

This version of the ESI replaces the one published on 04.03.2024 as the NMR spectra were missing from the previous version.

Supporting Information for

Catalyst-free Activation of N-C(O) Amide Bonds – Efficient cascade Synthesis of *N*-acyl thiocarbamides in Batch and Continuous-Flow

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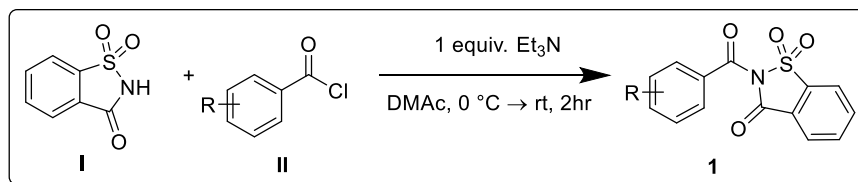
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1. General information

All chemicals were purchased from commercial providers (Sigma Aldrich, Alfa Aesar, TCI, and matrix scientific) and used directly without further purification, unless otherwise noted. Well cleaned and oven dried glassware was used for the experiments. Reaction was monitored by Thin Layer Chromatography (TLC), purchased as pre-coated with silica gel 60 F254 from Merck. Column chromatography was carried out using silica gel 230-400 mesh (purchased from Merck) with mixture of ethyl acetate/hexane or hexane as the eluent. ^1H NMR spectra were recorded on 400 MHz, ^{13}C -NMR spectra were recorded on 100 MHz, Varian mercury spectrometer using CDCl_3 or $\text{DMSO}-d_6$ as solvent. The spectra were recorded and presented in chemical shifts (ppm) with tetramethylsilane (TMS) used as internal standard. Multiplicities were provided in s (singlet), d (doublet), t (triplet), q (quartet), bs (broad singlet), m (multiplet) and dd (doublet of doublet). Coupling constants (J) were reported in Hz. All the compounds were characterized by EI mass on Thermo Finnigan (TRACEGC- POLARISQ) and HRMS (EI^+ mode) on JMS-700 spectrometer. Melting points were determined using Fargo instruments. Micromixer (500 μm) was purchased from Sanko Seiki company, stainless tube, peek and back pressure regulator were purchased from Idex health & science. Harvard syringe pump model-11 elite were used for the reaction.

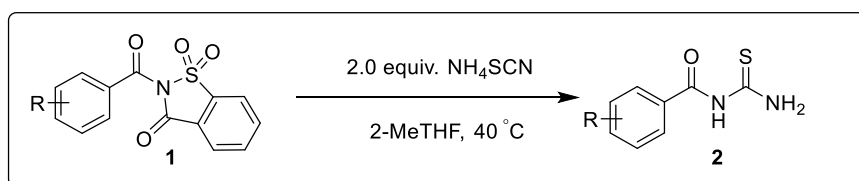
2. General procedure for the synthesis of starting materials (1a-p)



Scheme S1 General procedures for the synthesis of starting materials

An oven-dried round-bottomed flask (25 mL) equipped with a stir bar was charged with saccharin (**I**) (5 mmol, 1.0 equiv.), triethylamine (1.0 equiv.) and *N,N*-dimethylacetamide (5 mL), placed under nitrogen, and subjected to three evacuation/backfilling cycles under high vacuum. Acyl chloride (**II**) (1.0 equiv.) was added dropwise to the reaction mixture with vigorous stirring at 0 °C, and the reaction mixture was stirred 2 hr at room temperature. After the indicated time, the reaction mixture was diluted with H₂O (5 mL). The solid was collected by filtration, washed with Et₂O (1 x 10 mL), and dried. The crude product was purified by recrystallization (methanol or toluene) to give analytically pure product. All the data were matched with previous literature.¹⁻³

3. General procedure for the synthesis of *N*-acyl thiocarbamides (2a-2p)

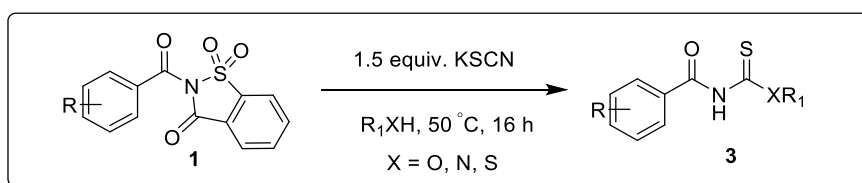


Scheme S2 General procedure for the synthesis of *N*-acyl thiocarbamides

An oven-dried vial was charged with **1a-1p** (0.2 mmol, 1.0 equiv.) and ammonium thiocyanate (0.4 mmol, 2.0 equiv.) in 2-methyltetrahydrofuran (0.2 M). The resulting mixture was allowed to stir at 40 °C in a close cap for 16-24h. After completion of reaction extracted with ethyl acetate (3 mL x 5) in water, organic layer combined and

washed with brine solution (5 mL). The organic layer was dried over anhydrous MgSO_4 , filtered, and concentrated under vacuum. The crude product was purified by column chromatography (Hexane/EtOAc, 19/1, silica gel) and obtained as solid up to 65-93% yields. Further, the obtained desired products were characterized by NMR, HRMS, Melting point and the data are shown given below.

4. General procedure for synthesis of *O*-alkyl benzoylcarbamothioates (3a-3ah)



Scheme S3 General procedure for the synthesis of *O*-alkyl benzoylcarbamothioates

An oven-dried vial was charged with **1a-1f**, **3aa-3ah** (0.2 mmol, 1.0 equiv.) and potassium thiocyanate (0.3 mmol, 1.5 equiv.) with various alcohol (0.2 M). The resulting mixture was allowed to stir at 50 °C in a close cap for 16h. After completion of the reaction was concentrated under vacuum to remove the solvent. The crude product was directly purified by column chromatography (Hexane/EtOAc, 19/1, silica gel) and obtained as solid up to 68-87% yields. Further, the obtained desired products were characterized by NMR, HRMS, Melting point and the data are shown given below. However, when the reaction of **1a**, potassium thiocyanate with butanethiol (0.2 M) or isopropyl amine (0.2 M) instead of the alcohol under the standard condition, the reaction was failed.

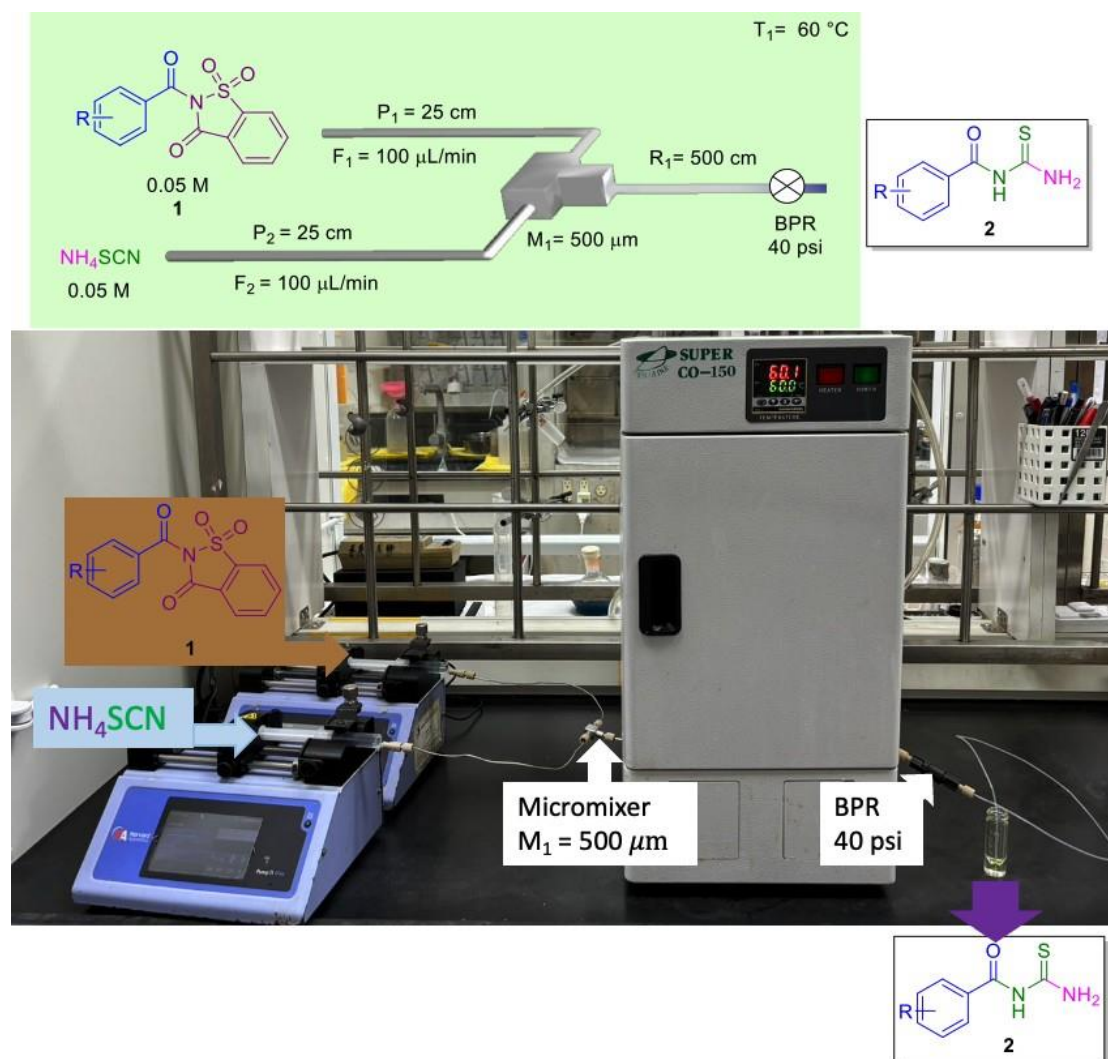
5. General procedure for the synthesis of *N*-acyl thiocarbamides in a continuous flow microreactor system.

A microreactor system consists of one T-shaped micromixer (M_1), one microtube reaction (R_1), two precooling units P_1 (inner diameter $\Phi = 800 \mu\text{m}$, length $L = 25 \text{ cm}$),

P₂ (Φ = 800 μ m, length L = 25 cm) and back pressure regulator (40 psi) were used. (**Scheme S4**). A solution of **1** (0.05 M in 2-MeTHF) (flow rate: F_1 = 100 μ L/min) and a solution of NH₄SCN (0.05 M in acetone) (flow rate: F_2 = 100 μ L/min) were introduced into **M₁** (Φ = 500 μ m) by syringe pumps. The resulting solution was passed through microtubing (**R₁** = 500 cm, Φ = 500 μ m) with a reaction temperature of 60°C. After a steady state was reached, the solution was collected for 30 minutes in a vial. The resulting solution was extracted with ethyl acetate (3 mL x 5), and the organic layer was combined and washed with brine solution (5 mL). The organic layer was dried over anhydrous MgSO₄, filtered, and concentrated under a vacuum. The crude product was purified by column chromatography (Hexane/EtOAc, 19/1, silica gel) and obtained **2** in 70-92% yields.

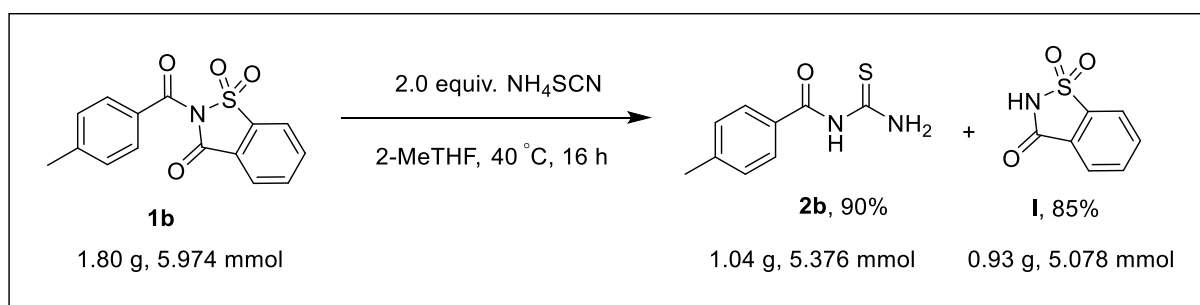
5.1 Scale up synthesis of **2a** in a continuous flow system.

The scale up synthesis of **2a** was conducted by applying the solution of **1** (0.1 M in 2-MeTHF) (flow rate: F_1 = 100 μ L/min) and a solution of NH₄SCN (0.1 M in acetone) (flow rate: F_2 = 100 μ L/min) into the same flow system. After a steady state was reached, the solution was collected for 120 minutes in a vial. With the same purification procedure, the final product **2a** was obtained as a white solid (191 mg, 88%).



Scheme S4. General procedure for the synthesis of *N*-acyl thiocarbamides in continuous-flow method and the photo of the continuous flow system.

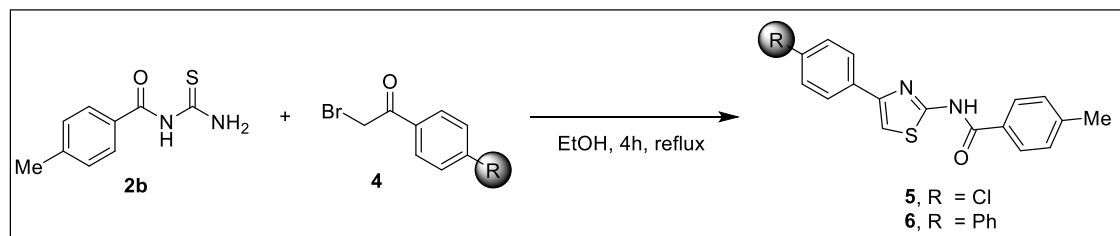
6. General procedure for gram-scale reaction



Scheme S5 General experimental procedure for gram-scale reaction

An oven-dried round-bottomed flask (100 mL) equipped with a stir bar was charged with **1b** (5.98 mmol, 1.0 equiv.) and ammonium thiocyanate (11.95 mmol, 2.0 equiv.) in 2-methyltetrahydrofuran (0.2 M). The resulting mixture was allowed to stir for 16h at 40 °C. The solvent was removed under reduced pressure to give a yellow solid. CH₂Cl₂ (8 mL) was added, and the mixture was sonicated for 3 min. The solid was collected on a glass frit, washed thoroughly with CH₂Cl₂, and dried in a high vacuum to give pure saccharin (**1**) as a white solid (0.63 g). The filtrate (yellow solution) was loaded onto silica gel and purified by column chromatography (Hexane/EtOAc, 19/1), after complete elution of *N*-carbamothioyl-4-methylbenzamide **2b**, the gradient was increased to 50% EtOAc in hexane to elute remaining saccharin). Saccharin was obtained after column chromatography was washed CH₂Cl₂ to give an off-white solid (0.30 g). *N*-carbamothioyl-4-methylbenzamide **2b** was obtained as a white solid (1.04 g, 90%).

7. Synthetic procedure for 5 and 6

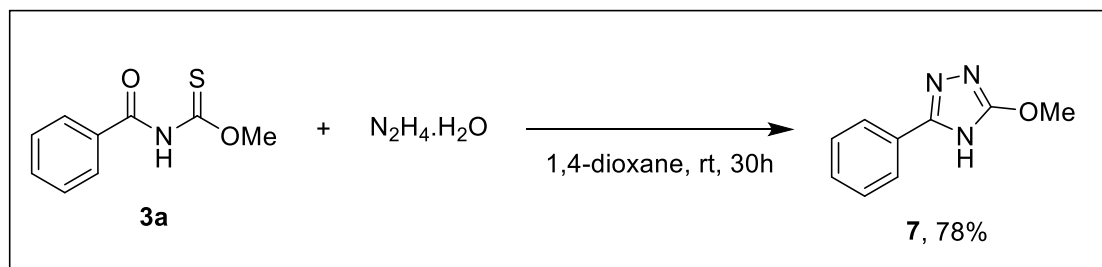


Scheme S6 General experimental procedure for the synthesis of 5 and 6

An oven-dried vial was charged with **2b** (0.2 mmol, 1.0 equiv.) and 2-bromoacetophenone derivatives **4** (0.2 mmol, 1.0 equiv.) in ethanol (0.1 M). The resulting mixture was allowed to stir at reflux in a closed vial for 4h. After the completion of the reaction, the mixture was cooled to room temperature and the resulting white solid was filtered off and thoroughly dried. The obtained white solids

(**5** and **6**) were characterized by NMR, HRMS, Melting point and the data are shown given below.

8. Synthetic procedure for **7**

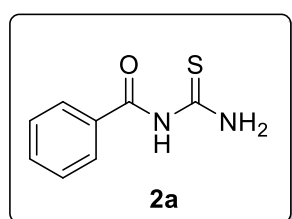


Scheme S7 General experimental procedure for the synthesis of **7**

An oven-dried vial was charged with **3a** (0.2 mmol, 1.0 equiv.) and hydrazine monohydrate (0.6 mmol, 3.0 equiv.) in 1,4-dioxane (0.1 M). The resulting mixture was allowed to stir at room temperature in a close cap for 30h. After completion of reaction extracted with ethyl acetate (3 mL x 5), organic layer combined and washed with brine solution (5 mL). The organic layer was dried over anhydrous MgSO_4 , filtered, and concentrated under vacuum. The crude product was purified by column chromatography (Hexane/EtOAc, 19/1, silica gel) and obtained as solid up to 78% yield. Further, the obtained desired product was characterized by NMR, HRMS, Melting point and the data are shown given below.

9. Characterization data:

N-carbamothioylbenzamide (2a). ^{4, 5} The title compound was synthesized according

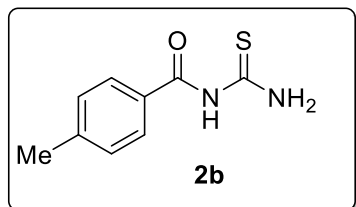


to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (31 mg, 85%); Mp.173-176 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ

11.23 (bs, 1H), 9.85 (bs, 1H), 9.56 (bs, 1H), 7.92 (d, *J* = 8.0 Hz, 2H), 7.63 (t, *J* = 7.6 Hz,

1H), 7.50 (t, $J = 8.0$ Hz, 2H) ; ^{13}C -NMR (100 MHz, $\text{DMSO}-d_6$) δ 182.5, 168.2, 133.4, 132.7, 129.0, 128.8.

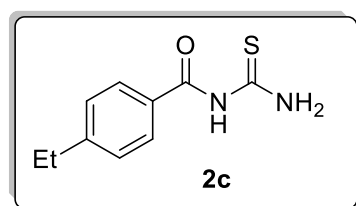
***N*-carbamothioyl-4-methylbenzamide (2b).** ⁶ The title compound was synthesized



according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (36 mg, 93%);

Mp. 218–223 °C; ^1H -NMR (400 MHz, $\text{DMSO}-d_6$) δ 11.10 (bs, 1H), 9.86 (bs, 1H), 9.51 (bs, 1H), 7.85 (d, $J = 8.2$ Hz, 2H), 7.31 (d, $J = 8.1$ Hz, 2H), 2.38 (s, 3H) ; ^{13}C -NMR (100 MHz, $\text{DMSO}-d_6$) δ 182.6, 168.0, 143.9, 129.8, 129.4, 129.1, 21.55.

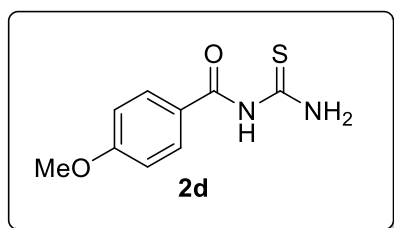
***N*-carbamothioyl-4-ethylbenzamide (2c).** The title compound was synthesized



according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (38 mg, 91%);

Mp. 210–213 °C; ^1H -NMR (400 MHz, $\text{DMSO}-d_6$) δ 11.06 (bs, 1H), 9.83 (bs, 1H), 9.47 (bs, 1H), 7.84 (d, $J = 8.4$ Hz, 2H), 7.31 (d, $J = 8.4$ Hz, 2H), 2.64 (q, $J = 7.6$ Hz, 2H), 1.16 (t, $J = 7.6$ Hz, 3H) ; ^{13}C -NMR (100 MHz, $\text{DMSO}-d_6$) δ 182.5, 168.0, 149.9, 130.0, 129.2, 128.3, 28.55, 15.60. HRMS (EI) m/z $[M]^+$ calcd for $\text{C}_{10}\text{H}_{12}\text{N}_2\text{OS}$, 208.0670; found, 208.0673.

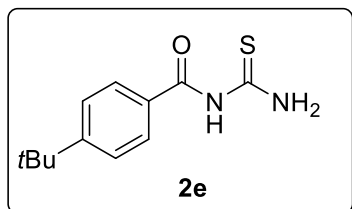
***N*-carbamothioyl-4-methoxybenzamide (2d).** ^{6, 7} The title compound was



synthesized according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a semi solid

(39 mg, 92%); $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 11.06 (bs, 1H), 9.89 (bs, 1H), 9.49 (bs, 1H), 7.96 (d, $J = 9.0$ Hz, 2H), 7.03 (d, $J = 9.0$ Hz, 2H), 3.84 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 182.0, 166.8, 162.9, 130.7, 123.9, 113.5, 55.8.

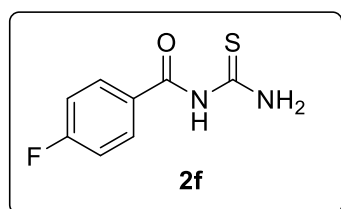
4-(tert-butyl)-*N*-carbamothioylbenzamide (2e). The title compound was synthesized



according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane

(1:19) and obtained as a white solid (41 mg, 88%); Mp. 209-211 °C; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 11.11 (bs, 1H), 9.88 (bs, 1H), 9.53 (bs, 1H), 7.89 (d, $J = 8.6$ Hz, 2H), 7.53 (d, $J = 8.7$ Hz, 2H), 1.30 (s, 9H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 182.6, 168.0, 156.6, 129.9, 129.0, 125.7, 35.3, 31.3. HRMS (EI) m/z $[M]^+$ calcd for $\text{C}_{12}\text{H}_{16}\text{N}_2\text{OS}$, 236.0983; found, 236.0983.

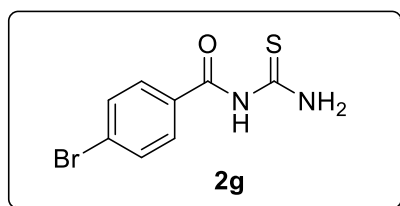
***N*-carbamothioyl-4-fluorobenzamide (2f).** ⁶ The title compound was synthesized



according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (36 mg, 90%);

Mp. 203-206 °C; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 11.28 (bs, 1H), 9.81 (bs, 1H), 9.54 (bs, 1H), 8.01 (dd, $J = 5.5$ Hz, 3.1 Hz, 2H), 7.34 (t, $J = 8.7$ Hz, 2H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 182.5, 167.1, 165.3 (d, $J = 249.5$ Hz), 132.1, 132.0 (d, $J = 9.4$ Hz), 115.9 (d, $J = 22.0$ Hz). ^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) δ -106.40.

4-bromo-*N*-carbamothioylbenzamide (2g). ⁶ The title compound was synthesized

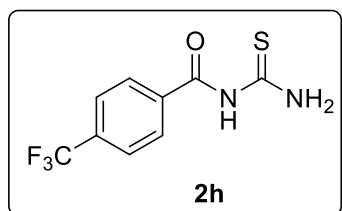


according to the general procedure (Scheme S2).

The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid

(45 mg, 87%); Mp.232-235 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 11.33 (bs, 1H), 9.78 (bs, 1H), 9.56 (bs, 1H), 7.85 (d, *J* = 8.6 Hz, 2H), 7.71 (d, *J* = 8.3 Hz, 2H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 182.4, 167.4, 131.9, 131.8, 131.1, 127.3.

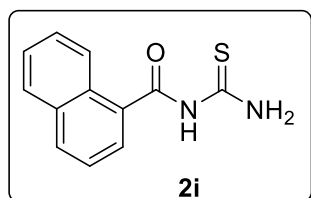
***N*-carbamothioyl-4-(trifluoromethyl)benzamide (2h).** The title compound was



synthesized according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (41

mg, 83%); Mp.231-236 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 11.51 (bs, 1H), 9.77 (bs, 1H), 9.63 (bs, 1H), 8.09 (d, *J* = 8.2 Hz, 2H), 7.87 (d, *J* = 8.2 Hz, 2H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 182.3, 167.2, 136.8, 132.8 (d, *J* = 31.8 Hz), 130.0, 125.7 (q, *J* = 3.7 Hz), 122.8 (q, *J* = 279.4). ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -61.58; HRMS (EI) *m/z* [*M*]⁺ calcd for C₉H₇F₃N₂OS, 248.0231; found, 248.0232.

***N*-carbamothioyl-1-naphthamide (2i).** ⁶ The title compound was synthesized

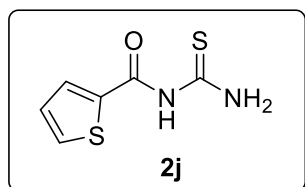


according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (43 mg, 93%); Mp.198-202 °C; ¹H-NMR (400

MHz, DMSO-*d*₆) δ 11.60 (bs, 1H), 9.86 (bs, 1H), 9.60 (bs, 1H), 8.10 (d, *J* = 8.6 Hz, 2H),

8.03-8.00 (m, 1H), 7.73 (d, $J = 7.0$ Hz, 1H), 7.65-7.55 (m, 3H); ^{13}C -NMR (100 MHz, DMSO- d_6) δ 182.8, 170.0, 133.4, 132.0, 131.8, 129.9, 128.9, 127.8, 127.4, 126.9, 125.2, 125.0.

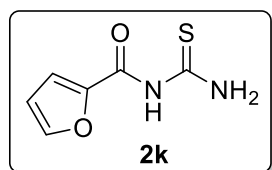
***N*-carbamothioylthiophene-2-carboxamide (2j).** ⁵ The title compound was



synthesized according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19)

and obtained as a white solid (28 mg, 75%); Mp.191-193 °C; ^1H -NMR (400 MHz, DMSO- d_6) δ 11.28 (bs, 1H), 9.72 (bs, 1H), 9.50 (bs, 1H), 8.33 (d, $J = 4.0$ Hz, 1H), 8.01 (d, $J = 4.9$ Hz, 1H), 7.22 (t, $J = 4.4$ Hz, 1H); ^{13}C -NMR (100 MHz, DMSO- d_6) δ 182.2, 162.1, 137.4, 135.5, 132.9, 129.1.

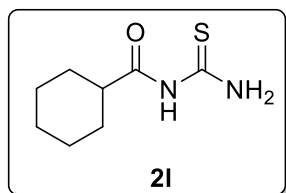
***N*-carbamothioylfuran-2-carboxamide (2k).** ⁵ The title compound was synthesized



according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a

white solid (27 mg, 78%); Mp.198-200 °C; ^1H -NMR (400 MHz, DMSO- d_6) δ 10.95 (bs, 1H), 9.66 (bs, 1H), 9.53 (bs, 1H), 8.03 (s, 1H), 7.79 (s, 1H), 6.73-6.72 (m, 1H); ^{13}C -NMR (100 MHz, DMSO- d_6) δ 182.6, 158.3, 149.3, 145.9, 119.3, 113.6.

***N*-carbamothioylcyclohexanecarboxamide (2l).** ⁸ The title compound was synthesized

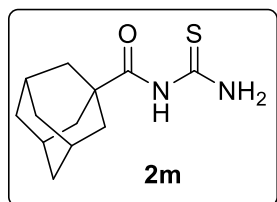


according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a

white solid (25 mg, 68%); Mp.226-228 °C; ^1H -NMR (400 MHz, DMSO- d_6) δ 10.97 (bs, 1H), 9.65 (bs, 1H), 9.30 (bs, 1H), 2.46-2.43 (m, 1H), 1.75-1.69 (m, 4H), 1.62-1.60 (m,

1H), 1.33-1.11 (m, 5H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 182.5, 178.0, 44.26, 29.05, 25.64, 25.38.

(1*s*,3*s*)-*N*-carbamothioyladamantane-1-carboxamide (2m). The title compound was

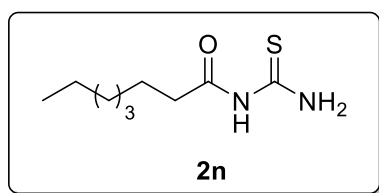


synthesized according to the general procedure (Scheme S2).

The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a

white solid (47 mg, 83%); Mp.205-210 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 10.12 (bs, 1H), 9.72 (bs, 1H), 9.33 (bs, 1H), 1.94 (s, 3H), 1.84 (s,6H), 1.63 (t, *J* = 15.48 Hz, 6H) ; ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 182.6, 179.3, 41.9, 37.3, 36.0, 27.8. HRMS (EI) *m/z* [M]⁺ calcd for C₁₂H₁₈N₂OS, 238.1140 ; found, 238.1144.

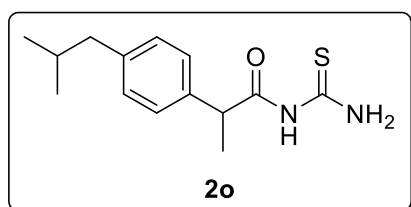
***N*-carbamothioylnonanamide (2n).** ⁹The title compound was synthesized according to



the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (30 mg, 70%); Mp.171-174

°C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 11.01 (bs, 1H), 9.63 (bs, 1H), 9.31 (bs, 1H), 2.33 (t, *J* = 7.4 Hz, 2H), 1.48 (t, *J* = 7.6 Hz, 2H), 1.26-1.22 (m, 9H), 0.86-0.82 (m, 3H) ; ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 182.1, 175.2, 36.1, 31.5, 28.8, 28.8, 24.7, 22.5, 14.4.

***N*-carbamothioyl-2-(4-isobutylphenyl)propanamide (2o).** The title compound was

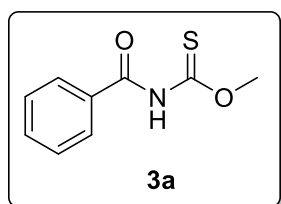


synthesized according to the general procedure (Scheme S2). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a semi solid

(34 mg, 65%); ¹H-NMR (400 MHz, DMSO-*d*₆) δ 11.16 (bs, 1H), 9.58 (bs, 1H), 9.37 (bs,

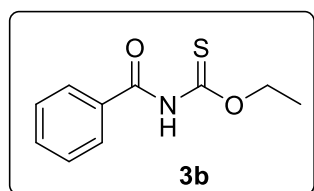
1H), 7.22 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 8.1$ Hz, 2H), 3.94 (q, $J = 7.2$ Hz, 1H), 2.39 (d, $J = 7.1$ Hz, 2H), 1.82-1.75 (m, 1H), 1.31 (d, $J = 7.0$ Hz, 3H), 0.83 (d, $J = 7.8$ Hz, 6H); ^{13}C -NMR (100 MHz, DMSO- d_6) δ 182.3, 176.0, 140.4, 138.1, 129.5, 127.6, 45.0, 44.7, 30.0, 22.6, 18.5. HRMS (EI) m/z $[M]^+$ calcd for $\text{C}_{14}\text{H}_{20}\text{N}_2\text{OS}$, 264.1296; found, 264.1295.

O-methyl benzoylcarbamothioate (3a). The title compound was synthesized



according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (32 mg, 81%); Mp.109-112 °C; ^1H -NMR (400 MHz, DMSO- d_6) δ 11.97 (bs, 1H), 7.85-7.83 (m, 2H), 7.63-7.59 (m, 1H), 7.51-7.47 (m, 2H), 4.03 (s, 3H); ^{13}C -NMR (100 MHz, DMSO- d_6) δ 190.8, 165.5, 133.6, 133.3, 129.1, 128.9, 58.3.

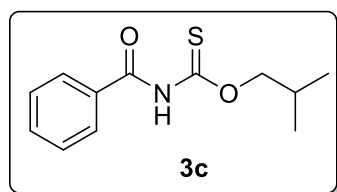
O-ethyl benzoylcarbamothioate (3b). ¹⁰, ¹¹ The title compound was synthesized



according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a

white solid (35 mg, 84%); Mp.80-83 °C; ^1H -NMR (400 MHz, DMSO- d_6) δ 11.90 (bs, 1H), 7.85 (d, $J = 7.8$ Hz, 2H), 7.62 (t, $J = 7.1$ Hz, 1H), 7.50 (t, $J = 7.4$ Hz, 2H), 4.53 (q, $J = 7.0$ Hz, 2H), 1.34-1.22 (m, 3H); ^{13}C -NMR (100 MHz, DMSO- d_6) δ 190.1, 165.6, 133.7, 133.2, 129.1, 128.8, 67.8, 14.0.

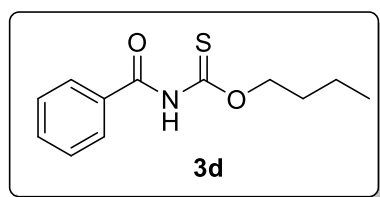
O-isobutyl benzoylcarbamothioate (3c). The title compound was synthesized



according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane

(1:19) and obtained as a semi solid (40 mg, 85%); $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 11.91 (bs, 1H), 7.83 (d, $J = 7.1$ Hz, 2H), 7.61 (t, $J = 7.4$ Hz, 1H), 7.49 (t, $J = 7.9$ Hz, 2H), 4.24 (d, $J = 6.5$ Hz, 2H), 2.05-1.95 (m, 1H), 0.93 (d, $J = 6.7$ Hz, 6H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 190.5, 165.4, 133.7, 133.2, 129.1, 128.8, 77.9, 27.6, 19.2. HRMS (EI) m/z $[\text{M}]^+$ calcd for $\text{C}_{12}\text{H}_{15}\text{NO}_2\text{S}$, 237.0823 ; found, 237.0820.

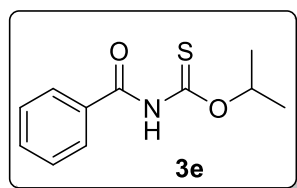
O-butyl benzoylcarbamothioate (3d). ^{12, 13} The title compound was synthesized



according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a semi solid (41 mg, 87%); $^1\text{H-}$

NMR (400 MHz, CDCl_3) δ 9.19 (bs, 1H), 7.82 (d, $J = 7.2$ Hz, 2H), 7.58 (t, $J = 7.3$ Hz, 1H), 7.48 (t, $J = 7.7$ Hz, 2H), 4.59 (t, $J = 6.6$ Hz, 2H), 1.83-1.76 (m, 2H), 1.50-1.76 (m, 2H), 0.96 (t, $J = 7.4$ Hz, 3H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 189.7, 162.5, 133.1, 132.9, 128.9, 127.6, 73.4, 30.1, 19.0, 13.6.

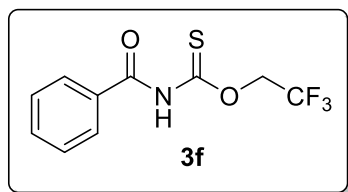
O-isopropyl benzoylcarbamothioate (3e). The title compound was synthesized



according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a

semi solid (38 mg, 85%); $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 9.05 (bs, 1H), 7.82 (d, $J = 7.5$ Hz, 2H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.49 (t, $J = 7.8$ Hz, 2H), 5.67-5.61 (m, 1H), 1.44 (d, $J = 6.2$ Hz, 6H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 184.0, 158.0, 128.5, 124.3, 123.0, 122.5, 73.3, 16.6. HRMS (EI) m/z $[\text{M}]^+$ calcd for $\text{C}_{11}\text{H}_{13}\text{NO}_2\text{S}$, 223.0667; found, 223.0666.

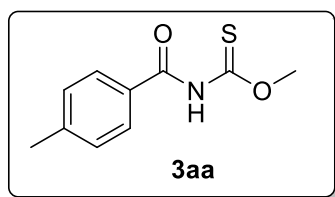
O-(2,2,2-trifluoroethyl) benzoylcarbamothioate (3f). The title compound was



synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane

(1:19) and obtained as a white solid (36 mg, 68%); Mp.97-100 °C; ¹H-NMR (400 MHz, CDCl₃) δ 9.27 (bs, 1H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 2H), 4.90 (q, *J* = 8.0 Hz, 2H); ¹³C-NMR (100 MHz, CDCl₃) δ 185.3, 162.9, 133.5, 132.5, 129.1, 127.7, 123.9 (d, *J* = 275.6 Hz), 66.15 (q, *J* = 73.5 Hz); ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -72.91; HRMS (EI) *m/z* [M]⁺ calcd for C₁₀H₈F₃NO₂S, 263.0228; found, 263.0222.

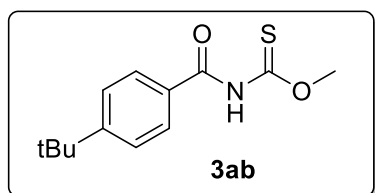
O-methyl (4-methylbenzoyl)carbamothioate (3aa). ¹⁴ The title compound was



synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (35

mg, 83%); Mp.138–140 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 11.91 (bs, 1H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 4.02 (s, 3H), 2.36 (s, 3H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 190.9, 165.2, 143.7, 130.7, 129.4, 129.2, 58.3, 21.5.

O-methyl (4-(tert-butyl)benzoyl)carbamothioate (3ab). The title compound was

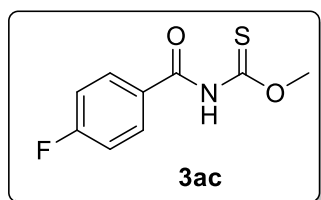


synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a semi solid (37

mg, 74%); ¹H-NMR (400 MHz, CDCl₃) δ 9.19 (bs, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 4.21 (s, 3H), 1.34 (s, 9H); ¹³C-NMR (100 MHz, CDCl₃) δ 190.5, 162.2, 157.2,

129.8, 127.6, 126.0, 59.6, 35.2, 31.0. HRMS (EI) m/z $[M]^+$ calcd for $C_{13}H_{17}NO_2S$, 251.0980; found, 251.0981.

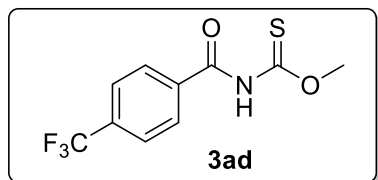
O-methyl (4-fluorobenzoyl)carbamothioate (3ac). ¹⁴ The title compound was



synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (31

mg, 72%); Mp.127-128 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 12.02 (bs, 1H), 7.92 (dd, J = 8.8 Hz, 5.2 Hz, 2H), 7.32 (t, J = 8.8 Hz, 2H), 4.02 (s, 3H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 190.7, 166.5, 164.4 (d, J = 249.4 Hz), 132.1 (d, J = 9.4 Hz), 130.1, 116.0 (d, J = 22.0 Hz), 58.4; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -106.57.

