Electronic Supplementary Information (ESI)

Iodine-mediated, chalcogen-chalcogen bond formation in water: Green synthesis of carbamo(dithioperoxo)thioates, carbamo(selenothioperoxo)thioates, carbono(dithioperoxo)thioates, and carbono(selenothioperoxo)thioates

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1. General consideration

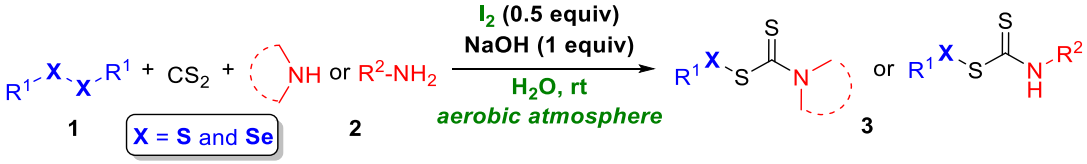
1.1 General reagent information

All reagents were purchased from BLD pharma, TCI chemicals, Sigma-Aldrich, AVRA, and SRL chemicals and used as such. Flash chromatography was performed using silica gel (100-200 mash).

1.2 General analytical information

All reagents were purchased from Spectrochem, Avra, BLD, and TCI chemicals. Potassium xanthate was prepared by following a literature protocol.[1] The products were characterized by ^{1}H, ^{13}C NMR spectra recorded on a Bruker 400 MHz instrument (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR). Copies of ^{1}H, and ^{13}C NMR spectra can be found at the end of the Supporting Information. ^{1}H NMR experiments are reported in units, parts per million (ppm), and were measured relative to residual chloroform (7.26 ppm) or DMSO (2.5 ppm) in the deuterated solvent. ^{13}C NMR spectra were reported in ppm relative to deuterochloroform (77.00 ppm) or DMSO-d_6 (40 ppm) and all were obtained with ^{1}H decoupling. Coupling constants were reported in Hz. Reactions were monitored by thin layer chromatography (TLC) and ^{1}H 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.

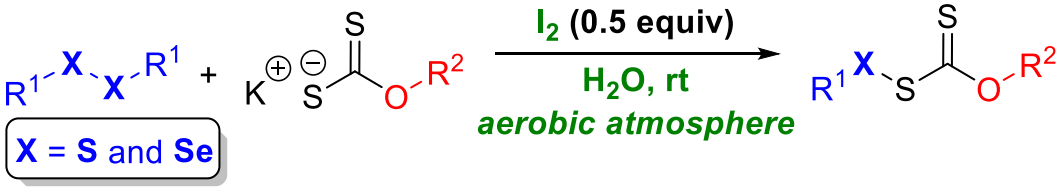
2. General experimental procedure for the synthesis of carbamo(dithioperoxo)thioates and carbamo(selenothioperoxo)thioates derivatives (3aa – 3af)



Representative experimental procedure for the synthesis of propyl piperidine-1-carbo(dithioperoxo)thioate (3aa): In a round-bottomed flask (RBF)**,** piperidine (0.085 g, 1 mmol, 1 equiv) and NaOH (0.04 g, 1 mmol, 1 equiv) were dissolved in water (2.5 mL), and then CS_2 (0.115 g, 1.5 mmol, 1.5 equiv) was added to the RBF at 0 °C and then the reaction mixture was stirred for 5 min (Solution A). In another 5 mL RBF, 1,2-dipropyldisulfane (0.150 g, 1 mmol, 1 equiv), crushed I_2 (0.127 g, 0.5 mmol, 0.5 equiv) were taken in H_2O (0.5 mL) and then solution A (aqueous solution of sodium piperidine-1-carbodithioate) was added dropwise over 15 min. The reaction mixture was stirred at room temperature for 30 min. The progress of the reaction was monitored by TLC. After the completion of the reaction, ethyl acetate (30 mL) was added to the reaction mixture and organic compounds were extracted and washed with water. The organic layer

was dried using anhydrous sodium sulfate and then the solvent was evaporated under reduced pressure. The crude product was purified by flash chromatography using 100-200 silica gel and 0-5% EtOAc in hexane as eluent to afford the pure propyl piperidine-1-carbo(dithioperoxo)thioate (**3aa**) (0.202 g, 0.858 mmol) as the yellow liquid in 86% yield. For the synthesis of **3ea**, **3fa**, **3ga**, **3ha**, **3ia**, **3ja**, **3ka, 3la, 3fc,** and **3ed** synthesis, the mixture of iodine and disulfide was heated at 60 ^{o}C and then solution A (aqueous solution of sodium piperidine-1-carbodithioate) was added dropwise over 15 min. For the synthesis of **3oa** and **3od**, a mixture of THF and water (2:1) was used as a solvent.

3. General experimental procedure for the synthesis of carbono(dithioperoxo)thioates, and carbono(selenothioperoxo)thioates (4a – 4f)

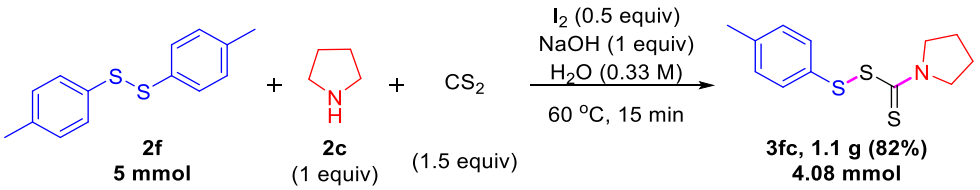


Representative experimental procedure for the synthesis of *O*-ethyl *SS*-propyl carbono(dithioperoxo)thioate (4b):

In a round-bottomed flask (RBF)**,** 1,2-dipropyldisulfane (0.150 g, 1 mmol, 1 equiv), crushed I_2 (0.127 g, 0.5 mmol, 0.5 equiv) were dissolved in H_2O (0.5 mL) to form (solution A). In another 5 mL RBF potassium ethylxanthate (0.161 g, 1 mmol, 1 equiv) was dissolved in 2.5 mL water (solution B). solution B (aqueous solution of potassium ethylxanthate) was added to the solution A dropwise over 15 min. The reaction mixture was stirred at room temperature for 30 min. The progress of the reaction was monitored by TLC. After completion of the reaction, ethyl acetate (30 mL) was added to the reaction mixture and organic compounds were extracted and washed with water. The organic layer was dried using anhydrous sodium sulfate and then the solvent was evaporated under reduced pressure. The crude product was purified by flash chromatography using 100-200 silica gel and hexane used as an eluent to afford the pure *O*-ethyl *SS*-propyl carbono(dithioperoxo)thioate (**4b**) (0.120 g, 0.896 mmol) as a yellow liquid in 61% yield.

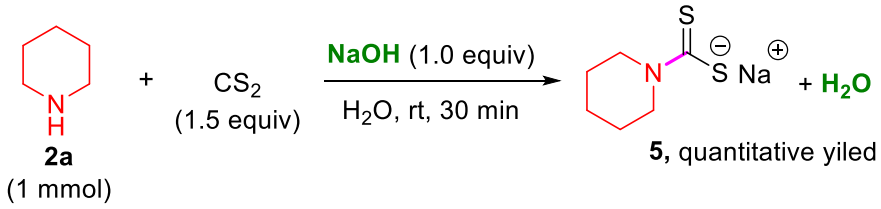
4. Scale up batch

Representative experimental procedure for the synthesis of *p*-tolyl pyrrolidine-1-carbo(dithioperoxo)thioate (3fc):



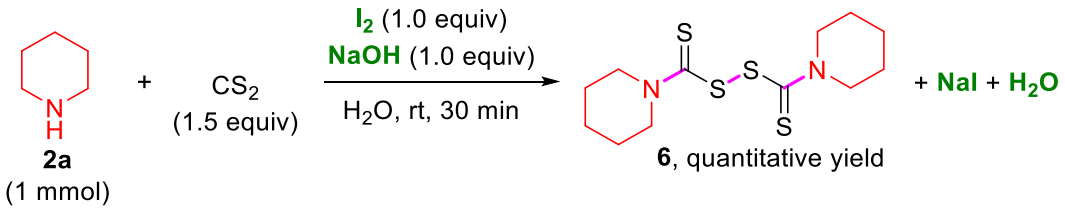
In a round-bottomed flask (RBF)**,** pyrrolidine (**2c)** (0.351 g, 5 mmol, 1 equiv) and NaOH (0.2 g, 1 equiv) were dissolved in water (3 mL), and then CS_2 (0.571 g, 7.5 mmol, 1.5 equiv) was added to thr RBF at 0 °C and then the reaction mixture was stirred for 5 min form a solution A (aqueous solution of sodium piperidine-1-carbodithioate). In another 10 mL RBF, 1,2-di-*p*-tolyldisulfane (**2f**) (1.23 g, 5 mmol, 1 equiv), crushed I_2 (0.635 g, 2.5 mmol, 0.5 equiv) were taken in H_2O (12 mL), heated the reaction mixture to 60 °C to form solution B and then solution A (aqueous solution of sodium piperidine-1-carbodithioate) was added to solution B dropwise over 15 min at the same temperature. The reaction mixture was immediately cooled to room temperature and stirred for 15 min. Progress of the reaction was monitored by TLC, After the completion of the reaction, ethyl acetate (30 mL) was added to the reaction mixture and organic compounds were extracted and washed with water. The organic layer was dried using anhydrous sodium sulfate and then the solvent was evaporated under reduced pressure. The crude product was purified by flash chromatography using 100-200 silica gel and 0-3% EtOAc in hexane as an eluent afford the pure *p*-tolyl pyrrolidine-1-carbo(dithioperoxo)thioate (**3fc**) (1.1 g, 4.08 mmol) as a yellow liquid in 82% yield.

5. General experimental procedure for the synthesis of sodium piperidine-1-carbodithioate (5)



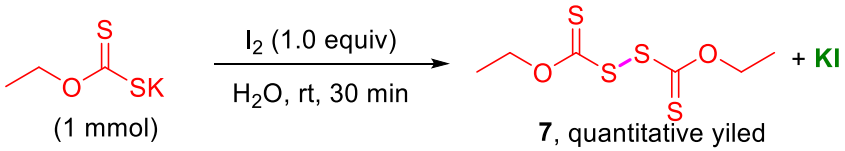
In a round-bottomed flask (RBF)**,** piperidine (0.425 g, 5 mmol, 1 equiv) and CS_2 (0.571 g, 7.5 mmol, 1.5 equiv) were dissolved in THF (5 mL), and then 0.2 mL aqueous solution of NaOH (0.2 g, 1 equiv) was added to the RBF at 0 °C and then the reaction mixture was stirred for 10 min and filtered the solid and dried to furnish sodium piperidine-1-carbodithioate (**5**) in quantitative yield as a white solid.

6. General experimental procedure for the synthesis of General experimental procedure for the synthesis of piperidine-1-carbothioic dithioperoxyanhydride (6)



In a round-bottomed flask (RBF)**,** sodium piperidine-1-carbodithioate (**5**) (0.184 g, 1 mmol) was dissolved in water (5 mL), and then crushed I_2 (0.254 g, 1 equiv) was added to the RBF at room temperature. The reaction mixture was stirred for 30 min at room temperature. After completion of the reaction, ethyl acetate (30 mL) was added to the reaction mixture and organic compounds were extracted and washed with water. The organic layer was dried using anhydrous sodium sulfate and then the solvent was evaporated under reduced pressure to afford piperidine-1-carbothioic dithioperoxyanhydride (**6**) in quantitative yield as a light yellow solid.

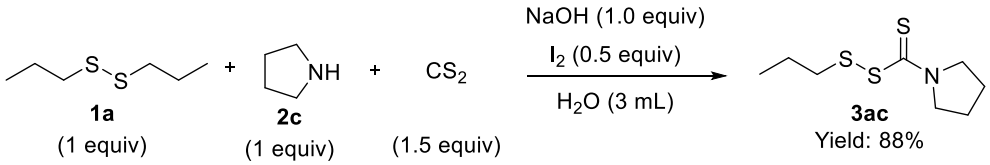
7. General experimental procedure for the synthesis of General experimental procedure for the synthesis of diethyl dithiobis(thionoformate) (7)



In a round-bottomed flask (RBF)**,** potassium *O*-ethyl carbonodithioate (0.161 g, 1 mmol) was dissolved in water (5 mL), and then crushed I_2 (0.254 g, 1 equiv) was added to the RBF at room temperature. The reaction mixture was stirred for 30 min at room temperature. After completion of the reaction, ethyl acetate (30 mL) was added to the reaction mixture and organic compounds were extracted and washed with water. The organic layer was dried using anhydrous sodium sulfate and then the solvent was evaporated under reduced pressure to afford piperidine-1-carbothioic dithioperoxyanhydride (**7**) in quantitative yield as a yellow liquid.

8. Evaluation of E-factor for compound 3ac

Table *S1*: Evaluation of E-factor of our protocol for the synthesis of propyl pyrrolidine-1-carbo(dithioperoxo)thioate **3ac**:



Reactant 1:	**1,2-dipropyldisulfane (1a)**	0.150 g	1 mmol	FW 150.3
Reactant 2:	**pyrrolidine (2c)**	0.0711 g	1 mmol	FW 71.12
Reactant 3:	**carbon disulfide (CS_2)**	0.114 g	1.5 mmol	FW 76.13
Reactant 4:	**sodium hydroxide (NaOH)**	0.04 g	1 mmol	FW 40
Reactant 5:	**iodine (I_2)**	0.127 g	0.5 mmol	FW 253.81
Solvent	**water (H_2O)**	3.0 g	--	FW 18
Product:	**propyl pyrrolidine-1-carbo(dithioperoxo)thioate (3ac)**	0.195 g	0.88 mmol	FW 221.4

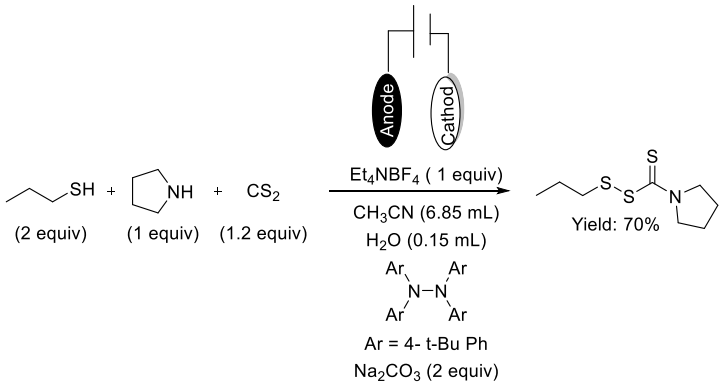
Consider water as a waste

$$\text{E-factor} = \frac{\text{total waste (g)}}{\text{total product (g)}} = \frac{(0.15 + 0.071 + 0.114 + 0.04 + 0.127 + 3) - 0.195}{0.195} = \mathbf{16.96}\ \text{g waste/g pdt}$$

Not considering water as a waste

$$\text{E-factor} = \frac{\text{total waste (g)}}{\text{total product (g)}} = \frac{(0.15 + 0.071 + 0.114 + 0.04 + 0.127) - 0.195}{0.195} = \mathbf{1.57}\ \text{g waste/g pdt}$$

Table *S2*: Evaluation of E-factor for recent literature (Green Chem., 2022, 24, 7362–7367) on the similar transformation, *i.e.*, for the synthesis of propyl pyrrolidine-1-carbo(dithioperoxo)thioate:



Reactant 1:	**propane-1-thiol**	0.0457 g	0.60 mmol	FW 76.16
Reactant 2:	**pyrrolidine**	0.0213 g	0.3 mmol	FW 71.12
Reactant 3:	**carbon disulfide**	0.0274 g	0.36 mmol	FW 76.13
Reactant 4:	**sodium carbonate**	0.0636 g	0.60 mmol	FW 105.99
Catalyst	**1,1,2,2-tetrakis(4-(*tert*-butyl)phenyl)hydrazine**	0.0336 g	0.02 mmol	FW 560.87
Electrolyte	**$(C_2H_5)_4N(BF_4)$**	0.0651 g	0.3 mmol	FW 217.16
Solvent 1	**acetonitrile**	5.38 g	--	FW 41.05
Solvent 2	**water**	0.15 g	--	FW 18
Product:	**propyl pyrrolidine-1-carbo(dithioperoxo)thioate**	0.047 g	0.209 mmol	FW 221.4

Considering water as a waste

$$\text{E-factor} = \frac{\text{total waste (g)}}{\text{total product (g)}} = \frac{(0.046 + 0.021 + 0.027 + 0.064 + 0.034 + 0.065 + 5.38 + 0.15) - 0.047}{0.047} = \mathbf{122.13}\ \text{g waste/g pdt}$$

Not considering water as a waste

$$\text{E-factor} = \frac{\text{total waste (g)}}{\text{total product (g)}} = \frac{(0.046 + 0.021 + 0.027 + 0.064 + 0.034 + 0.065 + 5.38\ ) - 0.047}{0.047} = \mathbf{118.94}\ \text{g waste/g pdt}$$

Not considering solvents (CH_3CN and H_2O) as a waste

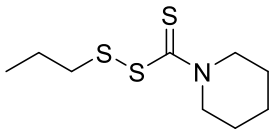
$$\text{E-factor} = \frac{\text{total waste (g)}}{\text{total product (g)}} = \frac{(0.046 + 0.021 + 0.027 + 0.064 + 0.034 + 0.065\ ) - 0.047}{0.047} = \mathbf{4.47}\ \text{g waste/g pdt}$$

9. Characteristic ^{13}C NMR signals of piperidine ring and a plausible explanation

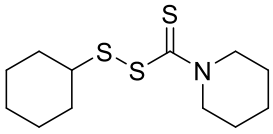
1. The nature of the ^{13}C peaks from the piperidine ring was found unique. The α-carbons and the β-carbons of the piperidine ring appeared non-equivalent in the ^{13}C-spectra, and these 13C-signals were found to be relatively broad with less intensity as compared to typical aliphatic carbons. Although these peaks are expected to be equivalent and similar in intensity to the typical aliphatic carbons, they appear to display partial double bond character, likely due to hindered rotation causing non-equivalence in the signals.

2. The intensity of ^{13}C NMR signals is dependent on relaxation time T1. Shorter relaxation times lead to shorter NMR signals. In piperidine, carbon attached to nitrogen might have shorter relaxation times, due to increased interaction with nearby nuclei.
3. The symmetry of piperidine causes multiple overlapping signals in its ^{13}C NMR spectra, particularly if it is the simple, unsubstituted form. Sometimes the signals appear less intense when peaks overlap or are closely spaced.

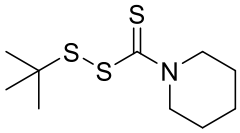
10. Analytical data of the synthesized products



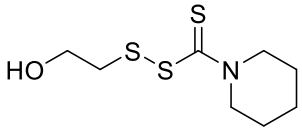
Propyl piperidine-1-carbo(dithioperoxo)thioate (3aa)[2] : yellow gummy (0.202 g, 86%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.26 (br s, 2H), 3.93 (br s, 2H), 2.78 (t, *J* = 7.4 Hz, 2H), 1.68 (br s, 6H), 1.67 – 1.59 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H)**; ^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.0, 55.0, 51.7, 40.5, 26.0, 25.3, 24.0, 21.7, 13.0; HRMS (ESI) m/z calcd for $C_9H_{18}NS_3$ $[M+H]^+$: 236.0601; found: 236.0603.



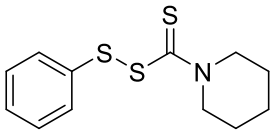
Cyclohexyl piperidine-1-carbo(dithioperoxo)thioate (3ba): yellow liquid (0.183 g, 66%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.27 (br s, 2H), 3.99 (br s, 2H), 2.99 (tt, *J* = 10.6, 3.7 Hz, 1H), 2.18 – 1.91 (m, 2H), 1.83 – 1.51 (m, 9H), 1.47 – 1.09 (m, 5H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.9, 55.2, 51.8, 49.2, 32.8, 32.6, 32.2, 26.1, 25.9, 25.7, 25.6, 24.1; HRMS (ESI) m/z calcd for $C_{12}H_{22}NS_3$ $[M+H]^+$: 276.0914; found: 276.0918.



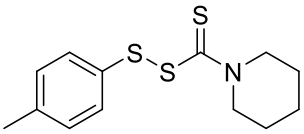
Tert-butyl piperidine-1-carbo(dithioperoxo)thioate (3ca)[2]: yellow liquid (0.121 g, 48%); **^{1}H NMR (400 MHz, DMSO-d_6)** δ 4.33 – 3.80 (m, 4H), 1.77 – 1.54 (m, 6H), 1.44 – 1.22 (m, 9H); **^{13}C NMR (100 MHz, DMSO-d_6)** δ 192.2, 55.6, 52.6, 49.7, 29.9, 26.7, 25.7, 23.9, 23.8; HRMS (ESI) m/z calcd for $C_{10}H_{20}NS_3$ $[M+H]^+$: 250.0758; found: 250.0761.



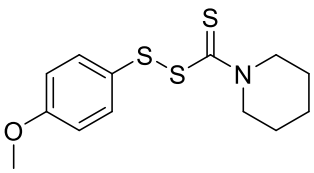
2-Hydroxyethyl piperidine-1-carbo(dithioperoxo)thioate (3da)[3]: off-white solid (0.118 g, 50%); melting point: 42- 43 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.29 (br s, 2H), 3.96 (br s, 2H), 3.68 (t, *J* = 4.0 Hz, 2H), 2.89 (t, *J* = 4.0 Hz, 2H), 1.70 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.7, 58.1, 55.8, 51.9, 43.1, 26.2, 25.3, 23.9.



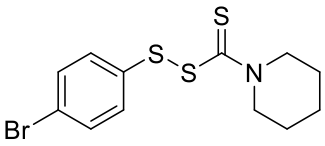
Phenyl piperidine-1-carbo(dithioperoxo)thioate (3ea): off-white solid (0.195 g, 73%); melting point: 91- 93 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.53 – 7.41 (m, 2H), 7.25 – 7.12 (m, 3H), 4.21 (br s, 2H), 3.91 (br s, 2H), 1.64 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 194.3, 136.1, 129.0, 128.8, 127.7, 55.5, 51.9, 26.2, 25.4, 24.0; HRMS (ESI) m/z calcd for $C_{12}H_{16}NS_3$ $[M+H]^+$: 270.0445; found: 270.0445.



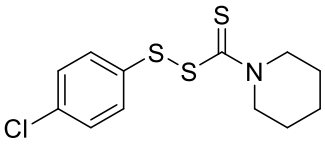
***p*-Tolyl piperidine-1-carbo(dithioperoxo)thioate (3fa):** off-white solid (0.255 g, 90%); melting point: 53- 55 °C; **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 7.40 (d, *J* = 8.2 Hz, 2H), 7.19 – 7.11 (m, 2H), 4.22 (s, 2H), 3.98 (br s, 2H), 2.27 (br s, 3H), 1.66 – 1.56 (m, 6H); **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 192.6, 138.2, 132.7, 130.2, 129.5, 55.5, 52.2, 26.6, 25.7, 23.8, 21.1; HRMS (ESI) m/z calcd for $C_{13}H_{18}NS_3$ $[M+H]^+$: 284.0601; found: 284.0601.



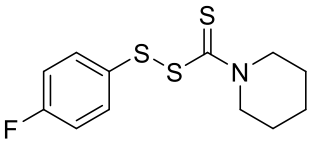
4-Methoxyphenyl piperidine-1-carbo(dithioperoxo)thioate (3ga): off-white solid (0.232 g, 77%); melting point: 70- 72 °C; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.62 (d, *J* = 8.9 Hz, 1H), 6.82 (d, *J* = 8.9 Hz, 1H), 4.28 (s, 1H), 3.94 (s, 1H), 3.86 (s, *J* = 65.1 Hz, 3H), 1.70 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 195.2, 160.3, 133.6, 127.1, 114.5, 55.3, 51.9, 26.1, 25.5, 24.1; HRMS (ESI) m/z calcd for $C_{13}H_{18}NOS_3$ $[M+H]^+$: 300.0551; found: 300.0550.



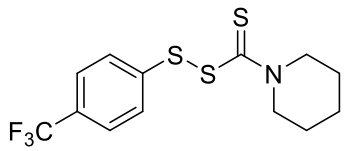
4-Bromophenyl piperidine-1-carbo(dithioperoxo)thioate (3ha): white solid (0.225 g, 65%); melting point: 90- 92 °C; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.44- 7.38 (m, 4H), 4.28 (br s, 2H), 3.98 (br s, 2H), 1.72 (s, 6H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 193.7, 135.4, 132.1, 131.8, 130.8, 129.3, 55.5, 52.0, 26.3, 25.4, 24.0; HRMS (ESI) m/z calcd for $C_{12}H_{15}BrNS_3$: $[M+H]^+$:347.9550; found: 347.9549.



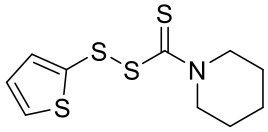
4-Chlorophenyl piperidine-1-carbo(dithioperoxo)thioate (3ia): yellow solid (0.218 g, 72%); melting point: 89- 91 °C; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.42 (d, *J* = 8.7 Hz, 2H), 7.18 (d, *J* = 8.7 Hz, 2H), 4.21 (br s, 2H), 3.9 (br s, 2H), 1.64 (s, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 193.7, 134.7, 133.8, 130.6, 128.9, 55.5, 52.0, 26.2, 25.4, 23.9; HRMS (ESI) m/z calcd for $C_{12}H_{15}ClNS_3$ $[M+H]^+$: 304.0055; found: 304.0055.



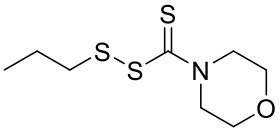
4-Fluorophenyl piperidine-1-carbo(dithioperoxo)thioate (3ja): light yellow solid (0.210 g, 73%); melting point: 95- 97 °C; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.46- 7.42 (m, 2H), 7.13- 709 (m, 2H), 4.27 (br s, 2H), 3.98 (br s, 2H), 1.73 (s, 6H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 195.6, 163.8 (d, *J* = 250.8 Hz), 139.1 (d, *J* = 8.7 Hz), 126.9 (d, *J* = 2.7 Hz), 116.1 (d, *J* = 22.1 Hz), 53.3, 51.8, 26.0, 25.2, 24.0; **^{19}F NMR (377 MHz, CDCl$_3$)** δ -110.1; HRMS (ESI) m/z calcd for $C_{12}H_{15}FNS_3$ $[M+H]^+$ :: 288.0351; found: 288.0349.



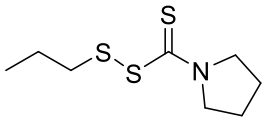
4-(Trifluoromethyl)phenylpiperidine-1-carbo(dithioperoxo) thioate (**3ka**): yellow gummy liquid (0.275 g ,82%); **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.43 (d, *J* = 8.2 Hz, 2H), 7.16 – 7.11 (m, 2H), 4.71 (q, *J* = 7.1 Hz, 2H), 2.34 (s, 3H), 1.47 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, CDCl$_3$)** δ 193.0, 140.9, 129.3 (q, *J* = 32.7 Hz), 125.7, 125.6, 55.7, 52.2, 26.3, 25.5, 24.0;**^{19}F NMR (377 MHz, CDCl$_3$)** δ -62.5; HRMS (ESI) m/z calcd for $C_{13}H_{15}F_3NS_3$ $[M+H]^+$: 338.0319; found: 338.0304.



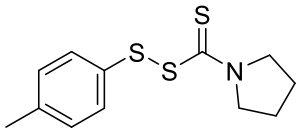
Thiophen-2-yl piperidine-1-carbo(dithioperoxo)thioate (3la): yellow solid (0.165 g, 60%); melting point: 62- 64 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.64 (dd, *J* = 5.3, 1.2 Hz, 1H), 7.27 – 7.23 (m, 1H), 7.14 (dd, *J* = 5.3, 3.6 Hz, 1H), 4.27 (s, 2H), 3.98 (s, 2H), 1.74 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.7, 138.9, 133.4, 129.1, 127.8, 53.8, 51.8, 26.2, 25.3, 24.1; HRMS (ESI) m/z calcd for $C_{10}H_{14}NS_4$ $[M+H]^+$:276.0009; found: 276.0007.



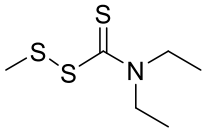
Propyl morpholine-4-carbo(dithioperoxo)thioate (3ab): off-white solid (0.166 g, 70%); melting point: 82- 84 °C; **^{1}H NMR (400 MHz, DMSO-d_6)** δ 4.14 (d, *J* = 77.4 Hz, 4H), 3.70 (s, 4H), 2.80 (t, *J* = 7.1 Hz, 2H), 1.70 – 1.52 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, DMSO-d_6)** δ 196.1, 66.1, 53.7, 51.3, 40.2, 21.9, 13.3; HRMS (ESI) m/z calcd for $C_8H_{16}NOS_3$ $[M+H]^+$: 238.0394; found: 238.0396.



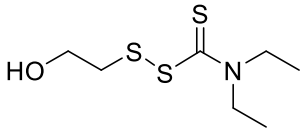
Propyl pyrrolidine-1-carbo(dithioperoxo)thioate (3ac)[2]: yellow liqiuid (0.195 g, 88%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 3.94 (t, *J* = 7.0 Hz, 2H), 3.73 (t, *J* = 6.9 Hz, 2H), 2.82 (t, *J* = 7.3 Hz, 2H), 2.14 – 2.06 (m, 2H), 2.01-1.94 (m, 2H), 1.74 – 1.62 (m, 2H), 0.99 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 192.9, 56.6, 50.5, 40.5, 26.4, 24.1, 21.9, 13.1.



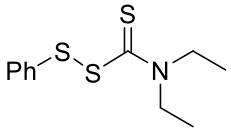
***p*-Tolyl pyrrolidine-1-carbo(dithioperoxo)thioate (3fc)[2]:** off-white solid (1.1 g, 82%); melting point: 121- 123 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.53 (d, *J* = 8.2 Hz, 2H), 7.10 (d, *J* = 7.9 Hz, 2H), 3.96 (t, *J* = 7.0 Hz, 2H), 3.74 (t, *J* = 6.9 Hz, 2H), 2.31 (s, 3H), 2.17 – 2.05 (m, 2H), 2.06 – 1.91 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 191.7, 138.5, 132.7, 130.6, 129.7, 56.8, 50.5, 26.6, 24.1, 21.1; HRMS (ESI) m/z calcd for $C_{12}H_{16}NS_3$ $[M+H]^+$: 270.0445; found: 270.0446.



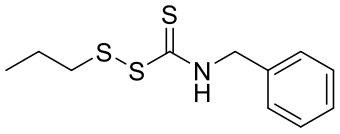
Methyl diethylcarbamo(dithioperoxo)thioate (3md): yellow liquid (0.101 g, 52%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.13 – 3.96 (m, 1H), 3.80-3.75 (m, 1H), 2.48 (s, 1H), 1.33-1.27 (m, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.5, 51.5, 46.9, 22.9, 13.0, 11.4; HRMS (ESI) m/z calcd for $C_6H_{14}NS_3$ $[M+H]^+$: 196.0288; found: 196.0288.



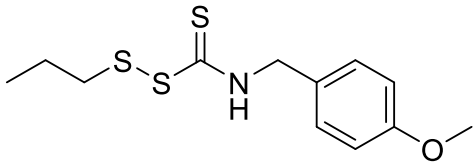
2-Hydroxyethyl diethylcarbamo(dithioperoxo)thioate (**3dd**)[3]: yellow liquid (0.118 g, 52%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.01 (q, *J* = 7.0 Hz, 2H), 3.80 (q, *J* = 7.2 Hz, 2H), 3.67 (t, *J* = 5.2 Hz, 2H), 3.28 (br s, 1H), 2.87 (t, *J* = 5.2 Hz, 1H), 1.28 (dt, *J* = 15.9, 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.7, 58.1, 52.1, 47.1, 43.1, 12.9, 11.2.



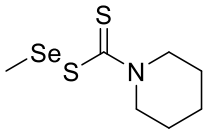
Phenyl diethylcarbamo(dithioperoxo)thioate (3ed): yellow solid (186 mg, 73%); melting point: 116- 118 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.60 – 7.50 (m, 2H), 7.38 – 7.17 (m, 3H), 4.06 (q, 2H), 3.84 (q, 1H), 1.37-1.29 (m, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 194.3, 136.1, 129.1, 128.9, 127.8, 51.9, 47.0, 13.2, 11.4; HRMS (ESI) m/z calcd for $C_{11}H_{16}NS_3$ $[M+H]^+$: 258.0445; found: 258.0448.



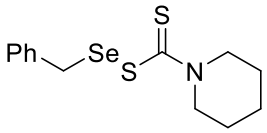
Propyl benzylcarbamo(dithioperoxo)thioate (3ae): yellow liquid (0.240 g, 94%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.78 (s, 1H), 7.50 – 7.31 (m, 5H), 4.94 (d, *J* = 5.4 Hz, 2H), 2.69 (t, *J* = 7.2 Hz, 2H), 1.68 – 1.56 (m, 3H), 0.91 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 191.6, 135.5, 129.0, 128.3, 128.1, 50.2, 41.7, 22.6, 12.9; HRMS (ESI) m/z calcd for $C_{11}H_{16}NS_3$ $[M+H]^+$: 258.0445; found: 258.0447.



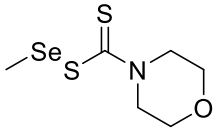
Propyl (4-methoxybenzyl)carbamo(dithioperoxo)thioate (3af): yellow solid (0.205 g, 71%); melting point: 85- 87 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.70 (s, 1H), 7.28 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 4.86 (d, *J* = 5.3 Hz, 2H), 3.82 (s, 3H), 2.67 (t, *J* = 8.0 Hz, 2H), 1.72 – 1.40 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 191.2, 159.6, 129.6, 127.4, 114.3, 55.3, 49.7, 41.6, 22.6, 12.9; HRMS (ESI) m/z calcd for $C_{12}H_{18}NOS_3$ $[M+H]^+$: 288.0551; found: 288.0474.



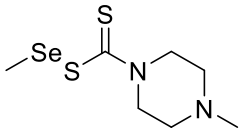
***SSe*-Methyl piperidine-1-carbo(selenothioperoxo)thioate (3na):** yellow oil (0.057 g, 45%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.29 (br s, 2H), 3.81 (br s, 2H), 2.55 (s, 3H), 1.71 – 1.67 (m, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 193.4, 53.5, 53.3, 26.1, 25.3, 24.2, 20.0, 14.8; **^{77}Se NMR (76 MHz, $CDCl_3$)** δ 483.7; HRMS (ESI) m/z calcd for $C_7H_{14}NS_2Se$ $[M+H]^+$: 255.9733; found: 255.9730.



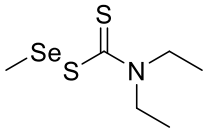
***SSe*-Benzyl piperidine-1-carbo(selenothioperoxo)thioate** (**3oa**): yellow liquid (0.182g, 56%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.31 (dd, *J* = 7.7, 1.0 Hz, 2H), 7.25 – 7.21 (m, 2H), 7.19 – 7.16 (m, 1H), 4.49 (s, 2H), 4.22 (br s, 2H), 3.78 (br s, 2H), 1.62 (br s, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.2, 136.0, 129.3, 128.5, 127.4, 52.8, 51.2, 42.2, 25.9, 25.4, 24.2; **^{77}Se NMR (76 MHz, $CDCl_3$)** δ 383.2; HRMS (ESI) m/z calcd for $C_{13}H_{18}NS_2Se$ $[M+H]^+$: 332.0046; found: 332.0059.



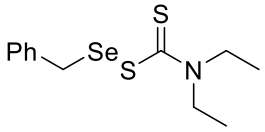
***SSe*-Methyl morpholine-4-carbo(selenothioperoxo)thioate (3nb)**: yellow liquid (0.154 g, 60%); **^{1}H NMR (400 MHz, DMSO-d_6)** δ 4.28 - 4.20 (m, 4H), 3.80- 3.72 (m, 4H), 2.55 (s, 3H); **^{13}C NMR (100 MHz, DMSO-d_6)** δ 195.1, 66.3, 66.2, 53.1, 14.6; **^{77}Se NMR (76 MHz, DMSO-d_6)** δ 357.6; HRMS (ESI) m/z calcd for $C_6H_{12}NOS_2Se$ $[M+H]^+$: 257.9526; found: 257.9537.



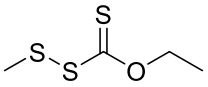
***SSe*-Methyl 4-methylpiperazine-1-carbo(selenothioperoxo)thioate** (**3ng**): yellow liquid (0.178 mg, 67%); **^{1}H NMR (400 MHz, DMSO-d_6)** δ 4.16 (br s, 4H), 2.49 (s, 3H), 2.45 – 2.38 (m, 4H), 2.22 (s, 3H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 194.5, 54.6, 52.5, 45.5, 14.6; **^{77}Se NMR (76 MHz, DMSO-d_6)** δ 254.2; HRMS (ESI) m/z calcd for $C_7H_{15}N_2S_2Se$ $[M+H]^+$: 270.9842; found: 270.9919.



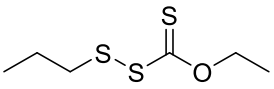
***SSe*-Methyl diethylcarbamo(selenothioperoxo)thioate (3nd):** yellow liquid (0.061 g, 50%); **^{1}H NMR (400 MHz, CDCl3)** δ 4.04 (dd, J = 13.2, 6.3 Hz, 2H), 3.74 (dd, J = 13.9, 6.9 Hz, 2H), 2.63 (s, 3H), 1.31 – 1.25 (m, 6H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 196.8, 49.5, 46.6, 20.0, 12.3, 11.6; **^{77}Se NMR (76 MHz, $CDCl_3$)** δ 385.2; HRMS (ESI) m/z calcd for $C_6H_{14}NS_2Se$ $[M+H]^+$: 243.9733; found: 243.9732.



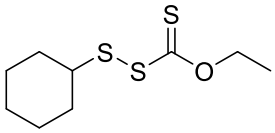
***SSe*-Benzyl diethylcarbamo(selenothioperoxo)thioate (3od):** yellow liquid (0.081 g, 51%); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 195.29, 136.01, 129.4, 129.0, 128.6, 127.5, 49.5, 46.7, 42.3, 12.5, 11.6; **^{77}Se NMR (76 MHz, $CDCl_3$)** δ 330.5; HRMS (ESI) m/z calcd for $C_{12}H_{18}NS_2Se$ $[M+H]^+$: 320.0046; found: 320.0044.



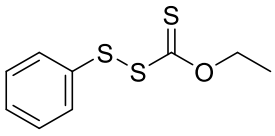
***O*-Ethyl *SS*-methyl carbono(dithioperoxo)thioate (4a)**: yellow liquid (0.088g, 53%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.75 – 4.70 (q, *J* = 8.0 Hz, 2H), 2.52 (s, 3H), 1.48 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 212.83, 71.35, 22.78, 13.67; HRMS (ESI) m/z calcd for $C_4H_9OS_3$ $[M+H]^+$: 168.9816; found: 168.9814.



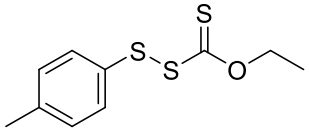
***O*-Ethyl *SS*-propyl carbono(dithioperoxo)thioate (4b)**: yellow liquid (0.140g, 71%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.70 (q, *J* = 7.1 Hz, 2H), 2.90 – 2.77 (m, 2H), 1.77 – 1.63 (m, 2H), 1.47 (t, *J* = 7.1 Hz, 3H), 1.00 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 213.2, 71.2, 41.2, 22.3, 13.6, 13.0; HRMS (ESI) m/z calcd for $C_6H_{13}OS_3$ $[M+H]^+$: 197.0129; found: 197.0128.



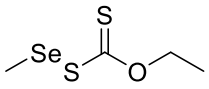
***SS*-Cyclohexyl *O*-ethyl carbono(dithioperoxo)thioate** (**4c**): yellow liquid (0.176 g, 75%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.70 (q, *J* = 7.1 Hz, 2H), 2.90 (tt, *J* = 10.7, 3.7 Hz, 1H), 2.05 – 1.95 (m, 2H), 1.82 – 1.75 (m, 2H), 1.64 – 1.58 (m, 1H), 1.47 (t, *J* = 7.1 Hz, 3H), 1.42 – 1.19 (m, 5H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 213.6, 71.2, 50.5, 32.7, 25.9, 25.4, 13.6; HRMS (ESI) m/z calcd for $C_9H_{17}OS_3$ $[M+H]^+$: 237.0442; found: 237.0443.



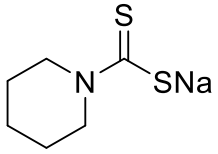
***O*-Ethyl *SS*-phenyl carbono(dithioperoxo)thioate** (**4d**): yellow liquid (0.165 g, 72%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.55 – 7.47 (m, 2H), 7.37 – 7.29 (m, 3H), 4.71 (q, *J* = 7.1 Hz, 2H), 1.45 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 211.1, 135.9, 131.5, 129.9, 129.1, 128.2, 71.5, 13.6; HRMS (ESI) m/z calcd for $C_9H_{11}OS_3$ $[M+H]^+$: 230.9972; found: 230.9972.



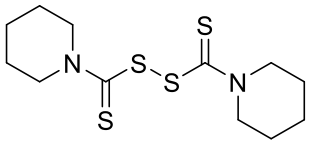
***O*-Ethyl *SS*-*p*-tolyl carbono(dithioperoxo)thioate** (**4e**): yellow liquid (0.159 g, 65%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.43 (d, *J* = 8.2 Hz, 2H), 7.16 – 7.11 (m, 2H), 4.71 (q, *J* = 7.1 Hz, 2H), 2.34 (s, 3H), 1.47 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 211.5, 138.8, 132.4, 132.3, 130.9, 129.9, 71.4, 21.1, 13.6; HRMS (ESI) m/z calcd for $C_{10}H_{13}OS_3$ $[M+H]^+$: 245.0129; found: 245.0124.



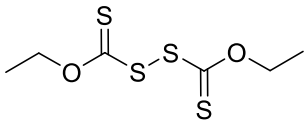
***O*-Ethyl *SSe*-methyl carbono(selenothioperoxo)thioate (4f):** yellow liquid (0.085 g, 40%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.69 (q, *J* = 7.1 Hz, 2H), 2.56 (s, 3H), 1.48 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 210.3, 71.8, 71.6, 13.6; NMR (76 MHz, $CDCl_3$) δ 389.8; HRMS (ESI) m/z calcd for $C_4H_9OS_2Se$ $[M+H]^+$: 216.9260; found: 216.9270.



Sodium piperidine-1-carbodithioate (**5**)[4]: white solid (0.183 g, 100%); **^{1}H NMR (400 MHz, DMSO)** δ 4.33 – 4.21 (m, 2H), 1.61 – 1.48 (m, 1H), 1.47 – 1.36 (m, 2H).



Piperidine-1-carbothioic dithioperoxyanhydride (**6**)[5]: light yellow solid (0.317 g, 99%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.20 (br s, 8H), 1.73 (br s, 12H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 192.6, 55.7, 52.5, 26.3, 25.5, 24.1.

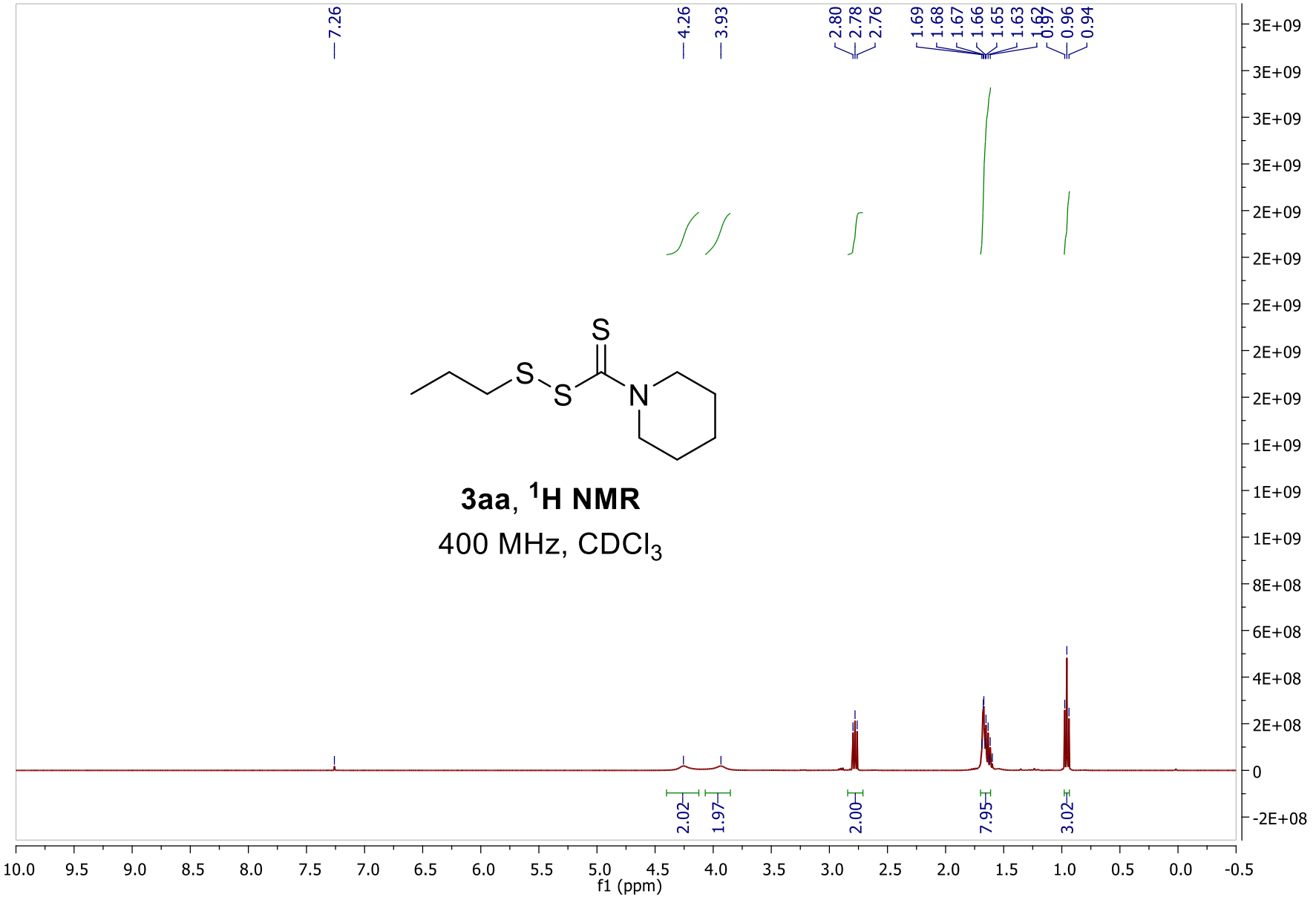


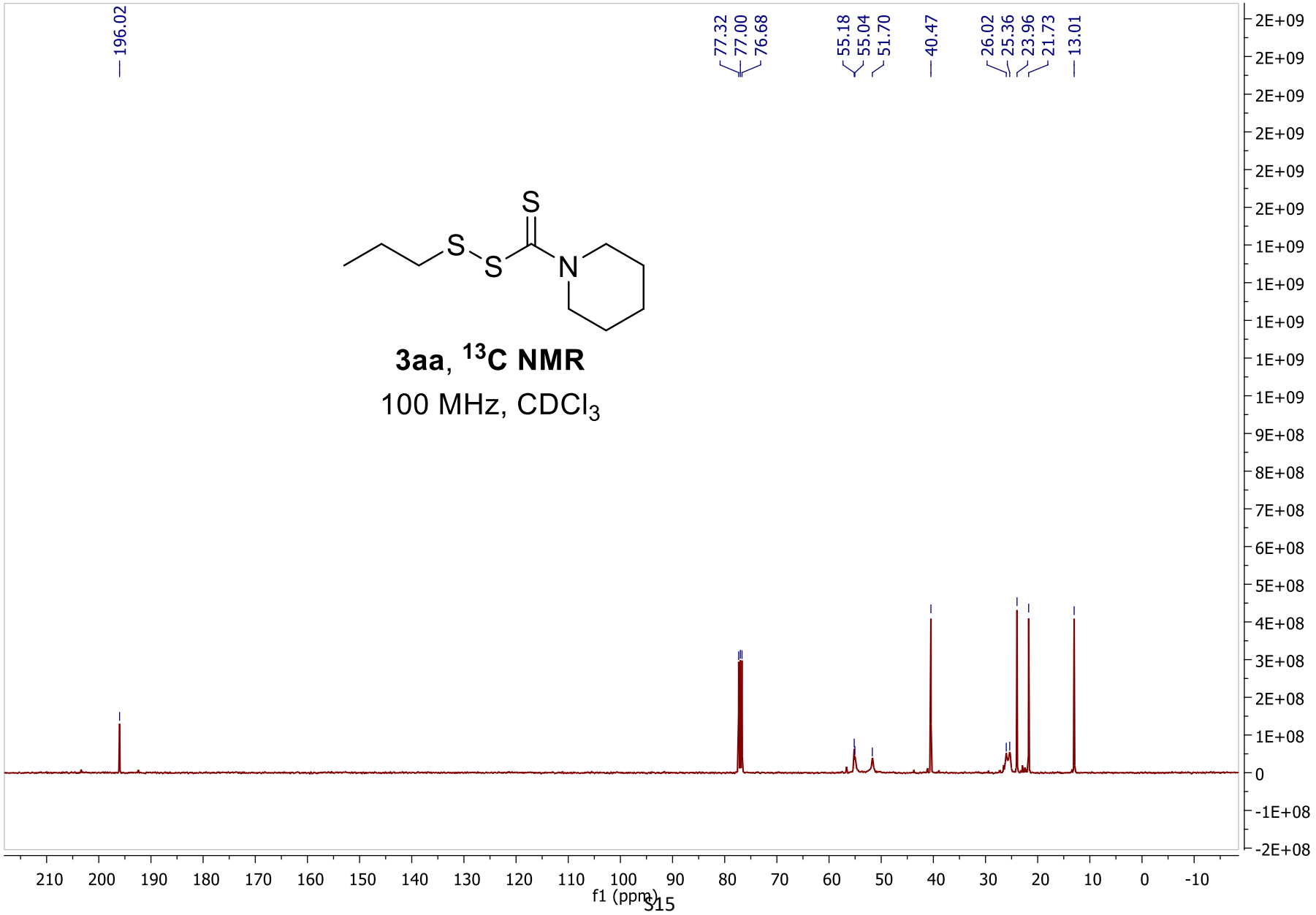
Diethyl dithiobis(thionoformate) (**7**)[6]: yellow gummy oil (0.24 g, 99%)**^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.69 (q, *J* = 7.1 Hz, 2H), 1.42 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$)** δ 207.4, 13.6.

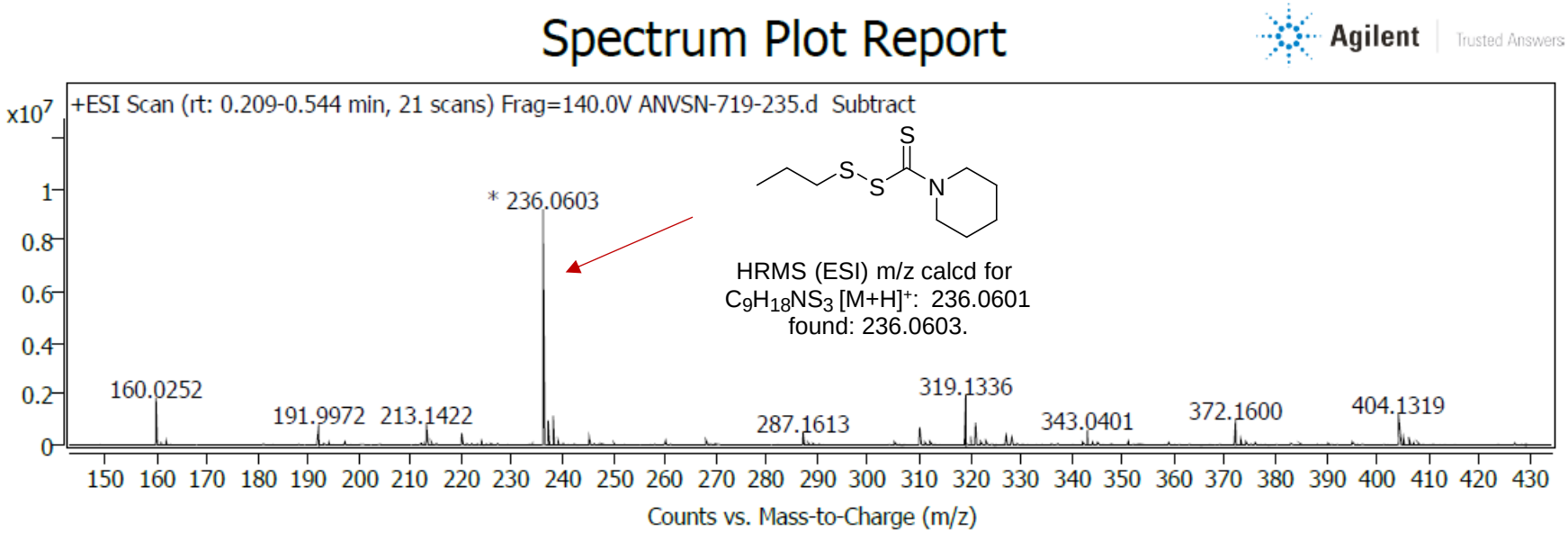
11.References

1. B. Wang and F. Q. Liu, *RSC Adv.*, 2020, **10**, 28541.
2. Q. Wang, C. H. Xu, Y. C. Wang, Y. M. Pan, W. G. Duan and H. T. Tang, *Green chem*., 2022, **24**, 7362-7367.
3. V. Cilibrasi, K. Tsang, M. Morelli, F. Solfa, H. L. Wiggins, A. T. Jones, A. D. Westwell, *Tetrahedron Letters*, 2015, **56**, 2583–2585.
4. E. Lieber and R. Orlowski, *J. Org. Chem.*,1957, **22**, 0022-3263.
5. Y. Xu, Y. Huang, R. Song, Y. Ren, X. Chen, C. Zhang, F. Mao, X. Li, J. Zhu, S. Ni, J. Wan, J. Li, *European Journal of Medicinal Chemistry*, 2020, **203**, 112500.
6. K. Hakobyan, C. S. P. McErlean and M. Mullner, *Macromolecules*, 2020, **53**, 10357−10365.

