

Supporting Information

Recyclable iodine-catalyzed oxidative C-H chalcogenation of 1,1-diarylethenes in water: Green synthesis of trisubstituted vinyl sulfides and selenides

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1. General reagent information

Starting materials such as ethene-1,1-diyldibenzene, α -methylstyrene, and all commercially available diorganyl disulphides, and solvents were purchased from Sigma-Aldrich, TCI, and other local chemical companies and used as such. Flash column chromatography was performed using silica gel (100-200 mesh). The de are commercially available.

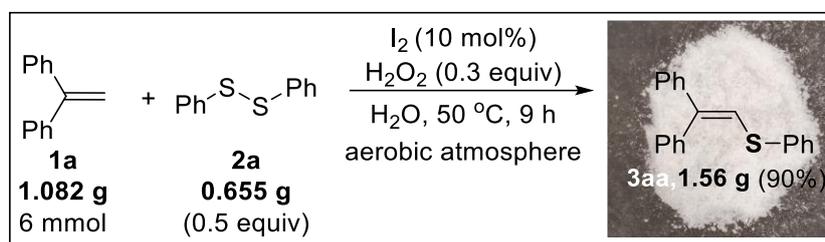
1.1. General analytical information

^1H , ^{13}C , ^{77}Se and ^{19}F NMR spectra were recorded on a Bruker 400 MHz instrument (400 MHz for ^1H NMR, 100 MHz for ^{13}C NMR, 76 MHz for ^{77}Se NMR and 376 MHz for ^{19}F NMR). Copies of ^1H , ^{13}C , ^{77}Se and ^{19}F NMR spectra can be found at the end of the Supporting Information. ^1H NMR data are reported in units, parts per million (ppm), and were measured relative to residual chloroform (7.26 ppm) in the deuterated solvent. $^{13}\text{C}\{^1\text{H}\}$ NMR spectra are reported in ppm relative to deuteriochloroform (77.00 ppm) and all were obtained with ^1H decoupling. Coupling constants were reported in Hz. Reactions were monitored by thin layer chromatography (TLC) and ^1H NMR of the crude reaction mixture using 1,3,5-trimethoxybenzene as the internal standard. Mass spectral data of unknown compounds were obtained on a high-resolution mass spectrometer, HRMS (6546 Q-TOF LC/MS, Agilent). Melting points of unknown compounds were recorded on a KRUSS Optronic M3000 apparatus. Single Crystal X-ray data were recorded on Rigaku Oxford Diffraction (XtalLab).

Representative experimental procedure for the synthesis of (2,2-diphenylvinyl)(phenyl)sulfane (**3aa**):

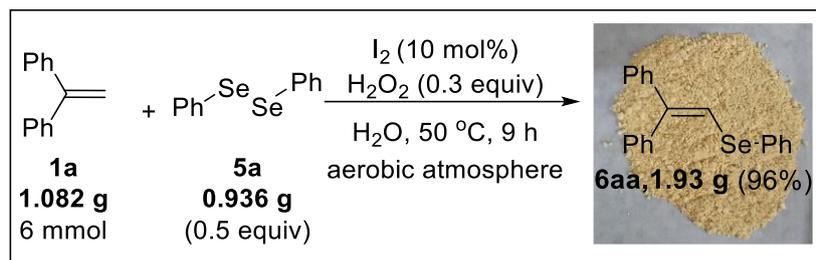
Ethene-1,1-diyldibenzene **1a** (0.088 mL, 0.5 mmol, 1 equiv), I₂ (0.013 g, 0.05 mmol) and 1,2-diphenyldisulfane **2a** (0.055 g, 0.25 mmol, 0.5 equiv) in a round-bottom flask (RBF) and H₂O (0.3 mL). was added to it. Then 30% aqueous H₂O₂ (v/v) (0.015 mL, 0.15 mmol, 0.3 equiv) was added and the reaction mixture was stirred at 50 °C under aerobic atmosphere. The progress of the reaction was monitored by TLC and after 7 h both the starting materials were found to be fully converted to product. To avoid the huge-solvent-consuming column chromatographic technique, we first quenched the iodine by Na₂S₂O₃ solution and then the product was separated from the reaction mixture through simple extraction using EtOAc (3 x 10 mL). The solvent was evaporated to dryness which furnished the pure product, (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (0.135 g, 0.47 mmol) in 94% yield. The reactions where starting materials were not fully consumed, column chromatographic purification were conducted to obtain the pure product.

4.1. Gram-scale synthesis of (2,2-diphenylvinyl)(phenyl)sulfane (**3aa**):



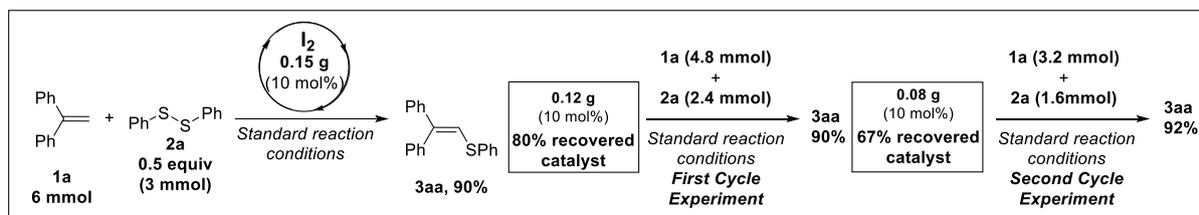
Ethene-1,1-diyldibenzene **1a** (1.06 mL, 6 mmol, 1 equiv), I₂ (0.152 g, 0.6 mmol) and 1,2-diphenyldisulfane **2a** (0.654 g, 3 mmol, 0.5 equiv) were taken in an RBF and H₂O (3.7 mL) was added to it. Then 30% aqueous H₂O₂ (v/v) (0.18 mL, 1.8 mmol, 0.3 equiv) was added and the reaction mixture was stirred at 50 °C under an aerobic atmosphere. The progress of the reaction was monitored by TLC and after 9 h both the starting materials were found to be fully converted to product. To avoid the huge-solvent-consuming column chromatographic technique, we first quenched the iodine by Na₂S₂O₃ solution and then the product was separated from the reaction mixture through simple extraction using EtOAc (3 x 25 mL). The solvent was evaporated to dryness which furnished the pure product, (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (1.56 g, 5.4 mmol) in yield 90% and around 72 mL EtOAc was recovered.

4.2. Gram-scale synthesis of (2,2-diphenylvinyl)(phenyl)selane (**6aa**):



Ethene-1,1-diyldibenzene **1a** (1.06 mL, 6 mmol, 1 equiv), I_2 (0.152 g, 0.6 mmol) and 1,2-diphenyldiselenane **2a** (0.94 g, 0.3 mmol, 0.5 equiv) were taken in a RBF and in H_2O (3.7 mL) was added to it. Then 30% aqueous H_2O_2 (v/v) (0.18 mL, 1.8 mmol, 0.3 equiv) was added and the reaction mixture was stirred at 50 °C under aerobic atmosphere. The progress of the reaction was monitored by TLC and after 9 h both the starting materials were found to be fully converted to product. To avoid the huge-solvent-consuming column chromatographic technique, we first quenched the iodine by $Na_2S_2O_3$ solution and then the product was separated from the reaction mixture through simple extraction using EtOAc (3 x 25 mL). The solvent was evaporated to dryness which furnished the pure product, (2,2-diphenylvinyl)(phenyl)selane **6aa** (1.93 g, 5.8 mmol) in 96% yield and around 72 mL EtOAc was recovered.

5. Recovery and recyclability of iodine:⁴



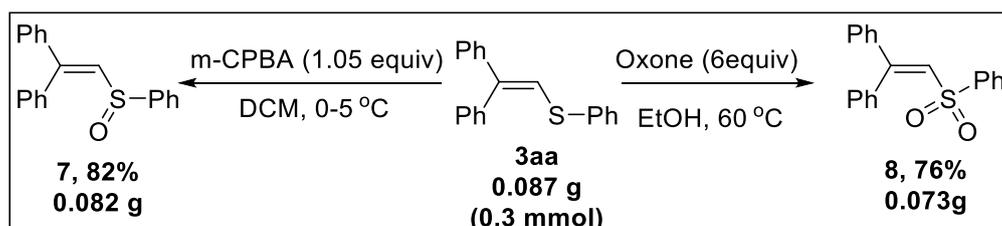
Step-1: Ethene-1,1-diyldibenzene **1a** (1.1 mL, 6 mmol, 1 equiv), I_2 (0.15 g, 0.6 mmol), 1,2-diphenyldisulfane **2a** (0.654 g, 3 mmol, 0.5 equiv) were taken in a RBF and H_2O (3.7 mL) was added to it. Then 30% aqueous H_2O_2 (v/v) (0.18 mL, 1.8 mmol, 0.3 equiv) was added and the reaction mixture was stirred at 50 °C under aerobic atmosphere for 9 h. The solution was then transferred to a separating funnel for extraction. The reaction mixture was extracted with ethyl acetate twice (2 X 20 mL) and the combined organic layer was washed with water (3 X 10 mL). The solvent was evaporated under reduced pressure and iodine (0.12 g, 80%) was recovered first from the crude reaction mixture by column chromatography using pentane as

eluent. The column chromatography process was further continued to obtain the pure product, (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (1.56 g, 5.4 mmol) in 90% yield.

Step-2: Recovered I₂ (0.12 g, 0.48 mmol), ethene-1,1-diylidibenzene **1a** (0.85 mL, 4.8 mmol, 1 equiv), and 1,2-diphenyldisulfane **2a** (0.523 g, 2.4 mmol, 0.5 equiv) were taken in a RBF and H₂O (2.93 mL) was added to it. Then 30% aqueous H₂O₂ (v/v) (0.14 mL, 1.44 mmol, 0.3 equiv) was added and the reaction mixture was stirred at 50 °C under an aerobic atmosphere for 9 h. The solution was then transferred to a separating funnel for extraction. The reaction mixture was extracted with ethyl acetate twice (2 X 20 mL) and the combined organic layer was washed with water (3 X 10 mL). The solvent was evaporated under reduced pressure and iodine (0.08 g, 67%) was recovered from the crude reaction mixture first by column chromatography using pentane as eluent. The column chromatography process was further continued to afford the pure product, (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (1.24g, 4.3 mmol) in 90% yield.

Step-3: To a solution of ethene-1,1-diylidibenzene **1a** (0.564 mL, 3.2 mmol, 1 equiv), I₂ (0.08 g, 0.32 mmol) and 1,2-diphenyldisulfane **2a** (0.35 g, 1.6 mmol, 0.5 equiv) in H₂O (1.95 mL) was added in a flame-dried RBF. Then 30% aqueous H₂O₂ (v/v) (0.096 mL, 0.96 mmol, 0.3 equiv) was added and the reaction mixture was stirred at 50 °C under an aerobic atmosphere for 9 h. The solution was then transferred to a separating funnel for extraction. The reaction mixture was extracted with ethyl acetate twice (2 X 20 mL) and the combined organic layer was washed with water (3 X 10 mL). The crude reaction mixture was purified by column chromatography to afford pure (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (0.85 g, 2.95 mmol) in 92% yield.

6. Experimental procedure for the synthesis of 2-(phenylsulfinyl)ethene-1,1-diylidibenzene **7** and (2-(phenylsulfonyl)ethene-1,1-diylidibenzene **8**.⁶



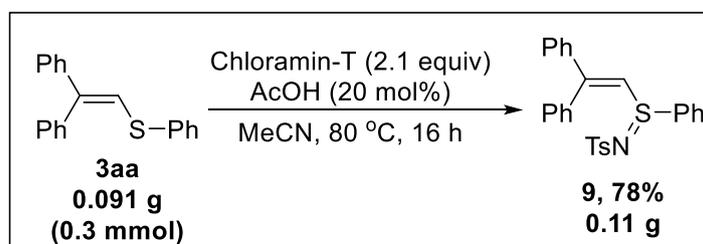
6.1. Experimental procedure for the synthesis of 2-(phenylsulfinyl)ethene-1,1-diyl)dibenzene **7**:

3-Chloroperoxybenzoic acid, *m*-CPBA (purity: 65-70%) (0.140 g, 0.32 mmol) was added to the solution of (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (0.087 g, 0.3 mmol, 1 equiv) dissolved in dichloromethane, DCM (2.5 mL) at 0 °C. Then, the reaction mixture was stirred vigorously for 2 h at room temperature. The progress of the reaction was monitored by TLC and after the completion of the reaction the solvent was evaporated under reduced pressure. The crude reaction mixture was extracted with DCM thrice (3 x 10 mL). The combined organic layer was washed with water (3 x 10 mL) and evaporated under reduced pressure. The crude product was purified by flash column chromatography through silica gel to afford the (2-(phenylsulfinyl)ethene-1,1-diyl)dibenzene **7** in (0.082 g, 0.25 mmol) in 82% yield.

6.2. Experimental procedure for the synthesis of (2-(phenylsulfonyl)ethene-1,1-diyl)dibenzene **8**:

Oxone (1.11 g, 1.8 mmol) was added to the solution of (2,2-diphenylvinyl)(phenyl)sulfane **3aa** (0.091 g, 0.3 mmol, 1 equiv) in ethanol (1.5 mL). The reaction mixture was stirred at 60 °C. The progress of the reaction was monitored by TLC until completion. After the completion of the reaction, the solvent was evaporated under reduced pressure. The crude reaction mixture was extracted with ethyl acetate thrice (3 x 10 mL). The combined organic layer was washed with water (3 x 10 mL) and evaporated under reduced pressure. The crude product was purified by flash column chromatography through silica gel to afford (2-(phenylsulfonyl)ethene-1,1-diyl)dibenzene **8** in (0.073 g, 0.23 mmol) 76% yield.

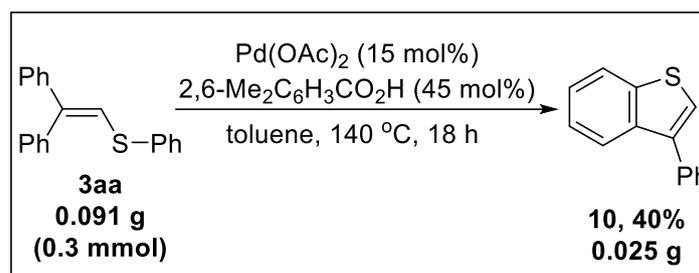
7. Experimental procedure for the synthesis of *N*-((2,2-diphenylvinyl)(phenyl)- λ -sulfanylidene)-4-methylbenzenesulfonamide (**9**):



To a solution of (2,2-Diphenylvinyl)(phenyl)sulfane **3aa** (0.091 g, 0.3 mmol) in MeCN (0.1 M) was added Chloramin-T trihydrate (0.18 g, 0.63 mmol) and glacial acetic acid (3 μ L, 0.06 mmol). The reaction mixture was then refluxed at 80 °C for 16 h. The mixture was allowed to

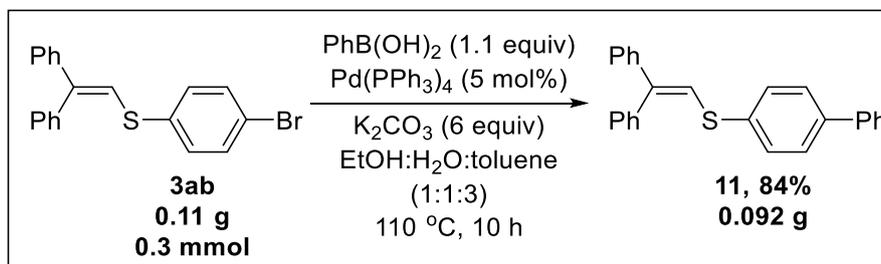
cool to room temperature. The solvent was removed under reduced pressure. The crude reaction mixture was extracted with ethyl acetate thrice (3 x 30 mL). The combined organic layer was washed with brine solution (30 mL) and concentrated under reduced pressure. The crude product was purified by flash column chromatography through silica gel to afford *N*-((2,2-diphenylvinyl)(phenyl)- λ -sulfanylidene)-4-methylbenzenesulfonamide **9** (0.11 g, 0.24 mmol) in 78% yield.

Experimental procedure for the synthesis of 3-phenylbenzo[*b*]thiophene ⁷



(2,2-diphenylvinyl)(phenyl)sulfane **3aa** (0.091 g, 0.3 mmol), diacetoxypalladium (0.010 g, 0.045 mmol, 1 equiv), 2,6-dimethylbenzoic acid (0.021 g, 0.135 mmol) in toluene (1.5 M) were taken in a 10 mL RBF. The reaction mixture was refluxed at 140 °C for 16 h. The mixture was cooled to room temperature and extracted with ethyl acetate (30x3 mL) three times. The combined organic layer was further washed with brine (30 mL) and subsequently dried over anhydrous Na₂SO₄. Finally, the solvent was evaporated under reduced pressure to get the crude product which was purified by flash column chromatography on silica gel to afford 3-phenylbenzo[*b*]thiophene **10** (0.025 g, 0.12 mmol) in 40% yield.

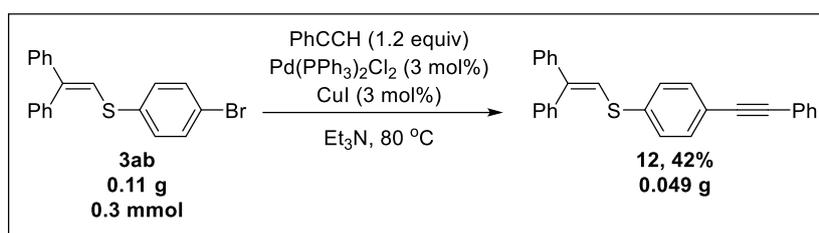
Experimental procedure for the synthesis of [1,1'-biphenyl]-4-yl(2,2-diphenylvinyl)sulfane (**11**):



4(4-Bromophenyl)(2,2-diphenylvinyl)sulfane **3ab** (0.11 g, 0.3 mmol, 1 equiv), phenyl boronic acid (0.040 g, 0.33 mmol), Pd(PPh₃)₄ (0.017 g, 0.015 mmol), K₂CO₃ (0.124 g, 0.9 mmol) and solvent (1 mL, EtOH : H₂O : PhMe = 1:1:3) were taken in a 25 mL RBF. The reaction mixture was refluxed at 110 °C and the progress of the reaction was monitored by thin layer

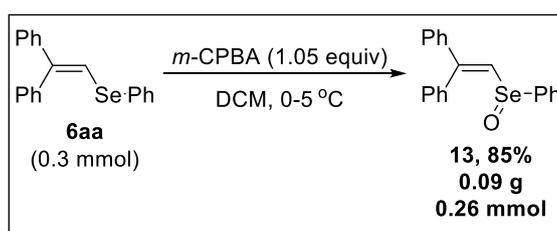
chromatography. The mixture was cooled to room temperature and extracted with ethyl acetate (30x3 mL) three times. The combined organic layer was further washed with brine (30 mL) and subsequently dried over anhydrous Na₂SO₄. Finally the solvent was evaporated under reduced pressure to get the crude product which was purified by flash column chromatography on silica gel to afford [1,1'-biphenyl]-4-yl(2,2-diphenylvinyl)sulfane **11** (0.092 g, 0.25 mmol) in 84% yield.

Experimental Procedure for The Synthesis of (2,2-diphenylvinyl)(4-(phenylethynyl)phenyl)sulfane (**12**):



To a solution of 4-(4-Bromophenyl)(2,2-diphenylvinyl)sulfane **3ab** (0.11 g, 0.3 mmol, 1 equiv) in Et₃N (3 mL) were added PdCl₂(PPh₃)₂ (0.006 g, 0.009 mmol) and CuI (0.0017 g, 0.009 mmol) under nitrogen atmosphere in a standard Schlenk-line process. The reaction mixture was stirred for 5 min under an inert atmosphere. Then, 2-phenyl acetylene (40 μL, 0.36 mmol, 1.2 equiv) was added to the reaction mixture. The resulting mixture was then heated under an inert atmosphere at 80 °C for 18 h. The mixture was allowed to cool to room temperature. The solvent was evaporated under reduced pressure. The crude reaction mixture was extracted with ethyl acetate thrice (3 x 30 mL). The combined organic layer was washed with brine solution (30 mL) and concentrated under reduced pressure. The crude product was purified by flash column chromatography through silica gel to afford the product (2,2-diphenylvinyl)(4-(phenylethynyl)phenyl)sulfane **12** (0.049 g, 0.13 mmol) in 42% yield.

Experimental procedure for the synthesis of (2-(phenylseleninyl)ethene-1,1-diyl)dibenzene (**13**)



3-Chloroperoxybenzoic acid, *m*-CPBA (purity: 65-70%) (0.140 g, 0.32 mmol) was added to a solution of (2,2-diphenylvinyl)(phenyl)selane **6aa** (0.168 g, 0.3 mmol) dissolved in dichloromethane (2.5 mL) at 0 °C. The reaction mixture was cooled at 0 °C. Then, the reaction mixture was stirred vigorously for 2 h. After the completion of the reaction the solvent was evaporated under reduced pressure. The crude reaction mixture was extracted with dichloromethane thrice (3 x 10 mL). The combined organic layer was washed with water (3 x 10 mL) and evaporated under reduced pressure. The crude product was purified by flash column chromatography through silica gel to afford the (2-(phenylseleninyl)ethene-1,1-diyl)dibenzene **13** in (0.09 g, 0.26 mmol) in 85% yield.

8. X-ray crystal structure of **3bi**.

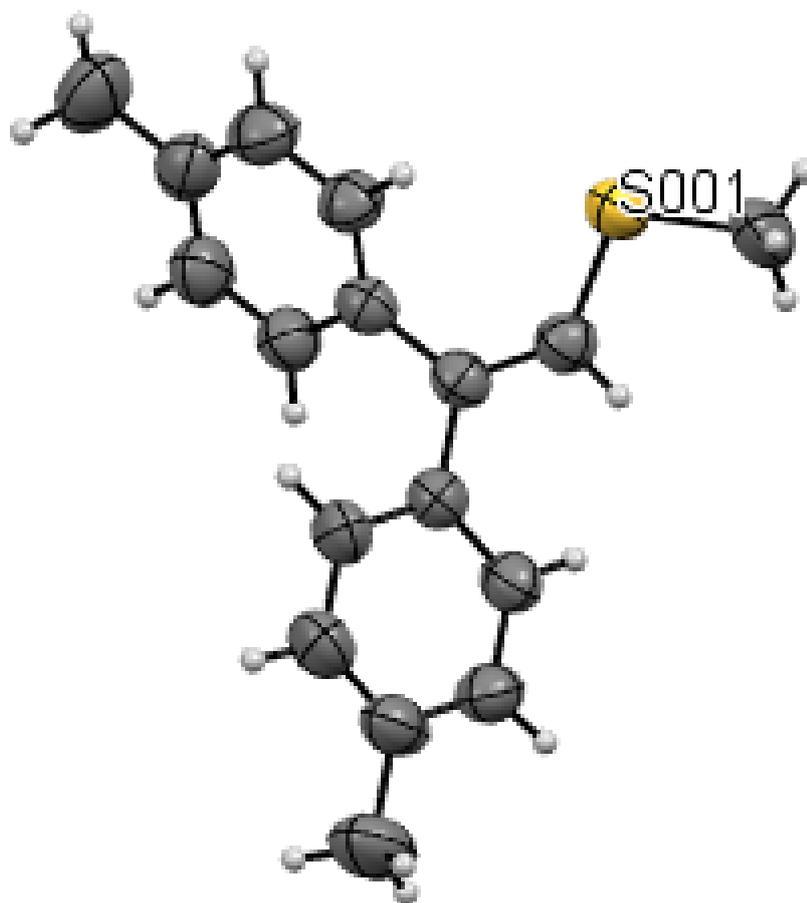


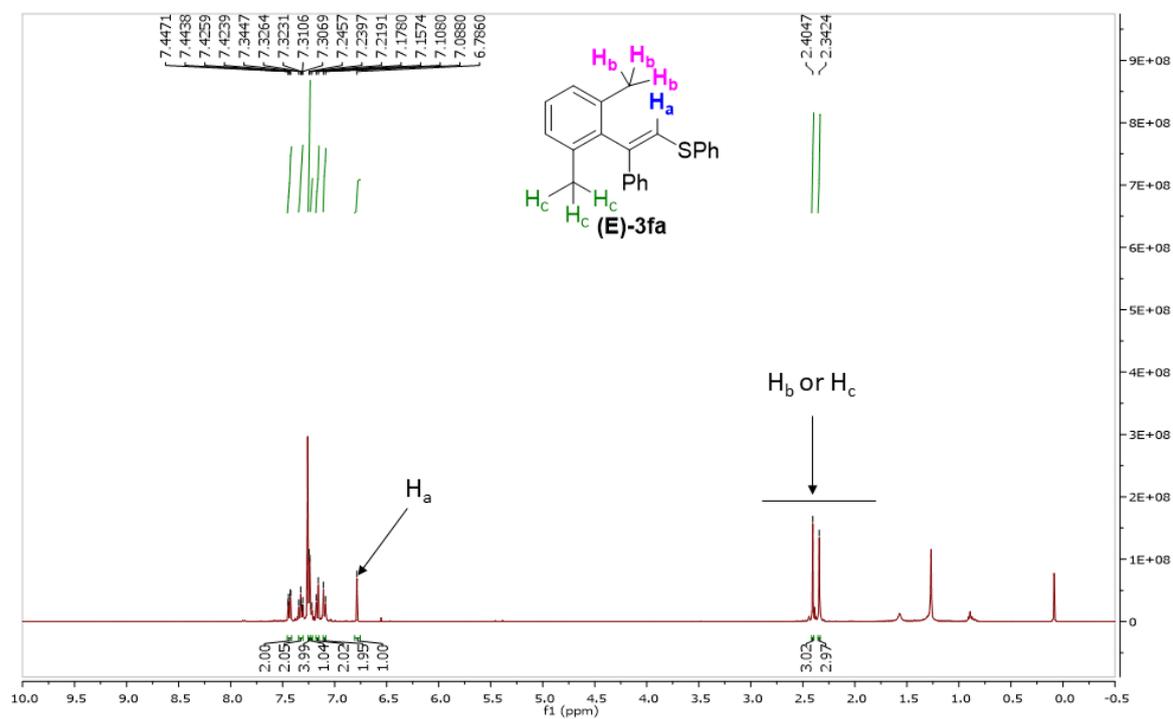
Figure S1. X-ray crystal structure of **3bi** (thermal ellipsoids shown at 50% probability) including hetero-atom numbering.

8.1. Table-S1. Selected crystal data of 3bi

<i>Parameters</i>	3bi
<i>Empirical formula</i>	$C_{17}H_{18}S$
<i>Formula weight</i>	254.37
<i>Temperature/K</i>	295
<i>Crystal system</i>	orthorhombic
<i>Space group</i>	$P\ 21\ 21\ 21$
$a/\text{\AA}$	9.4617(5)
$b/\text{\AA}$	11.8672(7)
$c/\text{\AA}$	13.2190(8)
α (°)	90
β (°)	90
γ (°)	90
<i>Volume/\AA^3</i>	1484.28(15)
<i>Z</i>	4
μ/mm^{-1}	1.754
D_x [g cm^{-3}]	1.138
<i>F(000)</i>	544
<i>2θ range for data collection (°)</i>	5.008-79.763
<i>Index ranges</i>	-11 ≤ h ≤ 12, - -10 ≤ k ≤ 14, - -16 ≤ l ≤ 15
<i>Reflections measured</i>	2648
<i>Unique reflections</i>	5041
<i>Parameters /restraints</i>	209/0
<i>Goodness-of-fit on F2</i>	1.084
R_1 [$I \geq 2\sigma(I)$]	0.0419
wR_2 (all data)	0.1226
<i>Largest diff. peak/hole/$e\ \text{\AA}^{-3}$</i>	0.204/-0.264
<i>CCDC</i>	2261525

9. Determination of stereochemistry of (2,2-diphenylvinyl)(phenyl)sulfane, 3fa.

(A)



(B)

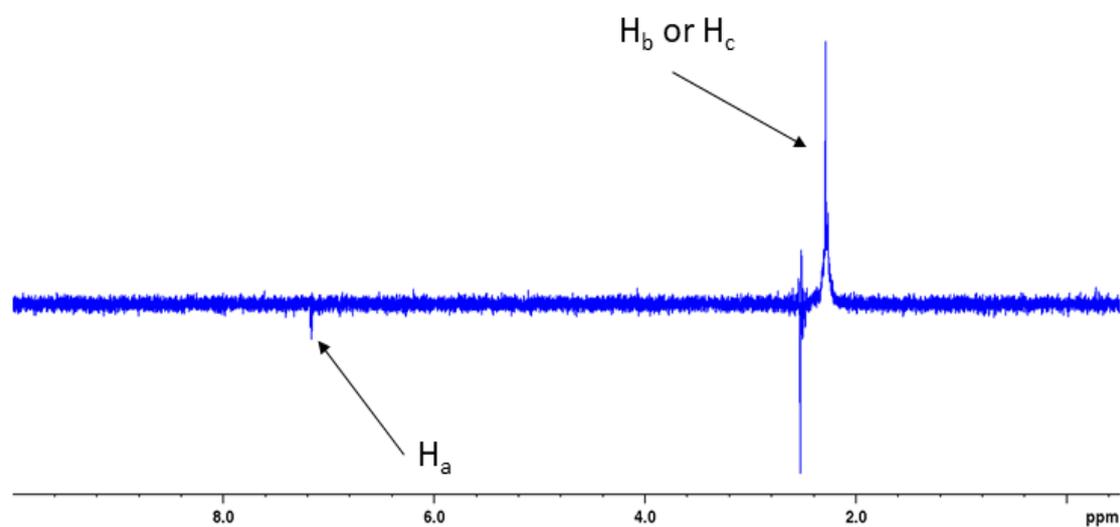


Figure S2: (A) ¹H-NMR spectrum of 3fa, (B) NOE difference spectrum, with irradiation at 6.79 ppm.

10.1. *In situ* detection of *tert*-butyl(2,2-diphenylvinyl)sulfane (3ao) by LC-MS

<Spectrum>

Line#: 1 R. Time: ---(Scan# ---)
MassPeaks: 36
Raw Mode: Averaged 0.391-0.852(135-293) BasePeak: 383.4(205721)
BG Mode: Averaged 1.167-1.983(401-681) Segment 1 - Event 1

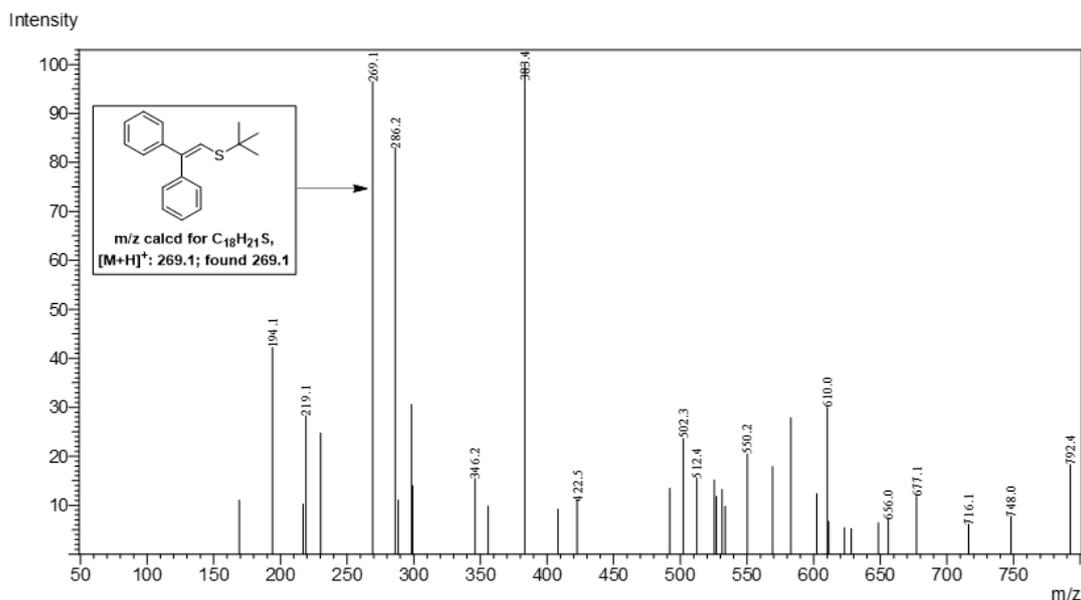


Figure S3. Mass spectrum of the reaction mixture of ethene-1,1-diyldibenzene, I₂ and 1,2-di-*tert*-butyldisulfane

10.2. *In situ* detection of 2,2-diphenylethene-1-thiol (C) by LC-MS

<Spectrum>

Line#: 1 R. Time: 1.359(Scan#: 467)
MassPeaks: 128
Raw Mode: Single 1.359(467) BasePeak: 73.2(5114225)
BG Mode: None Segment 1 - Event 1

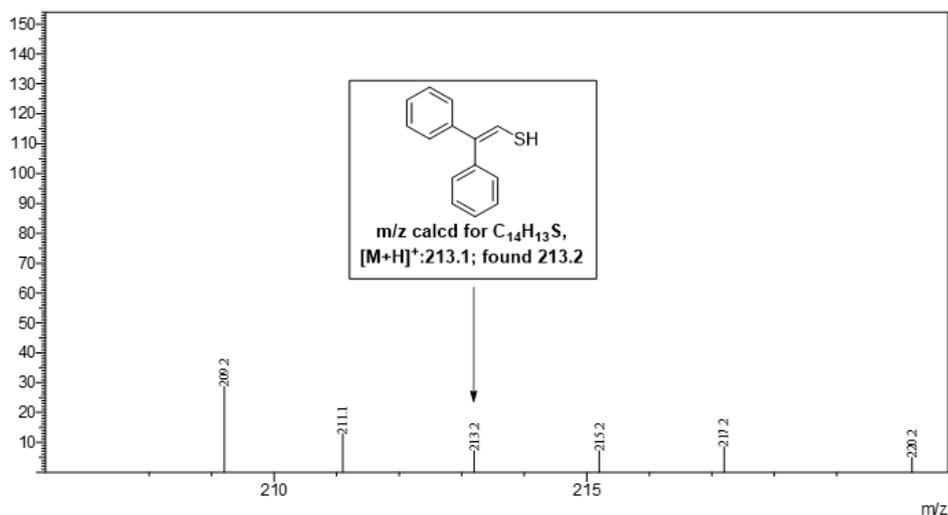
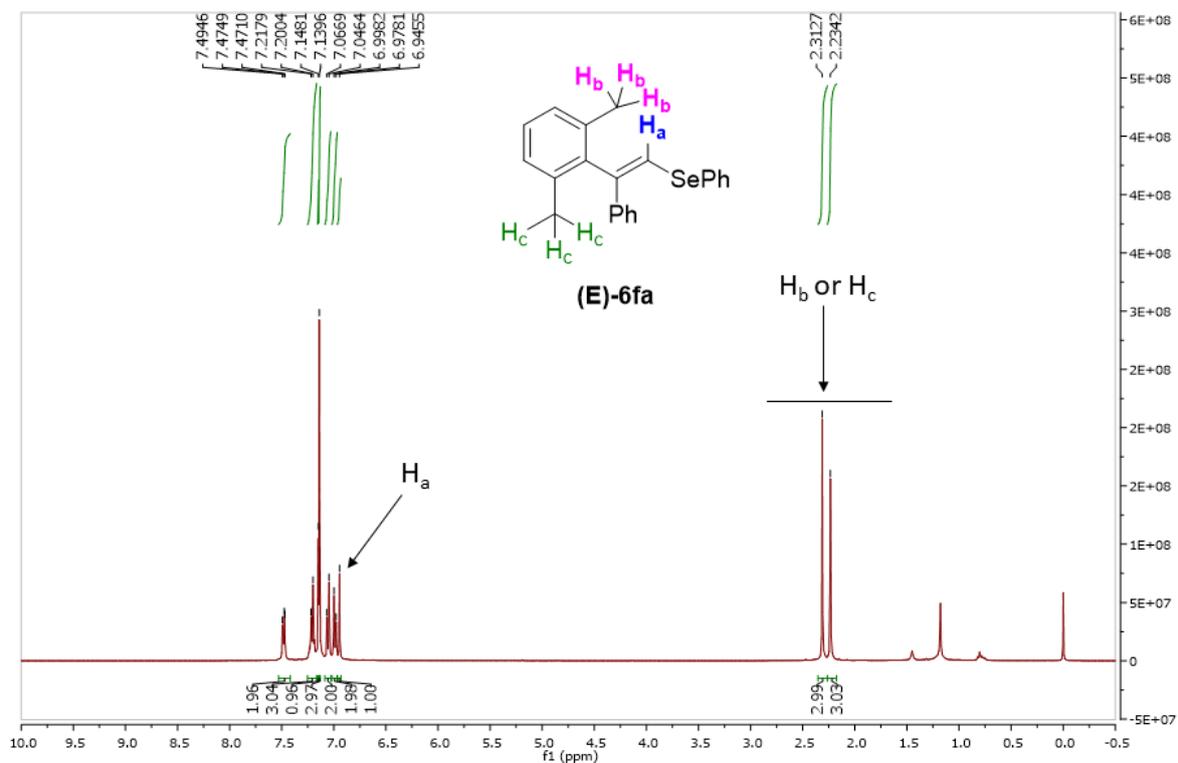


Figure S4. Mass spectrum of the reaction mixture of ethene-1,1-diyldibenzene, I₂ and 1,2-di-*tert*-butyldisulfane

11. Determination of stereochemistry of (2,2-diphenylvinyl)(phenyl)selane, **6fa**.

(A)



(B)

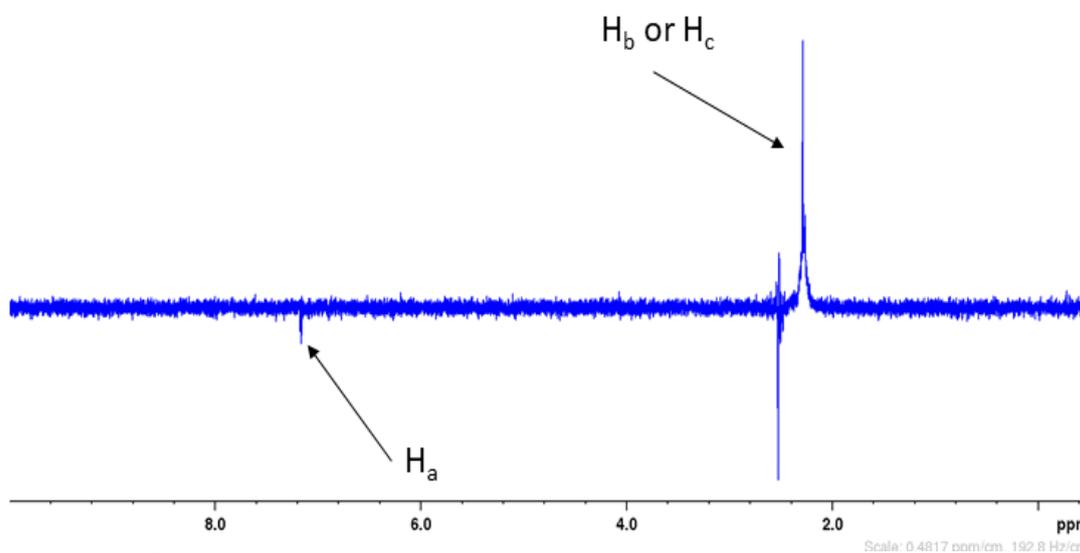
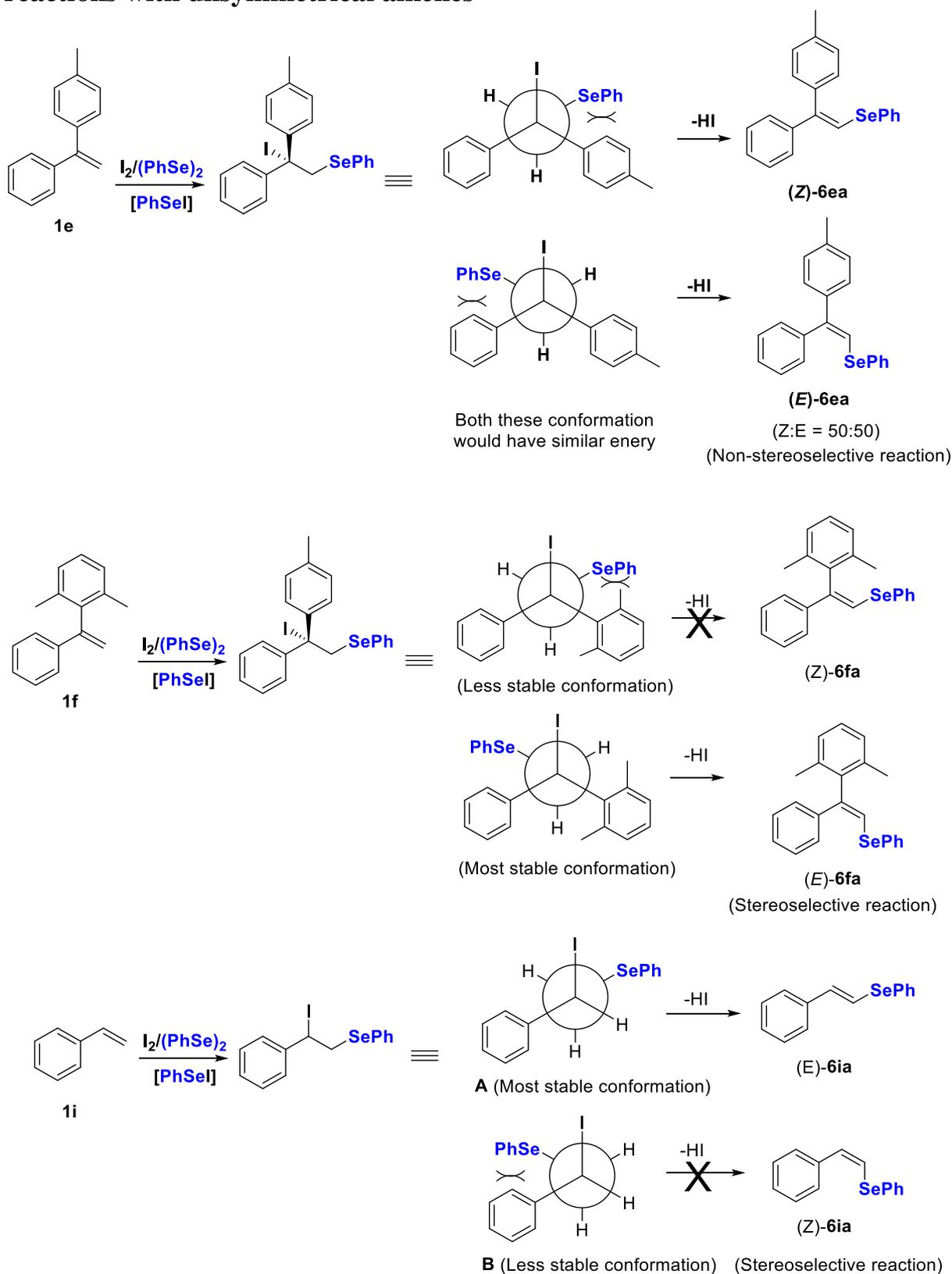


Figure S5: (A) ¹H-NMR spectrum of **6fa**, (B) NOE difference spectrum, with irradiation at 6.95 ppm.

12. Scheme S1. Explanation of stereoselectivity of C-H chalcogenation reactions with unsymmetrical alkenes



13. Table S2. Calculation of EcoScale score of the developed protocol for the synthesis of (2,2-diphenylvinyl)(phenyl)sulfane (3aa) ⁸

Eco Scale Calculation:

Eco Scale = 100 - Sum of individual penalties
Score on Eco Scale: > 75, Excellent; >50, acceptable; <50, Inadequate

Parameters	Penalty Points
1. Yield: $(100 - \% \text{ of yield})/2 = (100 - 90)/2 = 5$	5
2. Price of reaction components (To obtain 10 mmol of end product, 3aa)	
A. Calculation of Penalty Points :	
a. ethene-1,1-diyldibenzene = 11.37 mmol = 2.05 g = USD 7.08	
b. 1,2-diphenyldisulfane = 5.69 mmol = 1.24 g = USD 0.64	
c. Iodine (As catalyst) = 1.14 mmol = 0.289 g = USD 0.099	
d. Hydrogen Peroxide (30% of aqueous solution) = 0.33 mL = USD 0.087	
<hr/>	
Total cost of synthesis of 3aa = (6.49 + 0.64 + 0.099 + 0.087) = USD 7.32	0
Thus inexpensive, since <\$10 (total cost of synthesis of 10 mmol of 3aa) :	
3. Safety	
ethene-1,1-diyldibenzene	0
1,2-diphenyldisulfane (T)	5
Iodine (T)	5
4. Technical Setup	
Common Setup	0
5. Temperature/ Time	
50 °C, 7 h (Heating, > 1h)	3
6. Work up and purification :	
a. Adding solvent	0
b. No classical chromatography	0
<hr/>	
Total penalty points:	18

B. Ecoscale calculation:

EcoScale score: (100 - 18) = 82 (>75; it is an excellent synthesis)

14. Table S3. Calculation of EcoScale score of the developed protocol for the synthesis of (2,2-diphenylvinyl)(phenyl)sulfane (6aa) ⁸

Eco Scale Calculation:

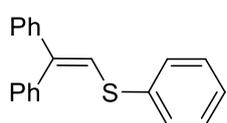
Eco Scale = 100 - Sum of individual penalties
Score on Eco Scale: > 75, Excellent; >50, acceptable; <50, Inadequate

Parameters	Penalty Points
1. Yield: $(100 - \% \text{ of yield})/2 = (100 - 96)/2 = 2$	2
2. Price of reaction components (To obtain 10 mmol of end product, 6aa)	
A. Calculation of Penalty Points :	
a. ethene-1,1-diyldibenzene = 10.42 mmol = 1.88 g = USD 6.49	
b. 1,2-diphenyldisulfane = 5.21 mmol = 1.63 g = USD 2.45	
c. Iodine (As catalyst) = 1.04 mmol = 0.264 g = USD 0.091	
d. Hydrogen Peroxide (30% of aqueous solution) = 0.31 mL = USD 0.082	
<hr/>	
Total cost of synthesis of 6aa = (6.49 + 2.45 + 0.091 + 0.082) = USD 9.113	0
Thus expensive, since < \$10 (total cost of synthesis of 10 mmol of 6aa):	
3. Safety	
ethene-1,1-diyldibenzene	0
1,2-diphenyldisulfane (T)	5
Iodine (T)	5
4. Technical Setup	
Common Setup	0
5. Temperature/ Time	
50 °C, 7 h (Heating, > 1h)	3
6. Work up and purification :	
a. Adding solvent	0
b. No classical chromatography	0
<hr/>	
Total penalty points:	15

B. Ecoscale calculation:

EcoScale score: (100 - 15) = 85 (>75; it is an excellent synthesis)

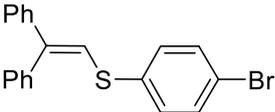
15. Analytical data of all synthesized products (3aa - 3an, 3ba – 3fa, 3bi, 3ci and 4, 6aa - 6aj, 6ba – 6ia, 7 - 14)

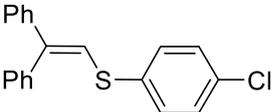


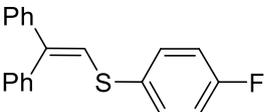
(2,2-Diphenylvinyl)(phenyl)sulfane (3aa):⁹ White solid (0.135 g, 94%);

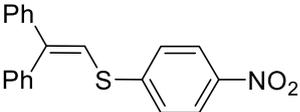
eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.54 – 7.48 (m, 4H), 7.45 (m, 3H), 7.42 – 7.36 (m, 3H), 7.35 (d, *J* = 2.9 Hz, 3H), 7.32 (m, 2H), 6.95

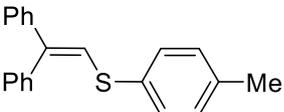
(s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 141.4, 141.0, 139.1, 136.5, 129.7, 129.5, 129.1, 128.3, 128.3, 127.7, 127.3, 127.2, 126.7, 124.1.

