



Supporting Information

for

Bifurcated synthesis of methylene-lactone- and methylene-lactam-fused spirolactams via electrophilic amide allylation of γ -phenylthio-functionalized γ -lactams

Tetsuya Sengoku, Koki Makino, Ayumi Iijima, Toshiyasu Inuzuka and Hidemi Yoda

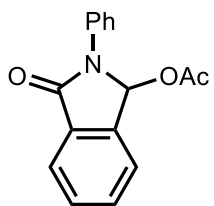
Beilstein J. Org. Chem. **2020**, *16*, 2769–2775. [doi:10.3762/bjoc.16.227](https://doi.org/10.3762/bjoc.16.227)

Copies of ^1H and ^{13}C NMR spectra of all new compounds

The following data are included in this material:

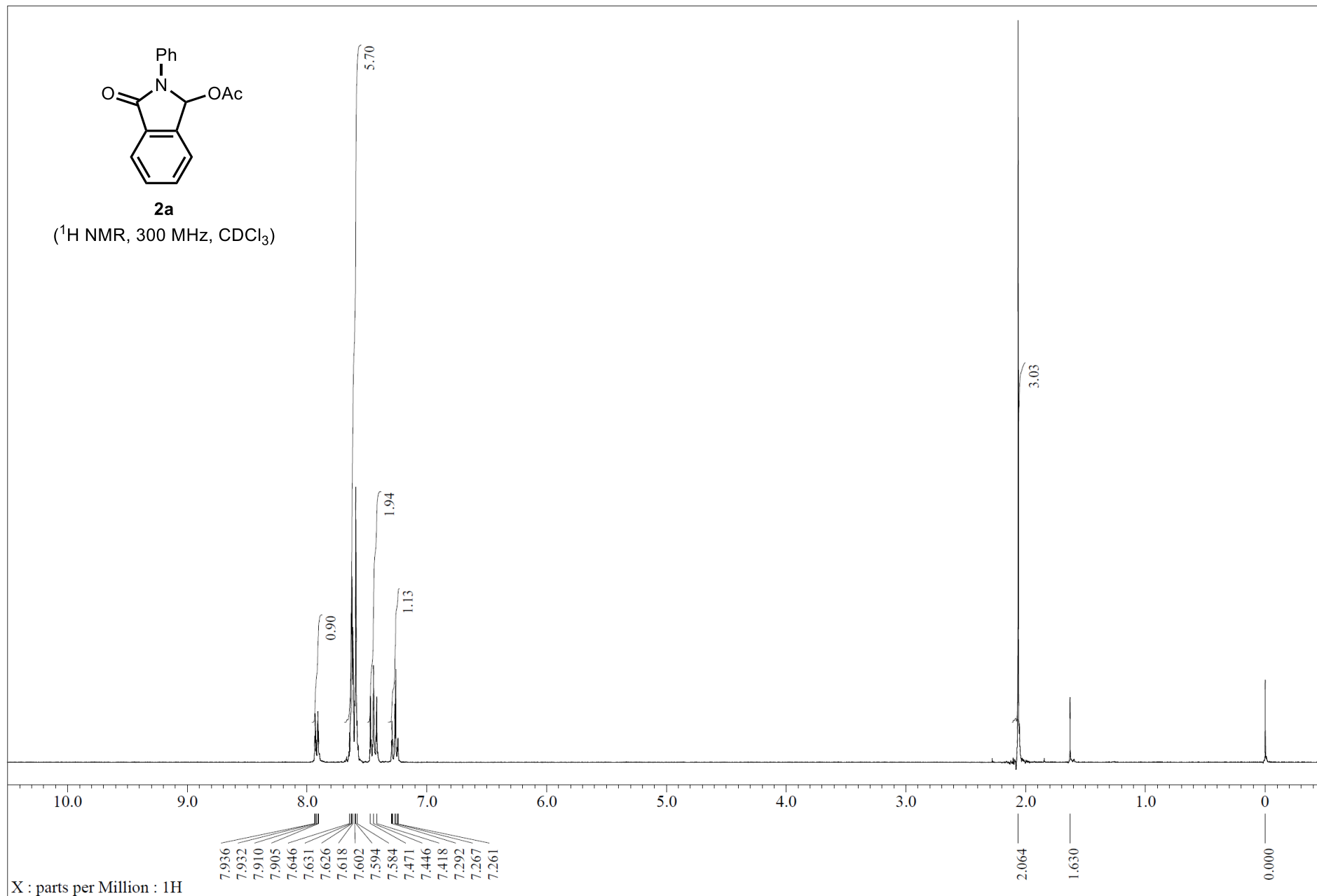
Copies of ^1H and ^{13}C NMR spectra

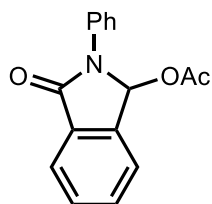
S2–S141



2a

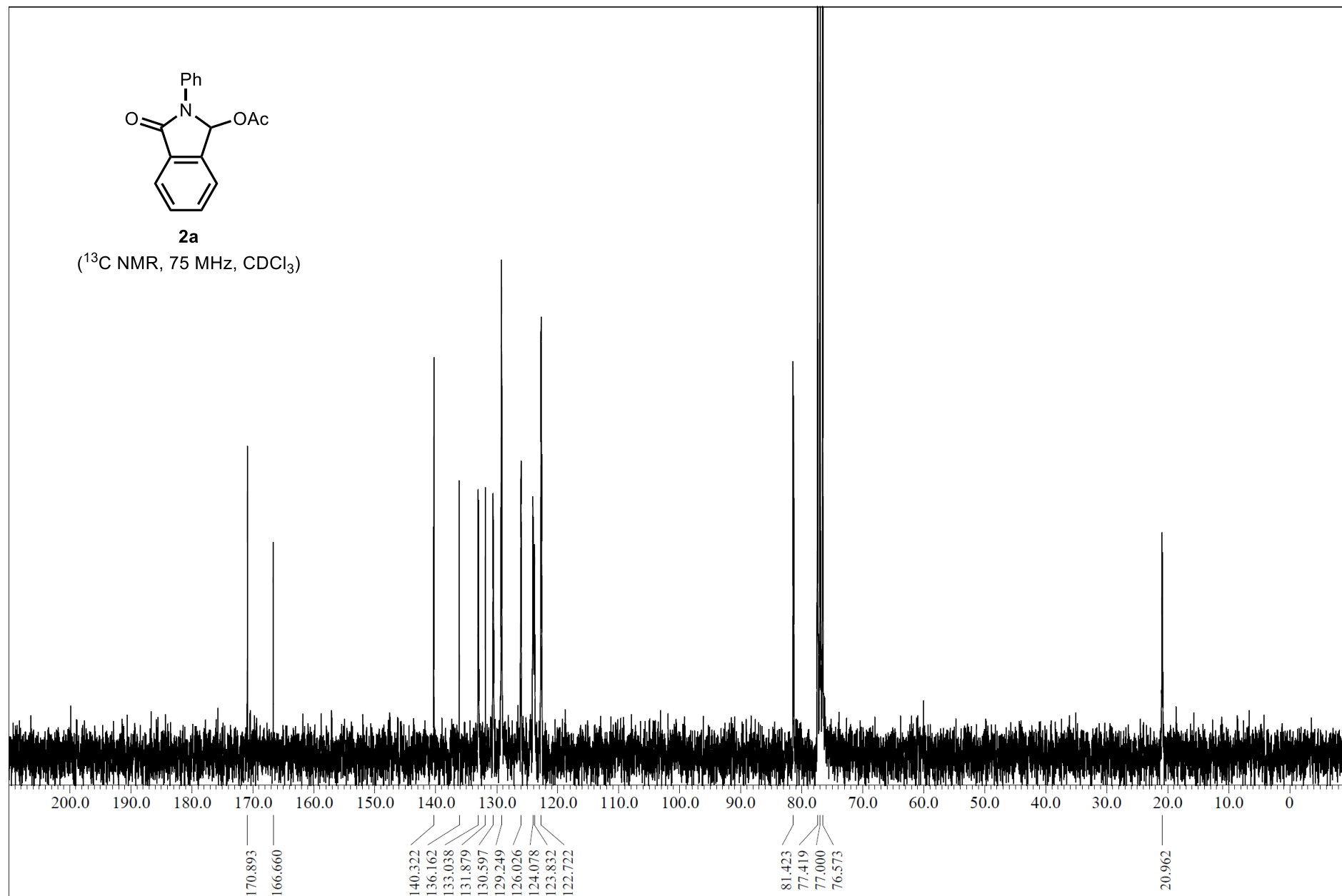
(¹H NMR, 300 MHz, CDCl₃)



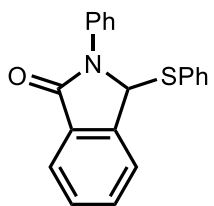


2a

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

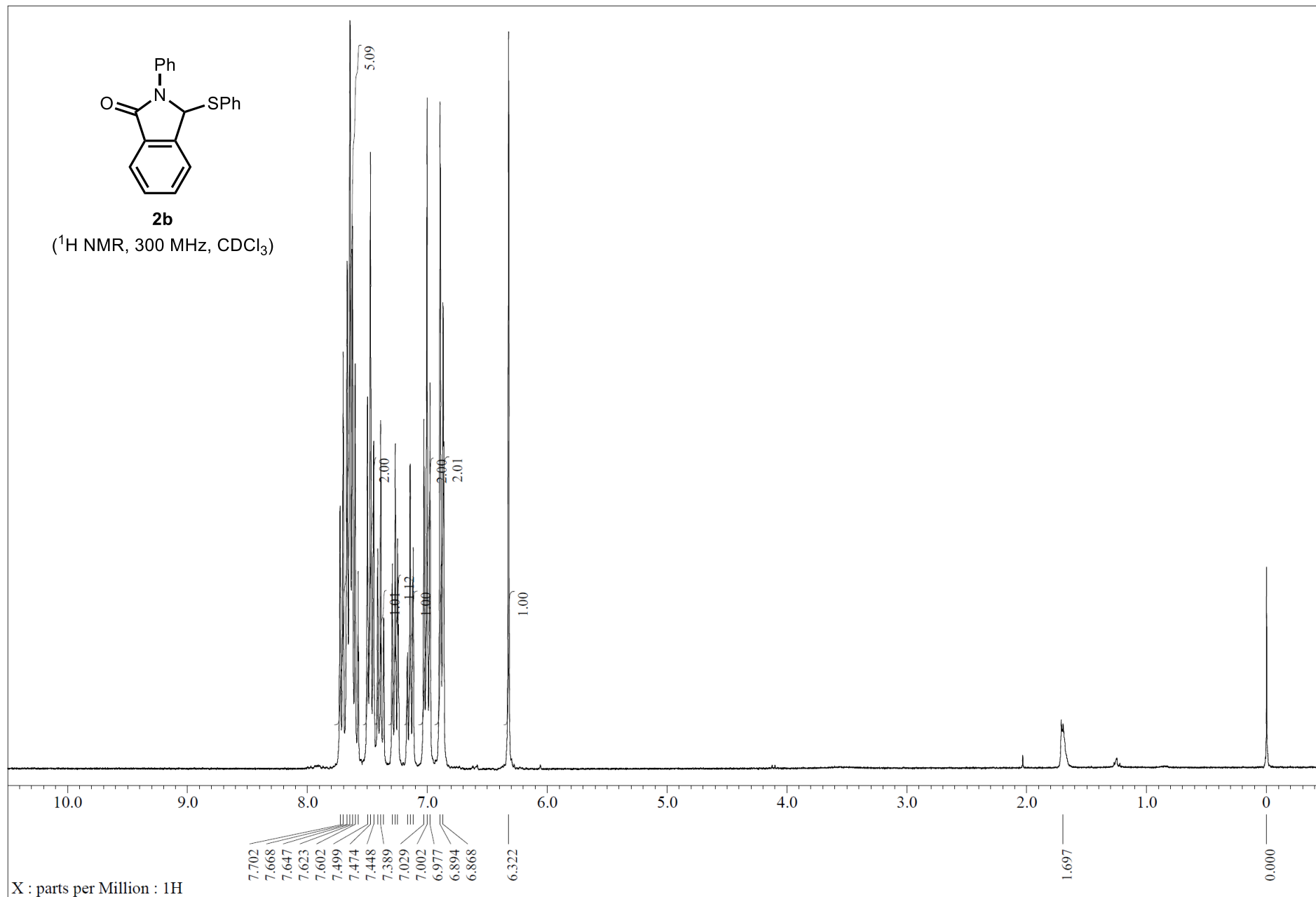


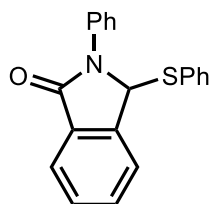
X : parts per Million : ^{13}C



2b

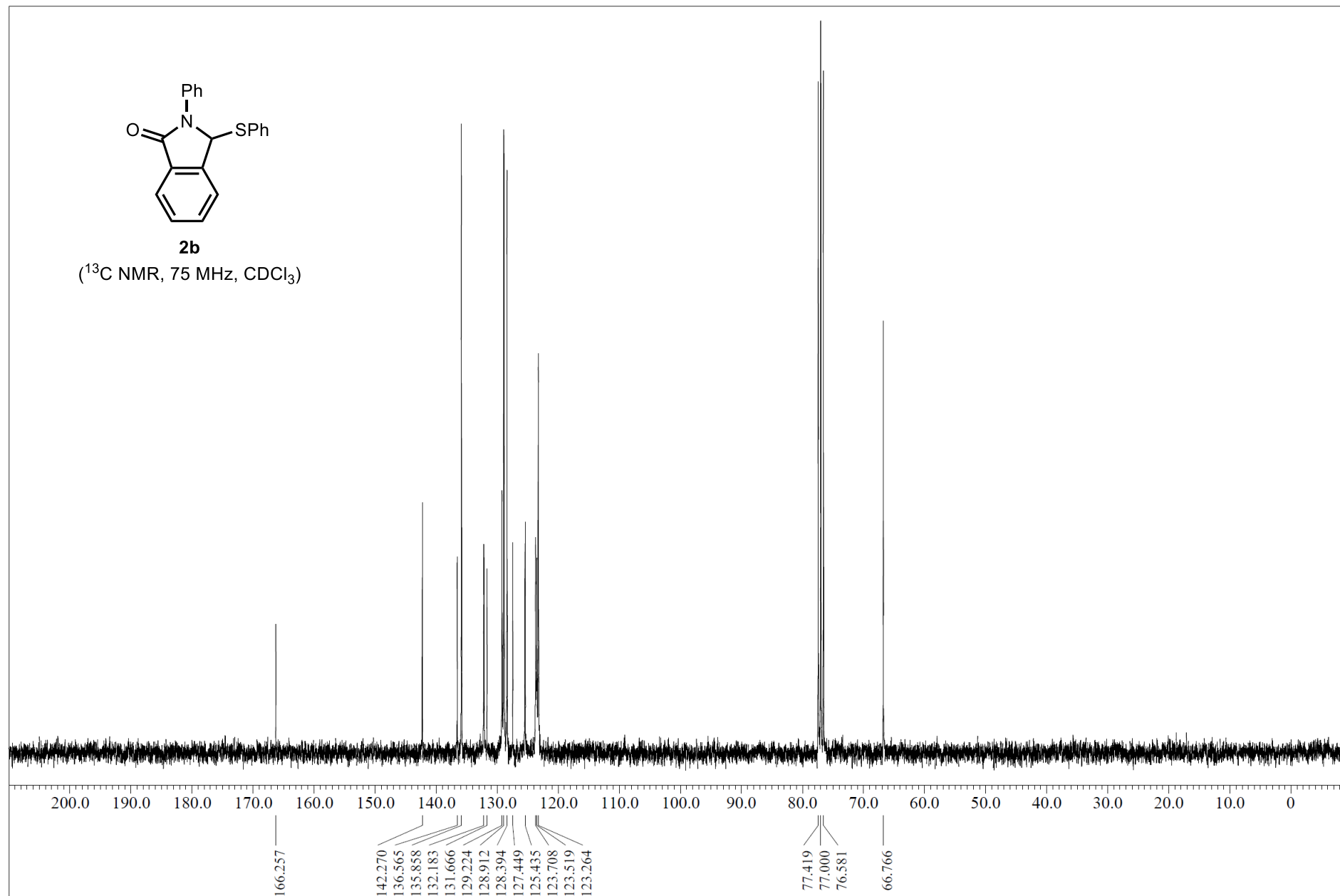
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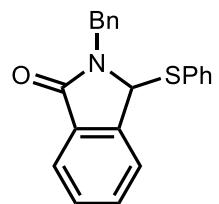


2b

(¹³C NMR, 75 MHz, CDCl₃)

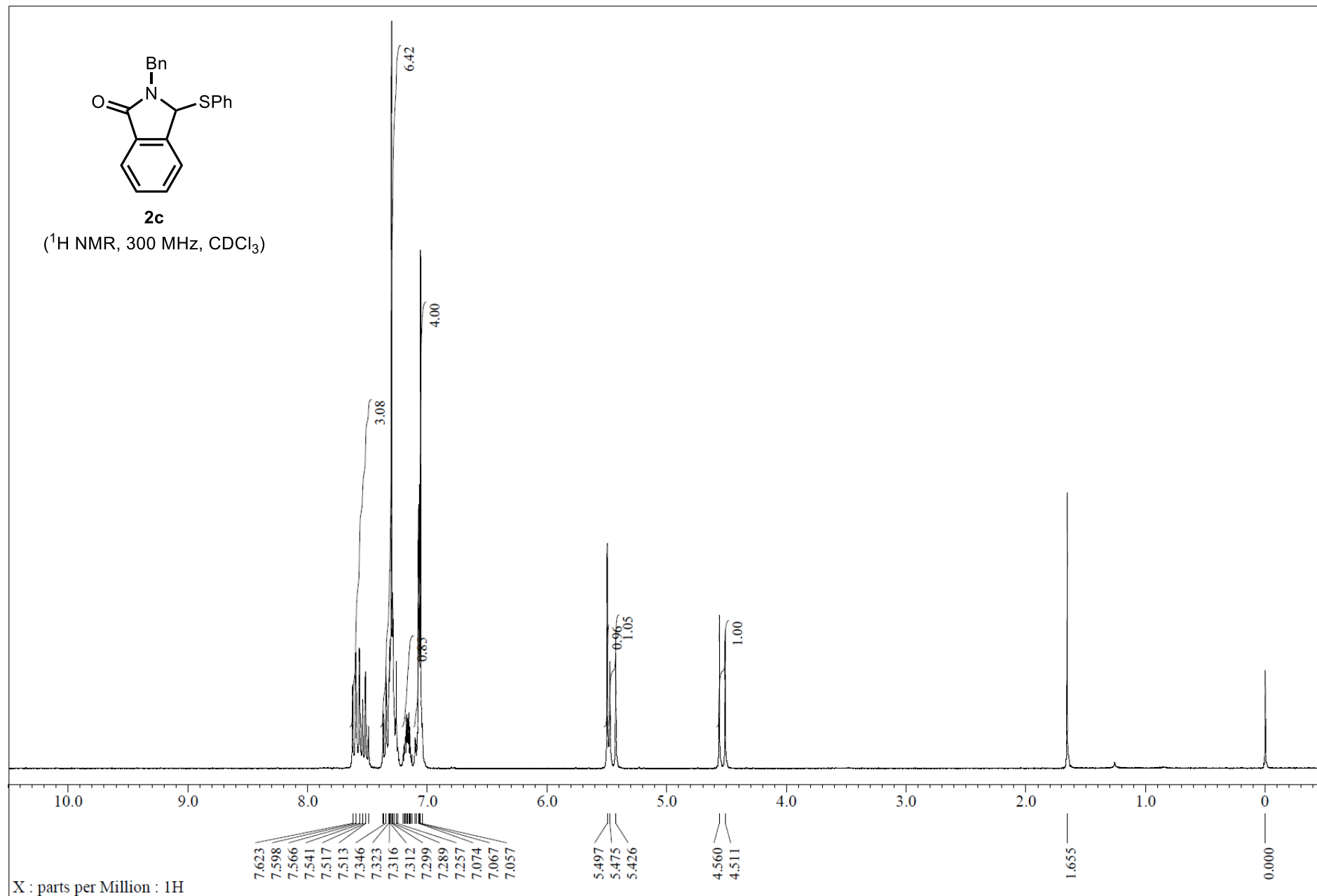


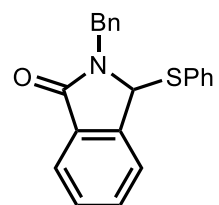
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2c

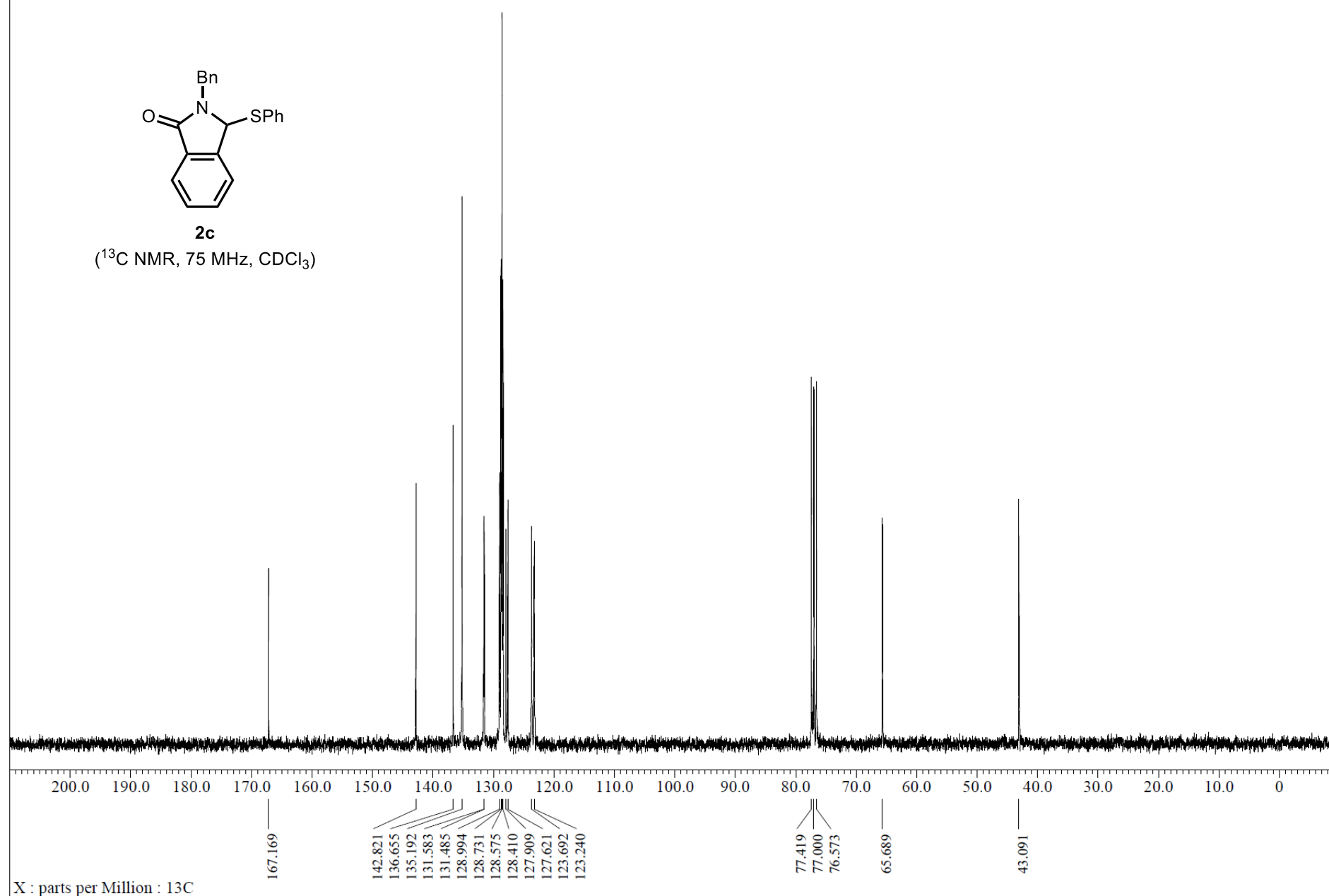
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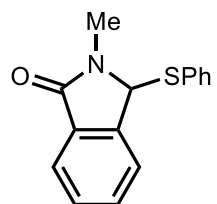




2c

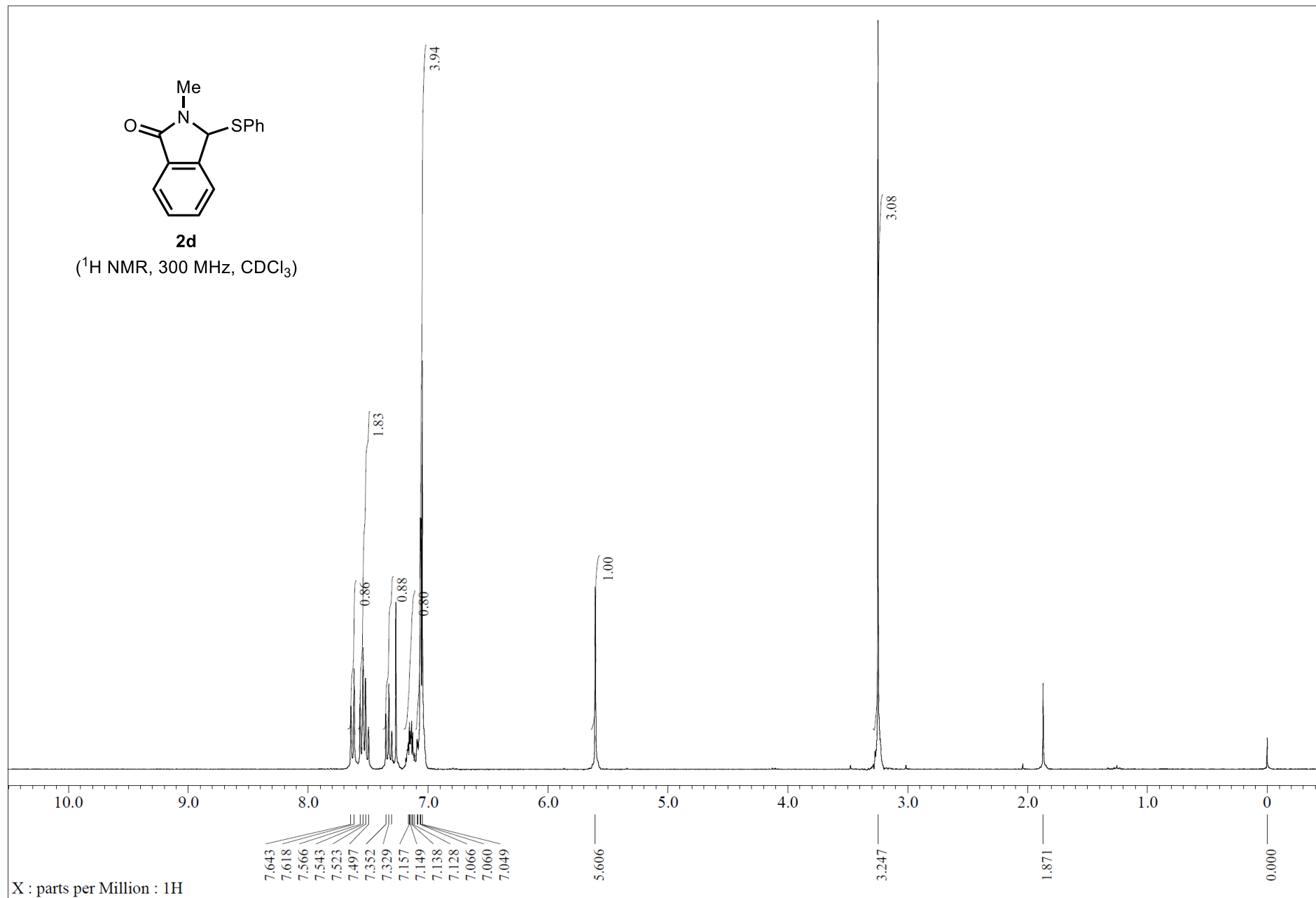
(^{13}C NMR, 75 MHz, CDCl_3)

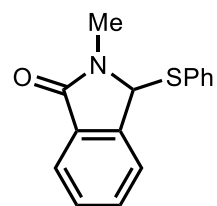




2d

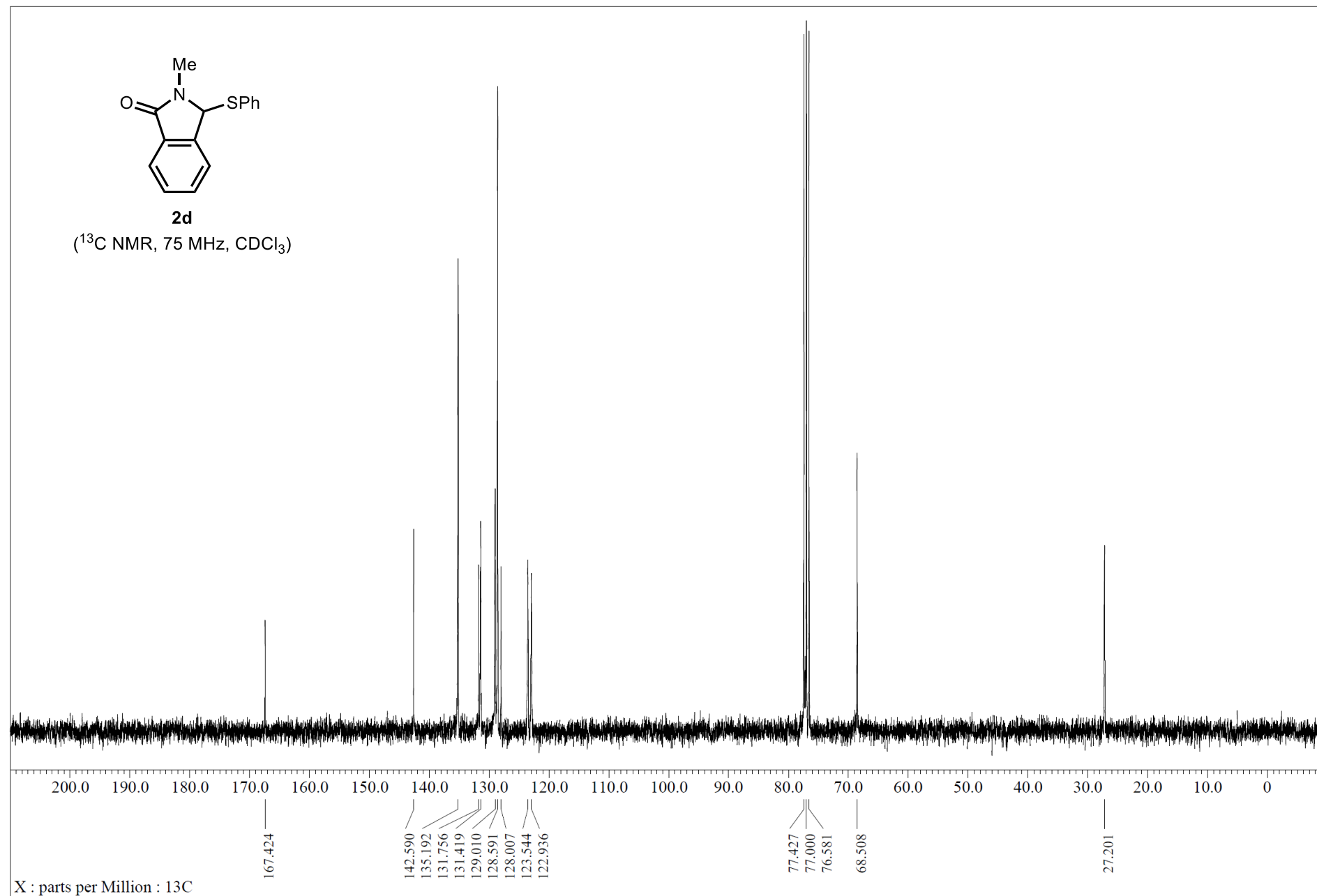
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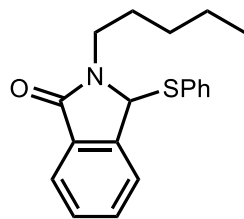




2d

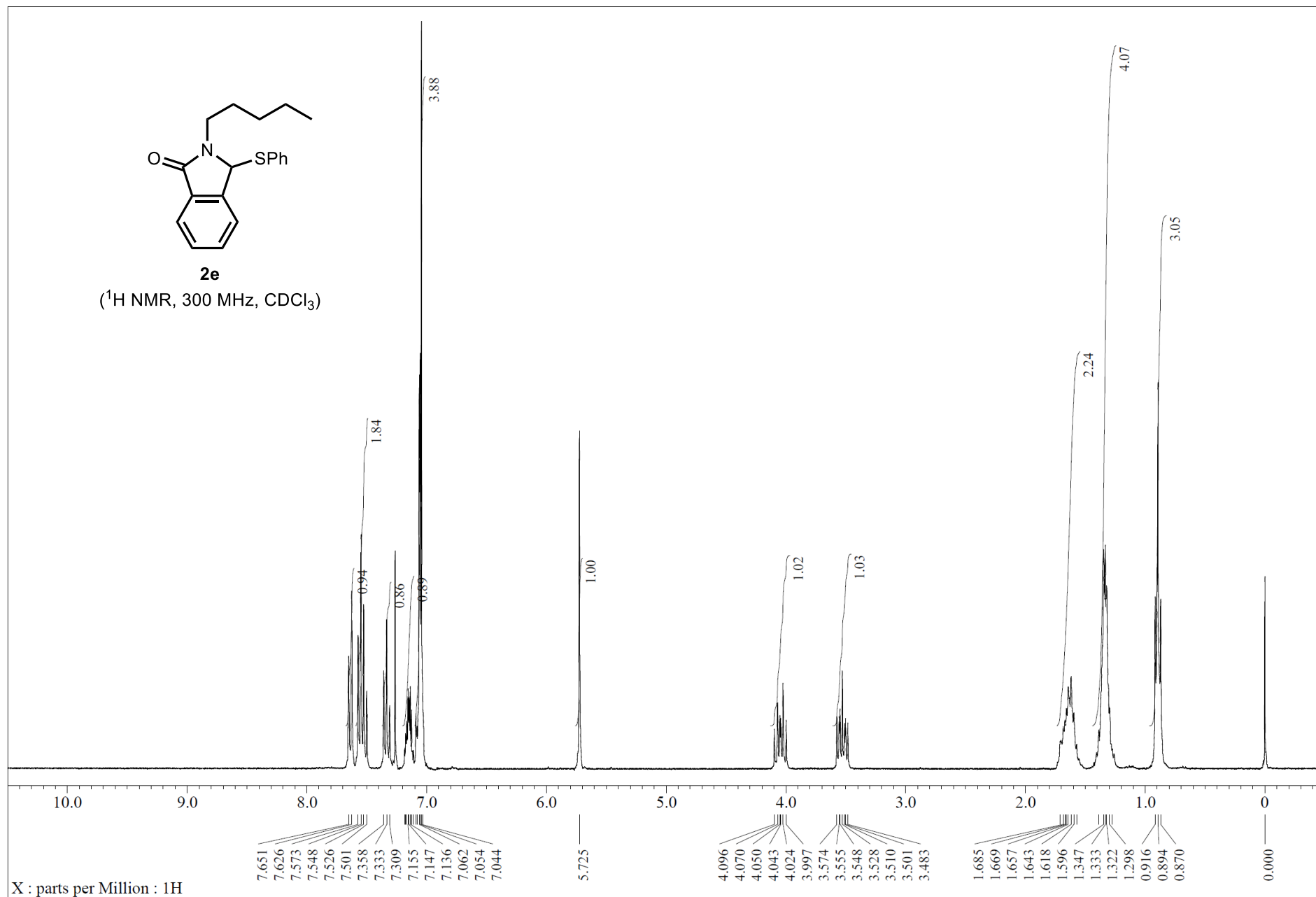
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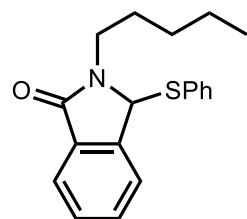




2e

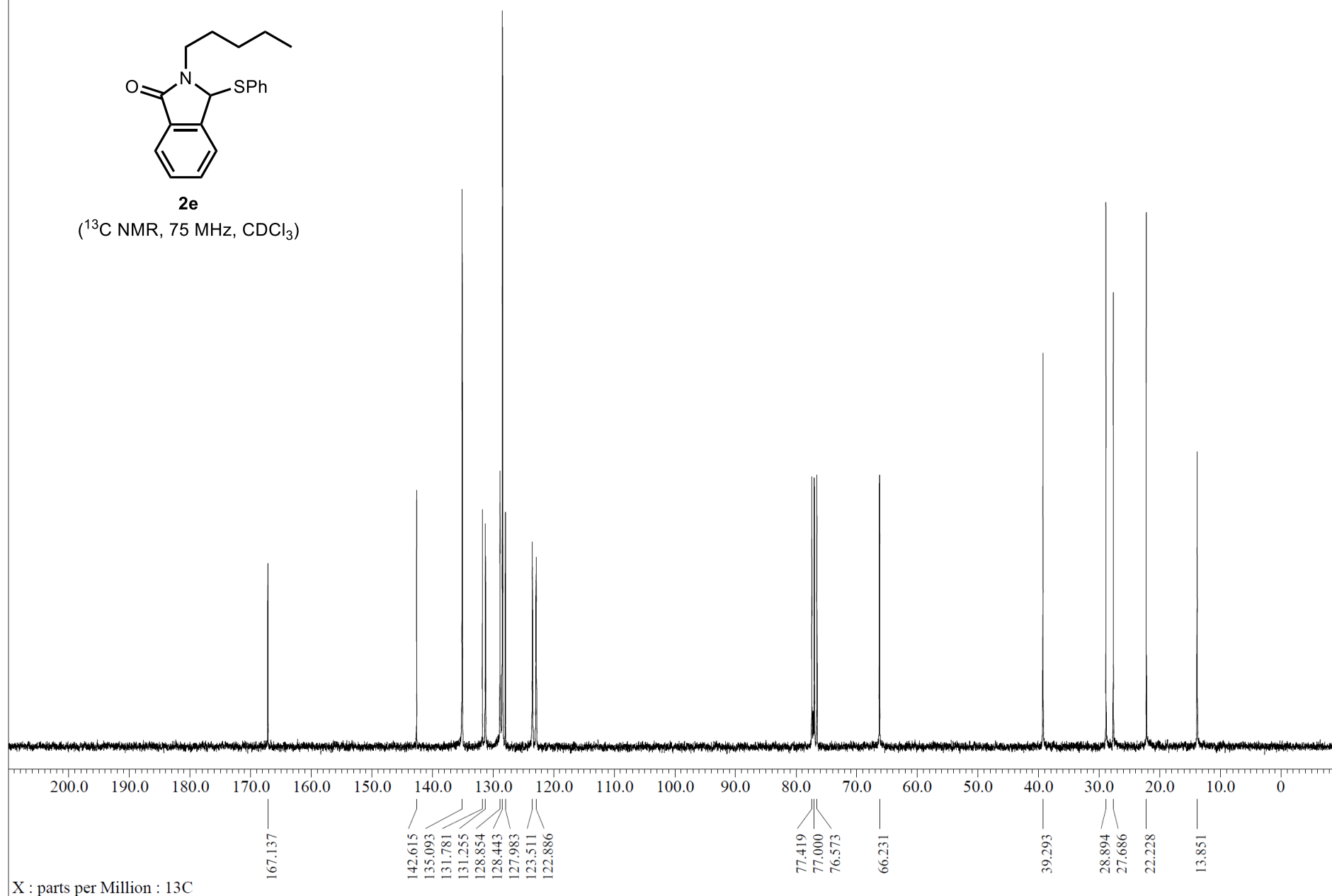
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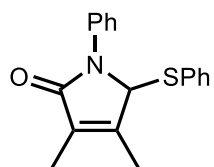


2e

(¹³C NMR, 75 MHz, CDCl₃)

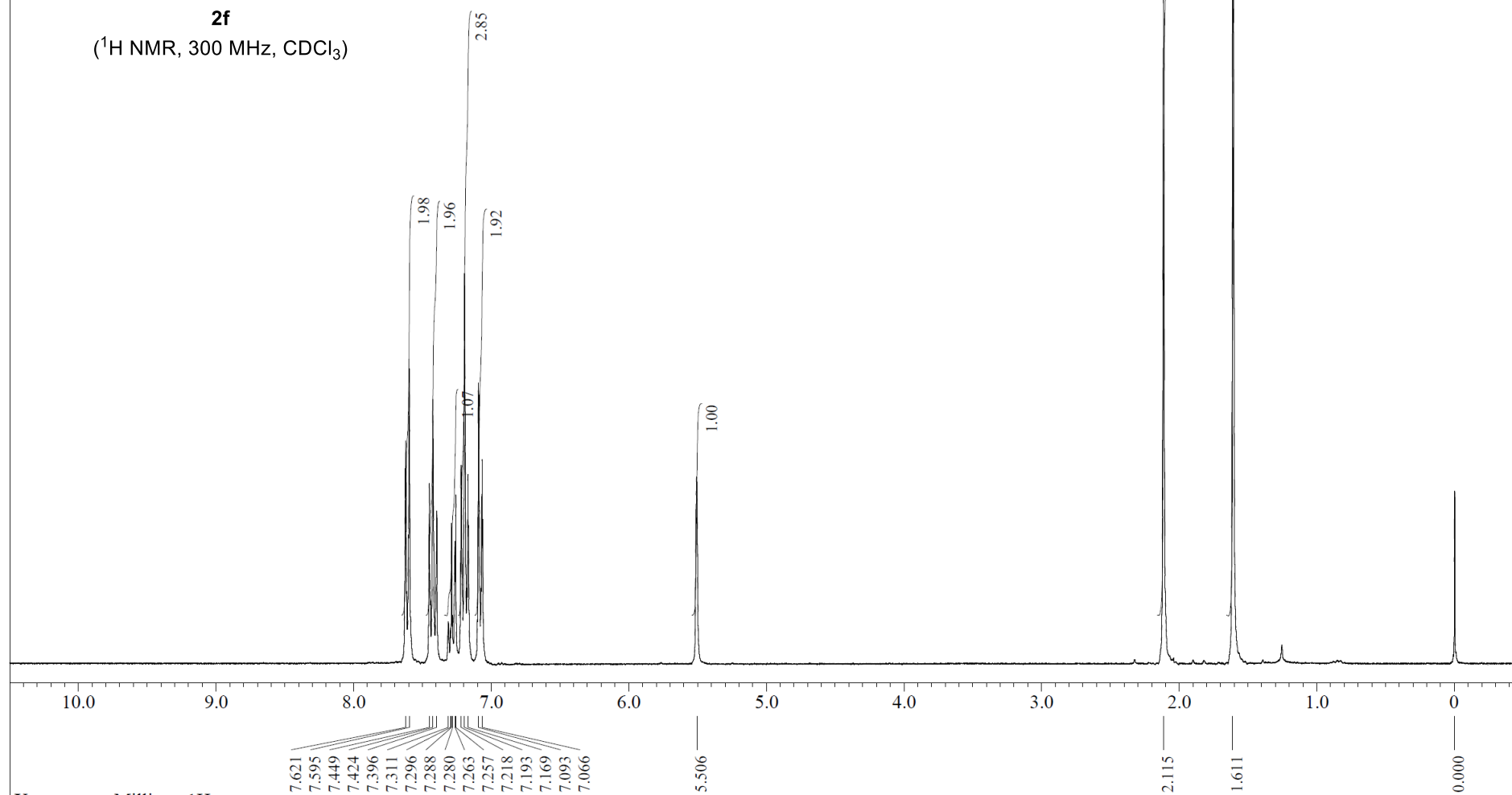


X : parts per Million : ¹³C

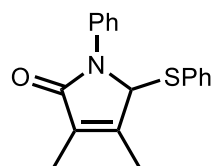


2f

(¹H NMR, 300 MHz, CDCl₃)

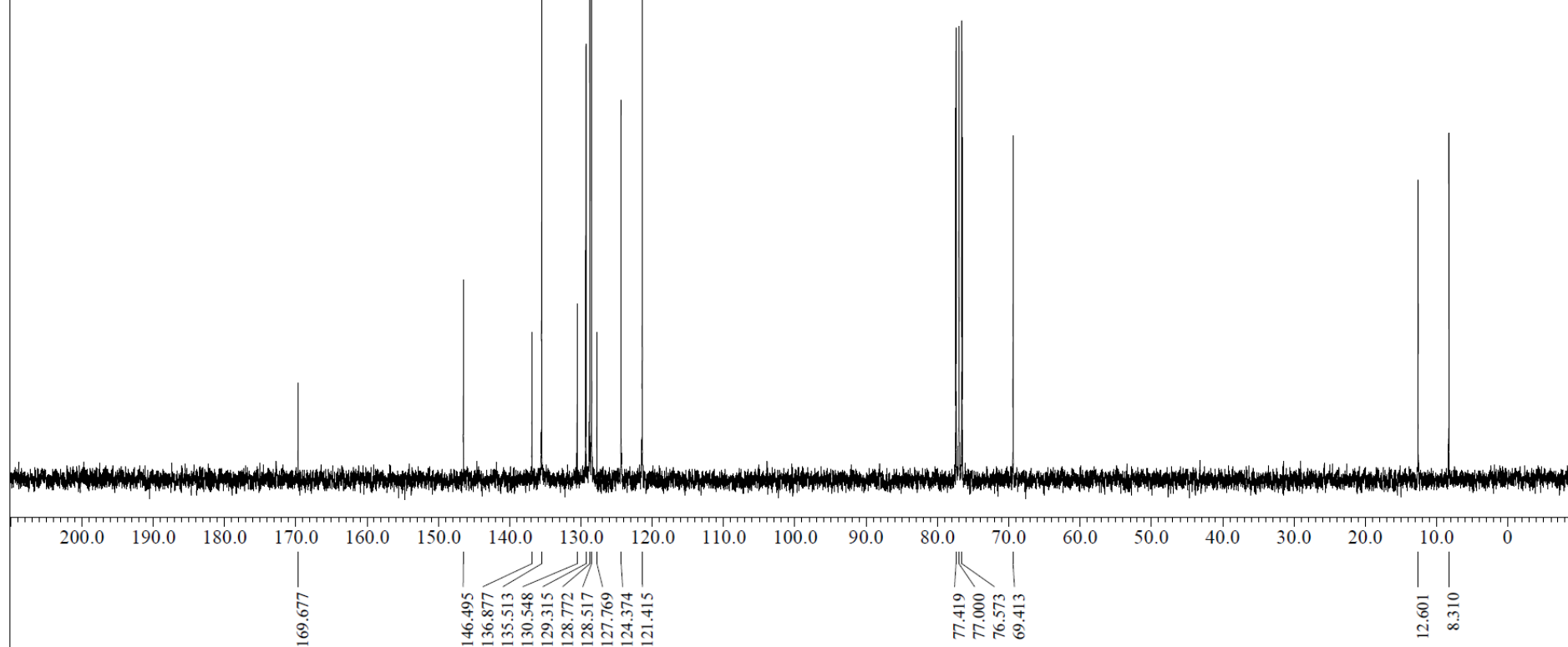


X : parts per Million : 1H

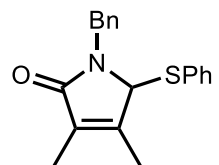


2f

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

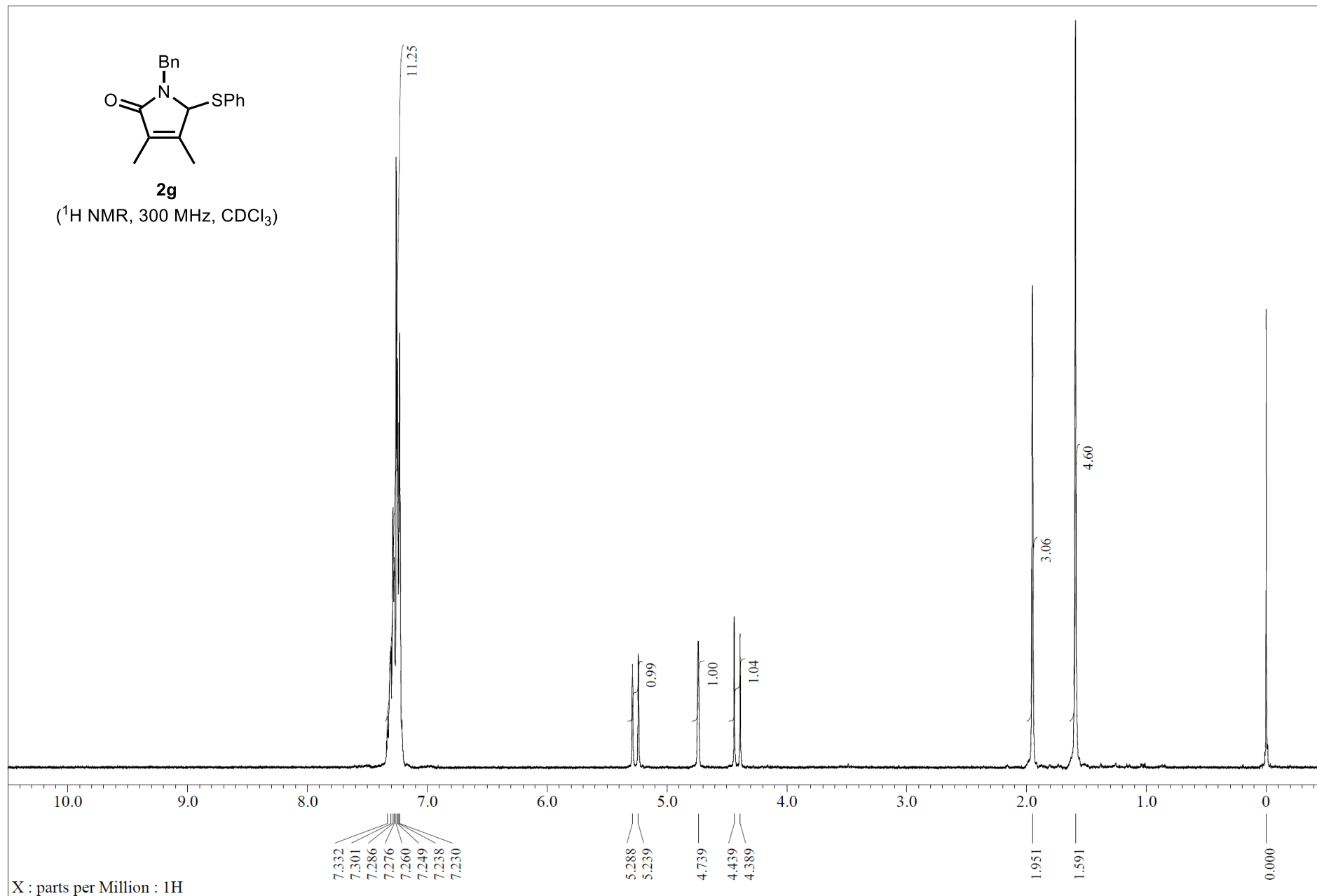


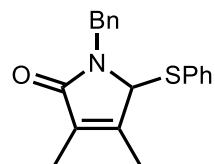
X : parts per Million : ^{13}C



2g

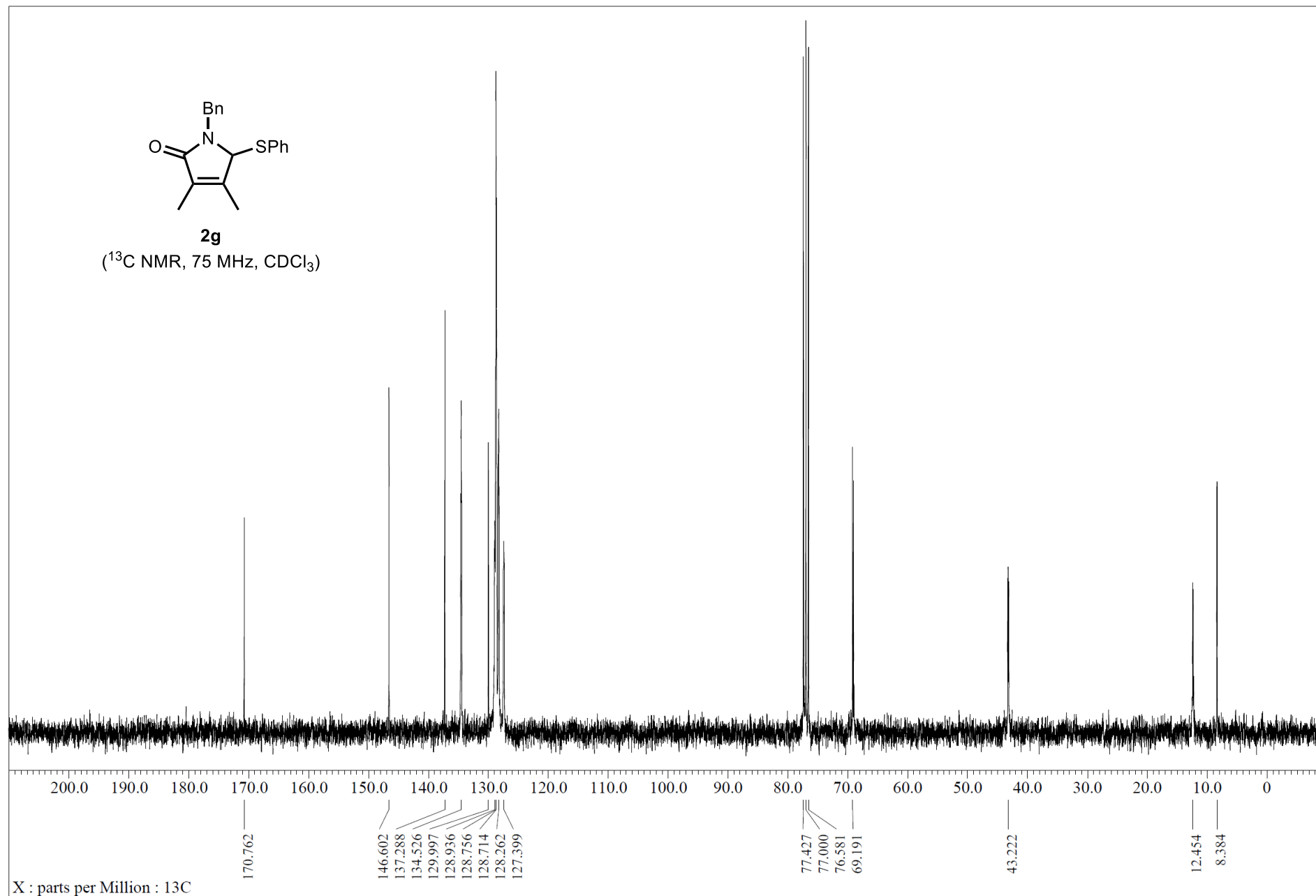
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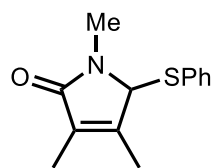




2g

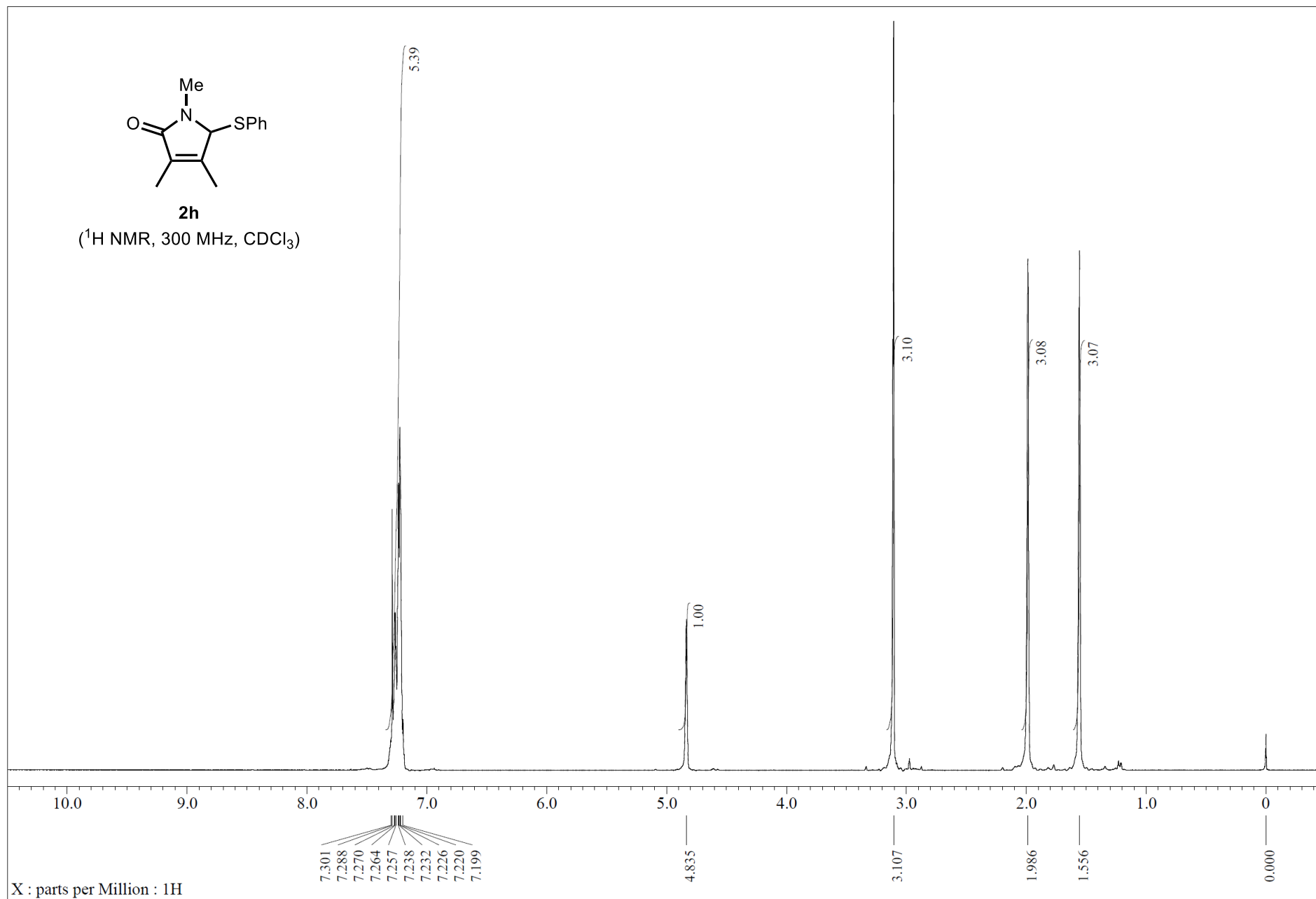
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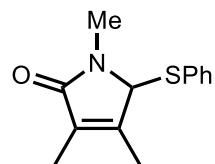




2h

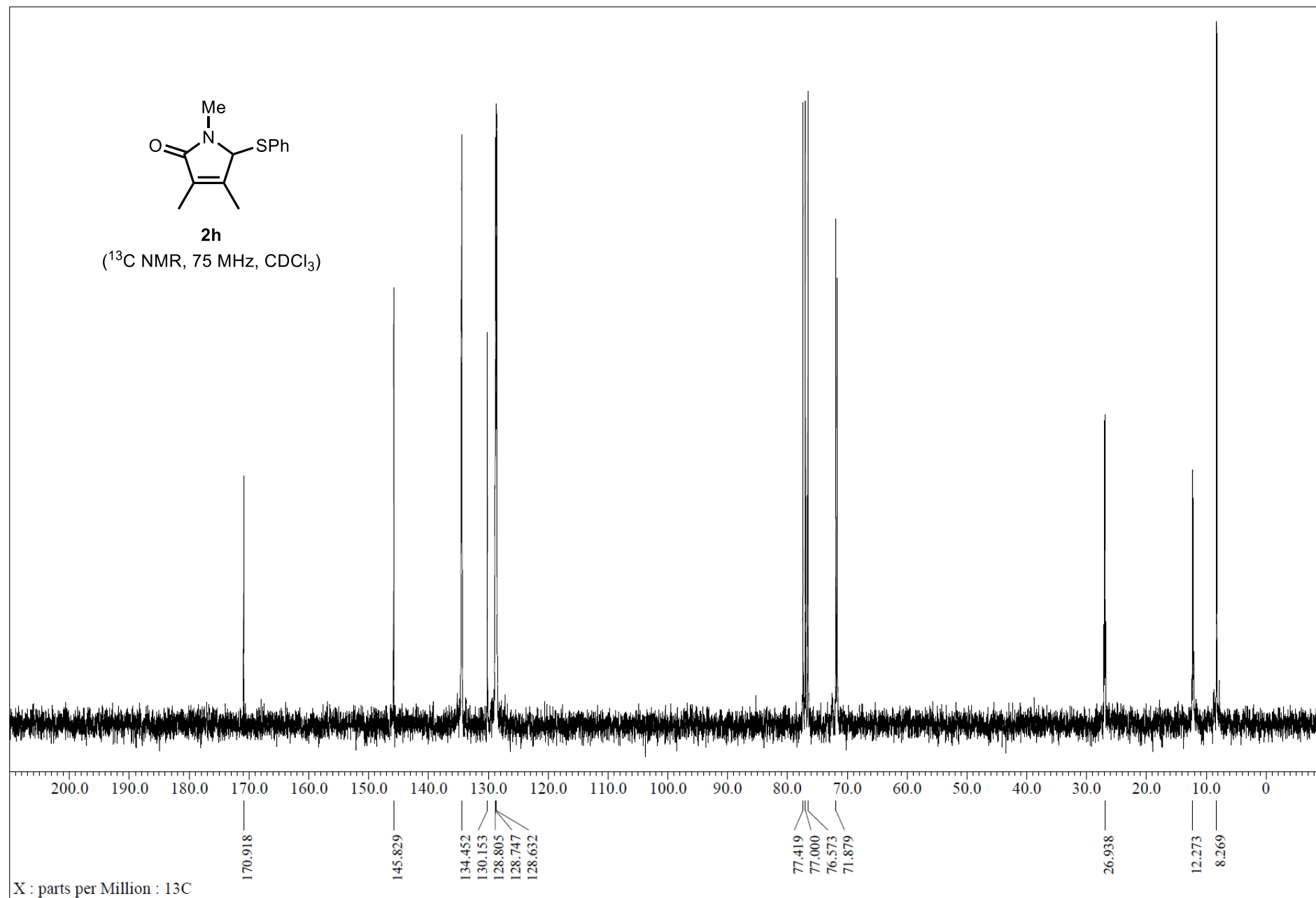
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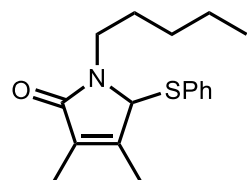




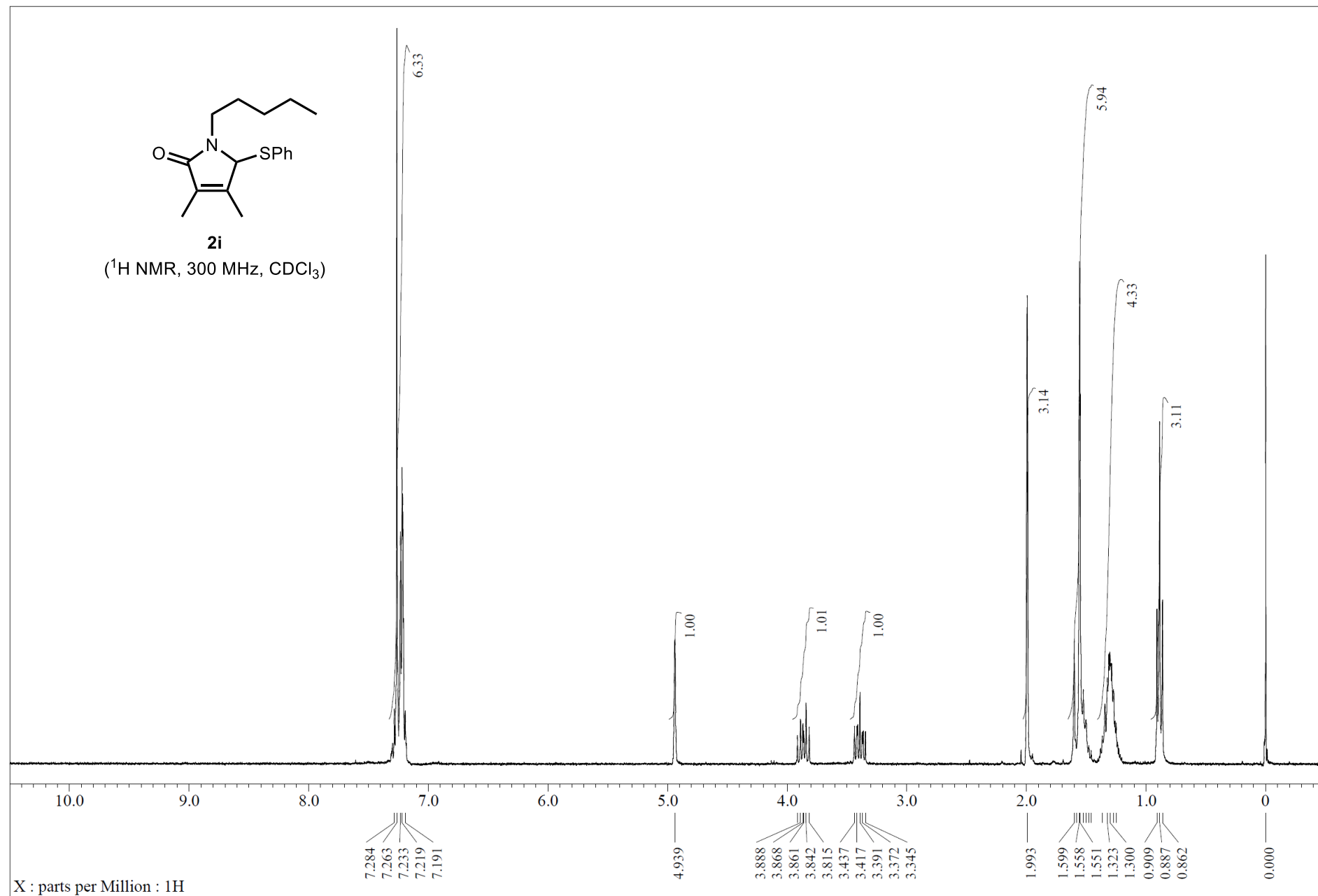
2h

(¹³C NMR, 75 MHz, CDCl₃)

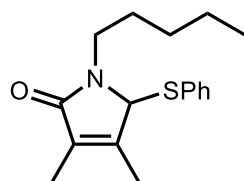




2i
 (^1H NMR, 300 MHz, CDCl_3)