12. ^{1}H, ^{13}C, ^{19}F, ^{77}Se NMR spectra


7.26
4.26
3.93
2.80
2.78
2.76
1.69
1.68
1.67
1.66
1.65
1.63
0.97
0.96
0.94
S
S
S
N
3aa, 1H NMR
400 MHz, CDCl3
2.02
1.97
2.00
7.95
3.02
f1 (ppm)


196.02
77.32
77.00
76.68
55.18
55.04
51.70
40.47
26.02
25.36
23.96
21.73
13.01
S
S
S
N
3aa, 13C NMR
100 MHz, CDCl3
2E+09
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
-2E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Spectrum Plot Report
Agilent
Trusted Answers
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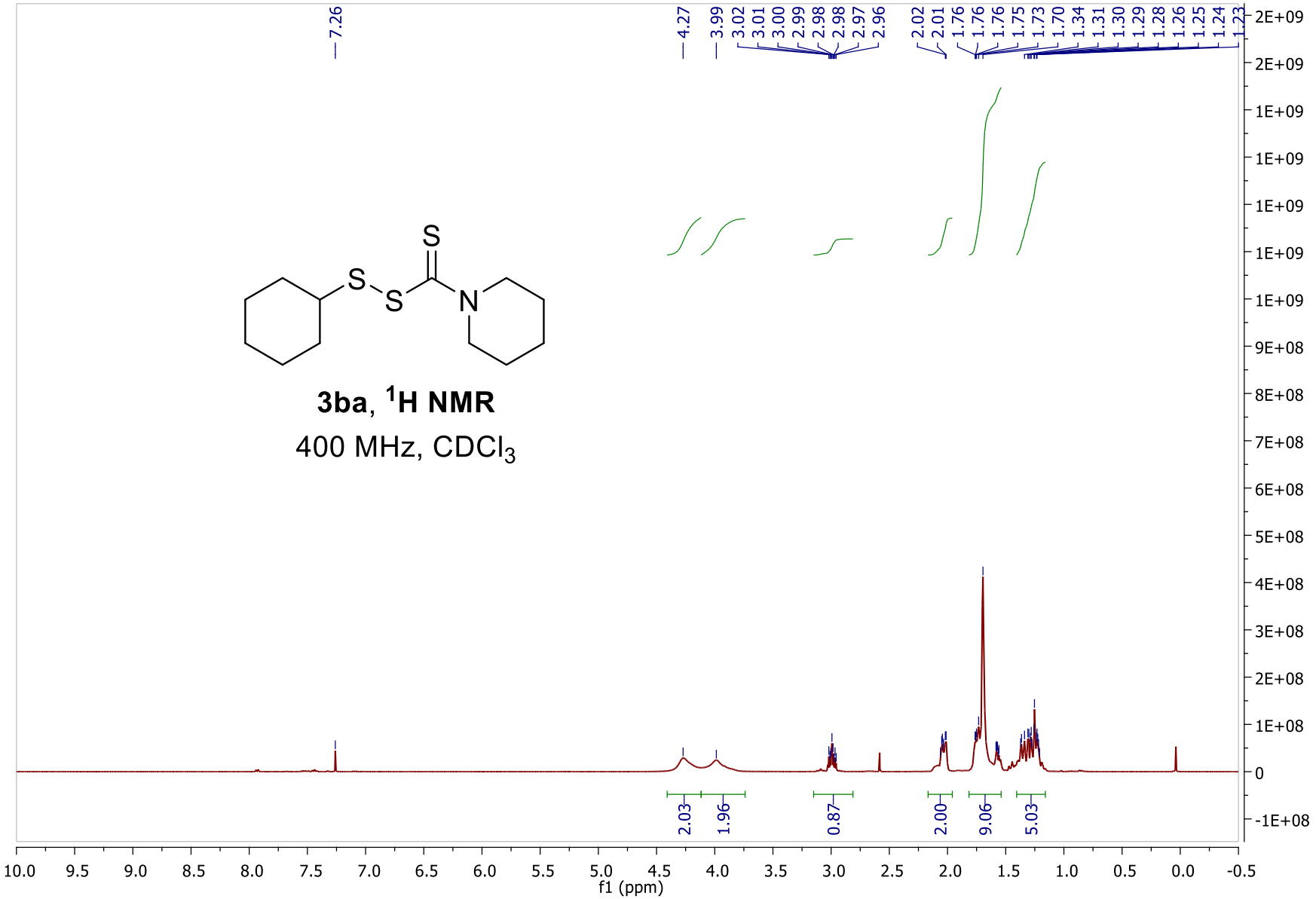
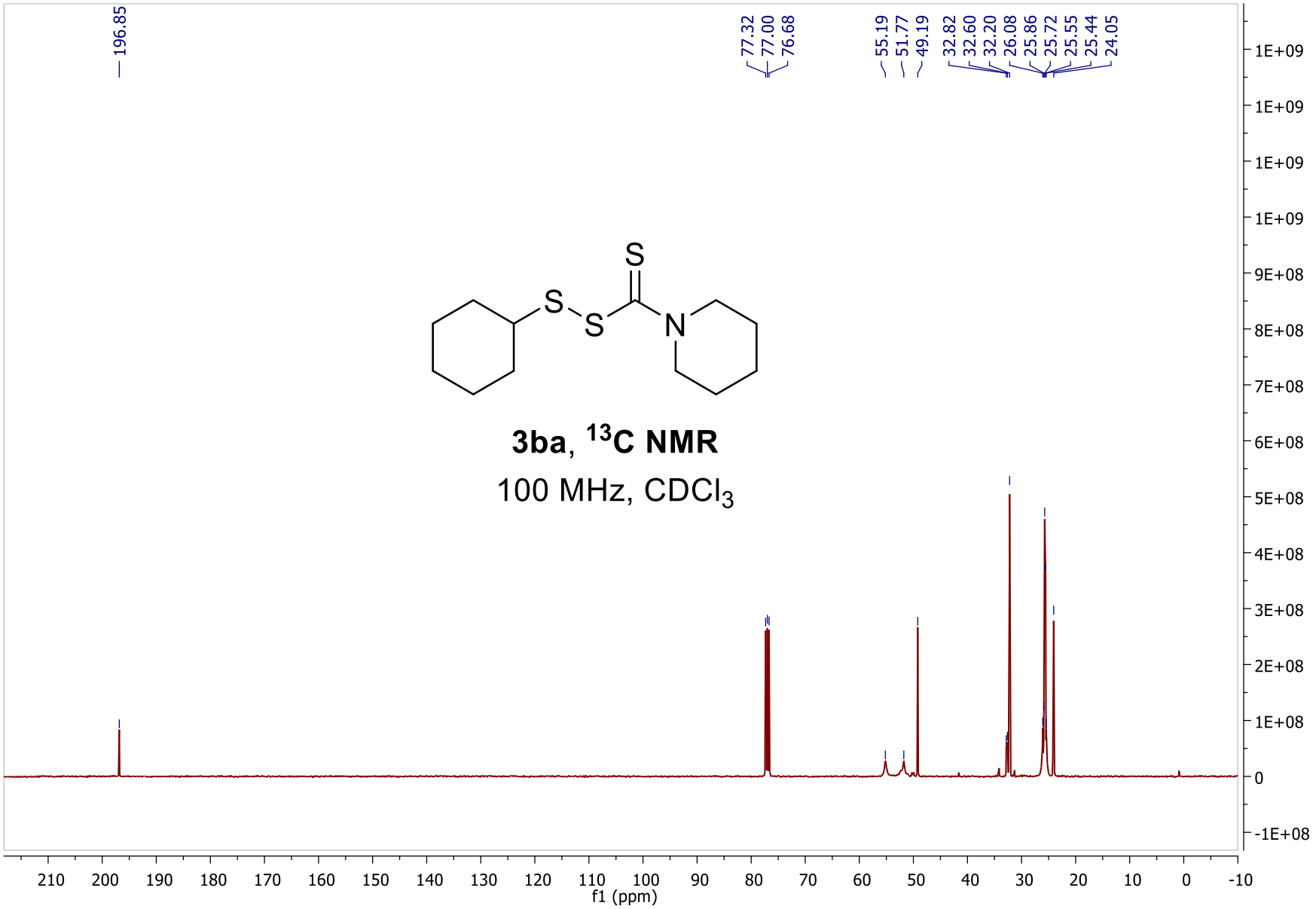
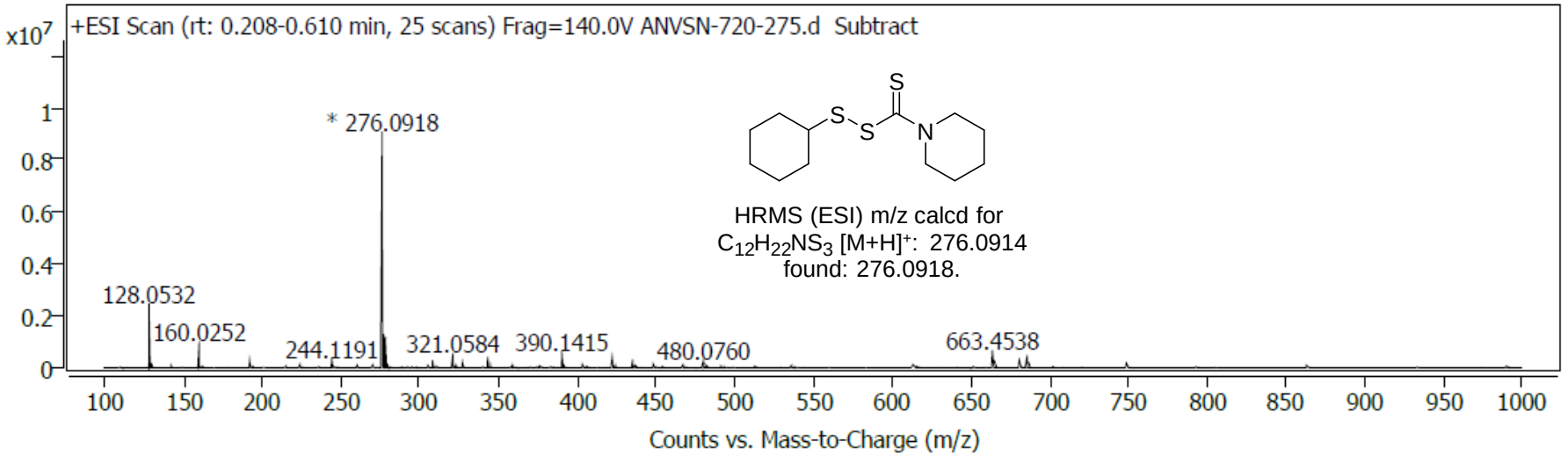
x10^7
1
0.8
0.6
0.4
0.2
0
* 236.0603
160.0252
191.9972
213.1422
287.1613
319.1336
343.0401
372.1600
404.1319
S
S
S
N
HRMS (ESI) m/z calcd for
C9H18NS3 [M+H]+: 236.0601
found: 236.0603.
150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430
Counts vs. Mass-to-Charge (m/z)

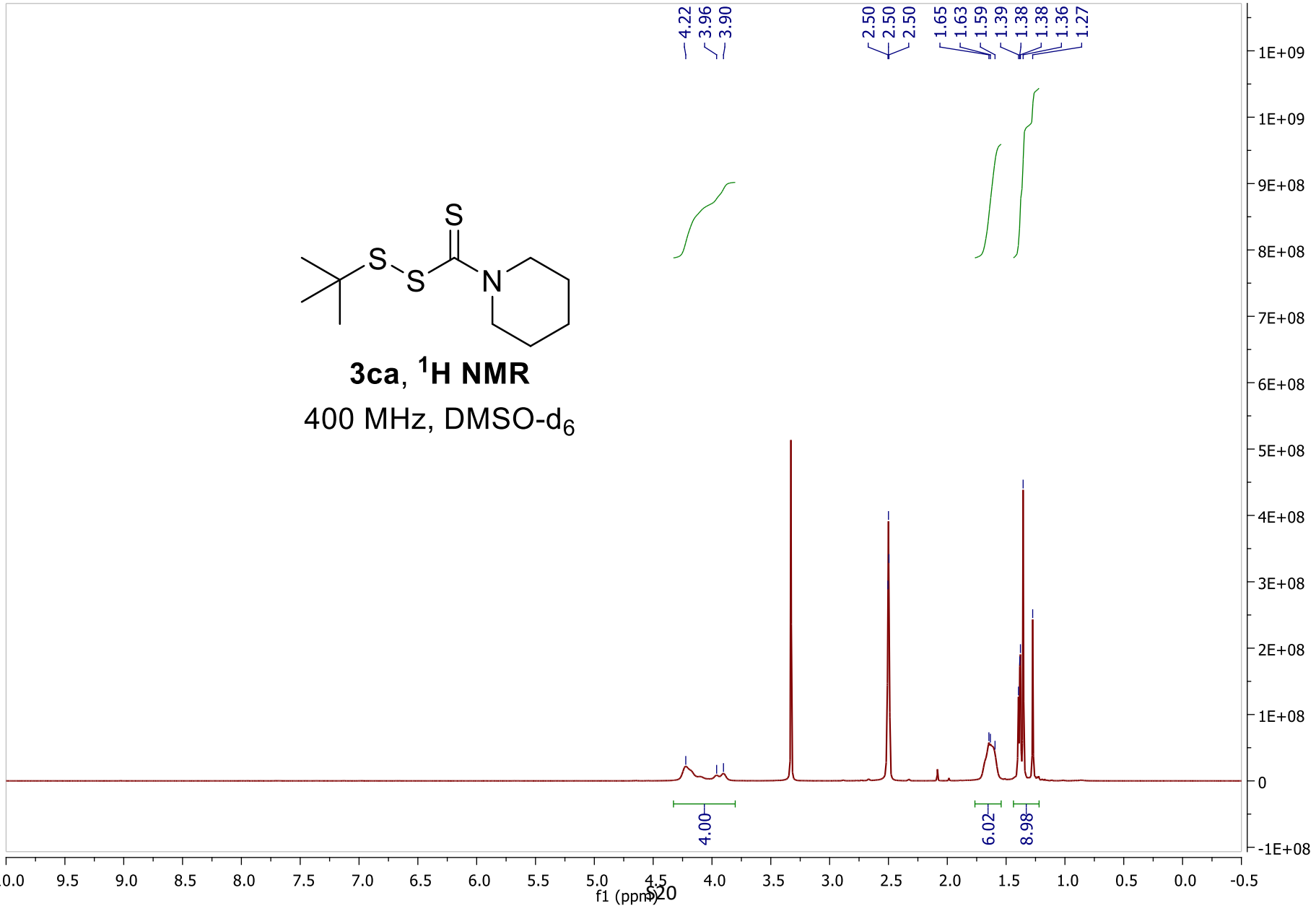

7.26
4.27
3.99
3.02
3.01
3.00
2.99
2.98
2.98
2.97
2.96
2.02
2.01
1.76
1.76
1.76
1.75
1.73
1.70
1.34
1.31
1.30
1.29
1.28
1.26
1.25
1.24
1.23
S
S
S
N
3ba, 1H NMR
400 MHz, CDCl3
2.03
1.96
0.87
2.00
9.06
5.03
f1 (ppm)

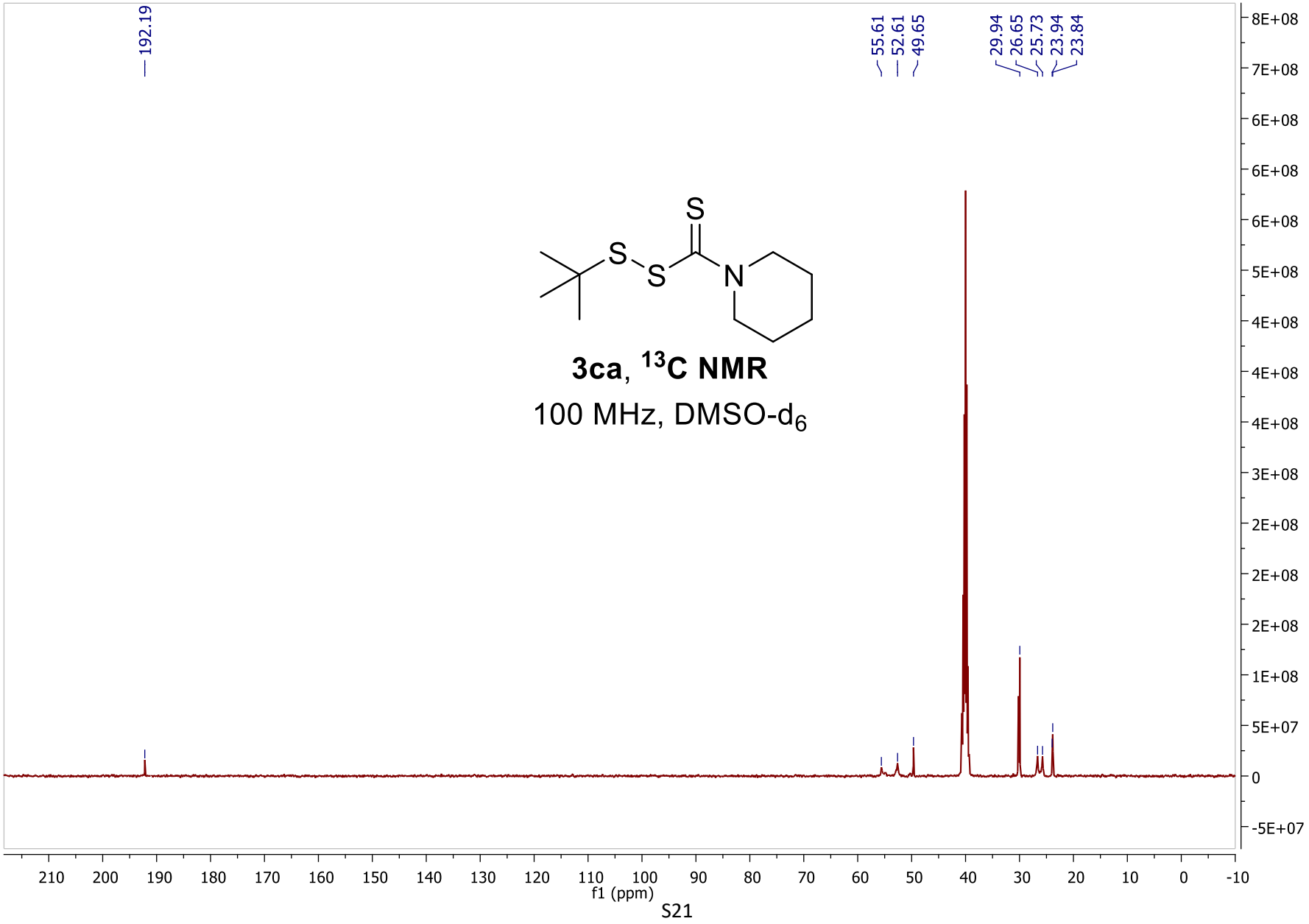

196.85
77.32
77.00
76.68
55.19
51.77
49.19
32.82
32.60
32.20
26.08
25.86
25.72
25.55
25.44
24.05
S
S
S
N
3ba, 13C NMR
100 MHz, CDCl3
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Spectrum Plot Report



HRMS (ESI) m/z calcd for $C_{12}H_{22}NS_3$ $[M+H]^+$: 276.0914
found: 276.0918.


4.22
3.96
3.90
2.50
2.50
2.50
1.65
1.63
1.59
1.39
1.38
1.38
1.36
1.27
S
S
S
N
3ca, 1H NMR
400 MHz, DMSO-d6
4.00
6.02
8.98
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


192.19
55.61
52.61
49.65
29.94
26.65
25.73
23.94
23.84
S
S
S
N
3ca, 13C NMR
100 MHz, DMSO-d6
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
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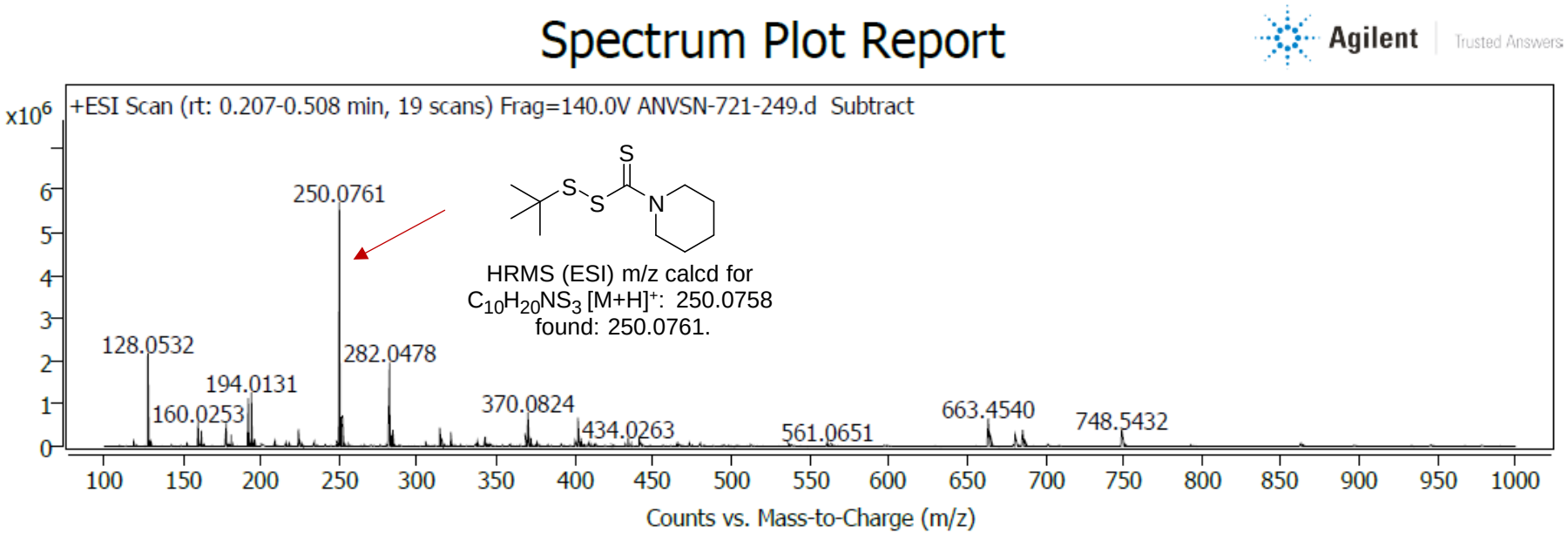
f1 (ppm)


Spectrum Plot Report
Agilent
Trusted Answers
x10^6
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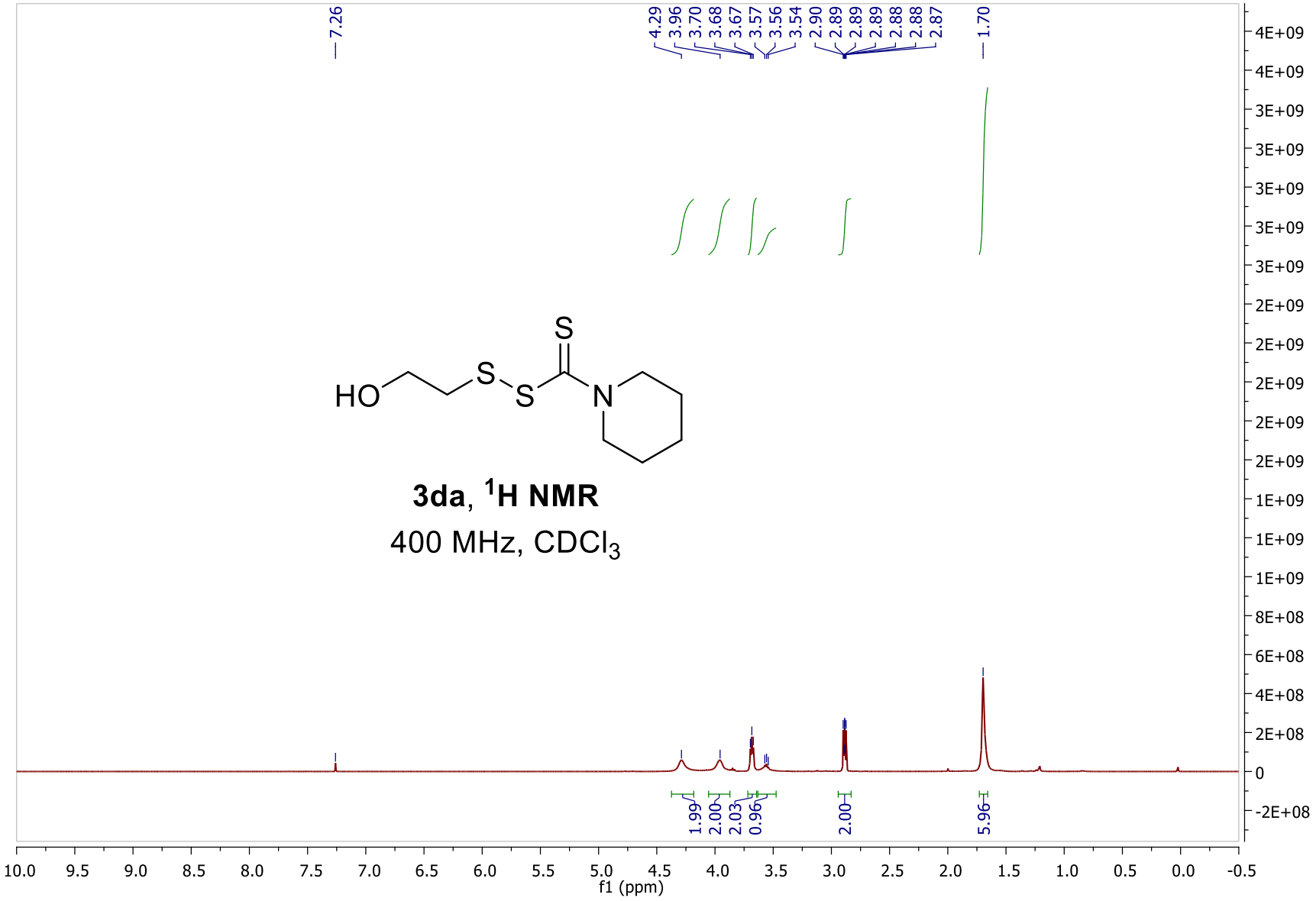
250.0761
128.0532
160.0253
194.0131
282.0478
370.0824
434.0263
561.0651
663.4540
748.5432
HRMS (ESI) m/z calcd for
C10H20NS3 [M+H]+: 250.0758
found: 250.0761.
Counts vs. Mass-to-Charge (m/z)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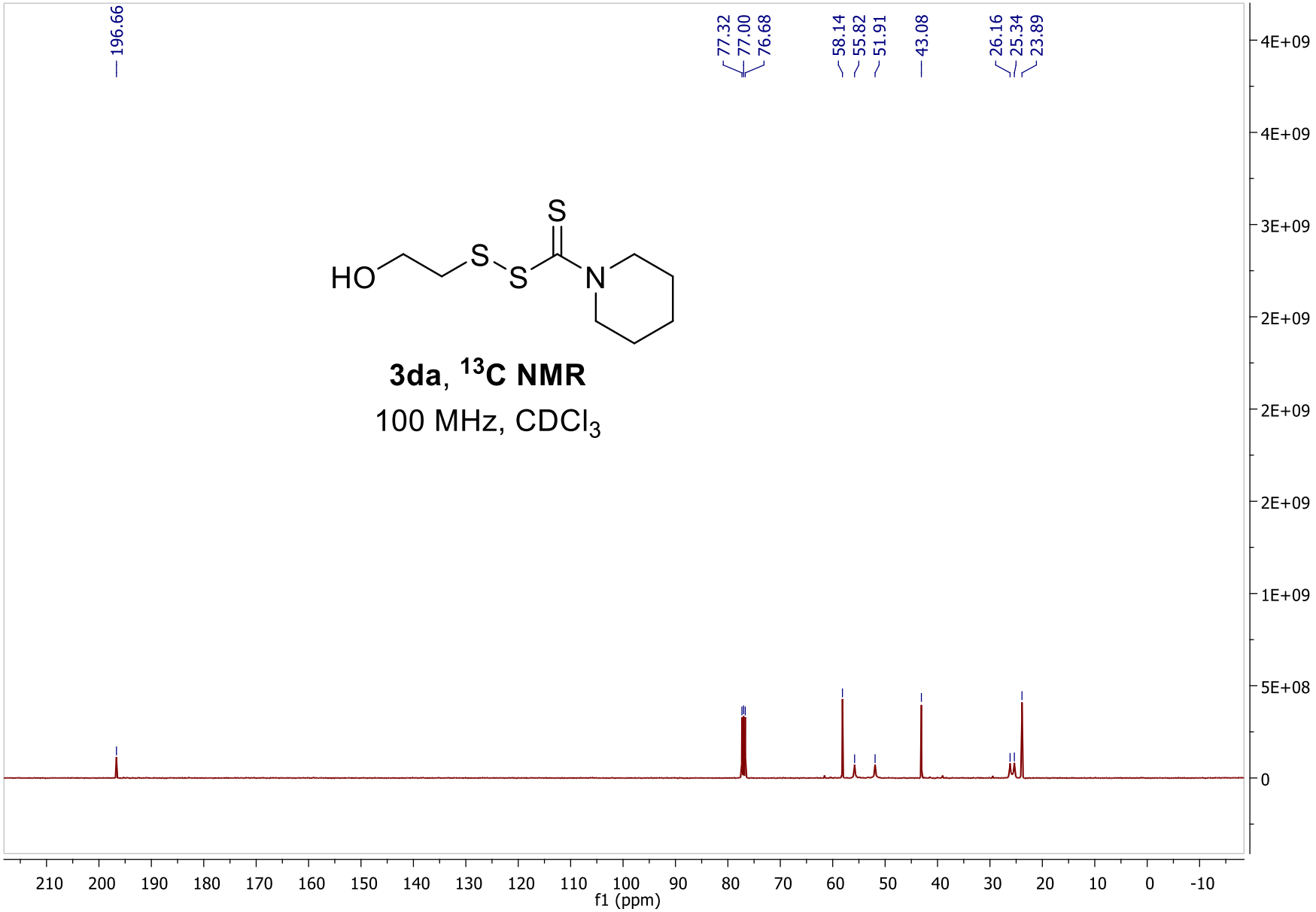
4.29
3.96
3.70
3.68
3.67
3.57
3.56
3.54
2.90
2.89
2.89
2.89
2.88
2.88
2.87
1.70
HO
S
S
S
N
3da, 1H NMR
400 MHz, CDCl3
1.99
2.00
2.03
0.96
2.00
5.96
f1 (ppm)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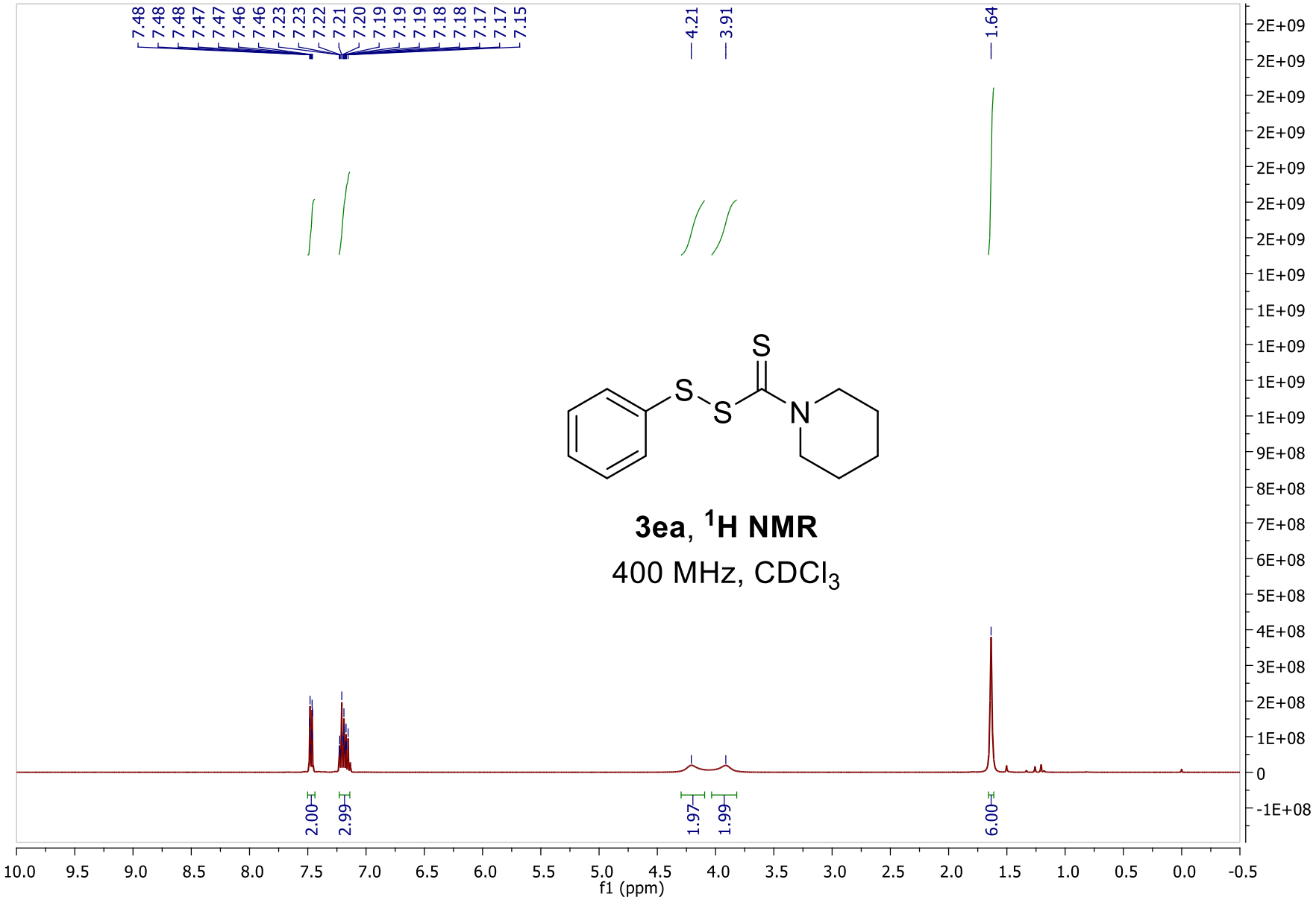
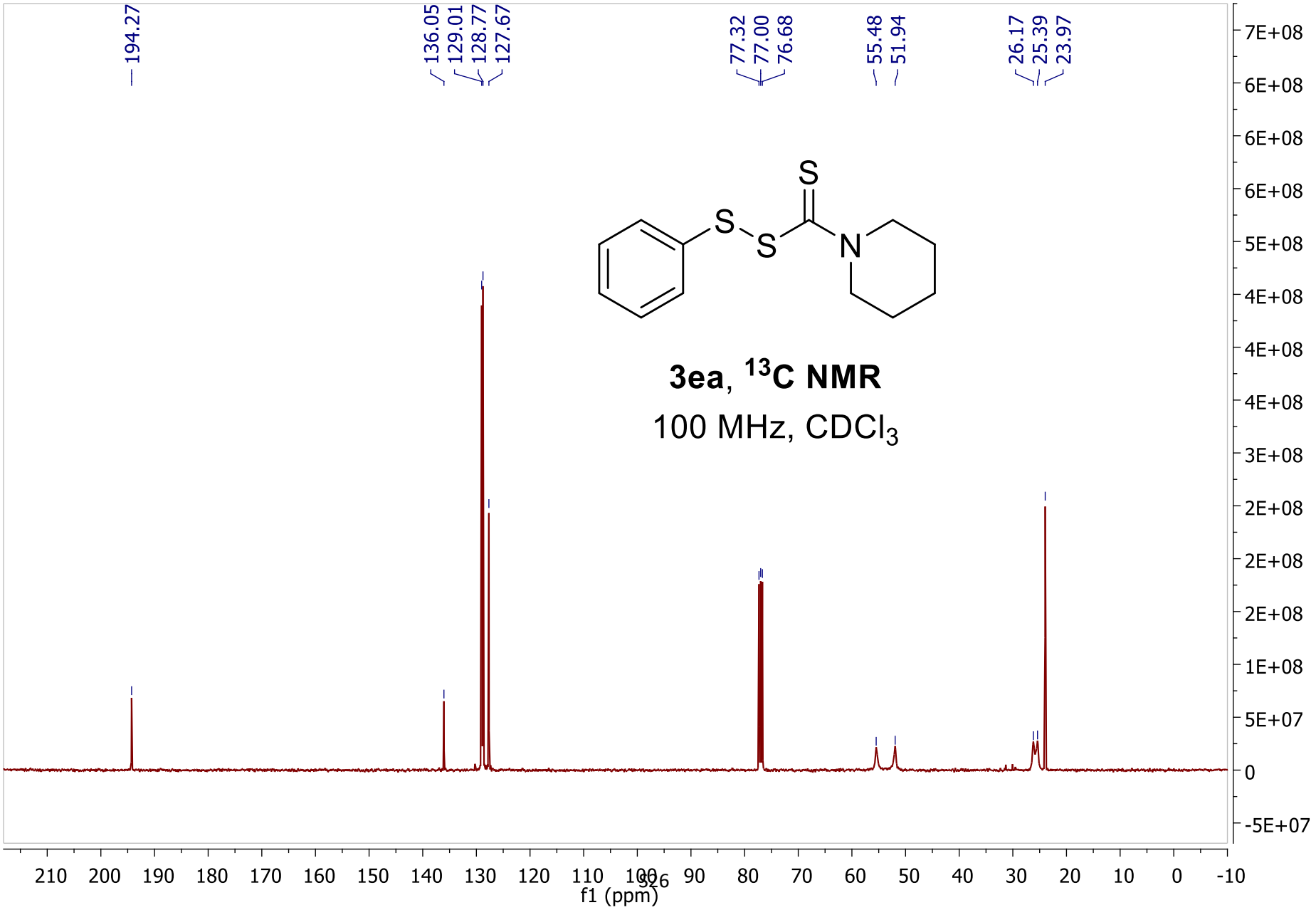
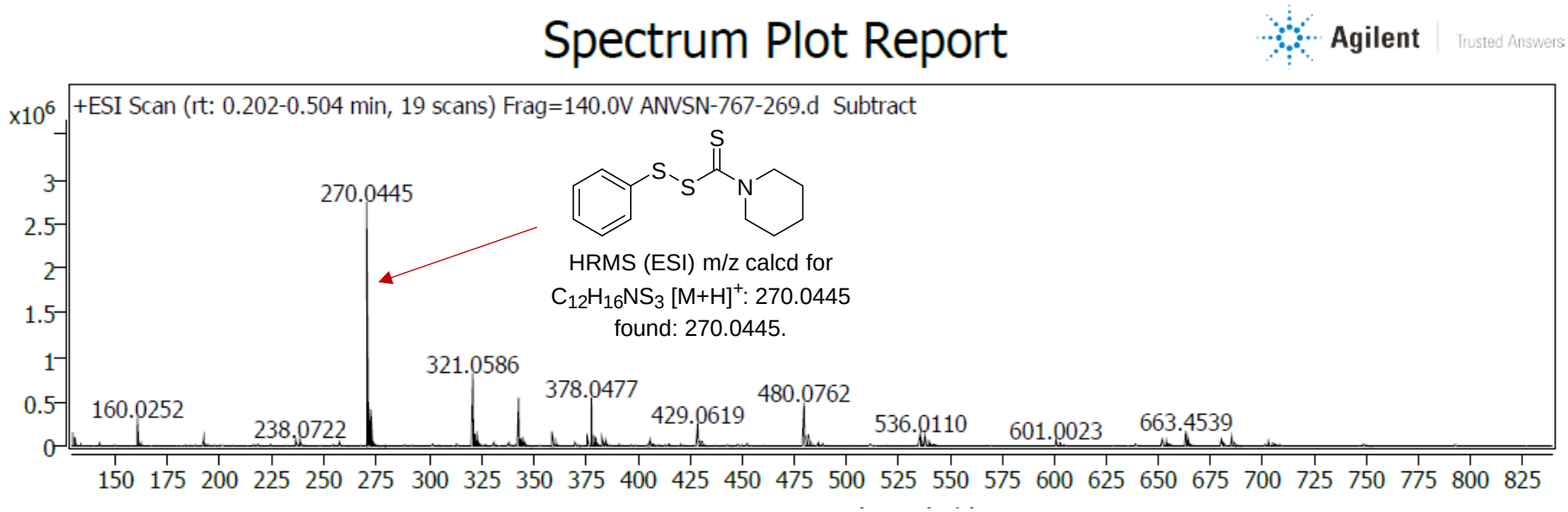
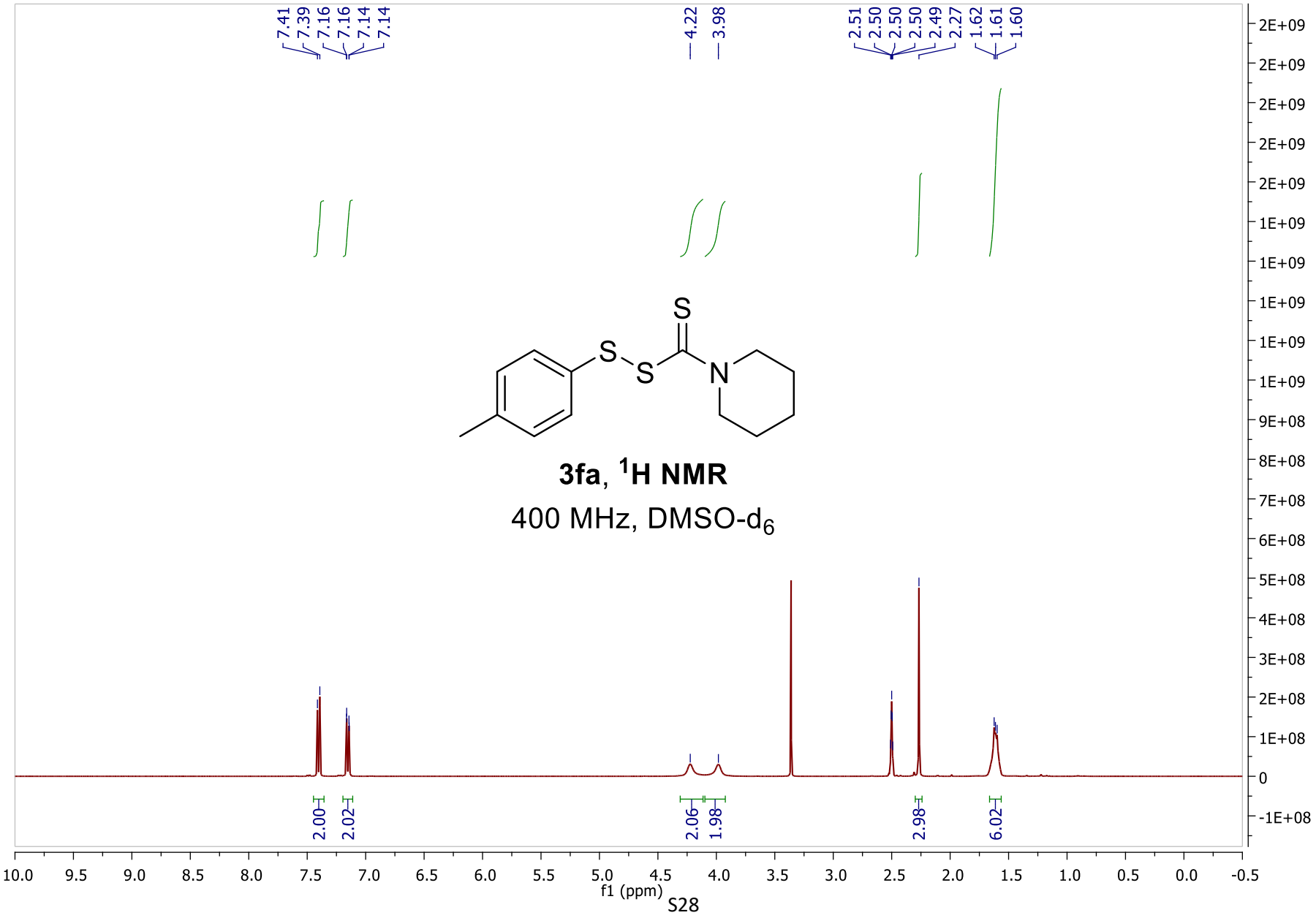
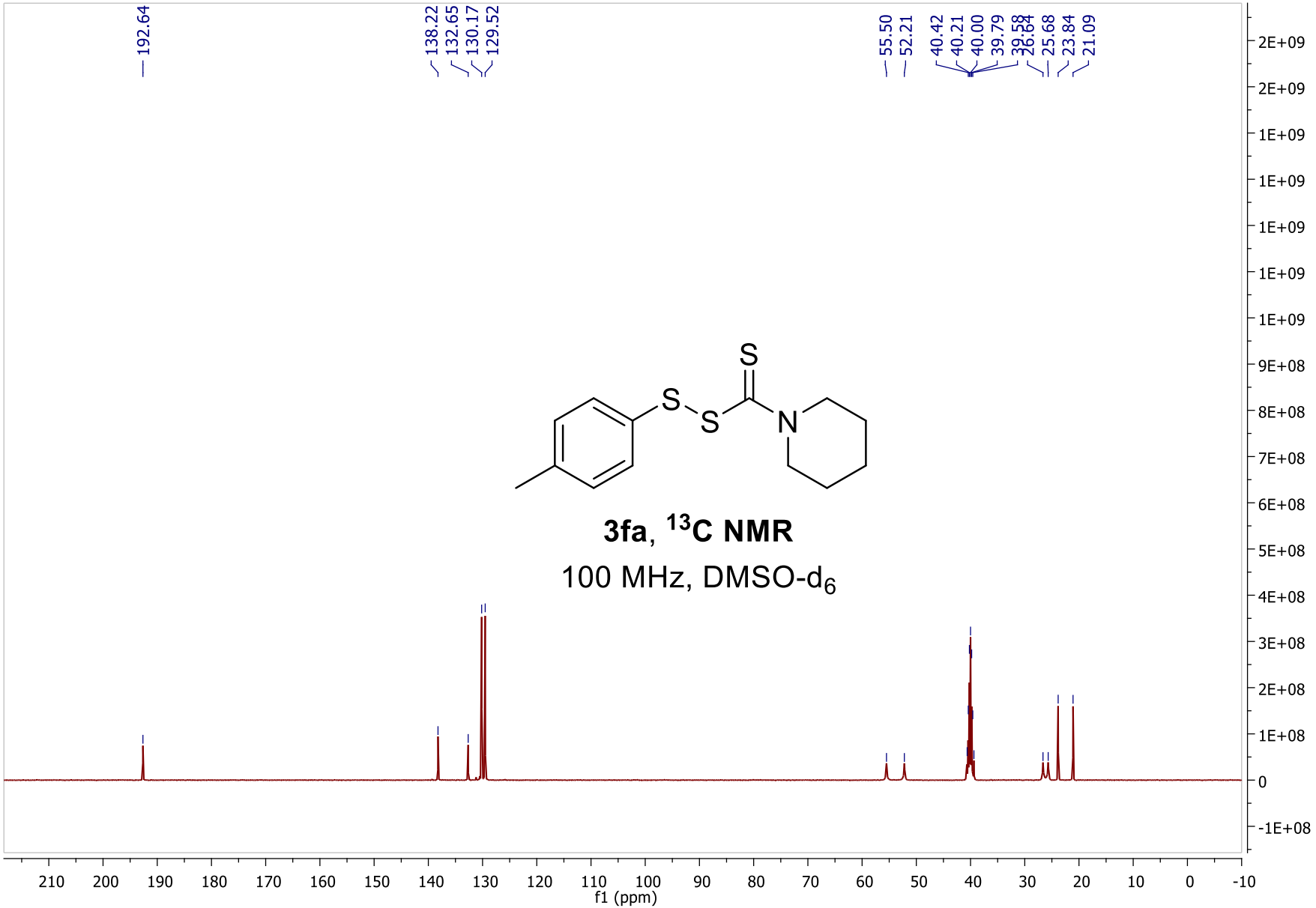
77.32
77.00
76.68
58.14
55.82
51.91
43.08
26.16
25.34
23.89
HO
S
S
S
N
3da, 13C NMR
100 MHz, CDCl3
4E+09
4E+09
3E+09
2E+09
2E+09
2E+09
1E+09
5E+08
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

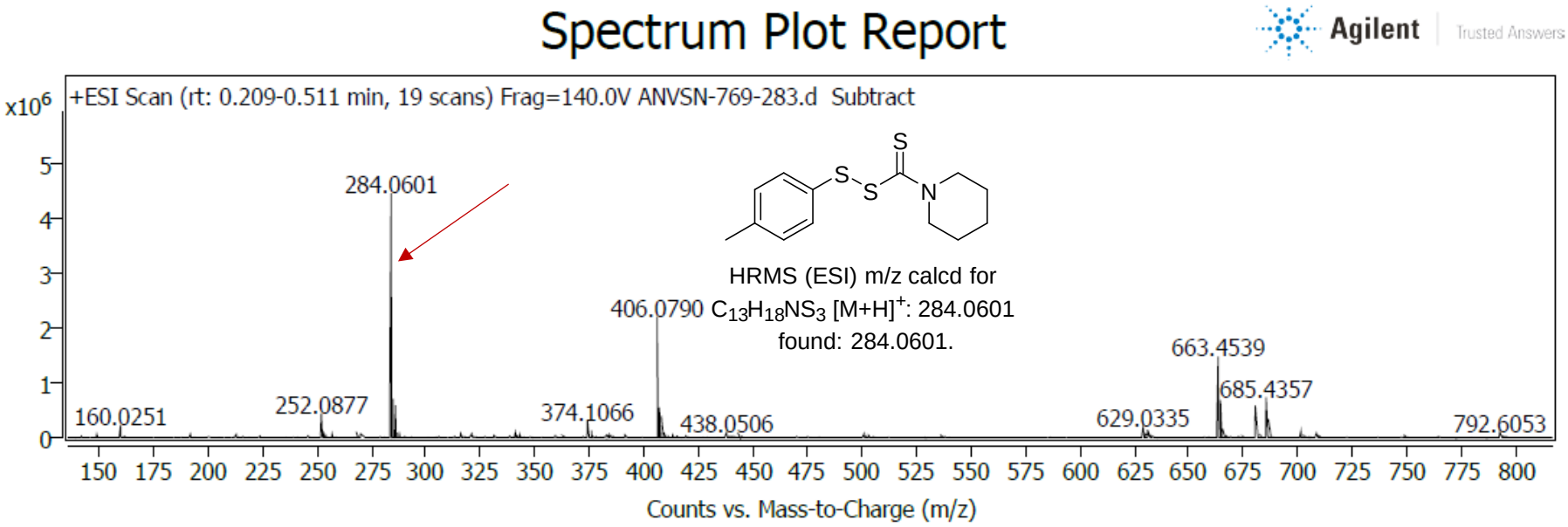

7.48
7.48
7.48
7.47
7.47
7.46
7.46
7.23
7.23
7.22
7.21
7.20
7.19
7.19
7.19
7.18
7.18
7.17
7.17
7.15
4.21
3.91
1.64
S
S
S
N
3ea, 1H NMR
400 MHz, CDCl3
2.00
2.99
1.97
1.99
6.00
f1 (ppm)

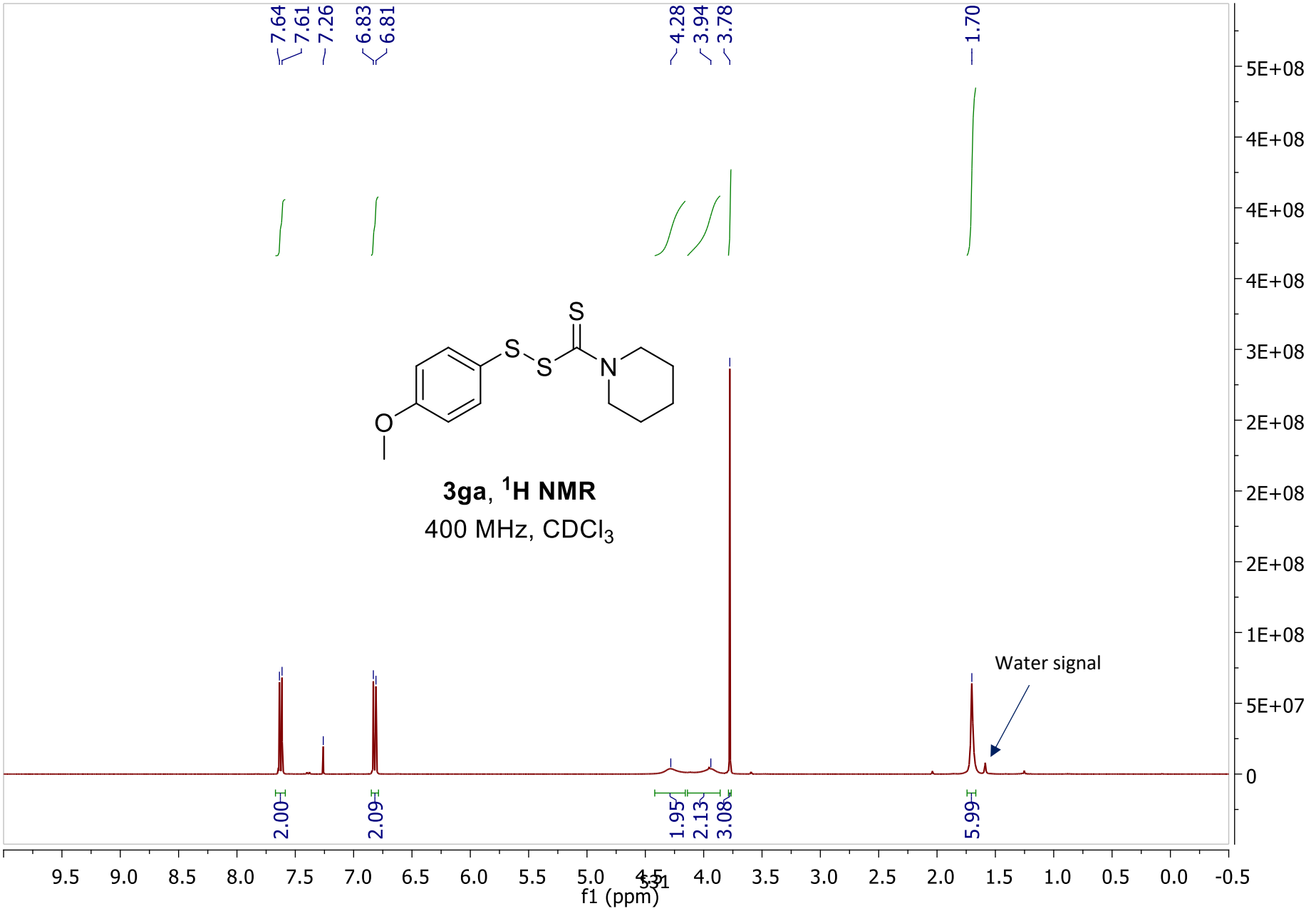

194.27
136.05
129.01
128.77
127.67
77.32
77.00
76.68
55.48
51.94
26.17
25.39
23.97
S
S
S
N
3ea, 13C NMR
100 MHz, CDCl3
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

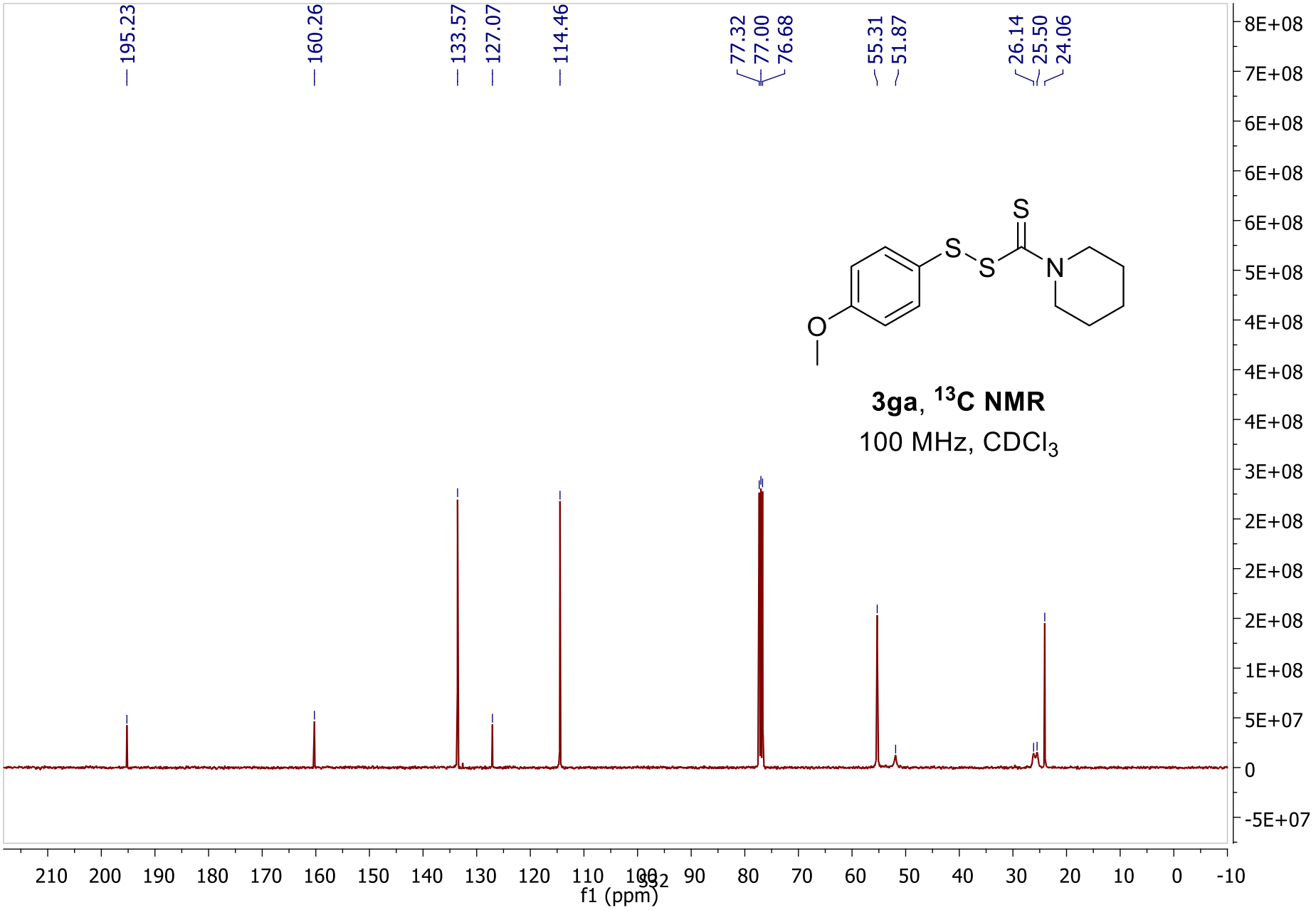

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.202-0.504 min, 19 scans) Frag=140.0V ANVSN-767-269.d Subtract
x10^6
3
2.5
2
1.5
1
0.5
0
270.0445
321.0586
378.0477
480.0762
160.0252
238.0722
429.0619
536.0110
601.0023
663.4539
150 175 200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775 800 825
S
S
S
N
HRMS (ESI) m/z calcd for
C12H16NS3 [M+H]+: 270.0445
found: 270.0445.