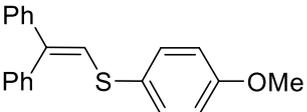
 **(4-Bromophenyl)(2,2-diphenylvinyl)sulfane (3ab):**⁹ White solid (0.138 g, 75%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.39 – 7.07 (m, 14H), 6.68 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 142.1, 141.2, 138.9, 135.7, 132.1, 130.8, 129.7, 128.4, 128.3, 127.9, 127.5, 127.2, 122.9, 120.6.

 **(4-Chlorophenyl)(2,2-diphenylvinyl)sulfane (3ac):**⁹ White solid (0.15 g, 94%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.49 – 7.44 (m, 2H), 7.43 – 7.37 (m, 5H), 7.36 – 7.29 (m, 7H), 6.83 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 141.9, 141.2, 138.9, 135.0, 132.7, 130.6, 129.7, 129.2, 128.4, 128.3, 127.9, 127.5, 127.2, 123.2.

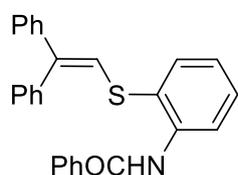
 **2,2-Diphenylvinyl(4-fluorophenyl)sulfane (3ad):**⁹ White solid (0.14 g, 92%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.30 (m, 4H), 7.23 (m, 3H), 7.17 – 7.10 (m, 5H), 6.94 – 6.85 (m, 2H), 6.65 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 162.1 (d, ¹J_{C-F} = 246 Hz), 141.3, 140.8, 139.0, 132.0 (d, ³J_{C-F} = 8 Hz), 131.5 (d, ⁴J_{C-F} = 4 Hz), 129.7, 128.4, 128.2, 127.8, 127.3, 127.1, 124.6, 116.2 (d, ²J_{C-F} = 22 Hz) (Overlapping peaks present). ¹⁹F NMR (376 MHz, CDCl₃) δ -114.45 (s).

 **(2,2-Diphenylvinyl)(4-nitrophenyl)sulfane (3ae):**⁹ Yellow solid (0.13 g, 79%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 9.0 Hz, 2H), 7.48 (d, *J* = 9.0 Hz, 2H), 7.45 – 7.42 (m, 2H), 7.36 – 7.30 (m, 8H), 6.87 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 146.61, 146.33, 145.70, 140.77, 138.56, 129.57, 128.47, 128.32, 128.19, 128.12, 127.42, 127.21, 124.13, 118.51.

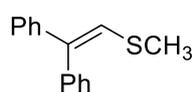
 **(2,2-Diphenylvinyl)(p-tolyl)sulfane (3af):**⁹ White solid (0.103 g, 68%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.27 (s, 1H), 7.27 – 7.18 (m, 6H), 7.13 – 7.06 (m, 5H), 6.99-6.97 (m, 2H), 6.70 (s, 1H), 2.18 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 141.5, 140.1, 139.2, 136.9, 132.8, 130.0, 129.8, 129.7, 128.3, 128.2, 127., 127.1, 125.2, 21.0 (overlapping peaks are present).

 **(2,2-Diphenylvinyl)(4-methoxyphenyl)sulfane (3ag):**⁹ Colourless liquid (0.13 g, 82%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.24 (m, 6H), 7.22 (s, 1H), 7.11-7.10 (m, 5H), 6.76-6.73 (m, 2H), 6.65 (s, 1H), 3.64 (s, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 159.2, 141.4, 139.1, 132.5,

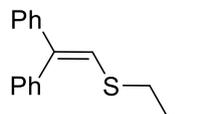
129.7, 128.3, 128.2, 127.6, 127.03, 126.99, 126.7, 126.5, 114.7, 55.3 (overlapping peaks are present).



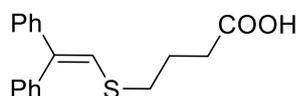
N-(2-((2,2-diphenylvinyl)thio)phenyl)benzamide (3ah): Yellow liquid (0.11 g, 52%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.92 (brs, 1H), 8.62 (dd, $J = 8.3, 1.1$ Hz, 1H), 7.90 – 7.78 (m, 2H), 7.67 (dd, $J = 7.7, 1.4$ Hz, 1H), 7.57 – 7.52 (m, 1H), 7.49 – 7.41 (m, 6H), 7.32 (dd, $J = 8.0, 1.5$ Hz, 2H), 7.23 (dd, $J = 5.1, 1.8$ Hz, 3H), 7.18 – 7.10 (m, 3H), 6.49 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 165.2, 142.4, 140.7, 139.3, 138.6, 134.8, 134.6, 132.0, 130.5, 129.6, 128.8, 128.5, 128.3, 128.2, 127.6, 127.2, 127.1, 124.4, 124.0, 123.2, 120.8. **HRMS (ESI)**, m/z calcd for $\text{C}_{27}\text{H}_{22}\text{NOS}$ [$\text{M} + \text{H}$] $^+$: 408.1417; found: 408.1390.



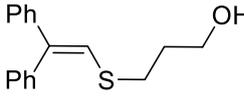
(2,2-diphenylvinyl)(methyl)sulfane (3ai):⁸ Yellow oil (0.109 g, 96%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.31 – 7.27 (m, 2H), 7.24 – 7.20 (m, 3H), 7.15 (s, 1H), 7.12 (m, 4H), 6.45 (s, 1H), 2.25 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.7, 139.4, 138.3, 129.63, 128.3, 128.2, 127.6, 127.5, 126.9, 126.8, 17.9.

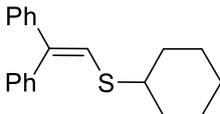


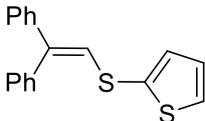
(2,2-Diphenylvinyl)(propyl)sulfane (3aj): Yellow liquid (0.12 g, 92%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.51-7.48 (m, 1H), 7.47 (d, $J = 2.0$ Hz, 1H), 7.45 (d, $J = 1.7$ Hz, 1H), 7.43 (m, 1H), 7.40 (m, 1H), 7.37-7.35 (m, 2H), 7.34 – 7.30 (m, 3H), 6.70 (s, 1H), 2.86 – 2.81 (m, 2H), 1.81 (d, $J = 7.3$ Hz, 2H), 1.11 (t, $J = 7.3$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.9, 139.5, 138.2, 129.62, 128.2, 128.1, 127.3, 126.9, 126.7, 126.3, 36.8, 23.6, 13.2. **HRMS (ESI)**, m/z calcd for $\text{C}_{18}\text{H}_{19}\text{S}$ [$\text{M} + \text{H}$] $^+$: 255.1202; found: 255.1169.

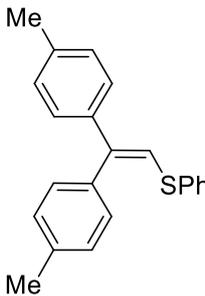


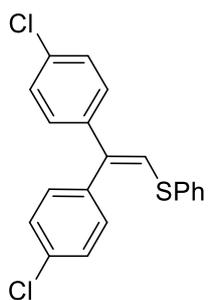
4-((2,2-Diphenylvinyl)thio)butanoic acid (3ak): Yellow viscous liquid (0.11 g, 76%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.31-7.29 (m, 3H), 7.25 – 7.20 (m, 3H), 7.14 – 7.12 (m, 4H), 6.47 (s, 1H), 2.73 (t, $J = 7.1$ Hz, 2H), 2.42 (t, $J = 7.2$ Hz, 2H), 1.97 – 1.89 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 179.0, 141.7, 139.4, 139.3, 129.6, 128.3, 128.2, 127.5, 126.95, 126.9, 125.2, 33.8, 32.3, 25.1. **HRMS (ESI)**, m/z calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2\text{S}$ [M]: 298.1028; found: 298.1053.


3-((2,2-Diphenylvinyl)thio)propan-1-ol (3al): Viscous liquid (0.084 g, 62%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.30 – 7.28 (m, 2H), 7.24 – 7.22 (m, 3H), 7.19 – 7.17 (m, 2H), 7.16 – 7.11 (m, 3H), 6.59 (s, 1H), 2.88 – 2.80 (m, 1H), 1.96 (d, $J = 2.9$ Hz, 1H), 1.74 – 1.68 (m, 2H), 1.35–1.25 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 178.6, 141.7, 139.5, 139.4, 129.7, 128.3, 128.2, 127.5, 127.0, 126.9, 125.2, 33.8, 32.2, 25.1. **Anal** calcd for $\text{C}_{17}\text{H}_{18}\text{OS}$: C, 75.52; H, 6.71; S, 11.86; found C, 75.40; H, 6.82; S, 11.79.

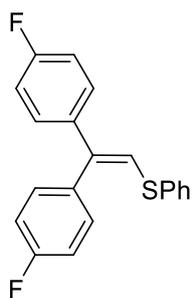

Cyclohexyl(2,2-diphenylvinyl)sulfane (3am): Colourless oil (0.097 g, 69%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.30 – 7.28 (m, 3H), 7.23 (d, $J = 7.4$ Hz, 2H), 7.119 – 7.17 (m, 2H), 7.16 – 7.11 (m, 3H), 6.59 (s, 1H), 2.88 – 2.80 (m, 1H), 2.02 – 1.94 (m, 2H), 1.74 – 1.68 (m, 2H), 1.58 – 1.53 (m, 1H), 1.40 – 1.16 (m, 5H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 142.2, 139.7, 138.2, 129.7, 128.2, 128.2, 127.3, 127.0, 126.7, 124.7, 46.8, 33.8, 26.1, 25.6. **HRMS (ESI)**, m/z calcd for $\text{C}_{20}\text{H}_{22}\text{S}$ [M]: 294.1442; found: 294.1437.


2-((2,2-Diphenylvinyl)thio)thiophene (3an):⁹ White solid (0.109 g, 74%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.37 – 7.32 (m, 2H), 7.31 – 7.25 (m, 4H), 7.20 – 7.14 (m, 3H), 7.13 – 6.91 (m, 4H), 6.63 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.0, 139.2, 138.8, 133.9, 132.7, 129.7, 129.3, 128.5, 128.3, 127.9, 127.6, 127.3, 127.1.

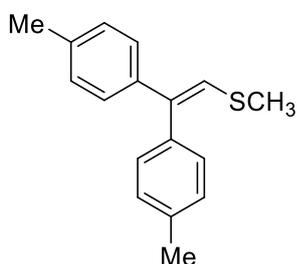

(2,2-Di-p-tolylvinyl)(phenyl)sulfane (3ba):⁹ Colourless liquid (0.124 g, 78%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.35 – 7.31 (m, 2H), 7.22 (t, $J = 7.6$ Hz, 2H), 7.15 – 7.14 (m, 5H), 7.07 (d, $J = 8.2$ Hz, 2H), 7.00 (d, $J = 7.9$ Hz, 2H), 6.68 (s, 1H), 2.30 (s, 3H), 2.24 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.4, 138.9, 137.5, 137.1, 136.8, 136.4, 129.6, 129.3, 129.03, 128.96, 128.2, 127.2, 126.6, 122.4, 21.3, 21.1.



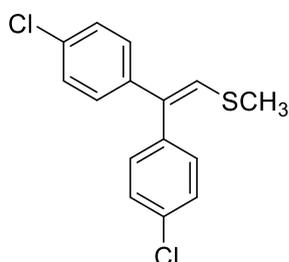
(2,2-Bis(4-chlorophenyl)vinyl)(phenyl)sulfane (3ca): White solid (0.16 g, 91%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 – 7.33 (m, 3H), 7.31 – 7.26 (m, 3H), 7.22 – 7.16 (m, 5H), 7.08 – 7.06 (m, 2H), 6.76 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 139.6, 138.2, 137.1, 135.7, 133.8, 133.3, 131.1, 129.8, 129.2, 128.8, 128.6, 128.34, 127.2, 125.7. **HRMS (ESI)**, m/z calcd for $\text{C}_{20}\text{H}_{15}\text{Cl}_2\text{S}$ [$\text{M} + \text{H}$] $^+$: 356.0193; found: 356.0160.



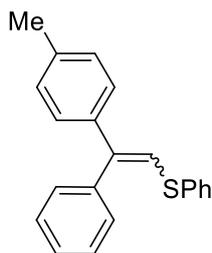
((2,2-Bis(4-fluorophenyl)vinyl)(phenyl)sulfane (3da): Yellow oil (0.126 g, 78%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.31 (d, $J = 7.4$ Hz, 2H), 7.23 – 7.18 (m, 4H), 7.13 (d, $J = 7.2$ Hz, 1H), 7.09 – 7.05 (m, 2H), 6.99 (t, $J = 8.7$ Hz, 2H), 6.85 (t, $J = 8.6$ Hz, 2H), 6.66 (s, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.20 (d, $^1J_{\text{C-F}} = 246$ Hz), 162.21 (d, $^1J_{\text{C-F}} = 246$ Hz), 138.9, 137.5, 136.0, 134.9, 131.45 (d, $^3J_{\text{C-F}} = 8$ Hz), 129.6, 129.2, 128.7 (d, $^3J_{\text{C-F}} = 8$ Hz), 127.0, 124.3, 115.2 (d, $^2J_{\text{C-F}} = 21$ Hz), 115.4 (d, $^2J_{\text{C-F}} = 21$ Hz). $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -113.28 (s), -114.51 (s). **HRMS (ESI)**, m/z calcd for $\text{C}_{20}\text{H}_{15}\text{F}_2\text{S}$ [$\text{M} + \text{H}$] $^+$: 325.0857; found: 325.0811.



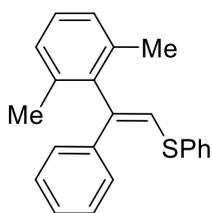
(2,2-Di-p-tolylvinyl)(methyl)sulfane (3bi): Yellow solid (0.12 g, 92%); eluent hexane; mp = 65 - 67 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.21 (s, 4H), 7.12 – 7.10 (m, 2H), 6.48 (s, 1H), 2.39 (s, 3H), 2.37 (s, 3H), 2.34 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 139.1, 138.4, 137.1, 136.6, 136.5, 129.5, 128.9, 128.9, 126.9, 126.1, 21.3, 21.0, 18.0. **HRMS (ESI)**, m/z calcd for $\text{C}_{17}\text{H}_{18}\text{S}$ [M]: 254.1129; found: 254.1199. The assignment is also supported by an X-ray crystallographic structure determination (CCDC 2261525).



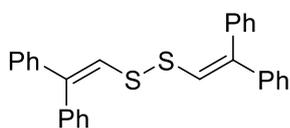
(2,2-Bis(4-chlorophenyl)vinyl)(methyl)sulfane (3ci):⁹ Orange solid (0.11 g, 72%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.27 (d, $J = 8.6$ Hz, 2H), 7.16 – 7.12 (m, 4H), 7.01 (d, $J = 8.7$ Hz, 2H), 6.46 (s, 1H), 2.29 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 139.8, 137.4, 135.8, 133.4, 132.8, 131.0, 128.9, 128.7, 128.4, 128.1, 17.9.



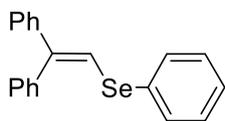
(Phenyl(2-phenyl-2-(p-tolyl)vinyl)sulfane (3ea): Colourless oil (0.114 g, 75%, Z:E = 55:45); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.39 – 7.35 (m, 5H), 7.33 – 7.25 (m, 9H), 7.22 – 7.19 (m, 10H), 7.07 (dd, $J = 21.0$, 5.0 Hz, 4H), 6.78 (s, 1H), 6.77 (s, 1H), 2.35 (s, 3H), 2.29 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.7, 141.4, 141.18, 139.3\3, 138.7, 137.6, 137.2, 136.7, 136.6, 136.2, 129.7, 129.6, 129.5, 129.4, 129.1, 129.0, 128.3, 128.3, 127.7, 127.3, 127.1, 126.7, 126.6, 123.6, 122.9 **Anal** calcd for $\text{C}_{21}\text{H}_{18}\text{S}$: C, 83.40; H, 6.00; S, 10.60; found C, 83.23; H, 5.82; S, 10.56.



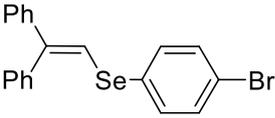
(E)-(2-(2,6-dimethylphenyl)-2-phenylvinyl)(phenyl)sulfane (3fa): Colourless liquid (0.068 g, 43%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.44 – 7.42 (m, 2H), 7.35 – 7.31 (m, 2H), 7.24 – 7.22 (m, 4H), 7.22 (s, 1H), 7.17 (d, $J = 8.2$ Hz, 2H), 7.10 (d, $J = 8.0$ Hz, 2H), 6.79 (s, 1H), 2.40 (s, 3H), 2.34 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.4, 138.9, 137.5, 137.1, 136.8, 136.4, 129.6, 129.4, 129.04, 128.96, 128.2, 127.2, 126.6, 122.4, 21.3, 21.1. **Anal** calcd for $\text{C}_{22}\text{H}_{20}\text{S}$: C, 83.50; H, 6.37; S, 10.13; found C, 83.64; H, 6.31; S, 10.05.

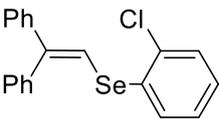


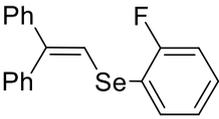
1,2-Bis(2,2-diphenylvinyl)disulfane (4): Yellow solid (0.13 g, 60%); eluent hexane; mp = 96 – 98 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49 – 7.45 (m, 8H), 7.42 – 7.40 (m, 5H), 7.37 (t, $J = 4.3$ Hz, 4H), 7.31 (d, $J = 1.4$ Hz, 3H), 6.91 (s, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 141.7, 139.9, 139.0, 129.7, 128.4, 128.3, 127.7, 127.2, 124.5 (Overlapping peaks are present). **Anal** calcd for $\text{C}_{28}\text{H}_{22}\text{S}_2$: C, 79.58; H, 5.25; S, 15.17; found C, 79.40; H, 5.43; S, 15.33.

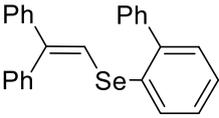


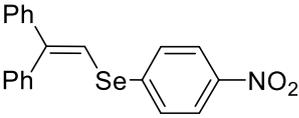
(2,2-Diphenylvinyl)(phenyl)selane (6aa):¹⁰ Yellow solid (0.16 g, 96%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.44 (dd, $J = 7.5$, 1.9 Hz, 2H), 7.32 – 7.19 (m, 5H), 7.19 – 7.02 (m, 8H), 7.00 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 143.0, 141.5, 140.3, 132.4, 131.2, 129.2, 129.2, 128.4, 128.2, 127.8, 127.3, 127.2, 127.1, 122.5.

 **(4-Bromophenyl)(2,2-diphenylvinyl)selane (6ab):**¹⁰ White solid (0.16 g, 77%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.32-7.31 (m, 5H), 7.30 – 7.26 (m, 2H), 7.22 – 7.19 (m, 2H), 7.16 – 7.13 (m, 5H), 6.93 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 143.8, 141.3, 140.1, 134.0, 132.4, 130.5, 129.3, 128.5, 128.3, 128.1, 127.4, 127.2, 121.7, 121.5.

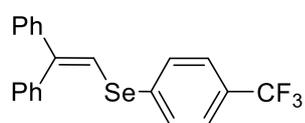
 **(2-Chlorophenyl)(2,2-diphenylvinyl)selane (6ac):**¹⁰ White solid (0.174 g, 94%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.54 (dd, *J* = 7.7, 1.7 Hz, 2H), 7.40 – 7.37 (m, 4H), 7.34 – 7.30 (m, 6H), 7.19 – 7.16 (m, 1H), 7.15 – 7.13 (m, 1H), 7.10 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 155.2, 141.5, 139.1, 135.4, 132.8, 130.3, 129.7, 128.9, 128.7, 128.7, 128.6, 128.3, 128.2, 128.0, 127.8, 127.6.

 **(2,2-Diphenylvinyl)(2-fluorophenyl)selane (6ad):** Yellow solid (0.164 g, 93%); eluent hexane; mp = 58 - 60 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.65 – 7.60 (m, 1H), 7.51 – 7.46 (m, 2H), 7.42 (m, 3H), 7.34 – 7.28 (m, 6H), 7.18 – 7.10 (m, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 161.3 (d, ¹*J*_{C-F} = 242Hz), 144.0, 141.4, 140.0, 133.9, 129.4, 129.3, 128.5, 128.3, 128.0, 127.4, 127.2, 124.9, 120.1, 118.2 (d, ²*J*_{C-F} = 23 Hz), 115.7 (d, ²*J*_{C-F} = 23 Hz). ⁷⁷Se NMR (76 MHz, CDCl₃) δ 302.08 (s). ¹⁹F NMR (376 MHz, CDCl₃) δ -103.64 (s). **Anal** calcd for C₂₀H₁₅FSe C, 67.99; H, 4.28 found C, 68.14; H, 4.16.

 **[1,1'-Biphenyl]-2-yl(2,2-diphenylvinyl)selane (6ae):** Yellow solid (0.15 g, 75%); eluent hexane; mp = 87 - 89 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.74 – 7.71 (m, 1H), 7.39 – 7.33 (m, 11H), 7.28 (s, 3H), 7.24 – 7.22 (m, 2H), 7.21 – 7.19 (m, 2H), 7.06 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 144.1, 143.9, 141.8, 141.6, 140.2, 134.2, 132.1, 132.0, 130.2, 129.4, 129.1, 128.3, 128.2, 128.1, 127.8, 127.5, 127.2, 127.0, 122.2 (Overlapping peaks are present). ⁷⁷Se NMR (76 MHz, CDCl₃) δ 350.06 (s). **Anal** calcd for C₂₆H₂₀Se: C, 75.91; H, 4.90; found C, 75.80; H, 5.02.

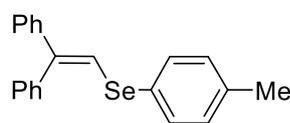
 **(2,2-Diphenylvinyl)(4-nitrophenyl)selane (6af):** Yellow solid (0.071 g, 37%); eluent hexane; mp = 55 - 57 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.34 – 7.28 (m, 4H), 7.27 – 7.21 (m, 3H), 7.20 – 7.13 (m, 7H), 6.66 (s, 1H). ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 142.1, 141.2, 138.9, 135.7, 132.1,

130.8, 129.7, 128.4, 128.3, 127.9, 127.5, 127.2, 122.9, 120.6.. **Anal** calcd for C₂₀H₁₅NO₂Se: C, 63.17; H, 3.98; N, 3.68; found C, 63.47; H, 3.56; N, 3.82.



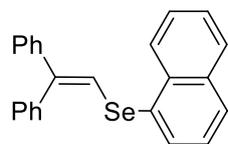
(2,2-Diphenylvinyl)(4-(trifluoromethyl)phenyl)selane (6ag):¹⁰

White solid (0.13 g, 62%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.35 – 7.27 (m, 3H), 7.21 – 7.12 (m, 7H), 6.97 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 145.1, 141.2, 140.06, 137.0, 131.7, 129.2, 128.6, 128.4, 128.2, 127.6, 127.2, 126.0, 125.4, 124.0 (d, ¹*J*_{C-F} = 270 Hz), 120.0.



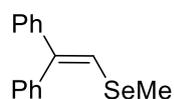
(2,2-Diphenylvinyl)(p-tolyl)selane (6ah):¹⁰ Yellow solid (0.13 g,

73%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.33 (m, 1H), 7.32 – 7.29 (m, 2H), 7.24 – 7.23 (m, 3H), 7.22 (s, 1H), 7.21 – 7.11 (m, 7H), 6.47 (s, 1H), 2.28 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 141.7, 139.5, 138.4, 133.4, 132.9, 129.7, 128.6, 128.3, 128.2, 127.6, 127.5, 127.0, 126.8, 18.0.



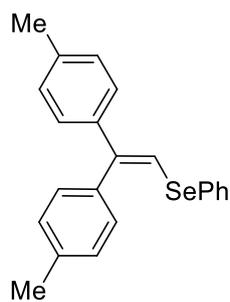
(2,2-Diphenylvinyl)(naphthalen-1-yl)selane (6ai):¹⁰ Yellow solid (0.16

g, 84%); eluent hexane; ¹H NMR (400 MHz, CDCl₃) δ 8.47 (d, *J* = 8.2 Hz, 1H), 8.03 (d, *J* = 7.1 Hz, 1H), 7.95 (t, *J* = 7.3 Hz, 2H), 7.69 – 7.60 (m, 3H), 7.59–7.57 (m, 3H), 7.55 – 7.49 (m, 2H), 7.33 (s, 5H), 7.21 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 142.9, 141.4, 140.3, 134.1, 134.0, 132.8, 130.4, 129.4, 128.9, 128.6, 128.5, 128.2, 127.9, 127.6, 127.1, 127.1, 126.8, 126.3, 125.8, 122.9.

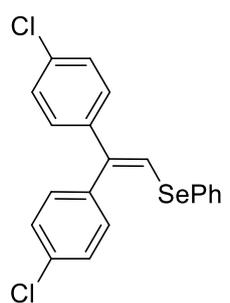


(2,2-Diphenylvinyl)(methyl)selane (6aj): White solid (0.126 g, 92%); eluent

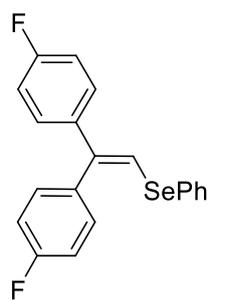
hexane; mp = 55 - 57 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.28 – 7.26 (m, 2H), 7.23 – 7.22 (m, 1H), 7.20 (d, *J* = 1.6 Hz, 1H), 7.18 (d, *J* = 1.4 Hz, 1H), 7.17 – 7.15 (m, 1H), 7.12 – 7.10 (m, 4H), 6.78 (s, 1H), 2.09 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 141.9, 141.8, 140.5, 129.2, 128.4, 128.2, 127.6, 126.9, 126.9, 123.0, 7.6. **Anal** calcd for C₁₅H₁₄Se: C, 65.94; H, 5.16 found C, 65.82; H, 5.32.



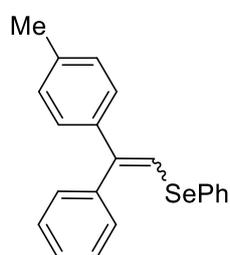
(2,2-Di-p-tolylvinyl)(phenyl)selane (6ba): Yellow solid (0.14 g, 78%); eluent hexane; mp = 85 - 87 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49 – 7.46 (m, 2H), 7.23 – 7.17 (m, 3H), 7.13 (s, 4H), 7.05 (d, $J = 8.2$ Hz, 2H), 6.98 (d, $J = 8.1$ Hz, 2H), 6.94 (s, 1H), 2.30 (s, 3H), 2.23 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.2, 139.0, 137.6, 137.5, 137.0, 132.3, 131.8, 129.2, 129.2, 129.1, 128.9, 127.2, 127.1, 120.8, 21.3, 21.1. $^{77}\text{Se NMR}$ (76 MHz, CDCl_3) δ 371.81 (s). **Anal** calcd for $\text{C}_{22}\text{H}_{20}\text{Se}$ C, 72.72; H, 5.55 found C, 72.96 ; H, 5.38.



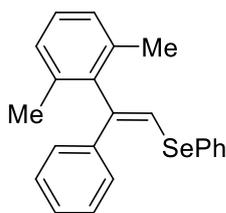
(2,2-Bis(4-chlorophenyl)vinyl)(phenyl)selane (6ca):¹⁰ Yellow oil (0.15 g, 74%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 – 7.47 (m, 2H), 7.33 – 7.30 (m, 2H), 7.25 – 7.22 (m, 3H), 7.18 (s, 1H), 7.17 – 7.15 (m, 2H), 7.14 (s, 1H), 7.05 (d, $J = 2.1$ Hz, 1H), 7.03 (d, $J = 2.2$ Hz, 2H). ^{13}C { ^1H } **NMR** (100 MHz, CDCl_3) δ 140.4, 139.7, 138.2, 133.9, 133.3, 132.7, 131.0, 130.7, 129.4, 128.9, 128.5, 128.3, 127.7, 124.2.



(2,2-Bis(4-fluorophenyl)vinyl)(phenyl)selane (6da): Yellow oil (0.169 g, 91%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.65 – 7.56 (m, 2H), 7.36 – 7.32 (m, 5H), 7.25 – 7.18 (m, 2H), 7.15 (t, $J = 8.7$ Hz, 2H), 7.08 (s, 1H), 6.99 (t, $J = 8.7$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 162.3 (d, $^1J_{\text{C-F}} = 246$ Hz), 162.2 (d, $^1J_{\text{C-F}} = 240$ Hz), 140.9, 137.7, 136.0, 132.6, 131.2, 131.0 (d, $^3J_{\text{C-F}} = 8$ Hz), 129.4, 128.5 (d, $^3J_{\text{C-F}} = 8$ Hz), 127.6, 122.7, 115.5 (d, $^2J_{\text{C-F}} = 39$ Hz), 115.3 (d, $^2J_{\text{C-F}} = 39$ Hz). $^{77}\text{SeNMR}$ (76 MHz, CDCl_3) δ 378.00 (s). $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -113.14 (s), -114.63 (s). **HRMS (ESI)**, m/z calcd for $\text{C}_{20}\text{H}_{14}\text{F}_2\text{Se}$ [M]: 372.0229; found: 372.0237.

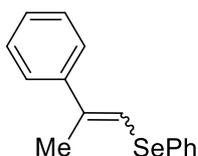


Phenyl(2-phenyl-2-(p-tolyl)vinyl)selane (6ea): Colourless oil (0.11 g, 65%, Z:E = 50:50); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.51-7.46 (m, 4H), 7.38 – 7.23 (m, 6H), 7.22 – 7.17 (m, 5H), 7.16 (dd, $J = 5.9$, 2.1 Hz, 4H), 7.15 (d, $J = 4.3$ Hz, 4H), 7.11 (d, $J = 3.8$ Hz, 2H), 7.06 – 6.99 (m, 3H), 6.98 (s, 1H), 6.97 (s, 1H), 2.31 (s, 3H), 2.22 (s, 3H). ^{13}C { ^1H } **NMR** (100 MHz, CDCl_3) δ 143.2, 143.1, 141.7, 140.5, 138.8, 137.7, 137.4, 137.1, 132.4, 132.4, 131.7, 129.3, 129.2, 129.2, 129.0, 128.7, 128.4, 128.2, 127.8, 127.3, 127.3, 127.2, 127.1, 127.0, 122.0, 121.3, 21.3, 21.1.



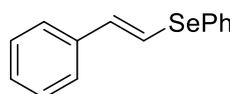
(E)-(2-(2,6-dimethylphenyl)-2-phenylvinyl)(phenyl)selane (6fa):

Pale yellow oil (0.1 g, 55%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.53 – 7.42 (m, 2H), 7.21 (d, $J = 7.0$ Hz, 3H), 7.15 (s, 1H), 7.14 (s, 3H), 7.06 (d, $J = 8.2$ Hz, 2H), 6.99 (d, $J = 8.1$ Hz, 2H), 6.95 (s, 1H), 2.31 (s, 3H), 2.23 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 143.2, 139.0, 137.6, 137.5, 137.0, 132.3, 131.8, 129.2, 129.2, 129.1, 128.9, 127.2, 127.1, 120.8, 21.3, 21.1. **Anal** calcd for $\text{C}_{22}\text{H}_{20}\text{Se}$: C, 72.72; H, 5.55; found C, 72.86; H, 5.38.



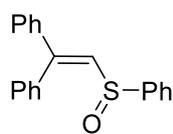
Phenyl(2-phenylprop-1-en-1-yl)selane (6ga): Yellow oil (0.042 g, 31%,

Z:E = 50:50); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.62 – 7.57 (m, 4H), 7.44 (dd, $J = 8.0, 6.6$ Hz, 3H), 7.40 – 7.37 (m, 2H), 7.35 – 7.31 (m, 5H), 7.28 – 7.25 (m, 6H), 7.15 (s, 1H), 7.10 (s, 1H), 2.43 (s, 3H), 2.35 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 143.1, 141.7, 140.5, 138.8, 137.7, 137.4, 137.1, 132.4, 132.4, 131.7, 129.2, 129.2, 129.0, 128.7, 128.4, 128.2, 127.8, 127.3, 127.3, 127.19, 127.2, 127.0, 21.3, 21.1. **Anal** calcd for $\text{C}_{15}\text{H}_{14}\text{Se}$: C, 65.94; H, 5.16; found C, 65.99; H, 5.04.



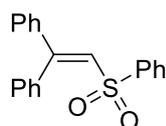
(E)-phenyl(styryl)selane (6ia):¹⁰ Yellow solid (0.032 g, 25%); eluent

hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.67 – 7.49 (m, 2H), 7.43 (dd, $J = 9.3, 5.6$ Hz, 2H), 7.40 – 7.32 (m, 3H), 7.29 – 7.25 (m, 3H), 7.24 (d, $J = 7.6$ Hz, 1H), 7.16 (d, $J = 7.8$ Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 143.1, 133.5, 132.3, 129.3, 129.0, 128.5, 128.2, 128.0, 127.7, 127.2.



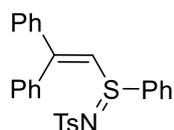
(2-(Phenylsulfinyl)ethene-1,1-diyl)dibenzene (7):¹² White solid (0.075 g,

82%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.64 – 7.57 (m, 2H), 7.46 – 7.38 (m, 6H), 7.32 – 7.25 (m, 3H), 7.21 (dt, $J = 10.8, 7.7$ Hz, 4H), 6.75 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 152.8, 144.8, 138.9, 136.9, 133.3, 130.8, 130.1, 129.8, 129.4, 129.2, 128.5, 128.4, 128.4, 124.6.

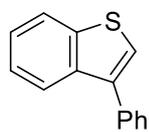


(2-(Phenylsulfinyl)ethene-1,1-diyl)dibenzene (8):¹³ White solid (0.073 g,

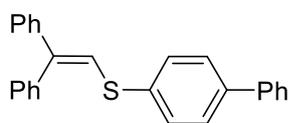
76%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.59 – 7.57 (m, 2H), 7.48 (t, $J = 4.0$ Hz, 1H), 7.40 – 7.34 (m, 4H), 7.33 – 7.29 (m, 4H), 7.23 – 7.20 (m, 2H), 7.10 – 7.06 (m, 2H), 7.03 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 155.2, 141.4, 139.0, 135.4, 132.8, 130.3, 129.7, 128.9, 128.7, 128.6, 128.6, 128.2, 127.8, 127.6.



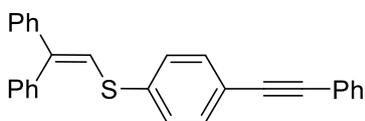
***N*-((2,2-diphenylvinyl)(phenyl)- λ_4 -sulfanylidene)-4-methylbenzenesulfonamide (9):** White solid (0.11 g, 78%); eluent 20% ethyl acetate : hexane; mp = 96 - 98 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.71 – 7.65 (m, 4H), 7.51 – 7.46 (m, 4H), 7.40 – 7.36 (m, 3H), 7.31 (t, $J = 7.5$ Hz, 2H), 7.19 – 7.13 (m, 4H), 7.08 – 7.06 (m, 2H), 6.67 (s, 1H), 2.35 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 155.5, 141.6, 141.4, 137.9, 136.9, 136.1, 131.8, 130.6, 129.8, 129.7, 129.7, 129.1, 128.7, 128.6, 128.5, 126.43, 126.4, 123.0, 21.4. **Anal** calcd for $\text{C}_{27}\text{H}_{23}\text{NO}_2\text{S}_2$: C, 70.87; H, 5.07; N, 3.06; S, 14.01; found C, 70.99; H, 5.03; N, 3.02; S, 14.04.



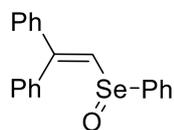
3-Phenylbenzo[b]thiophene (10):¹⁴ White solid (0.018 g, 40%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.78 (m, 2H), 7.45 (dd, $J = 8.2, 1.3$ Hz, 2H), 7.35 (dd, $J = 8.1, 6.8$ Hz, 2H), 7.25 – 7.18 (m, 2H), 7.16 – 7.08 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 140.7, 138.1, 137.9, 136.0, 131.0, 129.2, 128.7, 127.5, 124.4, 124.3, 123.4, 122.9.



[1,1'-Biphenyl]-4-yl(2,2-diphenylvinyl)sulfane (11):⁹ White solid (0.092 g, 84%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60 – 7.56 (m, 4H), 7.53 – 7.50 (m, 2H), 7.47 – 7.42 (m, 4H), 7.40 – 7.36 (m, 4H), 7.32 – 7.27 (m, 5H), 6.91 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.4, 141.3, 140.3, 139.8, 139.2, 135.5, 129.8, 129.8, 128.9, 128.4, 128.3, 127.8, 127.8, 127.5, 127.3, 127.2, 126.9, 123.9.

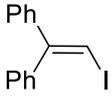


(2,2-Diphenylvinyl)(4-(phenylethynyl)phenyl)sulfane (12): Yellow viscous liquid (0.049 g, 42%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.70 (m, 1H), 7.40 – 7.29 (m, 12H), 7.25 (d, $J = 2.2$ Hz, 1H), 7.24 (d, $J = 2.2$ Hz, 2H), 7.22 – 7.21 (m, 1H), 7.20 – 7.17 (m, 2H), 7.04 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 143.4, 136.3, 132.3, 131.4, 131.2, 131.1, 131.0, 129.0, 128.1, 127.9, 127.8, 127.5, 127.24, 125.9, 125.7, 125.4, 124.9, 124.2, 123.9, 122.9, 92.9, 89.1. **Anal** calcd for $\text{C}_{28}\text{H}_{20}\text{S}$: C, 86.56; H, 5.19; S, 8.25; found C, 86.65; H, 5.14; S, 8.21.



(2-(Phenylseleninyl)ethene-1,1-diyl)dibenzene (13): Colourless oil (0.081 g, 77%); eluent hexane; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.66 (dd, $J = 7.7, 1.8$ Hz, 2H), 7.47 – 7.38 (m, 6H), 7.25-7.23 (m, 4H), 7.22 – 7.18 (m, 3H), 6.88 (s, 1H).

^{13}C NMR (100 MHz, CDCl_3) δ 153.9, 142.0, 138.3, 137.7, 133.6, 131.1, 129.8, 129.7, 129.5, 128.7, 128.7, 128.5, 128.2, 126.4. ^{77}Se NMR (76 MHz, CDCl_3) δ 848.85 (s). **Anal** calcd for $\text{C}_{20}\text{H}_{16}\text{OSe}$: C, 68.38; H, 4.59; found C, 68.49; H, 4.41.

 **(2-Iodoethene-1,1-diyl)dibenzene (14):**¹⁵ Pale yellow oil (0.138 g, 90%); eluent hexane; ^1H NMR (400 MHz, CDCl_3) δ 7.31 (d, $J = 7.2$ Hz, 3H), 7.20 – 7.14 (m, 5H), 7.12 (dd, $J = 3.7, 1.6$ Hz, 2H), 6.84 (s, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 152.7, 141.8, 141.1, 129.4, 128.3, 128.3, 128.1, 128.0, 127.5, 79.0.

