Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Electrochemically-Driven Difunctionalization of Isocyanide and Mumm Rearrangement Cascade: Expeditious Synthesis of N-acyl-N-alkyl S-thiocarbamates

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1. General information

All reactions were carried out in preheated glassware. All solvents and chemicals were used as received from the suppliers (Alfa, Sigma-Aldrich, Avra, TCI). Cs_2CO_3 was purchased from Avra Synthesis Pvt. Ltd. Carboxylic acids and isocyanides were purchased from Sigma-Aldrich. All the other reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous material. TLC was performed using 0.25 mm E. Merck Silica plates (60 F_{254}), using short-wave UV light, I_2, and $KMnO_4$ for visualization. Solvents for column chromatography, crystallization, and extractions have been distilled once. Column chromatography was performed on silica gel (100-200 mesh). ^{1}H, ^{13}C NMR and ^{19}F spectra were recorded on a JEOL-400 spectrometer. Chemical shifts (δ) were reported in ppm referenced to an internal TMS standard for ^{1}H NMR (δ 7.26). Chemical shifts of ^{13}C NMR are reported relative to $CDCl_3$ (δ 77.00). The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d =doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, *J*, were reported in hertz (Hz). Mass spectra were recorded on a Bruker MicroTOF Q II mass spectrometer. Melting points were recorded on a Fisher-Johns 12-144 melting point apparatus and are uncorrected. The instrument for electrolysis is an IKA ElectraSyn 2.O pro DC power supply with a three-electrode cyclic voltammetry attachment.

a. **Batch Reactor**

Figure S1: **A**: ElectraSyn 2.0 batch reactor.

Figure S2: Electrode pair

Figure S3: ElectraSyn 2.O Vial (10 mL) & Screw cap

Setting up electrochemical reaction

Figure S4: Setting up electrochemical reaction

b. Fabrication of micro-electrolysis flow reactor (µ-EFR)

Micro electro-flow outer body was fabricated using a stainless-steel body jacket (***A1***: 100 mmlength x 64 mm width x 3 mm thickness) and second layer was fabricated 10 mm thickness high dens nylon plate (***A2***: 100 mm length x 64 mm width), it has a hole in the middle that allows an easy connection of the electrodes to the power supply by a copper wire and also has 2 holes, one for the inlet and one for the outlet of the reaction solution. The third layer was fabricated with teflon (***A3***: 100 mm length x 64 mm width x 0.5 mm thickness) layer made with laser cutter for protecting the nylon and stainless steel from the corrosive acid base and insulator for the current flow. Fourth layer copper electrode was customized as per the reactorsize (***A4***: 72 mm length x 38 mm width x 1 mm thickness) these are fitted in same size rectangular shape groove, 1mm thickness PTFE core plate. For the solution flow under the constant current, fifth layer consisted of a laser cutted PTFE plate (***A5***: 100 mm x 64 mm x 1 mm thickness) zigzag groove with rectangular shape (2 mm x 350 mm x 1 mm = 700 µL vol.),exposed electrode surface: 7 cm^2. Next, graphite was customized as per the reactor size(***A6***; 100 mm length x 64 mm width x 2 mm thickness) which absolutely covered with Pt loaded Cu foil (thickness 0.1 mm). Last layer aluminium body (***A7***: 100 mm length x 64 mm width x 15 mm thickness). After fabrication of each layer and to align thepatterns order ***A1*** to ***A7***. Thereafter, both the electrodes were sandwiched by 2 mm teflon zig- zag channel sheets with identical dimension to fit groove channels and coupled to each other by inserting metal allenkey bolts through the eight holes on aluminium body plate. Finally, themetal holder was tightly pressed by help of allenkey driver to seal the device with no leak (**Figure S5**).

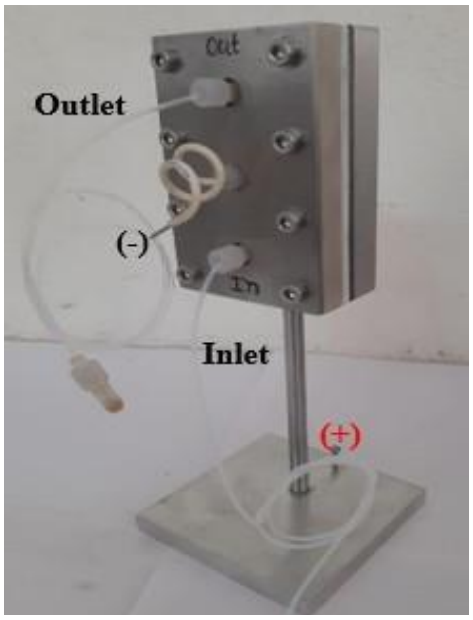
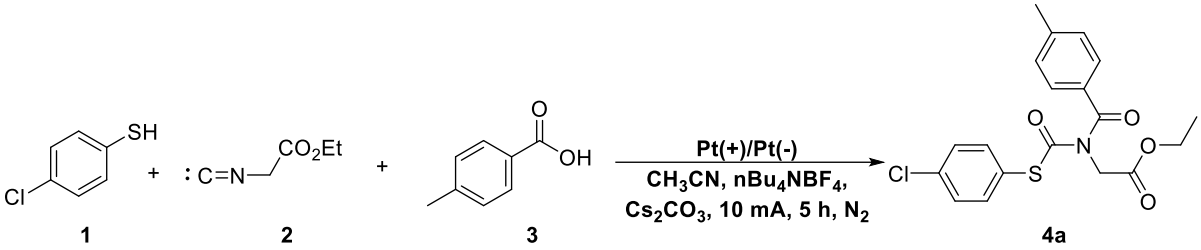


Figure S5. Original image of μ-EFR

Pt@Cu electrodes preparation: Modified phosphates bath-based method has been used for deposition of Pt over the Cu plate electrode. In this method, stock electrolytic solution containing mixture of Pt(IV) chlorides 150 mg, diammonium hydrogen phosphate $(NH4)_2HPO_4$ 80 mg, disodium hydrogen phosphate $(Na)_2HPO_4$ 200 mg, ammonium chloride 50 mg, and water 10 mL, was pumped with fix flow rate of 100 μL min^{-1} for 2 h at 70 ^oC under current density of 0.003 A/cm^2, which typically led to the generation of a silver-black colored patterning, and then washed with water thoroughly to remove any unreacted salt and nanoparticle.[1]

2. Reaction optimization

Table S1. Optimization of the reaction conditions[a].



Entry	Variation(s) from the standard conditions	**4a** yield (%)[b]
1.	No	87
2.	C(+)/Pt(-)	63
3.	C(+)/Ni(-)	60
4.	C(+)/C(-)	72
5.	nBu_4NI	74
6.	nBu_4NPF_6	62
7.	nBu_4NOTs	25
8.	MeCN/HFIP (5mL/1mL)	40
9.	THF	35
10.	DMF	33
11.	**3a** (0.5 mmol)	47
12.	Under air	25
13.	8 Ma	65
14.	12 Ma	73
15.	K_2CO_3	78
16.	No electric current	0

Standard conditions: 1 (0.5 mmol), **2** (0.7 mmol), **3** (1.0 mmol), nBu_4NBF_4 (0.5 mmol), Cs_2CO_3 (0.1 mmol), MeCN (6 mL), Pt anode, Pt cathode, undivided cell, constant current = 10 mA, room temperature, N_2, 5 h. isolated yield.

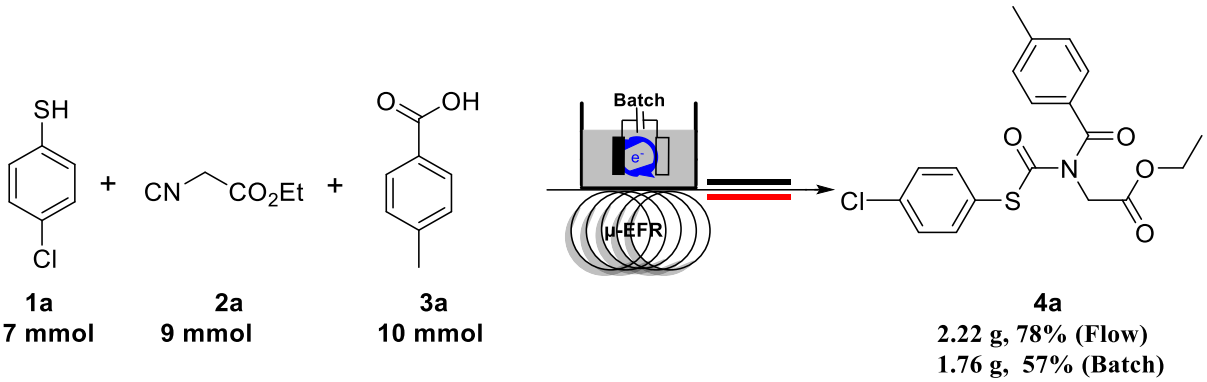
Our studies commenced with the electrochemical transformation of 4-chlorobenzenethiol (**1a**, 0.5 mmol), ethyl 2-isocyanoacetate (**2a**, 0.7 mmol), *p*-toluic acid (**3a**, 1.0 mmol) as coupling partners with platinum and platinum as the anode and the cathode, respectively. The optimized conditions are shown in Table 1. The thiocarbamate product **4a** was obtained in 87% isolated

yield with 10 mA constant current, and 1 equiv. nBu_4NBF_4 in CH_3CN at room temperature for 5 h under an N_2 atmosphere (Table 1, entry 1). When employing alternative electrode materials such as carbon or nickel plates for the model reaction, reduced yields were noted in undivided cell setups (entries 2-4). Subsequently, a notable decline in the yield of the desired product was observed when alternative salts were used in place of nBu_4NBF_4 (entries 5-7). The inclusion of a co-solvent, particularly HFIP, led to a sharp decrease in yield (entry 8). While solvents such as THF and DMF, with lower dielectric constants than MeCN, also resulted in diminished yields (entries 9, 10). An equimolar quantity of *p*-toluic acid resulted in a poor yield of 47% for the synthesis of **4a** (entry 11). The reaction was sensitive to air, and a drastic decrease in yield (25%) was obtained (entry 12). While investigating current variations, it was observed that the yield of the desired product decreased regardless of whether the applied current was increased or decreased (entries 13, 14). A decrease in yield was observed when Cs_2CO_3 was replaced with K_2CO_3, as indicated in entry 15. Finally, a control experiment demonstrated that electric current is indispensable for reactivity (entry 16).

Table S2: Base optimization

Entry	Base	Yield of 4a (%)
1.	Na_2CO_3	72
2.	K_2CO_3	78
3.	***Cs_2CO_3***	***87***
4.	Et_3N	20
5.	Et_2NH	46
6.	Py	64
7.	Bipy	75

Scheme S1. Gram-Scale Synthesis in Batch and Continuous Flow.



[a]**Reaction conditions for flow**: **1a** (1008 mg, 7.0 mmol, 175 mM), **2a** (1017 mg, 9.0 mmol, 225 mM), **3a** (1360 mg, 10 mmol, 250 mM), Cs_2CO_3 (1.5 mmol, 487 mg, 37.5 mM), nBu_4NBF_4 (1.5 mmol, 493 mg, 37.4 mM), CH_3CN (40 mL), constant current **10 mA for 11 h** (**4a**) reaction time, ***flow rate 60 µL min^{-1} (residence time = 12 min***, reactor vol. = 700 µL), productivity per day = 15.12 mmol day^{-1}, Pt loaded Cu foil (0.1 mm) anode, Pt loaded copper plate cathode.

[b]**Reaction conditions for batch**: **1a** (1008 mg, 7.0 mmol, 175 mM), **2a** (1017 mg, 9.0 mmol, 225 mM), **3a** (1360 mg, 10 mmol, 250 mM), Cs_2CO_3 (1.5 mmol, 487 mg, 37.5 mM), nBu_4NBF_4 (6 mmol, 1974 mg, 150 mM), CH_3CN (40 mL), constant current **30 mA for 43 h (4a)** reaction time, Pt plate anode, Pt plate cathode.

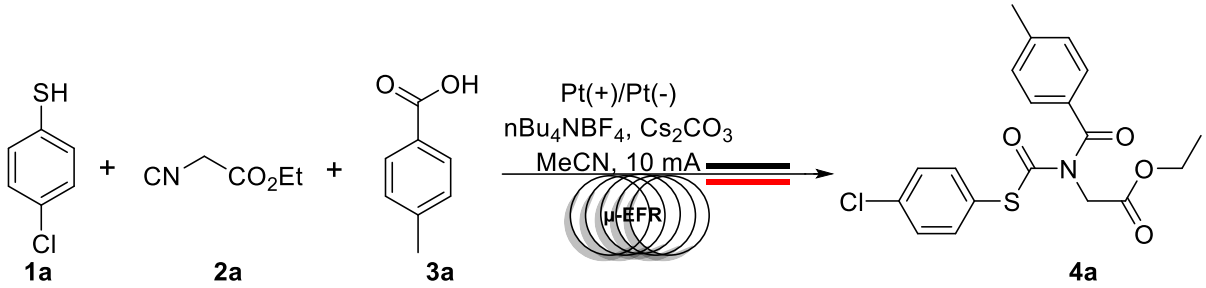


Table S3: Residence time optimization

Entry	Residence time (min.)	Yield (%)
1.	4 min.	25%
2.	8 min.	62%
3.	***12 min.***	***78%***

1.	16 min.	70%

After obtaining the optimized reaction conditions in batch, a procedure for the electrochemical synthesis of **4a** in flow using a simple micro electron flow reactor (μ-EFR) constructed from a PTFE sheets, tube and electrodes was developed to make the method more atom economical at gram scale synthesis with less time taking. This also allows us to make a comparison between batch and flow. Using the same conditions as those in batch with miner change in nBu_4NBF_4 with acetonitrile, 78% of **4a** was detected in **12 min.** reaction.

3. General procedures

3.1. **General procedure for preparation of diaryl deselenides (1l-n).**

Following a reported procedure, SeO_2 (1.0 equiv 5mmol), Substituted phenylboronic acid (1.2 equiv, 6mmol), and KI (1.0 equiv, 5mmol) were added to a round bottom flask with DMSO (12 mL). The mixture was stirred at 100 ℃ for 12 hours. After completion, the reaction was cooled to room temperature extracted (3 x 10 mL) with ethyl acetate. The organic layers were combined and washed with saturated NaCl and $Na_2S_2O_3$ solution and dried over anhydrous Na_2SO_4 and concentrated in vacuo. The crude residue was purified by silica gel column chromatography to give substituted diaryl deselenides.[2]

3.2. General Procedure for the Synthesis (4a-zt): In an oven-dried undivided cell (10 mL) equipped with thiol (0.5 mmol), ethyl 2-isocyanoacetate (0.7 mmol), carboxylic acid (1 mmol), nBu_4NBF_4 (0.5 mmol) and Cs_2CO_3 (0.1 mmol) were combined in MeCN (6.0 mL) as solvent. The reaction mixture was stirred and electrolyzed using Pt anode and Pt cathode at a constant current of 10 mA for 5 h at room temperature in inert (N_2) atmosphere. After the reaction was completed, the reaction was extracted with ethyl acetate (3 × 20 mL), and then the organic layer was washed with sodium bicarbonate and brine (20 mL) and dried with anhydrous Na_2SO_4. Subsequently, the solvent was removed under reduced pressure and the remaining crude product was purified by column chromatography over silica gel (EtOAc /hexane).

3.3. Cleaning procedures

This paragraph outlines the cleaning process for both the batch and microflow setups. Emphasizing the significance of electrode cleaning is crucial for ensuring the reaction's success, with potential differences in isolated yields of up to 20%.

3.3.1. Batch reactor

Initially, the vial, magnetic stirrer, and cap underwent a washing process using water followed by acetone, then they were dried. Next, the Pt electrode underwent washing with 0.01M HCl, after which it was immersed in a beaker containing acetone and subjected to sonication for 10 minutes. It's crucial to ensure the electrodes remain dry post-use (i.e., avoid submerging them in any solvent), as otherwise, the reaction ceases to progress.

3.3.2. Flow reactor

Initially, the gasket was cleaned on both sides with acetone. Subsequently, the Pt particle-loaded copper plate electrode underwent washing with 0.01M HCl, followed by rinsing with acetone. Then, the gasket, loops, and Pt particle-loaded copper plate electrode were immersed in a beaker filled with acetone and sonicated for 10 minutes. The Pt loaded Cu foil (0.1 mm thickness) was wiped with paper and rinsed with acetone thrice. Electrode holders were washed with acetone and dried using tissue paper. Copper contacts were first washed with 0.5M HCl, then scrubbed with a sponge if carbon deposits (anode) were observed. Finally, they were rinsed with acetone and reassembled. Following these processes, all components were dried with tissue paper, and the reactor was reassembled.

4. Mechanistic experiments

4.1. Cyclic Voltammetry analysis

All the cyclic voltammetry experiments were performed in a batch setup (gram scale) similar to the one used for the other experiments. The analysis was performed with an Metrohm Autolab PGSTAT101 (electrochemical work station) controlled by the NOVA control software package from Metrohm. Measurements were performed using an undivided cell equipped with a **Pt disk** working electrode, a **Pt wire** counter electrode and a **Ag/AgCl** reference electrode. All measurements were carried out in CH_3CN with nBu_4NBF_4 (15 mM or 51.8 mg). Unless specified, a **scan rate** of 0.05 V/s was used at **room temperature**. **1a** (10 mM), **2a** (10 mM), and **3a** (10 mM). **Positive** switching potential or direction of initial scan and initial potential was zero. The initial scan direction is oxidative, towards positive potentials. The undivided cell with the corresponding analyte was purged with nitrogen for 15 minutes before each CV run.

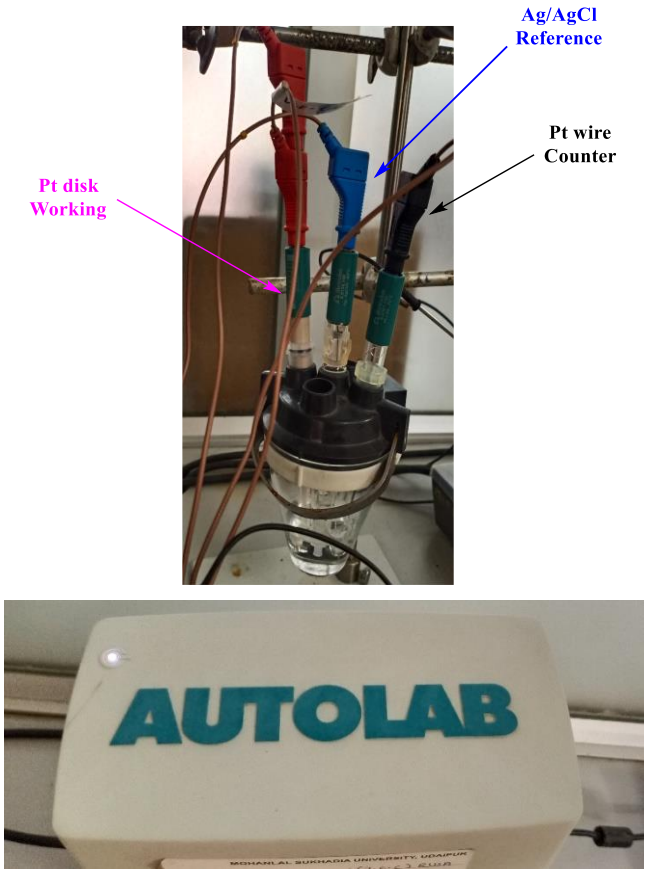


Figure S6: CV Set up

Blank:

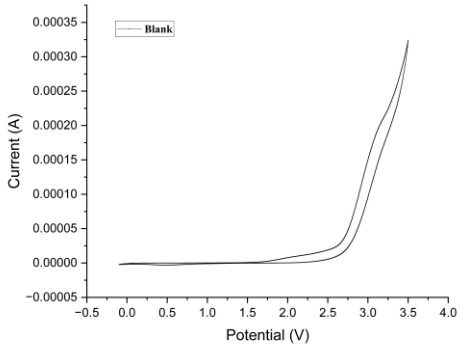


Figure S7: Blank

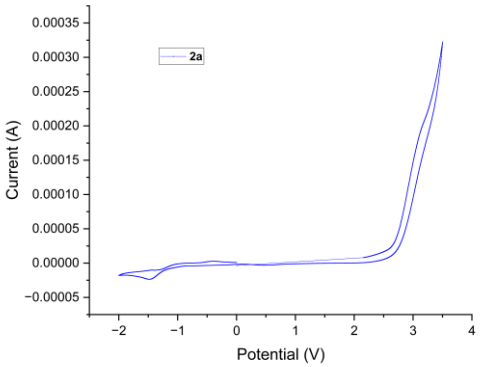


Figure S8: 2a

No significant oxidation peaks were observed for blank (Figure S7) and isocyanide (e.g., Ethyl isocynoacetate **2a**), which are required for a meaningful CV analysis. This lack of observation could be attributed to their poor interaction with the **Pt disk** electrode under the CV timescale. (Figure S8)

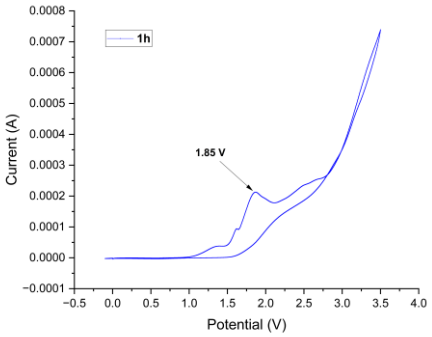


Figure S9: 1a

Cyclic voltammetry of 1a with a scan rate of 0.05 V/s. Onset potential was determined to be approximately 1.6 V vs Ag/AgCl while half-peak potential Ep/2 was determined to be around 1.8 V vs Ag/AgCl. (Figure S9)

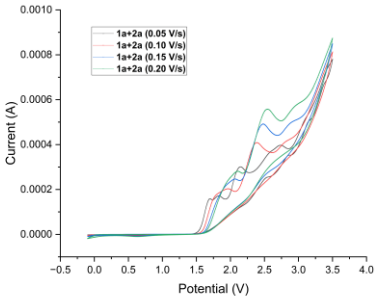


Figure S10: 1a+2a

Cyclic voltammetry of 1a+2a scanned after 1 min. at 0.05 V/s. The resulting CV plot shows anodic (oxidative) peak current, even at scan rates of 0.05 V/s. This possibly indicates an heterogenous electron transfer with a slow electron transfer rate from the **1a** to anode. This is further confirmed by the continuous increasing scan rate which shows clear interaction of **1a+2a.** (Figure S10)

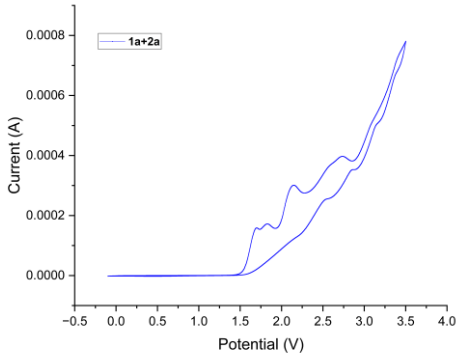


Figure S11

Cyclic voltammetry of **1a+2a** scanned after 10 min. at scan rate 0.05 V/s. Onset potential was determined to be approximately 1.7 V vs Ref, while half-peak potential Ep/2 was found to be around 1.8 V vs Ref. This suggests that the reaction is unlikely to proceed *via* the onset anodic oxidation of **1a** which would form a thiol radical cation. The thiol radical cation possibly interacts with **2a**, resulting in the emergence of another oxidation peak observed at 2.2 V.

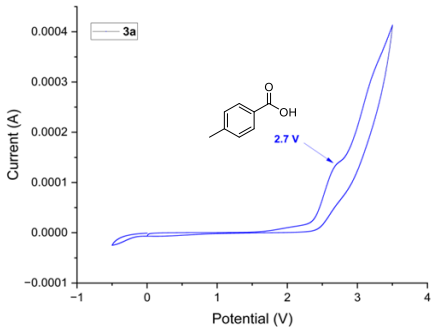


Figure S12

Cyclic voltammetry of **3a** scanned at scan rate 0.05 V/s. Onset potential was determined to be approximately 2.5 V vs Ref, while half-peak potential Ep/2 was found to be around 2.7 V vs Ref. This suggests that the **3a** is oxidized at higher potential respectively (Figure S12).

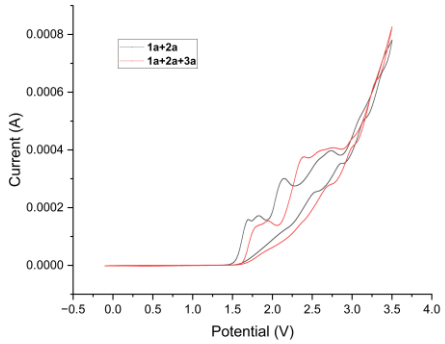


Figure S13

Comparative CV study of **1a+2a** and **1a+2a+3a** at scan rate 0.05 V/s in figure S13. This suggests that the possible intermediate peak observed by the addition **1a+2a** at 2.2 V was shifted to 2.4 V due to possible interaction with **3a.**

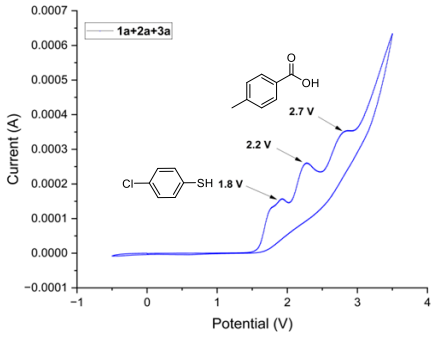


Figure S14

Cyclic voltammetry of **1a**+**2a**+**3a** scanned at scan rate 0.05 V/s.

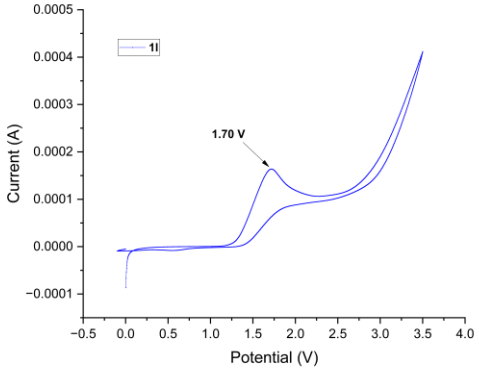


Figure S15

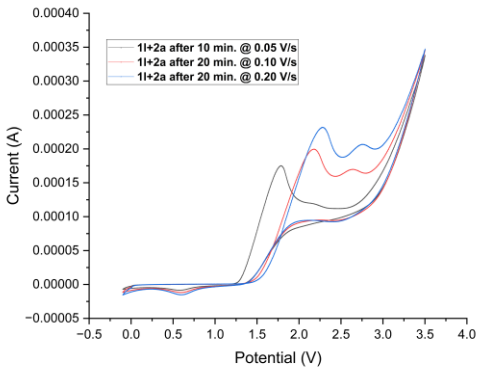


Figure S16

CV analysis of **1l** shows oxidation peak at 1.70 V (Figure S15). Further investigate reactivity of **1l** with **2a**, we take mixture CV of **1l**+**2a**. It shows another oxidation peak observed other than **1l** (Figure S16).

5. Calculation of Faradic efficiency

$$\text{Faradic efficiency } (n) = \frac{Q_{theoretical}}{Q_{experimental}} \times 100$$

$Q_{theoretical} = z \cdot N \cdot Y \cdot F$

$Q_{experimental} = z \cdot N \cdot Y \cdot$ electron equivlant(F/mol) {available directly from ElectraSyn 2.O}

n = Y/ electron equiv. or exp. charge

Yield (Y) = 87%

Experimental charge = 6.25 F/mol

n = 87/6.25 = 13.92%

6. Power consumes during reaction (P)

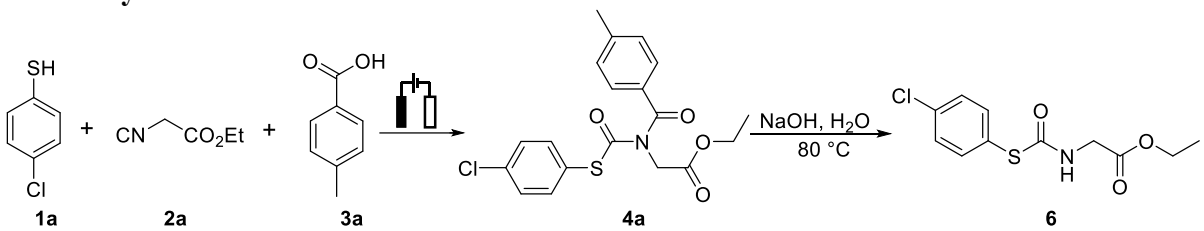
P = Voltage (V) x Current (A)

P = 5.63 x 0.01

P = 0.0563 W (*Watt*)

Power consumption per hour P/t = 0.0563 W/5 h = 0.01126 W/h

7. Post-Synthetic Modifications:



1a 2a 3a 4a 6

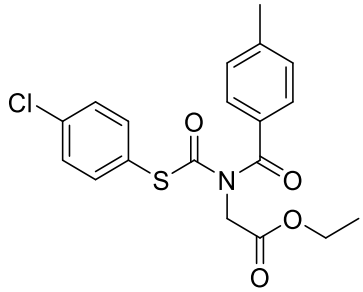
Prepared **4a** according to the above procedure. In a 10 mL round bottom flask equipped with a magnetic stir bar was added ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(4-methylbenzoyl)glycinate **4a** (156.4 mg, 0.4 mmol), and NaOH/H_2O (1.0 M, 5 mL) resulting

mixture was stirred at 80 °C temperature for 2 h. The aqueous layer was washed with brine and extracted with ethyl acetate (3 x 20 mL). The combined organic layer dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum. The crude material was purified by column chromatography (10% ethyl acetate in hexane) to furnish the desired product **6** (70 g, 65%).

8. Reference

1. M.E. Baumgärtner, J. Raub. *Platinum Metals Rev.* 1988, **32**, 188-197.
2. Y. Ren, B. Xu, Z. Zhong, C.U. Pittman, A. Zhou. *Org. Chem. Front.* 2019, **6**, 2023-2027.

9. Experimental data



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4a** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 170 mg (87%) of the title **4a**.

Physical State: Light brown solid.

Melting Point: 65-68 ^{0}C.

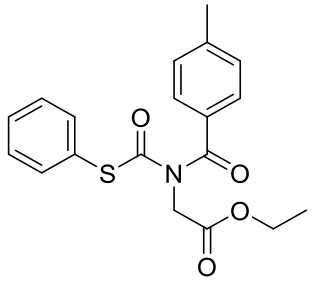
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 – 7.57 (m, 2H), 7.39 – 7.33 (m, 4H), 7.26 (s, 1H), 7.24 (s, 1H), 4.51 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 170.5, 168.0, 143.2, 136.5, 136.4, 131.2, 129.4, 128.3, 126.5, 61.8, 48.8, 21.7, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{19}ClNO_4S$ 392.0718; Found 392.0725.

FT-IR (vcm^{-1}): 2921, 2865, 1756, 1695, 1476, 1212, 884.

TLC: R_f = 0.55 (10% EtOAc/hexane).



Ethyl N-(4-methylbenzoyl)-N-((phenylthio)carbonyl)glycinate

The synthesis of **4b** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 132 mg (74%) of the title **4b**.

Physical State: White solid.

Melting Point: 62-65℃.

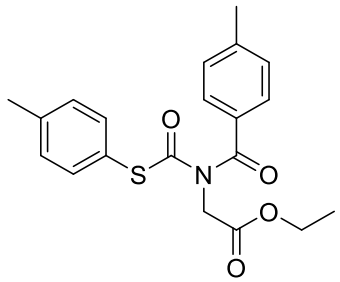
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 – 7.60 (m, 2H), 7.47 – 7.44 (m, 2H), 7.40 – 7.37 (m, 3H), 7.27 (s, 1H), 7.25 (s, 1H), 4.55 (s, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 2.41 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 170.0, 168.0, 143.1, 135.2, 131.5, 129.7, 129.3, 129.1, 128.4, 127.9, 61.7, 48.6, 21.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{20}NO_4S$ 358.1108; Found 358.1101.

FT-IR (vcm^{-1}): 2885, 2816, 1826, 1605, 1534, 1285, 804.

TLC: R_f = 0.50 (10% EtOAc/hexane).



Ethyl N-(4-methylbenzoyl)-N-((p-tolylthio)carbonyl)glycinate

The synthesis of **4c** was achieved according to the general procedure. Purification by column

chromatography (10% EtOAc/hexane) afforded 152 mg (82%) of the title **4c**.

Physical State: White solid.

Melting Point: 65-68 ^{0}C.

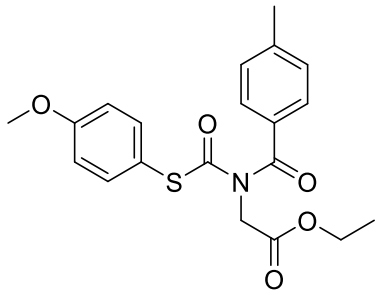
^{1}H NMR (400 MHz, CDCl$_3$): δ 7.62 – 7.60 (m, 2H), 7.27 – 7.26 (m, 5H), 7.21 – 7.19 (m, 1H), 4.56 (s, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 2.34 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 172.3, 170.0, 168.0, 142.9, 138.9, 135.6, 132.1, 131.5, 130.5, 129.1, 128.8, 128.3, 127.4, 61.6, 48.5, 21.5, 21.0, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_4S$ 372.1264; Found 372.1270.

FT-IR (vcm^{-1}): 2914, 2862, 1786, 1674, 1461, 1169, 863.

TLC: R_f = 0.45 (10% EtOAc/hexane).



Ethyl N-(((4-methoxyphenyl)thio)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4d** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 162 mg (84%) of the title **4d**.

Physical State: White solid.

Melting Point: 72-75 ^{0}C.

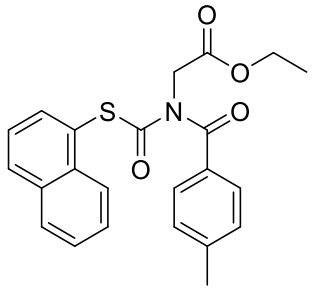
^{1}H NMR (400 MHz, CDCl$_3$): δ 7.61 – 7.59 (m, 2H), 7.37 – 7.34 (m, 2H), 7.25 – 7.23 (m, 2H), 6.90 – 6.88 (m, 2H), 4.54 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 3.77 (s, 3H), 2.39 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 172.2, 171.6, 168.0, 142.8, 136.7, 131.5, 129.1, 128.3, 118.3, 114.6, 61.5, 55.1, 48.4, 21.4, 13.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_5S$ 388.1213; Found 388.1207.

FT-IR (νcm^{-1}): 2958, 2886, 1862, 1691, 1563, 1123, 846.

TLC: R_f = 0.65 (15% EtOAc/hexane).



Ethyl N-(4-methylbenzoyl)-N-((naphthalen-1-ylthio)carbonyl)glycinate

The synthesis of **4e** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 142 mg (70%) of the title **4e**.

Physical State: Yellow solid.

Melting Point: 68-70 ^{0}C.

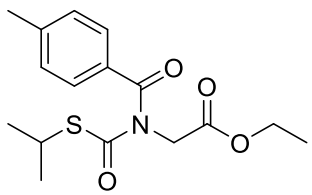
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 – 7.98 (m, 1H), 7.85 – 7.80 (m, 3H), 7.65 – 7.63 (m, 2H), 7.54 – 7.48 (m, 3H), 7.28 – 7.27 (m, 2H), 4.58 (s, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.42 (s, 3H), 1.29 (t, *J* = 7.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 171.0, 168.1, 143.1, 135.1, 133.5, 133.4, 131.5, 131.4, 129.3, 128.7, 128.5, 128.0, 127.7, 127.3, 126.5, 125.3, 61.7, 48.6, 21.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}NO_4S$ 408.1264; Found 408.1257.

FT-IR (νcm^{-1}): 2942, 2816, 1874, 1612, 1456, 1260, 815.

TLC: R_f = 0.55 (10% EtOAc/hexane).



Ethyl N-((isopropylthio)carbonyl)-N-(4-methylbenzoyl) glycinate

The synthesis of **4f** was achieved according to the general procedure. Purification by column chromatography (5% EtOAc/hexane) afforded 97 mg (60%) of the title **4f**.

Physical State: Light yellow viscous liquid.

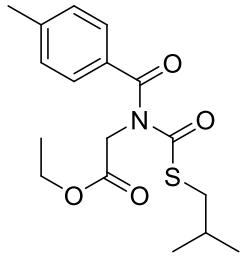
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 – 7.52 (m, 2H), 7.20 – 7.18 (m, 2H), 4.51 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 3.60 – 3.54 (m, 1H), 2.37 (s, 3H), 1.27 – 1.23 (m, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.0, 171.6, 168.1, 142.5, 131.9, 128.9, 128.3, 61.5, 47.8, 36.8, 22.5, 21.5, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{22}NO_4S$ 324.1264; Found 324.1261.

FT-IR (vcm^{-1}): 2912, 2841, 1724, 1654, 1406, 1185, 879.

TLC: R_f = 0.45 (5% EtOAc/hexane).



Ethyl N-((isobutylthio)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4g** was achieved according to the general procedure. Purification by column chromatography (5% EtOAc/hexane) afforded 108 mg (64%) of the title **4g**.

Physical State: Brown yellow solid.

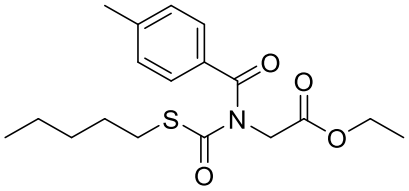
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 – 7.56 (m, 2H), 7.22 – 7.20 (m, 2H), 4.54 (s, 2H), 4.26 (q, *J* = 6.8 Hz, 2H), 2.39 (s, 3H), 1.43 (s, 9H), 1.30 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.1, 171.9, 168.4, 142.6, 132.2, 129.0, 128.5, 61.6, 49.4, 47.8, 29.7, 21.6, 14.1.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{24}NO_4S$ 338.1421; Found 338.1428.

FT-IR (vcm^{-1}): 2971, 2813, 1693, 1611, 1465, 1235, 819.

TLC: R_f = 0.45 (5% EtOAc/hexane).



Ethyl N-(4-methylbenzoyl)-N-((pentylthio)carbonyl)glycinate

The synthesis of **4h** was achieved according to the general procedure. Purification by column chromatography (5% EtOAc/hexane) afforded 123 mg (70%) of the title **4h**.

Physical State: Brown viscous liquid.

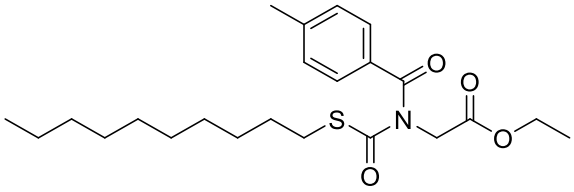
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 – 7.55 (m, 2H), 7.23 – 7.20 (m, 2H), 4.56 (s, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.87 (t, *J* = 7.2 Hz, 2H), 2.39 (s, 3H), 1.58 – 1.54 (m, 2H), 1.31 – 1.26 (m, 7H), 0.88 – 0.84 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.2, 172.0, 168.3, 142.8, 132.0, 129.0, 128.4, 61.6, 48.0, 31.2, 30.8, 28.8, 22.1, 21.6, 14.0, 13.8.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{26}NO_4S$ 352.1577; Found 352.1569.

FT-IR (νcm^{-1}): 2924, 2858, 1768, 1641, 1425, 1213, 880.

TLC: R_f = 0.45 (5% EtOAc/hexane).



Ethyl N-((decylthio)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4i** was achieved according to the general procedure. Purification by column chromatography (8% EtOAc/hexane) afforded 139 mg (66%) of the title **4i**.

Physical State: Yellowish white solid.

Melting Point: 65-67℃.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 – 7.54 (m, 2H), 7.21 – 7.19 (m, 2H), 4.55 (s, 2H), 4.24 (q, *J* = 7.2 Hz, 2H), 2.86 (t, *J* = 7.2 Hz, 2H), 2.38 (s, 3H), 1.58 – 1.51 (m, 2H), 1.28 – 1.23 (m,

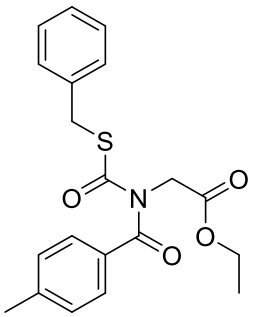
17H), 0.88 (t, J = 7.2 Hz, 3H),

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.0, 171.9, 168.1, 142.6, 131.9, 128.9, 128.3, 61.5, 47.9, 31.7, 31.1, 29.4, 29.3, 29.1, 29.0, 28.9, 28.6, 22.5, 21.5, 13.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{36}NO_4S$ 422.2360; Found 422.2354.

FT-IR (vcm^{-1}): 2916, 2850, 1731, 1643, 1422, 1212, 882.

TLC: R_f = 0.40 (8% EtOAc/hexane).



Ethyl N-((isobutylthio)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4j** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 124 mg (67%) of the title **4j**.

Physical State: White solid.

Melting Point: 70-73 ^{0}C.

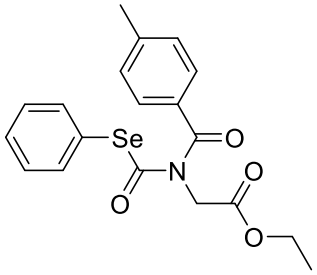
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 – 7.52 (m, 2H), 7.29 – 7.26 (m, 4H), 7.25 – 7.20 (m, 3H), 4.54 (s, 2H), 4.24 (q, J = 7.2 Hz, 2H), 4.13 (s, 2H), 2.40 (s, 3H), 1.27 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.1, 171.4, 168.1, 142.8, 136.5, 131.6, 129.1, 129.0, 128.5, 128.3, 127.3, 61.6, 48.2, 35.6, 21.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_4S$ 372.1264; Found 372.1261.

FT-IR (vcm^{-1}): 2951, 2832, 1753, 1640, 1461, 1187, 846.

TLC: R_f = 0.50 (15% EtOAc/hexane).



Ethyl N-(4-methylbenzoyl)-N-((phenylselanyl)carbonyl)glycinate

The synthesis of **4l** was achieved according to the general procedure. Purification by column chromatography (8% EtOAc/hexane) afforded 149 mg (74%) of the title **4l**.

Physical State: brown solid.

Melting Point: 65-68 ^{0}C.

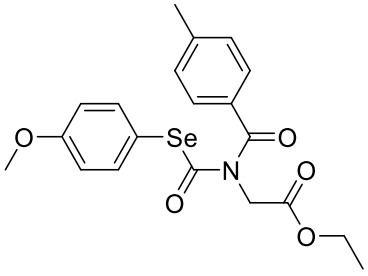
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 – 7.60 (m, 2H), 7.54 – 7.52 (m, 2H), 7.40 – 7.37 (m, 3H), 7.28 – 7.26 (m, 2H), 4.41 (s, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 2.42 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.2, 169.4, 167.8, 142.8, 136.3, 130.8, 129.5, 129.2, 129.1, 128.2, 127.7, 61.7, 49.4, 21.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{20}NO_4Se$ 406.0552; Found 406.0558.

FT-IR (vcm^{-1}): 2904, 2819, 1736, 1619, 1429, 1173, 853.

TLC: R_f = 0.55 (15% EtOAc/hexane).



Ethyl N-(((4-methoxyphenyl)selanyl)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4m** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 154 mg (71%) of the title **4m**.

Physical State: Light yellow solid.

Melting Point: 70-72 ^{0}C.

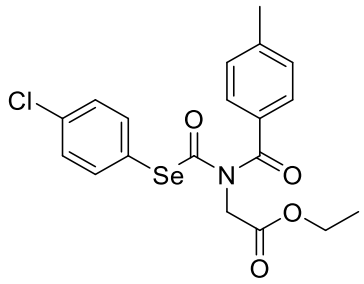
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 – 7.59 (m, 2H), 7.30 – 7.26 (m, 2H), 7.24 (s, 1H), 7.06 – 7.03 (m, 1H), 7.00 – 6.99 (m, 1H), 6.95 – 6.91 (m, 1H), 4.54 (s, 2H), 4.24 (q, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 2.40 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 170.8, 168.0, 159.6, 143.0, 131.5, 129.8, 129.2, 128.7, 128.4, 127.4, 120.2, 115.8, 61.6, 55.2, 48.6, 21.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_5Se$ 436.0658; Found 436.0653.

FT-IR (νcm^{-1}): 2911, 2867, 1738, 1672, 1421, 1142, 816.

TLC: R_f = 0.55 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)selanyl)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4n** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 142 mg (65%) of the title **4n**.

Physical State: Light brown solid.

Melting Point: 71-73 ^{0}C.

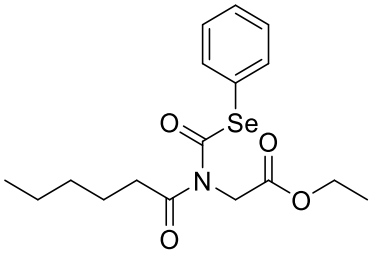
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 – 7.57 (m, 2H), 7.51 – 7.48 (m, 2H), 7.33 – 7.31 (m, 2H), 7.27 (s, 1H), 7.24 (s, 1H), 4.51 (s, 2H), 4.24 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 170.2, 167.9, 143.1, 136.6, 132.2, 131.1, 129.3, 128.2, 127.1, 124.4, 61.7, 48.7, 21.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{19}ClNO_4Se$ 440.0162; Found 440.0167.

FT-IR (νcm^{-1}): 2981, 2860, 1764, 1670, 1460, 1135, 828.

TLC: R_f = 0.50 (10% EtOAc/hexane).



Ethyl *N*-hexanoyl-N-((phenylselanyl)carbonyl)glycinate

The synthesis of **4o** was achieved according to the general procedure. Purification by column chromatography (5% EtOAc/hexane) afforded 117 mg (61%) of the title **4o**.

Physical State: White solid.

Melting Point: 70-73 ^{0}C.

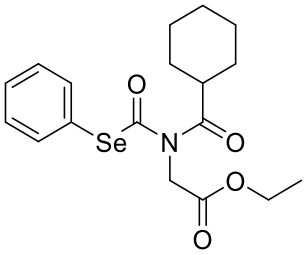
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 – 7.57 (m, 2H), 7.41 – 7.33 (m, 3H), 4.48 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 2.57 – 2.50 (m, 1H), 1.86 – 1.80 (m, 4H), 1.61 – 1.51 (m, 2H), 1.31 – 1.24 (m, 7H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 178.2, 169.2, 167.7, 136.1, 129.0, 128.9, 128.4, 61.7, 46.3, 43.6, 28.8, 25.3, 25.2, 13.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{24}NO_4Se$ 386.0865; Found 386.0859.

FT-IR (νcm^{-1}): 2970, 2854, 1763, 1650, 1433, 1226, 847.

TLC: R_f = 0.40 (5% EtOAc/hexane).



Ethyl N-(cyclohexanecarbonyl)-N-((phenylselanyl)carbonyl)glycinate

The synthesis of **4p** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 134 mg (68%) of the title **4p**.

Physical State: Bone white solid.

Melting Point: 65-68 ^{0}C.

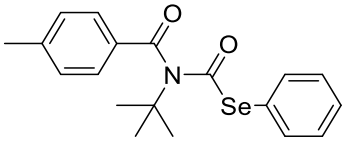
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 – 7.58 (m, 2H), 7.40 – 7.34 (m, 3H), 4.46 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 2.52 (t, *J* = 7.2 Hz, 2H), 1.74 – 1.67 (m, 2H), 1.35 – 1.31 (m, 4H), 1.28 (t, *J* = 6.8 Hz, 3H), 0.92 – 0.89 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 175.0, 168.9, 167.5, 136.2, 129.1, 128.9, 128.3, 61.8, 46.2, 35.8, 30.9, 23.5, 22.3, 13.9, 13.7.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{24}NO_4Se$ 398.0865; Found 398.0871.

FT-IR (vcm^{-1}): 2942, 2873, 1728, 1631, 1508, 1210, 830.

TLC: R_f = 0.40 (10% EtOAc/hexane).



Se-phenyltert-butyl(4-methylbenzoyl)carbamoselenoate

The synthesis of **4q** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 122 mg (55%) of the title **4q**.

Physical State: Light brown solid.

Melting Point: 65-68 ^{0}C.

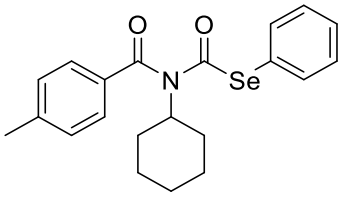
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 – 7.98 (m, 2H), 7.51 – 7.48 (m, 2H), 7.37 – 7.32 (m, 5H), 2.48 (s, 3H), 1.53 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.9, 160.3, 146.1, 136.2, 132.5, 131.2, 129.8, 128.9, 128.8, 127.7, 61.3, 28.5, 21.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{22}NO_2Se$ 376.0810; Found 376.0803.

FT-IR (vcm^{-1}): 1782, 1715, 1508, 1272, 1003, 846.

TLC: R_f = 0.40 (10% EtOAc/hexane).



Ethyl N-(cyclohexanecarbonyl)-N-((phenylselanyl)carbonyl)glycinate

The synthesis of **4r** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 114 mg (57%) of the title **4r**.

Physical State: Light brown solid.

Melting Point: 68-70 ^{0}C.

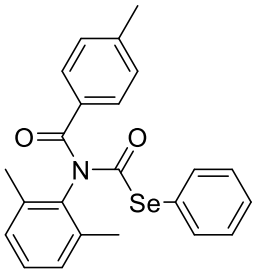
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 – 7.61 (m, 2H), 7.59 – 7.57 (m, 2H), 7.41 – 7.34 (m, 3H), 7.30 – 7.27 (m, 2H), 3.96 – 3.90 (m, 1H), 2.44 (s, 3H), 2.25 – 2.15 (m, 2H), 1.89 – 1.77 (m, 4H), 1.58 – 1.56 (m, 1H), 1.19 – 1.14 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 174.3, 167.3, 143.3, 136.2, 132.1, 129.2, 128.9, 128.8, 128.4, 128.2, 61.8, 30.1, 26.1, 24.8, 21.5.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}NO_2Se$ 402.0967; Found 402.0971.

FT-IR (vcm^{-1}): 1765, 1724, 1536, 1251, 1045, 806.

TLC: R_f = 0.50 (10% EtOAc/hexane).



Se-phenyl(2,6-dimethylphenyl)(4-methylbenzoyl)carbamoselenoate

The synthesis of **4s** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 88 mg (42%) of the title **4s**.

Physical State: White solid.

Melting Point: 75-77 ^{0}C.

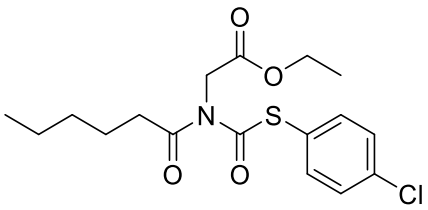
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 – 7.52 (m, 4H), 7.35 – 7.27 (m, 4H), 7.17 – 7.14 (m, 4H), 2.37 (s, 6H), 2.35 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.2, 169.1, 142.6, 137.7, 136.0, 131.5, 129.9, 129.1, 129.0, 128.9, 128.7, 128.6, 128.2, 127.6, 21.6, 18.4.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}NO_2Se$ 424.0810; Found 424.0813.

FT-IR (vcm^{-1}): 1794, 1770, 1567, 1278, 1086, 857.

TLC: R_f = 0.45 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-hexanoylglycinate

The synthesis of **4t** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 128 mg (69%) of the title **4t**.

Physical State: White solid.

Melting Point: 60-62 ^{0}C.

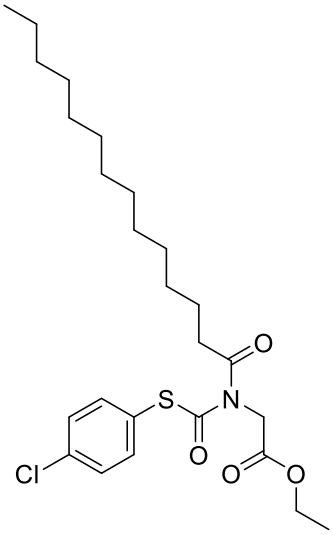
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.37 (m, 4H), 4.55 (s, 2H), 4.27 (q, J = 7.2 Hz, 2H), 2.27 (t, J = 7.6 Hz, 2H), 1.69 – 1.62 (m, 2H), 1.31 – 1.27 (m, 7H), 0.89 (t, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 174.6, 169.8, 167.7, 136.6, 136.4, 129.4, 126.1, 61.8, 46.0, 37.4, 31.0, 23.9, 22.3, 14.0, 13.8.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{23}ClNO_4S$ 372.1031; Found 372.1028.

FT-IR (vcm^{-1}): 2938, 2864, 1763, 1620, 1412, 1206, 891.

TLC: R_f = 0.45 (10% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-tetradecanoylglycinate

The synthesis of **4u** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 154 mg (64%) of the title **4u**.

Physical State: White solid.

Melting Point: 62-65 ^{0}C.

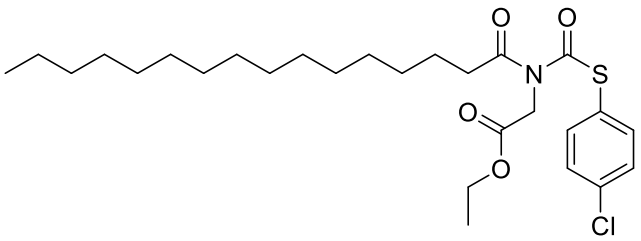
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.37 (m, 4H), 4.55 (s, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.71 (t, *J* = 7.6 Hz, 2H), 1.66 – 1.61 (m, 2H), 1.30 – 1.23 (m, 23H), 0.88 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 174.6, 169.8, 167.7, 136.6, 136.4, 129.4, 126.1, 61.8, 46.0, 37.4, 31.8, 29.6, 29.5, 29.4, 29.3, 28.9, 24.2, 22.6, 14.1, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{25}H_{39}ClNO_4S$ 484.2283; Found 484.2276.

FT-IR (vcm^{-1}): 2935, 2861, 1758, 1629, 1410, 1216, 865.

TLC: R_f = 0.45 (10% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-palmitoylglycinate

The synthesis of **4v** was achieved according to the general procedure. Purification by column

chromatography (10% EtOAc/hexane) afforded 157 mg (60%) of the title **4v**.

Physical State: White solid.

Melting Point: 65-68 ^{0}C.

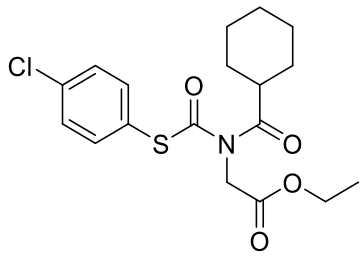
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.38 (m, 4H), 4.56 (s, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 1.65 – 1.63 (m, 2H), 1.31 – 1.24 (m, 27H), 0.89 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 174.7, 169.9, 167.7, 136.7, 136.5, 129.5, 126.1, 61.9, 46.0, 37.5, 31.9, 29.6, 29.5, 29.4, 29.3, 28.9, 24.3, 22.7, 14.1.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{27}H_{43}ClNO_4S$ 512.2596; Found 512.2588.

FT-IR (vcm^{-1}): 2932, 2866, 1753, 1635, 1418, 1213, 871.

TLC: R_f = 0.50 (10% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(cyclohexanecarbonyl)glycinate

The synthesis of **4w** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 134 mg (70%) of the title **4w**.

Physical State: Yellow solid.

Melting Point: 55-57 ^{0}C.

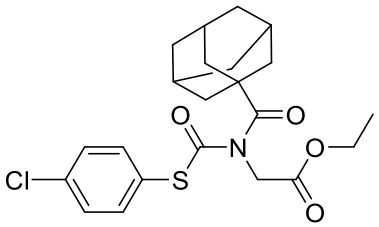
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 – 7.35 (m, 4H), 4.55 (s, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 3.00 – 2.93 (m, 1H), 1.88 – 1.73 (m, 4H), 1.65 – 1.61 (m, 1H), 1.50 – 1.41 (m, 2H), 1.29 – 1.20 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 177.7, 169.5, 167.7, 136.5, 136.3, 129.4, 126.0, 61.7, 46.1, 44.4, 29.1, 25.5, 25.3, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{23}ClNO_4S$ 384.1031; Found 384.1025.

FT-IR (vcm^{-1}): 2950, 2832, 1784, 1612, 1476, 1165, 813.

TLC: R_f = 0.40 (10% EtOAc/hexane).



Ethyl N-(adamantane-1-carbonyl)-N-(((4-chlorophenyl)thio)carbonyl)glycinate

The synthesis of **4x** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 126 mg (58%) of the title **4x**.

Physical State: White solid.

Melting Point: 58-62 ^{0}C.

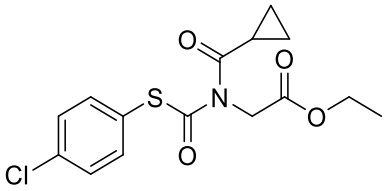
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.36 (m, 4H), 4.53 (s, 2H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.04 (s, 9H), 1.70 (s, 6H), 1.30 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 183.4, 170.6, 168.0, 136.4, 136.2, 129.4, 126.4, 61.7, 48.5, 45.4, 38.4, 36.2, 28.1, 14.1.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}ClNO_4S$ 436.1344; Found 436.1351.

FT-IR (vcm^{-1}): 2942, 2836, 1783, 1646, 1450, 1113, 854.

TLC: R_f = 0.45 (10% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(cyclopropanecarbonyl)glycinate

The synthesis of **4y** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 93 mg (55%) of the title **4y**.

Physical State: White solid.

Melting Point: 53-55 ^{0}C.

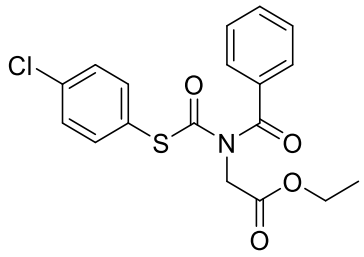
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 – 7.35 (m, 4H), 4.63 (s, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 2.17 – 2.11 (m, 1H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.20 – 1.16 (m, 2H), 1.00 – 0.96 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 175.5, 170.0, 167.7, 136.5, 136.2, 129.3, 126.6, 61.8, 46.4, 14.7, 14.0, 10.6.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{15}H_{17}ClNO_4S$ 342.0561; Found 342.0563.

