

# **An Oxidative Prins-Pinacol Tandem Process Mediated by a Hypervalent Iodine Reagent: Scope, Limitations and Applications**

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## **Supporting Information**

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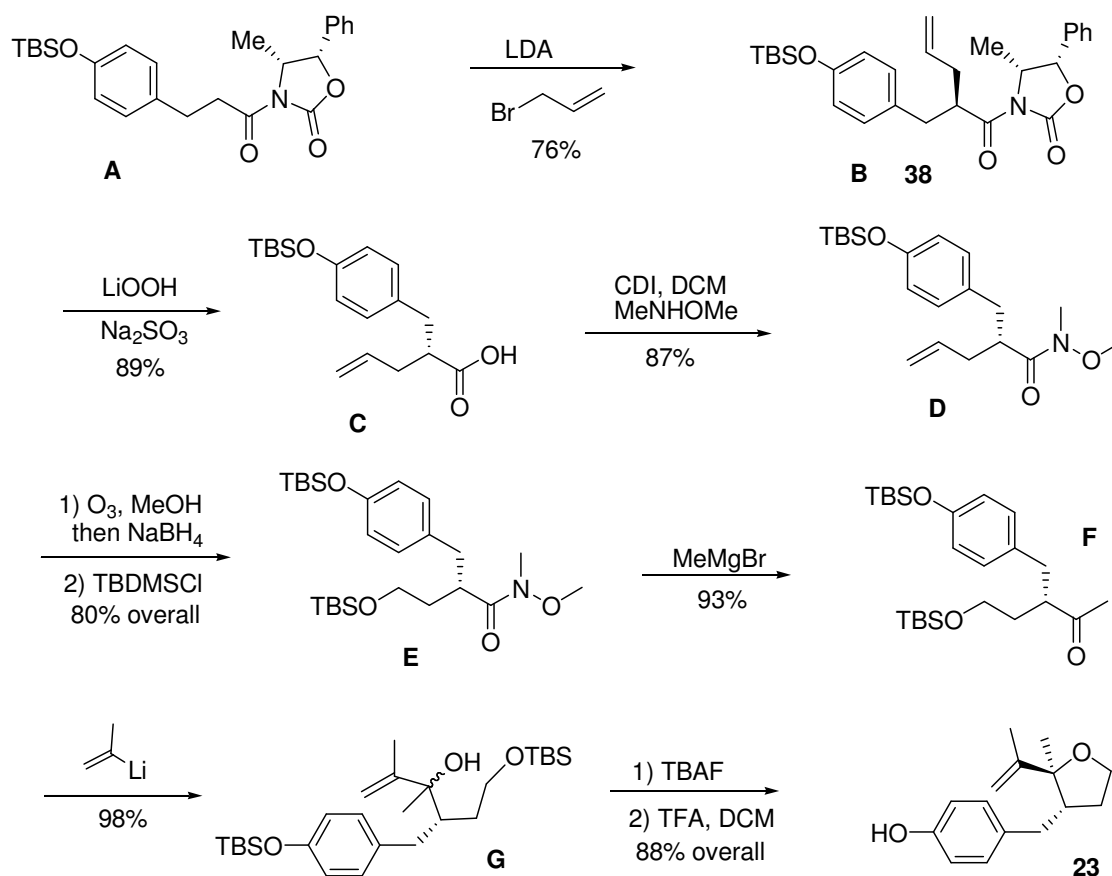
## I. General information and materials

Unless otherwise indicated,  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded at 300 and 75 MHz, respectively, in  $\text{CDCl}_3$  solutions. Chemical shifts are reported in ppm on the  $\delta$  scale. Multiplicities are described as s (singlet), d (doublet), dd, ddd, etc. (doublet of doublets, doublet of doublets of doublets, etc.), t (triplet), q (quartet), quin (quintuplet), m (multiplet), and further qualified as app (apparent), br (broad). Coupling constants,  $J$ , are reported in Hz. IR spectra ( $\text{cm}^{-1}$ ) were recorded from thin films. Mass spectra ( $m/e$ ) were measured in the electrospray (ESI) mode.

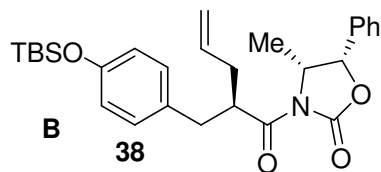
### II.4: Representative procedures for the formal synthesis of (-)-Platensimycin:

This experimental part is also present in reference 3i of this manuscript.

#### a) Synthesis of compound **23**.

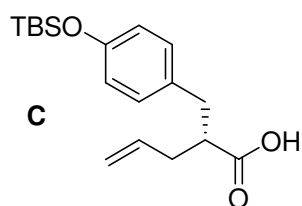


b) Procedures. (see reference 3i)



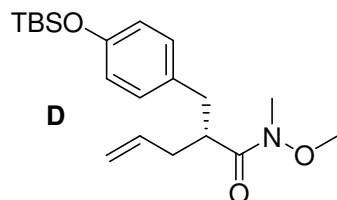
To a solution of the Evans oxazolidinone **A** (1.4 mmol, 615 mg) in THF (4.5 mL) was added LDA (4.2 mmol, 4.2 mL) at  $-78^{\circ}\text{C}$ . After 20 minutes, allyl bromide (7 mmol, 0.61 mL) was added dropwise. The reaction was then slowly warmed at  $5^{\circ}\text{C}$  and stirred until completion by TLC. The reaction was quenched by the addition of saturated  $\text{NH}_4\text{Cl}$  solution, the

aqueous layer was extracted with ethyl acetate. The combined organic extracts were dried ( $\text{Na}_2\text{SO}_4$ ), filtered and the solvent removed *in vacuo*. The crude product was purified by silica gel chromatography (hexane/ethyl acetate 90/10) to yield a yellow oil **B** (or **38**) (76%, 510 mg).  $[\alpha]_{\text{D}}^{20} = -50$  ( $c = 1$ , in  $\text{CHCl}_3$ ); **IR**  $\nu$  ( $\text{cm}^{-1}$ ) 1782, 1648, 1607, 1342, 1253, 1195;  **$^1\text{H}$**  NMR (600 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.38$  (t,  $J = 8.2$  Hz, 3H); 7.25 (t,  $J = 8.2$  Hz, 2H); 7.09 (d,  $J = 8.2$  Hz, 2H); 6.77 (d,  $J = 8.2$  Hz, 2H); 5.83 (m, 1H), 5.28 (d,  $J = 7.1$  Hz, 1H); 5.07 (d,  $J = 17.0$  Hz, 1H); 5.03 (d,  $J = 9.4$  Hz, 1H); 4.57 (q,  $J = 6.6$  Hz, 1H); 4.29 (m, 1H); 2.88 (dd,  $J = 13.7, 9.4$  Hz, 1H); 2.78 (dd,  $J = 13.7, 6.6$  Hz, 1H); 2.50 (m, 1H), 2.33 (m, 1H); 0.96 (s, 9H); 0.82 (d,  $J = 13.7$  Hz, 3H); 0.18 (s, 6H); 0.16 (s, 6H);  **$^{13}\text{C}$**  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 175.2, 154.1, 152.5, 134.9, 133.1, 131.5, 130.0, 128.5, 125.5, 119.8, 117.1, 78.6, 54.9, 44.1, 37.6, 36.3, 25.6, 18.1, 14.5, -4.4$ ; **HRMS** (ESI): Calc. for  $\text{C}_{28}\text{H}_{38}\text{NO}_4\text{Si}$  ( $\text{M}+\text{H}^+$ ): 480.2565; found: 480.2573.



To a solution of compound **B** (1.2 mmol, 575 mg) in THF/ $\text{H}_2\text{O}$  (3/1, 24 mL) was added  $\text{H}_2\text{O}_2$  (30% in  $\text{H}_2\text{O}$ , ~9.6 mmol) followed by  $\text{LiOH}\cdot\text{H}_2\text{O}$  (2.4 mmol, 101 mg). The mixture was allowed to warm to room temperature and stirred until completion by TLC (~ 10 min.). The mixture was cooled to  $0^{\circ}\text{C}$  and quenched with saturated  $\text{Na}_2\text{SO}_3$  solution, and diluted with ethyl acetate (25 mL) then citric

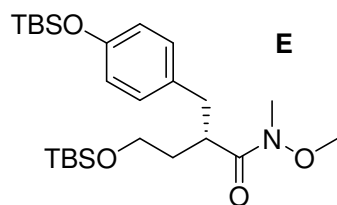
acid was added (460 mg, 3.6 mmol, 1.5 equiv.  $\text{pH} \sim 3$ ), the aqueous layer was extracted with ethyl acetate (3\*20 mL). The combined organic phases were washed with brine (30 mL), dried ( $\text{Na}_2\text{SO}_4$ ), filtered and the solvent removed *in vacuo*, the crude product was purified by silica gel chromatography (hexane/ethyl acetate 25/75) to yield a colourless oil **C** (89%, 342 mg).  $[\alpha]_{\text{D}}^{20} = +20$  ( $c = 0.6$  in  $\text{CHCl}_3$ ); **IR**  $\nu$  ( $\text{cm}^{-1}$ ) 2922, 1705, 1503, 1249;  **$^1\text{H}$**  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.04$  (d,  $J = 8.2$  Hz, 2H); 6.75 (d,  $J = 8.2$  Hz, 2H); 5.78 (m, 1H); 5.10 (d,  $J = 8.2$  Hz, 1H); 5.08 (d,  $J = 18.1$  Hz, 1H); 2.92 (dd,  $J = 10.4, 15.9$  Hz, 1H); 2.73 (m, 2H); 2.33 (m, 2H); 0.98 (s, 9H); 0.19 (s, 6H).  **$^{13}\text{C}$**  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 180.7, 154.1, 134.8, 131.4, 129.8, 119.9, 117.3, 47.1, 36.5, 35.4, 25.6, 18.1, -4.4$ ; **HRMS** (ESI): Calc. for  $\text{C}_{18}\text{H}_{29}\text{O}_3\text{Si}$  ( $\text{M}+\text{H}^+$ ): 321.1880; found: 321.1884.



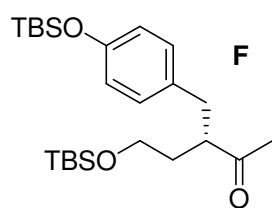
To a solution of compound **C** (320 mg, 1 mmol) in dichloromethane (5 mL) at  $0^{\circ}\text{C}$  was added carbonyldiimidazole (210 mg, 1.3 mmol) and a catalytic amount of DMAP (10 %, 12 mg, 0.1 mmol). The resulting solution was stirred for 1 hour. To this solution was added *O,N*-dimethylhydroxylamine hydrochloride (300 mg, 3 mmol) and the reaction was stirred overnight. The

reaction solution was then quenched with saturated  $\text{NH}_4\text{Cl}$  solution (10 mL) then diluted with ethyl acetate (20 mL). The combined organic phases were washed with brine (30 mL), dried ( $\text{Na}_2\text{SO}_4$ ), filtered and the solvent removed *in vacuo*, the crude product was purified by silica gel chromatography (hexane/ethyl acetate 80/20) to yield a pale yellow oil **D** (87%, 317 mg).  $[\alpha]_{\text{D}}^{20} = -26$  ( $c = 0.5$  in  $\text{CHCl}_3$ ); **IR**  $\nu$  ( $\text{cm}^{-1}$ ) 2929, 1661, 1608, 1510,

1256;  $^1\text{H}$  (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.03 (d,  $J$  = 8.2 Hz, 2H); 6.73 (d,  $J$  = 8.2 Hz, 2H); 5.76 (m, 1H); 5.07 (d,  $J$  = 17.0 Hz, 1H); 5.01 (d,  $J$  = 12.0 Hz, 1H); 3.31 (s, 3H); 3.15 (m, 1H); 3.08 (s, 3H); 2.89 (dd,  $J$  = 13.2; 9.3 Hz, 1H); 2.64 (dd,  $J$  = 13.2; 5.5 Hz, 1H); 2.42 (m, 1H); 2.23 (m, 1H); 0.97 (s, 9H); 0.16 (s, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 175.8, 153.9, 135.7, 132.7, 129.9, 119.9, 116.6, 61.1, 43.3, 37.5, 36.7, 31.9, 25.6, 18.1, -4.4; **HRMS** (ESI): Calc. for  $\text{C}_{20}\text{H}_{34}\text{NO}_3\text{Si}$  ( $\text{M}+\text{H}$ ) $^+$ : 364.2302; found: 364.2300.



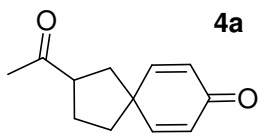
A solution of the allyl compound **D** (1.36 g, 3.74 mmol) in methanol (25 ml) at  $-78^\circ\text{C}$  was treated with ozone (bubbled through the solution until appearance of blue color). Argon was bubbled through the solution for 3 min. and  $\text{NaBH}_4$  (150 mg, 3.74 mmol) was added. The reaction was allowed to warm to room temperature and was stirred until the starting material disappears by TLC. The reaction was quenched with  $\text{NH}_4\text{Cl}$  (20 ml) and the mixture was concentrated in vacuo. The reaction was diluted with ethyl acetate (30 ml). The organic layer was removed and the aqueous layer was washed two times with ethyl acetate (15 mL). The organic layers were combined, dried over  $\text{Na}_2\text{SO}_4$  and concentrated in vacuo. The crude alcohol obtained was diluted as it in dry DMF at  $0^\circ\text{C}$  and imidazole (620 mg, 9 mmol), *tert*-butyldimethylsilyl chloride (685 mg, 4.50 mmol) were added. The resulting solution was stirred at room temperature for 12 hours and then was treated with sat. aq.  $\text{NaHCO}_3$  (10 mL). The aqueous phase was extracted with EtOAc (4 x 20 mL) and the combined organic layers were washed with brine (10 mL), dried over  $\text{Na}_2\text{SO}_4$ , concentrated under reduced pressure. The crude product was purified by chromatography over silica gel (*n*-hexane/ EtOAc, 88:12) to afford 1.45 g (80%, overall) of the product **E** as a yellow oil.  $[\alpha]_D^{20} = -9$  ( $c$  = 0.75 in  $\text{CHCl}_3$ ); **IR**  $\nu$  ( $\text{cm}^{-1}$ ) 2930, 1663, 1510, 1256;  $^1\text{H}$  (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.04 (d,  $J$  = 8.2 Hz, 2H); 6.73 (d,  $J$  = 8.2 Hz, 2H); 3.60 (m, 2H); 3.38 (s, 3H); 3.08 (s, 3H); 2.88 (dd,  $J$  = 13.2; 8.8 Hz, 1H); 2.62 (dd,  $J$  = 13.2; 6.0 Hz, 1H); 1.89 (m, 1H); 1.67 (m, 1H); 0.97 (s, 9H); 0.87 (s, 9H); 0.16 (s, 6H); 0.01 (s, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 176.4, 153.9, 132.8, 129.9, 119.8, 61.0, 60.7, 39.7, 37.7, 35.1, 31.9, 25.8, 25.6, 18.1, -4.4, -5.3; **HRMS** (ESI): Calc. for  $\text{C}_{25}\text{H}_{48}\text{NO}_4\text{Si}_2$  ( $\text{M}+\text{H}$ ) $^+$ : 482.3116; found: 482.3113.



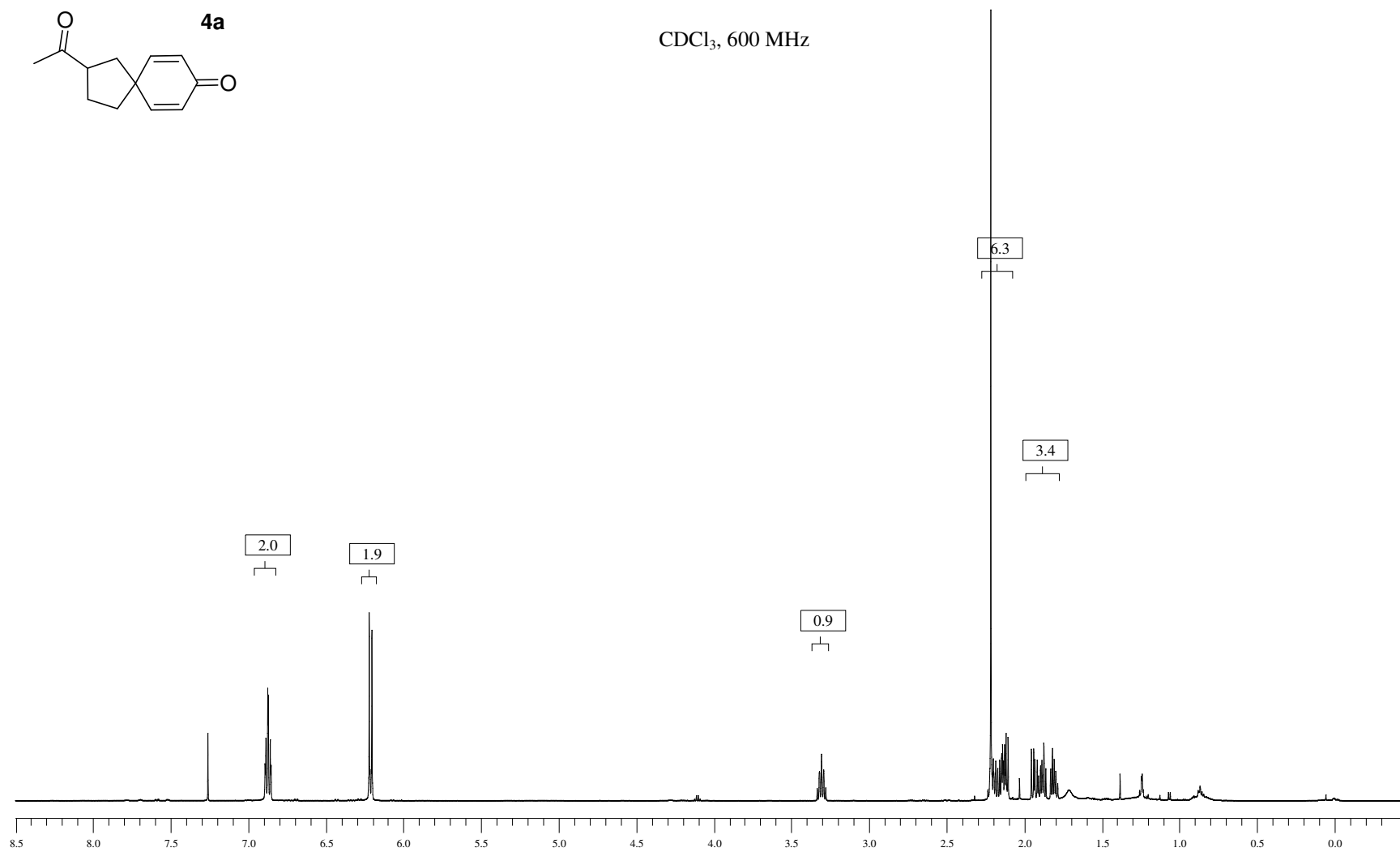
To a solution of the Weinreb amide **E** (1.45 g, 3 mmol) in dry THF (13 mL) at  $0^\circ\text{C}$  was added methylmagnesium bromide (3M in THF, 2 mL, 6 mmol, 2 equiv.) dropwise. The reaction mixture was stirred  $0^\circ\text{C}$  for 1 h and then a solution of 10 mL of sat. aq.  $\text{NH}_4\text{Cl}$  was added. The aqueous phase was extracted with EtOAc (3 \* 10 mL) and the combined organic layers were washed with brine, dried over  $\text{Na}_2\text{SO}_4$ , concentrated under reduced pressure. The crude product was purified by chromatography (*n*-hexane:EtOAc, 9:1) to afford 1.22 g (93%) of the compound **F** desired as a colorless oil.  $[\alpha]_D^{20} = -20$  ( $c$  = 0.9 in THF); **IR**  $\nu$  ( $\text{cm}^{-1}$ ) 2954, 1715, 1510, 1256, 1106;  $^1\text{H}$  (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.00 (d,  $J$  = 8.8 Hz, 2H); 6.74 (d,  $J$  = 8.8 Hz, 2H); 3.58 (t,  $J$  = 6.0 Hz, 2H); 2.96 (m, 1H); 2.80 (dd,  $J$  = 13.7; 8.2 Hz, 1H); 2.61 (dd,  $J$  = 13.7; 7.1 Hz, 1H); 1.98 (s, 3H); 1.87 (m, 1H); 1.62 (m, 1H); 0.98 (s, 9H); 0.88 (s, 9H); 0.18 (s, 6H); 0.02 (s, 6H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 212.5, 154.0, 132.1, 129.7, 120.0, 60.9, 51.4, 37.4, 34.4, 30.6, 25.8, 25.6, 18.2, 18.1, -4.4, -5.4. **HRMS** (ESI): Calc. for  $\text{C}_{24}\text{H}_{45}\text{O}_3\text{Si}$  ( $\text{M}+\text{H}$ ) $^+$ : 437.2902; found: 437.2899.

**Copies of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra**

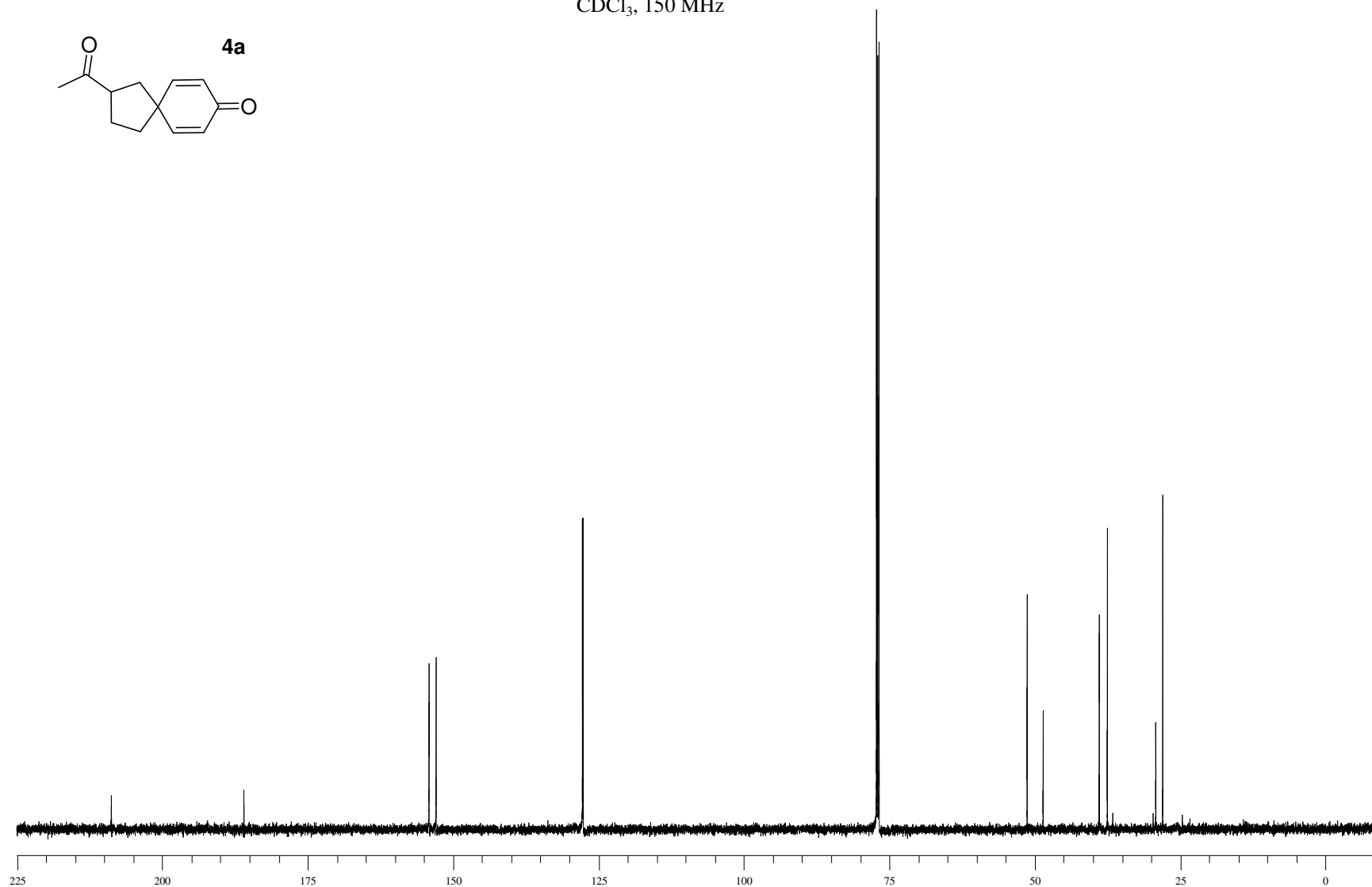
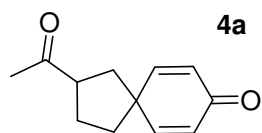


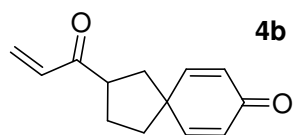


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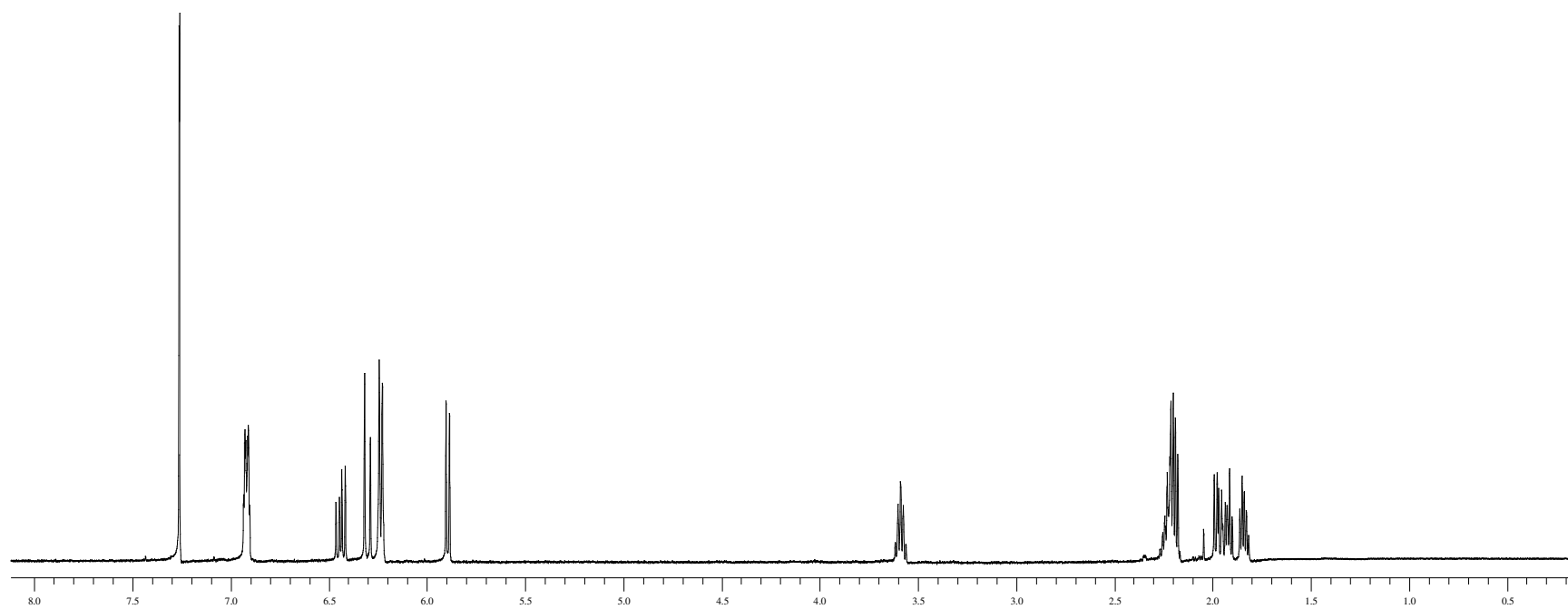


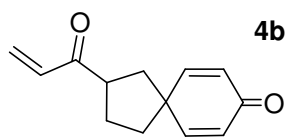
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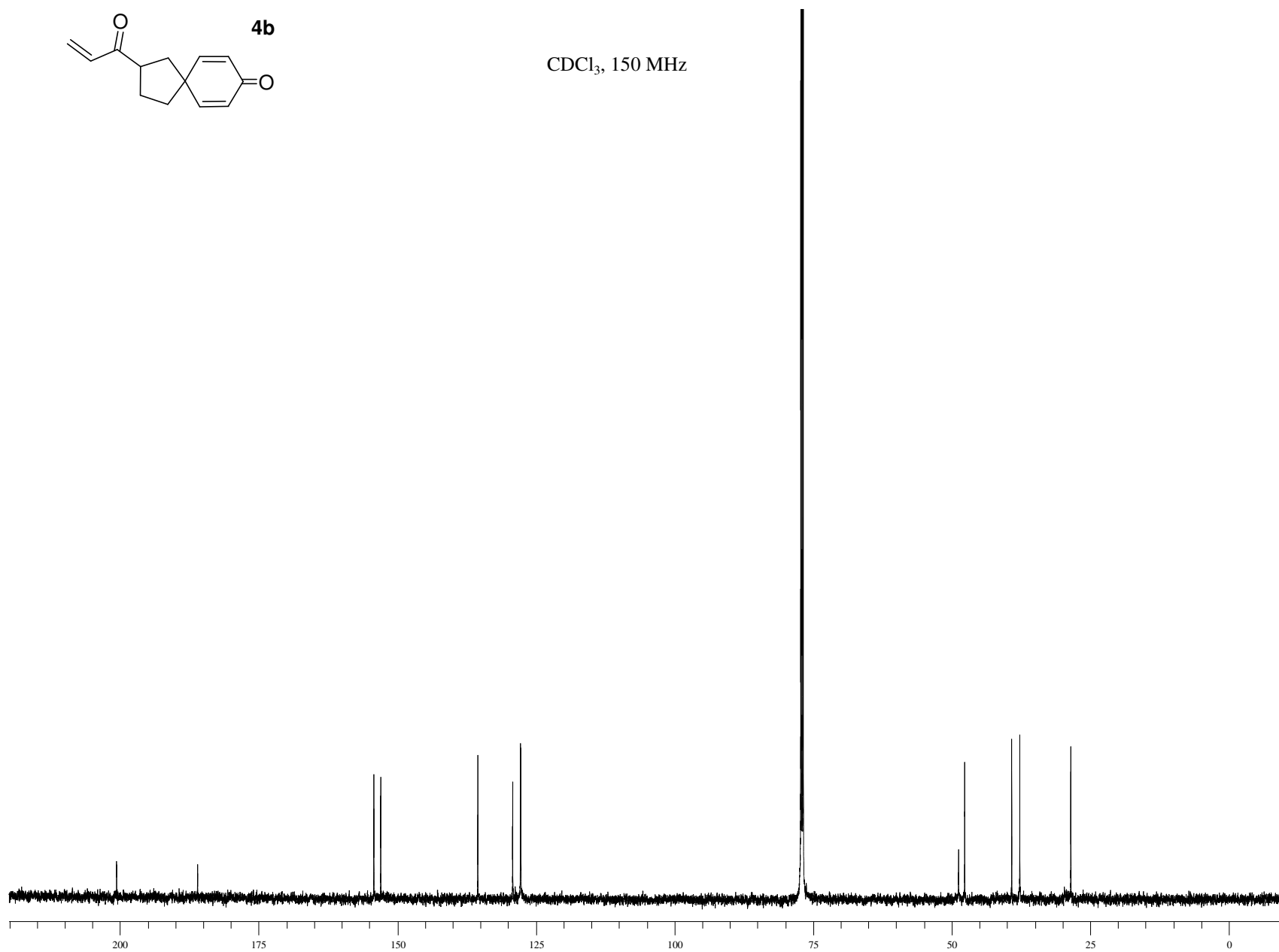


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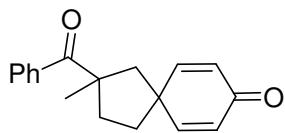




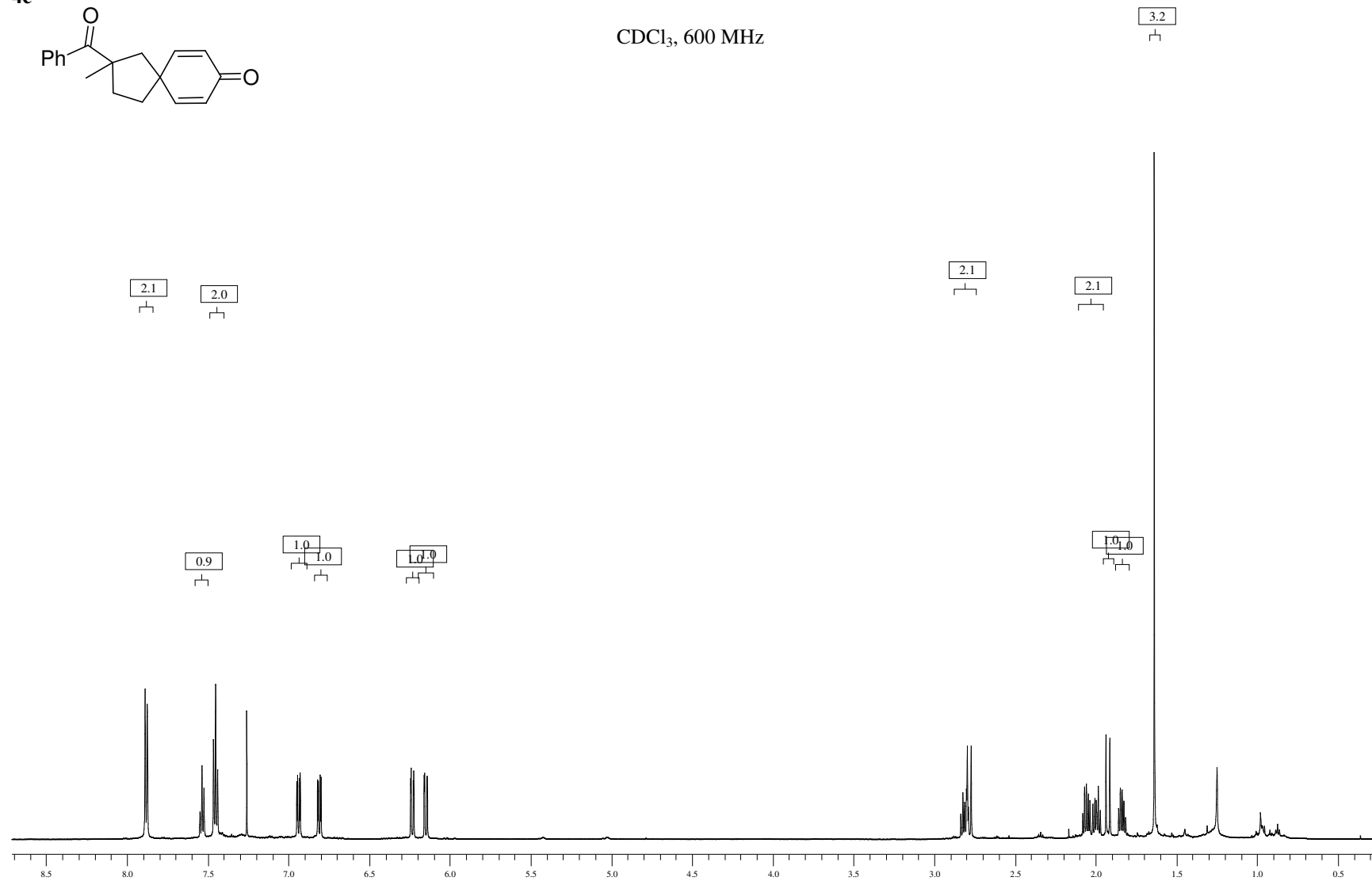
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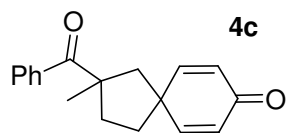


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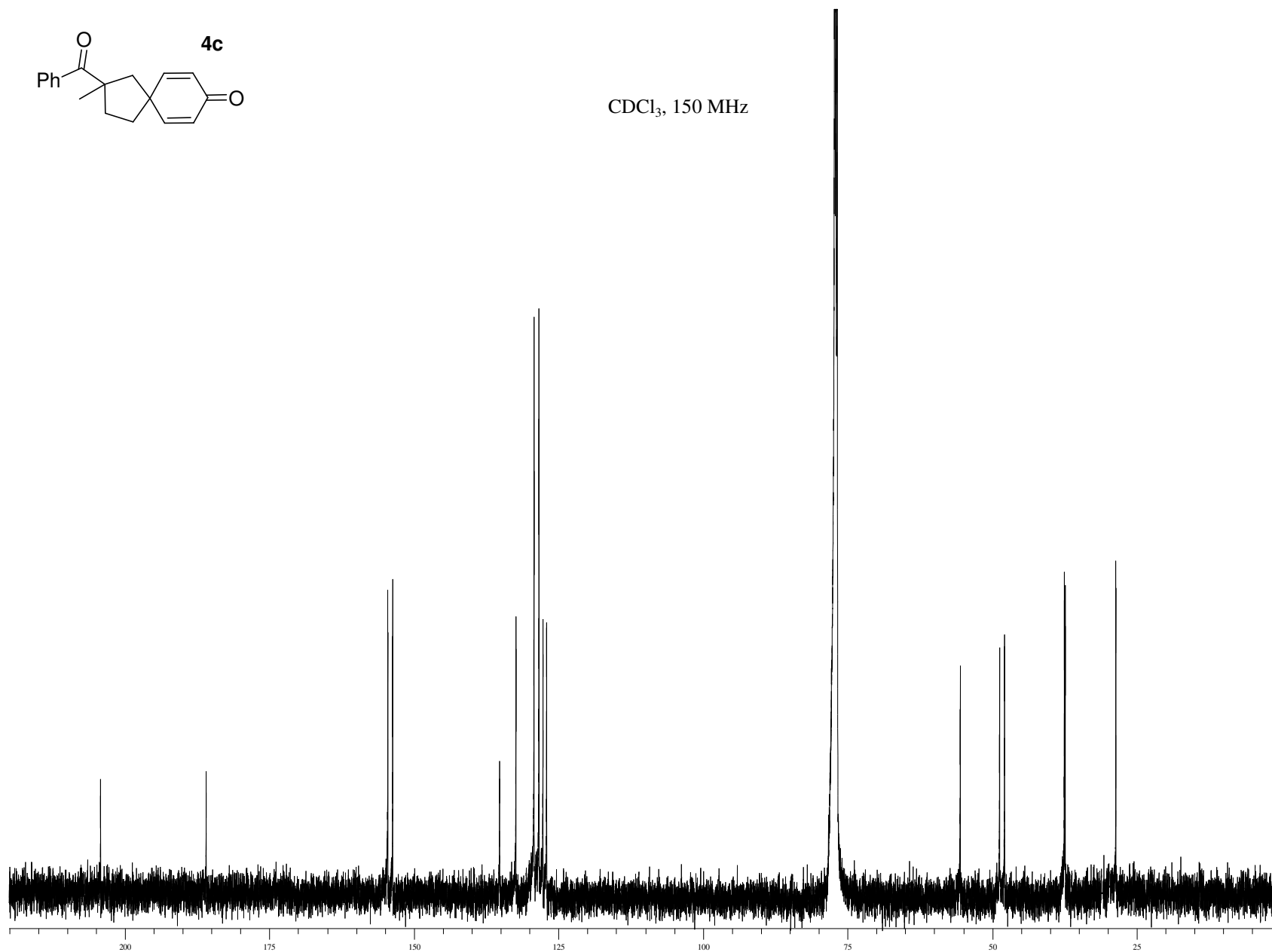


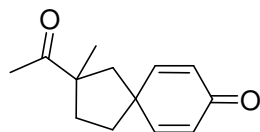
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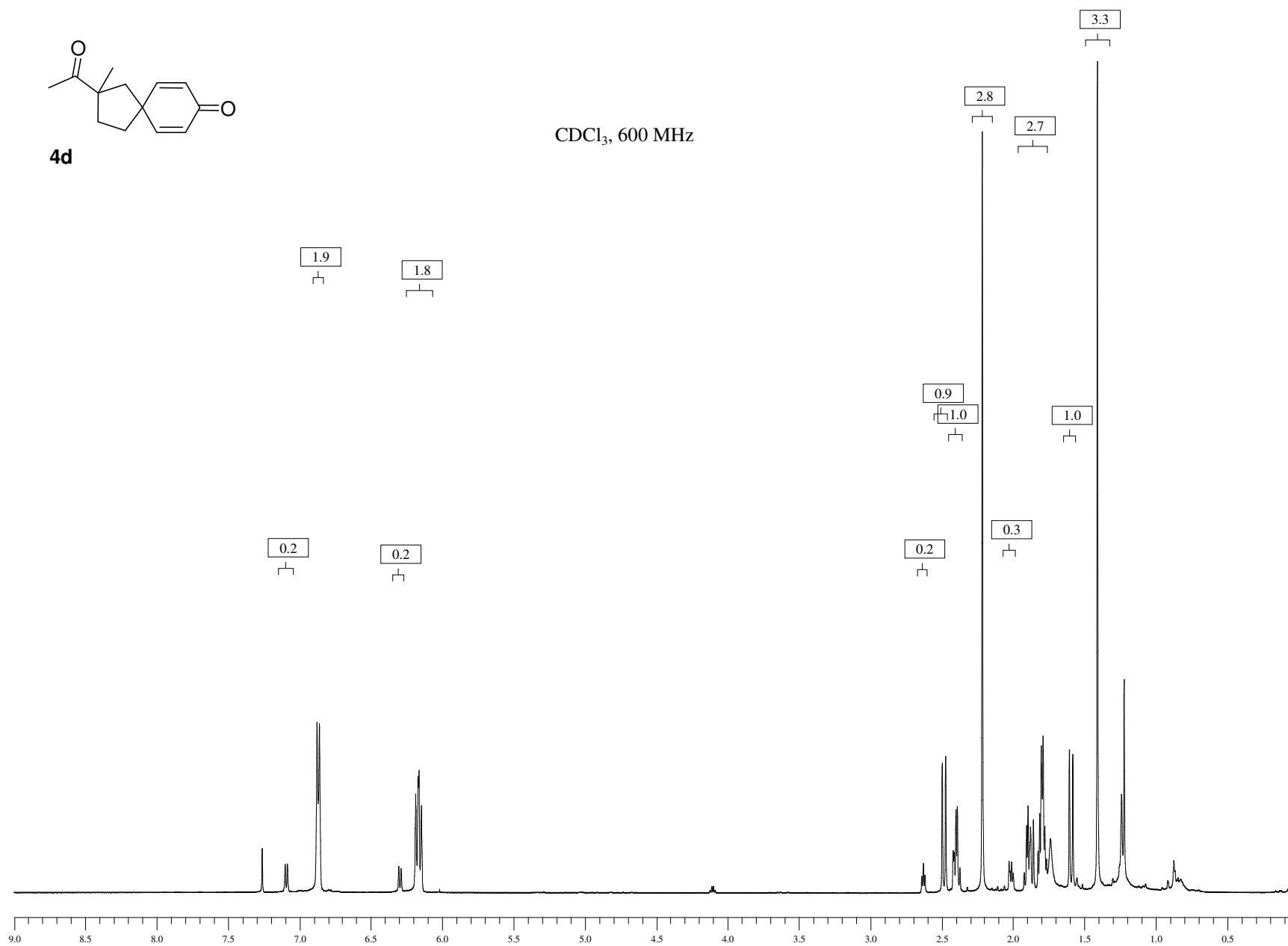
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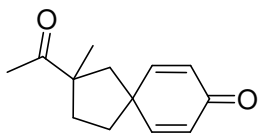




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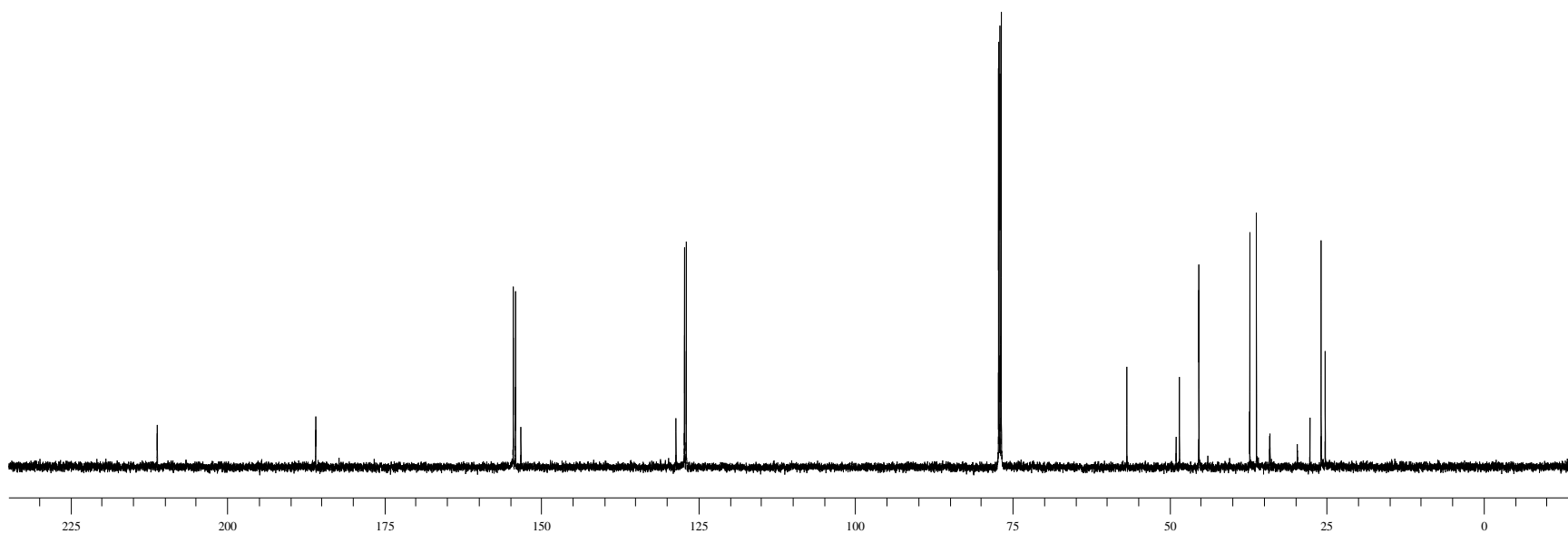
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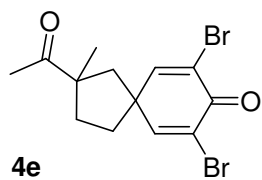