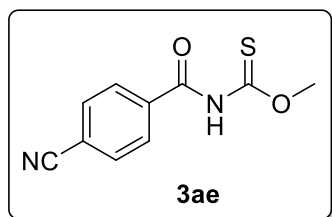
O-methyl (4-(trifluoromethyl)benzoyl)carbamothioate (3ad). The title compound



was synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid

(42 mg, 80%); Mp.105-109 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 12.21 (bs, 1H), 8.02 (d, J = 8.0 Hz, 2H), 7.86 (d, J = 8.0 Hz, 2H), 4.04 (s, 3H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 190.5, 164.6, 137.5, 132.6 (d, J = 31.8 Hz), 130.0, 125.8 (q, J = 3.8 Hz), 122.8 (s, J = 293.9 Hz), 58.5; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -63.20; HRMS (EI) m/z $[M]^+$ calcd for $C_{10}H_8F_3NO_2S$, 263.0228; found, 263.0223.

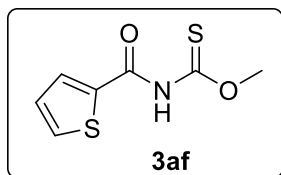
O-methyl (4-cyanobenzoyl)carbamothioate (3ae). The title compound was



synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a semi solid (33 mg, 75%); $^1\text{H-NMR}$

(400 MHz, $\text{DMSO-}d_6$) δ 12.22 (bs, 1H), 7.97 (s, 4H), 4.03 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 190.4, 164.5, 137.7, 132.8, 129.8, 118.5, 115.2, 58.5. HRMS (EI) m/z $[\text{M}]^+$ calcd for $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_2\text{S}$, 220.0306; found, 220.0303.

O-methyl (thiophene-2-carbonyl)carbamothioate (3af). The title compound was

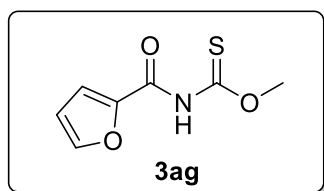


synthesized according to the general procedure (Scheme S3).

The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a

white solid (29 mg, 72%); Mp.108-110 °C; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 12.01 (bs, 1H), 8.09 (dd, J = 3.84 Hz, 1.08 Hz, 1H), 7.97 (dd, J = 5.00 Hz, 1.12 Hz, 1H), 7.19 (t, J = 4.4 Hz, 1H), 4.03 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 189.7, 169.4, 158.6, 134.7, 132.0, 128.4, 57.9. HRMS (EI) m/z $[\text{M}]^+$ calcd for $\text{C}_7\text{H}_7\text{NO}_2\text{S}_2$, 200.9918; found, 200.9916.

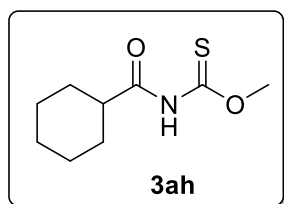
O-methyl (furan-2-carbonyl)carbamothioate (3ag). ¹⁵ The title compound was



synthesized according to the general procedure (Scheme S3). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a semi solid (26 mg, 69%); $^1\text{H-NMR}$

(400 MHz, $\text{DMSO-}d_6$) δ 11.82 (bs, 1H), 7.98 (s, 1H), 7.57 (s, 1H), 6.68 (s, 1H), 4.01 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 189.6, 170.0, 147.9, 145.7, 117.9, 112.3, 57.9.

O-methyl (cyclohexanecarbonyl)carbamothioate (3ah). ¹⁶ The title compound was

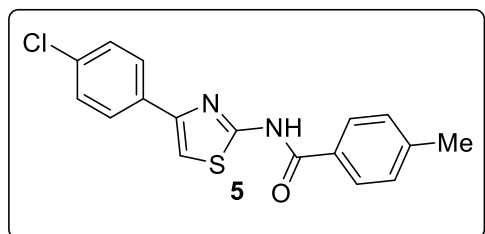


synthesized according to the general procedure (Scheme S3).

The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (27 mg, 68%); Mp.126-128 °C;

¹H-NMR (400 MHz, DMSO-*d*₆) δ 11.54 (bs, 1H), 4.00 (s, 3H), 2.44-2.39 (m, 1H), 1.76-1.69 (m, 5H), 1.29-1.15 (m, 5H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 190.0, 173.0, 57.8, 44.2, 26.8, 25.2, 24.9.

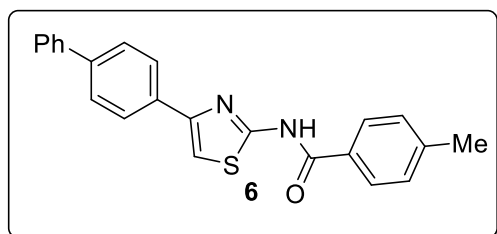
N-(4-(4-chlorophenyl)thiazol-2-yl)-4-methylbenzamide (5) The title compound was



synthesized according to the general procedure (Scheme S6). The obtained product as a white solid (62 mg, 95%); Mp.260-261 °C; ¹H-NMR (400 MHz, DMSO-*d*₆)

δ 12.69 (bs, 1H), 8.01 (d, *J* = 8.16 Hz, 2H), 7.95 (d, *J* = 8.72 Hz, 2H), 7.73 (s, 1H), 7.50 (d, *J* = 8.8 Hz, 2H), 7.35 (d, *J* = 7.9 Hz, 2H), 2.38 (s, 3H); ¹³C-NMR (100 MHz, DMSO-*d*₆) δ 165.6, 159.2, 148.3, 143.4, 133.7, 132.7, 129.6, 129.5, 129.2, 128.7, 127.9, 109.7, 21.5. HRMS (EI) *m/z* [M]⁺ calcd for C₁₇H₁₃ClN₂OS, 328.0437; found, 328.0436.

N-(4-([1,1'-biphenyl]-4-yl)thiazol-2-yl)-4-methylbenzamide (6) The title compound

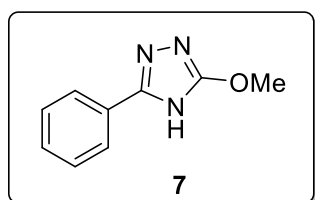


was synthesized according to the general procedure (Scheme S6). The obtained product as a white solid (69 mg, 93%); Mp.268-271 °C; ¹H-NMR (400 MHz, DMSO-*d*₆) δ 12.70 (bs, 1H), 8.03 (d, *J* = 7.0

Hz, 4H), 7.76-7.70 (m, 5H), 7.47 (t, *J* = 7.8 Hz, 2H), 7.38-7.34 (m, 3H), 2.39 (s, 3H); ¹³C-

NMR (100 MHz, DMSO- d_6) δ 165.5, 159.1, 149.2, 143.3, 140.0, 139.8, 133.9, 129.6, 129.4, 128.7, 128.0, 127.4, 126.9, 126.8, 109.1, 21.5. HRMS (EI) m/z $[M]^+$ calcd for $C_{23}H_{18}N_2OS$, 370.1140; found, 370.1131.

3-methoxy-5-phenyl-4H-1,2,4-triazole (7). The title compound was synthesized



according to the general procedure (Scheme S7). The crude mixture was purified using column chromatography by eluting with ethyl acetate/hexane (1:19) and obtained as a white solid (27 mg, 78%); Mp.158-160 °C; ^1H -NMR

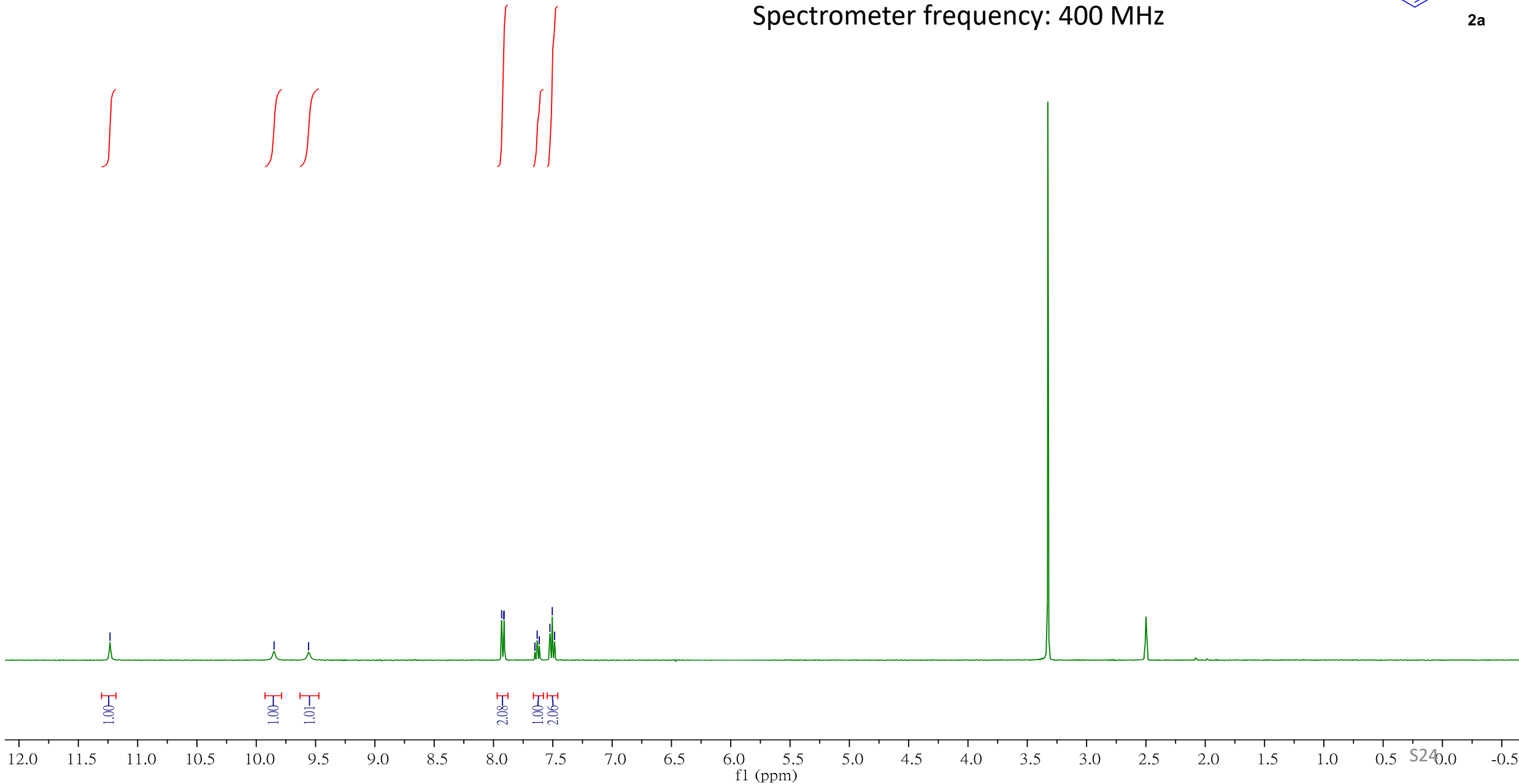
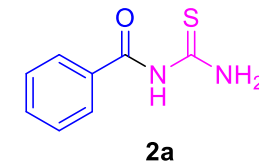
(400 MHz, $CDCl_3$) δ 7.98-7.95 (m, 2H), 7.42-7.41 (m, 3H), 6.67 (bs, 1H), 4.07 (s, 3H); ^{13}C -NMR (100 MHz, $CDCl_3$) δ 165.6, 156.0, 130.4, 128.9, 126.6, 126.3, 57.6. HRMS (EI) m/z $[M]^+$ calcd for $C_9H_9N_3O$, 175.0746; found, 175.0743.

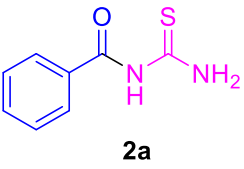
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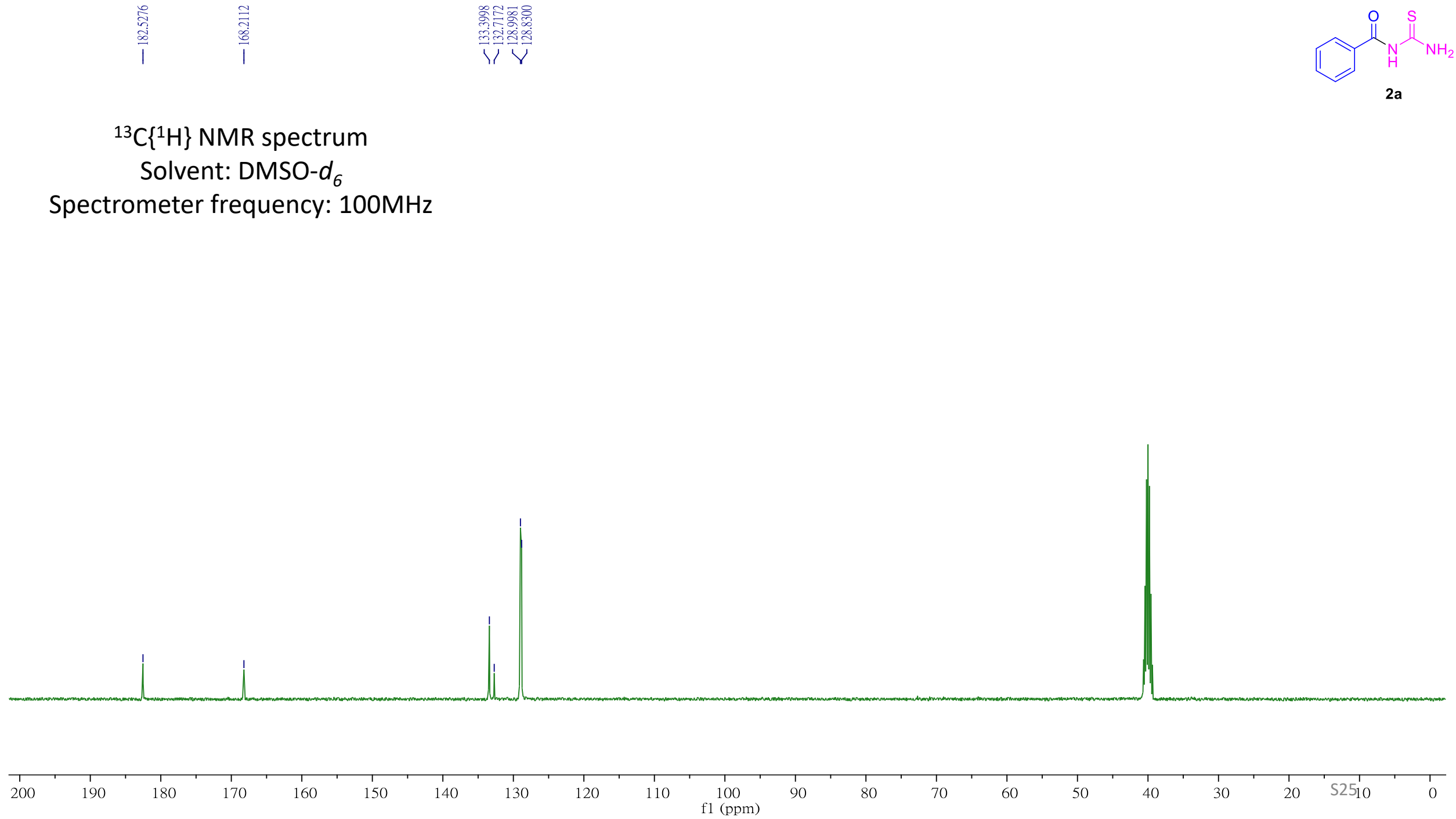
6. ^1H and ^{13}C spectra

¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz

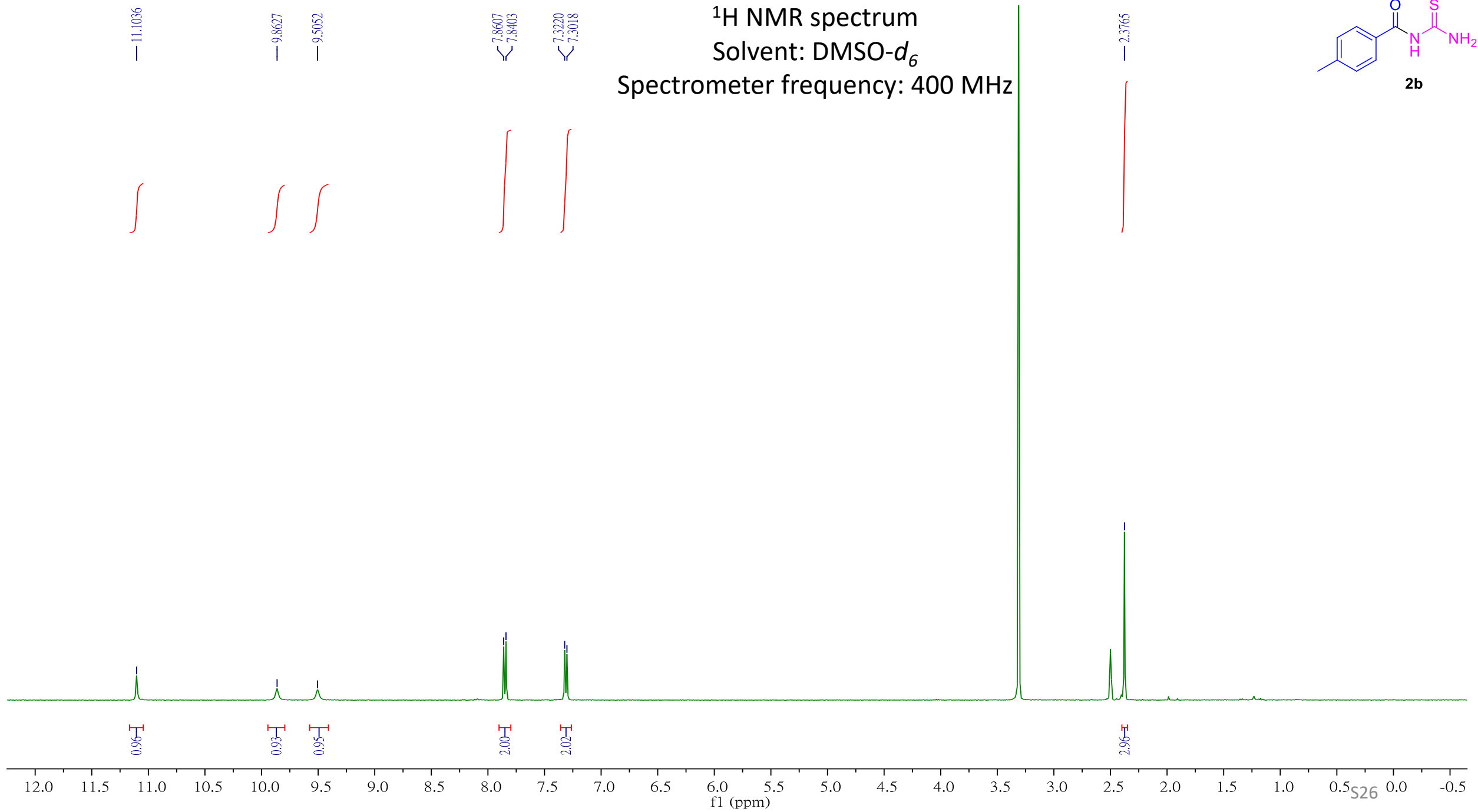
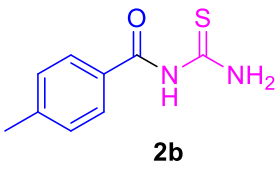




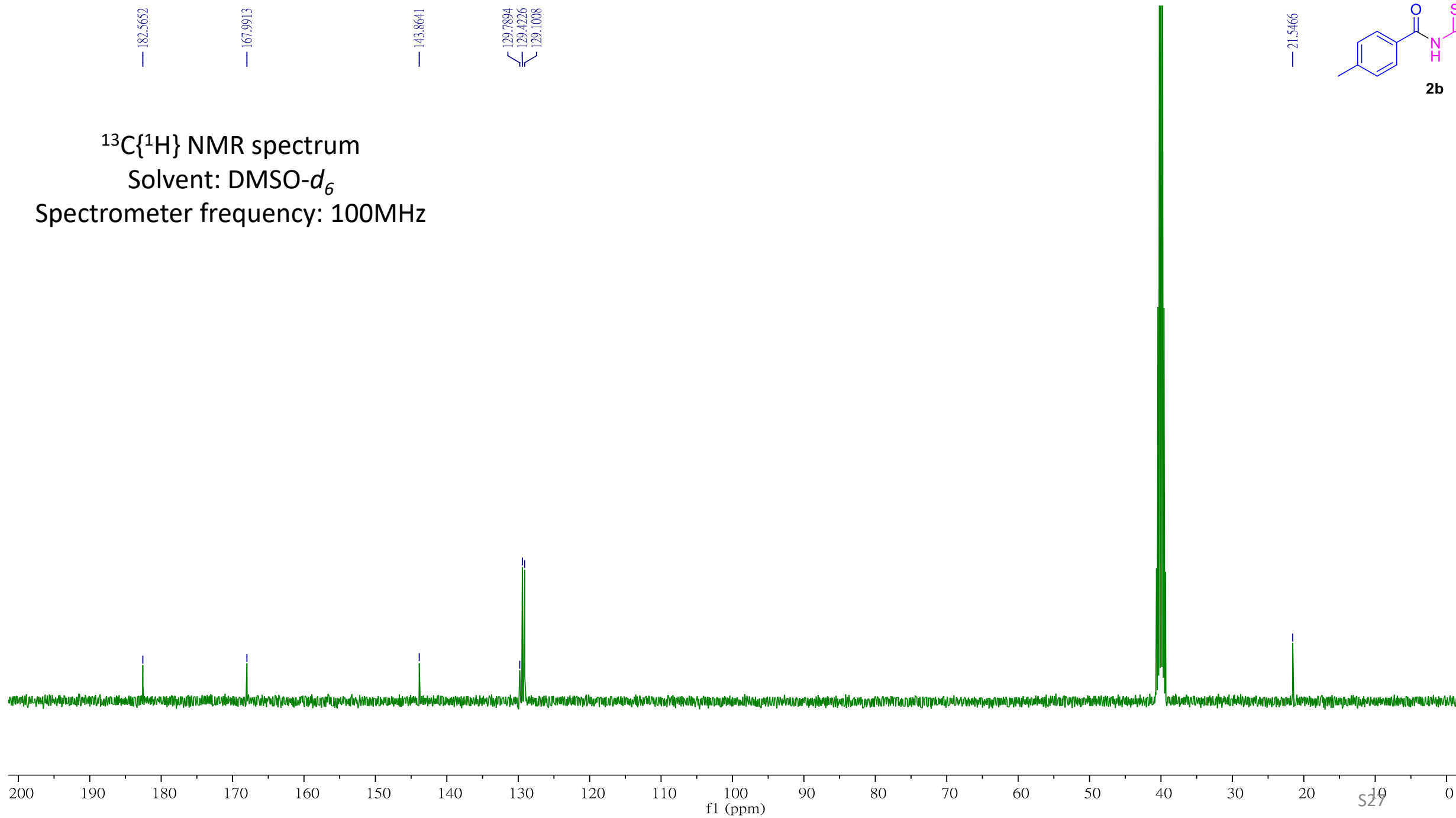
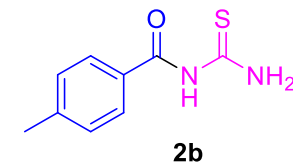
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



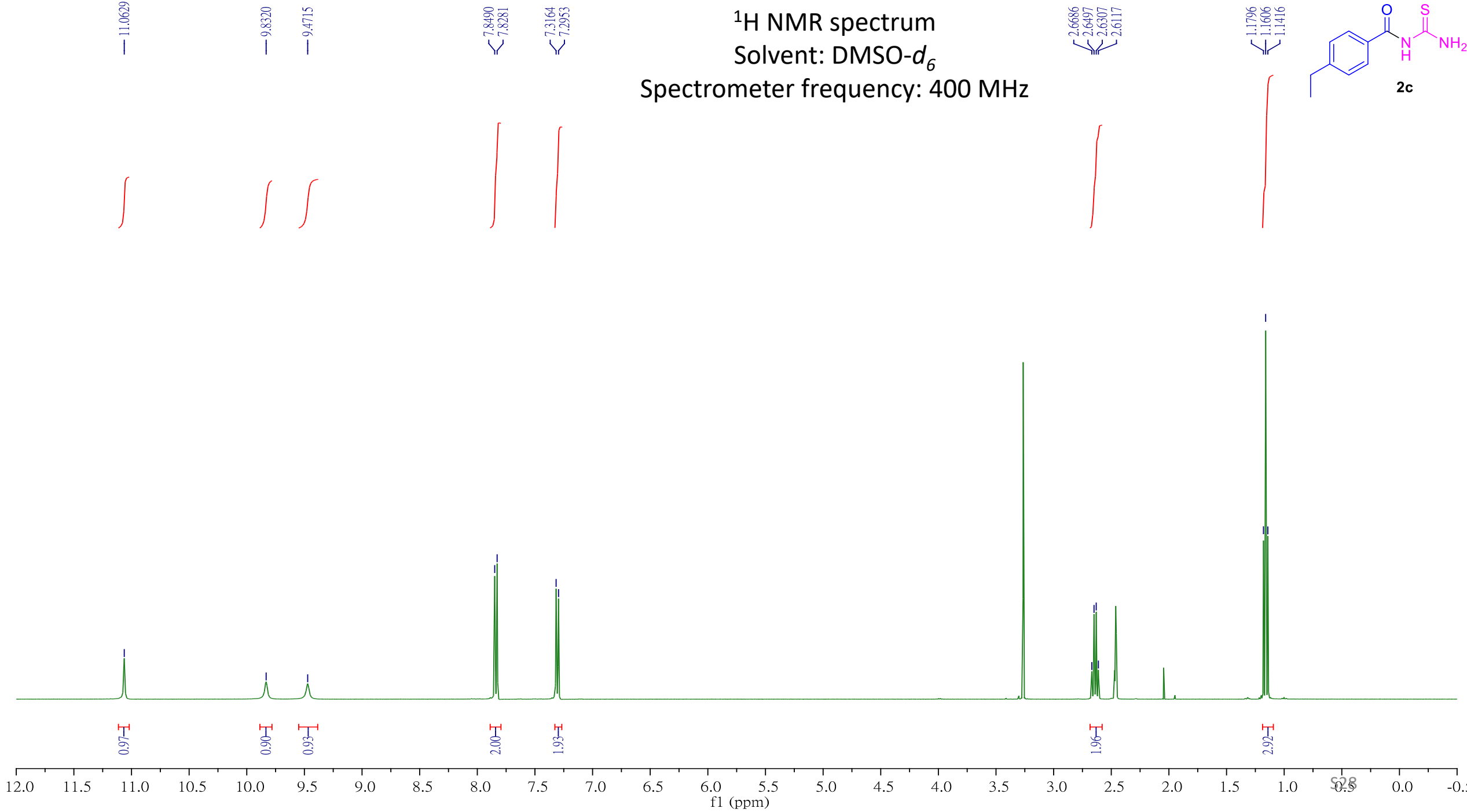
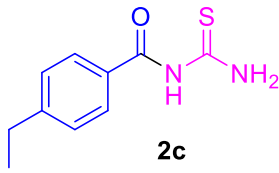
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



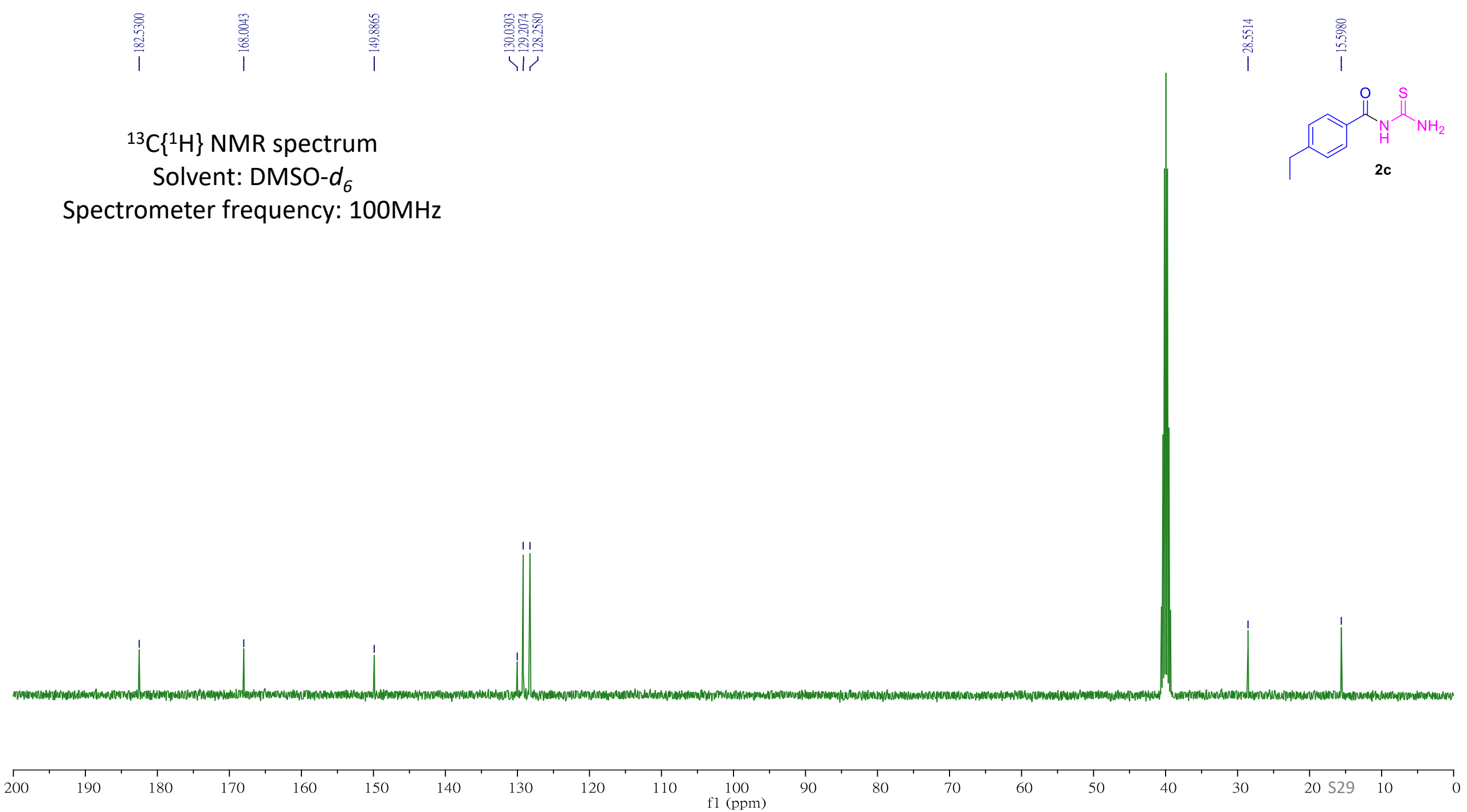
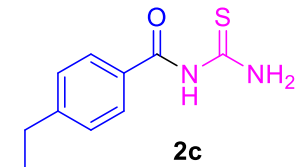
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz

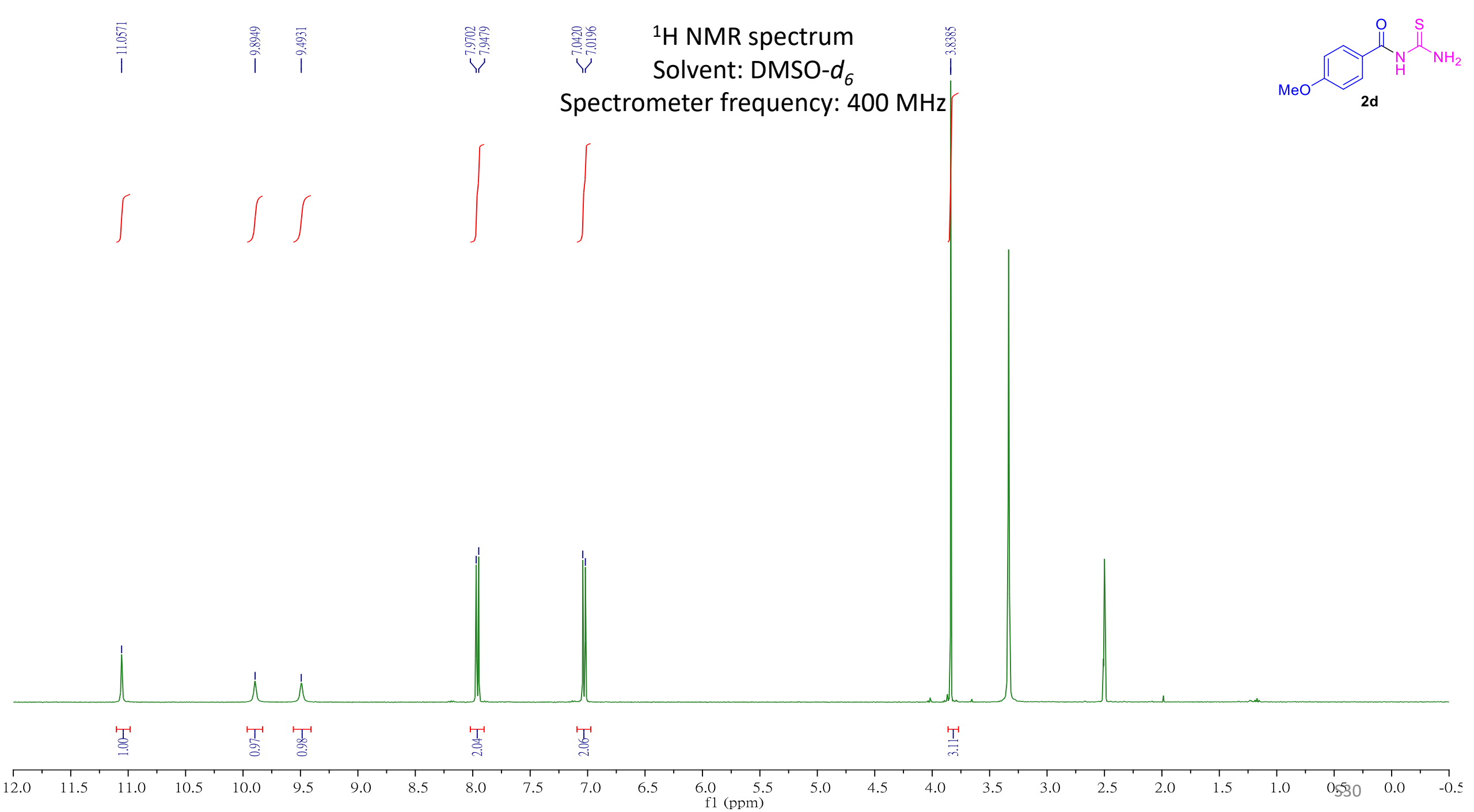


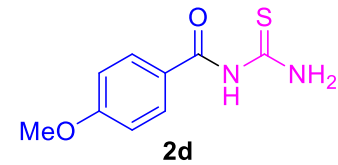
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



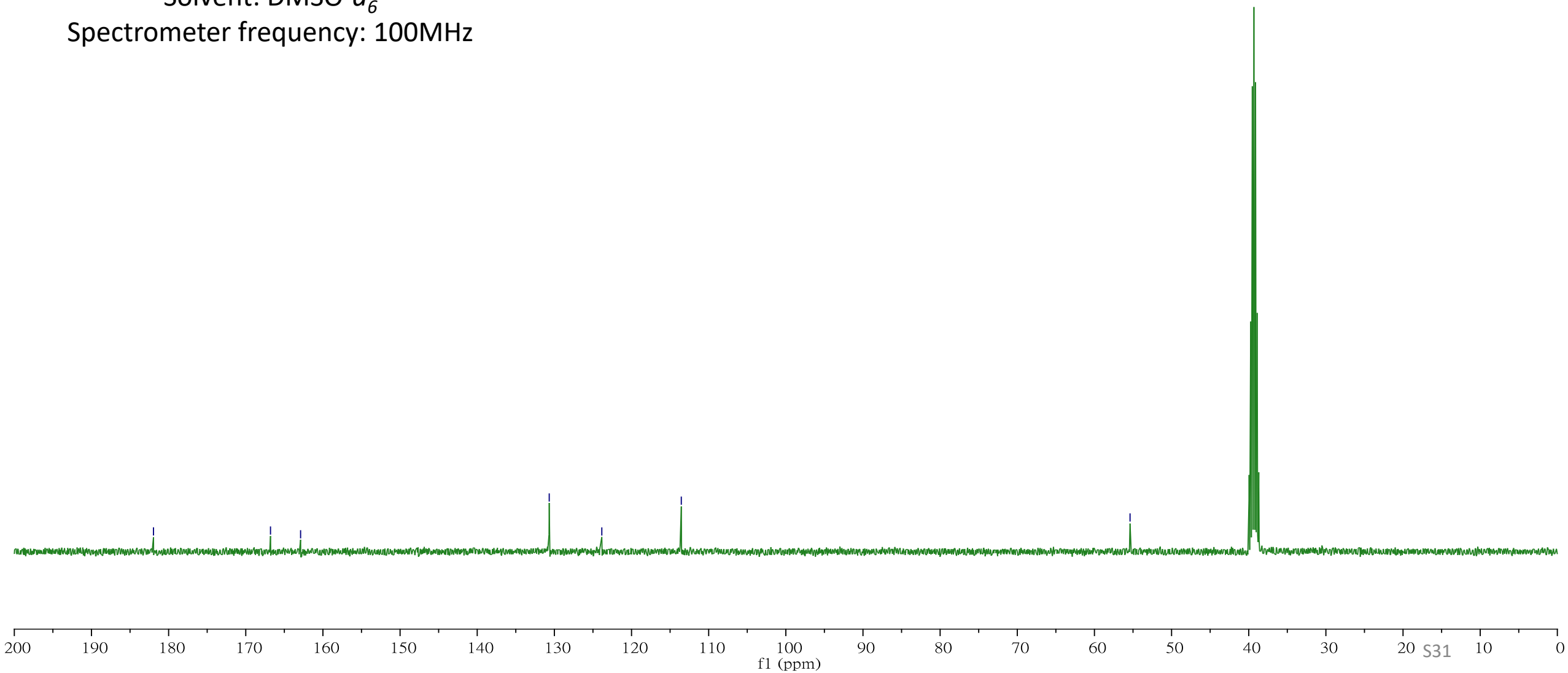
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



