

## Electrochemical selenylative *ipso*-Annulation of *N*-benzylacrylamides to seleno-azaspiro[4.5]decadienones

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and Uprety Ajaykumar<sup>a,b</sup>

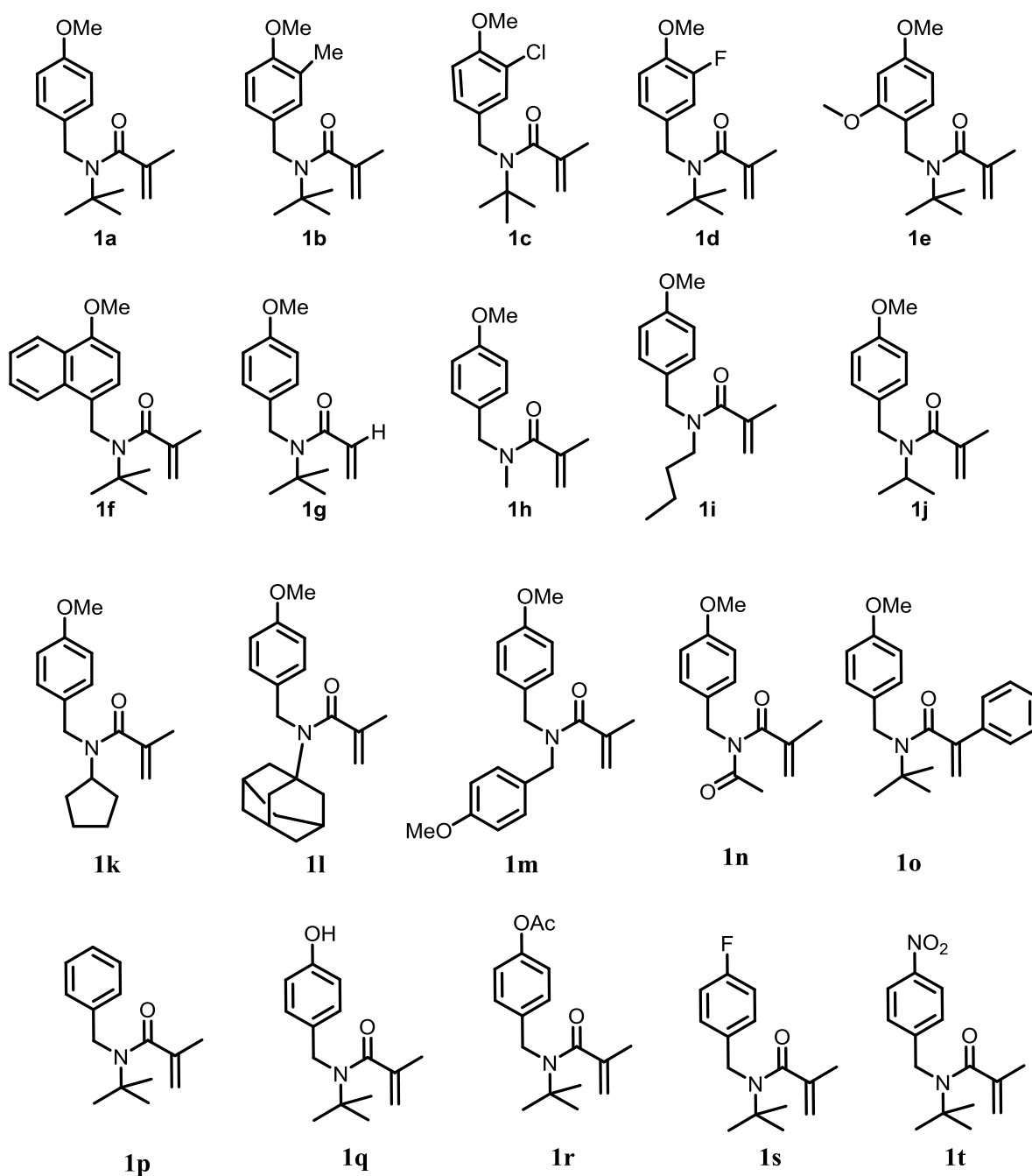
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## 1. Structures of *N*-Benzylacrylamides



All of the starting materials **1a** to **1m** and **1o** to **1t** were prepared based on literature reports, and the spectral data was compared.

### *N*-Acetyl-*N*-(4-methoxybenzyl)methacrylamide (**1n**):

White liquid, 667 mg, 90% yield,  $R_f = 0.8$  (Ethyl Acetate : Hexane = 2:8);  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20 (d,  $J = 8.6$  Hz, 2H), 6.84 (d,  $J = 8.6$  Hz, 2H), 5.38 (d,  $J = 31.6$  Hz, 2H), 4.88 (s, 2H), 3.78 (s, 3H), 2.28 (s, 3H), 1.95 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  175.3, 173.0, 158.9, 142.1, 129.3 (3C), 121.3, 113.9, 55.2, 48.0, 26.4, 18.8; HRMS (ESI):  $m/z$  calcd for  $\text{C}_{14}\text{H}_{18}\text{NO}_3$  ( $\text{M}+\text{H}$ ) $^+$  248.1287, Found 248.1294.

## 2. Visual representation of the reaction set-up:



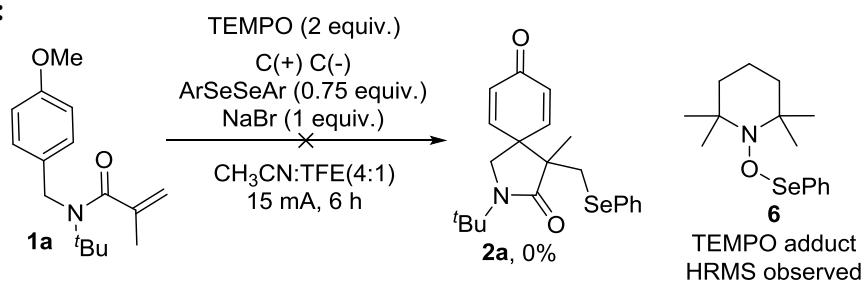
A. Initiation of the reaction



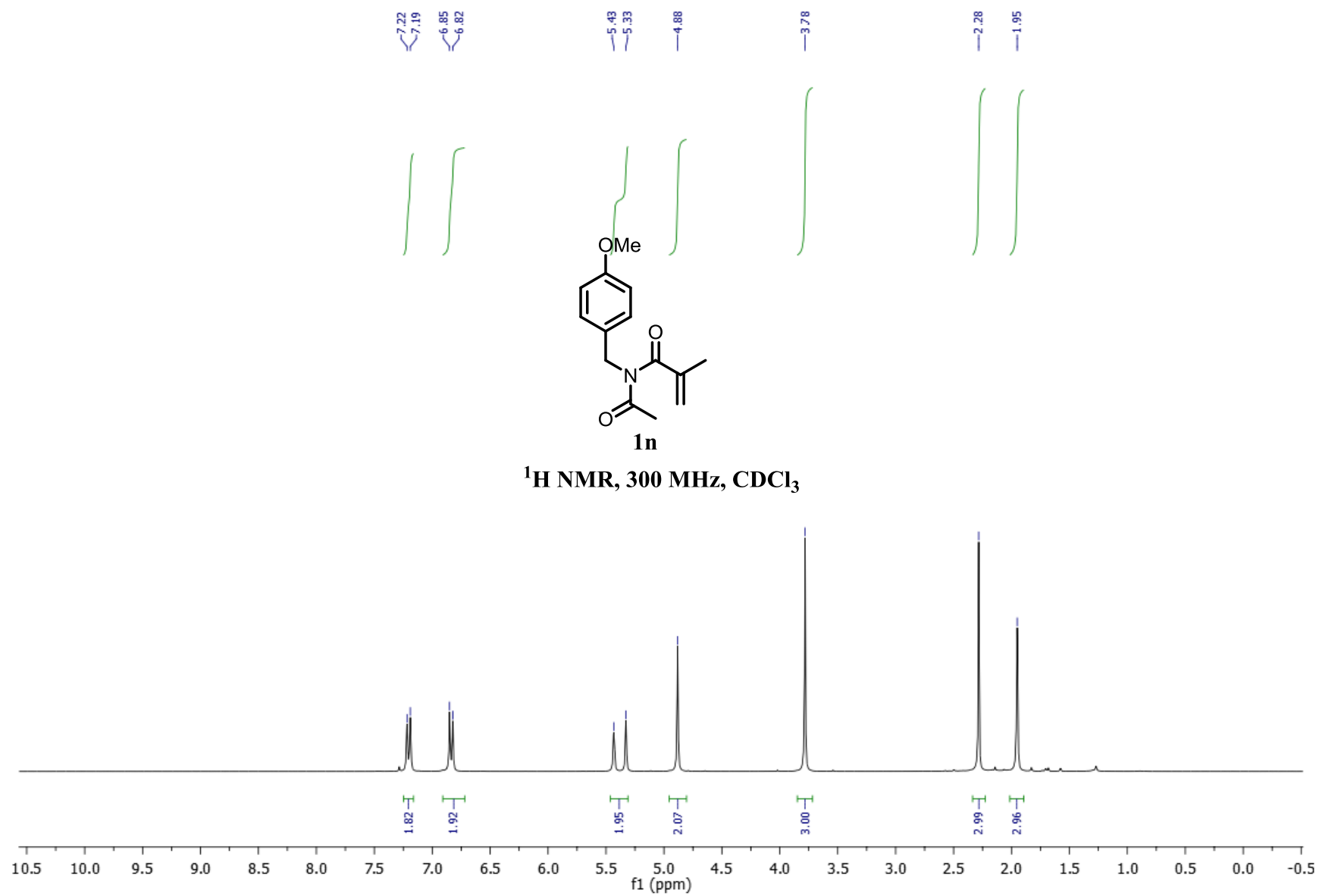
B. Completion of the reaction

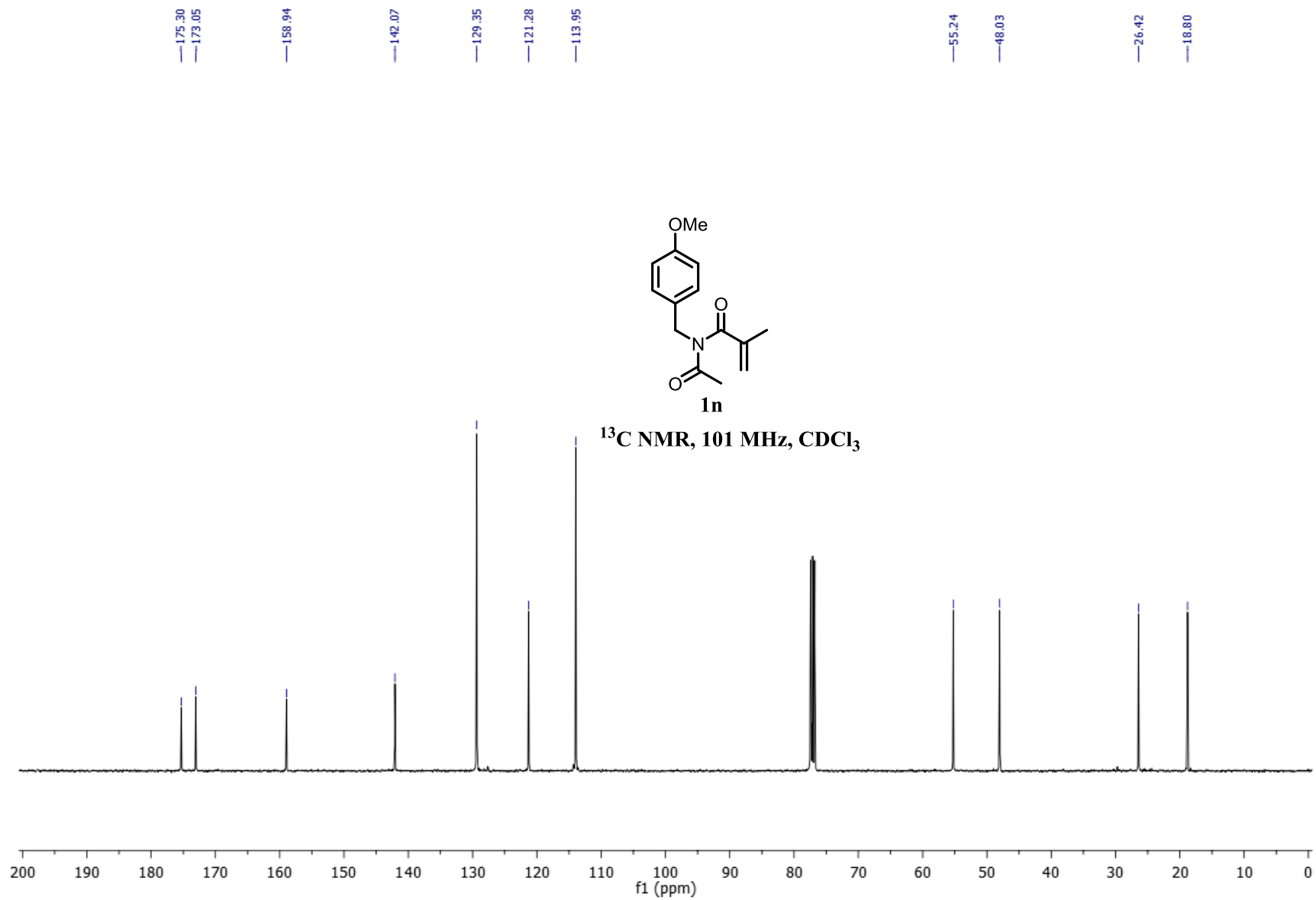
## 3. Control Experiment

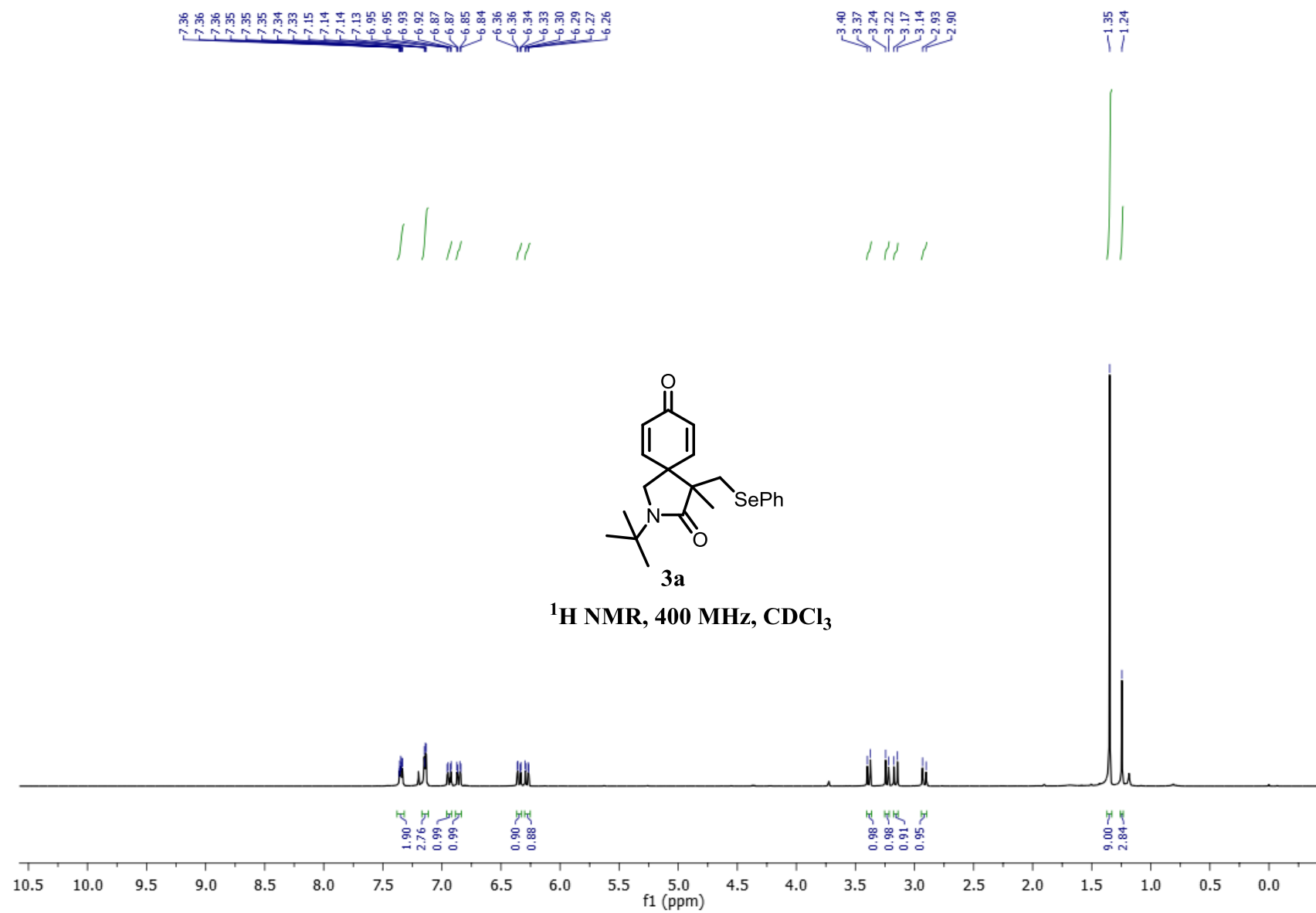
### A. Radical trapping reaction with 2,2,6,6-tetramethylpiperidin-1-yl)oxidanyl (TEMPO):

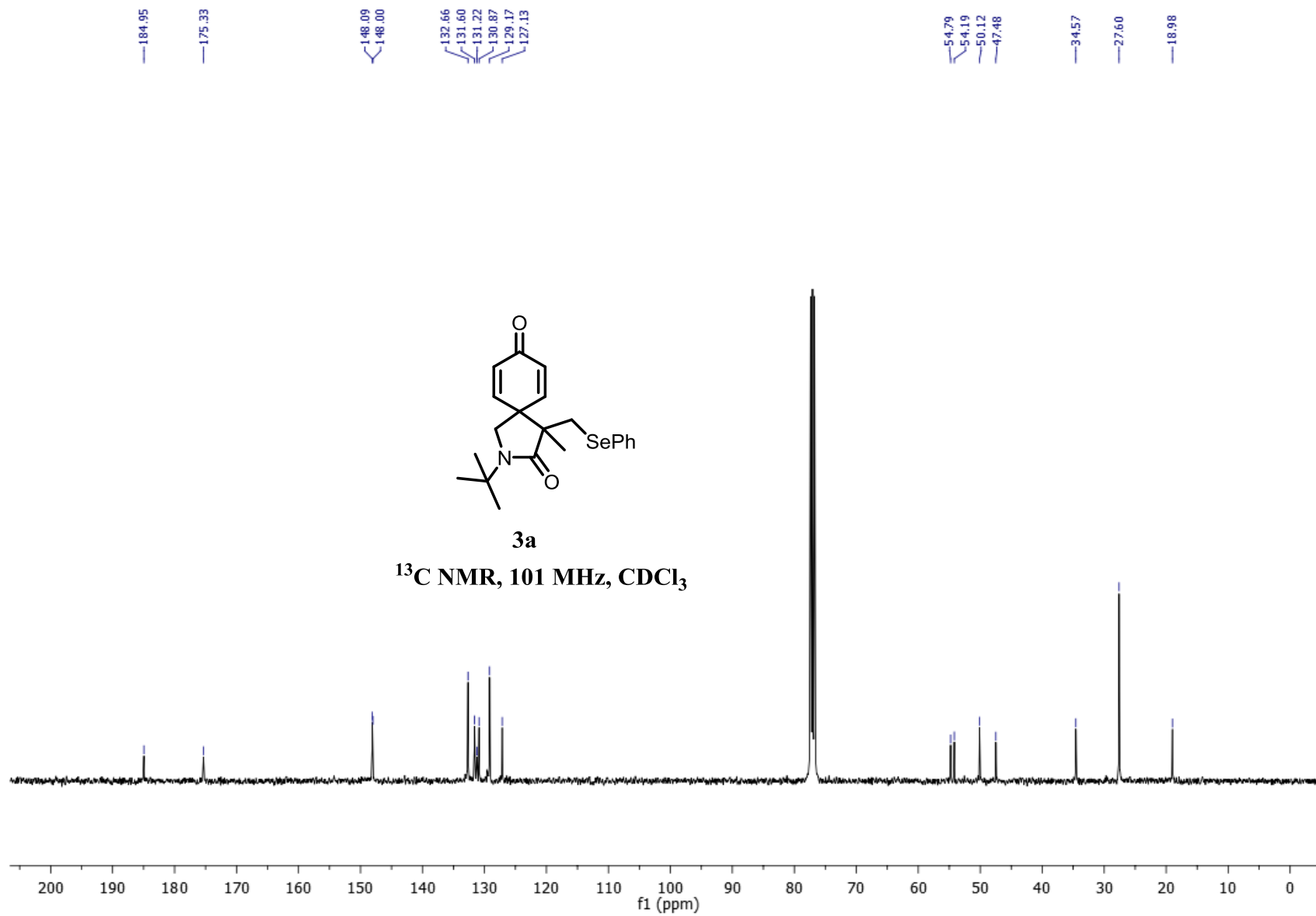


A 10 mL electro vial was charged with substrate (0.3 mmol, 1 equiv.), Diphenyl Diselenide ((PhSe)<sub>2</sub>) (0.225 mmol, 0.75 equiv.), sodium bromide (NaBr) (0.3 mmol, 1 equiv.), and TEMPO (0.6 mmol, 2 equiv.), CH<sub>3</sub>CN: TFE (4:1) (5mL) and a magnetic stir bar. The vial was equipped with carbon electrodes (8 x 52.5 x 2 mm) as the cathode and a carbon electrode (8 x 52.5 x 2 mm) as the anode. The whole cell was an undivided cell. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA at room temperature for 6 h (progress of the reaction was monitored by the TLC) and the reaction was found to be inhibited.

