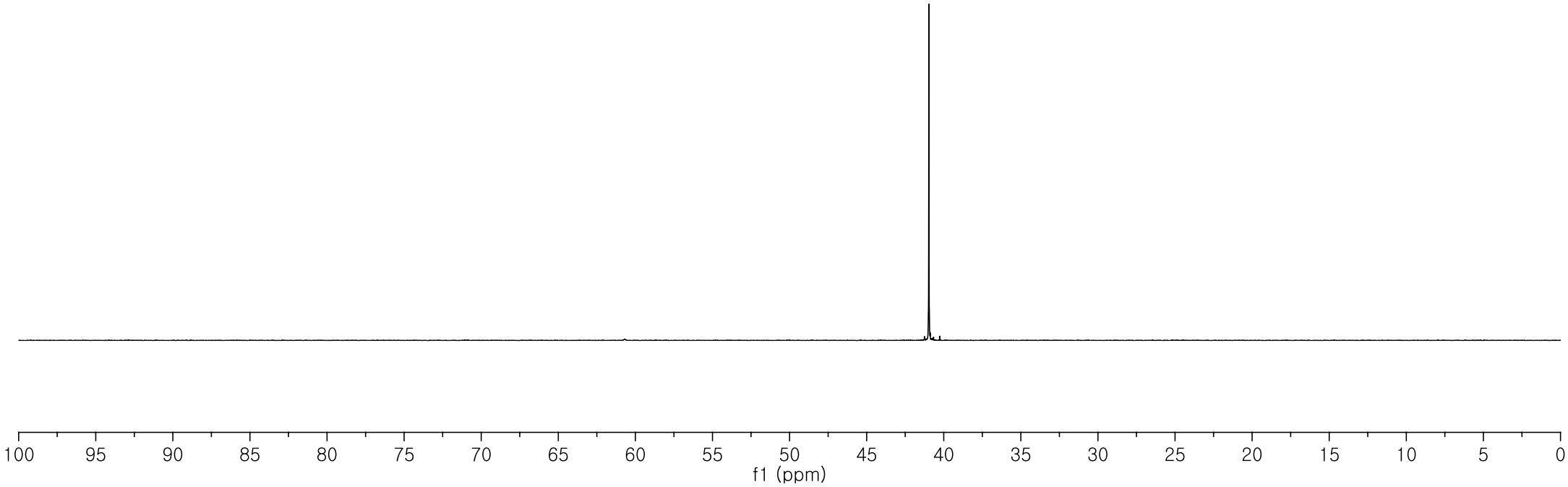


Table 3, 3i

^{31}P NMR (162 MHz, $CDCl_3$)



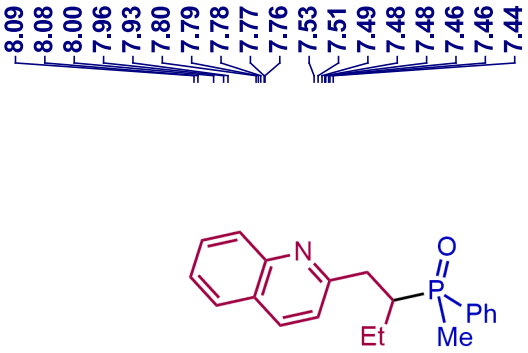
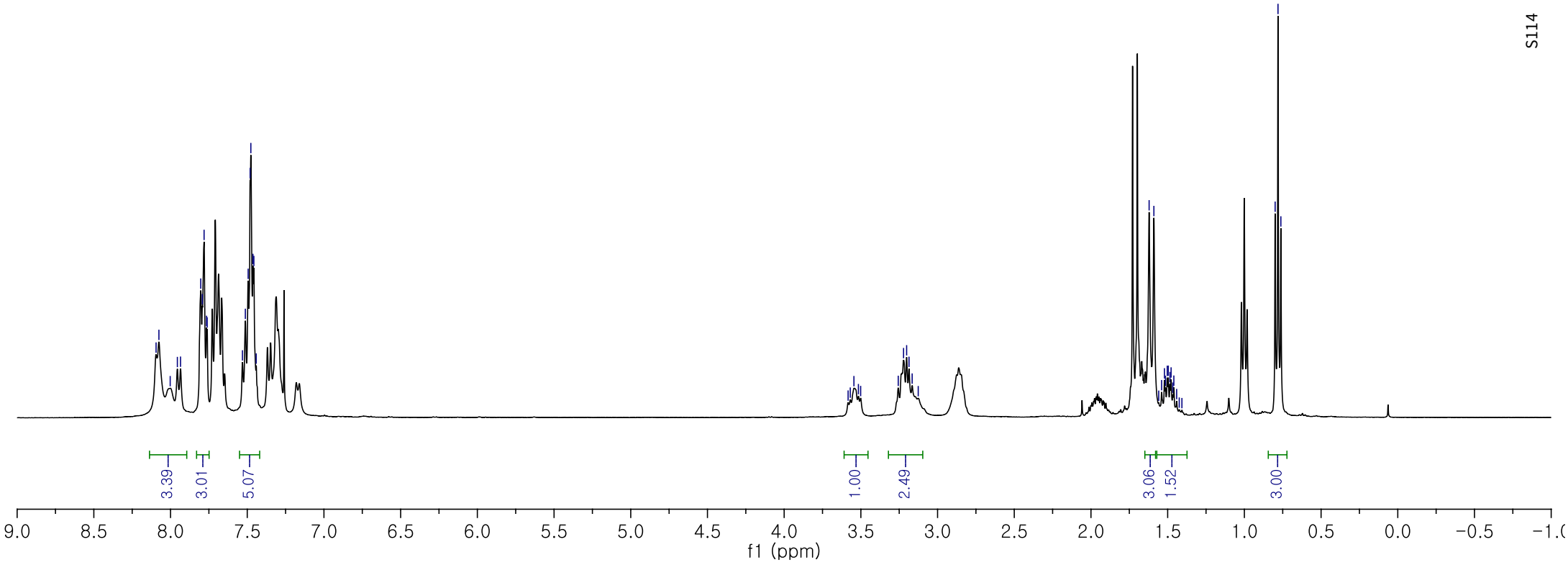


Table 4, 3jp (major)

^{1}H NMR (300 MHz, $CDCl_3$)



