

# Supporting Information

## In(OTf)<sub>3</sub>-Catalyzed *N*- $\alpha$ Phosphonylation of *N,O*-acetals with Triethyl phosphite

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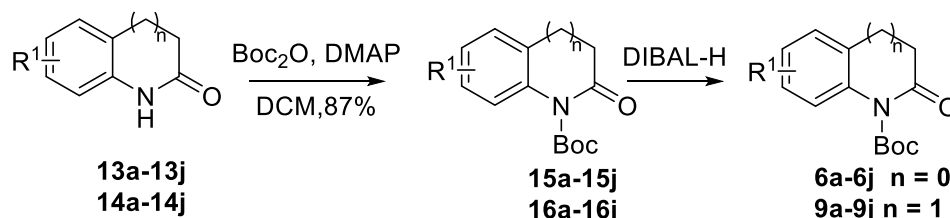
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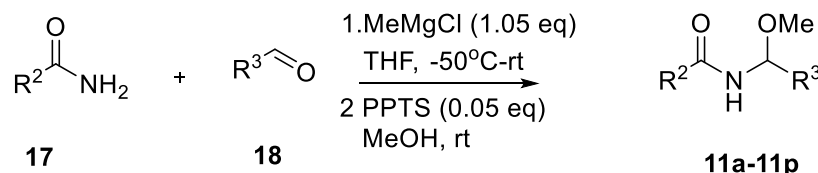
# 1. The synthetic details for the substrates 6a-6j, 9a-9j and 11a-11p.

## 1.1 General Procedure for the Synthesis of 6a-6j、9a-9j<sup>27b</sup>



To a solution of compound **13** or **14** (2.0 mmol) in dry DCM (10 mL) was added DMAP (0.2 mmol) and  $\text{Boc}_2\text{O}$  (5.0 mmol). After being stirred at room temperature for 12 h, the mixture was quenched with  $\text{NH}_4\text{Cl}$  and extracted with EtOAc (30 mL  $\times$  3). The combined organic layers were washed with brine, dried over  $\text{MgSO}_4$ , filtrated and concentrated. The residue was purified by flash chromatography on silica gel (PE/EA = 5:1) to give the desired product **15** or **16**. The compound **15** or **16** (0.5 mmol) was dissolved in dry THF (3 mL) under Ar atmosphere. Then DIBAL-H (1 M, 1.5 equiv) was dropped slowly. After being stirred at  $-78^\circ\text{C}$  for 2 h, the mixture was quenched with EtOAc and potassium sodium tartrate, and extracted with EtOAc (20 mL  $\times$  3). The combined organic layers were washed with brine, dried over  $\text{MgSO}_4$ , filtrated and concentrated, then obtained the crude product *N,O*-acetal **6** or **9** to be used without purification because of its unstability.

## 1.2 General Procedure for the Synthesis of 11a-11p<sup>28</sup>



First, amides **17** (10.0 mmol) were dissolved in anhydrous THF (15 mL) under an argon atmosphere, and the resulting solution was cooled at  $-50^\circ\text{C}$  for a while. Then  $\text{MeMgCl}$  (10.5 mmol, 3 M in THF) was added slowly under vigorous stirring, and the resulting mixture was allowed to warm to room temperature and stirred for 30 min.

Then the mixture was cooled to  $-50\text{ }^{\circ}\text{C}$ , and aldehydes (10.5 mmol) were added rapidly, and the resulting mixture was warmed to room temperature and stirred for 3 h. After completion of the reaction, the reaction was quenched with saturated aqueous  $\text{NaHCO}_3$  (50 mL). The organic layer was separated, and the aqueous phase was extracted with EtOAc (50 mL  $\times$  3); the combined organic layers were washed with brine, dried over anhydrous  $\text{MgSO}_4$  and filtered. The solvent was evaporated under reduced pressure to obtain crude hemiaminal.

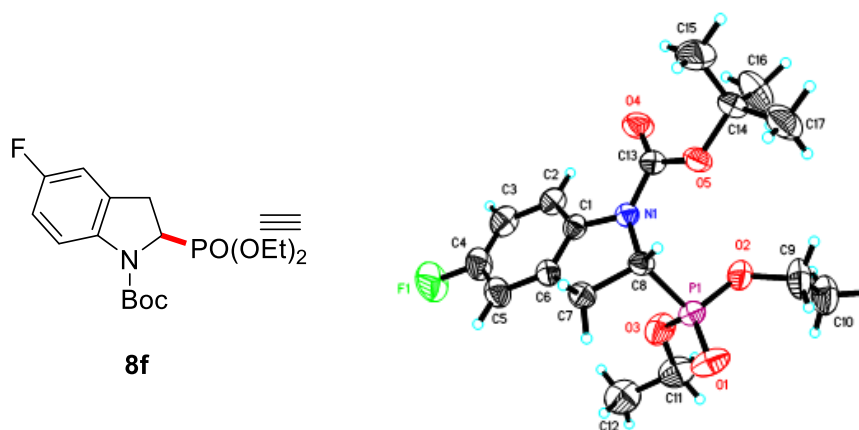
Next, the crude hemiaminal was dissolved in methanol (50 mL) at room temperature, and then pyridinium *p*-toluenesulfonate (0.1 mmol) was added. After the mixture was stirred for 3 h, the crude product was obtained using the same post-processing method described above, and then purified by flash chromatography with PE/EtOAc to afford the desired *N,O*-acetals **11a-11p**.

## 2 X-Ray Crystal data

### 2.1 X-Ray Crystal data of **8f**

ORTEP drawing of the X-ray crystallographic structure of compound **8f** (50% probability ellipsoids, dd21153 is the original test number for **8f**) The instrument is X-ray crystallographic diffractometer (Bruker/D8-VENTURE).

The single crystal of compound **8f** was prepared from its solution in petroleum ether/ethylacetate by slow evaporation of the solvent (**Table S1**)



**Table S1 . Crystal data and structure refinement for 8f**

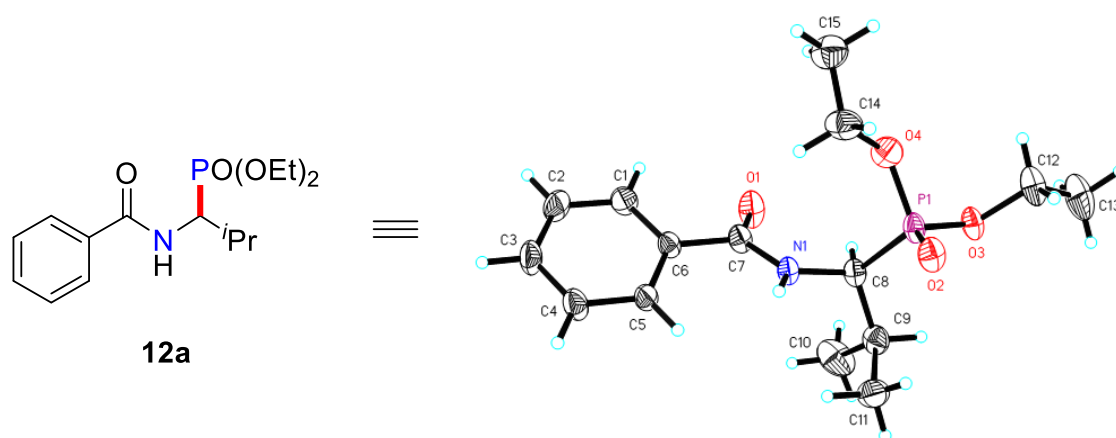
Table 1. Identification code	<b>8f</b>	
Empirical formula	C <sub>15</sub> H <sub>24</sub> N O <sub>4</sub> P	
Formula weight	313.32	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 10.2351(3) Å	a = 90 °
	b = 8.1643(2) Å	b =
	c = 20.5765(5) Å	g = 90 °
Volume	1702.00(8) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.223 Mg/m <sup>3</sup>	
Absorption coefficient	0.176 mm <sup>-1</sup>	
F(000)	672	
Crystal size	0.200 x 0.140 x 0.110 mm <sup>3</sup>	
Theta range for data collection	2.627 to 25.999 °	
Index ranges	-12 ≤ h ≤ 12, -10 ≤ k ≤ 10, -25 ≤ l ≤ 25	
Reflections collected	26255	
Independent reflections	6651 [R(int) = 0.0384]	
Completeness to theta = 25.242 °	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.6920	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	6651 / 83 / 425	
Goodness-of-fit on F <sup>2</sup>	1.049	
Final R indices [I > 2σ(I)]	R1 = 0.0536, wR2 = 0.1411	
R indices (all data)	R1 = 0.0588, wR2 = 0.1477	
Absolute structure parameter	0.20(3)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.579 and -0.310 e.Å <sup>-3</sup>	

CCDC **2175609**. For detailed crystallographic data, please refer to the Cambridge Crystallographic Data Centre at <http://ccdc.cam.ac.uk>.

## 2.2 X-Ray Crystal data of **12a**

ORTEP drawing of the X-ray crystallographic structure of compound **12a** (50% probability ellipsoids, dd21111 is the original test number for **12a**) The instrument is X-ray crystallographic diffractometer (Bruker/D8-VENTURE).

The single crystal of compound **12a** was prepared from its solution in petroleum ether/ethylacetate by slow evaporation of the solvent (**Table S2**)



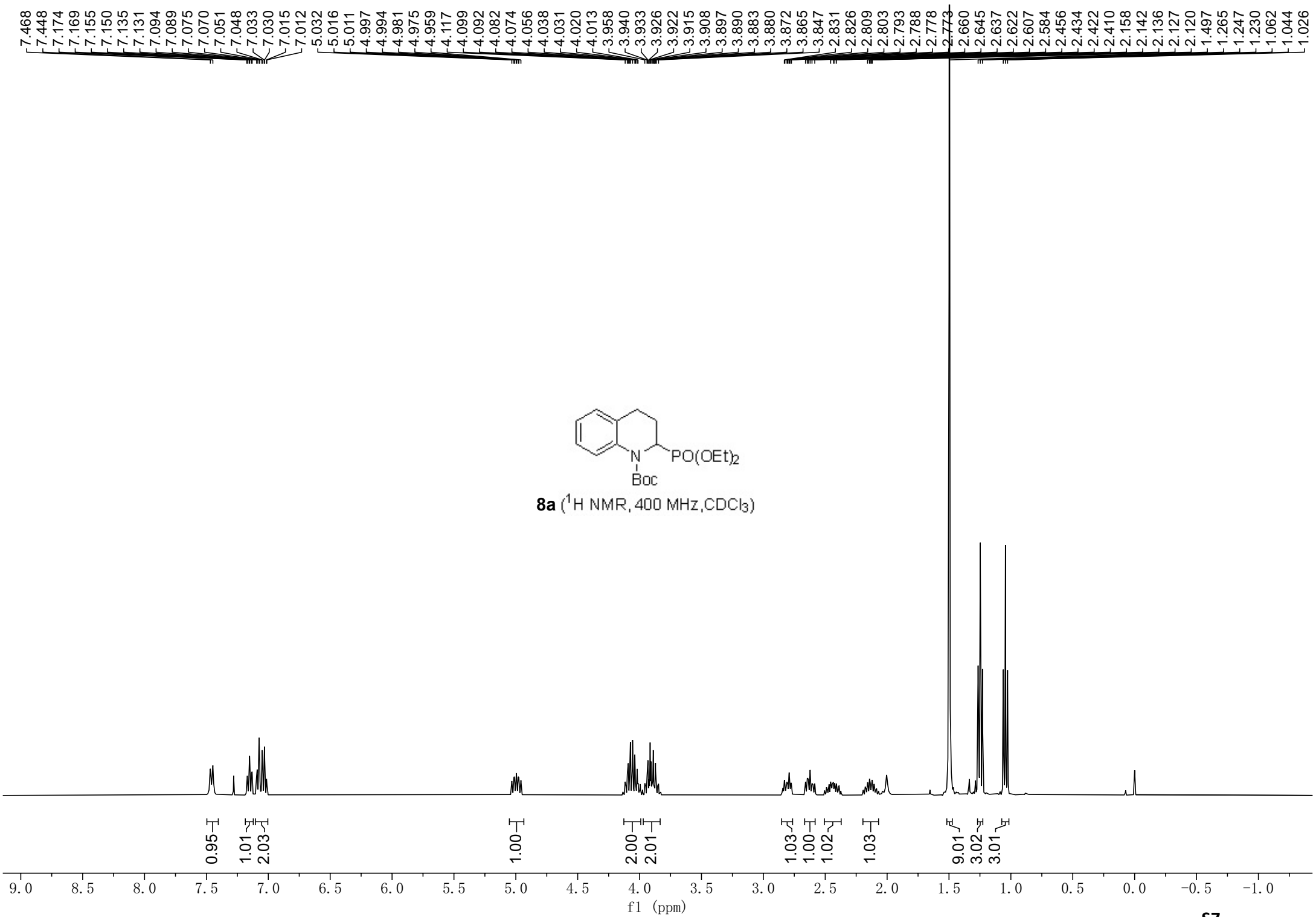
**Table S2 . Crystal data and structure refinement for **12a****

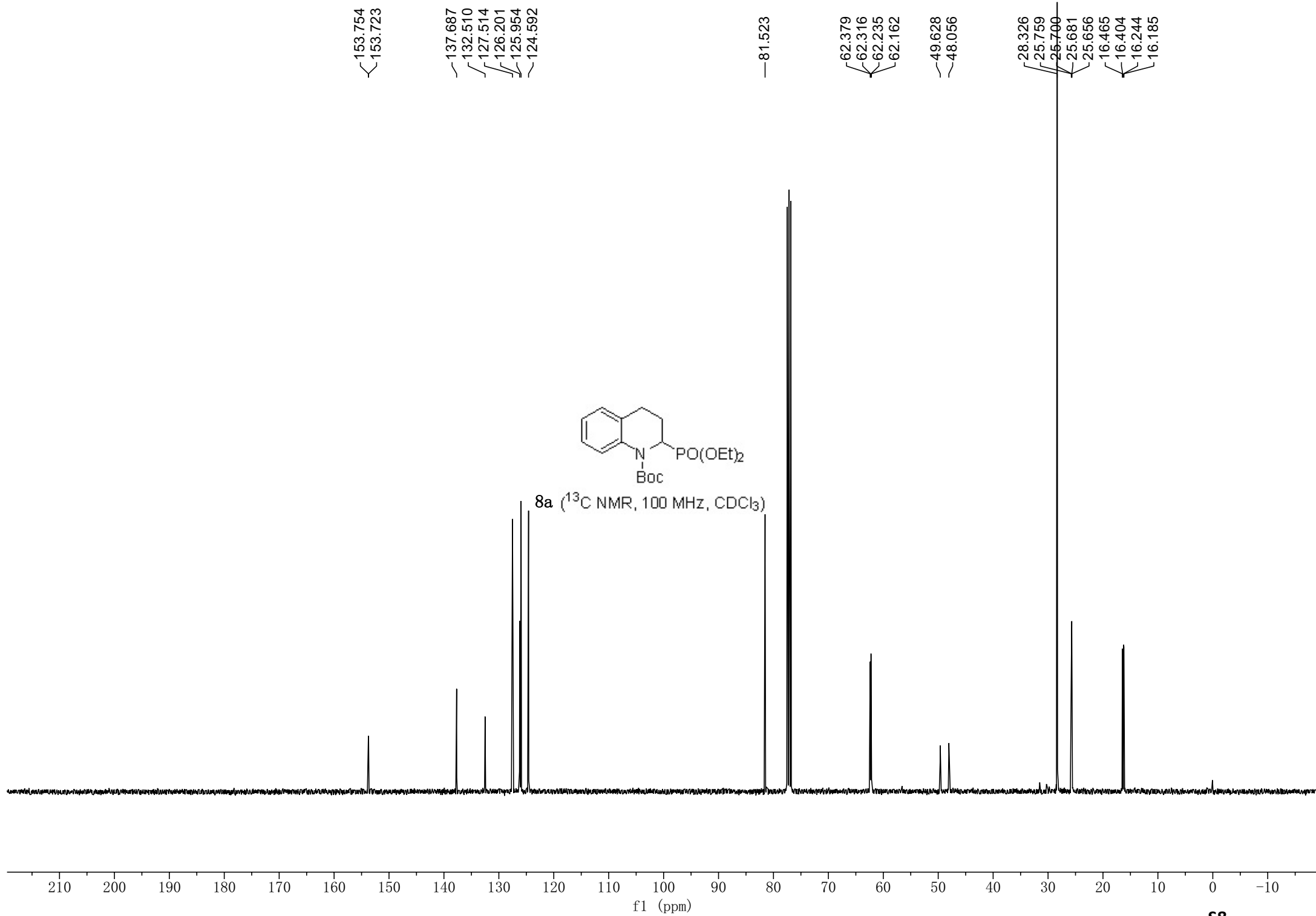
Identification code	<b>12a</b>	
Empirical formula	C <sub>17</sub> H <sub>25</sub> F N O <sub>5</sub> P	
Formula weight	373.35	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.2680(4) Å	α = 76.990(2) °
	b = 9.7857(5) Å	β =
	c = 11.9313(6) Å	γ = 77.696(2) °
Volume	969.02(8) Å <sup>3</sup>	
Z	2	

Density (calculated)	1.280 Mg/m <sup>3</sup>
Absorption coefficient	0.176 mm <sup>-1</sup>
F(000)	396
Crystal size	0.200 x 0.120 x 0.060 mm <sup>3</sup>
Theta range for data collection	2.483 to 25.999 °
Index ranges	-11<=h<=11, -12<=k<=12, -14<=l<=13
Reflections collected	16343
Independent reflections	3793 [R(int) = 0.0667]
Completeness to theta = 25.242 °	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.4652
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	3793 / 83 / 270
Goodness-of-fit on F <sup>2</sup>	1.023
Final R indices [I>2sigma(I)]	R1 = 0.0549, wR2 = 0.1272
R indices (all data)	R1 = 0.0844, wR2 = 0.1493
Extinction coefficient	0.050(7)
Largest diff. peak and hole	0.244 and -0.215 e.Å <sup>-3</sup>

CCDC **2175608**. For detailed crystallographic data, please refer to the Cambridge Crystallographic Data Centre at <http://ccdc.cam.ac.uk>.

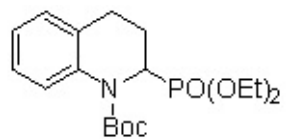
### 3. Copies of NMR Spectrum



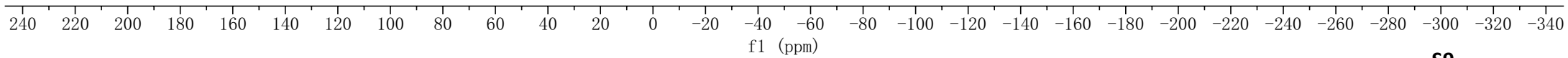


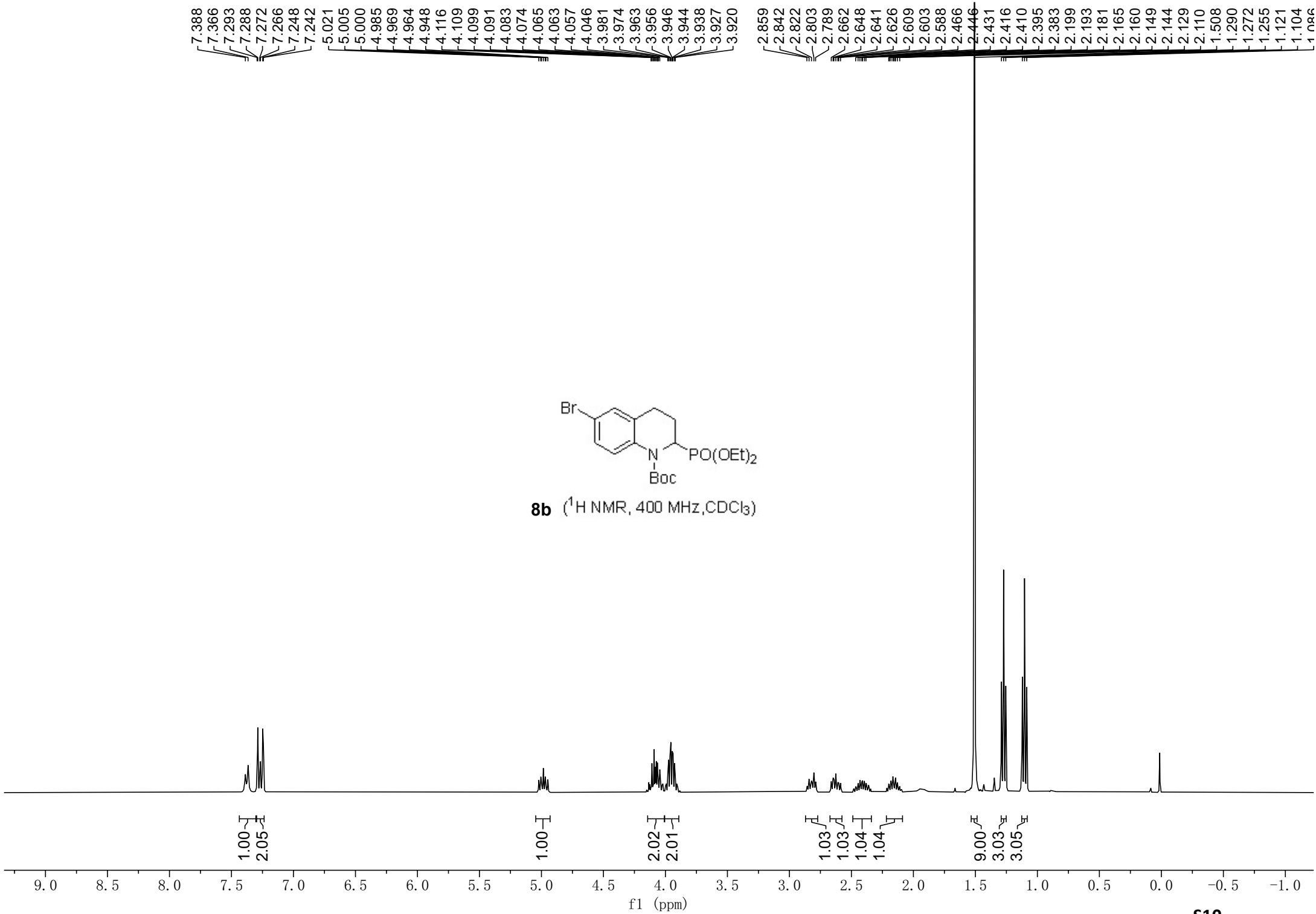


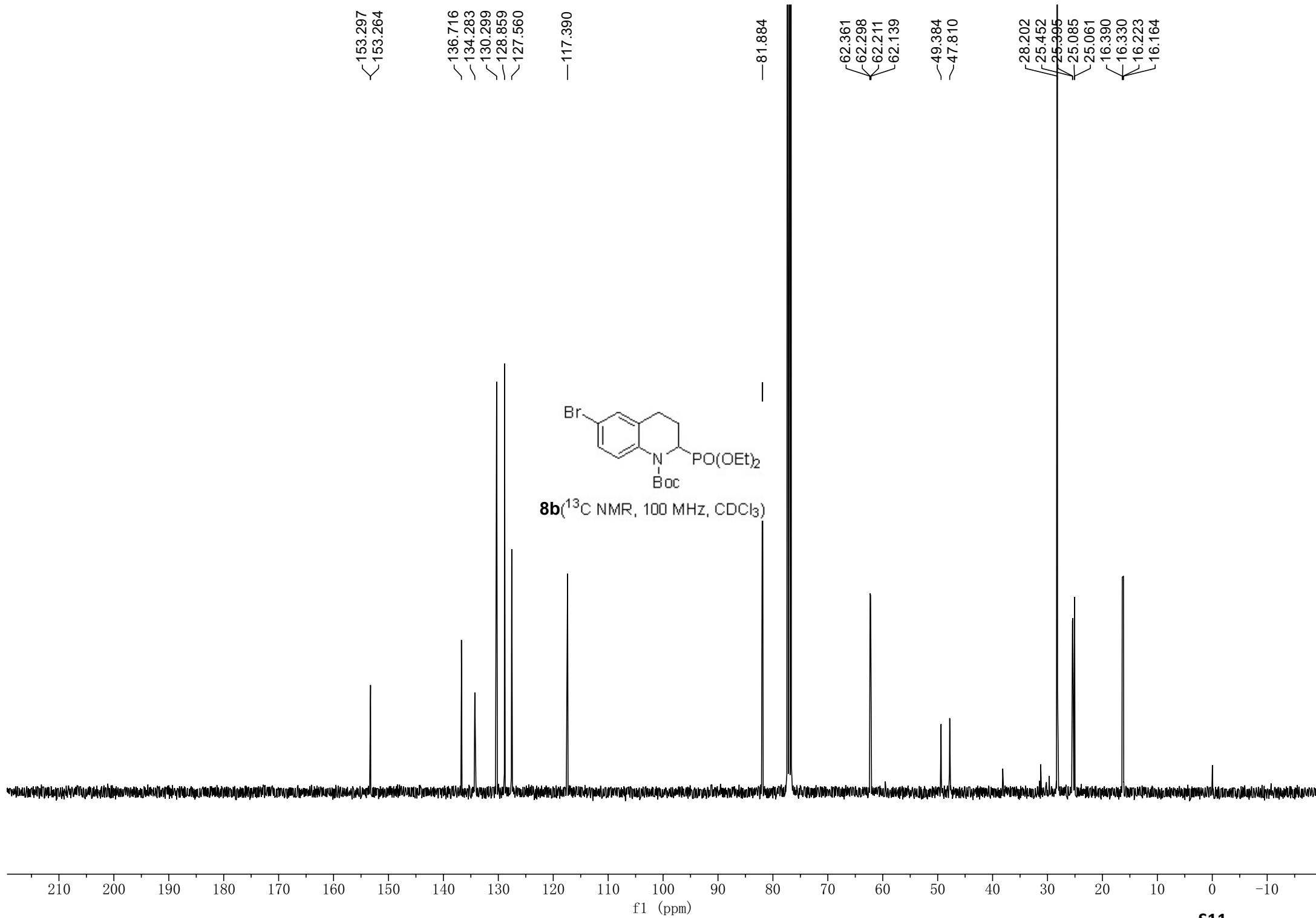
—24.167



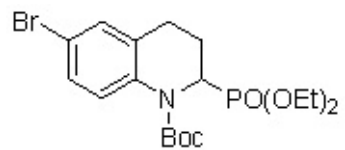
**8a** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )



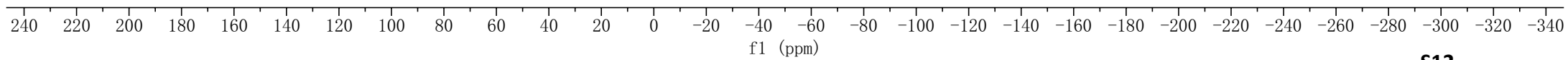


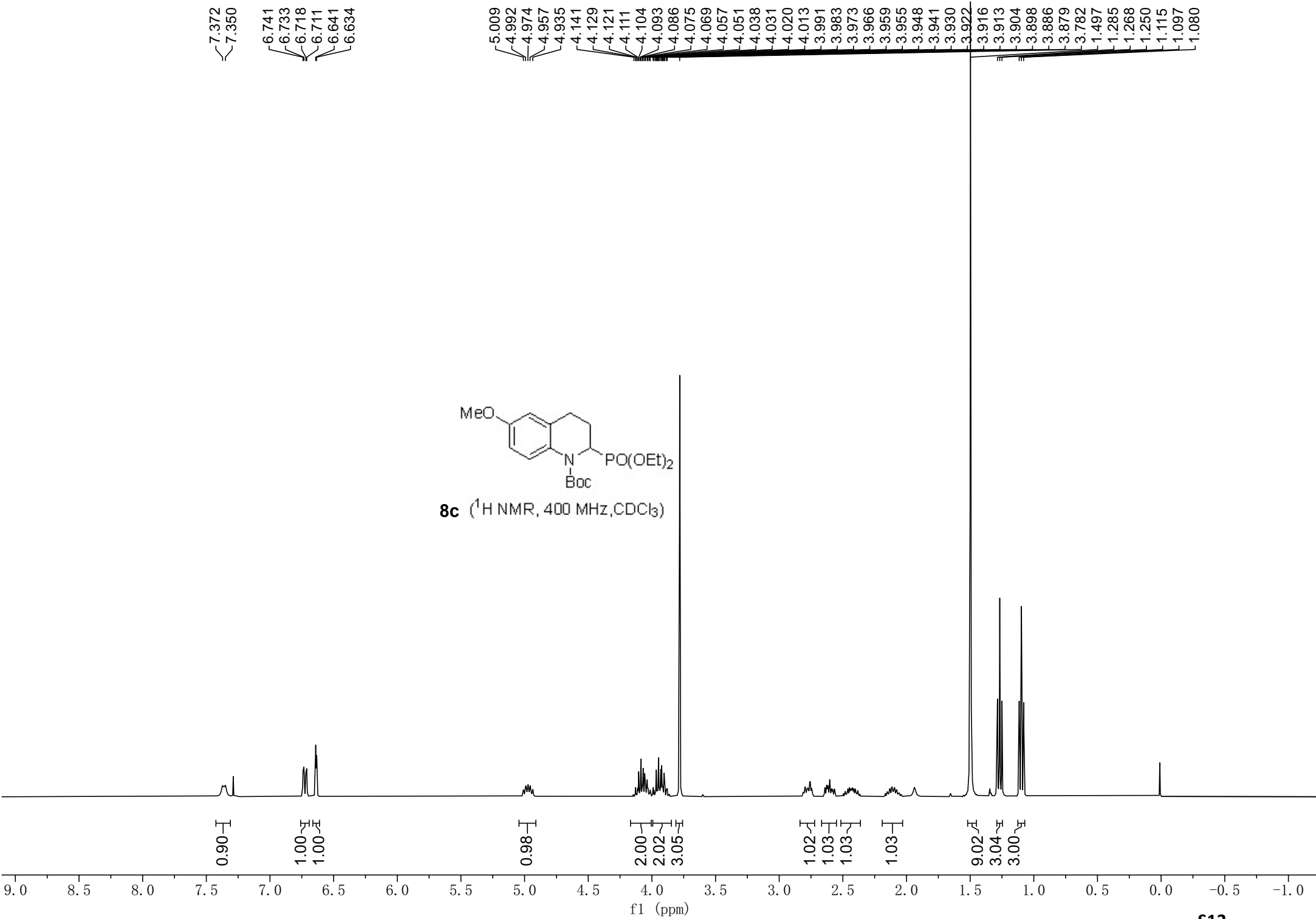


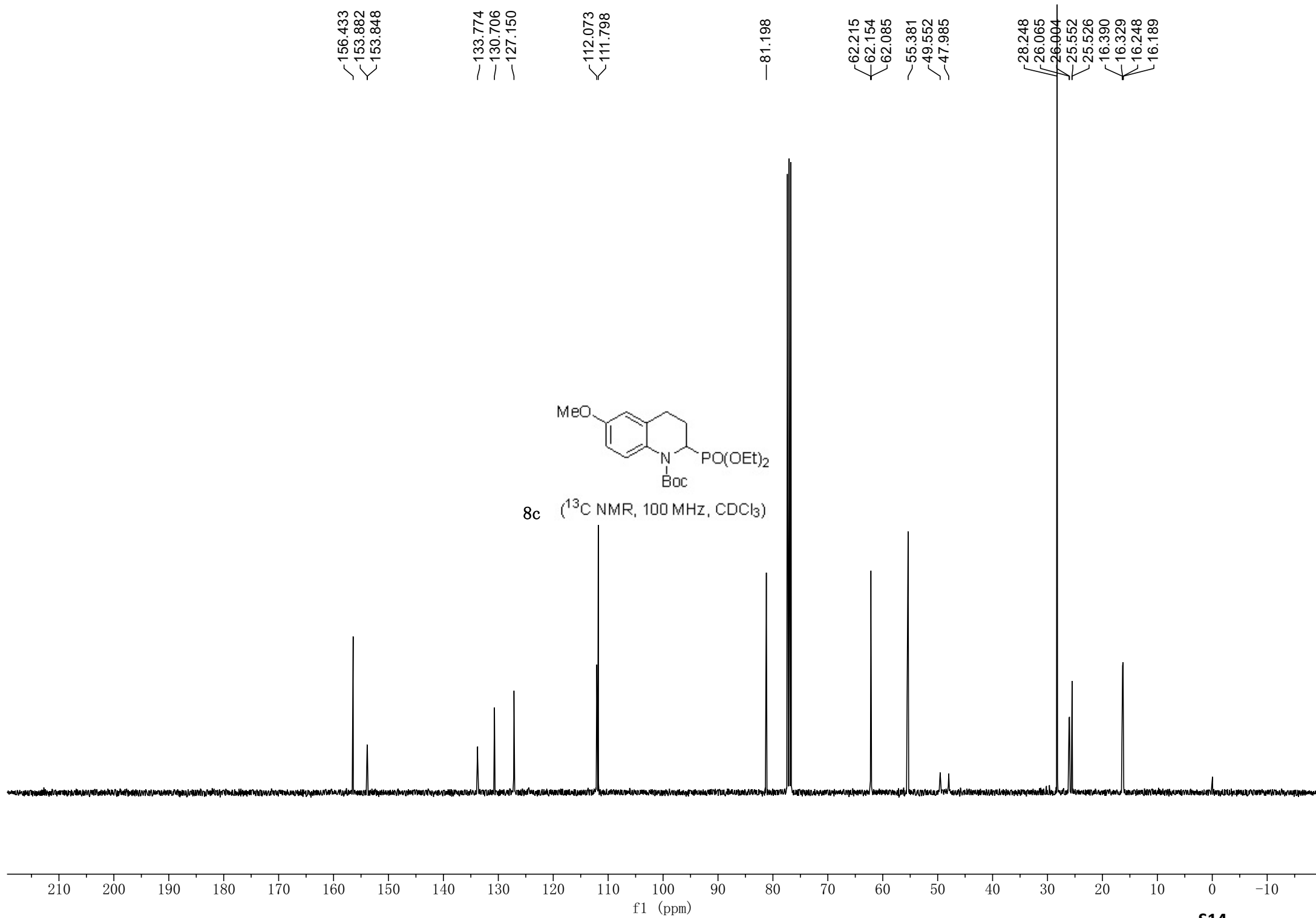
—23.705



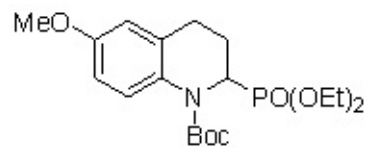
**8b** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



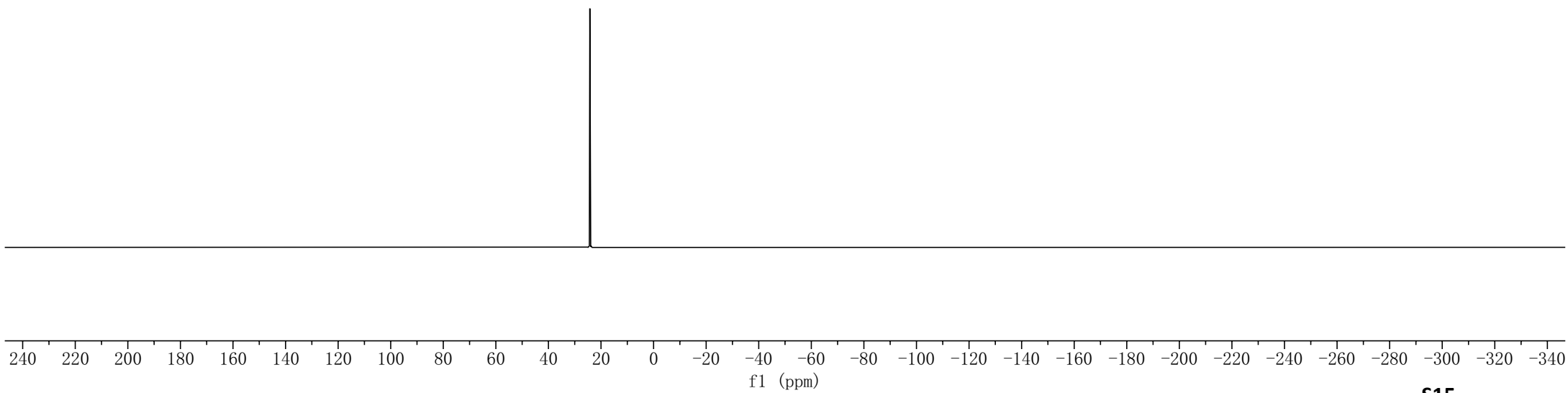


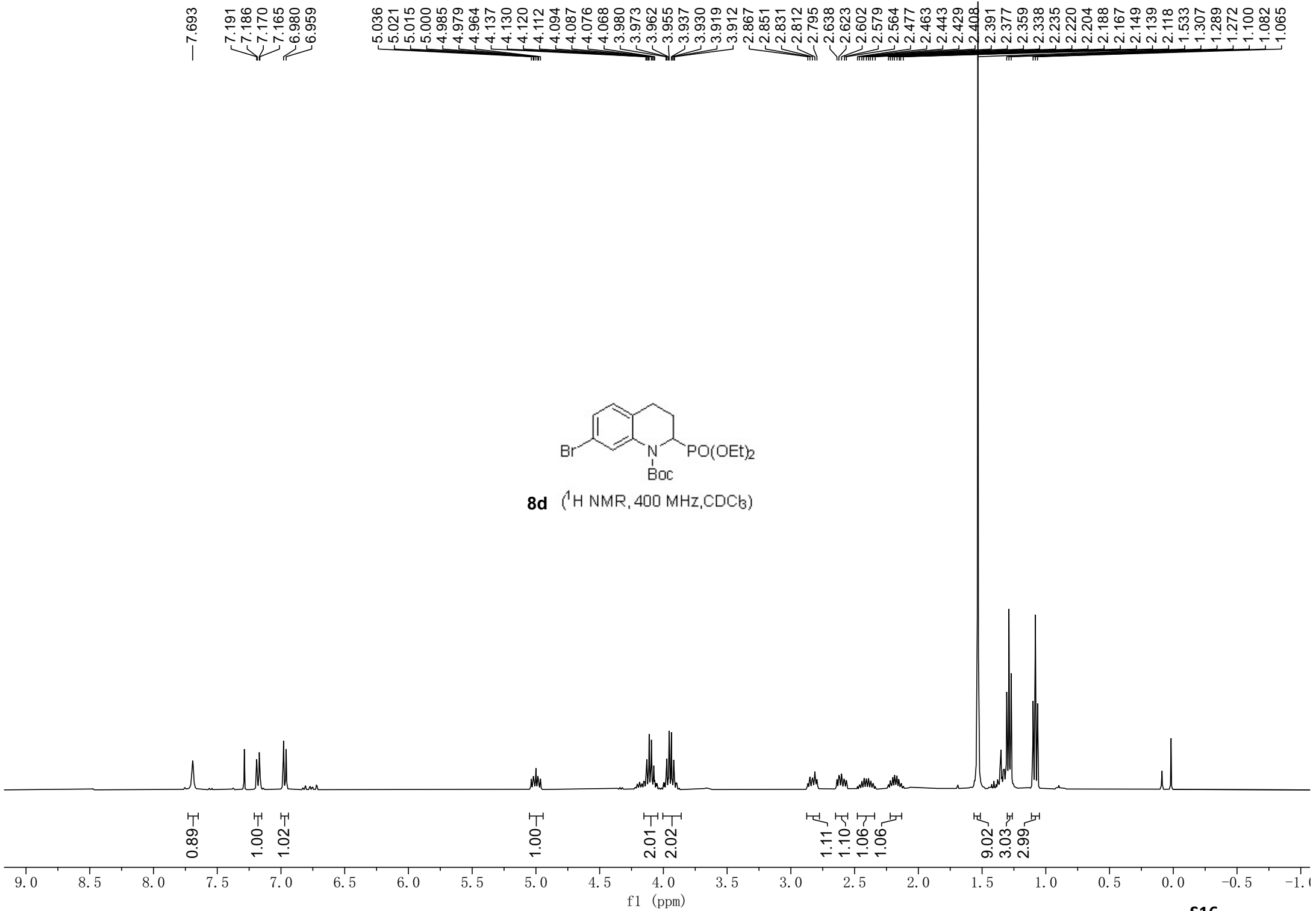


—24.253

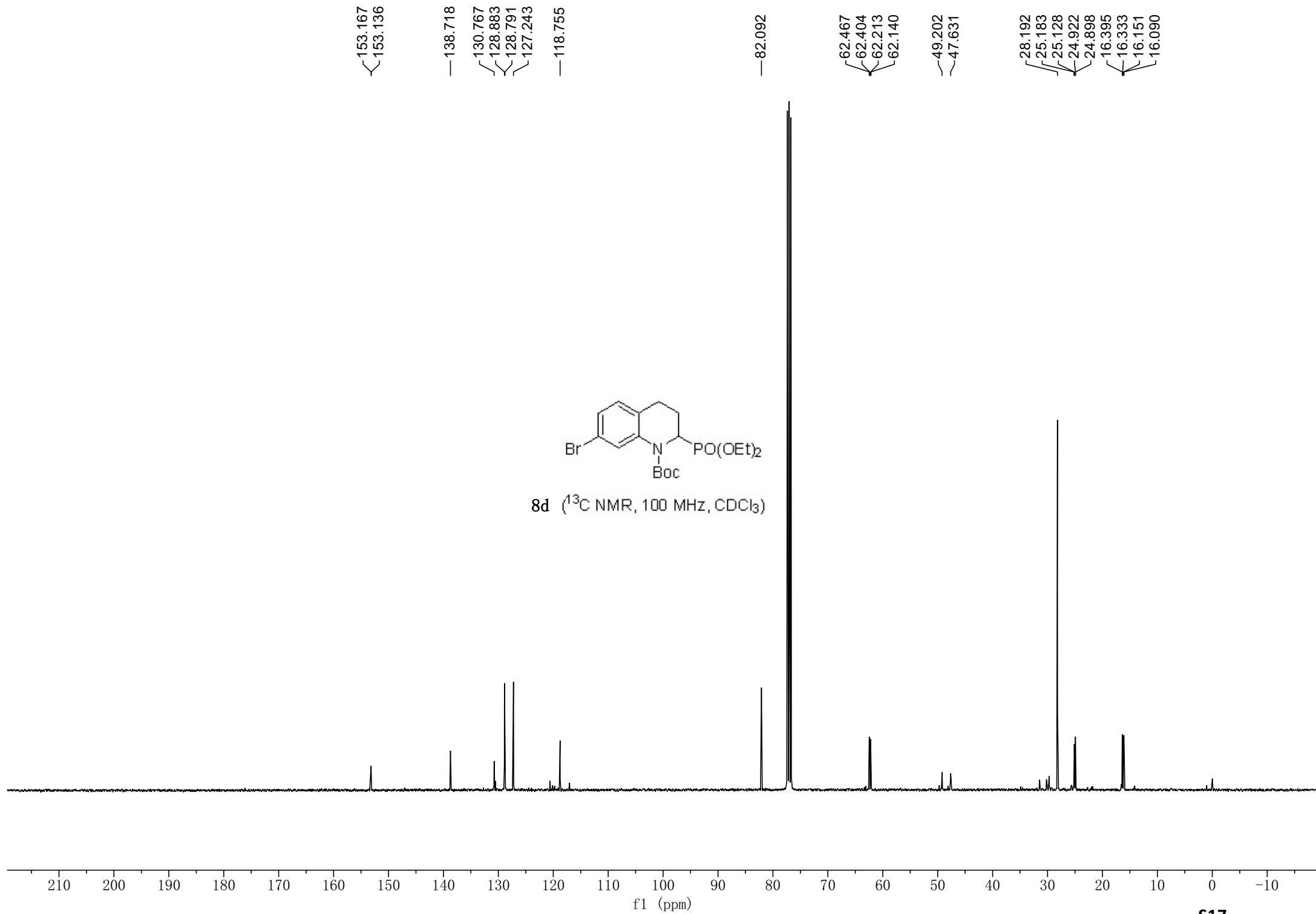


8c (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)

