

Synthesis of Sultams and Cyclic *N*-Sulfonyl Ketimines via Iron-Catalyzed Intramolecular Aliphatic C–H Amidation

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1. General Information

Unless otherwise stated, all experiments were carried out in flame-dried glassware using argon manifolds or in an argon-filled glovebox. Reactions were monitored by thin-layer chromatography (TLC). TLC was performed using Huanghai $8 \pm 0.2 \mu\text{m}$ precoated glass plates (0.25 mm) and visualized by UV fluorescence quenching, KMnO_4 , *p*-anisaldehyde, or phosphomolybdic acid staining. Huanghai silica gel (particle size 300 – 400 or 200 – 300 mesh) was used for column chromatography. ^1H NMR spectra were recorded at room temp. on a Bruker ADVANCE III 400 MHz spectrometer and were reported relative to residual CDCl_3 (δ 7.26 ppm) or CD_3CN (δ 1.94 ppm). ^{13}C NMR spectra were recorded on a Bruker ADVANCE III 400 MHz spectrometer (100 MHz) and were reported relative to CDCl_3 (δ 77.16 ppm) or CD_3CN (δ 1.32 ppm, 118.26 ppm). ^{19}F NMR spectra were recorded on a Bruker ADVANCE III 400 MHz spectrometer (376 MHz) and were reported relative to CFCl_3 (δ 0.0 ppm). Data for ^1H NMR were reported using standard abbreviations for multiplicities. Data for ^{13}C NMR and ^{19}F NMR were reported in terms of chemical shifts (δ ppm). High resolution mass spectra (HRMS) were obtained by use of a Bruker Compact TOF mass spectrometer in electrospray ionization mode (ESI+).

Unless otherwise noted, all reagents were purchased commercially and used without further purification. Petroleum ester (PE, 60 ~ 90 °C) was used as eluent for silica gel chromatography. Dry solvents were purchased commercially or were dried by passage through an activated alumina column under argon.¹

2. Optimization of Reaction Conditions

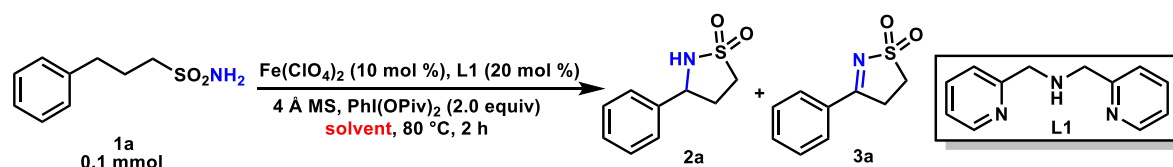
General procedure for reaction condition optimization: In an Argon-filled glovebox, iron salts, ligands and 0.5 mL solvent were added to a 4 mL vial (vial A) and stirred for half an hour. To another 4 mL vial (vial B) were added 4 Å MS (25 mg), oxidant (0.2 mmol, 2.0 equiv), **1a** (0.1 mmol, 1.0 equiv) and 0.5 mL solvent. Next, the solution in vial A was transferred to vial B. The vial B was sealed and the mixture was stirred at the indicated temperature for the indicated time. The vial was removed from the glovebox, and solvent was concentrated under reduced pressure. The residue was dissolved in CH₂Cl₂ (10 mL) and washed with sat. NaHCO₃ aqueous solution (5 mL). The aqueous layer was extracted with CH₂Cl₂ (3 × 5 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. Yields of **2a** and **3a** were determined by ¹H NMR analyses (relaxation delay was set as d1 = 10) of the crude mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S1. Oxidant effects

entry ^a	oxidant (x equiv)	2a (%) ^b	3a (%) ^b	1a (%) ^b
1	PhI(OAc) ₂ (1.5)	65	5	14
2	PhI(OAc) ₂ (2.0)	68	14	—
3	PhI(OAc) ₂ (2.5)	58	26	—
4	PhI(OAc) ₂ (3.0)	47	38	—
5	PhI(OAc) ₂ (4.0)	35	46	—
6	PhI(OCOCF ₃) ₂ (2.0)	—	32	53
7	PhI(OPiv) ₂ (2.0)	74 (73) ^c	9 (12) ^c	—
8	PhI(DMM) (2.0)	54	9	—
9	PhIO (2.0)	12	—	85

Reaction conditions: **1a** (0.1 mmol), oxidant (x equiv), 4 Å MS (25 mg), MeCN (1.0 mL), 80 °C. ^b¹H NMR yield using 1,3,5-trimethoxybenzene as an internal standard. ^cIsolated yield. PhI(DMM) = phenyliodonium dimethylmalonate.

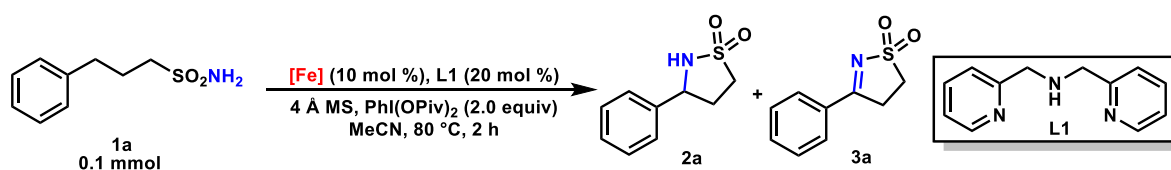
Table S2. Screening of solvents



entry ^a	solvent	2a (%) ^b	3a (%) ^b	1a (%) ^b
1	DCE	68	11	—
2	toluene	12	—	72
3	DMF	13	—	79
4	1,4-dioxane	17	—	63
5	MeCN	74	9	—

^aReaction conditions: **1a** (0.1 mmol), PhI(OPiv)₂ (0.2 mmol), 4 Å MS (25 mg), MeCN (1.0 mL), 80 °C. ^b¹H NMR yield using 1,3,5-trimethoxybenzene as an internal standard.

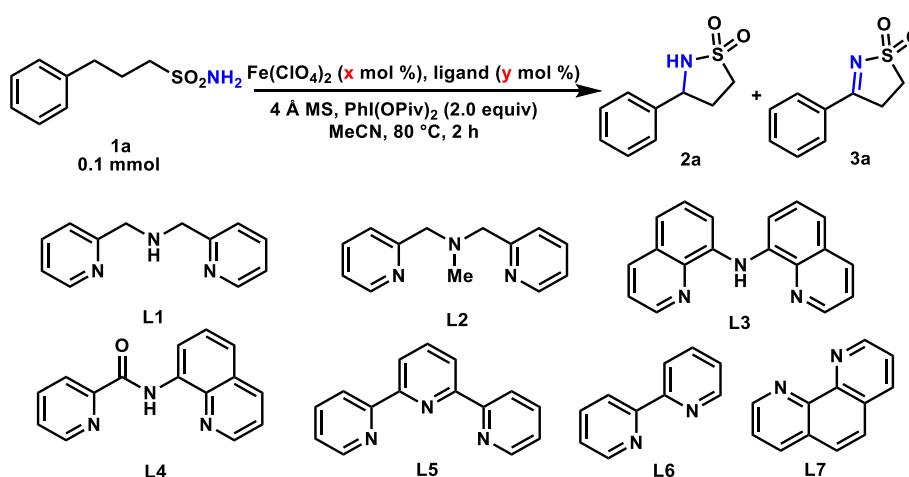
Table S3. Iron sources screen



entry ^a	[Fe]	2a (%) ^b	3a (%) ^b	1a (%) ^b
1	Fe(OTf) ₂	52	—	32
2	Fe(acac) ₂	61	—	31
3	FeCl ₂	50	6	32
4	Fe(ClO ₄) ₃	59	11	9
5	Fe(BF ₄) ₂	65	15	—
6	Fe(SbF ₆) ₂ ^c	71	13	—
7	Fe(ClO ₄) ₂	74	9	—

^aReaction conditions: **1a** (0.1 mmol), PhI(OPiv)₂ (0.2 mmol), 4 Å MS (25 mg), MeCN (1.0 mL), 80 °C. ^b¹H NMR yield using 1,3,5-trimethoxybenzene as an internal standard. ^cPrepared in situ with 10 mol % of FeCl₂ and 20 mol % of AgSbF₆ (2.0 equiv to Fe) in MeCN.

Table S4. Screening of catalyst loading and ligands

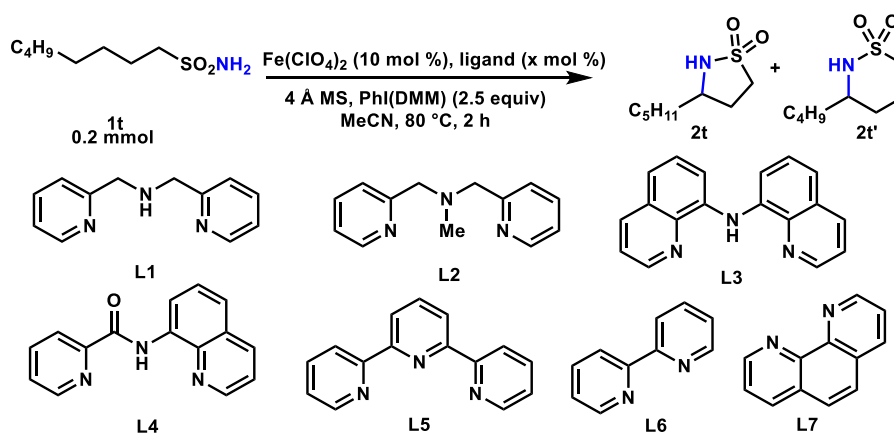


entry ^a	Fe(ClO ₄) ₂ (x mol %)	ligand (y mol %)	2a (%) ^b	3a (%) ^b	1a (%) ^b
1	10	L1 (20)	74	9	—
2	5	L1 (10)	46	22	13

3	2.5	L1 (5)	37	23	15
4	1	L1 (2)	13	20	13
5	10	L2 (20)	80 (81) ^c	10 (12) ^c	—
6	10	L3 (20)	47	—	41
7	10	L4 (20)	25	—	53
8	10	L5 (20)	59	16	6
9	10	L6 (30)	64	19	—
10	10	L7 (30)	63	21	—
11	10	no 4 Å MS	60	15	—
12	10	no ligand	—	—	71
13	—	L2 (20)	—	—	83
14 ^d	10	L2 (20)	65	14	—

^aReaction conditions: **1a** (0.1 mmol), PhI(OPiv)₂ (0.2 mmol), 4 Å MS (25 mg), MeCN (1.0 mL), 80 °C. ^b¹H NMR yield using 1,3,5-trimethoxybenzene as an internal standard. ^cIsolated yield. ^d60 °C.

Table S5. Optimization of ligands for the reaction of substrate 1t

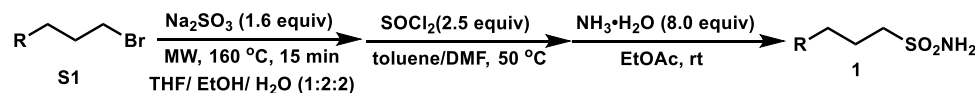


entry ^a	ligand (x mol %)	2t+2t' (%) ^b	2t:2t'	1t (%) ^b
1	L1 (20)	93 (85) ^c	3:5	2
2	L2 (20)	78	2:3	5
3	L3 (20)	52	1:1	26
4	L4 (20)	61	1:1	—
5	L5 (20)	66	1:1	8
6	L6 (30)	89 (75) ^c	2:1	—
7	L7 (30)	81	3:2	—

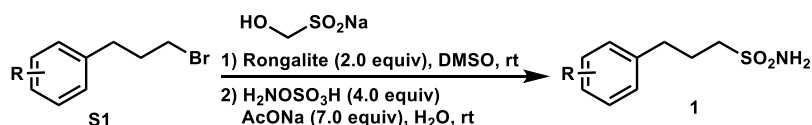
^aReaction conditions: **1a** (0.2 mmol), PhI(DMM) (0.5 mmol), 4 Å MS (50 mg), MeCN (2.0 mL), 80 °C. ^b¹H NMR yield using 1,3,5-trimethoxybenzene as an internal standard. ^cIsolated yield.