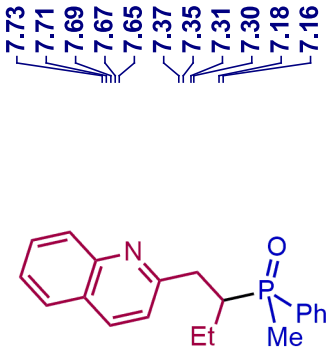
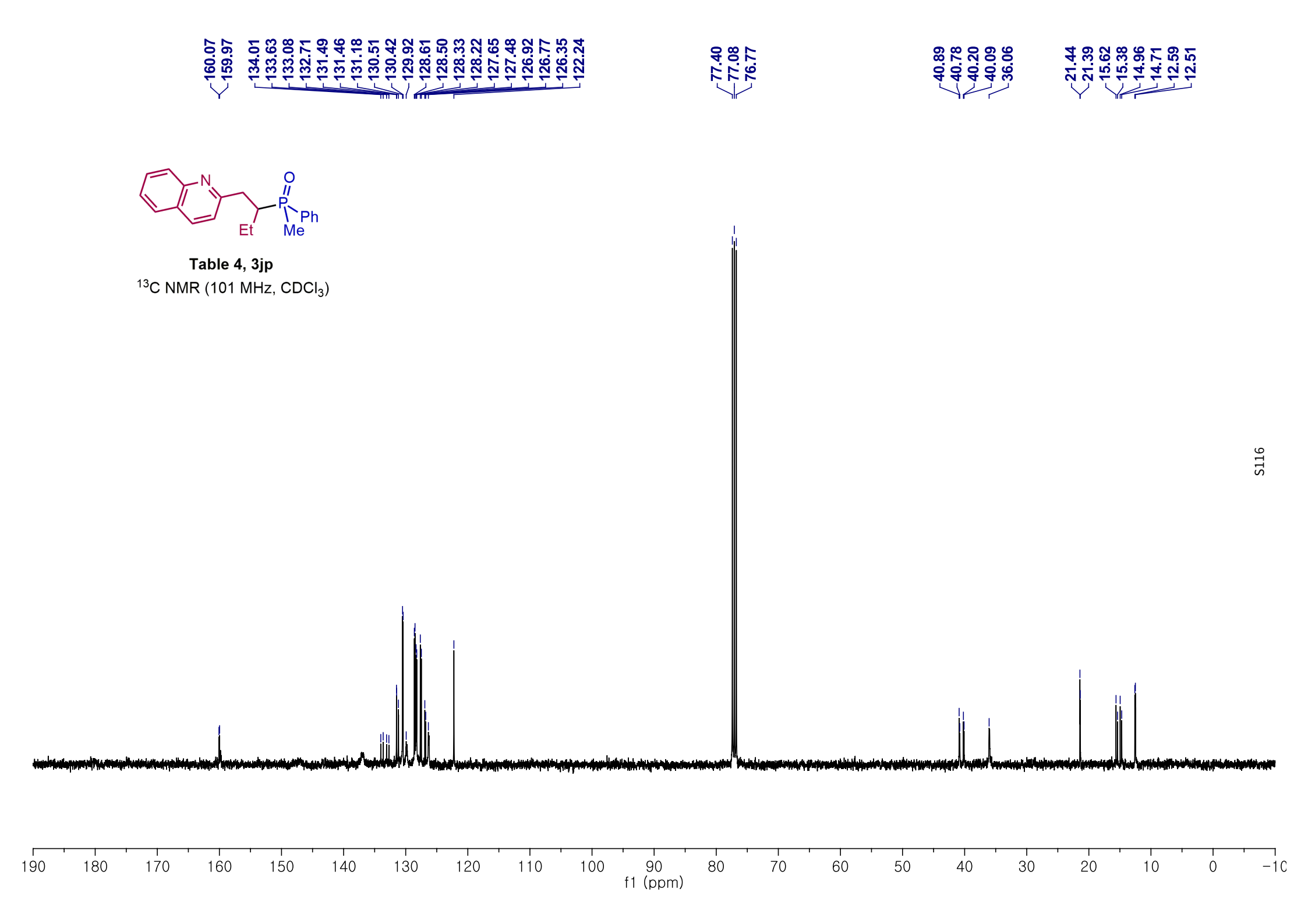


Table 4, 3jp (minor)

^{1}H NMR (300 MHz, $CDCl_3$)


160.07
159.97
134.01
133.63
133.08
132.71
131.49
131.46
131.18
130.51
130.42
129.92
128.61
128.50
128.33
128.22
127.65
127.48
126.92
126.77
126.35
122.24
77.40
77.08
76.77
40.89
40.78
40.20
40.09
36.06
21.44
21.39
15.62
15.38
14.96
14.71
12.59
12.51
N
O
P
Ph
Me
Et
Table 4, 3jp
13C NMR (101 MHz, CDCl3)
S116
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

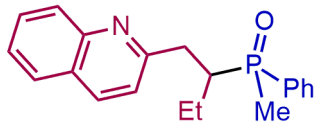
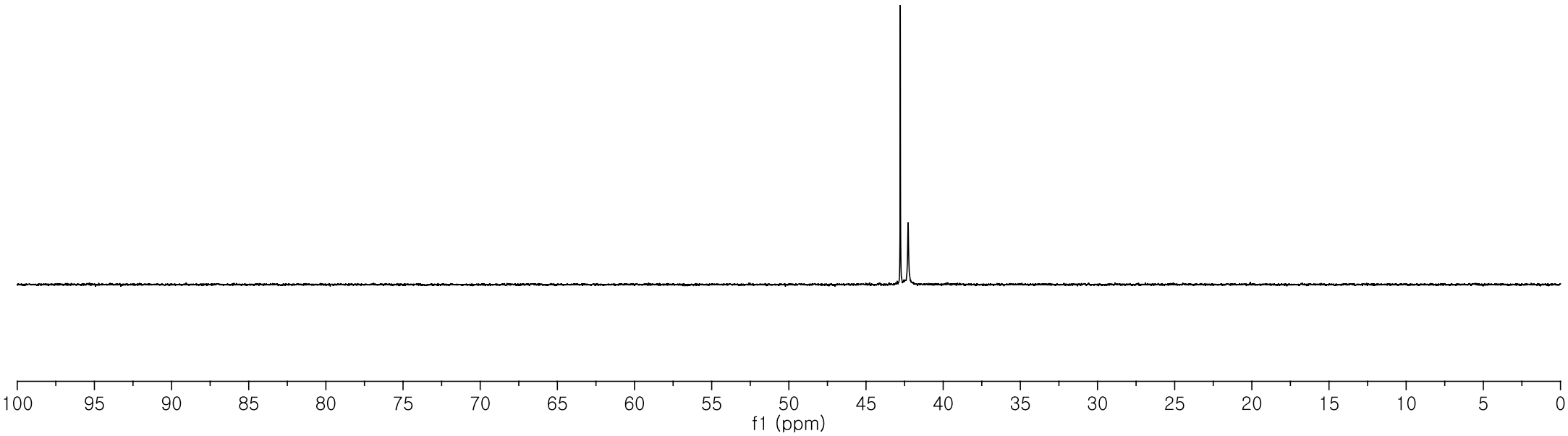


Table 4, 3jp

^{31}P NMR (122 MHz, $CDCl_3$)