FT-IR (vcm^{-1}): 2963, 2830, 1761, 1625, 1474, 1136, 814.

TLC: R_f = 0.40 (10% EtOAc/hexane).



Ethyl N-benzoyl-N-(((4-chlorophenyl)thio)carbonyl)glycinate

The synthesis of **4z** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 151 mg (80%) of the title **4z**.

Physical State: Bone white solid.

Melting Point: 70-73 ^{0}C.

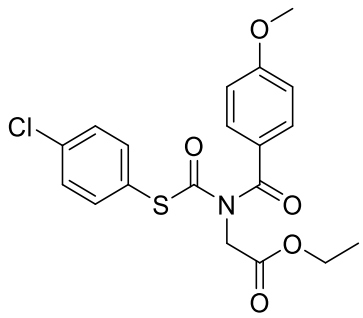
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 – 7.66 (m, 2H), 7.56 – 7.52 (m, 1H), 7.47 – 7.43 (m, 2H), 7.40 – 7.33 (m, 4H) 4.51 (s, 2H), 4.23 (q, *J* = 6.8 Hz, 2H), 1.27 (t, *J* = 7.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.3, 170.4, 167.8, 136.4, 136.1, 134.1, 132.2, 129.3, 128.6, 127.9, 126.4, 61.7, 48.7, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{17}ClNO_4S$ 378.0561; Found 378.0565.

FT-IR (vcm^{-1}): 2980, 2850, 1726, 1670, 1461, 1208, 829.

TLC: R_f = 0.50 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(4-methoxybenzoyl)glycinate

The synthesis of **4za** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 169 mg (83%) of the title **4za**.

Physical State: Light brown solid.

Melting Point: 71-73 ^{0}C.

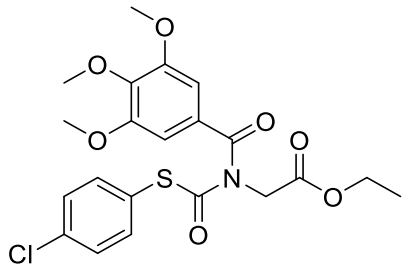
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 – 7.70 (m, 2H), 7.38 – 7.32 (m, 4H), 6.96 – 6.93 (m, 2H), 4.54 (s, 2H), 4.24 (q, *J* = 6.8 Hz, 2H), 3.86 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 170.3, 168.1, 163.3, 136.4, 136.2, 131.1, 129.3, 126.5, 126.1, 113.9, 61.7, 55.4, 48.8, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{19}ClNO_5S$ 408.0667; Found 408.0661.

FT-IR (vcm^{-1}): 2996, 2827, 1751, 1623, 1418, 1265, 821.

TLC: R_f = 0.65 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(3,4,5-trimethoxybenzoyl)glycinate

The synthesis of **4zb** was achieved according to the general procedure. Purification by column chromatography (25% EtOAc/hexane) afforded 182 mg (78%) of the title **4zb**.

Physical State: Brown solid.

Melting Point: 76-78 ^{0}C.

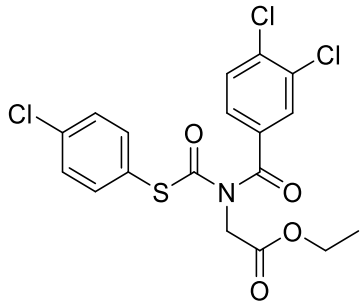
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 – 7.33 (m, 4H), 6.96 (s, 2H), 4.46 (s, 2H), 4.22 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 3.85 (s, 6H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.0, 170.6, 168.1, 153.2, 141.4, 136.4, 136.2, 129.3, 128.7, 126.5, 105.4, 61.8, 60.9, 56.1, 49.1, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{23}ClNO_7S$ 468.0878; Found 468.0876.

FT-IR (vcm^{-1}): 2978, 2861, 1748, 1653, 1452, 1196, 813.

TLC: R_f = 0.50 (25% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(3,4-dichlorobenzoyl)glycinate

The synthesis of **4zc** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 180 mg (81%) of the title **4zc**.

Physical State: Dark yellow solid.

Melting Point: 72-75 ^{0}C.

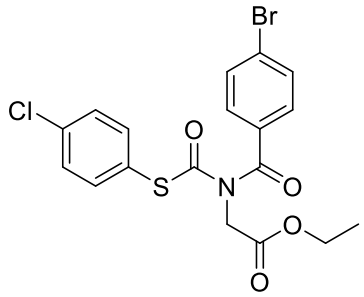
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 – 7.76 (m, 1H), 7.52 – 7.51 (m, 2H), 7.37 (m, 4H), 4.56 (s, 2H), 4.28 (q, *J* = 6.8 Hz, 2H), 1.31 (t, *J* = 7.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.3, 170.0, 167.7, 136.8, 136.7, 136.4, 134.1, 133.2, 130.7, 130.0, 129.6, 127.1, 125.3, 62.1, 48.1, 14.1.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{15}Cl_3NO_4S$ 445.9782; Found 445.9776.

FT-IR (vcm^{-1}): 2962, 2858, 1730, 1643, 1420, 1218, 856.

TLC: R_f = 0.45 (15% EtOAc/hexane).



Ethyl N-(4-bromobenzoyl)-N-(((4-chlorophenyl)thio)carbonyl)glycinate

The synthesis of **4zd** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 194 mg (85%) of the title **4zd**.

Physical State: Brown solid.

Melting Point: 68-70 ^{0}C.

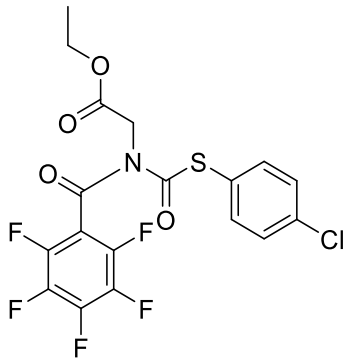
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 – 7.54 (m, 4H), 7.39 – 7.34 (m, 4H), 4.53 (s, 2H), 4.26 (q, J = 7.2 Hz, 2H), 1.29 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.4, 170.3, 167.8, 136.5, 136.4, 133.2, 132.0, 129.6, 129.5, 127.1, 125.8, 62.0, 48.4, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{16}BrClNO_4S$ 455.9666; Found 455.9658.

FT-IR (νcm^{-1}): 2954, 2868, 1728, 1624, 1432, 1209, 850.

TLC: R_f = 0.45 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(perfluorobenzoyl)glycinate

The synthesis of **4zf** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 175 mg (75%) of the title **4zf**.

Physical State: Orange white solid.

Melting Point: 72-75 ^{0}C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 – 7.36 (m, 4H), 4.73 (s, 2H), 4.34 (q, *J* = 7.2 Hz, 2H), 1.36 (t, *J* = 7.2 Hz, 3H).

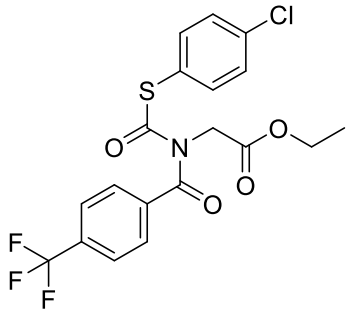
^{13}C NMR (100 MHz, $CDCl_3$): δ 169.7, 166.4, 137.2, 136.6, 129.8, 123.7, 62.3, 46.4, 14.0.

^{19}F NMR (376 MHz $CDCl_3$): δ 141.3, 150.4, 160.2.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{12}ClF_5NO_4S$ 468.0090; Found 468.0088.

FT-IR (νcm^{-1}): 2998, 2918, 1826, 1705, 1562, 1262, 952.

TLC: R_f = 0.55 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(4-(trifluoromethyl)benzoyl)glycinate

The synthesis of **4zg** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 182 mg (82%) of the title **4zg**.

Physical State: White solid.

Melting Point: 65-67 ^{0}C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 – 7.76 (m, 2H), 7.72 – 7.70 (m, 2H), 7.39 – 7.34 (m, 4H), 4.56 (s, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H)

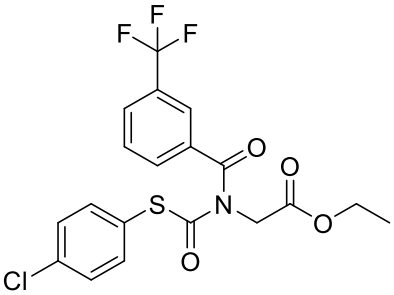
^{13}C NMR (100 MHz, $CDCl_3$): δ 171.0, 170.3, 167.7, 138.0, 136.6, 136.4, 133.6, 133.3, 129.6, 128.1, 125.7, 125.6, 125.5, 62.1, 48.1, 14.0.

^{19}F NMR (376 MHz $CDCl_3$): δ 63.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{16}ClF_3NO_4S$ 446.0435; Found 446.0433.

FT-IR (vcm^{-1}): 2960, 2846, 1740, 1654, 1425, 1204, 883.

TLC: R_f = 0.40 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(3-(trifluoromethyl)benzoyl)glycinate

The synthesis of **4zh** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 164 mg (74%) of the title **4zh**.

Physical State: White solid.

Melting Point: 63-65 ^{0}C.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (s, 1H), 7.87 – 7.85 (m, 1H), 7.80 – 7.77 (m, 1H), 7.60 – 7.57 (m, 1H), 7.38 – 7.33 (m, 4H), 4.57 (s, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H).

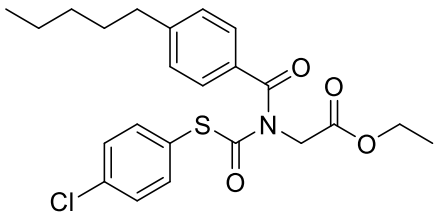
^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 170.3, 167.7, 136.5, 136.4, 135.2, 131.1, 130.9, 129.5, 129.3, 128.6, 128.5, 125.5, 124.9, 124.8, 122.0, 62.0, 48.1, 14.0.

^{19}F NMR (376 MHz $CDCl_3$): δ 62.7.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{16}ClF_3NO_4S$ 446.0435; Found 446.0437.

FT-IR (vcm^{-1}): 2945, 2860, 1721, 1651, 1456, 1223, 835.

TLC: R_f = 0.40 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(4-pentylbenzoyl)glycinate

The synthesis of **4zi** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 181 mg (81%) of the title **4zi**.

Physical State: White solid.

Melting Point: 55-57 ^{0}C.

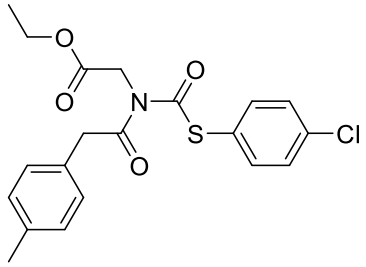
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 – 7.59 (m, 2H), 7.40 – 7.33 (m, 4H), 7.27 (s, 1H), 7.25 (s, 1H), 4.52 (s, 2H), 4.24 (q, *J* = 7.2 Hz, 2H), 4.26 (t, *J* = 7.6 Hz, 2H), 1.67 – 1.59 (m, 2H), 1.34 – 1.30 (m, 4H), 1.27 (t, *J* = 7.2 Hz, 3H), 0.90 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 170.5, 168.0, 148.1, 136.4, 136.1, 131.4, 129.3, 128.7, 128.3, 126.6, 61.7, 48.8, 35.9, 31.3, 30.7, 22.4, 14.0, 13.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{27}ClNO_4S$ 448.1344; Found 448.1339.

FT-IR (vcm^{-1}): 2965, 2843, 1735, 1647, 1434, 1260, 815.

TLC: R_f = 0.40 (10% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(2-(p-tolyl)acetyl)glycinate

The synthesis of **4zj** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 154 mg (76%) of the title **4zj**.

Physical State: White solid.

Melting Point: 60-62 ^{0}C.

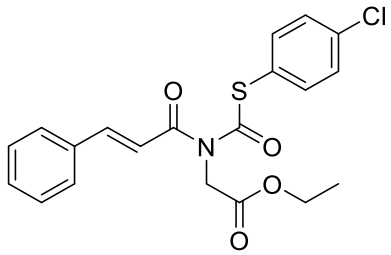
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 – 7.39 (m, 4H), 7.17 – 7.11 (m, 4H), 4.58 (s, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 4.04 (s, 2H), 2.34 (s, 3H), 1.31 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.8, 170.1, 167.5, 136.9, 136.6, 136.4, 129.9, 129.4, 129.3, 129.2, 126.0, 61.9, 46.2, 43.6, 21.3, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{21}ClNO_4S$ 406.0874; Found 406.0873.

FT-IR (vcm^{-1}): 2923, 2841, 1713, 1672, 1407, 1185, 829.

TLC: R_f = 0.55 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-cinnamoylglycinate

The synthesis of **4zk** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 143 mg (78%) of the title **4zk**.

Physical State: White solid.

Melting Point: 60-63 ^{0}C.

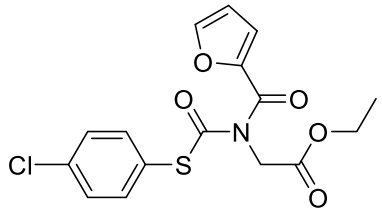
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 – 7.81 (m, 1H), 7.55 – 7.53 (m, 2H), 7.47 – 7.44 (m, 2H), 7.42 – 7.37 (m, 4H), 7.37 – 7.36 (m, 1H), 7.23 – 7.19 (m, 1H), 4.68 (s, 2H), 4.30 (q, *J* = 6.8 Hz, 2H), 1.33 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.0, 167.7, 167.2, 146.7, 136.6, 136.4, 134.2, 130.6, 129.4, 128.8, 128.4, 128.3, 125.8, 118.6, 61.8, 46.4, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{19}ClNO_4S$ 404.0718; Found 404.0711.

FT-IR (vcm^{-1}): 2953, 2824, 1765, 1687, 1640, 1468, 1241, 834.

TLC: R_f = 0.35 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(furan-2-carbonyl)glycinate

The synthesis of **4zl** was achieved according to the general procedure. Purification by column

chromatography (20% EtOAc/hexane) afforded 126 mg (69%) of the title **4zl**.

Physical State: Light red solid.

Melting Point: 70-72 ^{0}C.

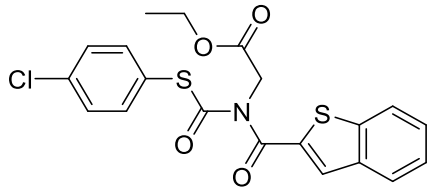
^{1}H NMR (400 MHz, CDCl$_3$): δ 7.57 – 7.56 (m, 1H), 7.43 – 7.40 (m, 2H), 7.38 – 7.34 (m, 3H), 6.58 – 6.57 (m, 1H), 4.72 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 170.6, 167.8, 160.2, 146.3, 145.9, 136.4, 136.0, 129.2, 127.0, 121.4, 112.6, 61.6, 47.6, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{15}ClNO_5S$ 368.0354; Found 368.03557.

FT-IR (vcm^{-1}): 2926, 2816, 1754, 1660, 1421, 1246, 882.

TLC: R_f = 0.60 (20% EtOAc/hexane).



Ethyl N-(benzo[b]thiophene-2-carbonyl)-N-(((4-chlorophenyl)thio)carbonyl)glycinate

The synthesis of **4zm** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 138 mg (78%) of the title **4zm**.

Physical State: Brown solid.

Melting Point: 56-58 ^{0}C.

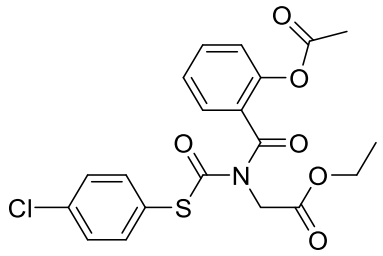
^{1}H NMR (400 MHz, CDCl$_3$): δ 7.90 – 7.87 (m, 3H), 7.50 – 7.39 (m, 4H), 7.37 – 7.35 (m, 2H), 4.66 (s, 2H), 4.29 (q, *J* = 7.2 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 170.4, 167.9, 166.4, 141.8, 138.1, 136.4, 136.3, 135.4, 130.0, 129.4, 127.4, 126.4, 125.7, 125.2, 122.6, 61.9, 49.1, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{17}ClNO_4S_2$ 434.0282; Found 434.0276.

FT-IR (vcm^{-1}): 2958, 2840, 1794, 1635, 1485, 1204, 862.

TLC: R_f = 0.50 (15% EtOAc/hexane).



Ethyl N-(2-acetoxybenzoyl)-N-(((4-chlorophenyl)thio)carbonyl)glycinate

The synthesis of **4zn** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 152 mg (70%) of the title **4zn**.

Physical State: White solid.

Melting Point: 51-53 ^{0}C.

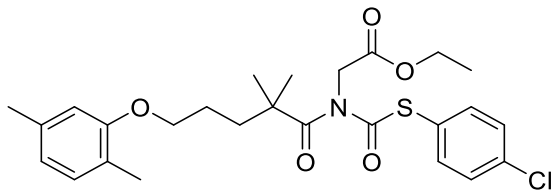
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 – 7.49 (m, 2H), 7.43 – 7.40 (m, 2H), 7.37 – 7.35 (m, 2H), 7.31 – 7.27 (m, 1H), 7.20 – 7.18 (m, 1H), 4.43 (s, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 2.31 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 169.3, 169.1, 167.9, 147.4, 136.6, 132.5, 129.5, 128.0, 127.9, 126.6, 126.2, 123.3, 61.9, 48.6, 21.1, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{19}ClNO_6S$ 436.0616; Found 436.0615.

FT-IR (νcm^{-1}): 2971, 2816, 1760, 1613, 1432, 1186, 818.

TLC: R_f = 0.40 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoyl)glycinate

The synthesis of **4zo** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 149 mg (59%) of the title **4zo**.

Physical State: White solid.

Melting Point: 55-58 ^{0}C.

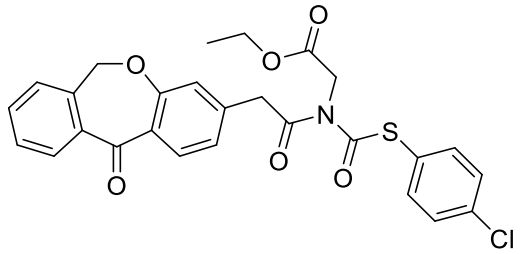
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 – 7.38 (m, 4H), 7.03 – 7.01 (m, 1H), 6.69 – 6.67 (m, 1H), 6.63 (s, 1H), 4.57 (s, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 3.95 (t, *J* = 6.0 Hz, 2H), 2.32 (s, 3H), 2.18 (s, 3H), 1.93 – 1.78 (m, 4H), 1.39 (s, 6H), 1.32 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 181.4, 170.7, 167.8, 156.7, 136.4, 136.3, 136.2, 130.2, 129.4, 126.4, 123.3, 120.6, 111.7, 67.5, 61.7, 48.2, 45.8, 37.8, 25.7, 25.0, 21.3, 15.7, 14.0.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{26}H_{33}ClNO_5S$ 506.1762; Found 506.1769.

FT-IR (νcm^{-1}): 2991, 2861, 1723, 1639, 1456, 1263, 876.

TLC: R_f = 0.55 (15% EtOAc/hexane).



Ethyl N-(((4-chlorophenyl)thio)carbonyl)-N-(2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-3-yl)acetyl)glycinate

The synthesis of **4zp** was achieved according to the general procedure. Purification by column chromatography (20% EtOAc/hexane) afforded 125 mg (48%) of the title **4zp**.

Physical State: Bone white solid.

Melting Point: 67-69 ^{0}C.

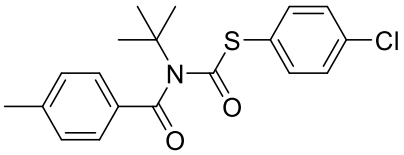
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 – 8.04 (m, 1H), 7.88 – 7.86 (m, 1H), 7.57 – 7.53 (m, 1H), 7.48 – 7.46 (m, 1H), 7.43 – 7.39 (m, 4H), 7.37 – 7.34 (m, 2H), 7.04 – 7.02 (m, 1H), 5.18 (s, 2H), 4.60 (s, 2H), 4.28 (q, *J* = 7.2 Hz, 2H), 4.12 (s, 2H), 1.31 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 190.8, 172.7, 170.1, 167.5, 160.5, 140.4, 136.7, 135.4, 132.7, 129.6, 129.4, 129.42 127.8, 127.1, 125.6, 125.0, 121.0, 73.5, 62.0, 46.2, 43.1, 14.1.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{27}H_{23}ClNO_6S$ 524.0929; Found 524.0921.

FT-IR (vcm^{-1}): 2976, 2852, 1751, 1624, 1468, 1263, 894.

TLC: R_f = 0.45 (20% EtOAc/hexane).



S-(4-chlorophenyl) tert-butyl(4-methylbenzoyl)carbamothioate

The synthesis of **4zq** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 121 mg (67%) of the title **4zq**.

Physical State: Bone white solid.

Melting Point: 60-62 ^{0}C.

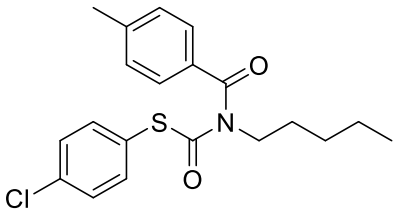
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 – 7.93 (m, 2H), 7.35 – 7.25 (m, 6H), 2.47 (s, 3H), 1.52 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.3, 164.2, 145.8, 136.5, 135.8, 132.9, 130.9, 129.8, 129.1, 126.9, 60.9, 28.5, 21.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{21}ClNO_2S$ 362.0976; Found 362.0975.

FT-IR (vcm^{-1}): 1771, 1723, 1565, 1216, 1015, 886.

TLC: R_f = 0.50 (10% EtOAc/hexane).



S-(4-chlorophenyl) (4-methylbenzoyl)(pentyl)carbamothioate

The synthesis of **4zr** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 112 mg (60%) of the title **4zr**.

Physical State: White solid.

Melting Point: 50-52 ^{0}C.

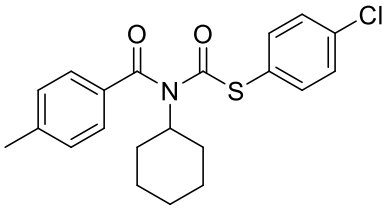
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 – 7.49 (m, 2H), 7.40 – 7.34 (m, 4H), 7.26 – 7.23 (m, 2H), 3.86 – 3.82 (m, 2H), 2.40 (s, 3H), 1.70 – 1.65 (m, 2H), 1.27 – 1.24 (m, 4H), 0.87 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.1, 170.3, 142.7, 136.5, 136.1, 132.2, 129.3, 129.2, 128.2, 126.8, 48.0, 28.9, 28.8, 22.2, 21.6, 13.9.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{23}ClNO_2S$ 376.1133; Found 376.1135.

FT-IR (vcm^{-1}): 1794, 1751, 1540, 1236, 1008, 865.

TLC: R_f = 0.40 (10% EtOAc/hexane).



S-(4-chlorophenyl) cyclohexyl(4-methylbenzoyl)carbamothioate

The synthesis of **4zs** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 126 mg (65%) of the title **4zs**.

Physical State: Brown solid.

Melting Point: 57-59 ^{0}C.

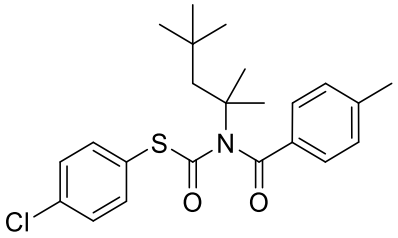
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 – 7.63 (m, 2H), 7.33 – 7.30 (m, 2H), 7.28 – 7.24 (m, 4H), 4.16 – 4.09 (m, 1H), 2.42 (s, 3H), 2.07 – 1.97 (m, 2H), 1.94 – 1.90 (m, 2H), 1.84 – 1.64 (m, 3H), 1.35 – 1.25 (m, 2H), 1.20 – 1.14 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.0, 169.5, 143.7, 136.3, 136.0, 133.1, 129.4, 129.3, 129.1, 126.5, 60.4, 30.6, 26.2, 25.0, 21.7.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{23}ClNO_2S$ 388.1133; Found 388.1127.

FT-IR (vcm^{-1}): 1785, 1763, 1531, 1242, 1034, 859.

TLC: R_f = 0.55 (10% EtOAc/hexane).



S-(4-chlorophenyl) (4-methylbenzoyl)(2,4,4-trimethylpentan-2-yl)carbamothioate

The synthesis of **4zt** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 127 mg (61%) of the title **4zt**.

Physical State: White solid.

Melting Point: 53-56 ^{0}C.

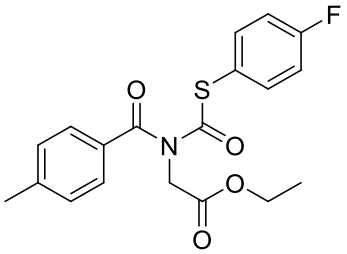
^{1}H NMR (400 MHz, CDCl$_3$): δ 7.91 – 7.89 (m, 2H), 7.34 – 7.28 (m, 4H), 7.22 – 7.19 (m, 2H), 2.46 (s, 3H), 2.10 (s, 2H), 1.53 (s, 6H), 1.07 (s, 9H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 172.7, 165.2, 145.2, 136.4, 135.8, 133.5, 130.5, 129.7, 129.1, 126.9, 65.0, 50.4, 31.7, 31.2, 29.1, 21.8.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}ClNO_2S$ 418.1602; Found 418.1597.

FT-IR (vcm^{-1}): 1787, 1739, 1532, 1228, 1009, 843.

TLC: R_f = 0.45 (10% EtOAc/hexane).



Ethyl N-(((4-fluorophenyl)thio)carbonyl)-N-(4-methylbenzoyl)glycinate

The synthesis of **4zu** was achieved according to the general procedure. Purification by column chromatography (10% EtOAc/hexane) afforded 153 mg (85%) of the title **4zu**.

Physical State: Brown solid.

Melting Point: 65-67 ^{0}C.

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.60 – 7.58 (m, 2H), 7.45 – 7.41 (m, 2H), 7.27 (s, 1H), 7.25

(s, 1H), 7.09 – 7.05 (m, 2H), 4.52 (s, 2H), 4.24 (q, *J* = 7.2 Hz, 2H), 2.41 (s, 3H), 1.28 (t, *J* = 7.2 Hz, 3H)

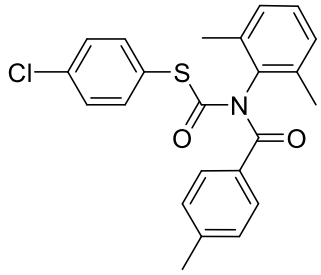
^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 170.9, 168.0, 164.9, 162.4, 143.2, 137.4, 137.3, 131.3, 129.35, 128.4, 116.5, 116.3, 61.7, 48.7, 21.6, 14.7.

^{19}F NMR (376 MHz, $CDCl_3$): 115.28

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{19}FNO_4S^+$ 376.1013; Found 376.1005.

FT-IR (vcm^{-1}): 2943, 2854, 1768, 1680, 1471, 1204, 892.

TLC: R_f = 0.55 (10% EtOAc/hexane).



S-(4-chlorophenyl) (2,6-dimethylphenyl)(4-methylbenzoyl)carbamothioate

The synthesis of **4zv** was achieved according to the general procedure. Purification by column chromatography (15% EtOAc/hexane) afforded 96 mg (47%) of the title **4zv**.

Physical State: Light brown solid.

Melting Point: 60-62 ^{0}C.

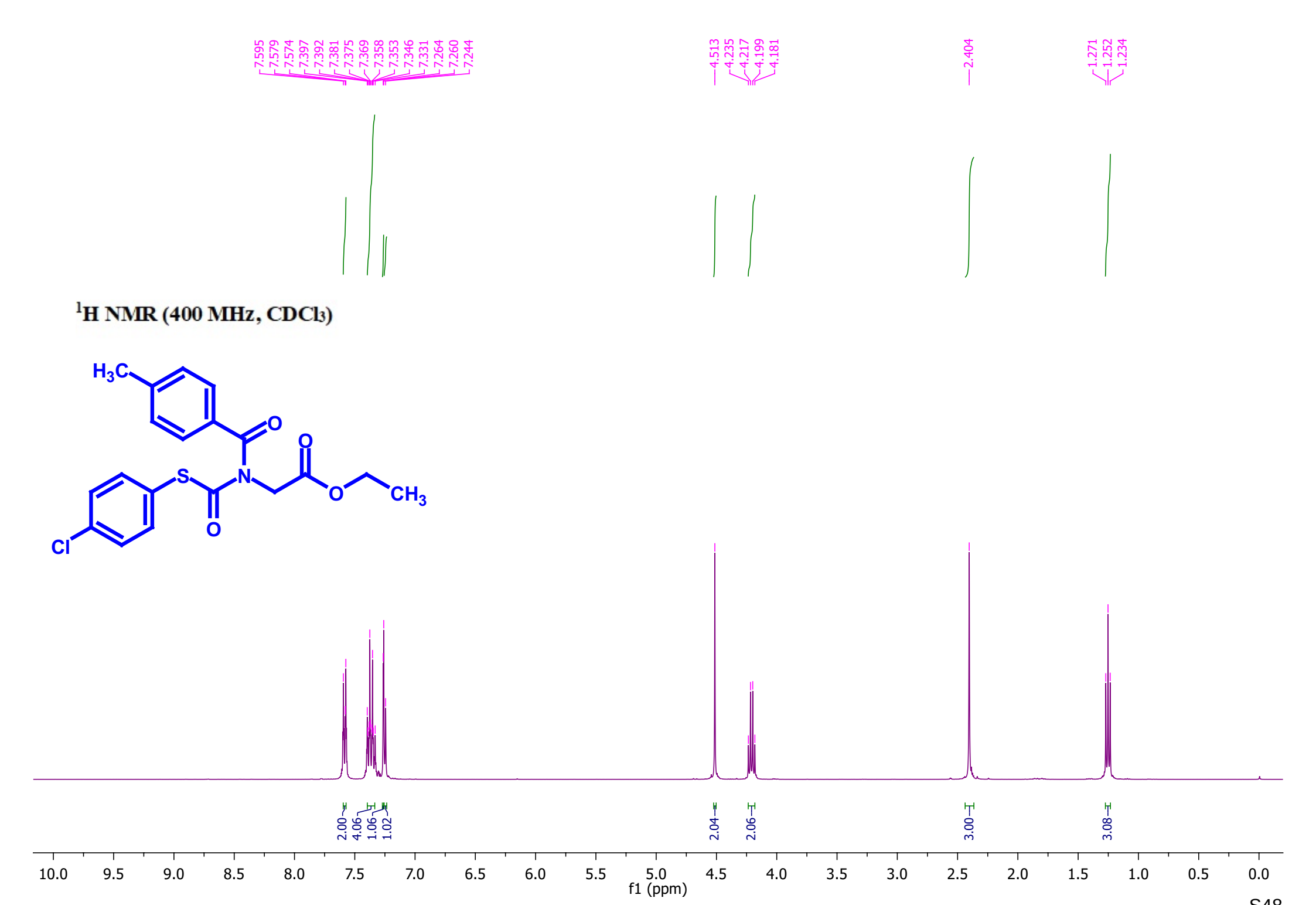
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 – 7.61 (m, 2H), 7.36 – 7.30 (m, 5H), 7.23 – 7.20 (m, 4H), 2.41 (s, 6H), 2.38 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.2, 169.9, 142.7, 137.8, 136.1, 136.0, 135.1, 131.9, 130.0, 129.3, 129.0, 128.9, 128.5, 126.4, 21.6, 18.4.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{21}ClNO_2S$ 410.0976; Found 410.0973.

FT-IR (vcm^{-1}): 1765, 1732, 1564, 1208, 1053, 862.

TLC: R_f = 0.50 (15% EtOAc/hexane).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O
CH3
Cl
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4.06
1.06
1.02
2.04
2.06
3.00
3.08
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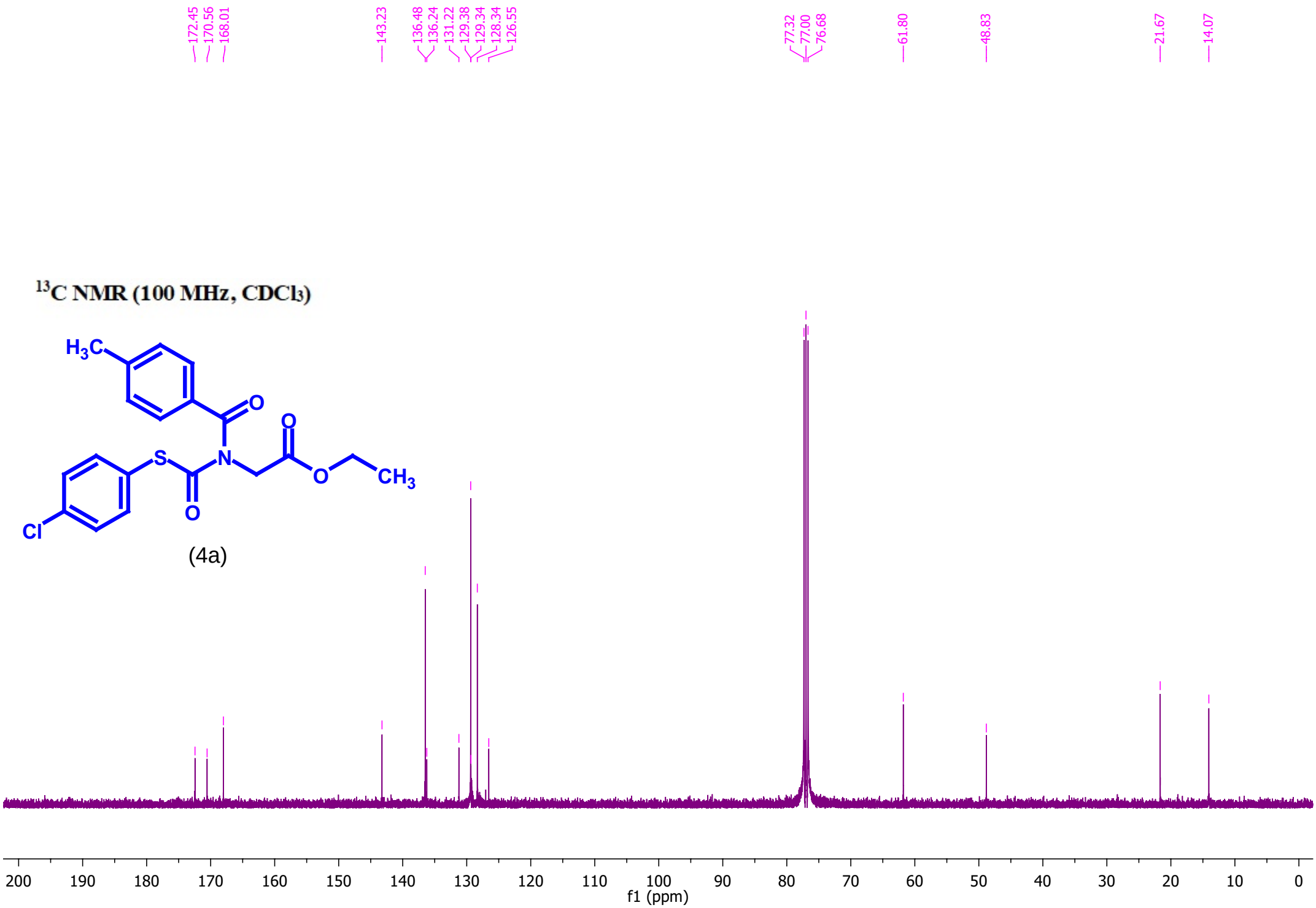
f1 (ppm)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131.22
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129.34
128.34
126.55
77.32
77.00
76.68
61.80
48.83
21.67
14.07
^{13}C NMR (100 MHz, CDCl$_3$)
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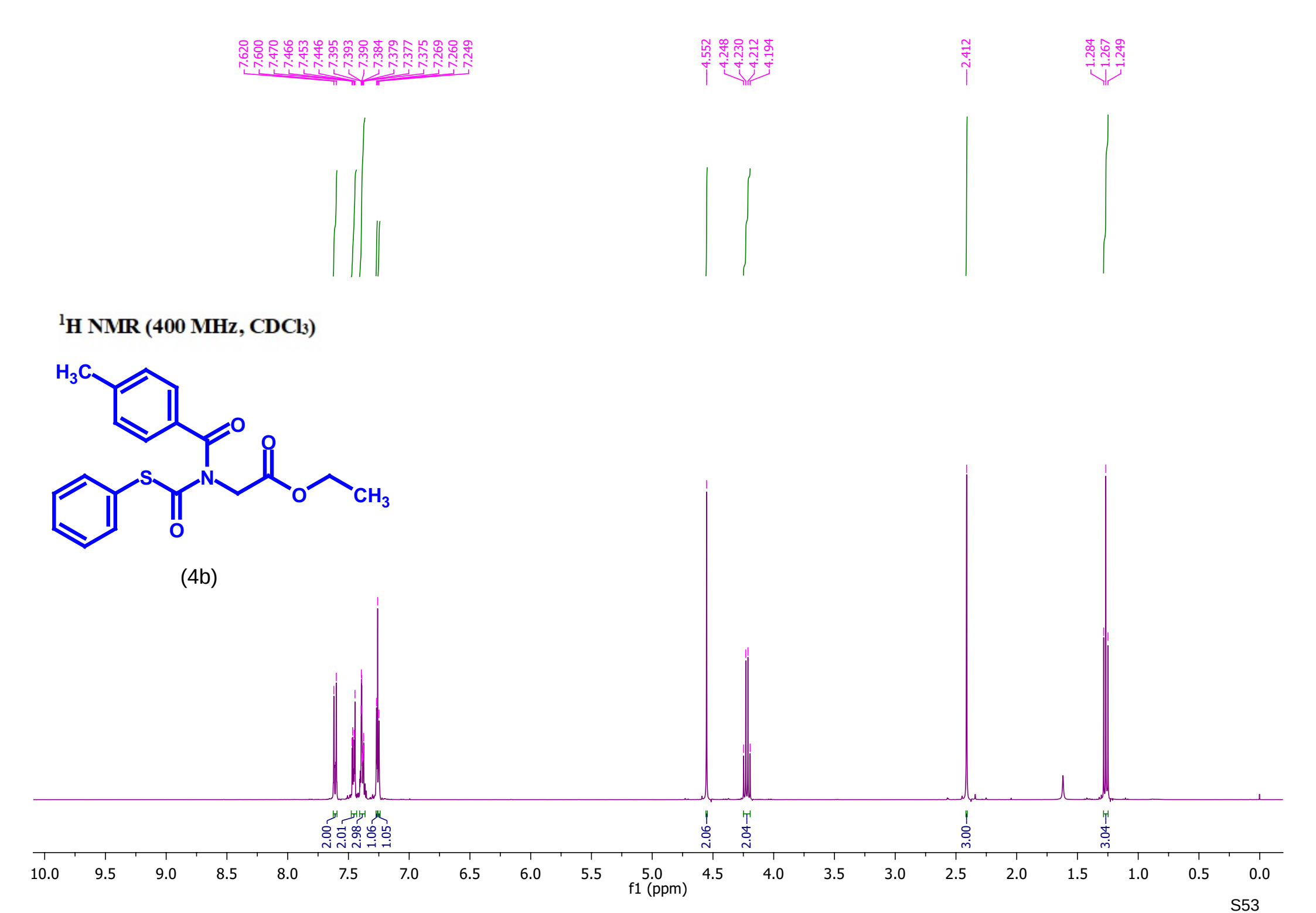
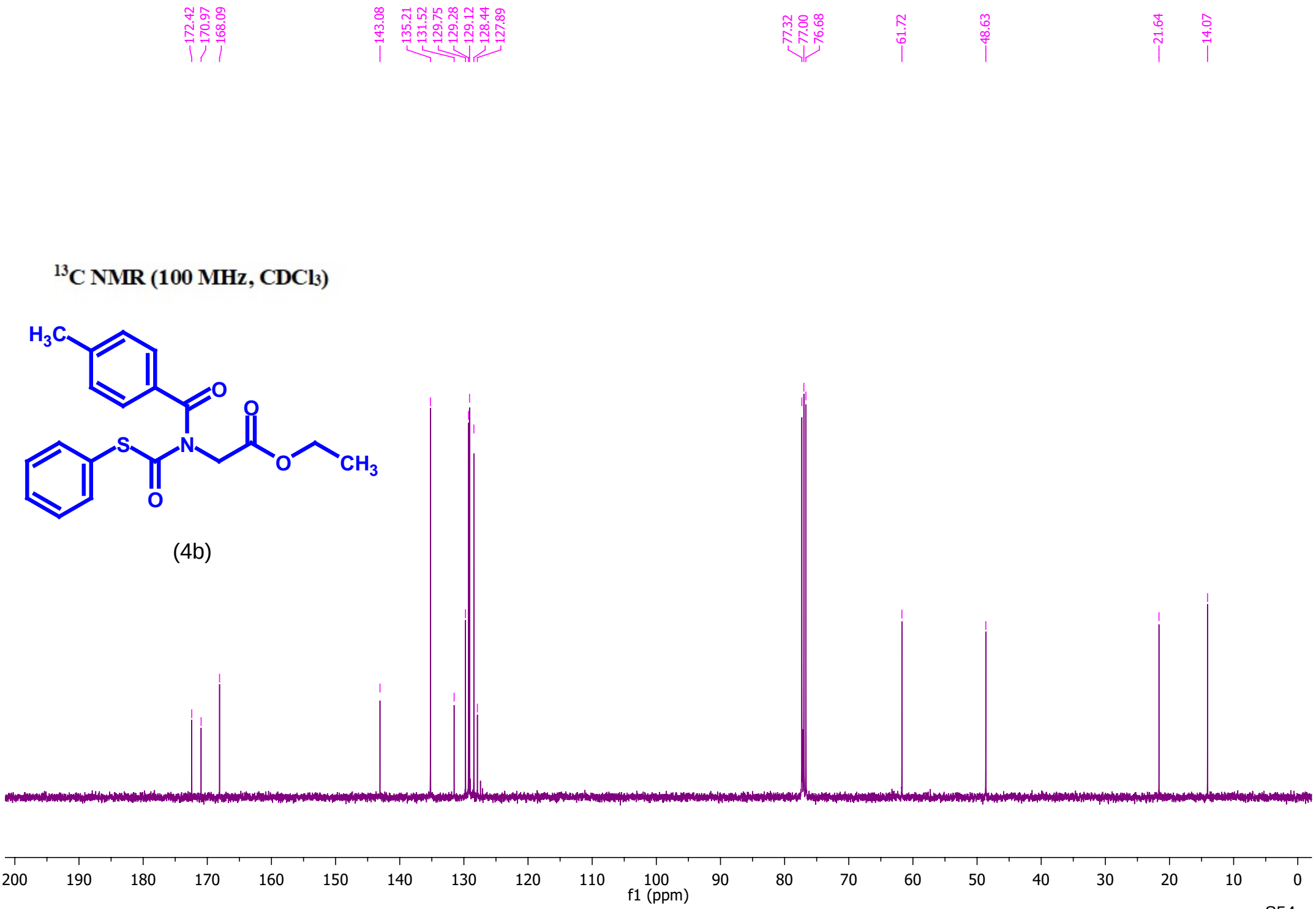
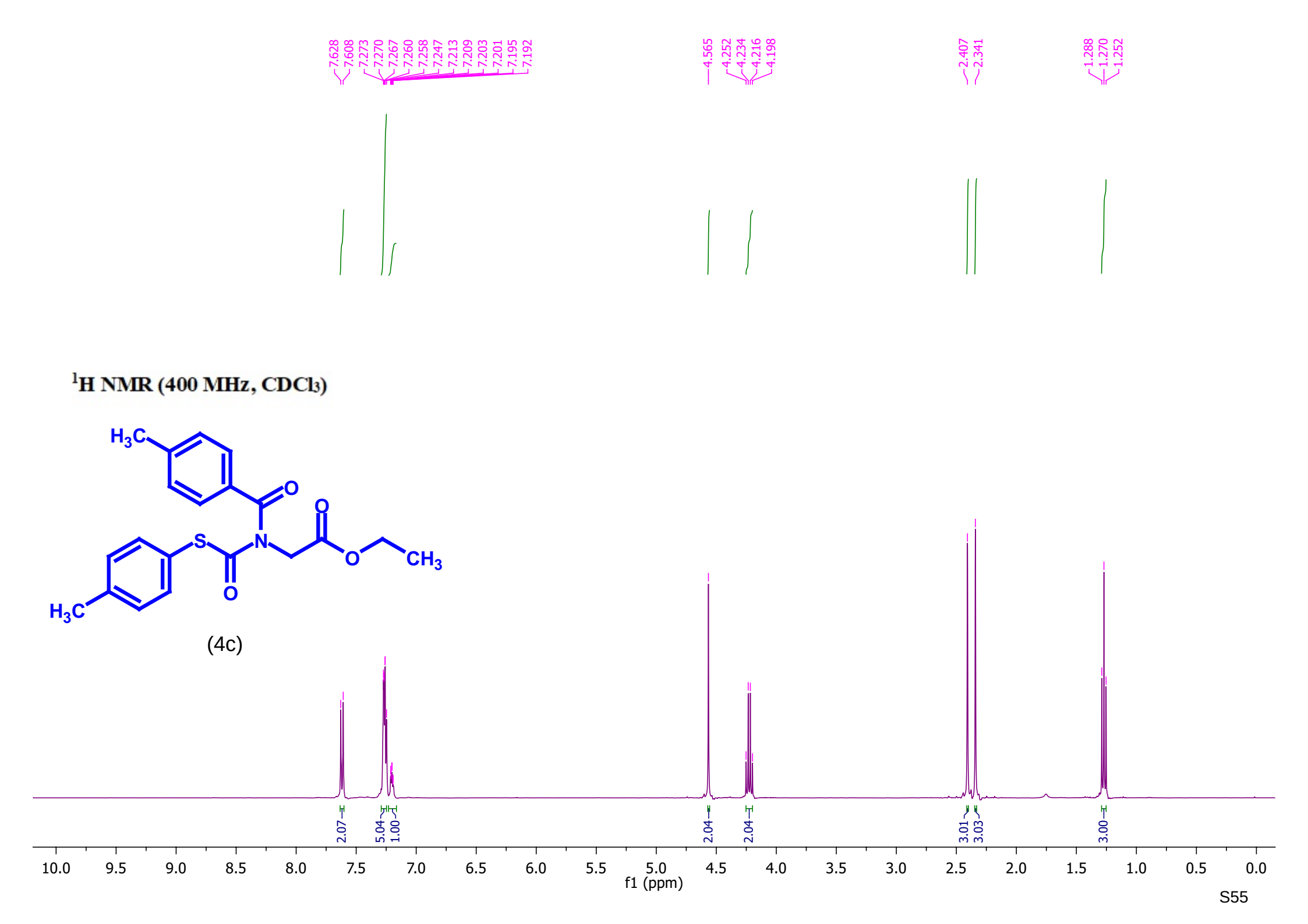
O
O
S
N
O
O
CH$_3$
O
Cl
(4a)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

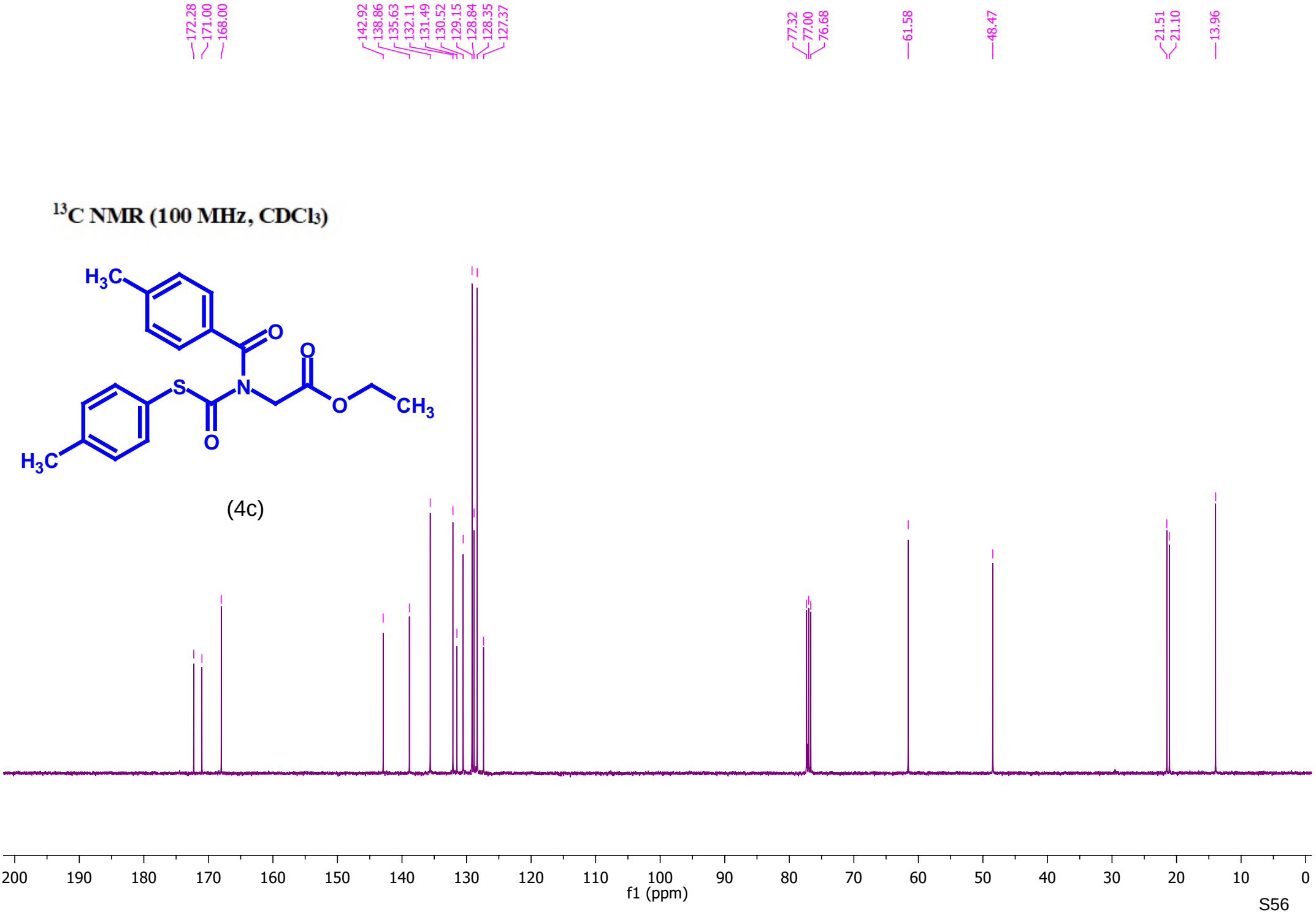

7.620
7.600
7.470
7.466
7.453
7.446
7.395
7.393
7.390
7.384
7.379
7.377
7.375
7.269
7.260
7.249
4.552
4.248
4.230
4.212
4.194
2.412
1.284
1.267
1.249
^{1}H NMR (400 MHz, CDCl$_3$)
H$_3$C
O
O
S
N
O
CH$_3$
O
(4b)
2.00
2.01
2.98
1.06
1.05
2.06
2.04
3.00
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

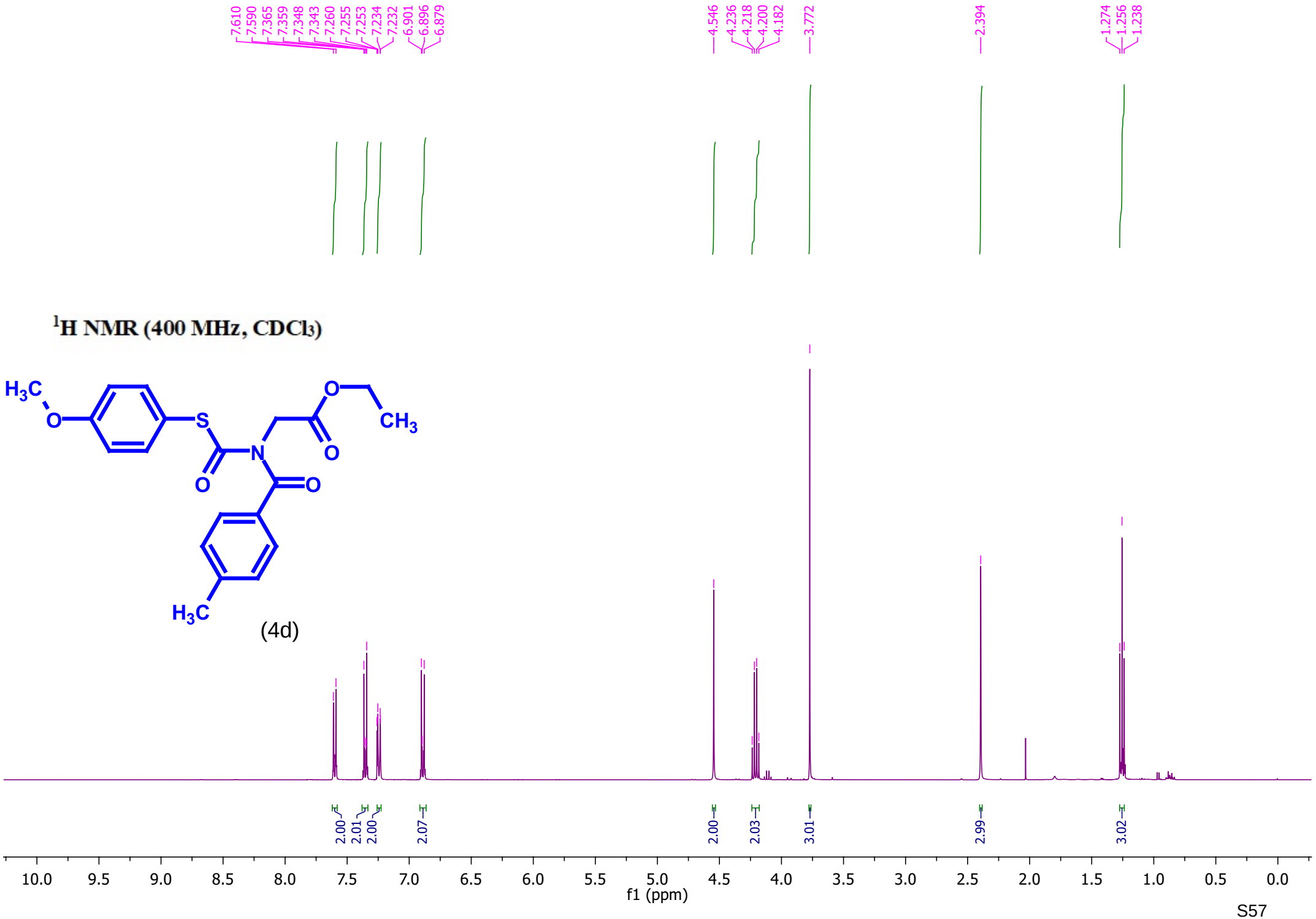

172.42
170.97
168.09
143.08
135.21
131.52
129.75
129.28
129.12
128.44
127.89
77.32
77.00
76.68
61.72
48.63
21.64
14.07
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
O
O
S
N
O
O
CH$_3$
(4b)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