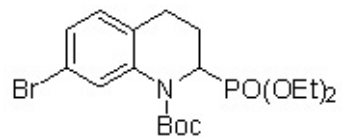




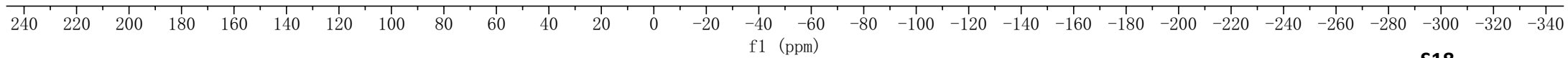


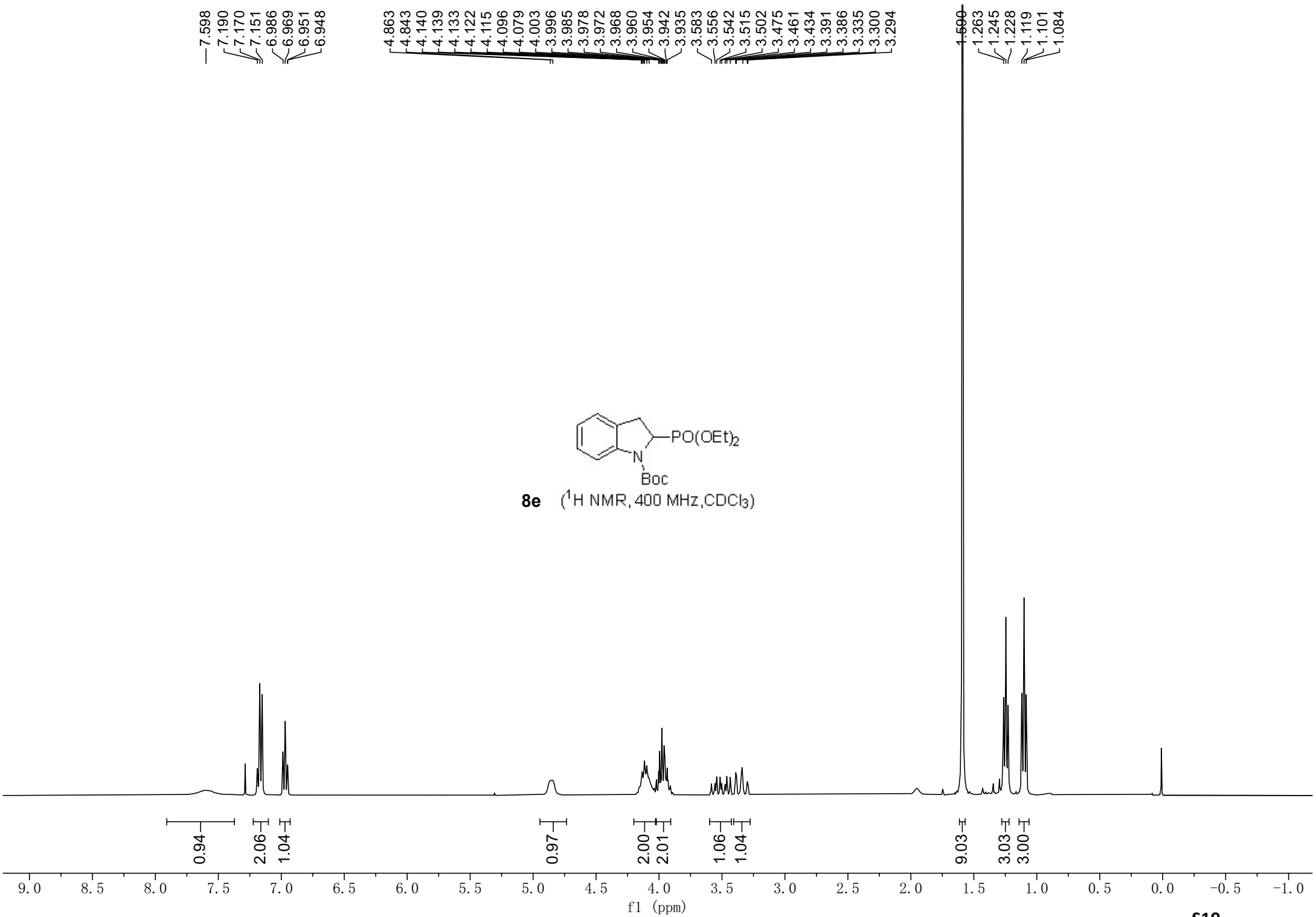


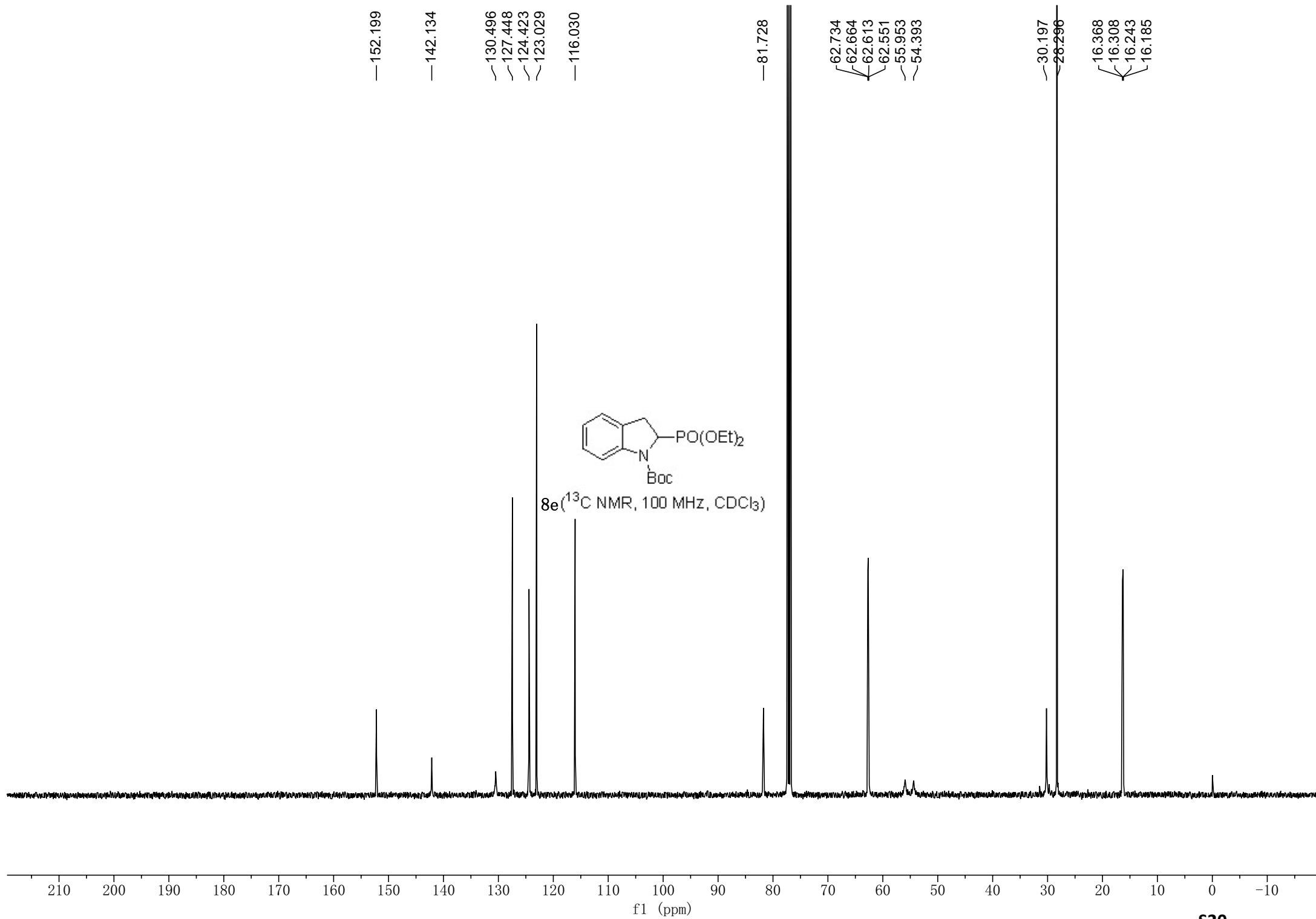
—23.655



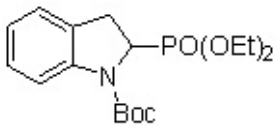
8d (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



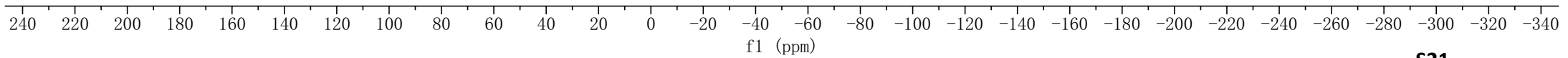


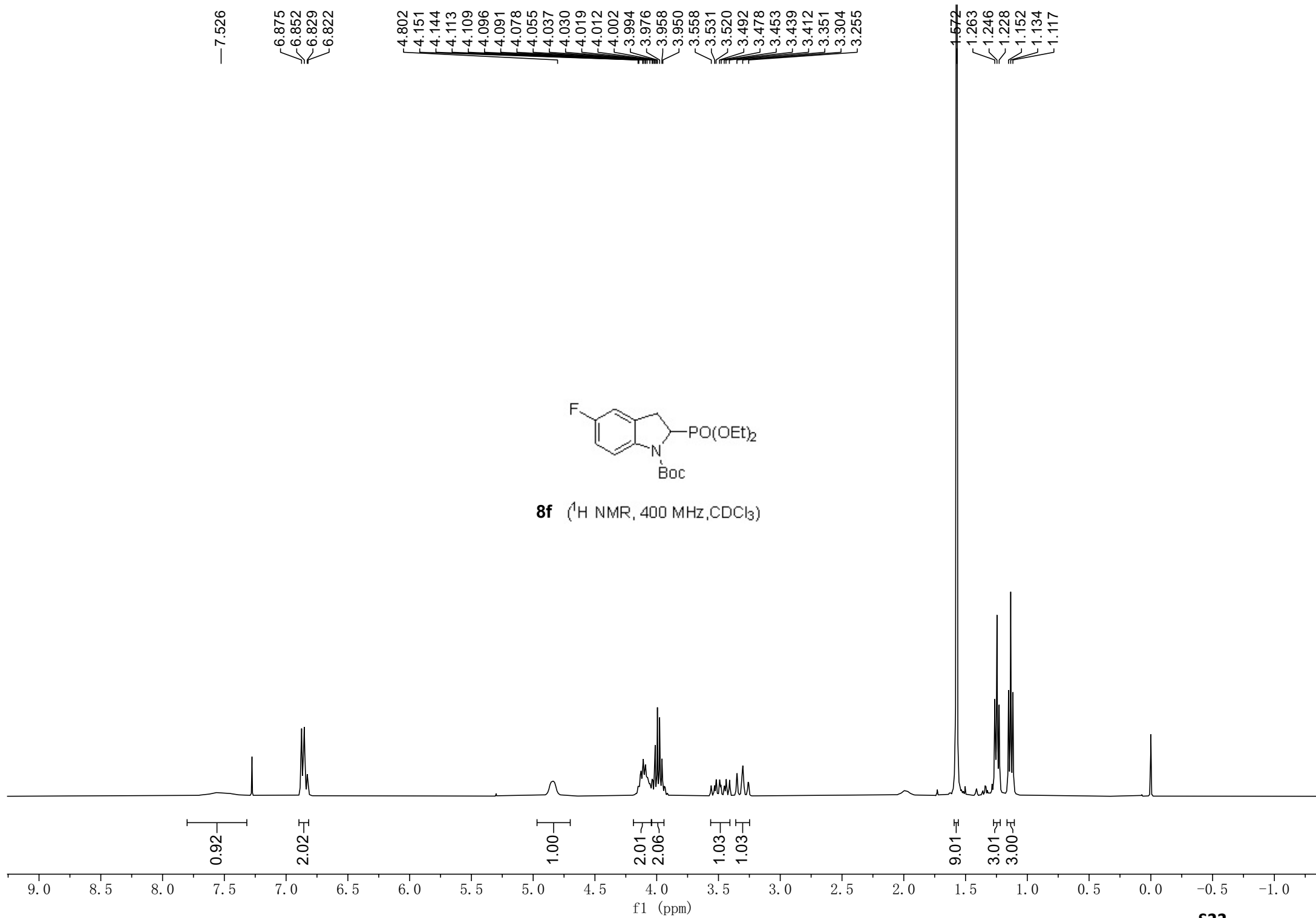


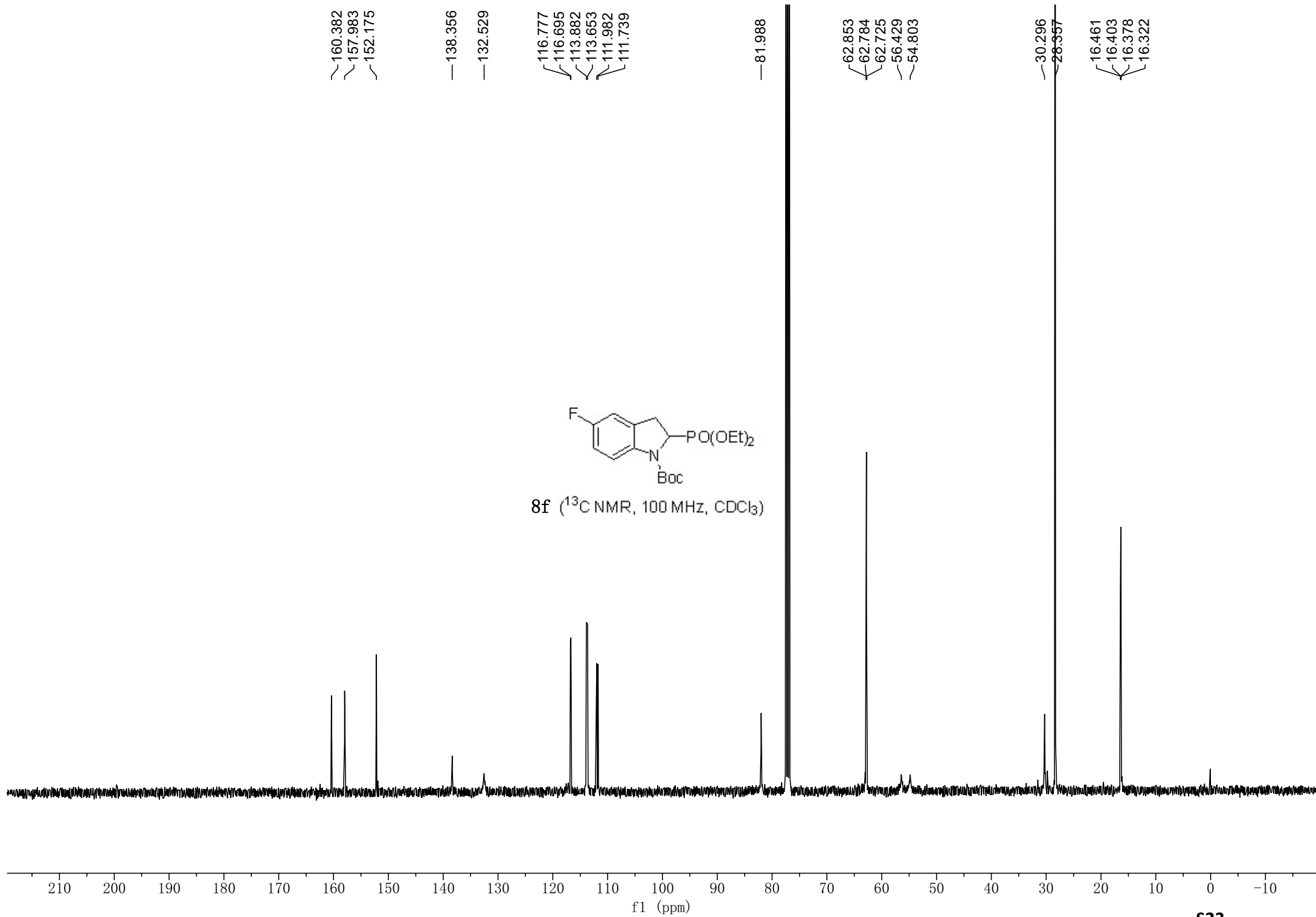
—23.031



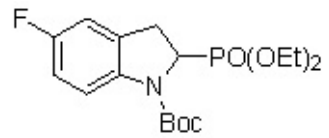
8e (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



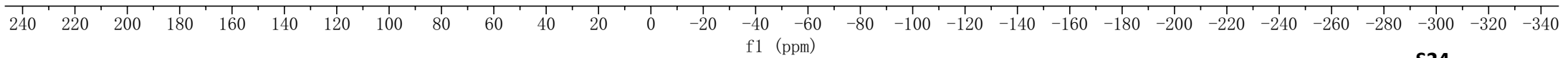




—22.710

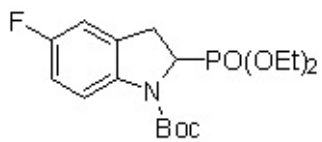


**8f** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

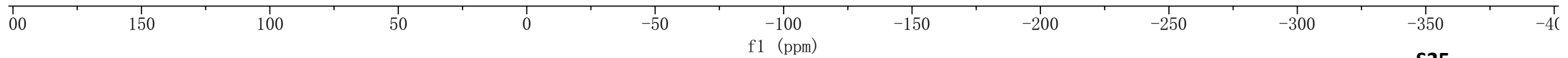


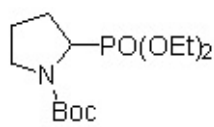


---120.761

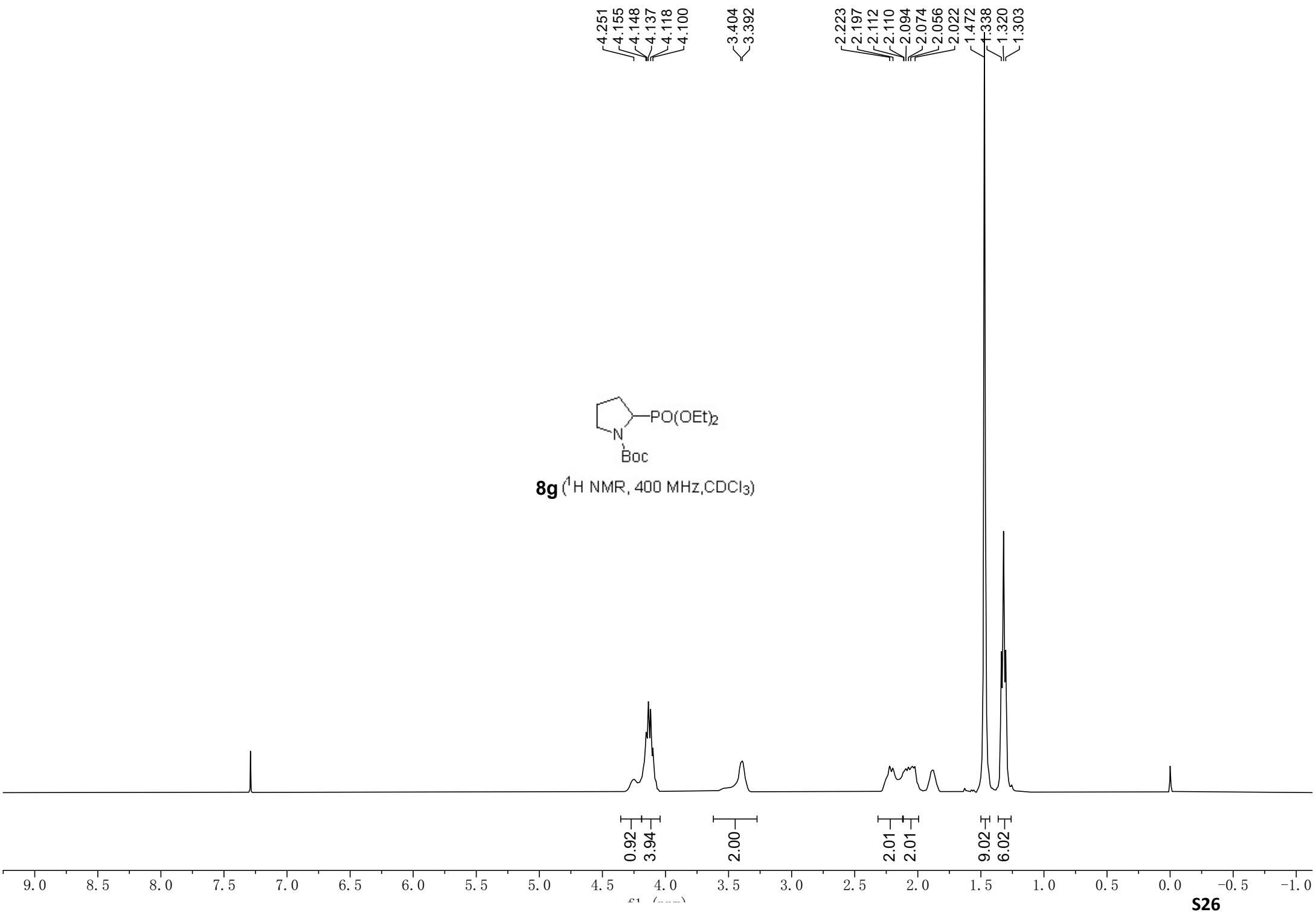


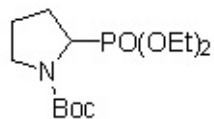
**8f** ( $^{19}\text{F}$  NMR, 376MHz,  $\text{CDCl}_3$ )



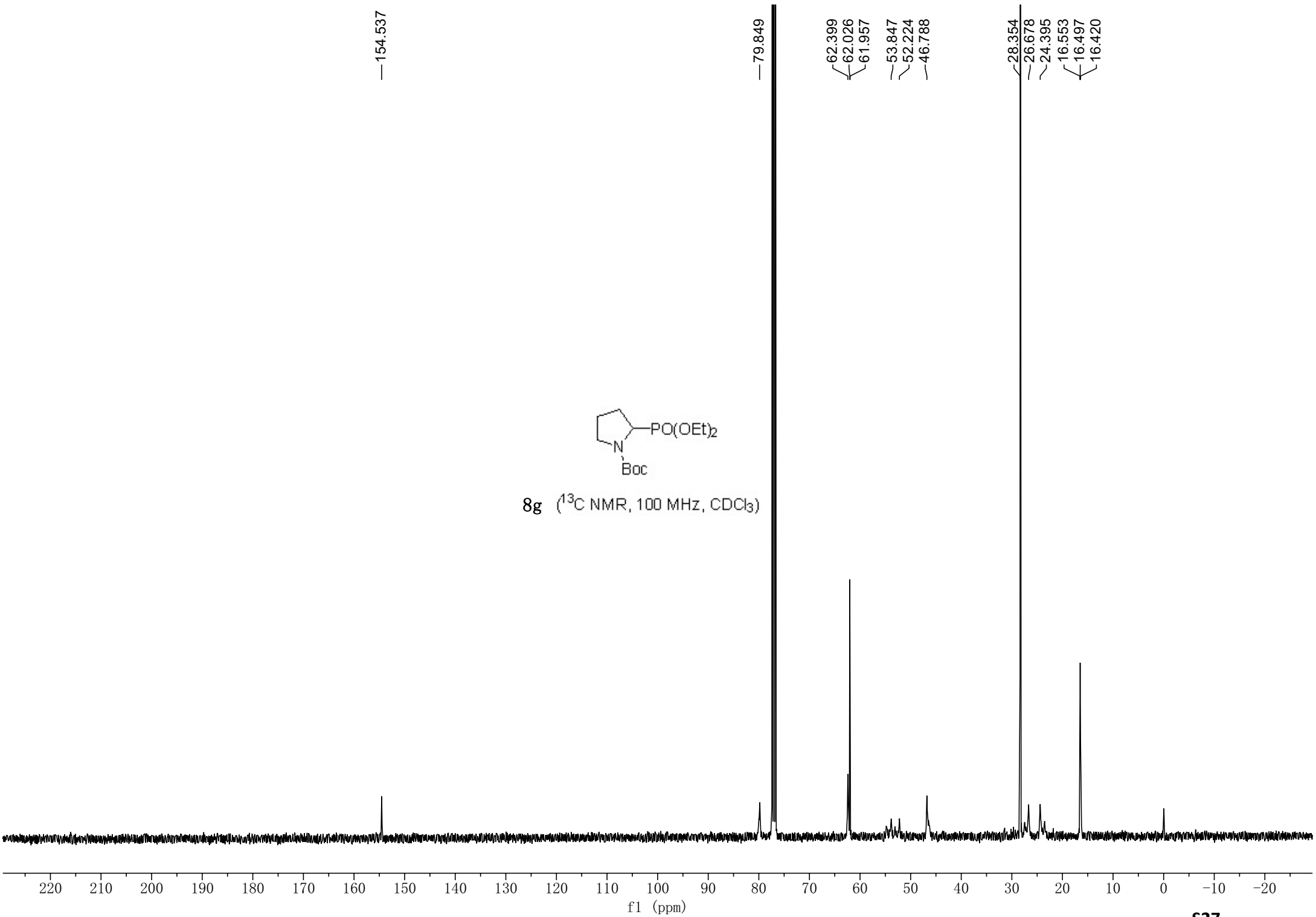


**8g** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

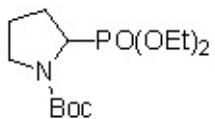




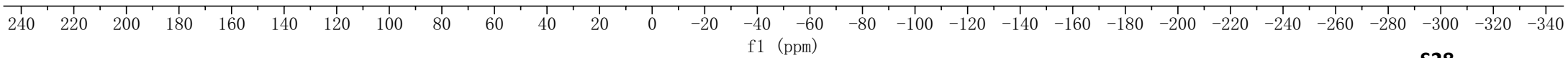
8g (<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>)

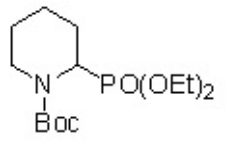


—25.427

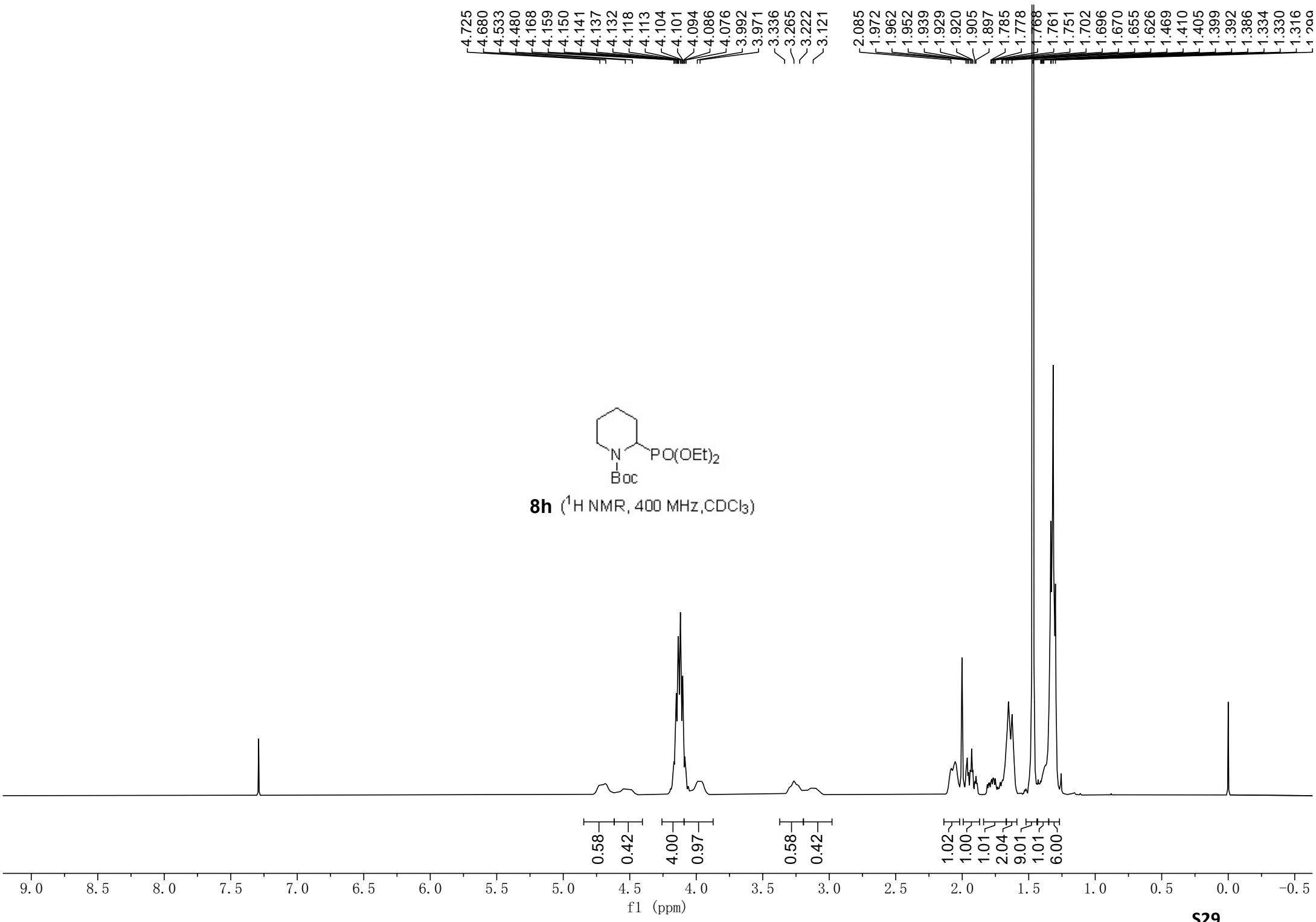


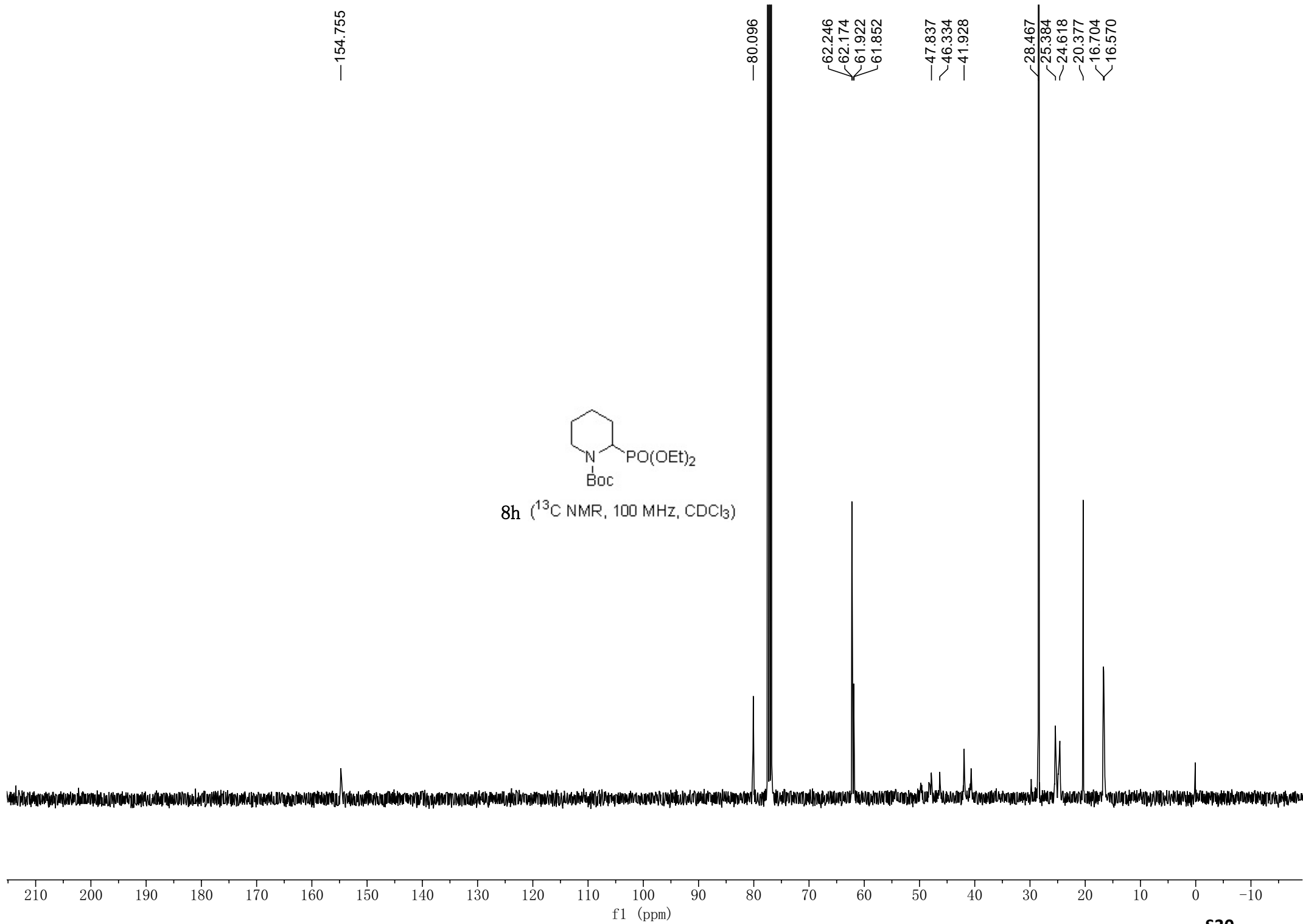
8g (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



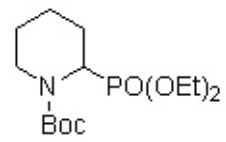


**8h** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

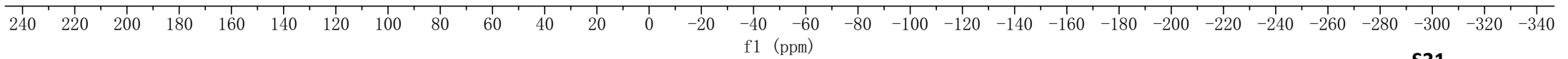


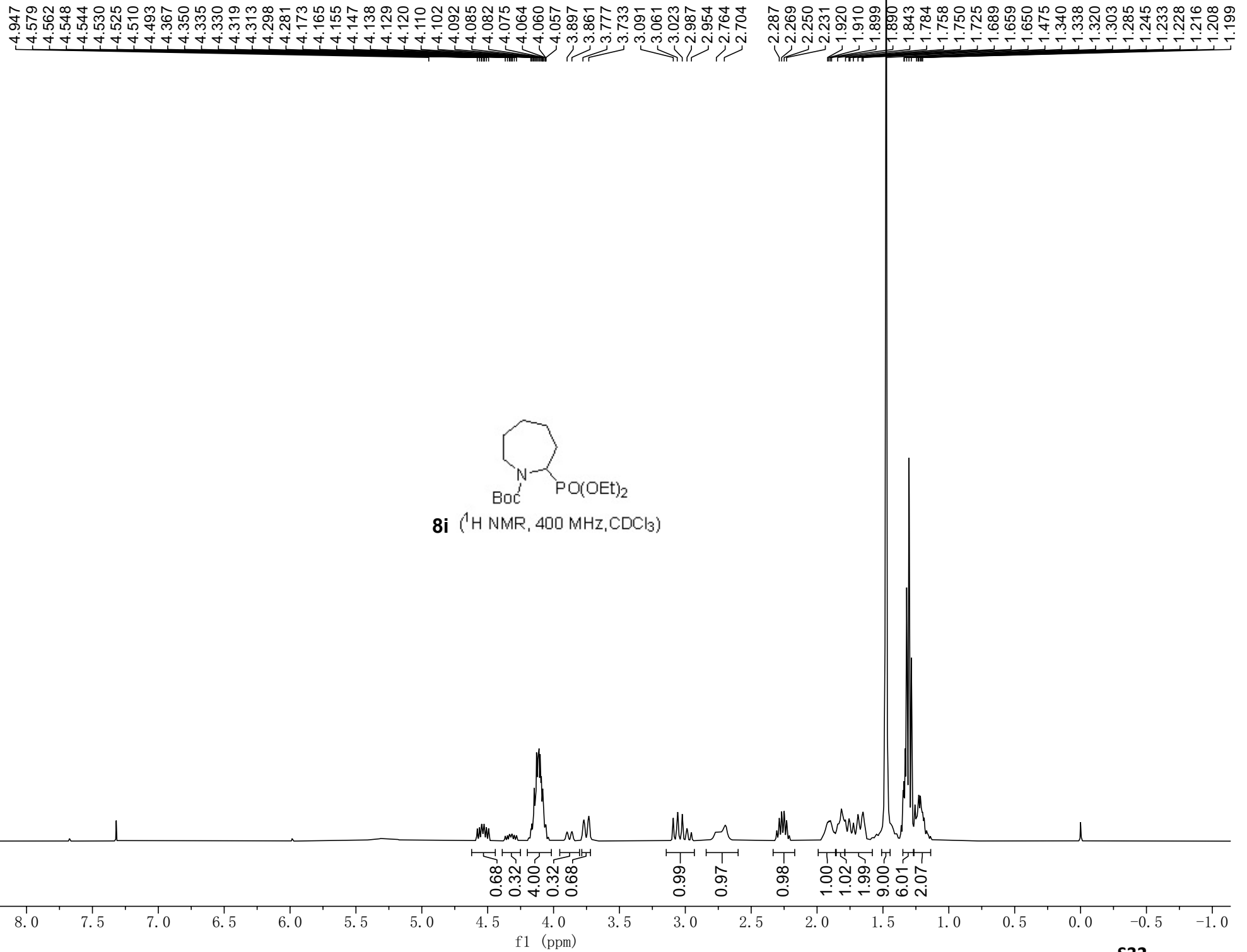


— 25.552



8h (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)