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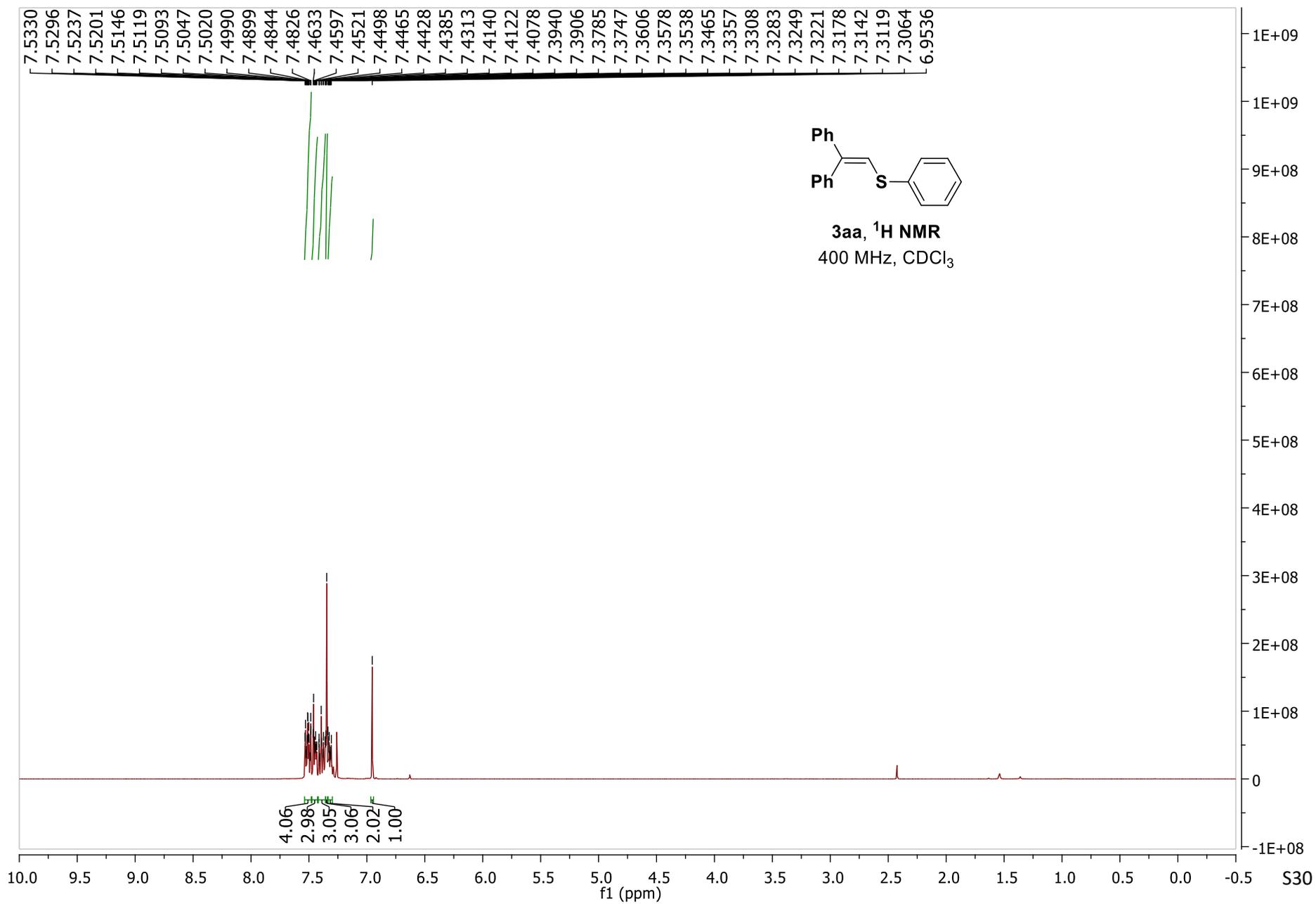
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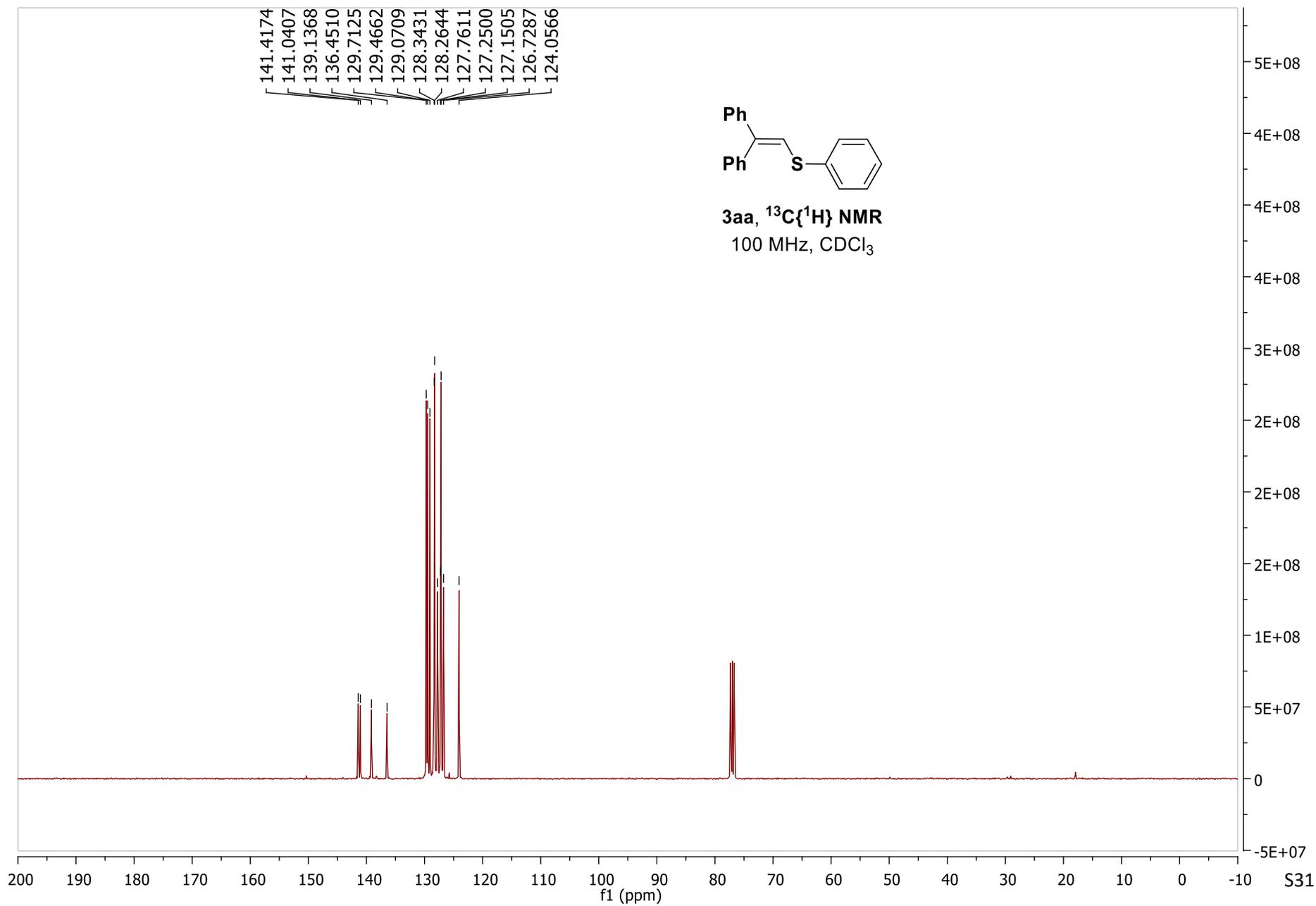
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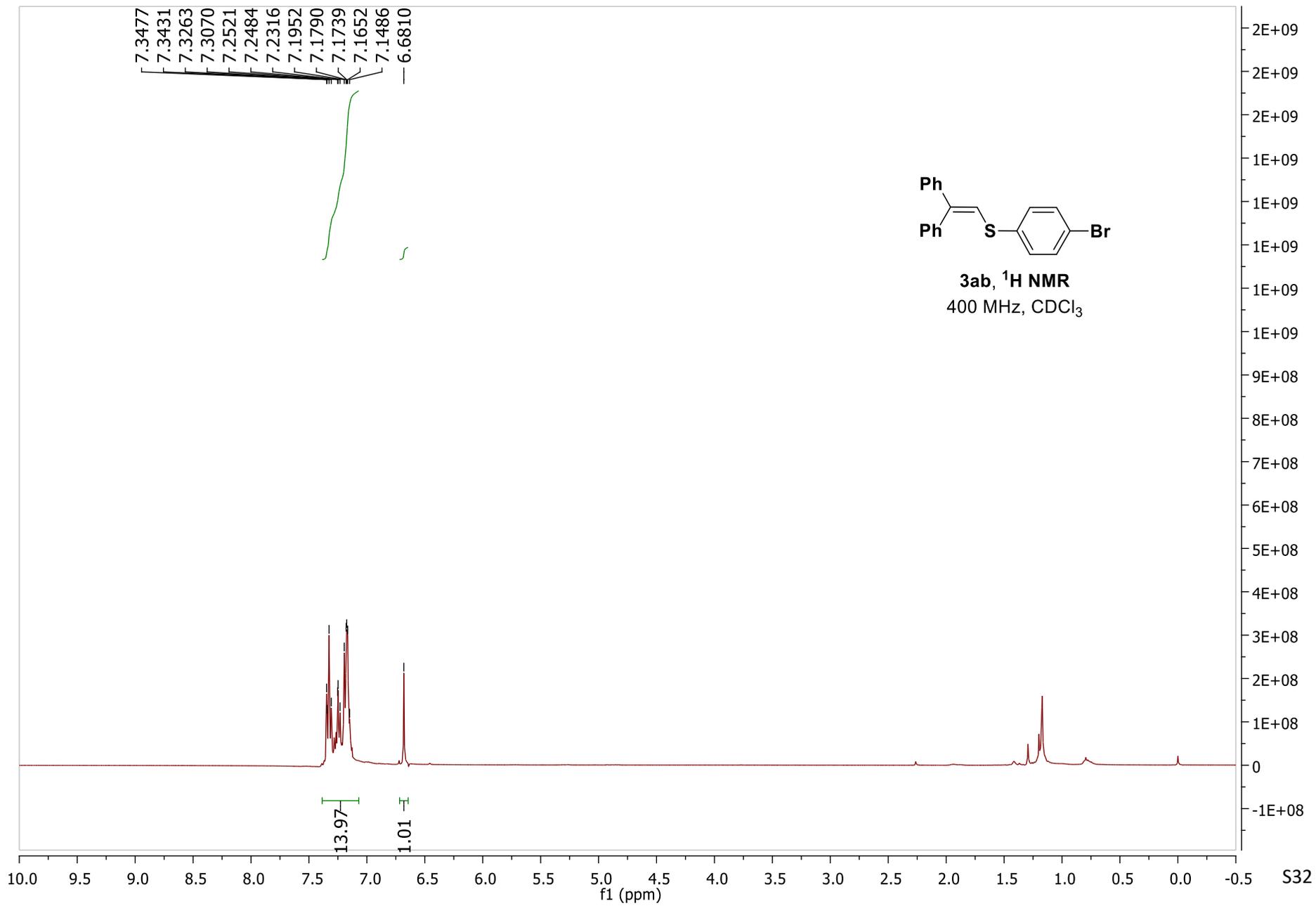
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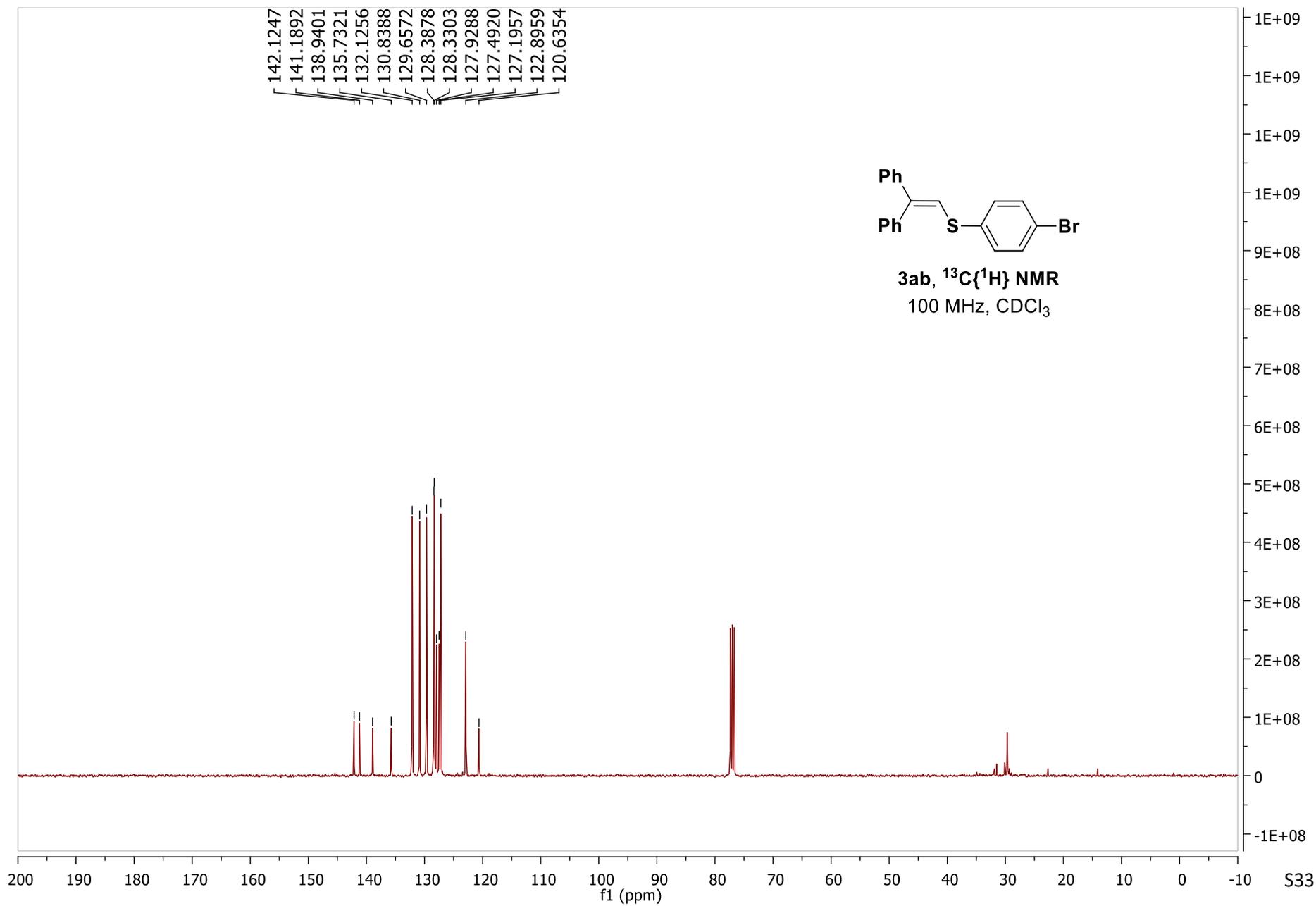
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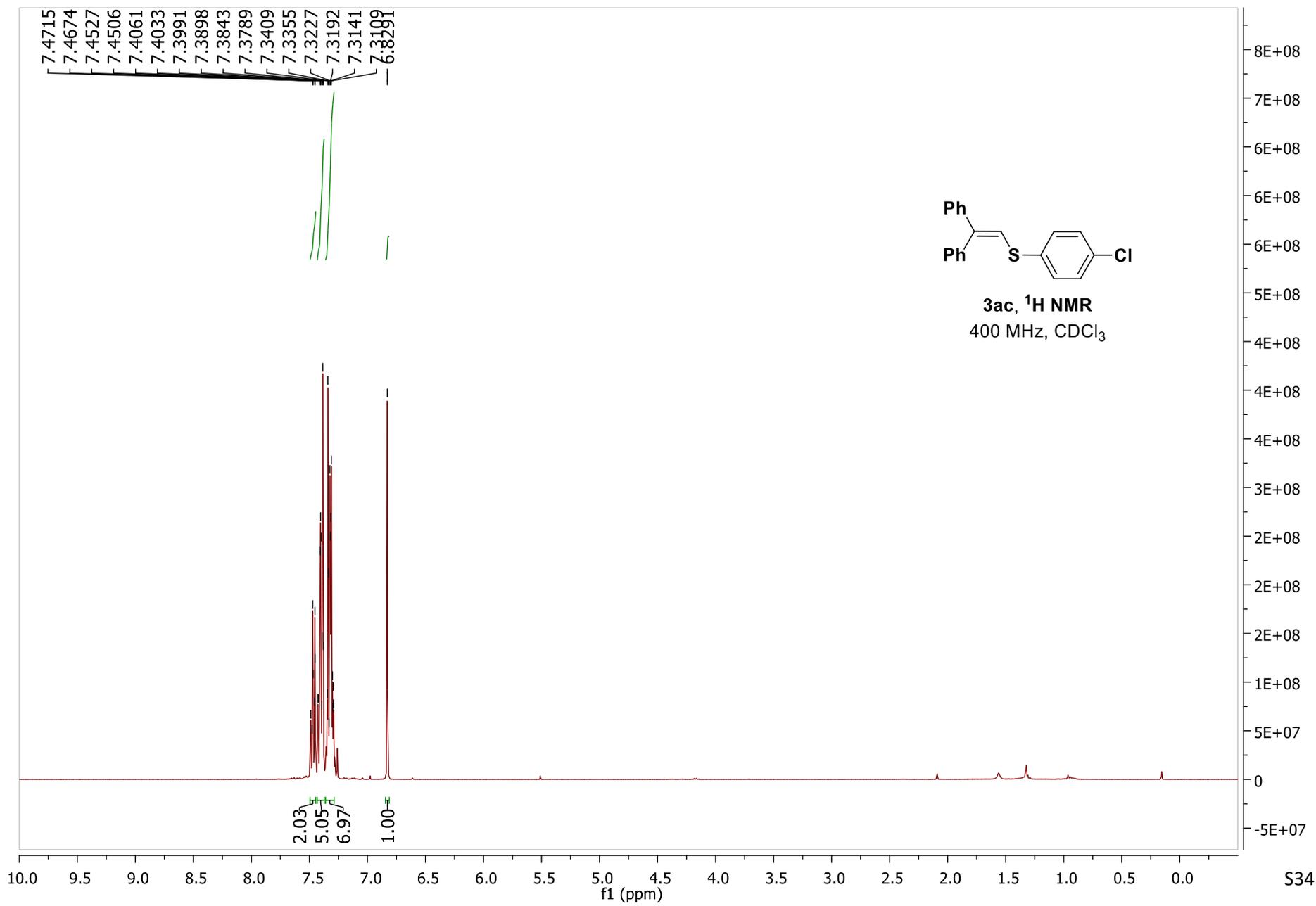
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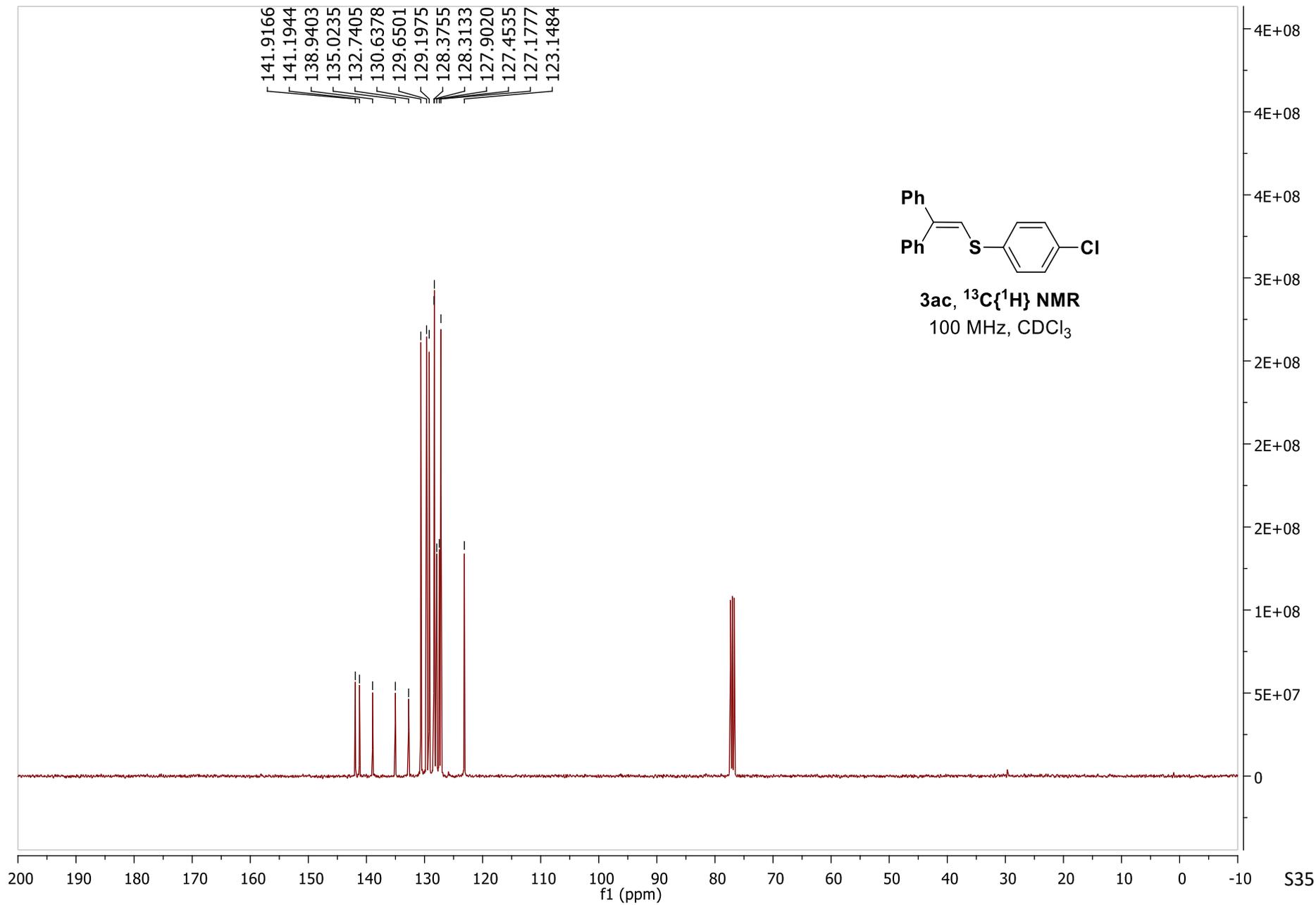