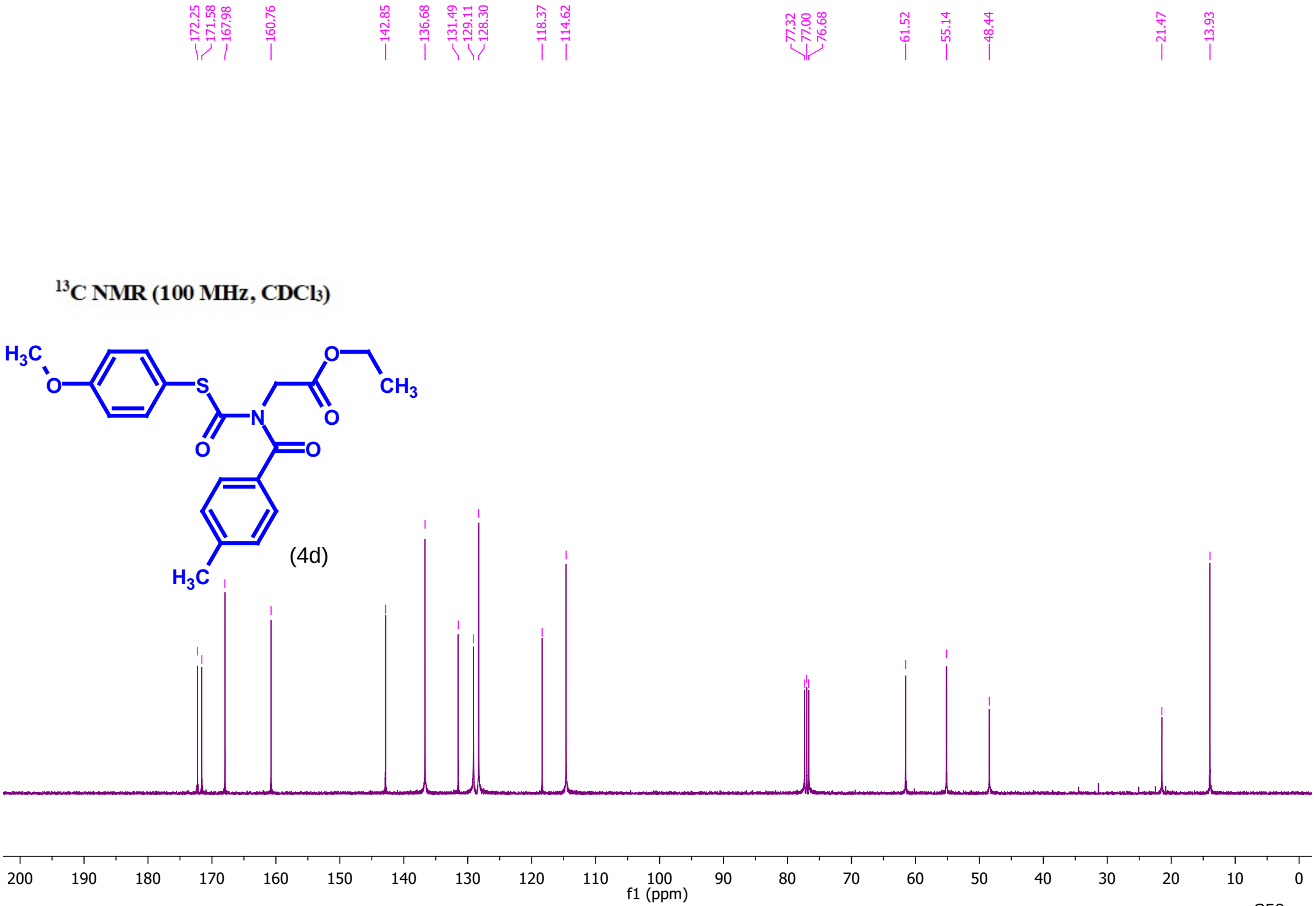


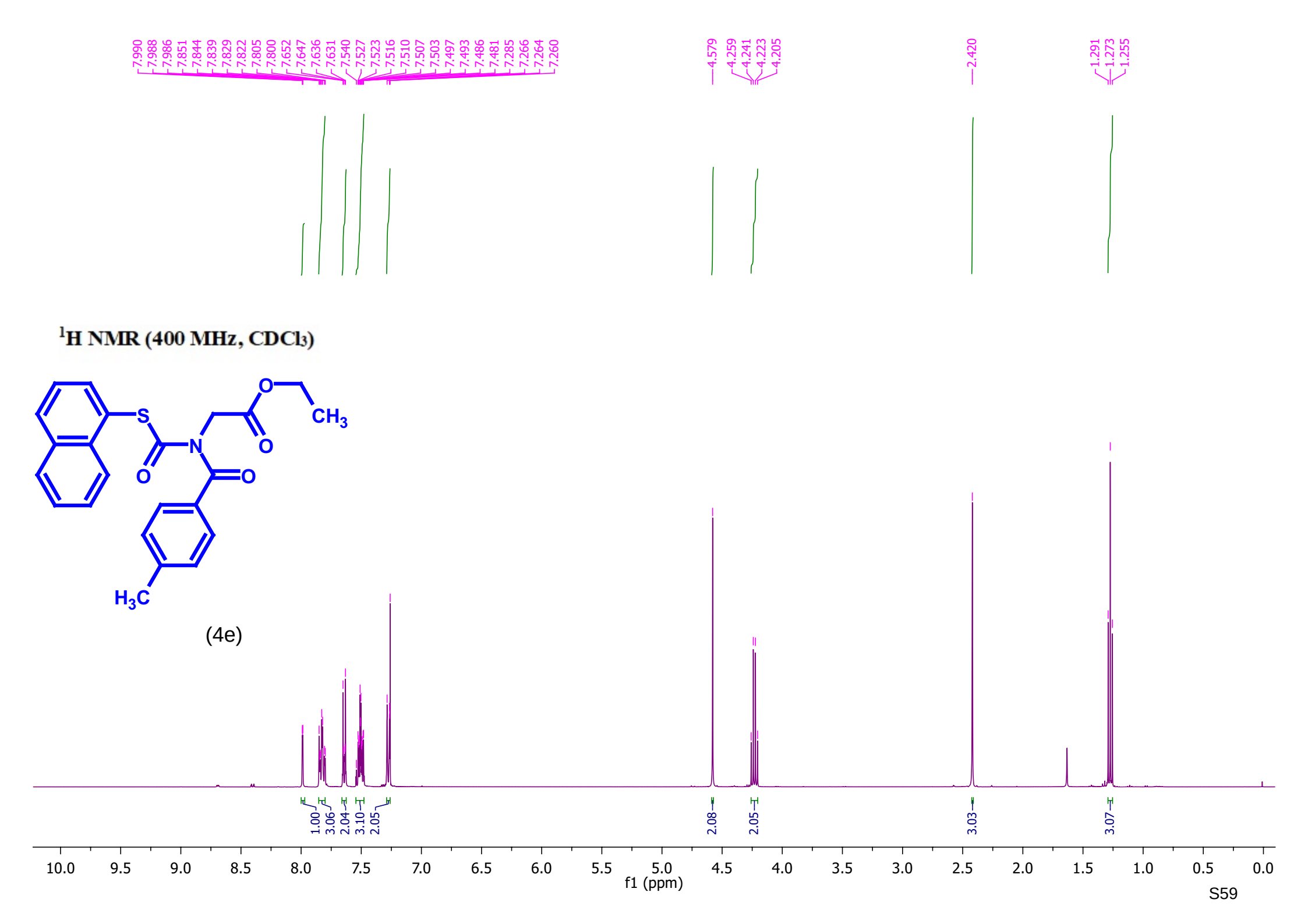
^{1}H NMR (400 MHz, $CDCl_3$)

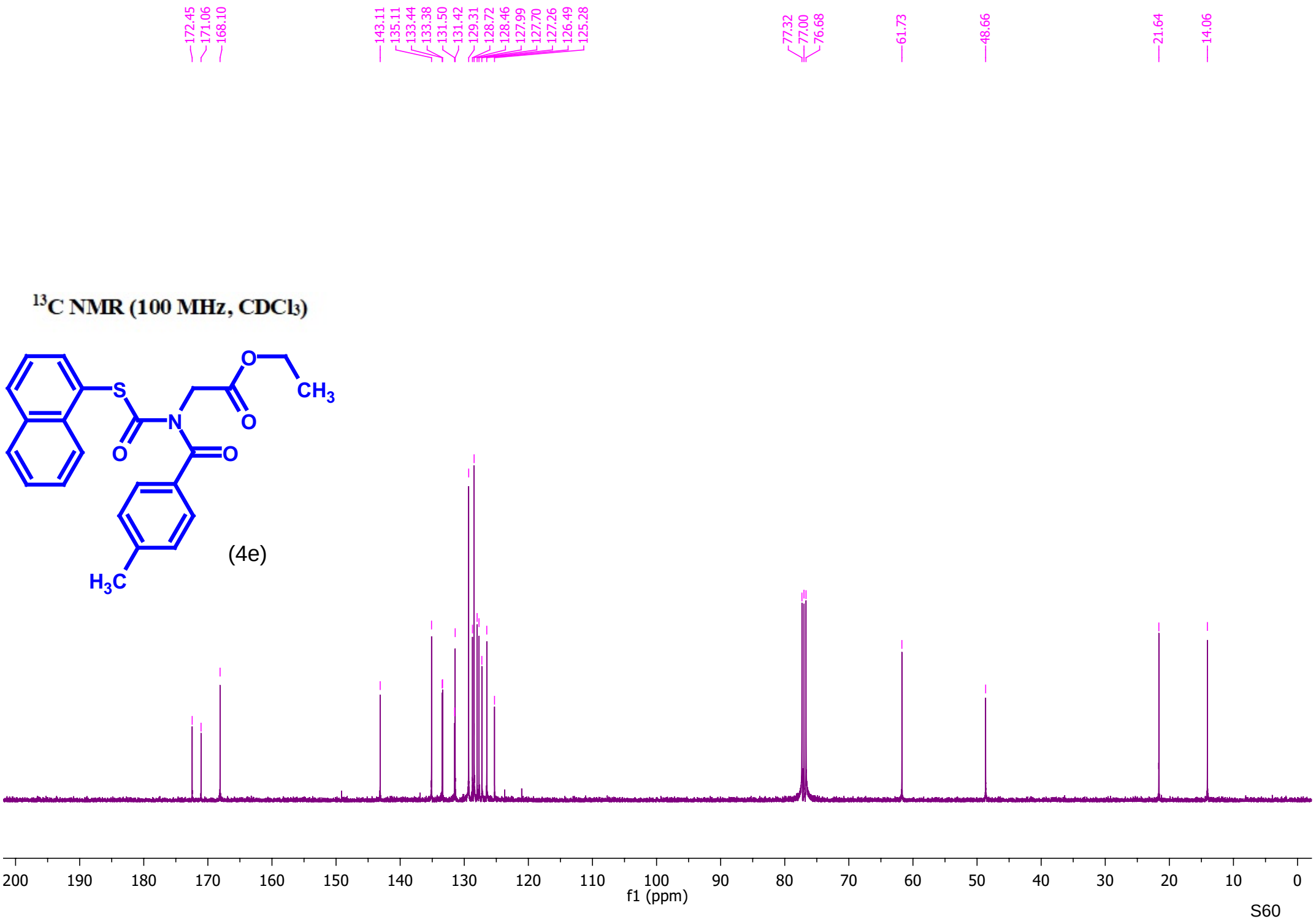
(4c)


172.28
171.00
168.00
142.92
138.86
135.63
132.11
131.49
130.52
129.15
128.84
128.35
127.37
77.32
77.00
76.68
61.58
48.47
21.51
21.10
13.96
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
O
O
S
N
O
CH$_3$
O
H$_3$C
(4c)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

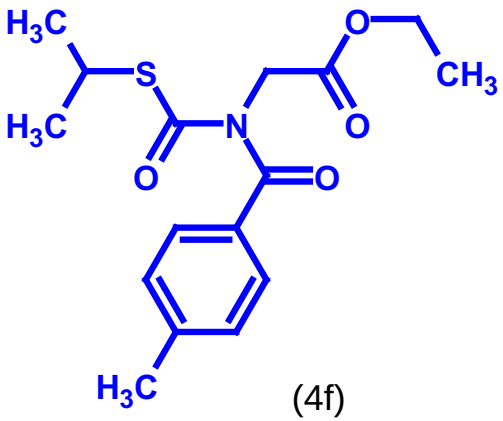

7.610
7.590
7.365
7.359
7.348
7.343
7.260
7.255
7.253
7.234
7.232
6.901
6.896
6.879
4.546
4.236
4.218
4.200
4.182
3.772
2.394
1.274
1.256
1.238
^{1}H NMR (400 MHz, CDCl$_3$)
H$_3$C
O
S
N
O
O
O
O
CH$_3$
O
H$_3$C
(4d)
2.00
2.01
2.00
2.07
2.00
2.03
3.01
2.99
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


172.25
171.58
167.98
160.76
142.85
136.68
131.49
129.11
128.30
118.37
114.62
77.32
77.00
76.68
61.52
55.14
48.44
21.47
13.93
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
O
S
N
O
O
O
O
CH$_3$
O
H$_3$C
(4d)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.990
7.988
7.986
7.851
7.844
7.839
7.829
7.822
7.805
7.800
7.652
7.647
7.636
7.631
7.540
7.527
7.523
7.516
7.510
7.507
7.503
7.497
7.493
7.486
7.481
7.285
7.266
7.264
7.260
4.579
4.259
4.241
4.223
4.205
2.420
1.291
1.273
1.255
1H NMR (400 MHz, CDCl3)
S
N
O
O
O
O
CH3
H3C
(4e)
1.00
3.06
2.04
3.10
2.05
2.08
2.05
3.03
3.07
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
f1 (ppm)

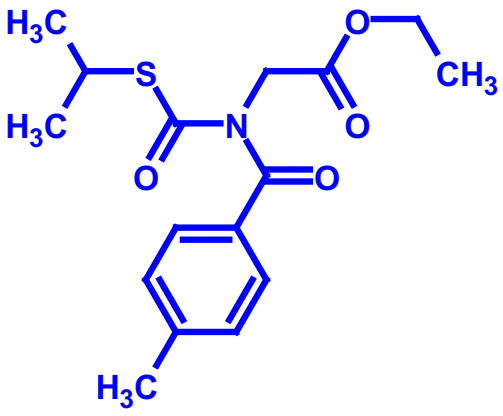

172.45
171.06
168.10
143.11
135.11
133.44
133.38
131.50
131.42
129.31
128.72
128.46
127.99
127.70
127.26
126.49
125.28
77.32
77.00
76.68
61.73
48.66
21.64
14.06
^{13}C NMR (100 MHz, CDCl$_3$)
S
N
O
O
O
O
CH$_3$
H$_3$C
(4e)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{1}H NMR (400 MHz, $CDCl_3$)



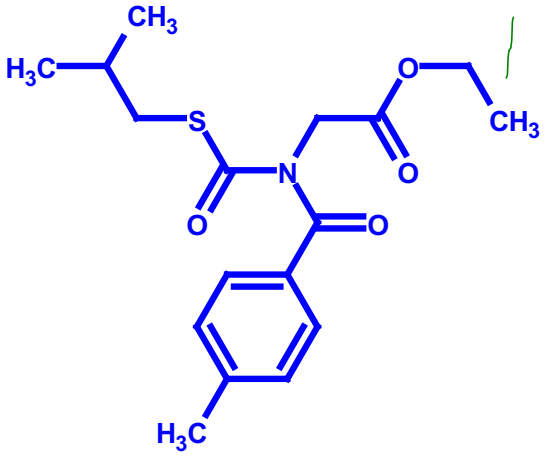
(4f)

^{13}C NMR (100 MHz, $CDCl_3$)

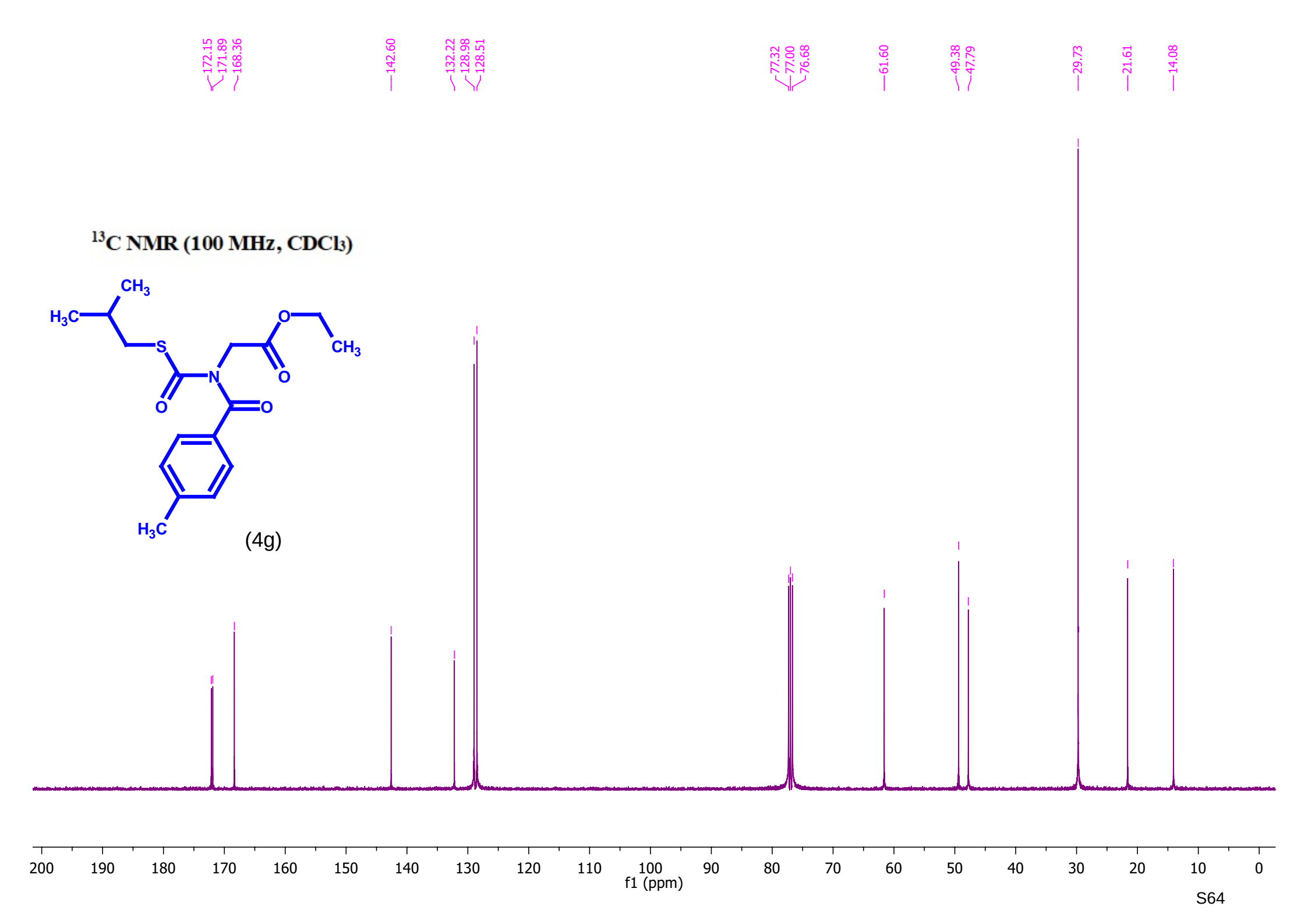


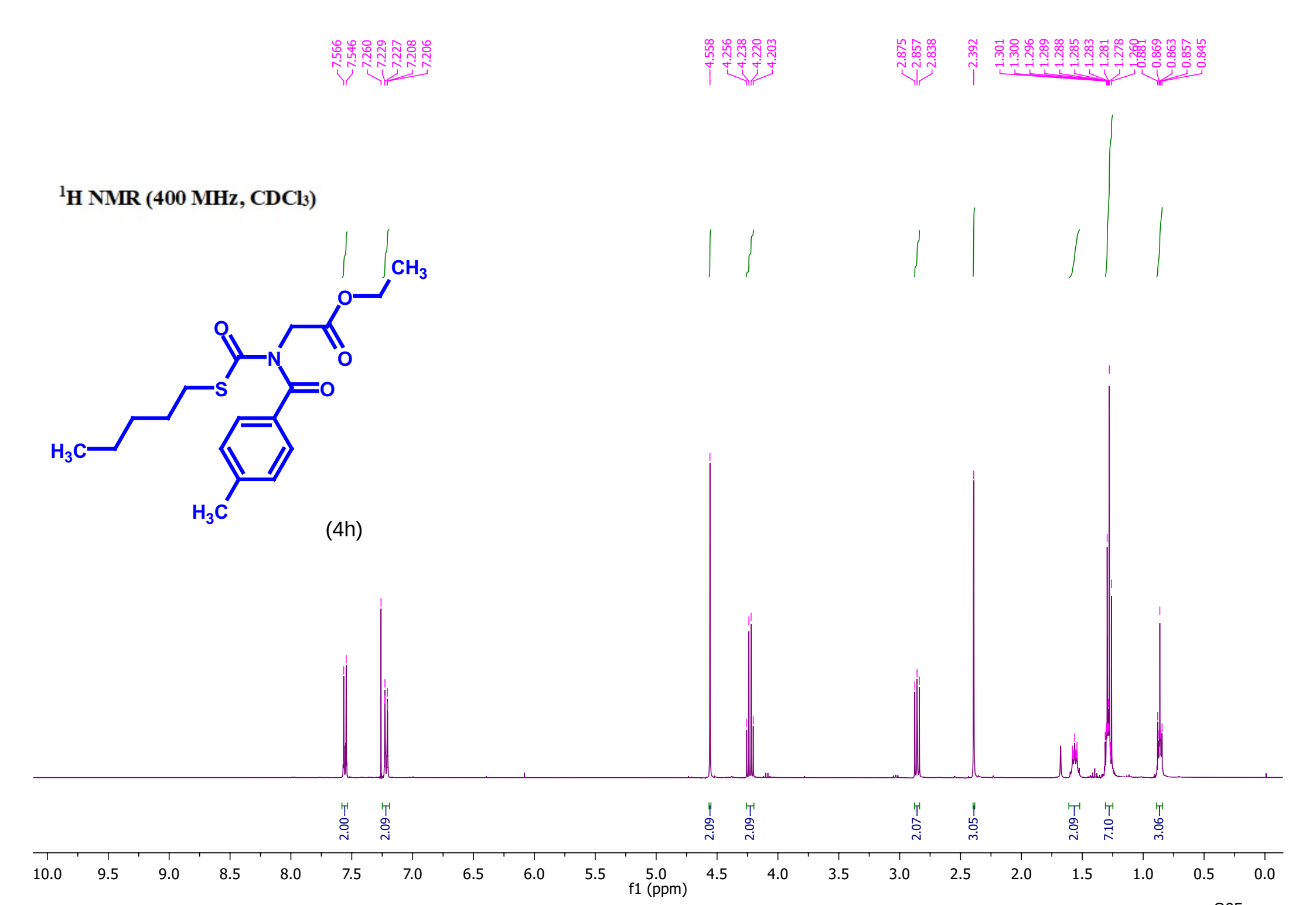
(4f)

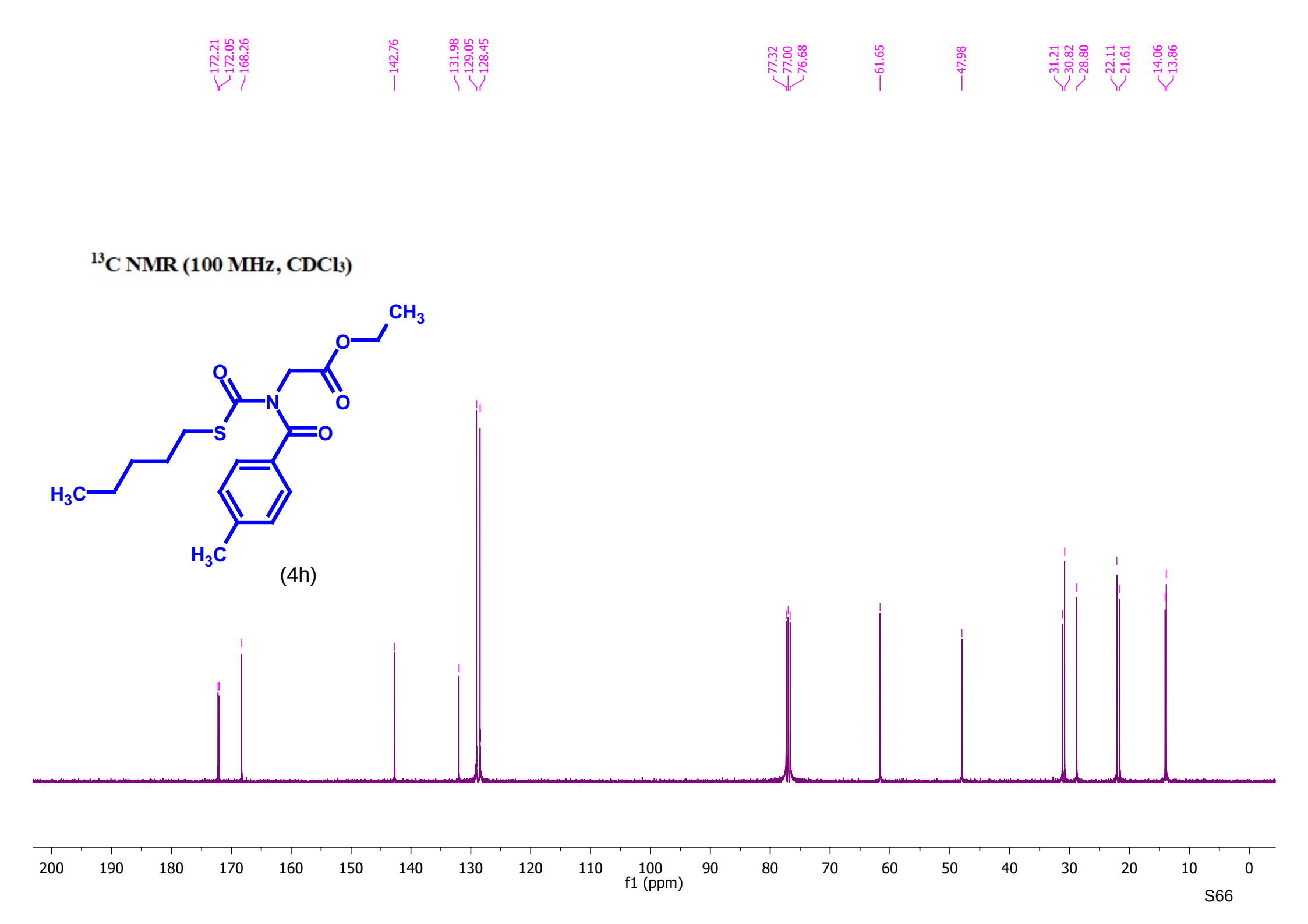
^{1}H NMR (400 MHz, $CDCl_3$)

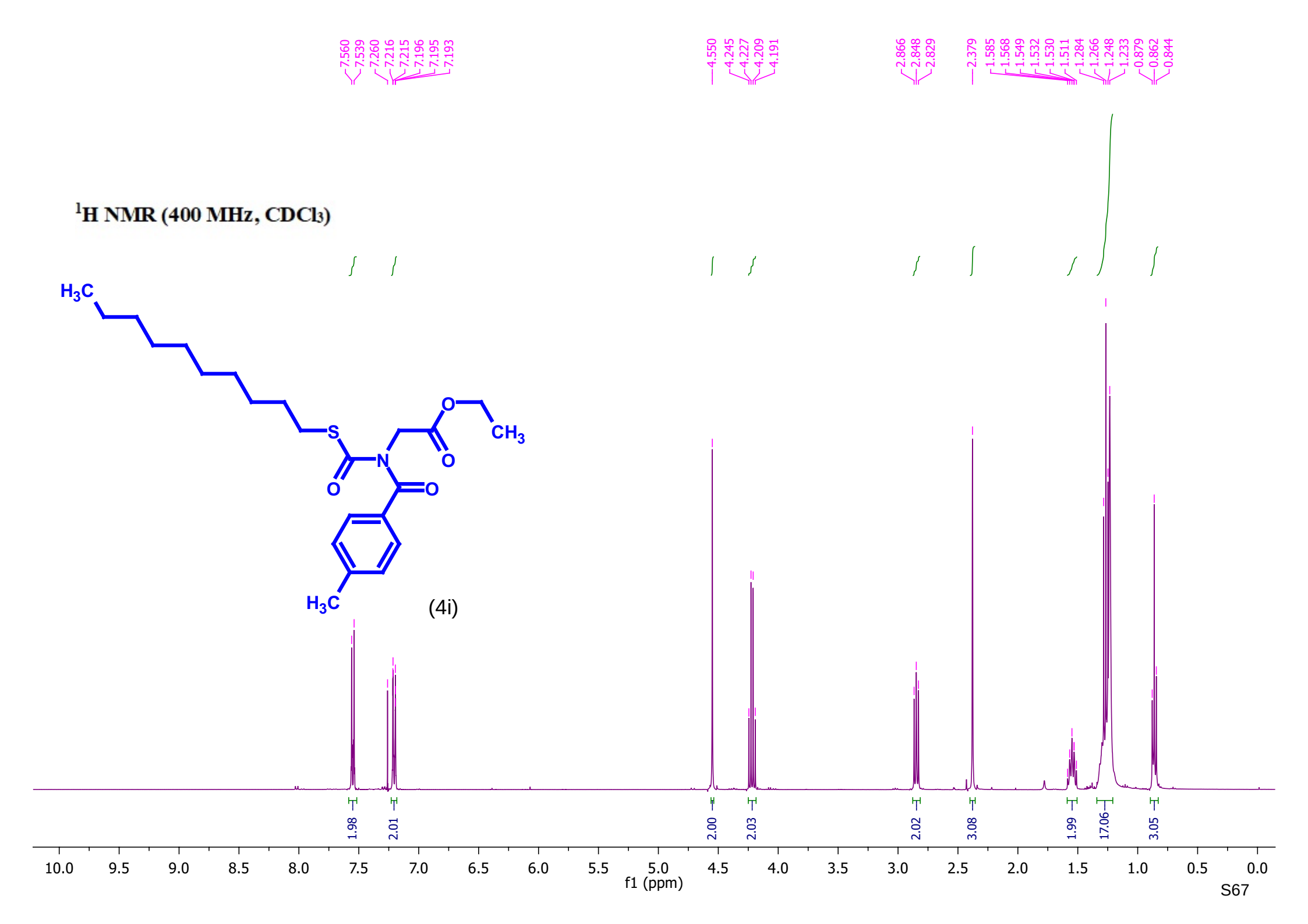


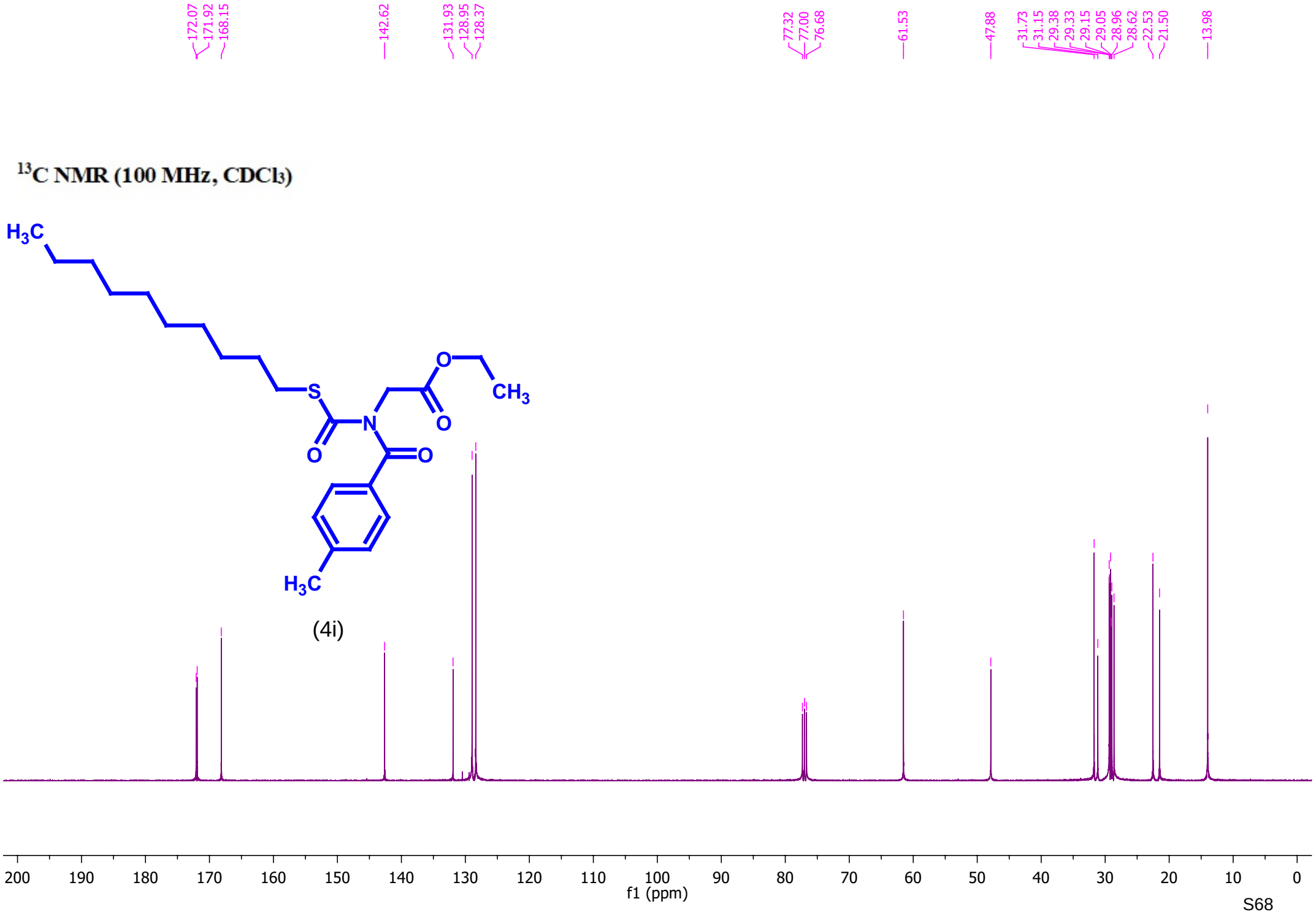
(4g)

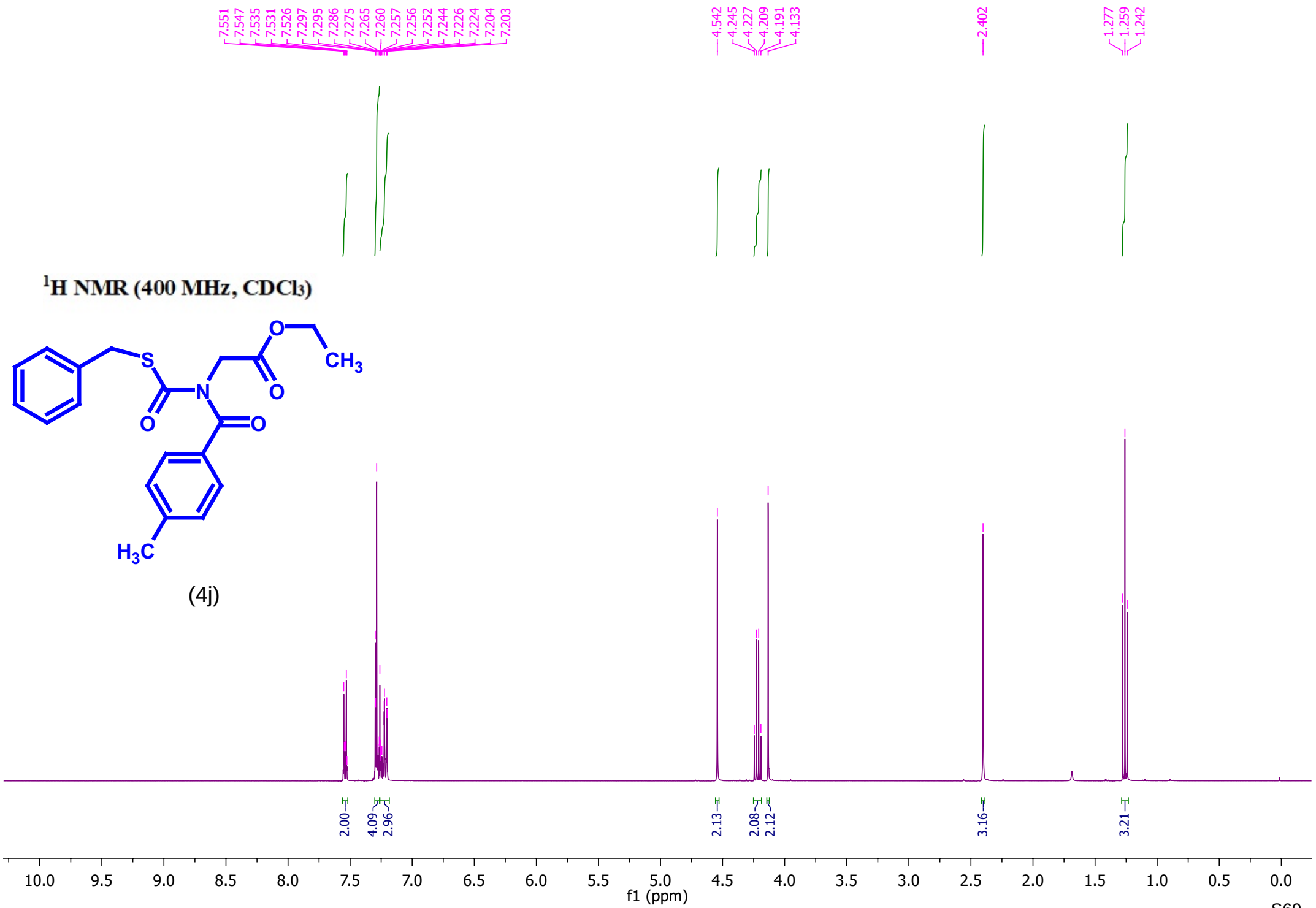

172.15
171.89
168.36
142.60
132.22
128.98
128.51
77.32
77.00
76.68
61.60
49.38
47.79
29.73
21.61
14.08
^{13}C NMR (100 MHz, CDCl$_3$)
(4g)
f1 (ppm)


^{1}H NMR (400 MHz, CDCl$_3$)
(4h)
7.566
7.546
7.260
7.229
7.227
7.208
7.206
4.558
4.256
4.238
4.220
4.203
2.875
2.857
2.838
2.392
1.301
1.300
1.296
1.289
1.288
1.285
1.283
1.281
1.278
0.869
0.863
0.857
0.845
2.00
2.09
2.09
2.09
2.07
3.05
2.09
7.10
3.06
f1 (ppm)


172.21
172.05
168.26
142.76
131.98
129.05
128.45
77.32
77.00
76.68
61.65
47.98
31.21
30.82
28.80
22.11
21.61
14.06
13.86
^{13}C NMR (100 MHz, CDCl$_3$)
CH$_3$
O
O
O
N
O
S
H$_3$C
H$_3$C
(4h)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

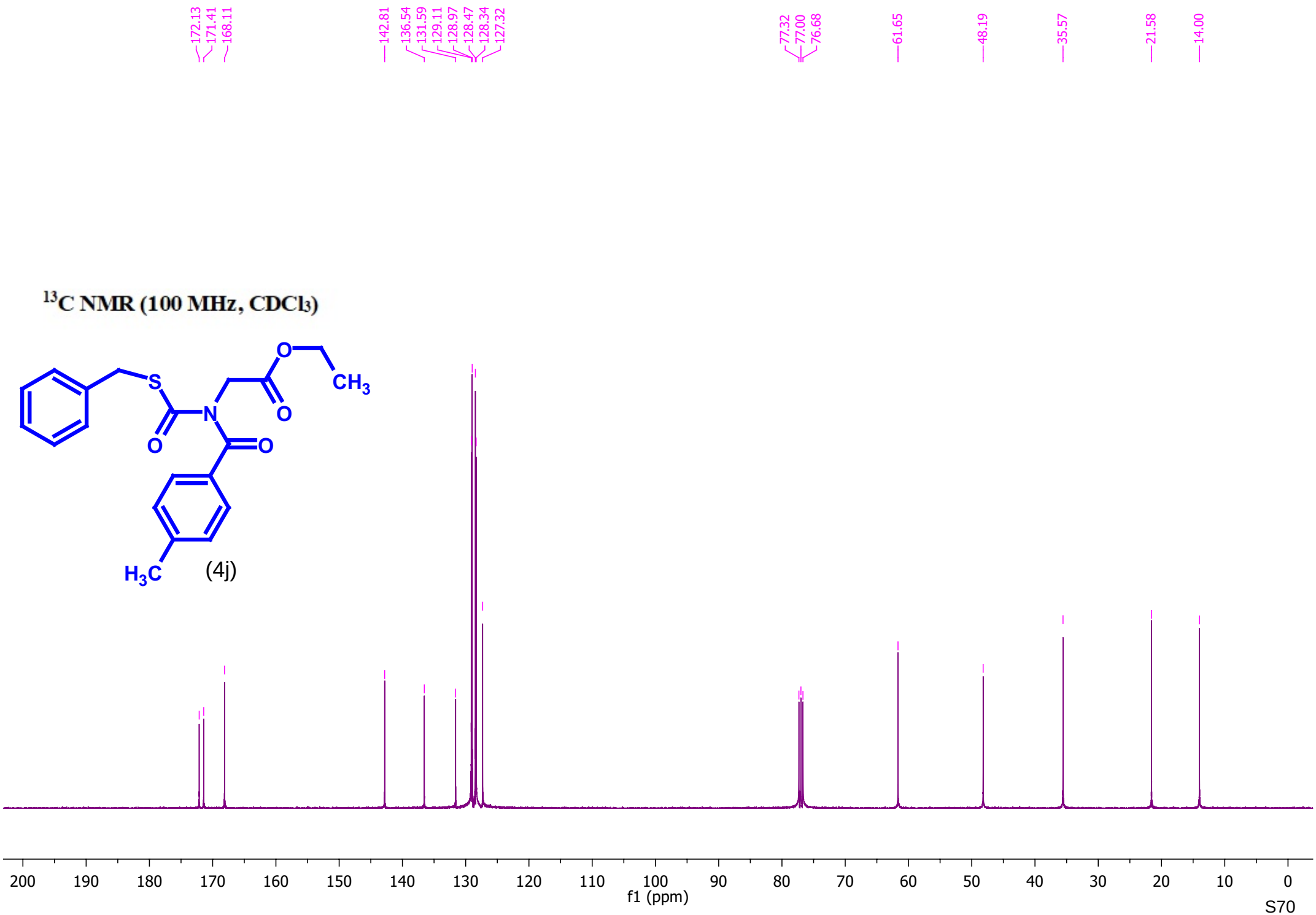

7.560
7.539
7.260
7.216
7.215
7.196
7.195
7.193
4.550
4.245
4.227
4.209
4.191
2.866
2.848
2.829
2.379
1.585
1.568
1.549
1.532
1.530
1.511
1.284
1.266
1.248
1.233
0.879
0.862
0.844
1H NMR (400 MHz, CDCl3)
H3C
S
N
O
O
O
O
CH3
O
H3C
(4i)
1.98
2.01
2.00
2.03
2.02
3.08
1.99
17.06
3.05
10.0
9.5
9.0
8.5
8.0
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)

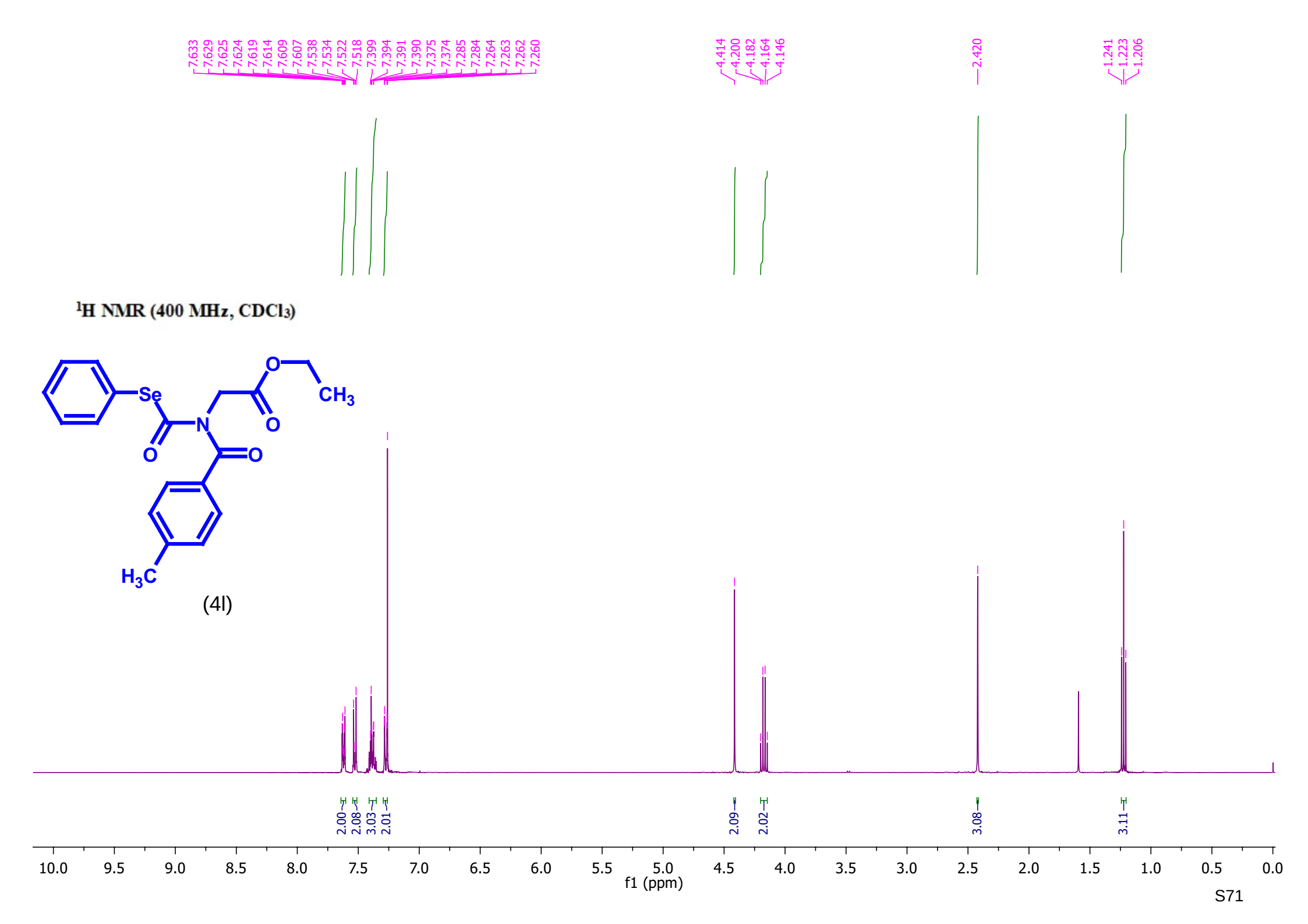

172.07
171.92
168.15
142.62
131.93
128.95
128.37
77.32
77.00
76.68
61.53
47.88
31.73
31.15
29.38
29.33
29.15
29.05
28.96
28.62
22.53
21.50
13.98
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
S
N
O
O
O
O
CH$_3$
H$_3$C
(4i)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{1}H NMR (400 MHz, $CDCl_3$)

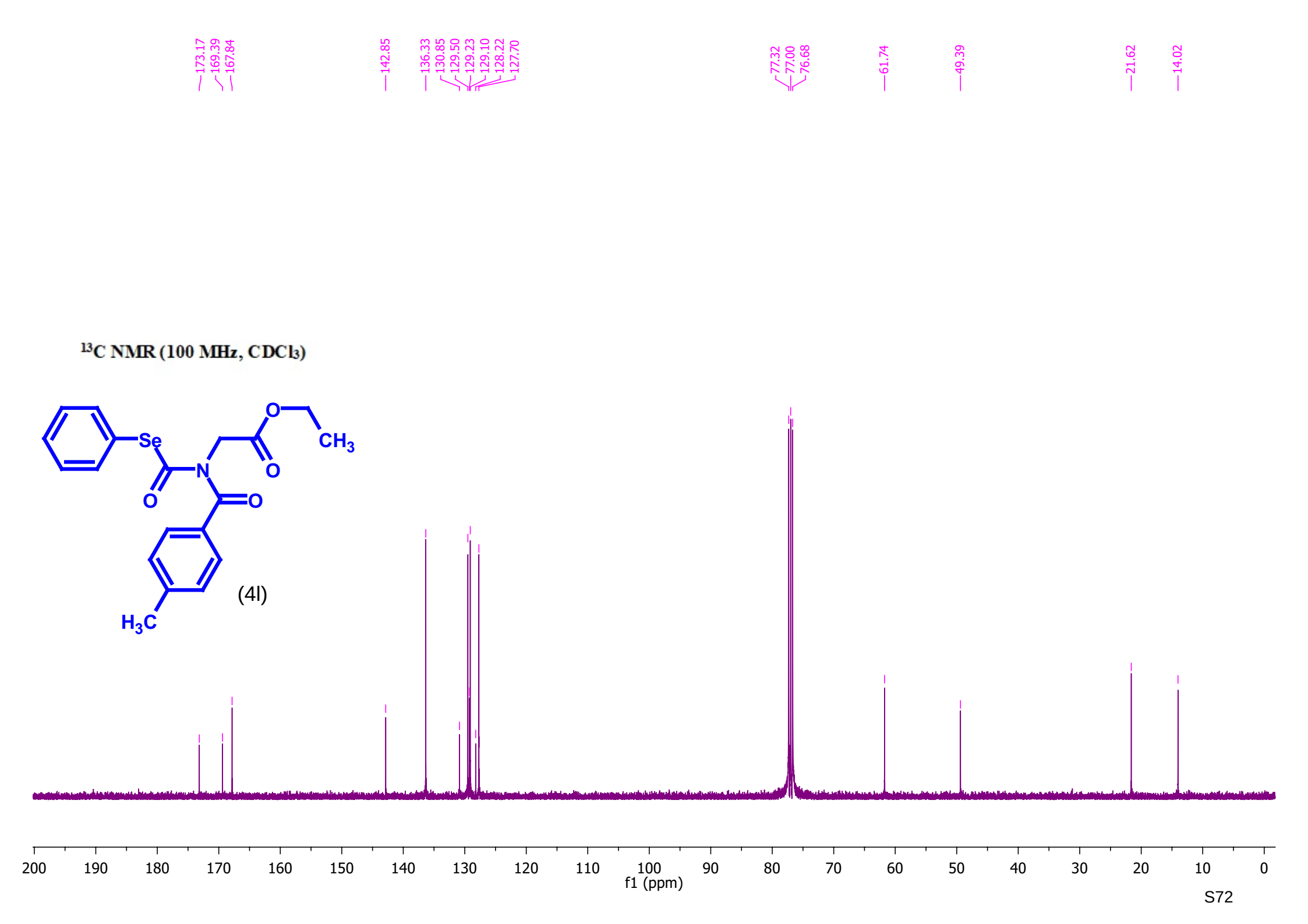
(4j)

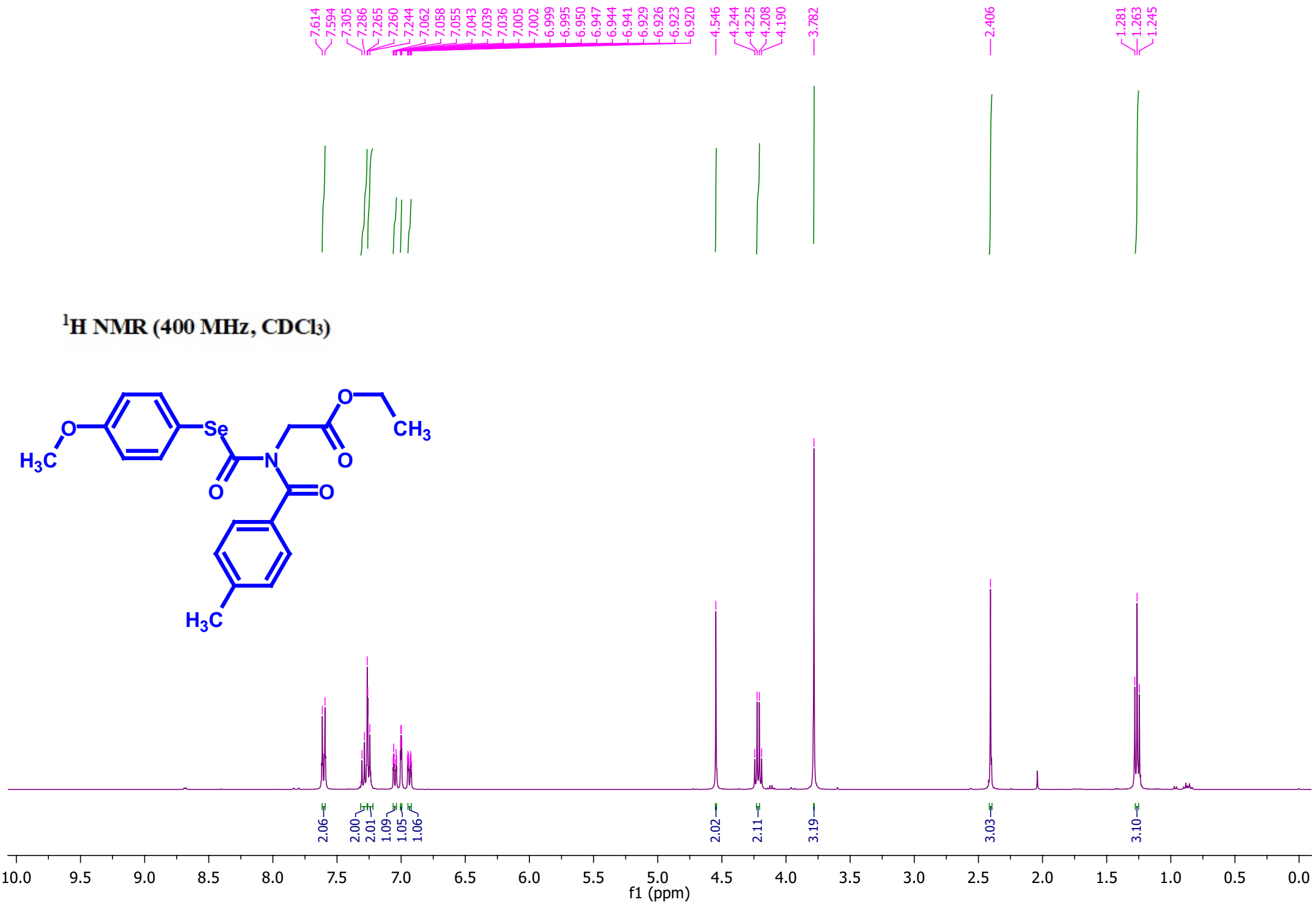

172.13
171.41
168.11
142.81
136.54
131.59
129.11
128.97
128.47
128.34
127.32
77.32
77.00
76.68
61.65
48.19
35.57
21.58
14.00
^{13}C NMR (100 MHz, CDCl$_3$)
S
N
O
O
O
O
CH$_3$
H$_3$C
(4j)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

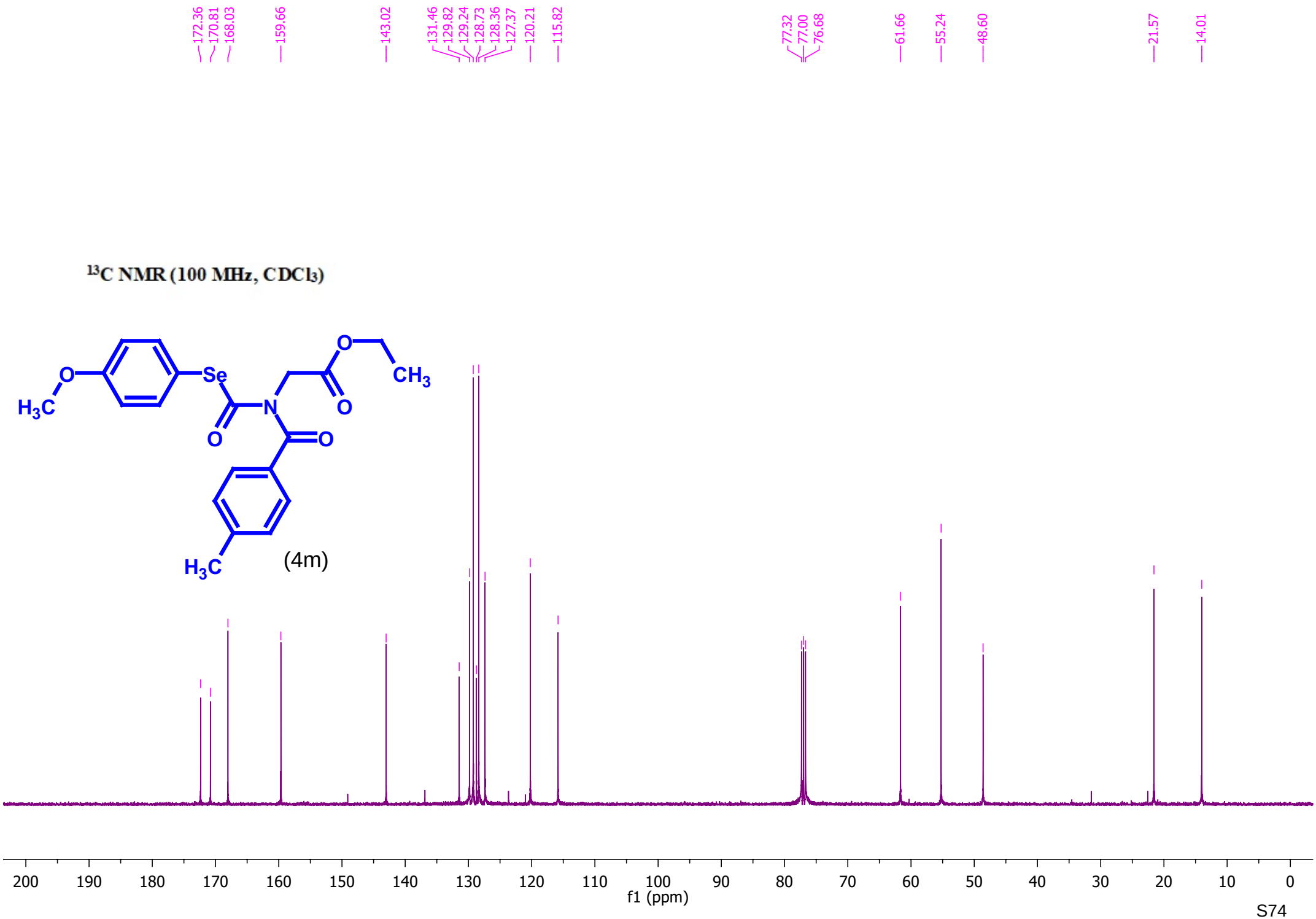


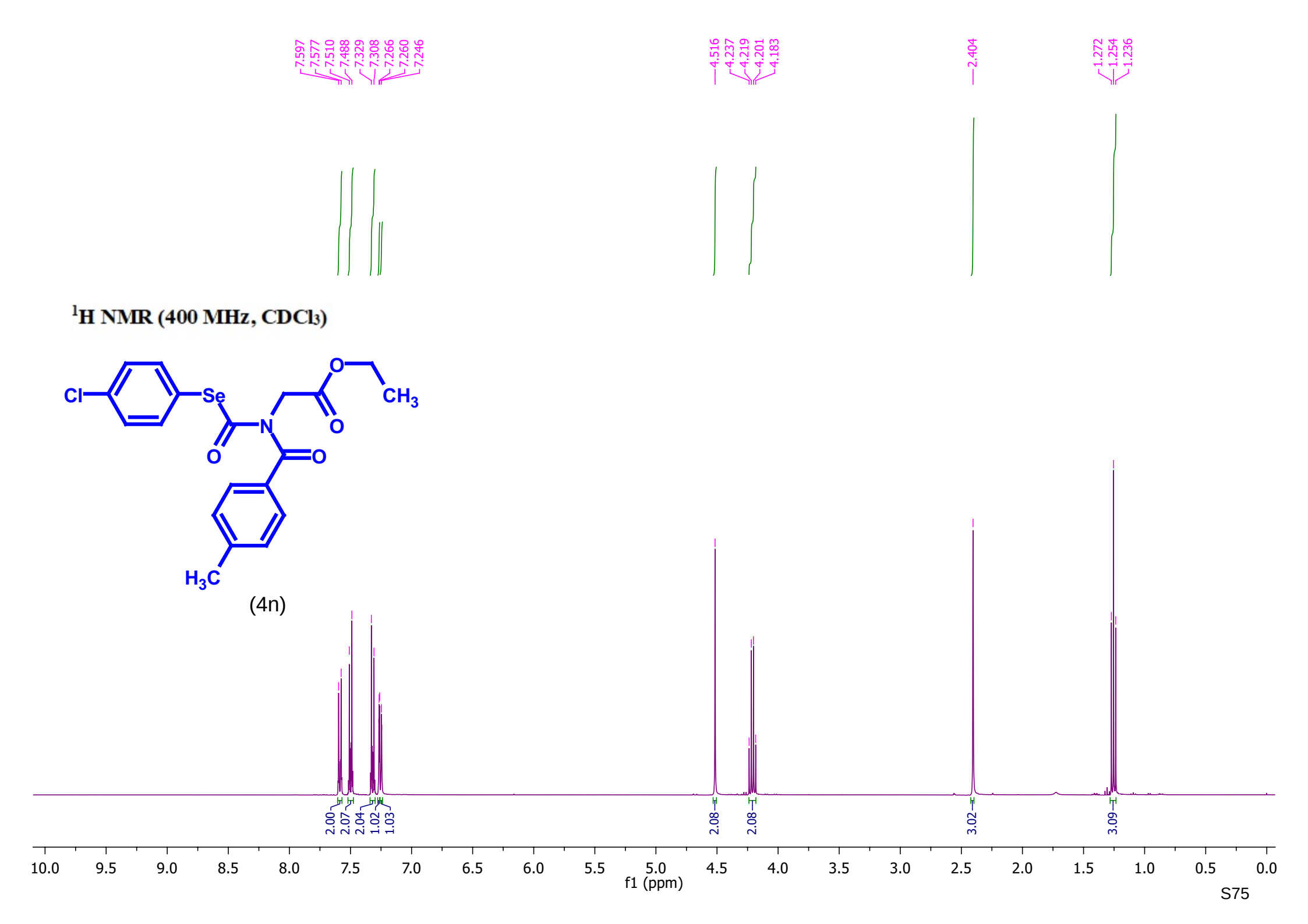
^{1}H NMR (400 MHz, $CDCl_3$)