7.41
7.39
7.16
7.16
7.14
7.14
4.22
3.98
2.51
2.50
2.50
2.50
2.49
2.27
1.62
1.61
1.60
S
S
S
N
3fa, 1H NMR
400 MHz, DMSO-d6
2.00
2.02
2.06
1.98
2.98
6.02
f1 (ppm)

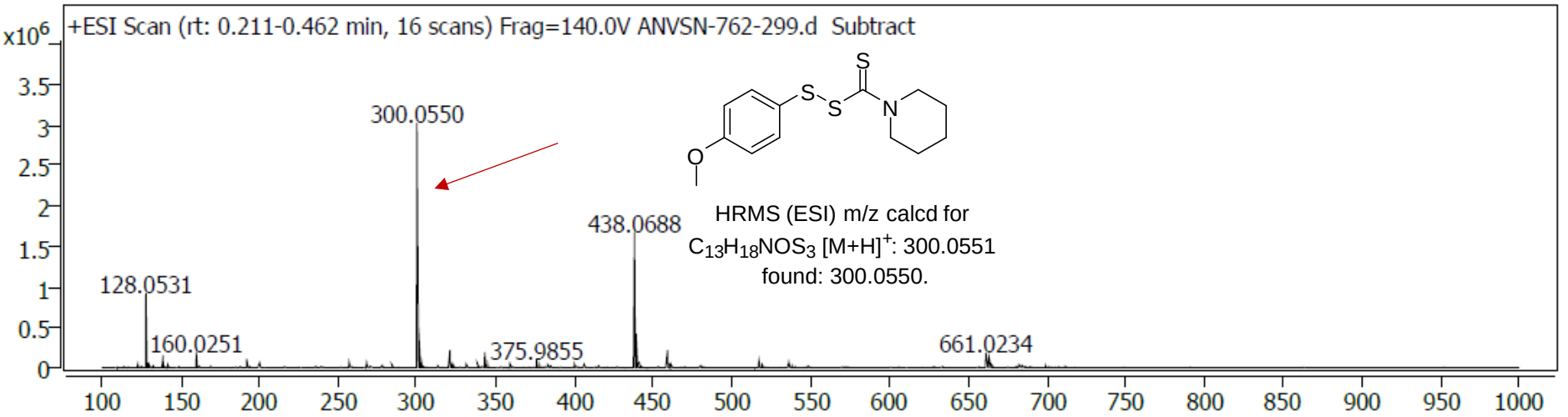

192.64
138.22
132.65
130.17
129.52
55.50
52.21
40.42
40.21
40.00
39.79
39.58
39.64
25.68
23.84
21.09
S
S
S
N
3fa, 13C NMR
100 MHz, DMSO-d6
f1 (ppm)


Spectrum Plot Report
Agilent
Trusted Answers
x10^6
+ESI Scan (rt: 0.209-0.511 min, 19 scans) Frag=140.0V ANVSN-769-283.d Subtract
284.0601
406.0790
663.4539
685.4357
160.0251
252.0877
374.1066
438.0506
629.0335
792.6053
HRMS (ESI) m/z calcd for
C13H18NS3 [M+H]+: 284.0601
found: 284.0601.
Counts vs. Mass-to-Charge (m/z)


7.64
7.61
7.26
6.83
6.81
4.28
3.94
3.78
1.70
S
S
S
N
O
3ga, ¹H NMR
400 MHz, CDCl3
Water signal
2.00
2.09
1.95
2.13
3.08
5.99
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


195.23
160.26
133.57
127.07
114.46
77.32
77.00
76.68
55.31
51.87
26.14
25.50
24.06
S
S
S
N
O
3ga, 13C NMR
100 MHz, CDCl3
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Spectrum Plot Report



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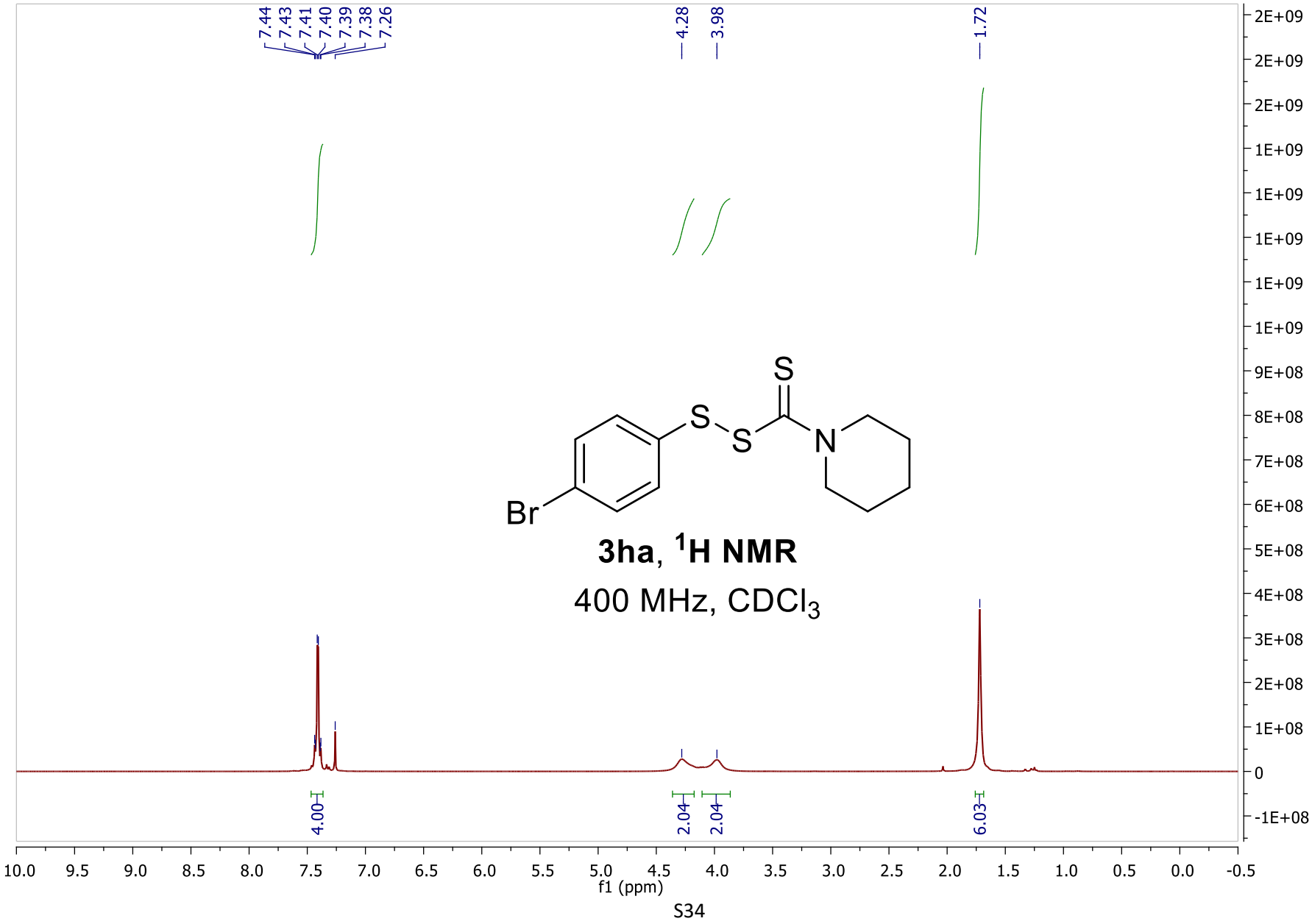
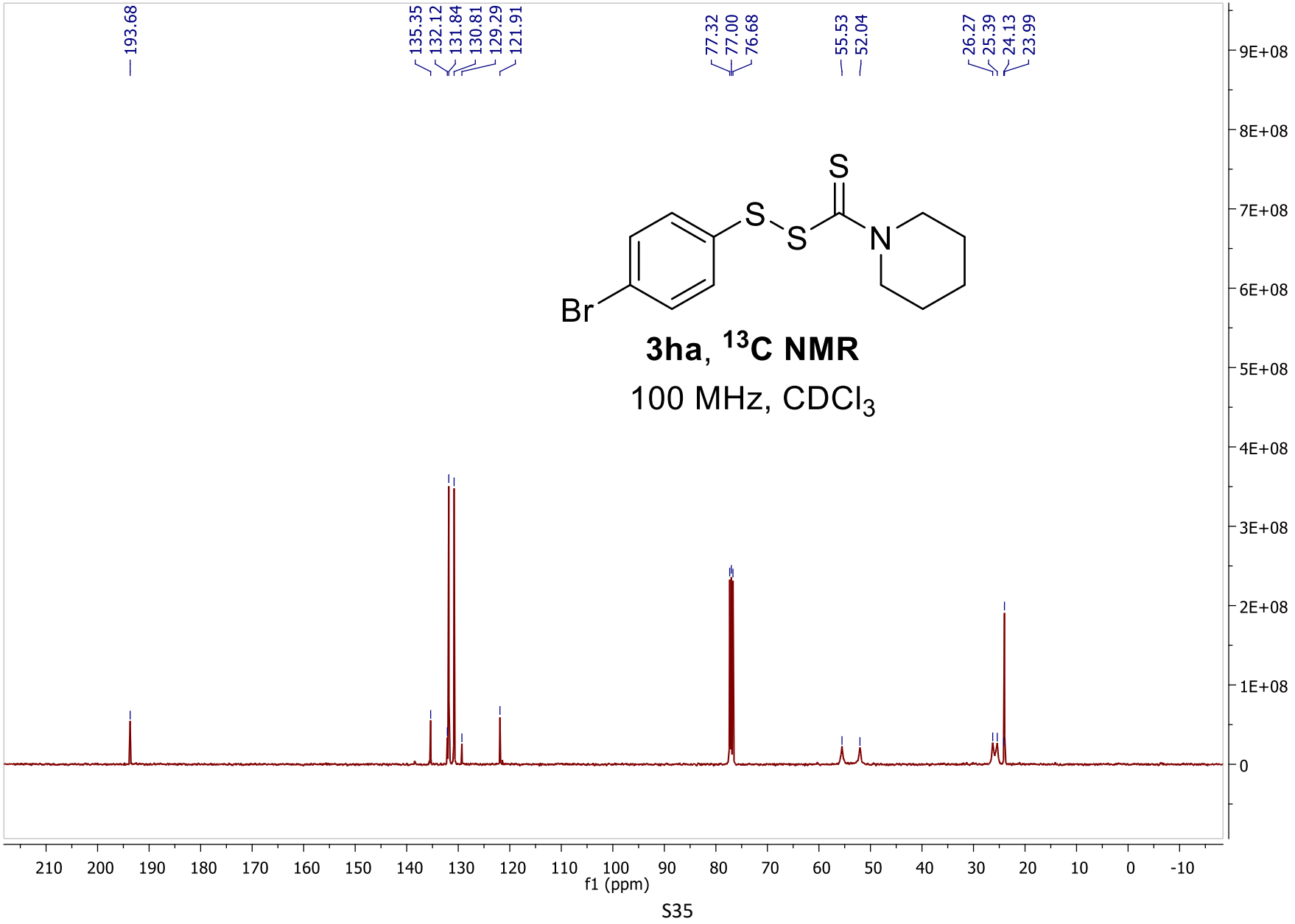
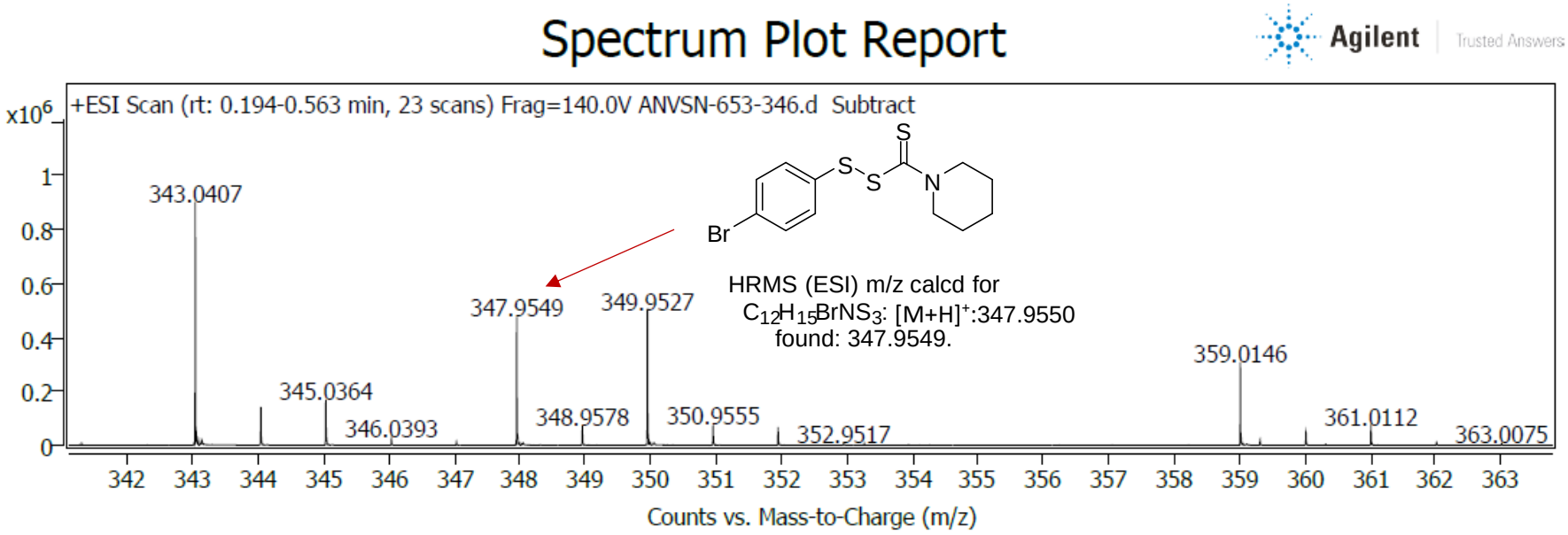
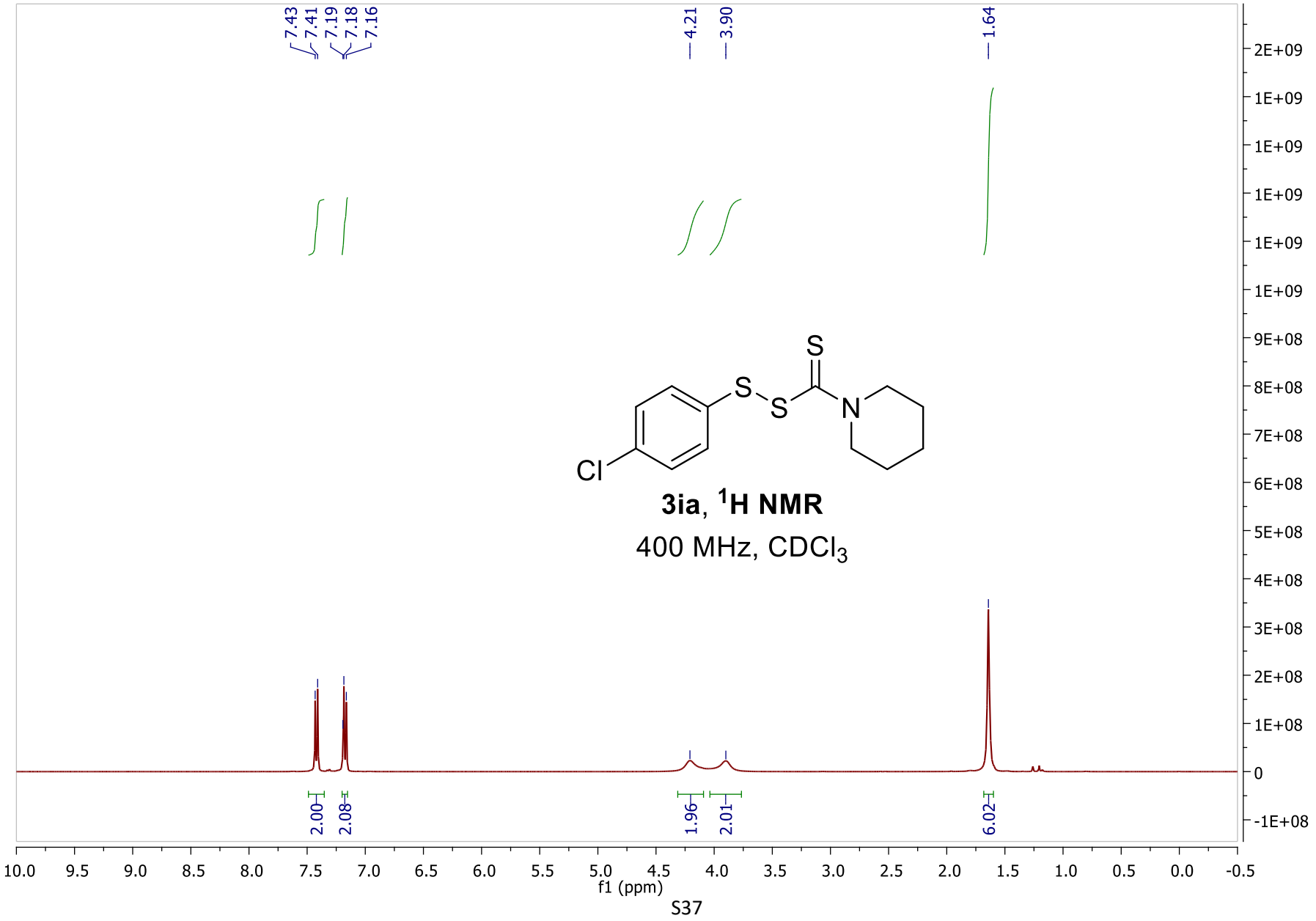
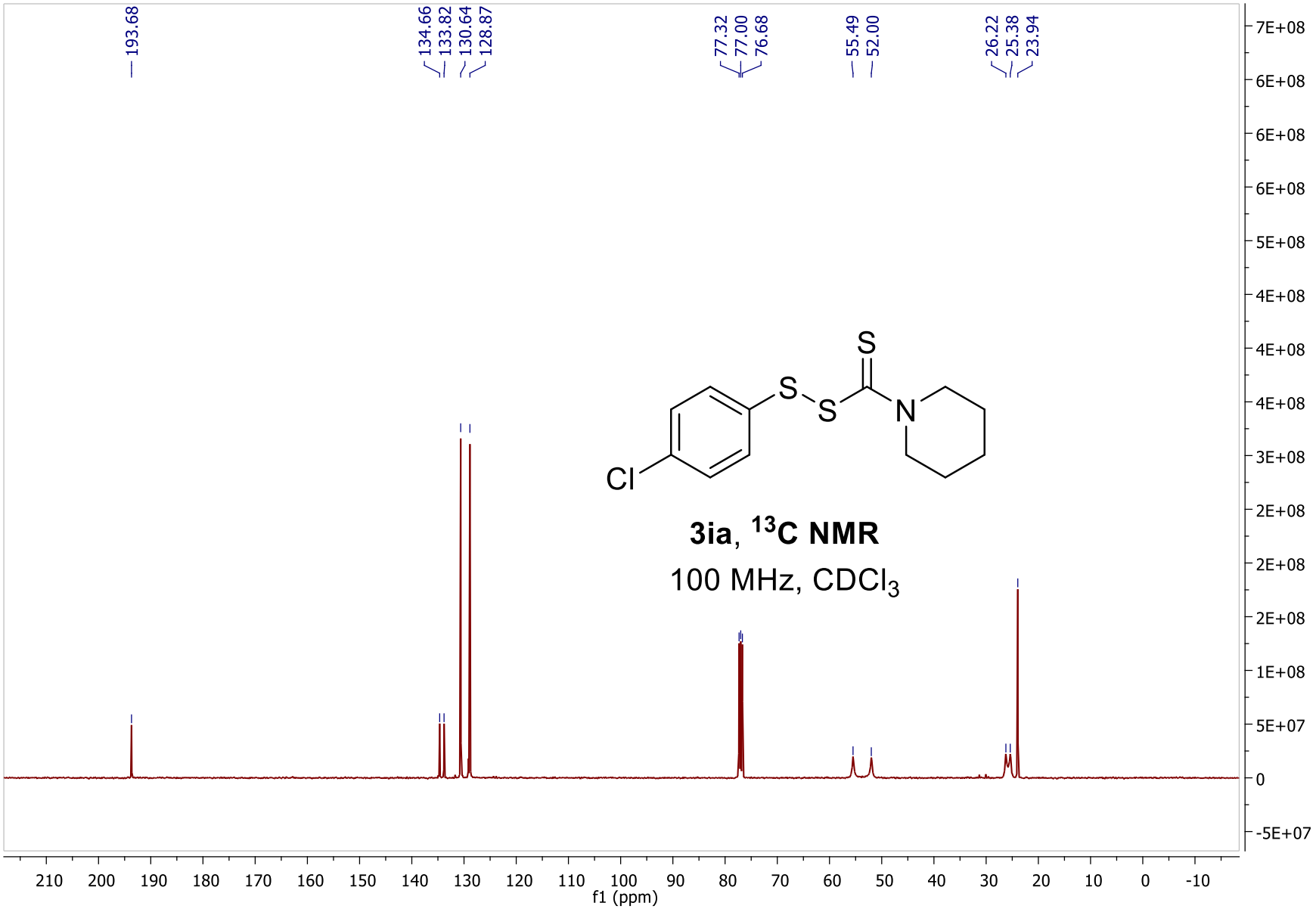
found: 300.0550.


7.44
7.43
7.41
7.40
7.39
7.38
7.26
4.28
3.98
1.72
S
S
S
N
Br
3ha, 1H NMR
400 MHz, CDCl3
4.00
2.04
2.04
6.03
f1 (ppm)

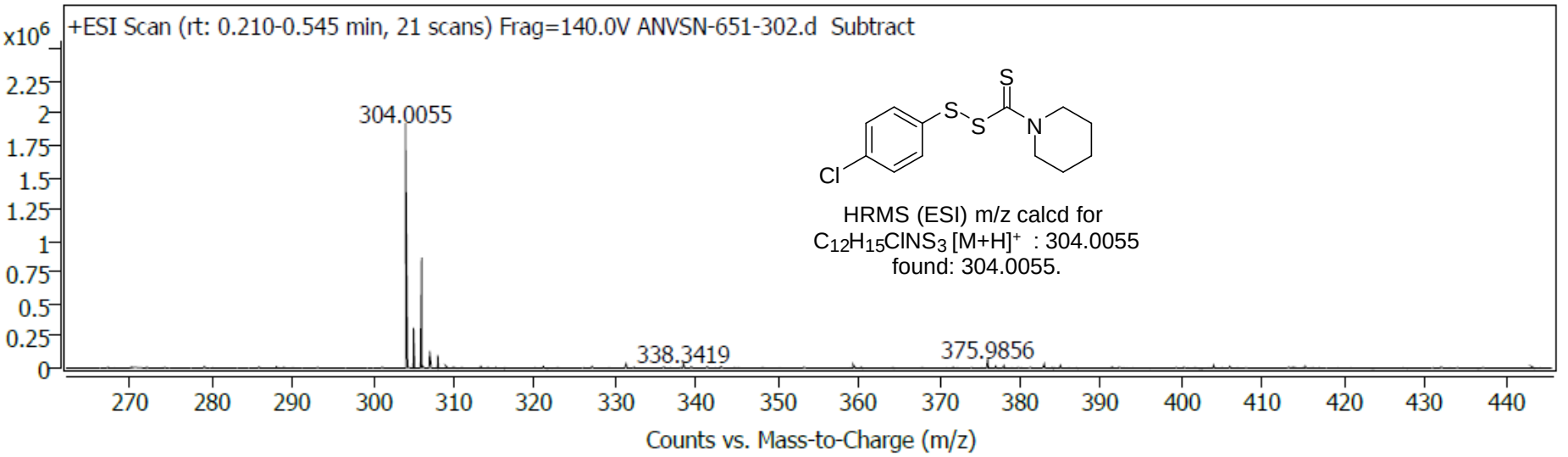

193.68
135.35
132.12
131.84
130.81
129.29
121.91
77.32
77.00
76.68
55.53
52.04
26.27
25.39
24.13
23.99
S
S
S
N
Br
3ha, 13C NMR
100 MHz, CDCl3
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.194-0.563 min, 23 scans) Frag=140.0V ANVSN-653-346.d Subtract
x10^6
1
0.8
0.6
0.4
0.2
0
343.0407
345.0364
346.0393
347.9549
348.9578
349.9527
350.9555
352.9517
359.0146
361.0112
363.0075
S
S
S
N
Br
HRMS (ESI) m/z calcd for
C12H15BrNS3: [M+H]+:347.9550
found: 347.9549.
342 343 344 345 346 347 348 349 350 351 352 353 354 355 356 357 358 359 360 361 362 363
Counts vs. Mass-to-Charge (m/z)

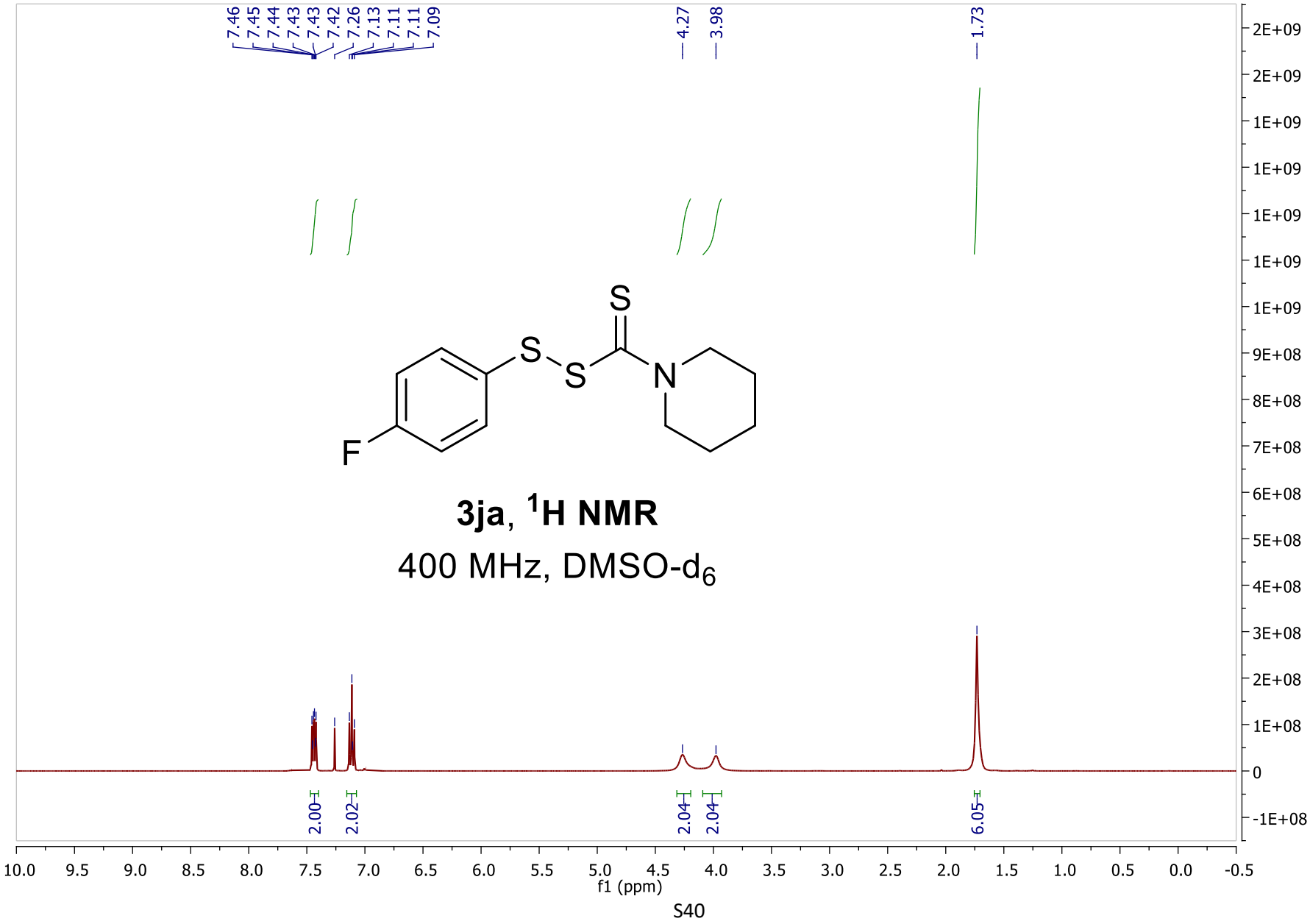

7.43
7.41
7.19
7.18
7.16
4.21
3.90
1.64
S
S
S
N
Cl
3ia, 1H NMR
400 MHz, CDCl3
2.00
2.08
1.96
2.01
6.02
f1 (ppm)

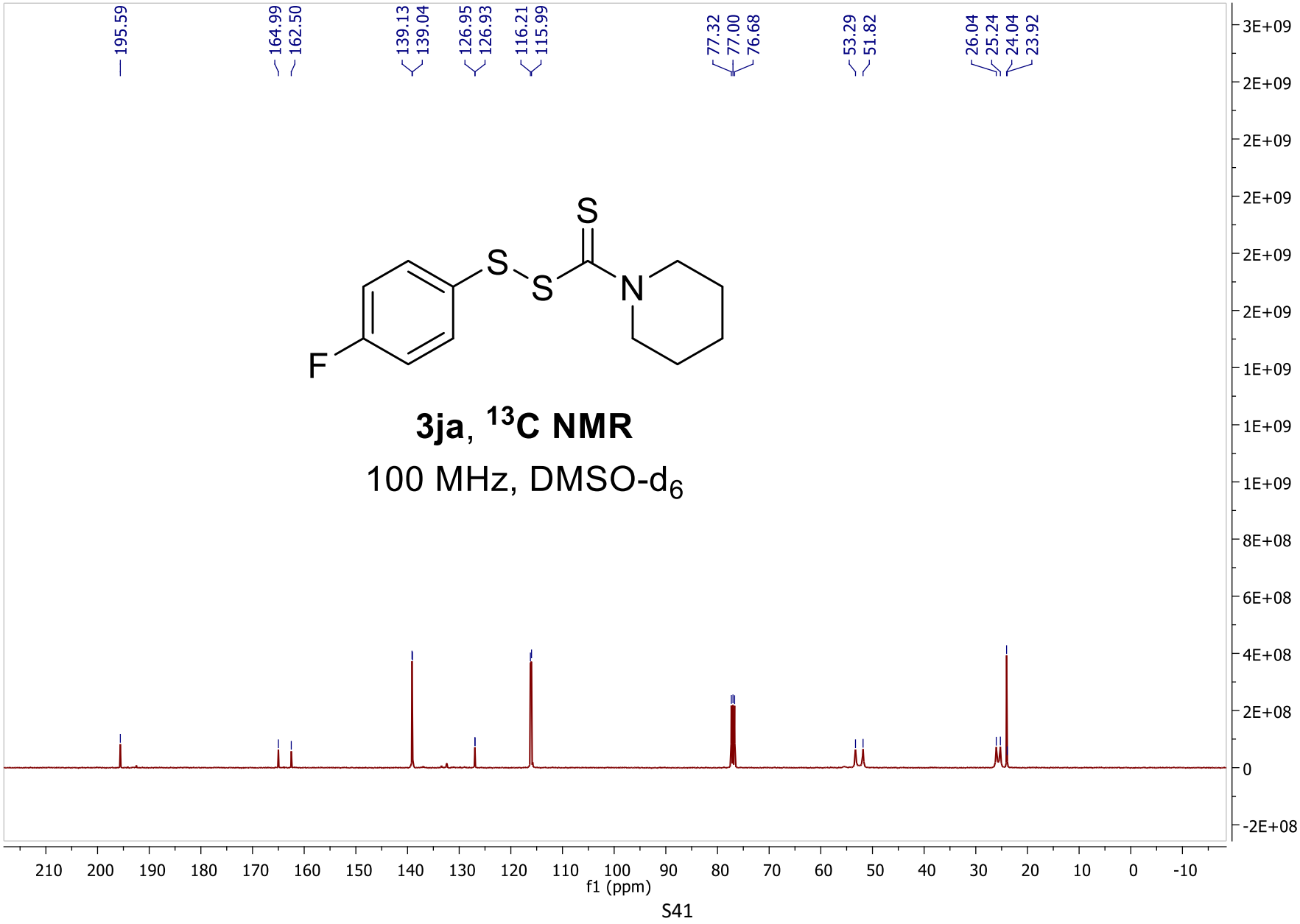

193.68
134.66
133.82
130.64
128.87
77.32
77.00
76.68
55.49
52.00
26.22
25.38
23.94
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
S
S
S
N
Cl
3ia, 13C NMR
100 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Spectrum Plot Report



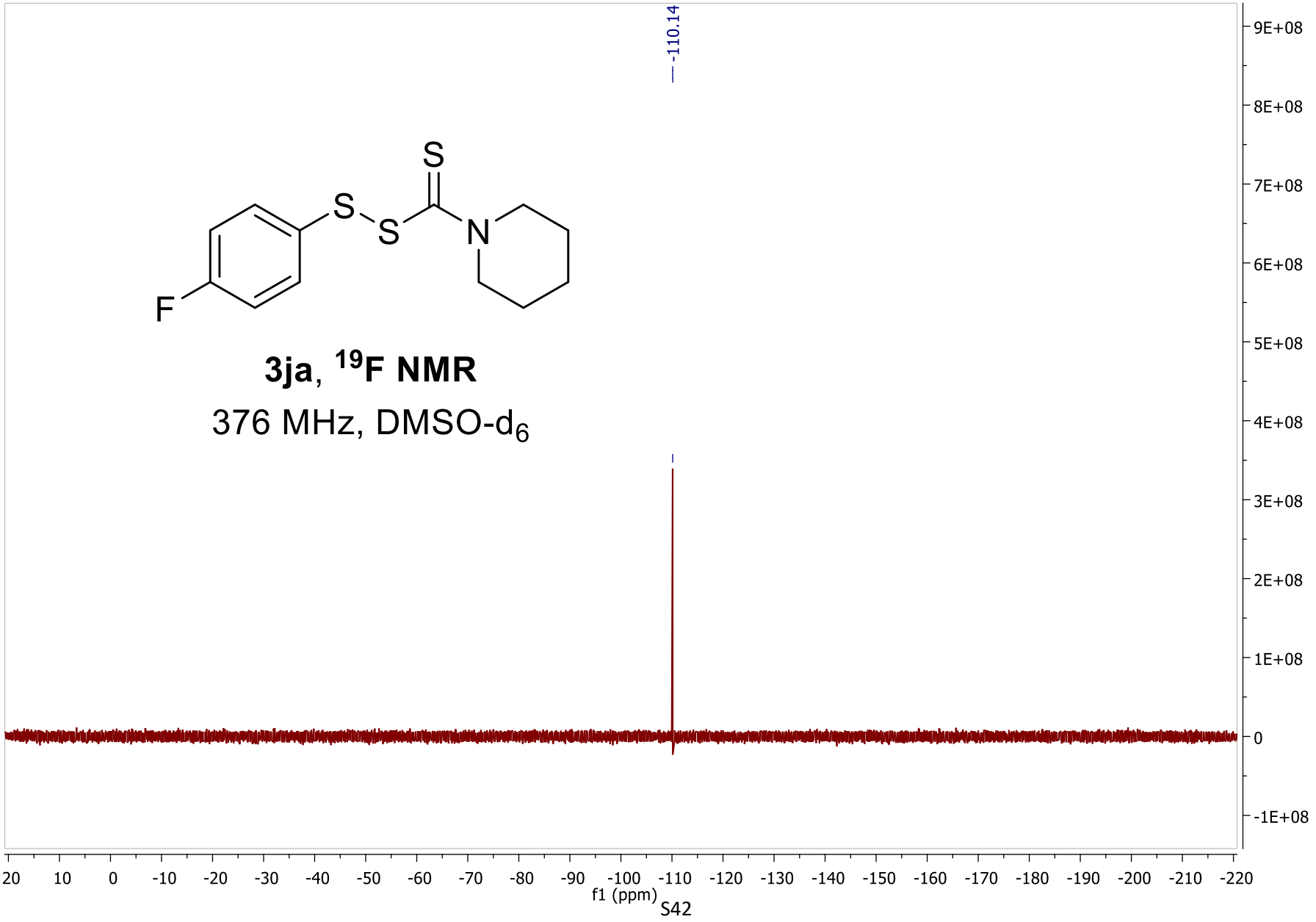
HRMS (ESI) m/z calcd for $C_{12}H_{15}ClNS_3$ $[M+H]^+$: 304.0055
found: 304.0055.

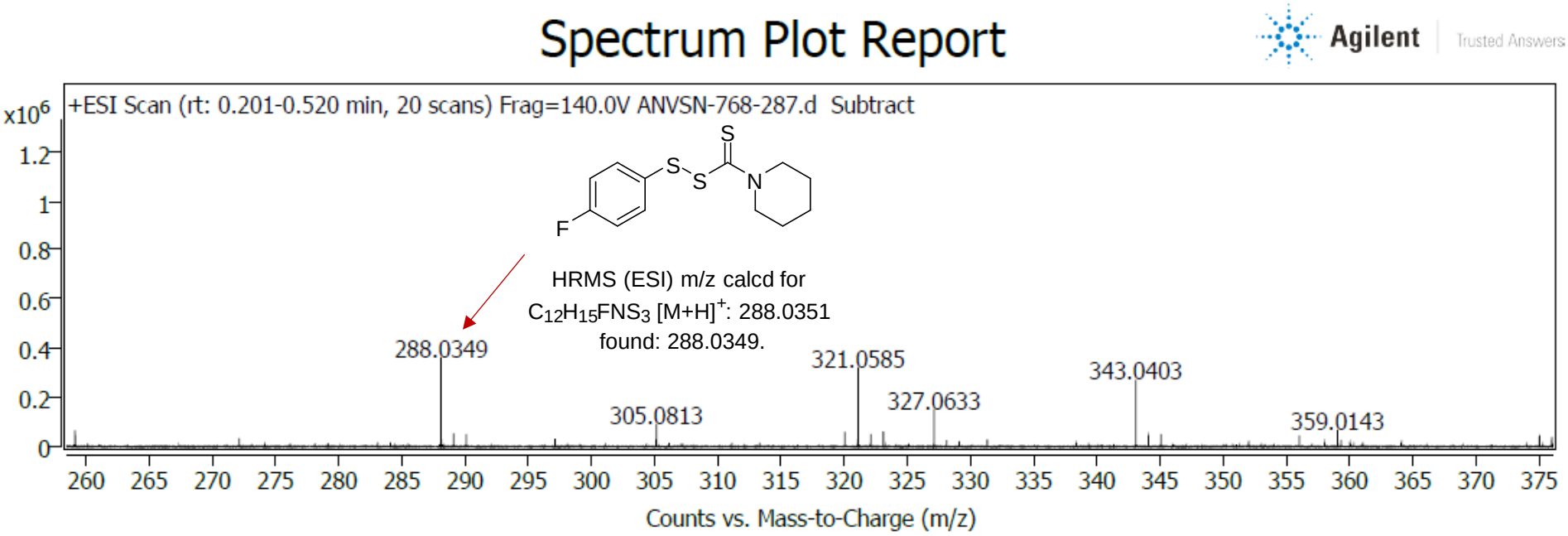

7.46
7.45
7.44
7.43
7.43
7.42
7.26
7.13
7.11
7.11
7.09
4.27
3.98
1.73
S
S
S
N
F
3ja, 1H NMR
400 MHz, DMSO-d6
2.00
2.02
2.04
2.04
6.05
f1 (ppm)

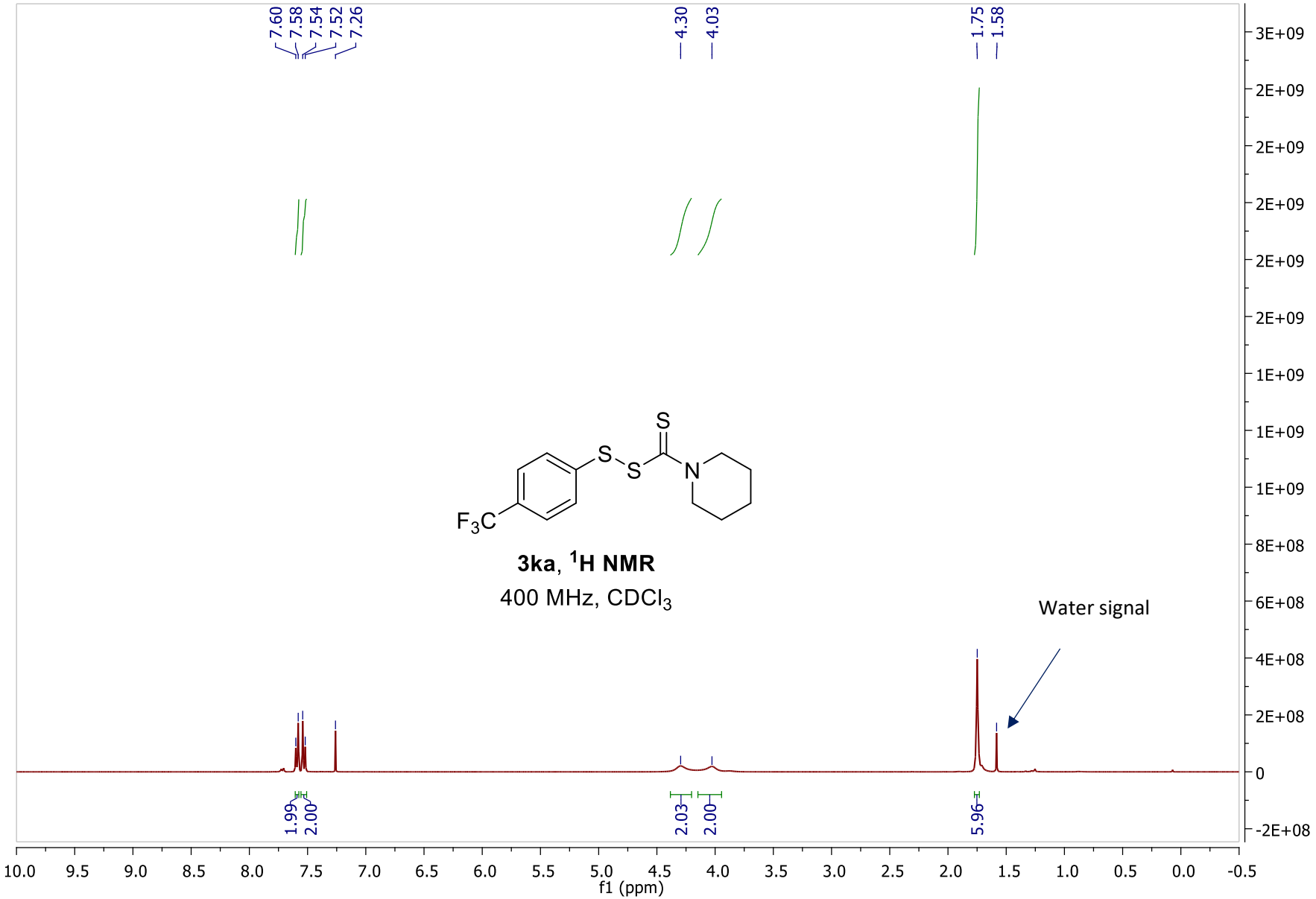


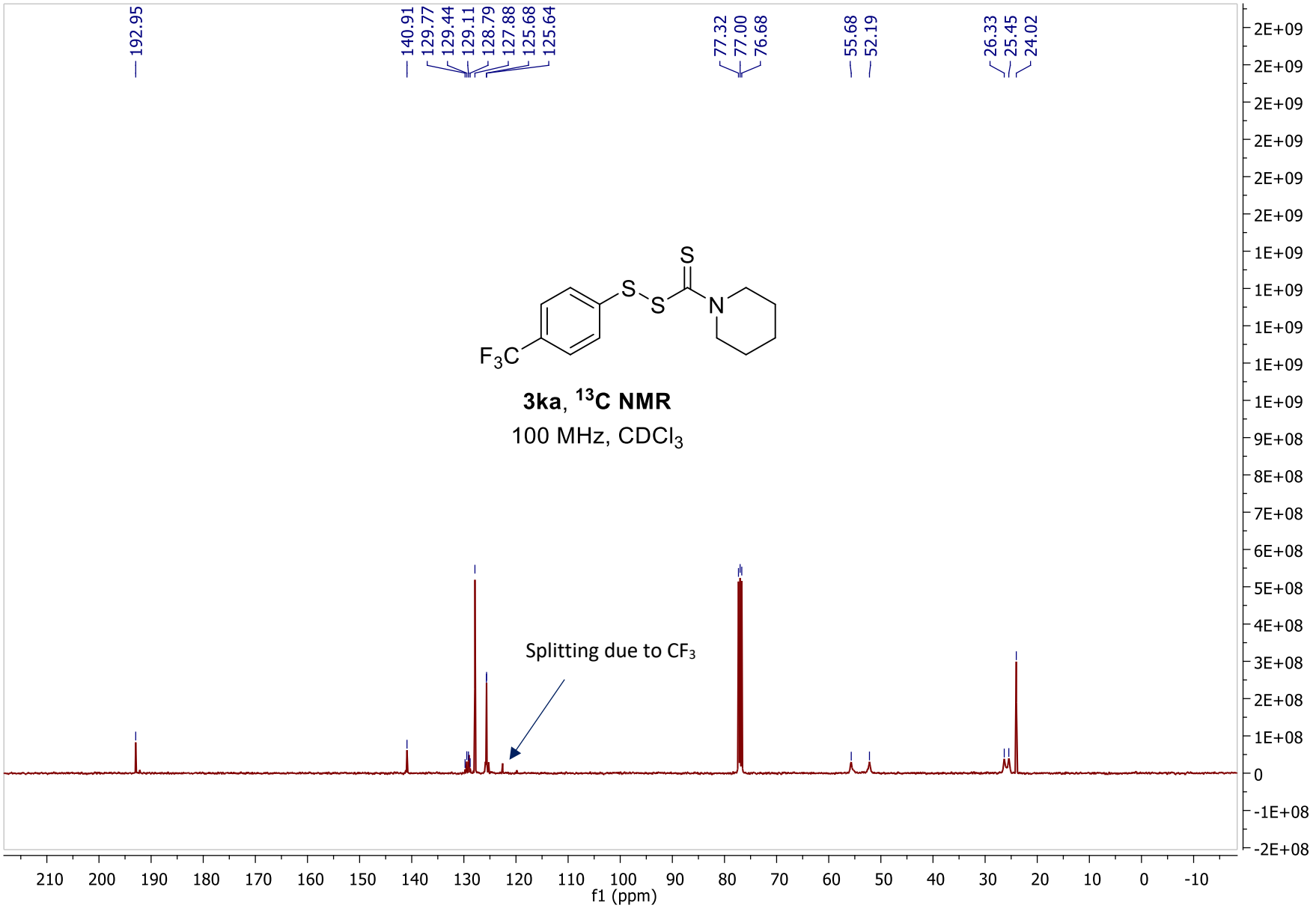
3ja, **^{13}C NMR**

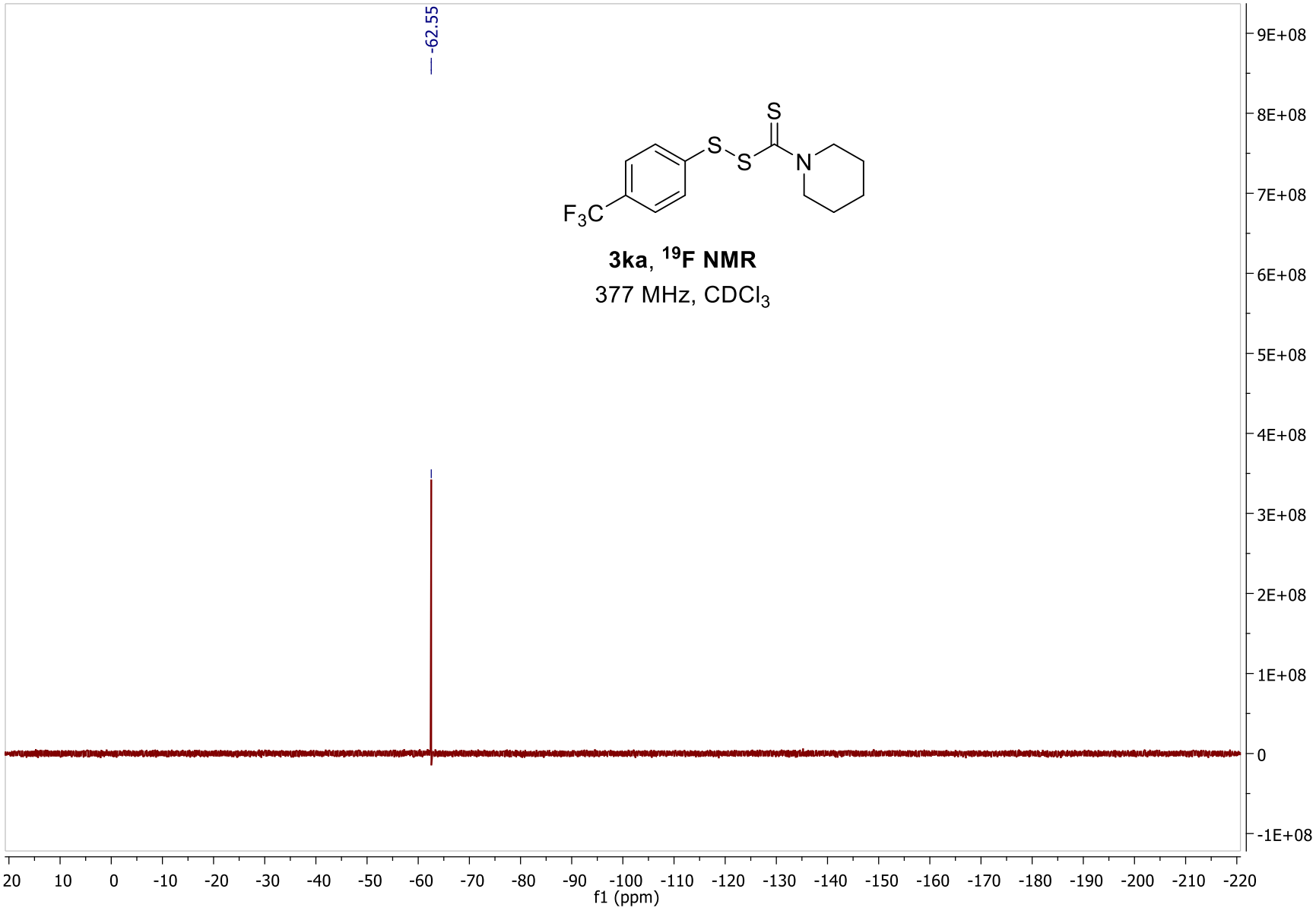
100 MHz, DMSO-d_6

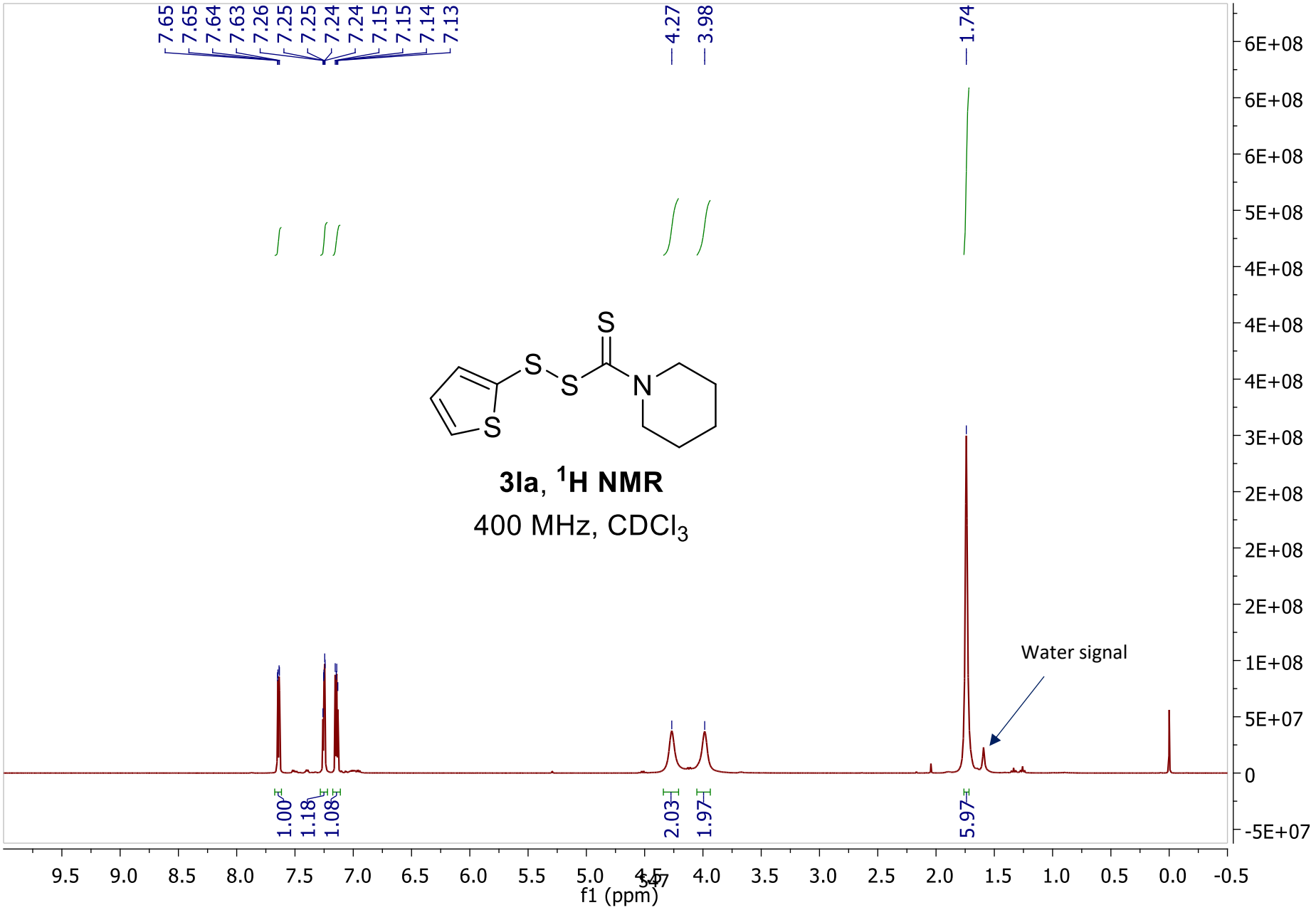

-110.14
S
S
S
N
F
3ja, 19F NMR
376 MHz, DMSO-d6
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

