

Supporting Information

In Situ Generation of Acyloxyphosphoniums for Mild and Efficient Synthesis of Thioesters

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A. General Information

All solvents and reagents were commercially obtained and used as received, except when noted otherwise. NMR spectra were recorded at ambient temperature on a JEOL-400 MHz spectrometer in deuterated solvents such as CDCl₃ ($\delta = 7.24$ in ¹H NMR, $\delta = 77.0$ in ¹³C NMR), with ¹H and ¹³C chemical shifts reported in ppm (δ) relative to tetramethylsilane ($\delta = 0.00$). Mass spectra were acquired on a Bruker Daltonics BioTOF III spectrometer (ESI-MS). Flash column chromatography was conducted using Merck Kieselgel Si60 (40–63 μ m), while thin-layer chromatography (TLC) plates were visualized through exposure to ultraviolet light at 254 nm and/or immersion in a staining solution (phosphomolybdic acid, ninhydrin, anisaldehyde, or potassium permanganate) followed by heating on a hot plate. Concentration was achieved through rotary evaporation, and column chromatography was performed on silica gel (70–230 mesh ASTM).

B. Experimental section

Characterization of the synthetic iodobenzene dicarboxylates

Efficient preparation of the required iodobenzene dicarboxylates **1a** and **1c–1j** was accomplished with minor modifications to a known method.^{S1}

Compound 1a. ¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, $J = 7.6$ Hz, 2H), 8.20 (d, $J = 8.8$ Hz, 4H), 8.07 (d, $J = 8.8$ Hz, 4H), 7.68 (t, $J = 4.3$ Hz, 1H), 7.59 (t, $J = 7.7$ Hz, 2H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 169.2, 150.3, 135.4, 135.0, 132.4, 131.4, 131.2, 131.0, 129.7, 123.4, 123.3, 122.1. HRMS (APCI): calculated for [C₂₀H₁₃IN₂O₈+Na]⁺ 558.9609, found 558.9600.

Compound 1c. ¹H NMR (400 MHz, CDCl₃) δ 8.28 (d, $J = 8.0$ Hz, 3H), 8.03–7.86 (m, 5H), 7.74 (t, $J = 7.4$ Hz, 2H), 7.47 (t, $J = 7.6$ Hz, 2H), 7.21 (t, $J = 7.6$ Hz, 1H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 171.8, 141.3, 136.4, 134.7, 133.4, 133.0, 131.6, 131.5, 129.8, 128.6, 128.1. HRMS (APCI): calculated for [C₂₀H₁₃I₃O₄+Na]⁺ 720.7840, found 720.7844.

Compound 1d. ¹H NMR (400 MHz, CDCl₃) δ 8.22 (dd, $J = 8.5, 1.1$ Hz, 2H), 7.80 (dd, $J = 12.2, 8.2$ Hz, 4H), 7.61–7.59 (m, 1H), 7.53 (t, $J = 7.5$ Hz, 2H), 7.17–7.14 (m, 4H), 2.36 (s, 6H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 171.4, 143.1, 134.9, 131.6, 130.9, 130.1, 128.9, 127.3, 122.2, 21.6. HRMS (APCI): calculated for [C₂₂H₁₉IO₄+Na]⁺ 497.0222, found 497.0220.

Compound 1e. ¹H NMR (400 MHz, CDCl₃) δ 8.25–8.23 (m, 2H), 7.94–7.92 (m, 4H), 7.63–7.48 (m, 5H), 7.39–7.35 (m, 4H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 171.3, 134.8, 132.5, 131.7, 131.0, 130.1, 130.0, 128.2, 122.3. HRMS (APCI): calculated for [C₂₀H₁₅IO₄+Na]⁺ 469.9909, found 469.9901.

Compound 1f. ^1H NMR (400 MHz, CDCl_3) δ 8.08 (dd, $J = 8.3, 0.9$ Hz, 2H), 7.58 (d, $J = 7.6$ Hz, 1H), 7.49 (t, $J = 7.8$ Hz, 2H), 2.24 (t, $J = 7.4$ Hz, 4H), 1.57 (quin, $J = 7.4$, 4H), 0.88 (t, $J = 7.4$ Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 178.8, 134.8, 131.6, 130.8, 121.7, 35.9, 19.1, 13.7. HRMS (APCI): calculated for $[\text{C}_{14}\text{H}_{19}\text{IO}_4+\text{Na}]^+$ 401.0222, found 401.0219.

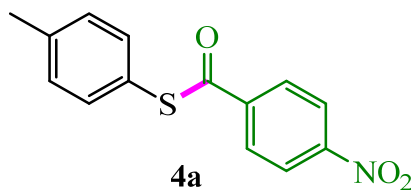
Compound 1g. ^1H NMR (400 MHz, CDCl_3) δ 7.94–7.92 (m, 2H), 7.55 (t, $J = 7.5$ Hz, 1H), 7.43–7.40 (m, 2H), 7.29–7.18 (m, 10H), 3.57 (s, 4H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 176.1, 134.5, 134.4, 131.4, 130.6, 129.0, 128.2, 126.6, 121.7, 40.8. HRMS (APCI): calculated for $[\text{C}_{22}\text{H}_{19}\text{IO}_4+\text{Na}]^+$ 497.0222, found 497.0224.

Compound 1h. ^1H NMR (400 MHz, CDCl_3) δ 8.01 (d, $J = 8.4$ Hz, 2H), 7.54 (d, $J = 7.5$ Hz, 1H), 7.47 (t, $J = 7.6$ Hz, 2H), 1.12 (s, 18H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 183.6, 134.2, 131.2, 130.6, 122.0, 38.9, 27.7. HRMS (APCI): calculated for $[\text{C}_{16}\text{H}_{23}\text{IO}_4+\text{Na}]^+$ 429.0535, found 429.0535.

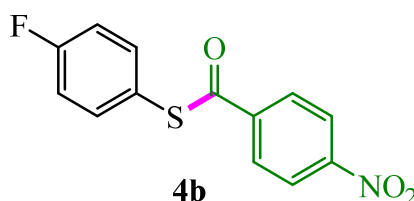
Compound 1i. ^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, $J = 8.1$ Hz, 2H), 7.70 (d, $J = 8.1$ Hz, 4H), 7.62 (d, $J = 7.3$ Hz, 1H), 7.53–7.40 (m, 8H), 7.27–7.23 (m, 6H), 7.10–7.08 (m, 4H), 6.64 (d, $J = 7.5$ Hz, 2H), 5.02 (dd, $J = 13.2, 6.0$ Hz, 2H), 3.17 (d, $J = 5.8$ Hz, 4H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 175.8, 166.8, 136.2, 134.9, 133.9, 132.1, 131.7, 131.1, 129.5, 129.3, 128.5, 128.5, 127.0, 121.6, 53.5, 38.4. HRMS (APCI): calculated for $[\text{C}_{38}\text{H}_{33}\text{IN}_2\text{O}_6+\text{Na}]^+$ 763.1274, found 763.1269.

Compound 1j. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (s, 15H), 5.26 (d, $J = 7.9$ Hz, 2H), 5.12 (s, 4H), 4.35 (s, 2H), 2.25–2.17 (m, 2H), 1.01 (d, $J = 6.6$ Hz, 6H), 0.93 (d, $J = 6.7$ Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 175.2, 156.4, 135.9, 128.2, 128.0, 127.9, 127.8, 127.8, 127.8, 127.6, 66.8, 58.7, 18.7, 17.1. HRMS (APCI): calculated for $[\text{C}_{32}\text{H}_{37}\text{IN}_2\text{O}_8+\text{Na}]^+$ 727.1485, found 727.1486.

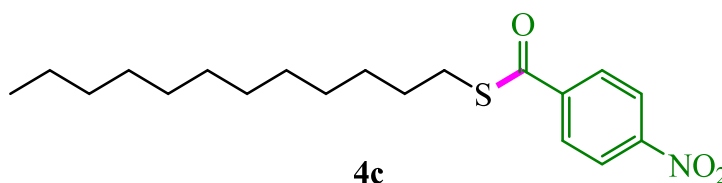
General procedure for the synthesis of thioesters. A sealed tube (5 mL) containing a mixture of **1a** (402 mg, 1.5 equiv) and Ph_3P (262 mg, 2.0 equiv) in toluene (2.5 mL) was heated at 80 °C for 1 hour. Then, **2a** (62.1 mg, 0.5 mmol) was added to the mixture, which was kept stirring at 80 °C for an additional 0.5 hour. Upon completion of the reaction, as determined by TLC analysis, the mixture was concentrated under reduced pressure and purified by column chromatography.



S-(4-Methylphenyl) 4-nitrobenzothioate (4a).^{S2} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a pale yellow solid (131 mg, 96%). $R_f = 0.7$ (hexanes/EtOAc = 3 : 1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.32 (d, $J = 8.5$ Hz, 2H), 8.16 (d, $J = 8.5$ Hz, 2H), 7.38 (d, $J = 8.1$ Hz, 2H), 7.28 (d, $J = 7.8$ Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 189.4, 150.7, 141.4, 140.6, 134.9, 130.5, 128.6, 124.1, 122.7, 21.5; HRMS (APCI): calculated for $\text{C}_{14}\text{H}_{11}\text{O}_3\text{NS}$: M^+ 273.0454, found 273.0459, found 273.0454.

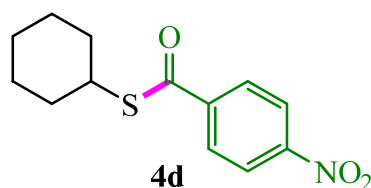


S-(4-Fluorophenyl) 4-nitrobenzothioate (4b). The reaction was carried out according on the general procedure. Starting from 4-fluorobenzenethiol (53 μL , 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a pale yellow solid (114 mg, 82%). $R_f = 0.6$ (hexanes/EtOAc = 3 : 1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.33 (d, $J = 8.8$ Hz, 2H), 8.15 (d, $J = 8.9$ Hz, 2H), 7.49–7.46 (m, 2H), 7.20–7.15 (m, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 188.9, 165.1, 162.6, 150.7, 141.0, 137.0, 128.5, 124.0, 117.0; HRMS (APCI): calculated for $\text{C}_{13}\text{H}_8\text{O}_3\text{NFS}$: M^+ 277.0203, found 277.0209.

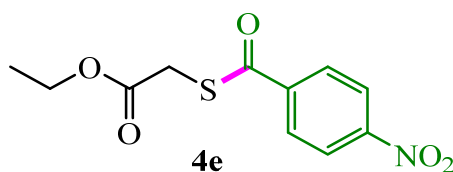


S-Dodecyl 4-nitrobenzothioate (4c).^{S3} The reaction was carried out according on the general procedure. Starting from 1-dodecanethiol (120 μL , 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a white solid (130 mg, 74%). $R_f = 0.6$ (hexanes/EtOAc = 12 : 1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.28 (d, $J = 8.8$ Hz, 2H), 8.10 (d, $J = 8.8$ Hz, 2H), 3.10 (t, $J = 7.4$ Hz, 2H), 1.71–1.63 (m, 2H), 1.43–1.37 (m, 2H), 1.28–1.24 (m, 17H), 0.86 (t, $J = 6.8$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 190.6, 150.4, 141.8, 128.2, 123.8, 31.9, 29.6, 29.5, 29.5, 29.3, 29.3, 29.1,

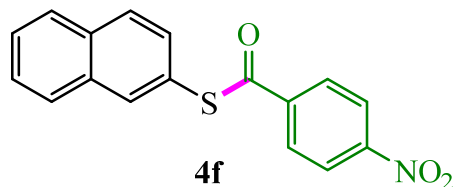
28.9, 22.7, 14.1; HRMS (APCI): calculated for C₁₉H₂₉O₃NS: M⁺ 351.1863, found 351.1859.



S-Cyclohexyl 4-nitrobenzothioate (4d).^{S4} The reaction was carried out according on the general procedure. Starting from cyclohexanethiol (61 μ L, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a pale yellow solid (133 mg, 100%). R_f = 0.7 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 8.26 (d, J = 9.1 Hz, 2H), 8.07 (d, J = 9.1 Hz, 2H), 3.78–3.75 (m, 1H), 2.02–2.00 (m, 2H), 1.76–1.73 (m, 2H), 1.63–1.60 (m, 1H), 1.55–1.44 (m, 4H), 1.33–1.30 (m, 1H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 190.6, 150.4, 141.8, 128.2, 123.8, 31.9, 29.6, 29.5, 29.3, 29.1, 28.9, 22.7, 14.1; HRMS (APCI): calculated for C₁₃H₁₆O₃NS: [M+H]⁺ 266.0845, found 266.0845.

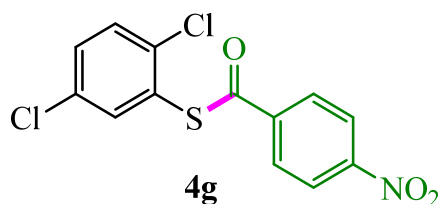


Ethyl 2-((4-nitrobenzoyl)thio)acetate (4e).^{S5} The reaction was carried out according on the general procedure. Starting from ethyl mercaptoacetate (548 μ L, 5 mmol) and compound **1a** (4.02 g, 1.5 equiv), the title compound was obtained as a white solid (1.21 g, 90%). R_f = 0.5 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 8.31 (d, J = 8.0 Hz, 2H), 8.12 (d, J = 8.0 Hz, 2H), 4.23 (q, J = 7.1 Hz, 2H), 3.91 (s, 2H), 1.29 (t, J = 7.1 Hz, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 188.8, 168.1, 150.7, 140.6, 128.4, 124.0, 62.2, 31.8, 14.1; HRMS (APCI): calculated for C₁₁H₁₂O₅NS: [M+H]⁺ 270.0431, found 270.0434.

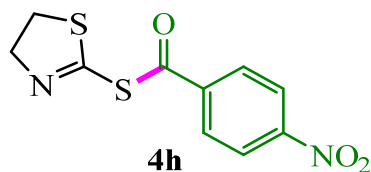


S-(2-Naphthyl) 4-nitrobenzothioate (4f).^{S6} The reaction was carried out according on the general procedure. Starting from 2-naphthalenethiol (80.1 mg, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a yellow solid (136 mg, 88%). R_f = 0.5 (hexanes/EtOAc = 6 : 1); ¹H NMR (400 MHz, CDCl₃) δ 8.35 (d, J = 8.7 Hz, 2H), 8.20 (d, J = 8.7 Hz, 2H), 8.05 (s, 1H), 7.94 (d, J = 8.5 Hz, 1H), 7.90–7.85 (m, 2H), 7.57–7.51 (m, 3H); ¹³C{H} NMR (100 MHz, CDCl₃)

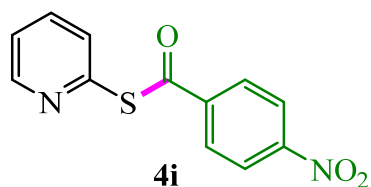
δ 189.1, 150.7, 141.3, 135.0, 133.6, 130.9, 129.2, 128.5, 128.0, 127.9, 127.5, 126.8, 124.0, 123.4;
HRMS (APCI): calculated for $C_{17}H_{12}O_3NS$: $[M+H]^+$ 310.0532, found 310.0535.



S-(2,5-Dichlorophenyl) 4-nitrobenzothioate (4g). The reaction was carried out according on the general procedure. Starting from 2,5-dichlorobenzenethiol (89.5 mg, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a white solid (126 mg, 77%). R_f = 0.8 (hexanes/EtOAc = 3 : 1); 1H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, J = 9.1 Hz, 2H), 8.17 (d, J = 8.9 Hz, 2H), 7.60 (d, J = 2.5 Hz, 1H), 7.51 (d, J = 8.7 Hz, 1H), 7.41 (dd, J = 8.5, 2.5 Hz, 1H); $^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$) δ 186.3, 150.8, 140.6, 137.3, 136.8, 133.0, 131.8, 131.3, 128.7, 127.4, 124.1; HRMS (APCI): calculated for $C_{13}H_8O_3NCl_2S$: $[M+H]^+$ 327.95965, found 327.95967.

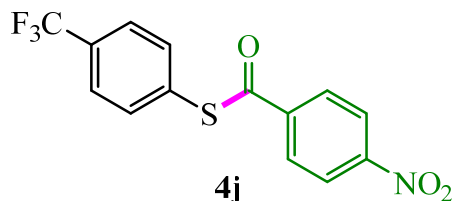


S-Thiazolinyl 4-nitrobenzothioate (4h). The reaction was carried out according on the general procedure. Starting from 2-thiazoline-2-thiol (59.6 mg, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a yellow solid (118 mg, 89%). R_f = 0.4 (hexanes/EtOAc = 1 : 1); 1H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 8.8 Hz, 2H), 7.77 (d, J = 8.9 Hz, 2H), 4.58 (t, J = 7.3 Hz, 2H), 3.51 (t, J = 7.2 Hz, 2H); $^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$) δ 202.1, 169.2, 149.5, 139.7, 129.8, 123.5, 56.0, 29.8; HRMS (APCI): calculated for $C_{10}H_9O_3N_2S_2$: $[M+H]^+$ 269.0049, found 269.0046.

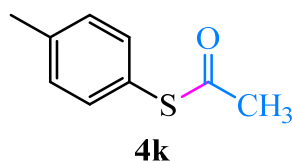


S-Pyridinyl 4-nitrobenzothioate (4i).^{S7} The reaction was carried out according on the general procedure. Starting from 2-mercaptopyridine (55.6 mg, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a yellow oil (89.8 mg, 69%). R_f = 0.5 (hexanes/EtOAc =

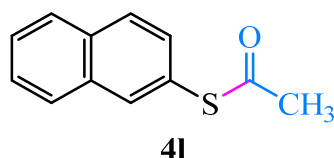
1 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.70 (dd, $J = 4.8, 1.1$ Hz, 1H), 8.35 (d, $J = 8.9$ Hz, 2H), 8.17 (d, $J = 8.9$ Hz, 2H), 7.83 (td, $J = 7.7, 1.9$ Hz, 1H), 7.74–7.72 (m, 1H), 7.39 (ddd, $J = 7.6, 4.9, 1.2$ Hz, 1H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 188.2, 150.9, 150.1, 141.2, 137.7, 130.9, 128.7, 124.3, 124.2, 124.1; HRMS (APCI): calculated for $\text{C}_{12}\text{H}_8\text{N}_2\text{O}_3\text{S}$: $[\text{M}+\text{Na}]^+$ 283.0148, found 283.0141.



S-(4-(Trifluoromethyl)phenyl) 4-nitrobenzothioate (4j). The reaction was carried out according on the general procedure. Starting from 4-(trifluoromethyl)thiophenol (89.1 mg, 0.5 mmol) and compound **1a** (402 mg, 1.5 equiv), the title compound was obtained as a white solid (124 mg, 76%). $R_f = 0.8$ (hexanes/EtOAc = 3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.36 (d, $J = 9.0$ Hz, 2H), 8.18 (d, $J = 8.9$ Hz, 2H), 7.75 (d, $J = 8.3$ Hz, 2H), 7.66 (d, $J = 8.1$ Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 187.7, 150.9, 140.8, 135.1, 132.1 (q, $J_{\text{C-F}} = 29.8$ Hz), 130.9, 128.6, 126.3, 124.1, 123.7 (q, $J_{\text{C-F}} = 268.4$ Hz); HRMS (EI): calculated for $\text{C}_{14}\text{H}_8\text{F}_3\text{NO}_3\text{S}$ (M^+), 327.0177, found 327.0180.

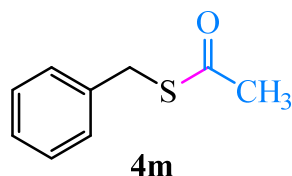


S-(p-Tolyl) ethanethioate (4k).^{S8} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1b** (242 mg, 1.5 equiv), the title compound was obtained as a yellow liquid (53.2 mg, 64%). $R_f = 0.6$ (hexanes/EtOAc = 3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.30–7.21 (m, 4H), 2.41 (s, 3H), 2.37 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 194.7, 139.7, 134.4, 130.0, 124.4, 30.1, 21.3; HRMS (EI): calculated for $\text{C}_9\text{H}_{10}\text{OS}$ (M^+), 166.0452, found 166.0453.

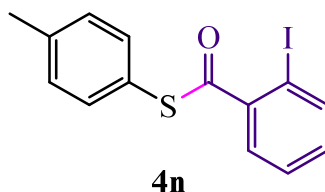


S-(Naphthalen-2-yl) ethanethioate (4l).^{S9} The reaction was carried out according on the general procedure. Starting from 2-naphthalenethiol (80.1 mg, 0.5 mmol) and compound **1b** (242 mg, 1.5 equiv), the title compound was obtained as a brown solid (64.7 mg, 64%). $R_f = 0.6$ (hexanes/EtOAc

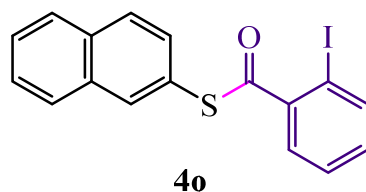
= 3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.95–7.82 (m, 4H), 7.53–7.44 (m, 3H), 2.46 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 194.3, 134.3, 133.5, 133.3, 130.8, 128.8, 127.9, 127.7, 127.1, 126.5, 125.2, 30.2; HRMS (EI): calculated for $\text{C}_{12}\text{H}_{10}\text{OS}$ (M^+), 202.0452, found 202.0455.



S-Benzyl ethanethioate (4m).^{S10} The reaction was carried out according on the general procedure. Starting from benzyl mercaptan (587 μL , 5 mmol) and compound **1b** (2.42 g, 1.5 equiv), the title compound was obtained as a yellow liquid (657 mg, 79%). R_f = 0.4 (hexanes/EtOAc = 15 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.30–7.26 (m, 5H), 4.12 (s, 2H), 2.35 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 195.0, 137.5, 128.7, 128.5, 127.2, 33.4, 30.2; HRMS (EI): calculated for $\text{C}_9\text{H}_{10}\text{OS}$ (M^+), 166.0452, found 166.0444.

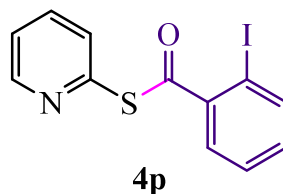


S-(p-Tolyl) 2-iodobenzothioate (4n).^{S11} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1c** (524 mg, 1.5 equiv), the title compound was obtained as a white solid (122 mg, 69%). a white solid (122 mg, 69%). R_f = 0.7 (hexanes/EtOAc = 3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, J = 8.0 Hz, 1H), 7.70 (dd, J = 7.6, 1.4 Hz, 1H), 7.46–7.42 (m, 3H), 7.29–7.26 (m, 2H), 7.19 (dd, J = 7.8, 1.5 Hz, 1H), 2.40 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 192.8, 142.4, 140.7, 140.0, 134.5, 132.3, 130.2, 128.5, 127.9, 123.8, 91.5, 21.4; HRMS (EI): calculated for $\text{C}_{14}\text{H}_{11}\text{IOS}$ (M^+), 353.9575, found 353.9575.

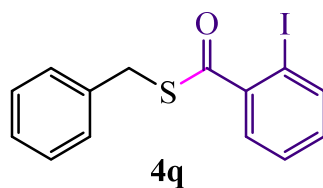


S-(Naphthalen-2-yl) 2-iodobenzothioate (4o).^{S12} The reaction was carried out according on the general procedure. Starting from 2-naphthalenethiol (801 mg, 5 mmol) and compound **1c** (5.24 g, 1.5 equiv), the title compound was obtained as a brown solid (1.50 g, 77%). R_f = 0.6 (hexanes/EtOAc =

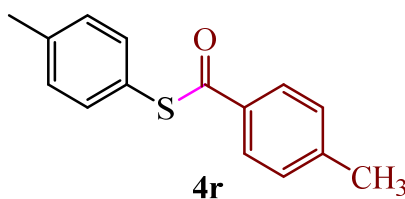
3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.08 (s, 1H), 7.99–7.87 (m, 4H), 7.77–7.75 (m, 1H), 7.61–7.47 (m, 4H), 7.20 (s, 1H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 192.4, 142.2, 140.8, 134.5, 133.5, 133.4, 132.4, 130.7, 128.9, 128.6, 128.0, 128.0, 127.8, 127.2, 126.6, 124.7, 91.6; HRMS (EI): calculated for $\text{C}_{17}\text{H}_{11}\text{IOS}$ (M^+), 389.9575, found 389.9585.



S-(Pyridin-2-yl) 2-iodobenzothioate (4p). The reaction was carried out according on the general procedure. Starting from 2-mercaptopyridine (55.6 mg, 0.5 mmol) and compound **1c** (524 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (113 mg, 66%). $R_f = 0.6$ (hexanes/EtOAc = 1 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.64 (d, $J = 4.6$ Hz, 1H), 7.94 (d, $J = 7.8$ Hz, 1H), 7.78–7.72 (m, 3H), 7.42 (t, $J = 7.5$ Hz, 1H), 7.31 (dd, $J = 8.6, 4.7$ Hz, 1H), 7.19–7.15 (m, 1H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 191.1, 151.2, 150.4, 141.8, 140.9, 137.3, 132.7, 130.2, 128.8, 128.0, 123.8, 91.5; HRMS (MALDI-TOF): calculated for $\text{C}_{12}\text{H}_9\text{INOS}$ ($\text{M}+\text{H}^+$), 341.9444, found 341.9451.

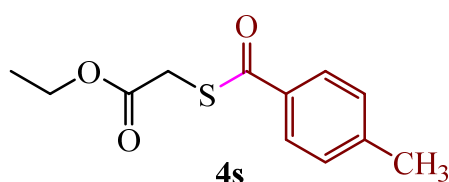


S-Benzyl 2-iodobenzothioate (4q).^{S13} The reaction was carried out according on the general procedure. Starting from benzyl mercaptan (59 μL , 0.5 mmol) and compound **1c** (524 mg, 1.5 equiv), the title compound was obtained as a yellow liquid (103 mg, 58%). $R_f = 0.5$ (hexanes/EtOAc = 15 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 7.7$ Hz, 1H), 7.59 (d, $J = 7.4$ Hz, 1H), 7.40–7.26 (m, 6H), 7.15 (t, $J = 7.4$ Hz, 1H), 4.34 (s, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 193.4, 142.3, 140.8, 137.0, 132.4, 129.0, 128.8, 128.7, 127.9, 127.4, 91.3, 34.4; HRMS (EI): calculated for $\text{C}_{14}\text{H}_{11}\text{IOS}$ (M^+), 353.9575, found 353.9567.

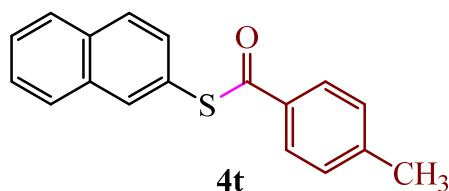


S-(p-Tolyl) 4-methylbenzothioate (4r).^{S14} The reaction was carried out according on the general

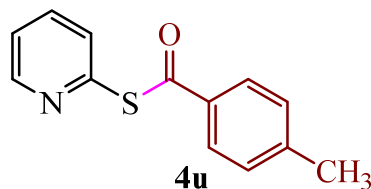
procedure. Starting from *p*-thiocresol (621 mg, 5 mmol) and compound **1d** (3.56 g, 1.5 equiv), the title compound was obtained as a white solid (957 mg, 79%). $R_f = 0.5$ (hexanes/EtOAc = 15 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.2$ Hz, 2H), 7.42 (d, $J = 8.1$ Hz, 2H), 7.28–7.26 (m, 4H), 2.42 (s, 3H), 2.41 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 189.8, 144.3, 139.5, 134.9, 134.0, 129.9, 129.2, 127.4, 123.8, 21.5, 21.2; HRMS (EI): calculated for $\text{C}_{15}\text{H}_{14}\text{OS}$ (M^+), 242.0765, found 242.0770.



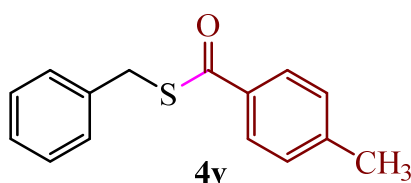
Ethyl 2-((4-methylbenzoyl)thio)acetate (4s). The reaction was carried out according on the general procedure. Starting from ethyl mercaptoacetate (55 μL , 0.5 mmol) and compound **1d** (356 mg, 1.5 equiv), the title compound was obtained as a white solid (91.7 mg, 77%). $R_f = 0.2$ (hexanes/EtOAc = 15 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, $J = 8.2$ Hz, 2H), 7.25 (d, $J = 8.1$ Hz, 2H), 4.22 (d, $J = 7.1$ Hz, 2H), 3.86 (s, 2H), 2.40 (s, 3H), 1.29 (t, $J = 7.1$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 189.6, 168.9, 144.7, 133.6, 129.3, 127.4, 61.8, 31.3, 21.6, 14.1; HRMS (EI): calculated for $\text{C}_{12}\text{H}_{14}\text{O}_3\text{S}$ (M^+), 238.0664, found 238.0659.



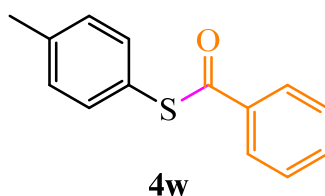
S-(Naphthalen-2-yl) 4-methylbenzothioate (4t).^{S21} The reaction was carried out according on the general procedure. Starting from 2-naphthalenethiol (80.1 mg, 0.5 mmol) and compound **1d** (356 mg, 1.5 equiv), the title compound was obtained as a white solid (107 mg, 77%). $R_f = 0.5$ (hexanes/EtOAc = 15 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.05 (s, 1H), 7.97–7.85 (m, 5H), 7.57–7.52 (m, 3H), 7.31 (d, $J = 8.0$ Hz, 2H), 2.45 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 189.9, 144.6, 135.0, 134.1, 133.6, 133.4, 131.5, 129.4, 128.7, 128.0, 127.8, 127.6, 127.1, 126.5, 124.9, 21.7; HRMS (EI): calculated for $\text{C}_{18}\text{H}_{14}\text{OS}$ (M^+), 278.0765, found 278.0770.



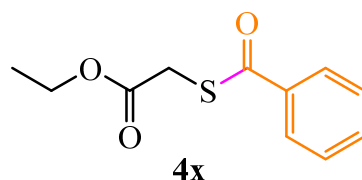
S-(Pyridin-2-yl) 4-methylbenzothioate (4u).^{S15} The reaction was carried out according on the general procedure. Starting from 2-mercaptopyridine (55.6 mg, 0.5 mmol) and compound **1d** (356 mg, 1.5 equiv), the title compound was obtained as a white solid (110 mg, 96%). $R_f = 0.3$ (hexanes/EtOAc = 3 : 1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.68 (dq, $J = 4.8, 0.9$ Hz, 1H), 7.92 (dd, $J = 8.2$ Hz, 2H), 7.81–7.71 (m, 2H), 7.35–7.27 (m, 3H), 2.43 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 188.8, 151.4, 150.4, 144.9, 137.1, 133.9, 130.9, 129.5, 127.6, 123.5, 77.3, 77.0, 76.7, 21.7; HRMS (EI): calculated for $\text{C}_{13}\text{H}_{11}\text{NOS}$ (M^+), 229.0561, found 229.0567.



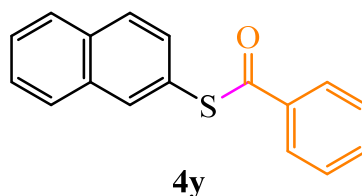
S-Benzyl 4-methylbenzothioate (4v).^{S16} The reaction was carried out according on the general procedure. Starting from benzyl mercaptan (59 μL , 0.5 mmol) and compound **1d** (356 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (89.7 mg, 74%). $R_f = 0.5$ (hexanes/EtOAc = 15 : 1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.87 (d, $J = 8.2$ Hz, 2H), 7.38–7.25 (m, 7H), 4.31 (s, 2H), 2.40 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 190.8, 144.3, 137.6, 134.2, 129.2, 128.9, 128.6, 127.3, 127.2, 76.7, 33.2, 21.6; HRMS (EI): calculated for $\text{C}_{15}\text{H}_{14}\text{OS}$ (M^+), 242.0765, found 242.0757.



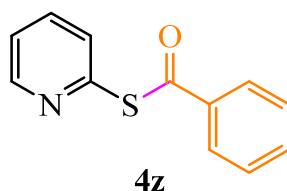
S-(*p*-Tolyl) benzothioate (4w).^{S17} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1e** (335 mg, 1.5 equiv), the title compound was obtained as a white solid (106 mg, 93%). $R_f = 0.6$ (hexanes/EtOAc = 5 : 1); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.04–8.02 (m, 2H), 7.62–7.58 (m, 1H), 7.50–7.46 (m, 2H), 7.40 (d, $J = 8.1$ Hz, 2H), 7.29–7.23 (m, 2H), 2.40 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 190.5, 139.8, 136.6, 135.0, 133.5, 130.1, 128.7, 127.4, 123.7, 21.3; HRMS (EI): calculated for $\text{C}_{14}\text{H}_{12}\text{OS}$ (M^+), 228.0609, found 228.0614.



Ethyl 2-(benzoylthio)acetate (4x).^{S18} The reaction was carried out according on the general procedure. Starting from ethyl mercaptoacetate (55 μ L, 0.5 mmol) and compound **1e** (335 mg, 1.5 equiv), the title compound was obtained as a yellow liquid (108 mg, 96%). R_f = 0.5 (hexanes/EtOAc = 3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 7.97–7.95 (m, 2H), 7.57 (d, J = 7.4 Hz, 1H), 7.47–7.43 (m, 2H), 4.22 (q, J = 7.1 Hz, 2H), 3.87 (s, 2H), 1.28 (t, J = 7.1 Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 190.0, 168.7, 136.1, 133.7, 128.6, 127.3, 61.8, 31.4, 14.0; HRMS (EI): calculated for $\text{C}_{11}\text{H}_{12}\text{O}_3\text{S}$ (M^+), 224.0507, found 224.0509.

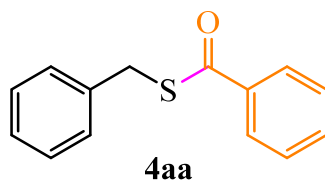


S-(Naphthalen-2-yl) benzothioate (4y).^{S19} The reaction was carried out according on the general procedure. Starting from 2-naphthalenethiol (80.1 mg, 0.5 mmol) and compound **1e** (335 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (185 mg, 70%). R_f = 0.5 (hexanes/EtOAc = 6 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.08 (d, J = 7.3 Hz, 3H), 7.94–7.85 (m, 3H), 7.62 (d, J = 7.4 Hz, 1H), 7.58–7.49 (m, 5H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 190.4, 136.6, 135.0, 133.7, 133.6, 133.4, 131.4, 128.8, 128.8, 128.0, 127.8, 127.5, 127.2, 126.5, 124.6; HRMS (EI): calculated for $\text{C}_{17}\text{H}_{12}\text{OS}$ (M^+), 264.0609, found 264.0602.

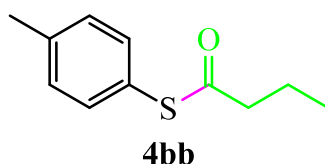


S-(Pyridin-2-yl) benzothioate (4z).^{S20} The reaction was carried out according on the general procedure. Starting from 2-mercaptopyridine (556 mg, 5 mmol) and compound **1e** (3.35 g, 1.5 equiv), the title compound was obtained as a yellow solid (689 mg, 64%). R_f = 0.3 (hexanes/EtOAc = 3 : 1); ^1H NMR (400 MHz, CDCl_3) δ 8.69–8.68 (m, 1H), 8.04–8.02 (m, 2H), 7.82–7.73 (m, 2H), 7.65–7.61 (m, 1H), 7.52–7.48 (m, 2H), 7.35–7.33 (m, 1H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 189.3, 151.3,

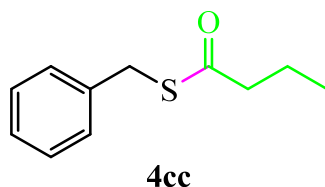
150.5, 137.2, 136.5, 133.9, 130.9, 128.8, 127.6, 123.6; HRMS (EI): calculated for C₁₂H₉NOS (M⁺), 215.0405, found 215.0409.



S-Benzyl benzothioate (4aa).^{S21} The reaction was carried out according on the general procedure. Starting from benzyl mercaptan (59 μ L, 0.5 mmol) and compound **1e** (335 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (83.3 mg, 73%). $R_f = 0.5$ (hexanes/EtOAc = 5 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.99–7.97 (m, 2H), 7.59–7.55 (m, 1H), 7.46–7.38 (m, 4H), 7.34–7.32 (t, $J = 7.6$ Hz, 2H), 7.28–7.25 (m, 1H), 4.33 (s, 2H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 191.3, 137.4, 136.7, 133.4, 128.9, 128.6, 128.6, 127.3, 127.3, 33.3; HRMS (EI): calculated for C₁₄H₁₂OS (M⁺), 228.0609, found 228.0601.

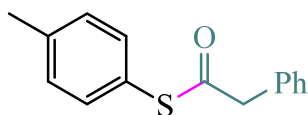


S-(*p*-Tolyl) butanethioate (4bb).^{S22} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1f** (284 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (68.0 mg, 70%). $R_f = 0.8$ (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.30–7.20 (m, 4H), 2.62 (t, $J = 7.3$ Hz, 2H), 2.37 (s, 3H), 1.74 (sextet, $J = 7.3$ Hz, 2H), 0.99 (t, $J = 7.4$ Hz, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 198.0, 139.5, 134.4, 130.0, 124.4, 45.4, 21.3, 19.1, 13.5; HRMS (EI): calculated for C₁₁H₁₄OS (M⁺), 194.0765, found 194.0776.



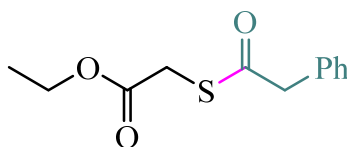
S-Benzyl butanethioate (4cc).^{S23} The reaction was carried out according on the general procedure. Starting from benzyl mercaptan (59 μ L, 0.5 mmol) and compound **1f** (284 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (66.1 mg, 68%). $R_f = 0.7$ (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.30–7.26 (m, 5H), 4.12 (s, 2H), 2.55 (t, $J = 7.4$ Hz, 2H), 1.71 (d, $J = 7.4$ Hz, 2H), 0.95 (t, $J = 7.4$ Hz, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 198.9, 137.7, 128.8, 128.6,

127.2, 45.6, 33.0, 19.1, 13.5; HRMS (EI): calculated for C₁₁H₁₄OS (M⁺), 194.0765, found 194.0772.



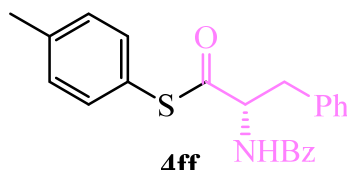
4dd

S-(*p*-Tolyl) 2-phenylethanethioate (4dd).^{S24} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1g** (356 mg, 1.5 equiv), the title compound was obtained as a white solid (90.9 mg, 75%). *R_f* = 0.7 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.35–7.25 (m, 7H), 7.19 (d, *J* = 7.8 Hz, 2H), 3.90 (s, 2H), 2.35 (s, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 176.1, 134.5, 134.4, 131.4, 130.6, 129.1, 129.0, 128.2, 126.6, 121.7, 40.8; HRMS (EI): calculated for C₁₅H₁₄OS (M⁺), 242.0765, found 242.0758.



4ee

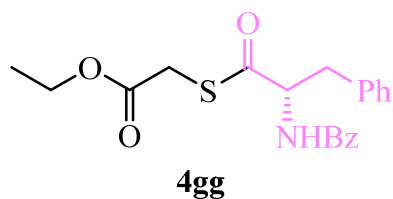
Ethyl 2-((2-phenylacetyl)thio)acetate (4ee).^{S25} The reaction was carried out according on the general procedure. Starting from ethyl mercaptoacetate (55 μL, 0.5 mmol) and compound **1g** (356 mg, 1.5 equiv), the title compound was obtained as a colorless liquid (82.2 mg, 69%). *R_f* = 0.4 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.30–7.24 (m, 5H), 4.13 (q, *J* = 7.0 Hz, 2H), 3.84 (s, 2H), 3.63 (s, 2H), 1.21 (t, *J* = 7.2 Hz, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 195.6, 168.5, 132.9, 129.6, 128.7, 127.6, 61.8, 49.9, 31.6, 14.0; HRMS (EI): calculated for C₁₂H₁₄O₃S (M⁺), 238.0664, found 238.0667.



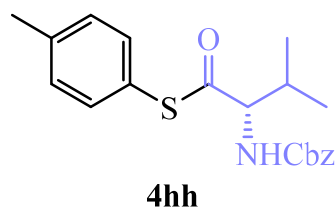
4ff

S-(*p*-Tolyl) (S)-2-benzamido-3-phenylpropanethioate (4ff).^{S26} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1i** (555 mg, 1.5 equiv), the title compound was obtained as a white solid (122 mg, 65%). *R_f* = 0.4 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 7.3 Hz, 2H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.36–7.22 (m, 9H), 6.76 (t, *J* = 6.4 Hz, 1H), 5.37–5.33 (m, 1H), 3.31 (d, *J* = 6.3 Hz, 2H), 2.39 (s, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 198.7, 167.0, 139.9, 135.4, 134.4,

133.6, 131.8, 130.1, 129.4, 128.7, 128.6, 127.2, 127.0, 123.1, 59.7, 38.2, 21.3; HRMS (EI): calculated for C₂₃H₂₁NO₂S (M⁺), 375.1293, found 375.1290.



Ethyl 2-((benzoyl-L-phenylalanyl)thio)acetate (4gg). The reaction was carried out according on the general procedure. Starting from ethyl mercaptoacetate (55 μ L, 0.5 mmol) and compound **1i** (555 mg, 1.5 equiv), the title compound was obtained as a white solid (130 mg, 70%). R_f = 0.1 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, J = 7.8 Hz, 2H), 7.53–7.47 (m, 1H), 7.41 (t, J = 7.3 Hz, 2H), 7.33–7.26 (m, 3H), 7.21 (d, J = 7.2 Hz, 2H), 6.50 (d, J = 7.9 Hz, 1H), 5.30–5.23 (m, 1H), 4.20 (dd, J = 13.7, 6.6 Hz, 2H), 3.75 (d, J = 15.0 Hz, 1H), 3.66 (d, J = 16.0 Hz, 1H), 3.30–3.27 (m, 2H), 1.29 (t, J = 7.1 Hz, 3H); ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 198.7, 168.4, 167.1, 135.2, 133.5, 132.0, 129.4, 128.8, 128.7, 127.4, 127.0, 61.9, 59.7, 38.0, 31.4, 14.1; HRMS (EI): calculated for C₂₀H₂₁NO₄S (M⁺), 371.1191, found 371.1187.



S-(*p*-Tolyl) (S)-2-(((benzyloxy)carbonyl)amino)-3-methylbutanethioate (4hh).^{S27} The reaction was carried out according on the general procedure. Starting from *p*-thiocresol (62.1 mg, 0.5 mmol) and compound **1j** (528 mg, 1.5 equiv), the title compound was obtained as a white solid (122 mg, 68%). R_f = 0.6 (hexanes/EtOAc = 3 : 1); ¹H NMR (400 MHz, CDCl₃) δ 7.42–7.32 (m, 7H), 7.25 (d, J = 7.8 Hz, 2H), 5.71 (d, J = 9.2 Hz, 1H), 5.23 (s, 2H), 4.57 (m, 1H), 2.41 (s, 3H), 2.41–2.39 (m, 1H), 1.09 (d, J = 6.6 Hz, 3H), 0.98 (d, J = 6.6 Hz, 3H); ¹³C {¹H} NMR (100 MHz, CDCl₃) δ 198.9, 156.1, 139.5, 136.0, 134.3, 129.8, 128.3, 128.0, 127.8, 123.4, 67.0, 65.6, 30.9, 21.0, 19.1, 16.7; HRMS (EI): calculated for C₂₀H₂₃NO₃S (M⁺), 357.1399, found 357.1403.