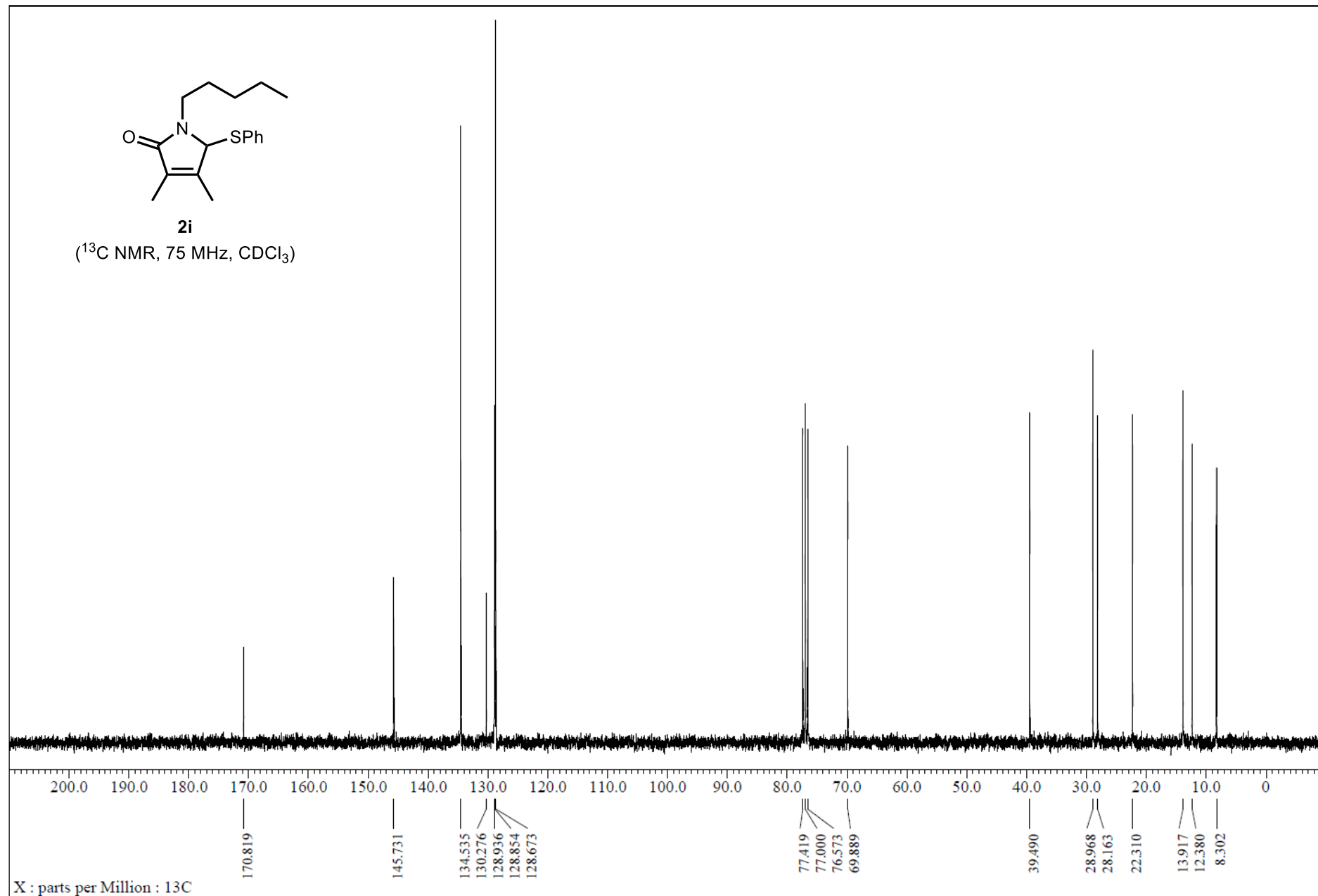


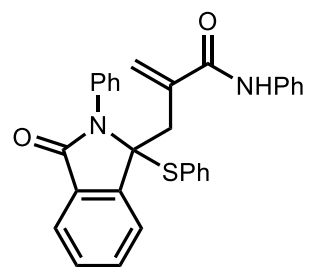
X : parts per Million : ^1H



2i

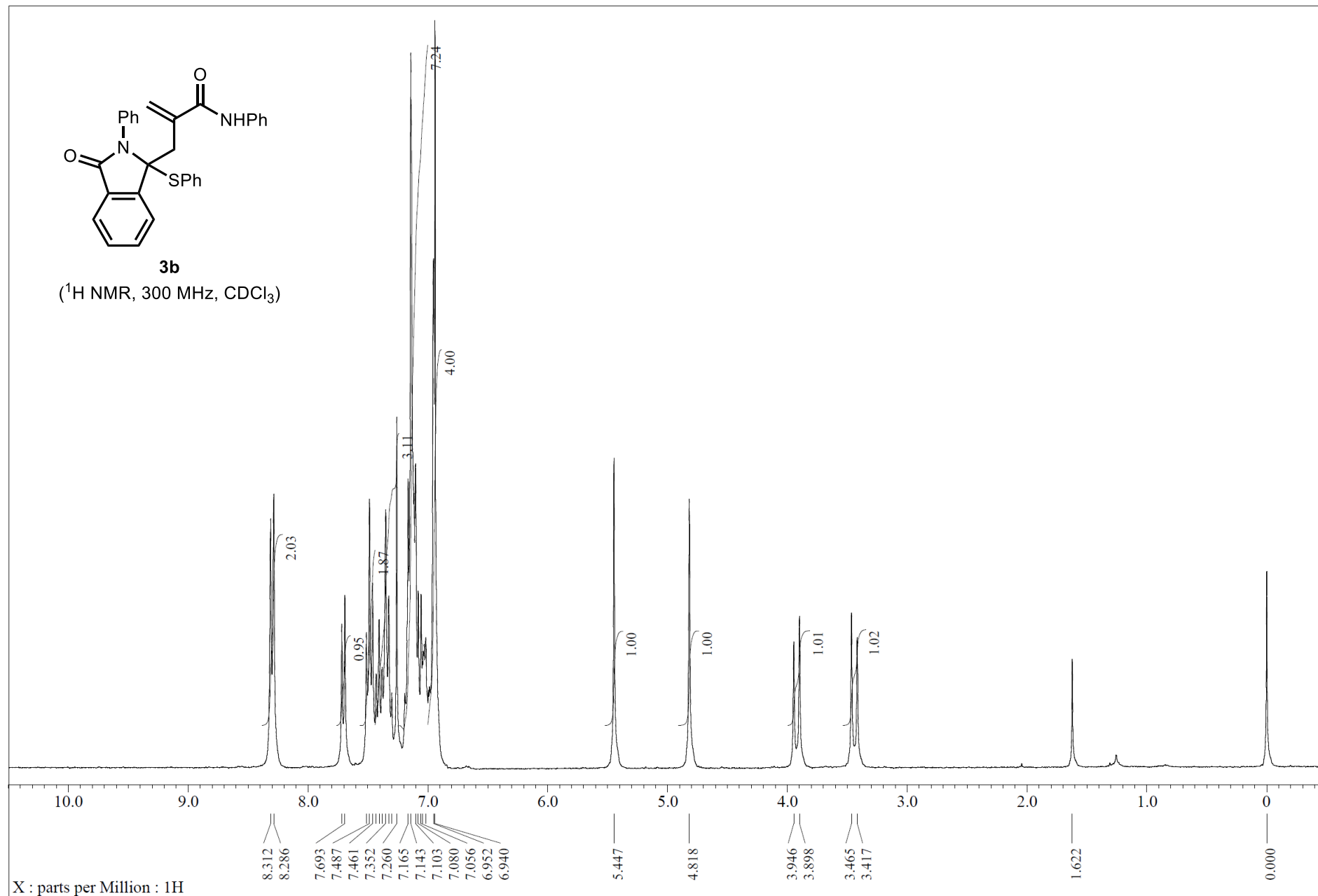
(^{13}C NMR, 75 MHz, CDCl_3)



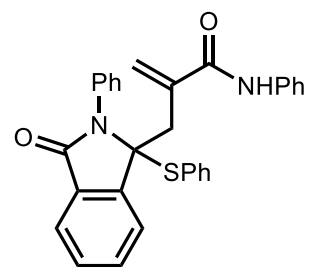


3b

(¹H NMR, 300 MHz, CDCl₃)

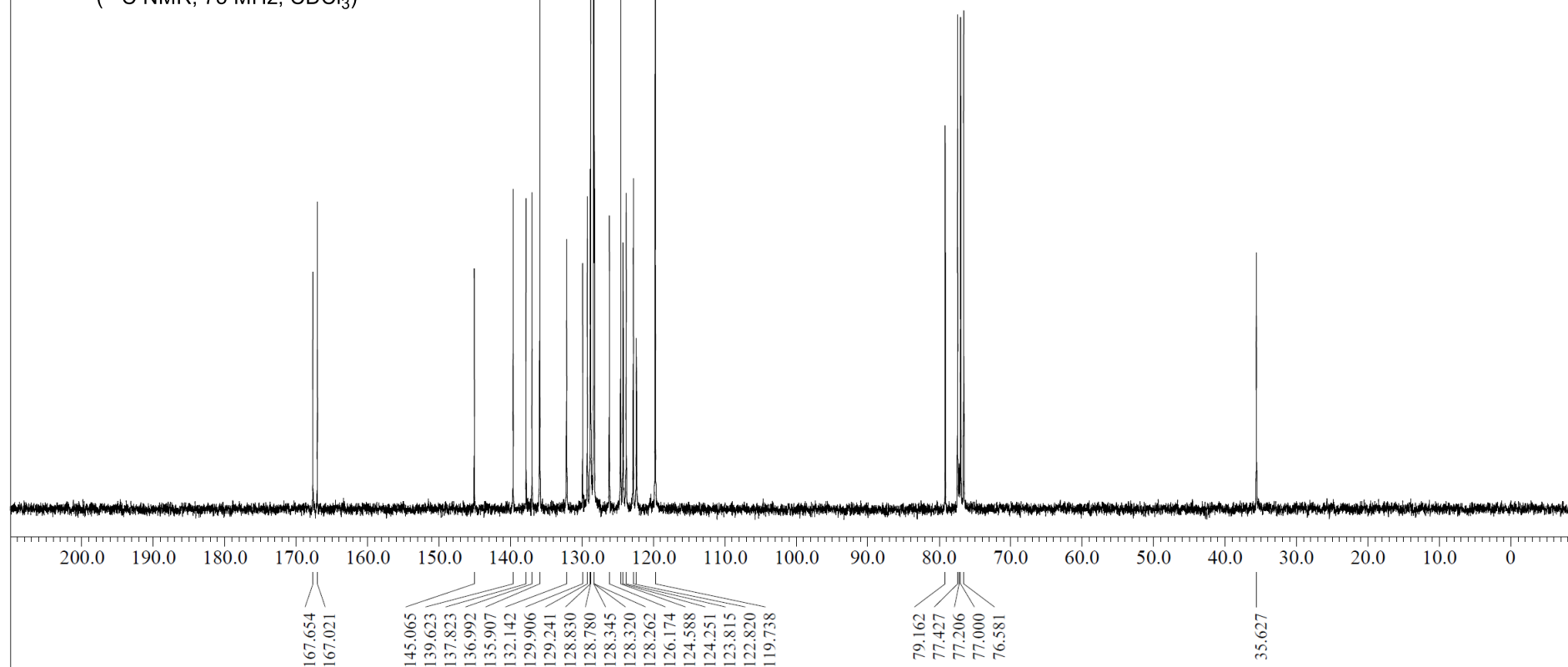


X : parts per Million : 1H

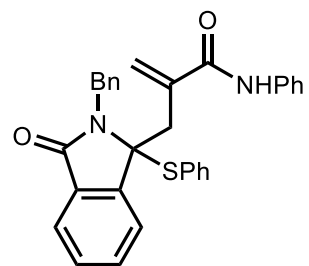


3b

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

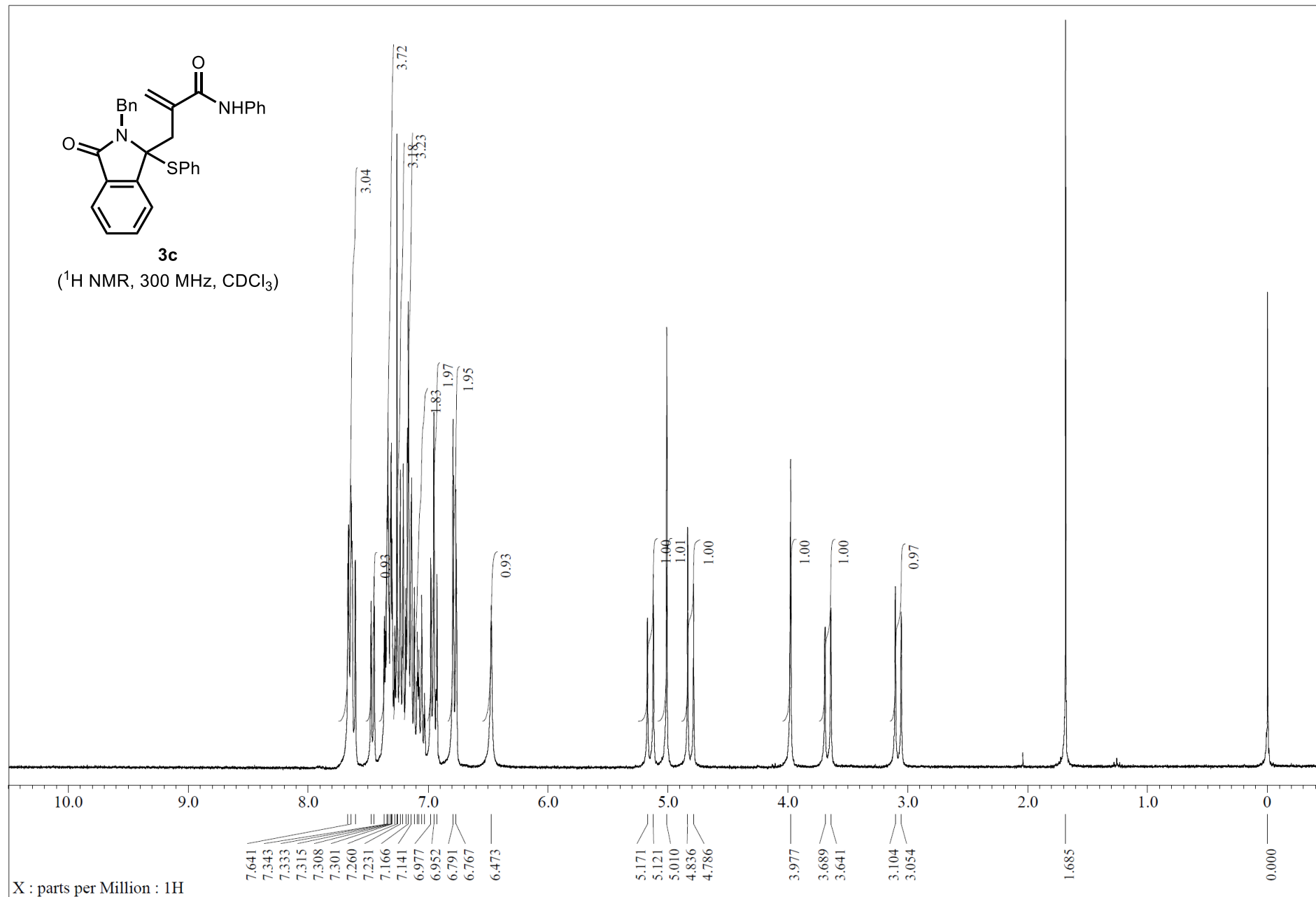


X : parts per Million : ^{13}C

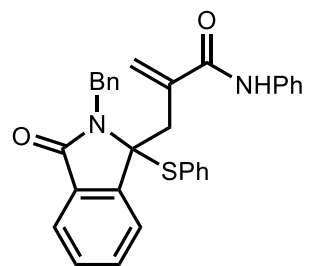


3c

(¹H NMR, 300 MHz, CDCl₃)

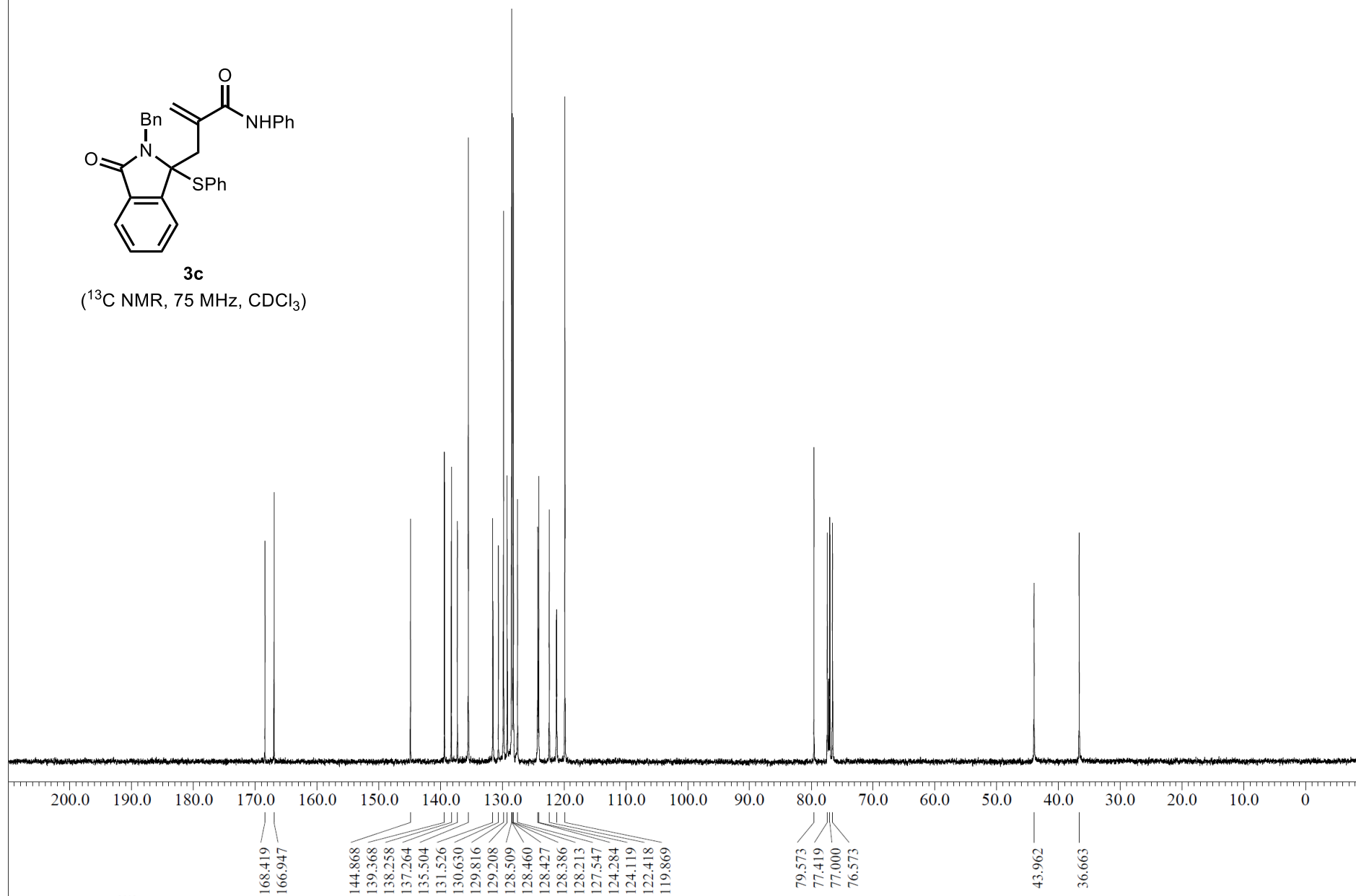


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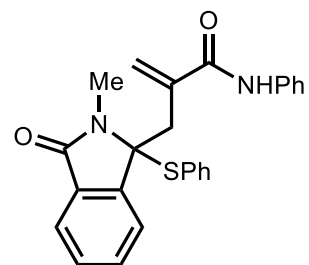


3c

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

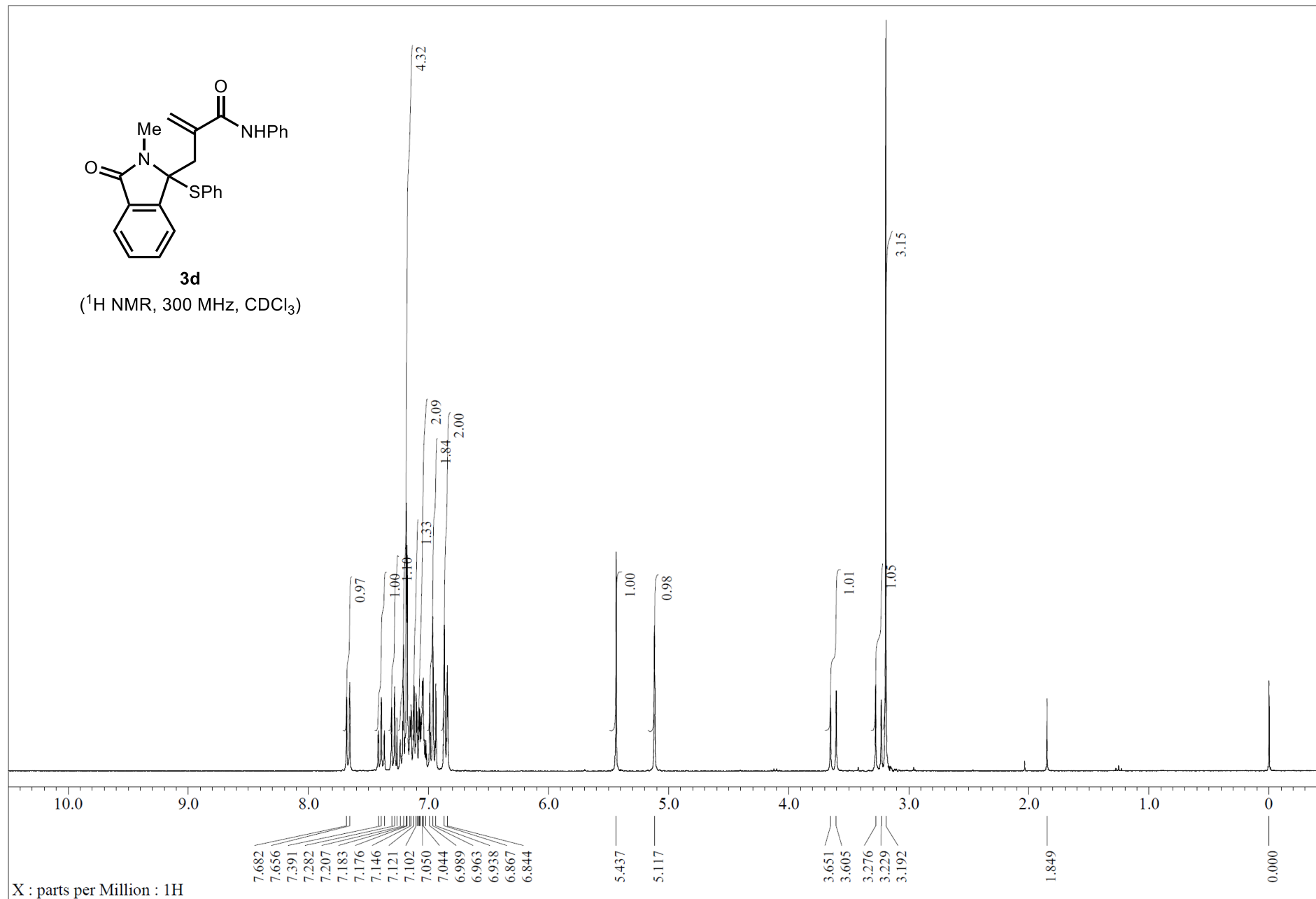


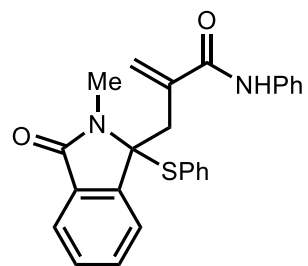
X : parts per Million : ^{13}C



3d

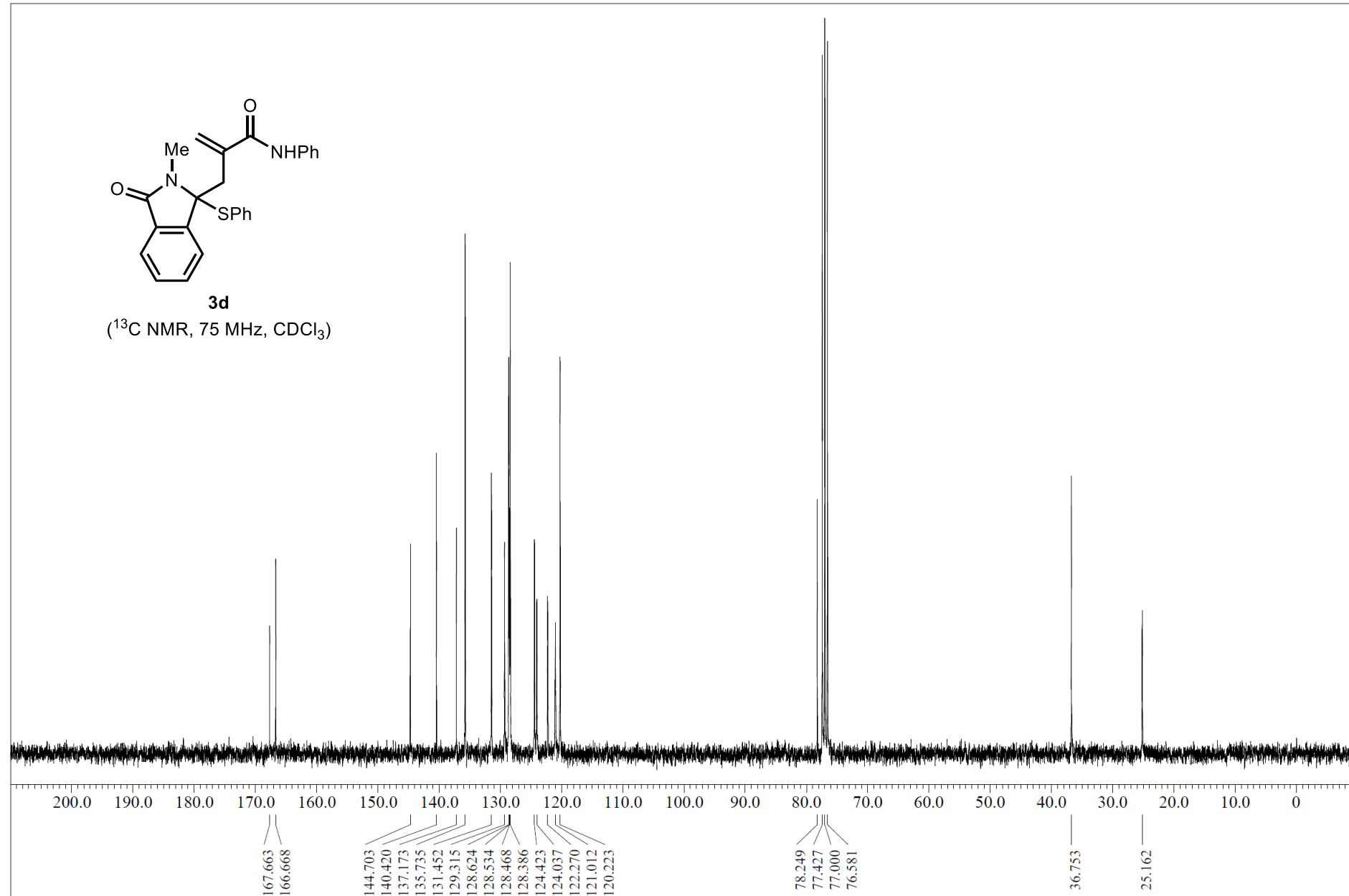
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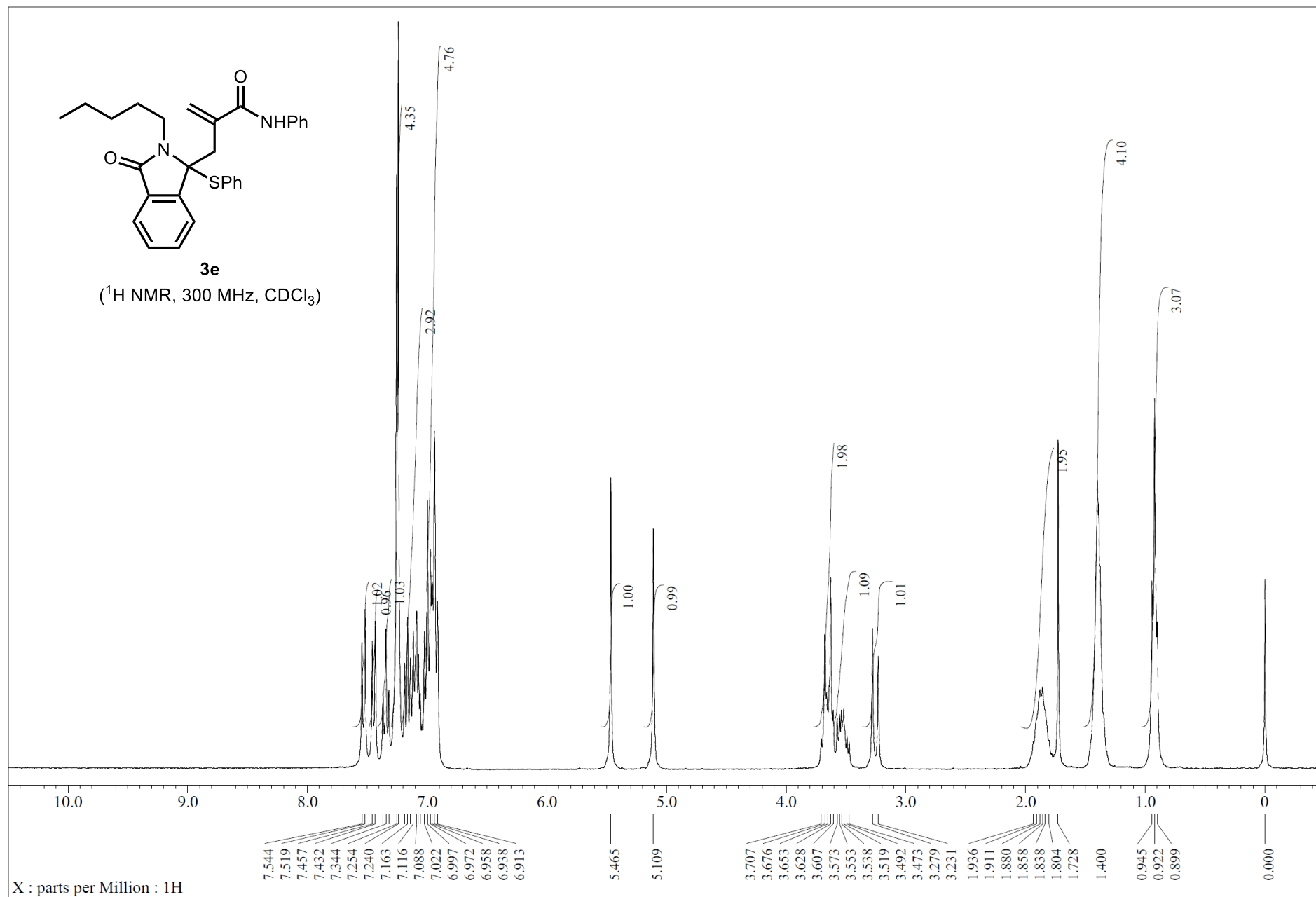


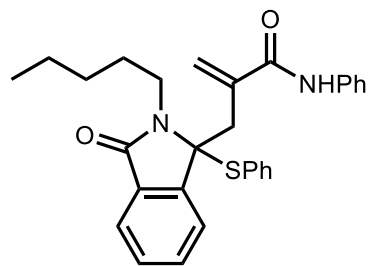
3d

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



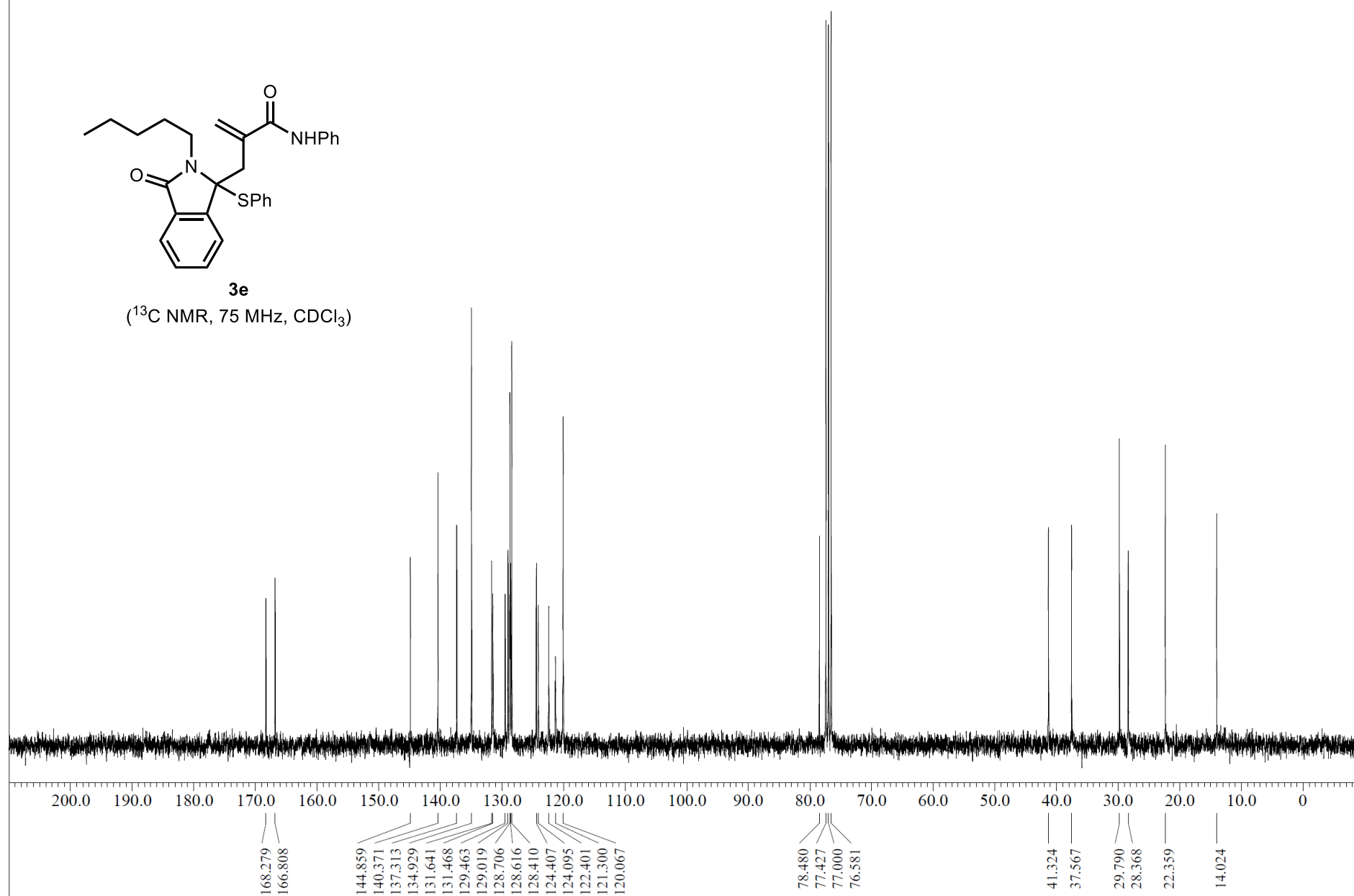
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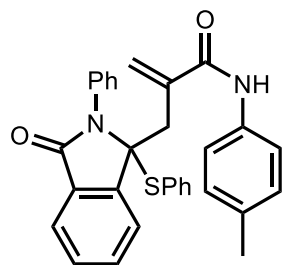


3e

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

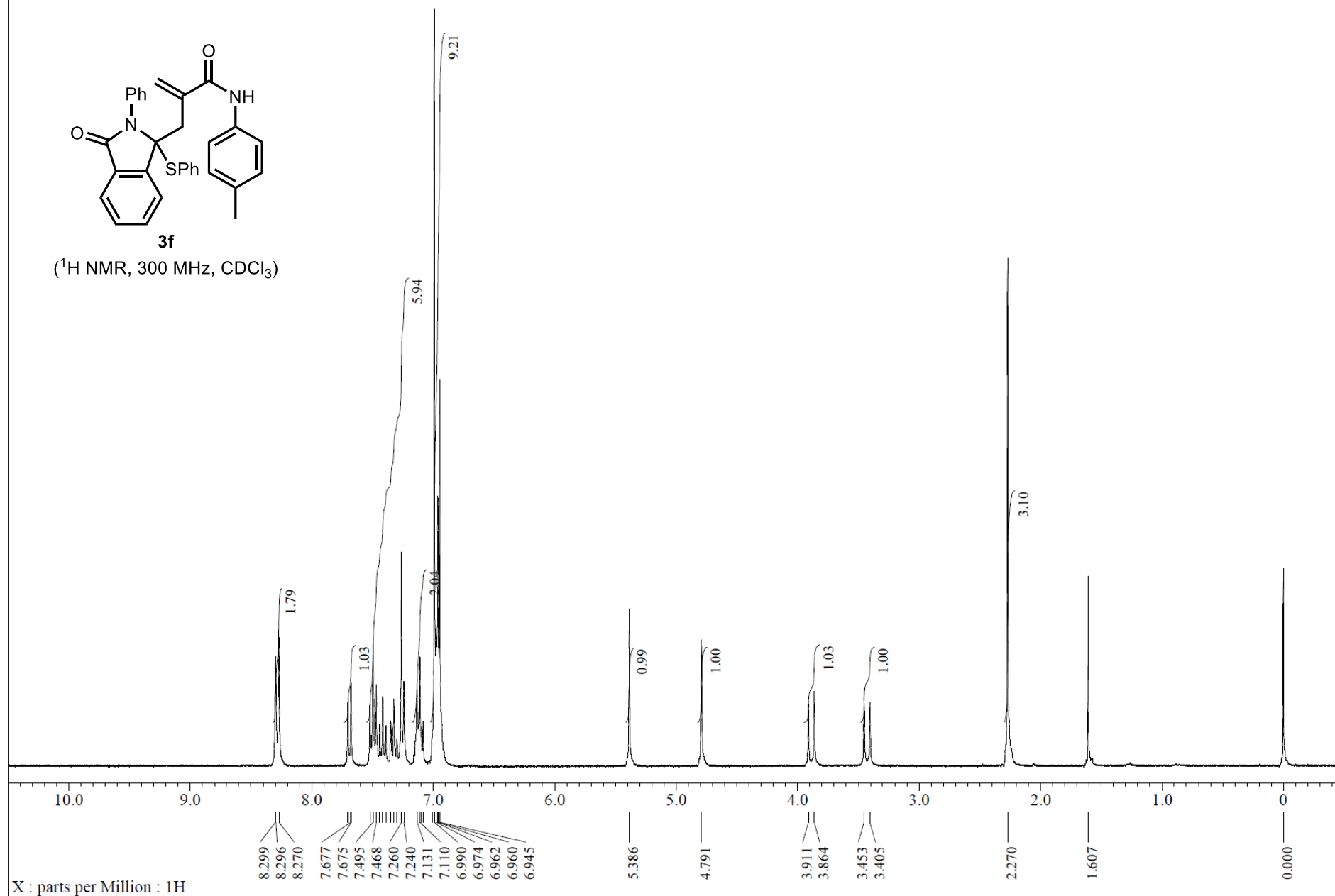


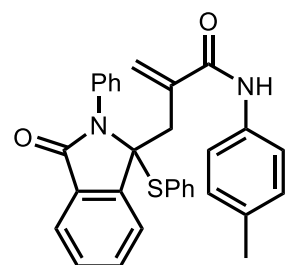
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3f

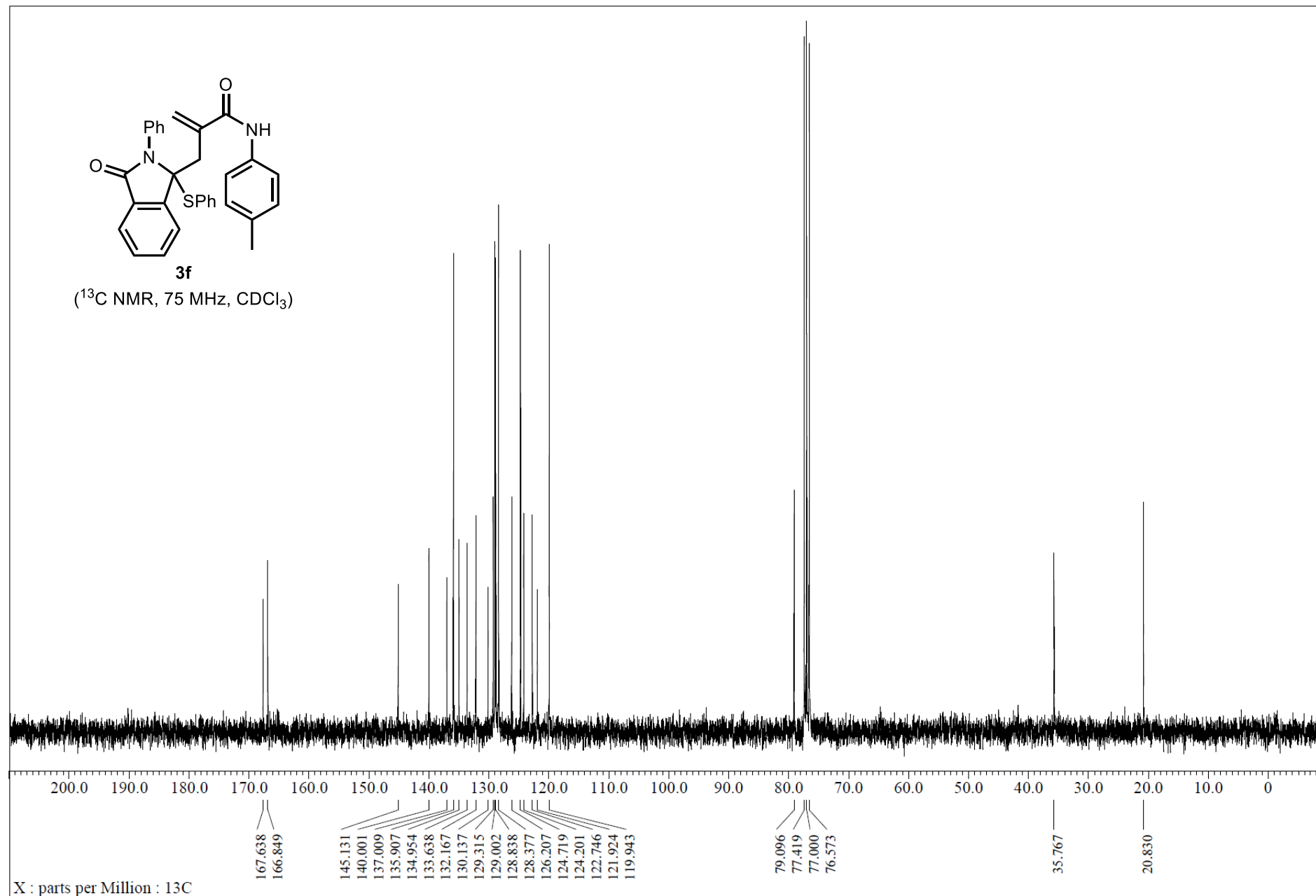
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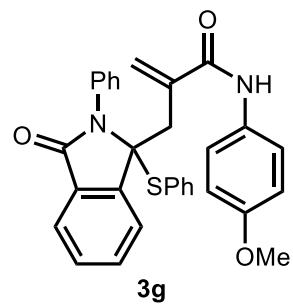


3f

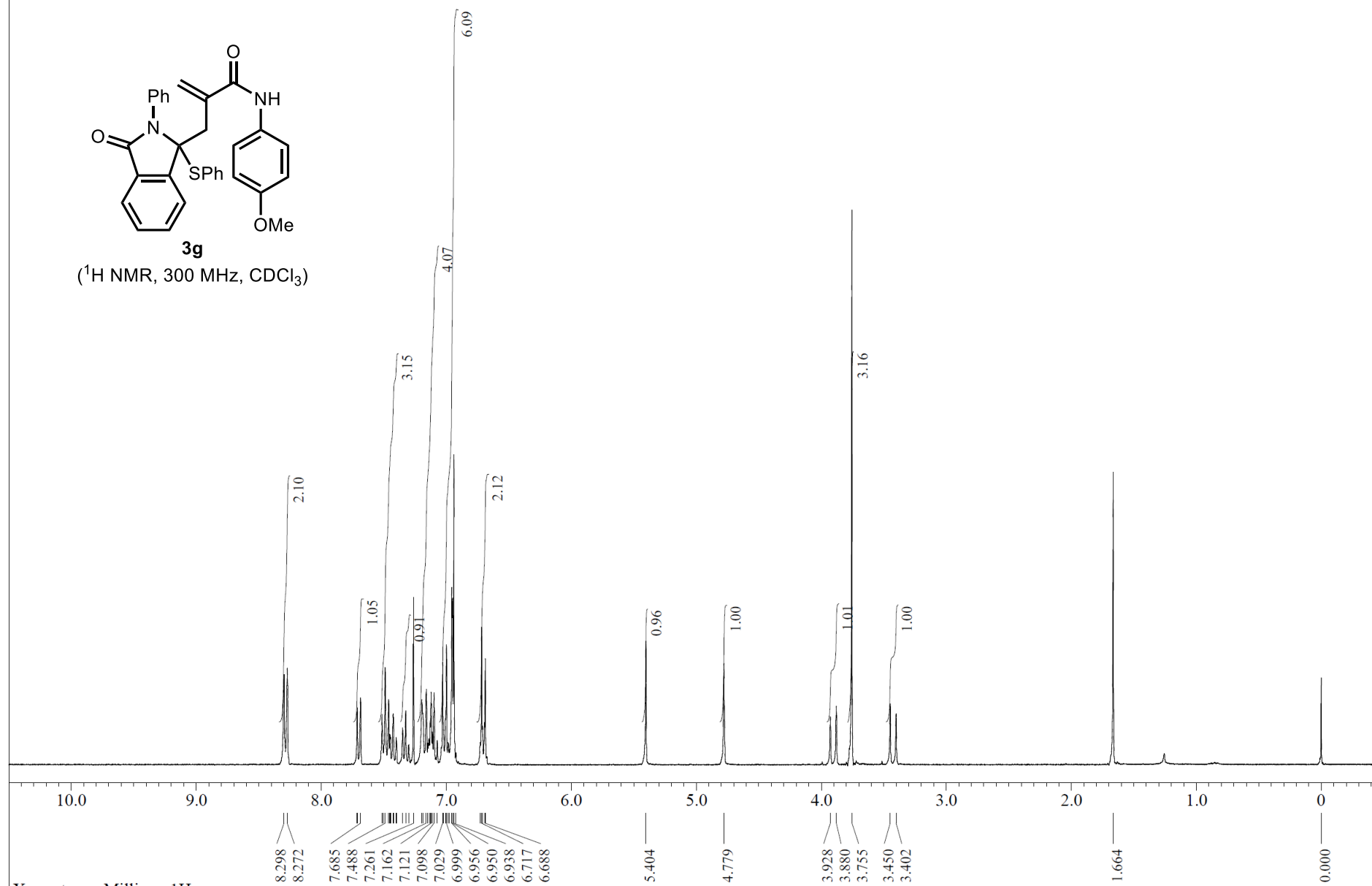
(^{13}C NMR, 75 MHz, CDCl_3)



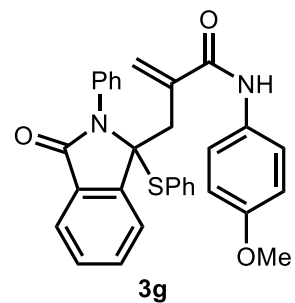
X : parts per Million : ^{13}C



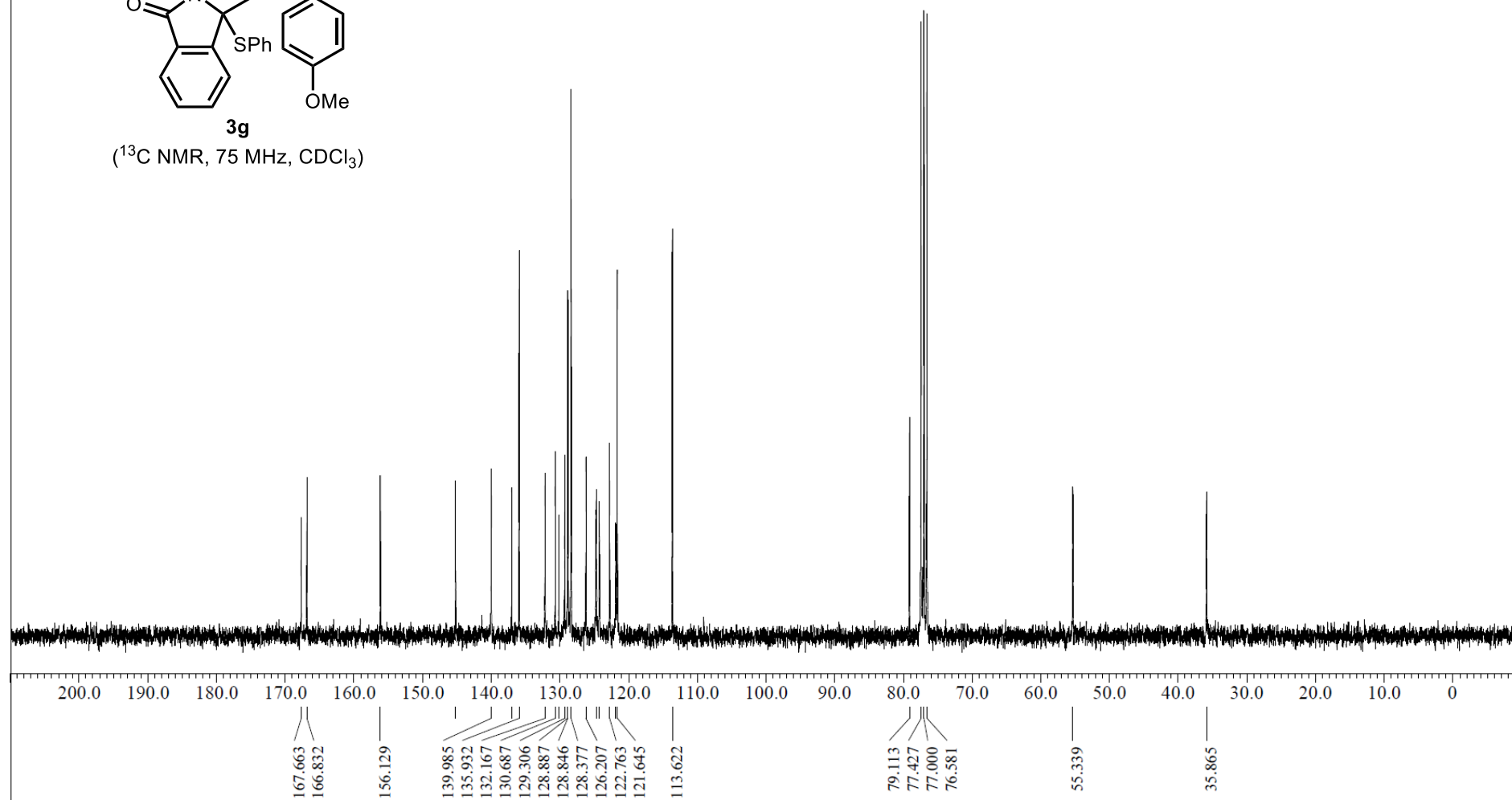
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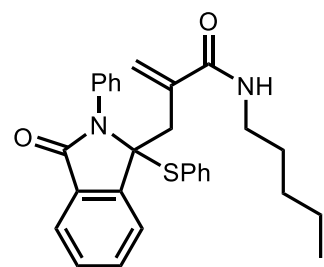
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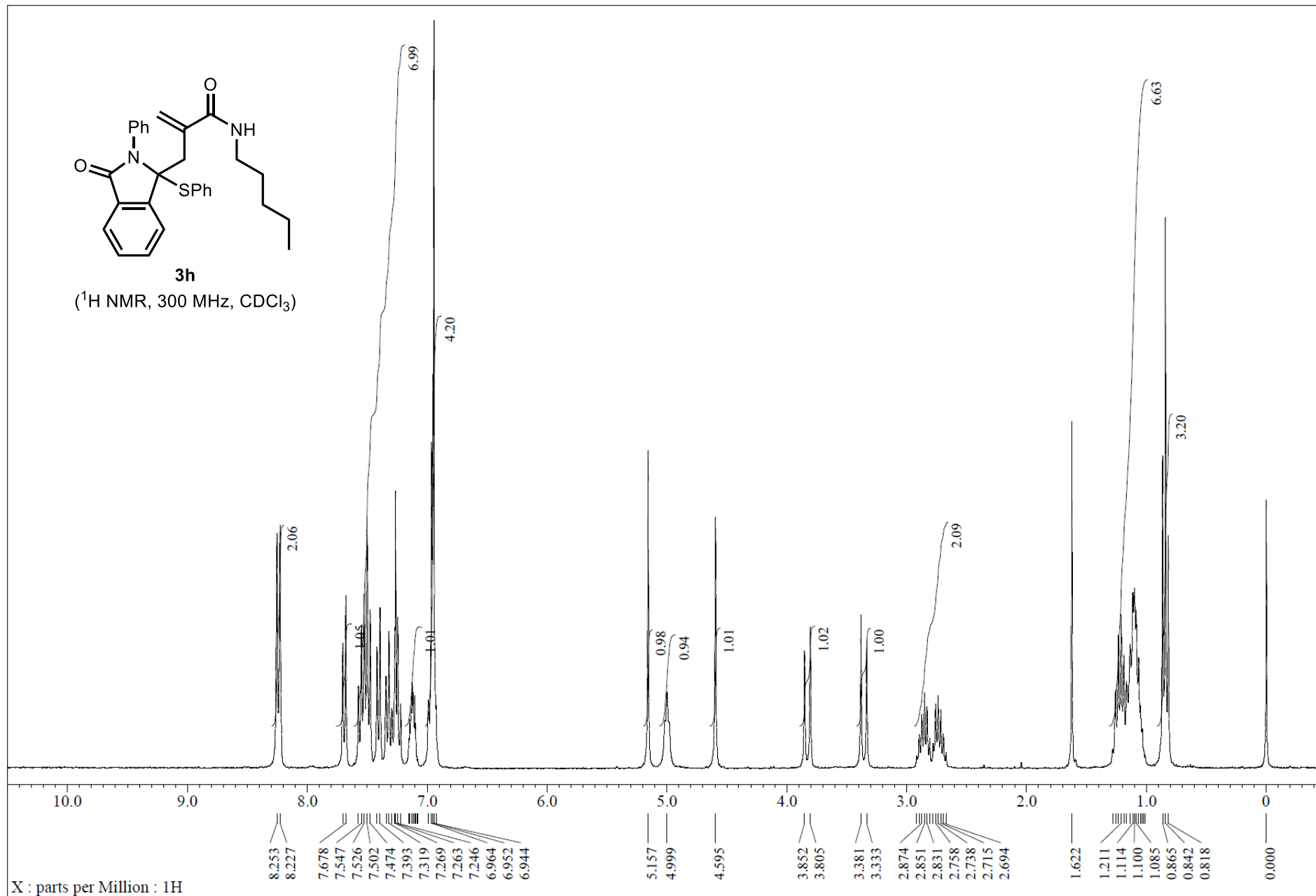
(^{13}C NMR, 75 MHz, CDCl_3)



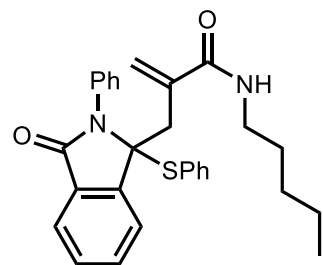
X : parts per Million : ^{13}C



3h
(^1H NMR, 300 MHz, CDCl_3)

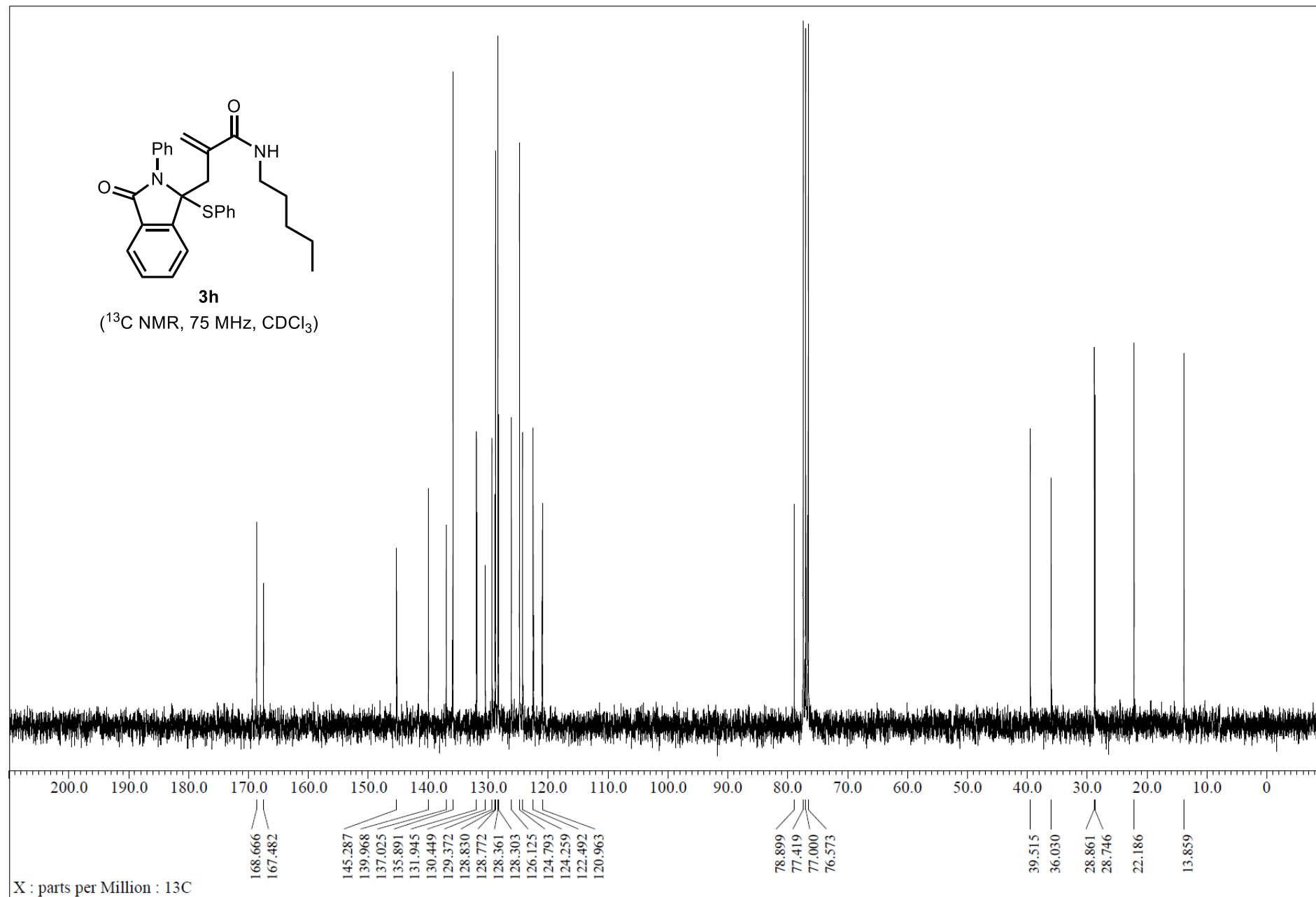


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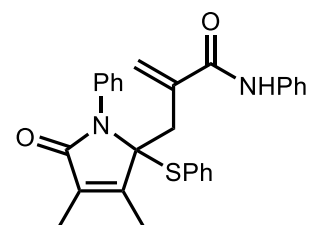


3h

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

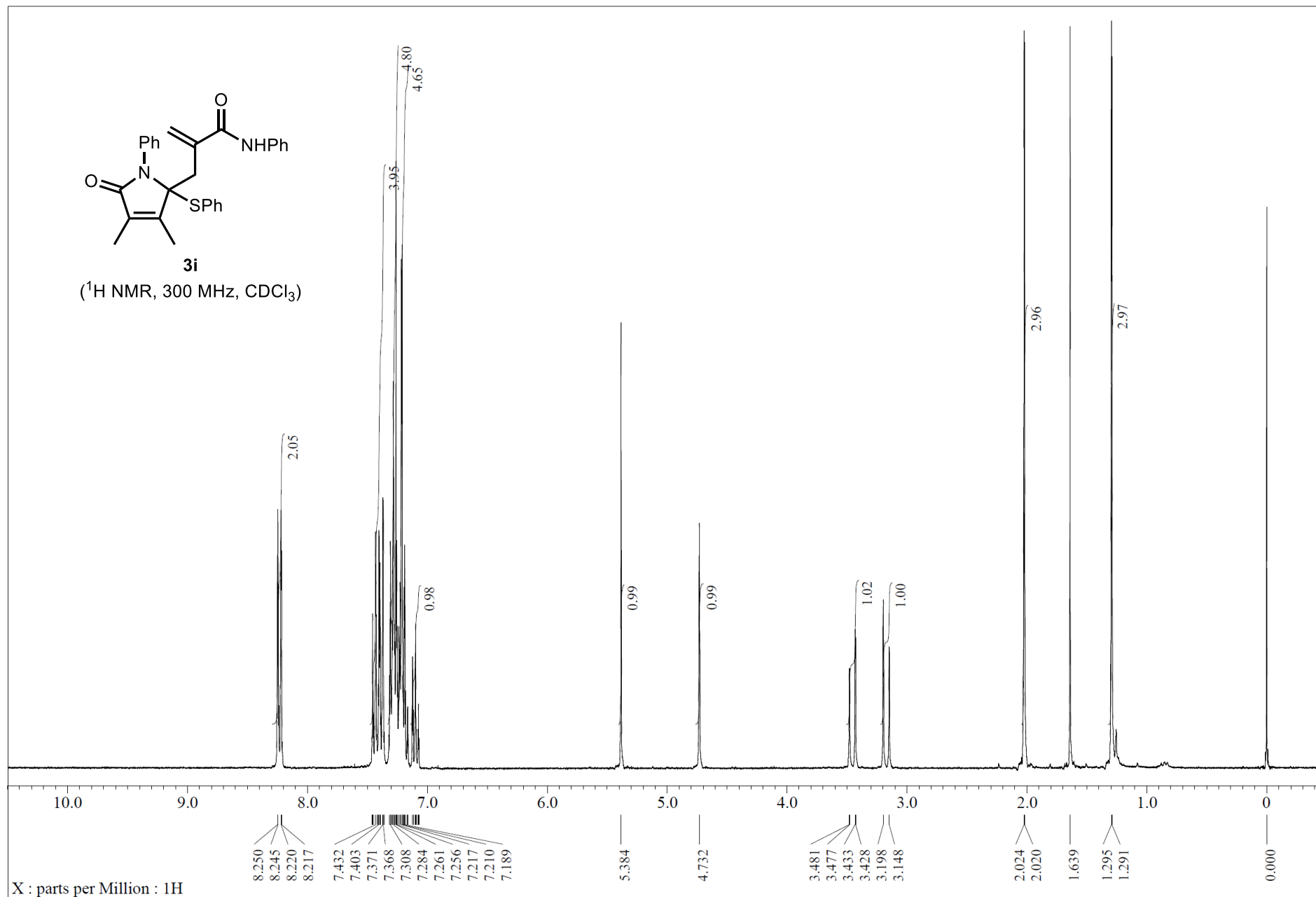


X : parts per Million : ^{13}C

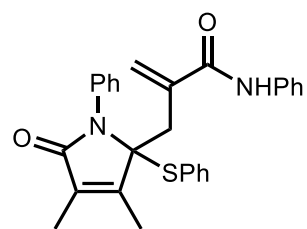


3i

(¹H NMR, 300 MHz, CDCl₃)

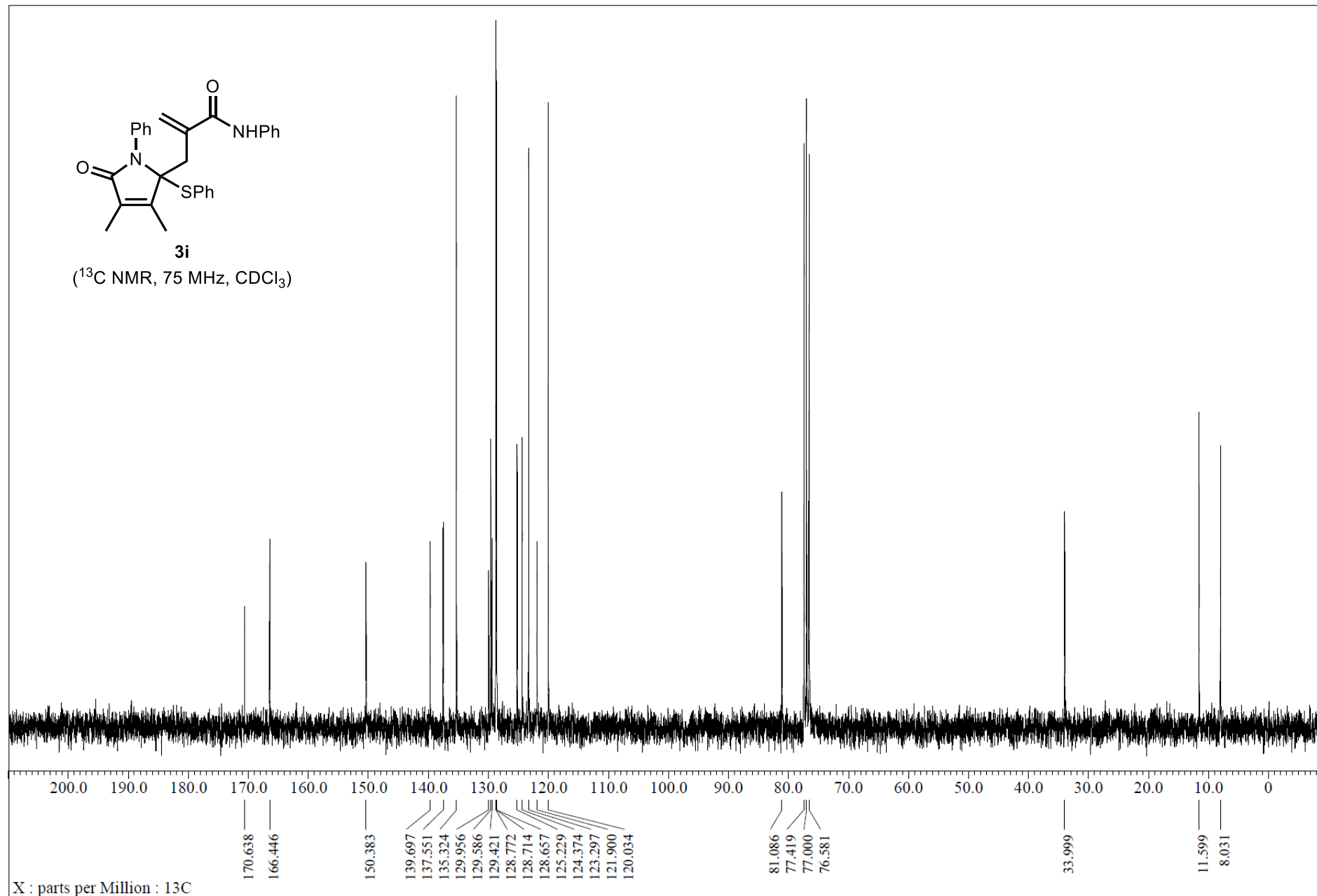


X : parts per Million : 1H

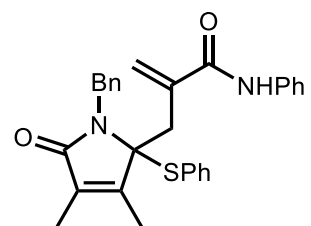


3i

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

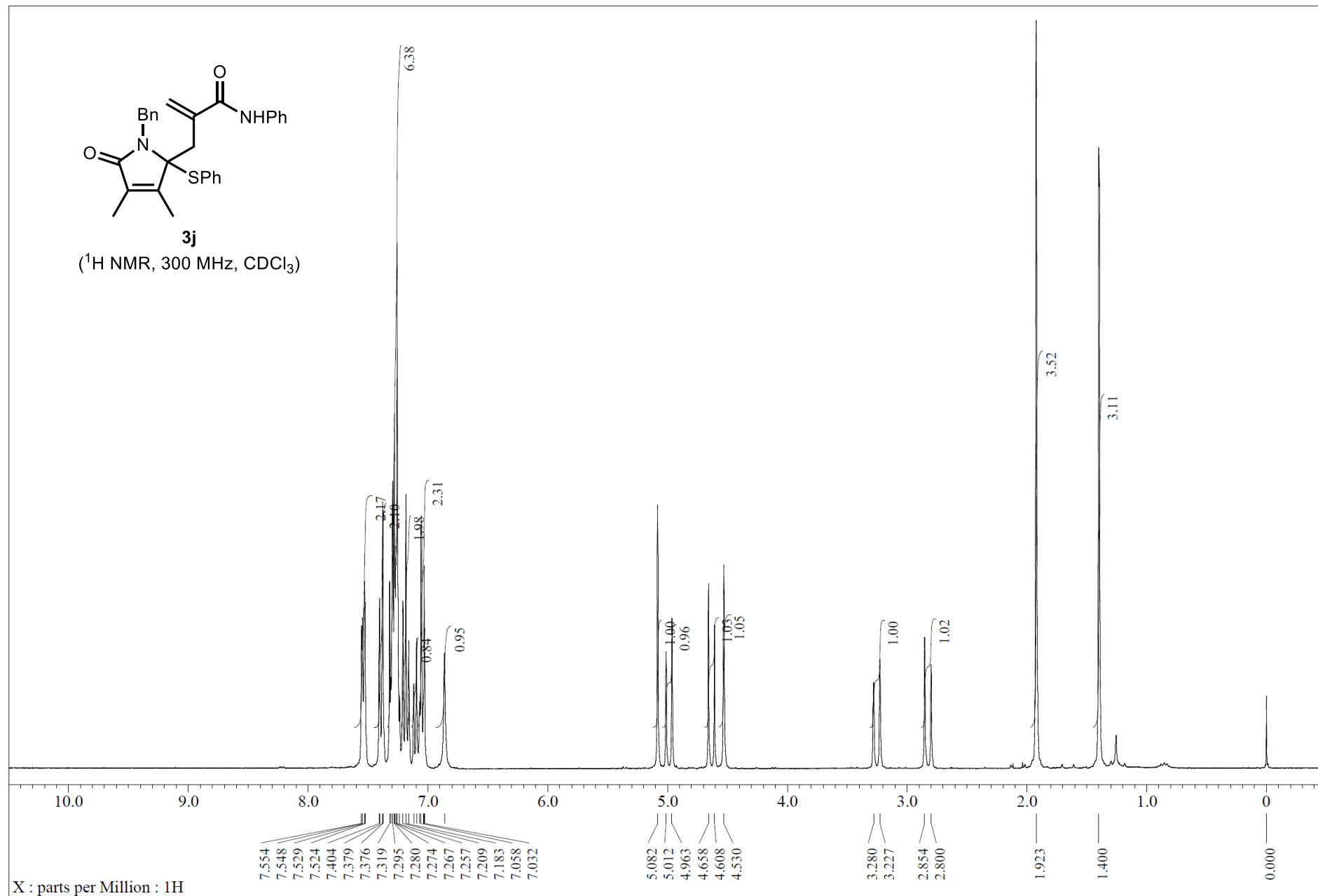


X : parts per Million : ^{13}C

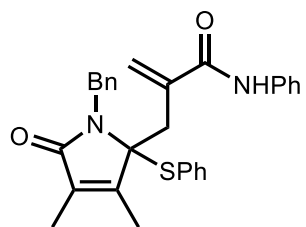


3j

(¹H NMR, 300 MHz, CDCl₃)