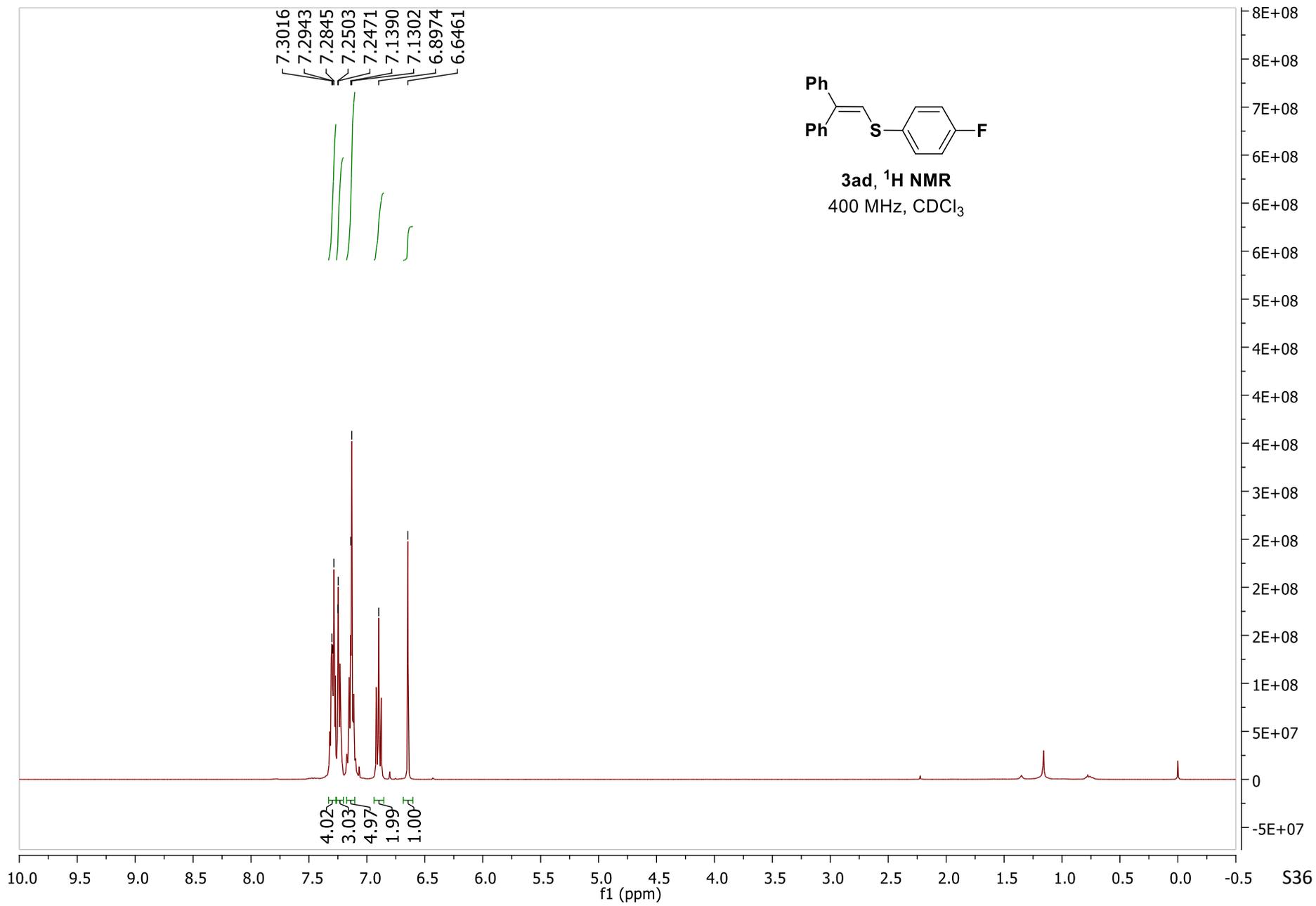


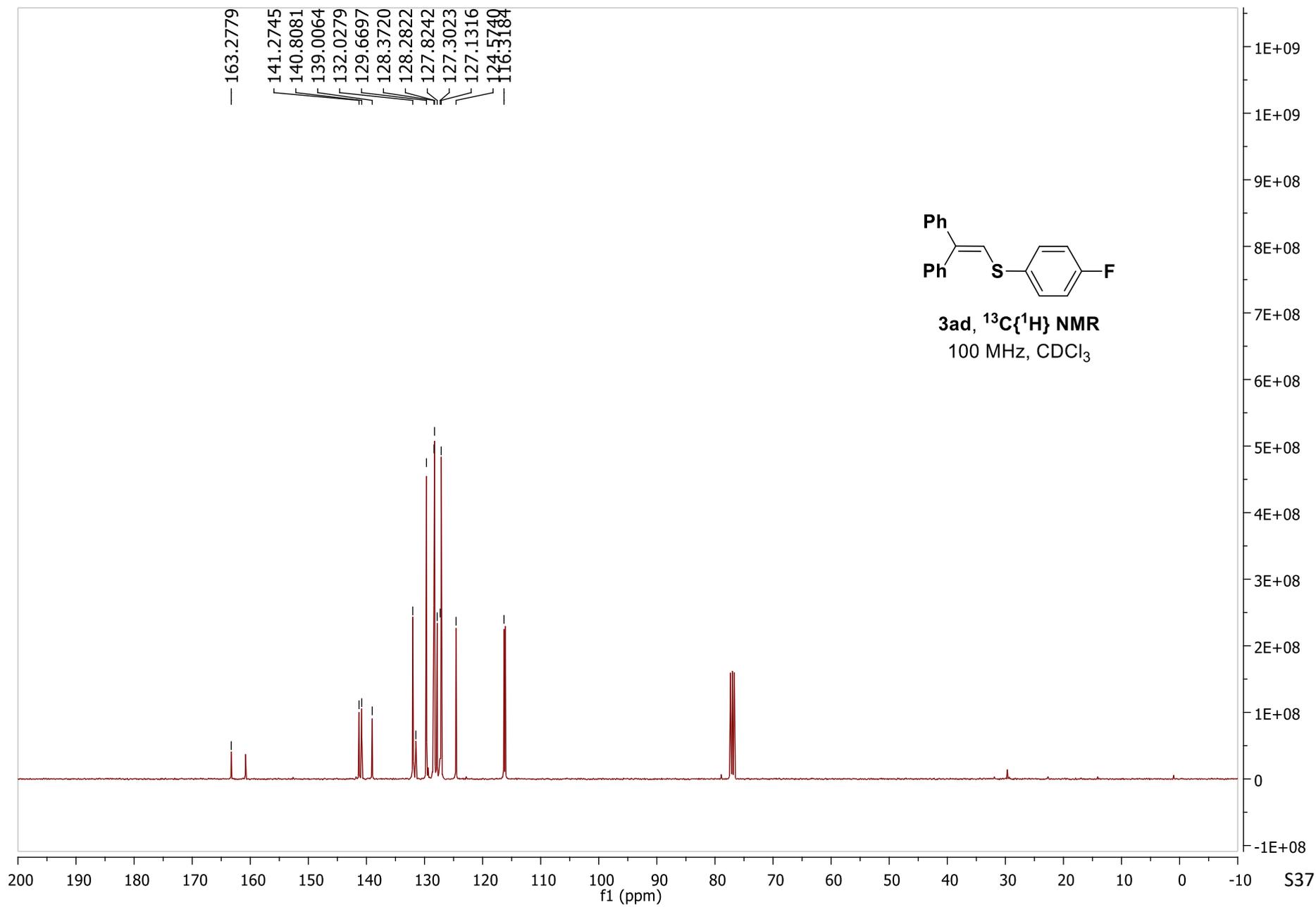


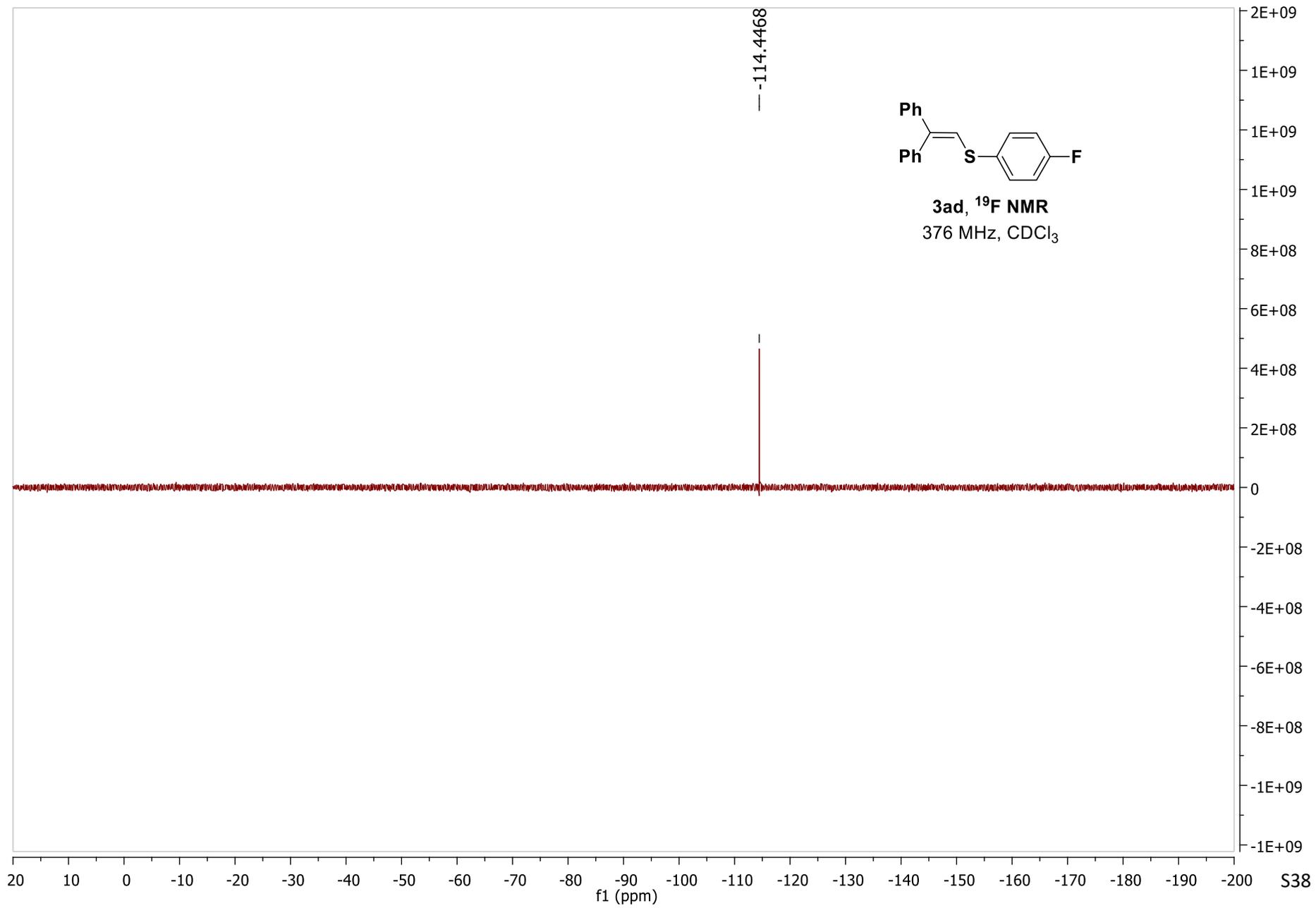


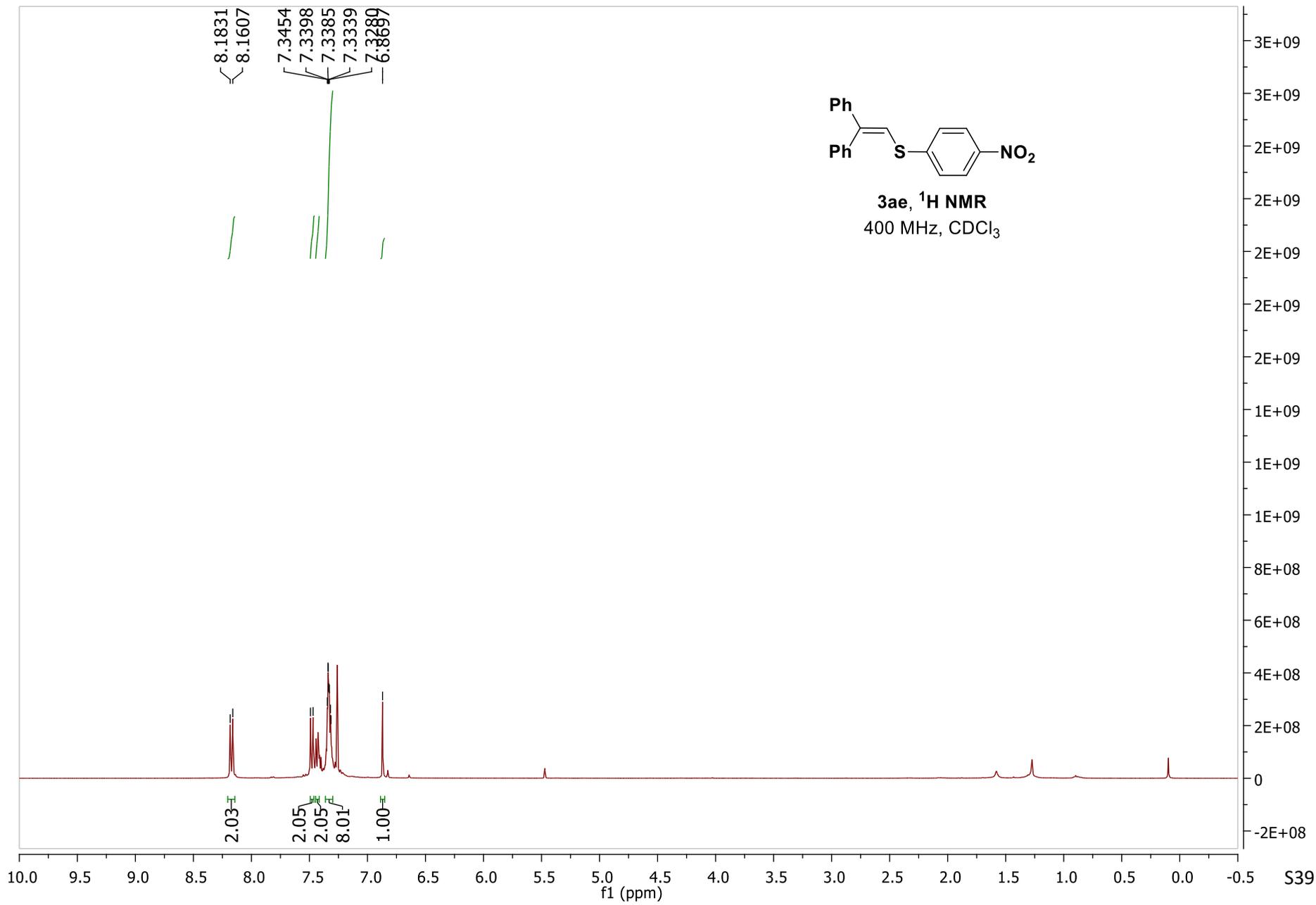


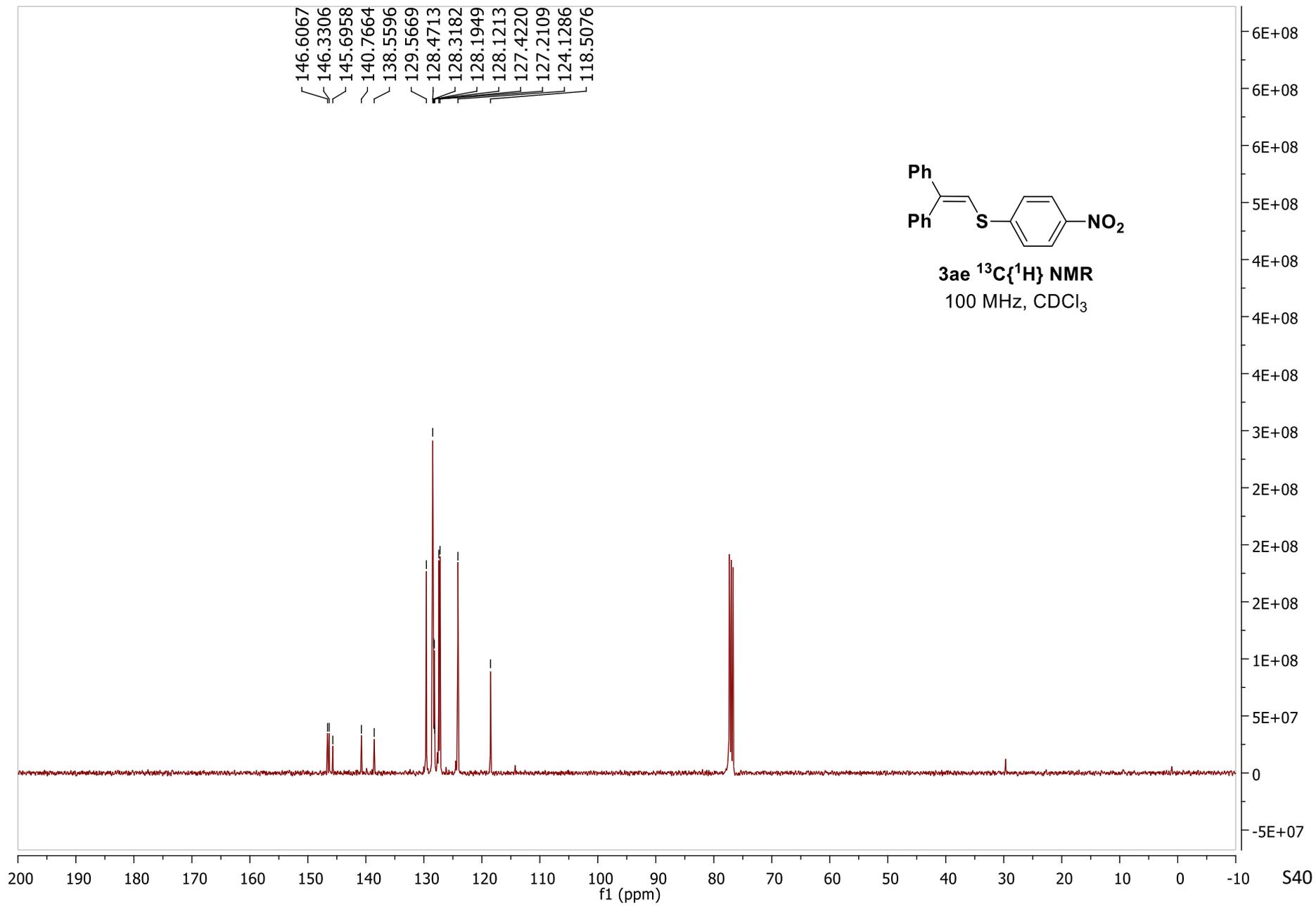


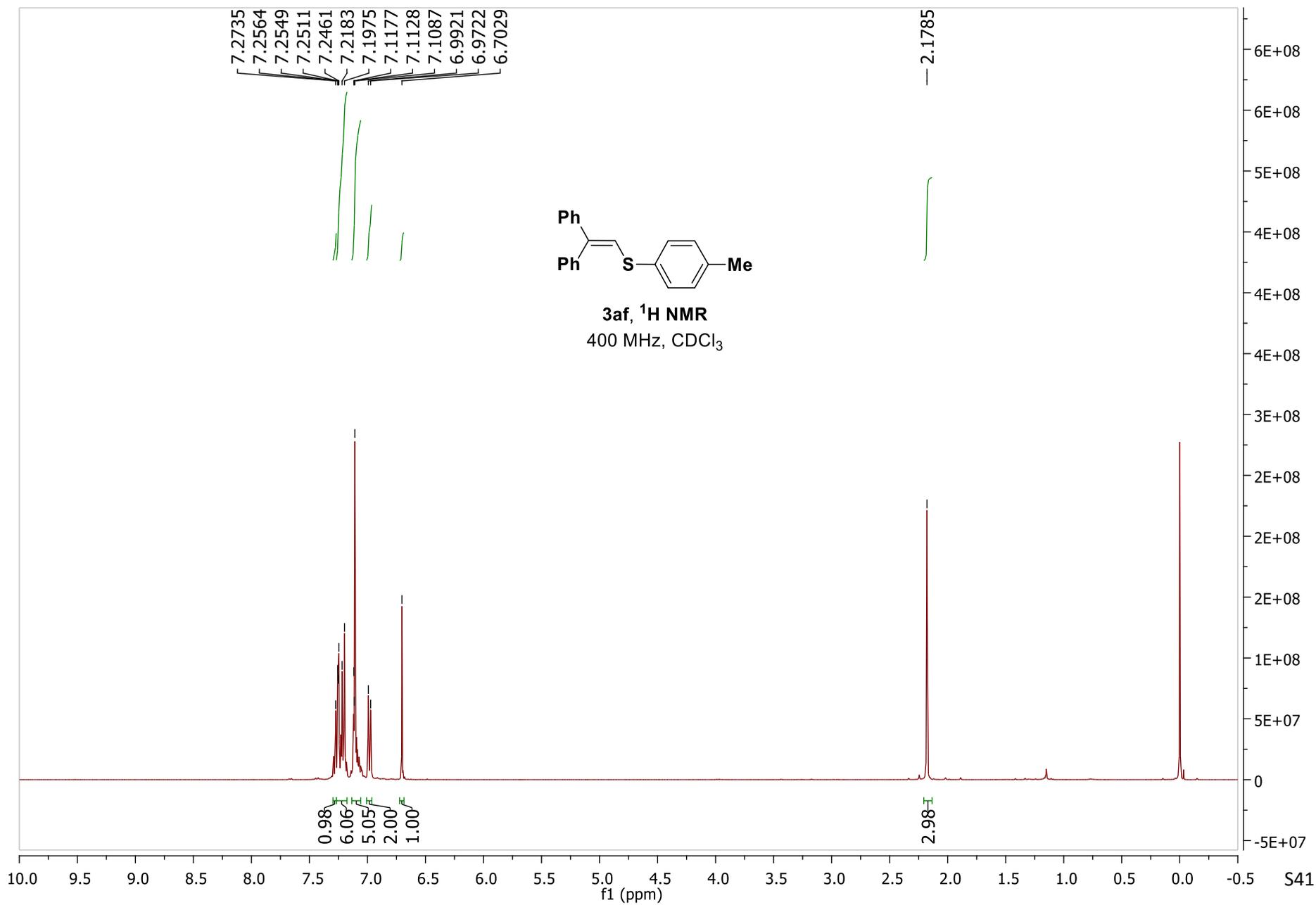


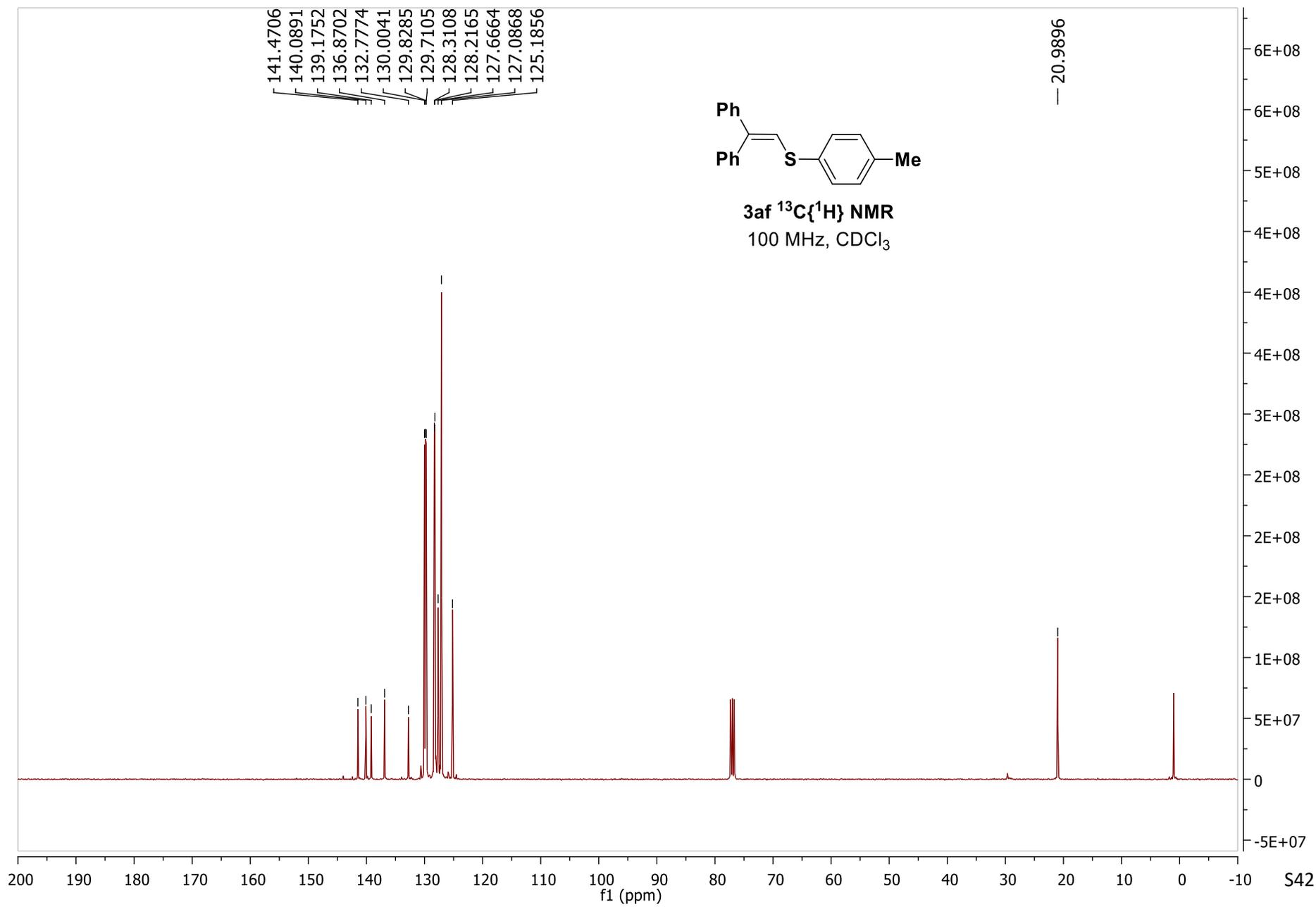


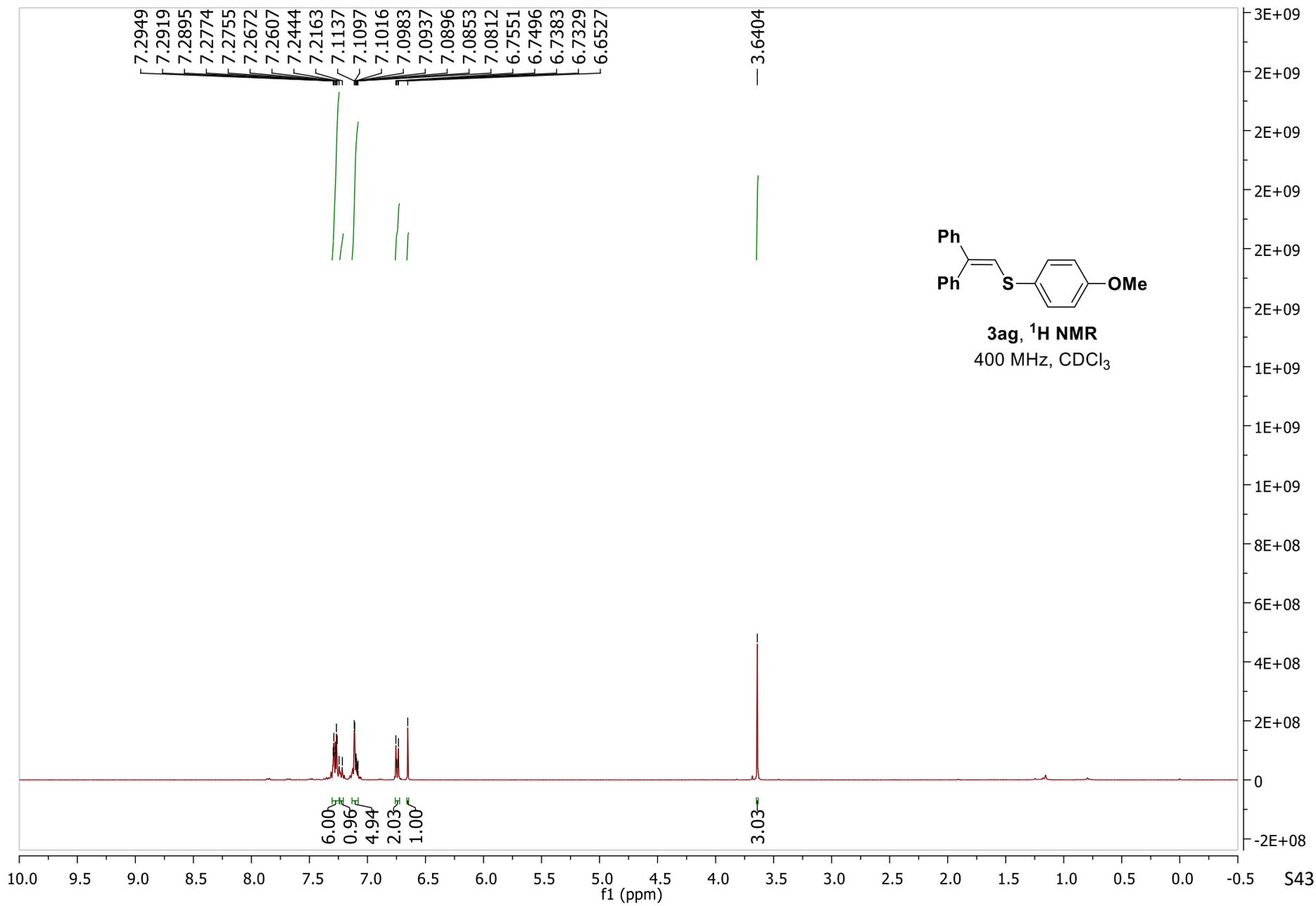


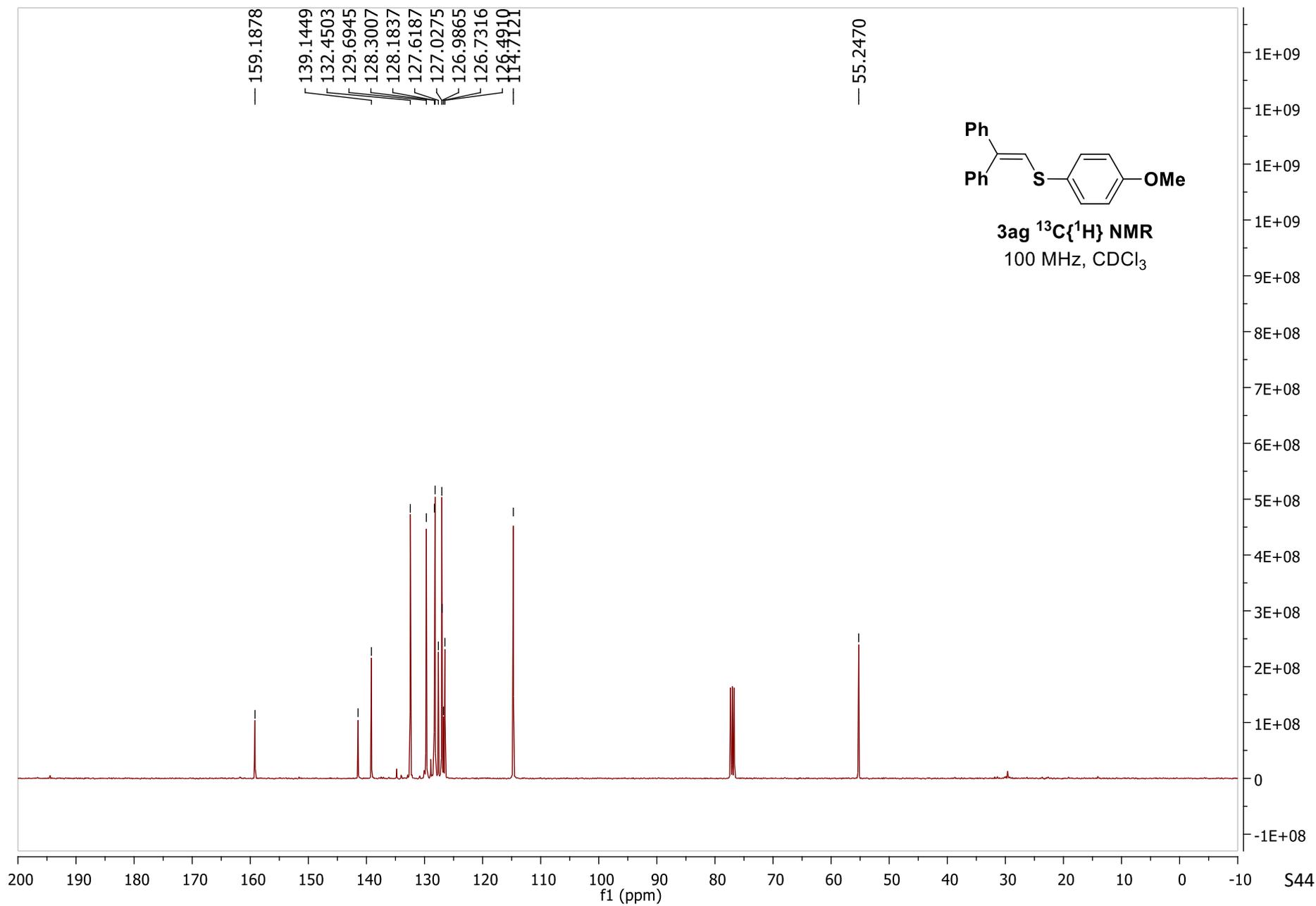


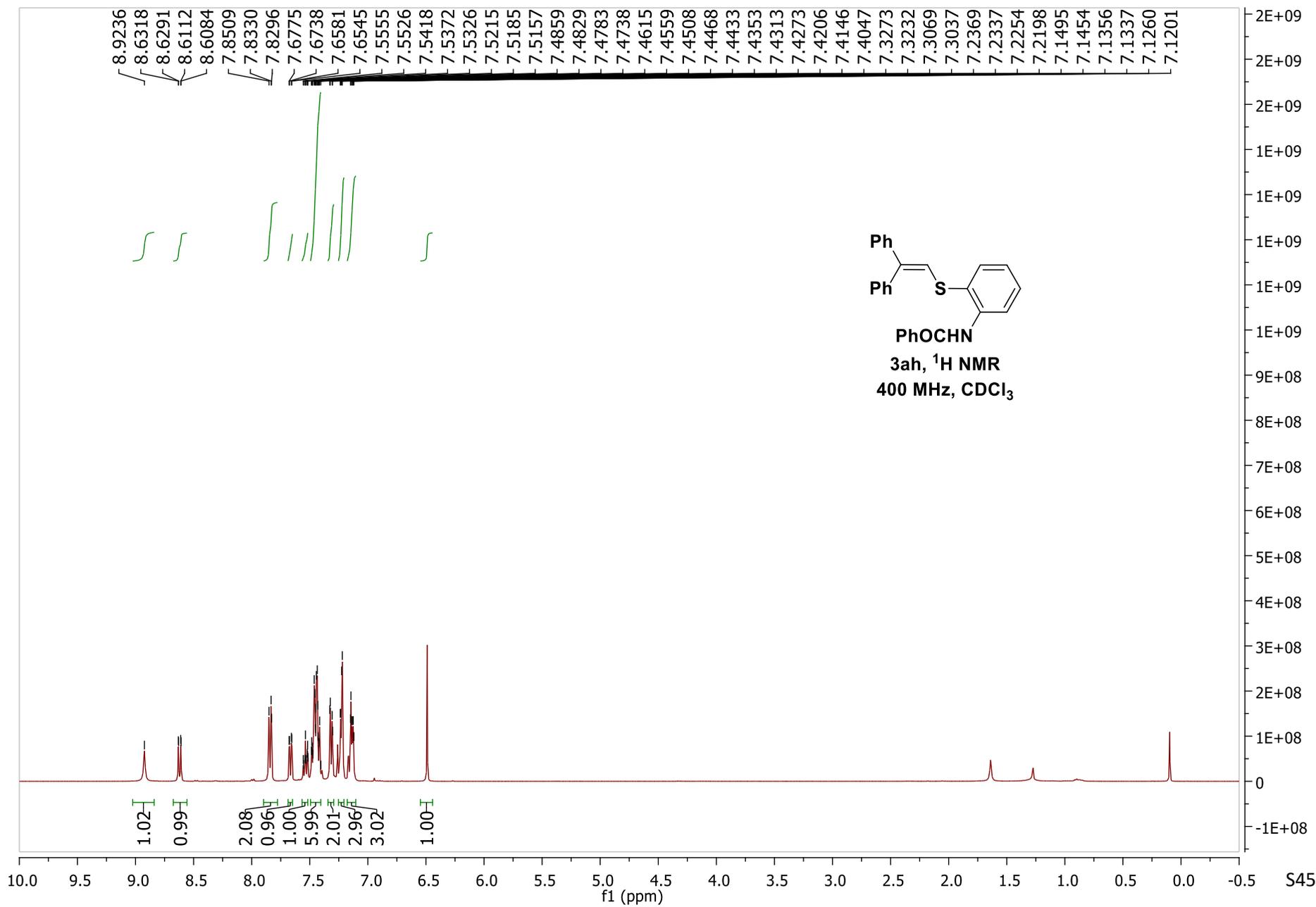


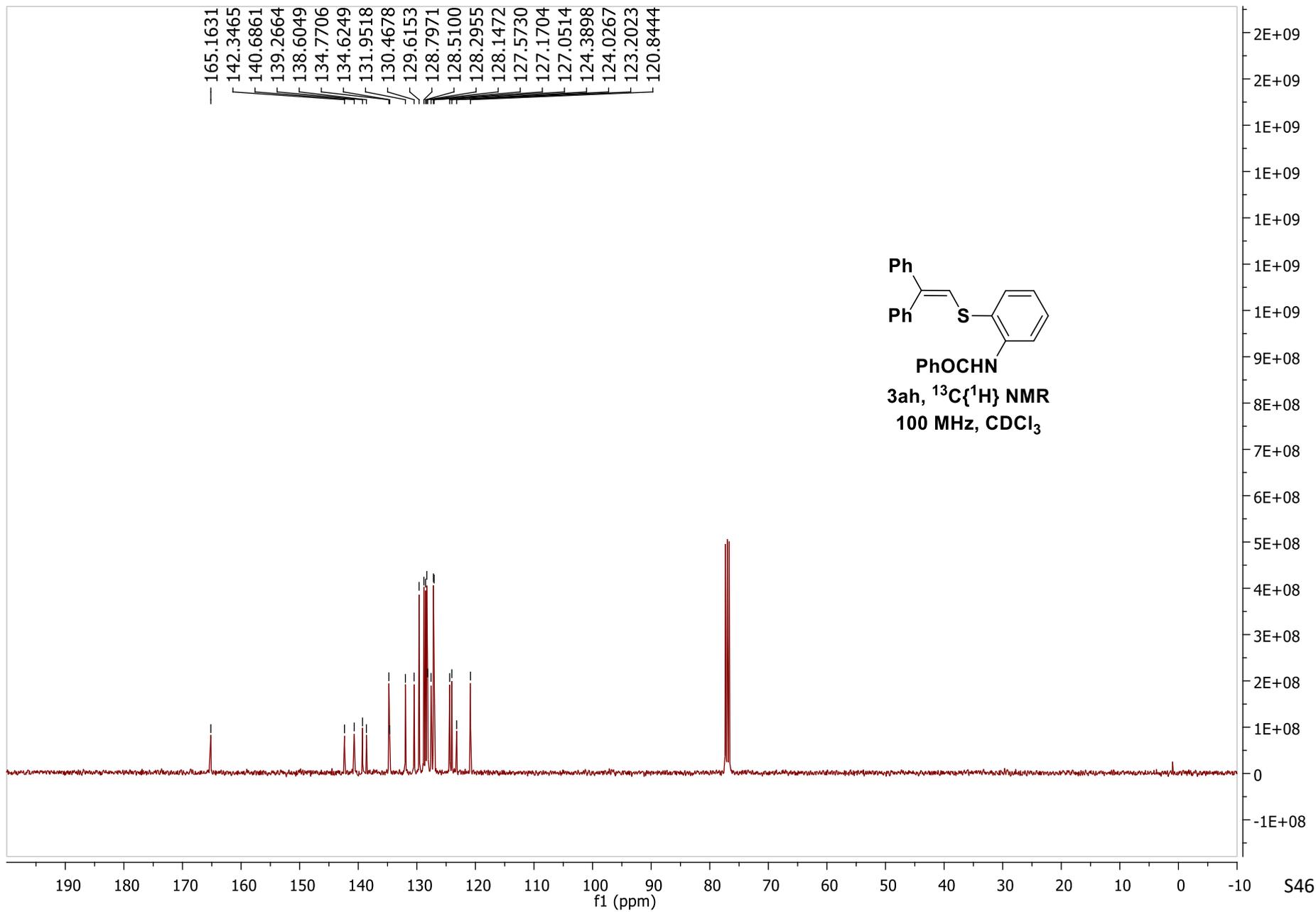


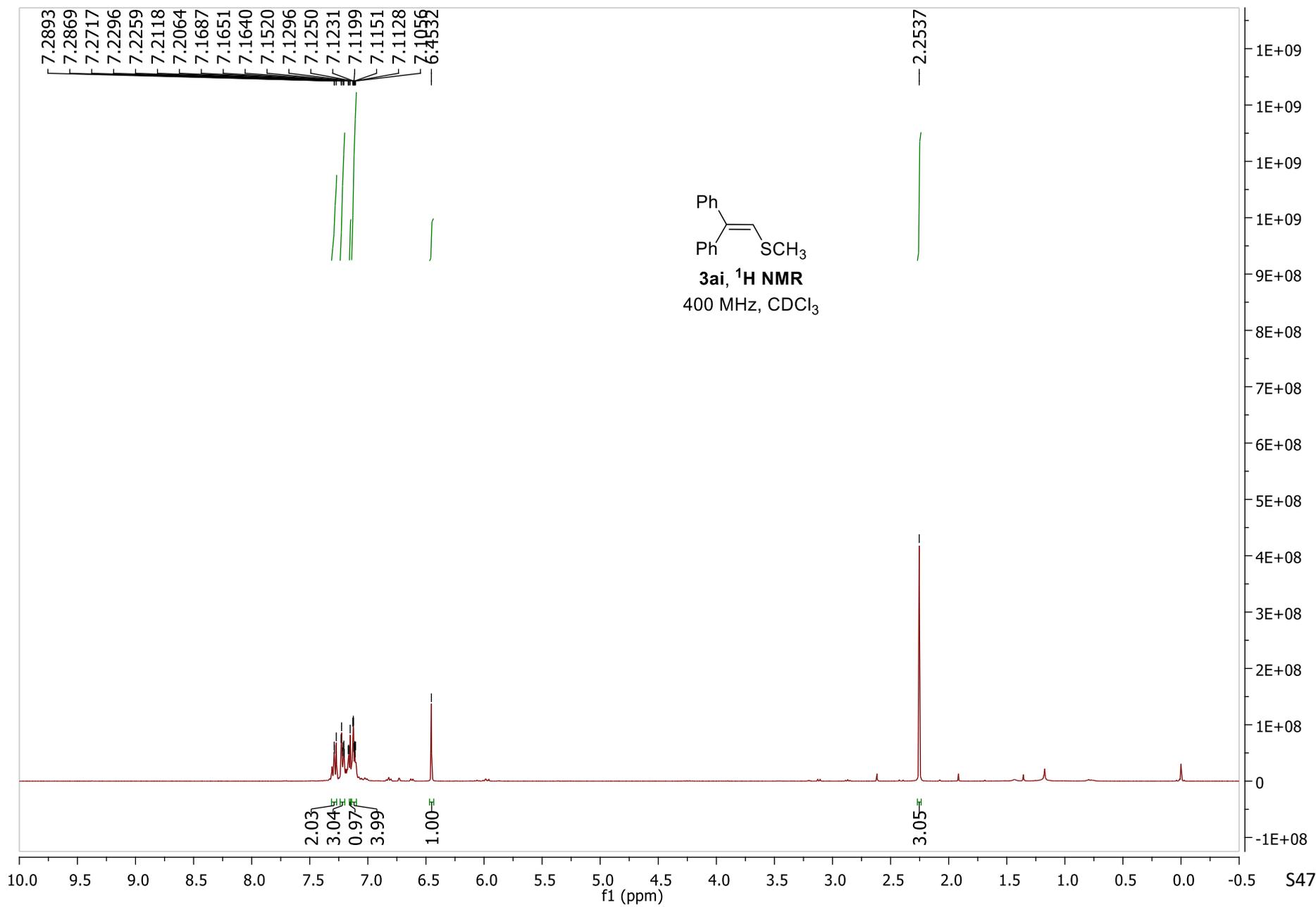


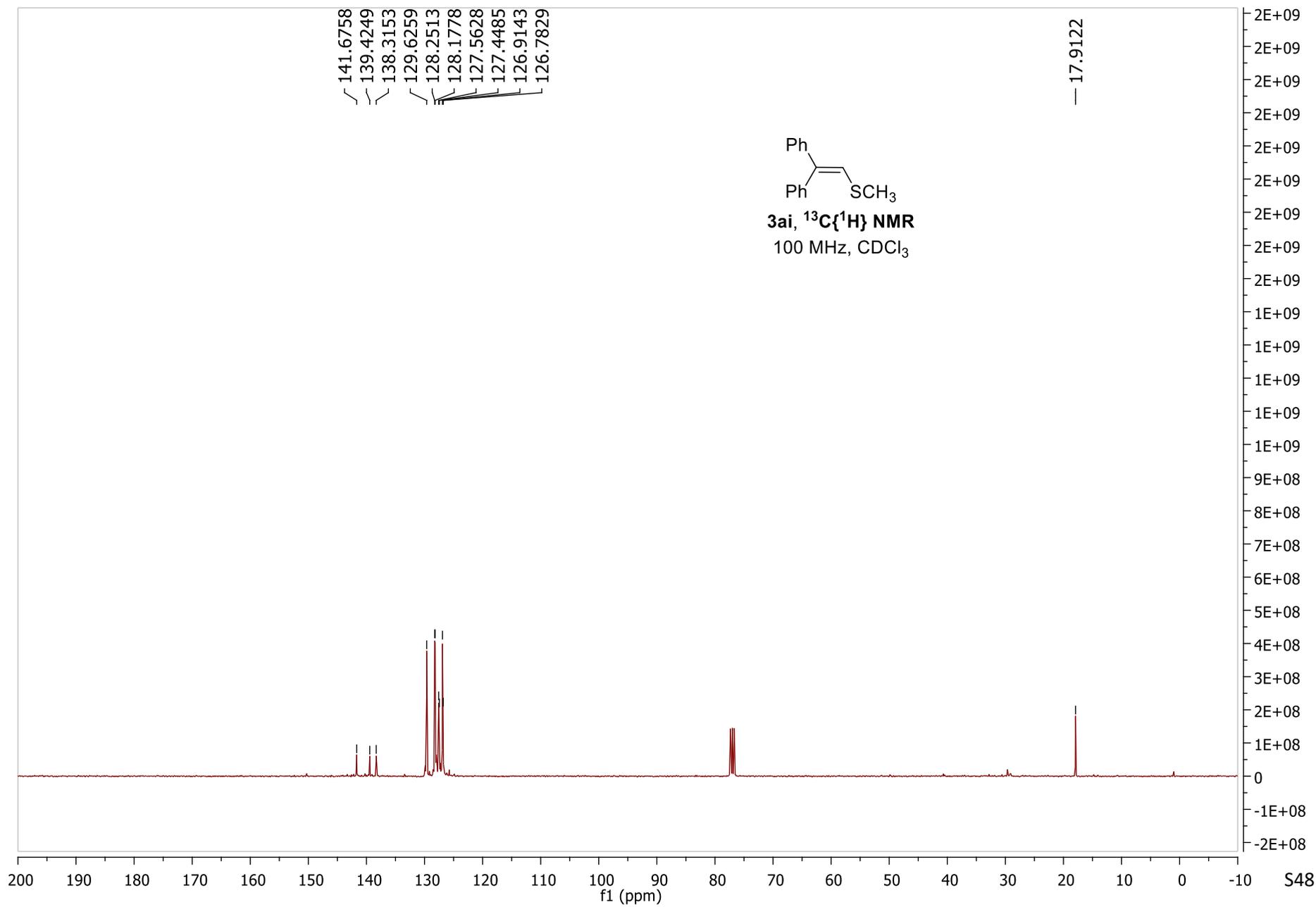


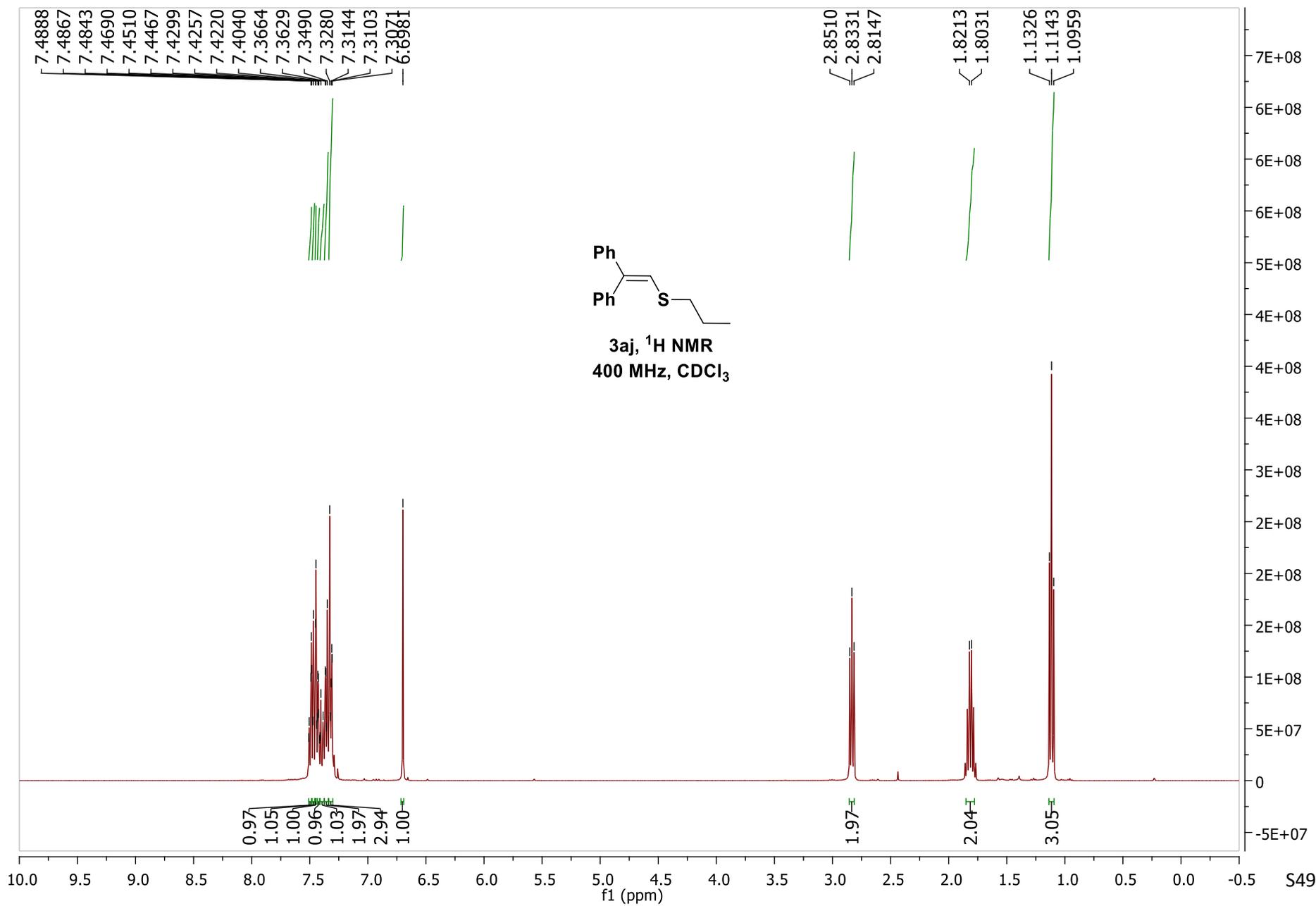


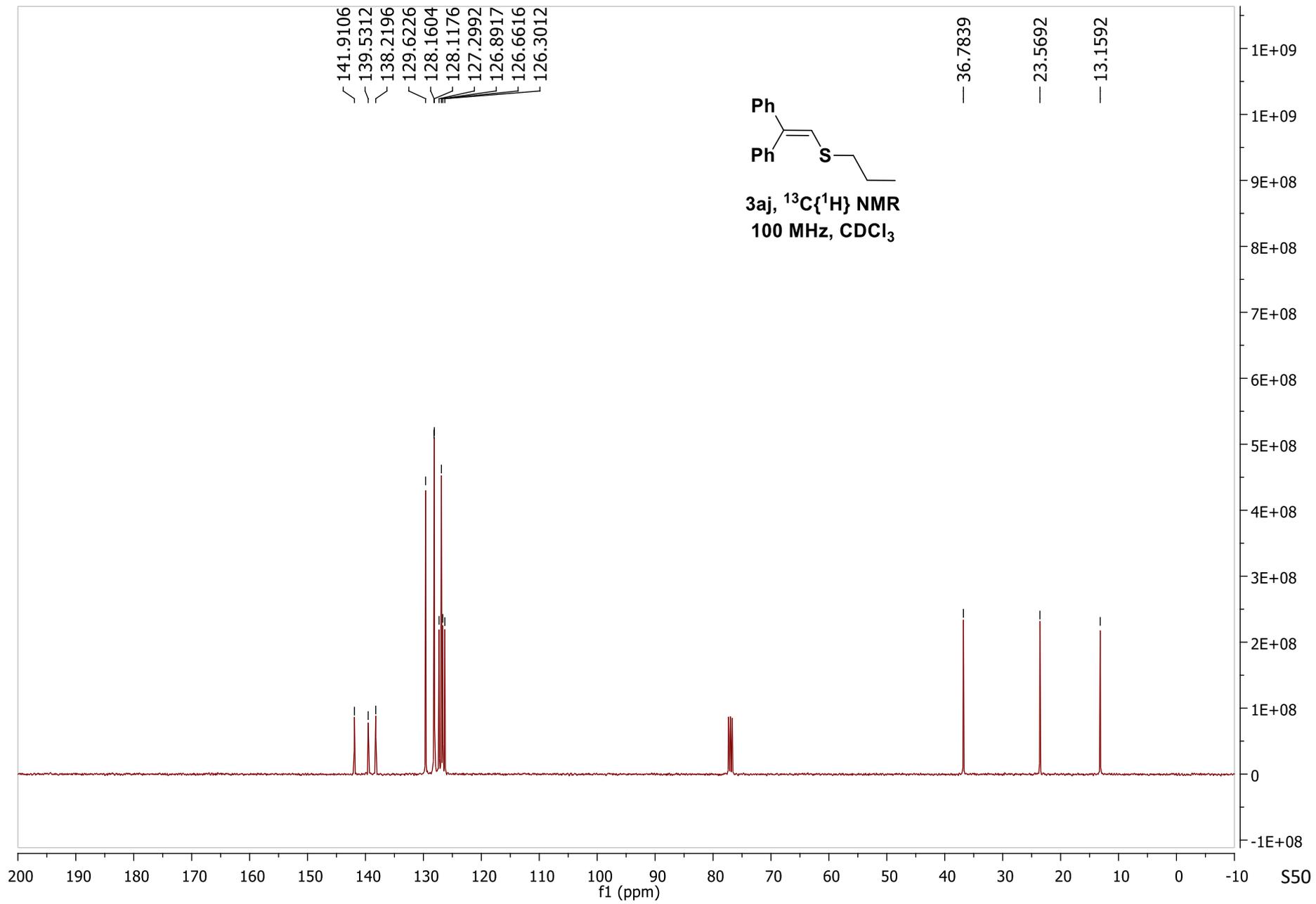