3. Preparation and Characterization of Substrates 1

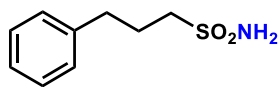
General procedure for preparation of sulfonamide substrates:



Method A:² A mixture of alkyl bromide **S1** (1.0 equiv, 0.2 mol/L) and Na₂SO₃ (2.0 equiv) in THF / EtOH / H₂O (1 / 2 / 2) was heated at 160 °C for 30 min under microwave irradiation (300 W). The reaction mixture was cooled to room temp. (~ 25 °C), then dried under high vacuo and the crude sulfonate product was used directly without further purification. To a stirring suspension of this sulfonate in anhydrous toluene / DMF (70 / 1) was added dropwise SOCl₂ (3.0 equiv) and heated at 50 °C under argon. After the completion of the reaction (monitored by TLC), H₂O (~ 10 mL) was added to quench the reaction and stirred until the solution turned clear. The organic phase was collected and the aqueous layer was extracted with Et₂O (3 × 15 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude products were purified by flash column chromatography (200 – 300 mesh SiO₂) to give the desired sulfonyl chloride. Then, to the solution of the sulfonyl chloride (1.0 equiv, 0.1 mol/L) in ethyl acetate (EA), was added dropwise ammonium hydroxide solution (25%, 8.0 equiv) at room temp. (~ 25 °C). After the completion of the reaction (monitored by TLC), the organic phase was collected and the aqueous layer was extracted with EA (3 × 15 mL). The combined organic layers were dried over Na₂SO₄, filtered and concentrated under reduced pressure. The crude products were purified by flash column chromatography (200 – 300 mesh SiO₂) to give the desired sulfonamide **1**.



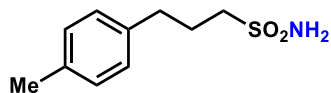
Method B:³ To a 25 mL Schlenk tube were added Rongalite (1.0 mmol, 2.0 equiv) and DMSO (3 mL) and the mixture was stirred at room temp. (~ 25 °C) for 30 min. Then alkyl bromide **S1** was added and the reaction mixture was stirred for 18 h. A solution of H₂NOSO₃H (2.0 mmol, 4.0 equiv) and AcONa (3.5 mmol, 7.0 equiv) in water was added dropwise under stirring at room temp. (~ 25 °C) and stirred at the same temperature for 12 h. The mixture was diluted with water (5 mL), the organic phase was collected and the aqueous layer was extracted with EA (3 × 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude products were purified by flash column chromatography (200 – 300 mesh SiO₂) to give the desired sulfonamide **1**.



3-Phenylpropane-1-sulfonamide **1a**:³

Prepared according to method B, 4.24 g (20.0 mmol) of (3-bromopropyl) benzene was used. The desired product **1a** was obtained as a white solid (1.77 g, 45% yield) after flash column chromatography using petroleum ether (PE) / ethyl acetate (EA) = 2 / 1 to 1 / 1 as eluent. R_f = 0.4 (PE / EA = 1 / 1, KMnO₄).

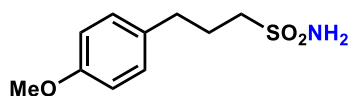
¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.29 (m, 2H), 7.24 – 7.17 (m, 3H), 4.73 (br s, 2H), 3.20 – 2.98 (m, 2H), 2.76 (t, J = 7.6 Hz, 2H), 2.28 – 2.07 (m, 2H).



3-(4-Tolyl)propane-1-sulfonamide **1b**:

Prepared according to method B, 220.0 mg (1.0 mmol) of 1-(3-bromopropyl)-4-methylbenzene was used. The desired product **1b** was obtained as a white solid (45.0 mg, 21% yield) after flash column chromatography using PE / EA = 5 / 1 to 1 / 1 as eluent. R_f = 0.4 (PE / EA = 1 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.12 – 7.06 (m, 4H), 4.58 (br s, 2H), 3.15 – 3.02 (m, 2H), 2.73 (t, J = 7.4 Hz, 2H), 2.32 (s, 3H), 2.22 – 2.12 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 137.0, 136.1, 129.5, 128.5, 54.6, 33.7, 25.7, 21.2; **HRMS** (ESI⁺) calc'd for C₁₀H₁₅NNaO₂S [M+Na]⁺: 236.0716, found 236.0716.

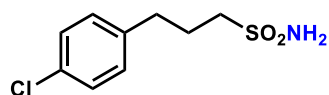


3-(4-Methoxyphenyl)propane-1-sulfonamide **1c**:

Prepared according to method A, 916.6 mg (4.0 mmol) of 1-(3-bromopropyl)-4-methoxybenzene was used. The desired product **1c** was obtained as a white solid (317.7 mg, 35% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. R_f = 0.3 (PE / EA = 2 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.10 (dd, 2H), 6.89 (dd, 2H), 4.63 (br s, 2H), 3.79 (s, 3H), 3.12 – 3.04 (m, 2H), 2.71 (t, J = 7.4 Hz, 2H), 2.21 – 2.10 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ

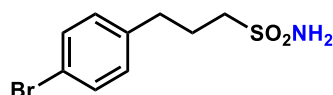
158.3, 132.1, 129.5, 114.2, 55.4, 54.6, 33.2, 25.8; **HRMS** (ESI⁺) calc'd for C₁₀H₁₅NNaO₃S [M+Na]⁺: 252.0665, found 252.0664.



3-(4-Chlorophenyl) propane-1-sulfonamide **1d**:

Prepared according to method B, 1.16 g (5.0 mmol) of 1-(3-bromopropyl)-4-chlorobenzene was used. The desired product **1d** was obtained as a white solid (297.2 mg, 44% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. R_f = 0.3 (PE / EA = 1 / 1, KMnO₄).

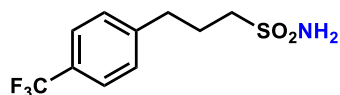
¹H NMR (400 MHz, CDCl₃) δ 7.27 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 8.4 Hz, 2H), 4.64 (br s, 2H), 3.15 – 3.00 (m, 2H), 2.75 (t, J = 7.5 Hz, 2H), 2.23 – 2.09 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 138.6, 132.4, 129.9, 128.9, 54.5, 33.5, 25.6; **HRMS** (ESI⁺) calc'd for C₉H₁₂ClNNaO₂S [M+Na]⁺: 256.0169, found 256.0166.



3-(4-Bromophenyl) propane-1-sulfonamide **1e**:

Prepared according to method B, 1.11 g (4.0 mmol) of 1-bromo-4-(3-bromopropyl) benzene was used. The desired product **1e** was obtained as a white solid (324.0 mg, 29% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. R_f = 0.4 (PE / EA = 1 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, J = 8.3 Hz, 2H), 7.06 (d, J = 8.2 Hz, 2H), 4.63 (br s, 2H), 3.08 (t, J = 8.0 Hz, 2H), 2.74 (t, J = 7.5 Hz, 2H), 2.21 – 2.13 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 139.1, 131.9, 130.3, 120.4, 54.5, 33.5, 25.5; **HRMS** (ESI⁺) calc'd for C₉H₁₂BrNNaO₂S [M+Na]⁺: 299.9664, found 299.9662.

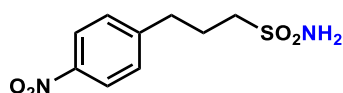


3-(4-(Trifluoromethyl) phenyl) propane-1-sulfonamide **1f**:

Prepared according to method A, 1.06 g (4.0 mmol) of 1-(3-bromopropyl)-4-(trifluoromethyl) benzene was used. The desired product **1f** was obtained as a white solid (530.5 mg, 46% yield)

after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. $R_f = 0.3$ (PE / EA = 2 / 1, KMnO_4).

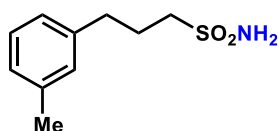
^1H NMR (400 MHz, CDCl_3) δ 7.56 (d, $J = 8.0$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 4.69 (br s, 2H), 3.19 – 3.05 (m, 2H), 2.85 (t, $J = 7.5$ Hz, 2H), 2.27 – 2.14 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 144.3 (q, $J = 1.3$ Hz), 129.0 (q, $J = 32.2$ Hz), 128.9, 127.1 (q, $J = 279.1$ Hz), 125.7 (q, $J = 3.8$ Hz), 54.5, 33.9, 25.3; **^{19}F NMR** (376 MHz, CDCl_3) δ –62.39 (s); **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{12}\text{F}_3\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 290.0433, found 290.0433.



3-(4-Nitrophenyl) propane-1-sulfonamide **1g**:

Prepared according to method B, 495.0 mg (2.0 mmol) of 1-(3-bromopropyl)-4-nitrobenzene was used. The desired product **1g** was obtained as a white solid (207.7 mg, 43% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. $R_f = 0.4$ (PE / EA = 1 / 1, KMnO_4).

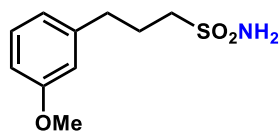
^1H NMR (400 MHz, CD_3CN) δ 8.16 (d, $J = 8.6$ Hz, 2H), 7.47 (d, $J = 8.5$ Hz, 2H), 5.26 (br s, 2H), 3.10 – 2.99 (m, 2H), 2.88 (t, $J = 7.7$ Hz, 2H), 2.15 – 2.05 (m, 2H); **^{13}C NMR** (100 MHz, CD_3CN) δ 150.2, 147.6, 130.5, 124.5, 54.6, 34.2, 26.0; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_{12}\text{N}_2\text{NaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 267.0410, found 267.0409.



3-(3-Tolyl) propane-1-sulfonamide **1h**:

Prepared according to method B, 530.0 mg (2.5 mmol) of 1-(3-bromopropyl)-3-methylbenzene was used. The desired product **1h** was obtained as a white solid (100.3 mg, 19% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. $R_f = 0.5$ (PE / EA = 1 / 1, KMnO_4).

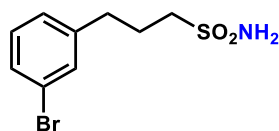
^1H NMR (400 MHz, CDCl_3) δ 7.21 – 7.17 (m, 1H), 7.05 – 6.97 (m, 3H), 4.55 (br s, 2H), 3.14 – 3.05 (m, 2H), 2.74 (t, $J = 7.4$ Hz, 2H), 2.33 (s, 3H), 2.25 – 2.12 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 140.1, 138.4, 129.4, 128.7, 127.4, 125.6, 54.7, 34.1, 25.6, 21.5; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{15}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 236.0716, found 236.0714.



3-(3-Methoxyphenyl) propane-1-sulfonamide **1i**:

Prepared according to method B, 1.14 g (5.0 mmol) of 1-(3-bromopropyl)-3-methoxybenzene was used. The desired product **1i** was obtained as a white solid (368.9 mg, 32% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. R_f = 0.5 (PE / EA = 1 / 1, KMnO₄).

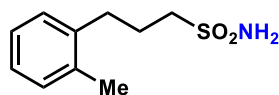
¹H NMR (400 MHz, CDCl₃) δ 7.22 (t, J = 7.8 Hz, 1H), 6.76 (dd, J = 8.1, 2.7 Hz, 2H), 6.73 (s, 1H), 4.71 (br s, 2H), 3.80 (s, 3H), 3.19 – 3.02 (m, 2H), 2.74 (t, J = 7.4 Hz, 2H), 2.22 – 2.16 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 159.9, 141.8, 129.8, 120.9, 114.4, 111.7, 55.3, 54.6, 34.1, 25.5; **HRMS** (ESI⁺) calc'd for C₁₀H₁₅NNaO₃S [M+Na]⁺: 252.0665, found 252.0662.