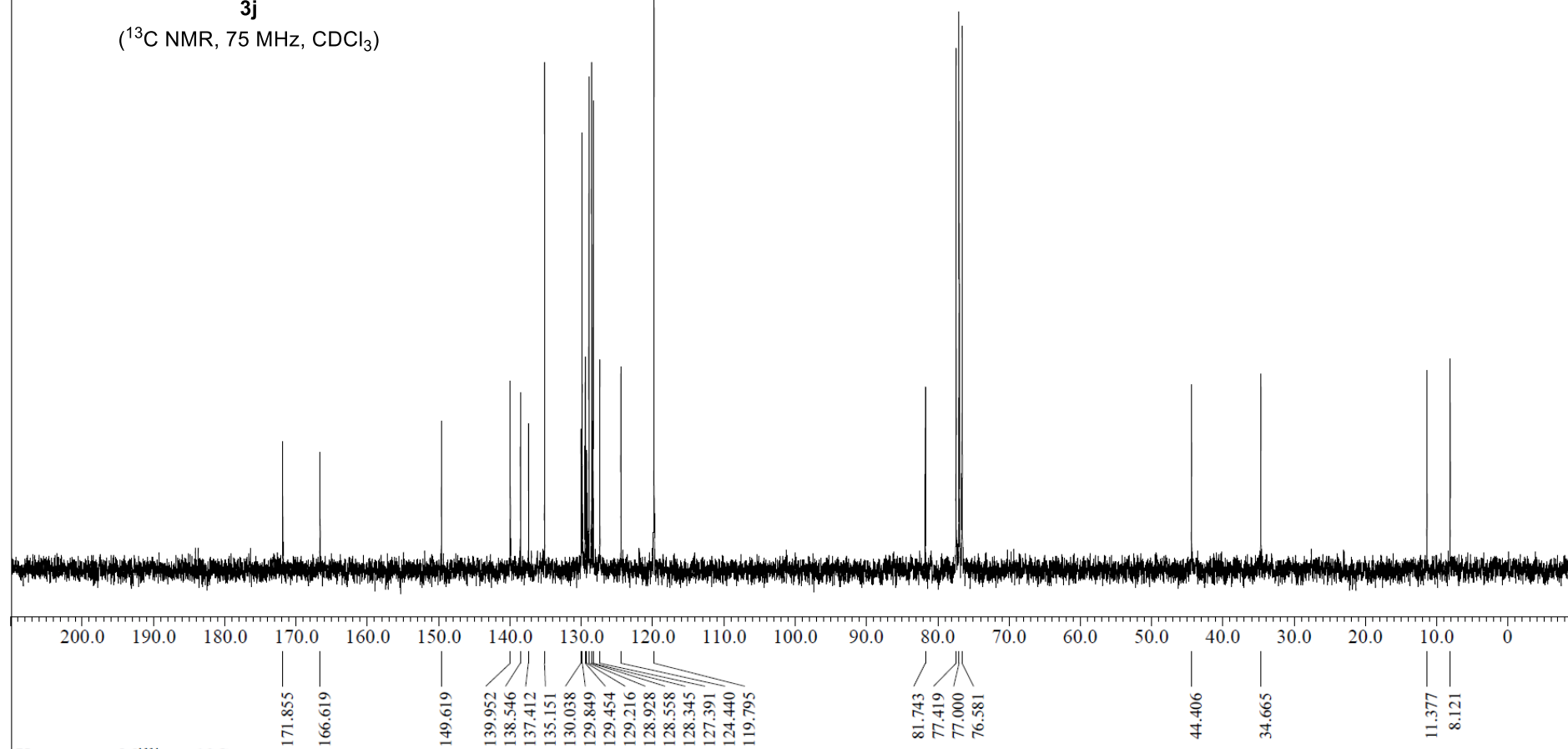


X : parts per Million : 1H

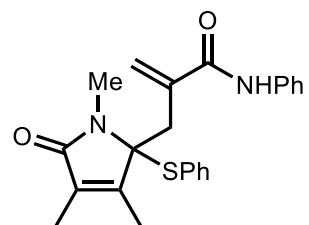


3j

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

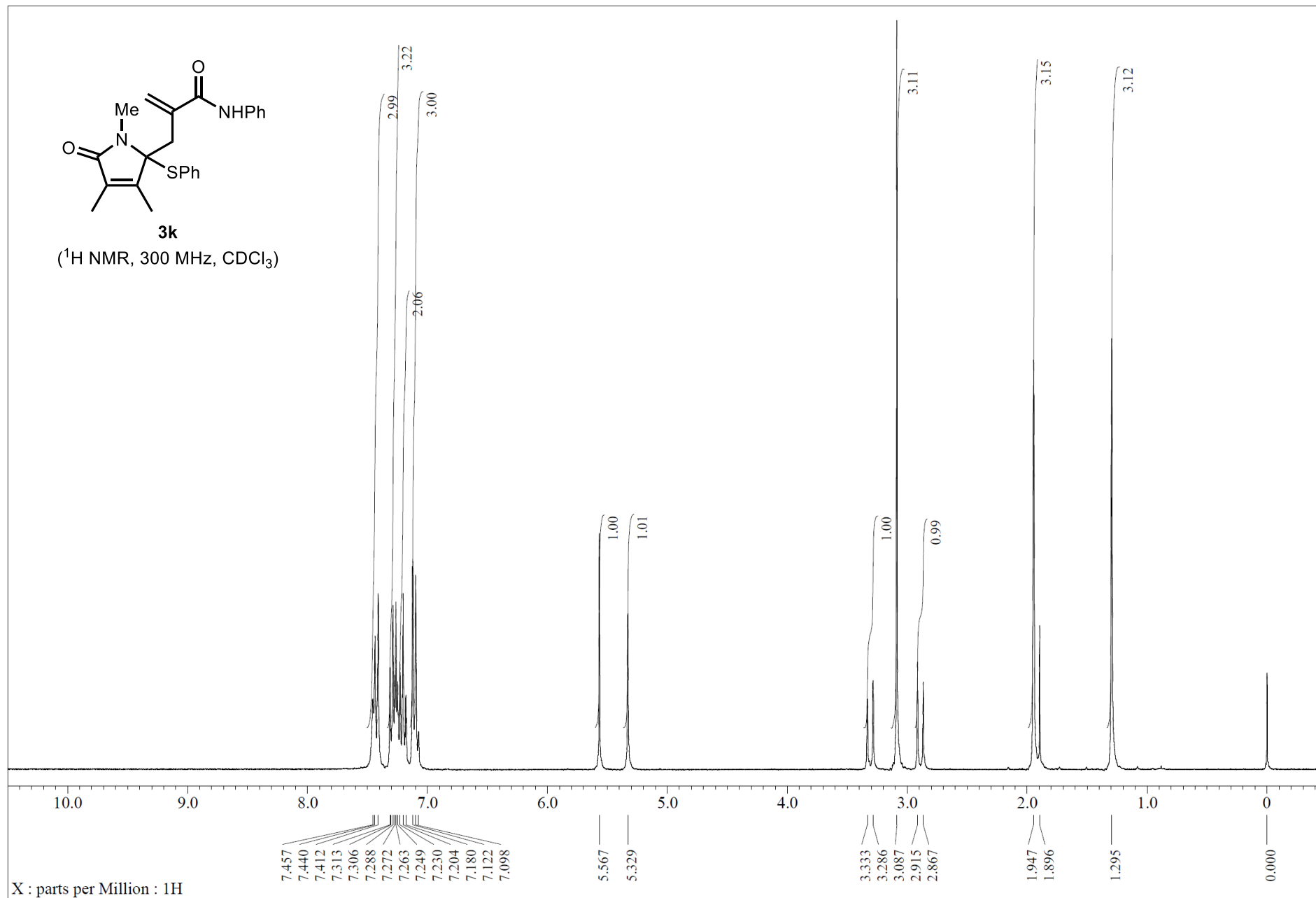


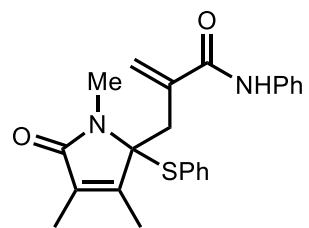
X : parts per Million : ^{13}C



3k

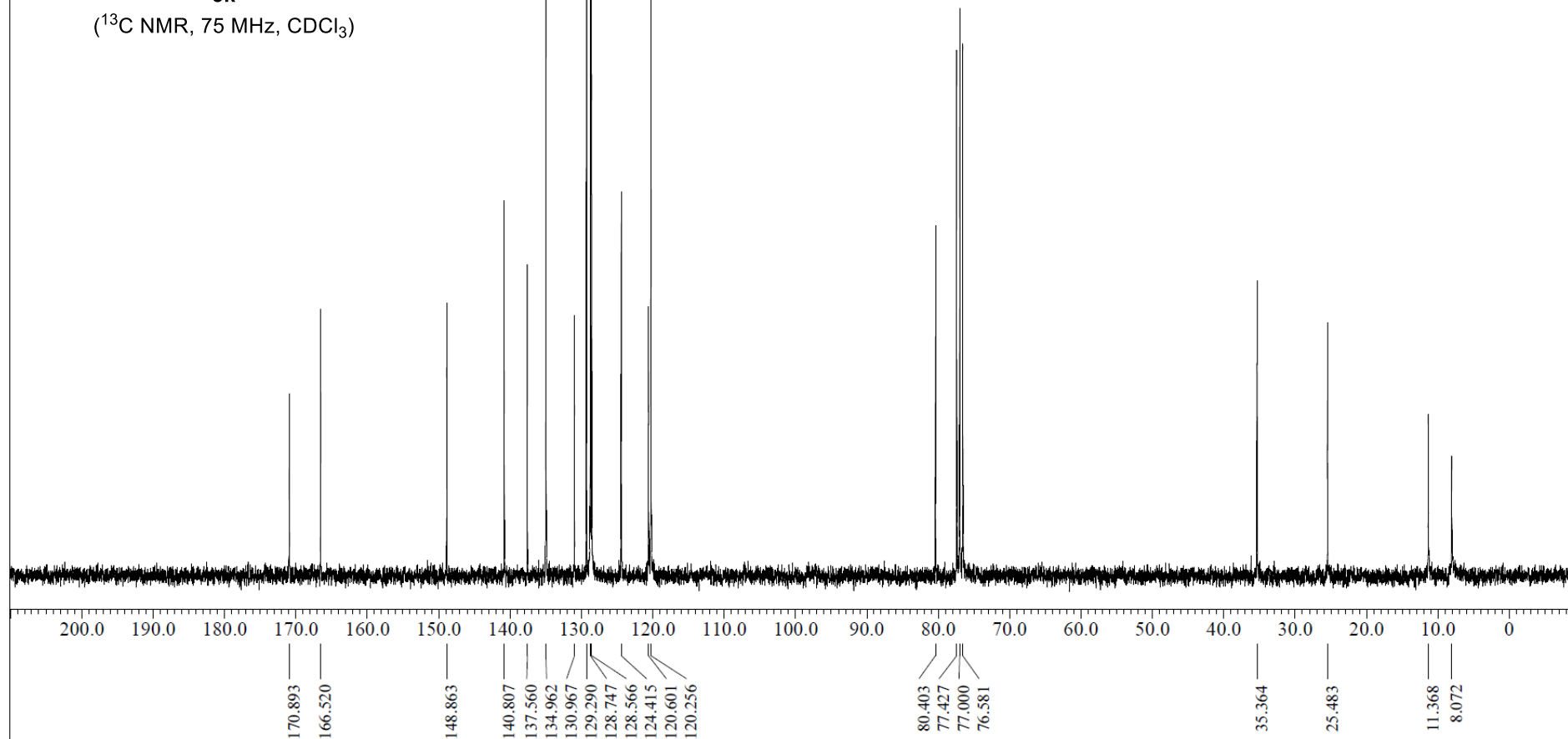
(^1H NMR, 300 MHz, CDCl_3)



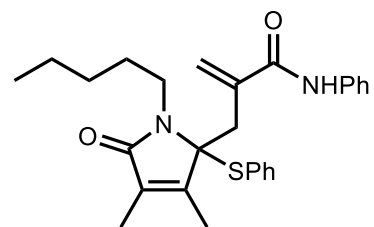


3k

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

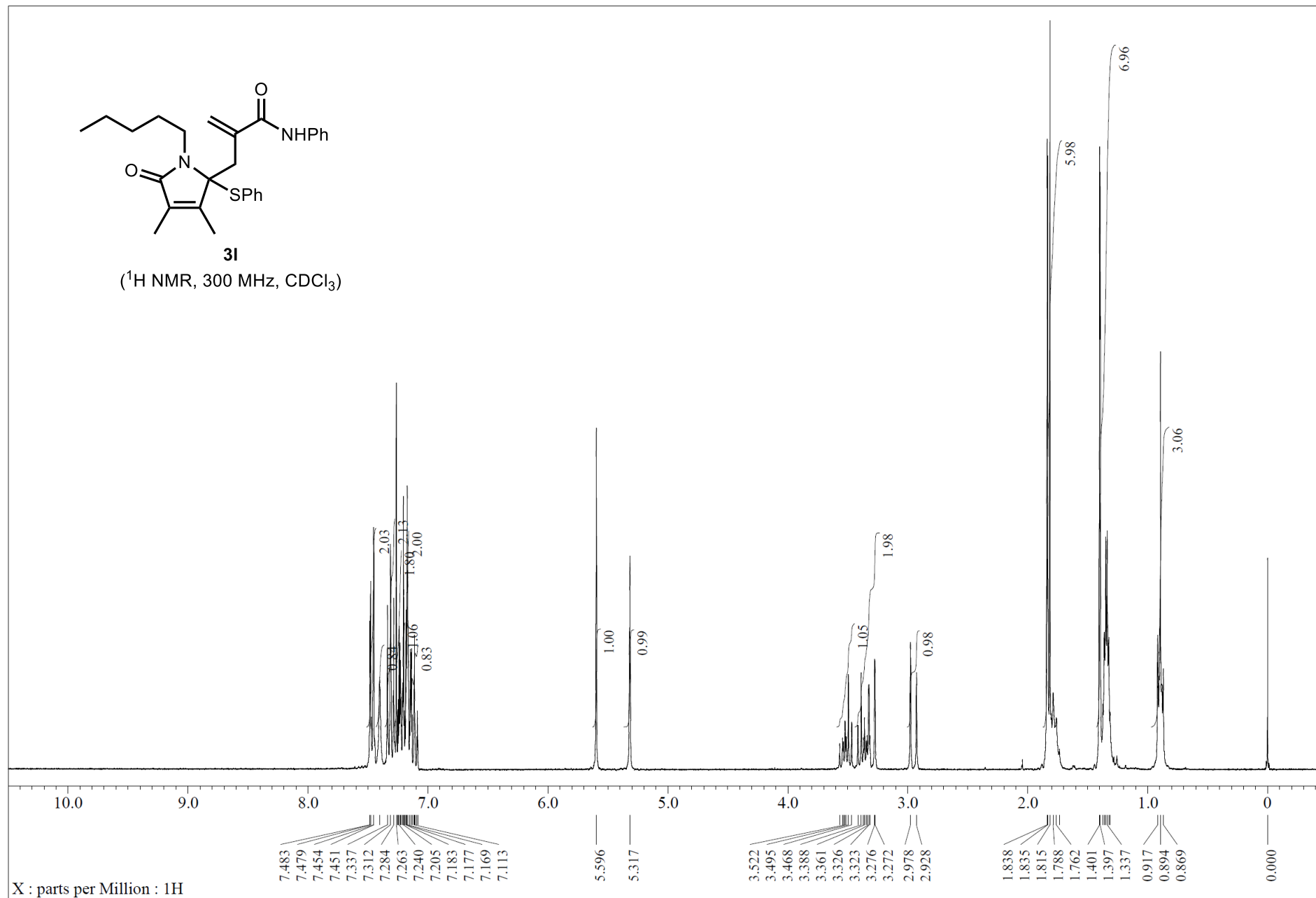


X : parts per Million : ^{13}C

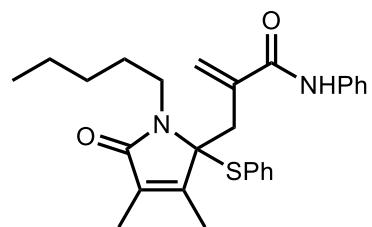


3l

(¹H NMR, 300 MHz, CDCl₃)

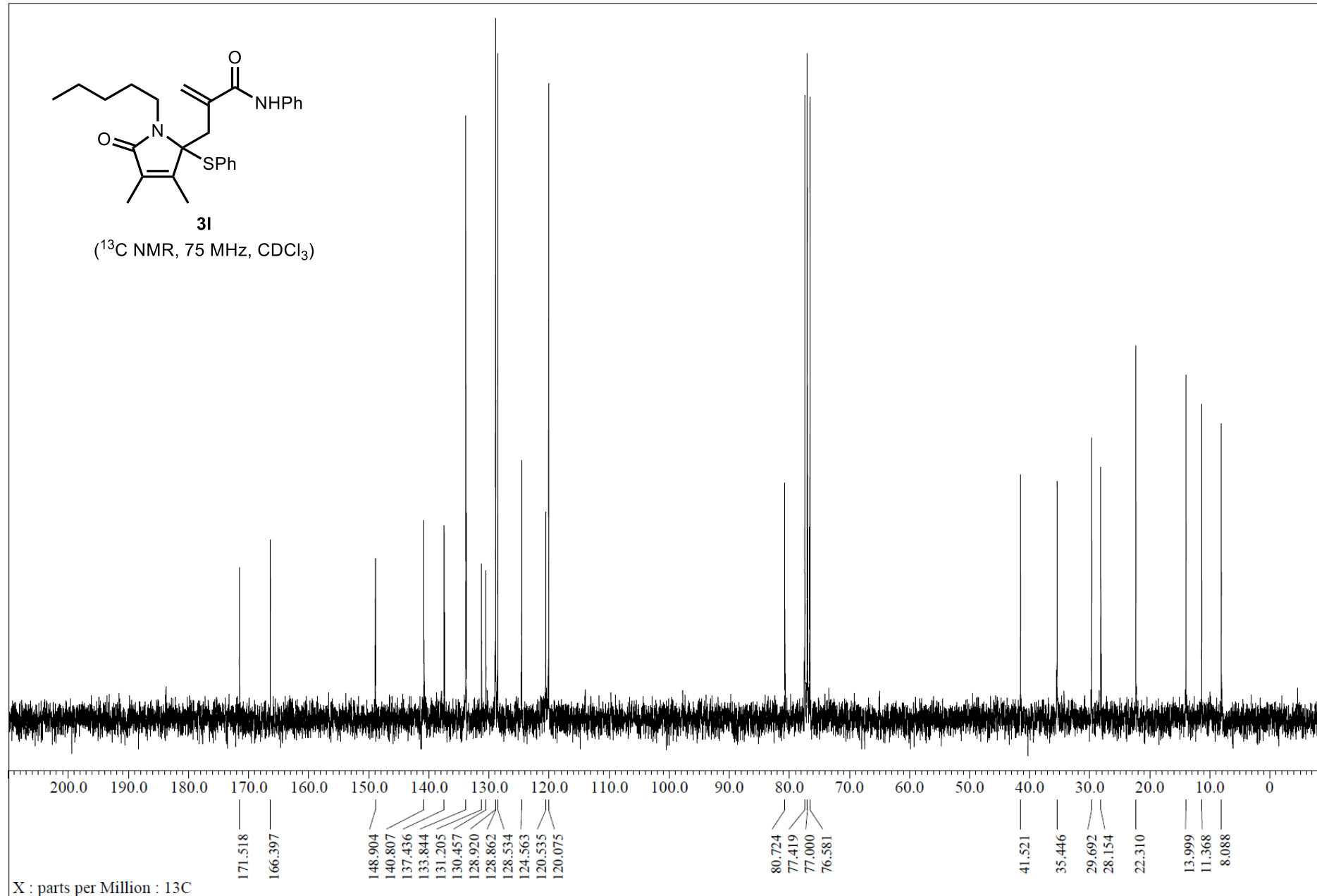


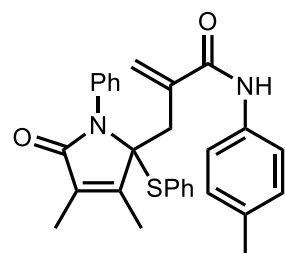
X : parts per Million : 1H



3I

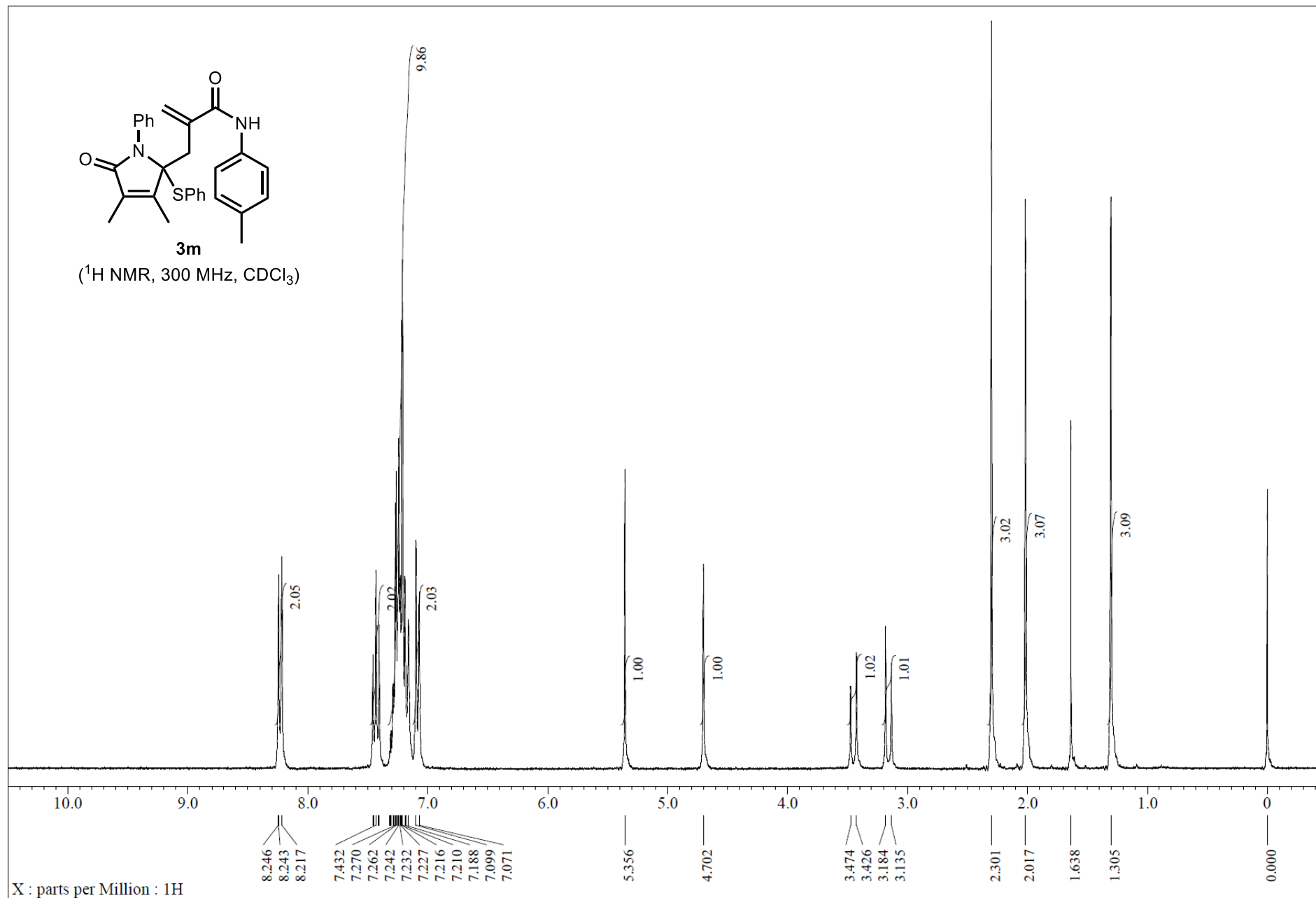
(^{13}C NMR, 75 MHz, CDCl_3)

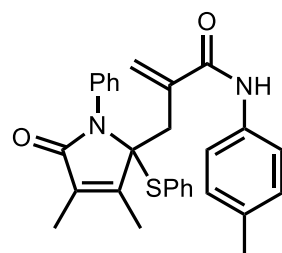




3m

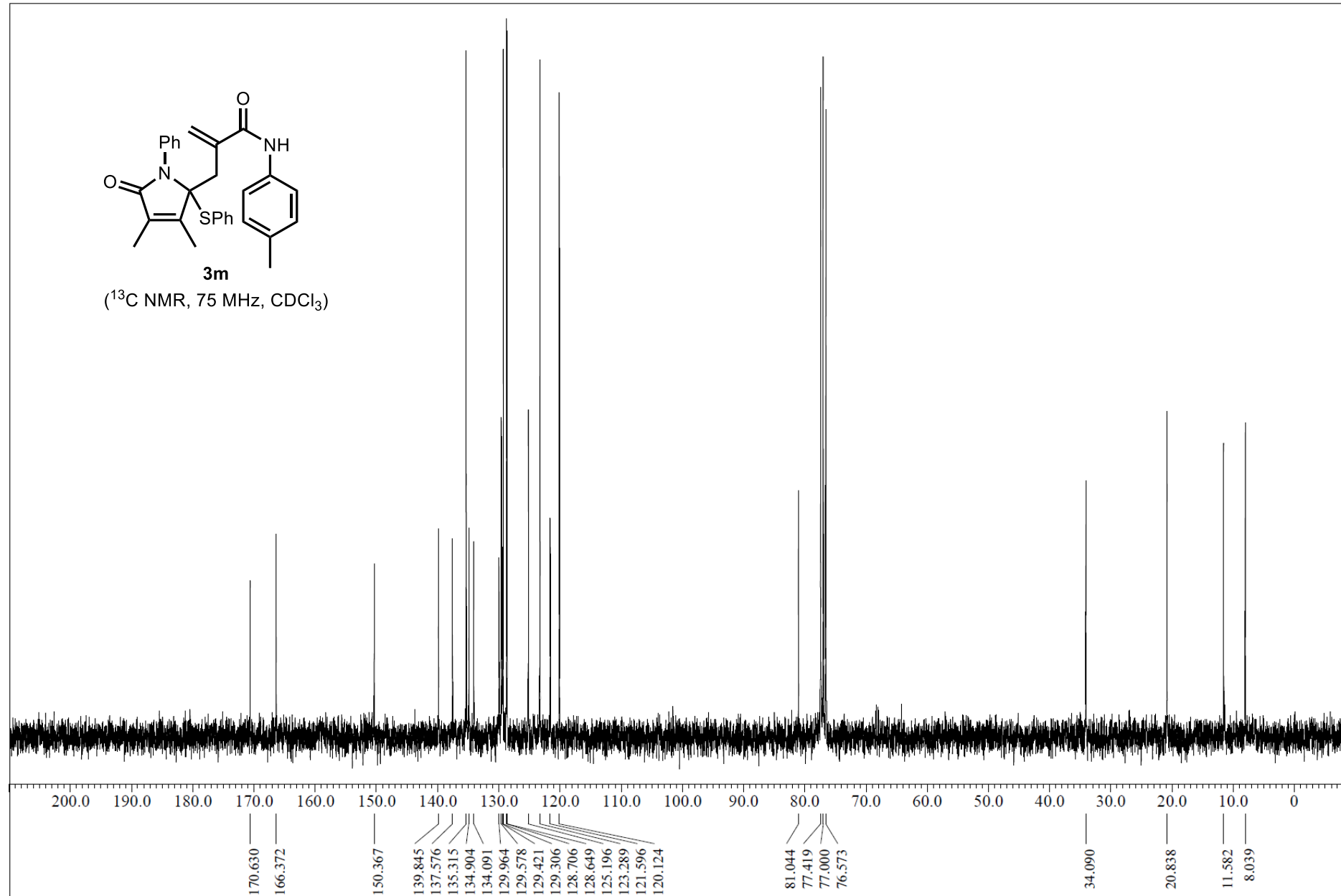
(¹H NMR, 300 MHz, CDCl₃)



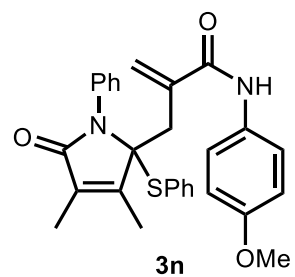


3m

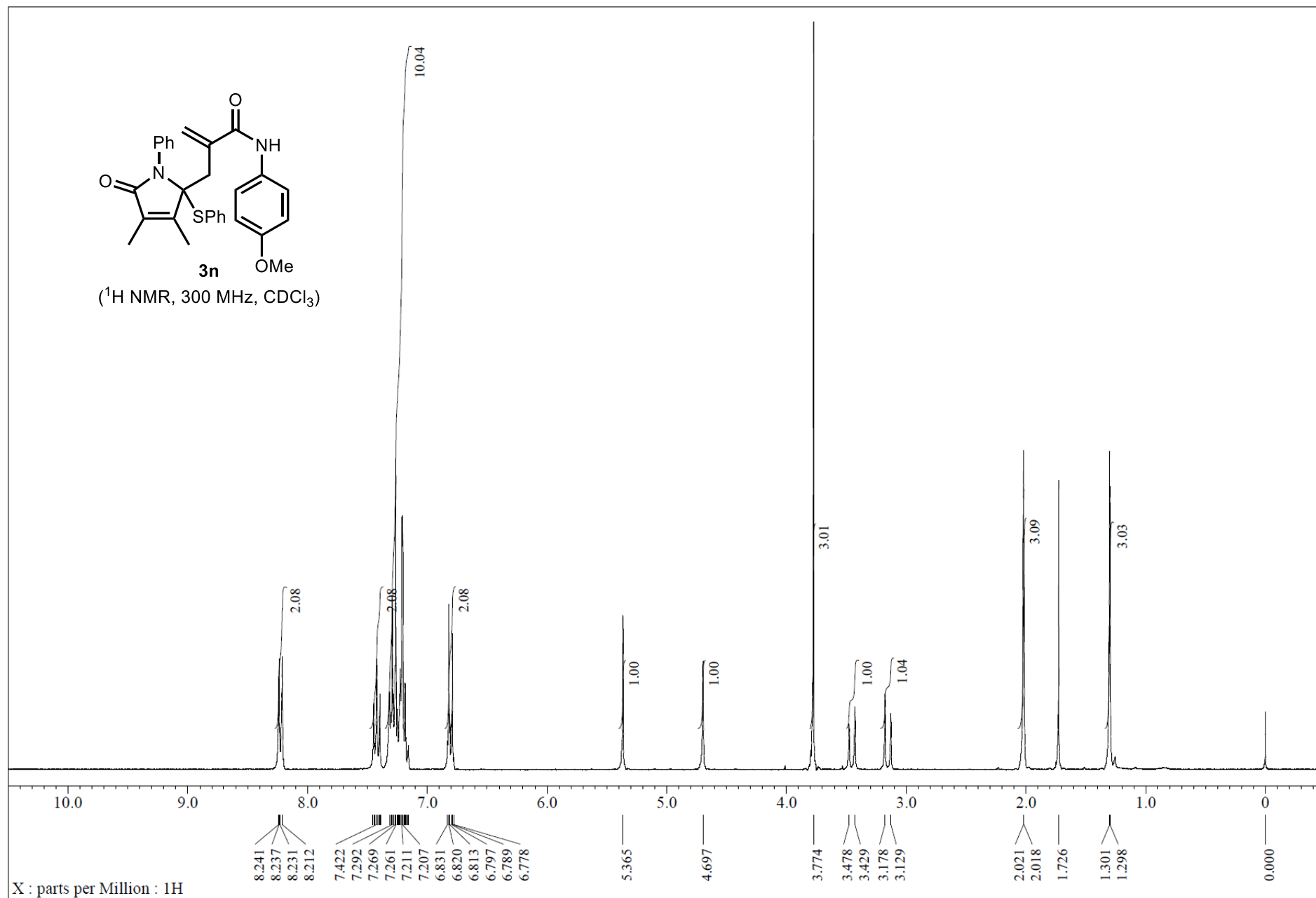
(^{13}C NMR, 75 MHz, CDCl_3)



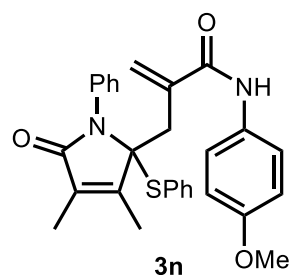
X : parts per Million : ^{13}C



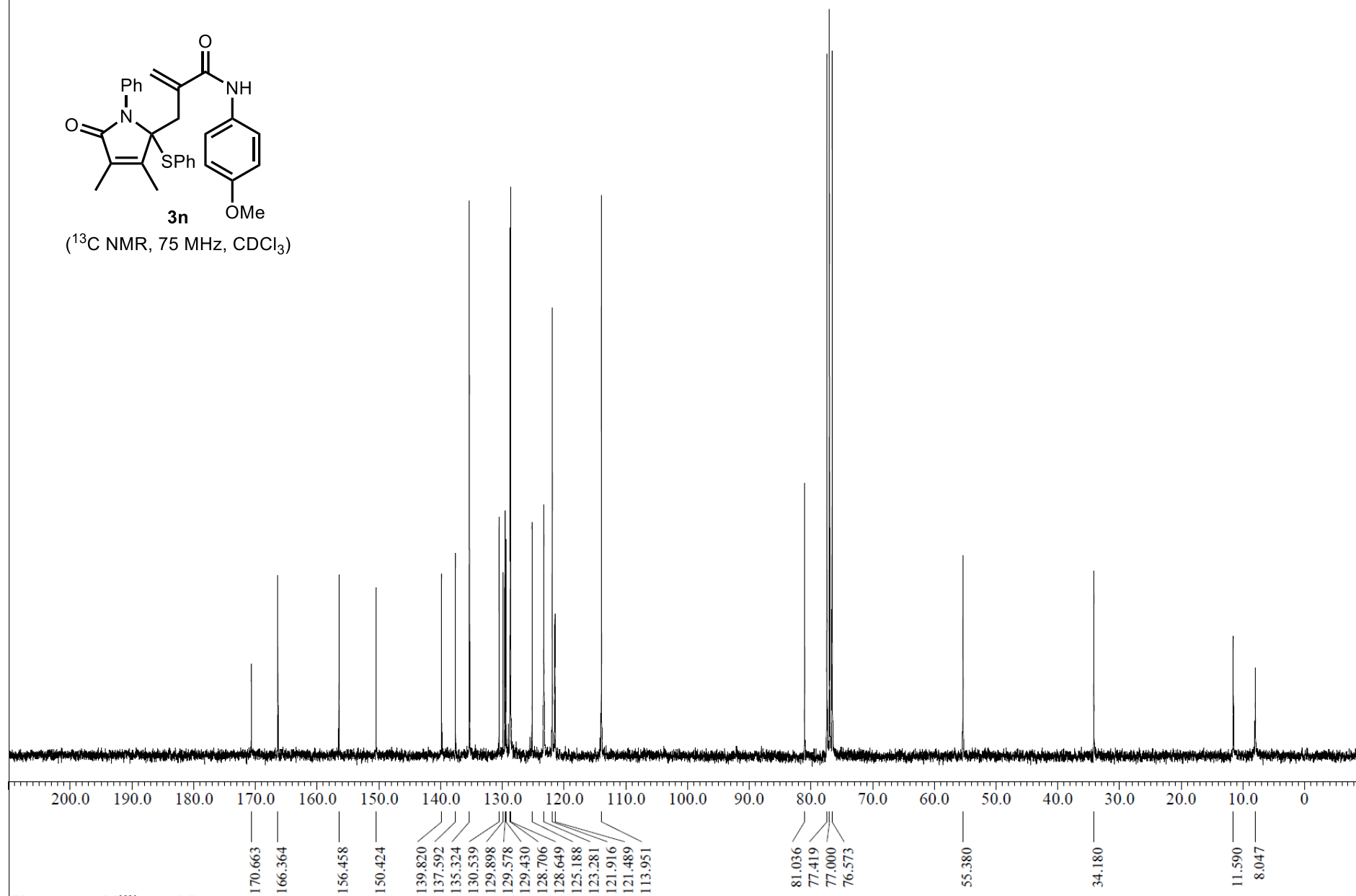
(¹H NMR, 300 MHz, CDCl₃)



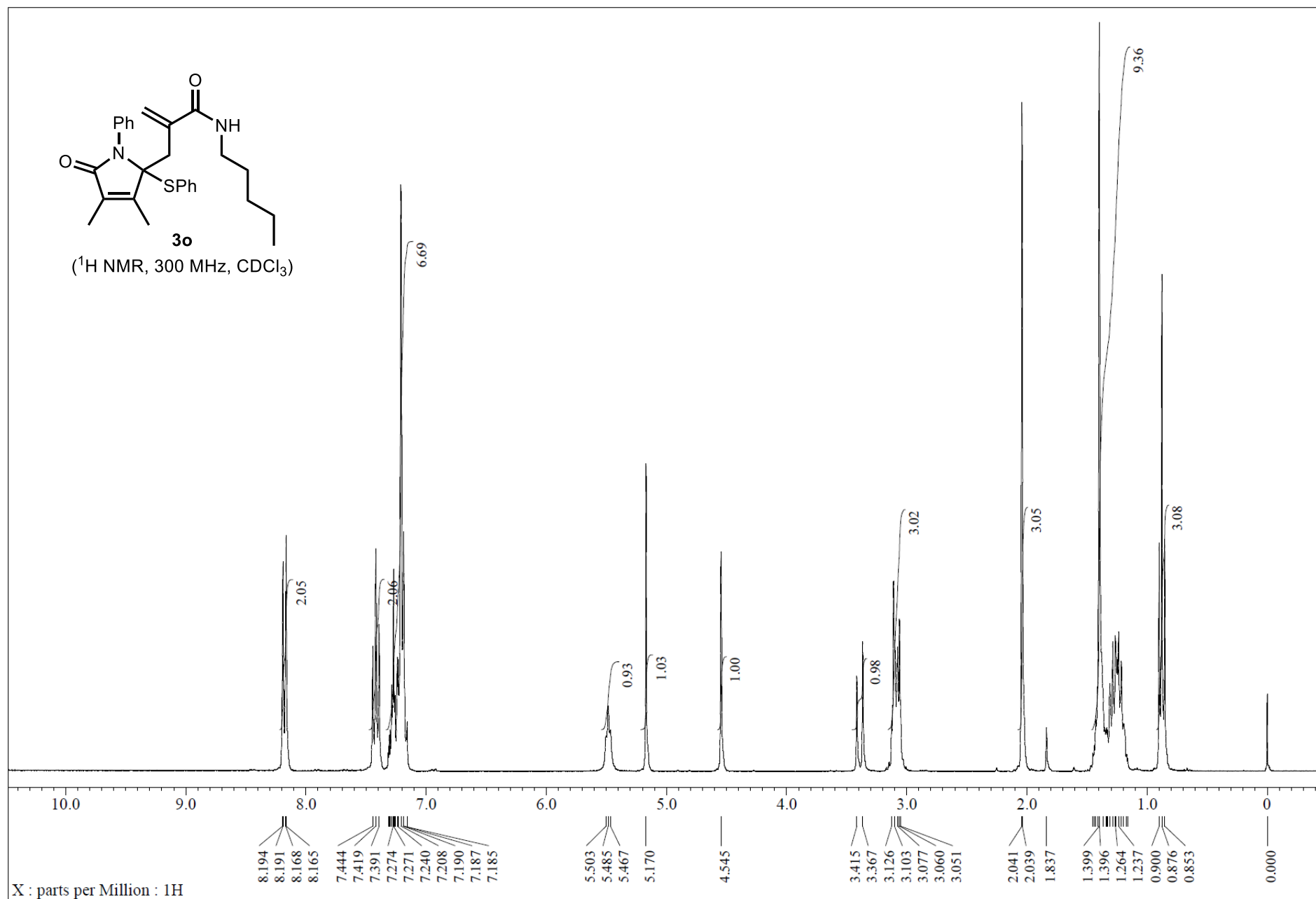
X : parts per Million : 1H

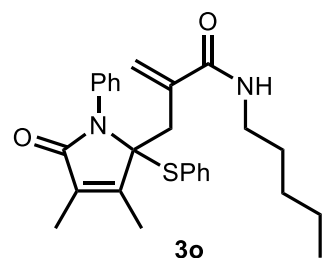


(^{13}C NMR, 75 MHz, CDCl_3)

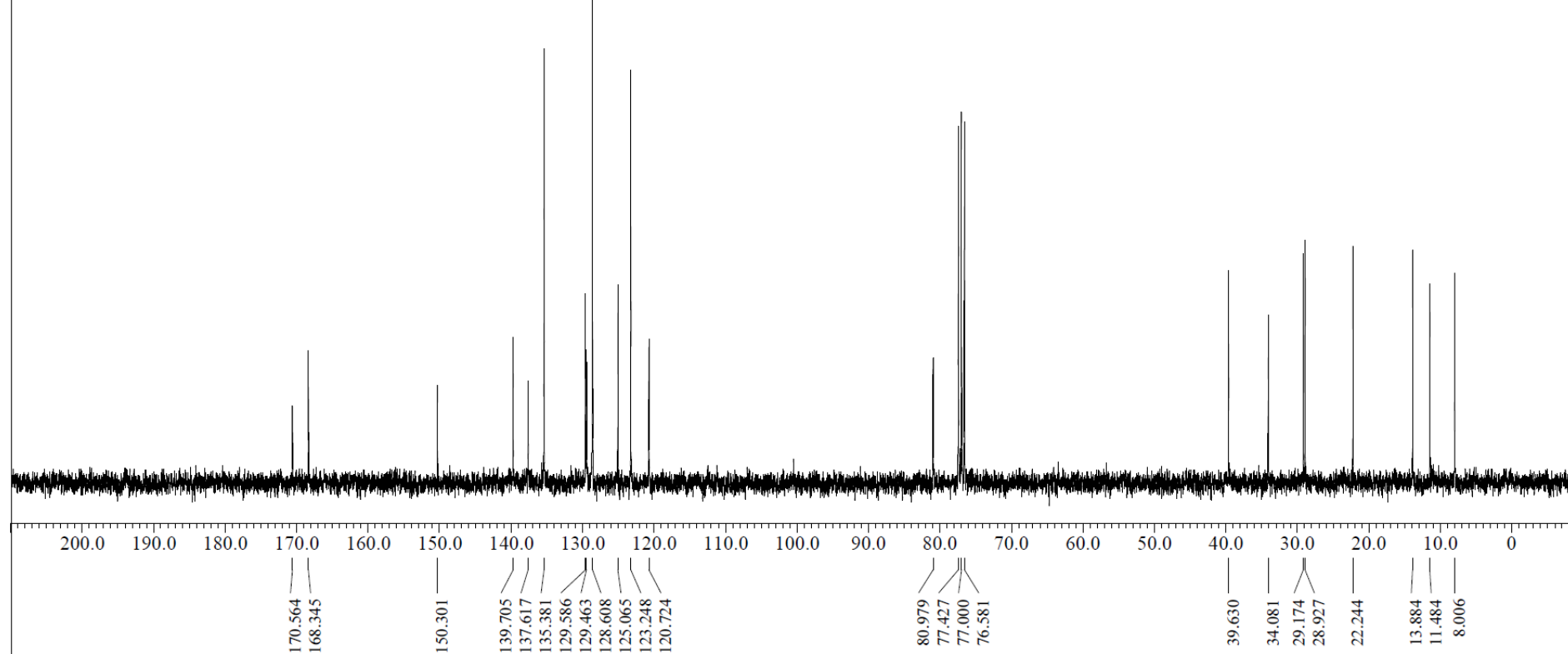


X : parts per Million : ^{13}C

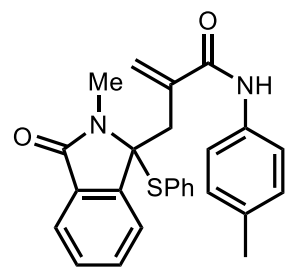




(^{13}C NMR, 75 MHz, CDCl_3)

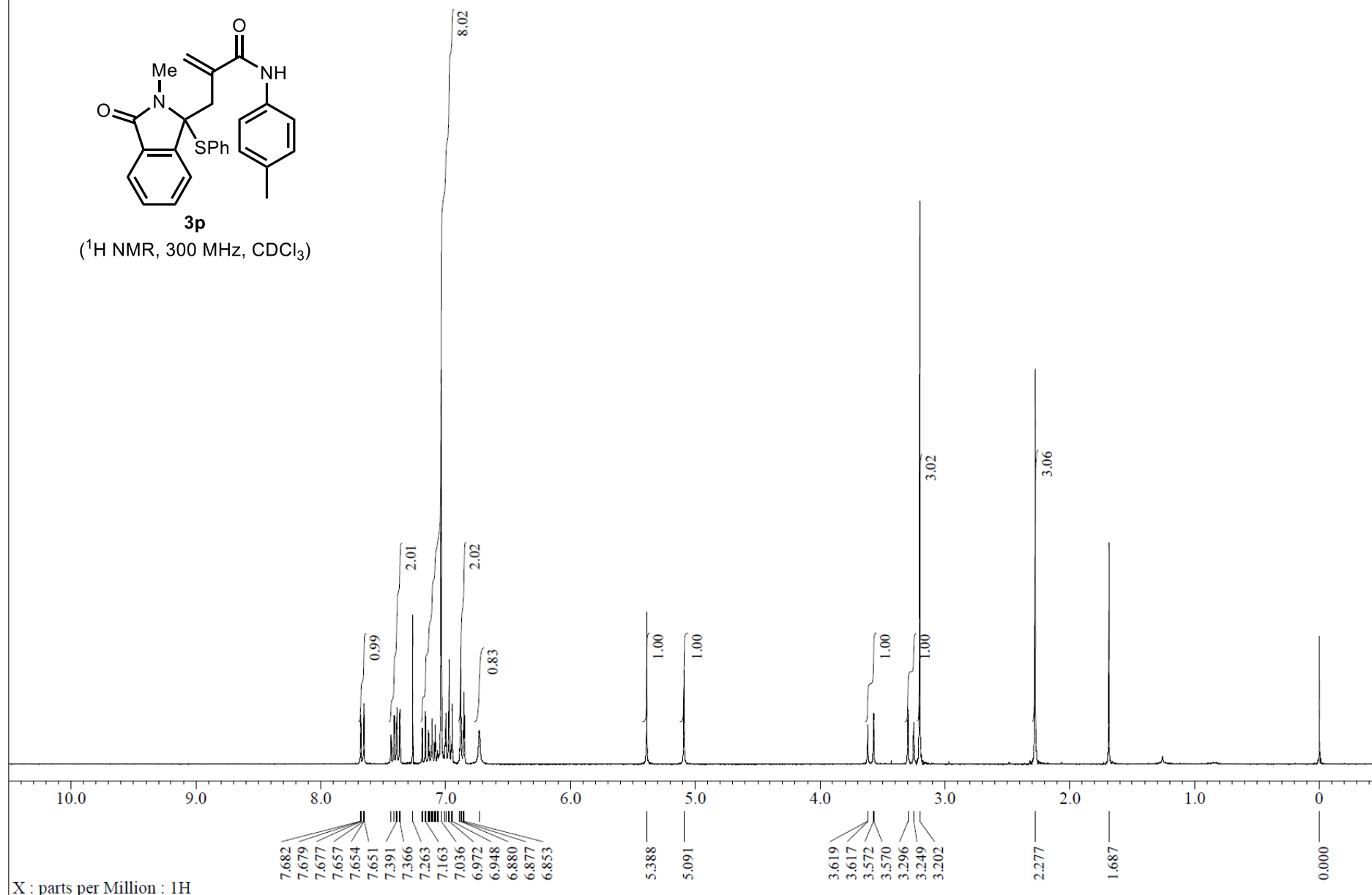


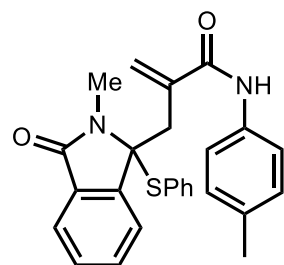
X : parts per Million : ^{13}C



3p

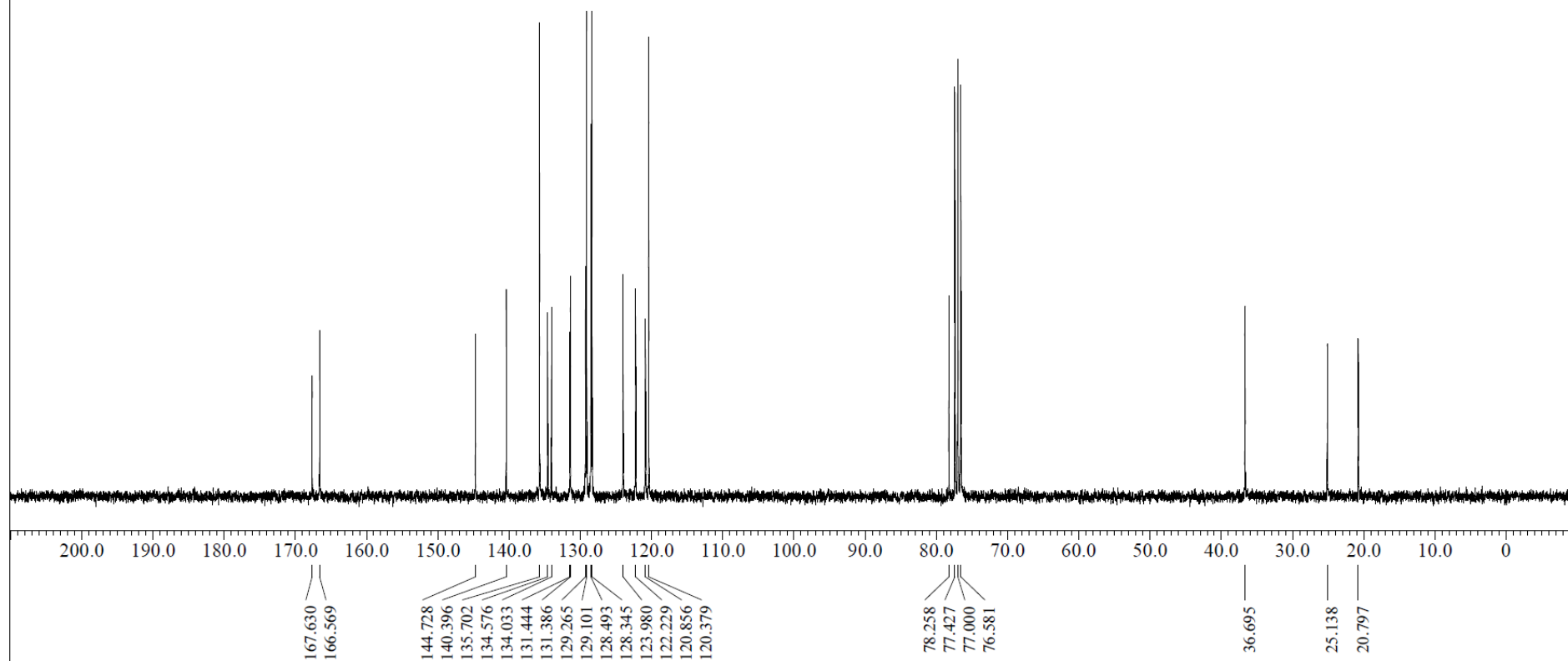
(¹H NMR, 300 MHz, CDCl₃)



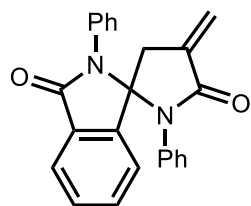


3p

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

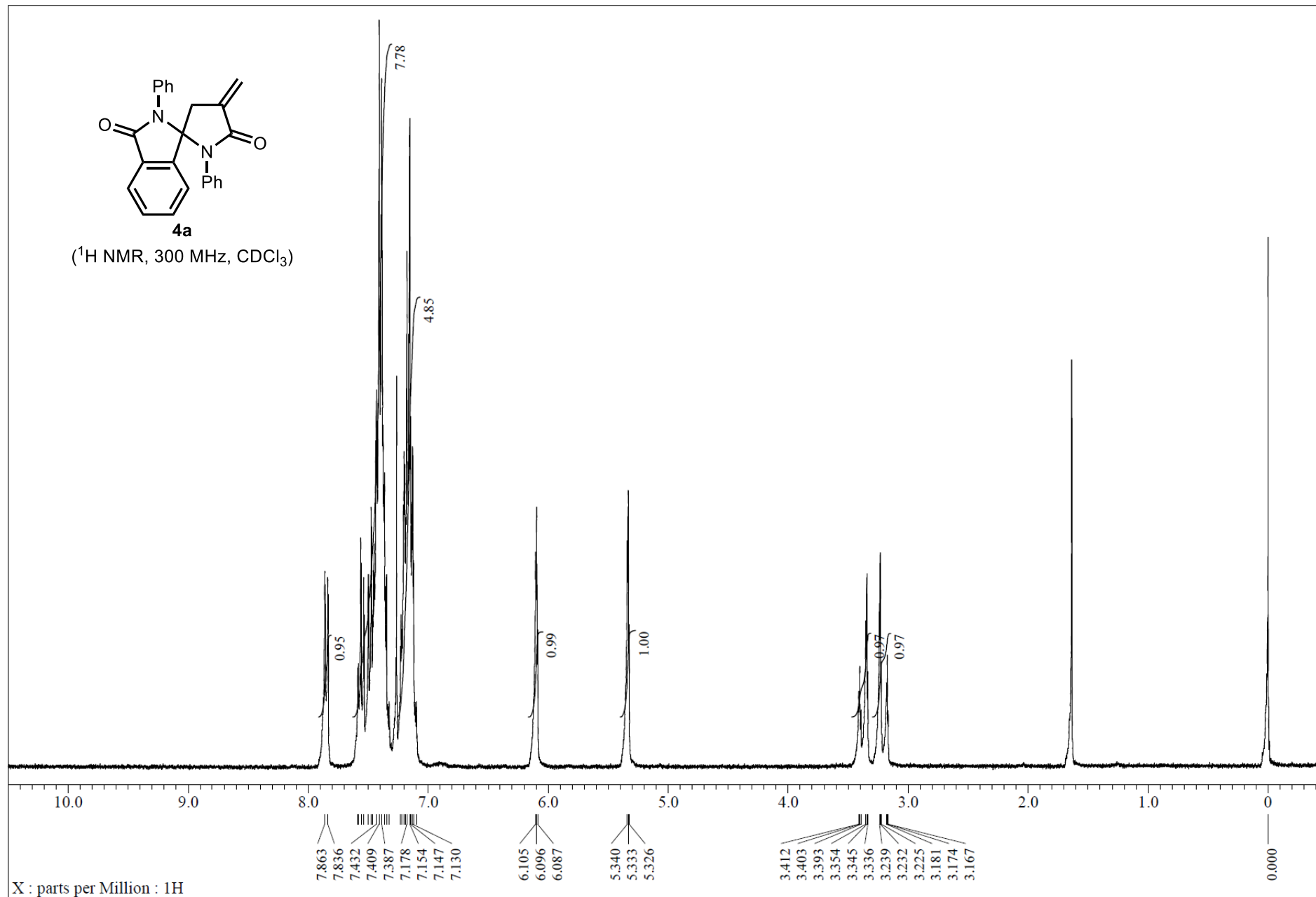


X : parts per Million : ^{13}C

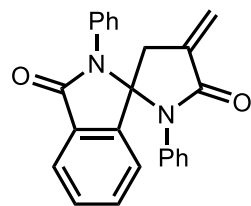


4a

(¹H NMR, 300 MHz, CDCl₃)

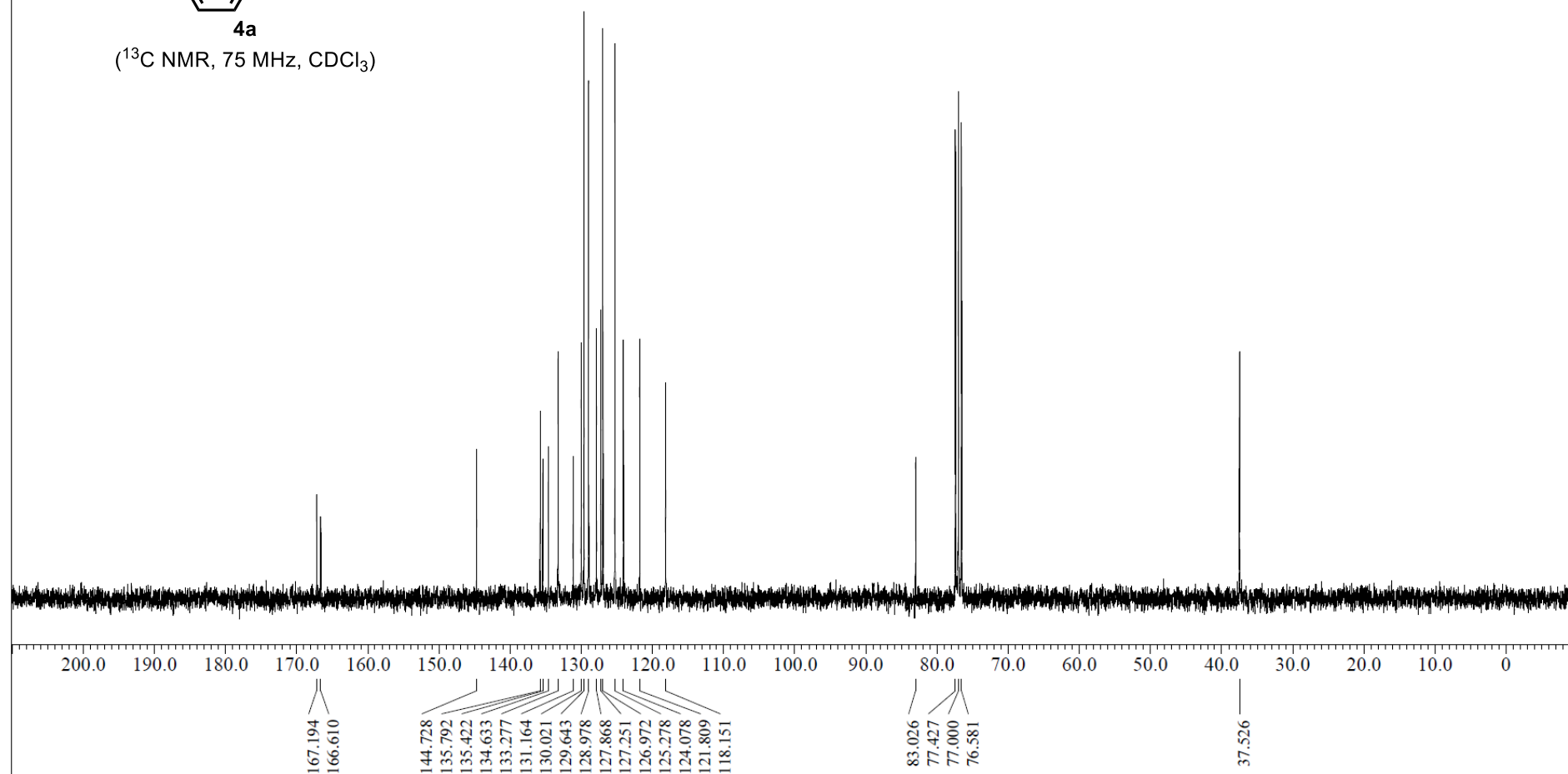


X : parts per Million : 1H

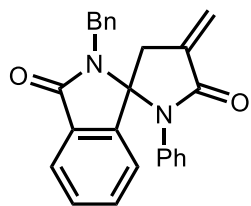


4a

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

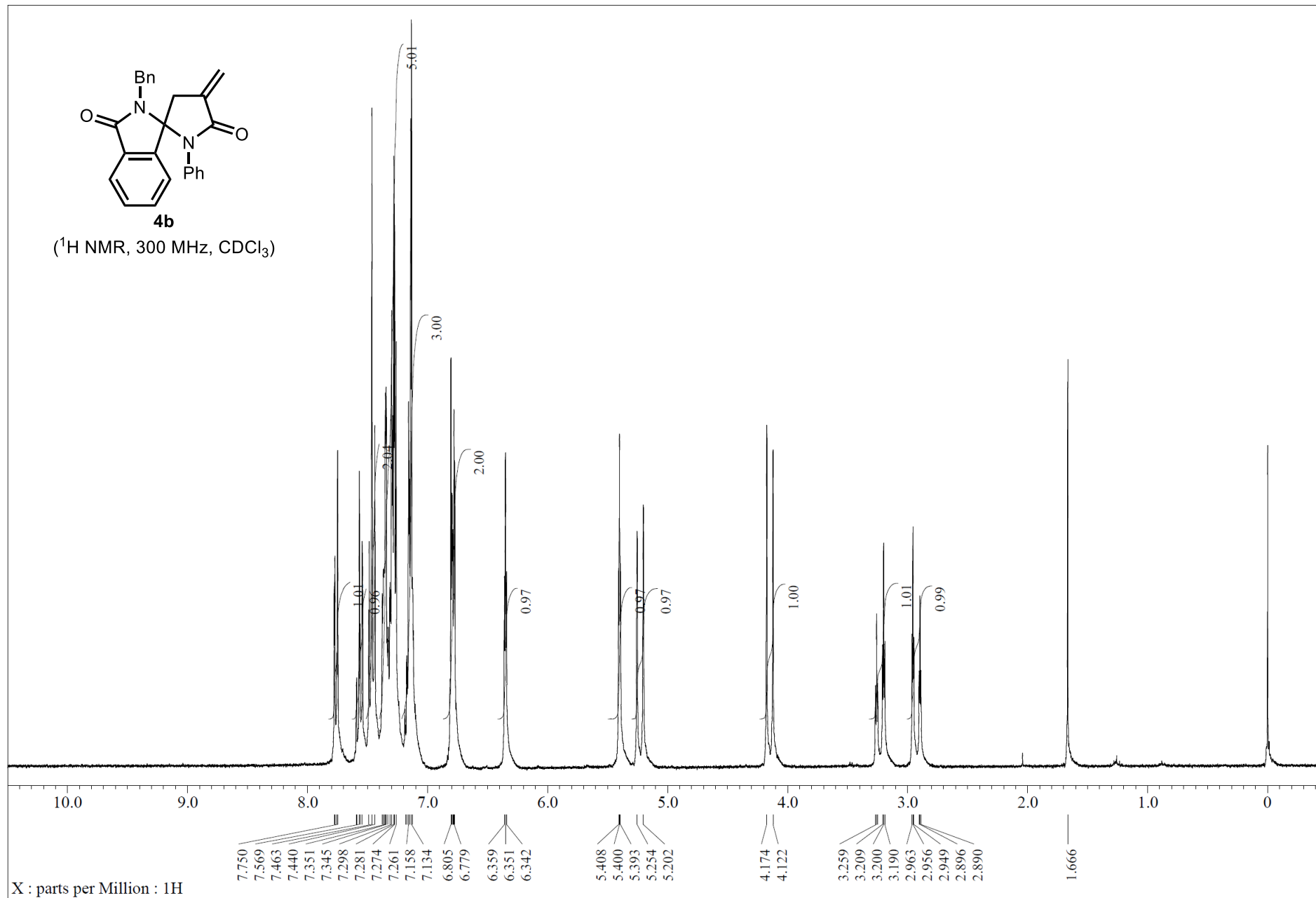


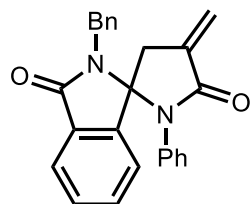
X : parts per Million : ^{13}C



4b

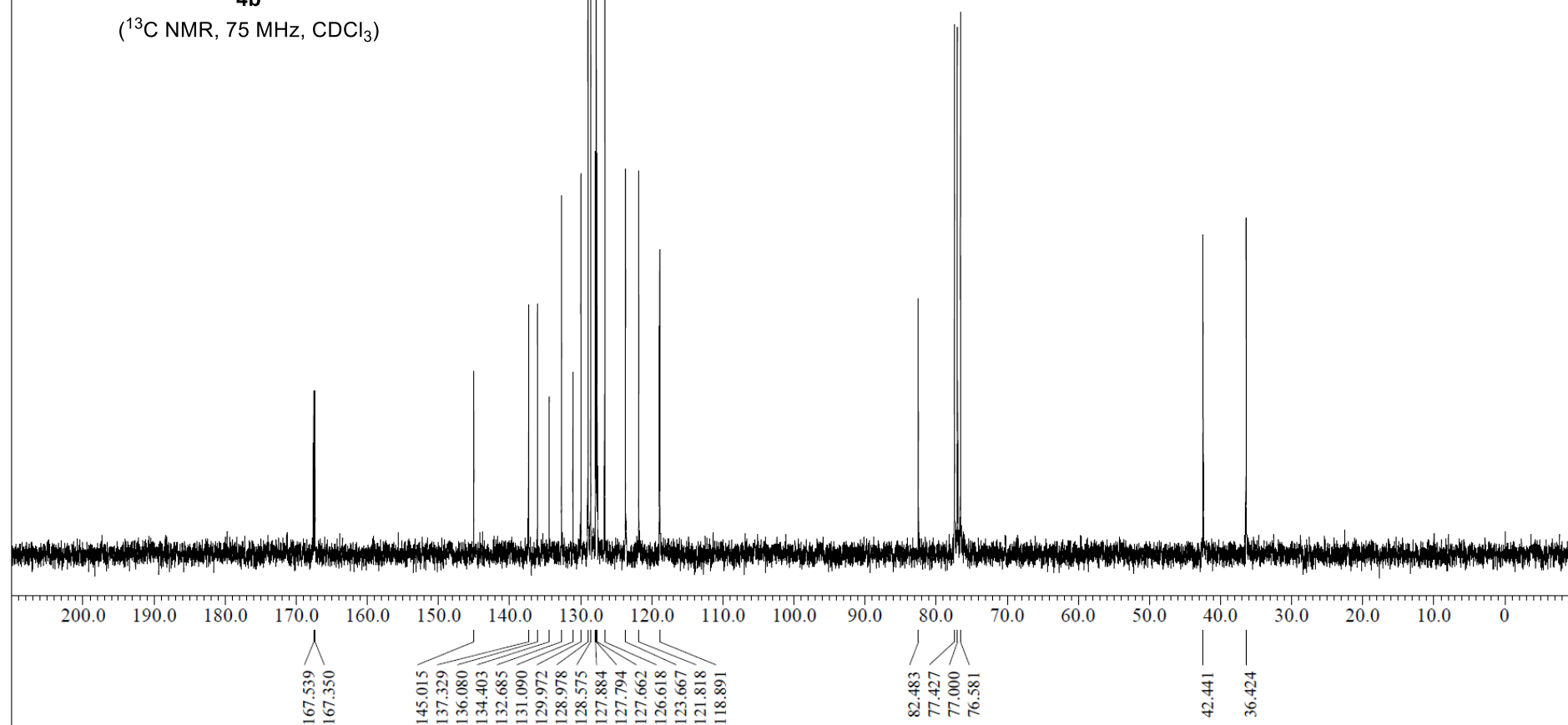
(¹H NMR, 300 MHz, CDCl₃)