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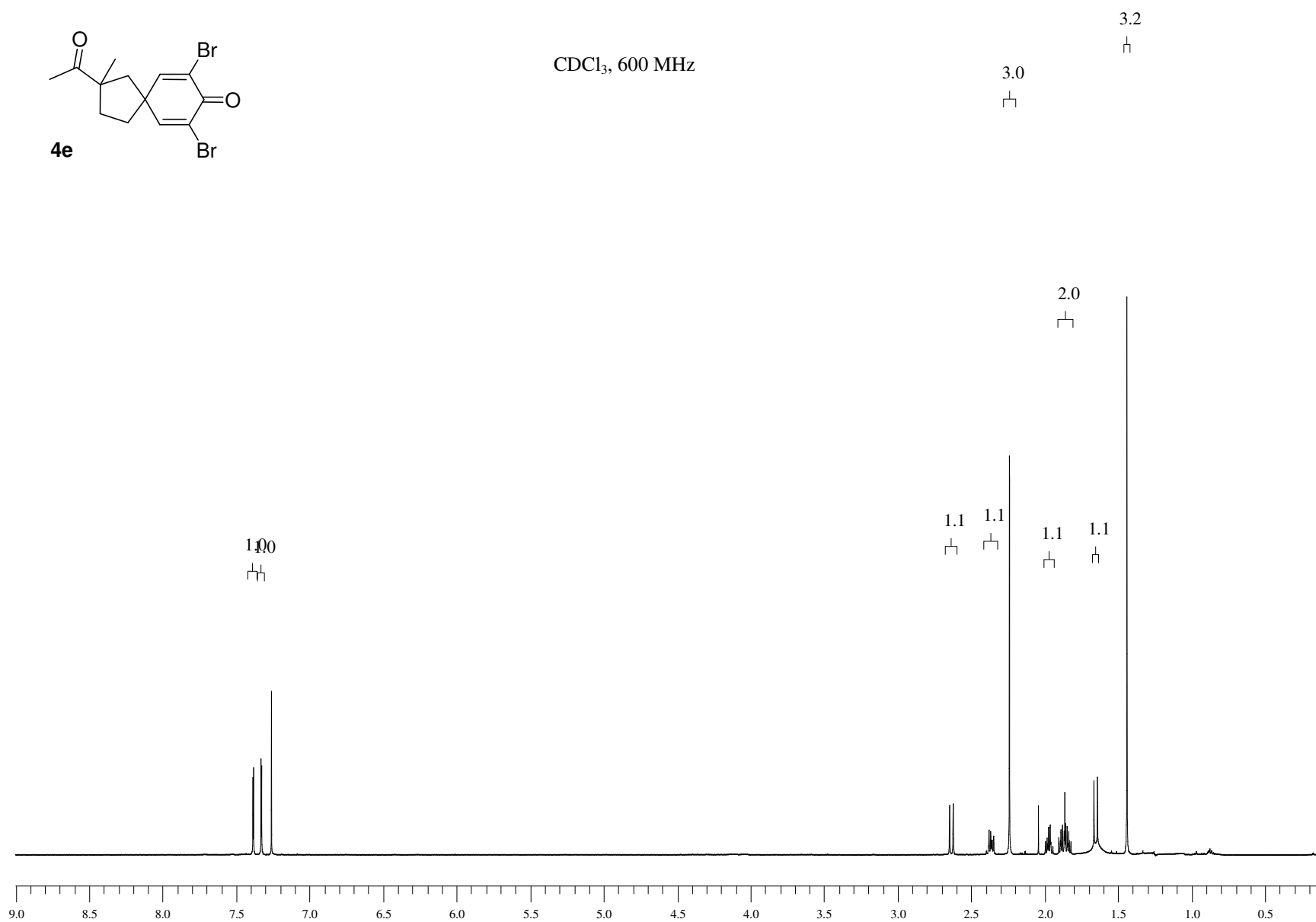
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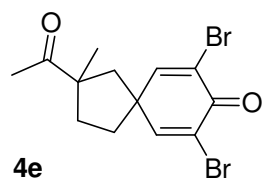




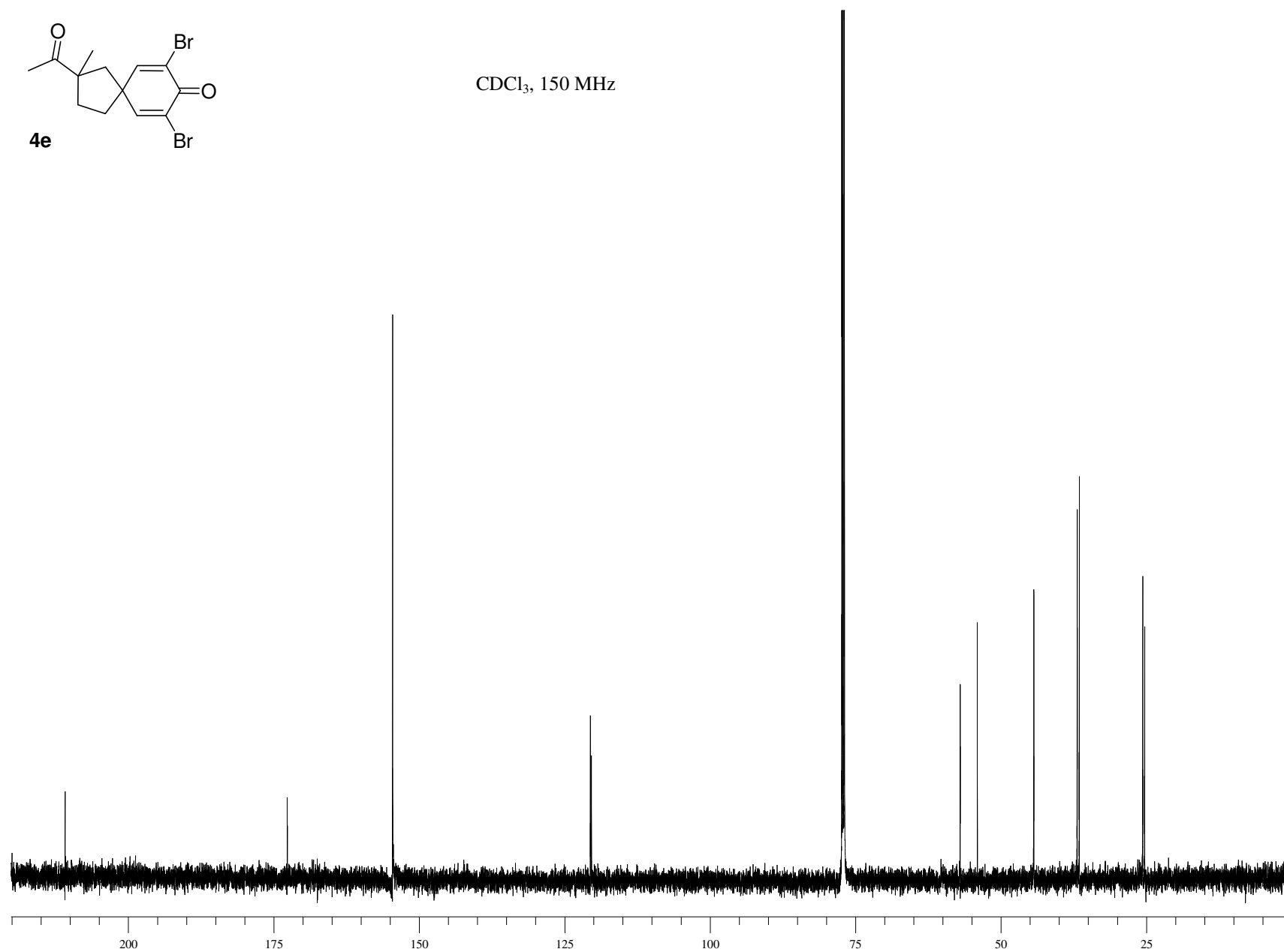


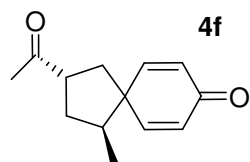
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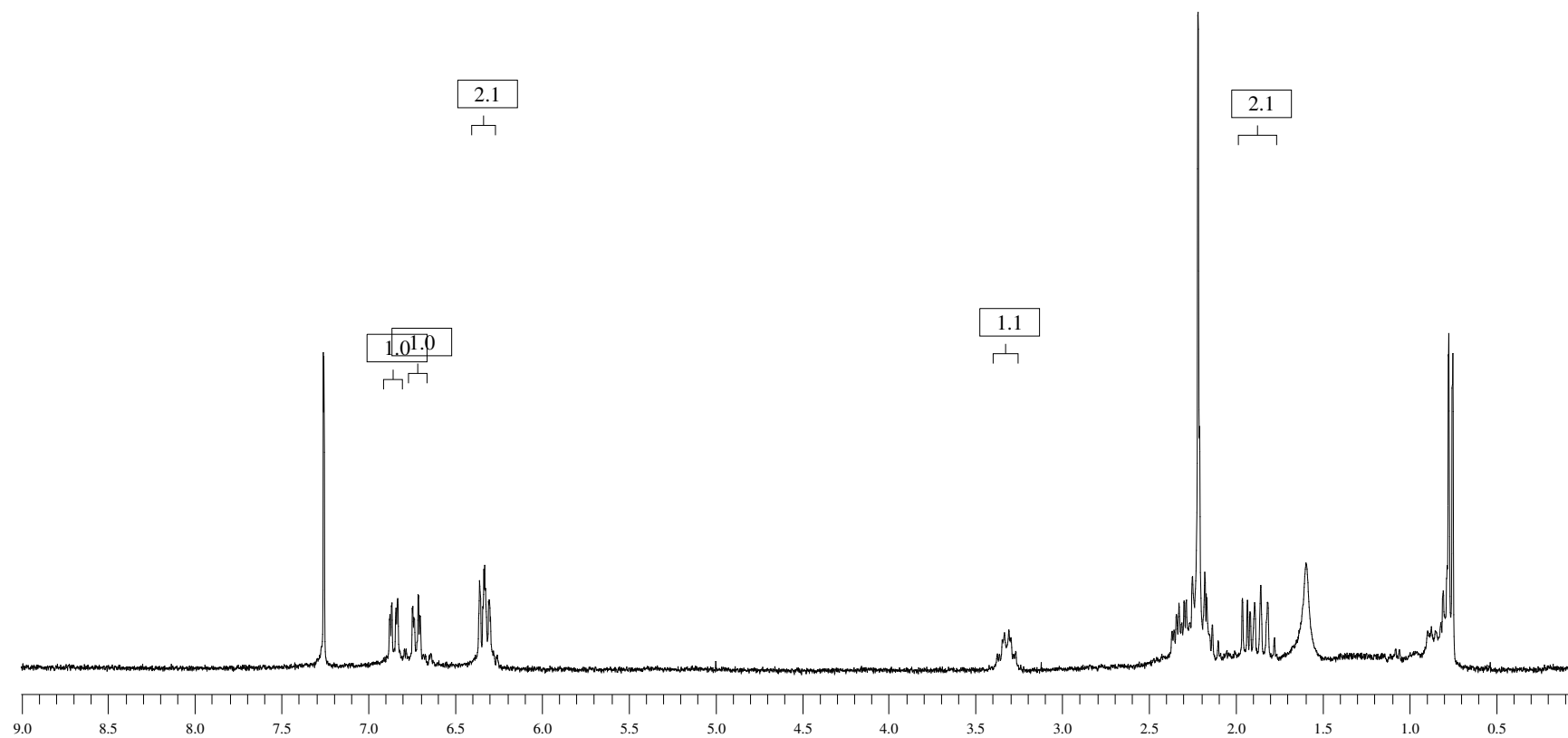


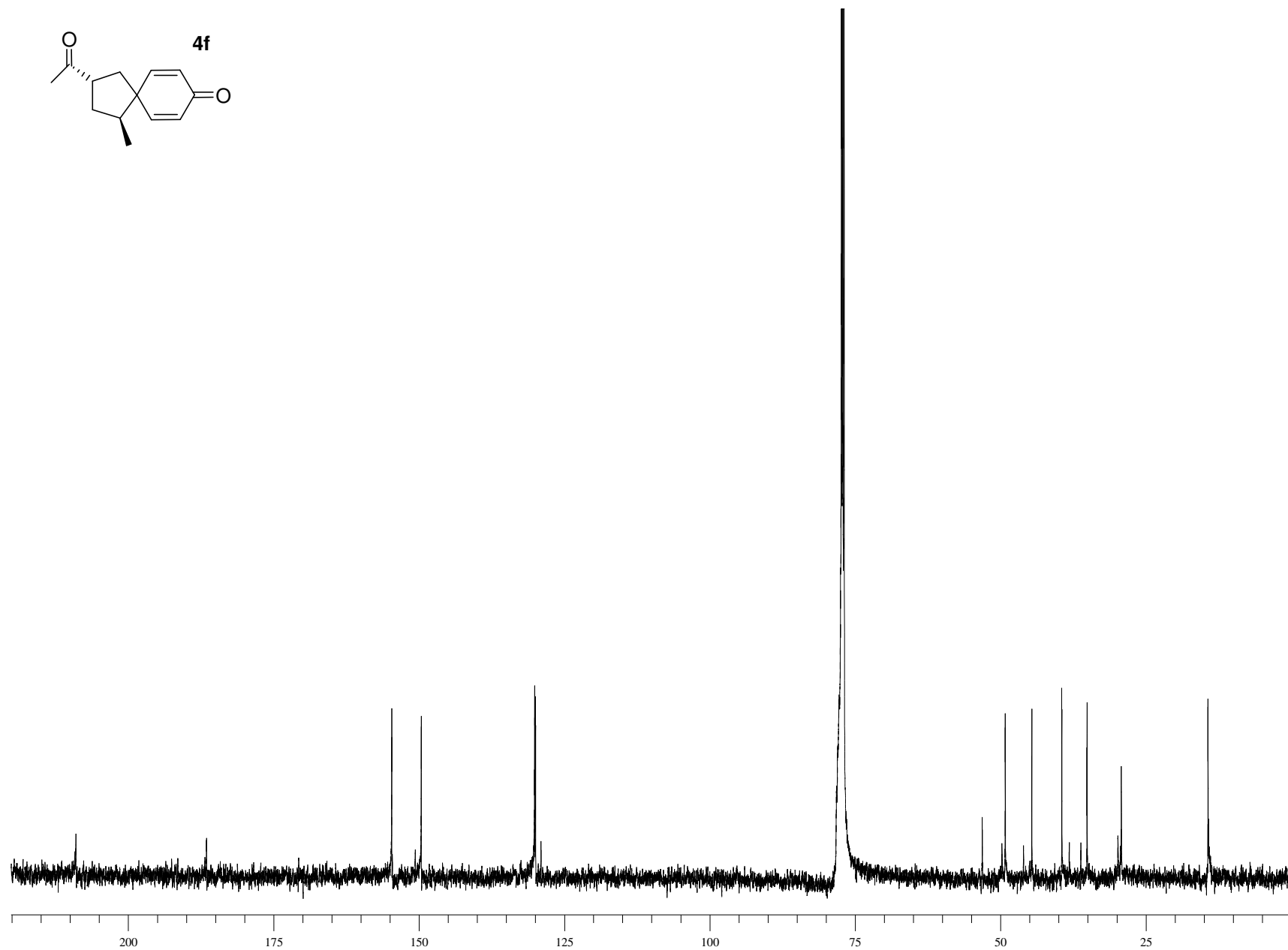
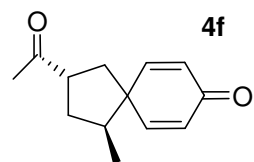
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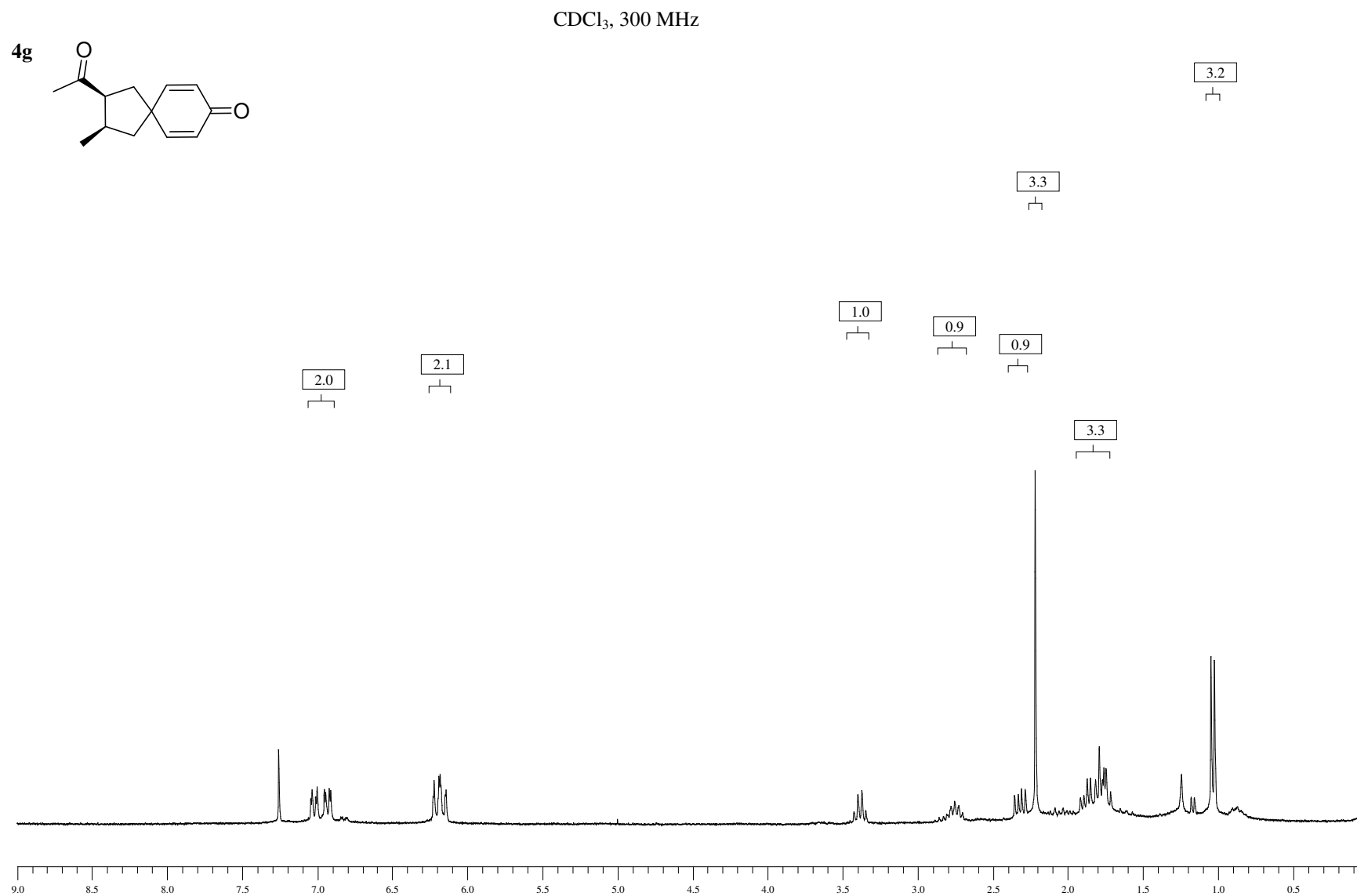


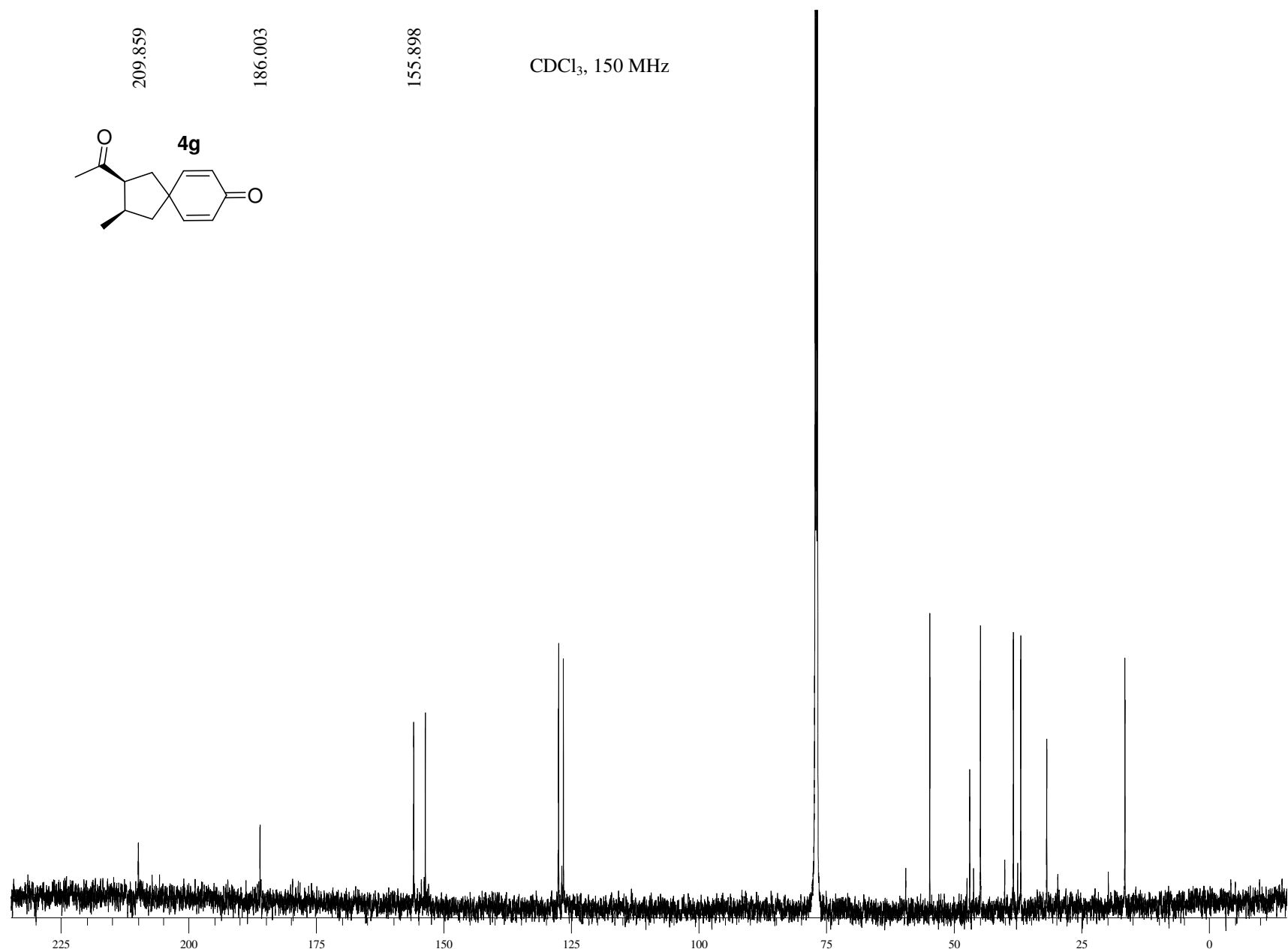


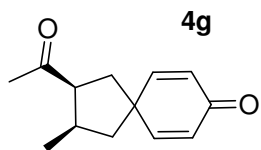
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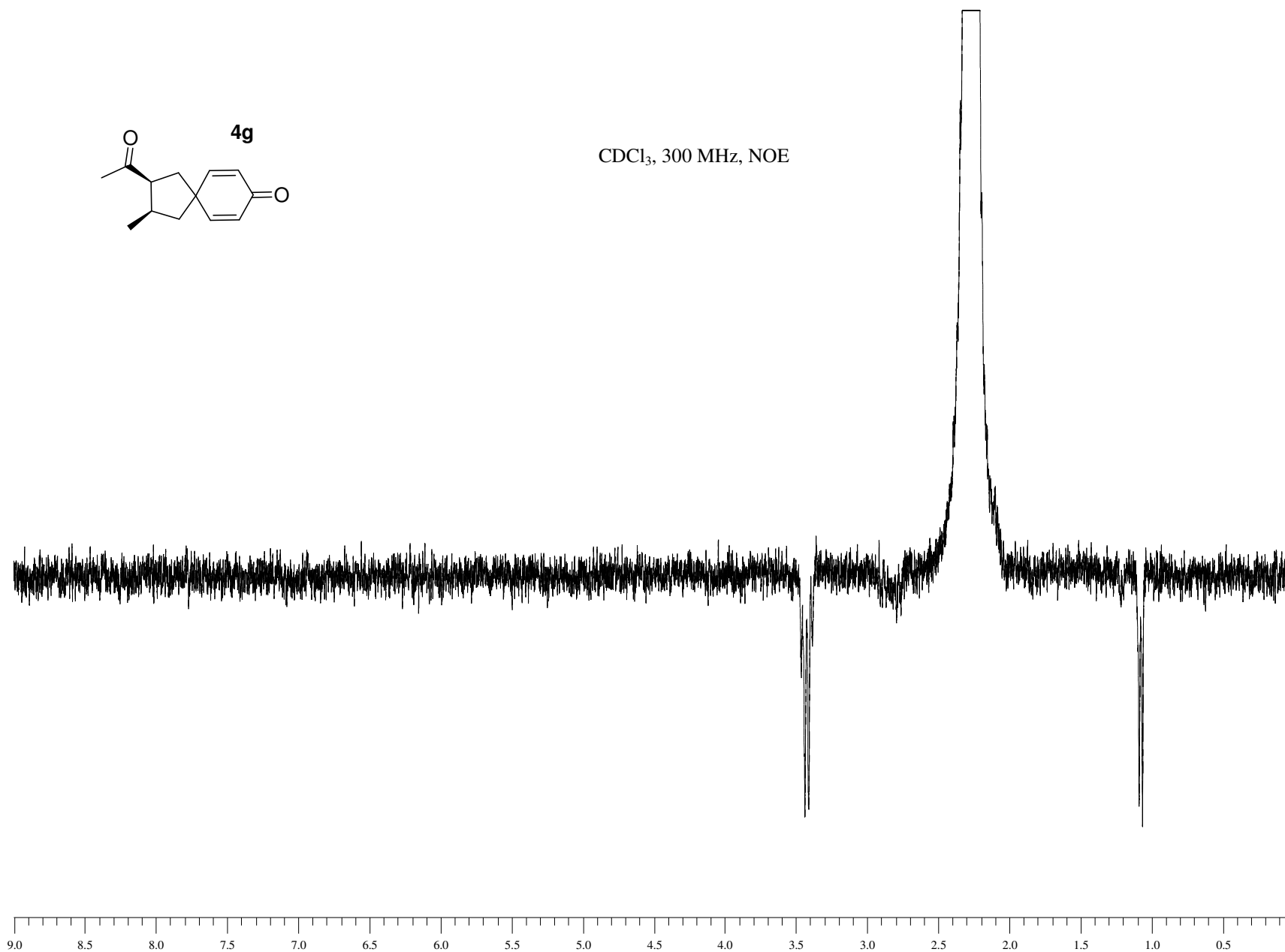


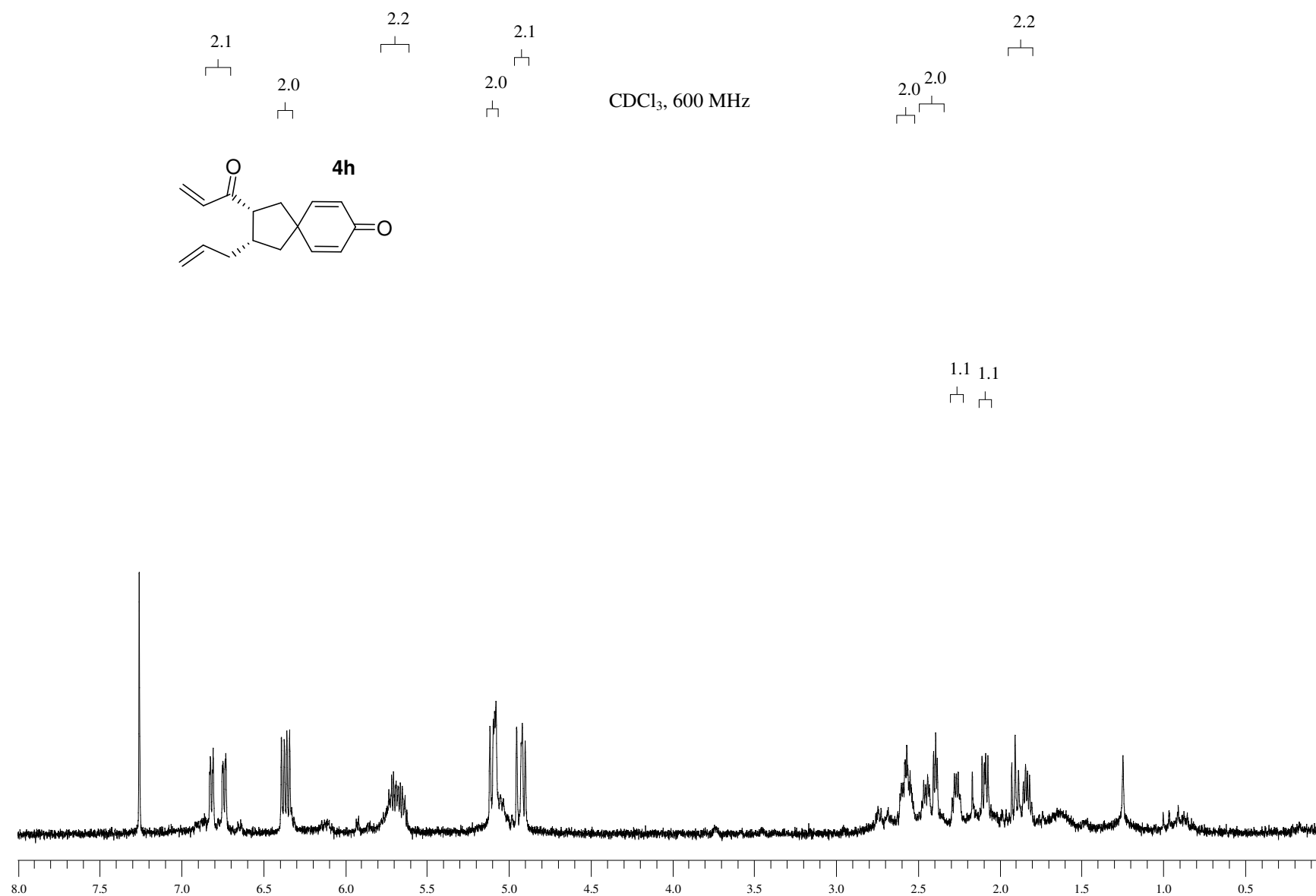




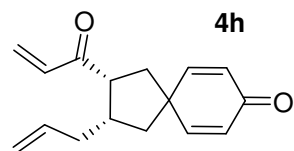


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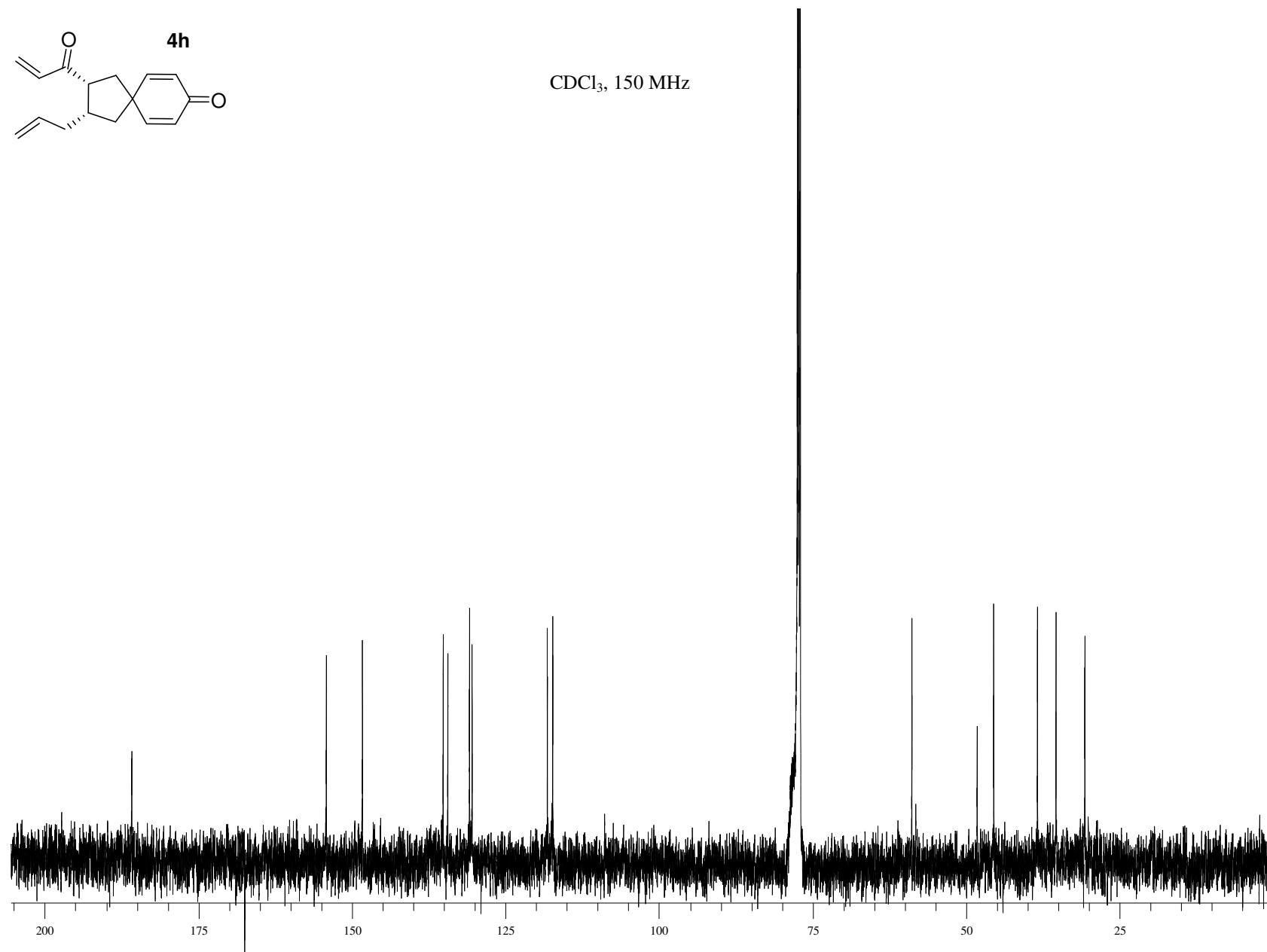


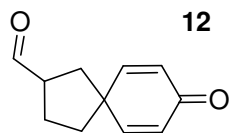




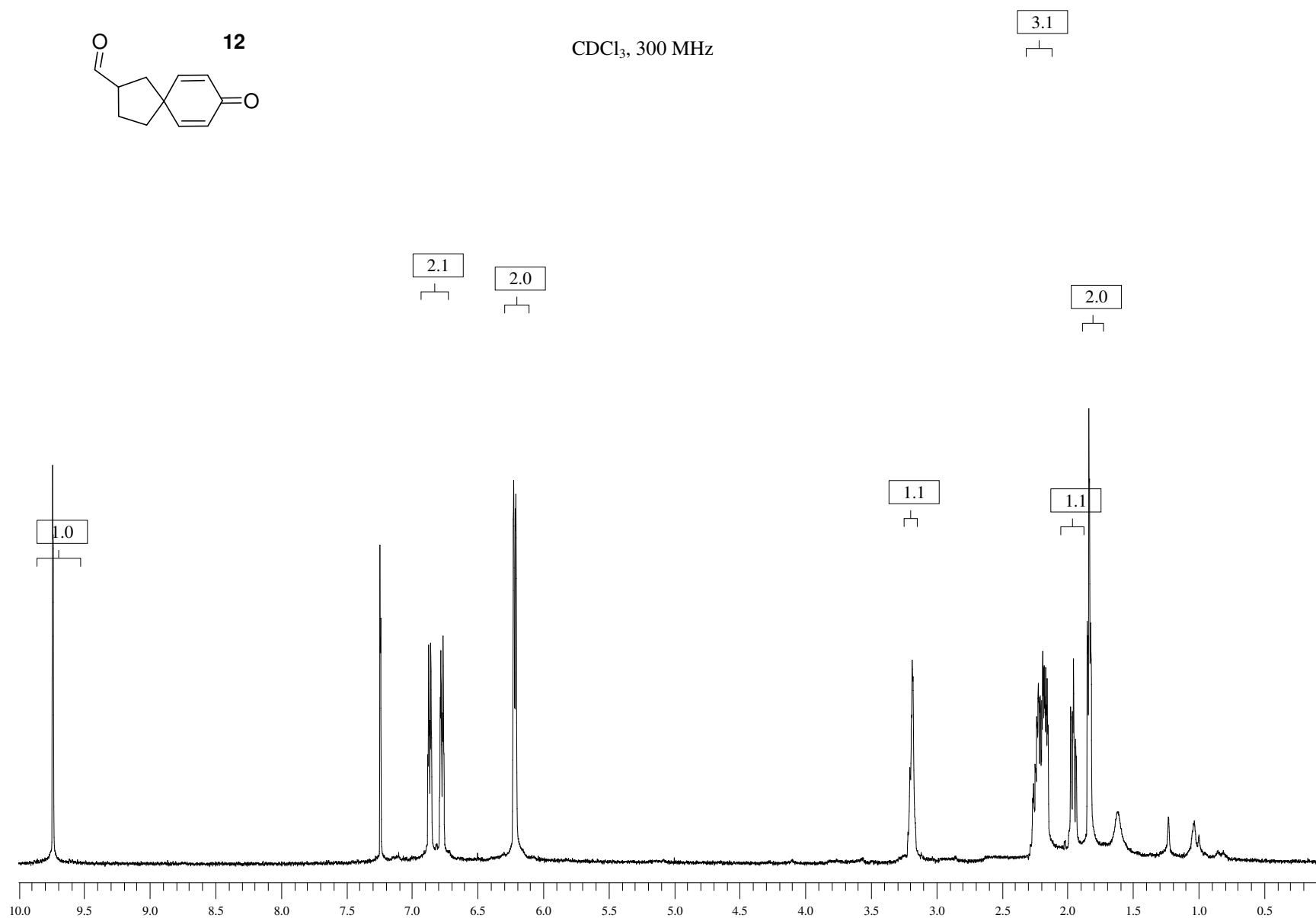


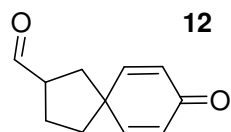
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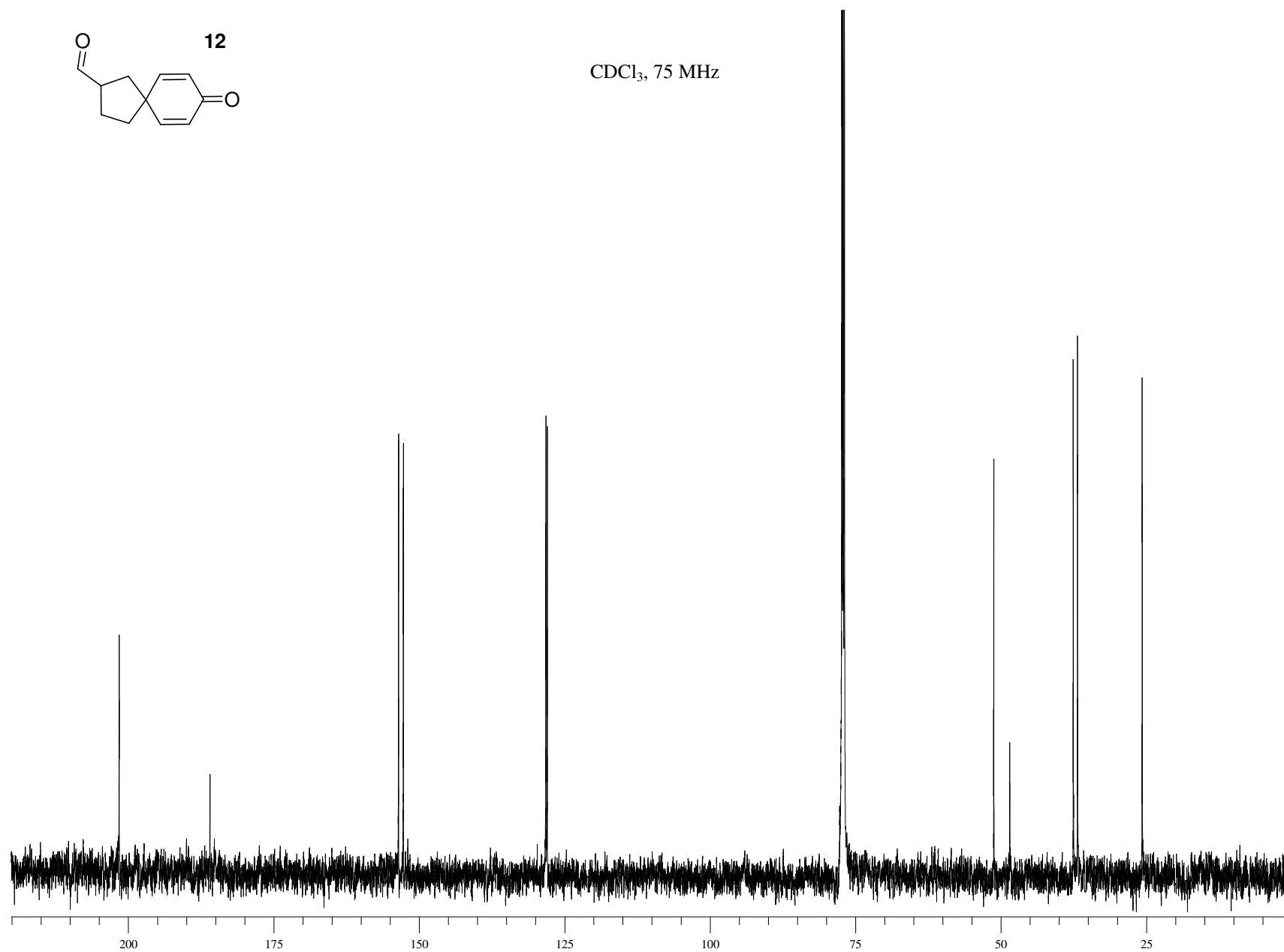


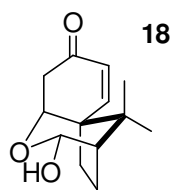
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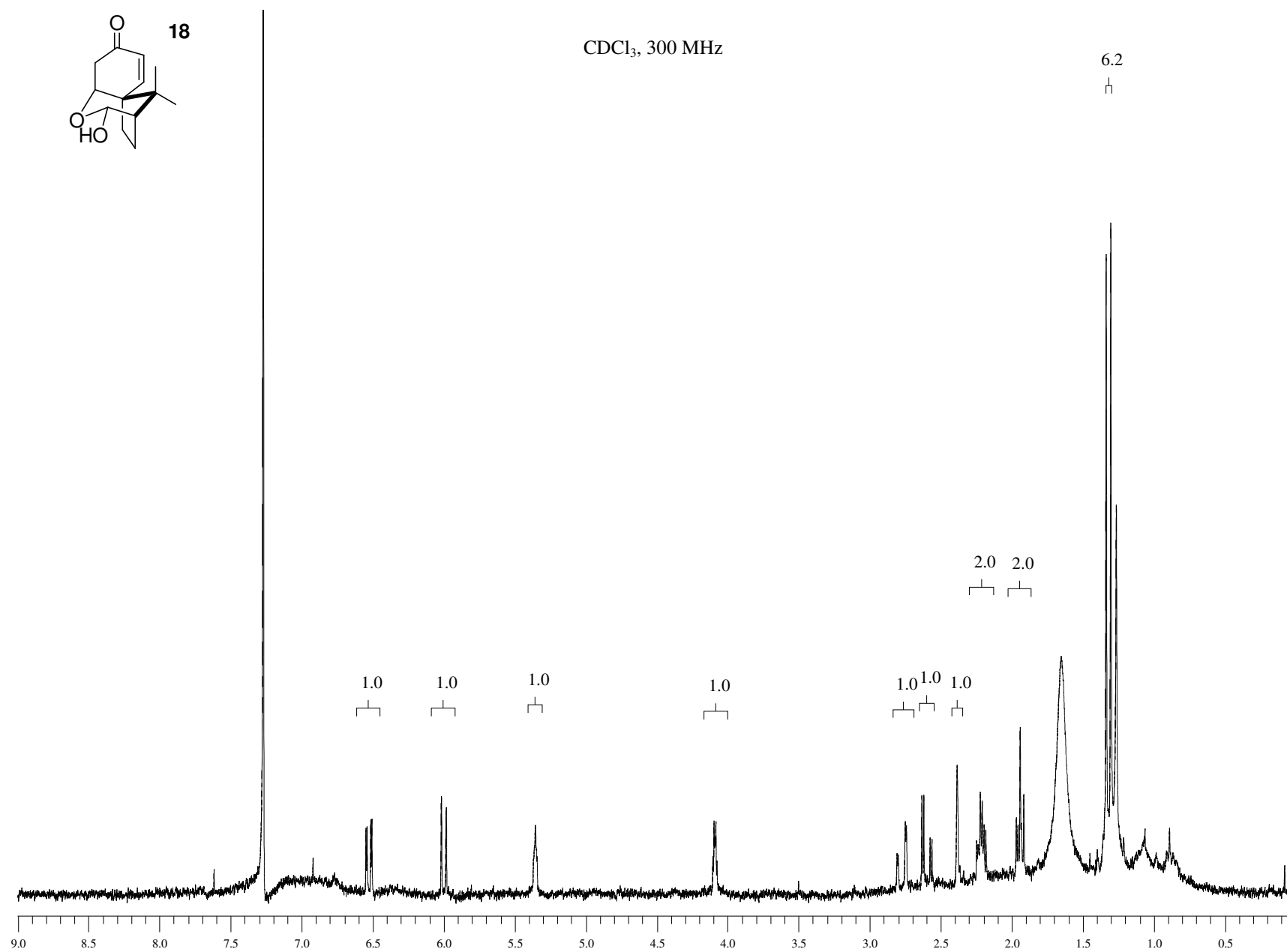