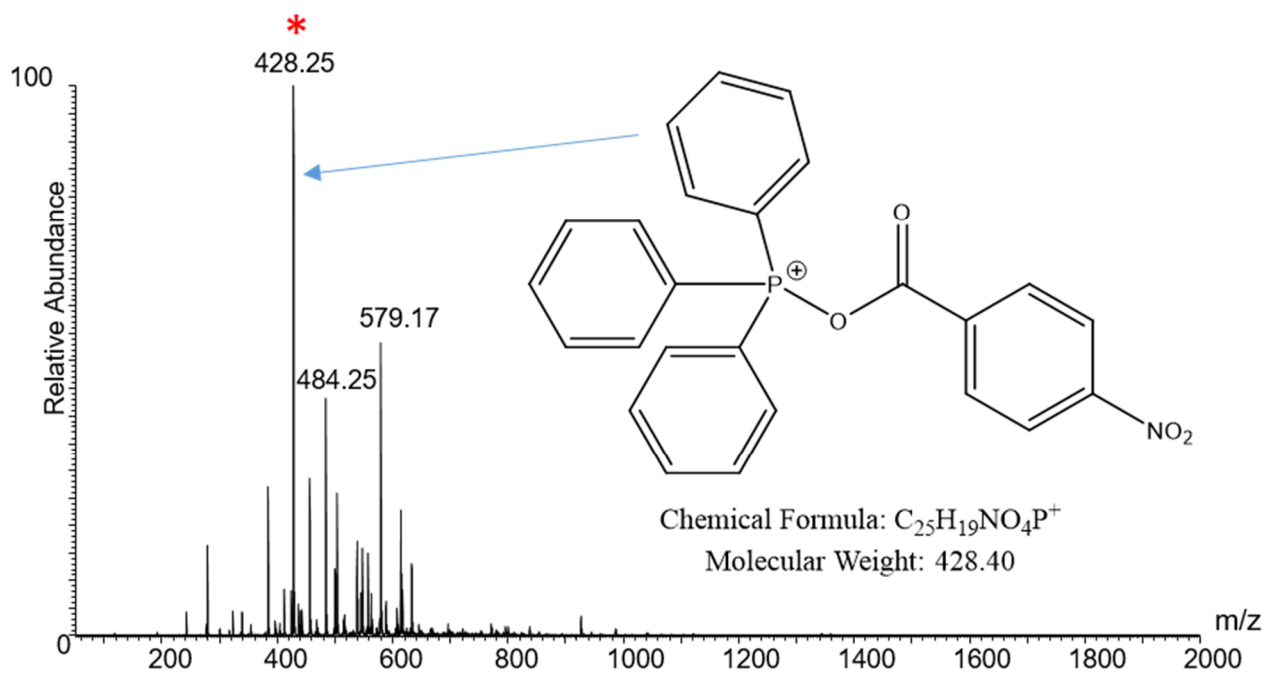
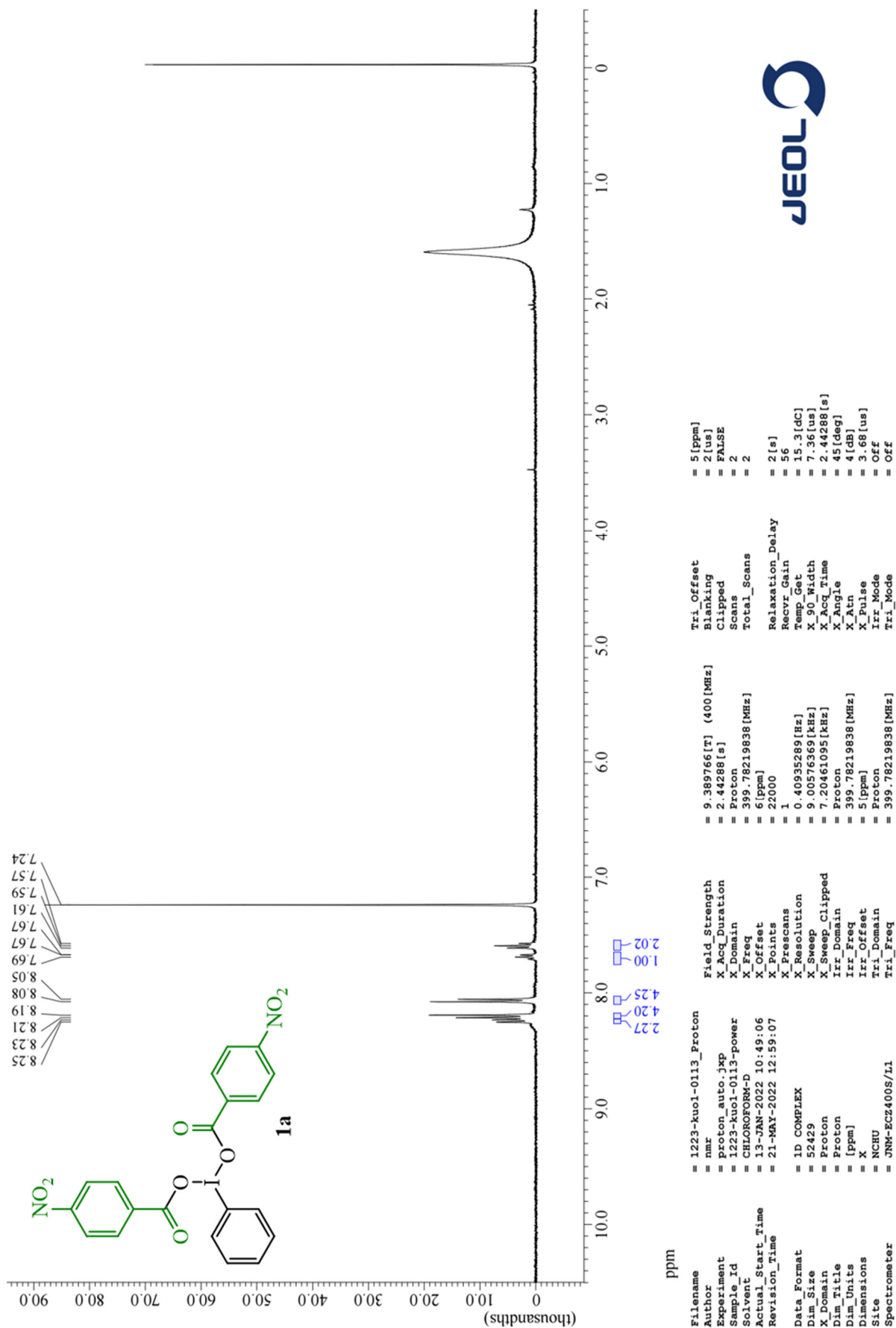
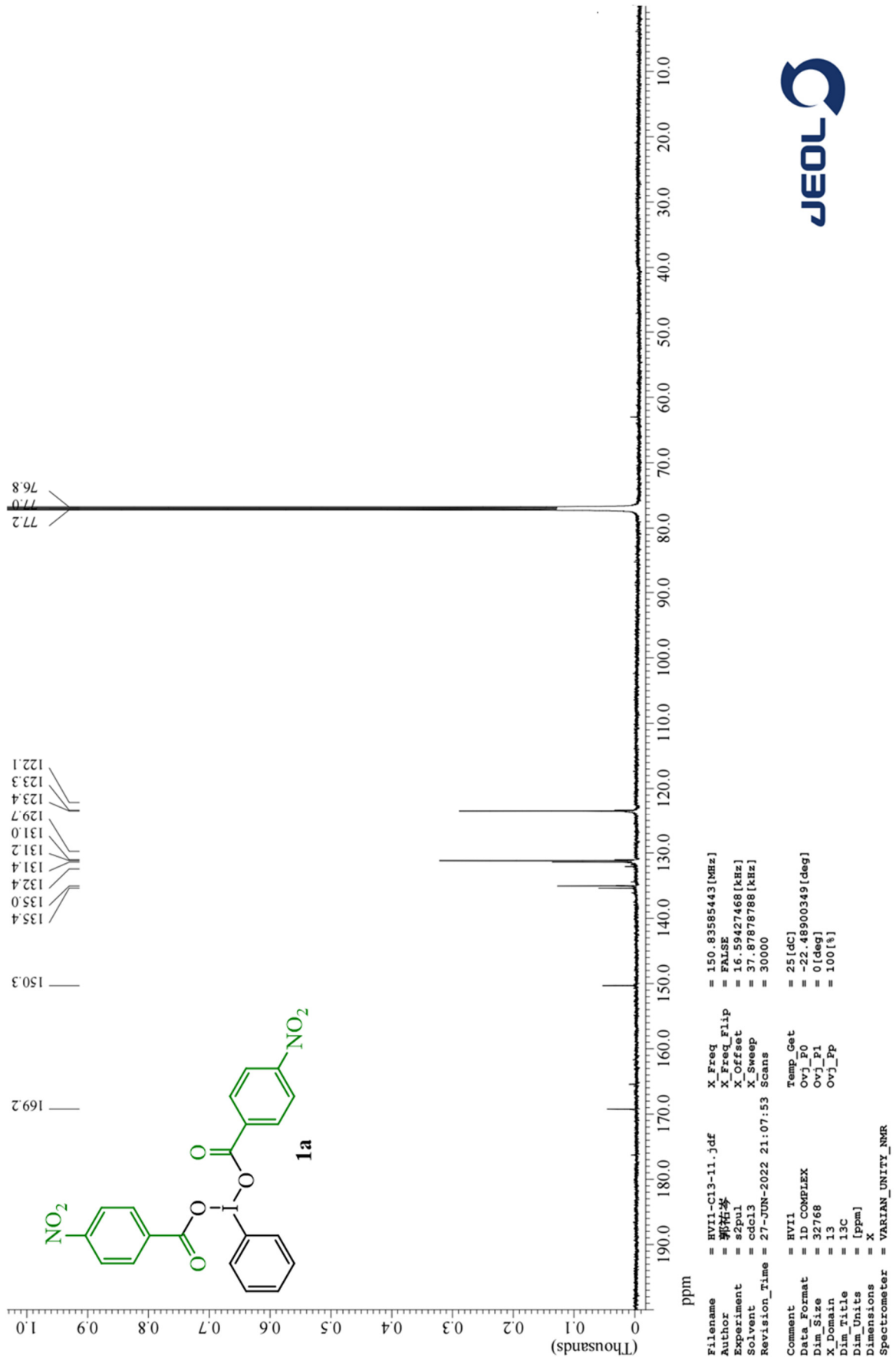


Figure S1. ESI-MS spectrum of the acyloxyphosphonium ion (**i**, R = $C_6H_4pNO_2$)

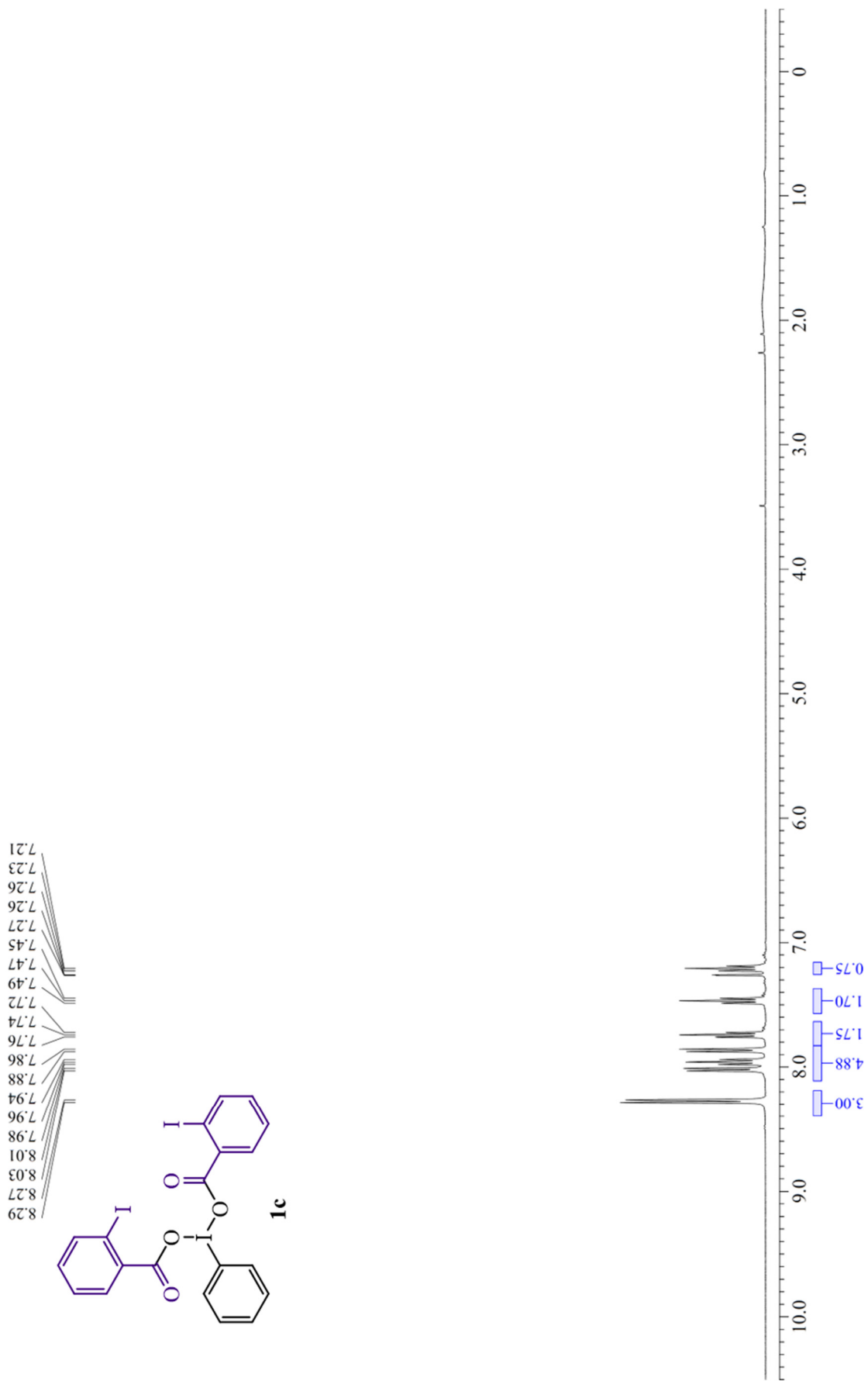
C. NMR Spectra for the synthesized compounds



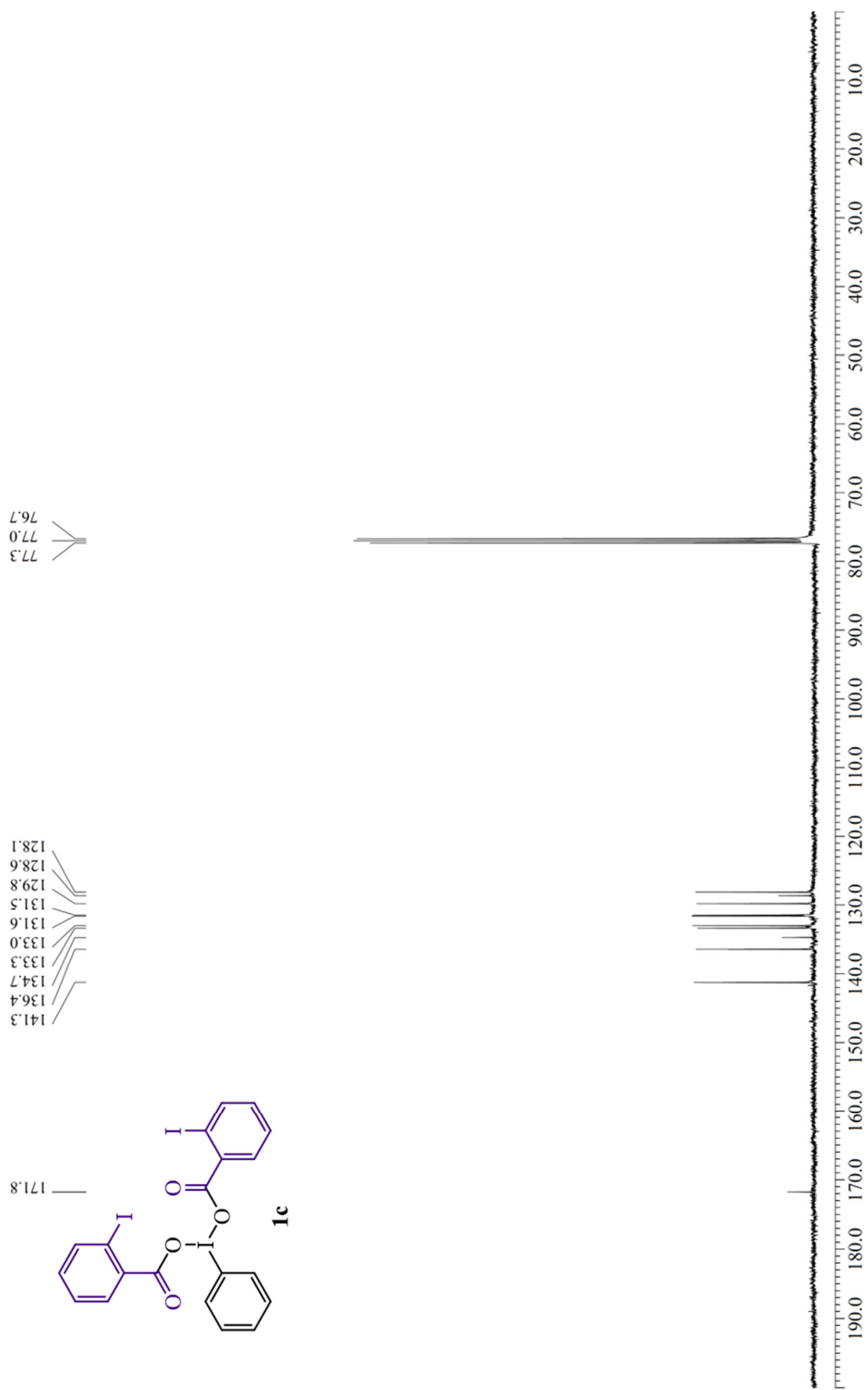
¹H NMR spectrum of compound **1a** (400 MHz, CDCl₃)



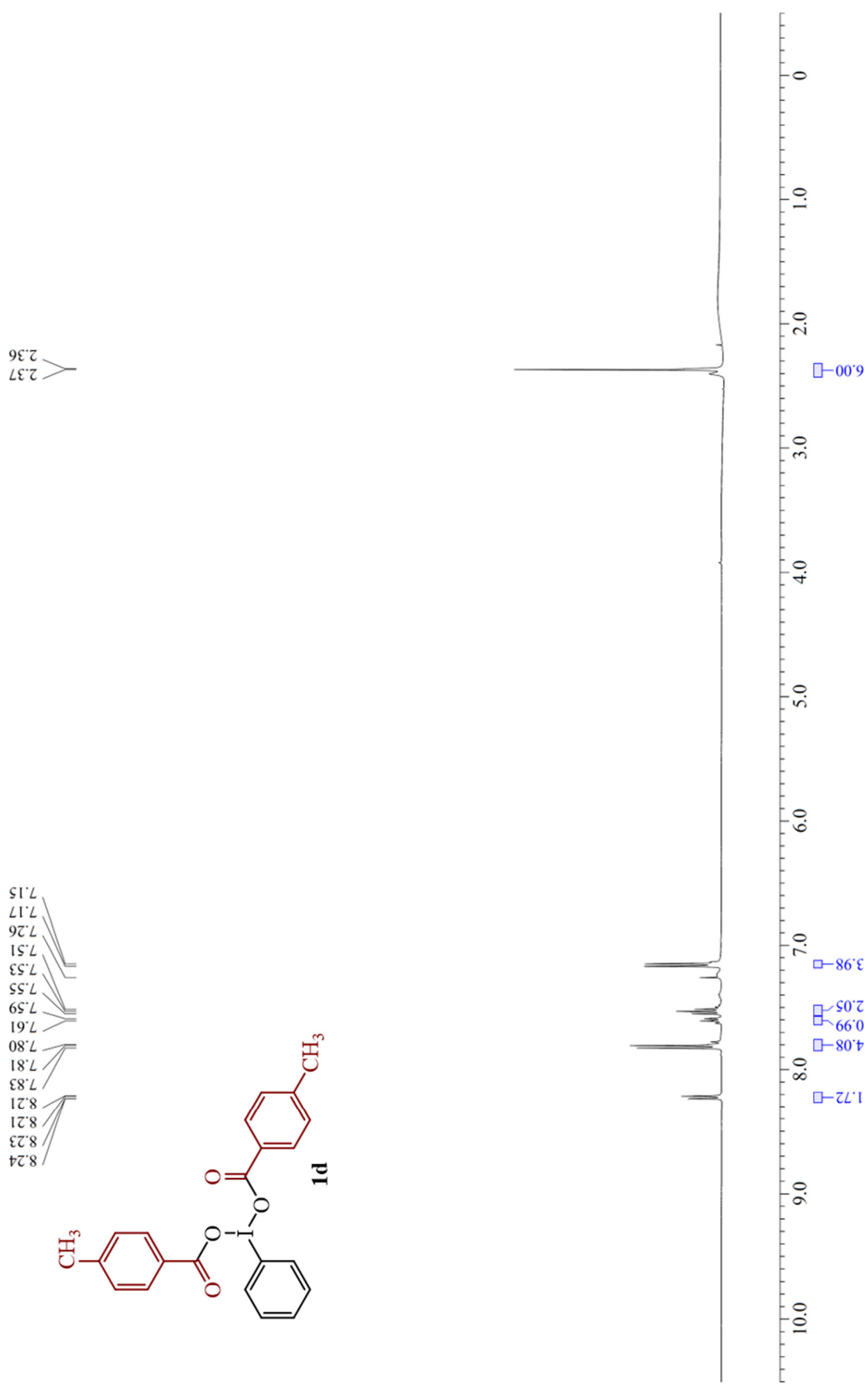
$^{13}\text{C}\{\text{H}\}$ NMR spectrum of compound **1a** (100 MHz, CDCl_3)



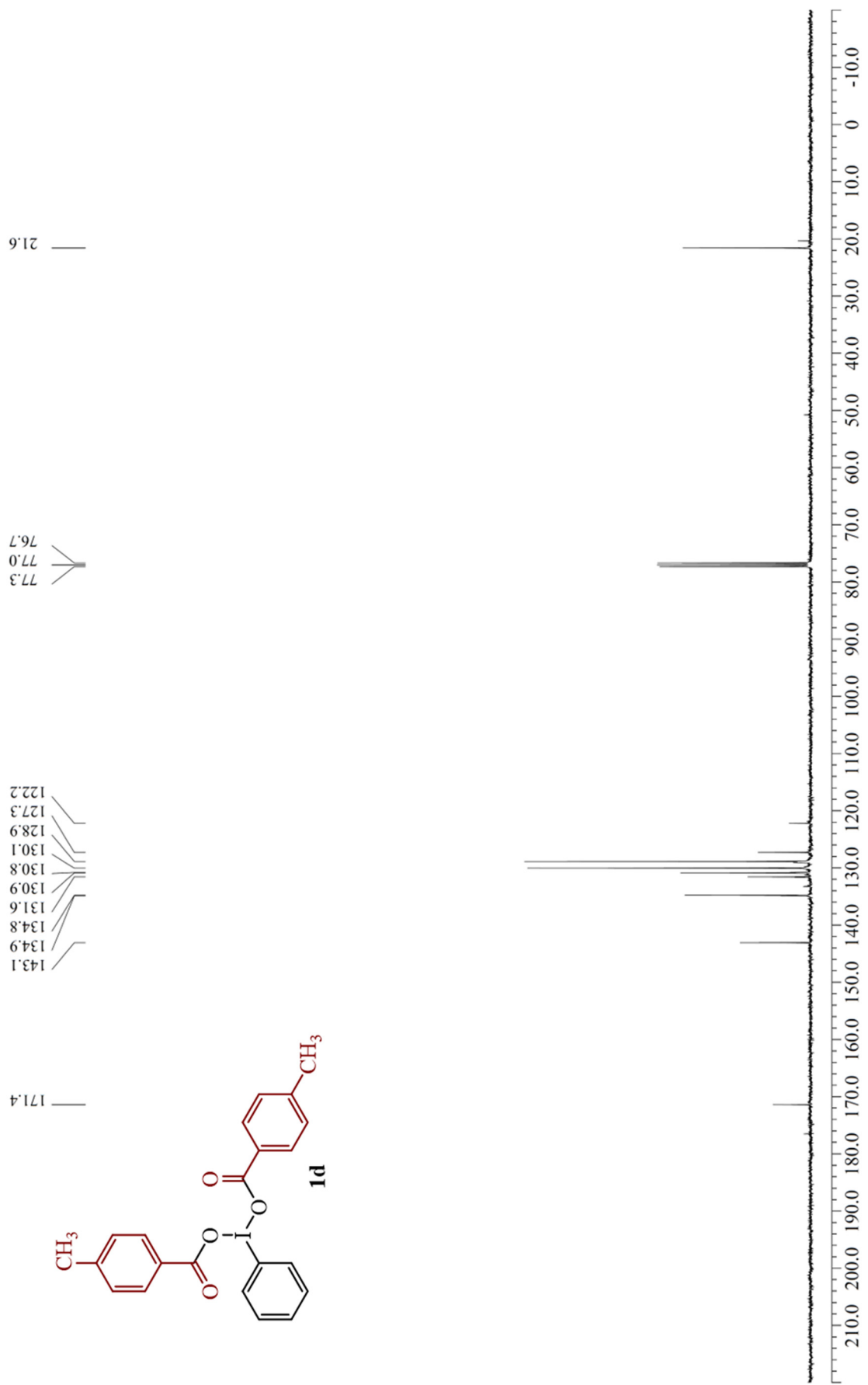
^1H NMR spectrum of compound **1c** (400 MHz, CDCl_3)



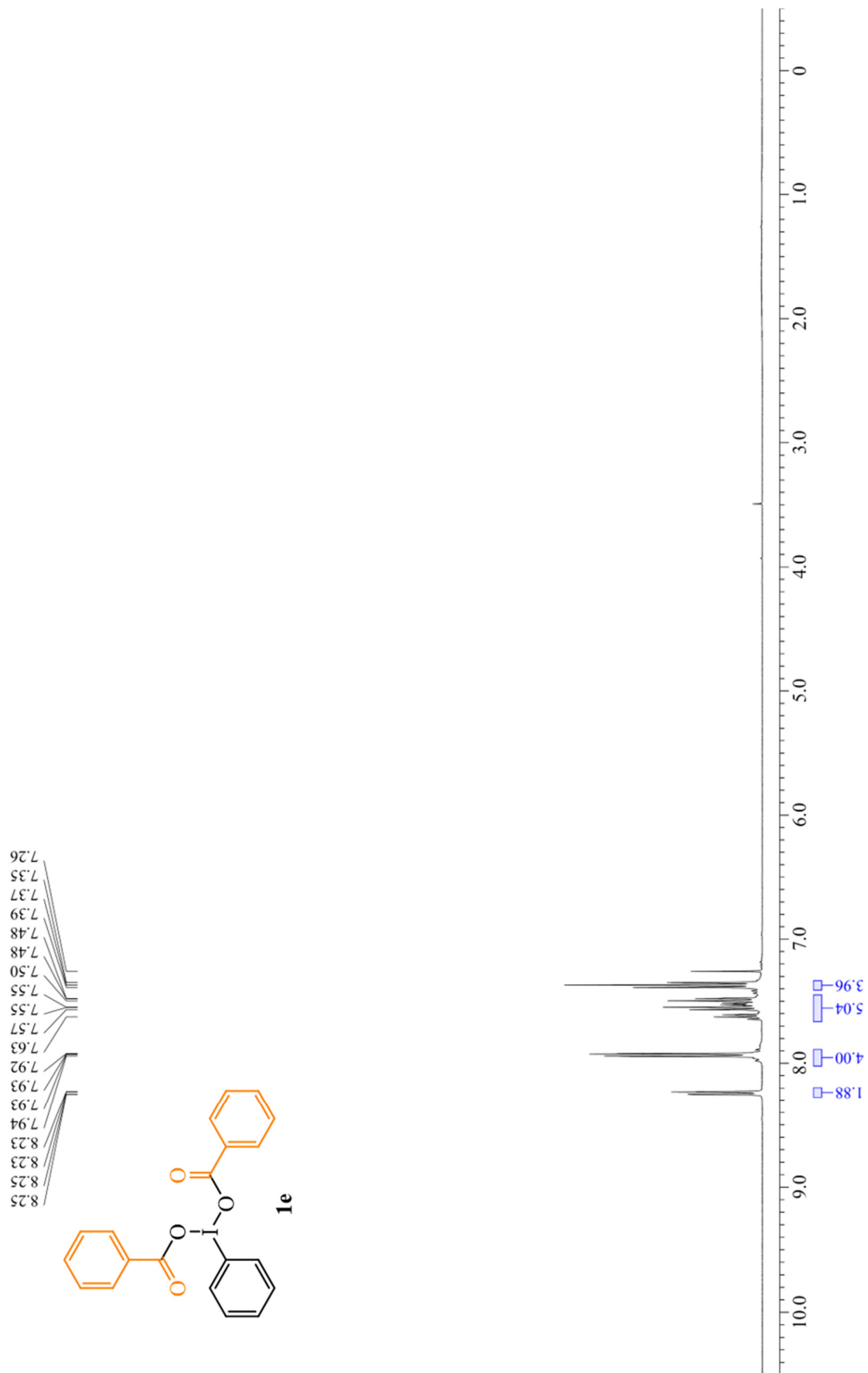
$^{13}\text{C}\{\text{H}\}$ NMR spectrum of compound **1c** (100 MHz, CDCl_3)



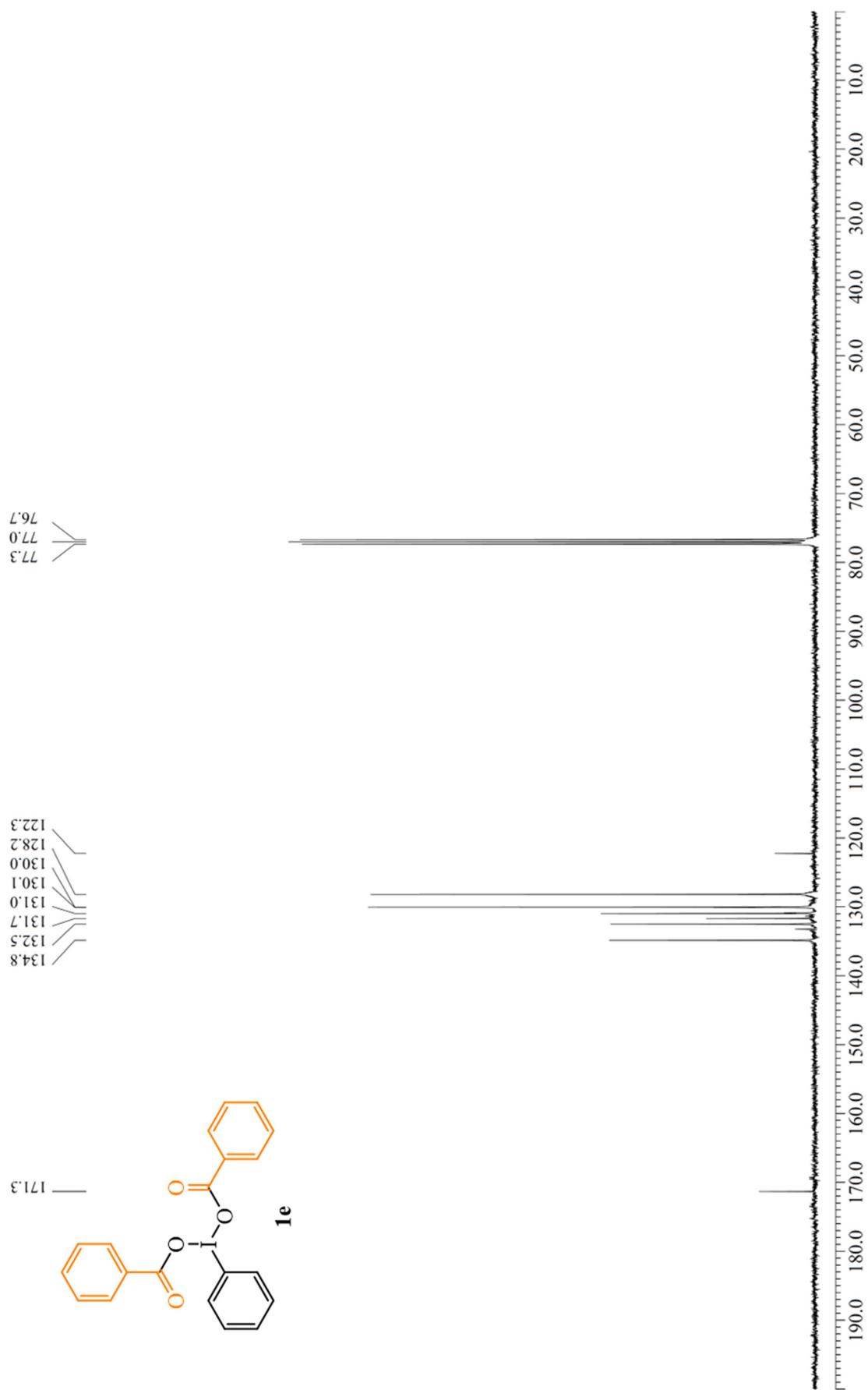
¹H NMR spectrum of compound **1d** (400 MHz, CDCl₃)



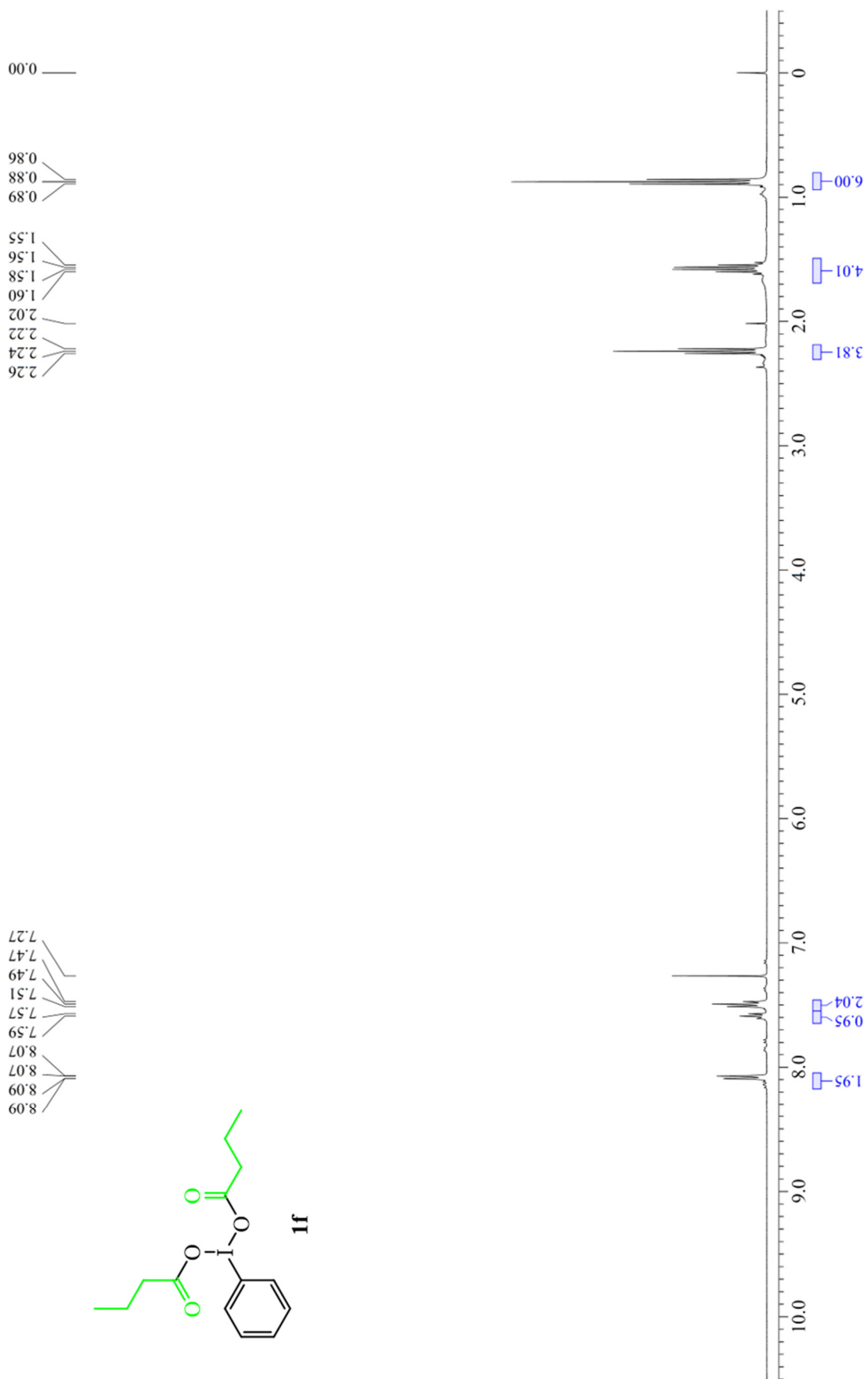
^{13}C {H} NMR spectrum of compound **1d** (100 MHz, CDCl_3)



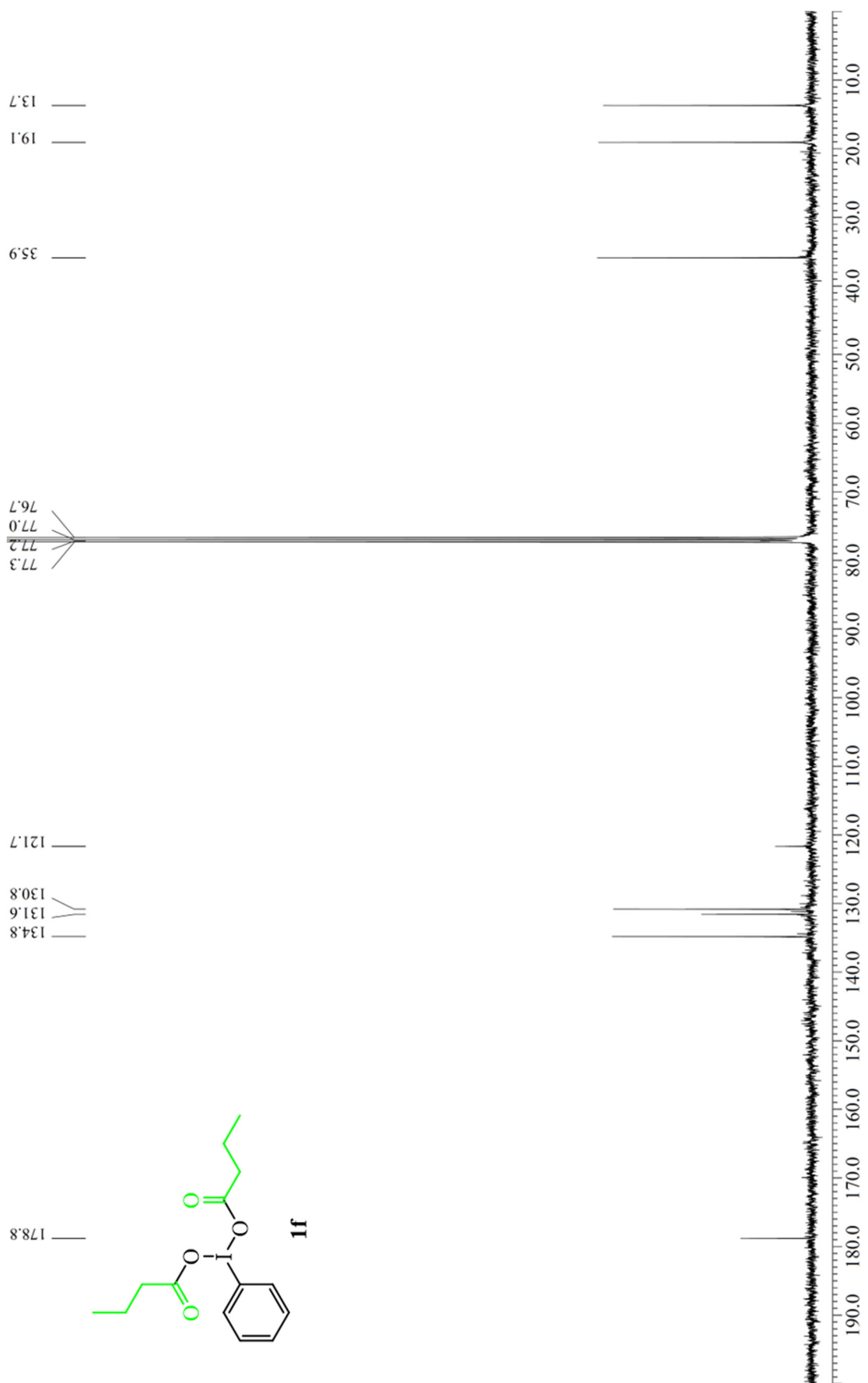
¹H NMR spectrum of compound **1e** (400 MHz, CDCl₃)



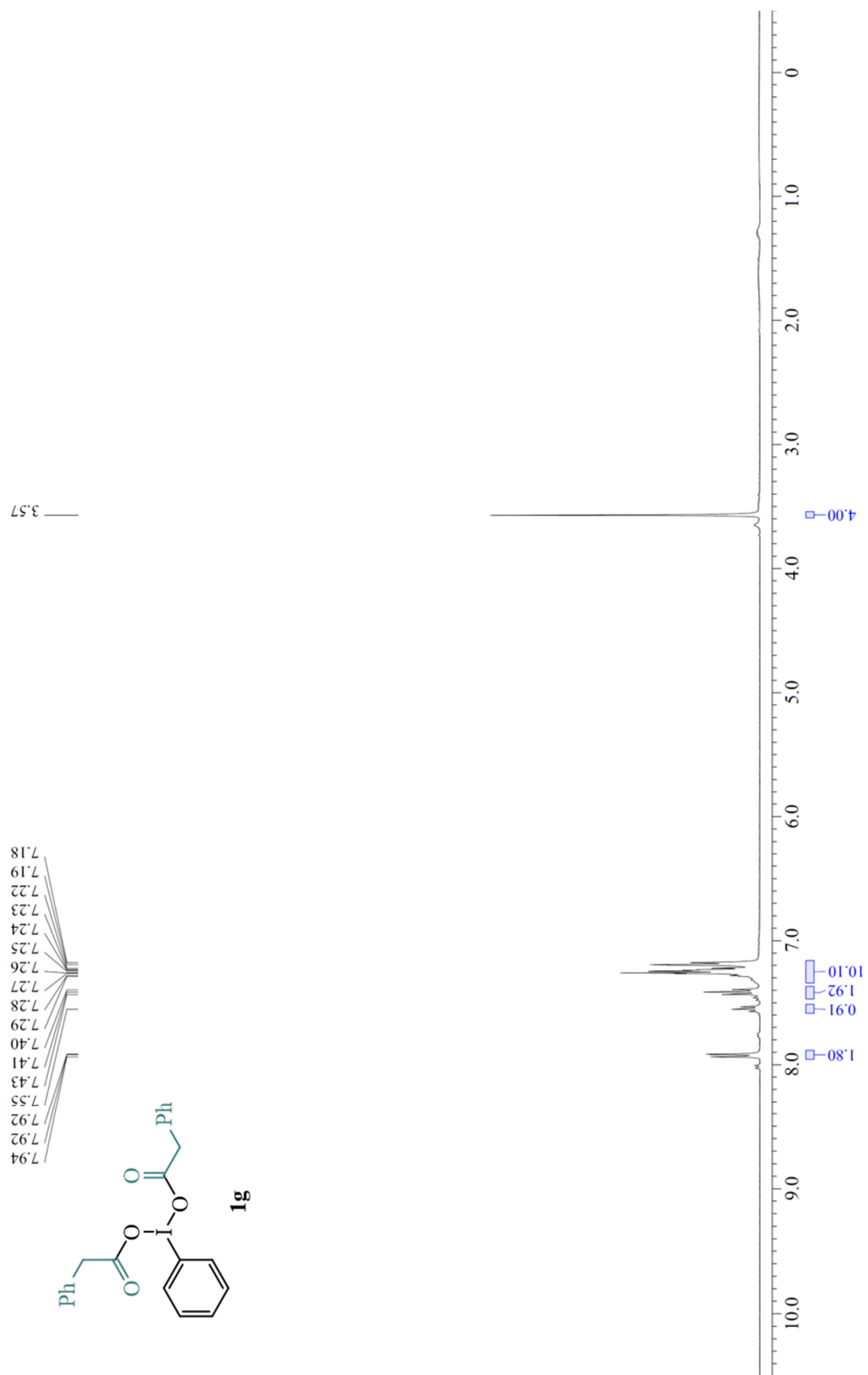
$^{13}\text{C}\{\text{H}\}$ NMR spectrum of compound **1e** (100 MHz, CDCl_3)



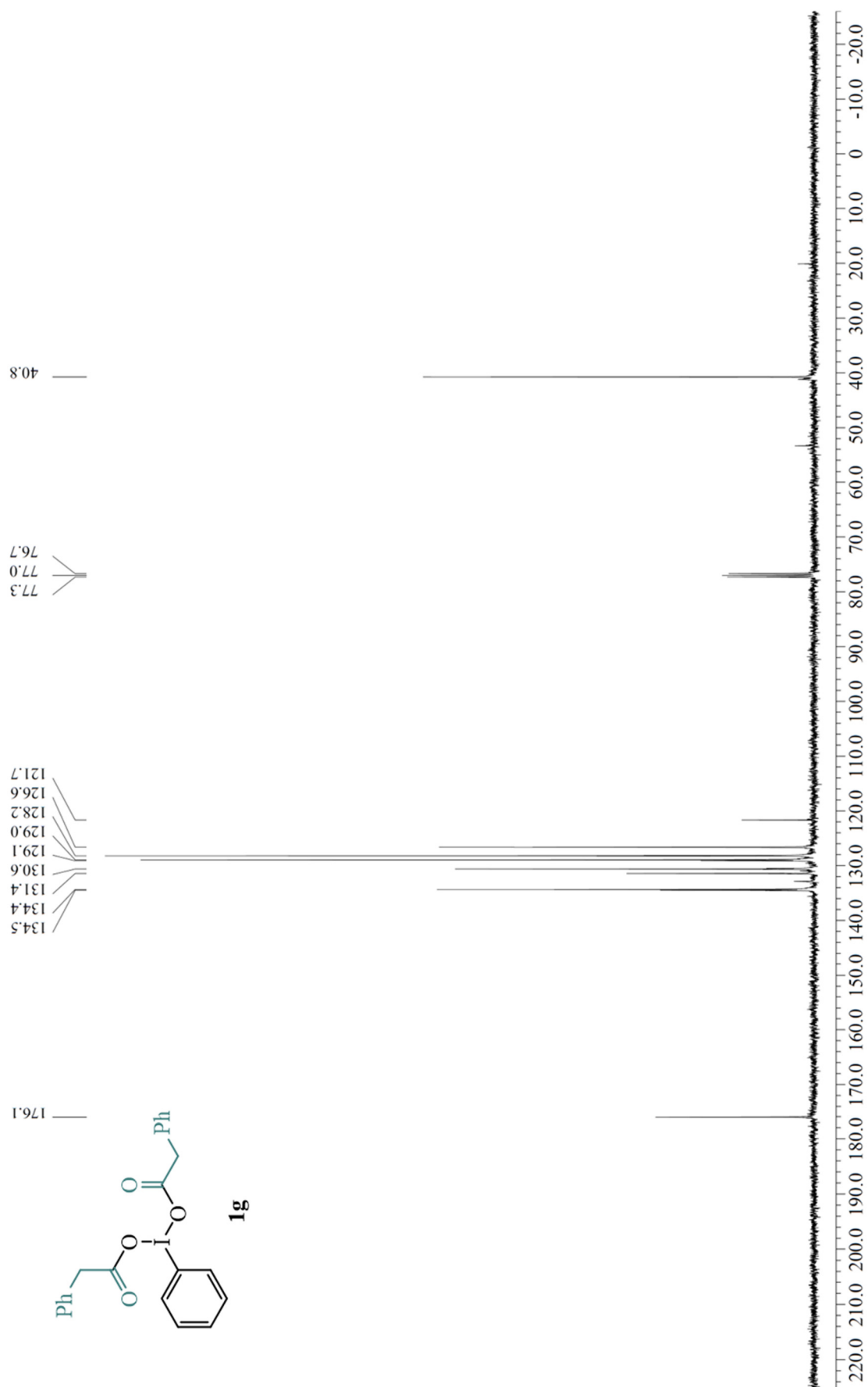
¹H NMR spectrum of compound **1f** (400 MHz, CDCl₃)