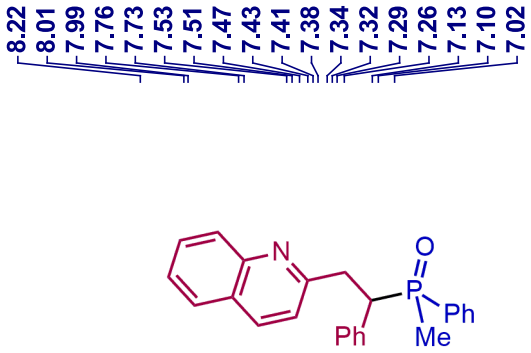
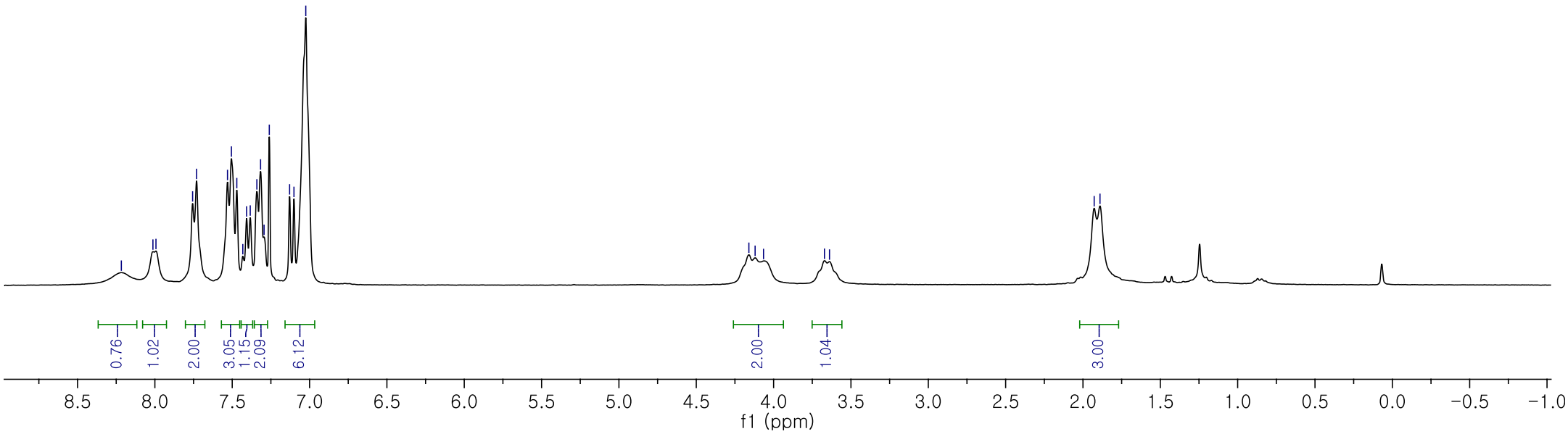
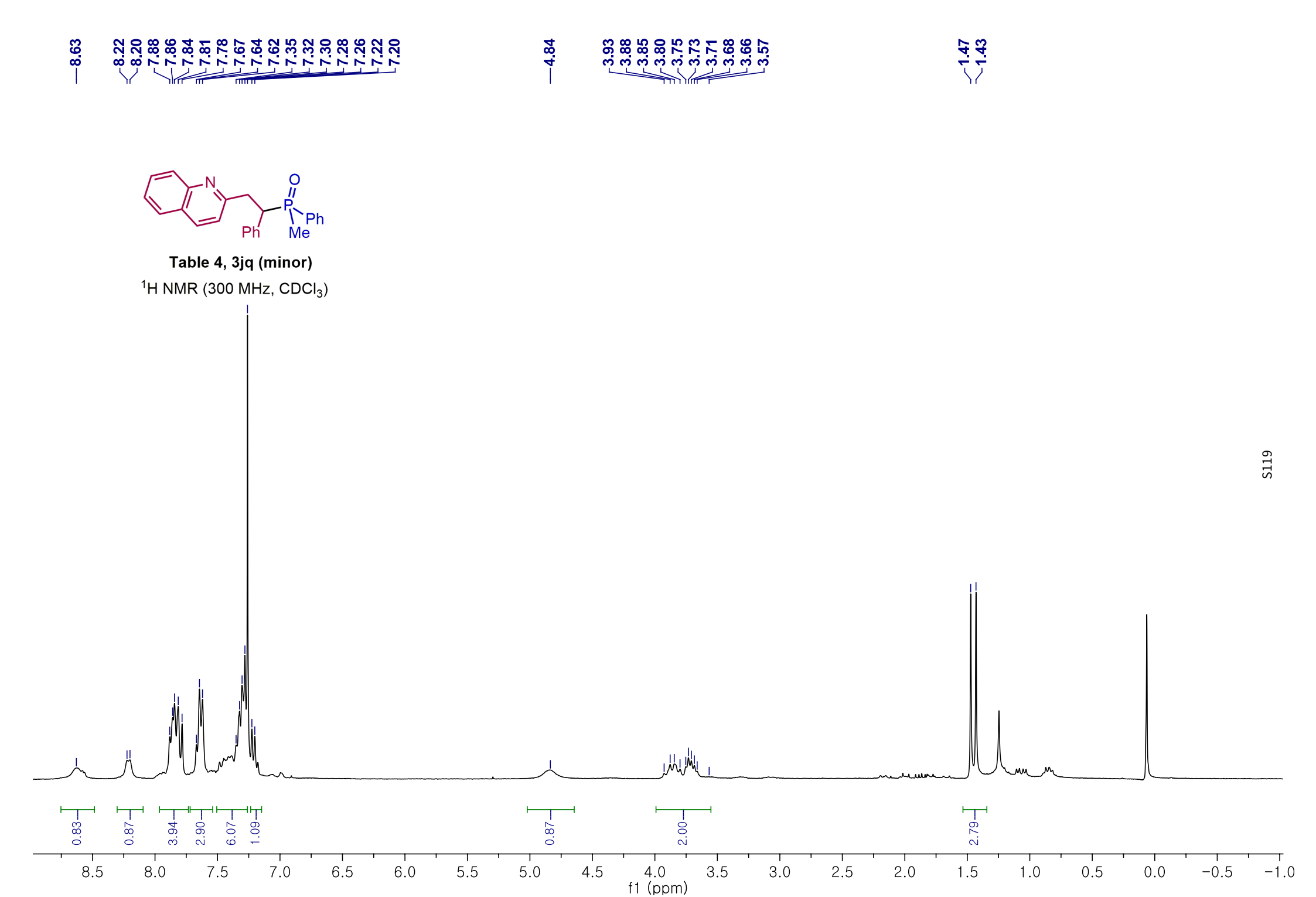
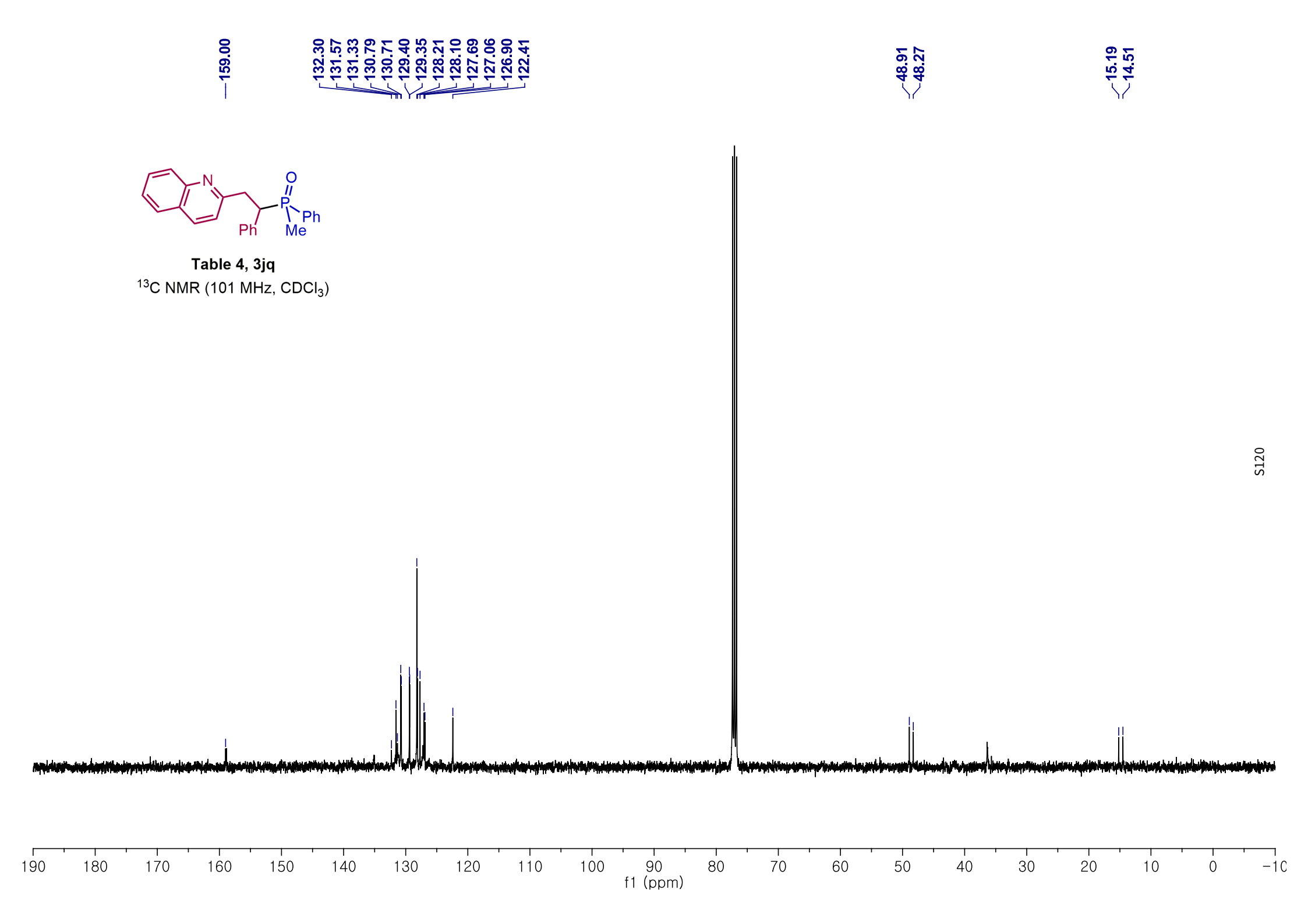


Table 4, 3jq (major)

^{1}H NMR (300 MHz, $CDCl_3$)




8.63
8.22
8.20
7.88
7.86
7.84
7.81
7.78
7.67
7.64
7.62
7.35
7.32
7.30
7.28
7.26
7.22
7.20
4.84
3.93
3.88
3.85
3.80
3.75
3.73
3.71
3.68
3.66
3.57
1.47
1.43
N
O
P
Ph
Me
Ph
Table 4, 3jq (minor)
1H NMR (300 MHz, CDCl3)
S119
0.83
0.87
3.94
2.90
6.07
1.09
0.87
2.00
2.79
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)