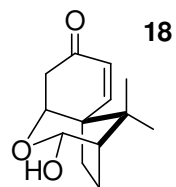


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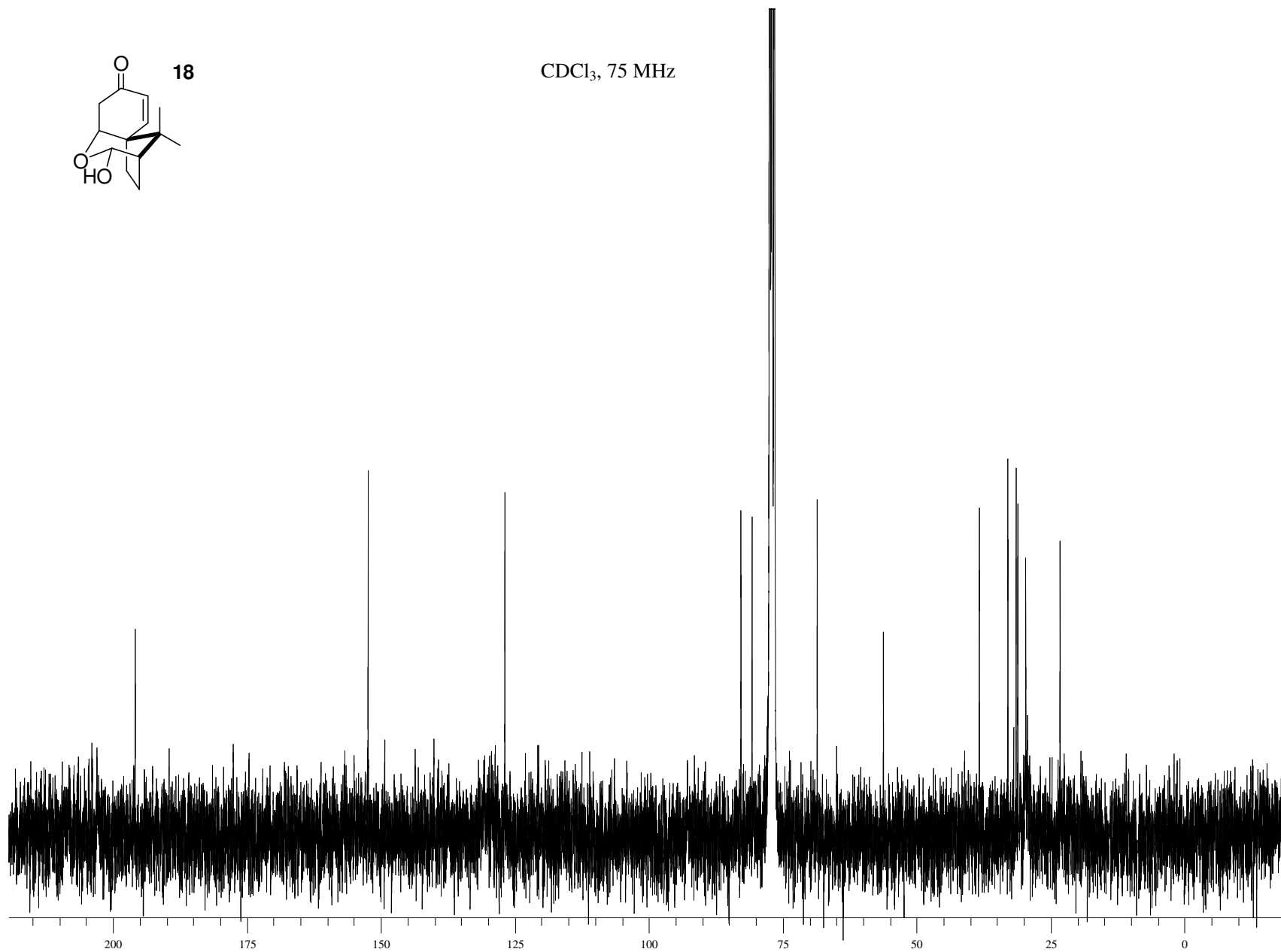
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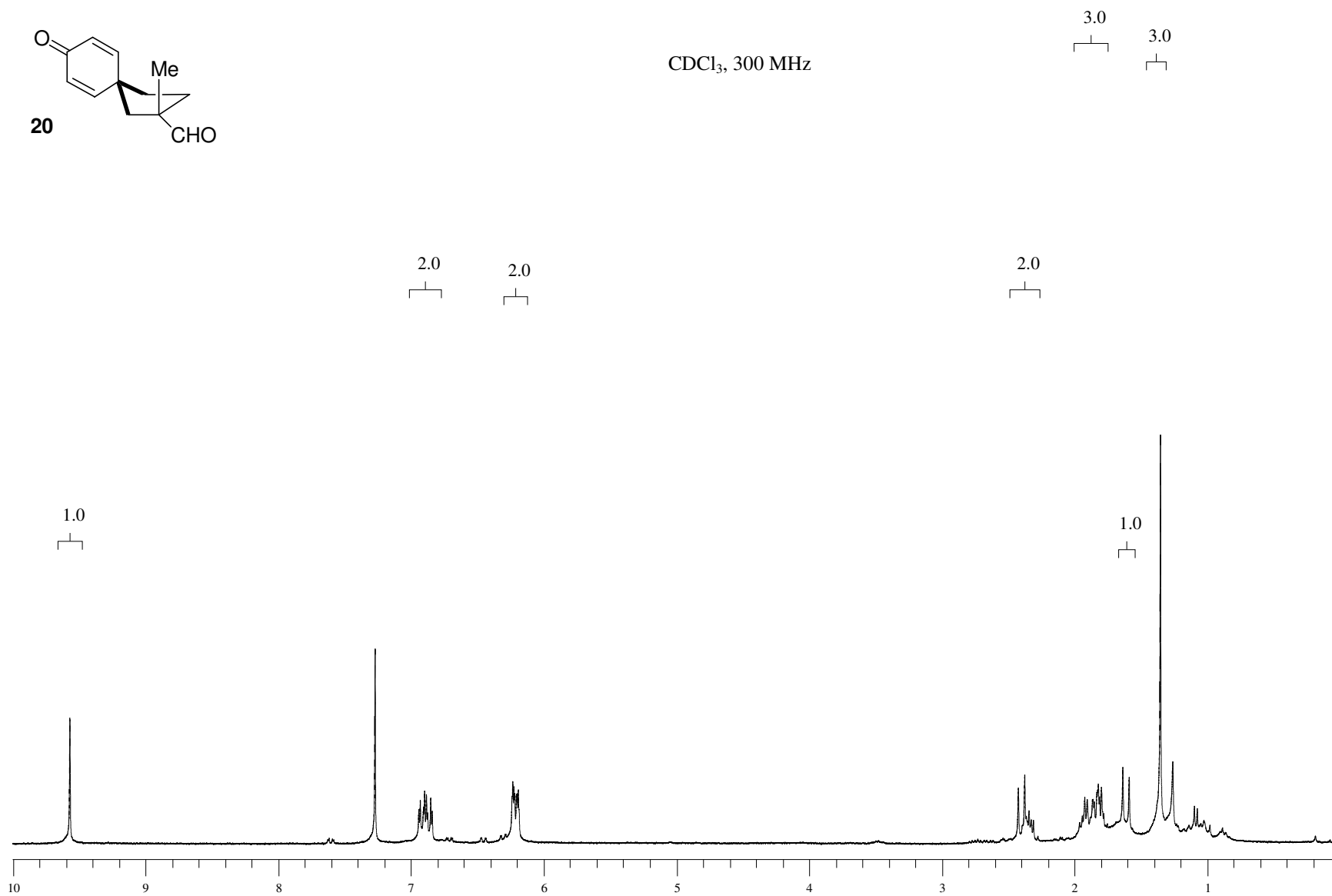
1

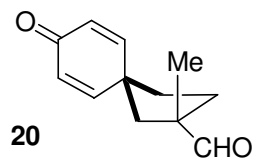




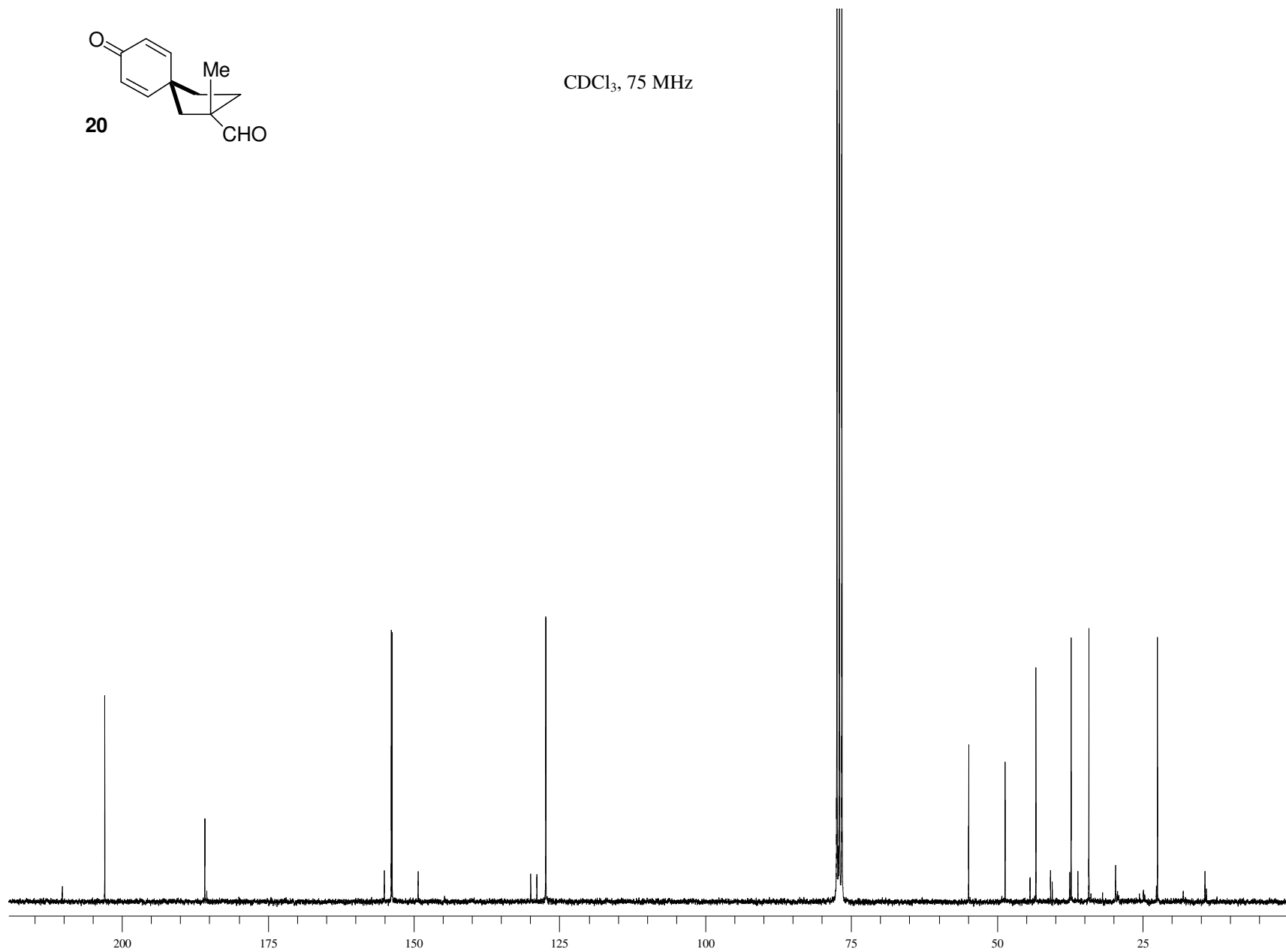
CDCl<sub>3</sub>, 75 MHz

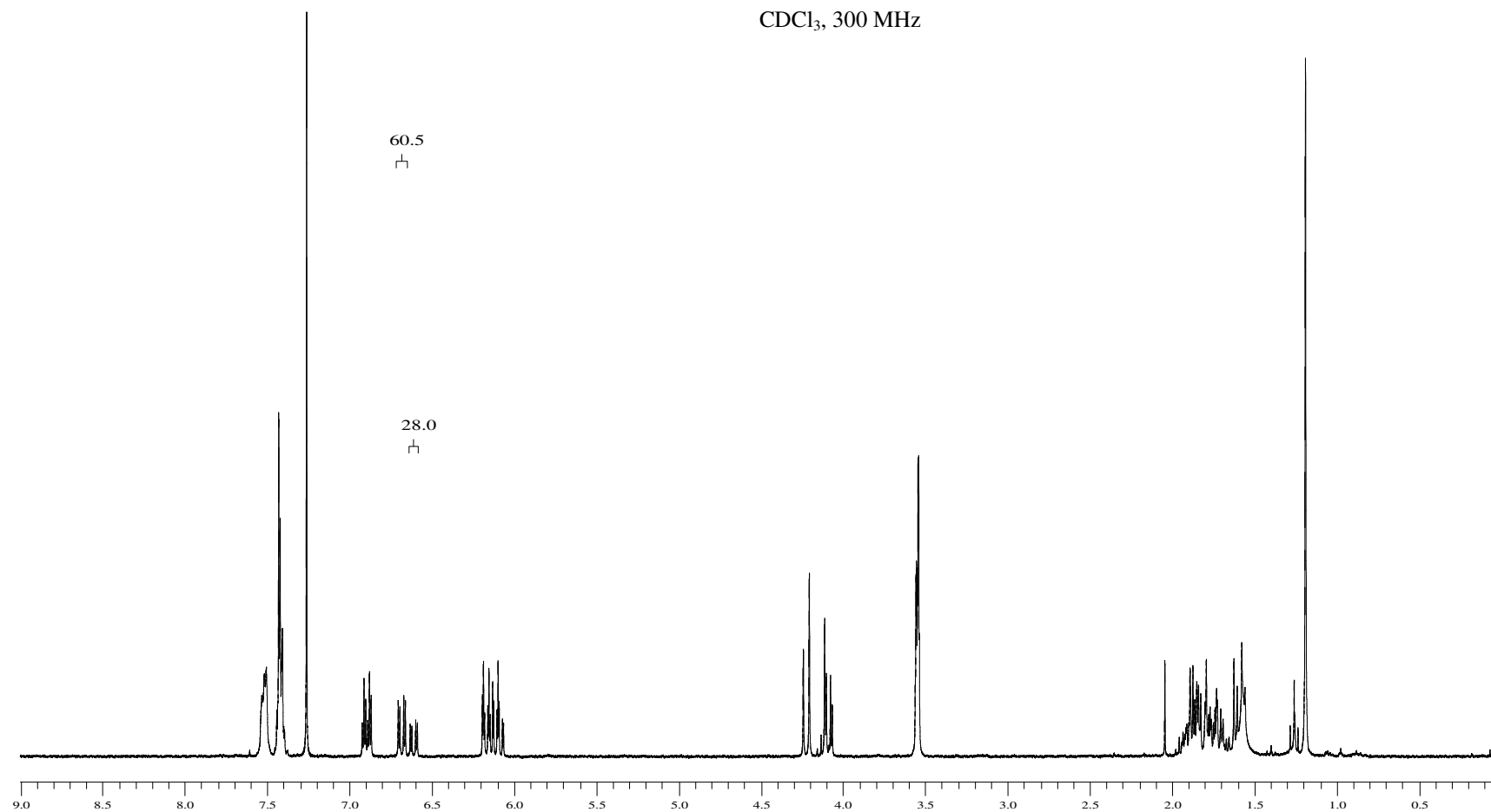
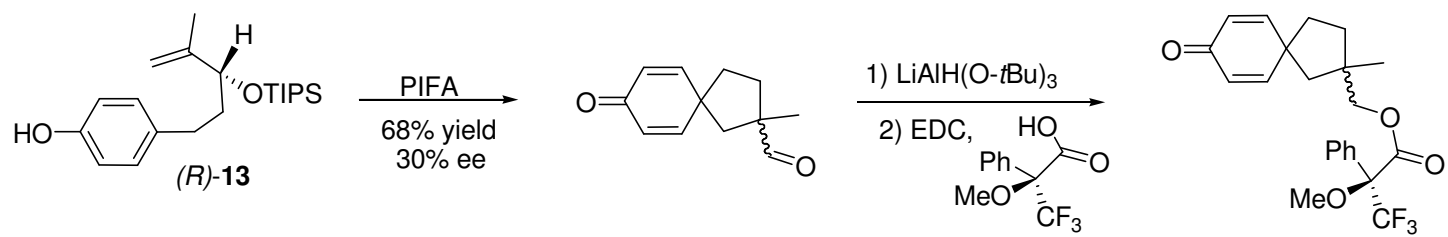




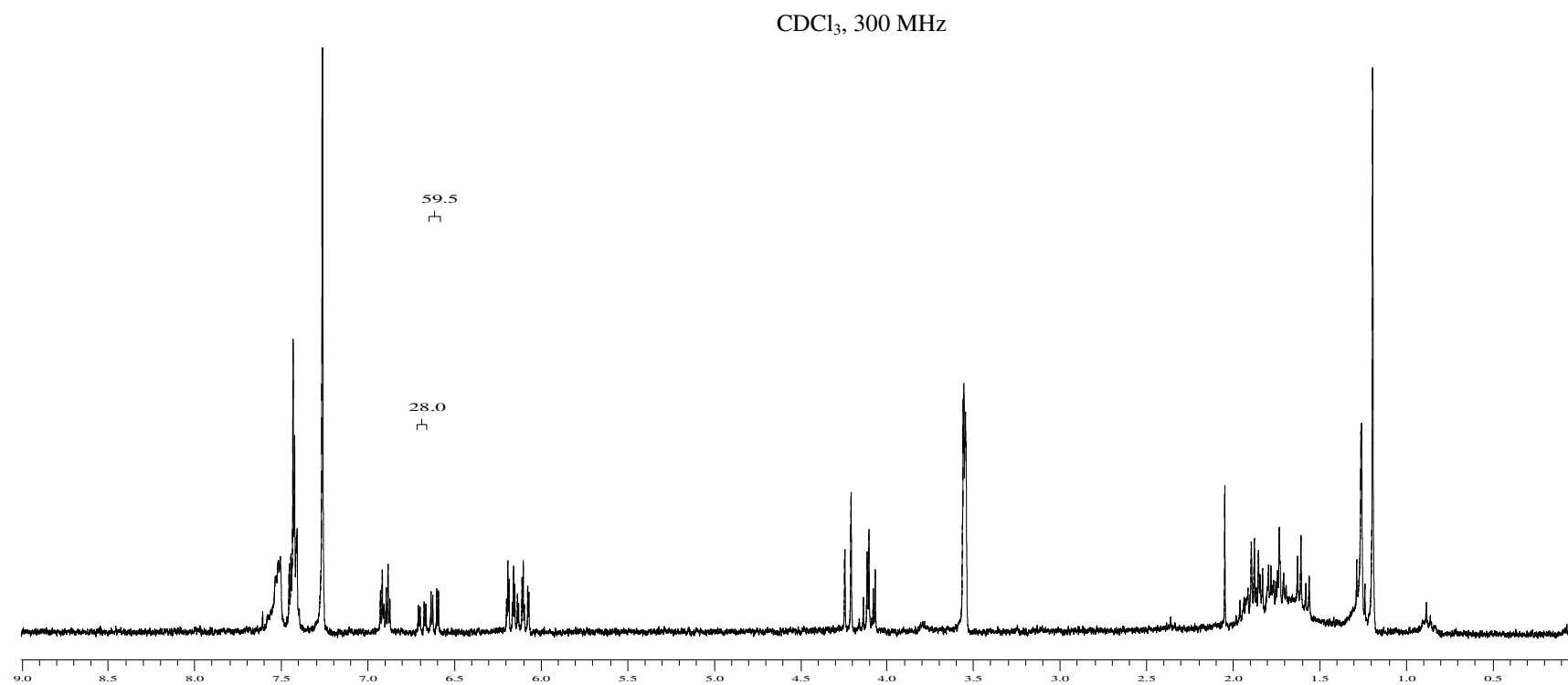
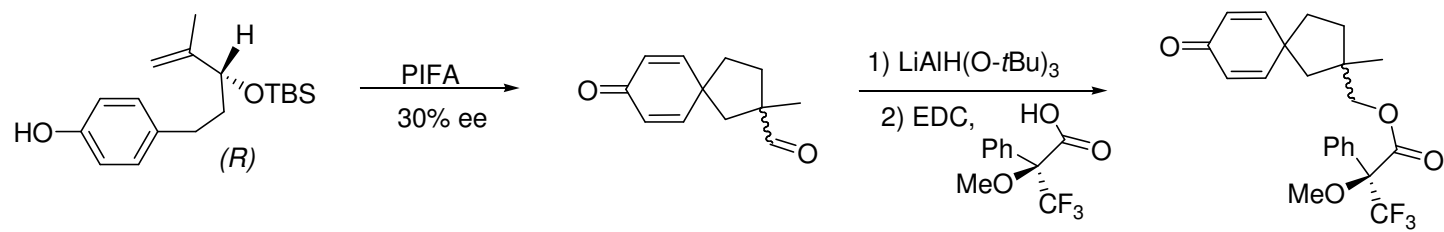


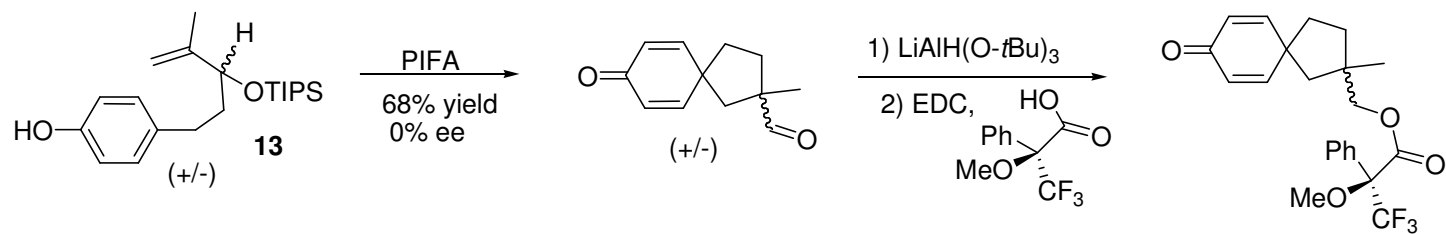
CDCl<sub>3</sub>, 75 MHz



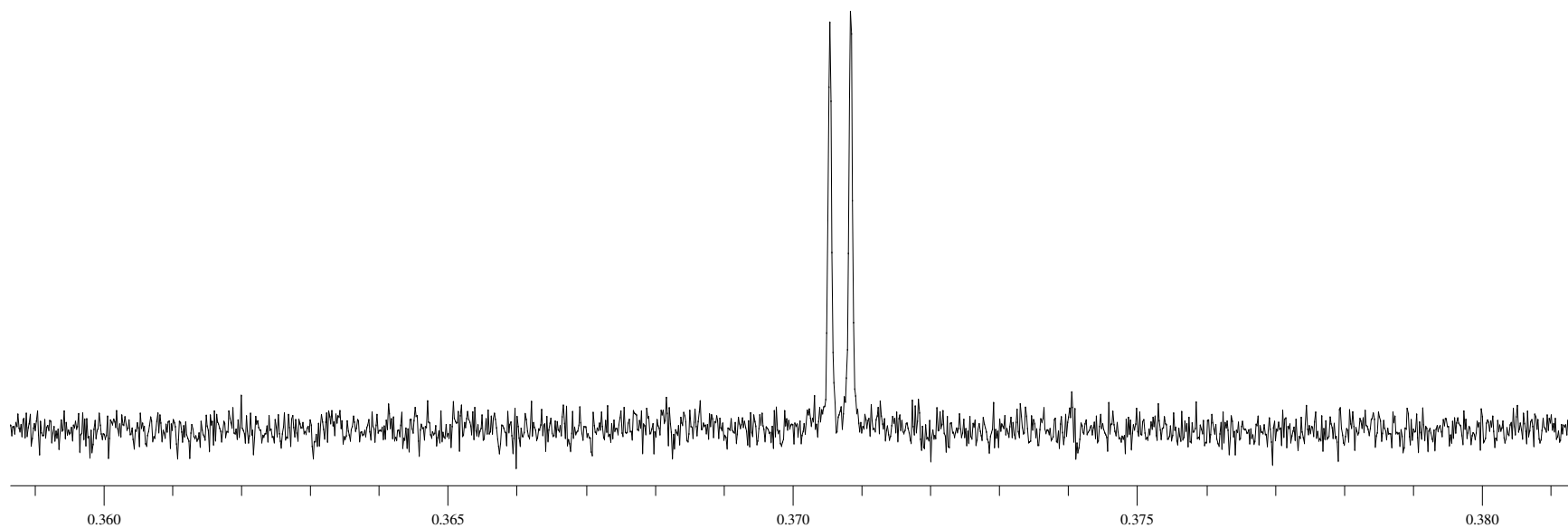


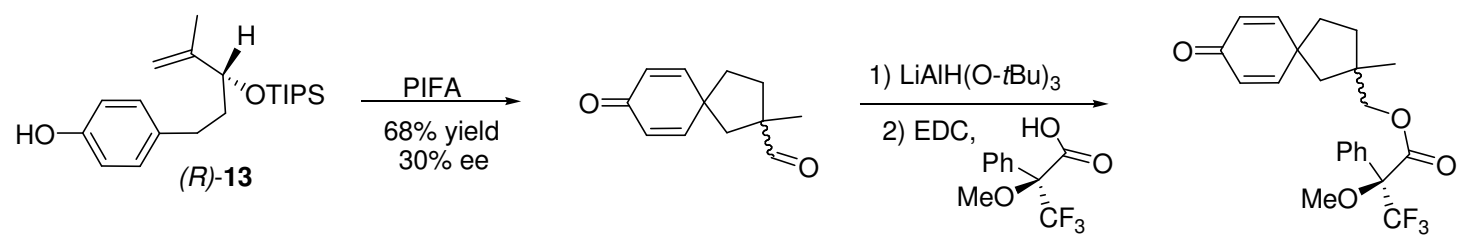






$\text{CDCl}_3$ , 300 MHz,  $^{19}\text{F}$



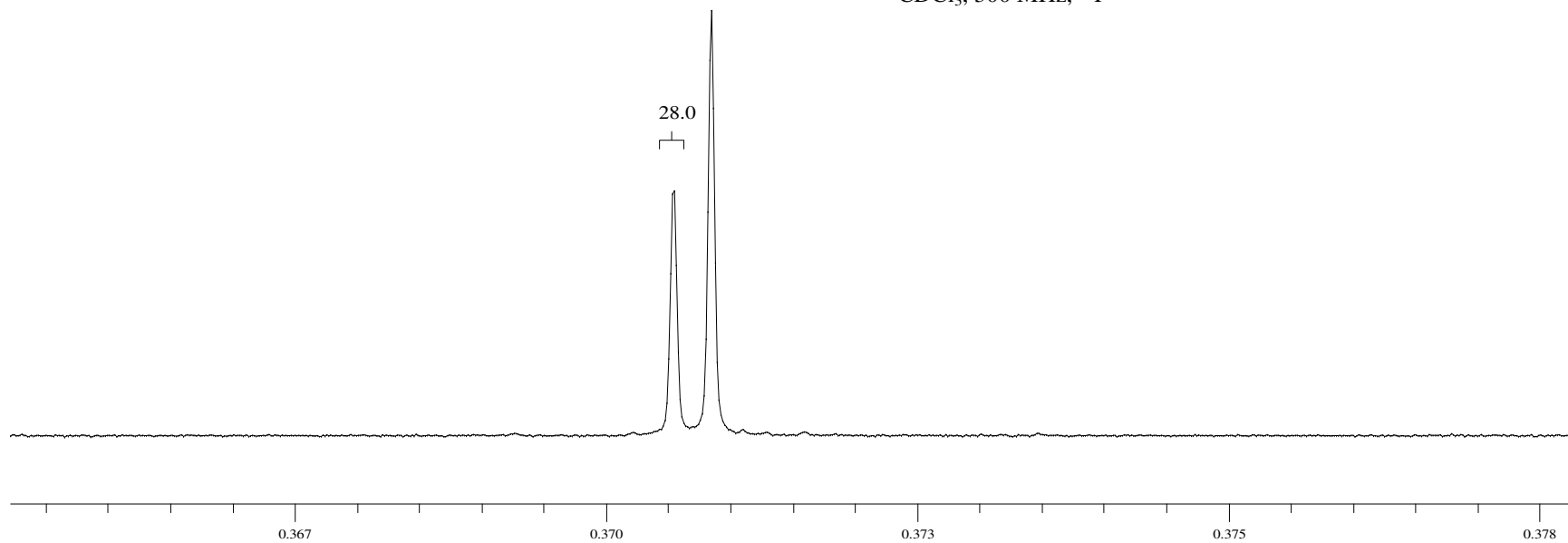


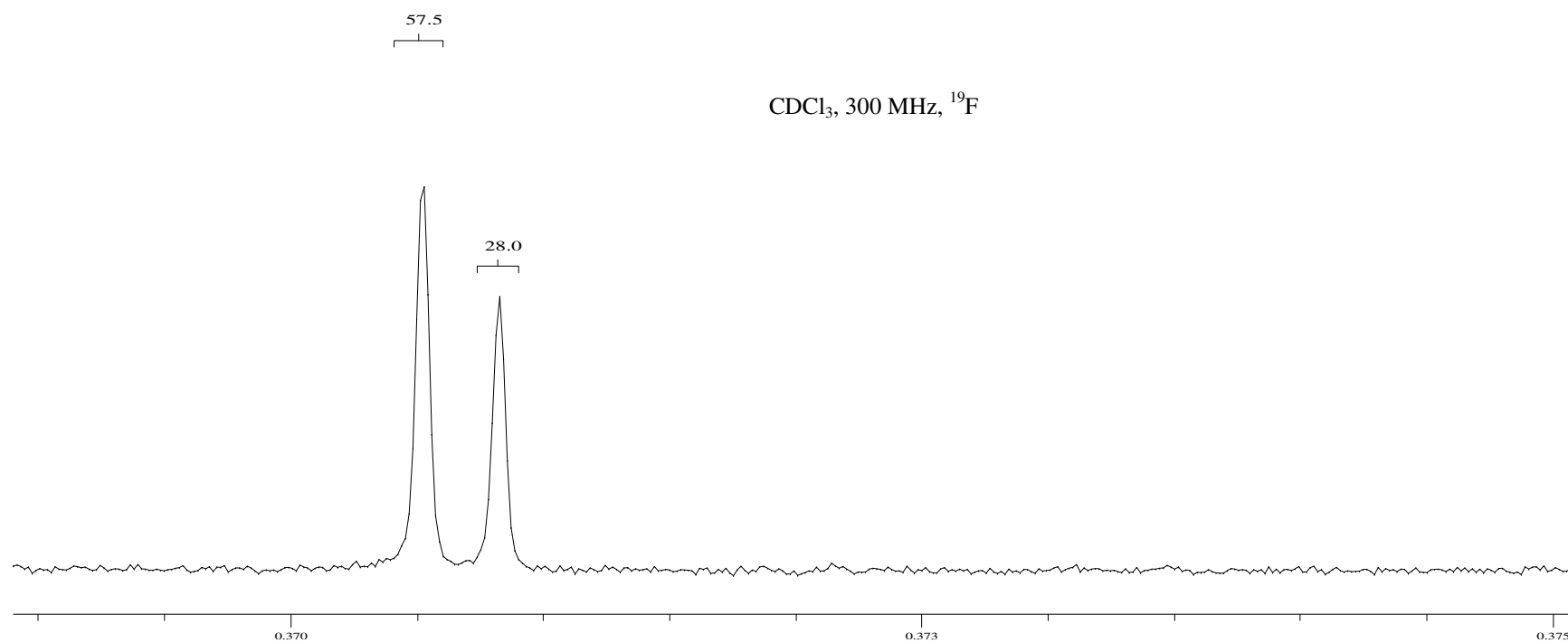
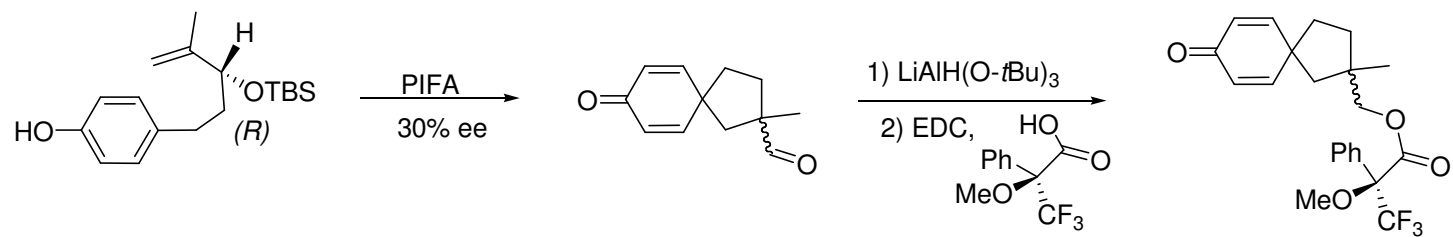
57.0

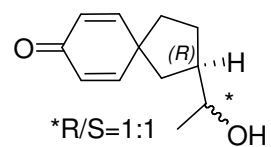
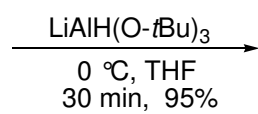
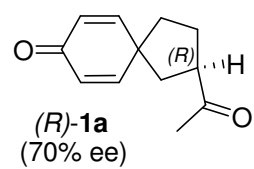


CDCl<sub>3</sub>, 300 MHz, <sup>19</sup>F

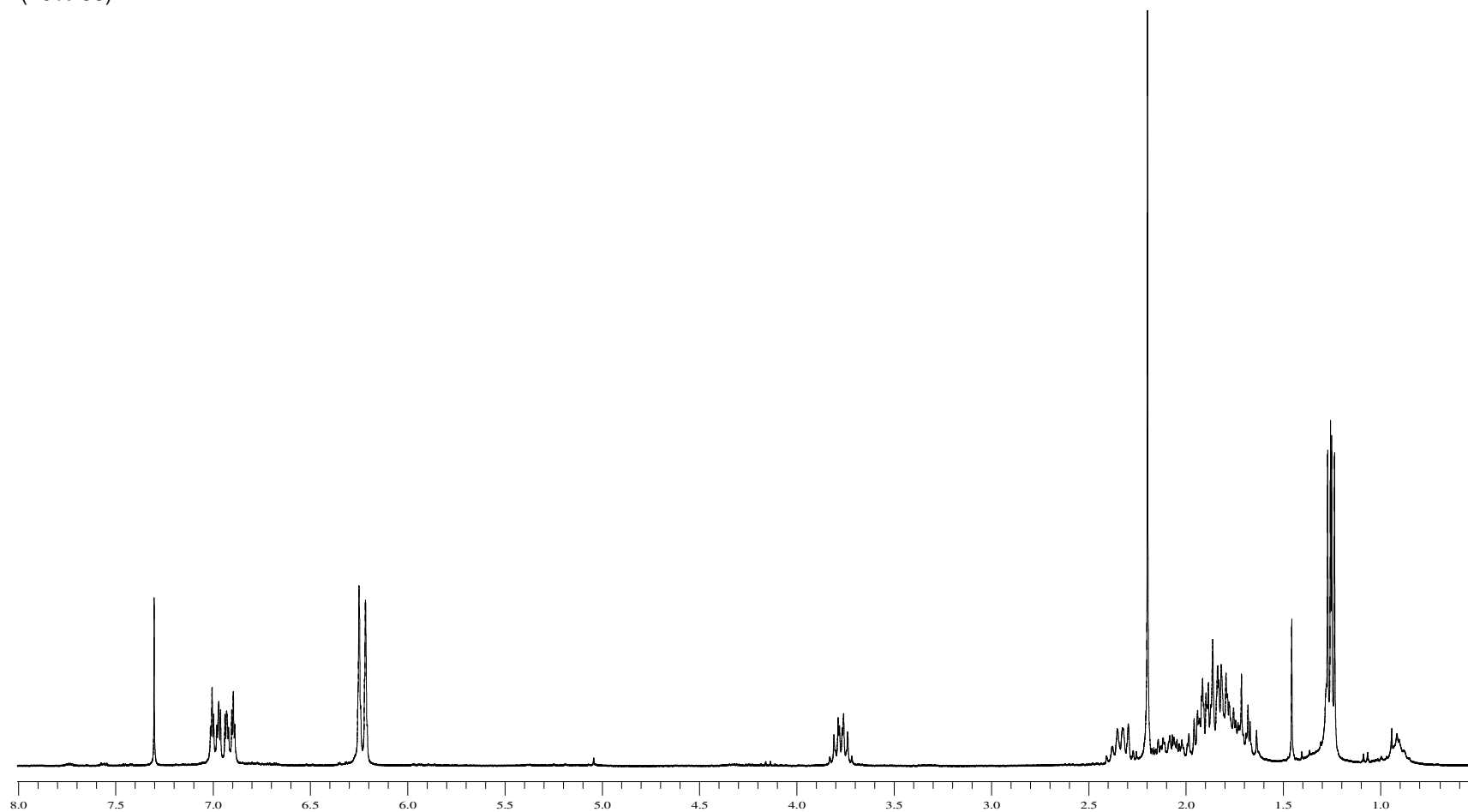
28.0

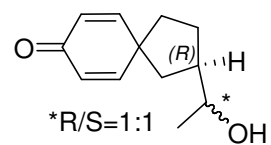
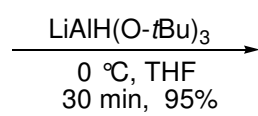
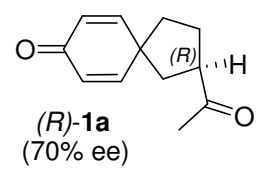




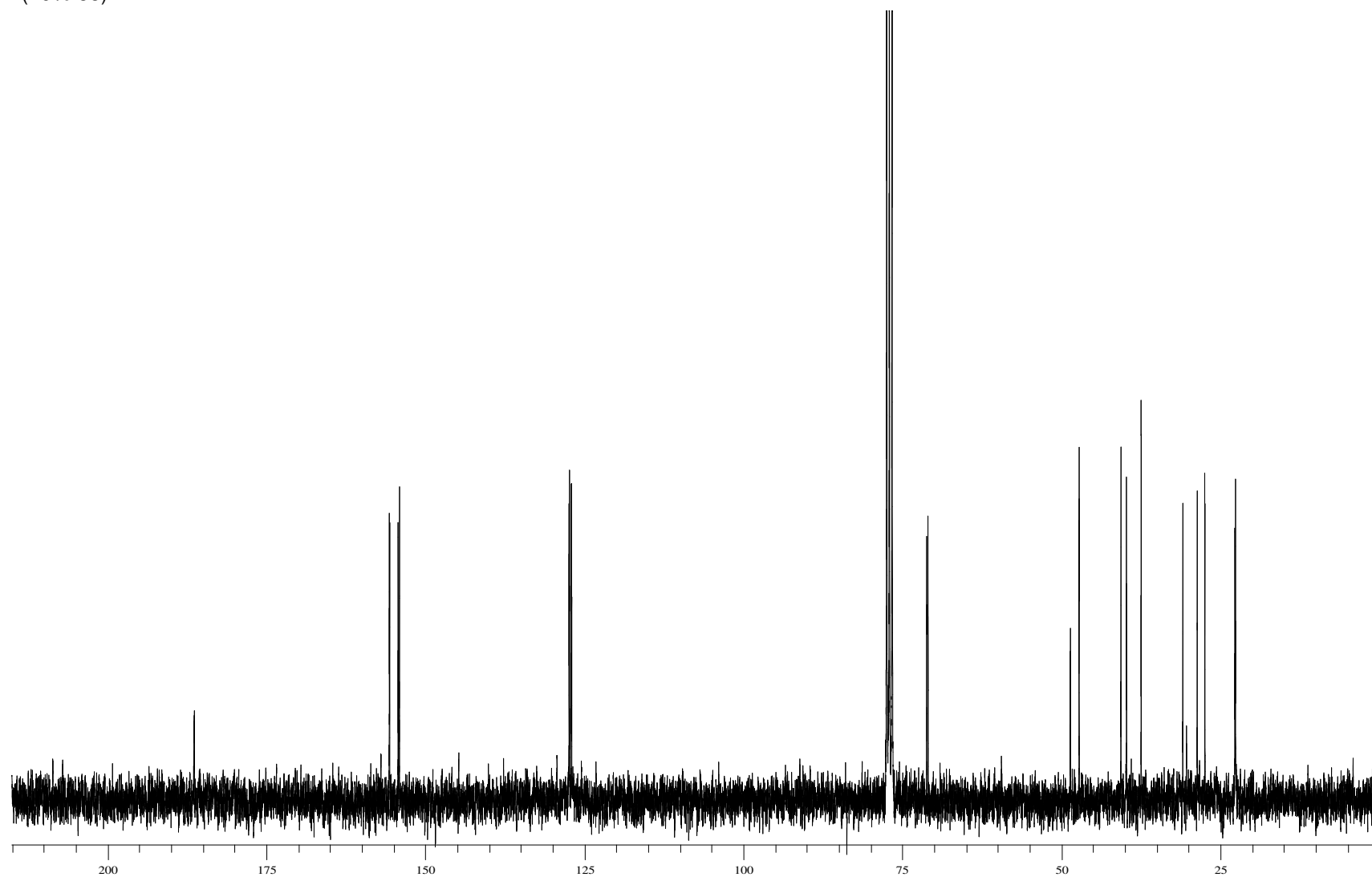


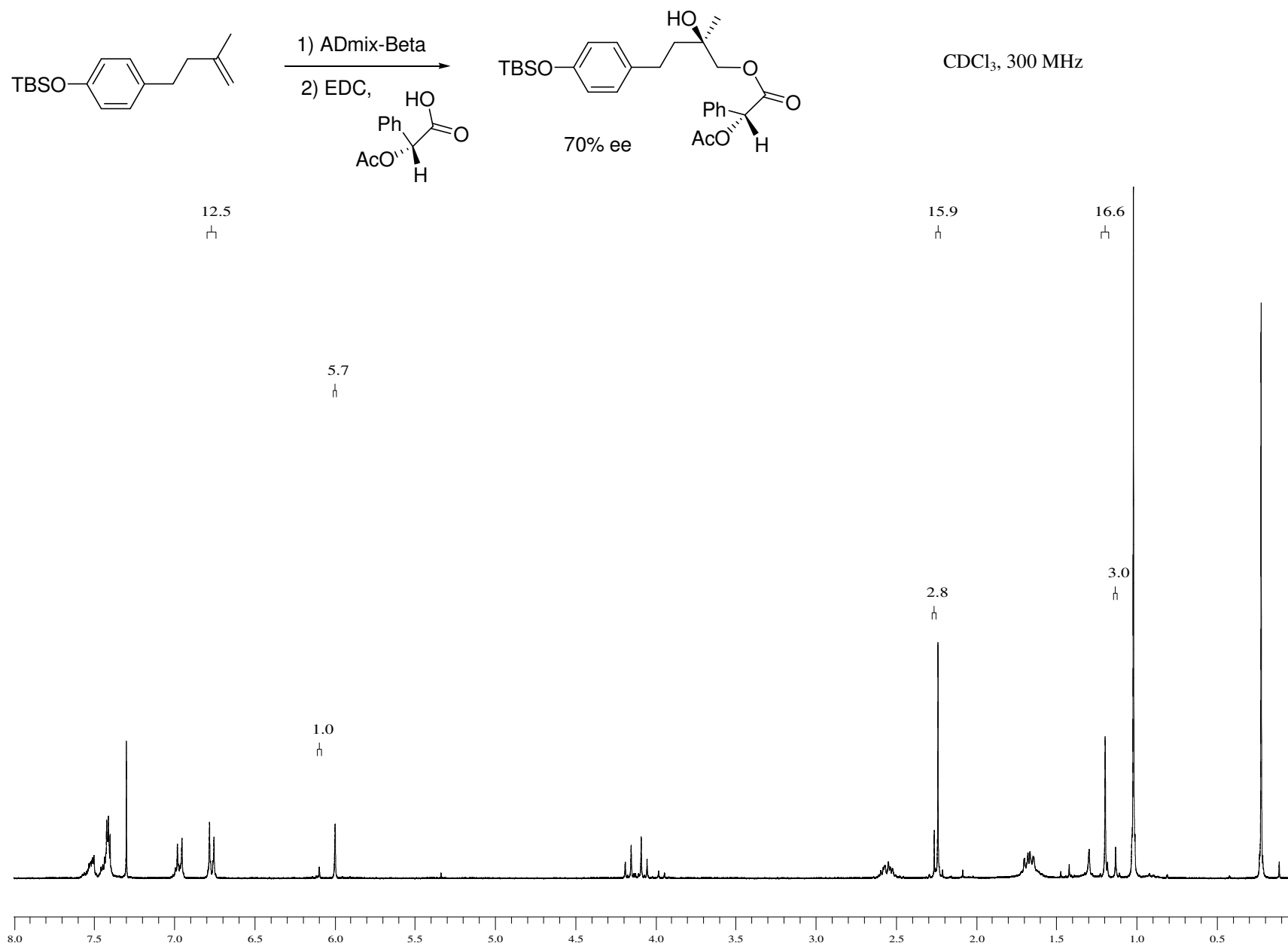
CDCl<sub>3</sub>, 300 MHz

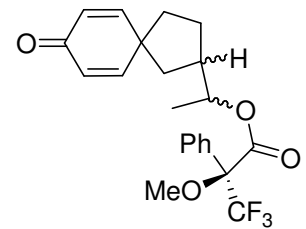
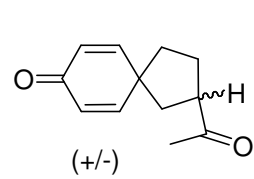