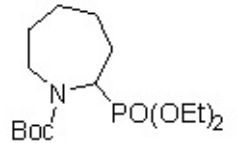




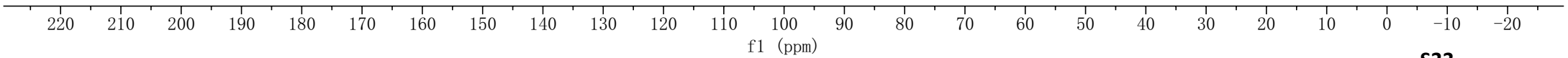


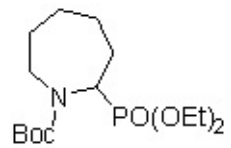
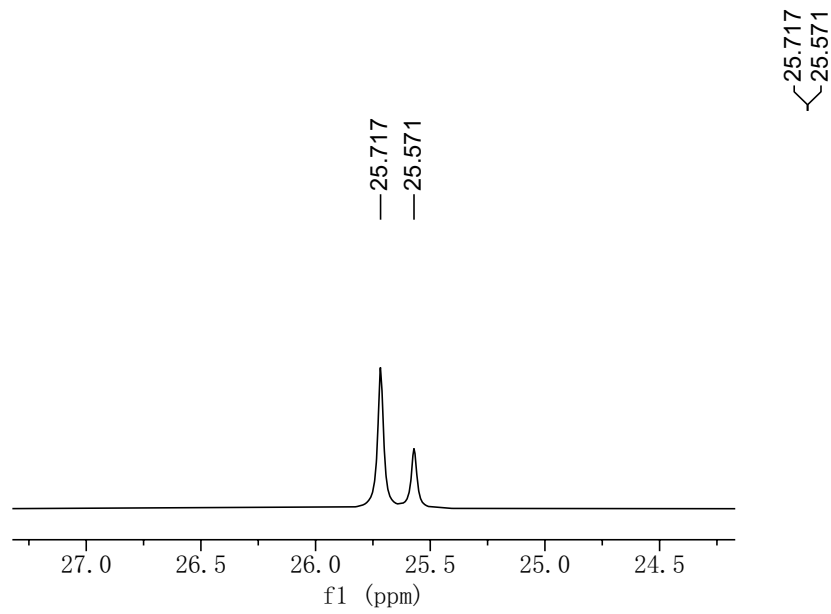
155.815  
155.782  
155.002  
154.980

80.188  
79.830  
62.529  
62.482  
62.458  
62.408  
62.032  
61.983  
61.964  
61.911  
52.968  
51.417  
51.275  
49.720  
43.796  
43.307  
29.733  
29.614  
29.152  
29.120  
28.536  
28.507  
28.460  
28.425  
28.363  
28.334  
28.112  
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16.429  
16.368

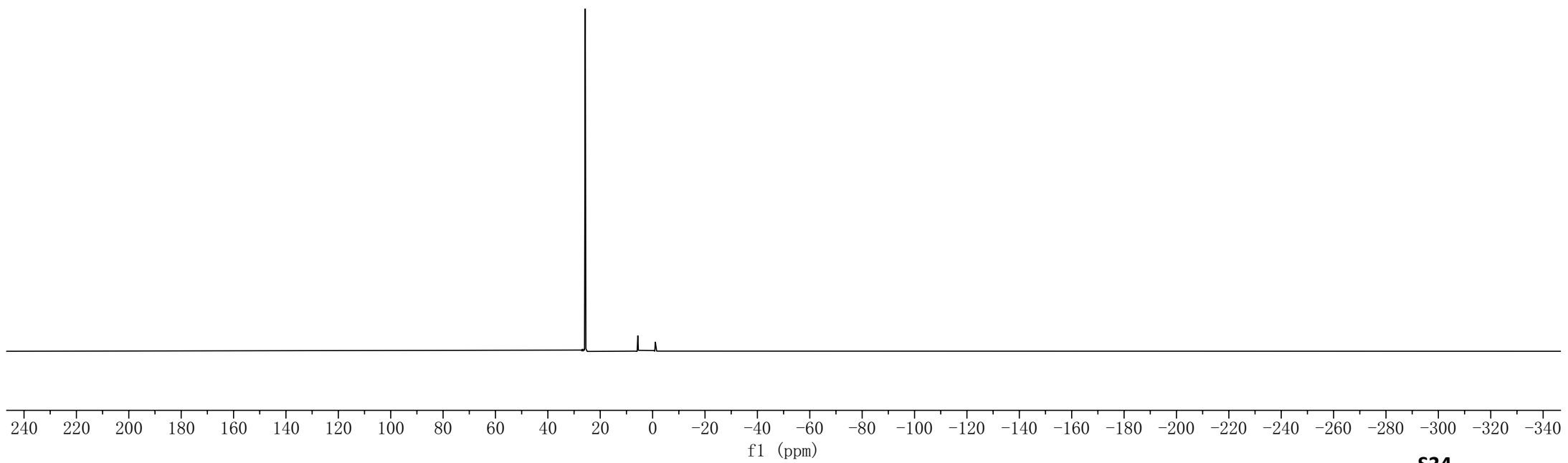


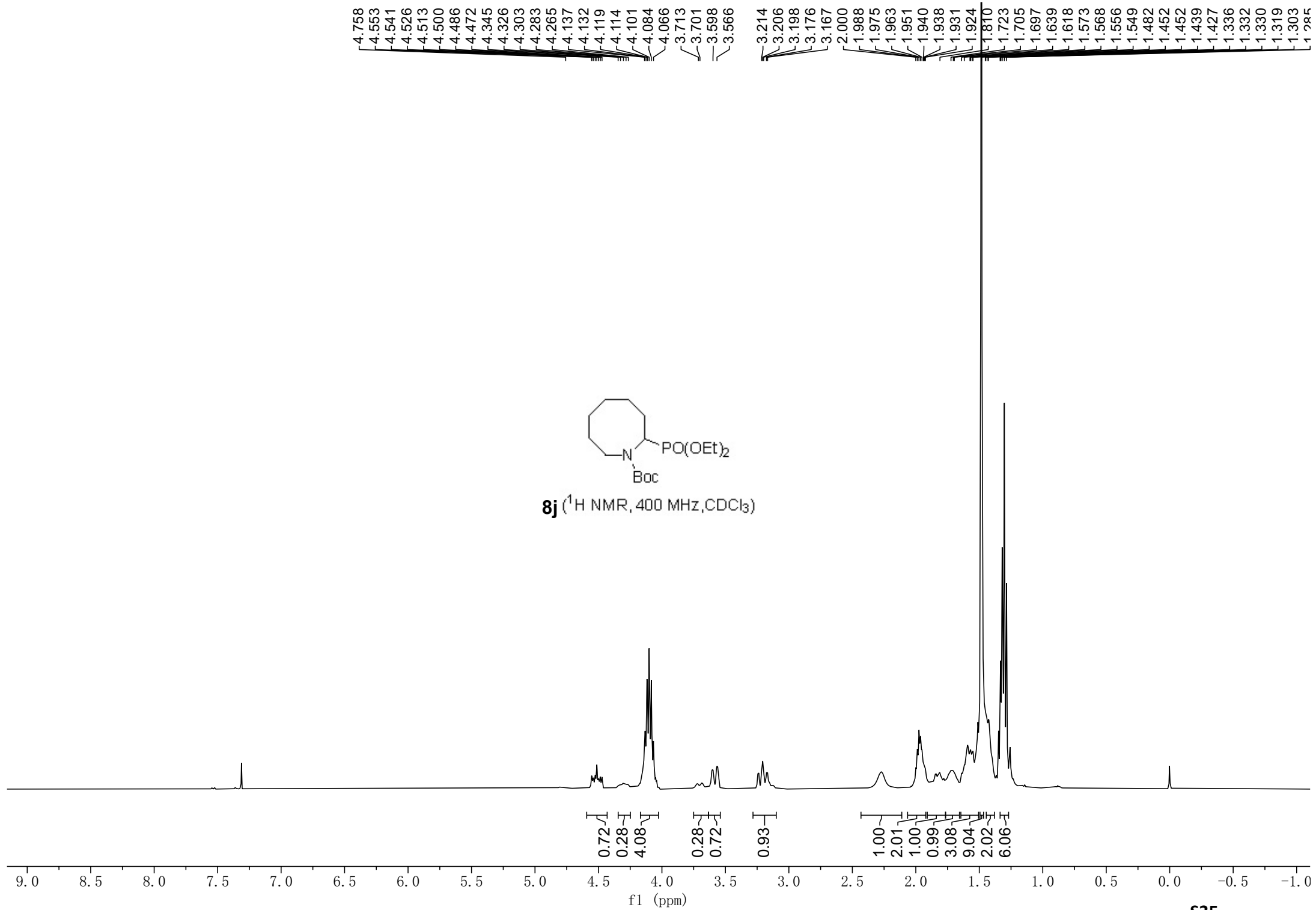
8i (<sup>13</sup>C NMR, 100 MHz, CDCl<sub>3</sub>)

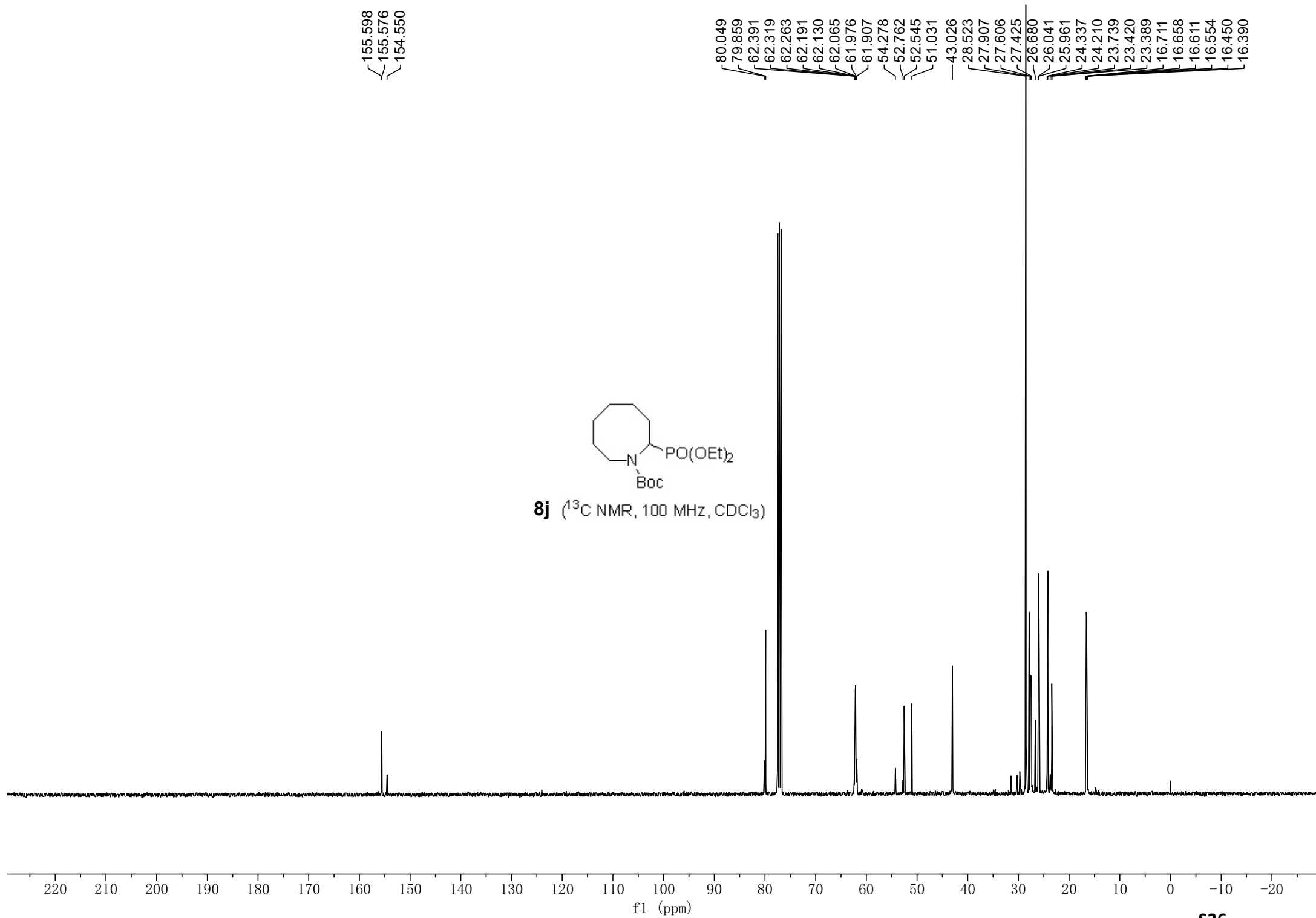


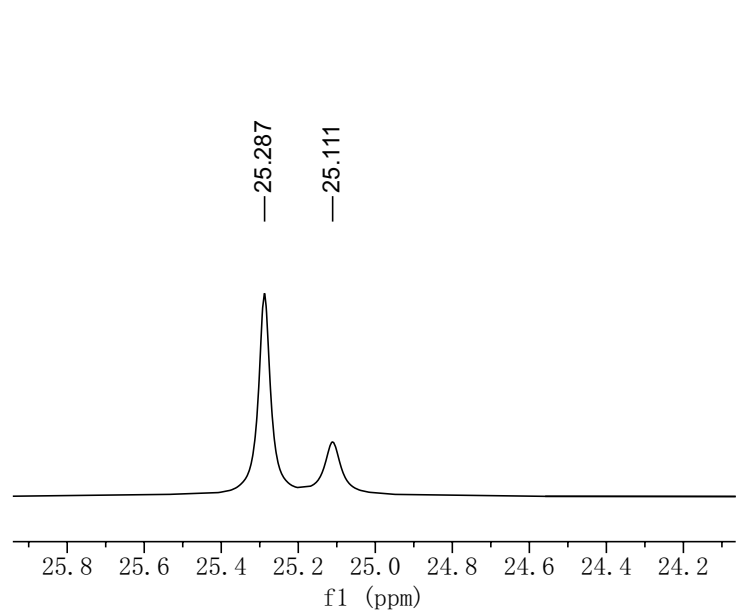


**8i** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)

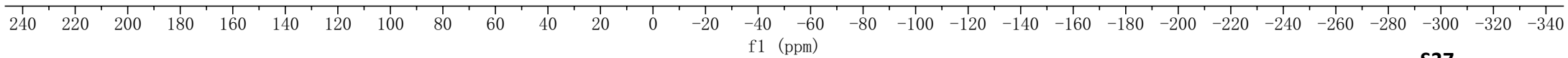
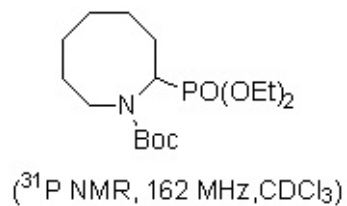


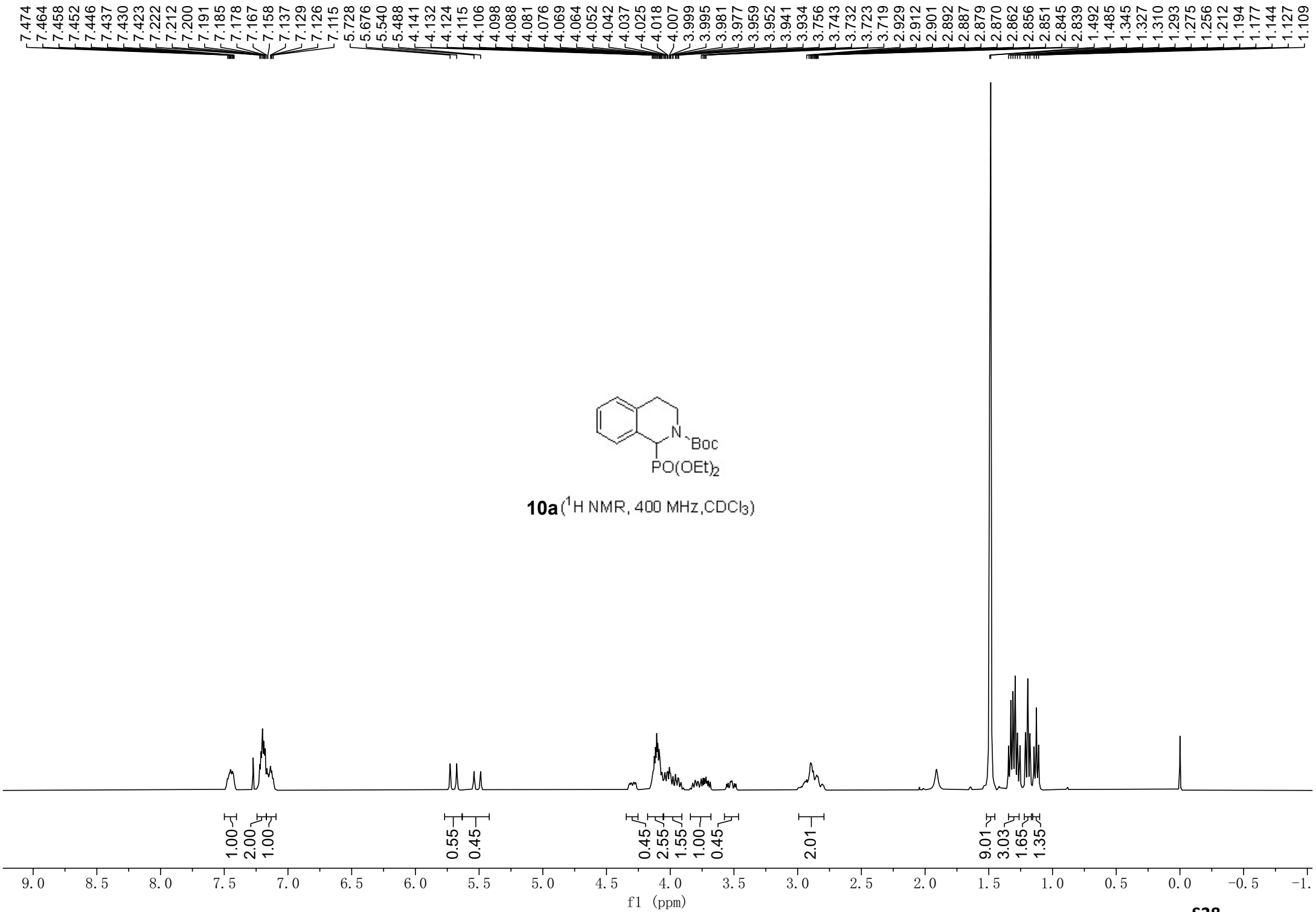


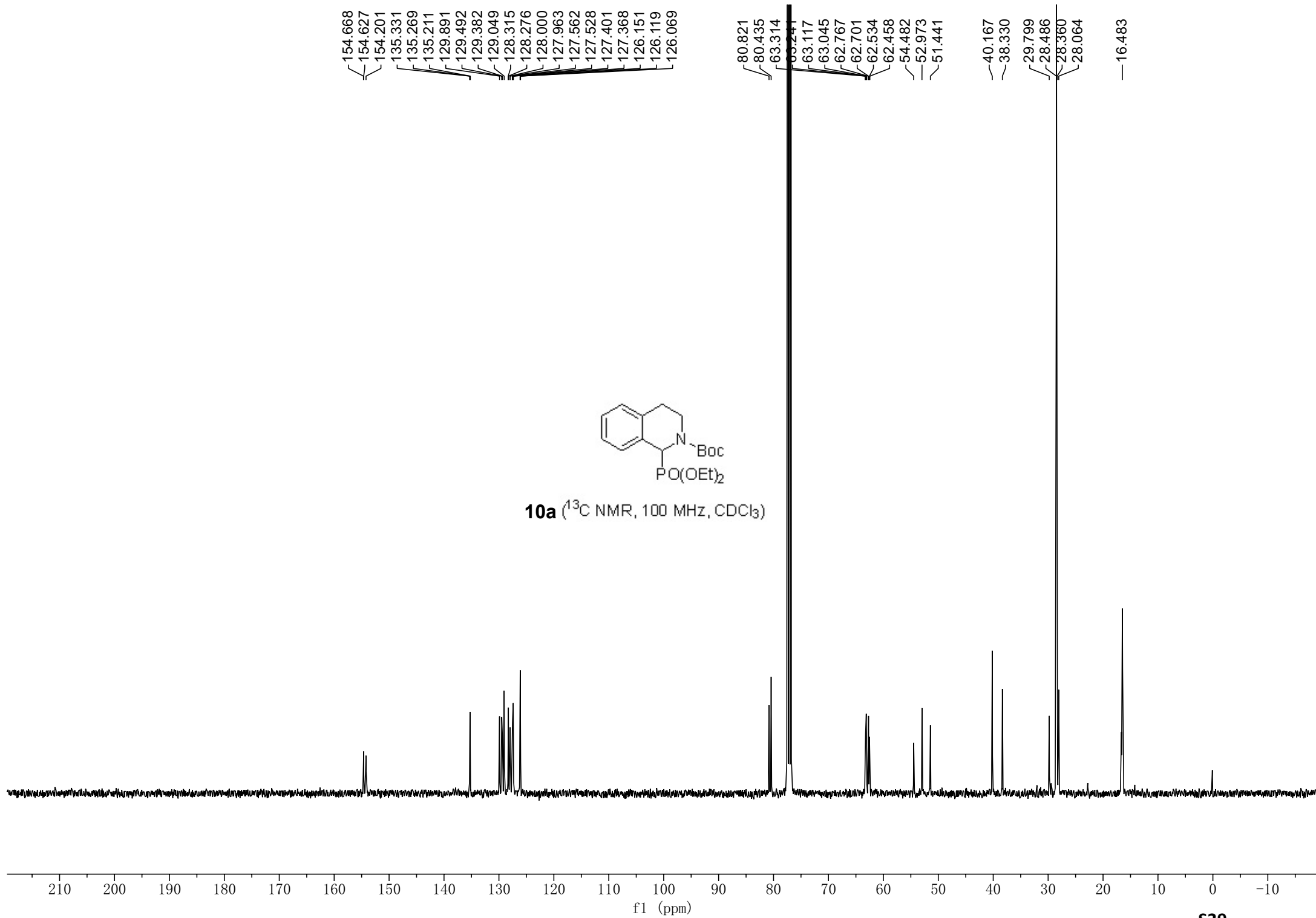


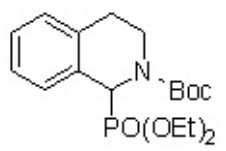
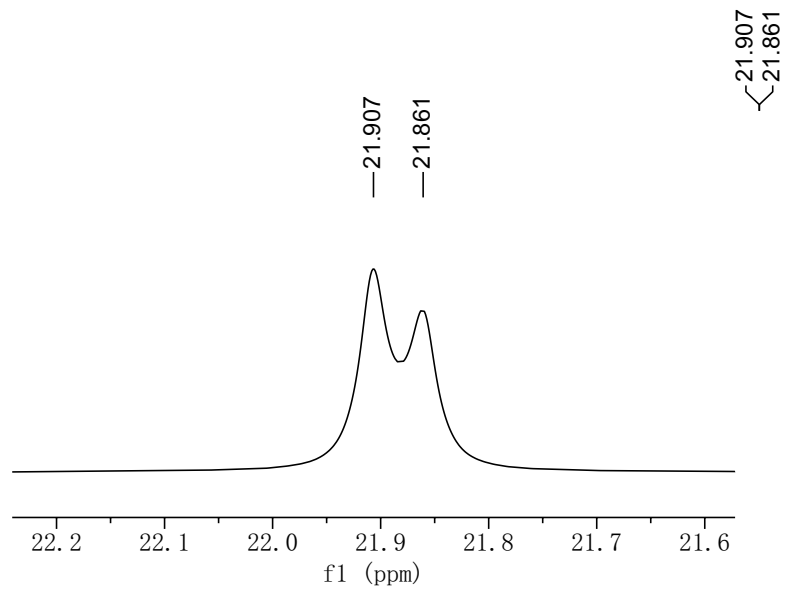


25.287  
25.111

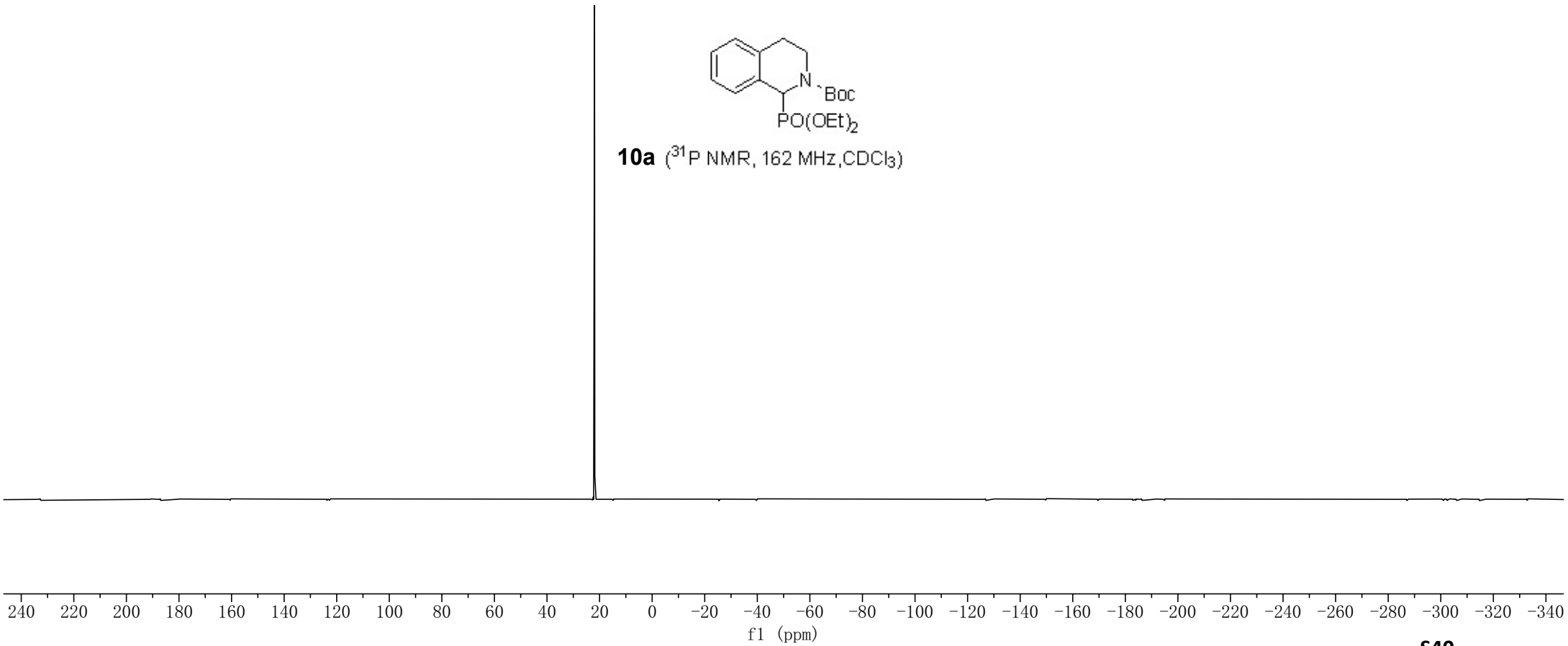




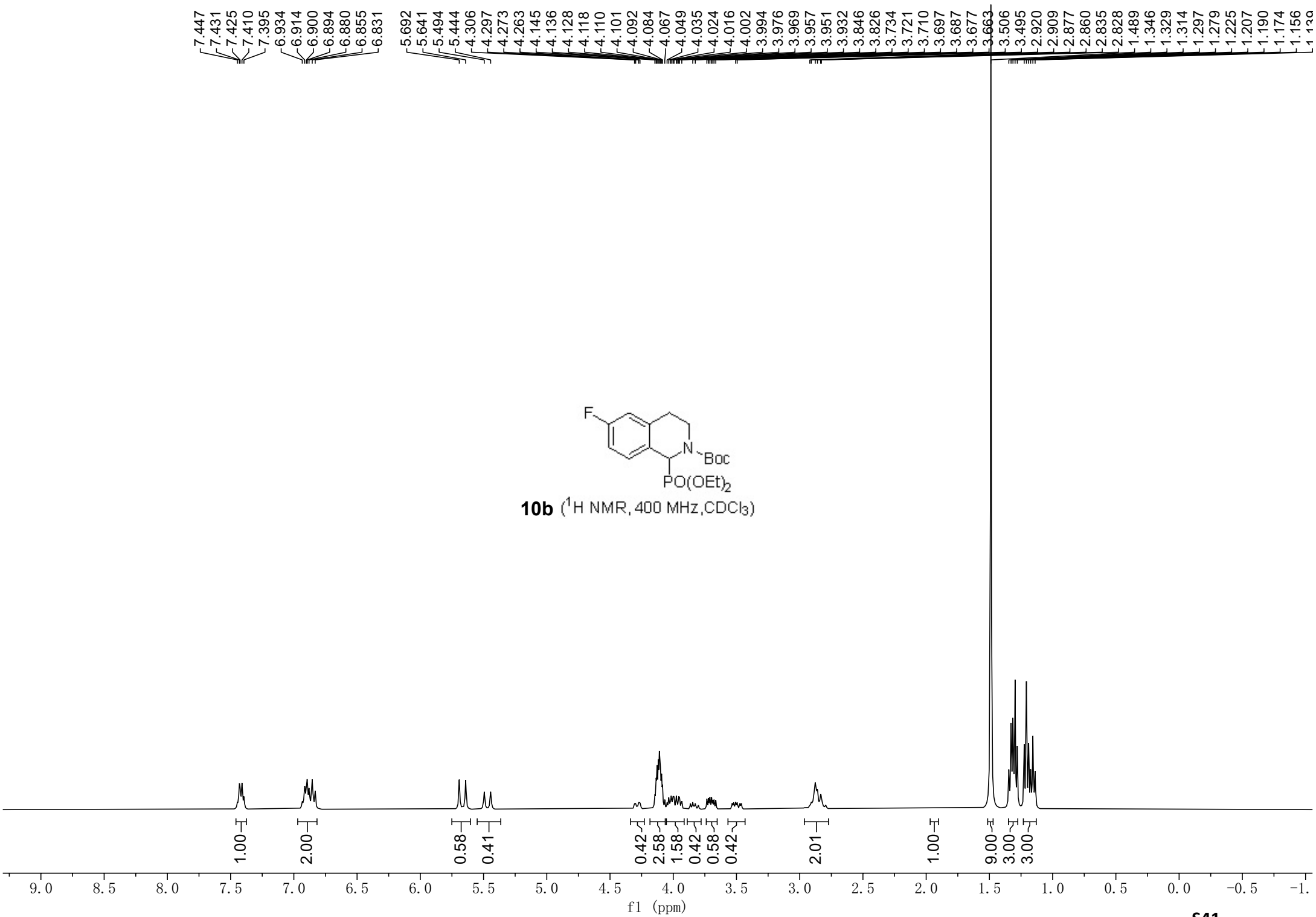


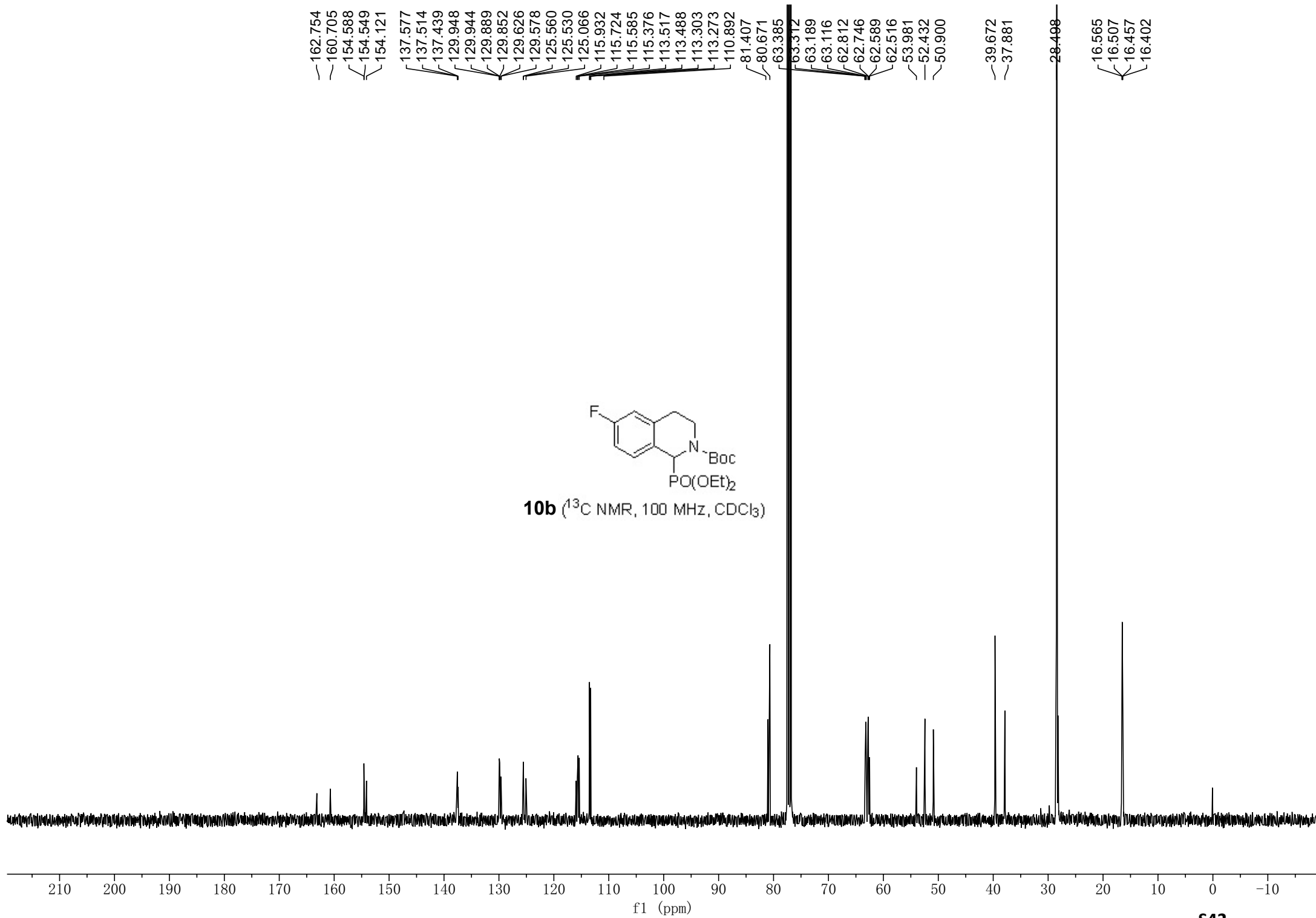


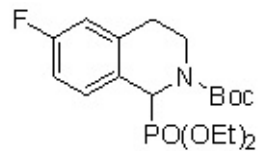
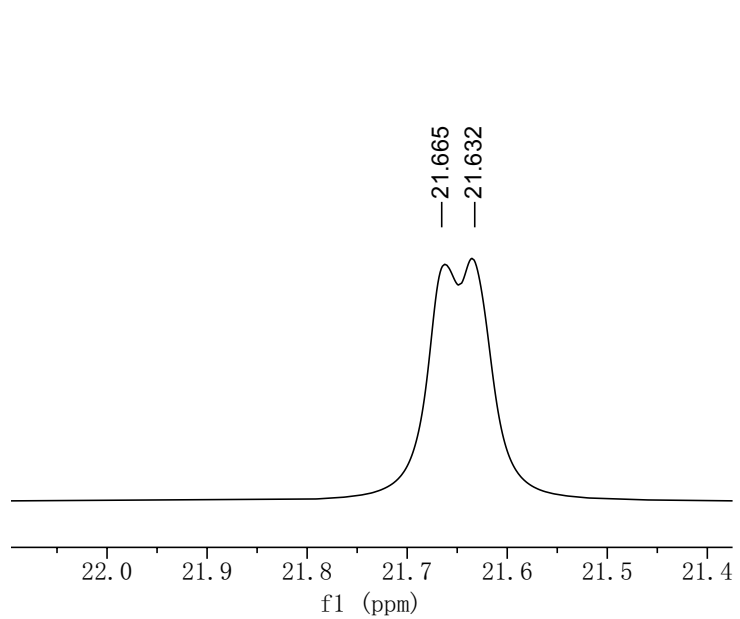
**10a** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



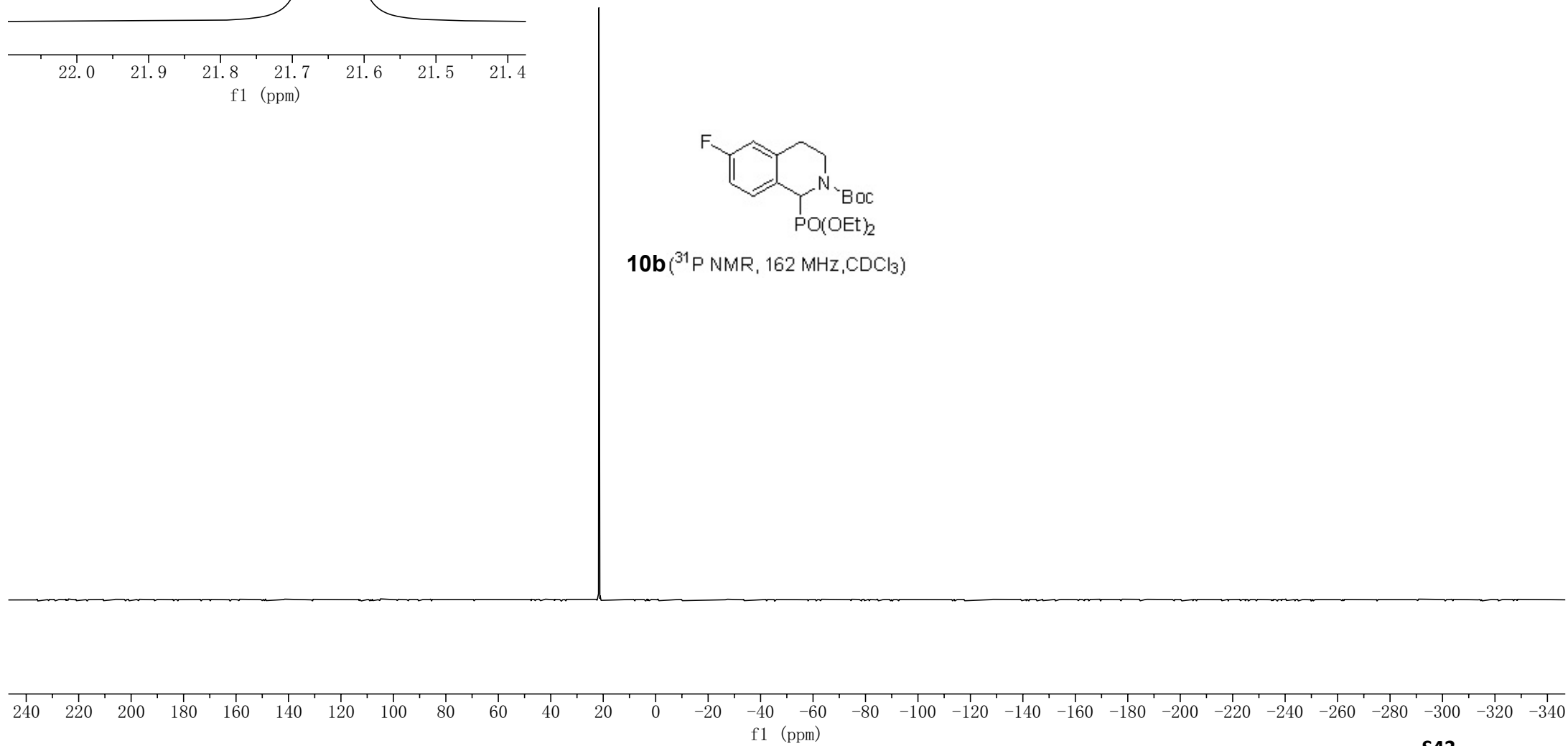


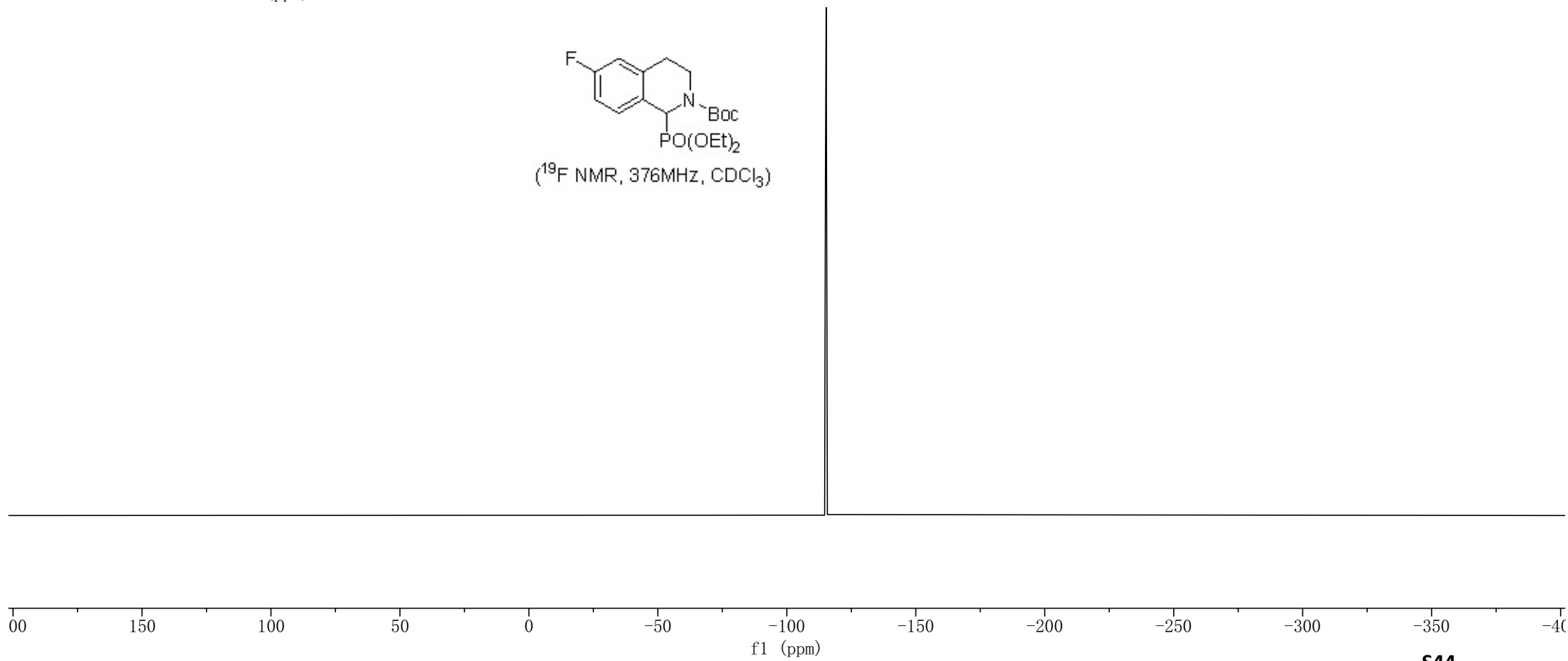
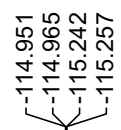
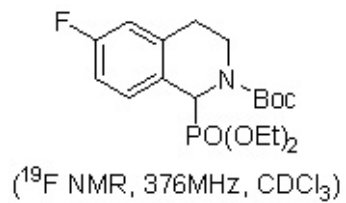
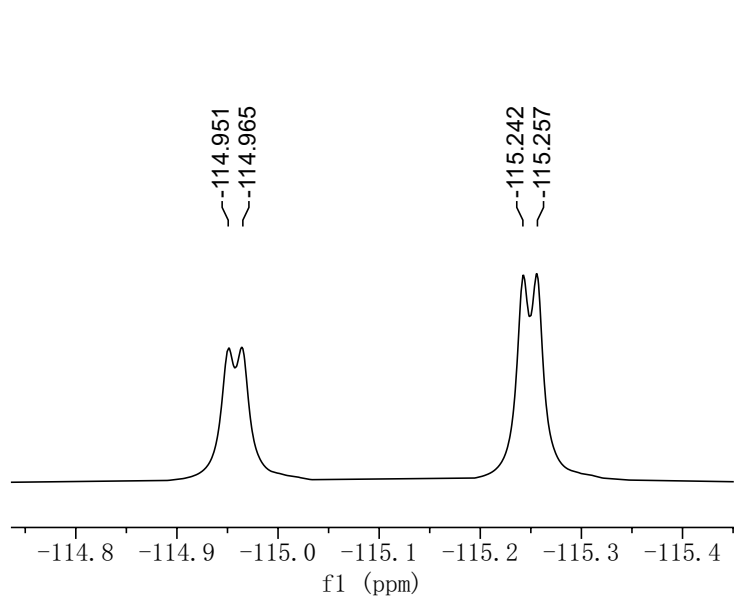