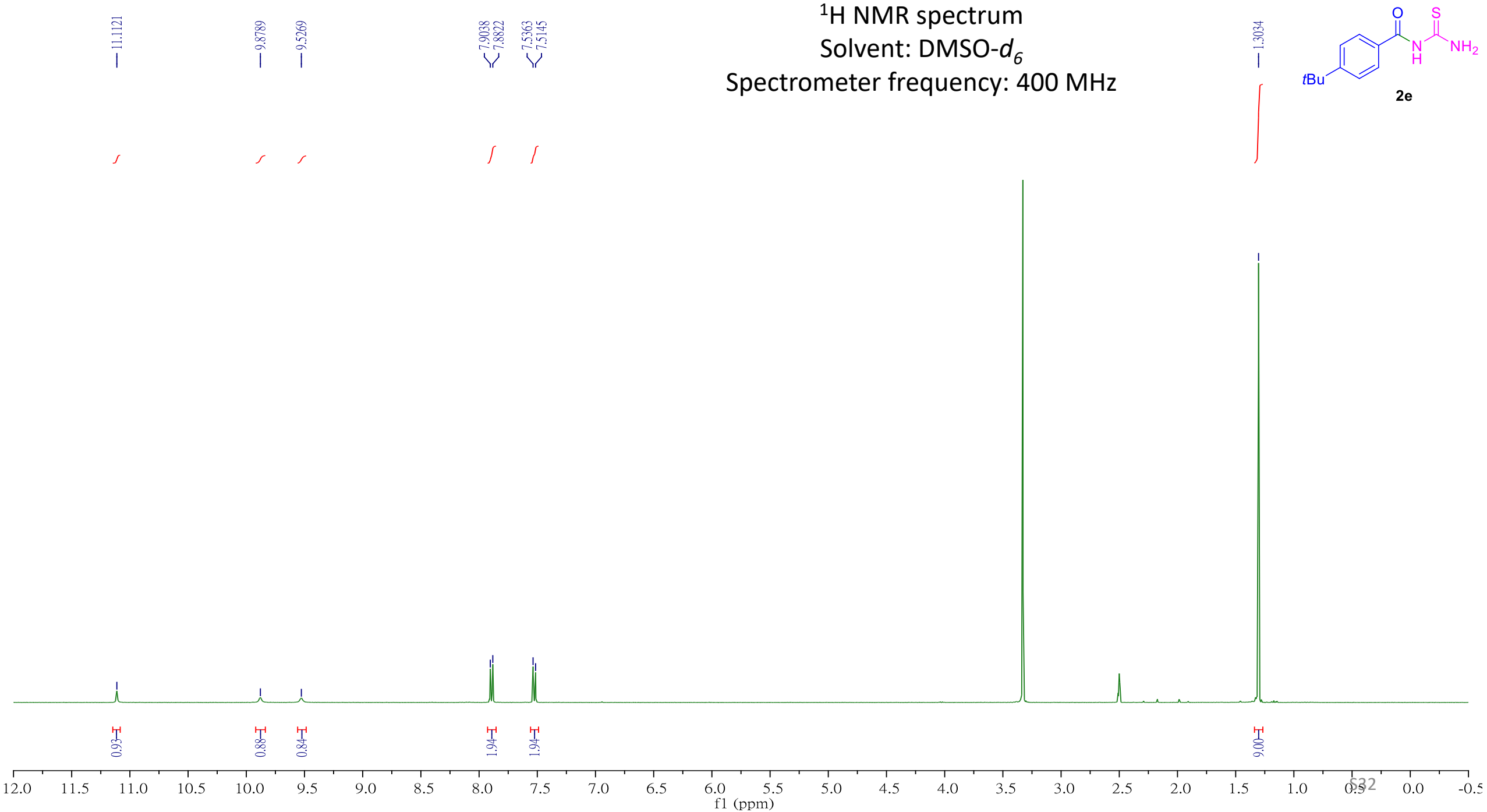
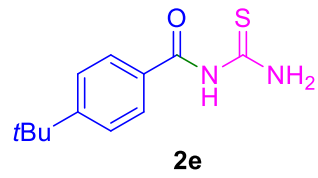


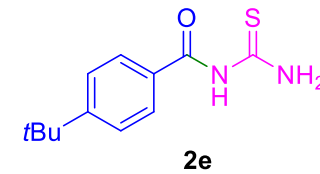


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz

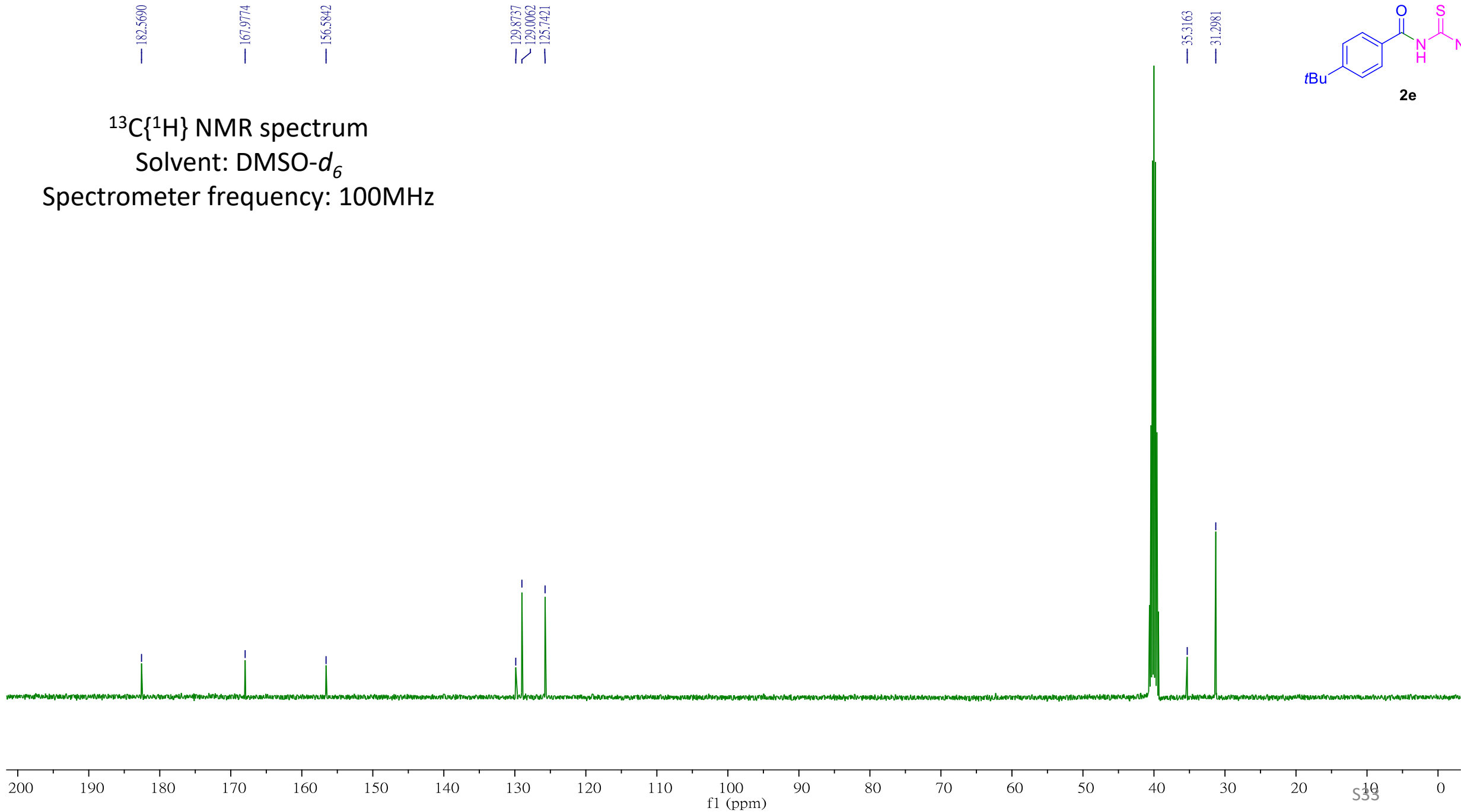


¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz

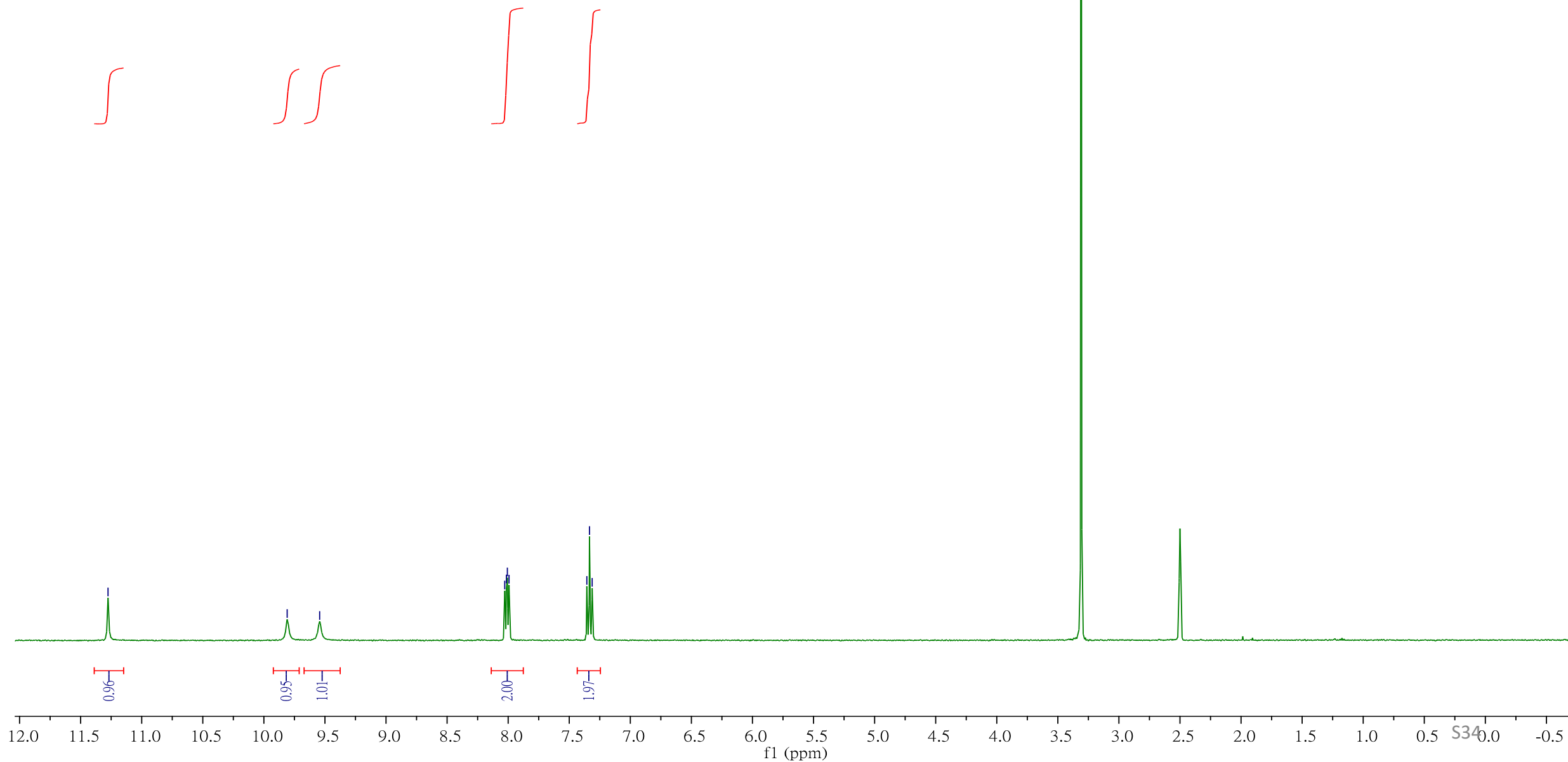
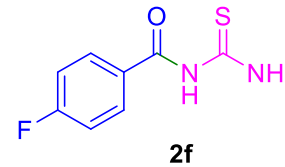


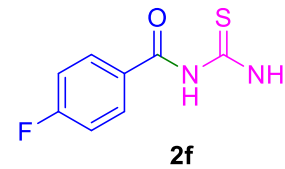


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz

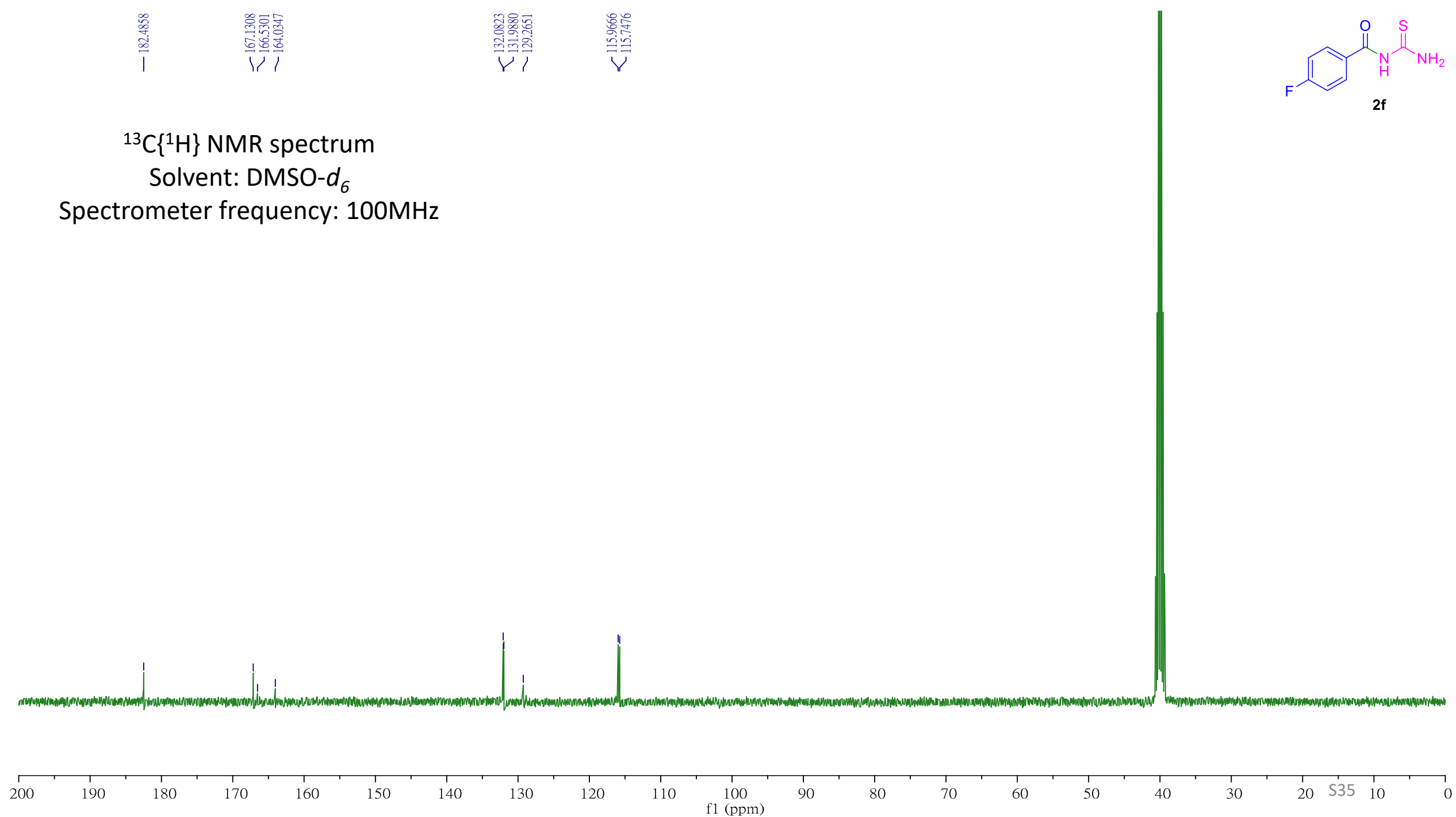


¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz





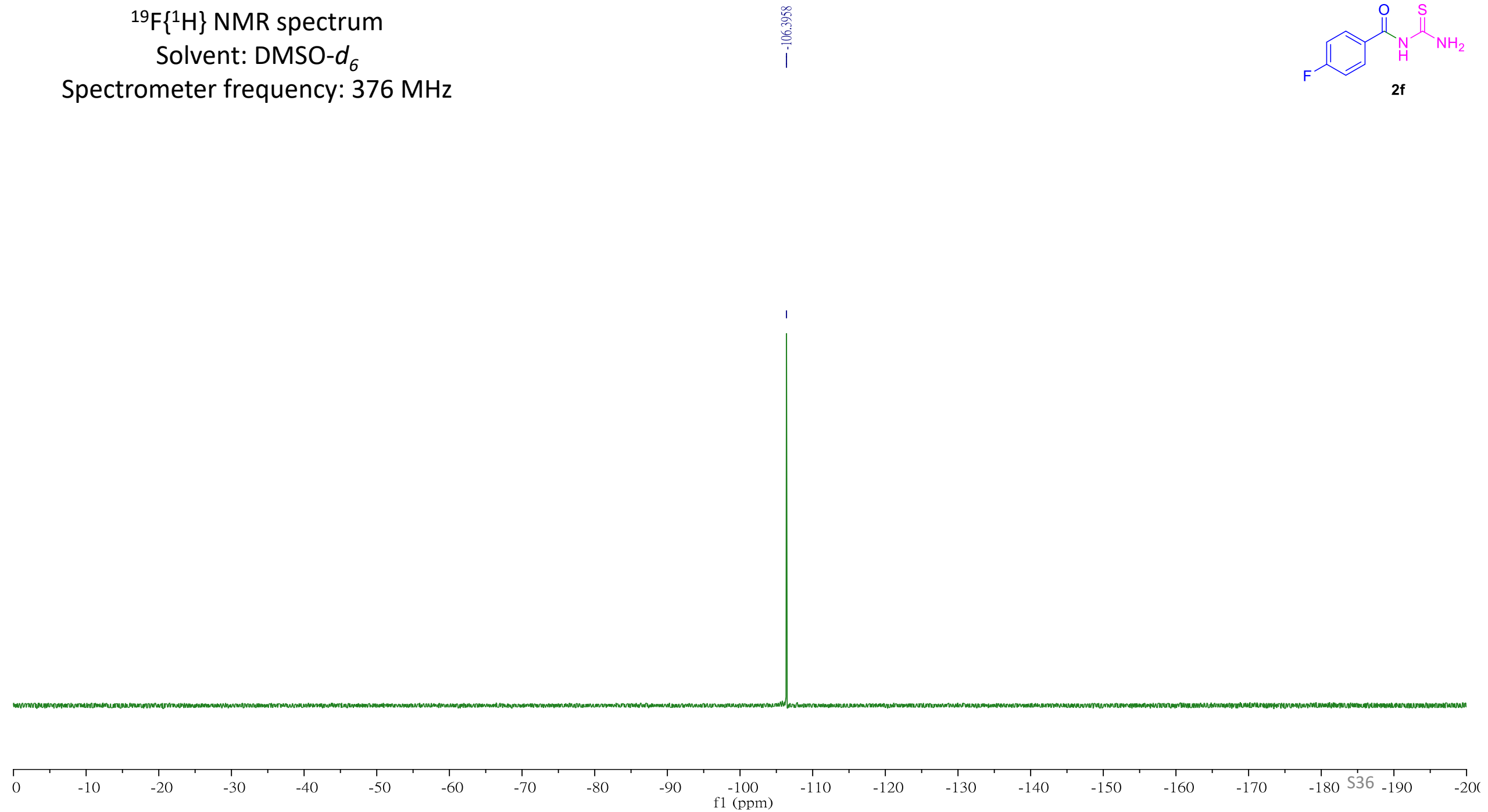
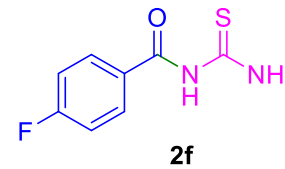
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



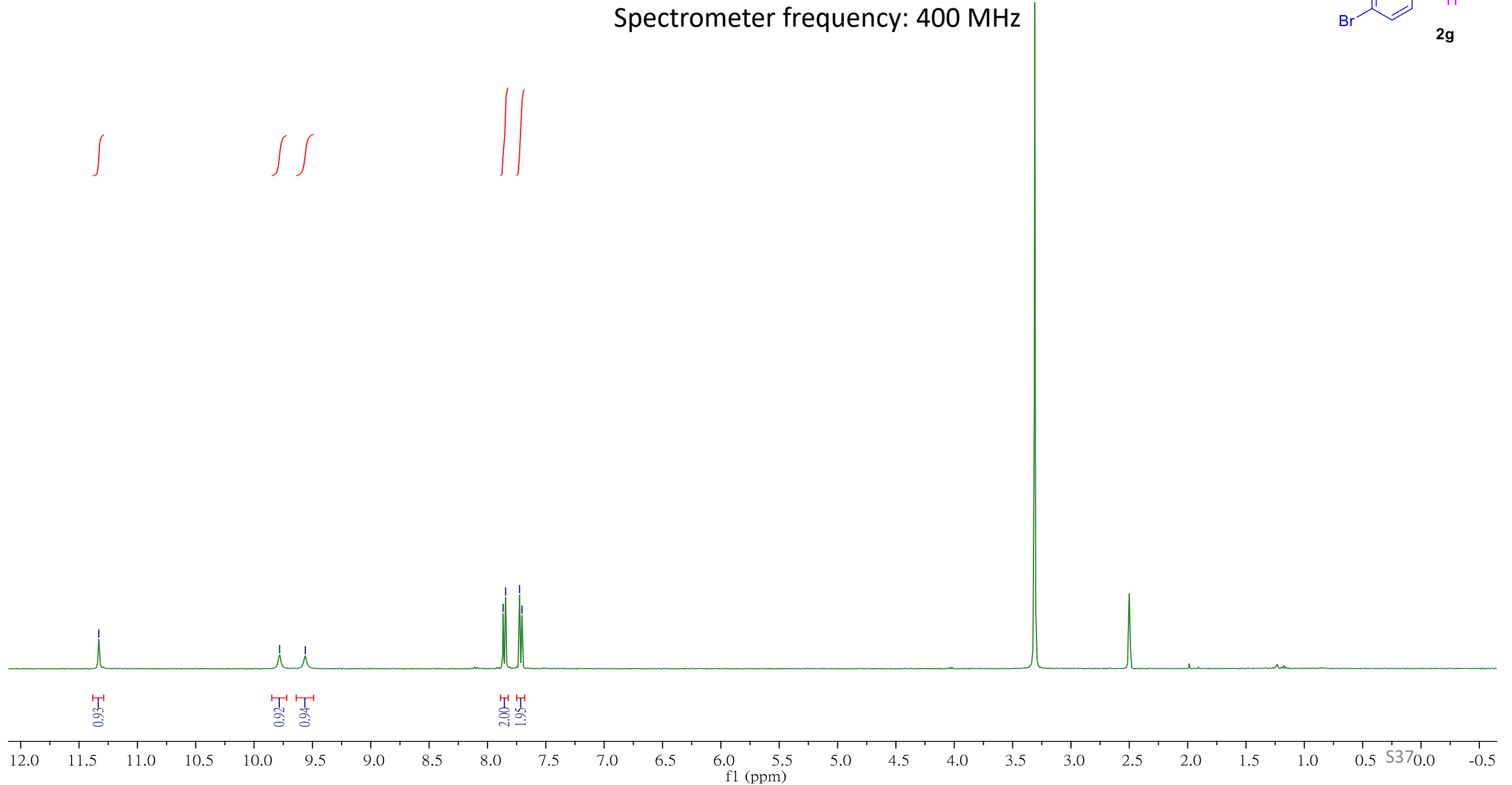
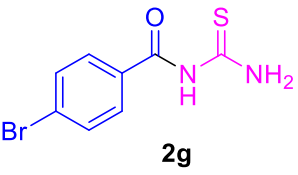
$^{19}\text{F}\{^1\text{H}\}$ NMR spectrum

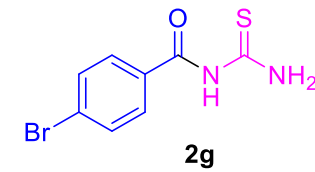
Solvent: $\text{DMSO-}d_6$

Spectrometer frequency: 376 MHz

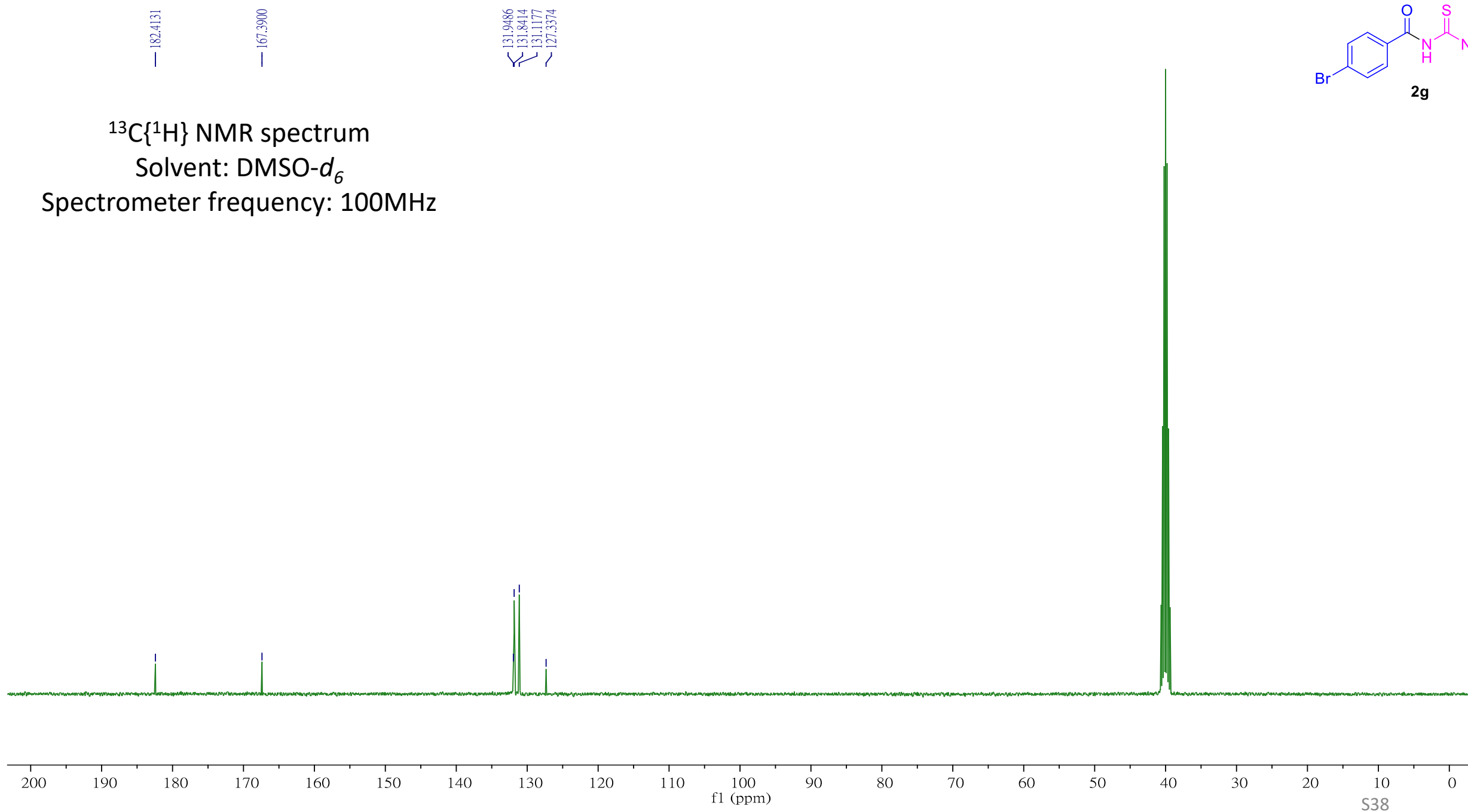


¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz

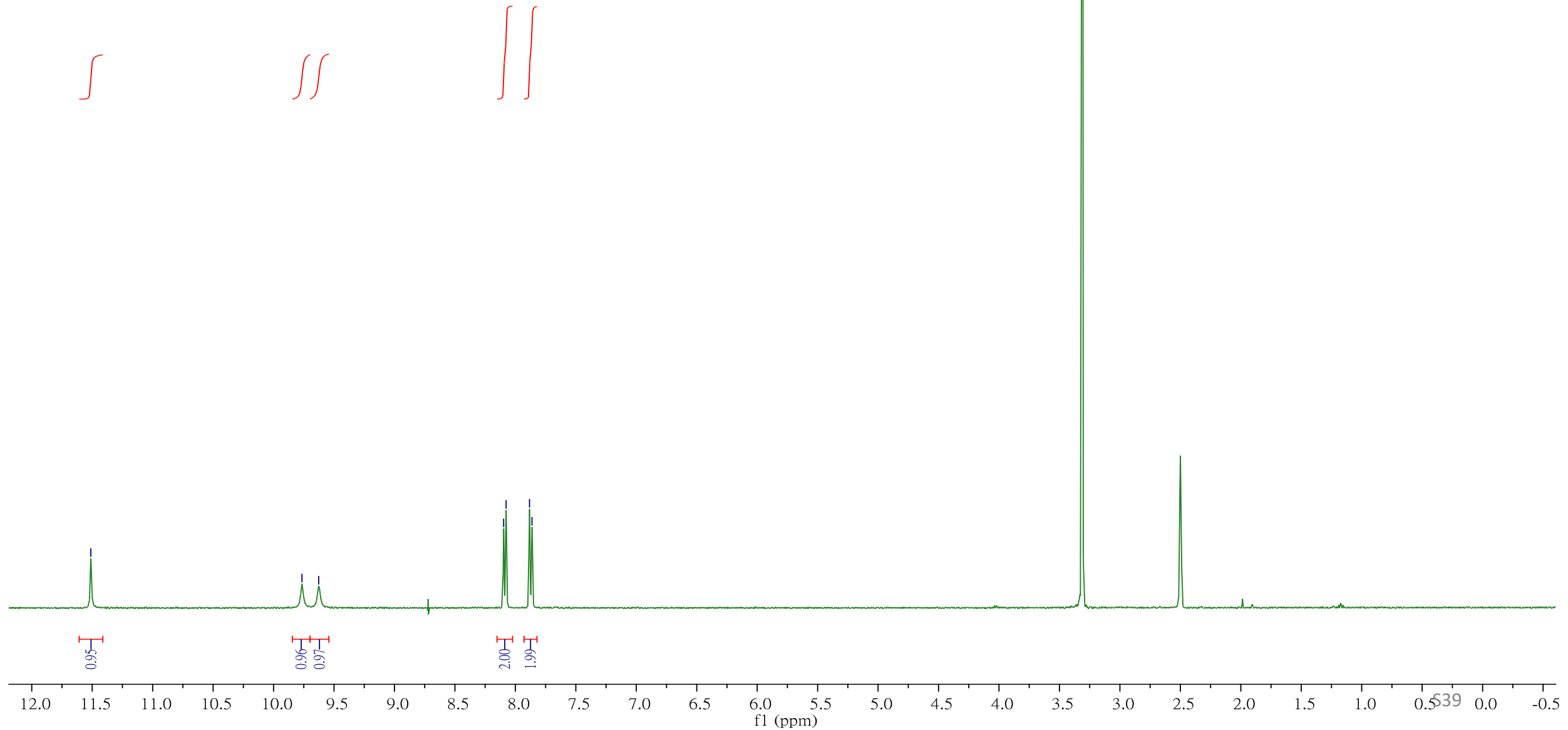
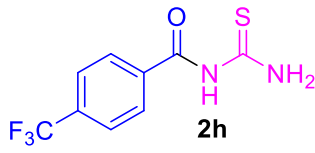


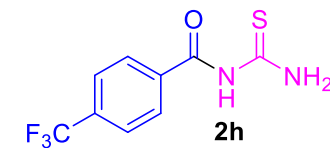


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz

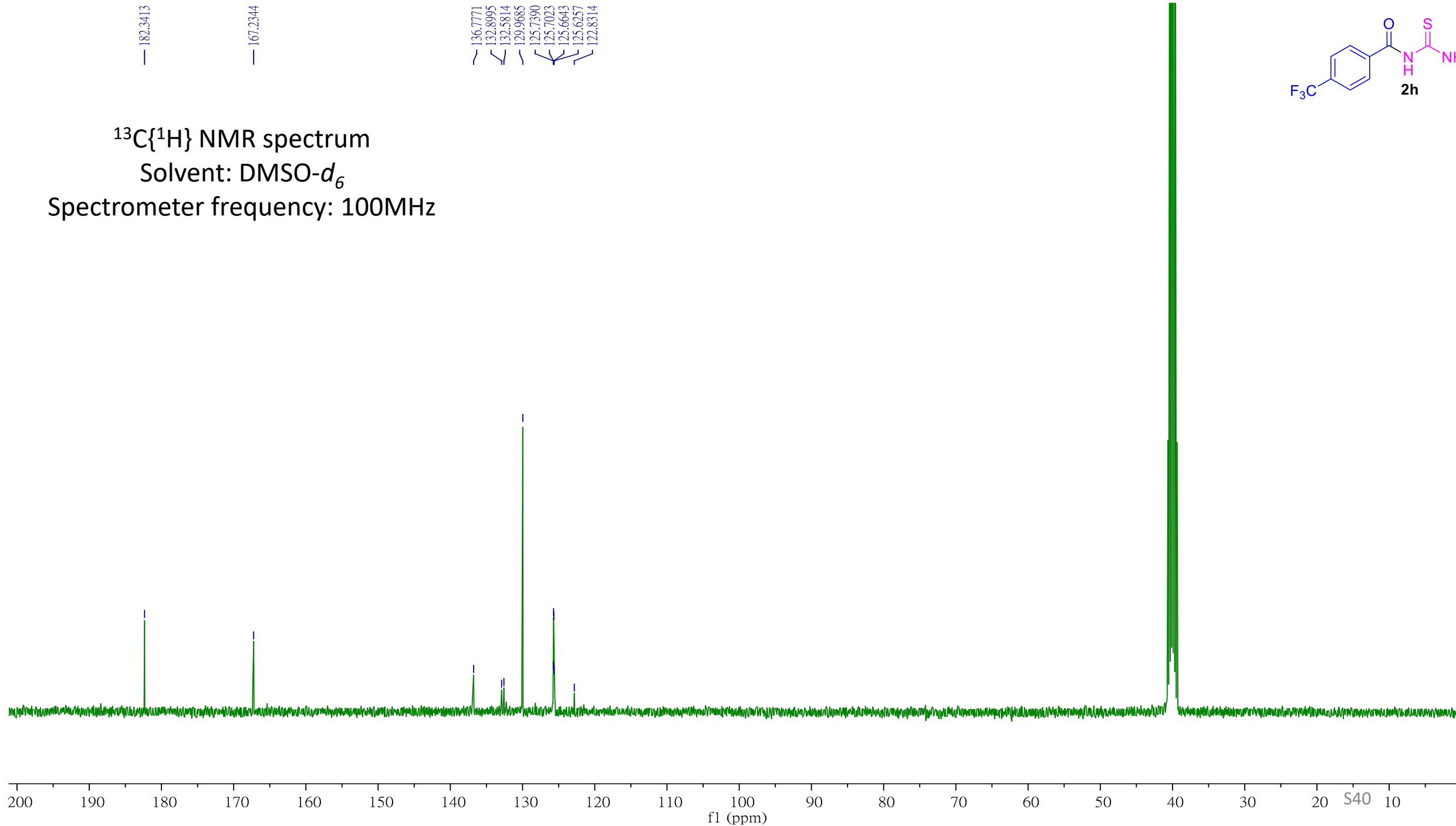


¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz





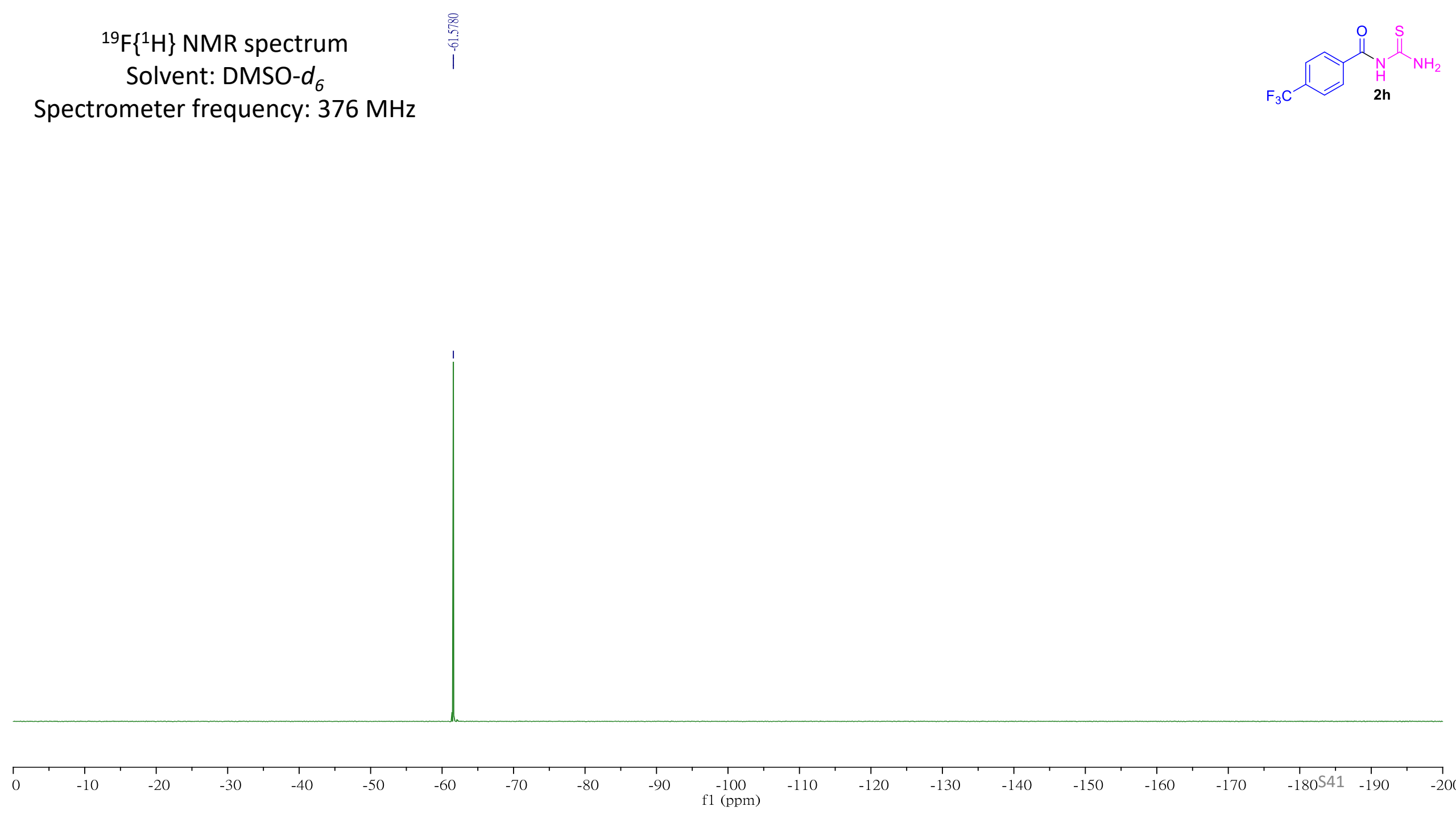
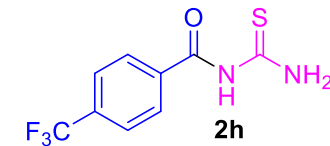
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
 Solvent: DMSO- d_6
 Spectrometer frequency: 100MHz



