

# Supplementary Materials

## Visible Light-Mediated Organoboron-Catalyzed Metal-Free Synthesis of Silanols from Silanes

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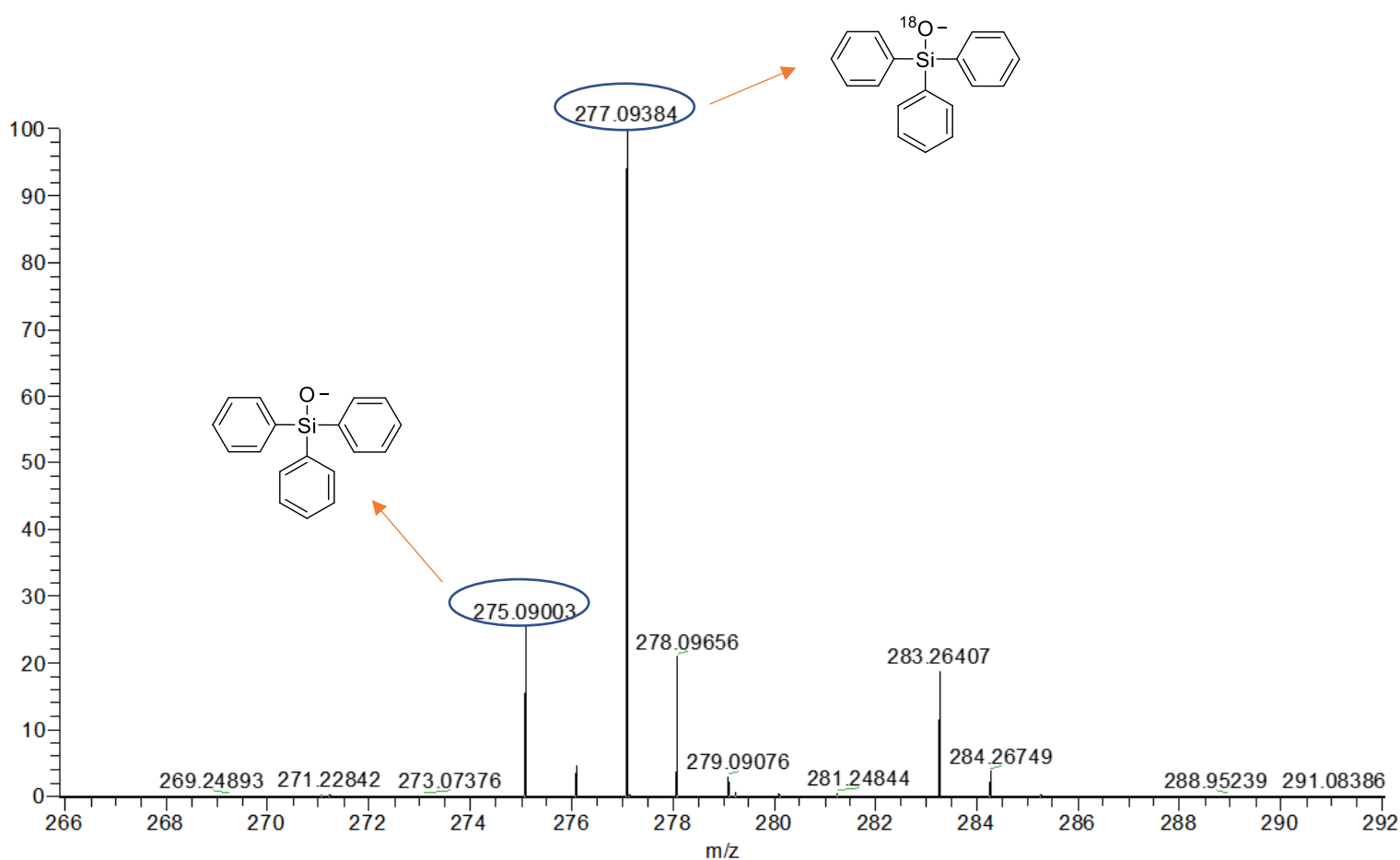
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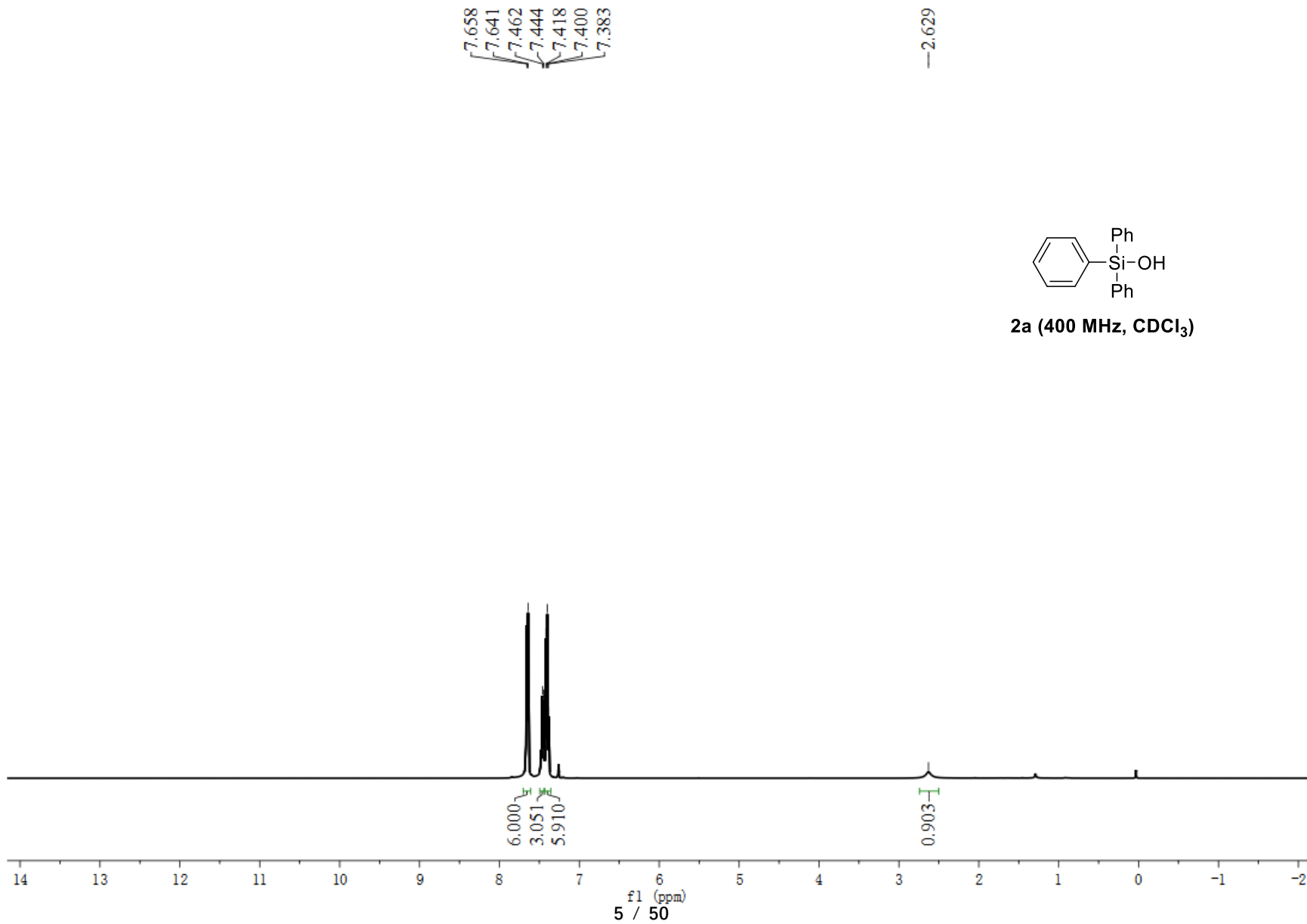
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## 1. $^{18}\text{O}$ Labelling experiments

flame-dried 25 mL quartz reaction tube was placed with a magnetic stir bar. Then, silane 1 (0.2 mmol, 1.0 equiv) were added to the flame-dried 25 mL quartz reaction tube, A triple oxygen replacement process was then performed using a double row of tubes.. After that, A mixture of AQDAB (0.9 mg, 0.002 mmol, 1.0 mol%), Dry DMSO (1 ml) and  $\text{H}_2^{18}\text{O}$  (50  $\mu\text{L}$ ) was rapidly added into the flame-dried 25 mL quartz reaction tube. The reaction tube was placed on a 25w blue Kessil reactor. Then the reaction mixture was stirred at 400-500 RPM and exposed to a blue case lamp at room temperature for 36 hours. After taking out the reaction tube, transfer the reaction mixture to the separator funnel and add 10 ml water to the separator funnel. Then, the reaction mixture was extracted with ethyl acetate ( $3 \times 10 \text{ mL}$ ). The combined organic phase was washed with brine ( $2 \times 5.0 \text{ mL}$ ) and then dried over anhydrous  $\text{Na}_2\text{SO}_4$ . After concentration, the silanol crude product was purified by column chromatography (silica gel) to give silanol 2, using petroleum ether/ethyl acetate (20 : 1) as the eluent.