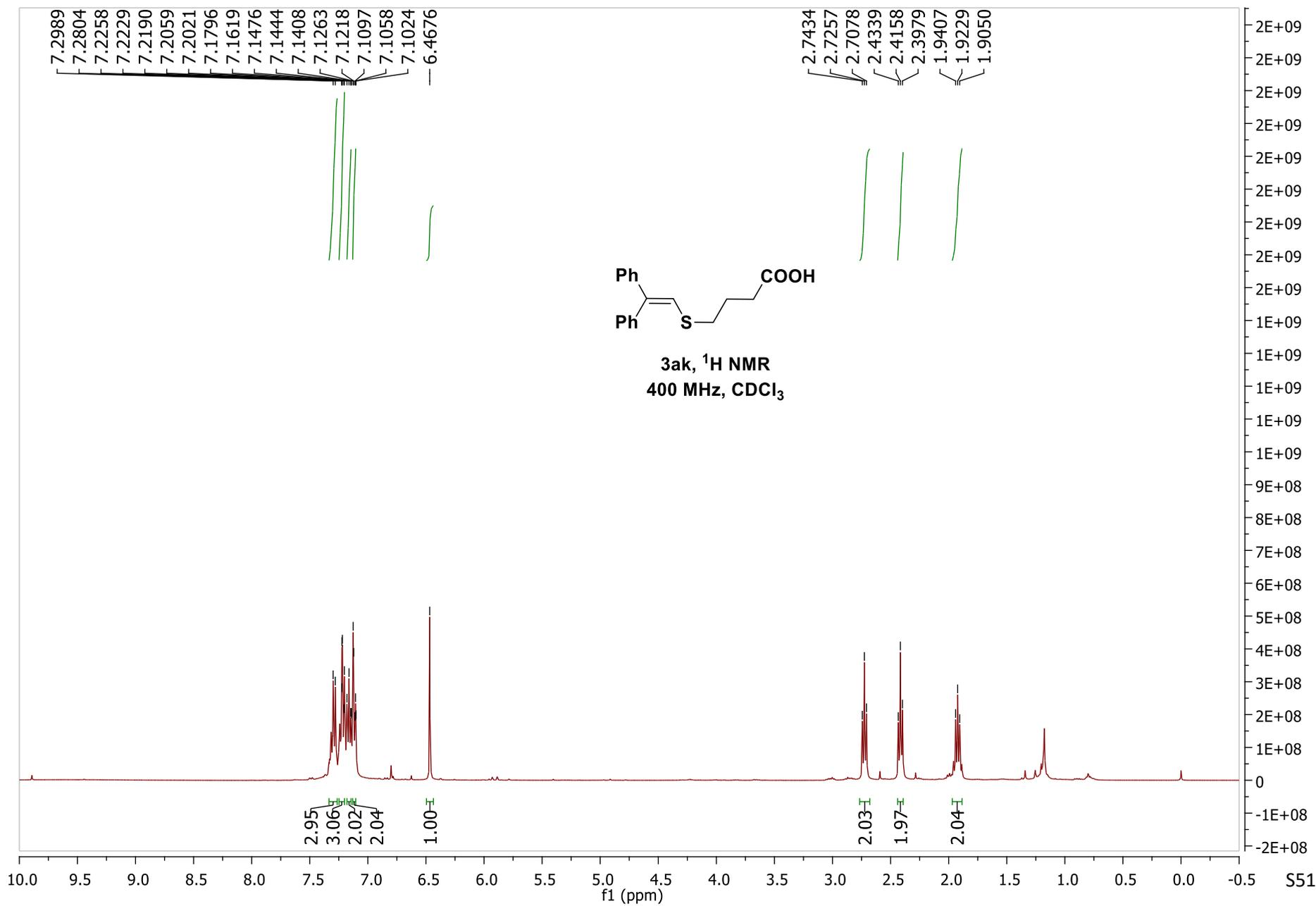


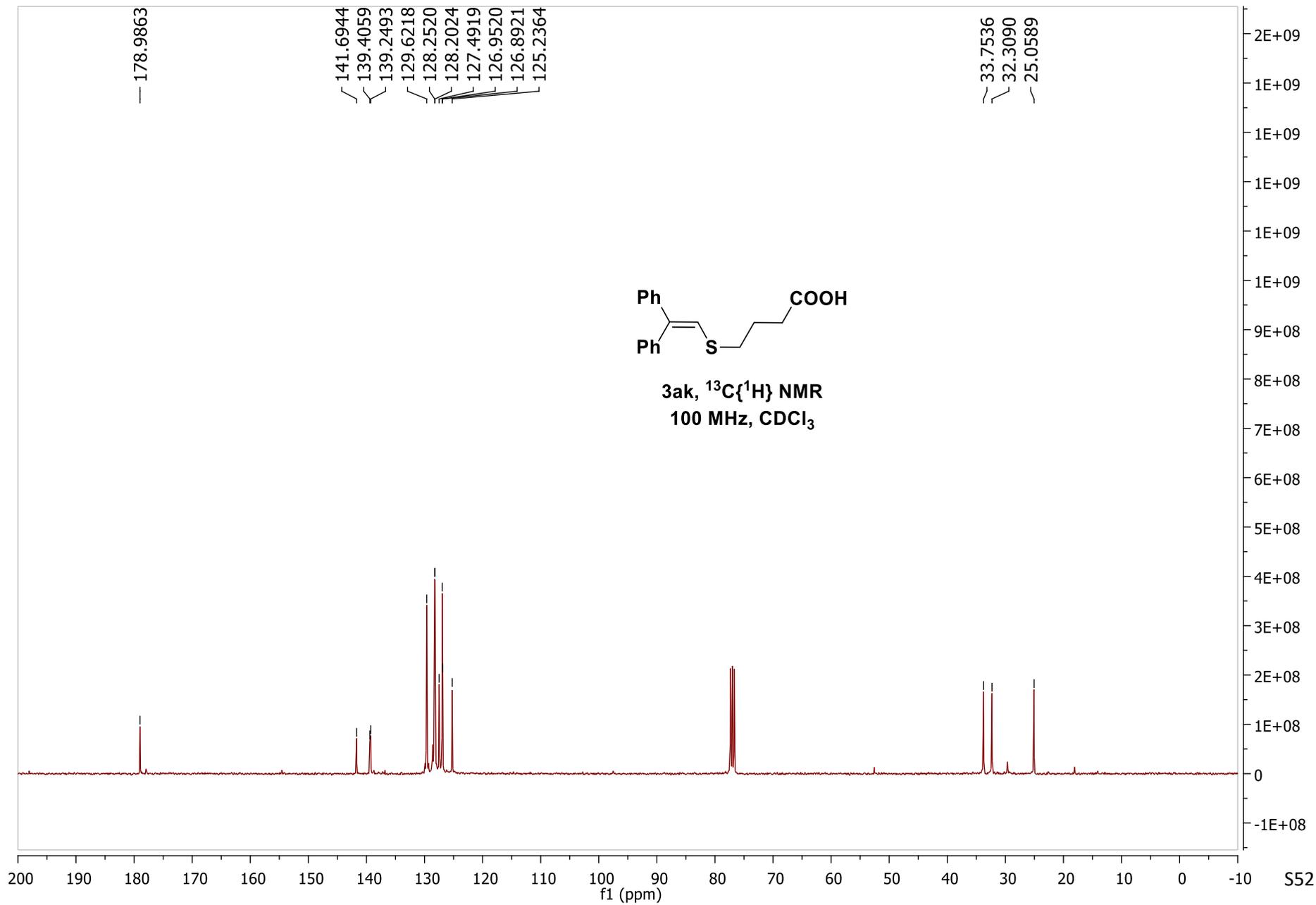


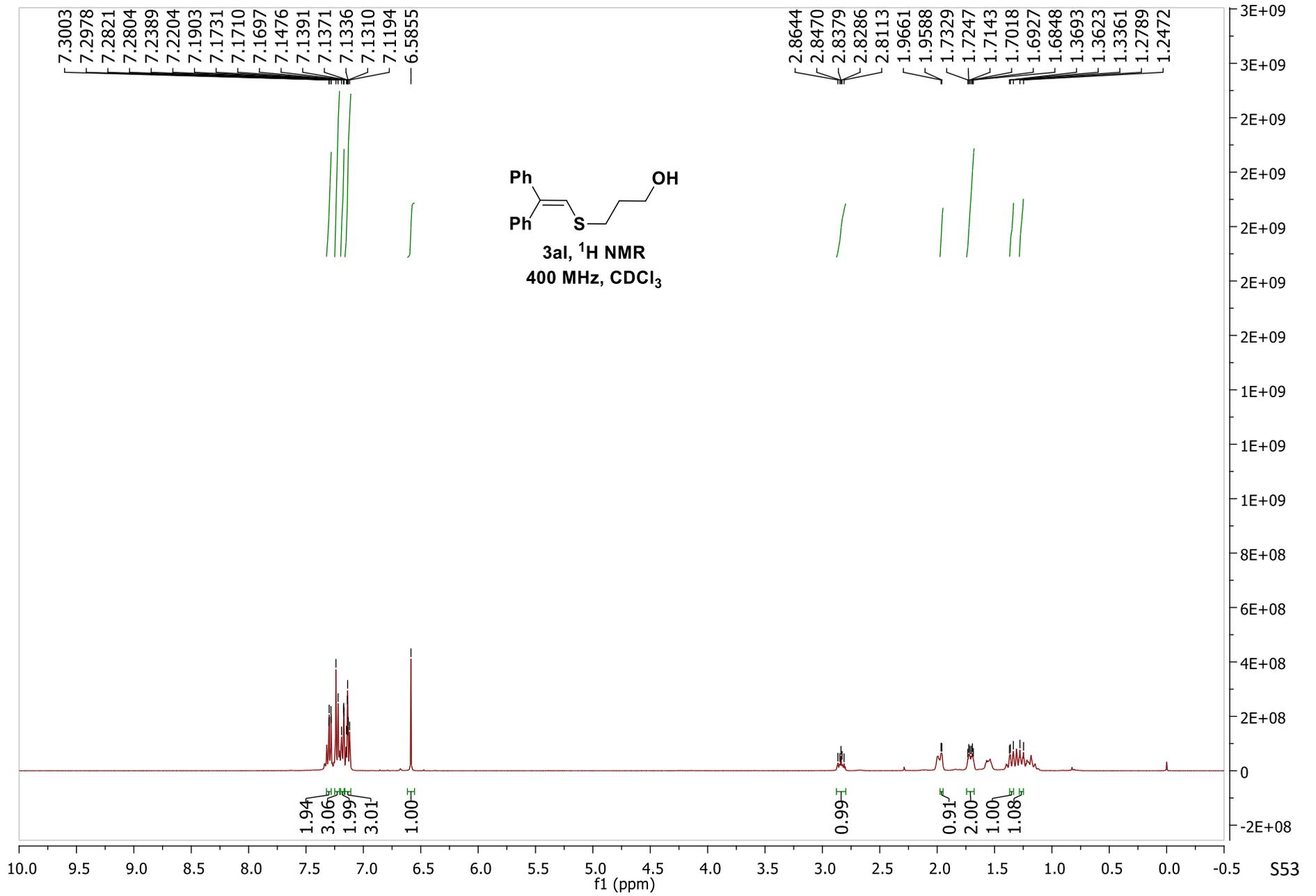


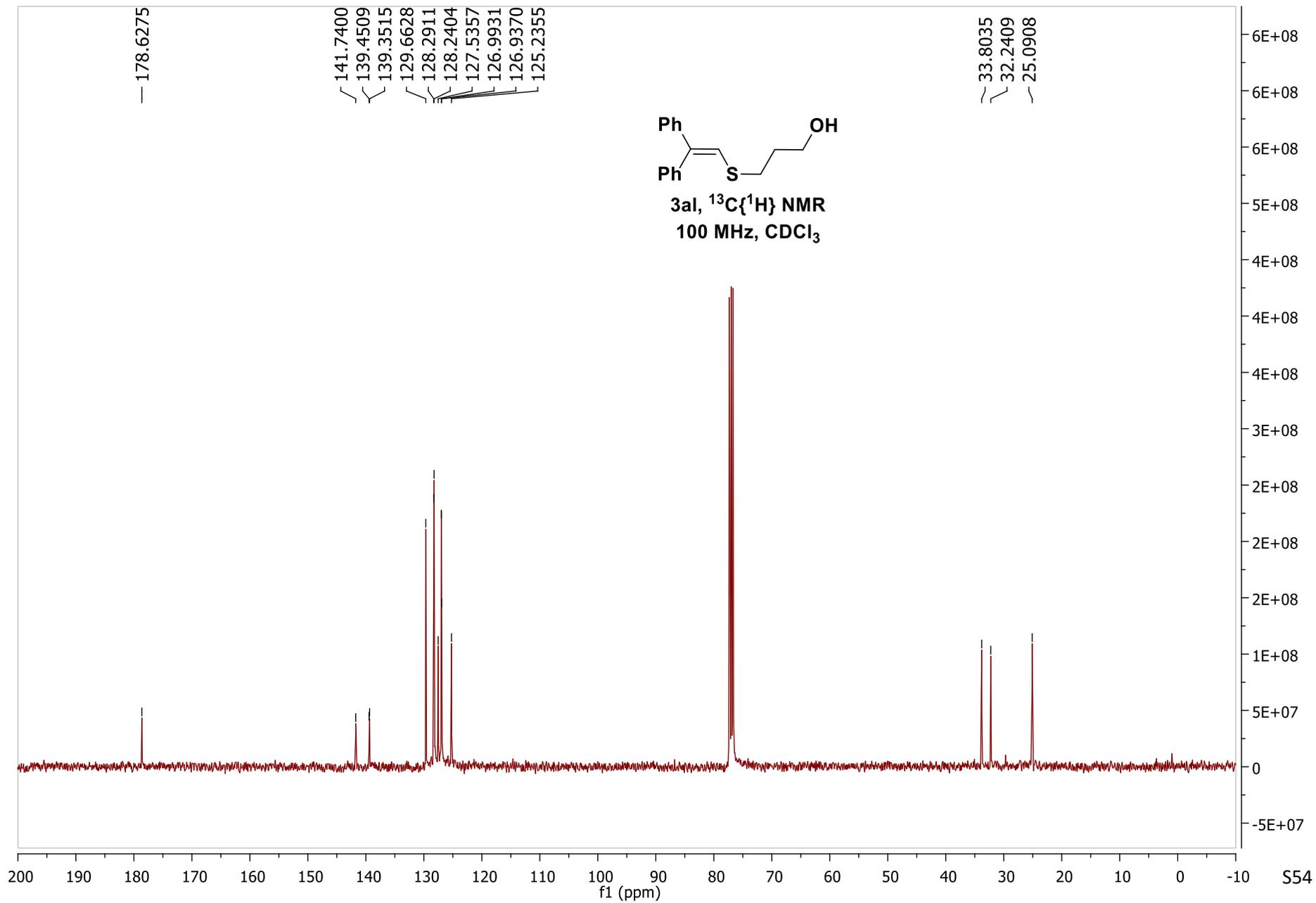


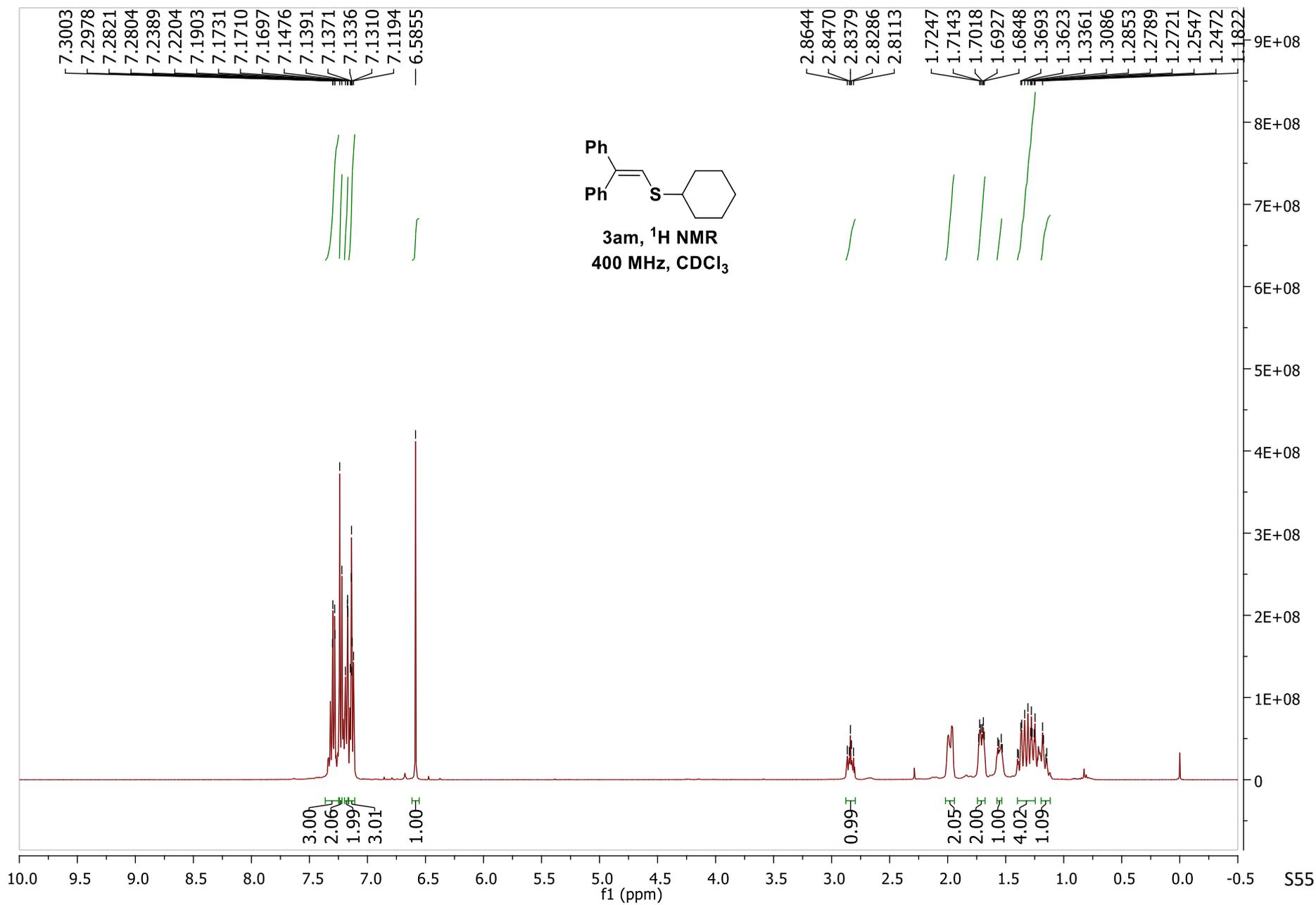


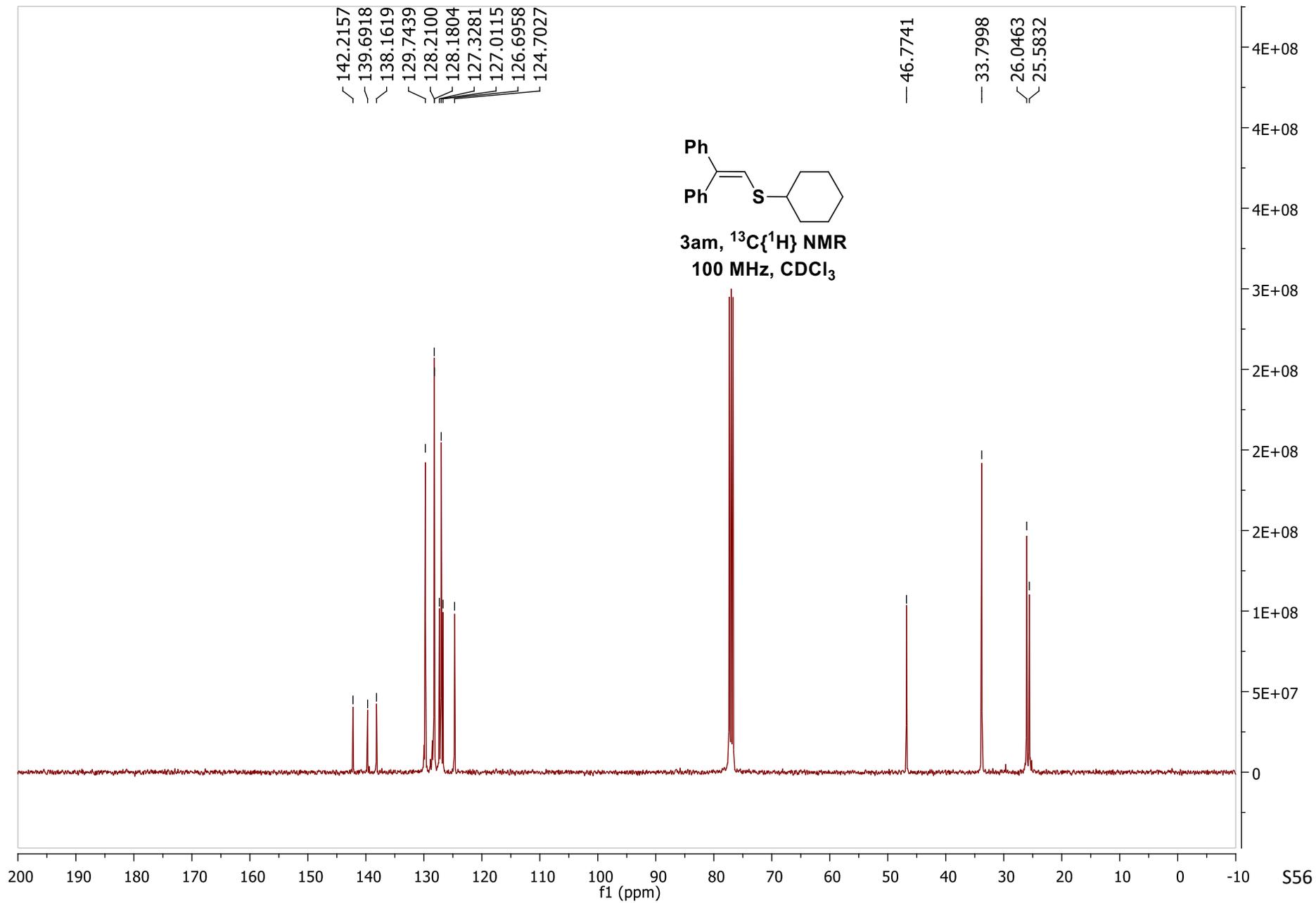


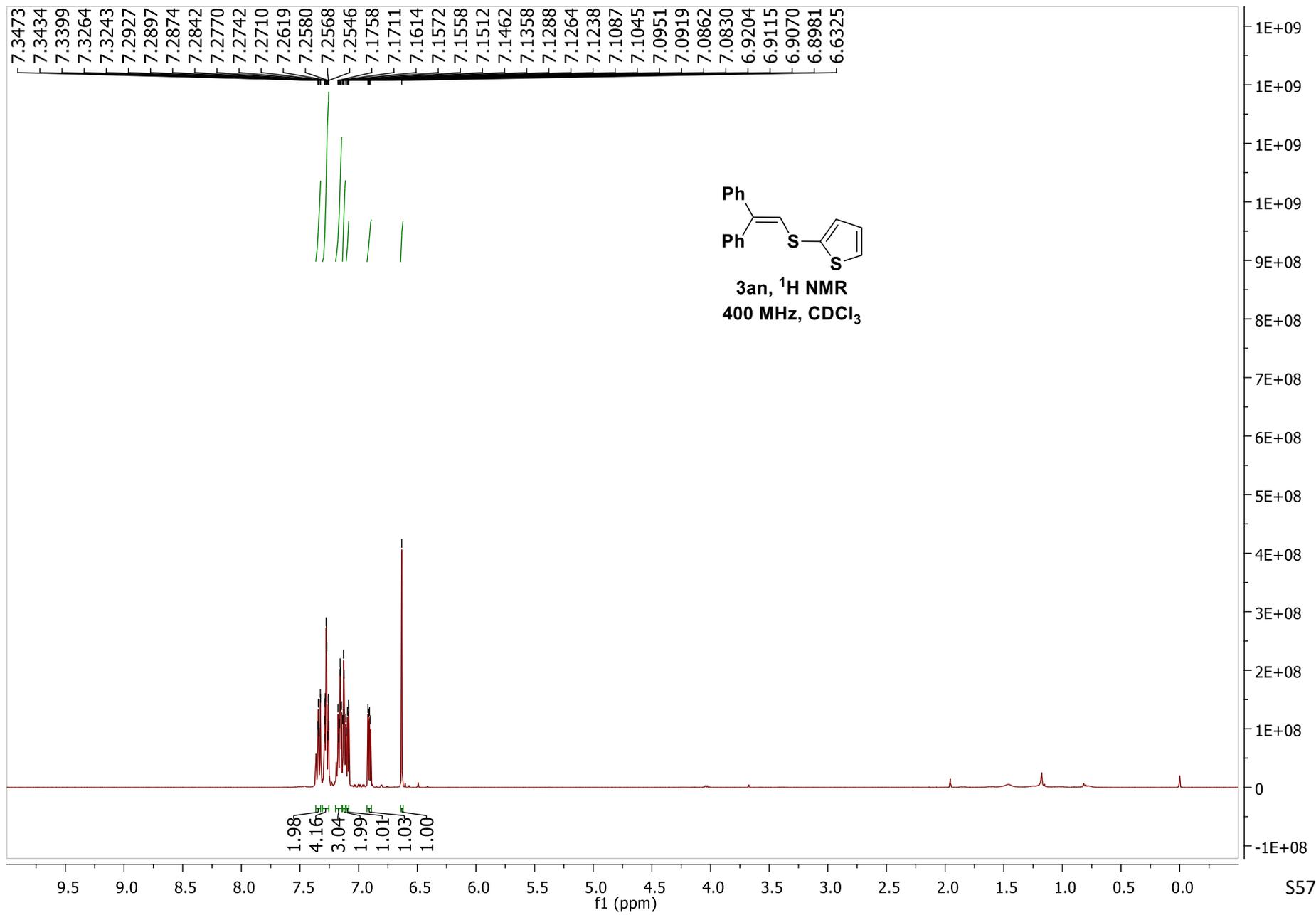


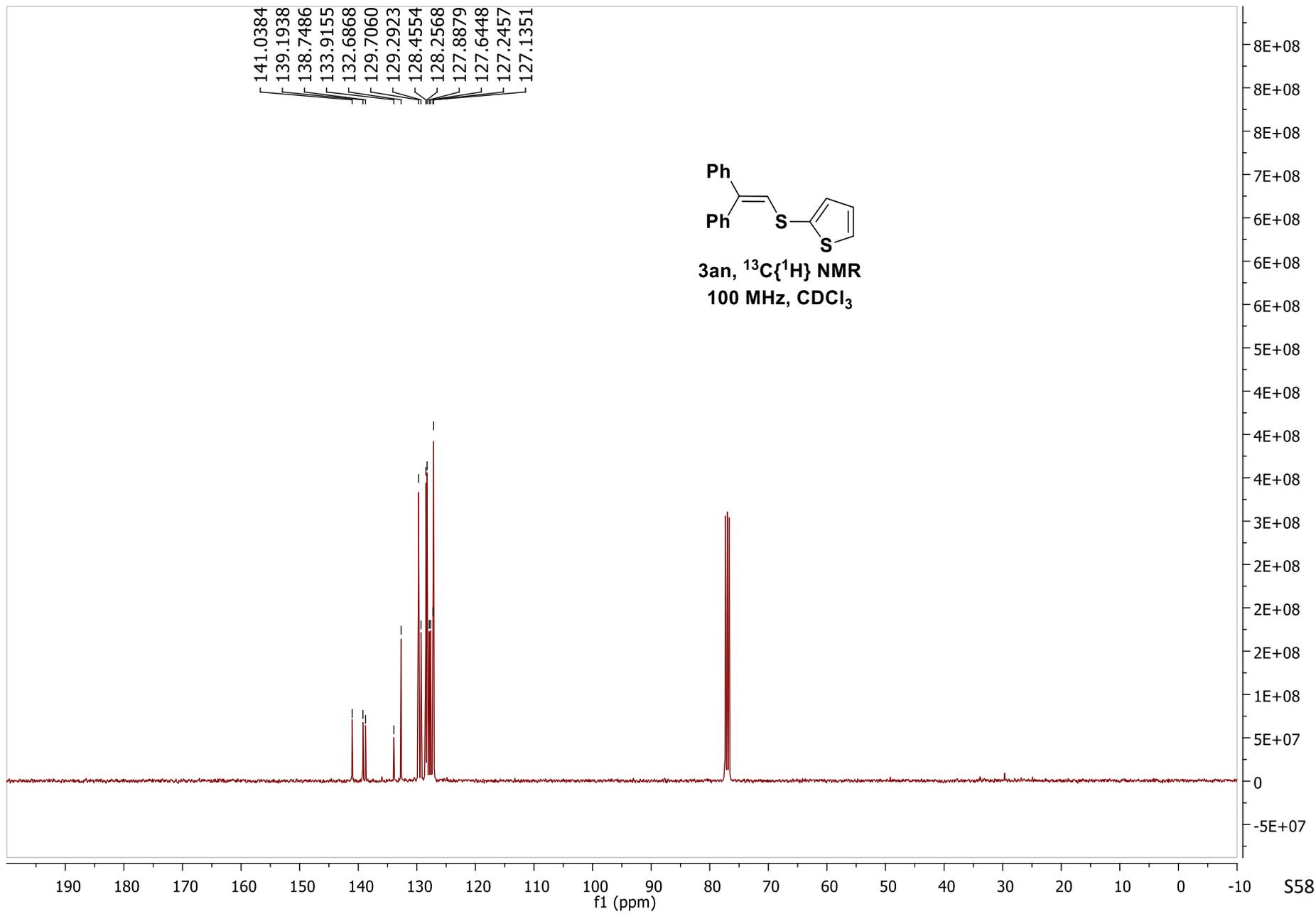


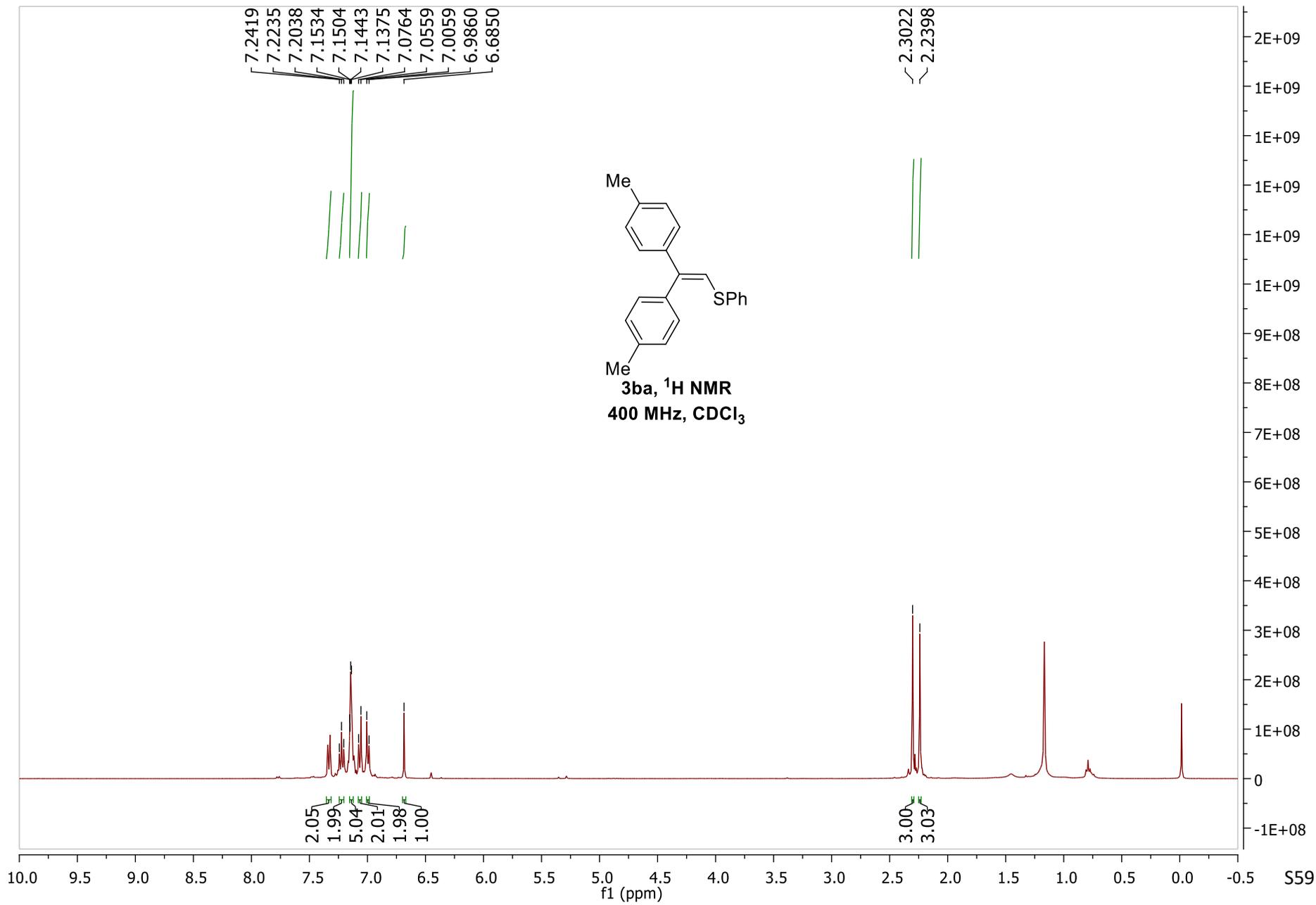


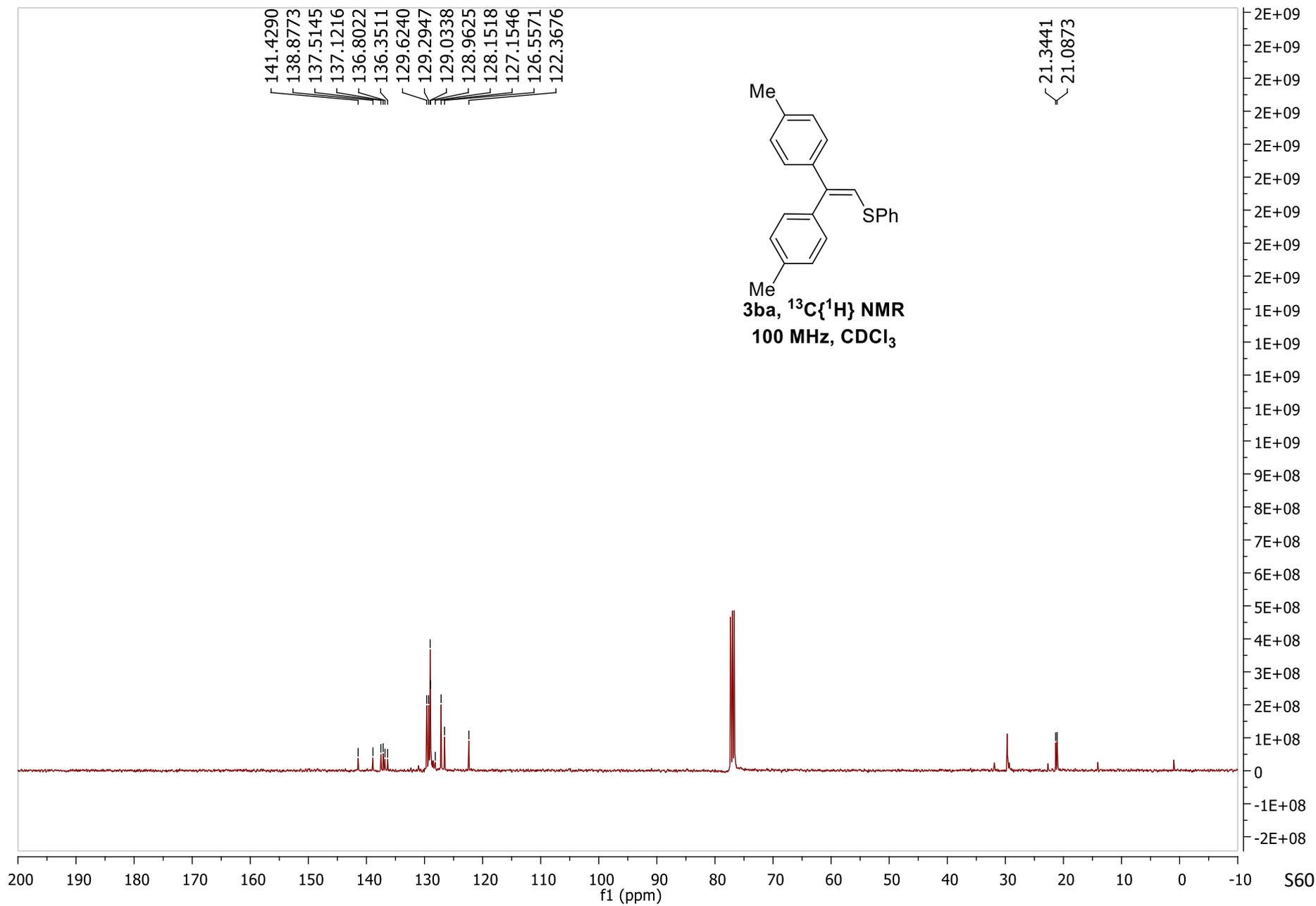


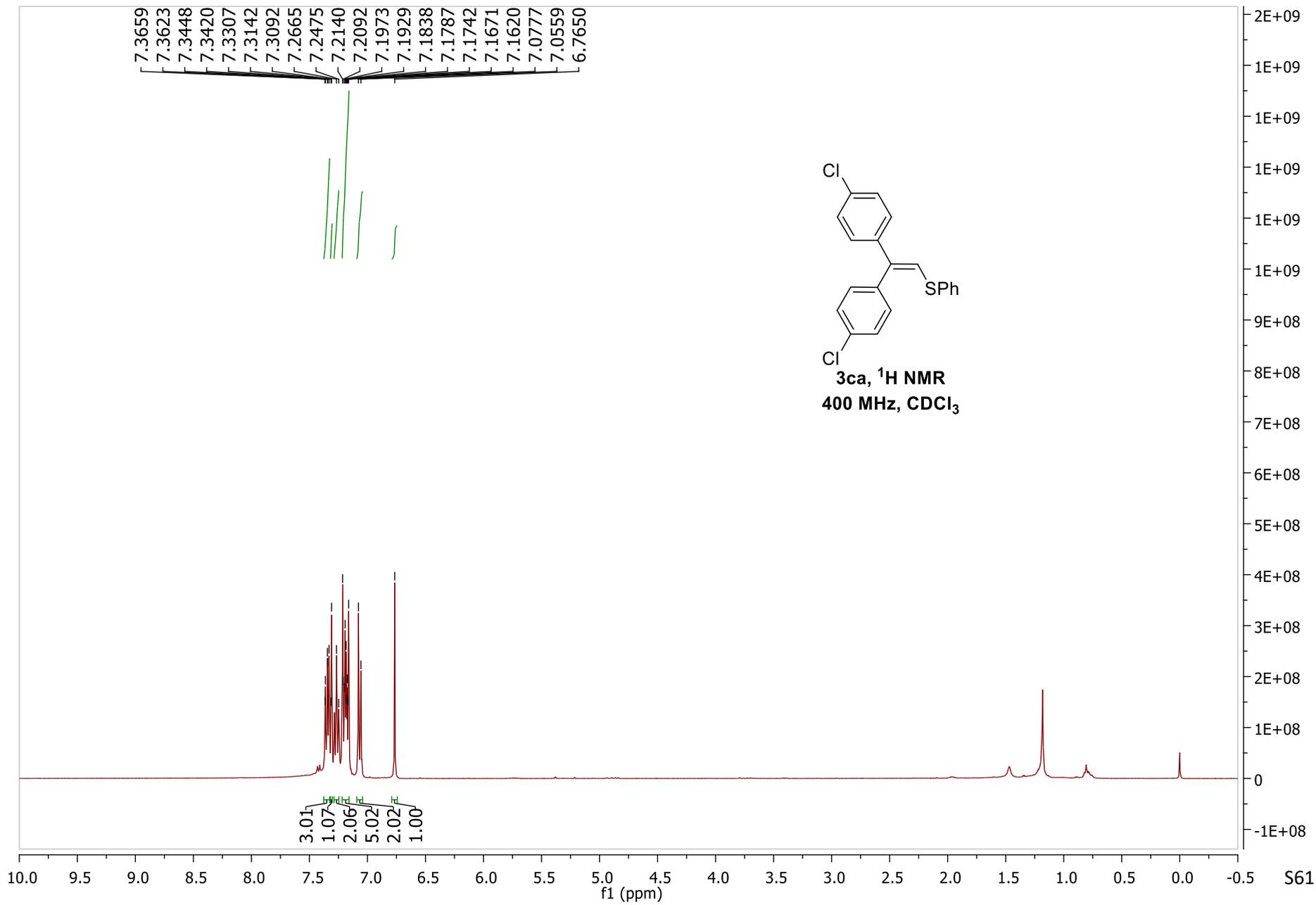


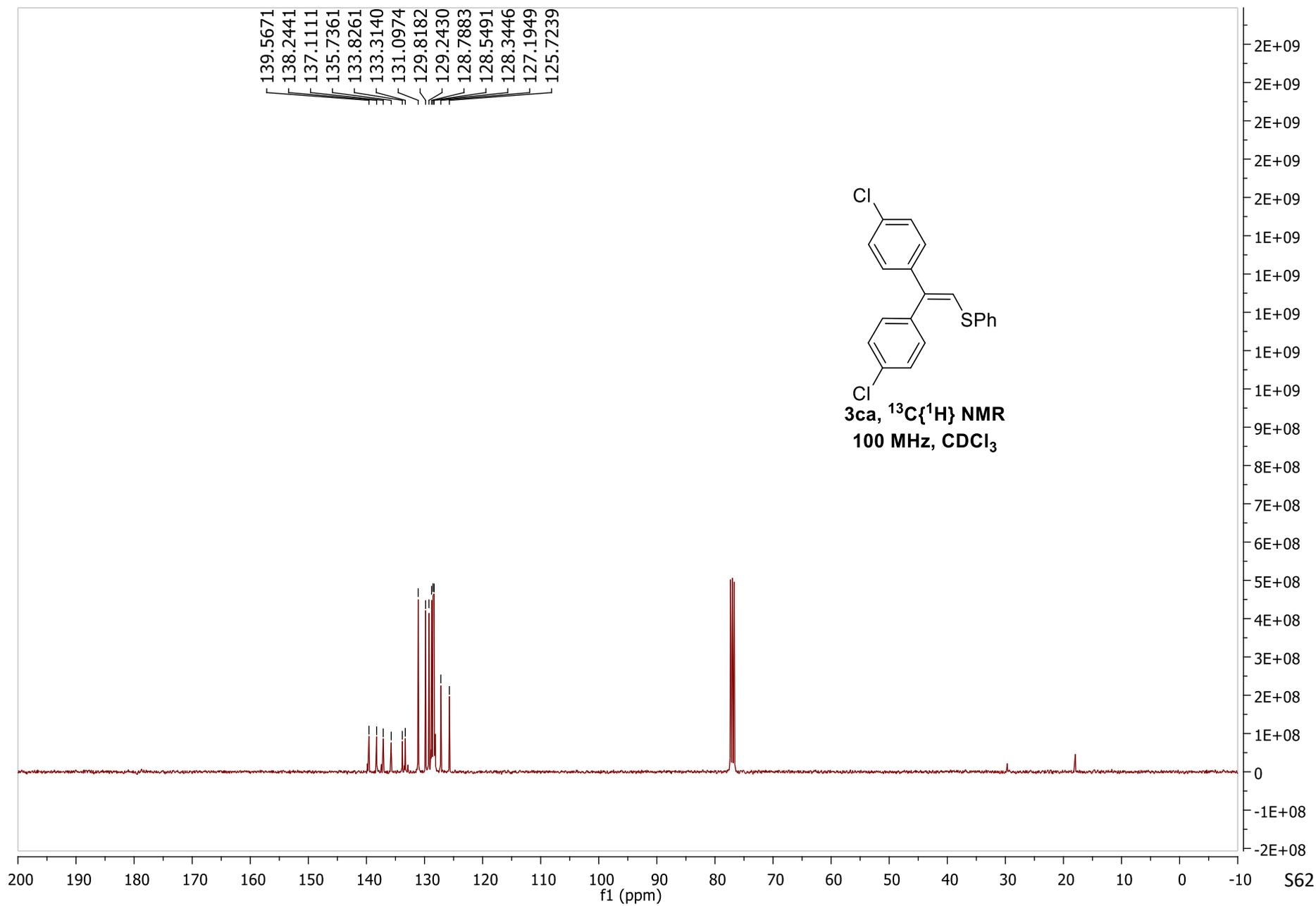


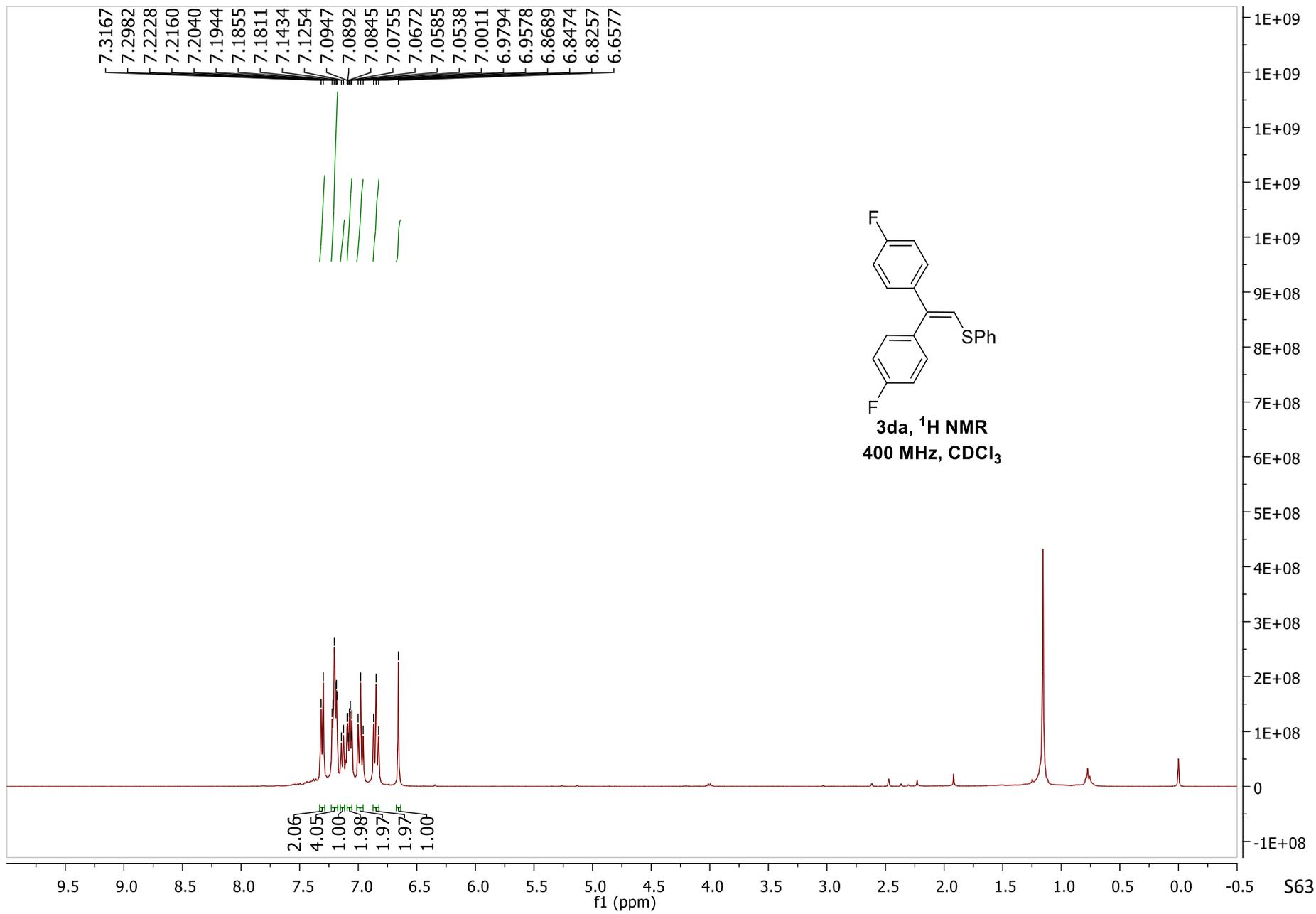


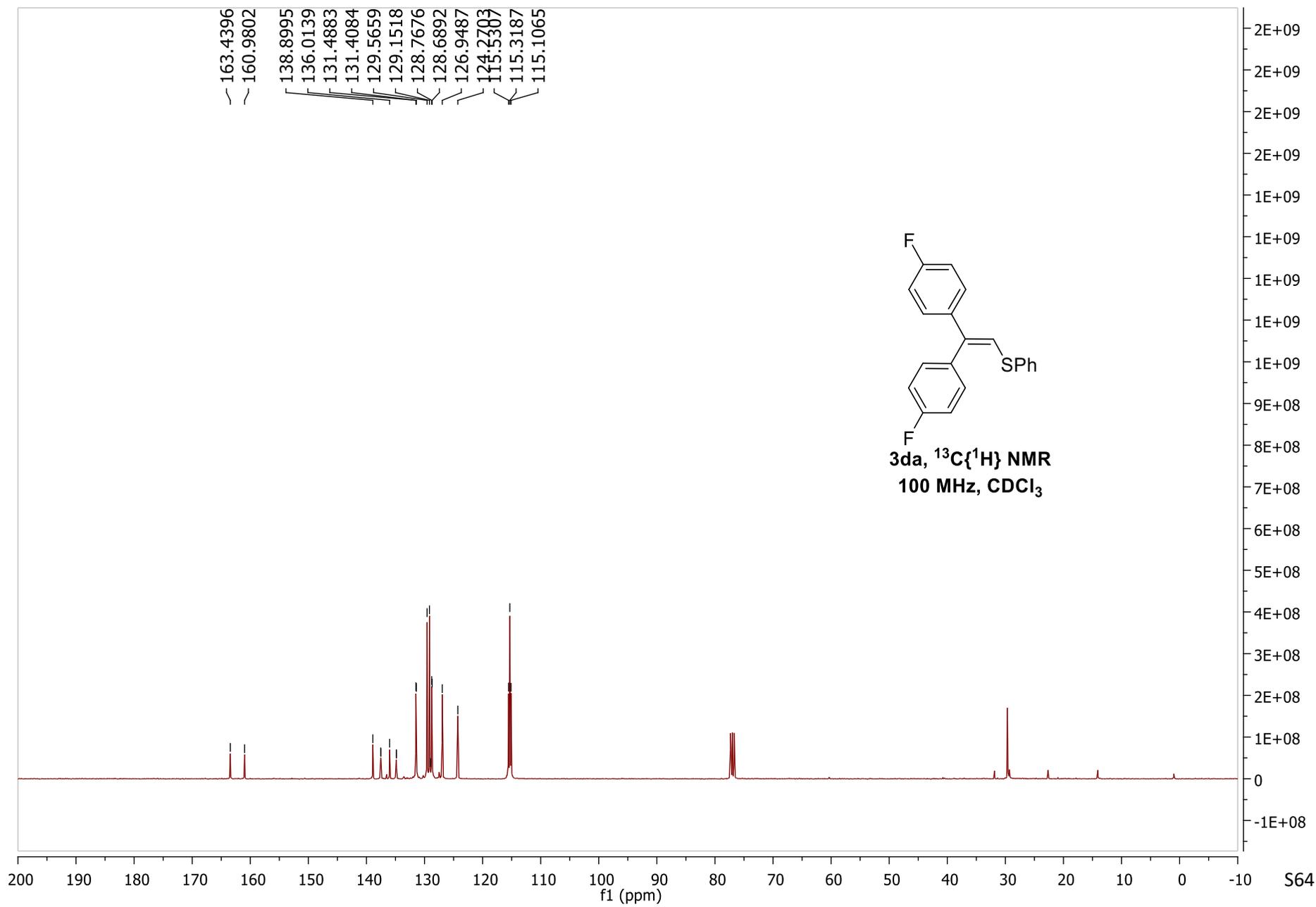