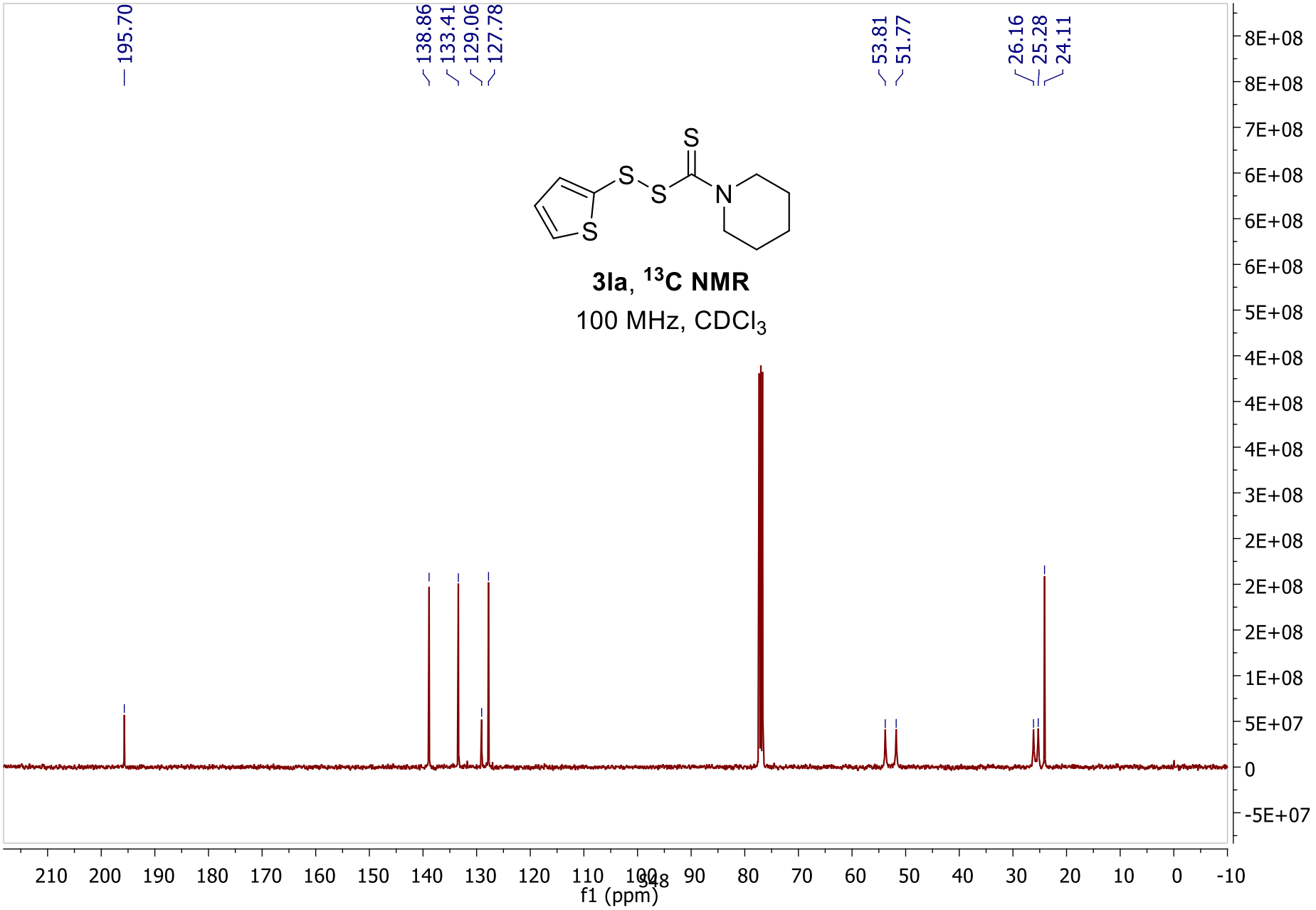

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.201-0.520 min, 20 scans) Frag=140.0V ANVSN-768-287.d Subtract
x10^6
1.2
1
0.8
0.6
0.4
0.2
0
S
S
S
N
F
HRMS (ESI) m/z calcd for
C12H15FNS3 [M+H]+: 288.0351
found: 288.0349.
288.0349
305.0813
321.0585
327.0633
343.0403
359.0143
260 265 270 275 280 285 290 295 300 305 310 315 320 325 330 335 340 345 350 355 360 365 370 375
Counts vs. Mass-to-Charge (m/z)


7.60
7.58
7.54
7.52
7.26
4.30
4.03
1.75
1.58
S
S
S
N
F3C
3ka, 1H NMR
400 MHz, CDCl3
Water signal
1.99
2.00
2.03
2.00
5.96
3E+09
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
8E+08
6E+08
4E+08
2E+08
0
-2E+08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

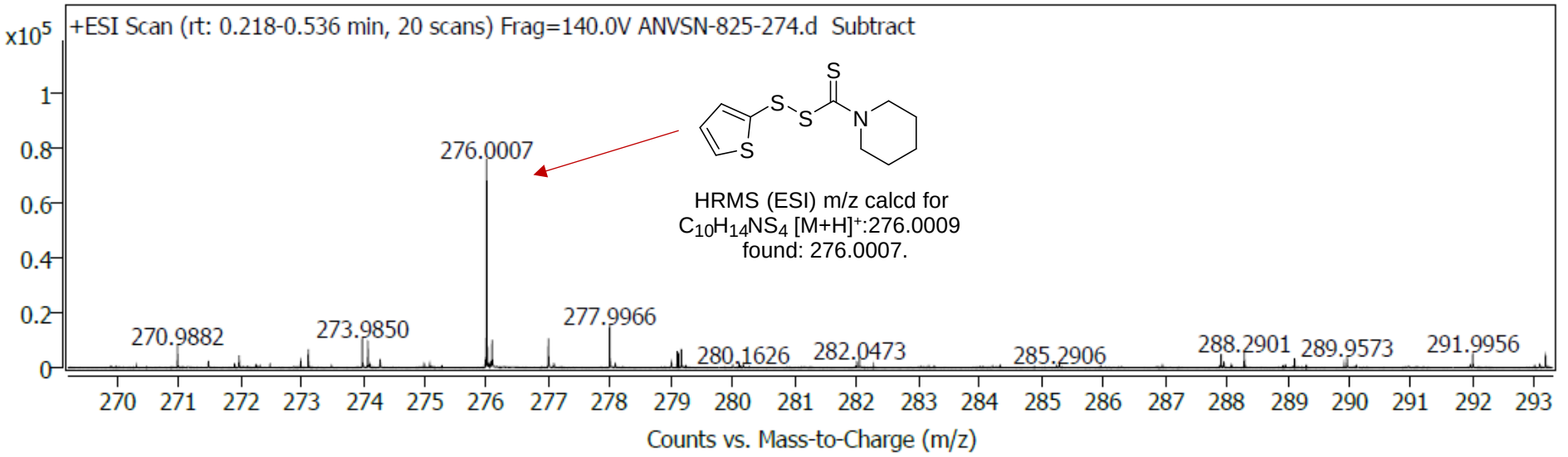

192.95
140.91
129.77
129.44
129.11
128.79
127.88
125.68
125.64
77.32
77.00
76.68
55.68
52.19
26.33
25.45
24.02
S
S
S
N
F3C
3ka, 13C NMR
100 MHz, CDCl3
Splitting due to CF3
2E+09
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
-2E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-62.55
S
S
S
N
F3C
3ka, 19F NMR
377 MHz, CDCl3
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

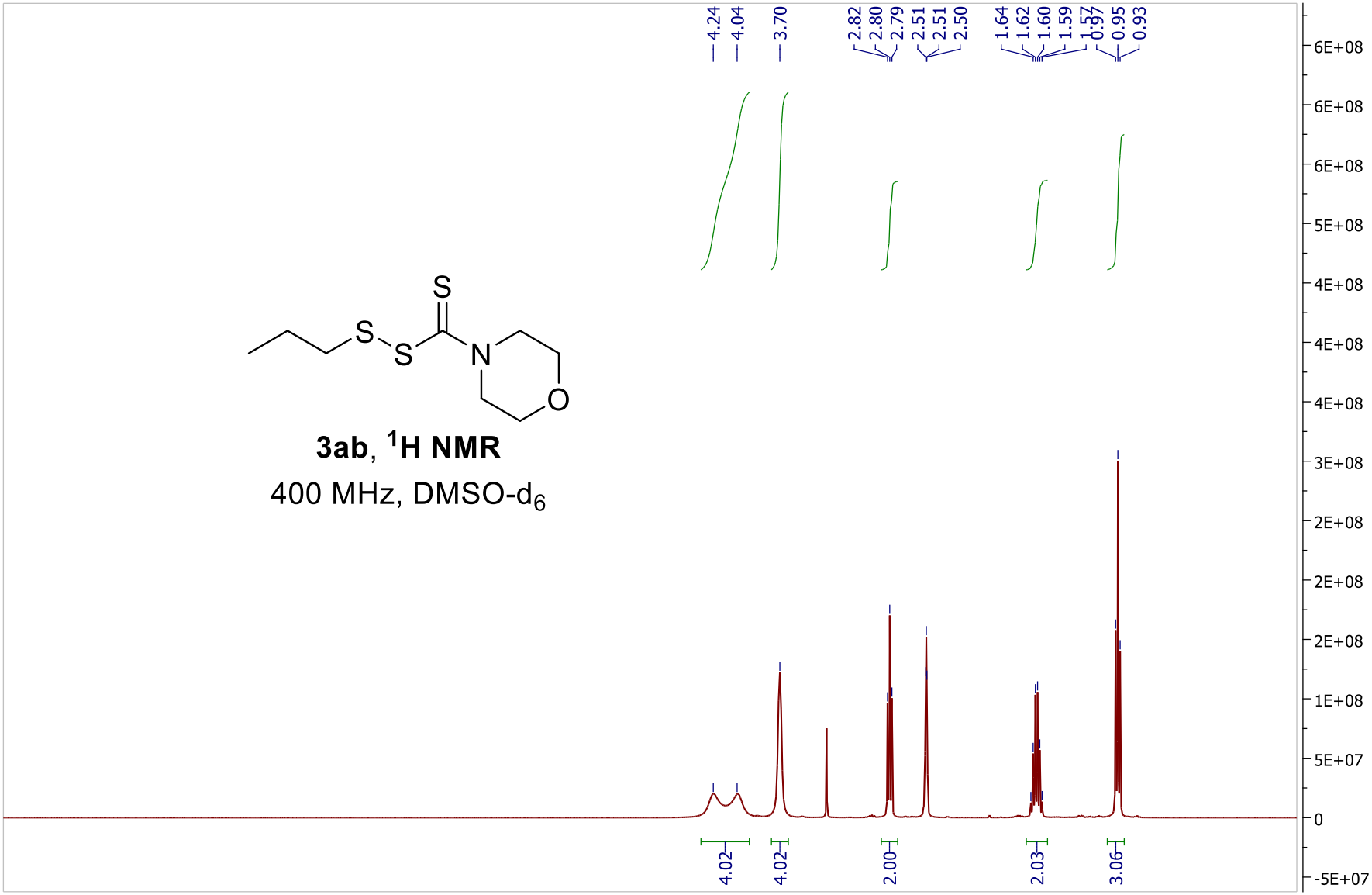

7.65
7.65
7.64
7.63
7.26
7.25
7.25
7.24
7.24
7.15
7.15
7.14
7.13
4.27
3.98
1.74
S
S
S
N
S
3la, 1H NMR
400 MHz, CDCl3
Water signal
1.00
1.18
1.08
2.03
1.97
5.97
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

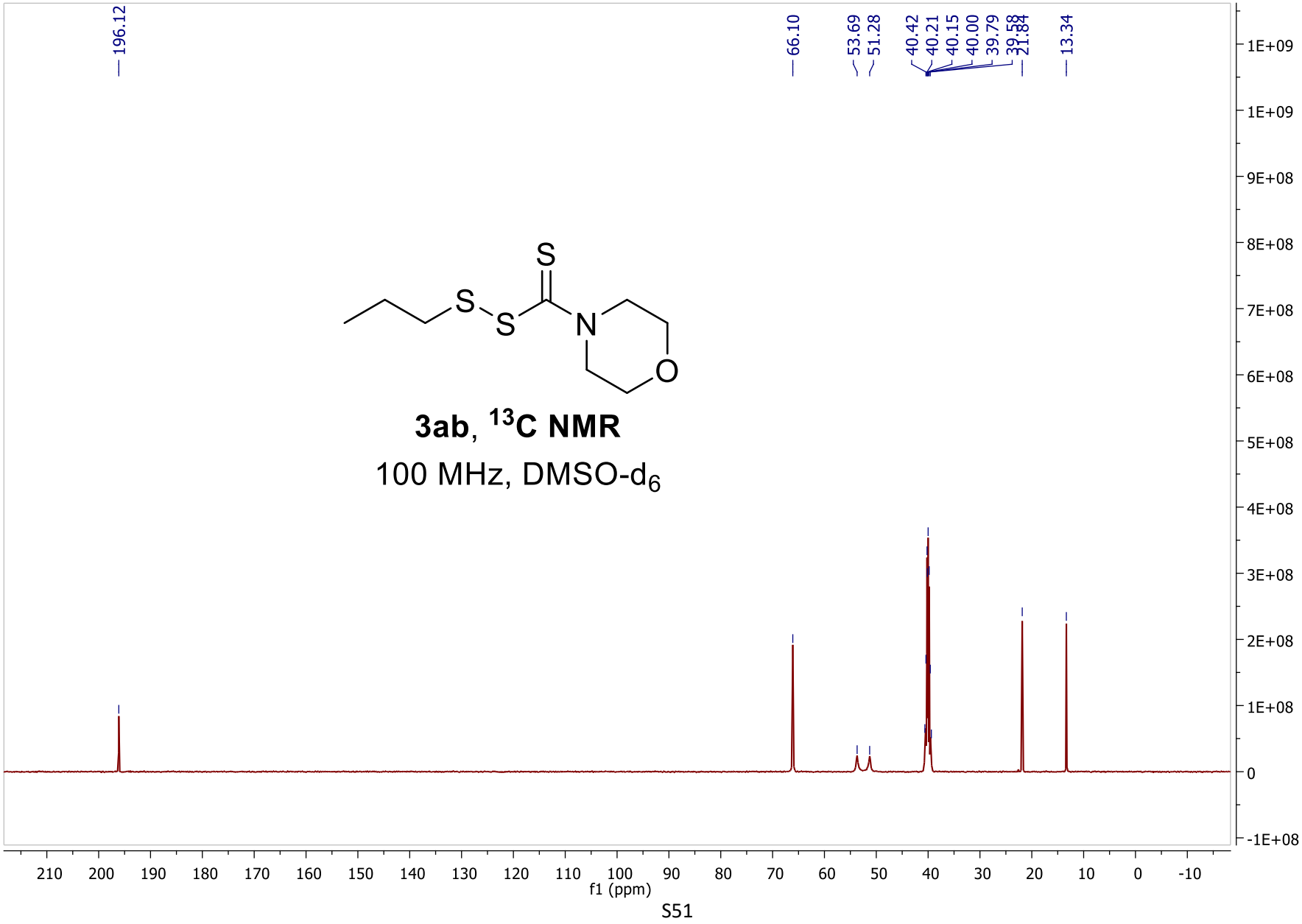

195.70
138.86
133.41
129.06
127.78
53.81
51.77
26.16
25.28
24.11
S
S
S
N
S
3Ia, 13C NMR
100 MHz, CDCl3
8E+08
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Spectrum Plot Report

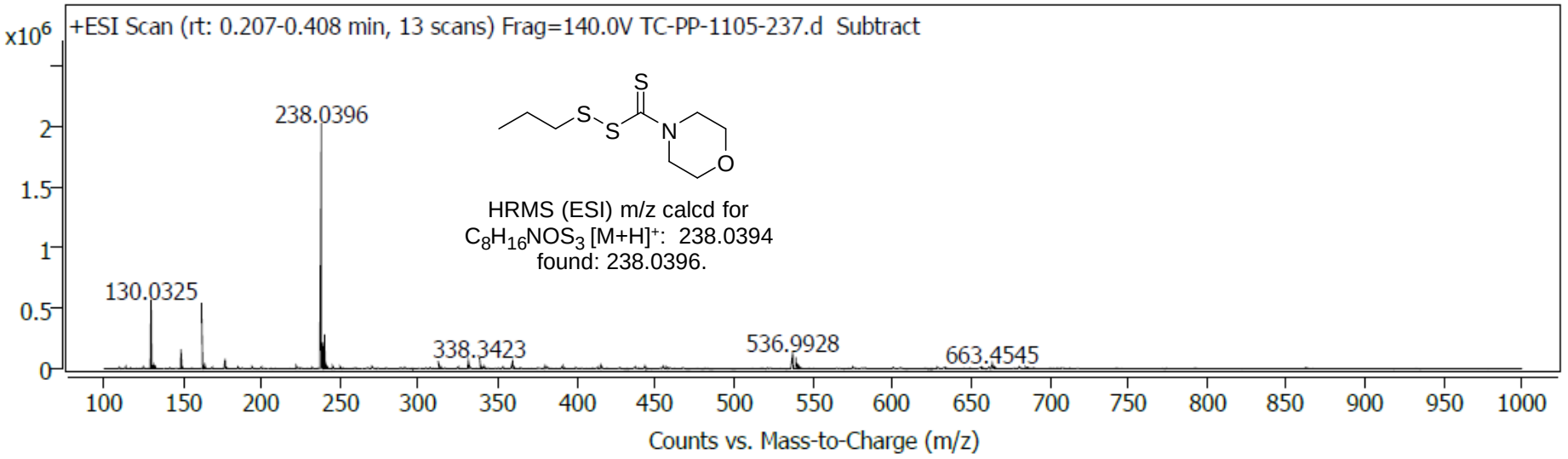


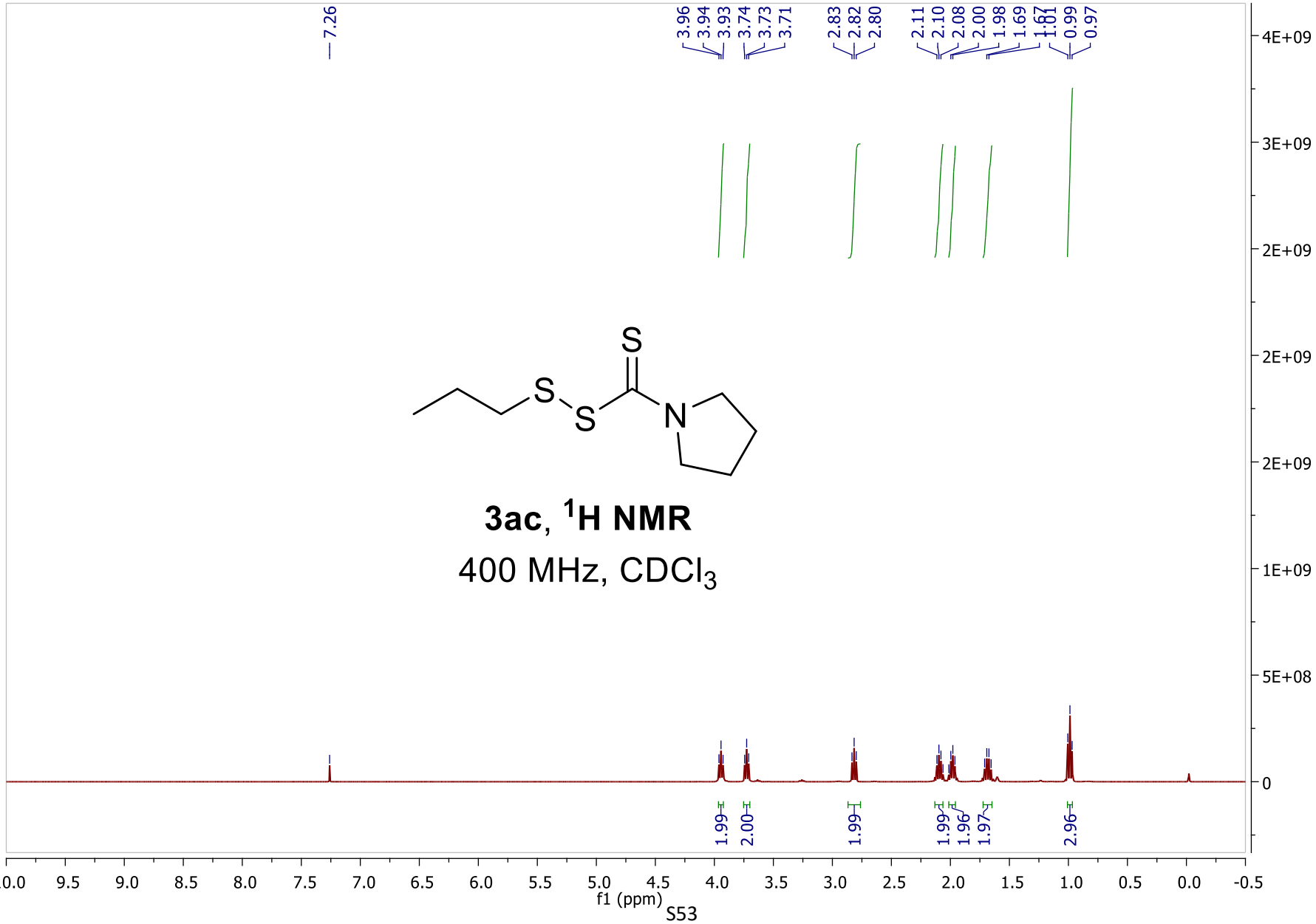
HRMS (ESI) m/z calcd for $C_{10}H_{14}NS_4$ $[M+H]^+$:276.0009
found: 276.0007.

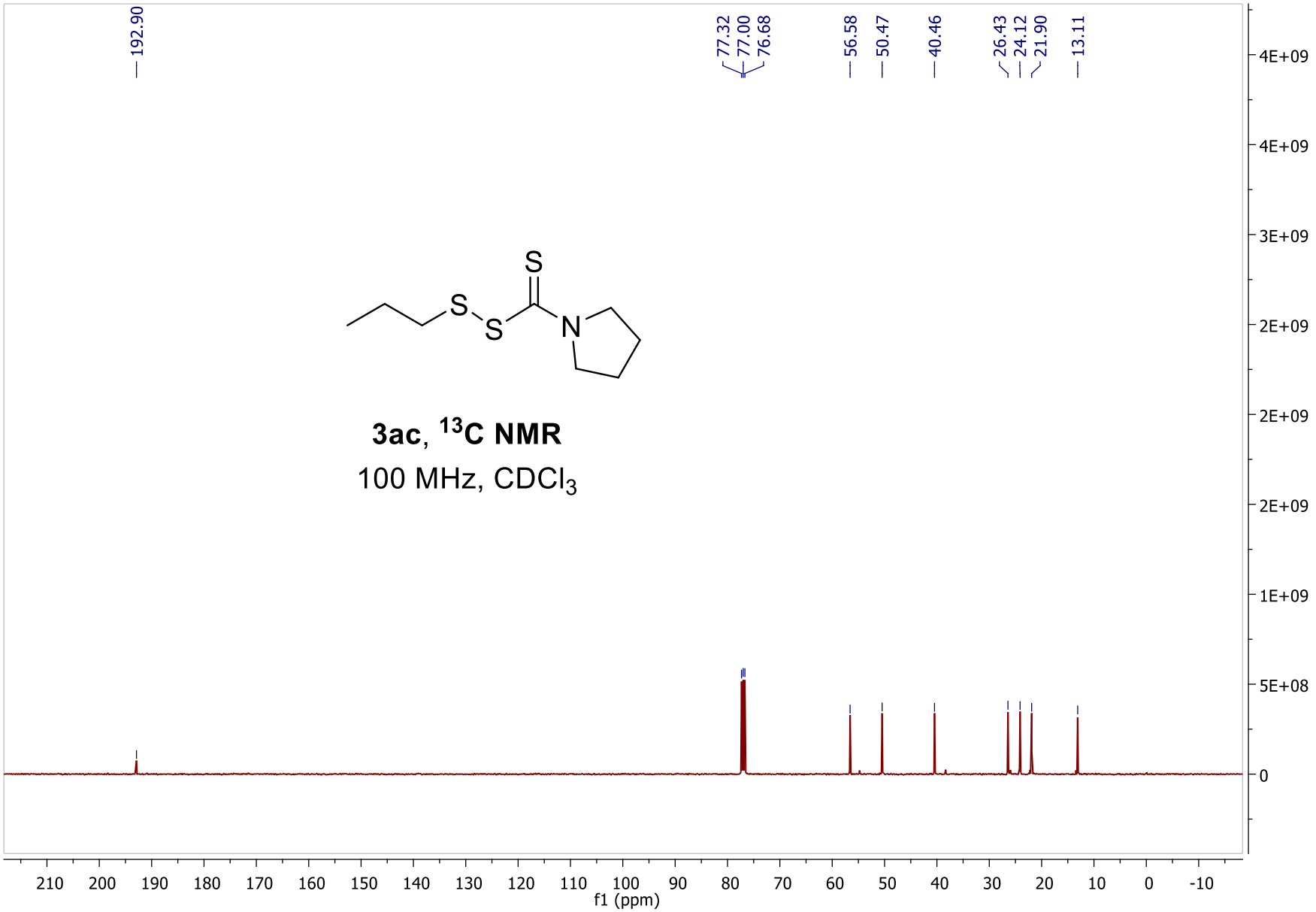

4.24
4.04
3.70
2.82
2.80
2.79
2.51
2.51
2.50
1.64
1.62
1.60
1.59
1.57
0.97
0.95
0.93
S
S
S
N
O
3ab, 1H NMR
400 MHz, DMSO-d6
4.02
4.02
2.00
2.03
3.06
f1 (ppm)

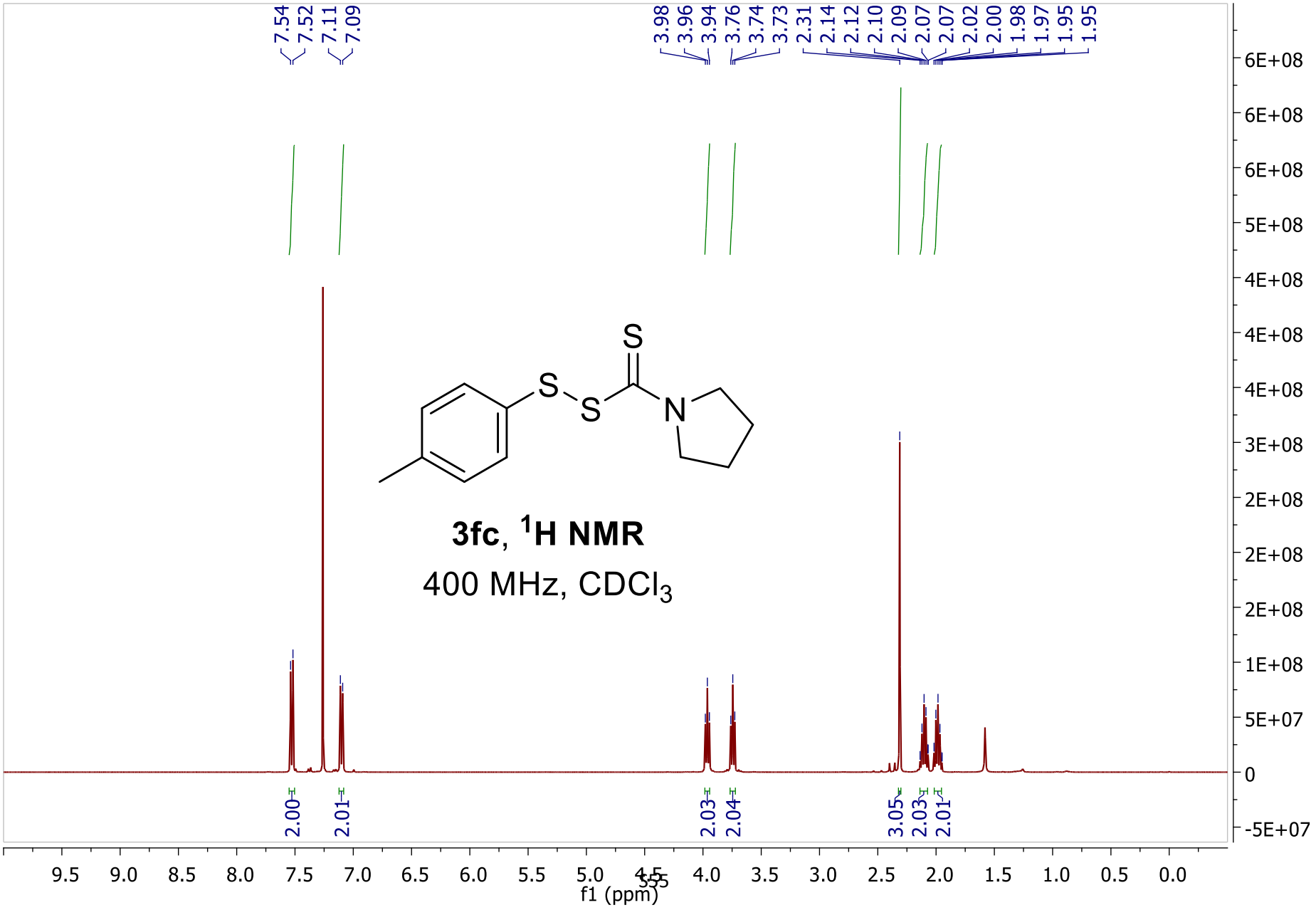

196.12
66.10
53.69
51.28
40.42
40.21
40.15
40.00
39.79
39.58
21.84
13.34
S
S
S
N
O
3ab, 13C NMR
100 MHz, DMSO-d6
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

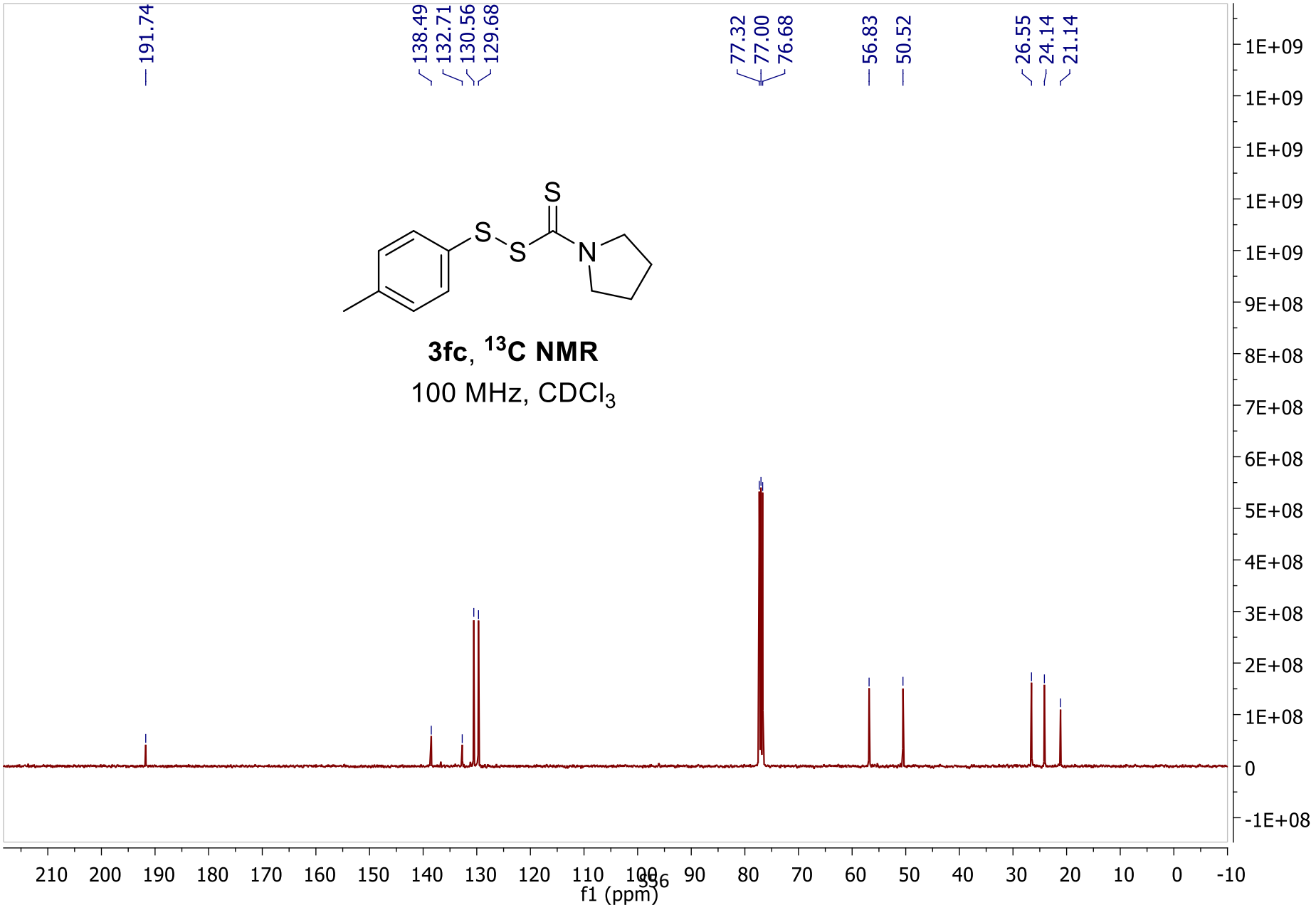
Spectrum Plot Report



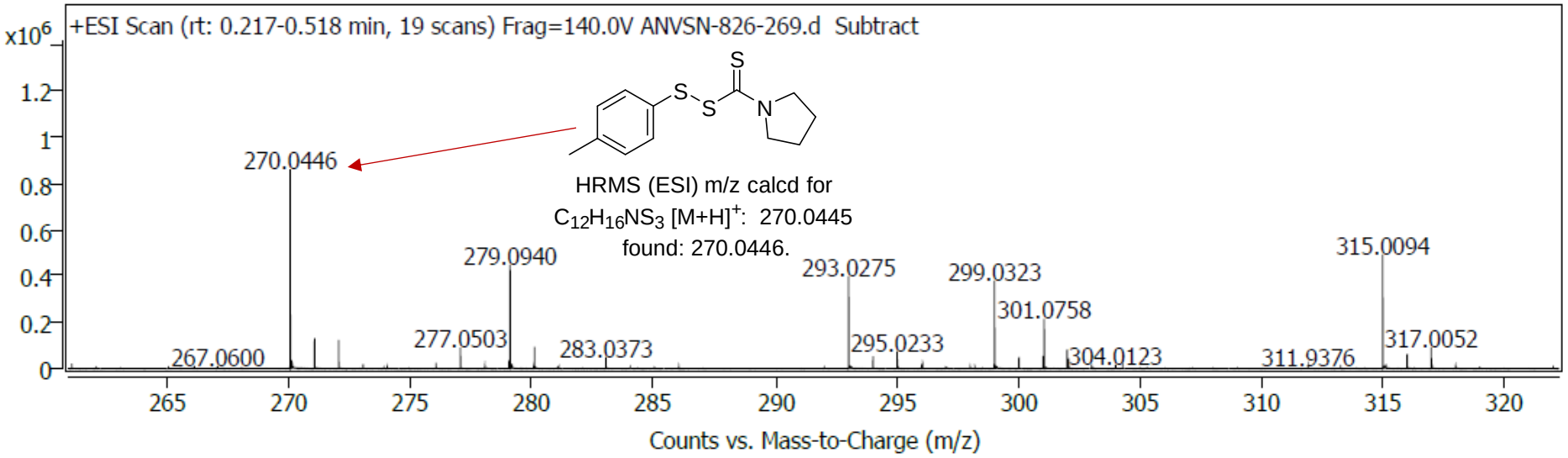

7.26
3.96
3.94
3.93
3.74
3.73
3.71
2.83
2.82
2.80
2.11
2.10
2.08
2.00
1.98
1.69
1.67
1.61
0.99
0.97
S
S
S
N
3ac, 1H NMR
400 MHz, CDCl3
4E+09
3E+09
2E+09
2E+09
2E+09
1E+09
5E+08
0
1.99
2.00
1.99
1.99
1.96
1.97
2.96
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


192.90
77.32
77.00
76.68
56.58
50.47
40.46
26.43
24.12
21.90
13.11
S
S
S
N
3ac, ^{13}C NMR
100 MHz, $CDCl_3$
4E+09
4E+09
3E+09
2E+09
2E+09
2E+09
1E+09
5E+08
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

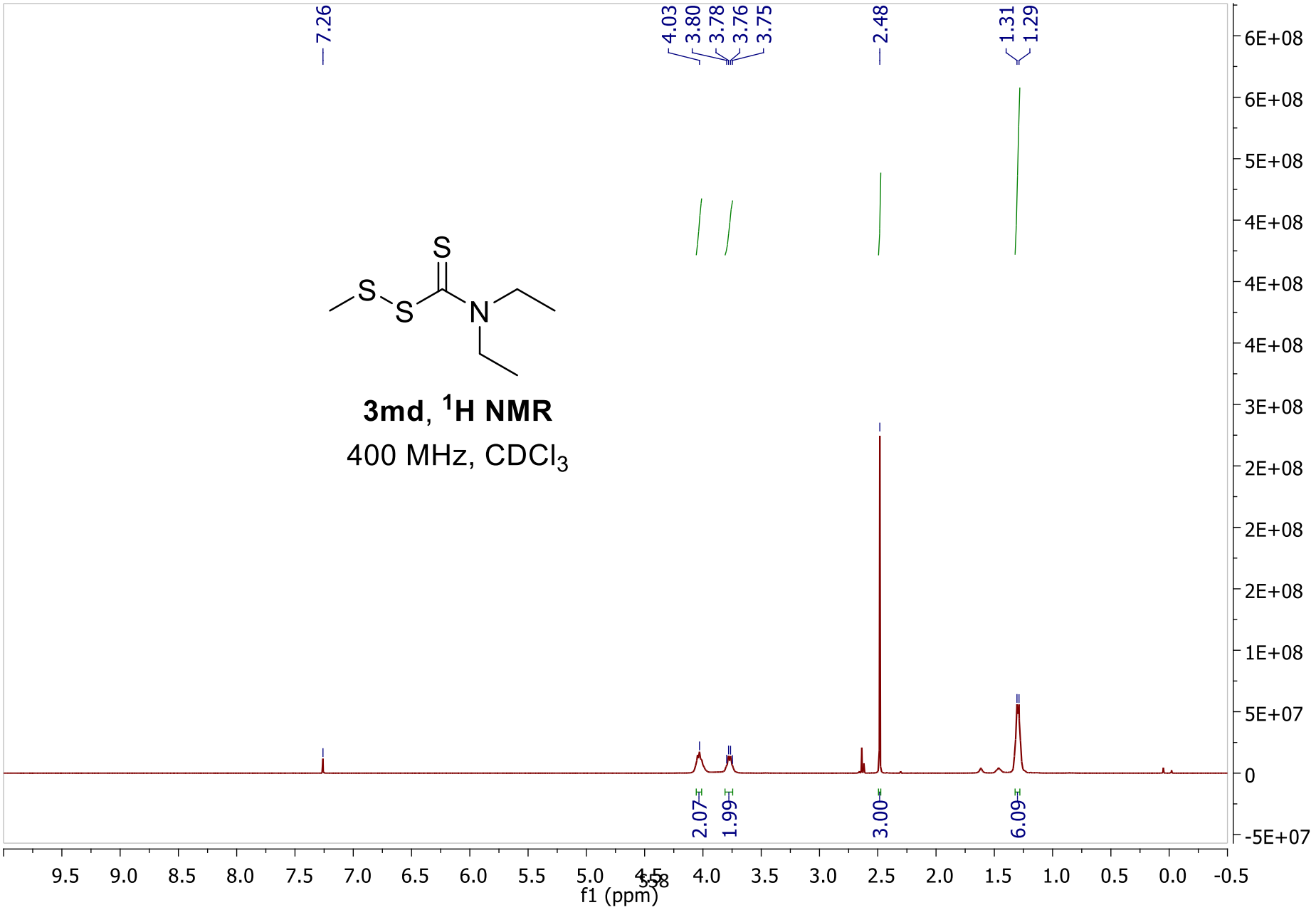

7.54
7.52
7.11
7.09
3.98
3.96
3.94
3.76
3.74
3.73
2.31
2.14
2.12
2.10
2.09
2.07
2.07
2.02
2.00
1.98
1.97
1.95
1.95
S
S
S
N
3fc, 1H NMR
400 MHz, CDCl3
2.00
2.01
2.03
2.04
3.05
2.03
2.01
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

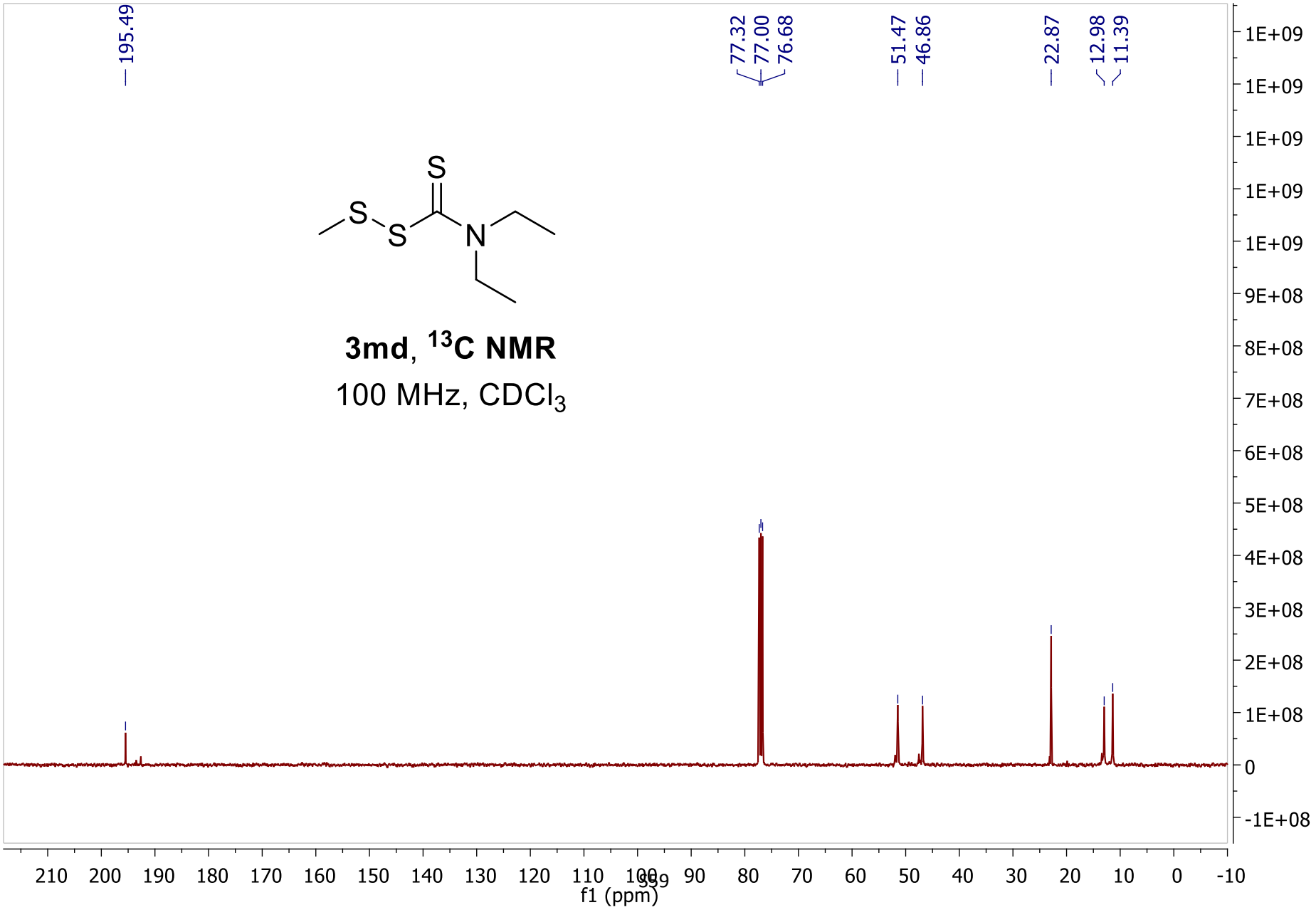

191.74
138.49
132.71
130.56
129.68
77.32
77.00
76.68
56.83
50.52
26.55
24.14
21.14
S
S
S
N
3fc, 13C NMR
100 MHz, CDCl3
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Spectrum Plot Report

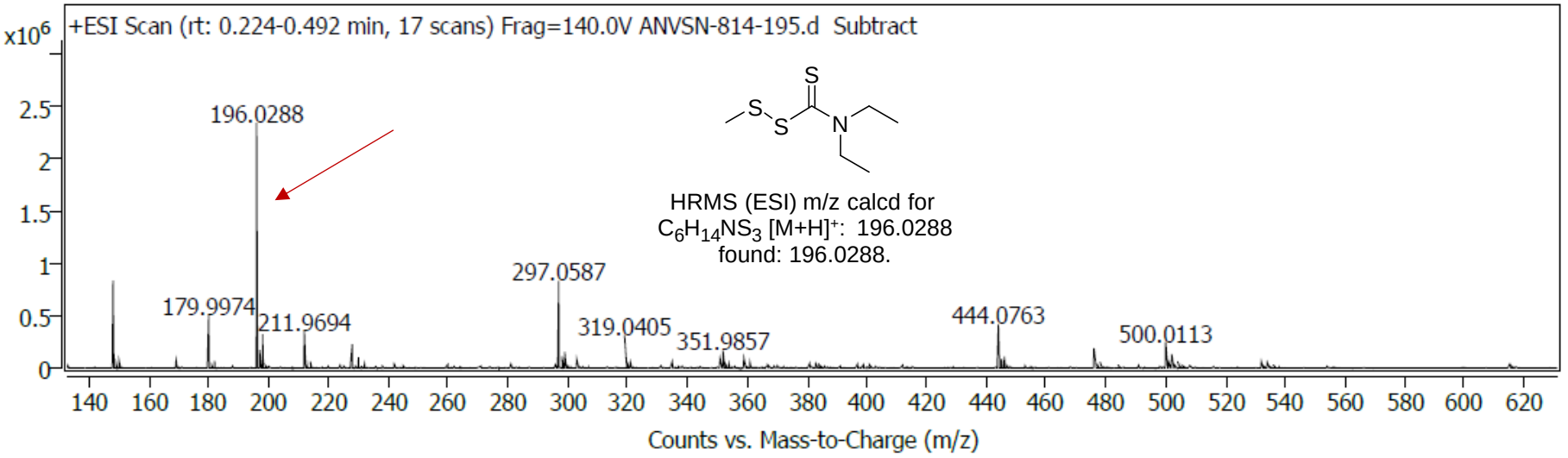


HRMS (ESI) m/z calcd for $C_{12}H_{16}NS_3$ $[M+H]^+$: 270.0445
found: 270.0446.