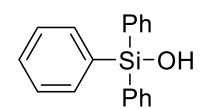


## 2. Copies of NMR spectra

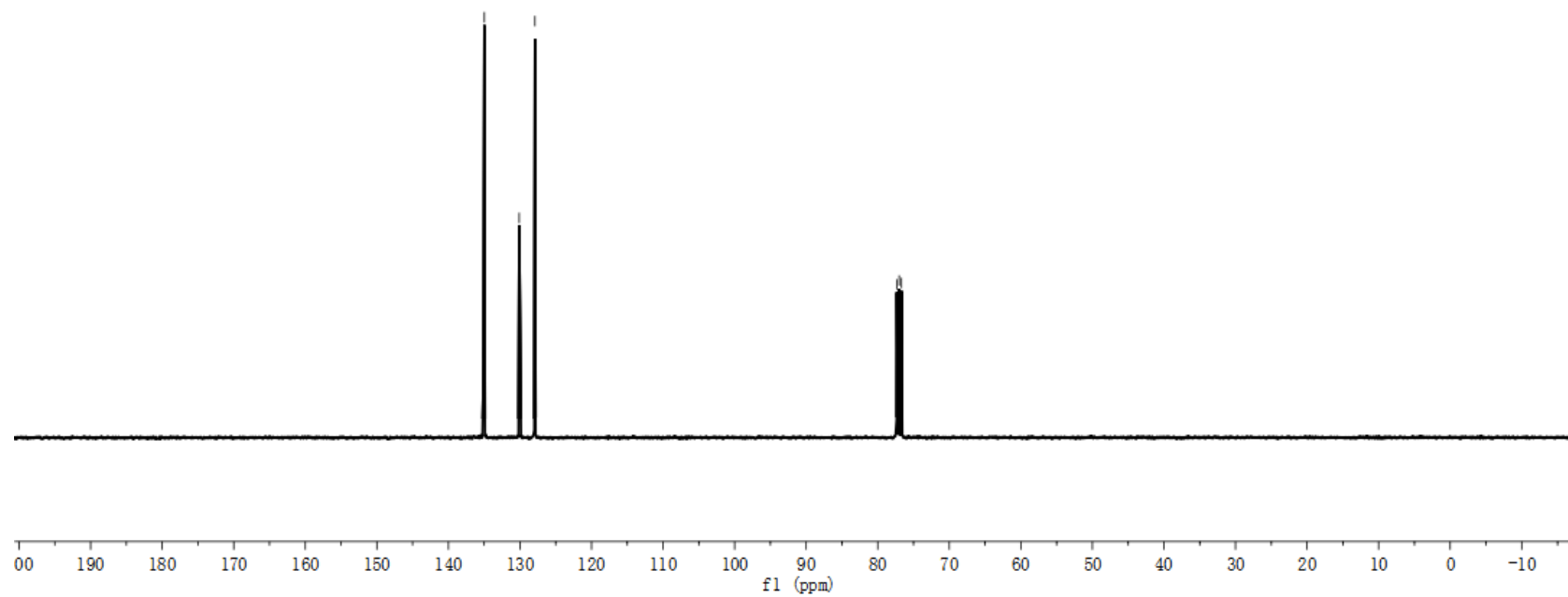


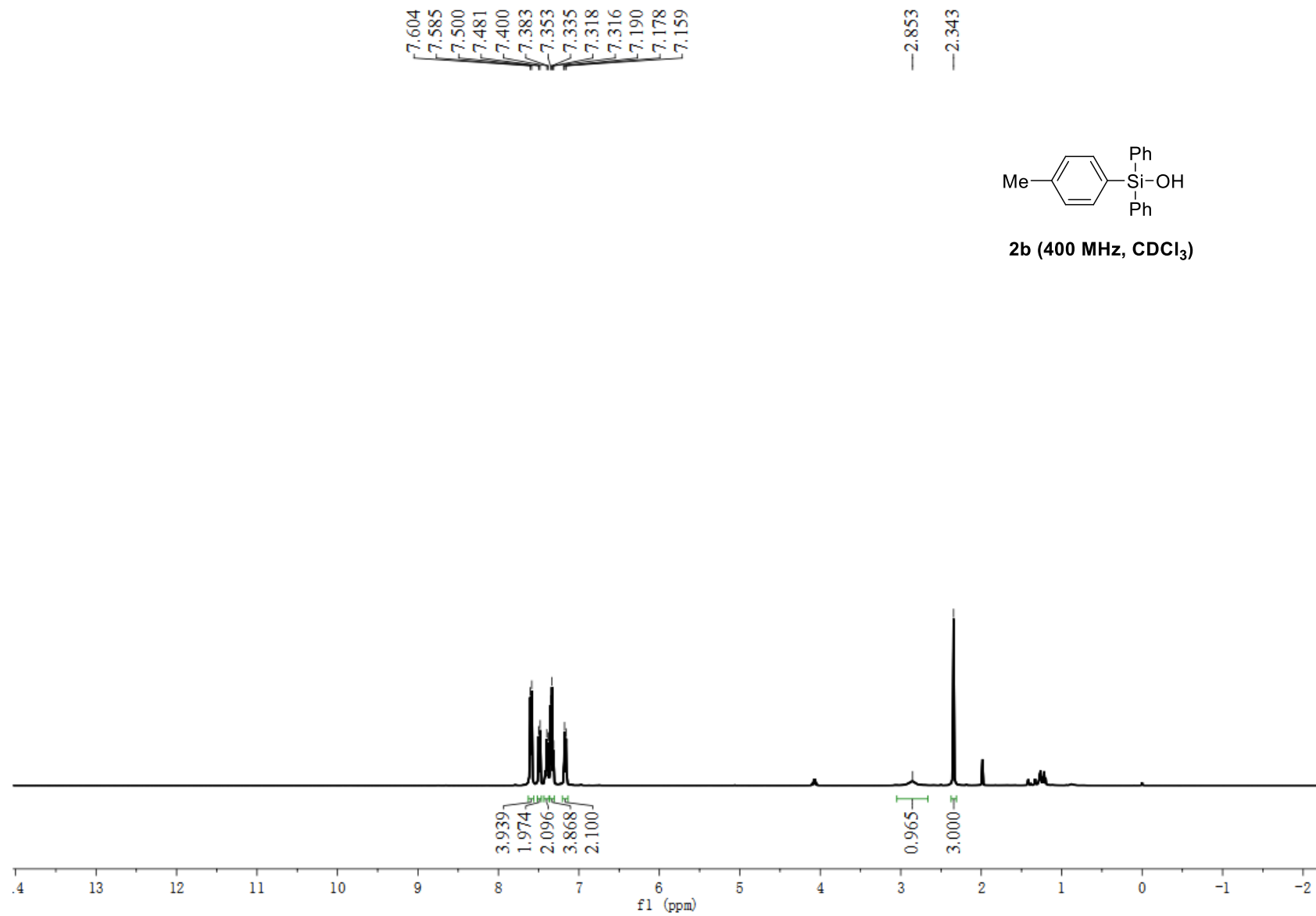
135.10  
134.97  
130.10  
127.91

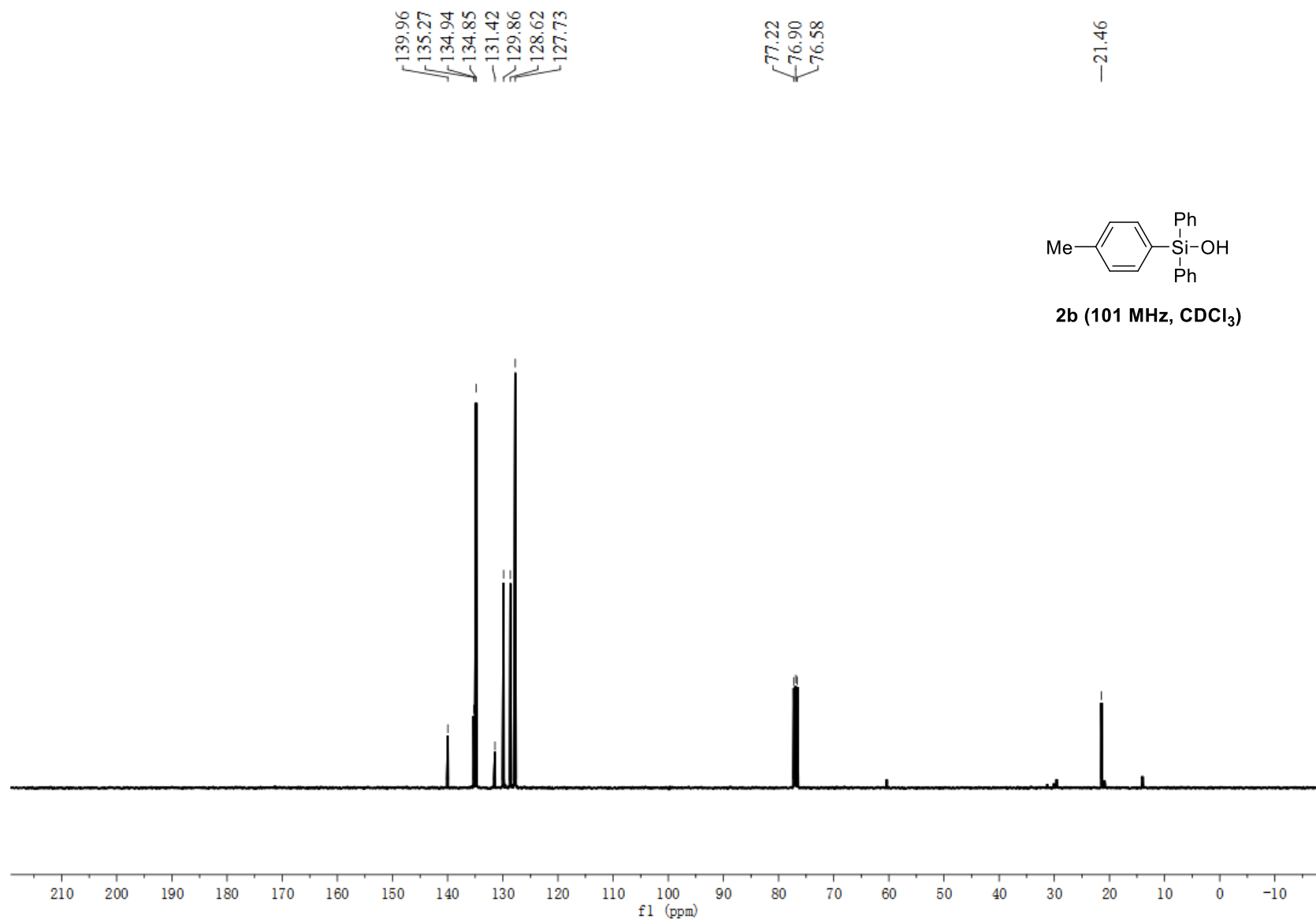
77.32  
77.00  
76.68



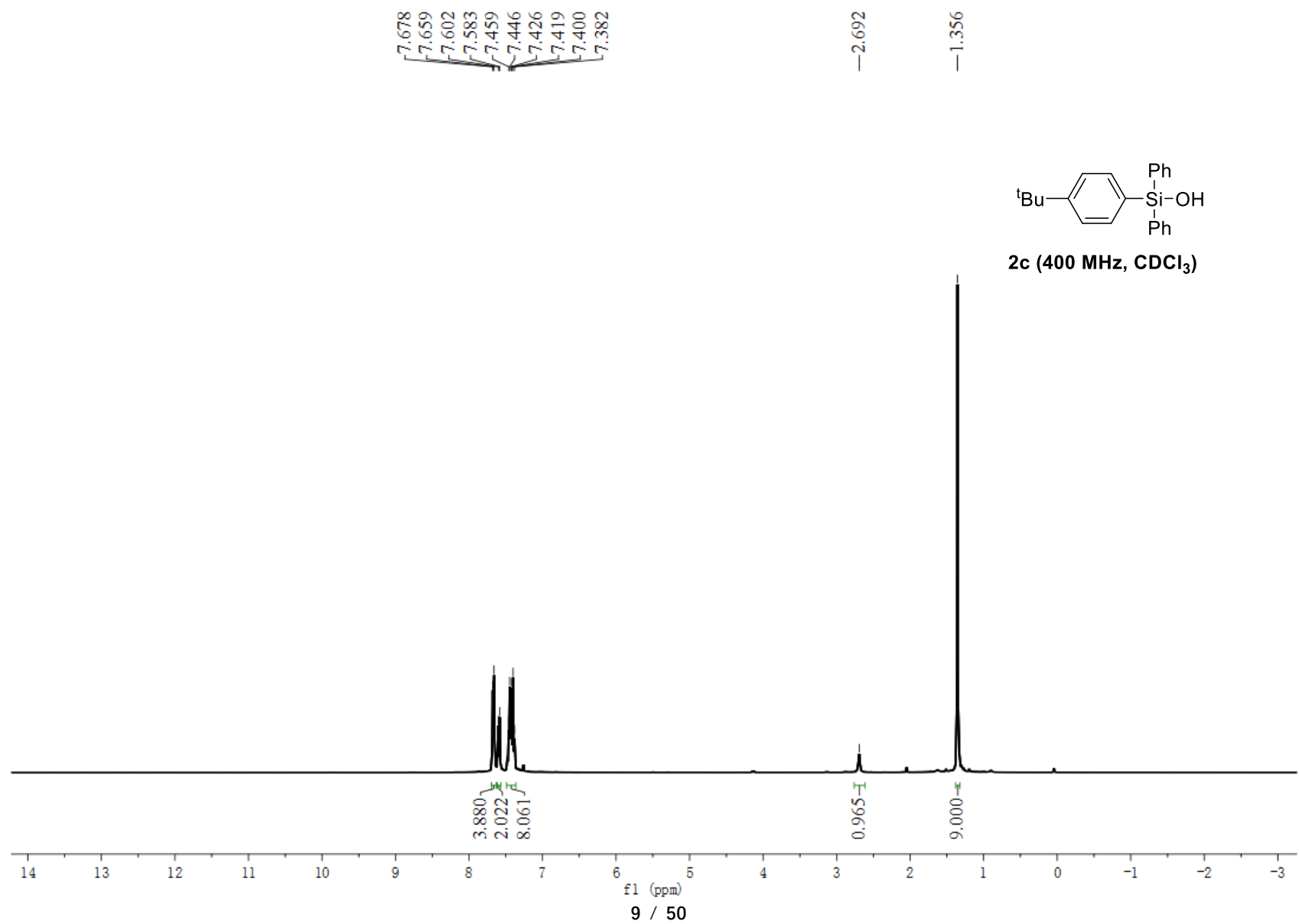
**2a** (101 MHz, CDCl<sub>3</sub>)