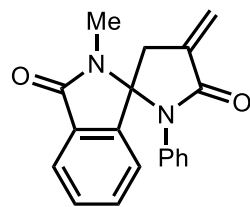


4b

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

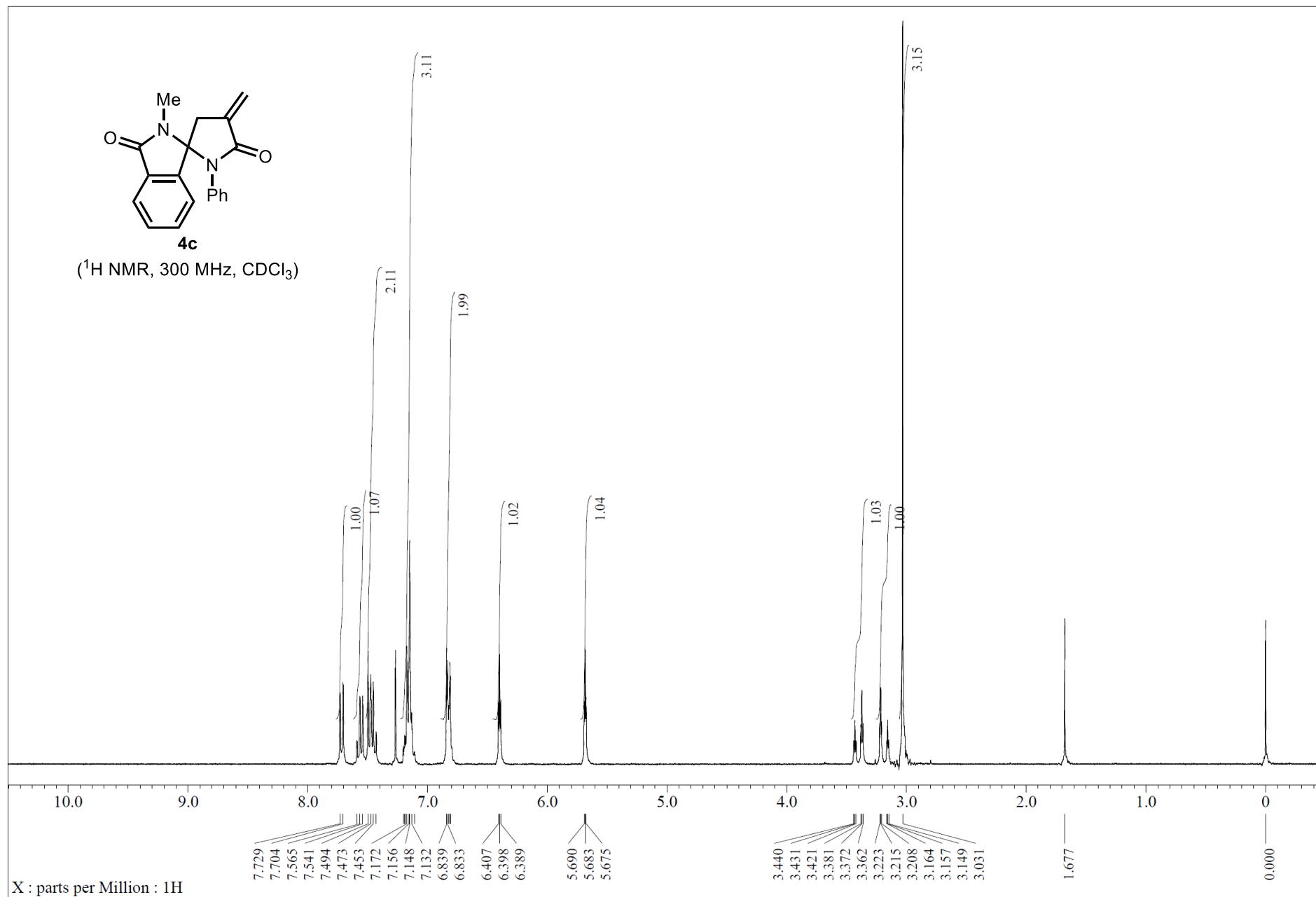


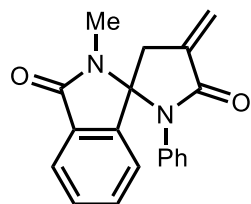
X : parts per Million : ^{13}C



4c

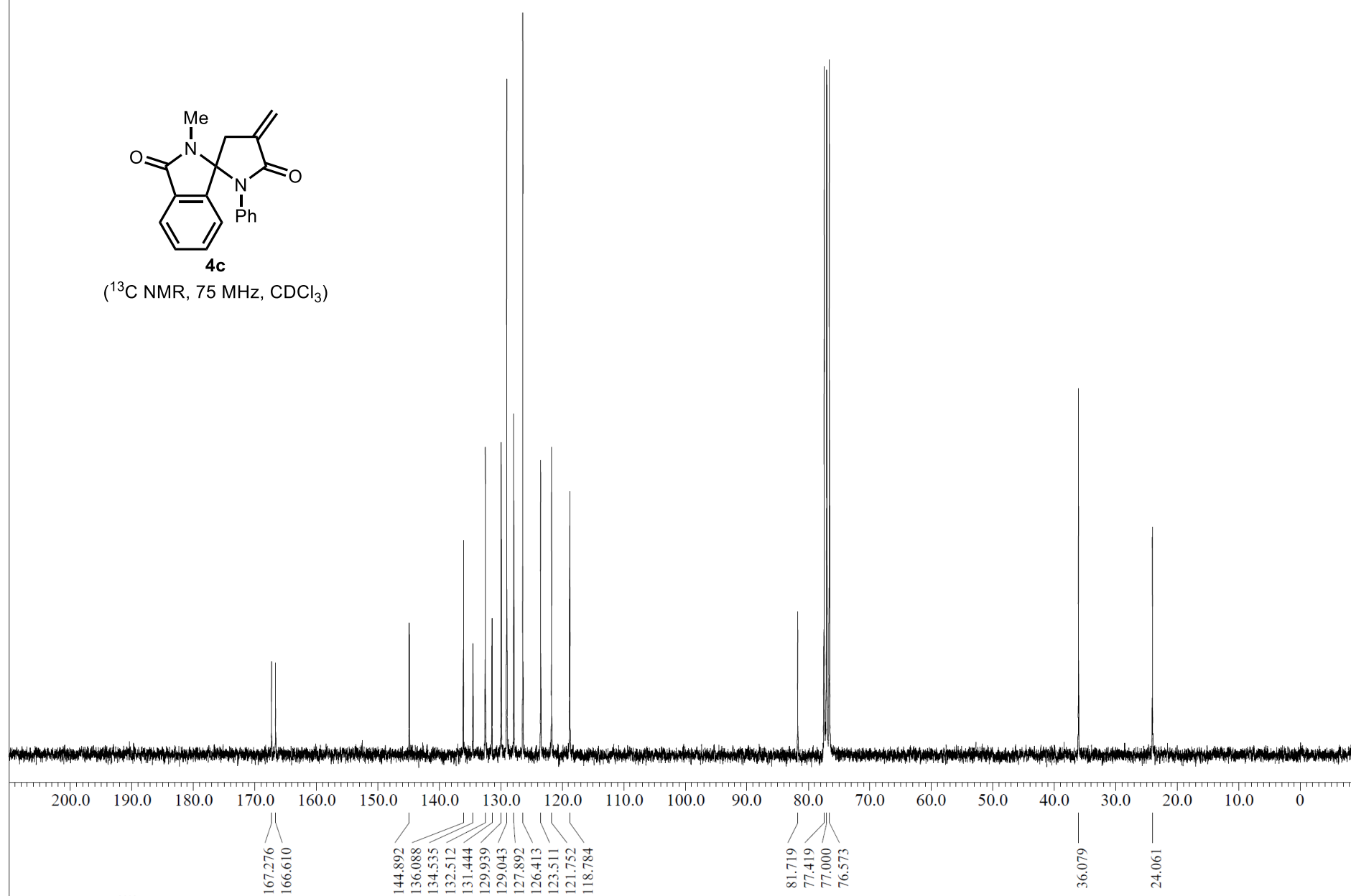
(^1H NMR, 300 MHz, CDCl_3)



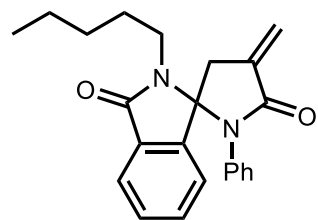


4c

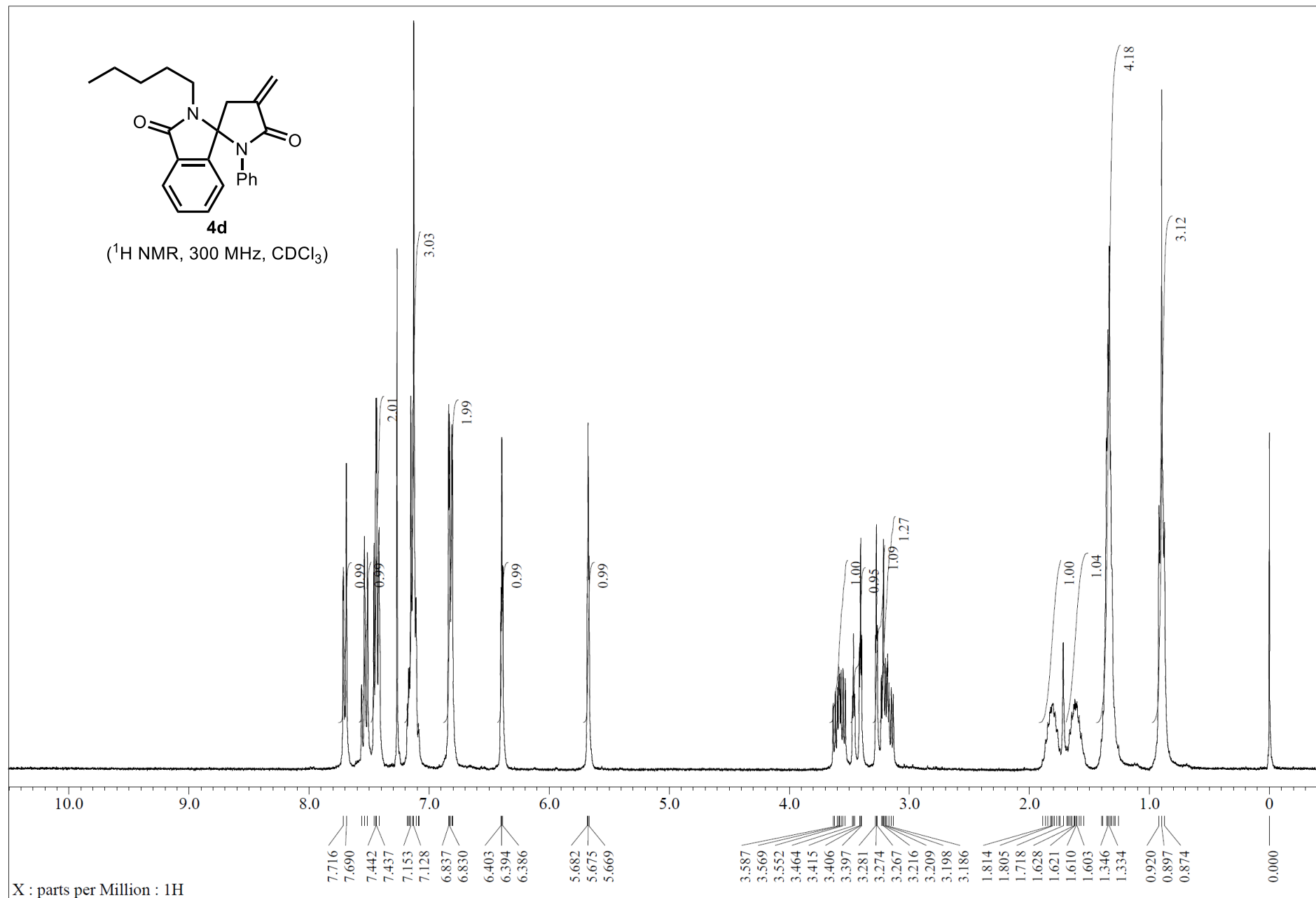
(^{13}C NMR, 75 MHz, CDCl_3)



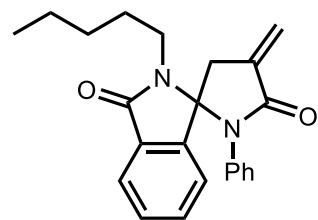
X : parts per Million : ^{13}C



4d
(¹H NMR, 300 MHz, CDCl₃)

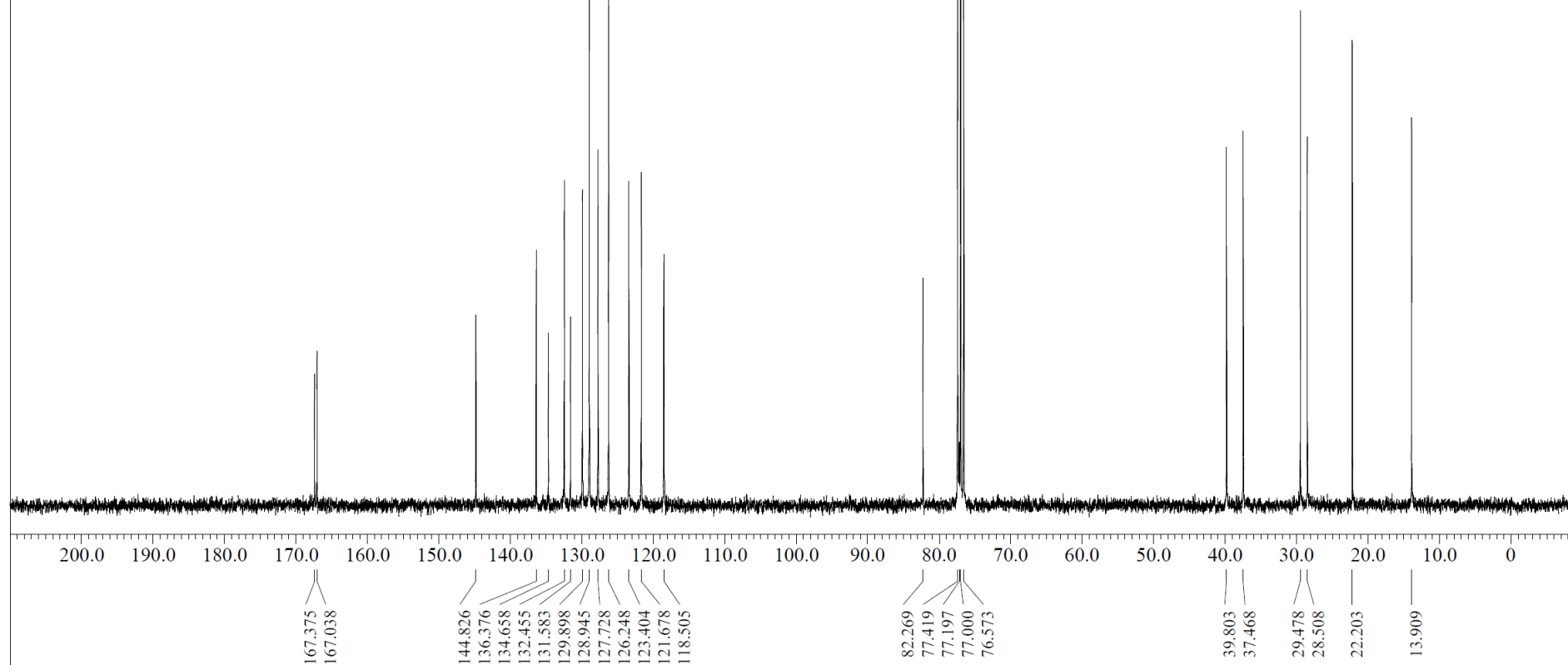


X : parts per Million : 1H

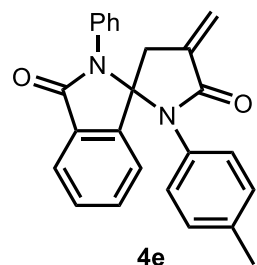


4d

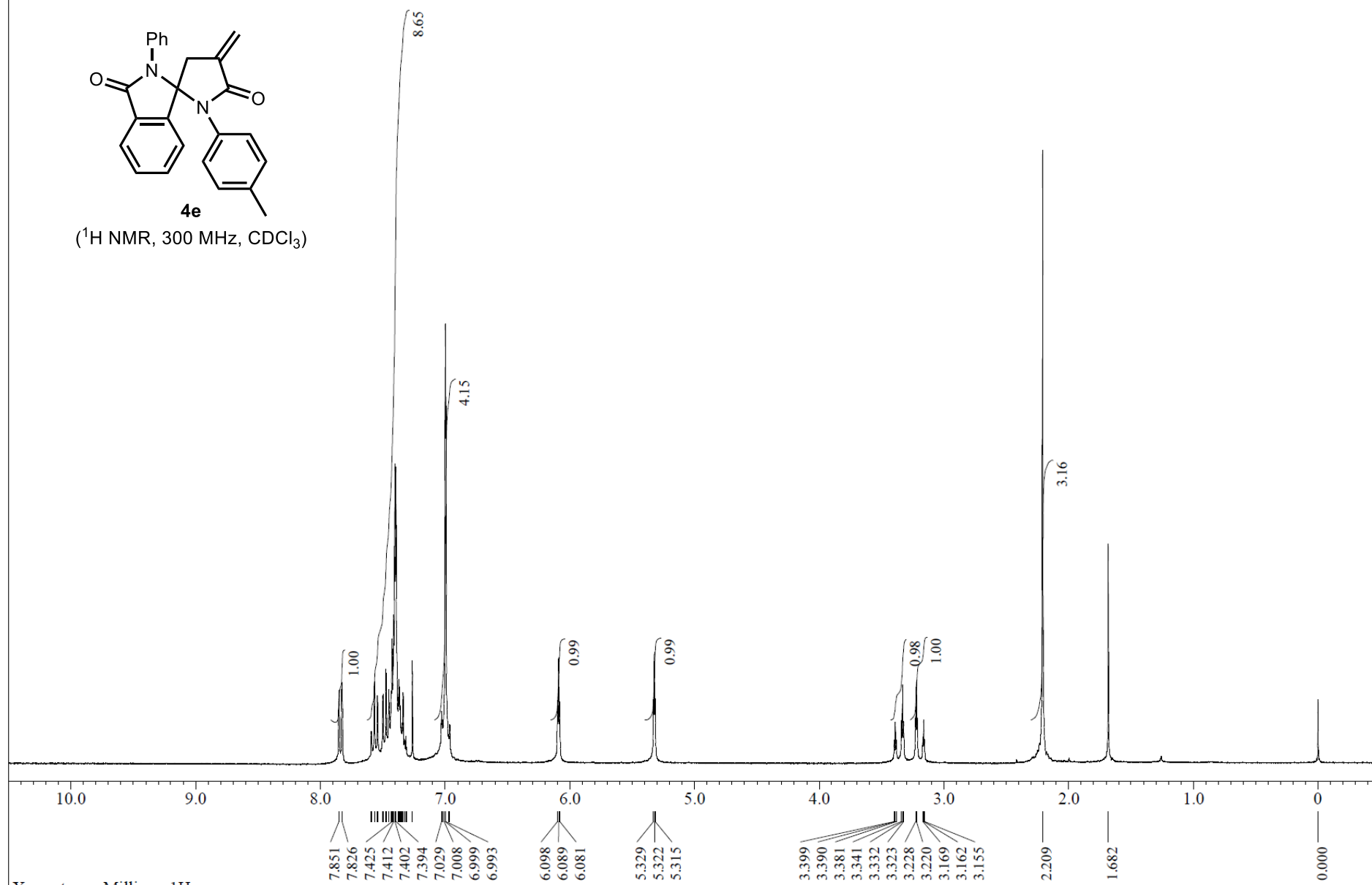
(^{13}C NMR, 75 MHz, CDCl_3)



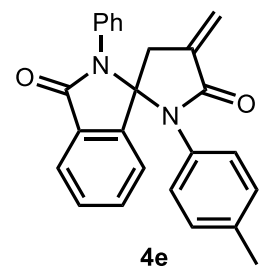
X : parts per Million : ^{13}C



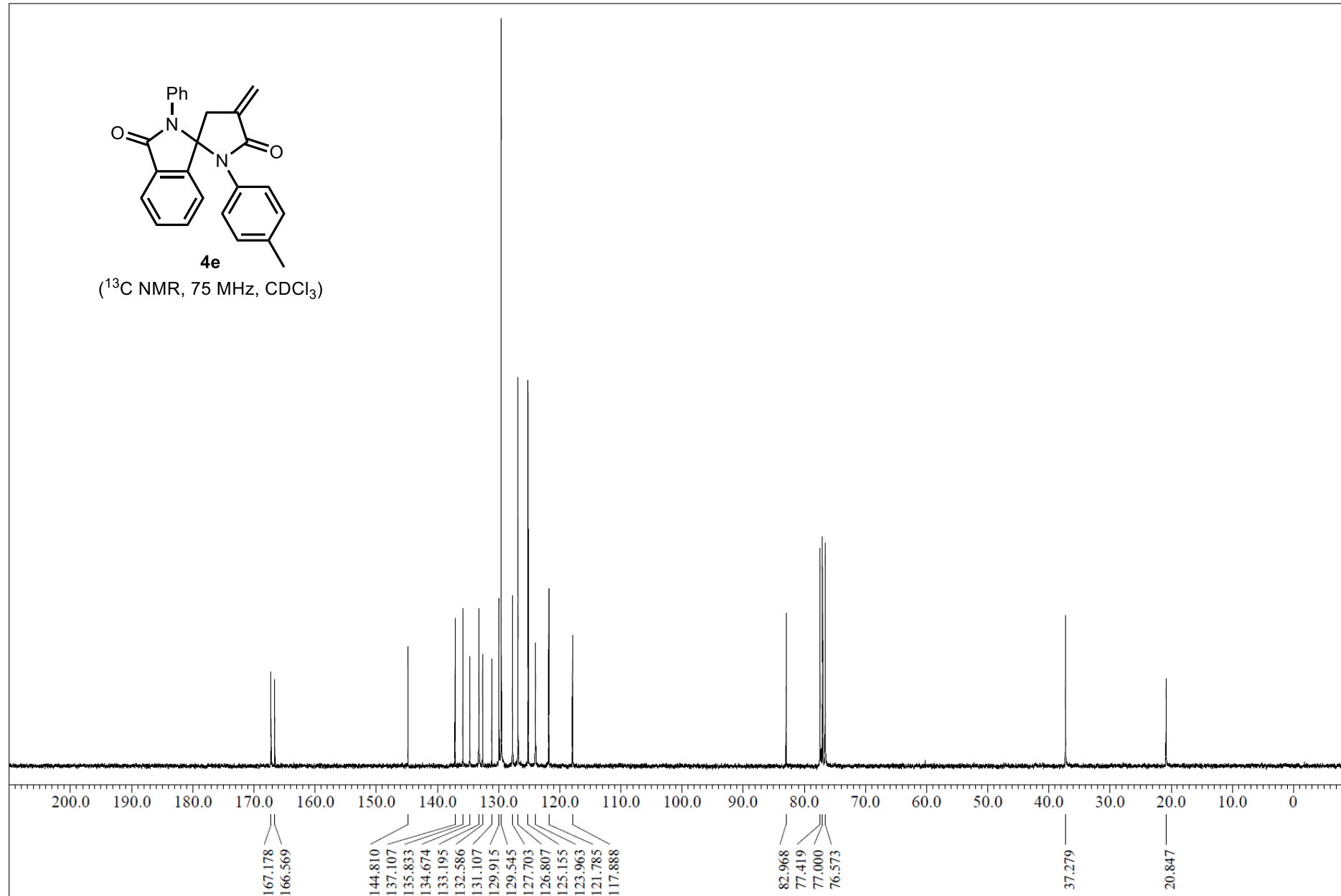
(^1H NMR, 300 MHz, CDCl_3)



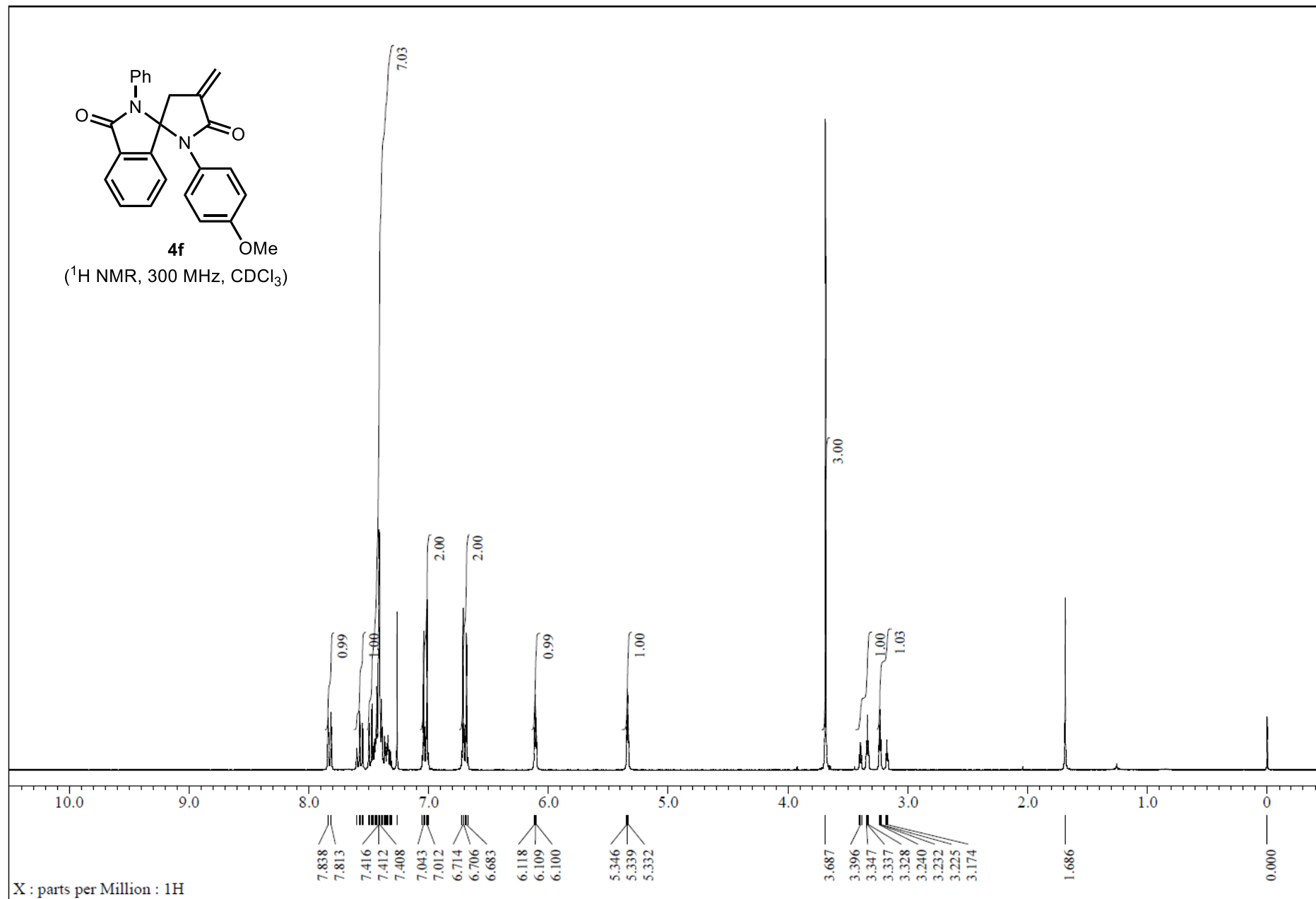
X : parts per Million : ^1H

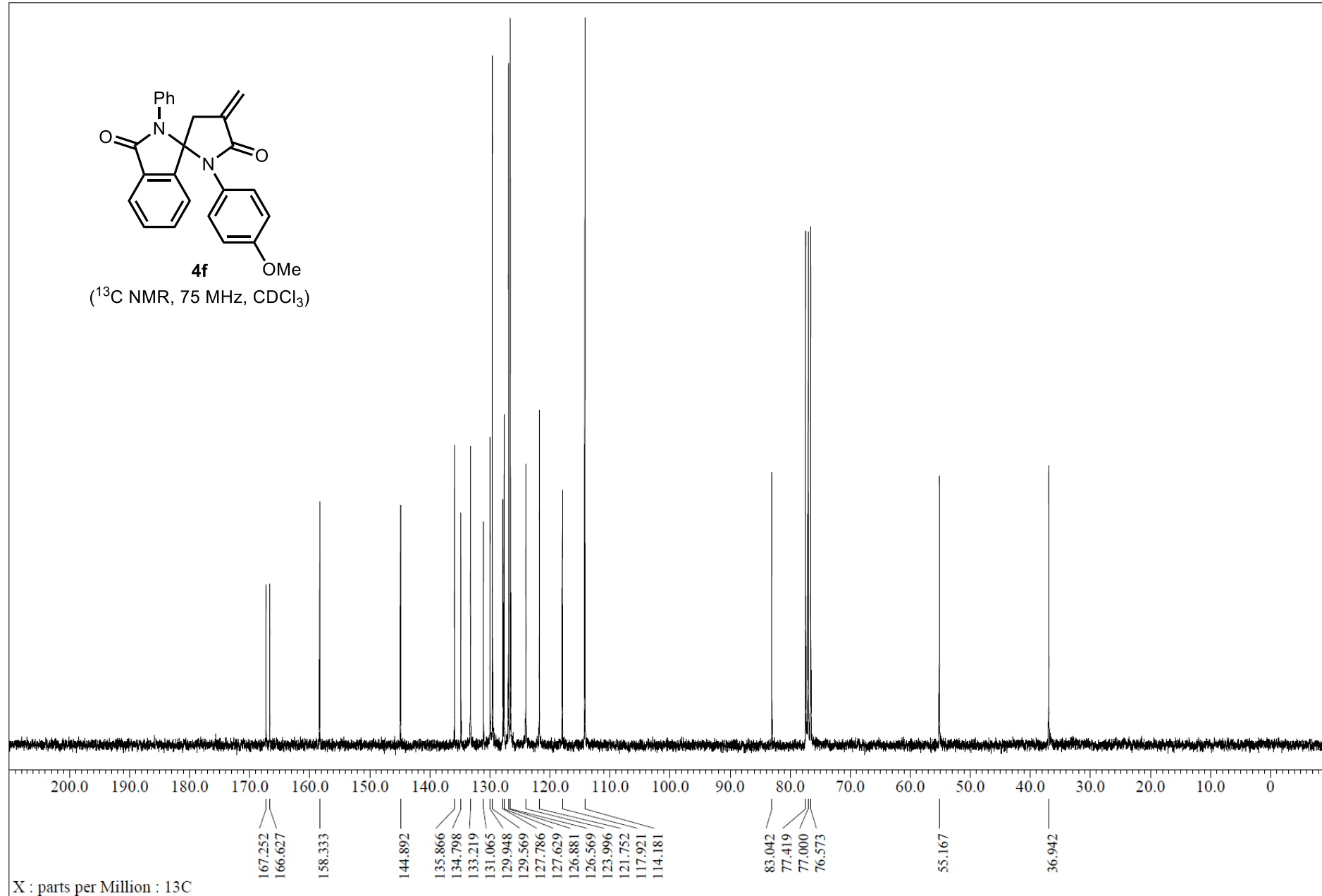
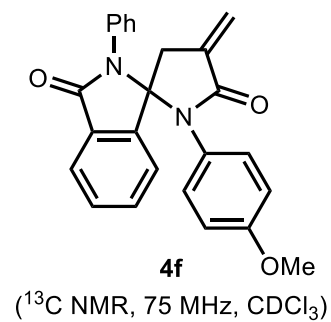


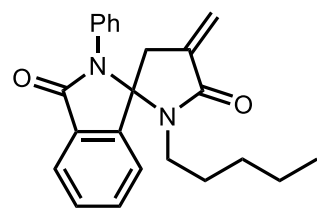
(^{13}C NMR, 75 MHz, CDCl_3)



X : parts per Million : ^{13}C

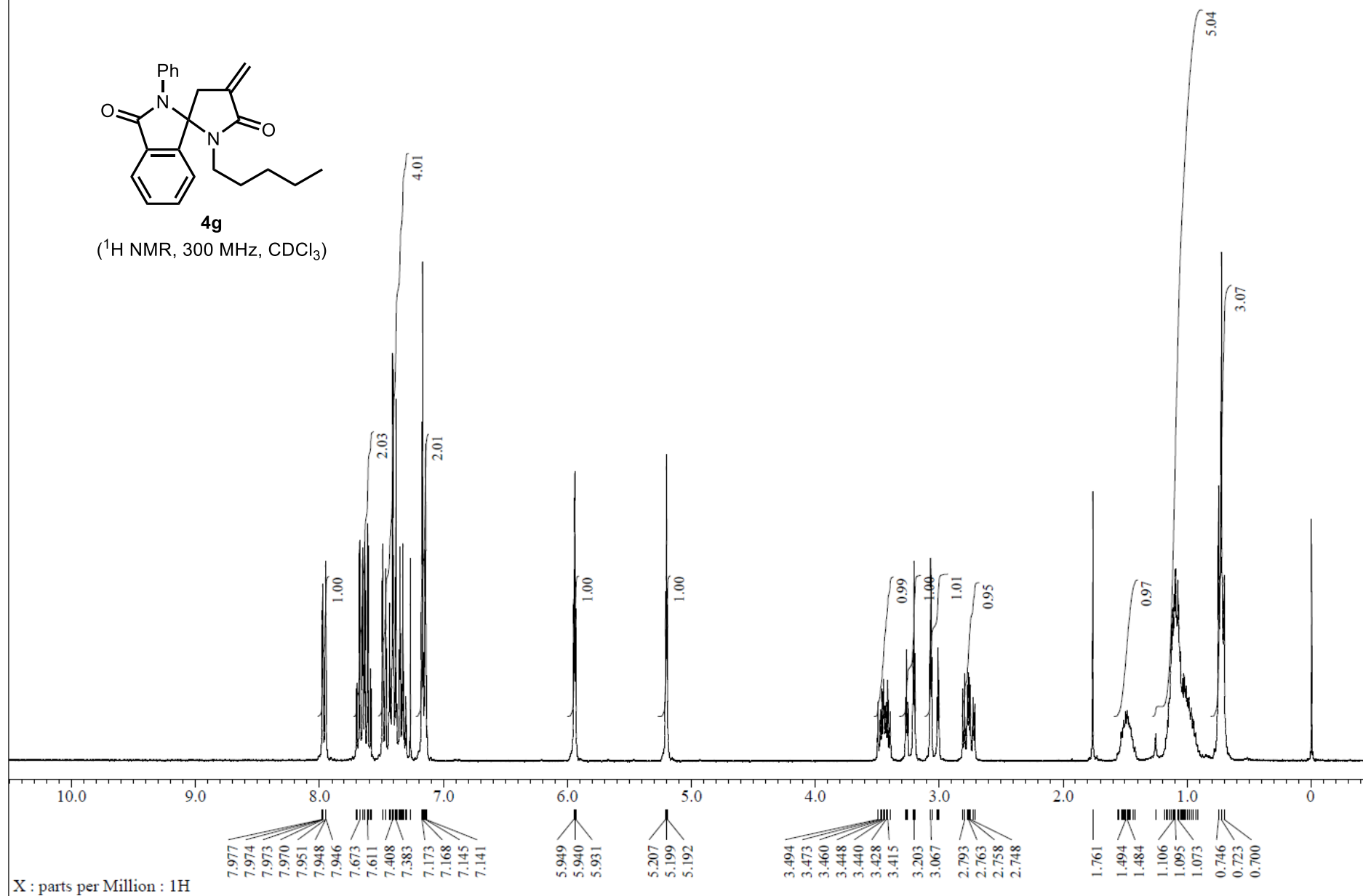




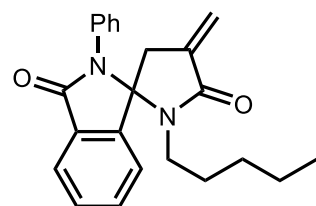


4g

(^1H NMR, 300 MHz, CDCl_3)

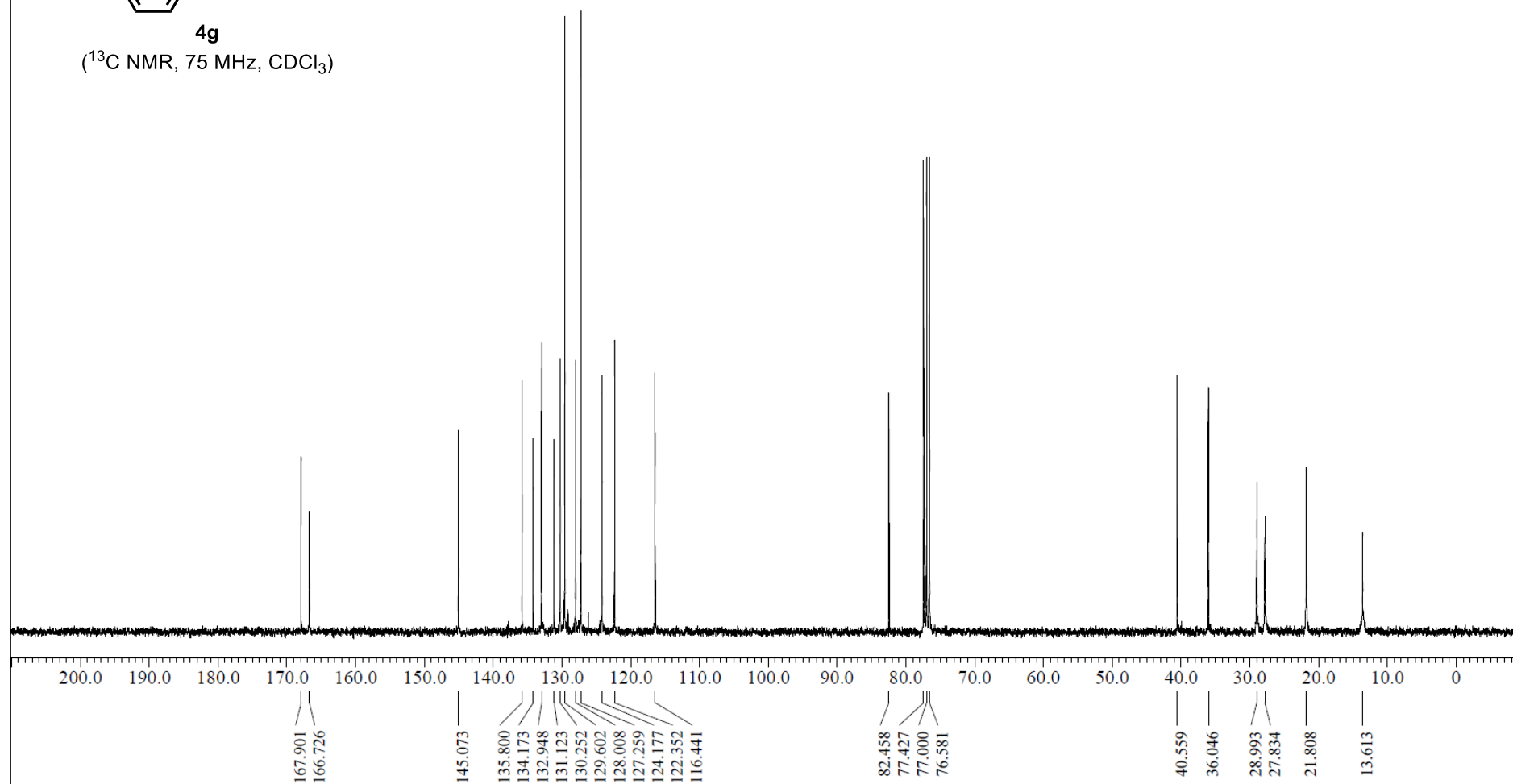


X : parts per Million : ^1H

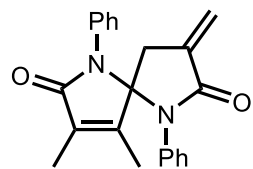


4g

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

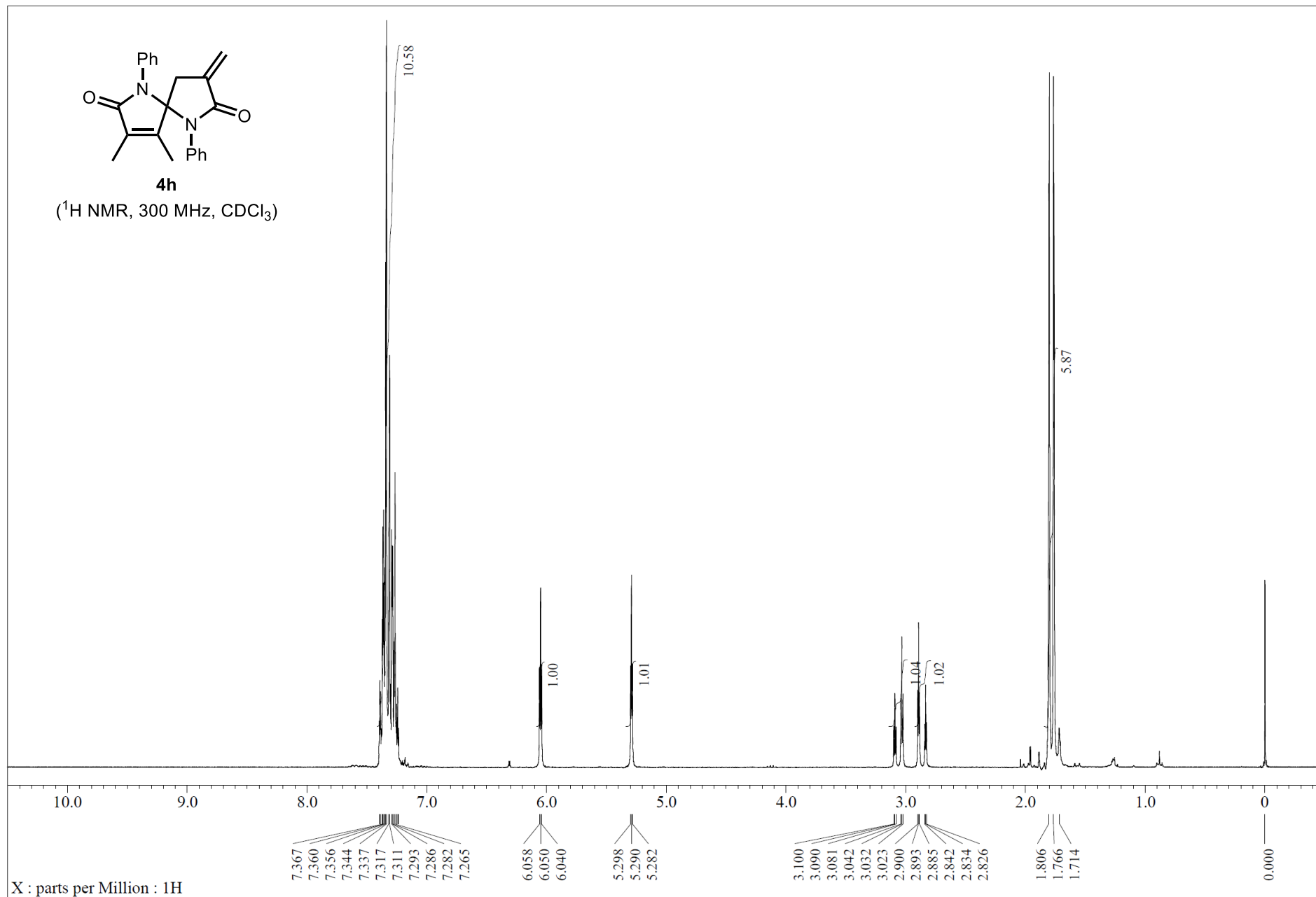


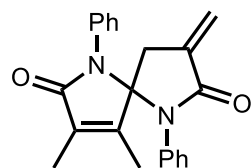
X : parts per Million : ^{13}C



4h

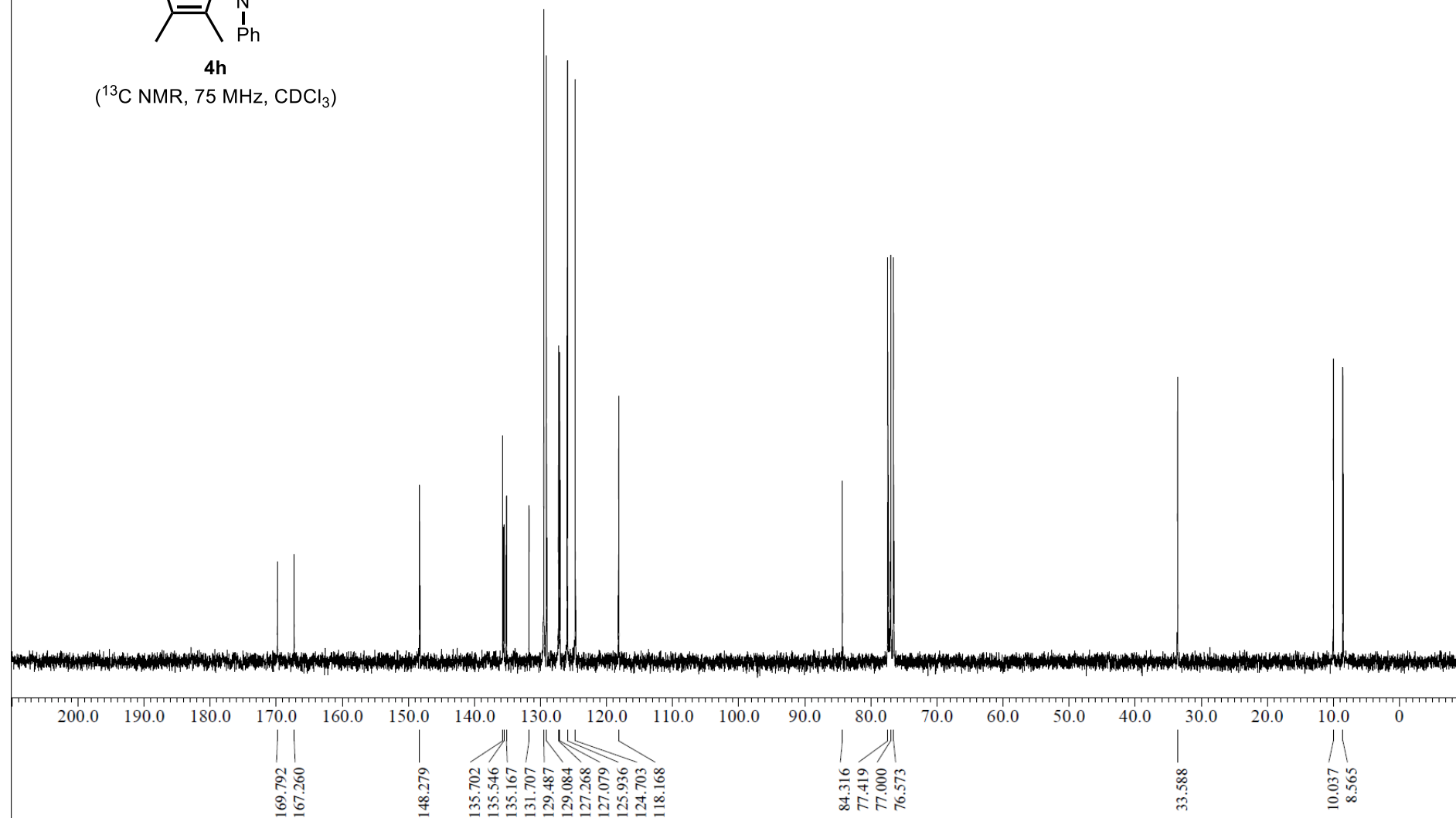
(^1H NMR, 300 MHz, CDCl_3)



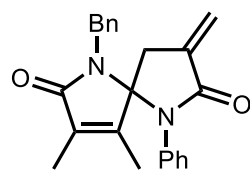


4h

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

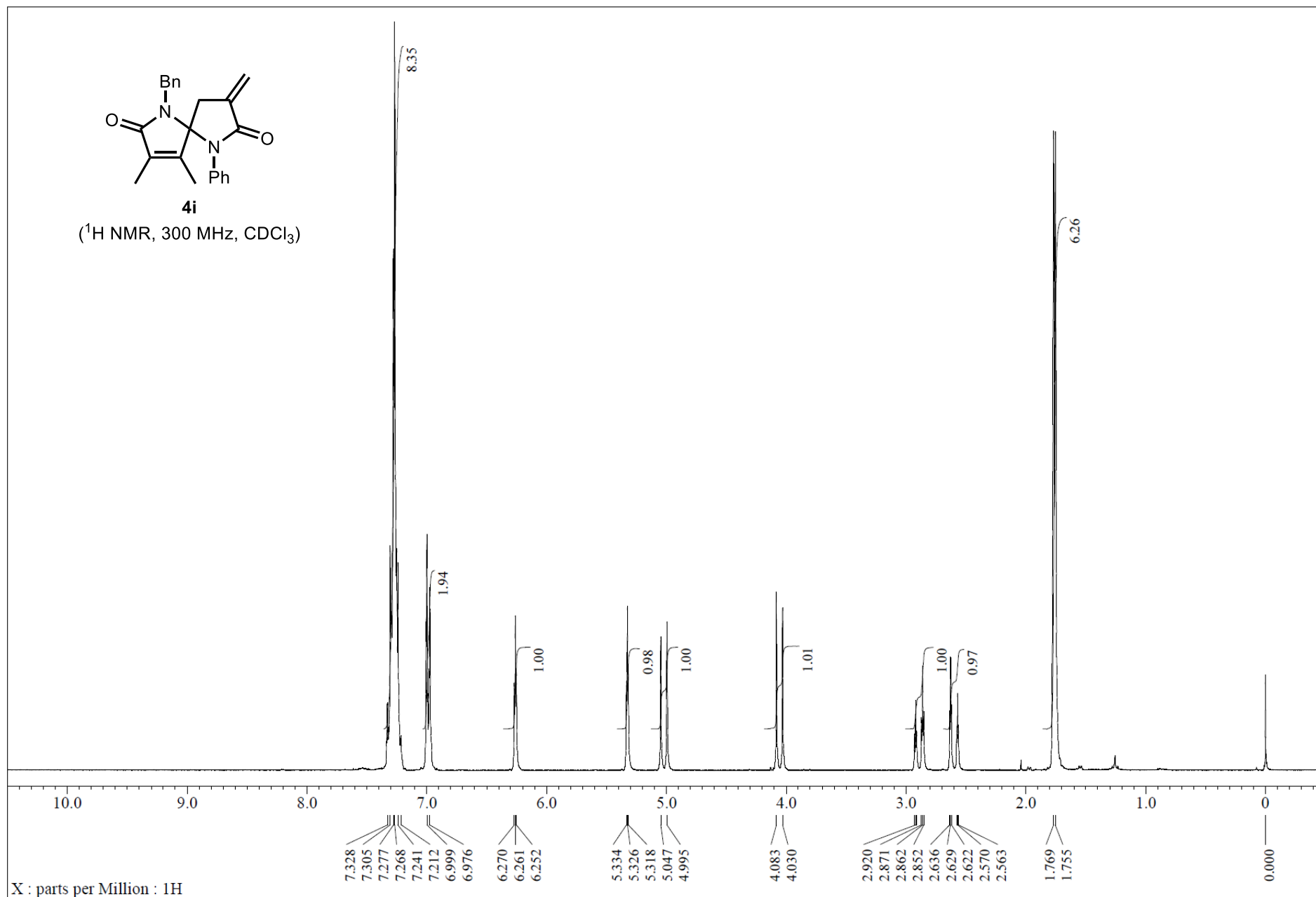


X : parts per Million : ^{13}C

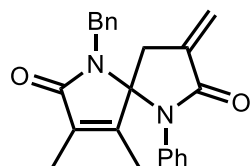


4i

(¹H NMR, 300 MHz, CDCl₃)

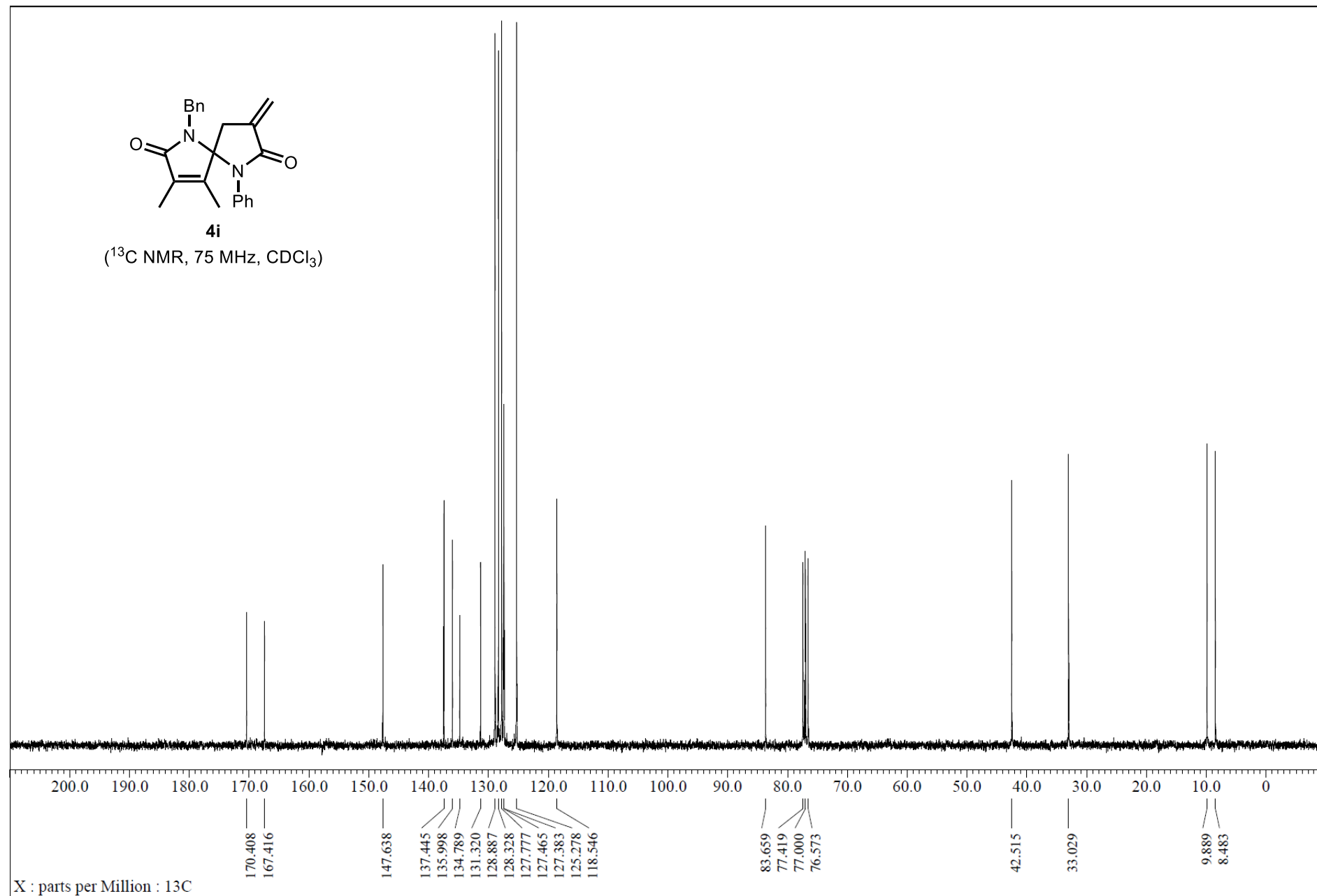


X : parts per Million : 1H

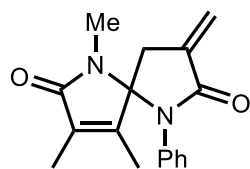


4i

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

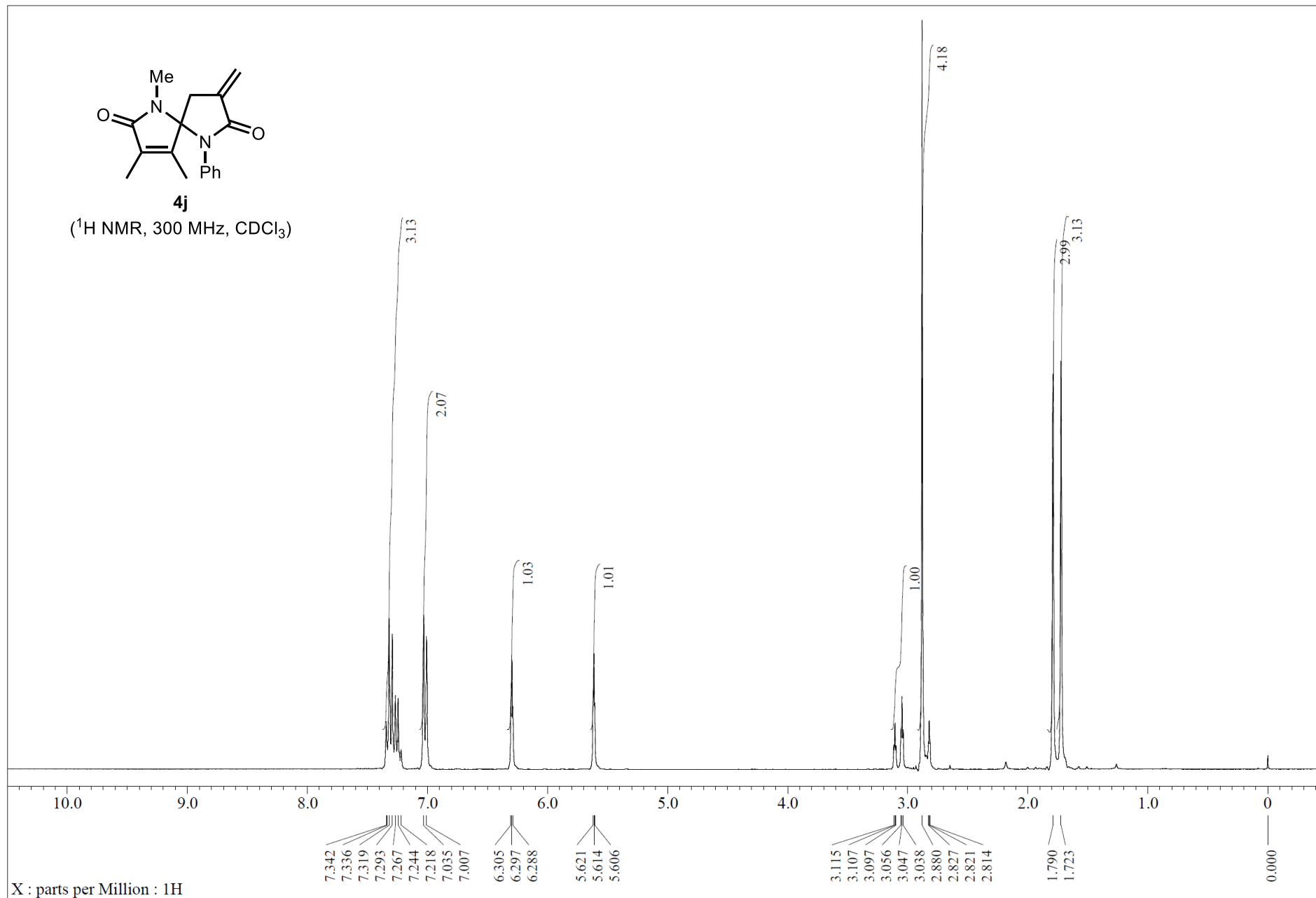


X : parts per Million : ^{13}C

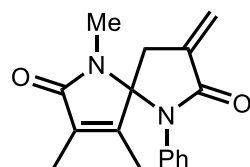


4j

(^1H NMR, 300 MHz, CDCl_3)

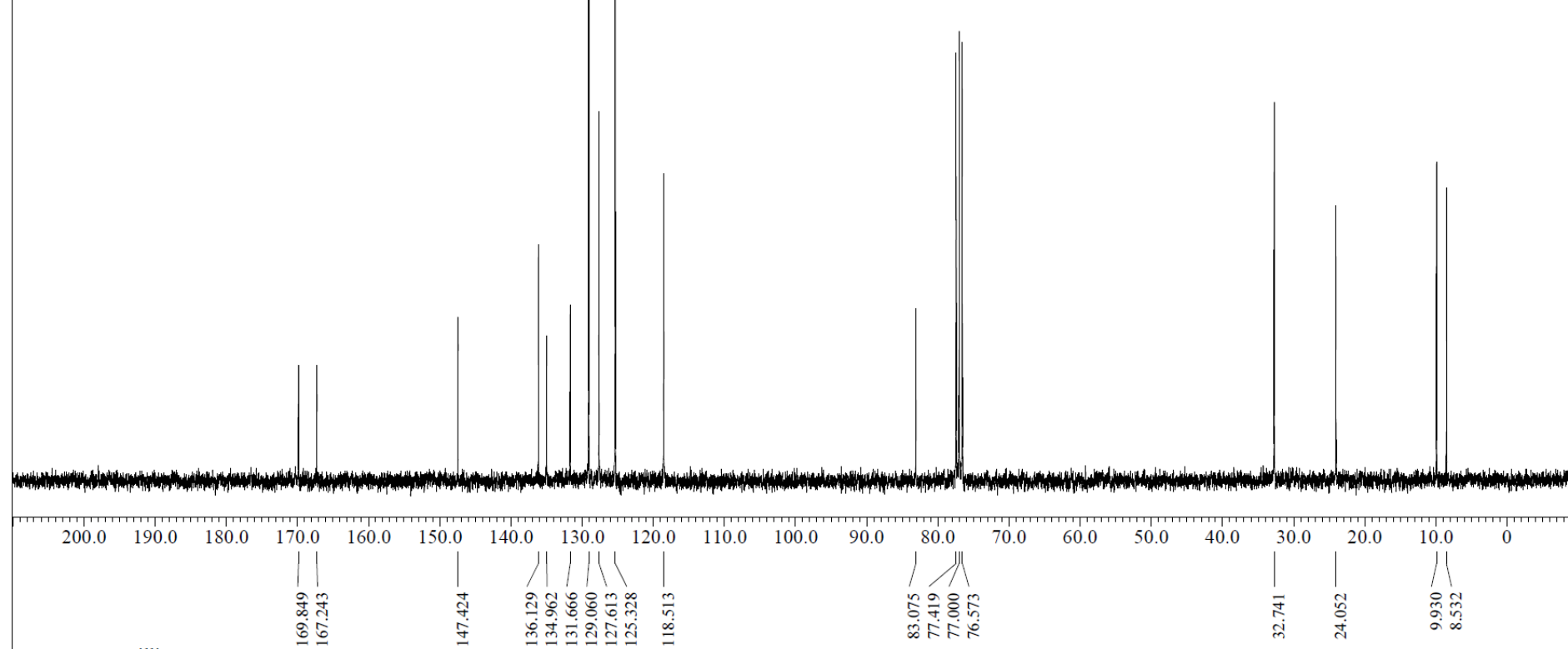


X : parts per Million : ^1H

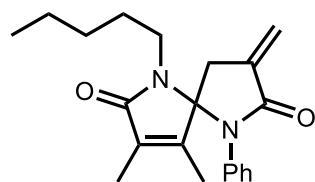


4j

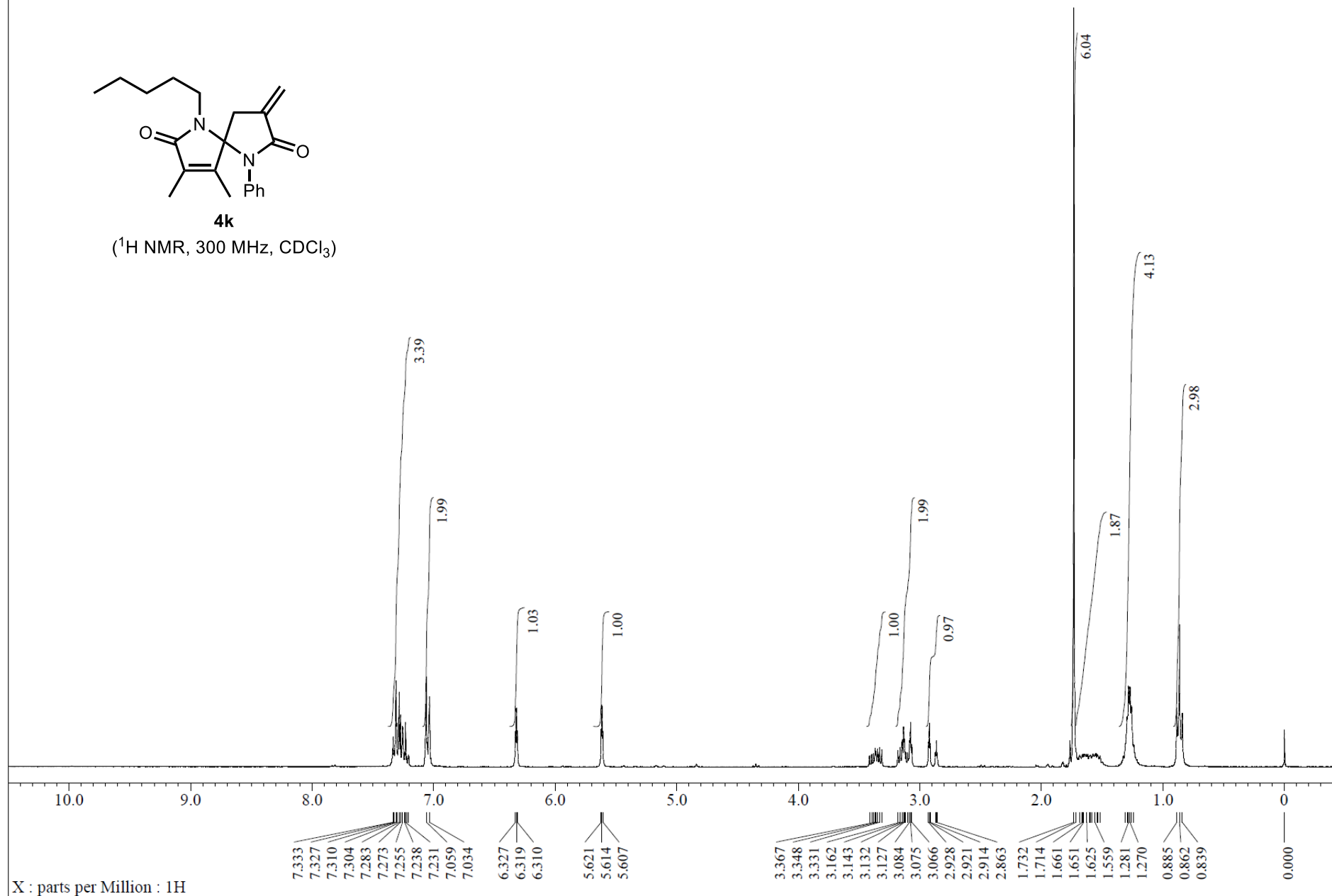
(^{13}C NMR, 75 MHz, CDCl_3)