3-(3-Bromophenyl) propane-1-sulfonamide **1j**:

Prepared according to method B, 666.6 mg (2.4 mmol) of 1-bromo-3-(3-bromopropyl) benzene was used. The desired product **1j** was obtained as a white solid (252.2 mg, 38% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. R_f = 0.4 (PE / EA = 1 / 1, KMnO₄).

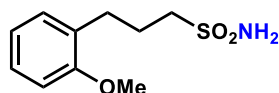
¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.32 (m, 2H), 7.20 – 7.16 (m, 1H), 7.12 (d, J = 7.6 Hz, 1H), 4.64 (br s, 2H), 3.16 – 3.03 (m, 2H), 2.75 (t, J = 7.5 Hz, 2H), 2.28 – 2.07 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 142.5, 131.6, 130.4, 129.8, 127.2, 122.8, 54.5, 33.8, 25.5; **HRMS** (ESI⁺) calc'd for C₉H₁₂BrNNaO₂S [M+Na]⁺: 299.9664, found 299.9662.



3-(2-Tolyl) propane-1-sulfonamide **1k**:

Prepared according to method B, 836.9 mg (4.0 mmol) of 1-(3-bromopropyl)-2-methylbenzene was used. The desired product **1k** was obtained as a white solid (337.4 mg, 40% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. R_f = 0.5 (PE / EA = 1 / 1, KMnO₄).

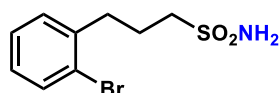
^1H NMR (400 MHz, CDCl_3) δ 7.19 – 7.08 (m, 4H), 4.71 (br s, 2H), 3.16 – 3.12 (m, 2H), 2.76 (t, $J = 7.6$ Hz, 2H), 2.32 (s, 3H), 2.21 – 2.03 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 138.4, 136.1, 130.6, 129.0, 126.7, 126.3, 54.9, 31.5, 24.3, 19.4; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{15}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 236.0716, found 236.0711.



3-(2-Methoxyphenyl) propane-1-sulfonamide **1l**:

Prepared according to method B, 337.2 mg (1.5 mmol) of 1-(3-bromopropyl)-2-methoxybenzene was used. The desired product **1l** was obtained as a white solid (139.1 mg, 40% yield) after flash column chromatography using PE / EA = 3 / 1 to 1 / 1 as eluent. $R_f = 0.5$ (PE / EA = 1 / 1, KMnO_4).

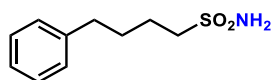
^1H NMR (400 MHz, CDCl_3) δ 7.26 – 7.18 (m, 1H), 7.12 (d, $J = 7.3$ Hz, 1H), 6.89 (dd, $J = 17.9$, 7.8 Hz, 2H), 4.70 (br s, 2H), 3.83 (s, 3H), 3.23 – 3.02 (m, 2H), 2.77 (t, $J = 7.3$ Hz, 2H), 2.12 – 2.07 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 157.5, 130.2, 128.5, 127.9, 120.7, 110.5, 55.4, 54.9, 28.8, 24.2; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{15}\text{NNaO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 252.0665, found 252.0667.



3-(2-Bromophenyl) propane-1-sulfonamide **1m**:

Prepared according to method A, 1.38 g (5.0 mmol) of 1-bromo-2-(3-bromopropyl) benzene was used. The desired product **1m** was obtained as a white solid (243.0 mg, 18% yield) after flash column chromatography using PE / EA = 2 / 1 to 1 / 1 as eluent. $R_f = 0.5$ (PE / EA = 1 / 1, KMnO_4).

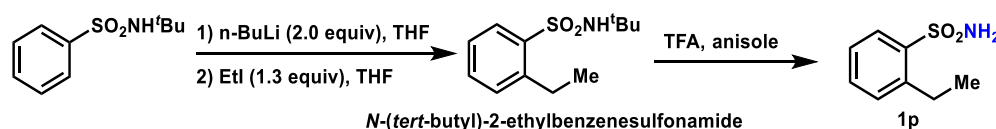
^1H NMR (400 MHz, CDCl_3) δ 7.55 (dd, $J = 8.0$, 1.0 Hz, 1H), 7.30 – 7.20 (m, 2H), 7.14 – 7.04 (m, 1H), 4.64 (br s, 2H), 3.22 – 3.09 (m, 2H), 2.91 (t, $J = 7.6$ Hz, 2H), 2.30 – 2.13 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 139.6, 133.2, 130.6, 128.4, 127.9, 124.5, 54.6, 34.4, 24.3; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_{12}\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 299.9664, found 299.9661.



4-Phenylbutane-1-sulfonamide **1n**:

Preparation according to method B, 216.5 mg (1.0 mmol) of 1-bromo-2-(3-bromopropyl) benzene was used. The desired product **1n** was obtained as a white solid (64.0 mg, 30% yield) after flash column chromatography using PE / EA = 5 / 1 to 1 / 1 as eluent. R_f = 0.4 (PE / EA = 1 / 1, KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.26 (m, 2H), 7.22 – 7.16 (m, 3H), 4.58 (br s, 2H), 3.28 – 3.04 (m, 2H), 2.66 (t, J = 7.5 Hz, 2H), 1.96 – 1.84 (m, 2H), 1.82 – 1.76 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 141.4, 128.6, 128.5, 126.2, 55.3, 35.5, 30.0, 23.7; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{15}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 236.0716, found 236.0719.

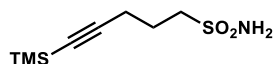


2-Ethylbenzenesulfonamide **1p**:⁴

To a 50 mL Schlenk tube were added *N*-(*tert*-butyl) benzenesulfonamide (0.64 g, 3.0 mmol, 1.0 equiv) and THF (8 mL) at -78°C , and then 2.5 mL of *n*-BuLi (2.4 M in hexane, 6 mmol, 2.0 equiv) was added dropwise. After stirring for 30 min, a solution of iodoethane (0.32 mL, 3.9 mmol, 1.3 equiv) in THF (2.0 mL) was added to the reaction mixture. After stirring for another 3 h, the mixture was quenched with sat. NH_4Cl aqueous solution (20 mL), and extracted with CH_2Cl_2 (3×20 mL). The combined organic layers were dried over Na_2SO_4 , filtered and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography (PE / EA = 10 / 1 as eluent) to give *N*-(*tert*-butyl)-2-ethylbenzenesulfonamide as a white solid (505.7 mg, 70% yield). R_f = 0.3 (PE / EA = 10 / 1, UV).

To a mixture of *N*-(*tert*-butyl)-2-ethylbenzenesulfonamide (505.7 mg) and anisole (7 mL, 3.0 equiv) was added $\text{CF}_3\text{CO}_2\text{H}$ (22.0 mL) and the mixture was stirred for 24 h at 0°C . After dilution with EA (30 mL), the solution was added sat. Na_2CO_3 aqueous solution at 0°C until pH = 13. The aqueous phase was extracted with EA (3×20 mL). The combined organic layers were dried over Na_2SO_4 , filtered and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography (PE to PE / EA = 10 / 1 as eluent) to give **1p** as a white solid (339.1 mg, 61% yield). R_f = 0.4 (PE / EA = 10 / 1, UV).

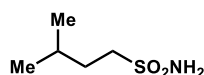
^1H NMR (400 MHz, CDCl_3) δ 8.01 (dd, J = 8.0, 0.9 Hz, 1H), 7.52 (dd, J = 7.6, 1.0 Hz, 1H), 7.40 (d, J = 7.6 Hz, 1H), 7.31 (t, J = 7.7 Hz, 1H), 4.87 (br s, 2H), 3.07 (t, J = 7.5 Hz, 2H), 1.33 (t, J = 7.5 Hz, 3H).



5-(Trimethylsilyl) pent-4-yne-1-sulfonamide **1q**:

Prepared according to method B, 327.2 mg (1.5 mmol) of (5-bromopent-1-yn-1-yl) trimethylsilane was used. The desired product **1q** was obtained as a white solid (142.3 mg, 43% yield) after flash column chromatography using PE / EA = 3 / 1 to 1 / 1 as eluent. R_f = 0.5 (PE / EA = 1 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 4.74 (br s, 2H), 3.36 – 3.15 (m, 2H), 2.43 (t, J = 6.8 Hz, 2H), 2.20 – 1.99 (m, 2H), 0.15 (s, 9H); **¹³C NMR** (100 MHz, CDCl₃) δ 104.5, 86.8, 54.3, 23.1, 18.7, 0.2; **HRMS** (ESI⁺) calc'd for C₈H₁₇NNaO₂SSi [M+Na]⁺: 242.0641, found 242.0642.

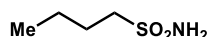


3-Methylbutane-1-sulfonamide **1r**:

Prepared according to method A, 1.19 g (8.0 mmol) of 1-bromo-3-methylbutane was used. The desired product **1r** was obtained as a colorless oil (315.1 mg, 26% yield) after flash column chromatography using PE / EA = 10 / 1 to 5 / 1 as eluent. R_f = 0.2 (PE / EA = 5 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 4.74 (br s, 2H), 3.29 – 2.95 (m, 2H), 1.95 – 1.57 (m, 3H), 0.95 (d, J = 6.3 Hz, 6H).

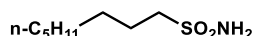
¹³C NMR (100 MHz, CDCl₃) δ 53.8, 32.6, 27.3, 22.3; **HRMS** (ESI⁺) calc'd for C₅H₁₃NNaO₂S [M+Na]⁺: 174.0559, found 174.0557.



Butane-1-sulfonamide **1s**:⁵

Prepared according to method A, 1.69 g (10.0 mmol) of butane-1-sulfonyl chloride was used. The desired product **1s** was obtained as a white solid (905.8 mg, 66% yield) after flash column chromatography using PE / EA = 3 / 1 to 2 / 1 as eluent. R_f = 0.5 (PE / EA = 1 / 1, KMnO₄).

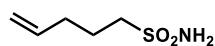
¹H NMR (400 MHz, CDCl₃) δ 4.98 (br s, 2H), 3.29 – 2.95 (m, 2H), 1.93 – 1.71 (m, 2H), 1.57 – 1.34 (m, 2H), 0.94 (t, J = 7.3 Hz, 3H).



Octane-1-sulfonamide **1t**:⁶

Prepared according to method A, 4.28 g (20.0 mmol) of octane-1-sulfonyl chloride was used. The desired product **1t** was obtained as a white solid (2.81 g, 73% yield) after flash column chromatography using PE / EA = 5 / 1 to 2 / 1 as eluent, $R_f = 0.5$ (PE / EA = 1 / 1, KMnO_4).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 4.78 (br s, 2H), 3.18 – 3.01 (m, 2H), 1.95 – 1.78 (m, 2H), 1.46 – 1.40 (m, 2H), 1.37 – 1.17 (m, 8H), 0.88 (t, $J = 6.8$ Hz, 3H).



Pent-4-ene-1-sulfonamide **1u**:⁷

Prepared according to method B, 1.49 g (10 mmol) of 5-bromopent-1-ene was used. The desired product **1u** was obtained as a colorless oil (632.1 mg, 42% yield) after flash column chromatography using PE / EA = 3 / 1 to 1 / 1 as eluent. $R_f = 0.3$ (PE / EA = 1 / 1, KMnO_4).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.81 – 5.71 (m, 1H), 5.15 – 5.01 (m, 2H), 4.89 (br s, 2H), 3.12 (dd, $J = 9.2, 6.6$ Hz, 2H), 2.21 (q, $J = 6.9$ Hz, 2H), 1.96 (dt, $J = 15.0, 7.3$ Hz, 2H).