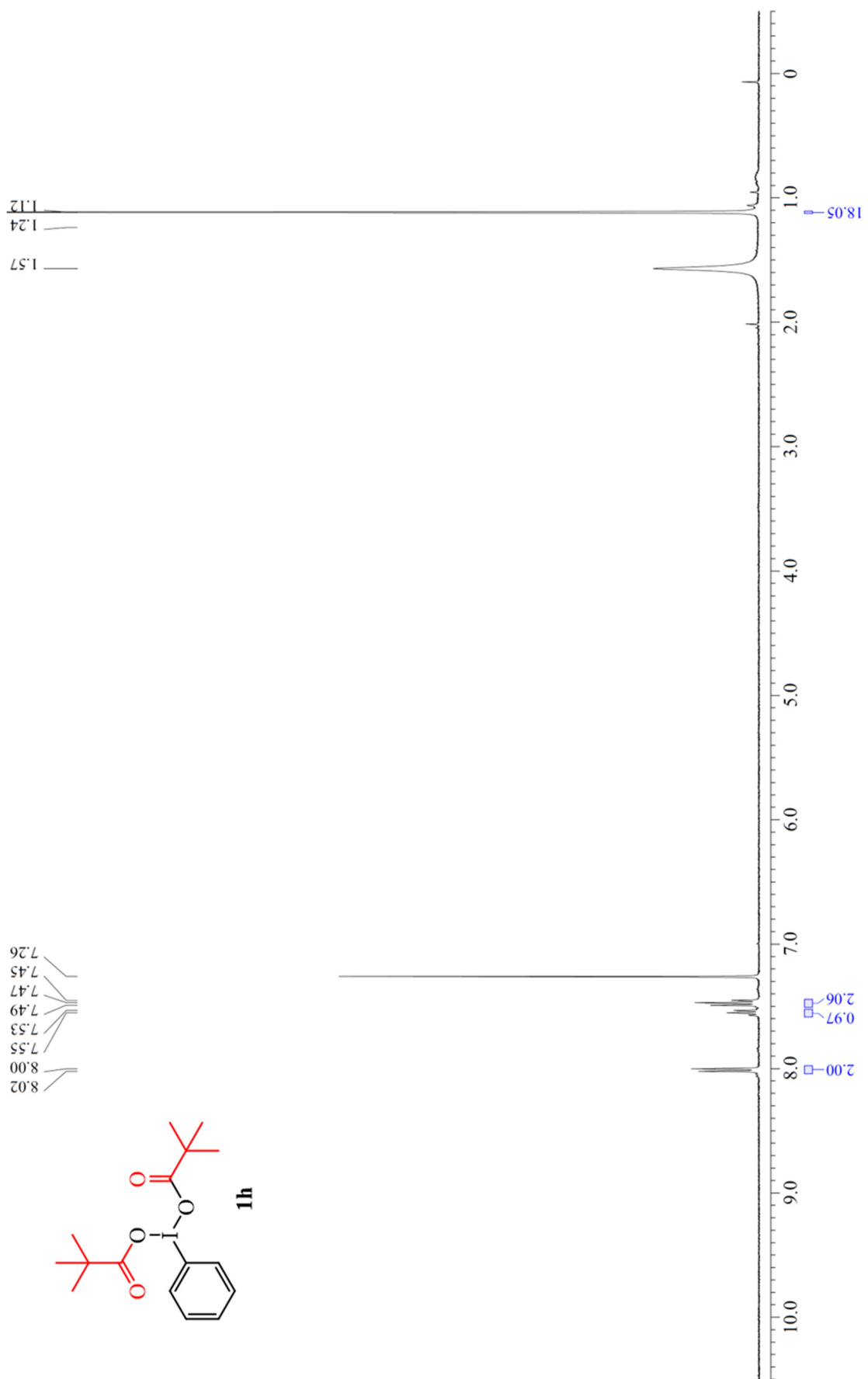
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **1f** (100 MHz, CDCl_3)



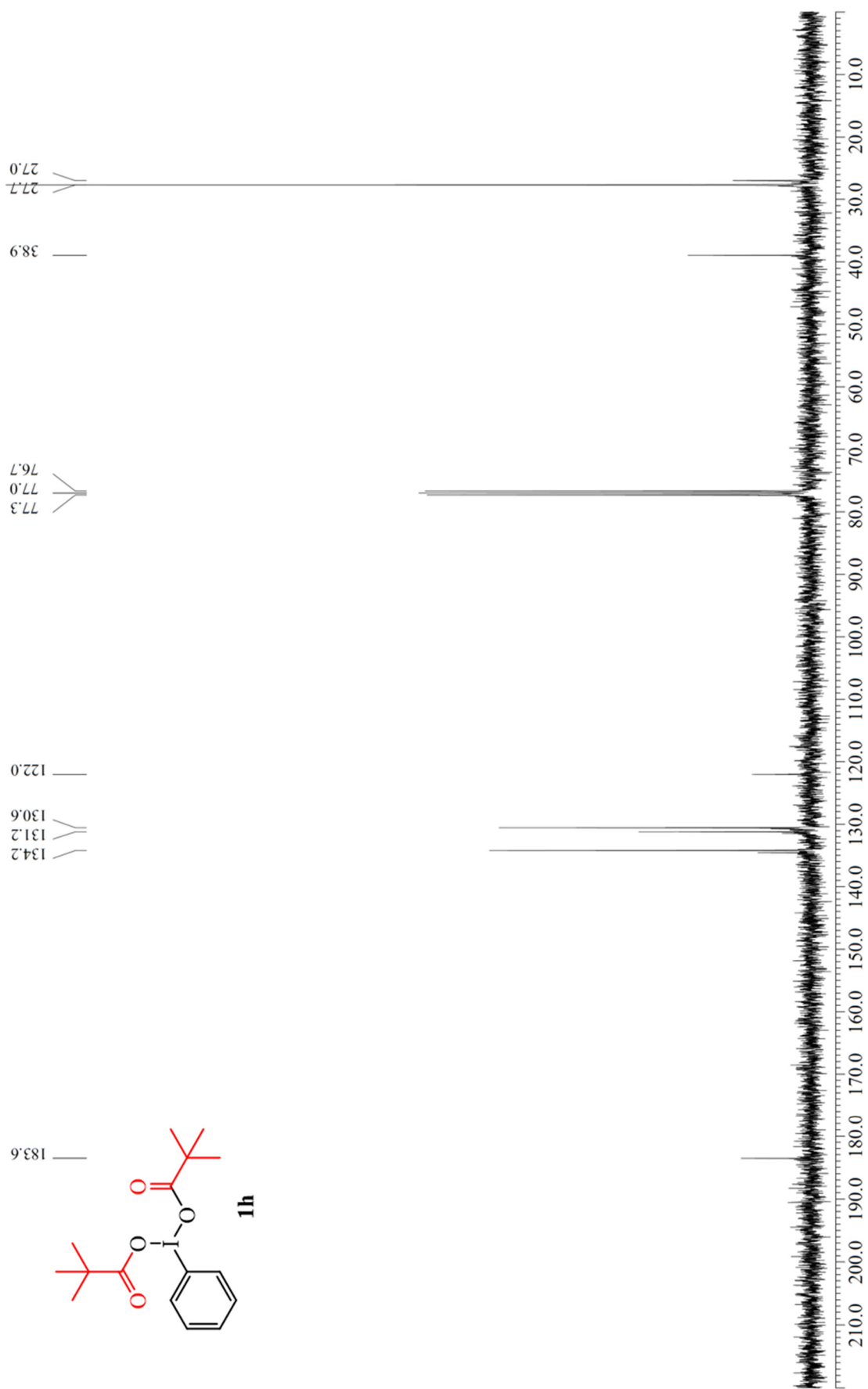
^1H NMR spectrum of compound **1g** (400 MHz, CDCl_3)



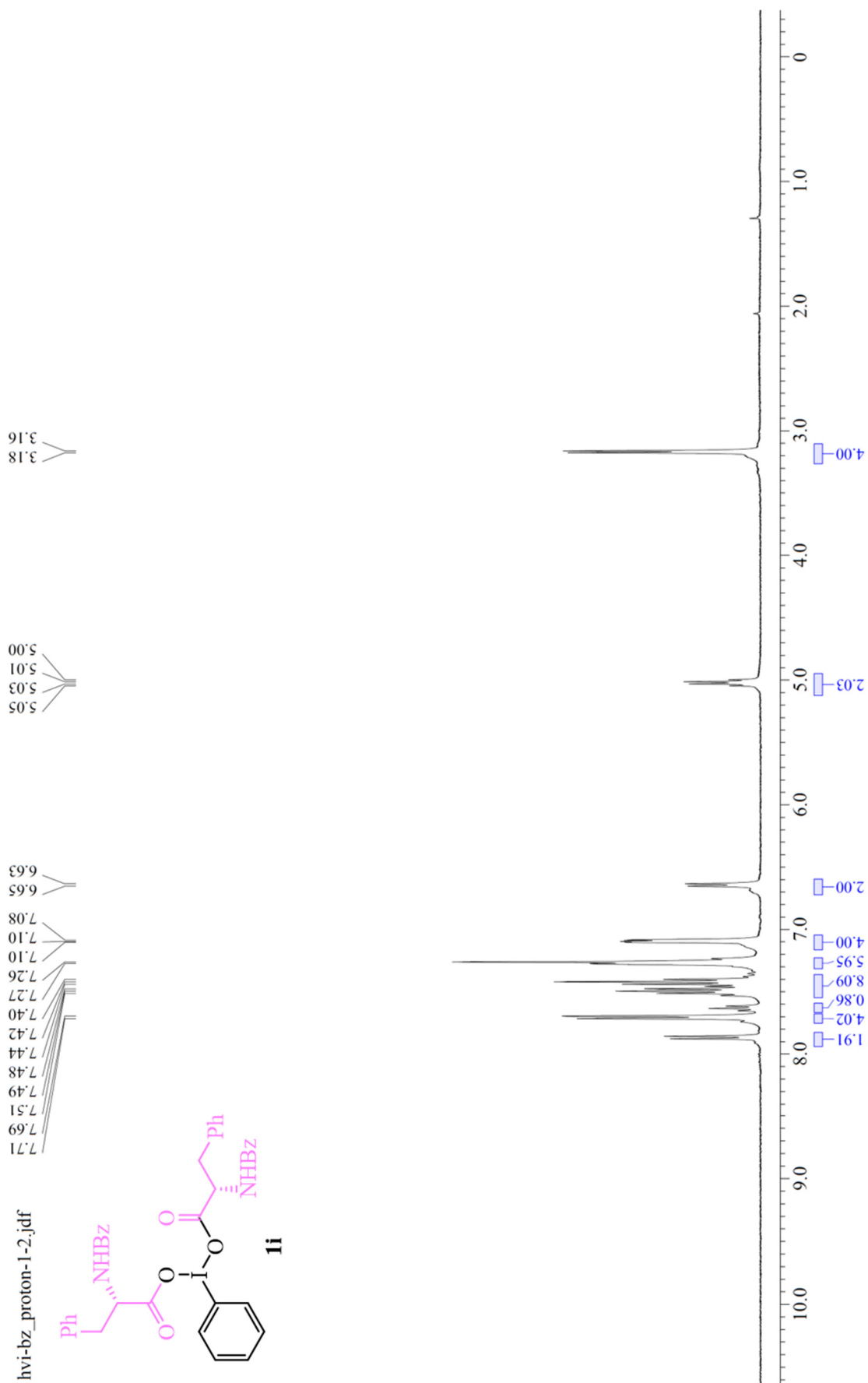
¹³C{H} NMR spectrum of compound **1g** (100 MHz, CDCl₃)



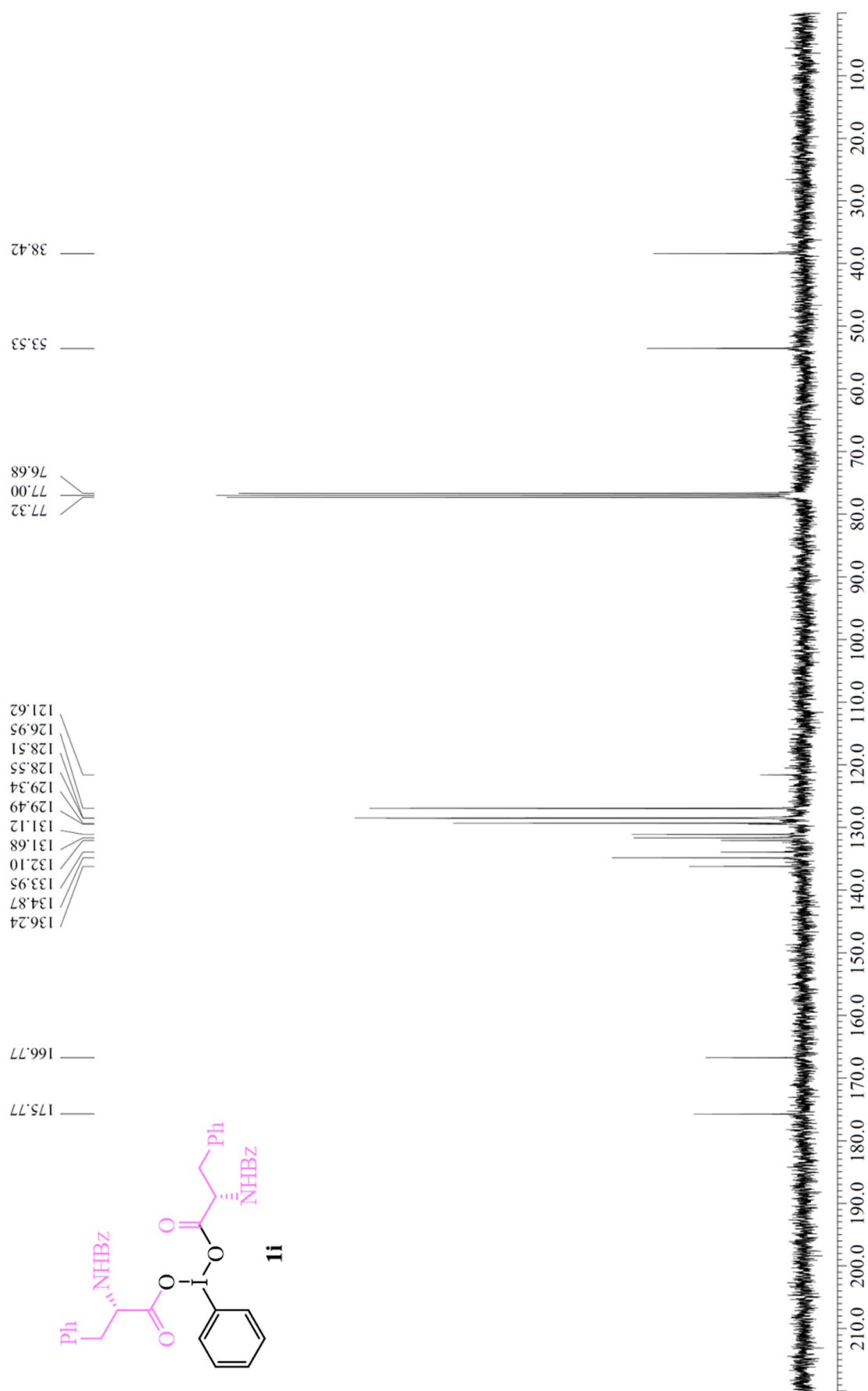
^1H NMR spectrum of compound **1h** (400 MHz, CDCl_3)



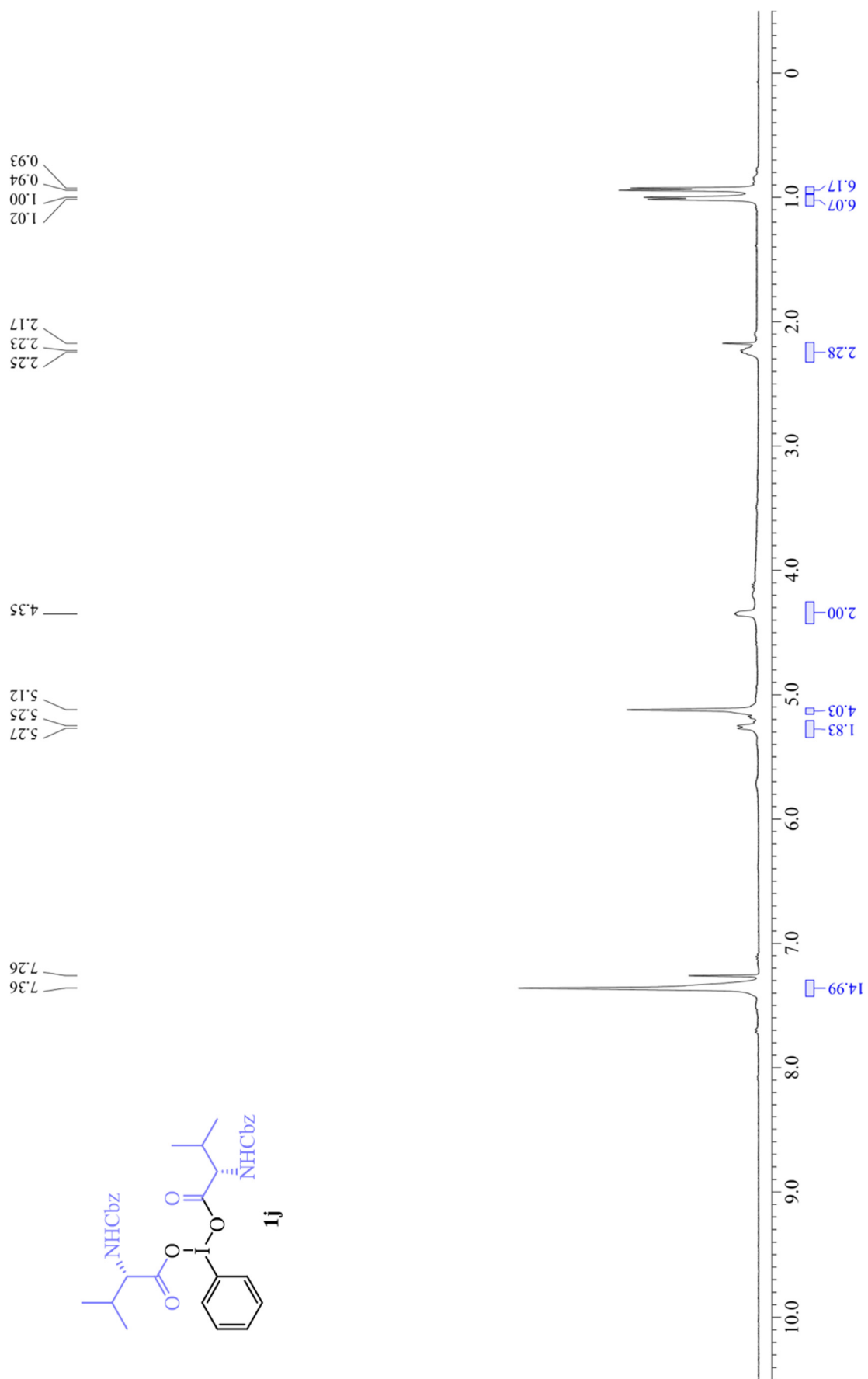
¹³C {H} NMR spectrum of compound **1h** (100 MHz, CDCl₃)



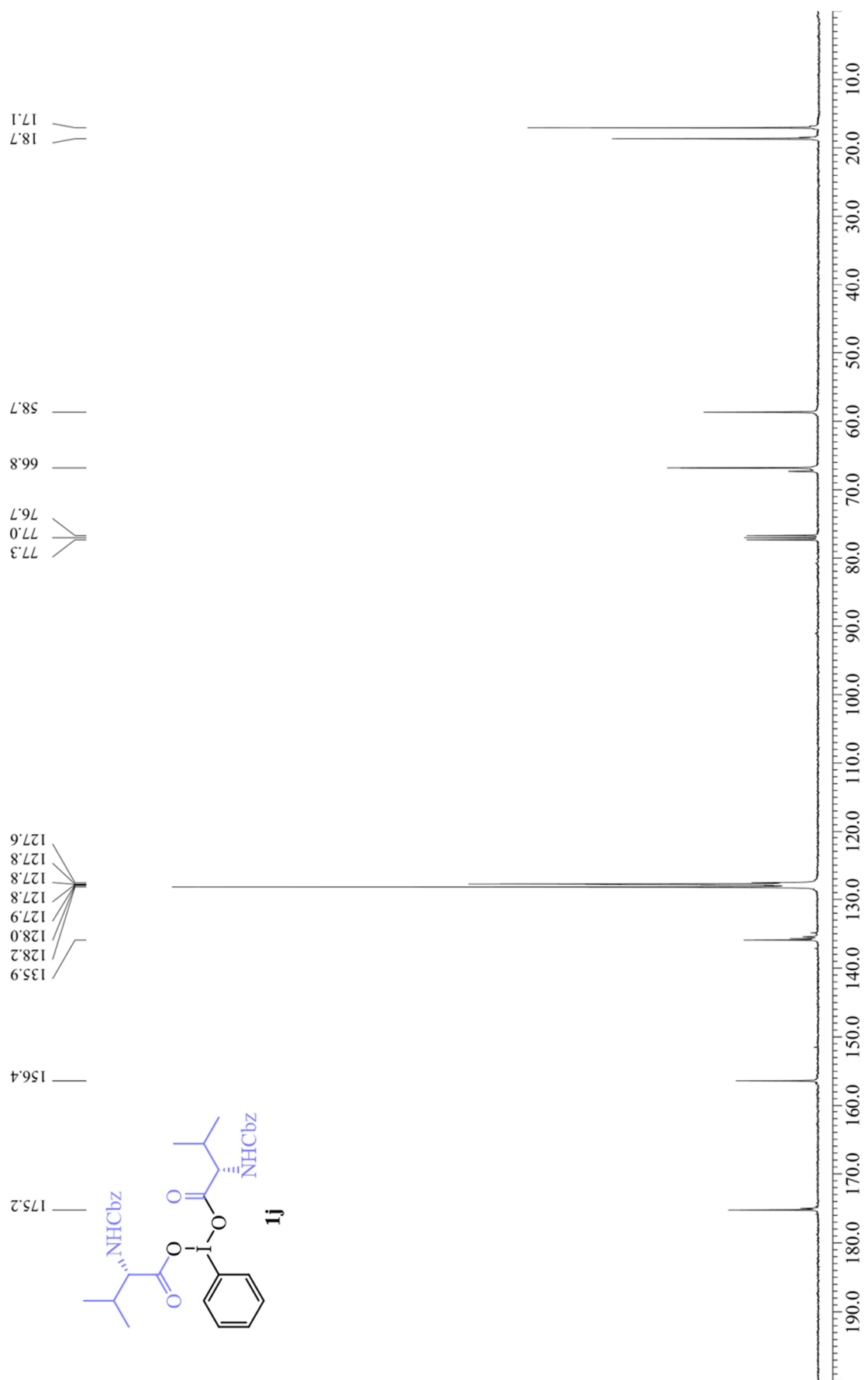
^1H NMR spectrum of compound **1i** (400 MHz, CDCl_3)

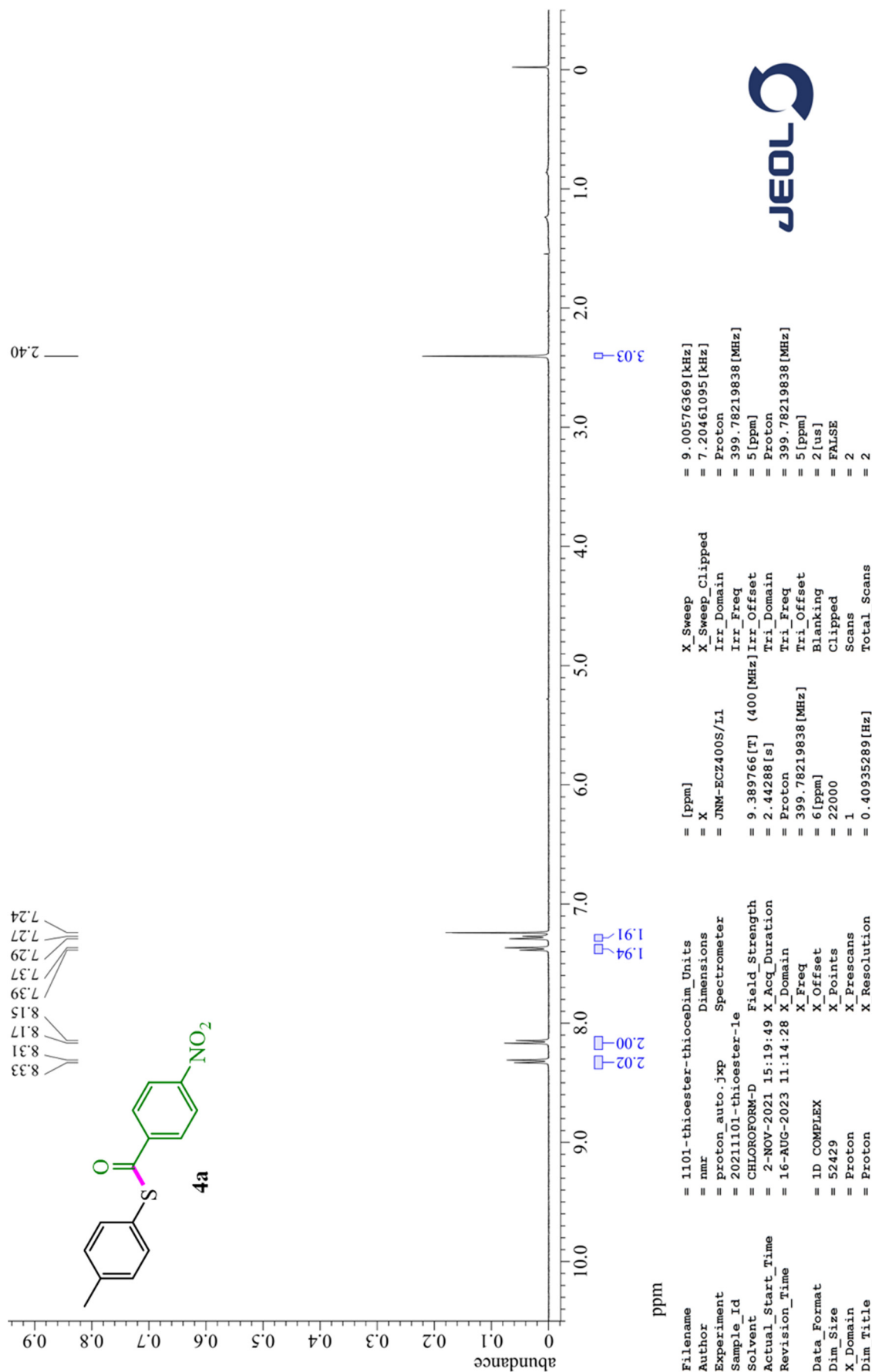


¹³C{H} NMR spectrum of compound **1i (100 MHz, CDCl₃)**

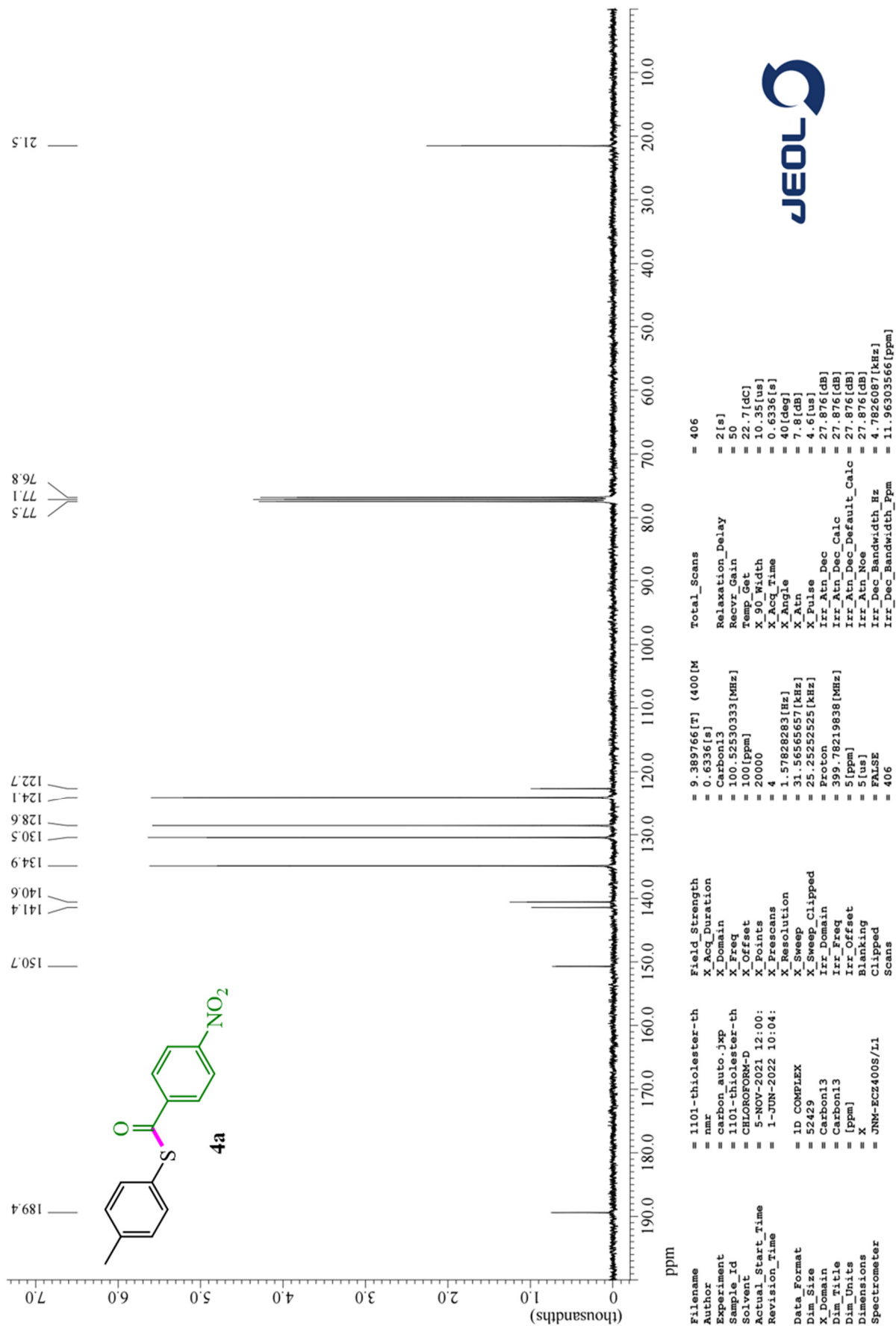


¹H NMR spectrum of compound 1j (400 MHz, CDCl₃)

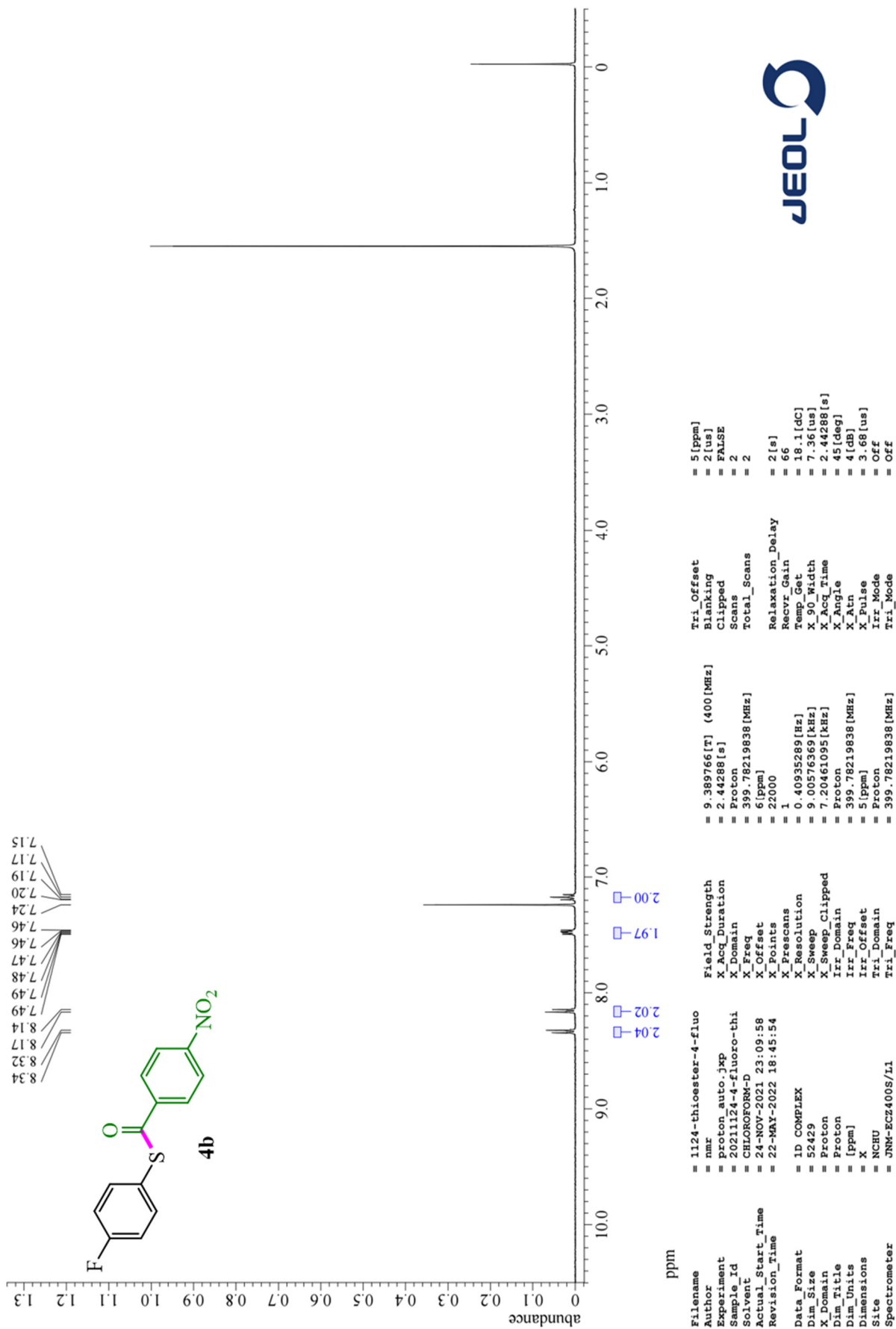




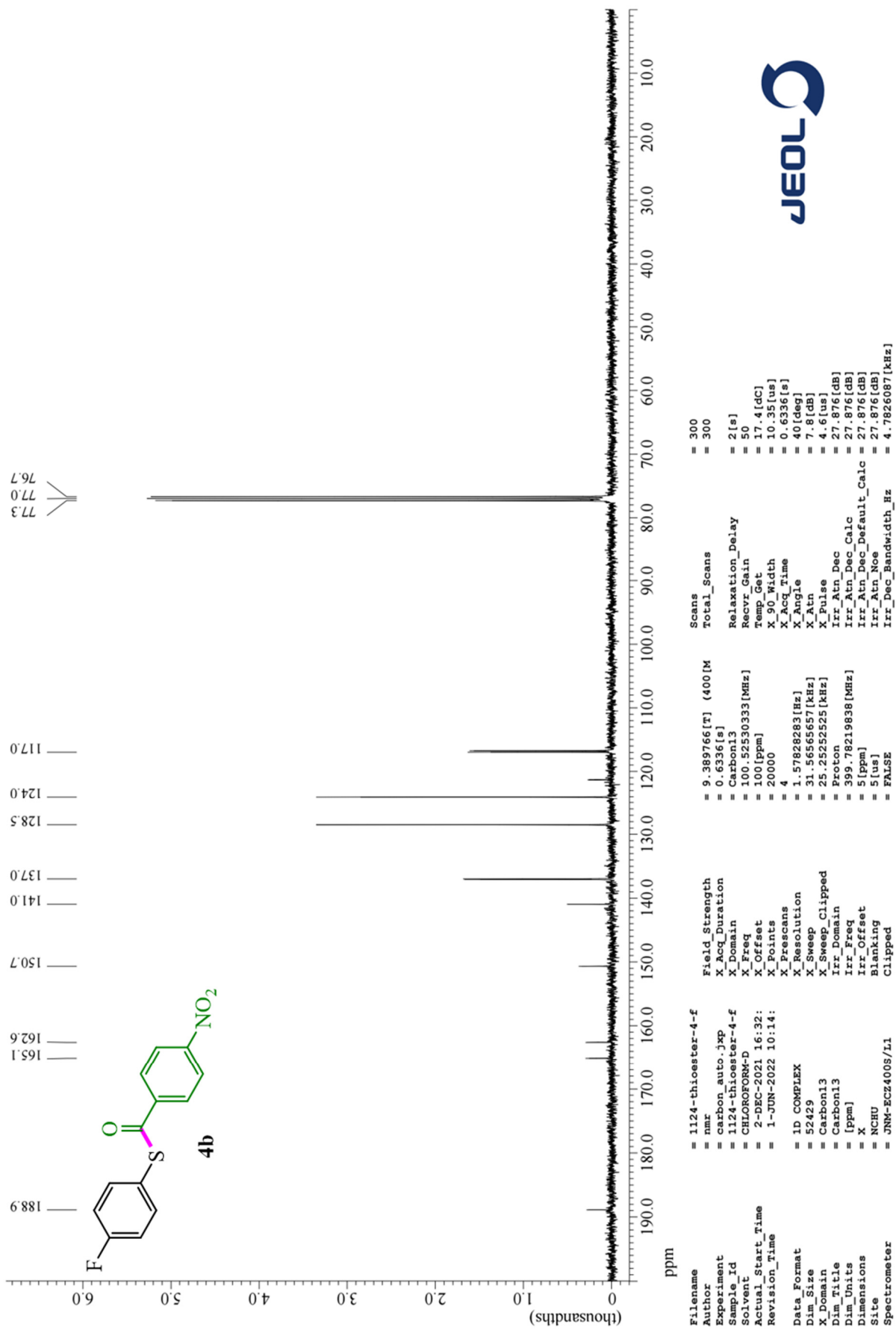
¹H NMR spectrum of compound **4a** (400 MHz, CDCl₃)



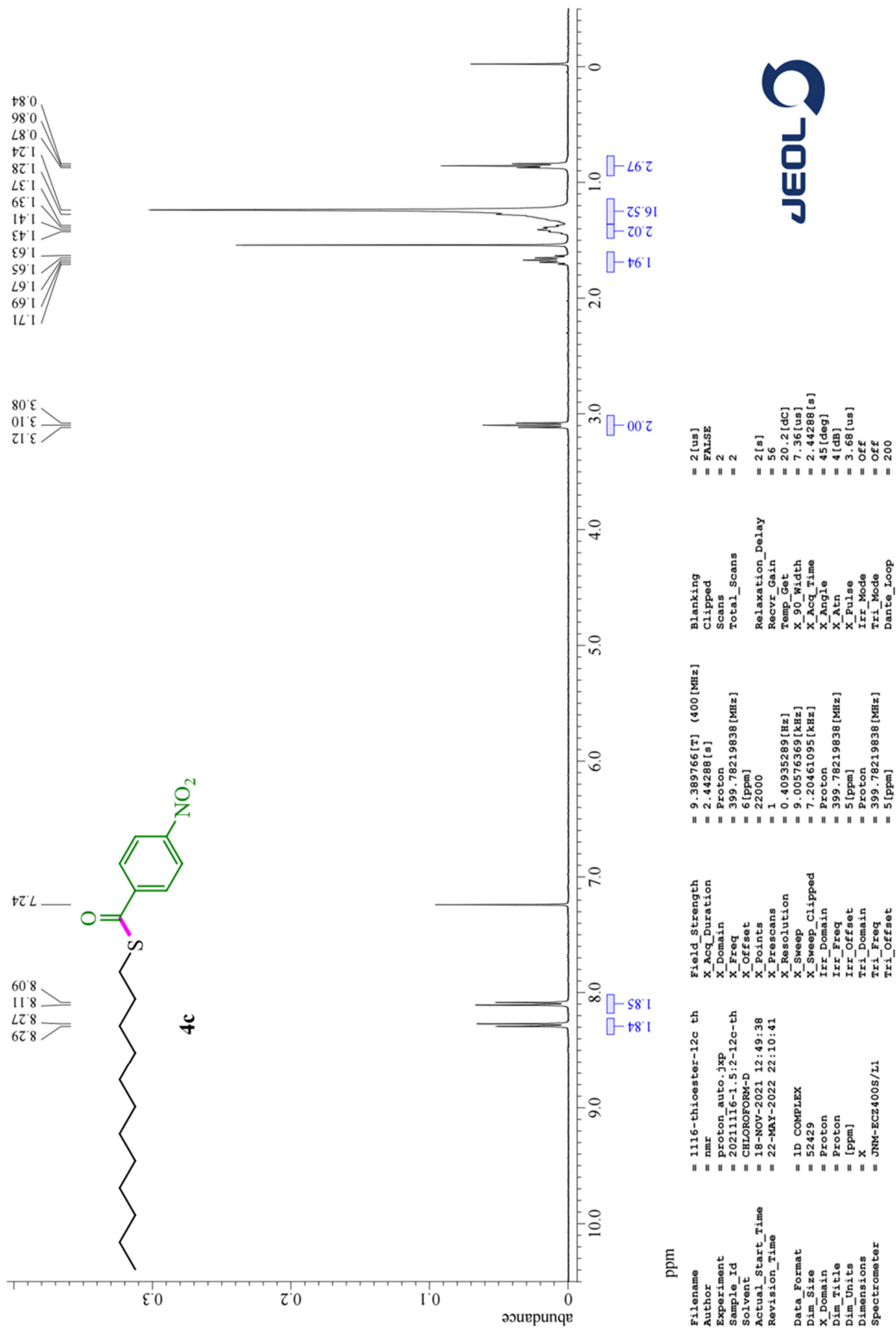
¹³C{H} NMR spectrum of compound **4a** (100 MHz, CDCl₃)



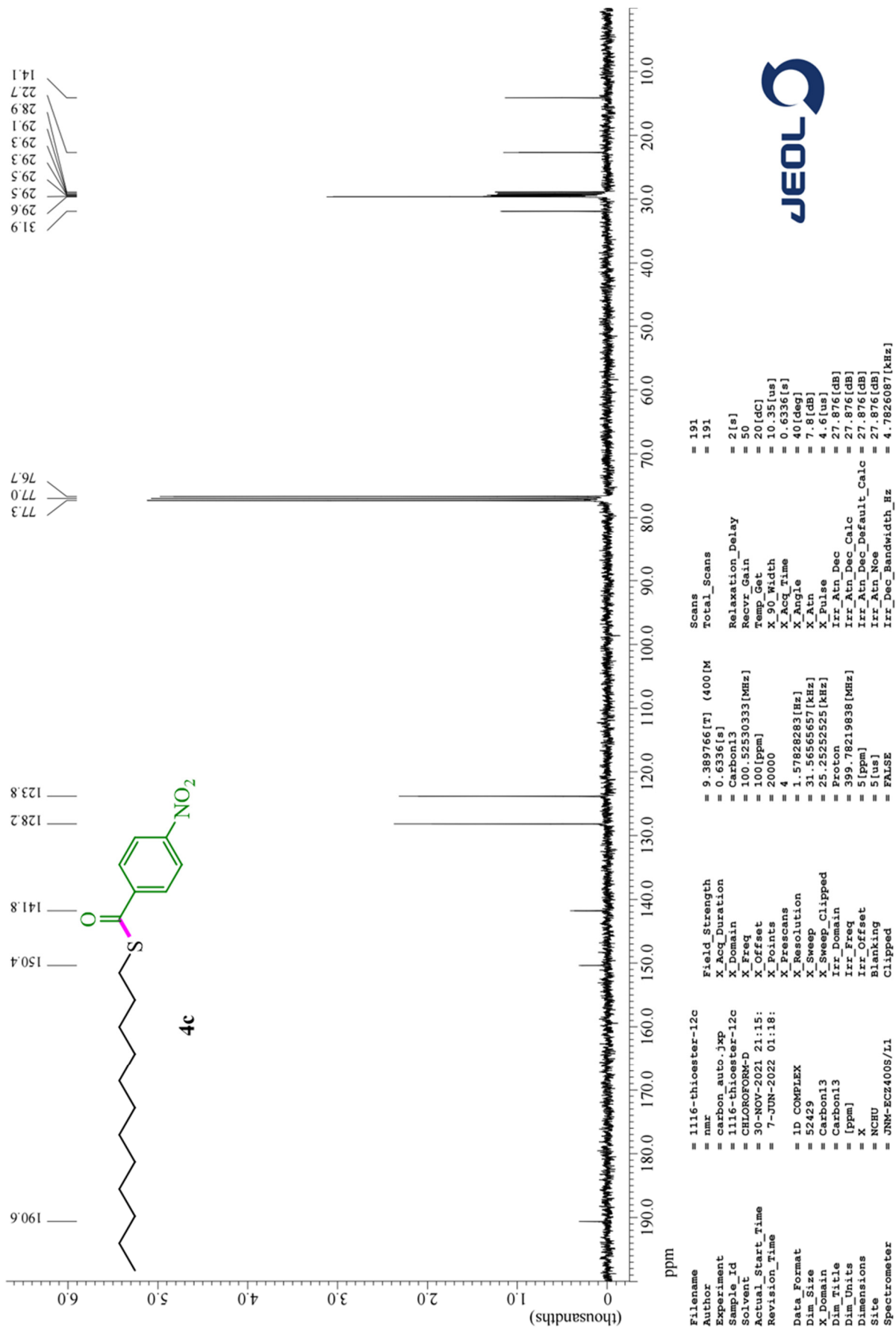
¹H NMR spectrum of compound **4b** (400 MHz, CDCl₃)