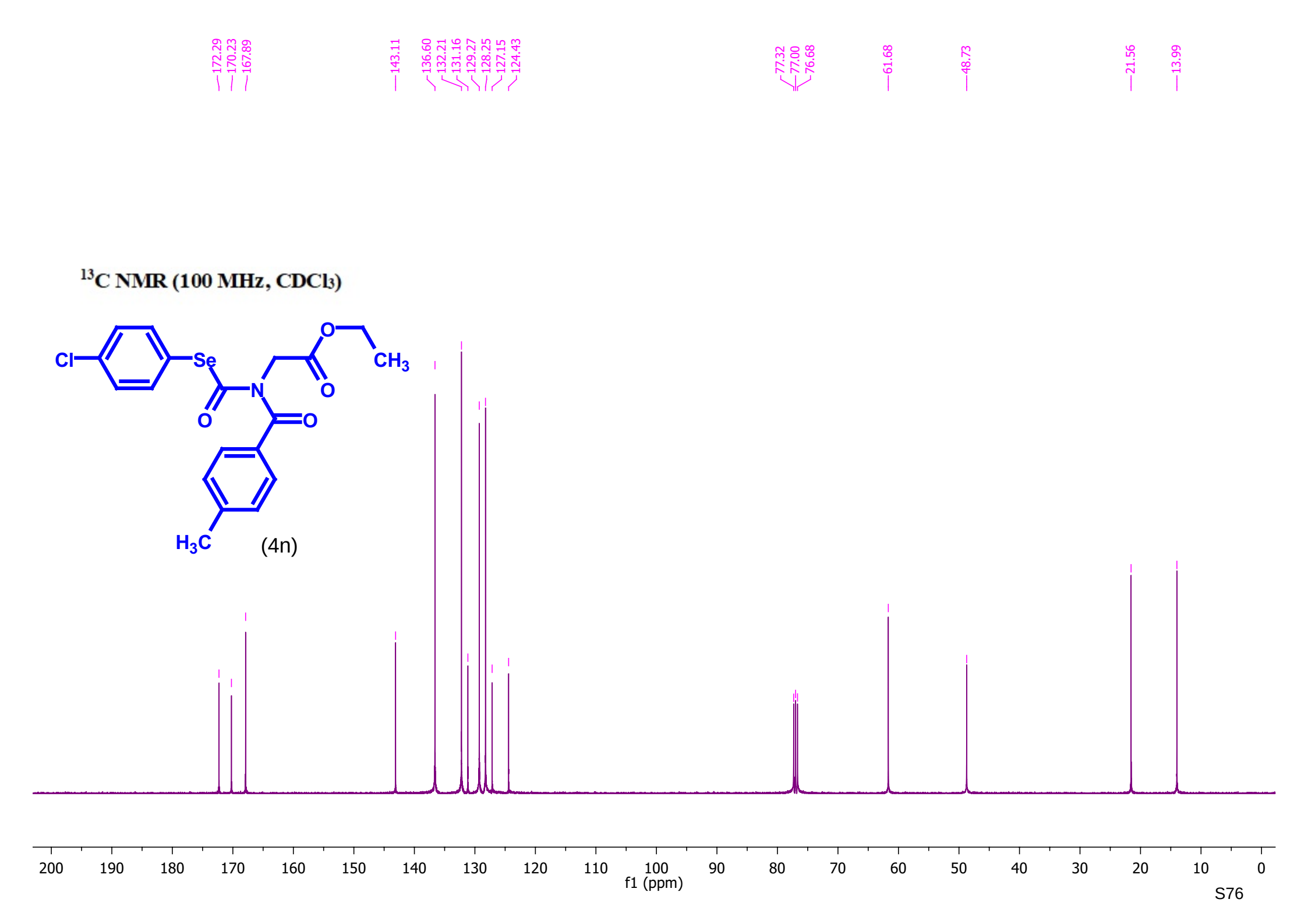
(4l)

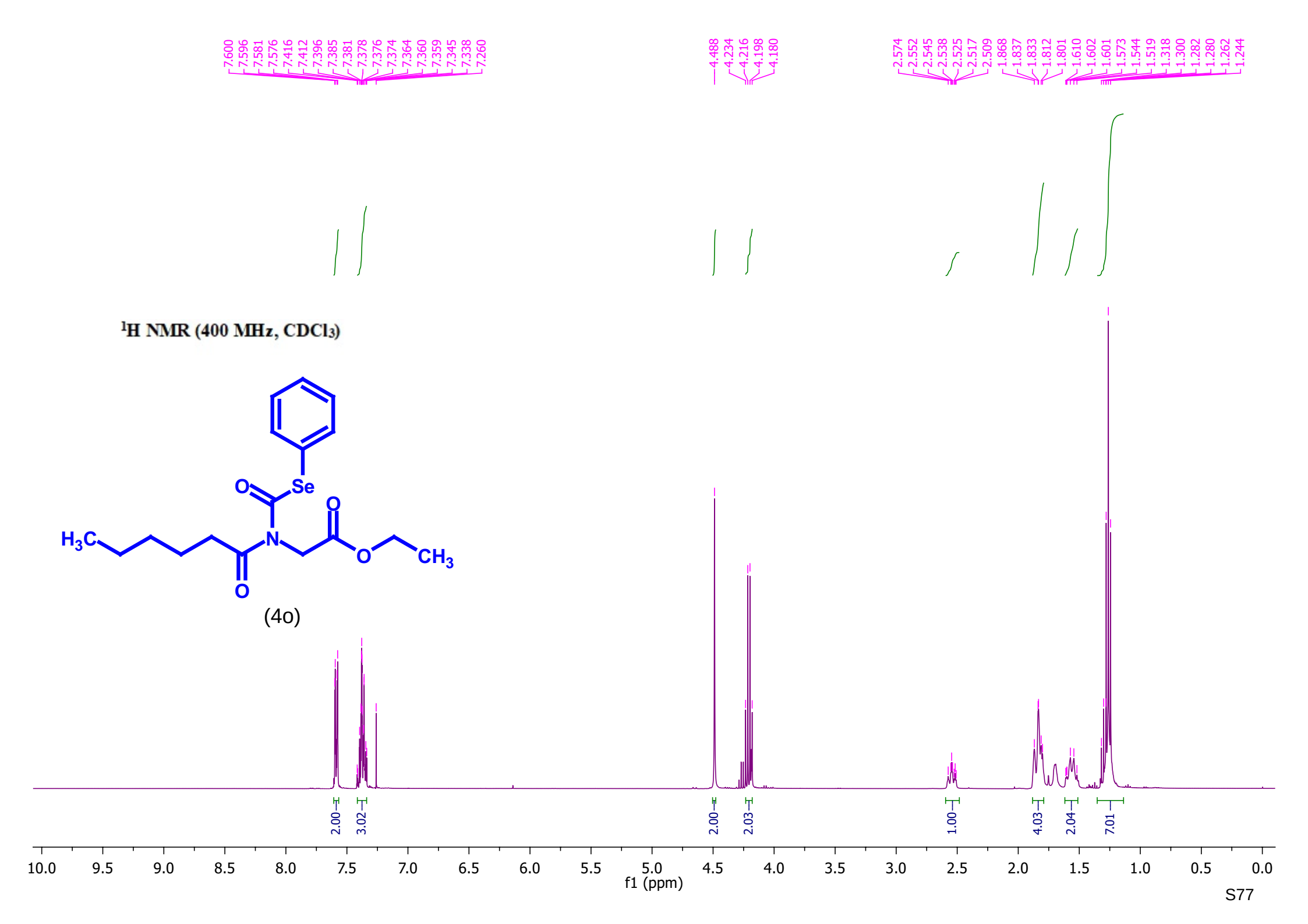

173.17
169.39
167.84
142.85
136.33
130.85
129.50
129.23
129.10
128.22
127.70
77.32
77.00
76.68
61.74
49.39
21.62
14.02
^{13}C NMR (100 MHz, CDCl$_3$)
Se
N
O
O
O
O
CH$_3$
H$_3$C
(4l)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

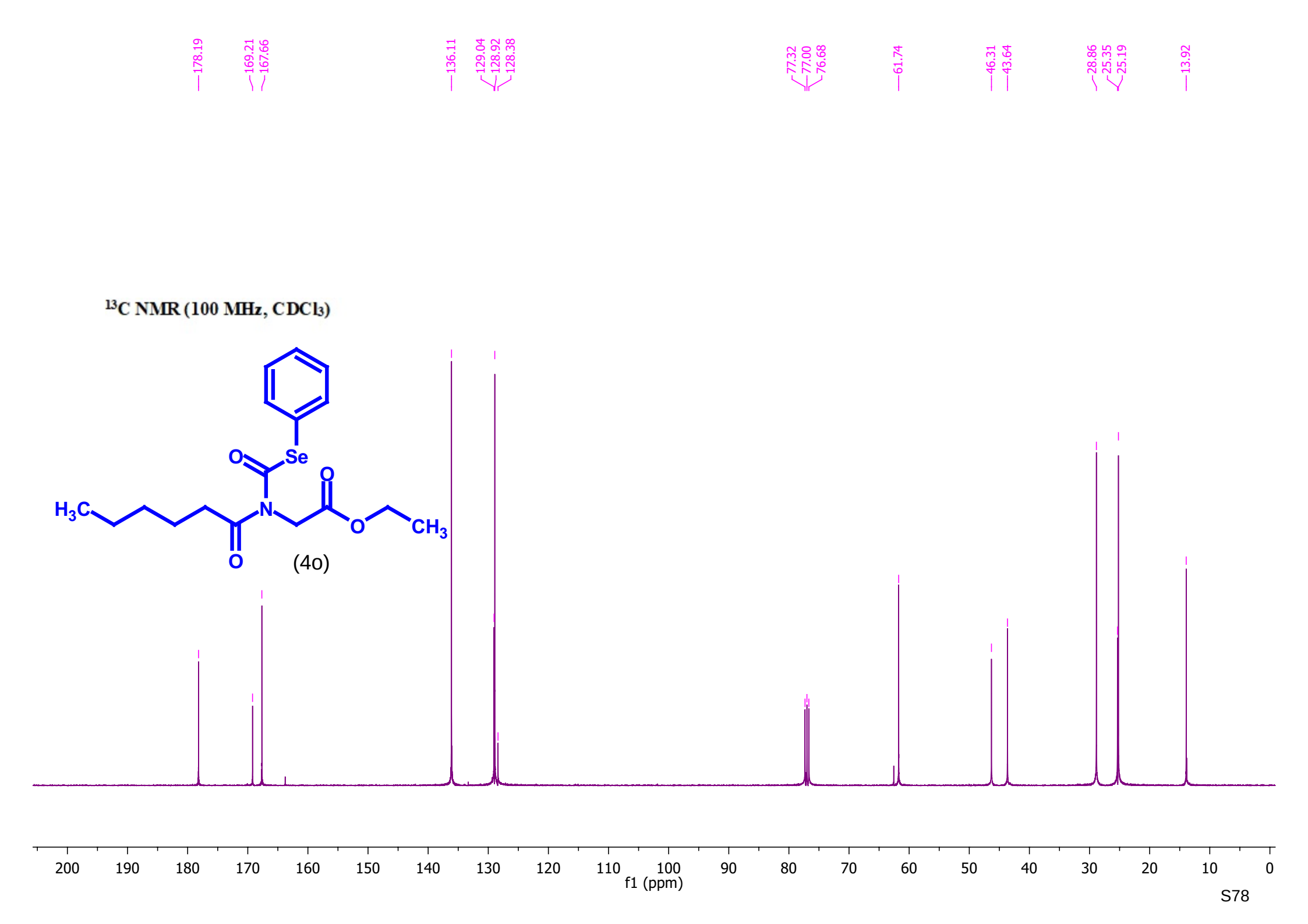

7.614
7.594
7.305
7.286
7.265
7.260
7.244
7.062
7.058
7.055
7.043
7.039
7.036
7.005
7.002
6.999
6.995
6.950
6.947
6.944
6.941
6.929
6.926
6.923
6.920
4.546
4.244
4.225
4.208
4.190
3.782
2.406
1.281
1.263
1.245
^{1}H NMR (400 MHz, CDCl$_3$)
O
Se
N
O
O
O
CH$_3$
H$_3$C
O
O
H$_3$C
2.06
2.00
2.01
1.09
1.05
1.06
2.02
2.11
3.19
3.03
3.10
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)

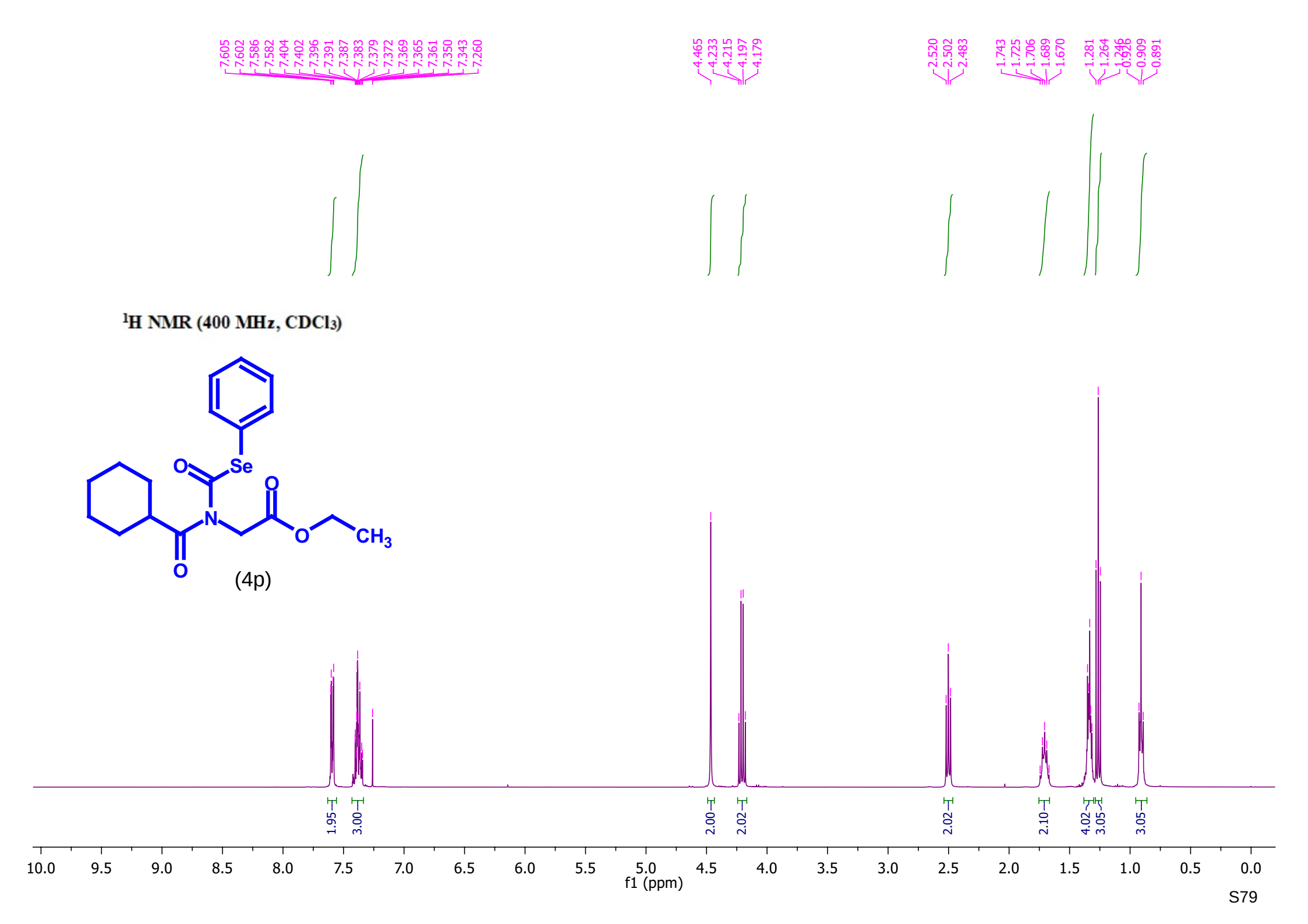

172.36
170.81
168.03
159.66
143.02
131.46
129.82
129.24
128.73
128.36
127.37
120.21
115.82
77.32
77.00
76.68
61.66
55.24
48.60
21.57
14.01
^{13}C NMR (100 MHz, CDCl$_3$)
O
H$_3$C
Se
N
O
O
O
O
CH$_3$
H$_3$C
(4m)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

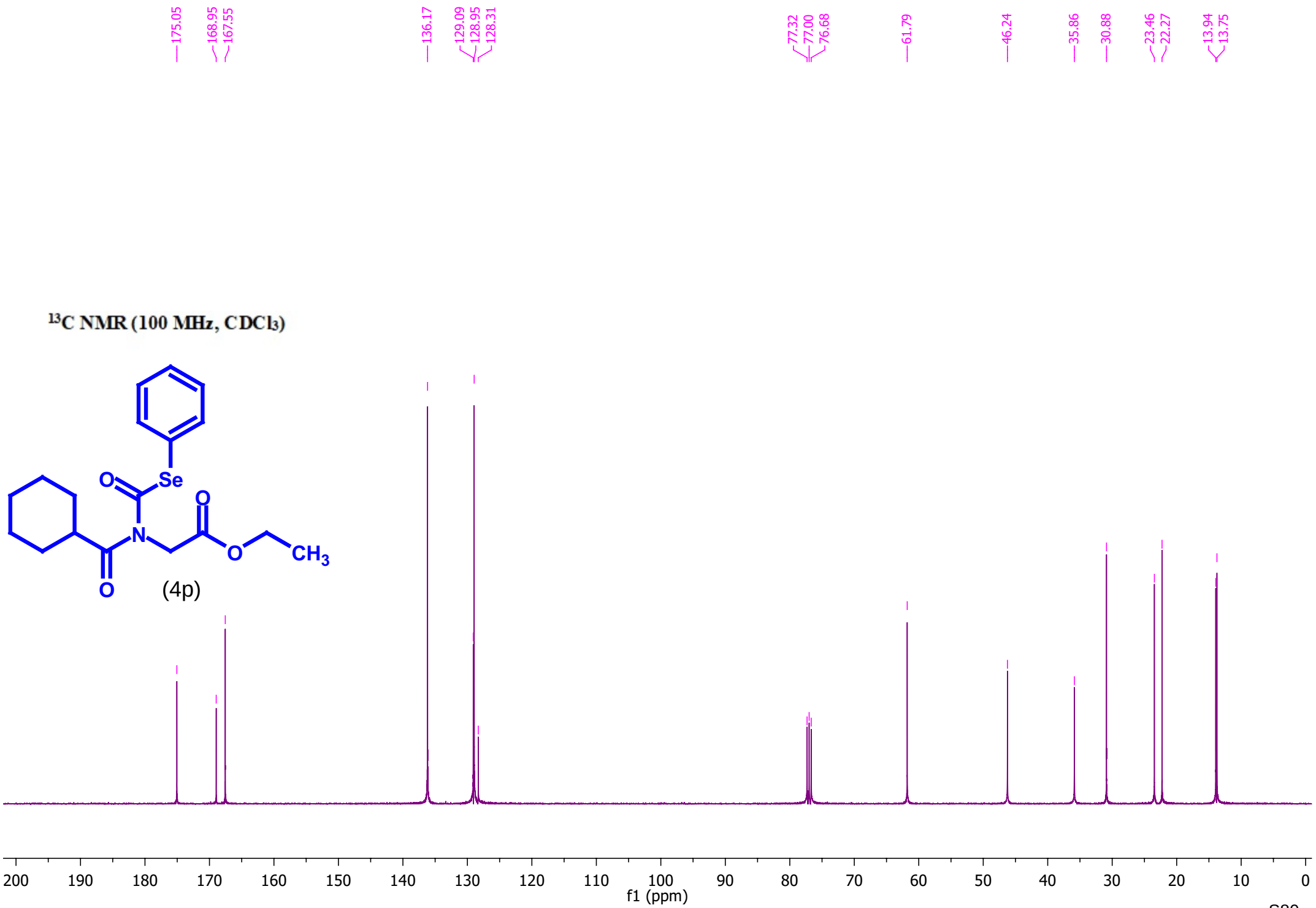

7.597
7.577
7.510
7.488
7.329
7.308
7.266
7.260
7.246
4.516
4.237
4.219
4.201
4.183
2.404
1.272
1.254
1.236
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
Se
N
O
O
O
O
CH$_3$
H$_3$C
(4n)
2.00
2.07
2.04
1.02
1.03
2.08
2.08
3.02
3.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

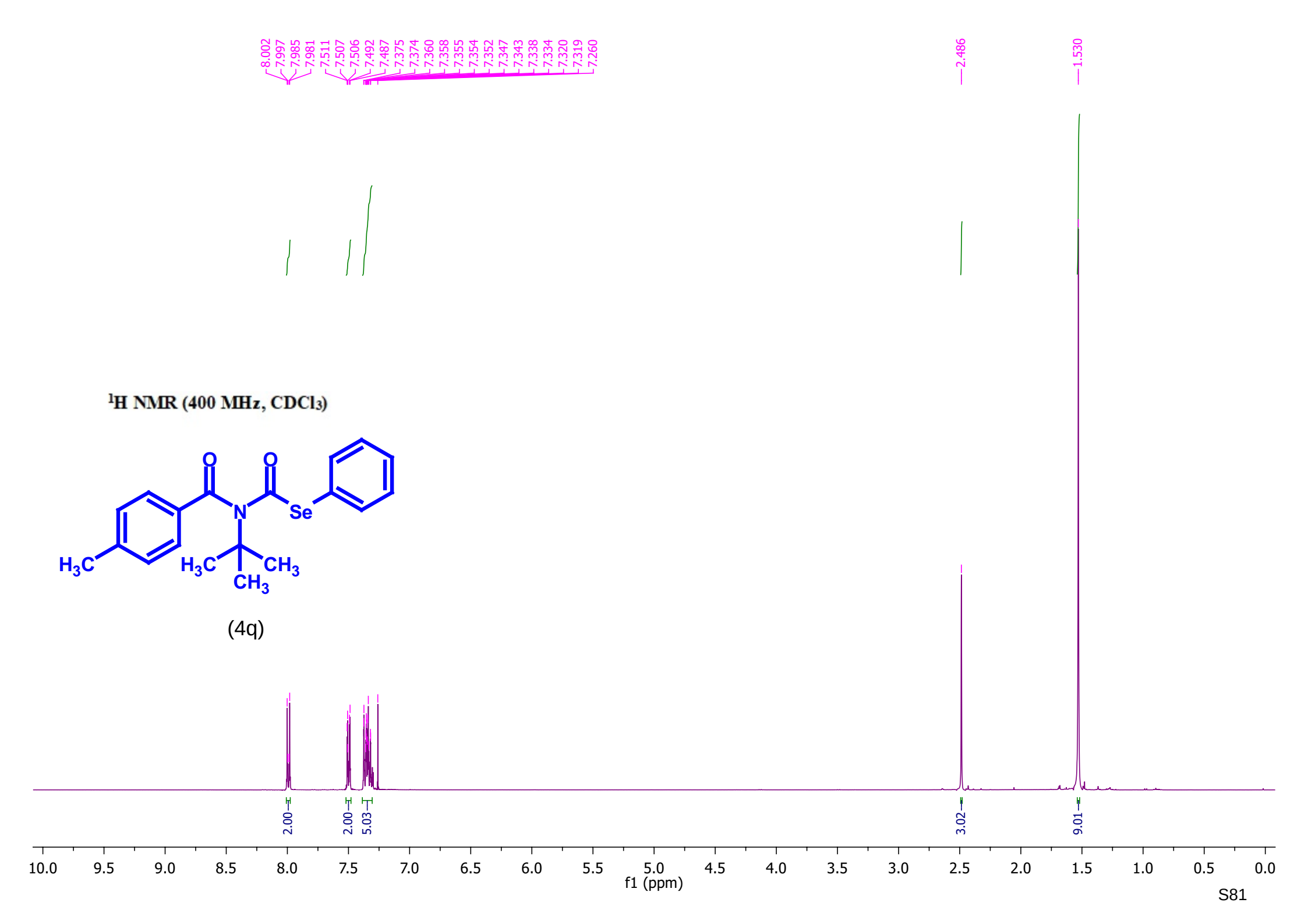

172.29
170.23
167.89
143.11
136.60
132.21
131.16
129.27
128.25
127.15
124.43
77.32
77.00
76.68
61.68
48.73
21.56
13.99
13C NMR (100 MHz, CDCl3)
Cl
Se
N
O
O
O
O
O
CH3
H3C
(4n)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.600
7.596
7.581
7.576
7.416
7.412
7.396
7.385
7.381
7.378
7.376
7.374
7.364
7.360
7.359
7.345
7.338
7.260
4.488
4.234
4.216
4.198
4.180
2.574
2.552
2.545
2.538
2.525
2.517
2.509
1.868
1.837
1.833
1.812
1.801
1.610
1.602
1.601
1.573
1.544
1.519
1.318
1.300
1.282
1.280
1.262
1.244
1H NMR (400 MHz, CDCl3)
O
Se
O
N
O
H3C
O
CH3
(4o)
2.00
3.02
2.00
2.03
1.00
4.03
2.04
7.01
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)


178.19
169.21
167.66
136.11
129.04
128.92
128.38
77.32
77.00
76.68
61.74
46.31
43.64
28.86
25.35
25.19
13.92
^{13}C NMR (100 MHz, CDCl$_3$)
O
Se
O
N
O
O
H_3C
CH_3
O
(4o)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

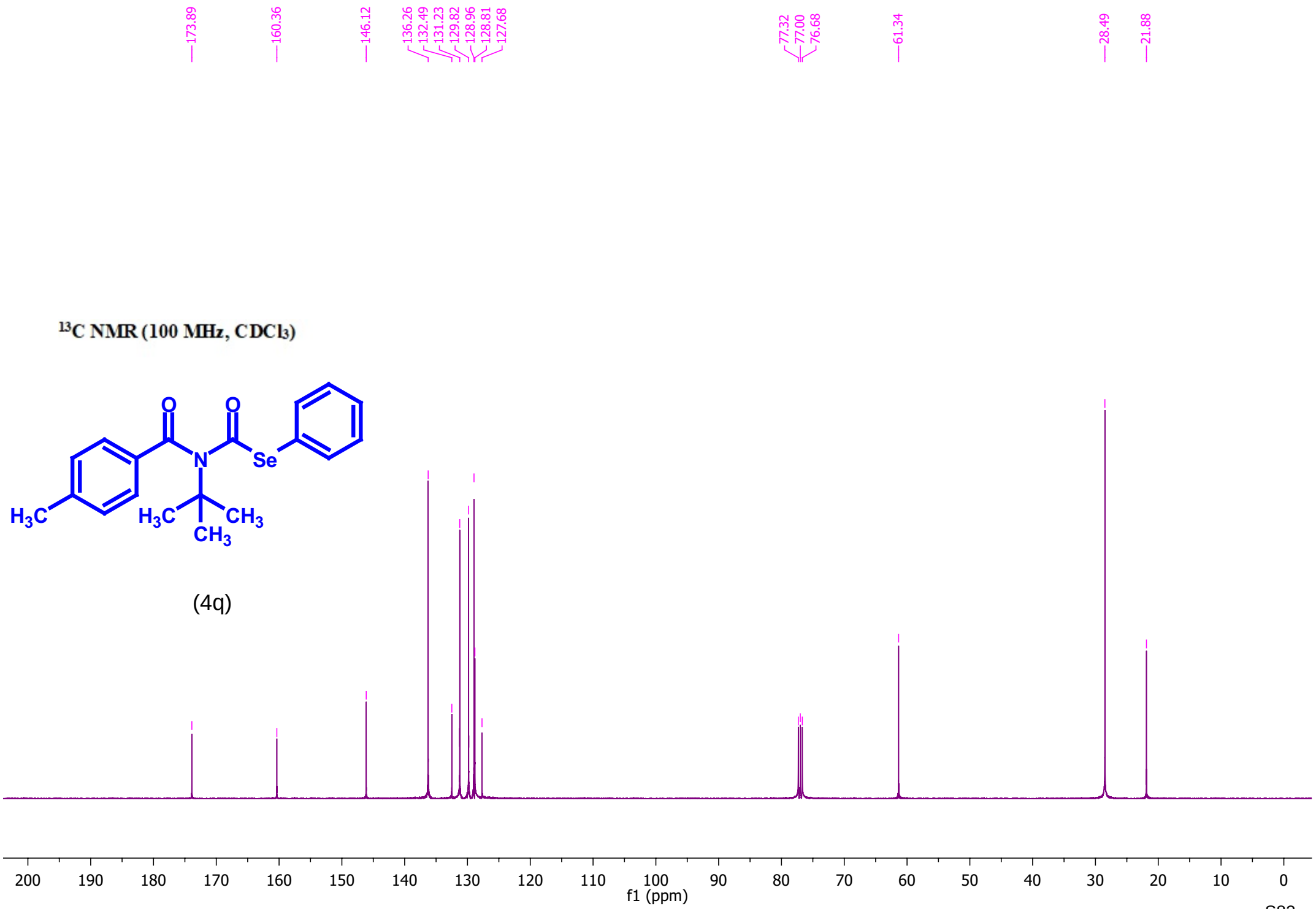

7.605
7.602
7.586
7.582
7.404
7.402
7.396
7.391
7.387
7.383
7.379
7.372
7.369
7.365
7.361
7.350
7.343
7.260
4.465
4.233
4.215
4.197
4.179
2.520
2.502
2.483
1.743
1.725
1.706
1.689
1.670
1.281
1.264
0.926
0.909
0.891
1H NMR (400 MHz, CDCl3)
O
Se
O
N
O
O
CH3
O
(4p)
1.95
3.00
2.00
2.02
2.02
2.10
4.02
3.05
3.05
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)

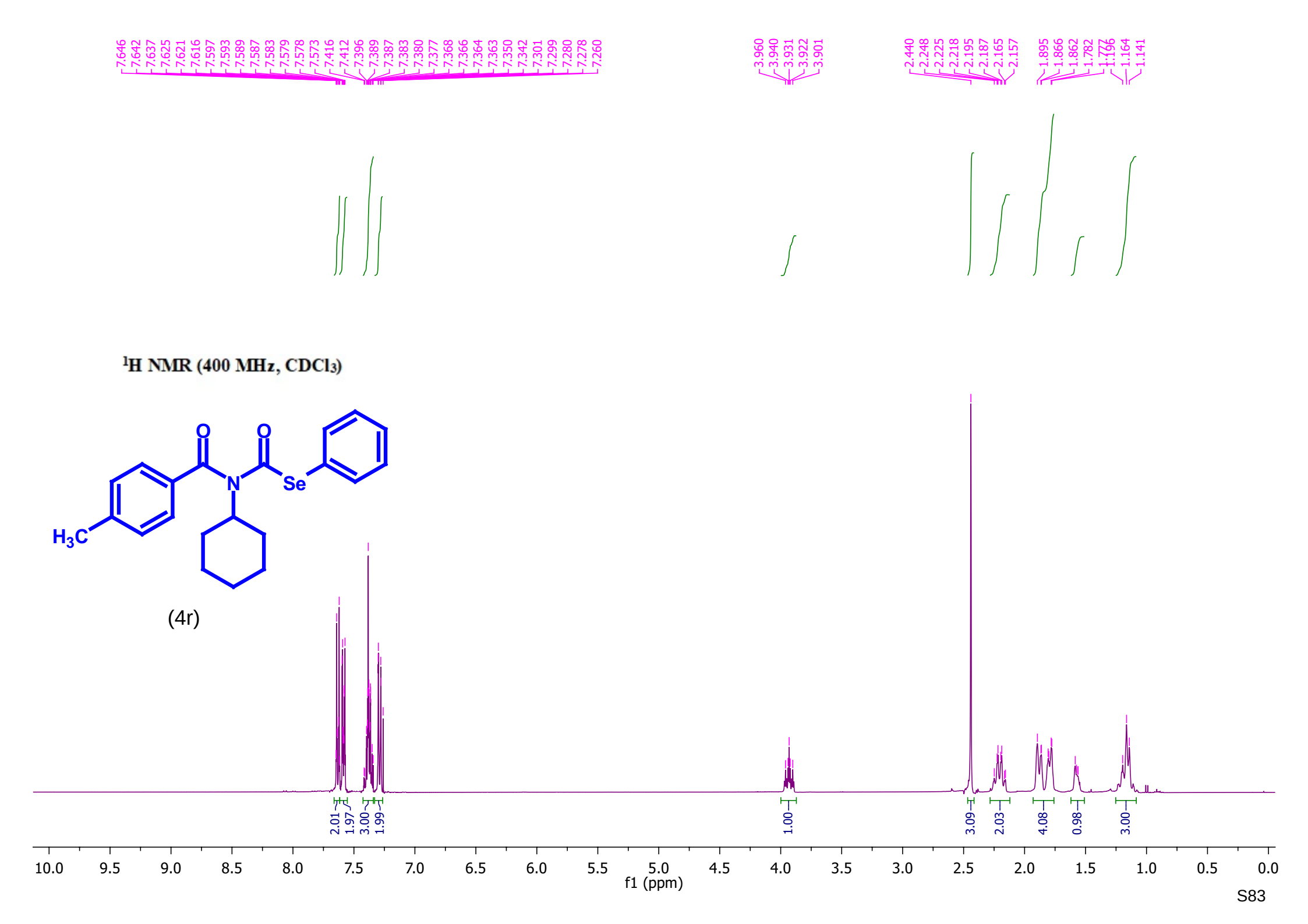

175.05
168.95
167.55
136.17
129.09
128.95
128.31
77.32
77.00
76.68
61.79
46.24
35.86
30.88
23.46
22.27
13.94
13.75
13C NMR (100 MHz, CDCl3)
O
Se
O
N
O
O
CH3
O
(4p)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

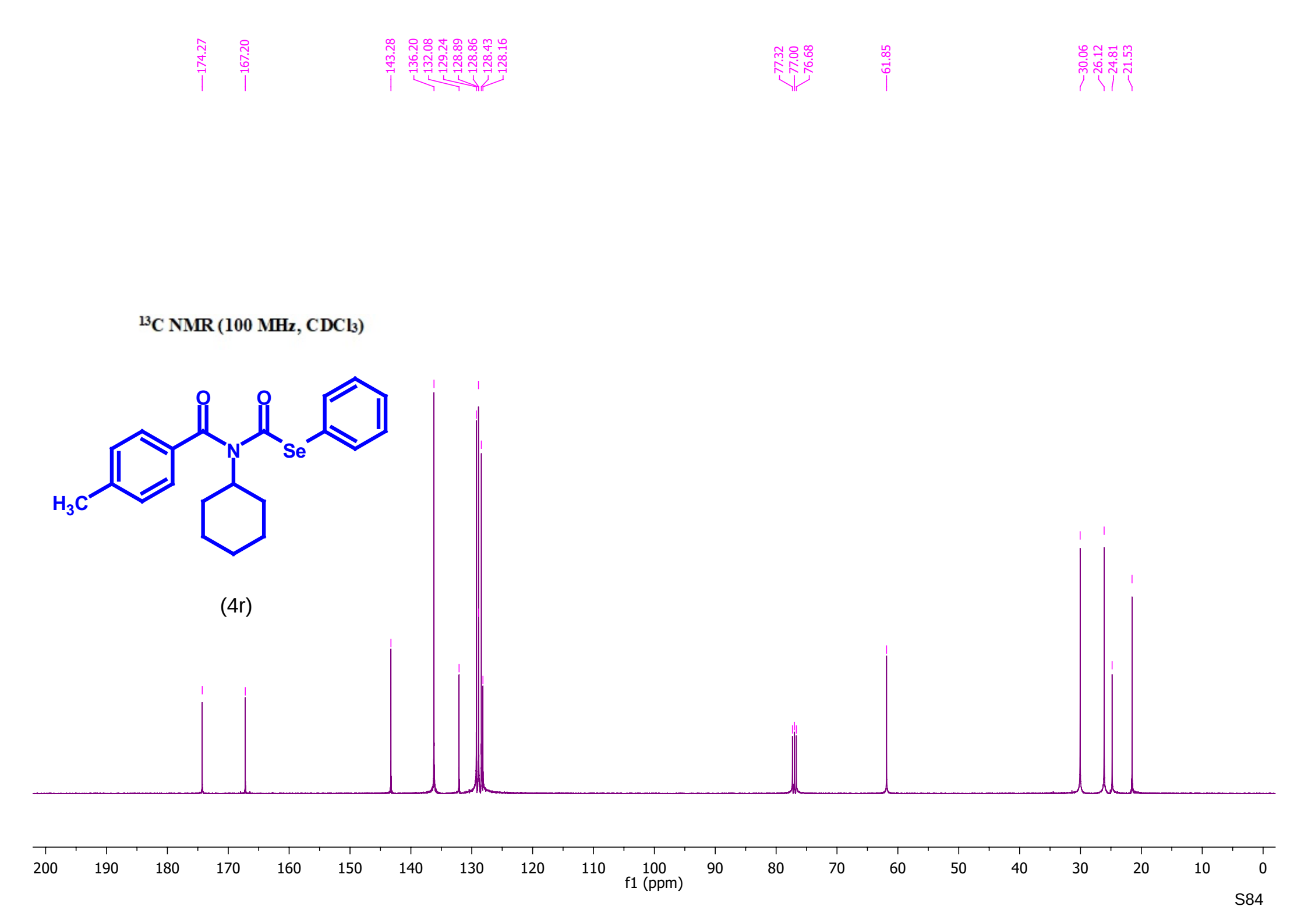


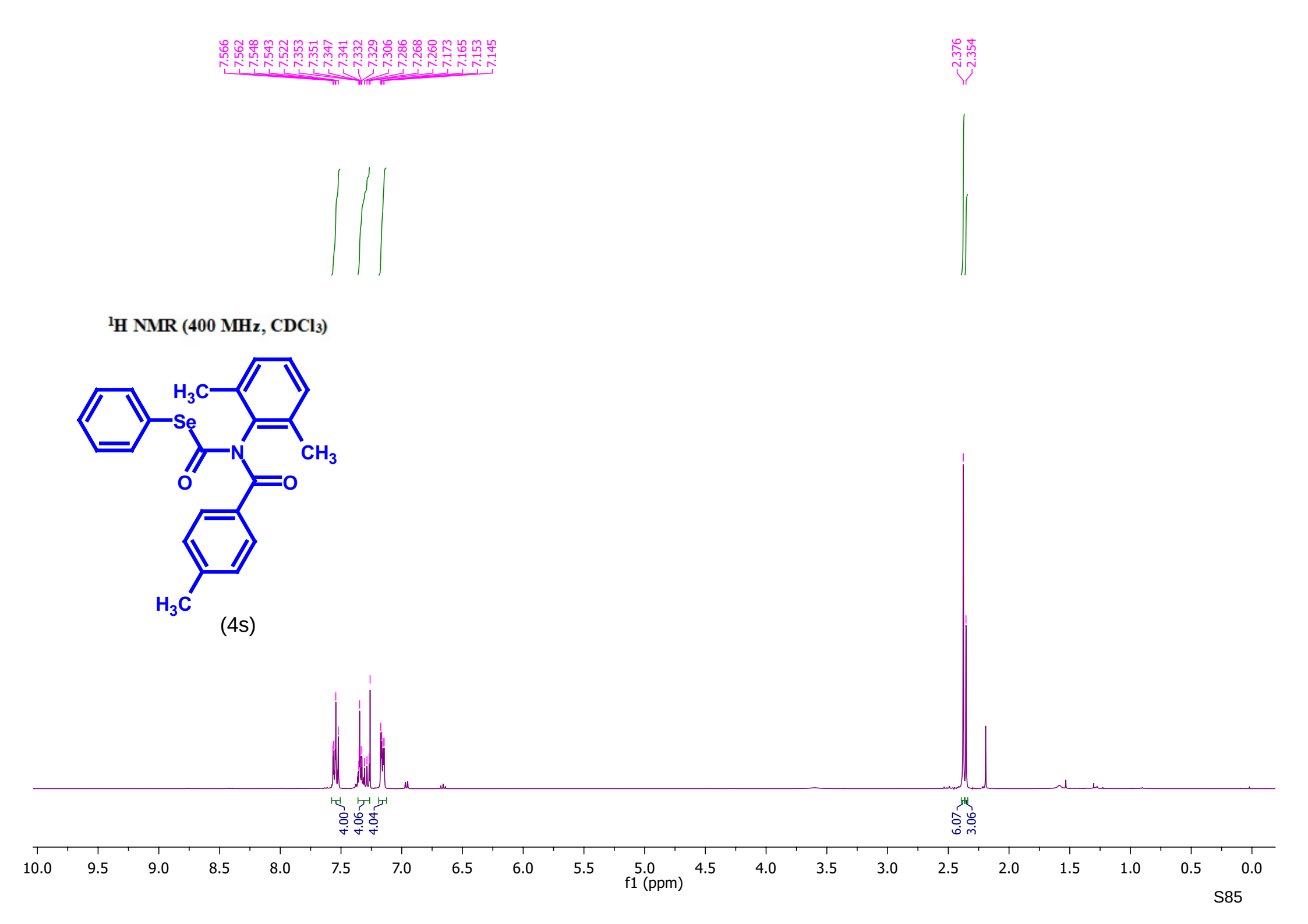
^{1}H NMR (400 MHz, $CDCl_3$)

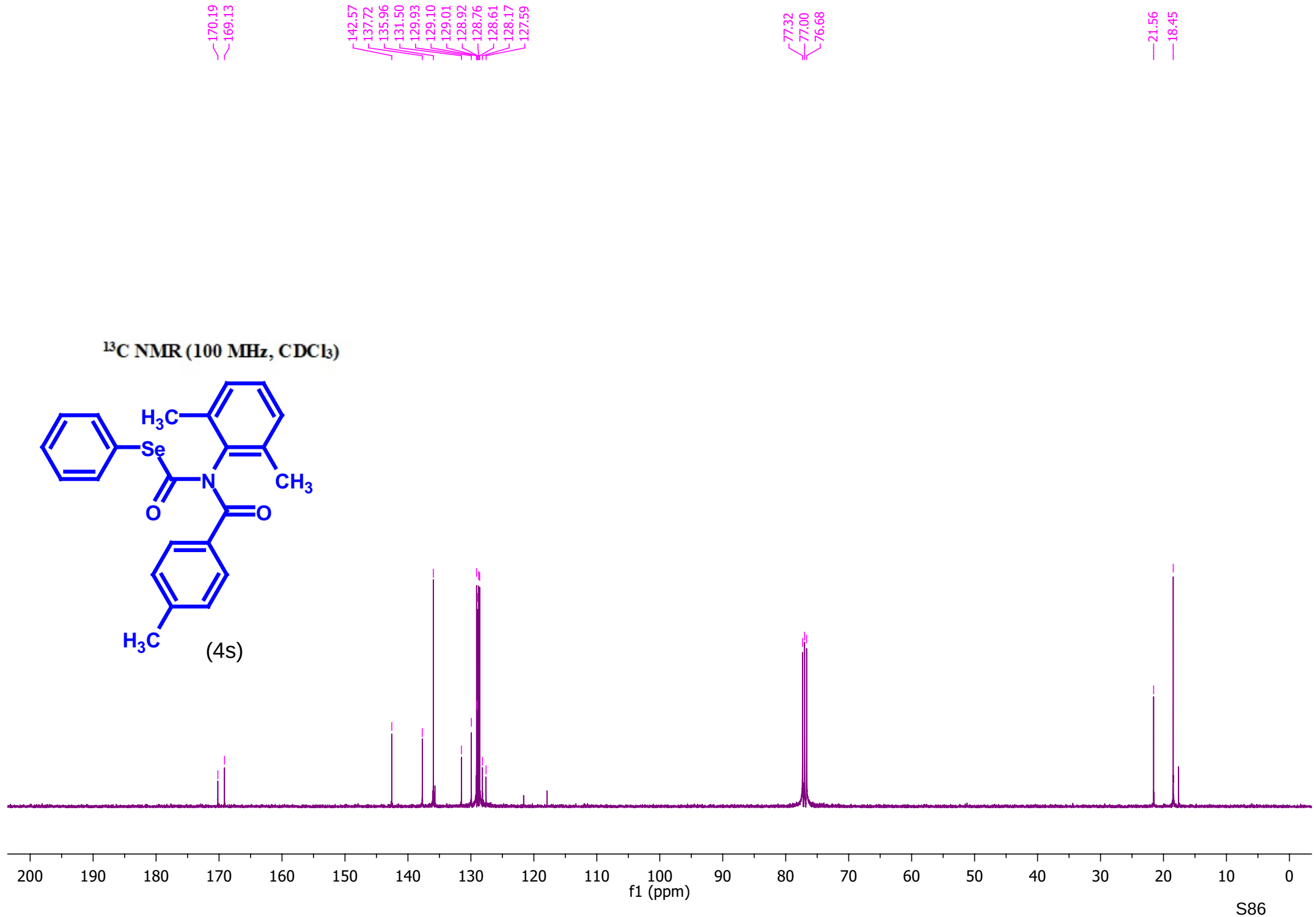
(4q)

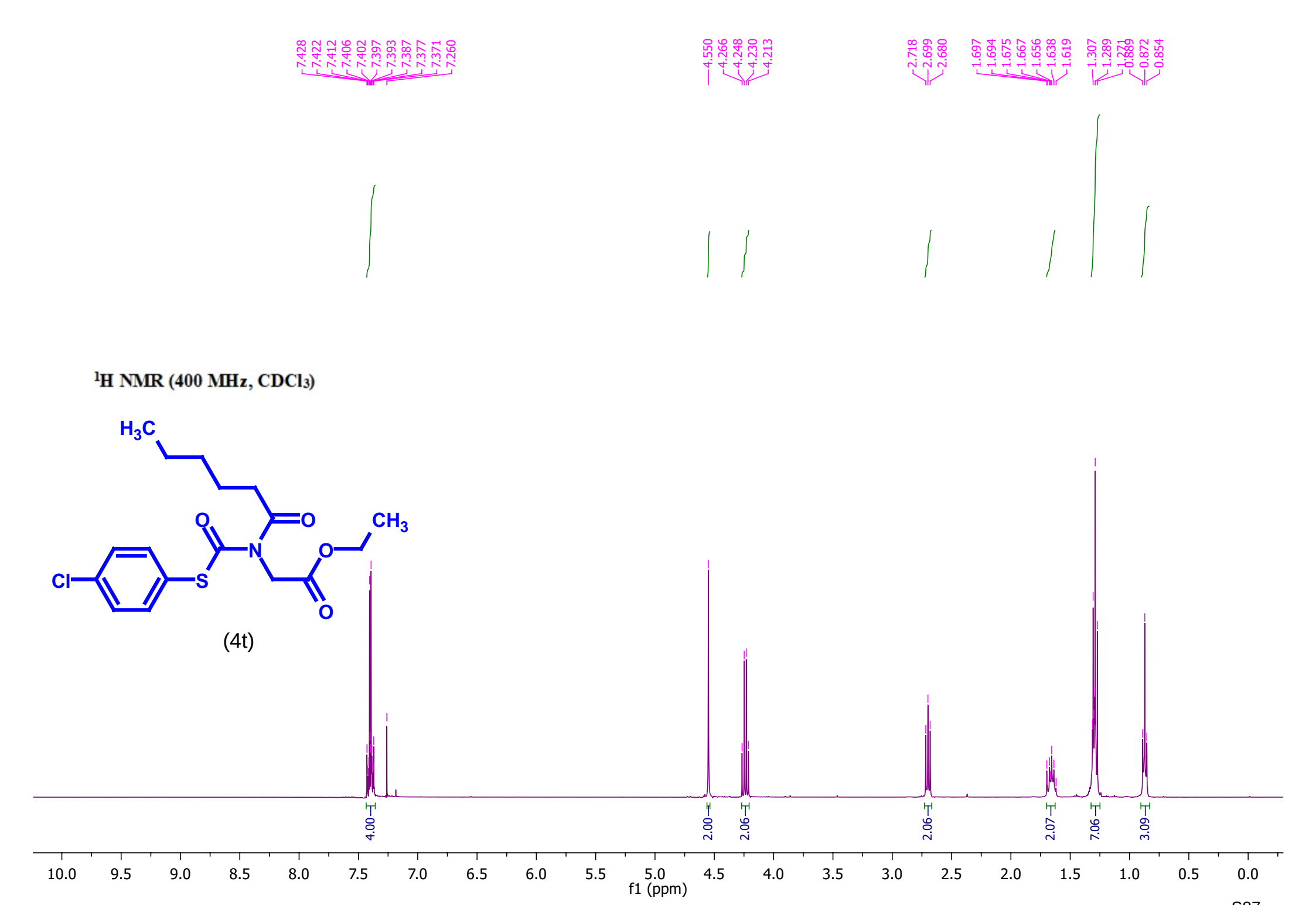

173.89
160.36
146.12
136.26
132.49
131.23
129.82
128.96
128.81
127.68
77.32
77.00
76.68
61.34
28.49
21.88
13C NMR (100 MHz, CDCl3)
O
O
N
Se
H3C
H3C
CH3
CH3
(4q)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

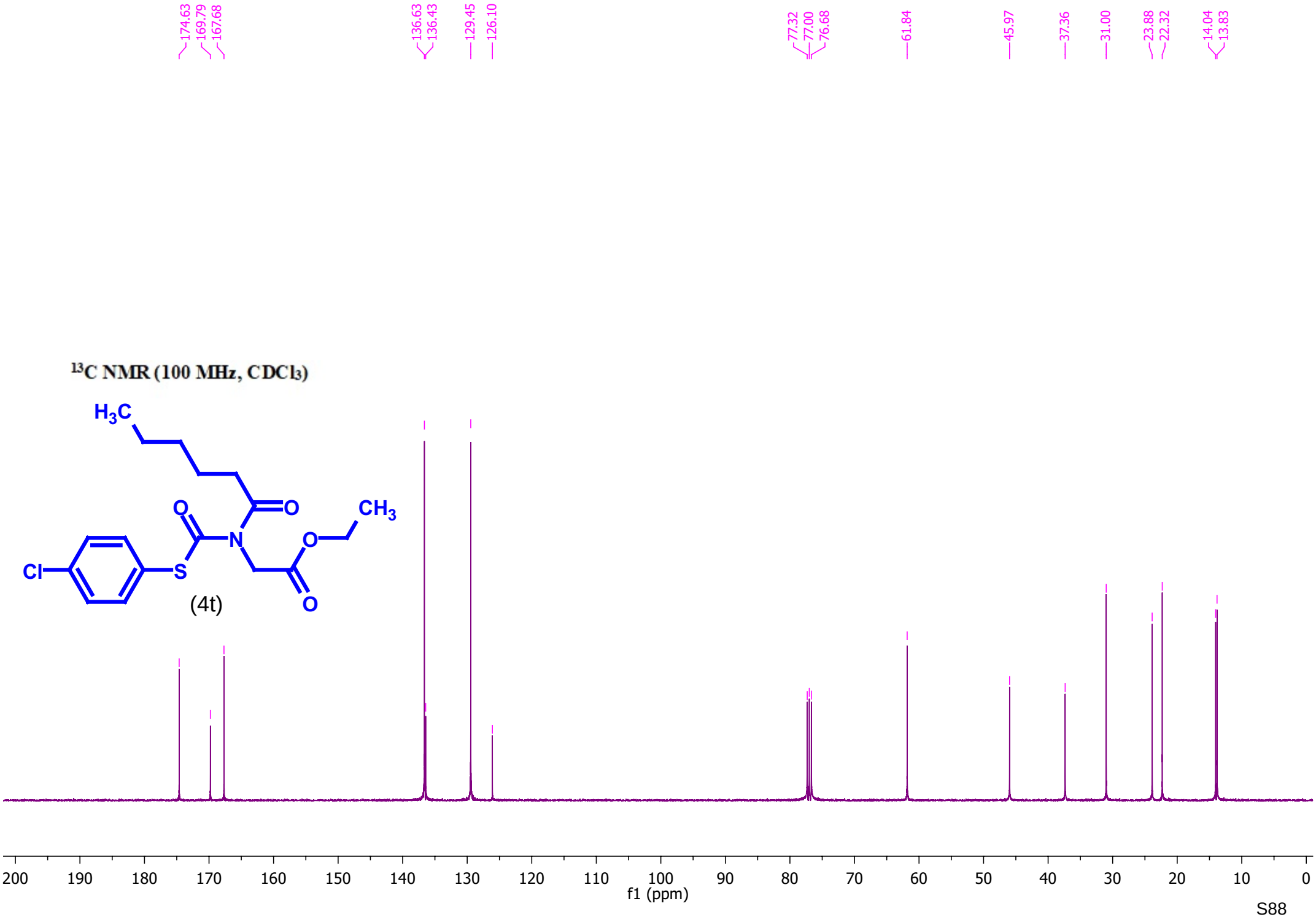

7.646
7.642
7.637
7.625
7.621
7.616
7.597
7.593
7.589
7.587
7.583
7.579
7.578
7.573
7.416
7.412
7.396
7.389
7.387
7.383
7.380
7.377
7.368
7.366
7.364
7.363
7.350
7.342
7.301
7.299
7.280
7.278
7.260
3.960
3.940
3.931
3.922
3.901
2.440
2.248
2.225
2.218
2.195
2.187
2.165
2.157
1.895
1.866
1.862
1.782
1.777
1.196
1.164
1.141
1H NMR (400 MHz, CDCl3)
O
O
N
Se
H3C
(4r)
2.01
1.97
3.00
1.99
1.00
3.09
2.03
4.08
0.98
3.00
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)

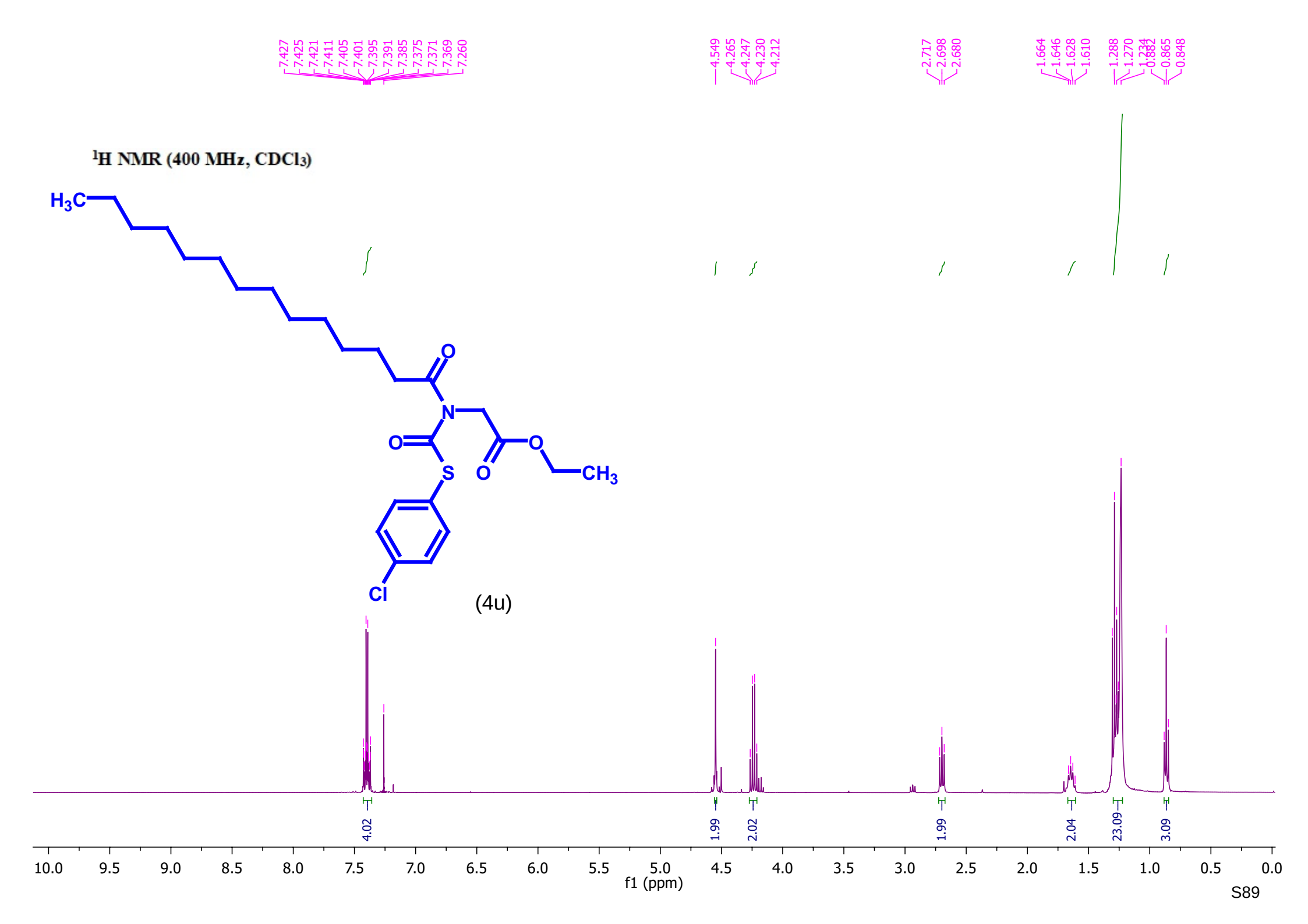

174.27
167.20
143.28
136.20
132.08
129.24
128.89
128.86
128.43
128.16
77.32
77.00
76.68
61.85
30.06
26.12
24.81
21.53
13C NMR (100 MHz, CDCl3)
O
O
N
Se
H3C
(4r)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