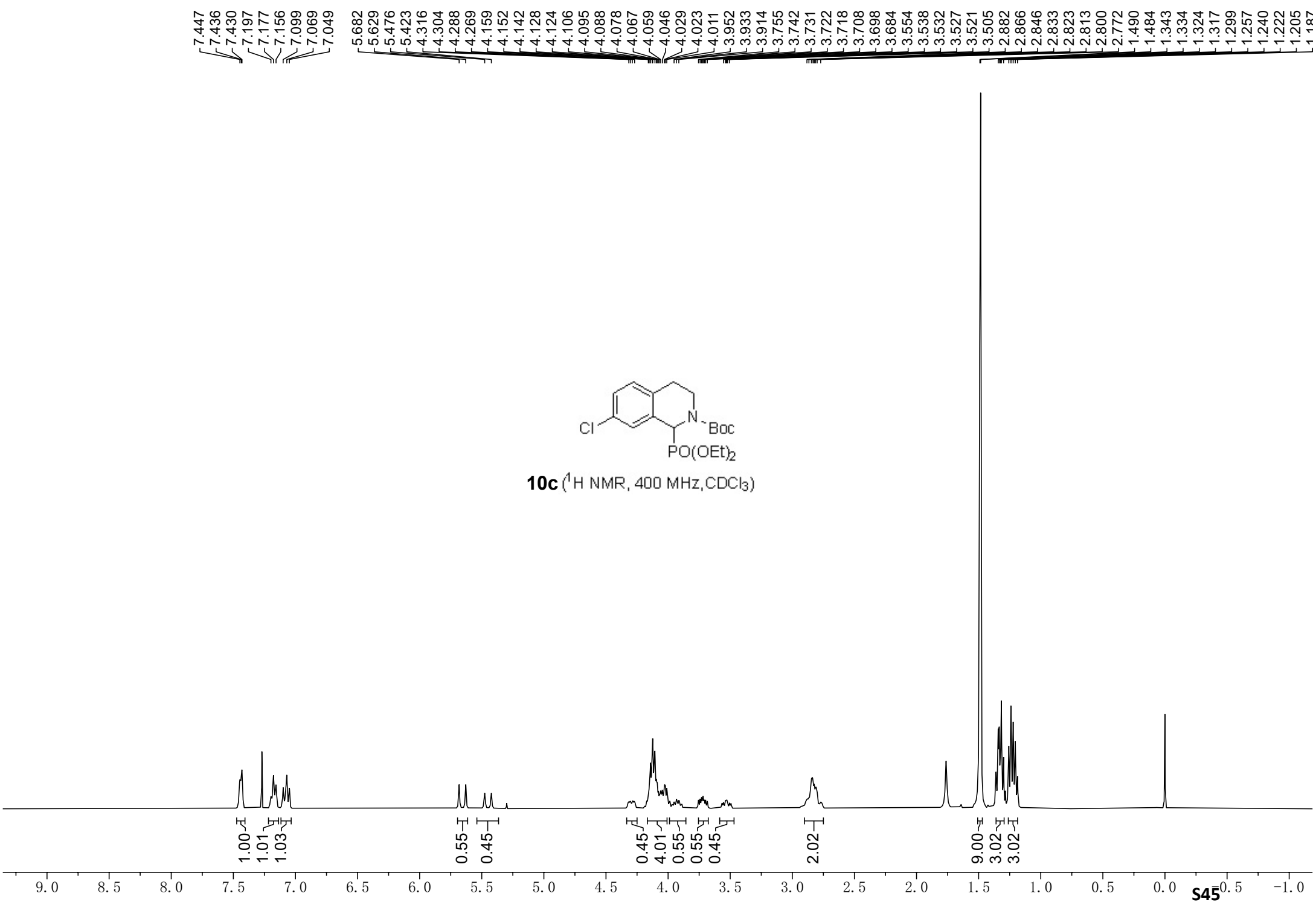


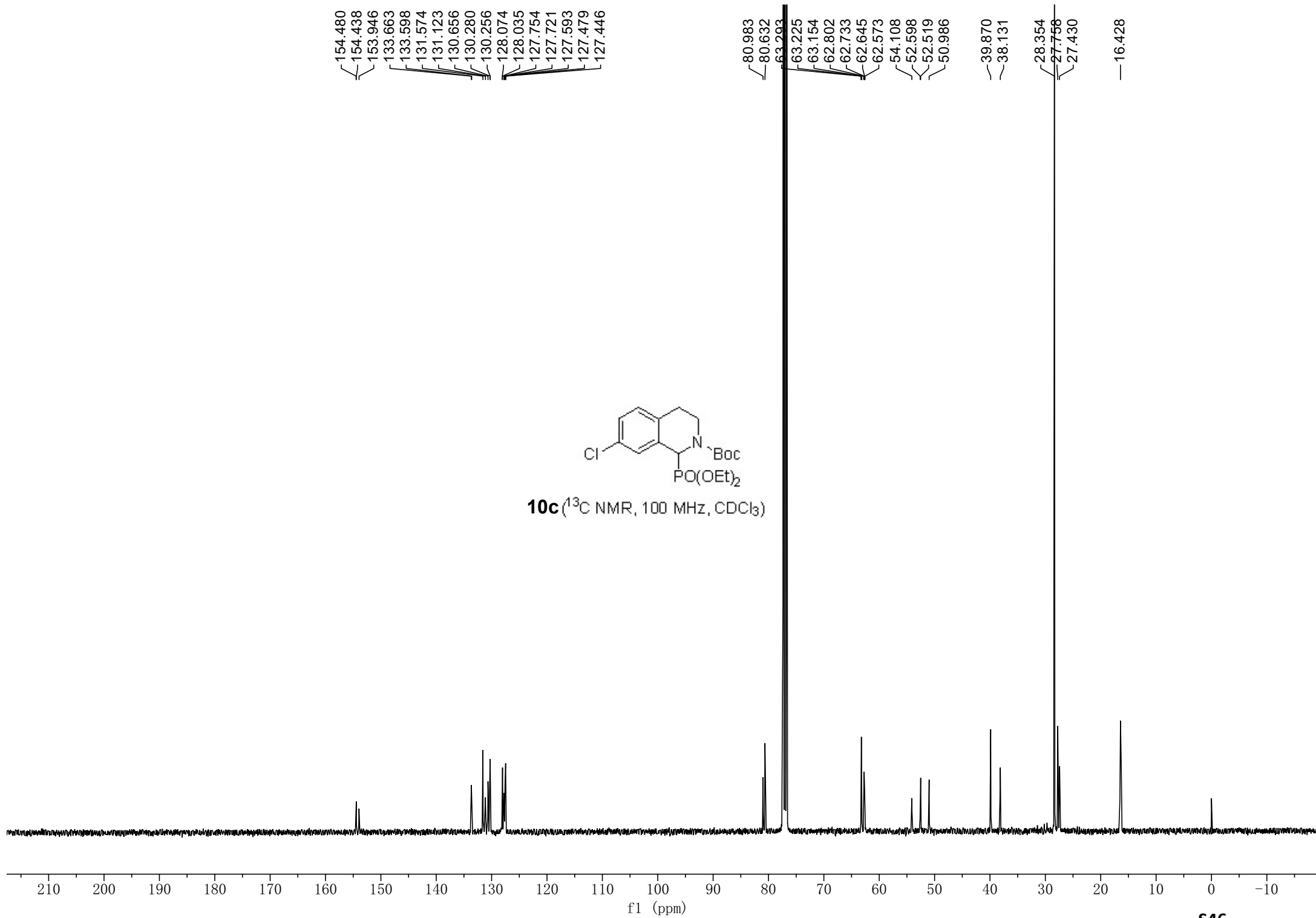


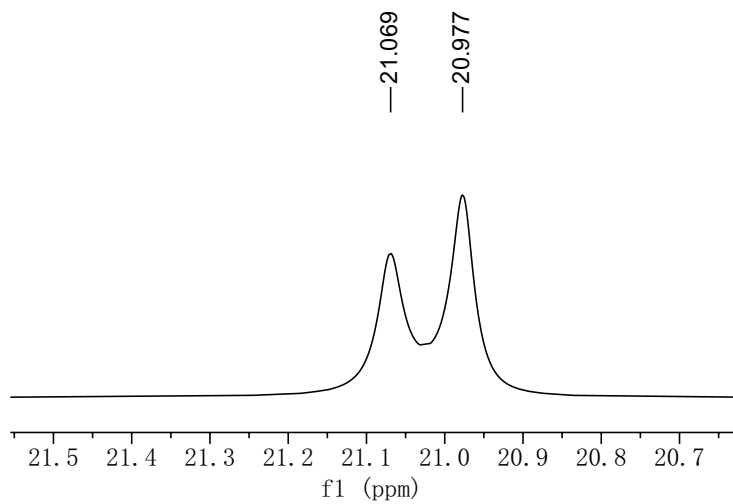
**10b** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )



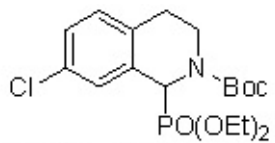




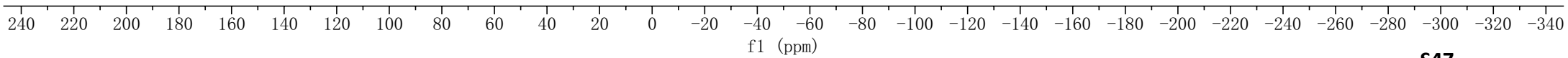




21.069  
20.977



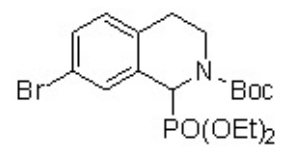
**10c** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



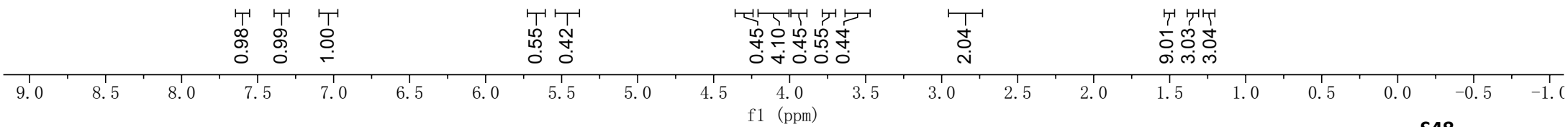
7.611  
7.595  
7.359  
7.342  
7.321  
7.054  
7.034  
7.026  
7.006

5.693  
5.640  
5.487  
5.433

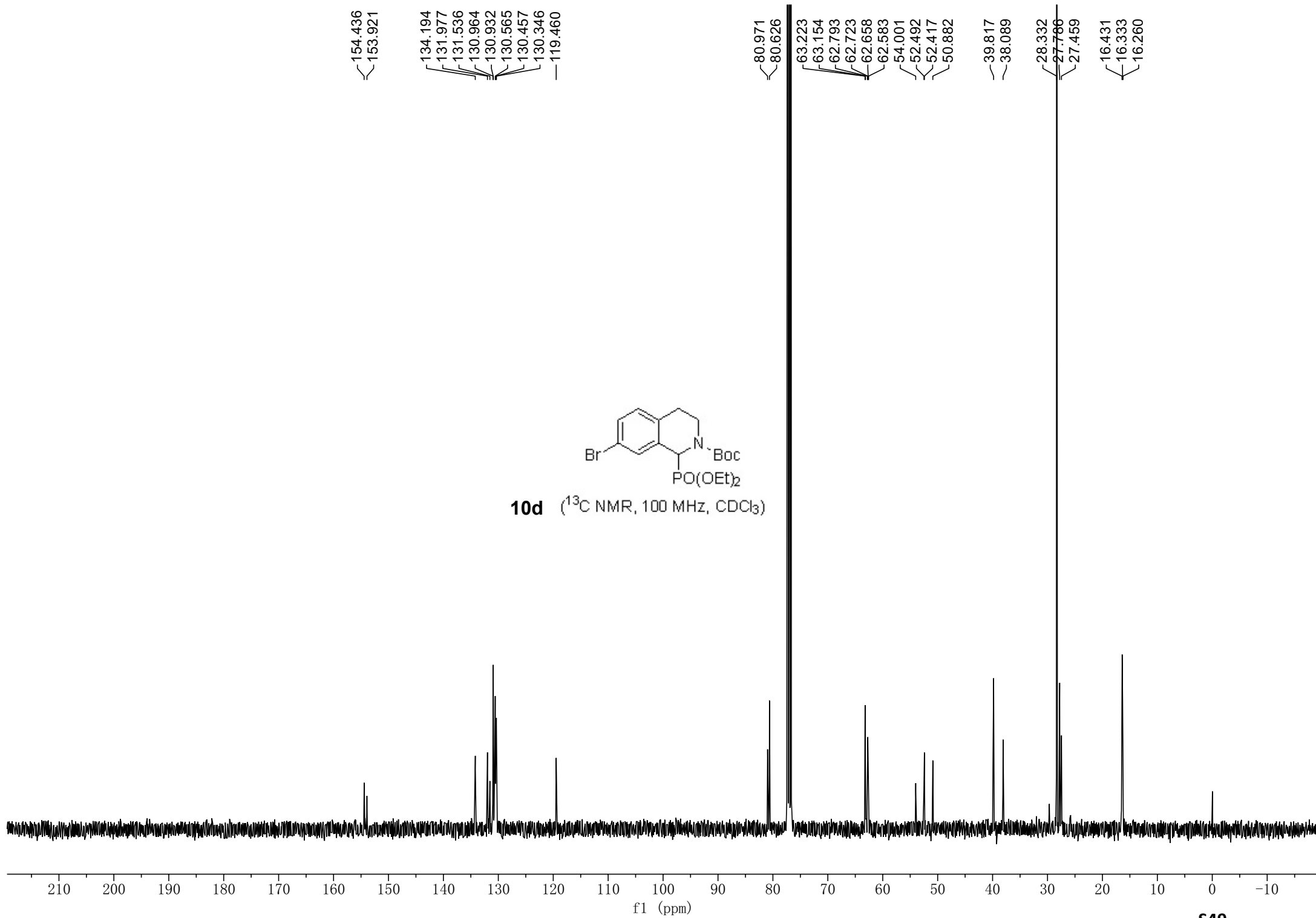
4.322  
4.307  
4.288  
4.274  
4.186  
4.161  
4.150  
4.132  
4.124  
4.078  
4.067  
4.062  
4.043  
4.031  
4.025  
4.025  
3.960  
3.954  
3.934  
3.776  
3.763  
3.751  
3.742  
3.729  
3.718  
3.705  
3.576  
3.561  
3.548  
3.527  
2.875  
2.851  
2.844  
2.826  
2.816  
2.808  
2.802  
2.794  
2.768  
1.497  
1.375  
1.357  
1.350  
1.339  
1.333  
1.315  
1.275  
1.257  
1.241  
1.227  
1.200

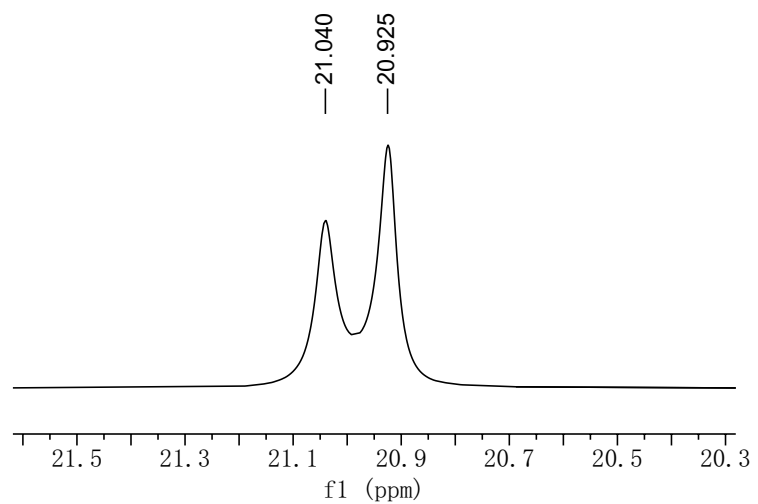


**10d** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

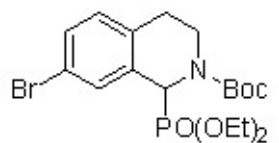




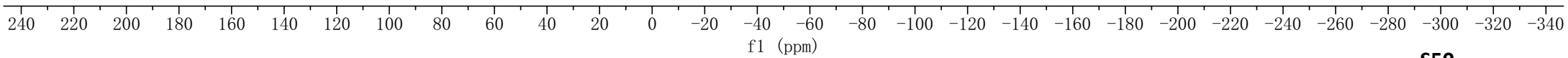


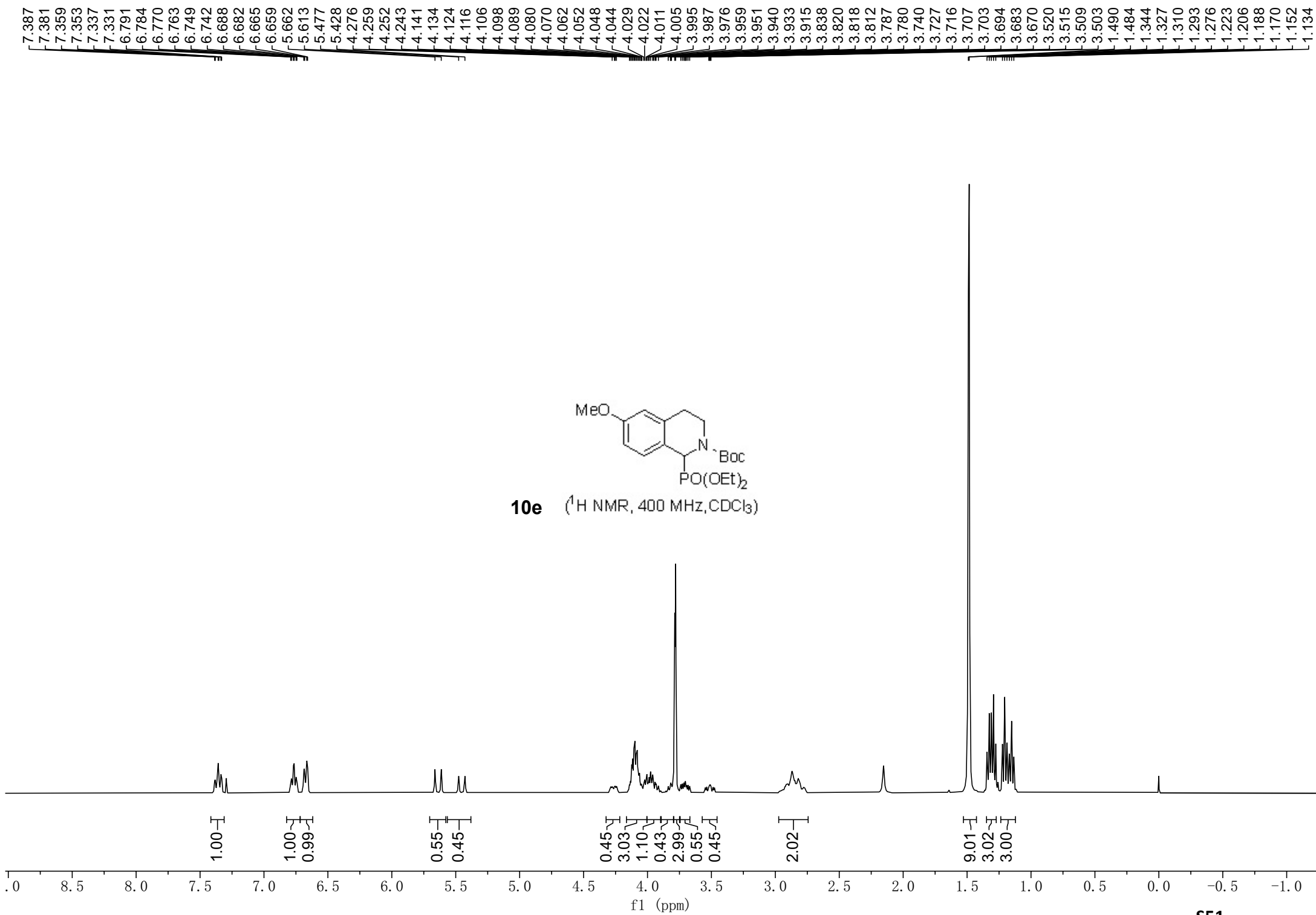


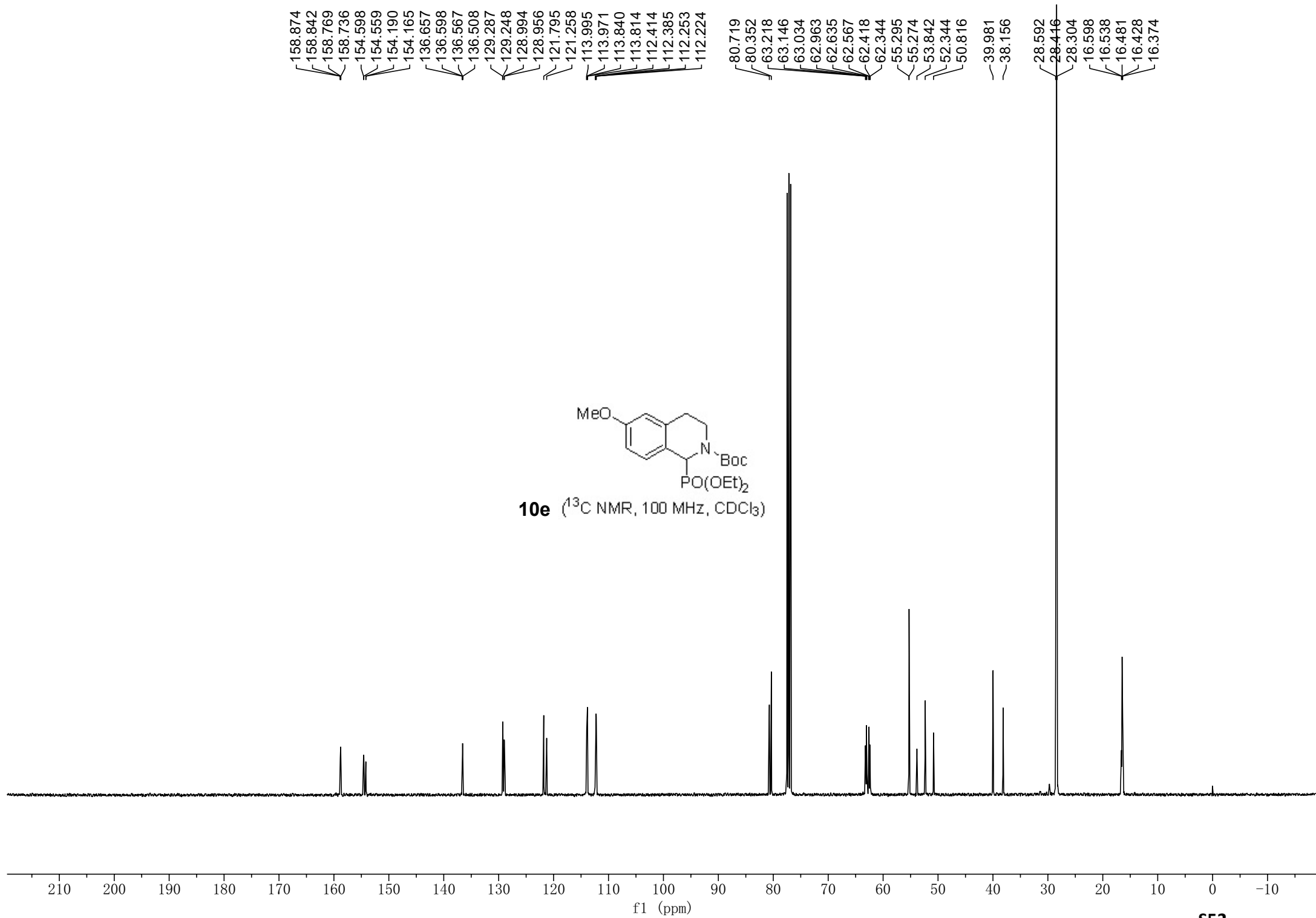
21.040  
20.925

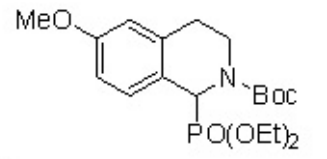
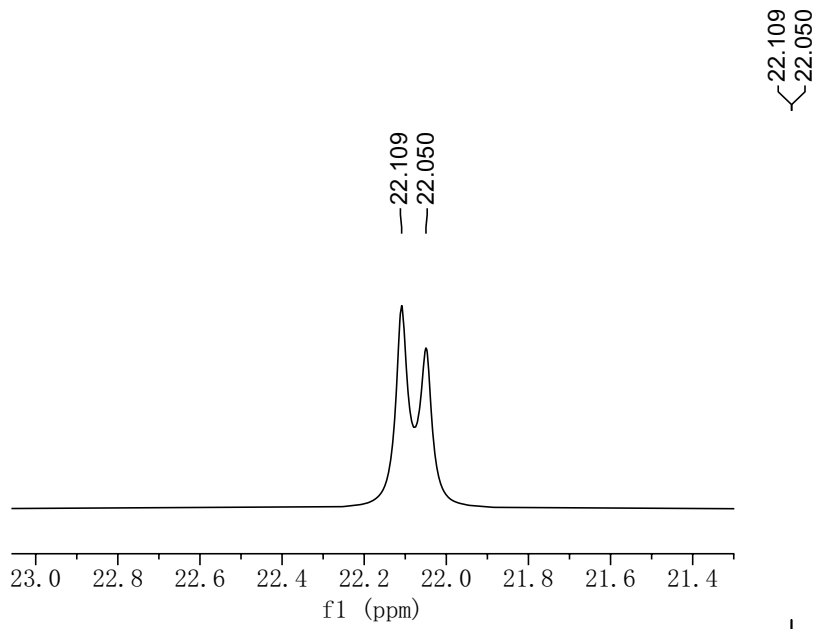


**10d** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)

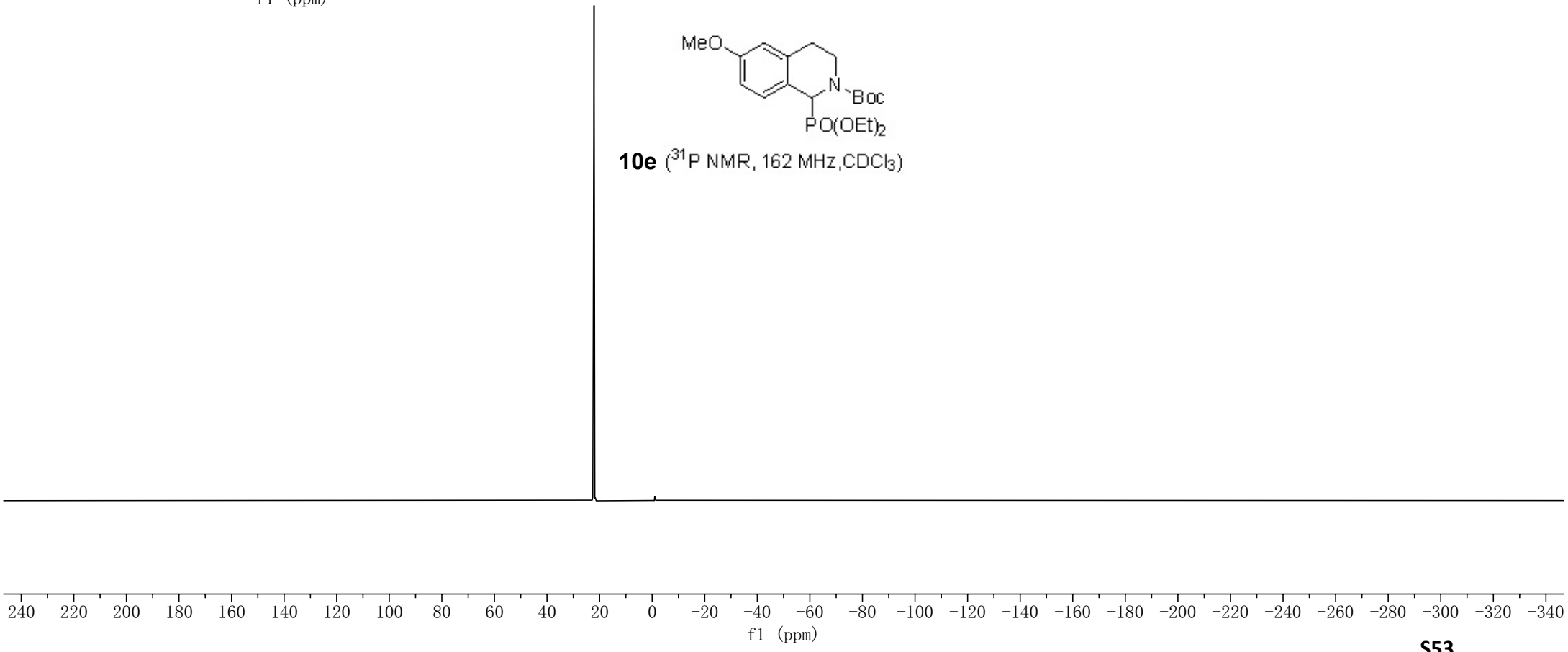


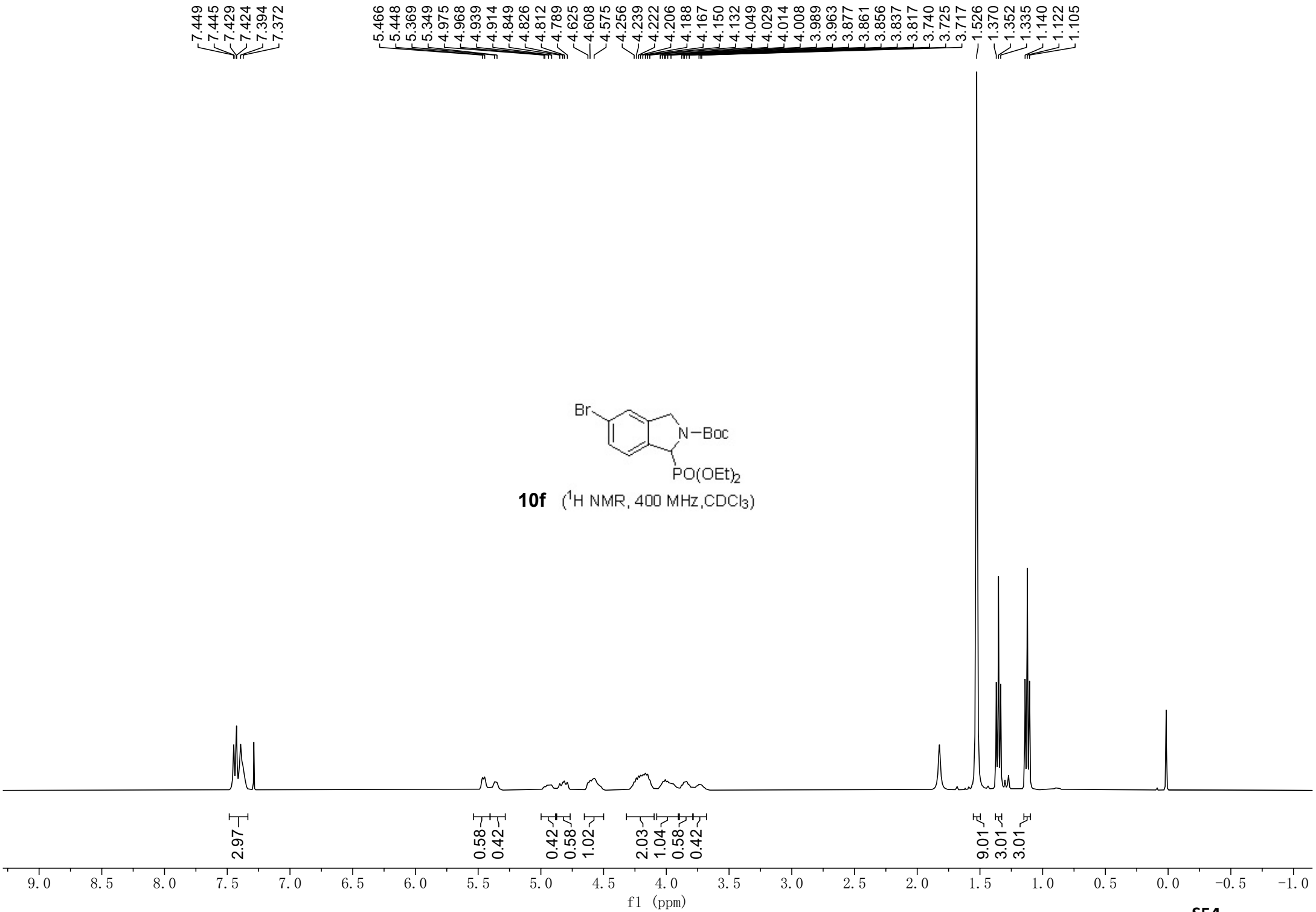


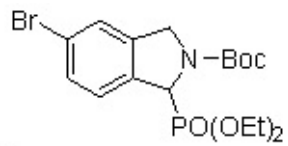




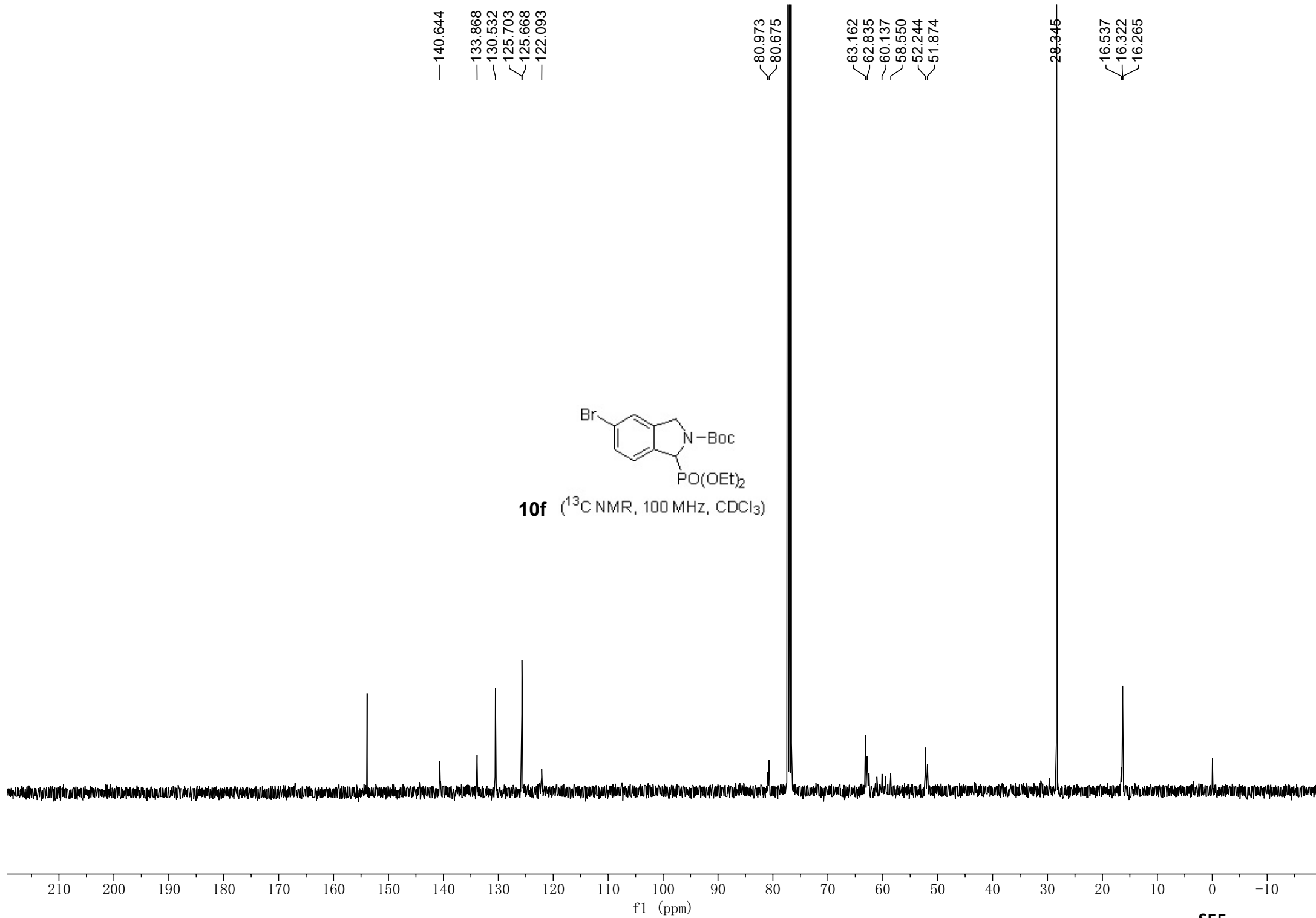
**10e** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



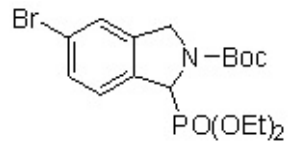




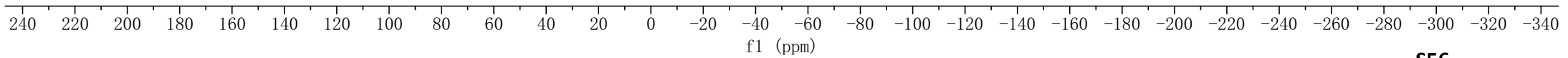
**10f** ( $^{13}\text{C}$  NMR, 100 MHz,  $\text{CDCl}_3$ )



—19.899



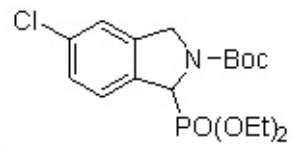
**10f** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )





7.439  
7.419  
7.287  
7.265  
7.228

5.483  
5.457  
5.378  
5.360  
4.962  
4.924  
4.900  
4.839  
4.815  
4.801  
4.778  
4.665  
4.597  
4.560  
4.243  
4.225  
4.213  
4.207  
4.197  
4.180  
4.175  
4.161  
4.157  
4.140  
4.122  
4.039  
4.020  
3.998  
3.978  
3.971  
3.960  
3.953  
3.942  
3.934  
3.930  
3.916  
3.867  
3.847  
3.831  
3.814  
3.792  
3.779  
3.757  
3.726  
3.705  
3.703  
3.688  
1.518  
1.361  
1.343  
1.326  
1.129  
1.111  
1.093