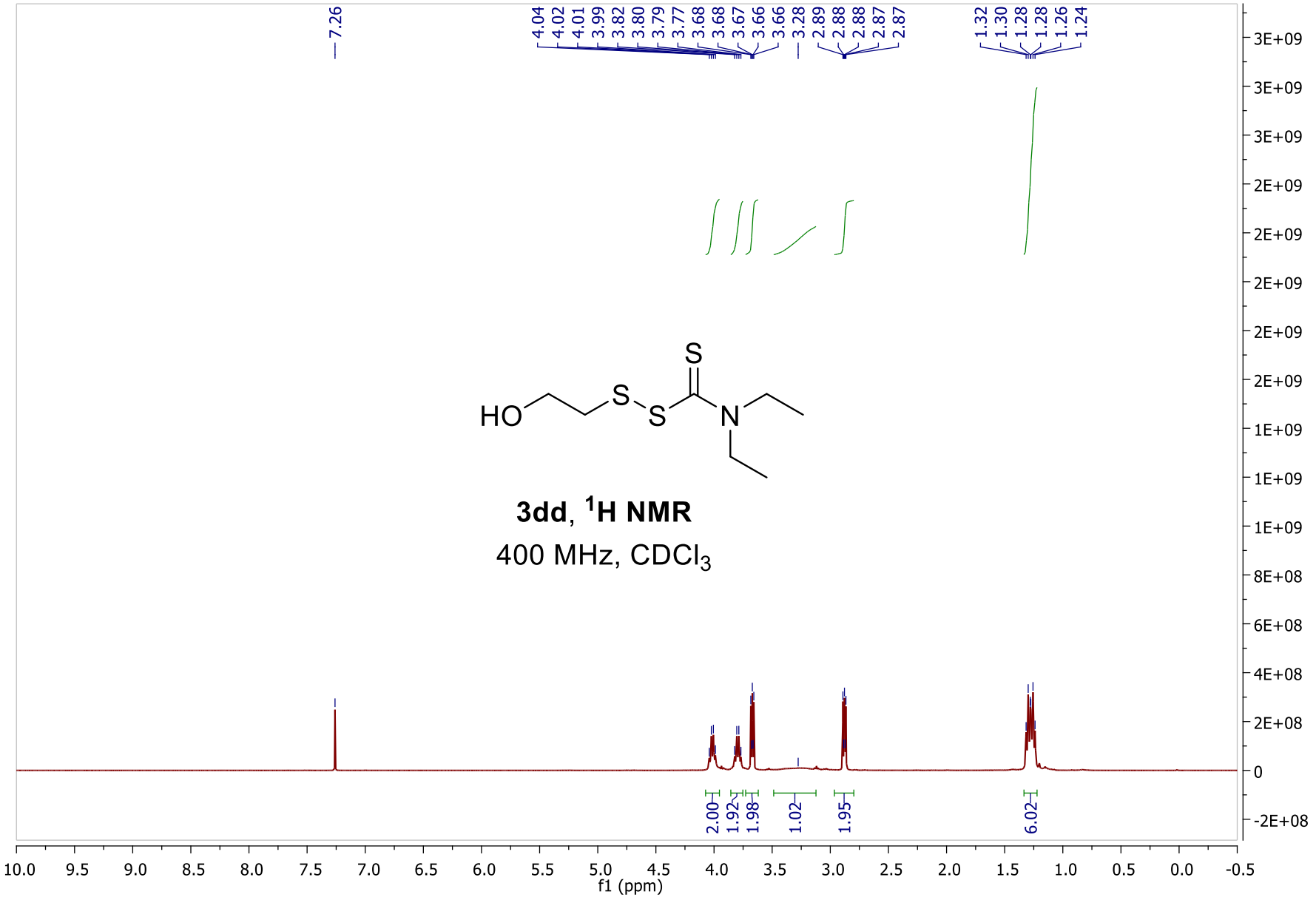

7.26
4.03
3.80
3.78
3.76
3.75
2.48
1.31
1.29
S
S
S
N
3md, 1H NMR
400 MHz, CDCl3
2.07
1.99
3.00
6.09
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
5E+07
0
-5E+07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

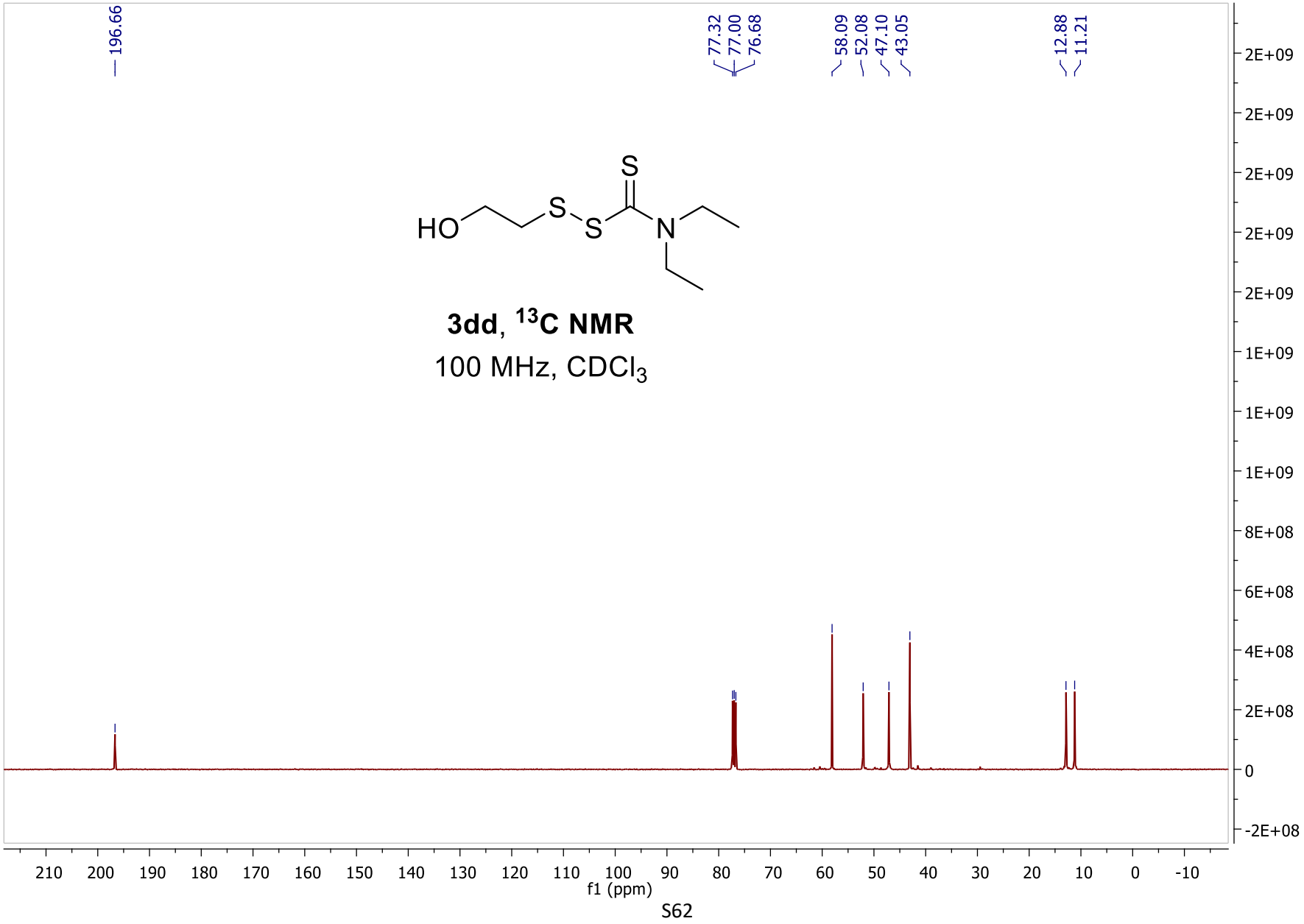

195.49
77.32
77.00
76.68
51.47
46.86
22.87
12.98
11.39
S
S
S
N
3md, 13C NMR
100 MHz, CDCl3
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

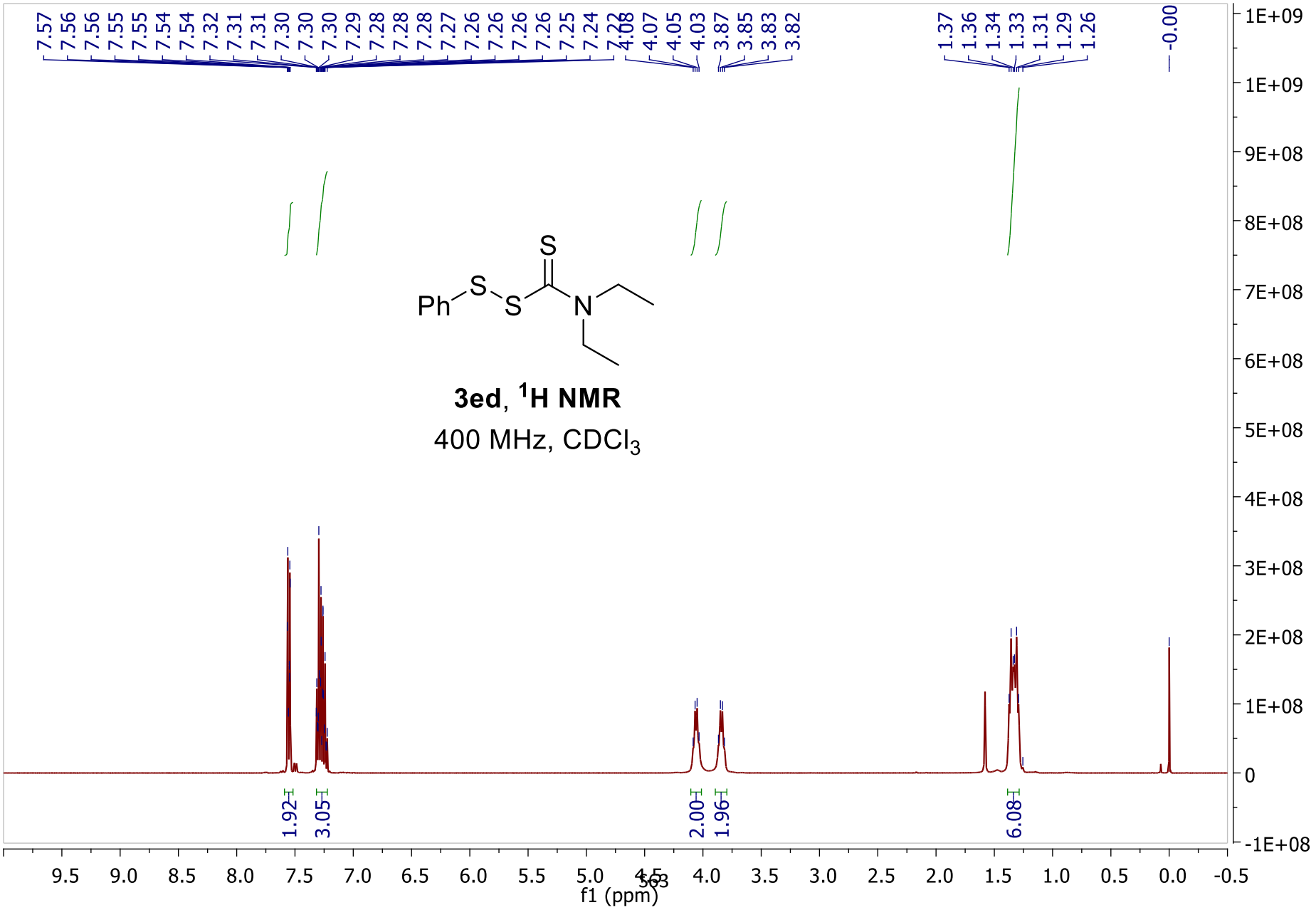
Spectrum Plot Report

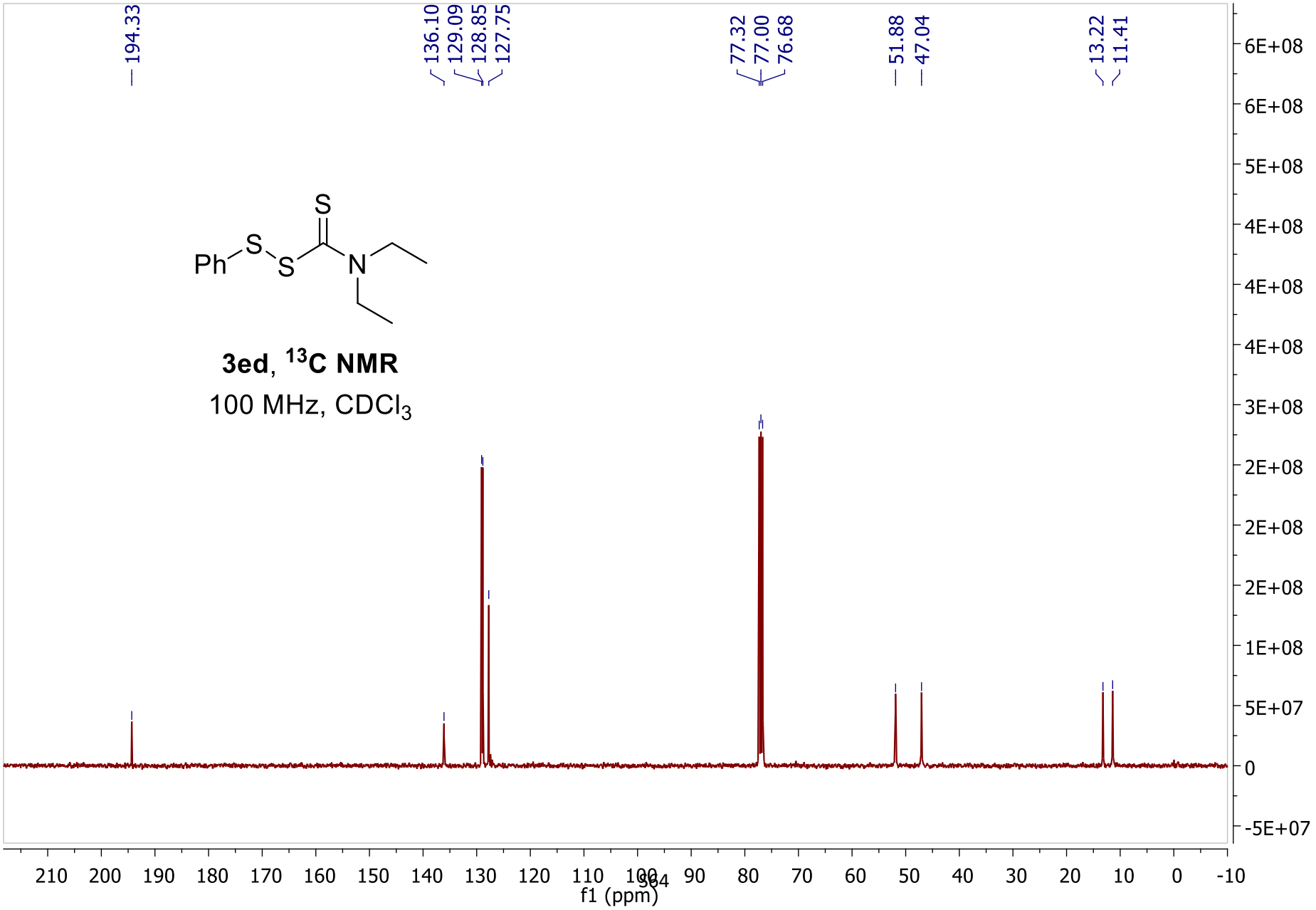


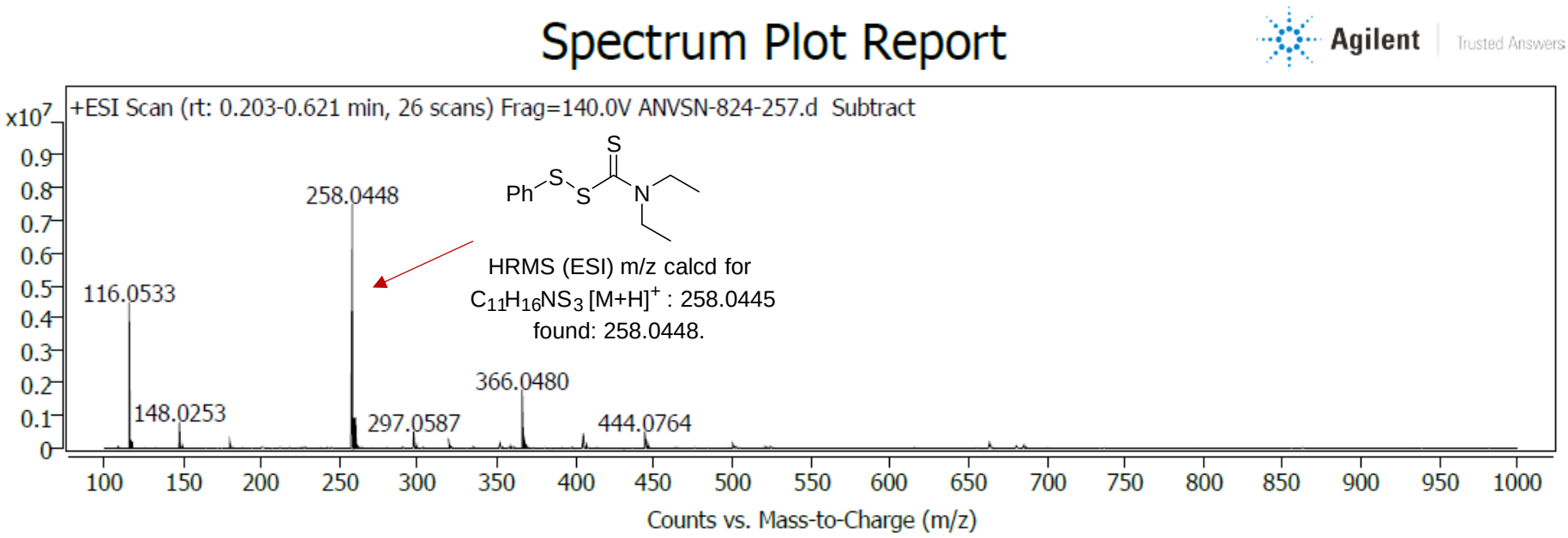
HRMS (ESI) m/z calcd for $C_6H_{14}NS_3$ $[M+H]^+$: 196.0288
found: 196.0288.

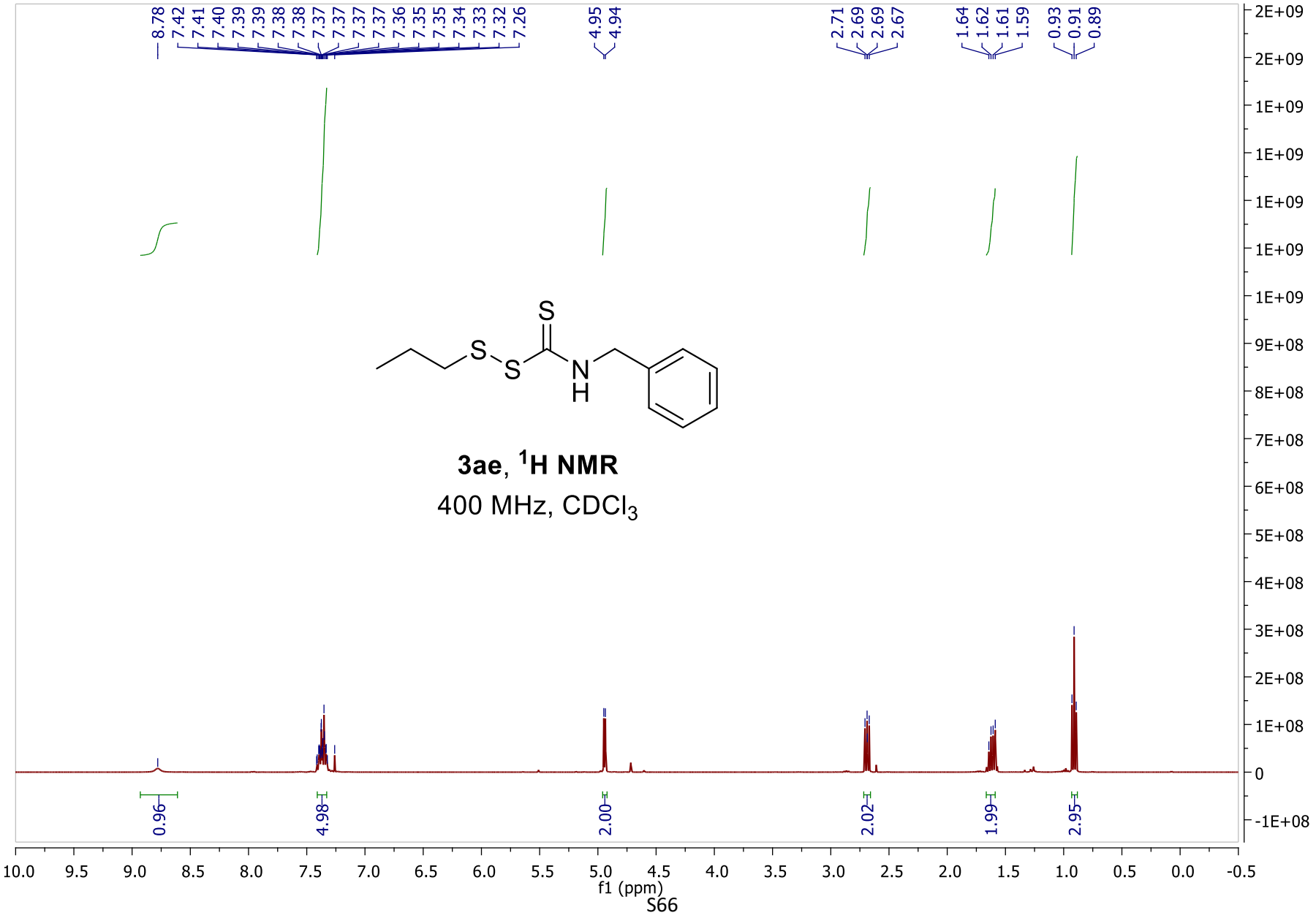

7.26
4.04
4.02
4.01
3.99
3.82
3.80
3.79
3.77
3.68
3.68
3.67
3.66
3.66
3.28
2.89
2.88
2.88
2.87
2.87
1.32
1.30
1.28
1.28
1.26
1.24
HO
S
S
S
N
3dd, 1H NMR
400 MHz, CDCl3
2.00
1.92
1.98
1.02
1.95
6.02
f1 (ppm)

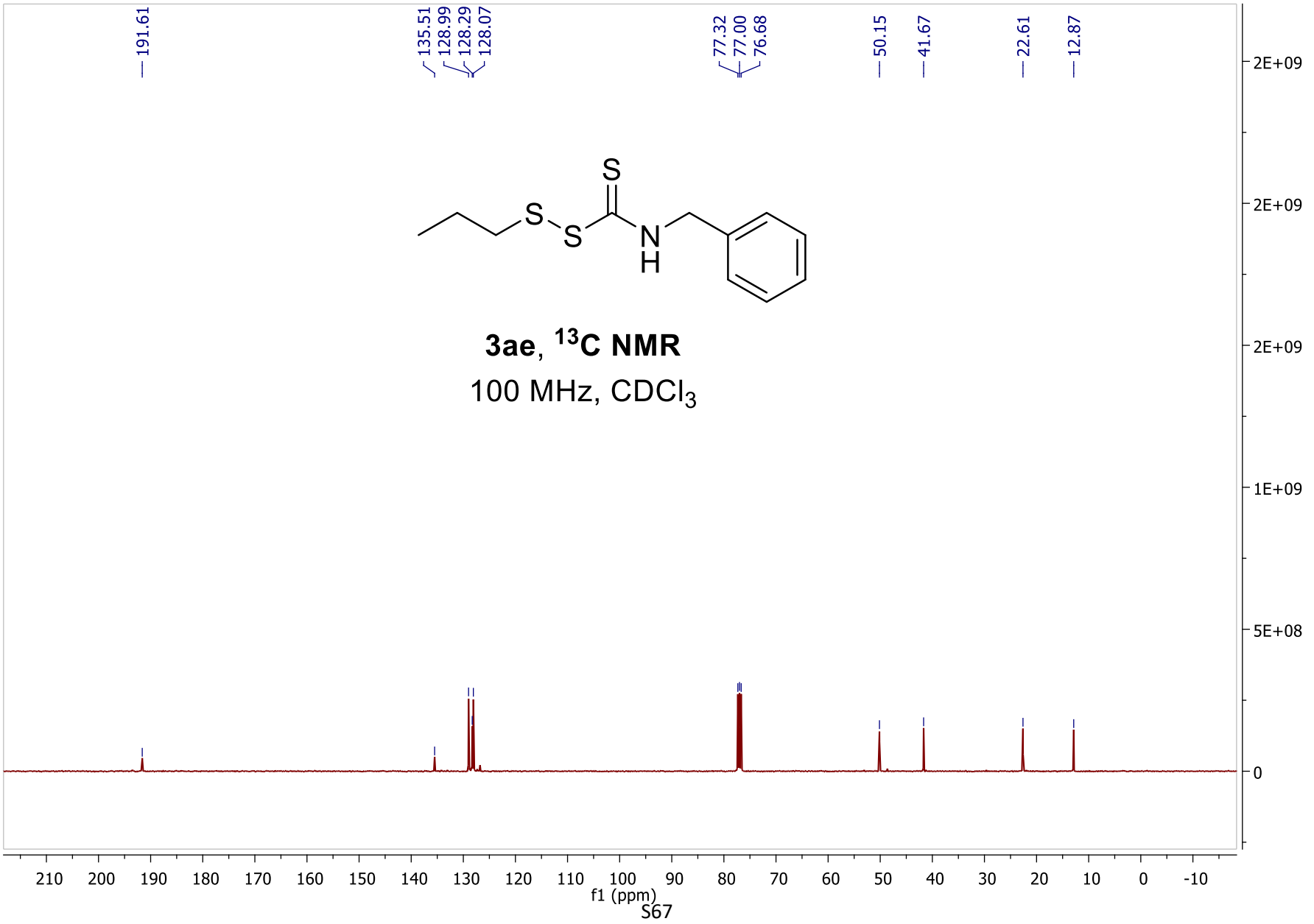

196.66
77.32
77.00
76.68
58.09
52.08
47.10
43.05
12.88
11.21
HO
S
S
S
N
3dd, 13C NMR
100 MHz, CDCl3
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
8E+08
6E+08
4E+08
2E+08
0
-2E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.57
7.56
7.56
7.55
7.55
7.54
7.54
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.28
7.28
7.28
7.27
7.26
7.26
7.26
7.26
7.25
7.24
7.22
4.08
4.07
4.05
4.03
3.87
3.85
3.83
3.82
1.37
1.36
1.34
1.33
1.31
1.29
1.26
-0.00
S
S
Ph
S
N
3ed, 1H NMR
400 MHz, CDCl3
1.92
3.05
2.00
1.96
6.08
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


194.33
136.10
129.09
128.85
127.75
77.32
77.00
76.68
51.88
47.04
13.22
11.41
S
Ph
S
S
N
3ed, 13C NMR
100 MHz, CDCl3
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.203-0.621 min, 26 scans) Frag=140.0V ANVSN-824-257.d Subtract
x10^7
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
258.0448
116.0533
148.0253
297.0587
366.0480
444.0764
Ph
S
S
S
N
HRMS (ESI) m/z calcd for
C11H16NS3 [M+H]+ : 258.0445
found: 258.0448.
100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000
Counts vs. Mass-to-Charge (m/z)

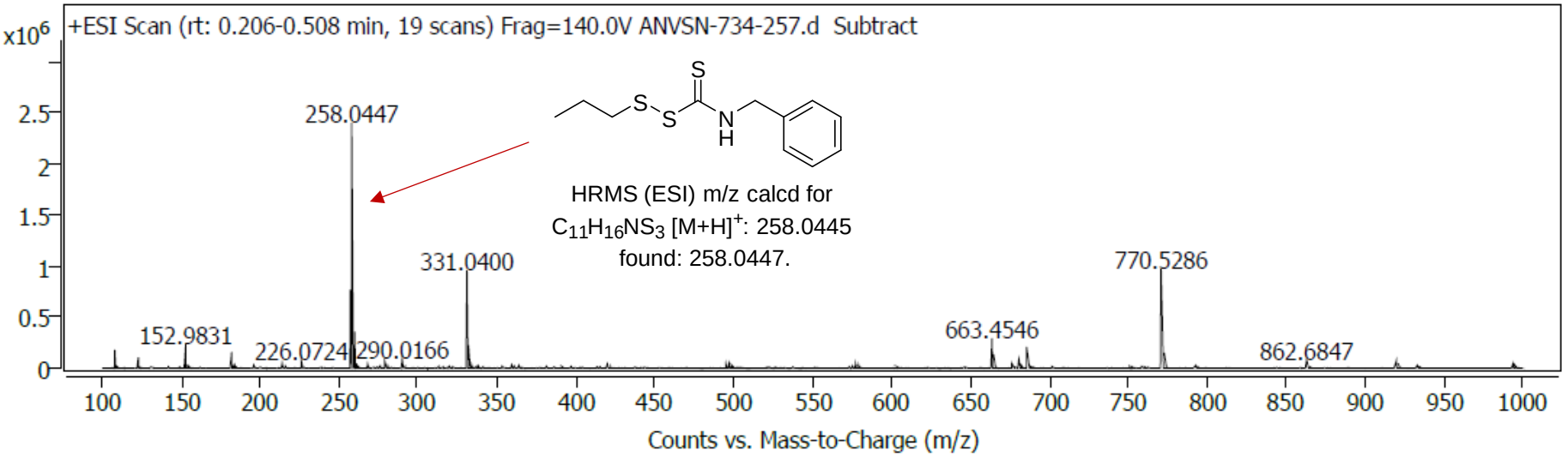

8.78
7.42
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.37
7.36
7.35
7.35
7.34
7.33
7.32
7.26
4.95
4.94
2.71
2.69
2.69
2.67
1.64
1.62
1.61
1.59
0.93
0.91
0.89
S
S
S
N
H
3ae, 1H NMR
400 MHz, CDCl3
0.96
4.98
2.00
2.02
1.99
2.95
f1 (ppm)



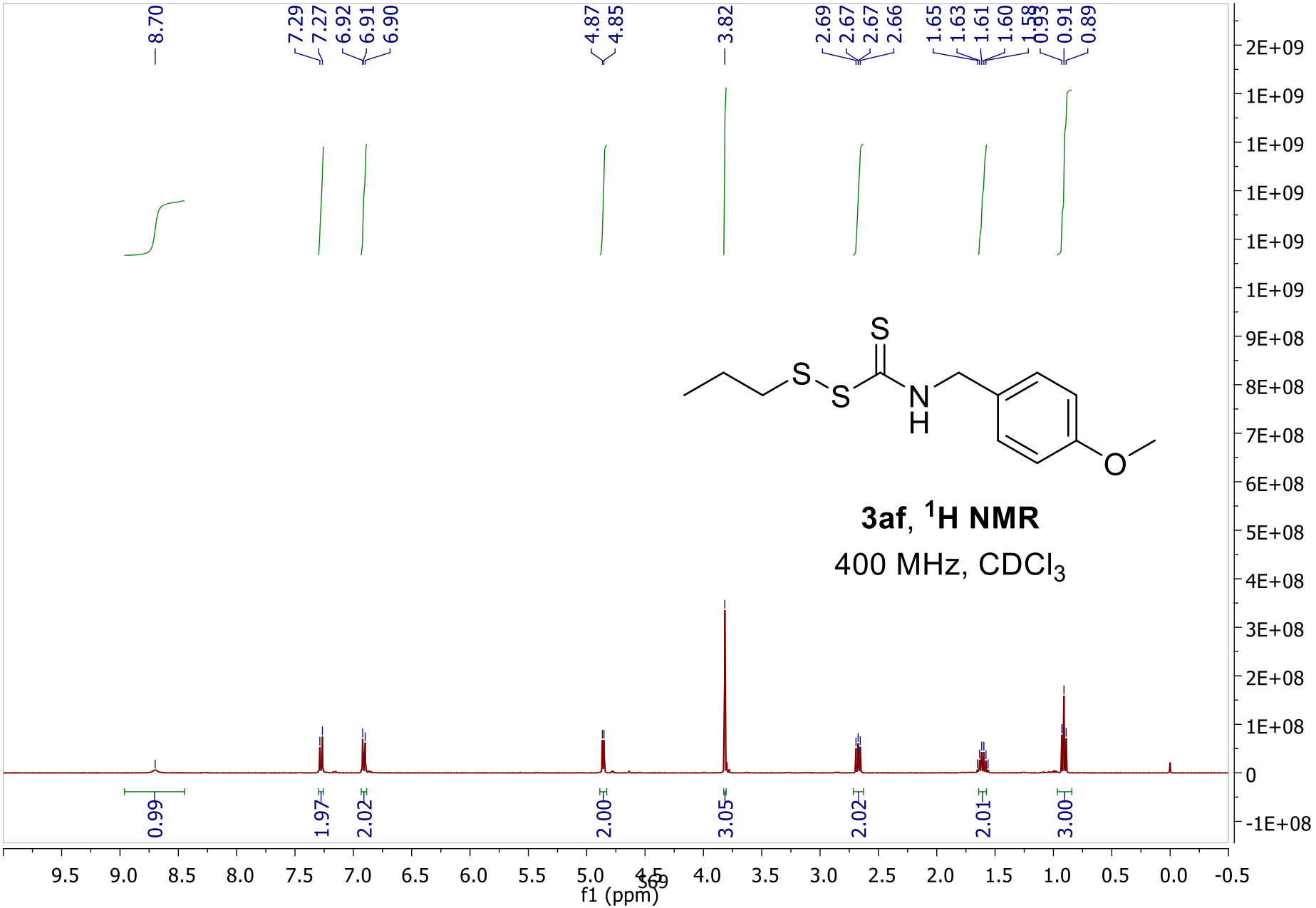
3ae, **^{13}C NMR**

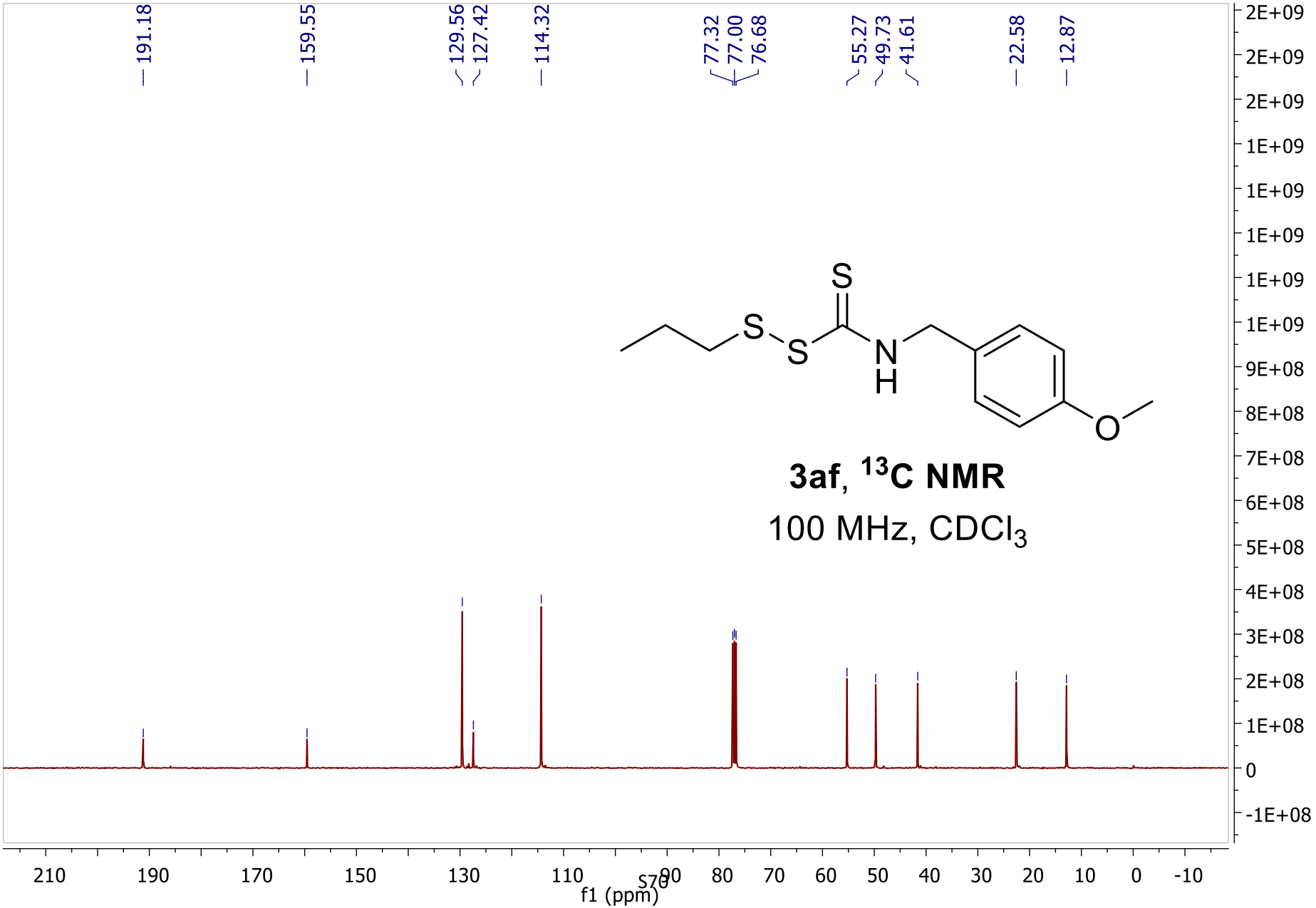
100 MHz, $CDCl_3$

Spectrum Plot Report

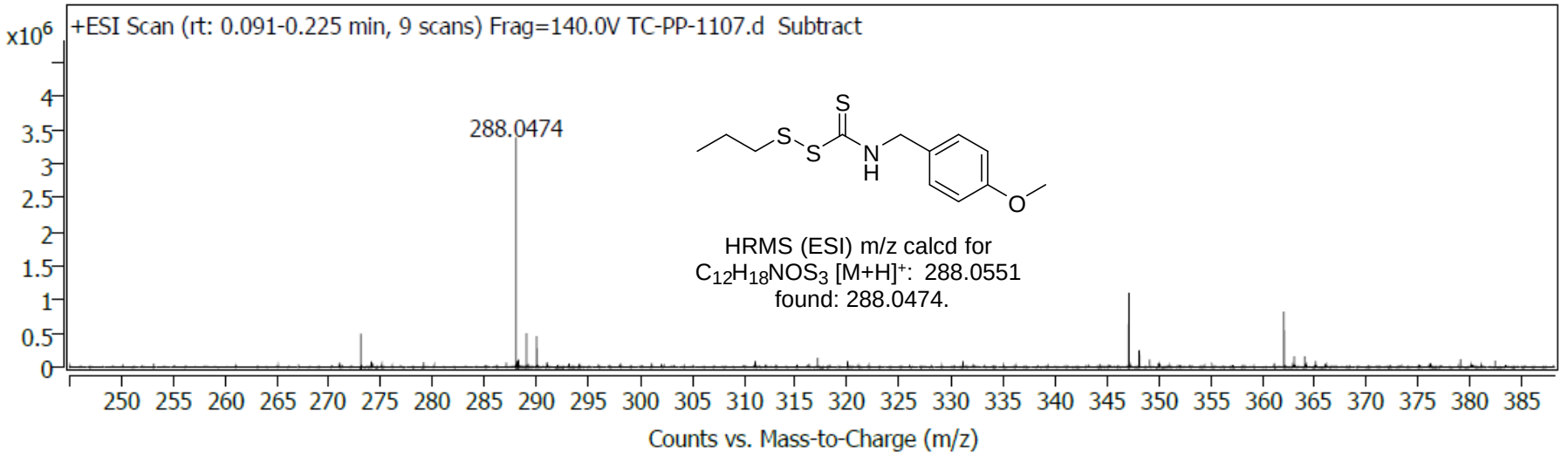


HRMS (ESI) m/z calcd for $C_{11}H_{16}NS_3$ $[M+H]^+$: 258.0445
found: 258.0447.

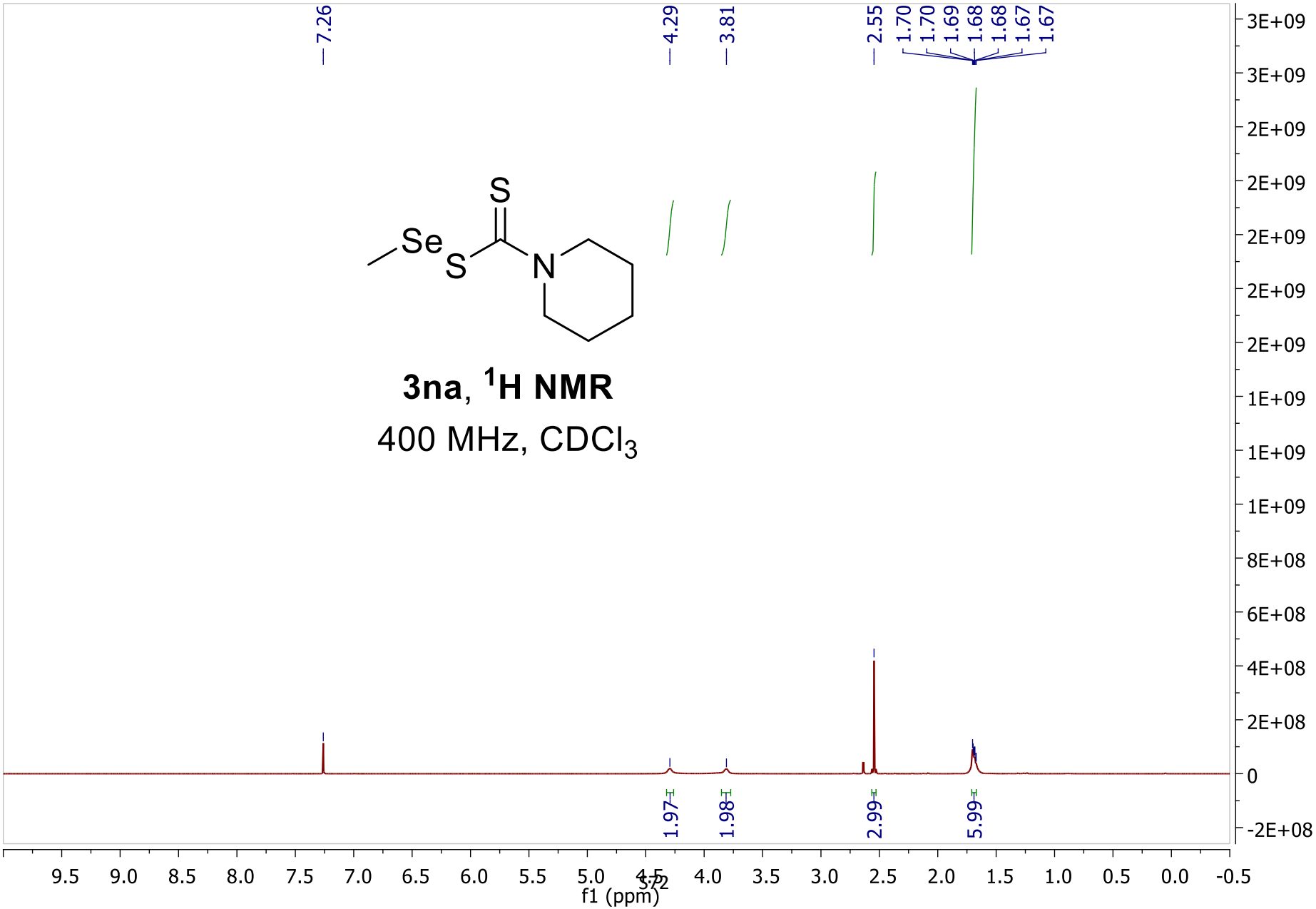

8.70
7.29
7.27
6.92
6.91
6.90
4.87
4.85
3.82
2.69
2.67
2.67
2.66
1.65
1.63
1.61
1.60
1.58
0.93
0.91
0.89
S
S
S
N
H
O
3af, 1H NMR
400 MHz, CDCl3
0.99
1.97
2.02
2.00
3.05
2.02
2.01
3.00
f1 (ppm)

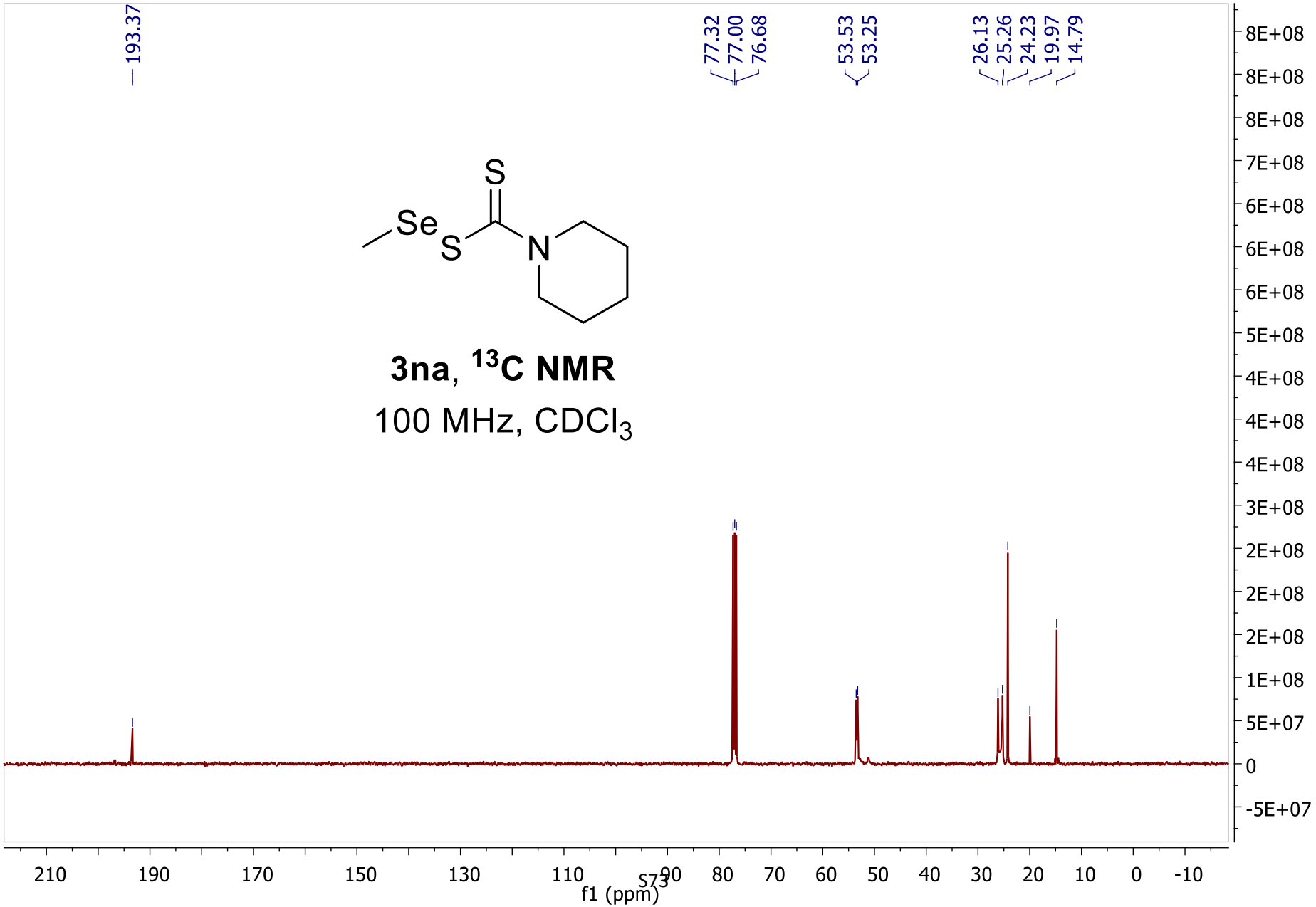

191.18
159.55
129.56
127.42
114.32
77.32
77.00
76.68
55.27
49.73
41.61
22.58
12.87
S
S
S
N
H
O
3af, 13C NMR
100 MHz, CDCl3
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

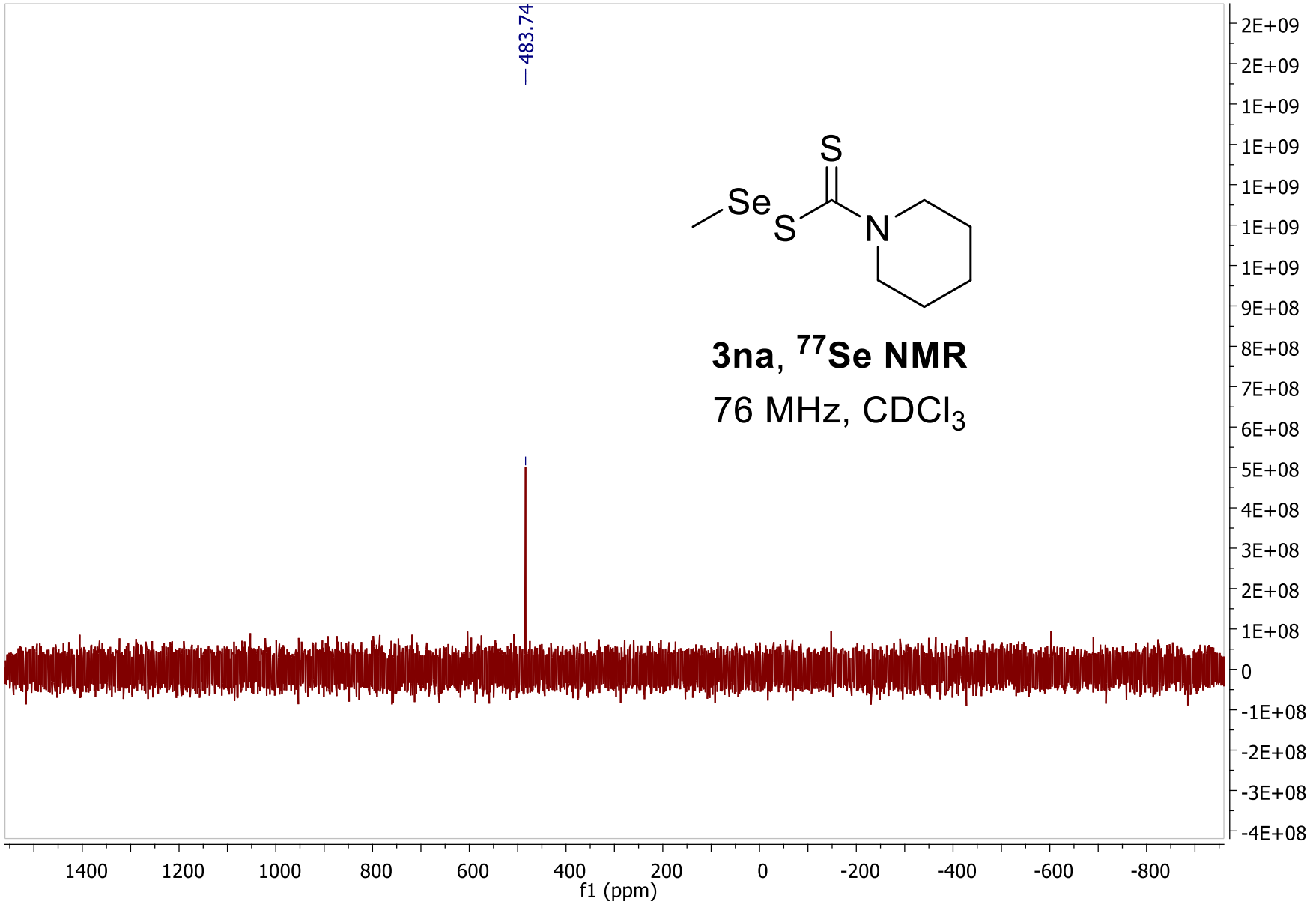
Spectrum Plot Report

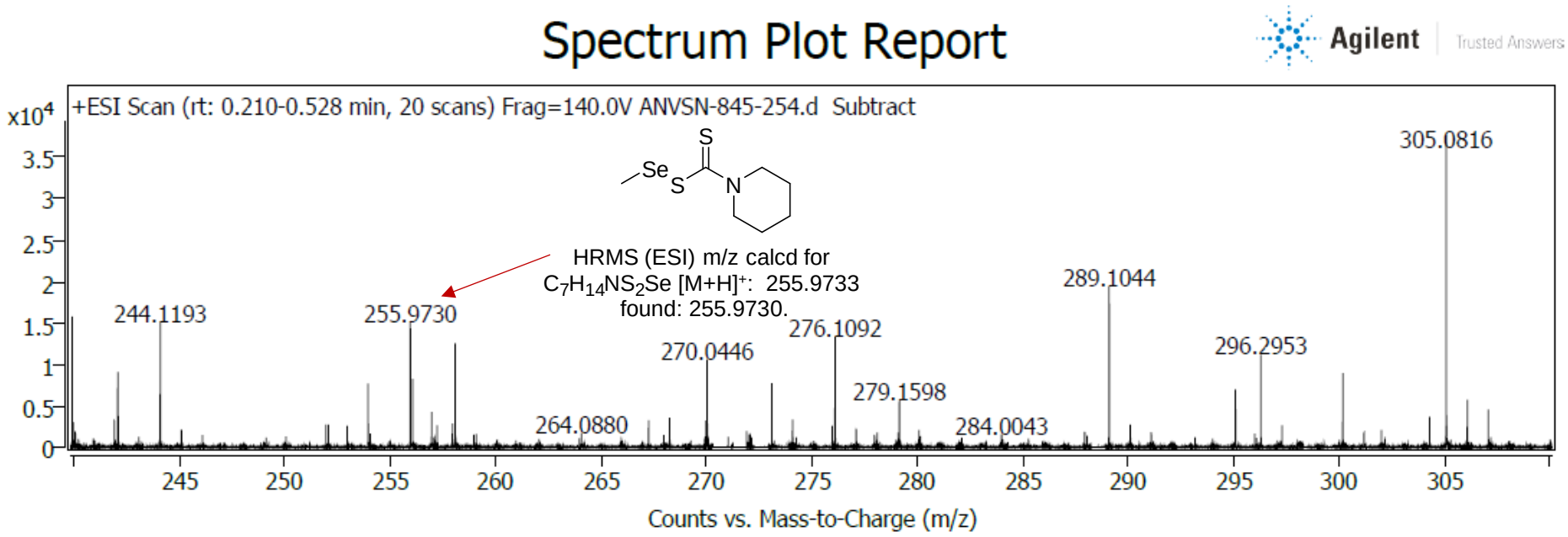


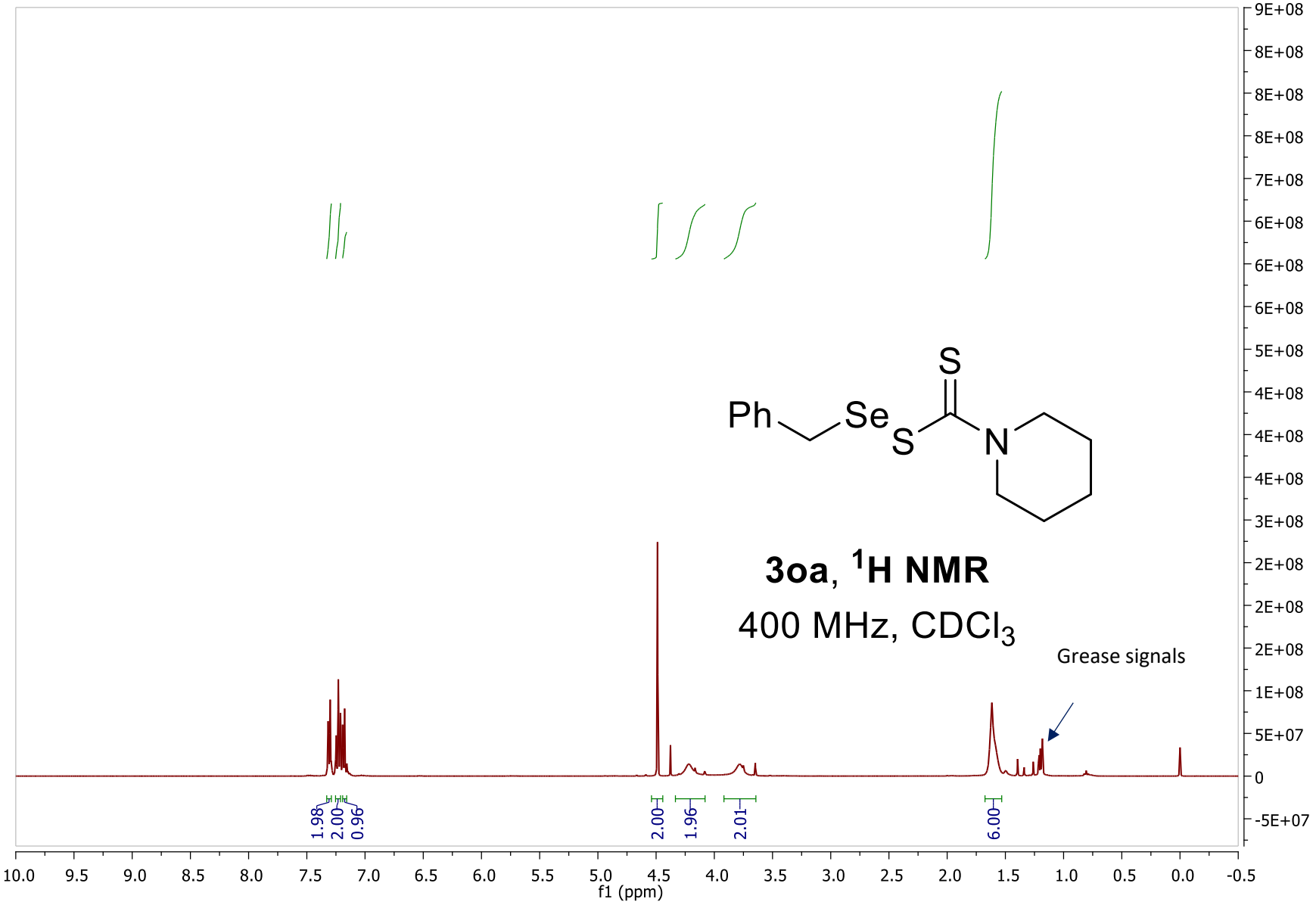
HRMS (ESI) m/z calcd for $C_{12}H_{18}NOS_3$ $[M+H]^+$: 288.0551
found: 288.0474.