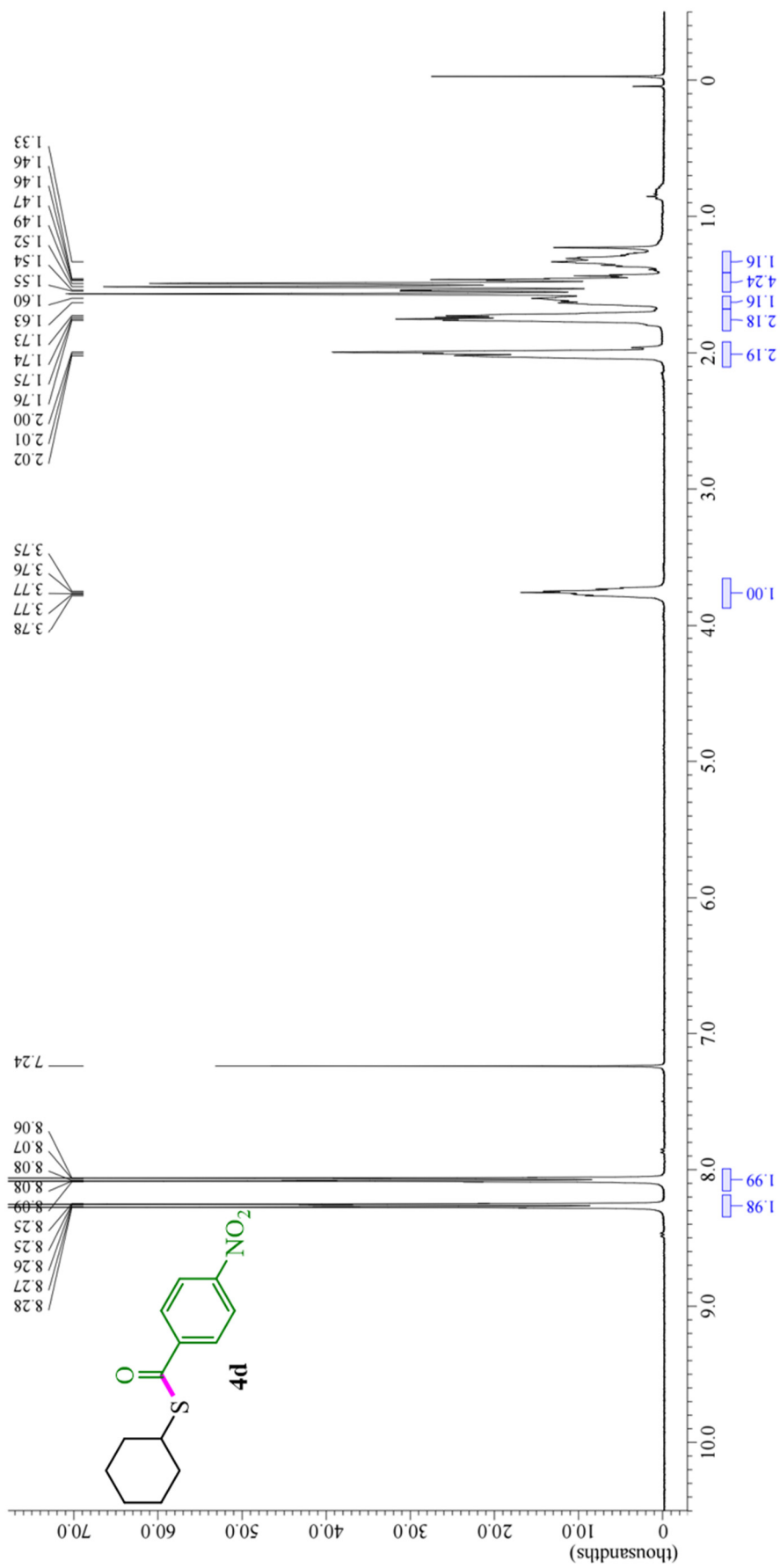
¹³C {H} NMR spectrum of compound **4b** (100 MHz, CDCl₃)



¹H NMR spectrum of compound **4c** (400 MHz, CDCl₃)



¹³C{H} NMR spectrum of compound **4c** (100 MHz, CDCl₃)



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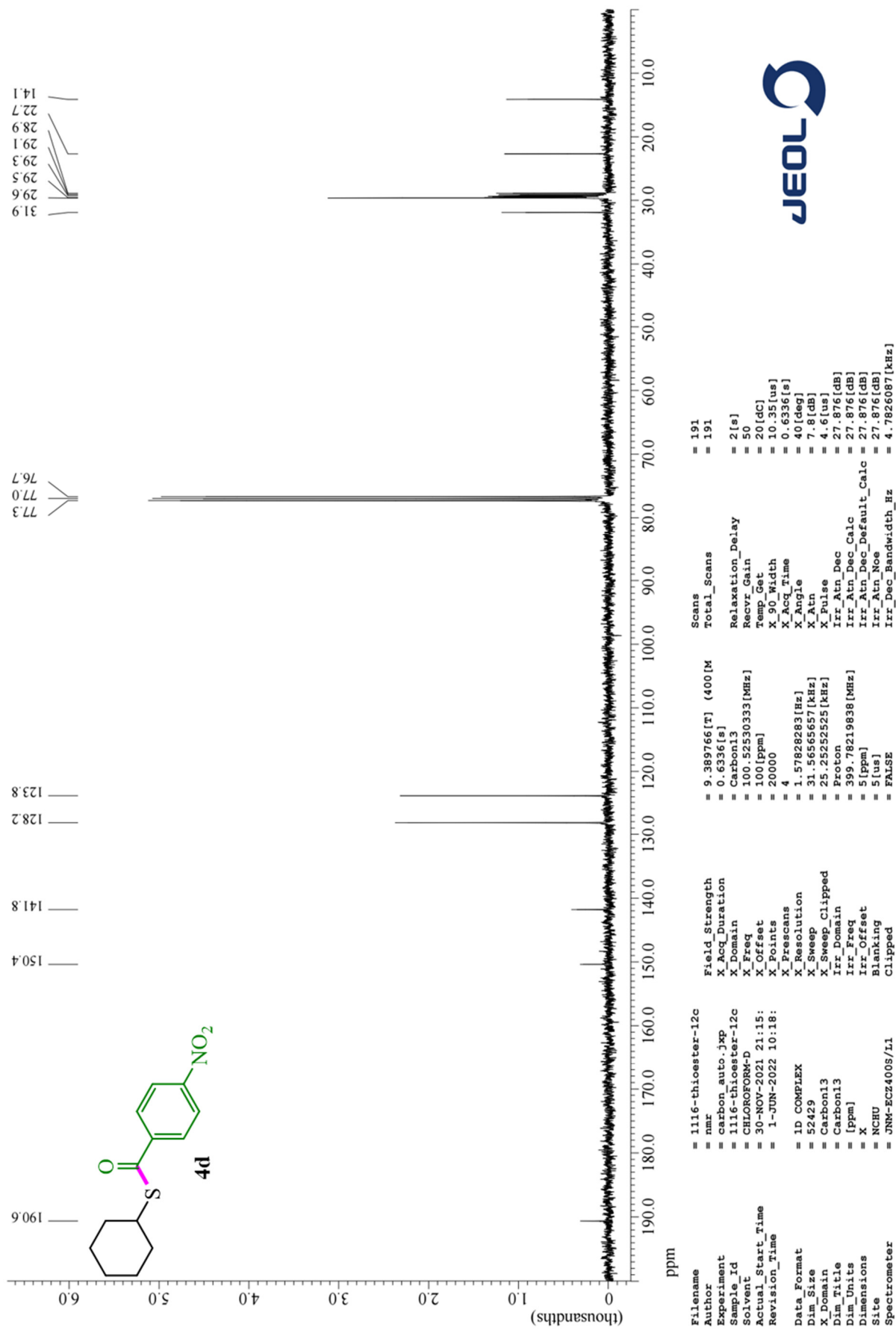
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 Experiment = proton_auto.jpg
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 Revision_Time = 26-MAY-2022 22:06:41

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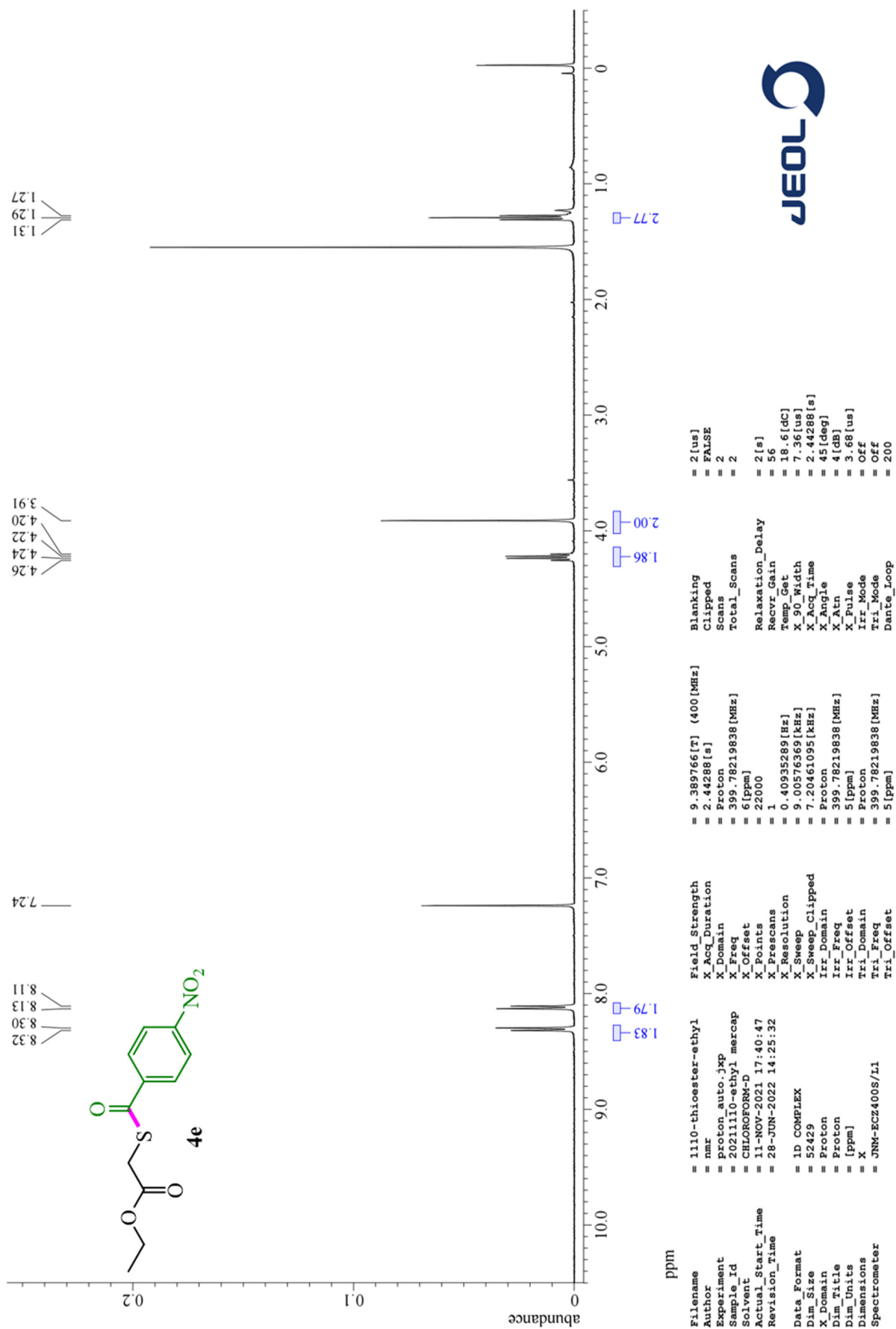
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 X_Sweep_Clip = 7.20461095 [kHz]
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 Irr_Freq = 399.78219838 [MHz]
 Irr_Offset = 5 [ppm]
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Tri_Offset = 5 [ppm]
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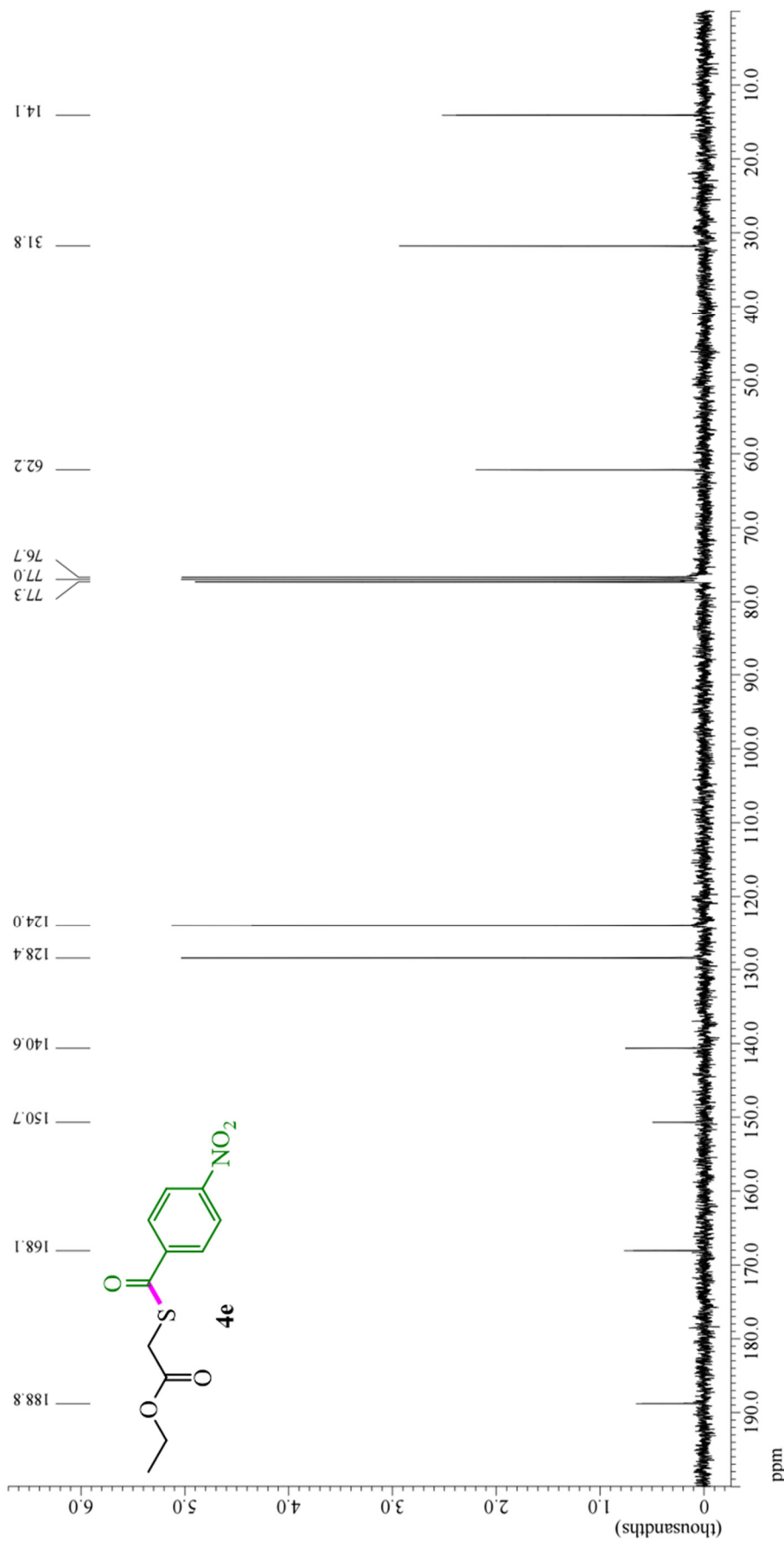
¹H NMR spectrum of compound **4d** (400 MHz, CDCl₃)



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4d** (100 MHz, CDCl_3)



¹H NMR spectrum of compound **4e** (400 MHz, CDCl₃)



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4e** (100 MHz, CDCl_3)

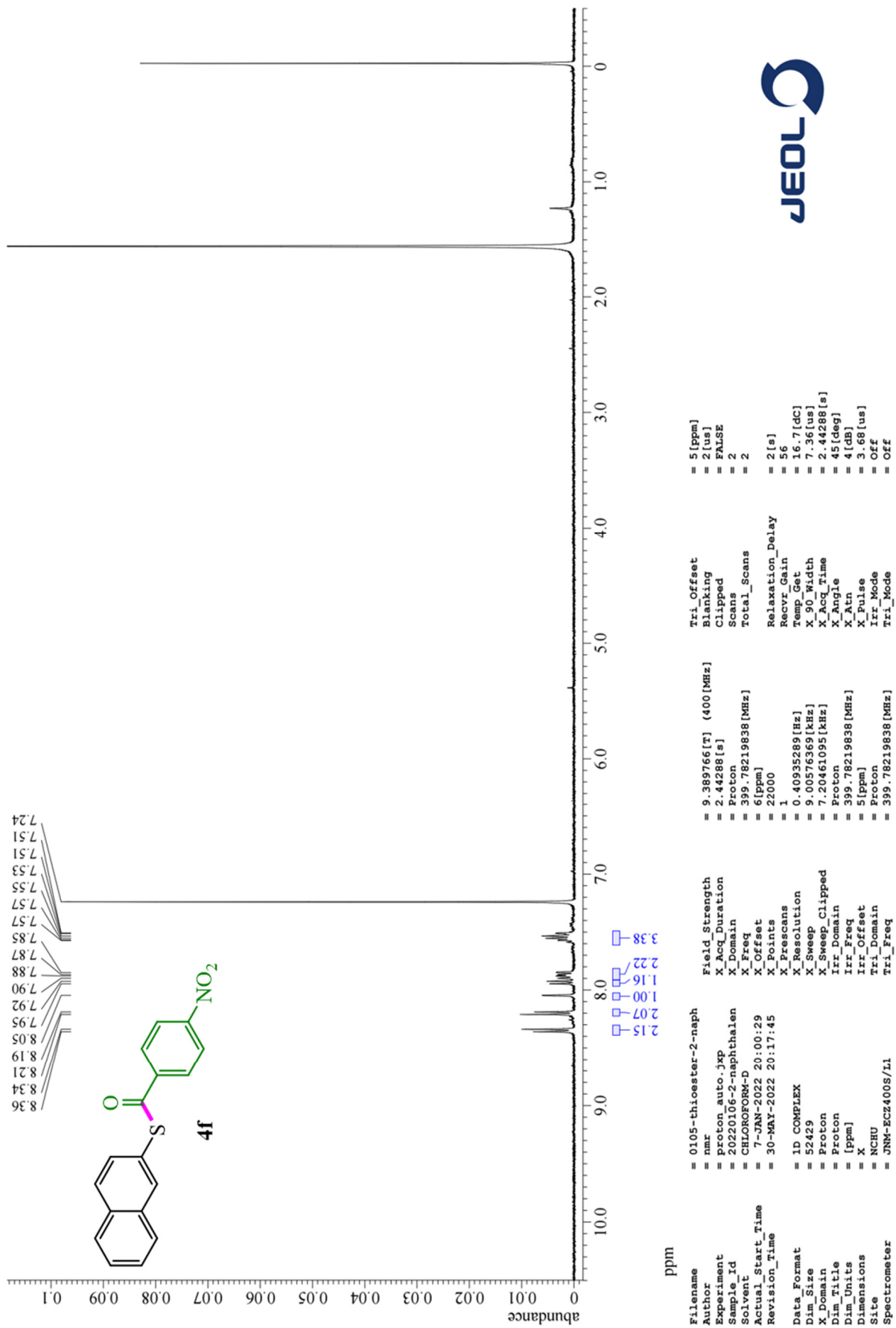
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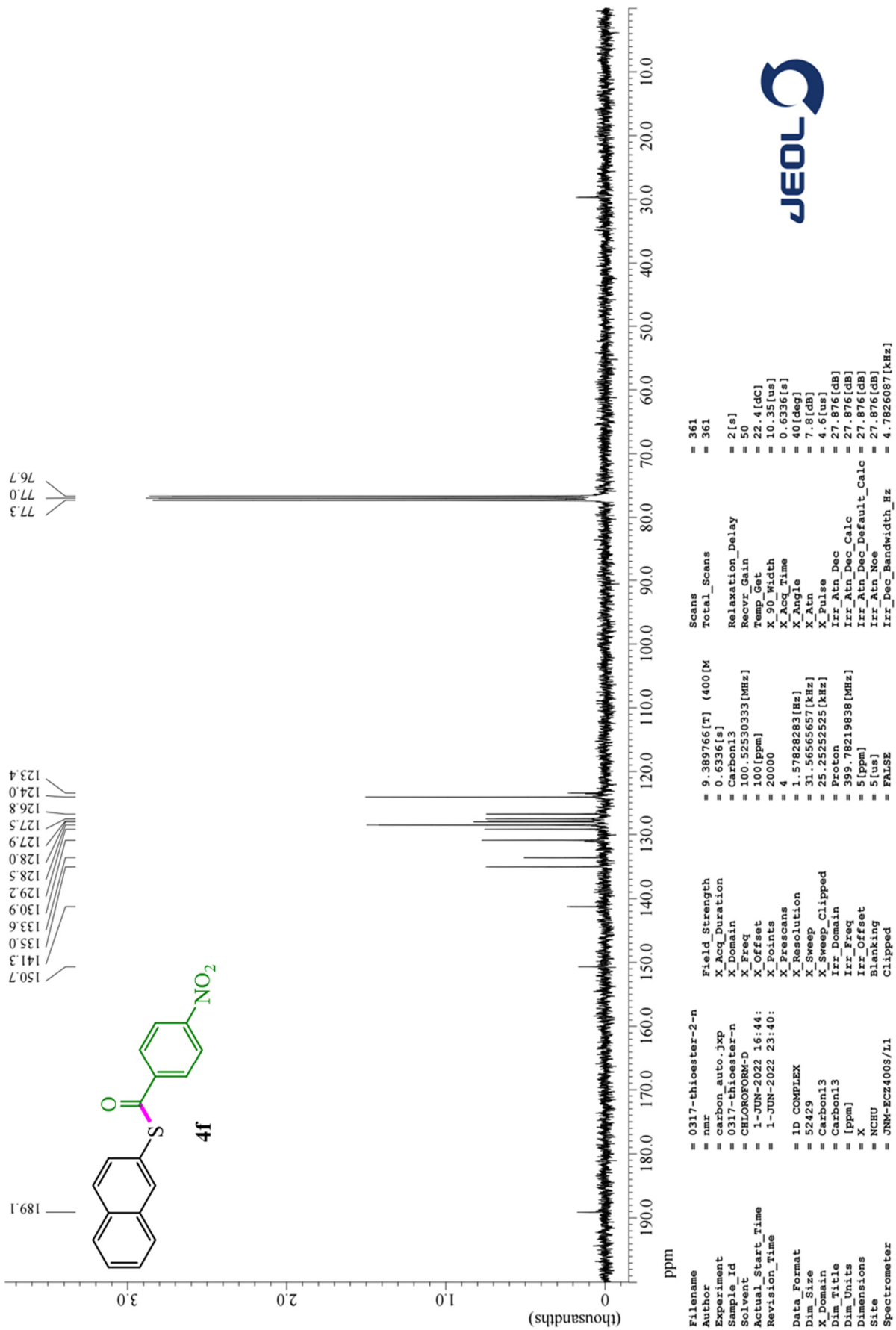
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Dimensions = X
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¹H NMR spectrum of compound **4f** (400 MHz, CDCl₃)



¹³C{H} NMR spectrum of compound **4f** (100 MHz, CDCl₃)

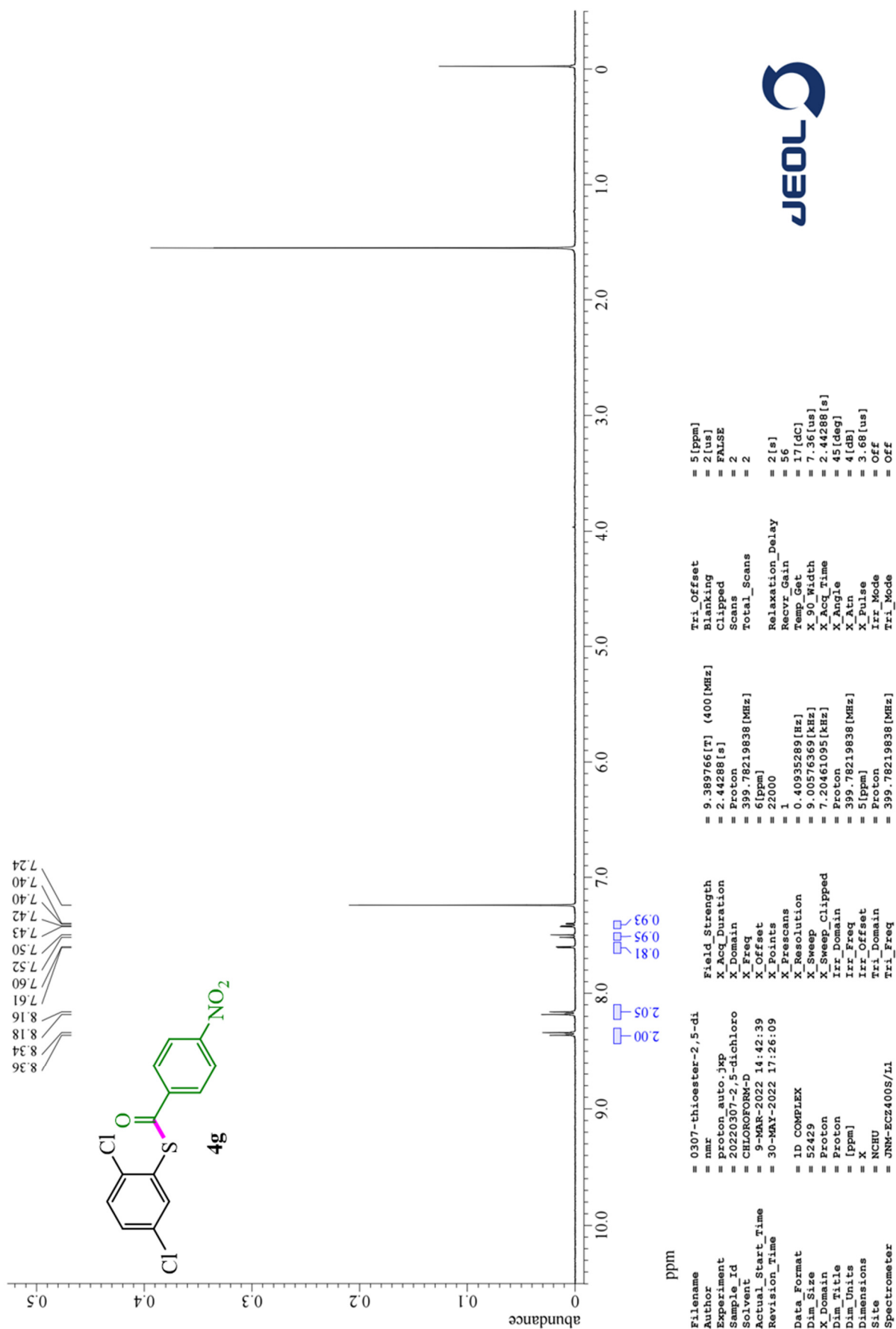
JEOL

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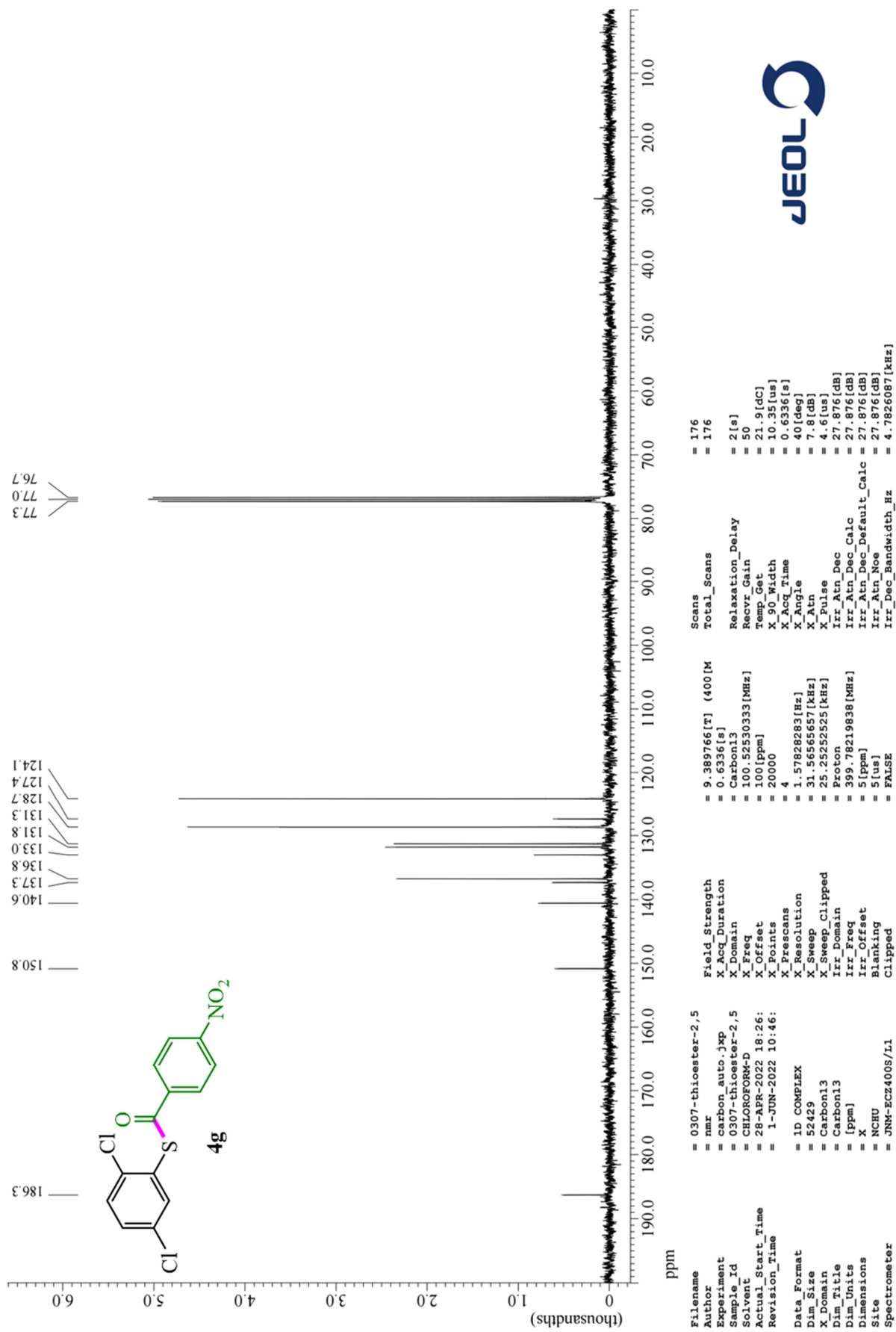
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Dimensions = X
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Spectrometer = JNM-ECZ400S/L1

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X_Points = 20000
X_Prescans = 4
X_Resolution = 1.57828283[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clippped = 25.25252525[kHz]
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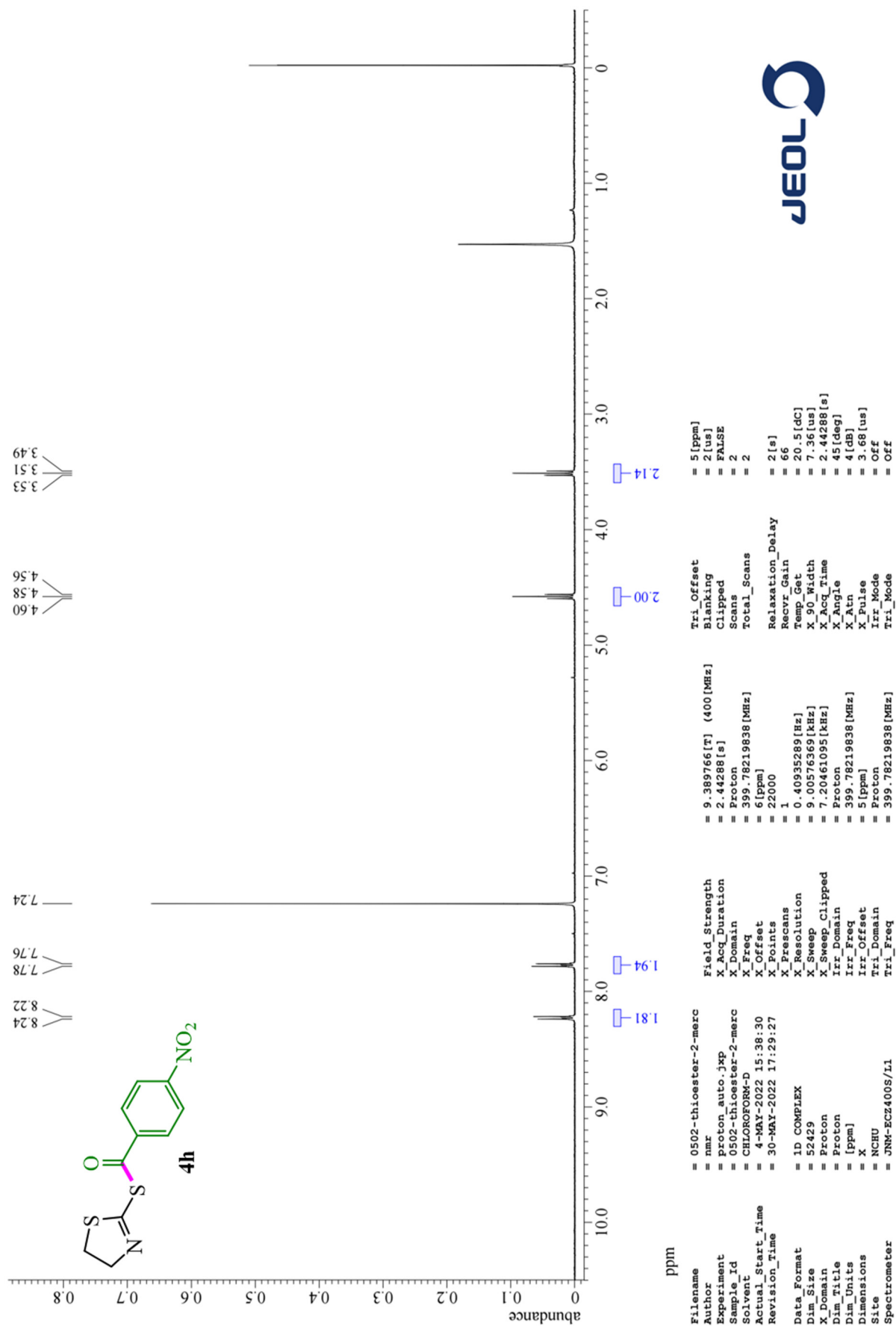
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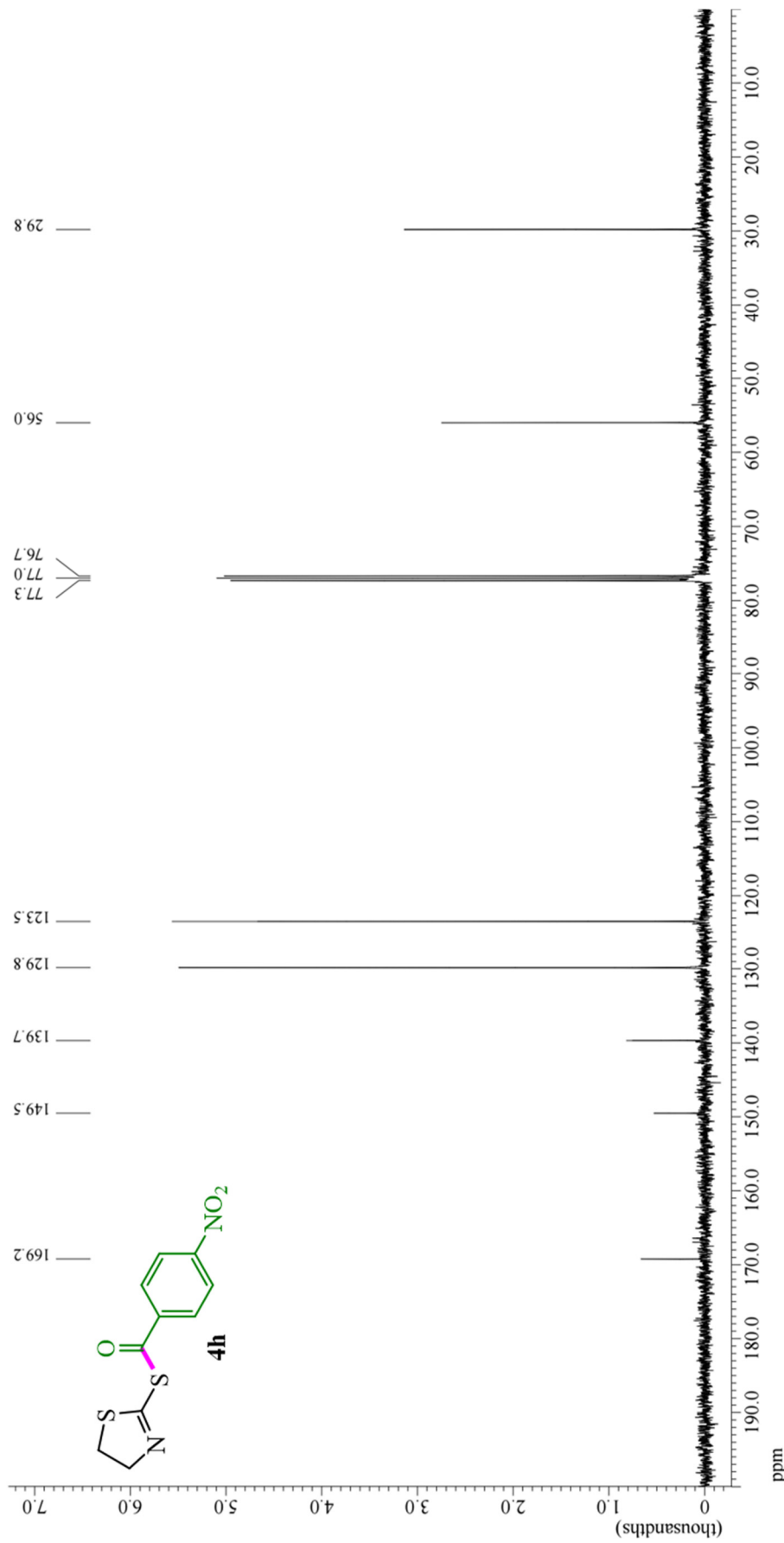
¹H NMR spectrum of compound **4g** (400 MHz, CDCl₃)



¹³C{H} NMR spectrum of compound **4g** (100 MHz, CDCl₃)



¹H NMR spectrum of compound **4h** (400 MHz, CDCl₃)



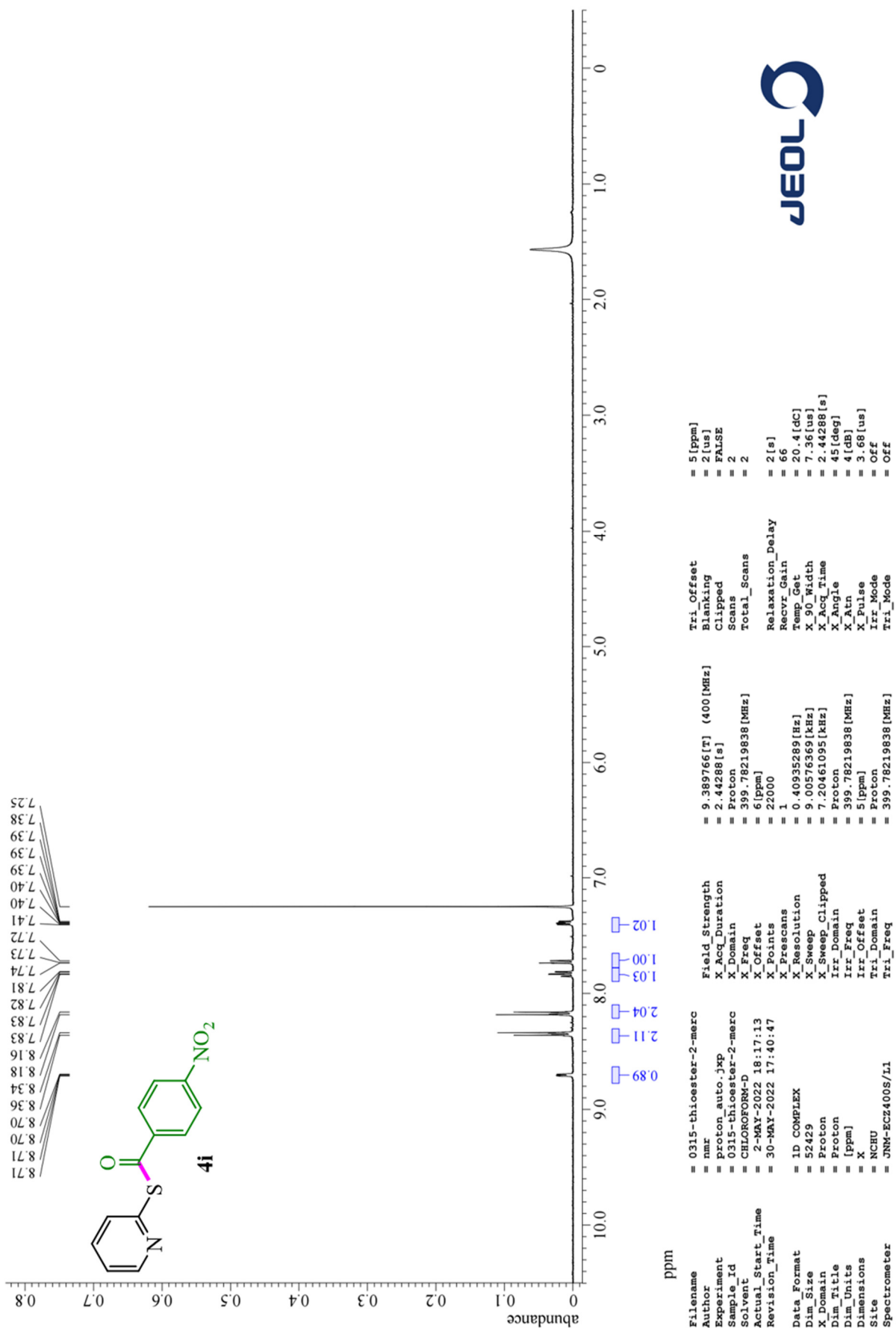
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4h** (100 MHz, CDCl_3)