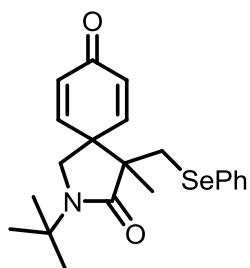






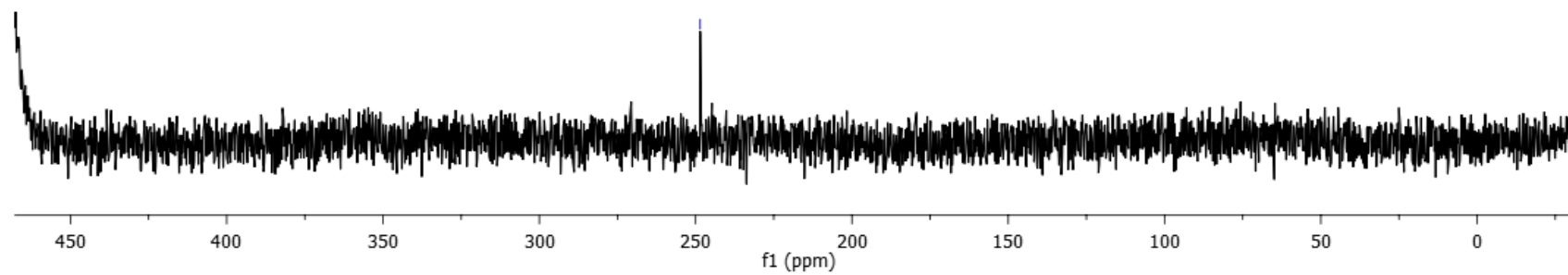


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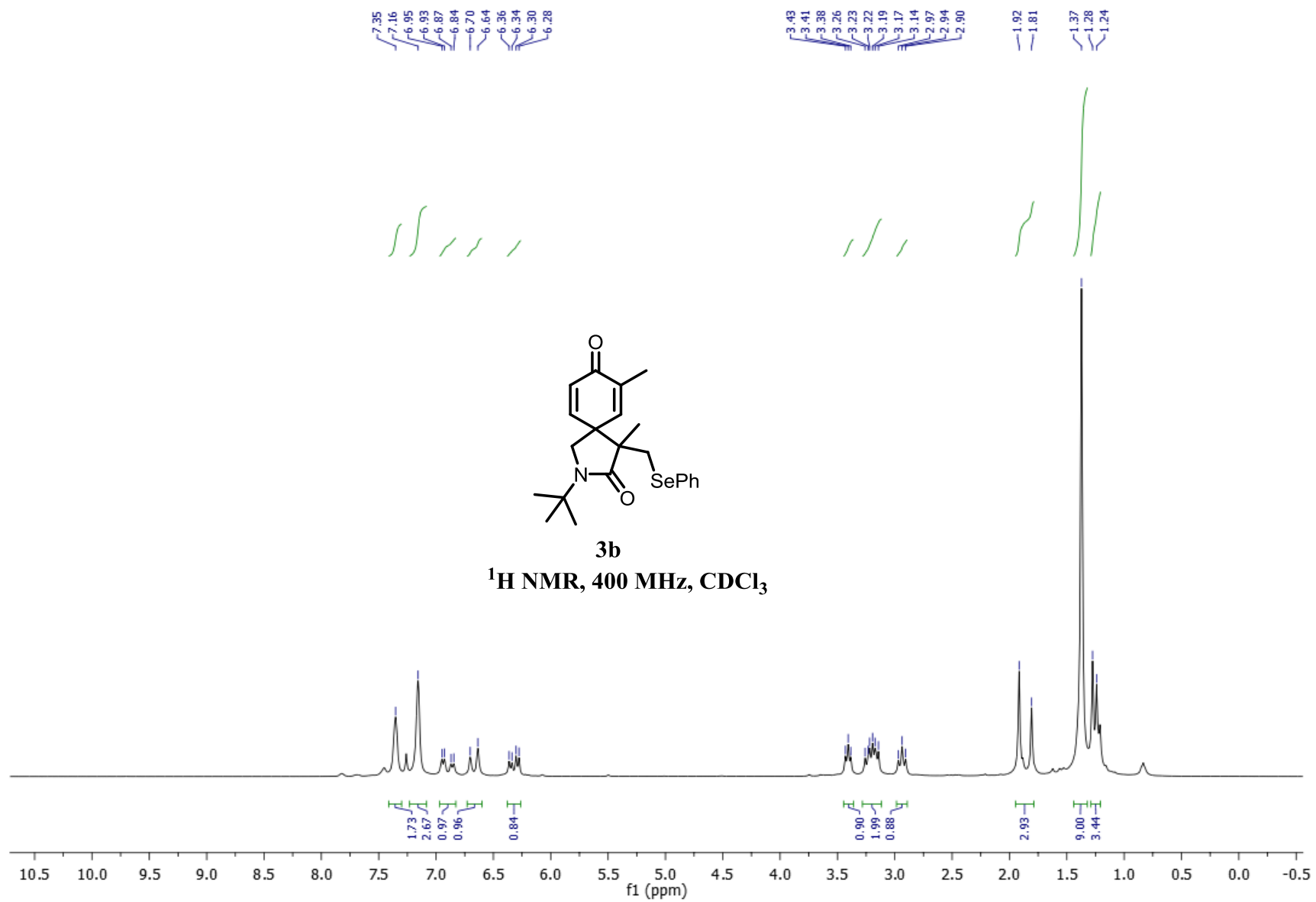


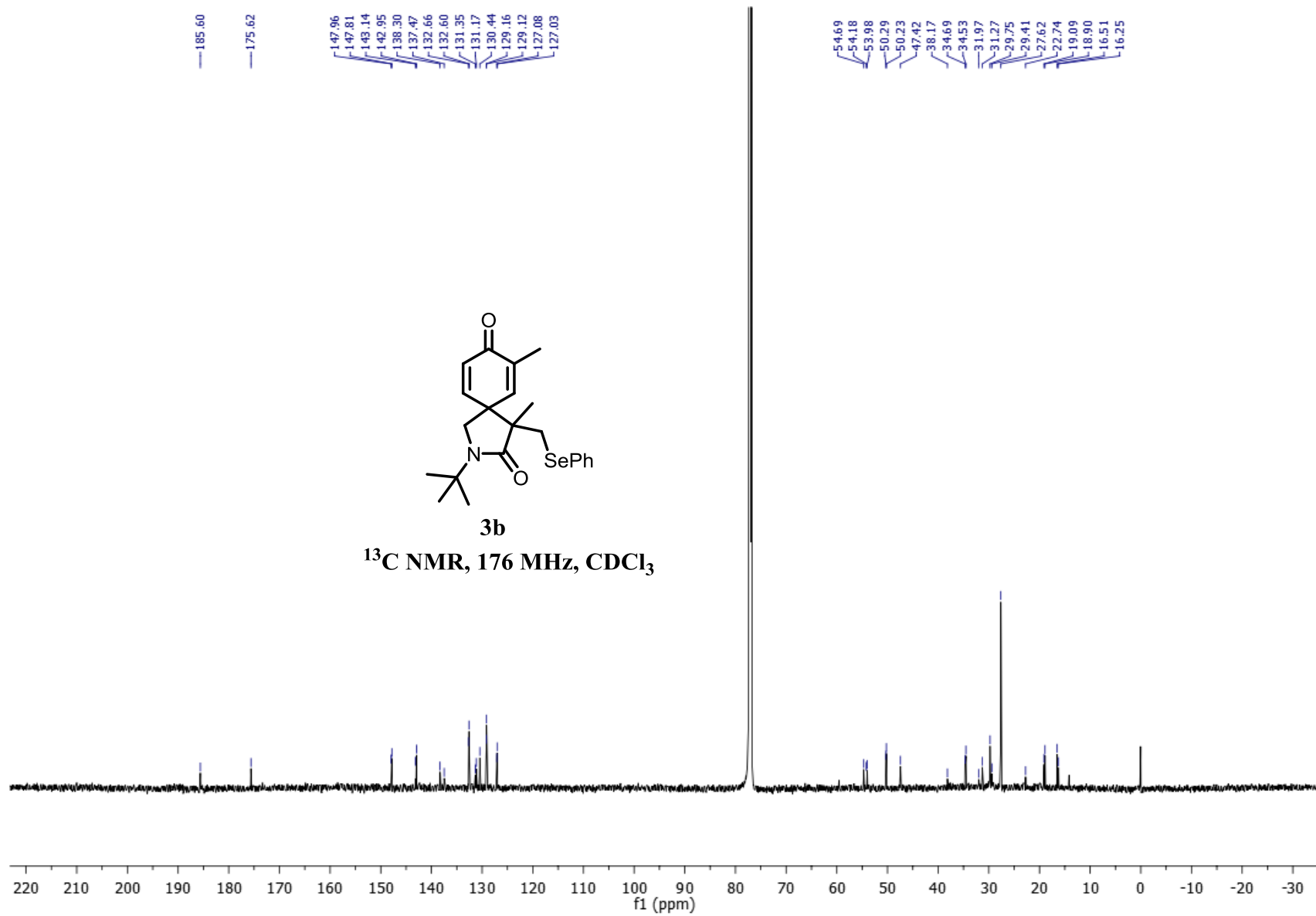
**3a**

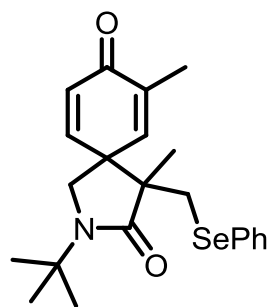
<sup>77</sup>Se NMR, 76 MHz, CDCl<sub>3</sub>