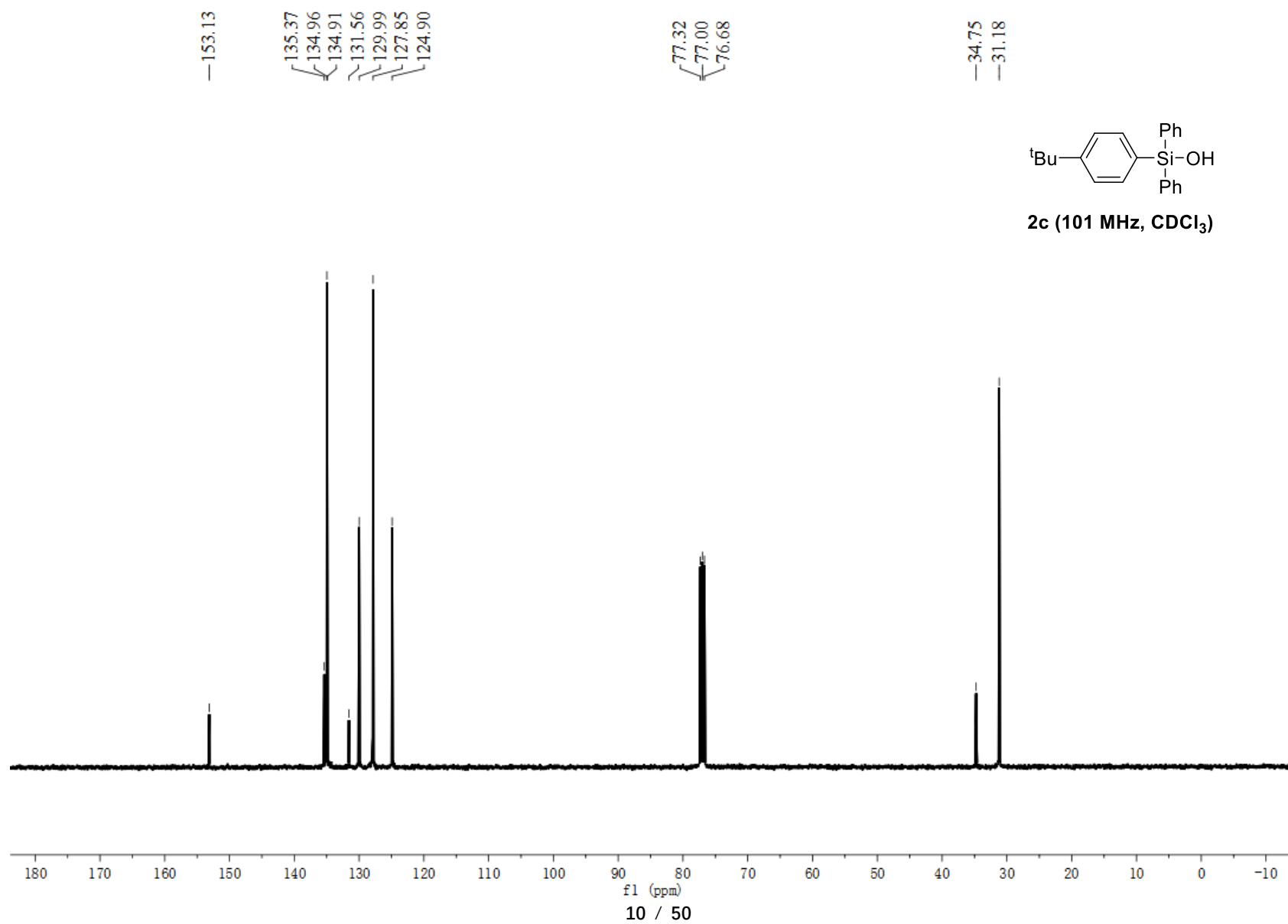


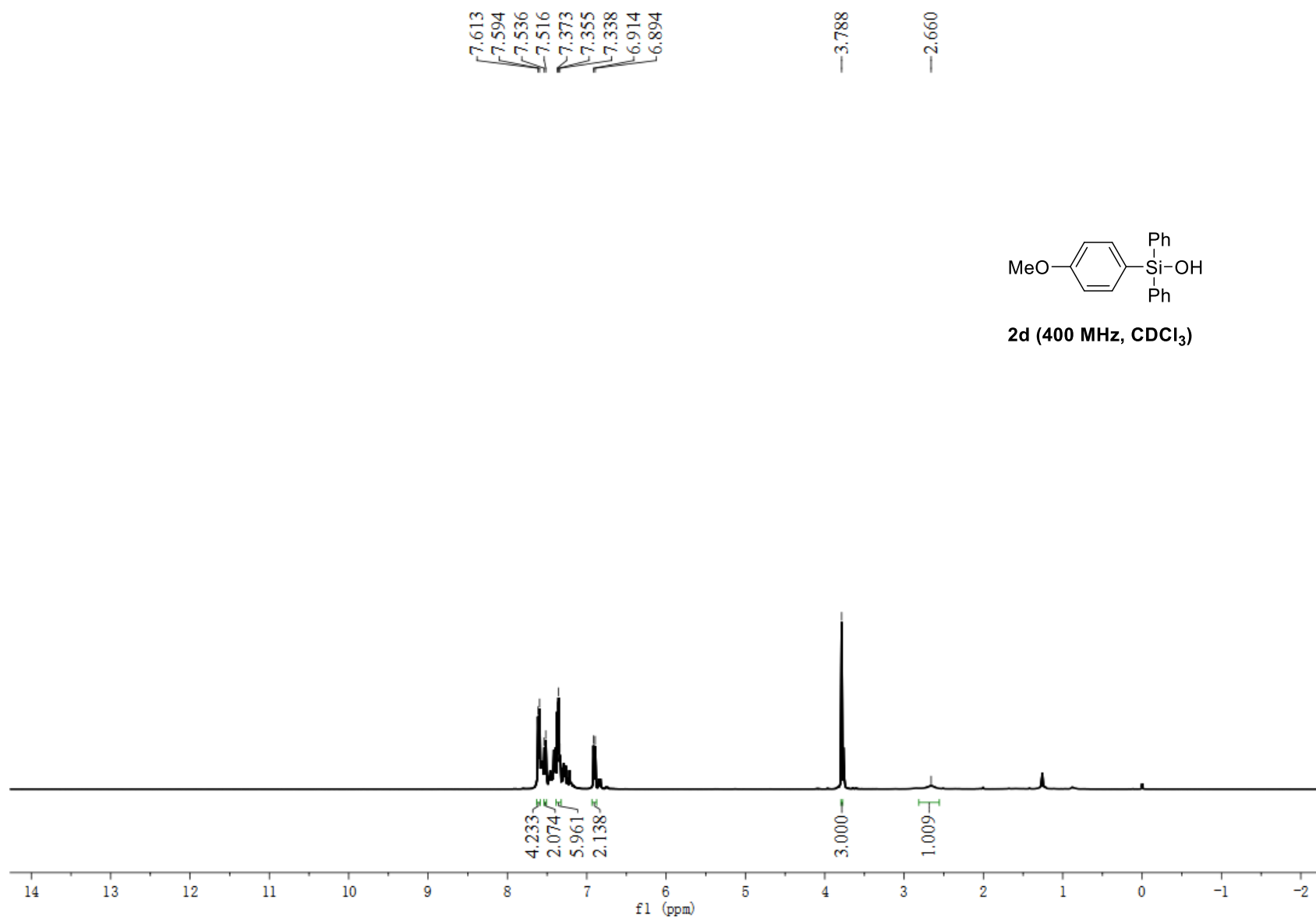


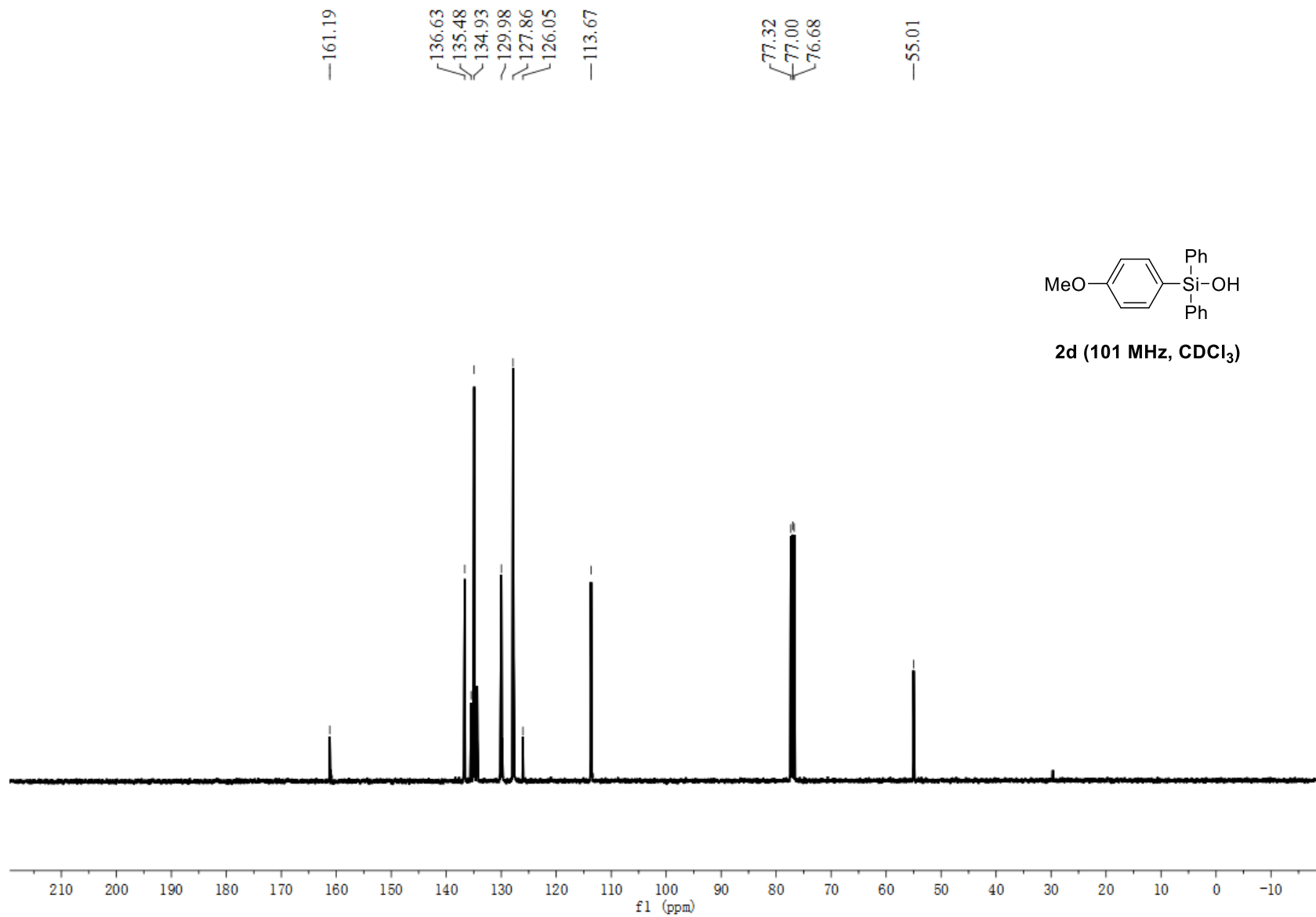


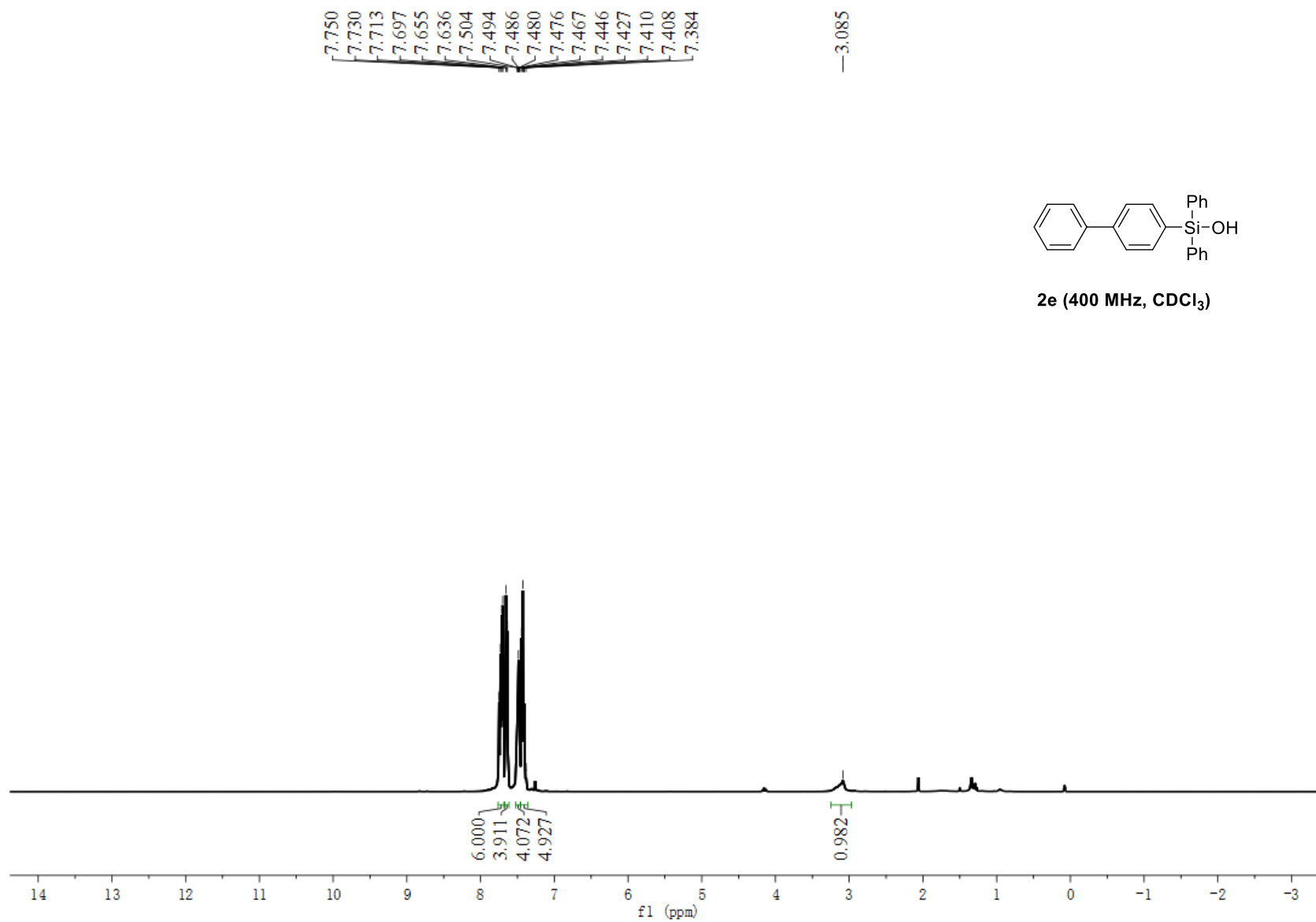


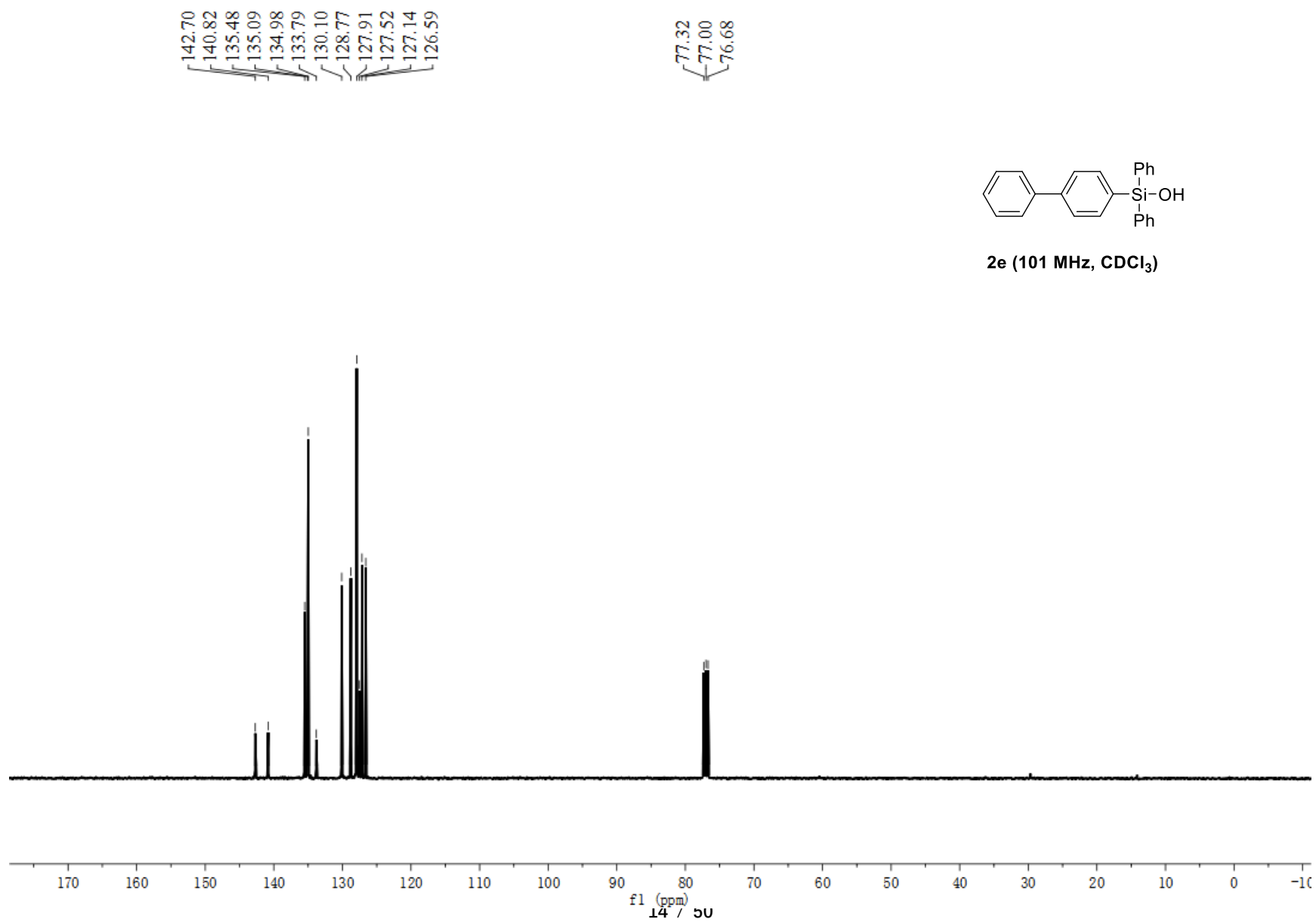


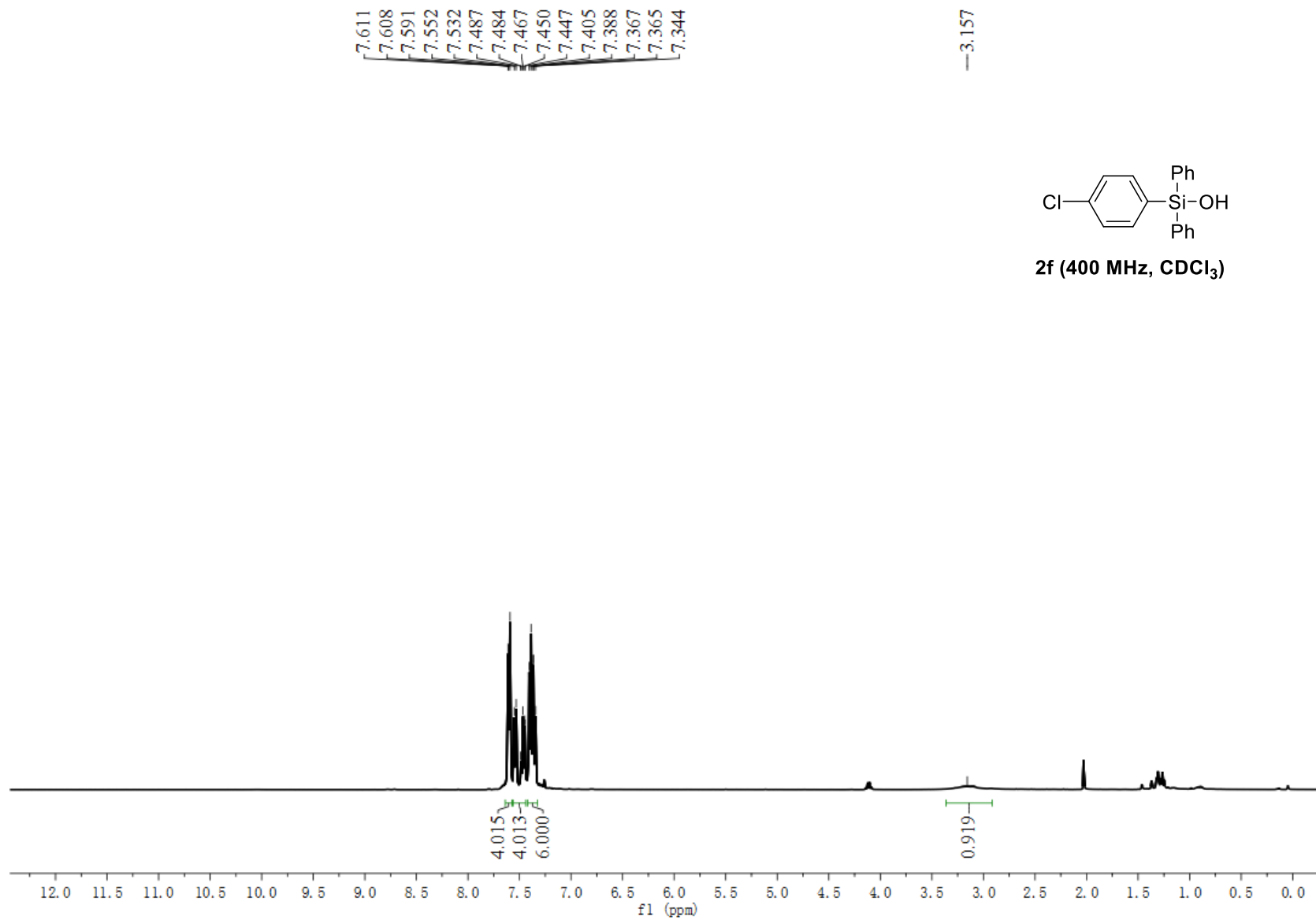






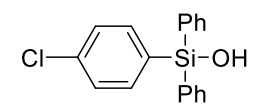




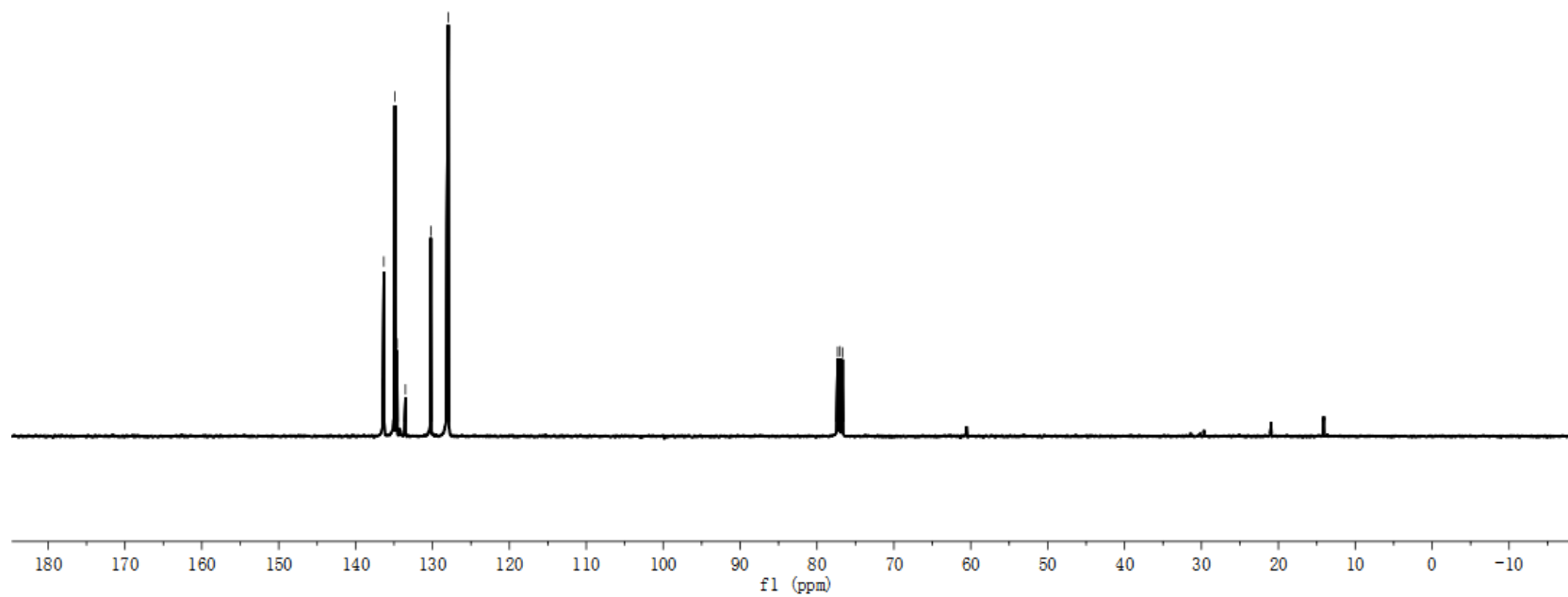


136.47  
136.33  
134.88  
134.61  
133.52  
130.23  
128.14  
127.95

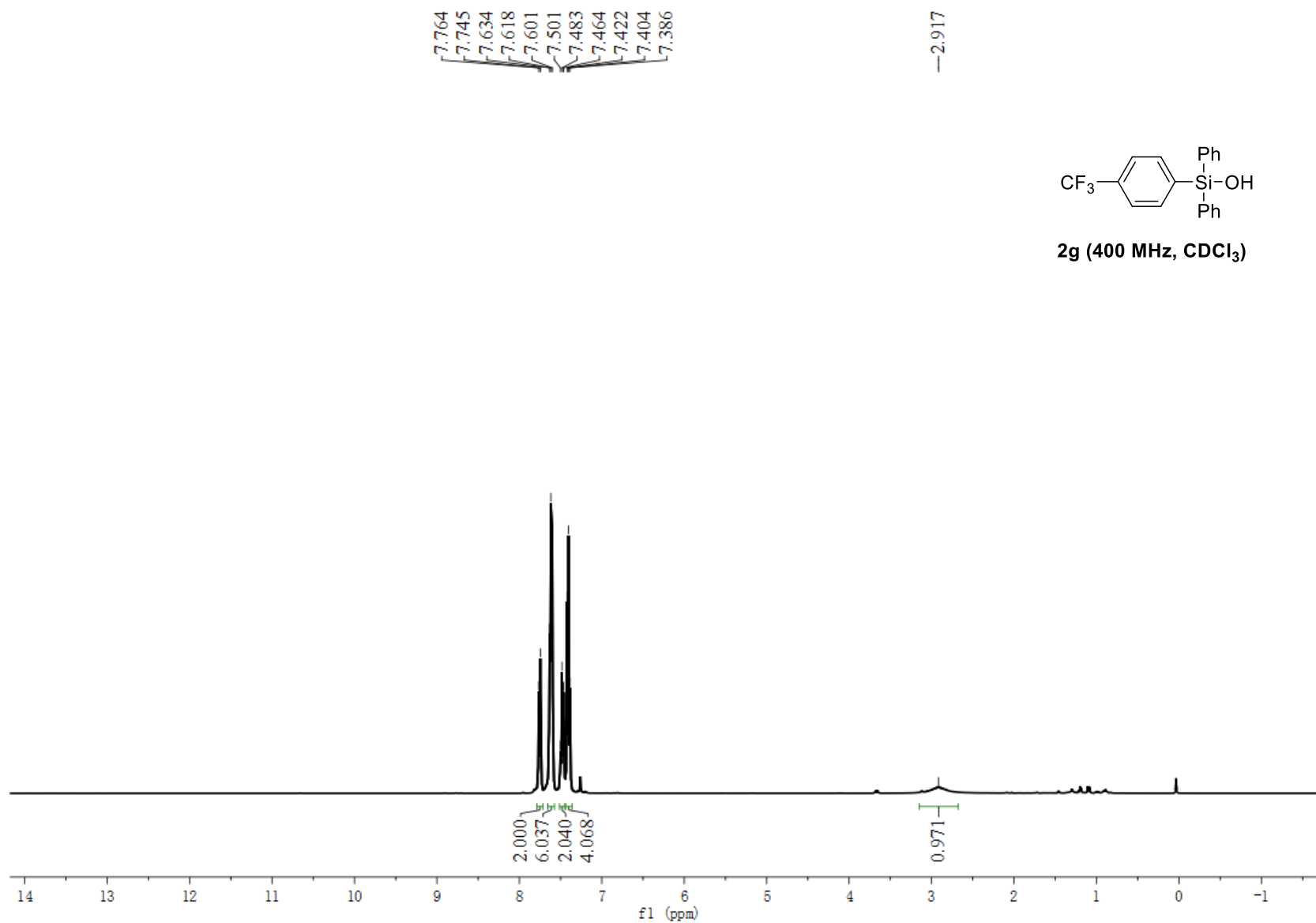
77.32  
77.00  
76.68

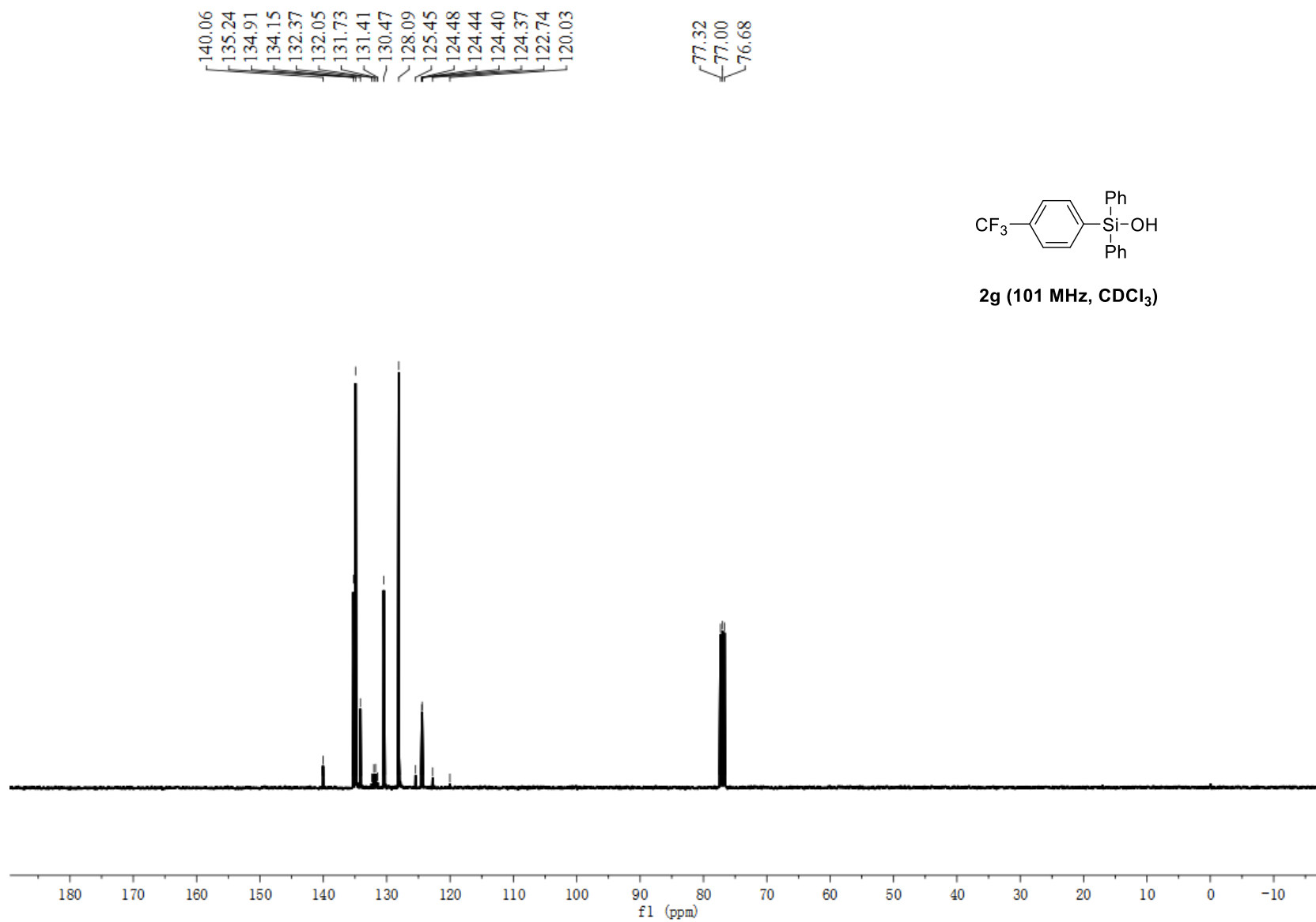