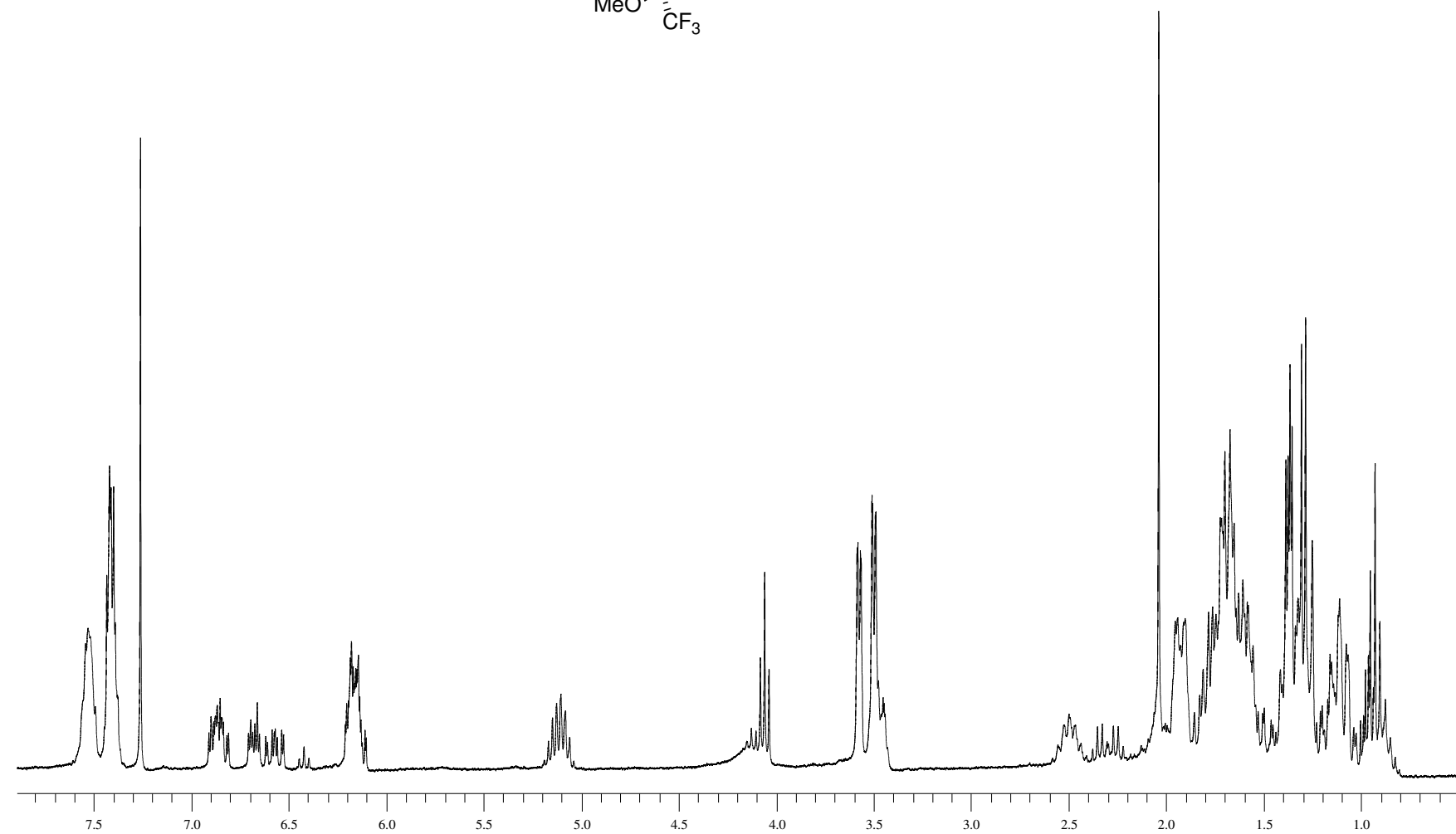
CDCl<sub>3</sub>, 75 MHz



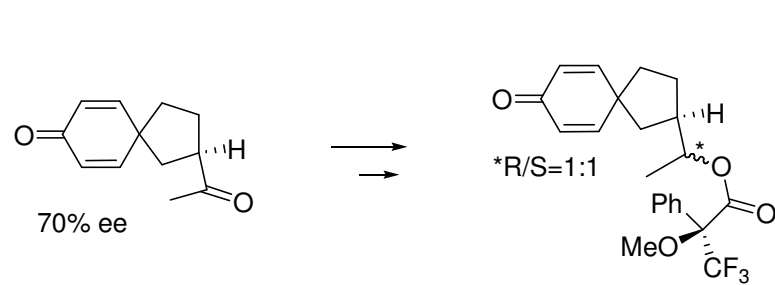




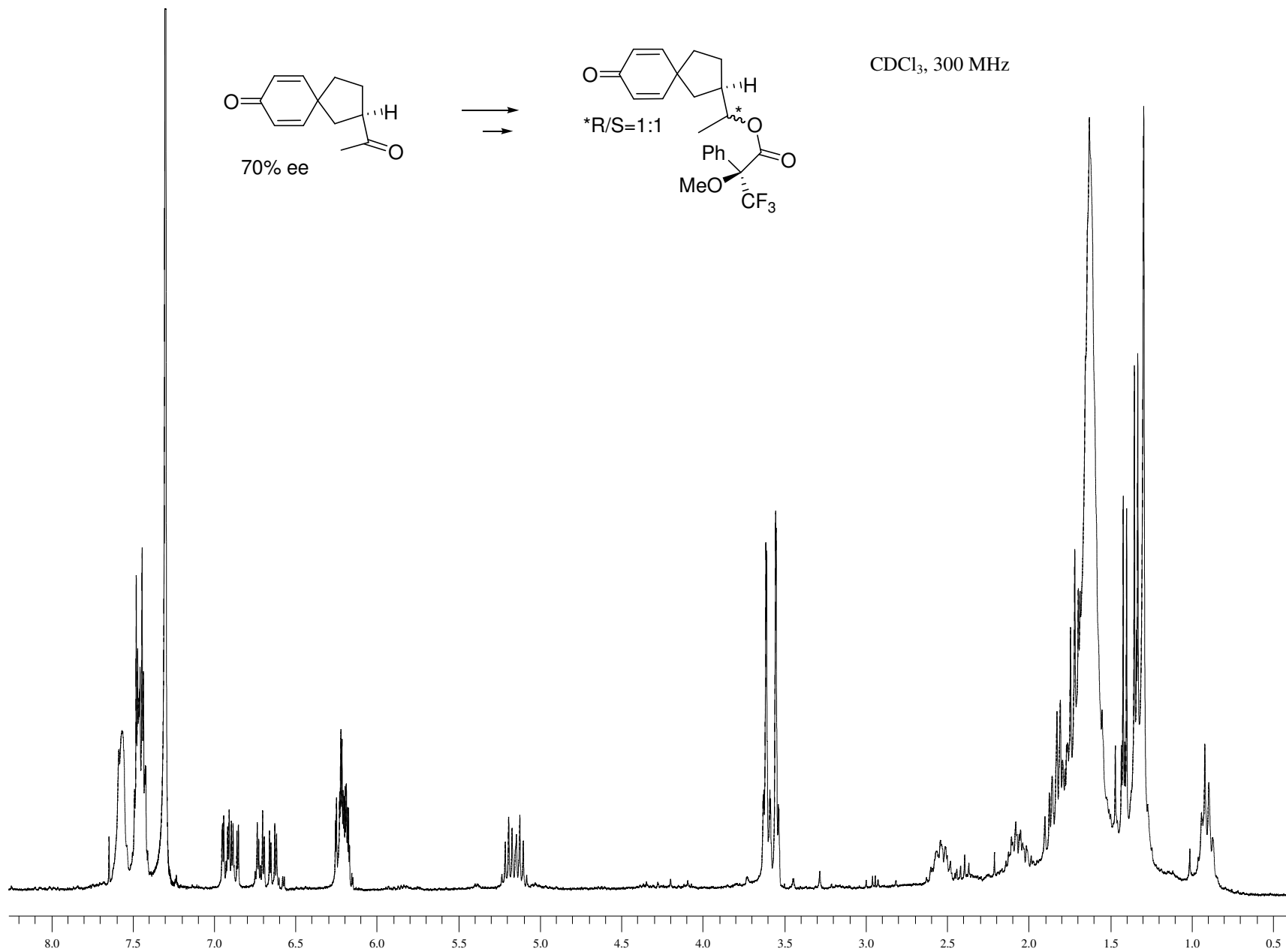
CDCl<sub>3</sub>, 300 MHz

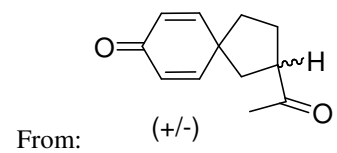




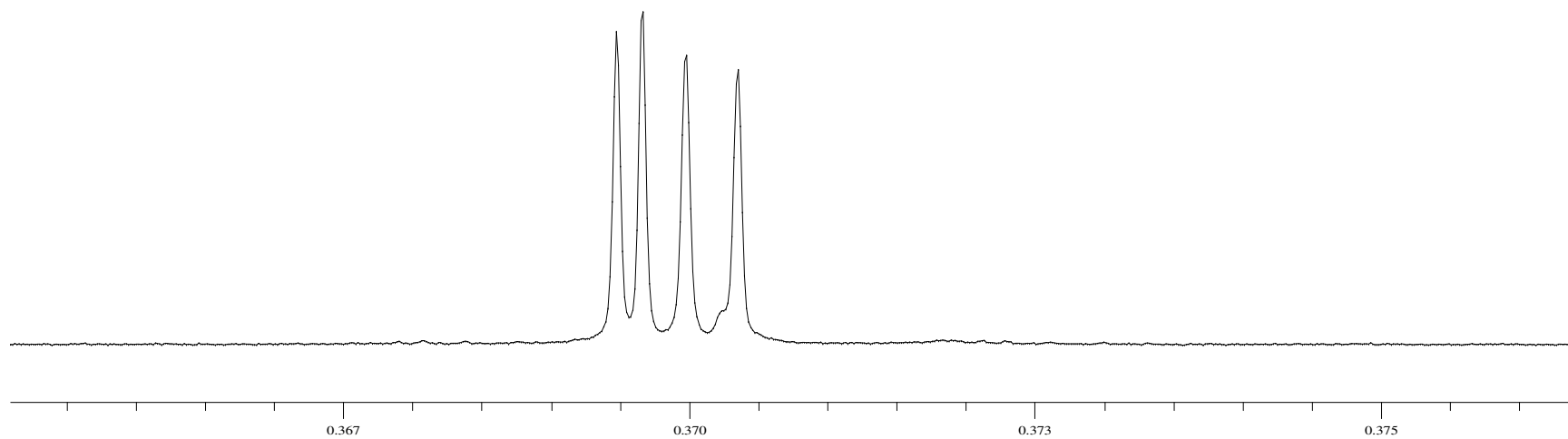
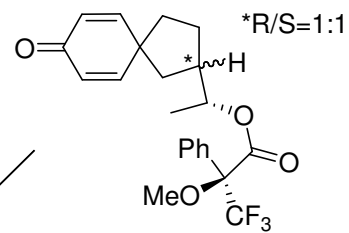
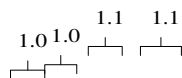
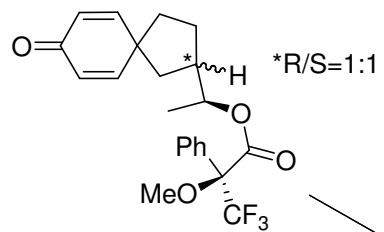


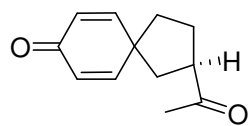
CDCl<sub>3</sub>, 300 MHz





CDCl<sub>3</sub>, 300 MHz, <sup>19</sup>F

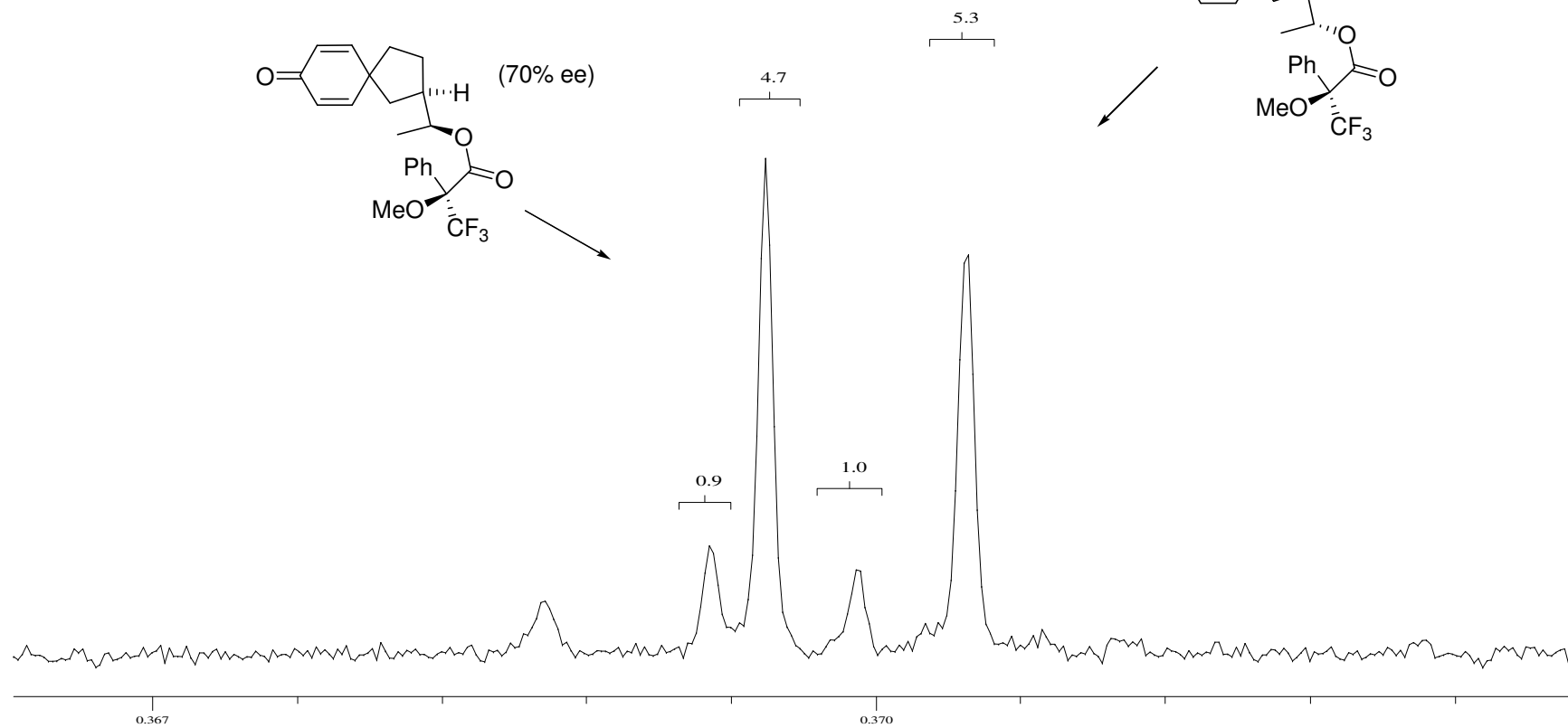
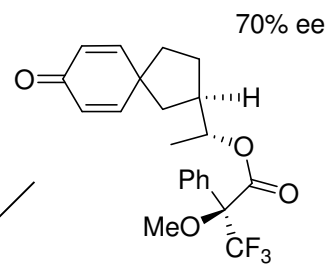
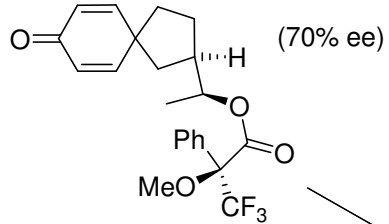


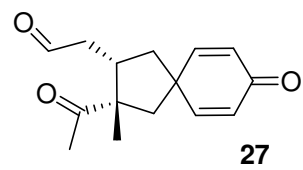


70% ee

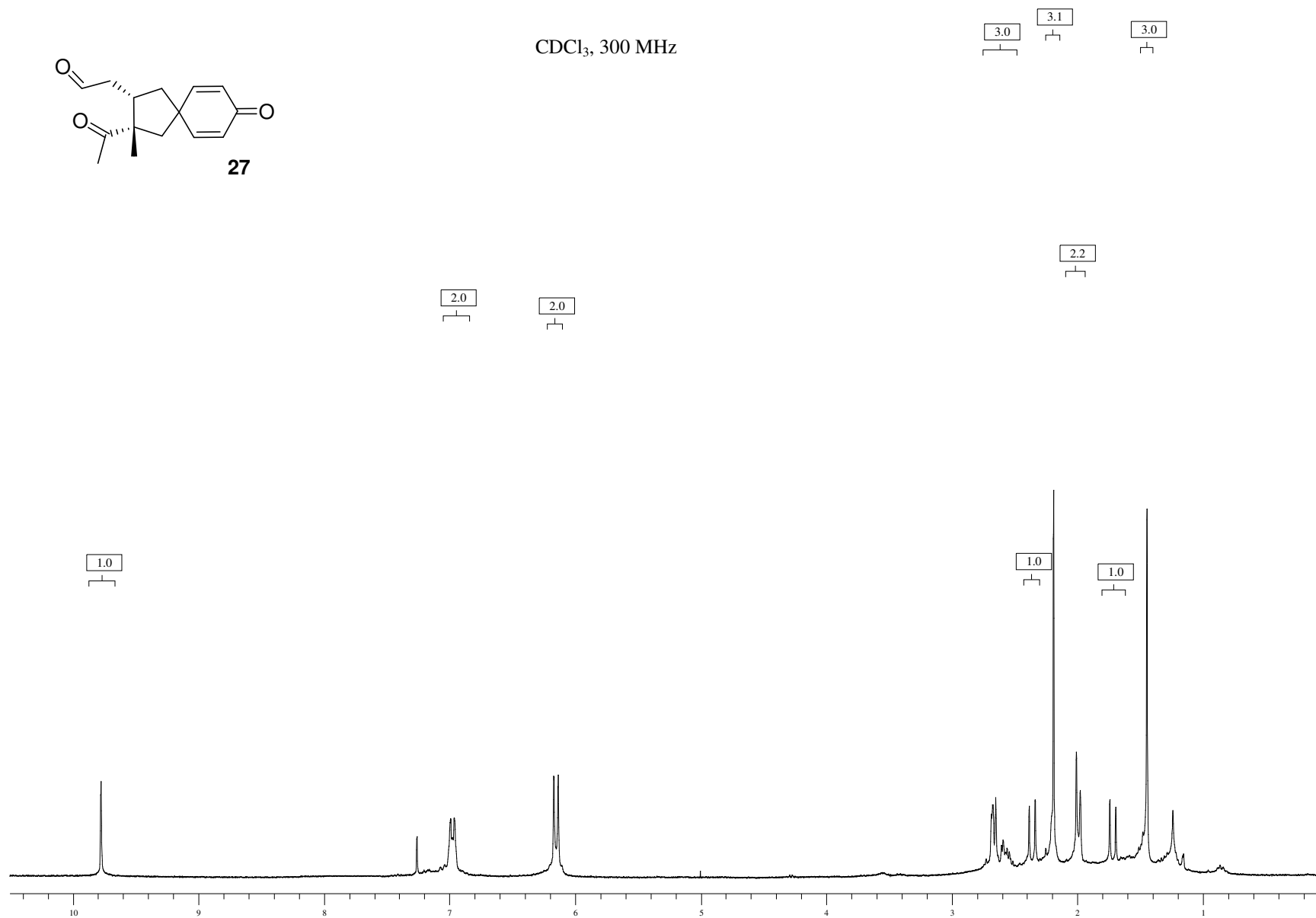
From

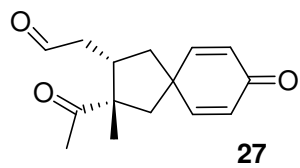
CDCl<sub>3</sub>, 300 MHz, <sup>19</sup>F



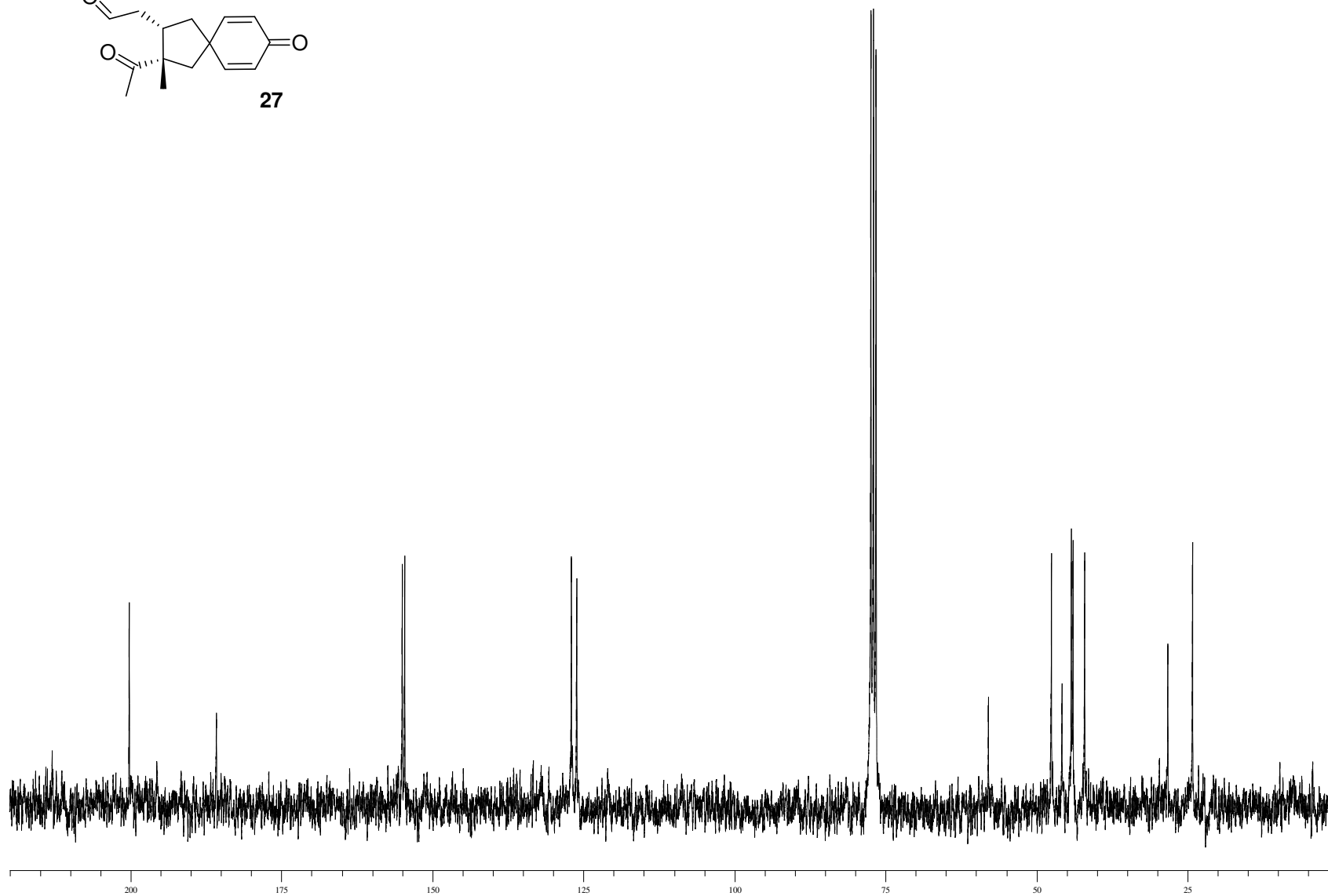


CDCl<sub>3</sub>, 300 MHz



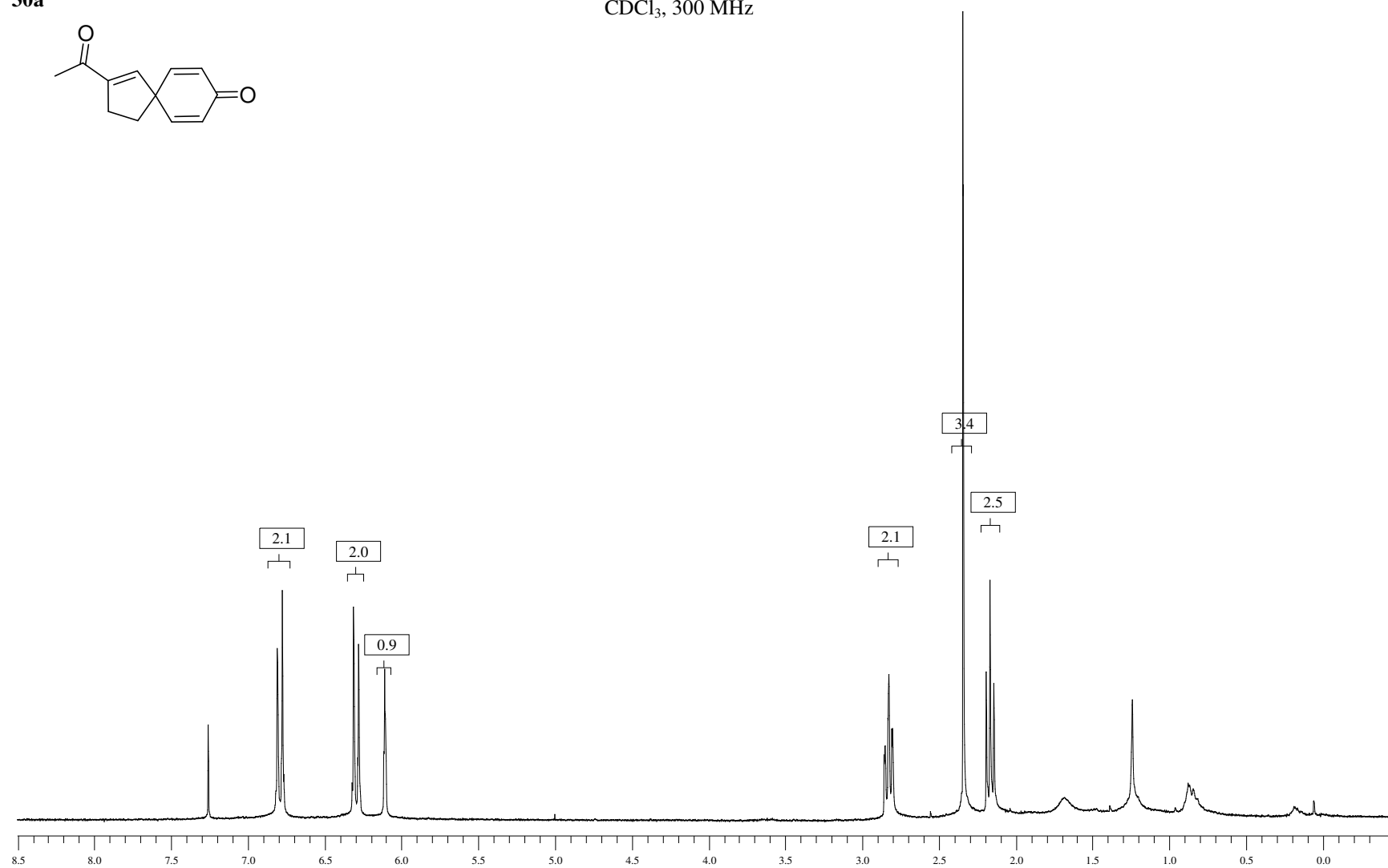
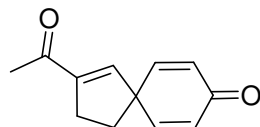