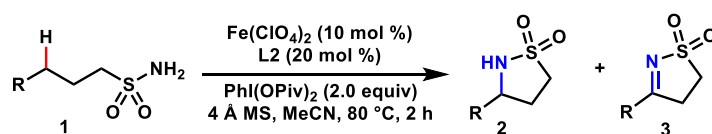


Methyl 4-sulfamoylbutanoate **1v**:⁸

Prepared according to method A, 904.5 mg (5 mmol) of methyl 4-bromobutanoate was used. The desired product **1v** was obtained as a colorless oil (96.9 mg, 11% yield) after flash column chromatography using PE / EA = 3 / 1 to 1 / 1 as eluent. $R_f = 0.3$ (PE / EA = 1 / 1, KMnO_4).

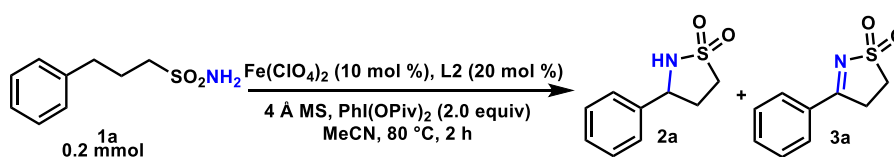
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.03 (br s, 2H), 3.69 (s, 3H), 3.27 – 3.10 (m, 2H), 2.53 (t, $J = 7.1$ Hz, 2H), 2.30 – 1.99 (m, 2H).

4. General Procedure for Fe-Catalyzed C–H Amidation and Characterization of Products

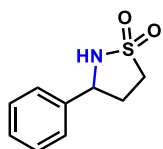


General procedure: In an argon-filled glovebox, 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 1.0 mL of MeCN were sequentially added to a 4 mL vial equipped with a magnetic stirring bar. After stirring at room temp. for 30 min, this solution was transferred to another 4 mL vial which contained 4 Å MS (50.0 mg), $\text{PhI}(\text{OPiv})_2$ (161.2 mg, 0.4 mmol, 2.0 equiv), substrate **1** (0.2 mmol), and then other 1.0 mL of MeCN was added.

After the vial was sealed and the mixture was stirred at 80 °C for 2 h, the vial was cooled to room temp. and removed from the glovebox, filtered and the filtrate was concentrated under reduced pressure. The residue was dissolved in CH₂Cl₂ (10 mL) and washed with sat. NaHCO₃ aqueous solution (5 mL). The aqueous layer was extracted with CH₂Cl₂ (3 × 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography (200 – 300 mesh SiO₂) to give the desired products.

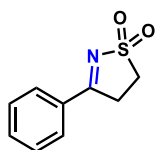


Following the general procedure, 39.7 mg of **1a** (0.2 mmol), 161.0 mg of PhI(OPiv)₂ (0.4 mmol), 4 Å MS (50.5 mg), 5.2 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave sultam **2a** (32.6 mg, 83% yield) and imine **3a** (2.4 mg, 6% yield).



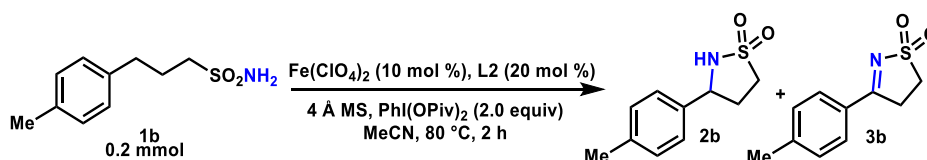
3-Phenylisothiazolidine 1,1-dioxide 2a:⁹ White solid; R_f = 0.3 (CH₂Cl₂, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.54 – 7.31 (m, 5H), 4.76 – 4.70 (m, 2H), 3.44 – 3.29 (m, 1H), 3.20 (m, *J* = 12.6, 10.6, 7.6 Hz, 1H), 2.80 – 2.73 (m, 1H), 2.45 – 2.34 (m, 1H).

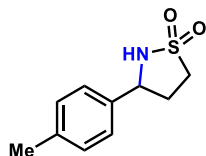


3-Phenyl-4,5-dihydroisothiazole 1,1-dioxide 3a:⁹ White solid; R_f = 0.4 (CH₂Cl₂, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 8.03 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.74 – 7.61 (m, 1H), 7.53 (t, *J* = 7.7 Hz, 2H), 3.75 – 3.61 (m, 2H), 3.56 – 3.35 (m, 2H).

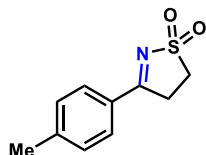


Following the general procedure, 42.3 mg of **1b** (0.2 mmol), 161.9 mg of PhI(OPiv)_2 (0.4 mmol), 4 Å MS (50.5 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **2b** (33.7 mg, 80% yield) and imine **3b** (4.7 mg, 11% yield).



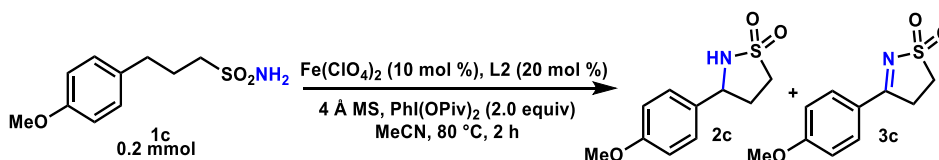
3-(4-Tolyl) isothiazolidine 1,1-dioxide 2b:⁹ White solid; R_f = 0.2 (CH_2Cl_2 , KMnO_4).

¹H NMR (400 MHz, CDCl_3) δ 7.29 (d, J = 8.1 Hz, 2H), 7.19 (d, J = 7.9 Hz, 2H), 4.70 (dt, J = 9.0, 5.9 Hz, 1H), 4.43 (br s, 1H), 3.36 (ddd, J = 12.1, 8.0, 3.8 Hz, 1H), 3.21 (ddd, J = 12.7, 10.3, 7.6 Hz, 1H), 2.75 (dtd, J = 11.0, 7.0, 3.8 Hz, 1H), 2.46 – 2.37 (m, 1H), 2.35 (s, 3H).

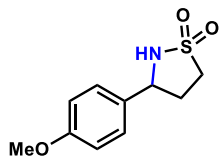


3-(4-Tolyl)-4,5-dihydroisothiazole 1,1-dioxide 3b:⁹ White solid; R_f = 0.3 (CH_2Cl_2 , KMnO_4).

¹H NMR (400 MHz, CDCl_3) δ 7.92 (d, J = 8.3 Hz, 2H), 7.32 (d, J = 8.3 Hz, 2H), 3.65 (dd, J = 8.2, 6.4 Hz, 2H), 3.43 (dd, J = 8.1, 6.5 Hz, 2H), 2.45 (s, 3H).



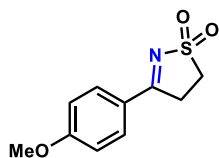
Following the general procedure, 45.9 mg of **1c** (0.2 mmol), 162.7 mg of PhI(OPiv)_2 (0.4 mmol), 4 Å MS (51.2 mg), 5.3 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **2c** (40.2 mg, 86% yield) and imine **3c** (5.7 mg, 11% yield).



3-(4-Methoxyphenyl) isothiazolidine 1,1-dioxide 2c: White solid; R_f = 0.2 (CH_2Cl_2 , KMnO_4).

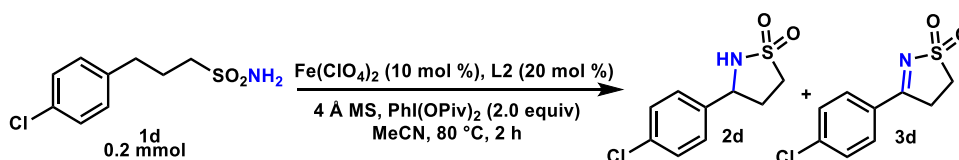
¹H NMR (400 MHz, CDCl_3) δ 7.32 (d, J = 8.8 Hz 2H), 6.90 (d, J = 8.8 Hz 2H), 4.79 – 4.63 (m, 1H), 4.50 (br s, 1H), 3.81 (s, 3H), 3.45 – 3.32 (m, 1H), 3.25 – 3.17 (m, 1H), 2.76 – 2.68

(m, 1H), 2.44 – 2.34 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.8, 132.0, 127.5, 114.5, 58.0, 55.5, 48.5, 32.4; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{13}\text{NNaO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 250.0508, found 250.0513.

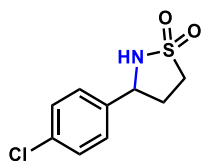


3-(4-Methoxyphenyl)-4,5-dihydroisothiazole 1,1-dioxide 3c: White solid; R_f = 0.3 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 8.9 Hz, 2H), 7.00 (d, J = 8.9 Hz, 2H), 3.90 (s, 3H), 3.74 – 3.58 (t, 2H), 3.49 – 3.35 (t, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 175.0, 164.9, 131.6, 123.5, 114.7, 55.8, 44.5, 33.4; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{11}\text{NNaO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 248.0352, found 248.0356.

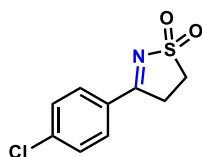


Following the general procedure, 46.6 mg of **1d** (0.2 mmol), 161.5 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.5 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **2d** (37.1 mg, 80% yield) and imine **3d** (2.8 mg, 6% yield).



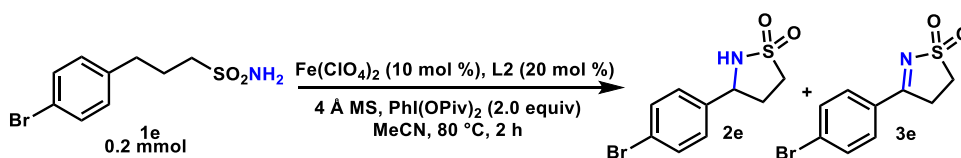
3-(4-Chlorophenyl) isothiazolidine 1,1-dioxide 2d: White solid; R_f = 0.2 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.34 – 7.32 (m, 4H), 4.71 (t, J = 6.6 Hz, 1H), 4.66 (br s, 1H), 3.36 – 3.30 (m, 1H), 3.22 – 3.14 (m, 1H), 2.79 – 2.71 (m, 1H), 2.38 – 2.27 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.9, 134.4, 129.3, 127.5, 57.6, 48.3, 32.3; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_{10}\text{ClNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 254.0013, found 254.0013.

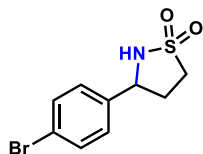


3-(4-Chlorophenyl)-4,5-dihydroisothiazole 1,1-dioxide 3d: White solid; $R_f = 0.3$ (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 8.6$ Hz, 2H), 7.51 (d, $J = 8.5$ Hz, 2H), 3.82 – 3.57 (t, 2H), 3.56 – 3.36 (t, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 174.8, 141.4, 130.5, 129.8, 129.4, 44.5, 33.6; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_8\text{ClNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 251.9856, found 251.9860.

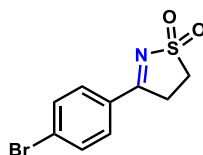


Following the general procedure, 55.5 mg of **1e** (0.2 mmol), 161.0 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (49.7 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / $\text{CH}_2\text{Cl}_2 = 1 / 1$ to CH_2Cl_2 , 200 – 300 mesh SiO_2) gave sultam **2e** (41.0 mg, 75% yield) and imine **3e** (4.4 mg, 8% yield).



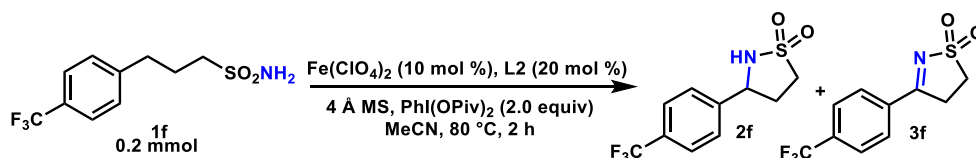
3-(4-Bromophenyl) isothiazolidine 1,1-dioxide 2e: White solid; $R_f = 0.2$ (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.50 (d, $J = 8.3$ Hz, 2H), 7.29 (d, $J = 8.4$ Hz, 2H), 4.87 – 4.59 (m, 2H), 3.35 (ddd, $J = 10.9, 7.6, 3.1$ Hz, 1H), 3.26 – 3.13 (m, 1H), 2.85 – 2.70 (m, 1H), 2.44 – 2.25 (m, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 139.5, 132.3, 127.9, 122.4, 57.6, 48.3, 32.2; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_{10}\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 297.9508, found 297.9512.