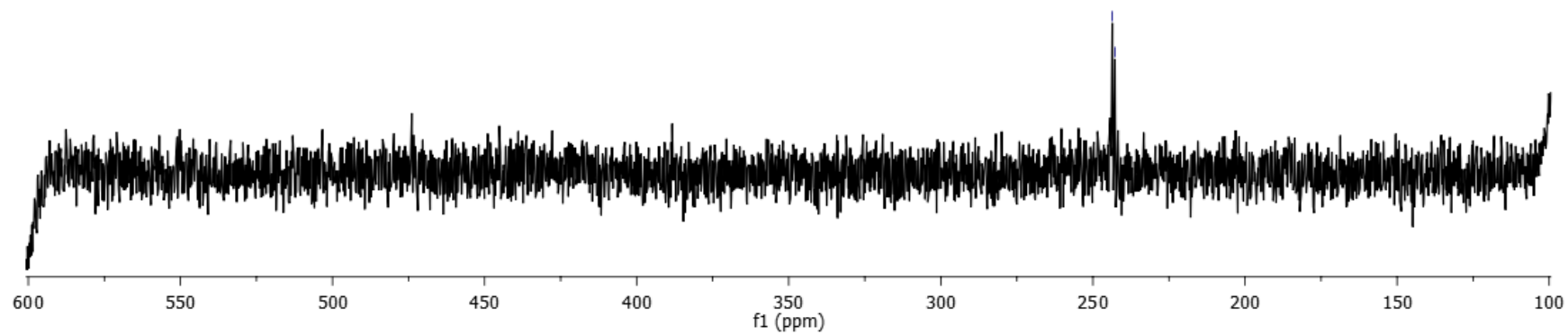


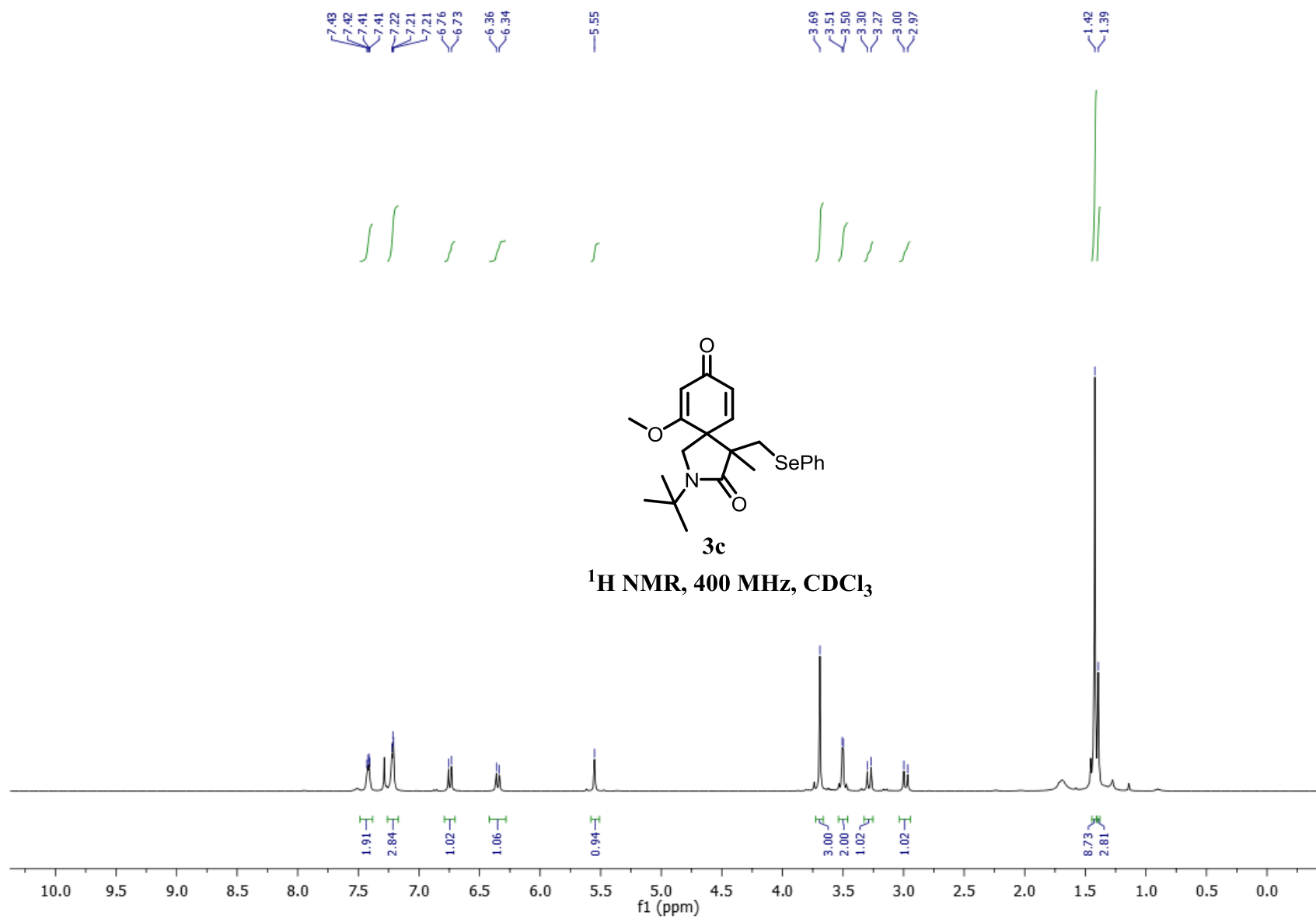


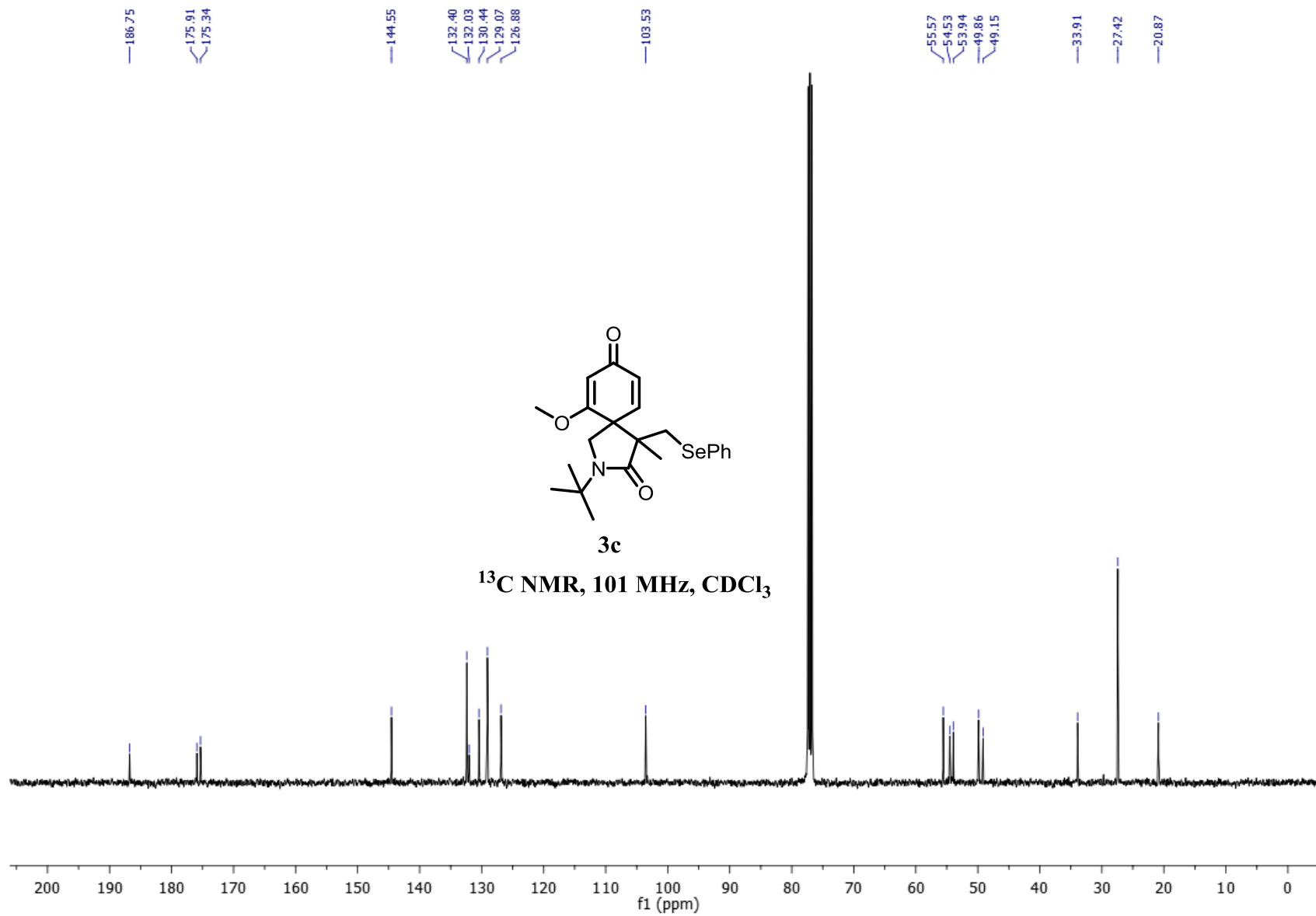


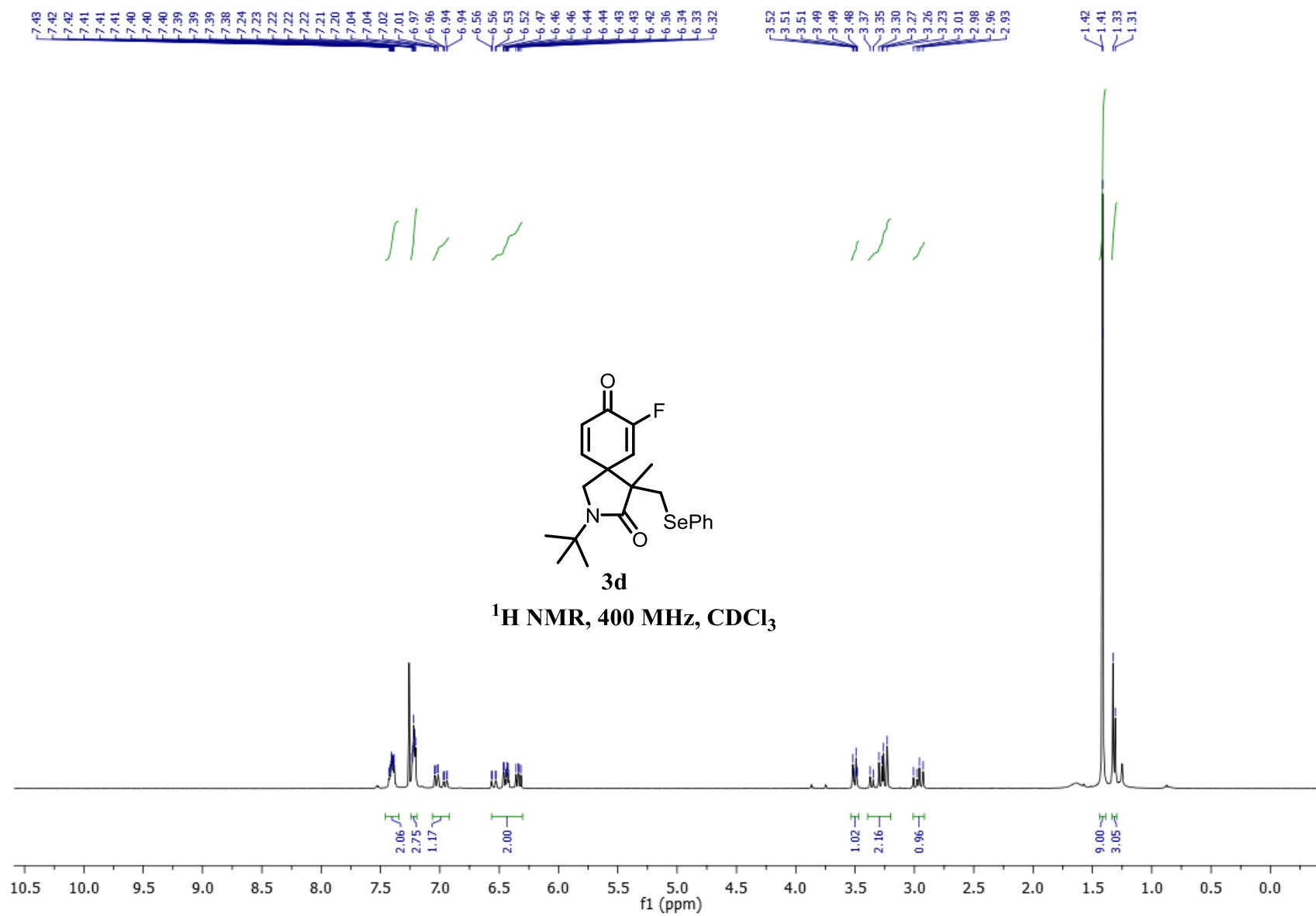
**3b**

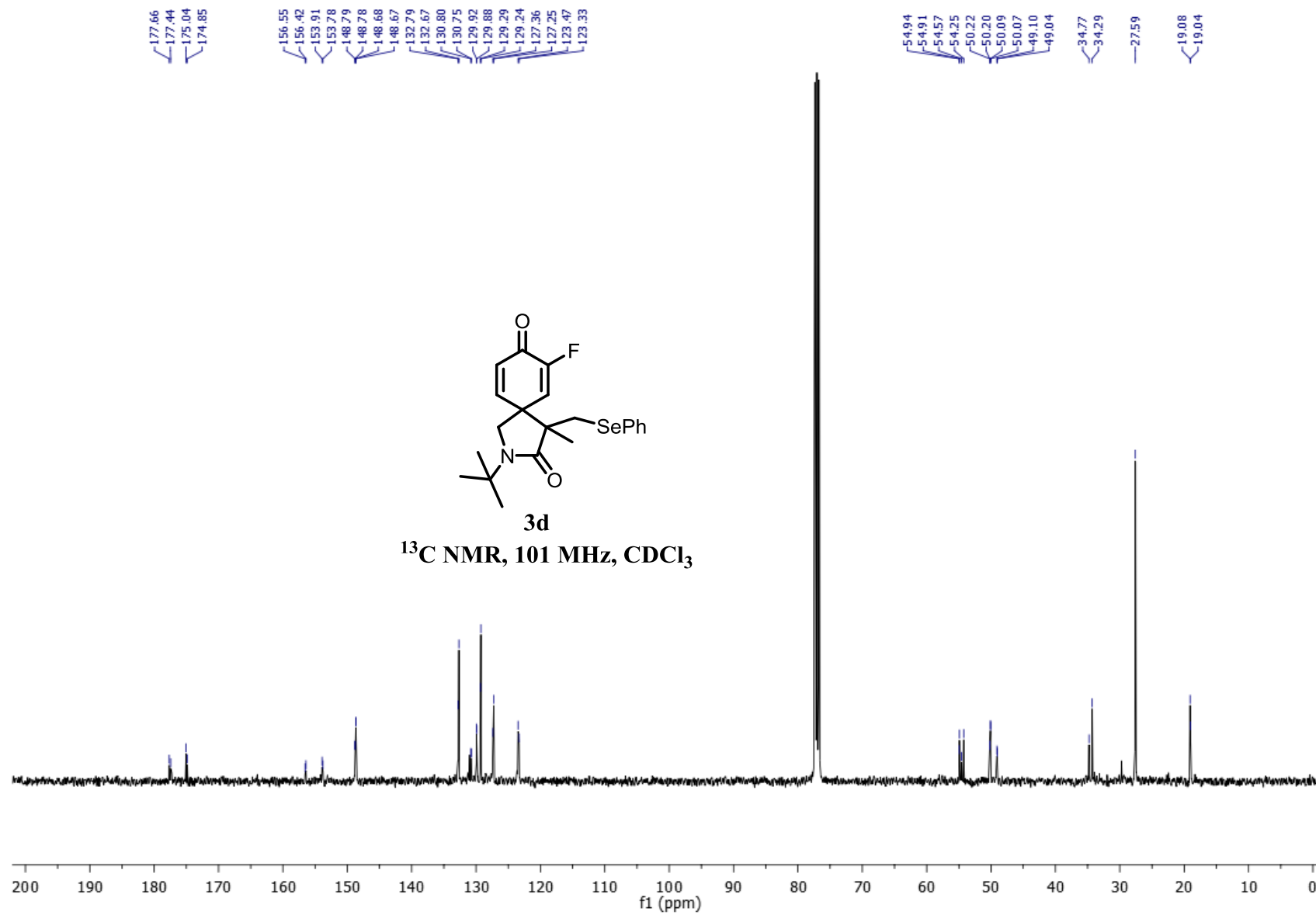
$^{77}\text{Se}$  NMR, 76 MHz,  $\text{CDCl}_3$