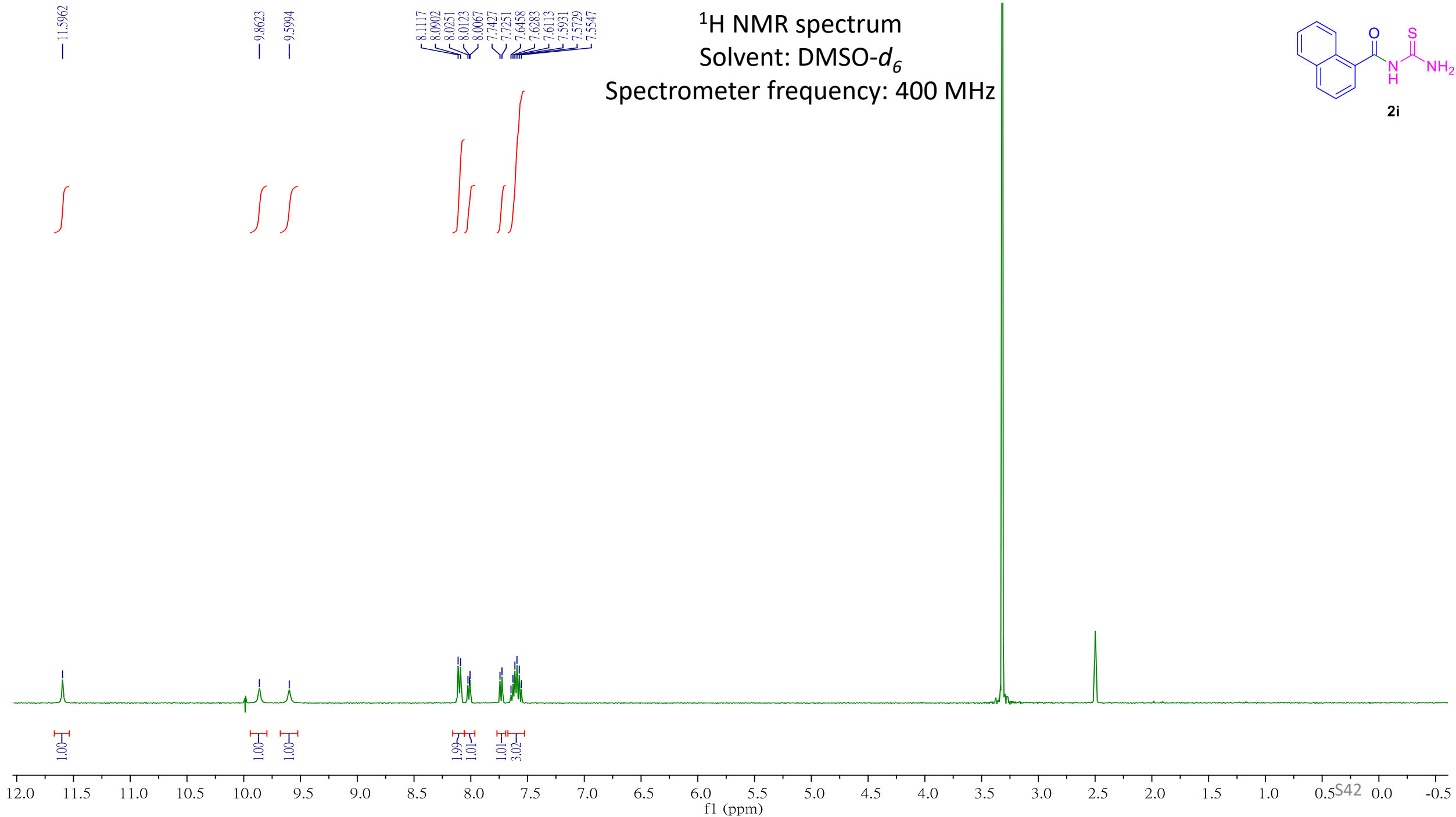
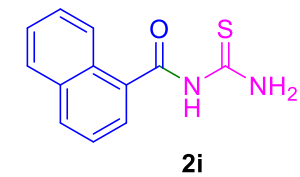
$^{19}\text{F}\{^1\text{H}\}$ NMR spectrum

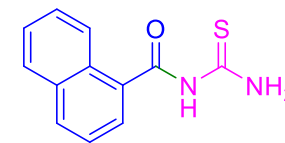
Solvent: $\text{DMSO-}d_6$

Spectrometer frequency: 376 MHz



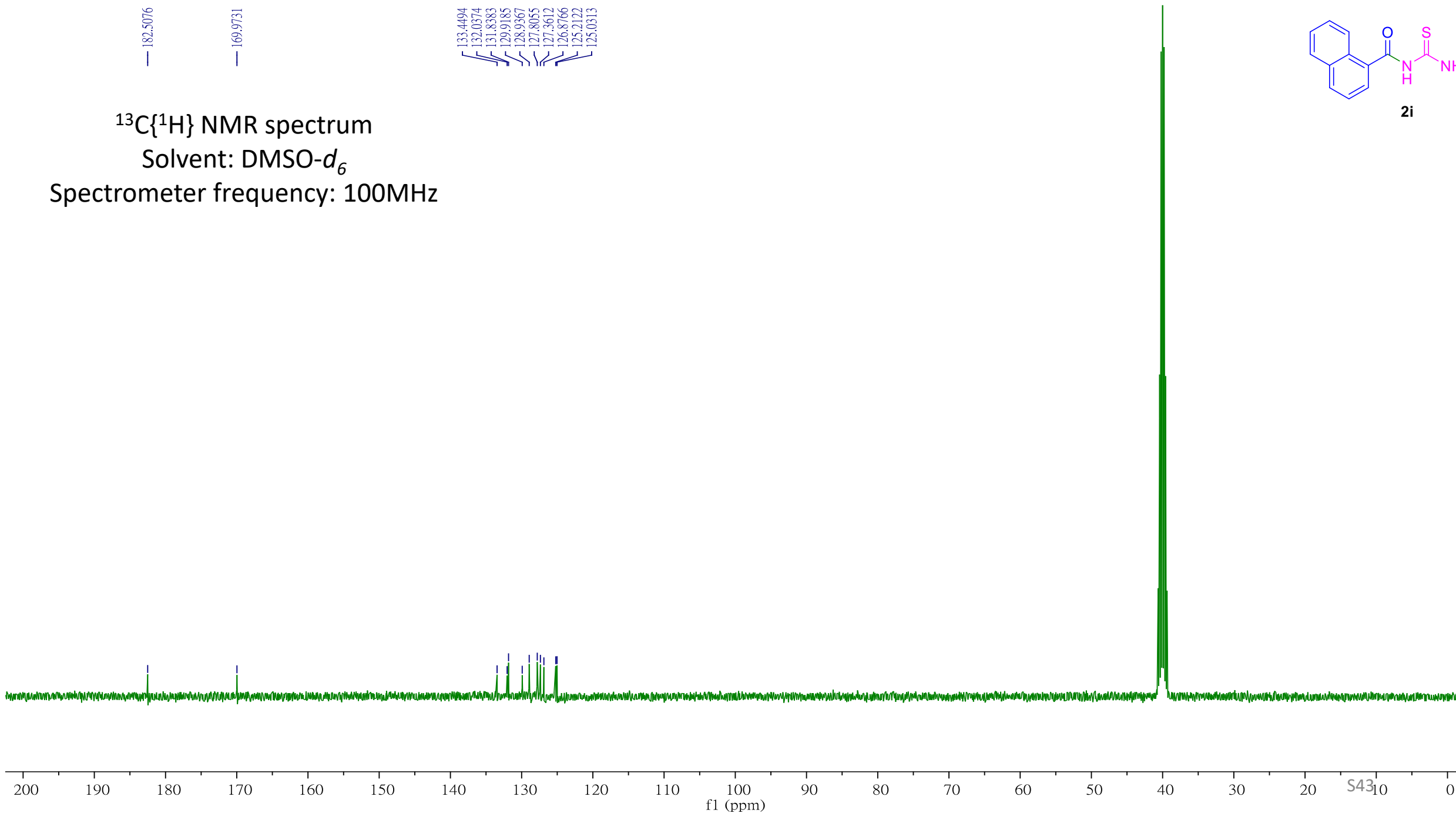
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



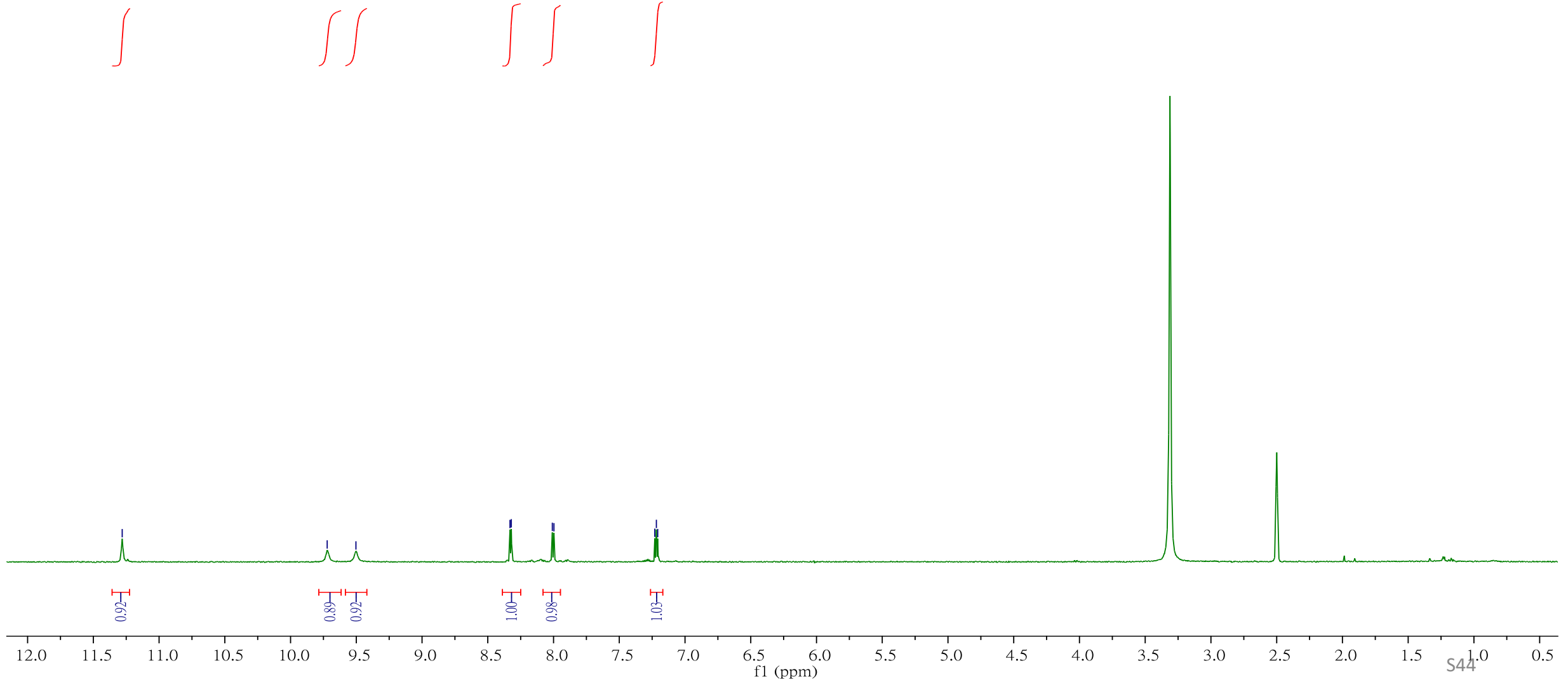
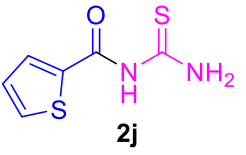


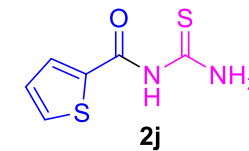
2i

$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz

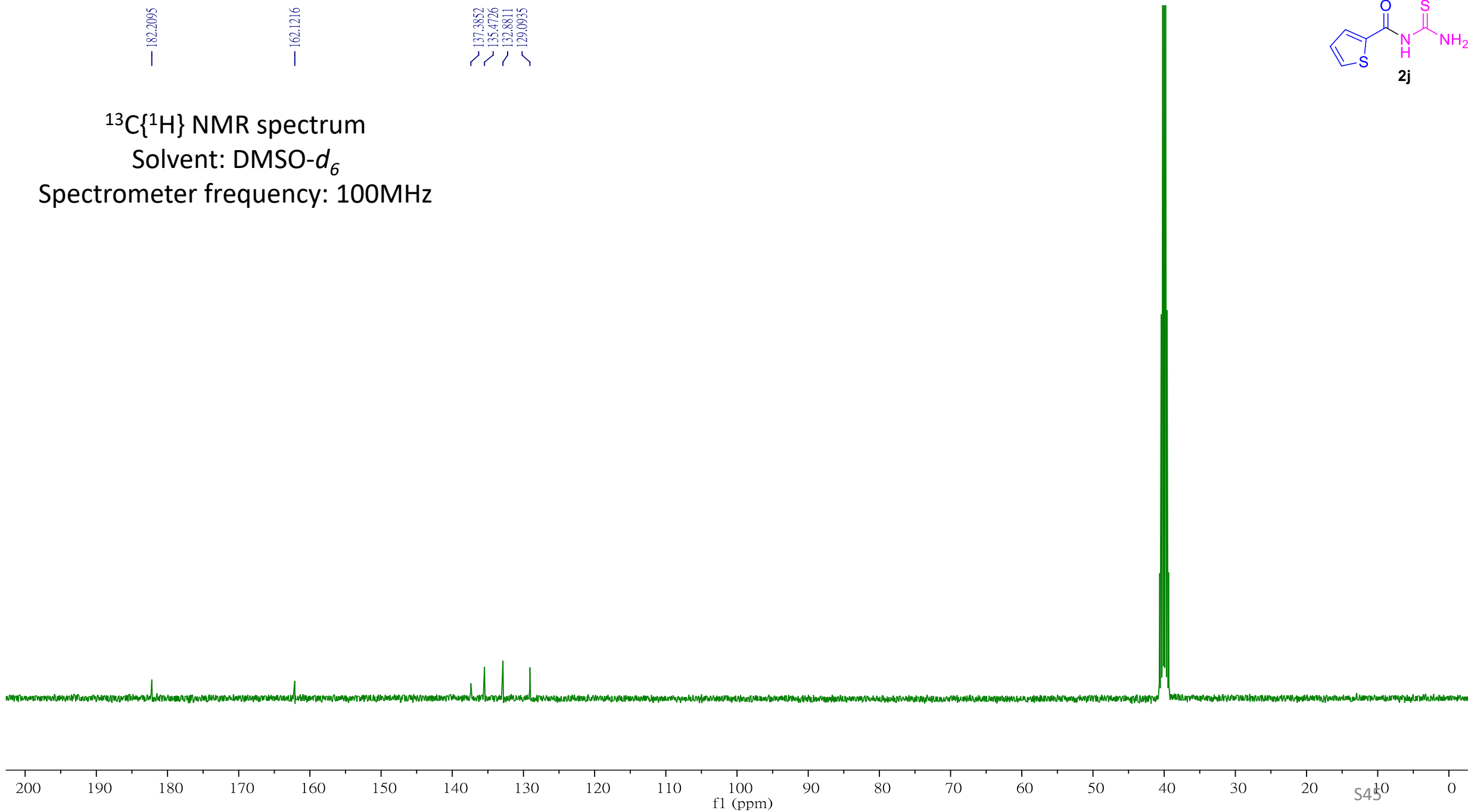


¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz





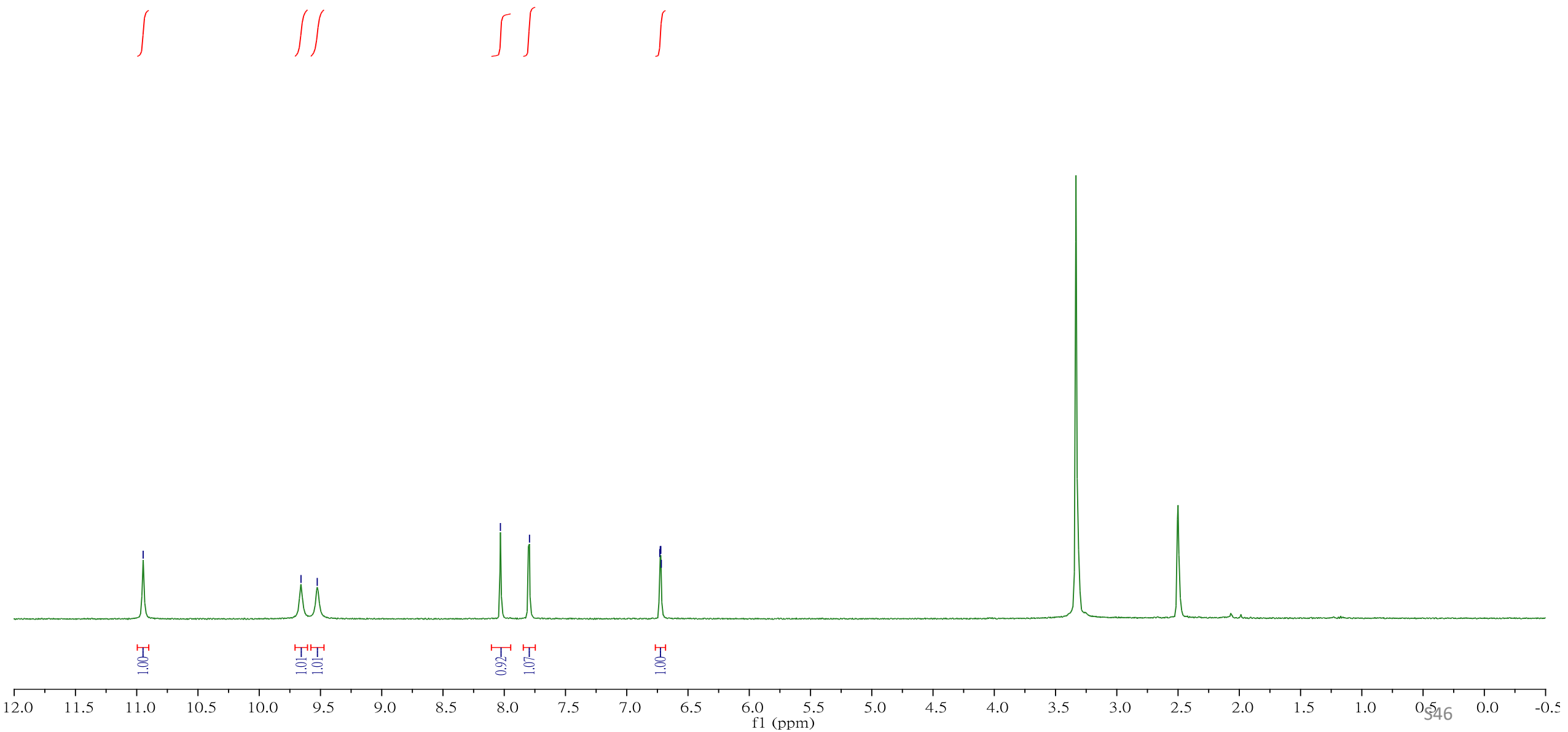
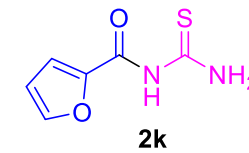
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
 Solvent: $\text{DMSO-}d_6$
 Spectrometer frequency: 100MHz

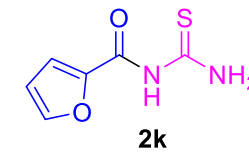


¹H NMR spectrum

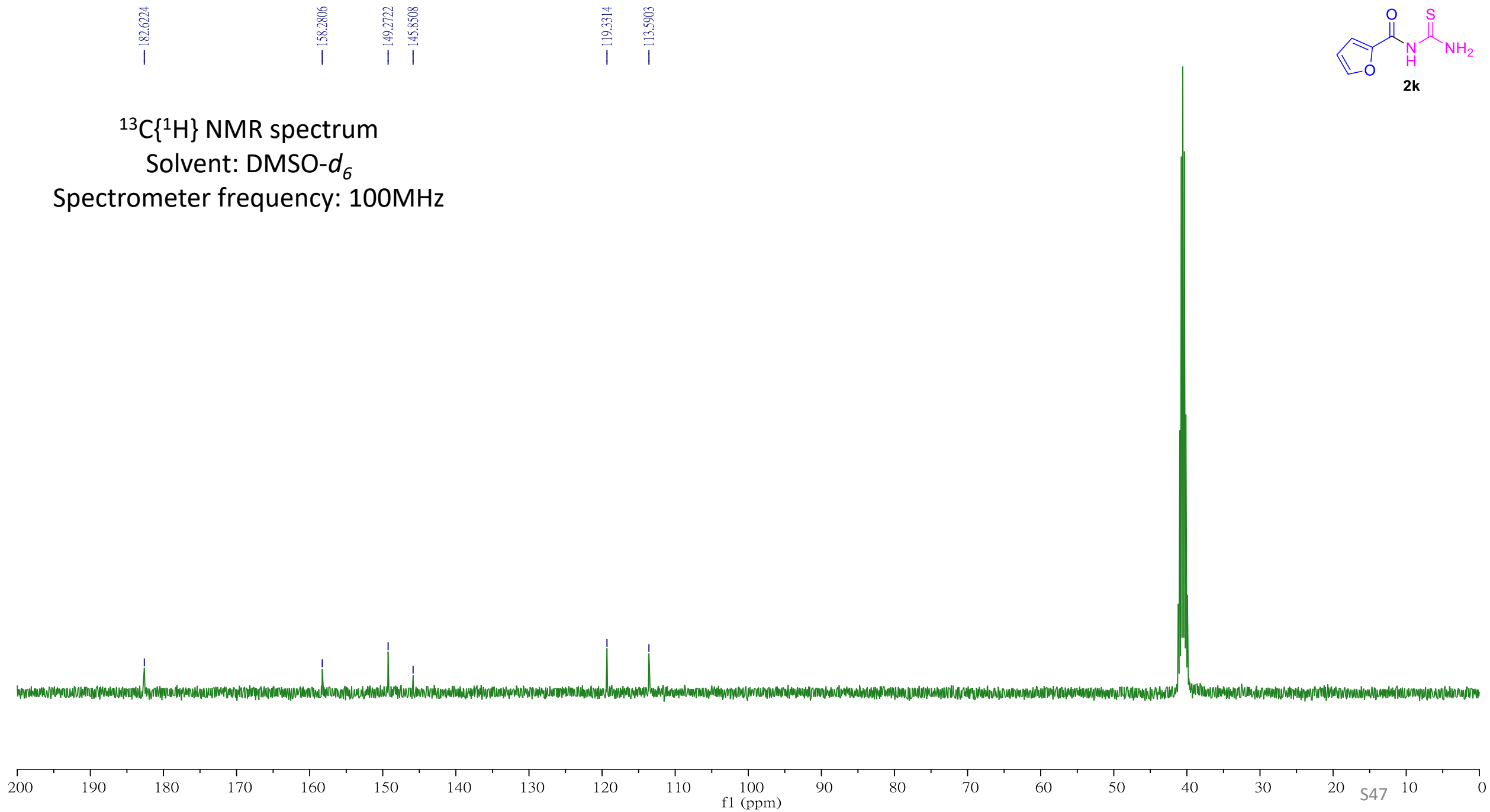
Solvent: DMSO-*d*₆

Spectrometer frequency: 400 MHz

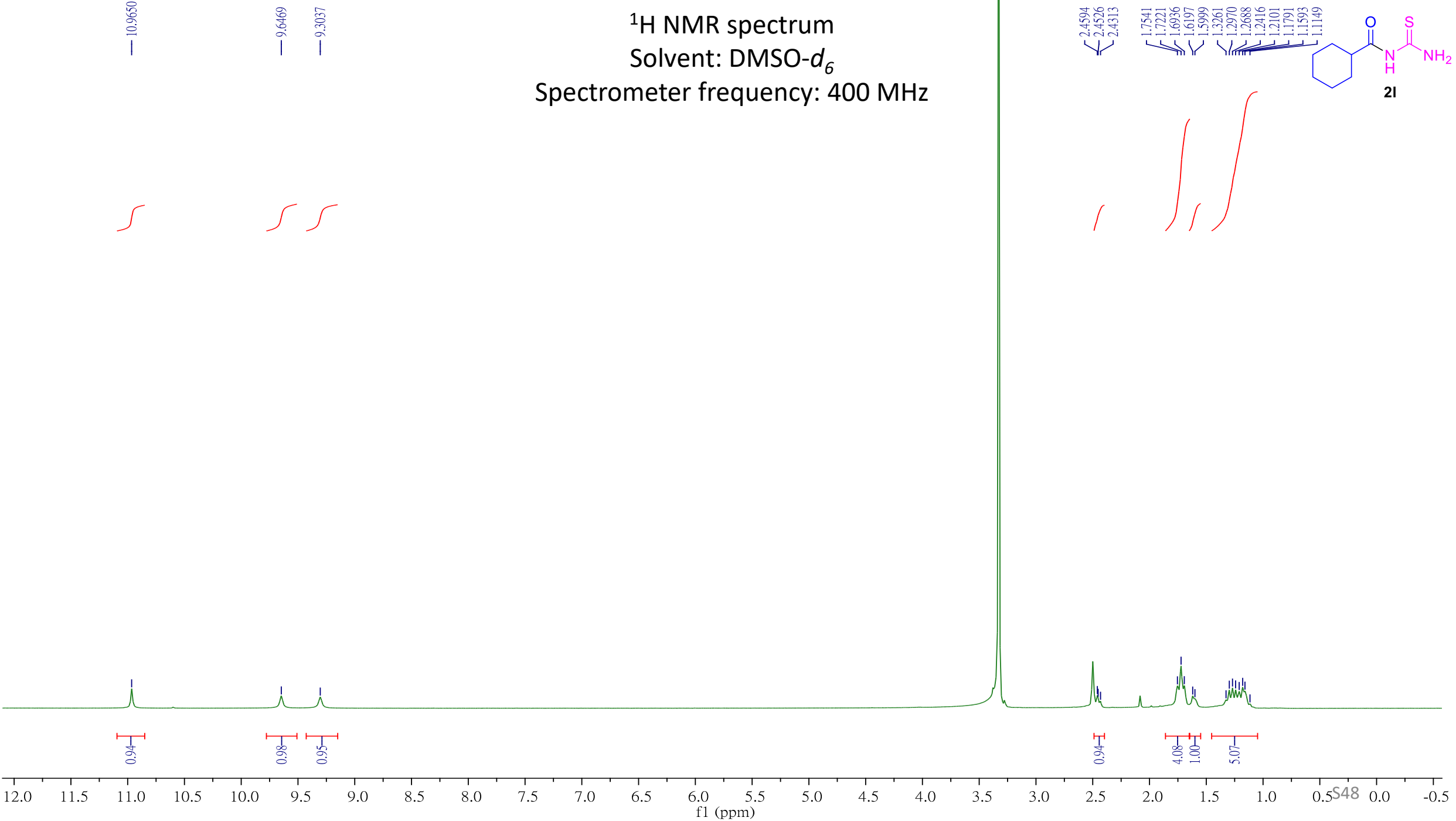
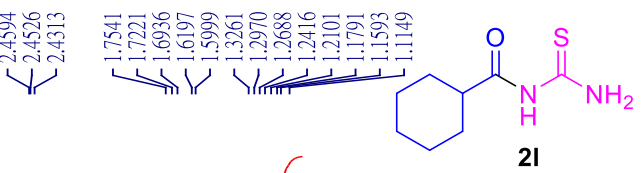


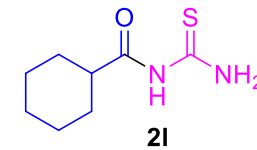


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



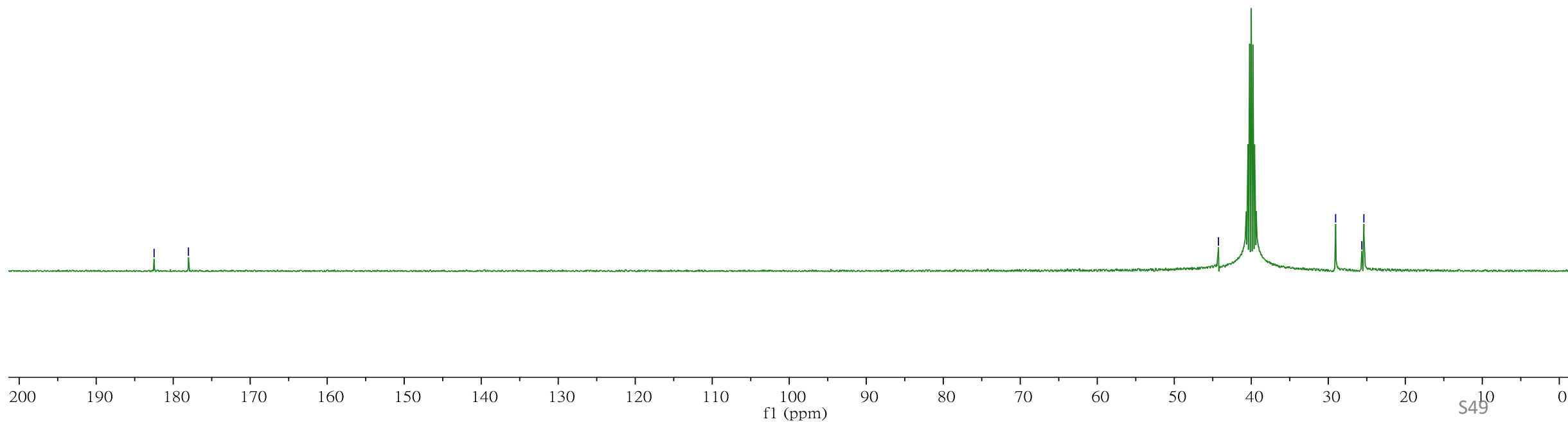


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz

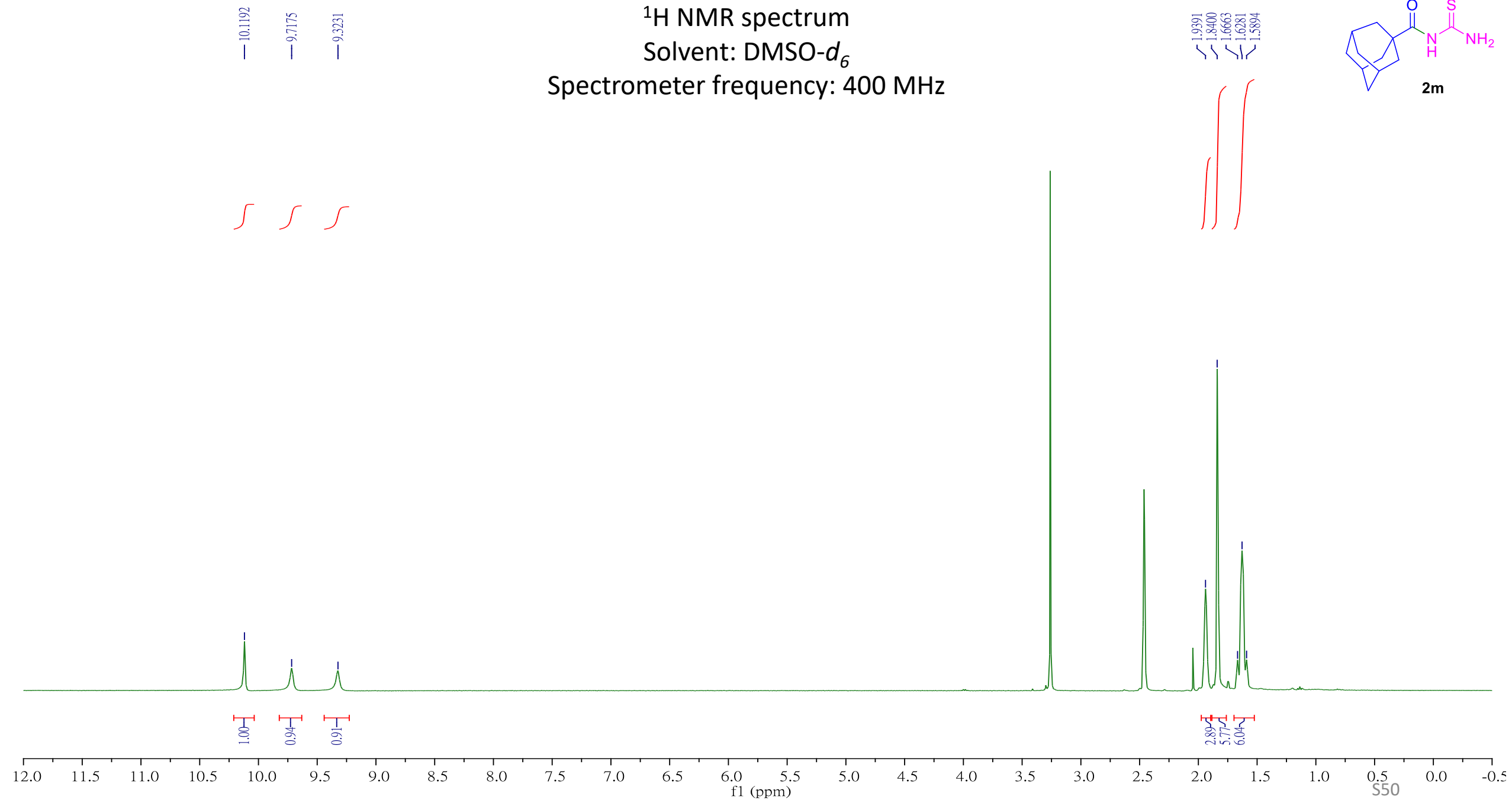
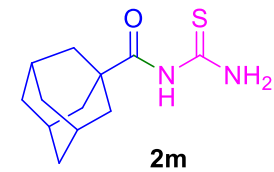
182.4821
178.0187

44.2596

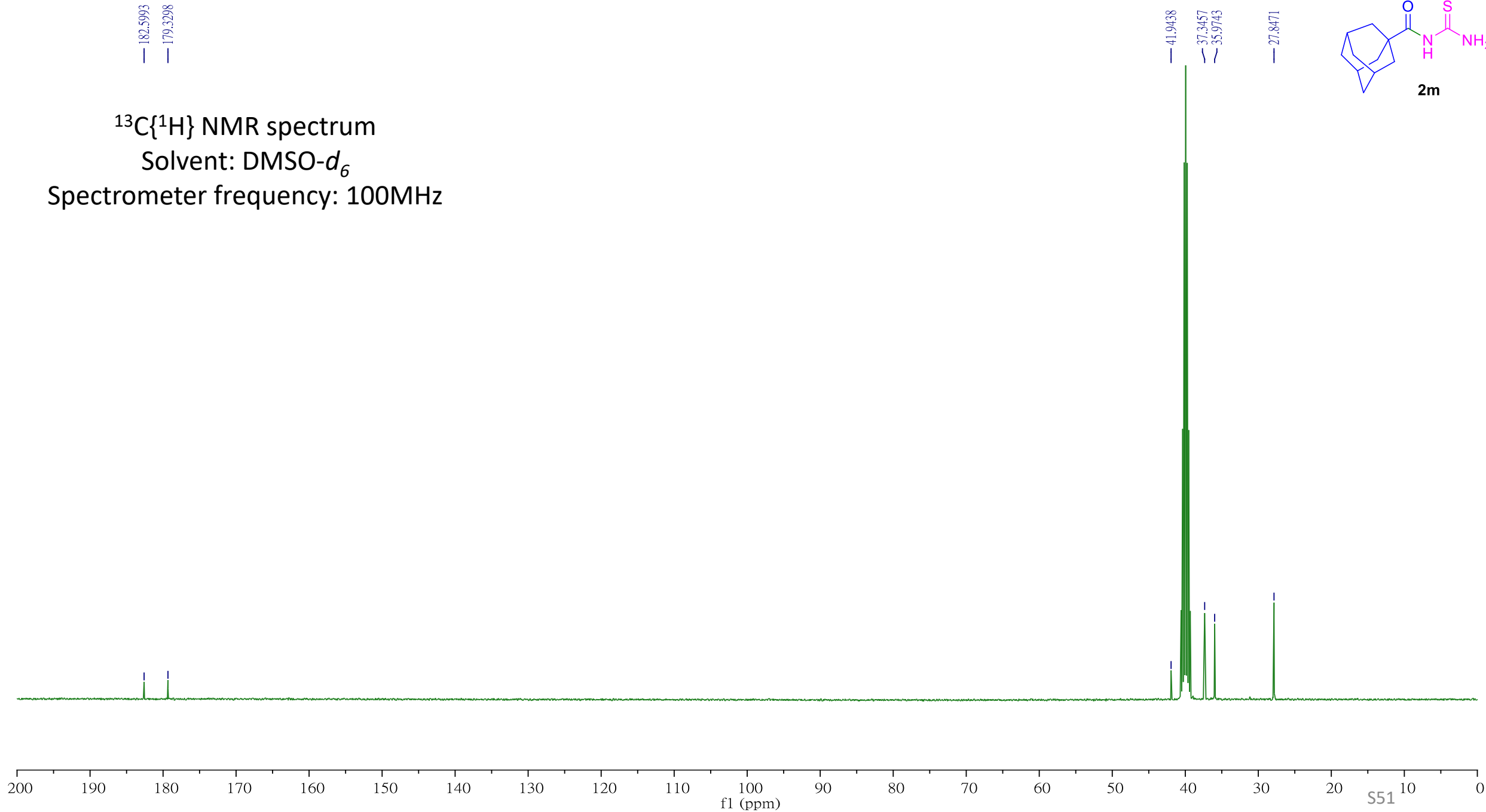
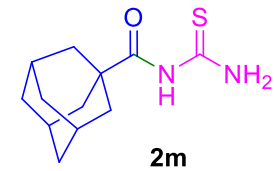
29.0450
25.6413
25.3771