CDCl<sub>3</sub>, 75 MHz

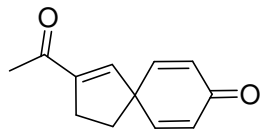


**30a**

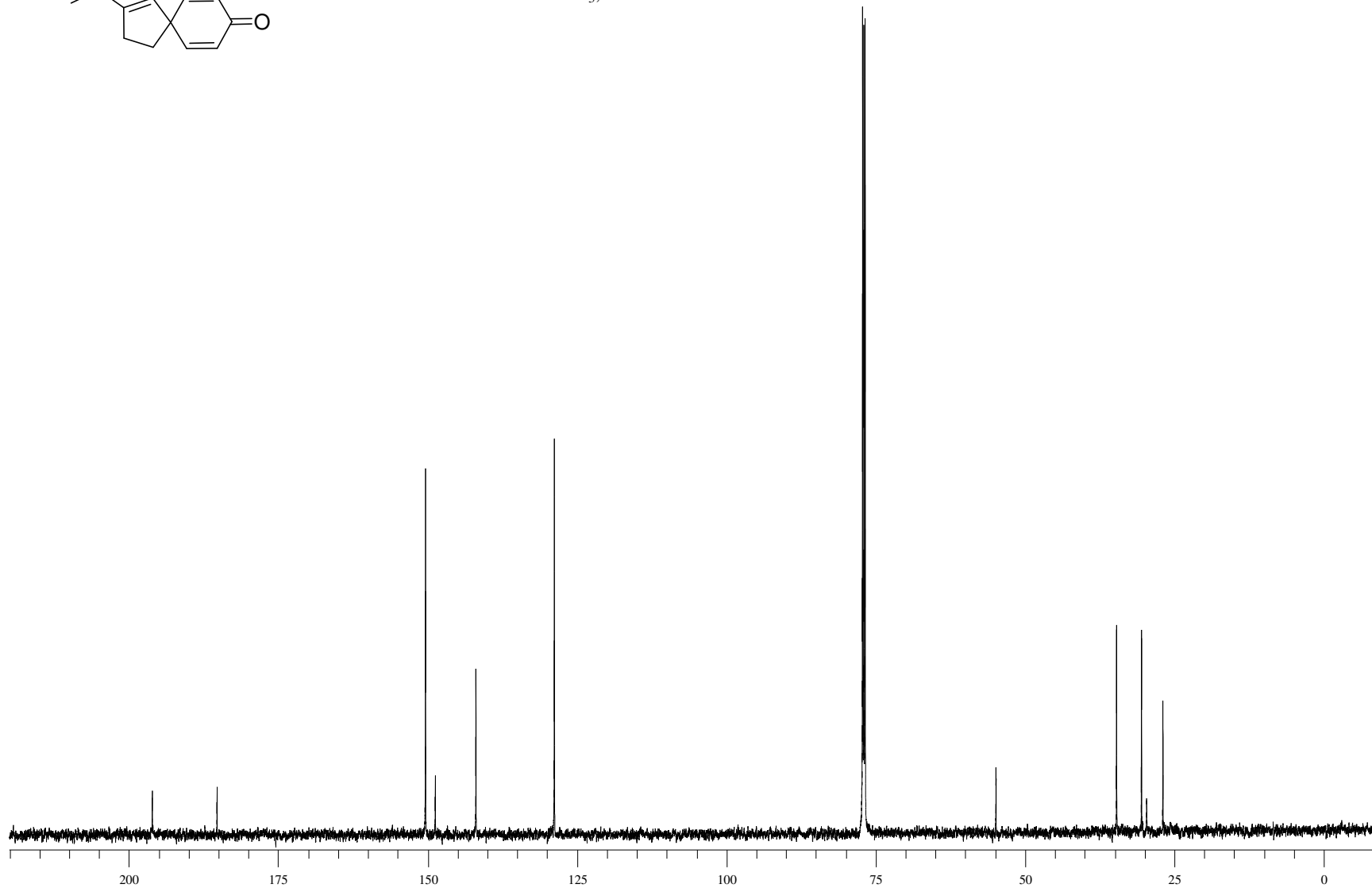
CDCl<sub>3</sub>, 300 MHz



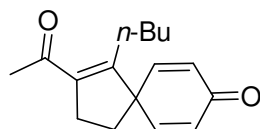
**30a**



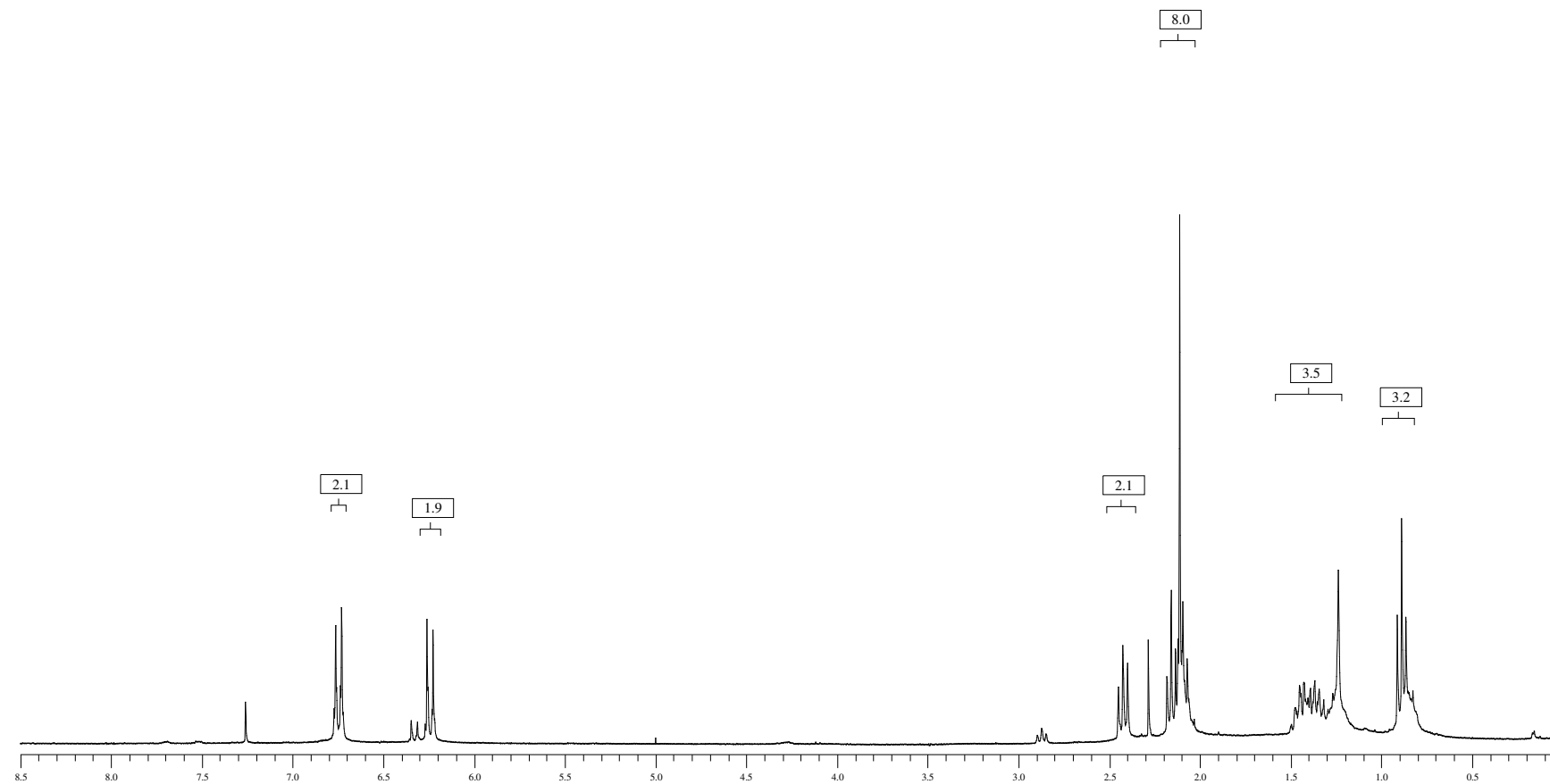
CDCl<sub>3</sub>, 150 MHz



**30b**

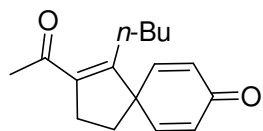


CDCl<sub>3</sub>, 300 MHz

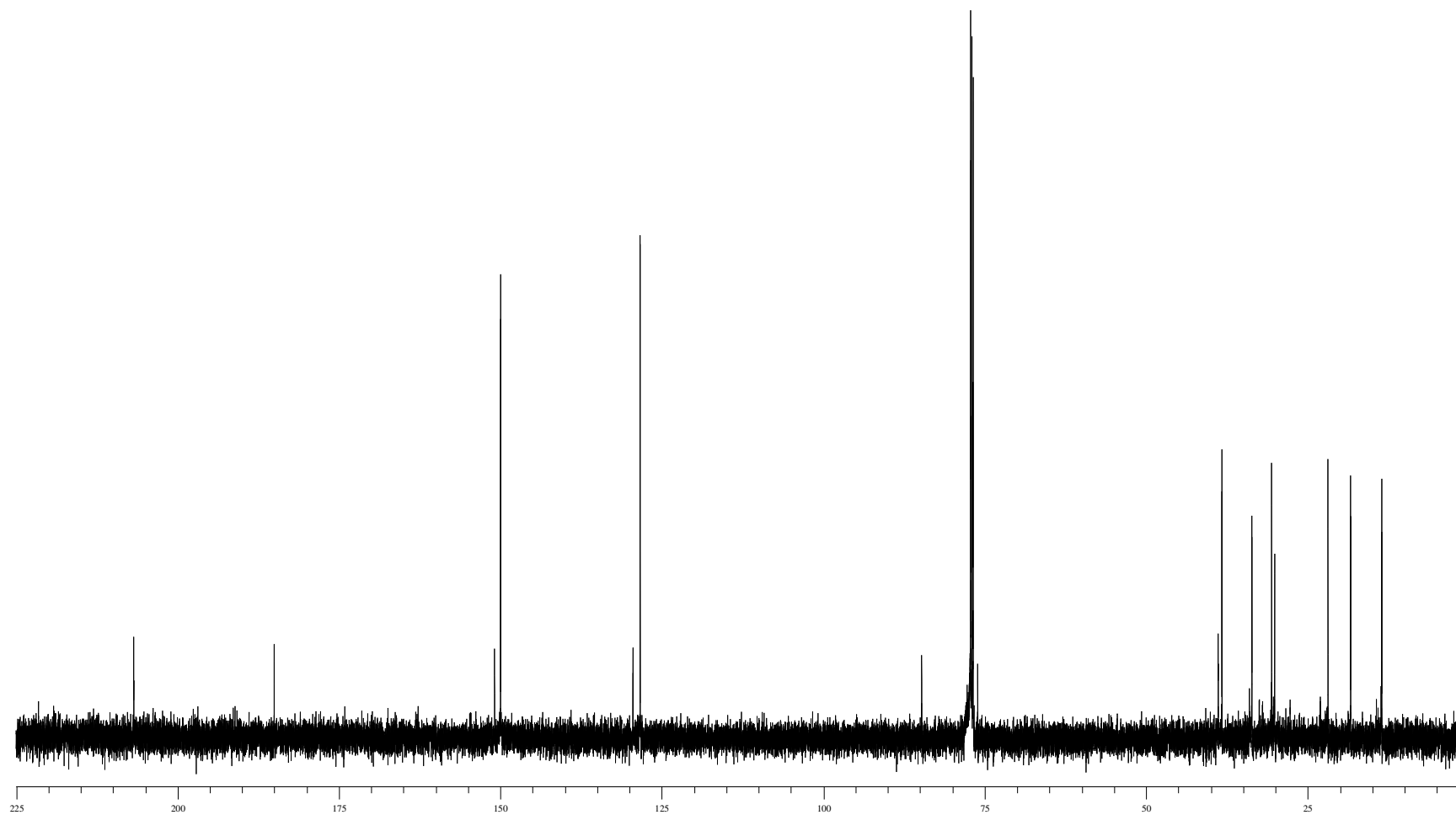


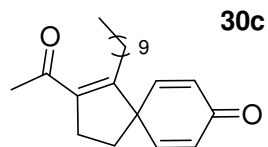


**30b**

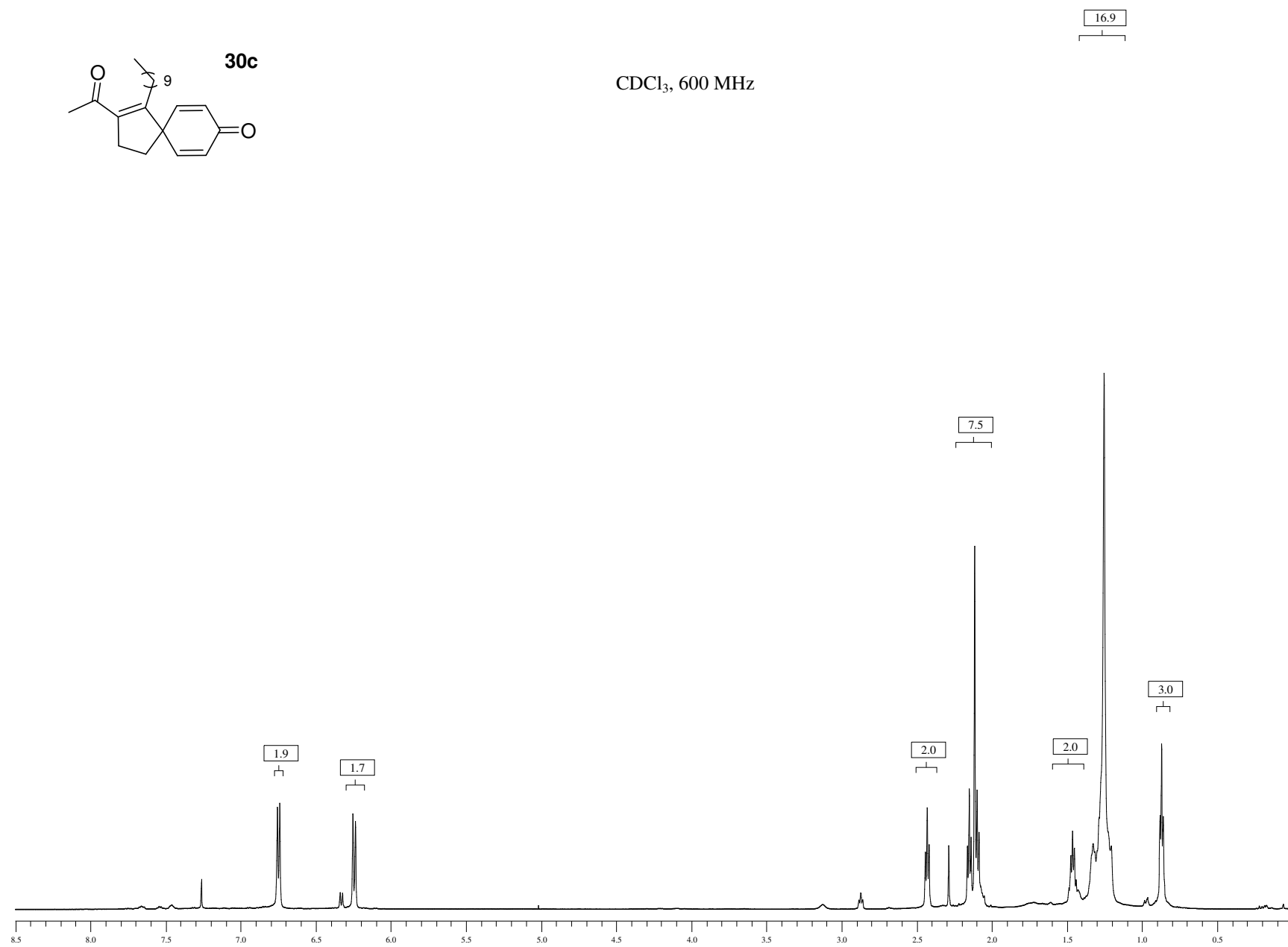


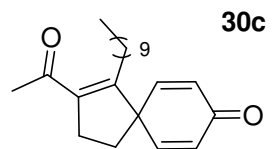
CDCl<sub>3</sub>, 150 MHz



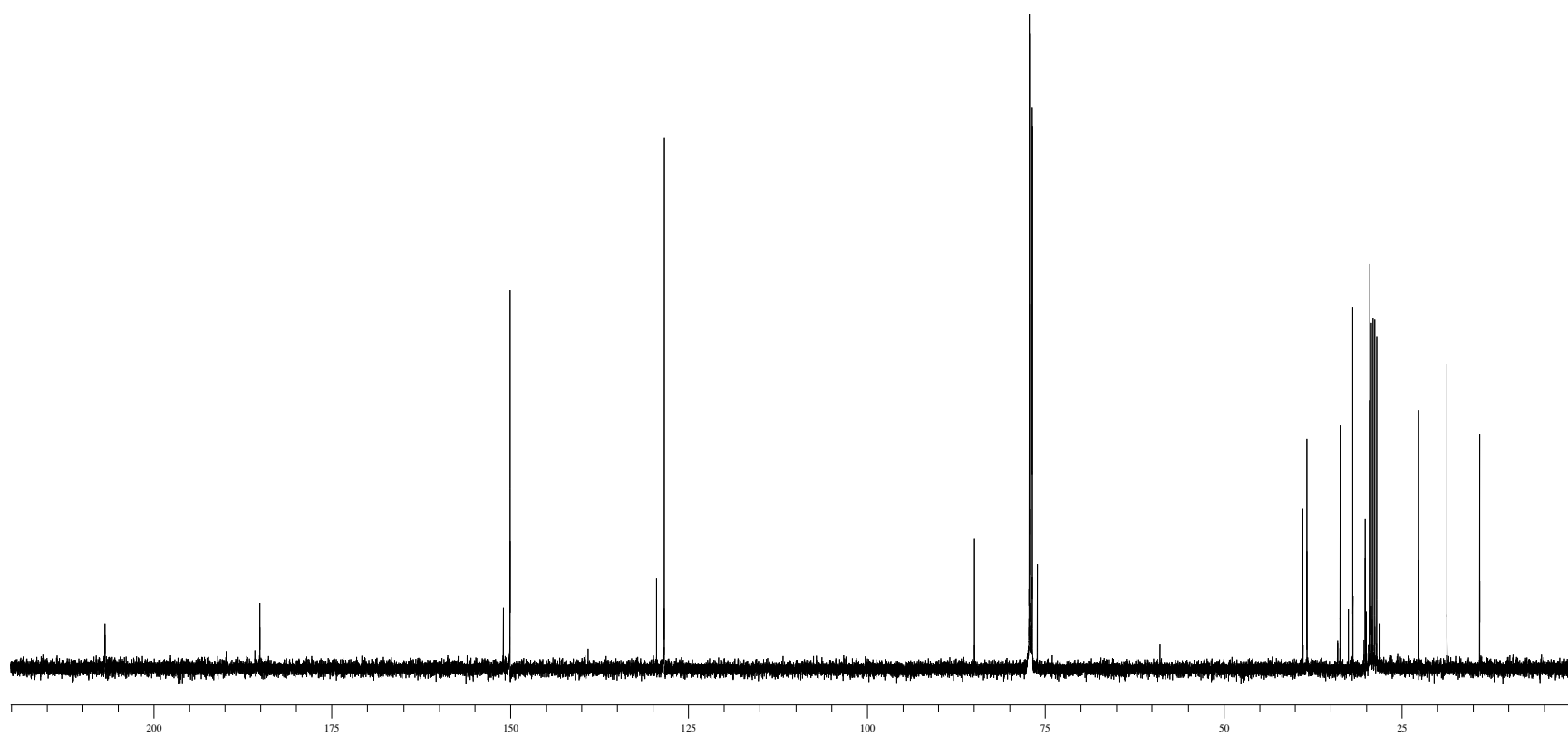


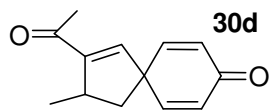
CDCl<sub>3</sub>, 600 MHz



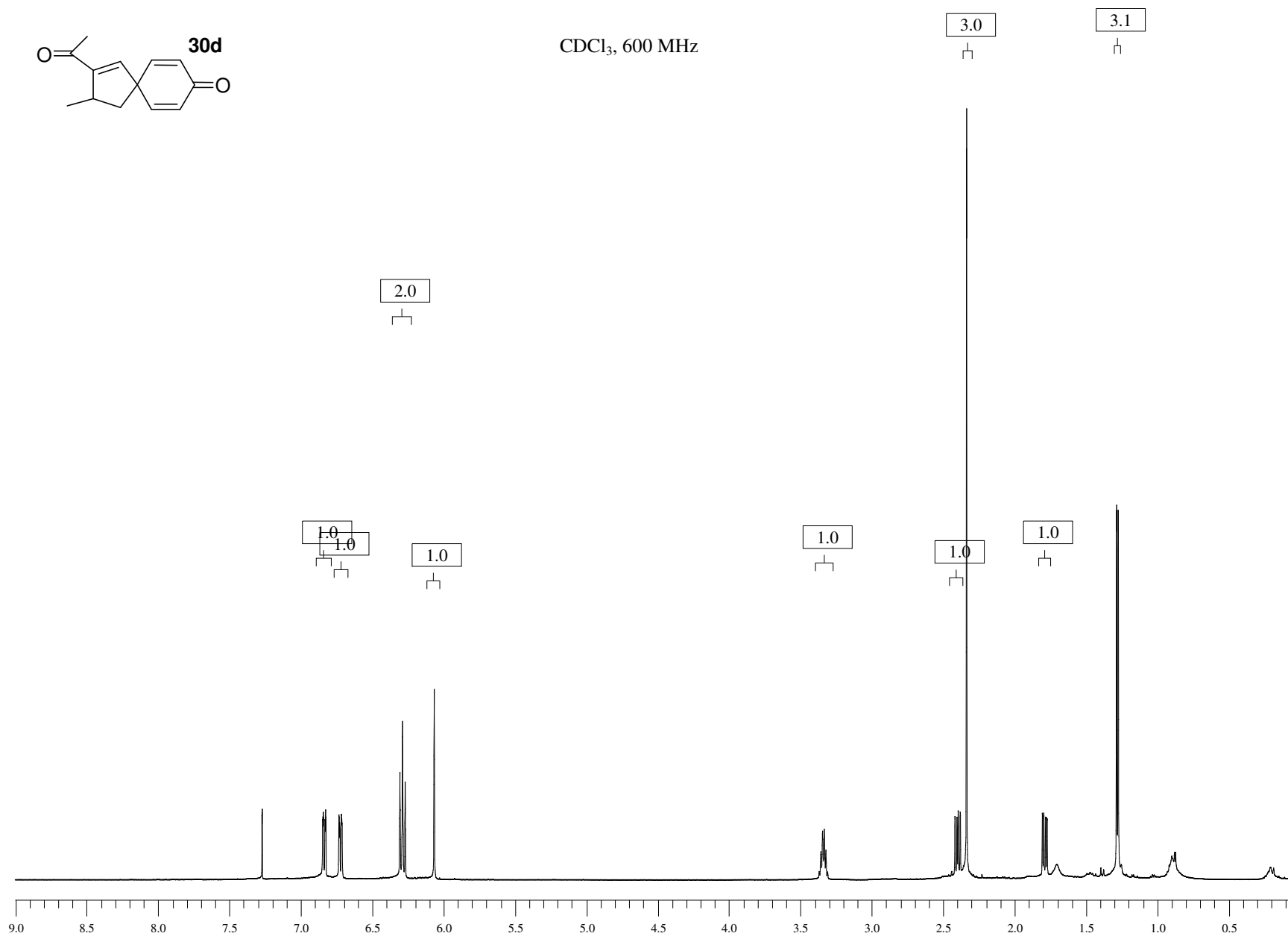


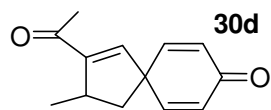
CDCl<sub>3</sub>, 150 MHz



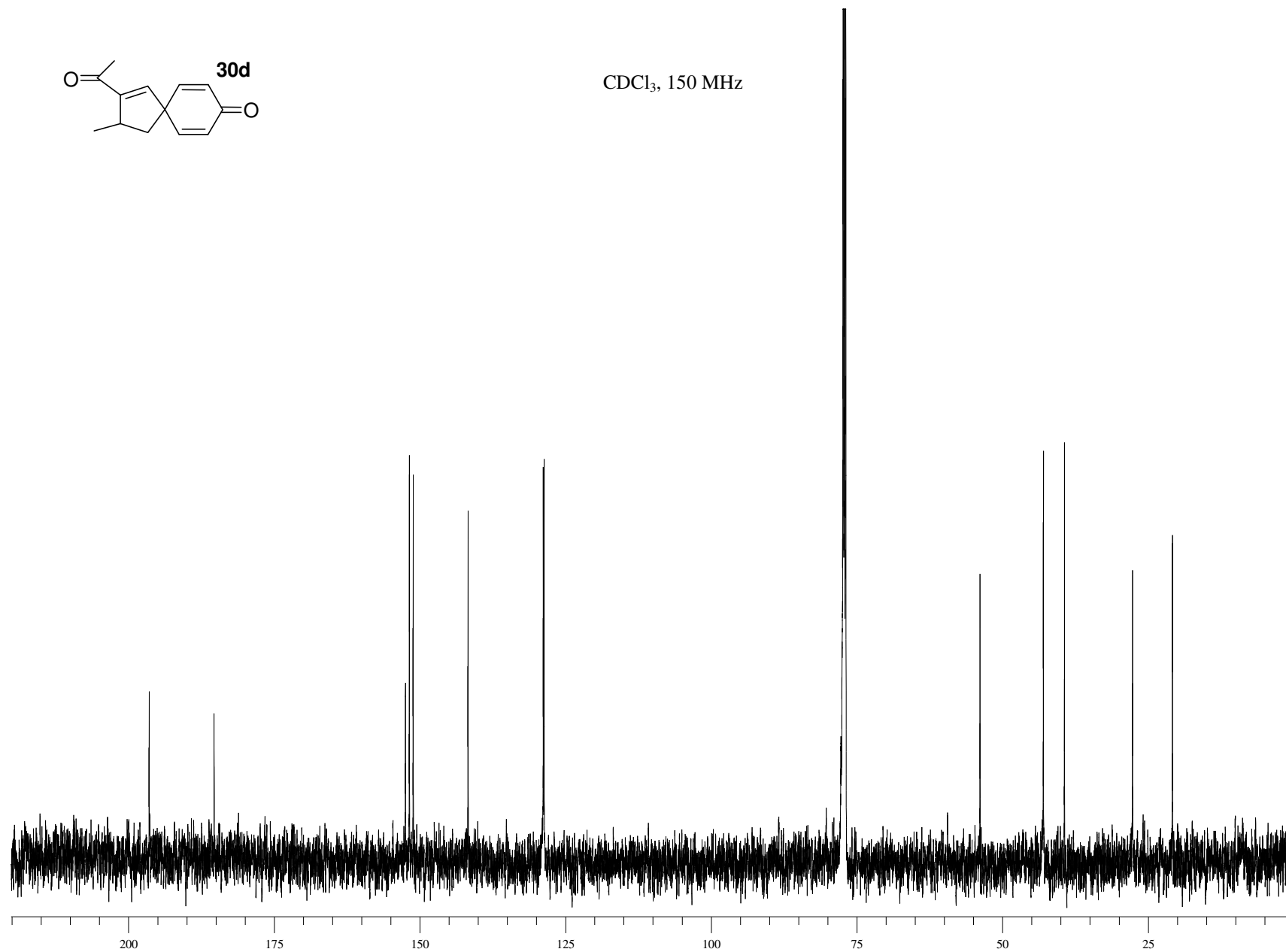


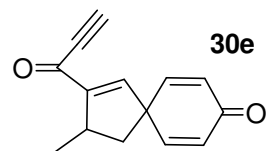
CDCl<sub>3</sub>, 600 MHz



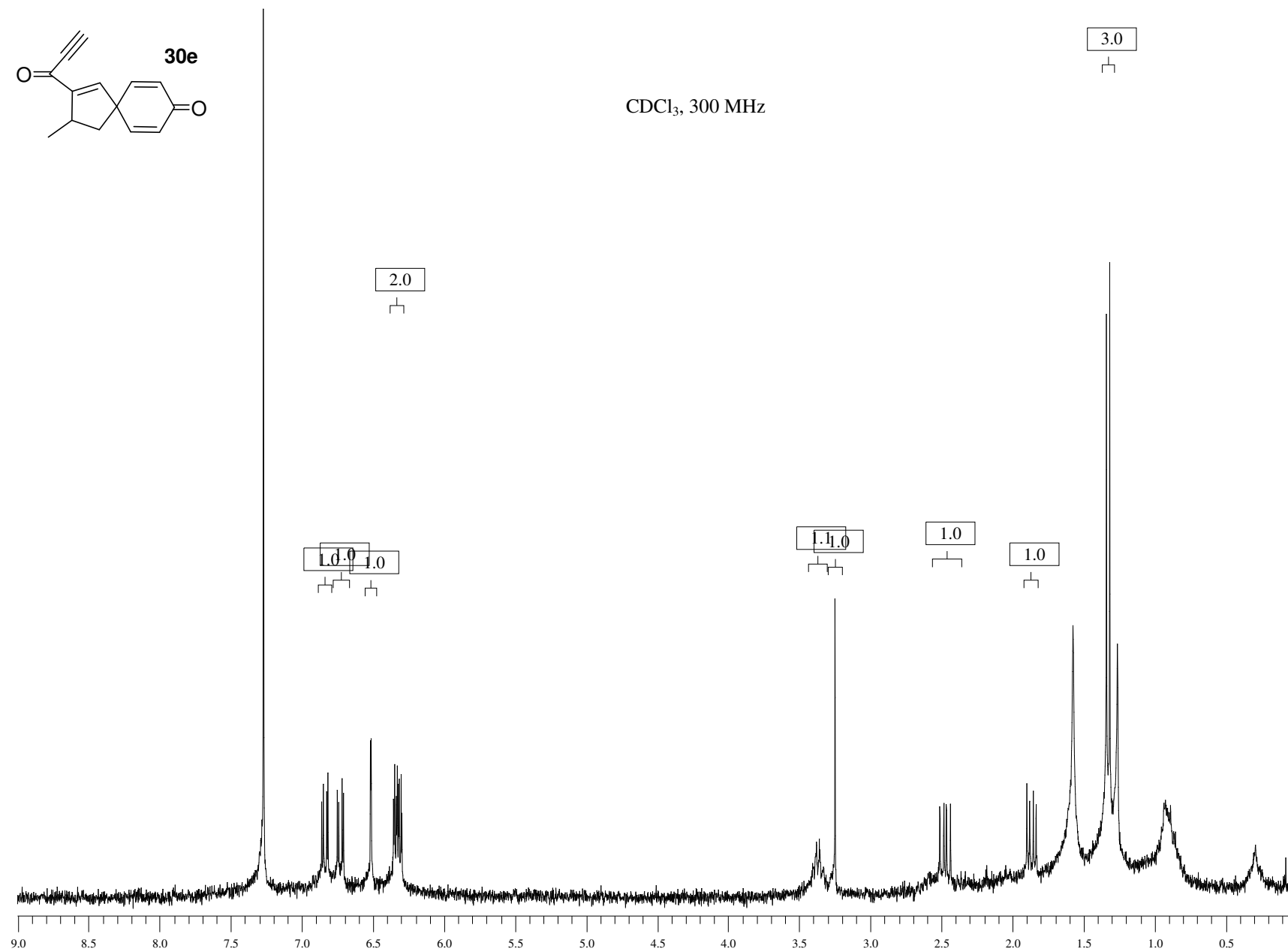


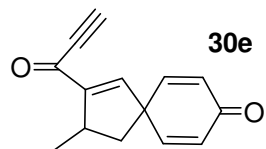
CDCl<sub>3</sub>, 150 MHz



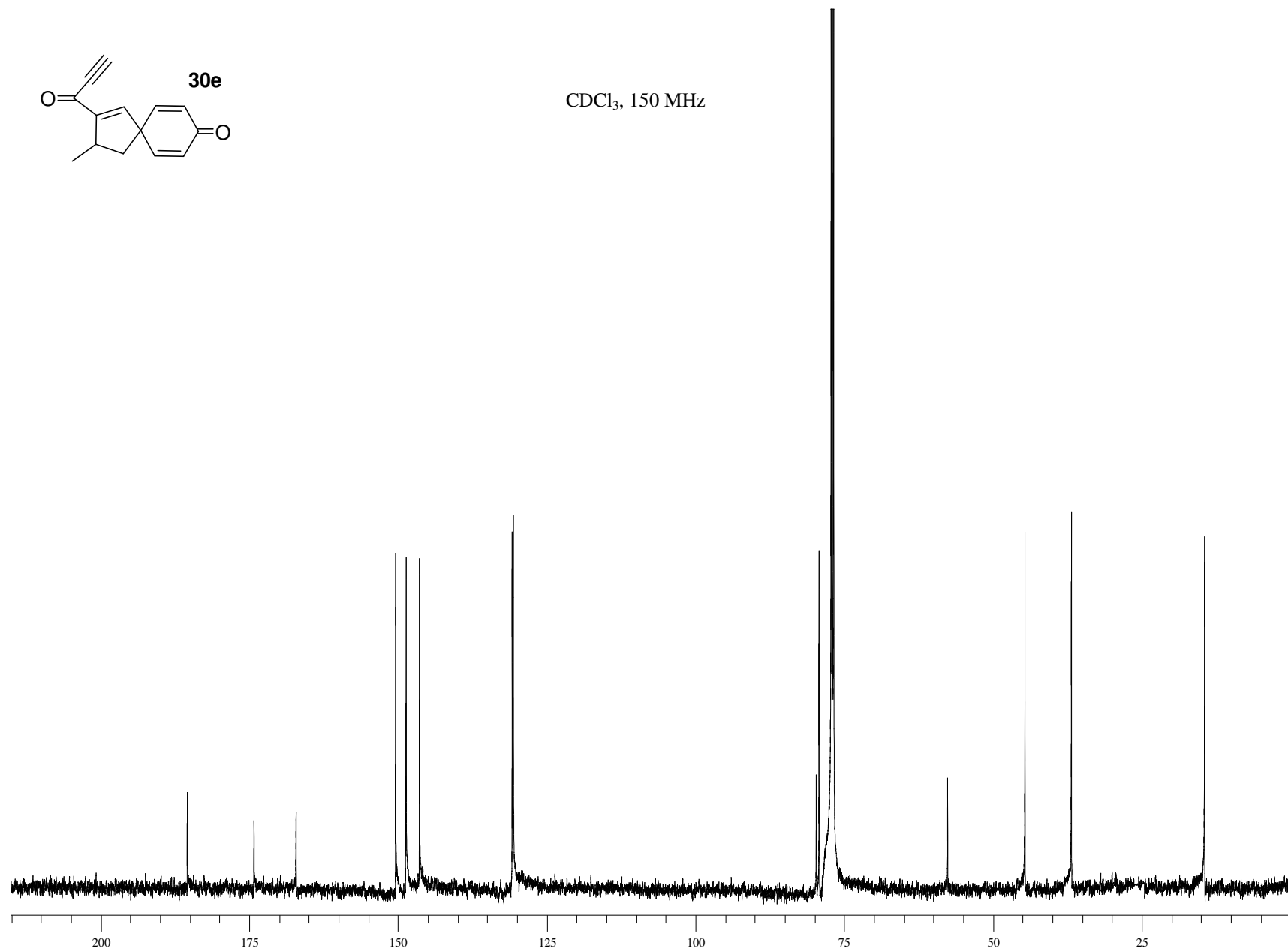


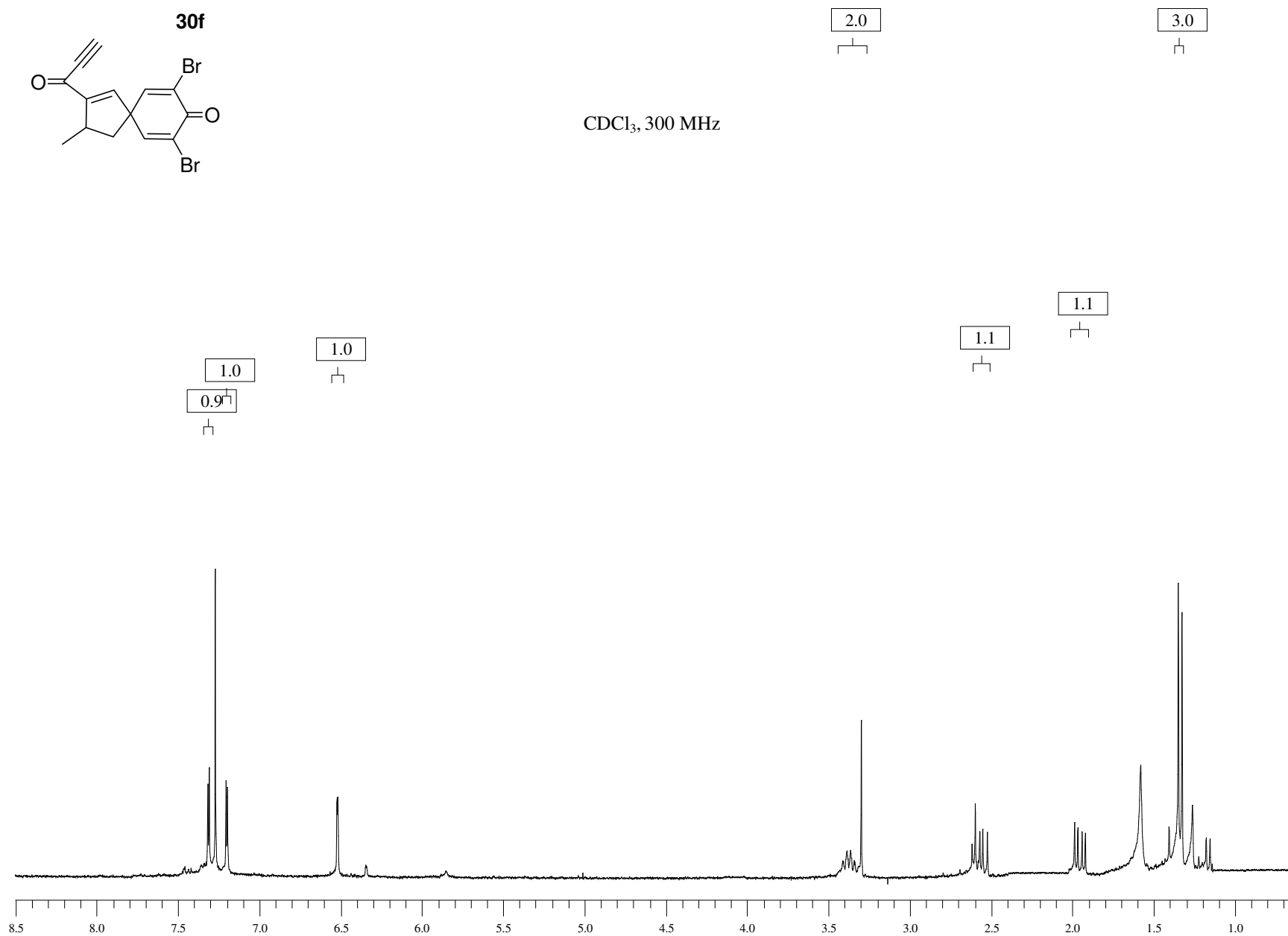
CDCl<sub>3</sub>, 300 MHz



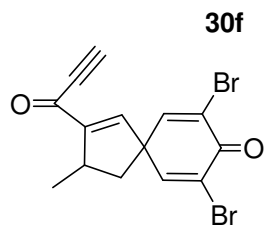


CDCl<sub>3</sub>, 150 MHz

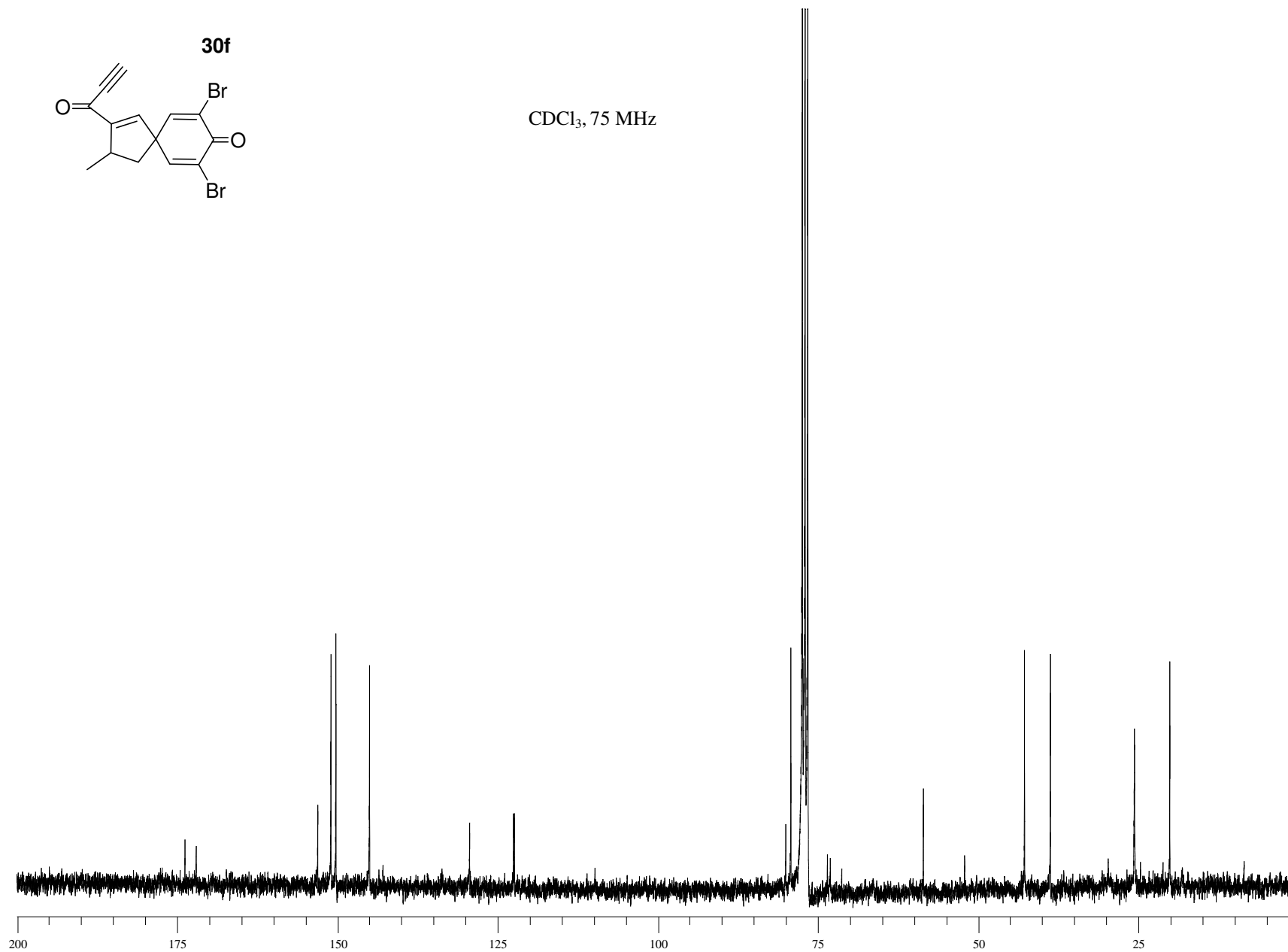


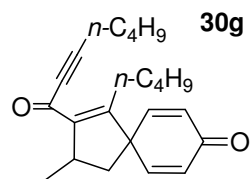








CDCl<sub>3</sub>, 75 MHz

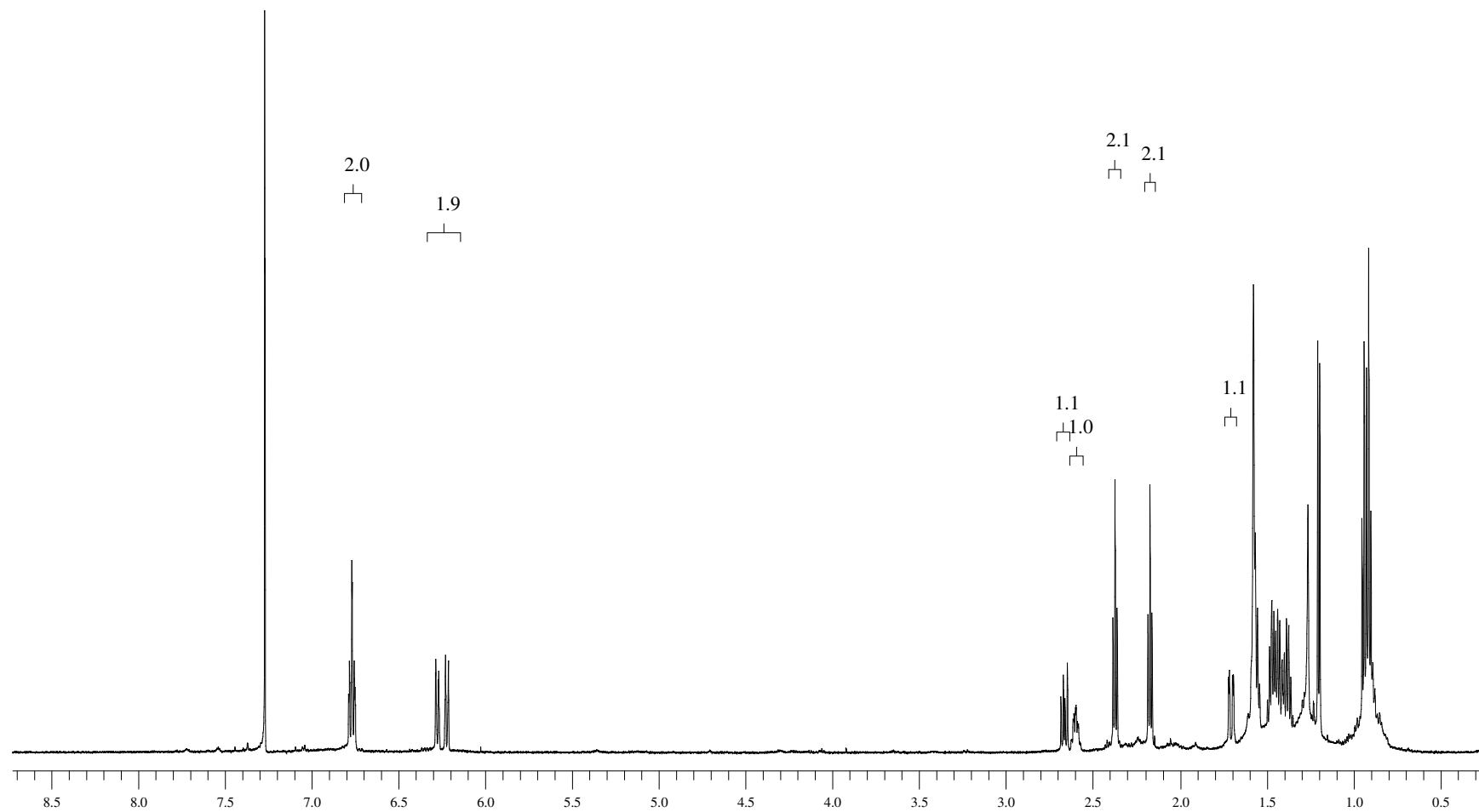


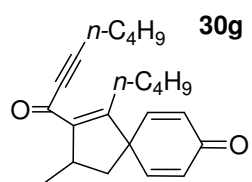


CDCl<sub>3</sub>, 600 MHz

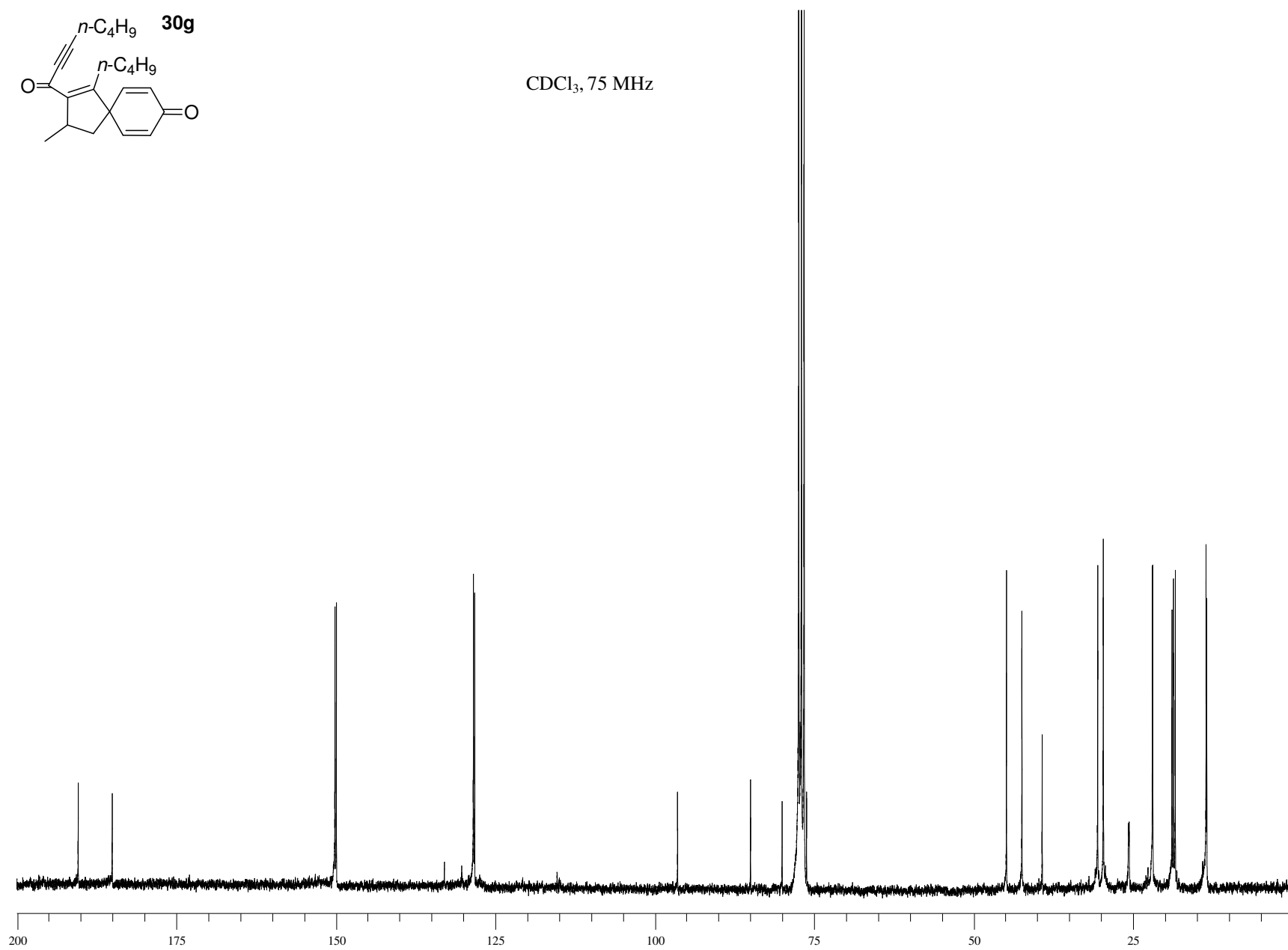
4.05.4 6.1  



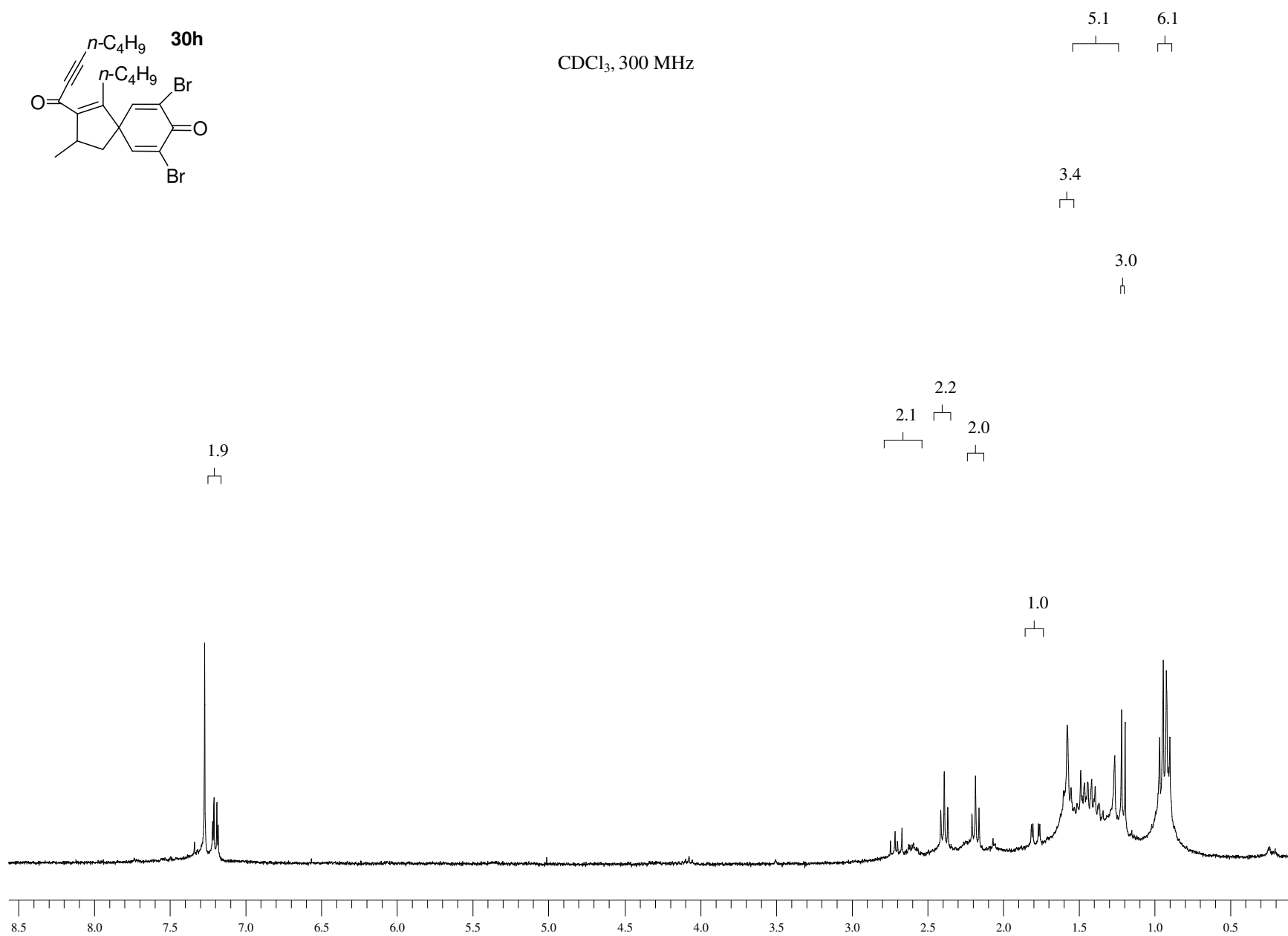
3.0  

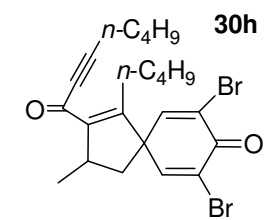





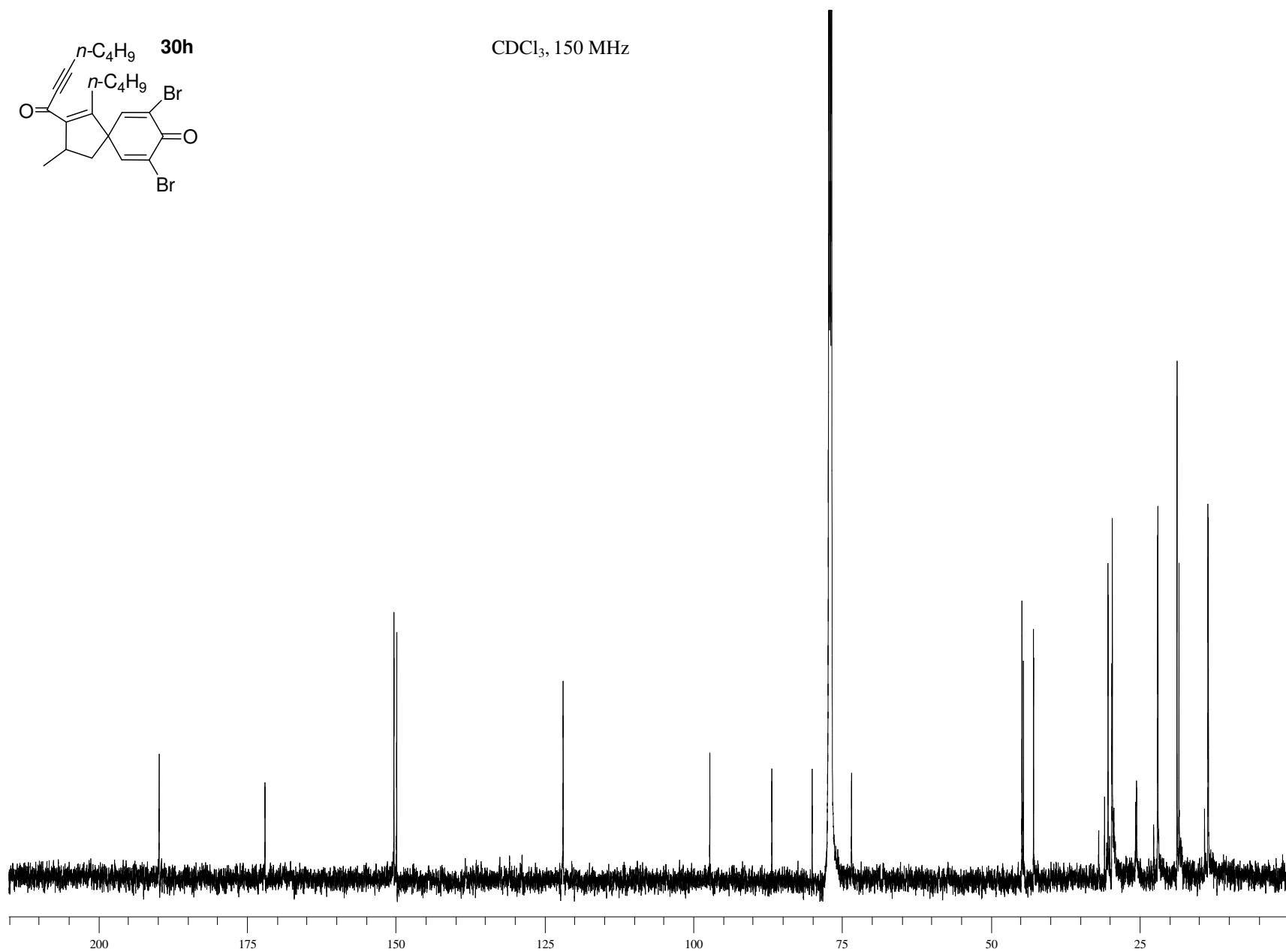