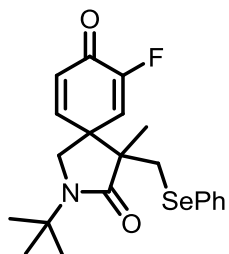






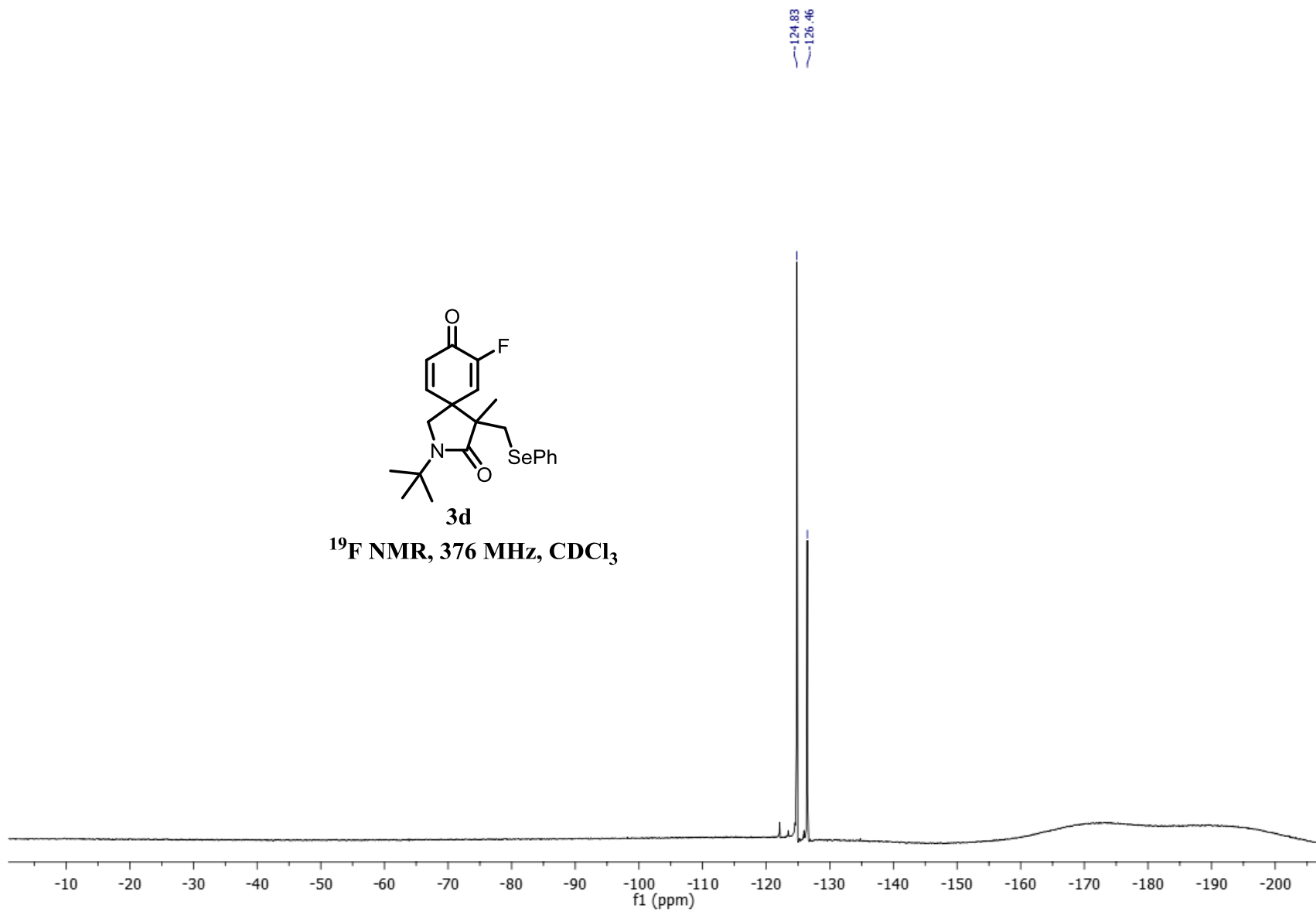




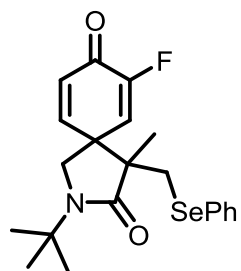


3d

<sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>

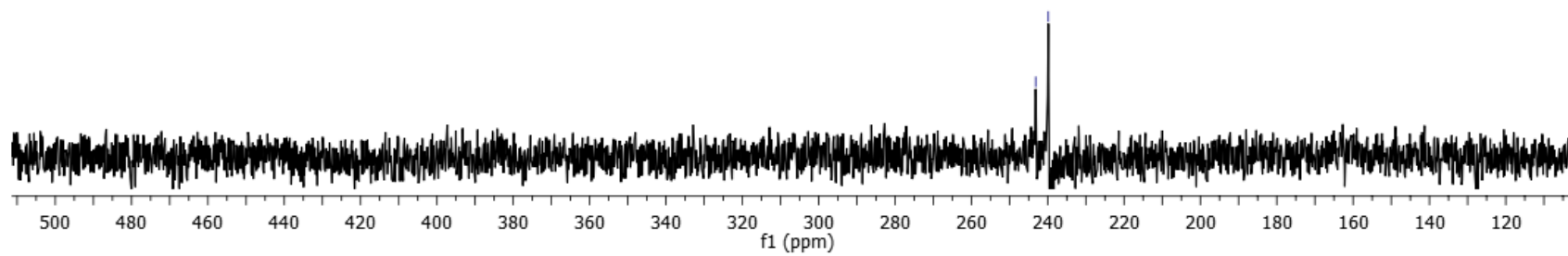


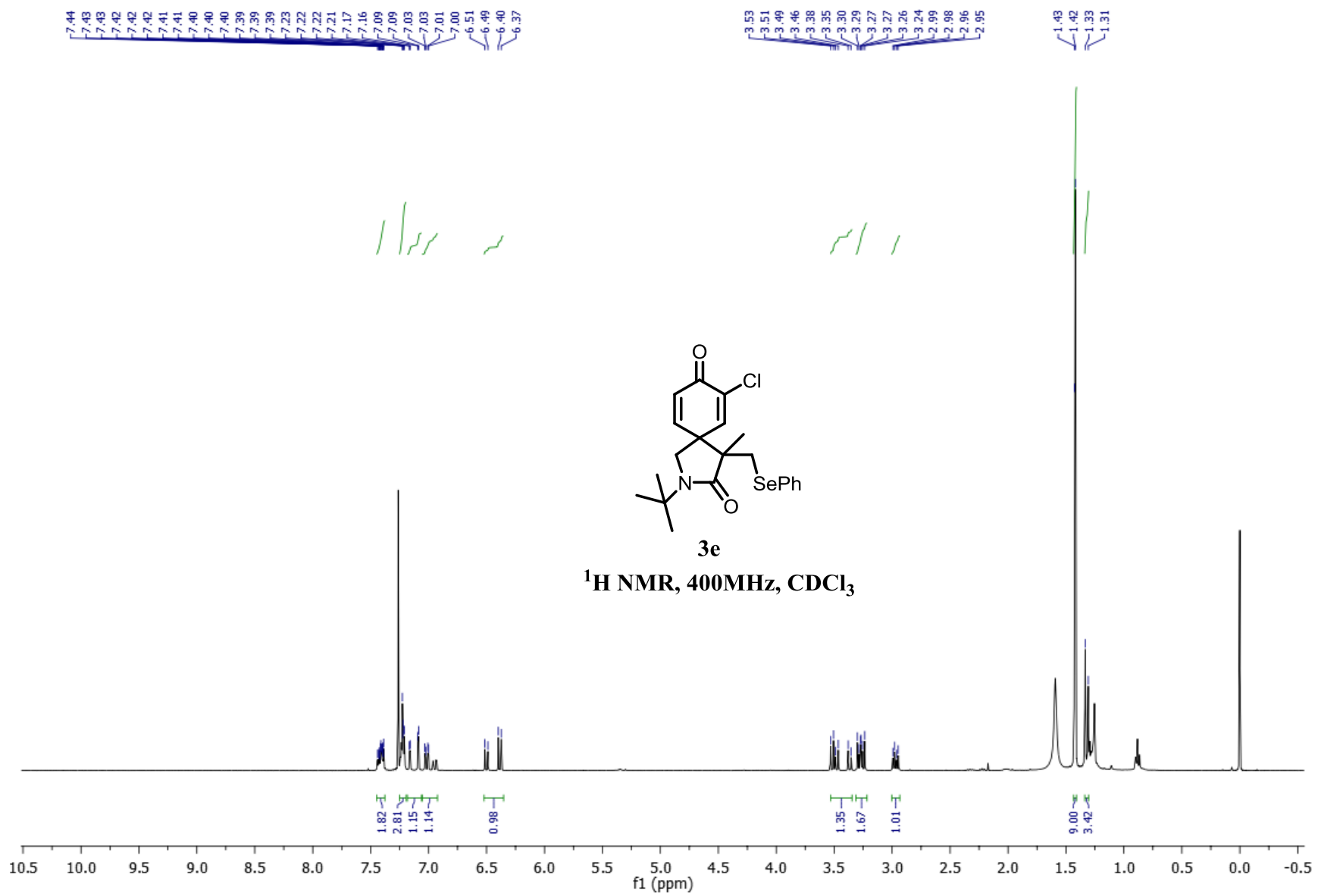


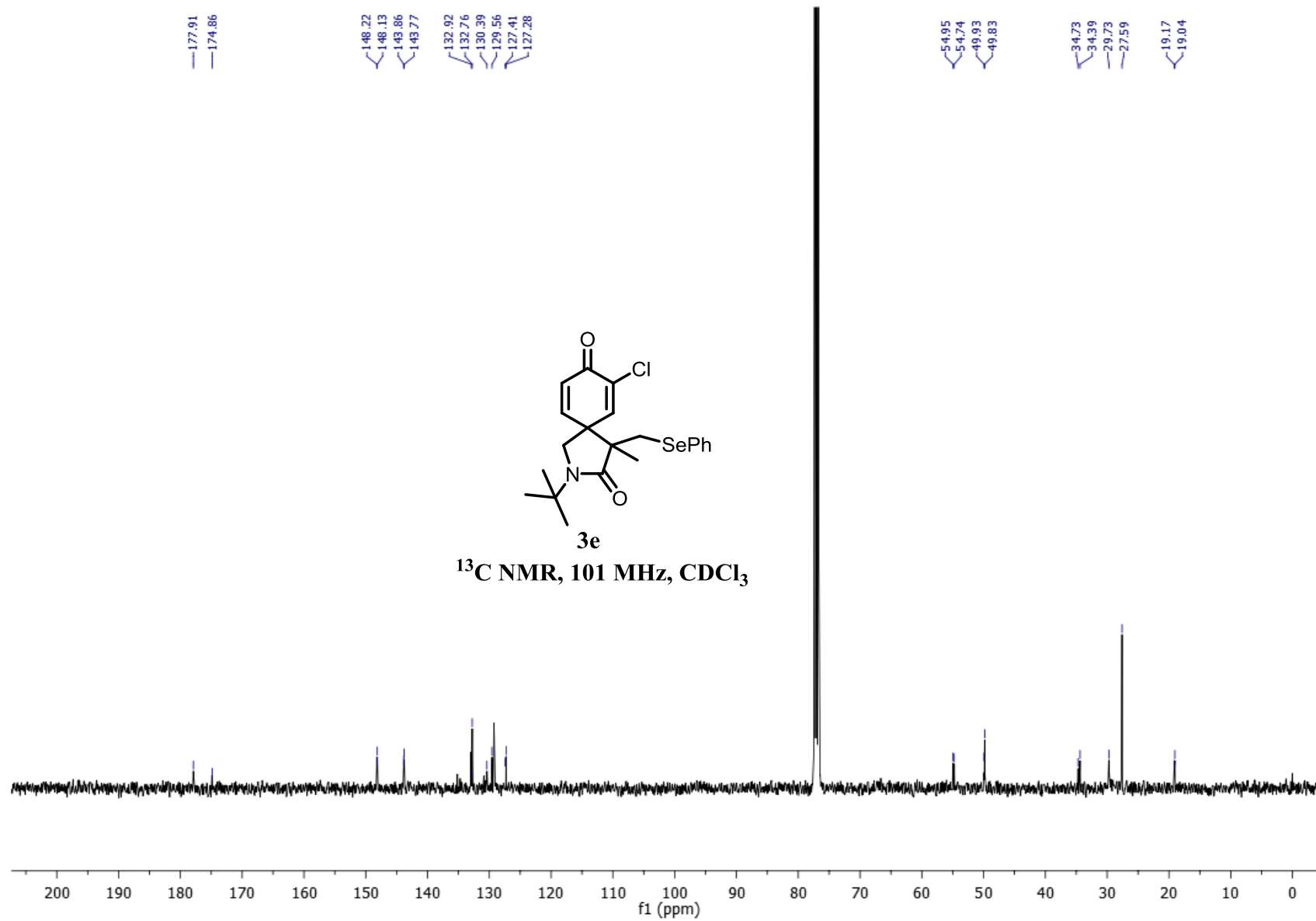


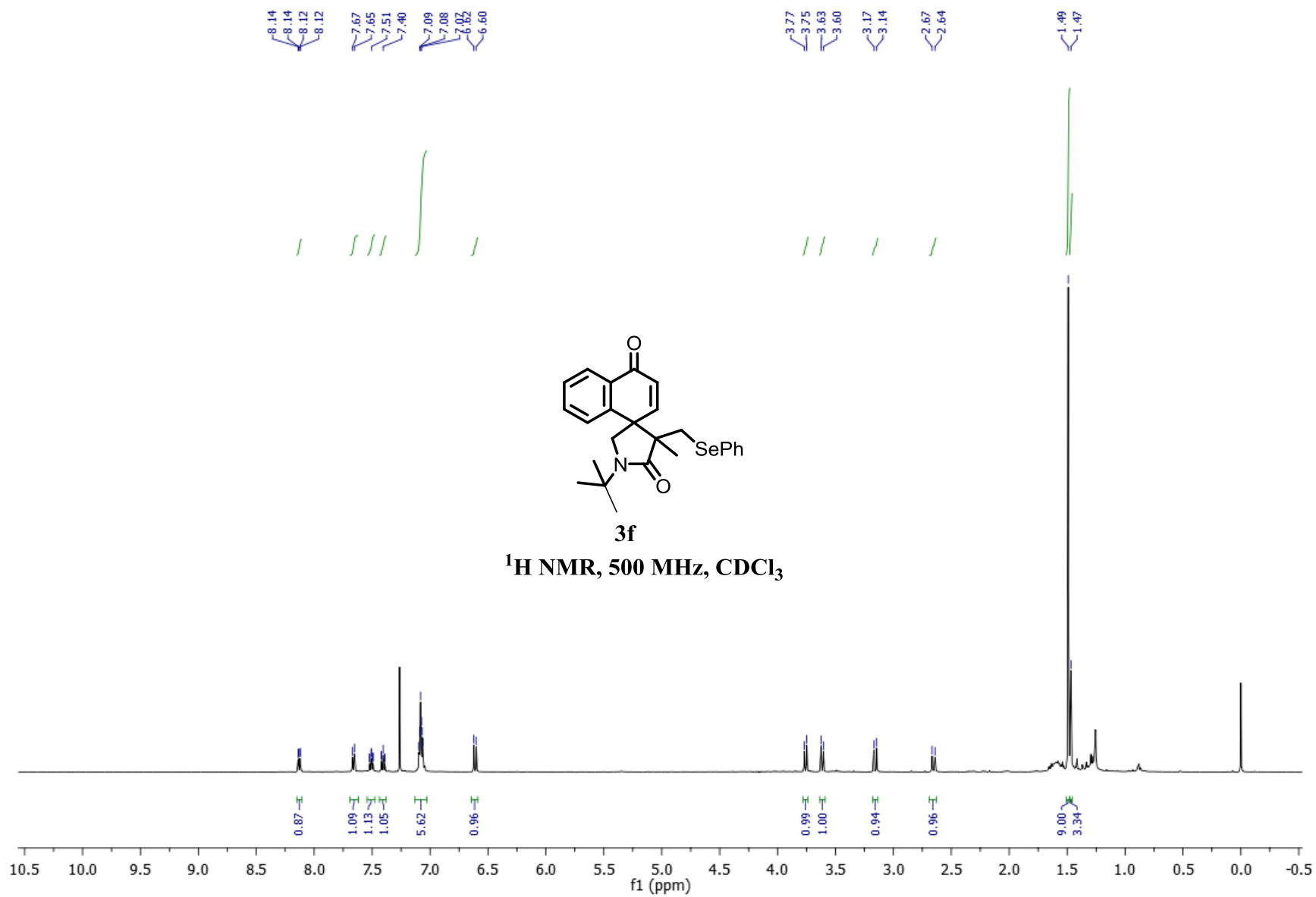
3d

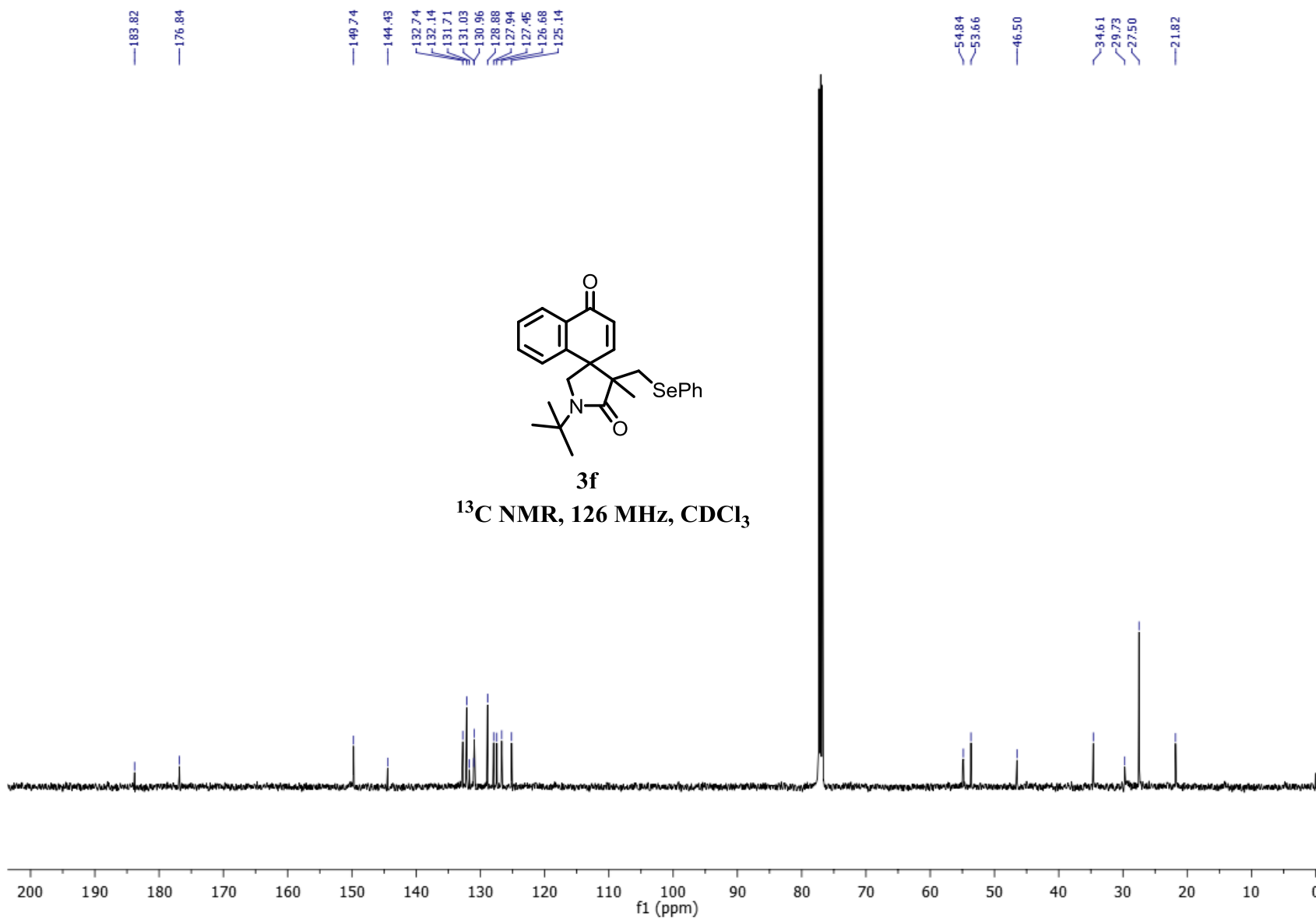
$^{77}\text{Se}$  NMR, 76 MHz,  $\text{CDCl}_3$

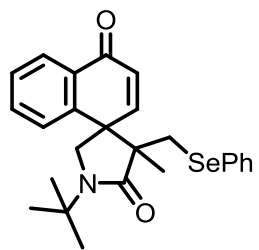






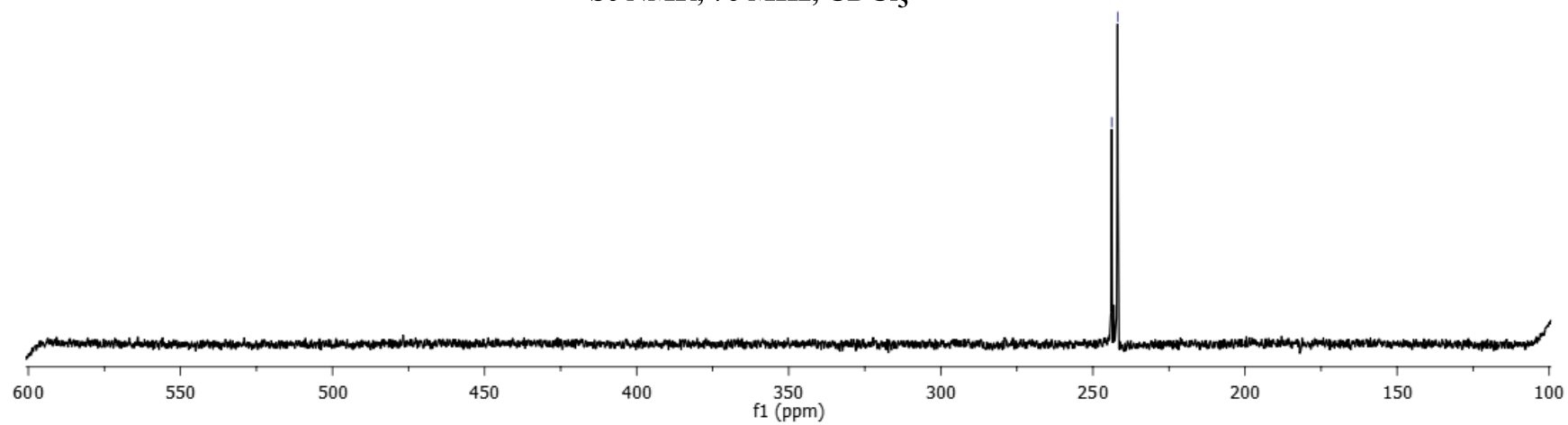


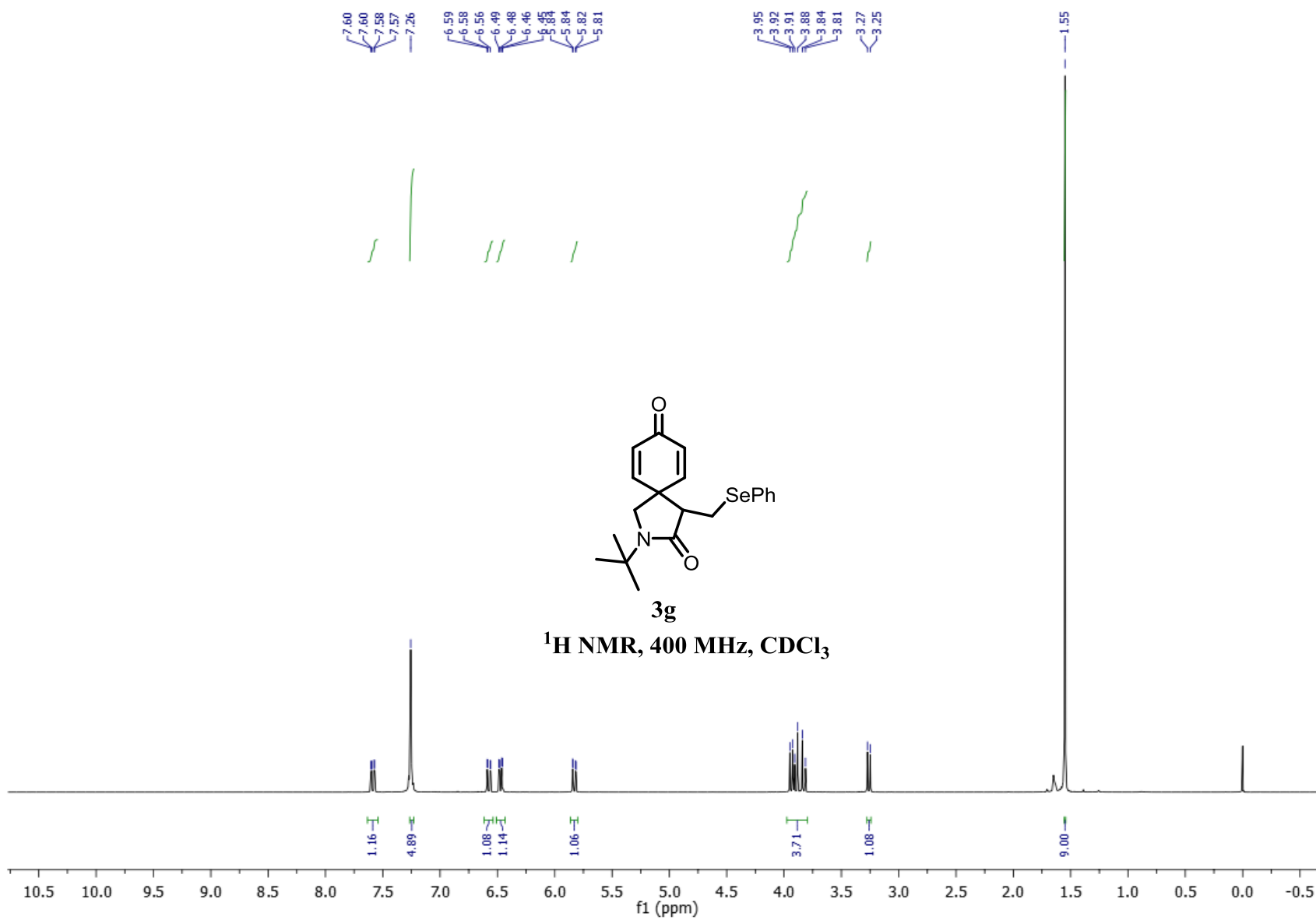


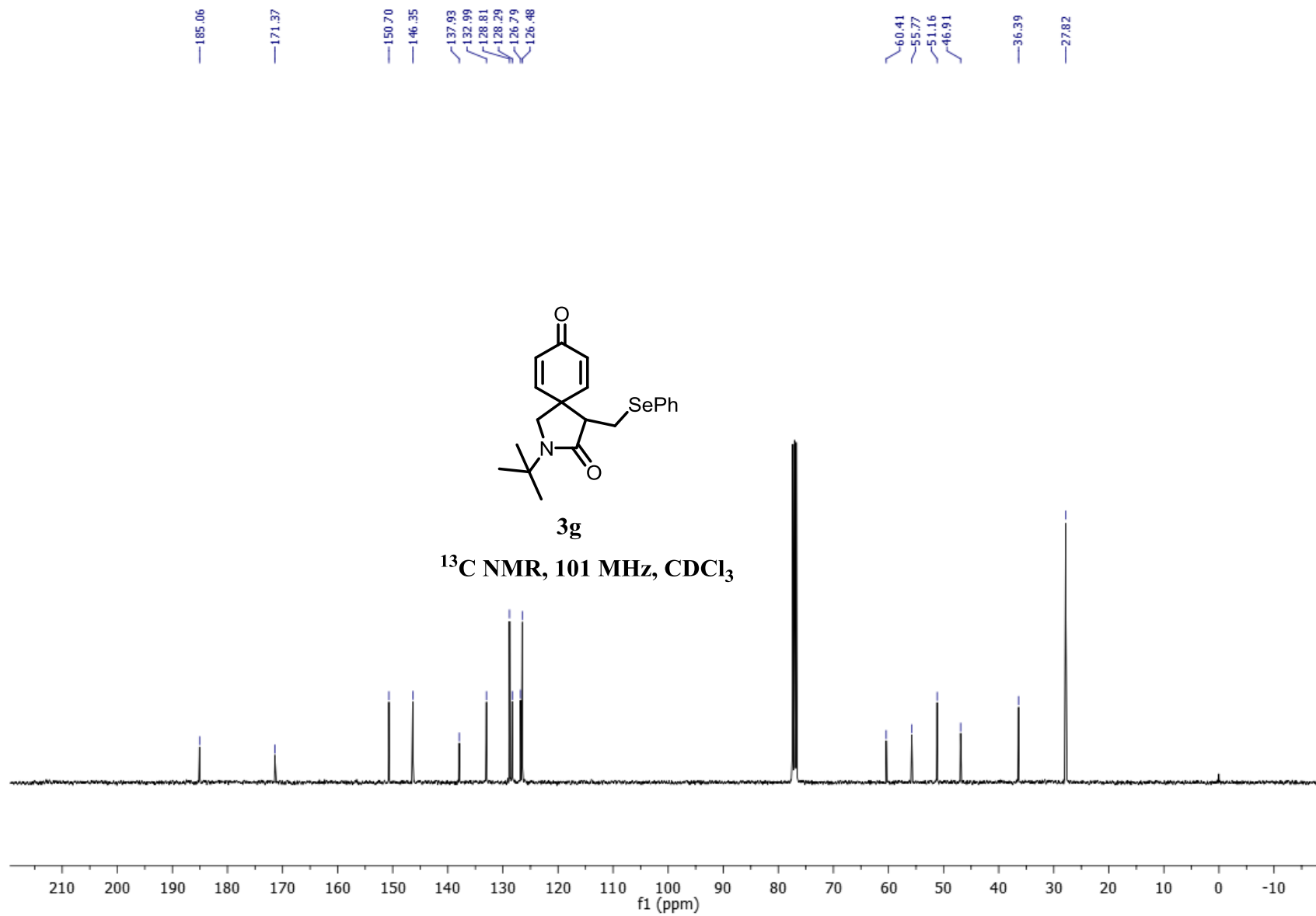


**3f**

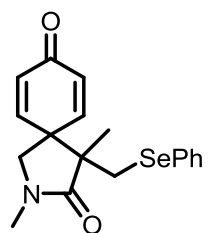
<sup>77</sup>Se NMR, 76 MHz, CDCl<sub>3</sub>