**10g** ( $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$ )

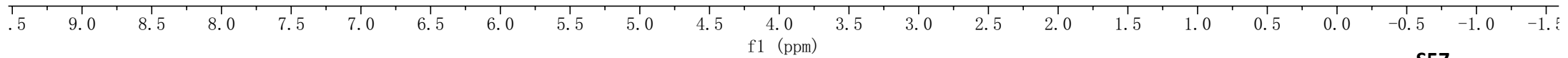
1.01  
2.00

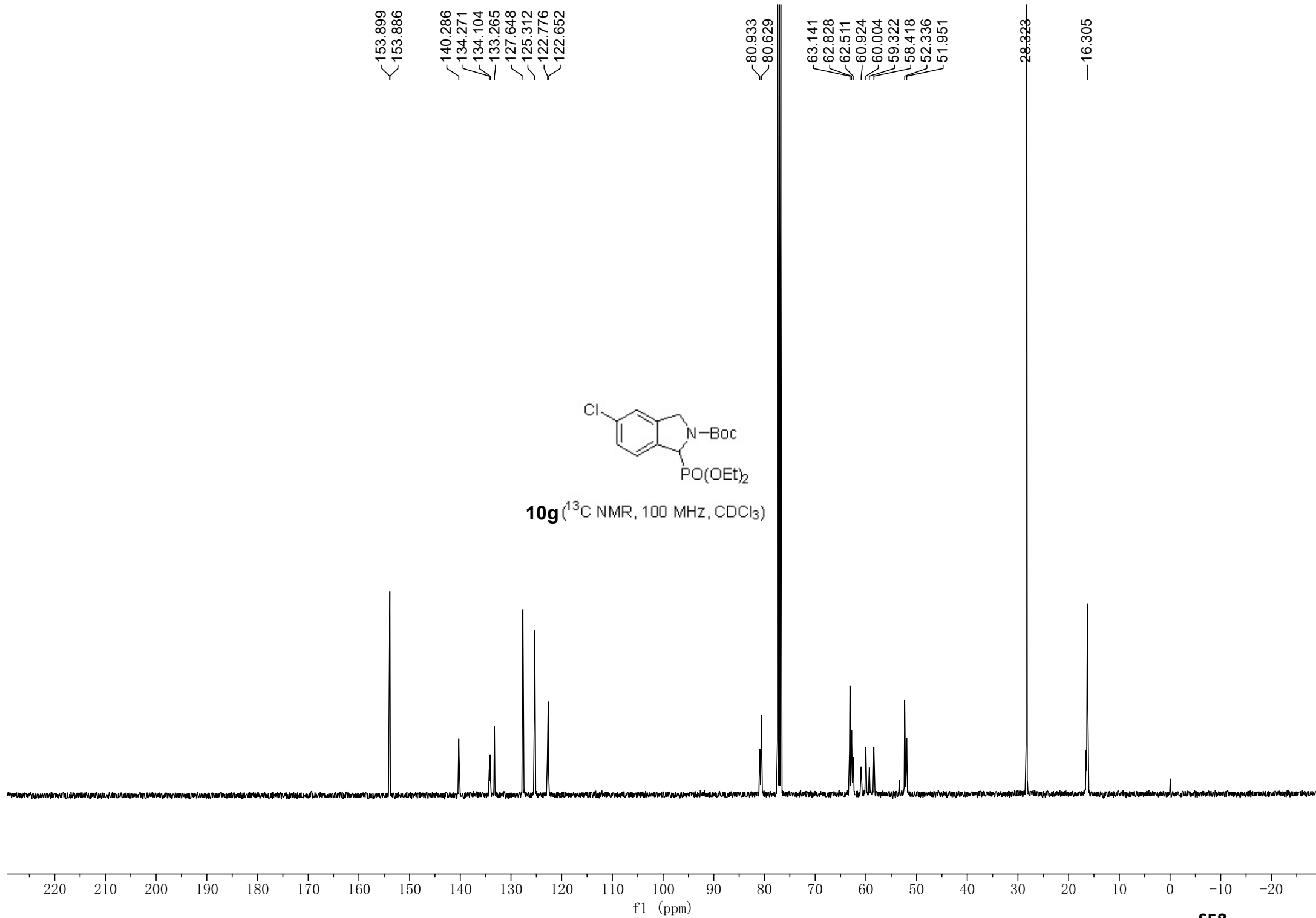
0.60  
0.40

0.40  
0.60  
1.00

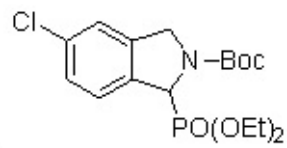
2.02  
1.00  
0.60  
0.40

9.00  
3.02  
3.00

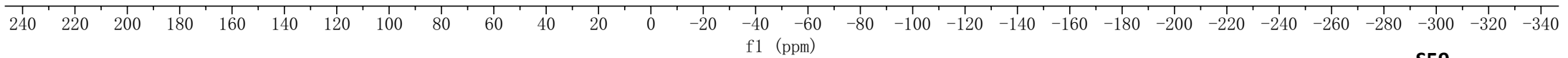


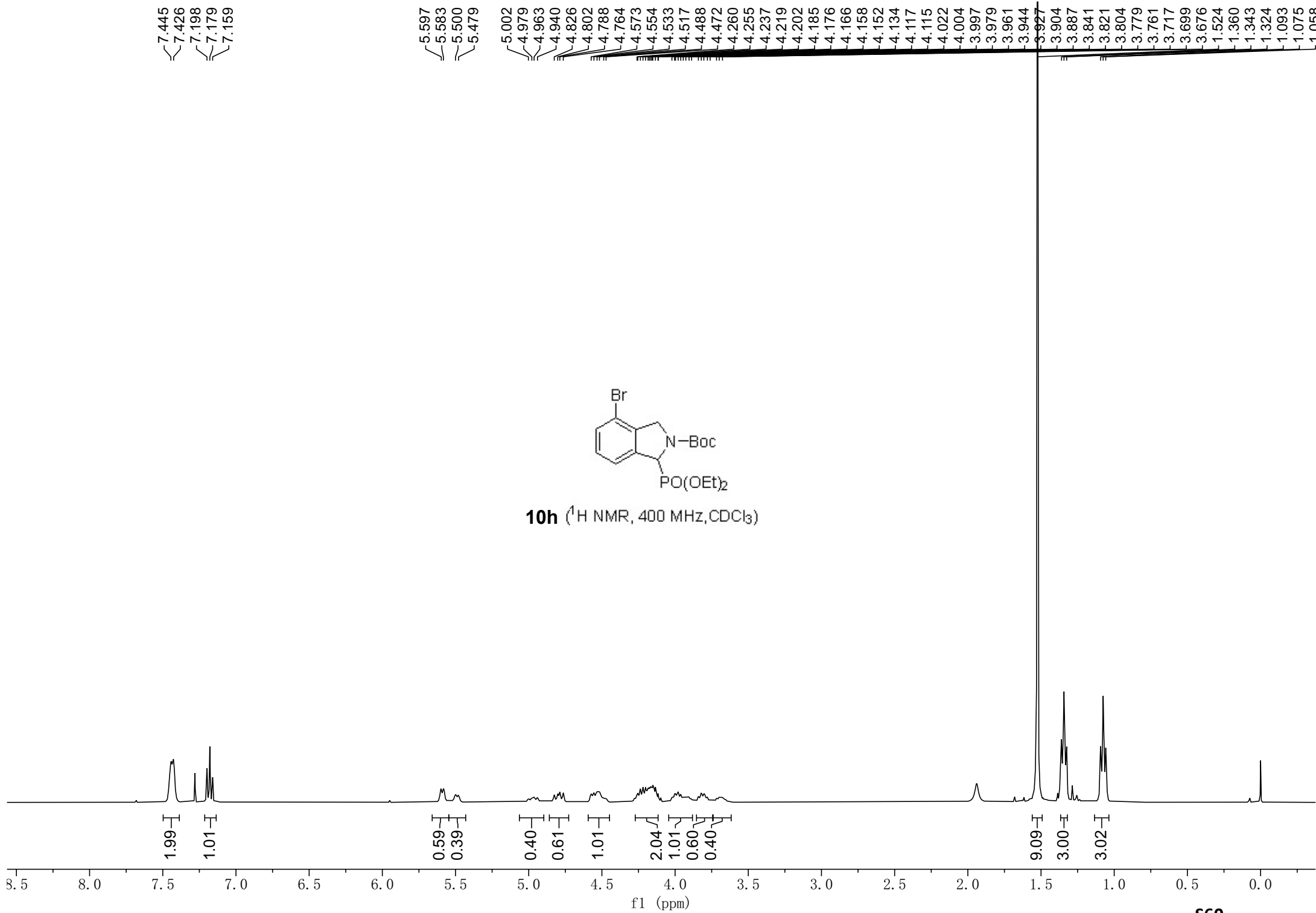


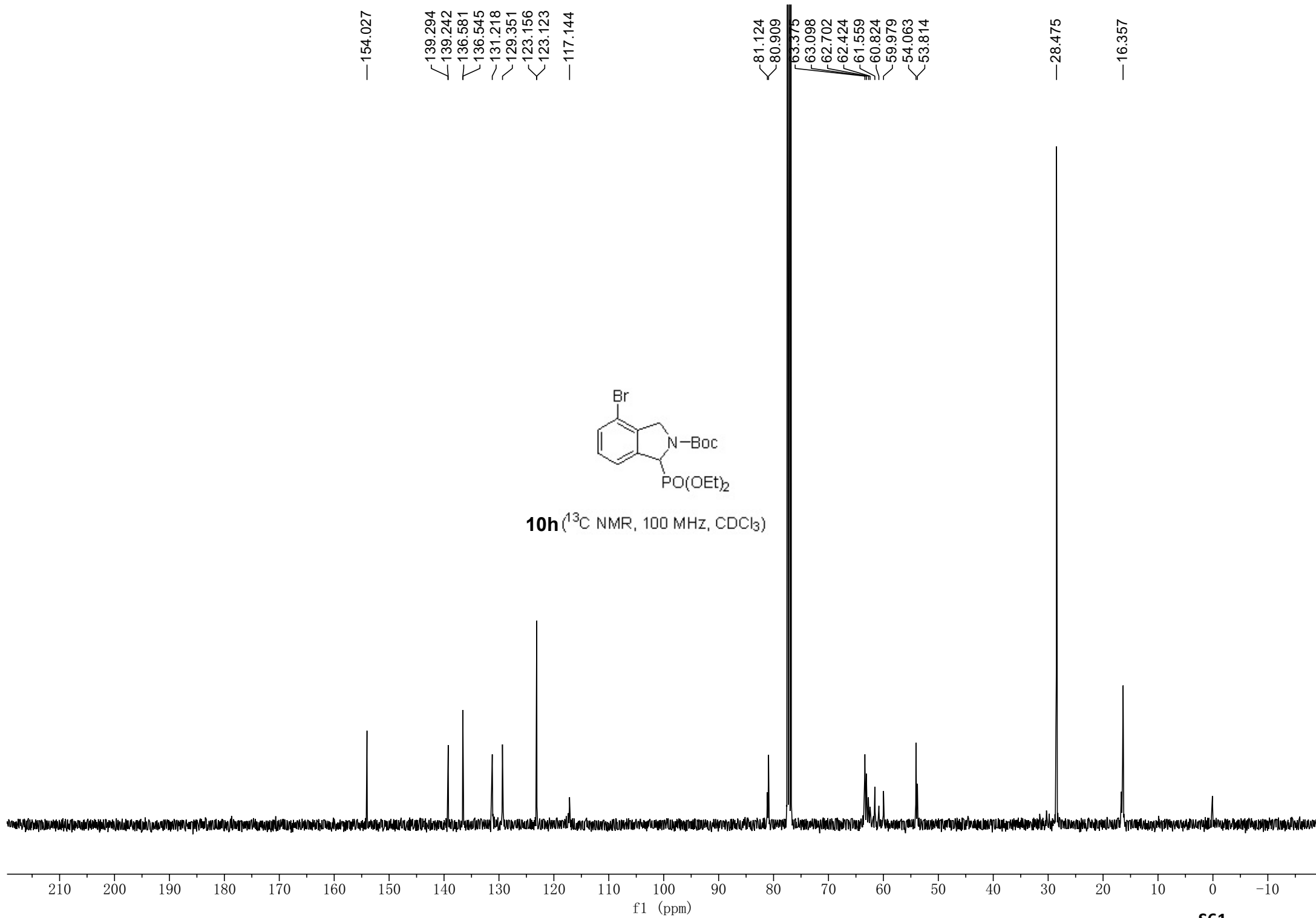
—20.063



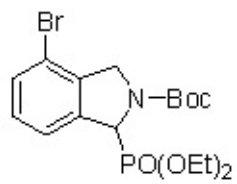
**10g** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



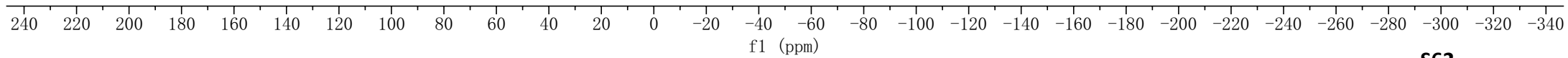




—19.987

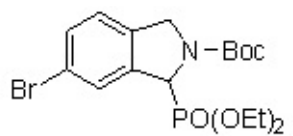


**10h** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)

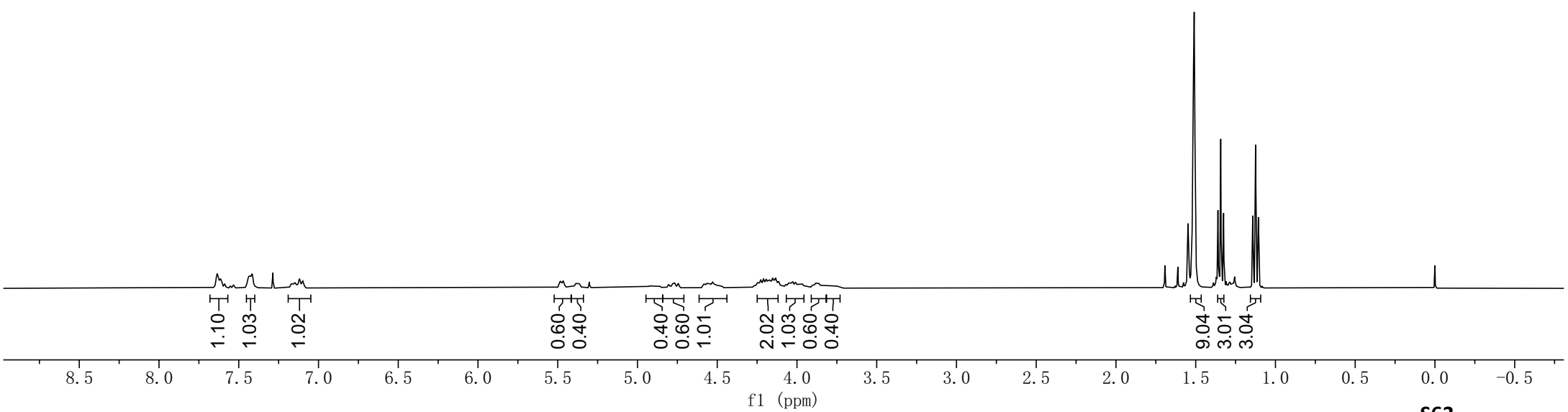


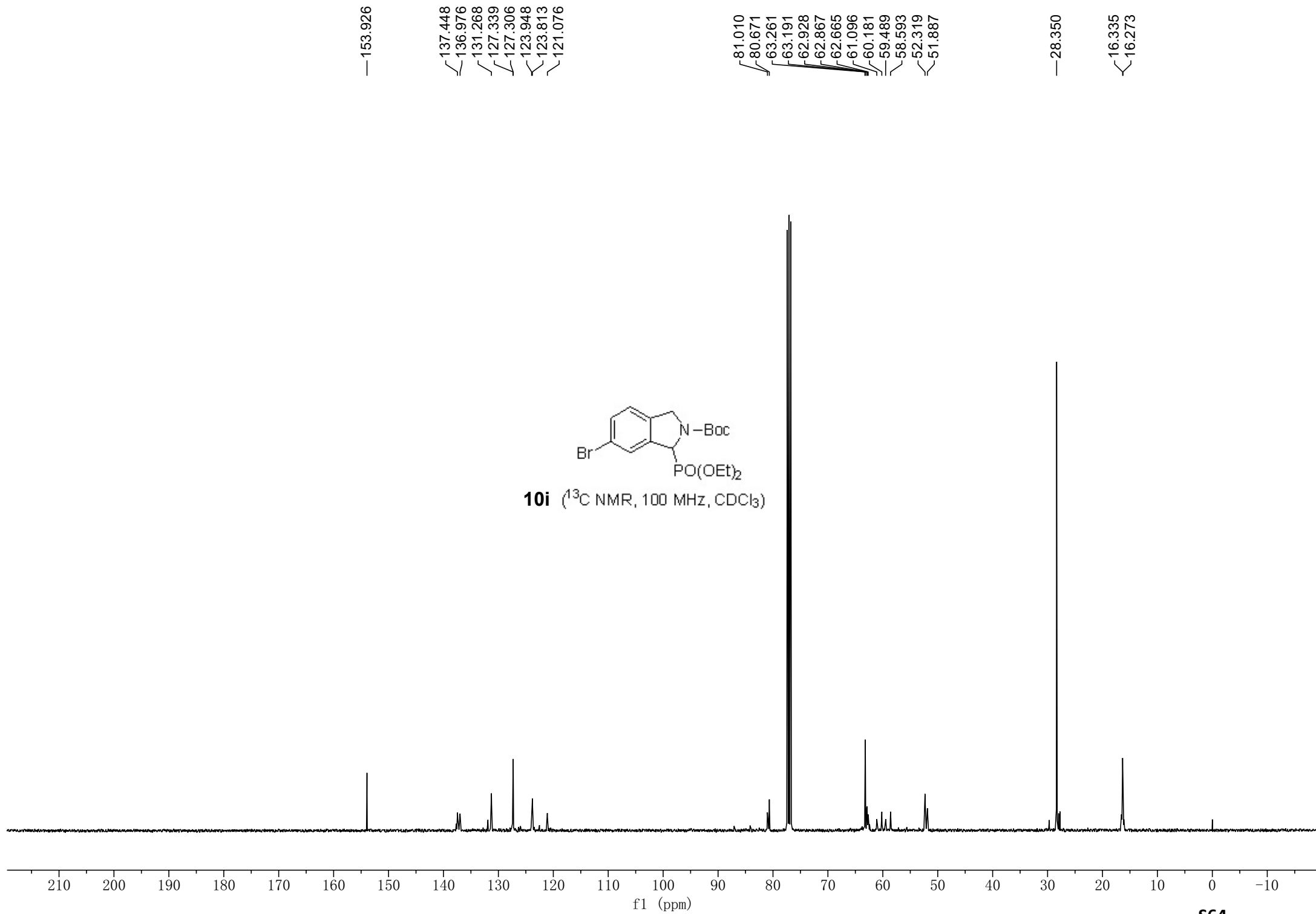
7.635  
7.613  
7.438  
7.419  
7.168  
7.148  
7.119  
7.099

5.484  
5.462  
5.386  
5.365  
4.924  
4.902  
4.887  
4.865  
4.804  
4.781  
4.767  
4.744  
4.580  
4.565  
4.547  
4.528  
4.508  
4.489  
4.472  
4.246  
4.219  
4.210  
4.201  
4.193  
4.183  
4.171  
4.165  
4.153  
4.134  
4.117  
4.052  
4.045  
4.034  
4.009  
3.972  
3.965  
3.903  
3.883  
3.861  
3.840  
3.822  
3.800  
3.777  
3.759  
3.733  
1.509  
1.361  
1.343  
1.325  
1.142  
1.125  
1.107



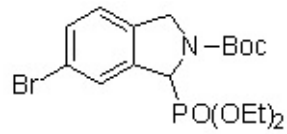
**10i** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)



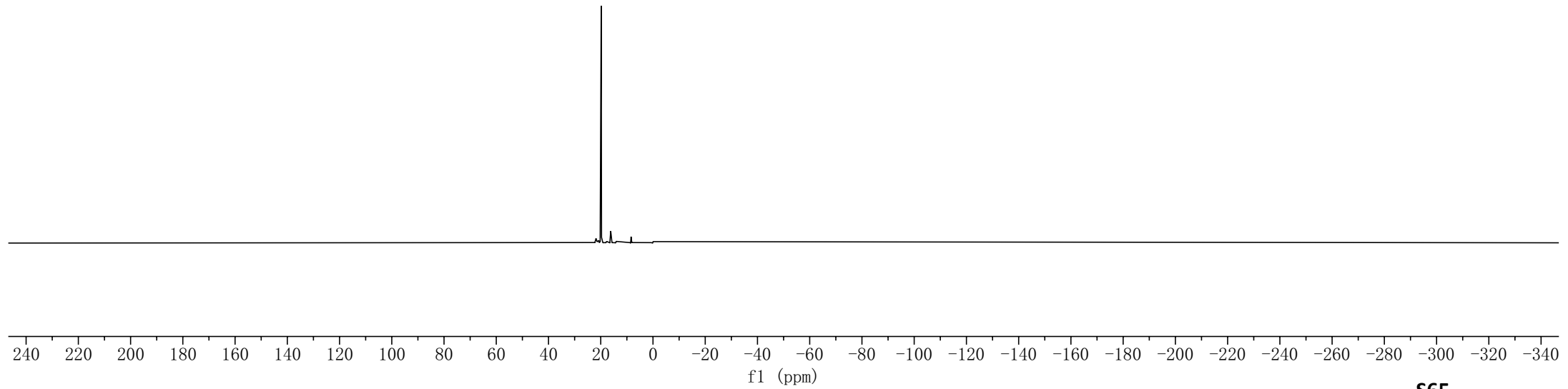


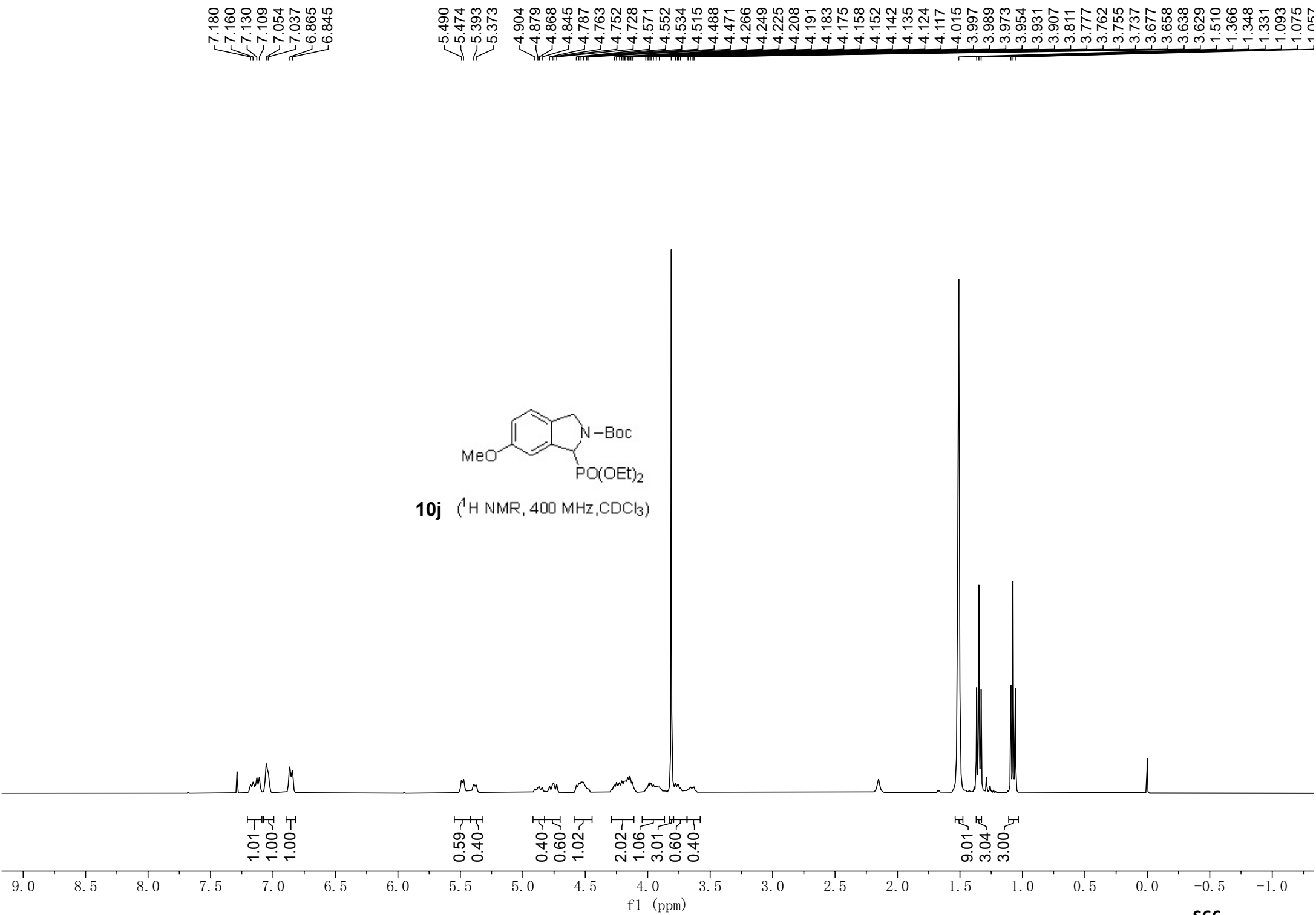


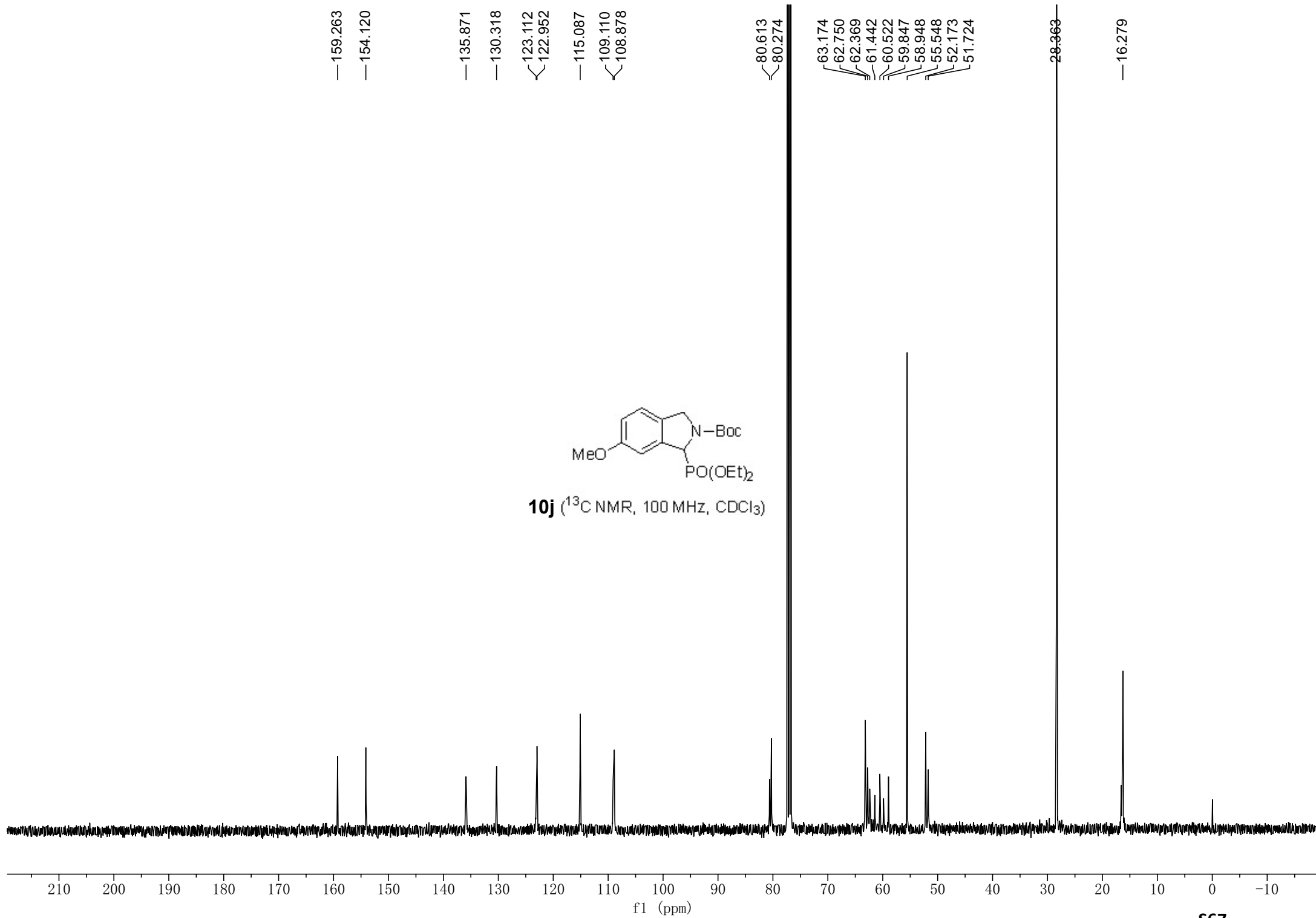
—19.824



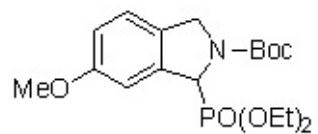
**10i** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )



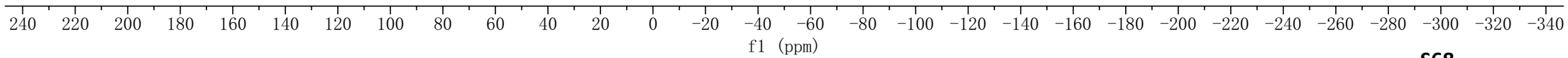




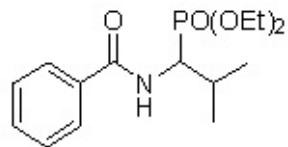
—20.593



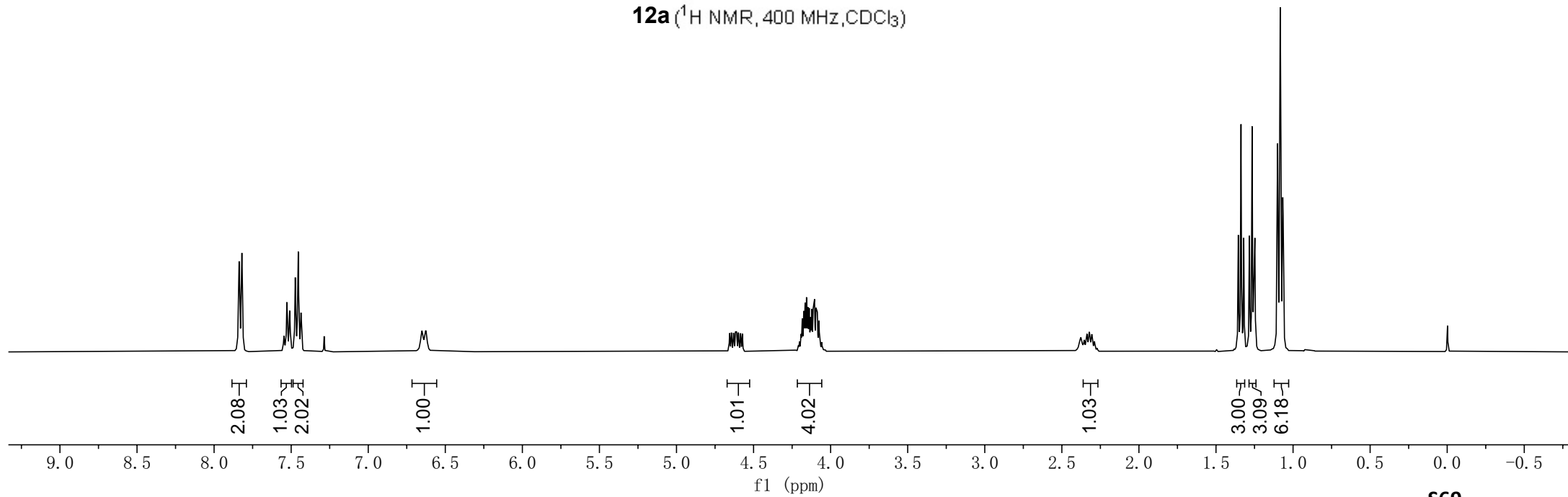
**10j** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



7.846  
7.840  
7.837  
7.833  
7.825  
7.820  
7.815  
7.546  
7.528  
7.522  
7.513  
7.510  
7.506  
7.473  
7.469  
7.458  
7.454  
7.441  
7.436  
7.433  
6.655  
6.648  
6.629  
6.622  
4.655  
4.644  
4.630  
4.618  
4.610  
4.598  
4.584  
4.572  
4.174  
4.166  
4.157  
4.147  
4.139  
4.130  
4.122  
4.113  
4.104  
4.096  
4.093  
4.086  
2.378  
2.364  
2.342  
2.337  
2.334  
2.325  
2.321  
2.317  
2.308  
2.305  
2.300  
2.288  
1.355  
1.338  
1.320  
1.283  
1.265  
1.248  
1.100  
1.083  
1.068  
1.065



**12a** ( $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$ )



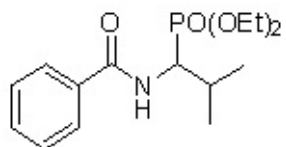
167.391  
167.337

134.144  
131.740  
128.641  
127.070

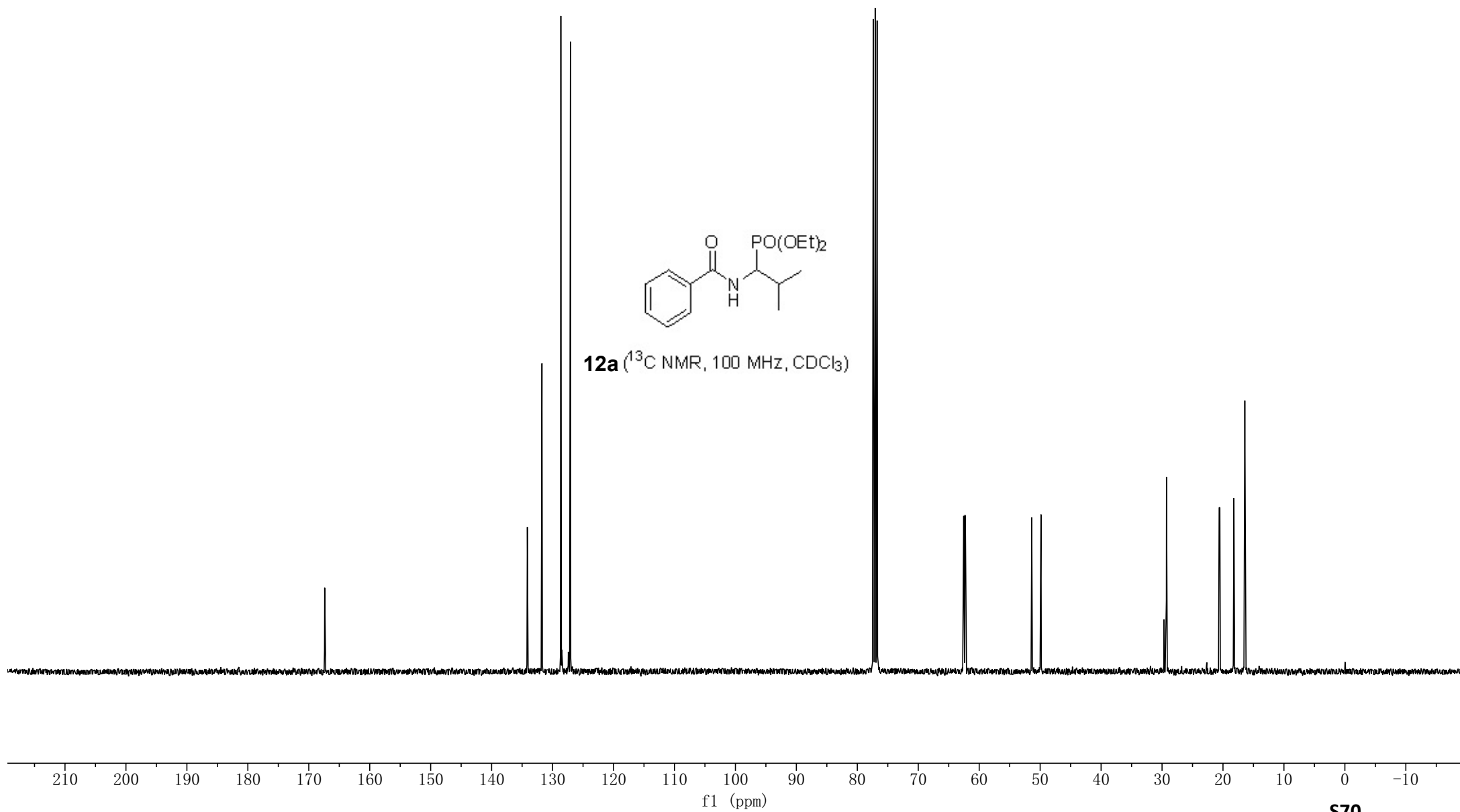
62.545  
62.475  
62.339  
62.271

51.378  
49.863

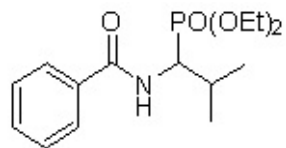
29.271  
29.237  
20.632  
20.514  
18.206  
18.155  
16.474  
16.414  
16.349



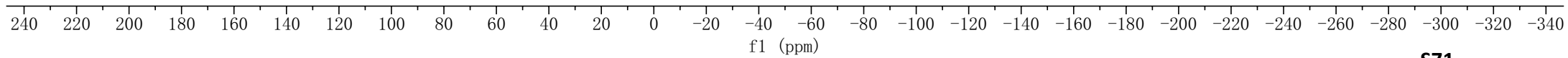
**12a** ( $^{13}\text{C}$  NMR, 100 MHz,  $\text{CDCl}_3$ )



—24.287



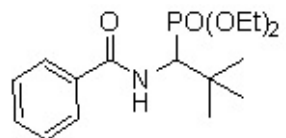
**12a** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



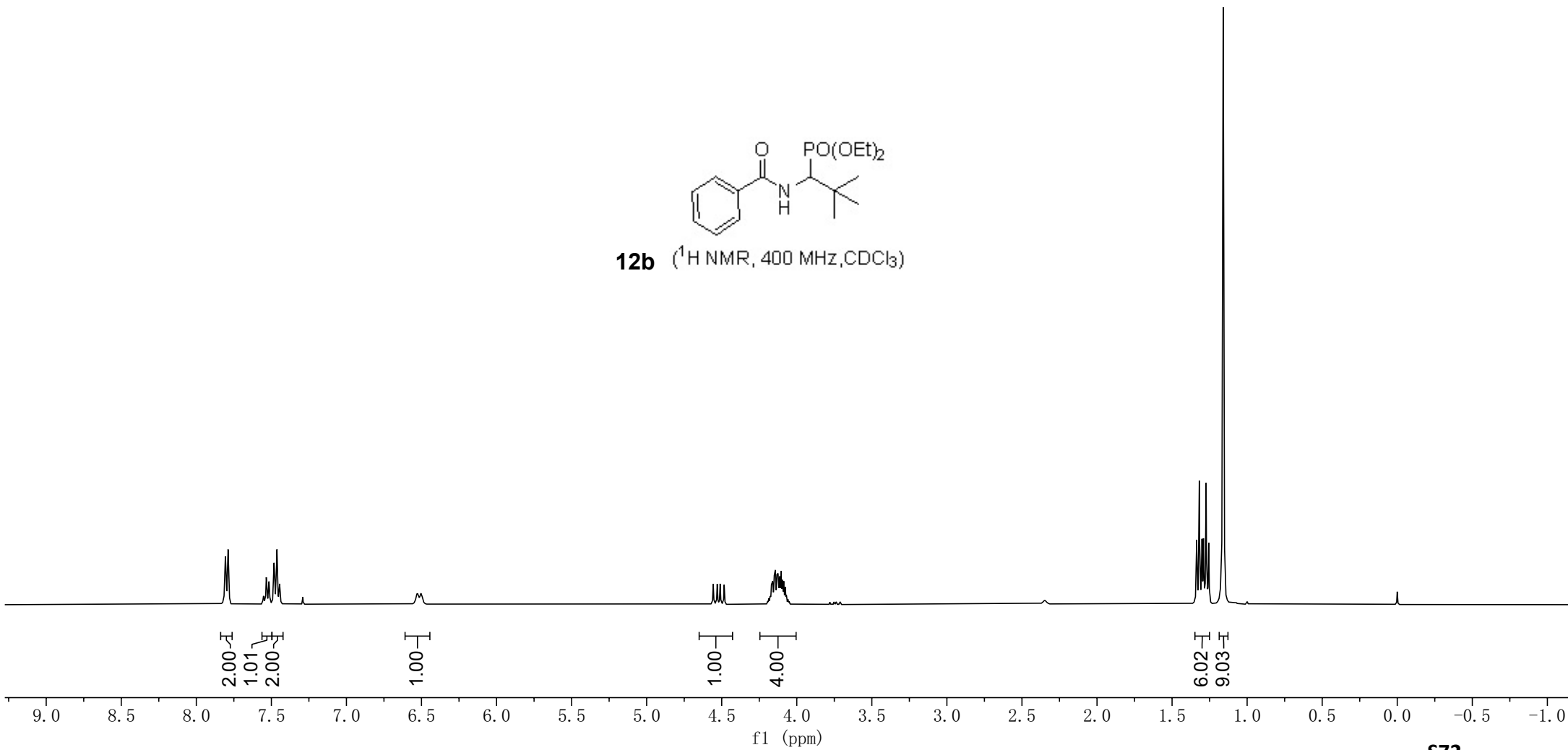
7.809  
7.806  
7.802  
7.788  
7.784  
7.556  
7.540  
7.534  
7.528  
7.519  
7.516  
7.512  
7.482  
7.467  
7.463  
7.450  
7.445  
7.442  
6.524  
6.505