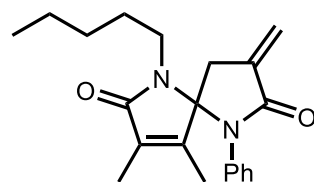


X : parts per Million : ^{13}C



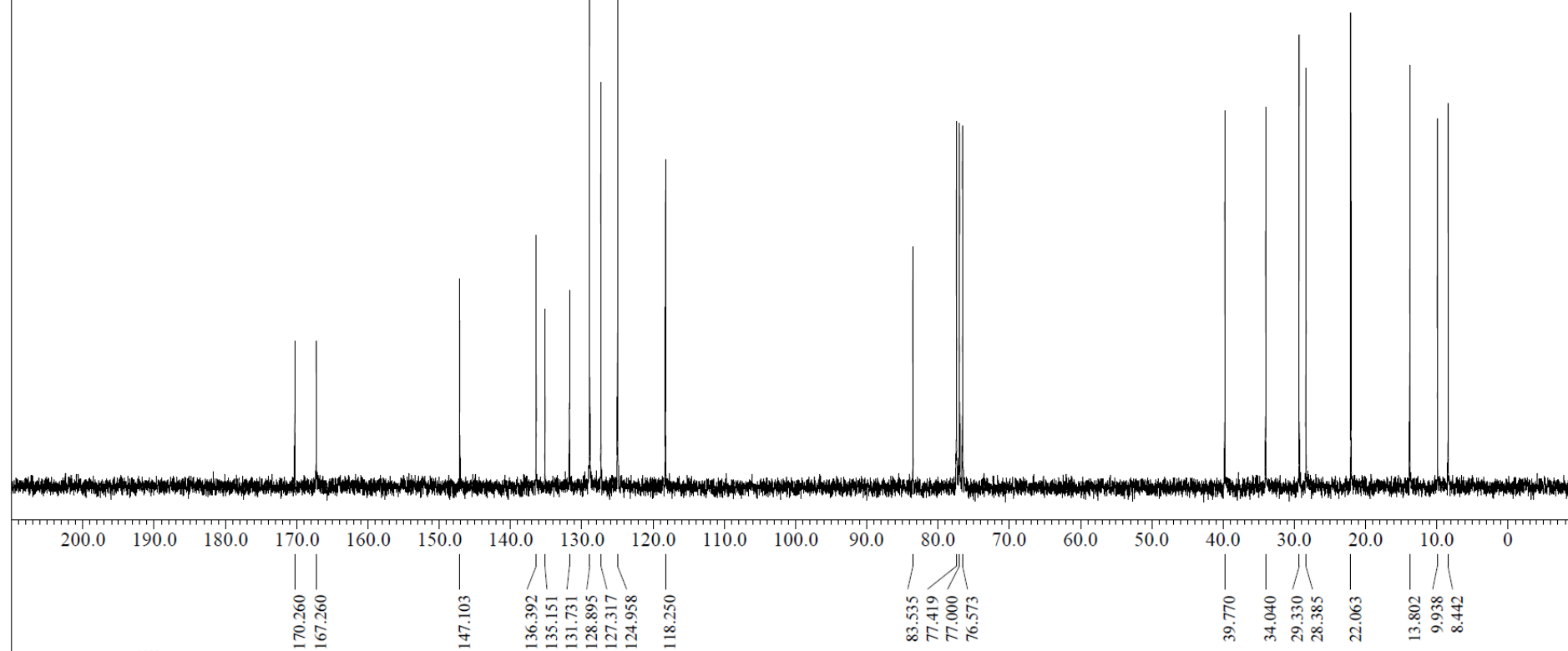
4k
 (¹H NMR, 300 MHz, CDCl₃)



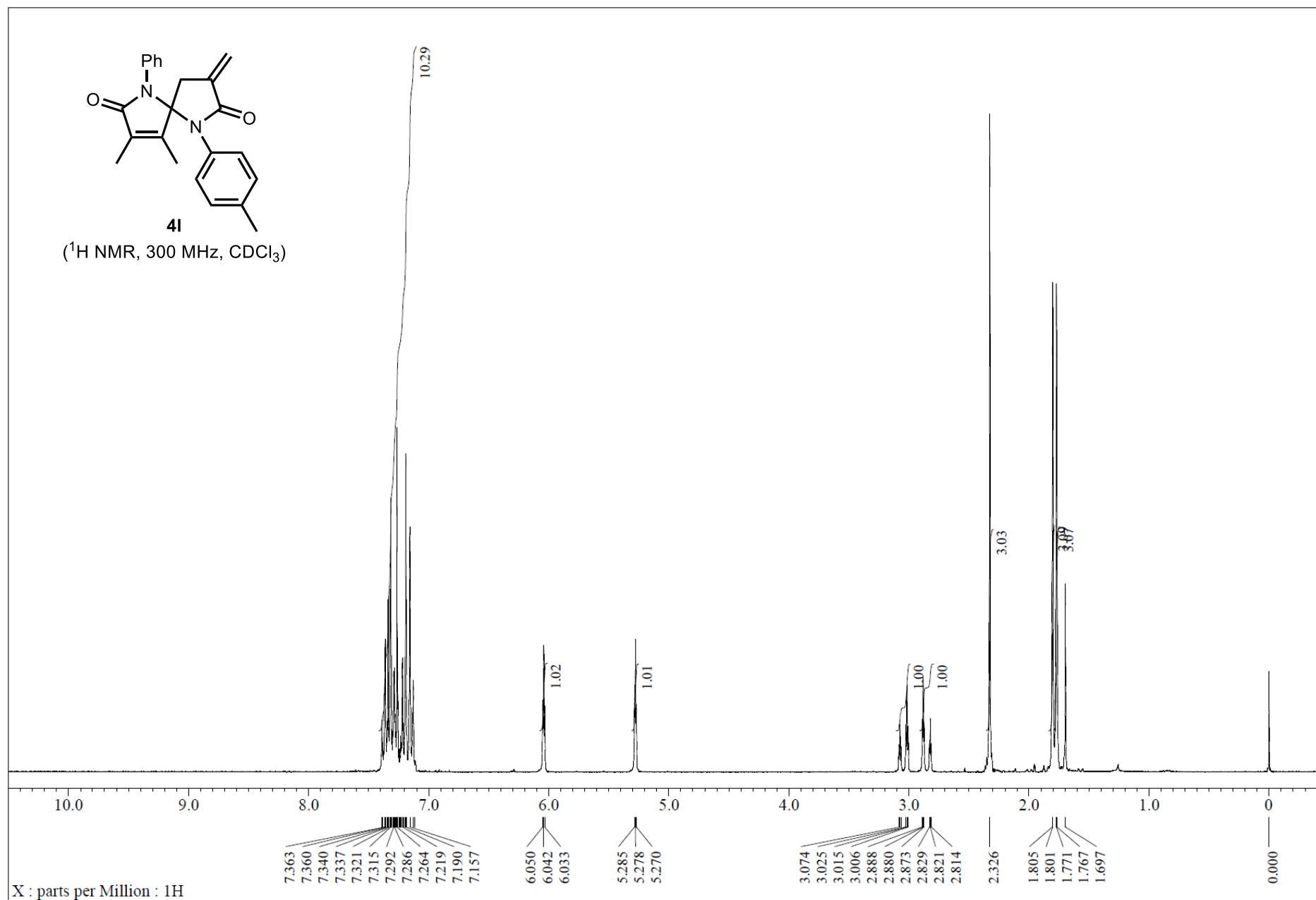


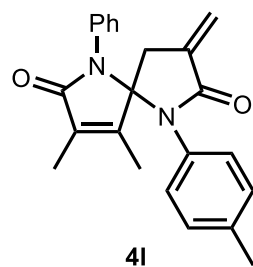
4k

(¹³C NMR, 75 MHz, CDCl₃)

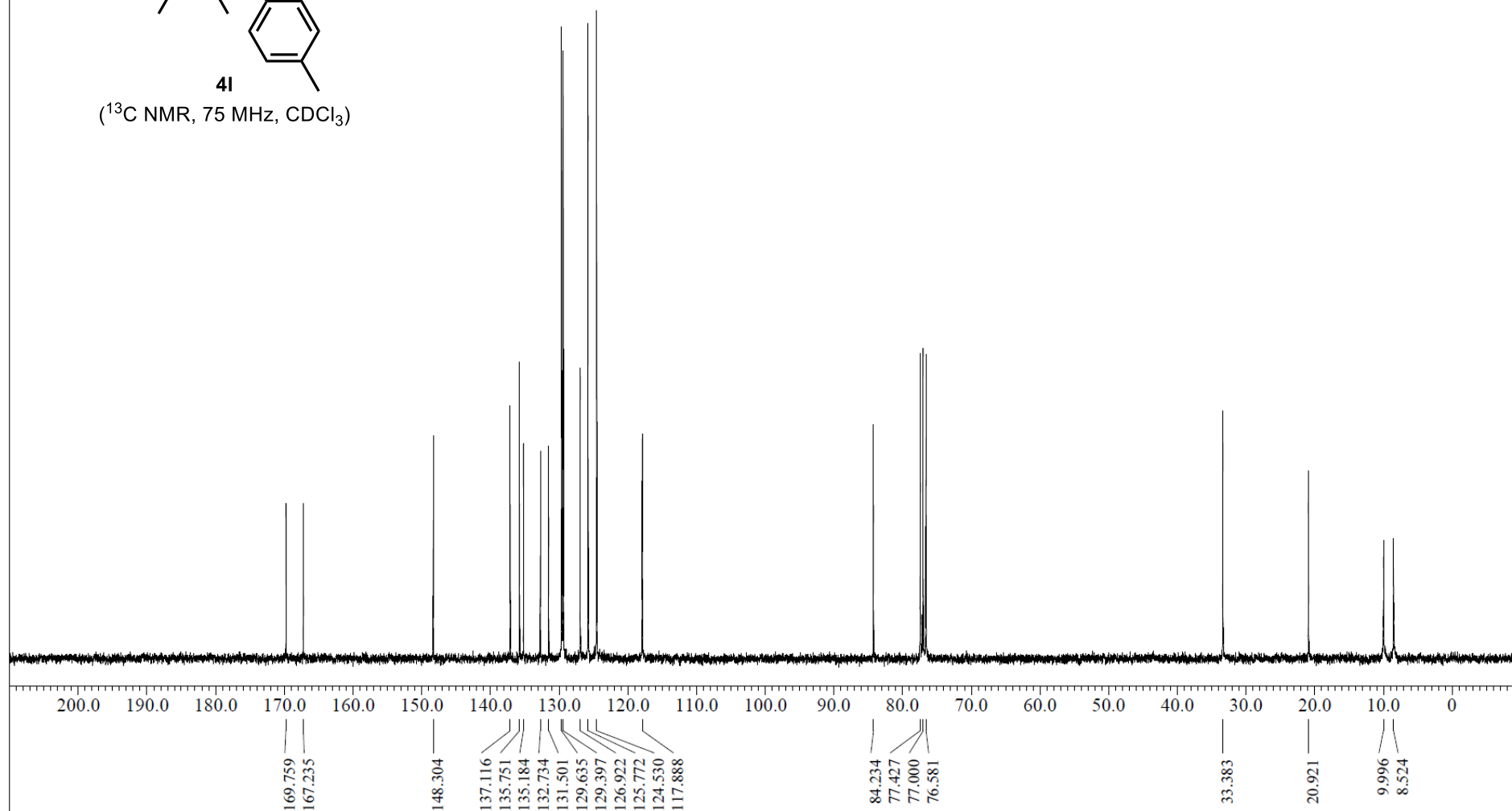


X : parts per Million : 13C

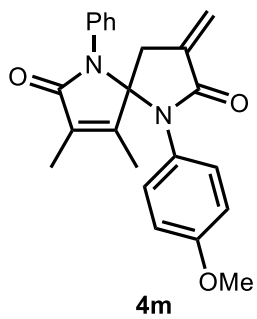




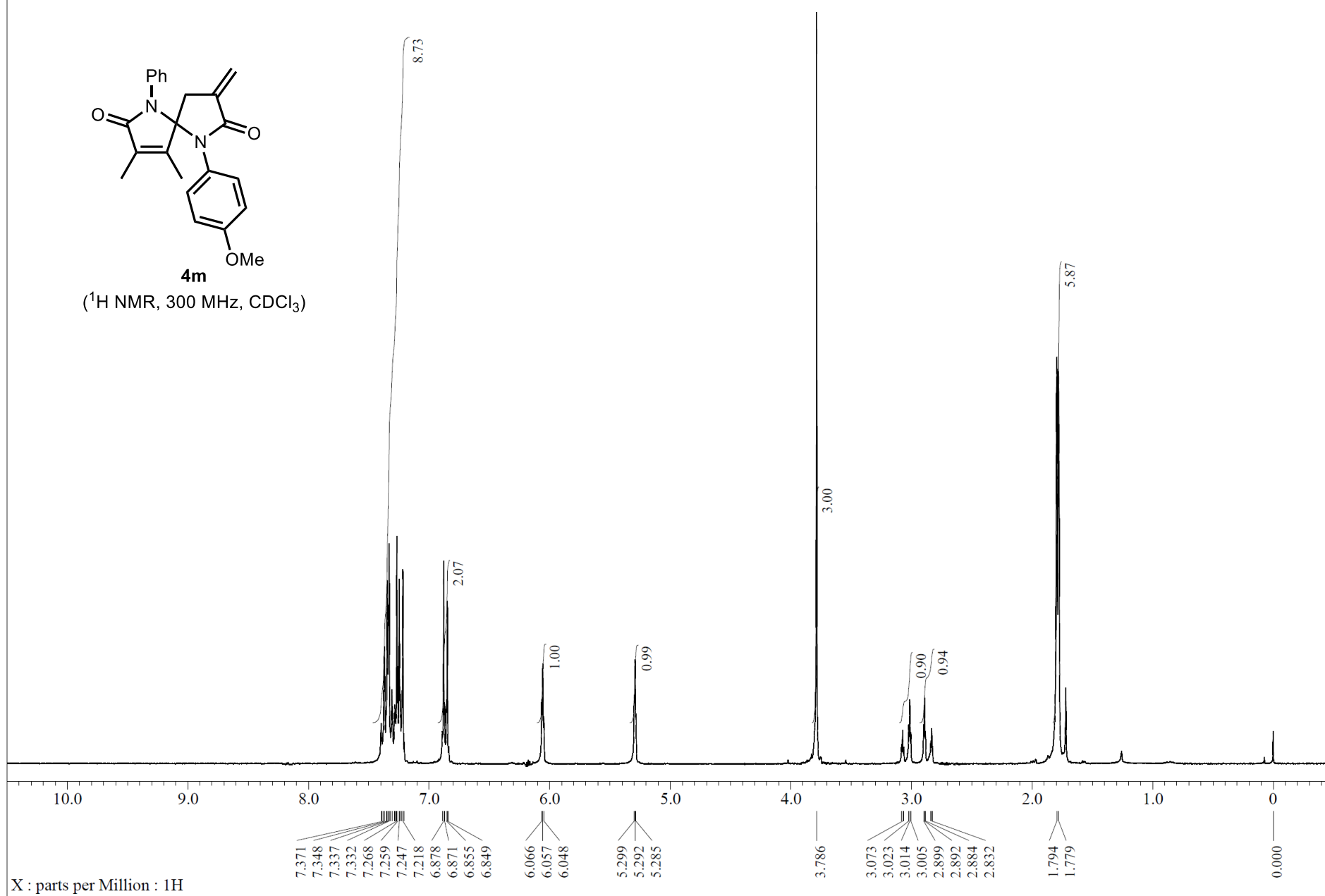
(^{13}C NMR, 75 MHz, CDCl_3)



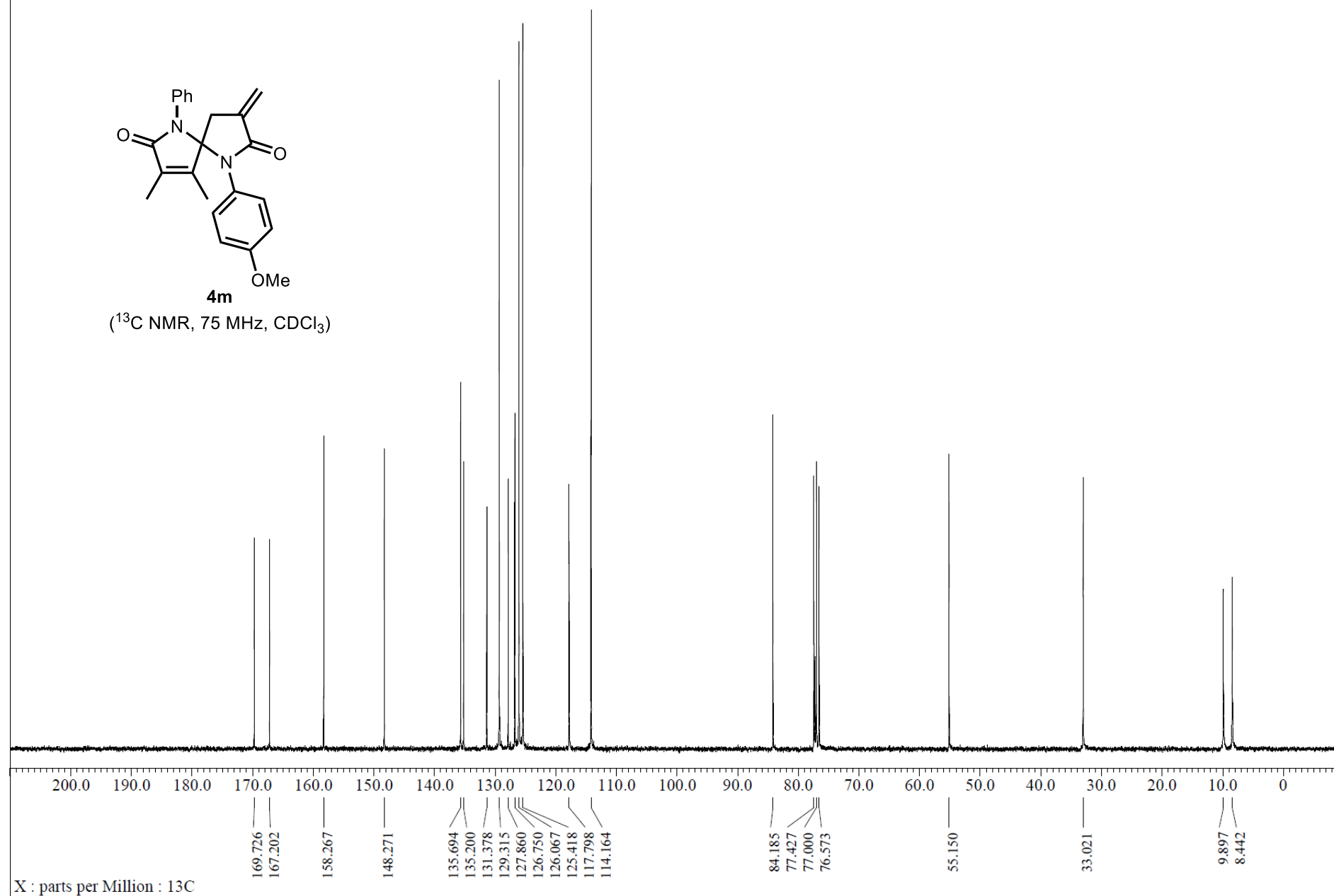
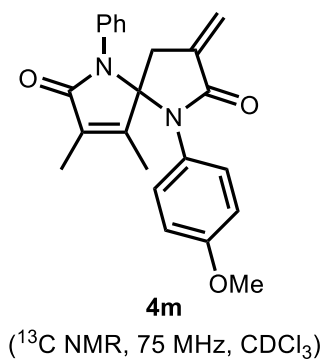
X : parts per Million : ^{13}C

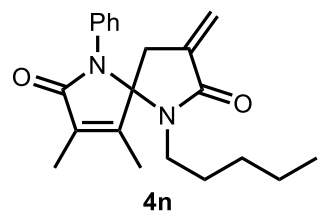


(¹H NMR, 300 MHz, CDCl₃)

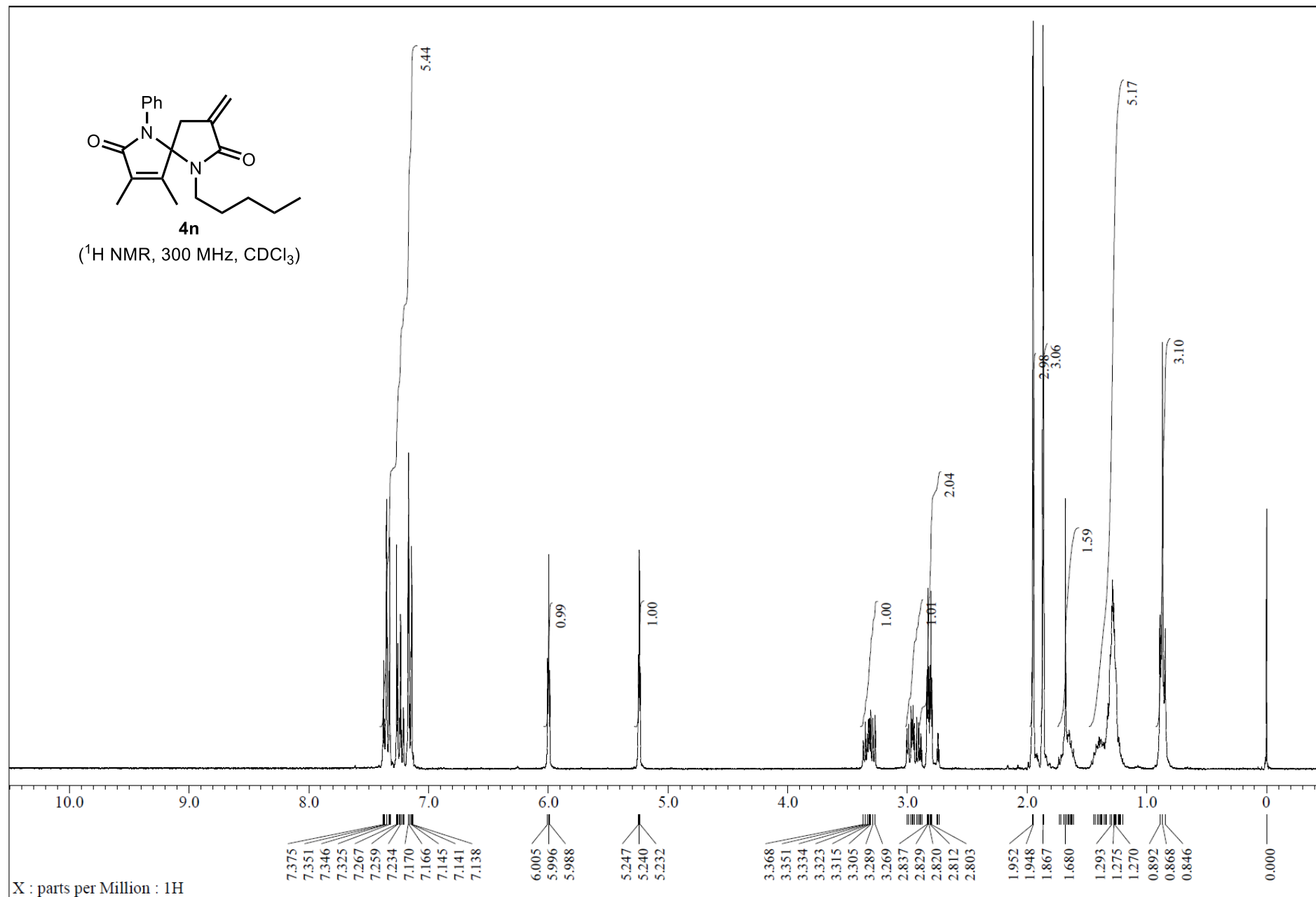


X : parts per Million : 1H

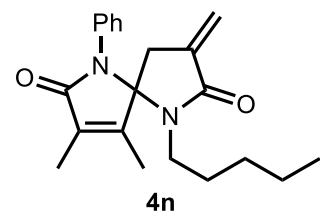




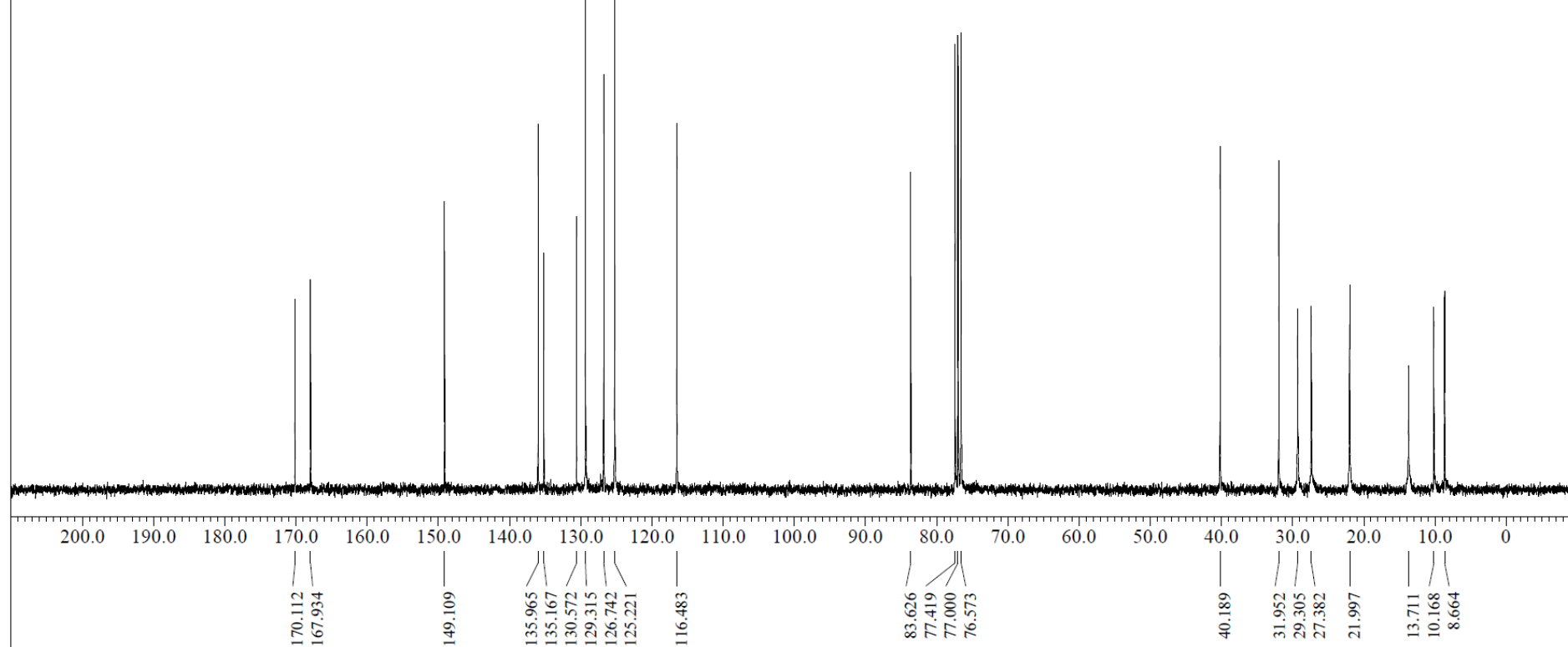
(¹H NMR, 300 MHz, CDCl₃)



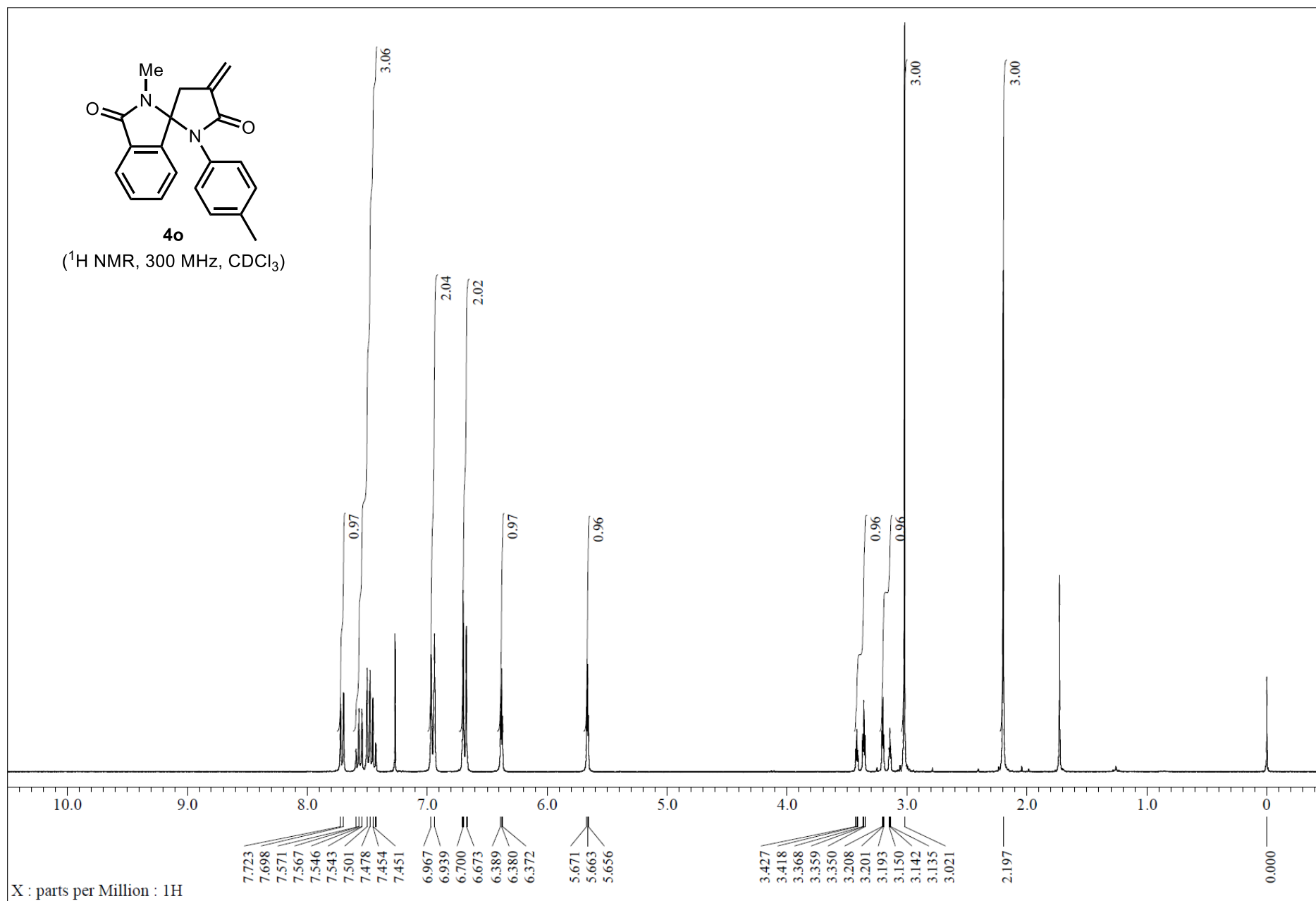
X : parts per Million : 1H

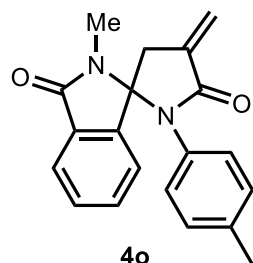


(^{13}C NMR, 75 MHz, CDCl_3)

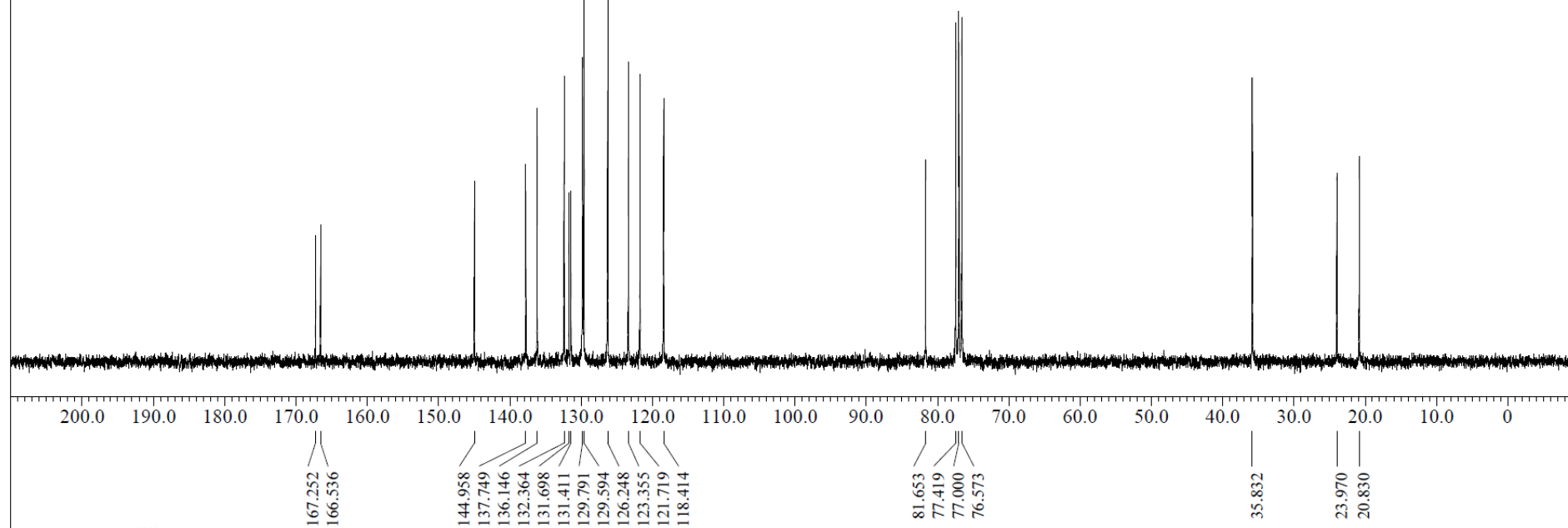


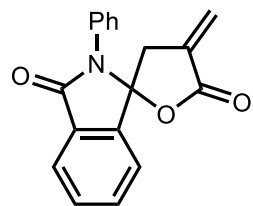
X : parts per Million : ^{13}C





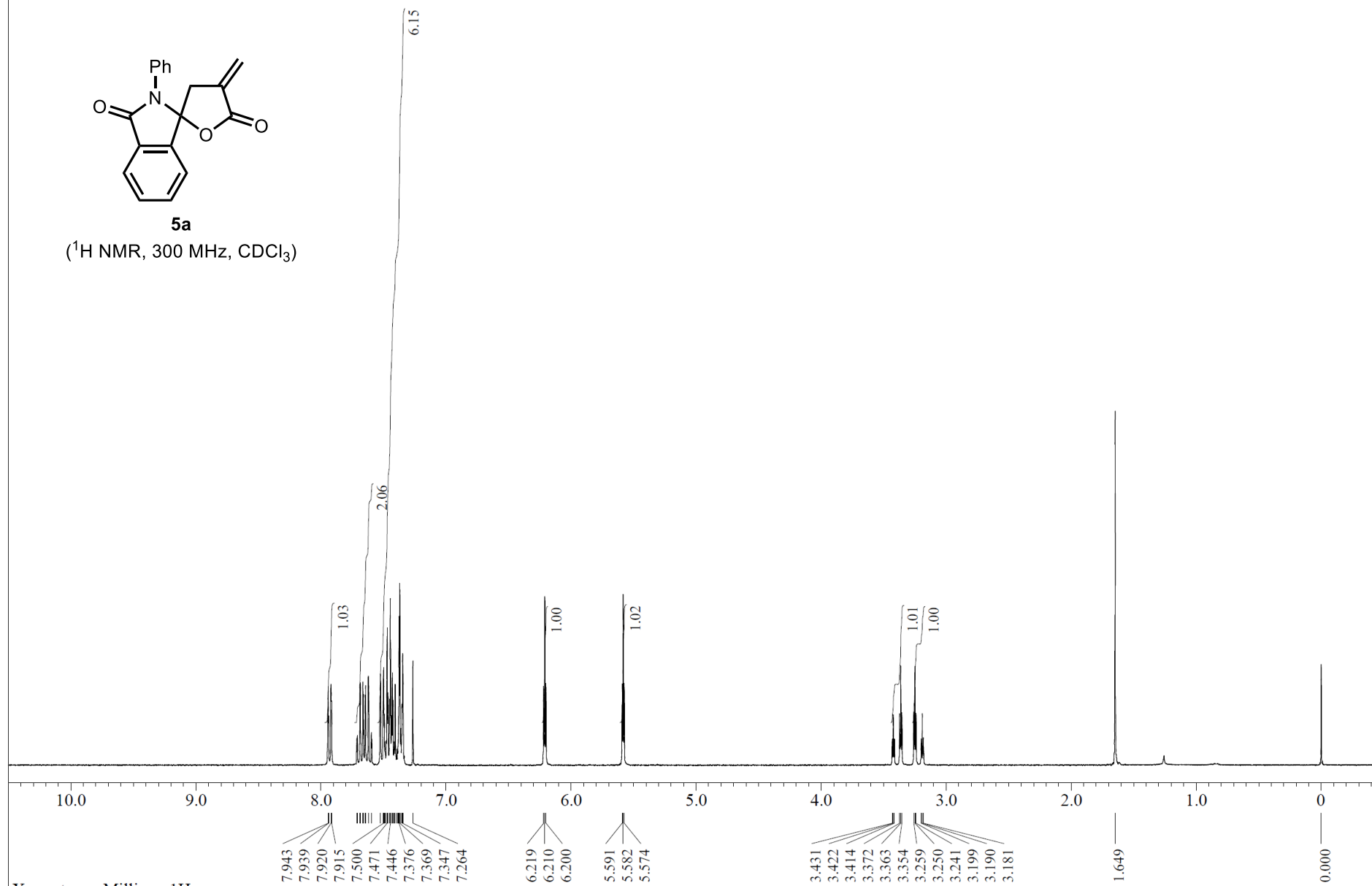
(^{13}C NMR, 75 MHz, CDCl_3)



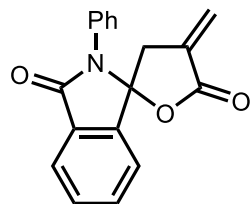


5a

(¹H NMR, 300 MHz, CDCl₃)

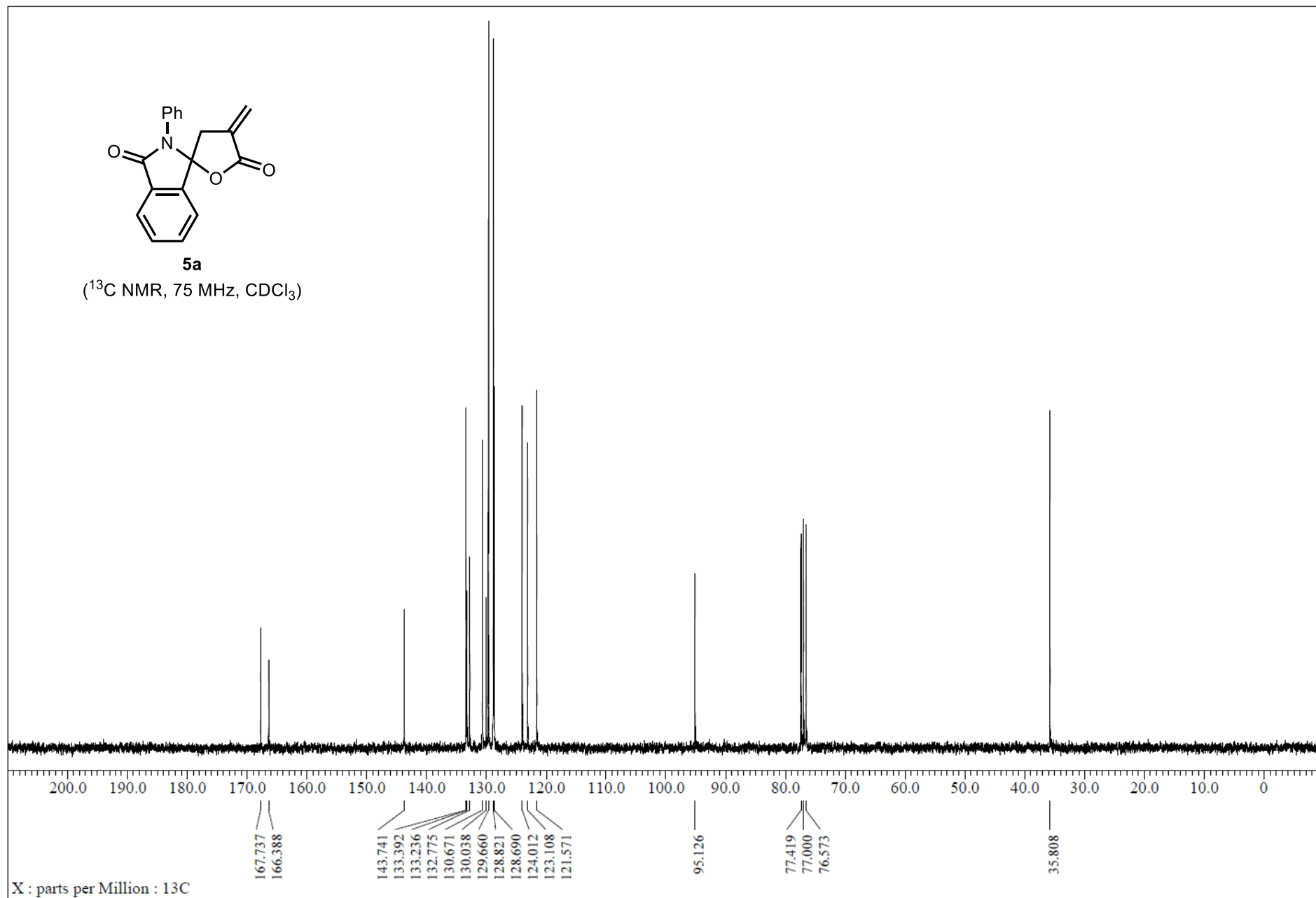


X : parts per Million : 1H

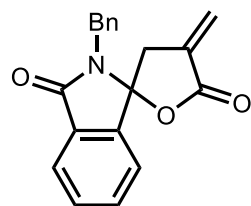


5a

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

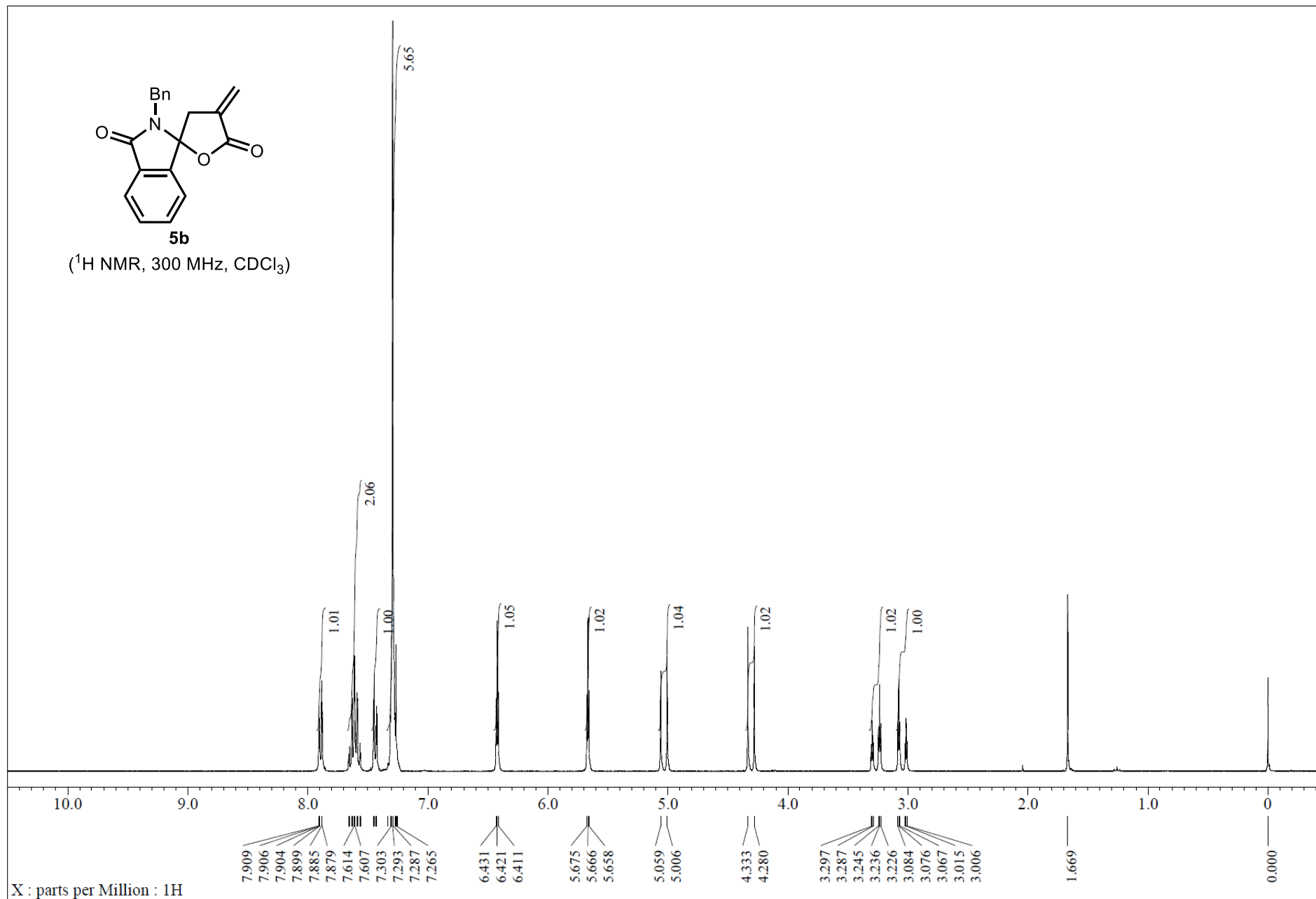


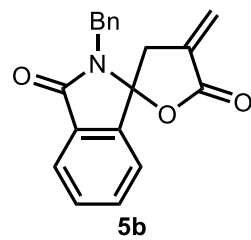
X : parts per Million : ^{13}C



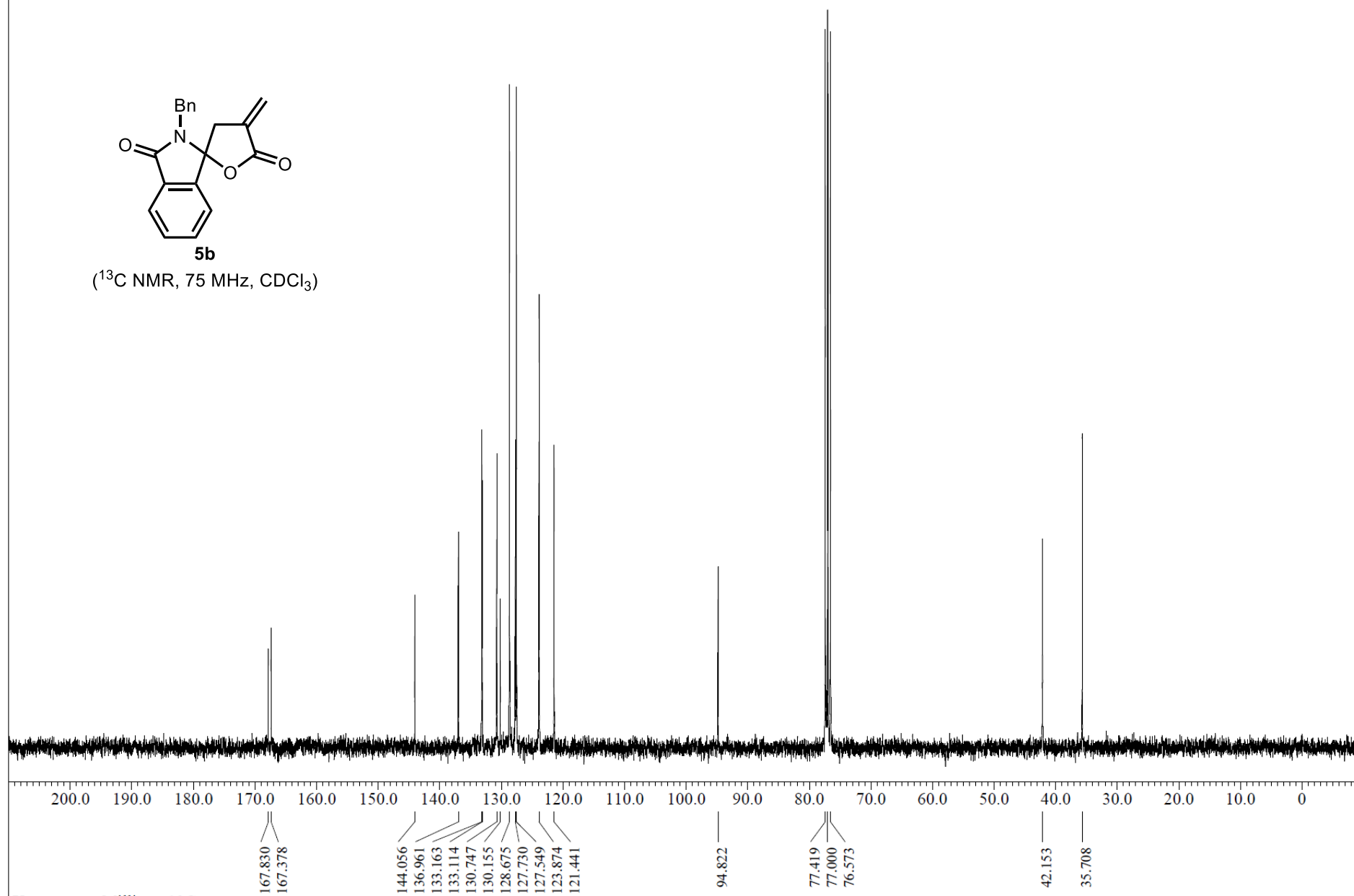
5b

(^1H NMR, 300 MHz, CDCl_3)

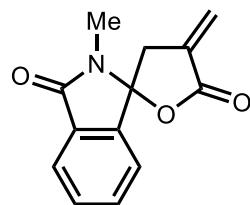




(^{13}C NMR, 75 MHz, CDCl_3)

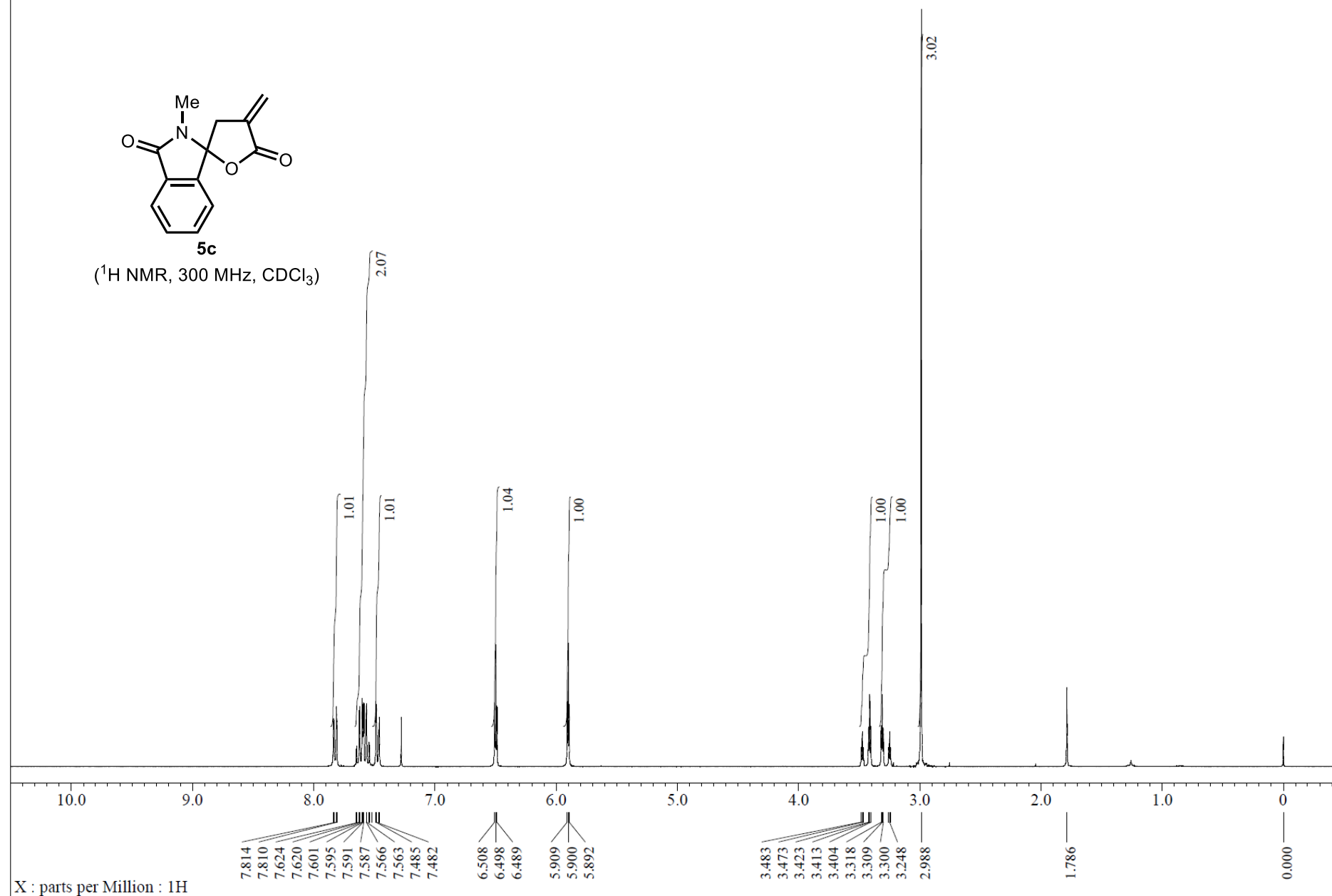


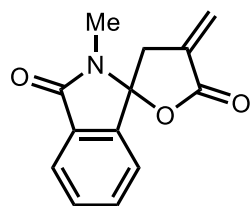
X : parts per Million : ^{13}C



5c

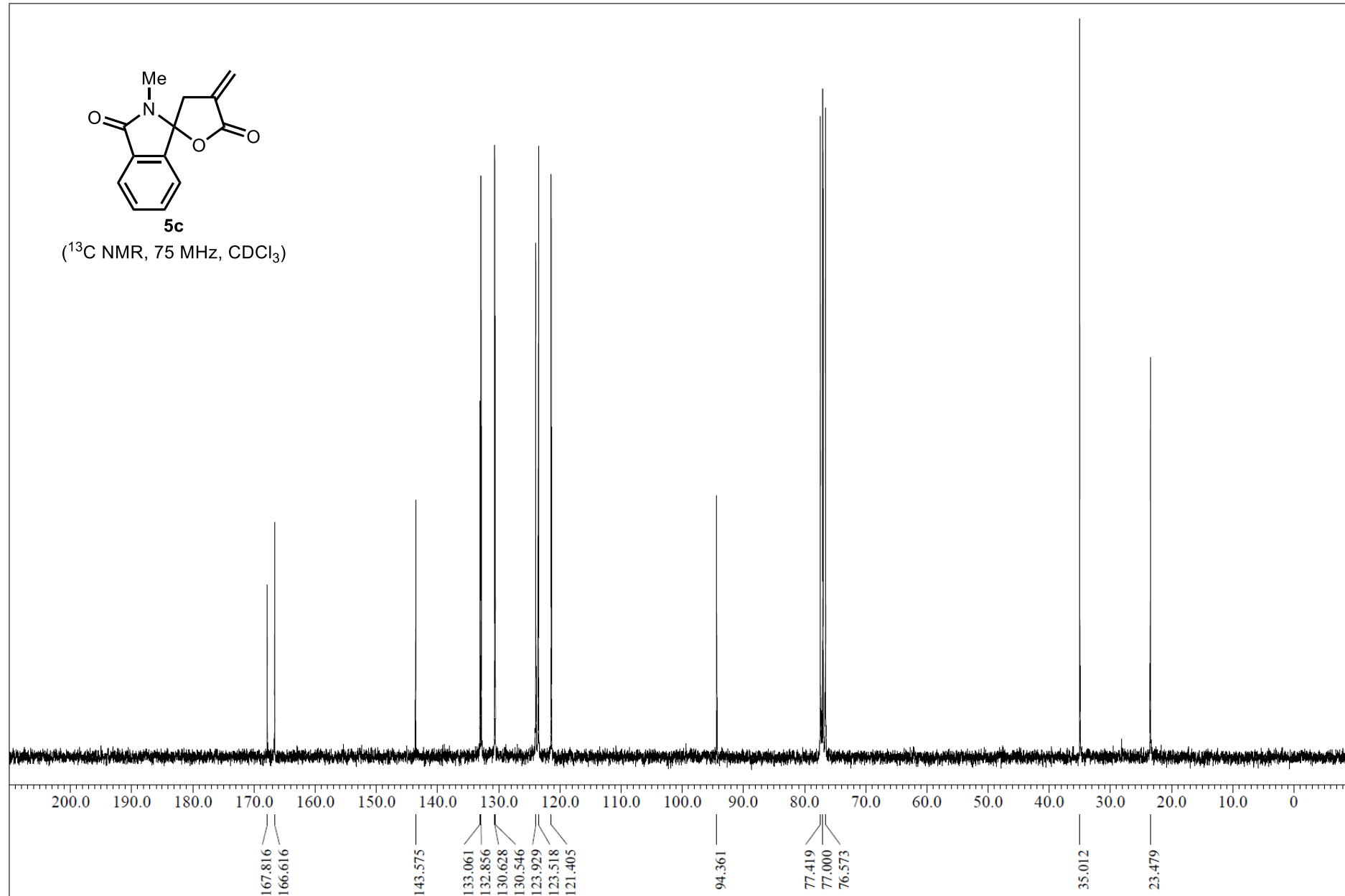
(^1H NMR, 300 MHz, CDCl_3)



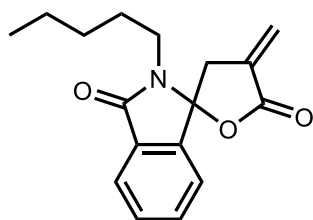


5c

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

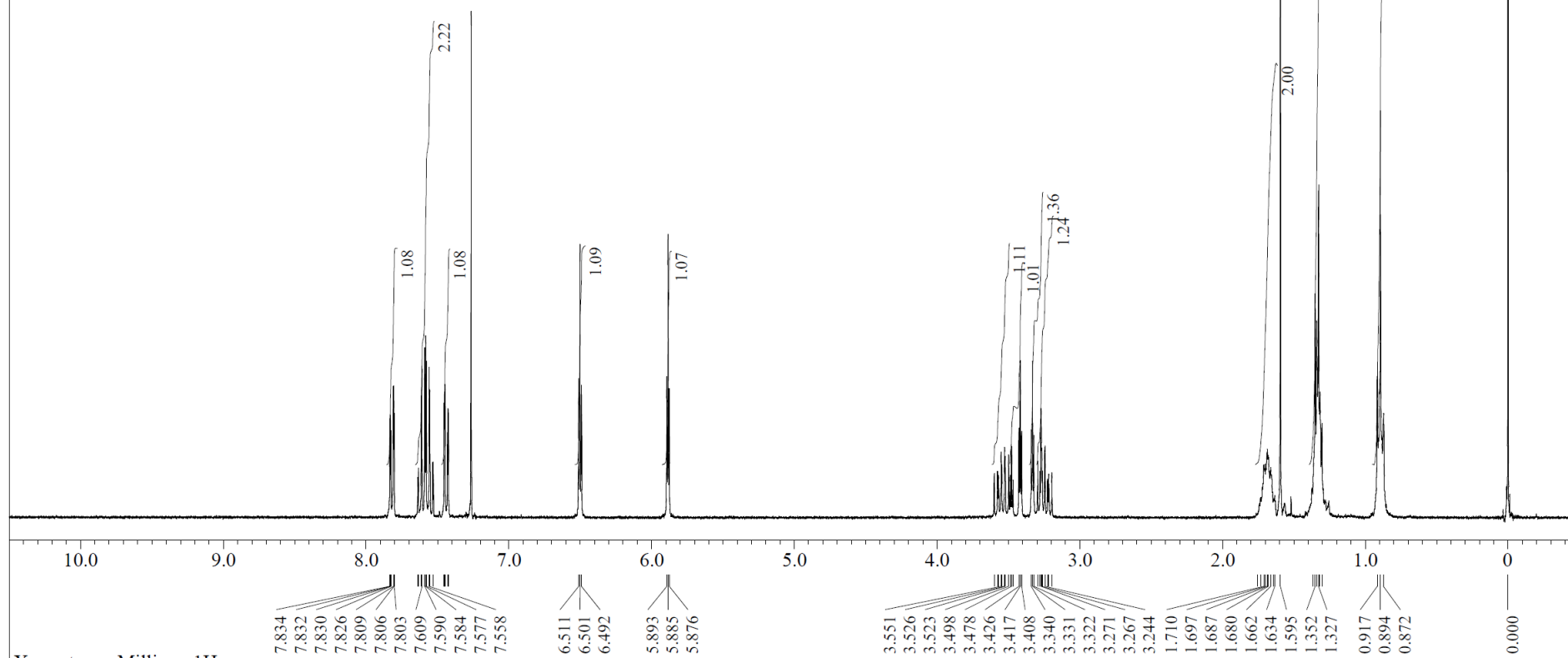


X : parts per Million : ^{13}C

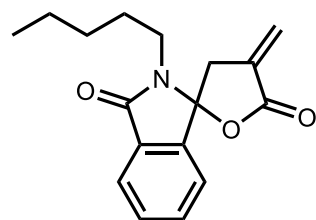


5d

(^1H NMR, 300 MHz, CDCl_3)

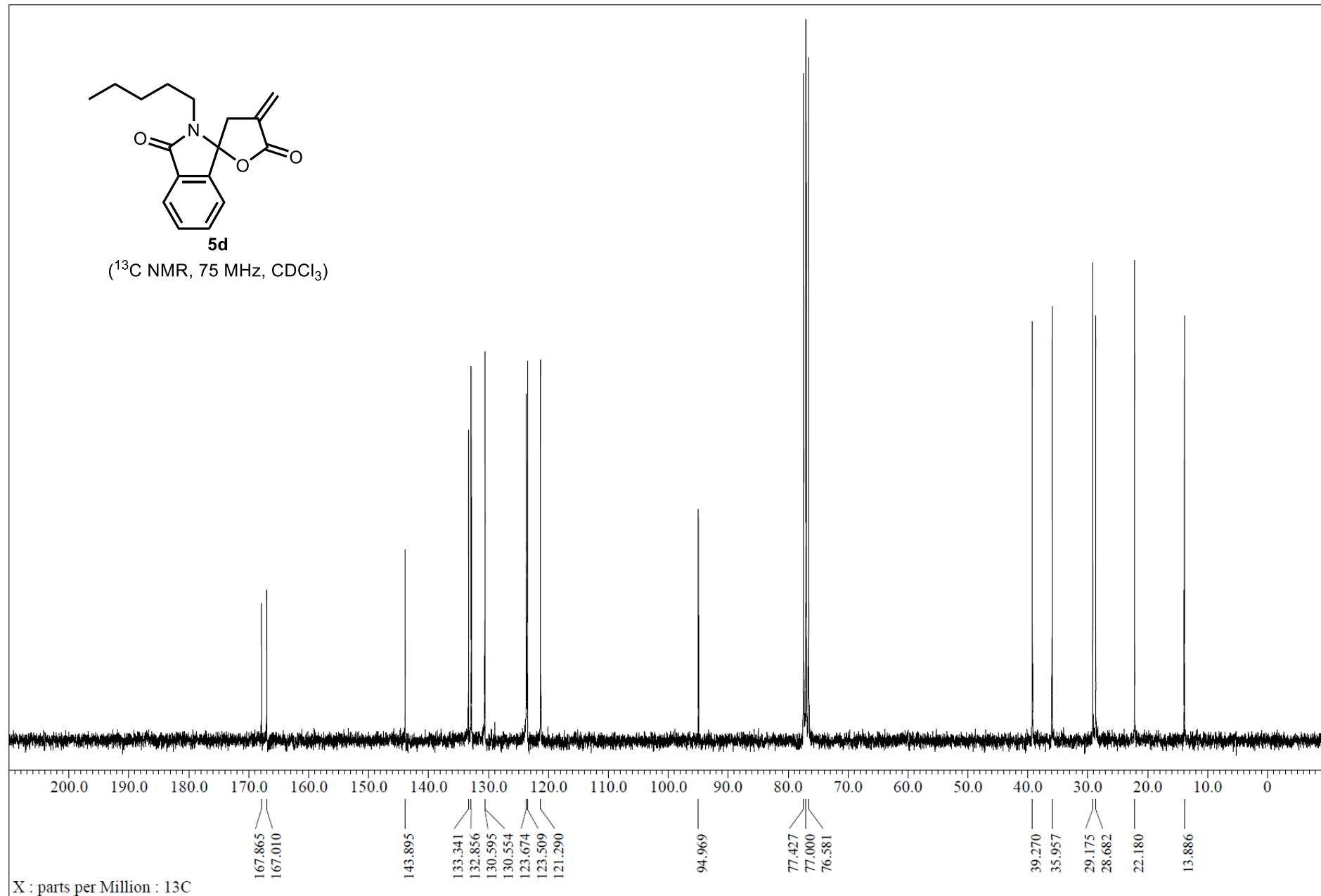


X : parts per Million : ^1H

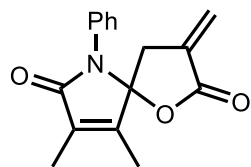


5d

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

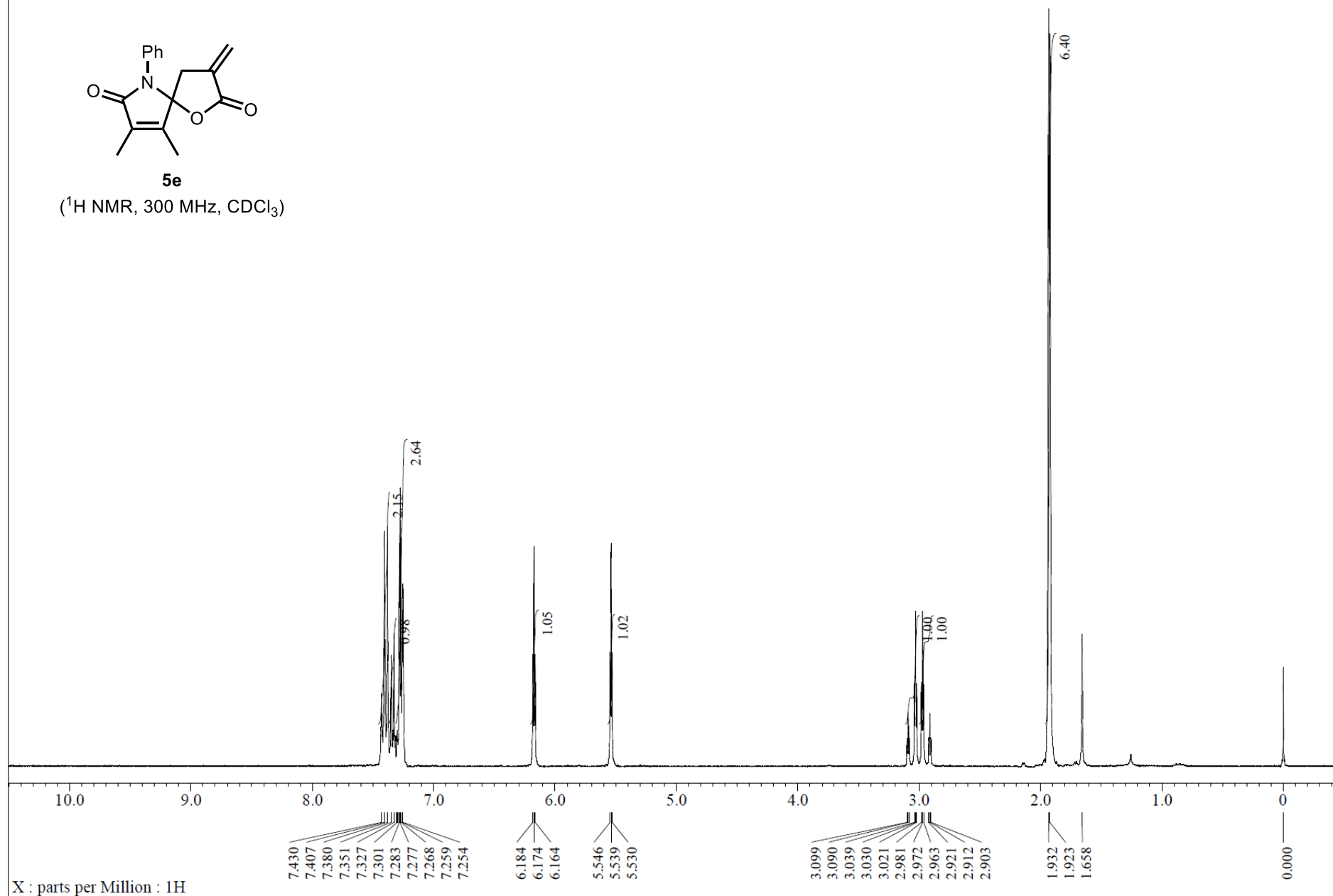


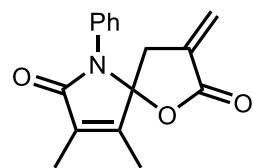
X : parts per Million : ^{13}C



5e

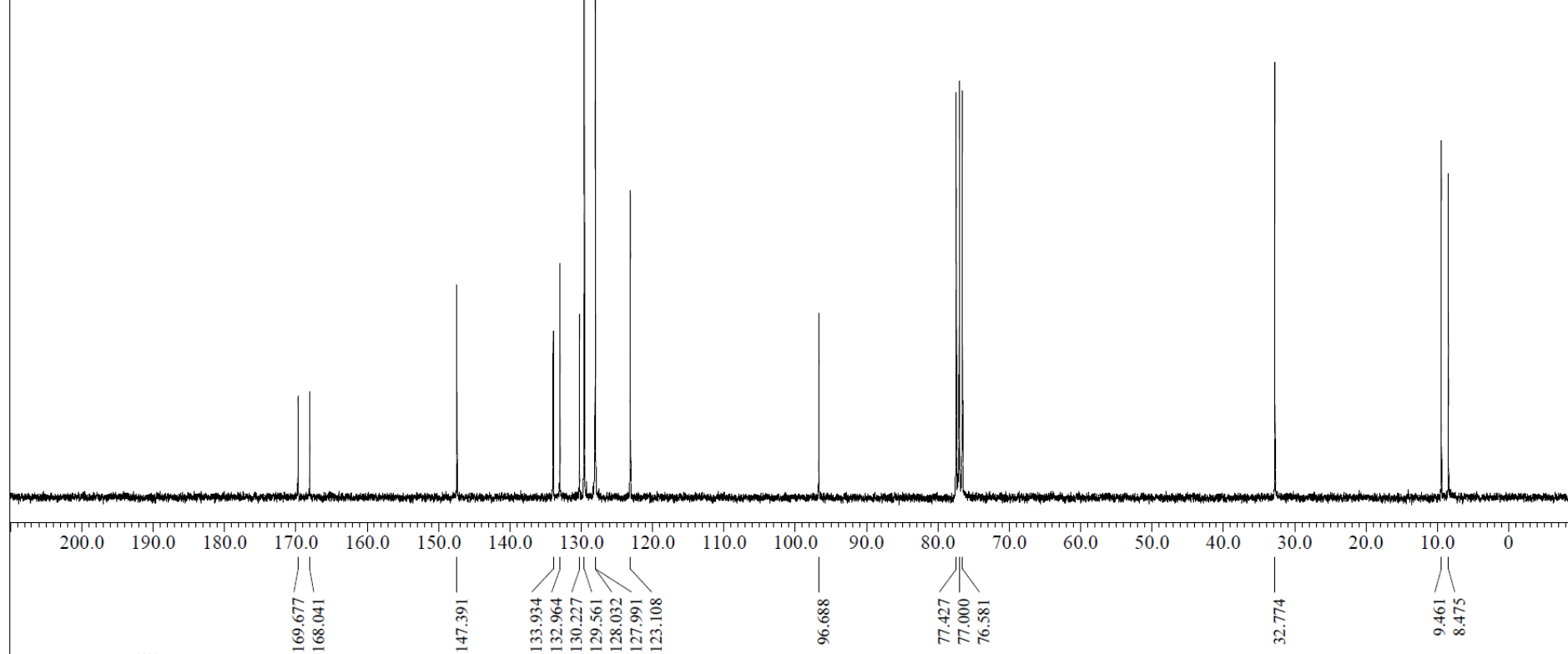
(¹H NMR, 300 MHz, CDCl₃)