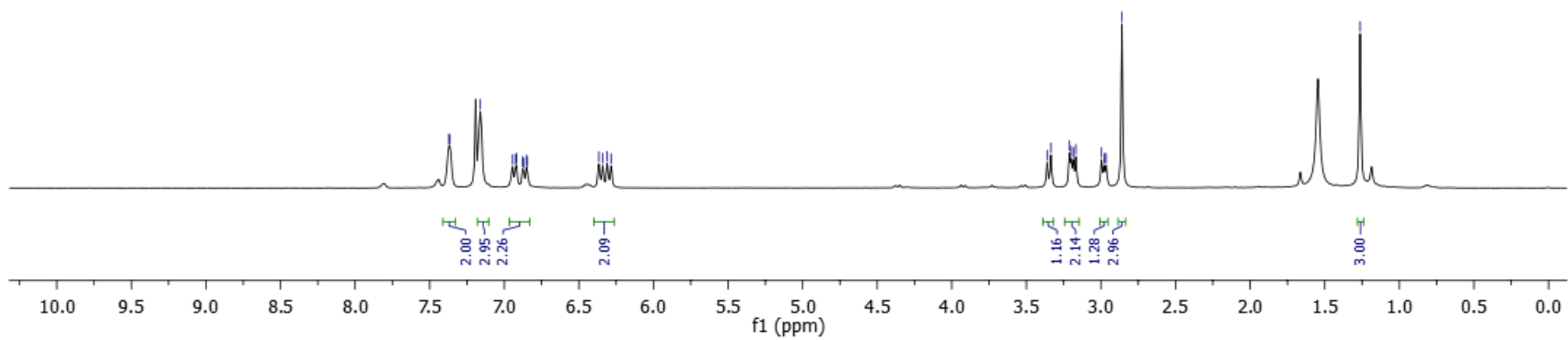


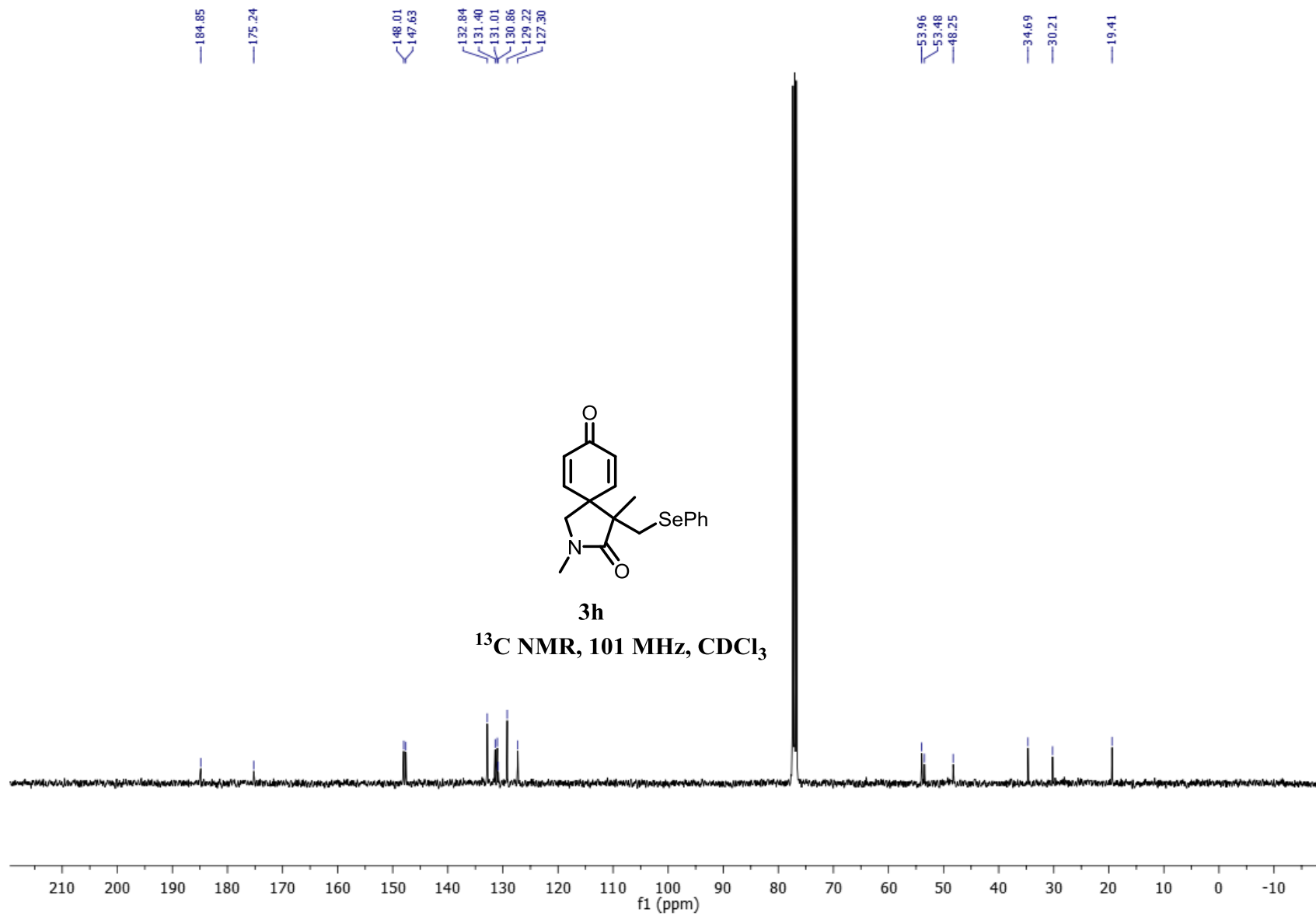


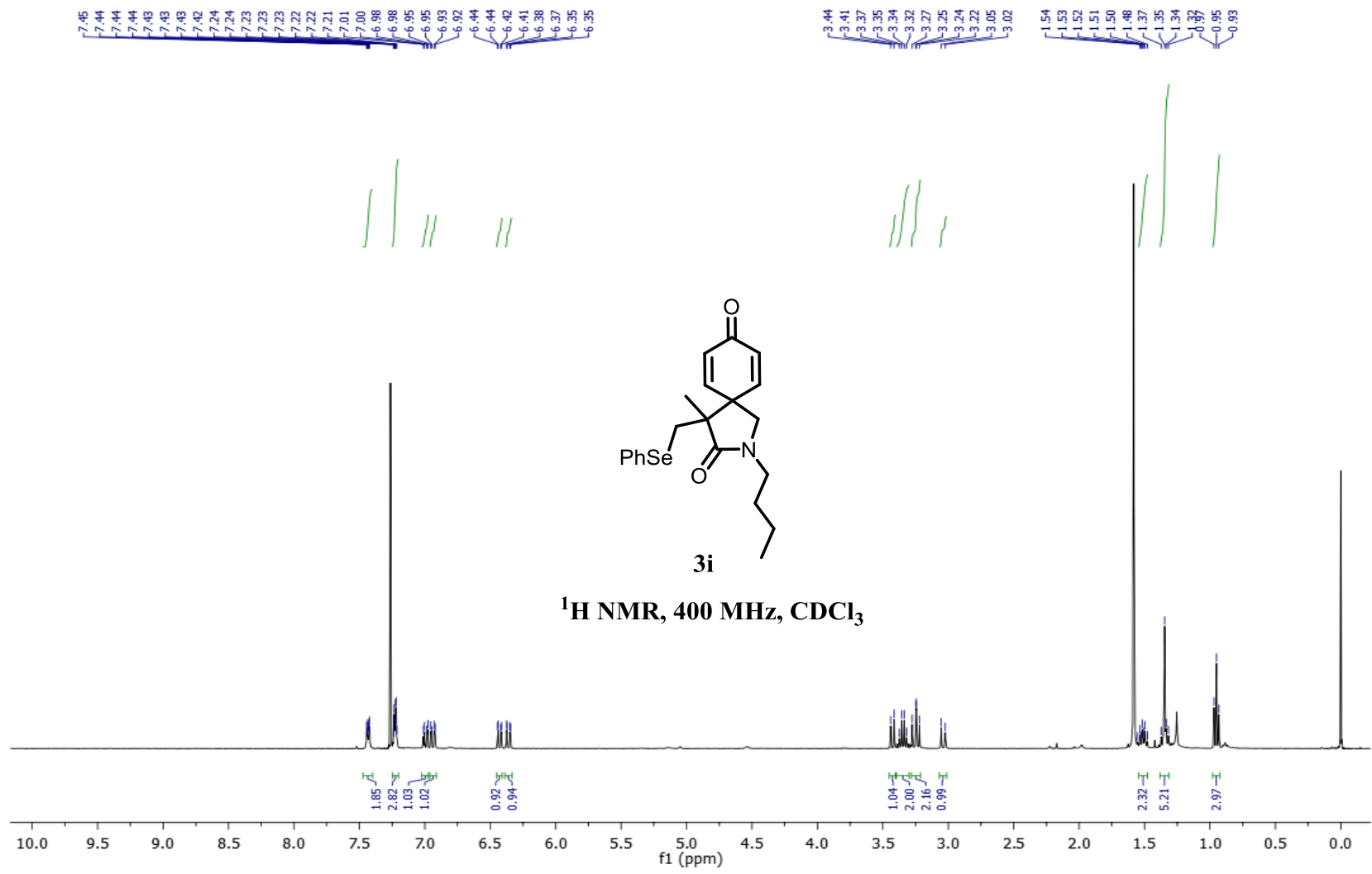


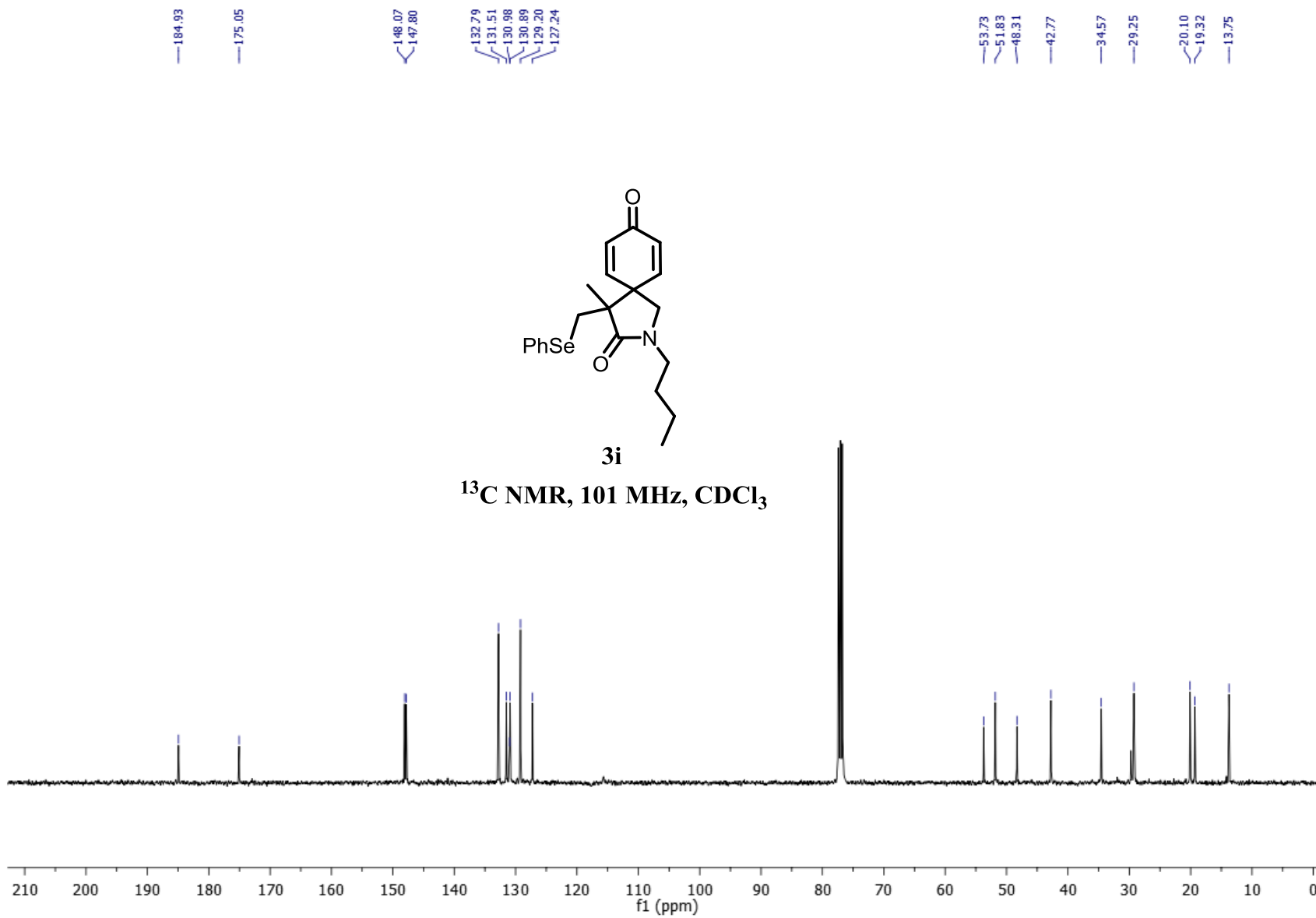
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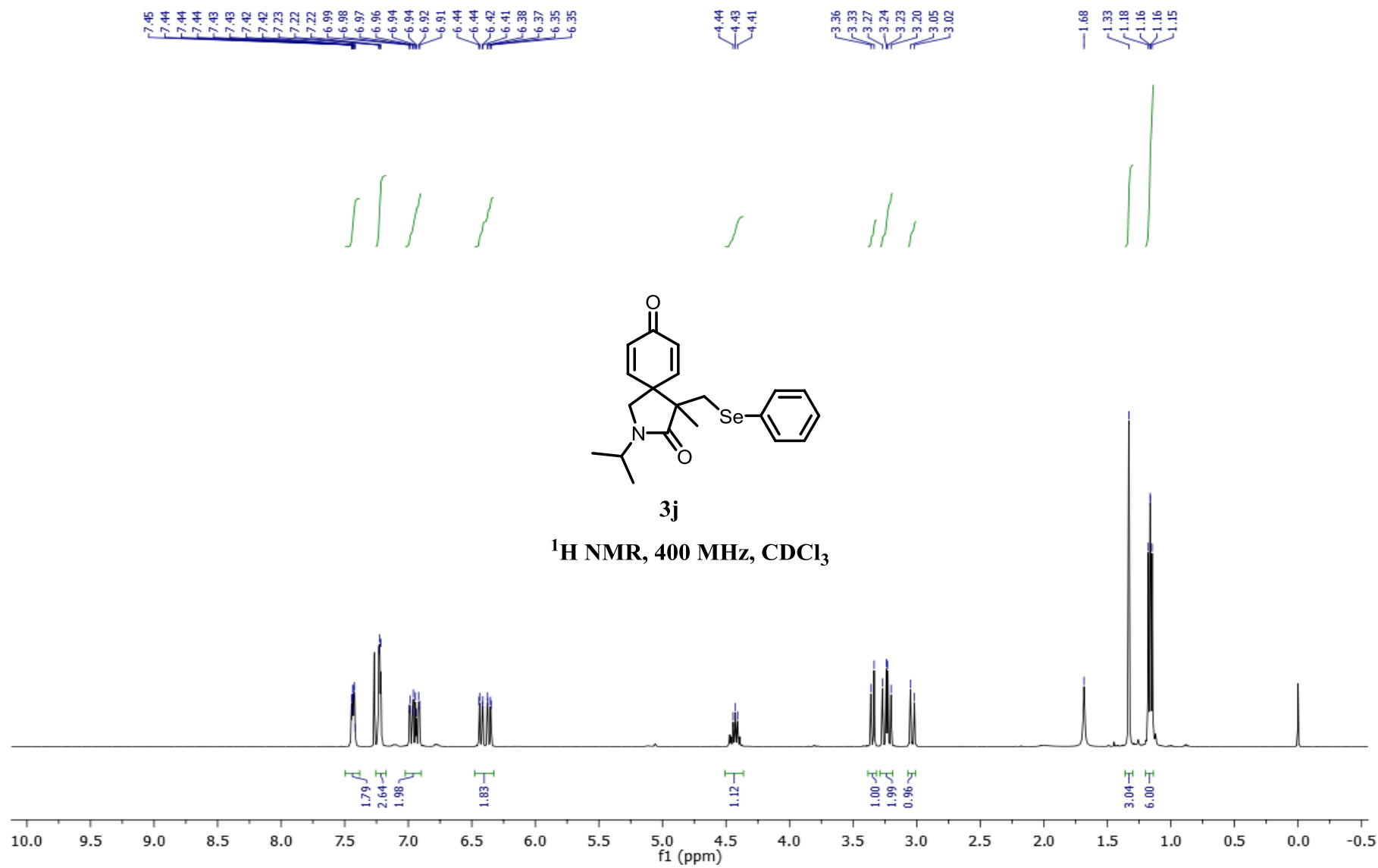
**$^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$**