159.00
132.30
131.57
131.33
130.79
130.71
129.40
129.35
128.21
128.10
127.69
127.06
126.90
122.41
48.91
48.27
15.19
14.51
N
O
P
Ph
Me
Ph
Table 4, 3jq
13C NMR (101 MHz, CDCl3)
S120
f1 (ppm)

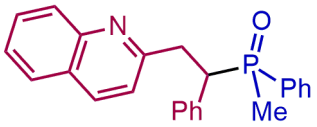
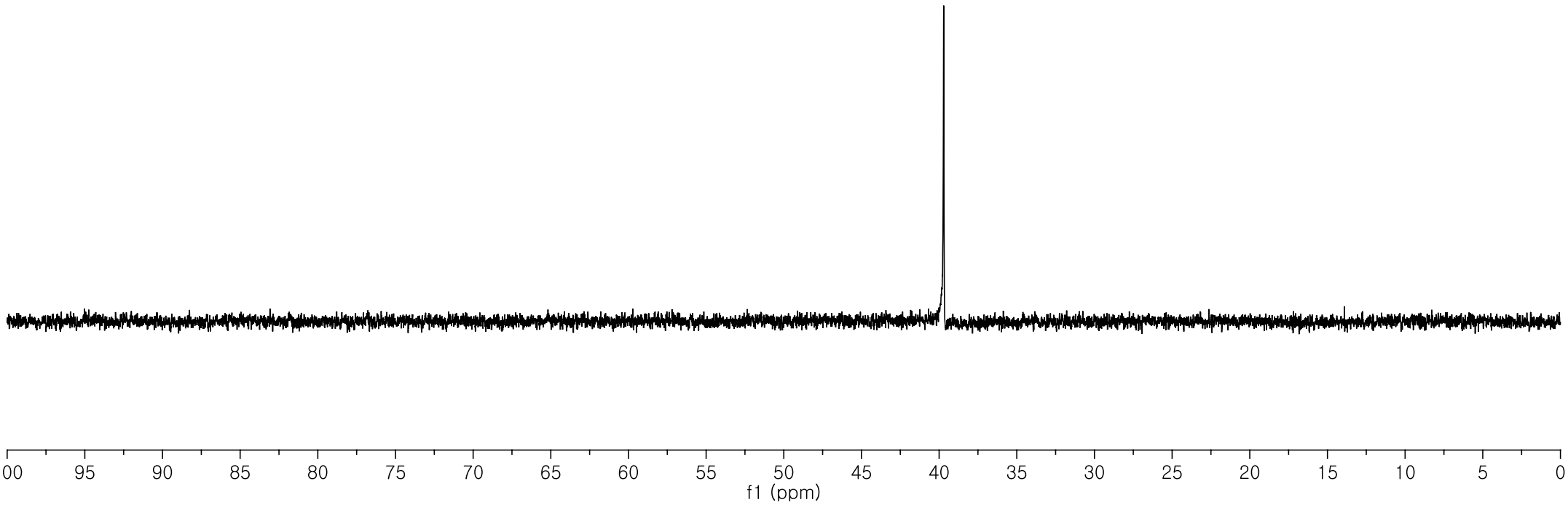


Table 4, 3jq (major)

^{31}P NMR (122 MHz, $CDCl_3$)



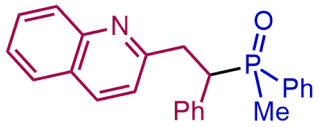
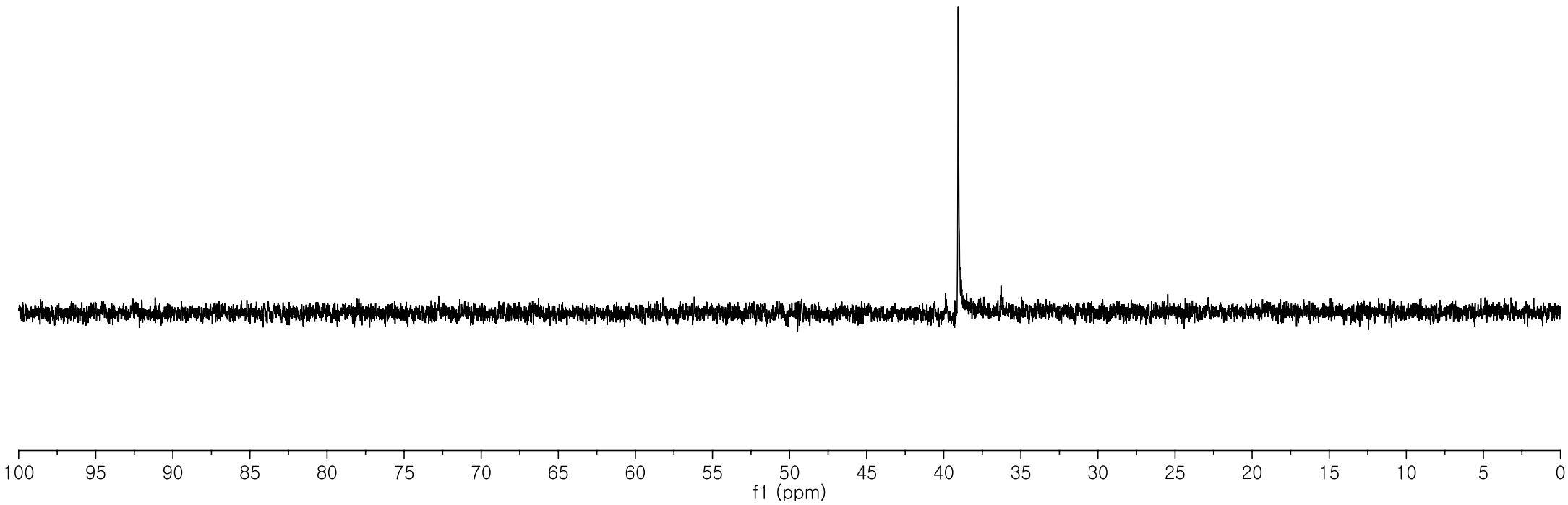


Table 4, 3jq (minor)

^{31}P NMR (122 MHz, $CDCl_3$)



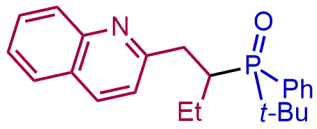


Table 4, 3kp

^{1}H NMR (300 MHz, $CDCl_3$)