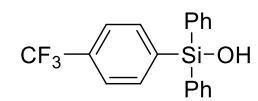
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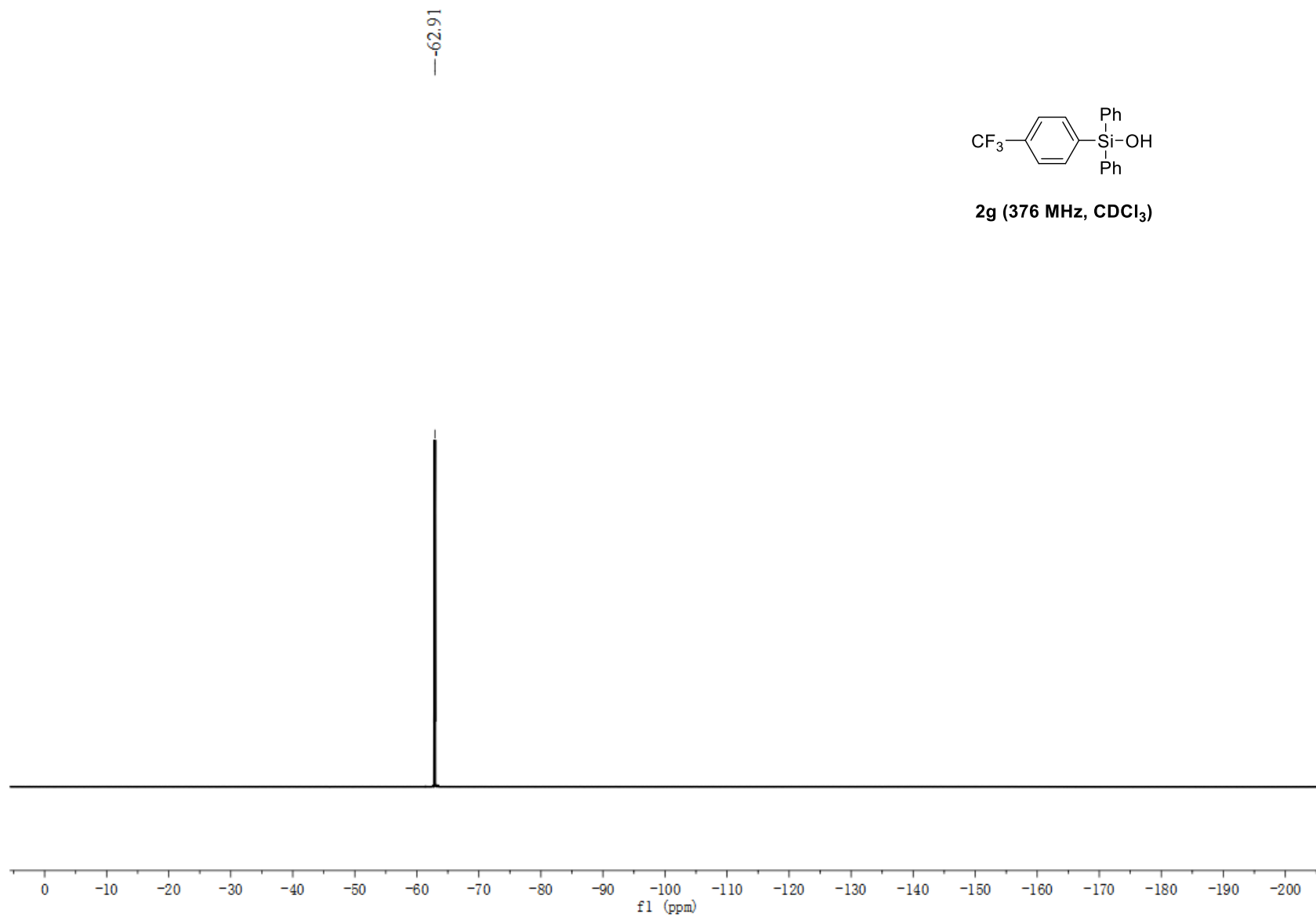


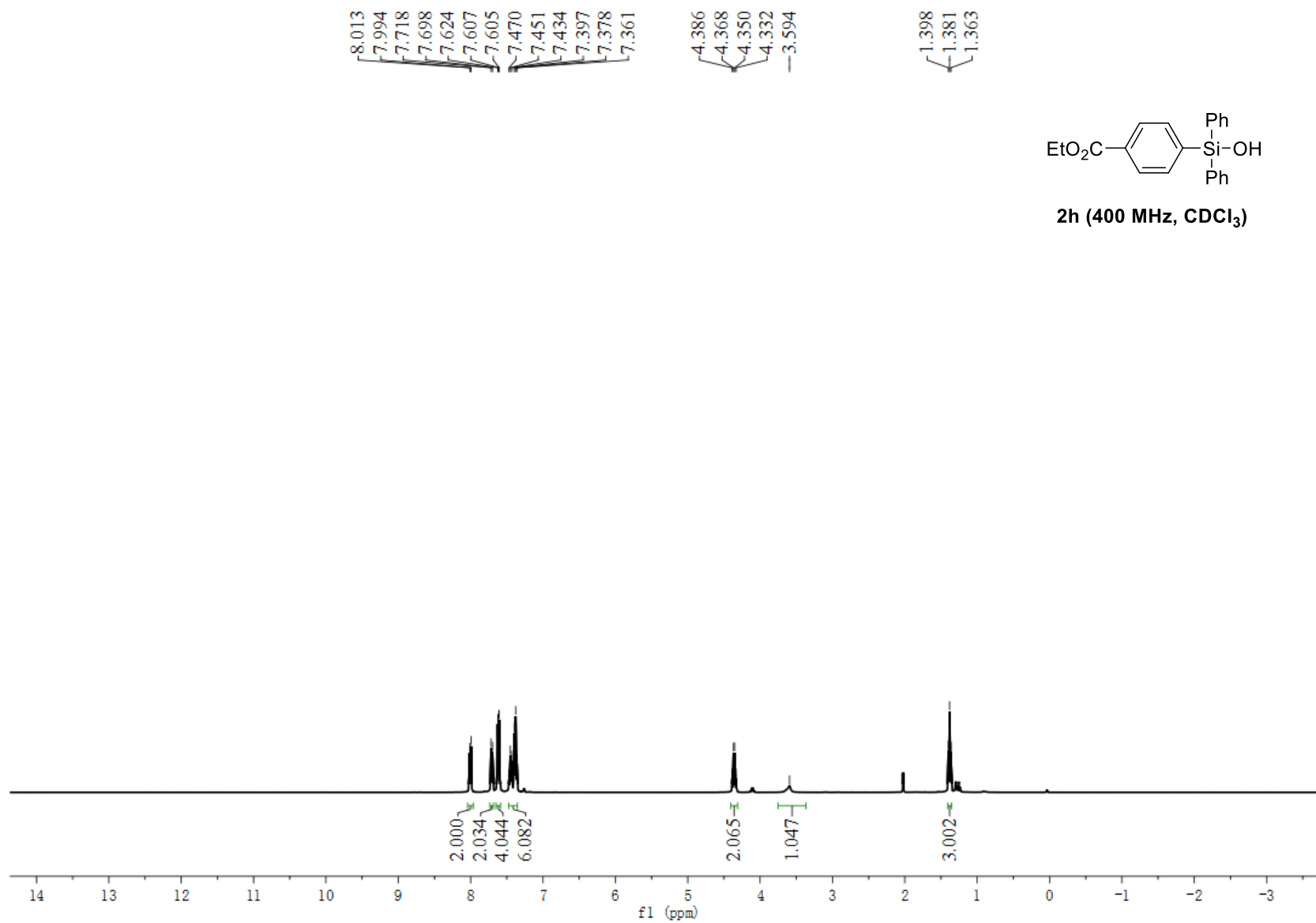


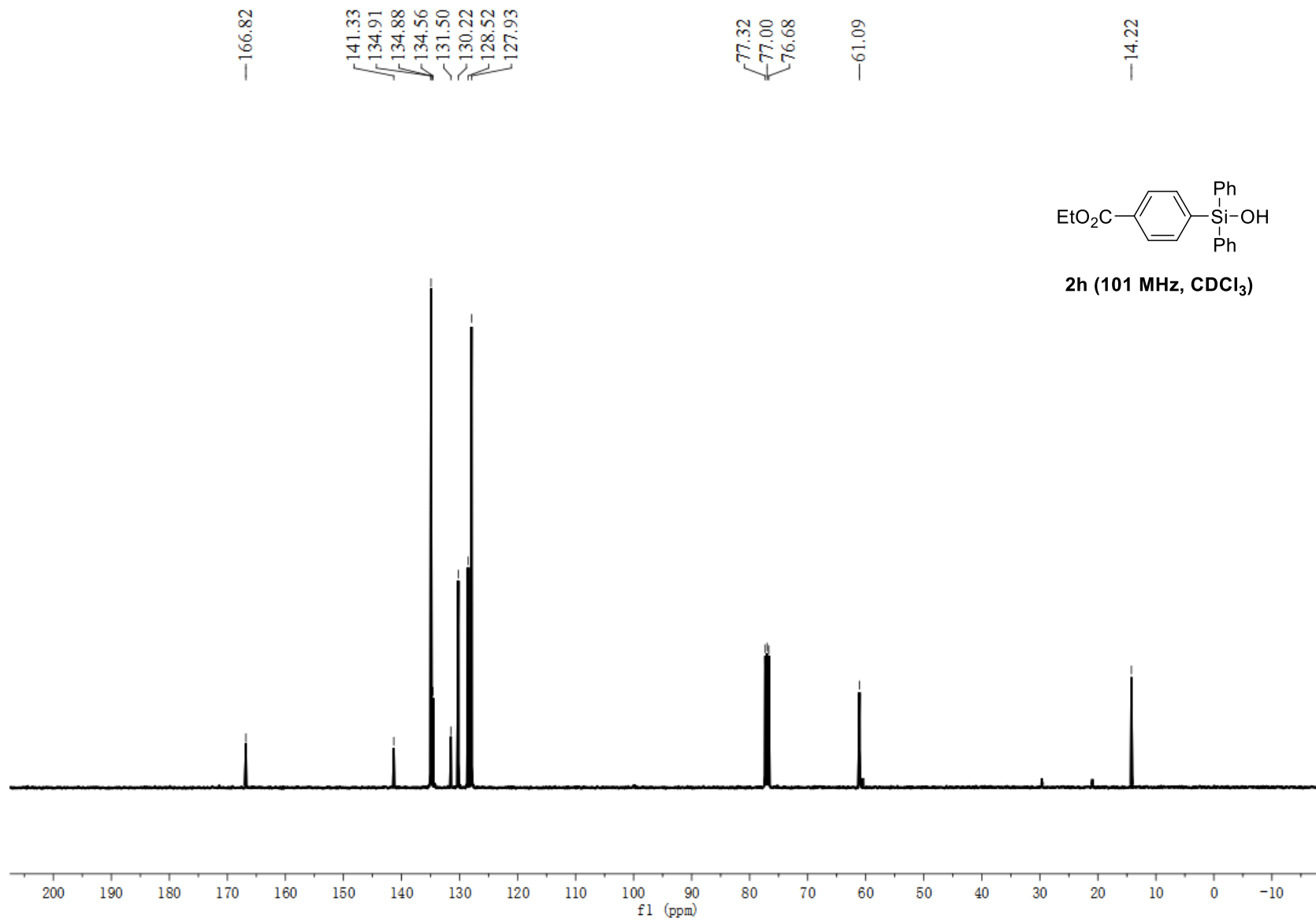




**2g (376 MHz, CDCl<sub>3</sub>)**

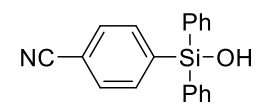




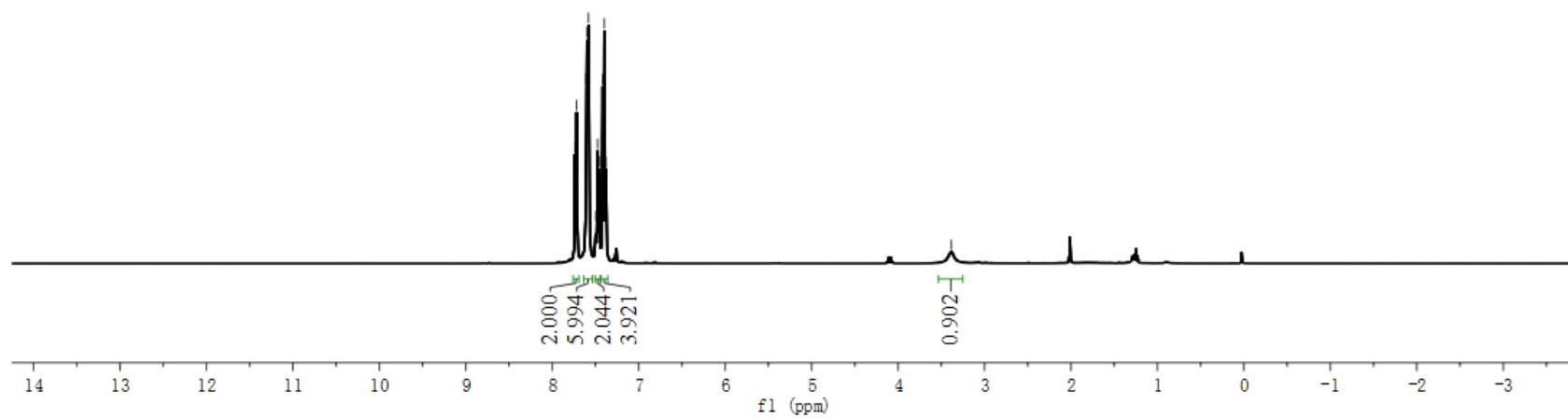


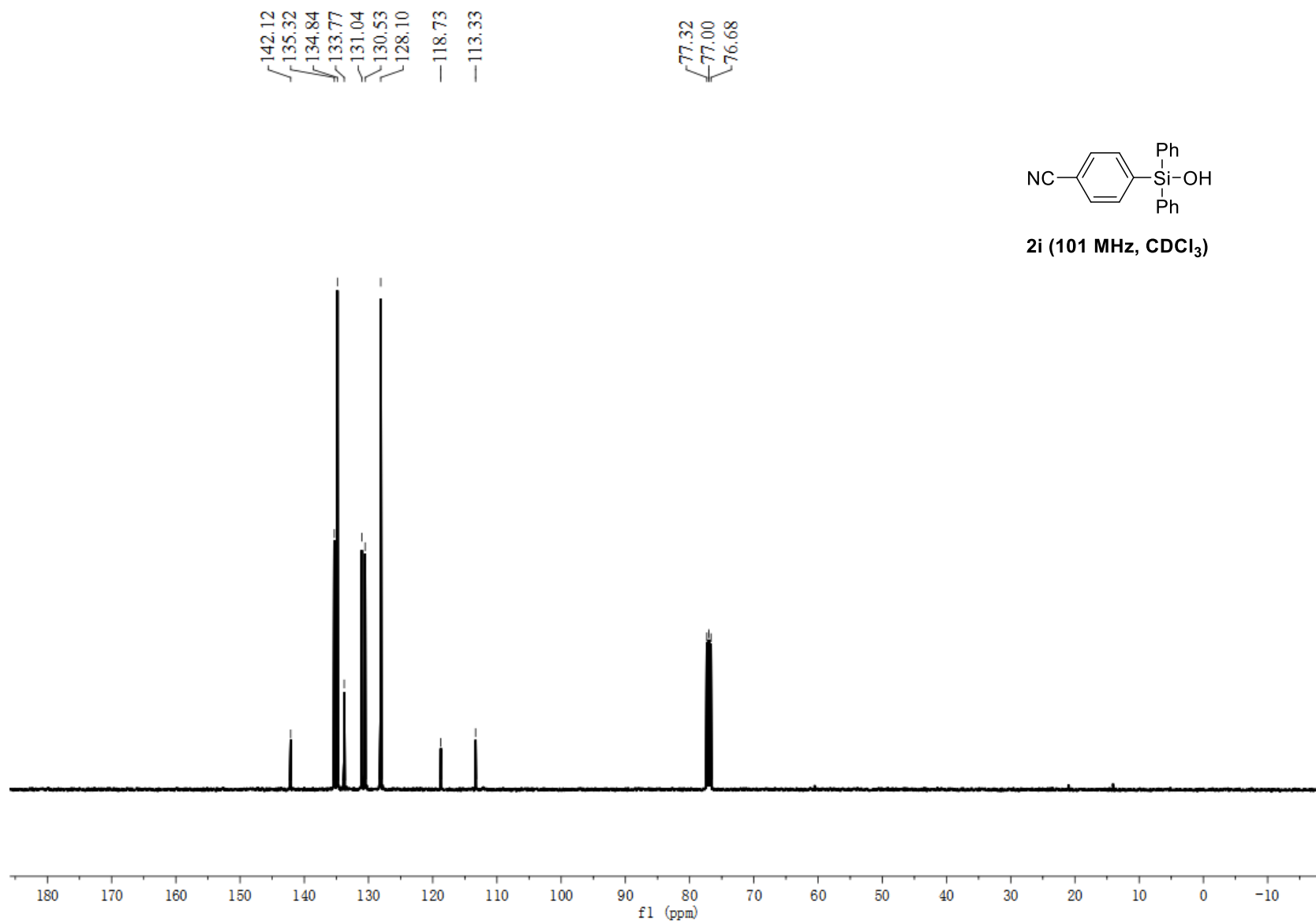
7.740  
7.720  
7.610  
7.606  
7.599  
7.590  
7.582  
7.579  
7.496  
7.478  
7.459  
7.418  
7.399  
7.381