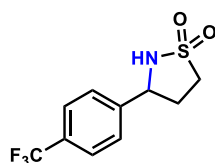


3-(4-Bromophenyl)-4,5-dihydroisothiazole 1,1-dioxide 3e: White solid; $R_f = 0.4$ (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.88 (d, $J = 8.6$ Hz, 2H), 7.68 (d, $J = 8.6$ Hz, 2H), 3.67 – 3.60 (t, 2H), 3.50 – 3.41 (t, 2H); **^{13}C NMR** (100 MHz, CDCl_3) δ 174.9, 132.8, 130.5, 130.1, 129.9, 44.4, 33.6; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_8\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 295.9351, found 295.9353.

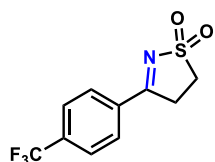


Following the general procedure, 53.3 mg of **1f** (0.2 mmol), 161.2 mg of PhI(OPiv)_2 (0.4 mmol), 4 Å MS (50.4 mg), 5.1 mg of $\text{Fe(ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.4 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2), R_f = 0.20 (CH_2Cl_2 , KMnO_4) gave sultam **2f** (46.0 mg, 88% yield) and imine **3f** (5.7 mg, 8% yield).

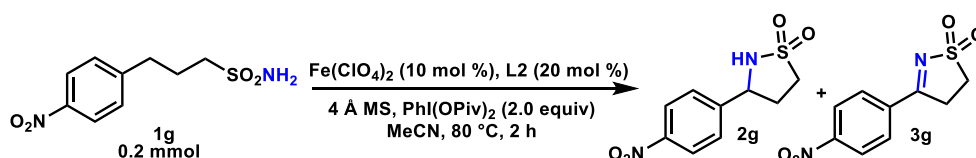


3-(4-(Trifluoromethyl) phenyl) isothiazolidine 1,1-dioxide 2f: White solid; R_f = 0.2 (CH_2Cl_2 , KMnO_4).

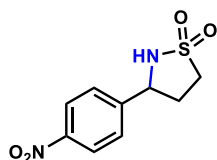
^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, J = 8.2 Hz, 2H), 7.54 (d, J = 8.2 Hz, 2H), 4.87 – 4.79 (m, 2H), 3.37 (ddd, J = 12.3, 7.5, 3.2 Hz, 1H), 3.27 – 3.14 (m, 1H), 2.84 – 2.80 (m, 1H), 2.47 – 2.26 (m, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 144.6 (q, J = 1.0 Hz), 130.8 (q, J = 32.4 Hz), 126.5, 126.1 (q, J = 3.8 Hz), 124.0 (q, J = 270.5 Hz), 57.6, 48.2, 32.1; **^{19}F NMR** (376 MHz, CDCl_3) δ –62.61 (s); **HRMS** (ESI⁺) calc'd for $\text{C}_{10}\text{H}_{10}\text{F}_3\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 288.0277, found 288.0282.



3-(4-(Trifluoromethyl) phenyl)-4,5-dihydroisothiazole 1,1-dioxide 3f: White solid; R_f = 0.4 (CH_2Cl_2 , KMnO_4), **^1H NMR** (400 MHz, CD_3CN) δ 8.21 (d, J = 8.2 Hz, 2H), 7.87 (d, J = 8.3 Hz, 2H), 3.75 (t, J = 7.9, 6.3 Hz, 2H), 3.53 – 3.32 (t, 2H); **^{13}C NMR** (100 MHz, CD_3CN) δ 177.7, 135.9 (q, J = 1.0 Hz), 135.1 (q, J = 32.5 Hz), 130.6, 126.9 (q, J = 3.8 Hz), 124.7 (q, J = 270.2 Hz), 45.3, 34.9; **^{19}F NMR** (376 MHz, CD_3CN) δ –63.77 (s); **HRMS** (ESI⁺) calc'd for $\text{C}_{10}\text{H}_8\text{F}_3\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 286.0120, found 286.0118.

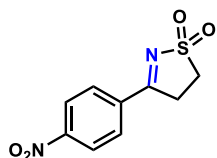


Following the general procedure, 48.6 mg of **1g** (0.2 mmol), 161.2 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.5 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.5 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **2g** (25.2 mg, 52% yield) and imine **3g** (11.1 mg, 23% yield).



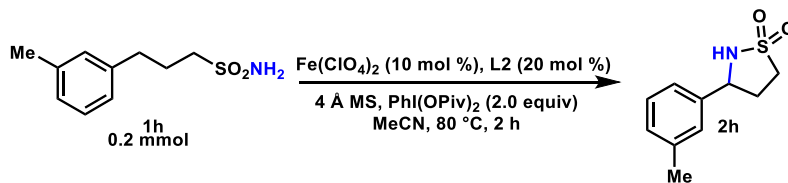
3-(4-Nitrophenyl) isothiazolidine 1,1-dioxide 2g: White solid; R_f = 0.2 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 8.25 (d, J = 8.7 Hz, 2H), 7.62 (d, J = 8.7 Hz, 2H), 4.85 (m, J = 7.9 Hz, 1H), 4.74 (br s, 1H), 3.40 (ddd, J = 12.3, 7.3, 2.7 Hz, 1H), 3.21 (td, J = 12.0, 7.6 Hz, 1H), 2.88 (ddd, J = 13.8, 7.1, 4.6 Hz, 1H), 2.50 – 2.25 (m, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 148.0, 147.9, 127.0, 124.5, 57.2, 48.2, 32.0; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_9\text{ClNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 265.0253, found 265.0254.



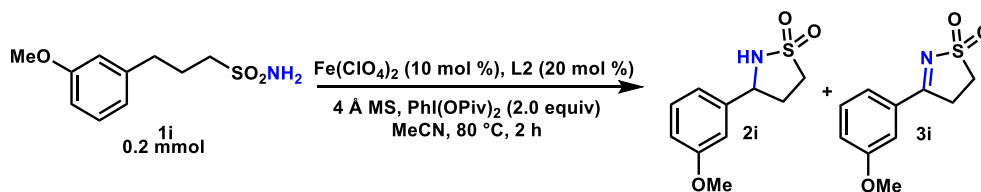
3-(4-Nitrophenyl)-4,5-dihydroisothiazole 1,1-dioxide 3g: White solid; R_f = 0.3 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CD_3CN) δ 8.42 – 8.28 (m, 2H), 8.28 – 8.15 (m, 2H), 3.76 (dd, J = 8.0, 6.3 Hz, 2H), 3.47 (dd, J = 7.9, 6.3 Hz, 2H); **^{13}C NMR** (100 MHz, CD_3CN) δ 177.2, 151.9, 137.5, 131.1, 124.9, 45.4, 35.1; **HRMS** (ESI+) calc'd for $\text{C}_9\text{H}_8\text{N}_2\text{NaO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 263.0097, found 263.0093.

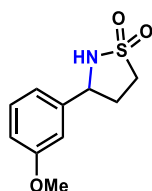


Following the general procedure, 42.6 mg of **1h** (0.2 mmol), 161.8 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.6 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / EA = 5 / 1 to CH_2Cl_2) gave **3-(3-tolyl) isothiazolidine 1,1-dioxide 2h⁹** (37.9 mg, 89% yield) as a white solid. R_f = 0.2 (CH_2Cl_2 , KMnO_4).

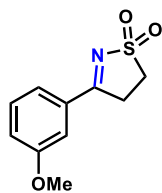
¹H NMR (400 MHz, CDCl₃) δ 7.30 – 7.25 (m, 1H), 7.24 – 7.12 (m, 3H), 4.72 – 4.68 (m, 1H), 4.51 (br s, 1H), 3.39 – 3.33 (m, 1H), 3.25 – 3.17 (m, 1H), 2.78 – 2.73 (m, 1H), 2.46 – 2.38 (m, 1H), 2.37 (s, 3H).



Following the general procedure, 46.0 mg of **1i** (0.2 mmol), 160.4 mg of PhI(OPiv)₂ (0.4 mmol), 4 Å MS (50.5 mg), 5.2 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave sultam **2i** (30.1 mg, 66% yield) and imine **3i** (6.9 mg, 15% yield).

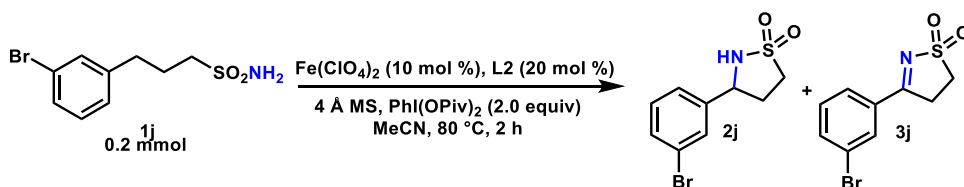


3-(3-Methoxyphenyl) isothiazolidine 1,1-dioxide 2i: White solid; *R*_f = 0.2 (CH₂Cl₂, KMnO₄). **¹H NMR** (400 MHz, CDCl₃) δ 7.30 (t, *J* = 7.9 Hz, 1H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.87 (d, *J* = 8.2 Hz, 1H), 4.75 – 4.67 (m, 1H), 4.55 (br s, 1H), 3.82 (s, 3H), 3.44 – 3.29 (m, 1H), 3.24 – 3.16 (m, 1H), 2.81 – 2.73 (m, 1H), 2.45 – 2.37 (m, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 160.2, 141.9, 130.3, 118.2, 114.0, 111.6, 58.2, 55.5, 48.2, 32.2; **HRMS** (ESI⁺) calc'd for C₁₀H₁₃NNaO₃S [M+Na]⁺: 250.0508, found 250.0513.

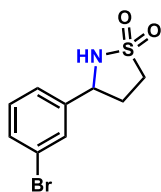


3-(3-Methoxyphenyl)-4,5-dihydroisothiazole 1,1-dioxide 3i: White solid; *R*_f = 0.4 (CH₂Cl₂, KMnO₄).

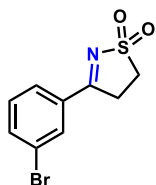
¹H NMR (400 MHz, CDCl₃) δ 7.63 – 7.58 (m, 1H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 7.19 (dd, *J* = 8.2, 2.4 Hz, 1H), 3.87 (s, 3H), 3.75 – 3.55 (m, 2H), 3.56 – 3.31 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 176.0, 160.1, 132.2, 130.2, 121.6, 121.4, 113.3, 55.7, 44.4, 33.7; **HRMS** (ESI⁺) calc'd for C₁₀H₁₁NNaO₃S [M+Na]⁺: 248.0352, found 248.0352.



Following the general procedure, 55.4 mg of **1j** (0.2 mmol), 161.3 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (51.2 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **2j** (41.1 mg, 74% yield) and imine **3j** (6.6 mg, 12% yield).

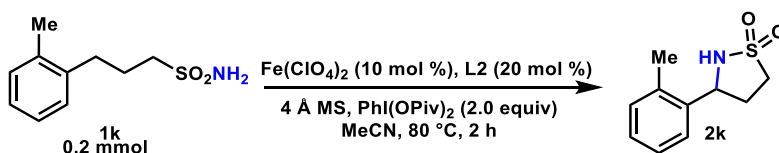


3-(3-Bromophenyl) isothiazolidine 1,1-dioxide 2j: White solid; R_f = 0.2 (CH_2Cl_2 , KMnO_4). ^1H NMR (400 MHz, CDCl_3) δ 7.56 (s, 1H), 7.46 (dd, J = 7.9, 1.0 Hz, 1H), 7.35 (d, J = 7.8 Hz, 1H), 7.30 – 7.21 (m, 1H), 4.73 – 4.65 (m, 2H), 3.43 – 3.31 (m, 1H), 3.19 (m, J = 12.5, 11.0, 7.6 Hz, 1H), 2.79 (m, J = 10.4, 7.3, 3.4 Hz, 1H), 2.49 – 2.28 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.7, 131.7, 130.8, 129.2, 124.8, 123.2, 57.5, 48.2, 32.1; HRMS (ESI⁺) calc'd for $\text{C}_9\text{H}_{10}\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 297.9508, found 297.9509.