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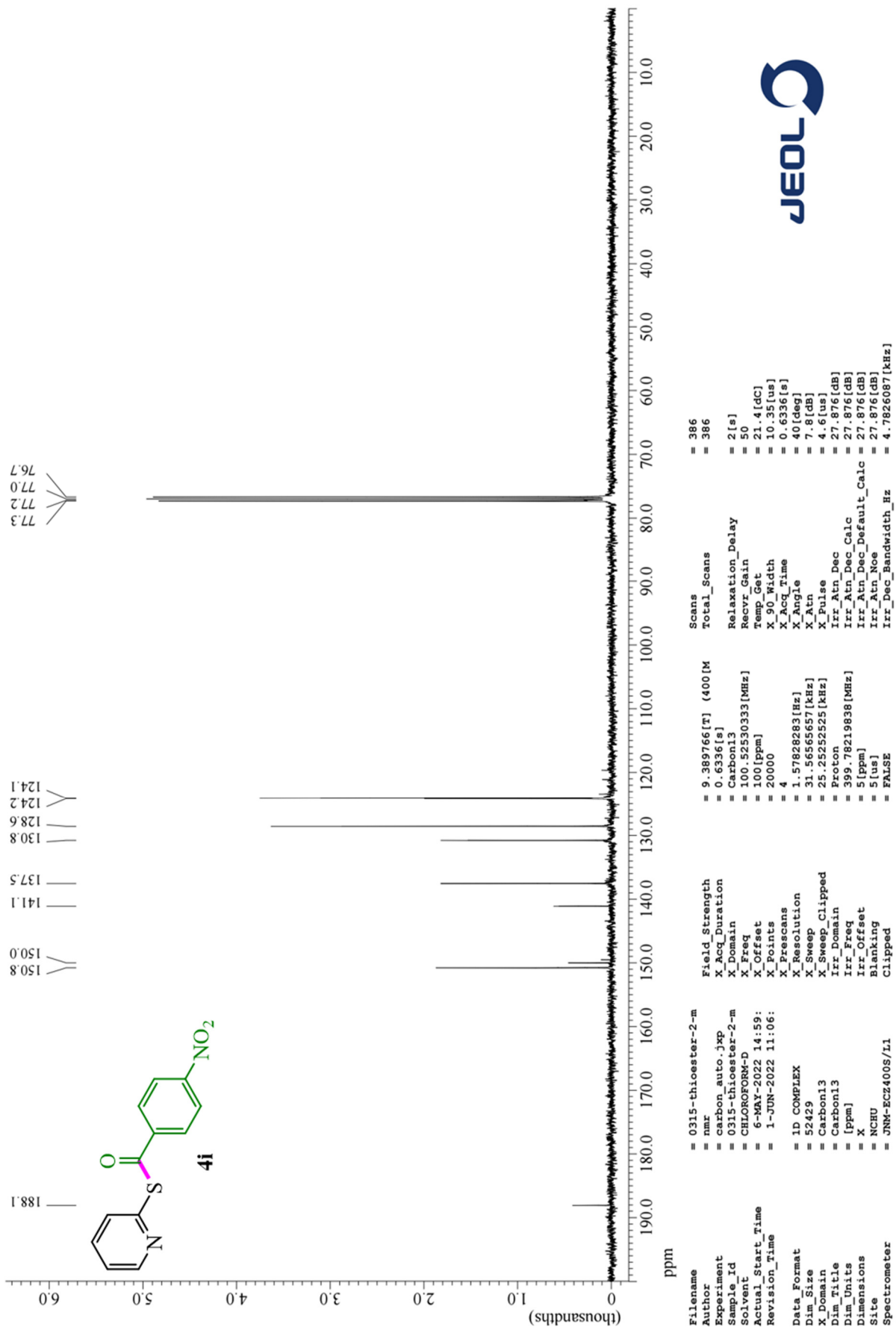
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 X_Sweep = 31.56565657[kHz]
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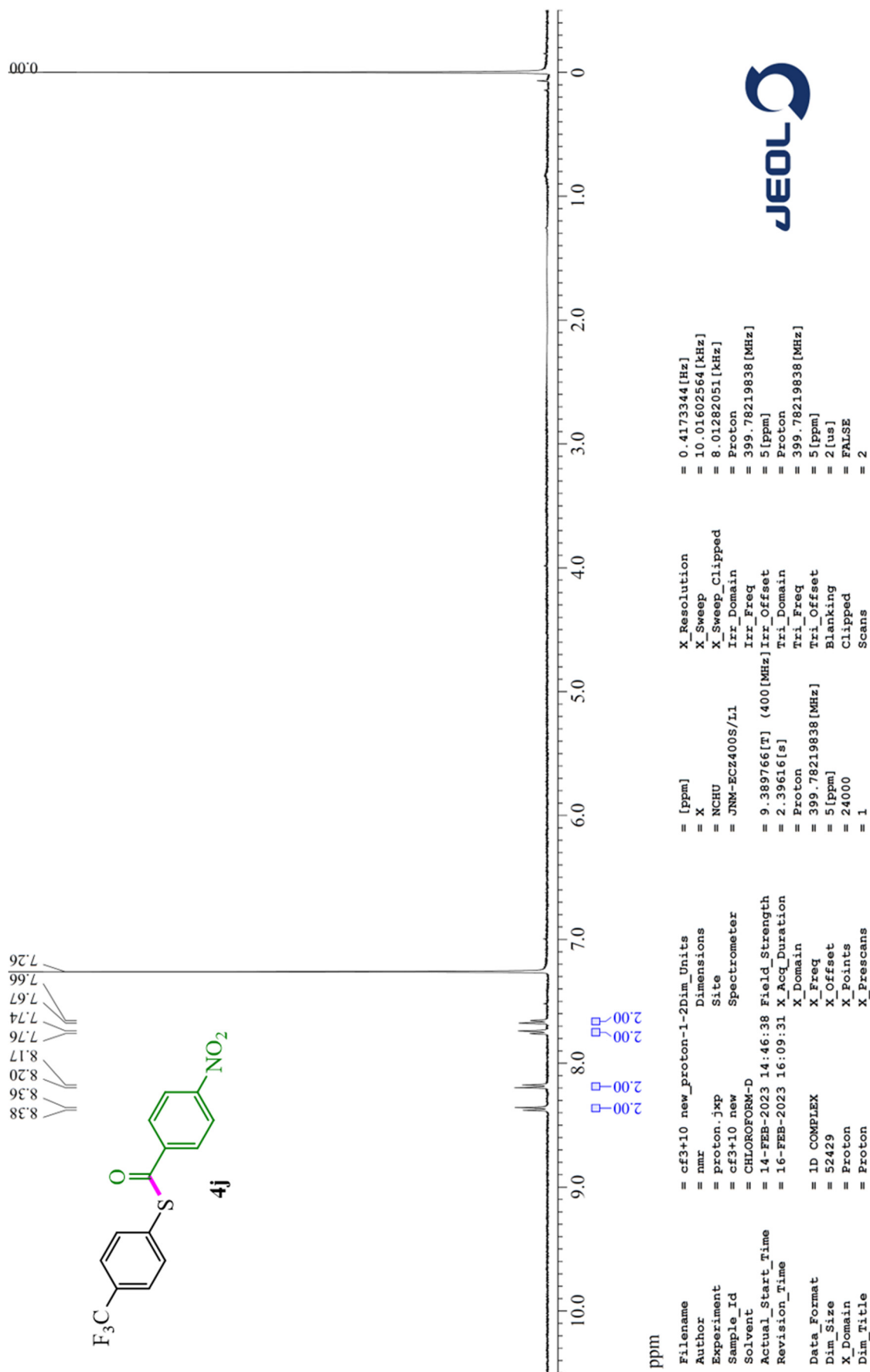




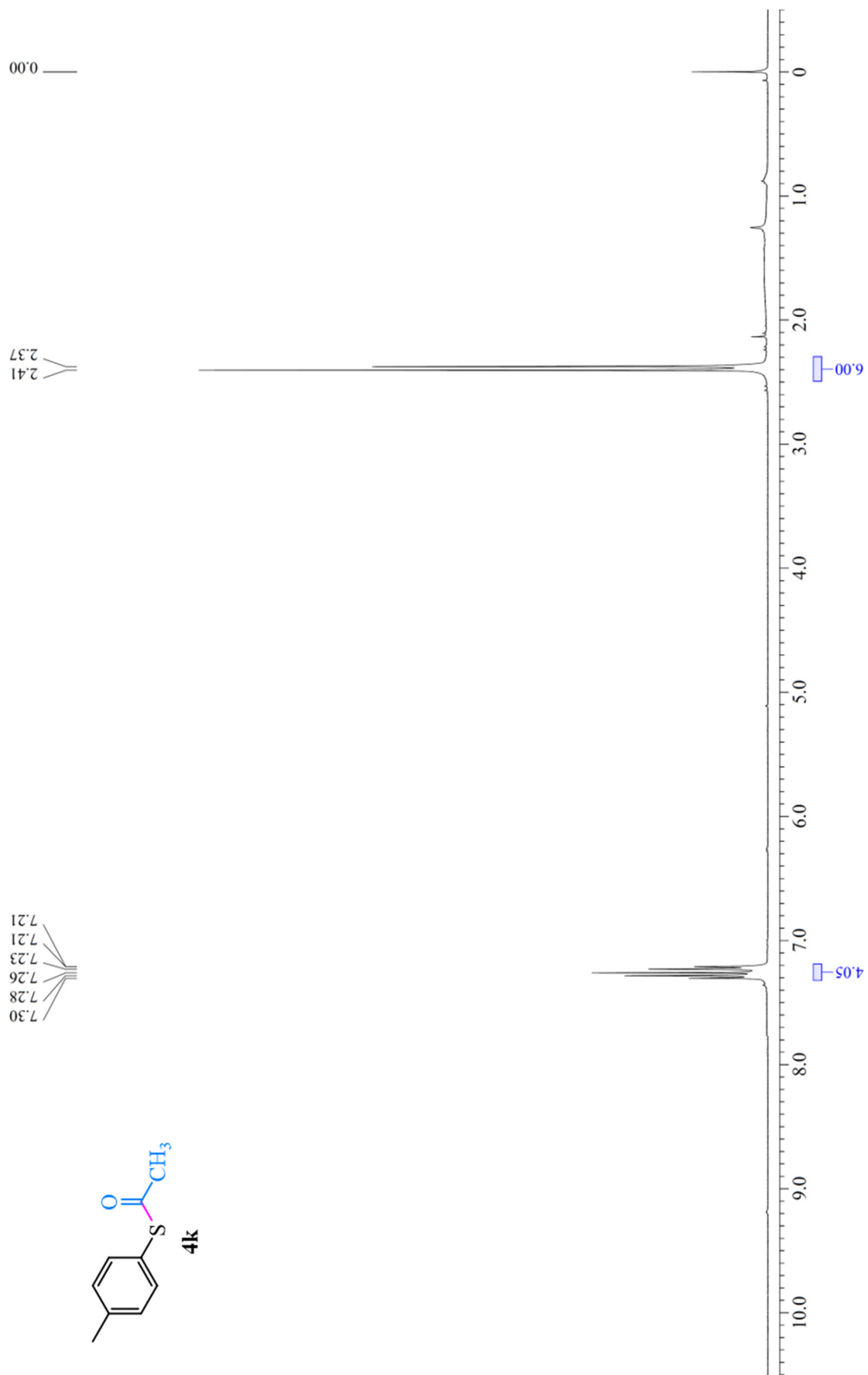
¹H NMR spectrum of compound **4i** (400 MHz, CDCl₃)



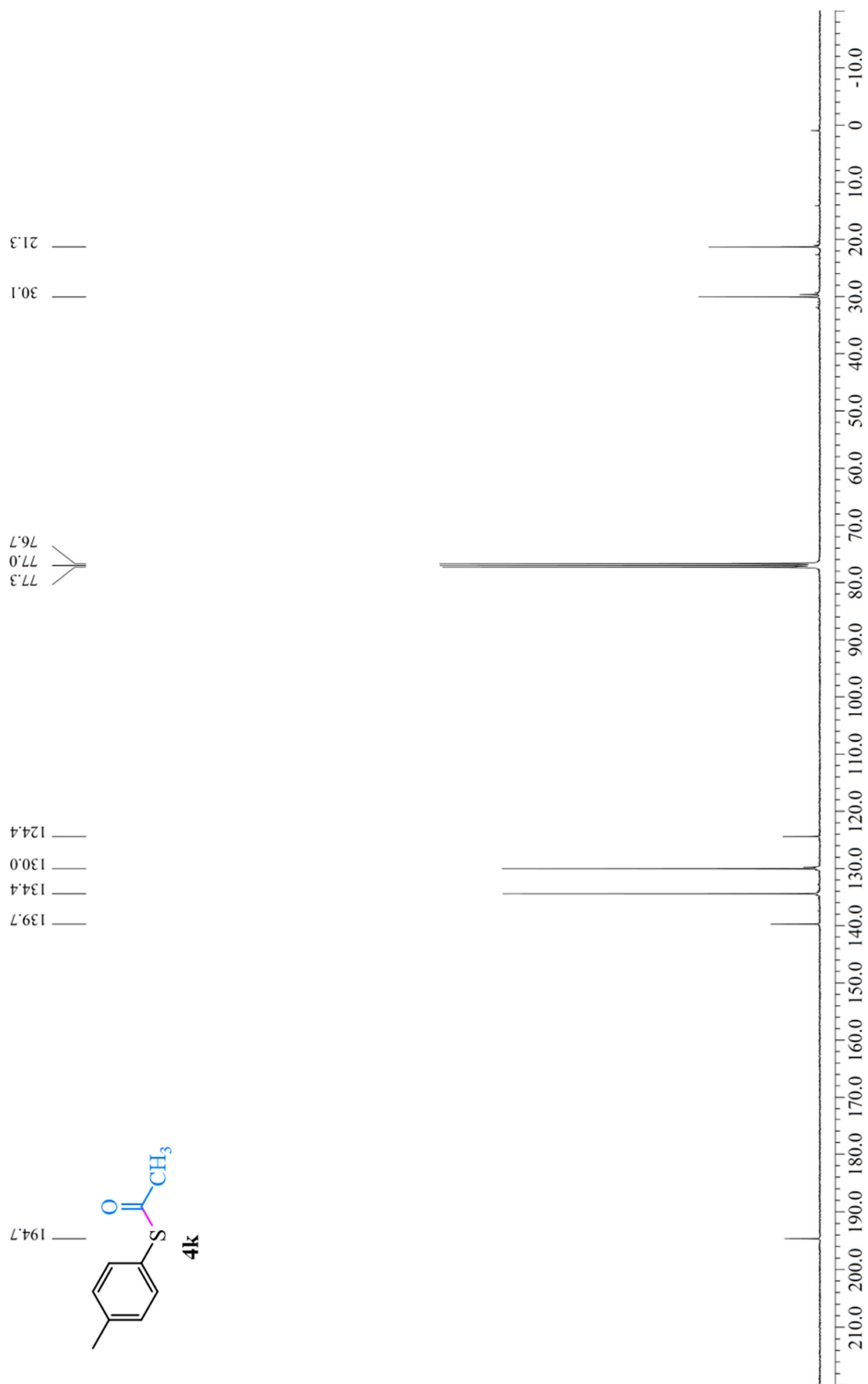
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4i** (100 MHz, CDCl_3)



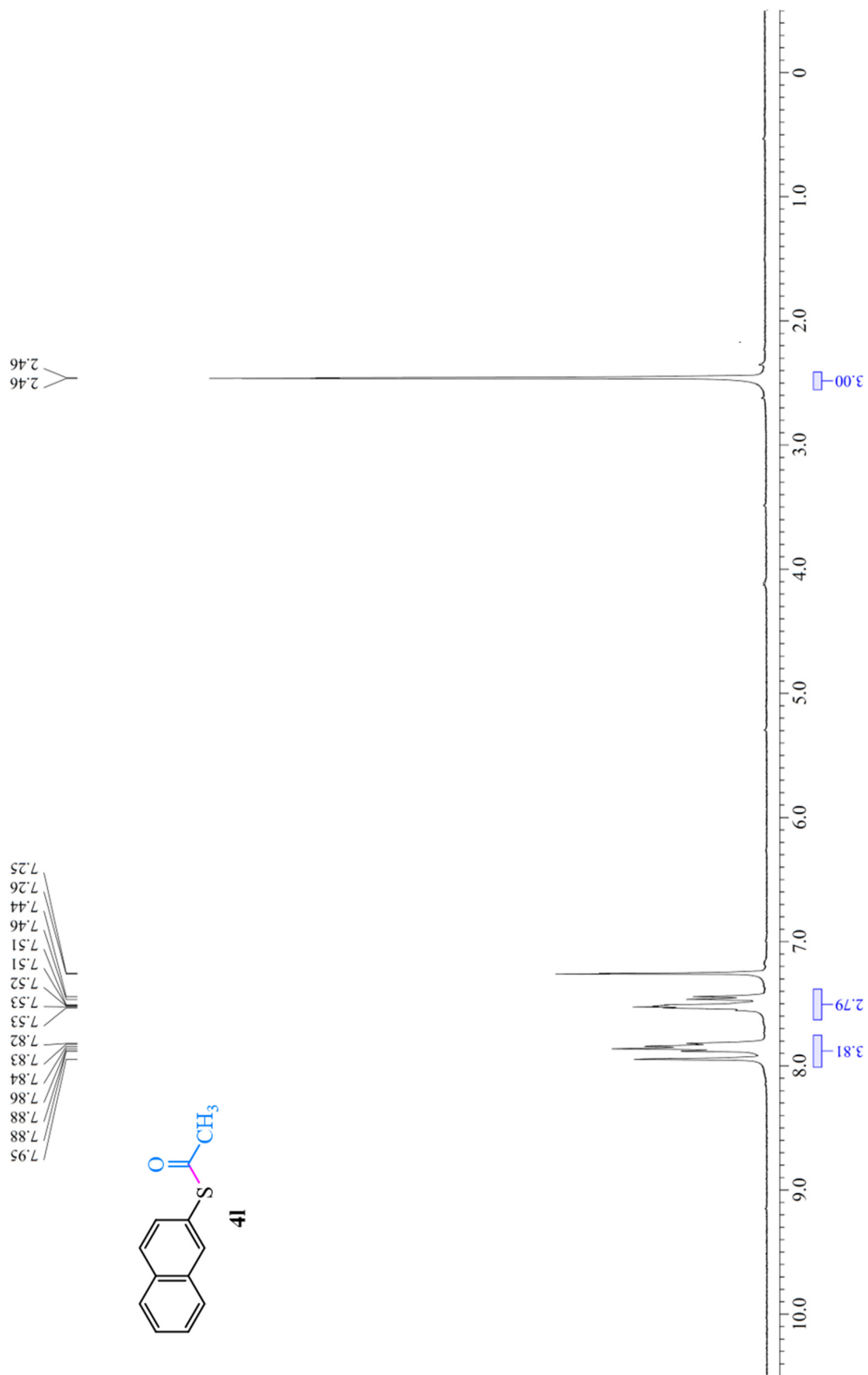
¹H NMR spectrum of compound **4j** (400 MHz, CDCl₃)



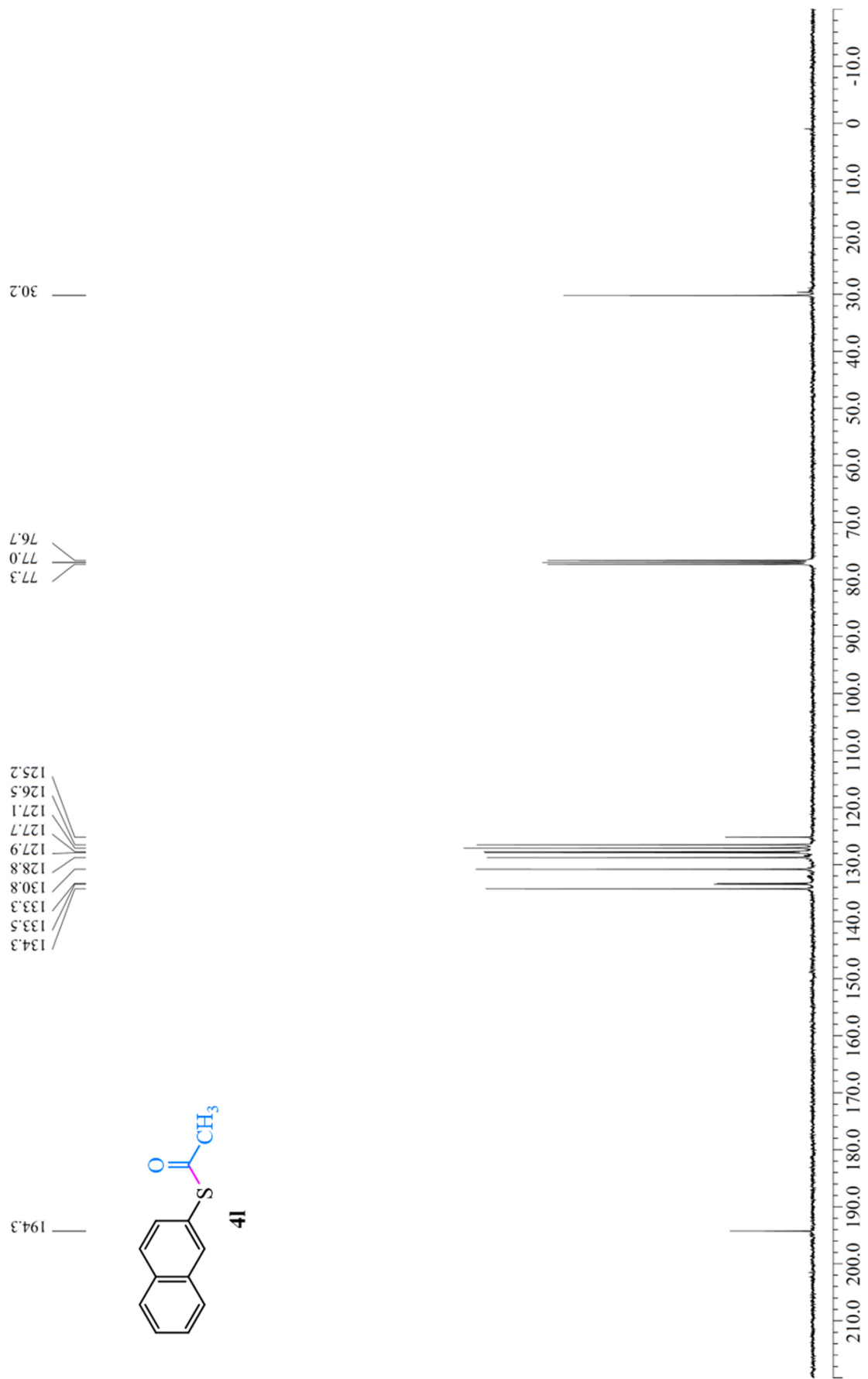
^1H NMR spectrum of compound **4k** (400 MHz, CDCl_3)



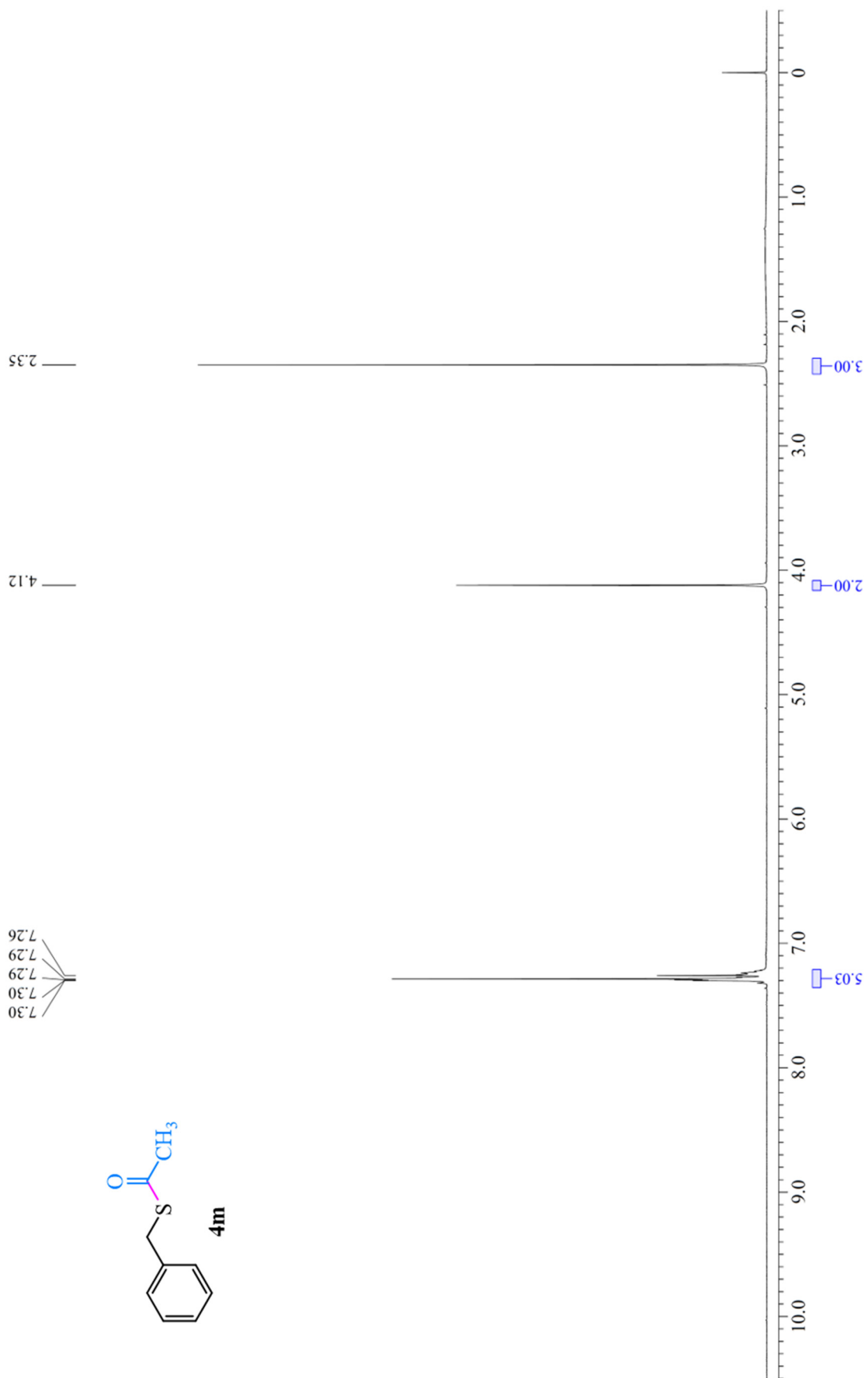
¹³C {H} NMR spectrum of compound **4k** (100 MHz, CDCl₃)



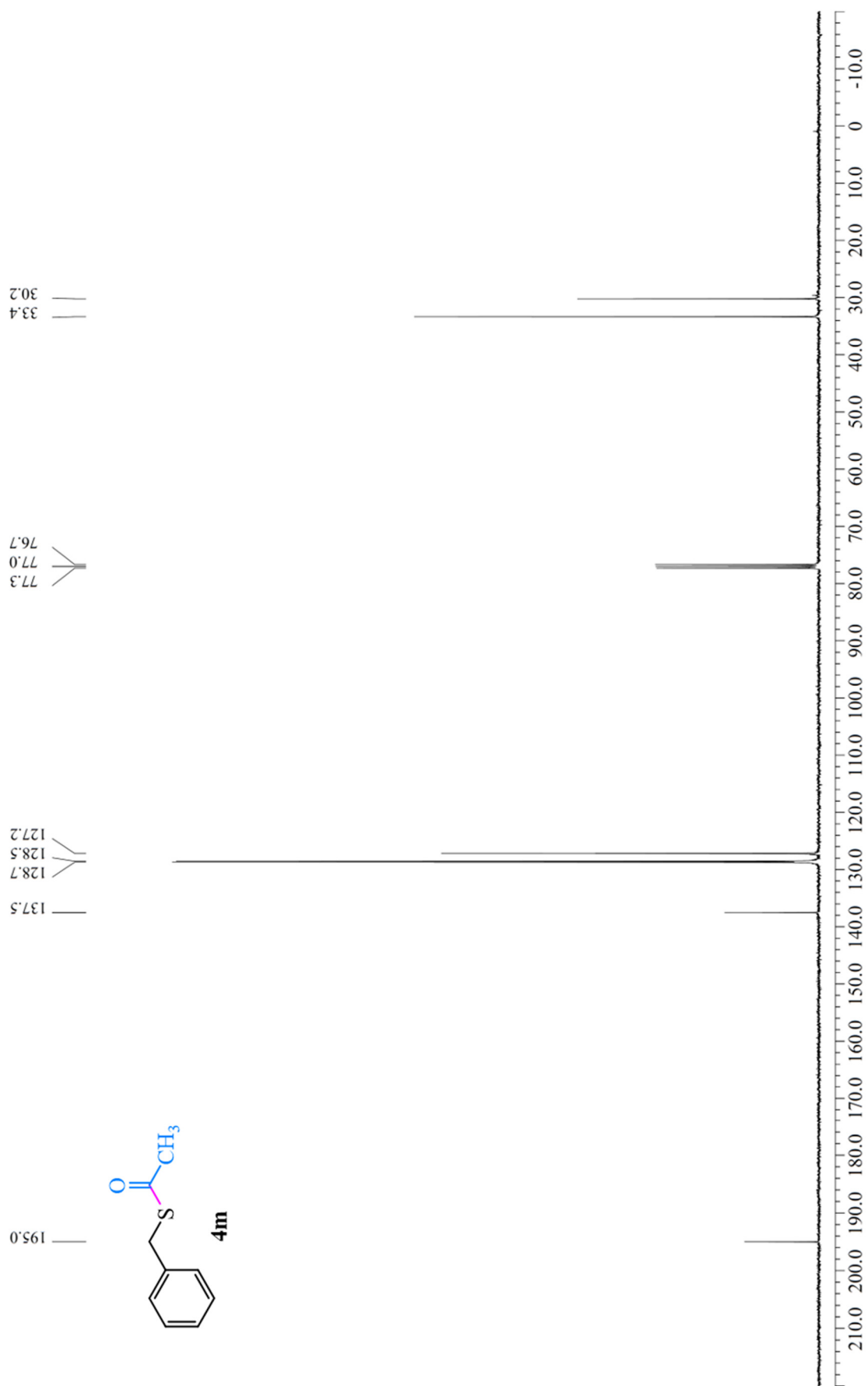
¹H NMR spectrum of compound **4I** (400 MHz, CDCl₃)



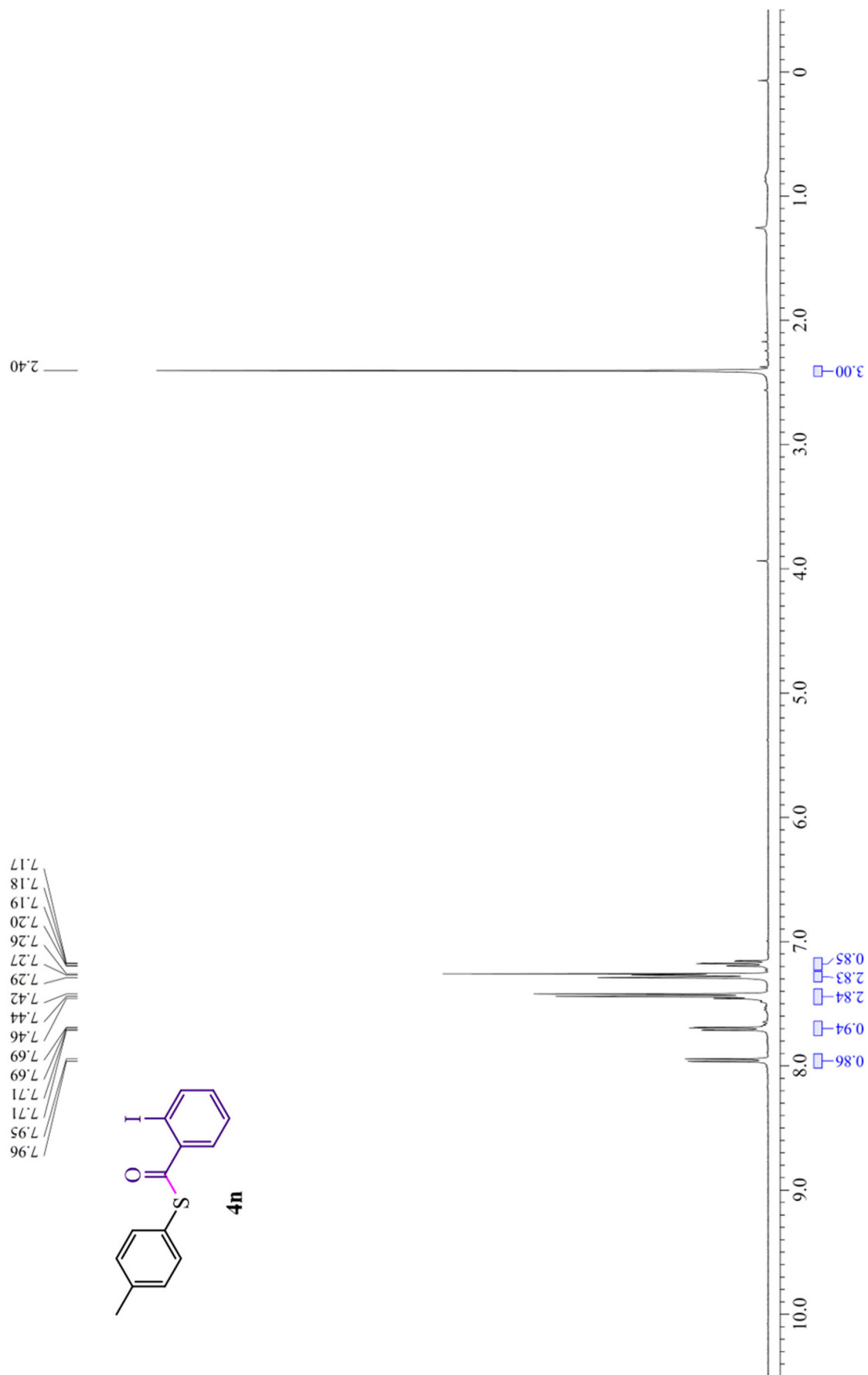
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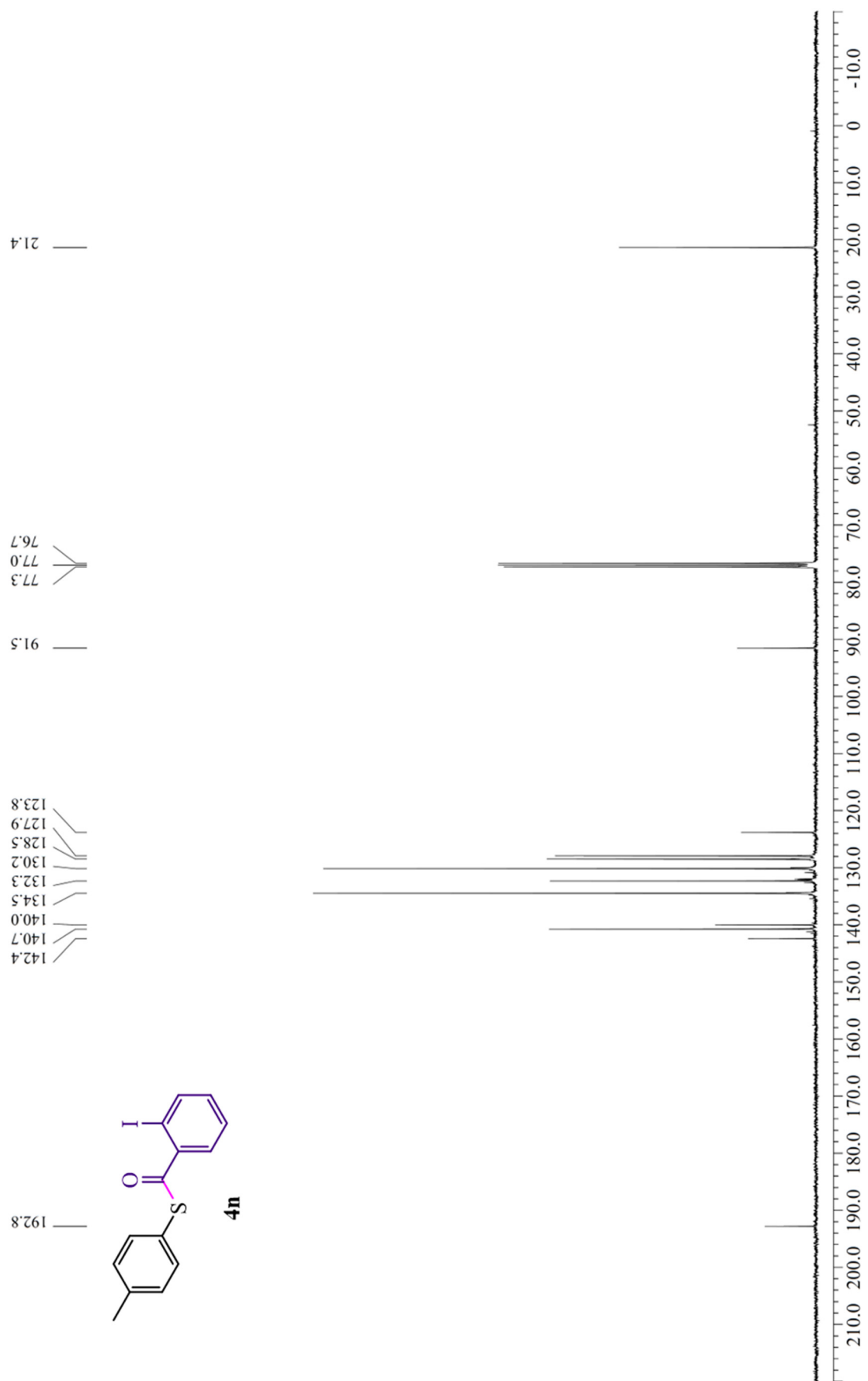
^1H NMR spectrum of compound **4m** (400 MHz, CDCl_3)



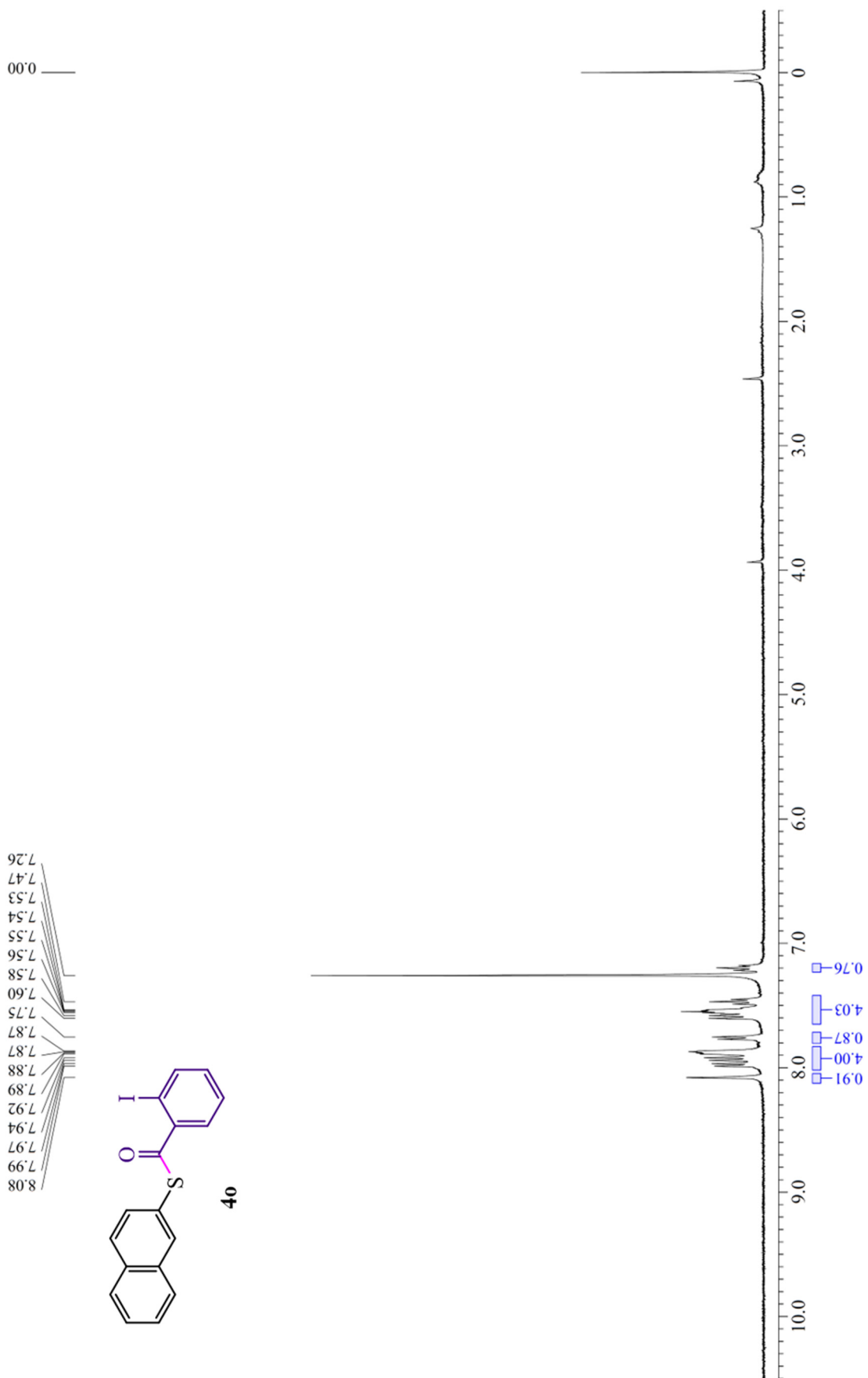
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4m** (100 MHz, CDCl₃)



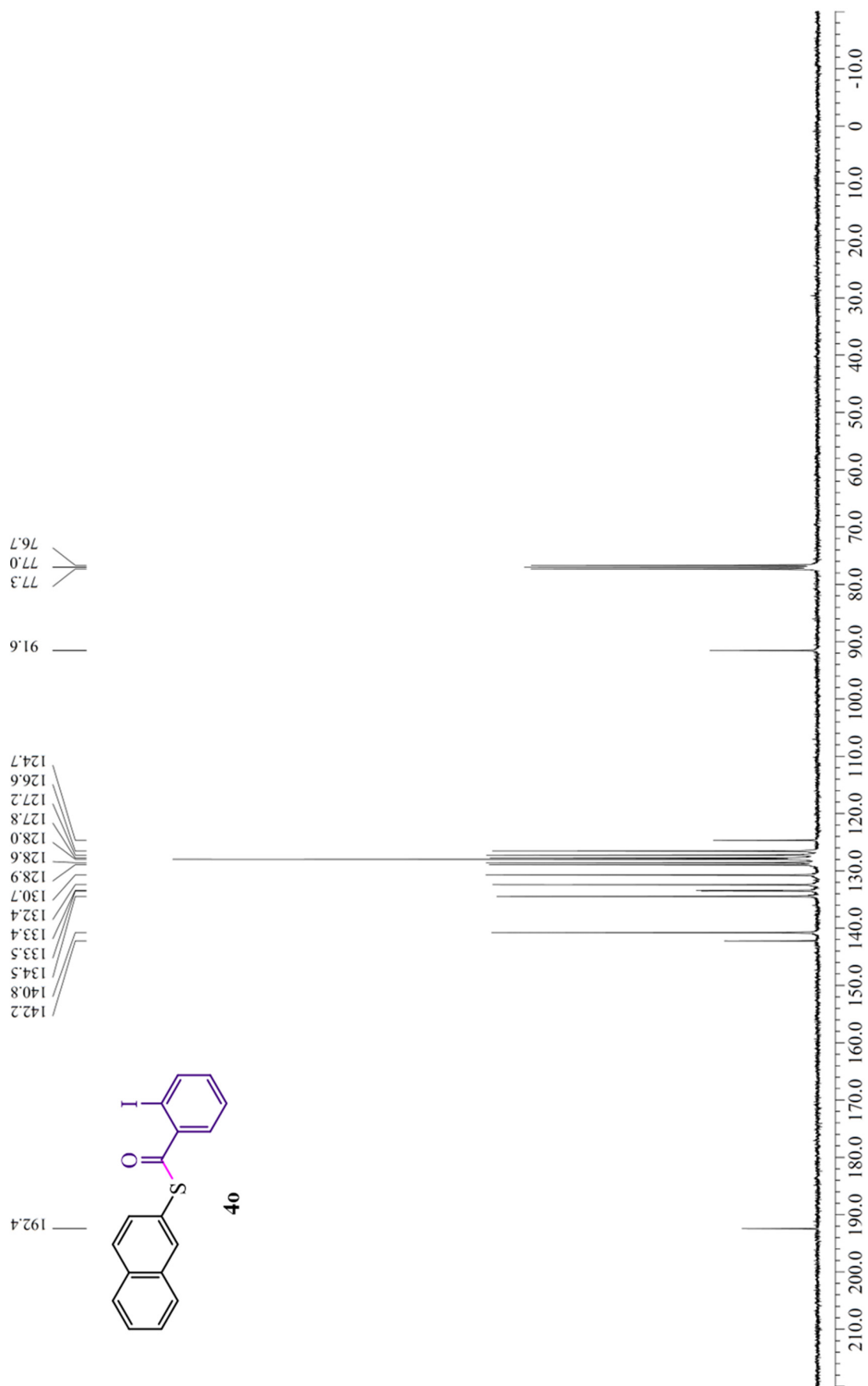
¹H NMR spectrum of compound **4n** (400 MHz, CDCl₃)