$\text{CDCl}_3$ , 75 MHz



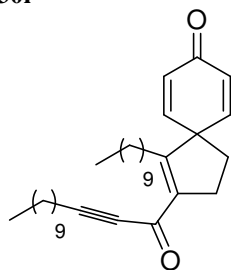




CDCl<sub>3</sub>, 150 MHz

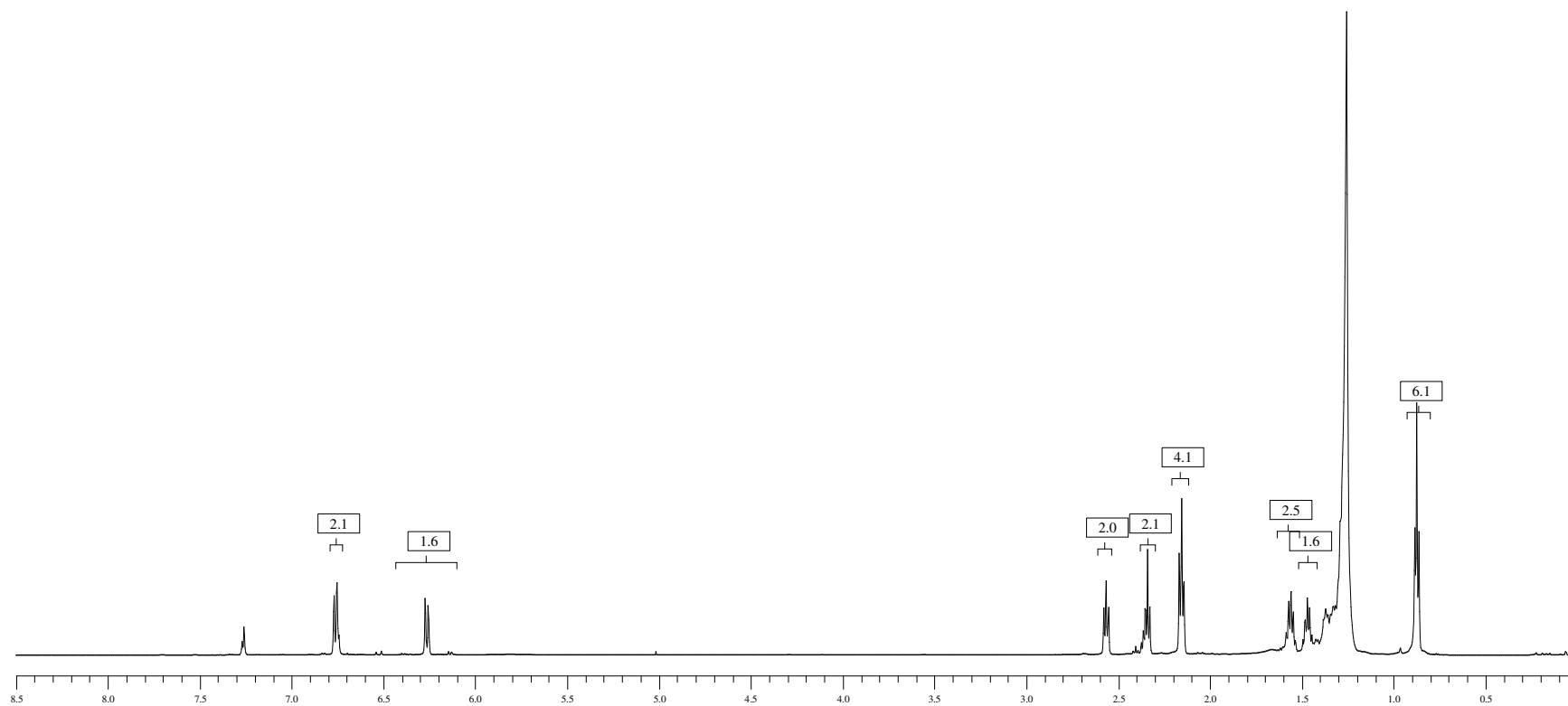


30i

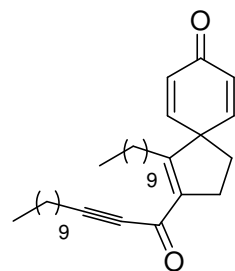


CDCl<sub>3</sub>, 600 MHz

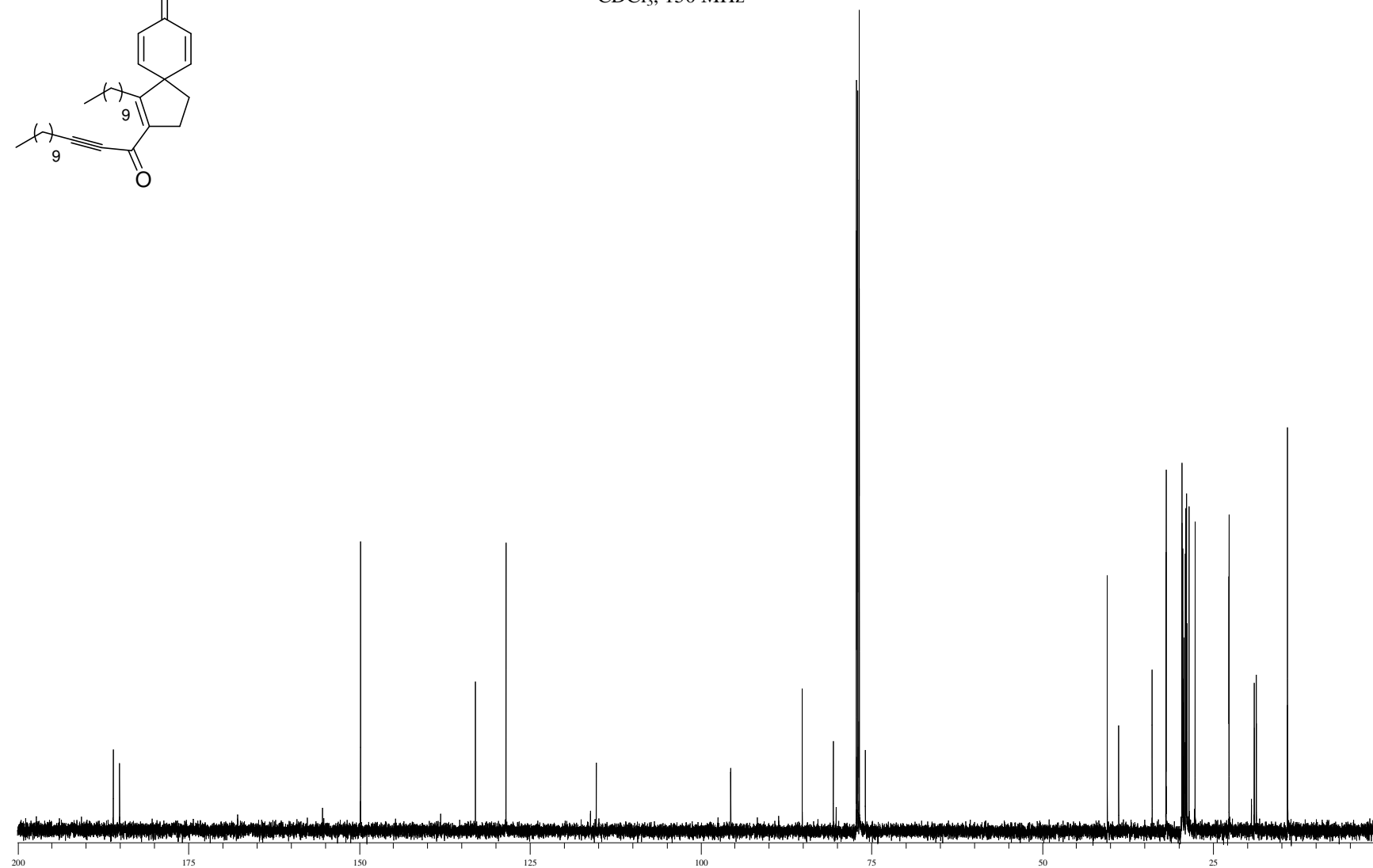
29.2

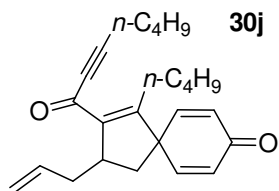


30i

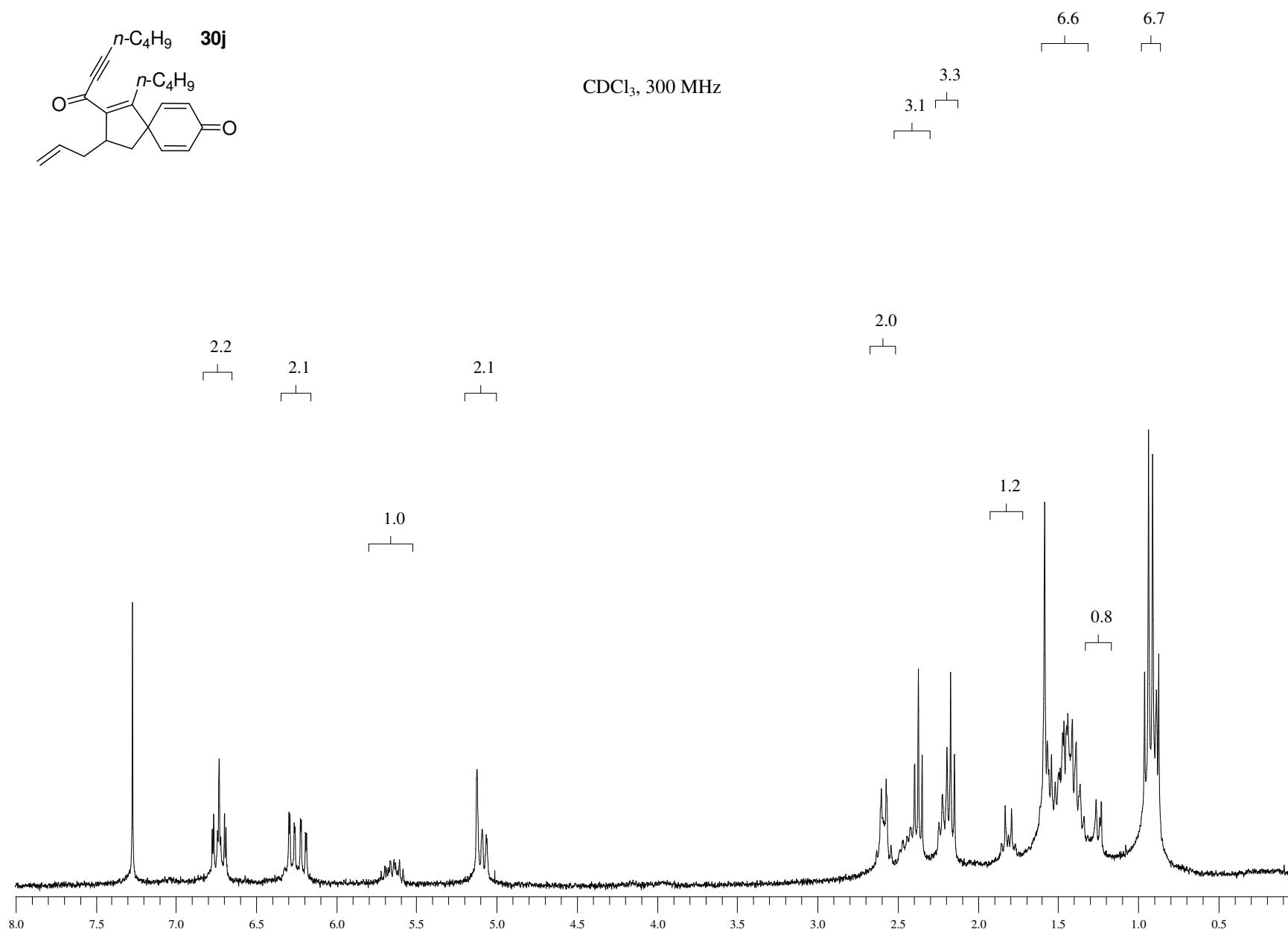


CDCl<sub>3</sub>, 150 MHz

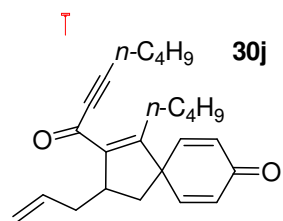




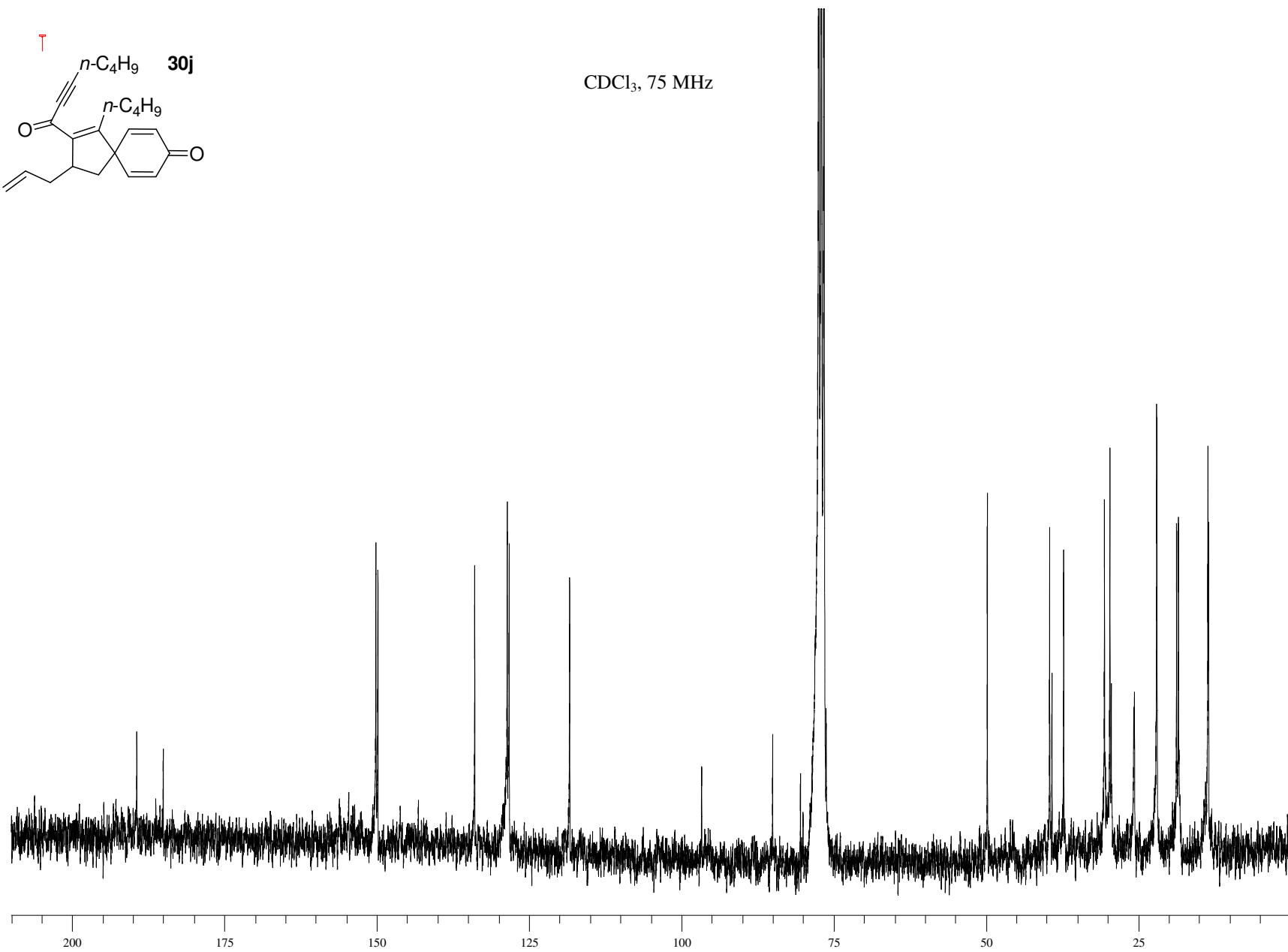
CDCl<sub>3</sub>, 300 MHz

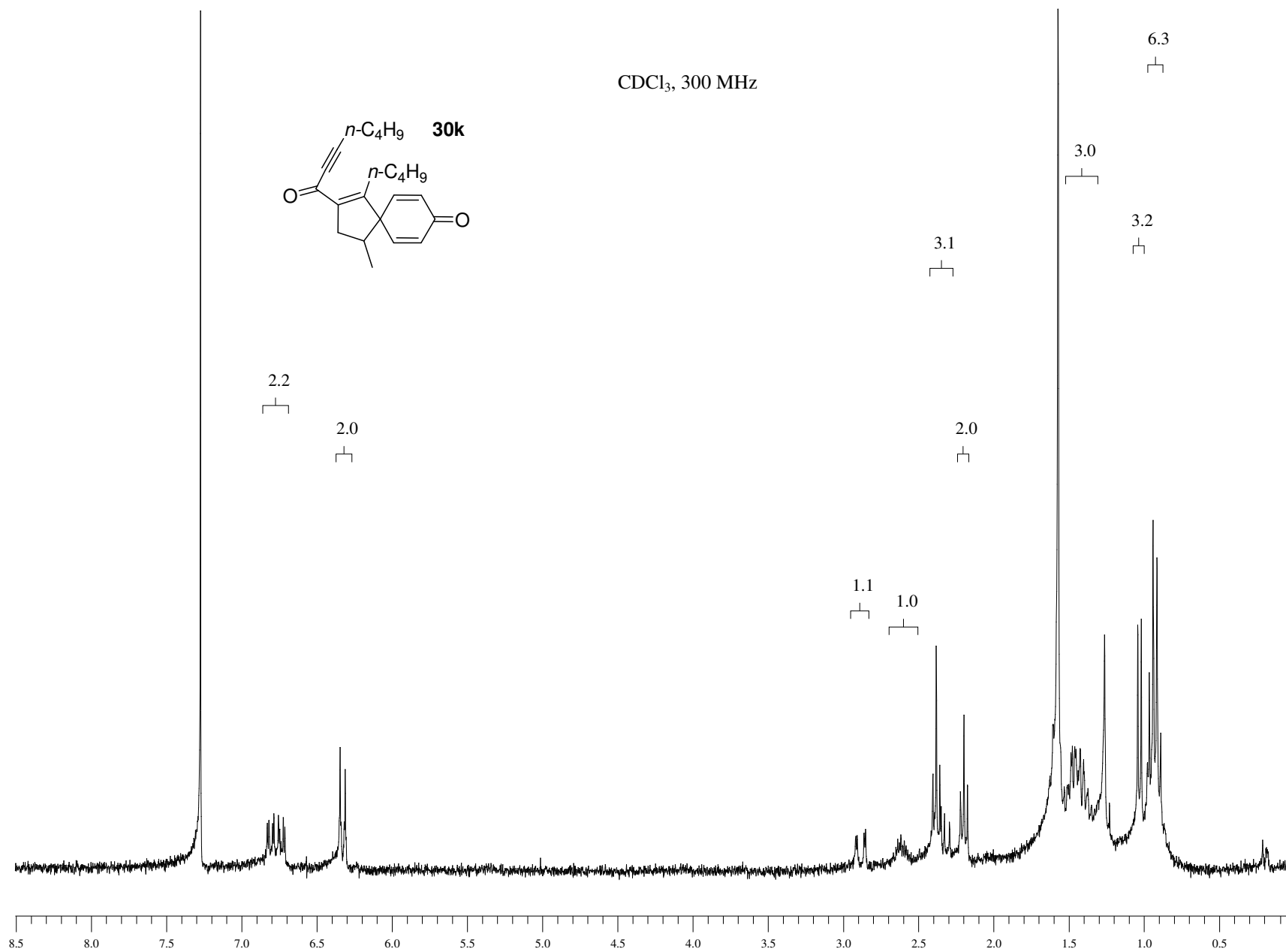


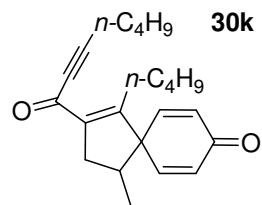




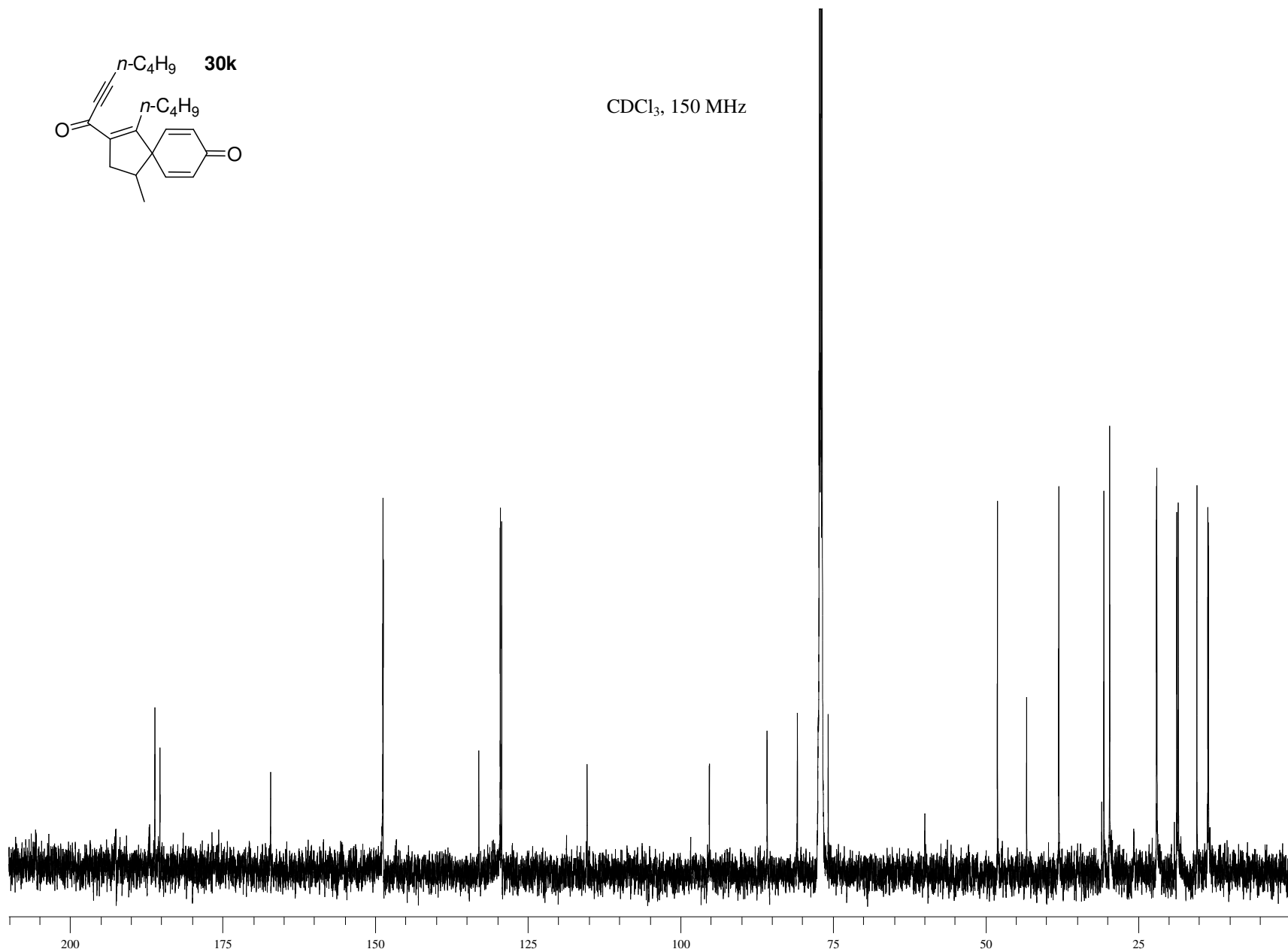
CDCl<sub>3</sub>, 75 MHz

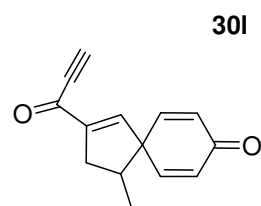






CDCl<sub>3</sub>, 150 MHz

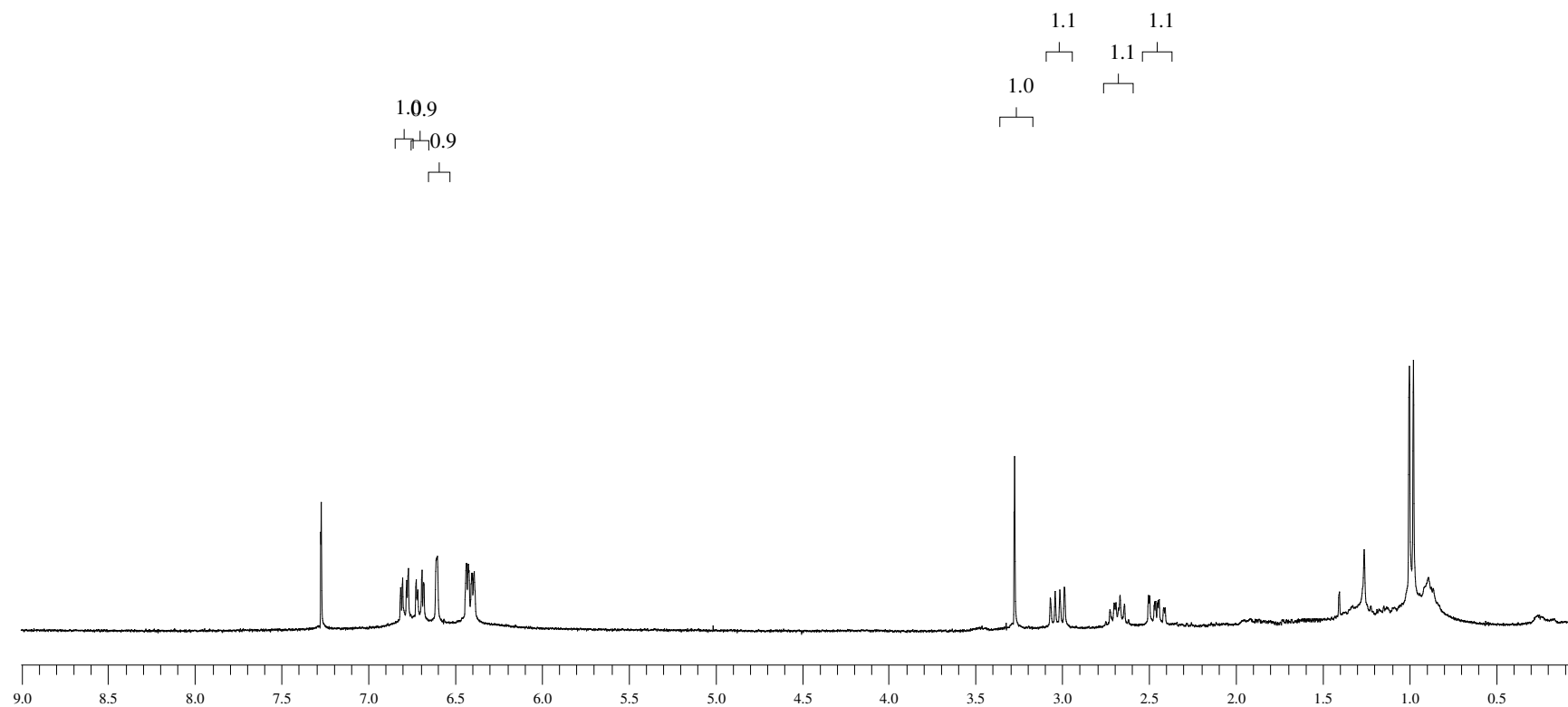


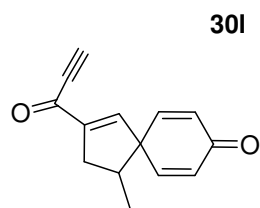


2.1  
└─┘

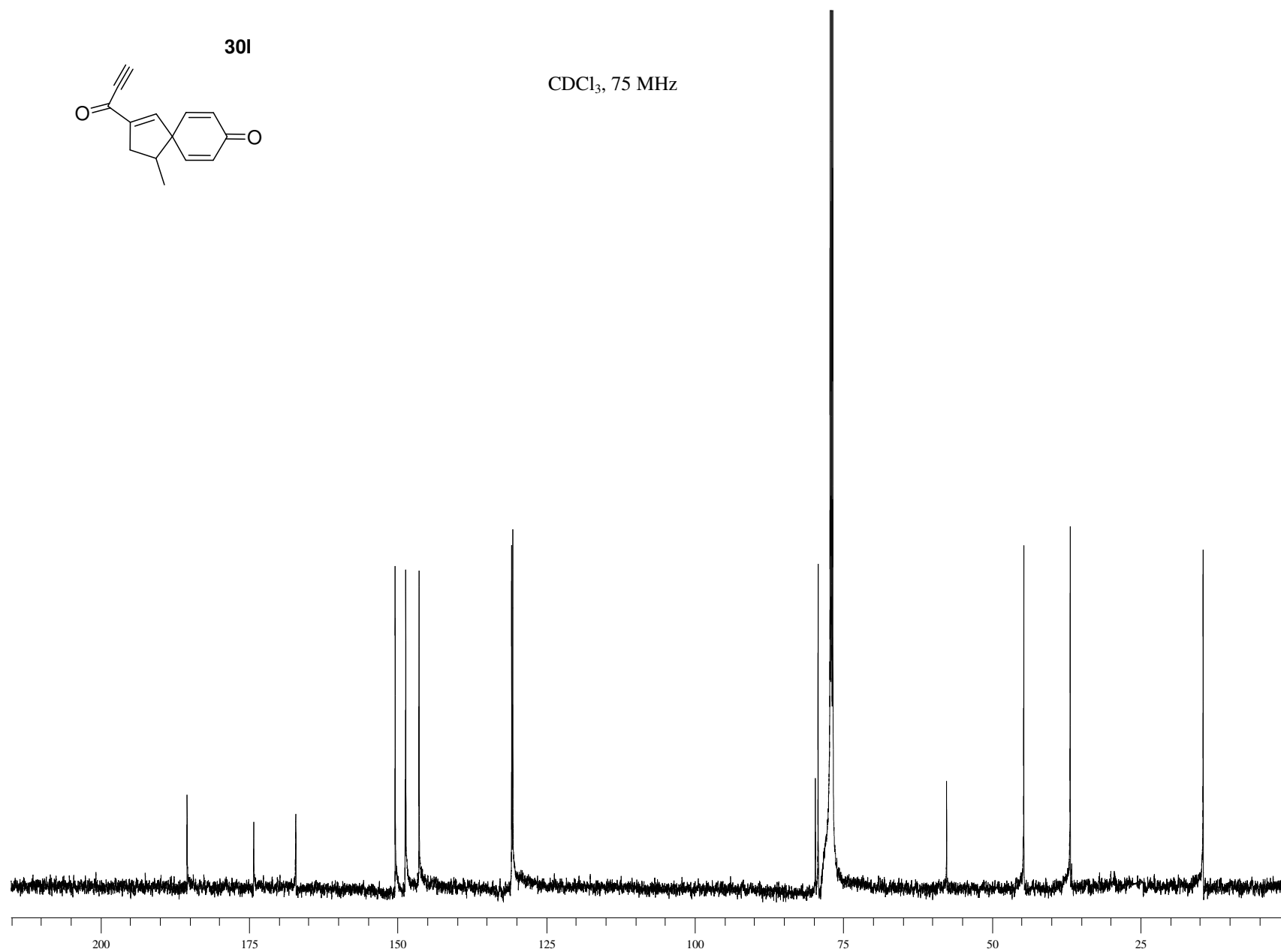
3.2  
└─┘

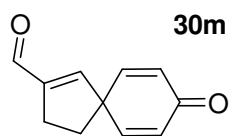
CDCl<sub>3</sub>, 300 MHz





CDCl<sub>3</sub>, 75 MHz





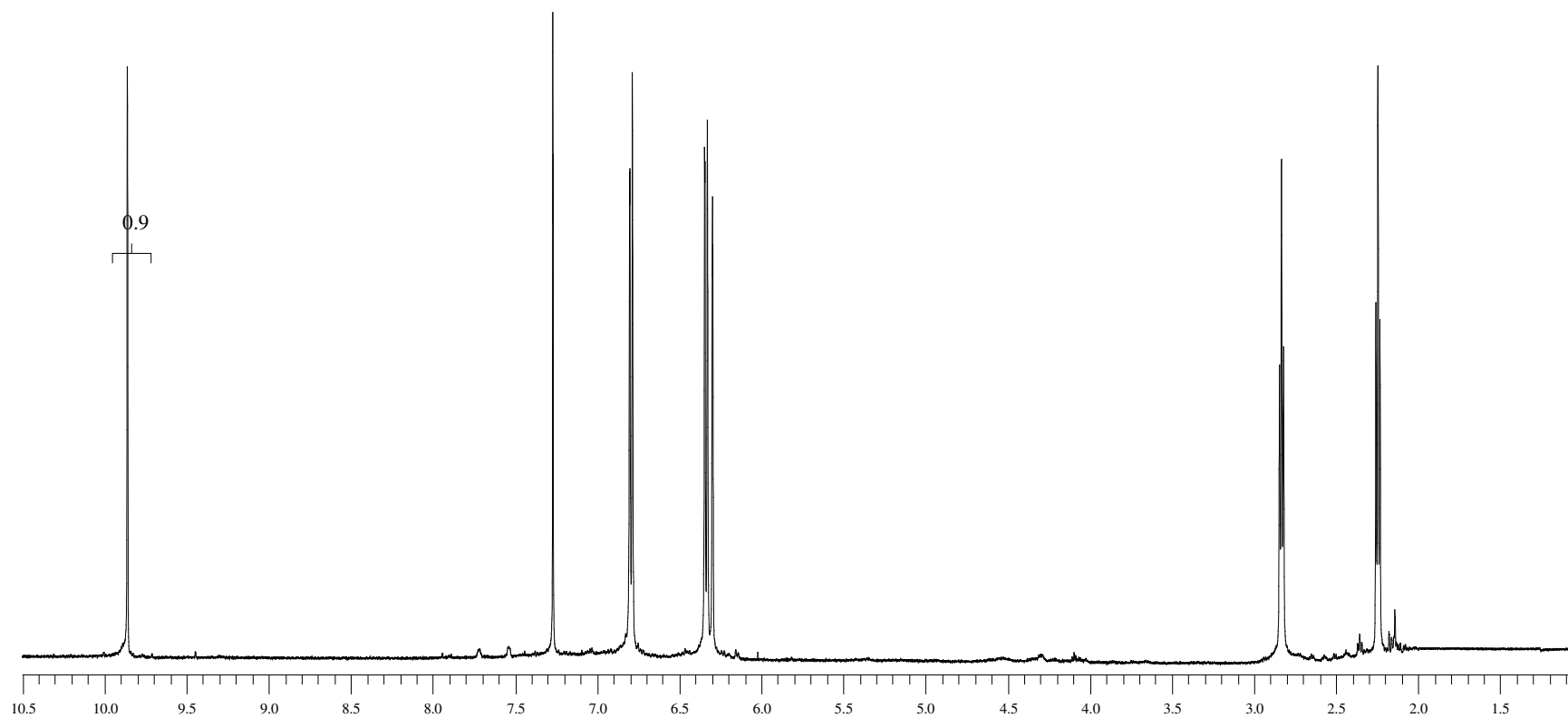
3.0

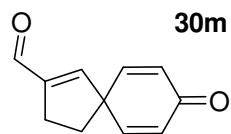
CDCl<sub>3</sub>, 600 MHz

2.1

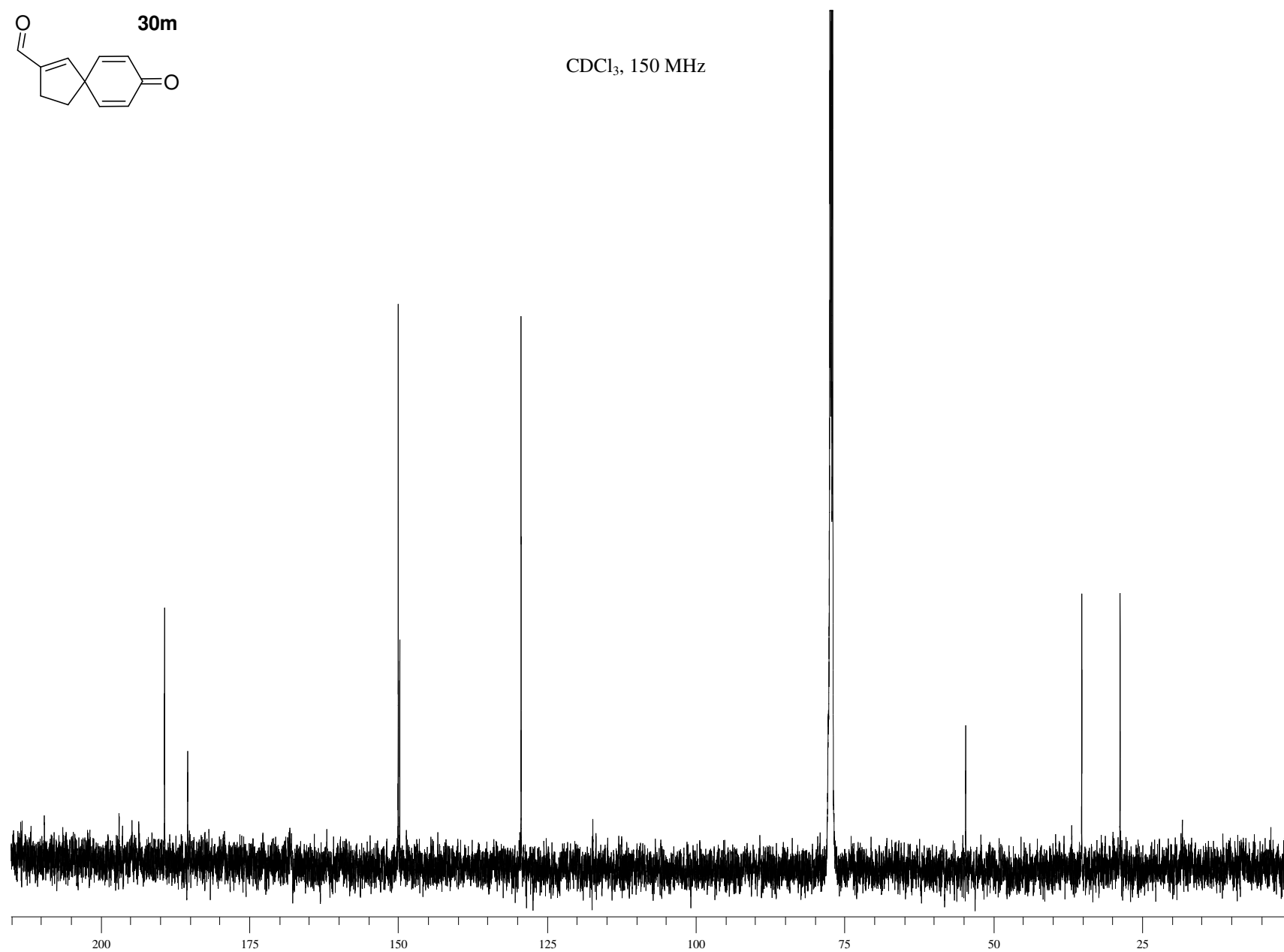
2.1

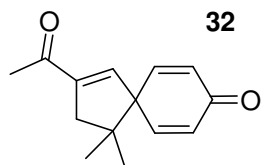
2.1





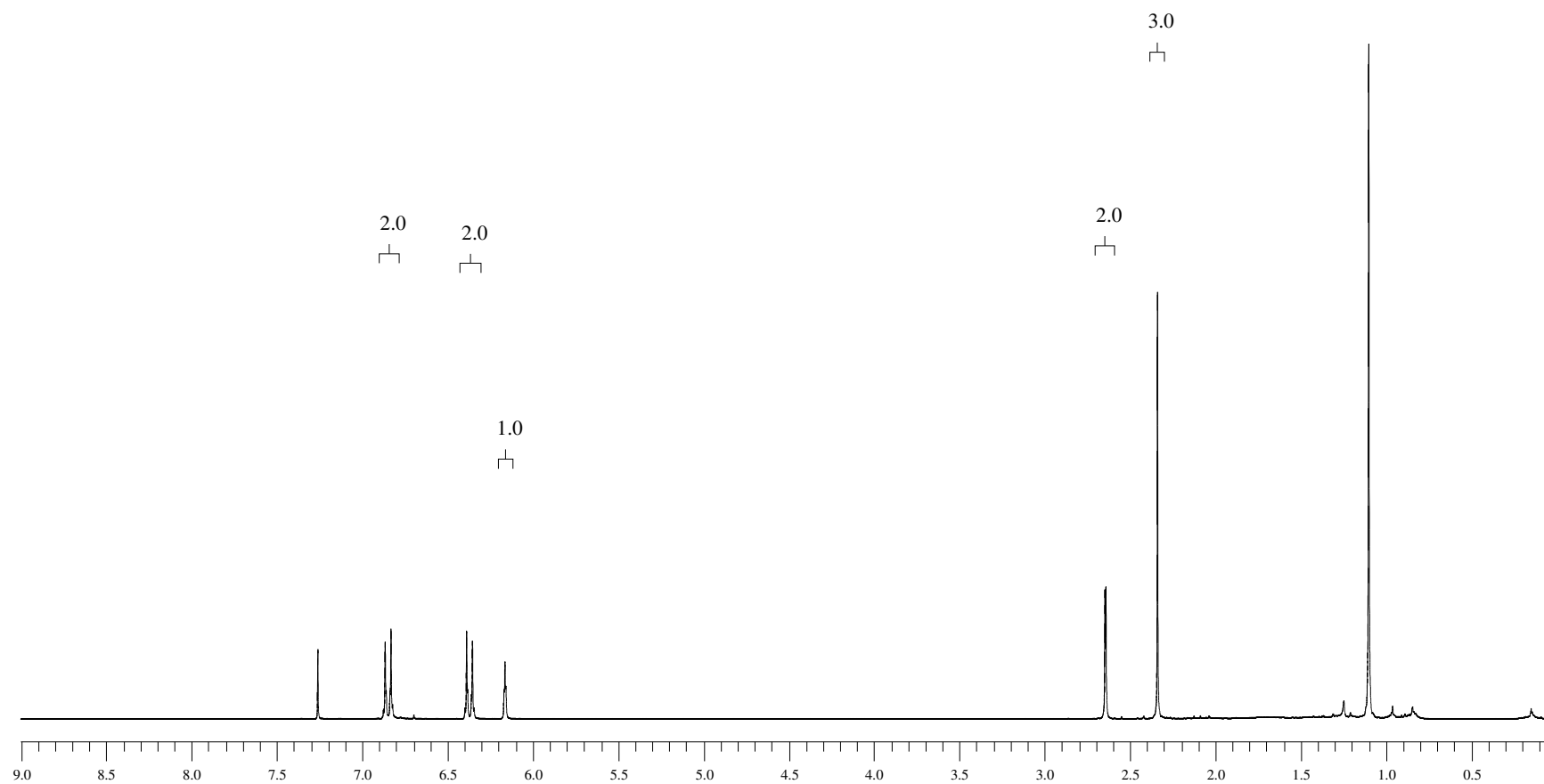
CDCl<sub>3</sub>, 150 MHz



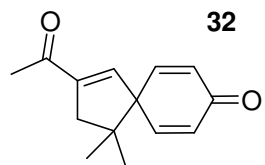


CDCl<sub>3</sub>, 300 MHz

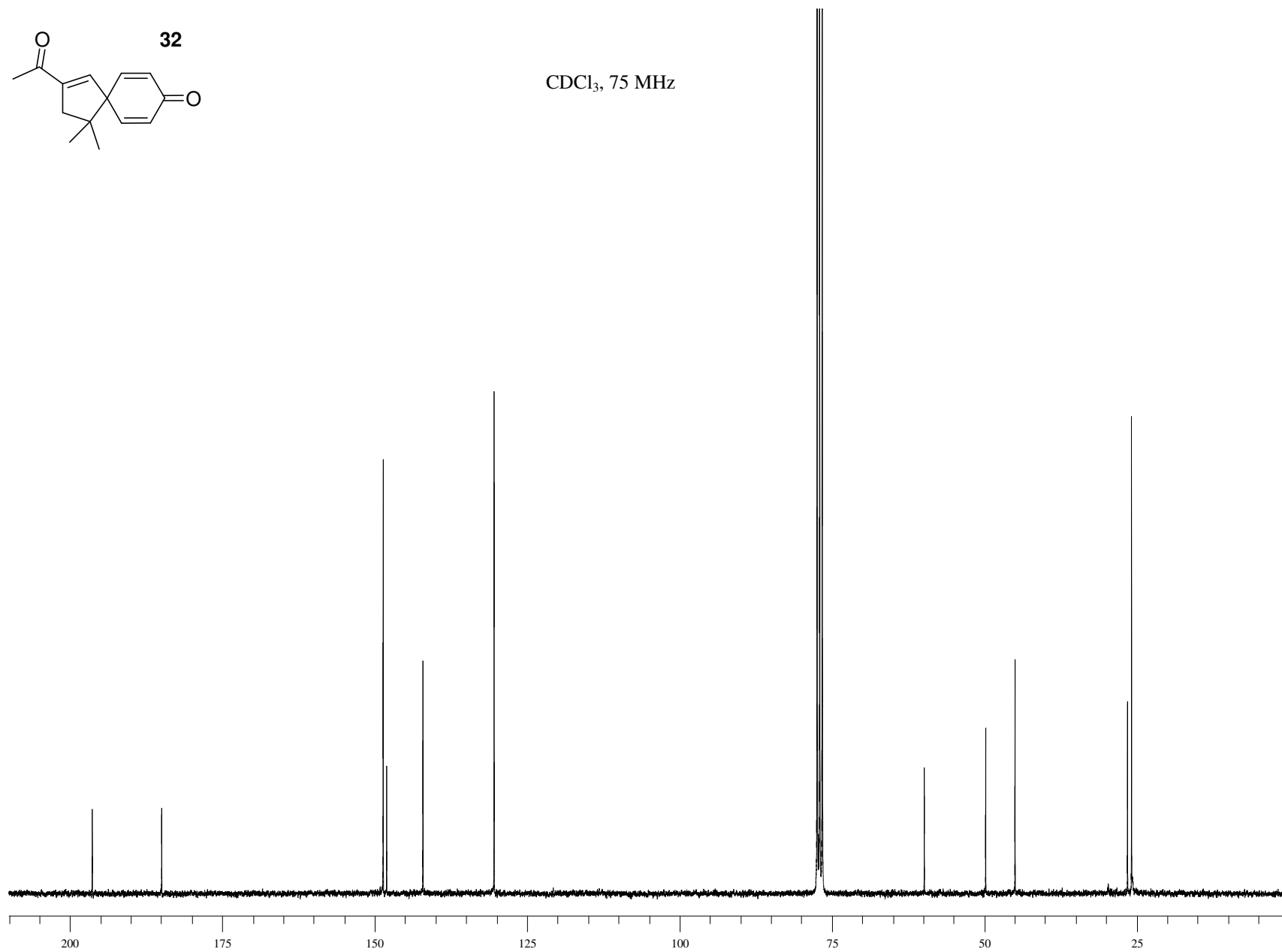
6.0  
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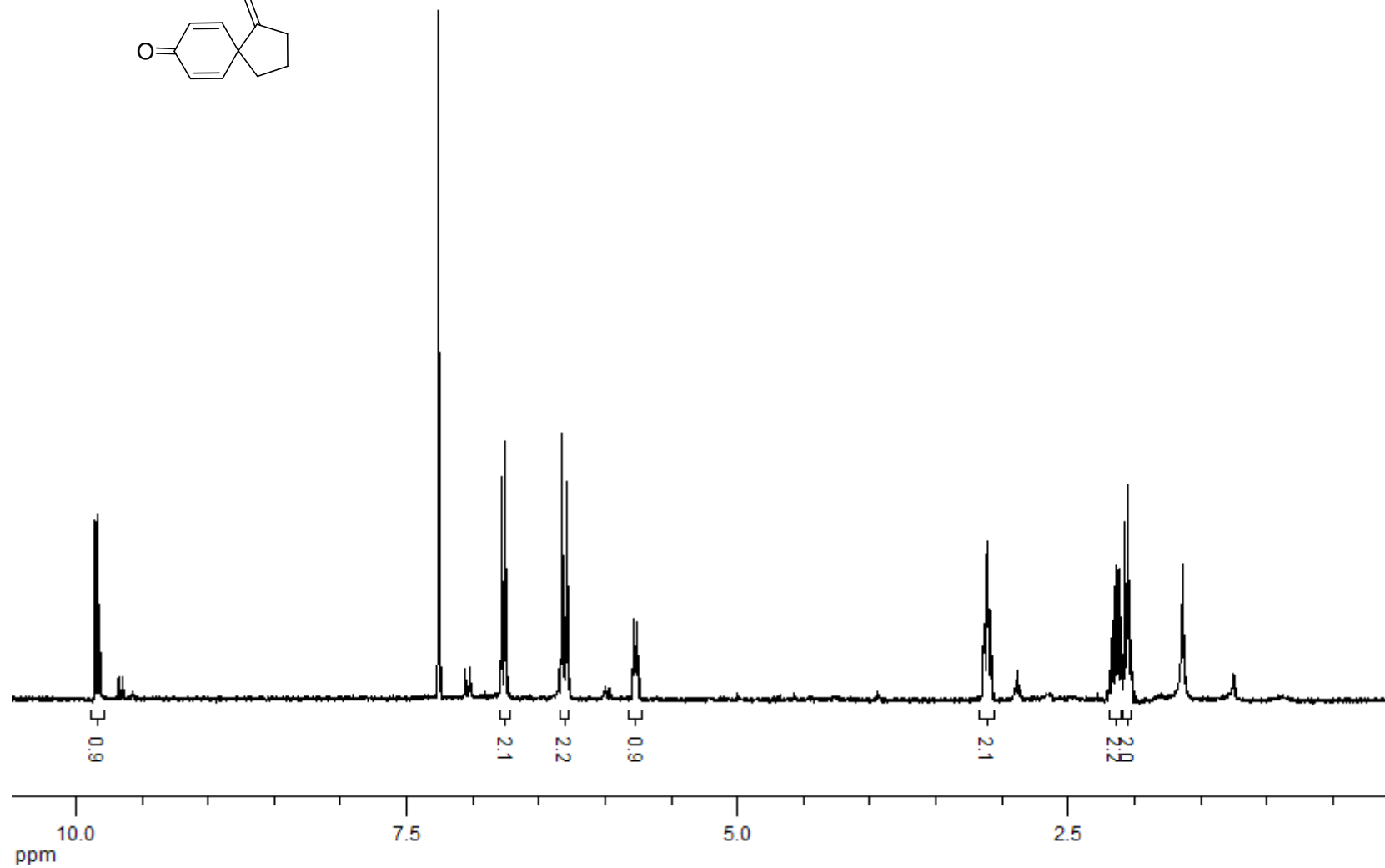
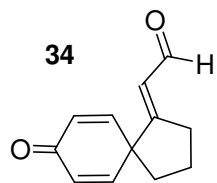


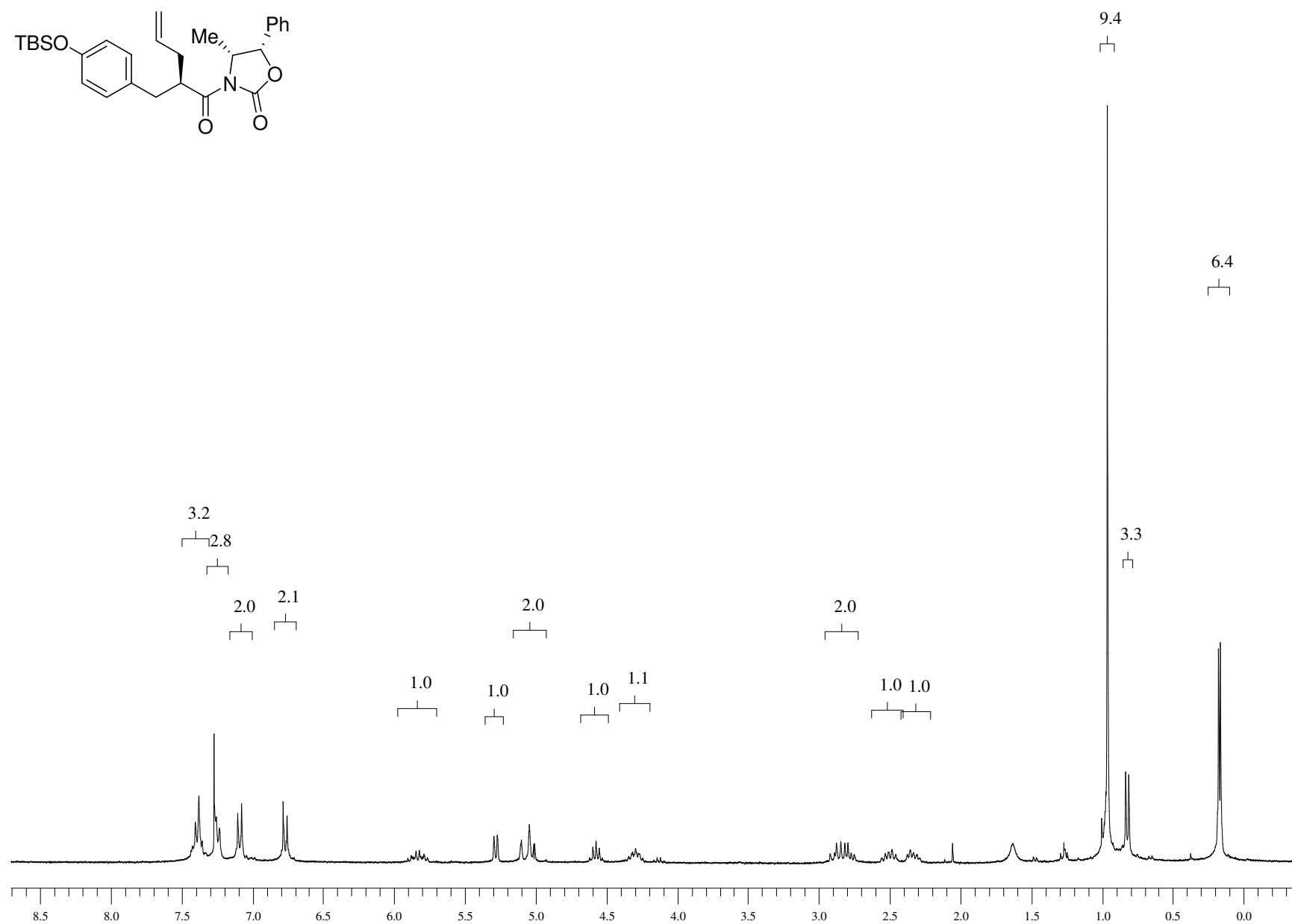
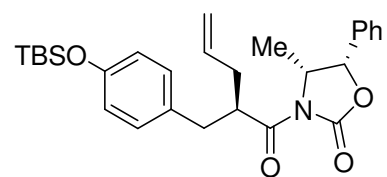
CDCl<sub>3</sub>, 75 MHz