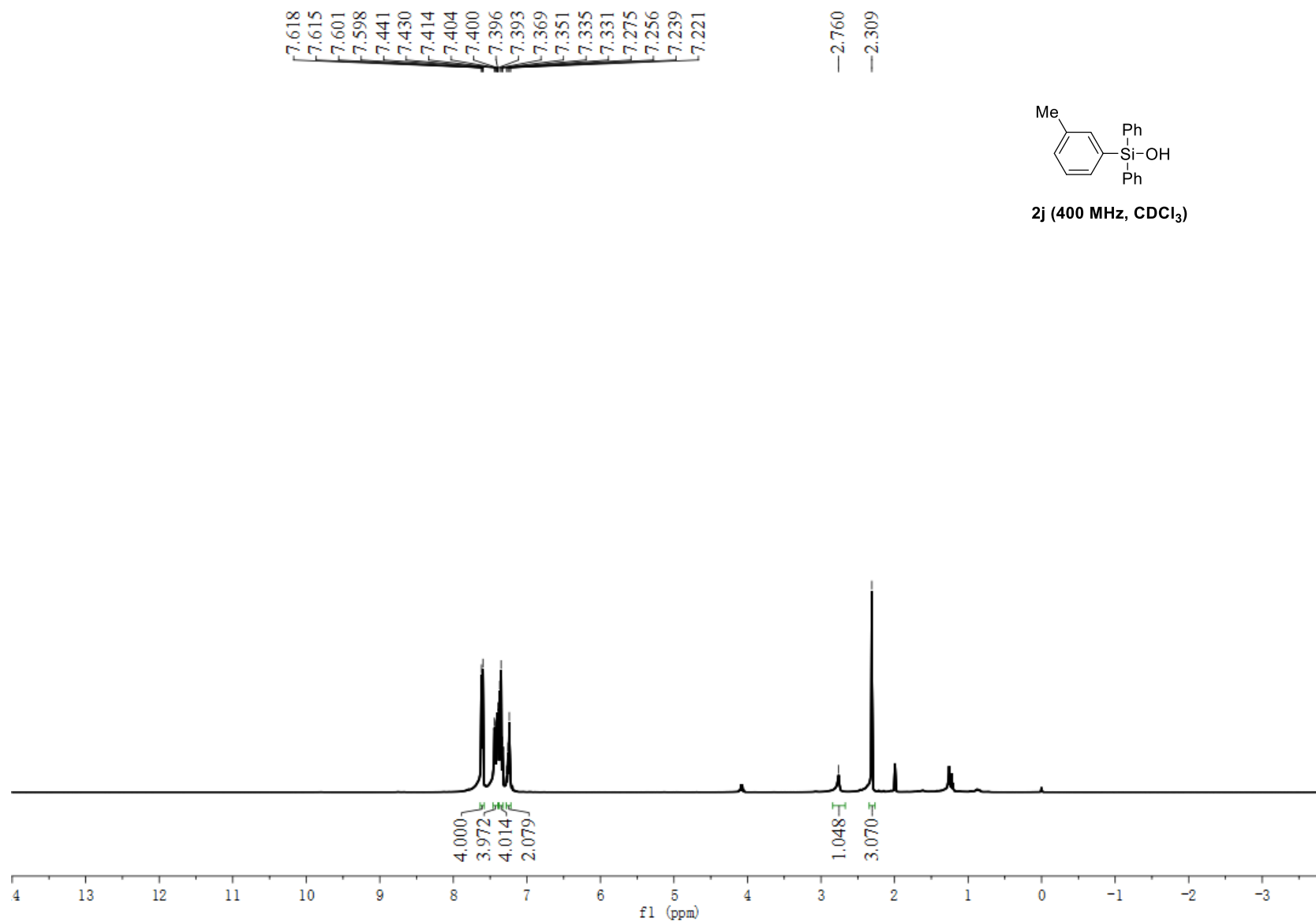
— 3.384



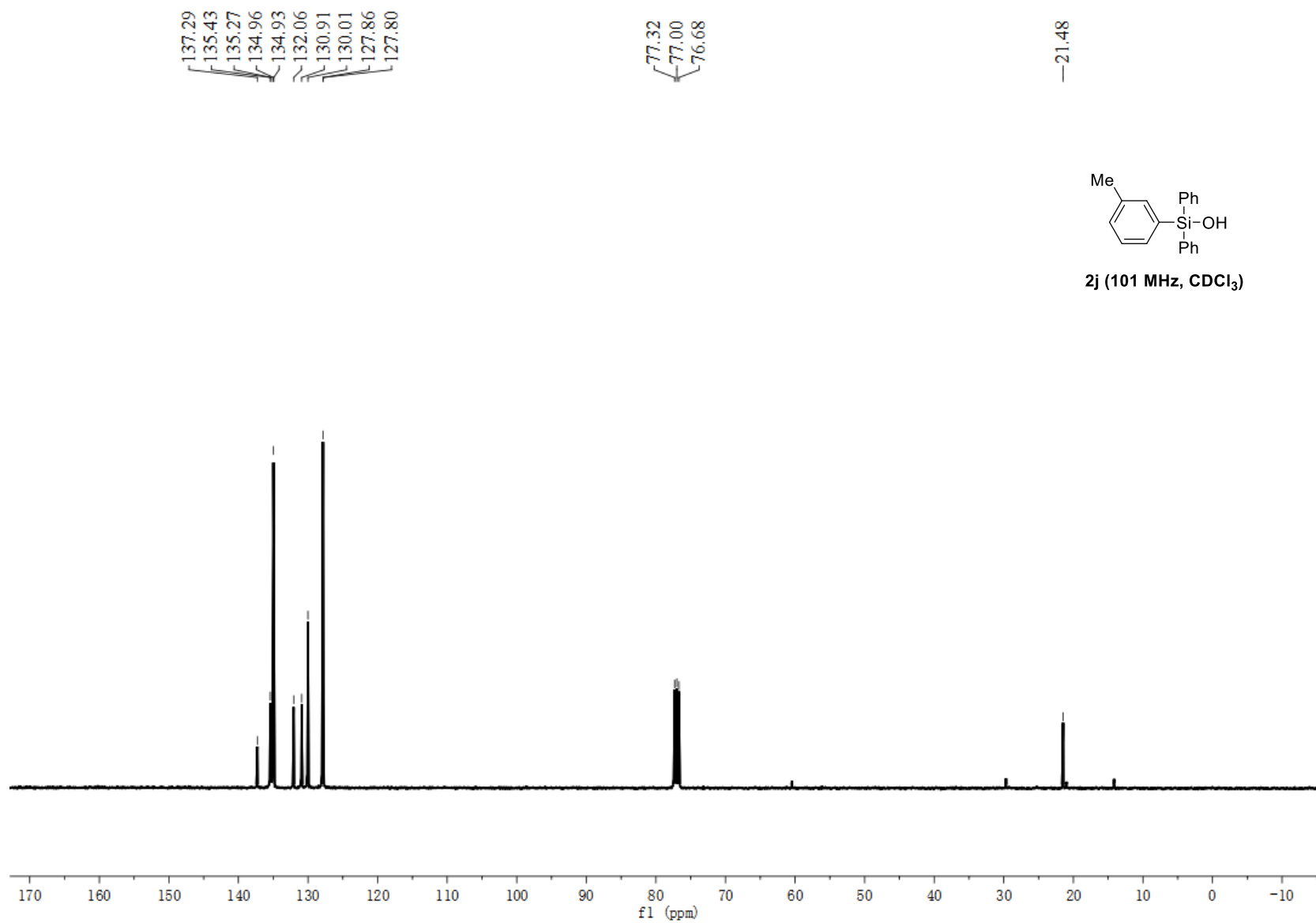
2i (400 MHz, CDCl<sub>3</sub>)