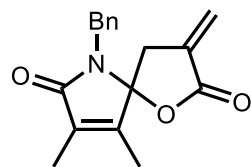


5e

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

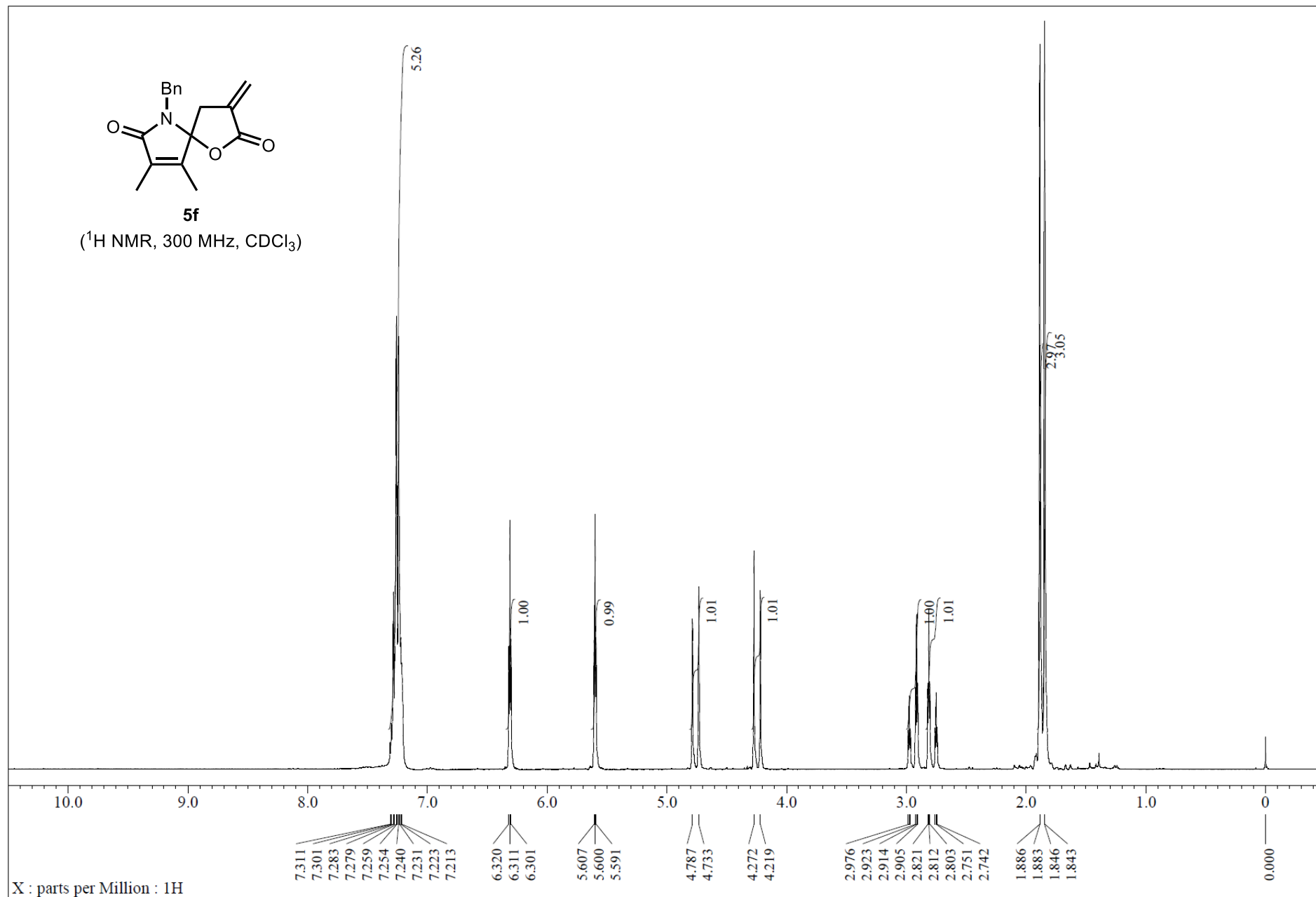


X : parts per Million : ^{13}C

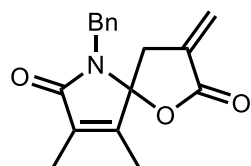


5f

(^1H NMR, 300 MHz, CDCl_3)

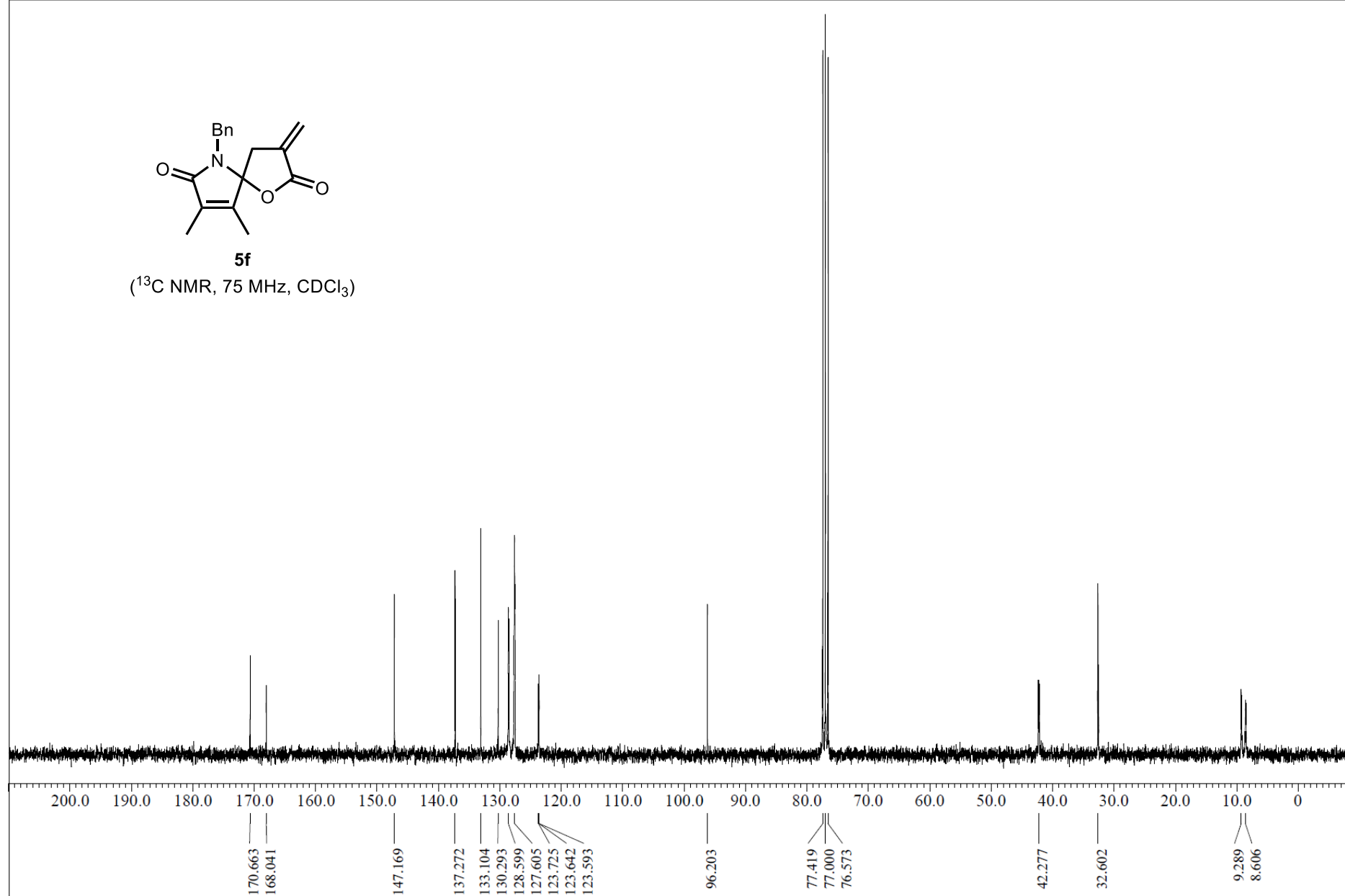


X : parts per Million : ^1H

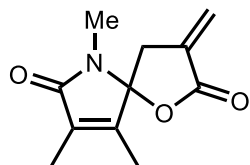


5f

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

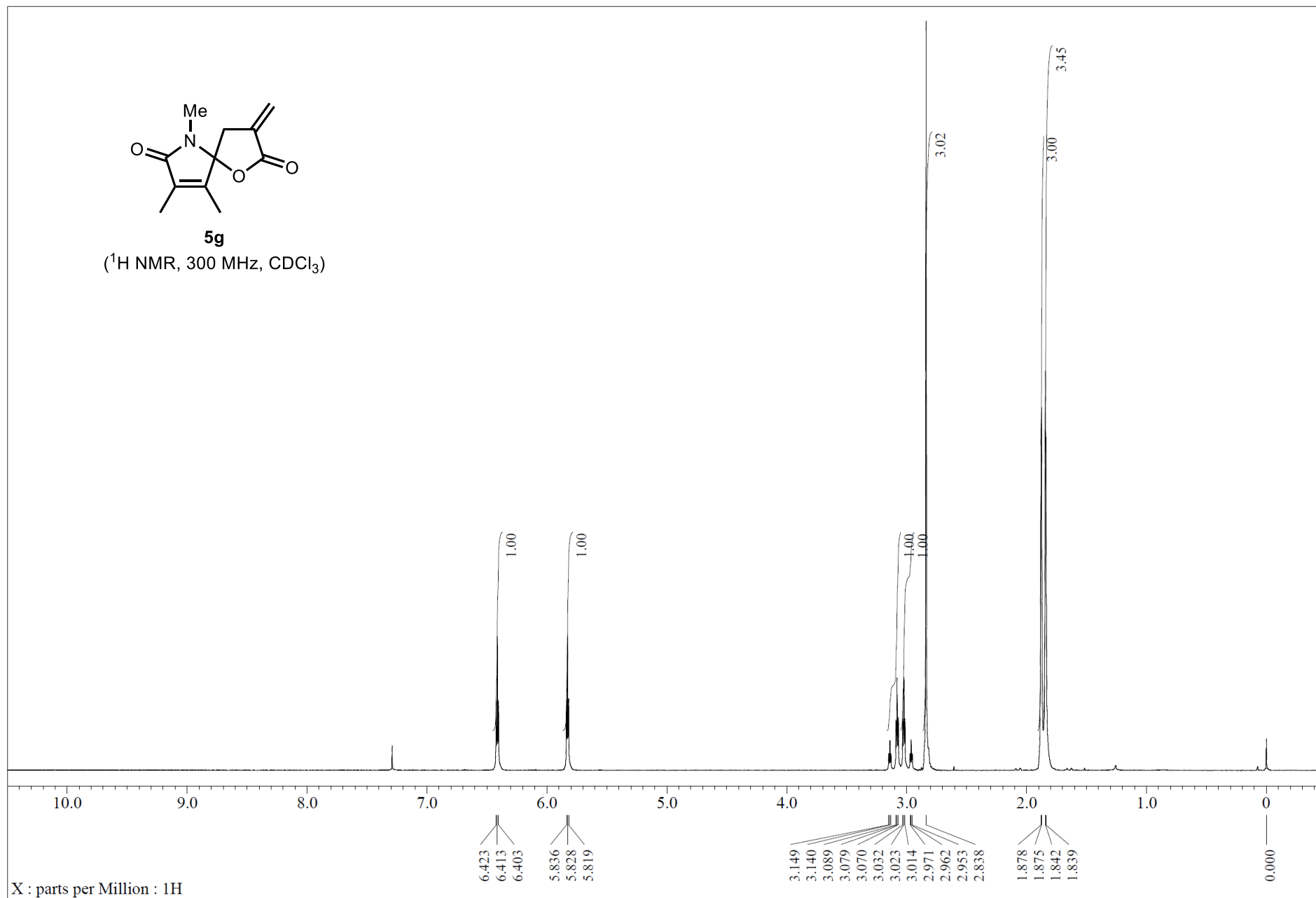


X : parts per Million : ^{13}C

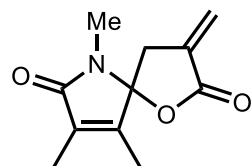


5g

(¹H NMR, 300 MHz, CDCl₃)

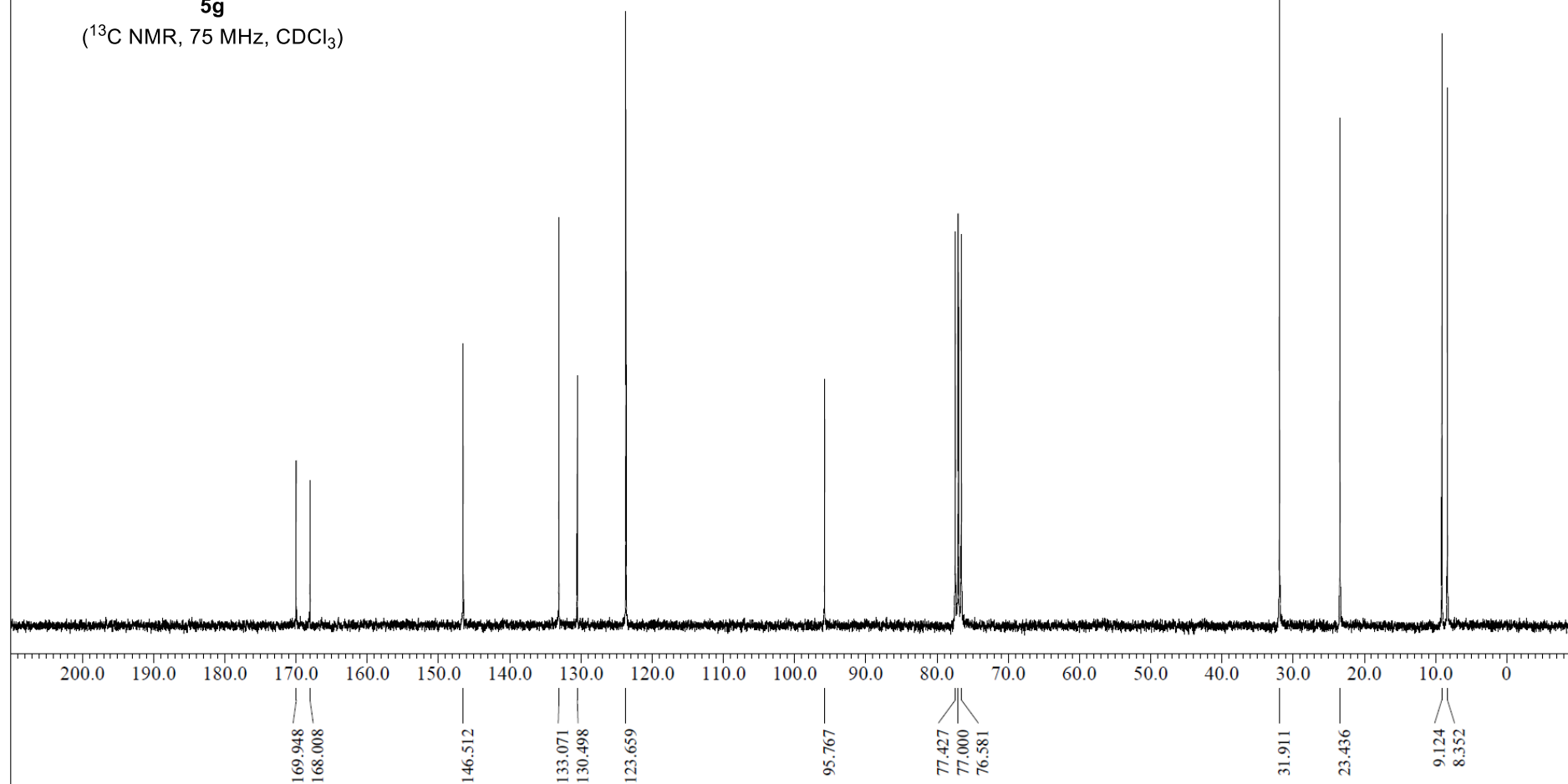


X : parts per Million : 1H

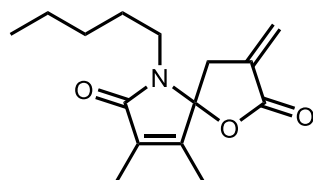


5g

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

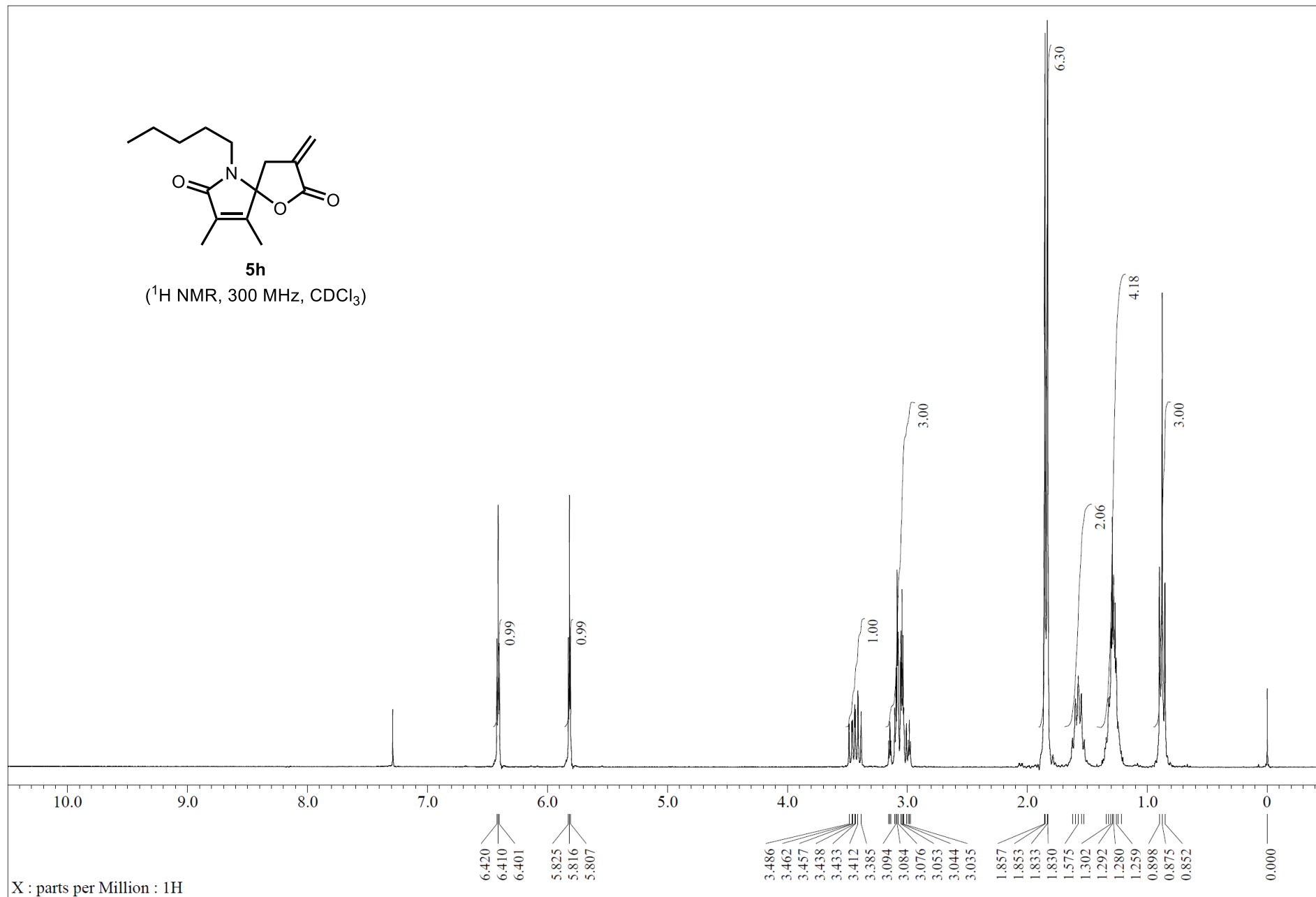


X : parts per Million : ^{13}C

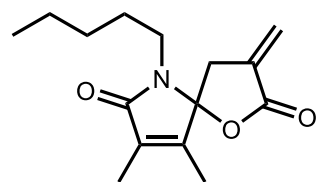


5h

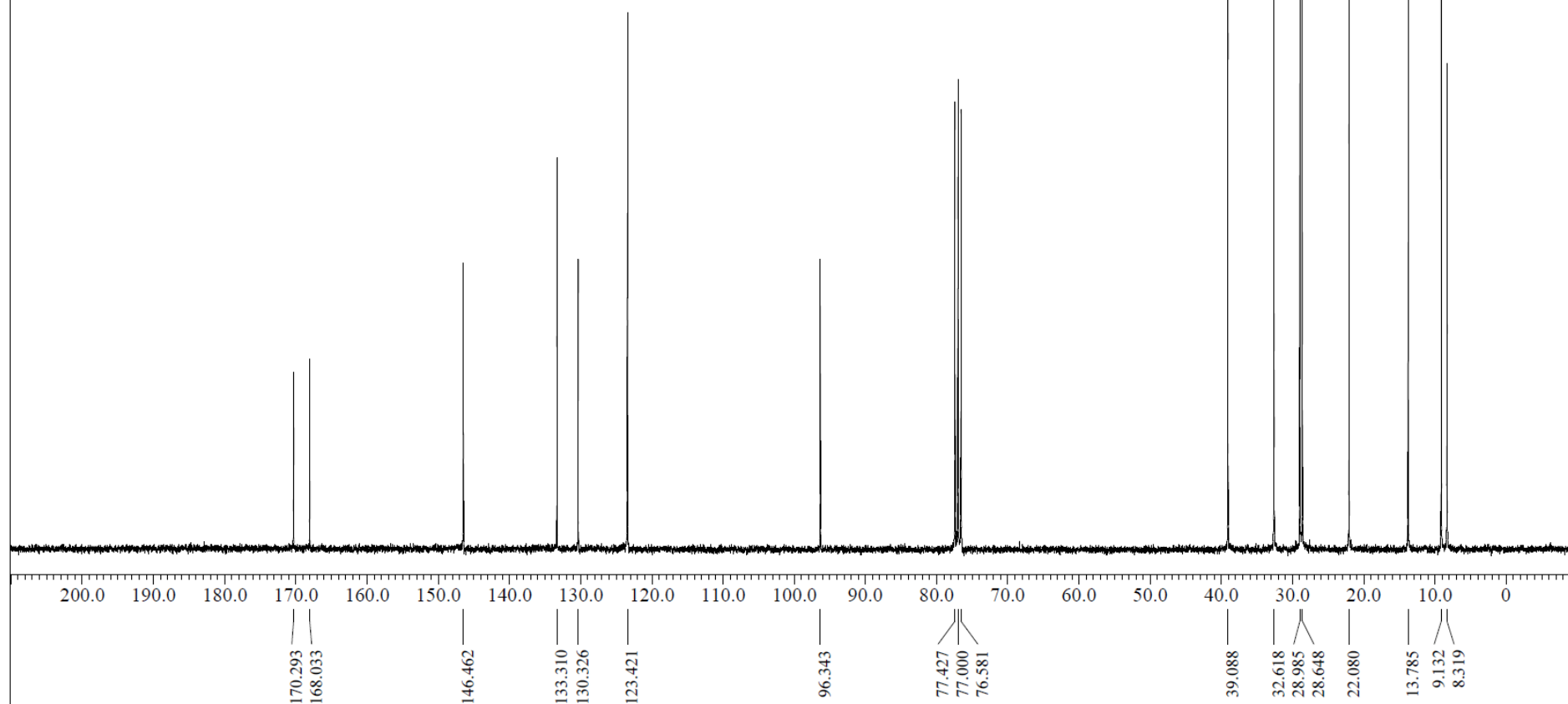
(^1H NMR, 300 MHz, CDCl_3)



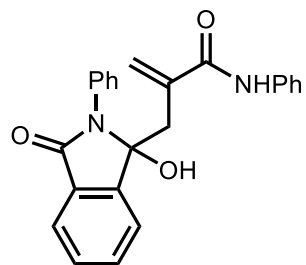
X : parts per Million : ^1H



5h
 (^{13}C NMR, 75 MHz, CDCl_3)

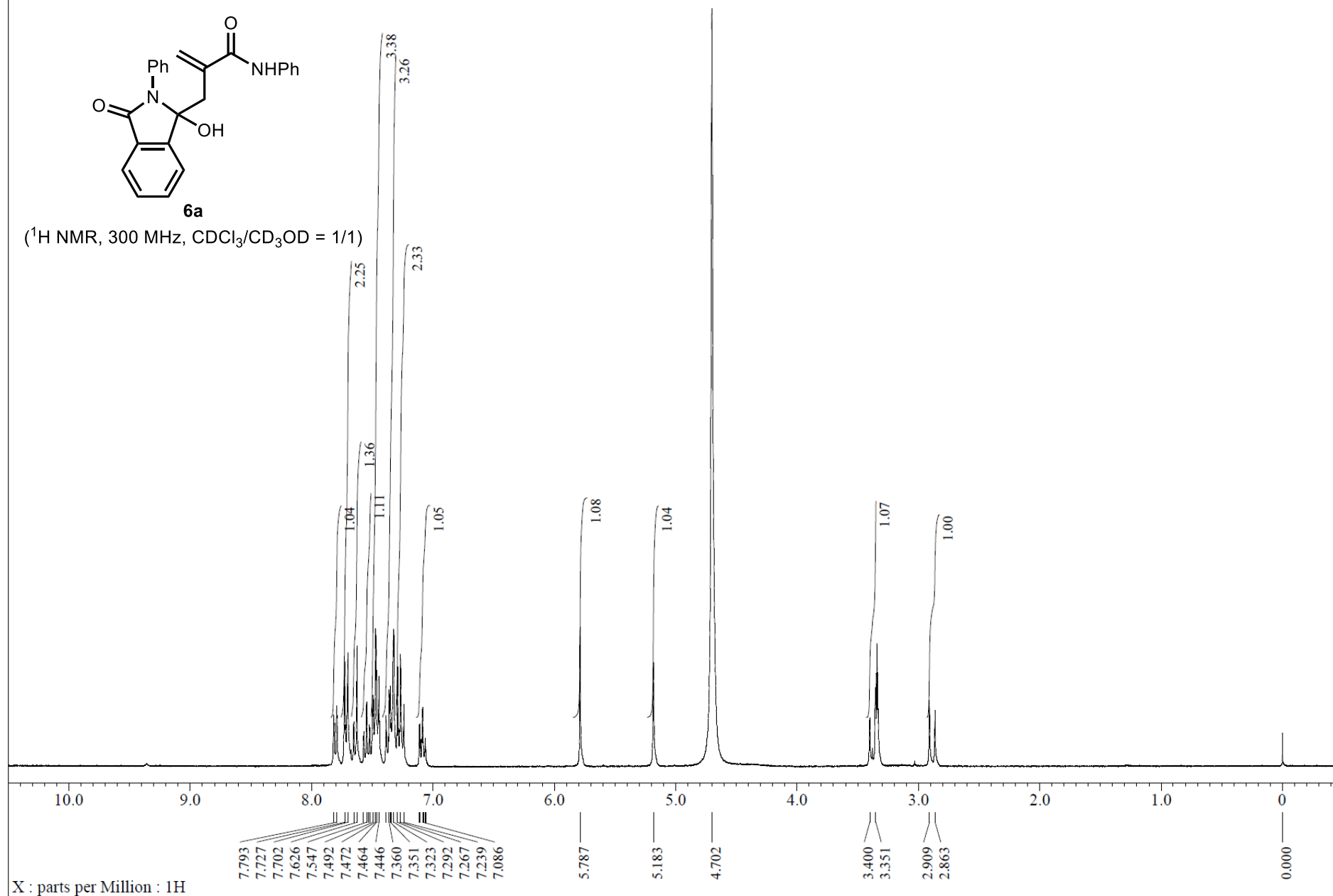


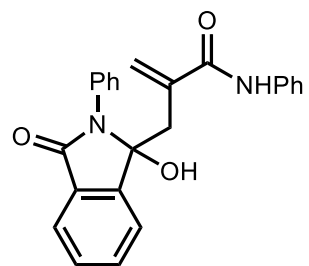
X : parts per Million : ^{13}C



6a

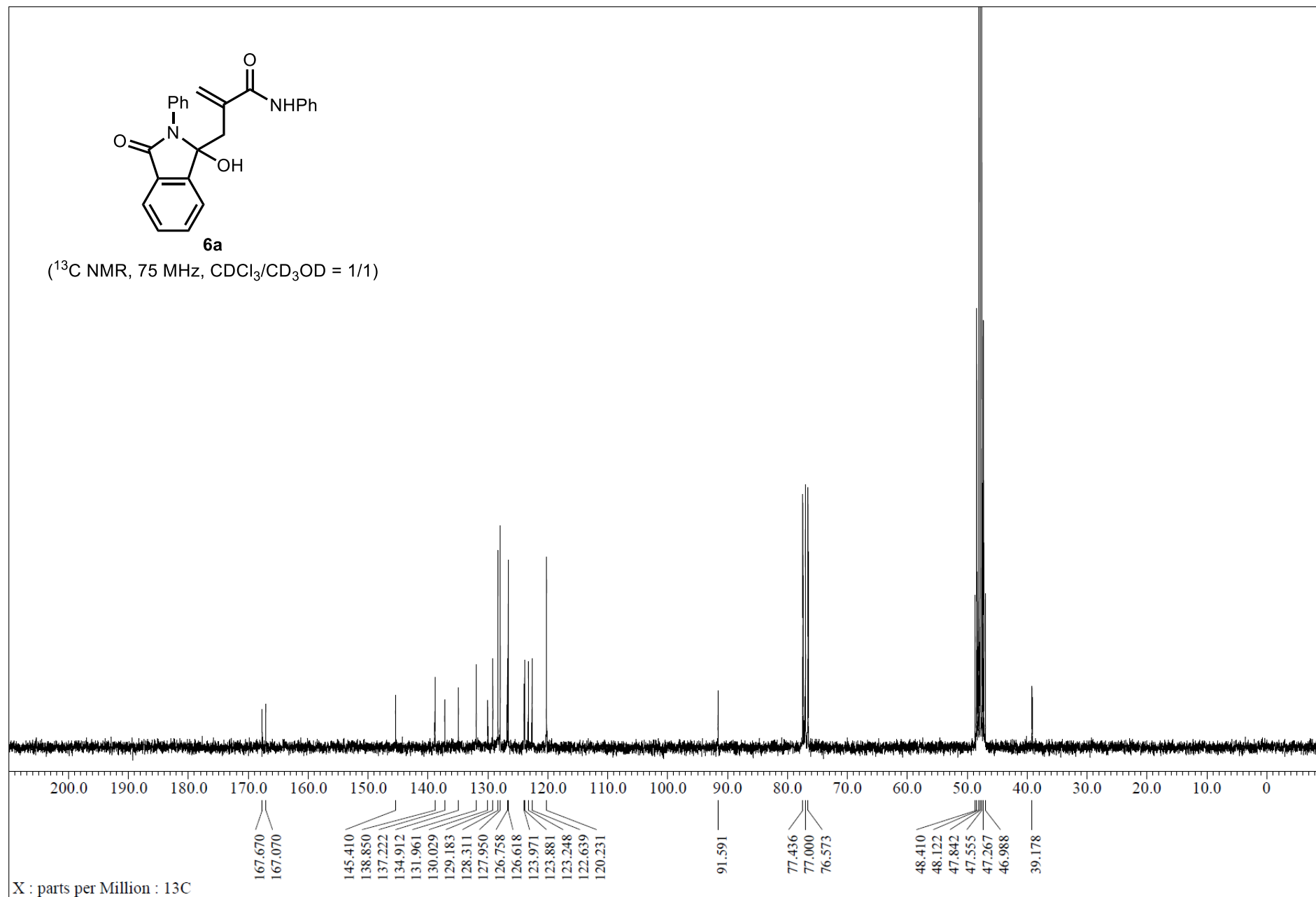
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

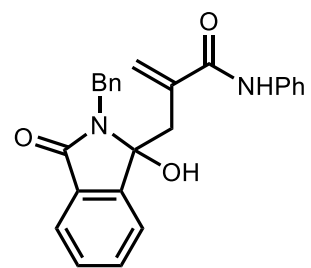




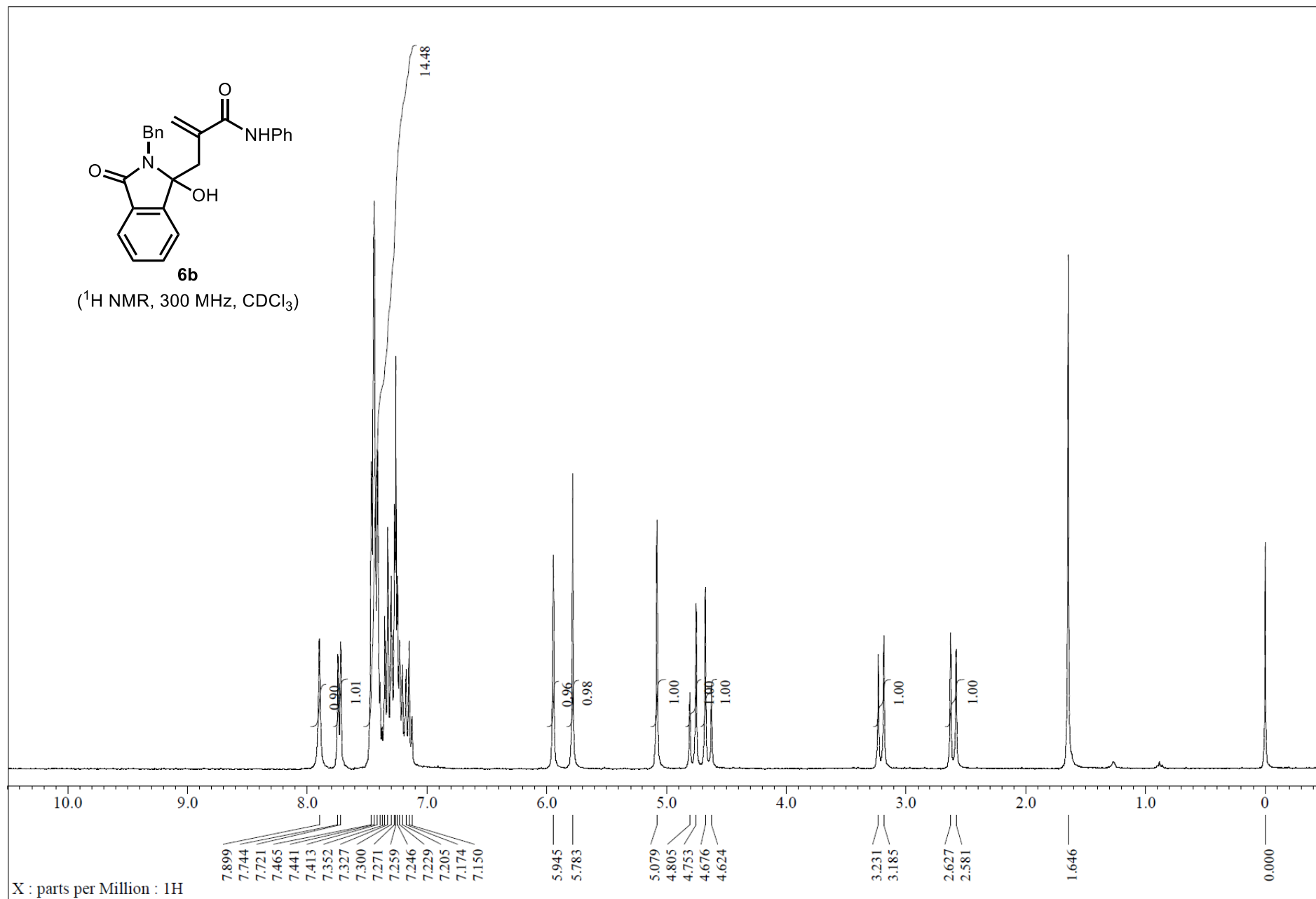
6a

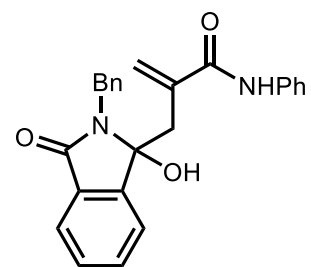
(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)





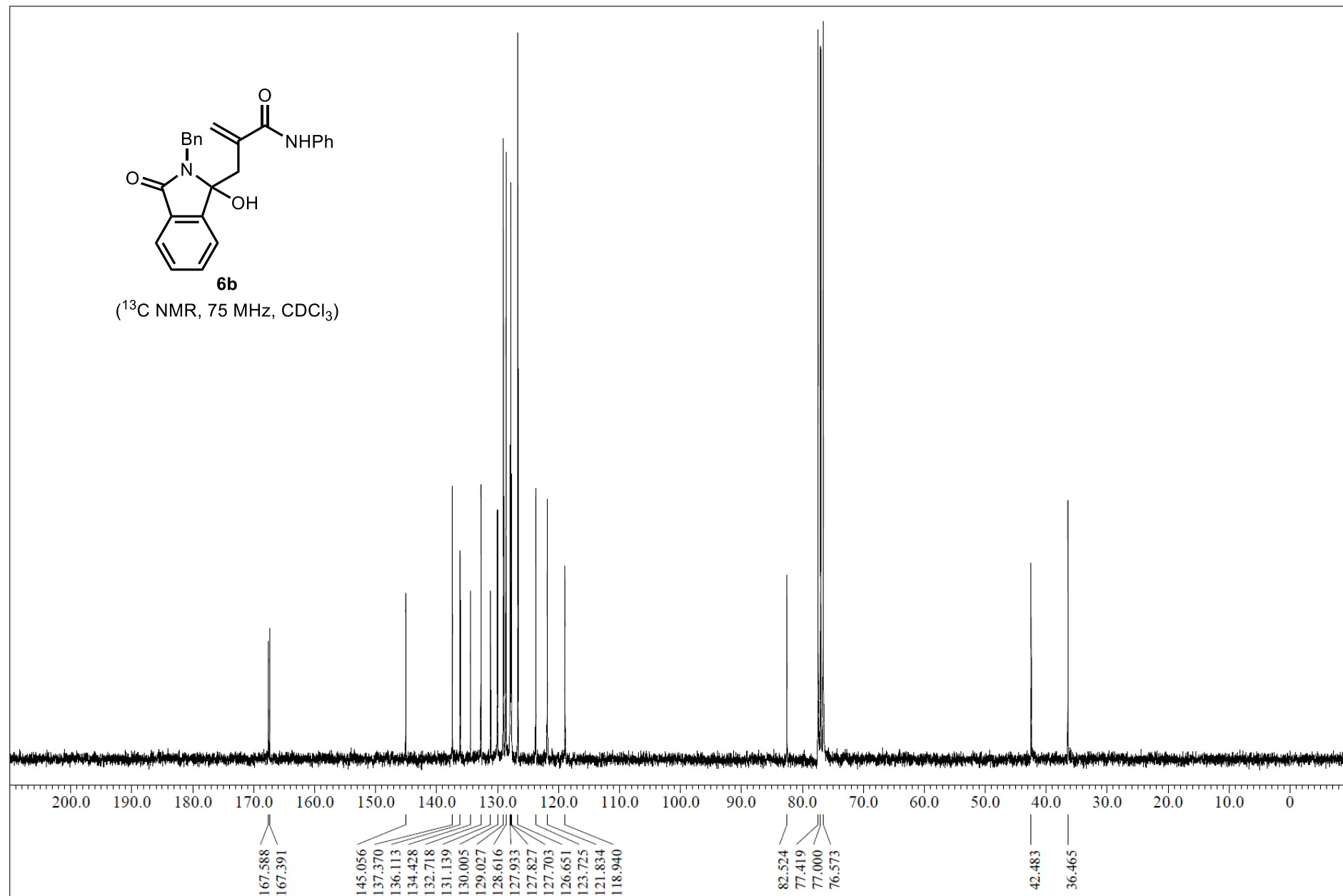
6b
 ^1H NMR, 300 MHz, CDCl_3)



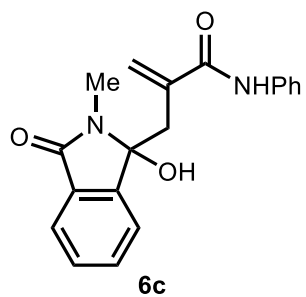


6b

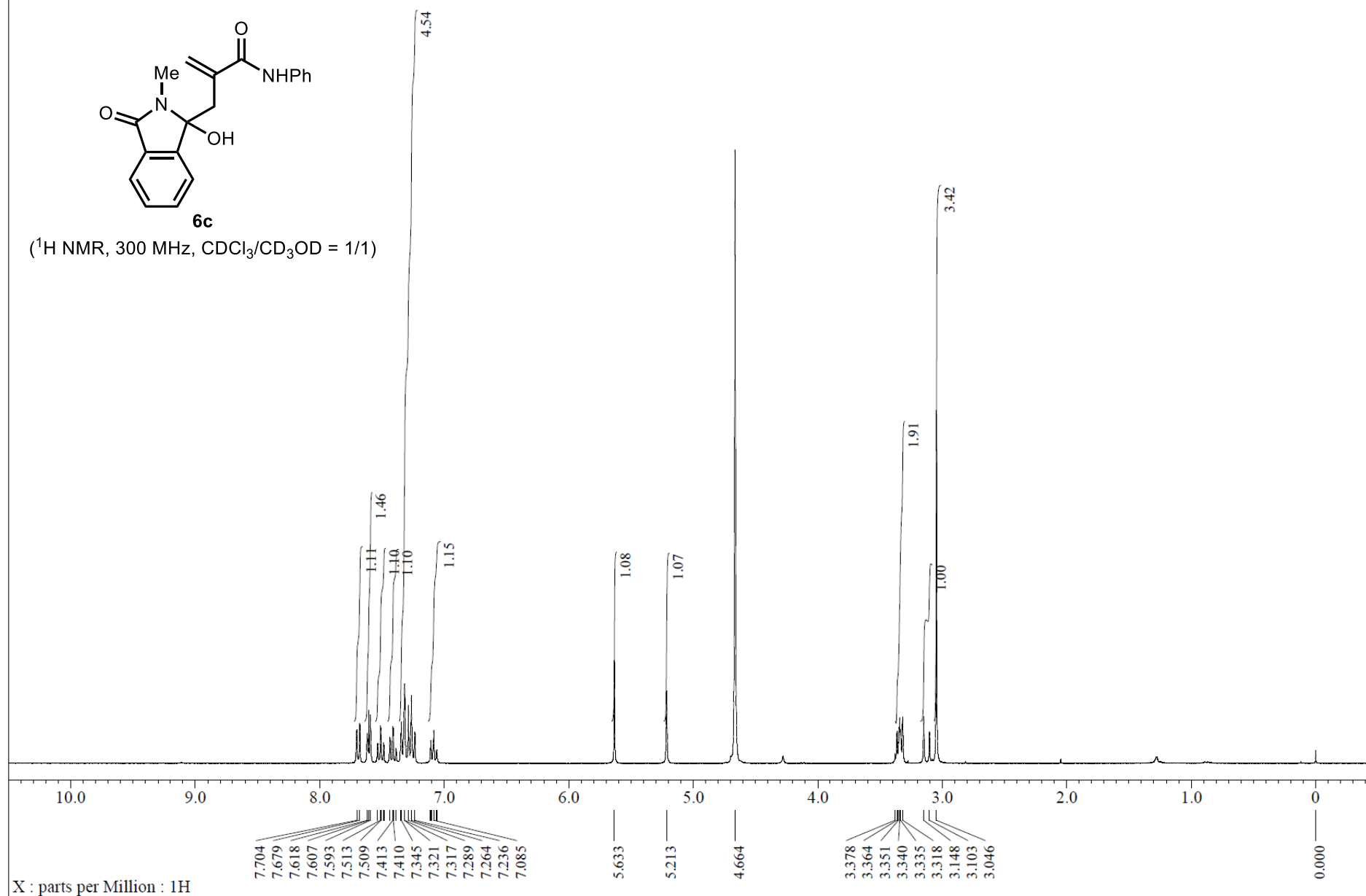
(^{13}C NMR, 75 MHz, CDCl_3)

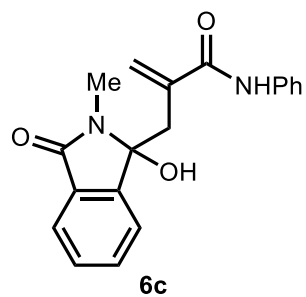


X : parts per Million : ^{13}C

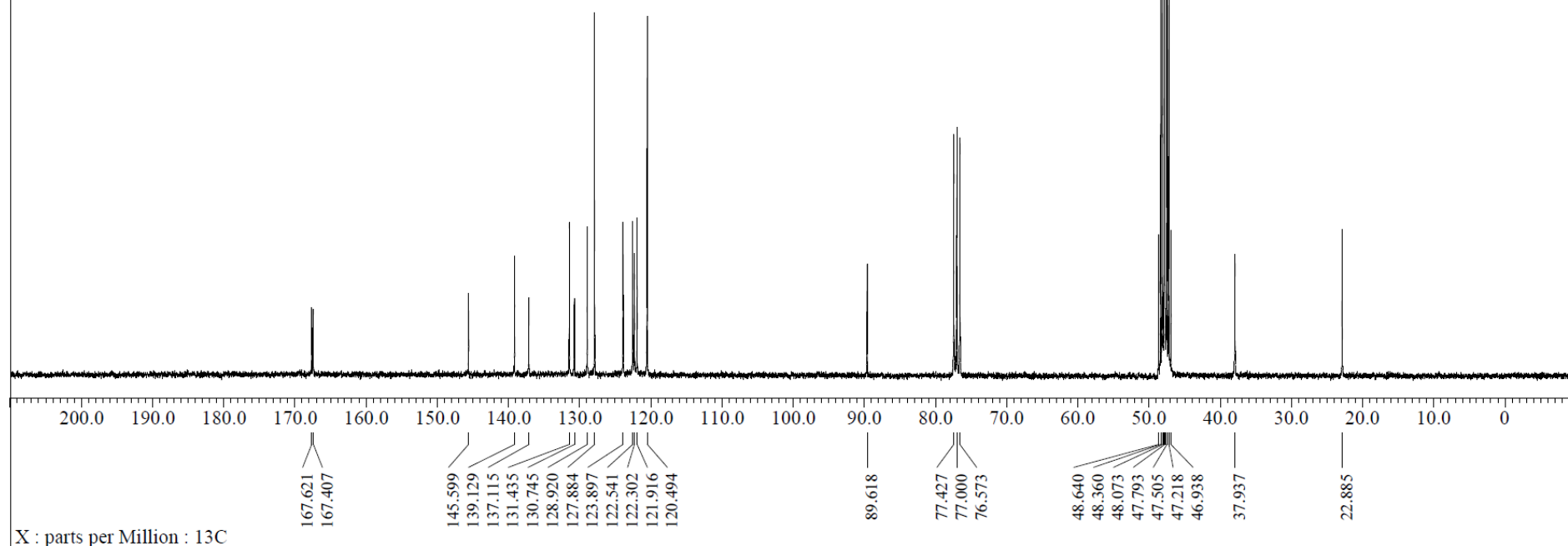


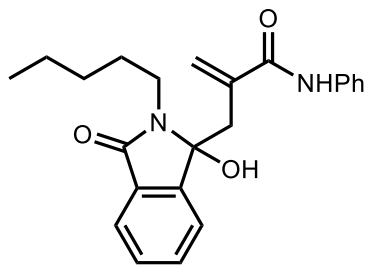
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)





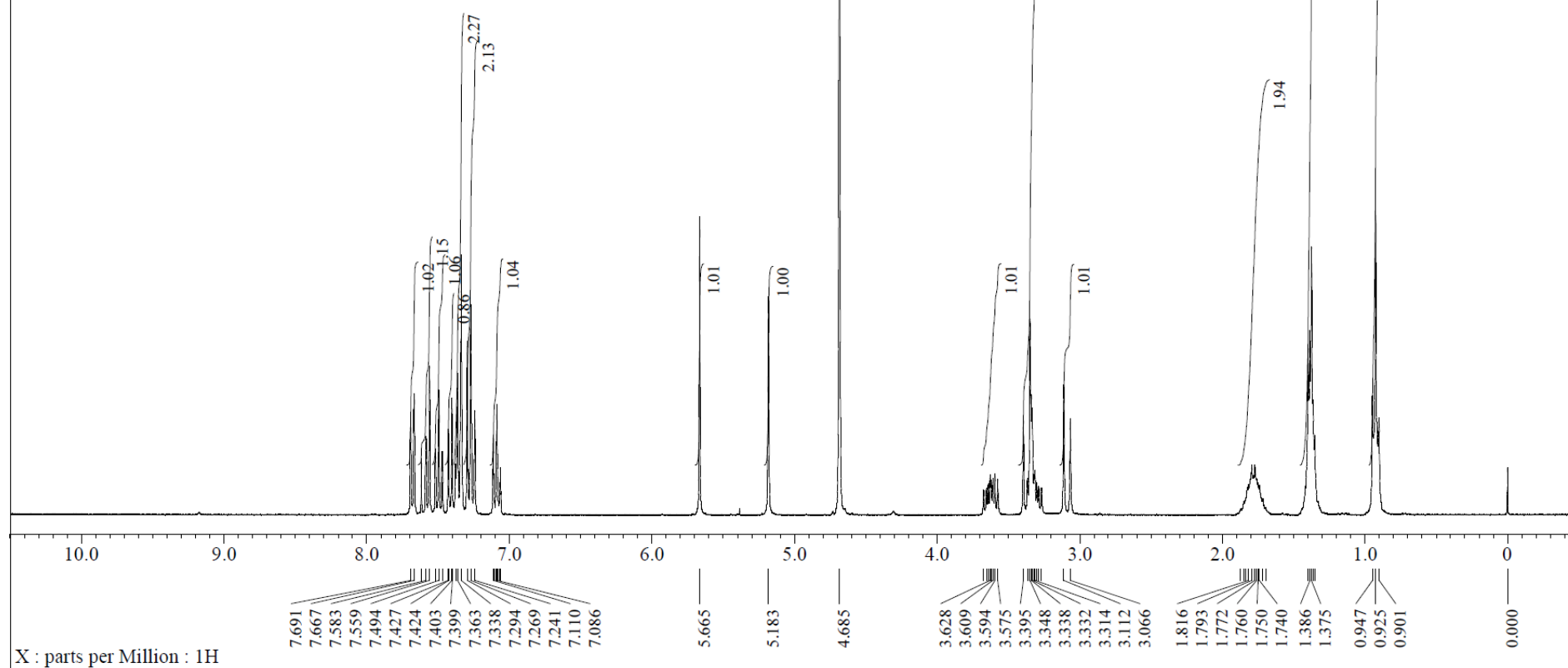
(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

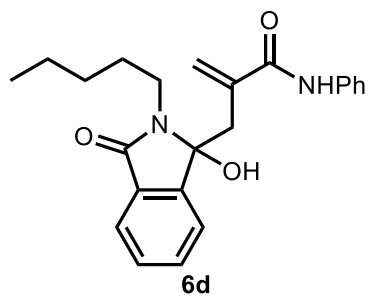




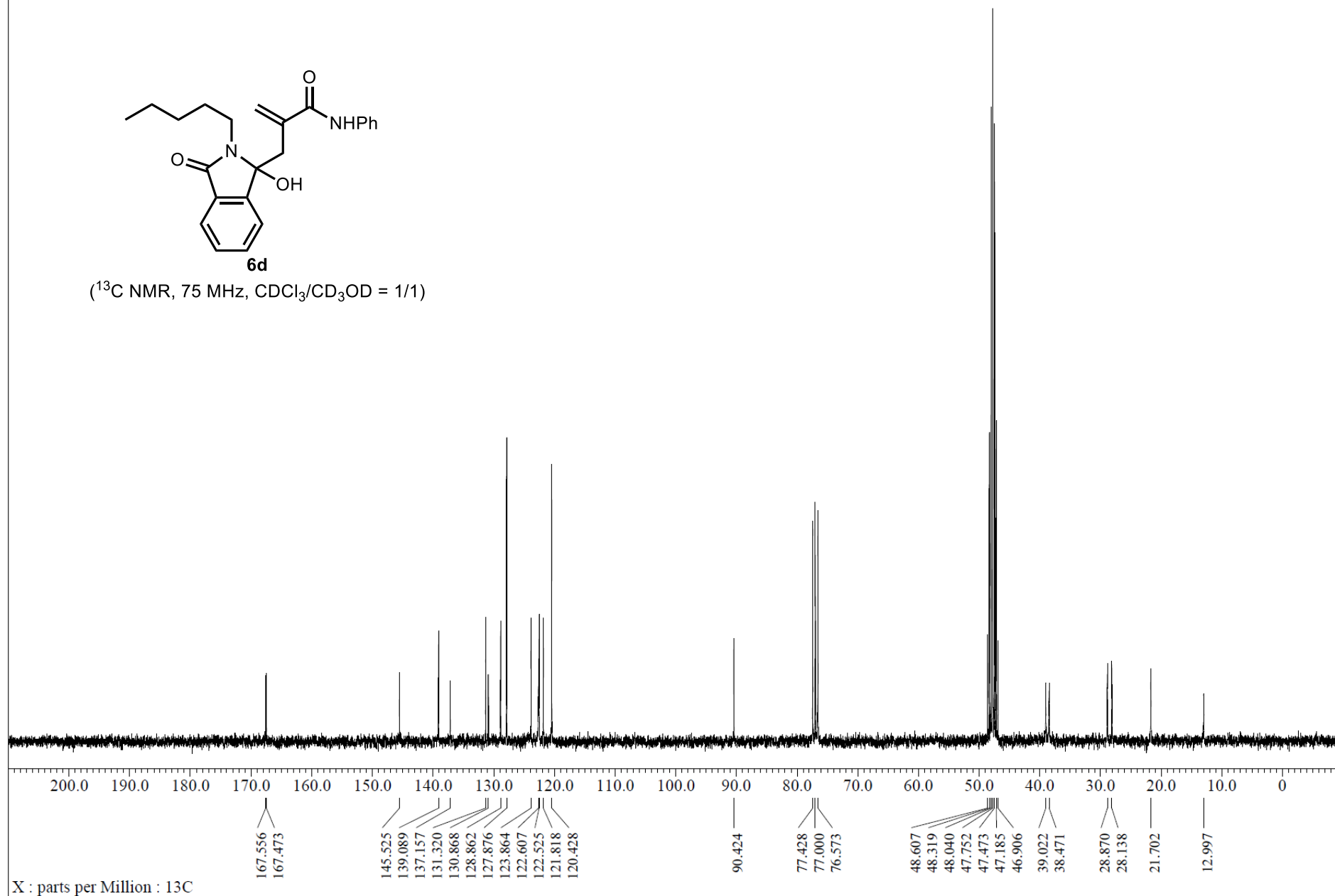
6d

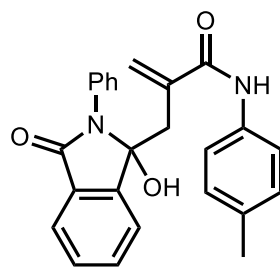
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)





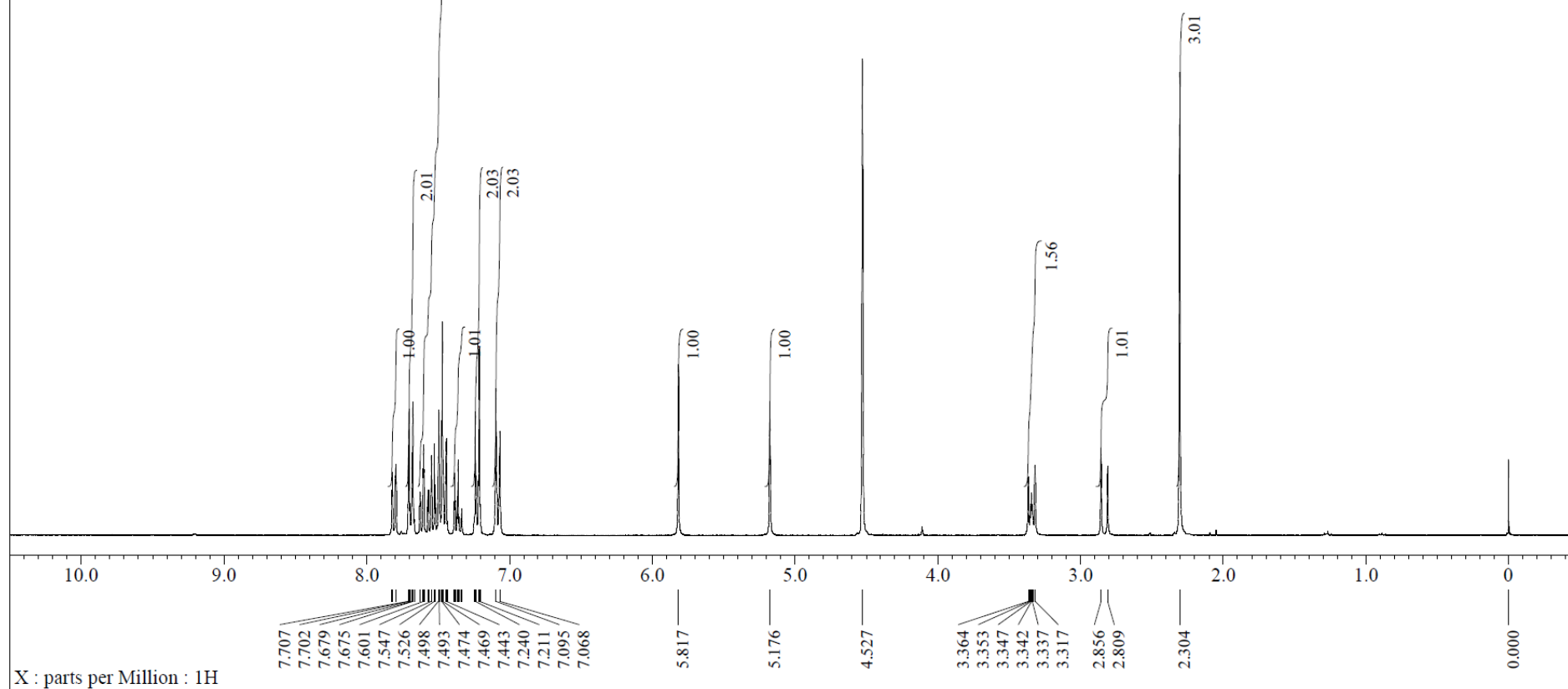
(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

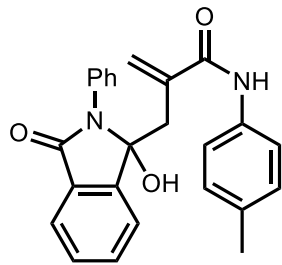




6e

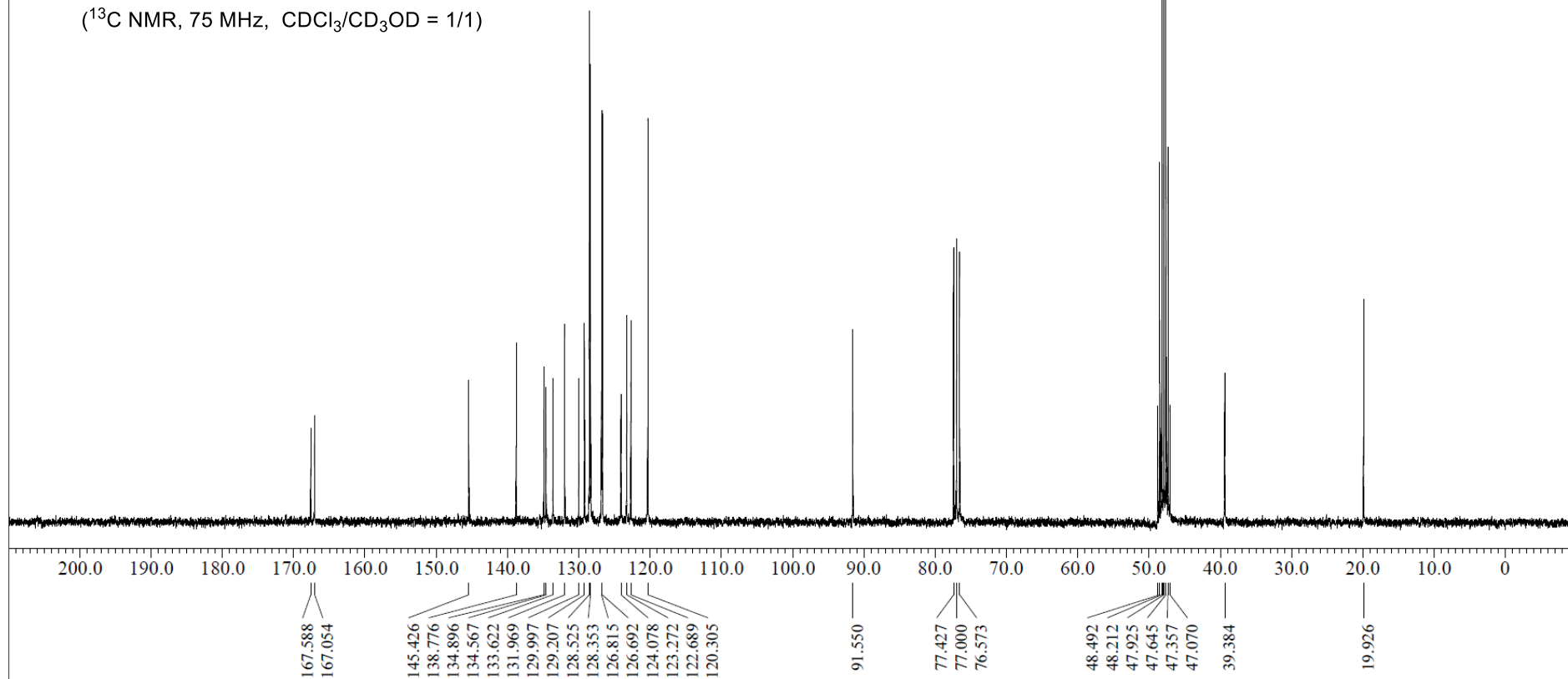
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