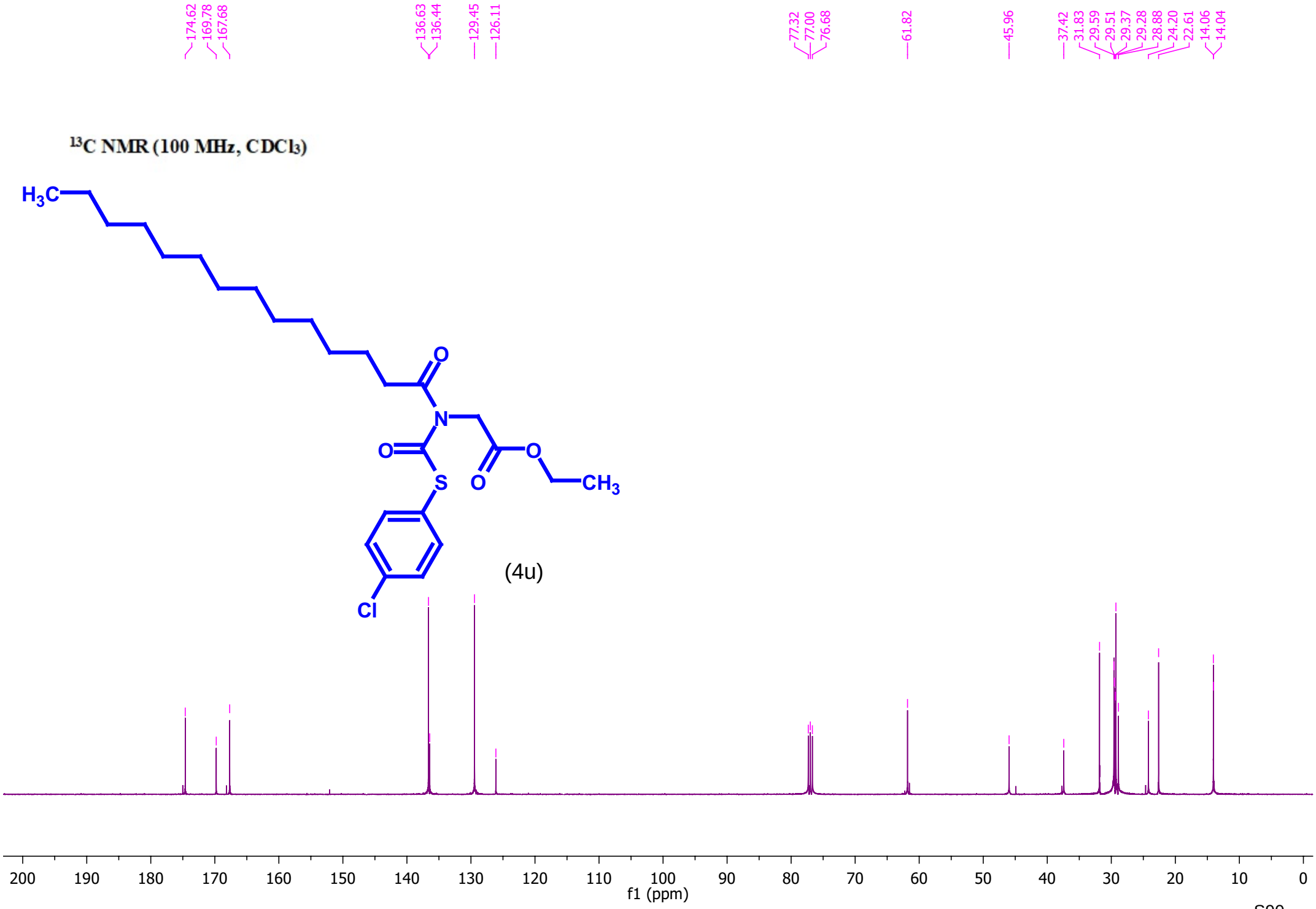

7.566
7.562
7.548
7.543
7.522
7.353
7.351
7.347
7.341
7.332
7.329
7.306
7.286
7.268
7.260
7.173
7.165
7.153
7.145
2.376
2.354
^{1}H NMR (400 MHz, CDCl$_3$)
H$_3$C
Se
N
CH$_3$
O
O
H$_3$C
(4s)
4.00
4.06
4.04
6.07
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

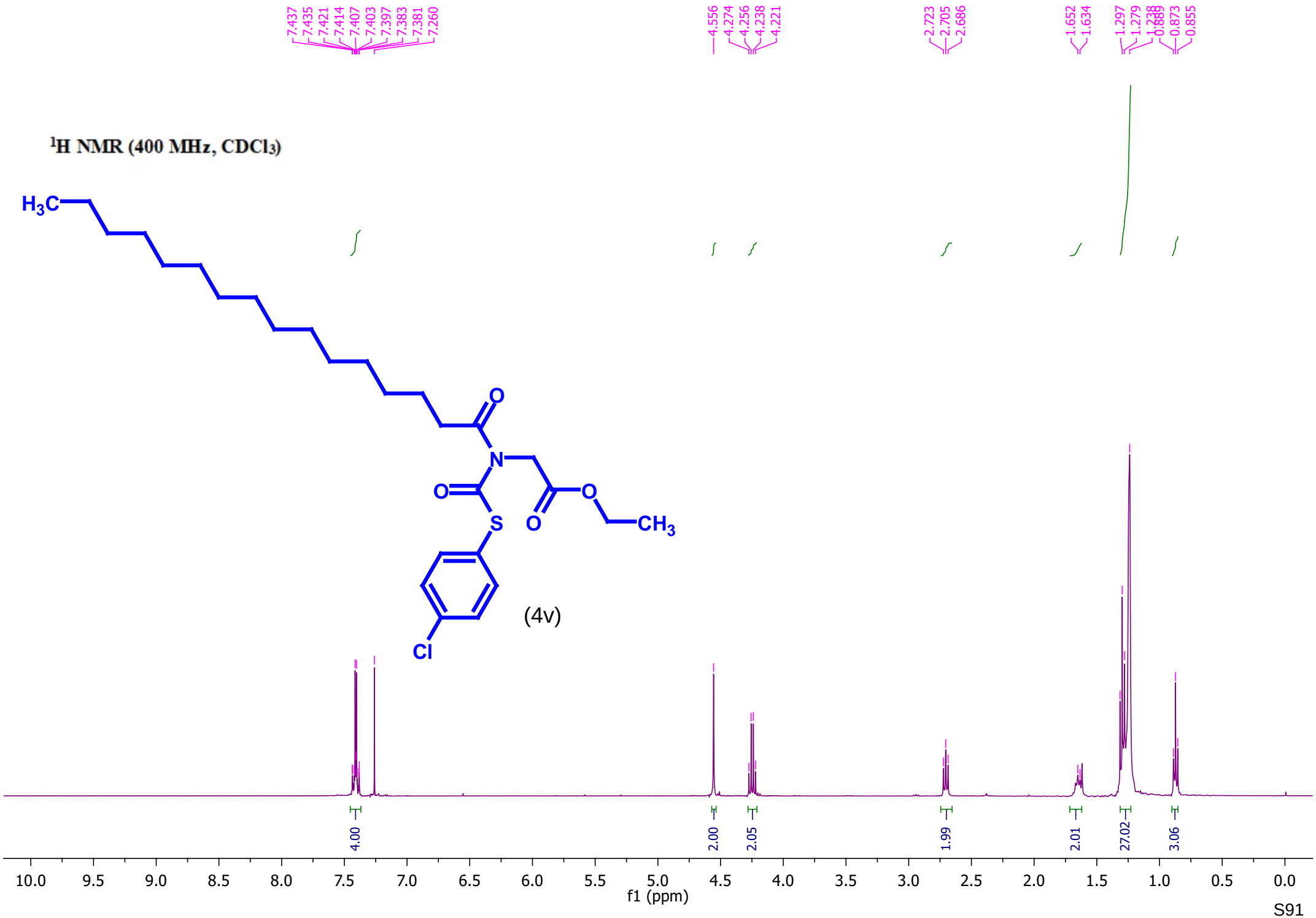

170.19
169.13
142.57
137.72
135.96
131.50
129.93
129.10
129.01
128.92
128.76
128.61
128.17
127.59
77.32
77.00
76.68
21.56
18.45
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
Se
N
CH$_3$
O
O
H$_3$C
(4s)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

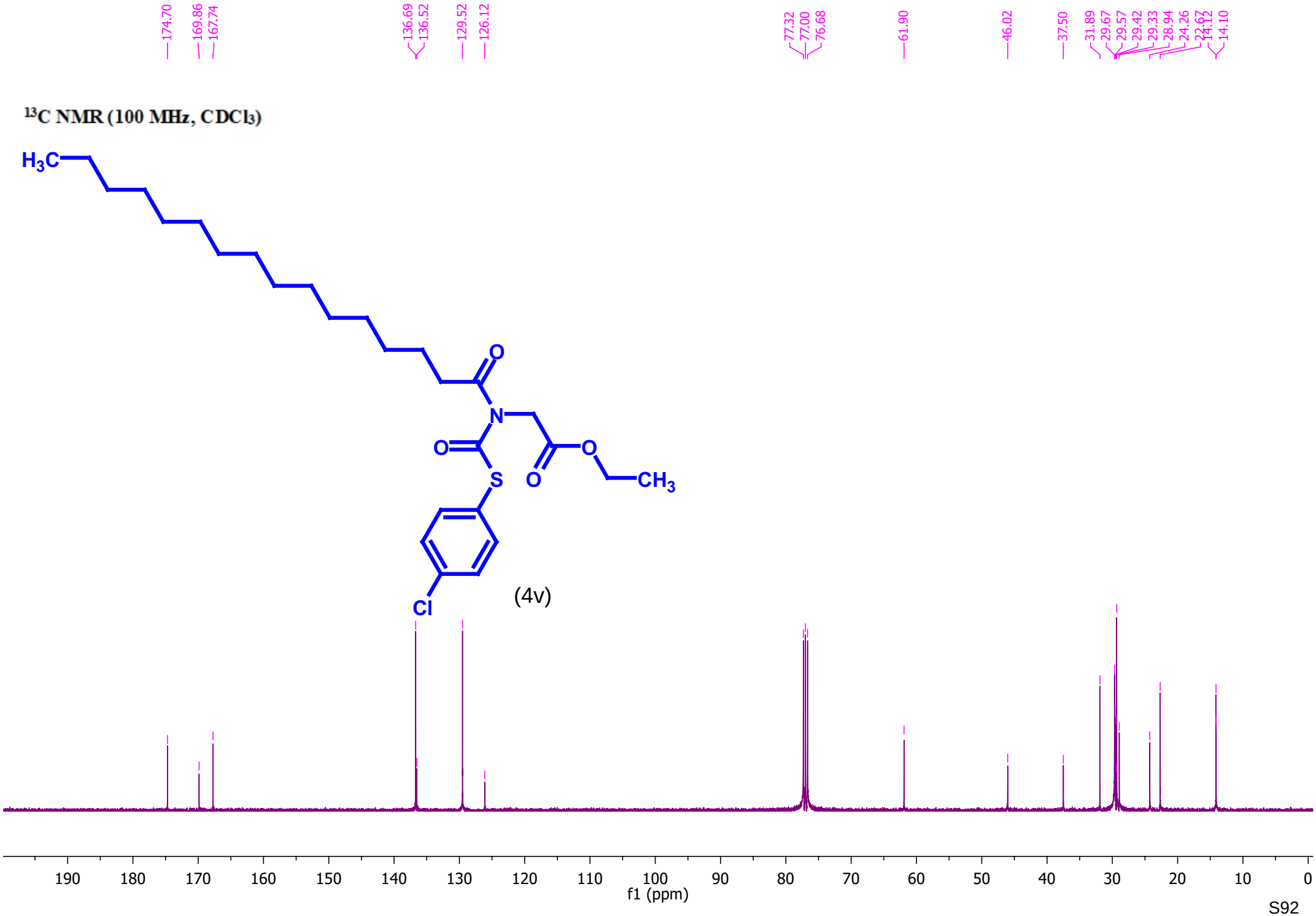

7.428
7.422
7.412
7.406
7.402
7.397
7.393
7.387
7.377
7.371
7.260
4.550
4.266
4.248
4.230
4.213
2.718
2.699
2.680
1.697
1.694
1.675
1.667
1.656
1.638
1.619
1.307
1.289
1.271
0.889
0.872
0.854
¹H NMR (400 MHz, CDCl₃)
H3C
O
O
CH3
O
N
O
Cl
S
O
(4t)
4.00
2.00
2.06
2.06
2.07
7.06
3.09
10.0
9.5
9.0
8.5
8.0
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)

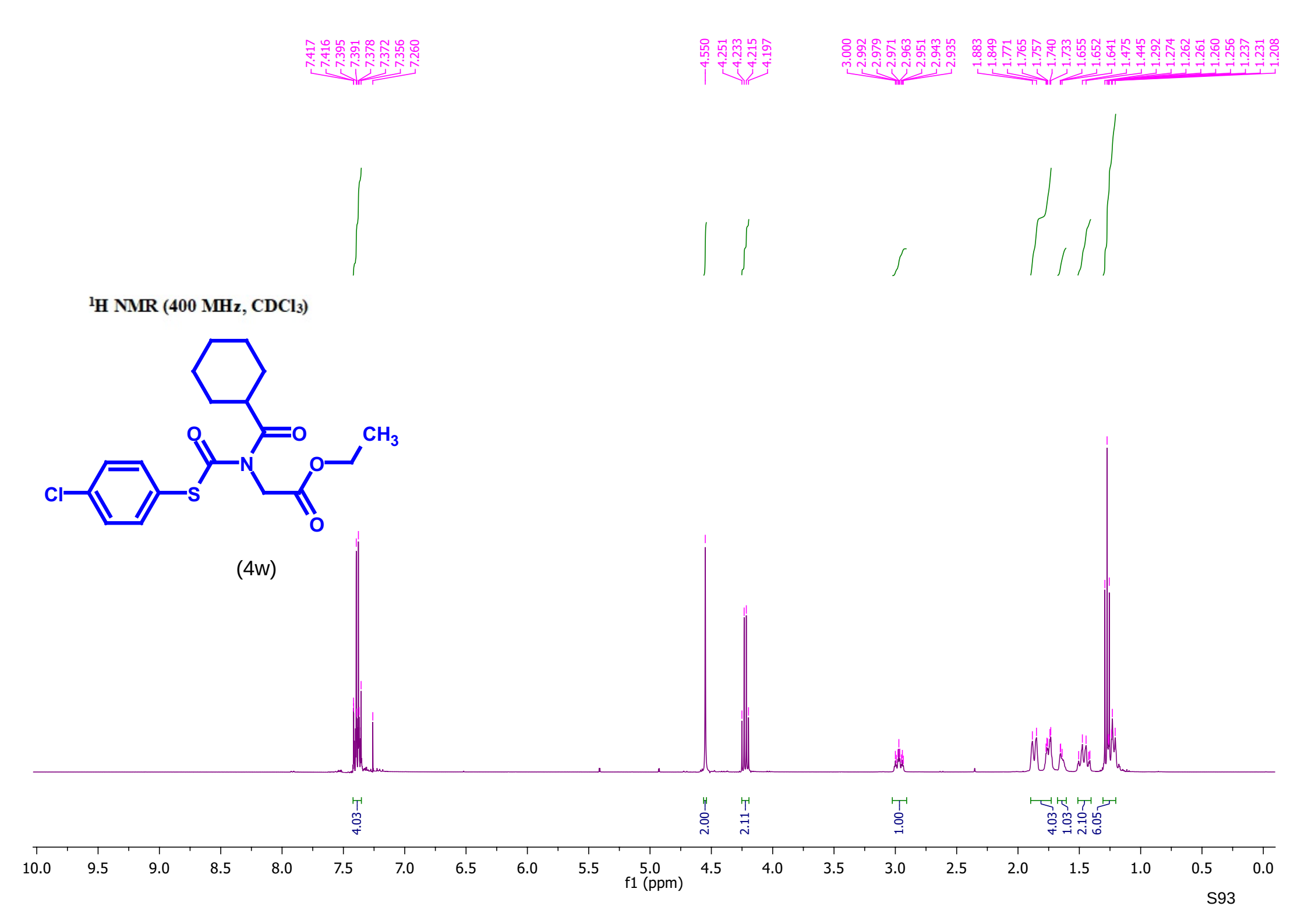

174.63
169.79
167.68
136.63
136.43
129.45
126.10
77.32
77.00
76.68
61.84
45.97
37.36
31.00
23.88
22.32
14.04
13.83
13C NMR (100 MHz, CDCl3)
H3C
O
O
CH3
O
N
O
Cl
S
O
(4t)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

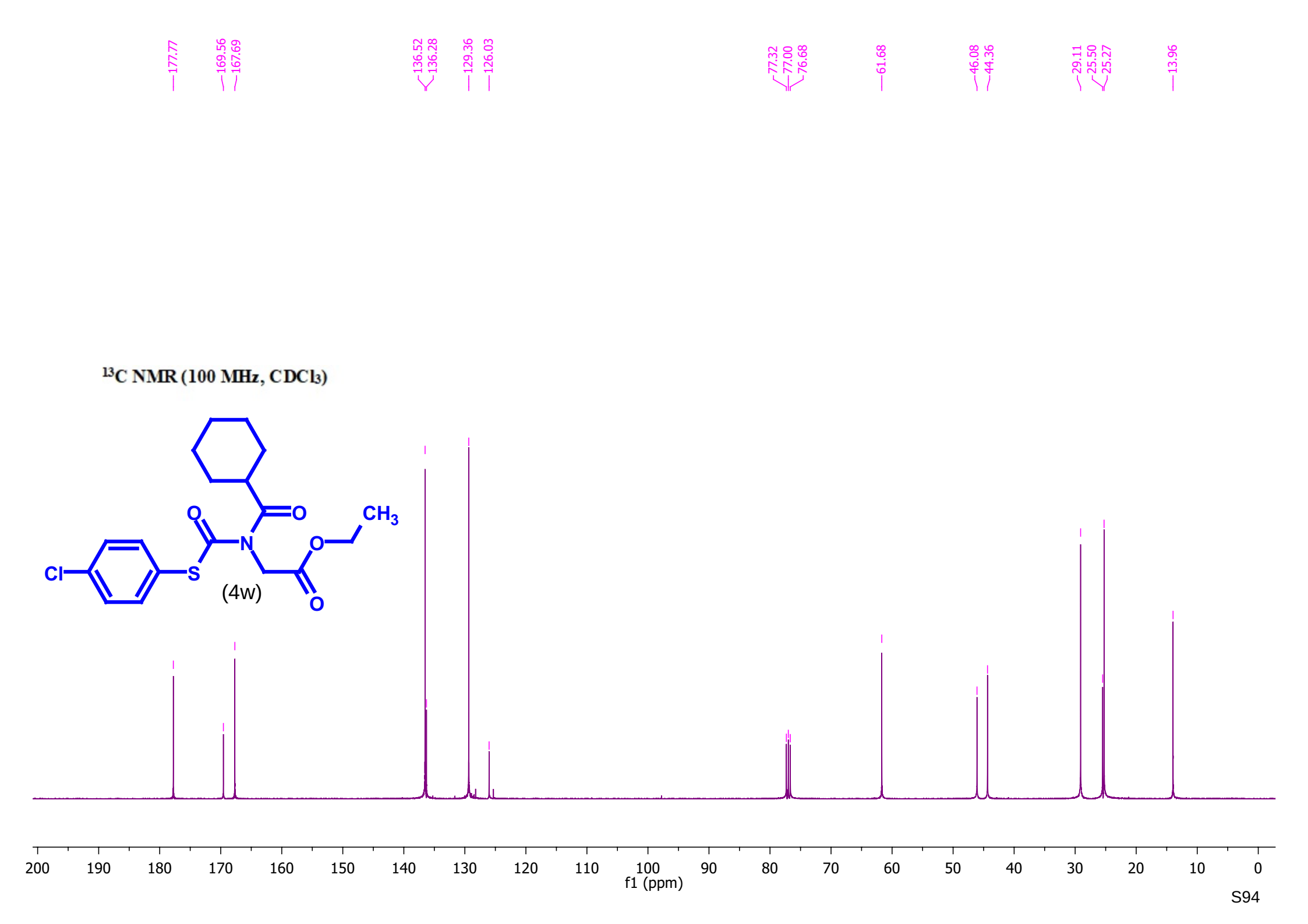

7.427
7.425
7.421
7.411
7.405
7.401
7.395
7.391
7.385
7.375
7.371
7.369
7.260
4.549
4.265
4.247
4.230
4.212
2.717
2.698
2.680
1.664
1.646
1.628
1.610
1.288
1.270
1.234
0.882
0.865
0.848
¹H NMR (400 MHz, CDCl₃)
H3C
O
N
O
O
S
O
CH3
Cl
(4u)
4.02
1.99
2.02
1.99
2.04
23.09
3.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
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)

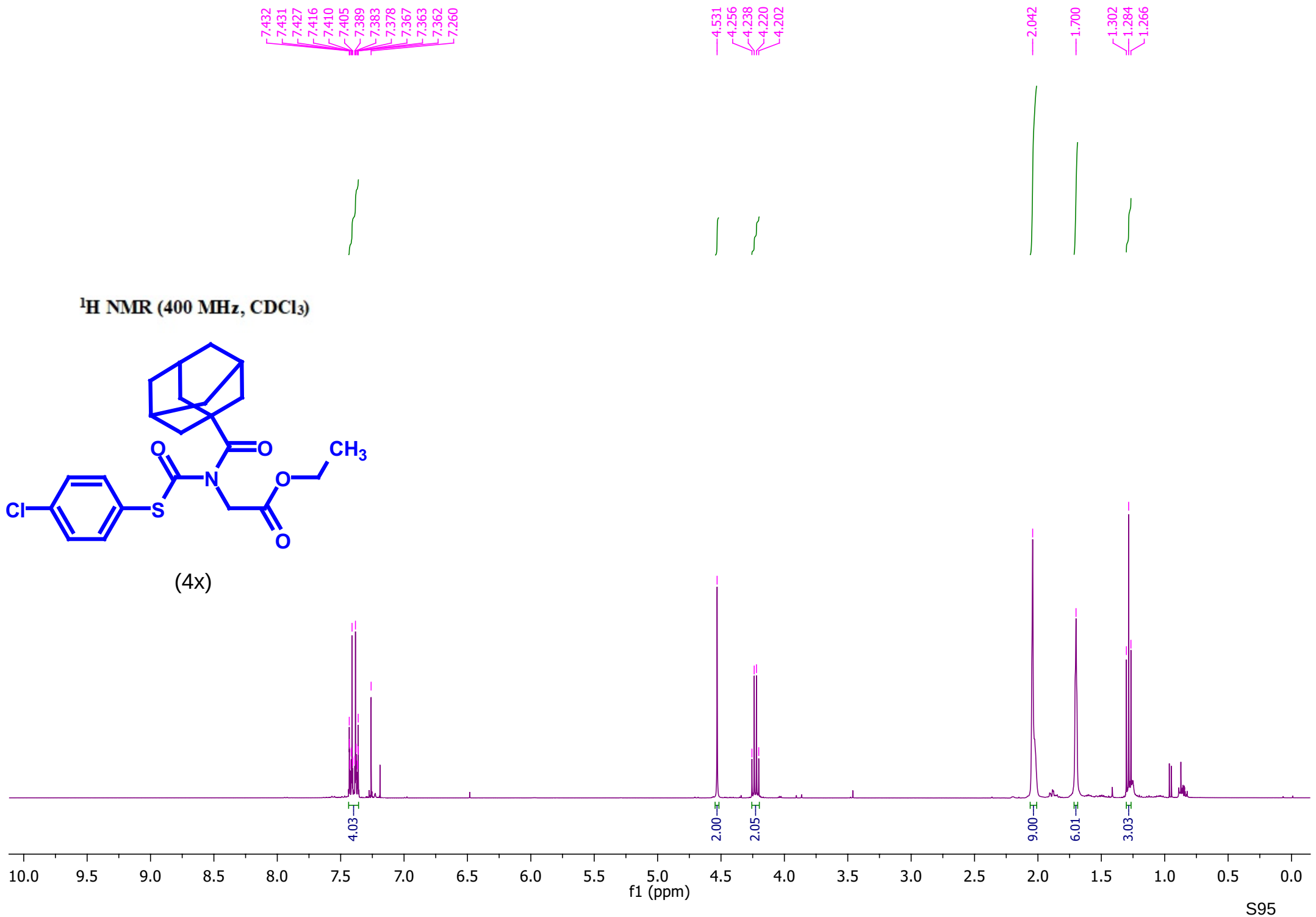

174.62
169.78
167.68
136.63
136.44
129.45
126.11
77.32
77.00
76.68
61.82
45.96
37.42
31.83
29.59
29.51
29.37
29.28
28.88
24.20
22.61
14.06
14.04
13C NMR (100 MHz, CDCl3)
H3C
O
N
O
S
O
O
CH3
Cl
(4u)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

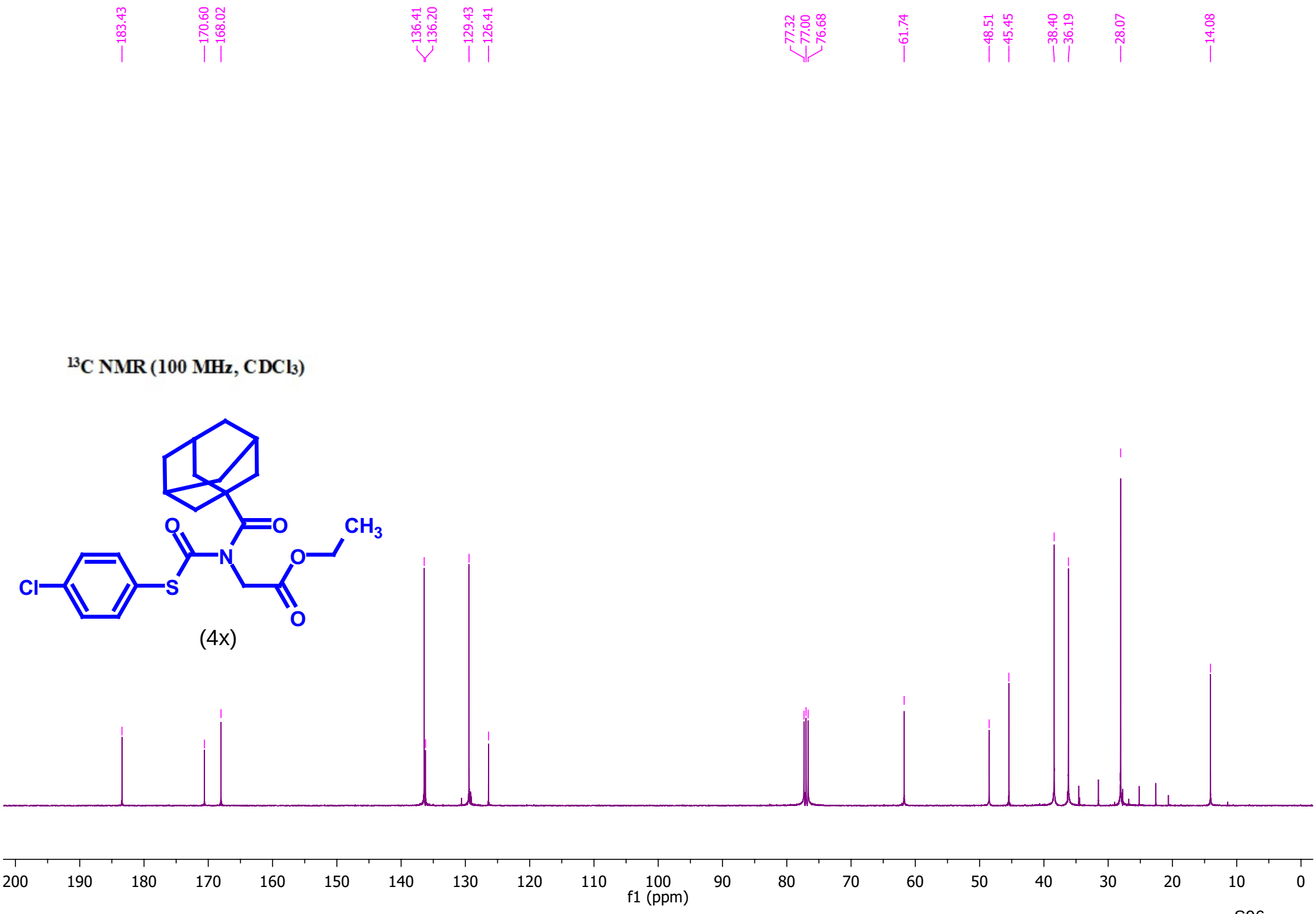

7.437
7.435
7.421
7.414
7.407
7.403
7.397
7.383
7.381
7.260
4.556
4.274
4.256
4.238
4.221
2.723
2.705
2.686
1.652
1.634
1.297
1.279
1.238
0.889
0.873
0.855
^{1}H NMR (400 MHz, CDCl$_3$)
H$_3$C
O
N
O
O
S
O
CH$_3$
Cl
(4v)
4.00
2.00
2.05
1.99
2.01
27.02
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

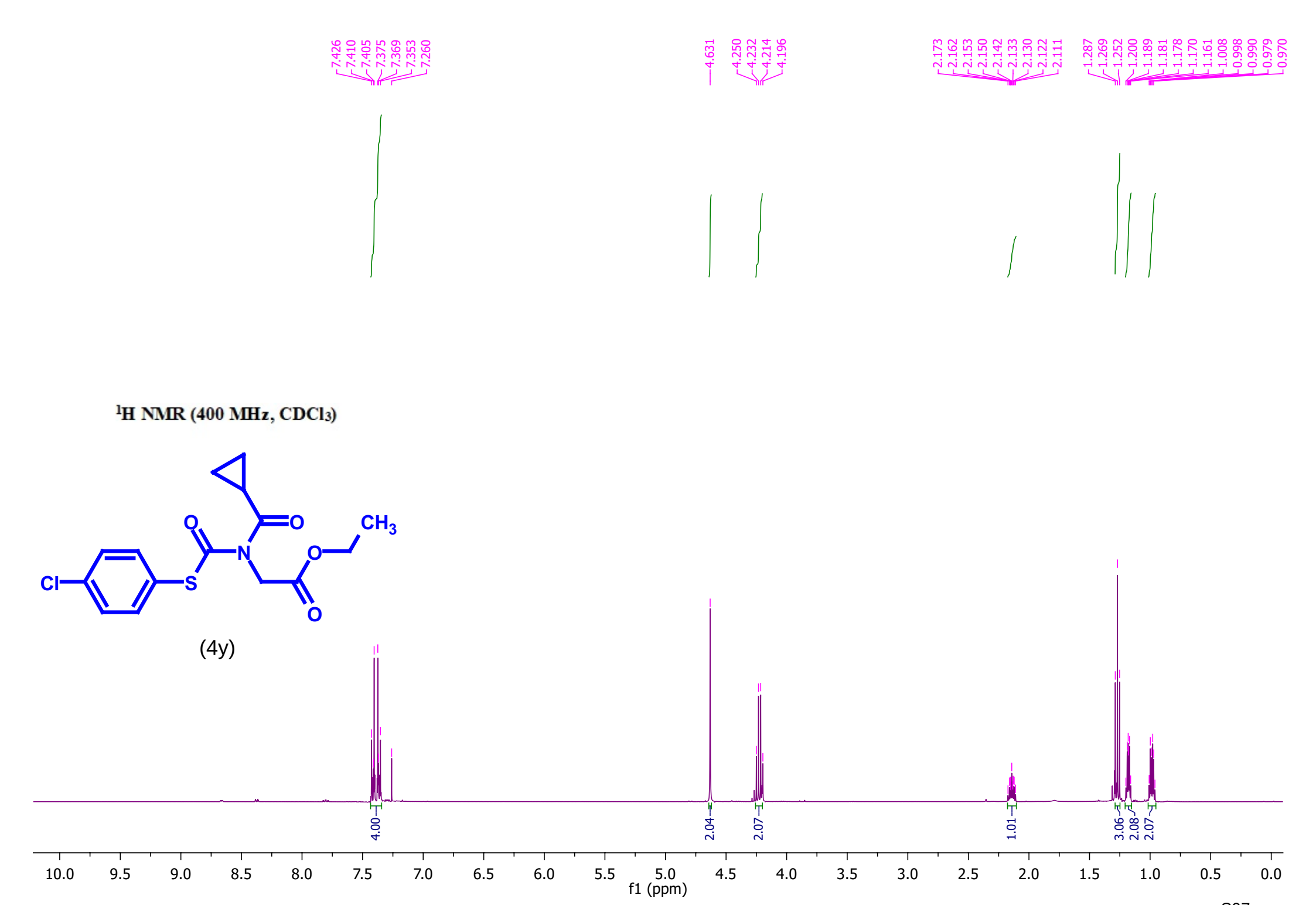

174.70
169.86
167.74
136.69
136.52
129.52
126.12
77.32
77.00
76.68
61.90
46.02
37.50
31.89
29.67
29.57
29.42
29.33
28.94
24.26
22.67
14.12
14.10
13C NMR (100 MHz, CDCl3)
H3C
O
N
O
S
O
O
CH3
Cl
(4v)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

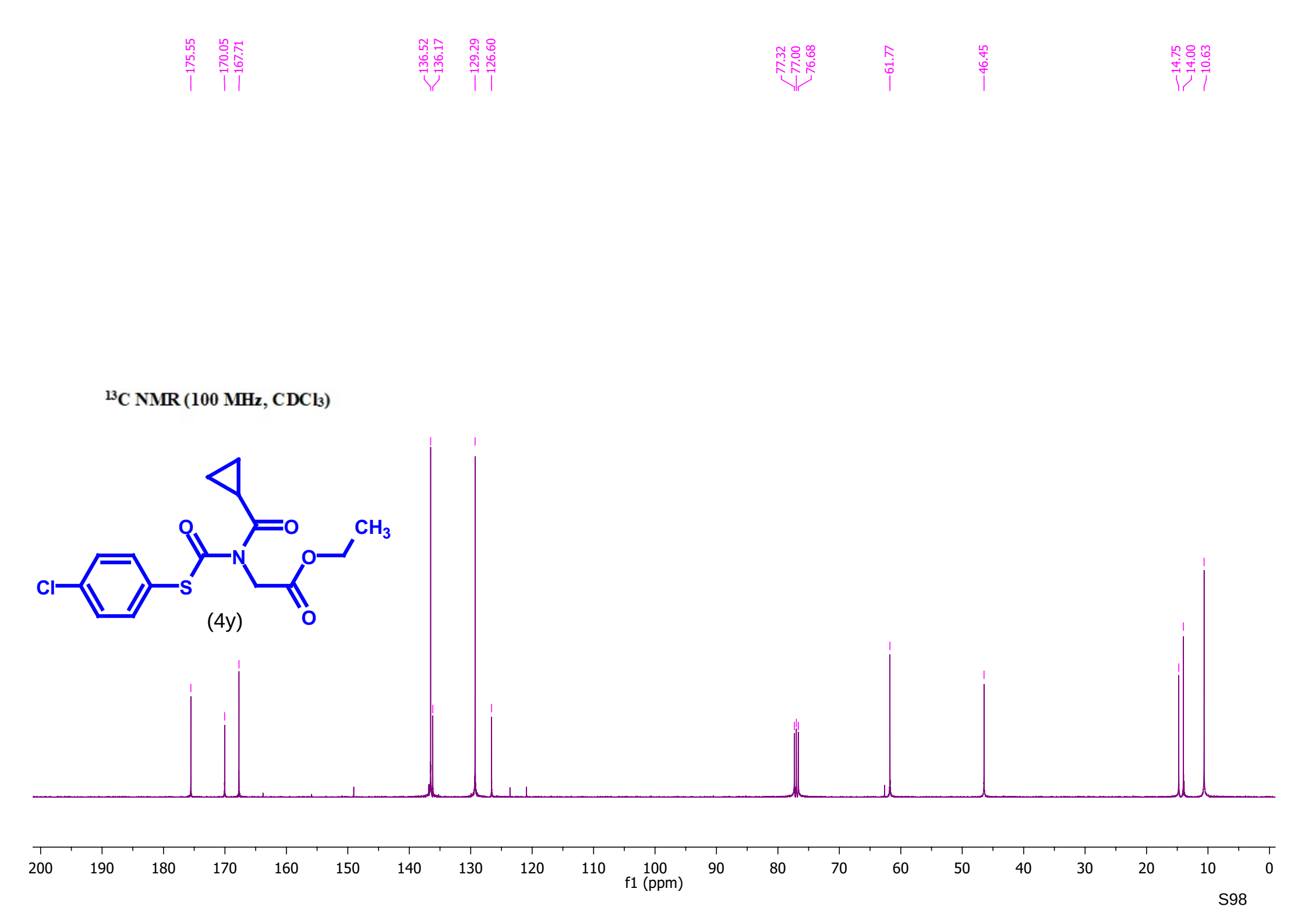

7.417
7.416
7.395
7.391
7.378
7.372
7.356
7.260
4.550
4.251
4.233
4.215
4.197
3.000
2.992
2.979
2.971
2.963
2.951
2.943
2.935
1.883
1.849
1.771
1.765
1.757
1.740
1.733
1.655
1.652
1.641
1.475
1.445
1.292
1.274
1.262
1.261
1.260
1.256
1.237
1.231
1.208
¹H NMR (400 MHz, CDCl₃)
Cl
S
N
O
O
O
O
O
CH₃
(4w)
4.03
2.00
2.11
1.00
4.03
1.03
2.10
6.05
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)

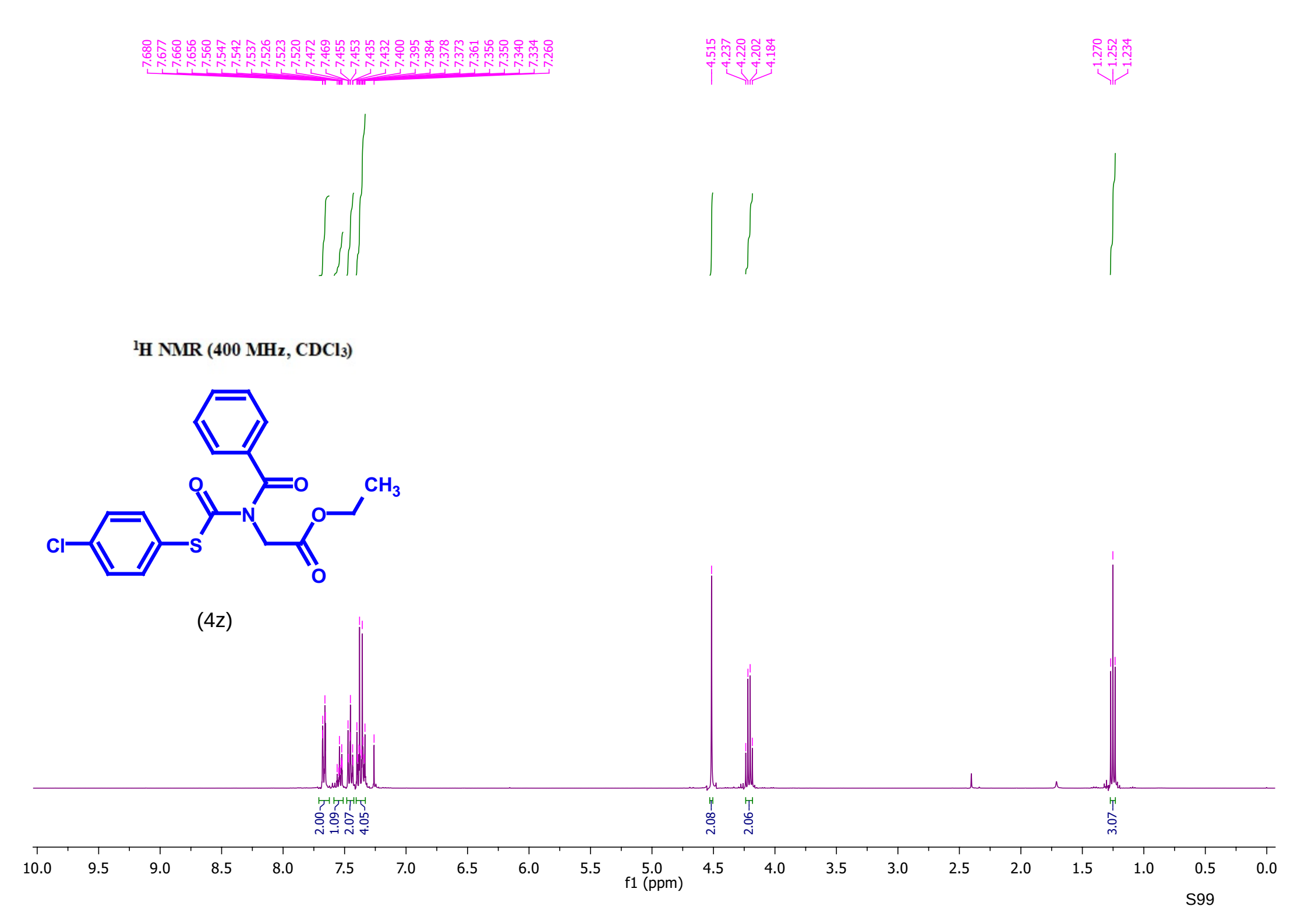

177.77
169.56
167.69
136.52
136.28
129.36
126.03
77.32
77.00
76.68
61.68
46.08
44.36
29.11
25.50
25.27
13.96
13C NMR (100 MHz, CDCl3)
O
O
CH3
N
O
Cl
S
(4w)
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

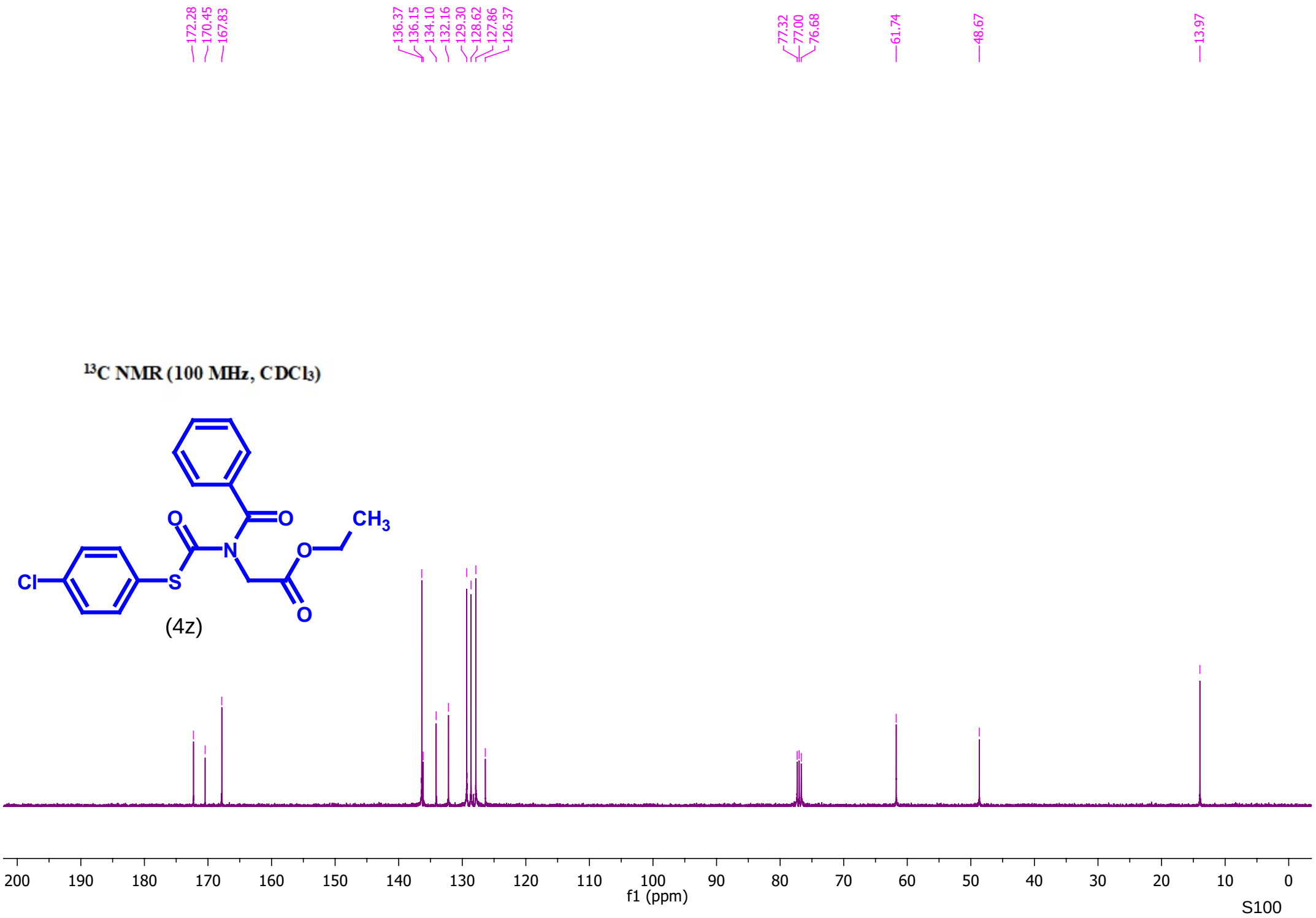

7.432
7.431
7.427
7.416
7.410
7.405
7.389
7.383
7.378
7.367
7.363
7.362
7.260
4.531
4.256
4.238
4.220
4.202
2.042
1.700
1.302
1.284
1.266
¹H NMR (400 MHz, CDCl₃)
Cl
S
N
O
O
O
O
O
CH₃
(4x)
4.03
2.00
2.05
9.00
6.01
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

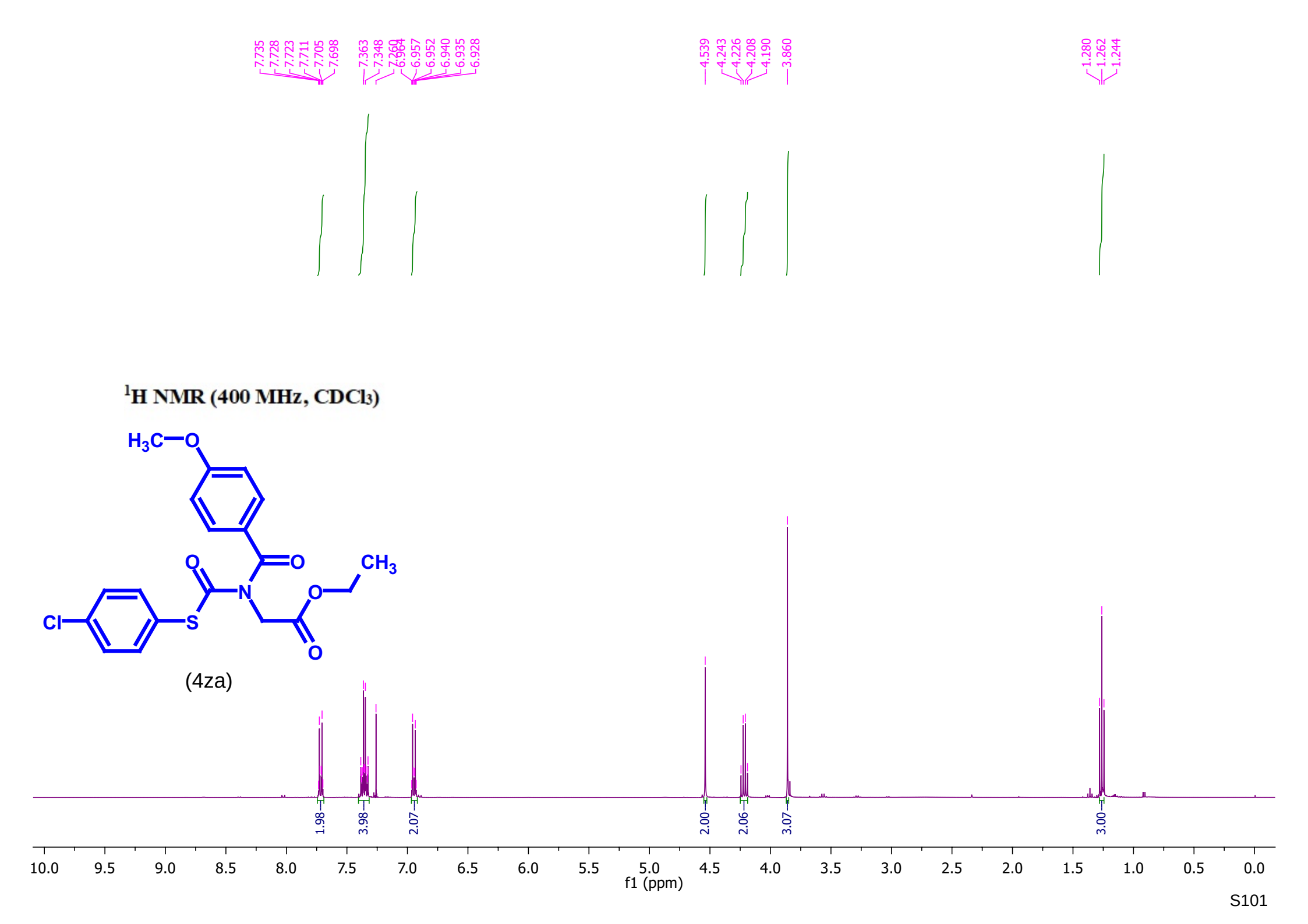

183.43
170.60
168.02
136.41
136.20
129.43
126.41
77.32
77.00
76.68
61.74
48.51
45.45
38.40
36.19
28.07
14.08
^{13}C NMR (100 MHz, CDCl$_3$)
Cl
S
N
O
O
O
O
O
CH$_3$
(4x)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

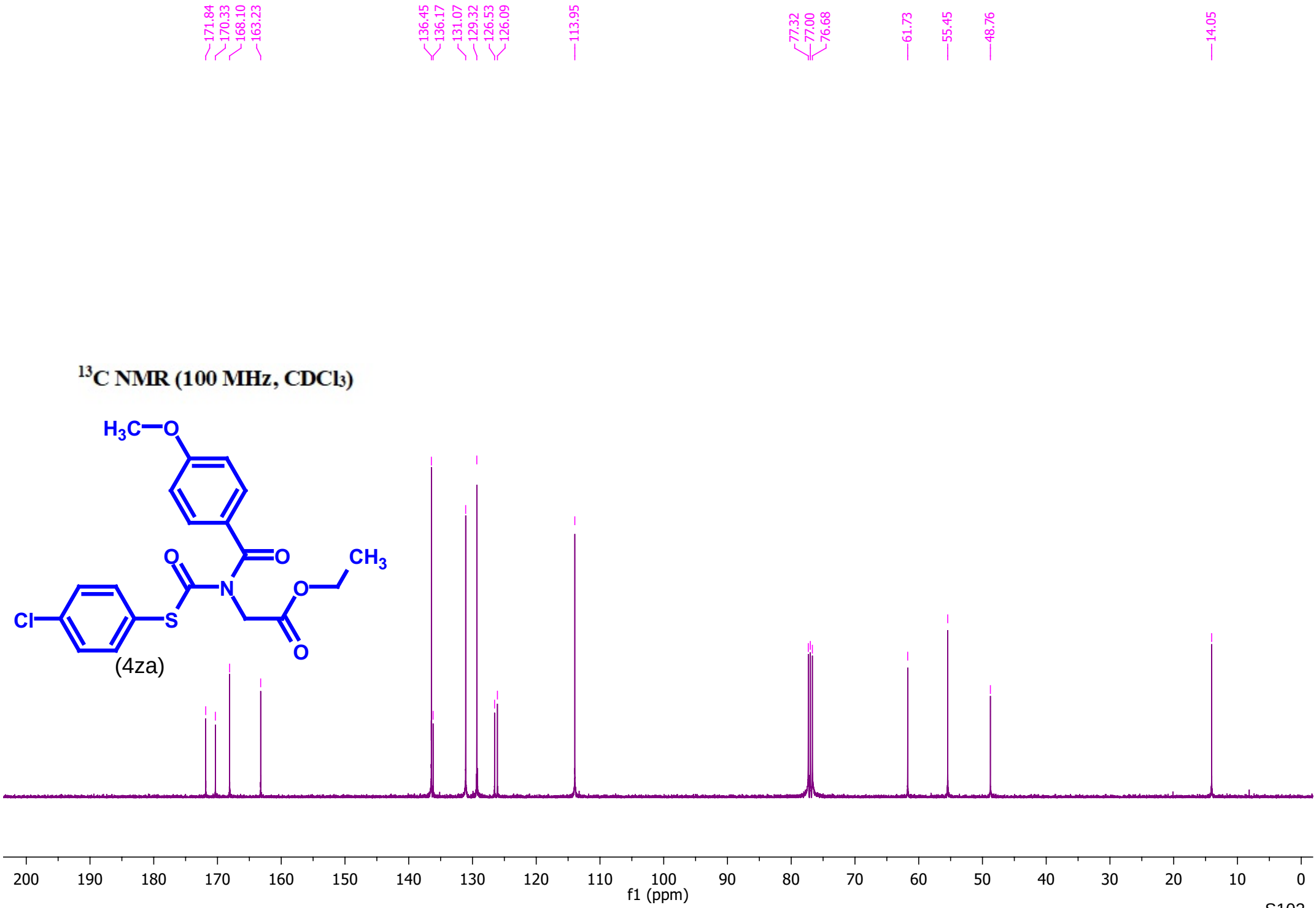

7.426
7.410
7.405
7.375
7.369
7.353
7.260
4.631
4.250
4.232
4.214
4.196
2.173
2.162
2.153
2.150
2.142
2.133
2.130
2.122
2.111
1.287
1.269
1.252
1.200
1.189
1.181
1.178
1.170
1.161
1.008
0.998
0.990
0.979
0.970
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
S
N
O
O
O
O
O
CH$_3$
(4y)
4.00
2.04
2.07
1.01
3.06
2.08
2.07
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
f1 (ppm)


175.55
170.05
167.71
136.52
136.17
129.29
126.60
77.32
77.00
76.68
61.77
46.45
14.75
14.00
10.63
13C NMR (100 MHz, CDCl3)
Cl
S
N
O
O
O
O
CH3
(4y)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

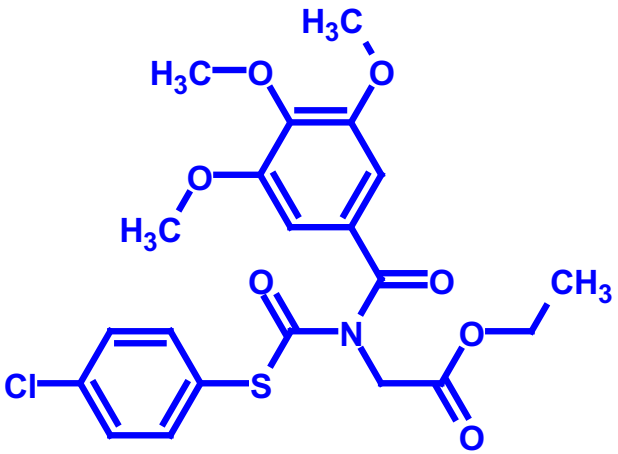

7.680
7.677
7.660
7.656
7.560
7.547
7.542
7.537
7.526
7.523
7.520
7.472
7.469
7.455
7.453
7.435
7.432
7.400
7.395
7.384
7.378
7.373
7.361
7.356
7.350
7.340
7.334
7.260
4.515
4.237
4.220
4.202
4.184
1.270
1.252
1.234
1H NMR (400 MHz, CDCl3)
O
O
CH3
N
O
Cl
S
O
(4z)
2.00
1.09
2.07
4.05
2.08
2.06
3.07
10.0
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
f1 (ppm)


172.28
170.45
167.83
136.37
136.15
134.10
132.16
129.30
128.62
127.86
126.37
77.32
77.00
76.68
61.74
48.67
13.97
^{13}C NMR (100 MHz, CDCl$_3$)
Cl
S
N
O
O
O
O
O
CH$_3$
(4z)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