S123

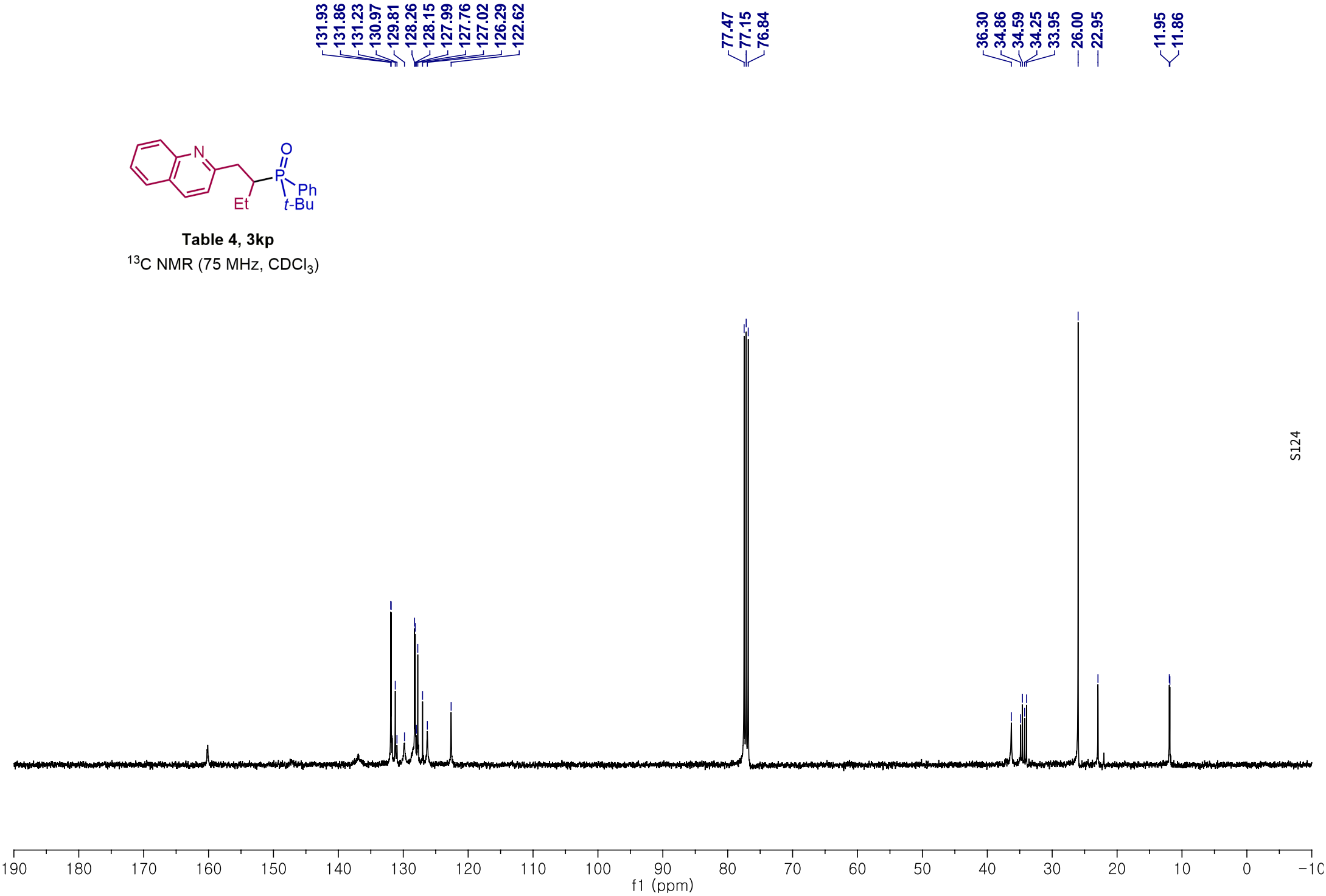


Table 4, 3kp

^{13}C NMR (75 MHz, $CDCl_3$)

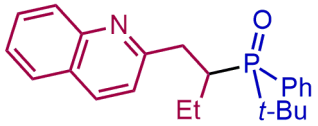
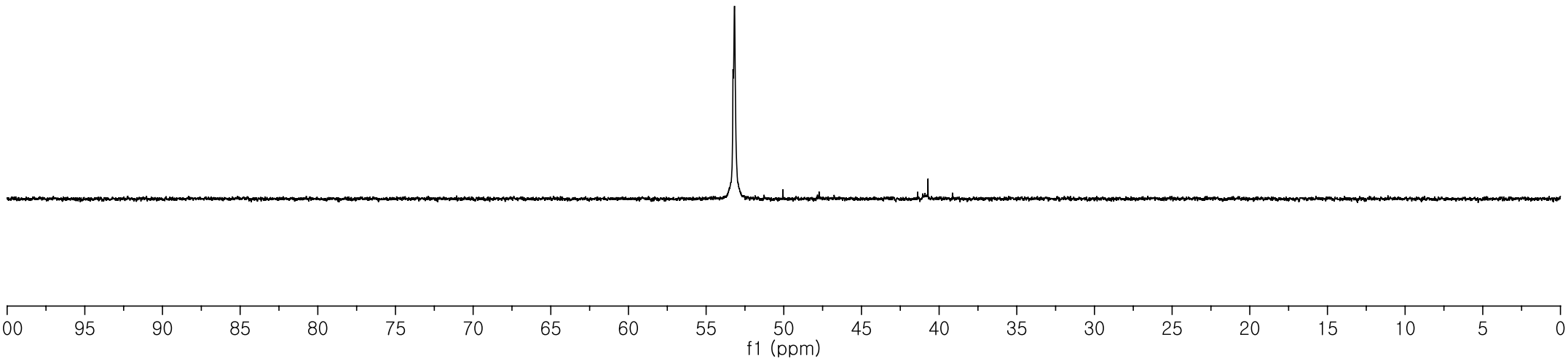
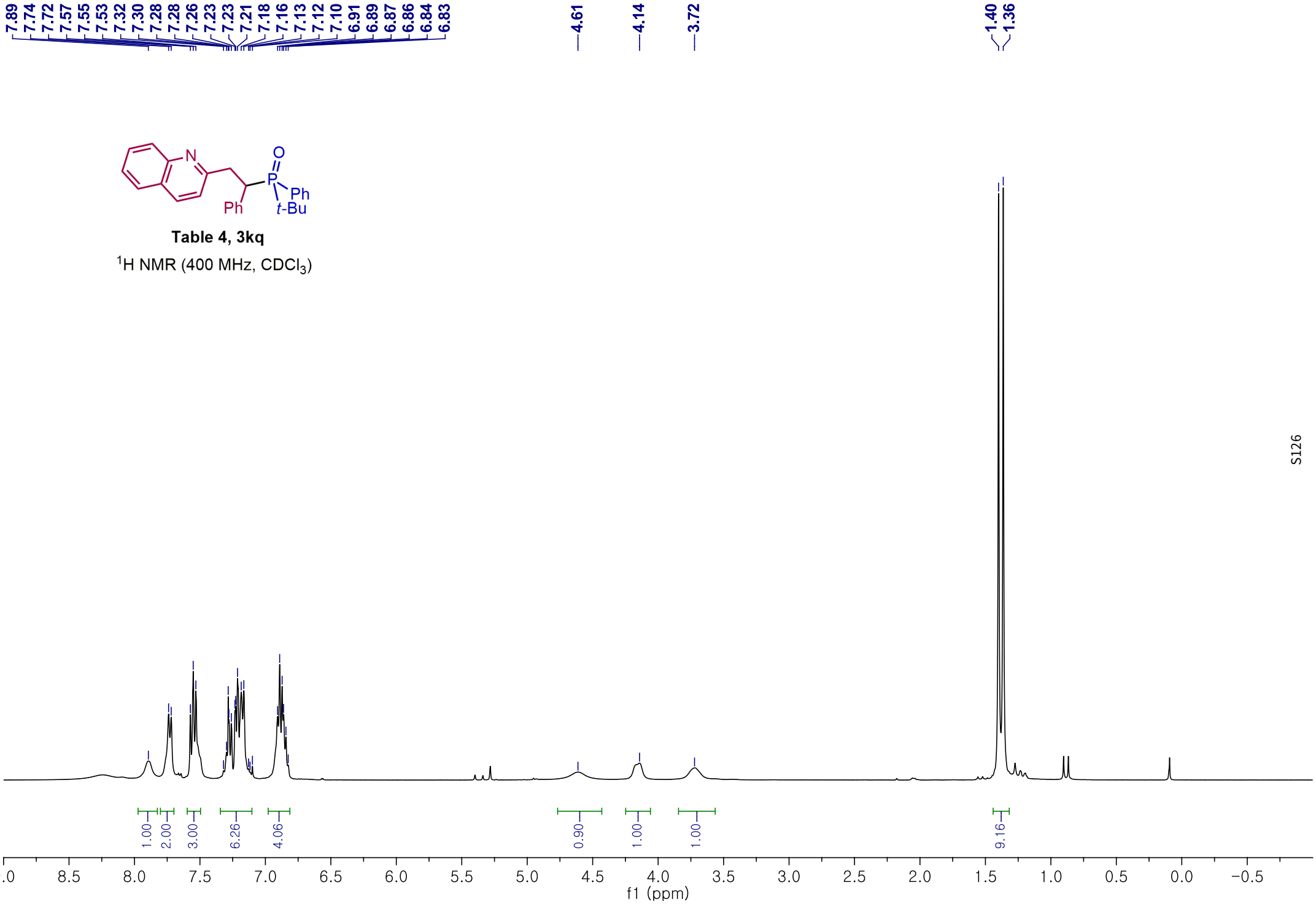
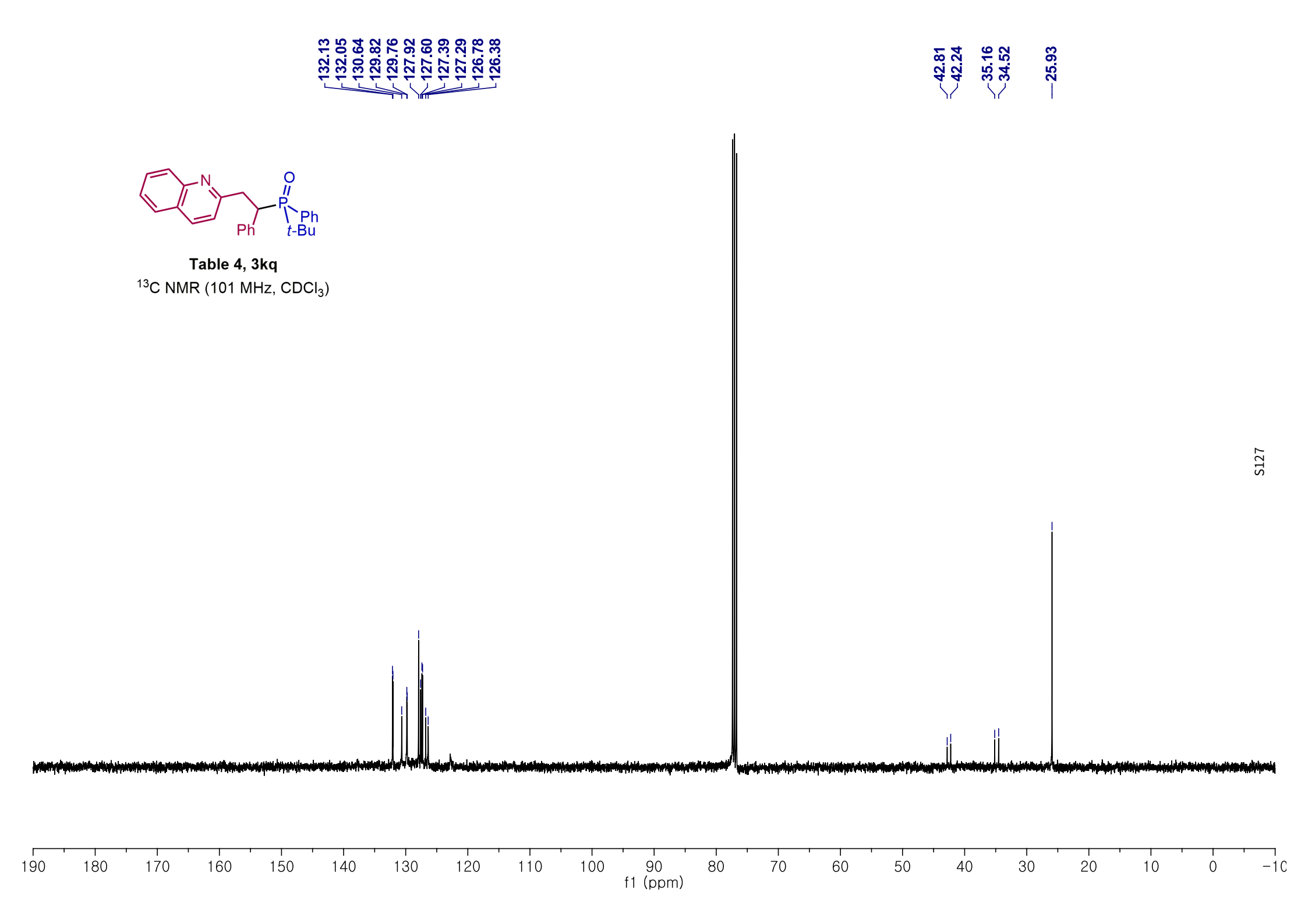