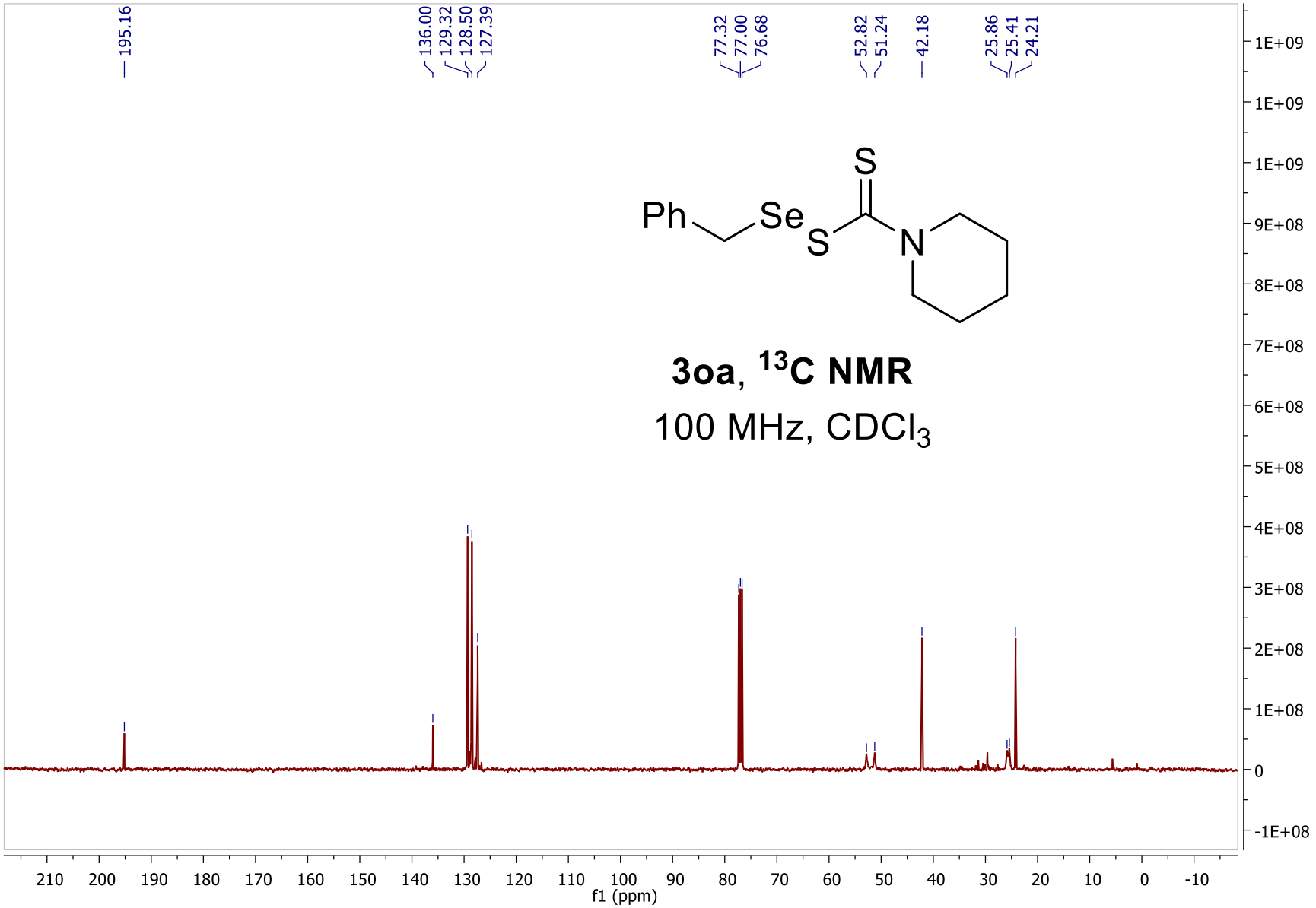

7.26
4.29
3.81
2.55
1.70
1.70
1.69
1.68
1.68
1.67
1.67
S
Se
S
N
3na, 1H NMR
400 MHz, CDCl3
1.97
1.98
2.99
5.99
f1 (ppm)

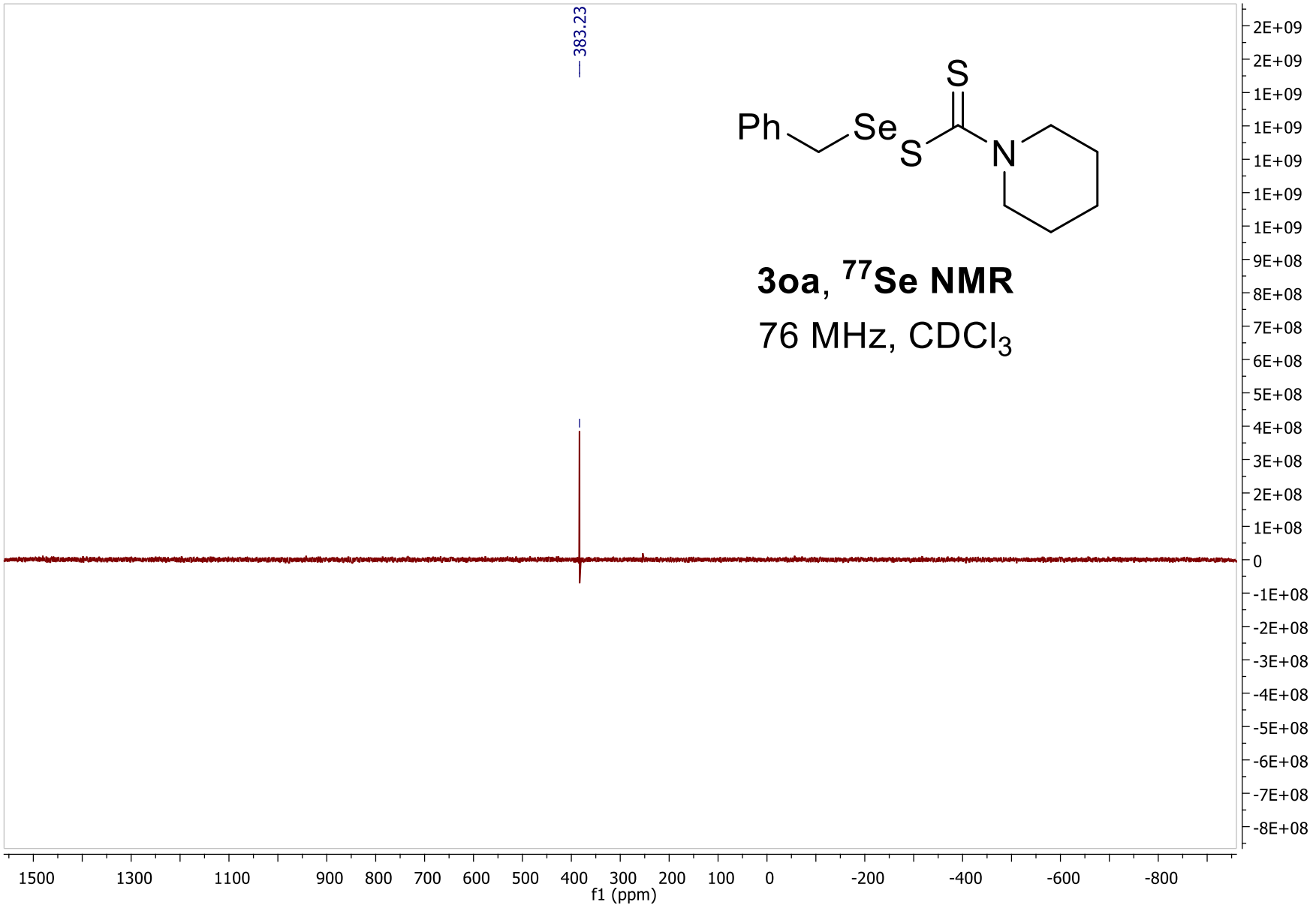

193.37
77.32
77.00
76.68
53.53
53.25
26.13
25.26
24.23
19.97
14.79
S
Se
S
N
3na, 13C NMR
100 MHz, CDCl3
8E+08
8E+08
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

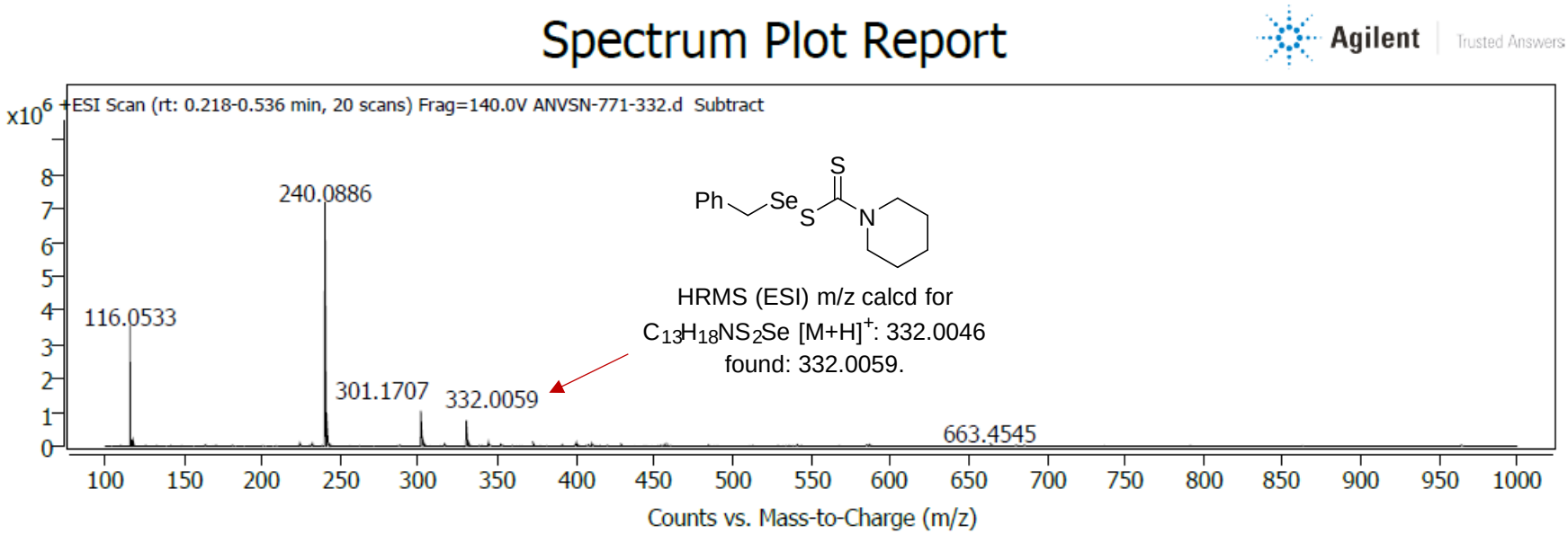

483.74
S
Se
S
N
3na, 77Se NMR
76 MHz, CDCl3
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
-2E+08
-3E+08
-4E+08
1400
1200
1000
800
600
400
200
0
-200
-400
-600
-800
f1 (ppm)

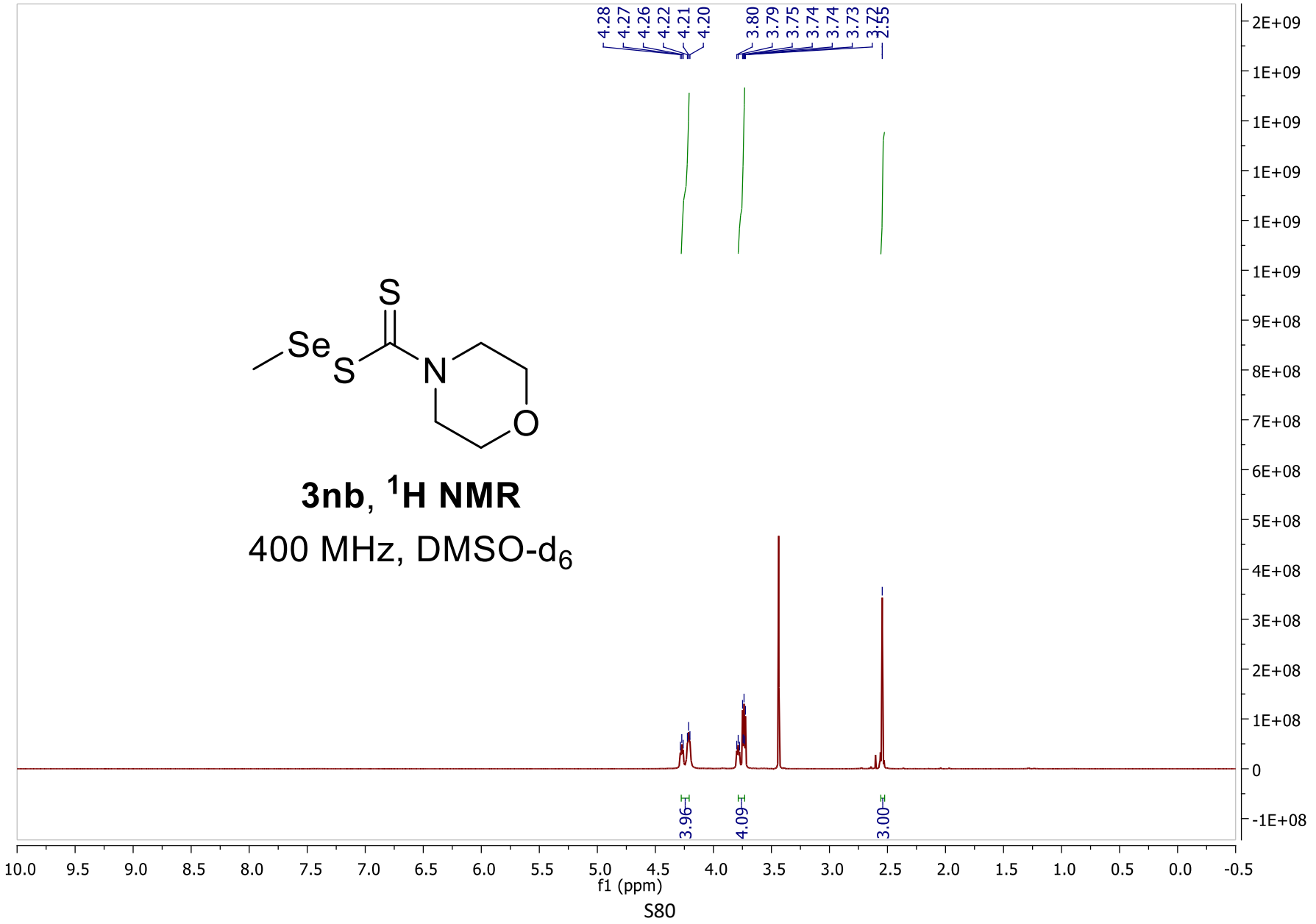

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.210-0.528 min, 20 scans) Frag=140.0V ANVSN-845-254.d Subtract
x10⁴
3.5
3
2.5
2
1.5
1
0.5
0
Se
S
S
N
HRMS (ESI) m/z calcd for
C7H14NS2Se [M+H]+: 255.9733
found: 255.9730.
244.1193
255.9730
264.0880
270.0446
276.1092
279.1598
284.0043
289.1044
296.2953
305.0816
245
250
255
260
265
270
275
280
285
290
295
300
305
Counts vs. Mass-to-Charge (m/z)

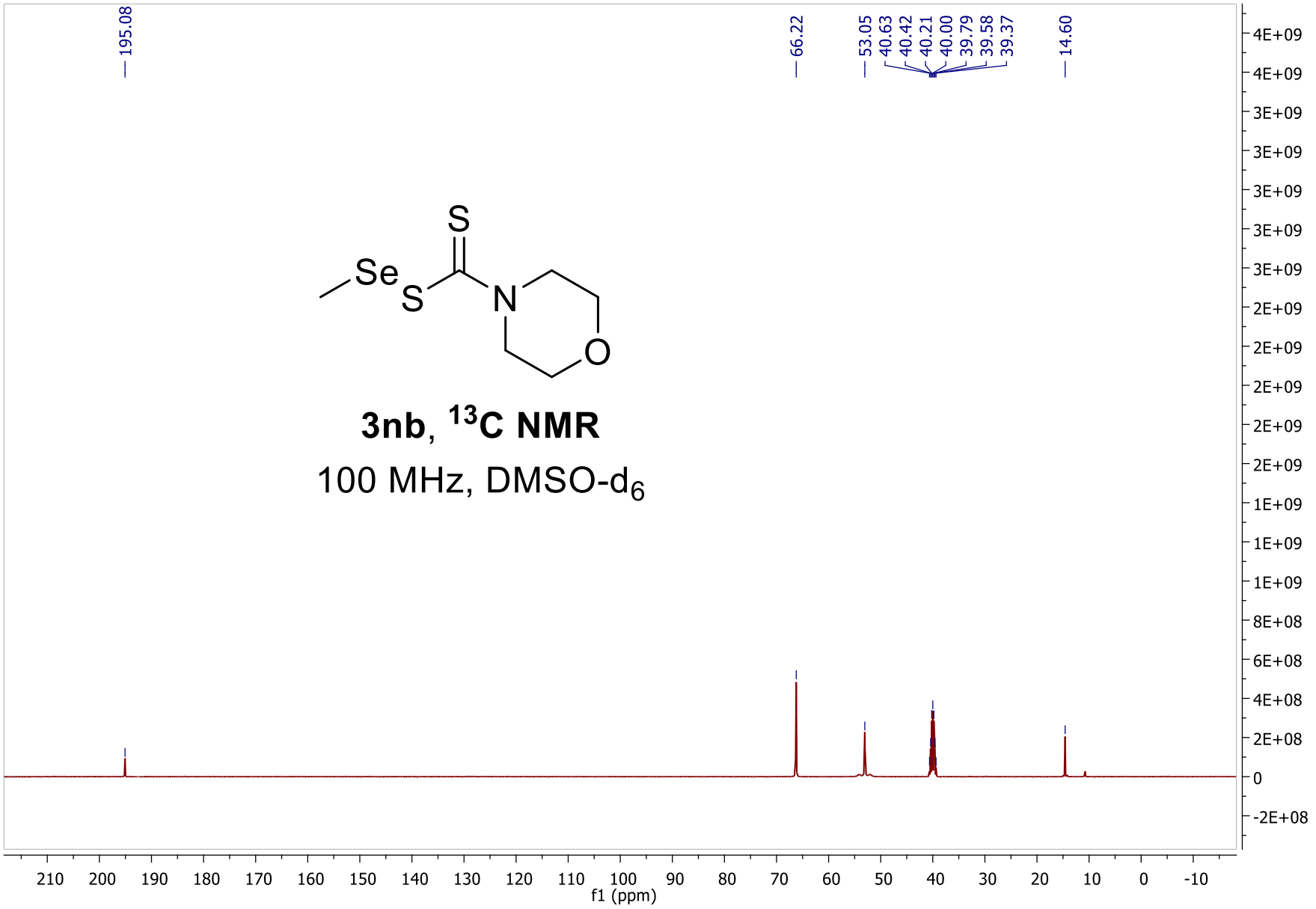

Ph
Se
S
S
N
3oa, 1H NMR
400 MHz, CDCl3
Grease signals
1.98
2.00
0.96
2.00
1.96
2.01
6.00
f1 (ppm)

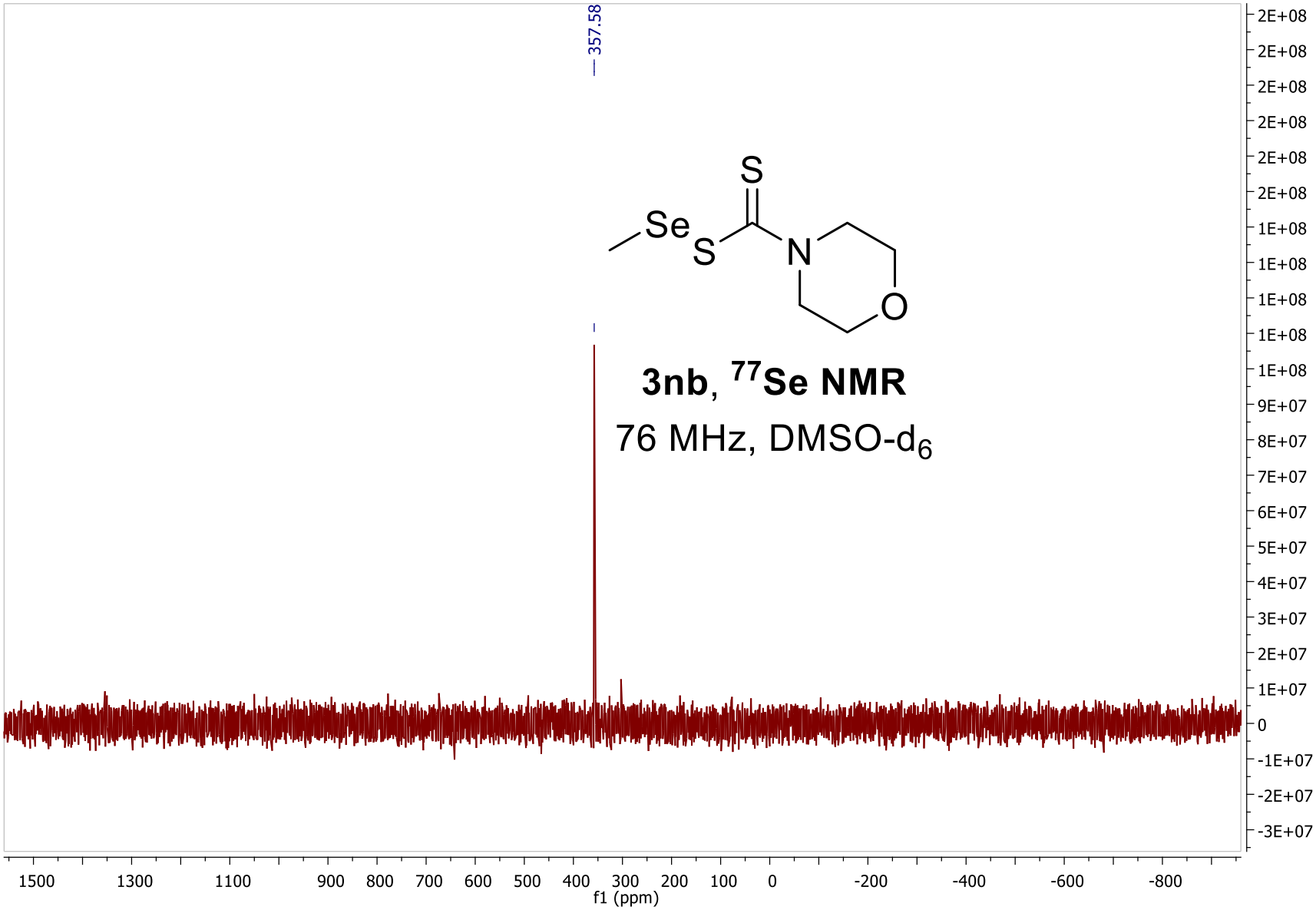

195.16
136.00
129.32
128.50
127.39
77.32
77.00
76.68
52.82
51.24
42.18
25.86
25.41
24.21
Ph
Se
S
S
N
3oa, 13C NMR
100 MHz, CDCl3
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

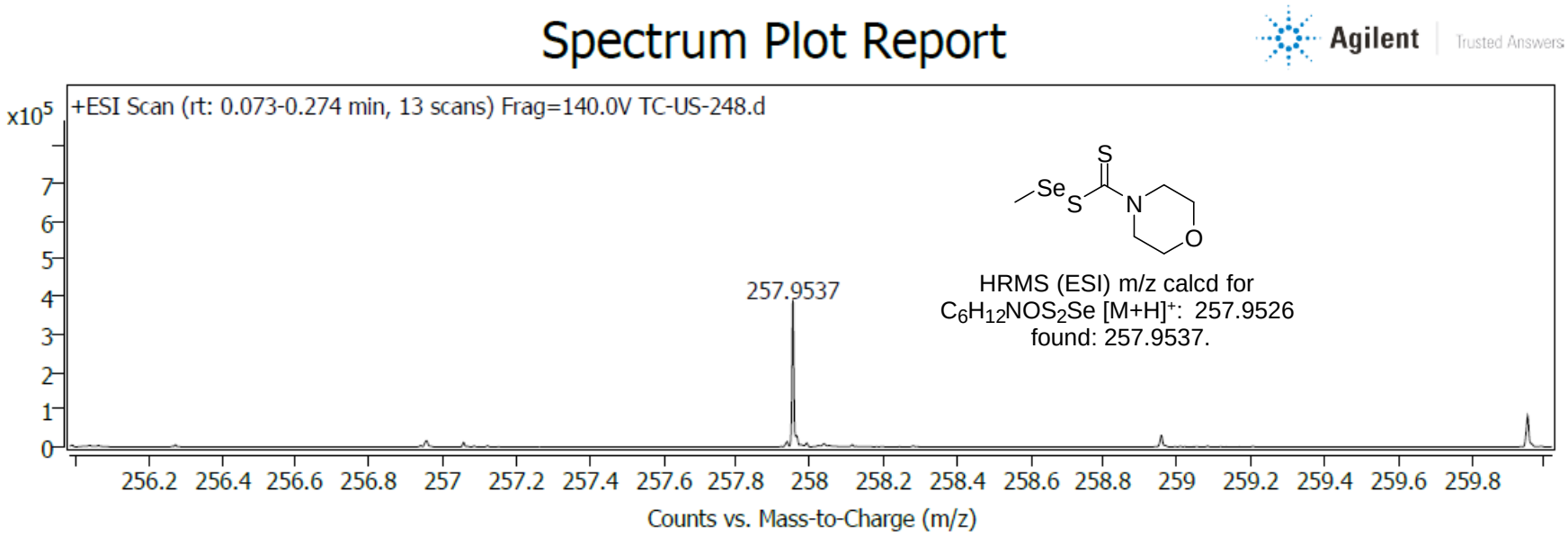

383.23
Ph
Se
S
S
N
3oa, 77Se NMR
76 MHz, CDCl3
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
-2E+08
-3E+08
-4E+08
-5E+08
-6E+08
-7E+08
-8E+08
1500
1300
1100
900
800
700
600
500
400
300
200
100
0
-200
-400
-600
-800
f1 (ppm)

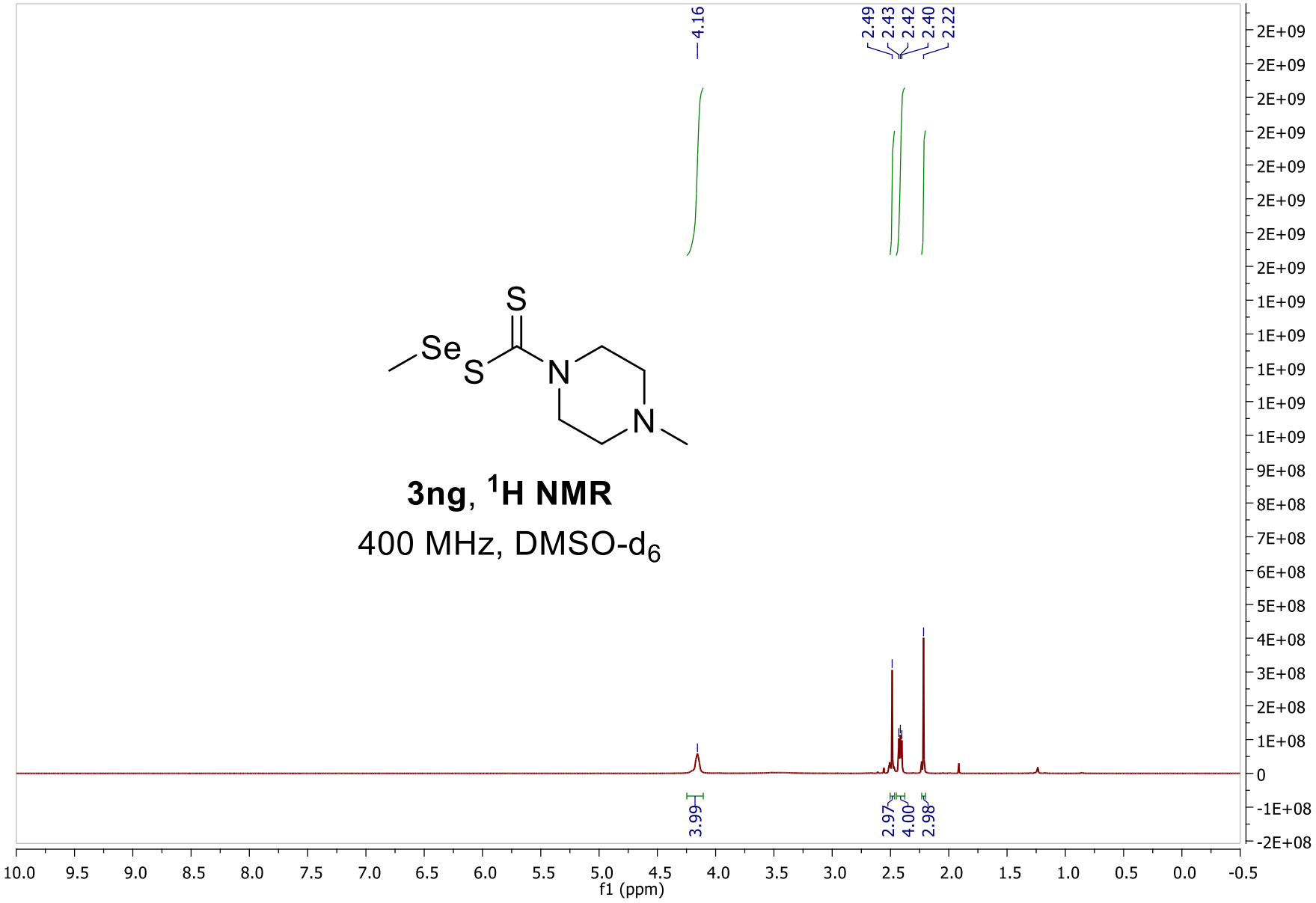

Spectrum Plot Report
Agilent
Trusted Answers
x10^6
ESI Scan (rt: 0.218-0.536 min, 20 scans) Frag=140.0V ANVSN-771-332.d Subtract
240.0886
116.0533
301.1707
332.0059
663.4545
Ph
Se
S
S
N
HRMS (ESI) m/z calcd for
C13H18NS2Se [M+H]+: 332.0046
found: 332.0059.
Counts vs. Mass-to-Charge (m/z)

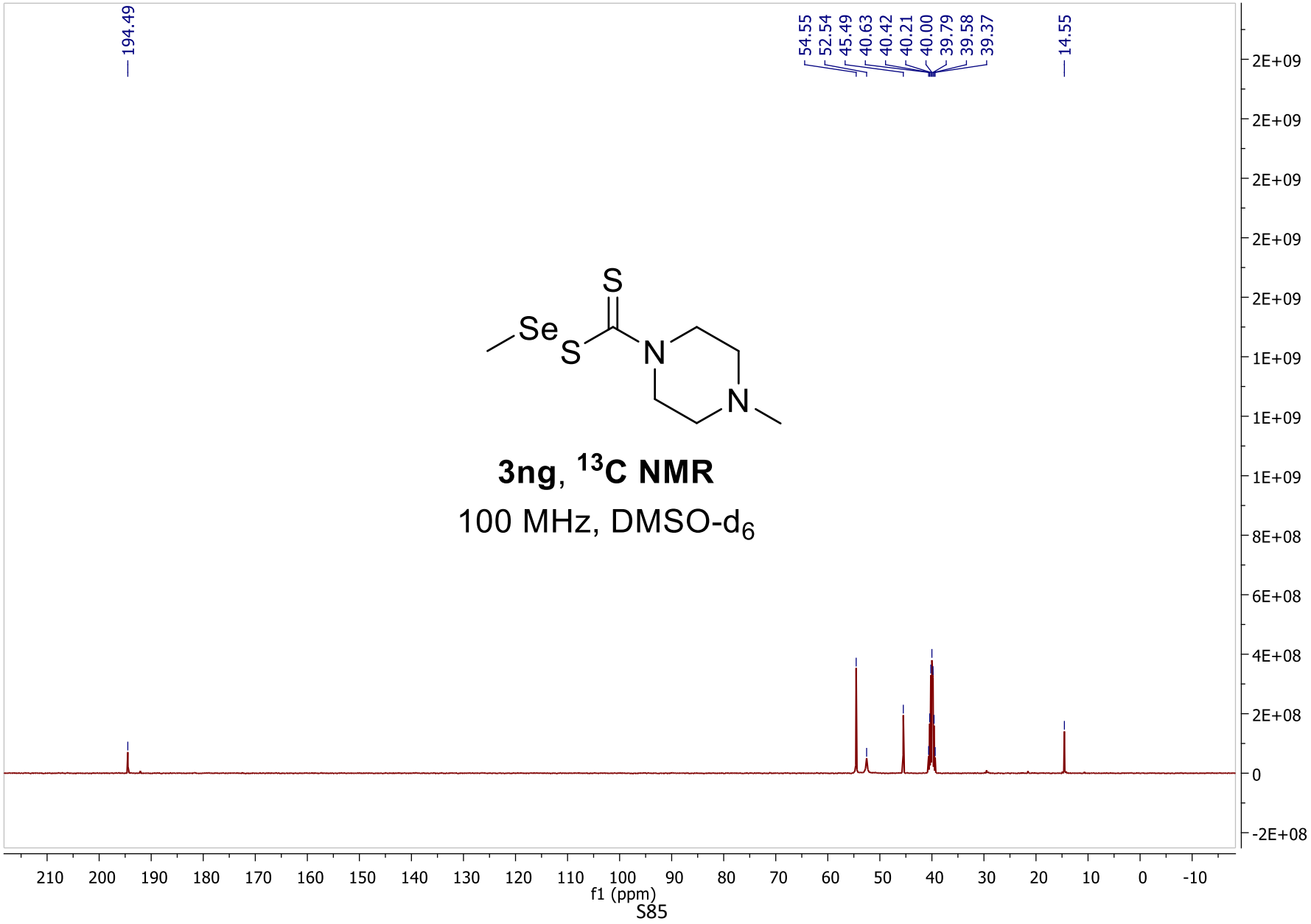

4.28
4.27
4.26
4.22
4.21
4.20
3.80
3.79
3.75
3.74
3.74
3.73
3.72
2.55
S
Se
S
N
O
3nb, 1H NMR
400 MHz, DMSO-d6
3.96
4.09
3.00
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

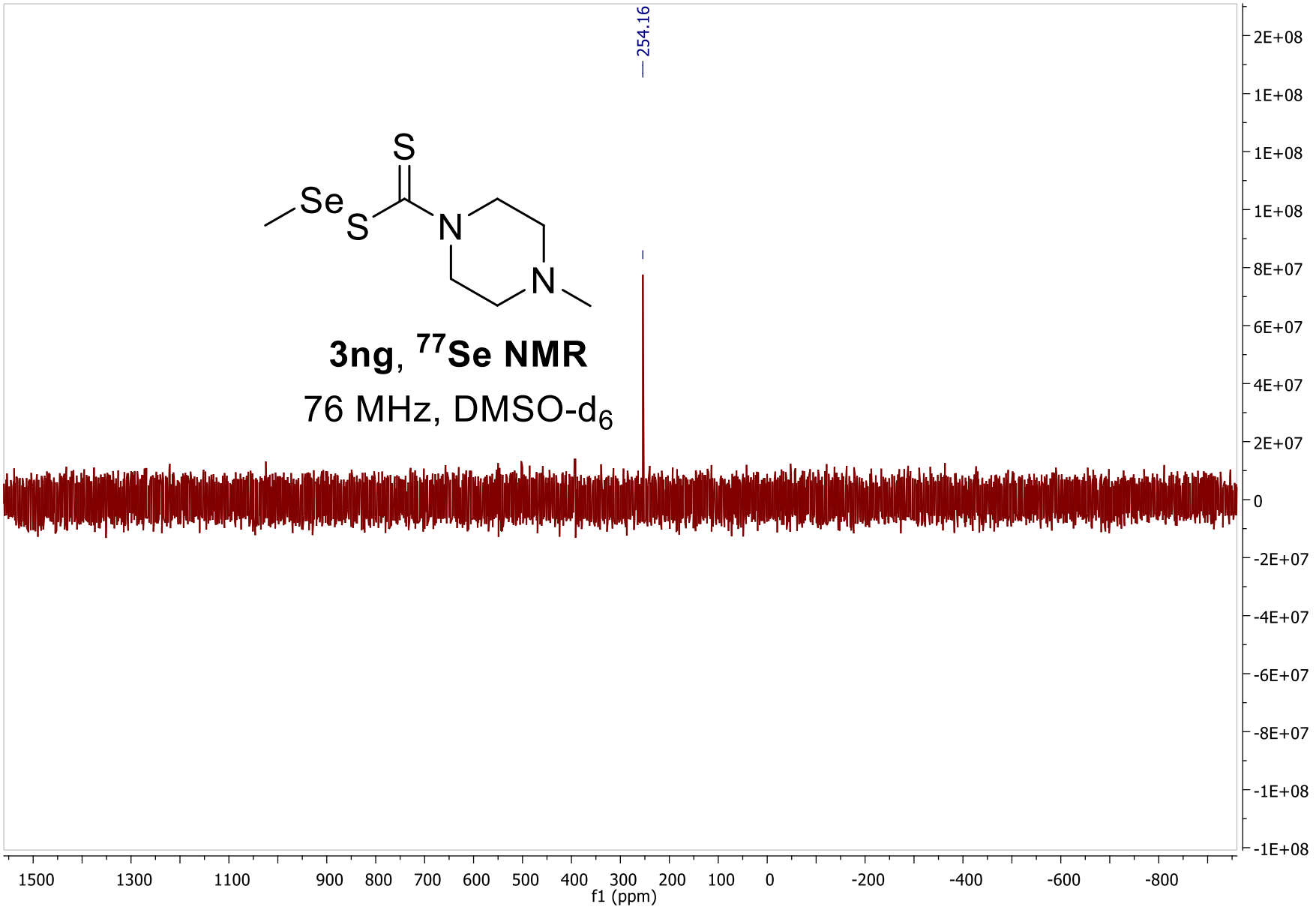

195.08
66.22
53.05
40.63
40.42
40.21
40.00
39.79
39.58
39.37
14.60
S
Se
S
N
O
3nb, 13C NMR
100 MHz, DMSO-d6
4E+09
4E+09
3E+09
3E+09
3E+09
3E+09
3E+09
2E+09
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
8E+08
6E+08
4E+08
2E+08
0
-2E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

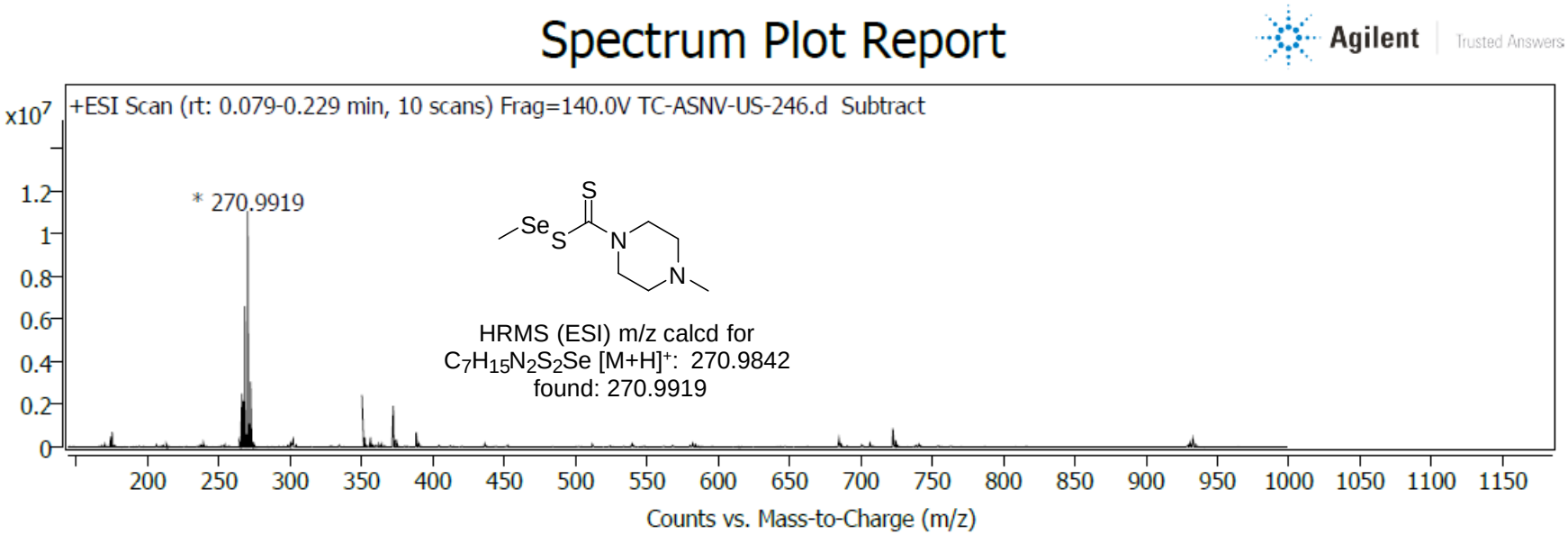

357.58
S
Se
S
N
O
3nb, 77Se NMR
76 MHz, DMSO-d6
2E+08
2E+08
2E+08
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
1E+08
1E+08
1E+08
9E+07
8E+07
7E+07
6E+07
5E+07
4E+07
3E+07
2E+07
1E+07
0
-1E+07
-2E+07
-3E+07
1500
1300
1100
900
800
700
600
500
400
300
200
100
0
-200
-400
-600
-800
f1 (ppm)

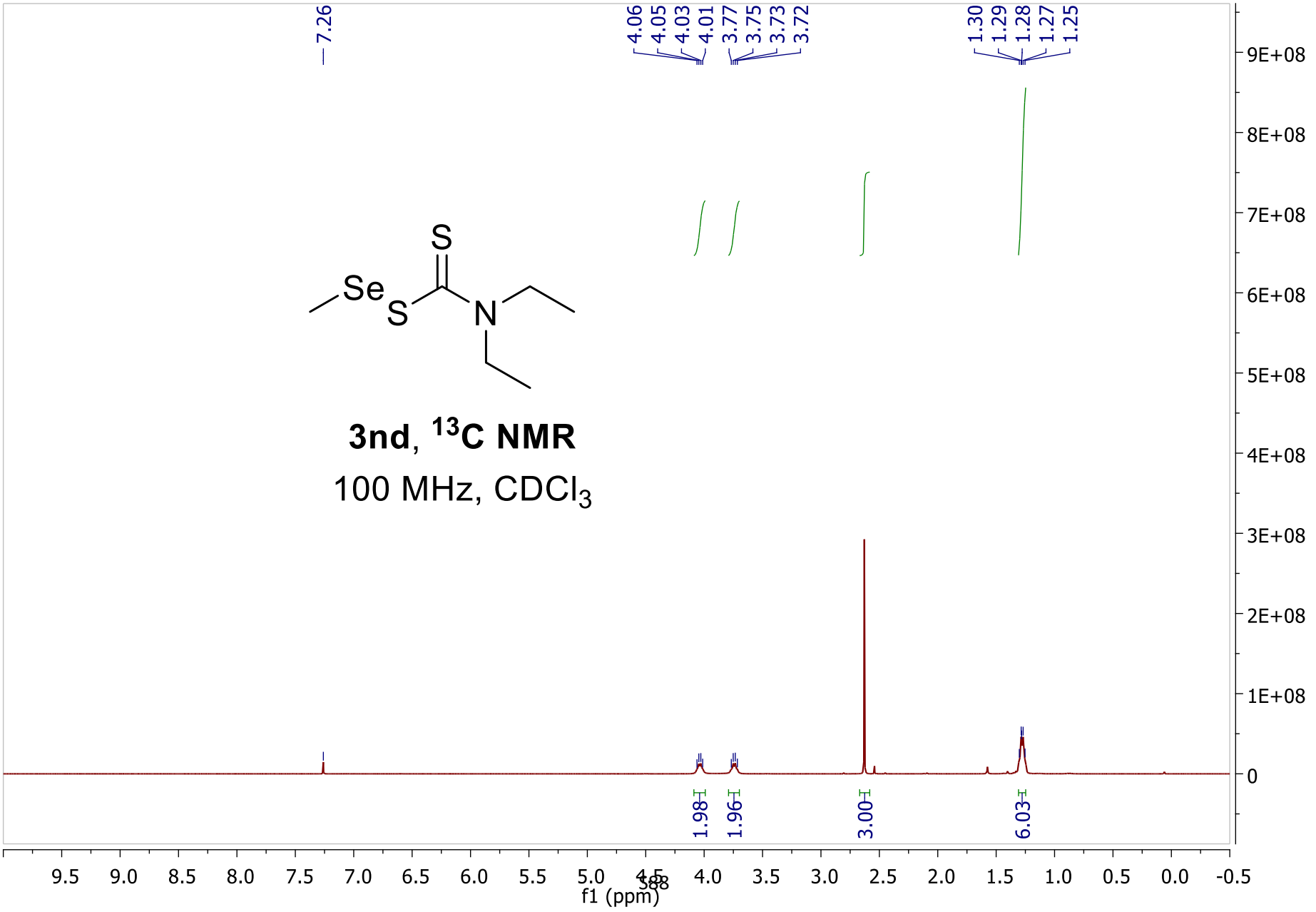

Spectrum Plot Report
Agilent
Trusted Answers
x10^5
+ESI Scan (rt: 0.073-0.274 min, 13 scans) Frag=140.0V TC-US-248.d
257.9537
HRMS (ESI) m/z calcd for
C6H12NOS2Se [M+H]+: 257.9526
found: 257.9537.
Counts vs. Mass-to-Charge (m/z)

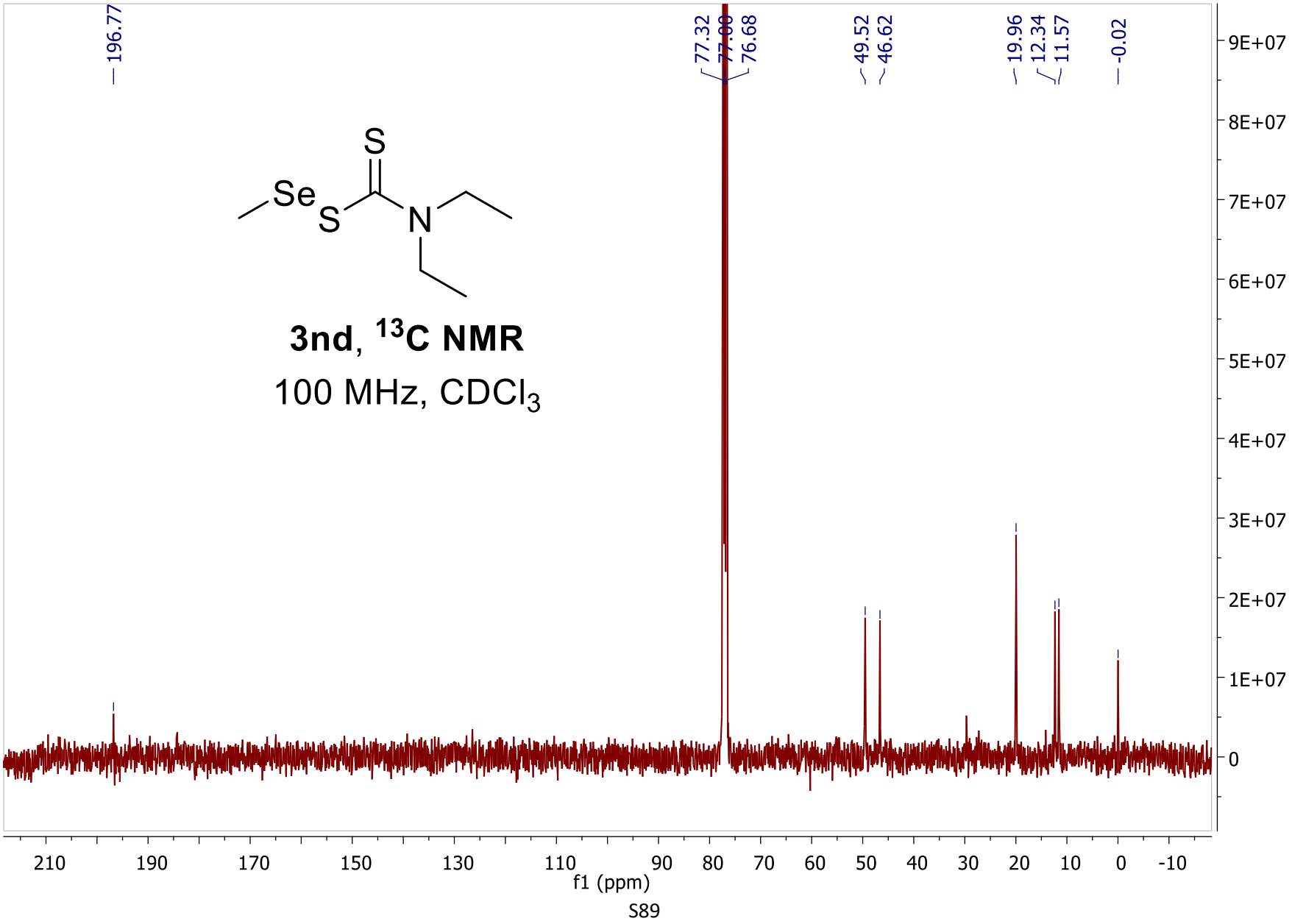

4.16
2.49
2.43
2.42
2.40
2.22
S
Se
S
N
N
3ng, 1H NMR
400 MHz, DMSO-d6
3.99
2.97
4.00
2.98
f1 (ppm)

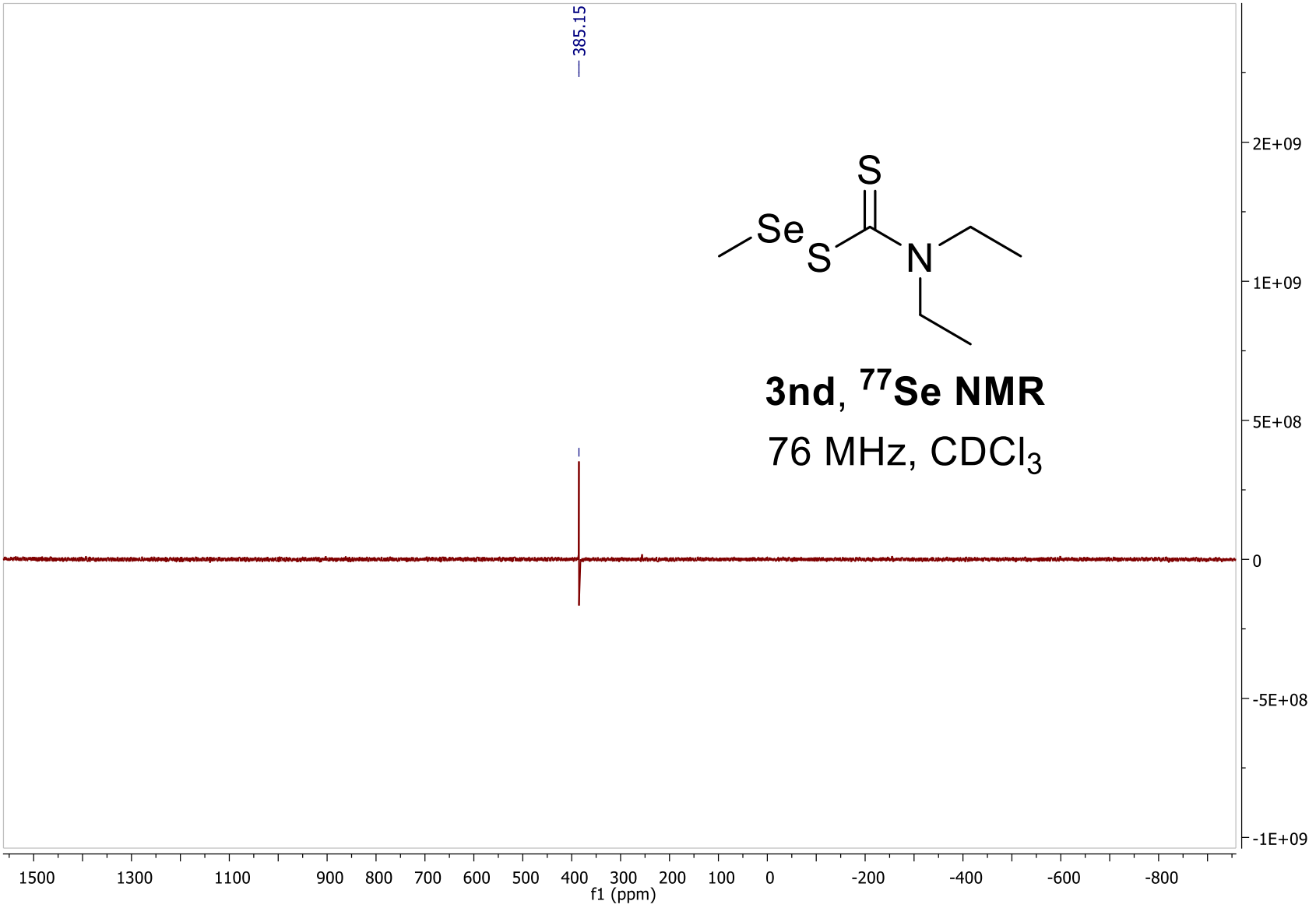

194.49
54.55
52.54
45.49
40.63
40.42
40.21
40.00
39.79
39.58
39.37
14.55
Se
S
S
N
N
3ng, 13C NMR
100 MHz, DMSO-d6
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
8E+08
6E+08
4E+08
2E+08
0
-2E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

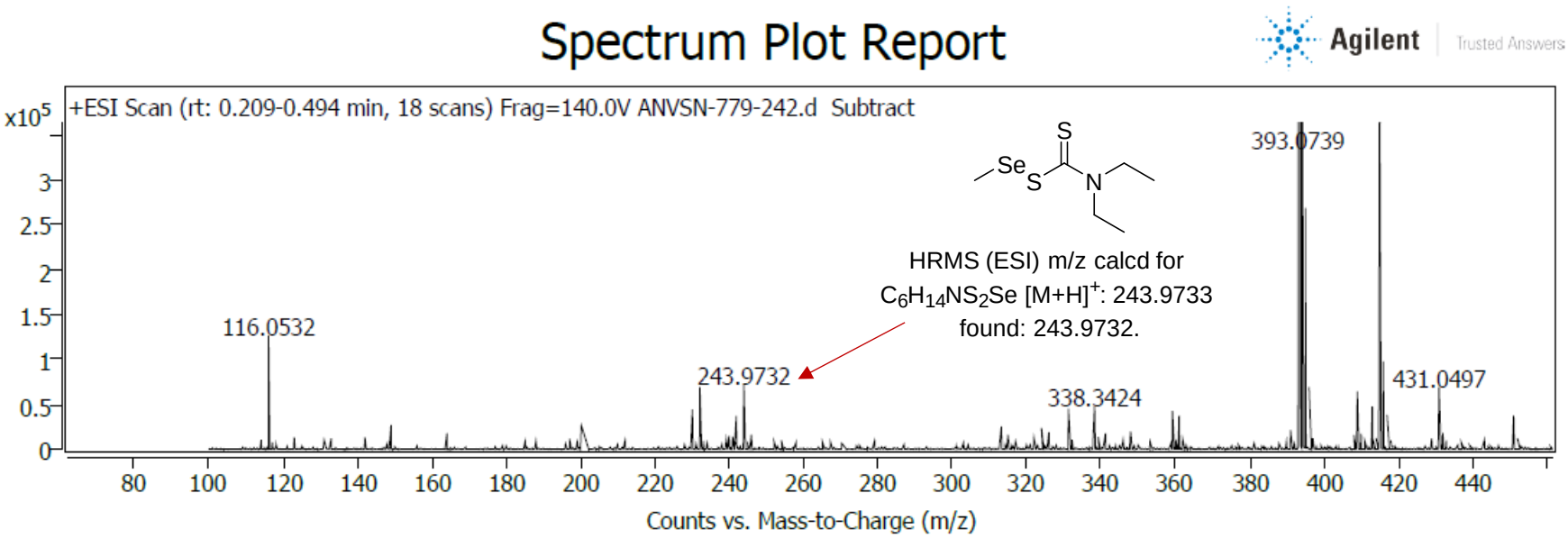

254.16
S
Se
S
N
N
3ng, 77Se NMR
76 MHz, DMSO-d6
2E+08
1E+08
1E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
-4E+07
-6E+07
-8E+07
-1E+08
-1E+08
1500
1300
1100
900
800
700
600
500
400
300
200
100
0
-200
-400
-600
-800
f1 (ppm)