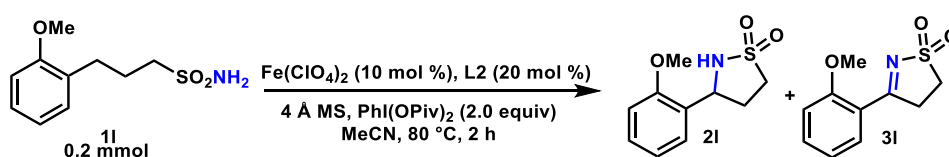
3-(3-Bromophenyl)-4,5-dihydroisothiazole 1,1-dioxide 3j: White solid; R_f = 0.5 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 8.16 (s, 1H), 8.05 – 7.90 (m, 1H), 7.80 – 7.77 (m, 1H), 7.44 – 7.40 (m, 1H), 3.64 (dd, J = 8.3, 6.2 Hz, 2H), 3.46 (dd, J = 8.1, 6.3 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 174.6, 137.5, 132.9, 132.1, 130.8, 127.7, 123.5, 44.4, 33.6; HRMS (ESI⁺) calc'd for $\text{C}_9\text{H}_8\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 295.9351, found 295.9347.

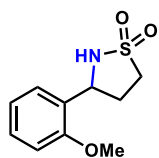


Following the general procedure, 42.5 mg of **1k** (0.2 mmol), 161.8 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.3 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.4 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **3-(2-tolyl) isothiazolidine 1,1-dioxide 2k**⁹ (34.2 mg, 81% yield) as a white solid. R_f = 0.2 (CH_2Cl_2 , KMnO_4).

¹H NMR (400 MHz, CDCl_3) δ 7.58 (d, J = 7.4 Hz, 1H), 7.30 – 7.23 (m, 1H), 7.21 (dd, J = 7.2, 1.2 Hz, 1H), 7.17 (d, J = 7.2 Hz, 1H), 4.97 (t, J = 6.8 Hz, 1H), 4.51 (br s, 1H), 3.35 (ddd, J = 12.3, 7.8, 4.3 Hz, 1H), 3.21 (ddd, J = 12.6, 9.8, 7.6 Hz, 1H), 2.78 (ddd, J = 11.6, 10.2, 5.8 Hz, 1H), 2.37 (s, 3H), 2.37 – 2.28 (m, 1H).

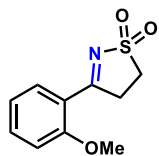


Following the general procedure, 45.6 mg of **1l** (0.2 mmol), 161.6 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (51.0 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.0 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave sultam **2l** (34.5 mg, 77% yield) and imine **3l** (2.7 mg, 3% yield).



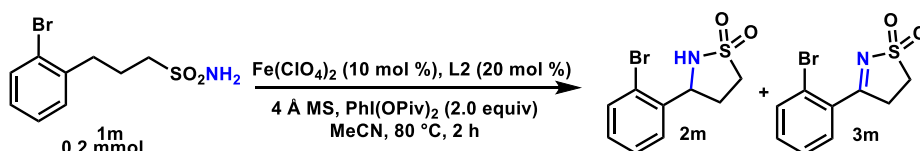
3-(2-Methoxyphenyl) isothiazolidine 1,1-dioxide 2l: White solid; R_f = 0.3 (CH_2Cl_2 , KMnO_4).

¹H NMR (400 MHz, CDCl_3) δ 7.45 (dd, J = 7.6, 1.5 Hz, 1H), 7.34 – 7.28 (m, 1H), 7.00 – 6.97 (m, 1H), 6.90 (d, J = 8.2 Hz, 1H), 4.96 (dd, J = 8.3, 7.0 Hz, 1H), 3.86 (s, 3H), 3.34 – 3.27 (m, 1H), 3.24 – 3.16 (m, 1H), 2.84 – 2.76 (m, 1H), 2.45 – 2.35 (m, 1H); **¹³C NMR** (100 MHz, CDCl_3) δ 156.6, 129.5, 127.5, 127.3, 121.2, 110.7, 55.5, 54.4, 48.2, 30.4; **HRMS** (ESI+) calc'd for $\text{C}_{10}\text{H}_{13}\text{NNaO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 250.0508, found 250.0505.

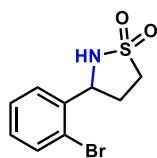


3-(2-Methoxyphenyl)-4,5-dihydroisothiazole 1,1-dioxide 3l: White solid; R_f = 0.4 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 8.15 (dd, $J = 7.9, 1.8$ Hz, 1H), 7.66 – 7.48 (m, 1H), 7.09 – 7.04 (m, 1H), 7.01 (d, $J = 8.5$ Hz, 1H), 3.93 (s, 3H), 3.82 (t, $J = 7.2$ Hz, 2H), 3.35 (t, $J = 6.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 178.0, 160.5, 135.8, 132.5, 121.4, 120.1, 112.0, 55.8, 44.2, 37.9; **HRMS** (ESI $^+$) calc'd for $\text{C}_{10}\text{H}_{11}\text{NNaO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 248.0352, found 248.0352.

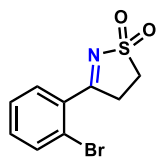


Following the general procedure, 55.2 mg of **1m** (0.2 mmol), 161.8 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.2 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.0 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / $\text{CH}_2\text{Cl}_2 = 1 / 1$ to CH_2Cl_2) gave sultam **2m** (33.7 mg, 61% yield) and imine **3m** (3.1 mg, 6% yield).



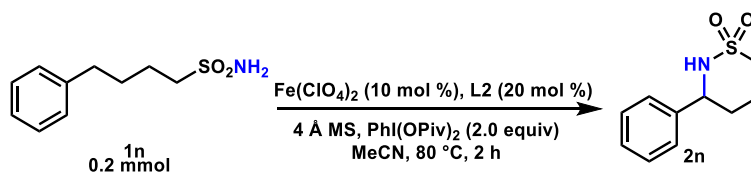
3-(2-Bromophenyl) isothiazolidine 1,1-dioxide 2m: White solid; $R_f = 0.4$ (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.73 (dd, $J = 7.8, 1.6$ Hz, 1H), 7.54 (dd, $J = 8.0, 1.1$ Hz, 1H), 7.42 – 7.34 (m, 1H), 7.18 (td, $J = 7.7, 1.7$ Hz, 1H), 5.16 (dd, $J = 14.8, 6.7$ Hz, 1H), 4.70 (br s, 1H), 3.33 (ddd, $J = 12.1, 7.1, 3.7$ Hz, 1H), 3.23 – 3.12 (m, 1H), 3.06 – 2.98 (m, 1H), 2.30 – 2.20 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 139.7, 133.1, 129.8, 128.5, 127.6, 121.8, 57.2, 48.0, 30.1; **HRMS** (ESI $^+$) calc'd for $\text{C}_9\text{H}_9\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 297.9508, found 297.9505.



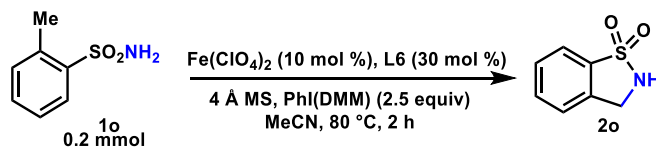
3-(2-Bromophenyl)-4,5-dihydroisothiazole 1,1-dioxide 3m: White solid; $R_f = 0.6$ (CH_2Cl_2 , UV).

^1H NMR (400 MHz, CDCl_3) δ 7.71 (dd, $J = 7.8, 1.3$ Hz, 1H), 7.66 (dd, $J = 7.6, 1.9$ Hz, 1H), 7.46 (dd, $J = 7.5, 1.4$ Hz, 1H), 7.43 – 7.38 (m, 1H), 3.94 – 3.68 (t, 2H), 3.45 (t, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 178.9, 134.6, 133.6, 133.3, 131.0, 128.0, 121.2, 44.7, 37.4; **HRMS** (ESI $^+$) calc'd for $\text{C}_9\text{H}_8\text{BrNNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 295.9351, found 295.9350.



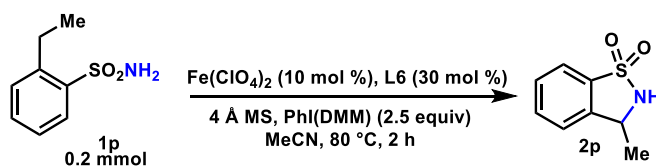
Following the general procedure, 40.7 mg of **1n** (0.19 mmol), 163.8 mg of PhI(OPiv)₂ (0.4 mmol), 4 Å MS (51.1 mg), 5.2 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.1 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave **3-phenyl-1,2-thiazinane 1,1-dioxide 2n**¹⁰ (28.1 mg, 70% yield) as a white solid; *R*_f = 0.2 (CH₂Cl₂, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.43 – 7.29 (m, 5H), 4.59 (dd, *J* = 11.9, 3.4 Hz, 1H), 4.24 (br s, 1H), 3.33 – 3.19 (m, 1H), 3.01 (td, *J* = 13.1, 5.2 Hz, 1H), 2.43 – 2.23 (m, 2H), 2.06 (ddd, *J* = 13.9, 5.9, 3.0 Hz, 1H), 1.73 (ddd, *J* = 17.1, 13.0, 8.5 Hz, 1H).



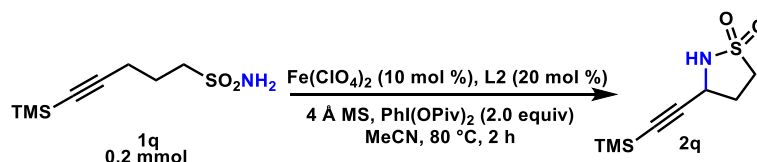
Following the general procedure, 34.4 mg of **1o** (Adamas, 0.2 mmol), 163.4 mg of PhI(DMM) (0.5 mmol), 4 Å MS (50.6 mg), 5.3 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 9.3 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave **2,3-dihydrobenzo[d]isothiazole 1,1-dioxide 2o**¹¹ (16.8 mg, 50% yield) as a white solid; *R*_f = 0.3 (CH₂Cl₂, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.80 (d, *J* = 7.8 Hz, 1H), 7.62 (td, *J* = 7.6, 1.1 Hz, 1H), 7.56 – 7.51 (m, 1H), 7.40 (d, *J* = 7.7 Hz, 1H), 4.81 (br s, 1H), 4.54 (s, 2H).



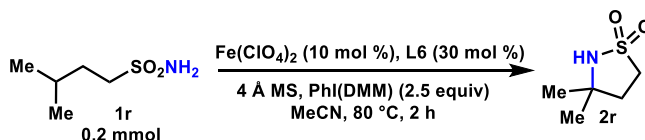
Following the general procedure, 37.2 mg of **1p** (0.2 mmol), 167.0 mg of PhI(DMM) (0.5 mmol), 4 Å MS (50.2 mg), 5.3 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 9.3 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave **3-methyl-2,3-dihydrobenzo[d]isothiazole 1,1-dioxide 2p**⁹ (18.9 mg, 51% yield) as a white solid; *R*_f = 0.3 (CH₂Cl₂, KMnO₄).