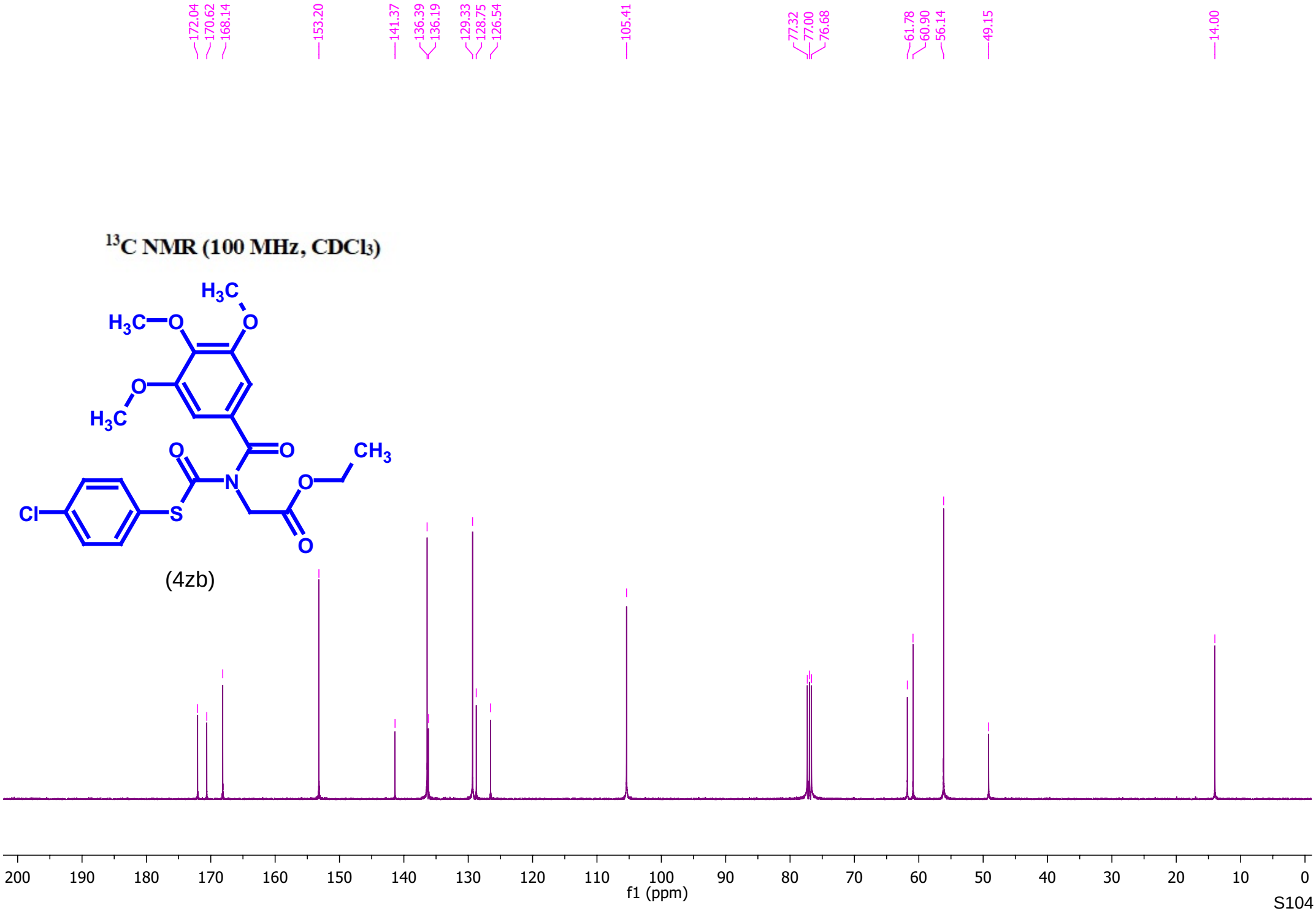

7.735
7.728
7.723
7.711
7.705
7.698
7.363
7.348
7.260
6.964
6.957
6.952
6.940
6.935
6.928
4.539
4.243
4.226
4.208
4.190
3.860
1.280
1.262
1.244
1H NMR (400 MHz, CDCl3)
H3C
O
O
O
CH3
O
N
O
Cl
S
O
(4za)
1.98
3.98
2.07
2.00
2.06
3.07
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

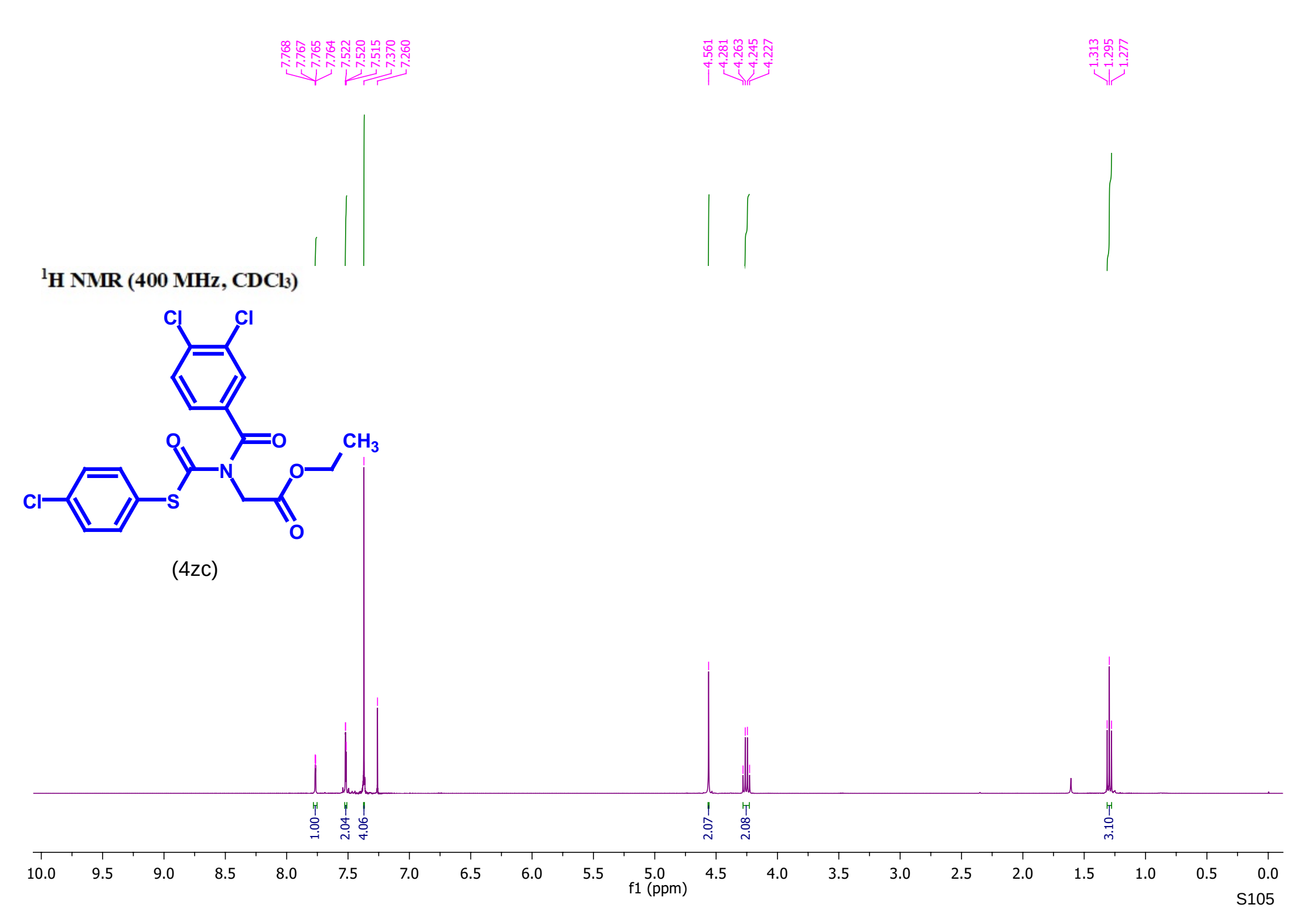

171.84
170.33
168.10
163.23
136.45
136.17
131.07
129.32
126.53
126.09
113.95
77.32
77.00
76.68
61.73
55.45
48.76
14.05
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
O
O
O
CH$_3$
N
O
Cl
S
O
(4za)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{1}H NMR (400 MHz, $CDCl_3$)

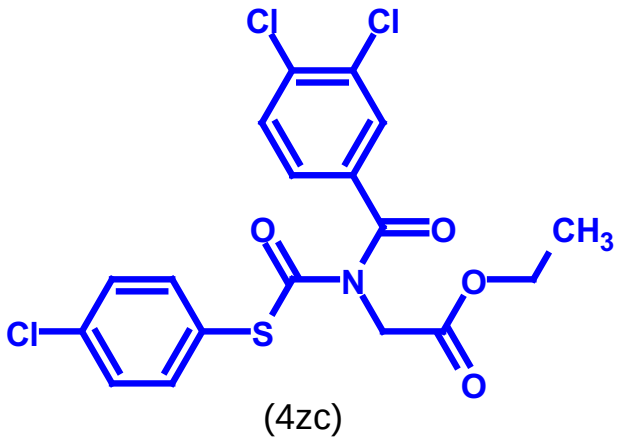


(4zb)

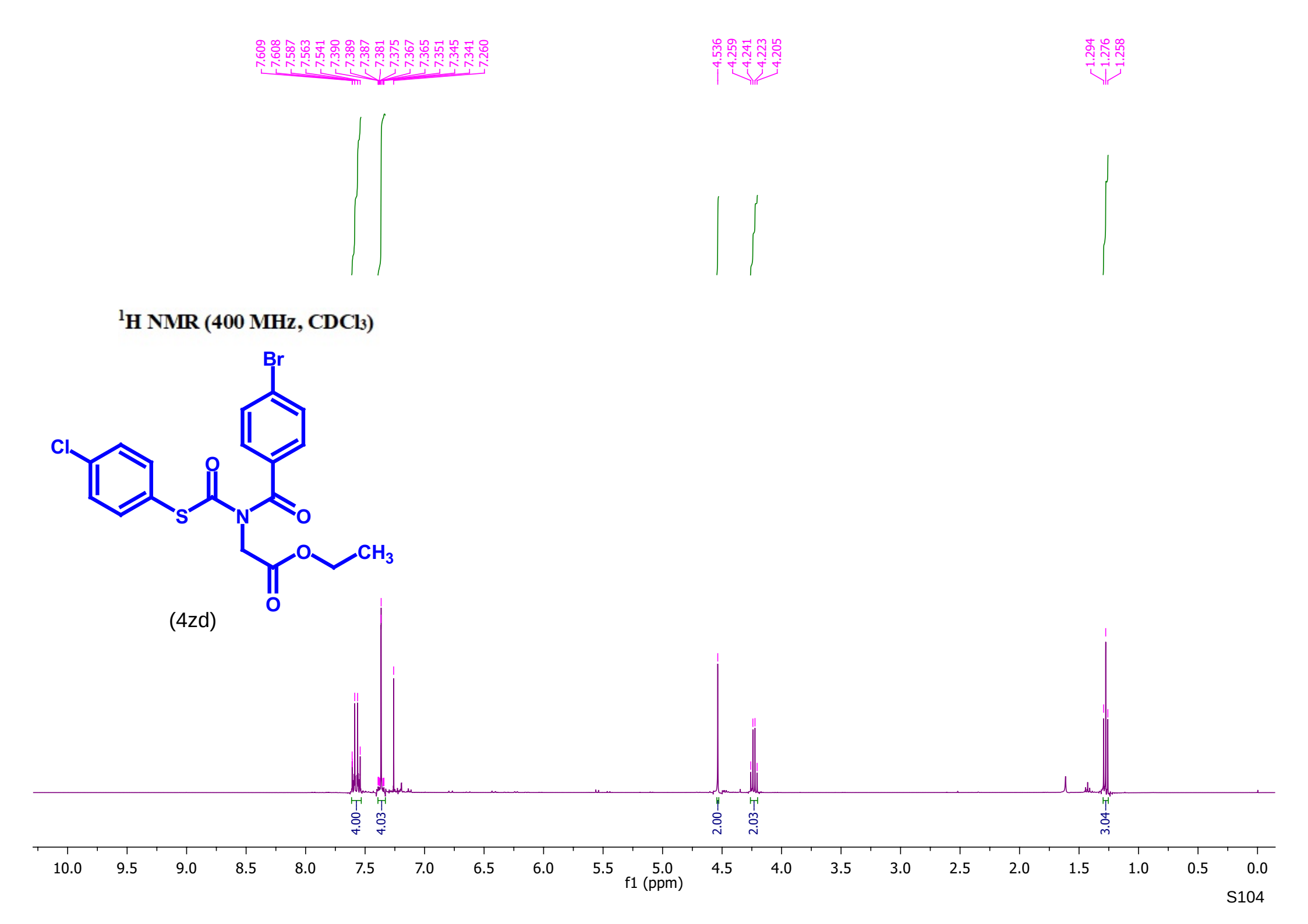

172.04
170.62
168.14
153.20
141.37
136.39
136.19
129.33
128.75
126.54
105.41
77.32
77.00
76.68
61.78
60.90
56.14
49.15
14.00
^{13}C NMR (100 MHz, CDCl$_3$)
(4zb)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

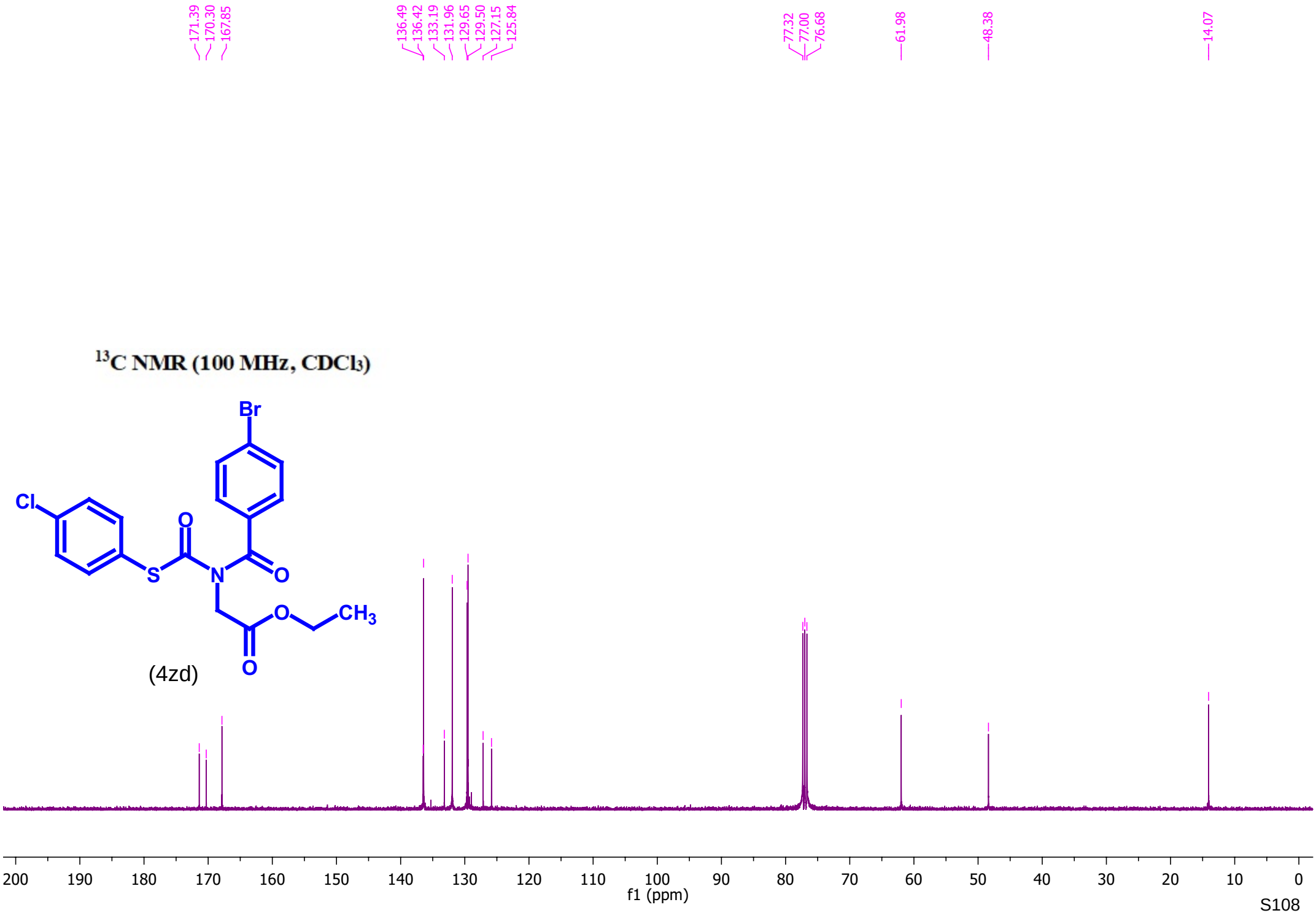

7.768
7.767
7.765
7.764
7.522
7.520
7.515
7.370
7.260
4.561
4.281
4.263
4.245
4.227
1.313
1.295
1.277
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
Cl
O
O
CH$_3$
N
O
Cl
S
O
(4zc)
1.00
2.04
4.06
2.07
2.08
3.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

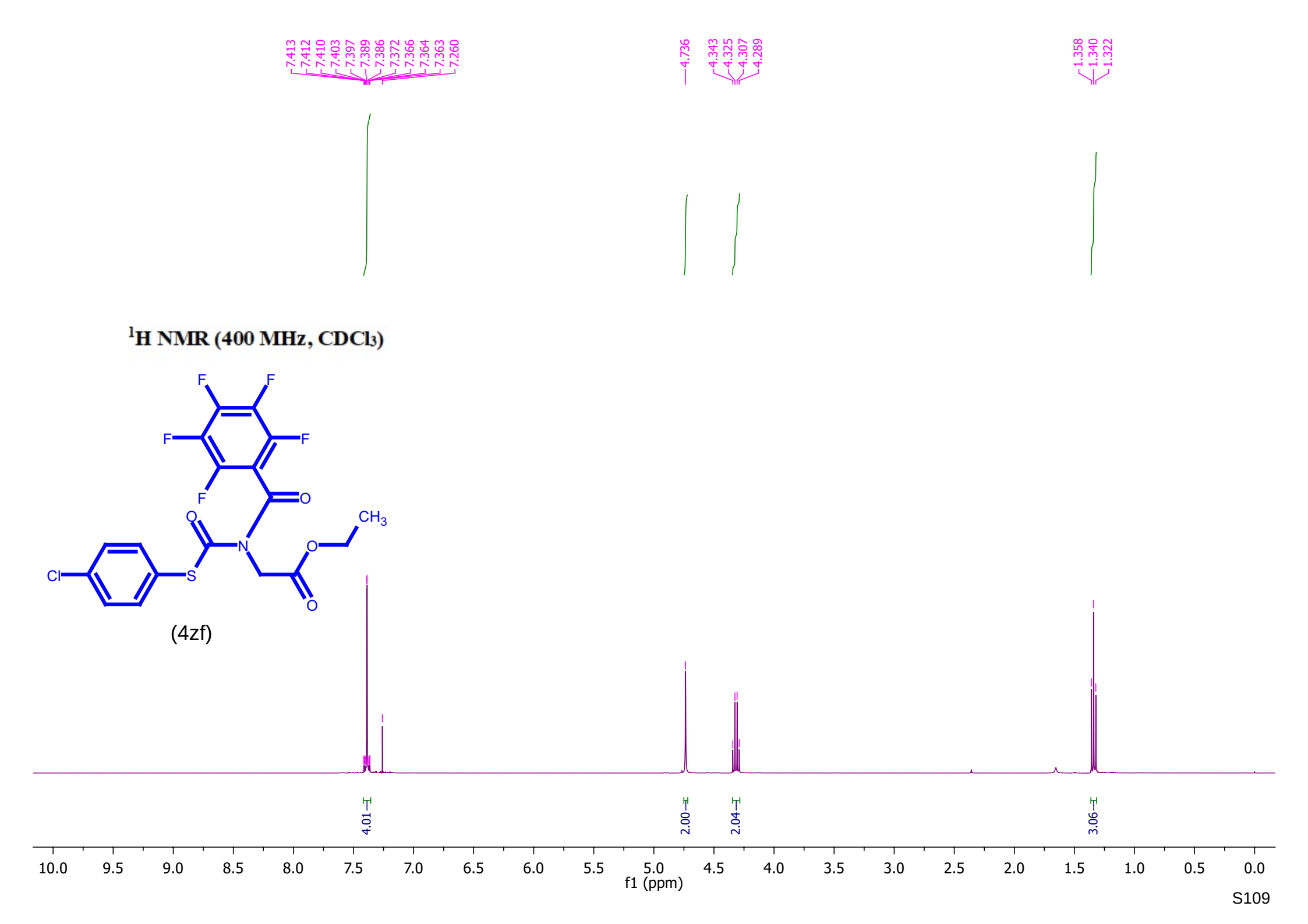
^{13}C NMR (100 MHz, $CDCl_3$)

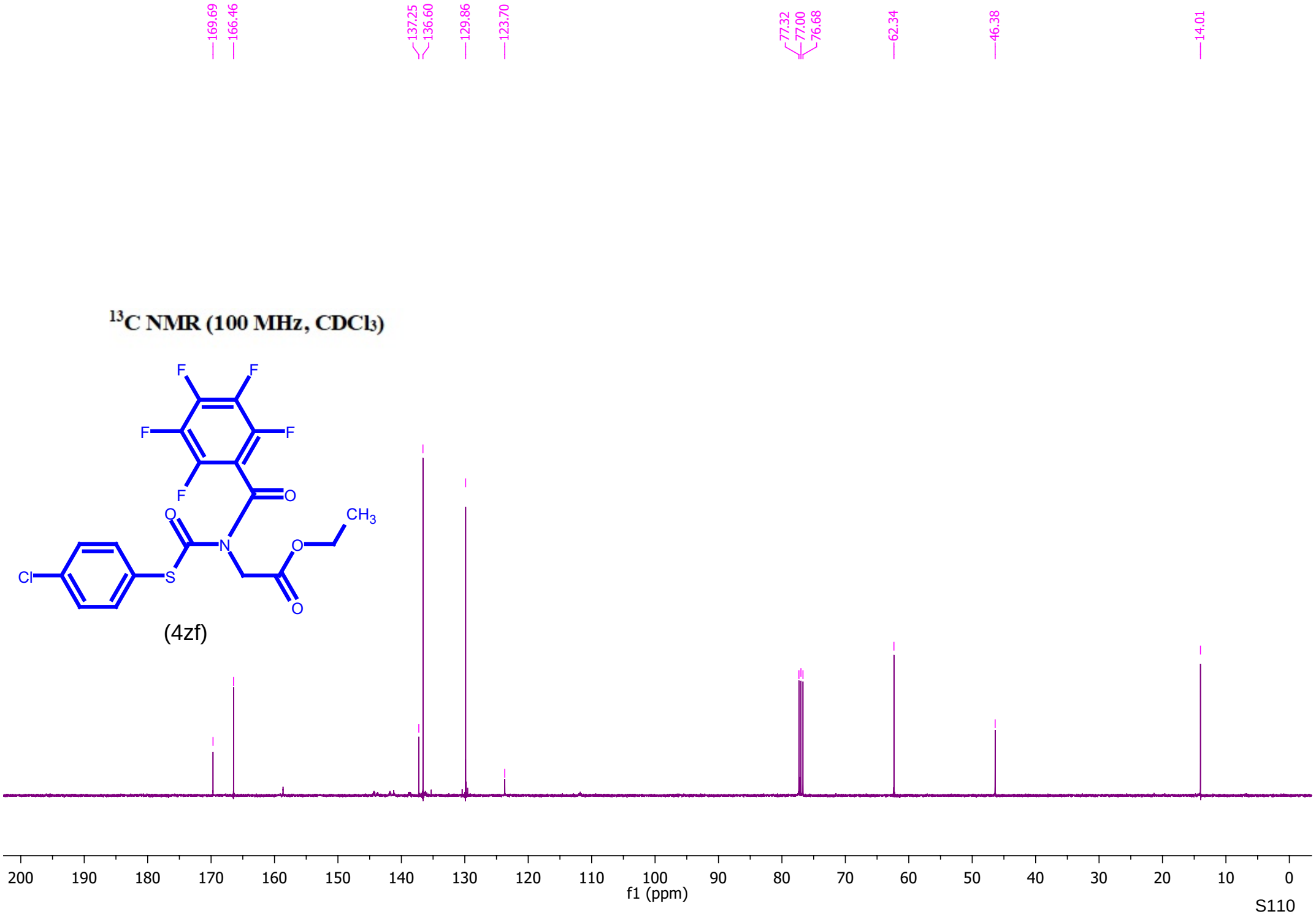


(4zc)

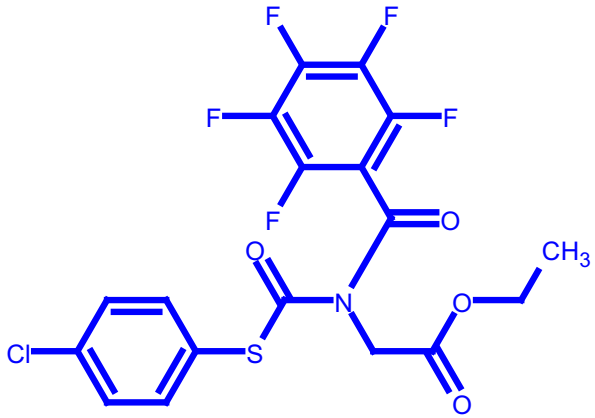

7.609
7.608
7.587
7.563
7.541
7.390
7.389
7.387
7.381
7.375
7.367
7.365
7.351
7.345
7.341
7.260
4.536
4.259
4.241
4.223
4.205
1.294
1.276
1.258
^{1}H NMR (400 MHz, CDCl$_3$)
Br
Cl
O
S
N
O
O
O
CH$_3$
O
(4zd)
4.00
4.03
2.00
2.03
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


171.39
170.30
167.85
136.49
136.42
133.19
131.96
129.65
129.50
127.15
125.84
77.32
77.00
76.68
61.98
48.38
14.07
^{13}C NMR (100 MHz, CDCl$_3$)
Br
Cl
O
S
N
O
O
O
CH$_3$
O
(4zd)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

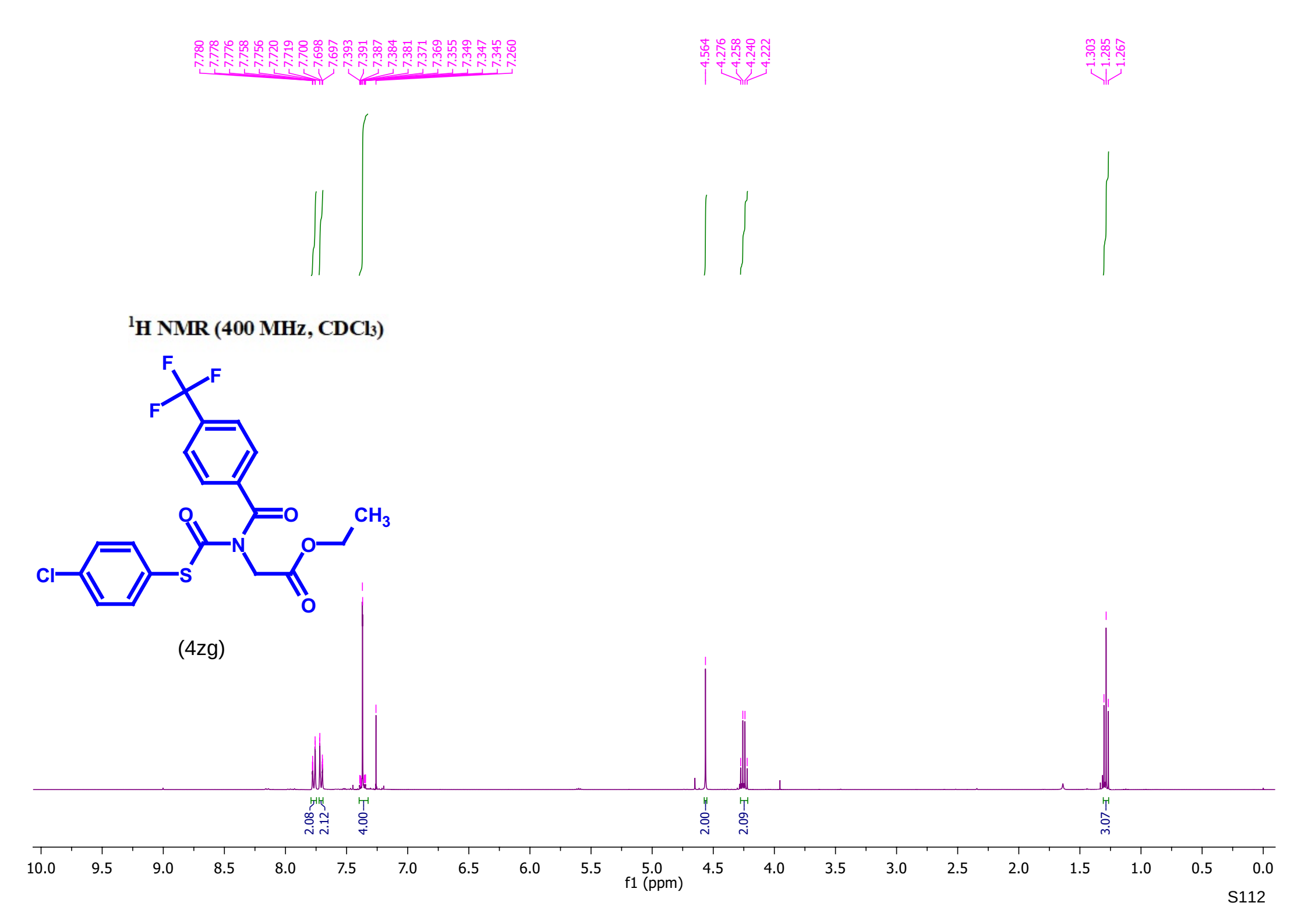

7.413
7.412
7.410
7.403
7.397
7.389
7.386
7.372
7.366
7.364
7.363
7.260
4.736
4.343
4.325
4.307
4.289
1.358
1.340
1.322
1H NMR (400 MHz, CDCl3)
F
F
F
F
F
O
O
CH3
N
O
Cl
S
O
(4zf)
4.01
2.00
2.04
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

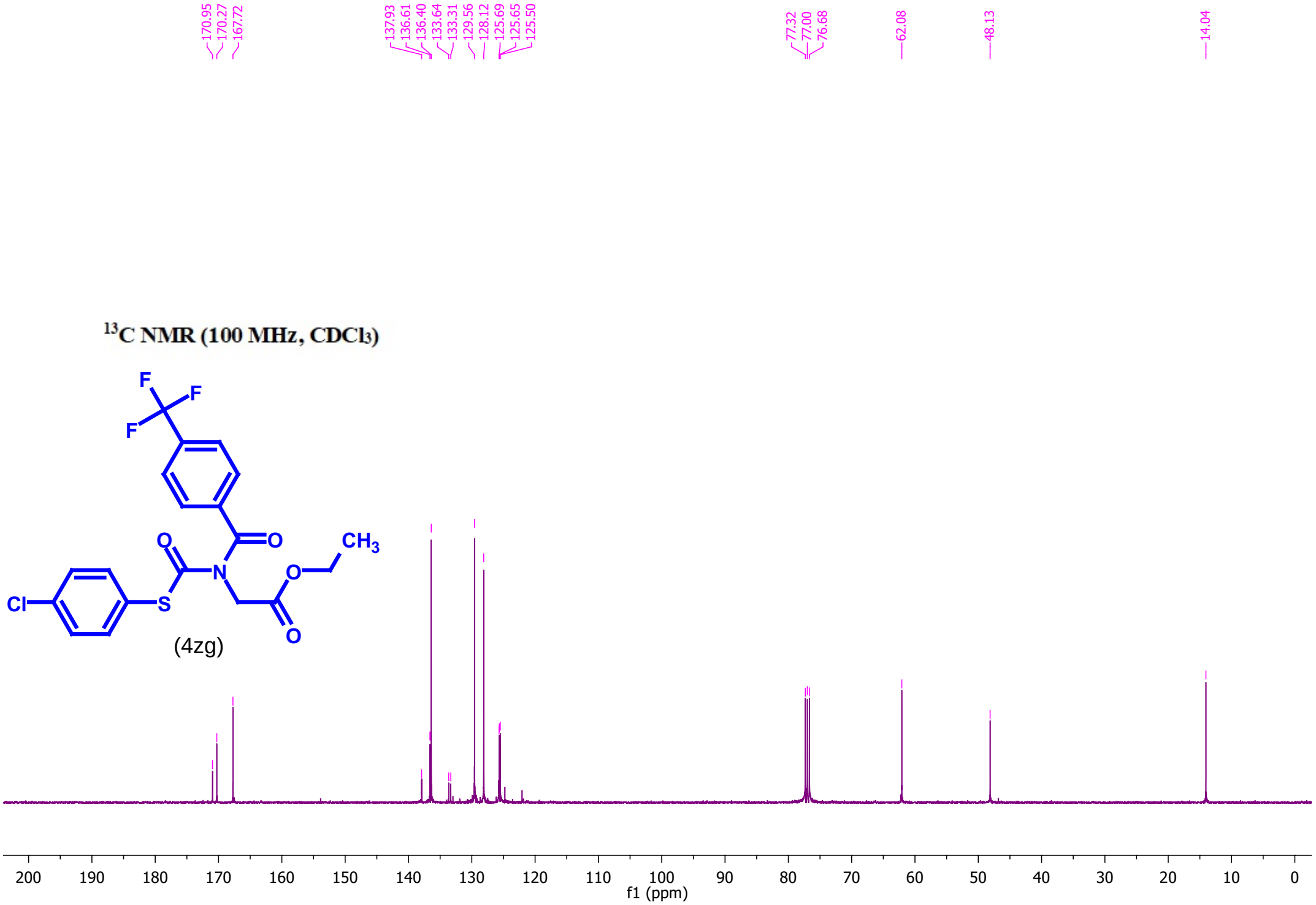

169.69
166.46
137.25
136.60
129.86
123.70
77.32
77.00
76.68
62.34
46.38
14.01
13C NMR (100 MHz, CDCl3)
F
F
F
F
F
O
O
CH3
N
O
Cl
S
O
(4zf)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{19}F NMR (376 MHz, $CDCl_3$)



(4zf)


7.780
7.778
7.776
7.758
7.756
7.720
7.719
7.700
7.698
7.697
7.393
7.391
7.387
7.384
7.381
7.371
7.369
7.355
7.349
7.347
7.345
7.260
4.564
4.276
4.258
4.240
4.222
1.303
1.285
1.267
^{1}H NMR (400 MHz, CDCl$_3$)
F
F
F
O
O
CH$_3$
N
O
Cl
S
O
(4zg)
2.08
2.12
4.00
2.00
2.09
3.07
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
f1 (ppm)


170.95
170.27
167.72
137.93
136.61
136.40
133.64
133.31
129.56
128.12
125.69
125.65
125.50
77.32
77.00
76.68
62.08
48.13
14.04
^{13}C NMR (100 MHz, CDCl$_3$)
F
F
F
O
O
CH$_3$
N
O
Cl
S
O
(4zg)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

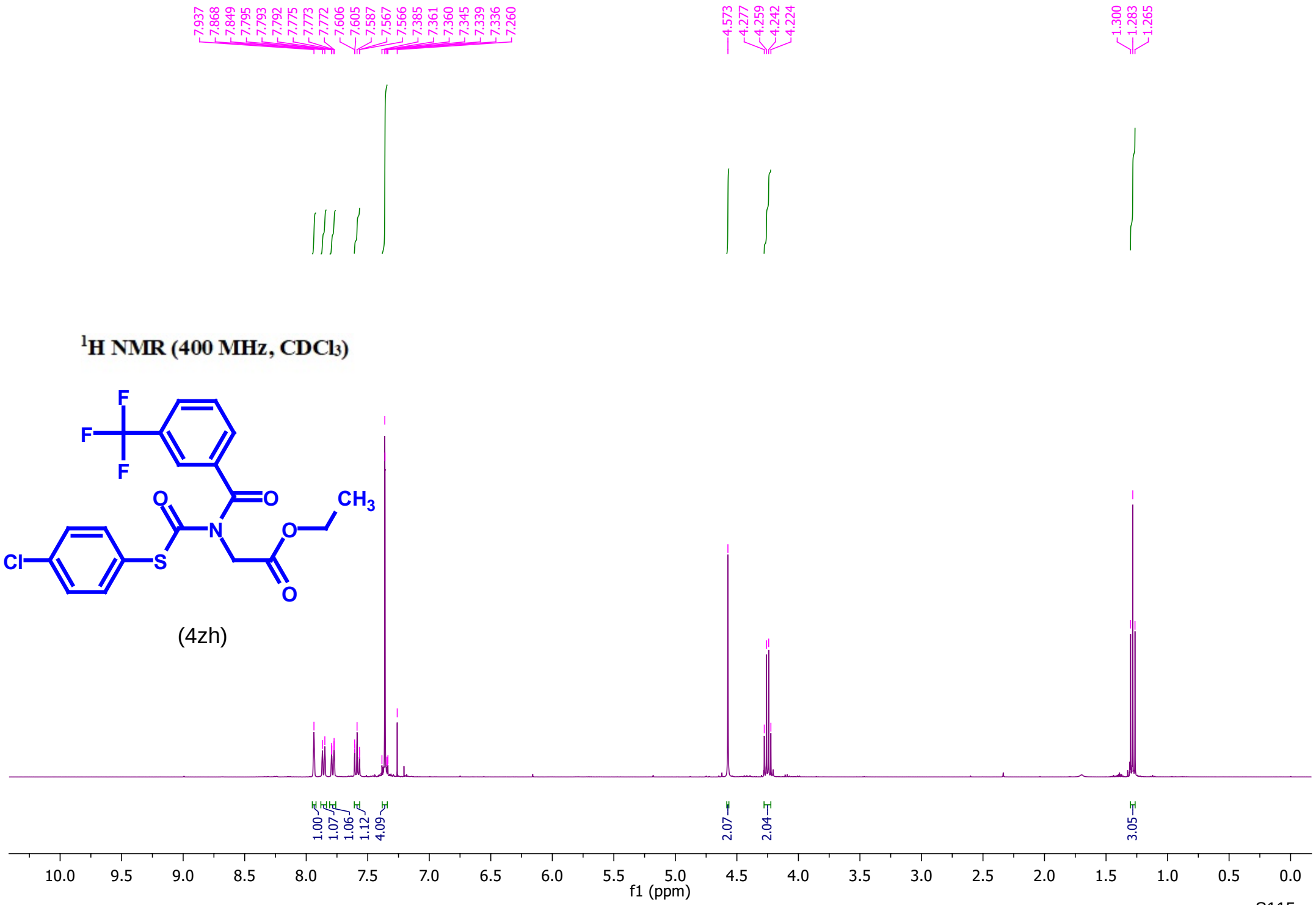
-62.97

^{19}F NMR (376 MHz, $CDCl_3$)

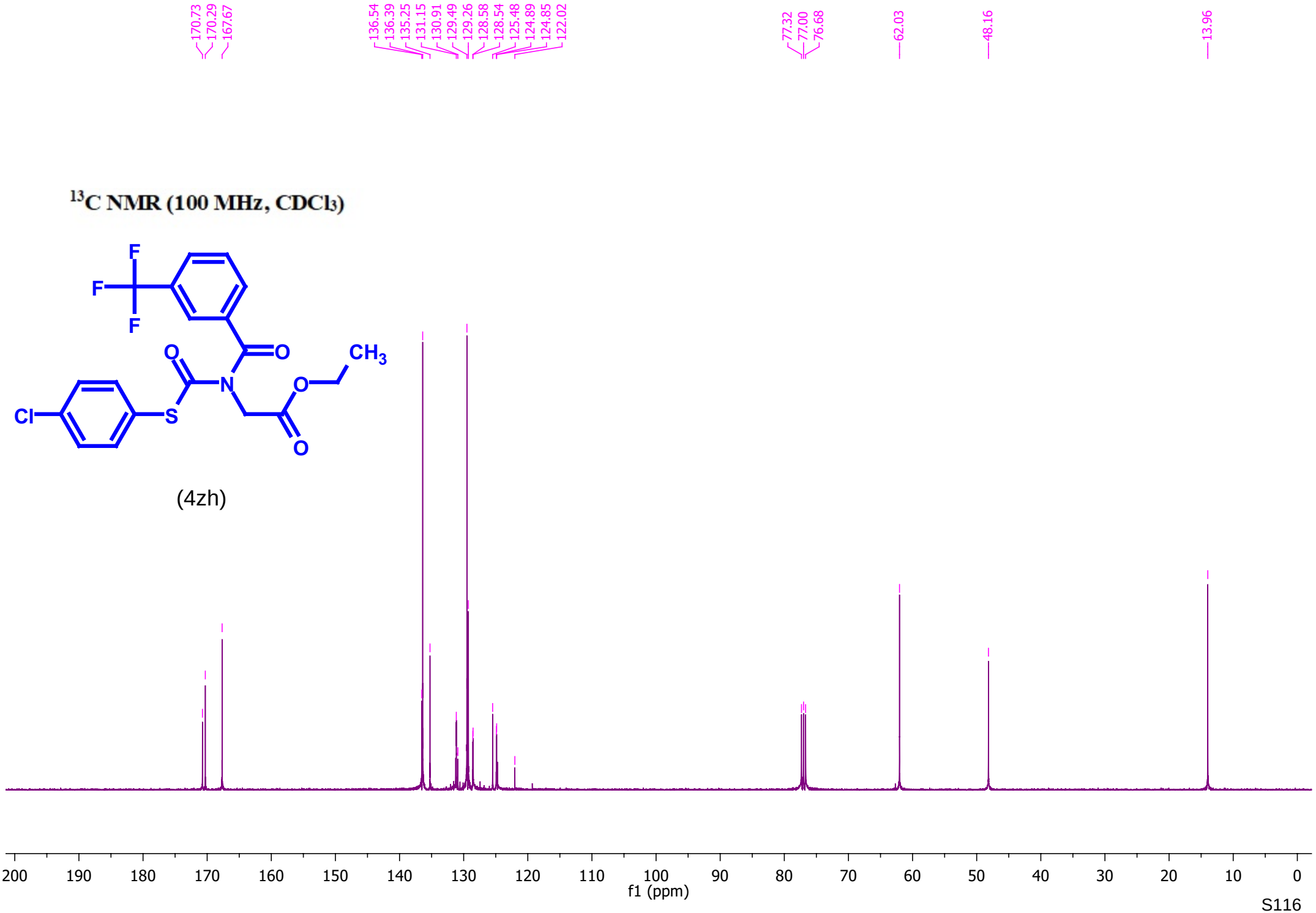
(4zg)

-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200

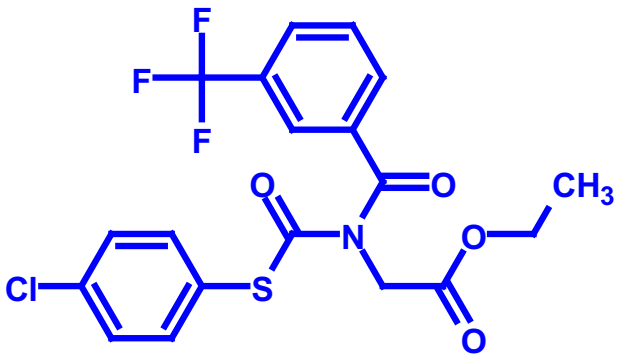
f1 (ppm)


7.937
7.868
7.849
7.795
7.793
7.792
7.775
7.773
7.772
7.606
7.605
7.587
7.567
7.566
7.385
7.361
7.360
7.345
7.339
7.336
7.260
4.573
4.277
4.259
4.242
4.224
1.300
1.283
1.265
1H NMR (400 MHz, CDCl3)
F
F
F
O
O
CH3
N
O
Cl
S
O
1.00
1.07
1.06
1.12
4.09
2.07
2.04
3.05
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)

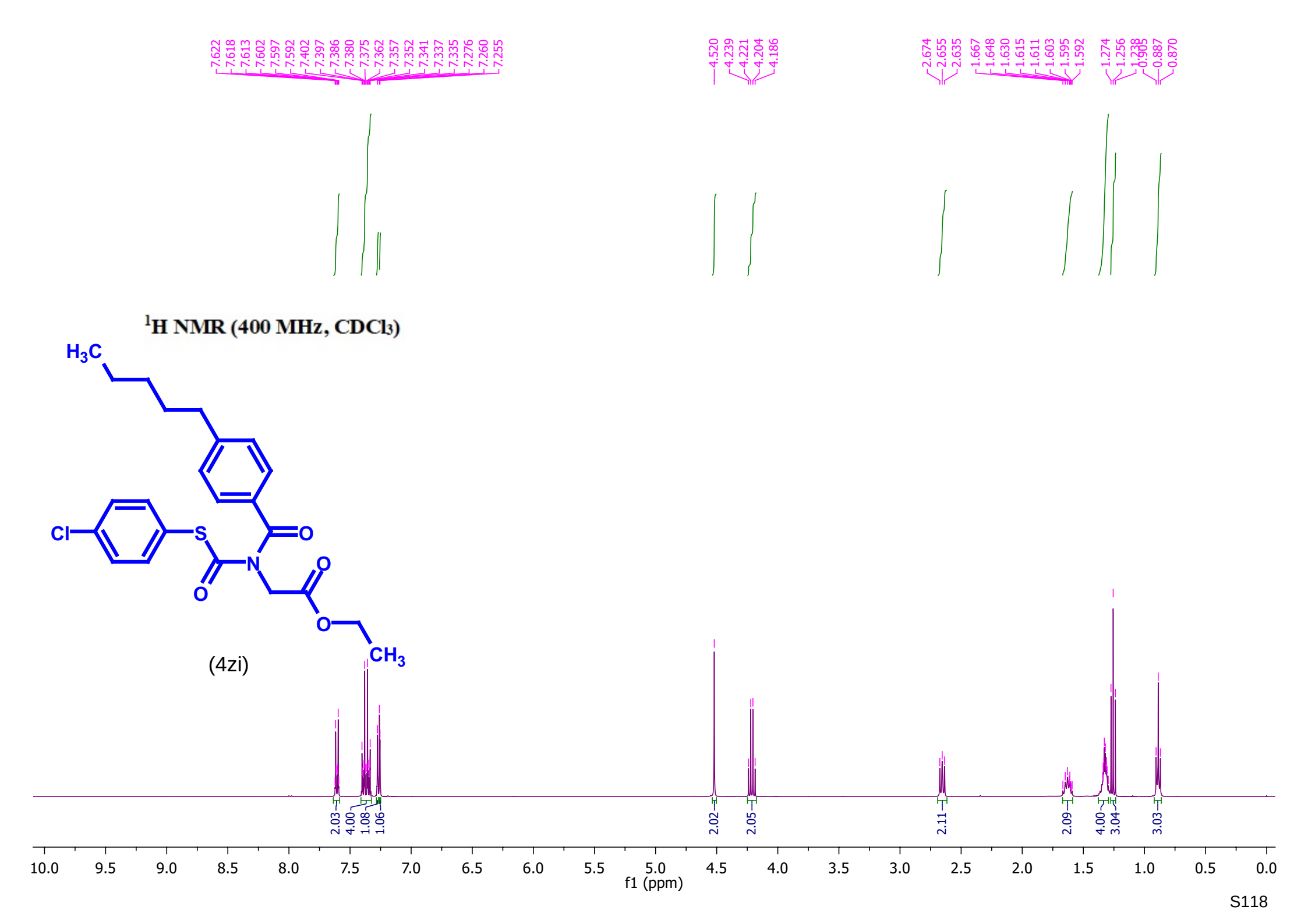
(4zh)

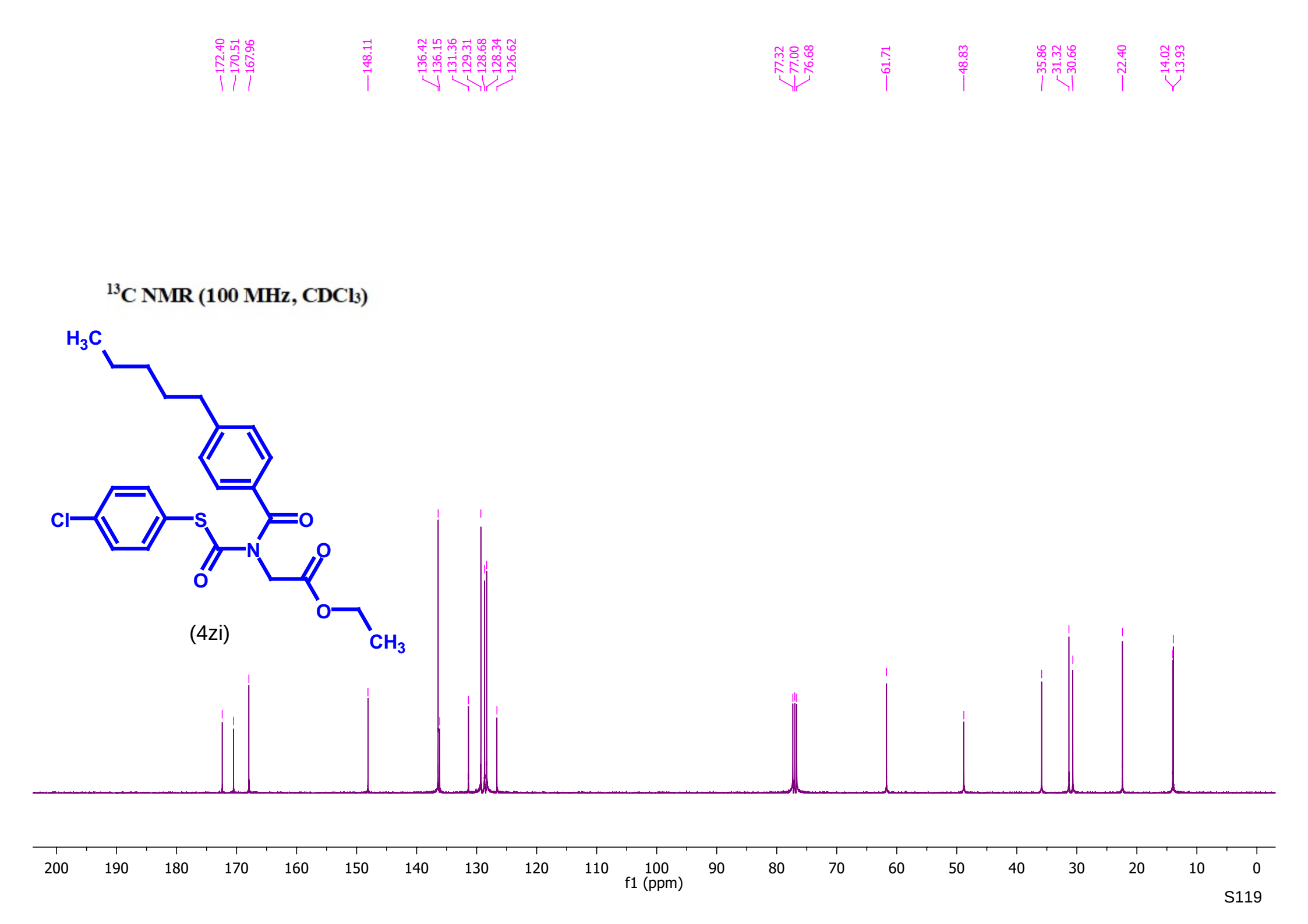

170.73
170.29
167.67
136.54
136.39
135.25
131.15
130.91
129.49
129.26
128.58
128.54
125.48
124.89
124.85
122.02
77.32
77.00
76.68
62.03
48.16
13.96
^{13}C NMR (100 MHz, CDCl$_3$)
F
F
F
O
O
CH$_3$
N
O
Cl
S
O
(4zh)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

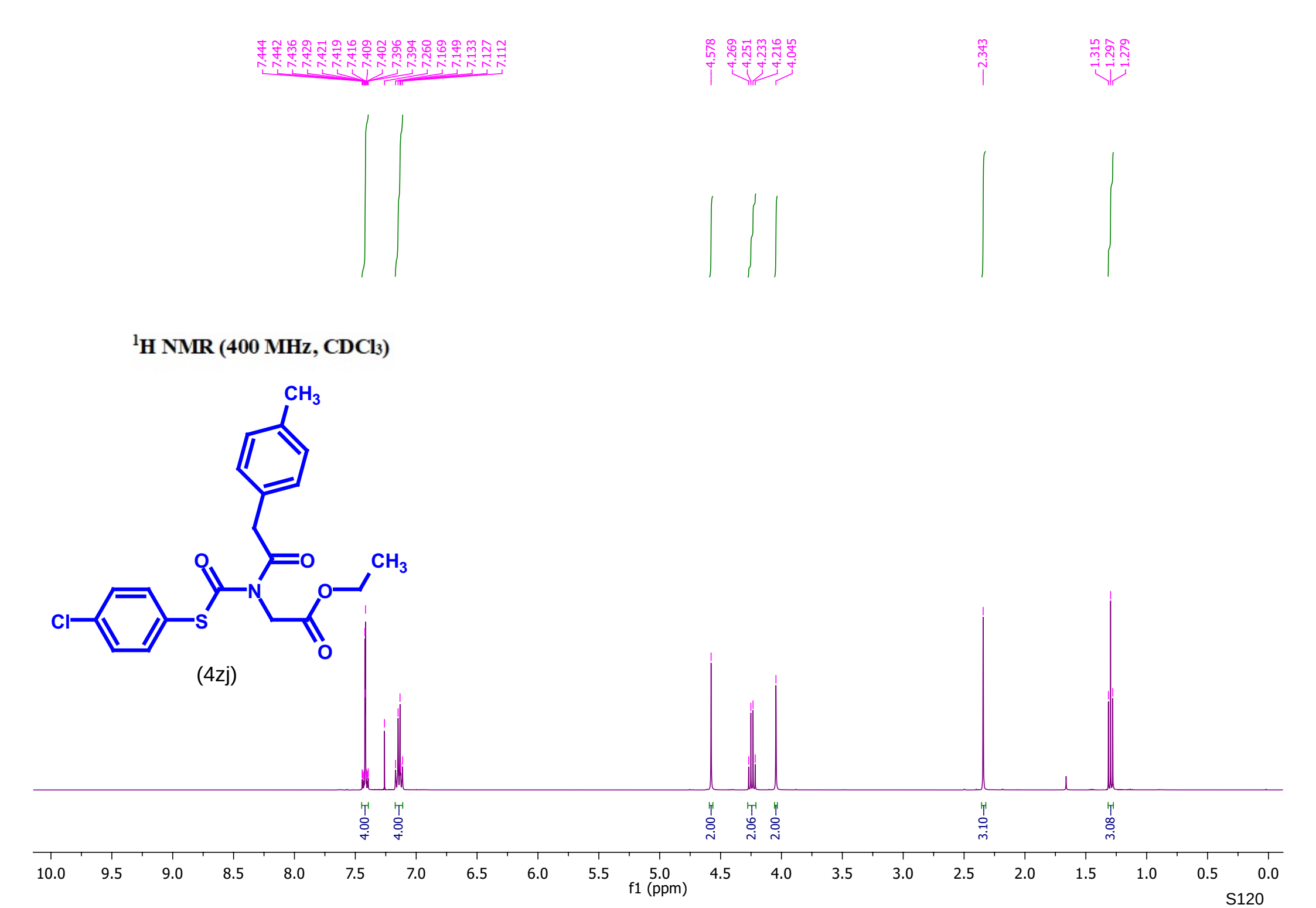
^{19}F NMR (376 MHz, $CDCl_3$)