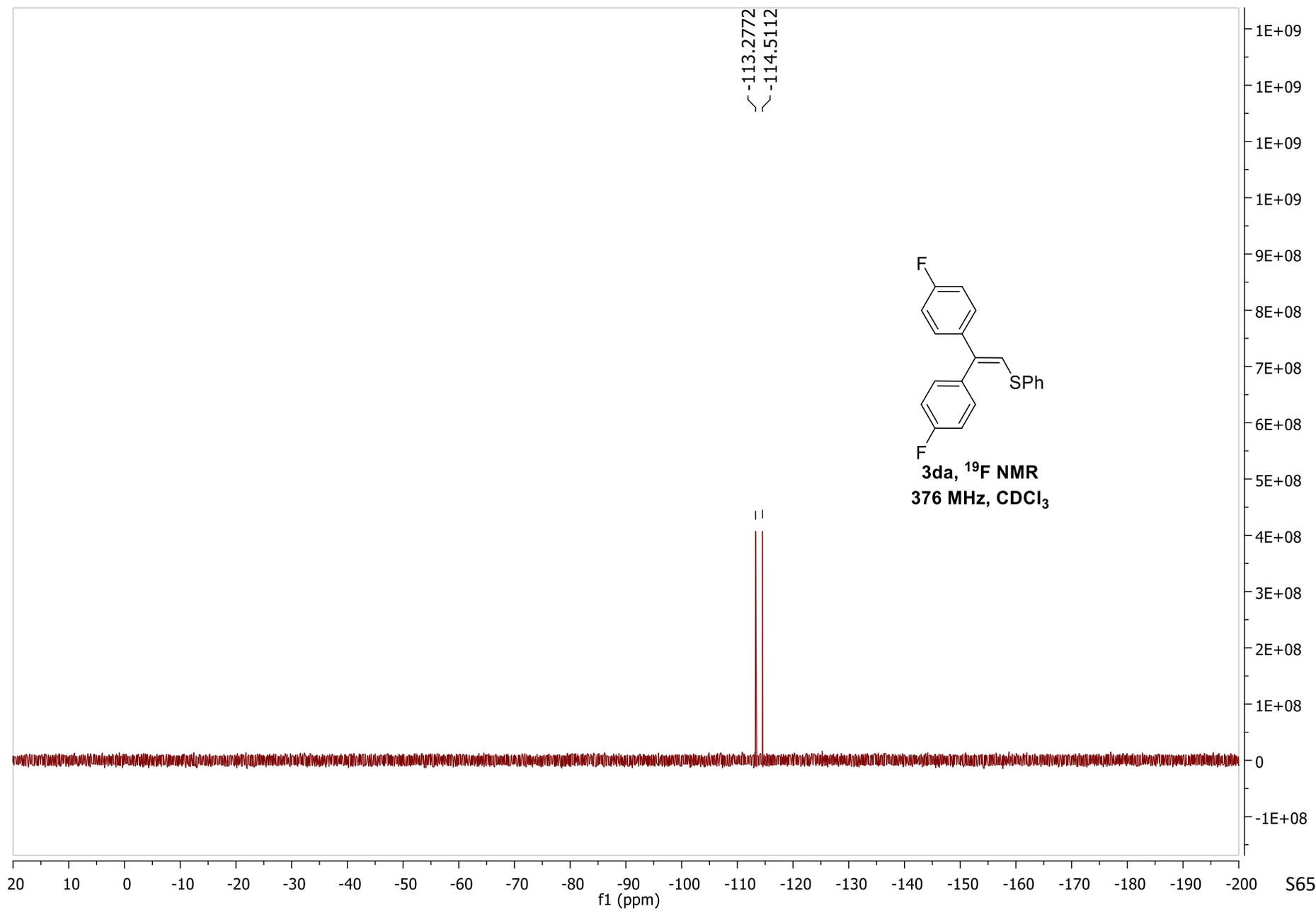


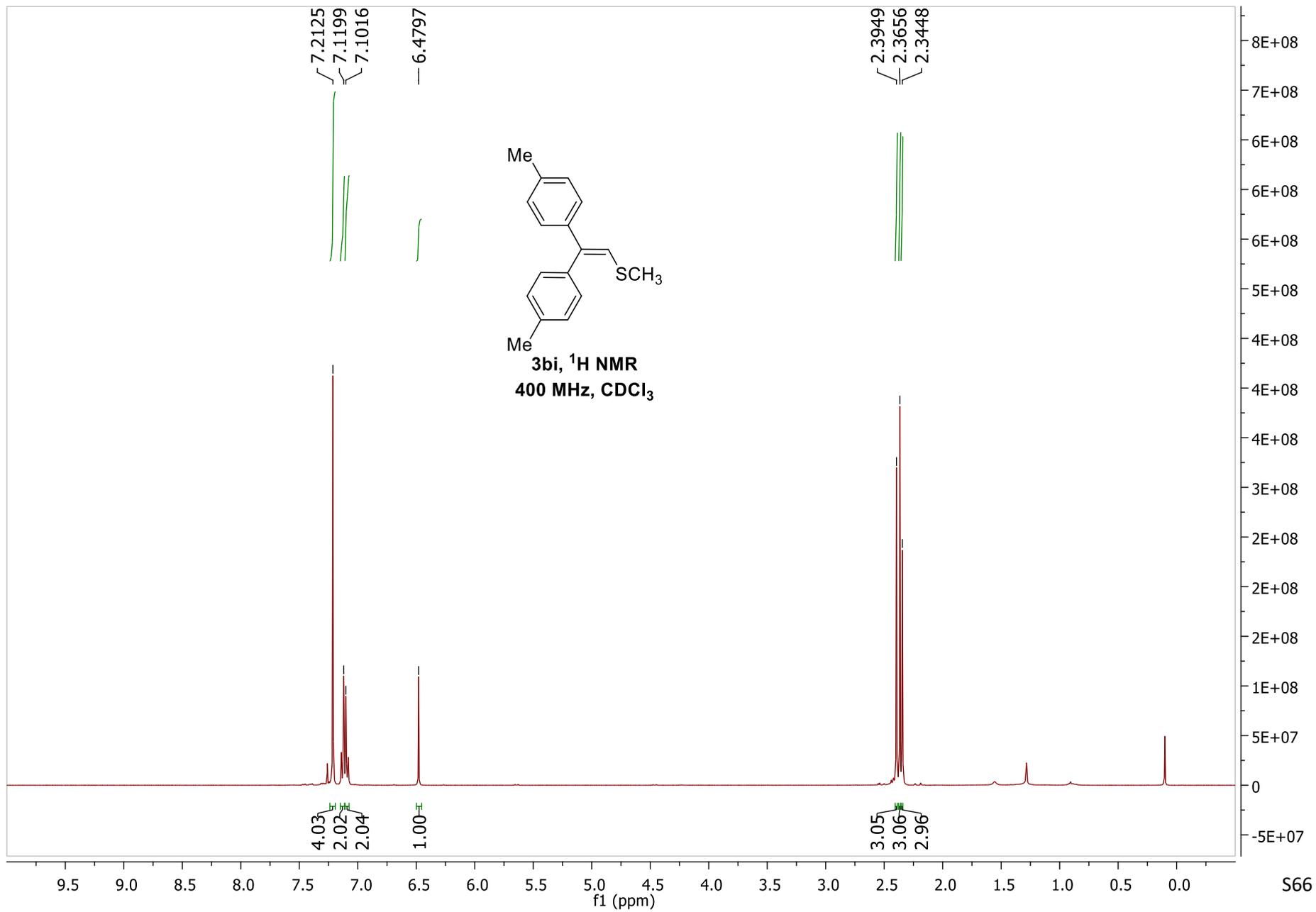


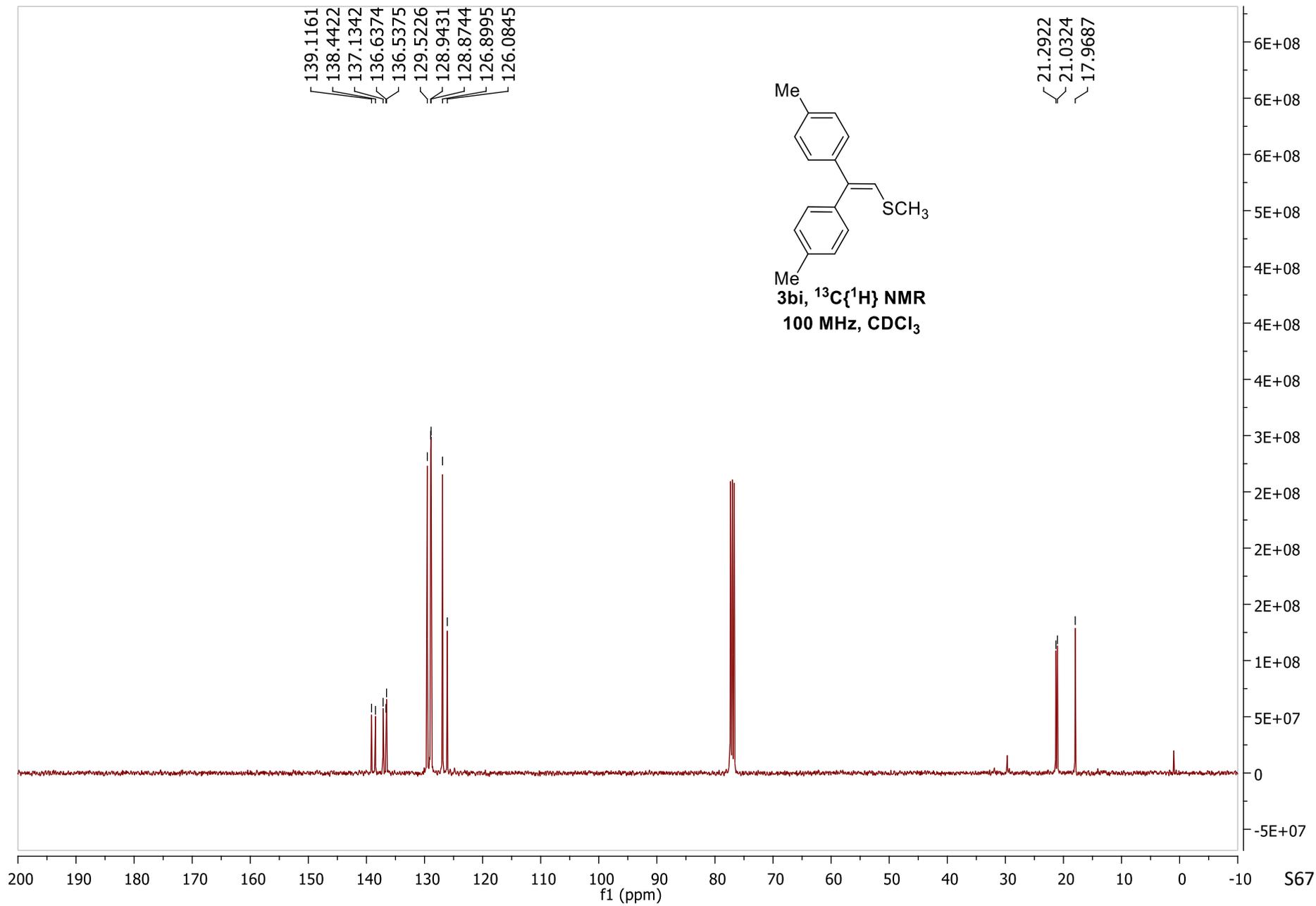


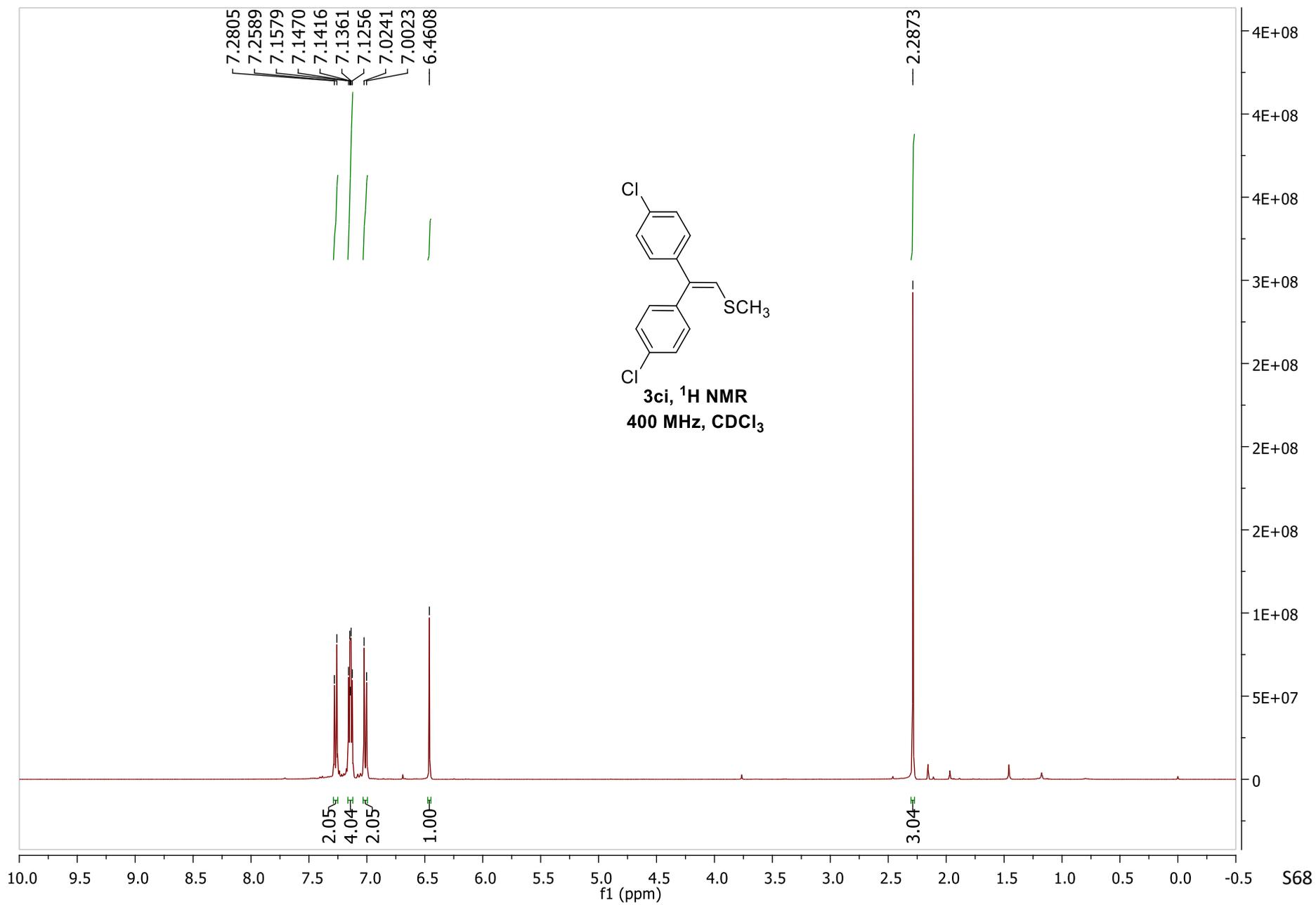


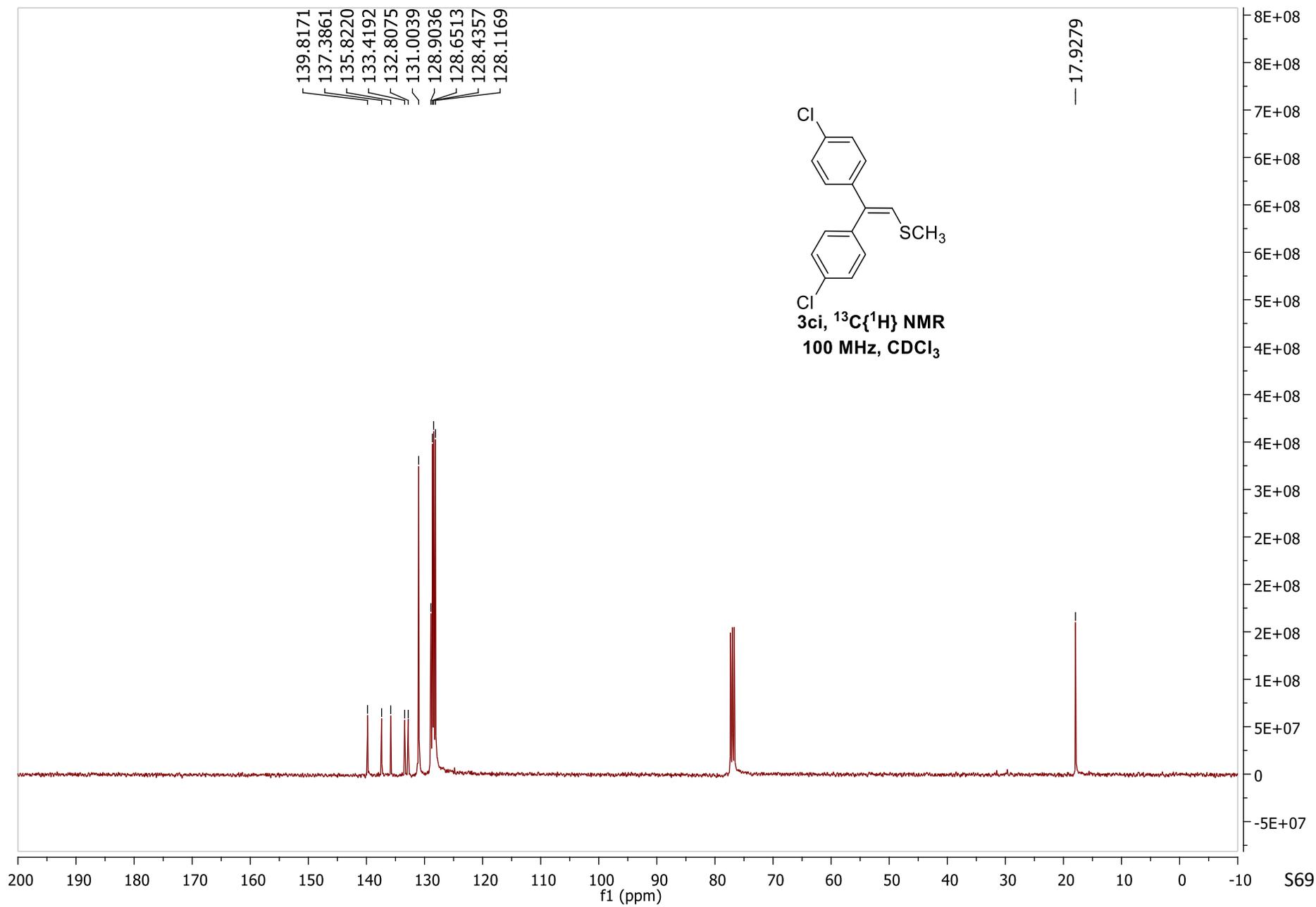


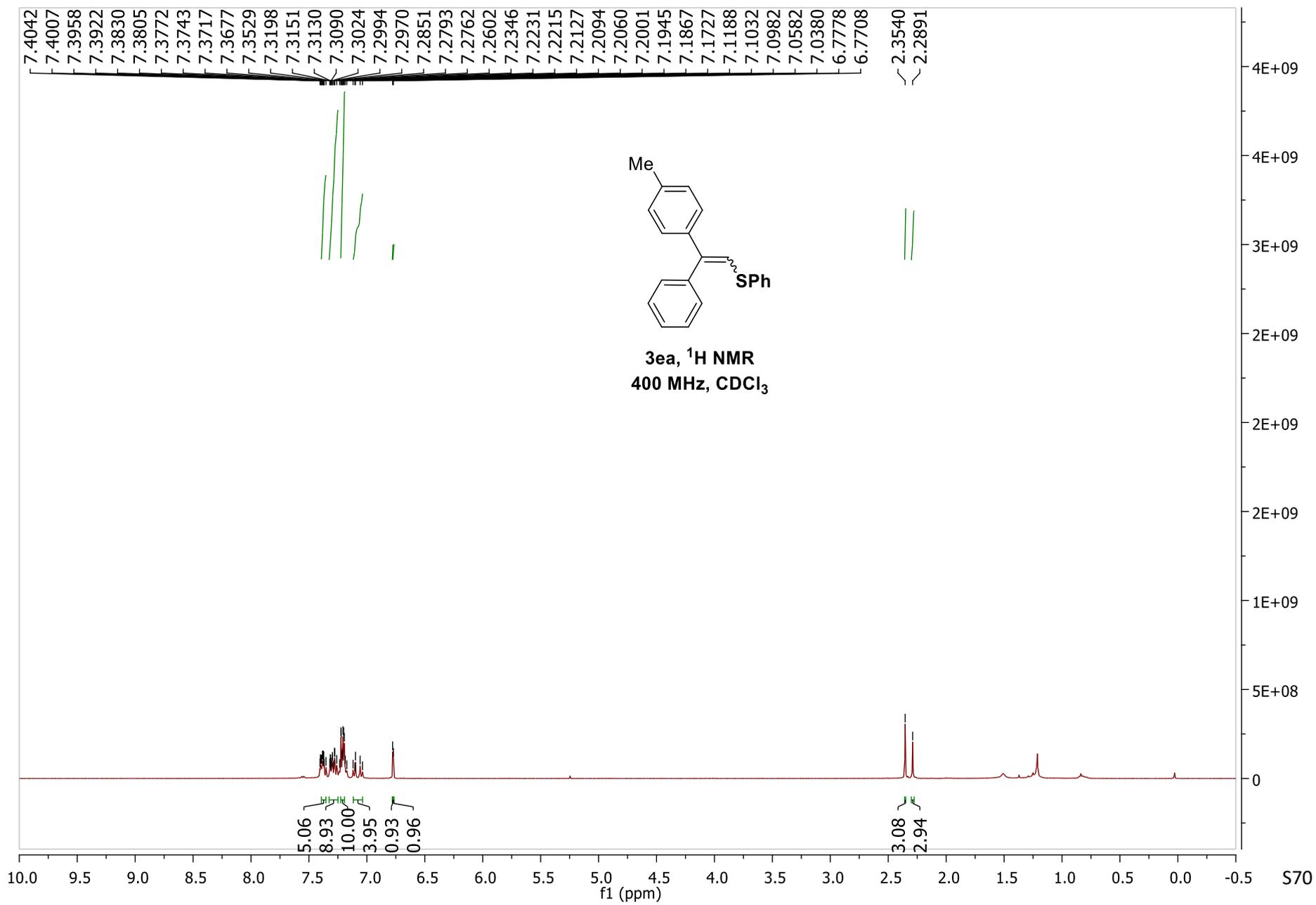


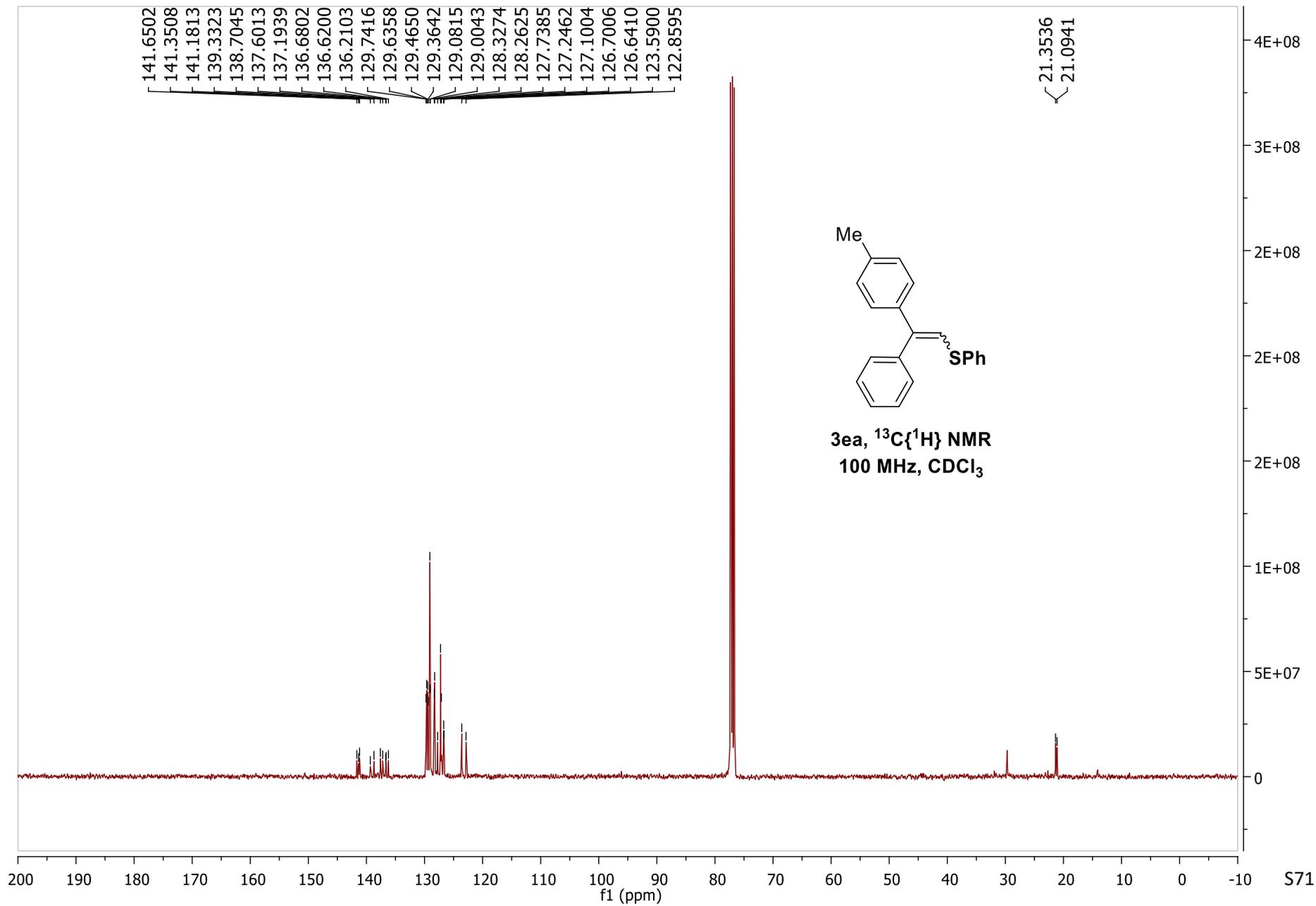


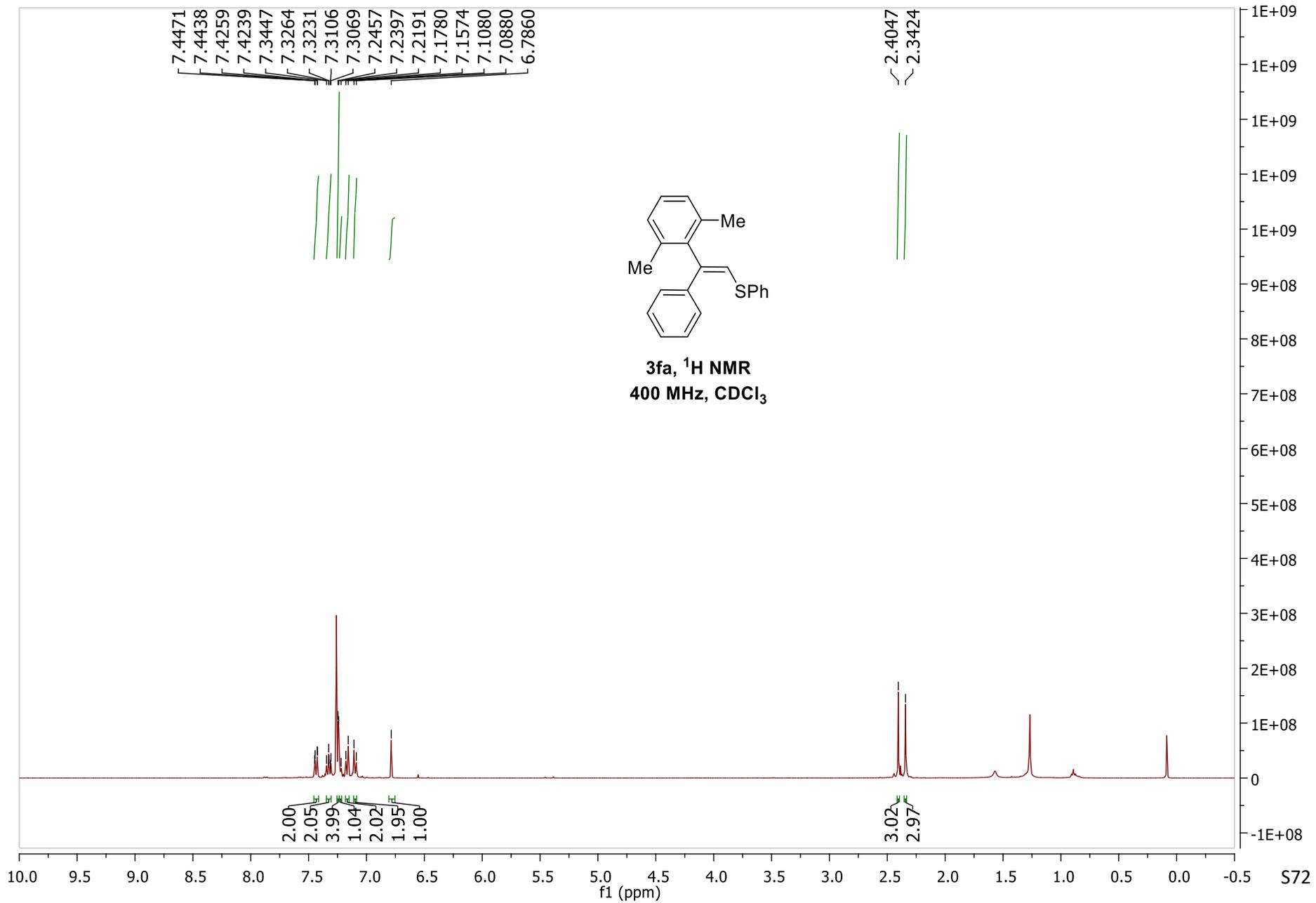


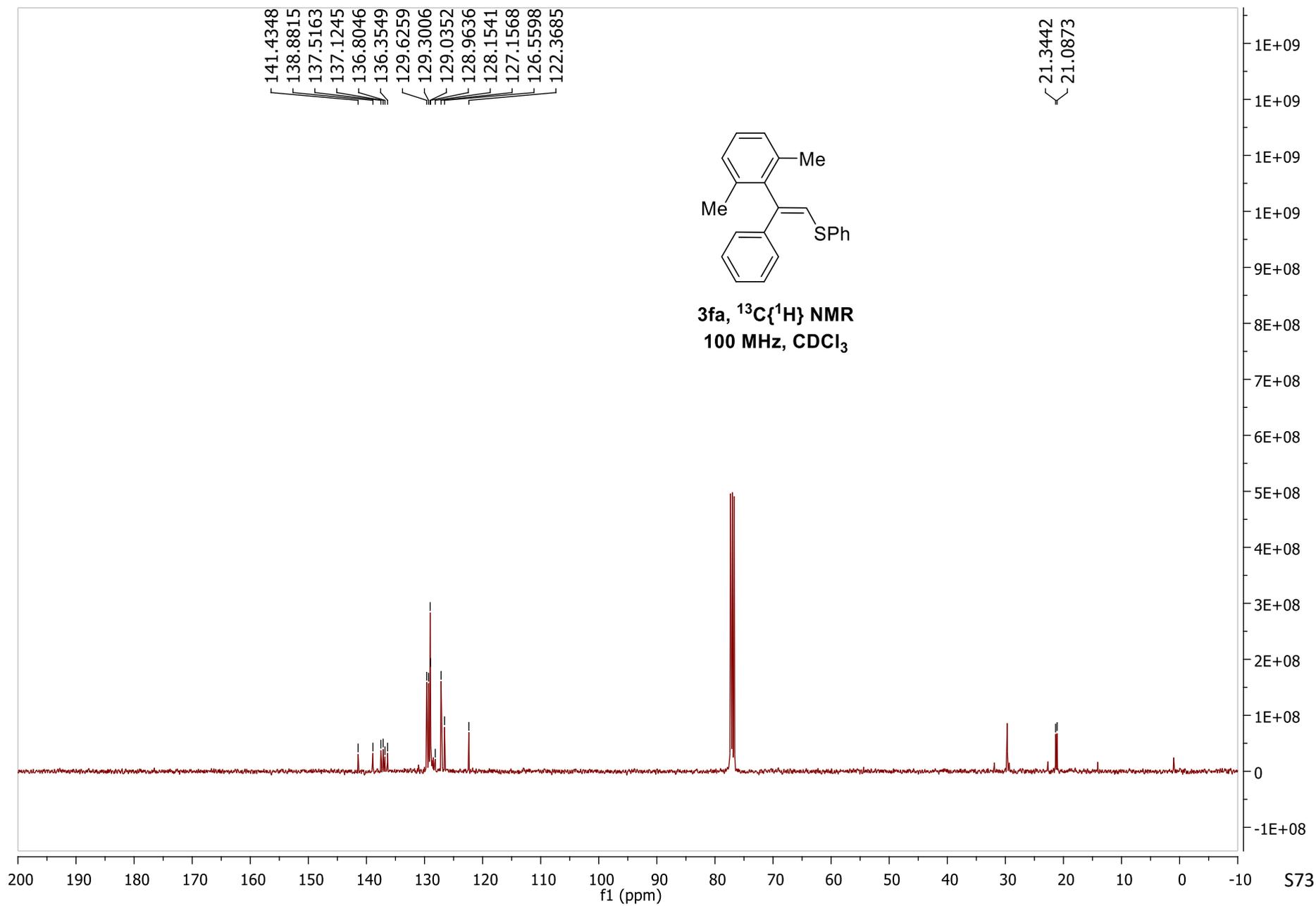


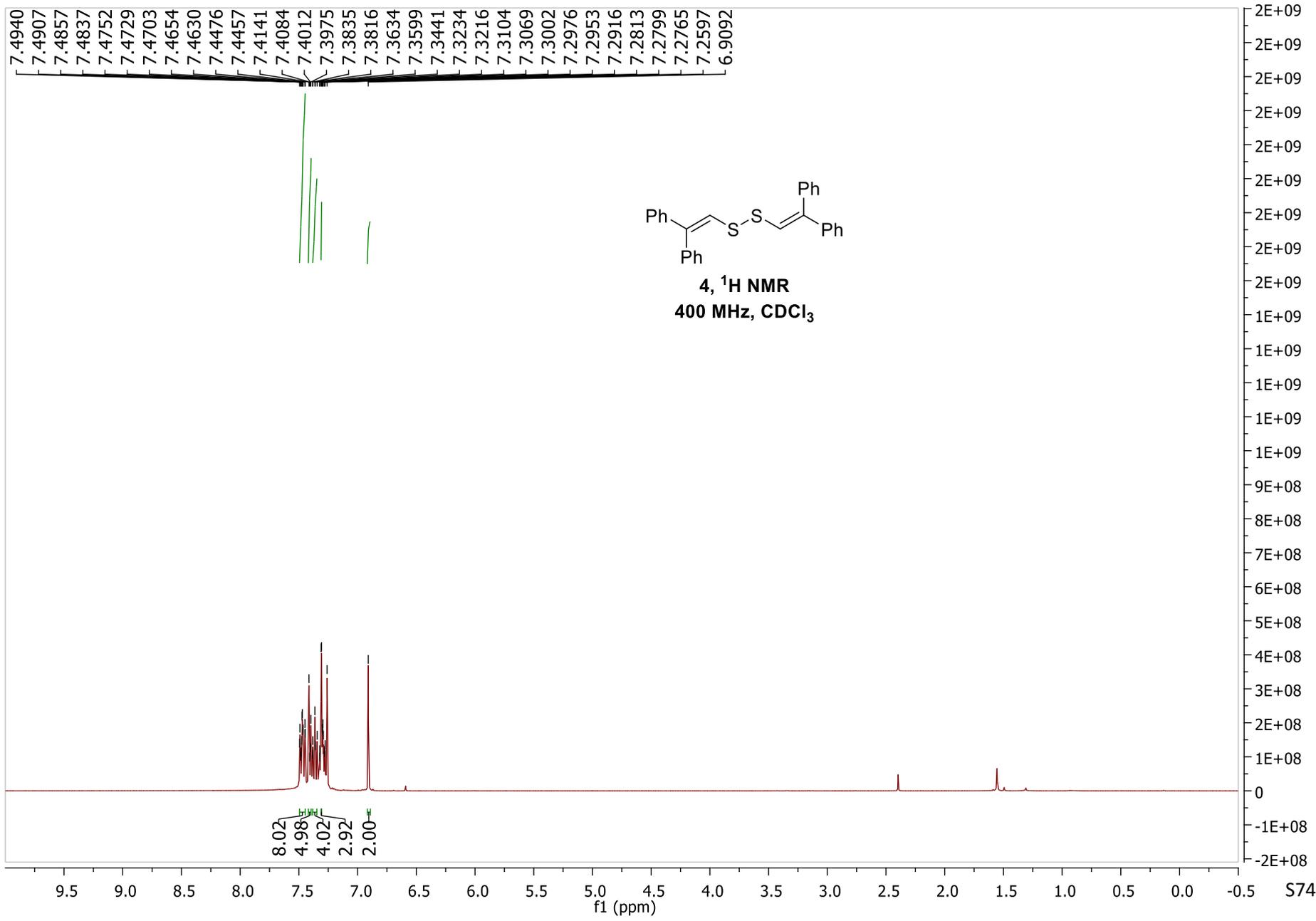


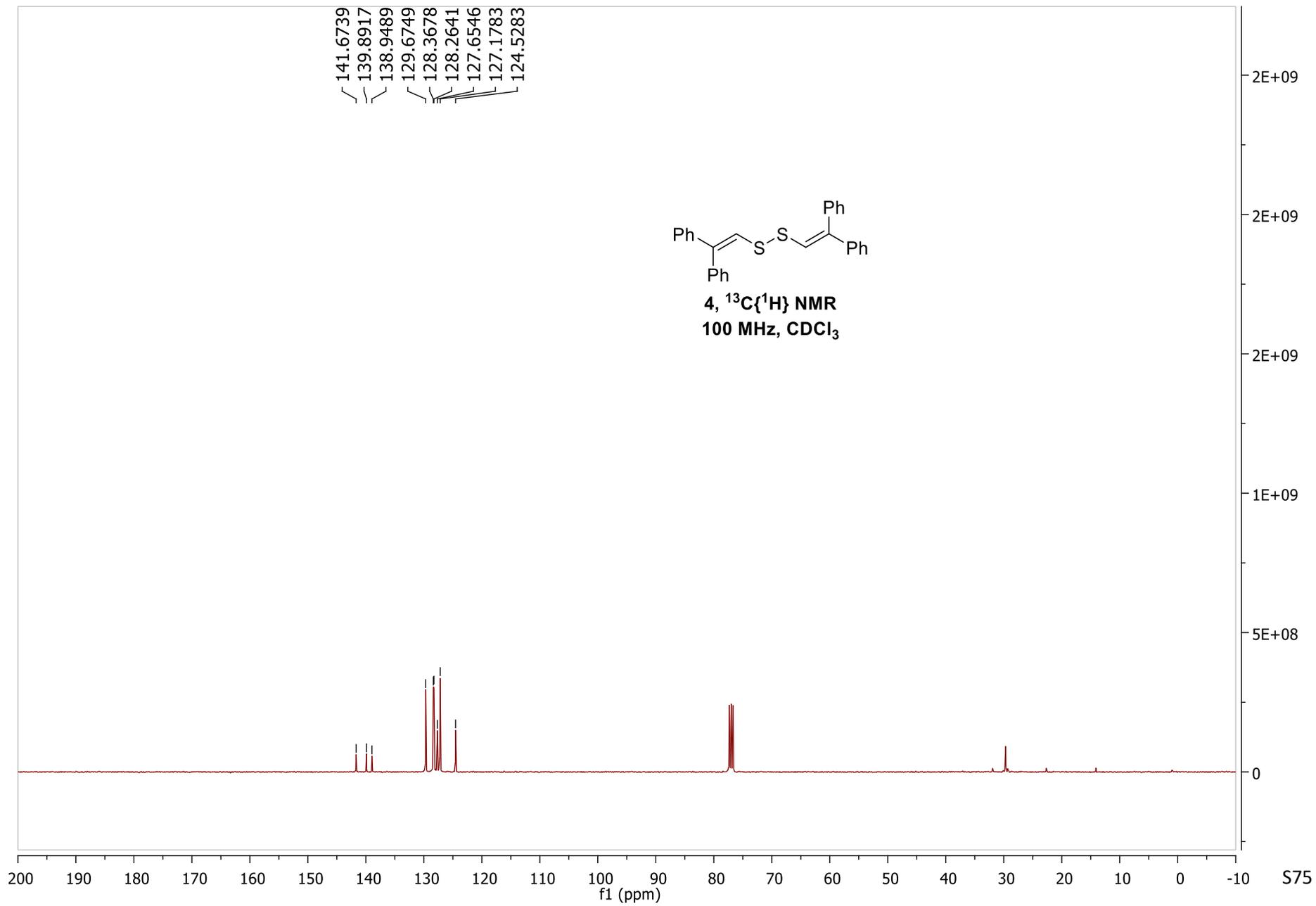


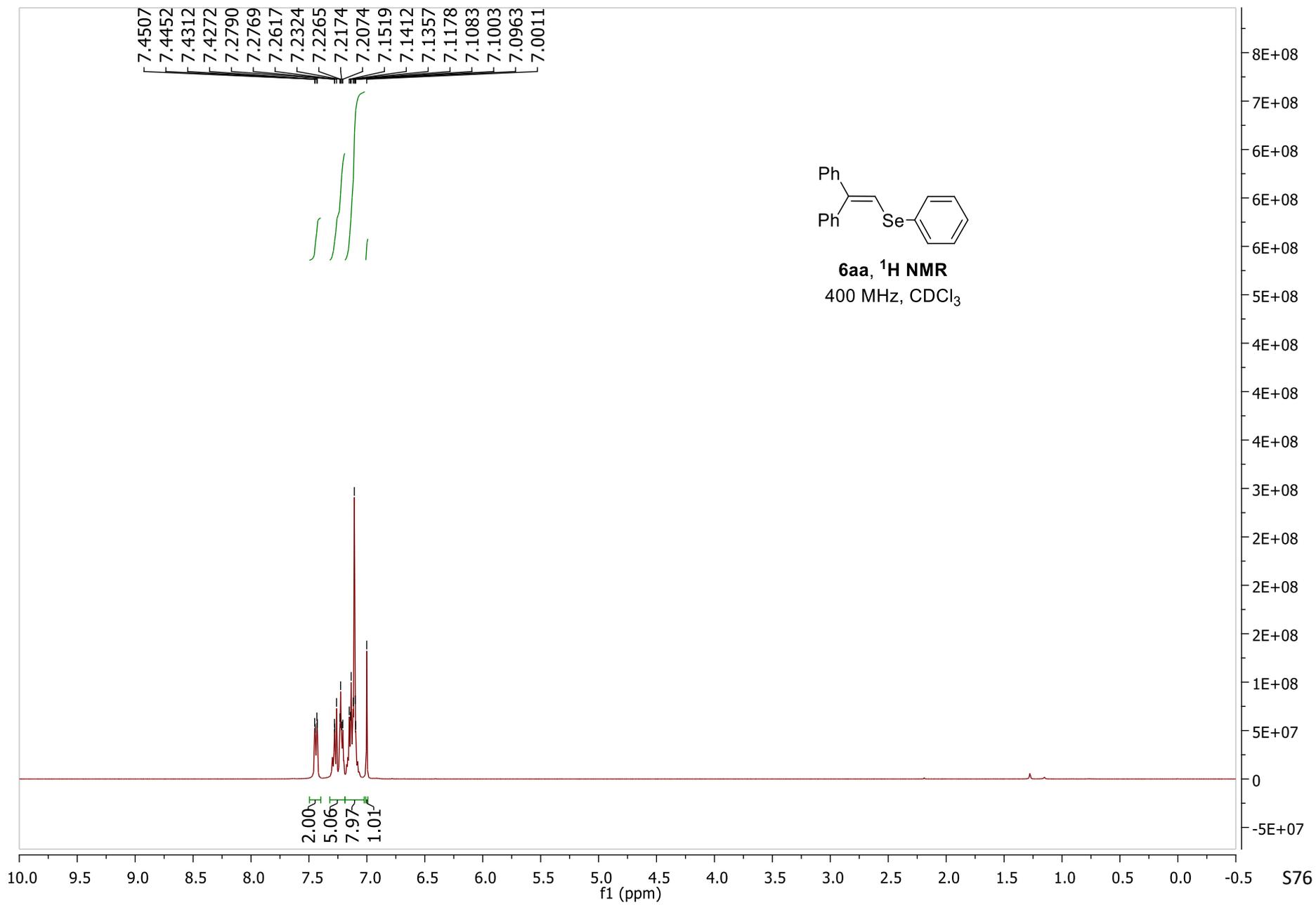


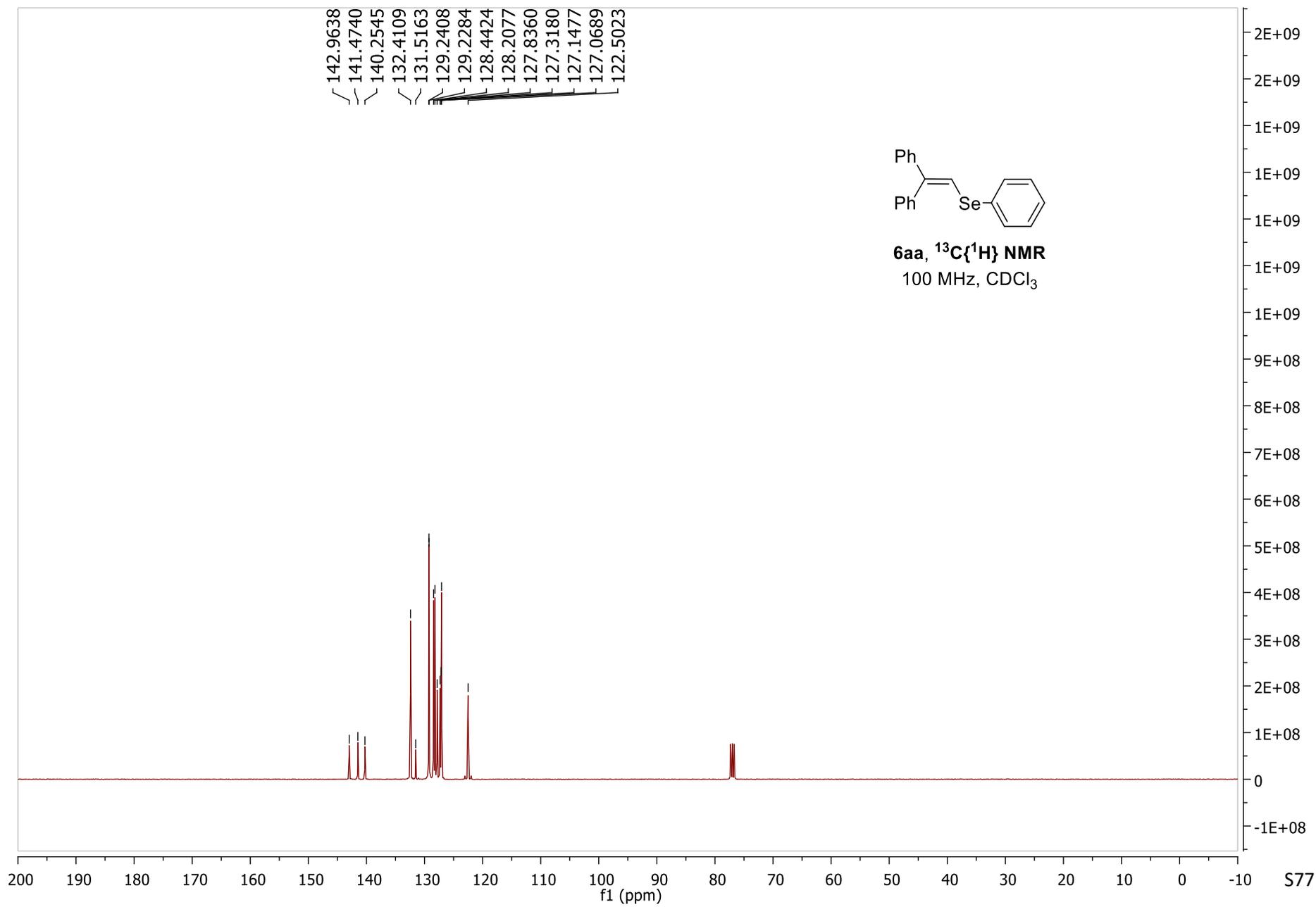


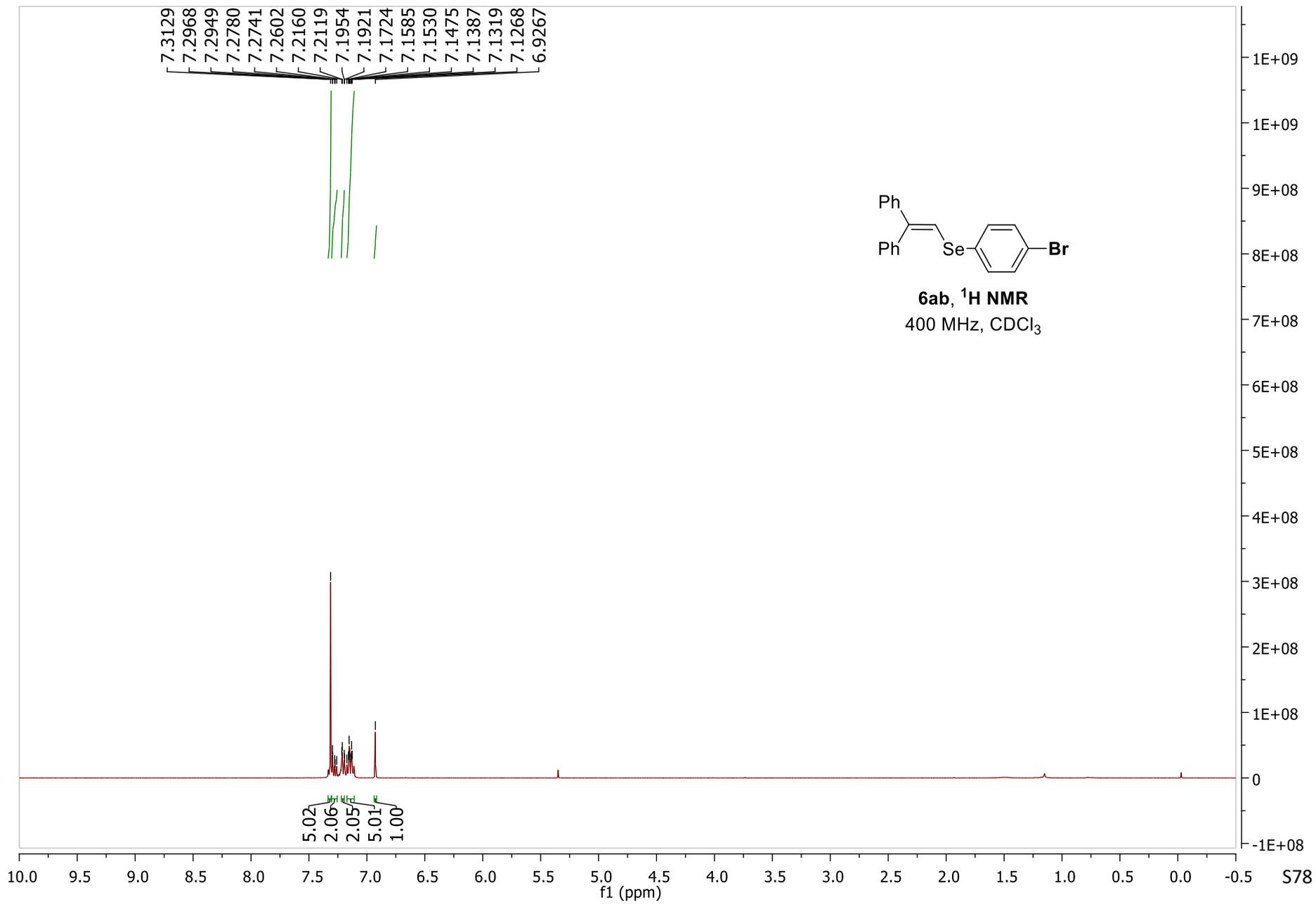


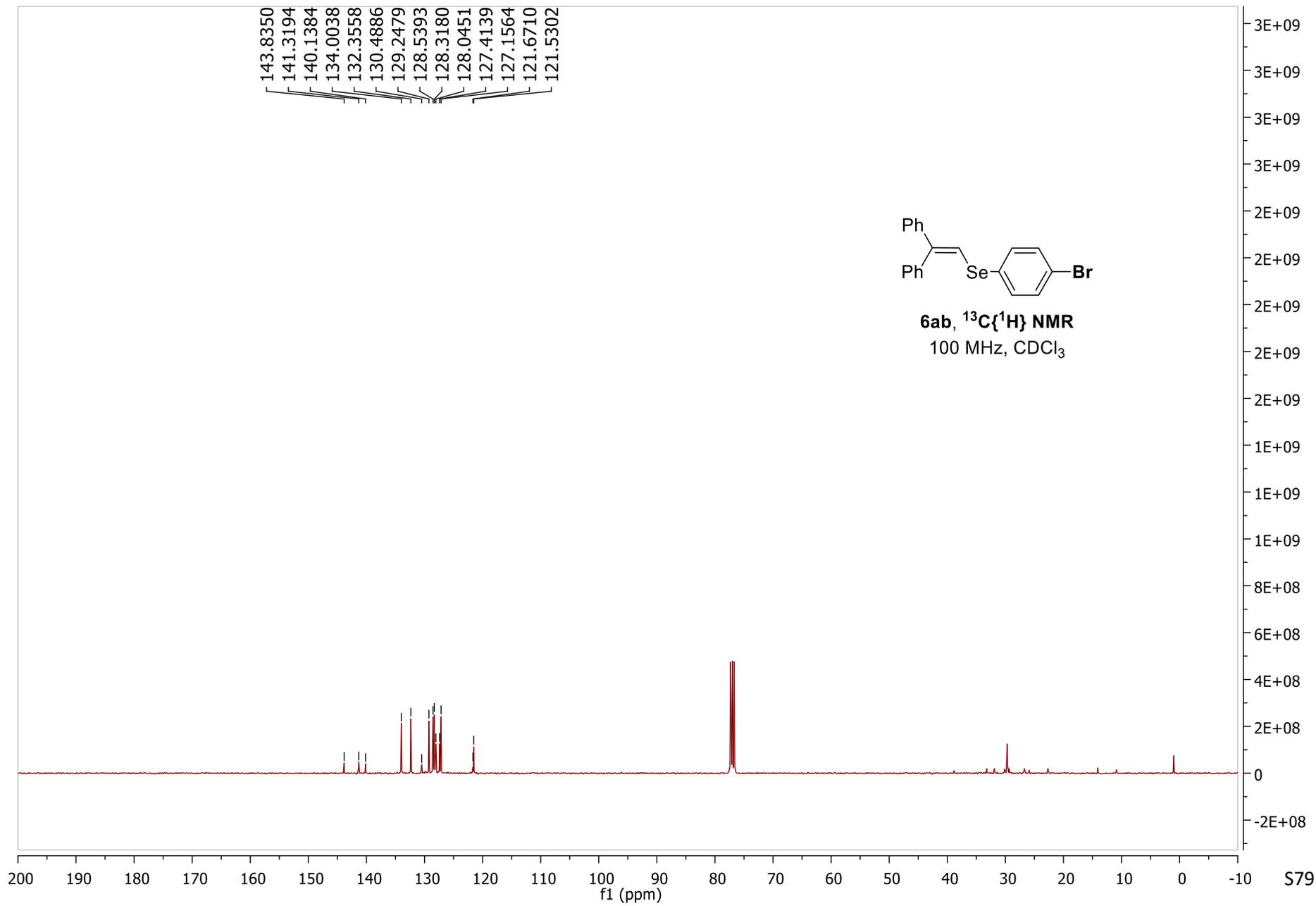