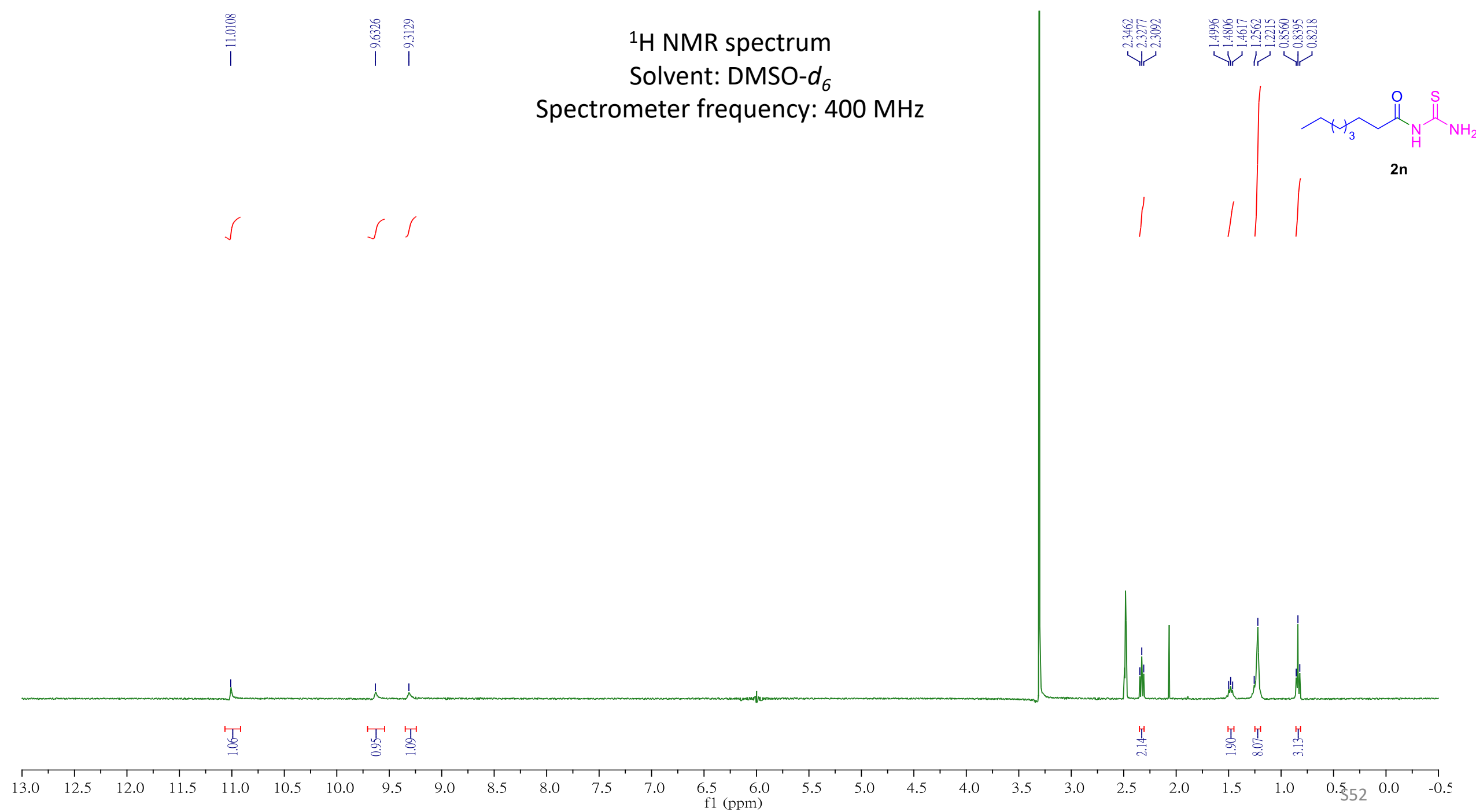
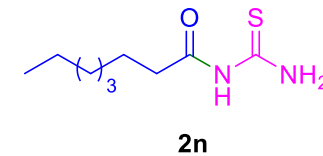
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



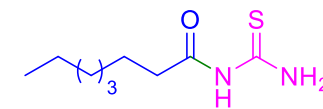
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz



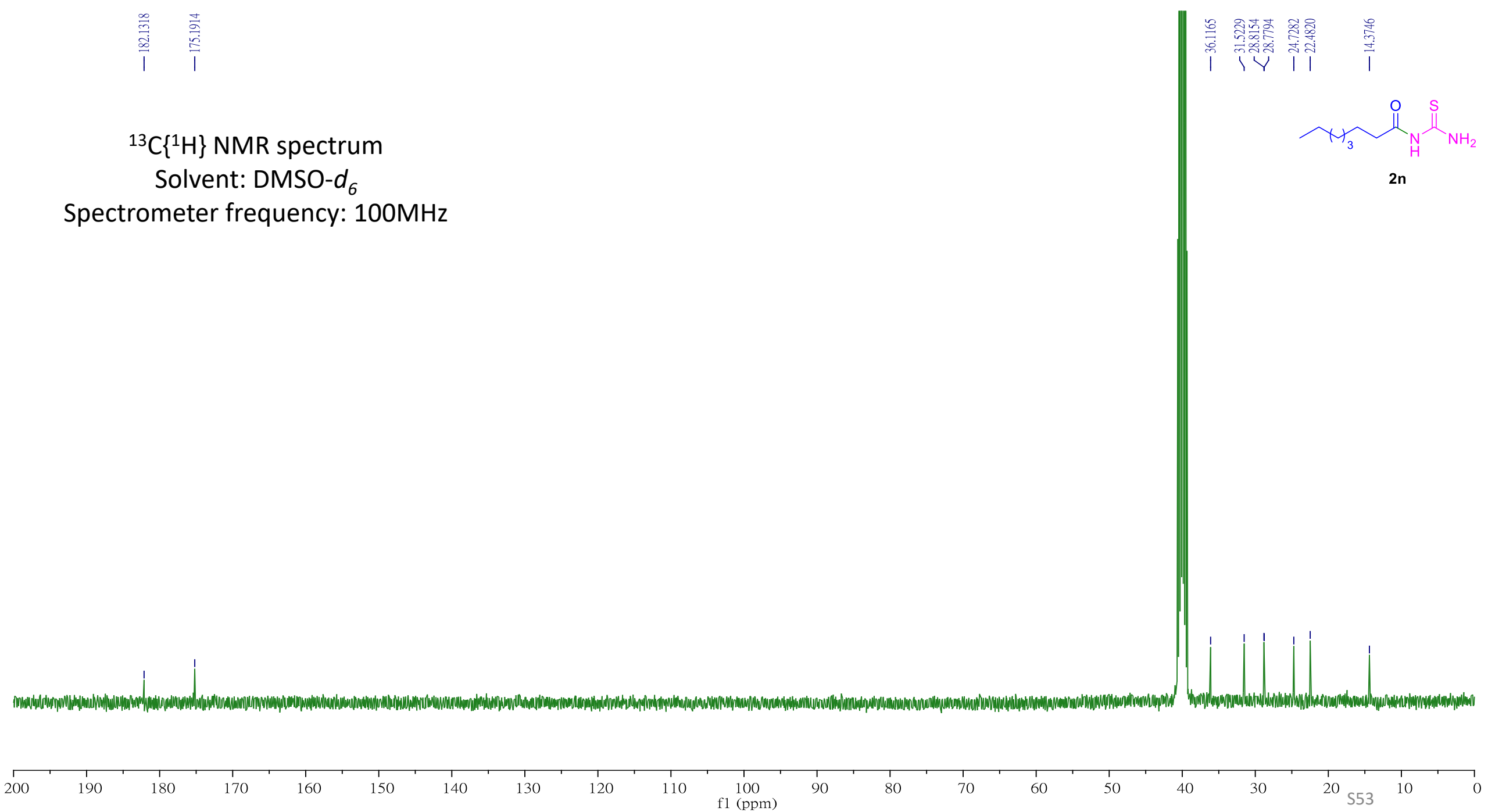
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



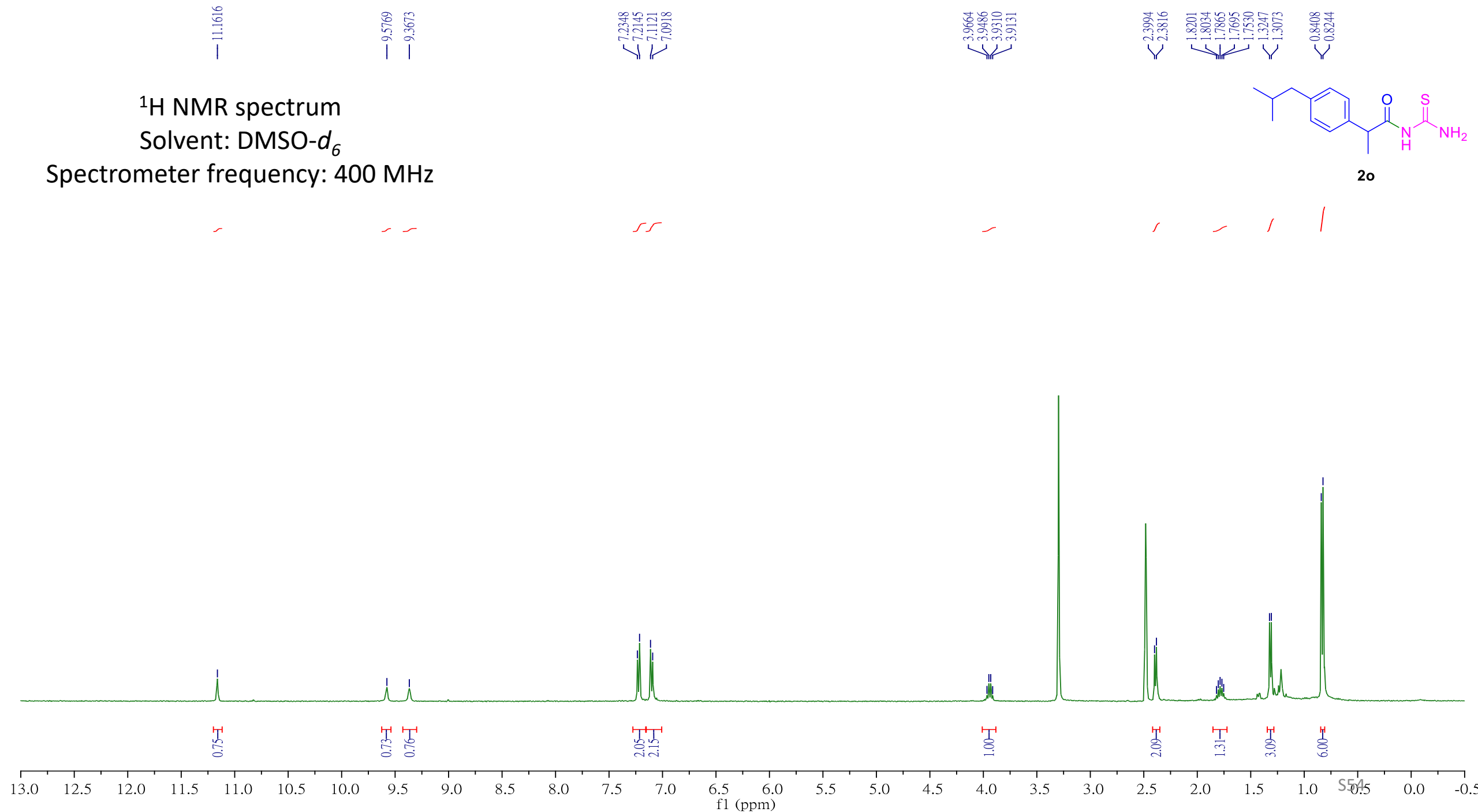
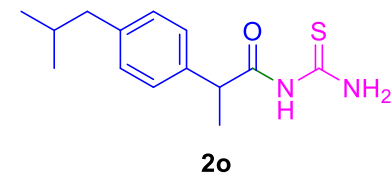
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz



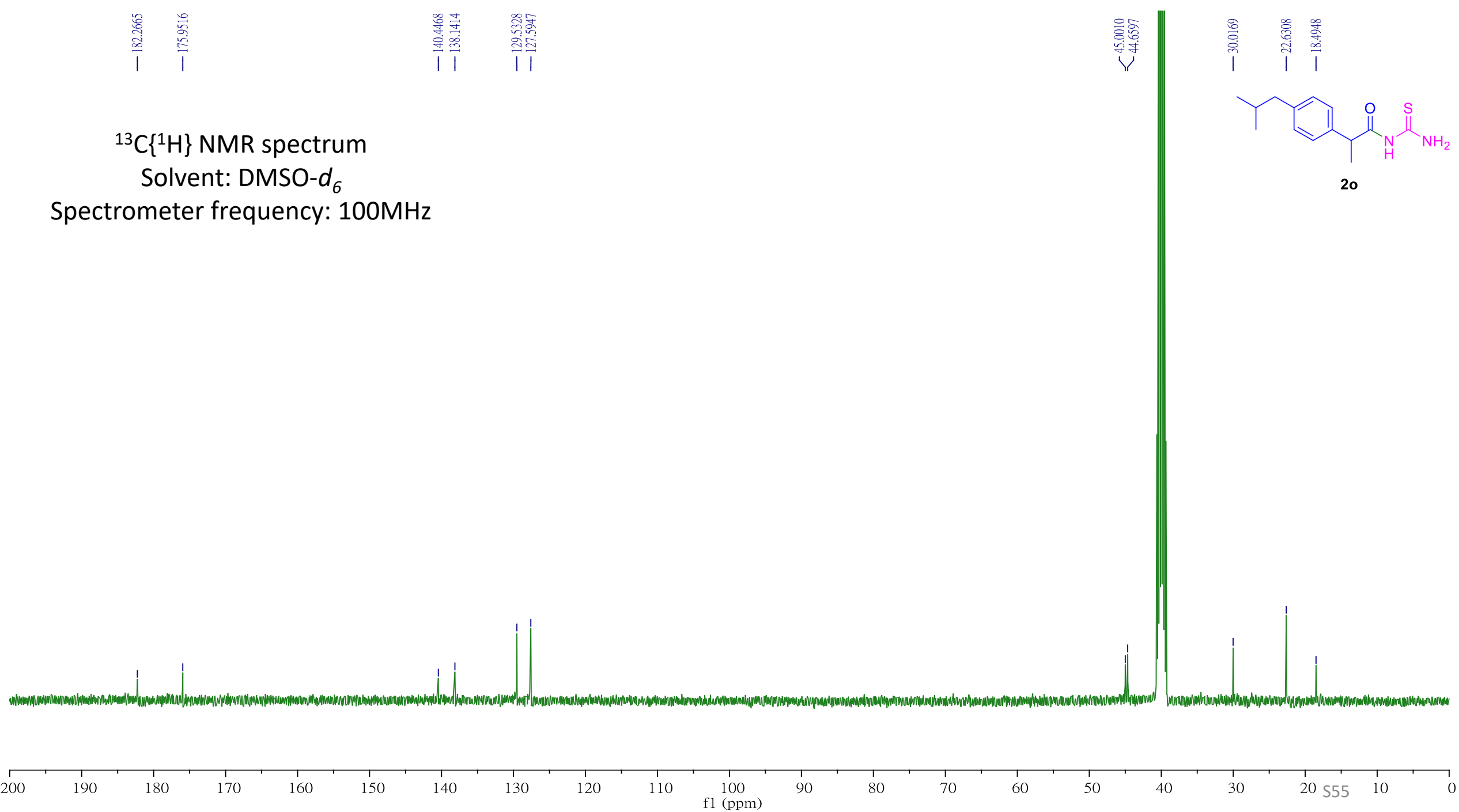
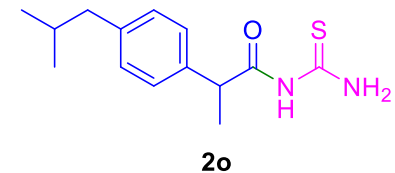
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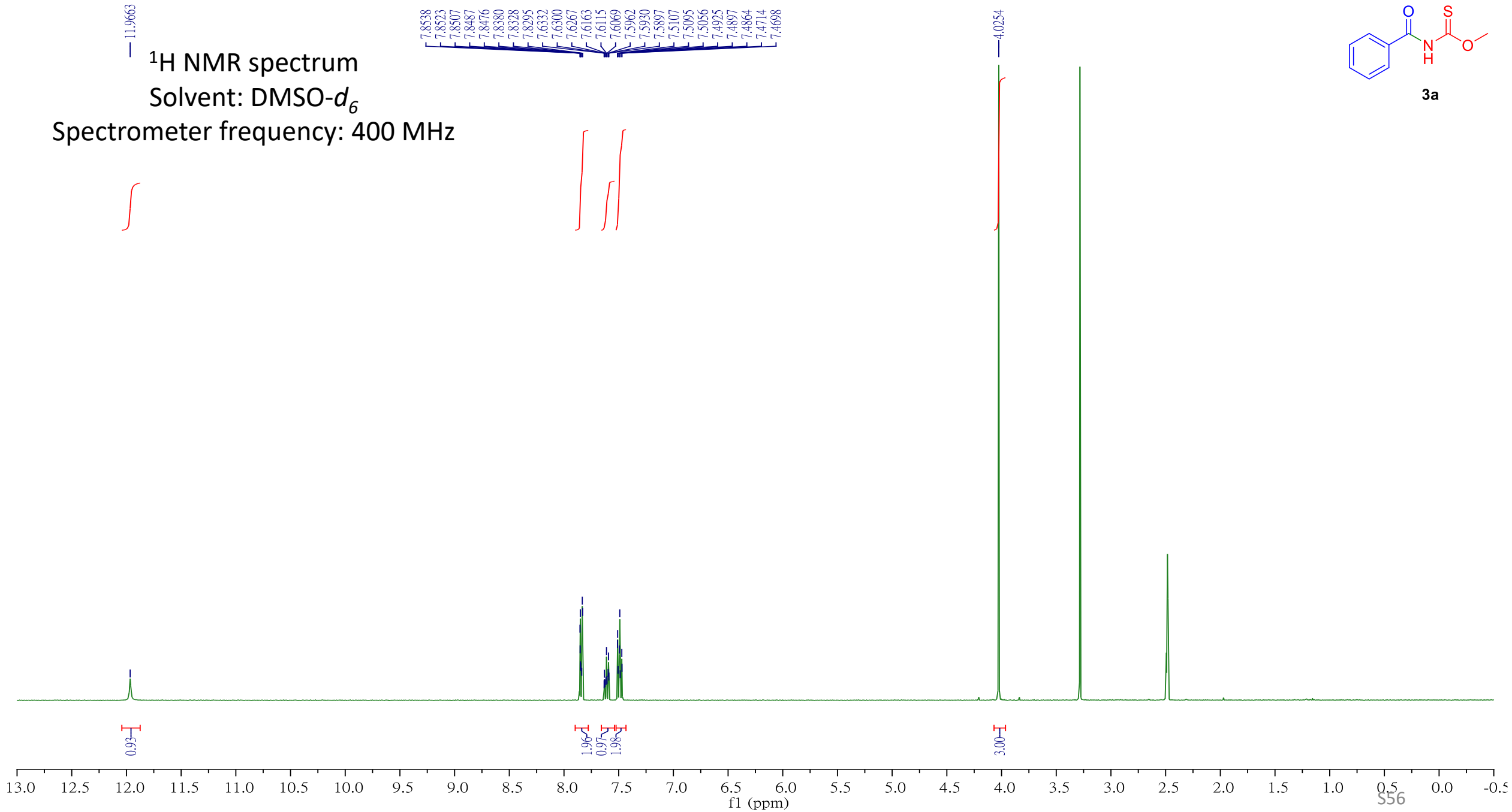
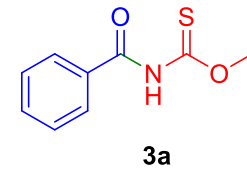
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz

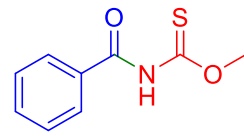


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



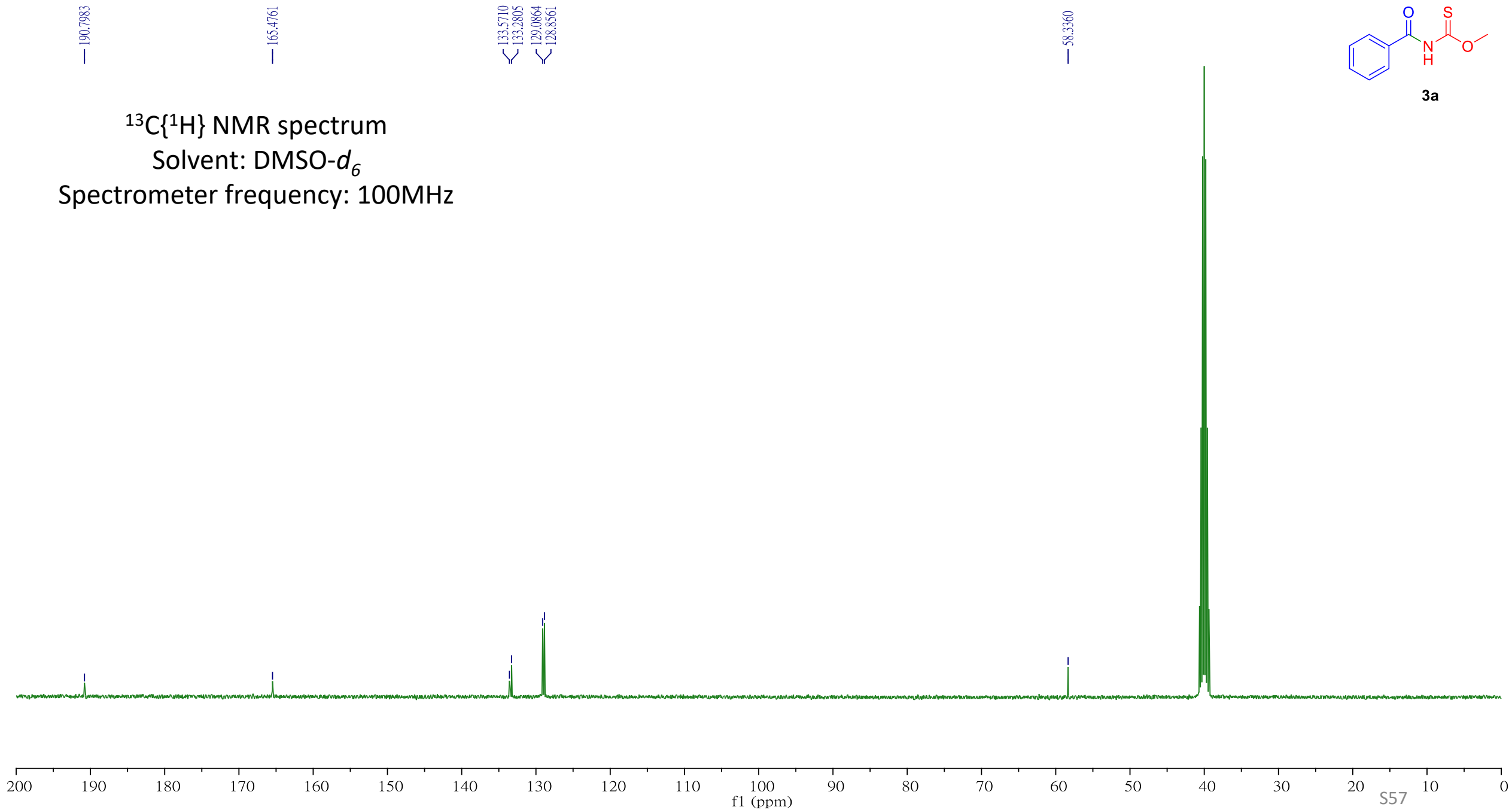
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



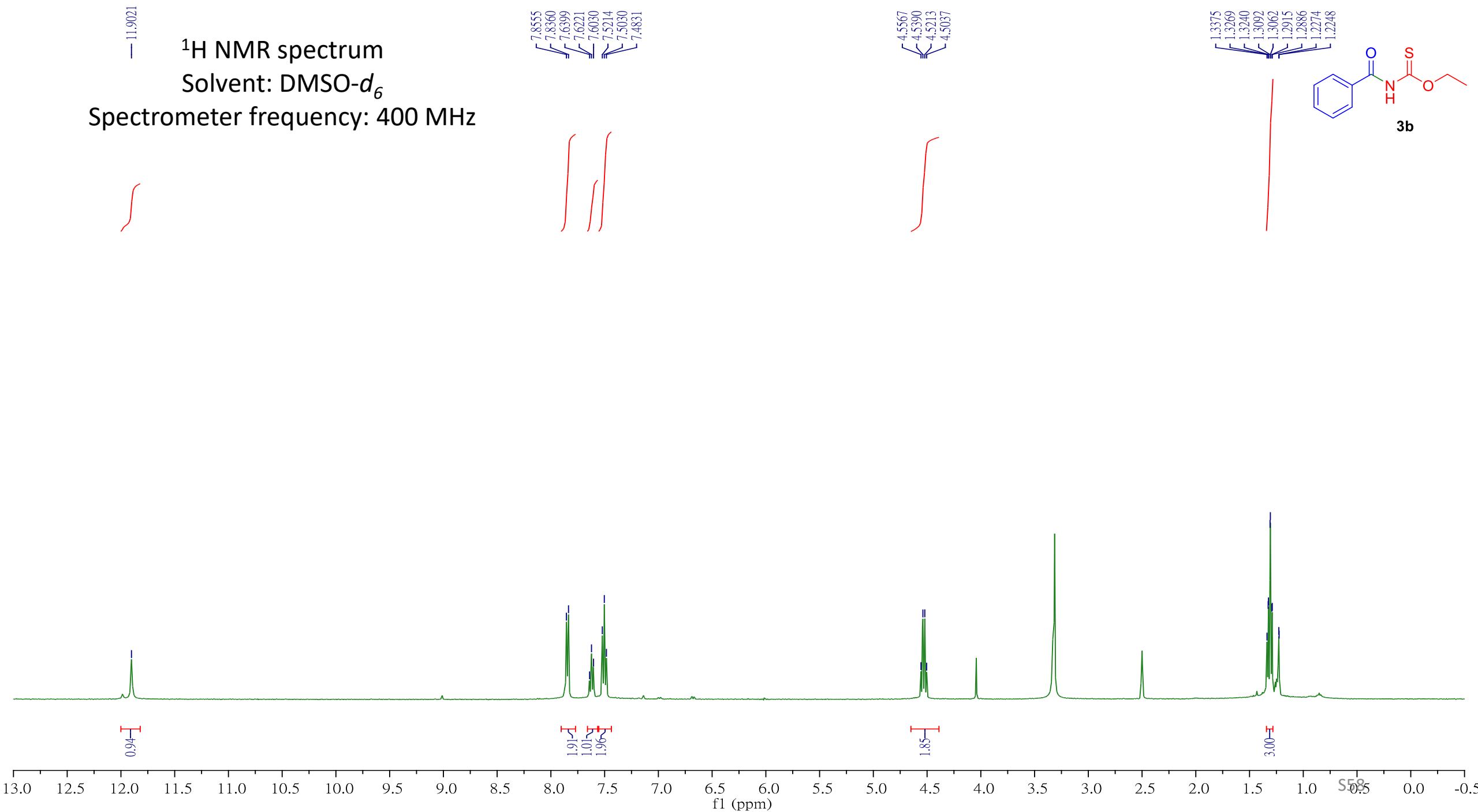
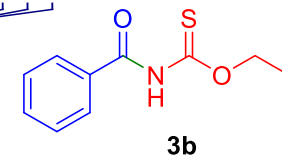


3a

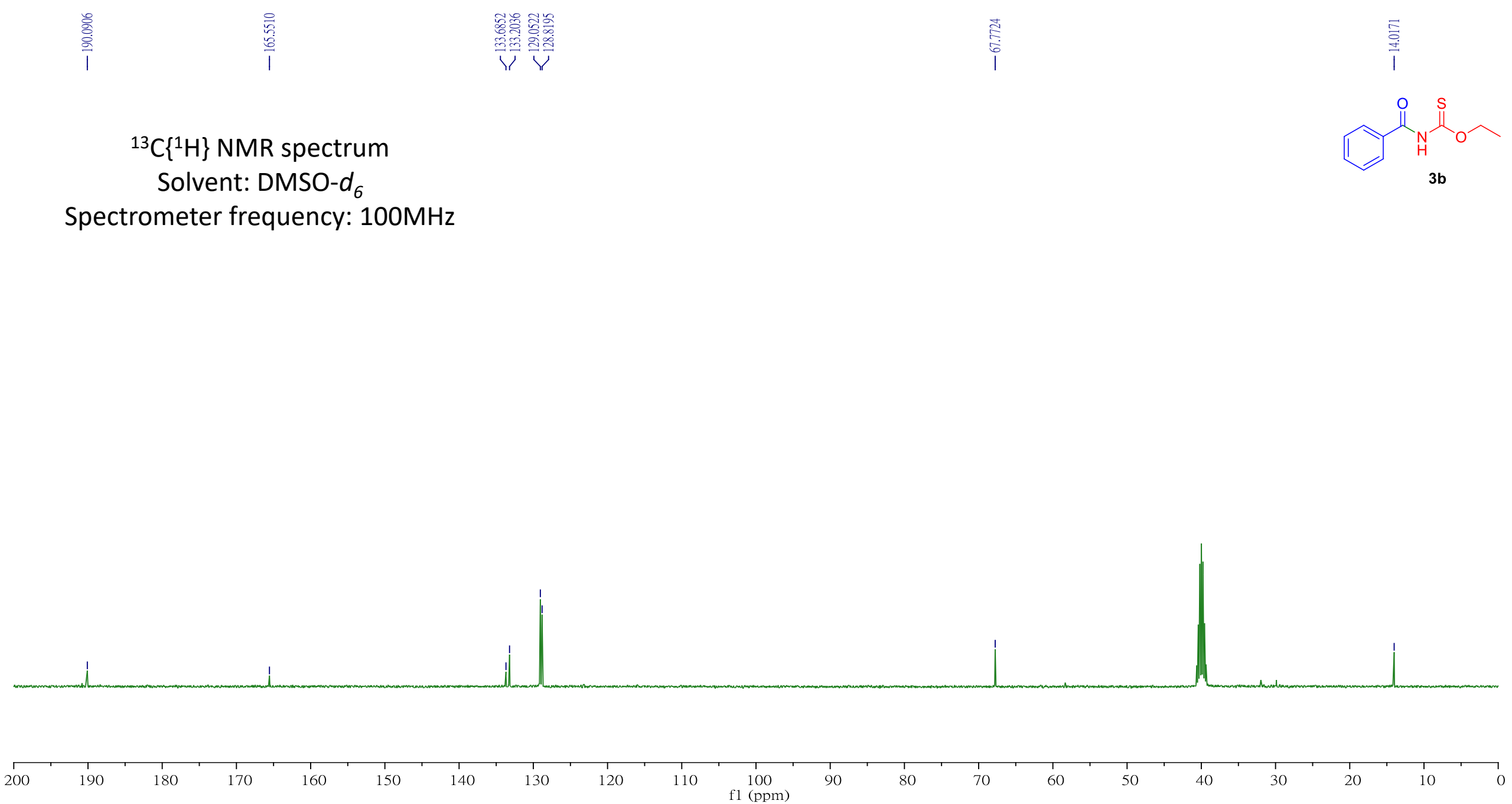
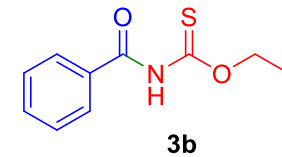
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz



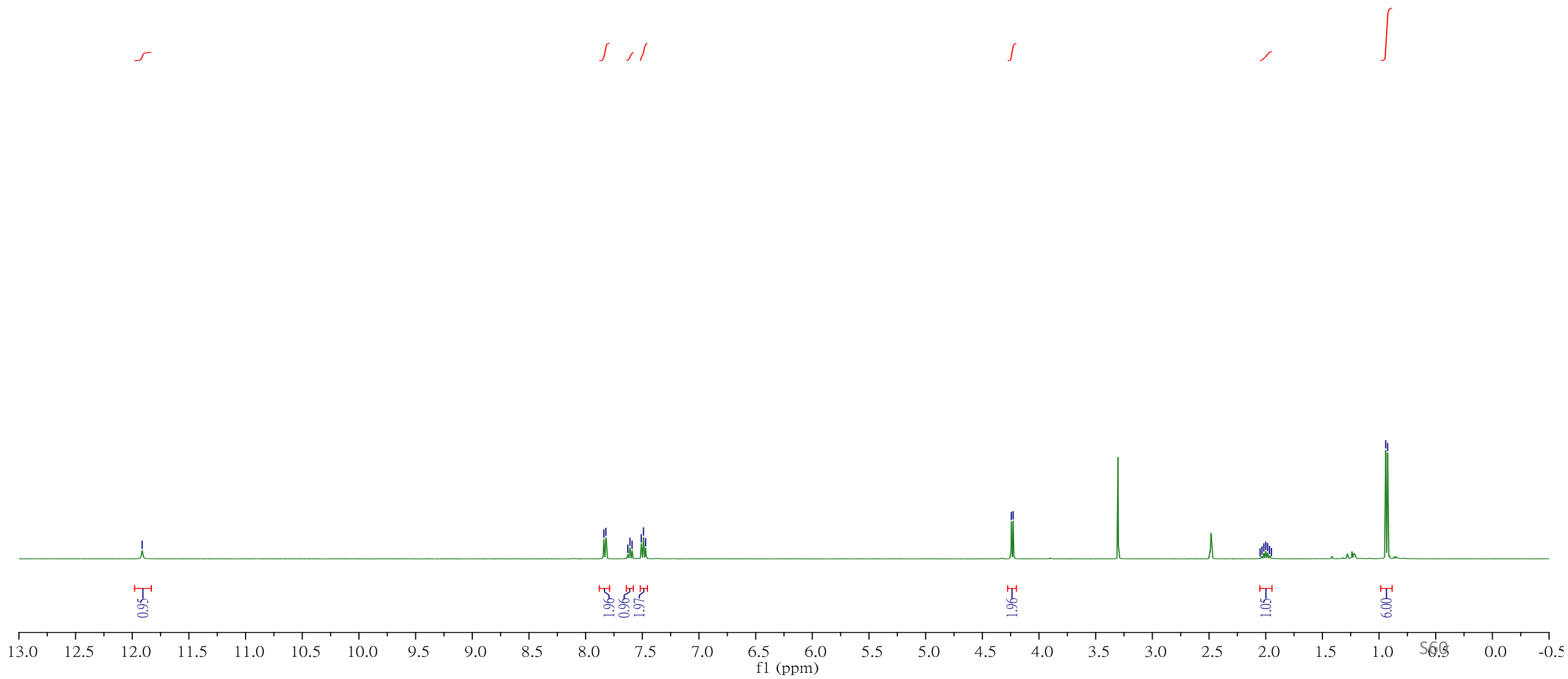
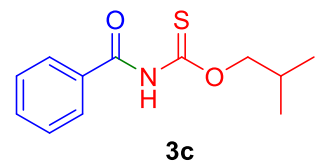
¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz

7.8389
7.8212
7.6276
7.6091
7.5906
7.5096
7.4898
7.4704

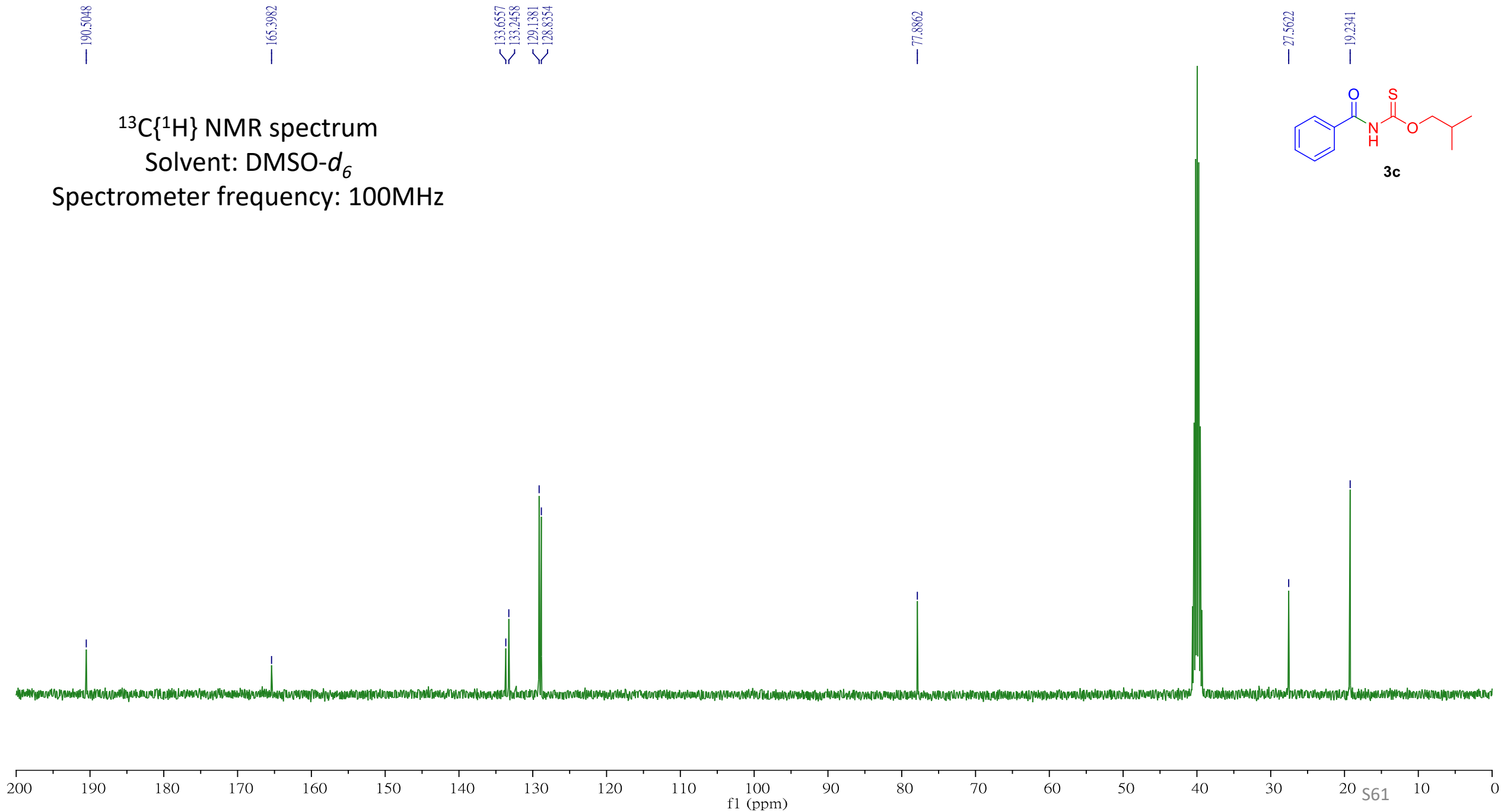
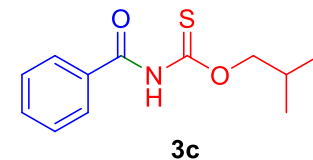
4.2442
4.2280