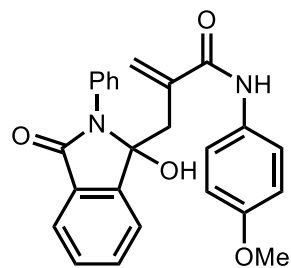


6e

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

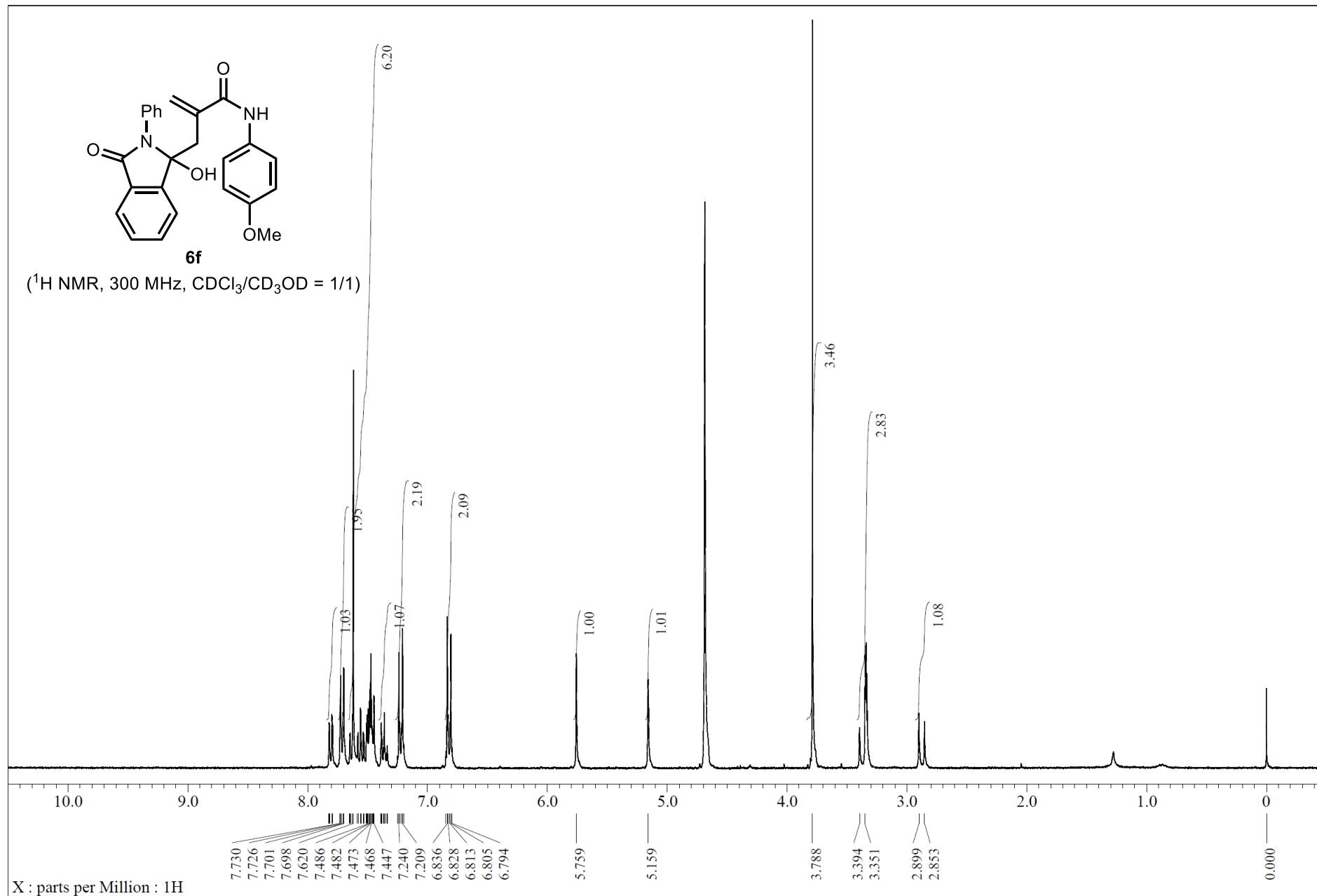


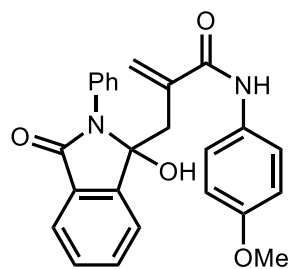
X : parts per Million : ^{13}C



6f

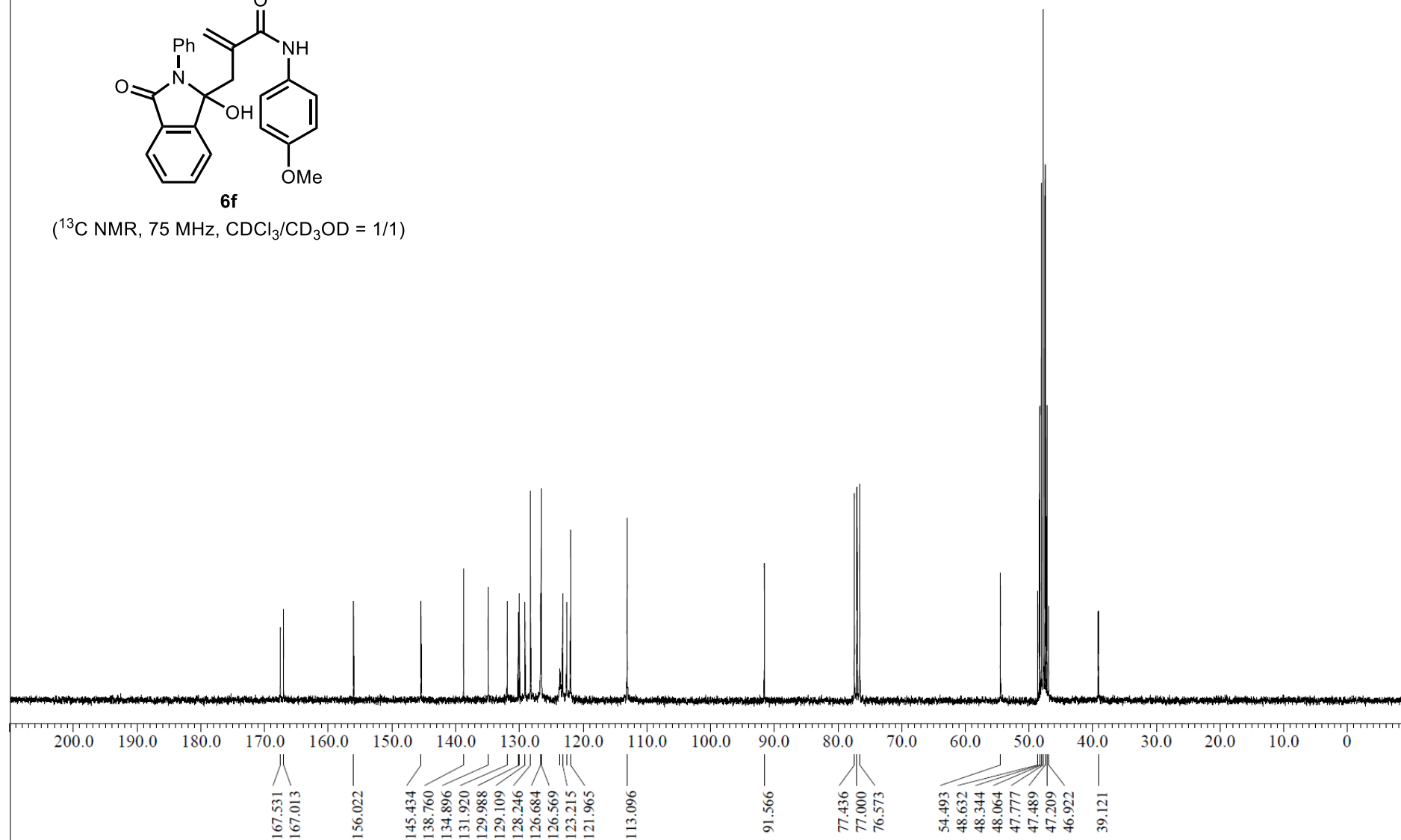
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



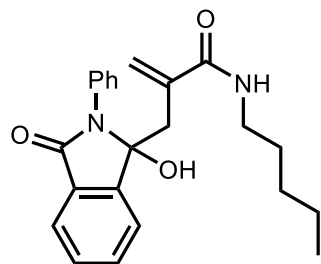


6f

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

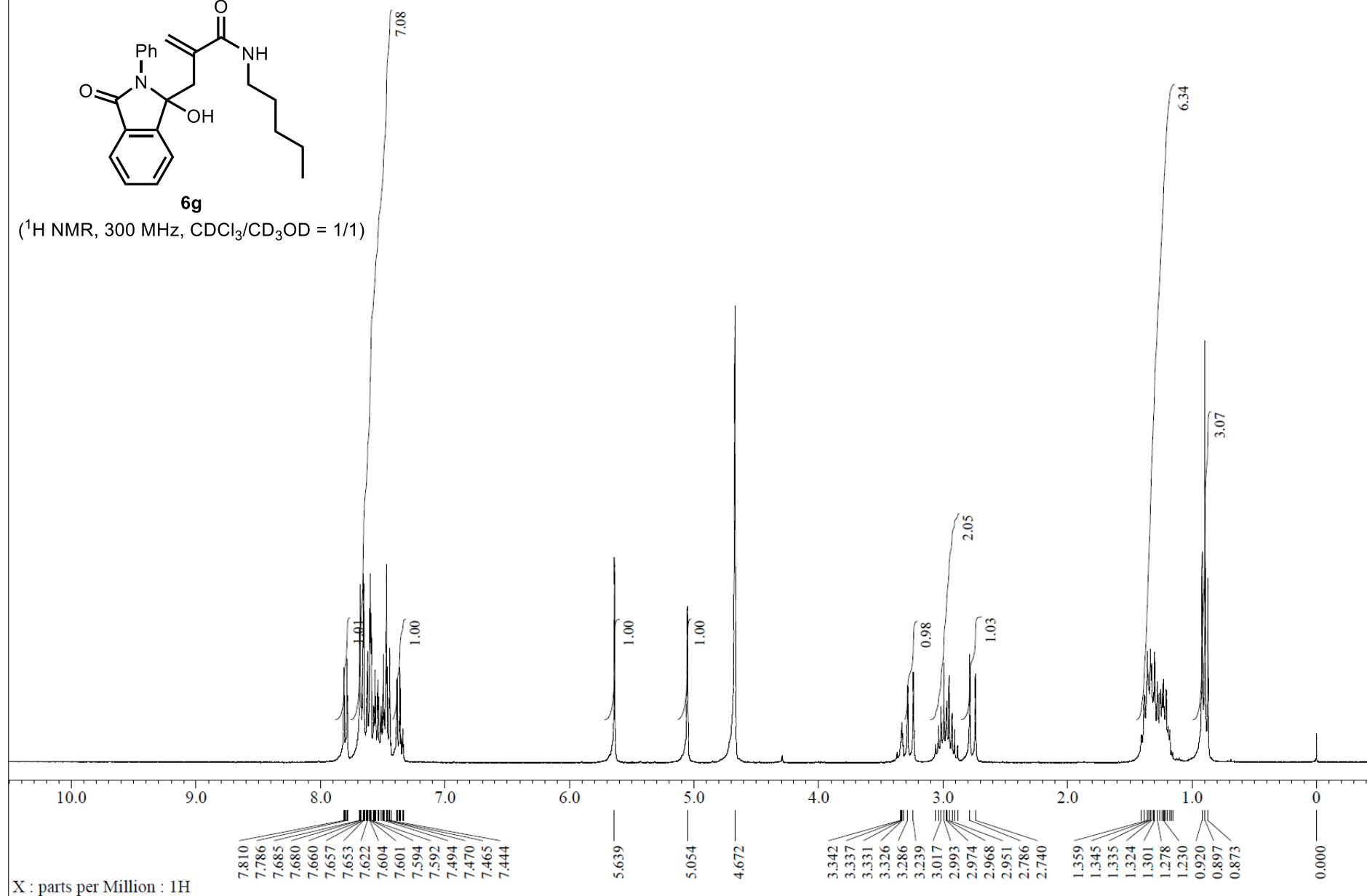


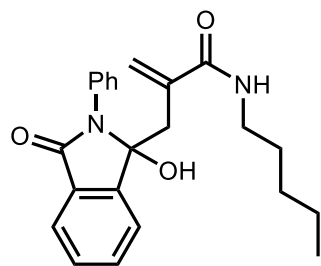
X : parts per Million : ^{13}C



6g

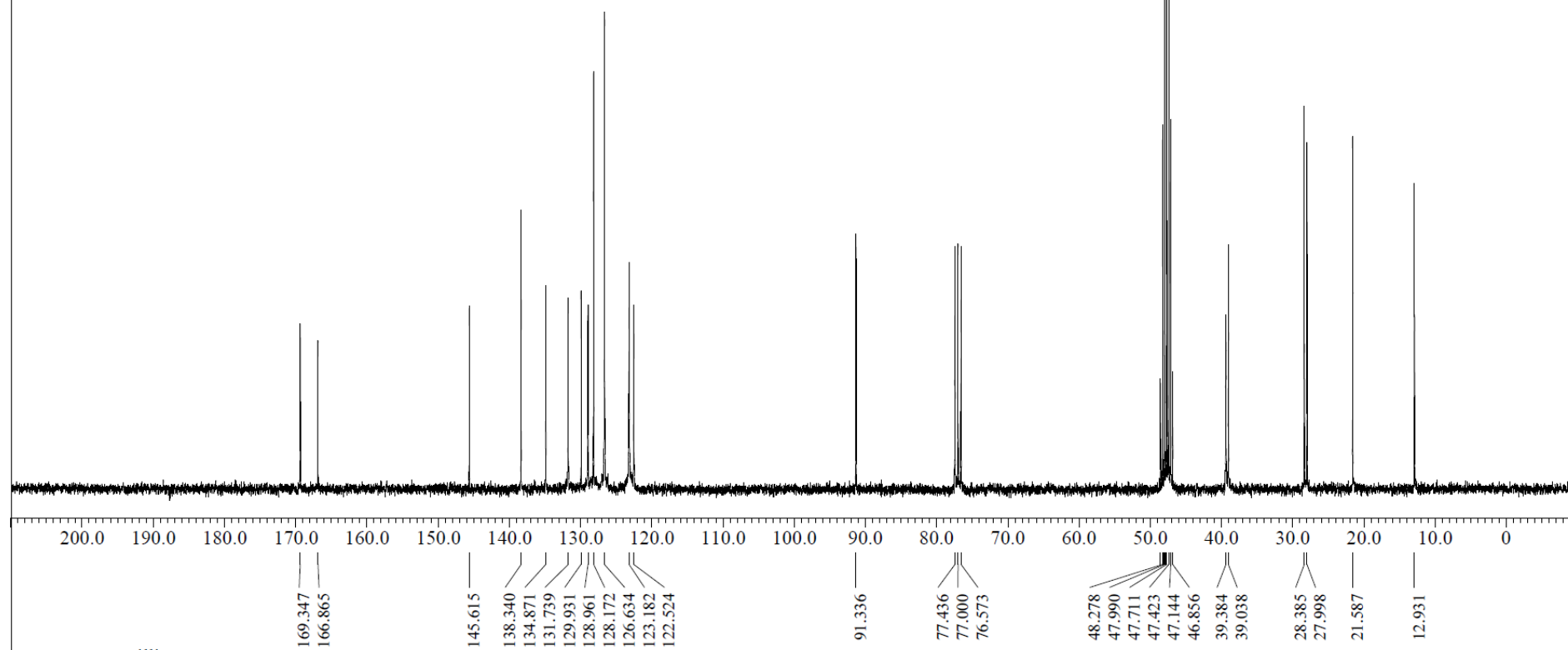
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



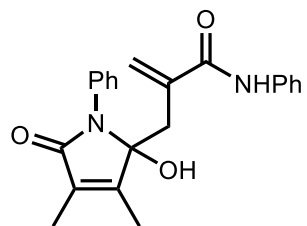


6g

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

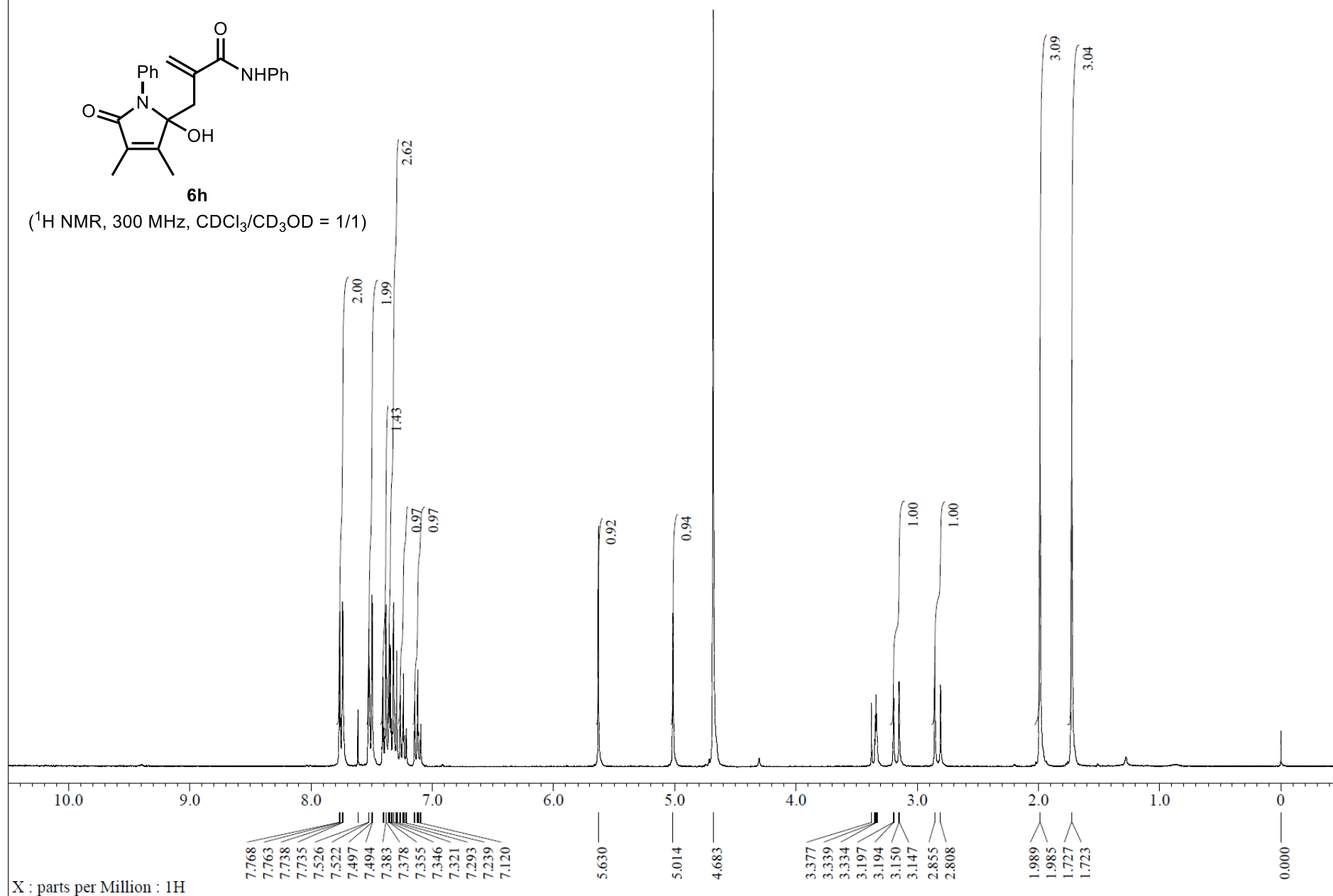


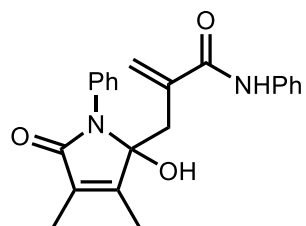
X : parts per Million : ^{13}C



6h

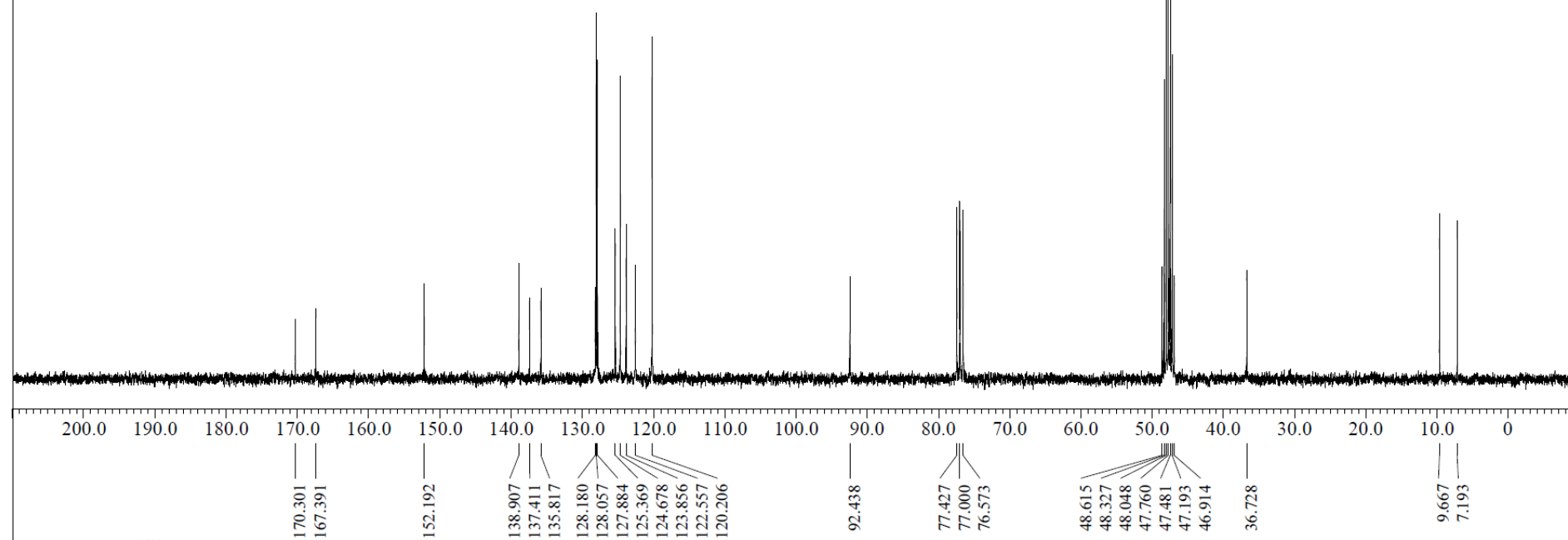
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



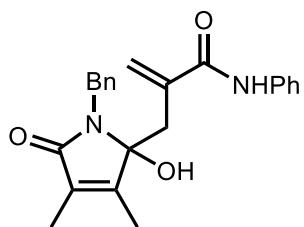


6h

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

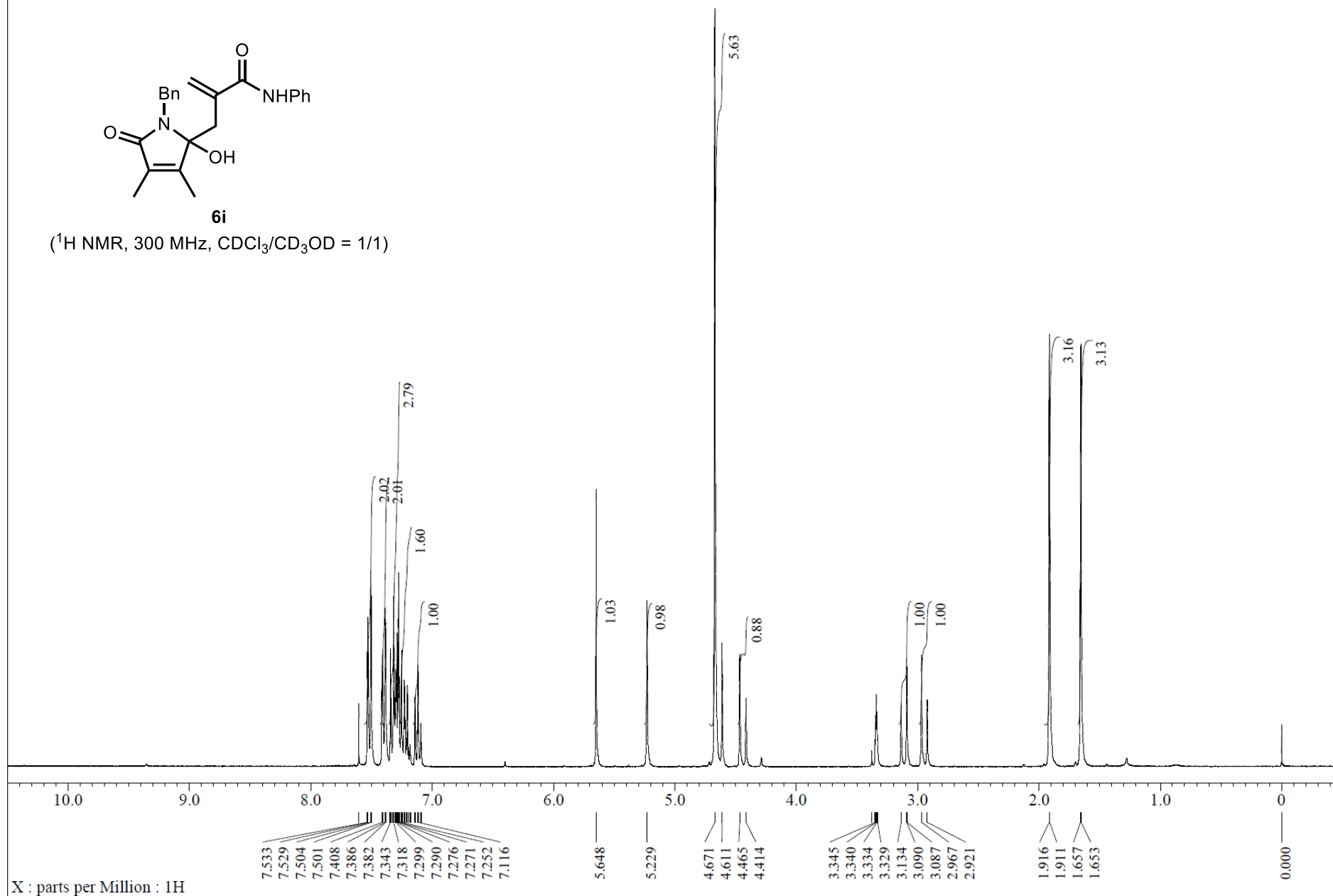


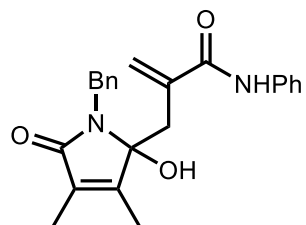
X : parts per Million : ^{13}C



6i

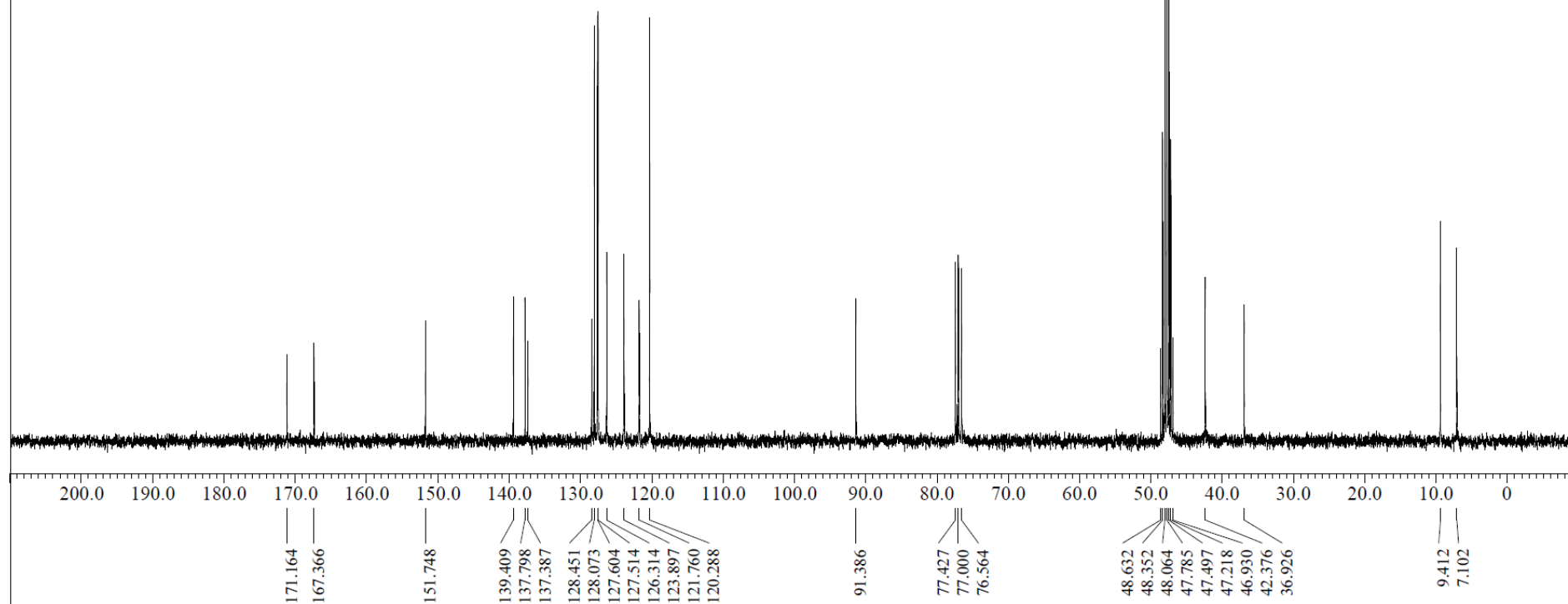
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



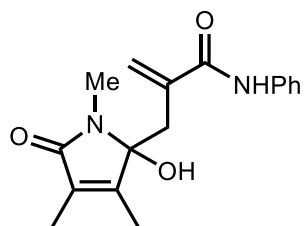


6i

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

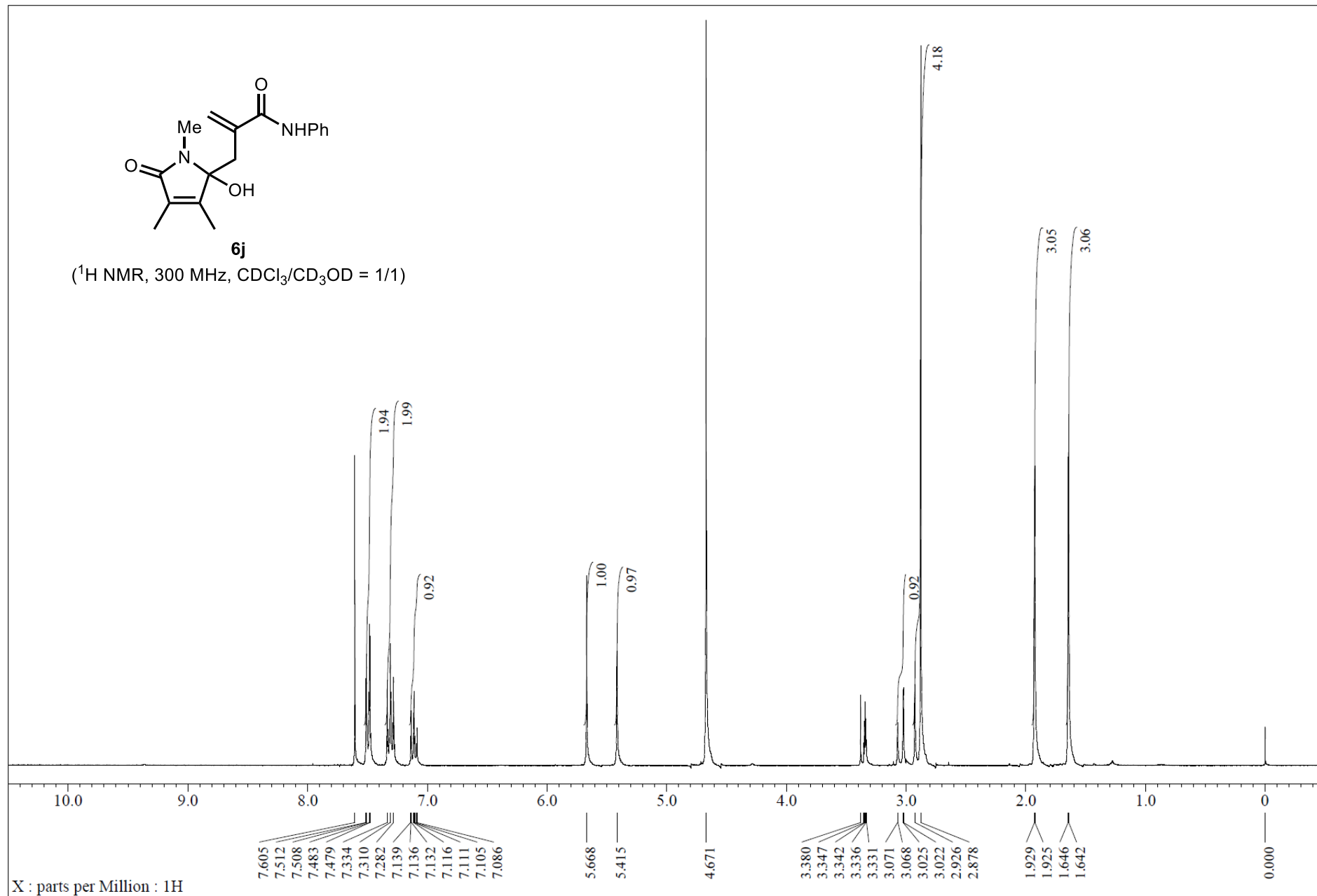


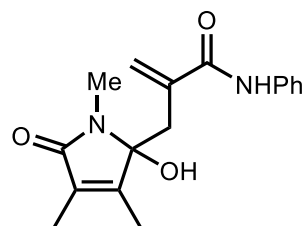
X : parts per Million : ^{13}C



6j

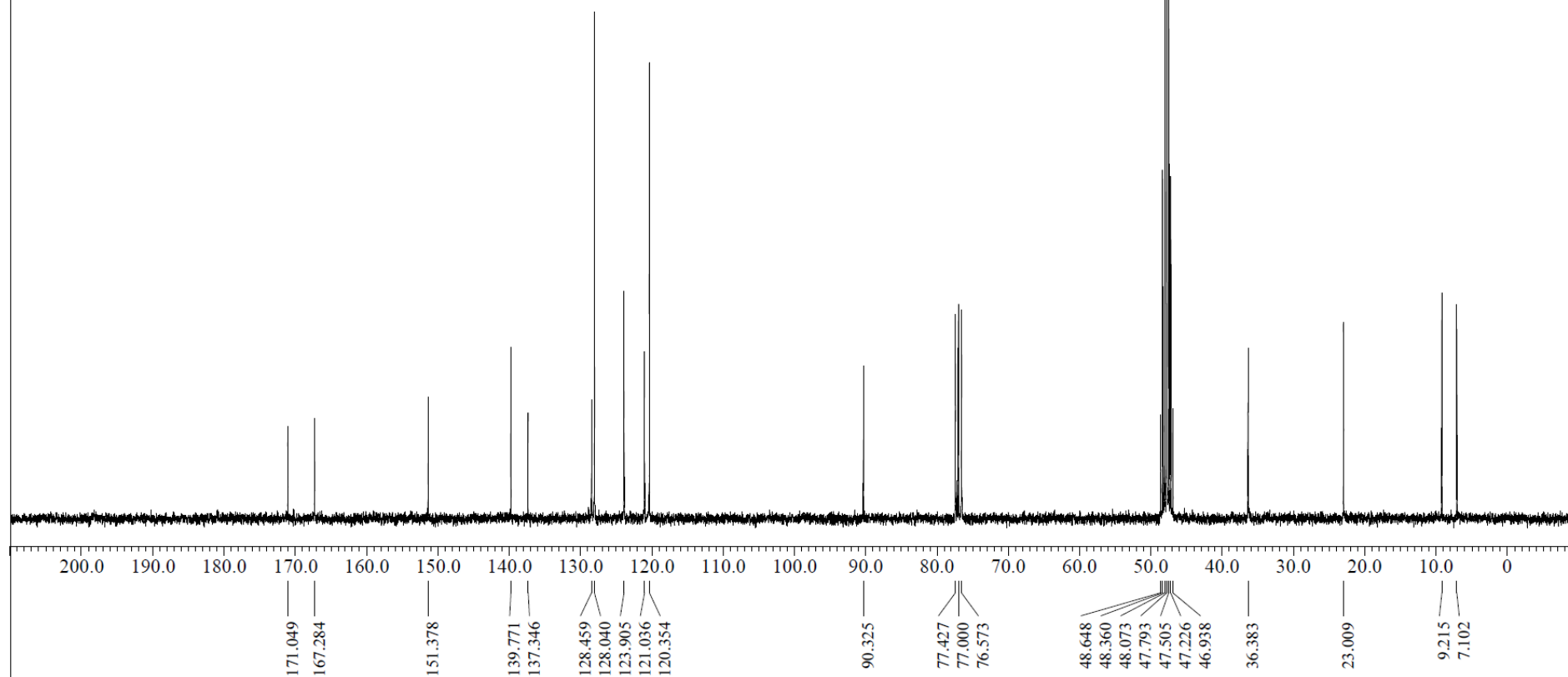
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



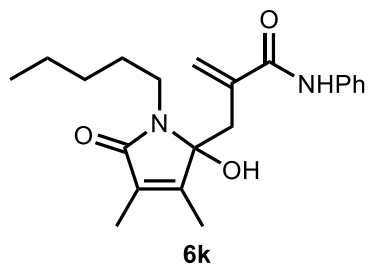


6j

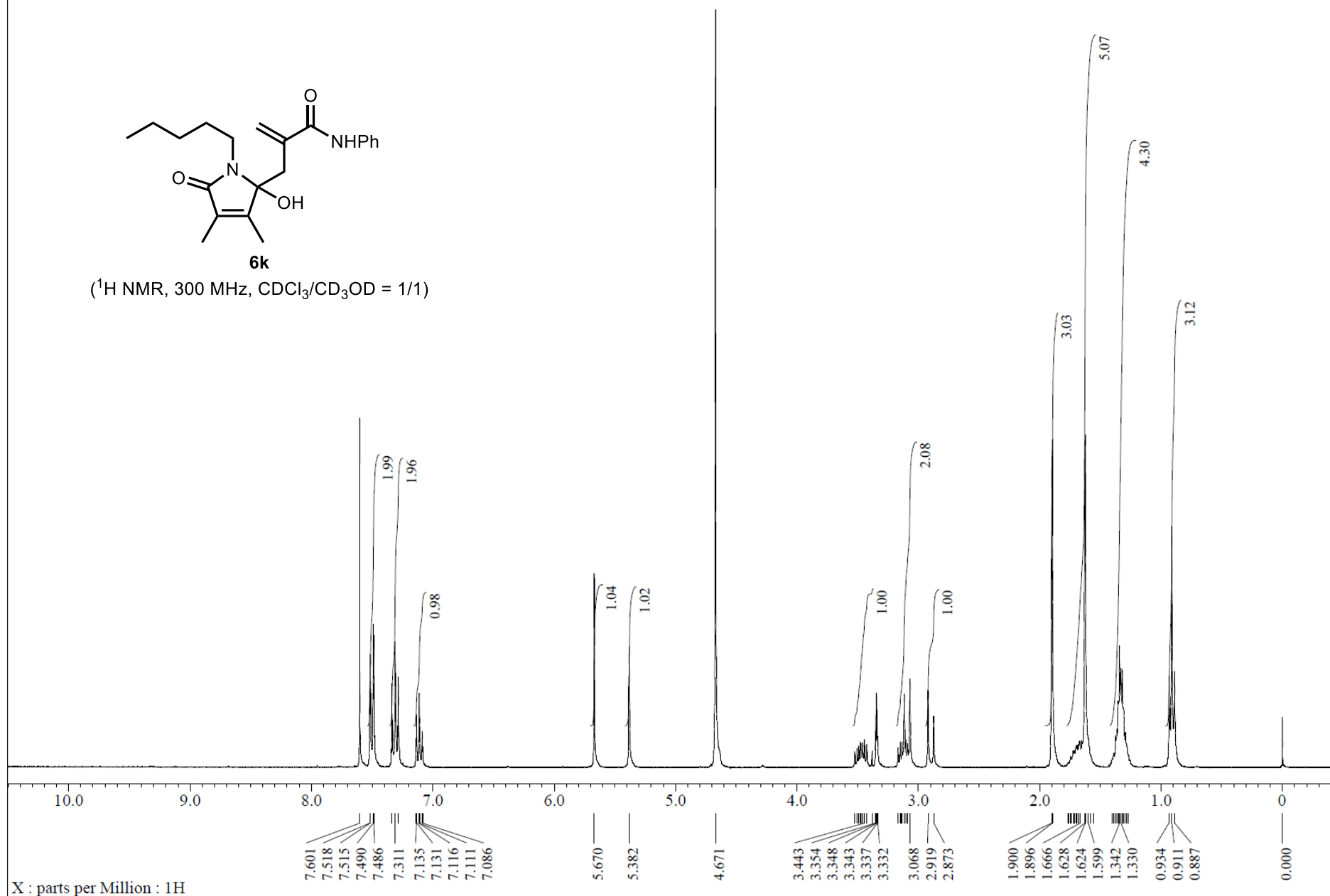
(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

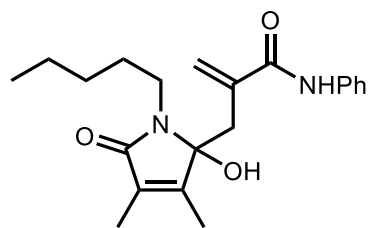


X : parts per Million : ^{13}C



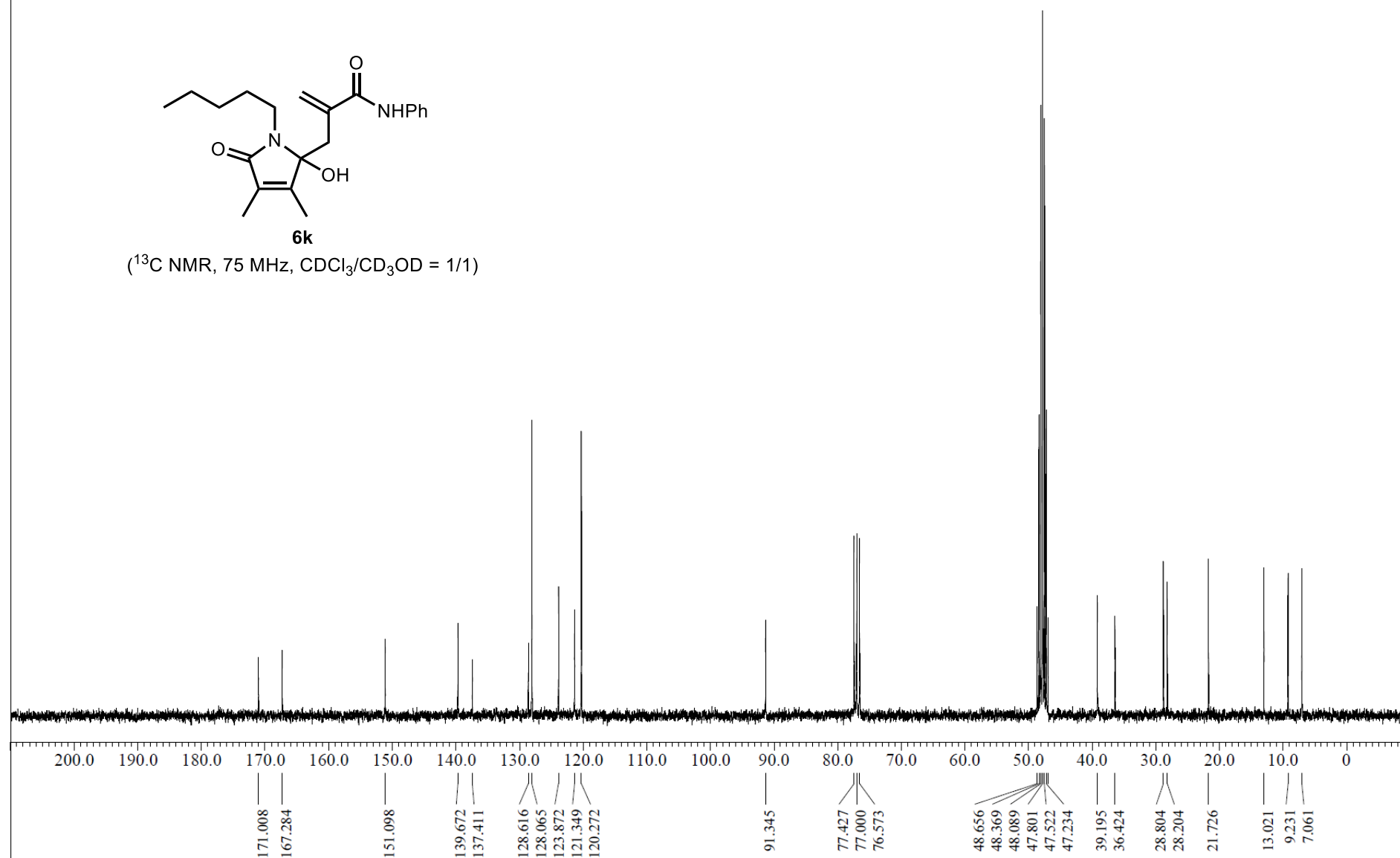
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



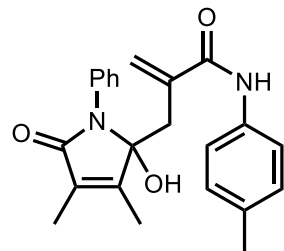


6k

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

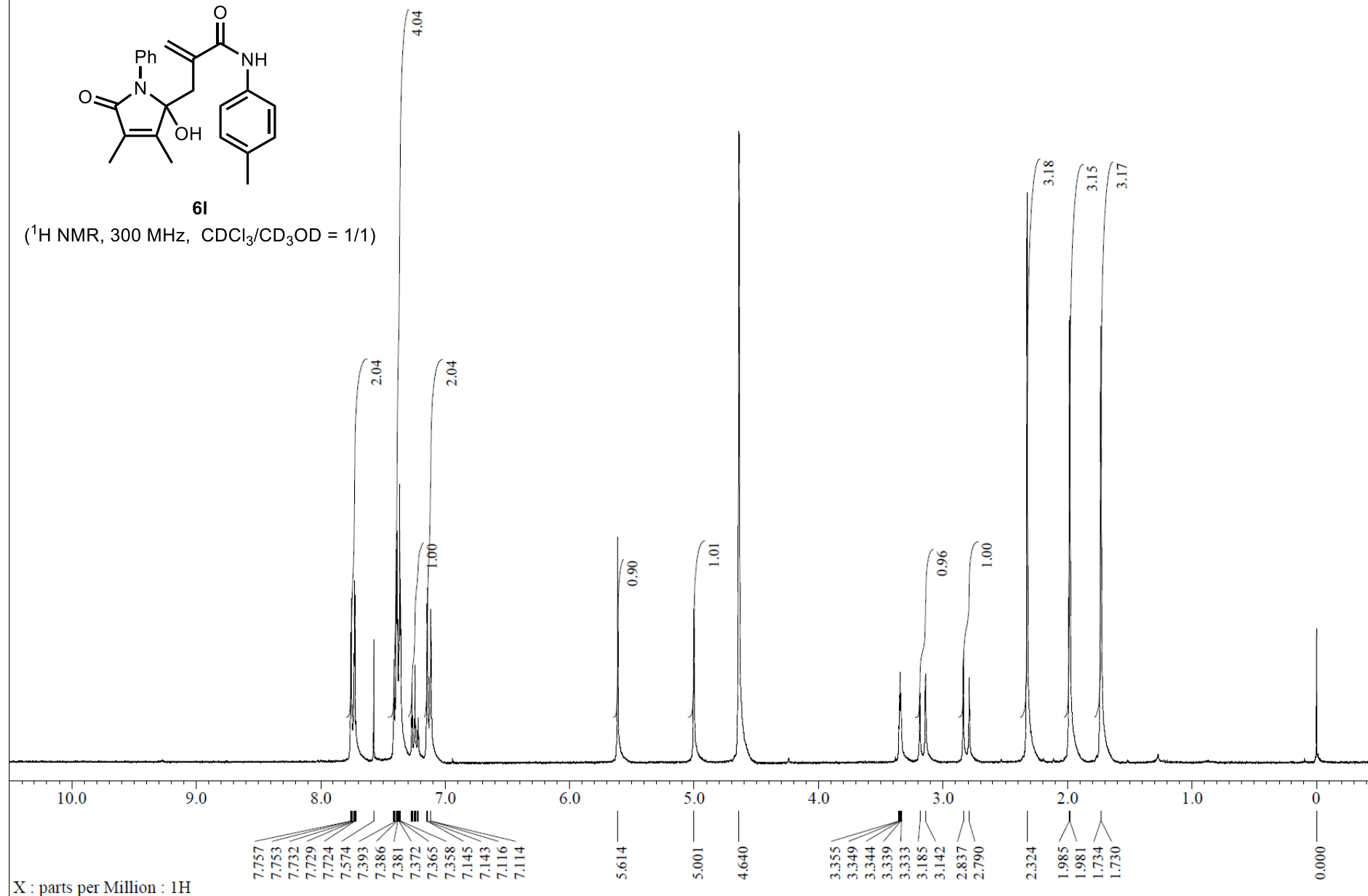


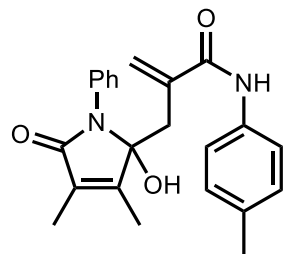
X : parts per Million : ^{13}C



6l

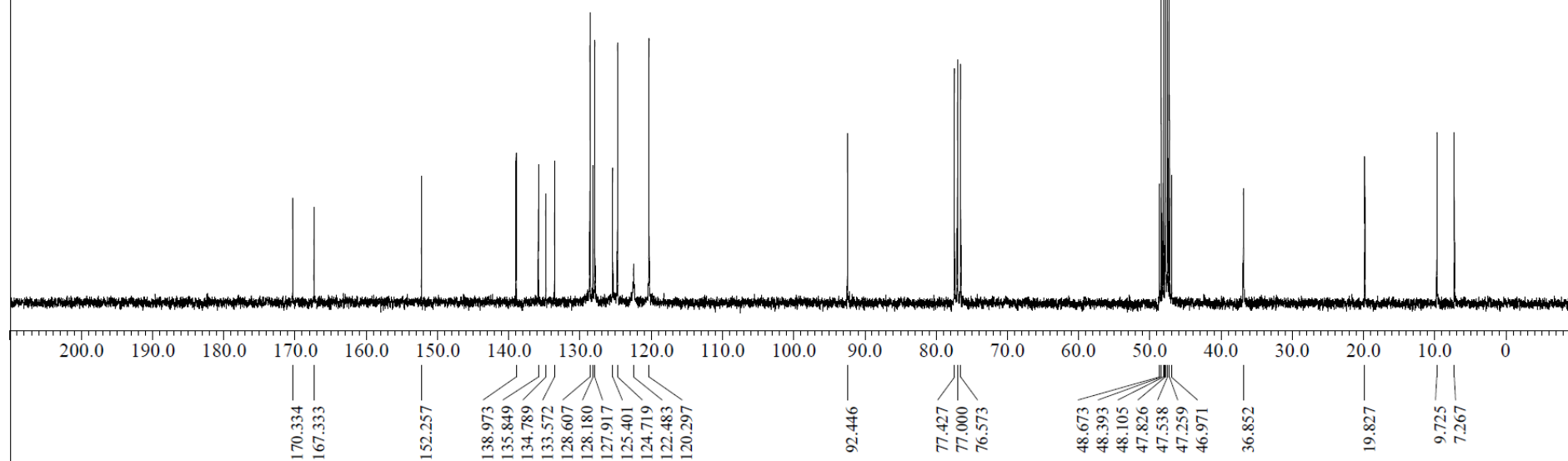
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



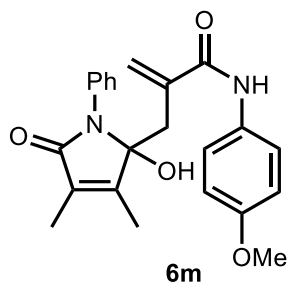


6I

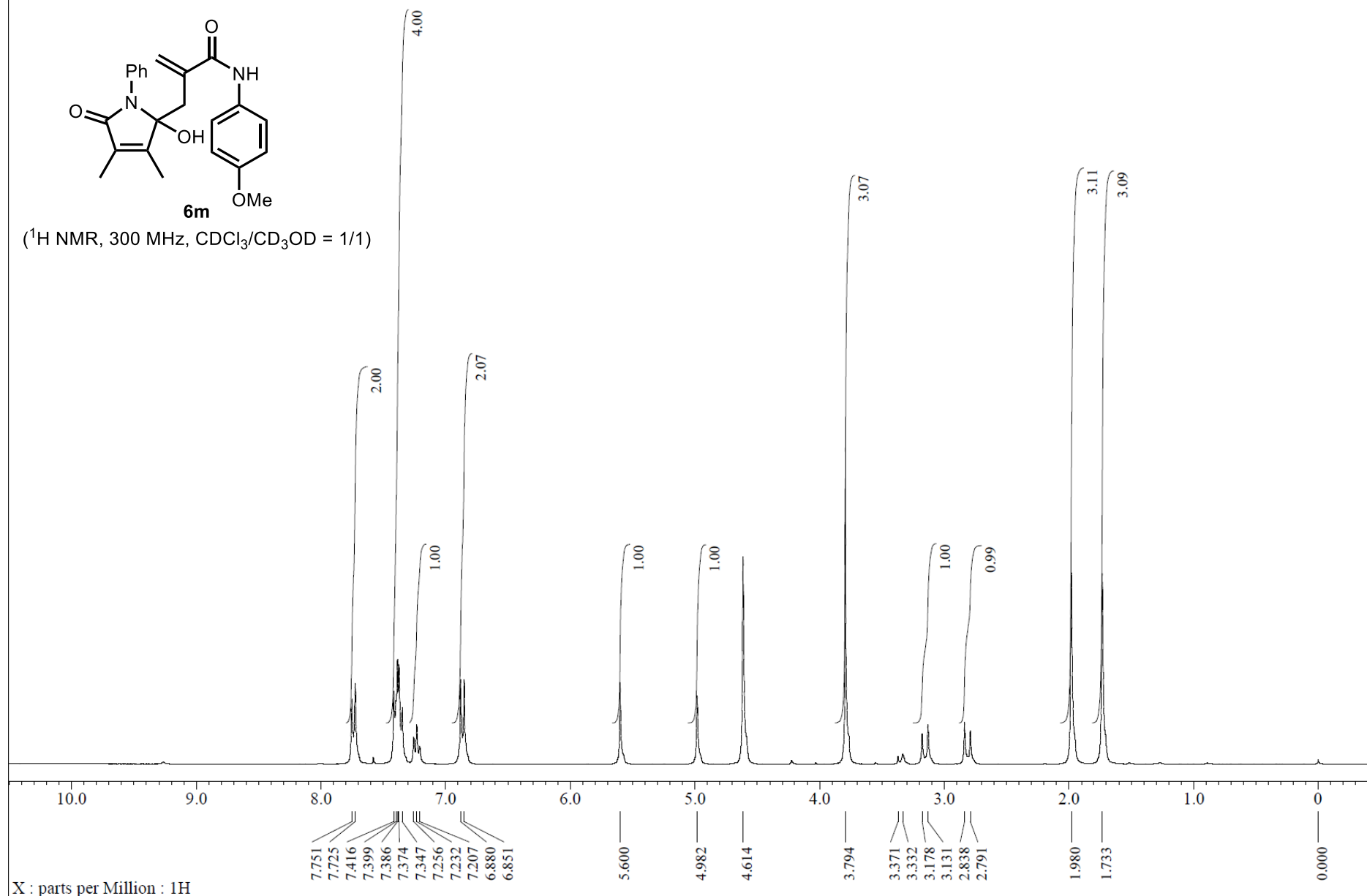
(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

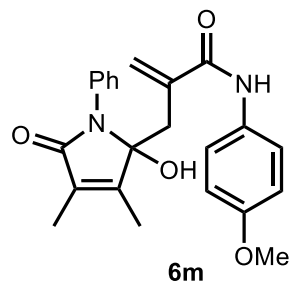


X : parts per Million : ^{13}C

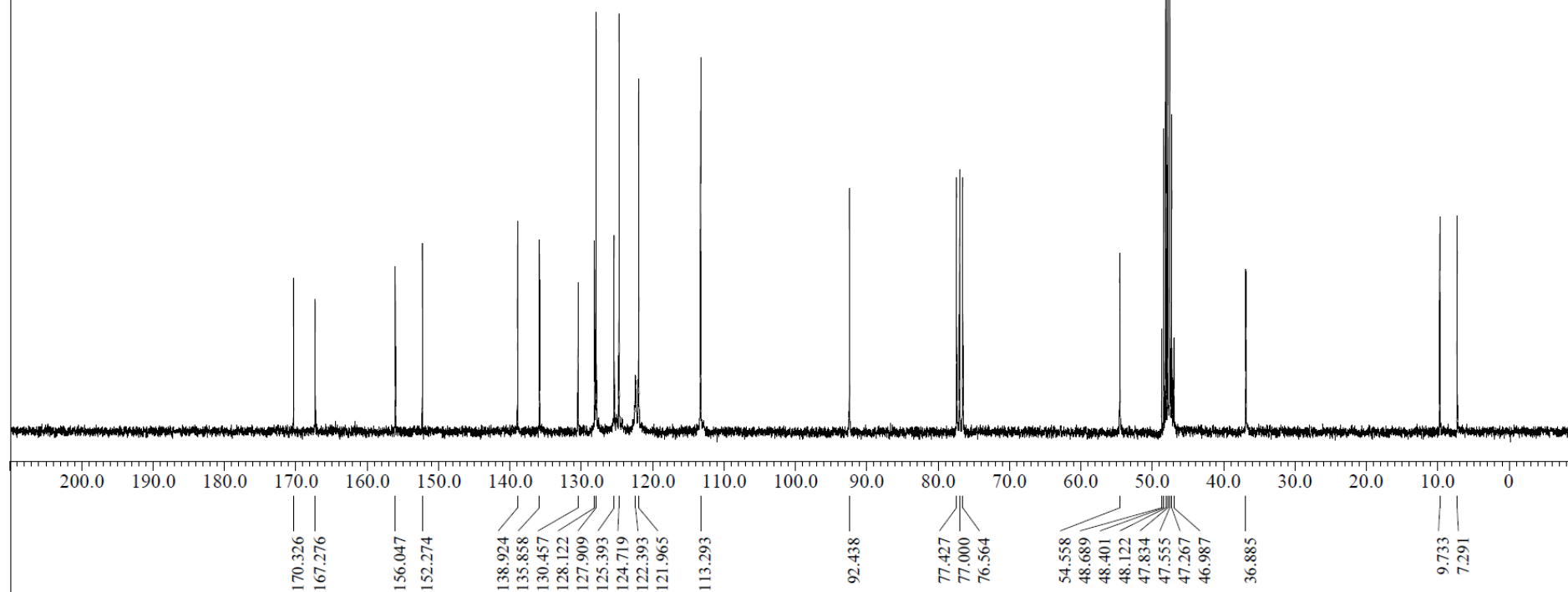


(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

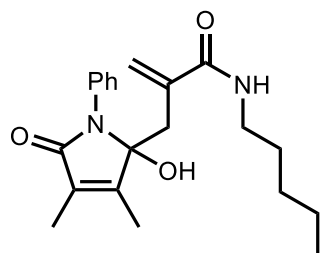




(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

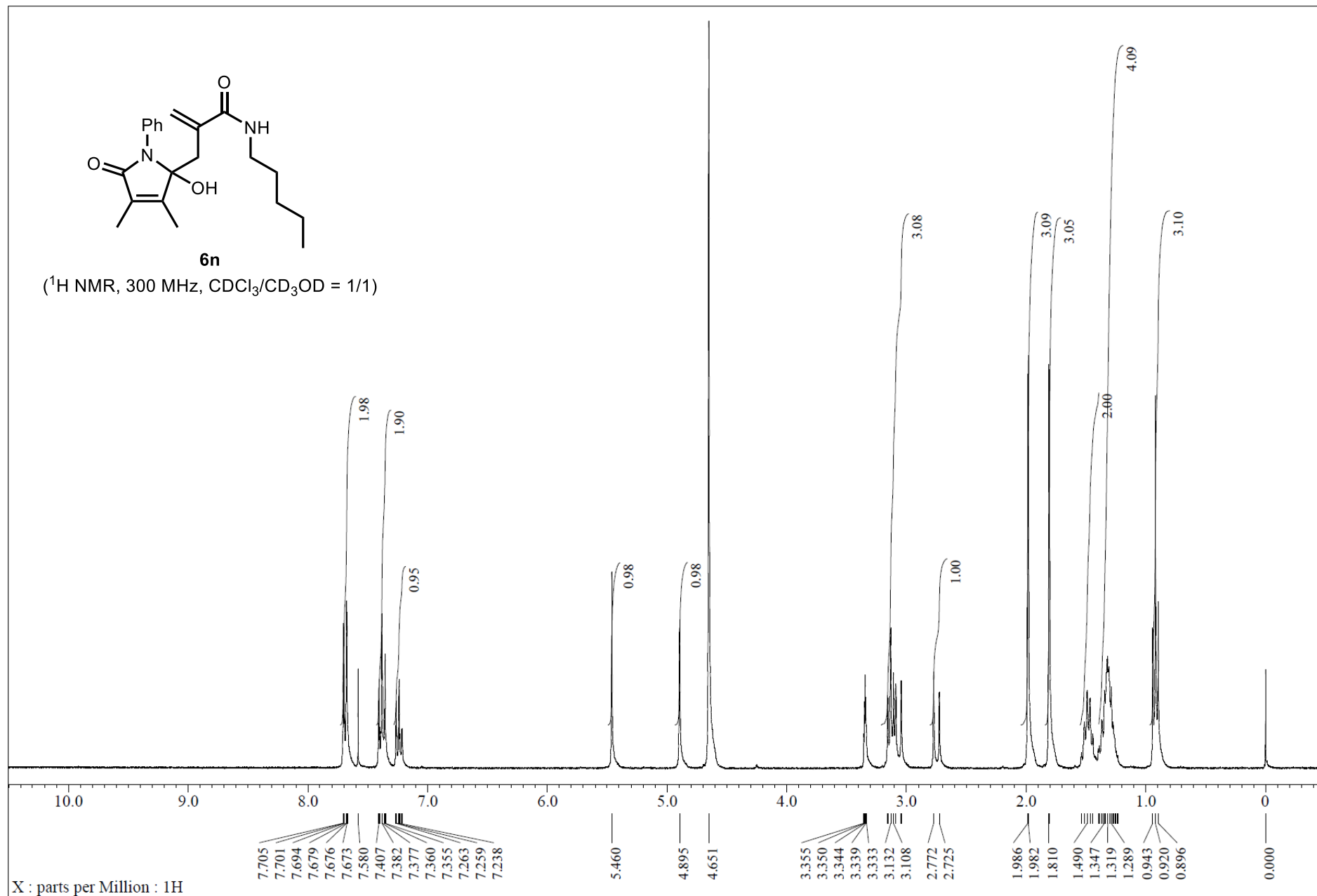


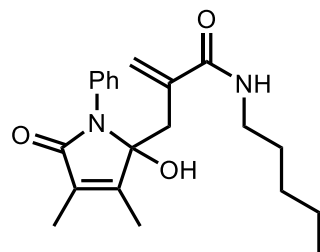
X : parts per Million : ^{13}C



6n

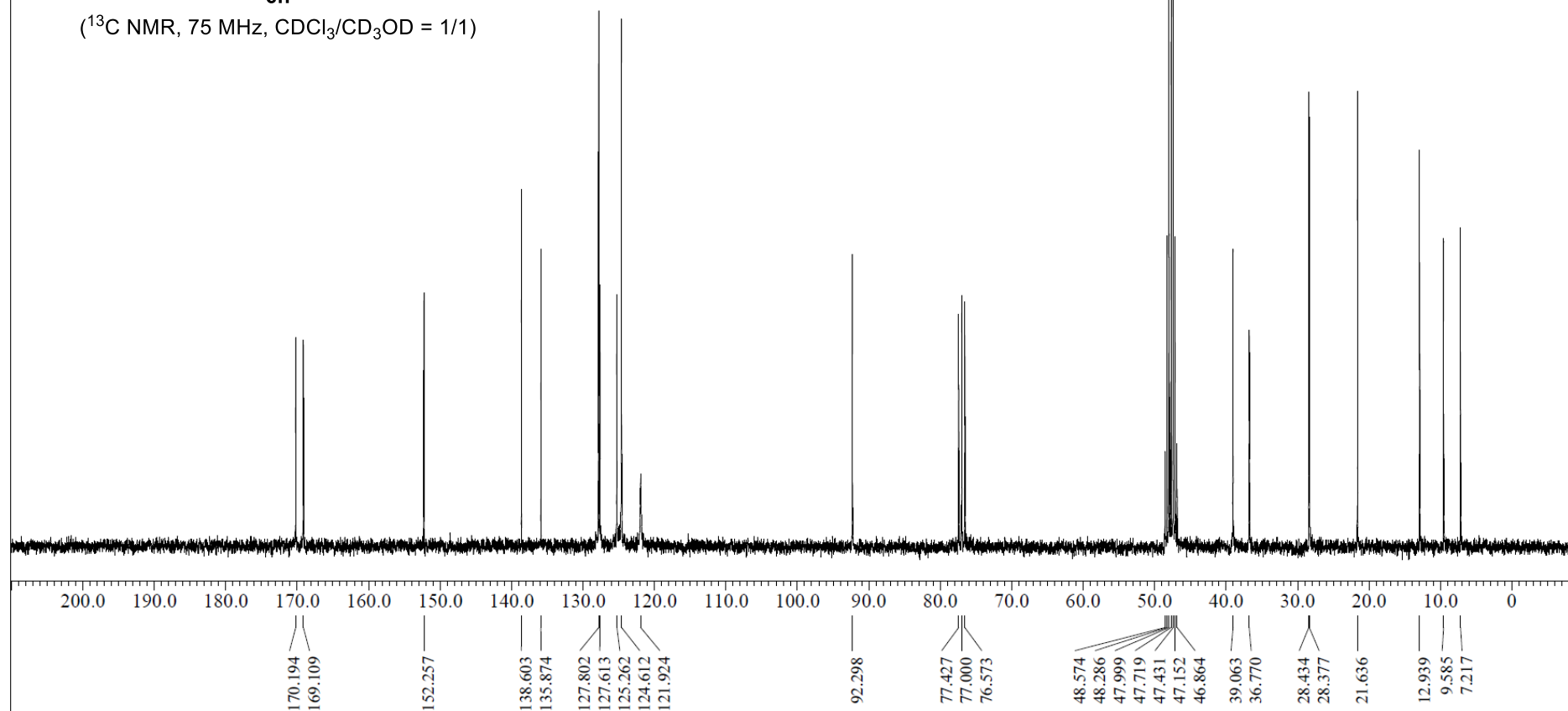
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