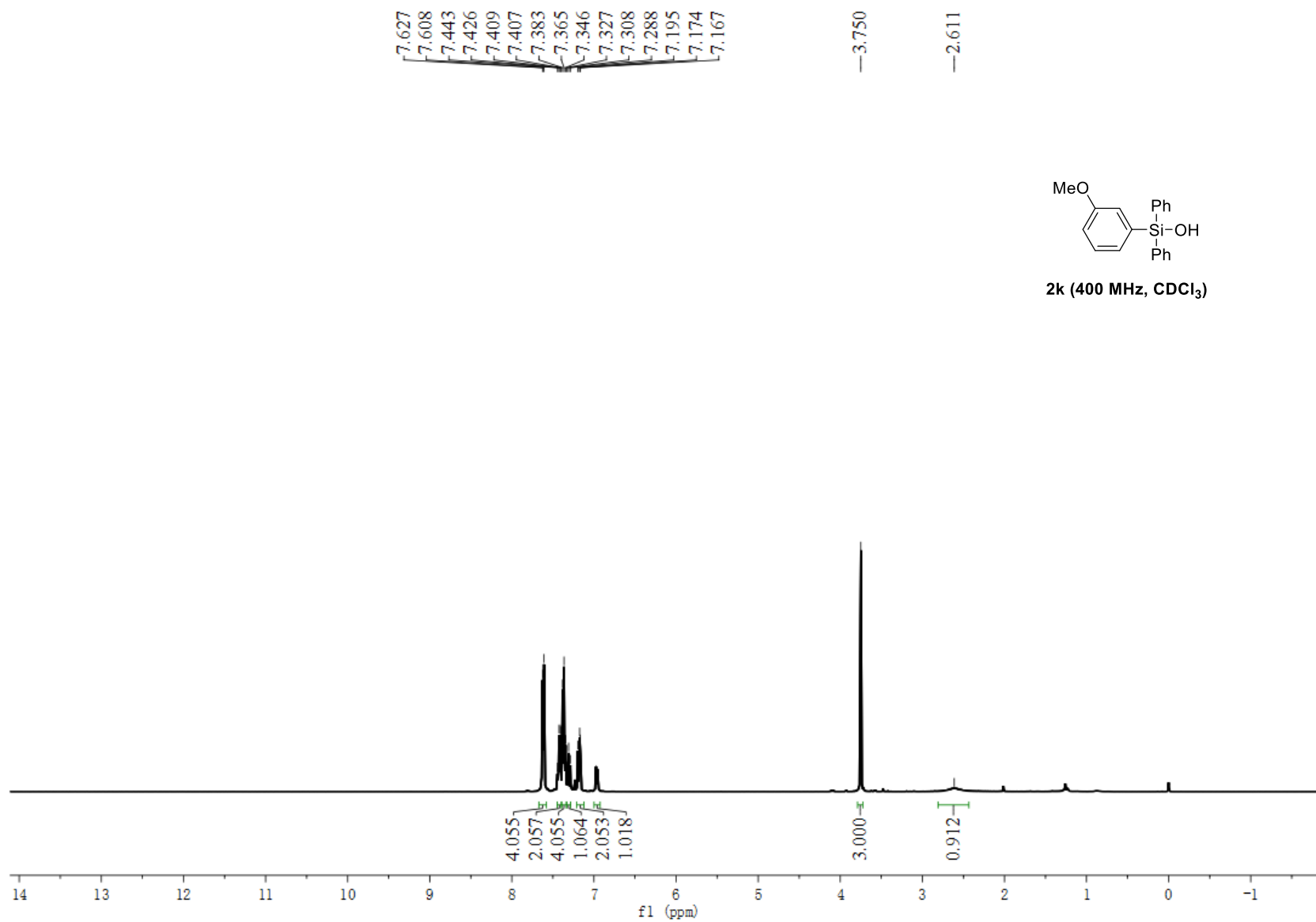


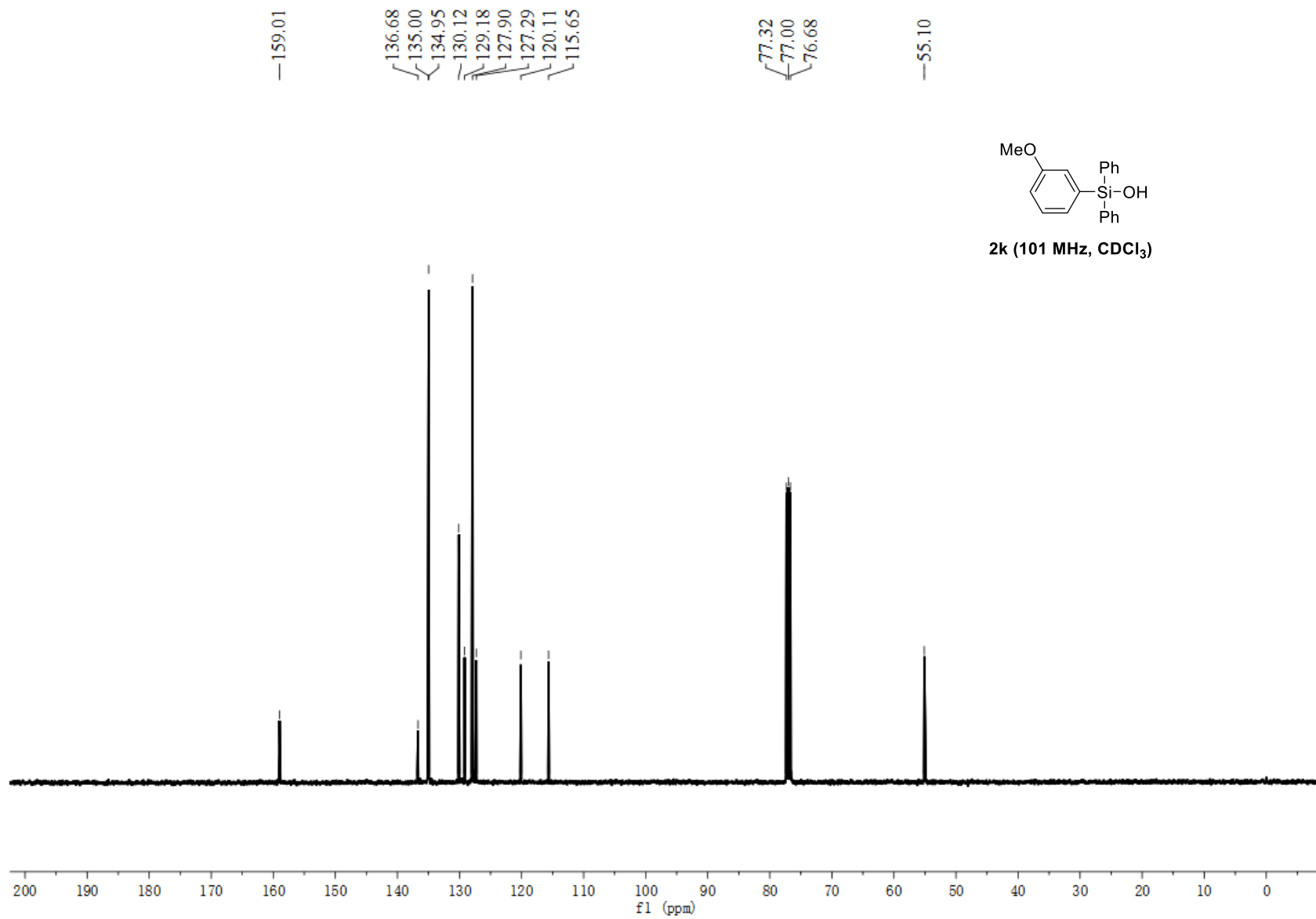


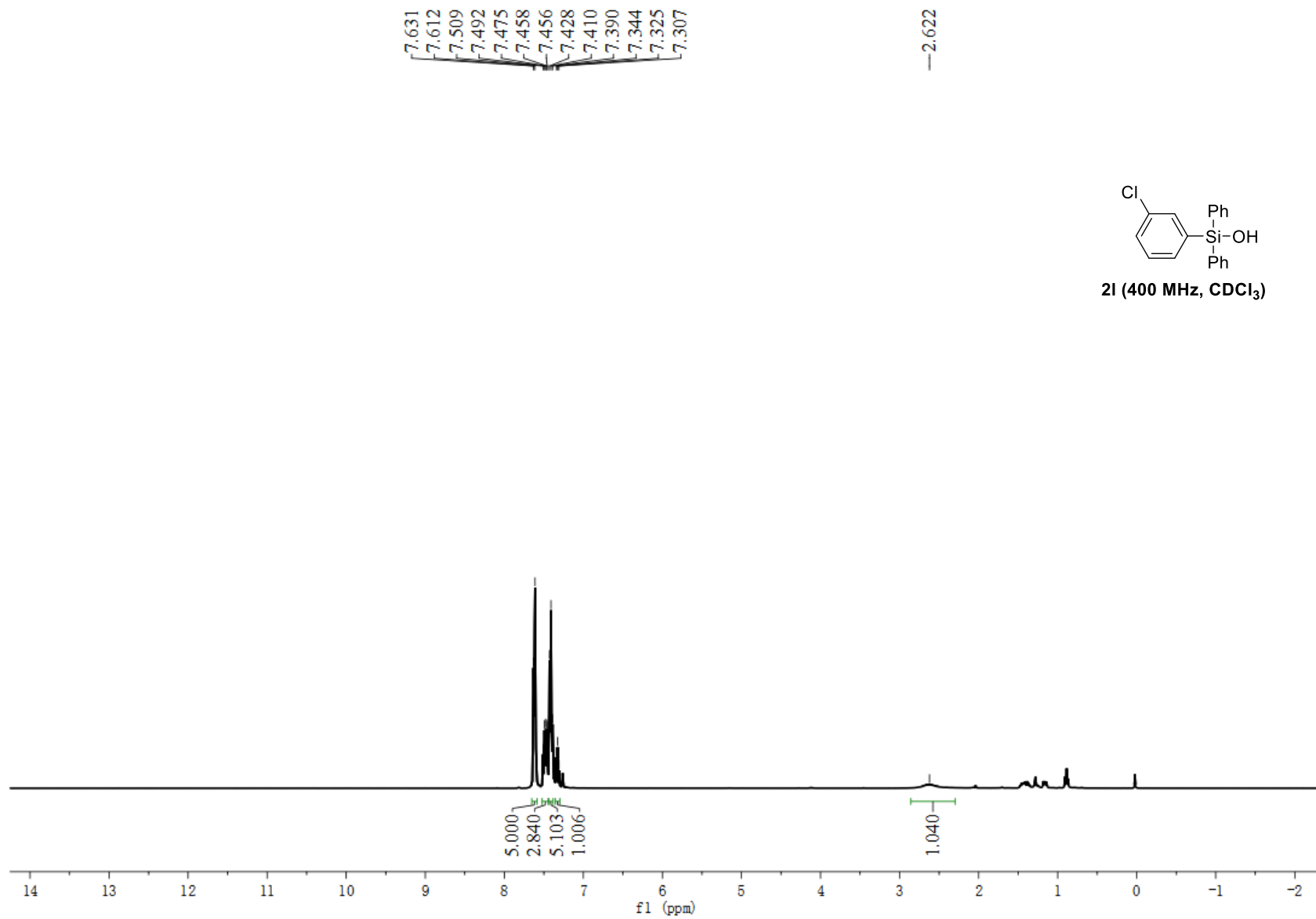


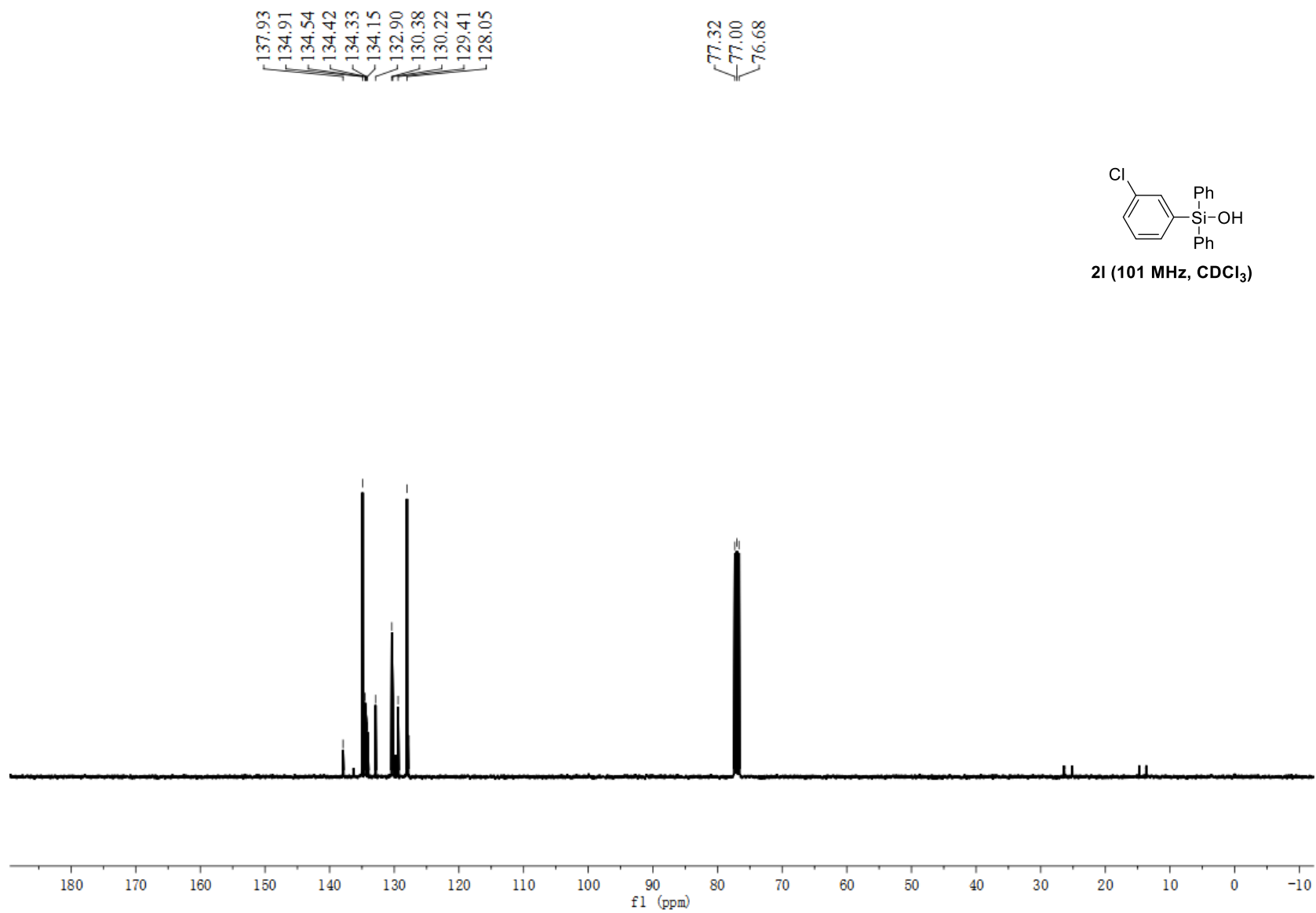


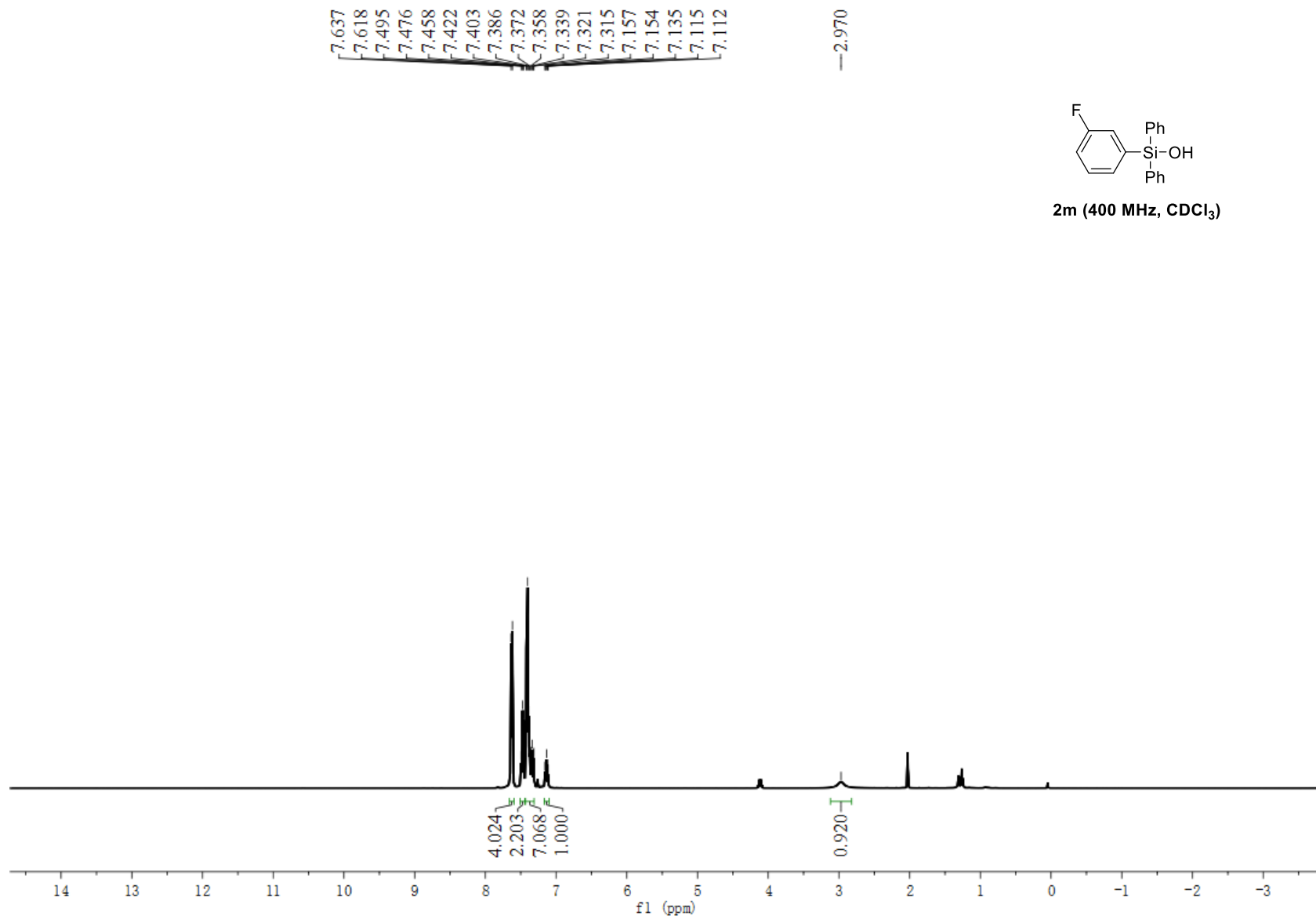


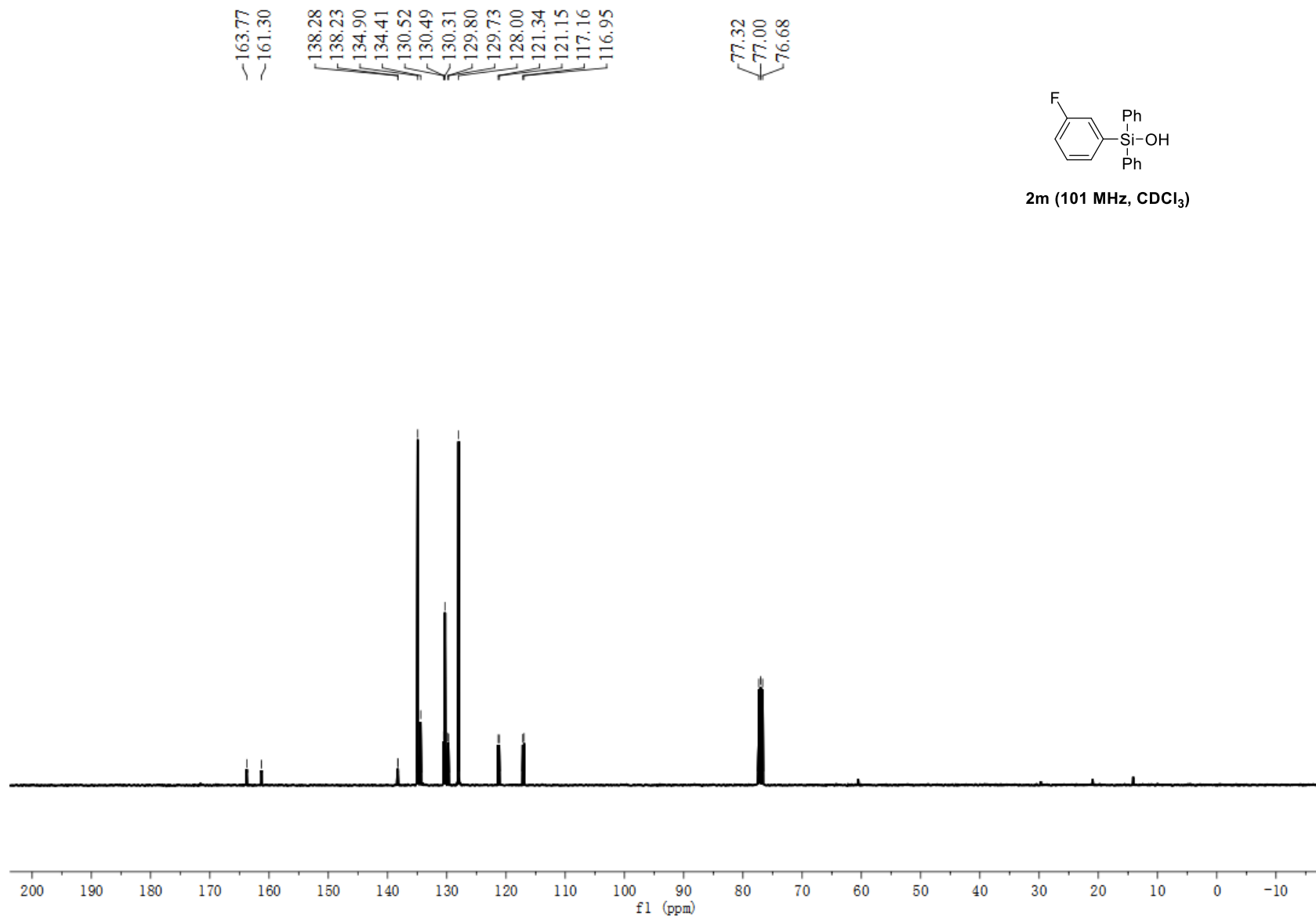


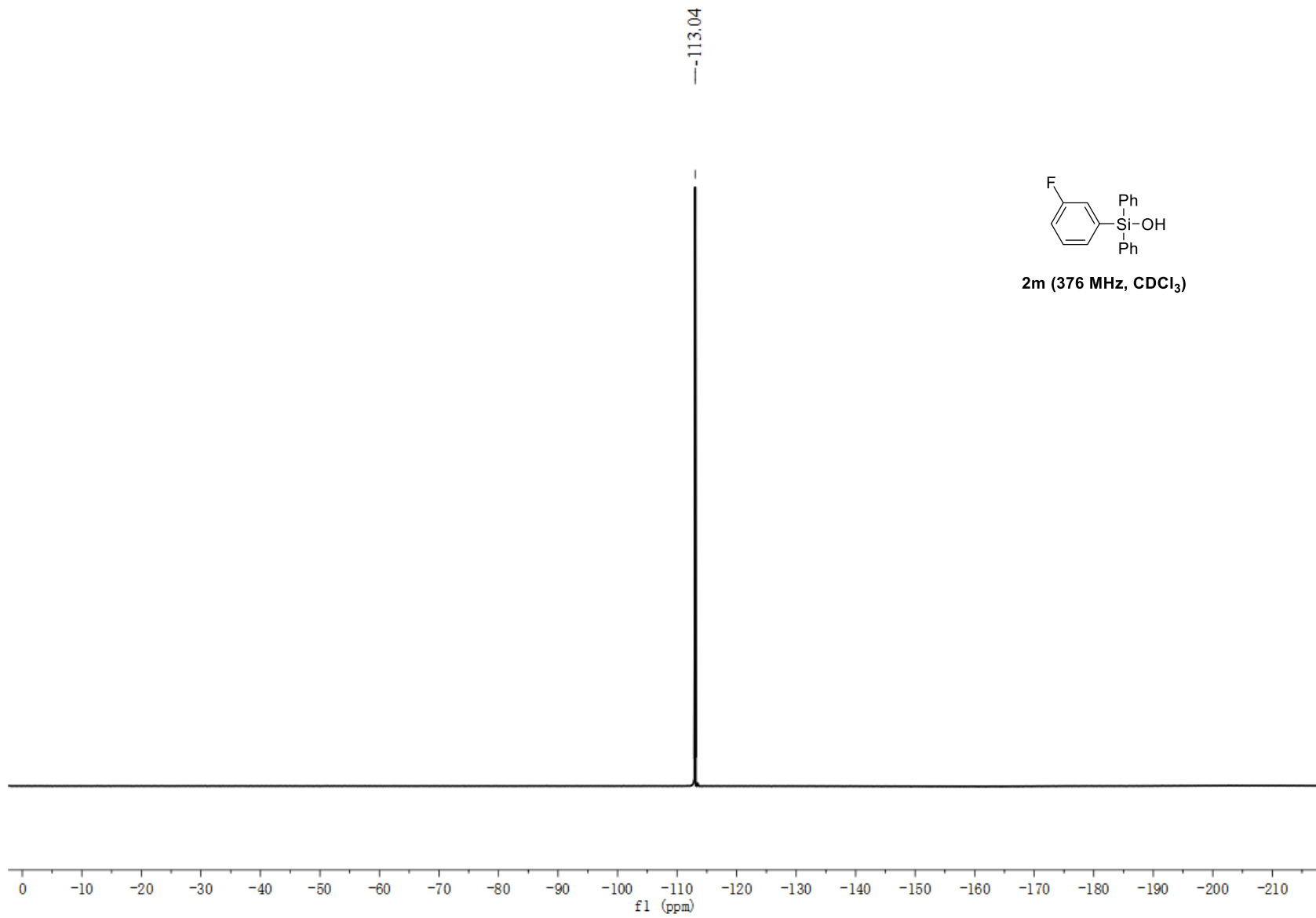








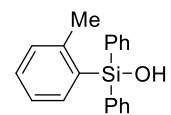




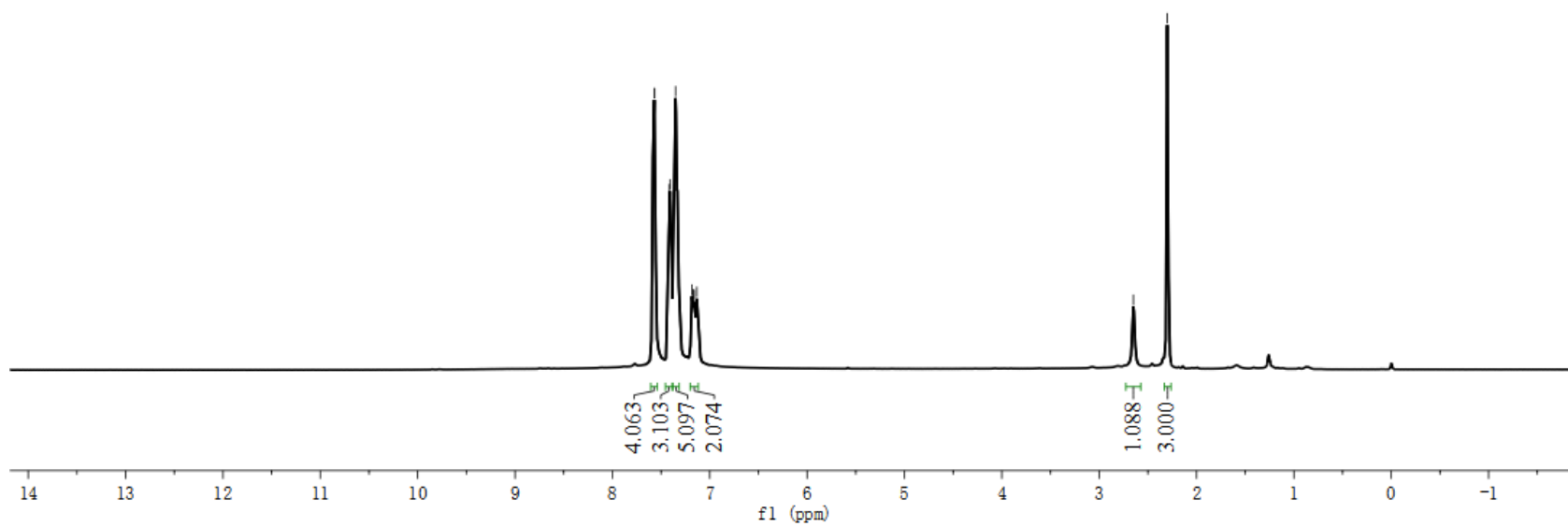


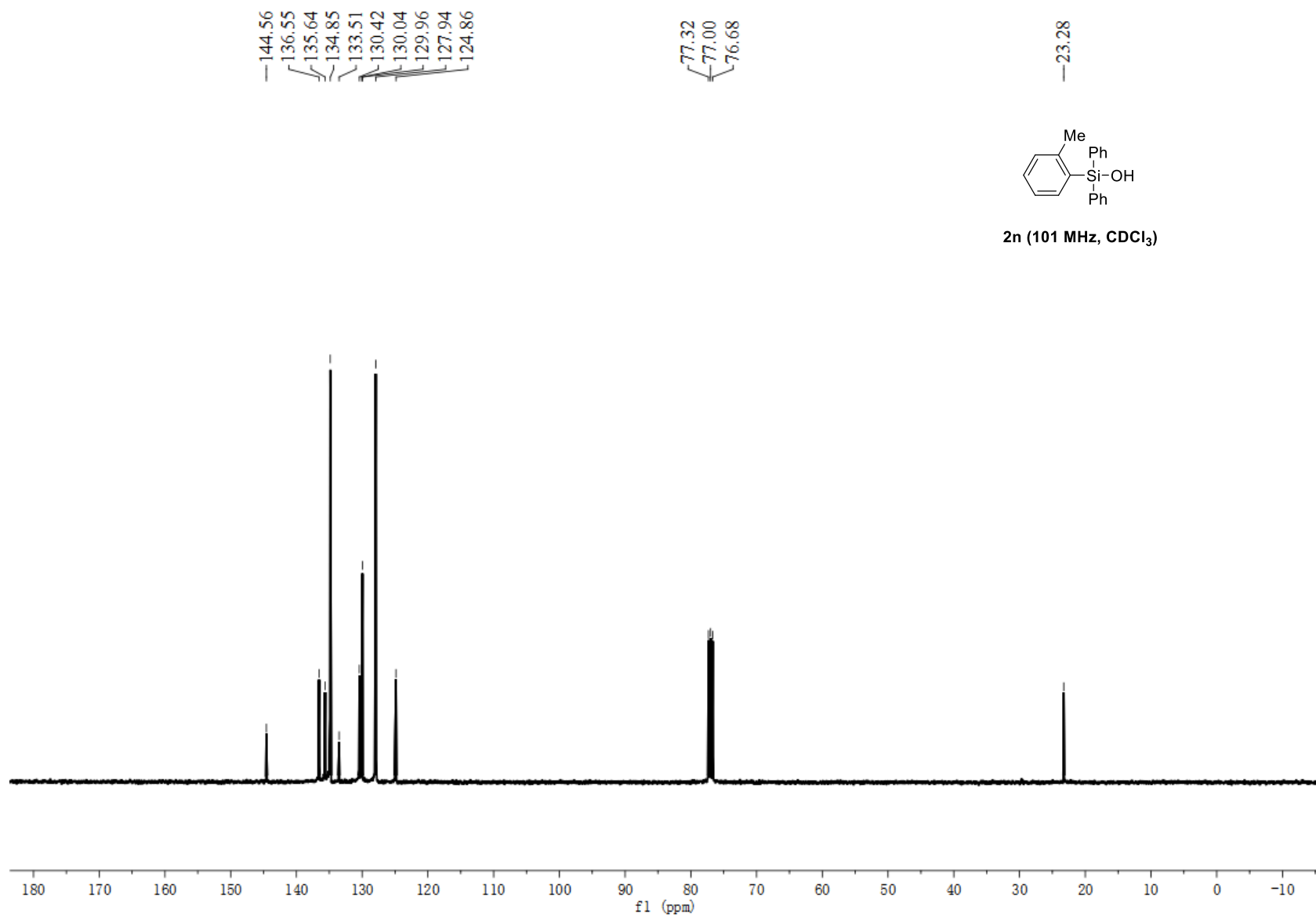
7.582  
7.578  
7.575  
7.569  
7.566  
7.415  
7.408  
7.351  
7.335  