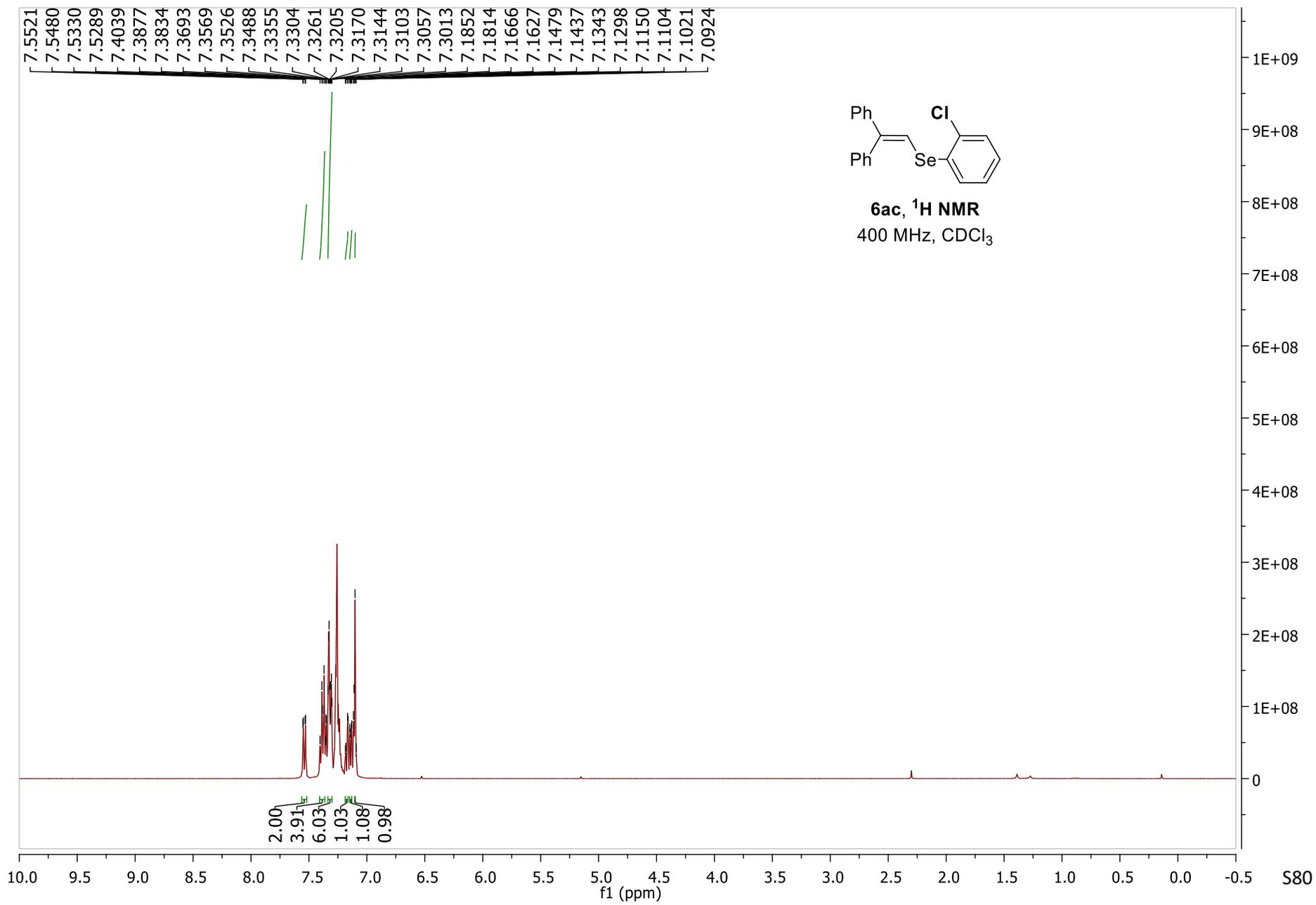


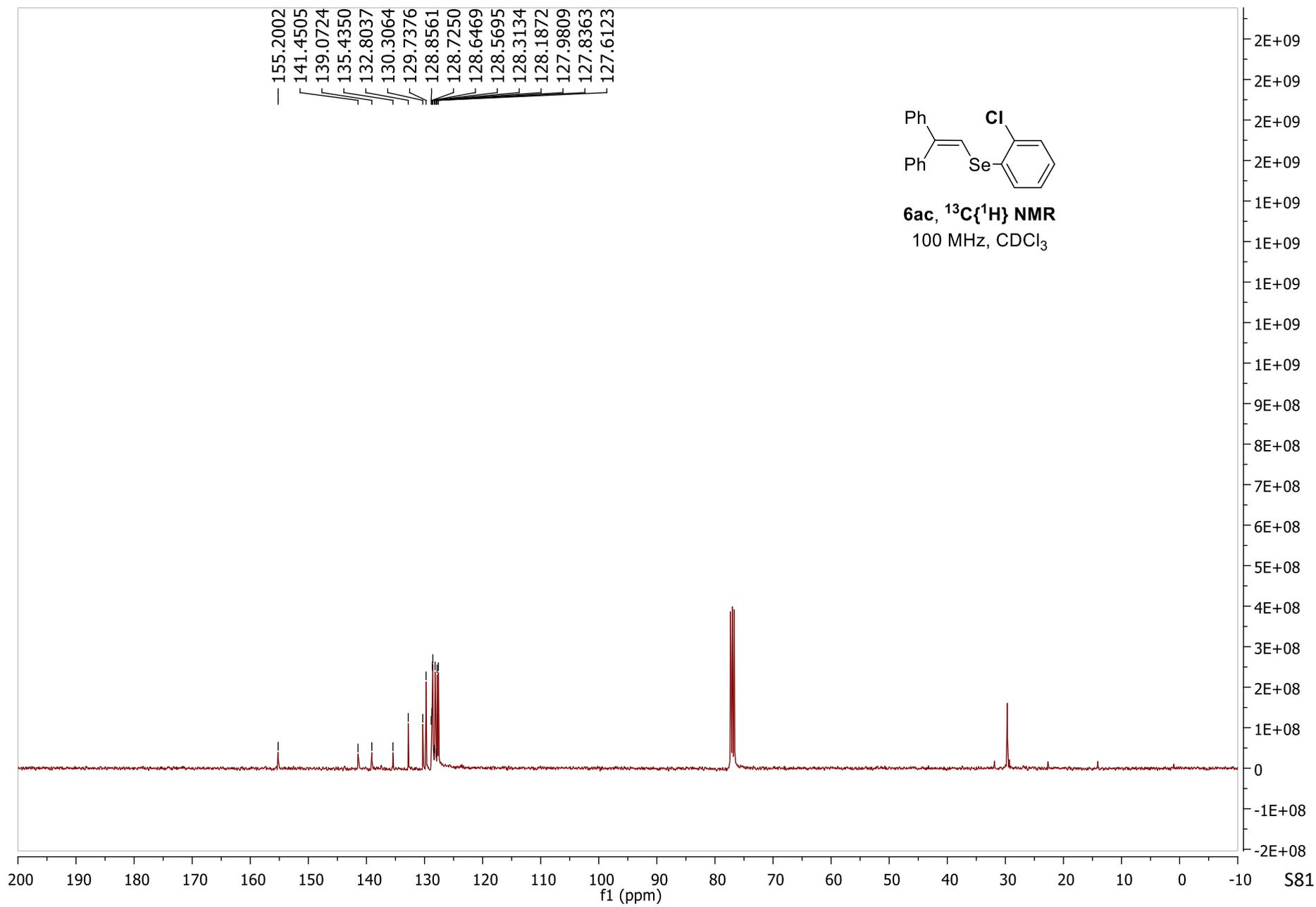


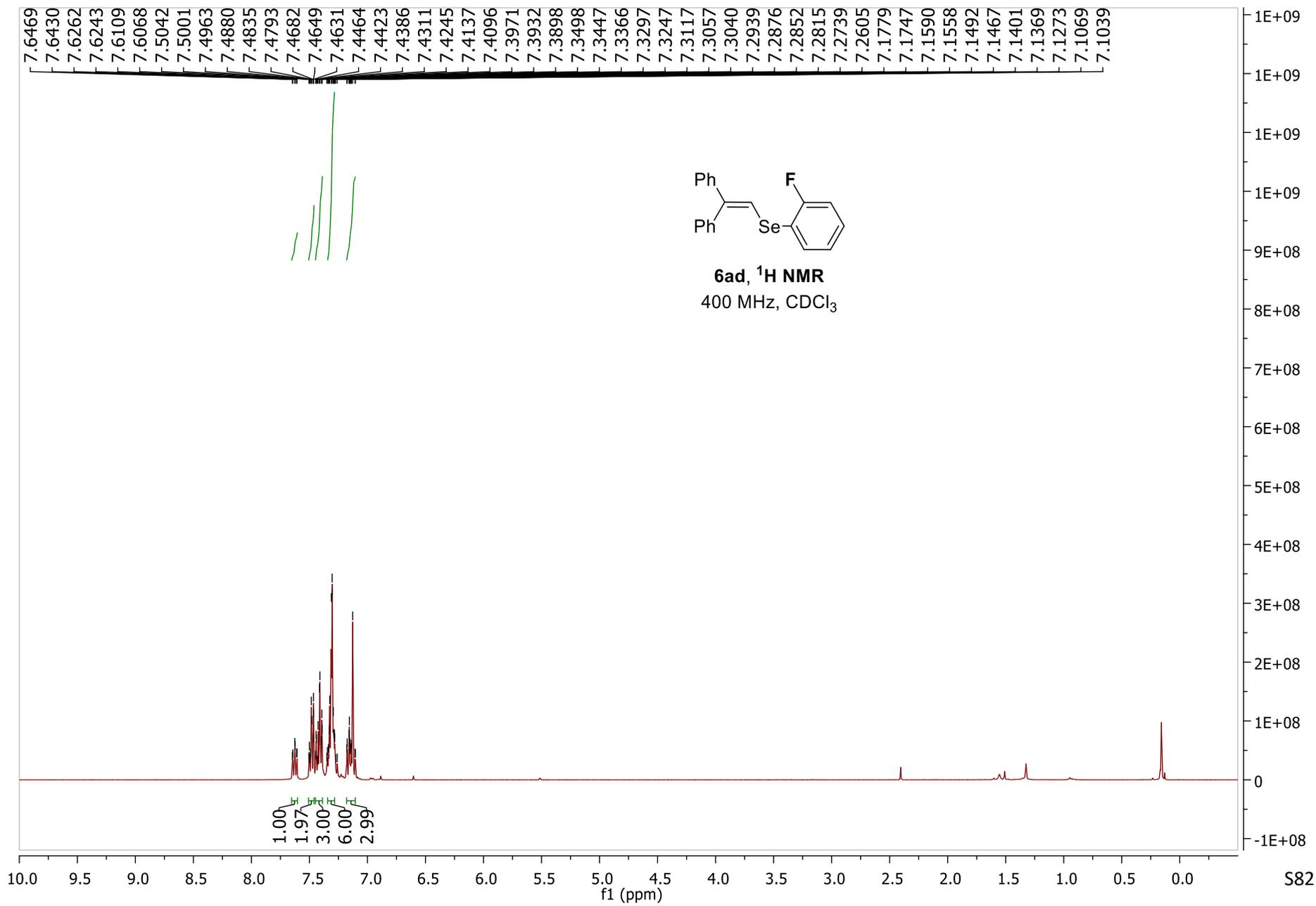


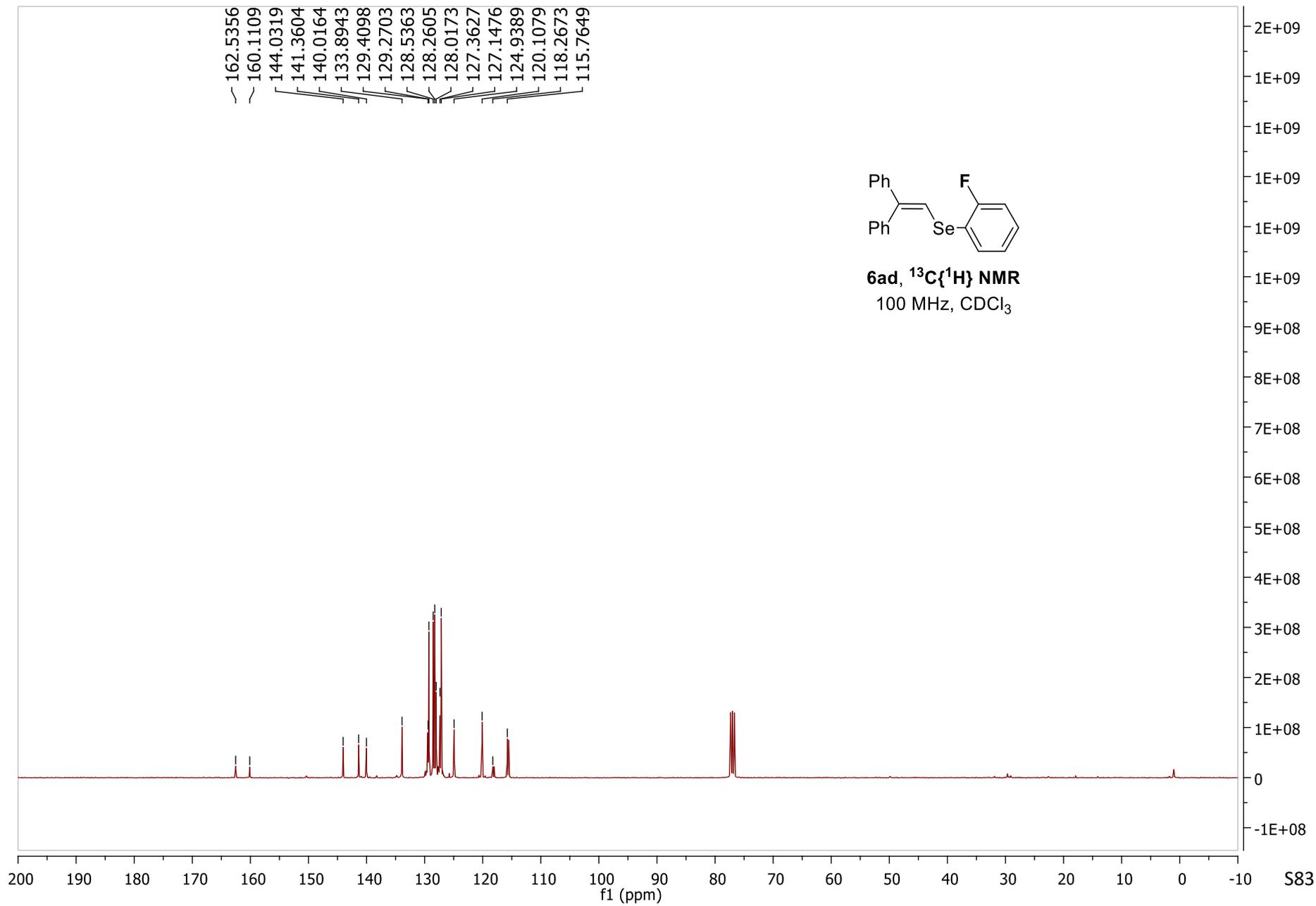


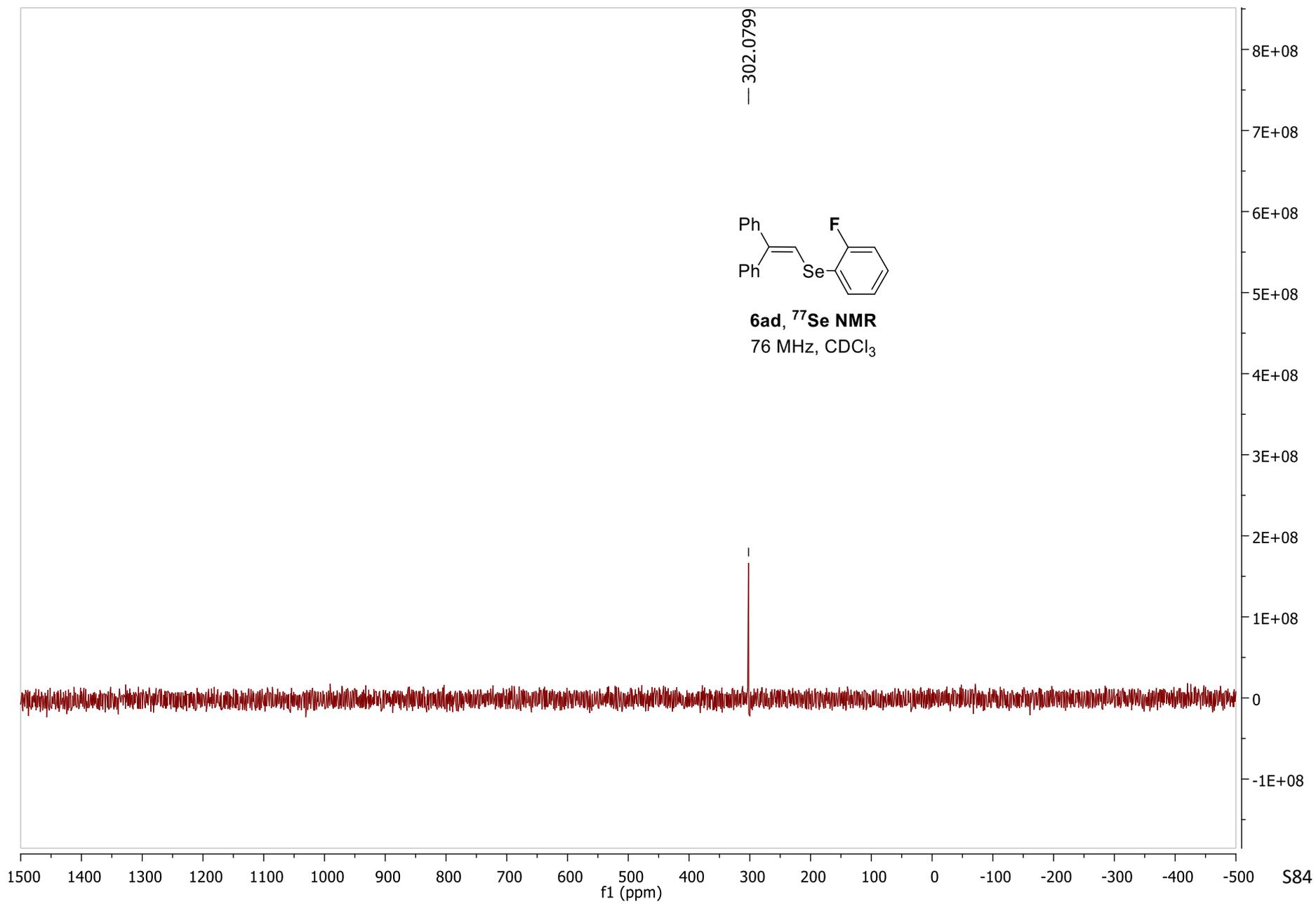


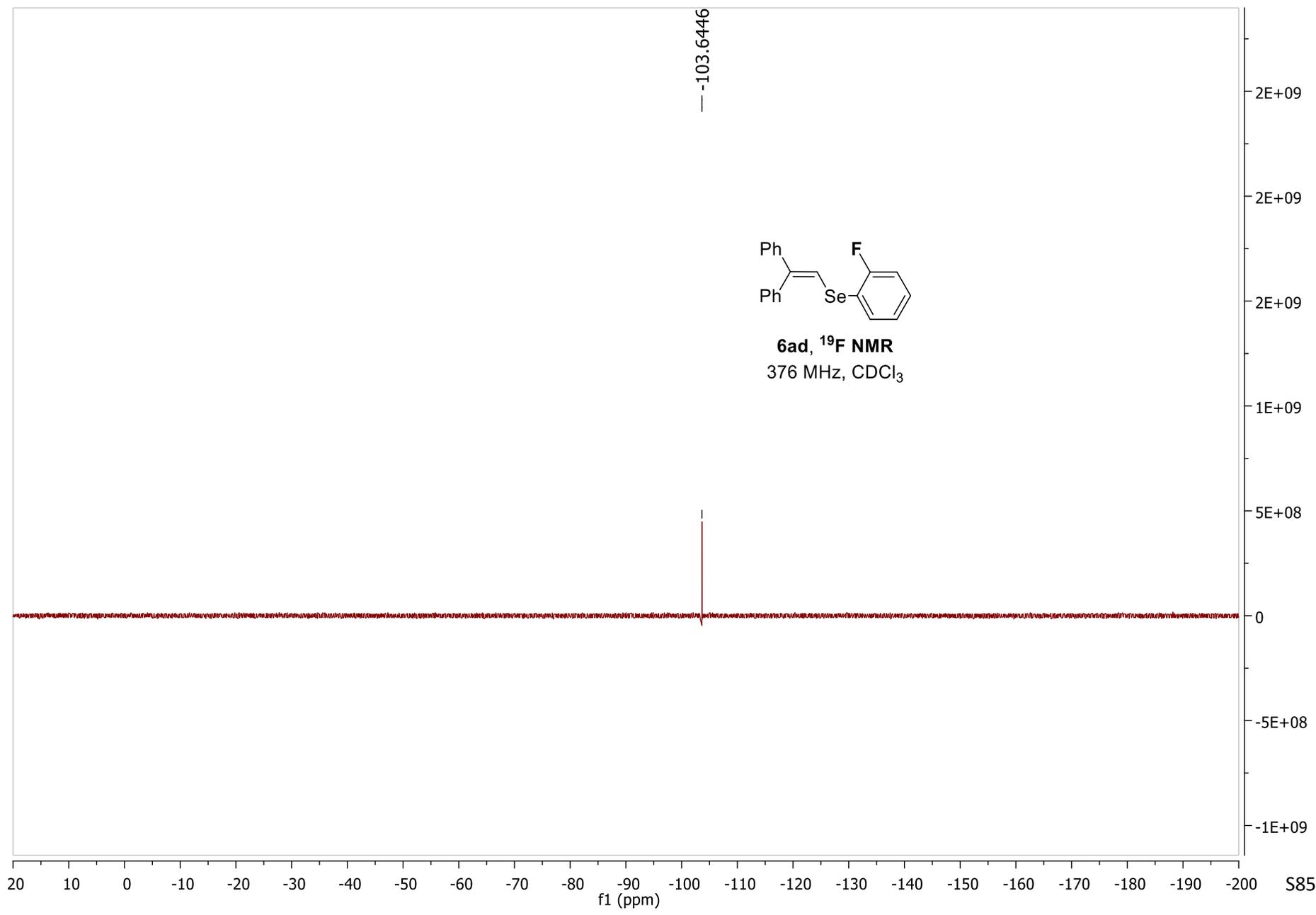


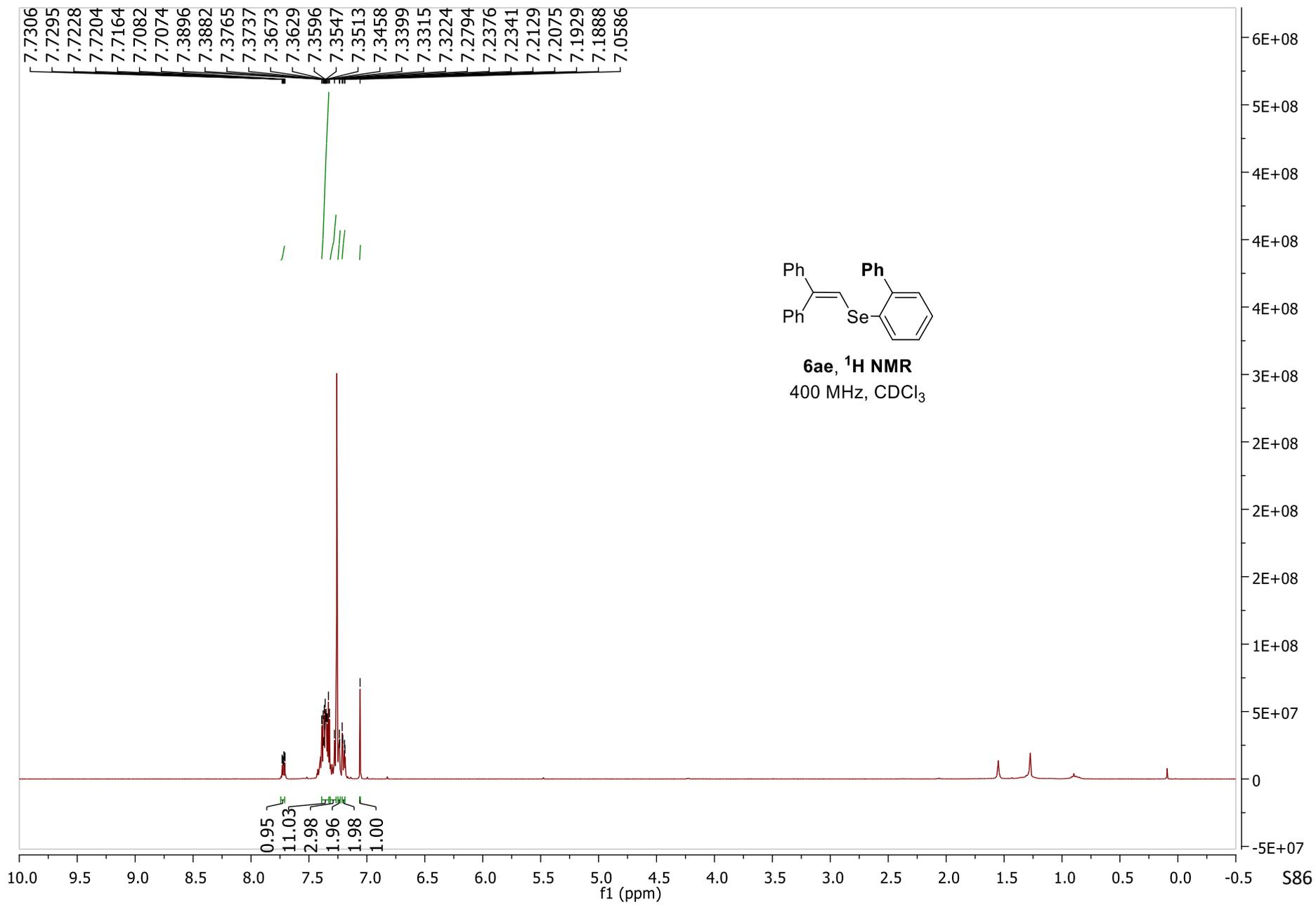


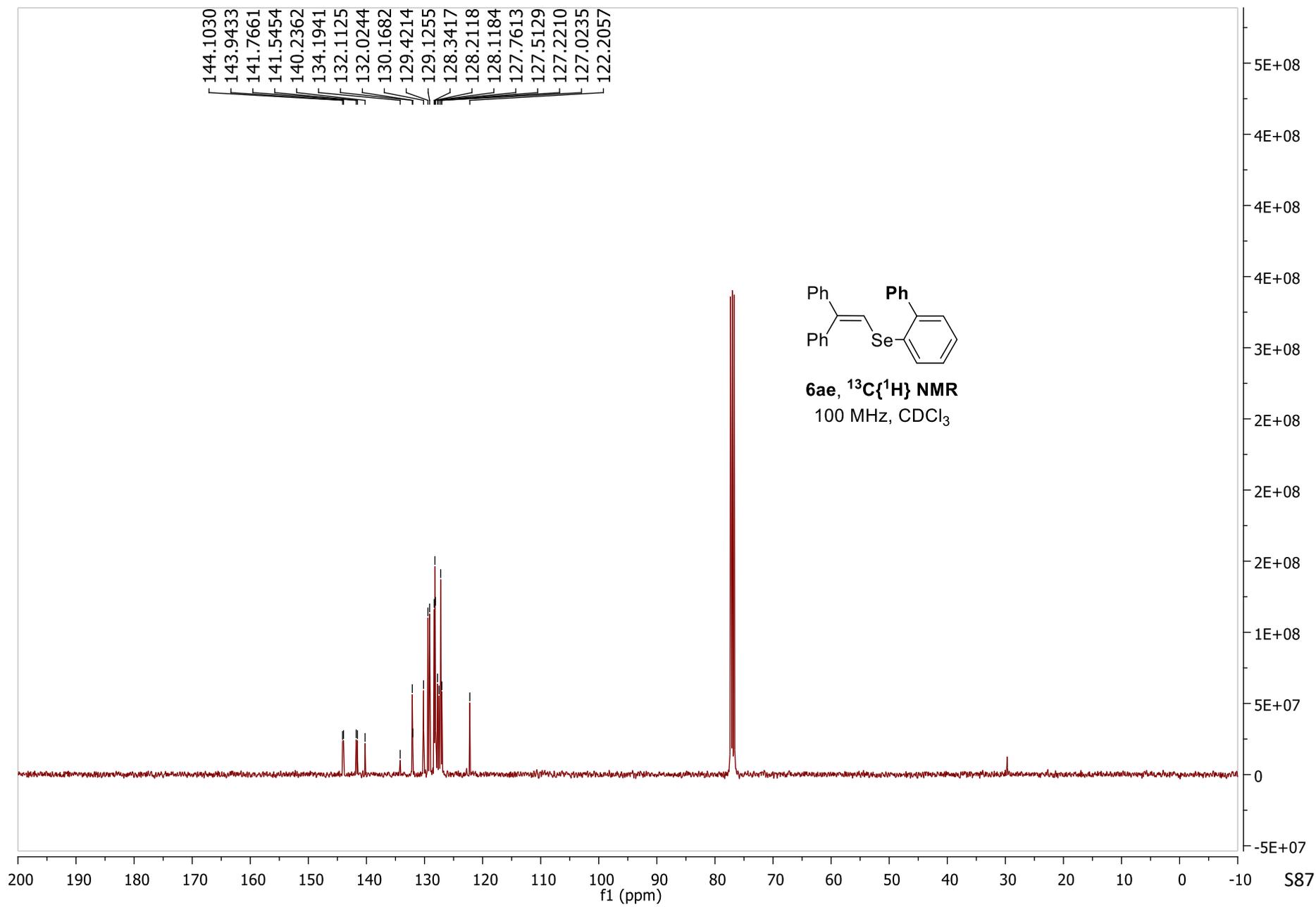


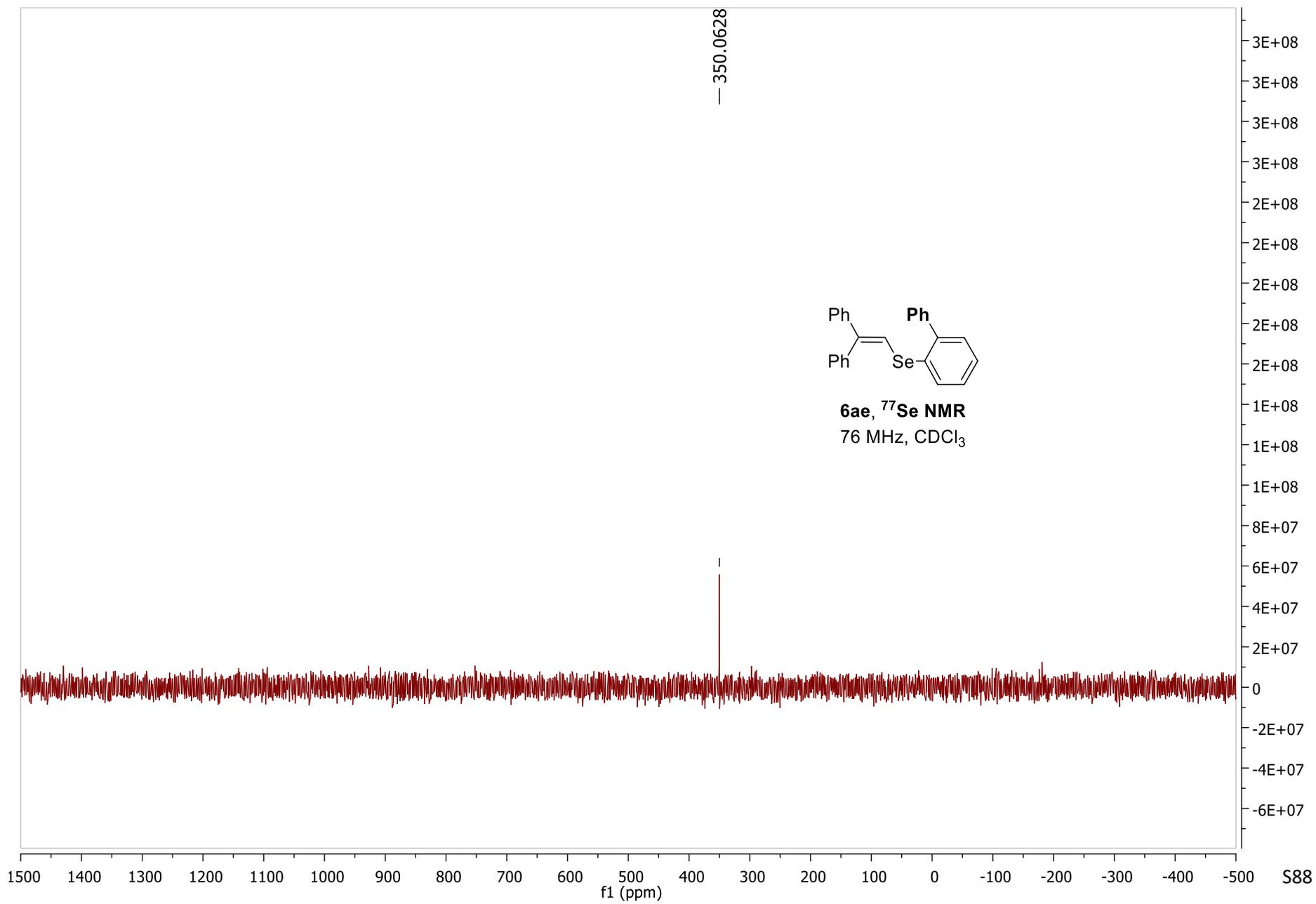


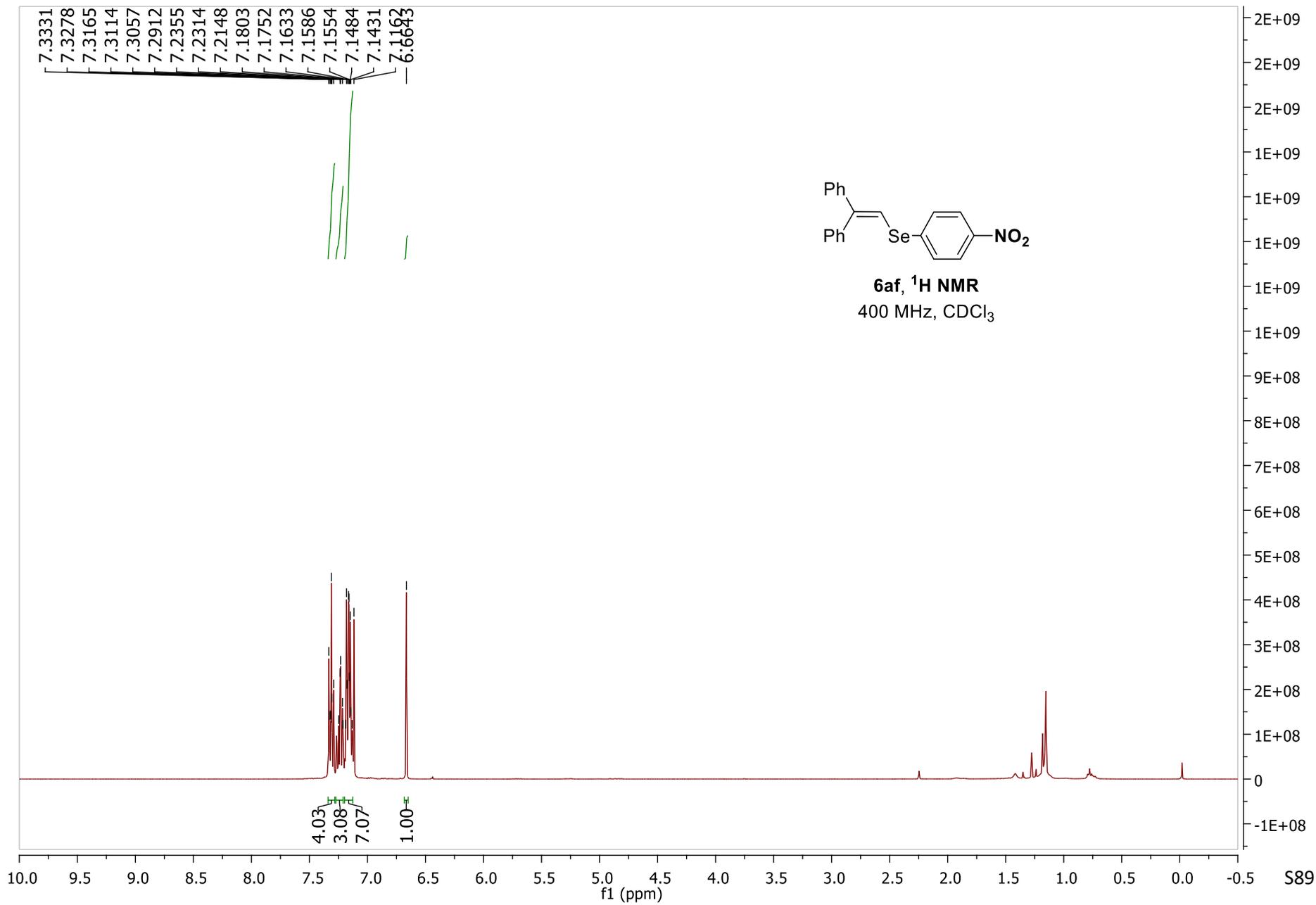


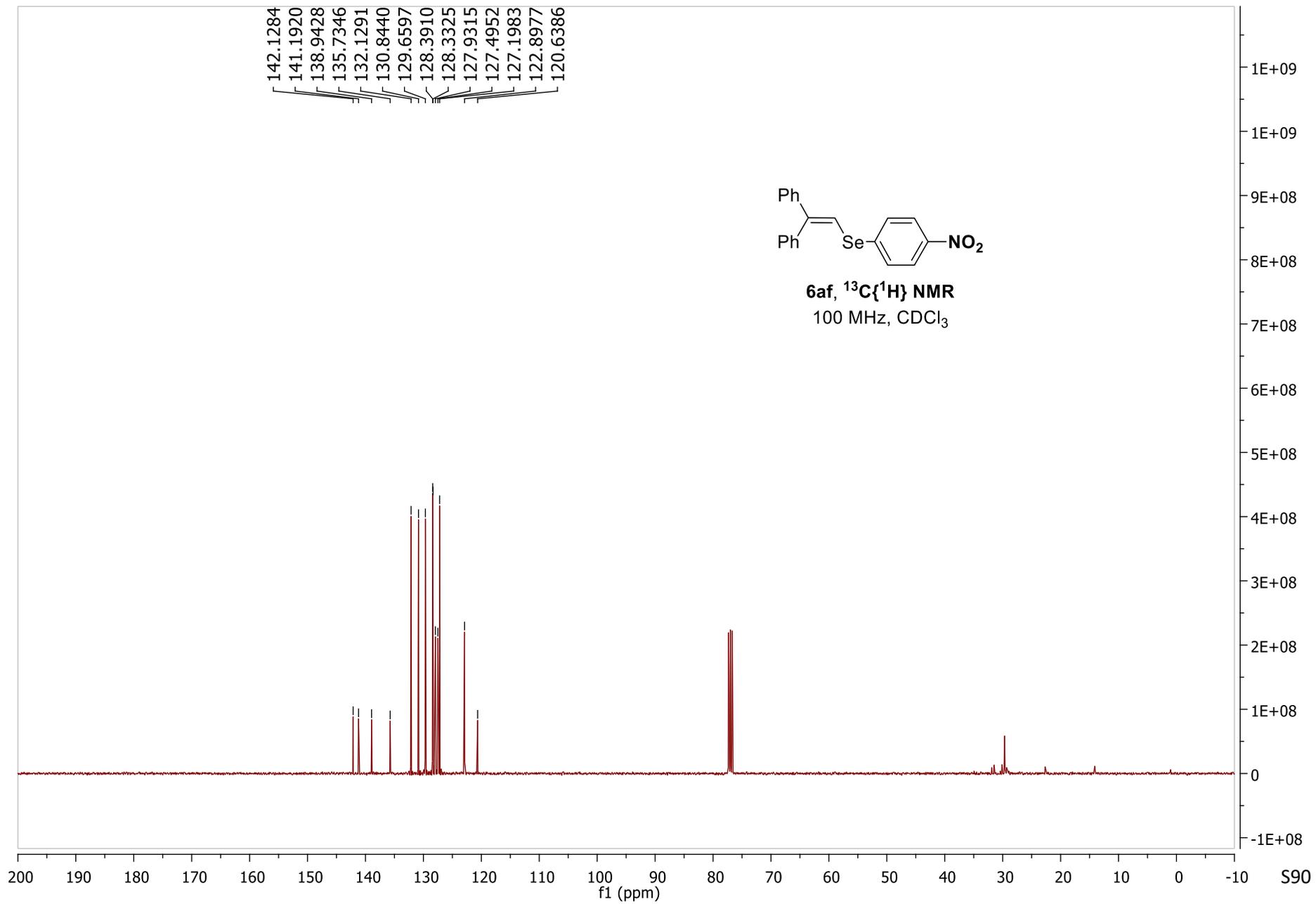


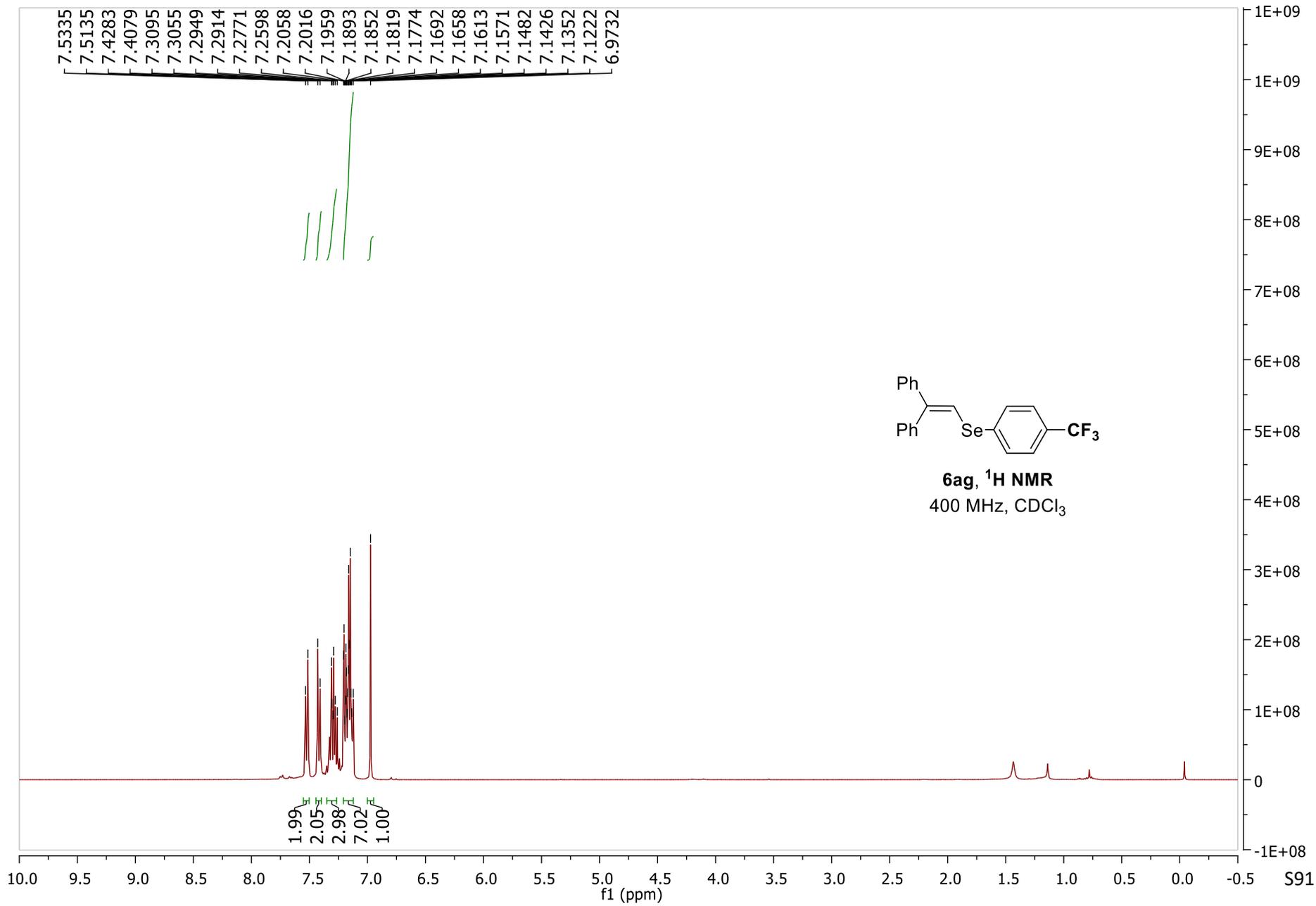


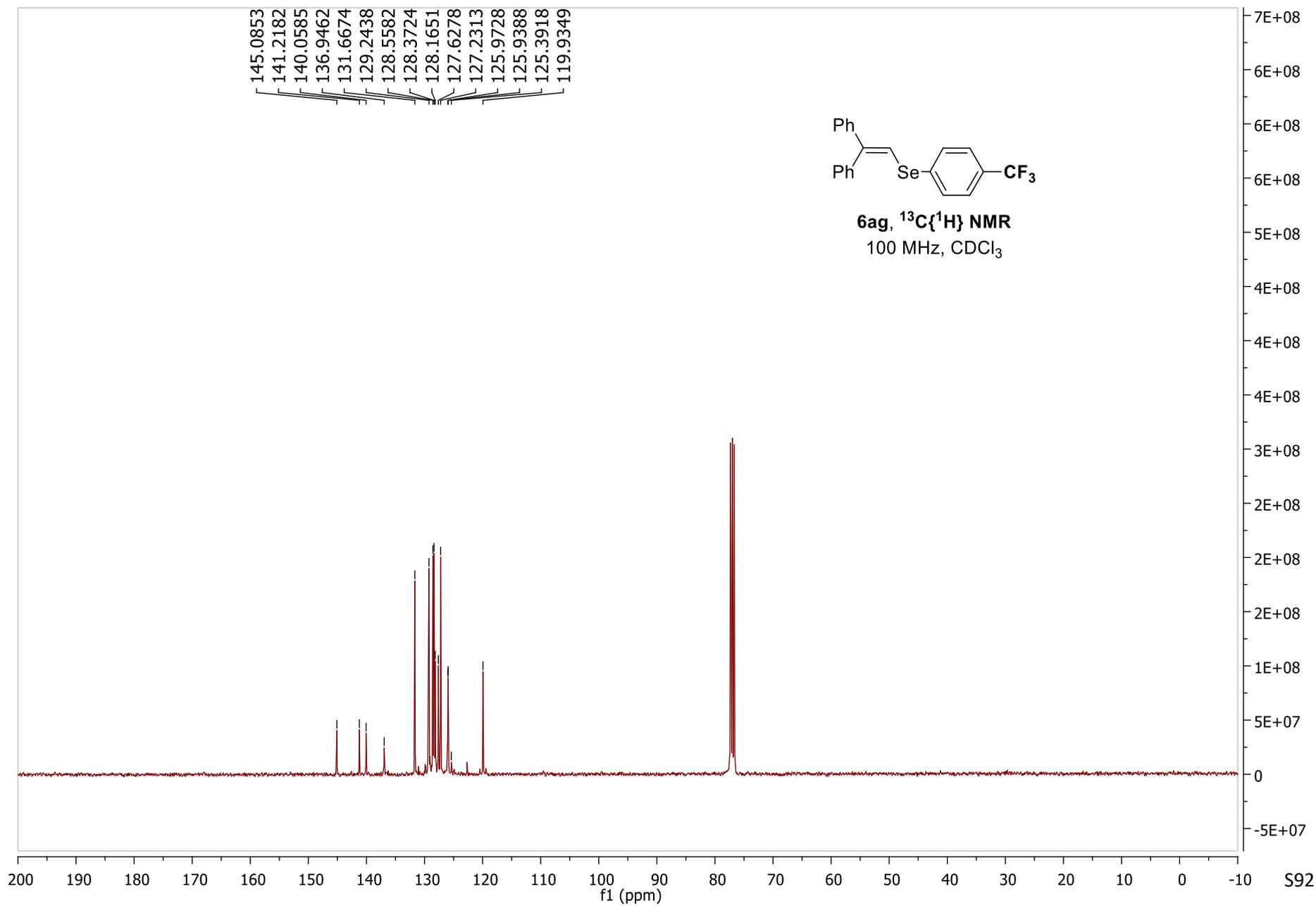


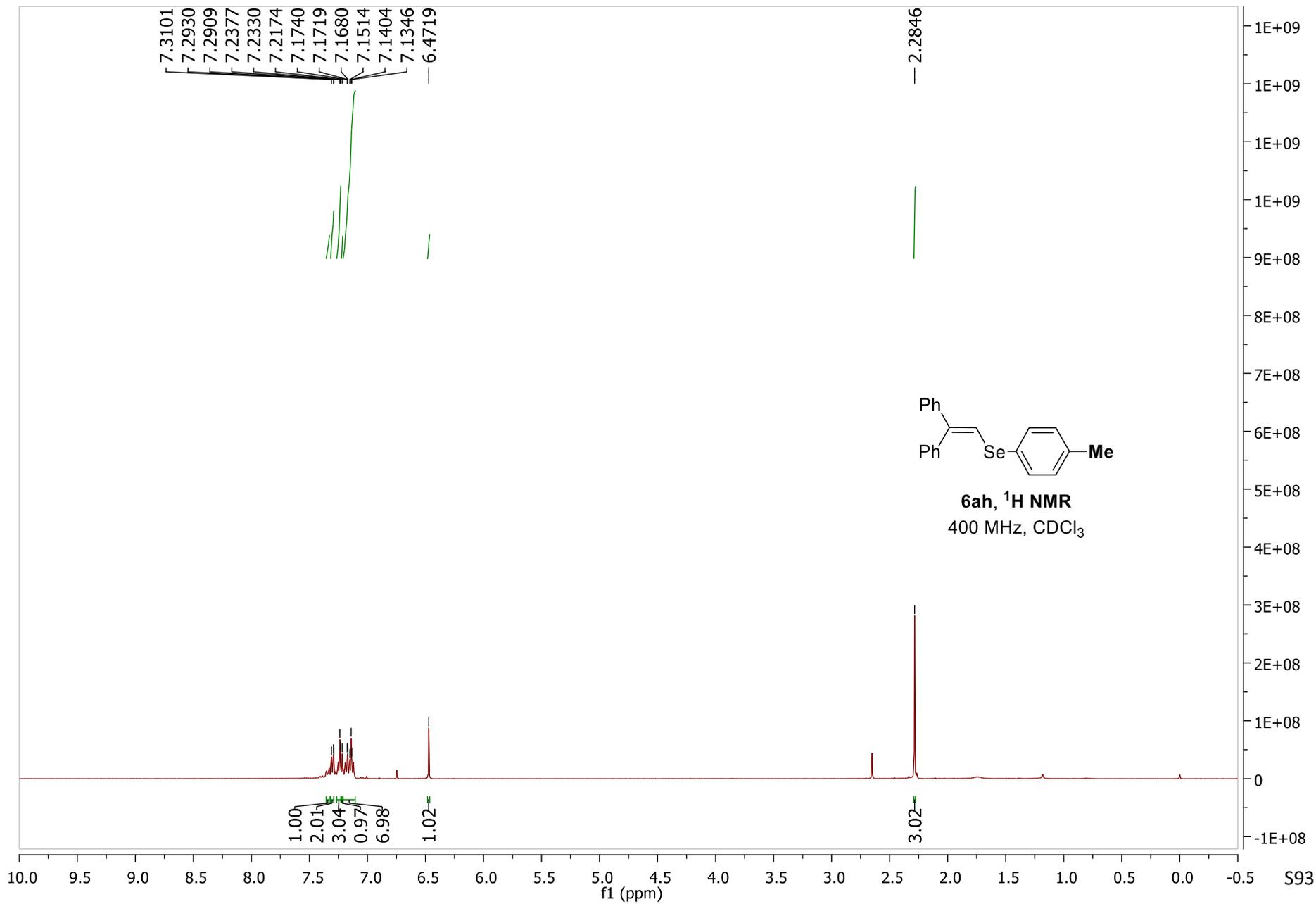


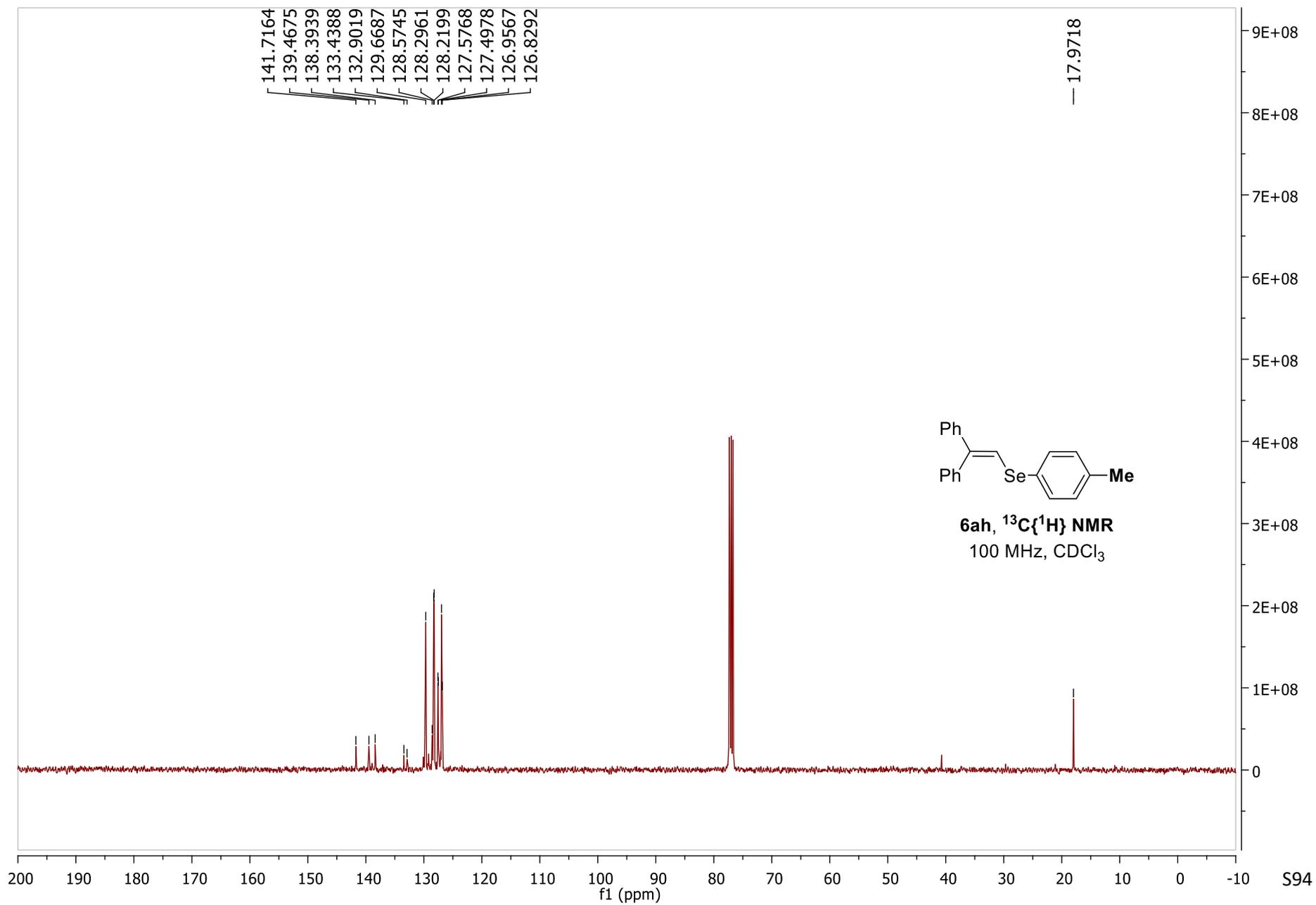