2.0496
2.0331
2.0162
1.9995
1.9830
1.9664
1.9498

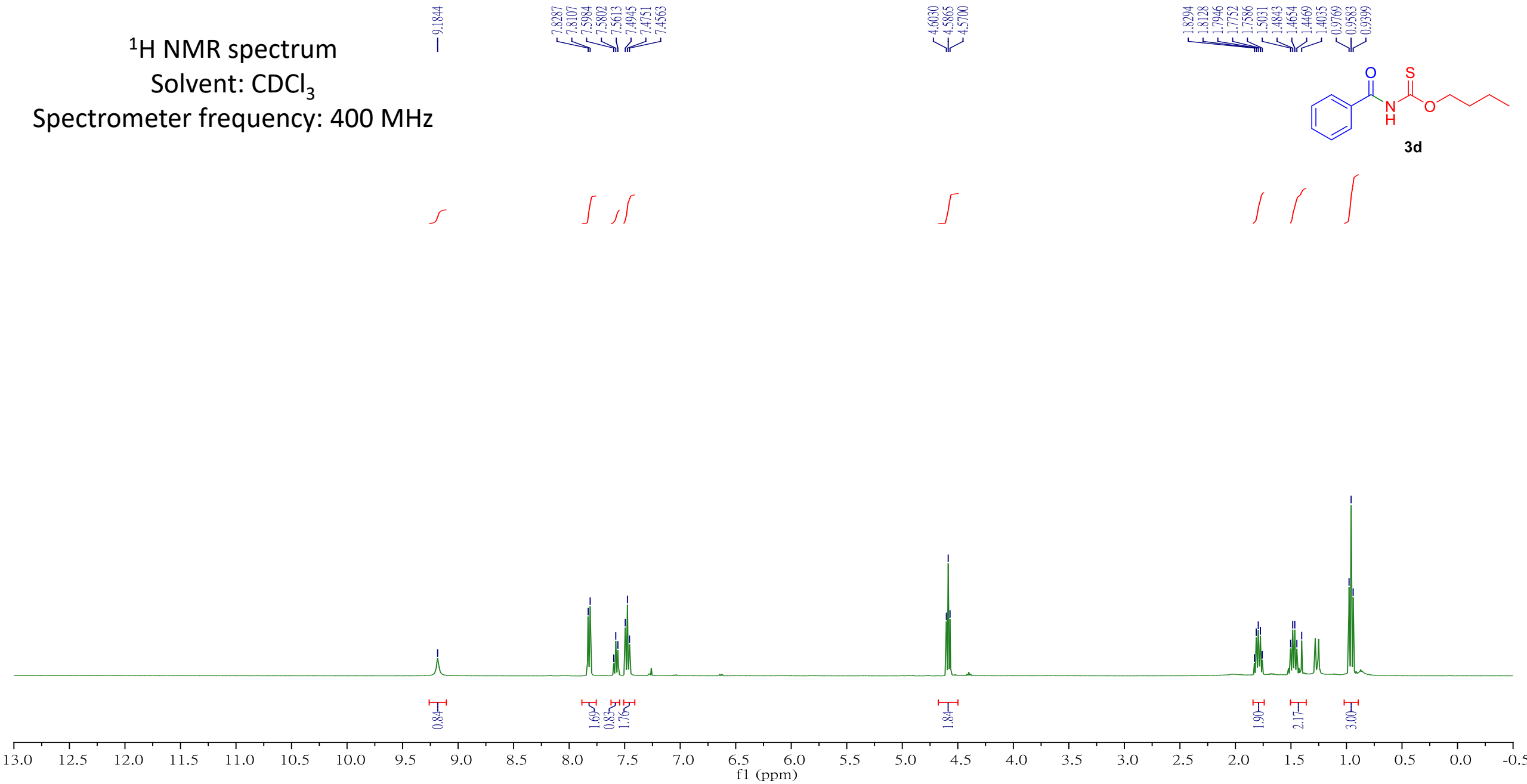
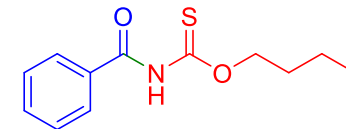
0.9413
0.9245



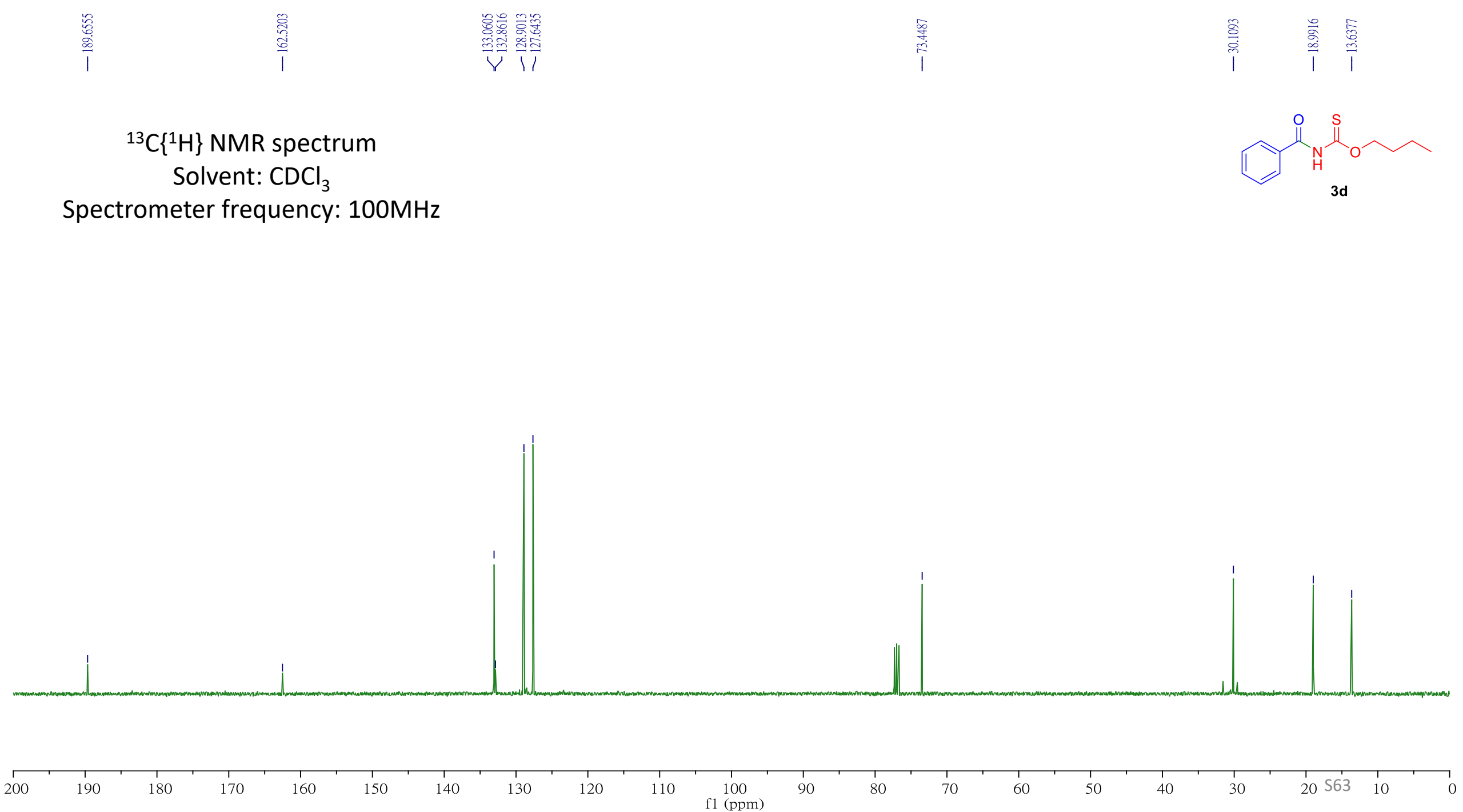
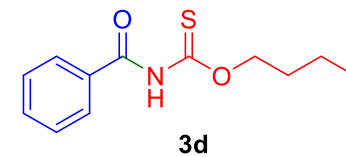
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



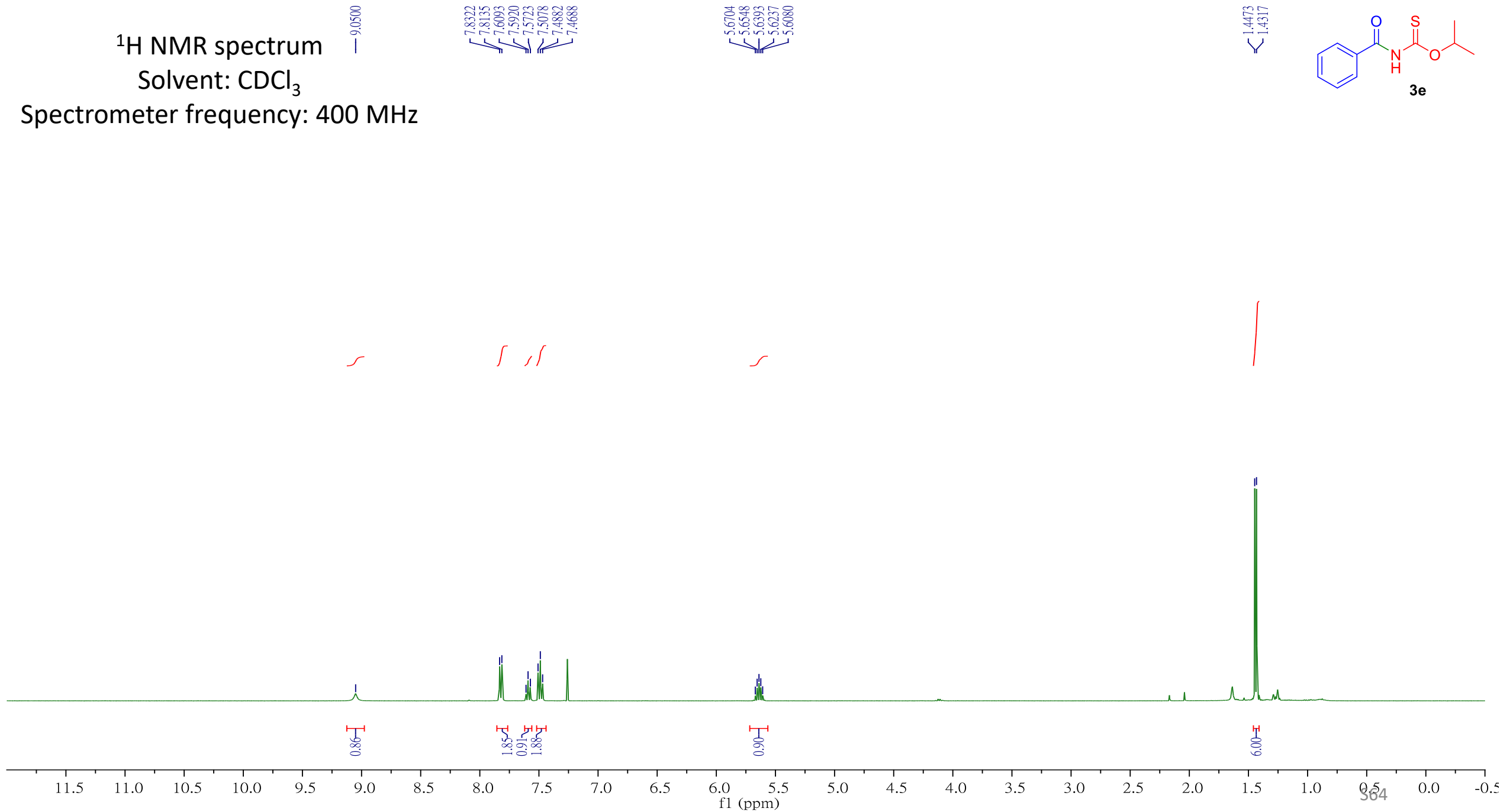
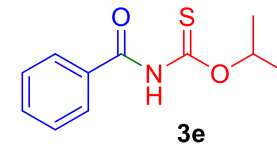
^1H NMR spectrum
Solvent: CDCl_3
Spectrometer frequency: 400 MHz



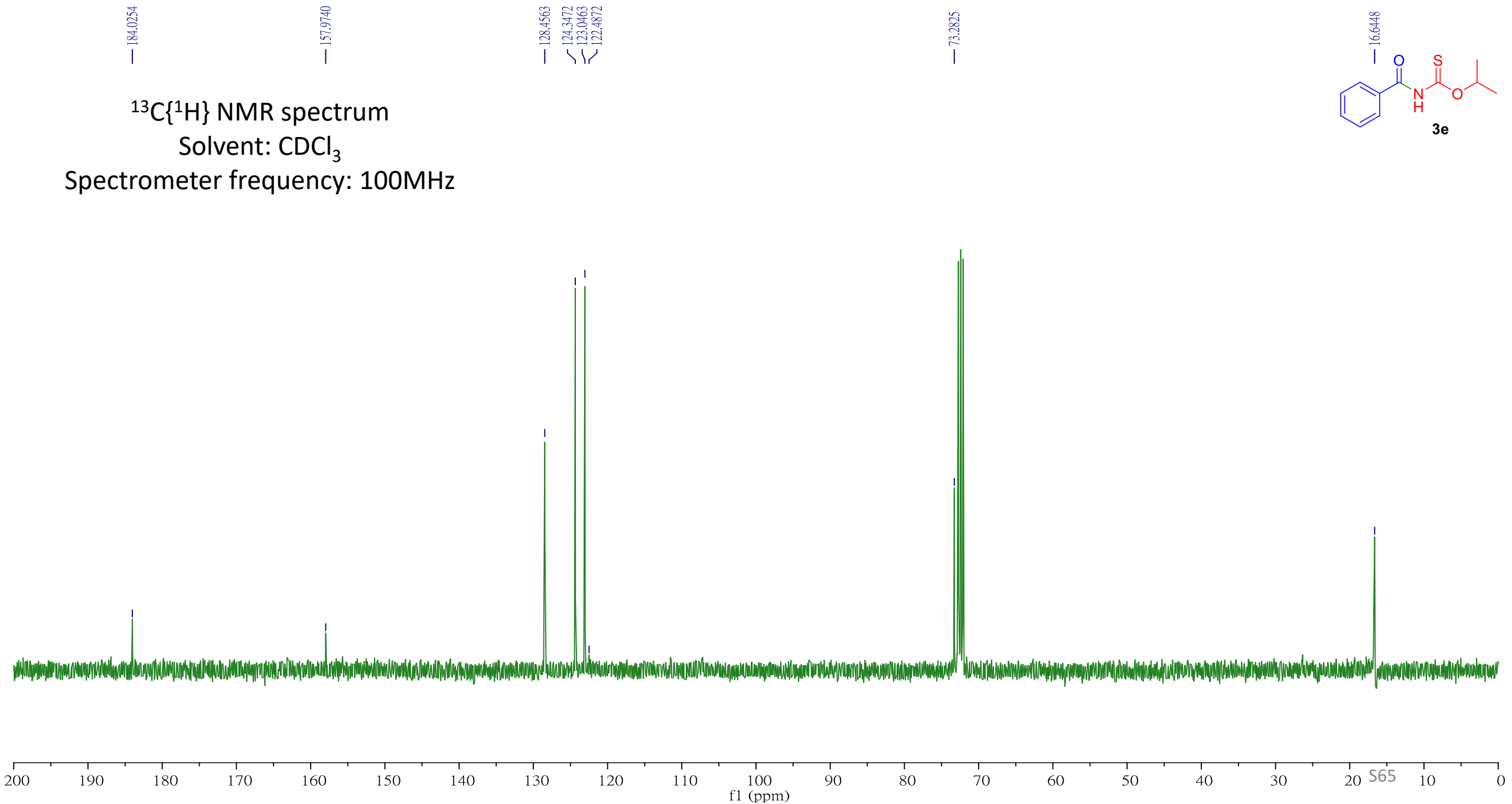
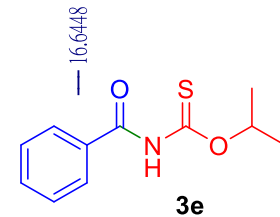
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: CDCl_3
Spectrometer frequency: 100MHz

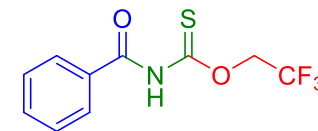


¹H NMR spectrum
Solvent: CDCl₃
Spectrometer frequency: 400 MHz



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: CDCl_3
Spectrometer frequency: 100MHz





3f

9.267

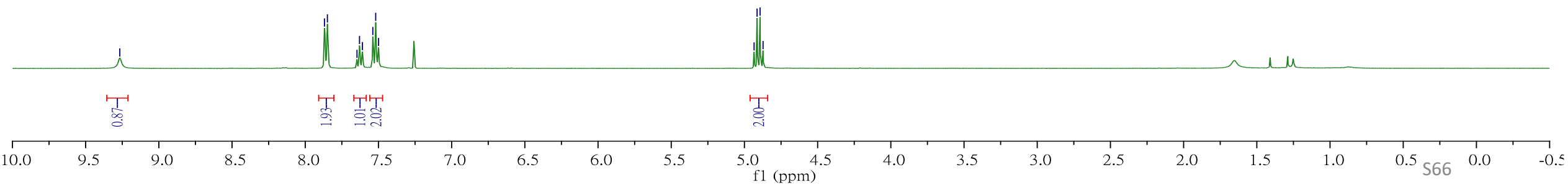
¹H NMR spectrum

Solvent: CDCl₃

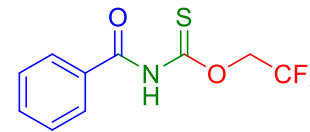
Spectrometer frequency: 400 MHz

7.869
7.849
7.647
7.629
7.609
7.538
7.519
7.499

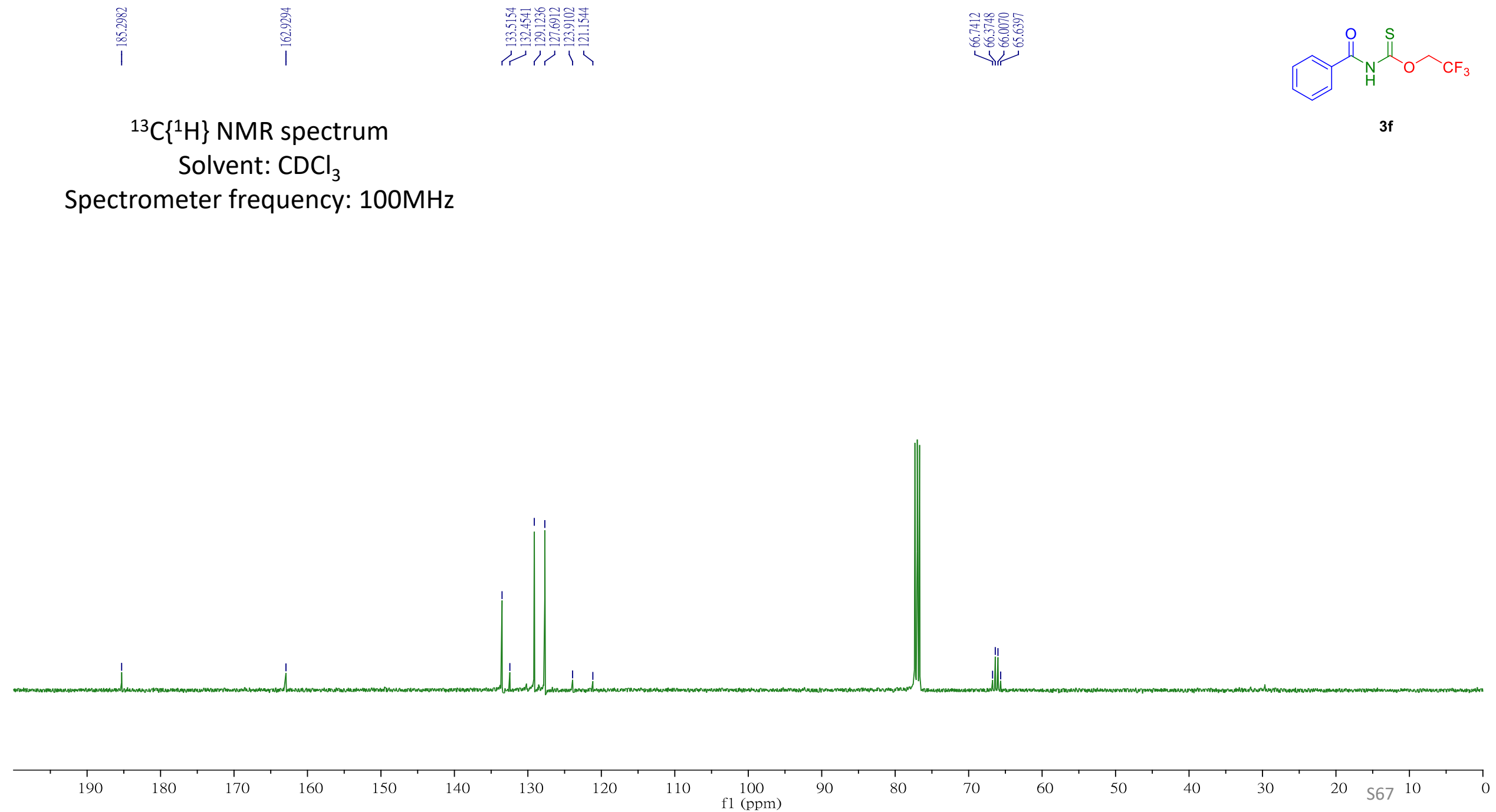
4.934
4.914
4.893
4.873



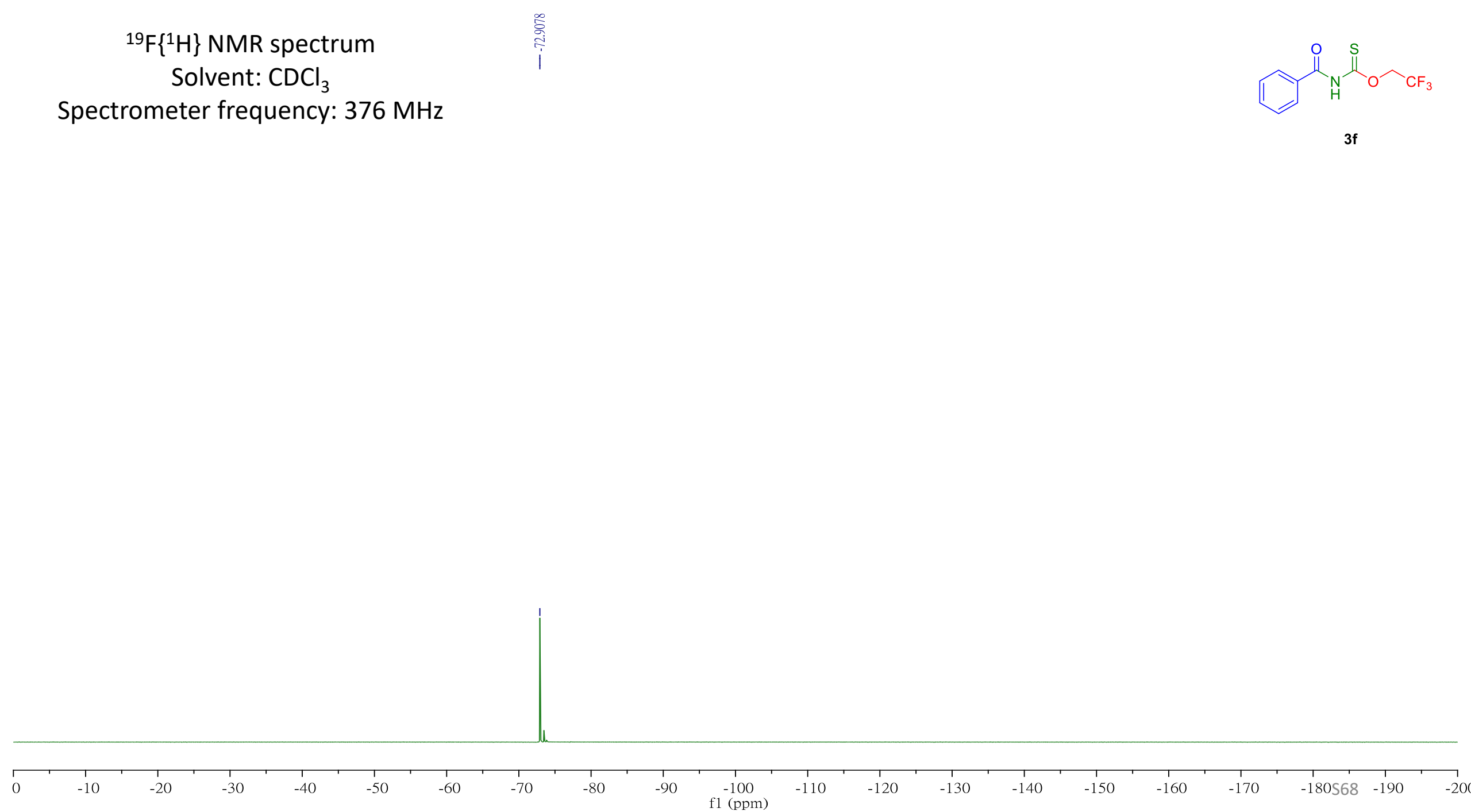
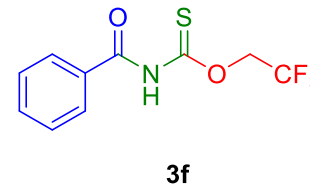
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: CDCl_3
Spectrometer frequency: 100MHz



3f



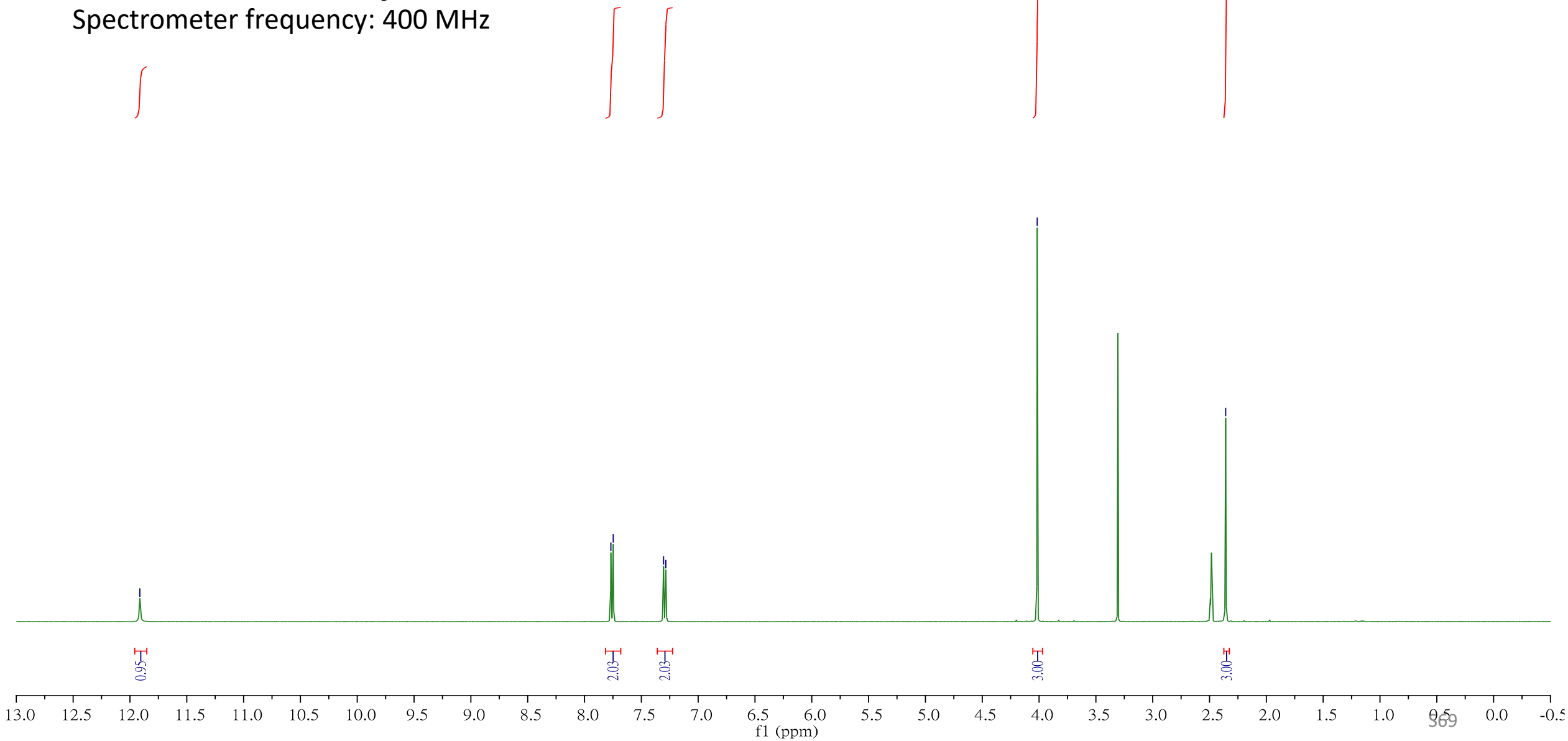
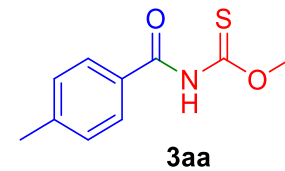
$^{19}\text{F}\{^1\text{H}\}$ NMR spectrum
Solvent: CDCl_3
Spectrometer frequency: 376 MHz

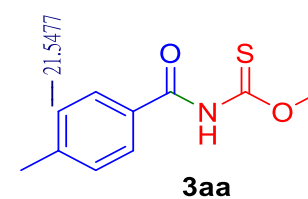


¹H NMR spectrum

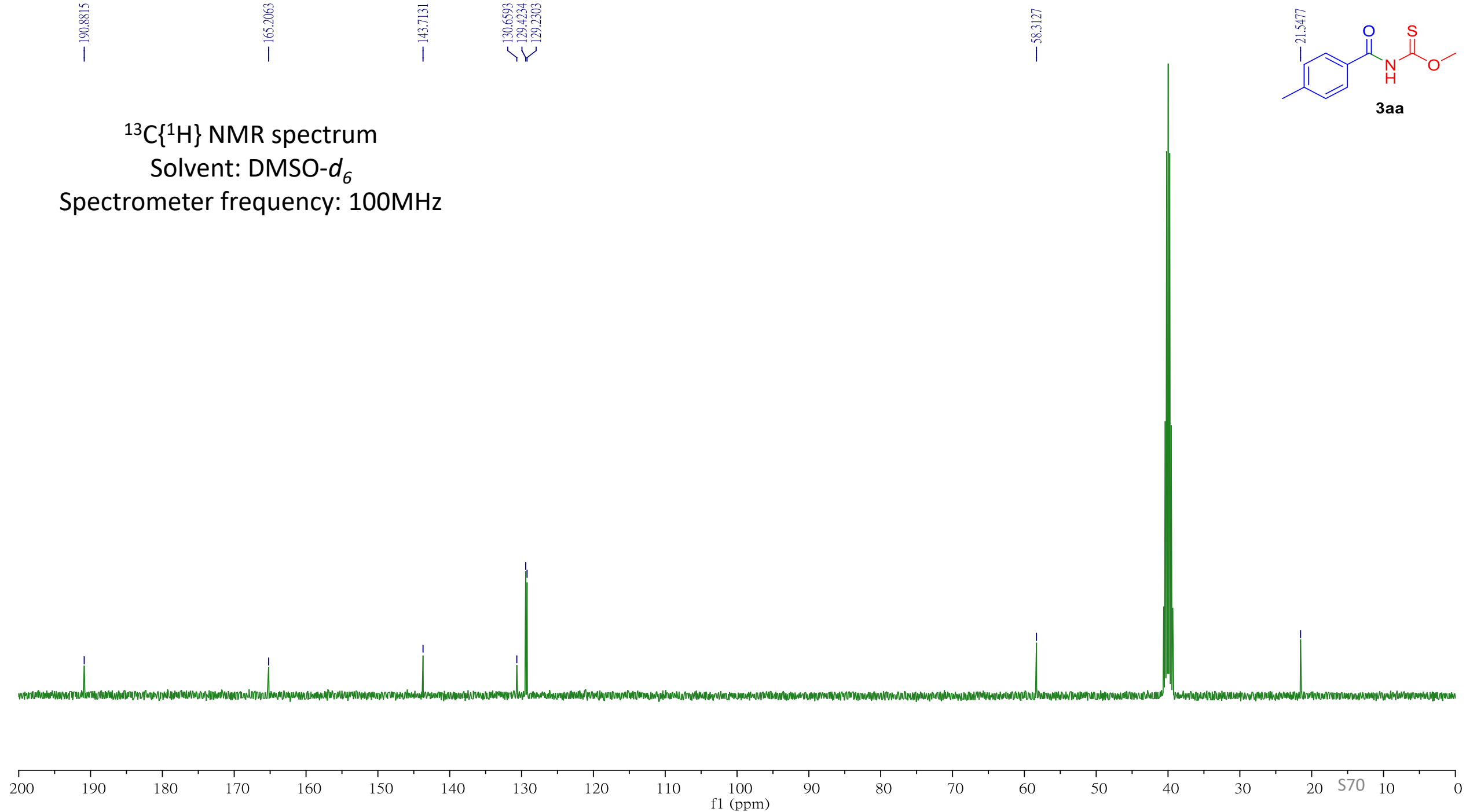
Solvent: DMSO-*d*₆

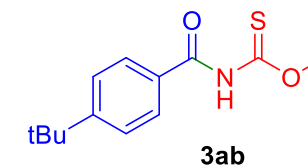
Spectrometer frequency: 400 MHz



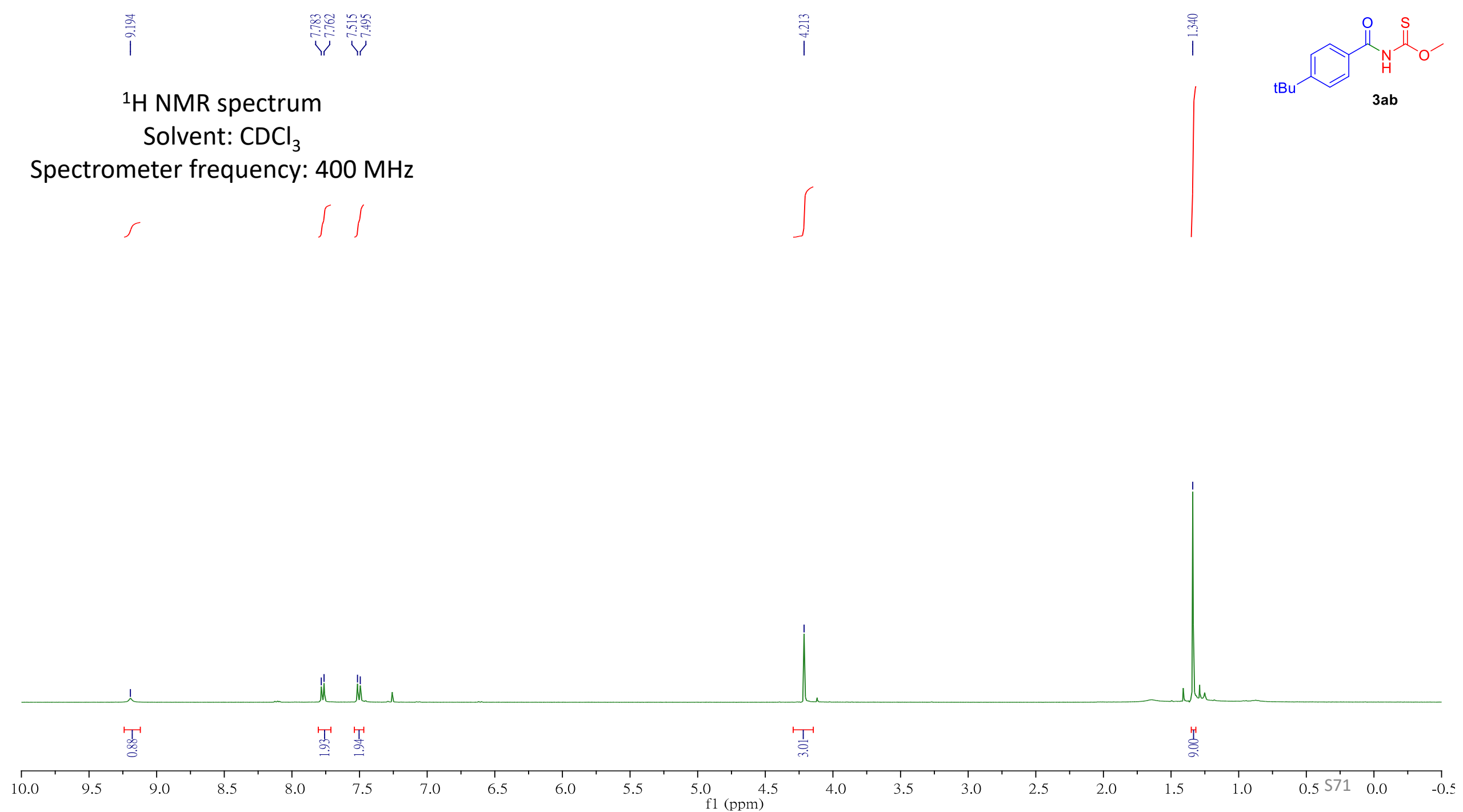


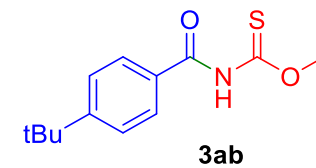
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