7.184  
7.167  
7.161  
7.133

2.649  
2.301

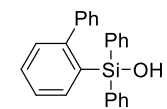


2n (400 MHz, CDCl<sub>3</sub>)

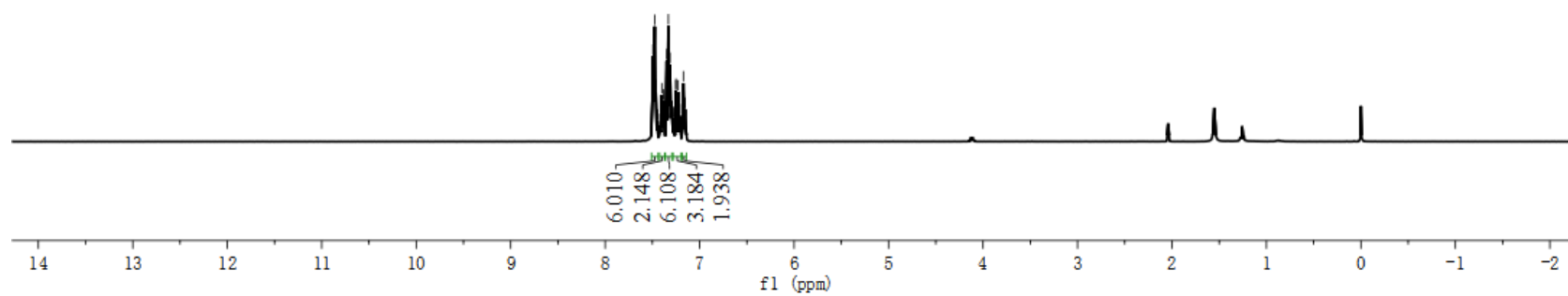


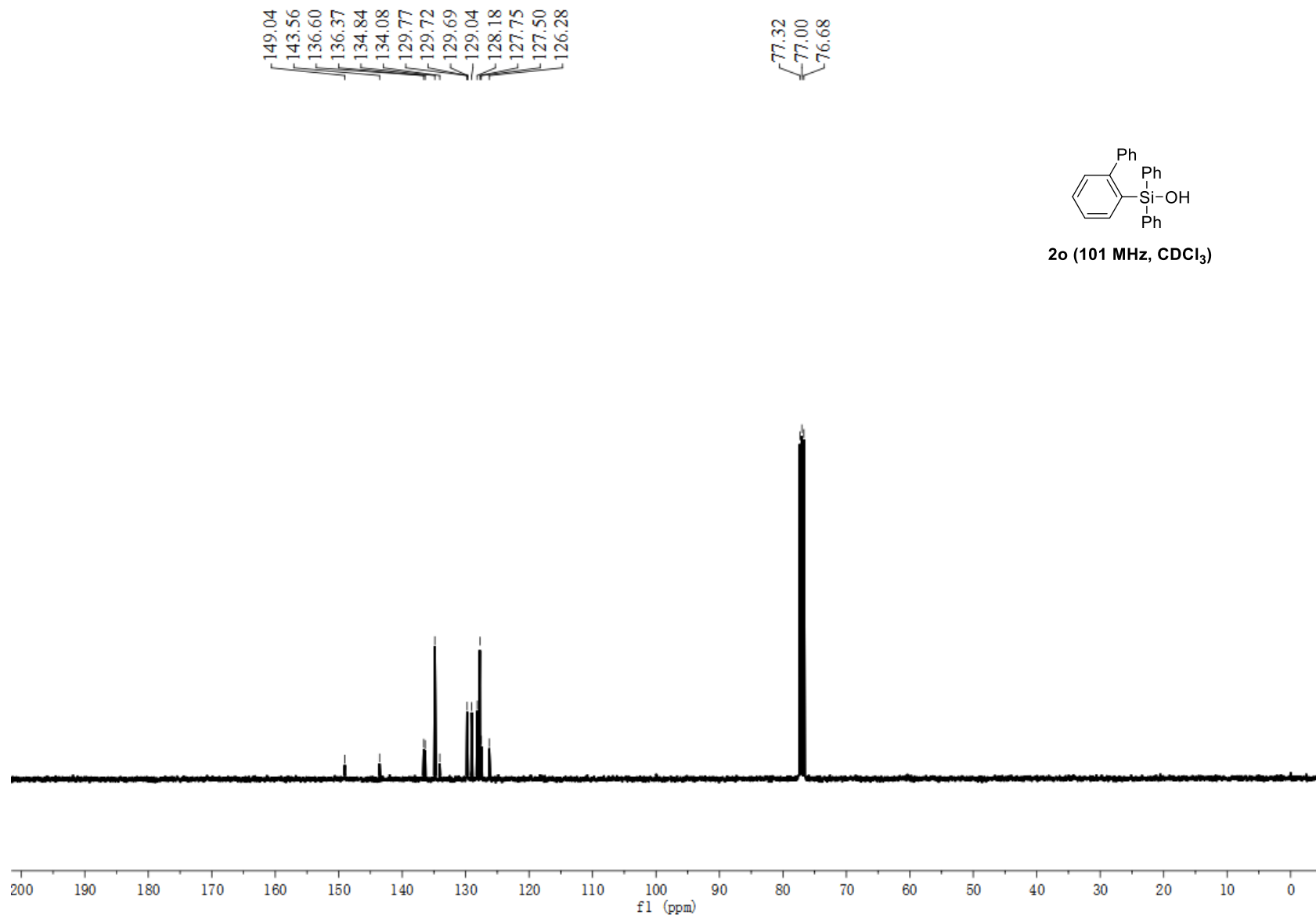


7.495  
7.476  
7.464  
7.447  
7.420  
7.401  
7.383  
7.349  
7.330  
7.313  
7.273  
7.252  
7.250  
7.227  
7.209  
7.172  
7.154

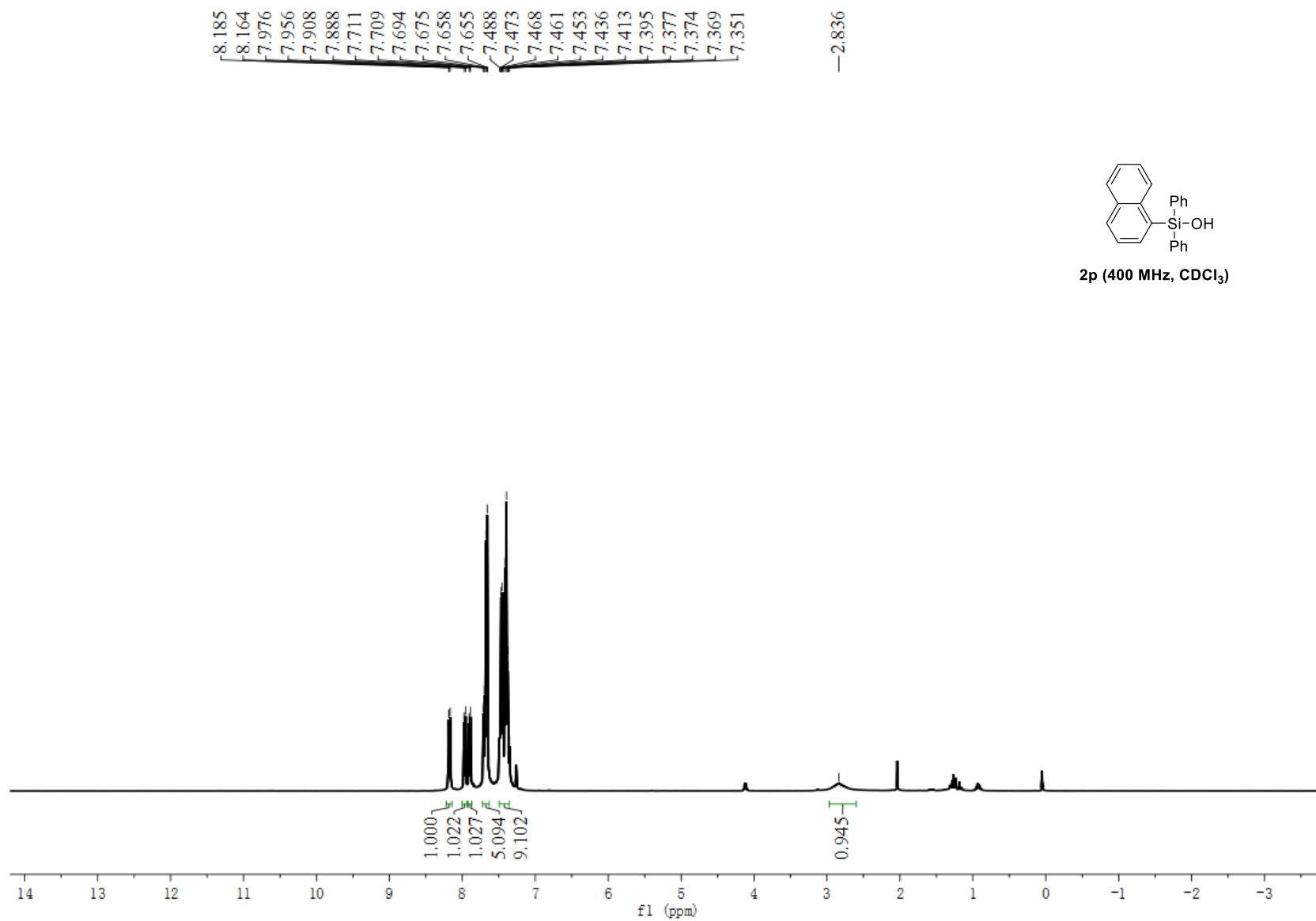


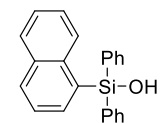
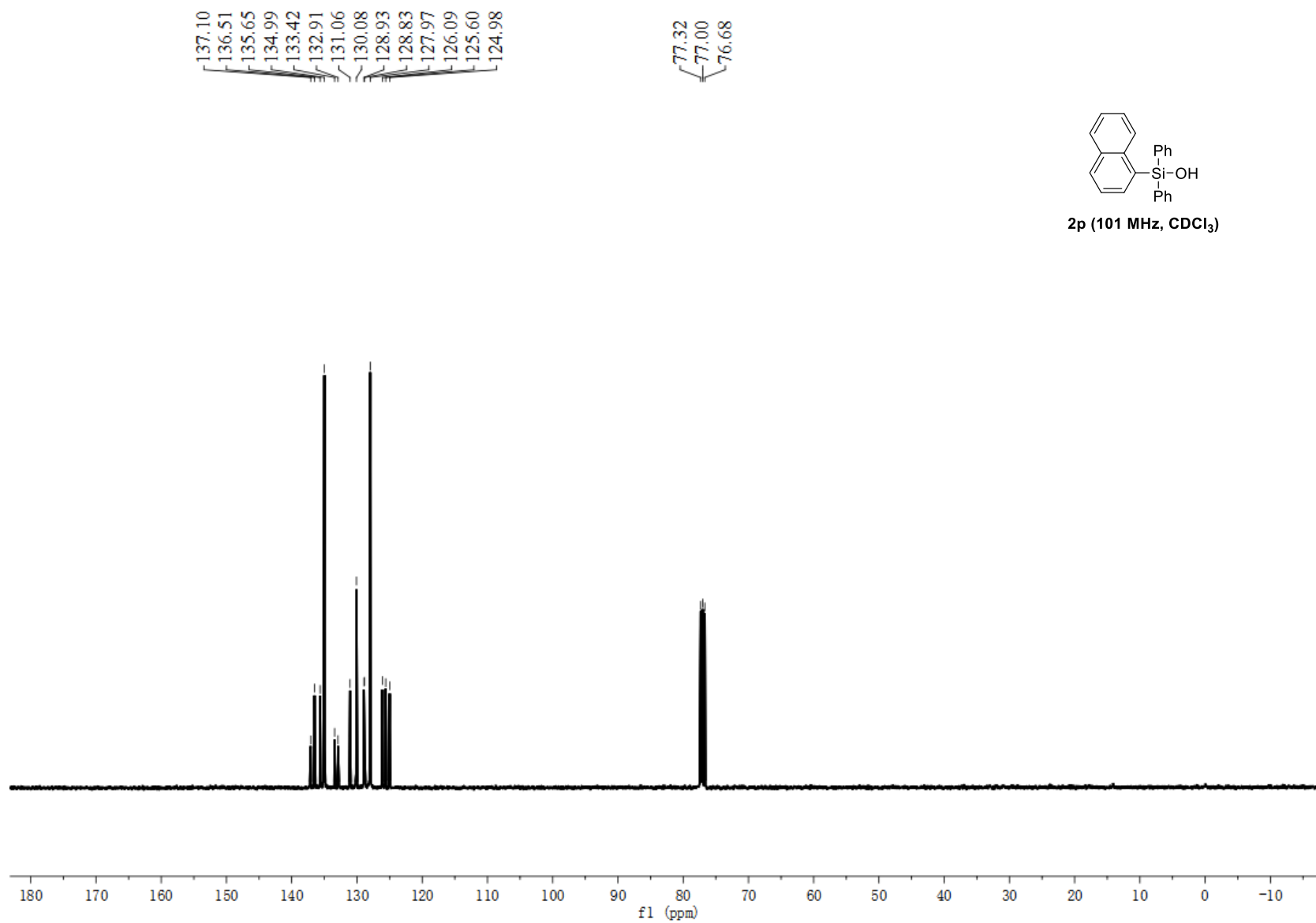
2o (400 MHz, CDCl<sub>3</sub>)