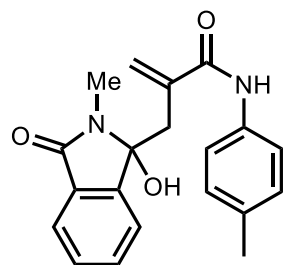


6n

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

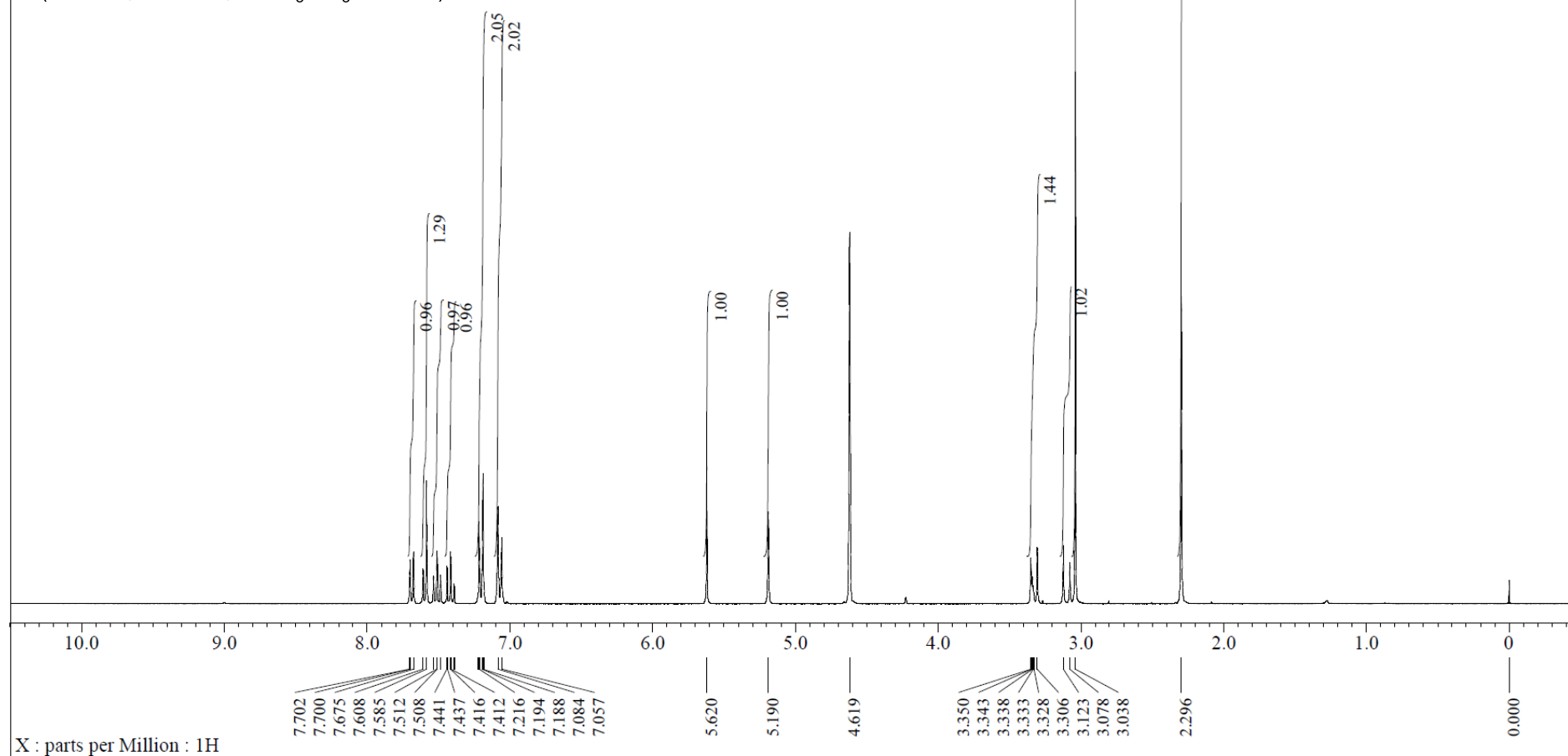


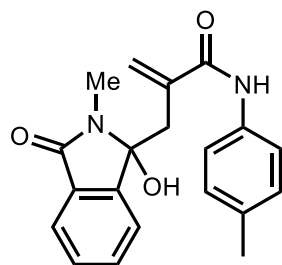
X : parts per Million : ^{13}C



6o

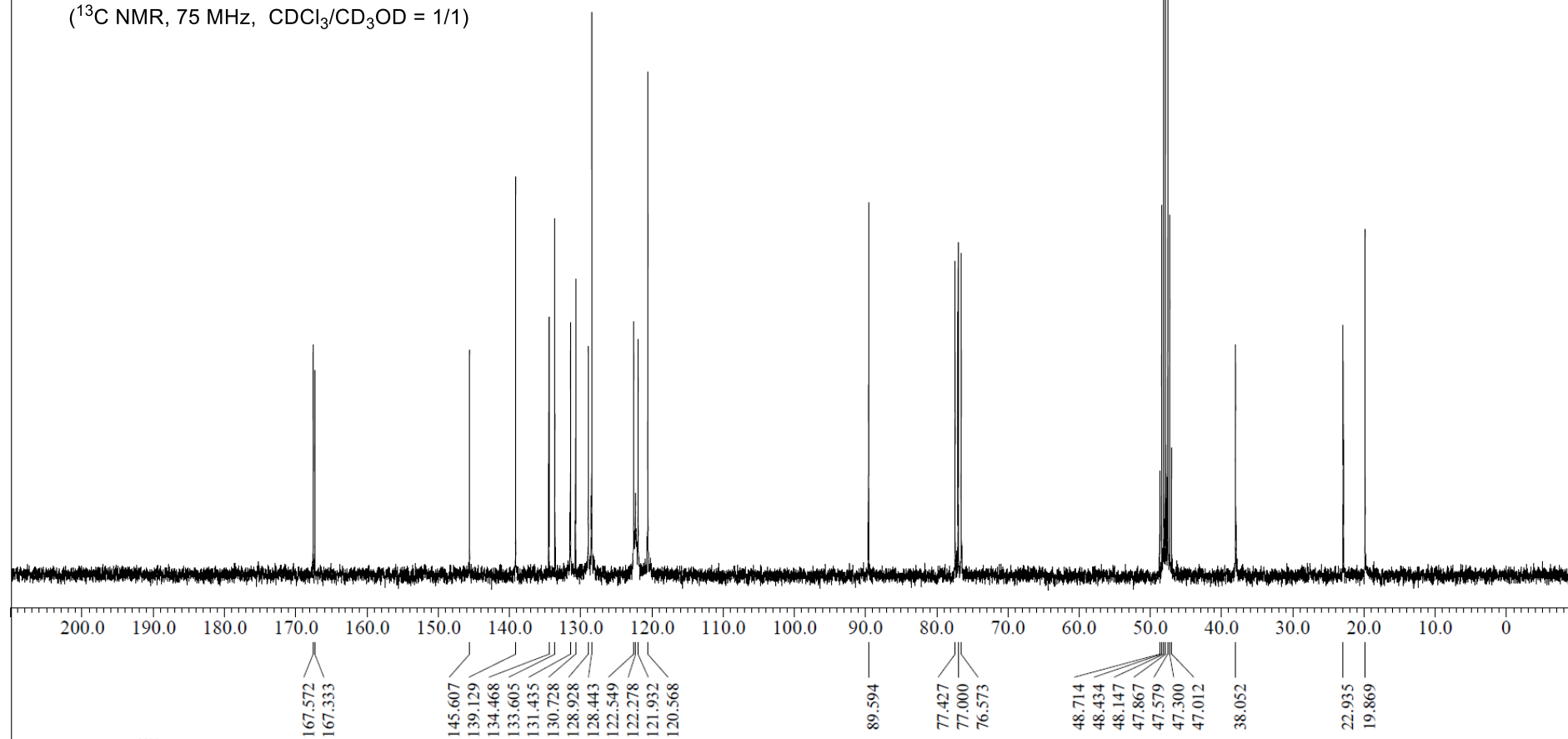
(^1H NMR, 300 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)



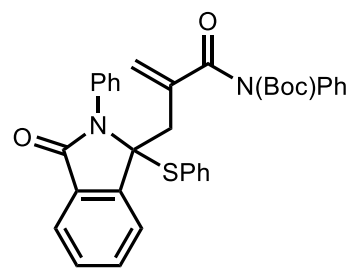


6o

(^{13}C NMR, 75 MHz, $\text{CDCl}_3/\text{CD}_3\text{OD} = 1/1$)

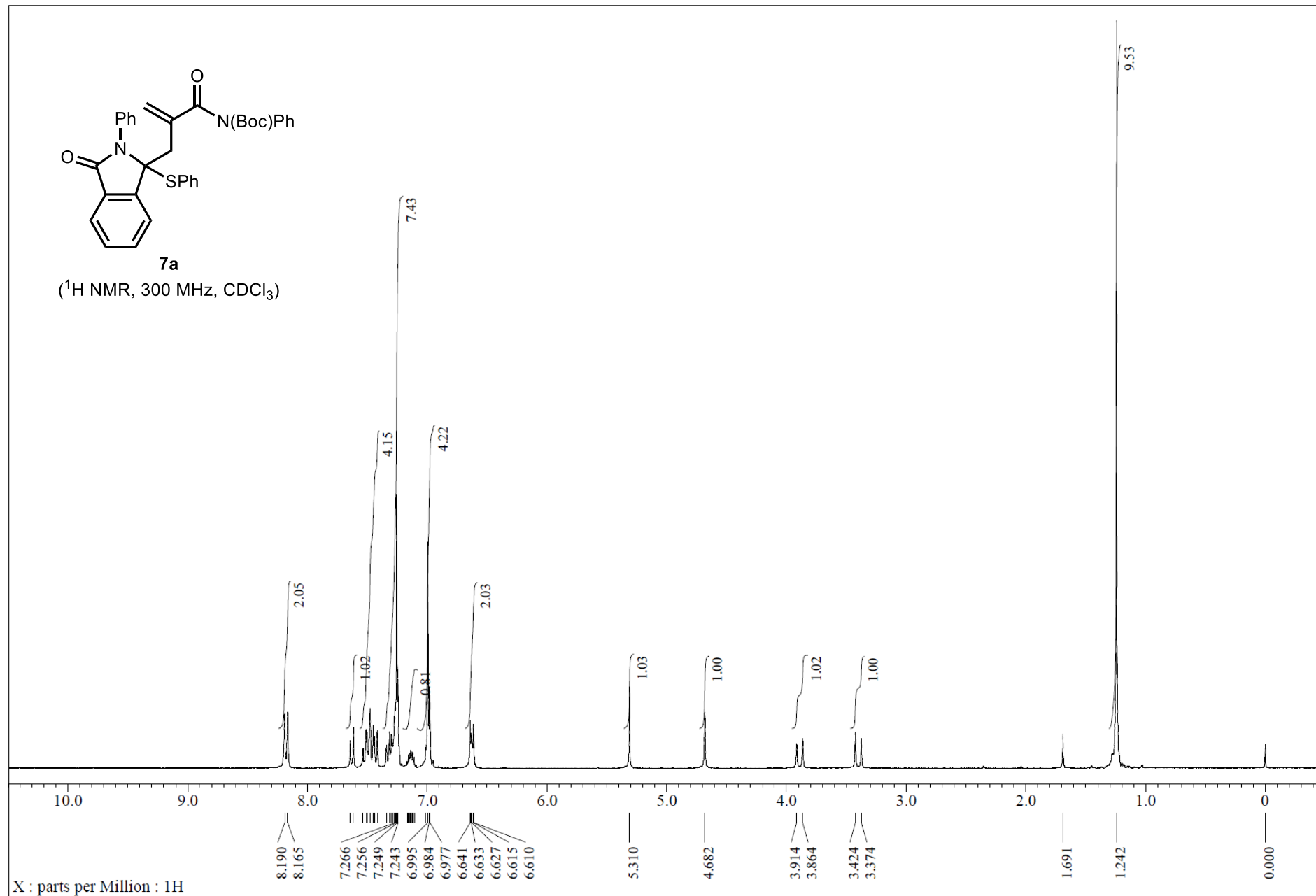


X : parts per Million : ^{13}C

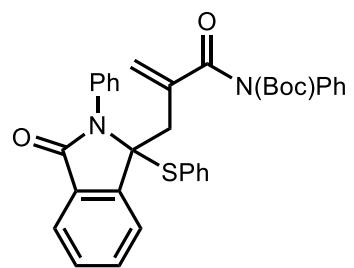


7a

(¹H NMR, 300 MHz, CDCl₃)

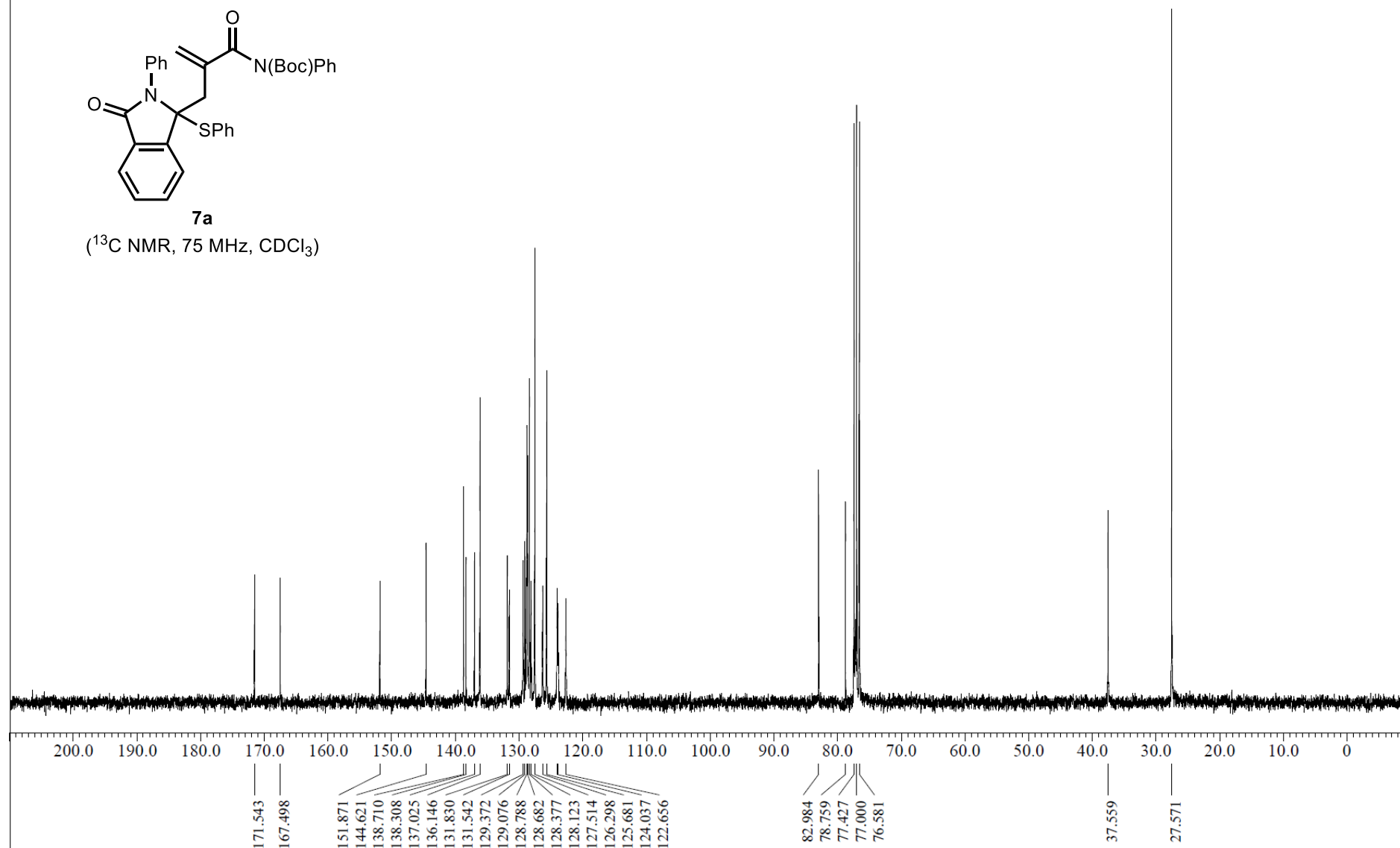


X : parts per Million : 1H

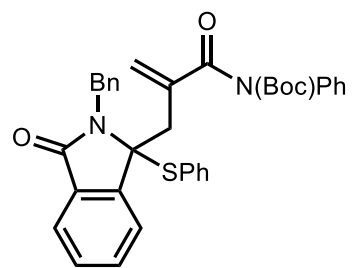


7a

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

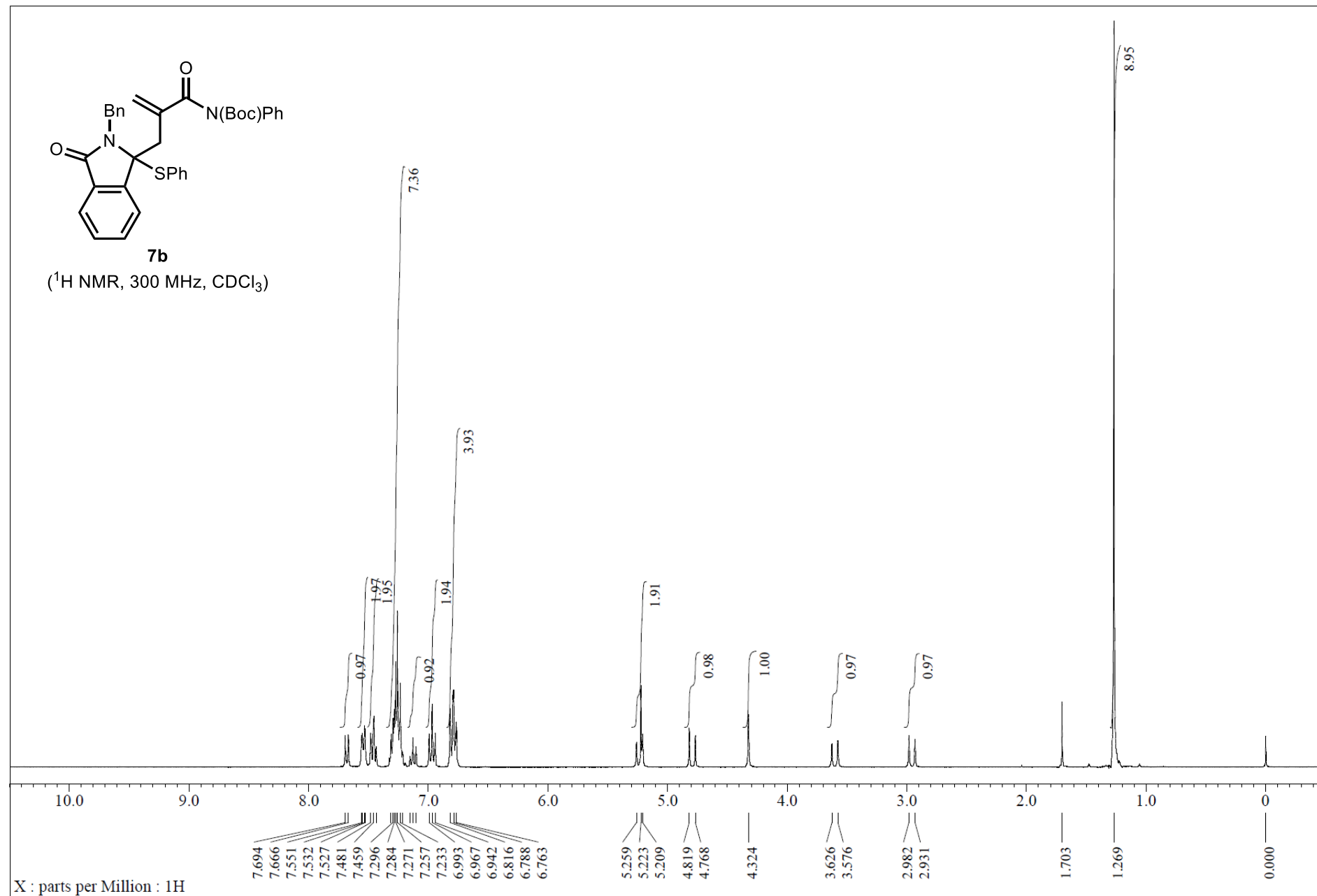


X : parts per Million : ^{13}C

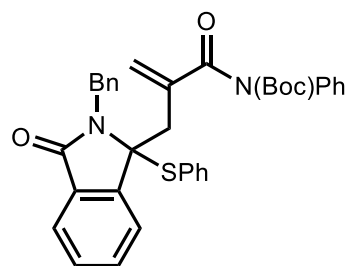


7b

(¹H NMR, 300 MHz, CDCl₃)

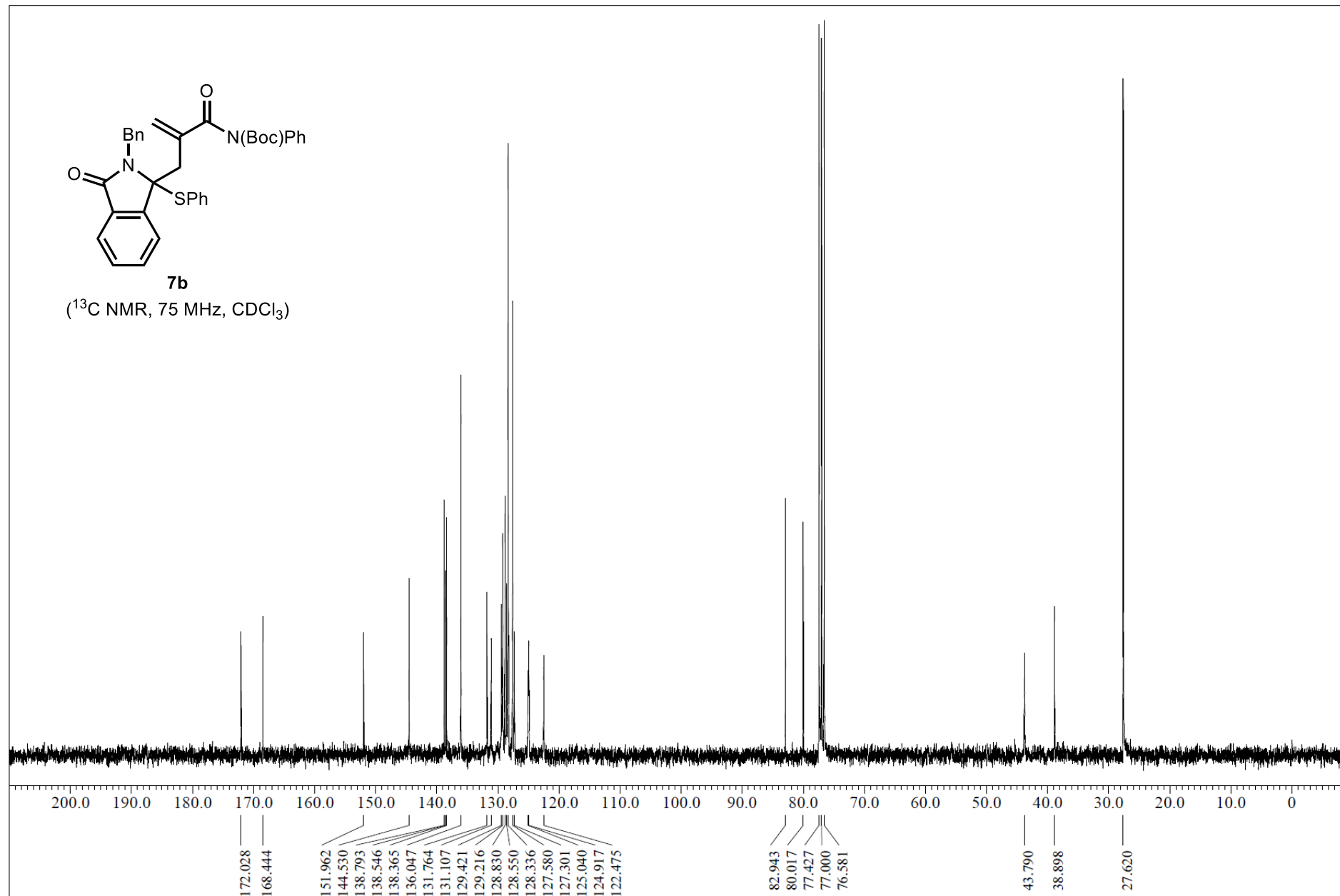


X : parts per Million : 1H

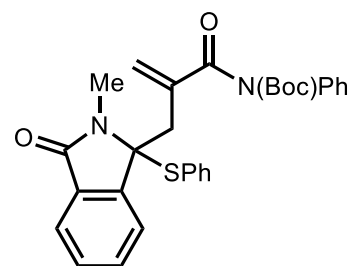


7b

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

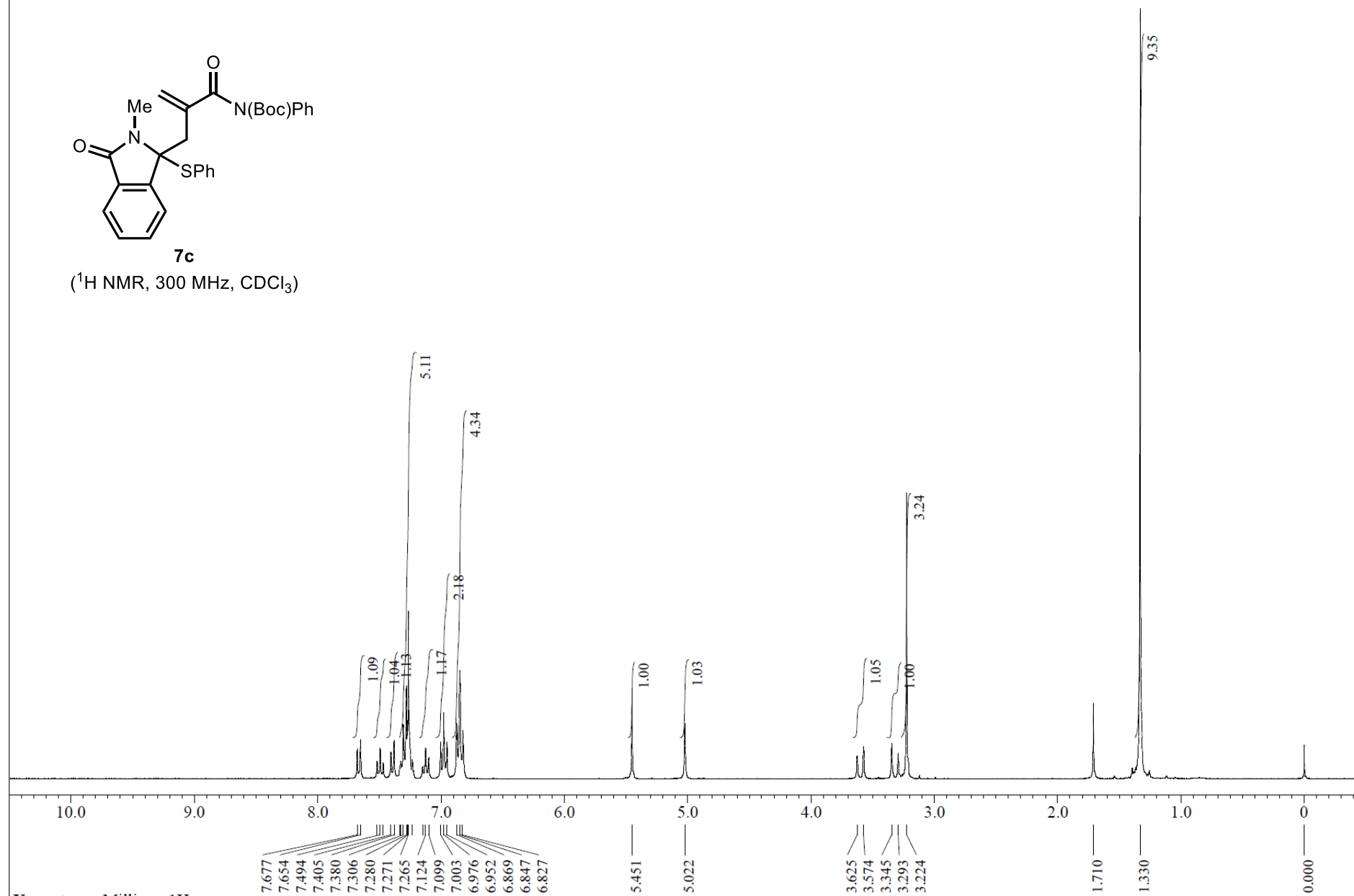


X : parts per Million : ^{13}C

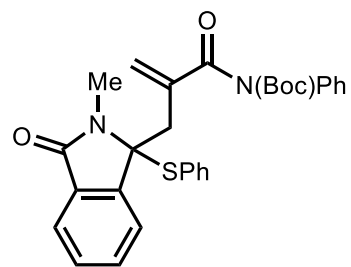


7c

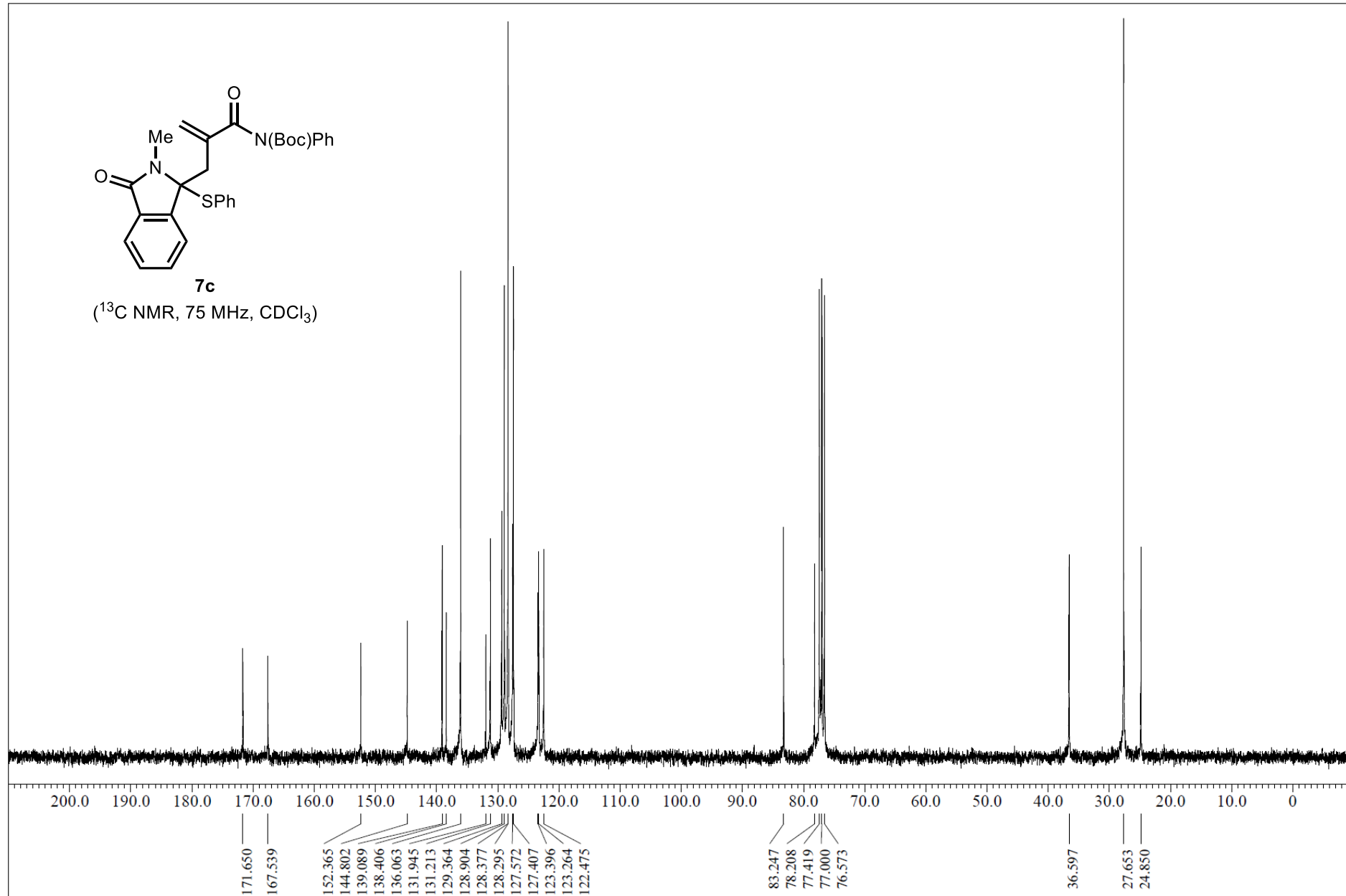
(¹H NMR, 300 MHz, CDCl₃)



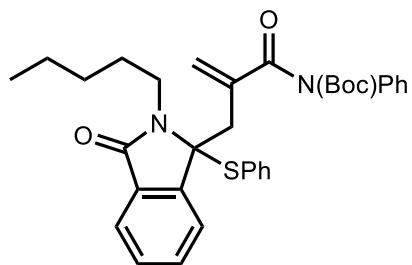
X : parts per Million : 1H



7c
(^{13}C NMR, 75 MHz, CDCl_3)

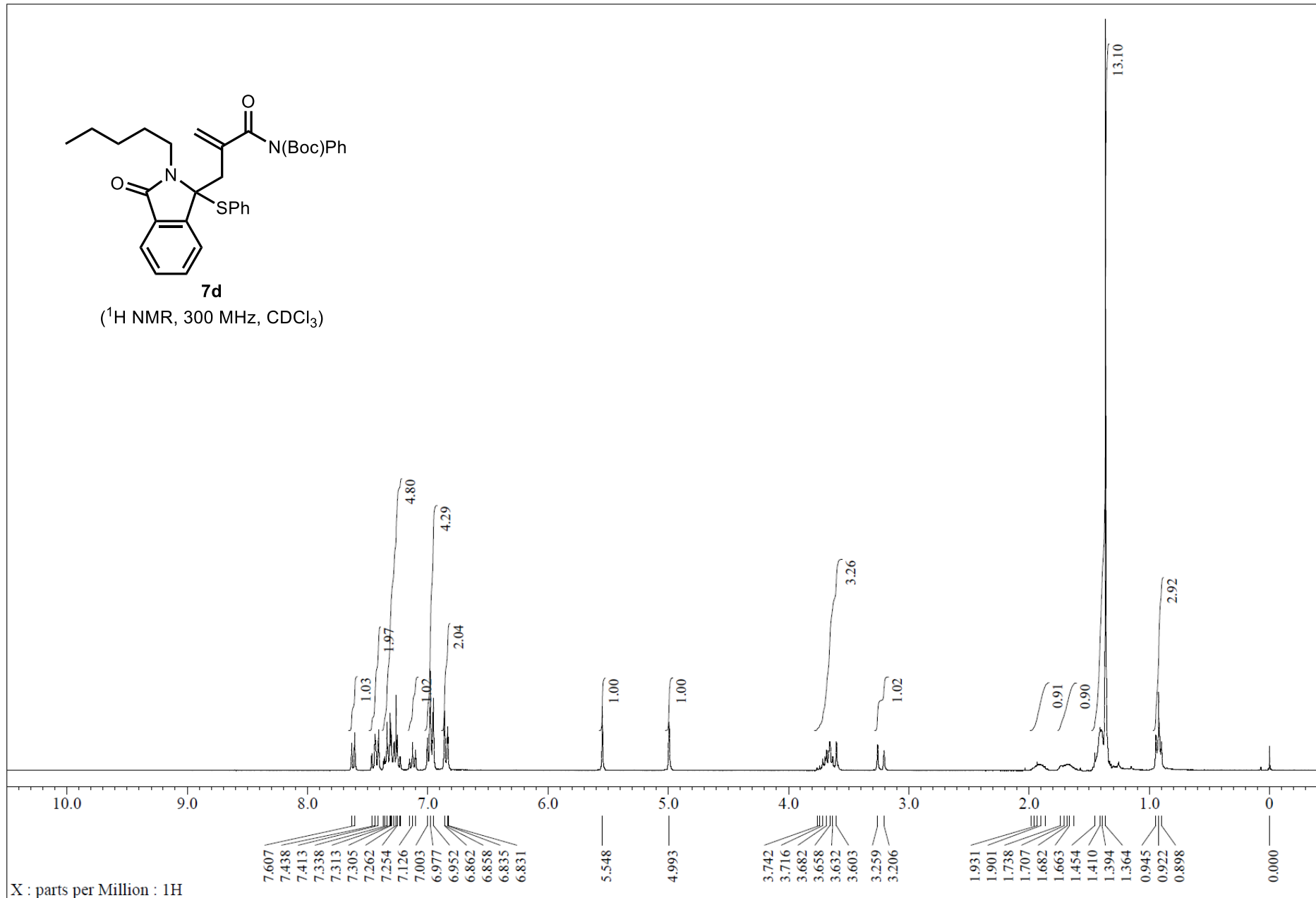


X : parts per Million : ^{13}C

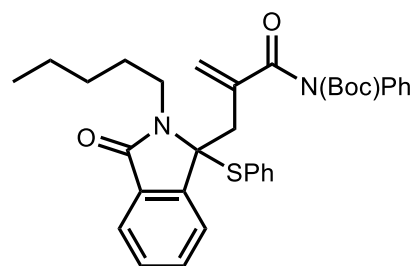


7d

(^1H NMR, 300 MHz, CDCl_3)

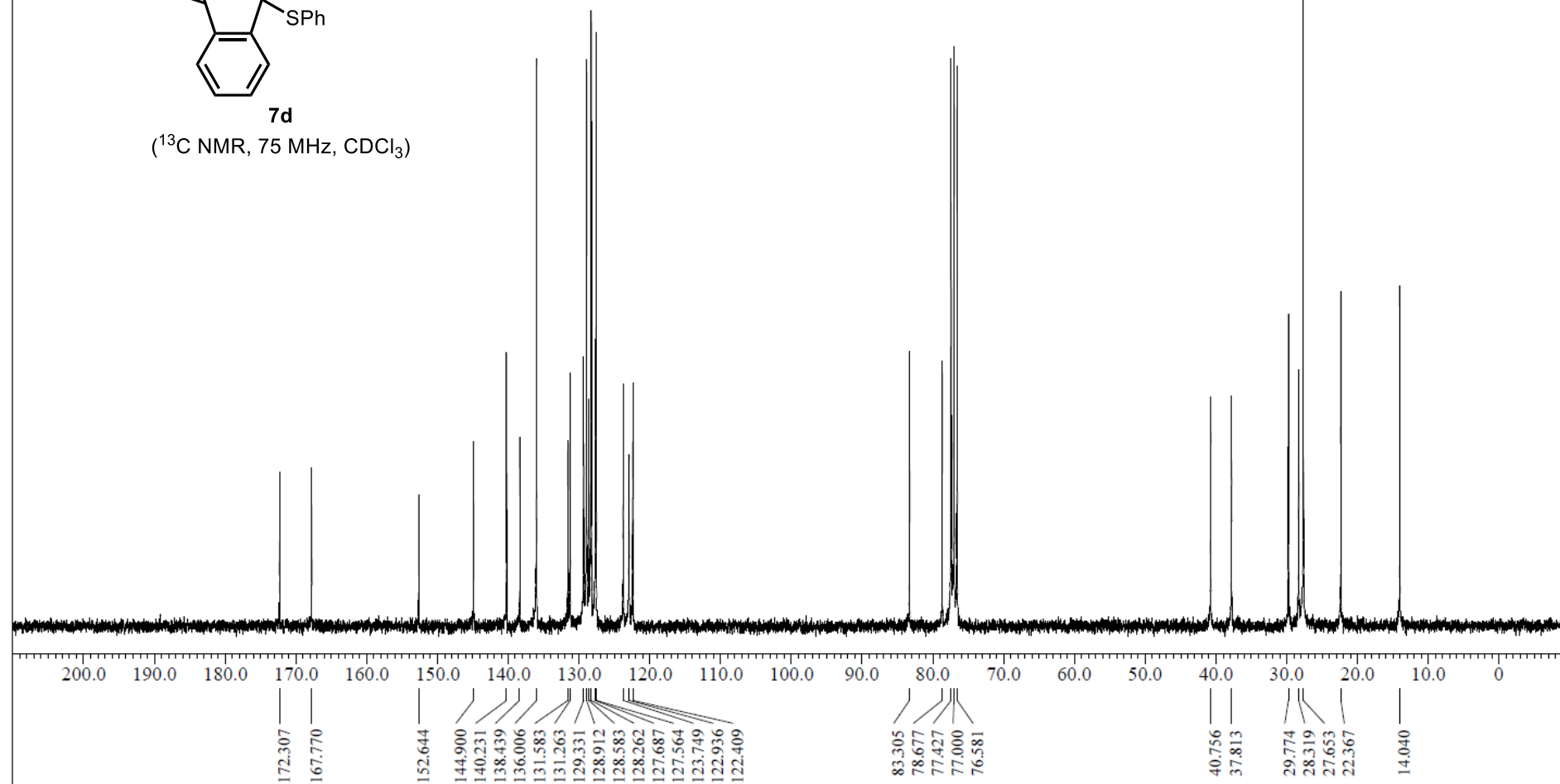


X : parts per Million : ^1H

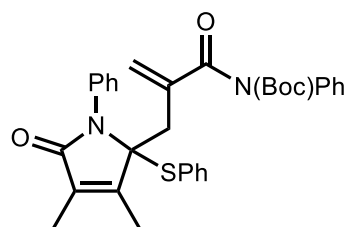


7d

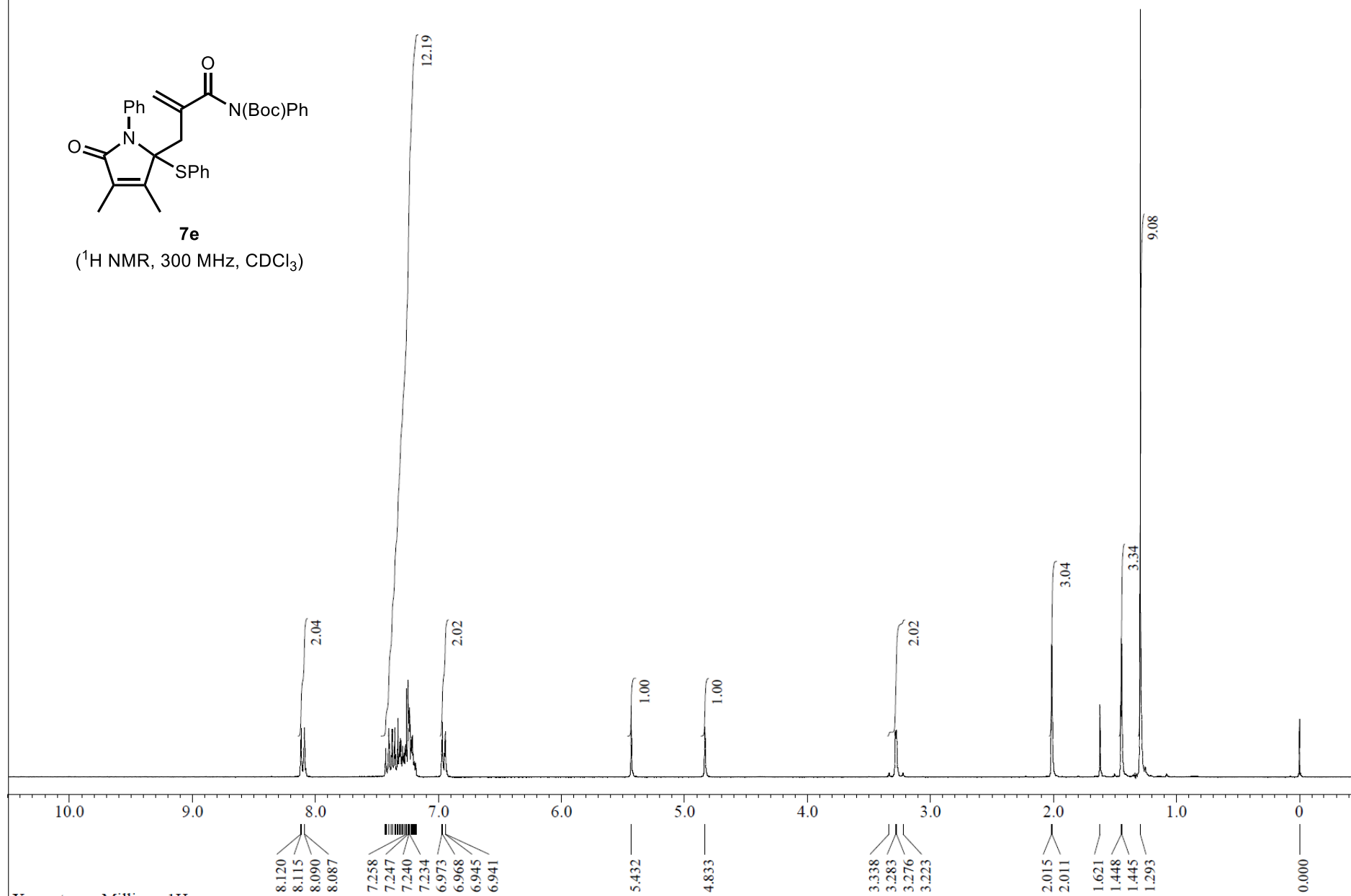
(^{13}C NMR, 75 MHz, CDCl_3)



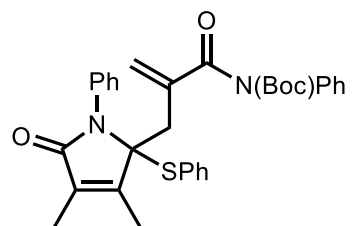
X : parts per Million : ^{13}C



7e
(^1H NMR, 300 MHz, CDCl_3)

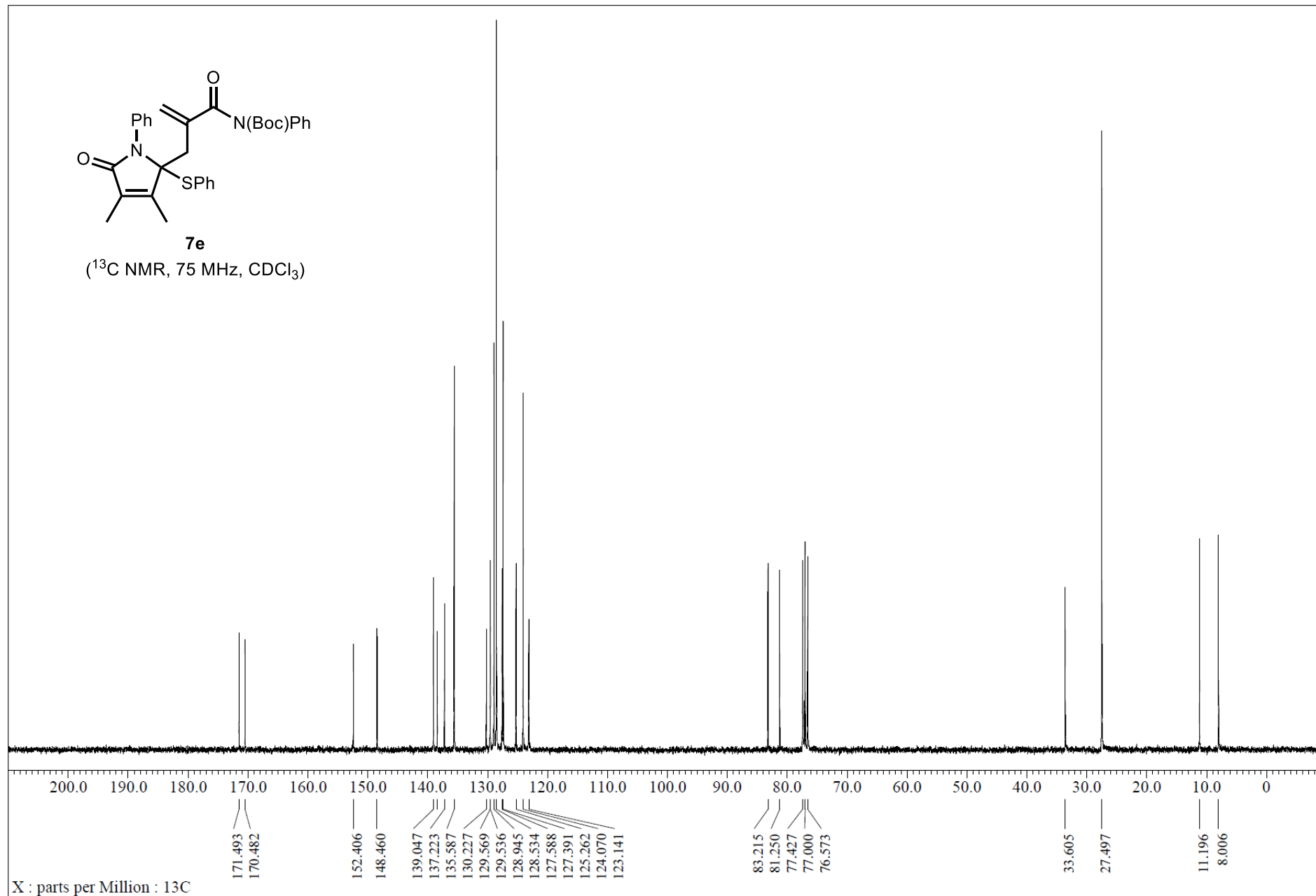


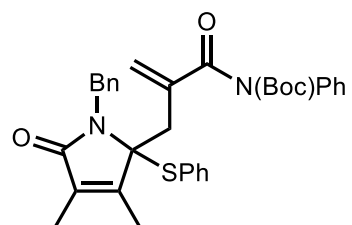
X : parts per Million : ^1H



7e

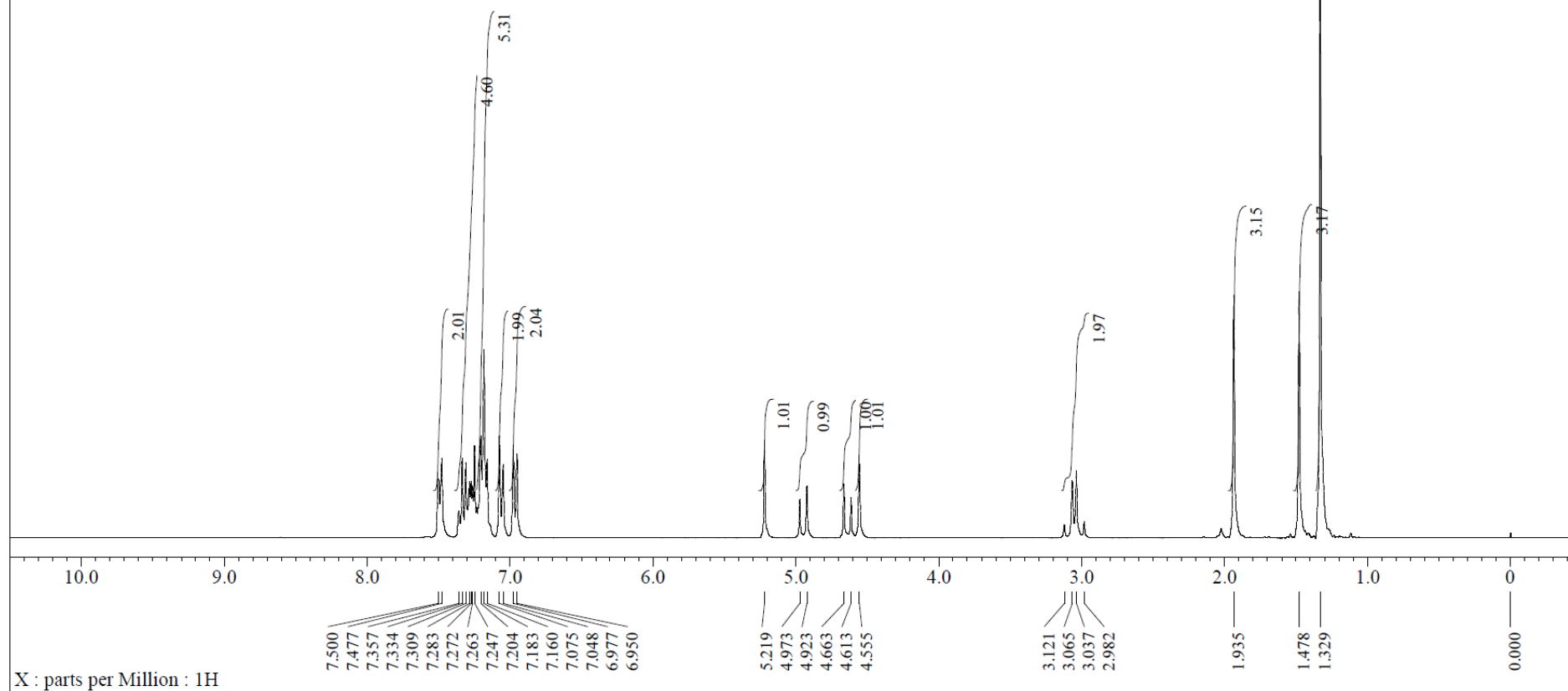
(^{13}C NMR, 75 MHz, CDCl_3)

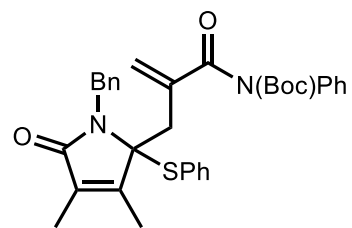




7f

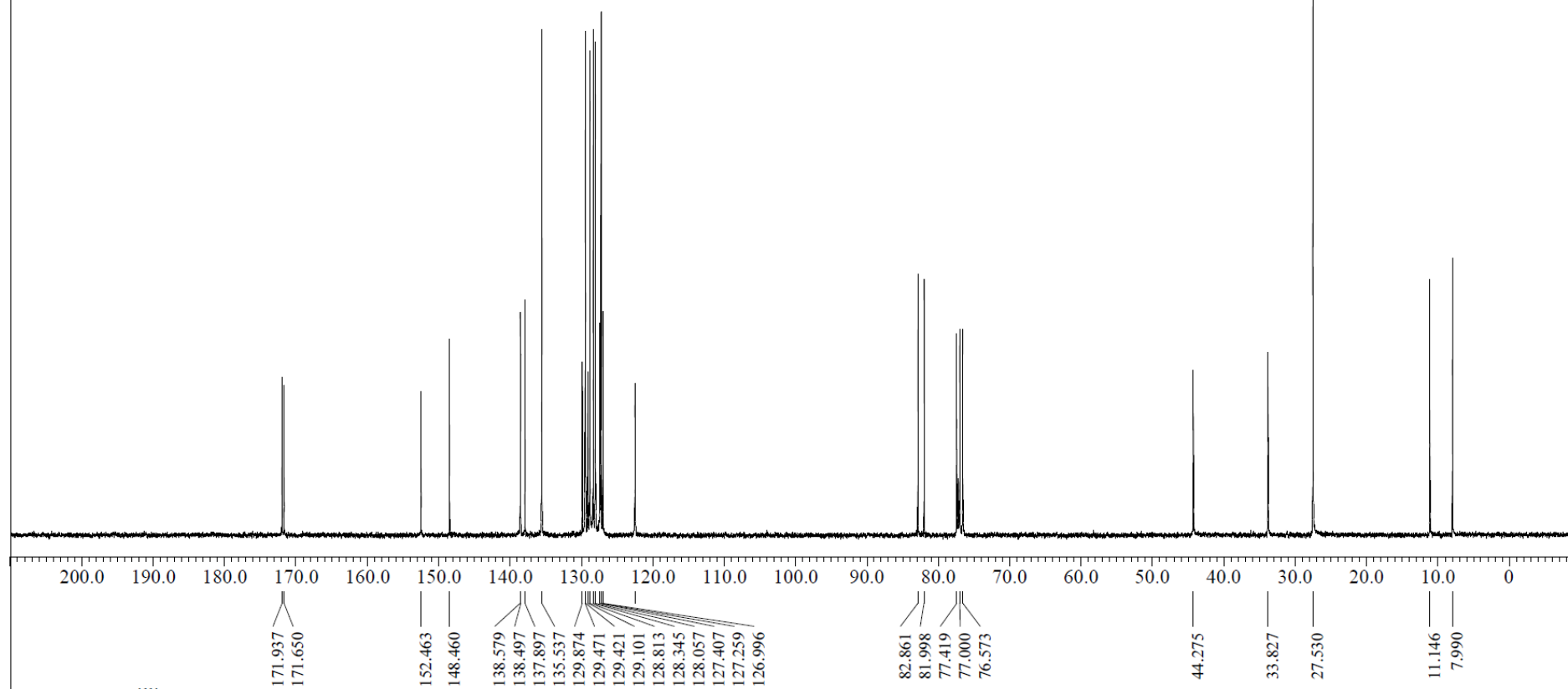
(¹H NMR, 300 MHz, CDCl₃)



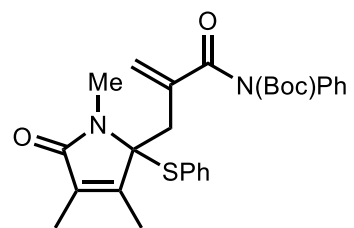


7f

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

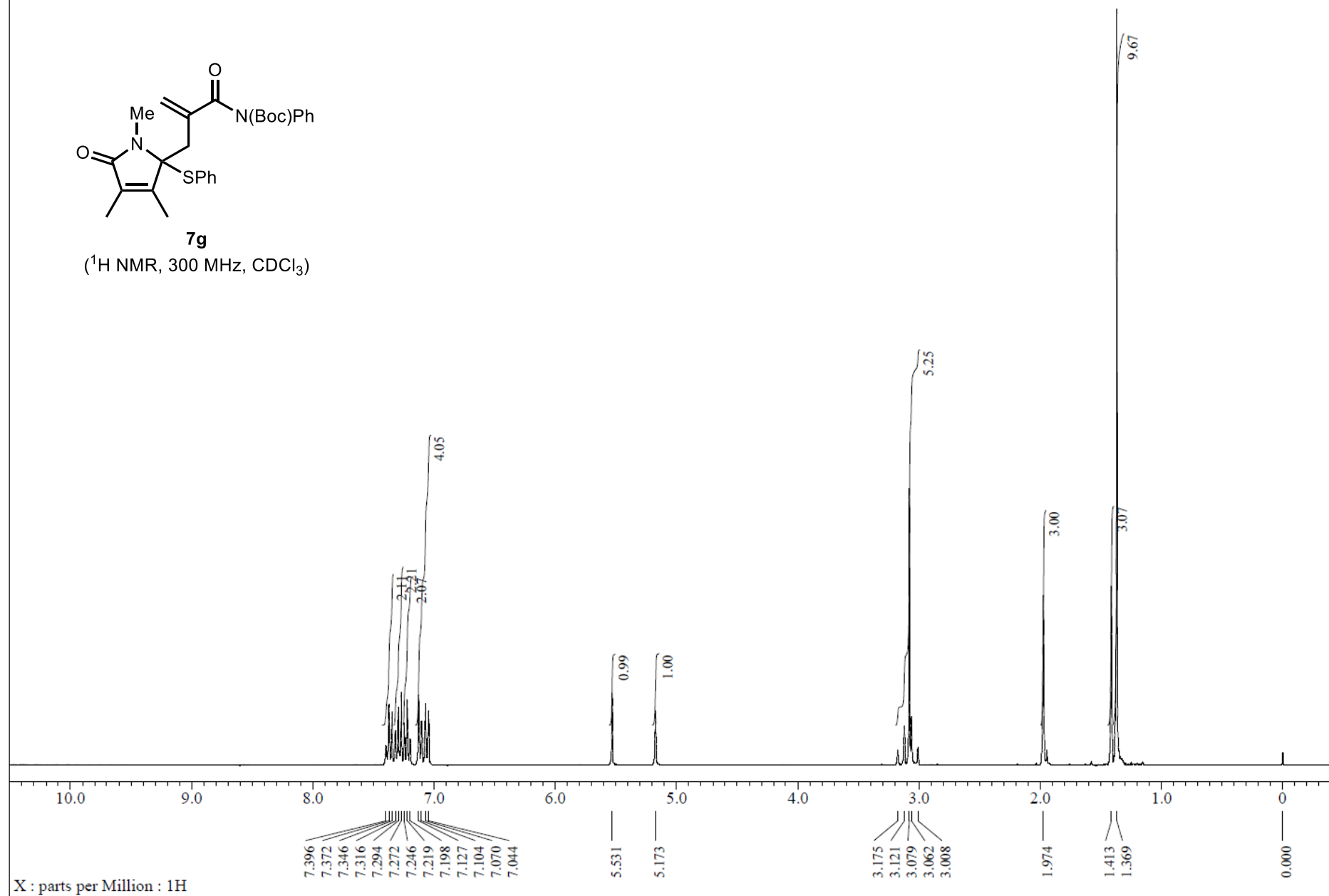


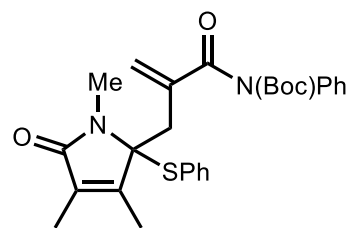
X : parts per Million : ^{13}C



7g

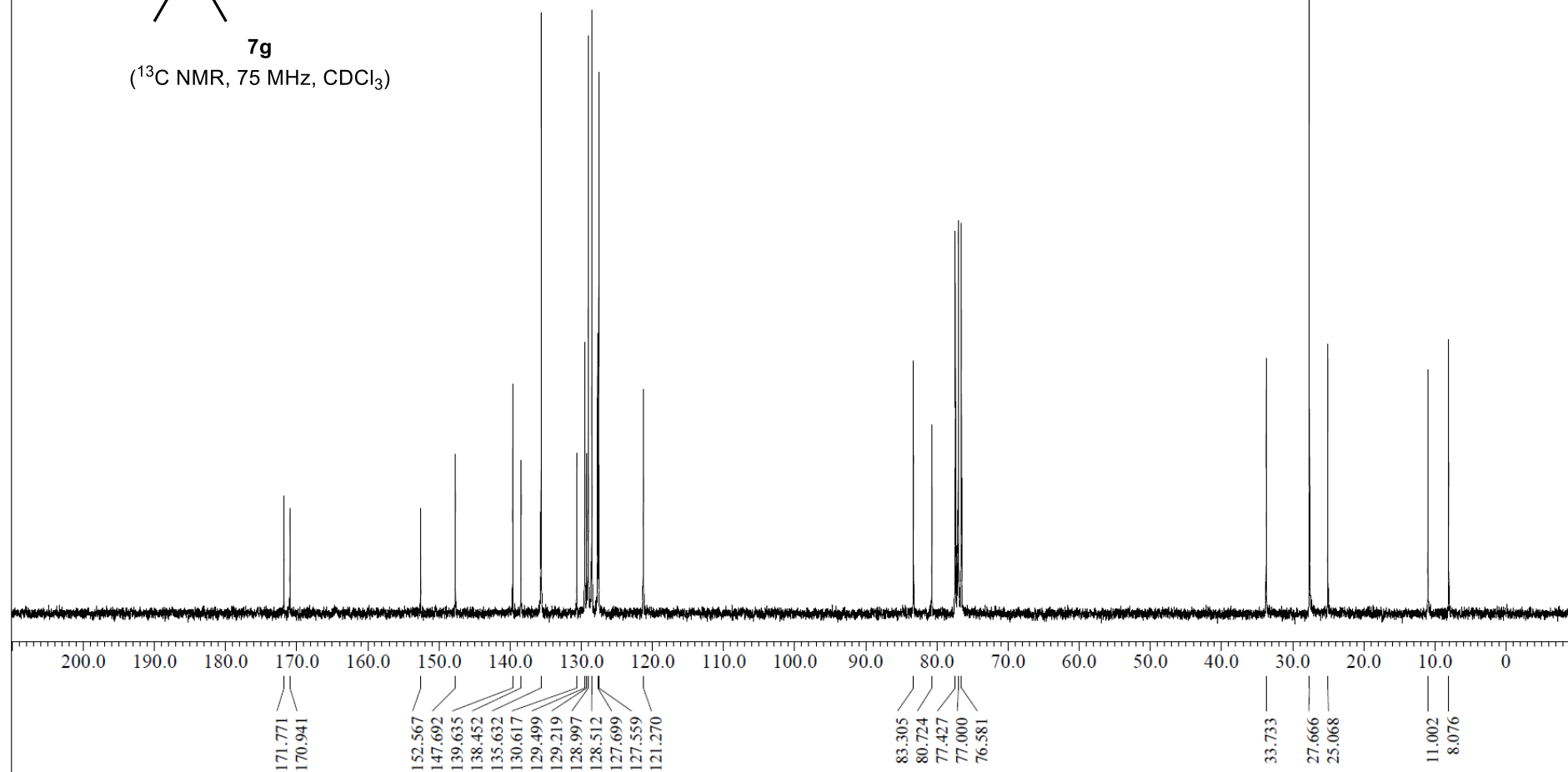
(^1H NMR, 300 MHz, CDCl_3)



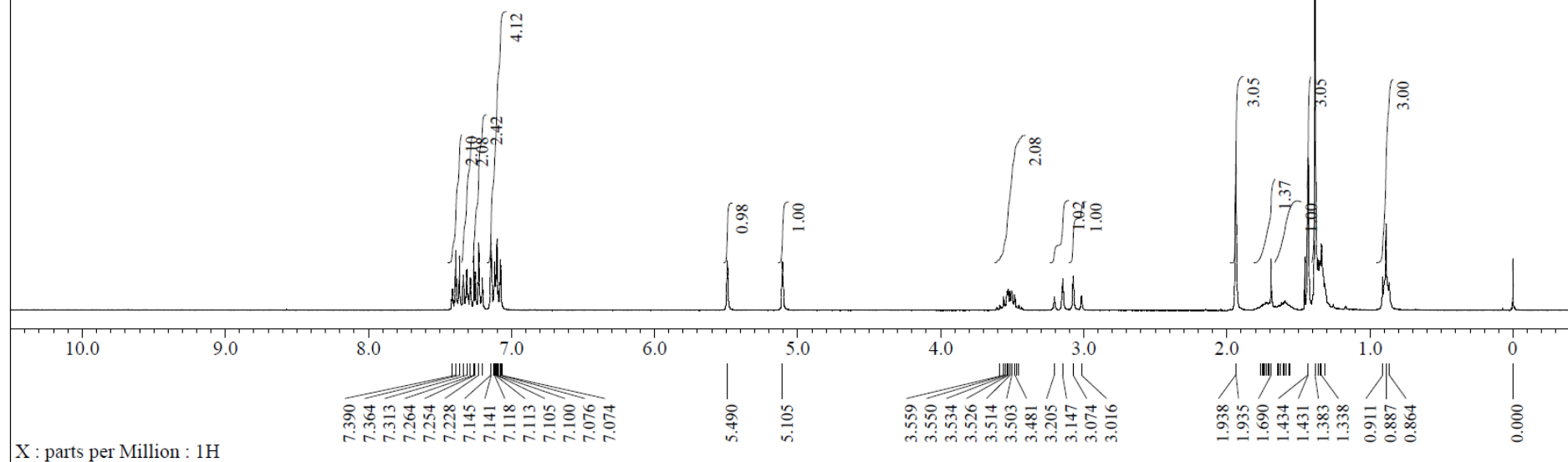
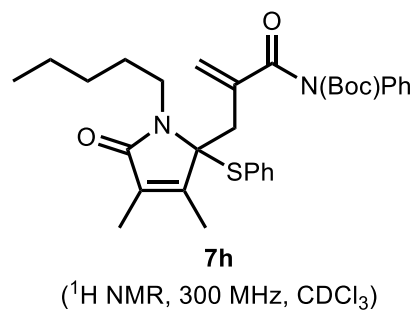


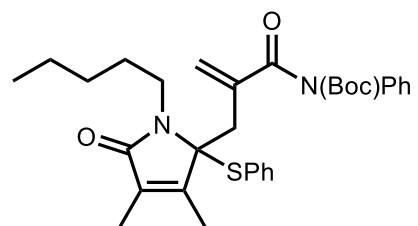
7g

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



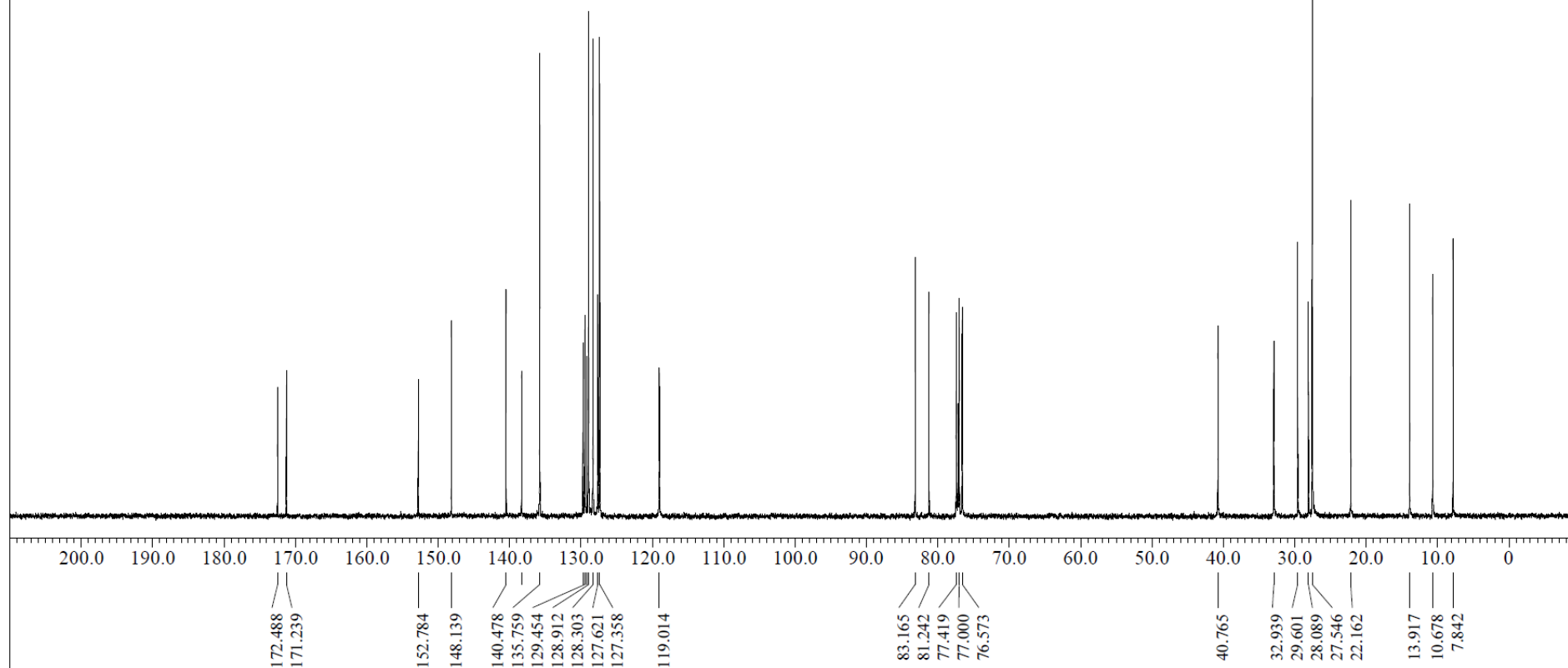
X : parts per Million : ^{13}C





7h

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



X : parts per Million : ^{13}C