4.556  
4.530  
4.510  
4.484  
4.178  
4.175  
4.169  
4.161  
4.150  
4.143  
4.132  
4.125  
4.123  
4.114  
4.105  
4.097  
4.094  
4.087  
4.077  
4.069

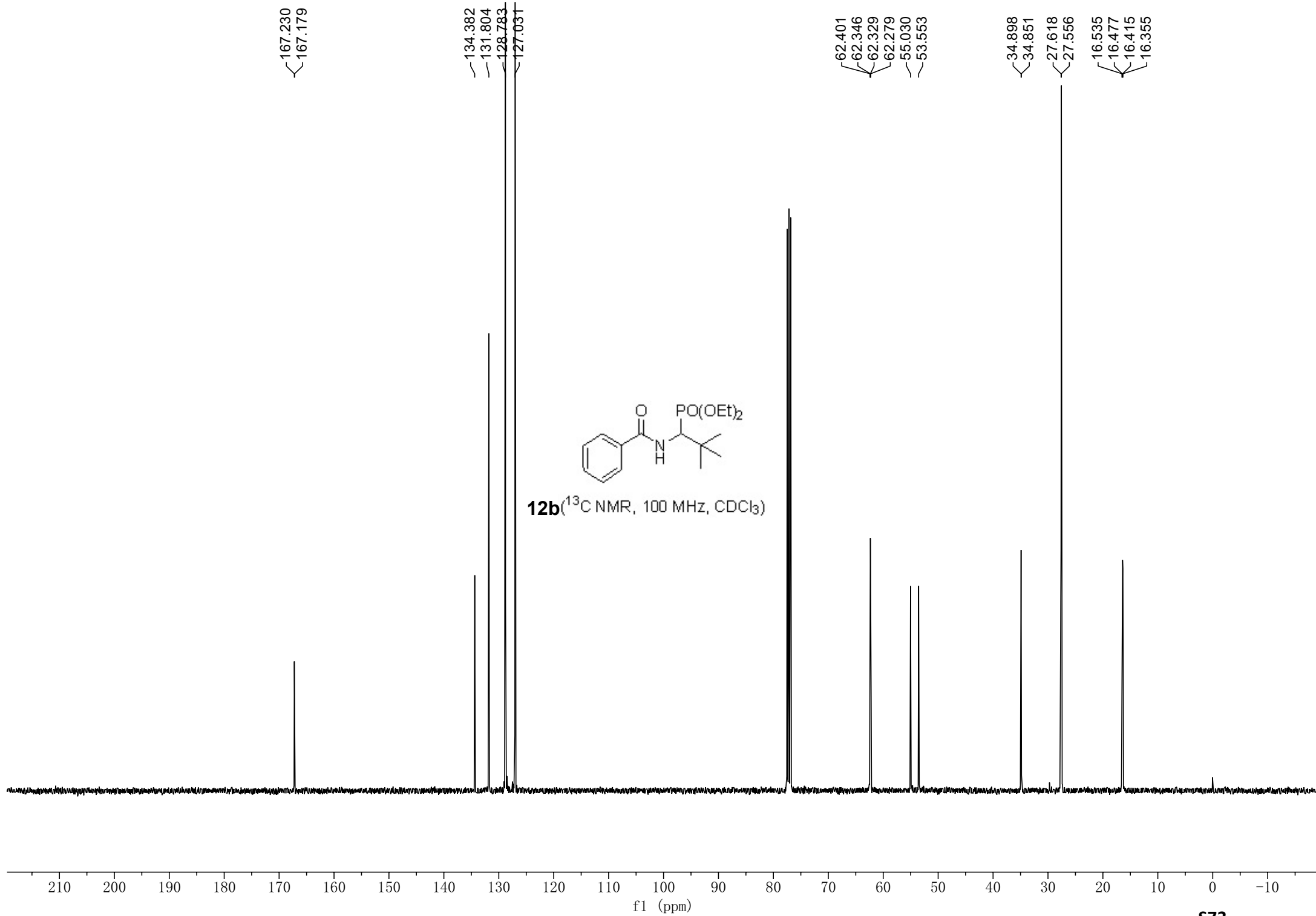
1.336  
1.319  
1.301  
1.291  
1.273  
1.256  
1.159



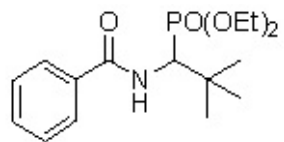
**12b** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)



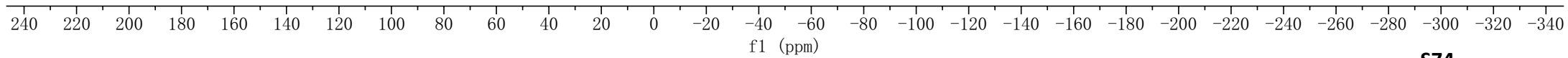




—23.711



**12b** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

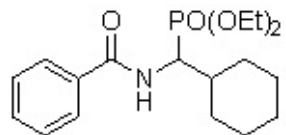


7.840  
7.822  
7.818  
7.540  
7.522  
7.503  
7.467  
7.448  
7.431  
6.691  
6.684  
6.665  
6.658

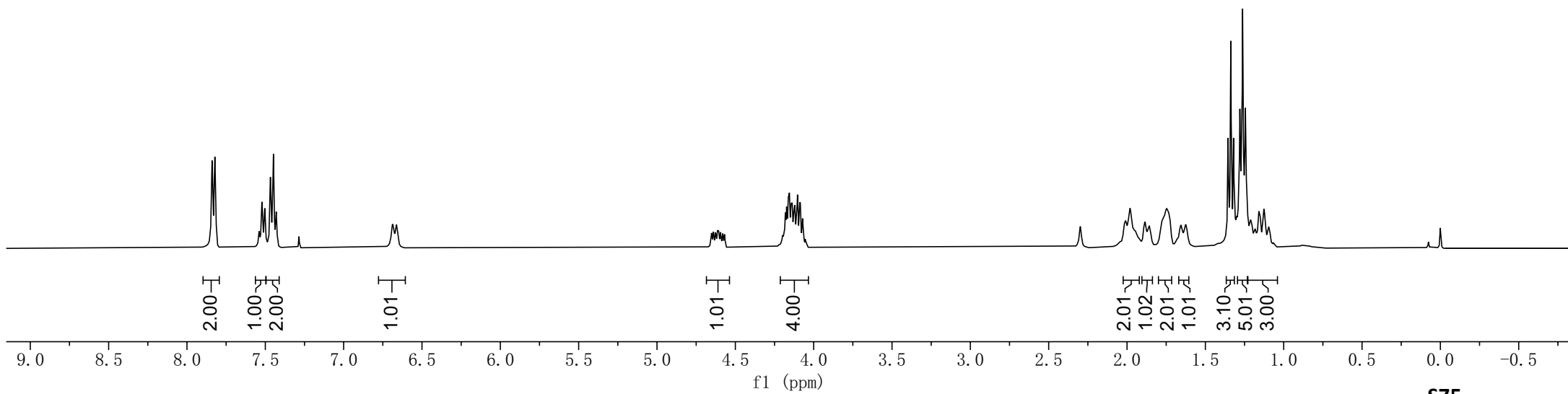
4.652  
4.640  
4.626  
4.614  
4.606  
4.594  
4.580  
4.569

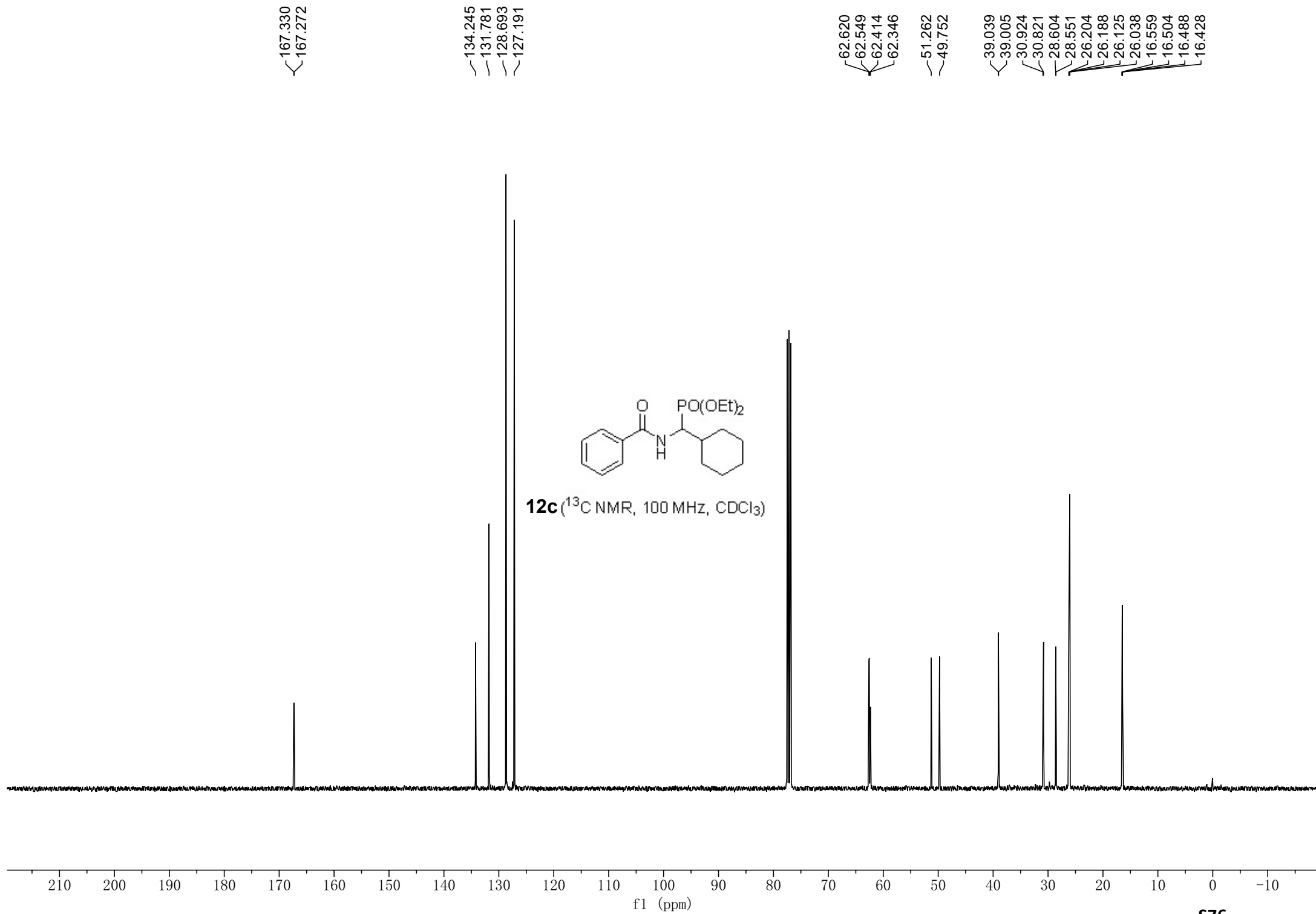
4.181  
4.173  
4.163  
4.155  
4.144  
4.137  
4.127  
4.121  
4.109  
4.103  
4.090  
4.085  
4.071

2.012  
1.987  
1.979  
1.967  
1.890  
1.884  
1.858  
1.777  
1.768  
1.757  
1.747  
1.736  
1.726  
1.656  
1.624  
1.355  
1.338  
1.320  
1.280  
1.262  
1.244  
1.236  
1.218  
1.211  
1.204  
1.159  
1.151  
1.127  
1.119

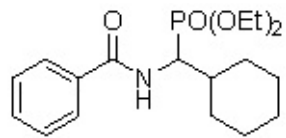


**12c** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

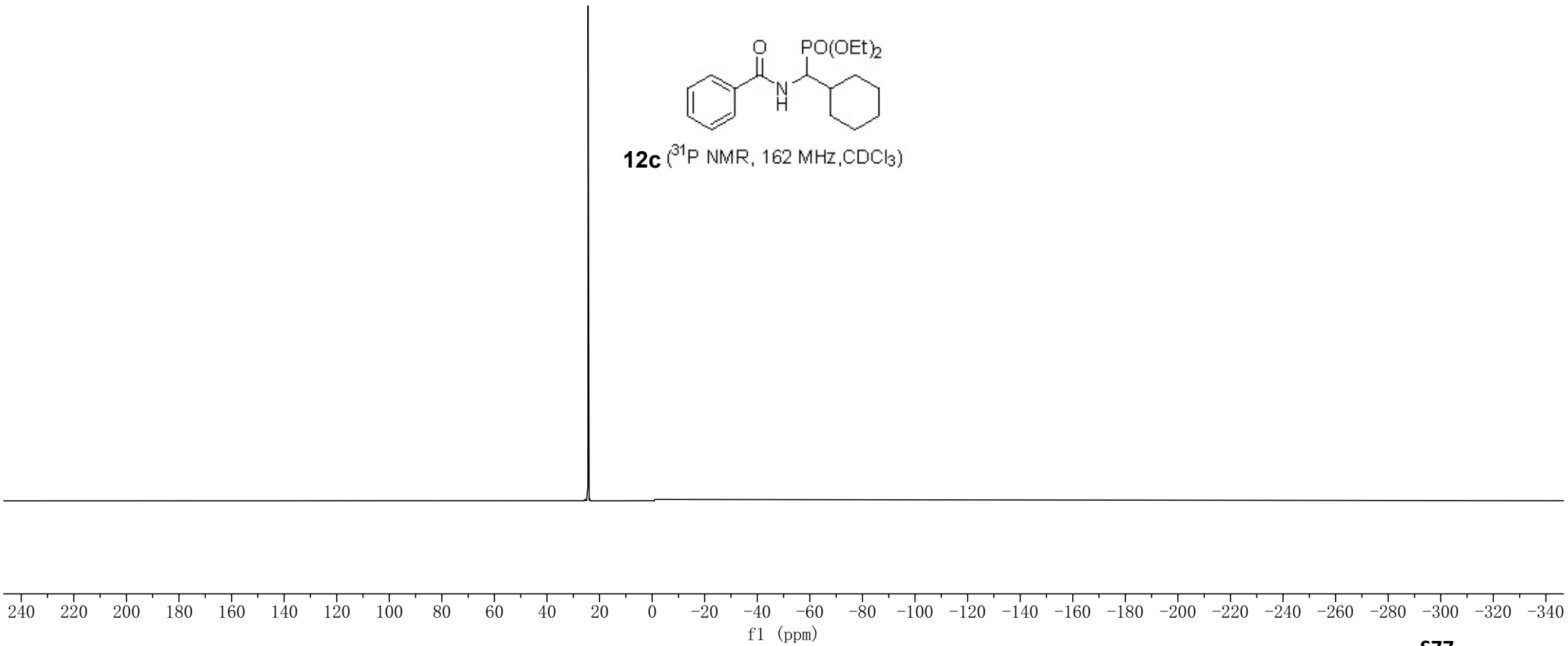




—24.363



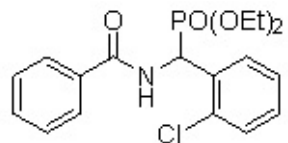
**12c** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )



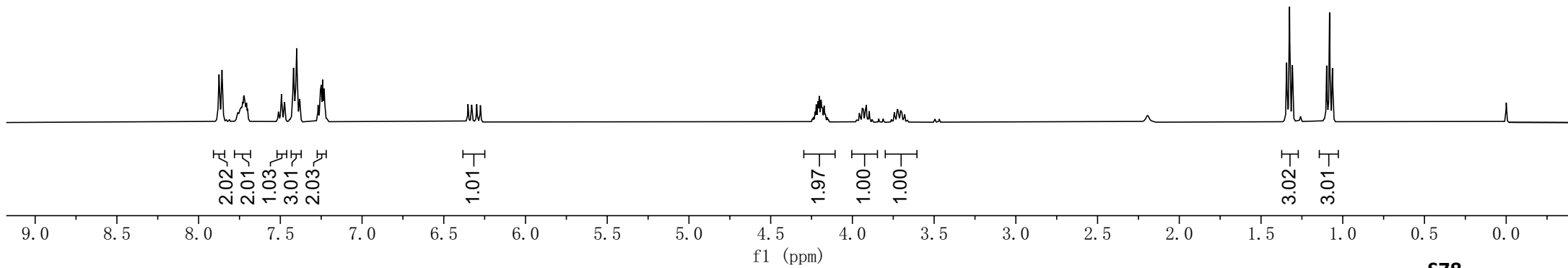
7.878  
7.857  
7.853  
7.740  
7.730  
7.724  
7.713  
7.706  
7.700  
7.511  
7.493  
7.488  
7.478  
7.420  
7.399  
7.386  
7.269  
7.255  
7.250  
7.241  
7.236  
7.232  
6.352  
6.329  
6.298  
6.276

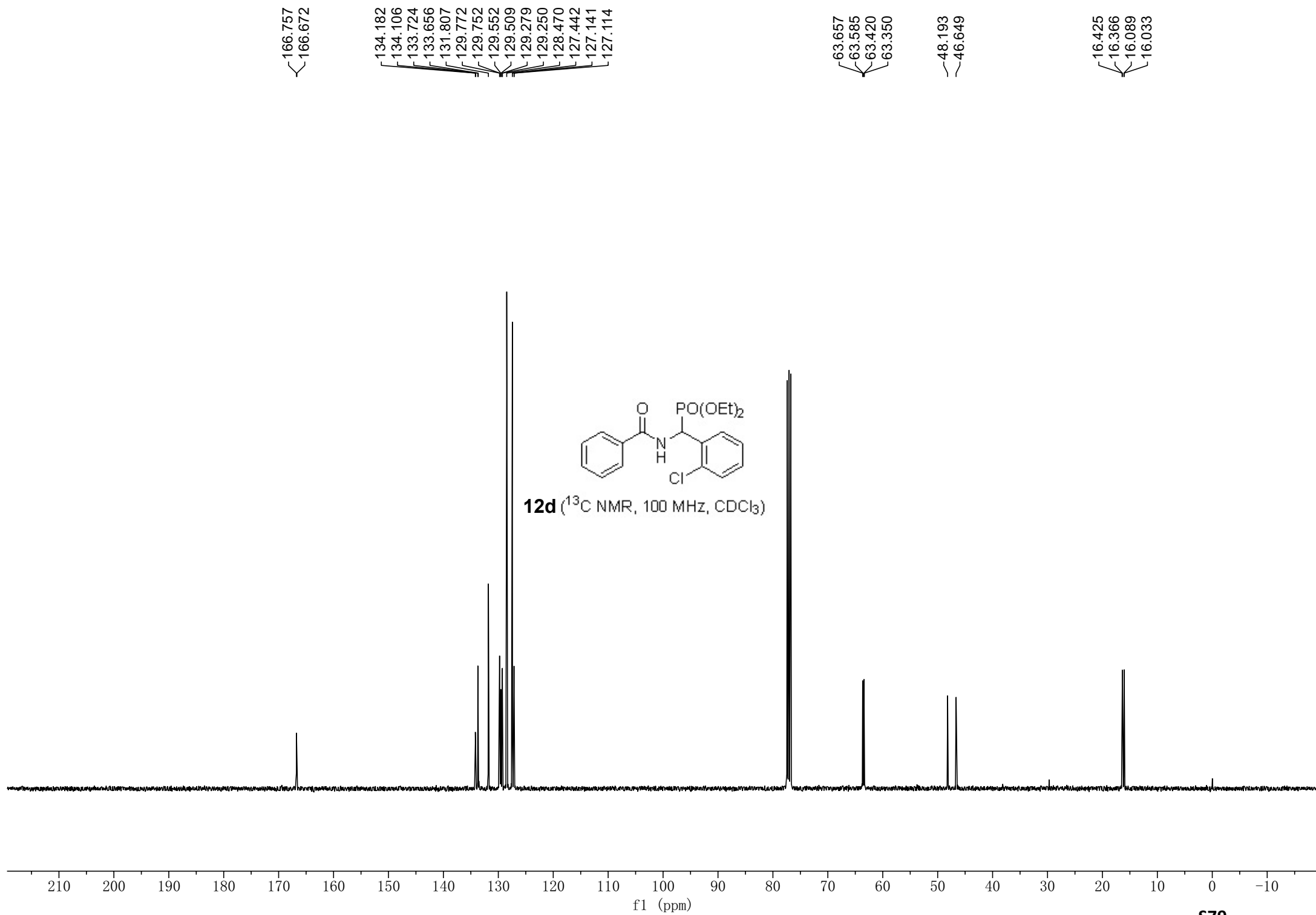
4.228  
4.221  
4.211  
4.203  
4.193  
4.190  
4.185  
4.173  
4.166  
3.958  
3.950  
3.940  
3.932  
3.922  
3.915  
3.897  
3.761  
3.743  
3.740  
3.736  
3.726  
3.723  
3.718  
3.715  
3.709  
3.705  
3.701  
3.698  
3.688  
3.683  
3.680

1.343  
1.326  
1.308  
1.098  
1.081  
1.063

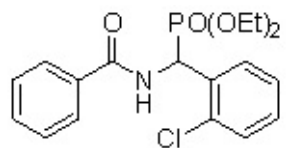


**12d** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

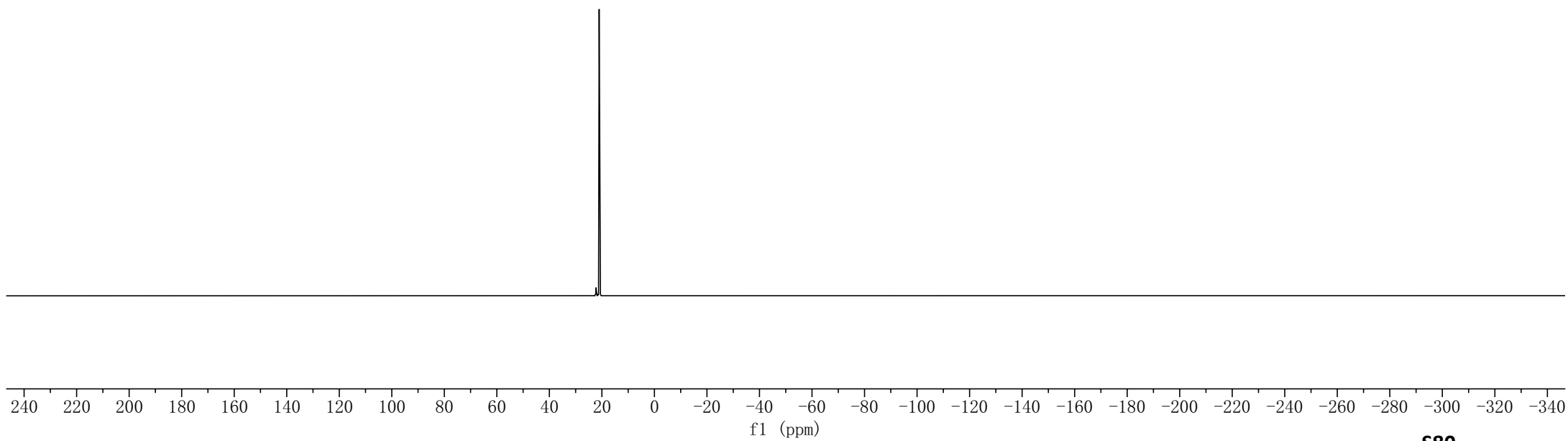




—21.040

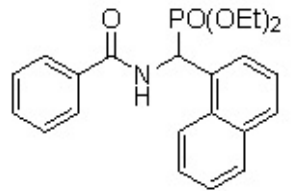


**12d** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

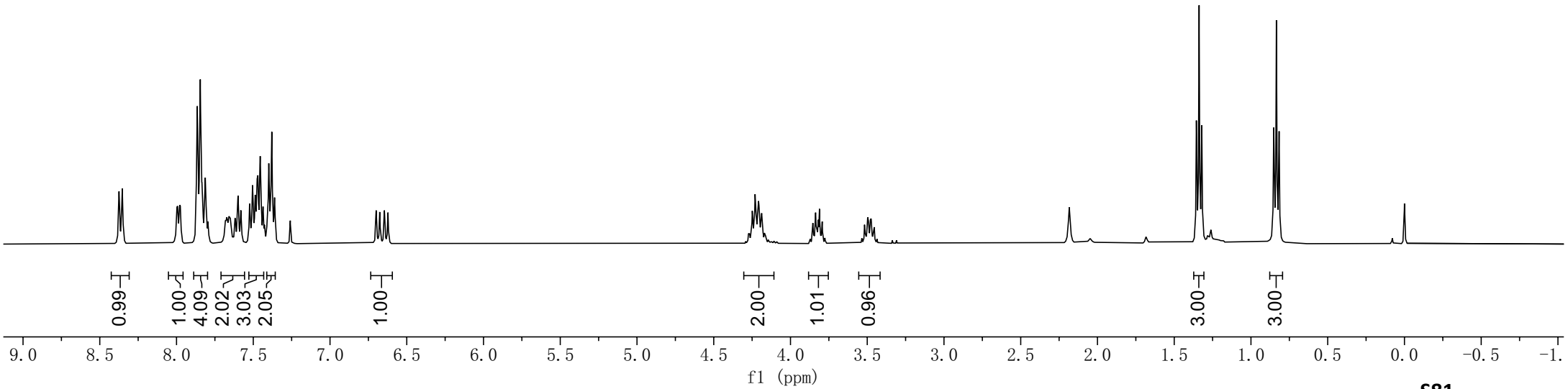


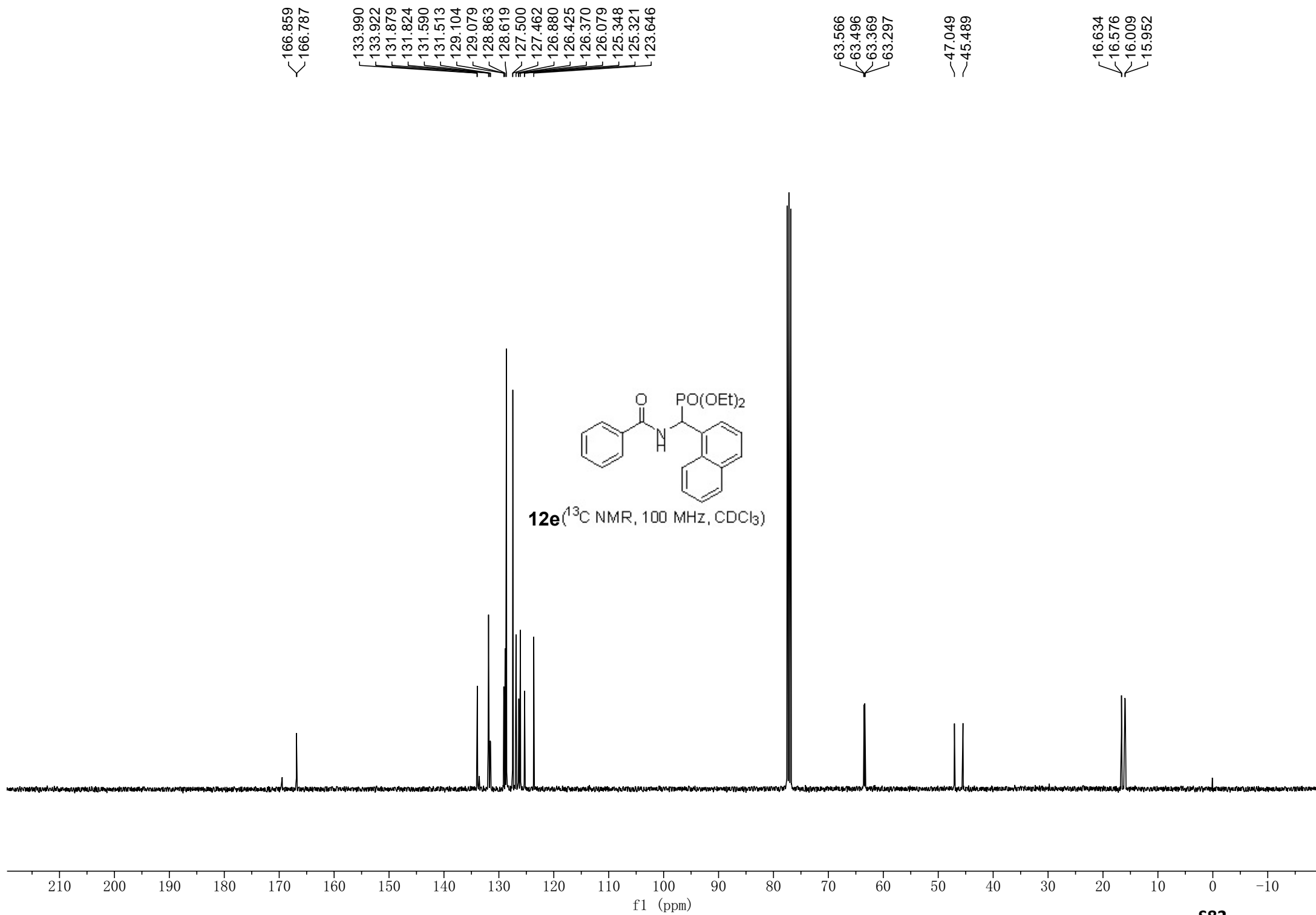


8.375  
8.353  
8.000  
7.993  
7.980  
7.973  
7.865  
7.846  
7.841  
7.833  
7.812  
7.682  
7.672  
7.658  
7.648  
7.620  
7.616  
7.603  
7.599  
7.595  
7.581  
7.578  
7.525  
7.522  
7.508  
7.505  
7.501  
7.488  
7.485  
7.475  
7.470  
7.464  
7.455  
7.451  
7.448  
7.436  
7.407  
7.399  
7.394  
7.380  
7.366  
7.361  
7.361  
6.999  
6.676  
6.646  
6.623  
4.256  
4.249  
4.244  
4.238  
4.231  
4.225  
4.213  
4.208  
4.205  
4.201  
4.194  
4.188  
4.183  
4.180  
3.854  
3.836  
3.829  
3.819  
3.811  
3.793  
3.517  
3.500  
3.497  
3.492  
3.479  
3.474  
3.471  
3.454  
1.356  
1.338  
1.320  
-0.852  
-0.834  
0.817

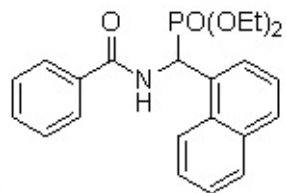


**12e** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

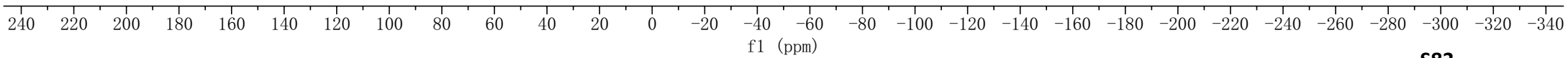




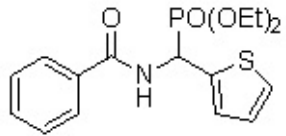
—22.134



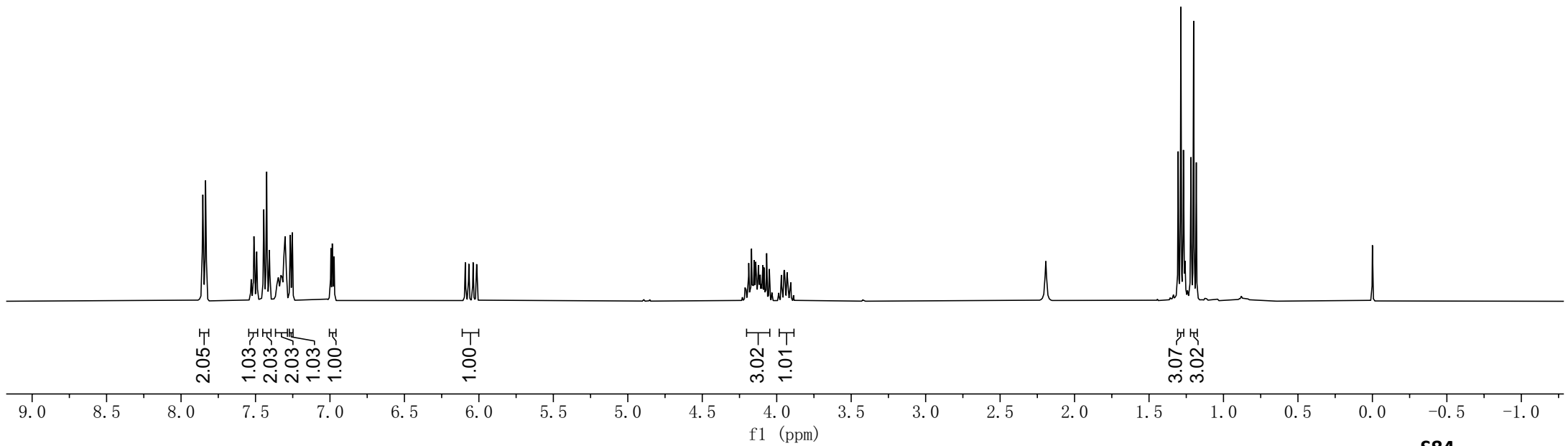
**12e** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

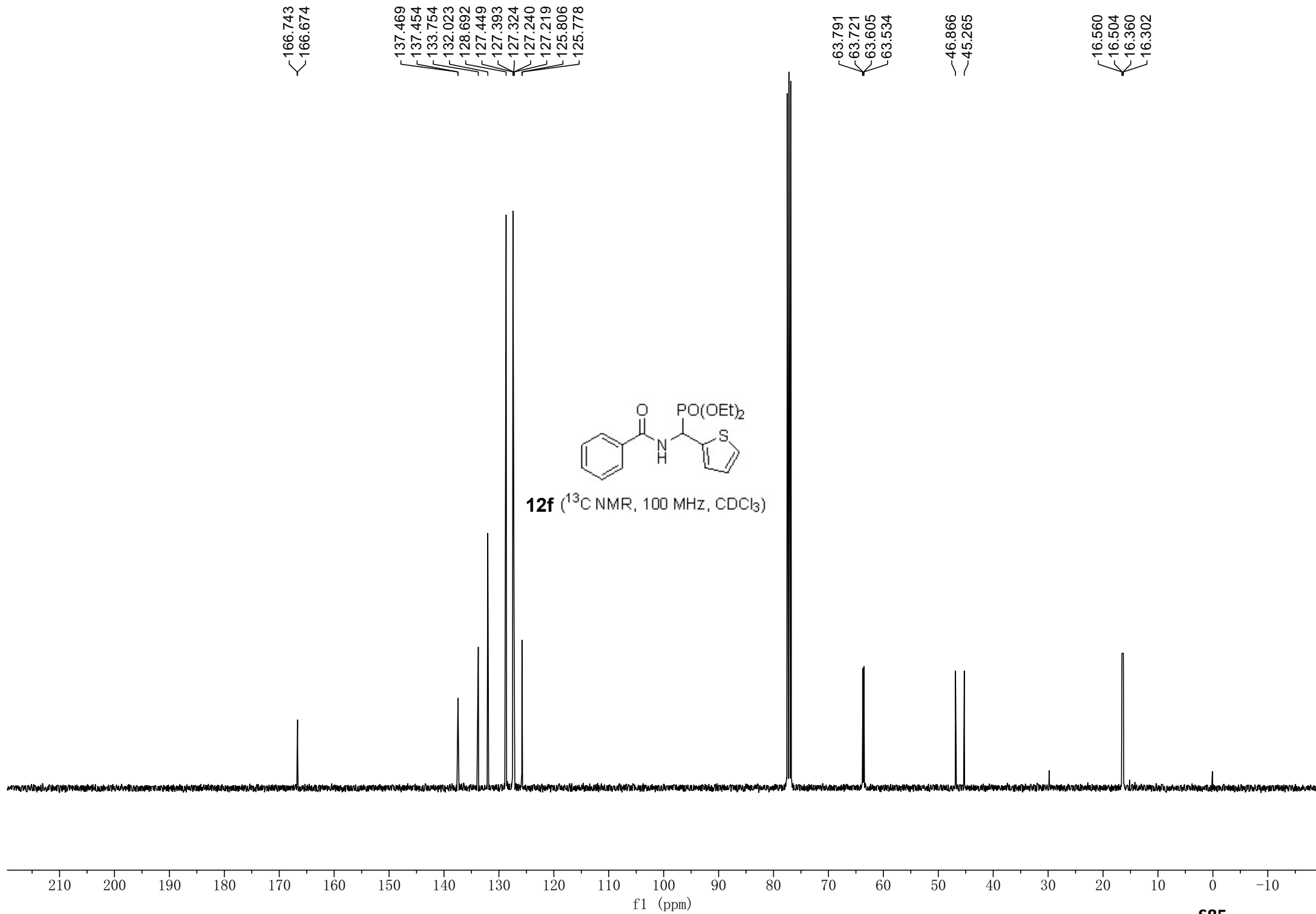


7.856  
7.853  
7.849  
7.836  
7.832  
7.529  
7.516  
7.510  
7.505  
7.495  
7.492  
7.489  
7.444  
7.429  
7.425  
7.407  
7.404  
7.353  
7.345  
7.329  
7.328  
7.321  
7.313  
7.310  
7.307  
7.304  
7.301  
7.298  
7.295  
7.292  
7.269  
7.266  
7.255  
7.253  
6.994  
6.985  
6.981  
6.972  
6.091  
6.067  
6.039  
6.015  
4.195  
4.188  
4.179  
4.176  
4.169  
4.162  
4.158  
4.151  
4.144  
4.141  
4.137  
4.133  
4.127  
4.123  
4.119  
4.116  
4.111  
4.106  
4.098  
4.093  
4.086  
4.075  
4.068  
4.050  
3.969  
3.951  
3.948  
3.943  
3.930  
3.926  
3.923  
3.905  
1.304  
1.286  
1.269  
1.218  
1.200  
1.183