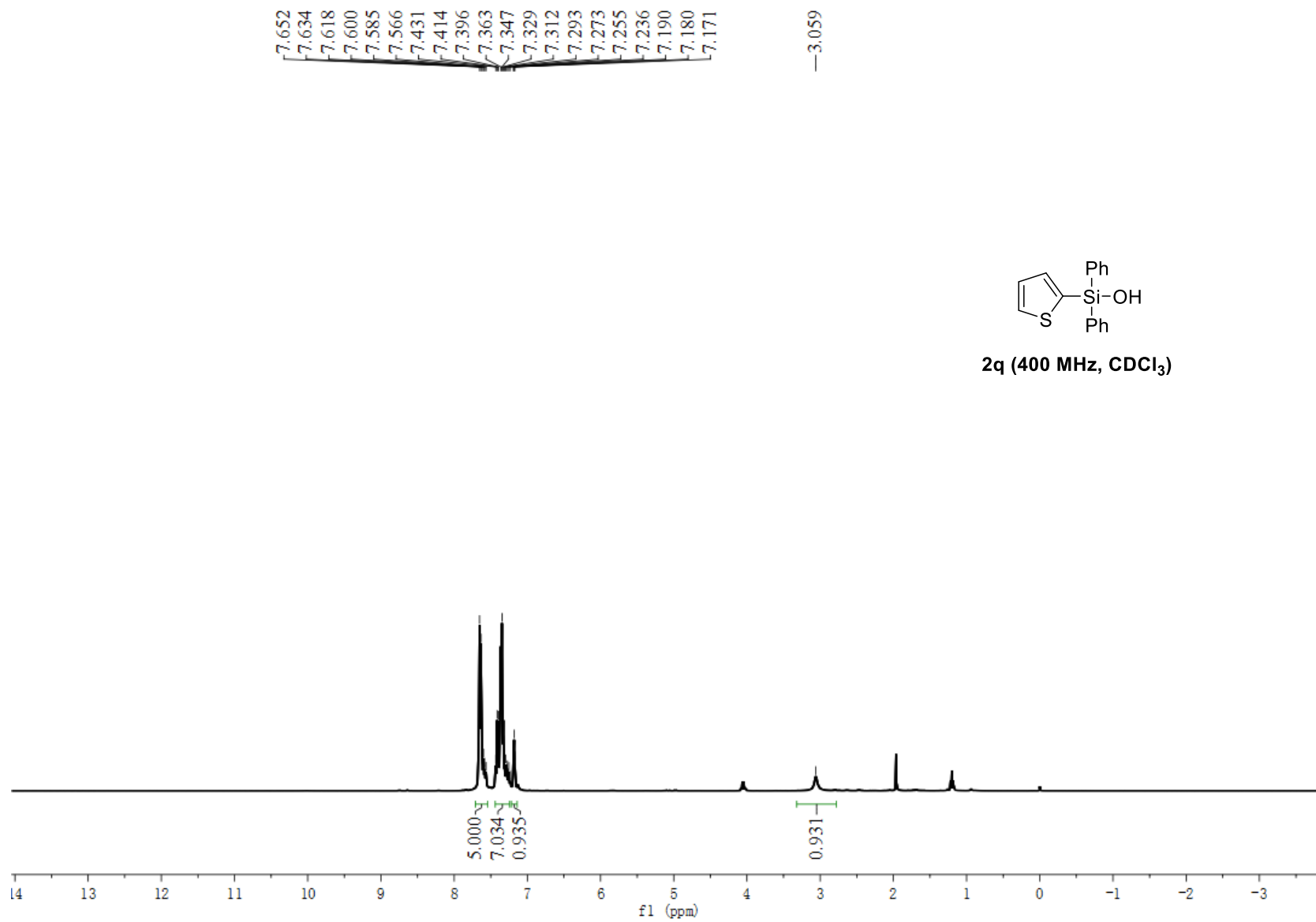


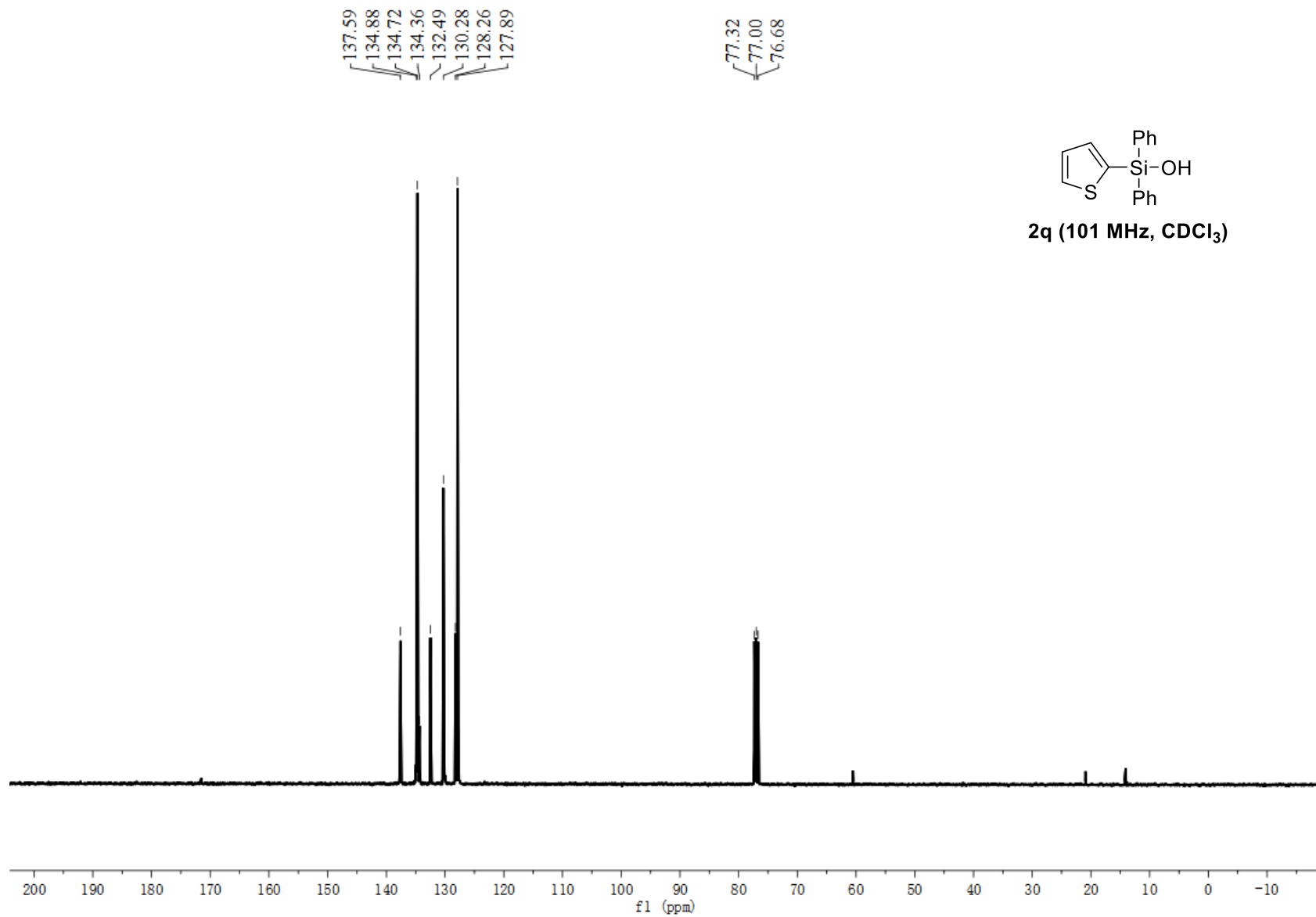
**2o** (101 MHz, CDCl<sub>3</sub>)



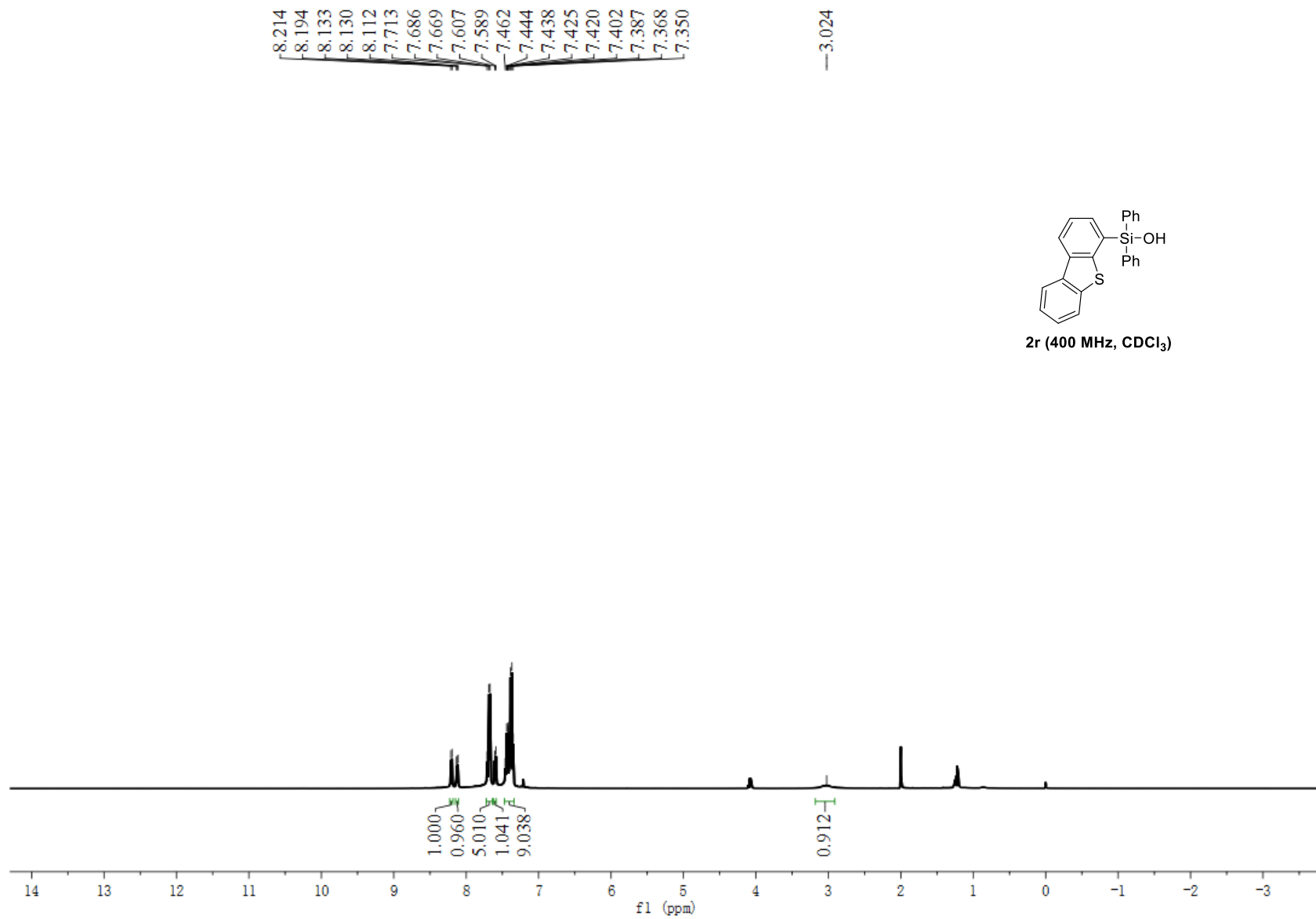


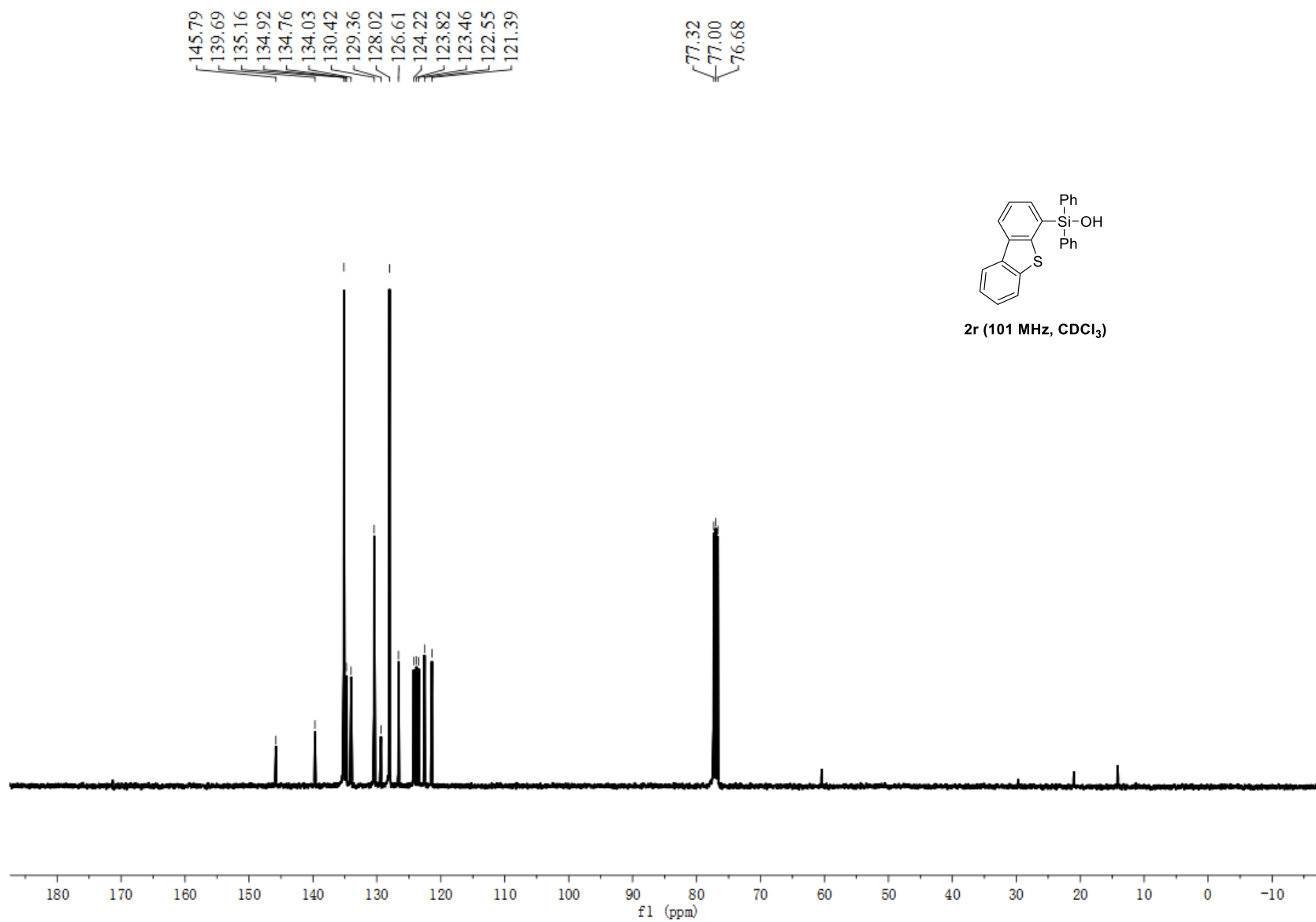
2p (101 MHz, CDCl<sub>3</sub>)



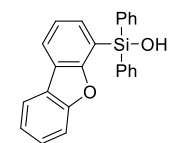








8.077  
8.058  
7.996  
7.977  
7.754  
7.734  
7.543  
7.525  
7.502  
7.481  
7.463  
7.444  
7.434  
7.415  
7.399  
7.397  
7.390  
7.380  
7.371  
7.362  
7.353  
7.343



2s (400 MHz, CDCl<sub>3</sub>)