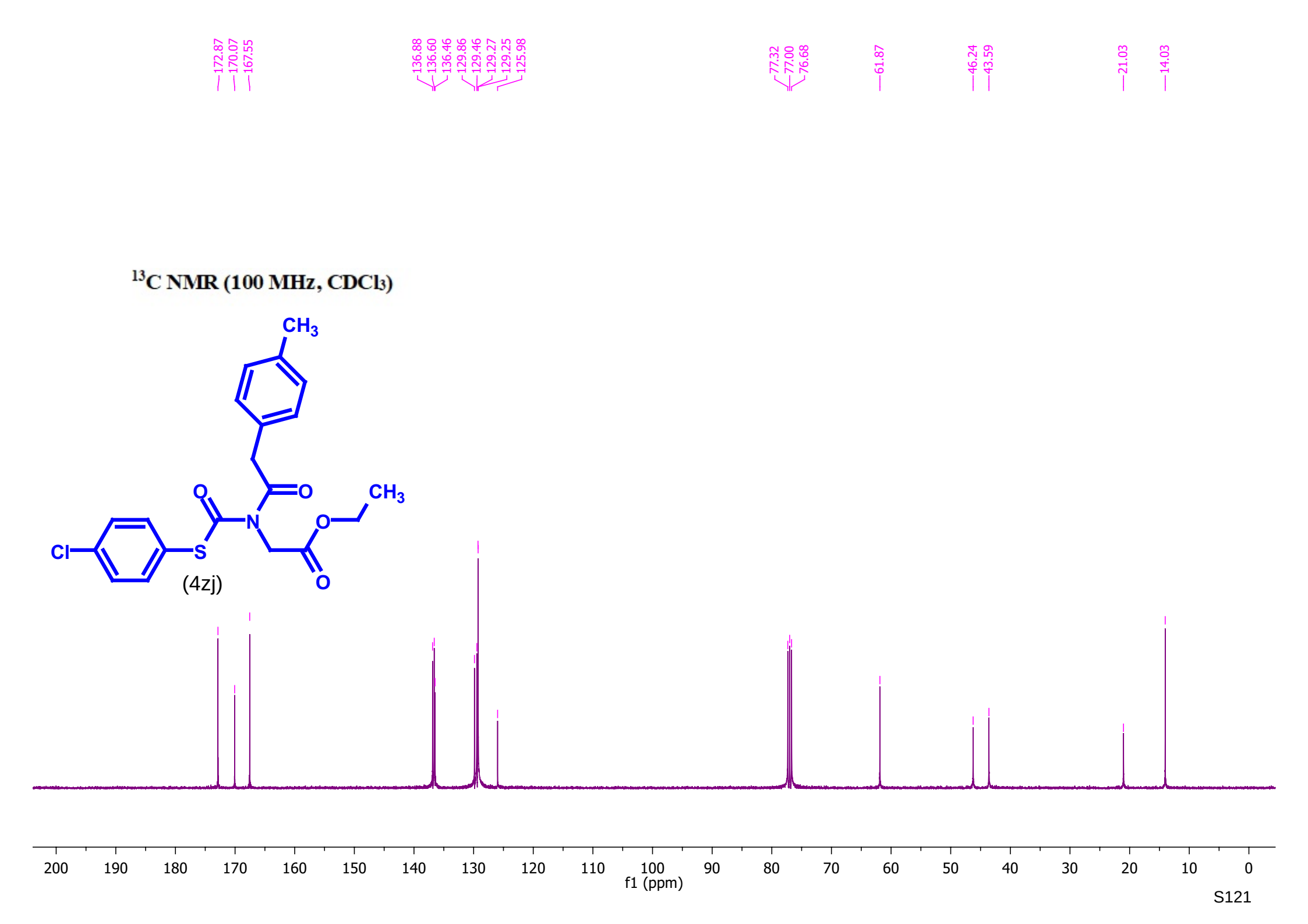


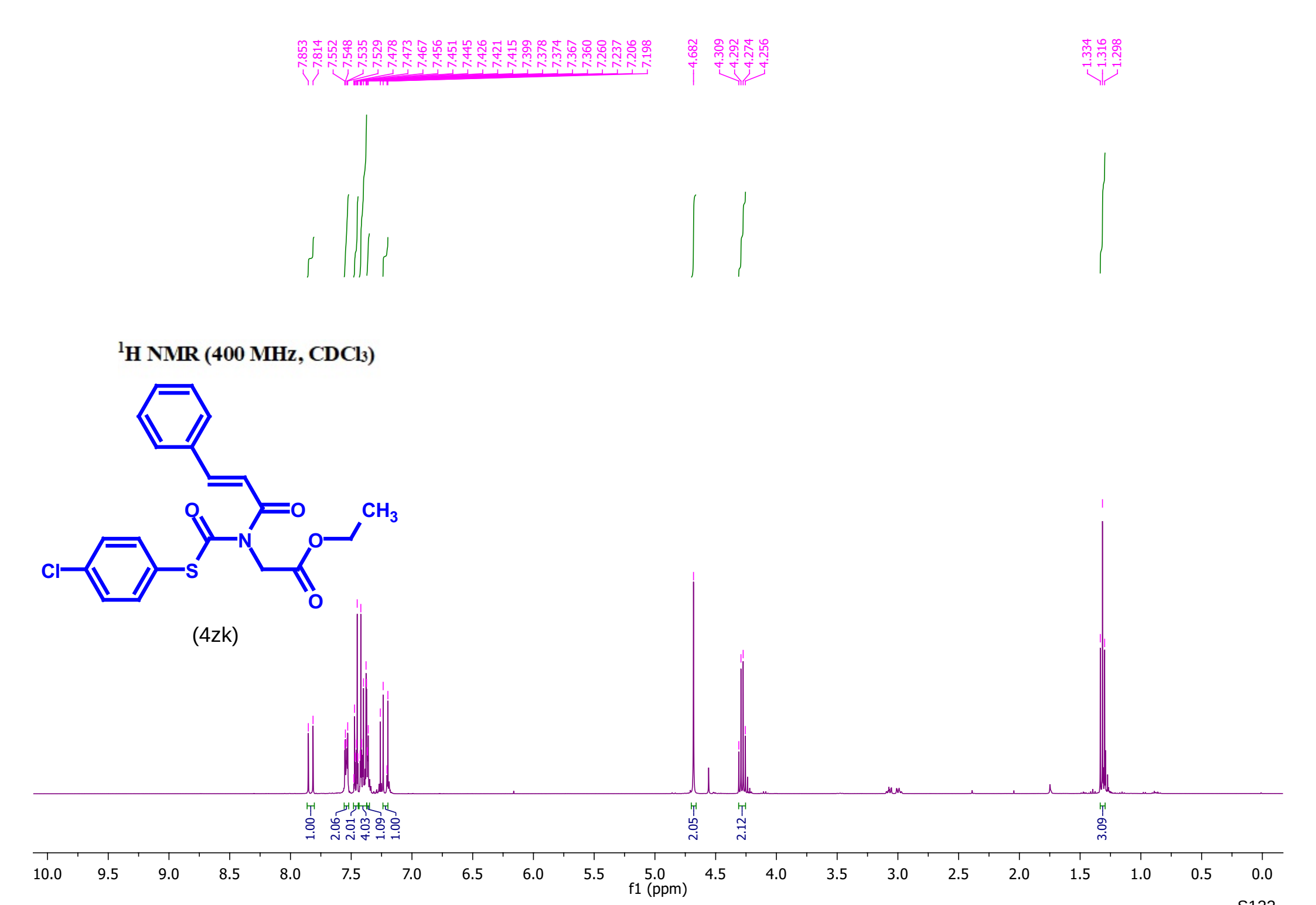
(4zh)

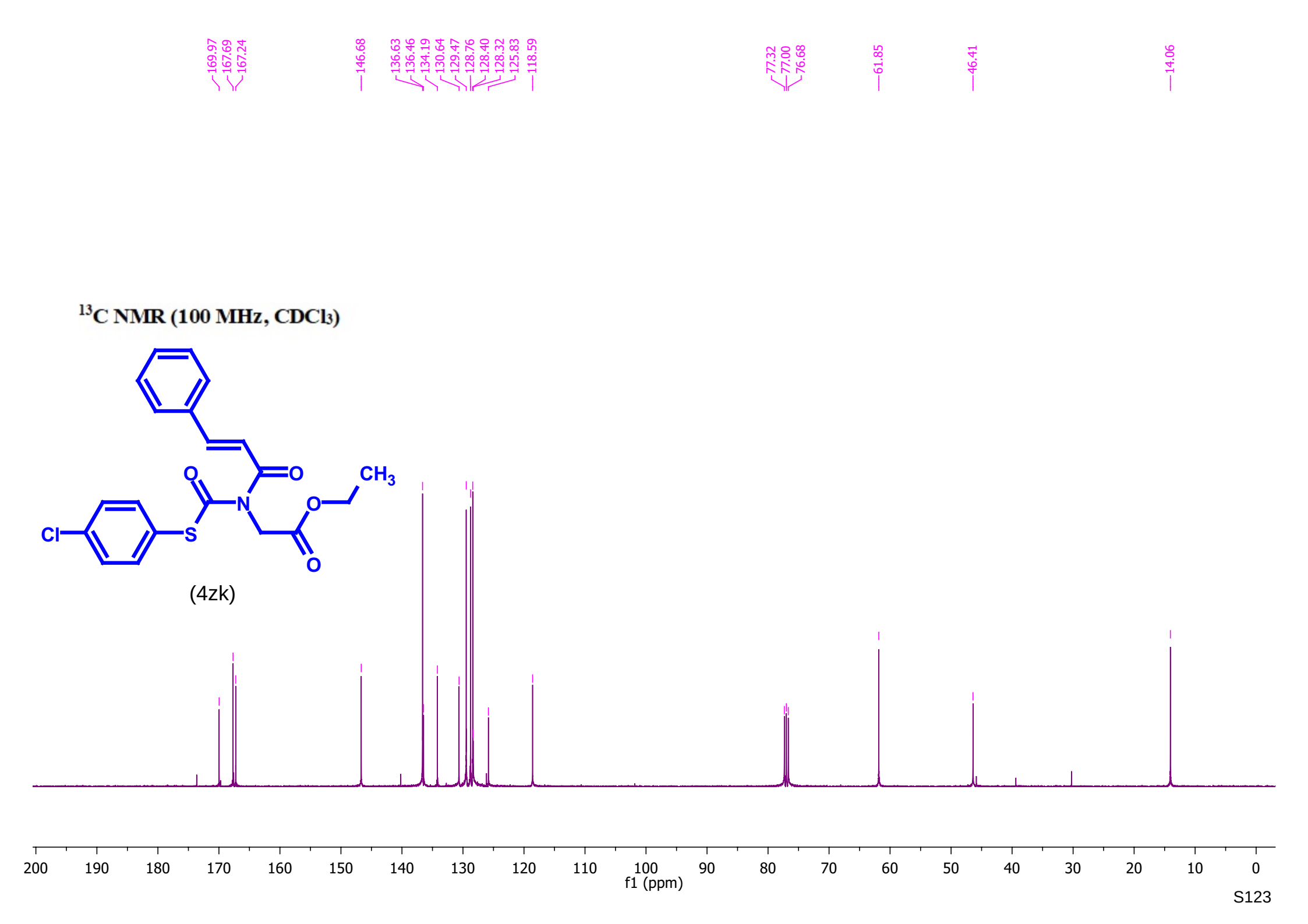

7.622
7.618
7.613
7.602
7.597
7.592
7.402
7.397
7.386
7.380
7.375
7.362
7.357
7.352
7.341
7.337
7.335
7.276
7.260
7.255
4.520
4.239
4.221
4.204
4.186
2.674
2.655
2.635
1.667
1.648
1.630
1.615
1.611
1.603
1.595
1.592
1.274
1.256
1.238
0.905
0.887
0.870
^{1}H NMR (400 MHz, CDCl$_3$)
H$_3$C
Cl
S
O
N
O
O
O
O
CH$_3$
(4zi)
2.03
4.00
1.08
1.06
2.02
2.05
2.11
2.09
4.00
3.04
3.03
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)

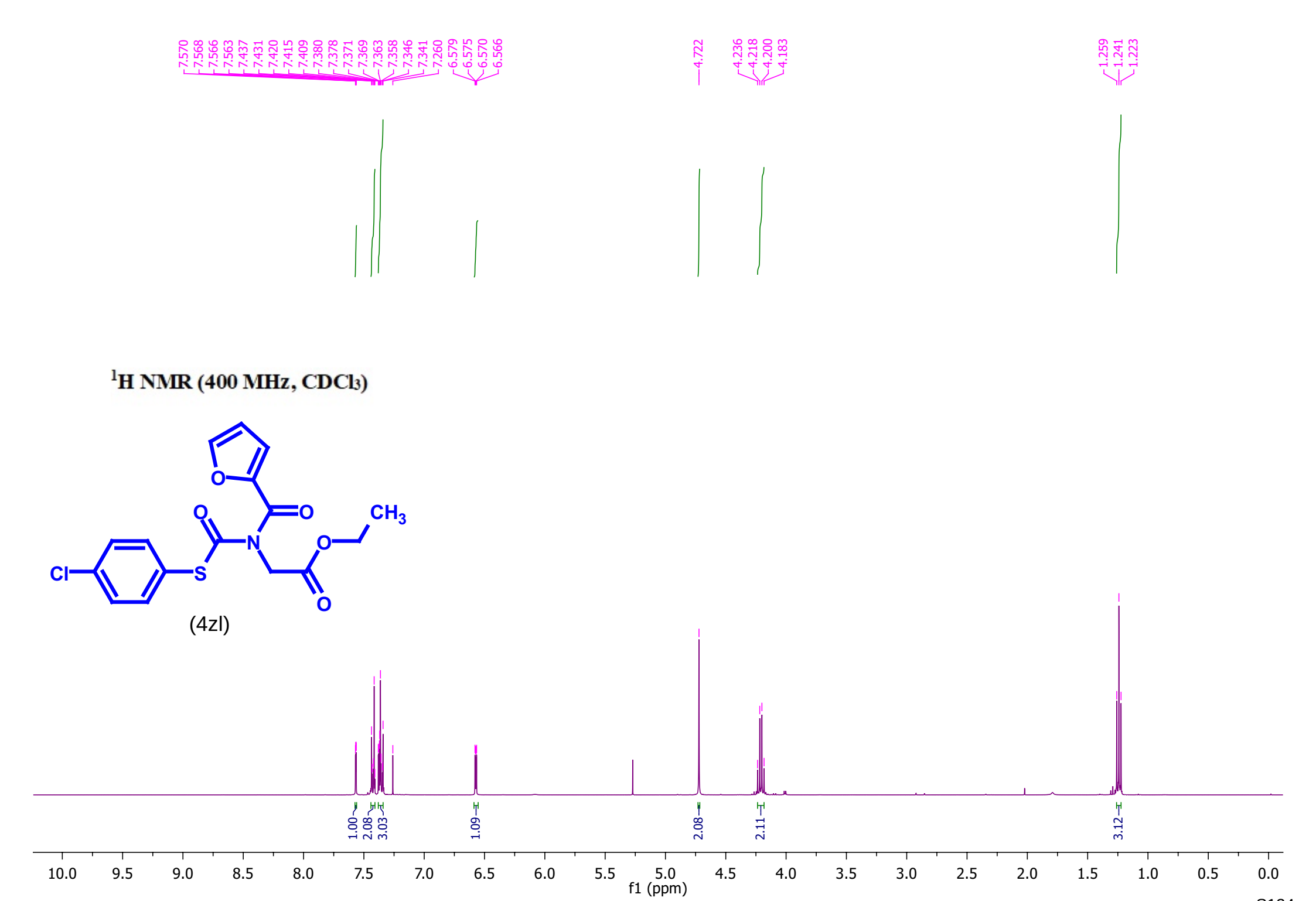

172.40
170.51
167.96
148.11
136.42
136.15
131.36
129.31
128.68
128.34
126.62
77.32
77.00
76.68
61.71
48.83
35.86
31.32
30.66
22.40
14.02
13.93
13C NMR (100 MHz, CDCl3)
H3C
Cl
S
O
N
O
O
O
O
CH3
(4zi)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

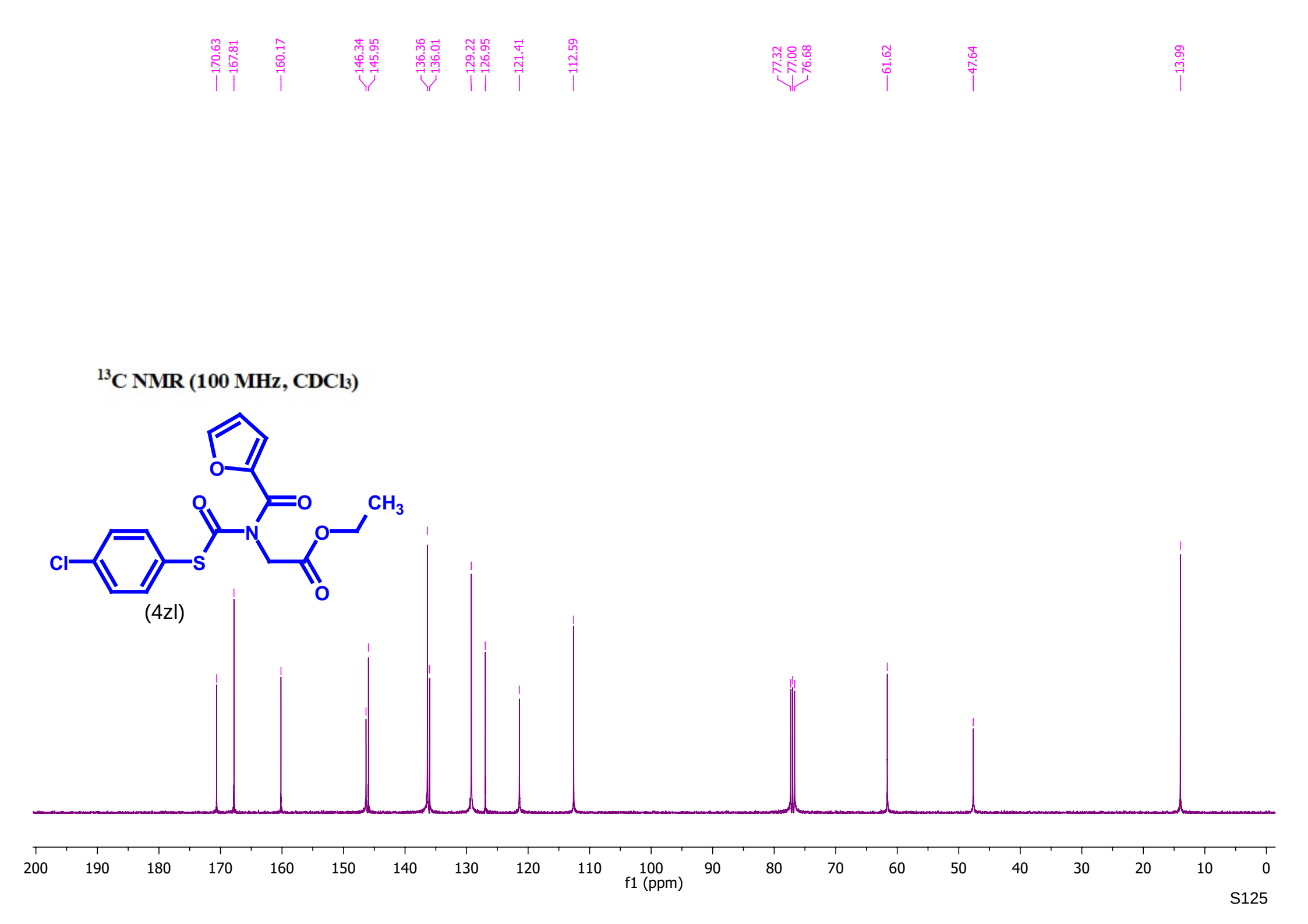

7.444
7.442
7.436
7.429
7.421
7.419
7.416
7.409
7.402
7.396
7.394
7.260
7.169
7.149
7.133
7.127
7.112
4.578
4.269
4.251
4.233
4.216
4.045
2.343
1.315
1.297
1.279
^{1}H NMR (400 MHz, CDCl$_3$)
CH$_3$
O
O
CH$_3$
N
O
Cl
S
O
(4zj)
4.00
4.00
2.00
2.06
2.00
3.10
3.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

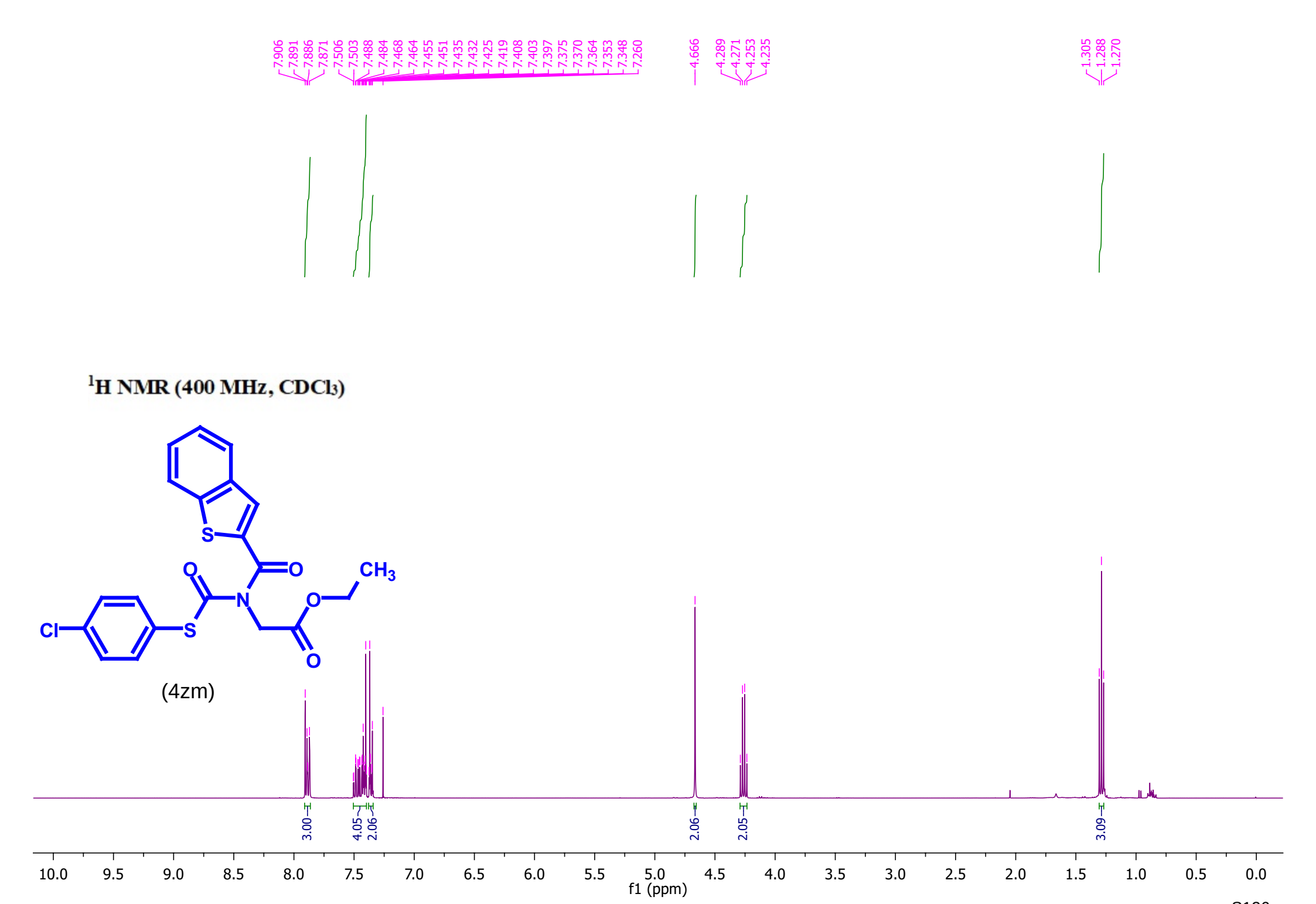

172.87
170.07
167.55
136.88
136.60
136.46
129.86
129.46
129.27
129.25
125.98
77.32
77.00
76.68
61.87
46.24
43.59
21.03
14.03
13C NMR (100 MHz, CDCl3)
CH3
O
O
CH3
N
O
Cl
S
O
(4zj)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.853
7.814
7.552
7.548
7.535
7.529
7.478
7.473
7.467
7.456
7.451
7.445
7.426
7.421
7.415
7.399
7.378
7.374
7.367
7.360
7.260
7.237
7.206
7.198
4.682
4.309
4.292
4.274
4.256
1.334
1.316
1.298
1H NMR (400 MHz, CDCl3)
Cl
S
N
O
O
O
O
O
CH3
(4zk)
1.00
2.06
2.01
4.03
1.09
1.00
2.05
2.12
3.09
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)


169.97
167.69
167.24
146.68
136.63
136.46
134.19
130.64
129.47
128.76
128.40
128.32
125.83
118.59
77.32
77.00
76.68
61.85
46.41
14.06
13C NMR (100 MHz, CDCl3)
O
O
CH3
N
O
Cl
S
O
(4zk)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

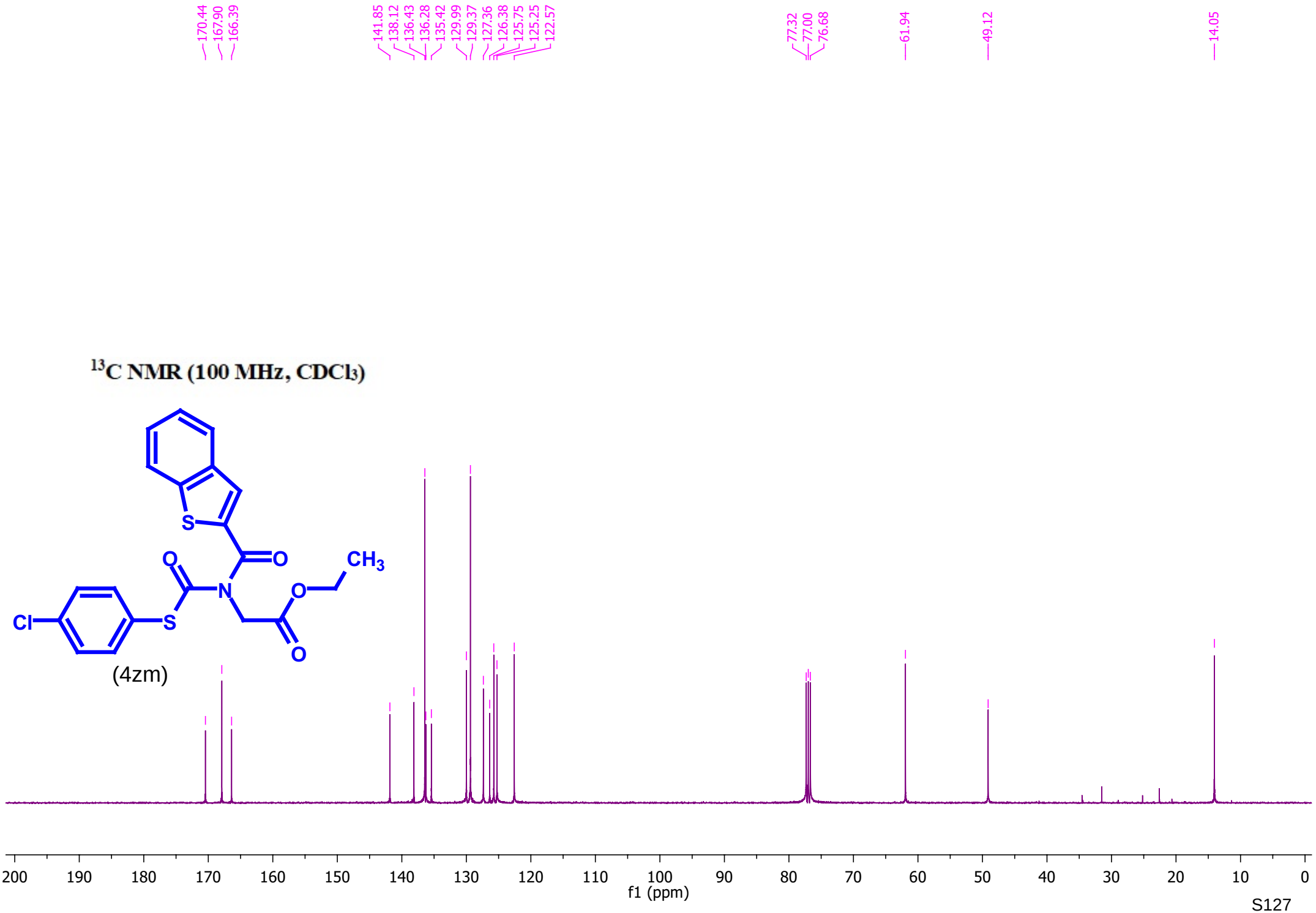

7.570
7.568
7.566
7.563
7.437
7.431
7.420
7.415
7.409
7.380
7.378
7.371
7.369
7.363
7.358
7.346
7.341
7.260
6.579
6.575
6.570
6.566
4.722
4.236
4.218
4.200
4.183
1.259
1.241
1.223
1H NMR (400 MHz, CDCl3)
O
O
O
CH3
N
O
Cl
S
O
(4zl)
1.00
2.08
3.03
1.09
2.08
2.11
3.12
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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)

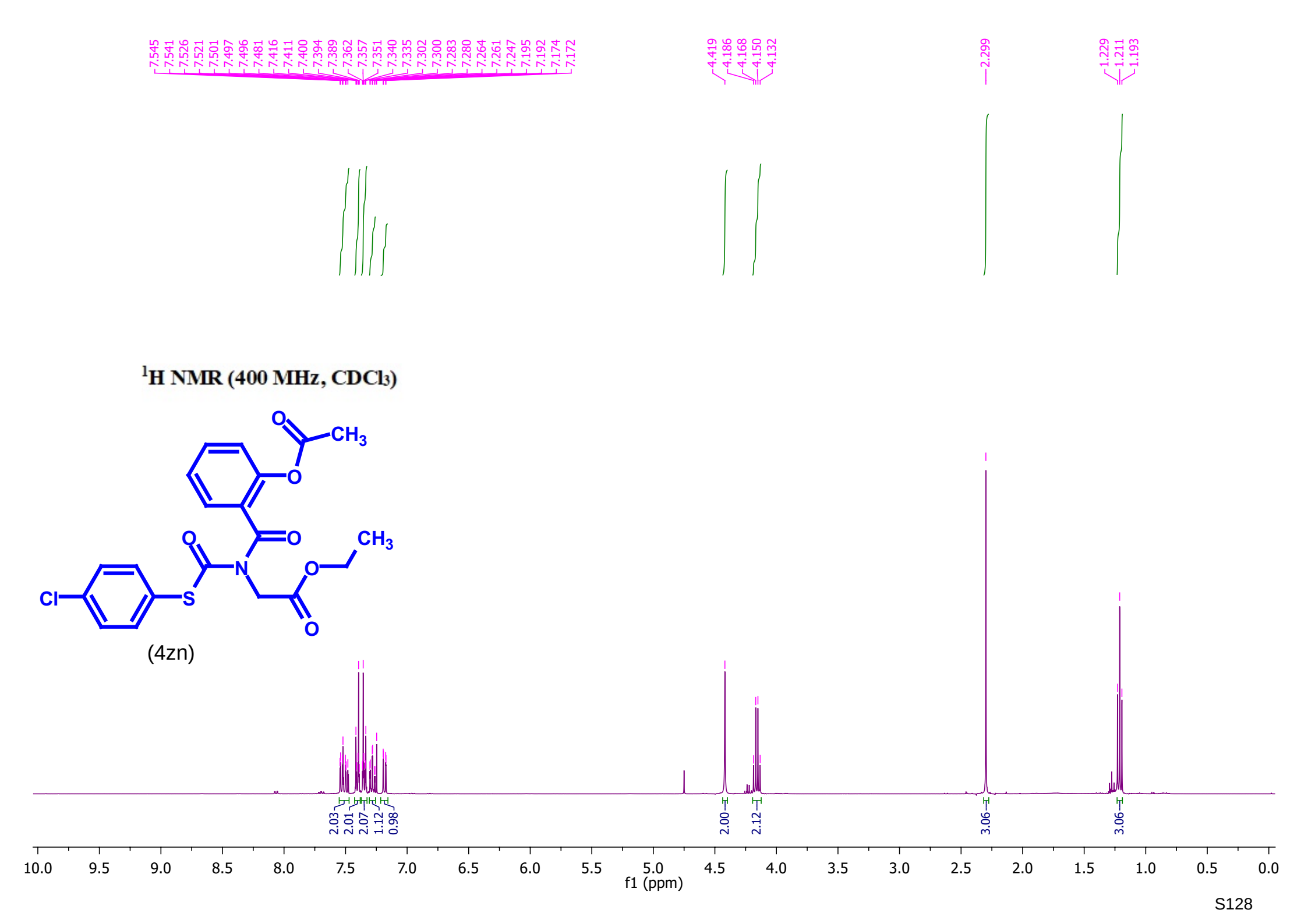

170.63
167.81
160.17
146.34
145.95
136.36
136.01
129.22
126.95
121.41
112.59
77.32
77.00
76.68
61.62
47.64
13.99
13C NMR (100 MHz, CDCl3)
O
O
O
CH3
N
O
Cl
S
O
(4zl)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

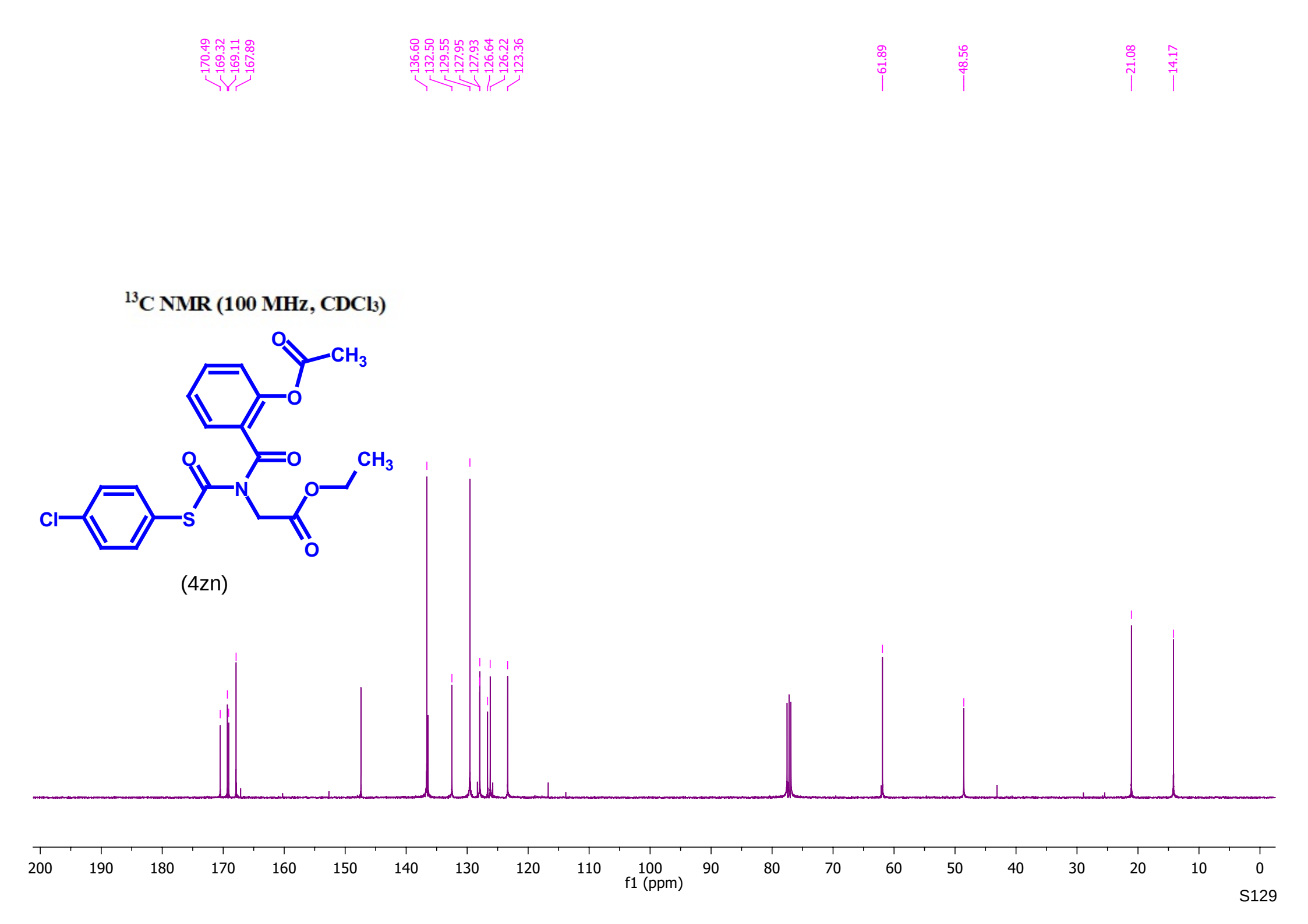


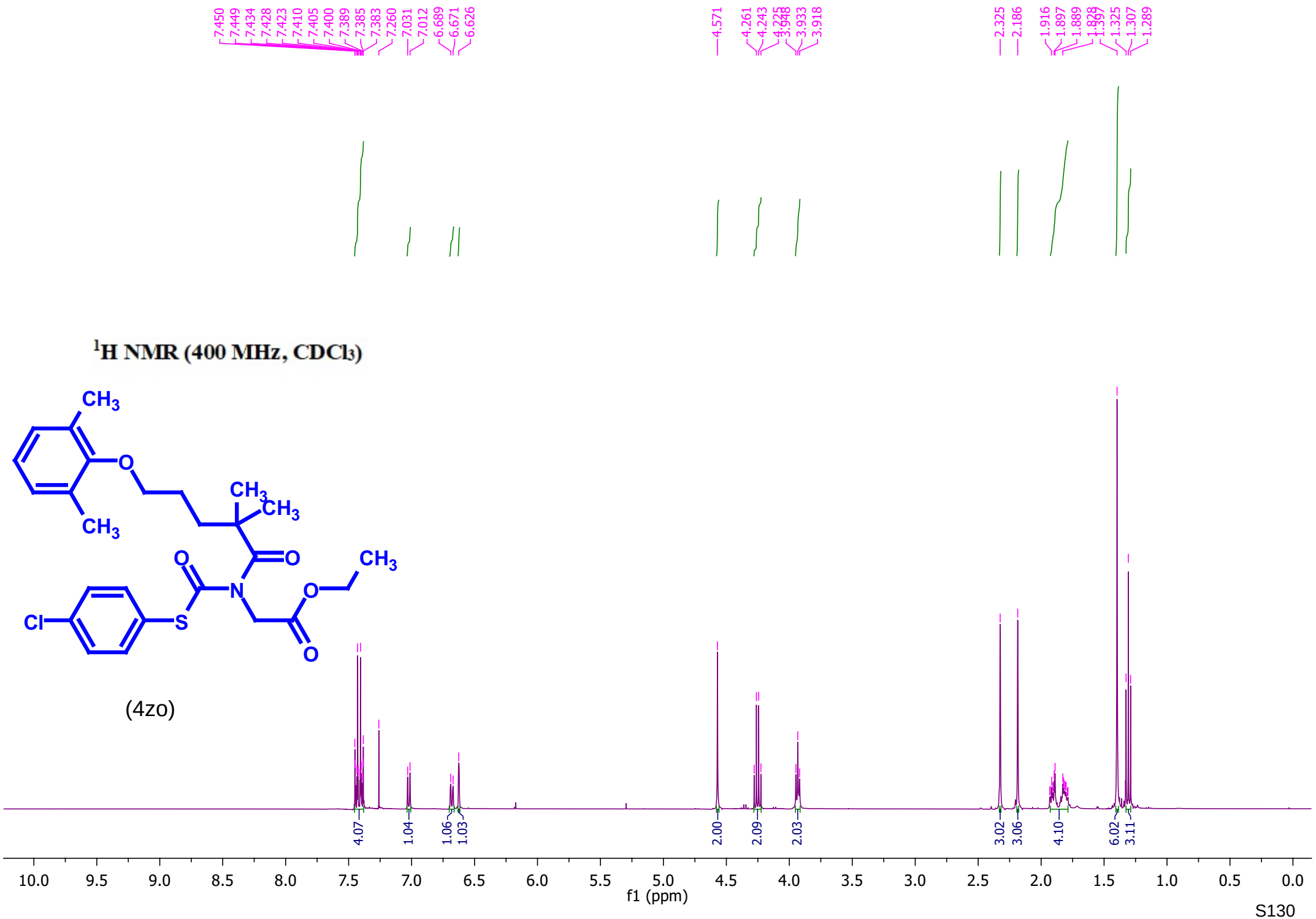
^{1}H NMR (400 MHz, $CDCl_3$)

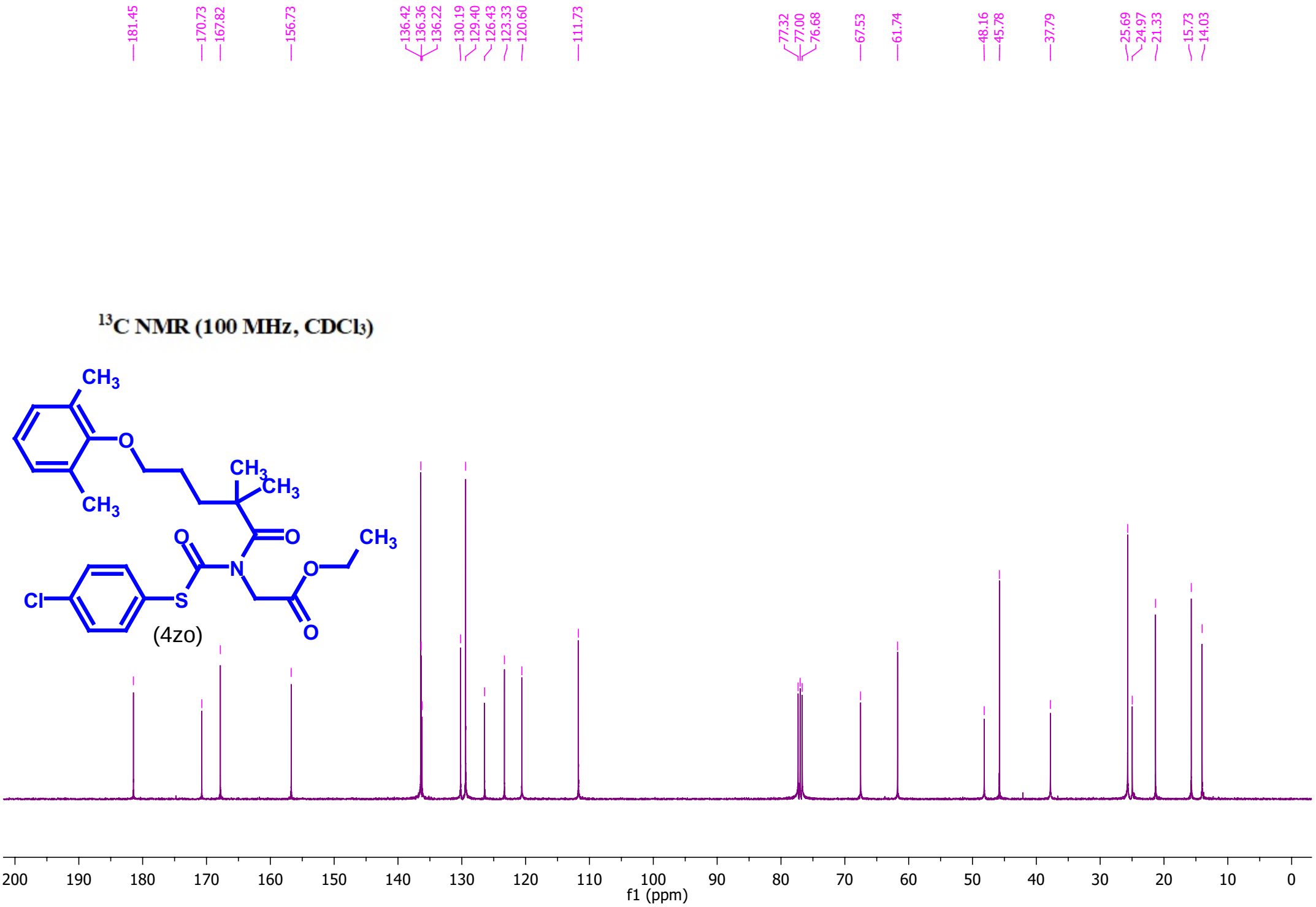
(4zm)

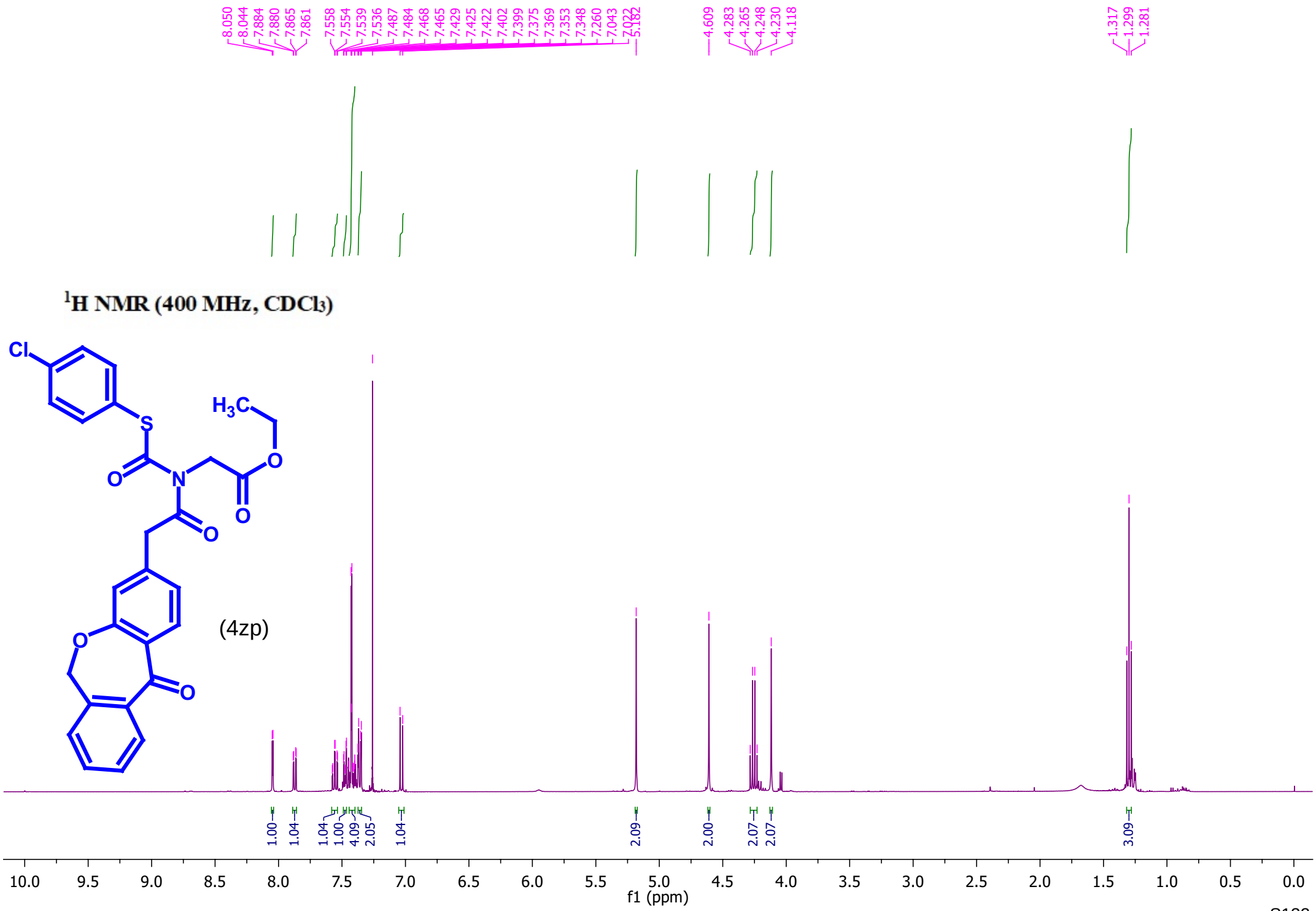

170.44
167.90
166.39
141.85
138.12
136.43
136.28
135.42
129.99
129.37
127.36
126.38
125.75
125.25
122.57
77.32
77.00
76.68
61.94
49.12
14.05
^{13}C NMR (100 MHz, CDCl$_3$)
S
O
O
N
O
CH$_3$
Cl
S
O
O
(4zm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

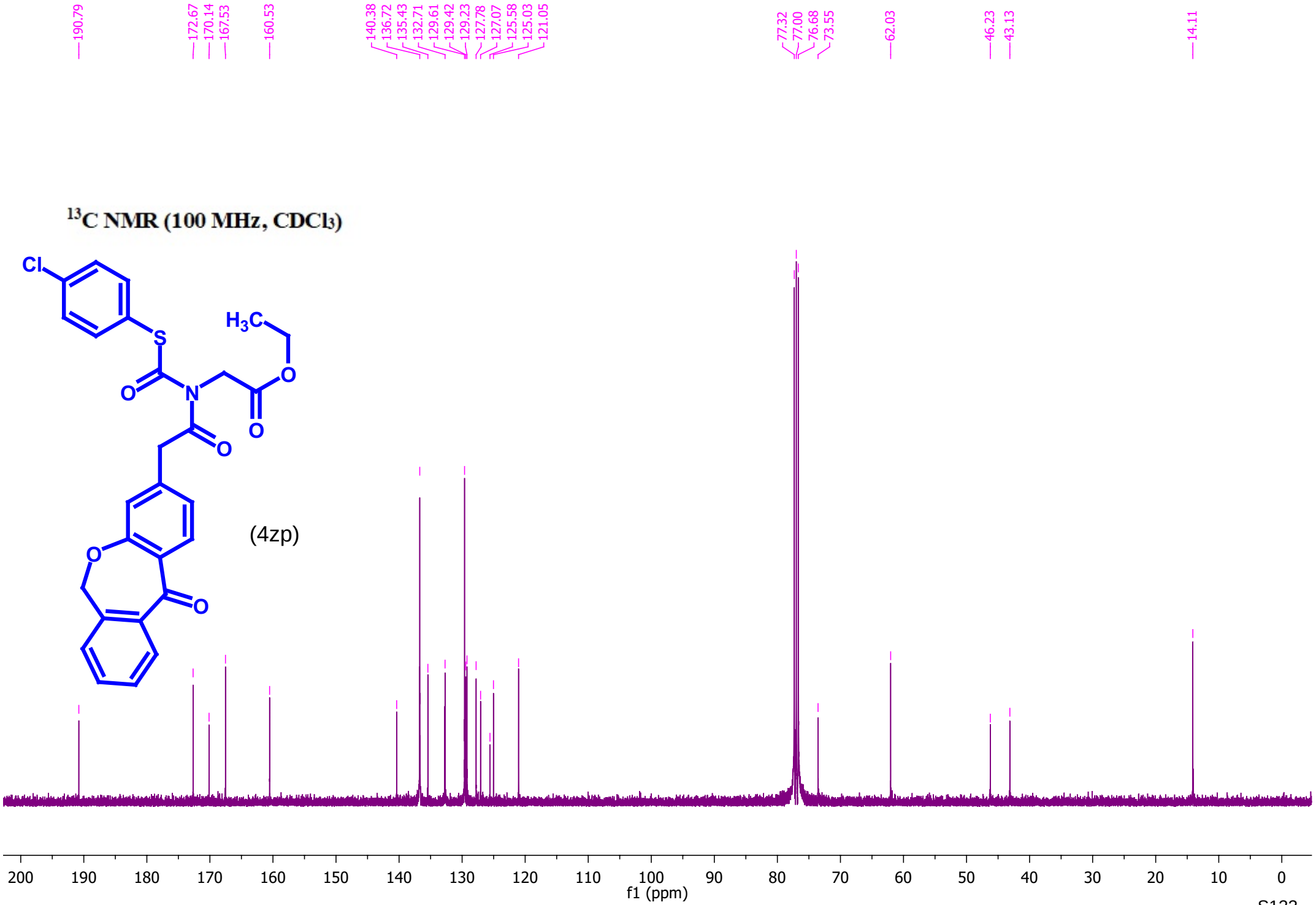

7.545
7.541
7.526
7.521
7.501
7.497
7.496
7.481
7.416
7.411
7.400
7.394
7.389
7.362
7.357
7.351
7.340
7.335
7.302
7.300
7.283
7.280
7.264
7.261
7.247
7.195
7.192
7.174
7.172
4.419
4.186
4.168
4.150
4.132
2.299
1.229
1.211
1.193
^{1}H NMR (400 MHz, CDCl$_3$)
(4zn)
2.03
2.01
2.07
1.12
0.98
2.00
2.12
3.06
3.06
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)

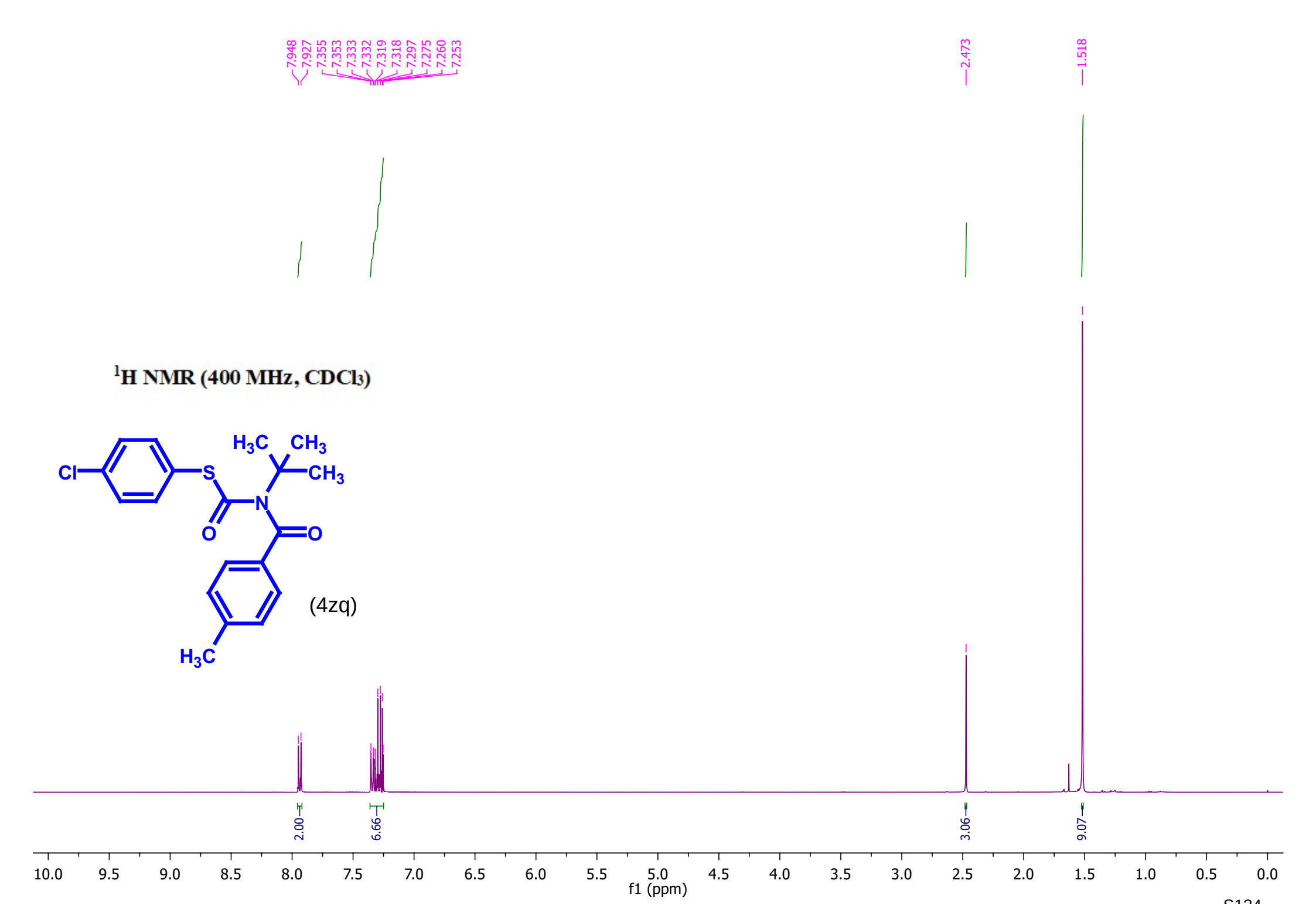

170.49
169.32
169.11
167.89
136.60
132.50
129.55
127.95
127.93
126.64
126.22
123.36
61.89
48.56
21.08
14.17
13C NMR (100 MHz, CDCl3)
O
CH3
O
O
O
CH3
N
O
Cl
S
O
(4zn)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

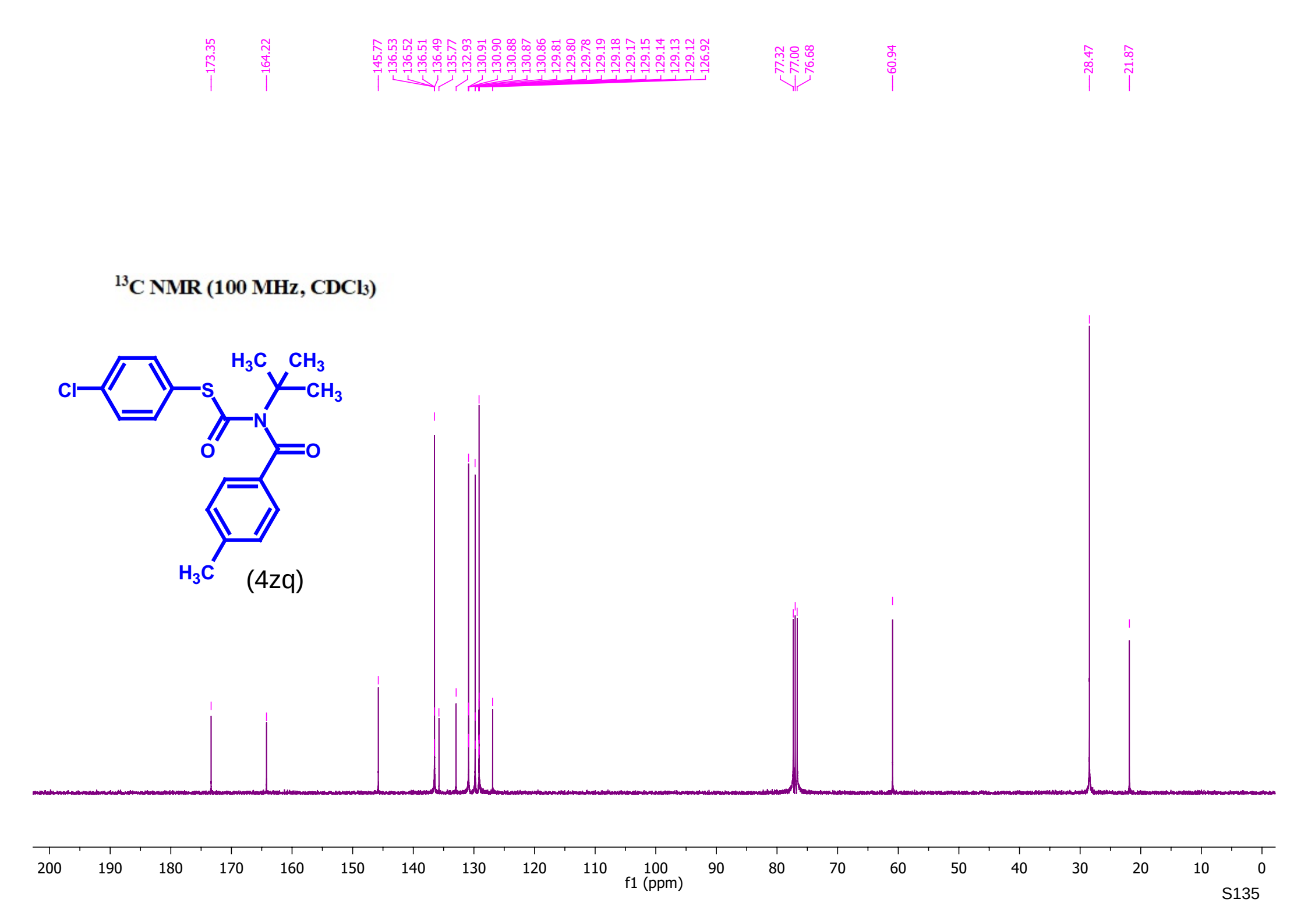

7.450
7.449
7.434
7.428
7.423
7.410
7.405
7.400
7.389
7.385
7.383
7.260
7.031
7.012
6.689
6.671
6.626
4.571
4.261
4.243
4.225
3.948
3.933
3.918
2.325
2.186
1.916
1.897
1.889
1.828
1.397
1.325
1.307
1.289
1H NMR (400 MHz, CDCl3)
CH3
O
CH3
CH3
CH3
O
O
CH3
N
O
Cl
S
O
O
(4zo)
4.07
1.04
1.06
1.03
2.00
2.09
2.03
3.02
3.06
4.10
6.02
3.11
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)