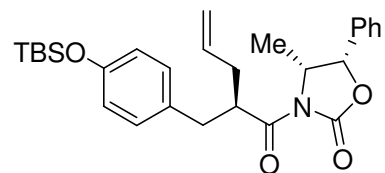


Ratio E/Z= (9/1)

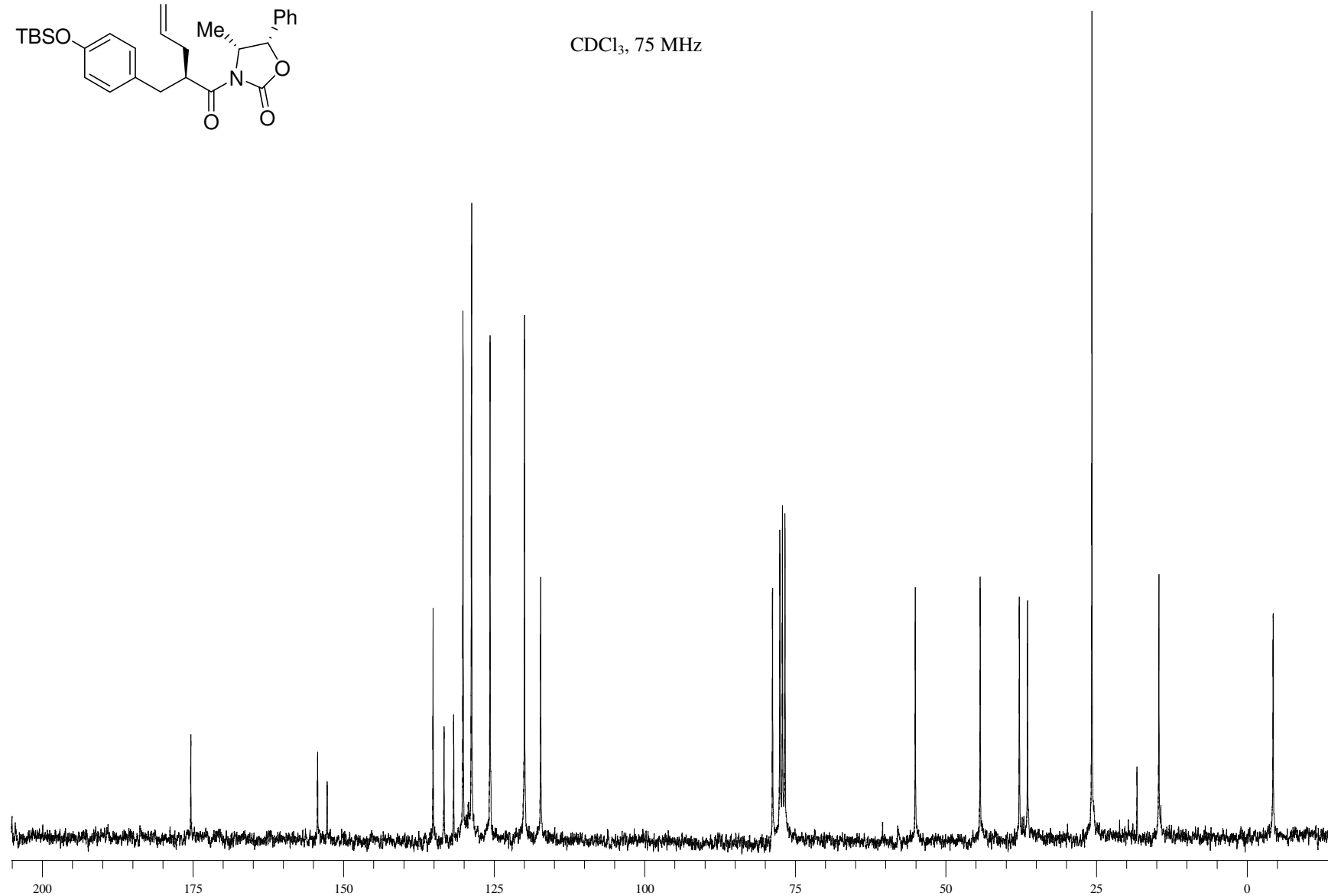
CDCl<sub>3</sub>, 300 MHz

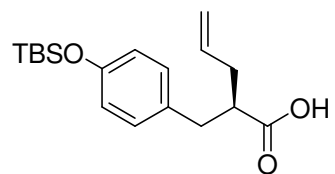




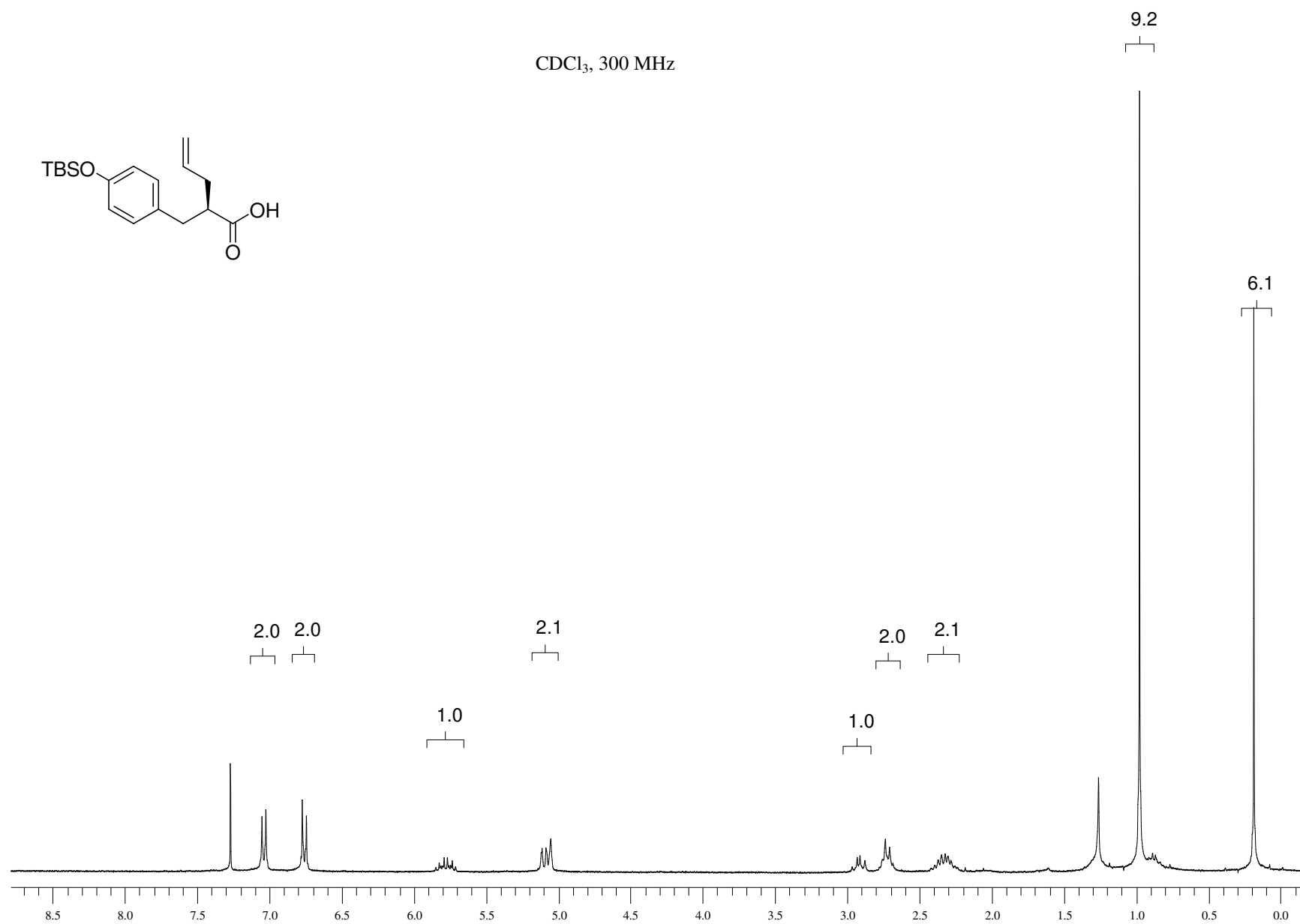


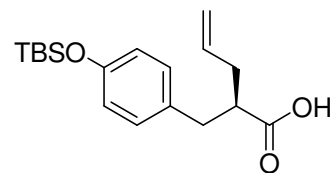
CDCl<sub>3</sub>, 75 MHz



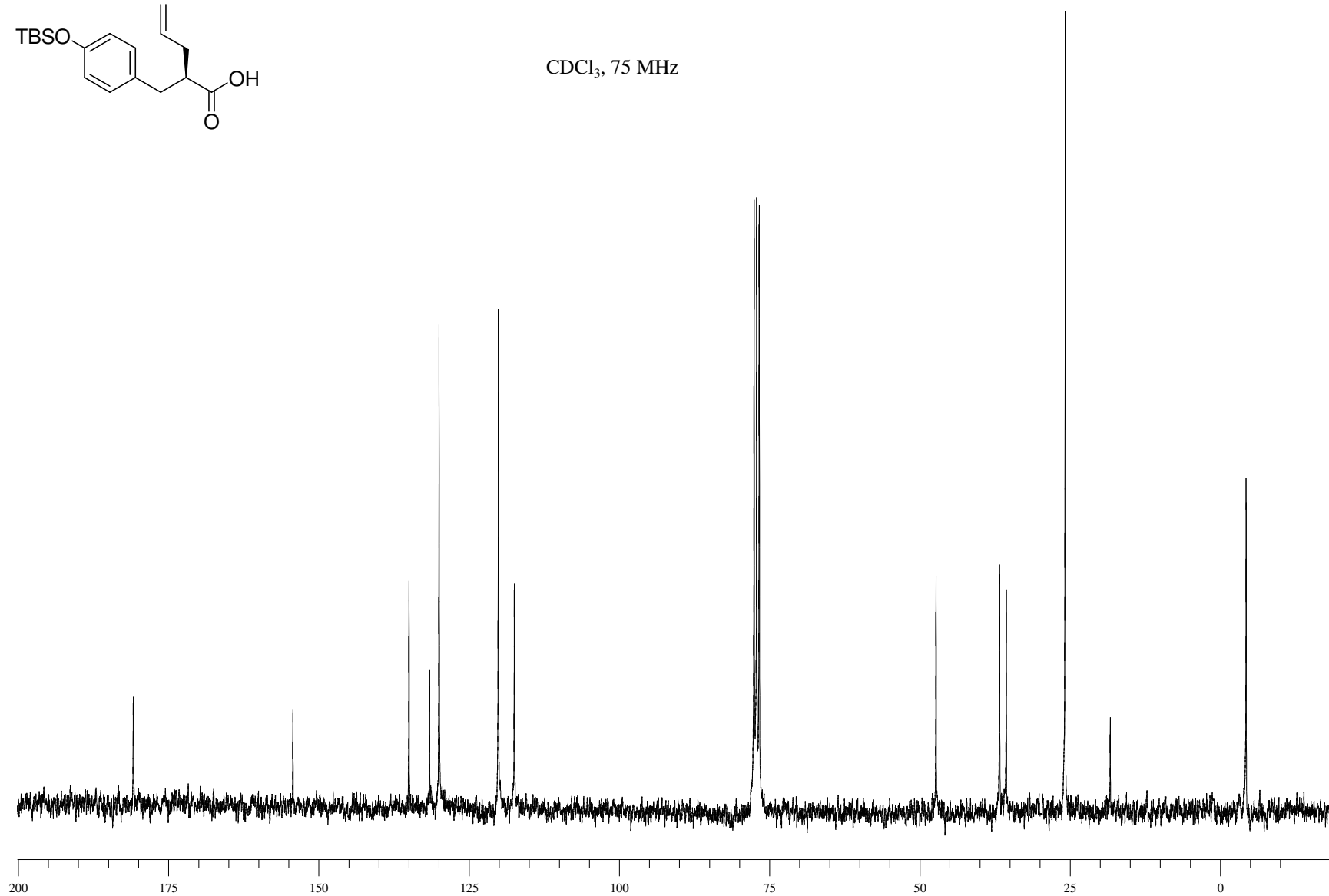


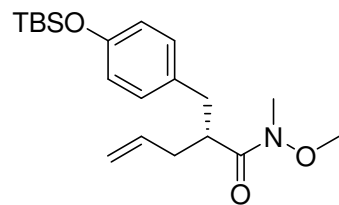
CDCl<sub>3</sub>, 300 MHz



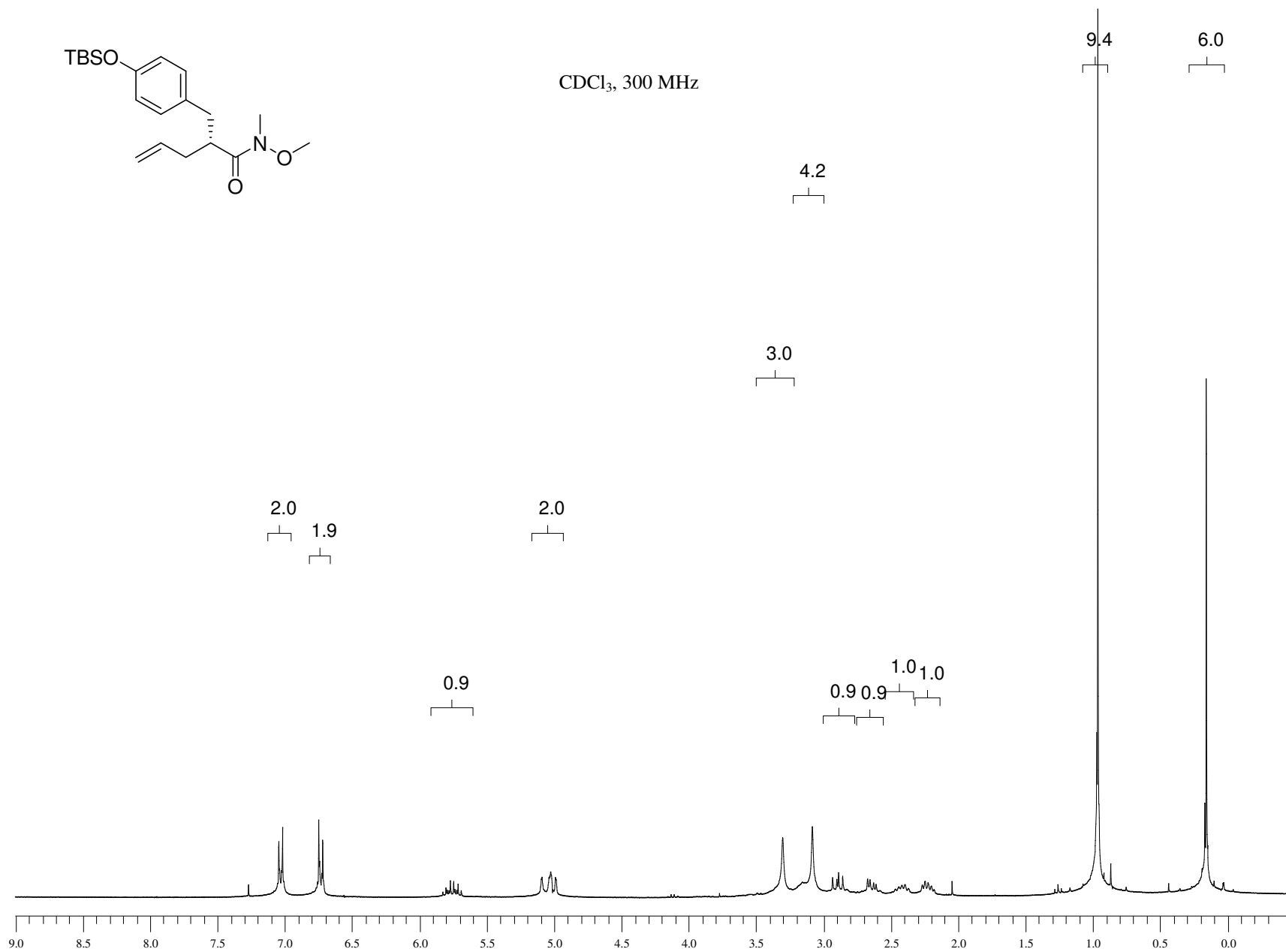


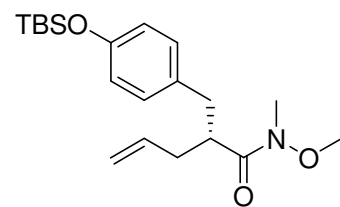
CDCl<sub>3</sub>, 75 MHz



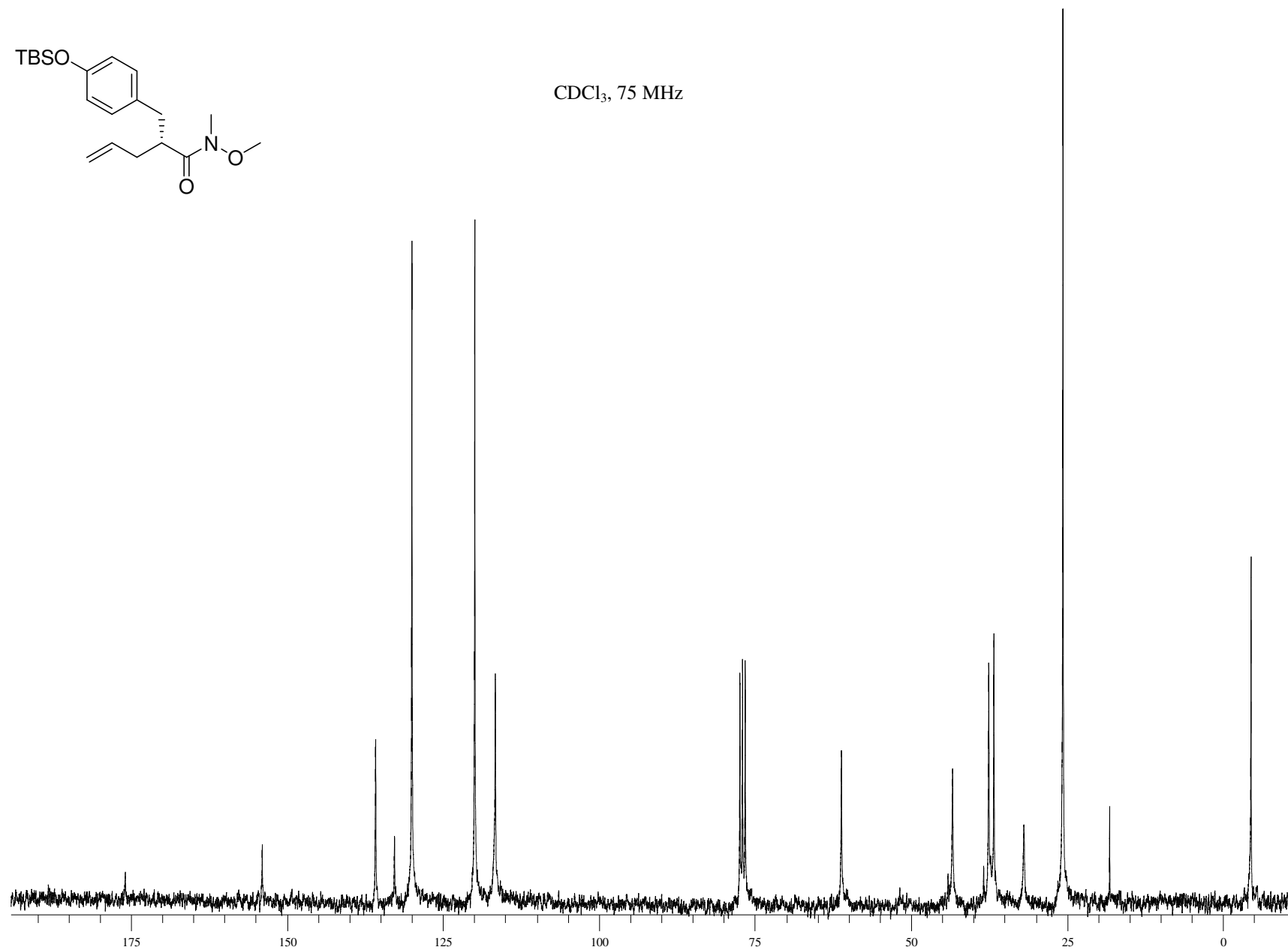


CDCl<sub>3</sub>, 300 MHz

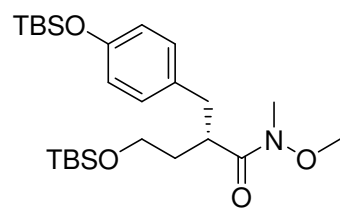




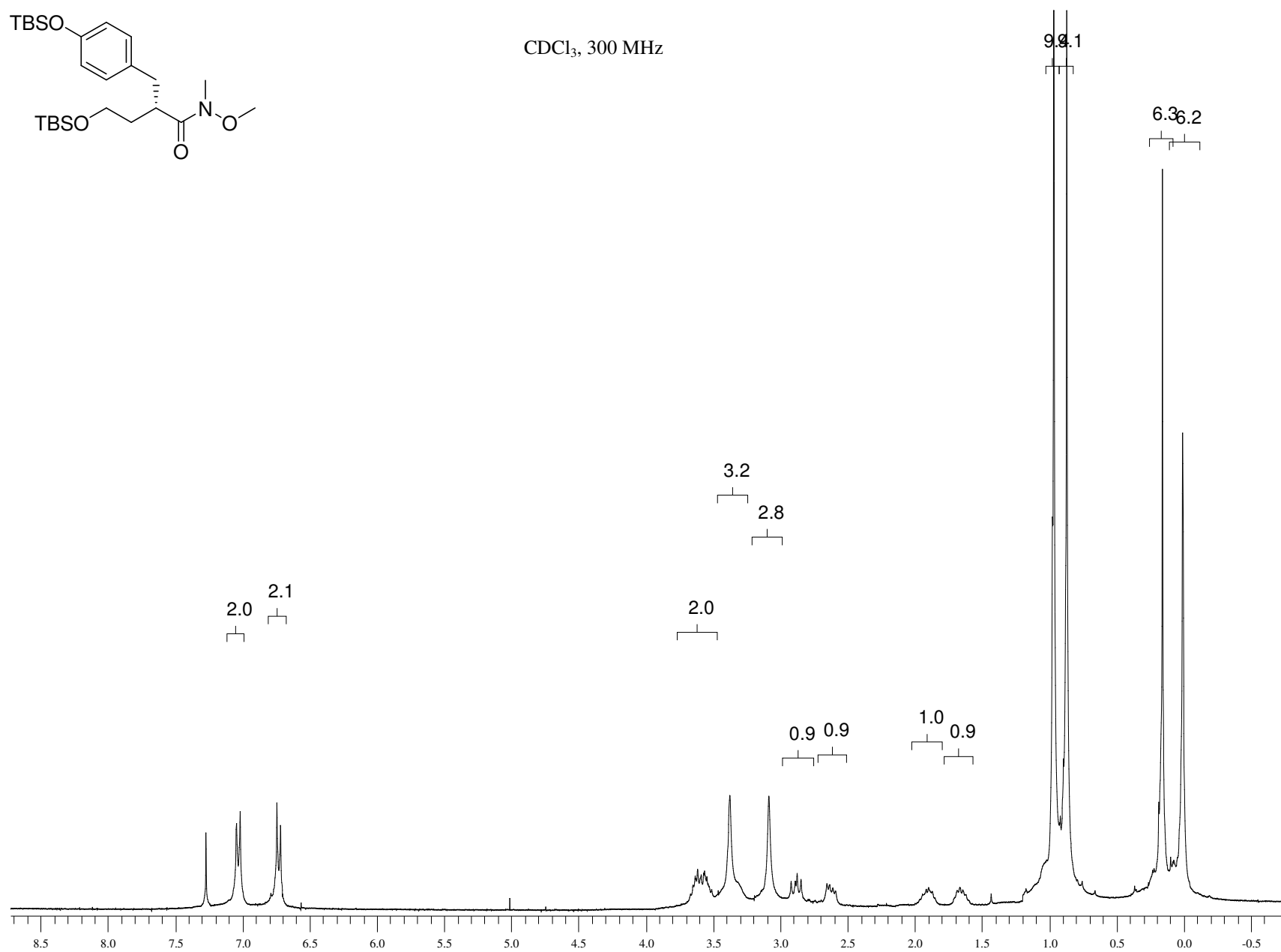
CDCl<sub>3</sub>, 75 MHz

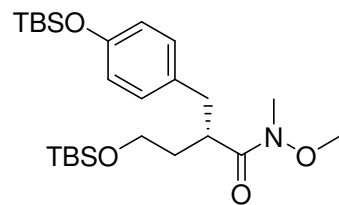




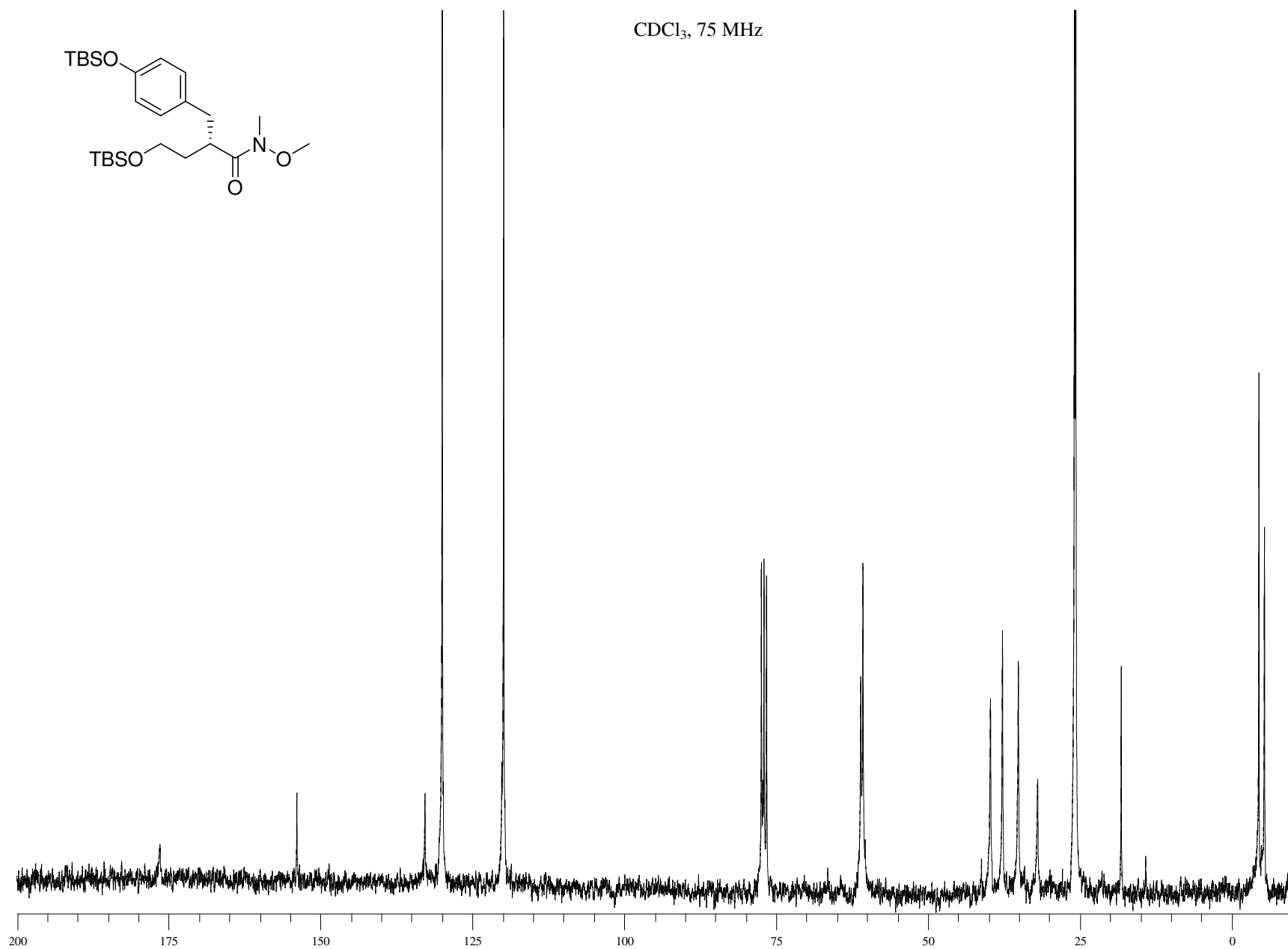


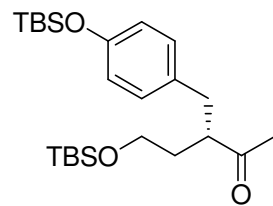
CDCl<sub>3</sub>, 300 MHz



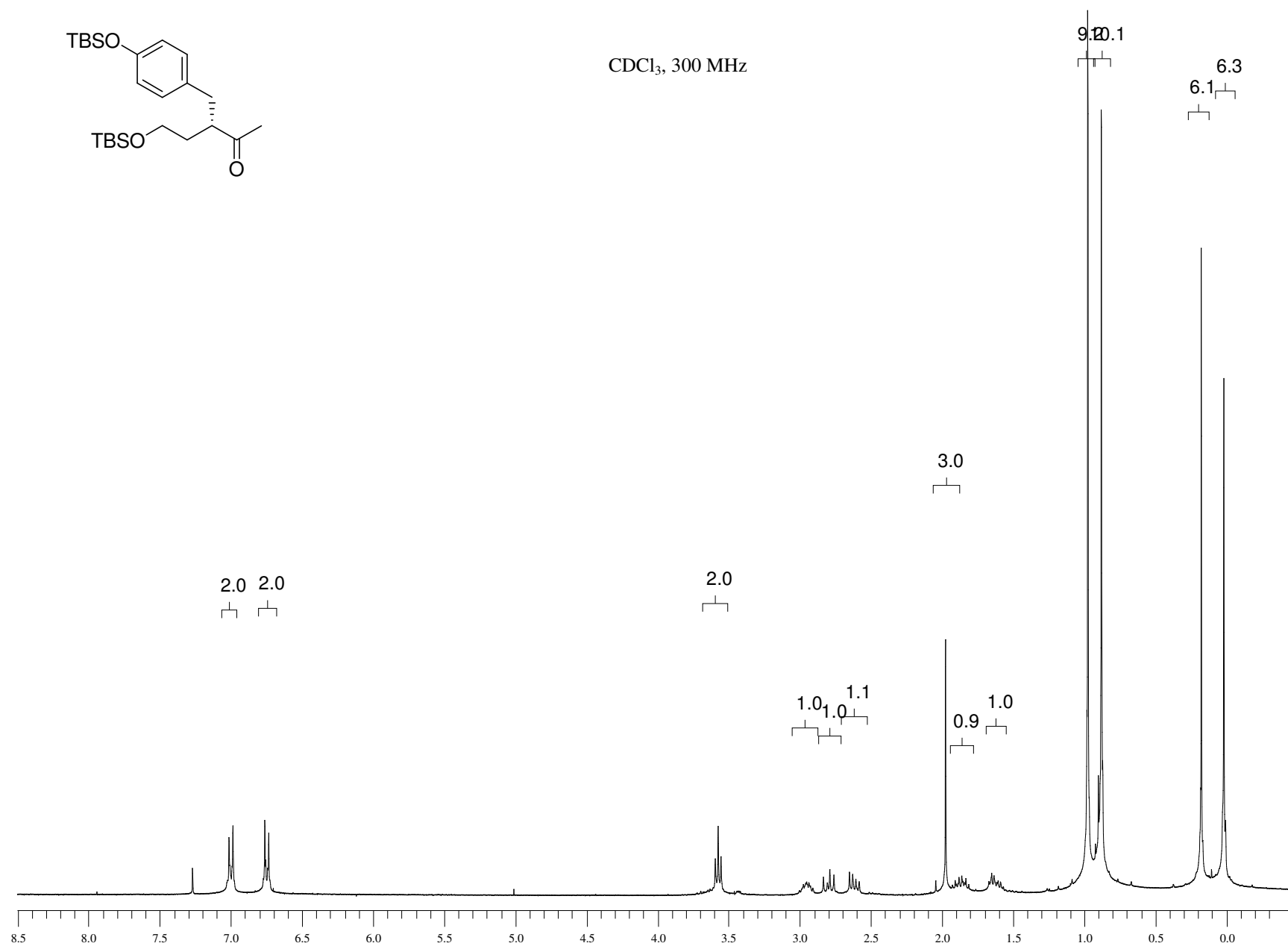


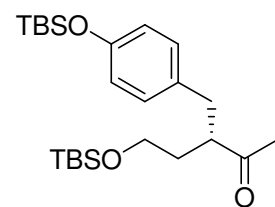
CDCl<sub>3</sub>, 75 MHz



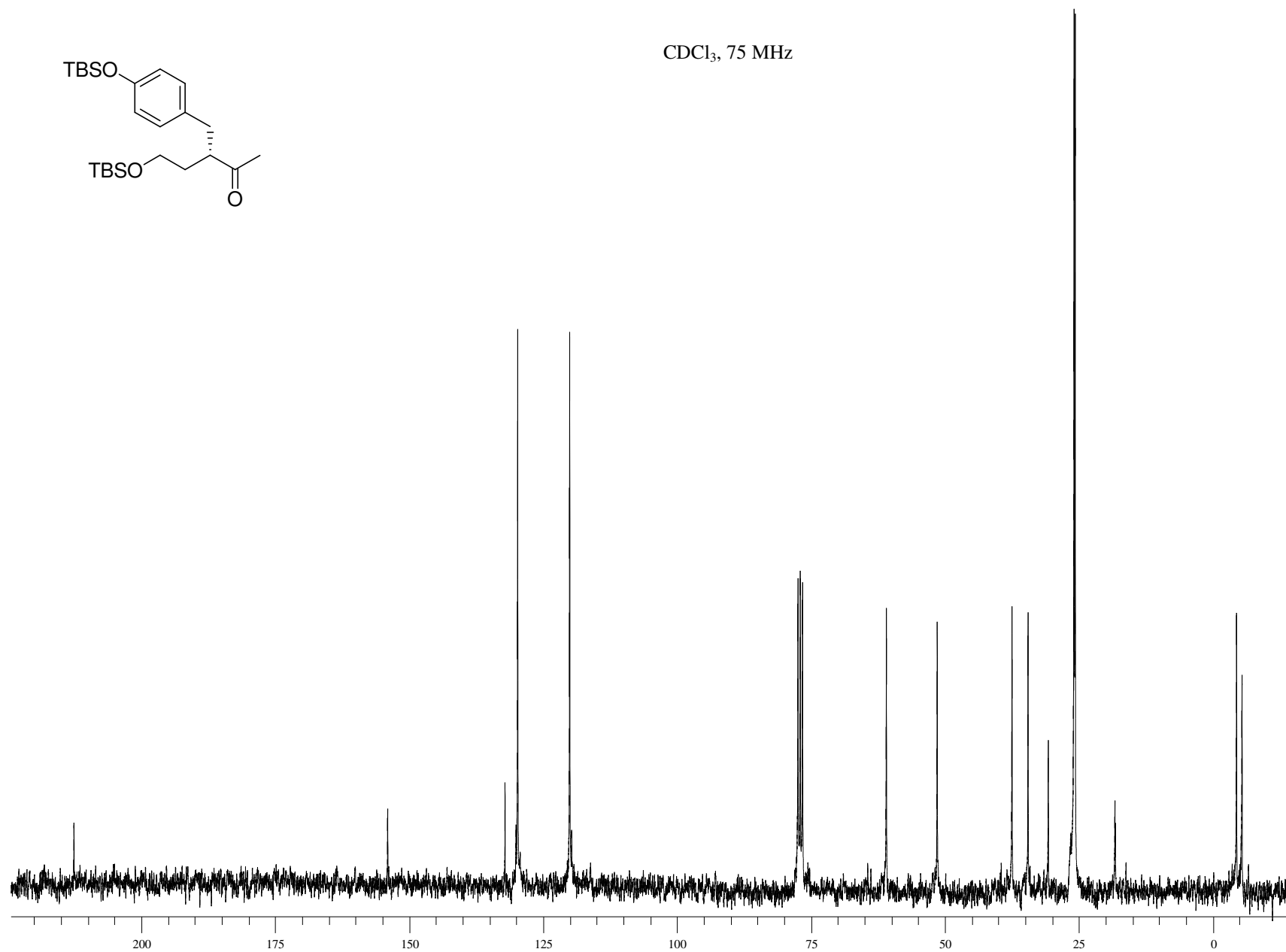


CDCl<sub>3</sub>, 300 MHz

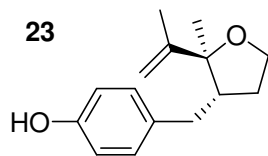




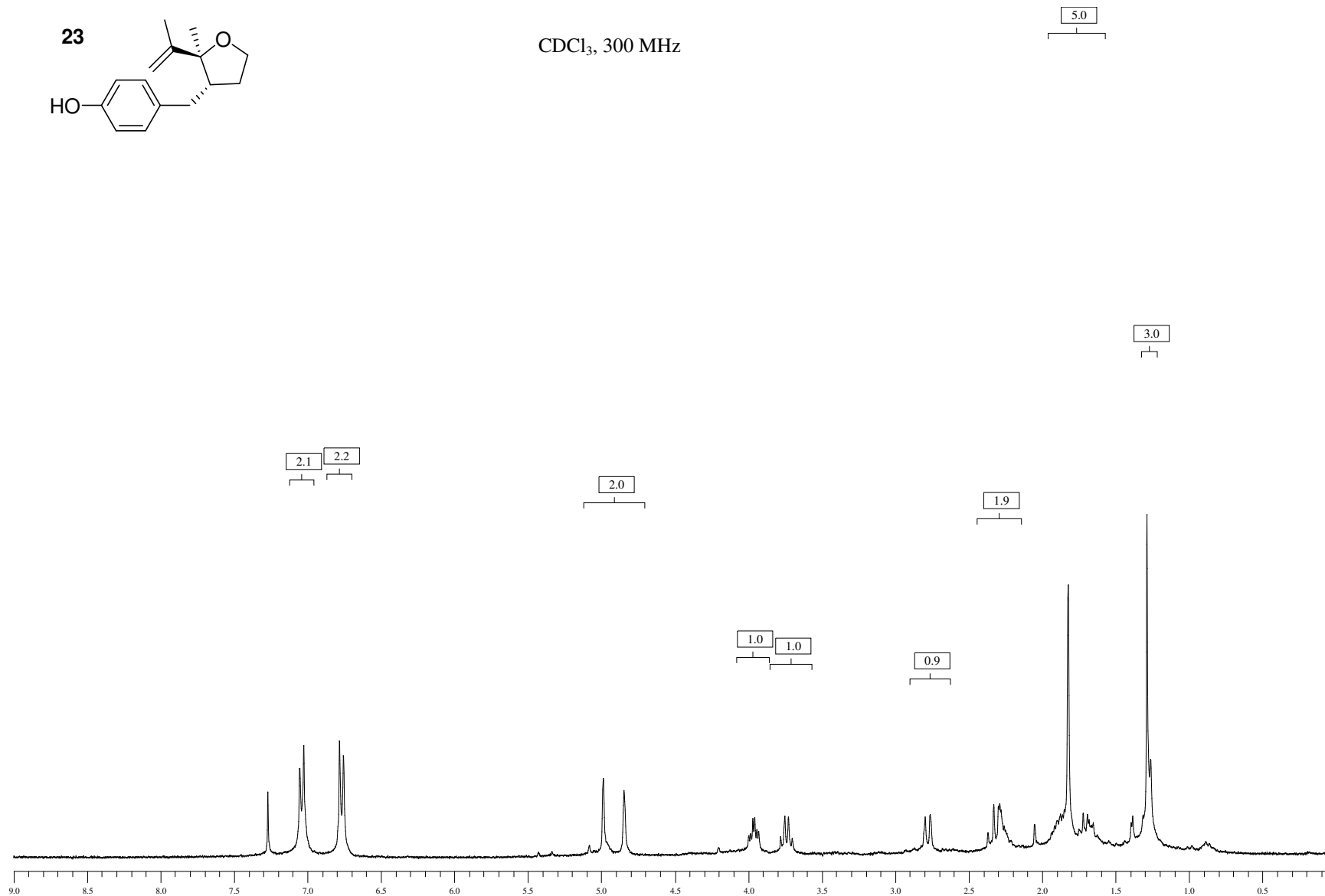
CDCl<sub>3</sub>, 75 MHz



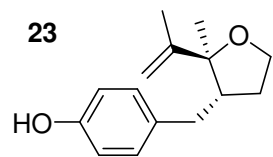
**23**



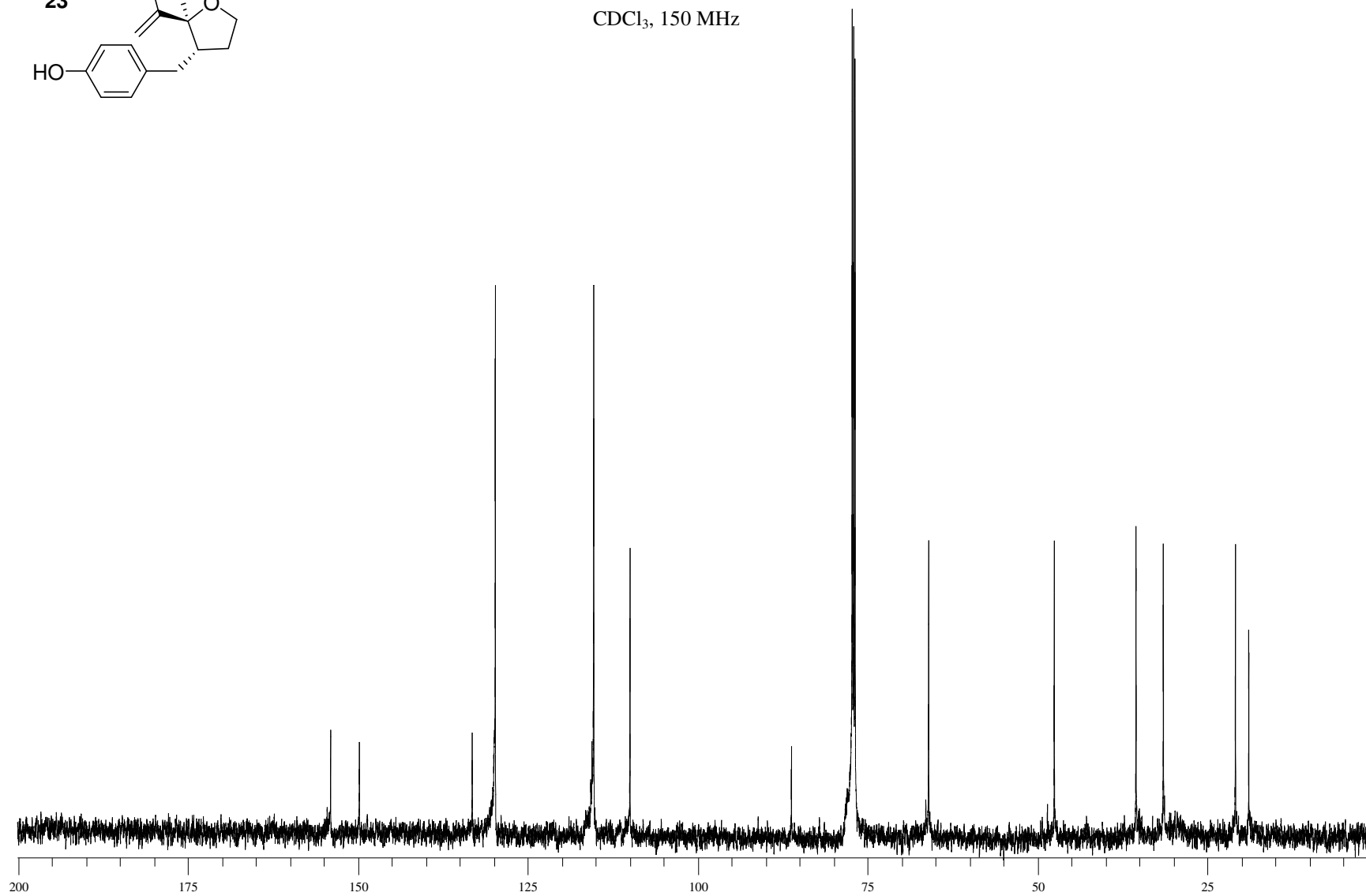
CDCl<sub>3</sub>, 300 MHz



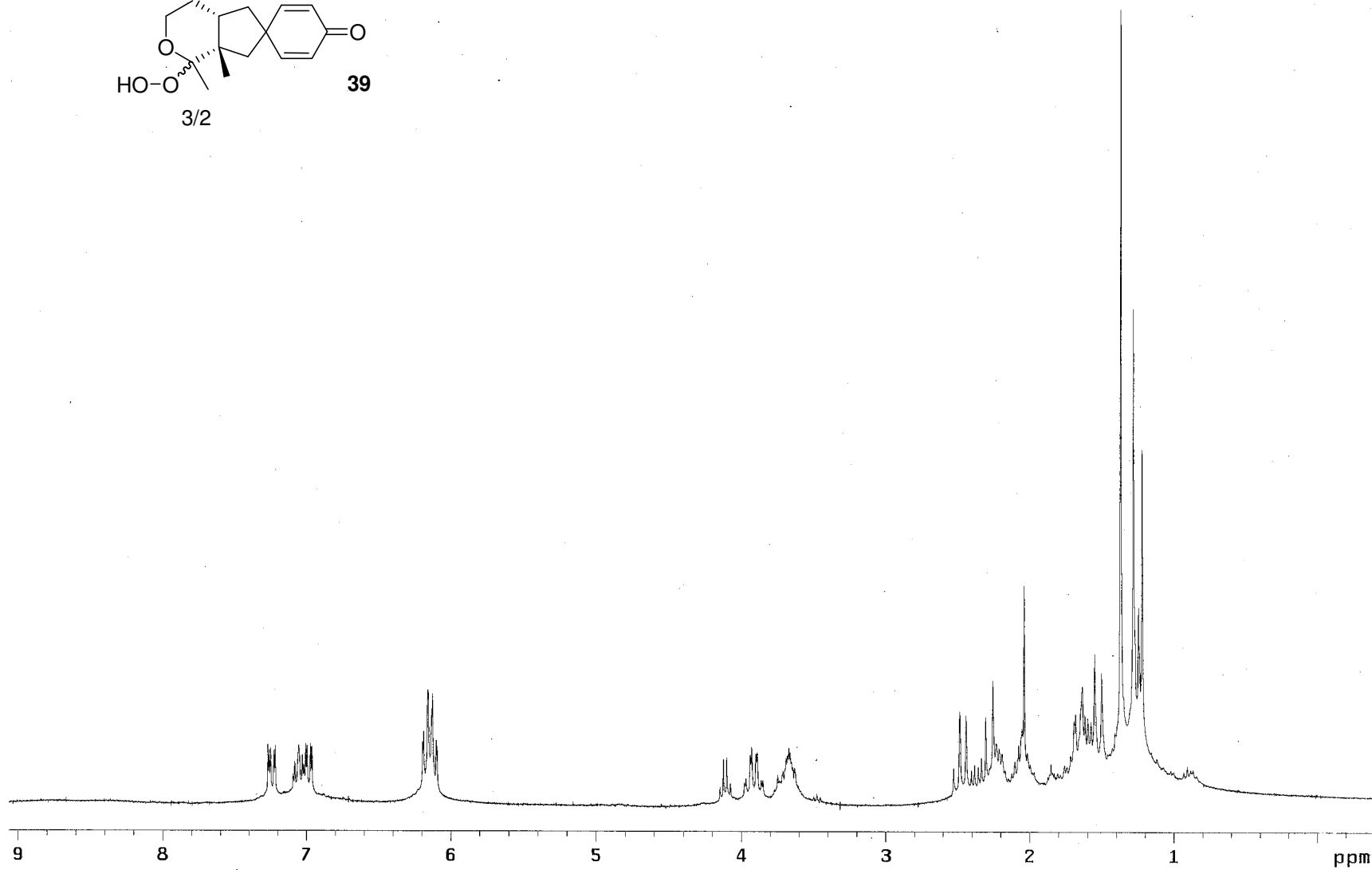
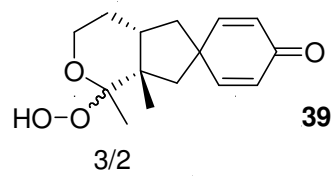
**23**