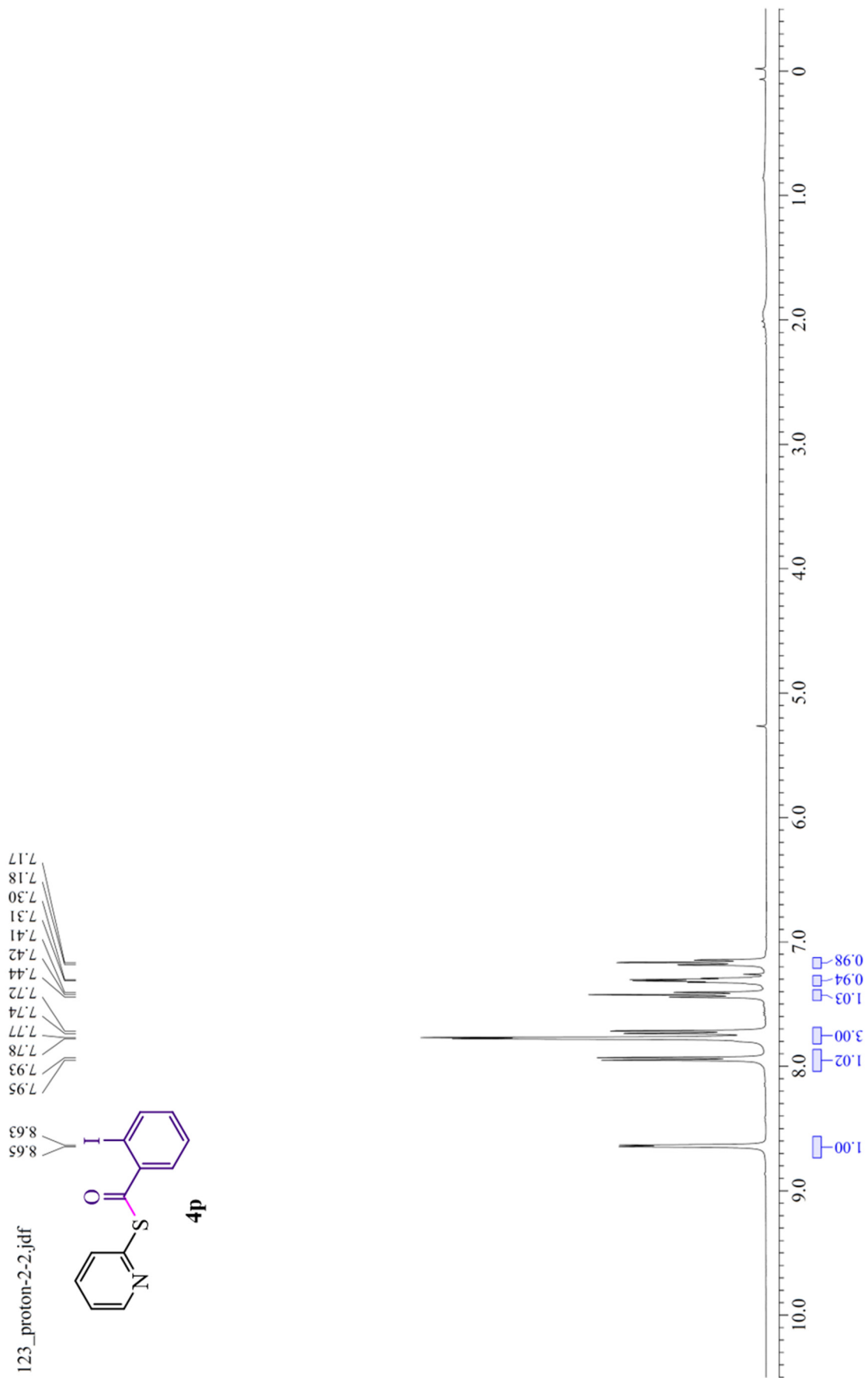
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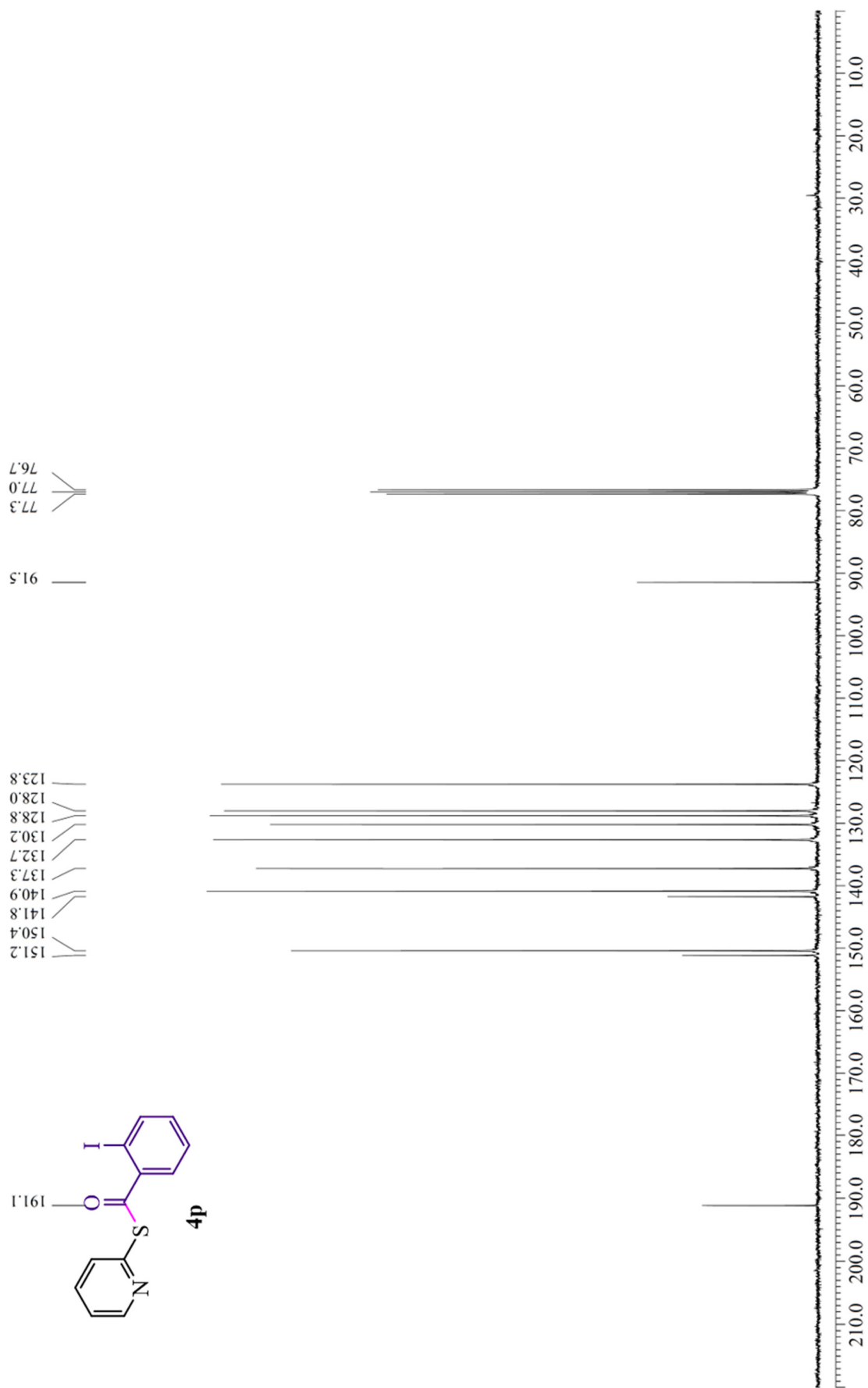
^1H NMR spectrum of compound **4o** (400 MHz, CDCl_3)



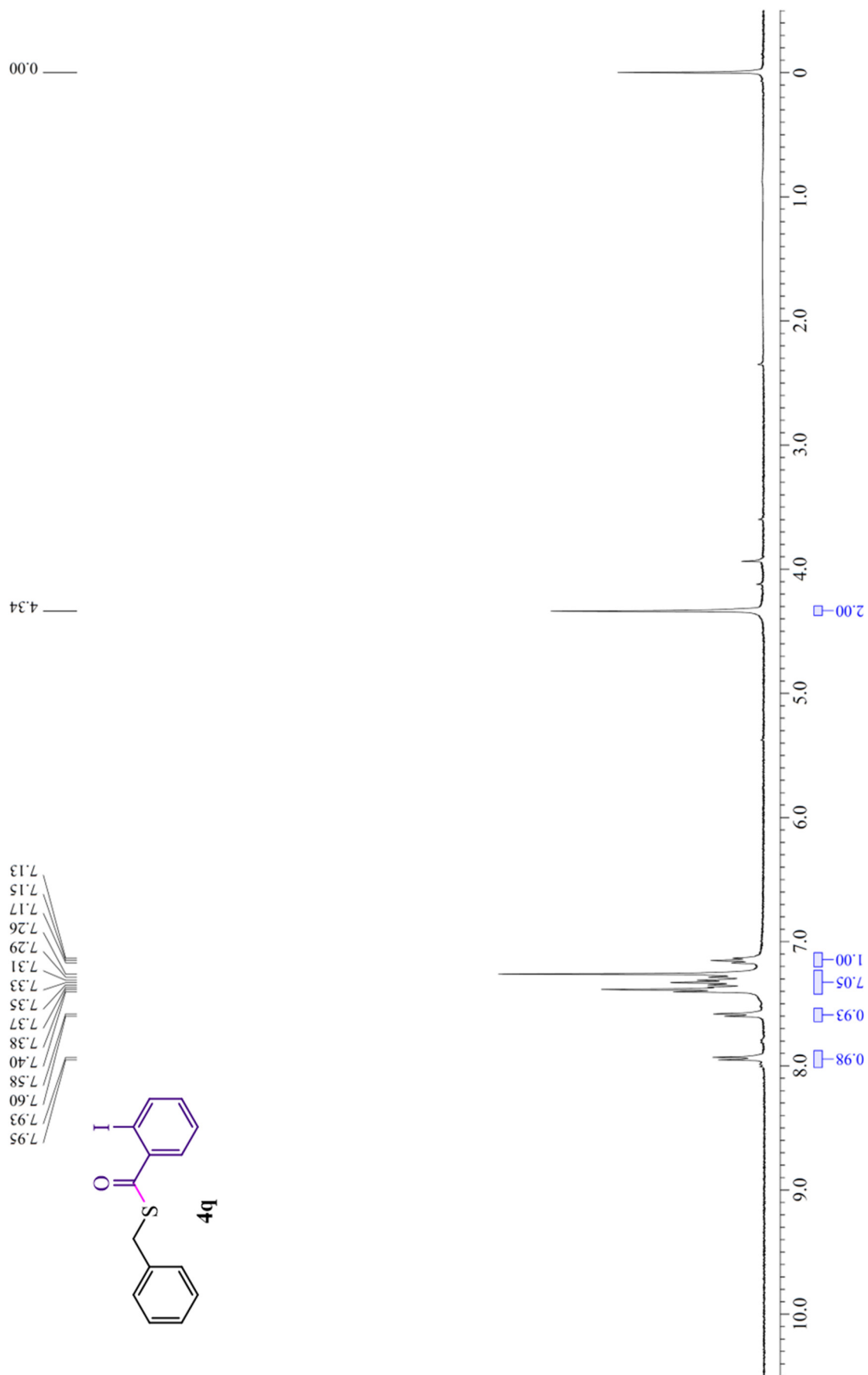
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4o** (100 MHz, CDCl_3)



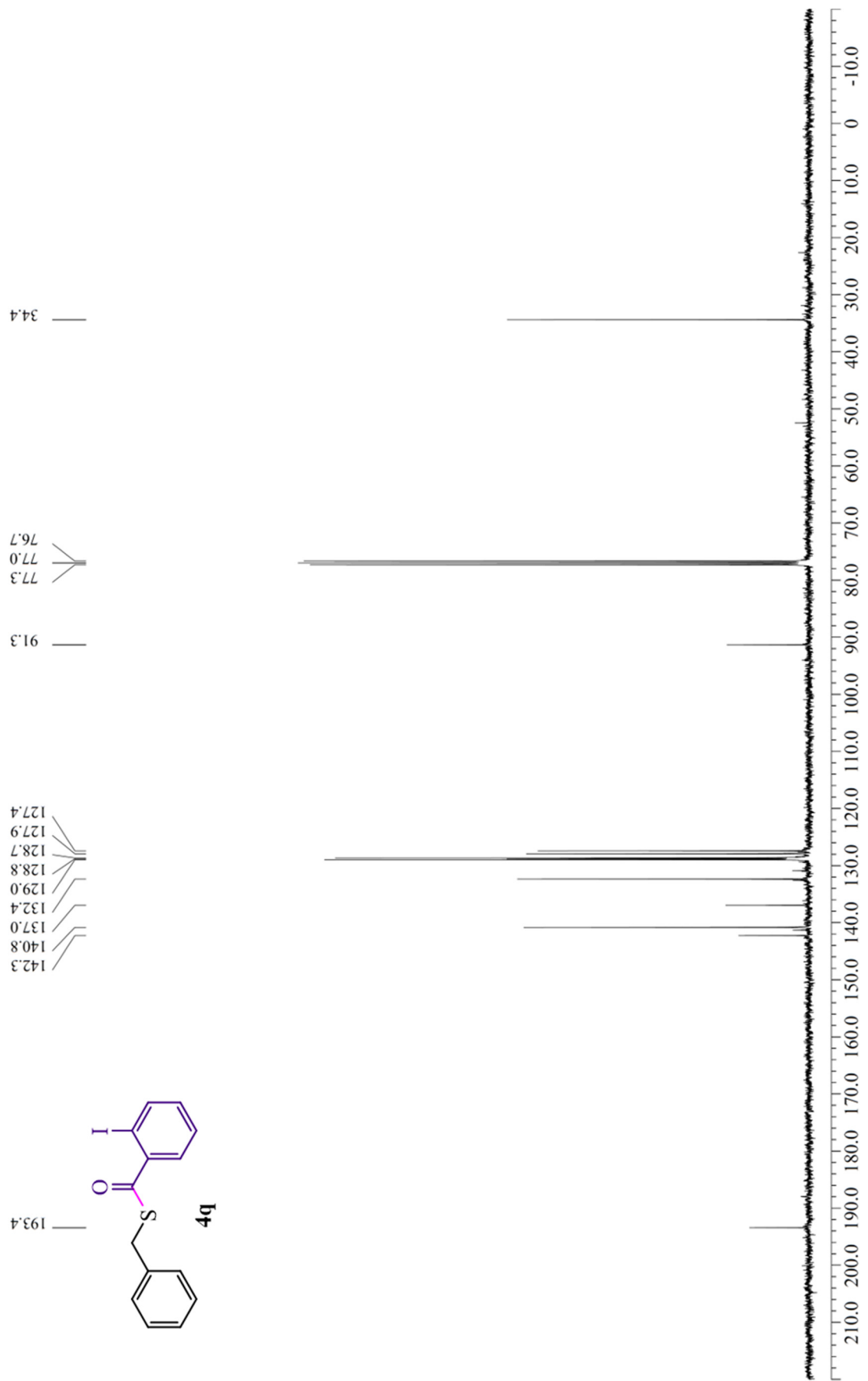
¹H NMR spectrum of compound **4p** (400 MHz, CDCl₃)



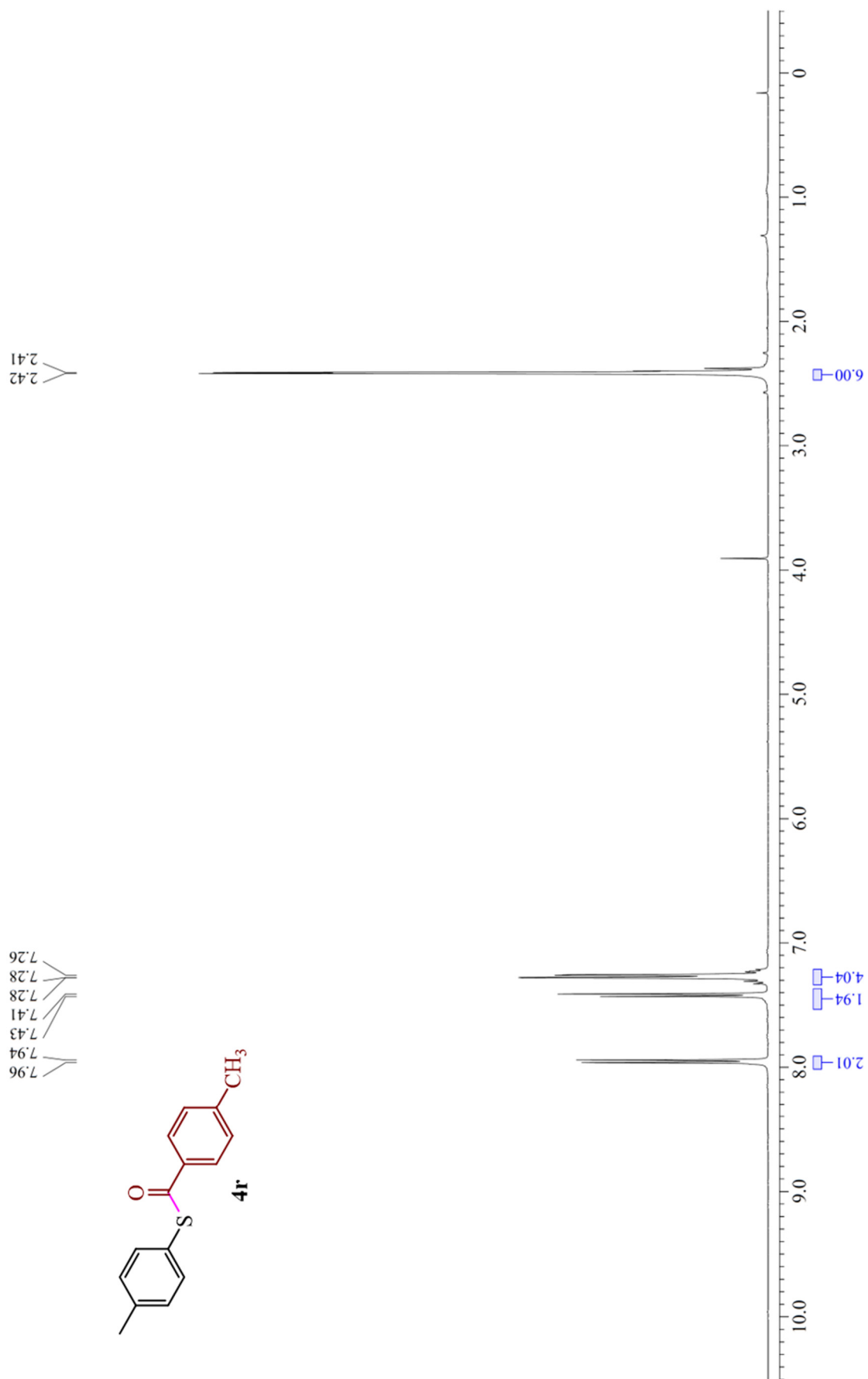
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4p** (100 MHz, CDCl_3)



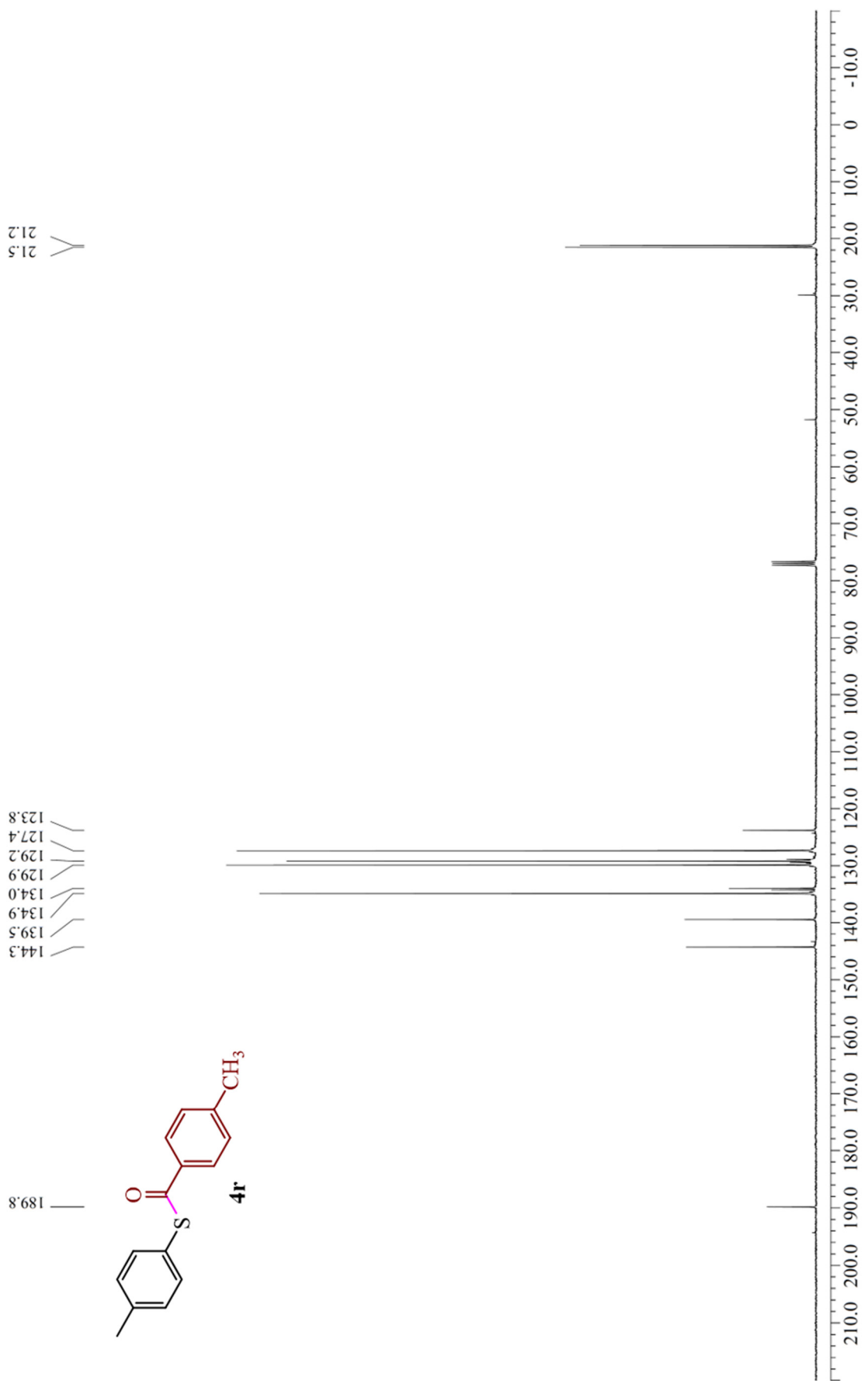
¹H NMR spectrum of compound **4q** (400 MHz, CDCl₃)



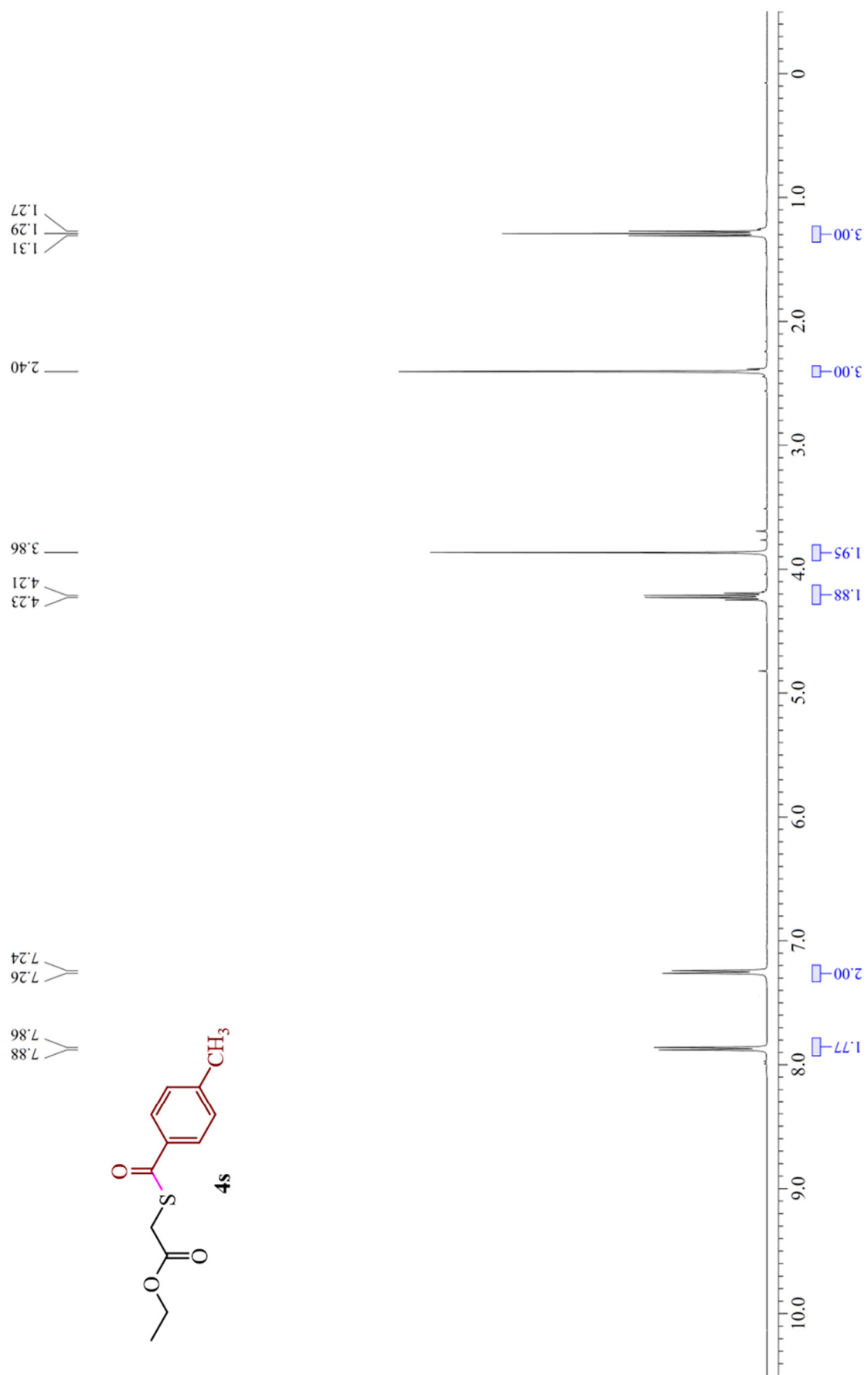
^{13}C NMR spectrum of compound **4q** (100 MHz, CDCl_3)



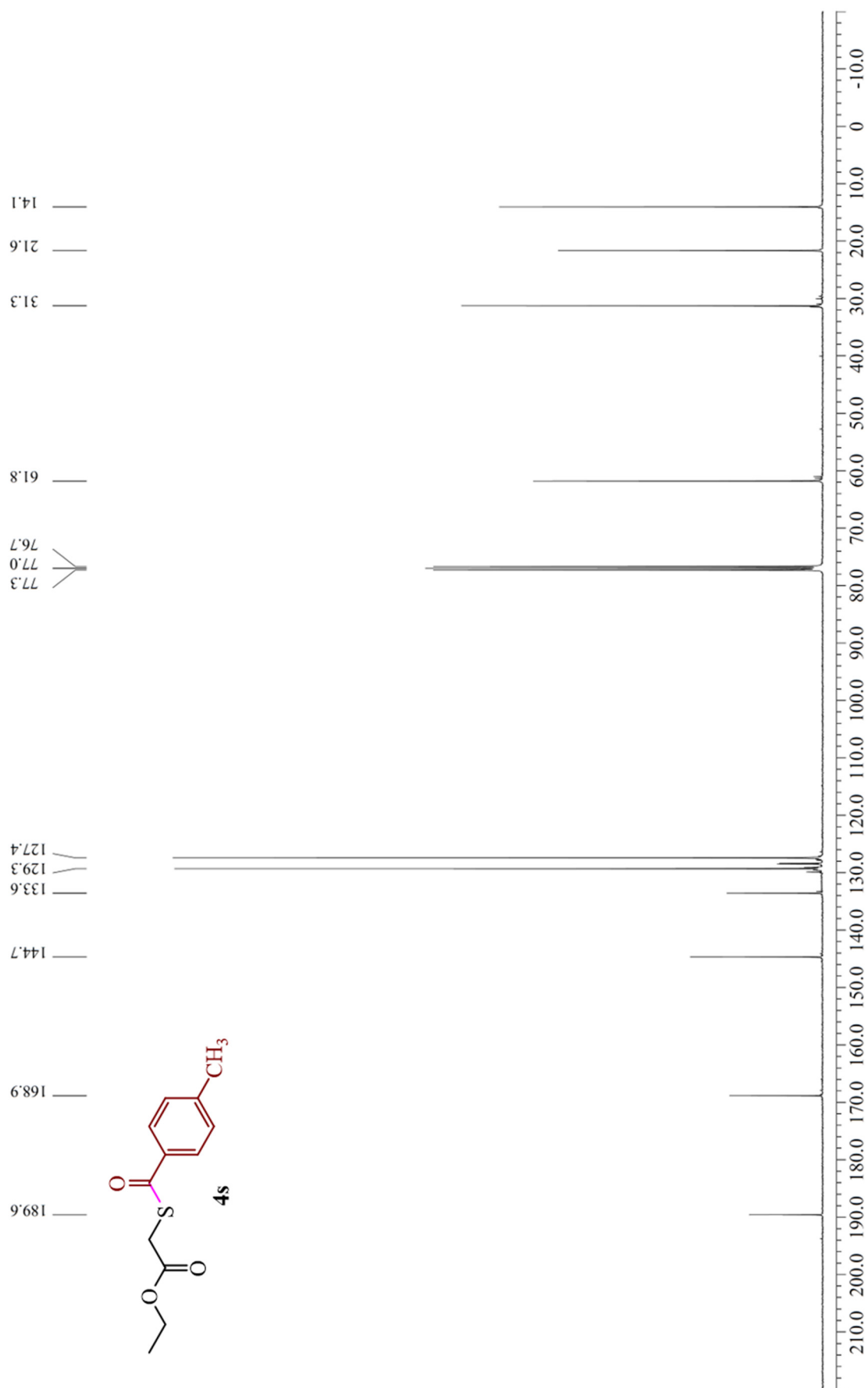
¹H NMR spectrum of compound **4r** (400 MHz, CDCl₃)



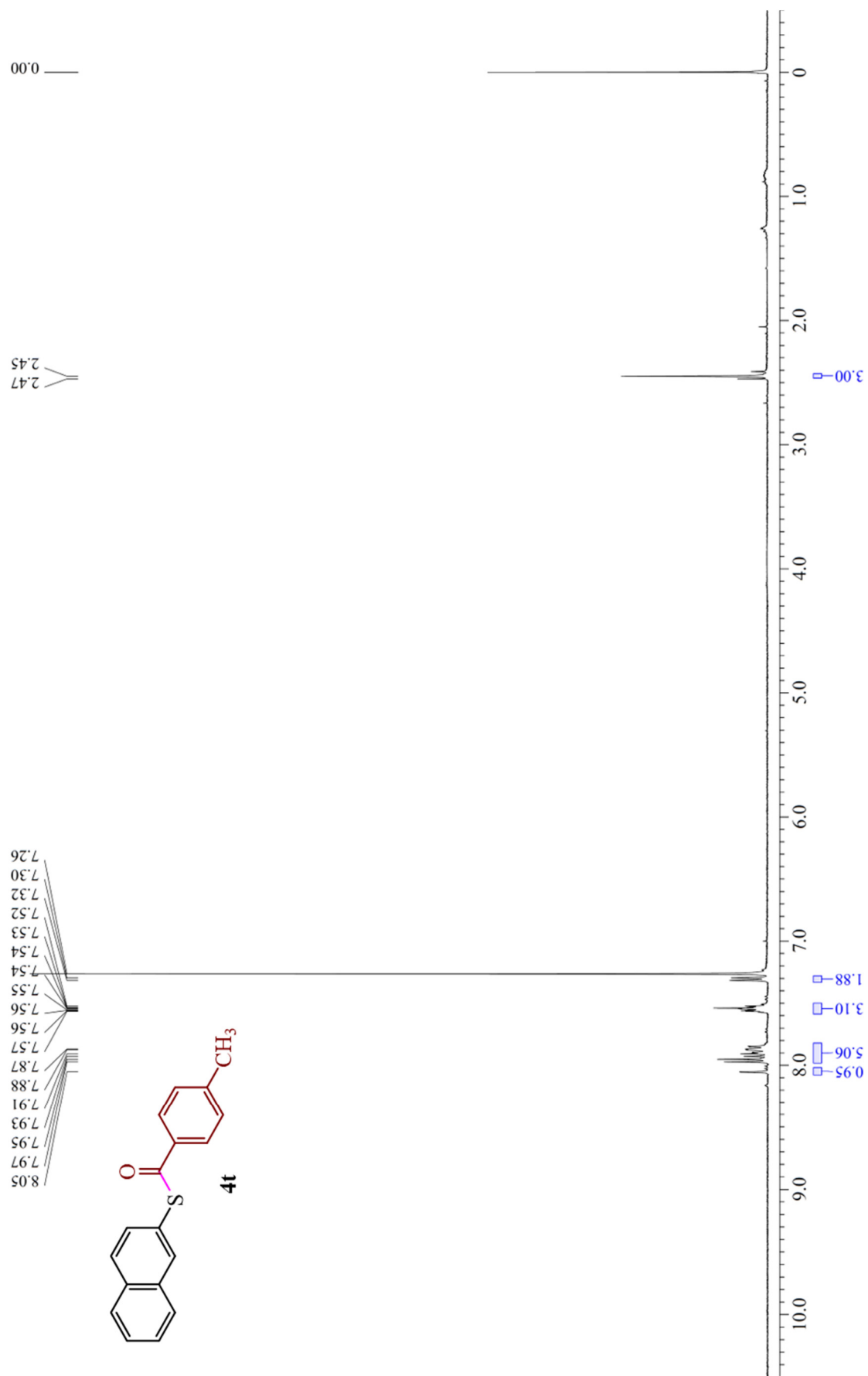
¹³C{H} NMR spectrum of compound **4r** (100 MHz, CDCl₃)



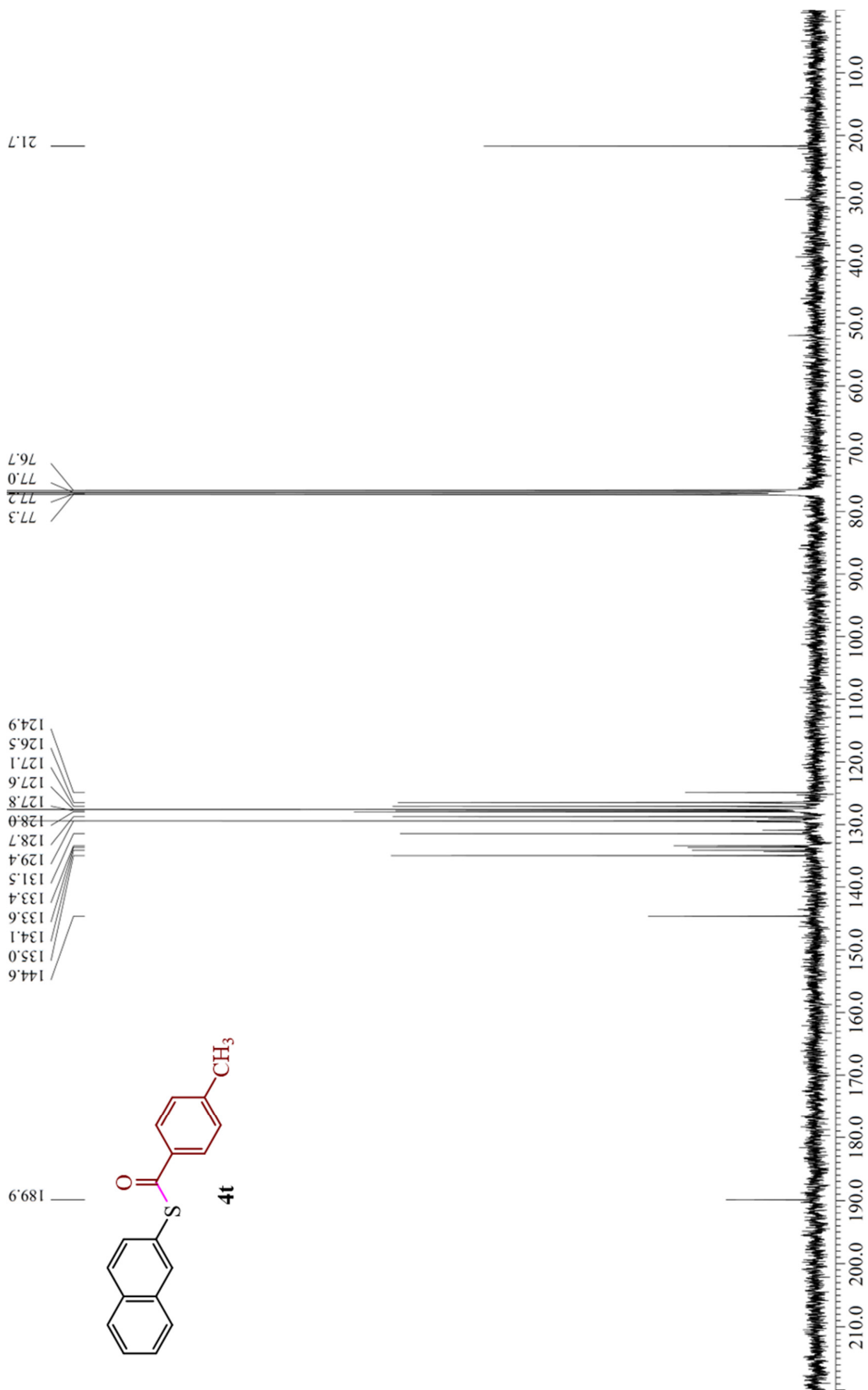
¹H NMR spectrum of compound **4s** (400 MHz, CDCl₃)



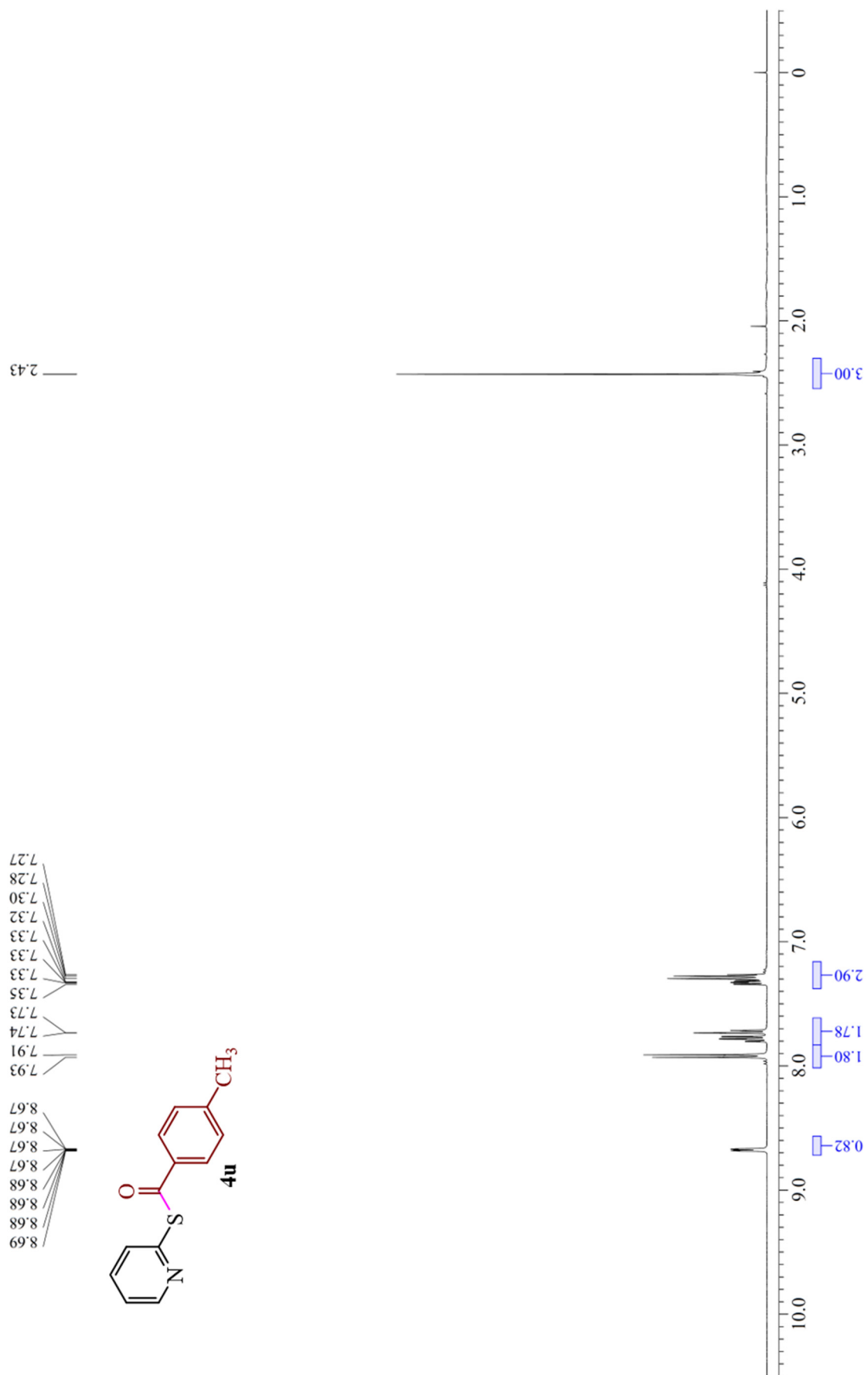
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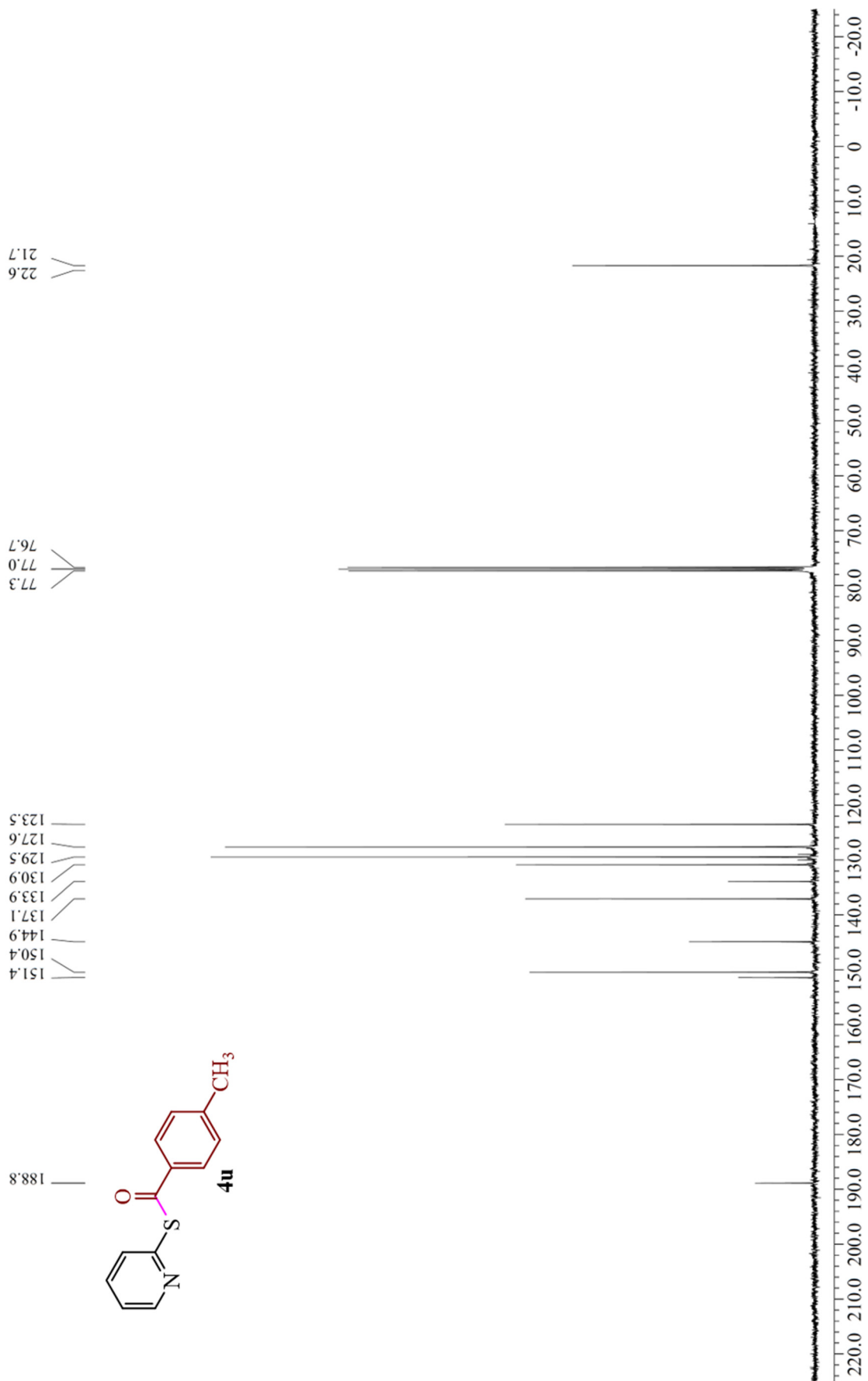
^1H NMR spectrum of compound **4t** (400 MHz, CDCl_3)