^1H NMR (400 MHz, CDCl_3) δ 7.78 (d, J = 7.7 Hz, 1H), 7.64 (t, J = 7.5 Hz, 1H), 7.54 (t, J = 7.4 Hz, 1H), 7.40 (d, J = 7.7 Hz, 1H), 4.82–4.78 (m, 1H), 1.63 (d, J = 6.4 Hz, 3H).



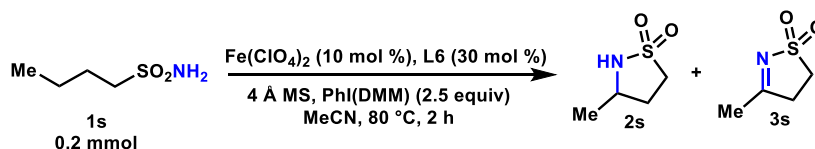
Following the general procedure, 44.0 mg of **1q** (0.2 mmol), 162.5 mg of PhI(OPiv)_2 (0.4 mmol), 4 Å MS (51.0 mg), 5.3 mg of $\text{Fe(ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave **3-((trimethylsilyl)ethynyl)isothiazolidine 1,1-dioxide 2q** (23.3 mg, 53% yield) as a yellow solid; R_f = 0.3 (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 4.59–4.22 (m, 2H), 3.31–3.20 (m, 1H), 3.19–3.14 (m, 1H), 2.86–2.66 (m, 1H), 2.61–2.40 (m, 1H), 0.17 (s, 9H); **^{13}C NMR** (100 MHz, CDCl_3) δ 102.0, 91.2, 46.7, 46.0, 30.9, -0.21; **HRMS** (ESI⁺) calc'd for $\text{C}_8\text{H}_{15}\text{NNaO}_2\text{SSi}$ [$\text{M}+\text{Na}$]⁺: 240.0485, found 240.0489.

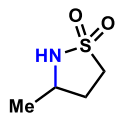


Following the general procedure, 30.1 mg of **1r** (0.2 mmol), 162.7 mg of PhI(DMM) (0.5 mmol), 4 Å MS (51.2 mg), 5.3 mg of $\text{Fe(ClO}_4)_2$ (0.02 mmol, 10 mol %), 10.3 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to PE / EA = 2 / 1) gave **3,3-dimethylisothiazolidine 1,1-dioxide 2r** (20.3 mg, 68% yield) as a white solid; R_f = 0.3 (PE / EA = 2 / 1, KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 4.06 (br s, 1H), 3.23 (t, J = 7.5 Hz, 2H), 2.29 (t, J = 7.5 Hz, 2H), 1.40 (s, 6H); **^{13}C NMR** (100 MHz, CDCl_3) δ 57.9, 48.3, 37.0, 29.7; **HRMS** (ESI⁺) calc'd for $\text{C}_5\text{H}_{11}\text{NNaO}_2\text{S}$ [$\text{M}+\text{Na}$]⁺: 172.0403, found 172.0404.

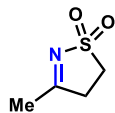


Following the general procedure, 27.8 mg of **1s** (0.2 mmol), 161.2 mg of PhI(DMM) (0.5 mmol), 4 Å MS (50.4 mg), 5.8 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 9.8 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to PE / EA = 2 / 1) gave sultam **2s** (20.1 mg, 74% yield) and imine **3s** (2.3 mg, 8% yield).



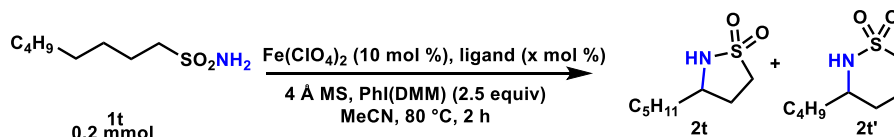
3-Methylisothiazolidine 1,1-dioxide 2s:⁹ Colorless oil; R_f = 0.2 (PE / EA = 2 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 4.04 (br s, 1H), 3.76 – 3.68 (m, 1H), 3.26 – 3.20 (m, 1H), 3.17 – 3.09 (m, 1H), 2.55 – 2.48 (m, 1H), 2.09 – 1.99 (m, 1H), 1.33 (d, J = 6.3 Hz, 3H).



3-Methyl-4,5-dihydroisothiazole 1,1-dioxide 3s:⁹ Colorless oil; R_f = 0.5 (PE / EA = 2 / 1, KMnO₄).

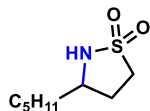
¹H NMR (400 MHz, CDCl₃) δ 3.31 – 3.24 (t, 2H), 3.23 – 3.16 (t, 2H), 2.34 (s, 3H).



For conditions A: Following the general procedure, 38.6 mg of **1t** (0.2 mmol), 162.5 mg of PhI(DMM) (0.5 mmol), 4 Å MS (53.2 mg), 5.1 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 11.3 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave an unseparated mixture of **2t** and **2t'** (28.6 mg, 75% yield, **2t** / **2t'** = 2 / 1).

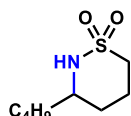
For conditions B: Following the general procedure, 38.6 mg of **1t** (0.2 mmol), 167.1 mg of PhI(DMM) (0.5 mmol), 4 Å MS (50.2 mg), 5.1 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.1 mg of **L1** (0.06 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave an unseparated mixture of **2t** and **2t'** (32.6 mg, 85% yield, **2t** / **2t'** = 3 / 5).

*Note: Treatment of the mixture (**2t** and **2t'**) with PIFA led to the oxidation of **2t** to the corresponding imine and recovered analytic pure **2t'** which was used for characterization. The characterization data of **2t** were collected as a mixture with **2t'**.*



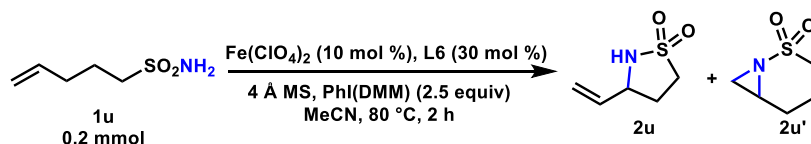
3-Pentylisothiazolidine 1,1-dioxide 2t: $R_f = 0.4$ (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 4.31 (br s, 1H), 3.61 – 3.53 (m, 1H), 3.26 – 3.15 (m, 1H), 3.13 – 3.06 (m, 1H), 2.59 – 2.43 (m, 1H), 2.12 – 1.97 (m, 1H), 1.64 – 1.53 (m, 2H), 1.42 – 1.15 (m, 6H), 0.89 (t, $J = 6.8$ Hz, 3H); **^{13}C NMR** (100 MHz, CDCl_3) δ 55.4, 48.2, 36.1, 31.6, 30.0, 25.9, 22.6, 14.1; **HRMS** (ESI+) calc'd for $\text{C}_8\text{H}_{17}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 214.0872, found 214.0873.

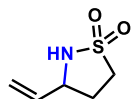


3-Butyl-1,2-thiazinane 1,1-dioxide 2t':¹⁰ Colorless oil; $R_f = 0.4$ (CH_2Cl_2 , KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 3.74 (br s, 1H), 3.50 – 3.43 (m, 1H), 3.20 (dt, $J = 13.3, 3.5$ Hz, 1H), 2.98 – 2.72 (m, 1H), 2.28 – 2.12 (m, 2H), 1.84 – 1.80 (m, 1H), 1.52 – 1.14 (m, 7H), 0.90 (t, $J = 7.0$ Hz, 3H); **^{13}C NMR** (100 MHz, CDCl_3) δ 56.8, 49.6, 35.6, 30.7, 27.6, 23.2, 22.5, 14.1.

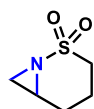


Following the general procedure, 30.1 mg of **1u** (0.2 mmol), 167.1 mg of $\text{PhI}(\text{DMM})$ (0.5 mmol), 4 Å MS (50.4 mg), 5.3 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 9.8 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / $\text{CH}_2\text{Cl}_2 = 1 / 1$ to PE / EA = 1 / 1) gave sultam **2u** (10.8 mg, 36% yield) and aziridine product **2u'** (7.4 mg, 25% yield).



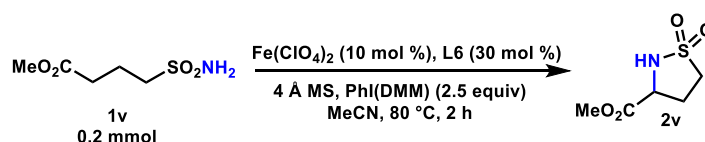
3-Vinylisothiazolidine 1,1-dioxide 2u:¹² Colorless oil; $R_f = 0.2$ (PE / EA = 1 / 1, KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 5.88 – 5.80 (m, 1H), 5.34 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.24 (dt, $J = 10.2, 1.0$ Hz, 1H), 4.28 (br s, 1H), 4.23 – 4.03 (m, 1H), 3.26 – 3.09 (m, 2H), 2.63 – 2.55 (m, 1H), 2.28 – 2.18 (m, 1H).



2-Thia-1-azabicyclo[4.1.0]heptane 2,2-dioxide **2u'**:⁷ yellow solid; R_f = 0.3 (PE / EA = 1 / 1, KMnO_4).

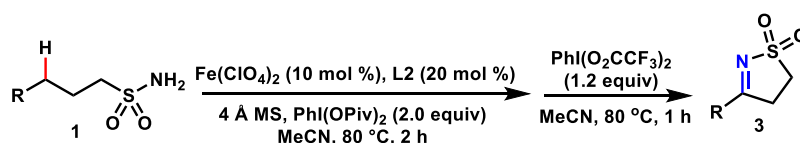
^1H NMR (400 MHz, CDCl_3) δ 3.30 – 3.13 (m, 2H), 3.02 (ddd, J = 14.8, 11.6, 4.3 Hz, 1H), 2.67 (d, J = 5.1 Hz, 1H), 2.57 (d, J = 5.2 Hz, 1H), 2.23 – 2.12 (m, 2H), 2.10 (dt, J = 15.3, 5.1 Hz, 1H), 1.98 – 1.88 (m, 1H).



Following the general procedure, 36.1 mg of **1v** (0.2 mmol), 166.7 mg of $\text{PhI}(\text{DMM})$ (0.5 mmol), 4 Å MS (51.4 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 9.7 mg of **L6** (0.06 mmol, 30 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to PE / EA = 1 / 1) gave **methyl isothiazolidine-3-carboxylate 1,1-dioxide **2v**** (19.3 mg, 54% yield) as a colorless oil; R_f = 0.4 (PE / EA = 1 / 1, KMnO_4).

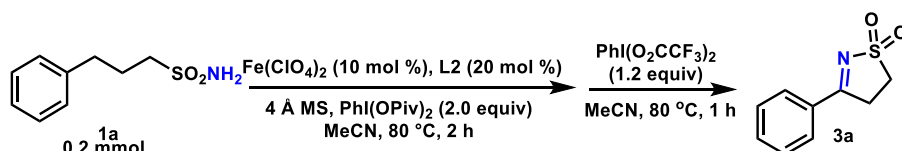
^1H NMR (400 MHz, CDCl_3) δ 4.95 (br s, 1H), 4.20 (dt, J = 8.3, 4.7 Hz, 1H), 3.85 (s, 3H), 3.21 – 3.15 (m, 1H), 3.01 – 2.94 (m, 1H), 2.87 – 2.77 (m, 1H), 2.64 – 2.56 (m, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 171.4, 54.3, 53.6, 45.9, 26.9; **HRMS** (ESI⁺) calc'd for $\text{C}_5\text{H}_9\text{NNaO}_4\text{S}$ [$\text{M}+\text{Na}$]⁺: 202.0144, found 202.0144.