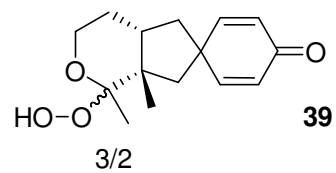


CDCl<sub>3</sub>, 150 MHz

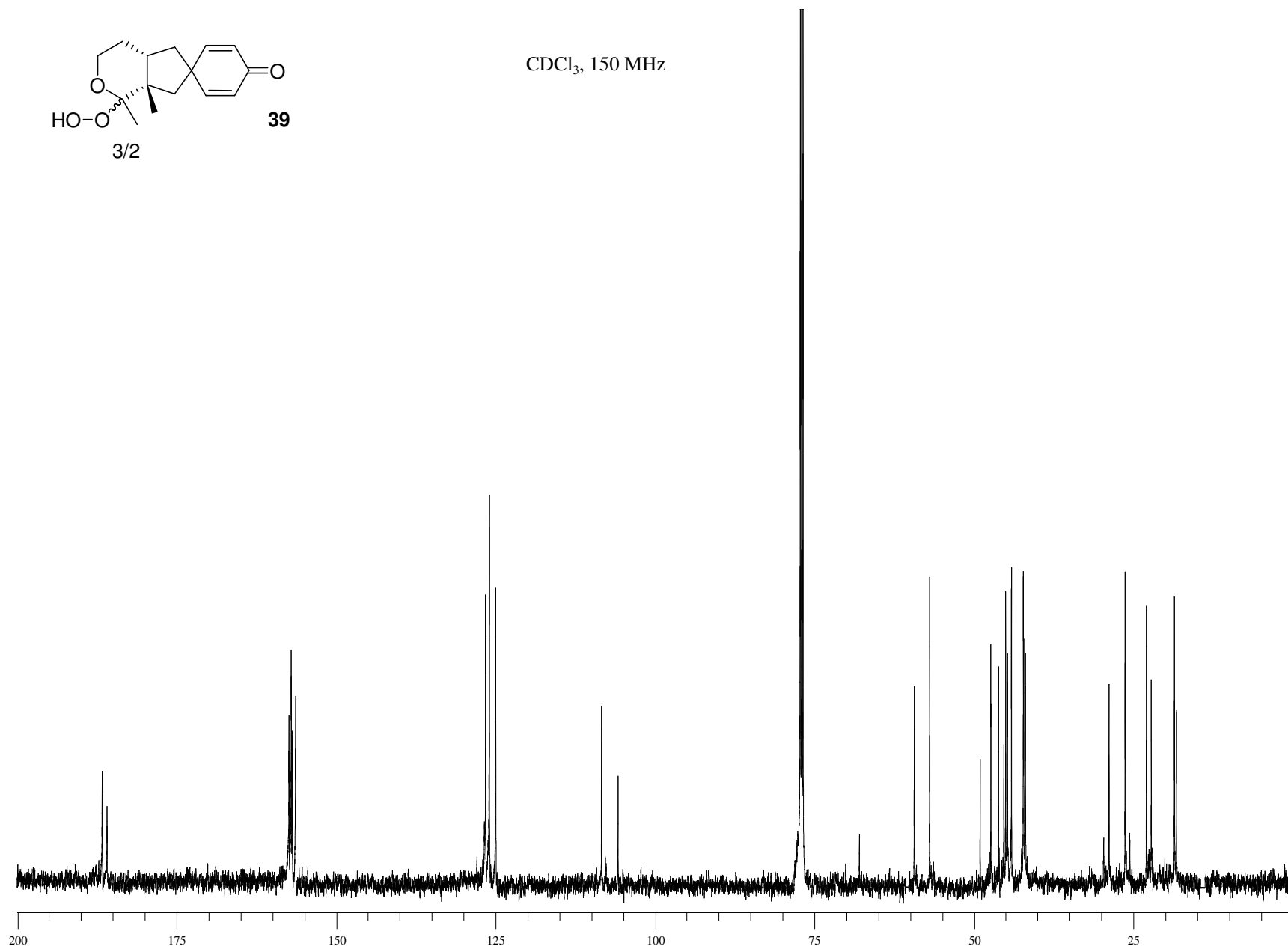


CDCl<sub>3</sub>, 300 MHz

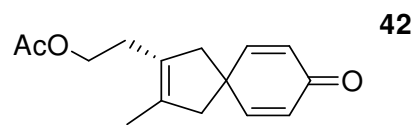




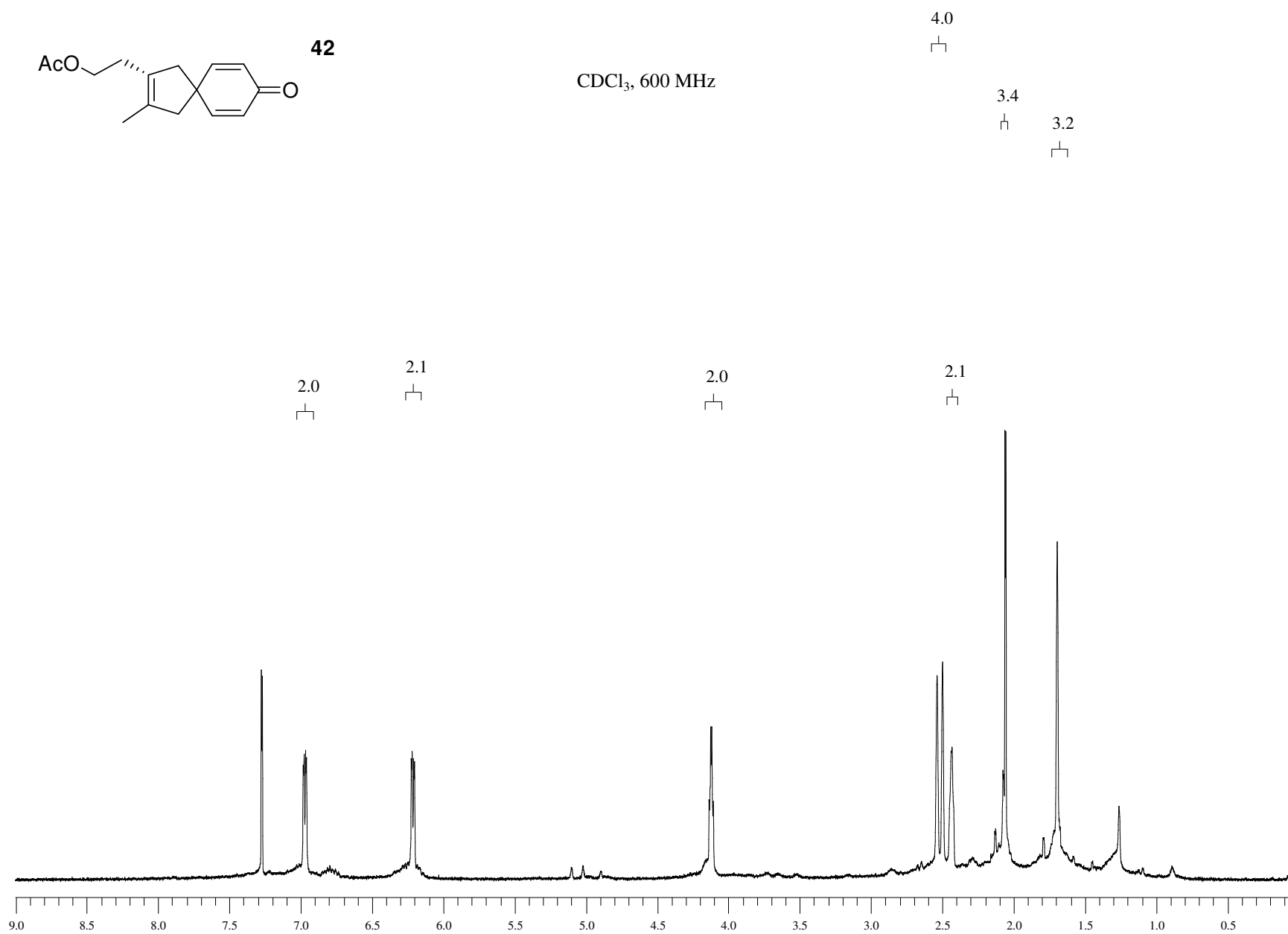
CDCl<sub>3</sub>, 150 MHz

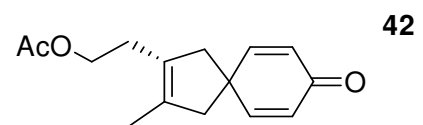




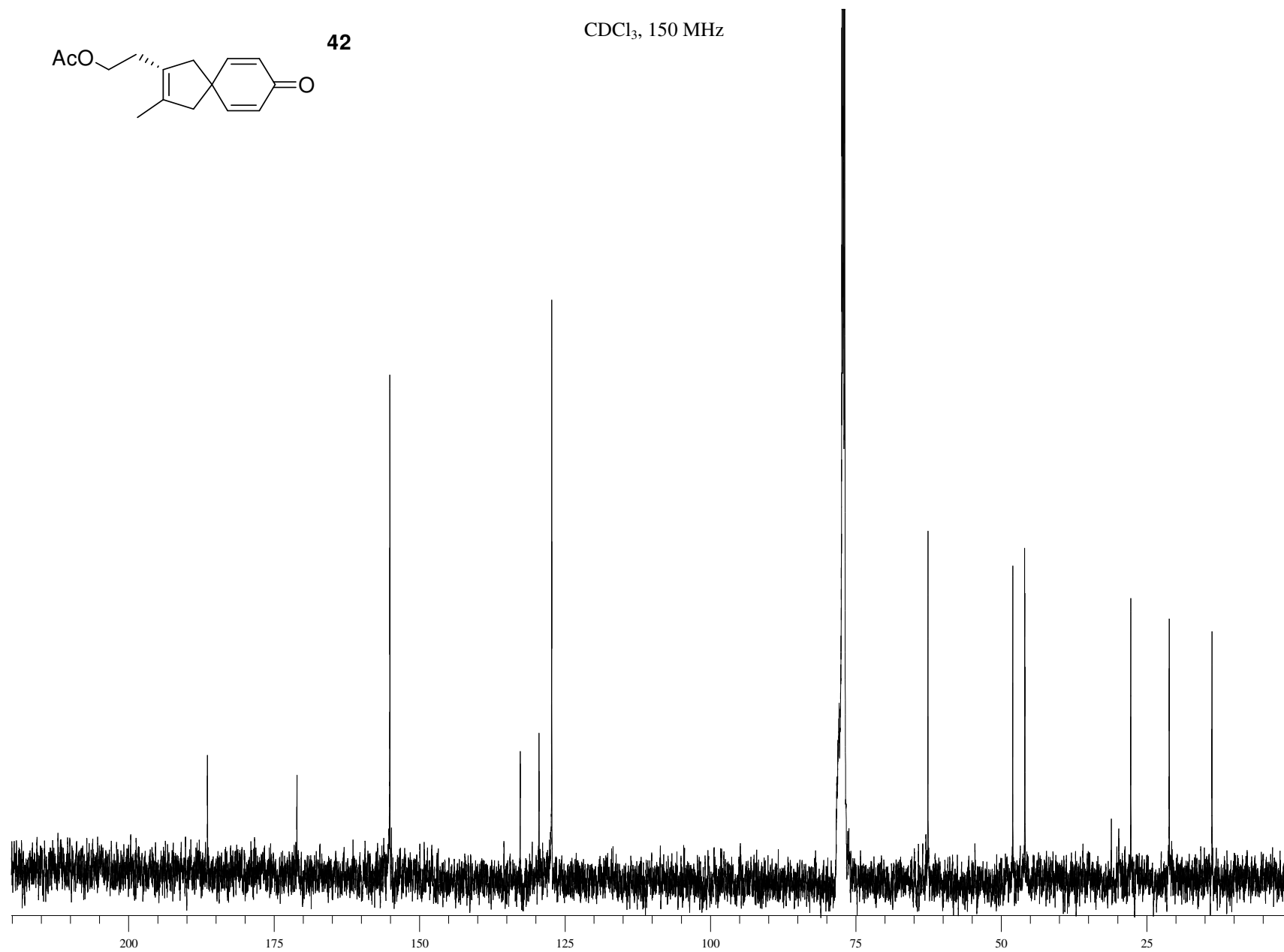


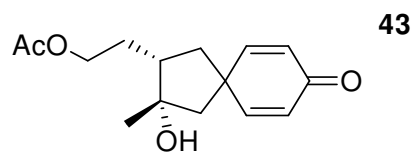
CDCl<sub>3</sub>, 600 MHz





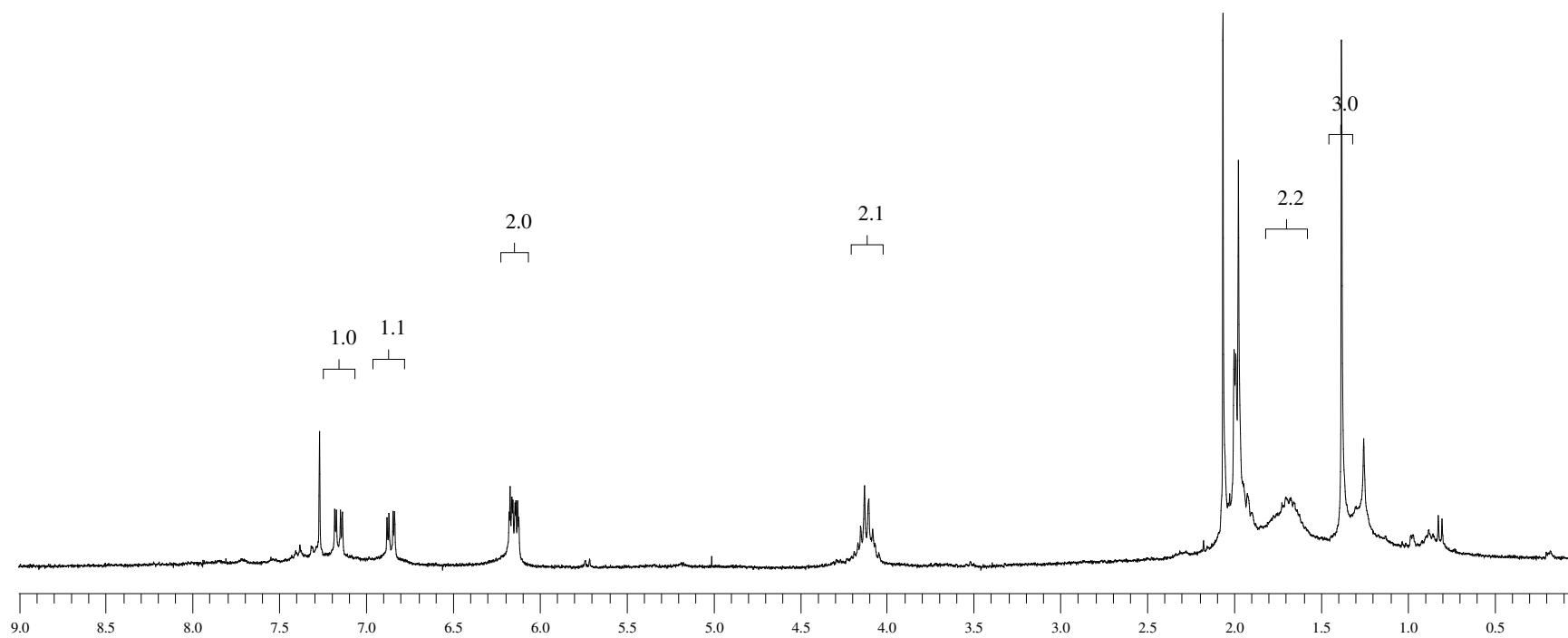
CDCl<sub>3</sub>, 150 MHz

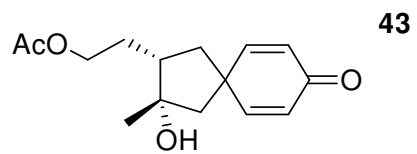




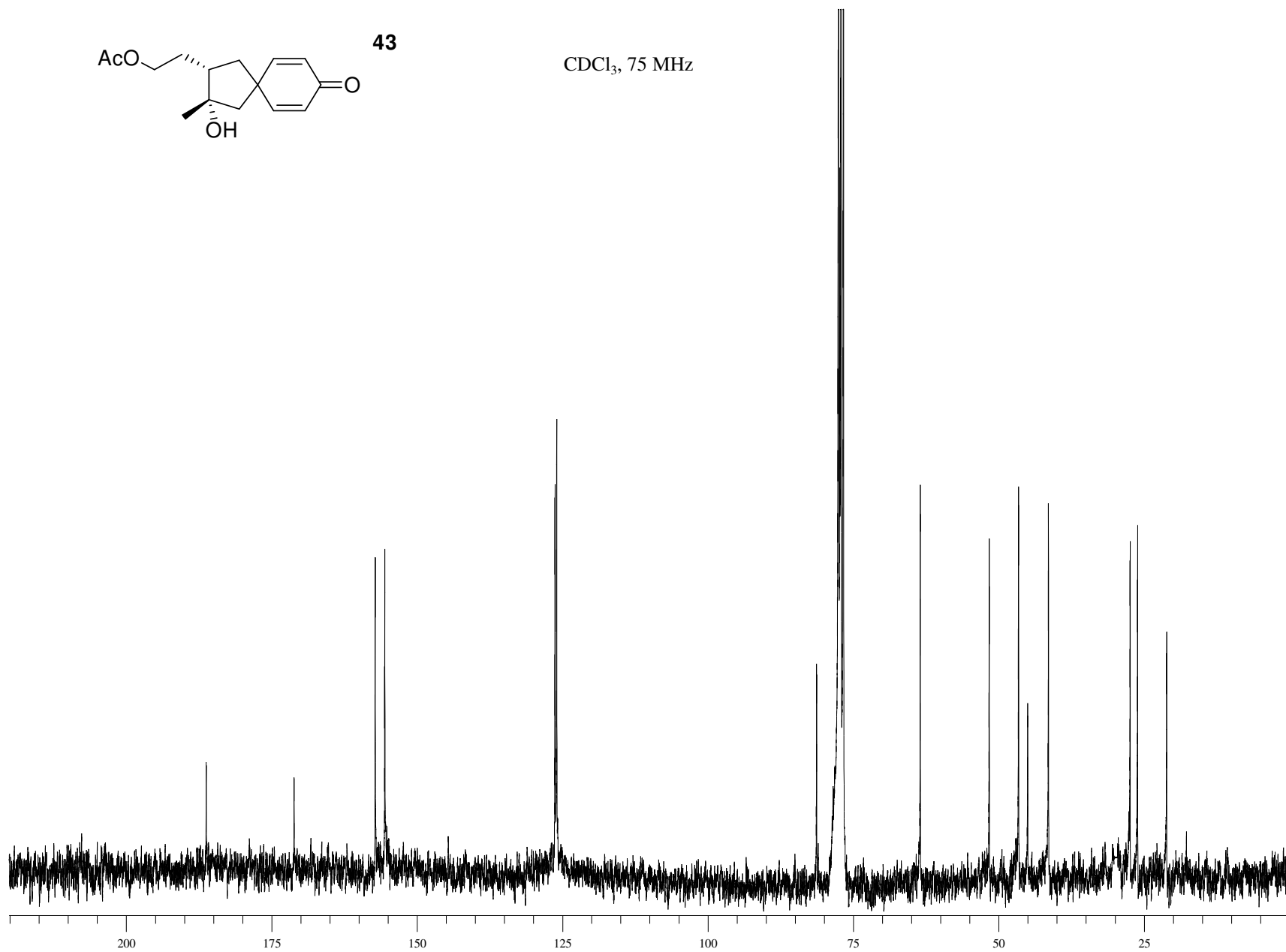
CDCl<sub>3</sub>, 300 MHz

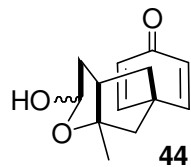
10.3



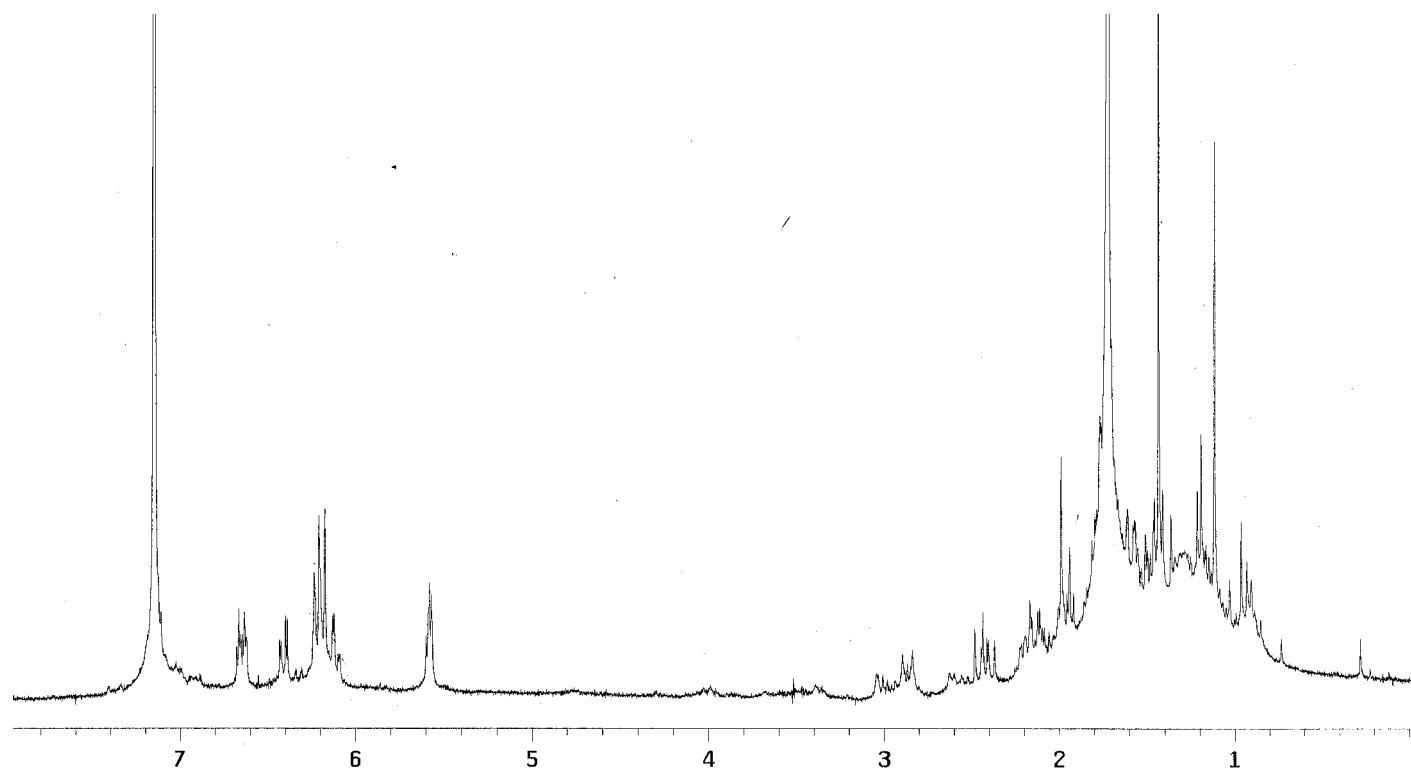


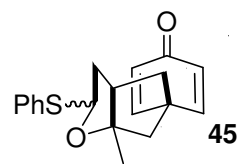
CDCl<sub>3</sub>, 75 MHz



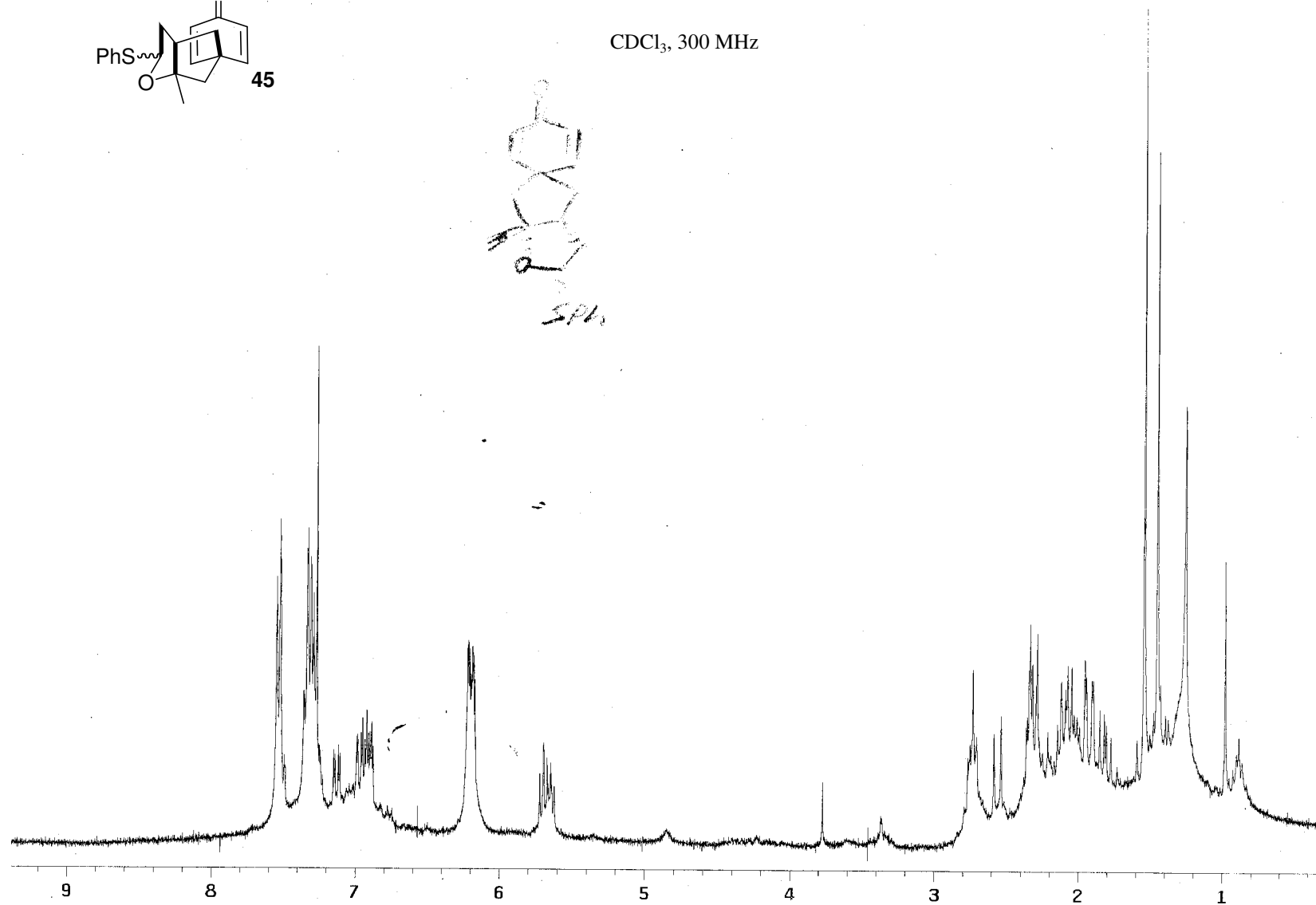
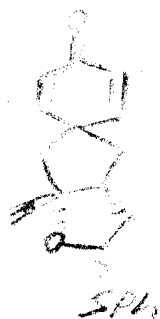


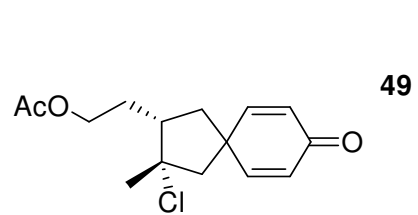
C<sub>6</sub>D<sub>6</sub>, 300 MHz



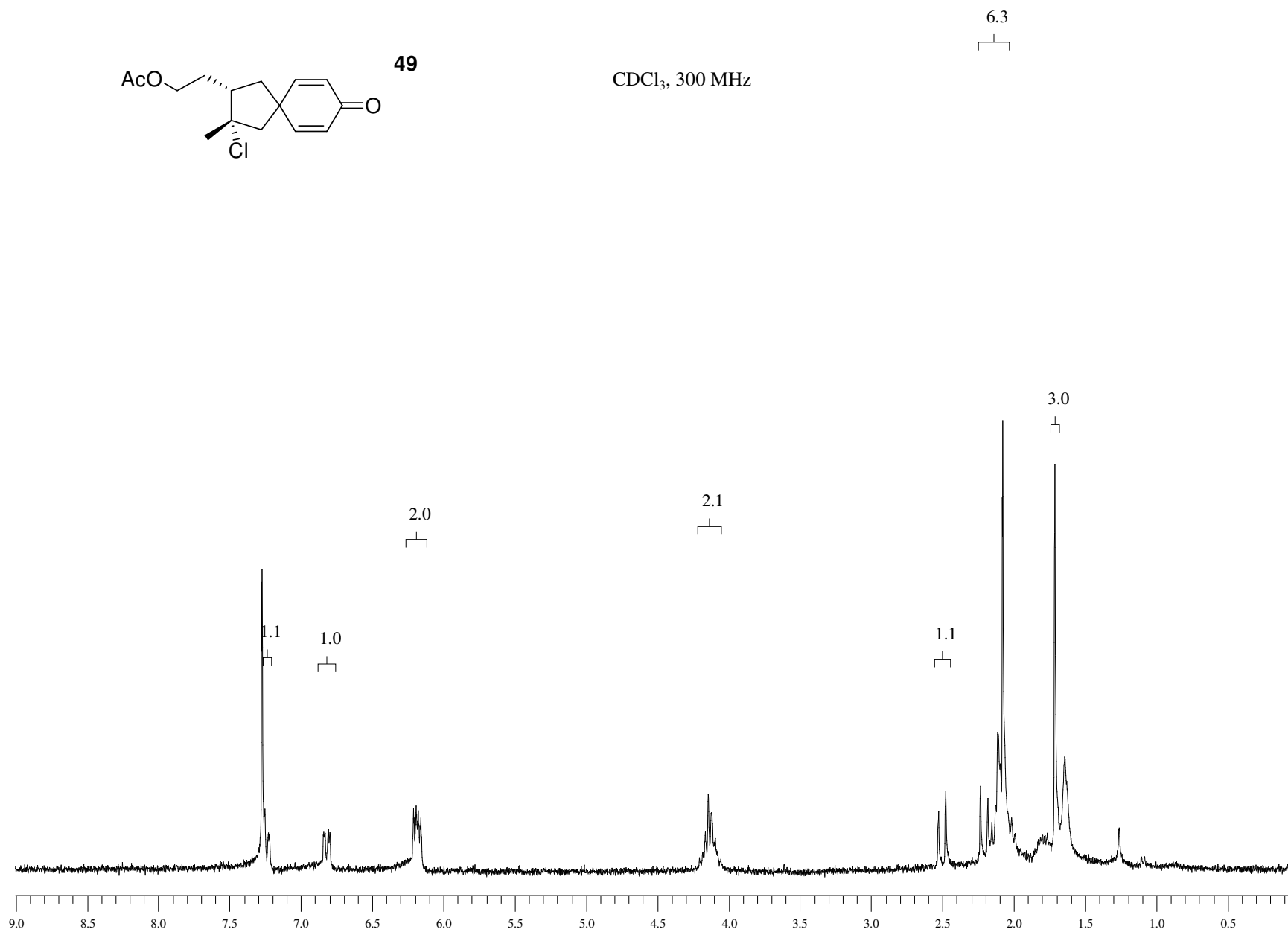


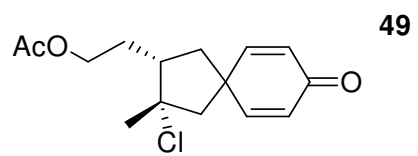
CDCl<sub>3</sub>, 300 MHz



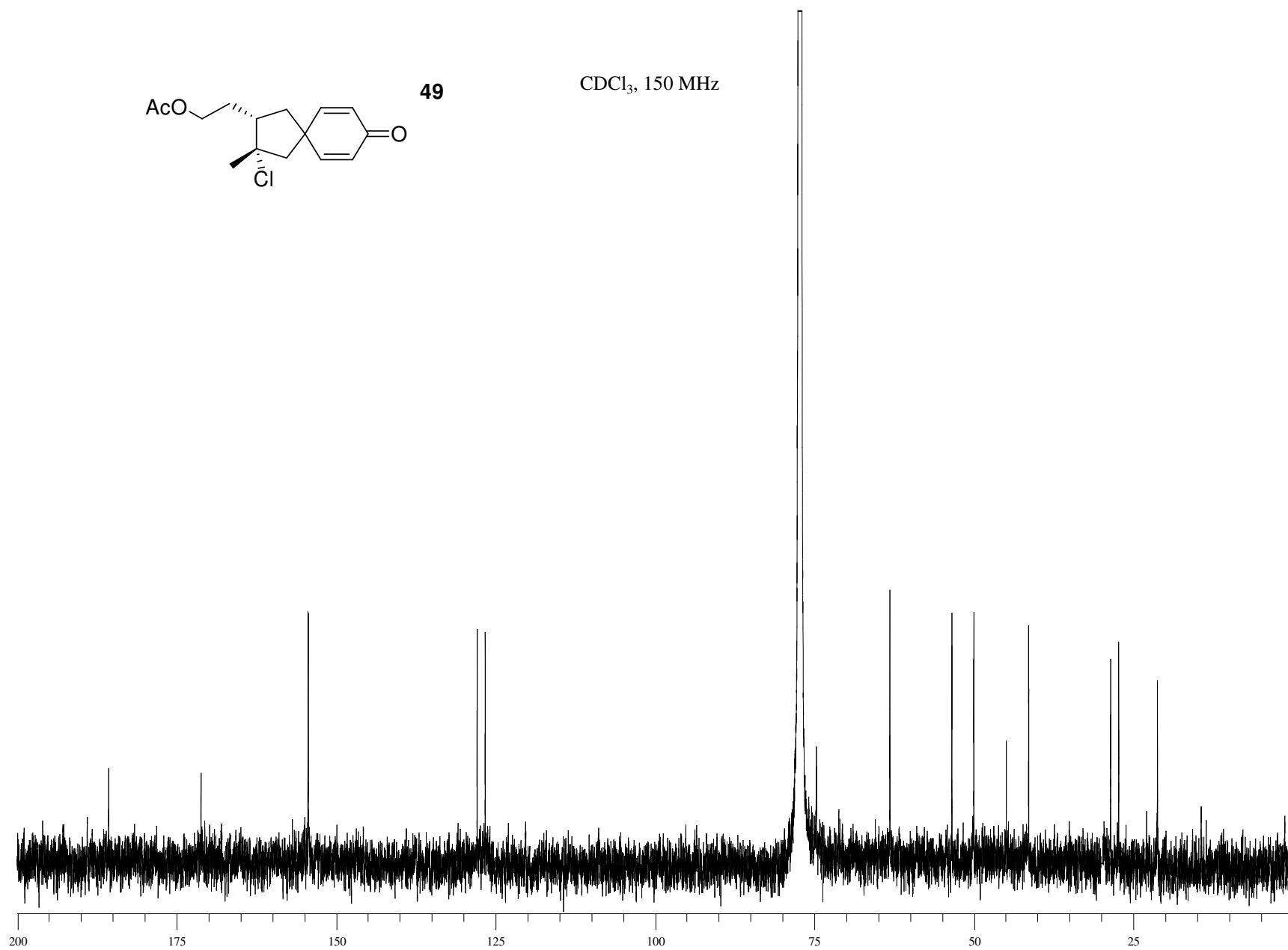


CDCl<sub>3</sub>, 300 MHz

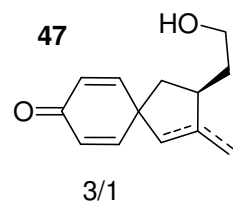




CDCl<sub>3</sub>, 150 MHz

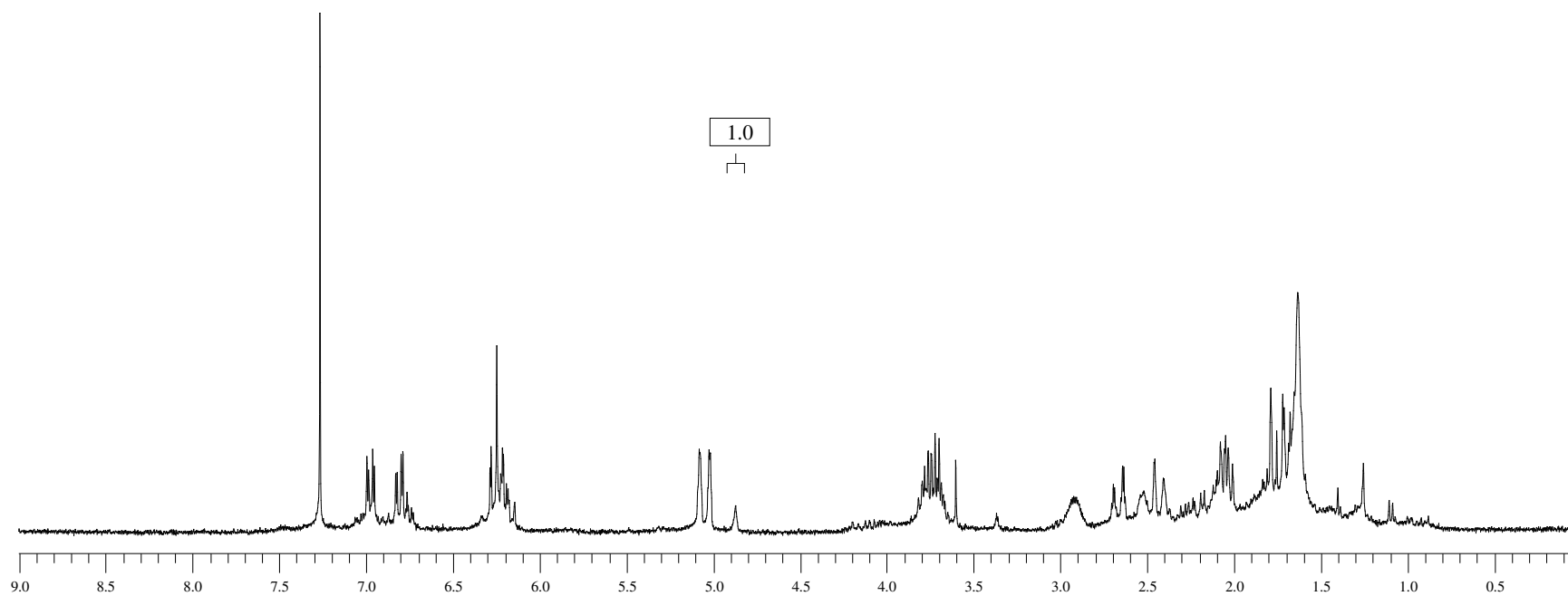


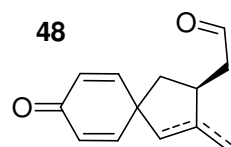




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CDCl<sub>3</sub>, 300 MHz



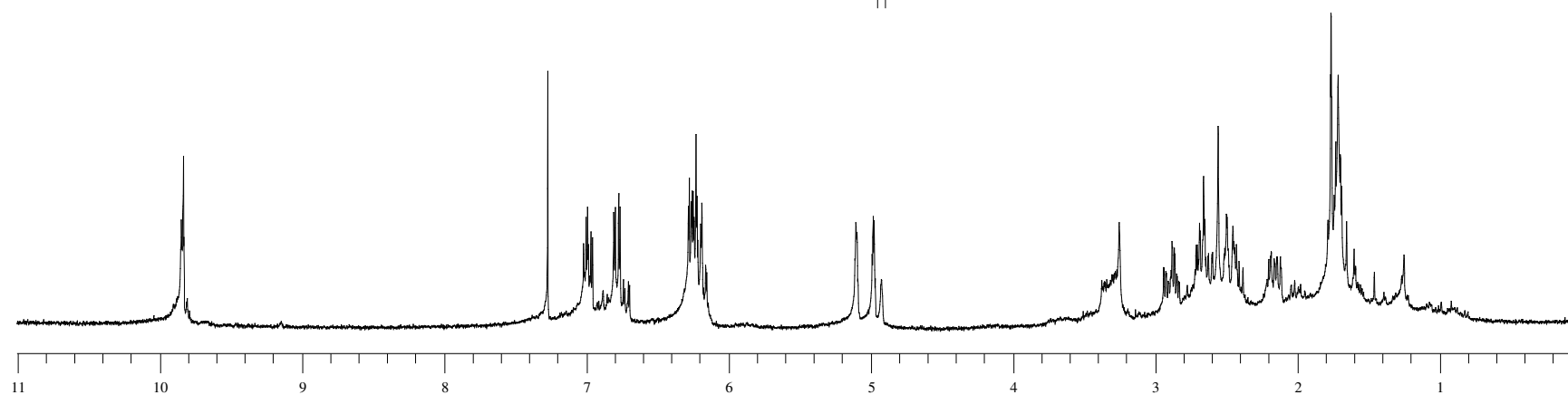


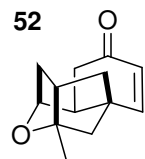
3/1

CDCl<sub>3</sub>, 300 MHz

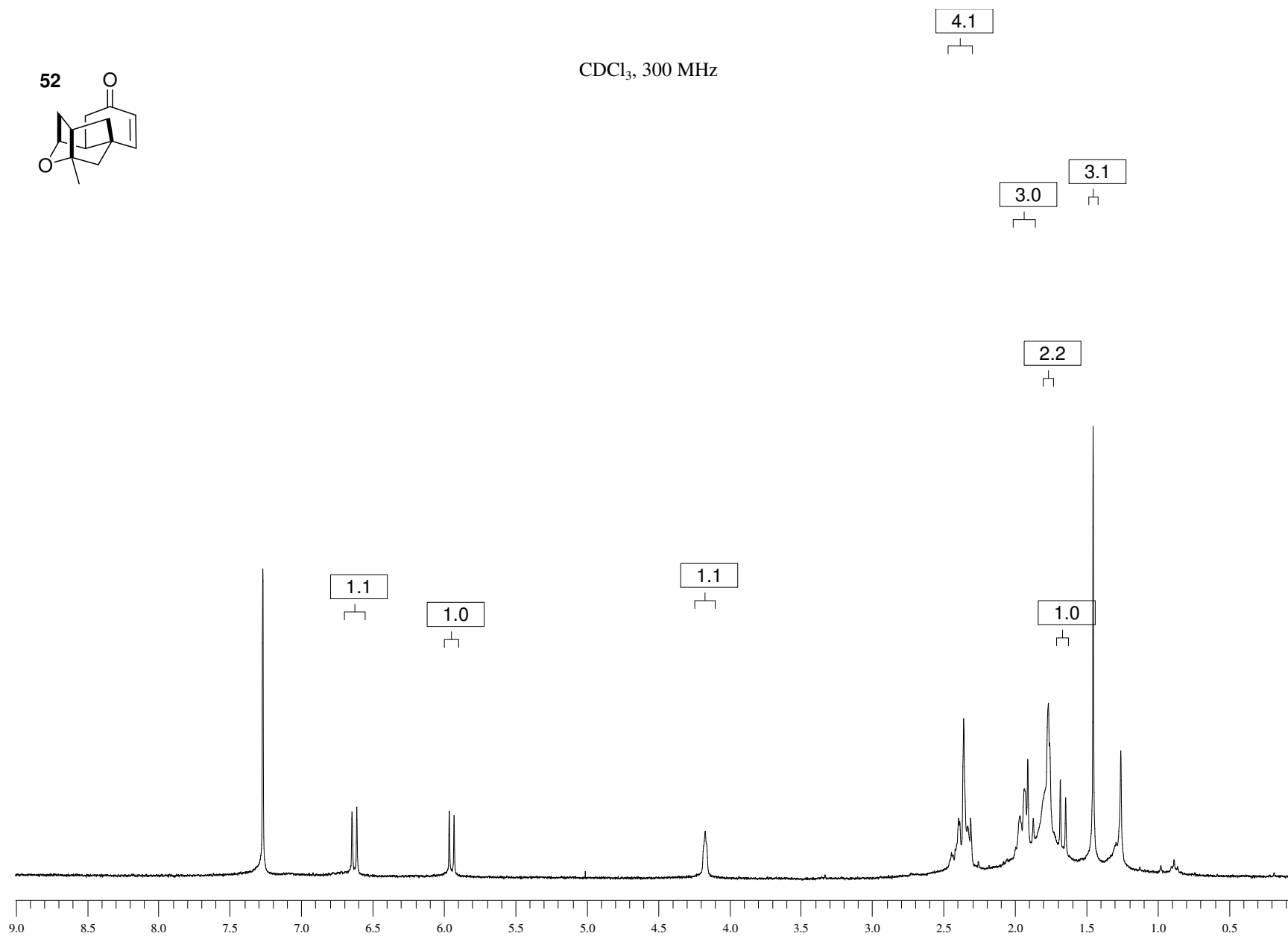
3.0  
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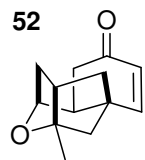
1.0  
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CDCl<sub>3</sub>, 300 MHz





CDCl<sub>3</sub>, 600 MHz