Table 4, 3kp

^{31}P NMR (122 MHz, $CDCl_3$)




7.89
7.74
7.72
7.57
7.55
7.53
7.32
7.30
7.28
7.28
7.26
7.23
7.23
7.21
7.18
7.16
7.13
7.12
7.10
6.91
6.89
6.87
6.86
6.84
6.83
4.61
4.14
3.72
1.40
1.36
N
O
P
Ph
t-Bu
Ph
Table 4, 3kq
1H NMR (400 MHz, CDCl3)
S126
1.00
2.00
3.00
6.26
4.06
0.90
1.00
1.00
9.16
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


132.13
132.05
130.64
129.82
129.76
127.92
127.60
127.39
127.29
126.78
126.38
42.81
42.24
35.16
34.52
25.93
N
O
P
Ph
Ph
t-Bu
Table 4, 3kq
13C NMR (101 MHz, CDCl3)
S127
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

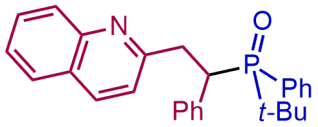
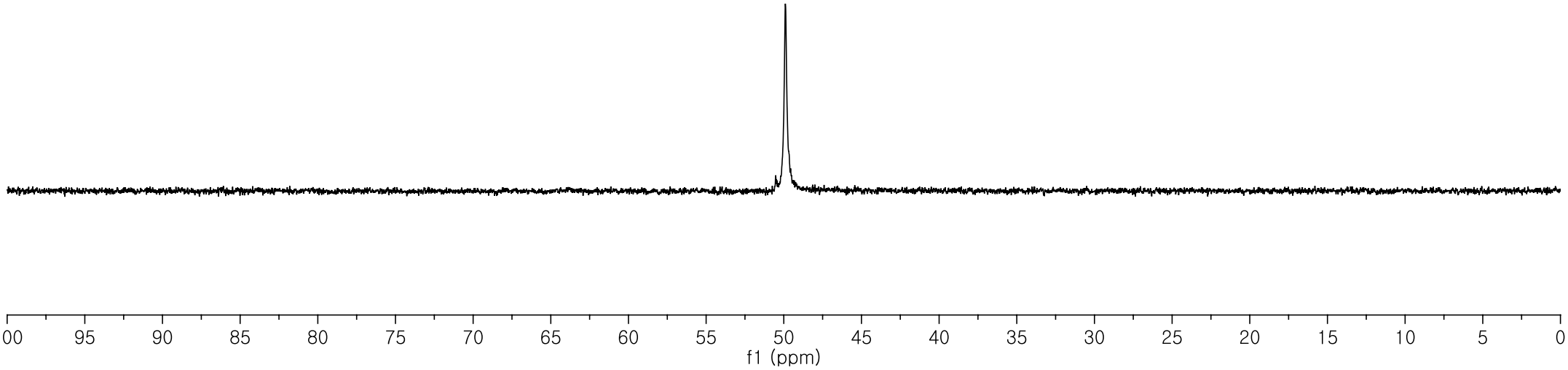
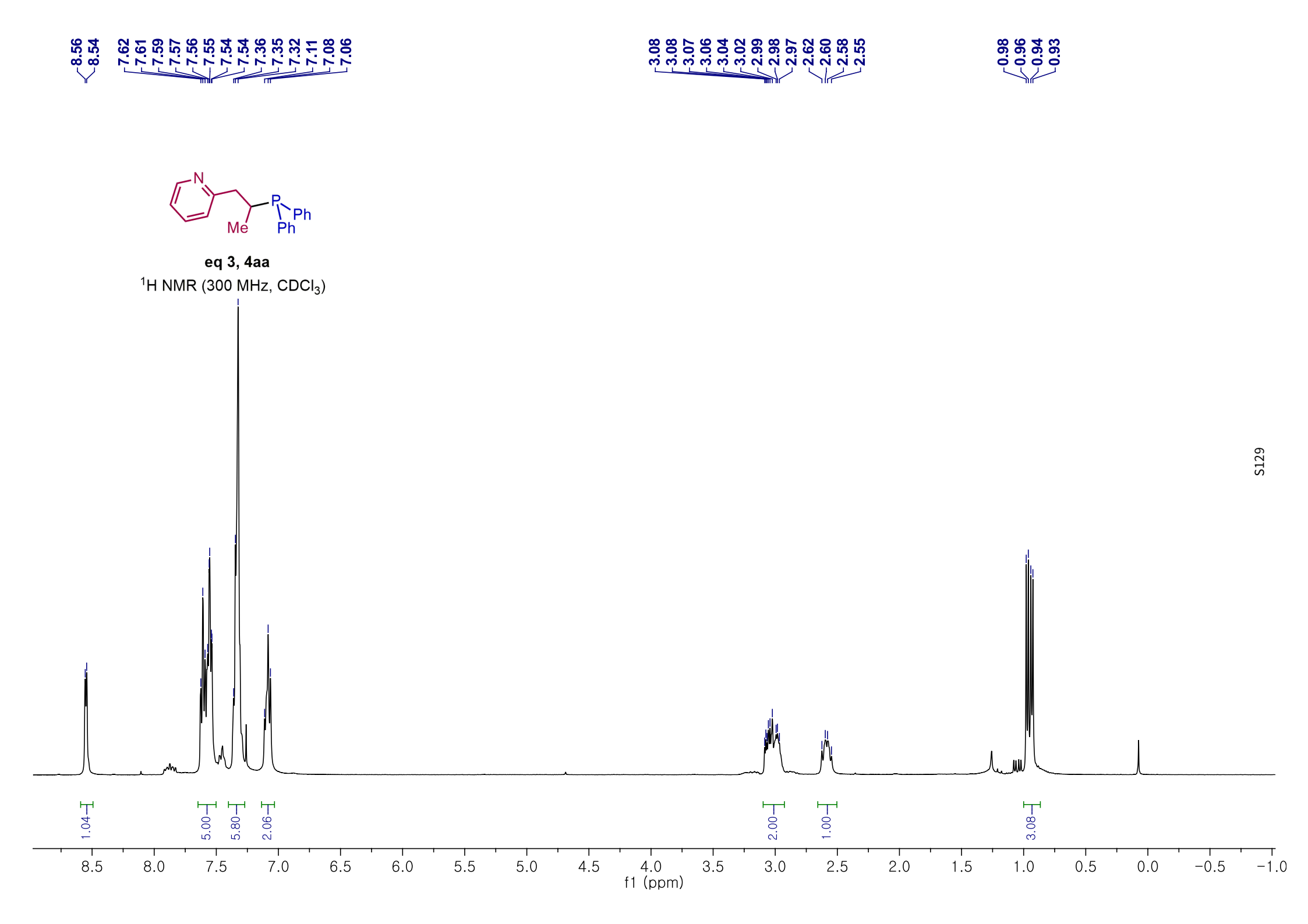
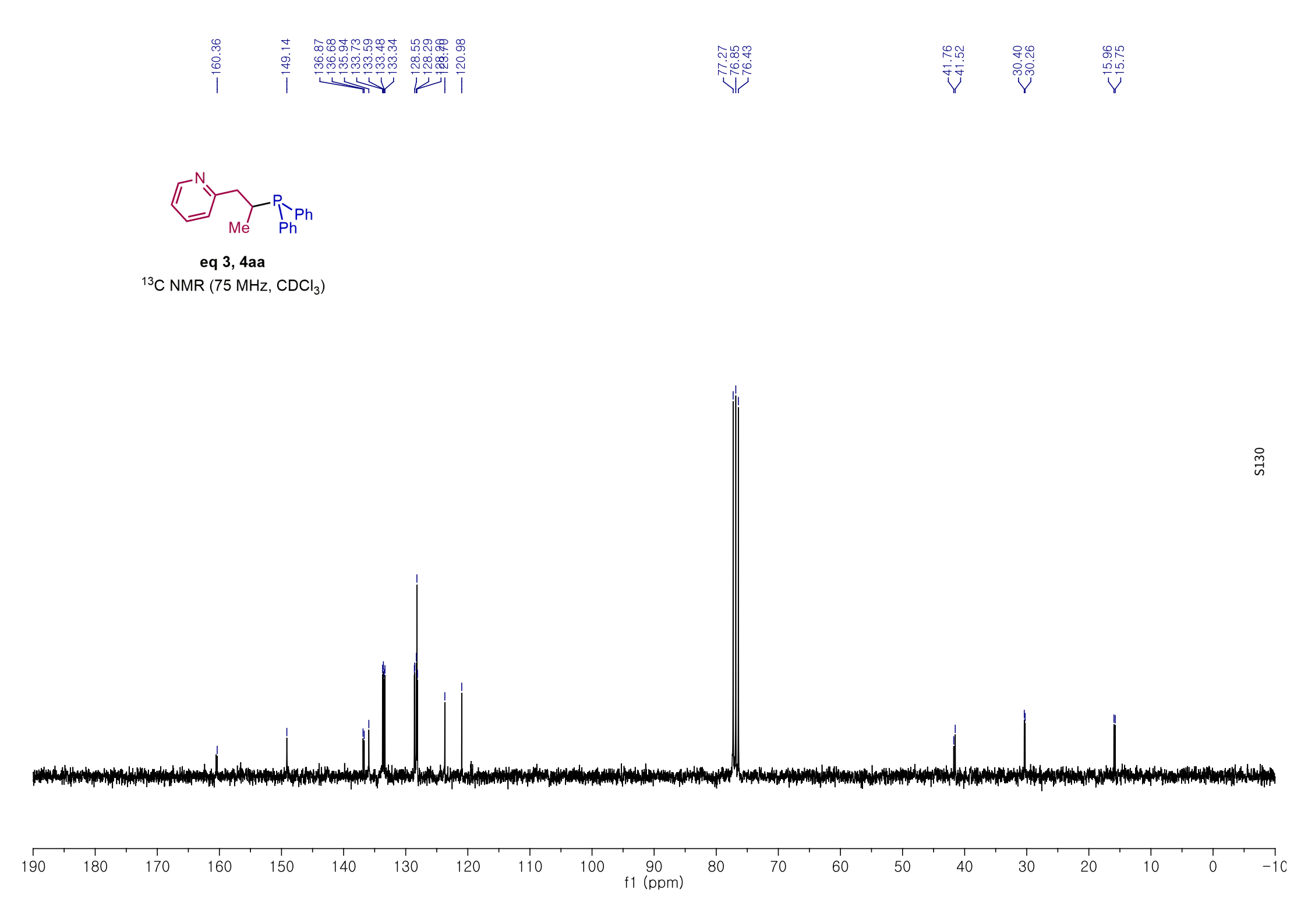


Table 4, 3kq

^{31}P NMR (162 MHz, $CDCl_3$)

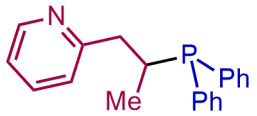



8.56
8.54
7.62
7.61
7.59
7.57
7.56
7.55
7.54
7.54
7.36
7.35
7.32
7.11
7.08
7.06
3.08
3.08
3.07
3.06
3.04
3.02
2.99
2.98
2.97
2.62
2.60
2.58
2.55
0.98
0.96
0.94
0.93
N
P
Ph
Ph
Me
eq 3, 4aa
1H NMR (300 MHz, CDCl3)
S129
1.04
5.00
5.80
2.06
2.00
1.00
3.08
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)



eq 3, 4aa

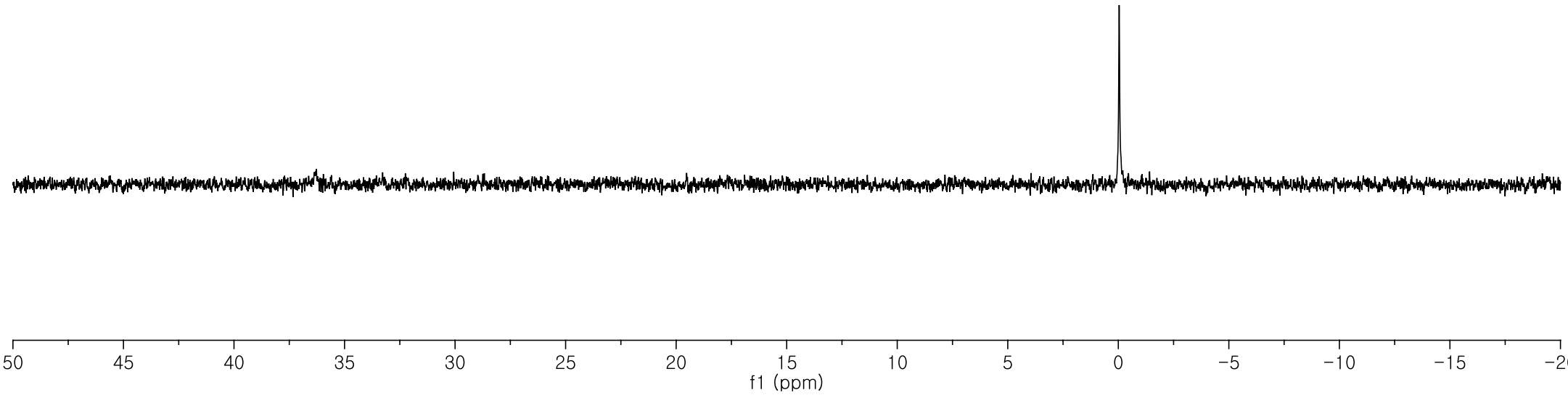
^{13}C NMR (75 MHz, $CDCl_3$)