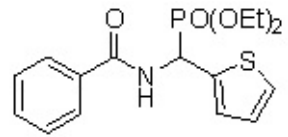


**12f** ( $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$ )

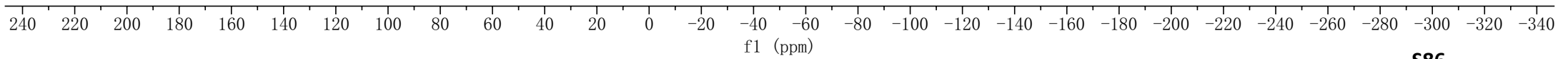




—19.886

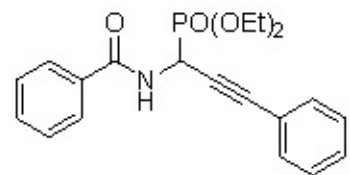


**12f** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

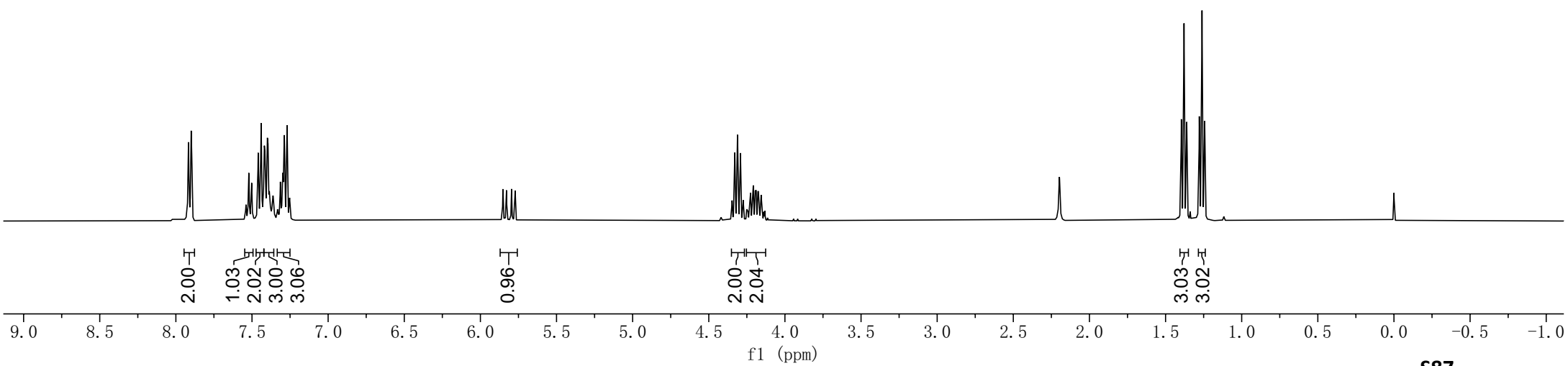


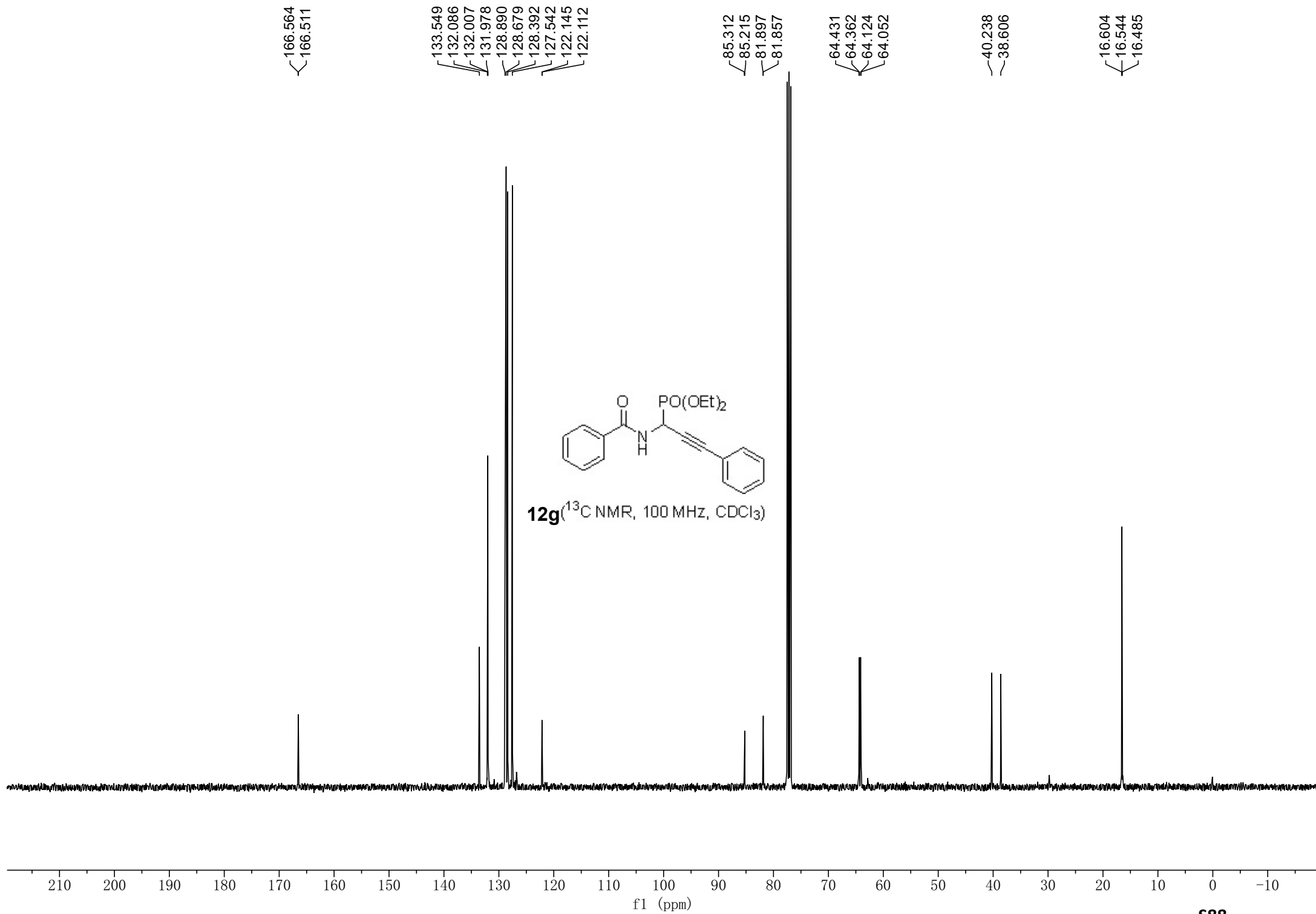
7.920  
7.917  
7.899  
7.895

7.539  
7.536  
7.527  
7.521  
7.515  
7.506  
7.502  
7.499  
7.459  
7.455  
7.444  
7.440  
7.436  
7.426  
7.422  
7.419  
7.415  
7.410  
7.405  
7.399  
7.395  
7.386  
7.383  
7.364  
7.359  
7.314  
7.307  
7.301  
7.297  
7.292  
7.288  
7.284  
7.274  
7.270  
7.265  
7.253  
5.852  
5.829  
5.795  
5.772  
4.347  
4.333  
4.329  
4.312  
4.309  
4.292  
4.274  
4.251  
4.233  
4.226  
4.214  
4.207  
4.195  
4.191  
4.189  
4.178  
4.174  
4.170  
4.160  
4.156  
4.152  
4.149  
4.131  
1.396  
1.379  
1.361  
1.278  
1.261  
1.243



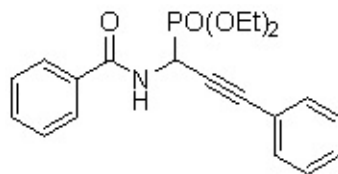
**12g** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)



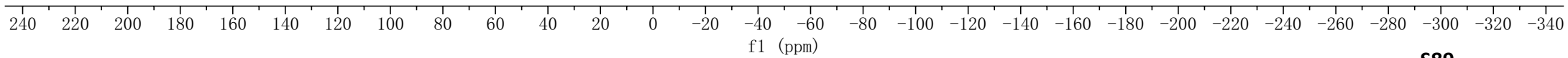




—17.199

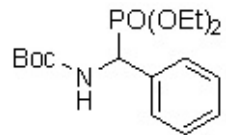


**12g** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

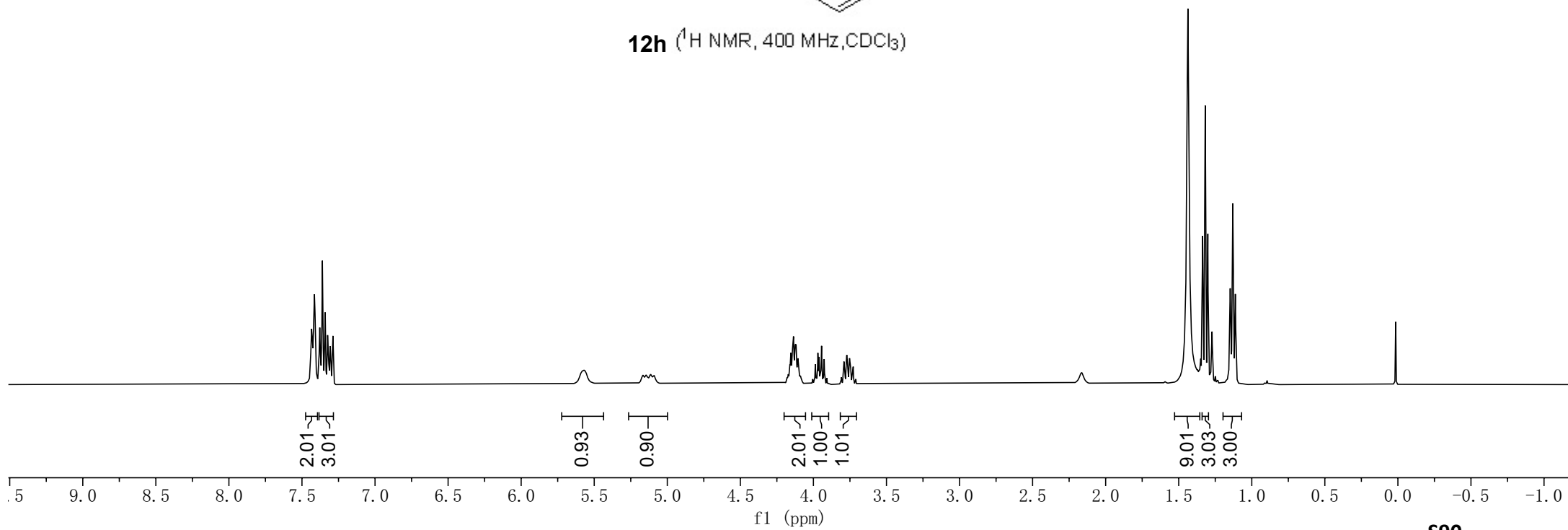


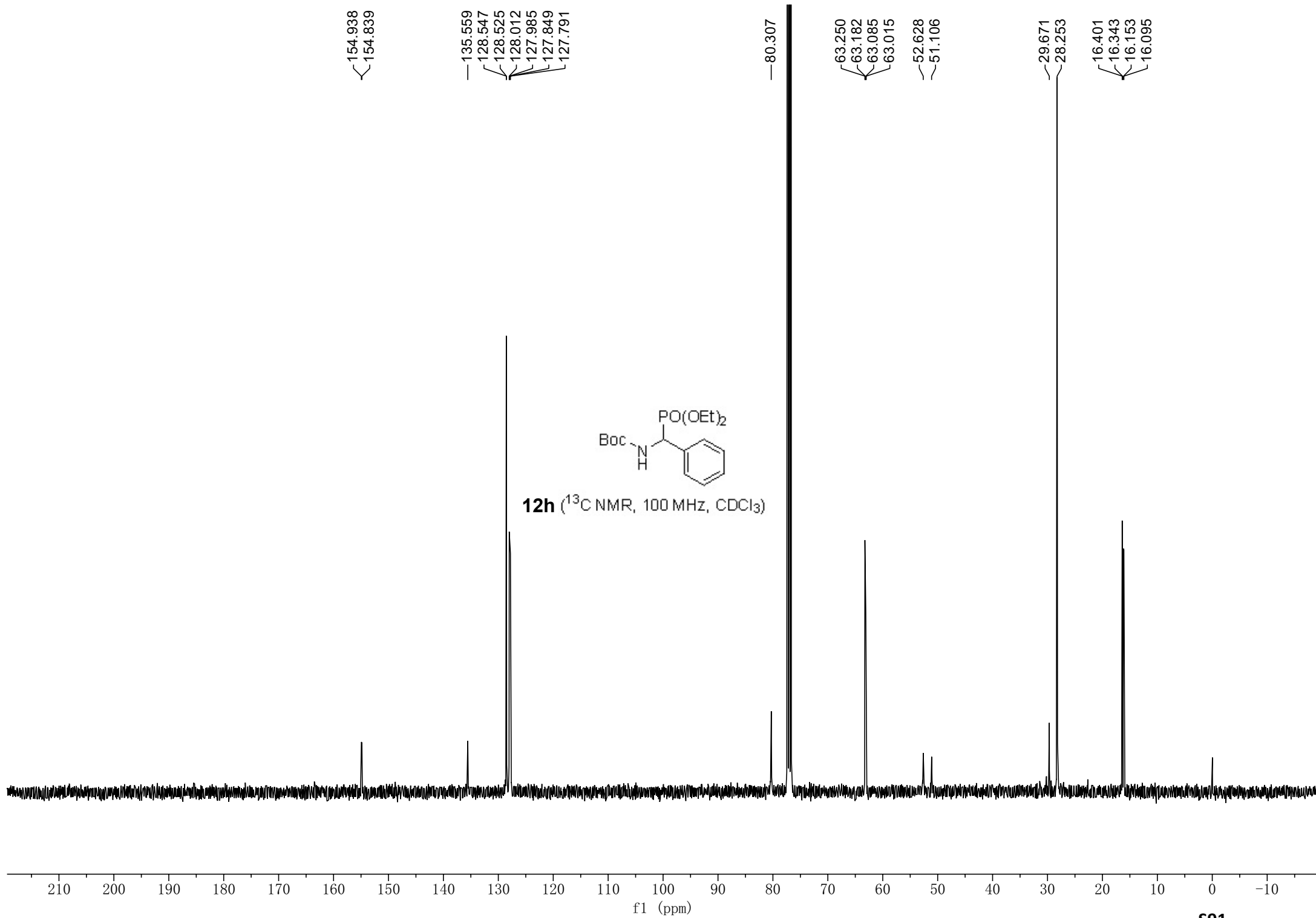
7.435  
7.430  
7.414  
7.378  
7.360  
7.341  
7.325  
7.321  
7.307  
7.303  
7.288

5.572  
5.168  
5.144  
5.115  
5.095  
4.179  
4.172  
4.161  
4.154  
4.143  
4.136  
4.125  
4.118  
4.106  
4.088  
4.005  
3.987  
3.980  
3.969  
3.962  
3.952  
3.944  
3.934  
3.926  
3.909  
3.809  
3.791  
3.783  
3.773  
3.770  
3.765  
3.753  
3.748  
3.745  
3.735  
3.731  
3.714  
3.710  
1.437  
1.336  
1.319  
1.301  
1.148  
1.130  
1.112

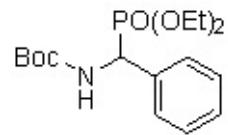


**12h** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

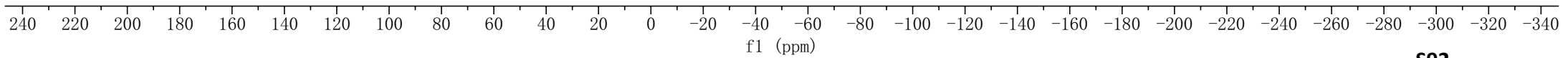


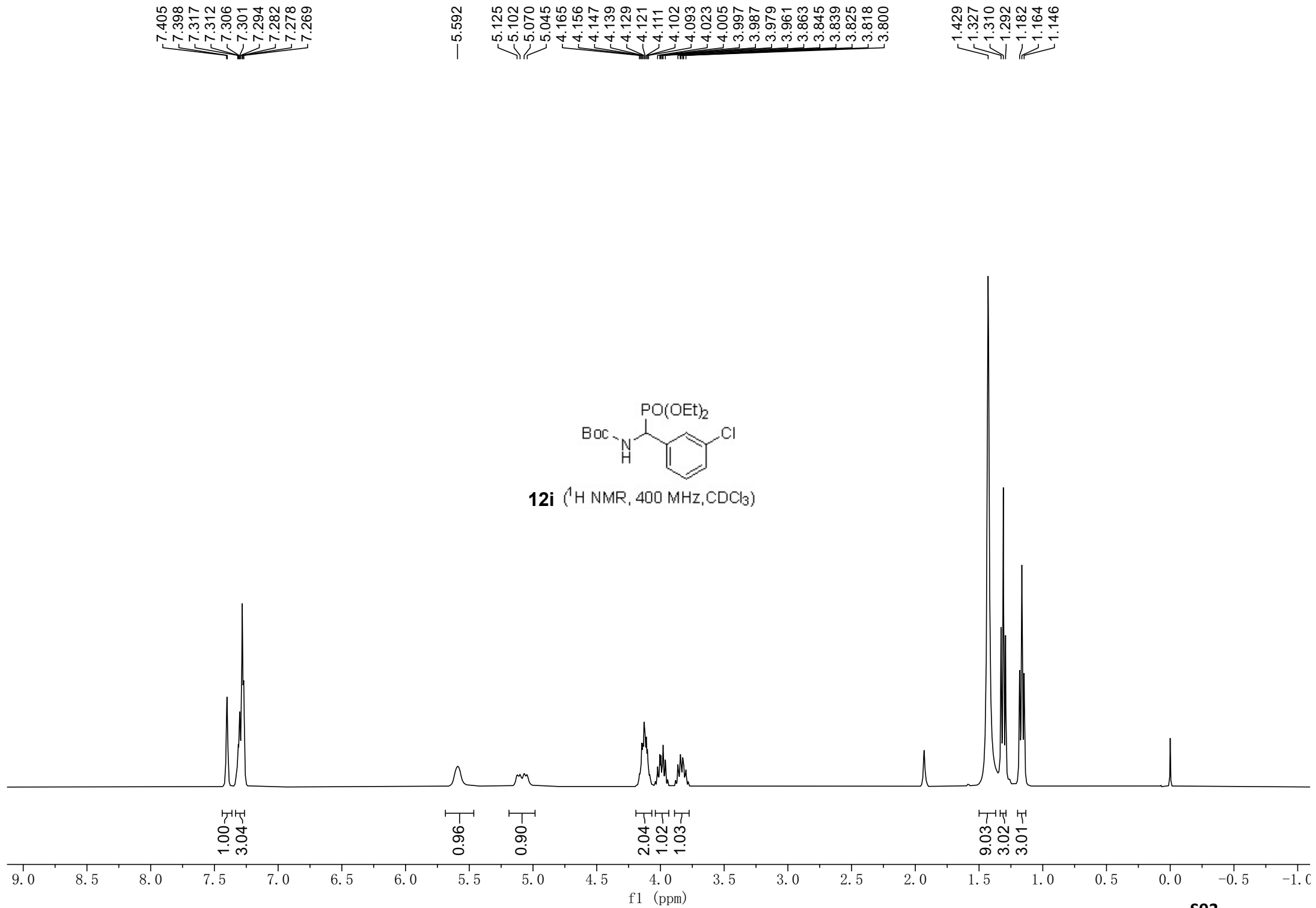


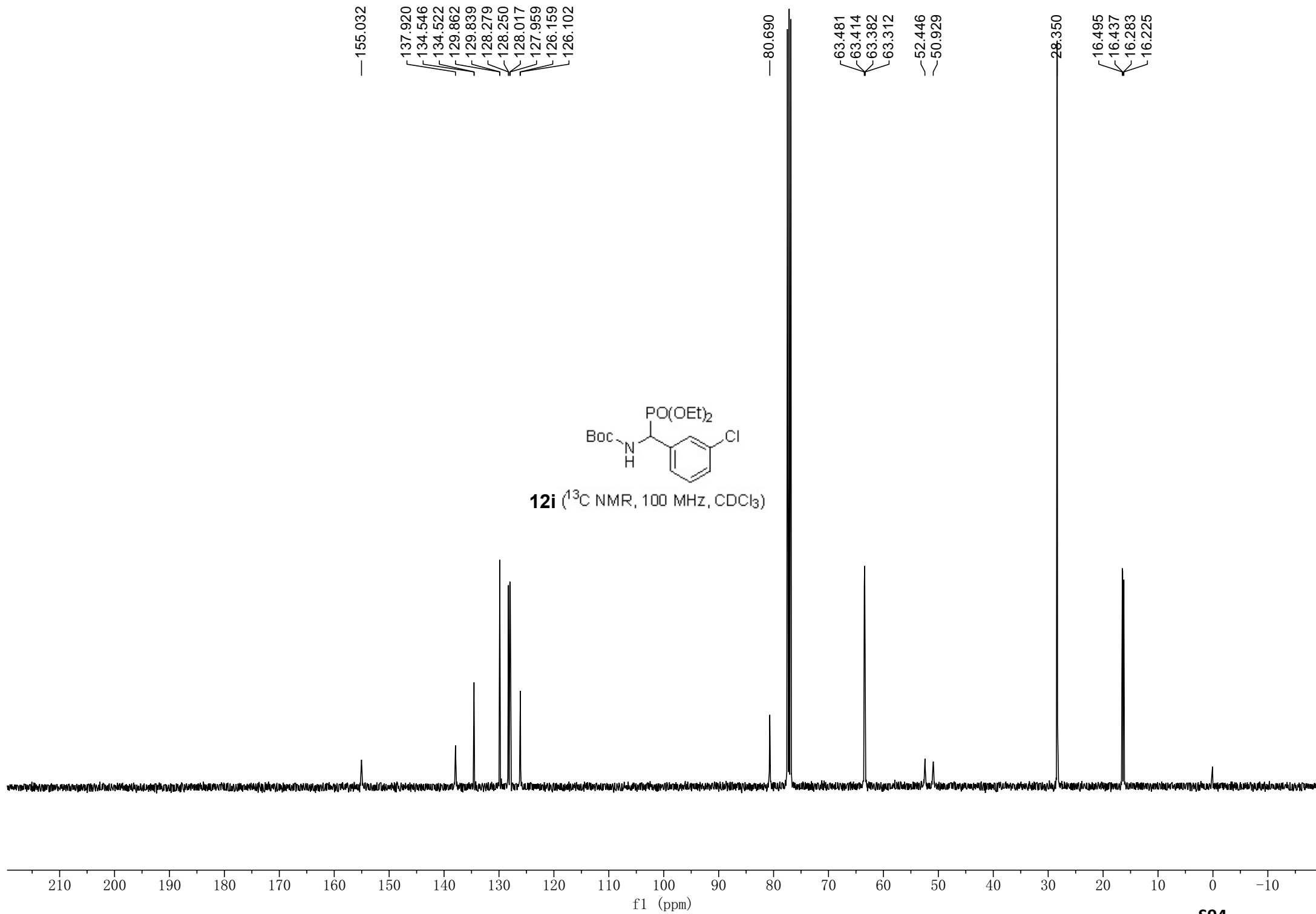
—21.859



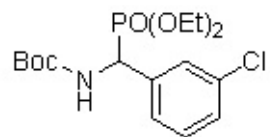
**12h** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



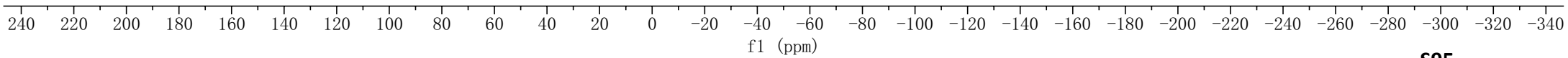




—21.054



**12i** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



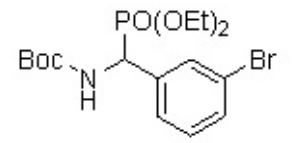
7.550  
7.544  
7.539  
7.440  
7.426  
7.420  
7.366  
7.346  
7.239  
7.219  
7.200

5.550

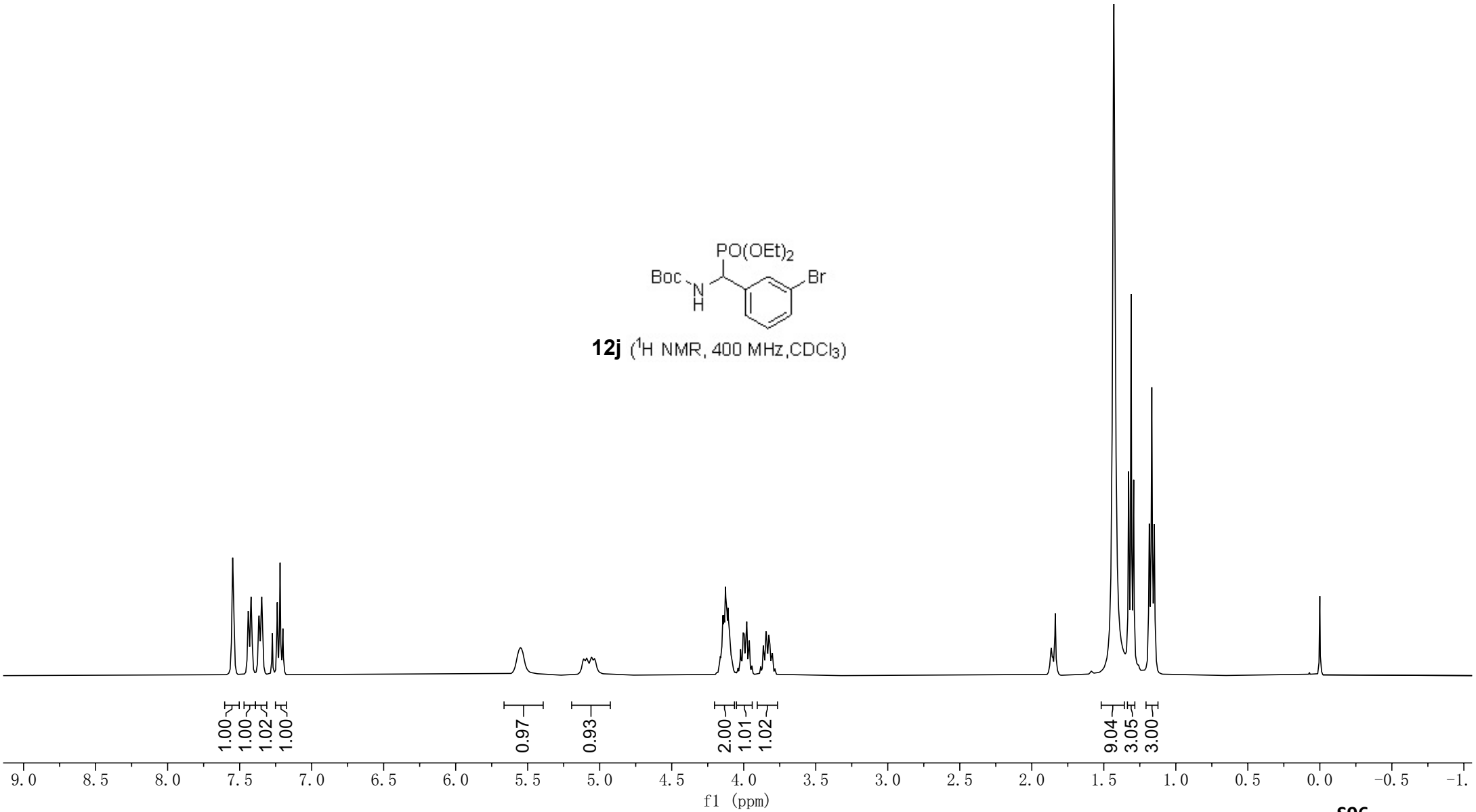
5.113  
5.090  
5.057  
5.033

4.145  
4.138  
4.127  
4.119  
4.109  
4.100  
4.086  
4.040  
4.022  
4.015  
4.004  
3.997  
3.987  
3.979  
3.968  
3.961  
3.944  
3.882  
3.864  
3.846  
3.839  
3.826  
3.820  
3.808  
3.801  
3.783

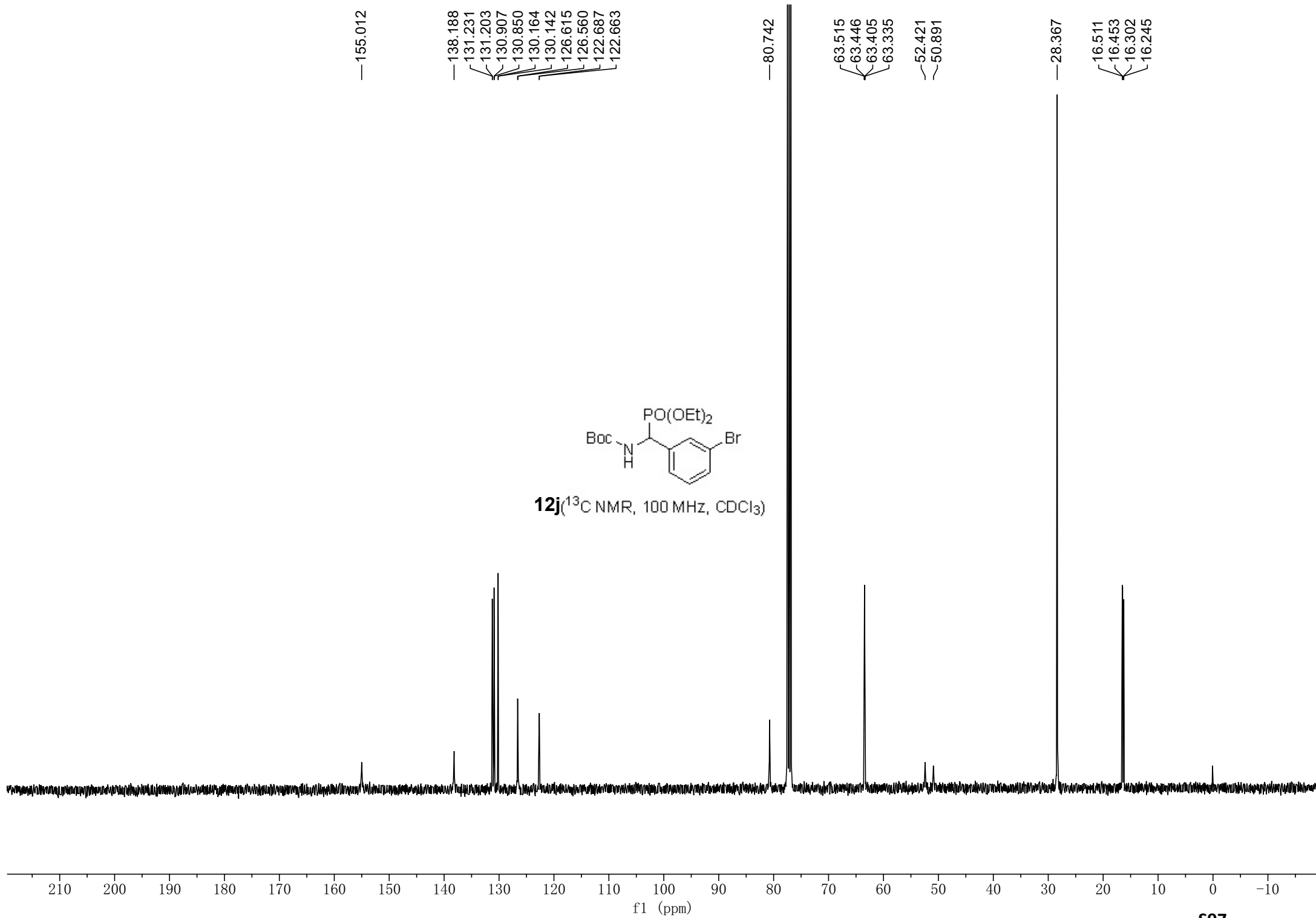
1.430  
1.328  
1.310  
1.293  
1.184  
1.167  
1.149



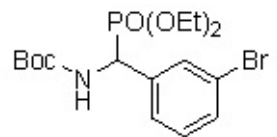
**12j** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)



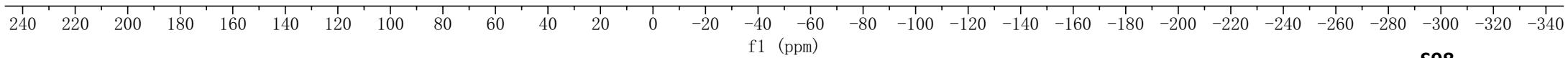


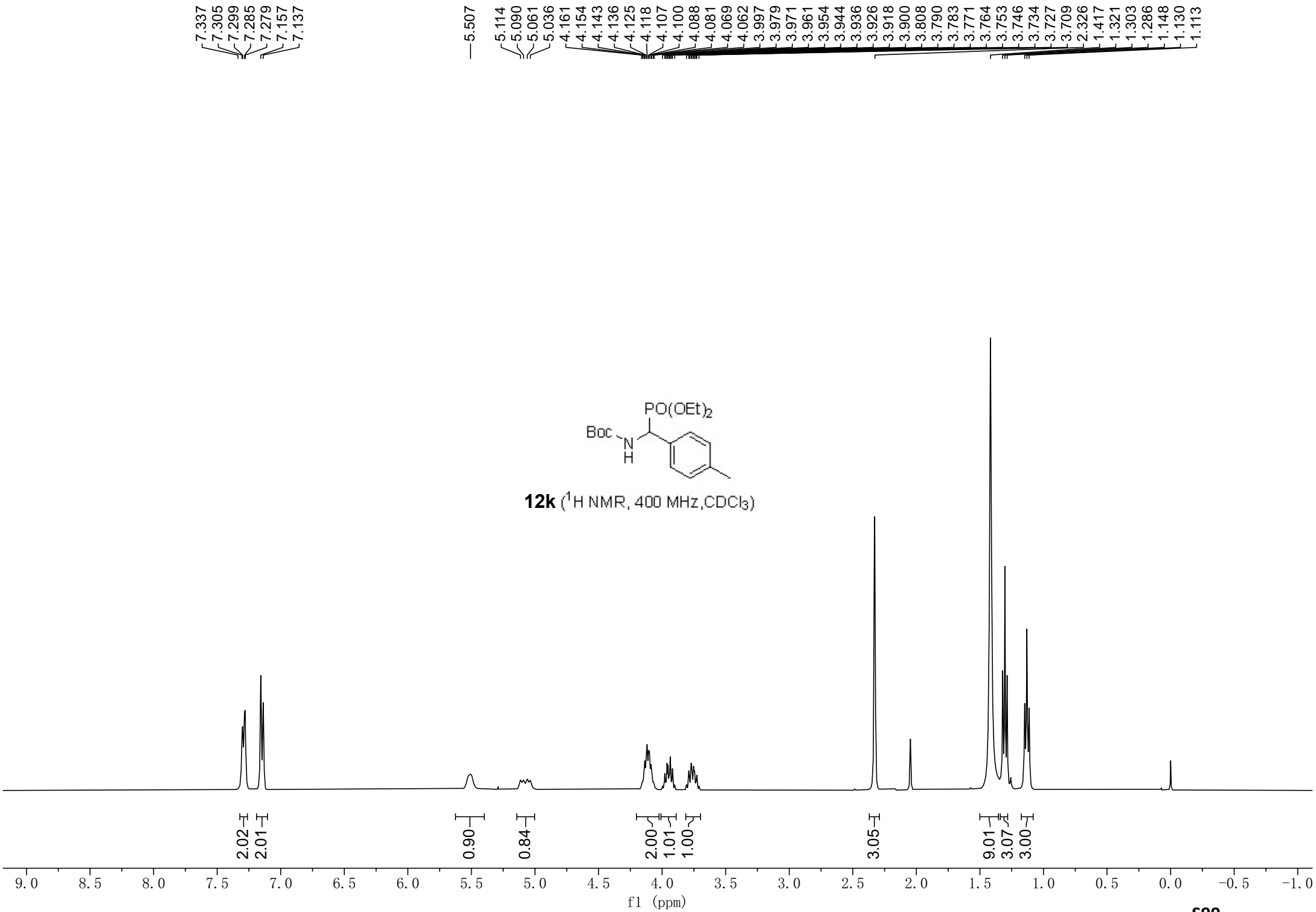


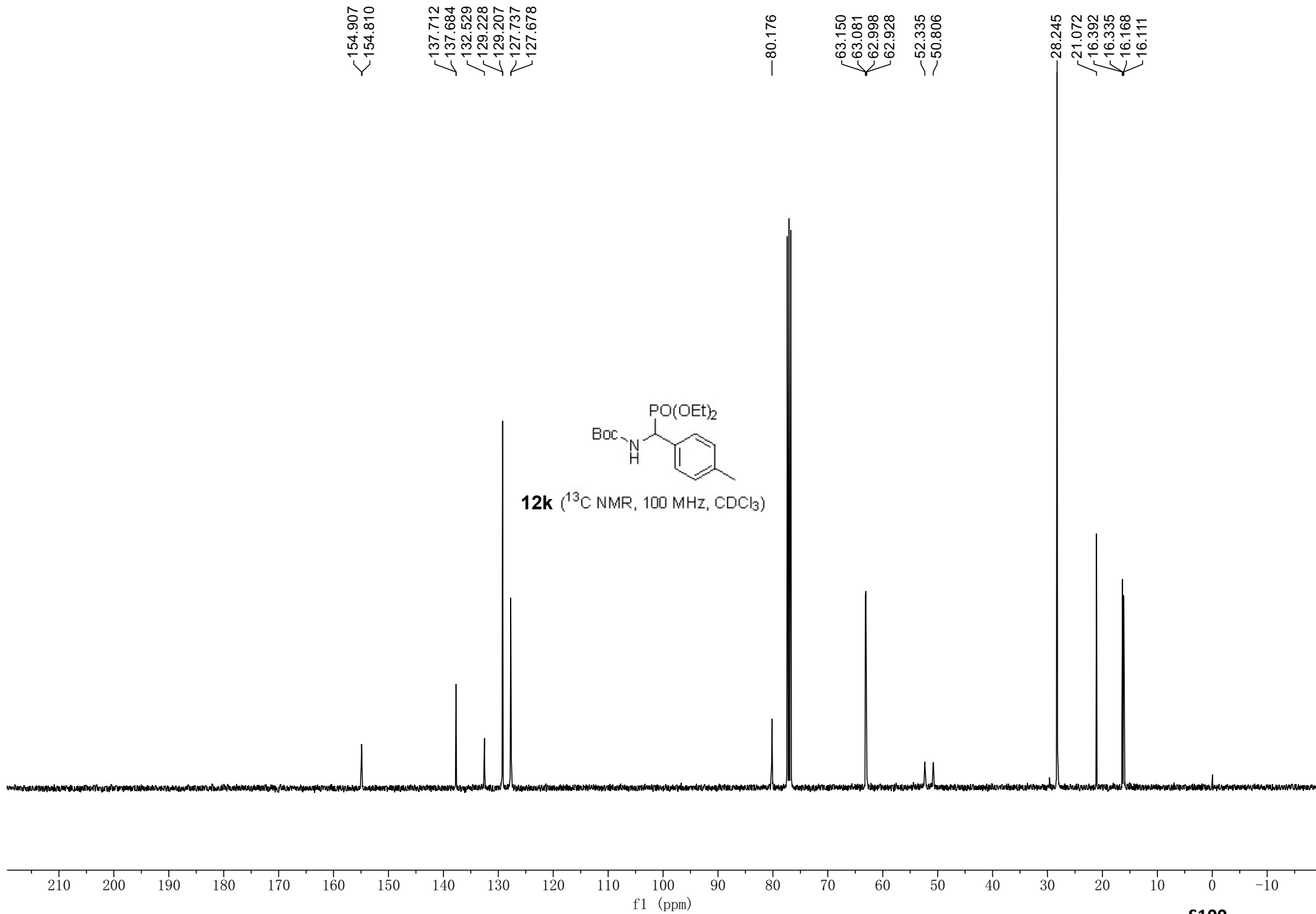
—21.046



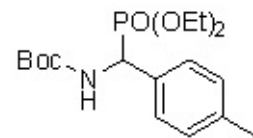
**12j** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )



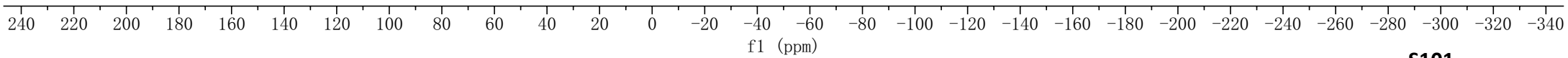




22.018



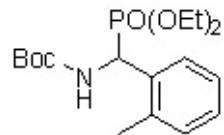
**12k** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )



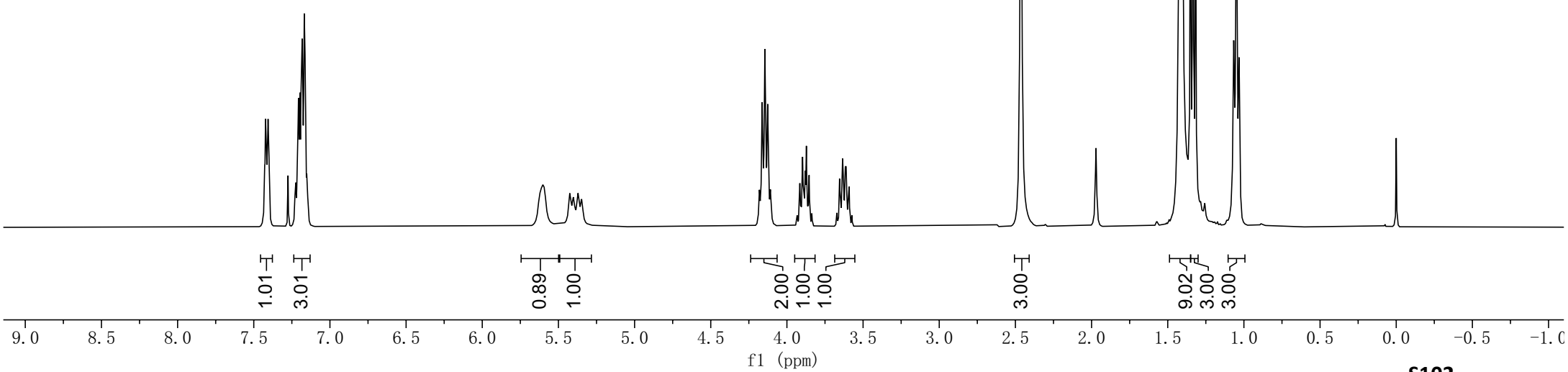
7.429  
7.423  
7.417  
7.407  
7.400  
7.224  
7.214  
7.206  
7.197  
7.189  
7.181  
7.169  
7.163  
7.152  
5.602  
5.426  
5.401  
5.371  
5.347  
4.181  
4.163  
4.145  
4.126  
4.108  
3.915  
3.908  
3.897  
3.890  
3.880  
3.872  
3.862  
3.854  
3.672  
3.654  
3.647  
3.636  
3.629  
3.617  
3.610  
3.599  
3.592  
3.574