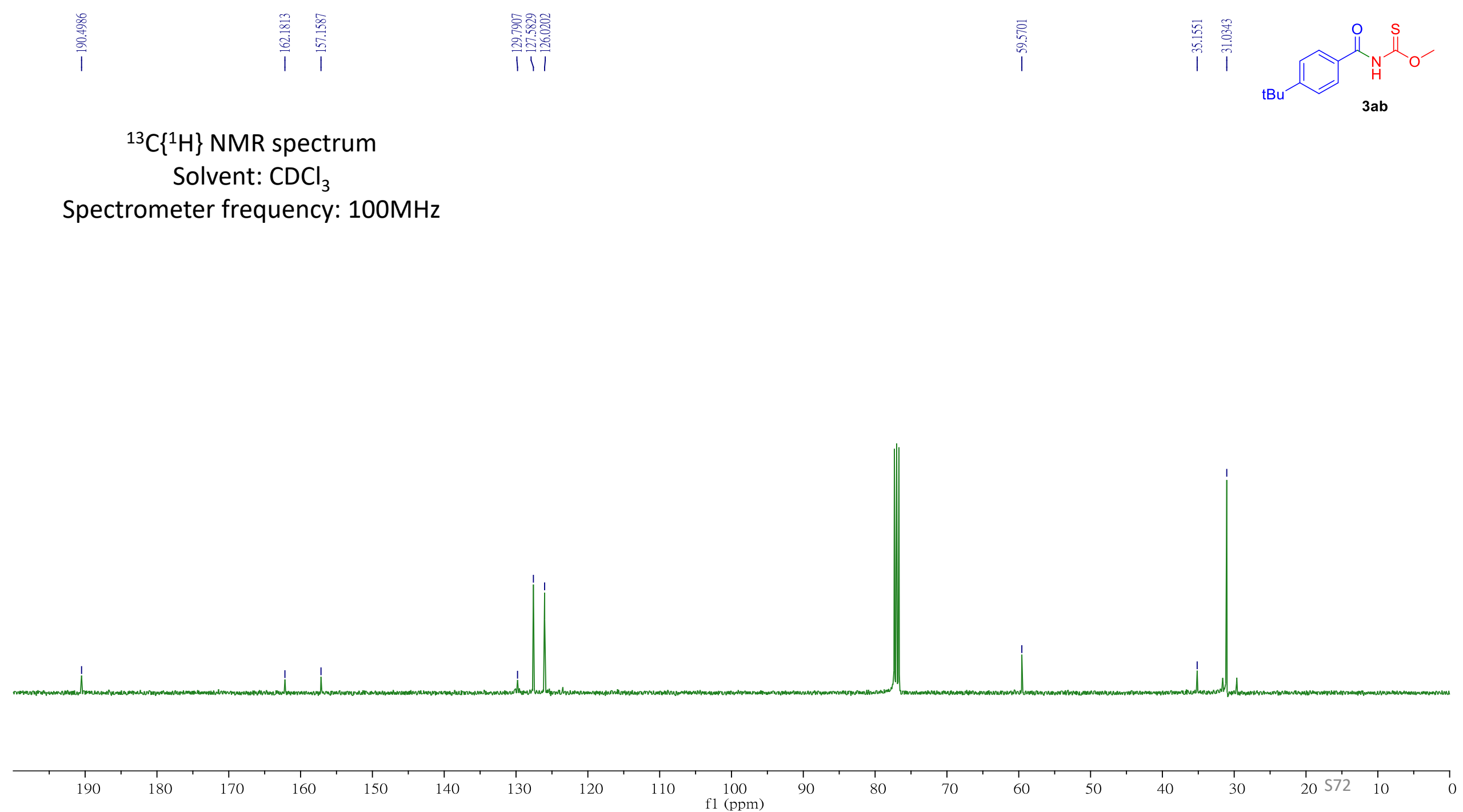


¹H NMR spectrum
Solvent: CDCl₃
Spectrometer frequency: 400 MHz





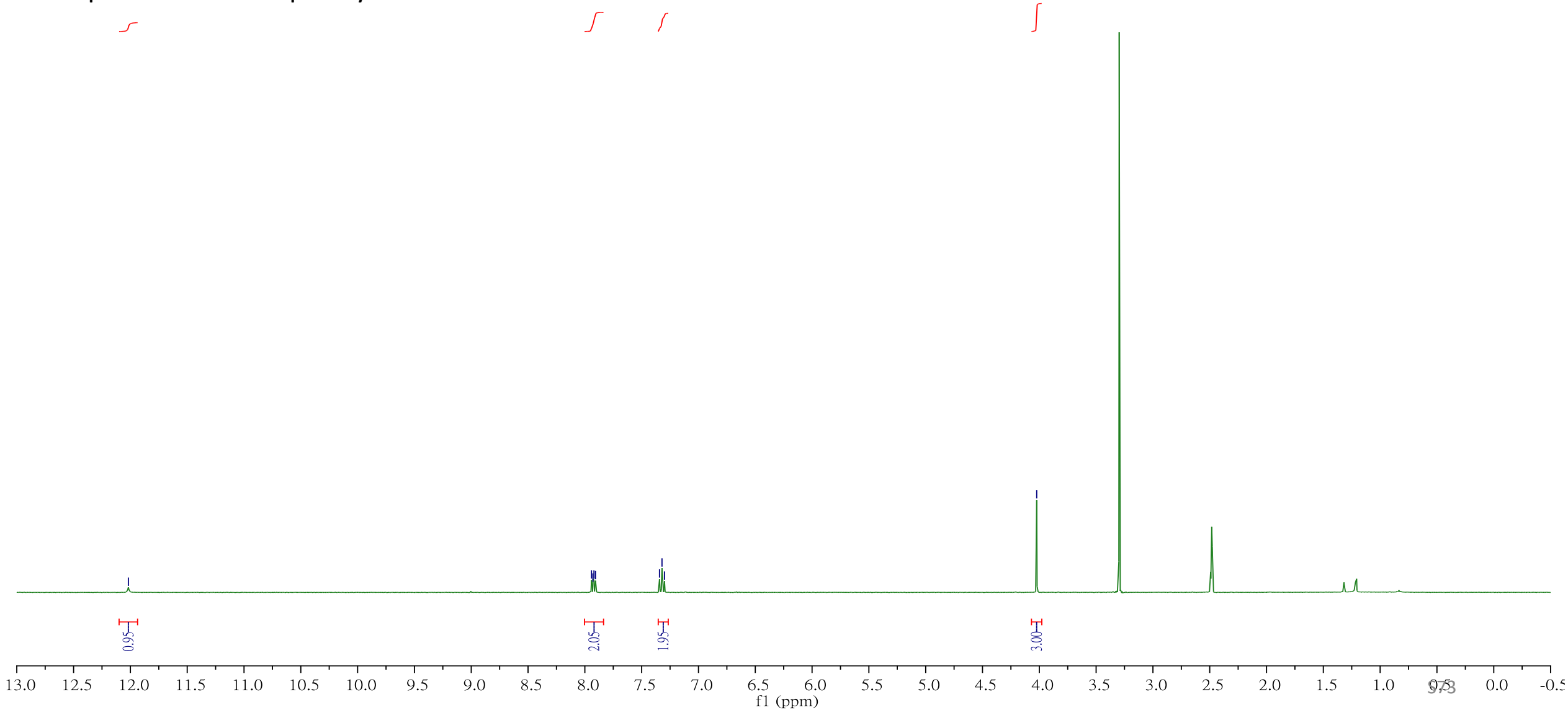
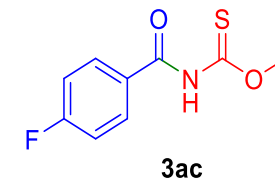
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
 Solvent: CDCl_3
 Spectrometer frequency: 100MHz

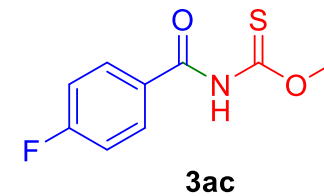


¹H NMR spectrum

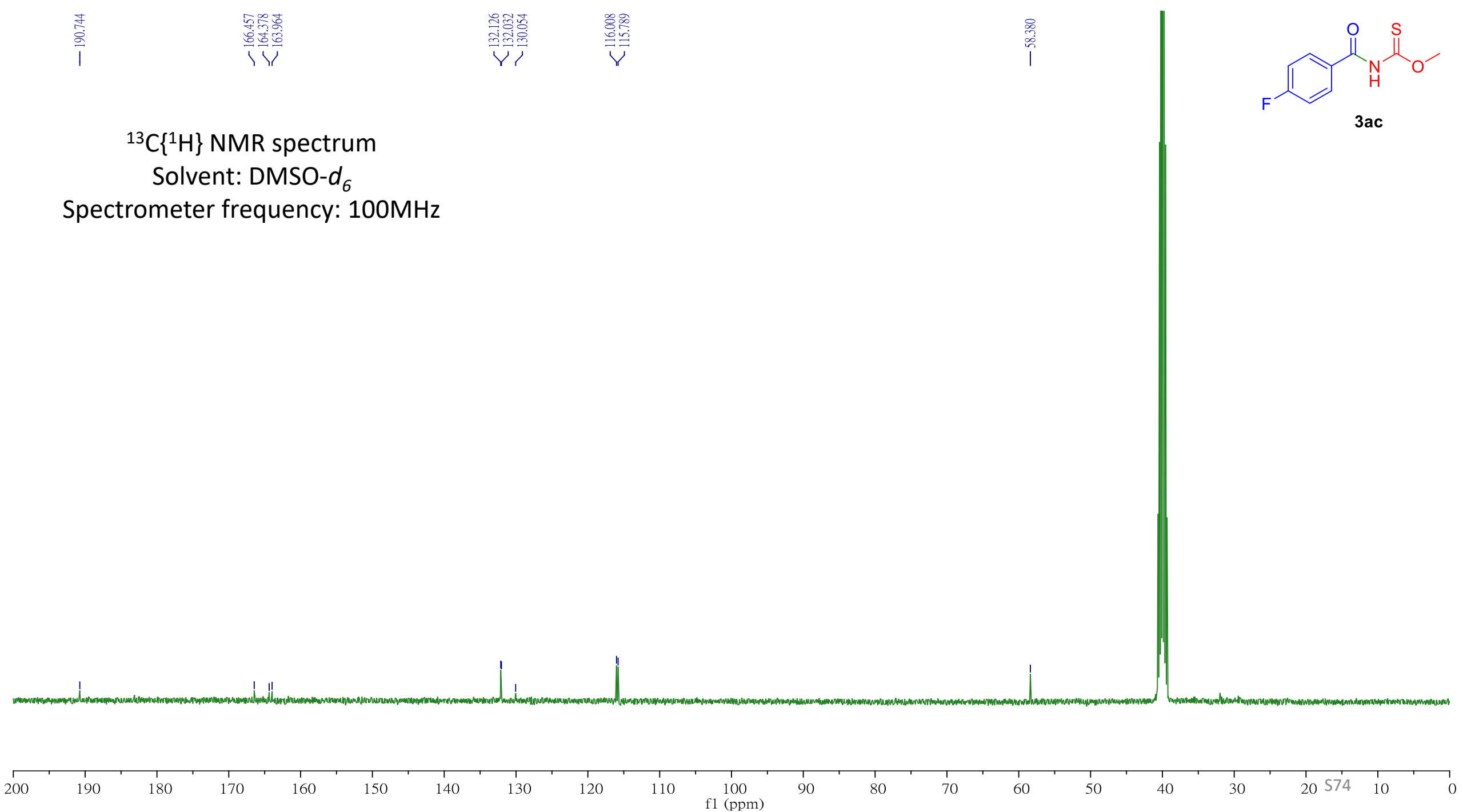
Solvent: DMSO-*d*₆

Spectrometer frequency: 400 MHz

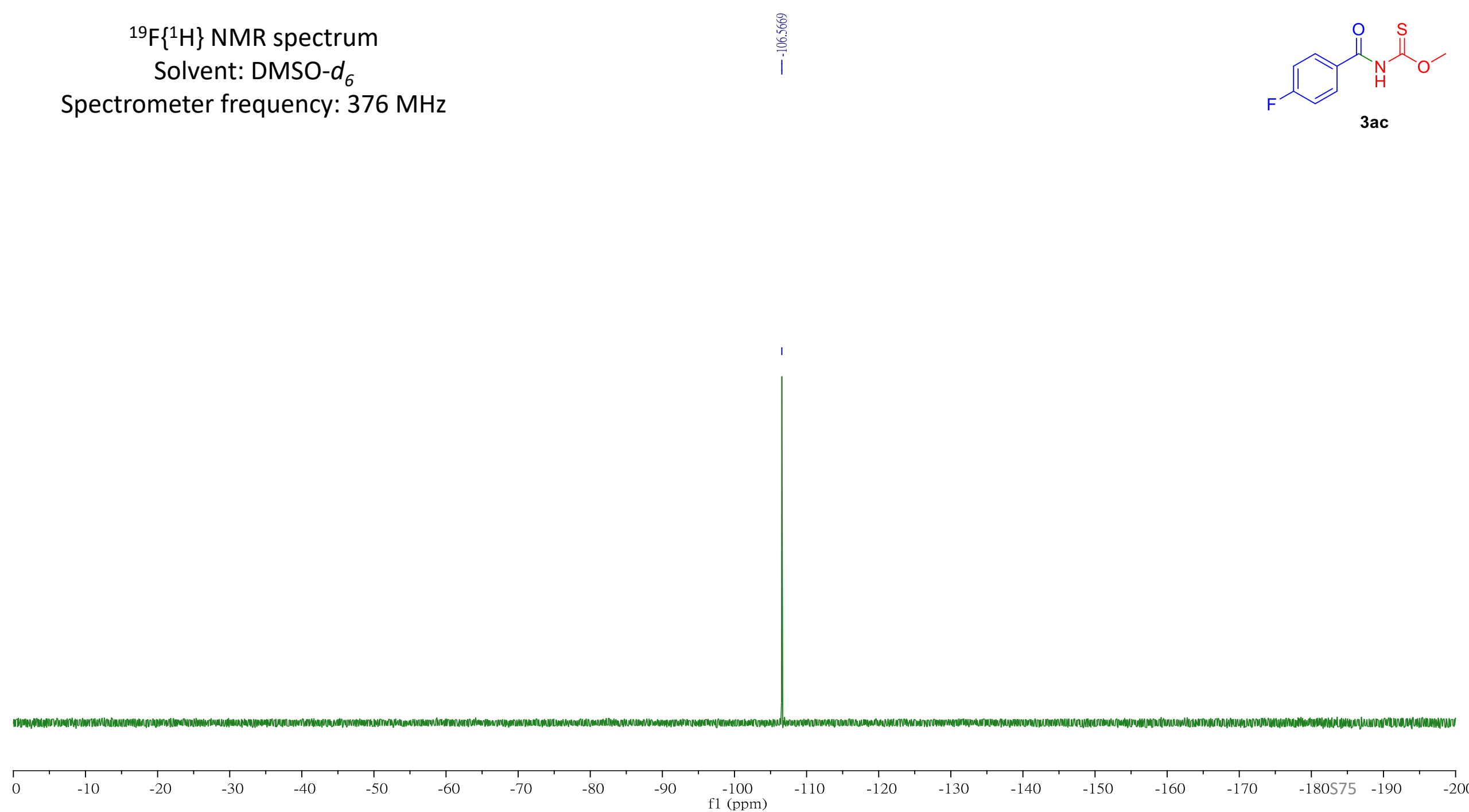
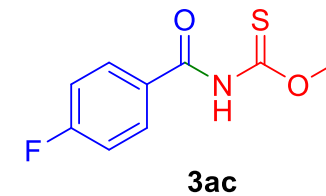




$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



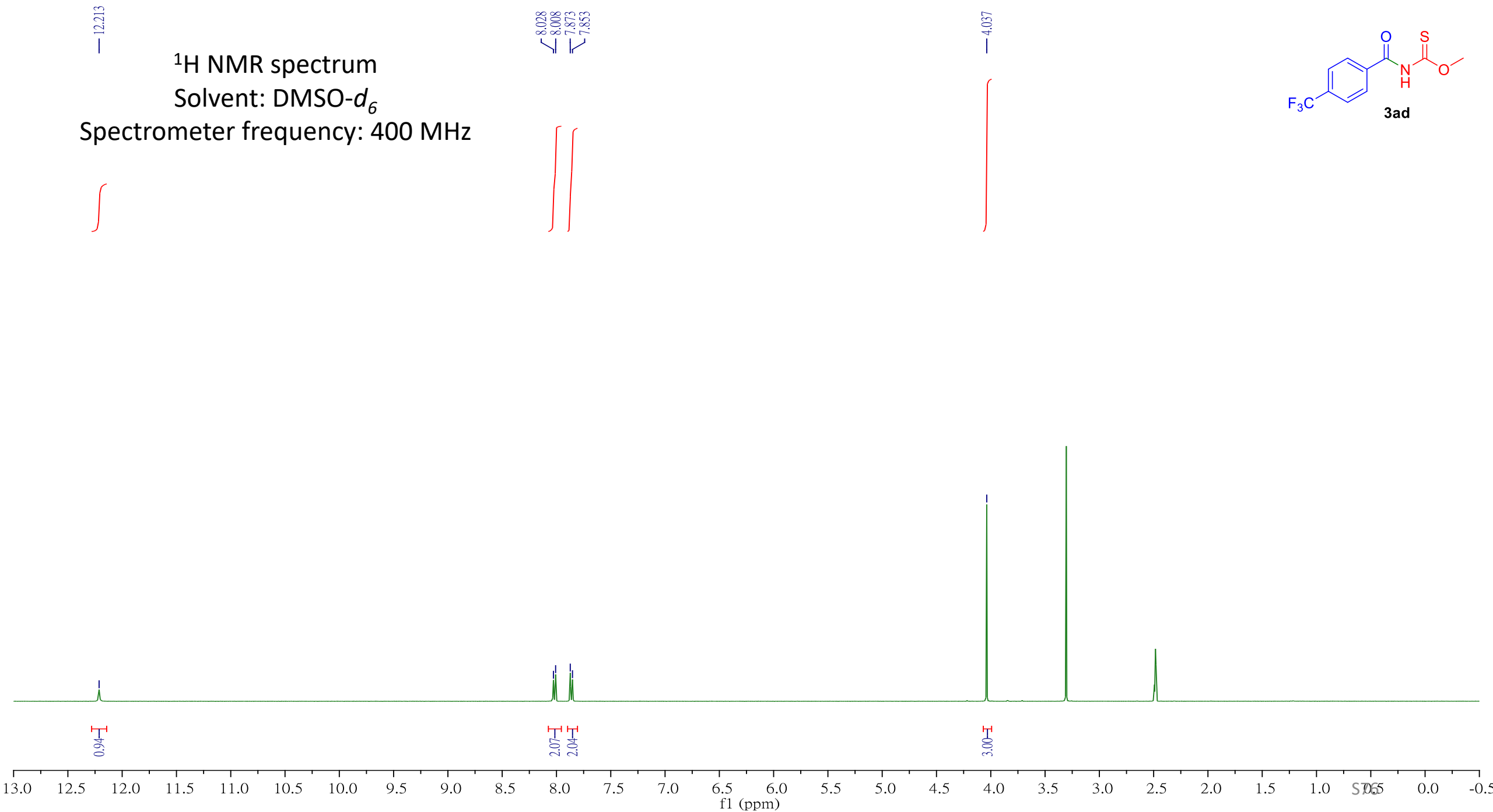
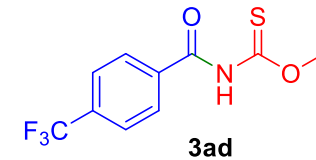
$^{19}\text{F}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 376 MHz

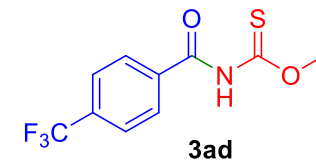


¹H NMR spectrum

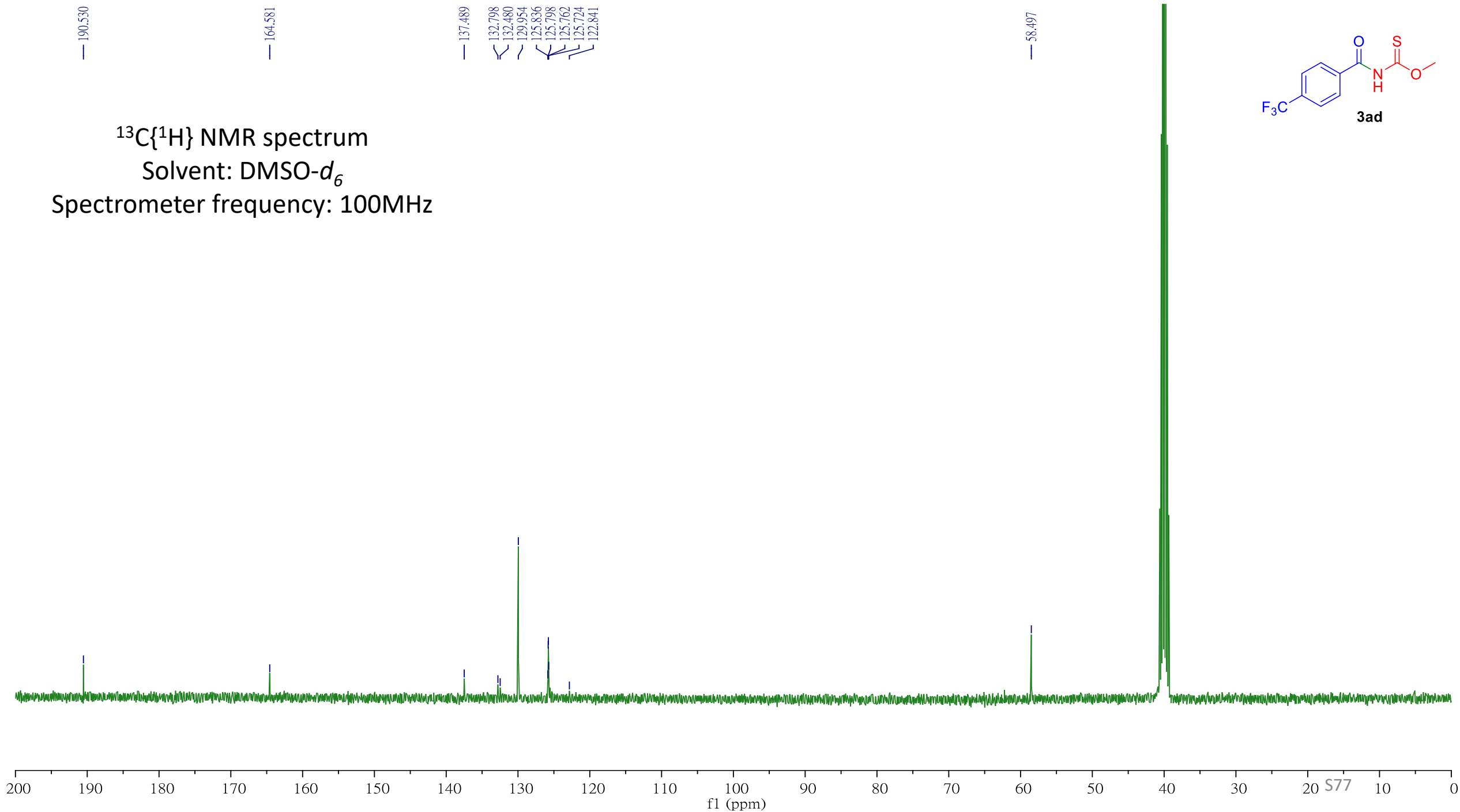
Solvent: DMSO-*d*₆

Spectrometer frequency: 400 MHz

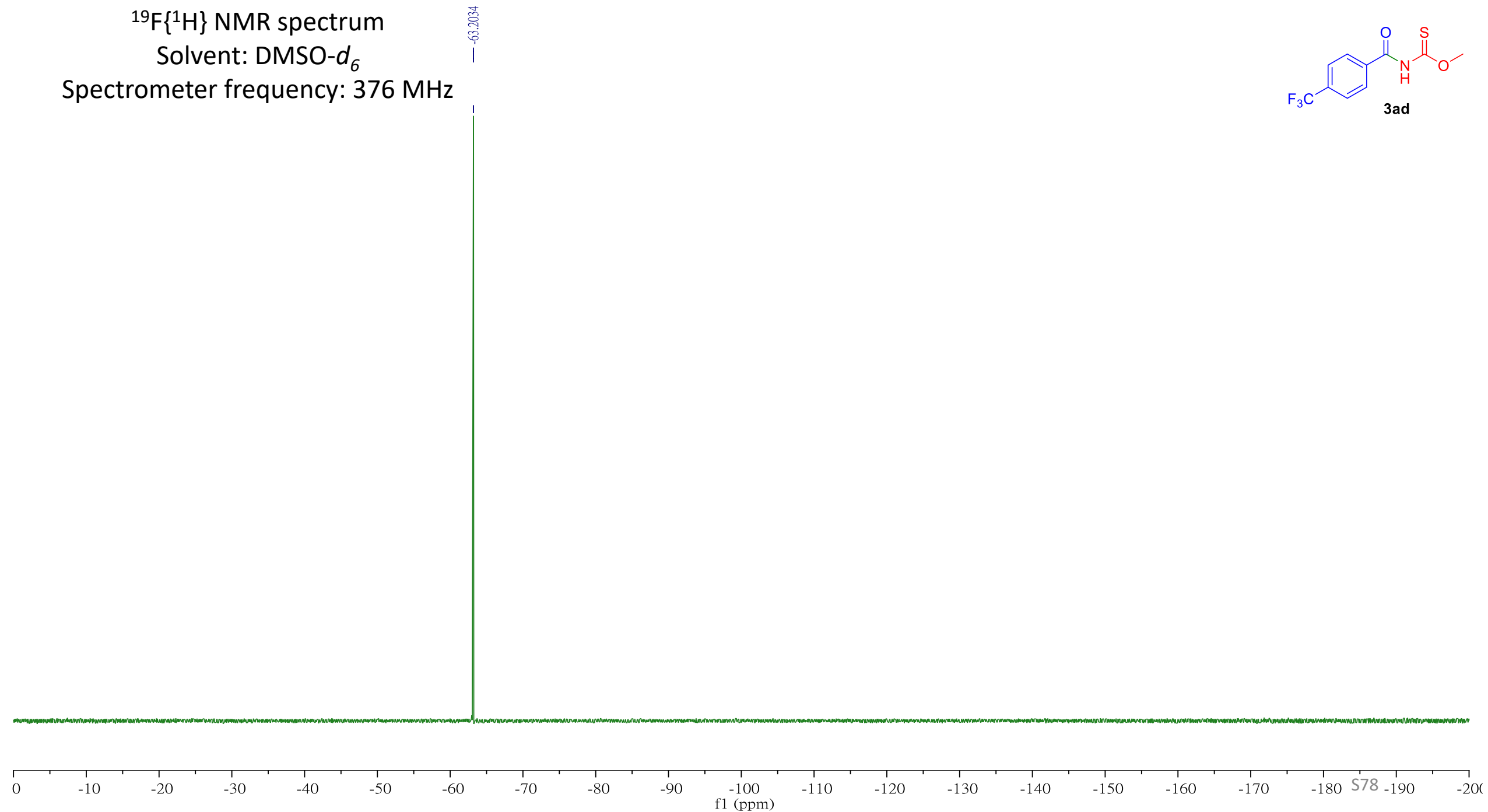
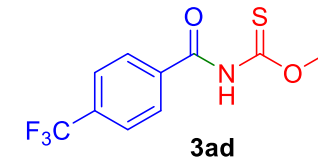




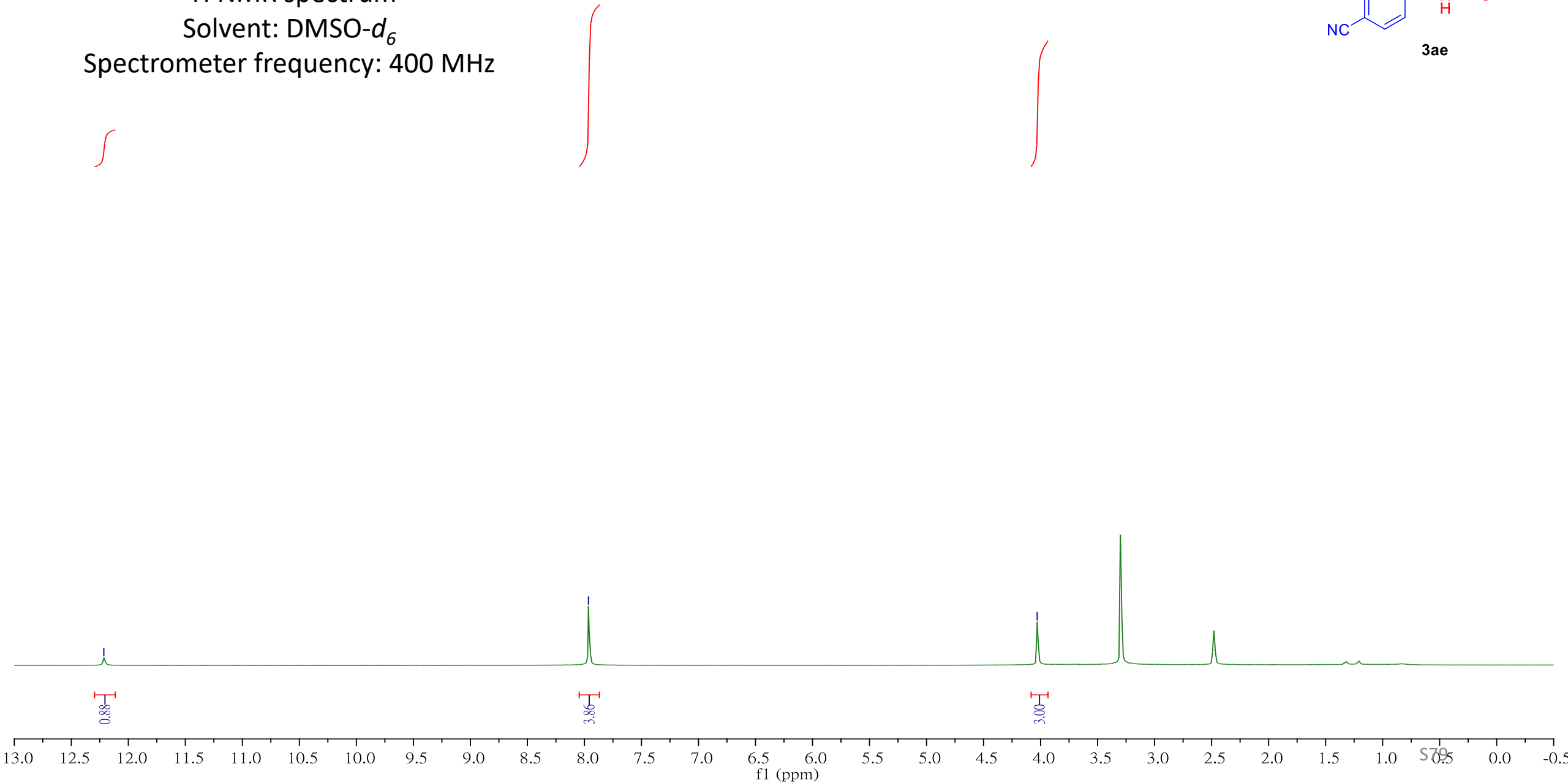
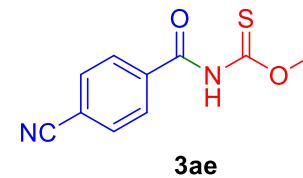
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
 Solvent: $\text{DMSO-}d_6$
 Spectrometer frequency: 100MHz

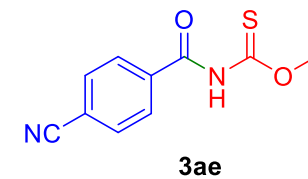


$^{19}\text{F}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 376 MHz

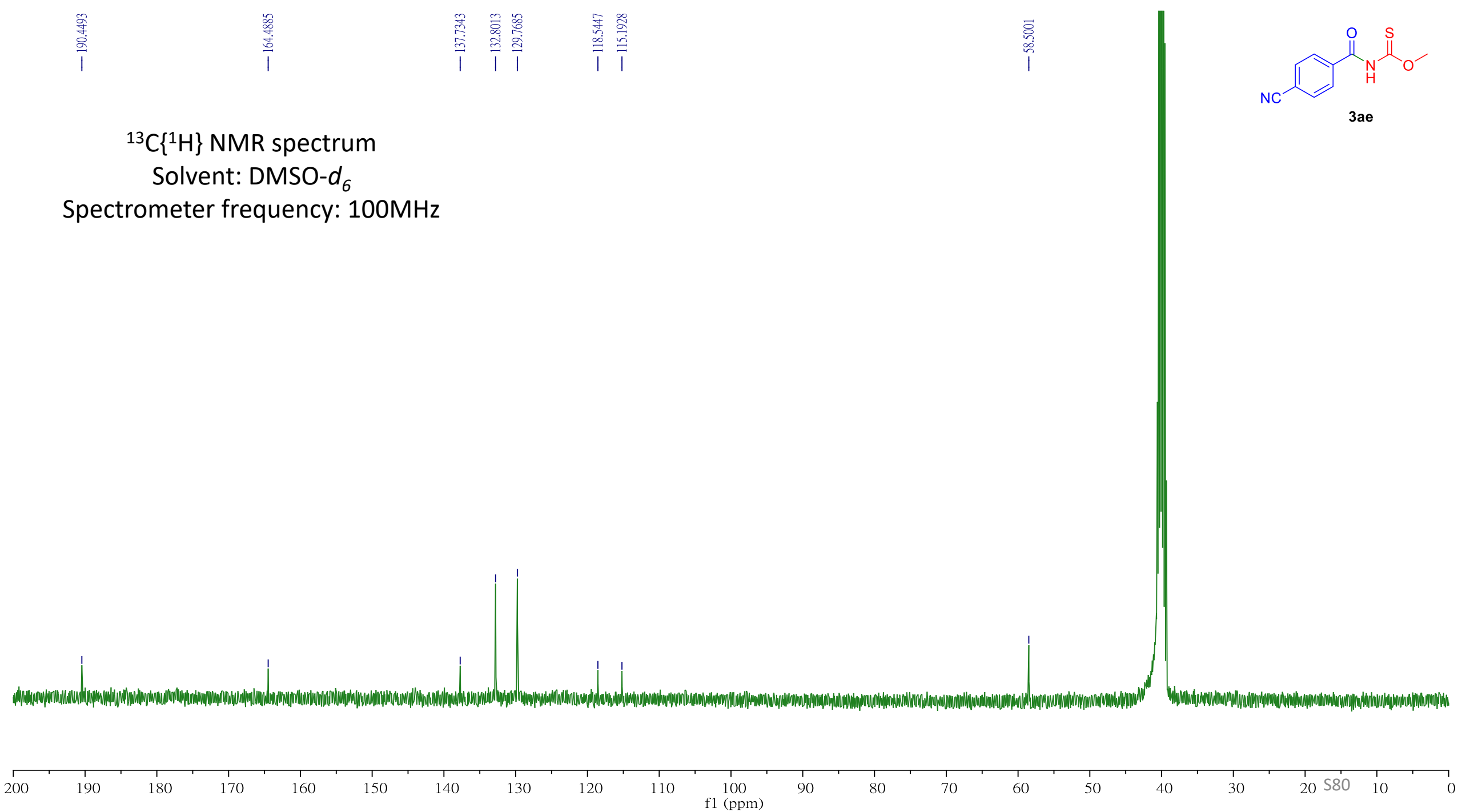


¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz





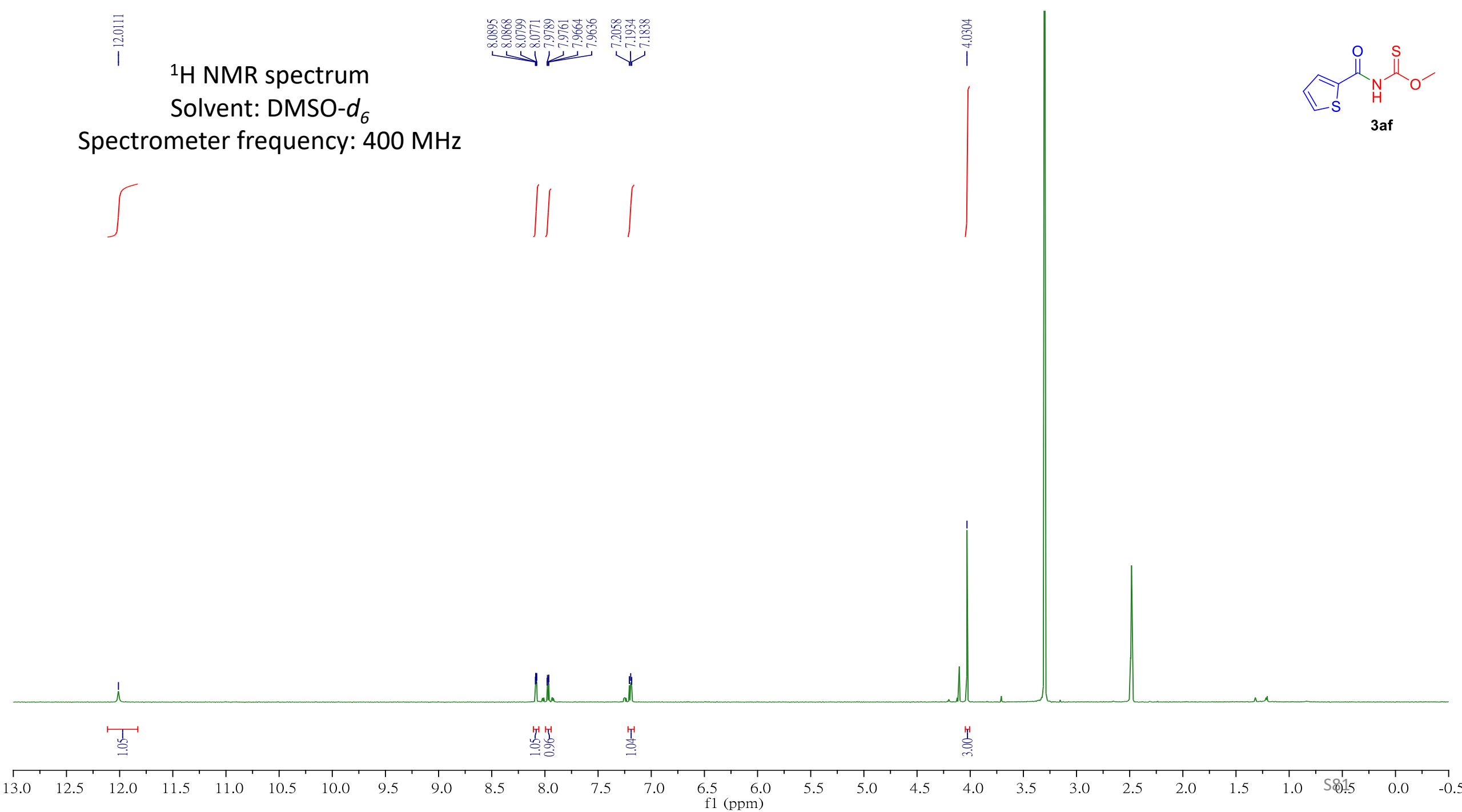
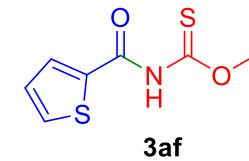
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