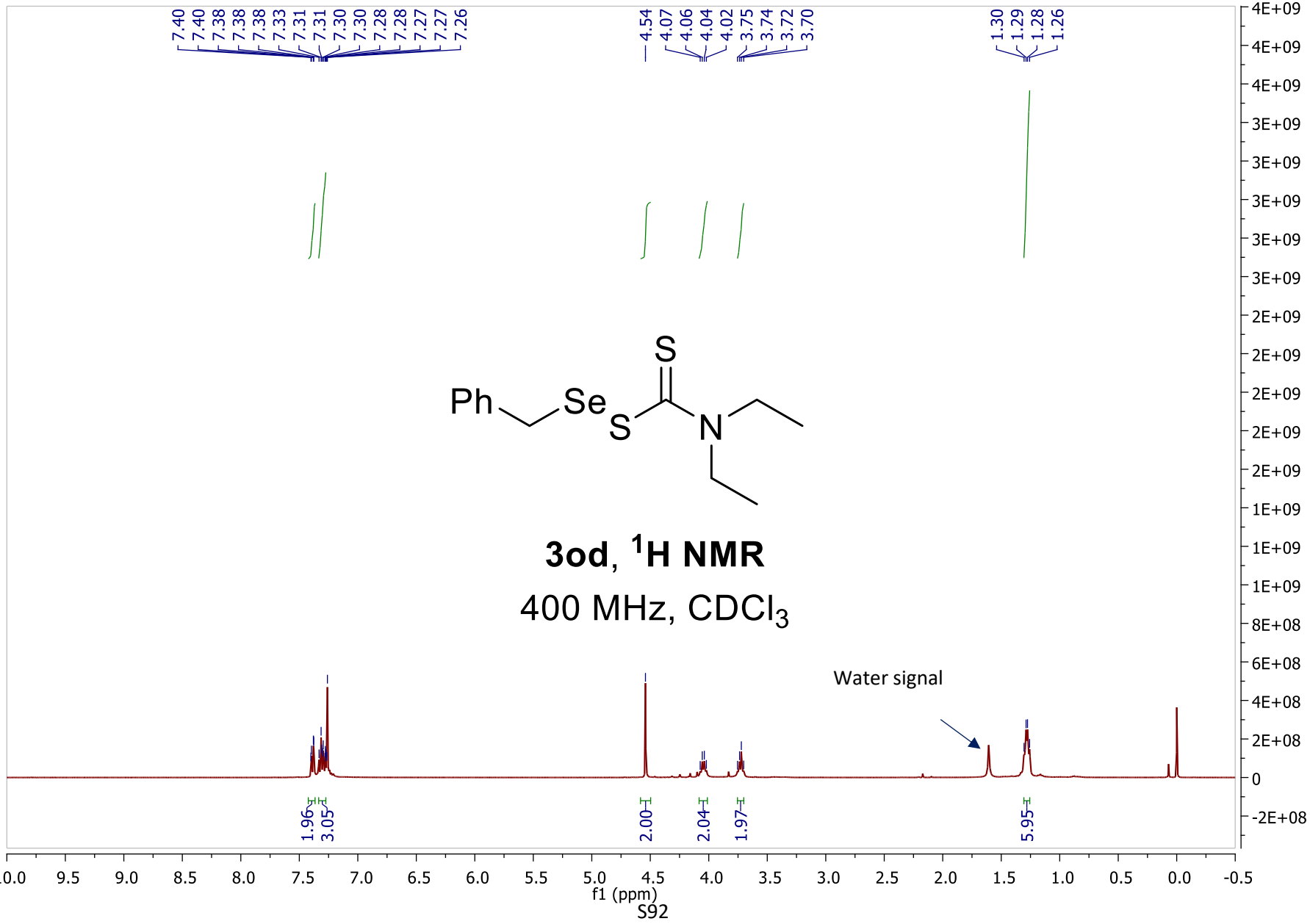

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.079-0.229 min, 10 scans) Frag=140.0V TC-ASNV-US-246.d Subtract
x10^7
* 270.9919
HRMS (ESI) m/z calcd for
C7H15N2S2Se [M+H]+: 270.9842
found: 270.9919
Counts vs. Mass-to-Charge (m/z)

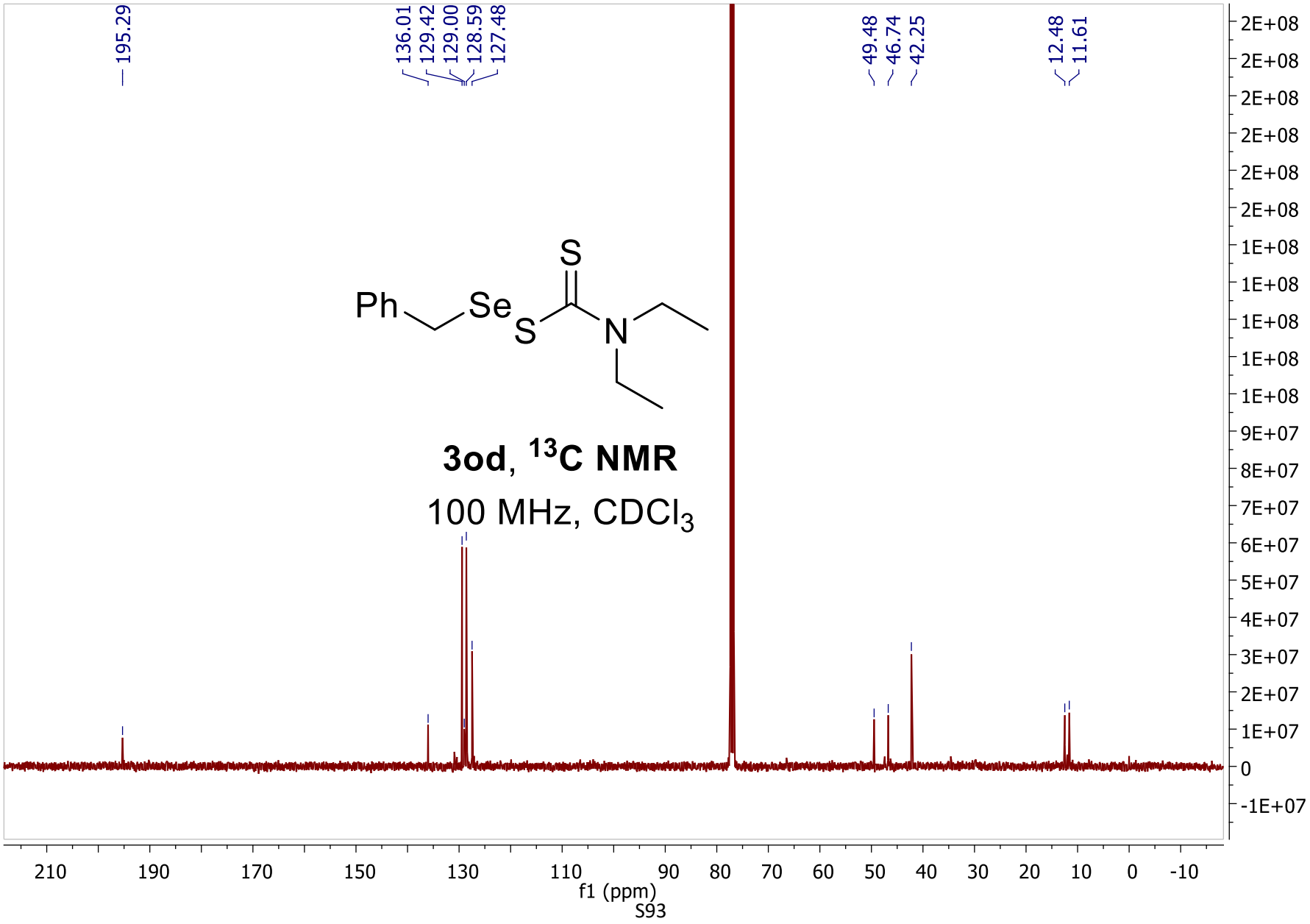

7.26
4.06
4.05
4.03
4.01
3.77
3.75
3.73
3.72
1.30
1.29
1.28
1.27
1.25
S
Se
S
N
3nd, 13C NMR
100 MHz, CDCl3
1.98
1.96
3.00
6.03
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

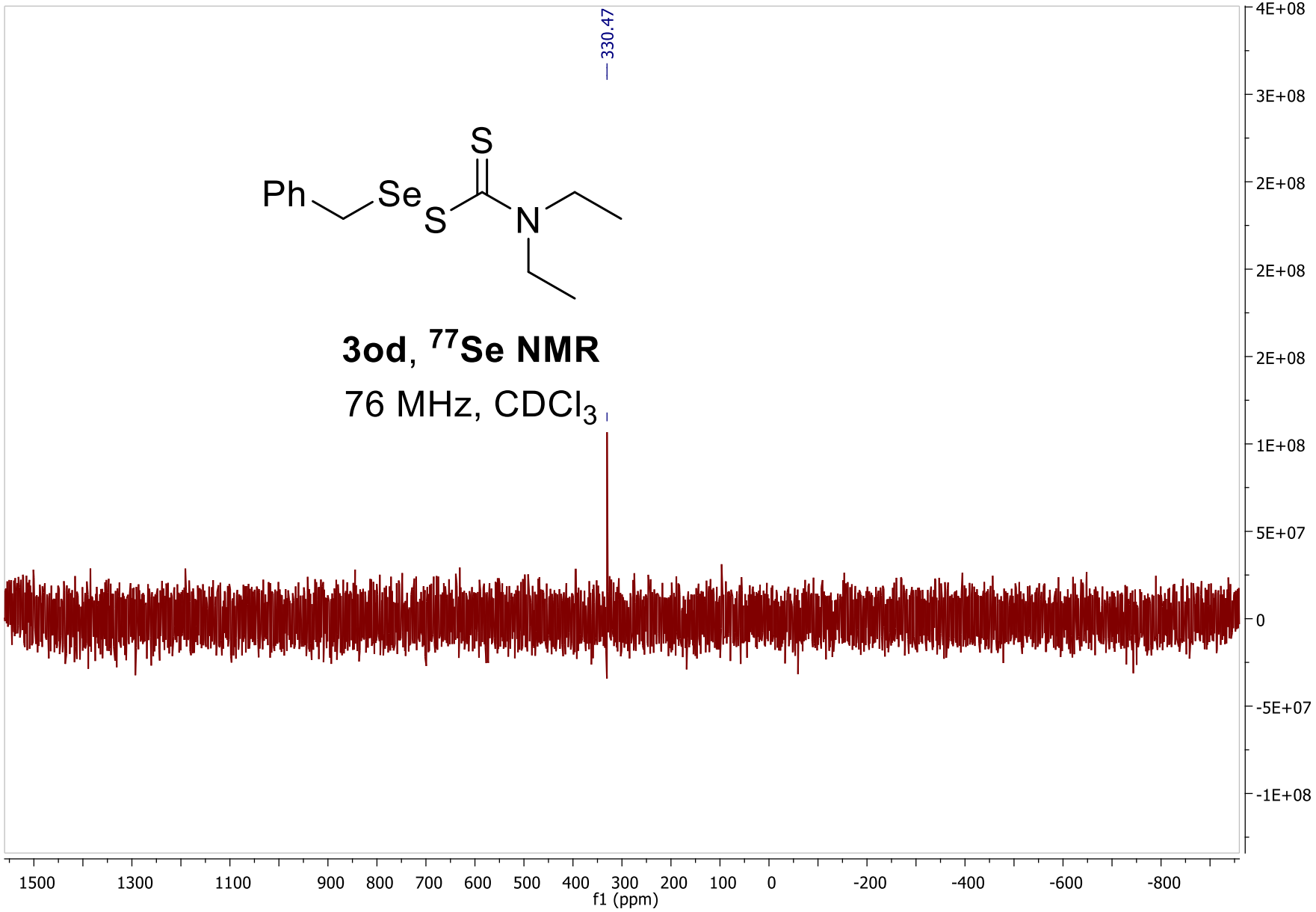

196.77
77.32
77.00
76.68
49.52
46.62
19.96
12.34
11.57
-0.02
Se
S
S
N
3nd, 13C NMR
100 MHz, CDCl3
9E+07
8E+07
7E+07
6E+07
5E+07
4E+07
3E+07
2E+07
1E+07
0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

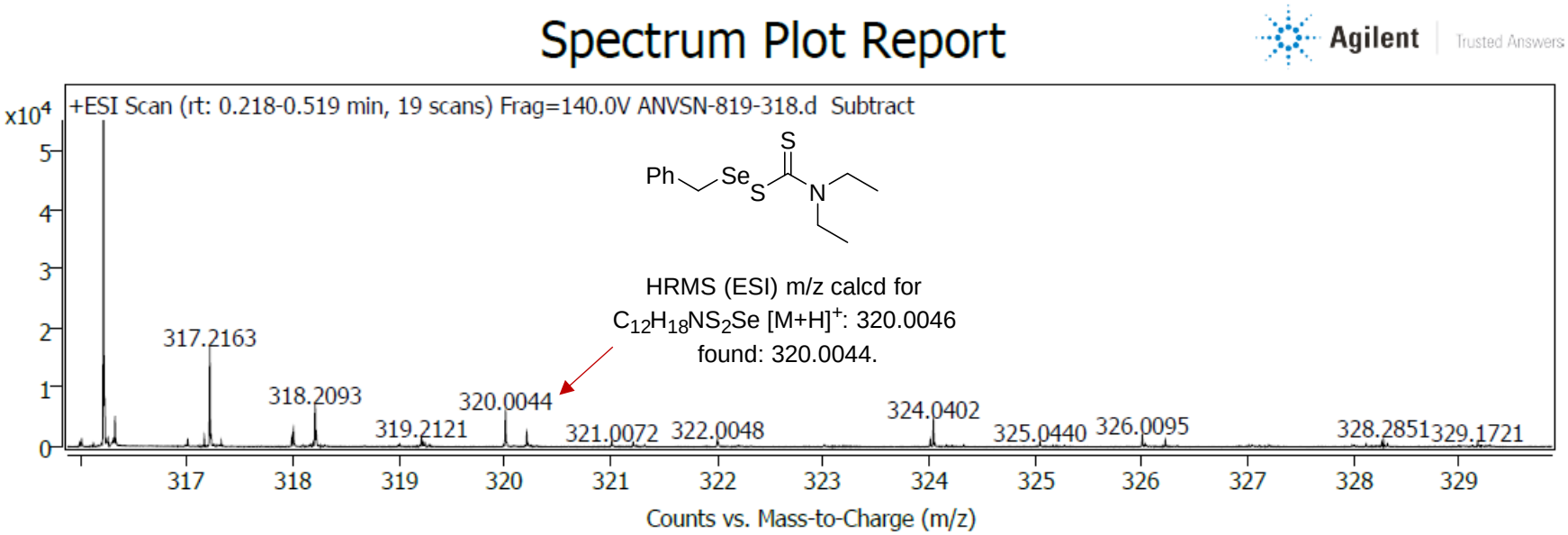

385.15
S
Se
S
N
3nd, 77Se NMR
76 MHz, CDCl3
2E+09
1E+09
5E+08
0
-5E+08
-1E+09
1500
1300
1100
900
800
700
600
500
400
300
200
100
0
-200
-400
-600
-800
f1 (ppm)

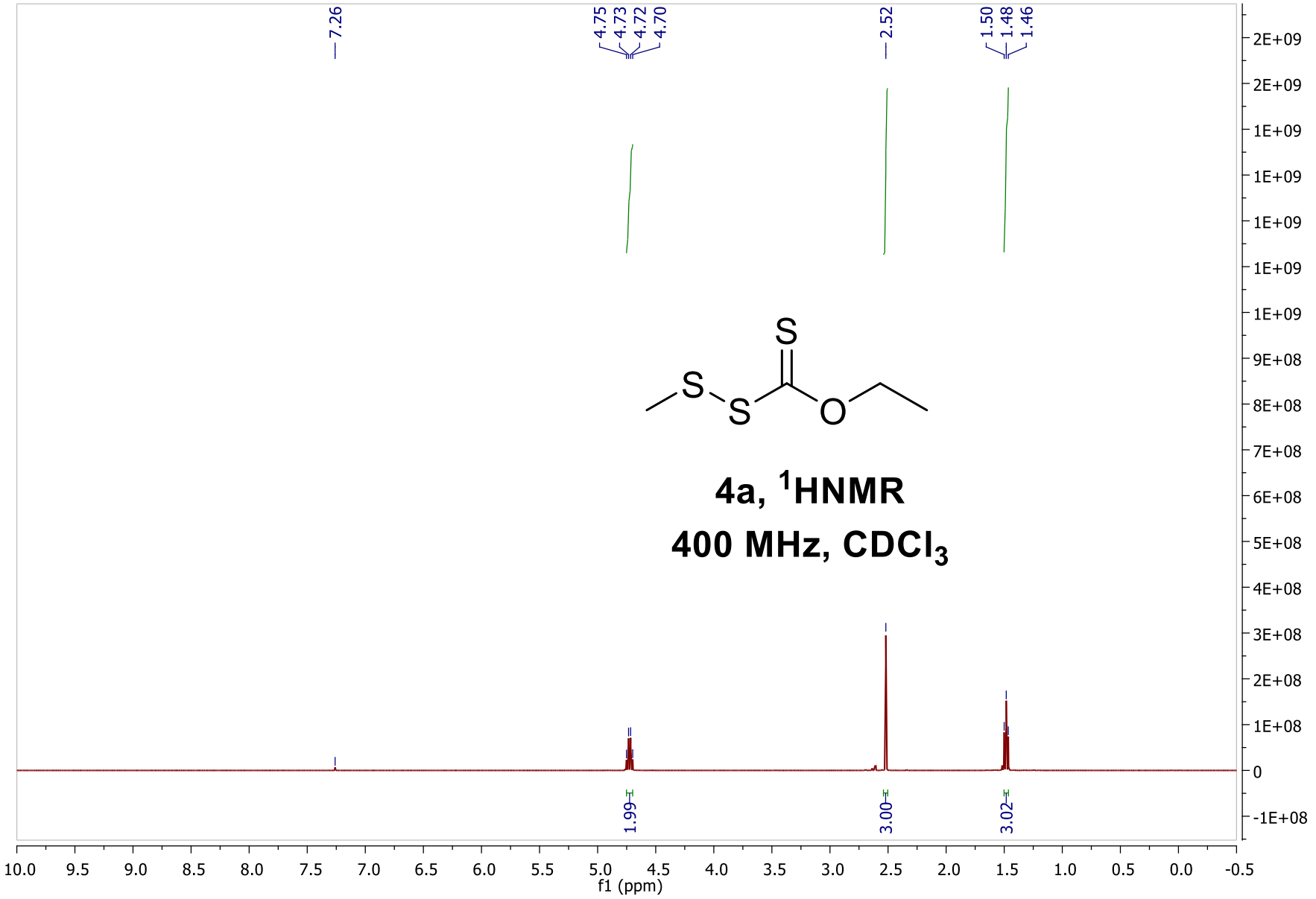

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.209-0.494 min, 18 scans) Frag=140.0V ANVSN-779-242.d Subtract
x10^5
3
2.5
2
1.5
1
0.5
0
116.0532
243.9732
338.3424
393.0739
431.0497
Se
S
S
N
HRMS (ESI) m/z calcd for
C6H14NS2Se [M+H]+: 243.9733
found: 243.9732.
80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440
Counts vs. Mass-to-Charge (m/z)

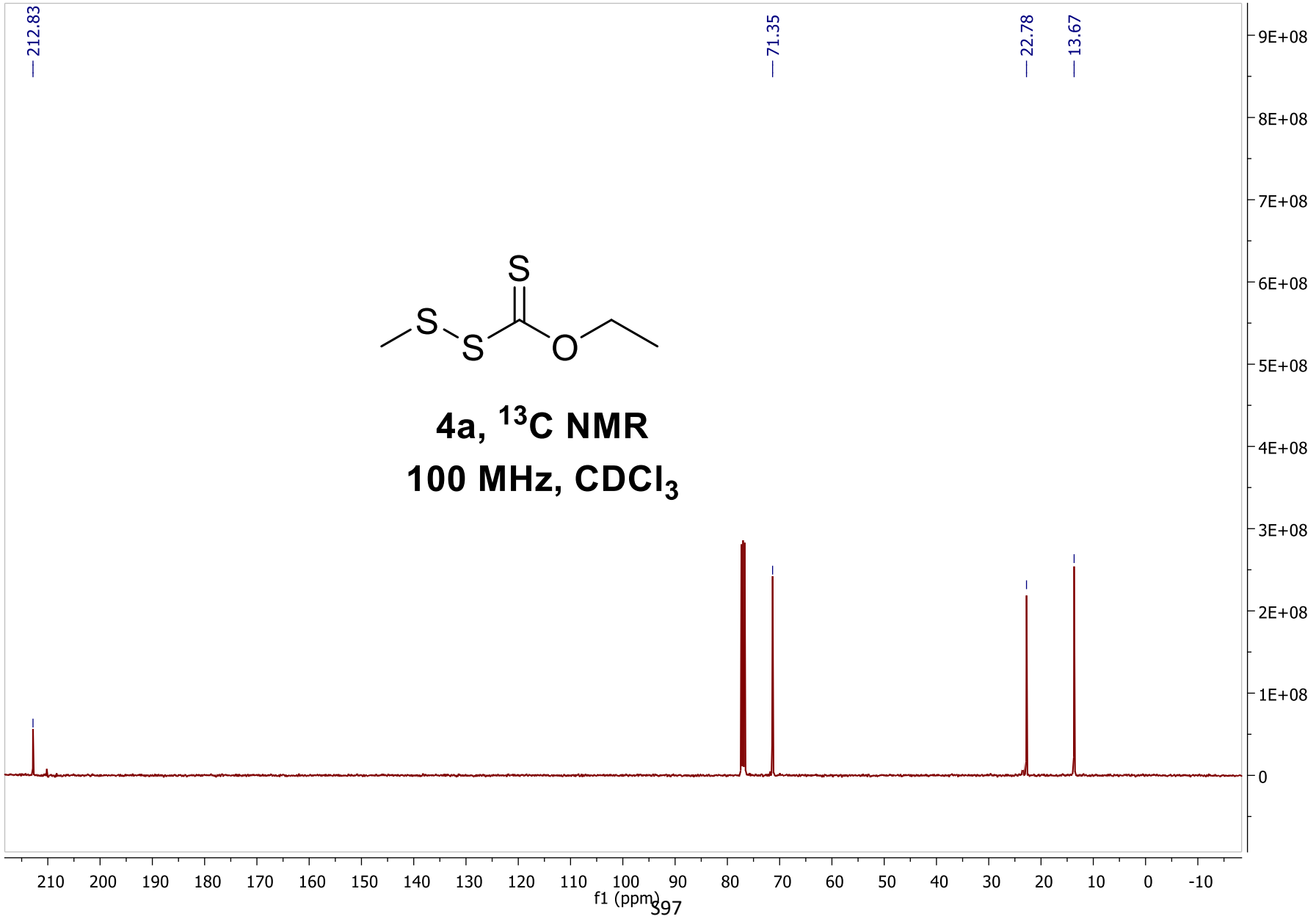

7.40
7.40
7.38
7.38
7.38
7.33
7.31
7.31
7.30
7.30
7.28
7.28
7.27
7.27
7.26
4.54
4.07
4.06
4.04
4.02
3.75
3.74
3.72
3.70
1.30
1.29
1.28
1.26
Ph
Se
S
S
N
3od, 1H NMR
400 MHz, CDCl3
Water signal
1.96
3.05
2.00
2.04
1.97
5.95
f1 (ppm)


195.29
136.01
129.42
129.00
128.59
127.48
49.48
46.74
42.25
12.48
11.61
Ph
Se
S
S
N
3od, 13C NMR
100 MHz, CDCl3
2E+08
2E+08
2E+08
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
1E+08
1E+08
9E+07
8E+07
7E+07
6E+07
5E+07
4E+07
3E+07
2E+07
1E+07
0
-1E+07
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

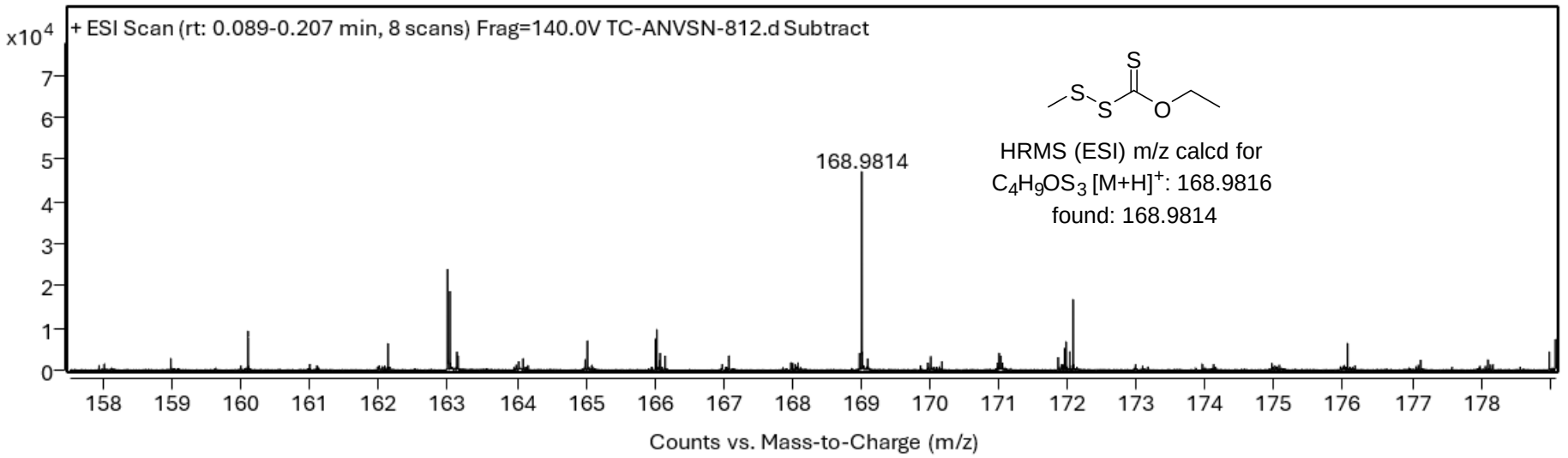

330.47
Ph
Se
S
S
N
3od, 77Se NMR
76 MHz, CDCl3
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
-1E+08
1500
1300
1100
900
800
700
600
500
400
300
200
100
0
-200
-400
-600
-800
f1 (ppm)


Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.218-0.519 min, 19 scans) Frag=140.0V ANVSN-819-318.d Subtract
x10⁴
5
4
3
2
1
0
Ph
Se
S
S
N
HRMS (ESI) m/z calcd for
C12H18NS2Se [M+H]+: 320.0046
found: 320.0044.
317.2163
318.2093
319.2121
320.0044
321.0072
322.0048
324.0402
325.0440
326.0095
328.2851
329.1721
317
318
319
320
321
322
323
324
325
326
327
328
329
Counts vs. Mass-to-Charge (m/z)

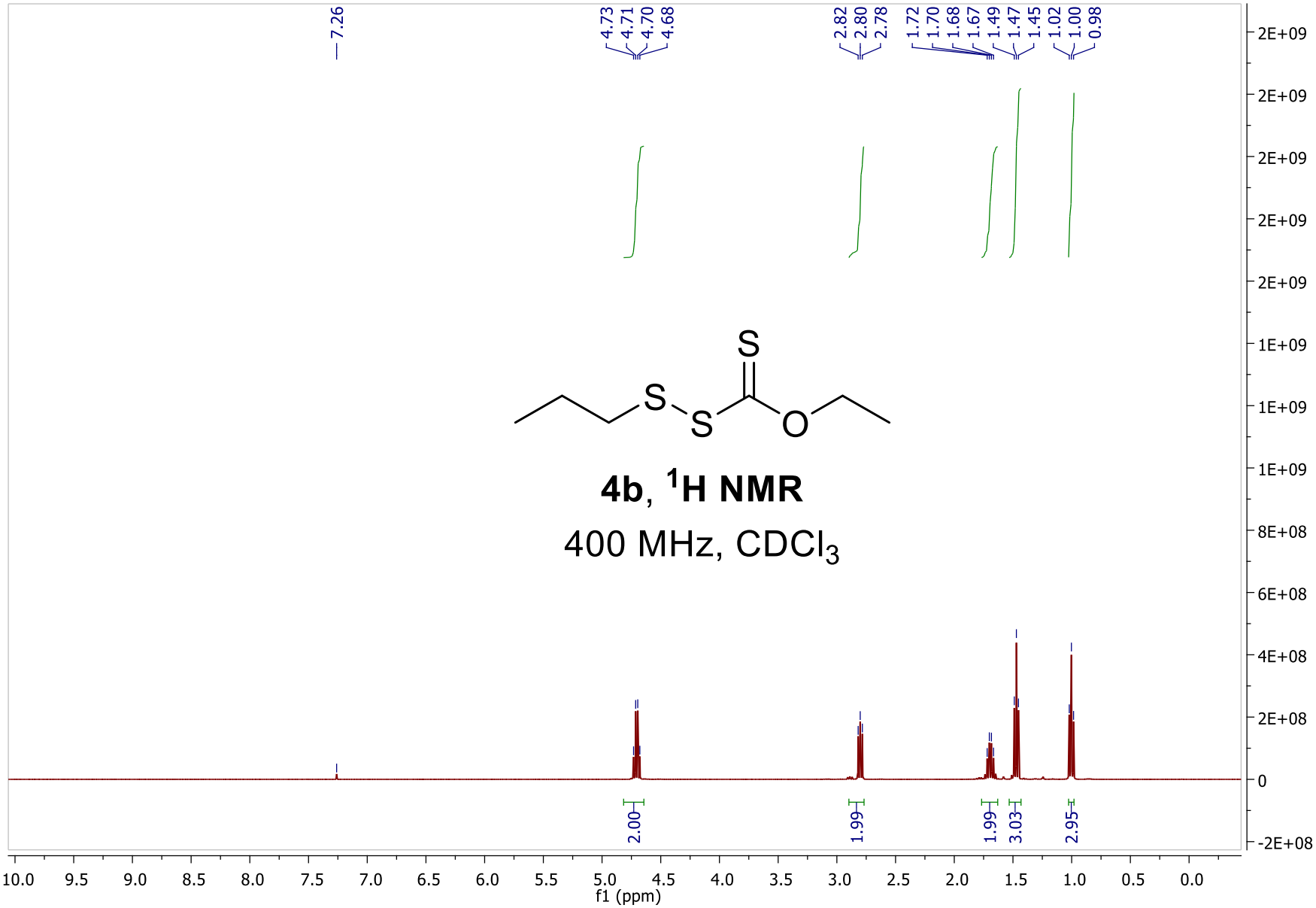

7.26
4.75
4.73
4.72
4.70
2.52
1.50
1.48
1.46
S
S
S
O
4a, ^{1}HNMR
400 MHz, $CDCl_3$
1.99
3.00
3.02
f1 (ppm)

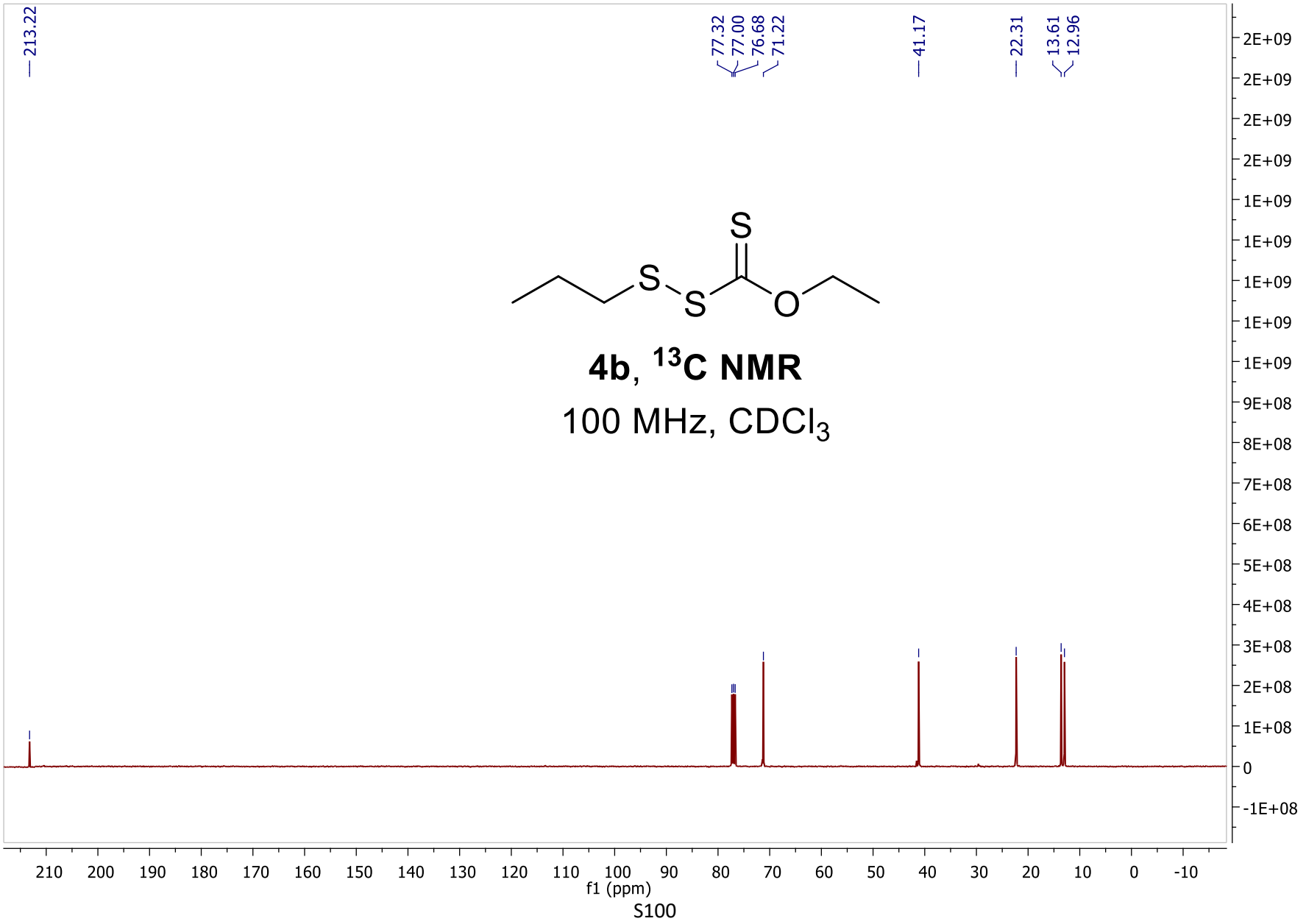

212.83
71.35
22.78
13.67
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
S
S
S
O
4a, 13C NMR
100 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Spectrum Plot Report

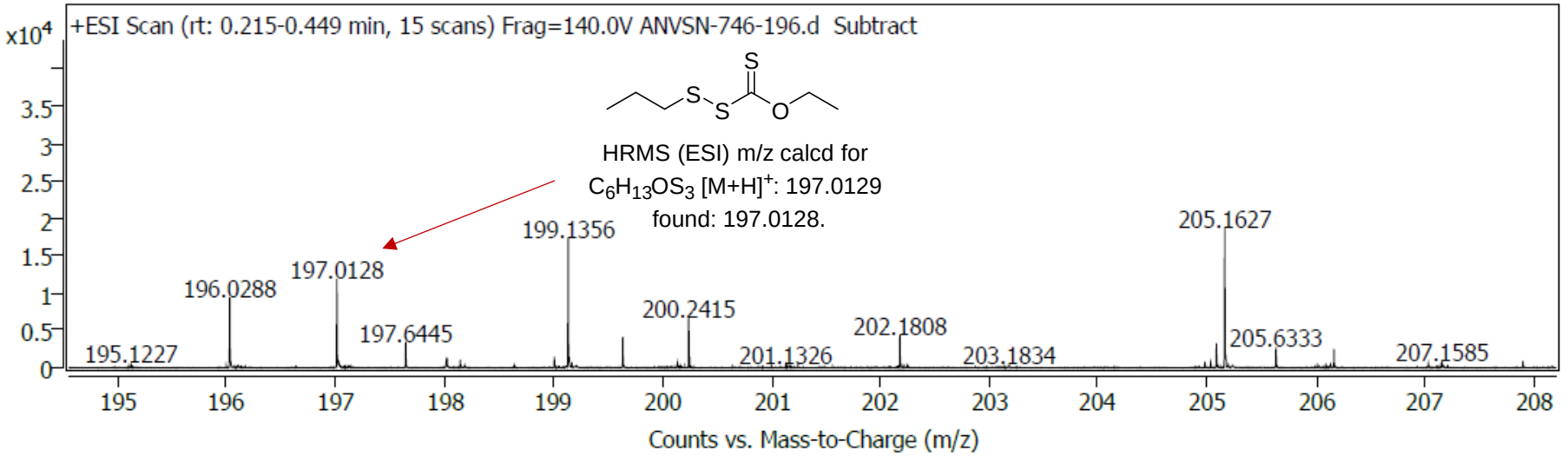


HRMS (ESI) m/z calcd for $C_4H_9OS_3$ $[M+H]^+$: 168.9816
found: 168.9814

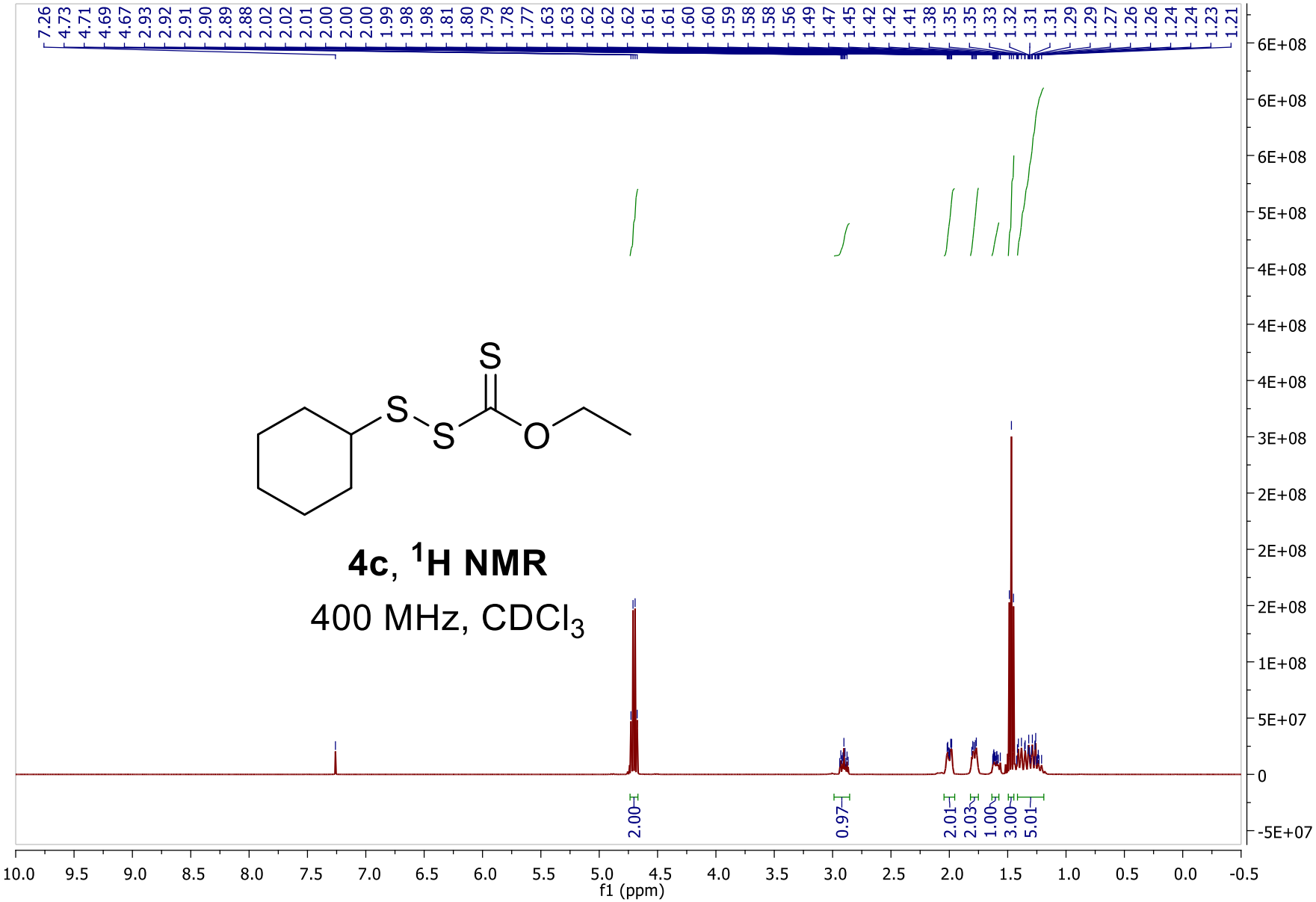

7.26
4.73
4.71
4.70
4.68
2.82
2.80
2.78
1.72
1.70
1.68
1.67
1.49
1.47
1.45
1.02
1.00
0.98
S
S
S
O
4b, 1H NMR
400 MHz, CDCl3
2.00
1.99
1.99
3.03
2.95
f1 (ppm)

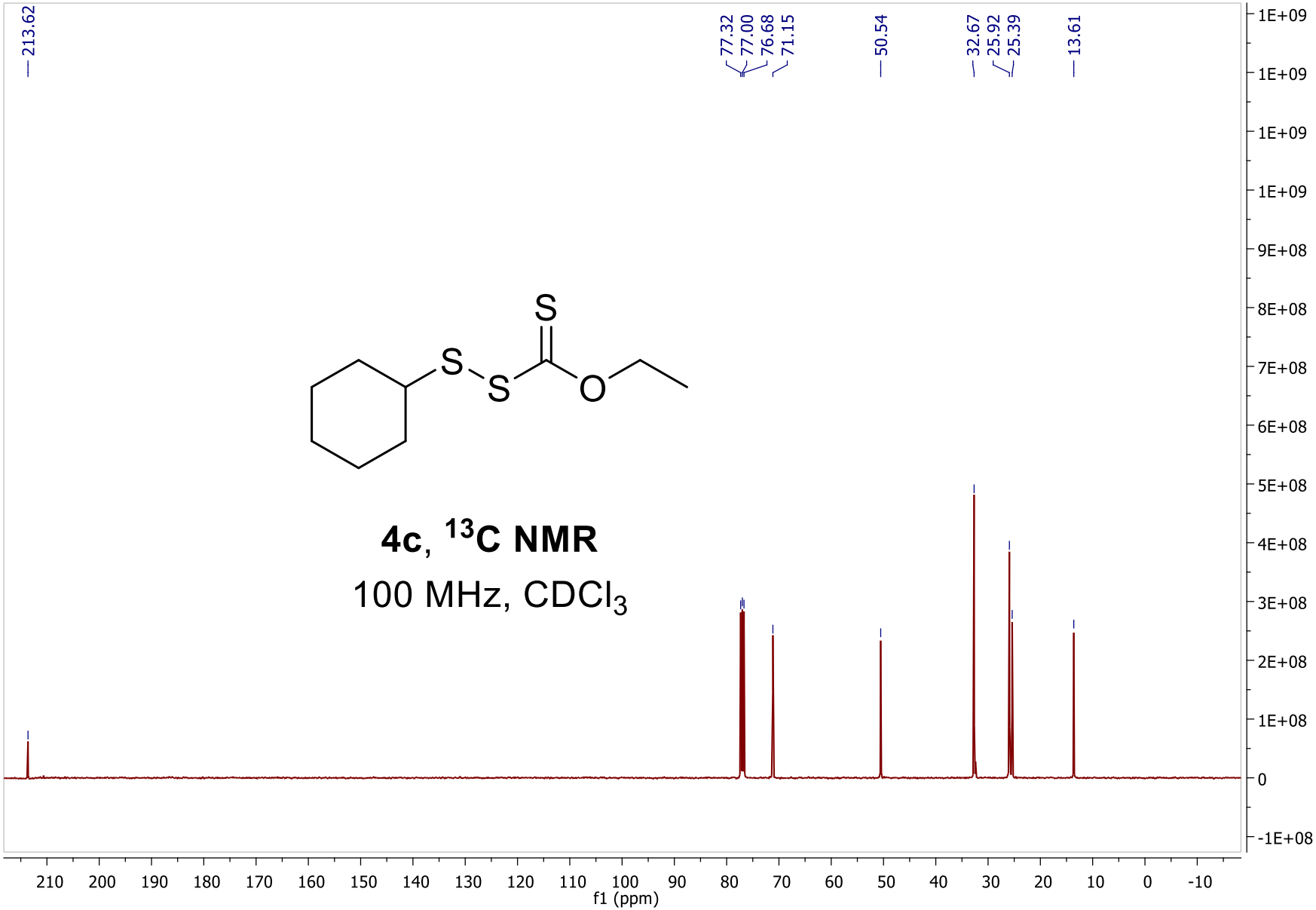

213.22
77.32
77.00
76.68
71.22
41.17
22.31
13.61
12.96
S
S
S
O
4b, 13C NMR
100 MHz, CDCl3
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

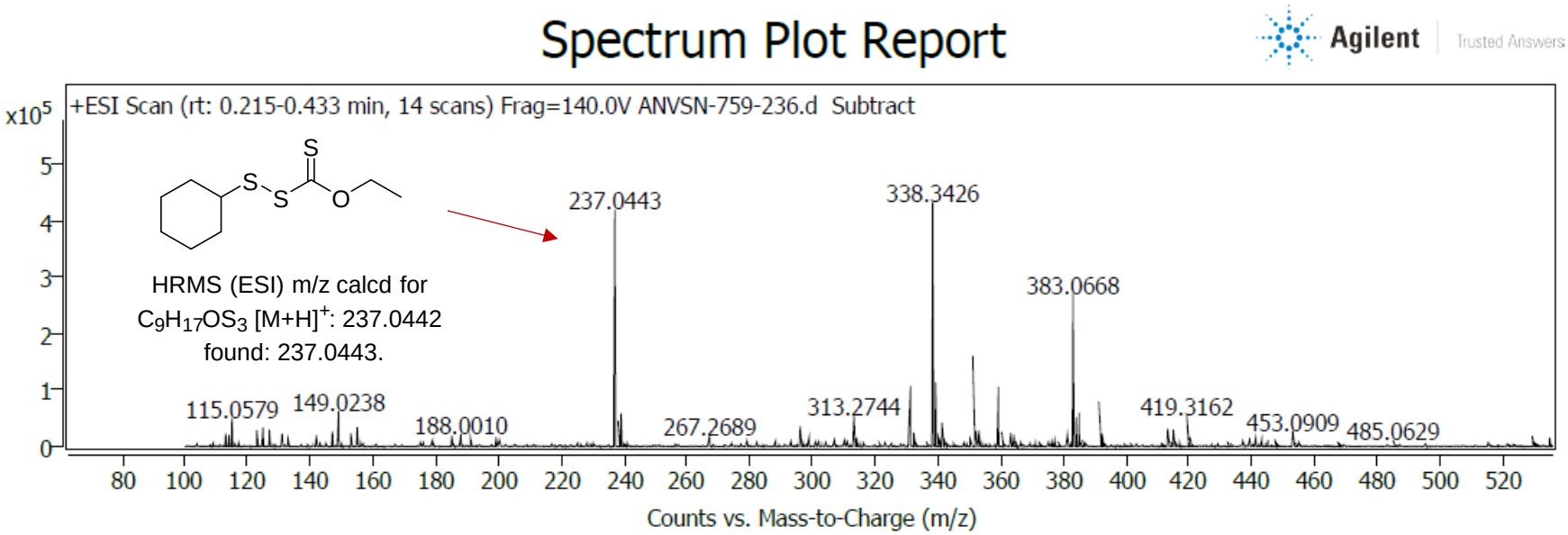
Spectrum Plot Report

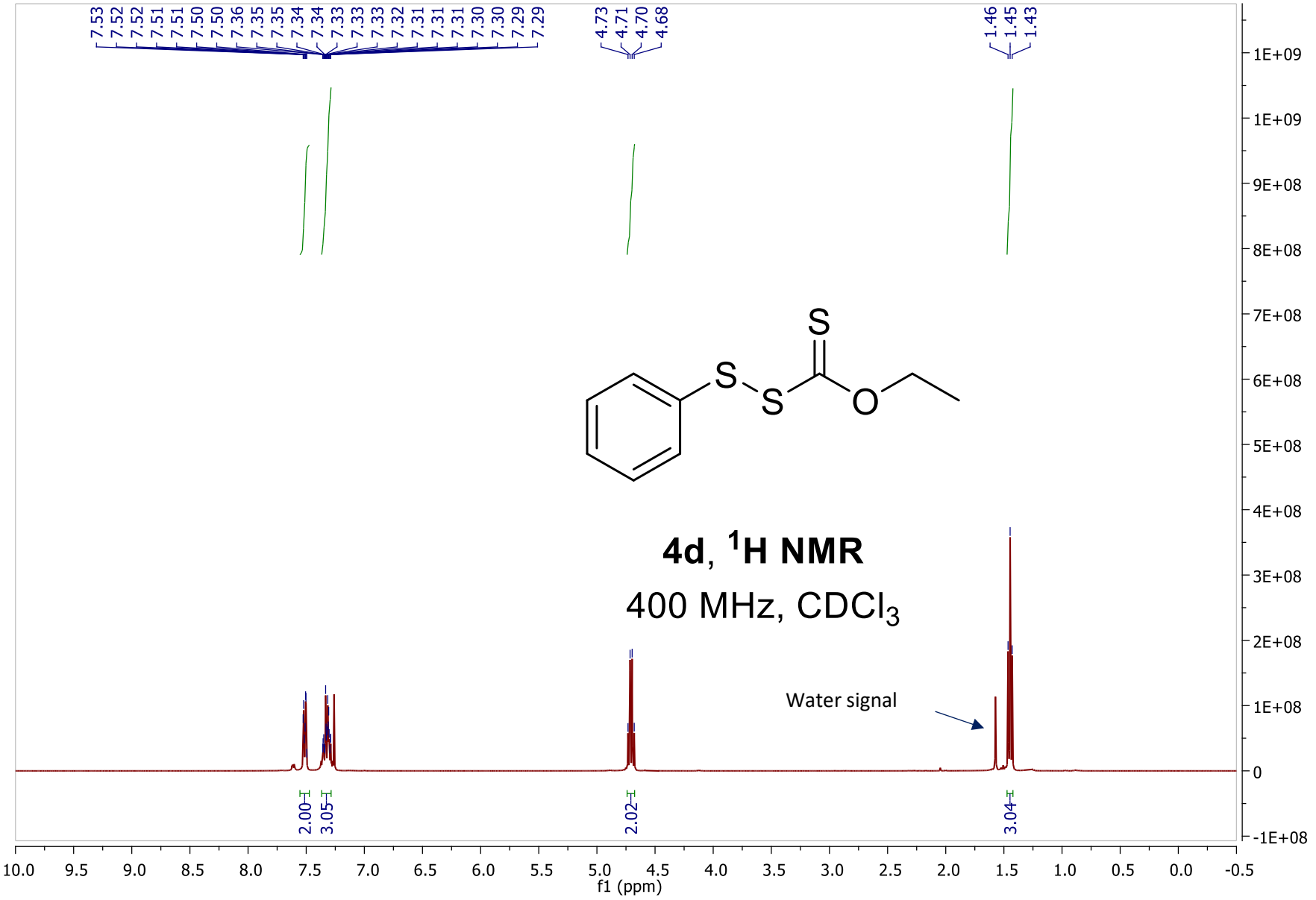


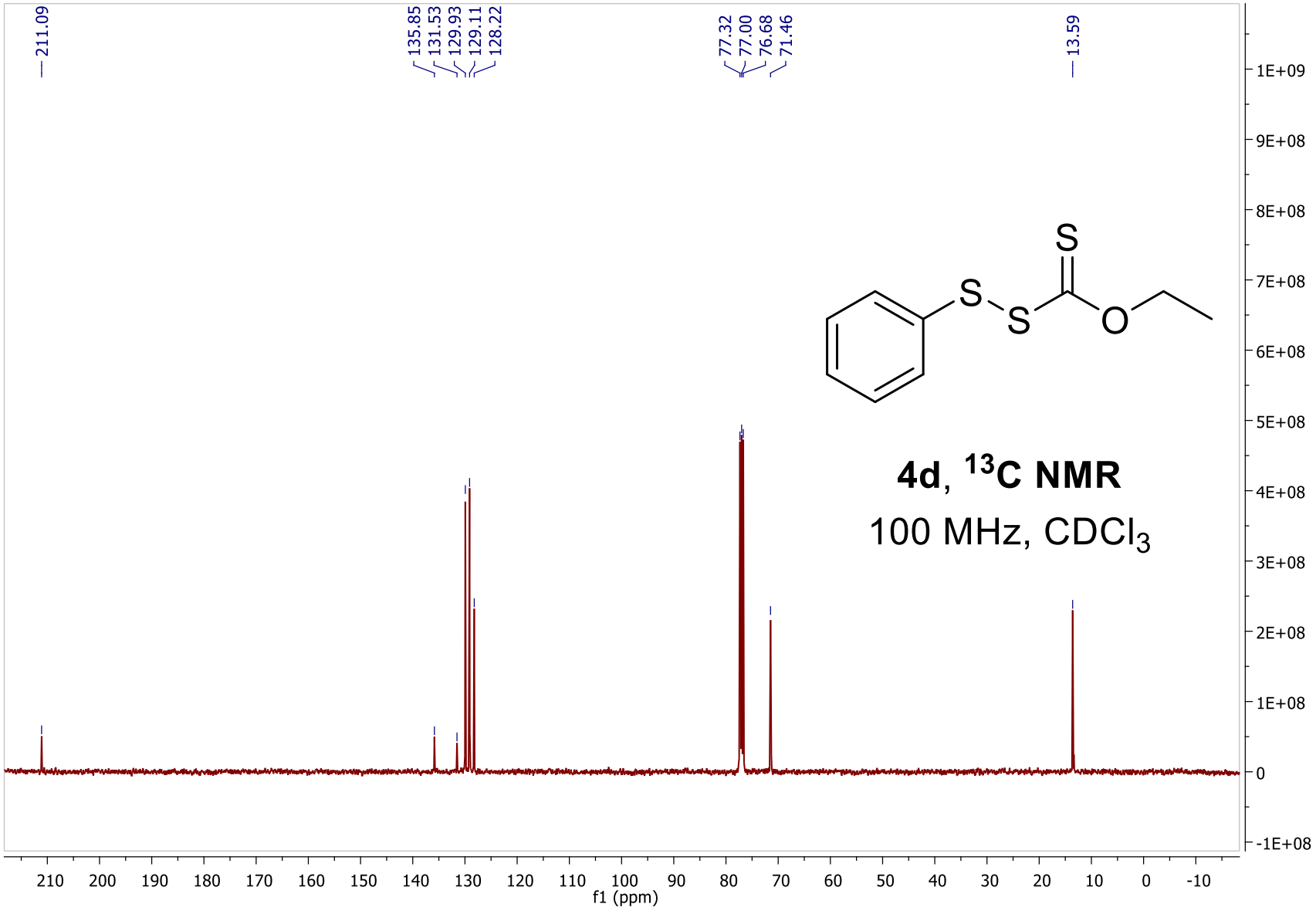
HRMS (ESI) m/z calcd for $C_6H_{13}OS_3$ $[M+H]^+$: 197.0129
found: 197.0128.