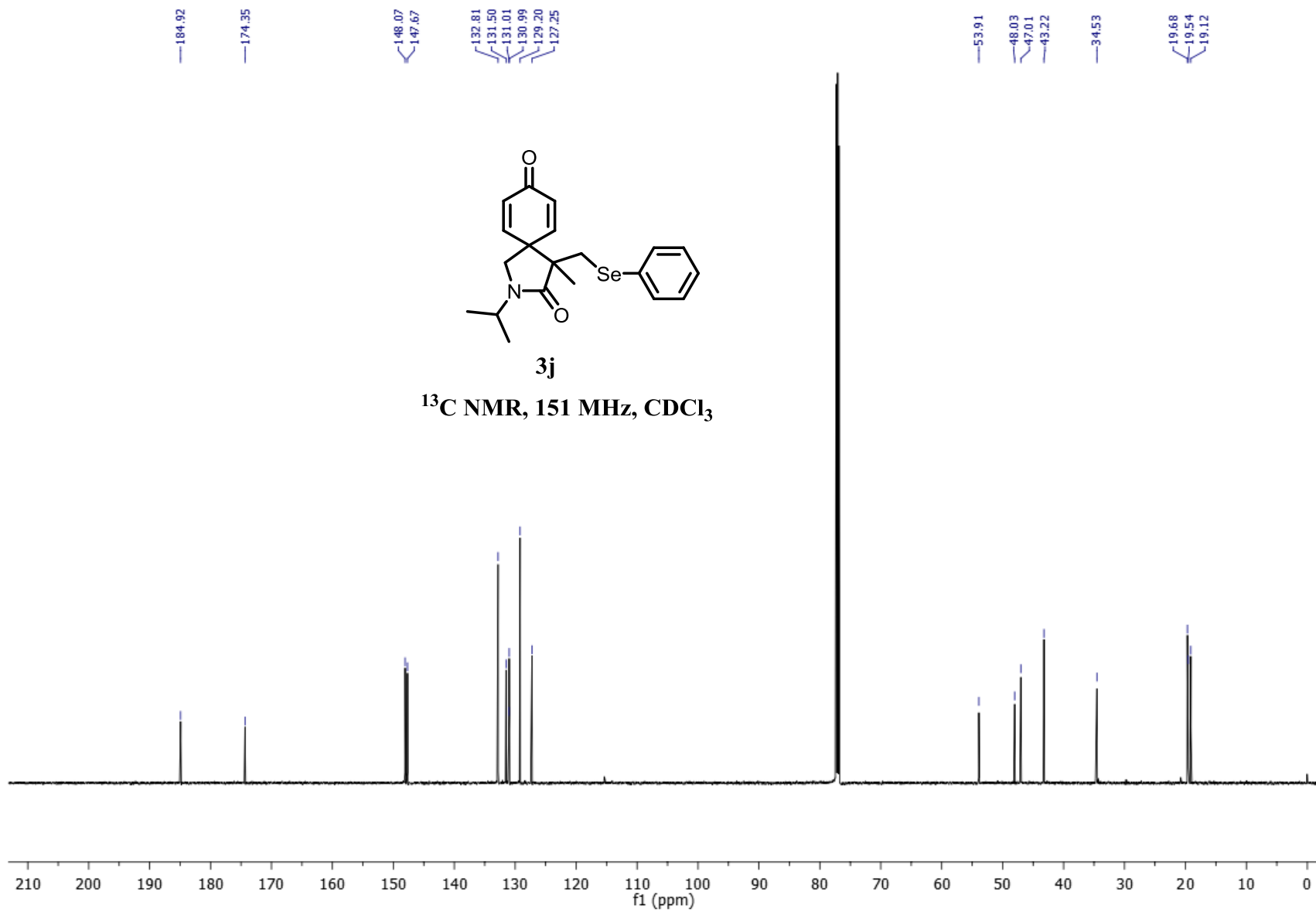


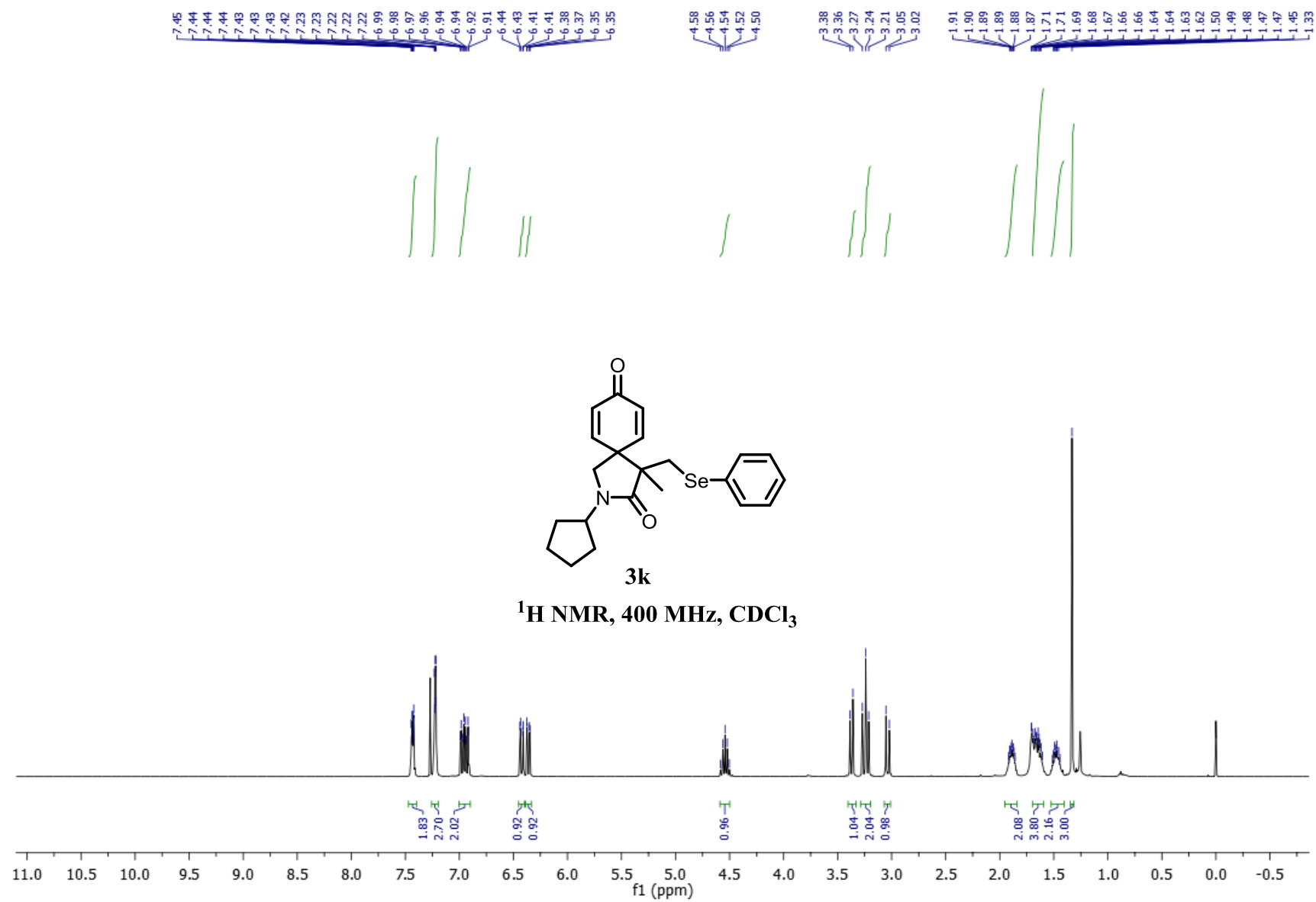


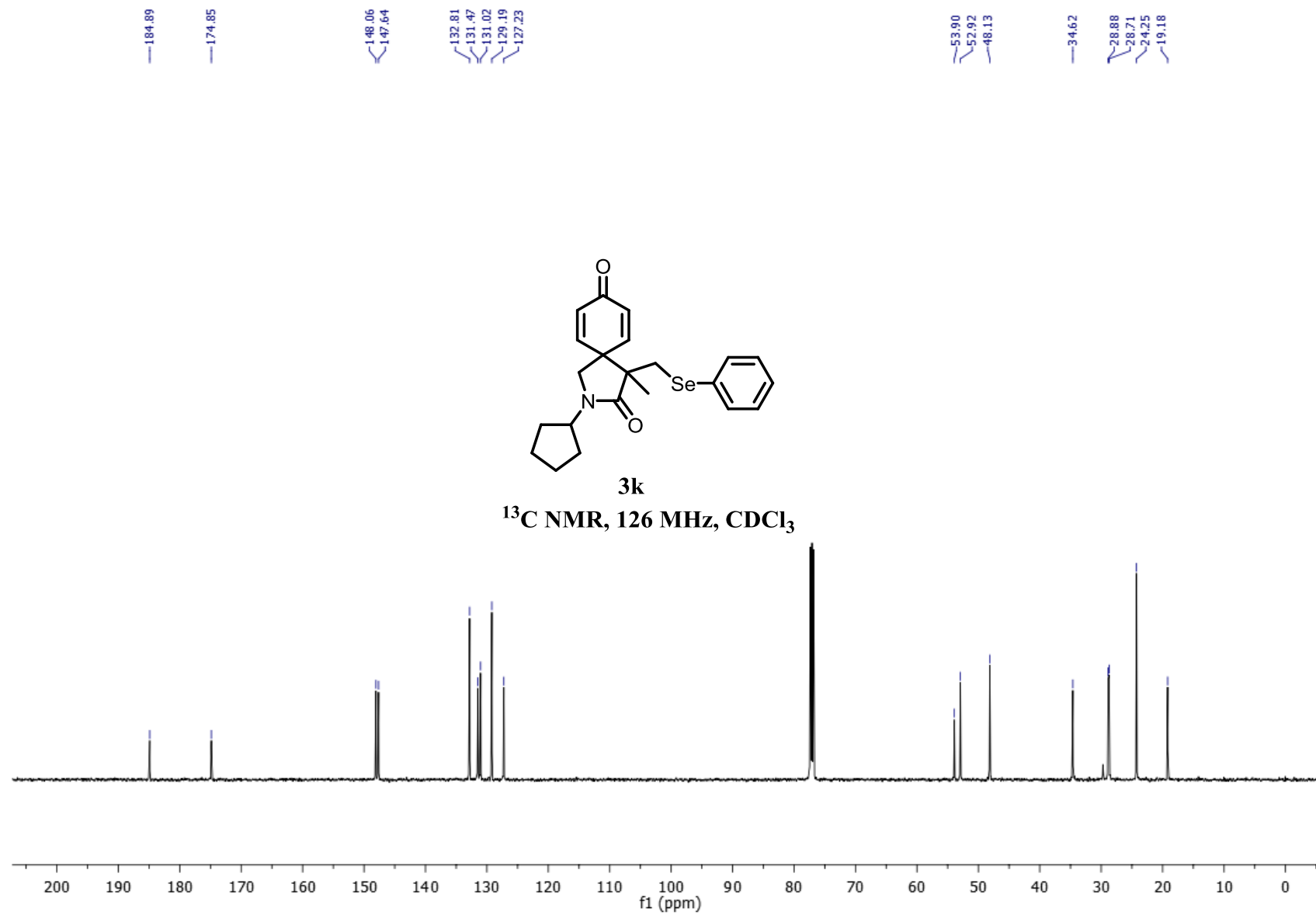






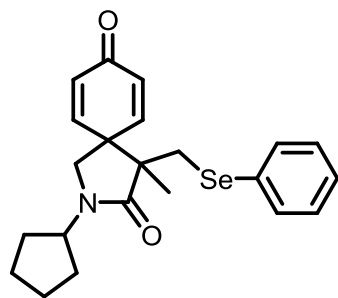






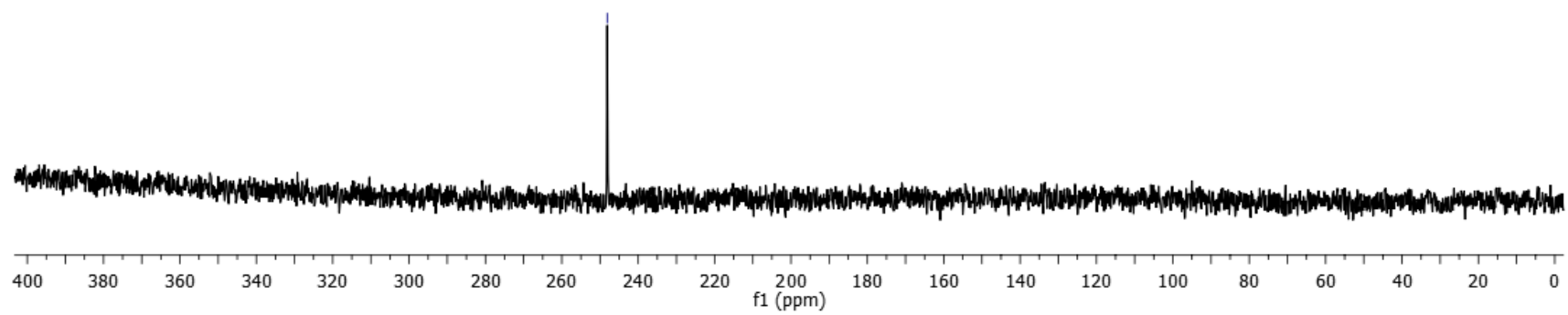


—248.12

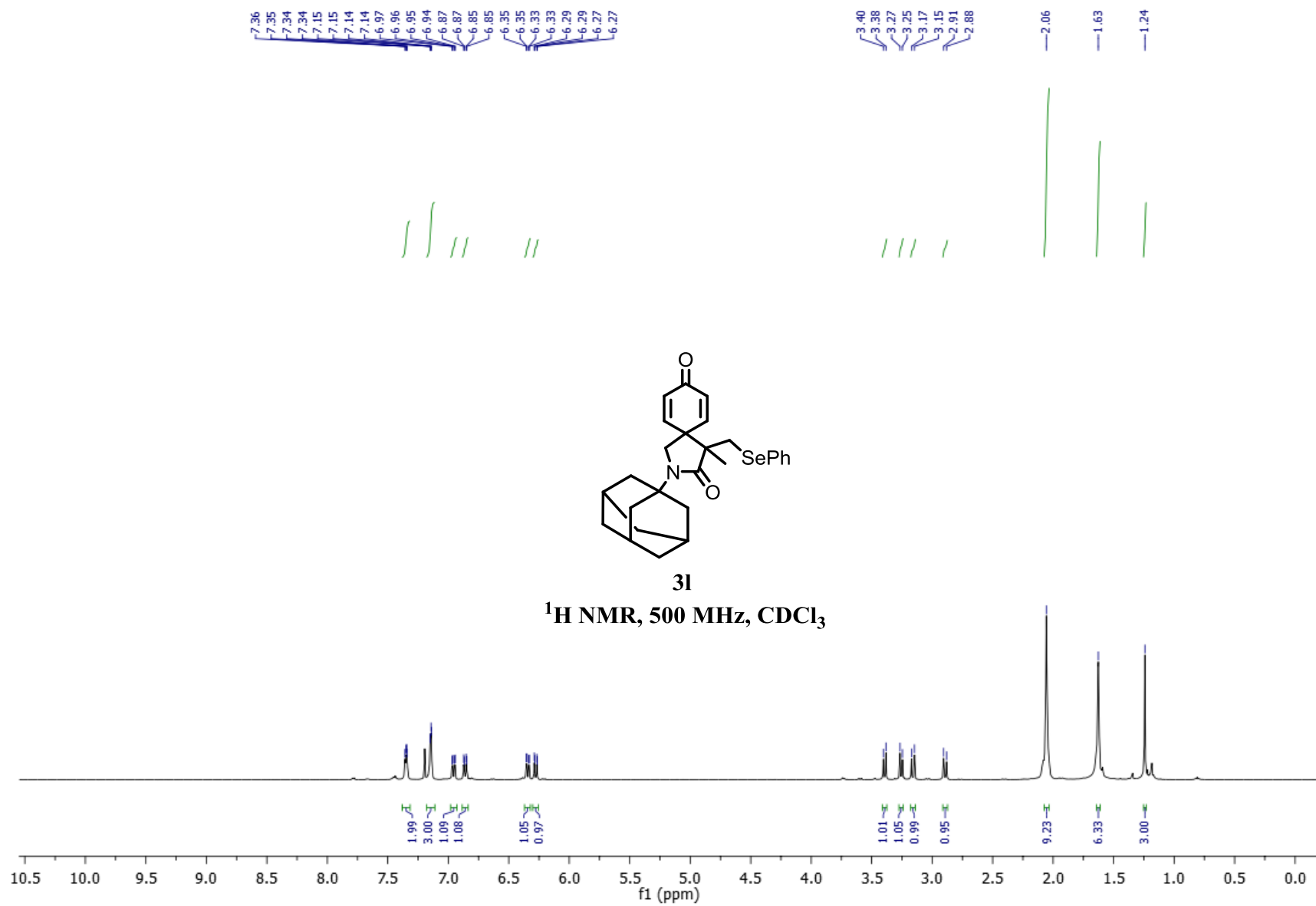


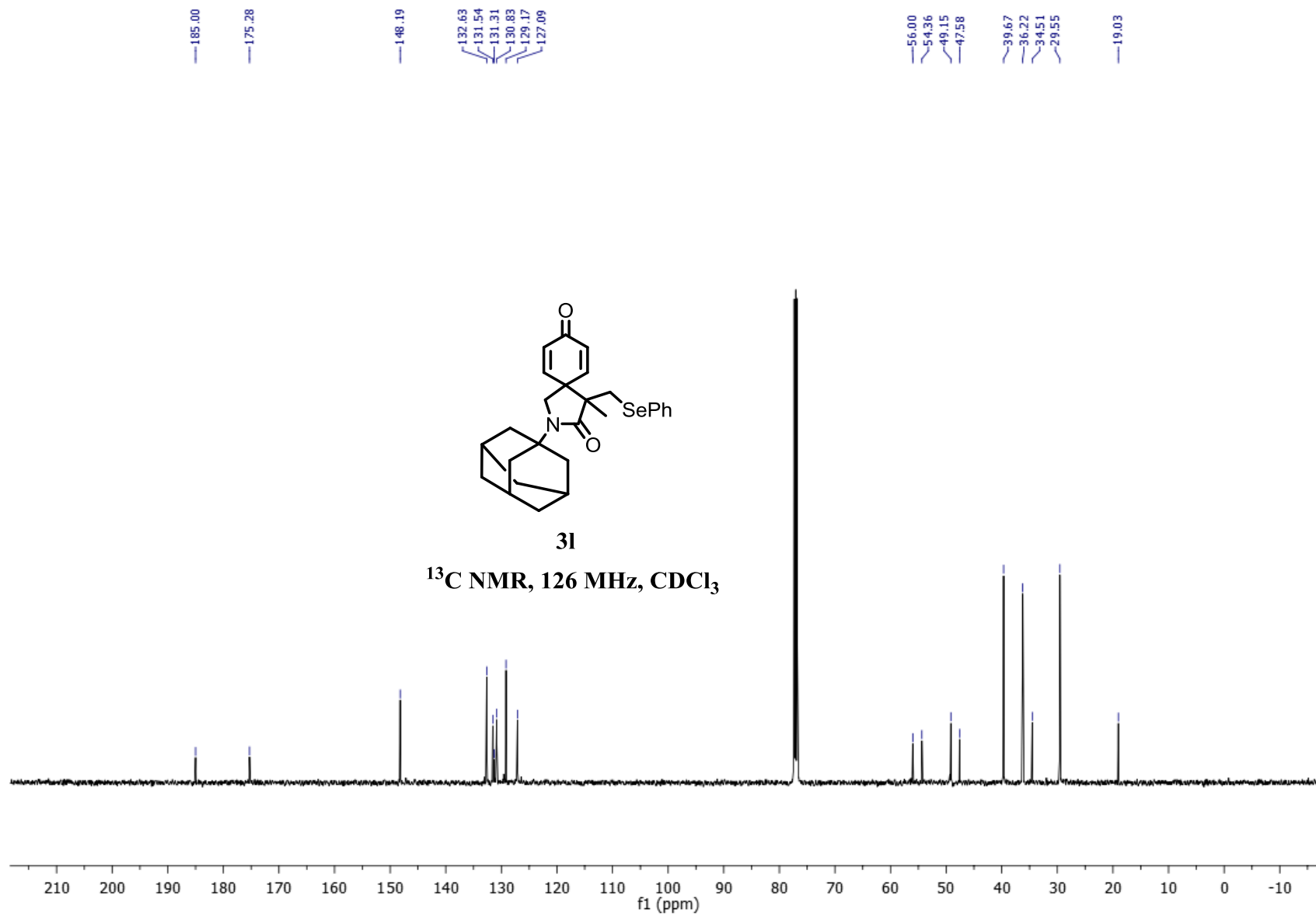
**3k**

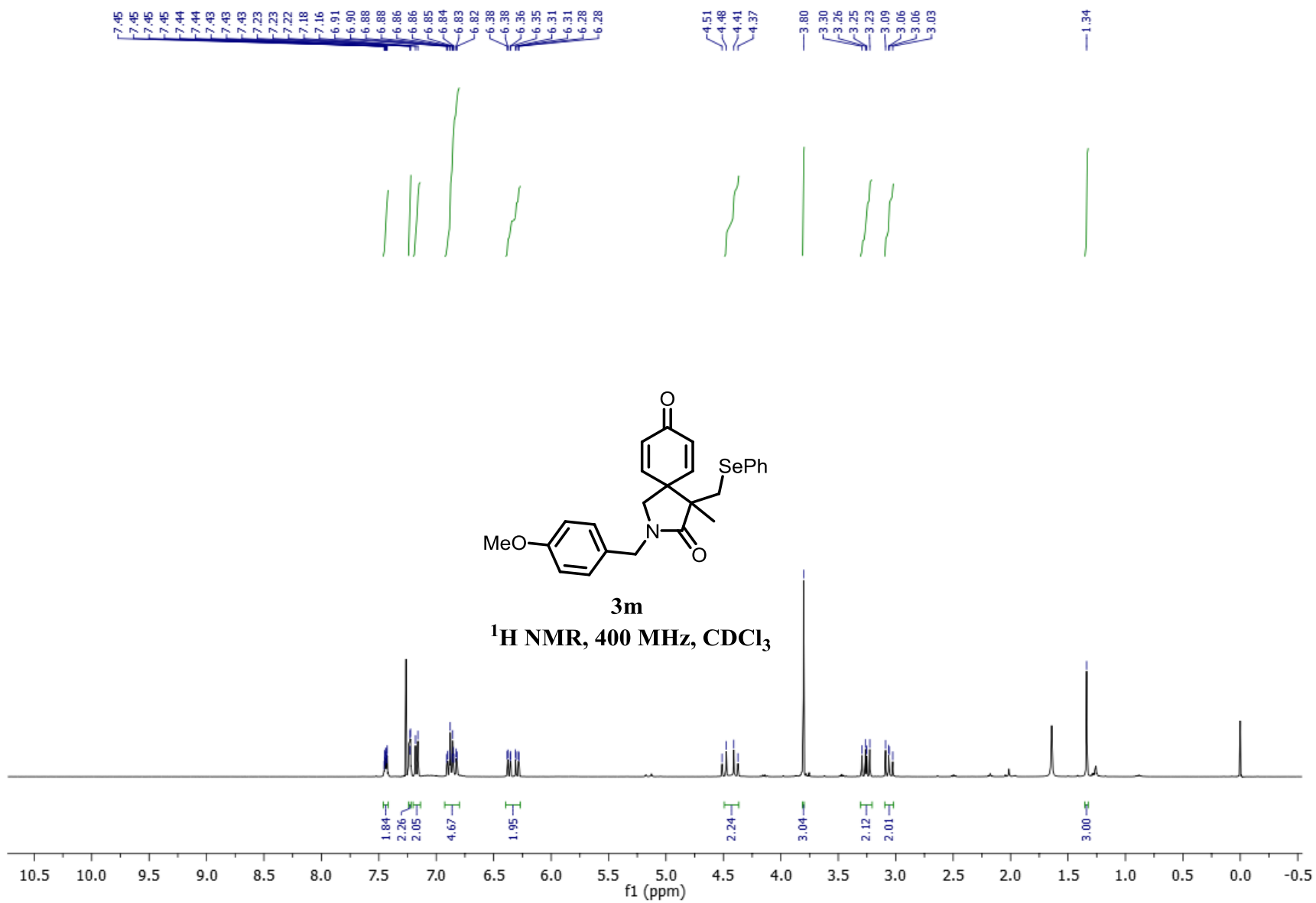
<sup>77</sup>Se NMR, 76 MHz, CDCl<sub>3</sub>