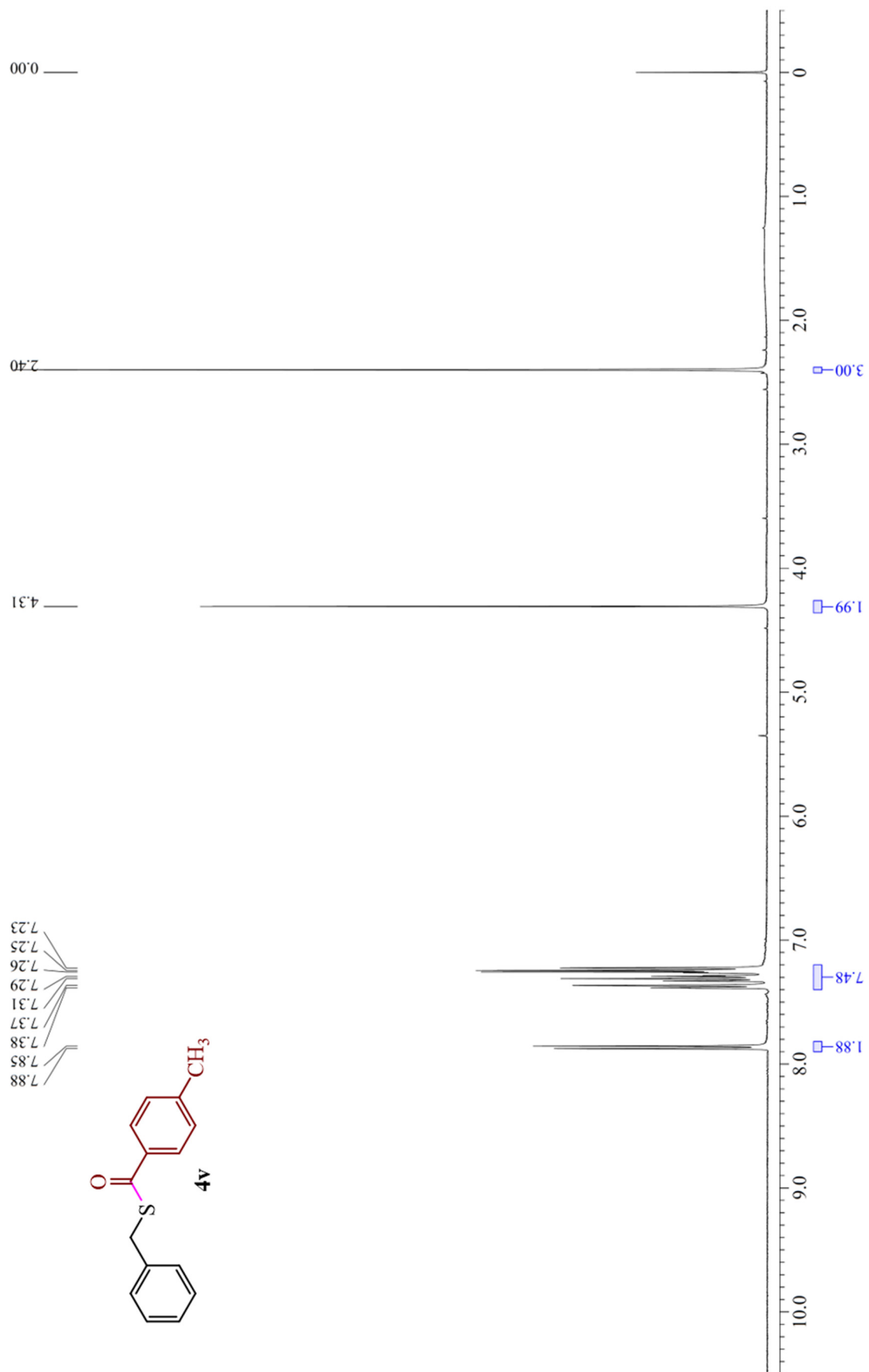
$^{13}\text{C}\{\text{H}\}$ NMR spectrum of compound **4t** (100 MHz, CDCl_3)



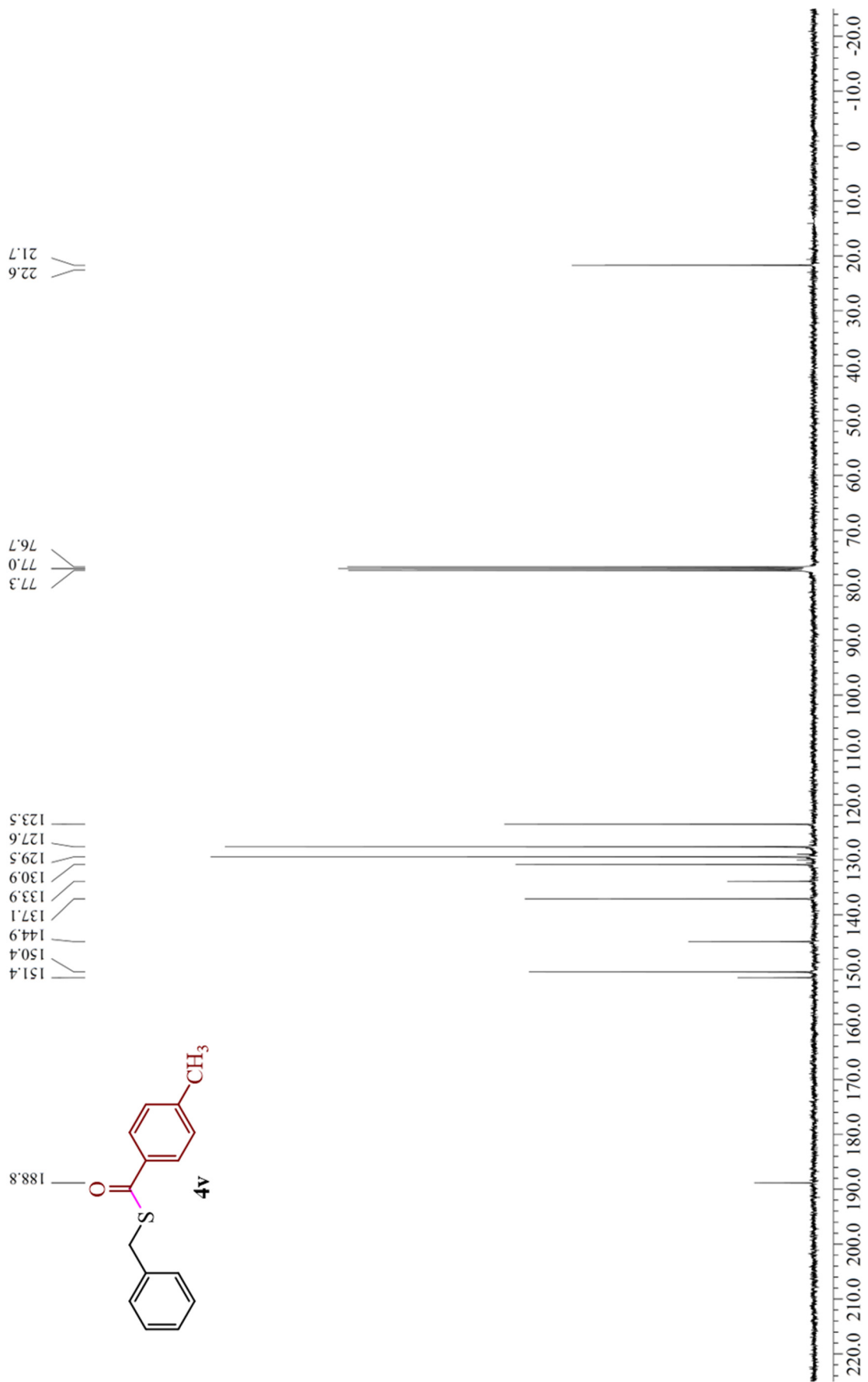
^1H NMR spectrum of compound **4u** (400 MHz, CDCl_3)



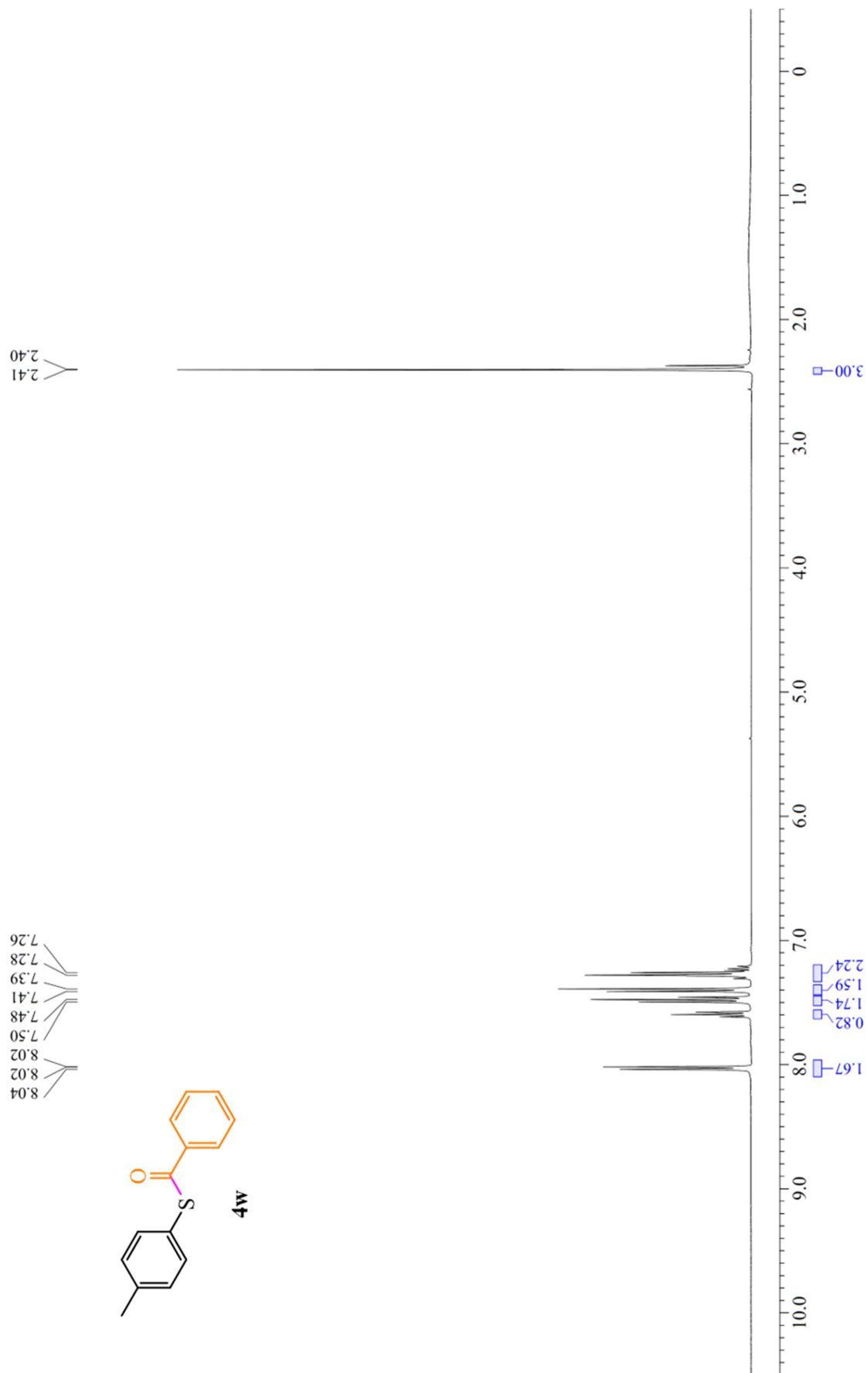
^{13}C {H} NMR spectrum of compound **4u** (100 MHz, CDCl_3)



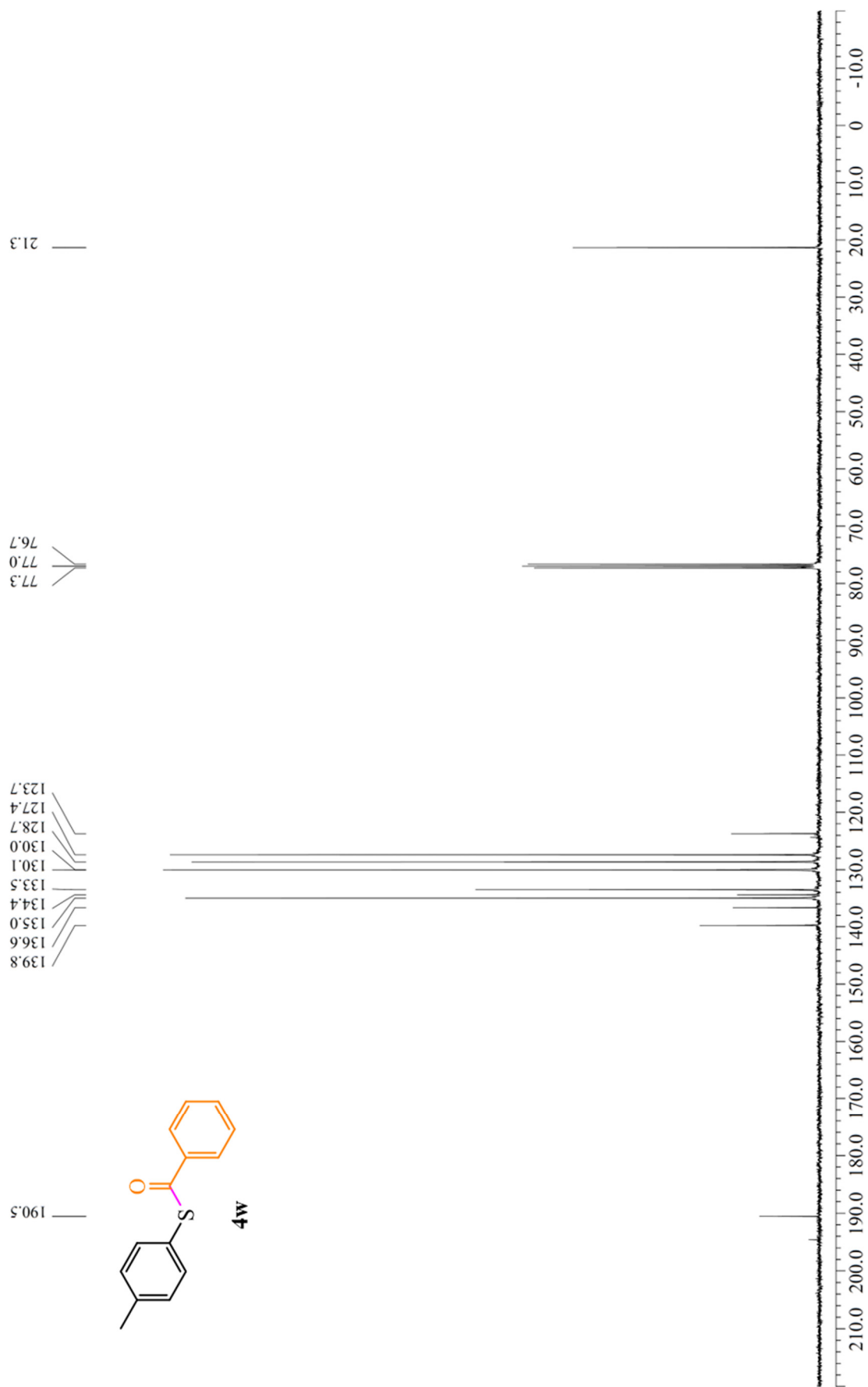
¹H NMR spectrum of compound **4v** (400 MHz, CDCl₃)



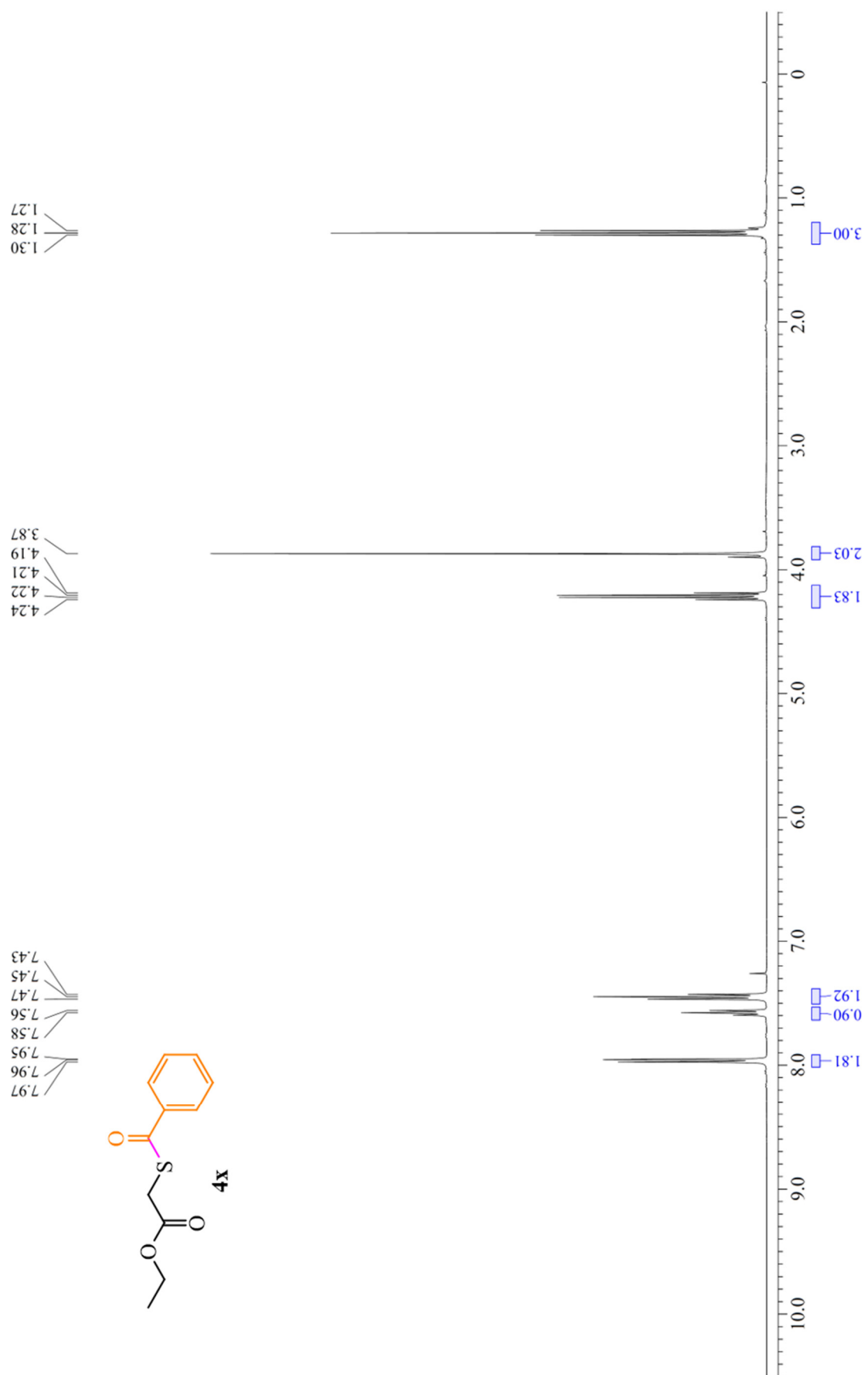
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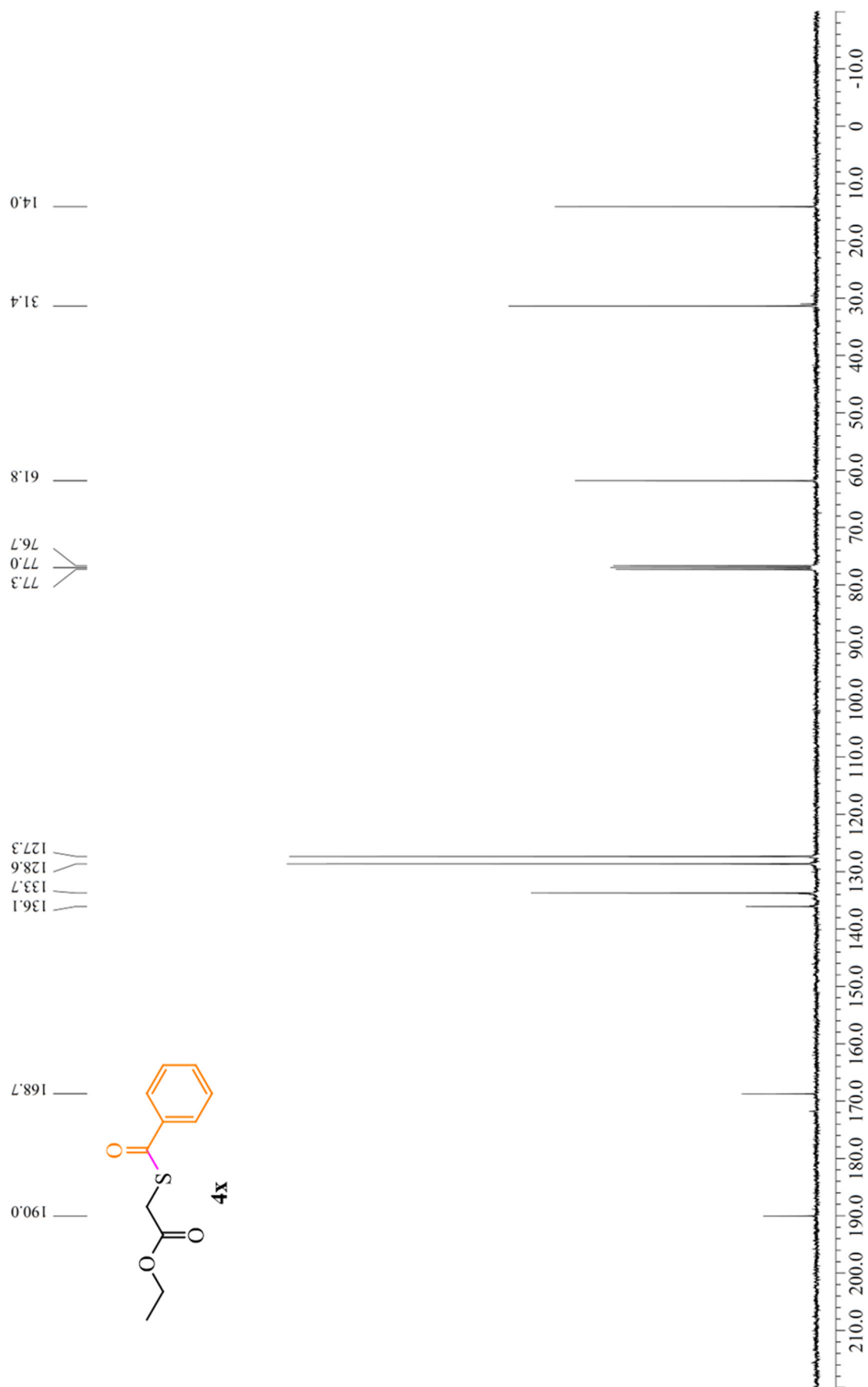
^1H NMR spectrum of compound **4w** (400 MHz, CDCl_3)



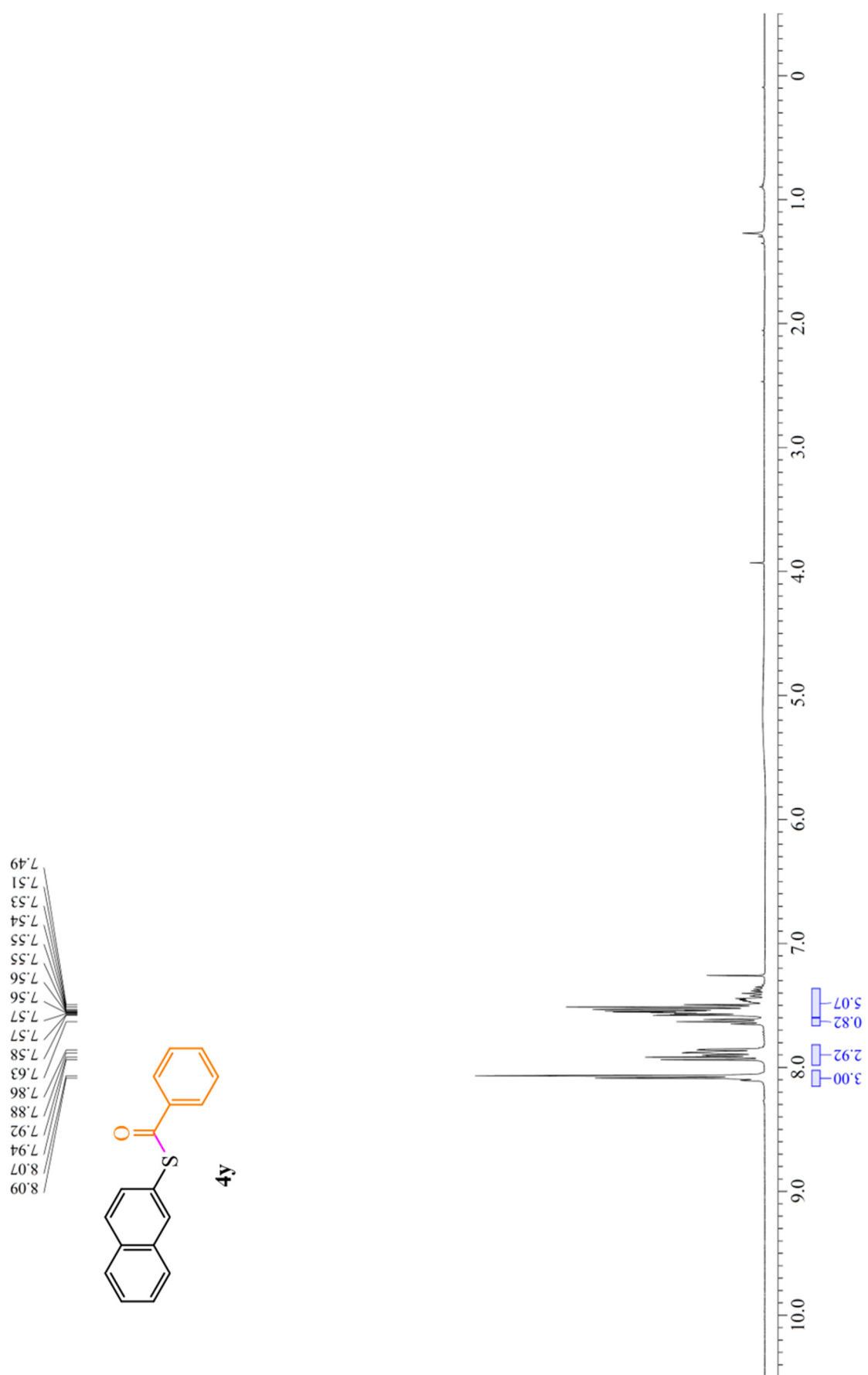
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4w** (100 MHz, CDCl_3)



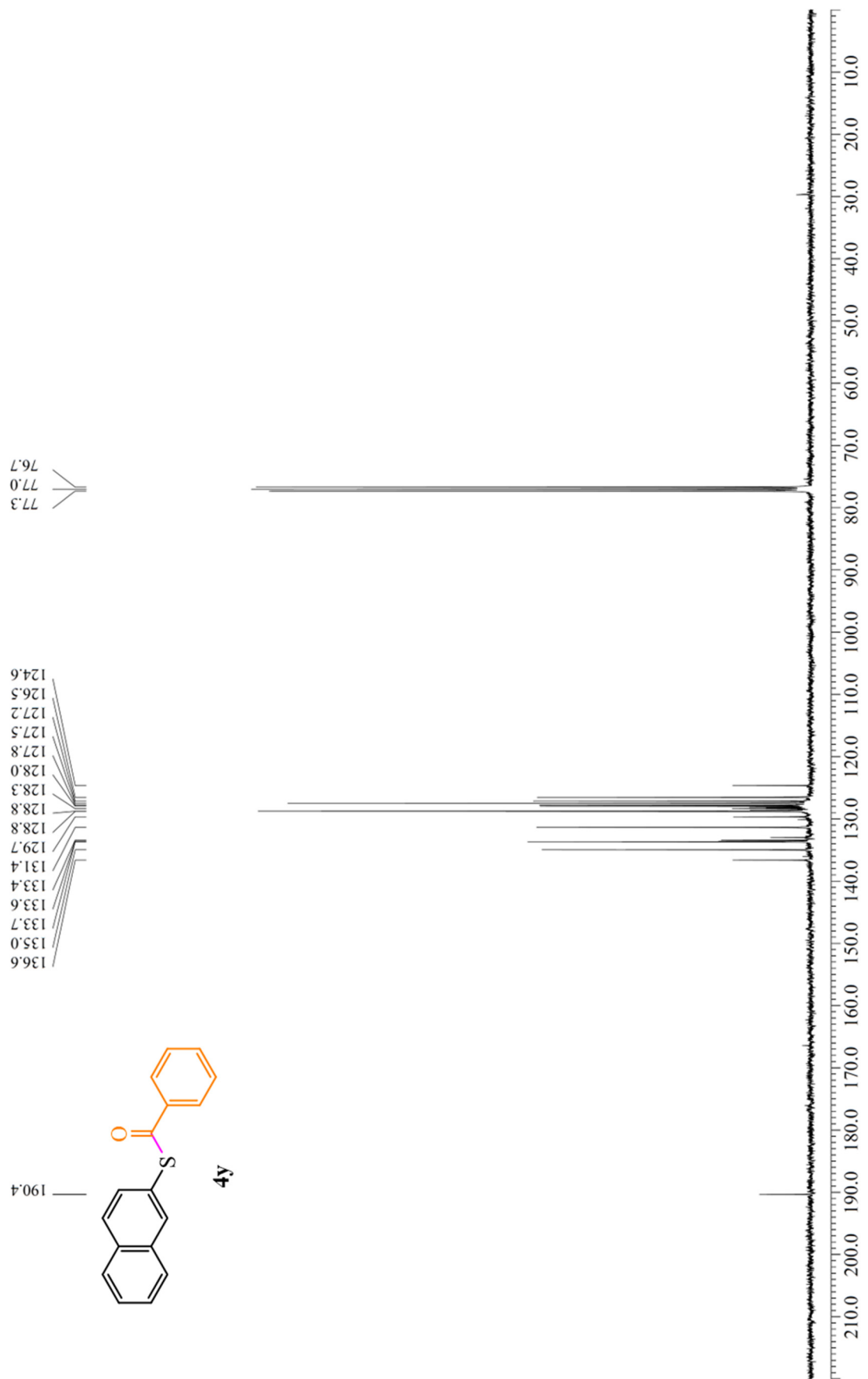
¹H NMR spectrum of compound **4x** (400 MHz, CDCl₃)



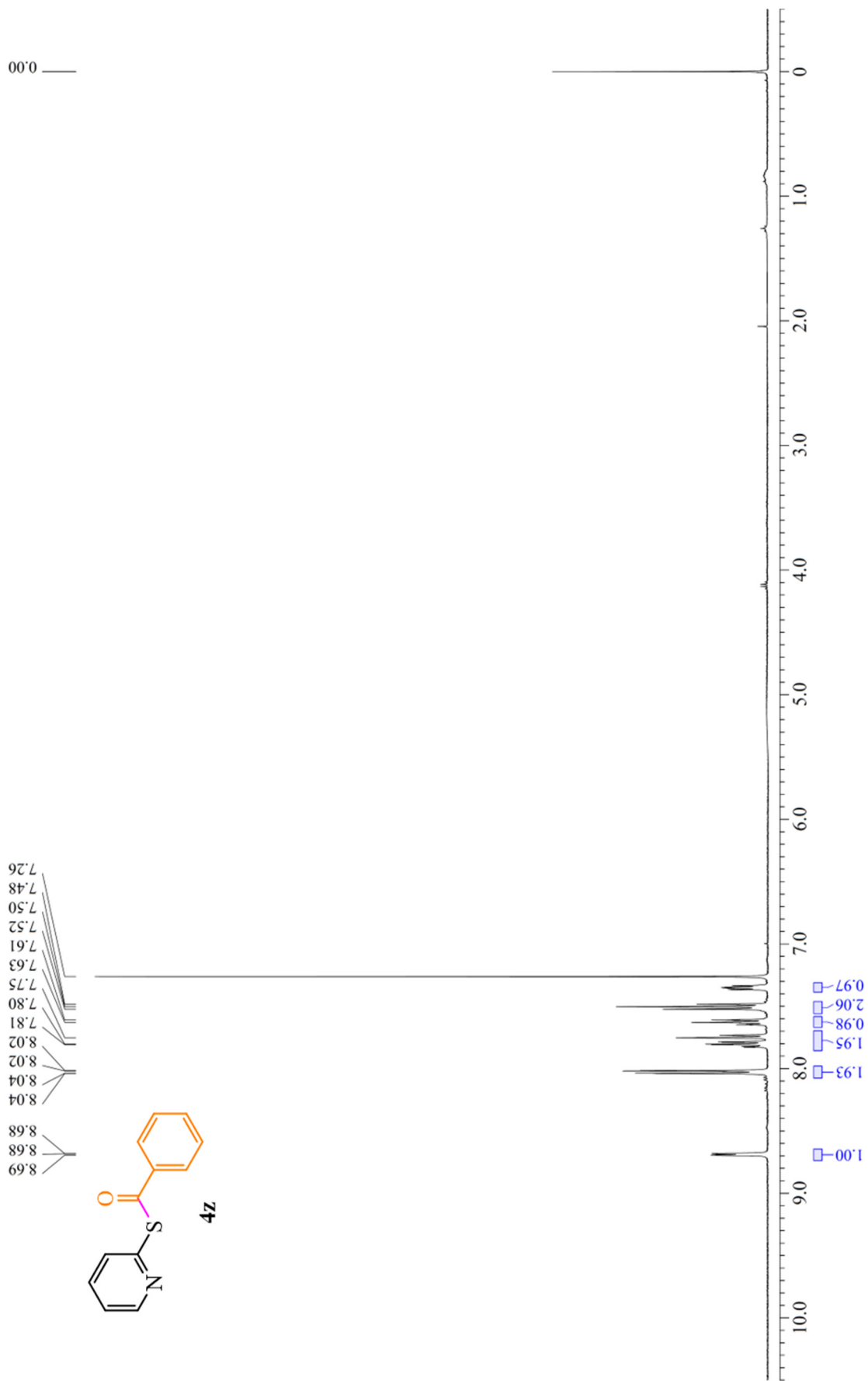
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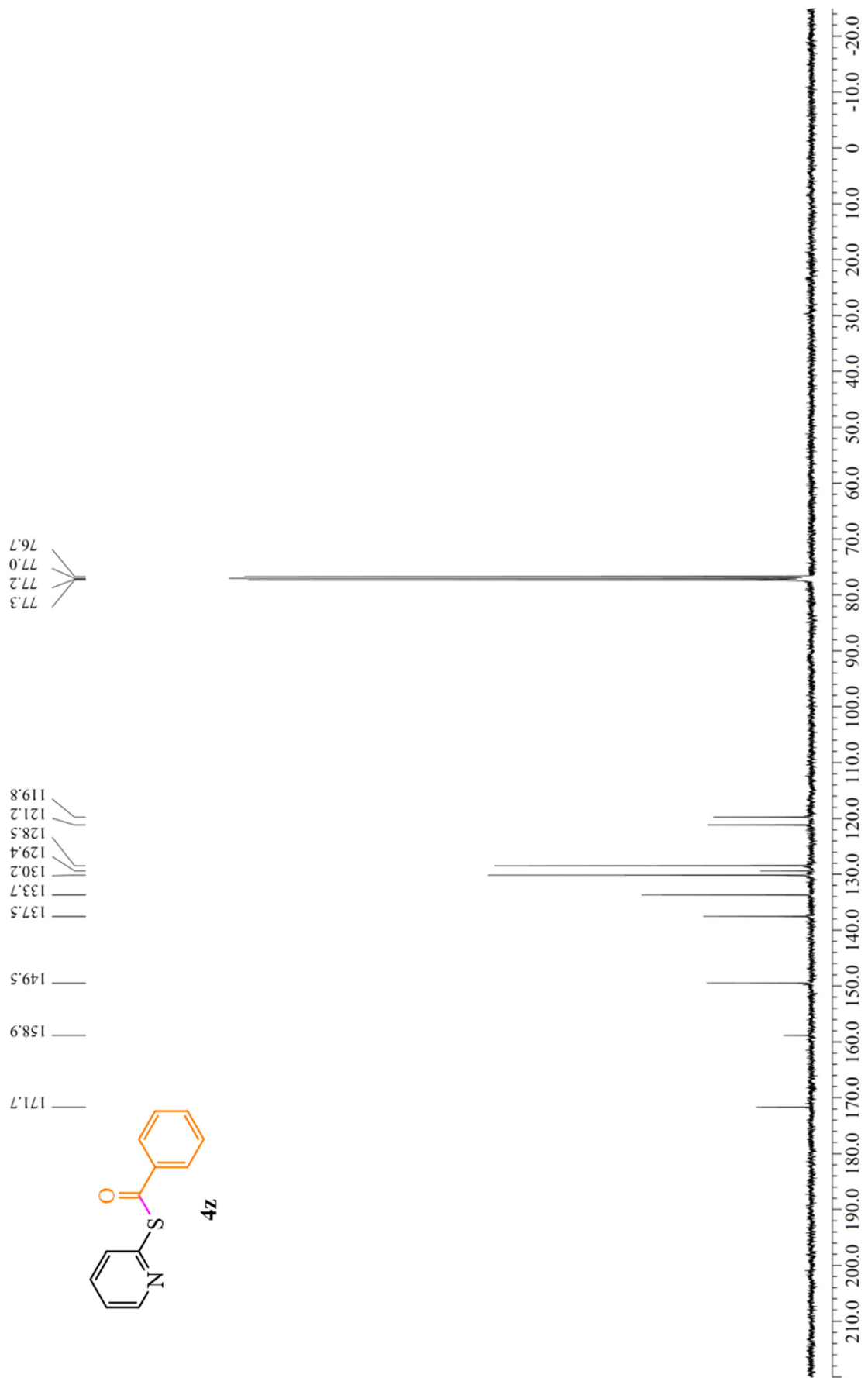
^1H NMR spectrum of compound **4y** (400 MHz, CDCl_3)



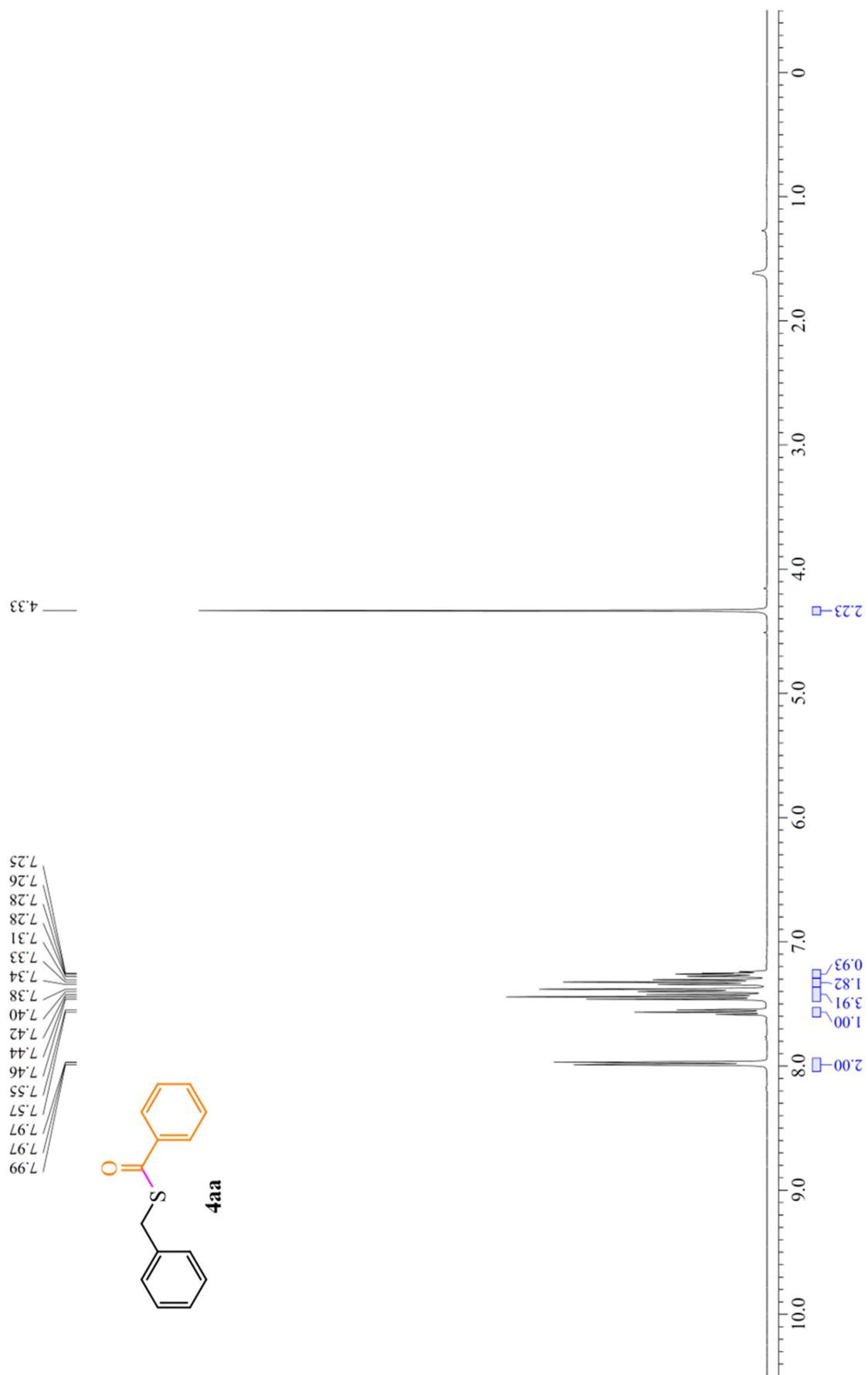
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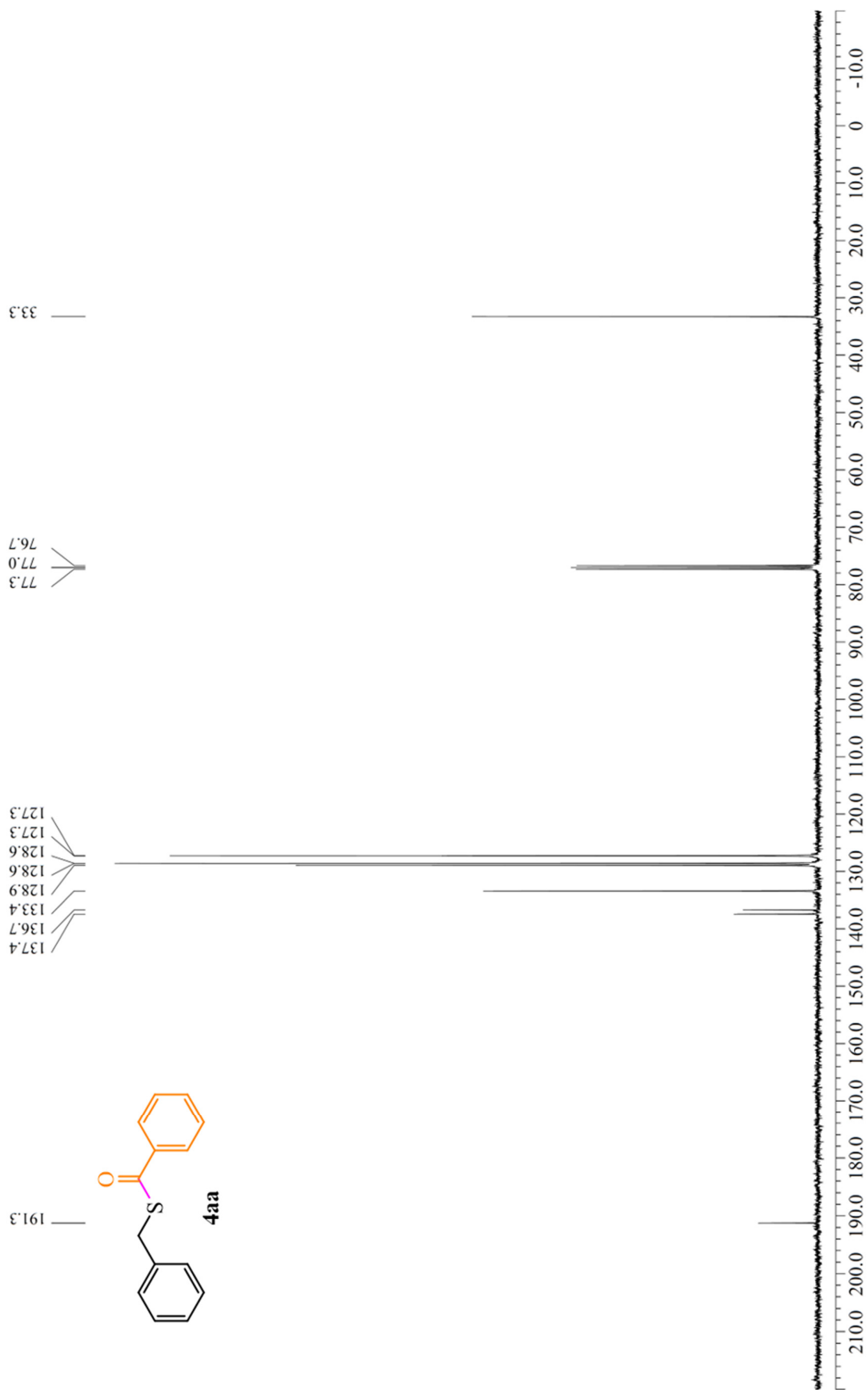
¹H NMR spectrum of compound **4z** (400 MHz, CDCl₃)