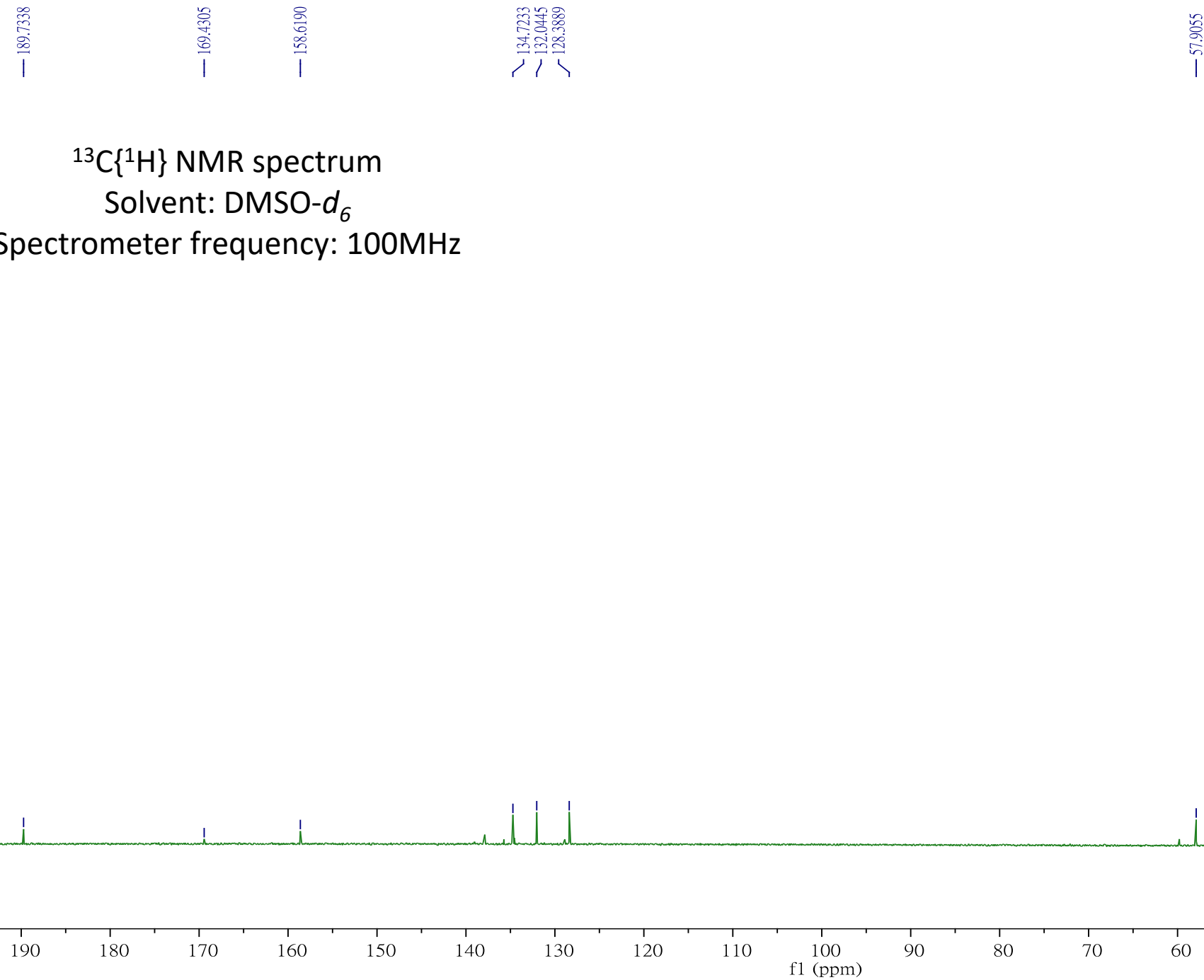
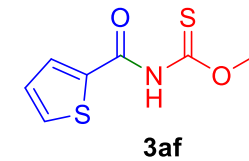
¹H NMR spectrum

Solvent: DMSO-*d*₆

Spectrometer frequency: 400 MHz



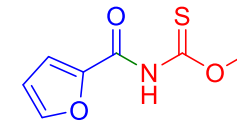
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Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz



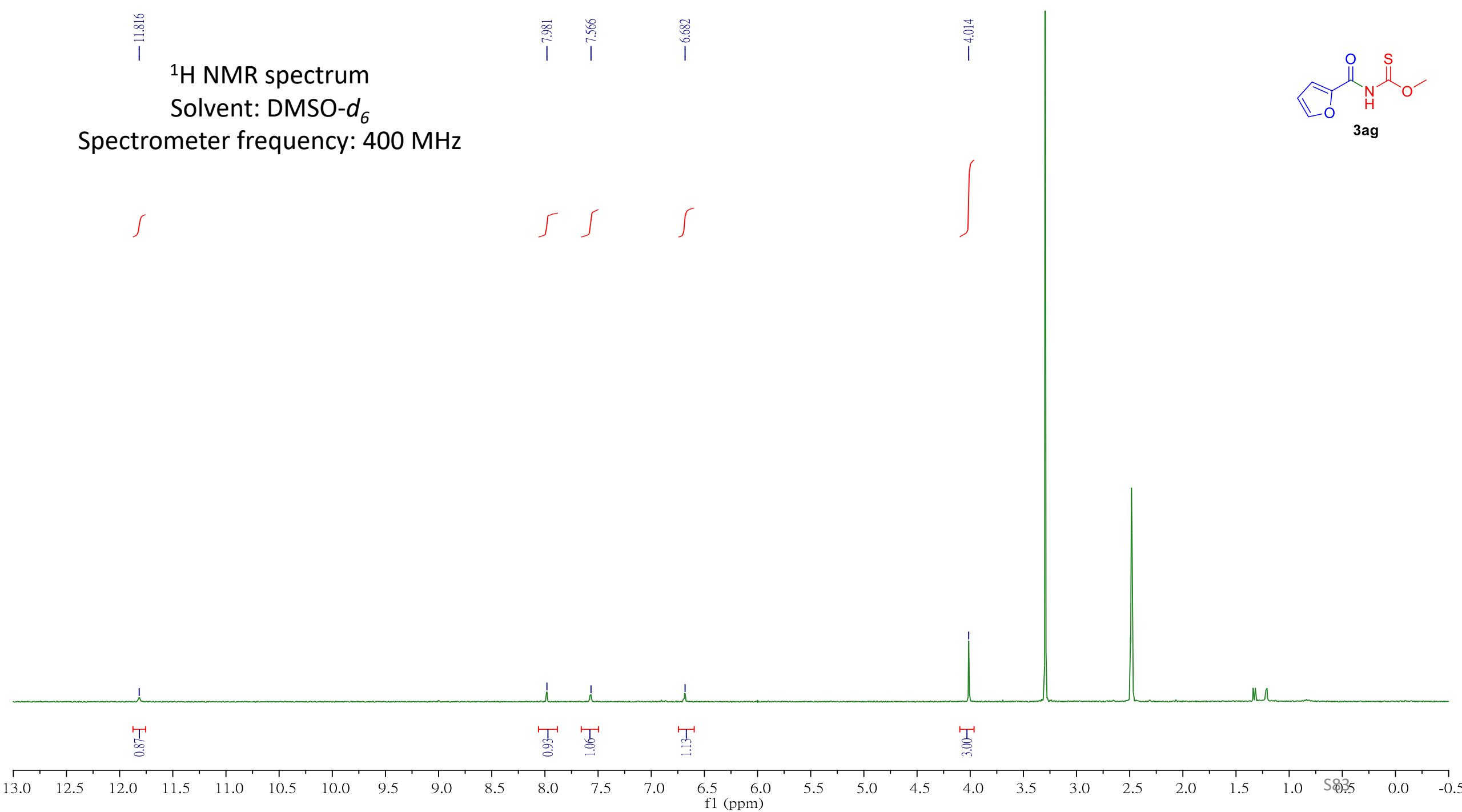
¹H NMR spectrum

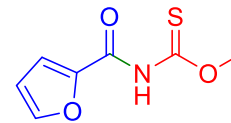
Solvent: DMSO-*d*₆

Spectrometer frequency: 400 MHz



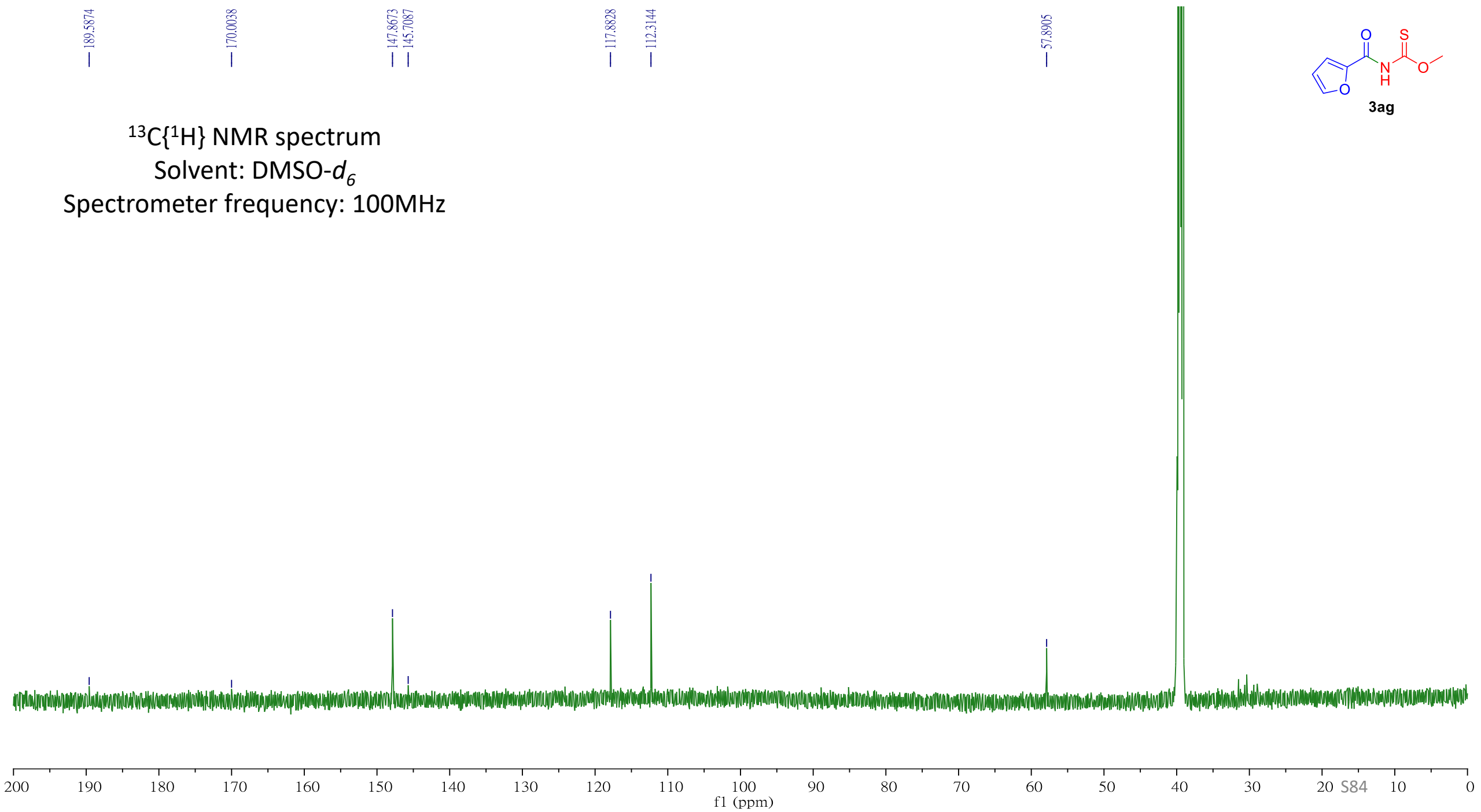
3ag



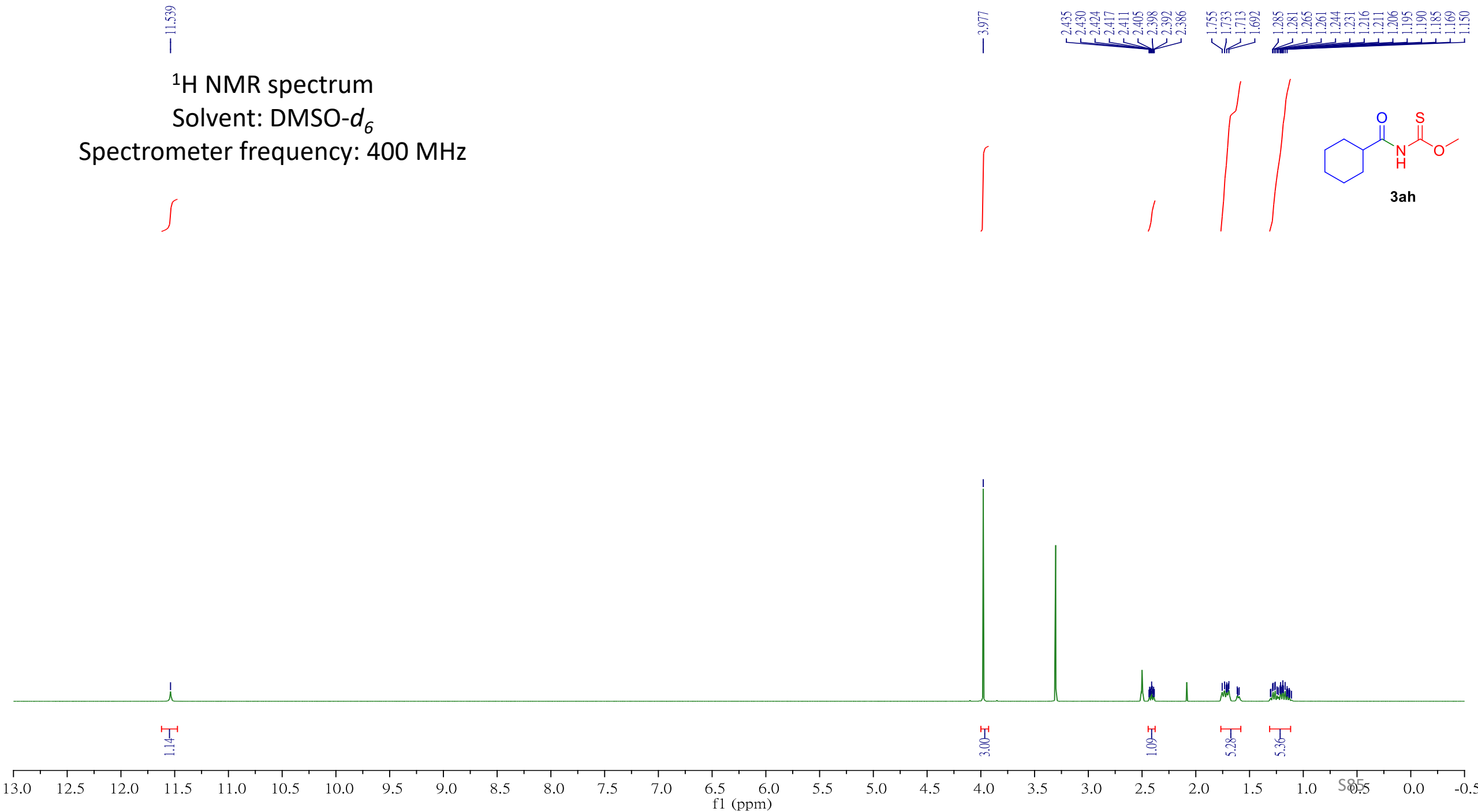
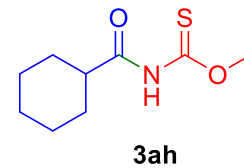


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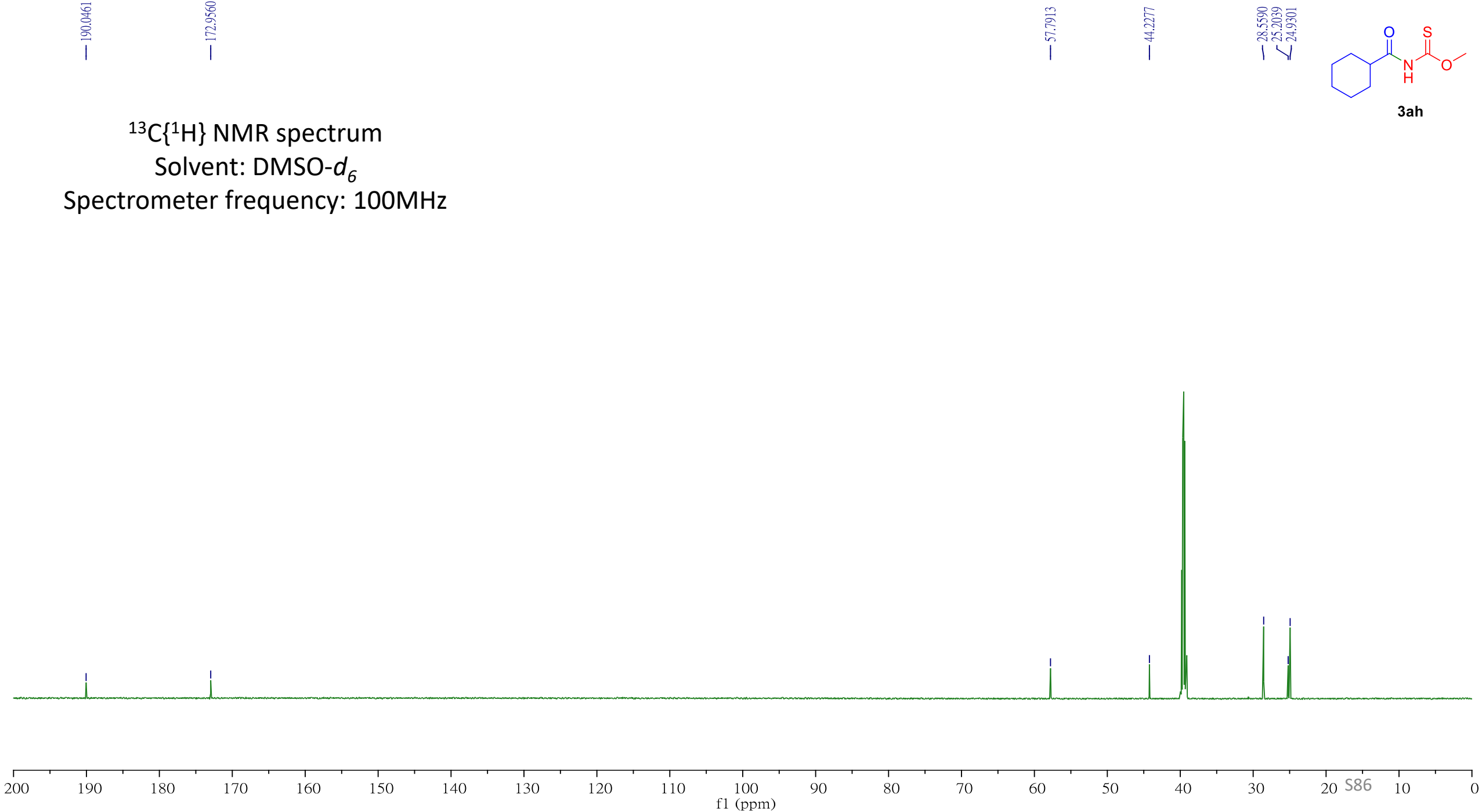
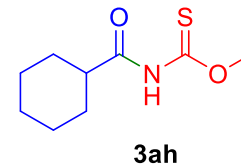
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



¹H NMR spectrum
Solvent: DMSO-*d*₆
Spectrometer frequency: 400 MHz



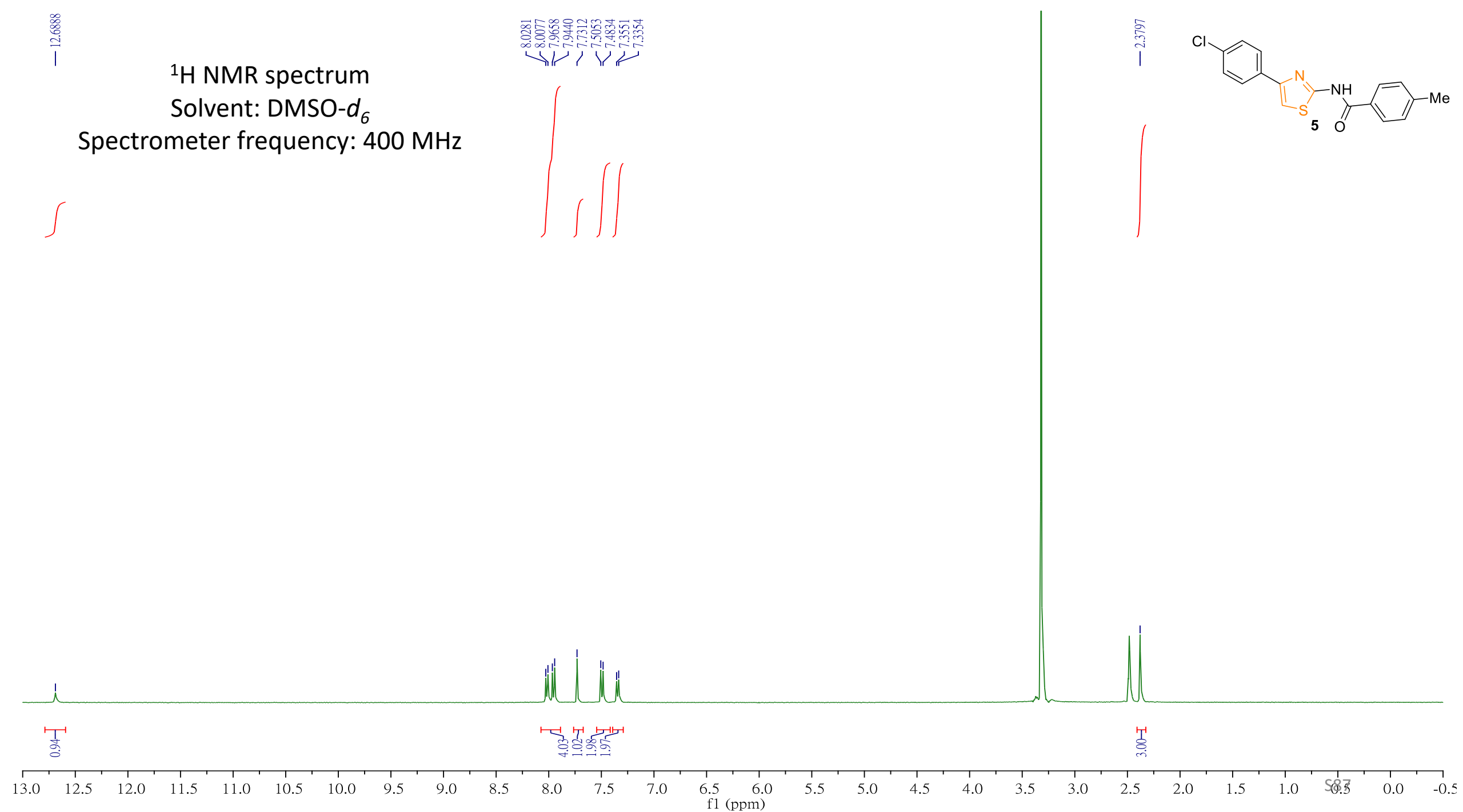
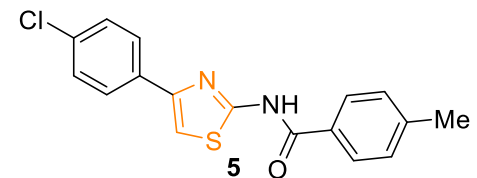
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



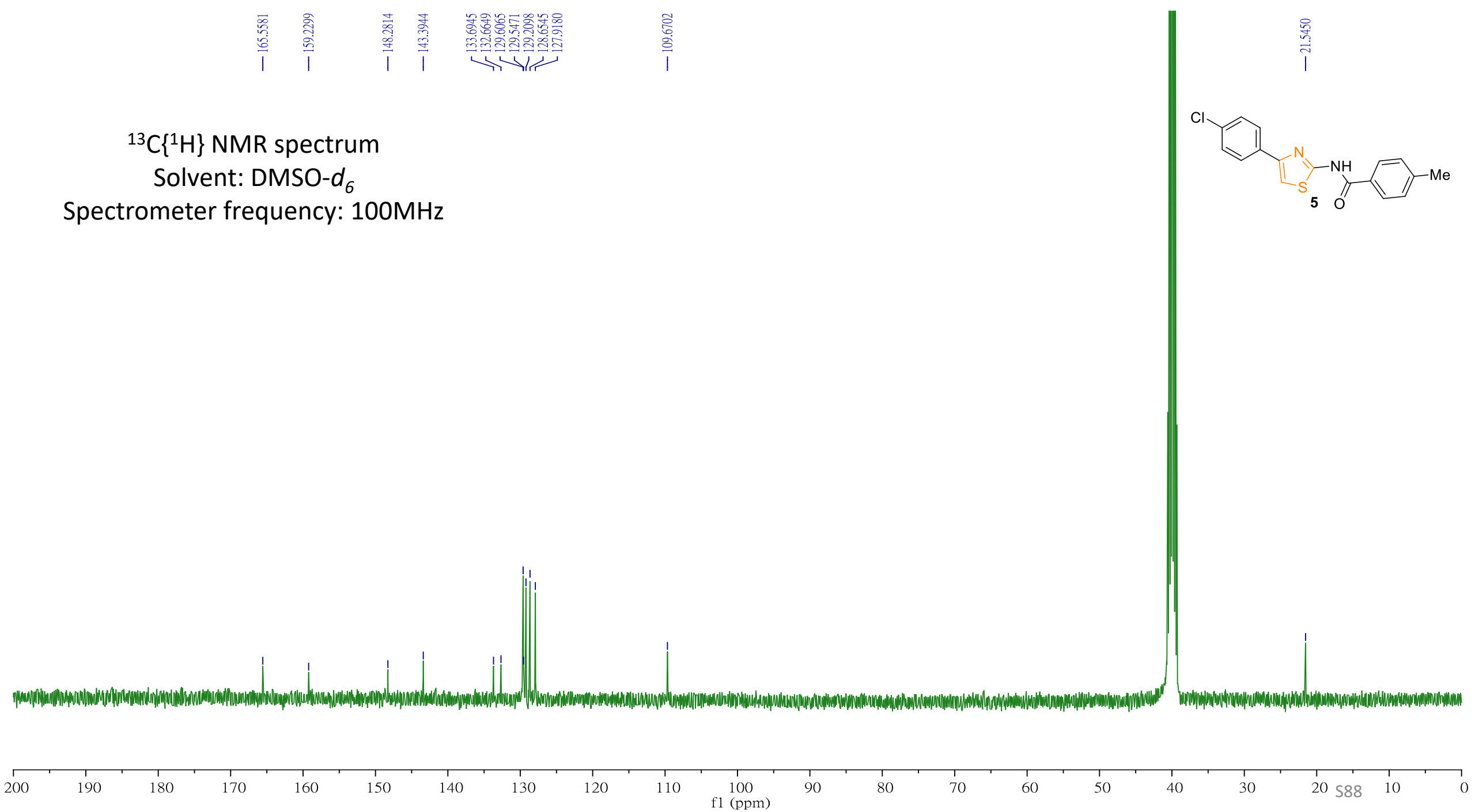
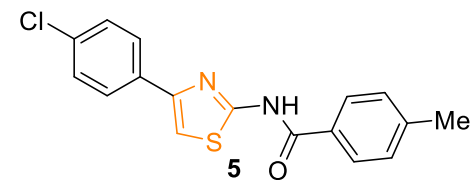
¹H NMR spectrum

Solvent: DMSO-*d*₆

Spectrometer frequency: 400 MHz



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: DMSO- d_6
Spectrometer frequency: 100MHz



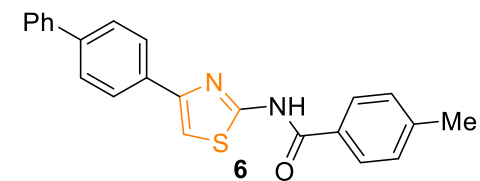
¹H NMR spectrum

Solvent: DMSO-*d*₆

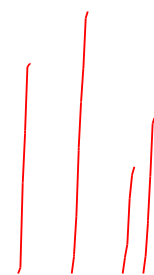
Spectrometer frequency: 400 MHz

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2.3864



0.87

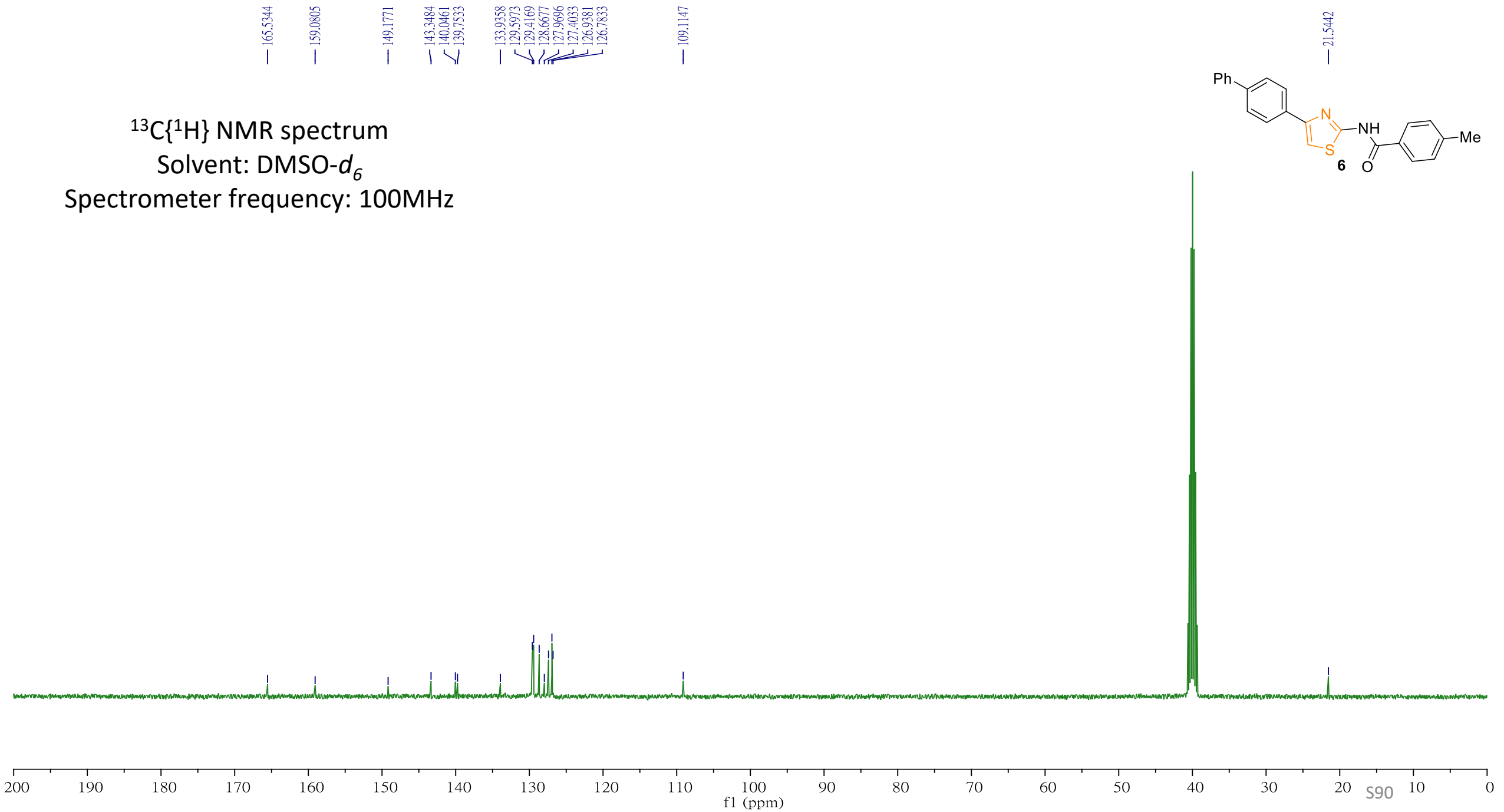


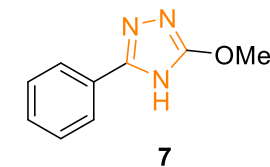
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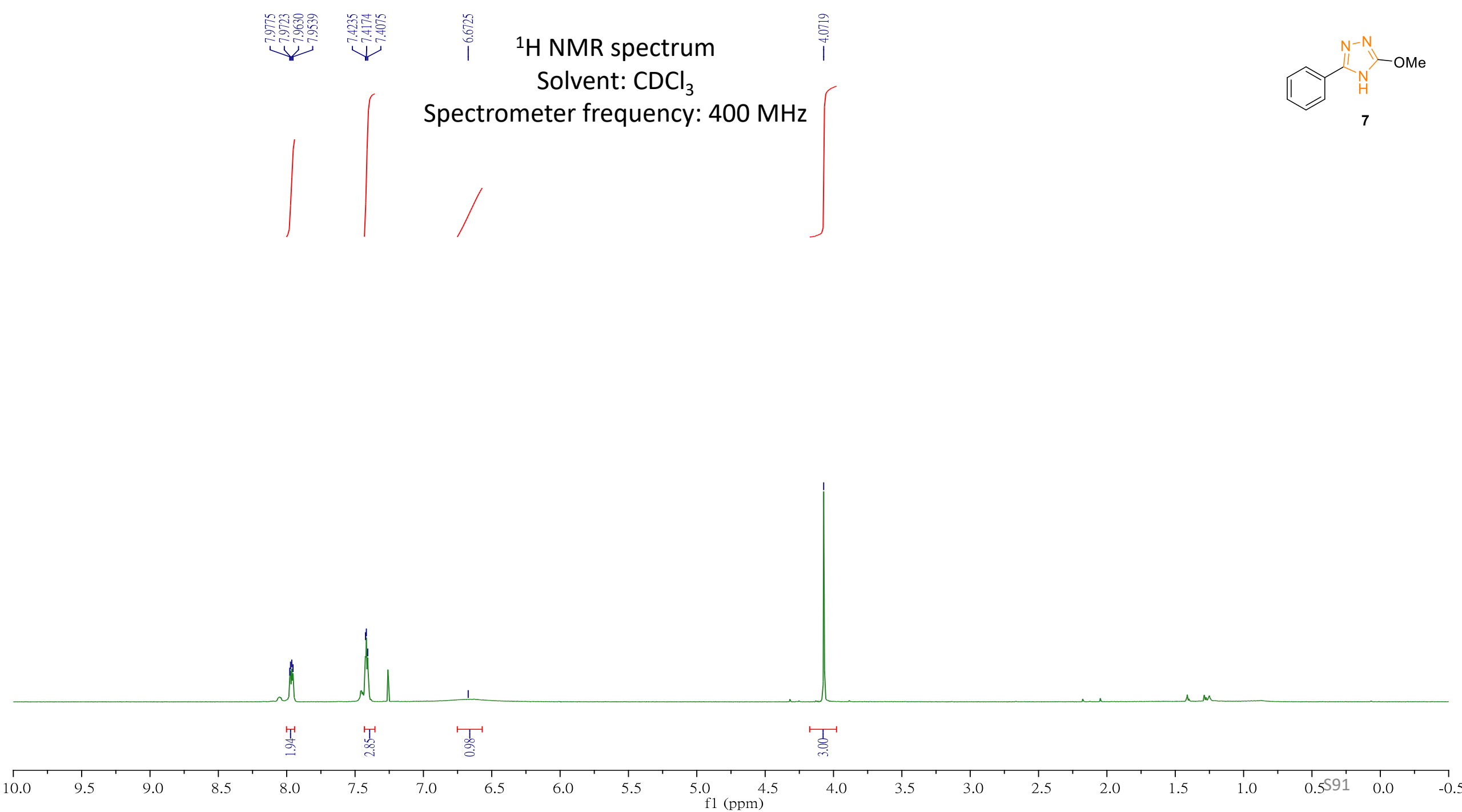
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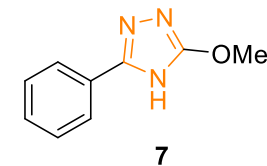
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: $\text{DMSO-}d_6$
Spectrometer frequency: 100MHz





¹H NMR spectrum
Solvent: CDCl₃
Spectrometer frequency: 400 MHz





$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum
Solvent: CDCl_3
Spectrometer frequency: 100MHz