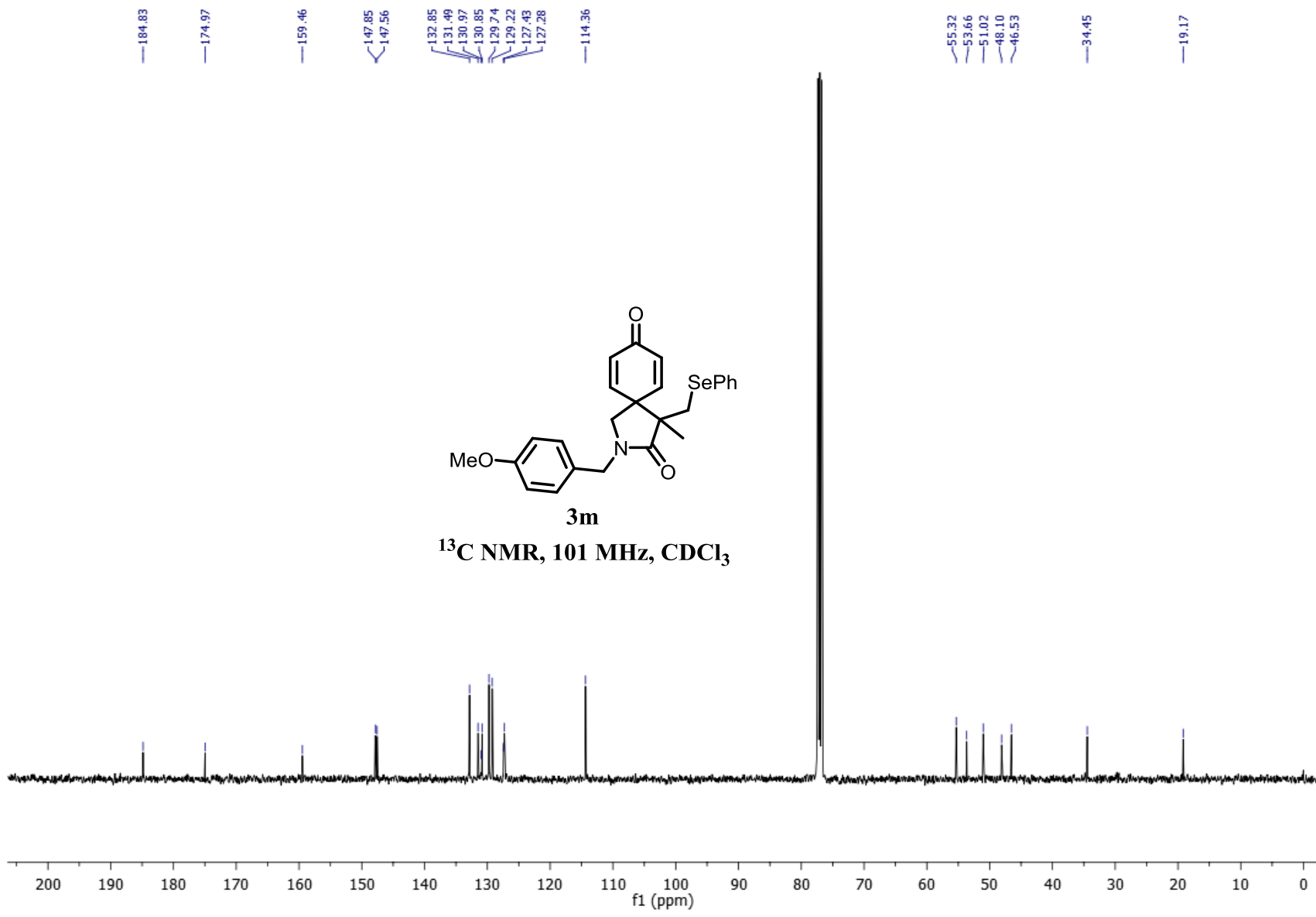


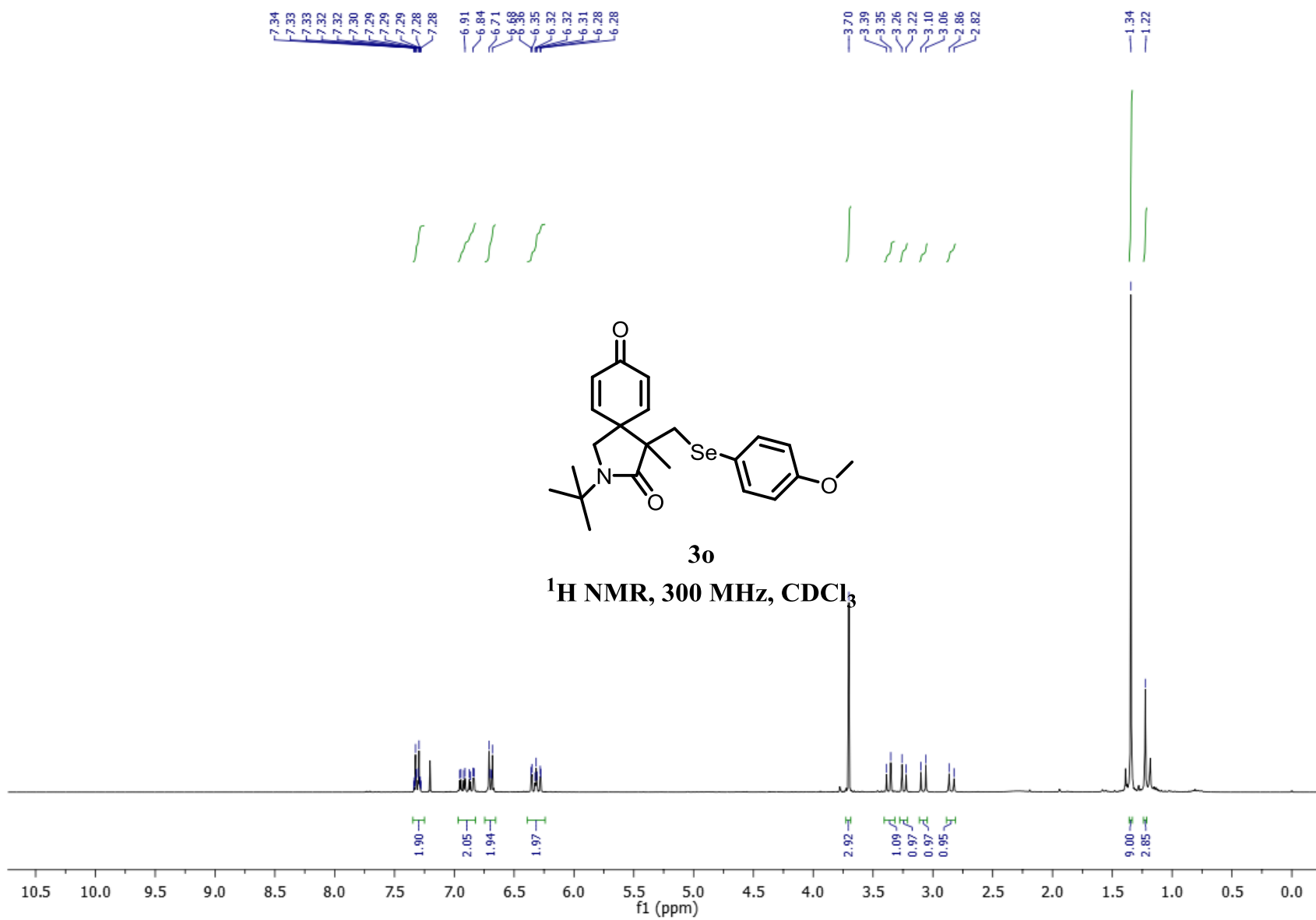
S33

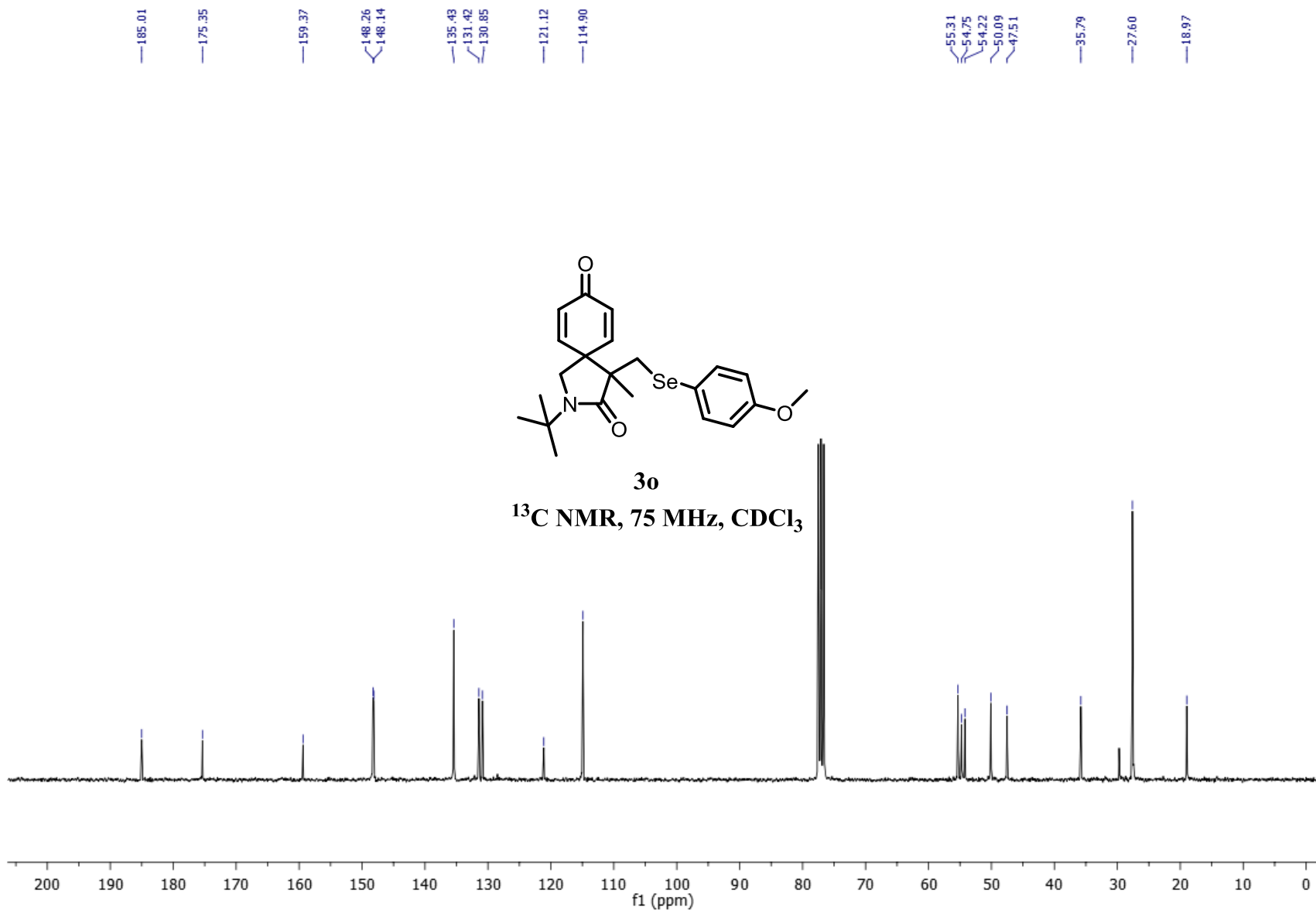


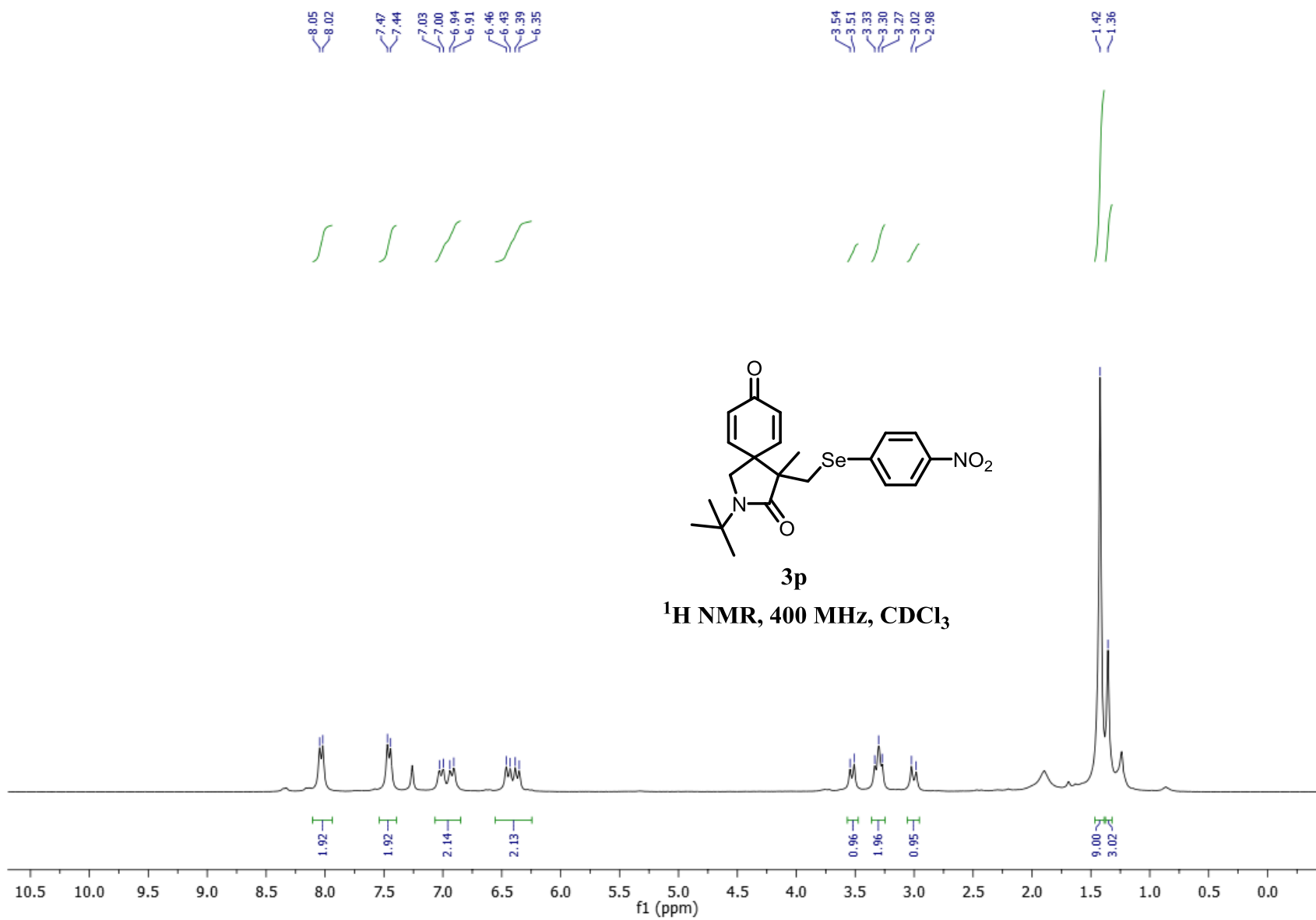




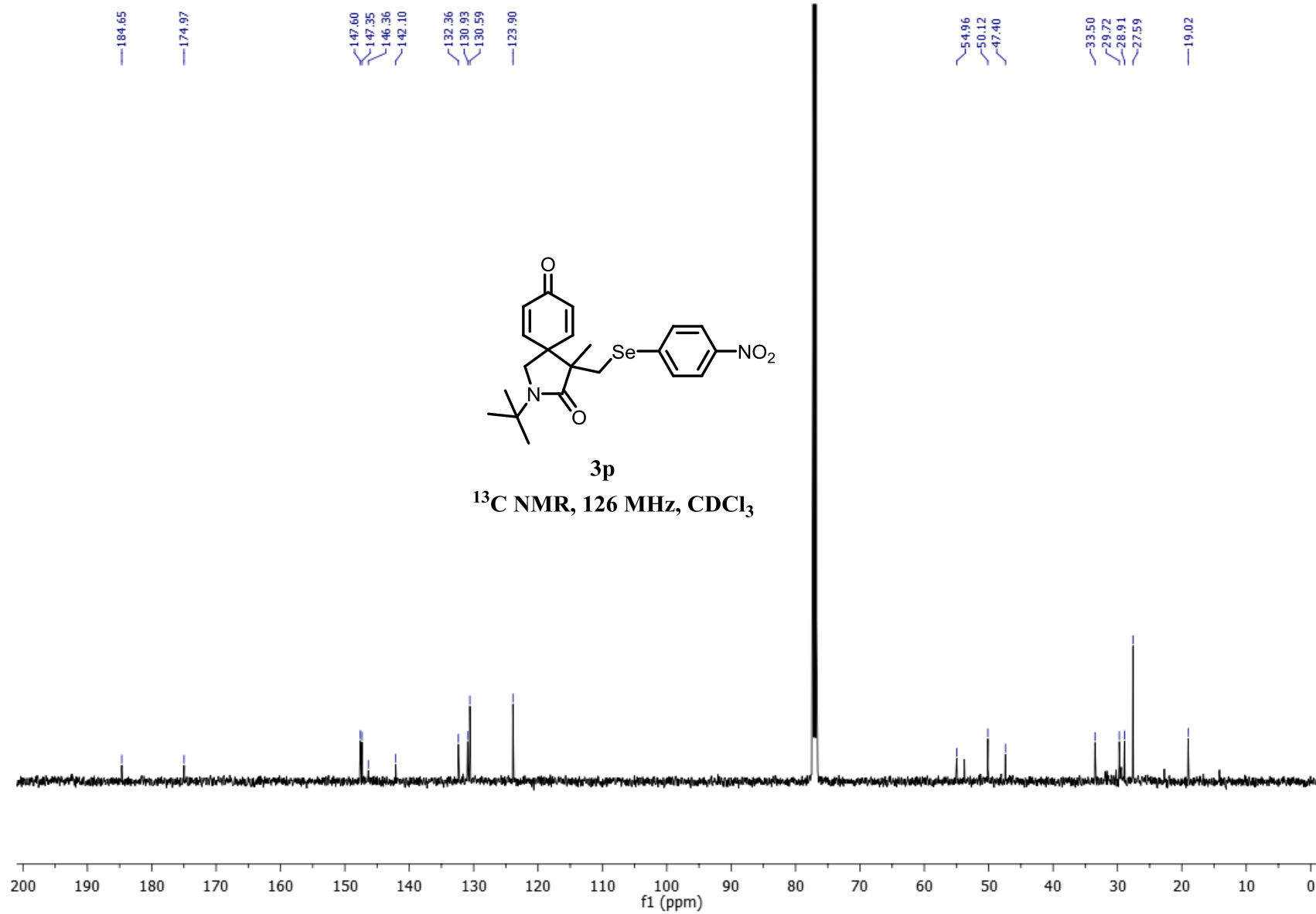


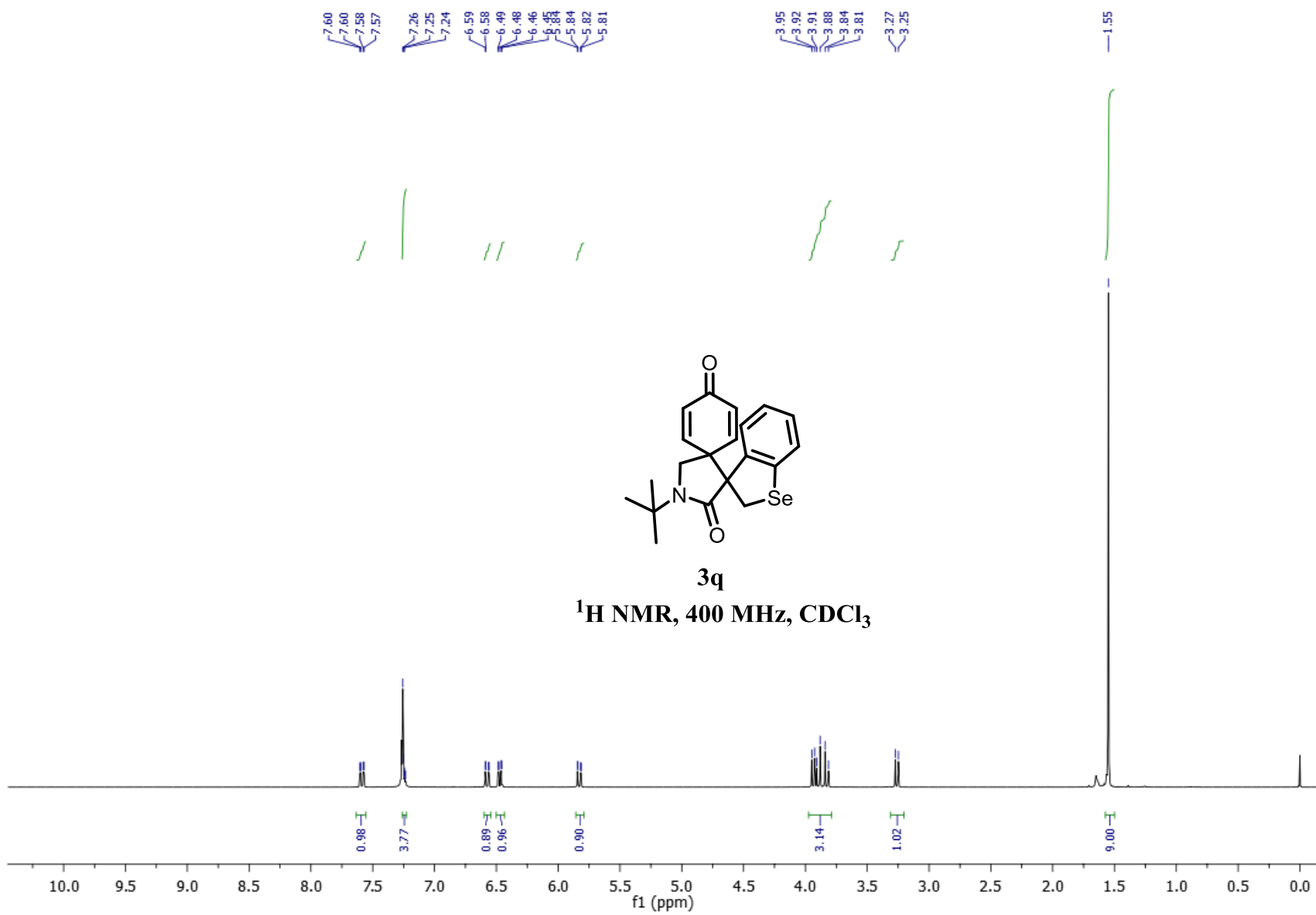


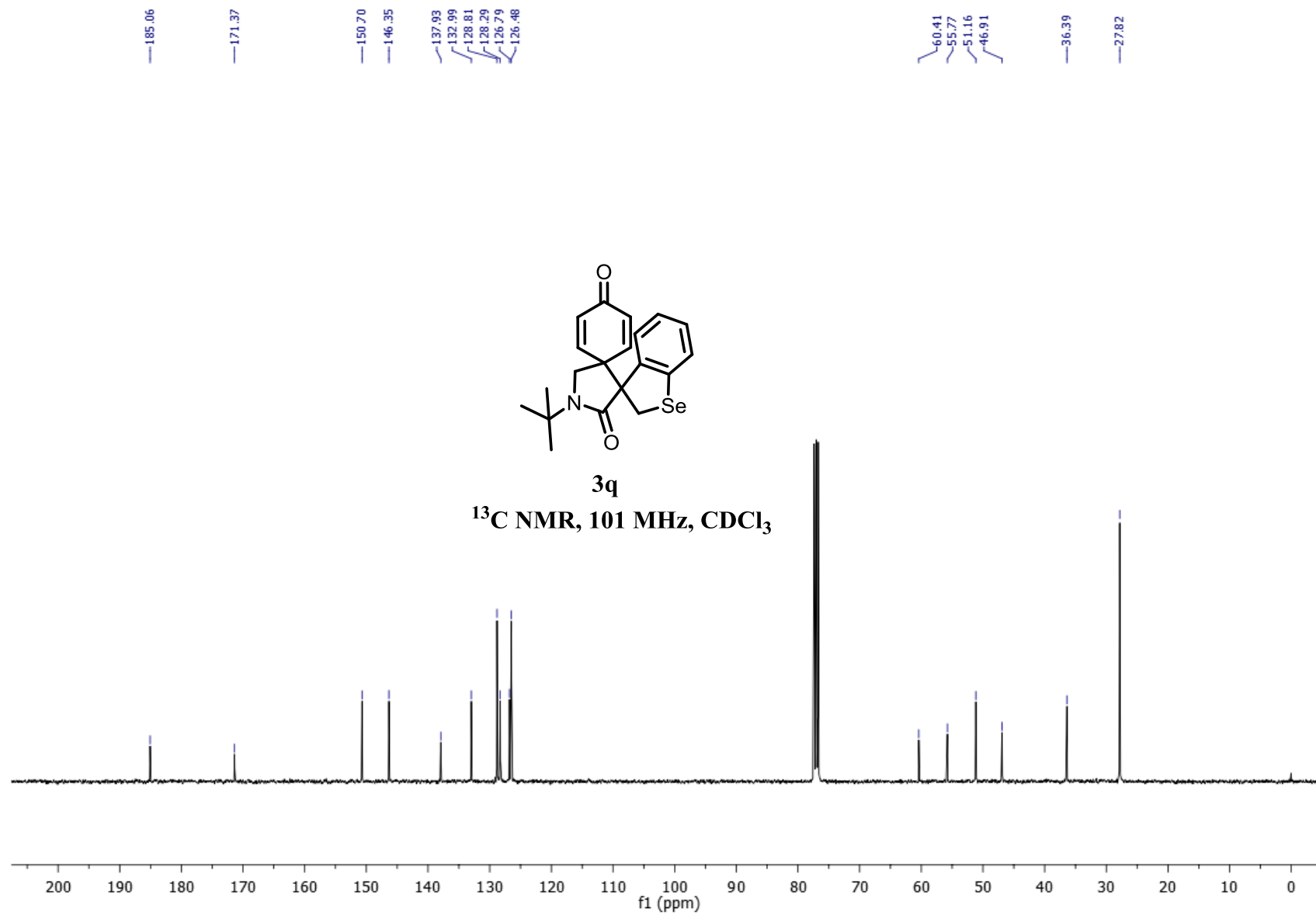


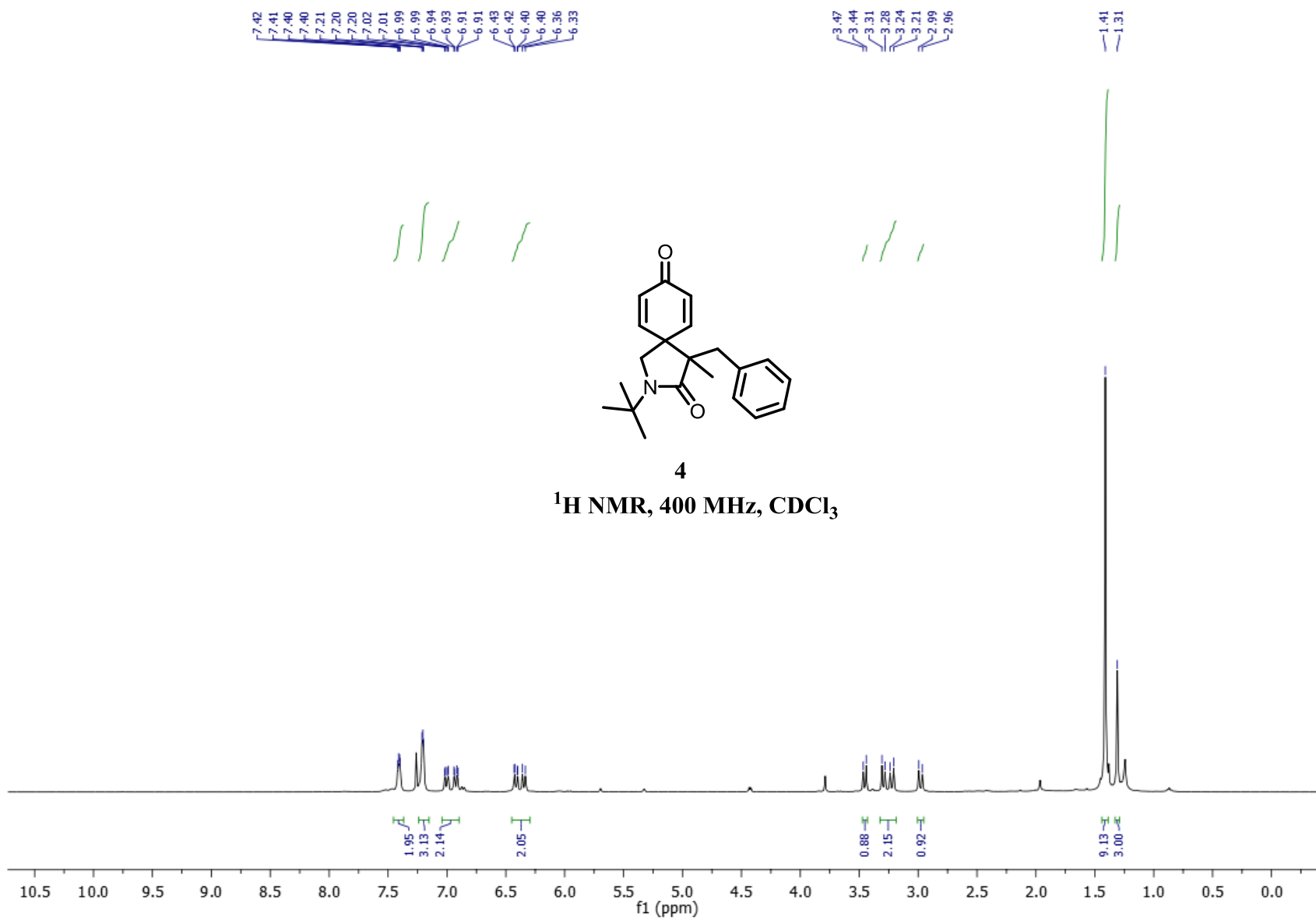


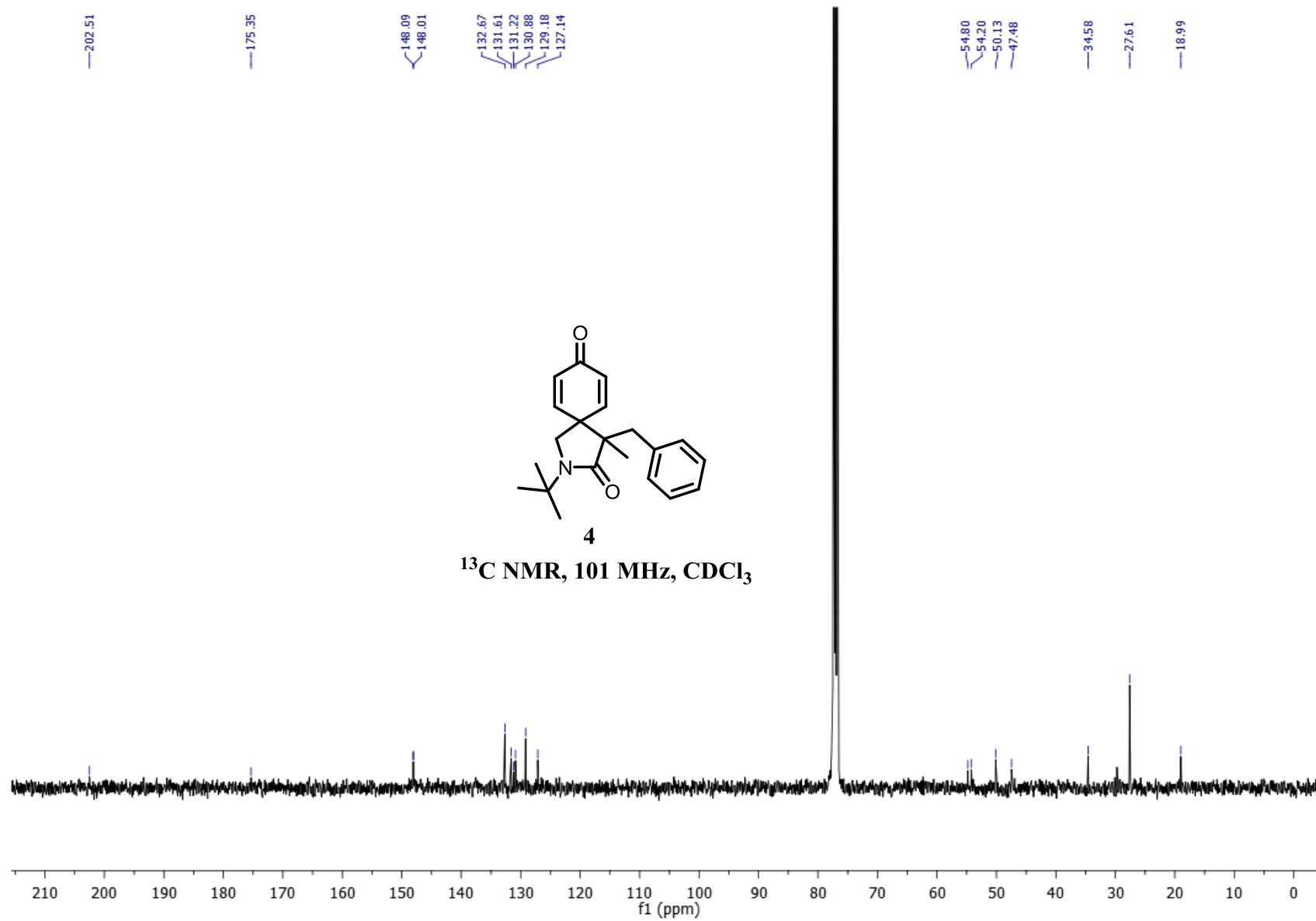


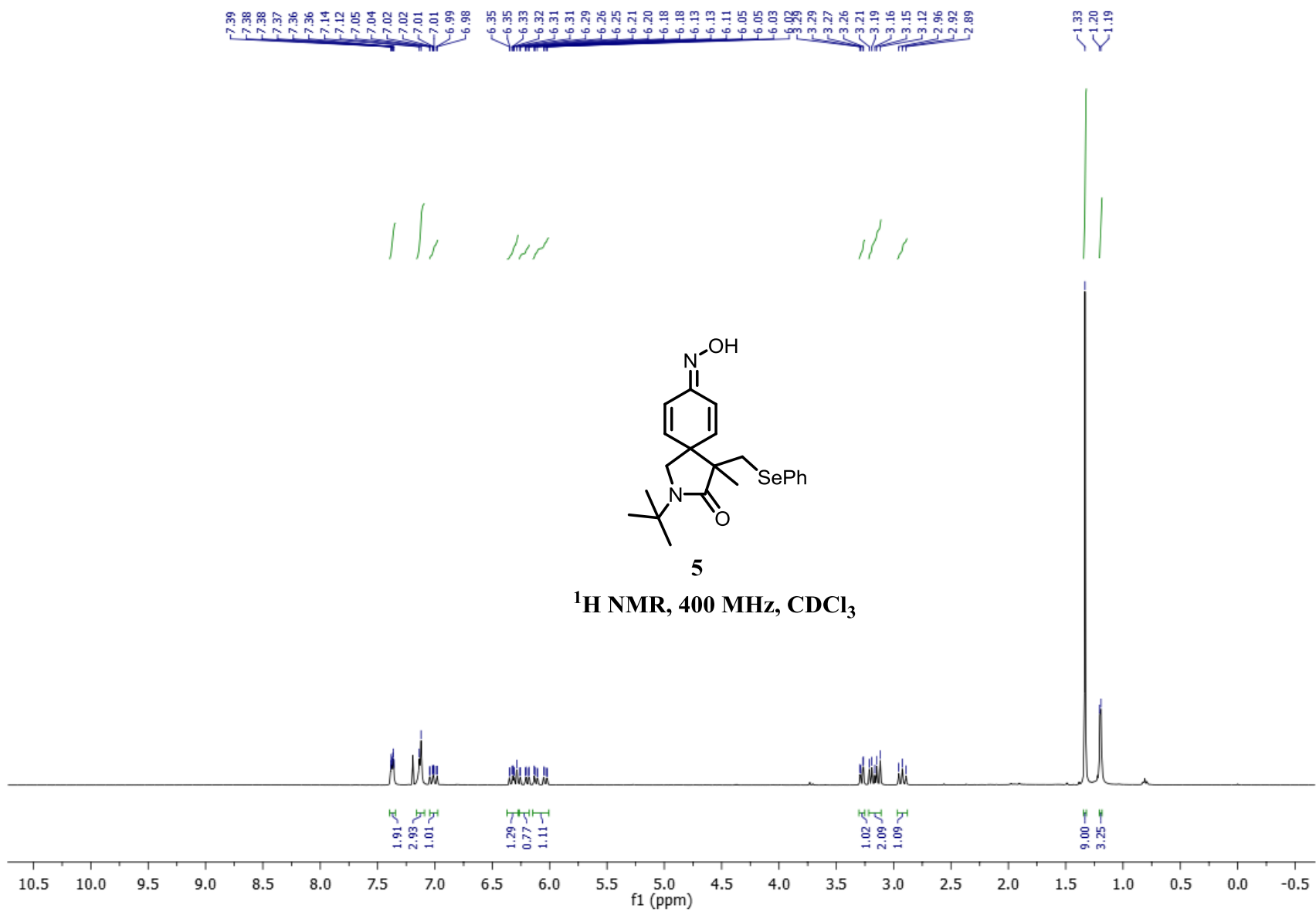


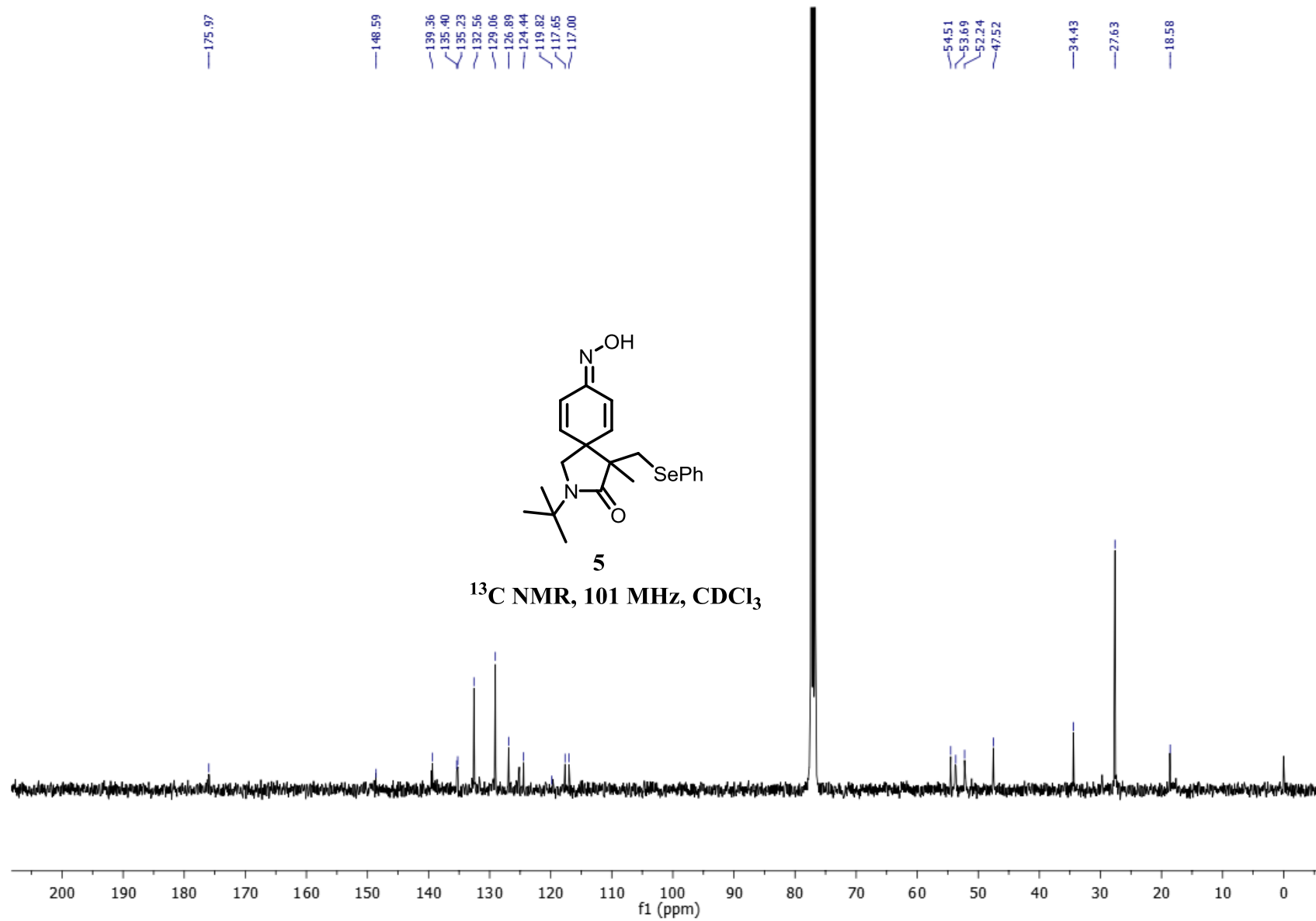




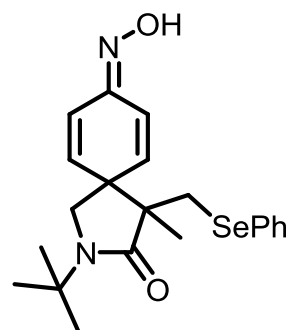






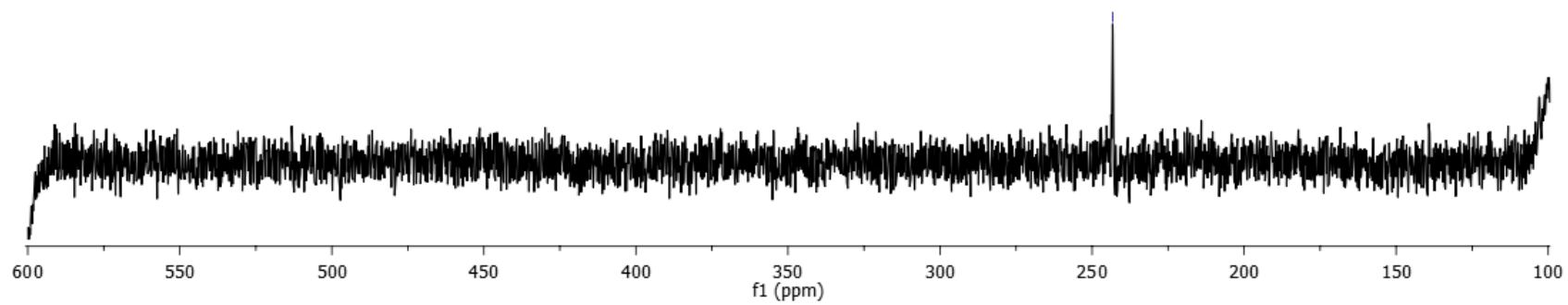


—248.22



5

<sup>77</sup>Se NMR, 76 MHz, CDCl<sub>3</sub>