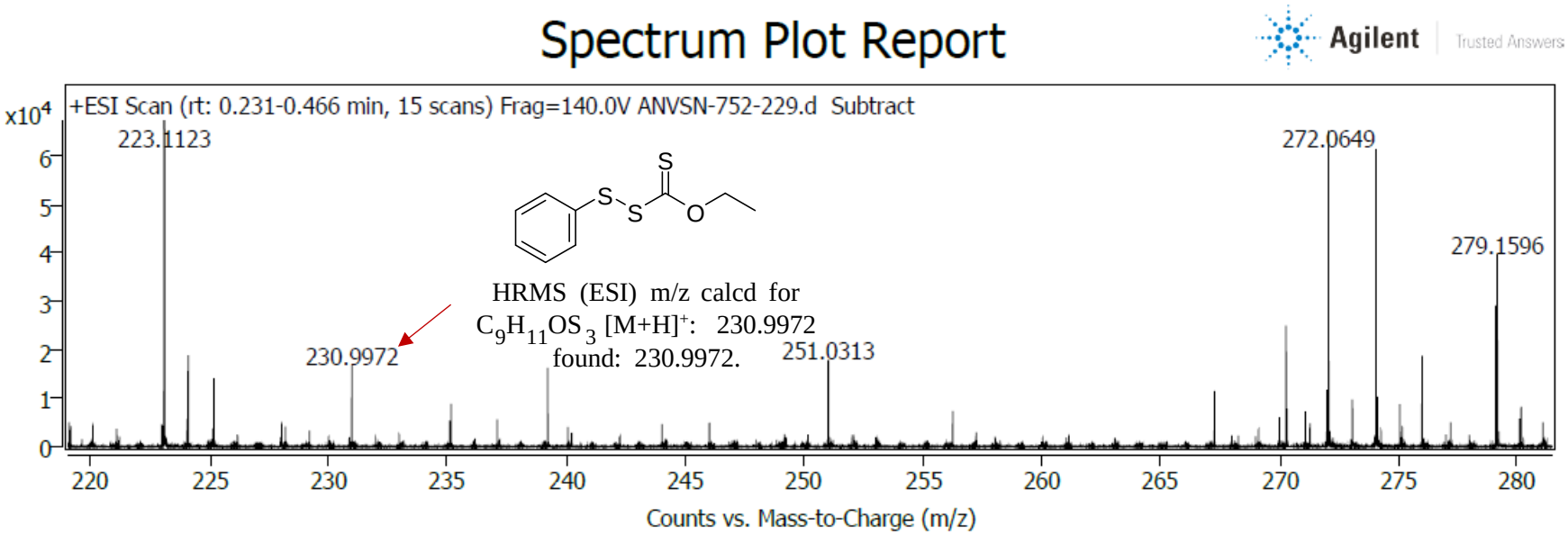

7.26 4.73 4.71 4.69 4.67 2.93 2.92 2.91 2.90 2.89 2.88 2.02 2.02 2.01 2.00 2.00 2.00 1.99 1.98 1.98 1.81 1.80 1.79 1.78 1.77 1.63 1.63 1.62 1.62 1.62 1.61 1.61 1.60 1.60 1.59 1.58 1.58 1.56 1.49 1.47 1.45 1.42 1.42 1.41 1.38 1.35 1.35 1.33 1.32 1.31 1.31 1.29 1.29 1.27 1.26 1.26 1.24 1.24 1.23 1.21
S
S
S
O
4c, 1H NMR
400 MHz, CDCl3
2.00
0.97
2.01
2.03
1.00
3.00
5.01
f1 (ppm)

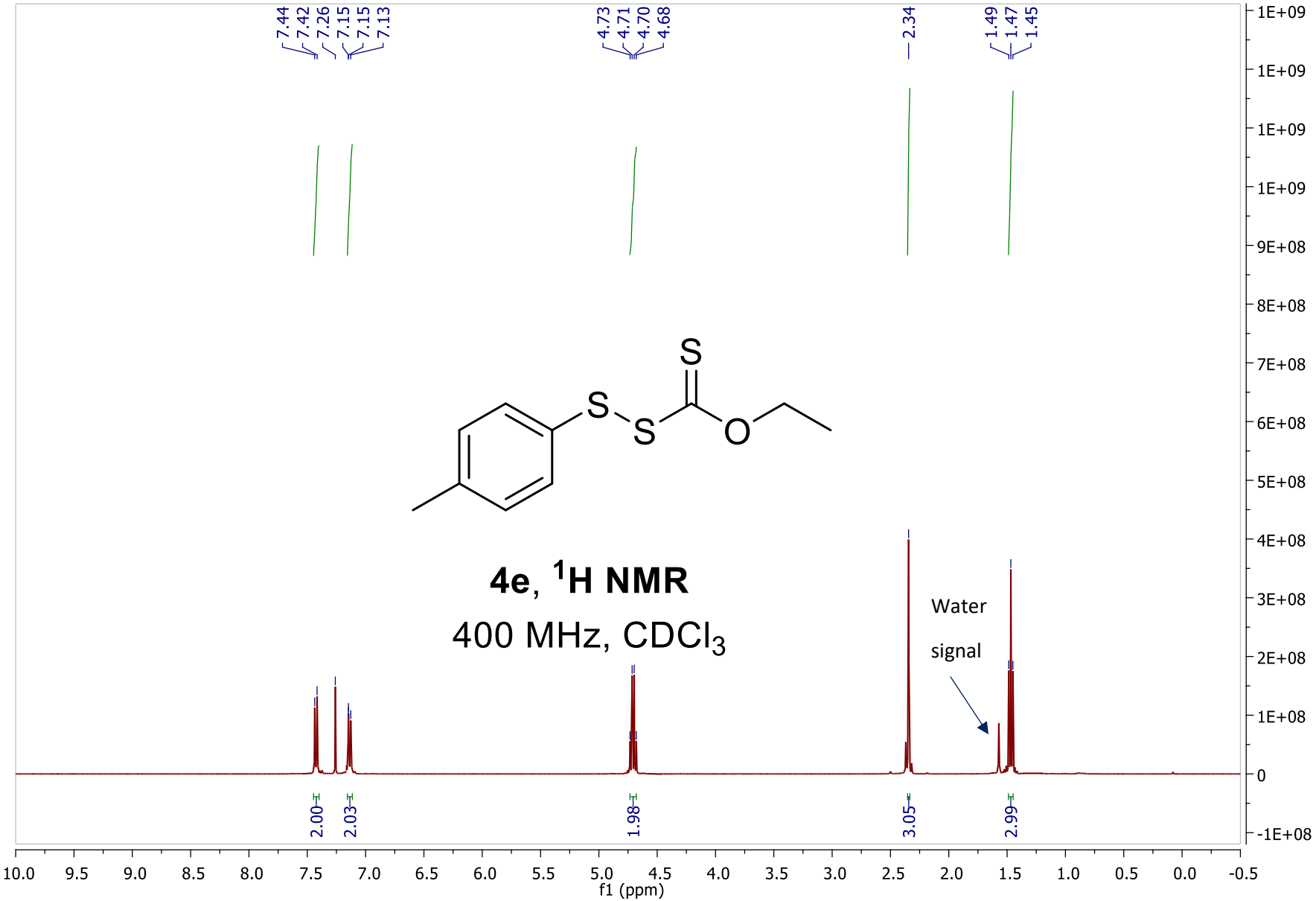

213.62
77.32
77.00
76.68
71.15
50.54
32.67
25.92
25.39
13.61
S
S
S
O
4c, 13C NMR
100 MHz, CDCl3
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

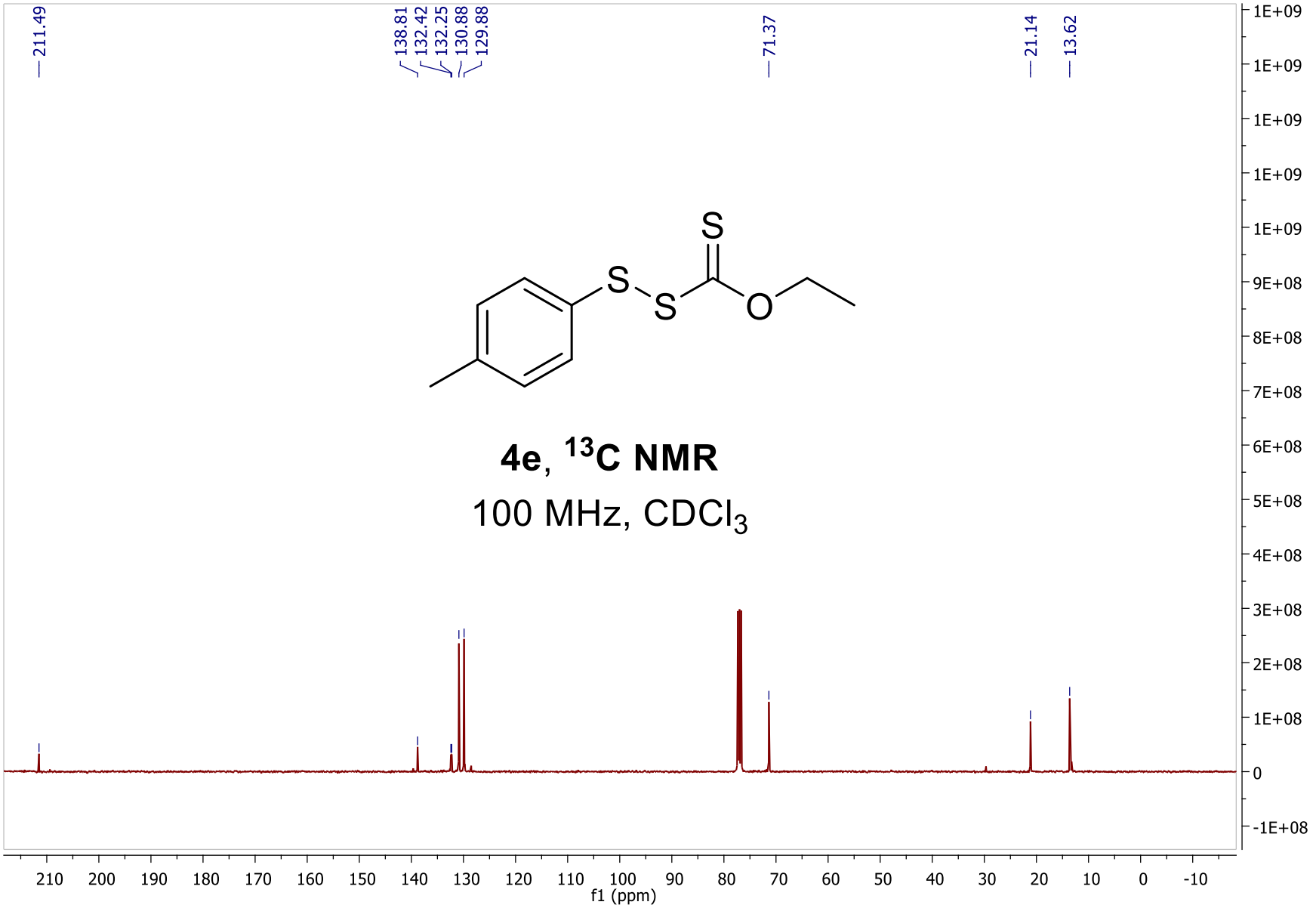

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.215-0.433 min, 14 scans) Frag=140.0V ANVSN-759-236.d Subtract
x10^5
HRMS (ESI) m/z calcd for
C9H17OS3 [M+H]+: 237.0442
found: 237.0443.
237.0443
338.3426
383.0668
115.0579
149.0238
188.0010
267.2689
313.2744
419.3162
453.0909
485.0629
Counts vs. Mass-to-Charge (m/z)

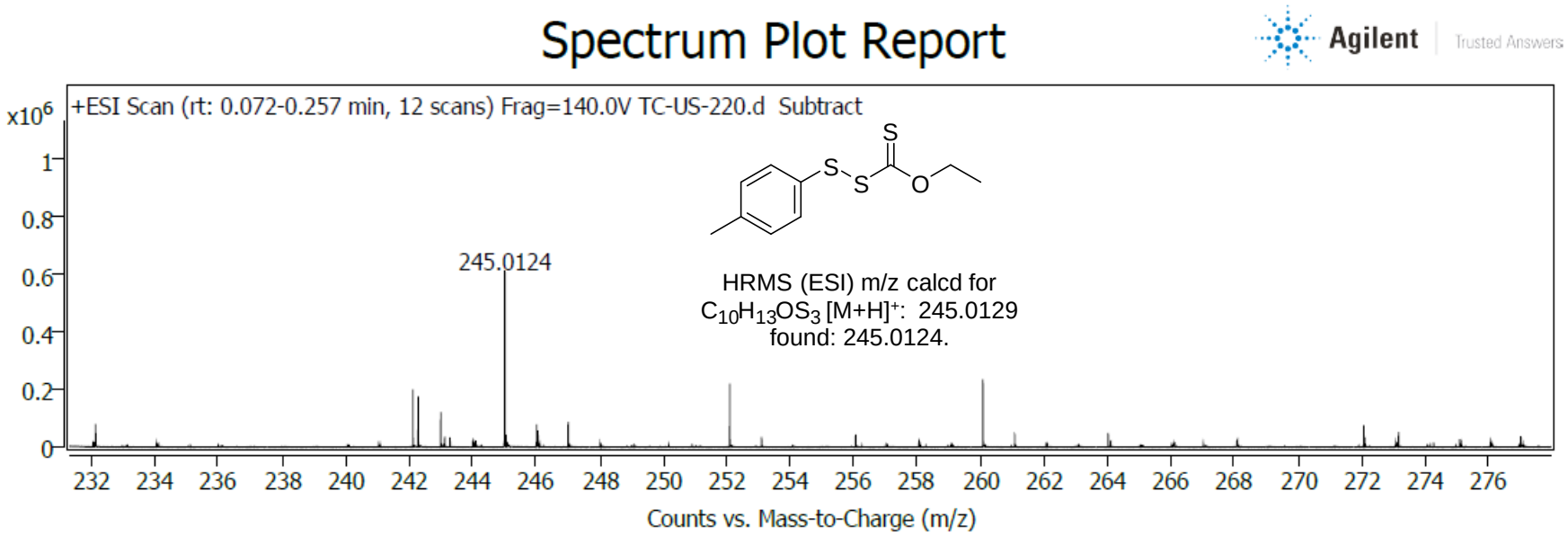

7.53 7.52 7.52 7.51 7.51 7.50 7.50 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.33 7.32 7.31 7.31 7.31 7.30 7.30 7.29 7.29
4.73 4.71 4.70 4.68
1.46 1.45 1.43
S
S
S
O
4d, 1H NMR
400 MHz, CDCl3
Water signal
2.00
3.05
2.02
3.04
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

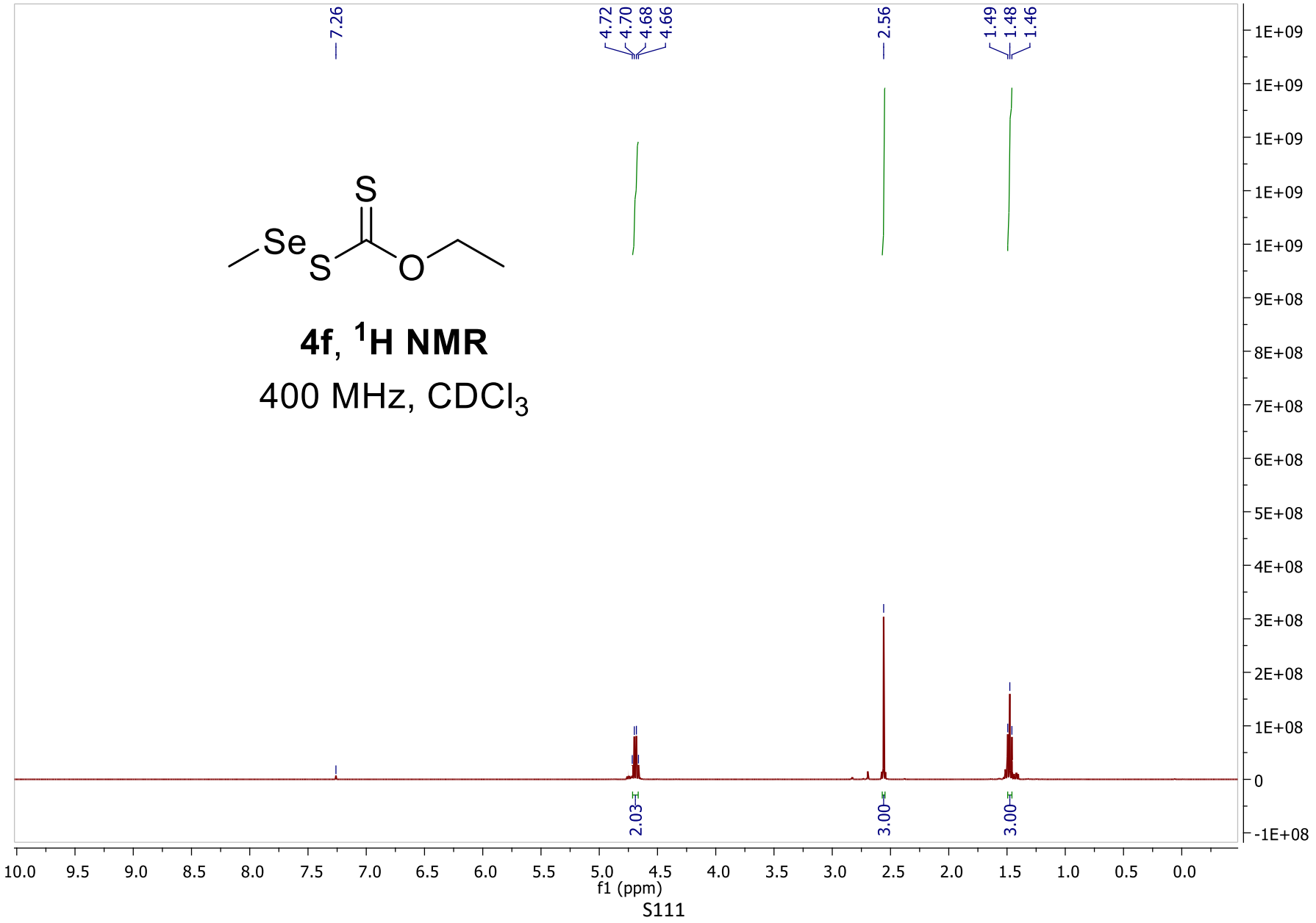

211.09
135.85
131.53
129.93
129.11
128.22
77.32
77.00
76.68
71.46
13.59
S
S
S
O
4d, 13C NMR
100 MHz, CDCl3
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

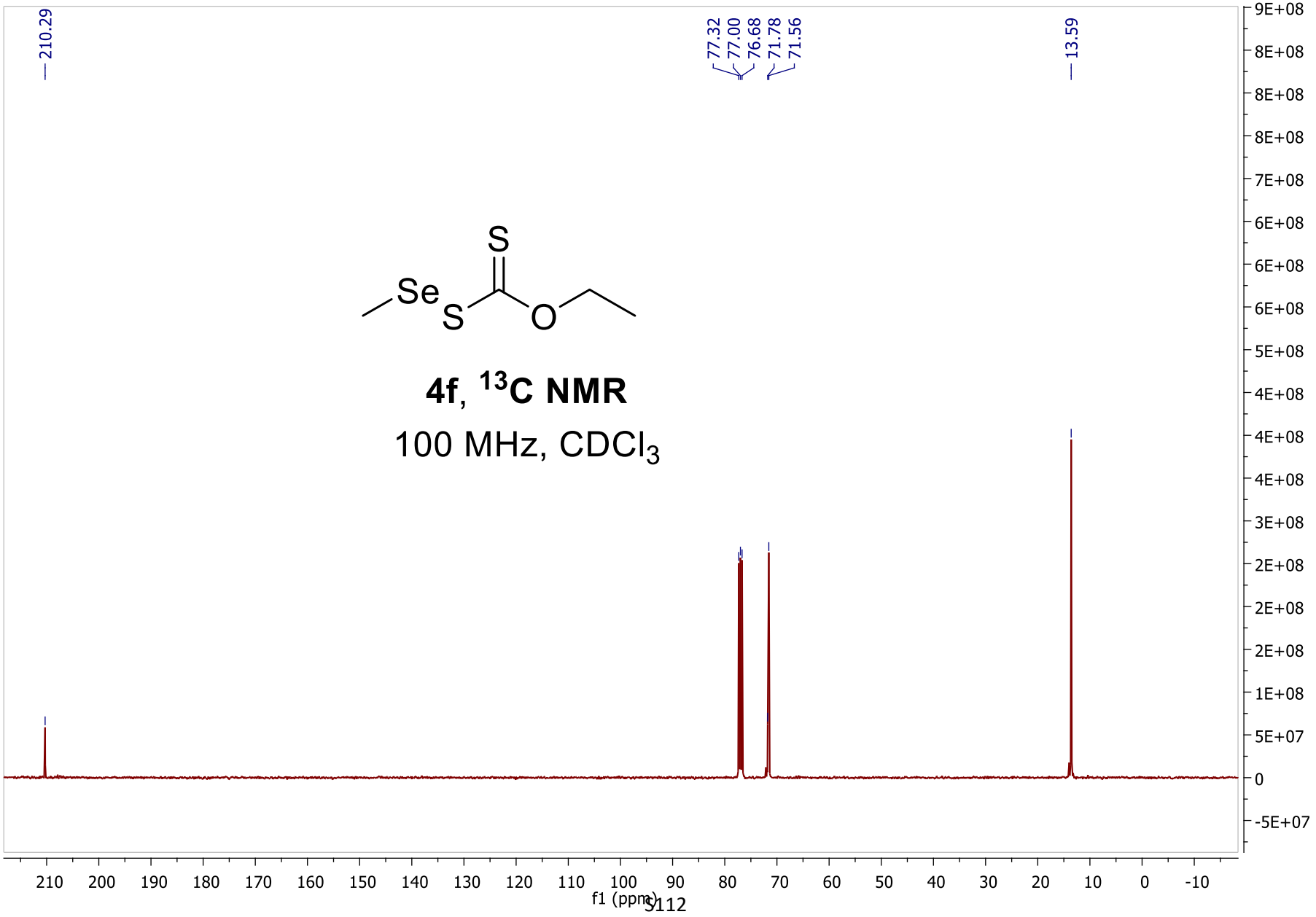

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.231-0.466 min, 15 scans) Frag=140.0V ANVSN-752-229.d Subtract
x10^4
223.1123
272.0649
279.1596
230.9972
251.0313
HRMS (ESI) m/z calcd for
C9H11OS3 [M+H]+: 230.9972
found: 230.9972.
Counts vs. Mass-to-Charge (m/z)

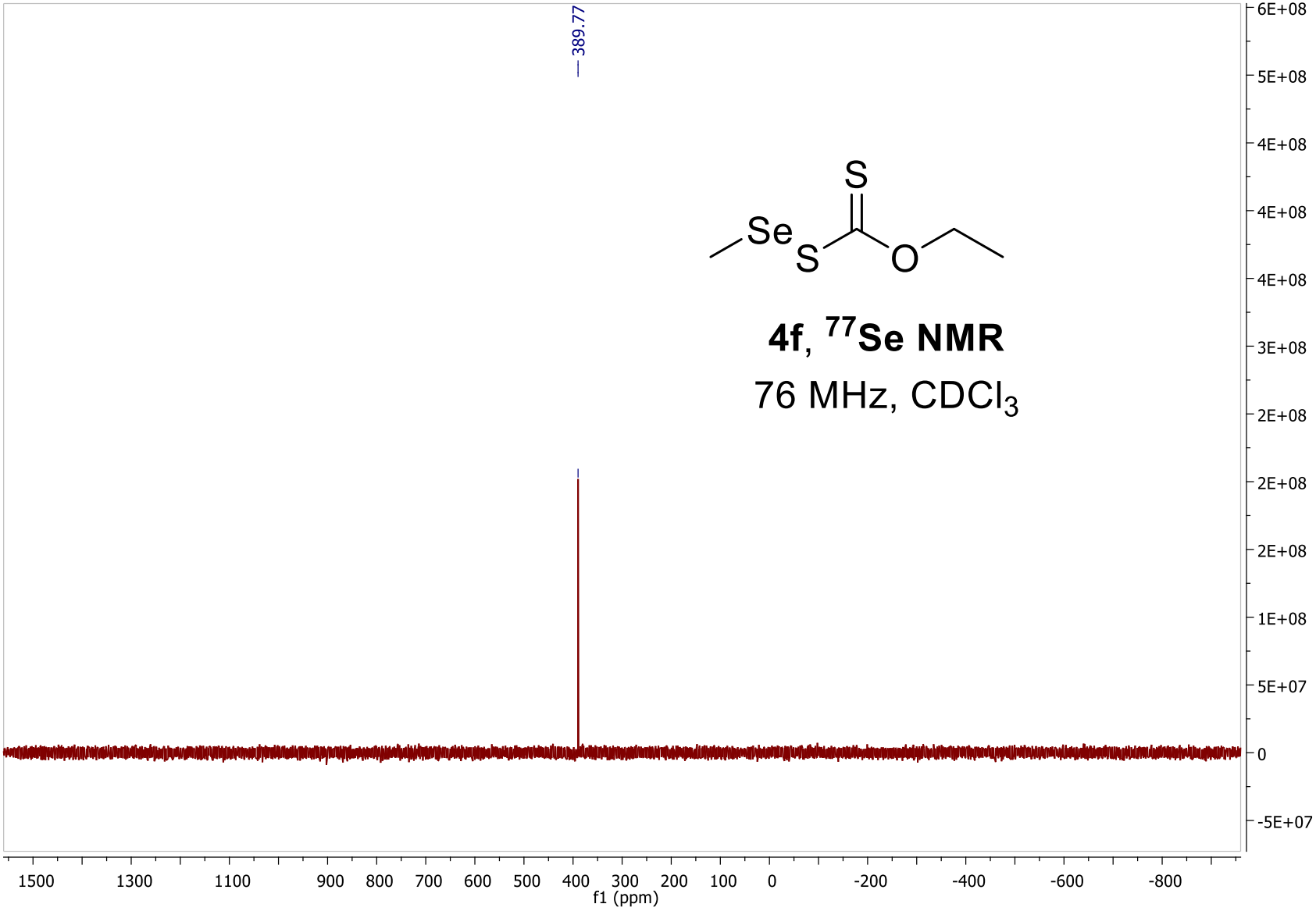

7.44
7.42
7.26
7.15
7.15
7.13
4.73
4.71
4.70
4.68
2.34
1.49
1.47
1.45
S
S
S
O
4e, 1H NMR
400 MHz, CDCl3
Water
signal
2.00
2.03
1.98
3.05
2.99
f1 (ppm)


211.49
138.81
132.42
132.25
130.88
129.88
71.37
21.14
13.62
S
S
S
O
4e, 13C NMR
100 MHz, CDCl3
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

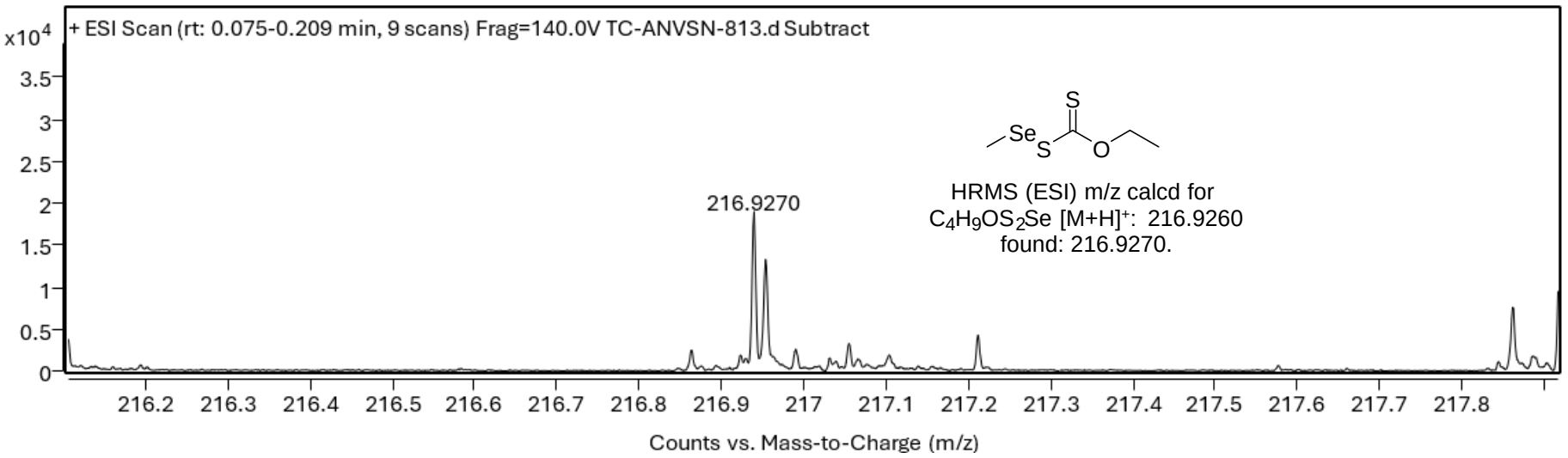

Spectrum Plot Report
Agilent
Trusted Answers
+ESI Scan (rt: 0.072-0.257 min, 12 scans) Frag=140.0V TC-US-220.d Subtract
x10^6
1
0.8
0.6
0.4
0.2
0
245.0124
HRMS (ESI) m/z calcd for
C10H13OS3 [M+H]+: 245.0129
found: 245.0124.
232 234 236 238 240 242 244 246 248 250 252 254 256 258 260 262 264 266 268 270 272 274 276
Counts vs. Mass-to-Charge (m/z)

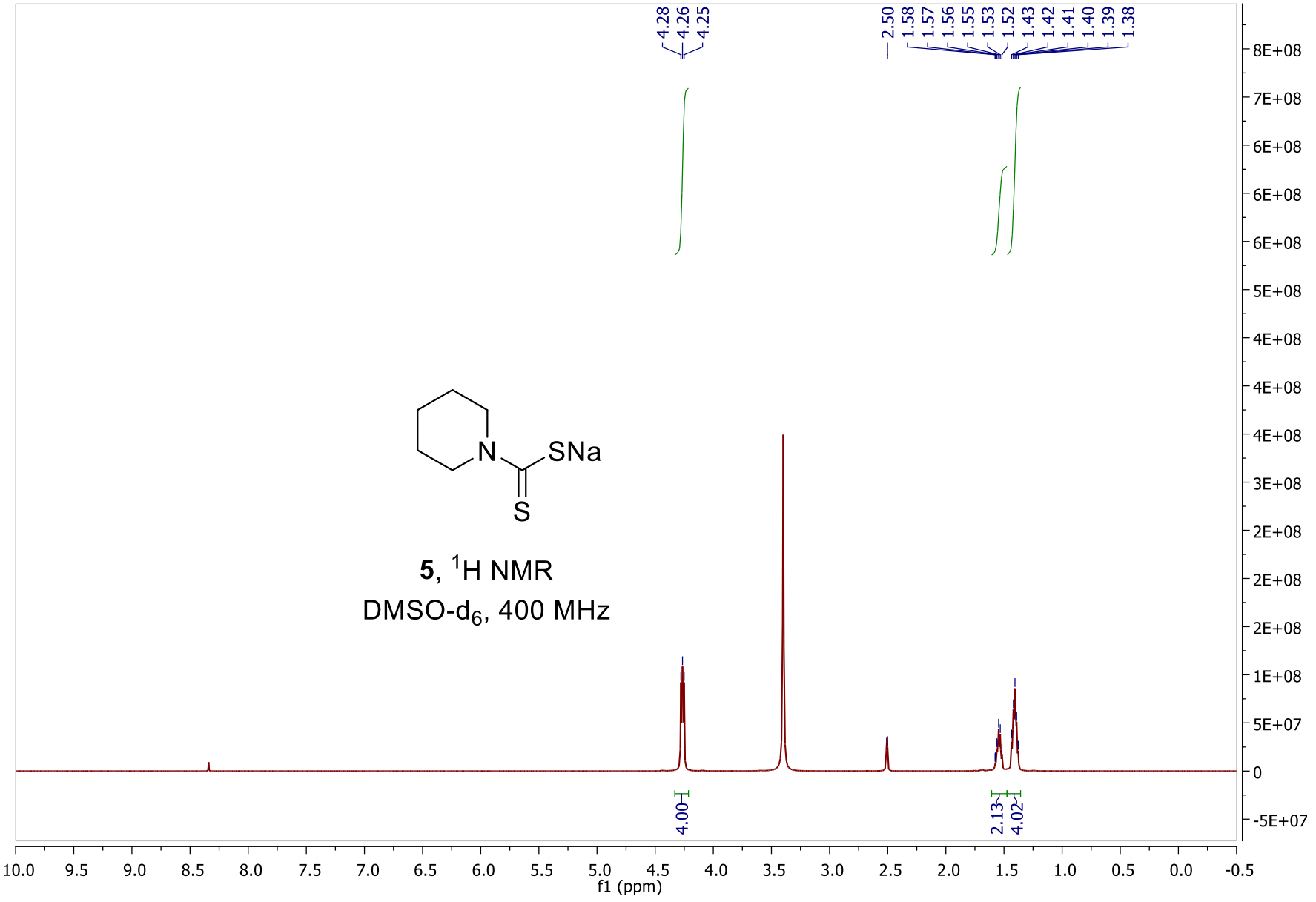

7.26
4.72
4.70
4.68
4.66
2.56
1.49
1.48
1.46
Se
S
S
O
4f, 1H NMR
400 MHz, CDCl3
2.03
3.00
3.00
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


210.29
77.32
77.00
76.68
71.78
71.56
13.59
S
Se
S
O
4f, 13C NMR
100 MHz, CDCl3
9E+08
8E+08
8E+08
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

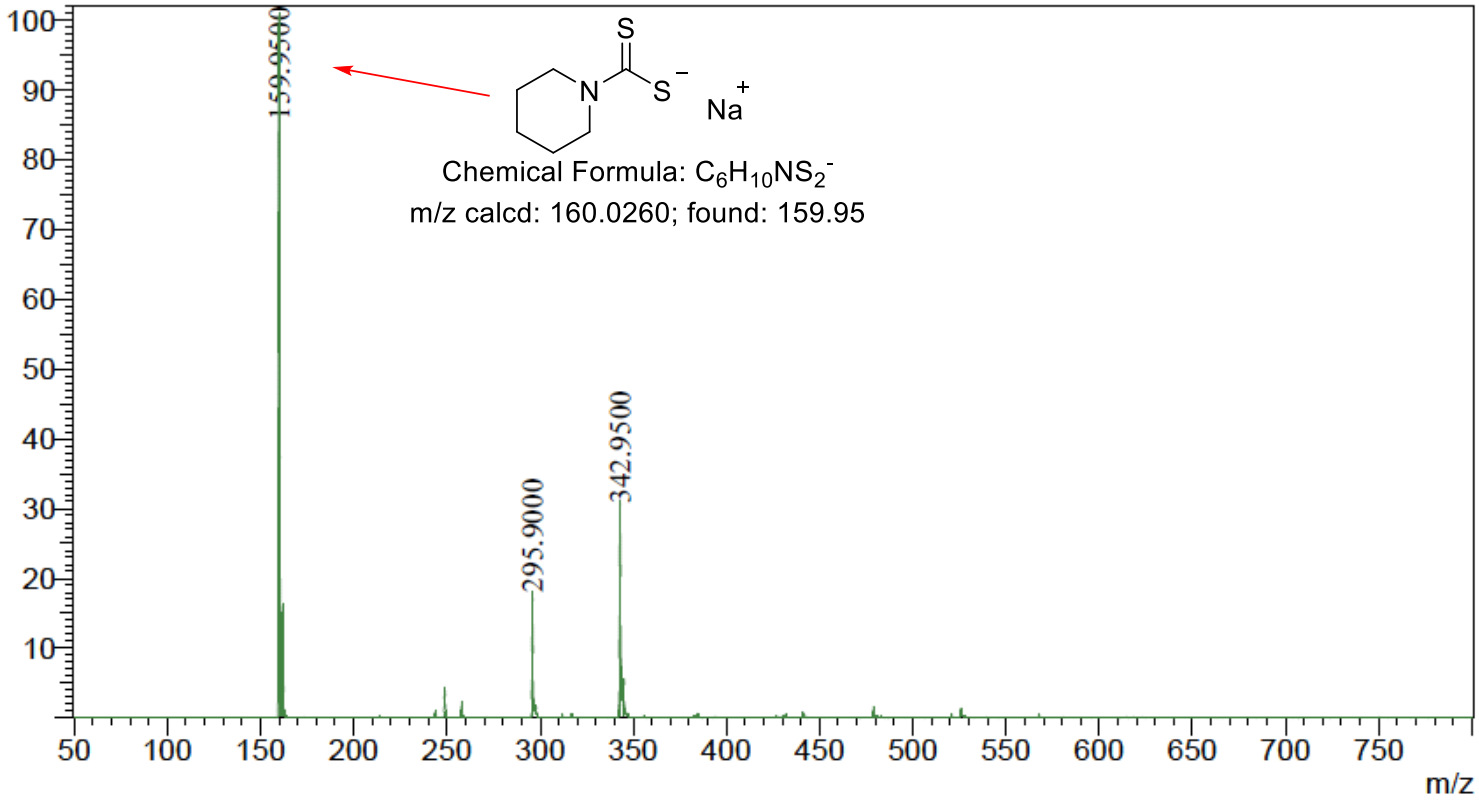

389.77
S
Se
S
O
4f, 77Se NMR
76 MHz, CDCl3
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
1500
1300
1100
900
800
700
600
500
400
300
200
100
0
-200
-400
-600
-800
f1 (ppm)

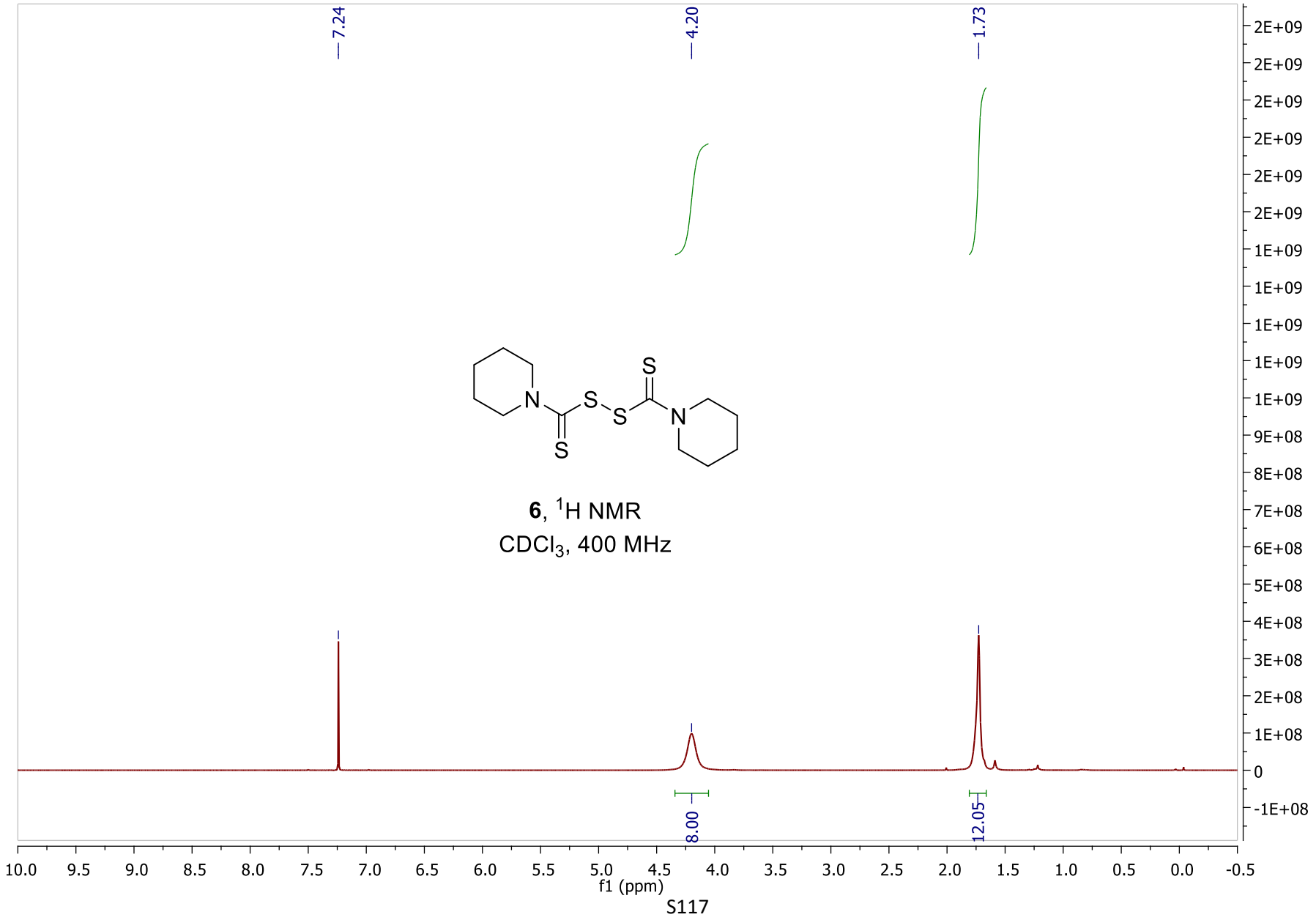
Spectrum Plot Report

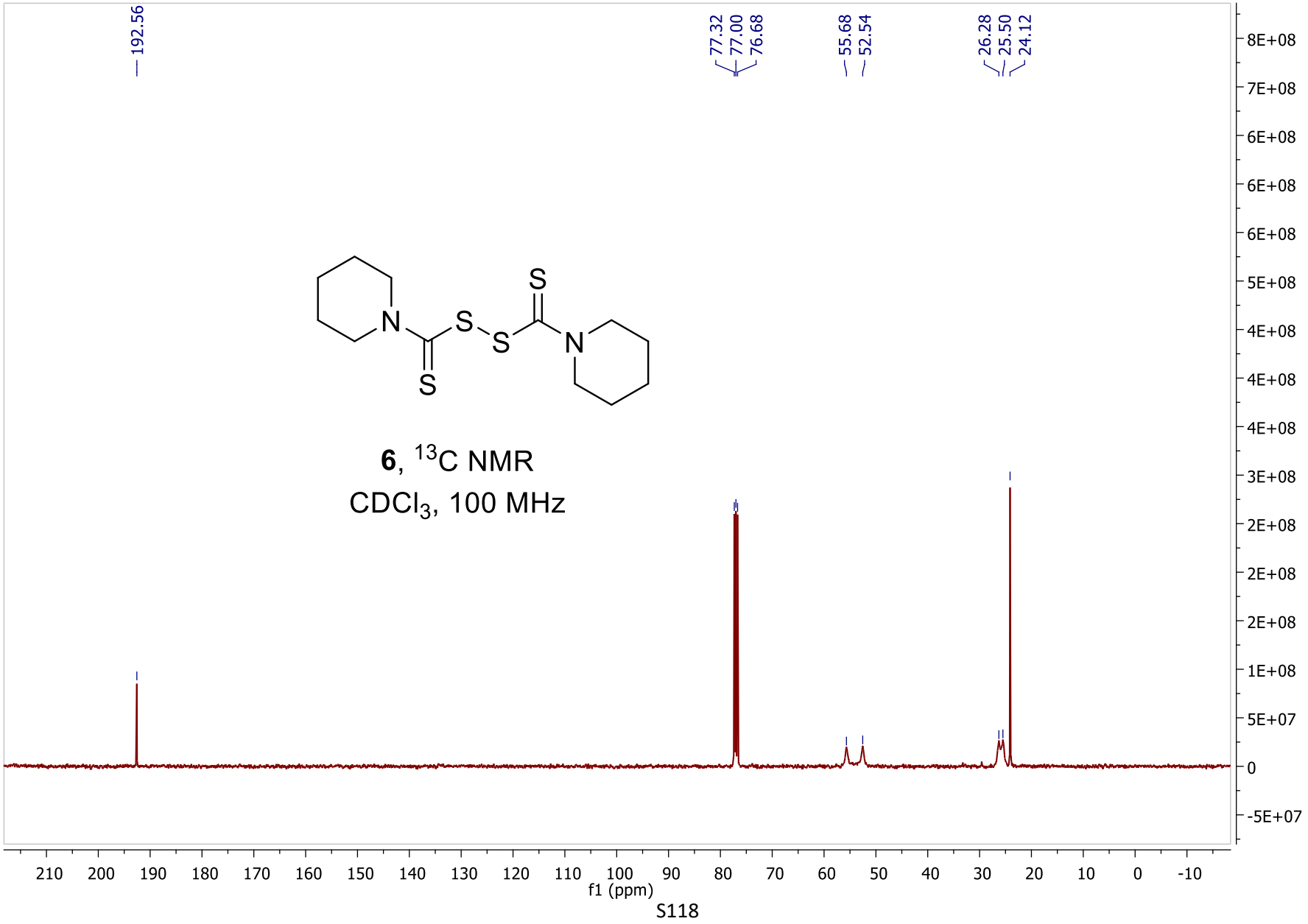


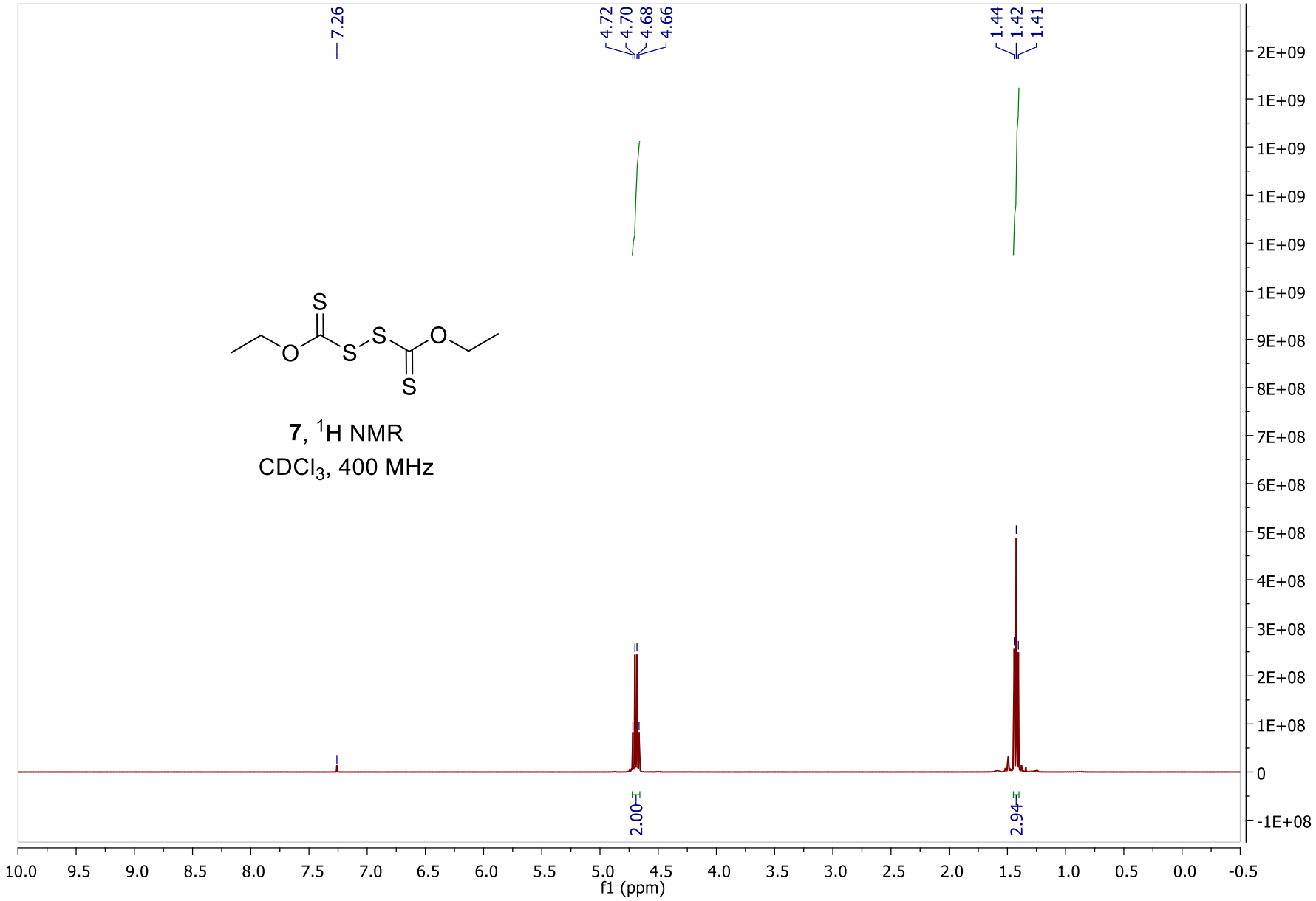

4.28
4.26
4.25
2.50
1.58
1.57
1.56
1.55
1.53
1.52
1.43
1.42
1.41
1.40
1.39
1.38
N
SNa
S
5, 1H NMR
DMSO-d6, 400 MHz
4.00
2.13
4.02
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

Line#:2 R.Time:----(Scan#:----)
MassPeaks:6
RawMode:Averaged 0.324-1.082(112-372) BasePeak:159.9500(10797484)
BG Mode:Averaged 0.044-0.330(16-114) Segment 1 - Event 2



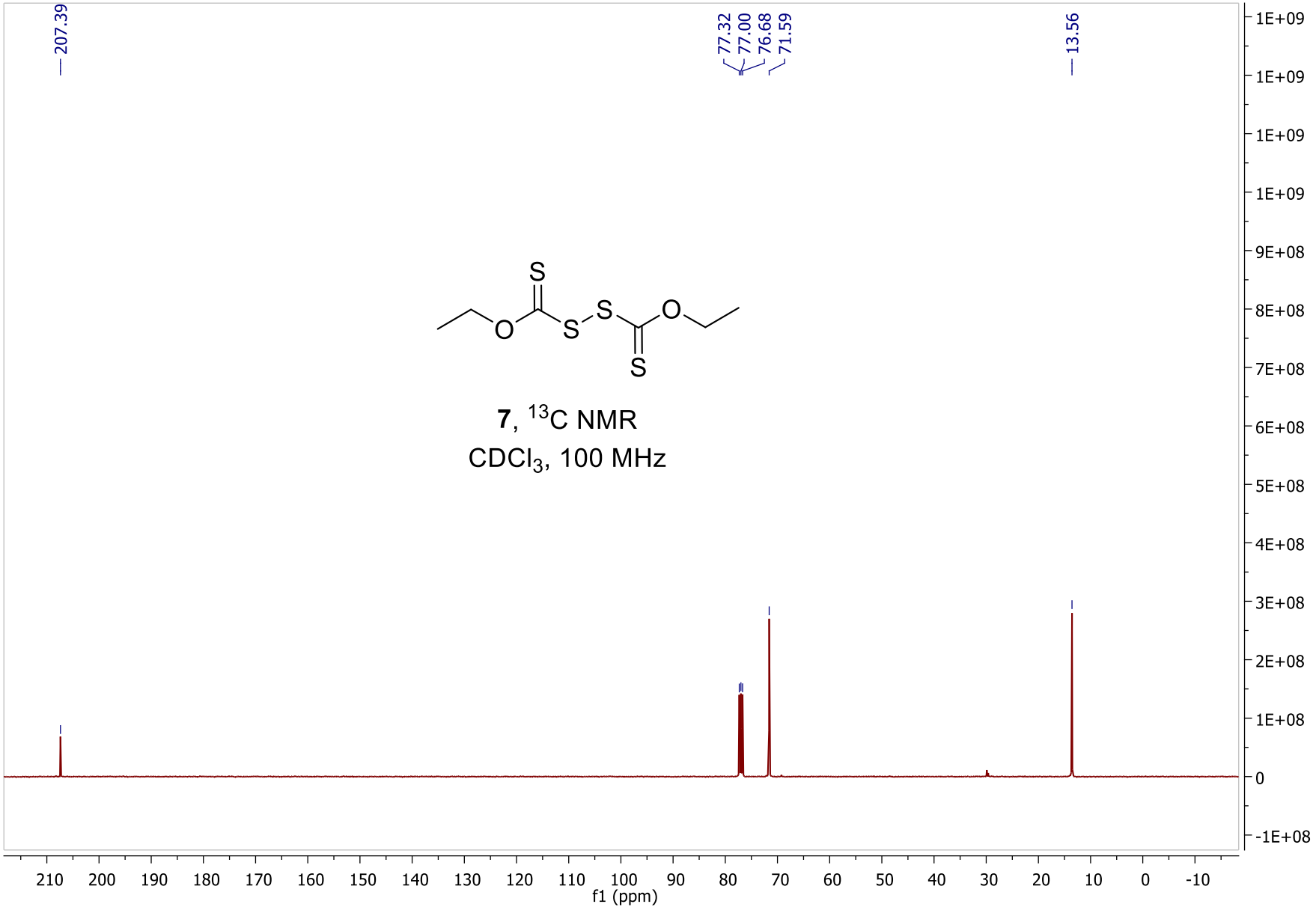

7.24
4.20
1.73
N
S
S
S
S
N
6, ^{1}H NMR
$CDCl_3$, 400 MHz
8.00
12.05
2E+09
2E+09
2E+09
2E+09
2E+09
2E+09
1E+09
1E+09
1E+09
1E+09
1E+09
1E+09
9E+08
8E+08
7E+08
6E+08
5E+08
4E+08
3E+08
2E+08
1E+08
0
-1E+08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


192.56
77.32
77.00
76.68
55.68
52.54
26.28
25.50
24.12
N
S
S
S
N
S
6, 13C NMR
CDCl3, 100 MHz
8E+08
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



7, ^{1}H NMR

$CDCl_3$, 400 MHz



7, ^{13}C NMR

$CDCl_3$, 100 MHz