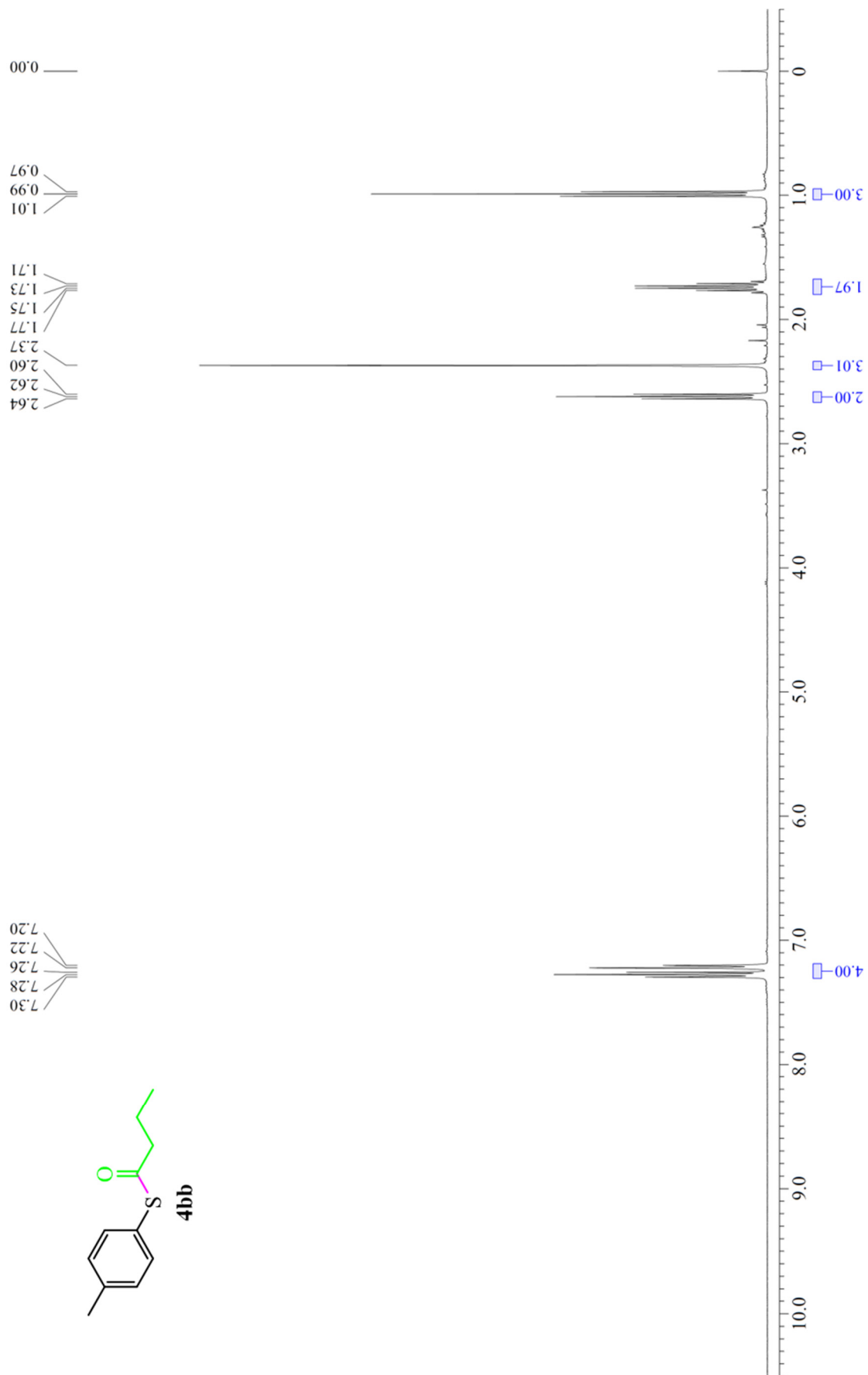
$^{13}\text{C}\{\text{H}\}$ NMR spectrum of compound **4z** (100 MHz, CDCl_3)



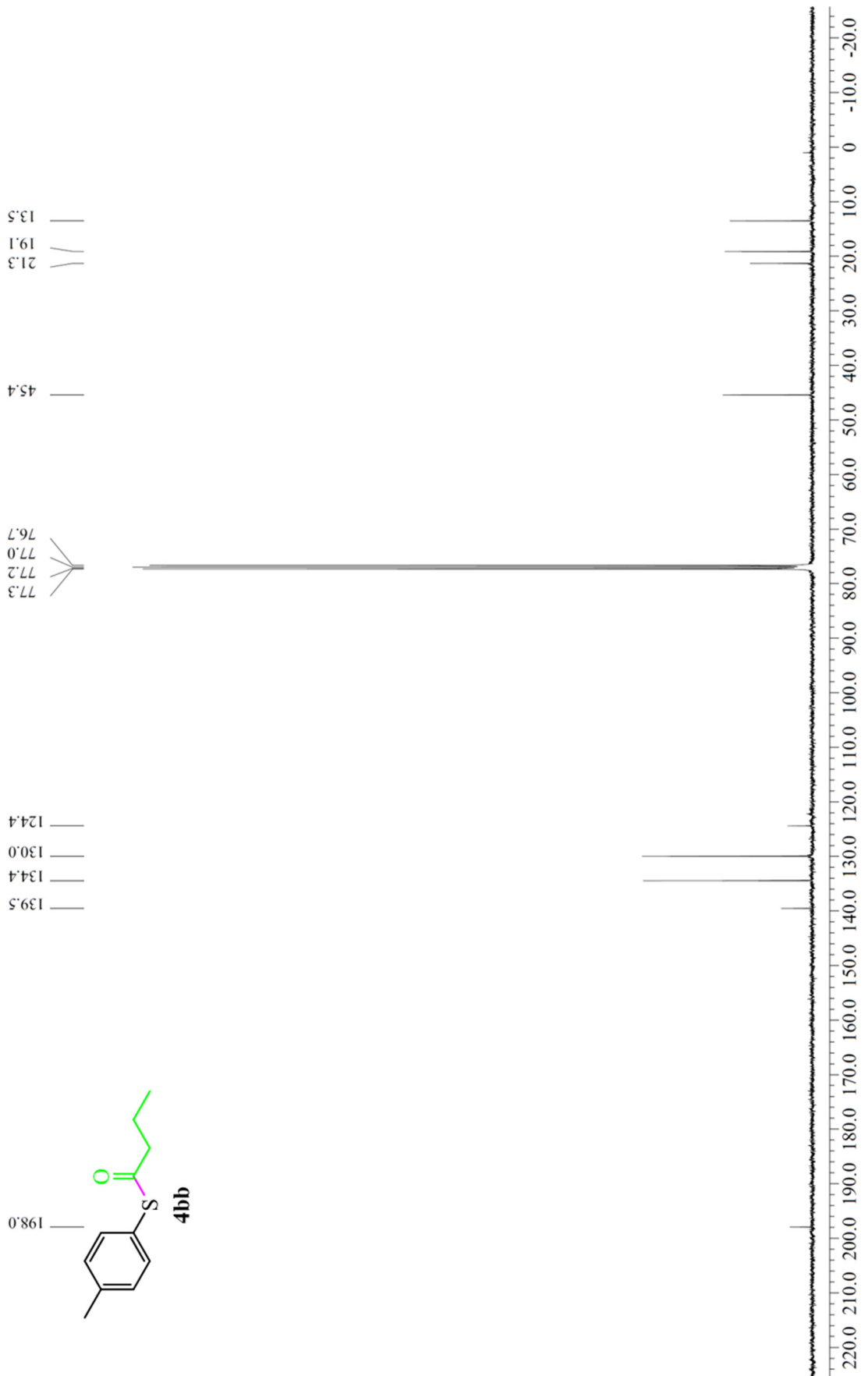
¹H NMR spectrum of compound **4aa** (400 MHz, CDCl₃)



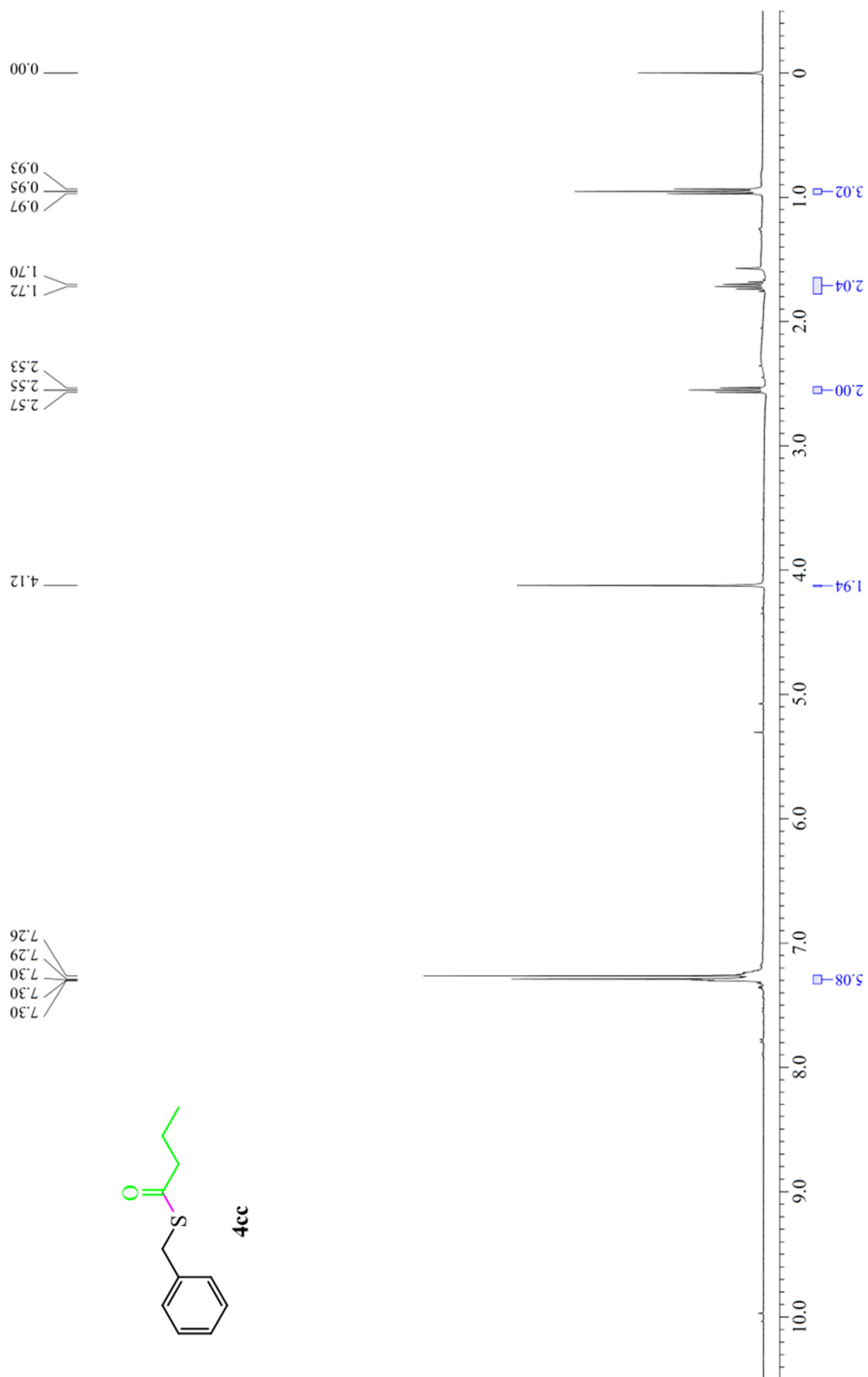
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4aa** (100 MHz, CDCl_3)



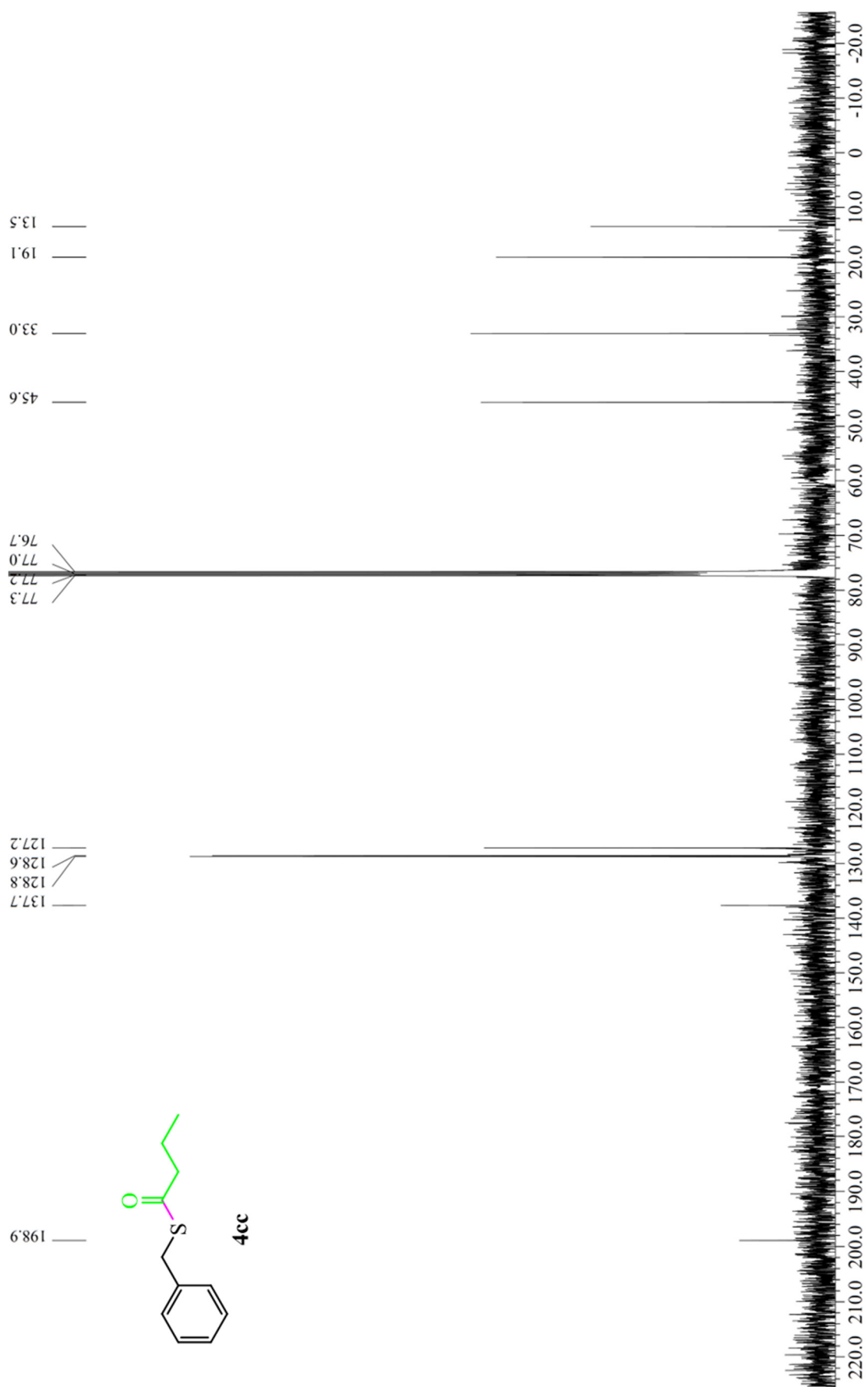
¹H NMR spectrum of compound **4bb** (400 MHz, CDCl₃)



¹³C{¹H} NMR spectrum of compound **4bb** (100 MHz, CDCl₃)



^1H NMR spectrum of compound **4cc** (400 MHz, CDCl_3)



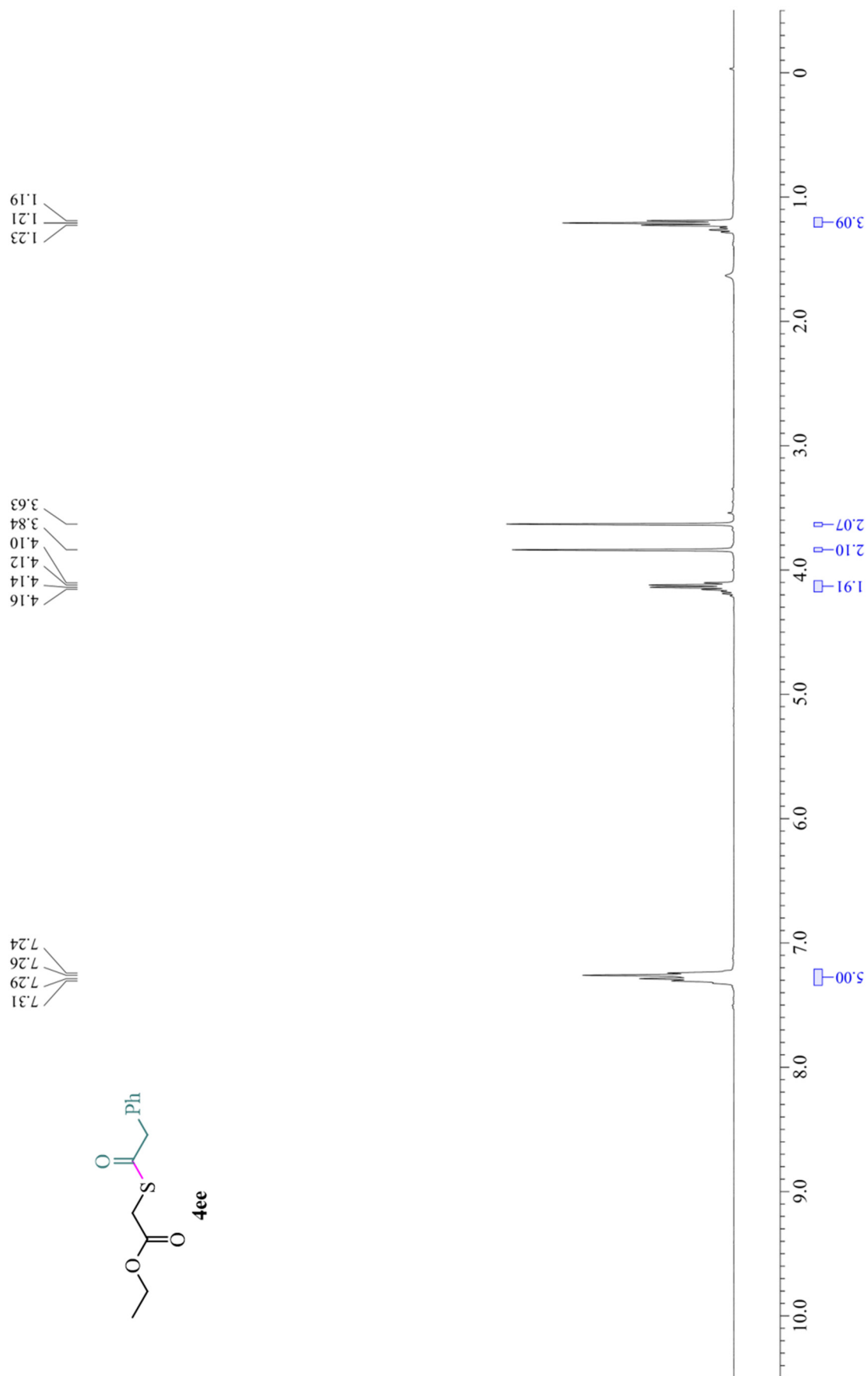
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4cc** (100 MHz, CDCl_3)



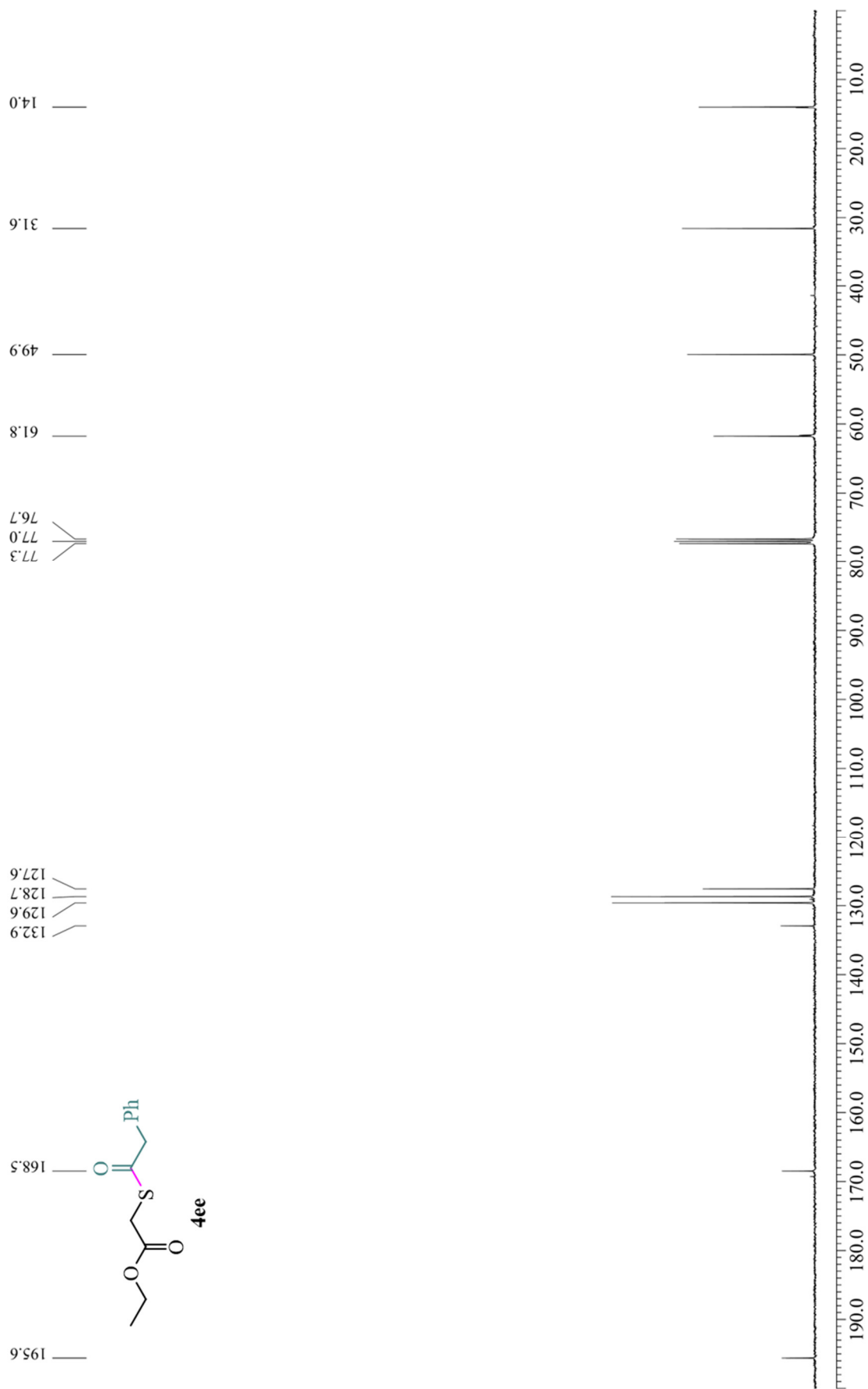
¹H NMR spectrum of compound **4dd** (400 MHz, CDCl₃)



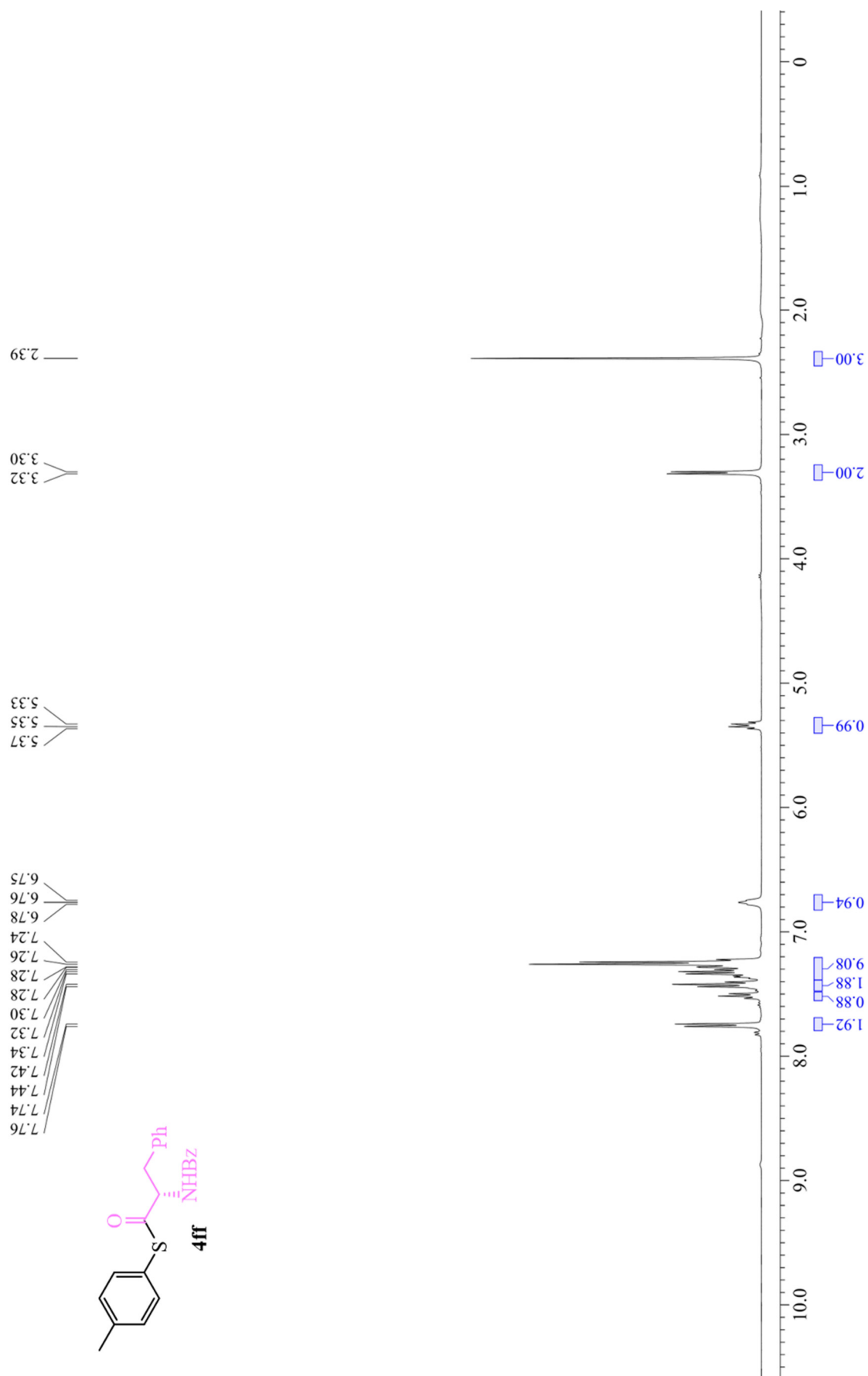
¹³C{H} NMR spectrum of compound **4dd** (100 MHz, CDCl₃)



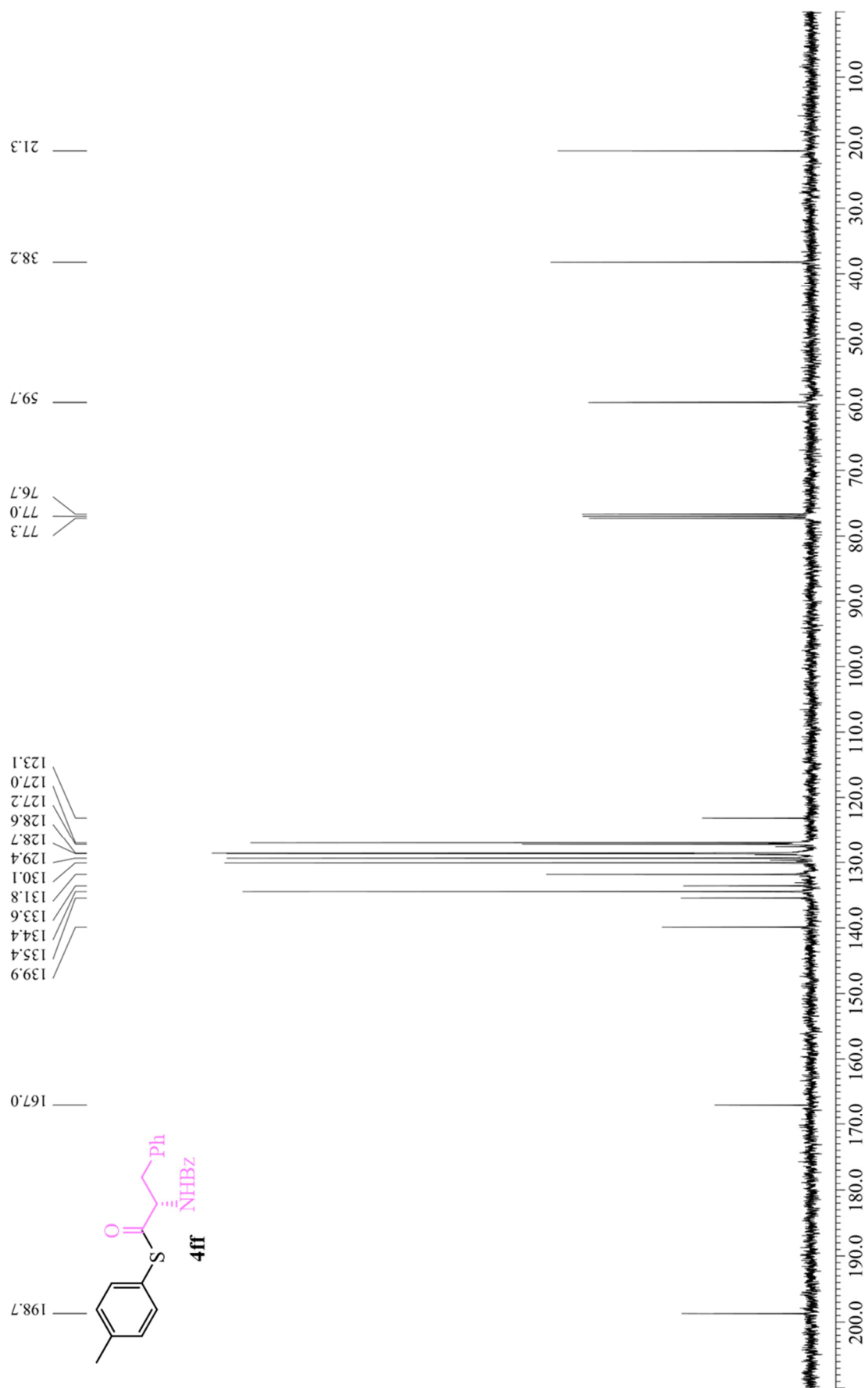
¹H NMR spectrum of compound **4ee** (400 MHz, CDCl₃)



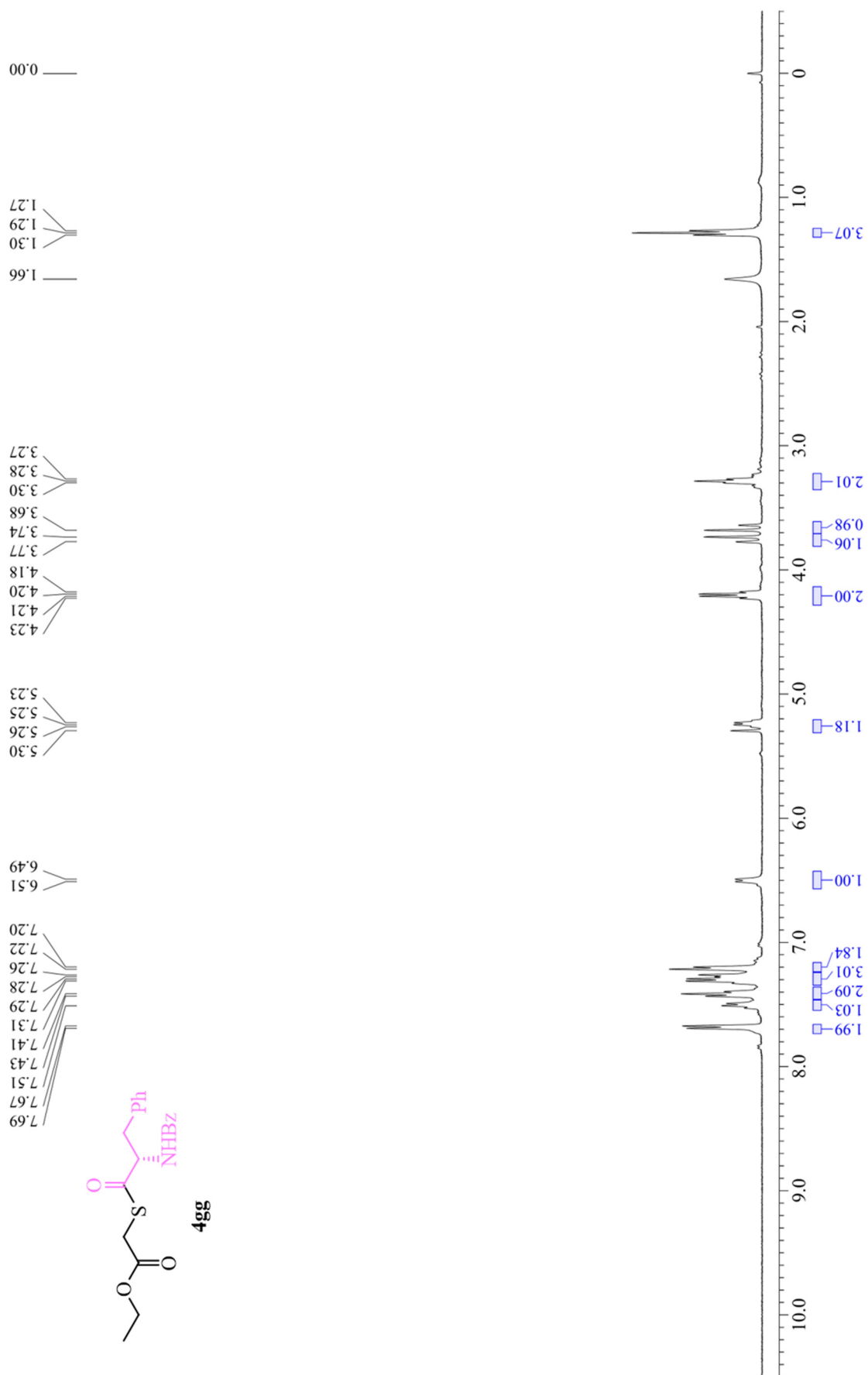
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4ee** (100 MHz, CDCl_3)

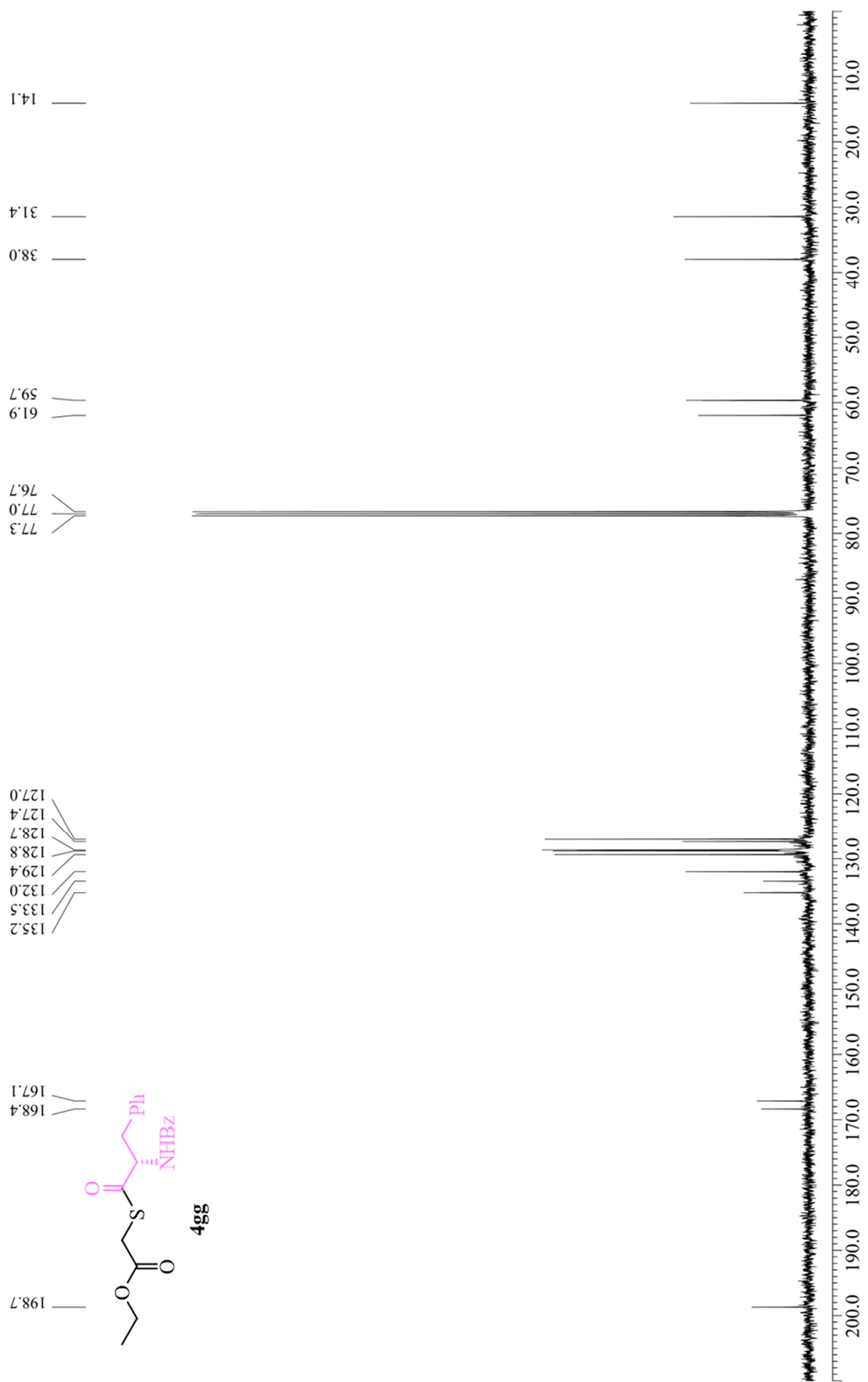


¹H NMR spectrum of compound **4ff** (400 MHz, CDCl₃)

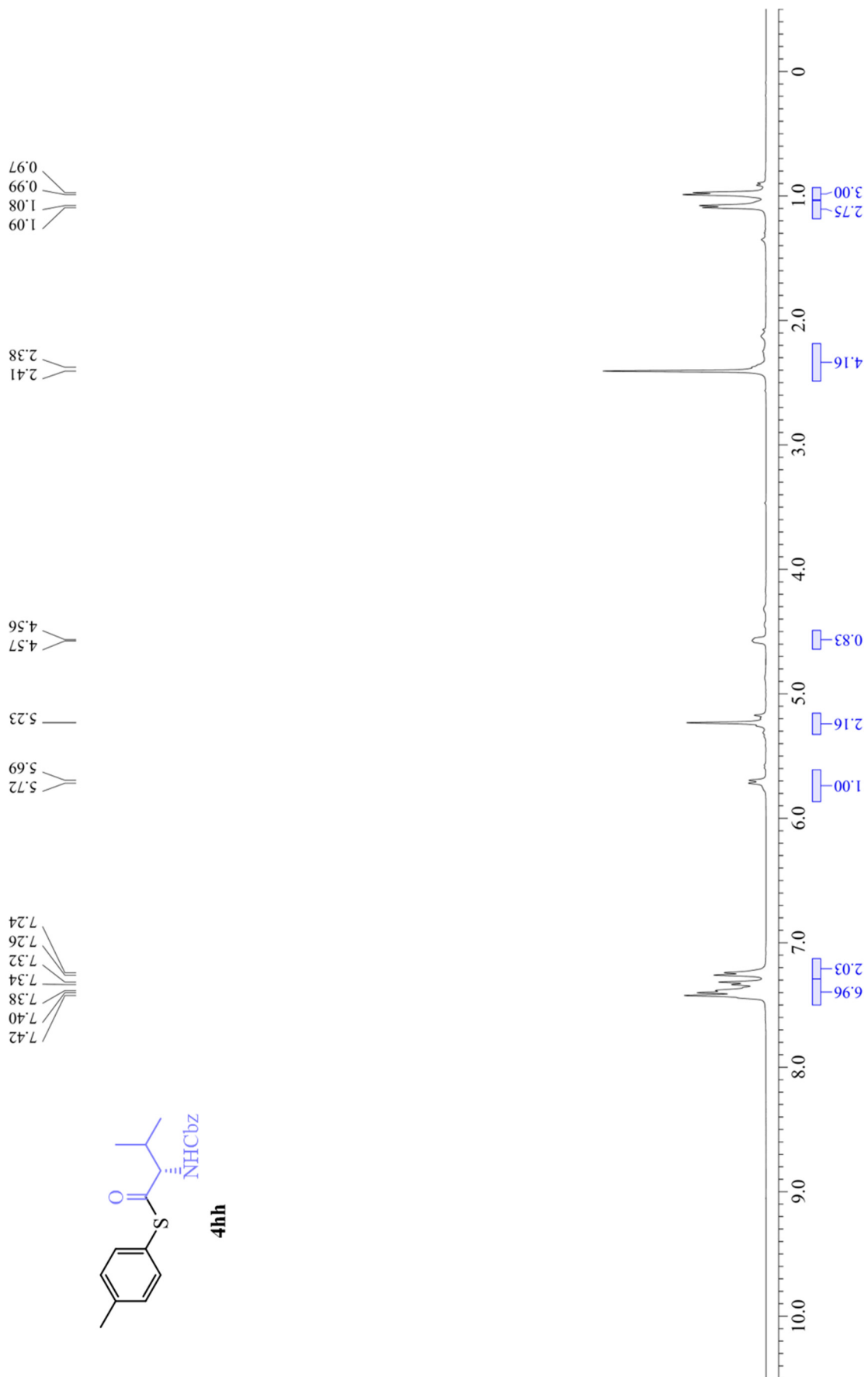


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4ff** (100 MHz, CDCl_3)

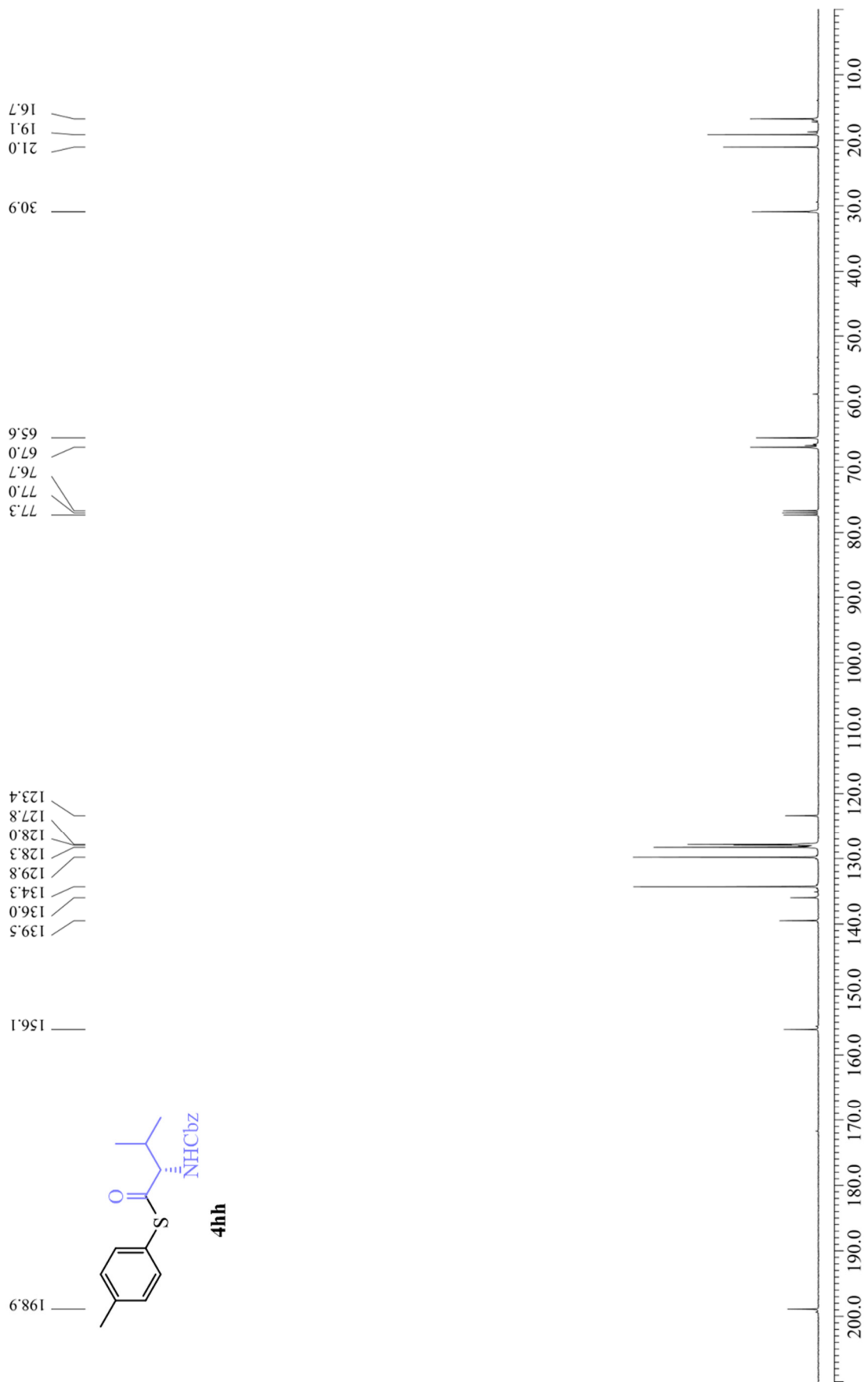




$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4gg** (100 MHz, CDCl_3)



^1H NMR spectrum of compound **4hh** (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4hh** (100 MHz, CDCl_3)

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