## Elemental Composition Report

### Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = 1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

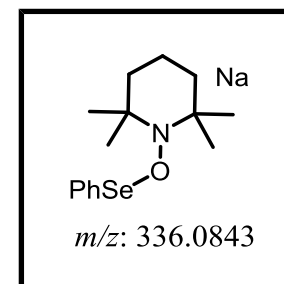
Monoisotopic Mass, Even Electron Ions

113 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

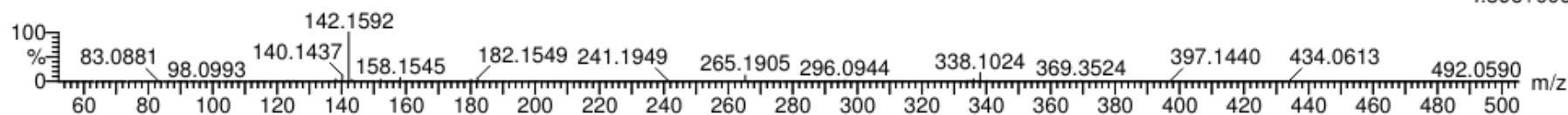
Elements Used:

C: 0-15 H: 0-23 B: 0-1 N: 0-1 O: 0-1 Na: 0-1 P: 0-2 Se: 0-1

300523\_CRR-AR-5-313 8 (0.290) AM2 (Ar,12000.0,0.00,0.00); Cm (7:15-(16:26+3:4))



1: TOF MS ES+  
4.39e+006



Minimum: 1.5  
Maximum: 5.0 100.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
336.1038	336.0843	19.5	58.0	5.5	301.7	n/a	n/a	C15 H23 N O Na Se