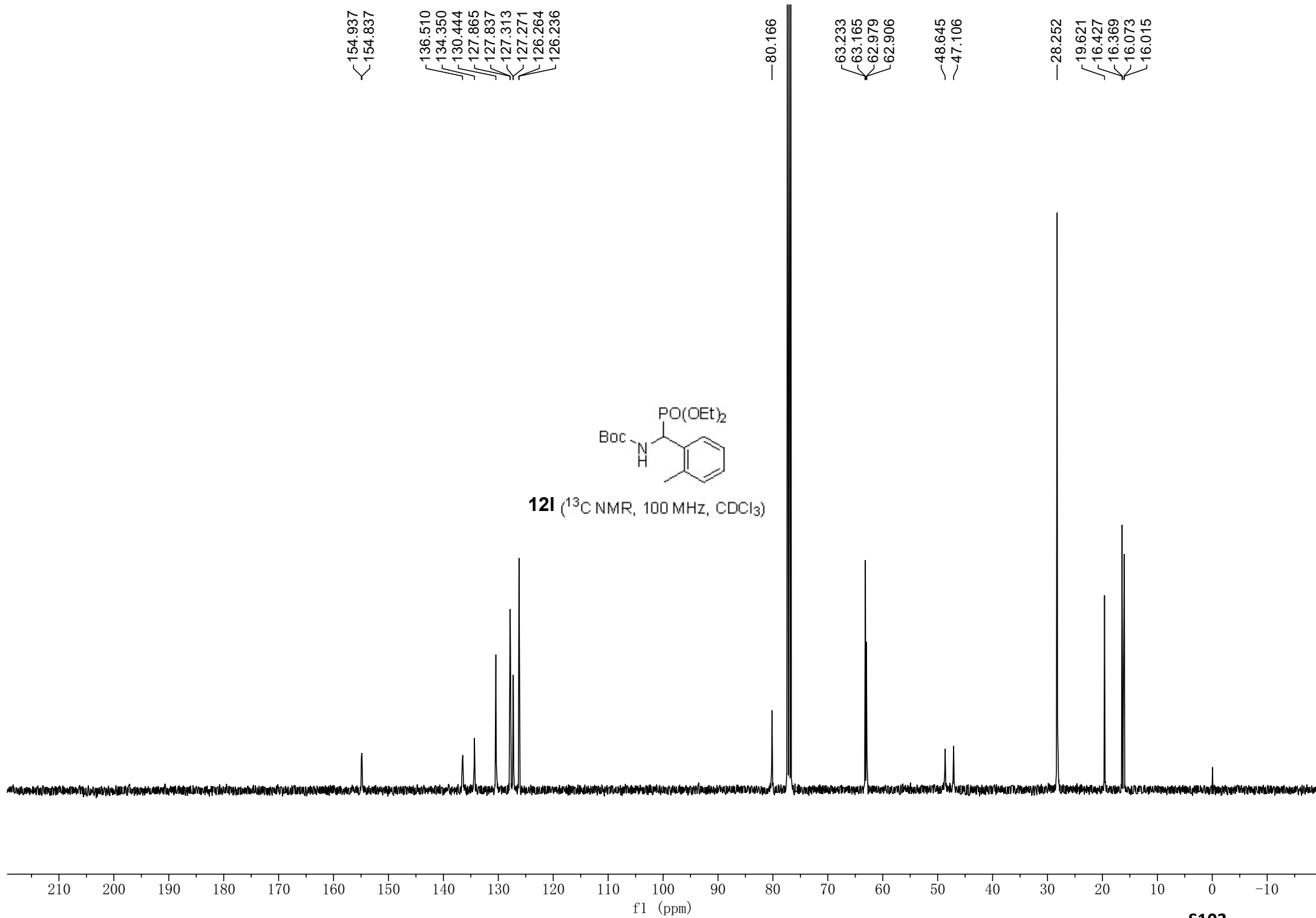
—2.464

1.415  
1.352  
1.334  
1.317  
1.067  
1.049  
1.032

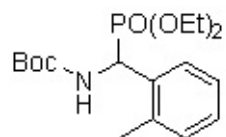


**121** (<sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>)

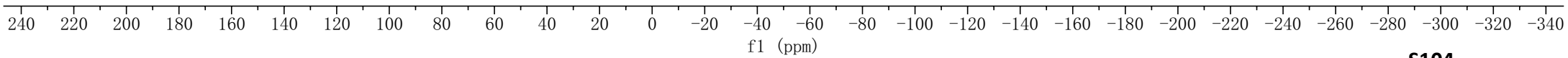




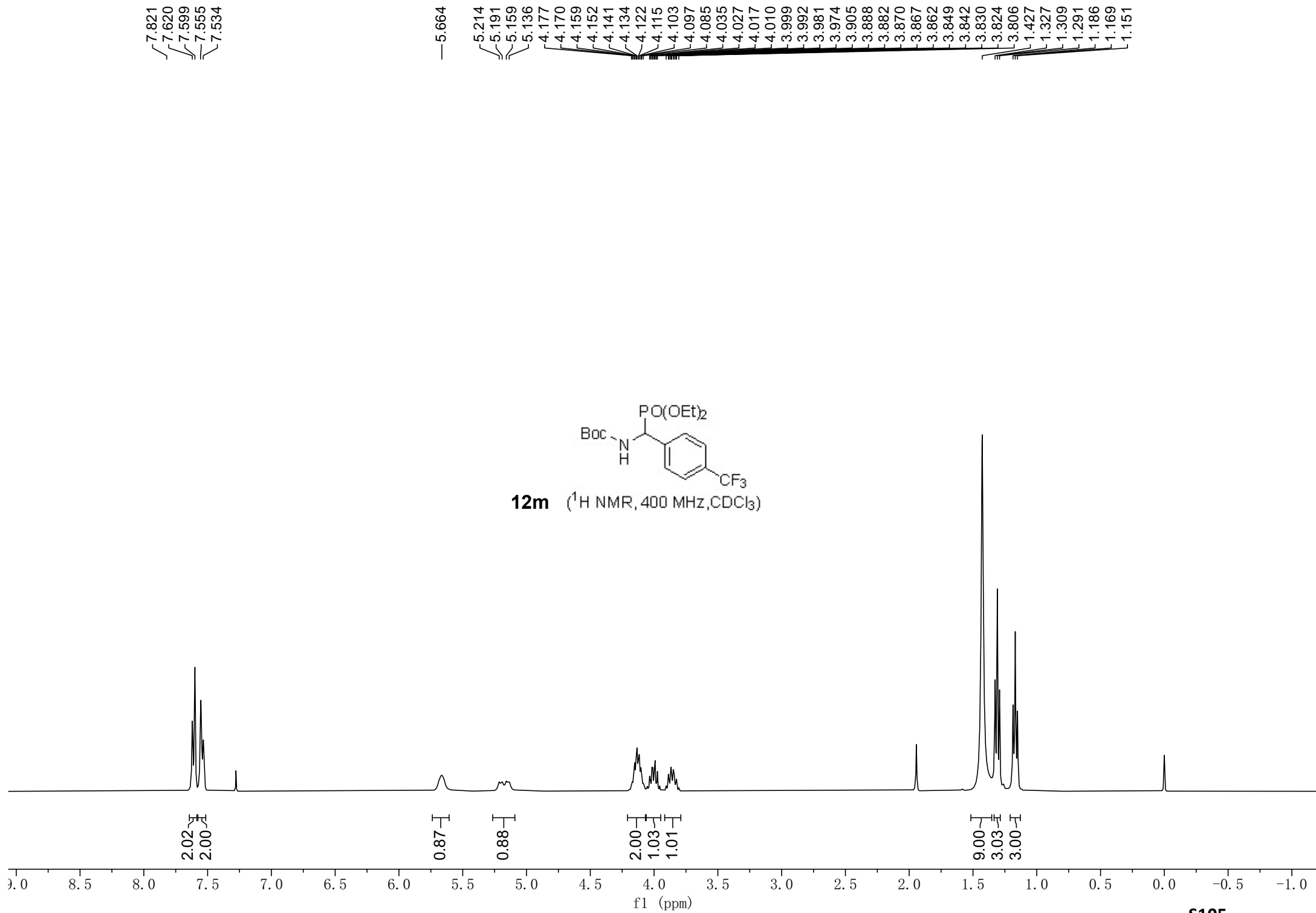
—22.641

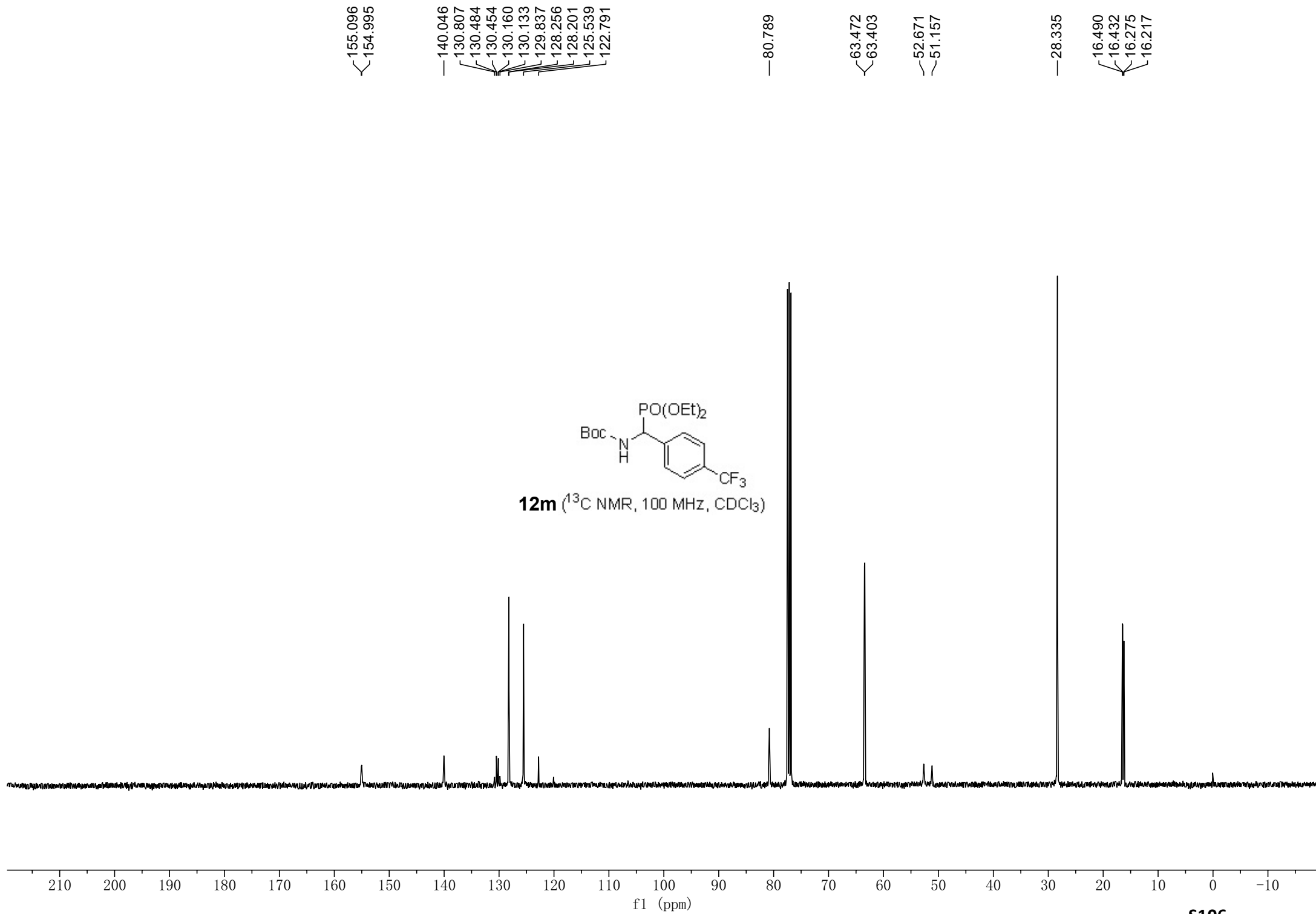


**12I** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

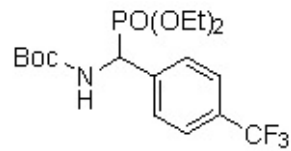




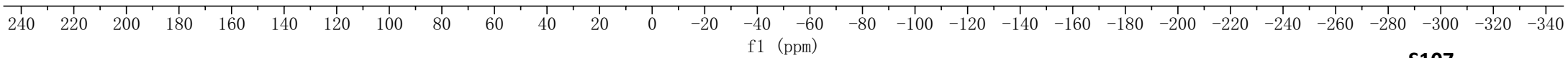




20.825

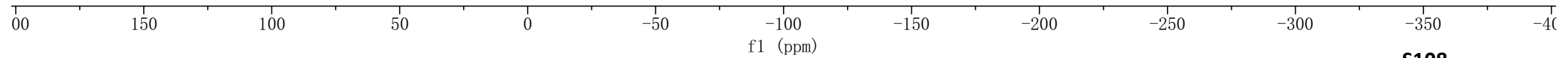
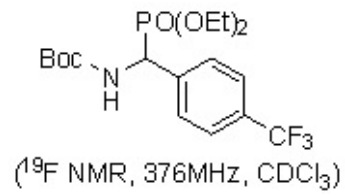


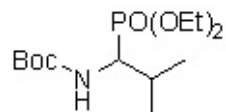
**12m** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)



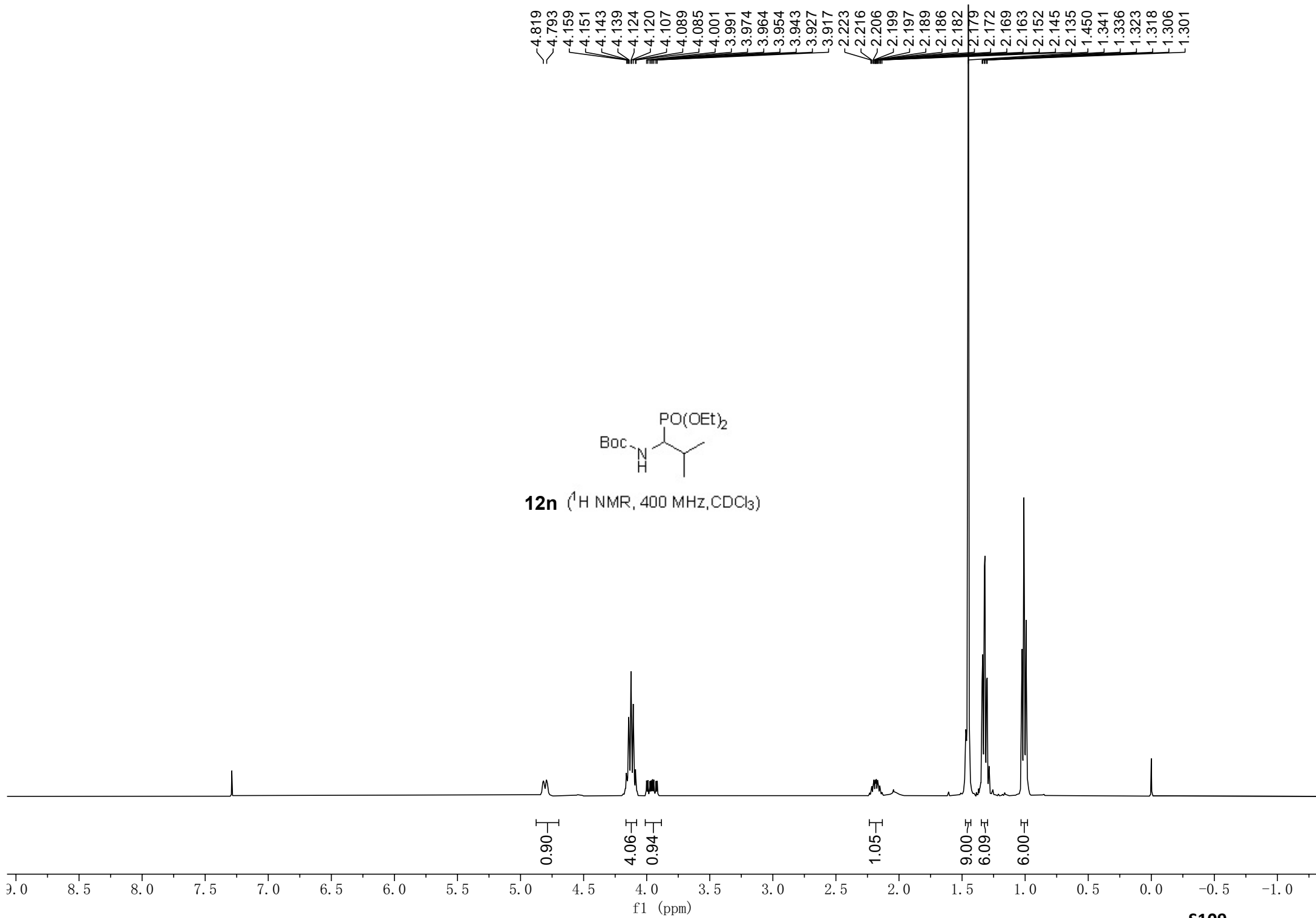
-62.655

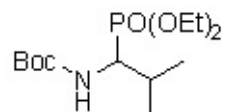
12m



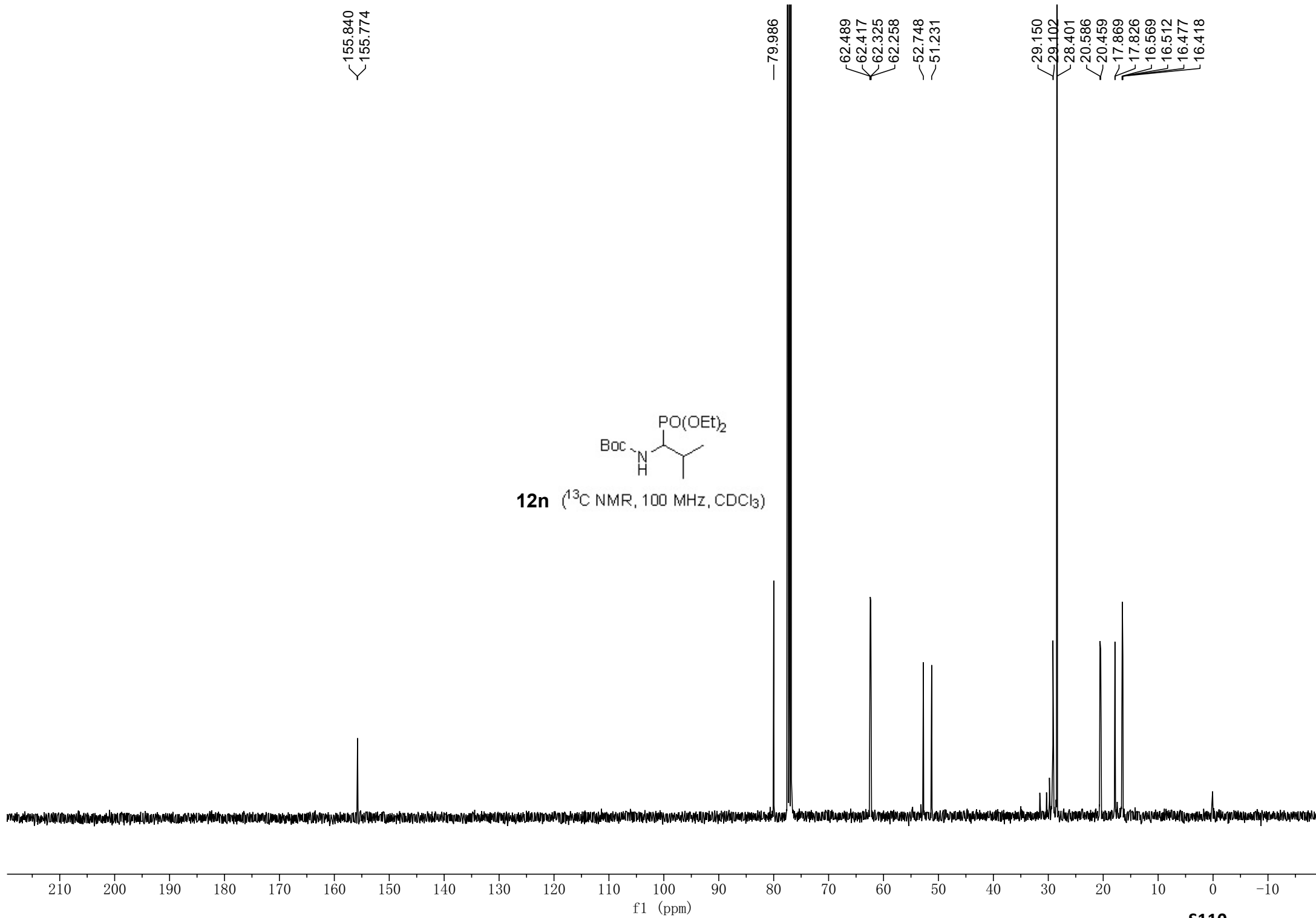


**12n** ( $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$ )

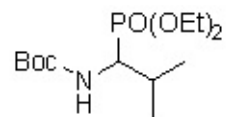




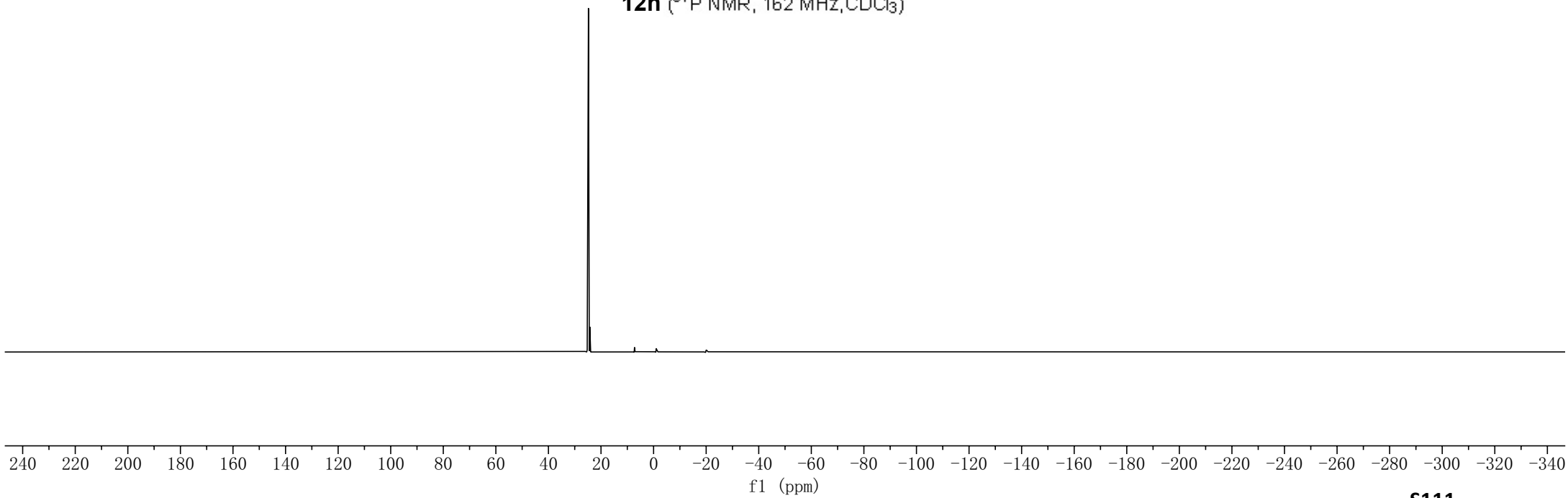
**12n** ( $^{13}\text{C}$  NMR, 100 MHz,  $\text{CDCl}_3$ )

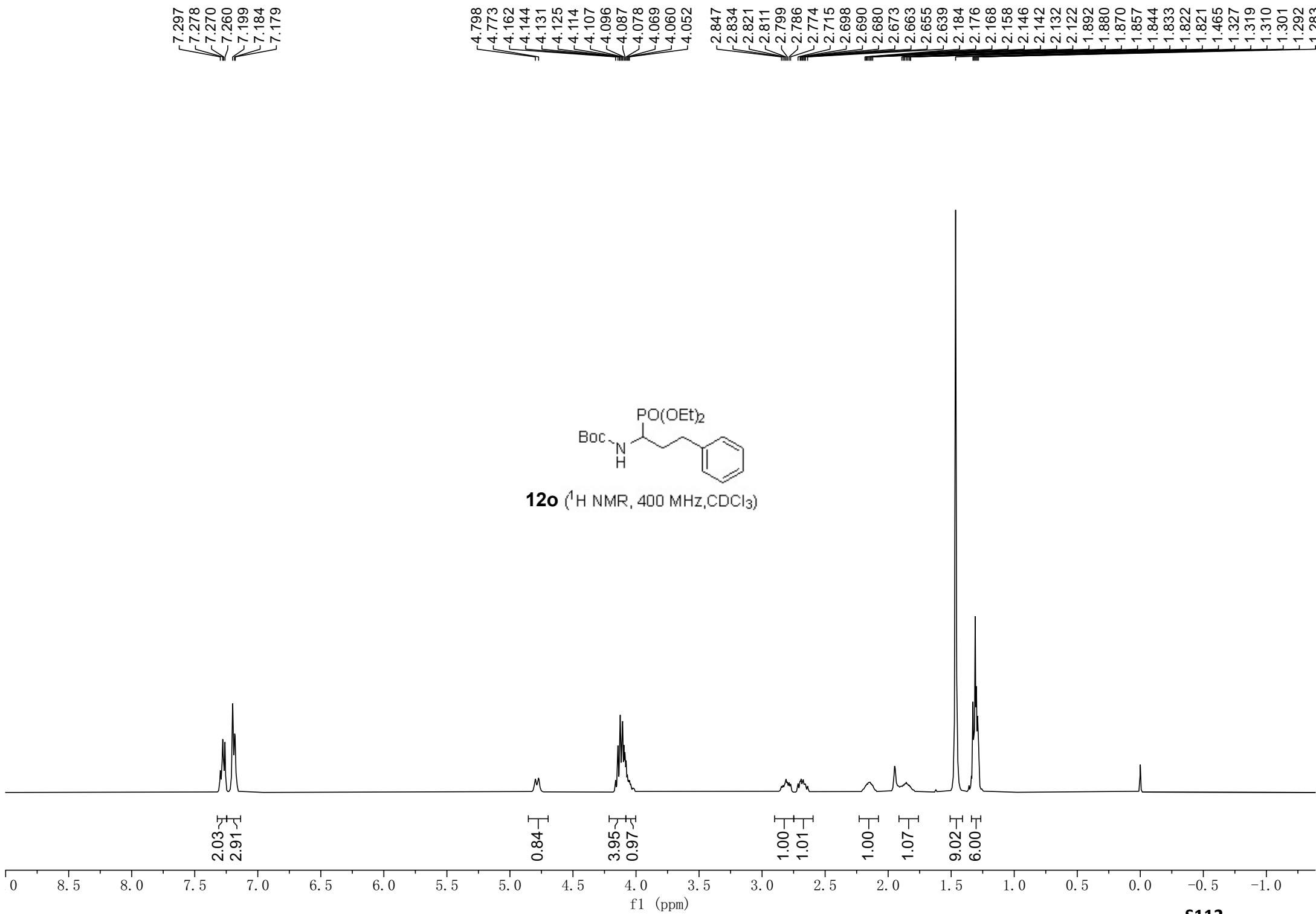


—24.781

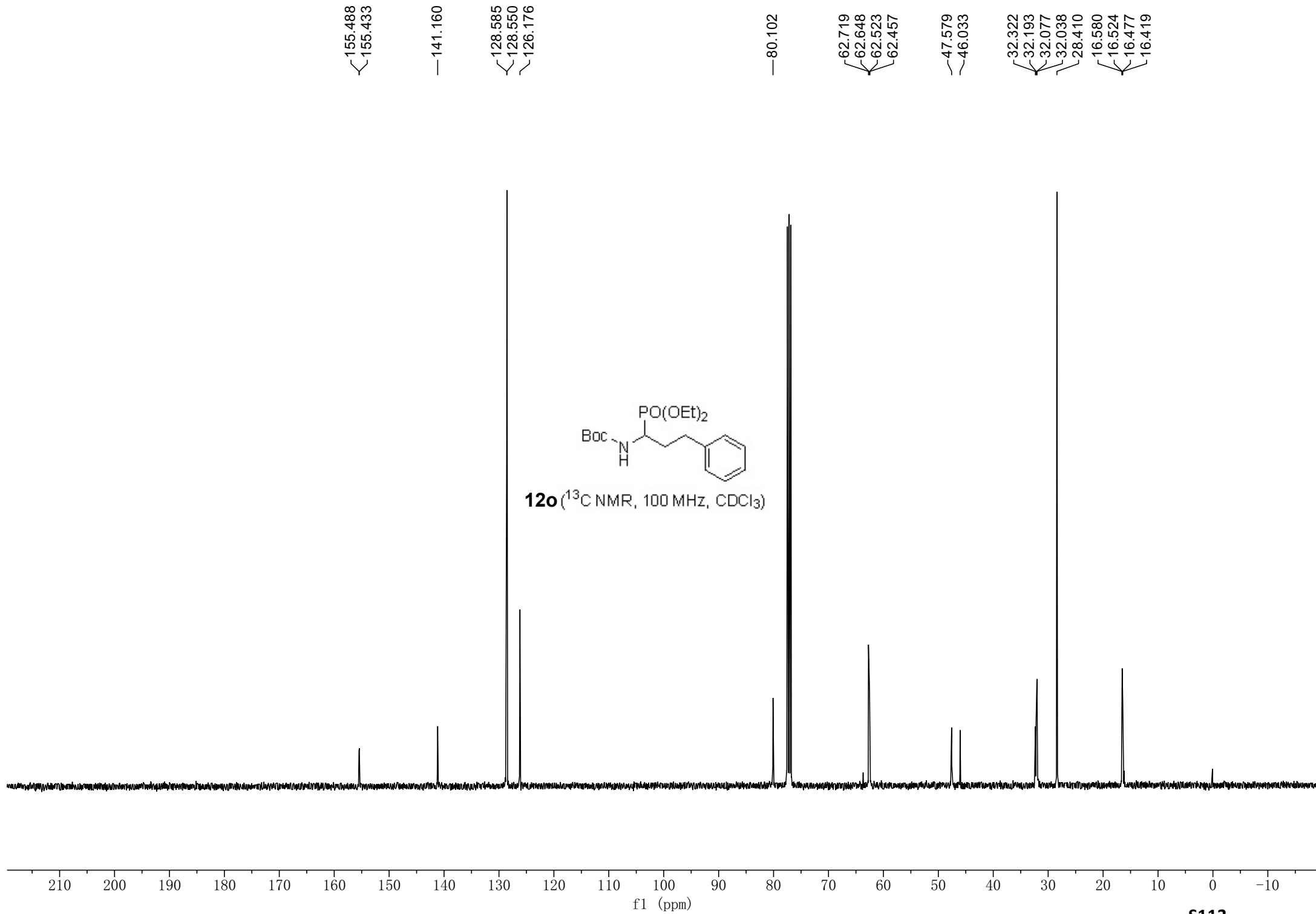


**12n** (<sup>31</sup>P NMR, 162 MHz, CDCl<sub>3</sub>)

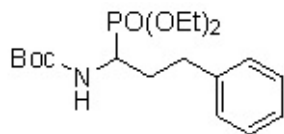




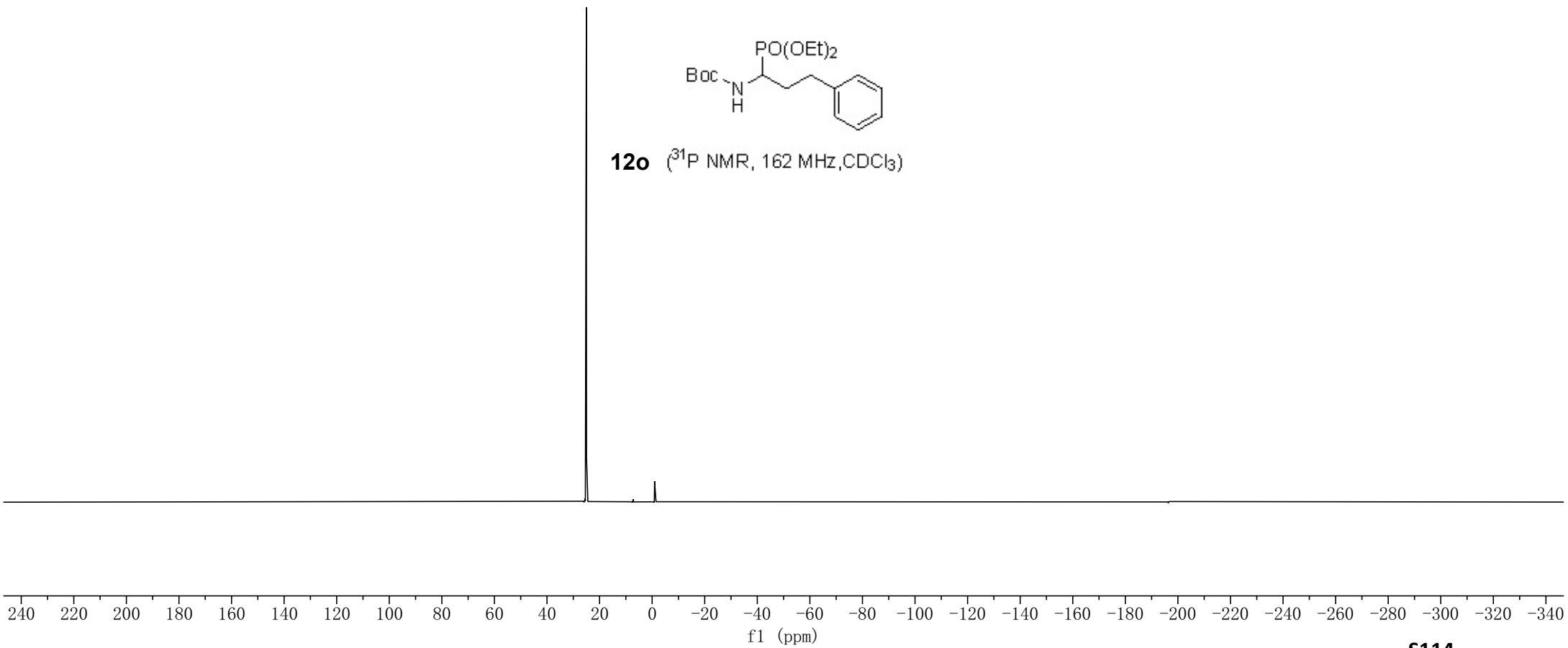


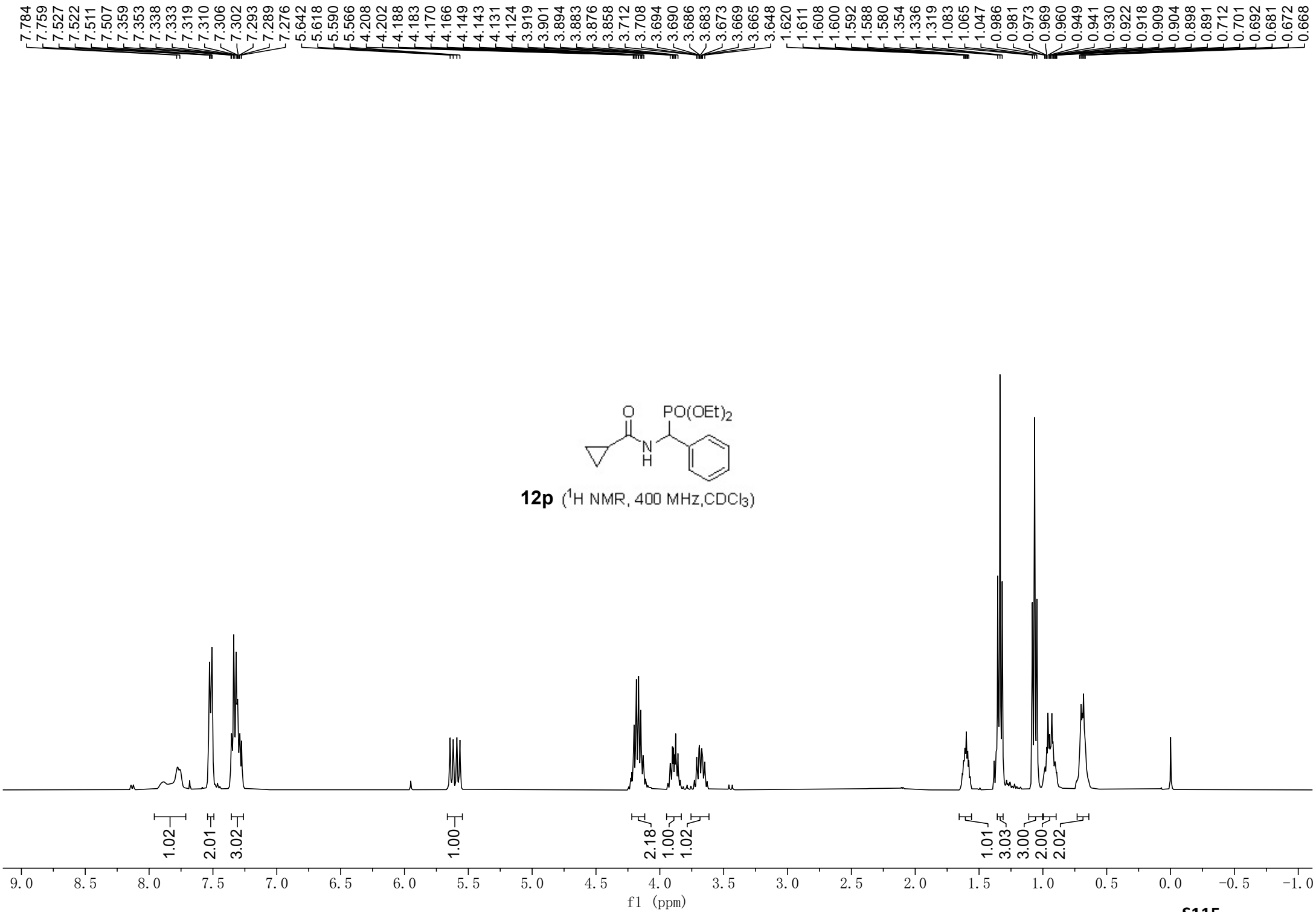


—25.020



**12o** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )





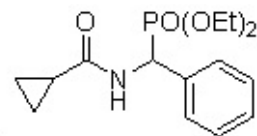
173.323  
173.247

135.407  
128.517  
128.497  
128.386  
128.324  
127.969  
127.942

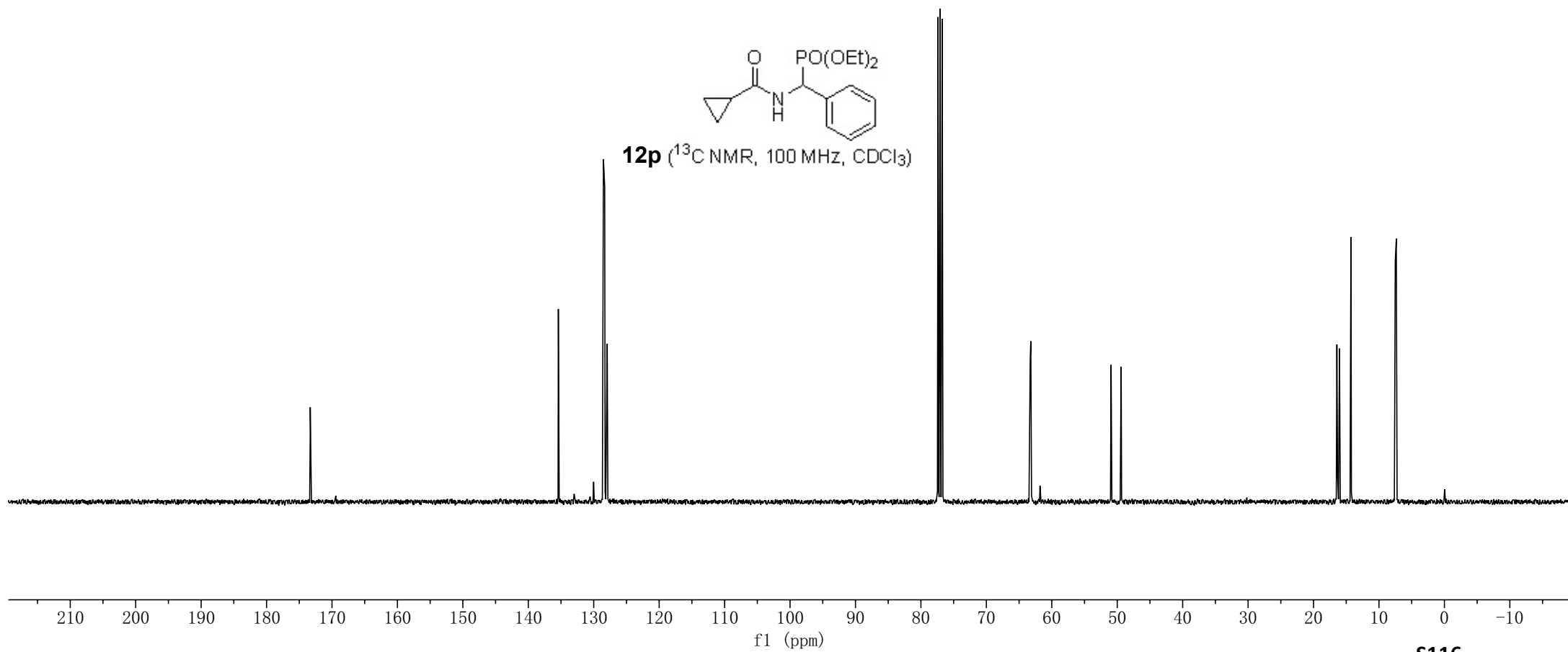
63.303  
63.231  
63.209  
63.138

50.937  
49.402

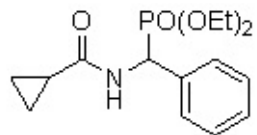
16.432  
16.375  
16.089  
16.033  
14.280  
7.509  
7.353



**12p** ( $^{13}\text{C}$  NMR, 100 MHz,  $\text{CDCl}_3$ )



—21.857



**12p** ( $^{31}\text{P}$  NMR, 162 MHz,  $\text{CDCl}_3$ )