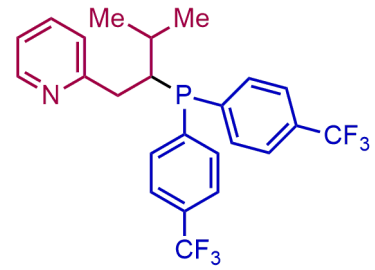


eq 3, 4aa

^{31}P NMR (122 MHz, $CDCl_3$)

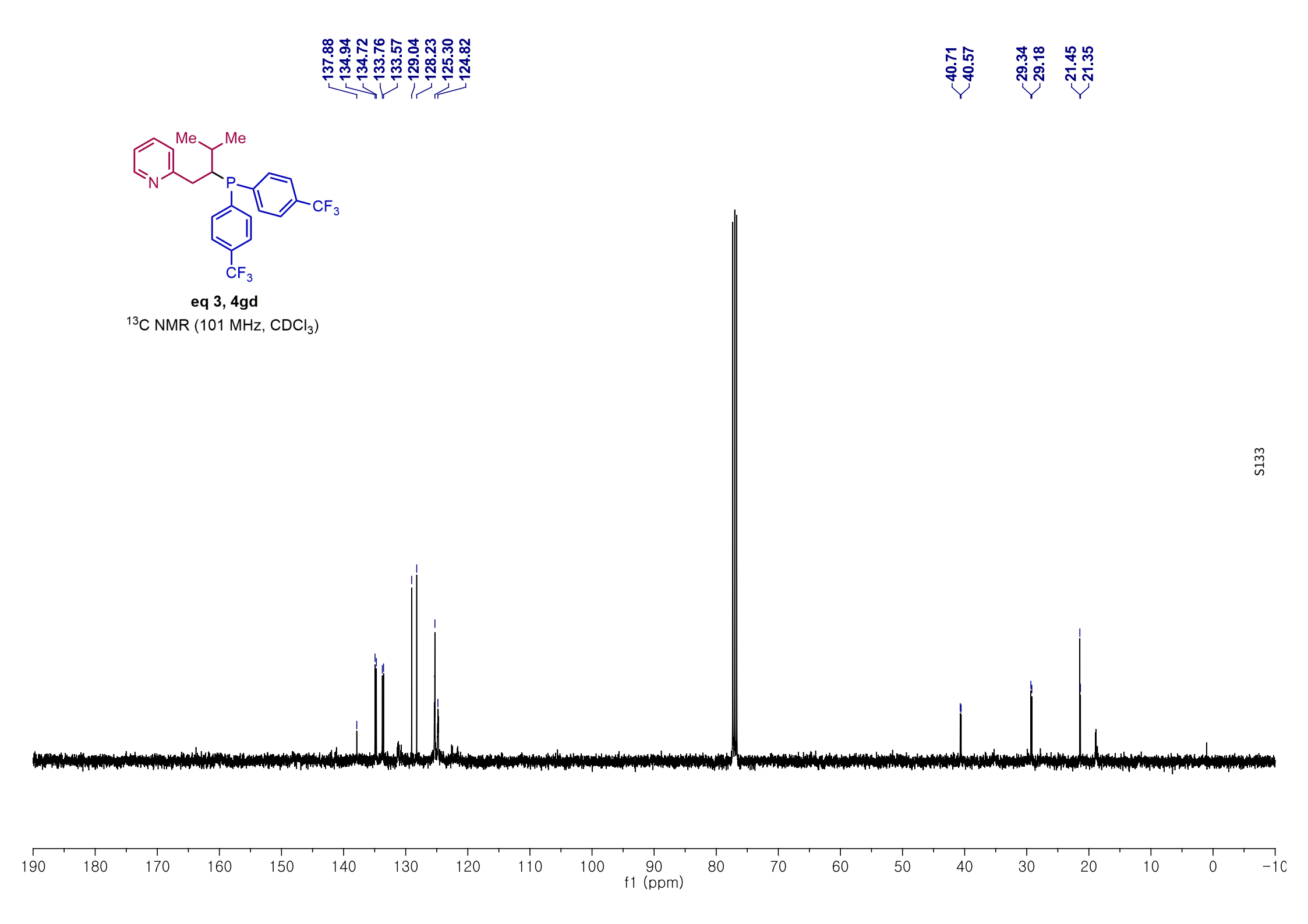
S131

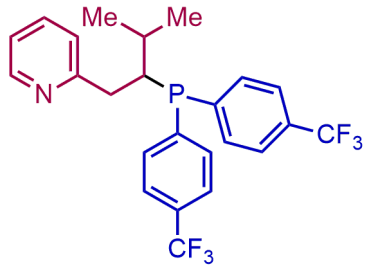




eq 3, 4gd

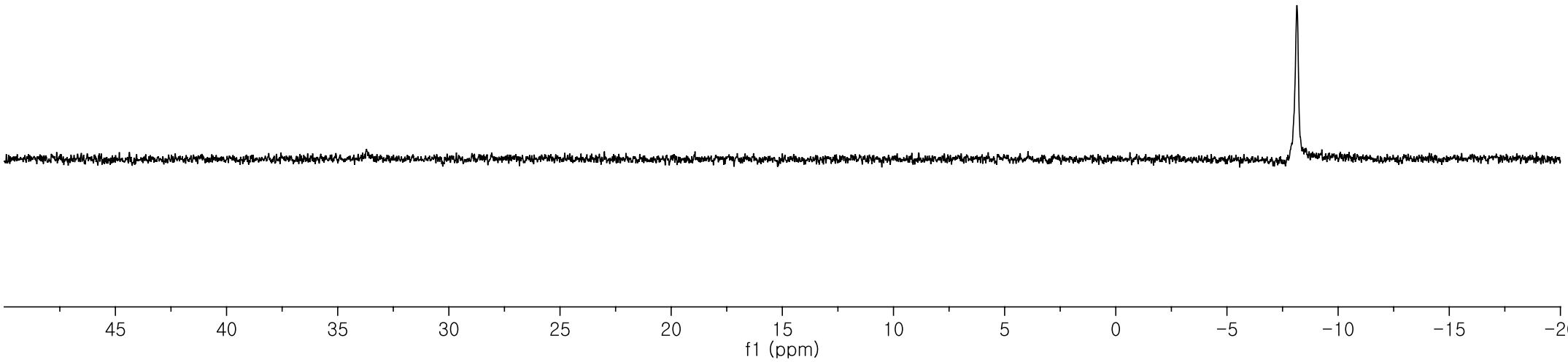
^{1}H NMR (400 MHz, $CDCl_3$)


137.88
134.94
134.72
133.76
133.57
129.04
128.23
125.30
124.82
40.71
40.57
29.34
29.18
21.45
21.35
Me
Me
N
P
CF3
CF3
eq 3, 4gd
13C NMR (101 MHz, CDCl3)
S133
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)



eq 3, 4gd

^{31}P NMR (122 MHz, $CDCl_3$)



VII. References

(1) Arvai, A. J.; Nielsen, C. ADSC Quantum-210 ADX Program, Area Detector System Corporation: Poway, CA, USA, 1983.
(2) Otwinowski, Z.; Minor, W. *Methods in Enzymology*; Carter Jr., C. W. Jr.; Sweet, R. M., Eds.; Academic Press: New York, 1997, vol. 276, part A, pp. 307–326.
(3) Sheldrick, G. M. SHELXT – Integrated space-group and crystal structure determination. *Acta Cryst.* **2015**, *A71*, 3–8.
(4) Sheldrick, G. M. Crystal structure refinement with SHELXL. *Acta. Cryst*. **2015**, *C71*, 3-8.
(5) Nie, S.-Z.; Davison, R. T.; Dong, V. M. Enantioselective Coupling of Dienes and Phosphine Oxides. *J. Am. Chem. Soc.* **2018**, *140*, 16450–16454.