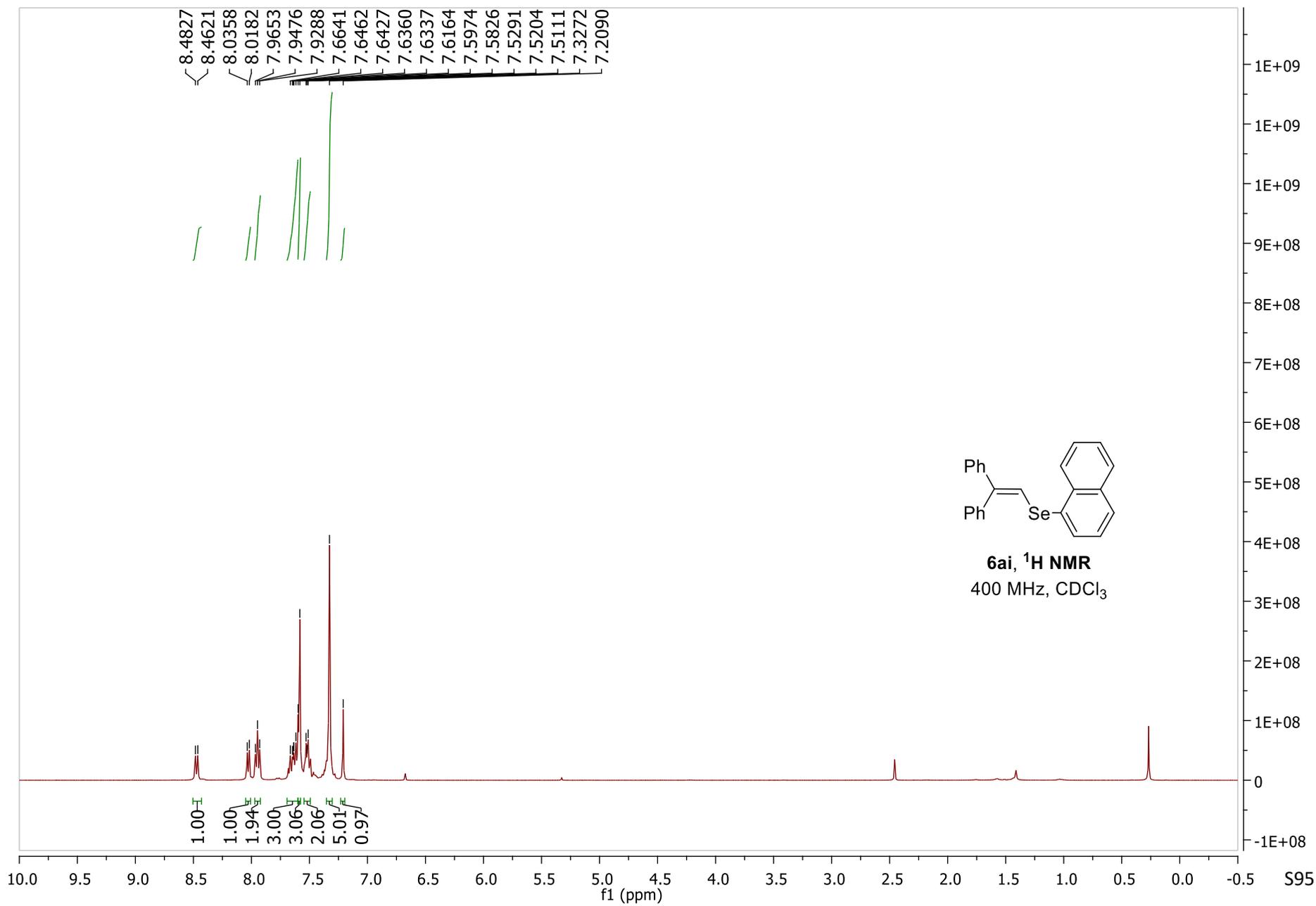


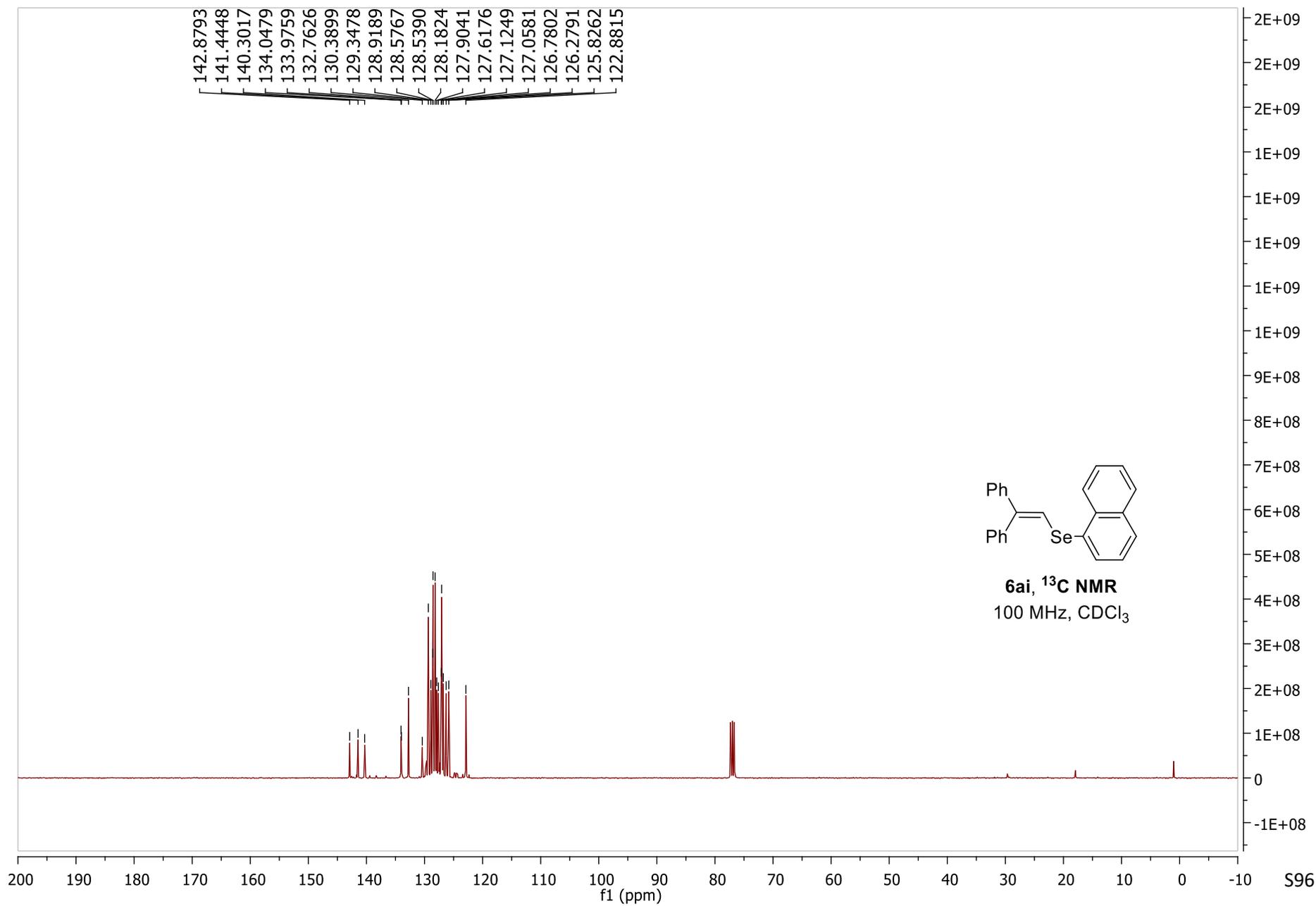


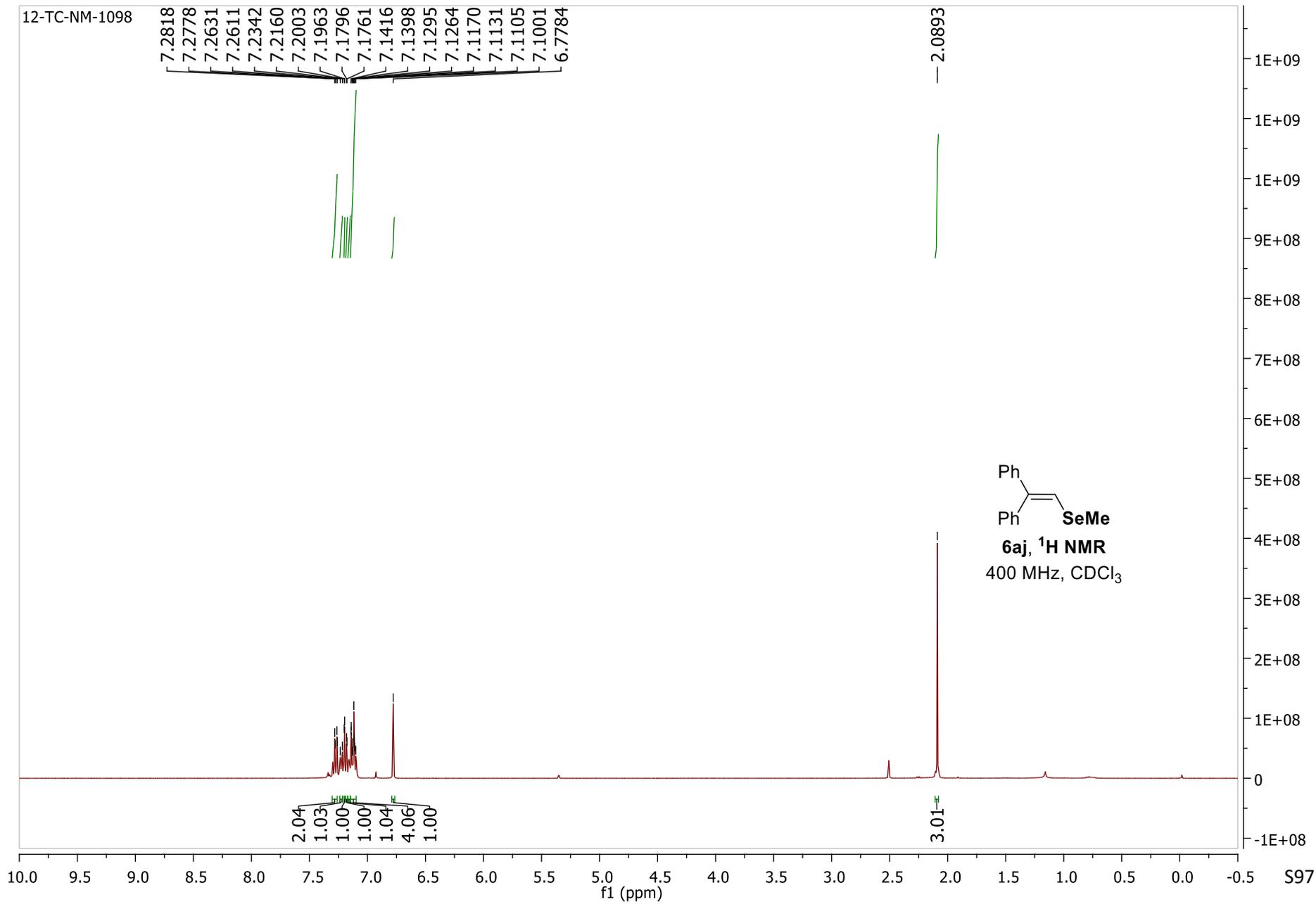


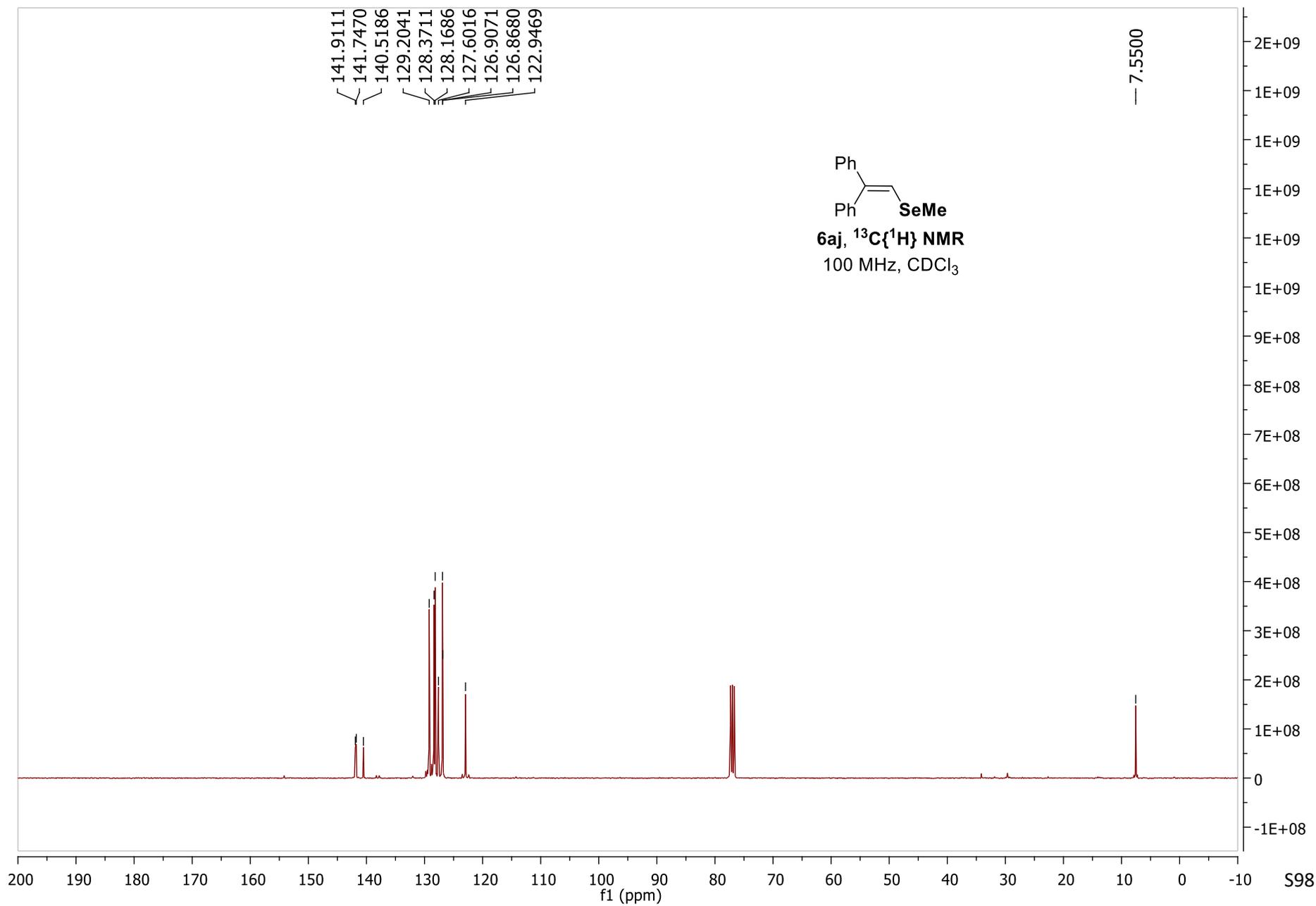


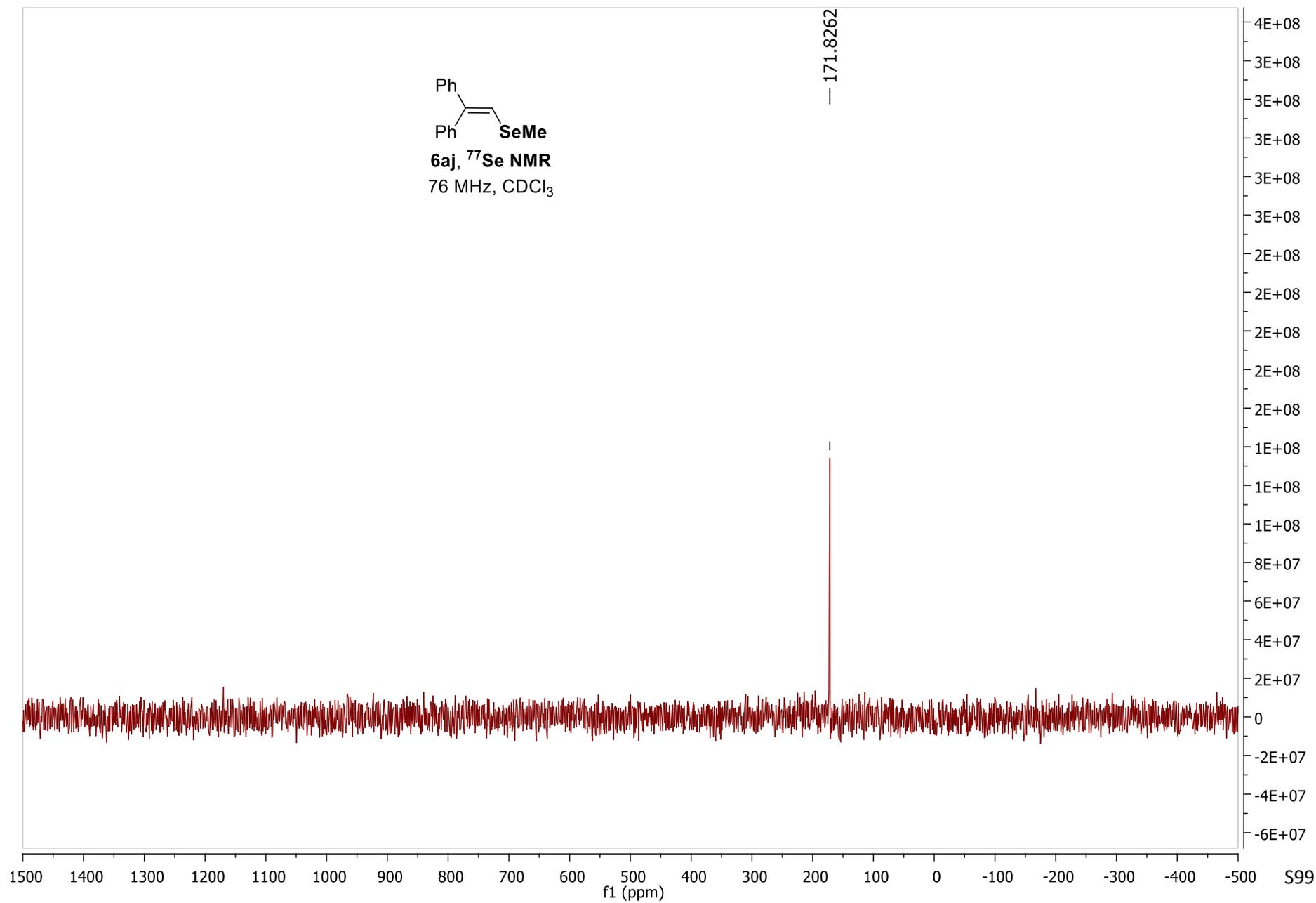


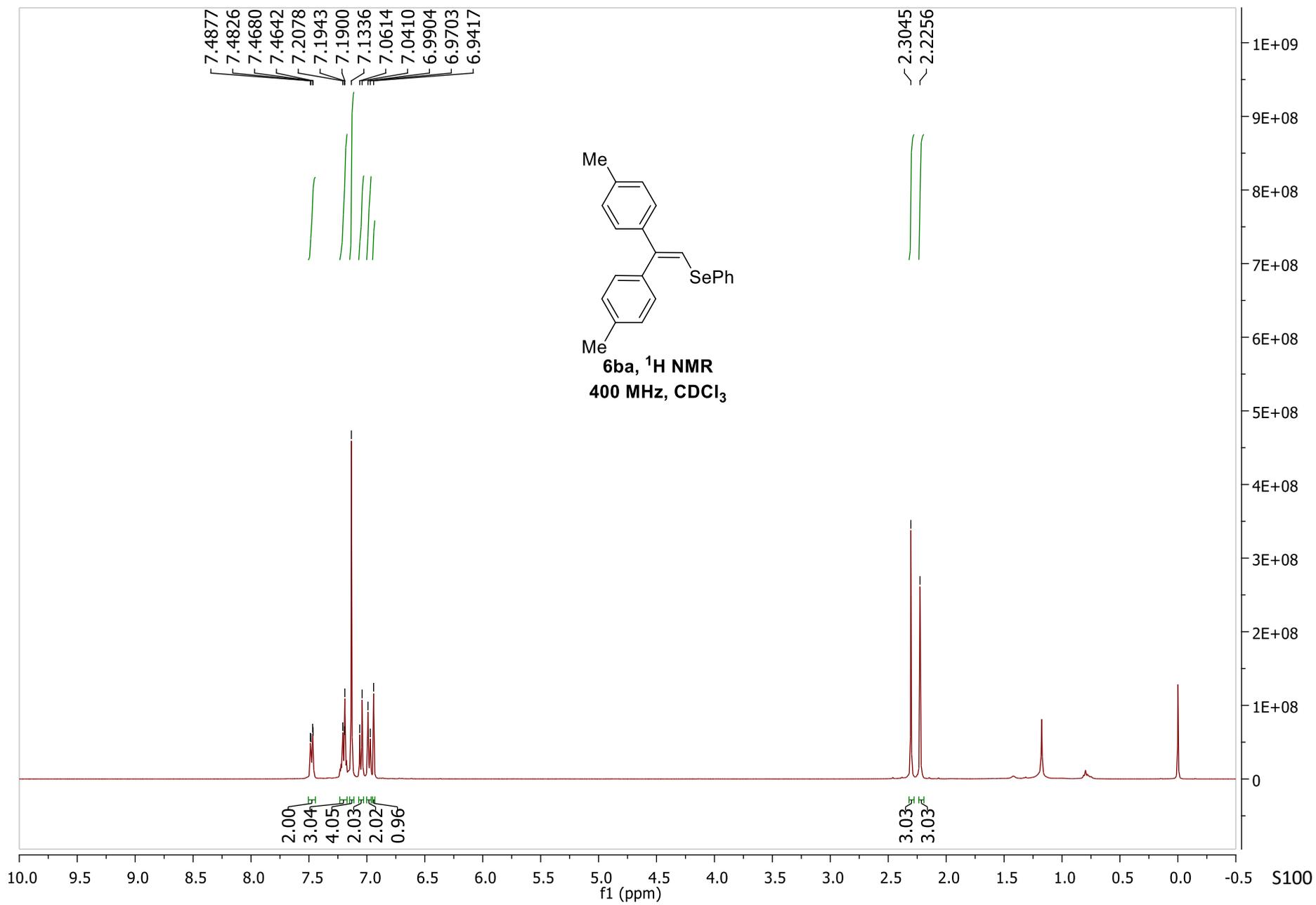


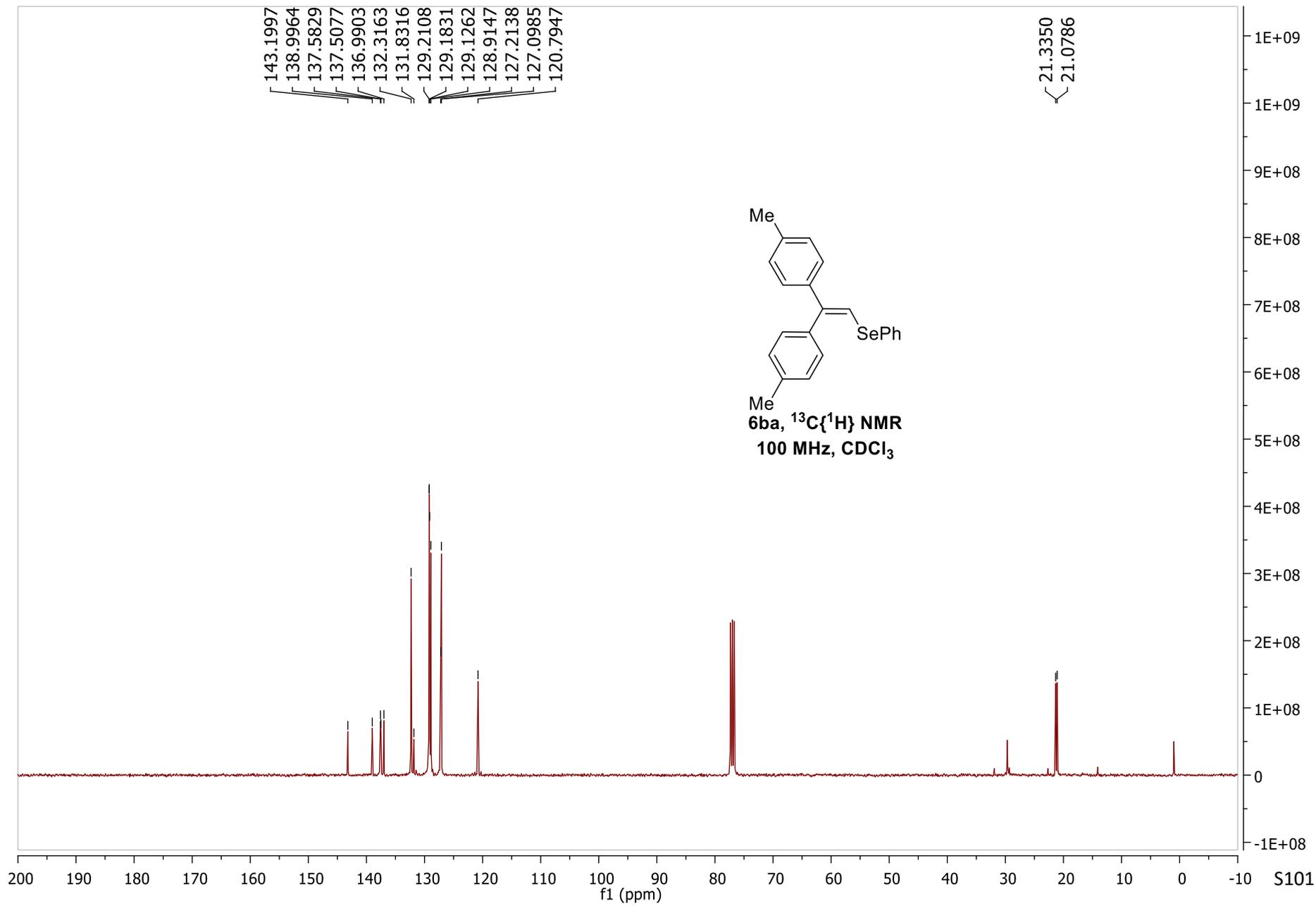


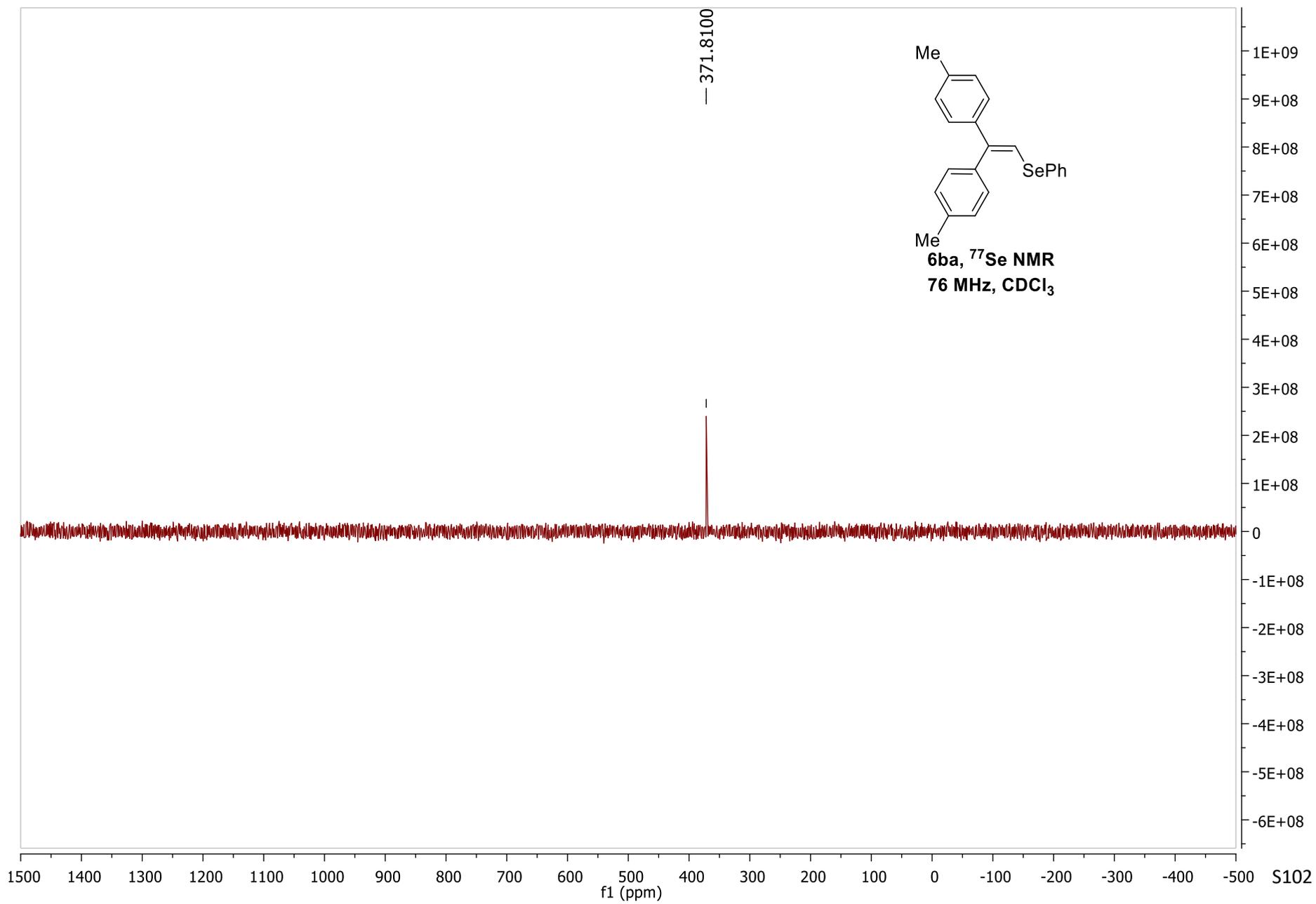


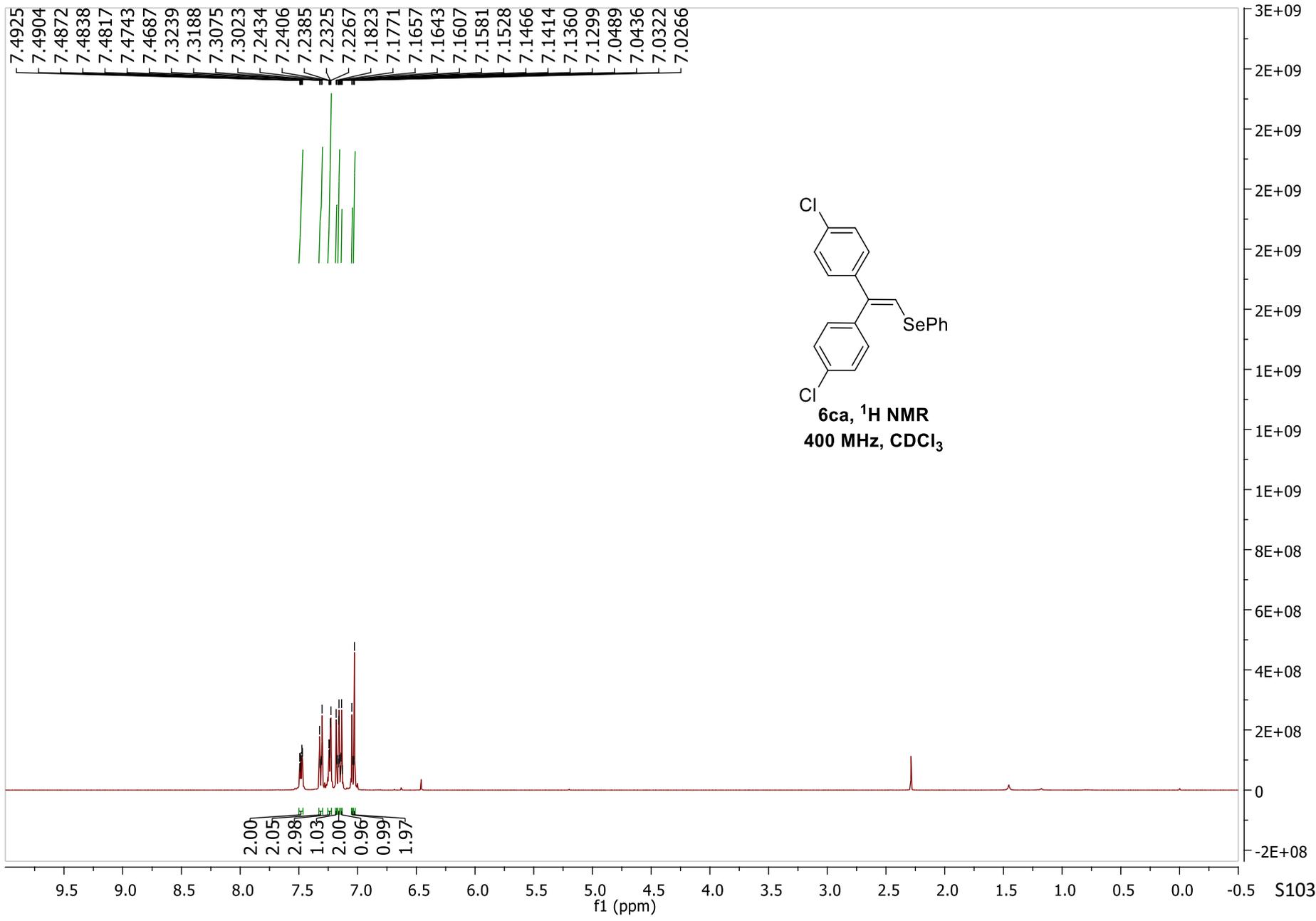


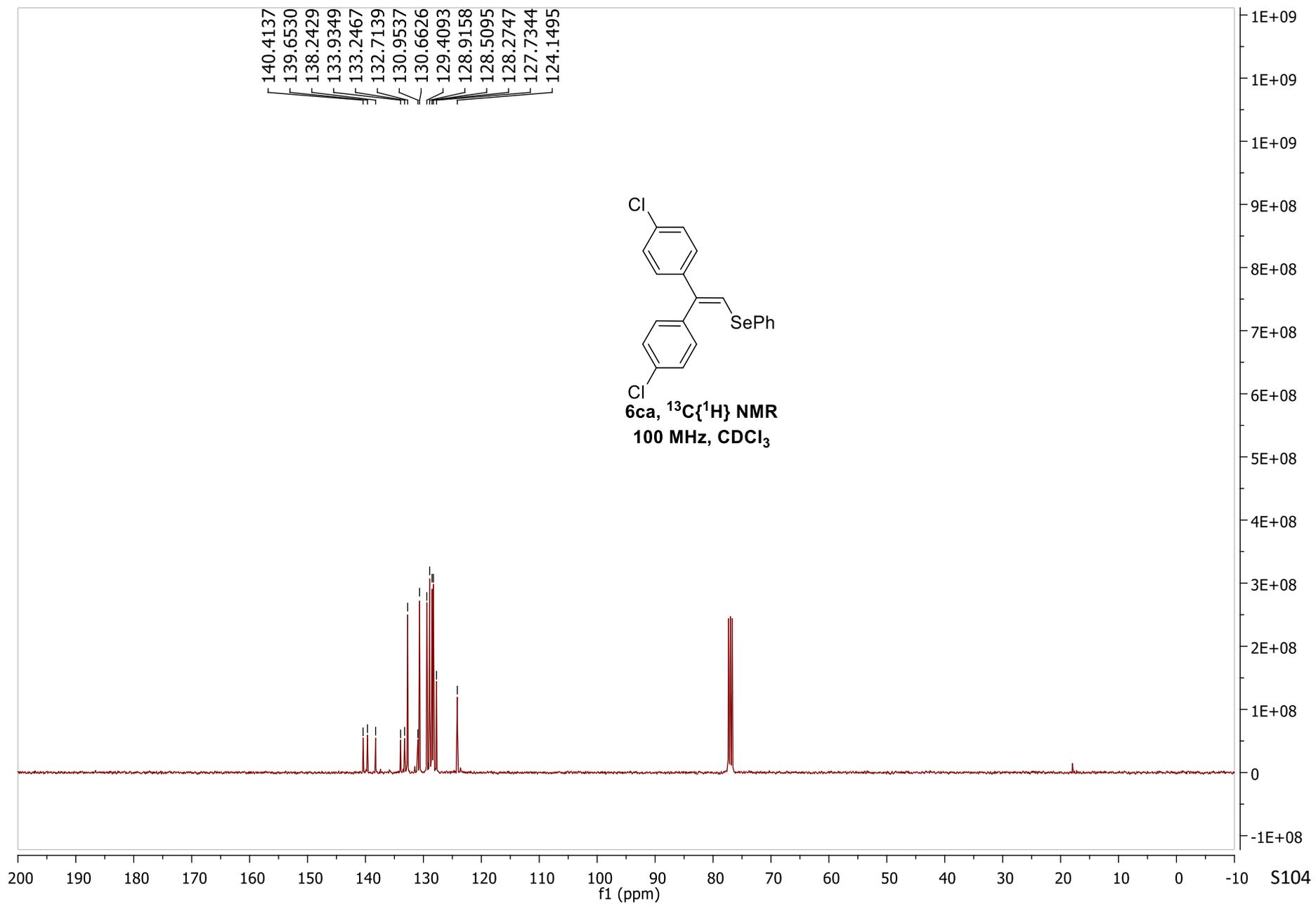


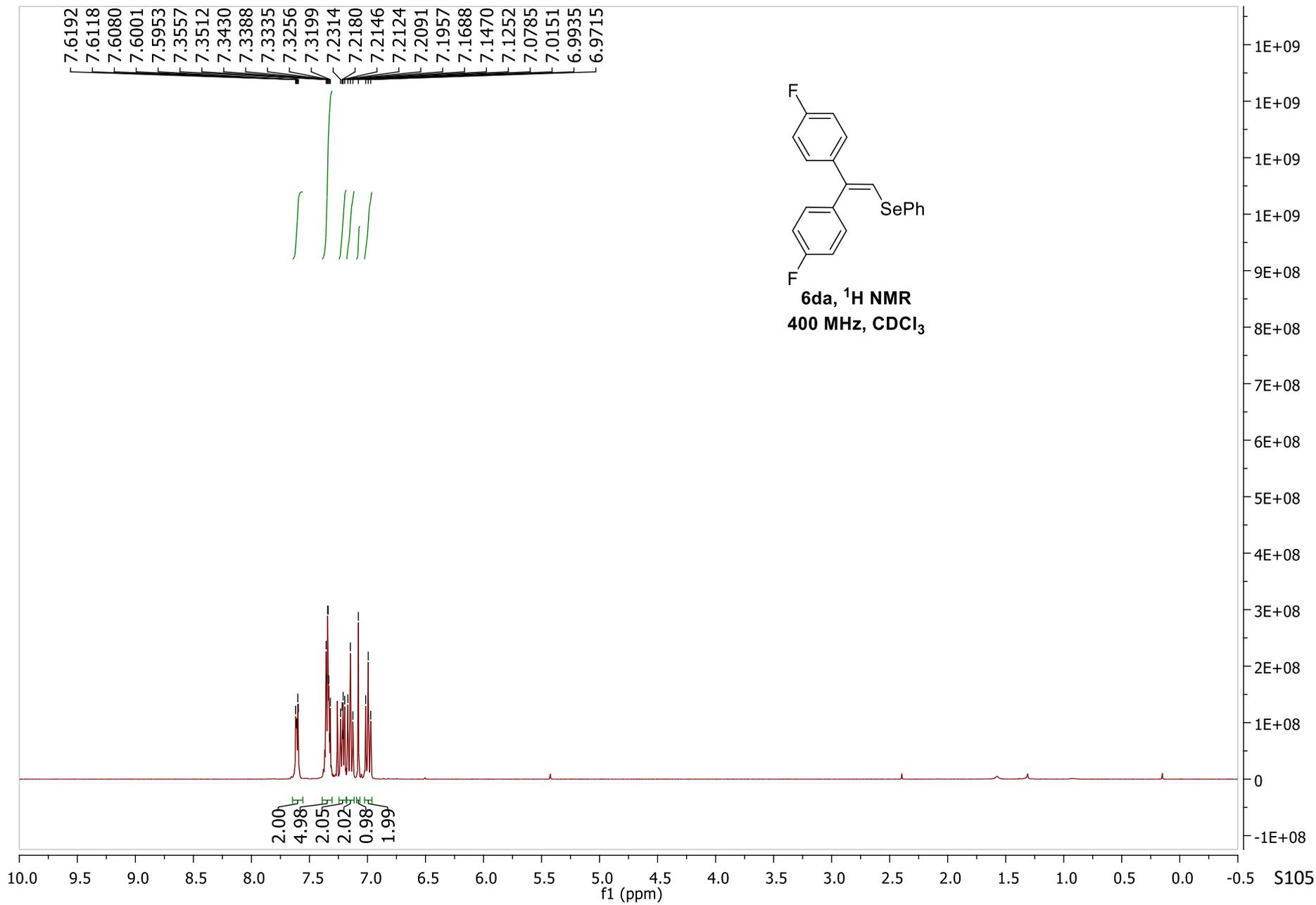


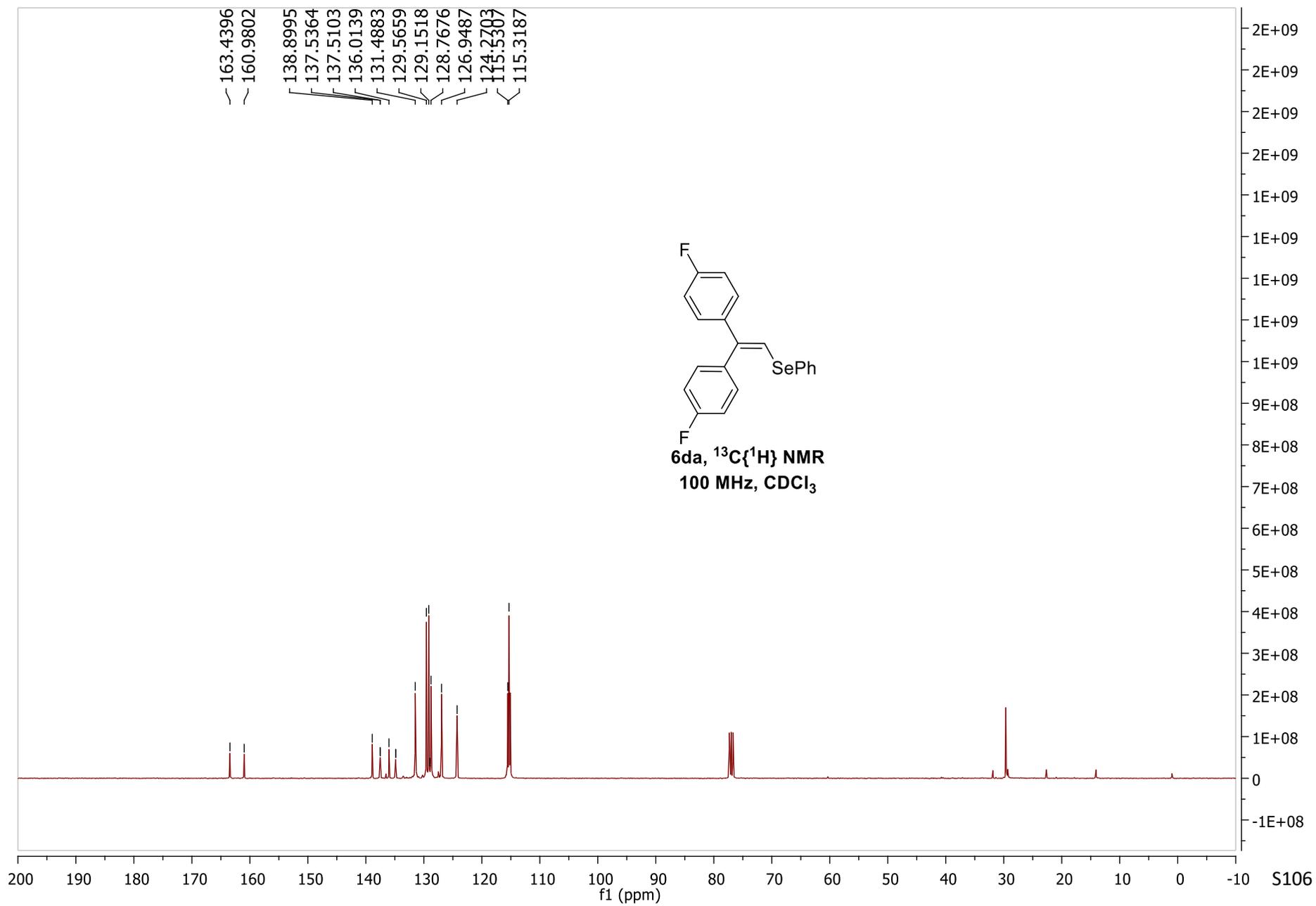


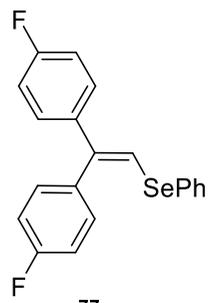












6da, ⁷⁷Se NMR
76 MHz, CDCl₃

— 378.0044