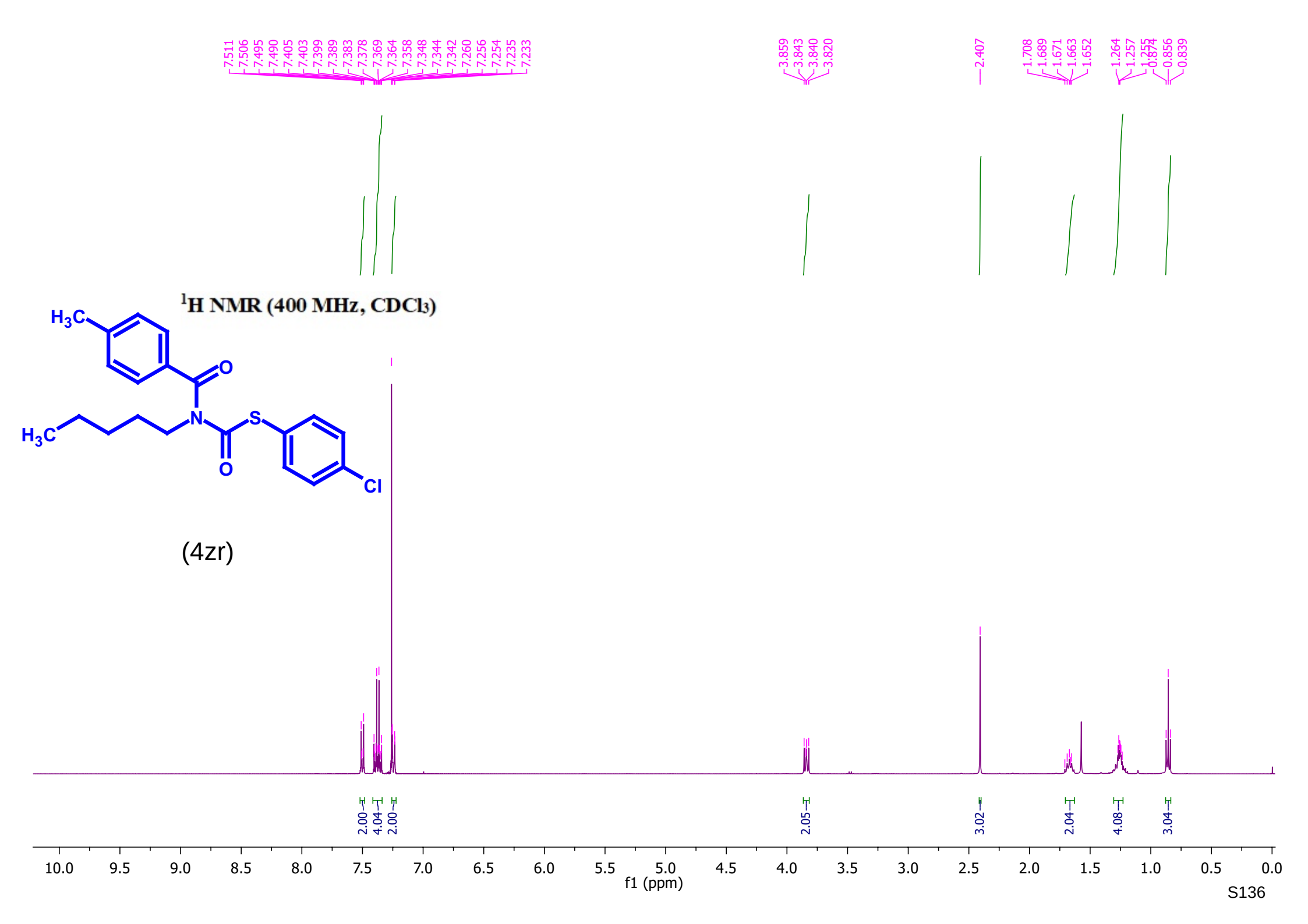

181.45
170.73
167.82
156.73
136.42
136.36
136.22
130.19
129.40
126.43
123.33
120.60
111.73
77.32
77.00
76.68
67.53
61.74
48.16
45.78
37.79
25.69
24.97
21.33
15.73
14.03
^{13}C NMR (100 MHz, CDCl$_3$)
CH$_3$
O
CH$_3$
CH$_3$
CH$_3$
O
O
CH$_3$
N
O
Cl
S
O
(4zo)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

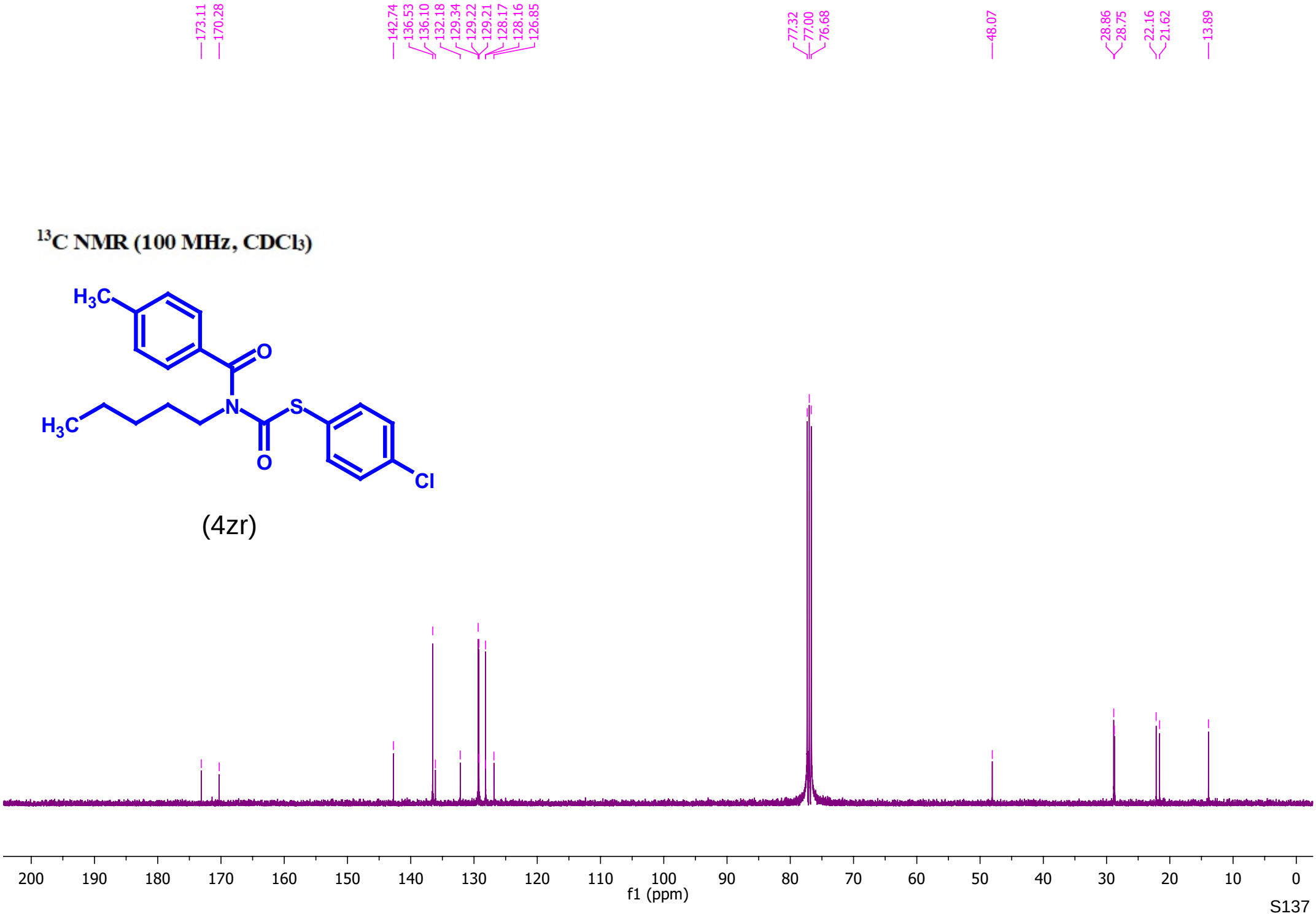

8.050
8.044
7.884
7.880
7.865
7.861
7.558
7.554
7.539
7.536
7.487
7.484
7.468
7.465
7.429
7.425
7.422
7.402
7.399
7.375
7.369
7.353
7.348
7.260
7.043
7.022
5.182
4.609
4.283
4.265
4.248
4.230
4.118
1.317
1.299
1.281
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
S
O
N
O
O
O
H_3C
O
O
(4zp)
1.00
1.04
1.04
1.00
4.09
2.05
1.04
2.09
2.00
2.07
2.07
3.09
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)

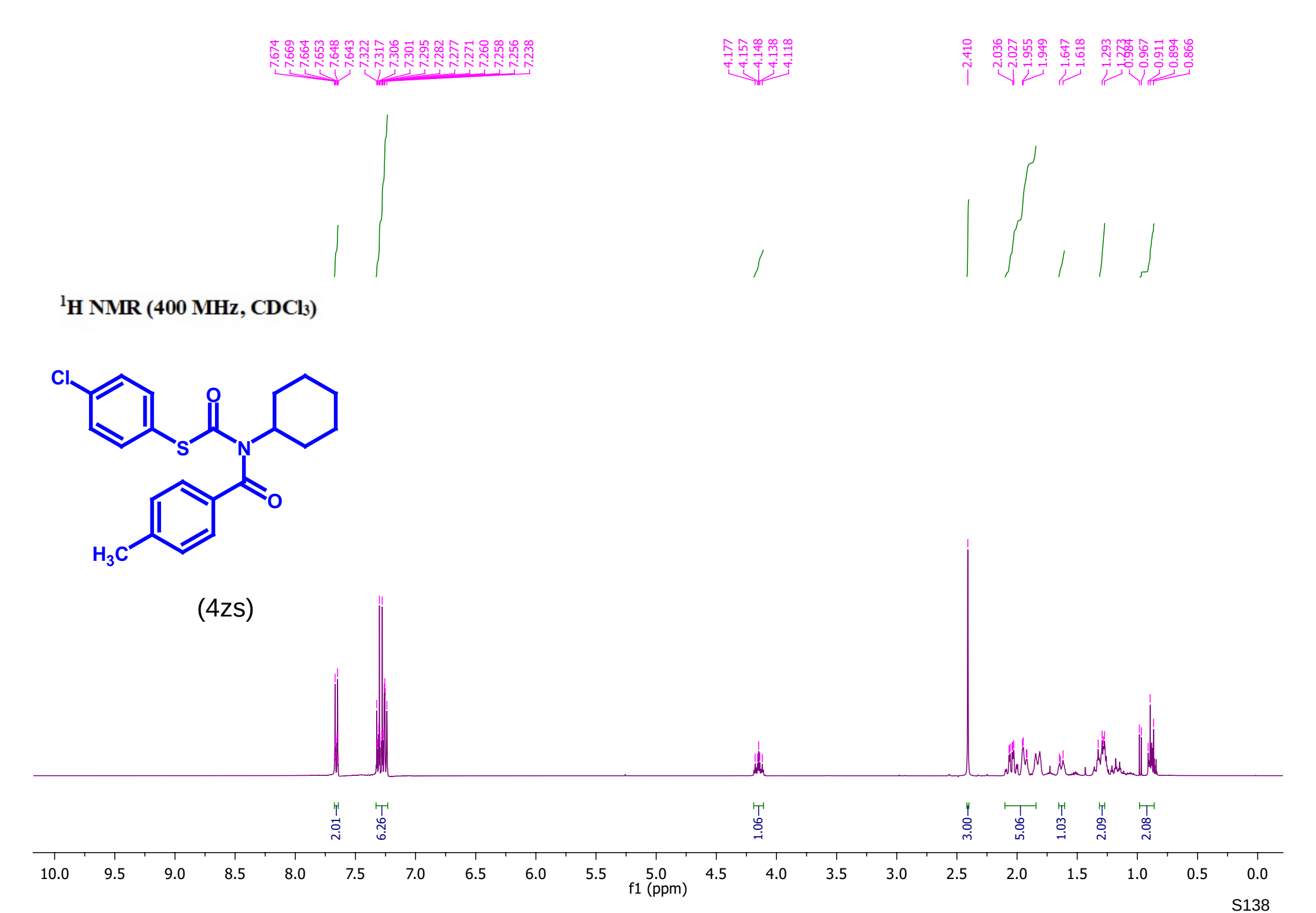

190.79
172.67
170.14
167.53
160.53
140.38
136.72
135.43
132.71
129.61
129.42
129.23
127.78
127.07
125.58
125.03
121.05
77.32
77.00
76.68
73.55
62.03
46.23
43.13
14.11
^{13}C NMR (100 MHz, CDCl$_3$)
Cl
S
O
N
O
O
H_3C
O
O
O
O
(4zp)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.948
7.927
7.355
7.353
7.333
7.332
7.319
7.318
7.297
7.275
7.260
7.253
2.473
1.518
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
S
N
O
O
H$_3$C
CH$_3$
CH$_3$
H$_3$C
(4zq)
2.00
6.66
3.06
9.07
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
f1 (ppm)


173.35
164.22
145.77
136.53
136.52
136.51
136.49
135.77
132.93
130.91
130.90
130.88
130.87
130.86
129.81
129.80
129.78
129.19
129.18
129.17
129.15
129.14
129.13
129.12
126.92
77.32
77.00
76.68
60.94
28.47
21.87
13C NMR (100 MHz, CDCl3)
Cl
S
N
O
O
H3C
CH3
CH3
H3C
(4zq)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.511
7.506
7.495
7.490
7.405
7.403
7.399
7.389
7.383
7.378
7.369
7.364
7.358
7.348
7.344
7.342
7.260
7.256
7.254
7.235
7.233
3.859
3.843
3.840
3.820
2.407
1.708
1.689
1.671
1.663
1.652
1.264
1.257
1.255
0.874
0.856
0.839
^{1}H NMR (400 MHz, CDCl$_3$)
H$_3$C
O
N
S
H$_3$C
O
Cl
(4zr)
2.00
4.04
2.00
2.05
3.02
2.04
4.08
3.04
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)


173.11
170.28
142.74
136.53
136.10
132.18
129.34
129.22
129.21
128.17
128.16
126.85
77.32
77.00
76.68
48.07
28.86
28.75
22.16
21.62
13.89
^{13}C NMR (100 MHz, CDCl$_3$)
H$_3$C
O
N
S
H$_3$C
O
Cl
(4zr)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

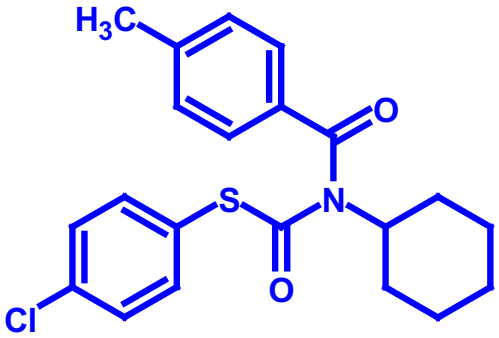


^{1}H NMR (400 MHz, $CDCl_3$)

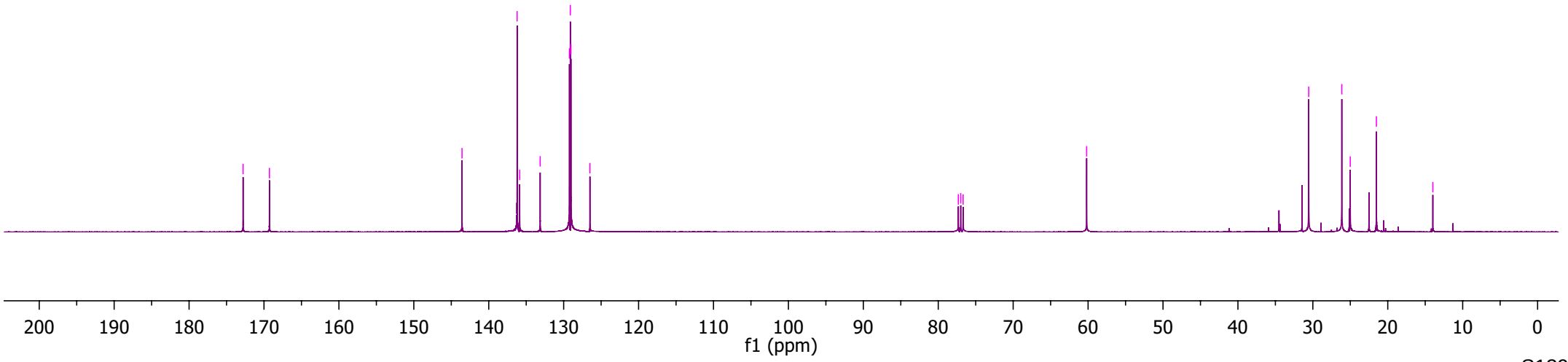
(4zs)

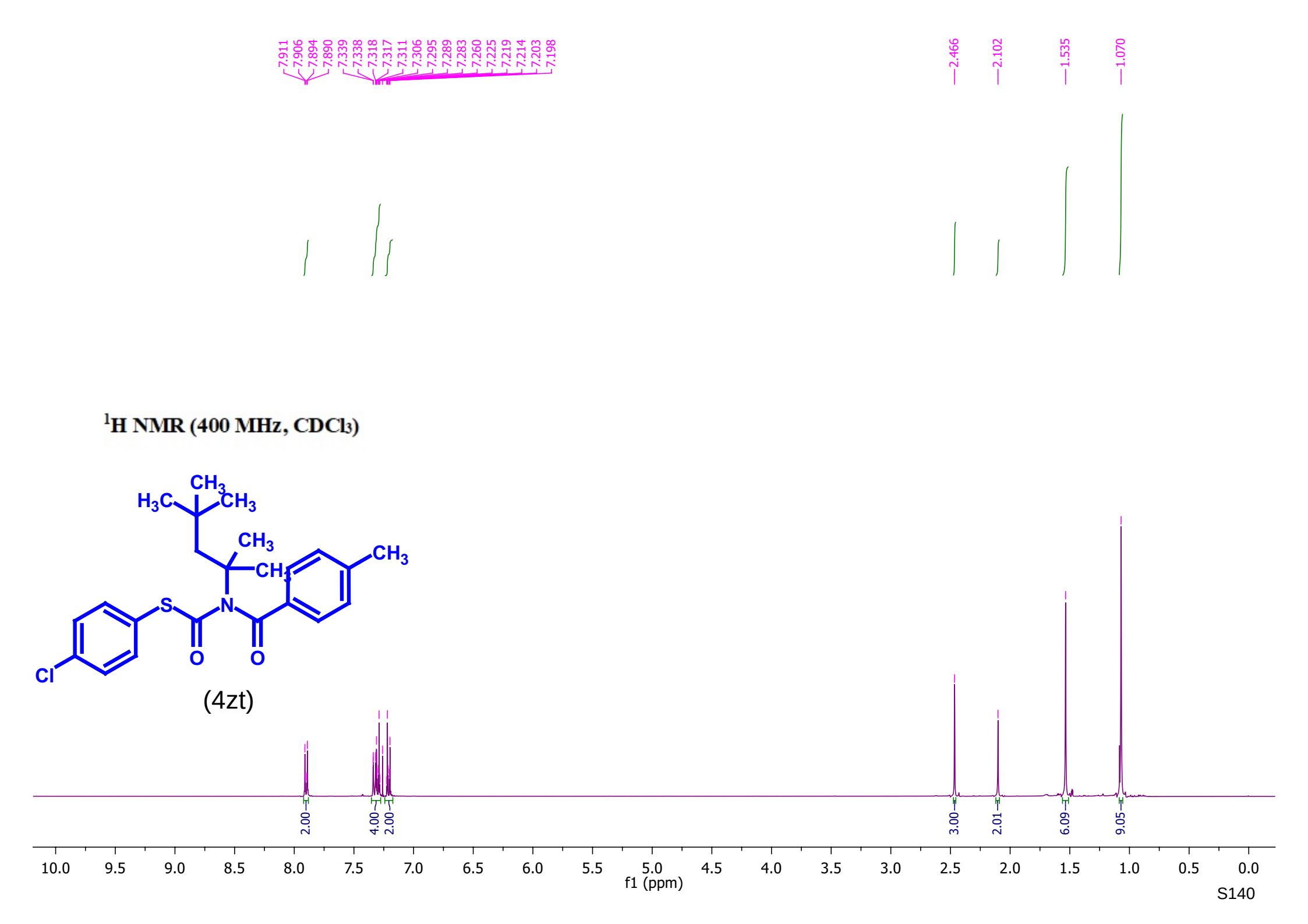
172.79
169.27
143.57
136.21
135.87
133.13
129.23
129.11
129.03
126.50
77.32
77.00
76.68
60.19
30.55
26.13
25.01
21.52
13.98

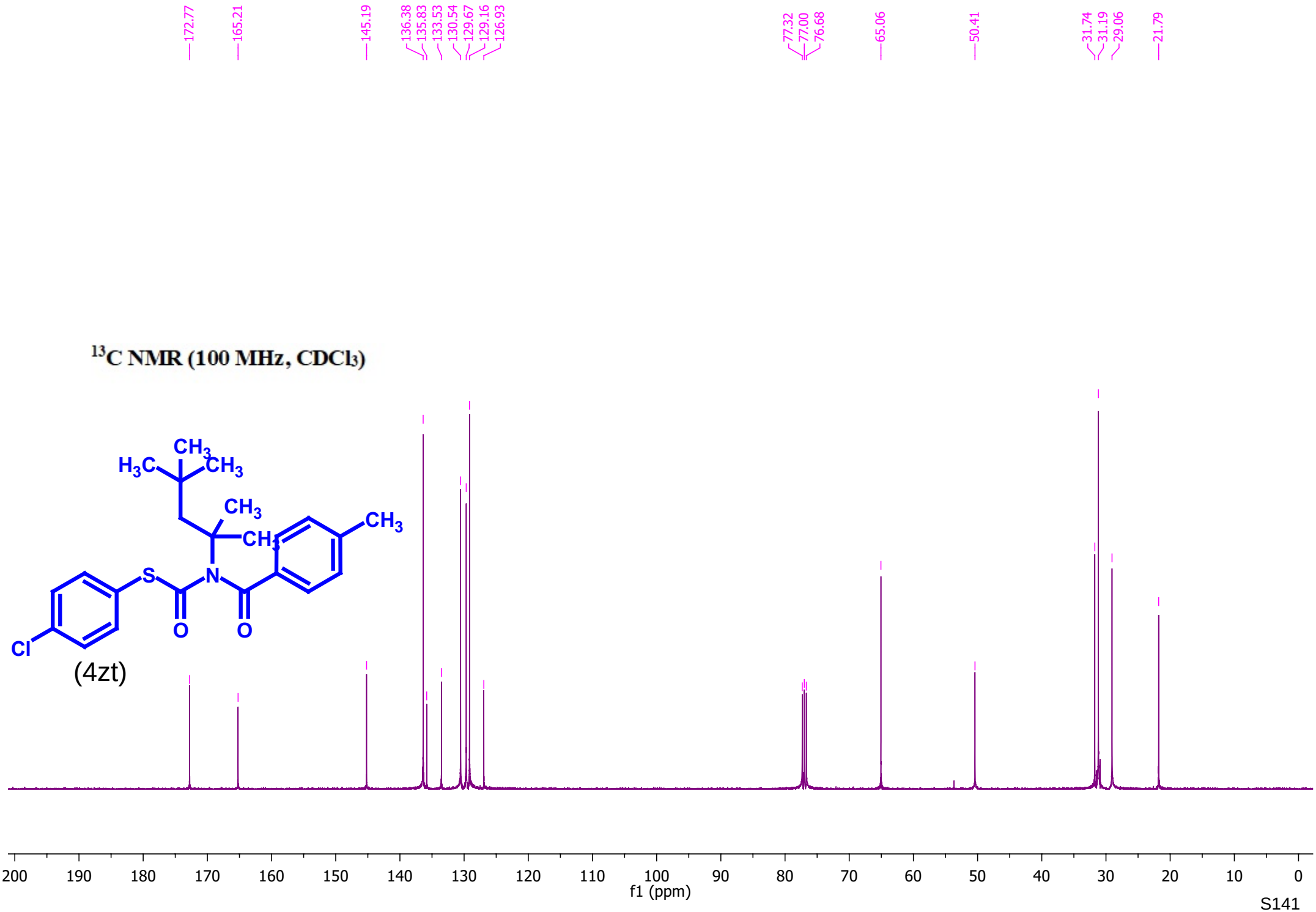
^{13}C NMR (100 MHz, $CDCl_3$)

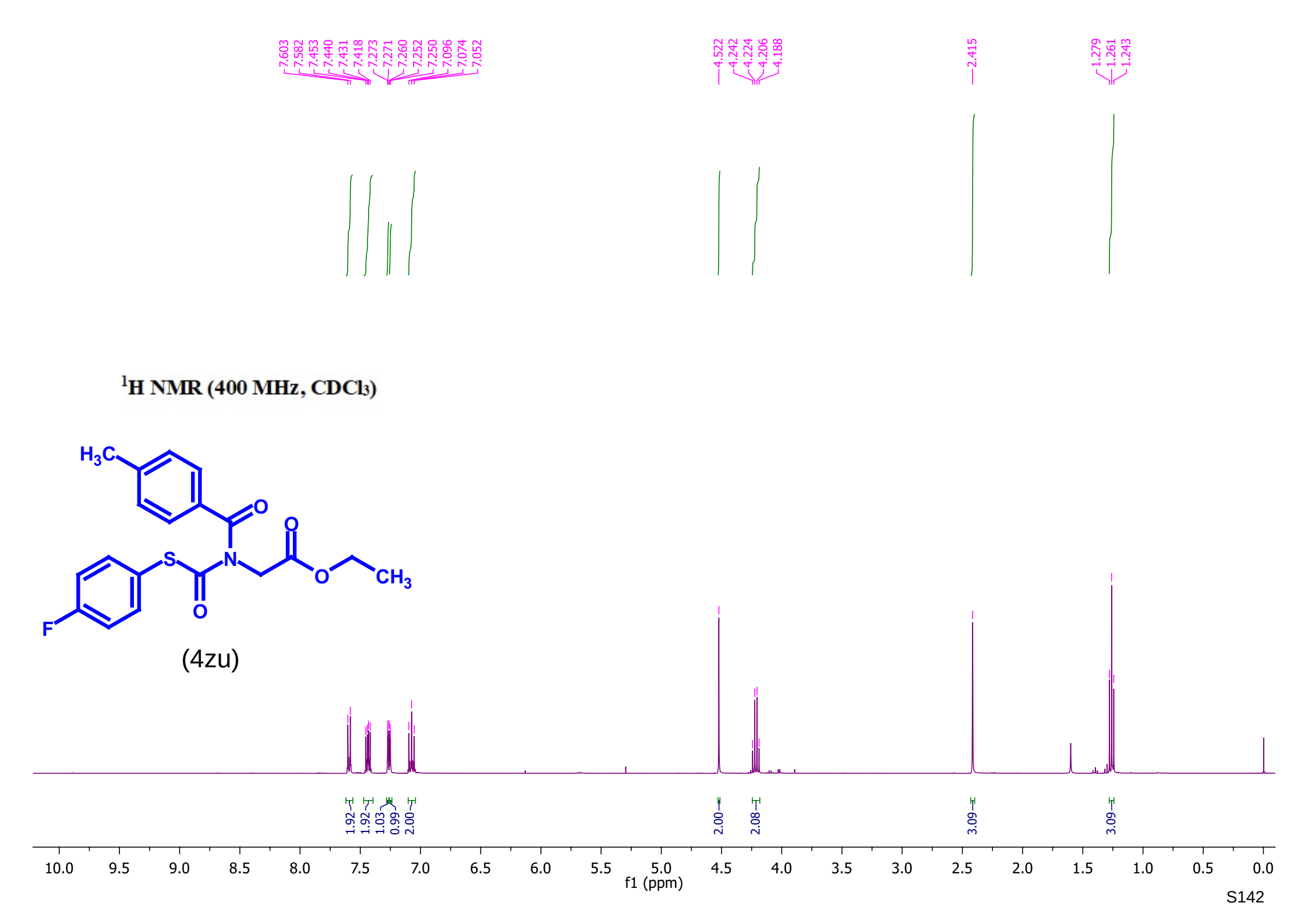


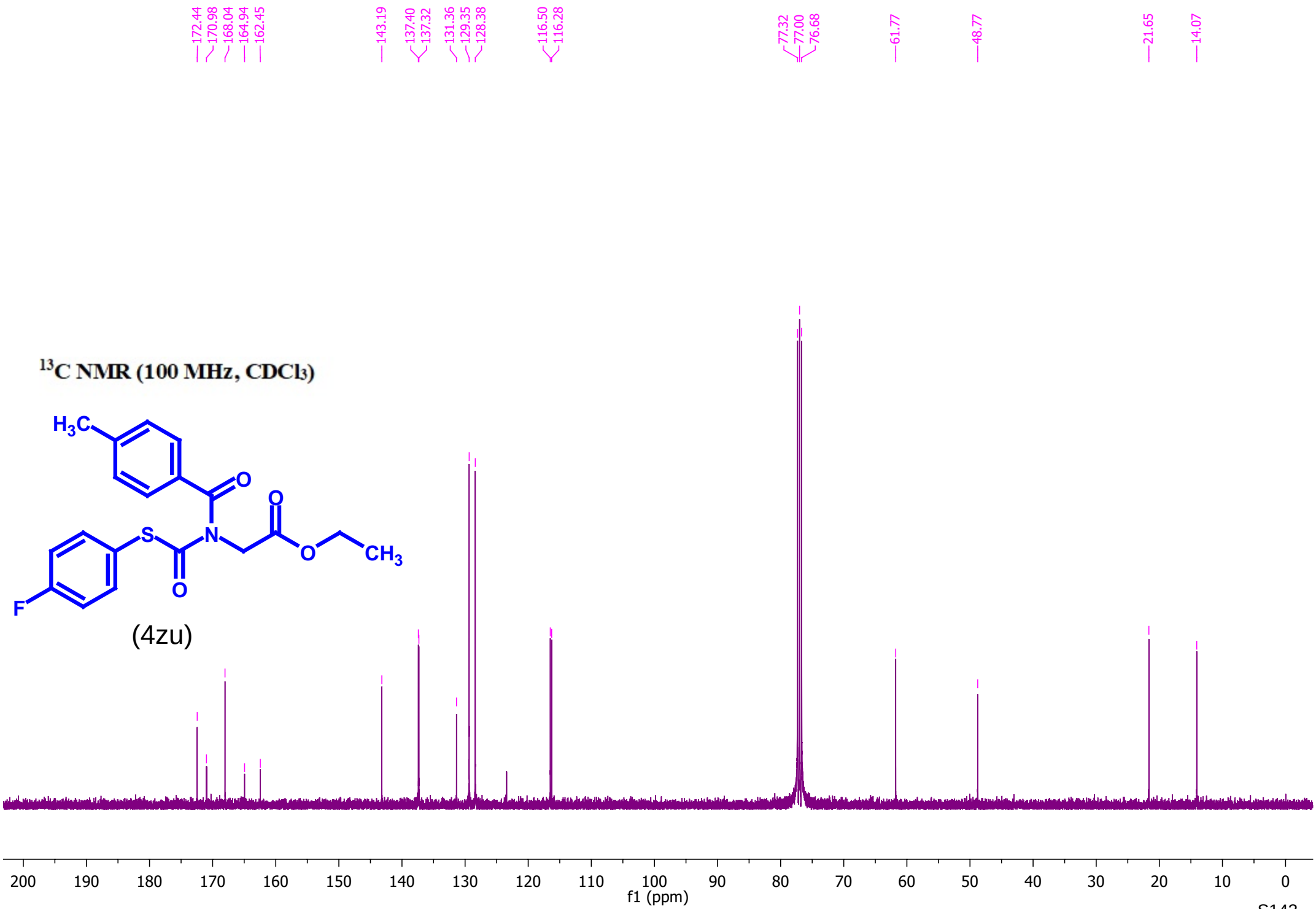
(4zs)



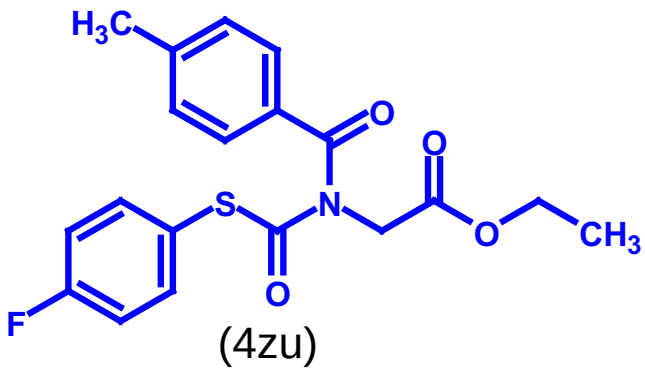

7.911
7.906
7.894
7.890
7.339
7.338
7.318
7.317
7.311
7.306
7.295
7.289
7.283
7.260
7.225
7.219
7.214
7.203
7.198
2.466
2.102
1.535
1.070
^{1}H NMR (400 MHz, CDCl$_3$)
CH$_3$
H$_3$C
CH$_3$
CH$_3$
CH$_3$
CH$_3$
S
N
O
O
Cl
(4zt)
2.00
4.00
2.00
3.00
2.01
6.09
9.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


172.77
165.21
145.19
136.38
135.83
133.53
130.54
129.67
129.16
126.93
77.32
77.00
76.68
65.06
50.41
31.74
31.19
29.06
21.79
^{13}C NMR (100 MHz, CDCl$_3$)
CH$_3$
H$_3$C
CH$_3$
CH$_3$
CH$_3$
CH$_3$
S
N
O
O
Cl
(4zt)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

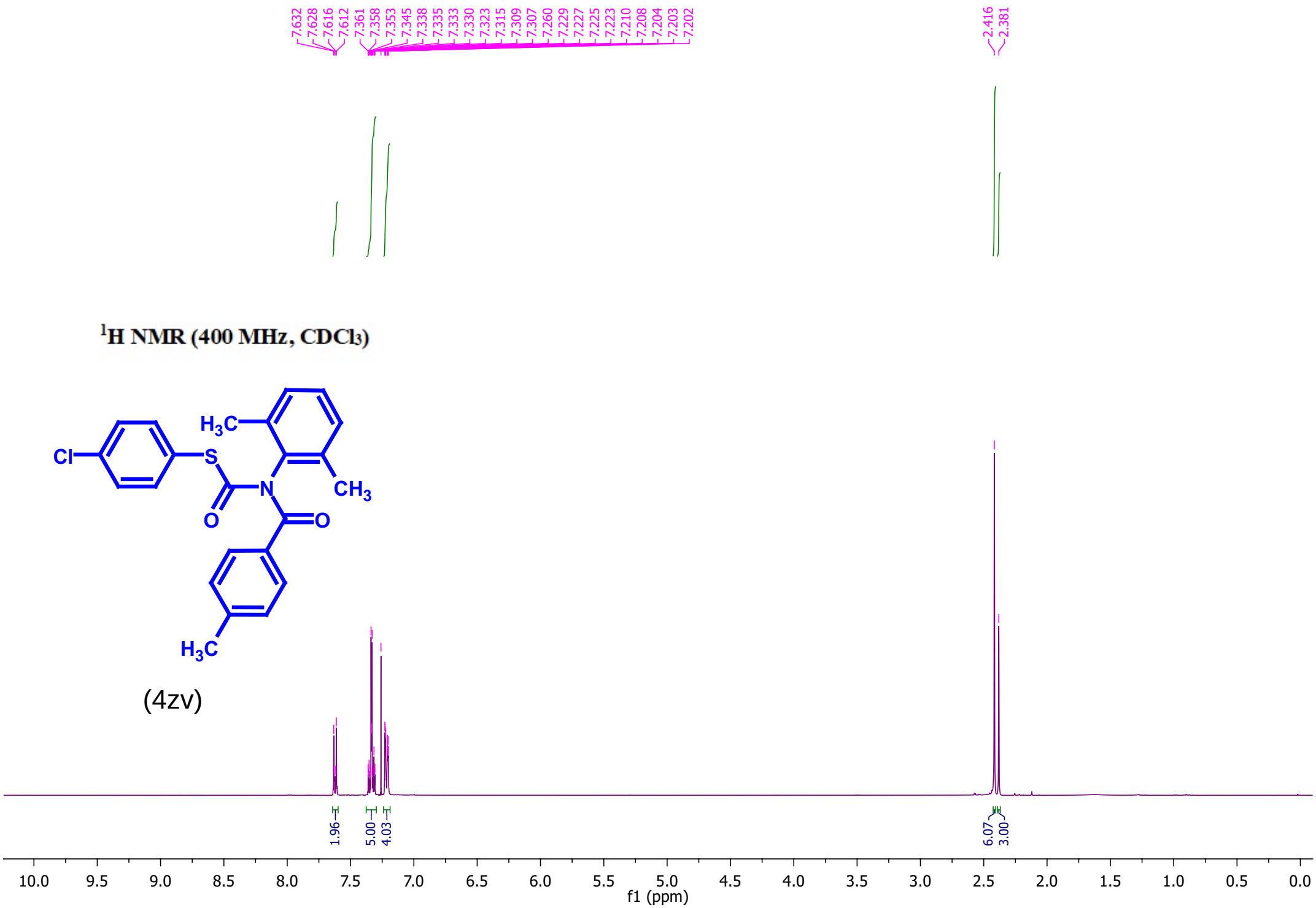

7.603
7.582
7.453
7.440
7.431
7.418
7.273
7.271
7.260
7.252
7.250
7.096
7.074
7.052
4.522
4.242
4.224
4.206
4.188
2.415
1.279
1.261
1.243
1H NMR (400 MHz, CDCl3)
H3C
O
O
S
N
O
O
CH3
O
F
(4zu)
1.92
1.92
1.03
0.99
2.00
2.00
2.08
3.09
3.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

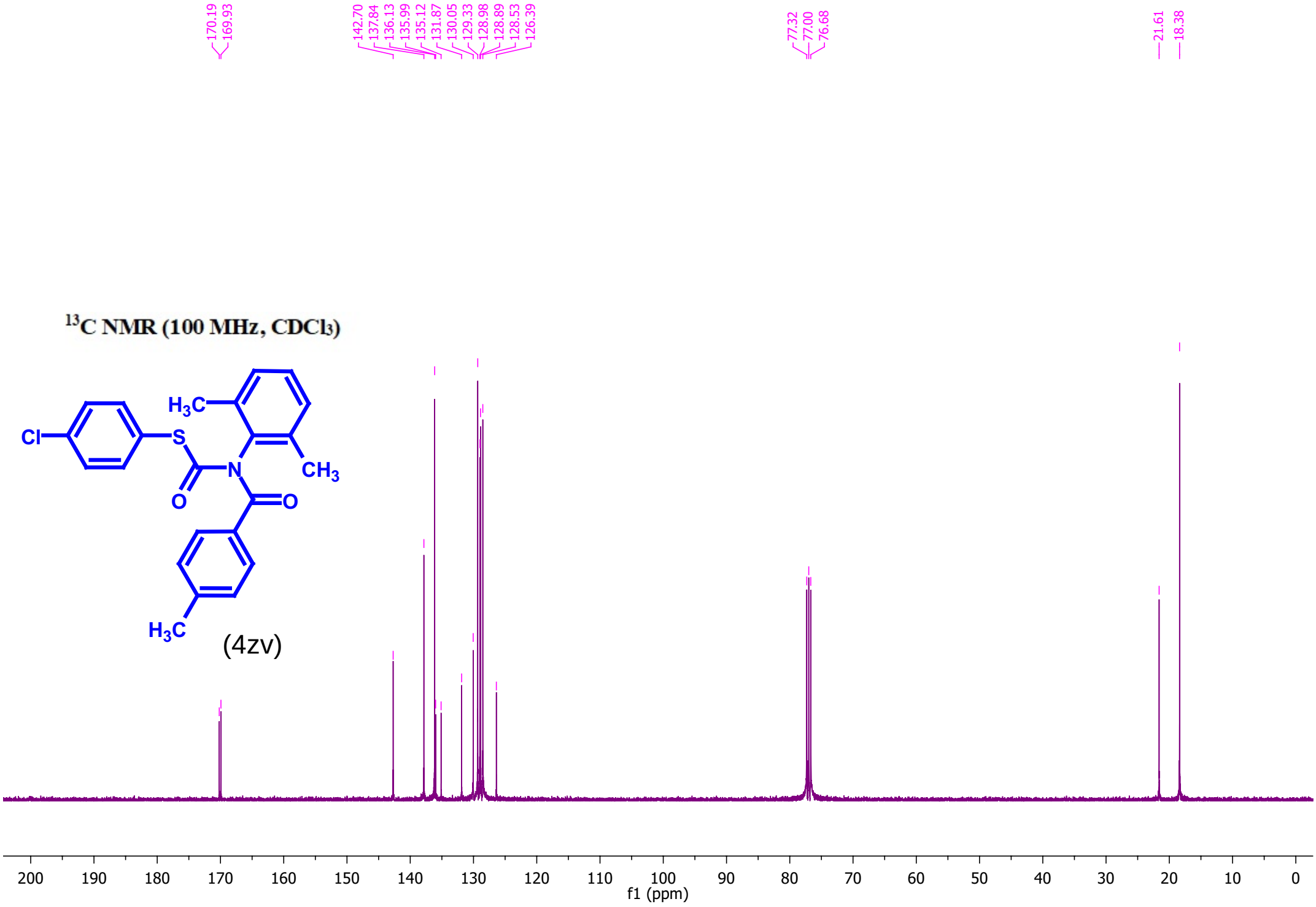

172.44
170.98
168.04
164.94
162.45
143.19
137.40
137.32
131.36
129.35
128.38
116.50
116.28
77.32
77.00
76.68
61.77
48.77
21.65
14.07
13C NMR (100 MHz, CDCl3)
H3C
O
O
S
N
O
O
CH3
F
O
(4zu)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

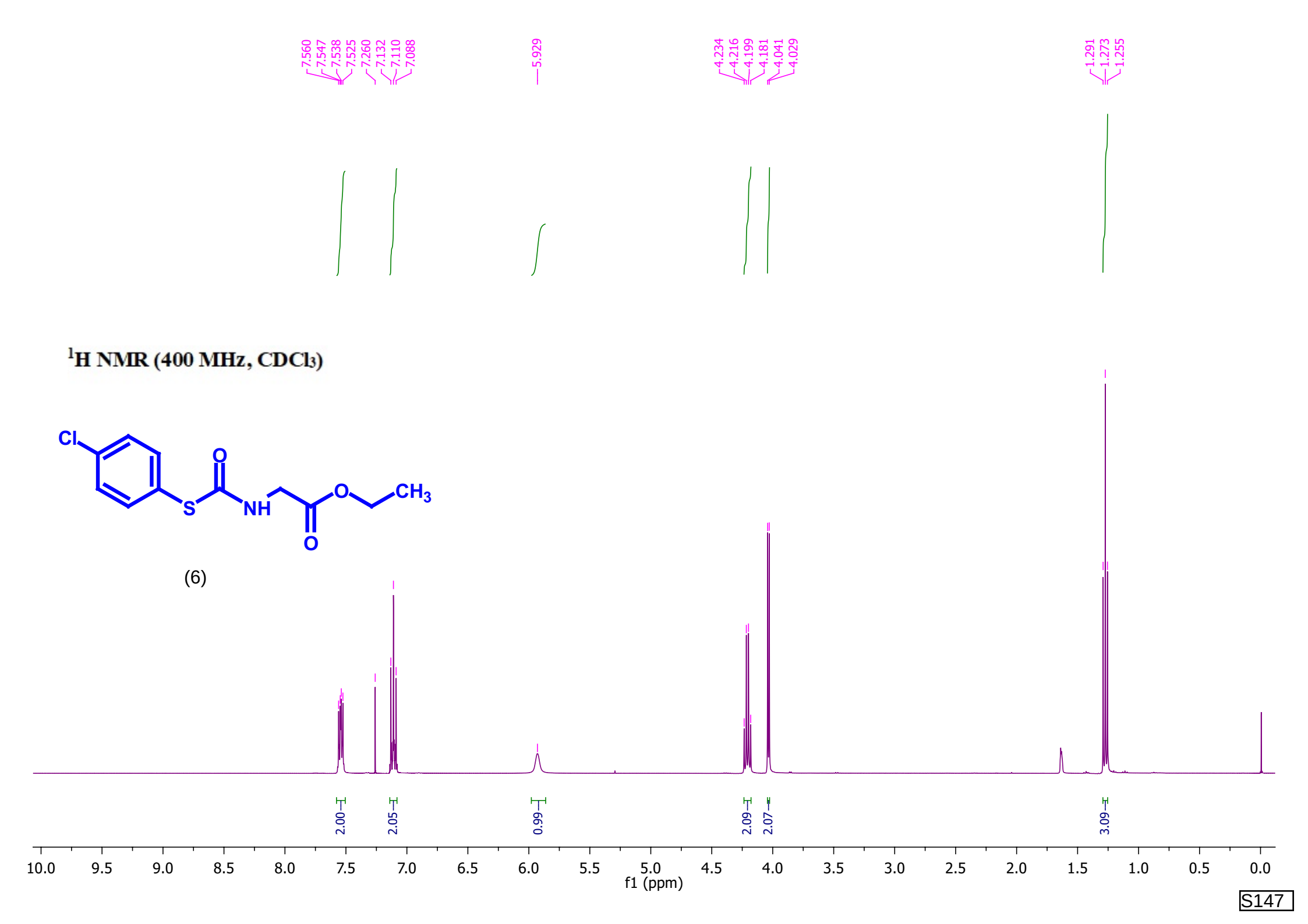
^{19}F NMR (376 MHz, $CDCl_3$)

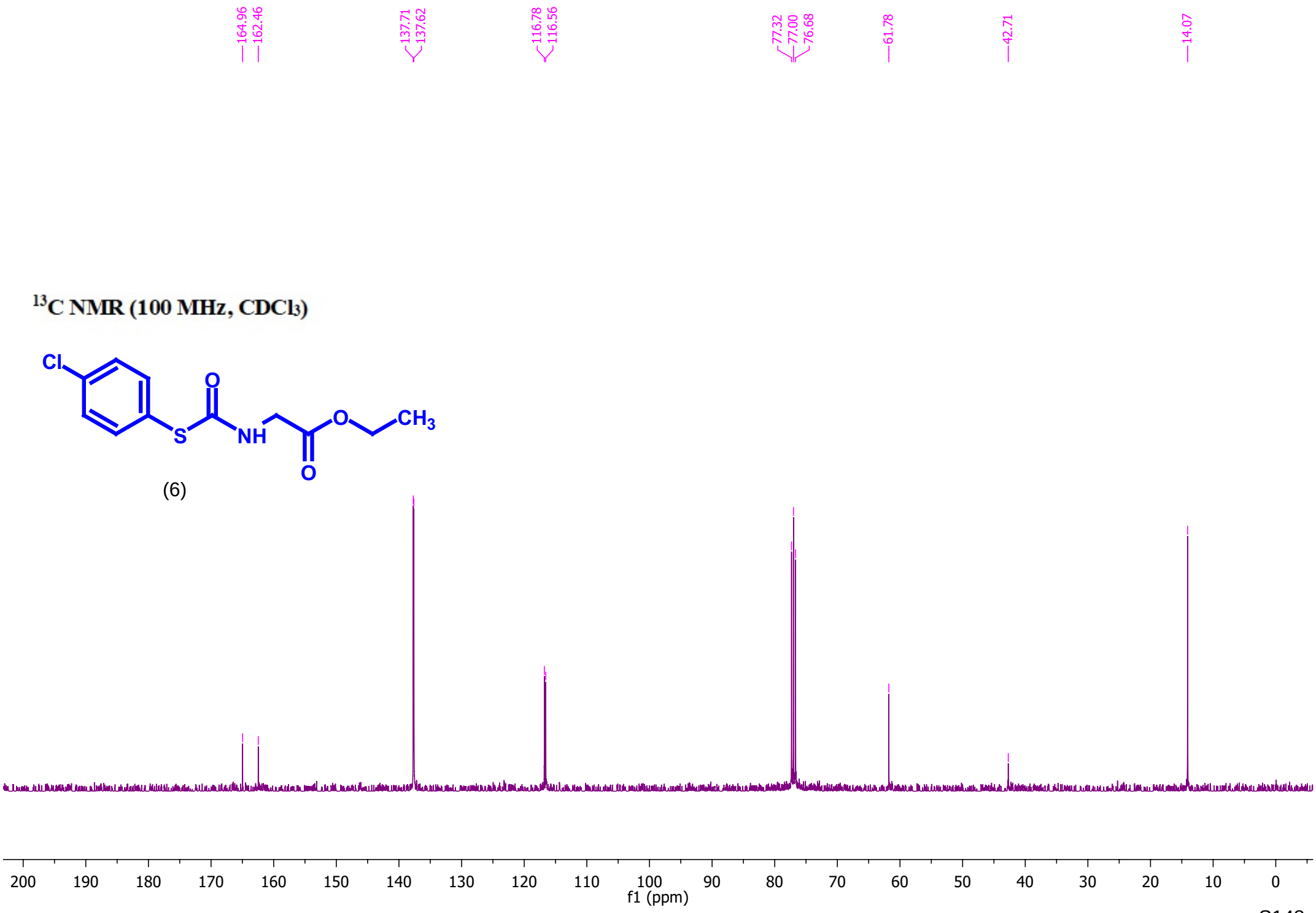


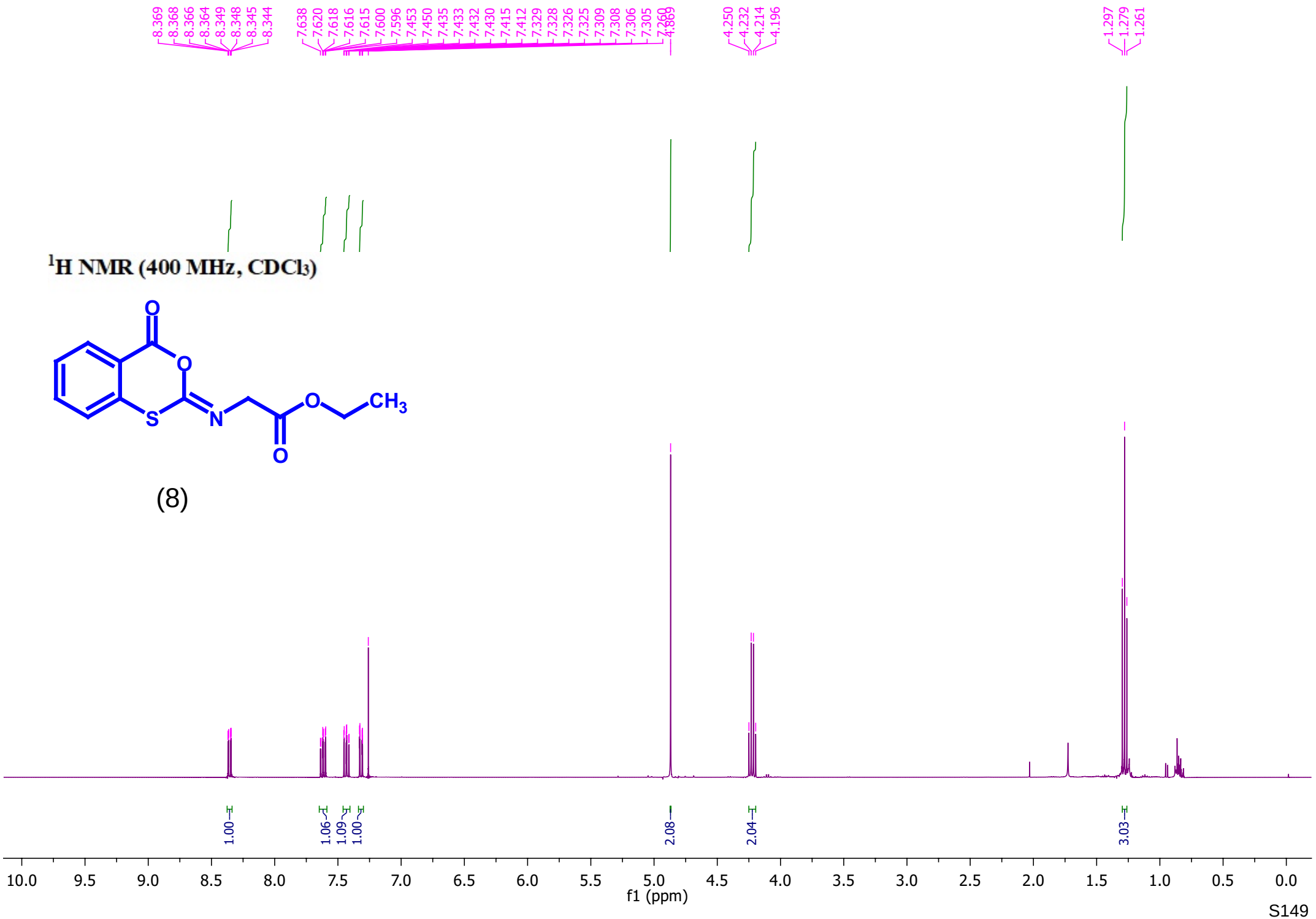
(4zu)


7.632
7.628
7.616
7.612
7.361
7.358
7.353
7.345
7.338
7.335
7.333
7.330
7.323
7.315
7.309
7.307
7.260
7.229
7.227
7.225
7.223
7.210
7.208
7.204
7.203
7.202
2.416
2.381
1H NMR (400 MHz, CDCl3)
Cl
S
N
O
O
H3C
CH3
H3C
(4zv)
1.96
5.00
4.03
6.07
3.00
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)

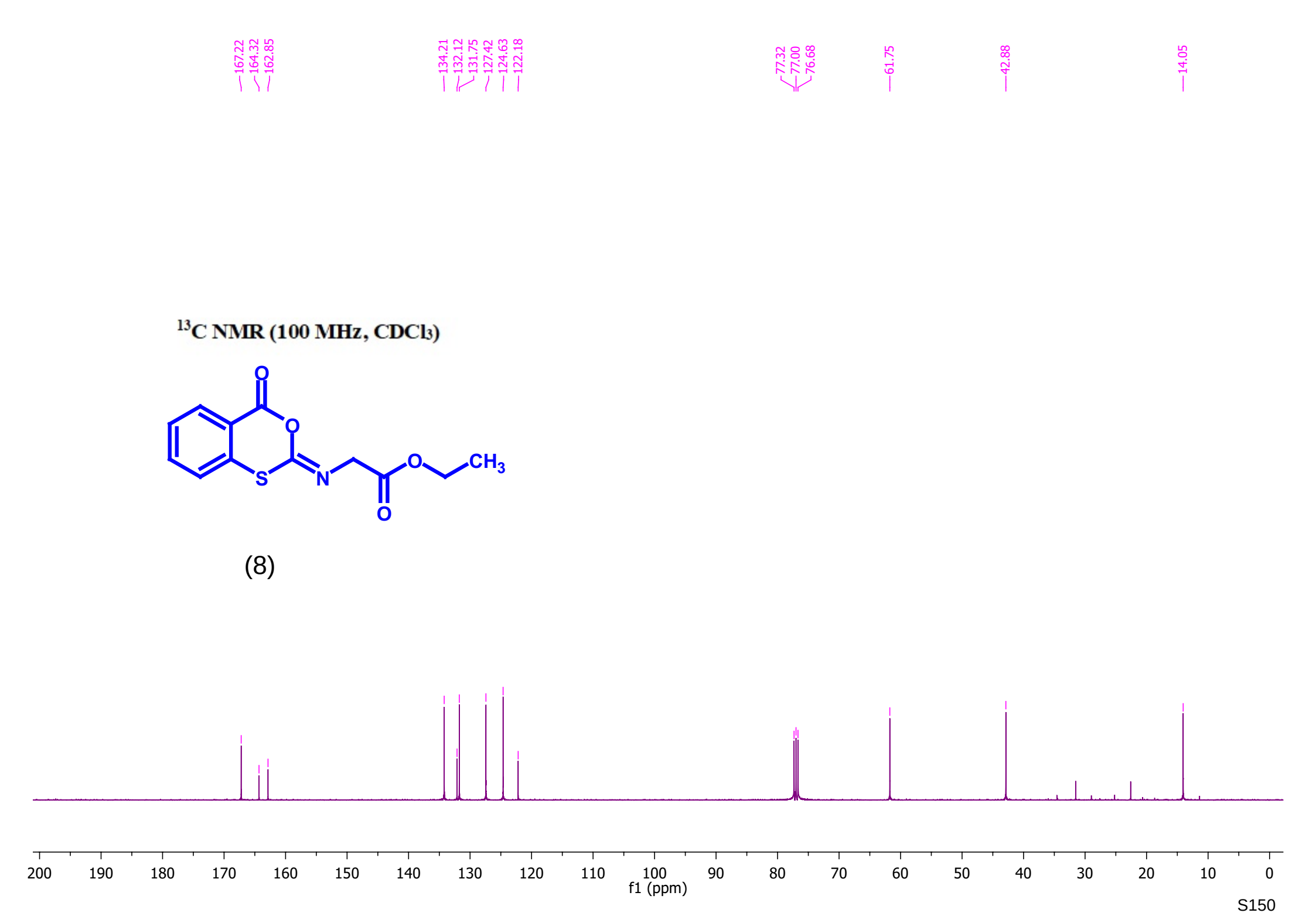

170.19
169.93
142.70
137.84
136.13
135.99
135.12
131.87
130.05
129.33
128.98
128.89
128.53
126.39
77.32
77.00
76.68
21.61
18.38
^{13}C NMR (100 MHz, CDCl$_3$)
Cl
S
N
O
O
H$_3$C
CH$_3$
H$_3$C
(4zv)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.560
7.547
7.538
7.525
7.260
7.132
7.110
7.088
5.929
4.234
4.216
4.199
4.181
4.041
4.029
1.291
1.273
1.255
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
O
S
NH
O
O
CH$_3$
(6)
2.00
2.05
0.99
2.09
2.07
3.09
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)


164.96
162.46
137.71
137.62
116.78
116.56
77.32
77.00
76.68
61.78
42.71
14.07
^{13}C NMR (100 MHz, CDCl$_3$)
Cl
O
S
NH
O
O
CH$_3$
(6)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.369
8.368
8.366
8.364
8.349
8.348
8.345
8.344
7.638
7.620
7.618
7.616
7.615
7.600
7.596
7.453
7.450
7.435
7.433
7.432
7.430
7.415
7.412
7.329
7.328
7.326
7.325
7.309
7.308
7.306
7.305
7.260
4.869
4.250
4.232
4.214
4.196
1.297
1.279
1.261
1H NMR (400 MHz, CDCl3)
O
O
S
N
O
O
CH3
1.00
1.06
1.09
1.00
2.08
2.04
3.03
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)

(8)



^{13}C NMR (100 MHz, $CDCl_3$)

(8)