5. General Procedure for One-pot Synthesis of Cyclic *N*-Sulfonyl Imines

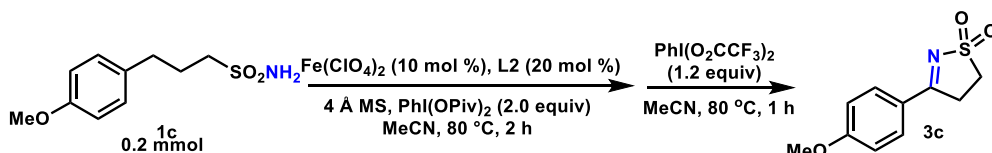


General procedure: In an argon-filled glovebox, 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 1.0 mL of MeCN were sequentially added to a 4 mL vial equipped with a magnetic stirring bar. After stirring at room temp. for 30 min, this catalyst solution was transferred to another 4 mL vial which contained 4 Å MS (50.0 mg), 161.2 mg of $\text{PhI}(\text{OPiv})_2$ (2.0 equiv), substrate (0.2 mmol), and other 1.0 mL of MeCN. Then the vial was sealed and the mixture was stirred at 80 °C. After 2 h, the vial was removed from the glovebox. To this vial was added 103.2 mg of $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ (1.2 equiv) and stirred at 80 °C for another 1 h. The mixture was filtered, and the filtrate was concentrated under reduced pressure. The residue was dissolved in CH_2Cl_2 (10 mL) and washed with sat. NaHCO_3 aqueous solution (5

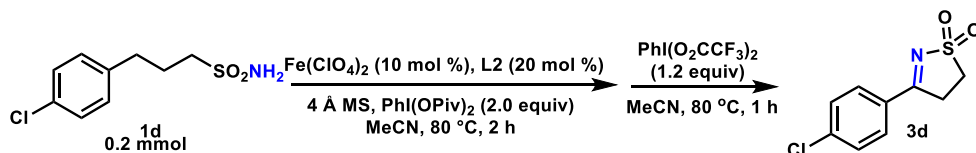
mL). The aqueous layer was extracted with CH_2Cl_2 (3×10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (200 – 300 mesh SiO_2) to give the desired product.



Following the general procedure, 40.1 mg of **1a** (0.2 mmol), 160.6 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.3 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 7.9 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.0 mg of $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave imine **3a** (34.1 mg, 88% yield) as a white solid. R_f = 0.4 (CH_2Cl_2 , KMnO_4). See page SI-15 for characterization data.

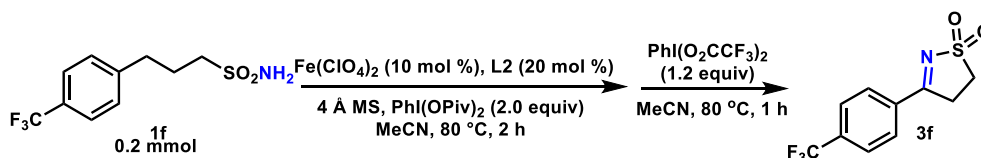


Following the general procedure, 45.6 mg of **1c** (0.2 mmol), 160.1 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.6 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.5 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.0 mg of $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave imine **3c** (35.2 mg, 78% yield) as a white solid. R_f = 0.4 (CH_2Cl_2 , KMnO_4). See page SI-16 for characterization data.

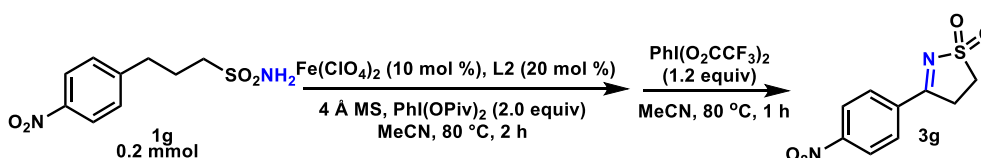


Following the general procedure, 46.7 mg of **1d** (0.2 mmol), 160.3 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.0 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.5 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.1 mg of $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column

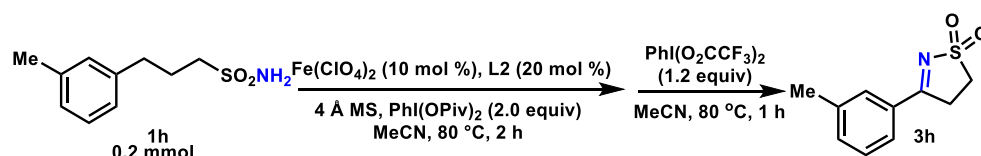
chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave imine **3d** (39.0 mg, 85% yield) as a white solid. R_f = 0.3 (CH₂Cl₂, KMnO₄). See page SI-18 for characterization data.



Following the general procedure, 53.3 mg of **1f** (0.2 mmol), 161.7 mg of PhI(OPiv)₂ (0.4 mmol), 4 Å MS (49.9 mg), 5.2 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.5 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.5 mg of PhI(O₂CCF₃)₂ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave imine **3f** (48.4 mg, 92% yield) as a white solid. R_f = 0.4 (CH₂Cl₂, KMnO₄). See page SI-19 for characterization data.

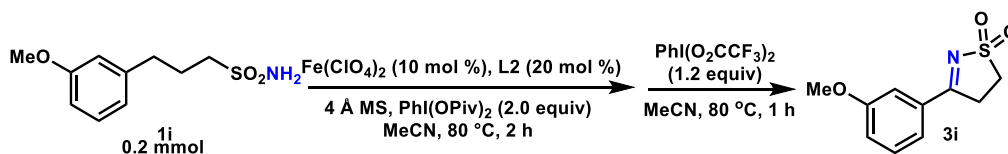


Following the general procedure, 48.6 mg of **1g** (0.2 mmol), 161.5 mg of PhI(OPiv)₂ (0.4 mmol), 4 Å MS (50.1 mg), 5.3 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.6 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.5 mg of PhI(O₂CCF₃)₂ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave imine **3g** (36.1 mg, 75% yield) as a white solid. R_f = 0.3 (CH₂Cl₂, KMnO₄). See page SI-20 for characterization data.

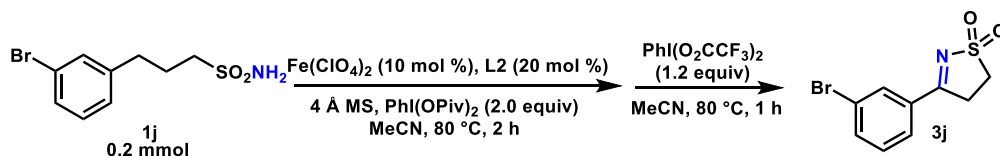


Following the general procedure, 42.6 mg of **1h** (0.2 mmol), 161.8 mg of PhI(OPiv)₂ (0.4 mmol), 4 Å MS (50.6 mg), 5.2 mg of Fe(ClO₄)₂ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.1 mg of PhI(O₂CCF₃)₂ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) gave **3-(3-tolyl)-4,5-dihydroisothiazole 1,1-dioxide 3h⁹** (37.1 mg, 89% yield) as a white solid. R_f = 0.4 (CH₂Cl₂, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.87 (s, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.43 (dt, J = 15.2, 7.6 Hz, 2H), 3.71 – 3.61 (m, 2H), 3.49 – 3.37 (m, 2H), 2.43 (s, 3H).

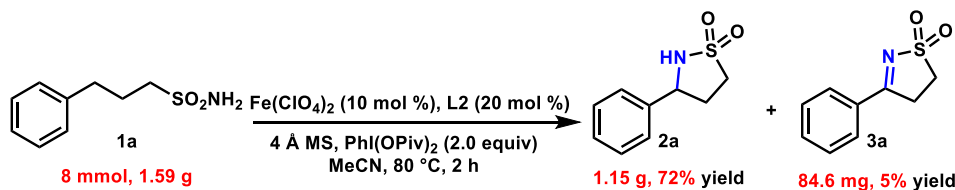


Following the general procedure, 45.8 mg of **1i** (0.2 mmol), 161.6 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.4 mg), 5.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.1 mg of $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave imine **3i** (36.3 mg, 81% yield) as a white solid. R_f = 0.3 (CH_2Cl_2 , KMnO_4). See page SI-21 for characterization data.



Following the general procedure, 55.1 mg of **1j** (0.2 mmol), 161.3 mg of $\text{PhI}(\text{OPiv})_2$ (0.4 mmol), 4 Å MS (50.8 mg), 5.1 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.02 mmol, 10 mol %), 8.3 mg of **L2** (0.04 mmol, 20 mol %) and 2.0 mL of MeCN were used (80 °C for 2 h); then 103.1 mg of $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ (0.24 mmol, 1.2 equiv) was added (80 °C for 1 h). Purification by flash column chromatography (PE / CH_2Cl_2 = 1 / 1 to CH_2Cl_2) gave product **3j** (42.5 mg, 78% yield) as a white solid. R_f = 0.5 (CH_2Cl_2 , KMnO_4). See page SI-22 for characterization data.

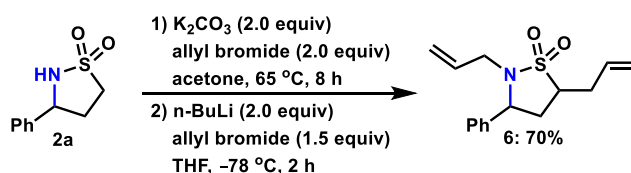
6. Gram-Scale Synthesis of **2a**



In an argon-filled glovebox, 203.2 mg of $\text{Fe}(\text{ClO}_4)_2$ (0.8 mmol, 10 mol %), 340.8 mg of **L2** (1.6 mmol, 20 mol %) and 10 mL of MeCN were sequentially added to a 20 mL vial equipped with a magnetic stirring bar. After stirring at room temp. for 30 min, this catalyst solution was transferred to a 250 mL Schlenk flask containing a stirring bar, 4 Å MS (2.01 g), **1a** (8.0 mmol, 1.59 g), and $\text{PhI}(\text{OPiv})_2$ (6.45 g, 2.0 equiv). Then 40 mL MeCN was injected. After stirring at

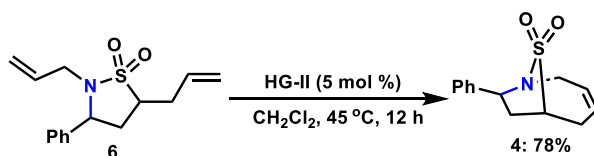
the 80 °C for 2 h, the mixture was filtered, washed with CH₂Cl₂ (50 mL) and the filtrate was concentrated under reduced pressure. The residue was dissolved in CH₂Cl₂ (50 mL) and washed with sat. NaHCO₃ aqueous solution (30 mL) and CH₂Cl₂ (50 mL) sequentially. The combined solutions were separated and the aqueous layer was extracted with CH₂Cl₂ (3 × 50 mL). The organic layers were combined, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography (PE / CH₂Cl₂ = 1 / 1 to CH₂Cl₂) to give the sultam **2a** (1.15 g, 72% yield) together with imine **3a** (84.6 mg, 5% yield).

7. Synthesis of [4.2.1]-Bridged Bicyclic Sultam **4** and [6.5]-Bicyclic Sultam **5**



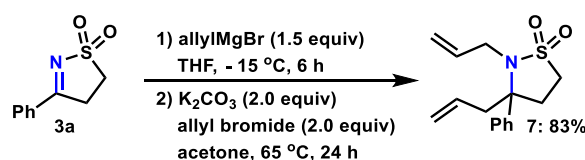
To a 25 mL Schlenk flask was charged with a K₂CO₃ (58.1 mg, 2.0 equiv), **2a** (39.6 mg, 0.2 mmol), and acetone (2.0 mL). The mixture was heated to 65 °C, and allyl bromide (49.6 mg, 2.0 equiv) was added dropwise. After stirring at 65 °C for 8 h, the mixture was filtered through a pad of celite, washed with EA, then concentrated under reduced pressure. The crude mixture was directly used in the next step without further purification. To a solution of the crude mixture in THF (2.0 mL) was added n-BuLi (0.18 mL, 2.4 M in hexane) at -78 °C. The mixture was stirred for 0.5 h and allyl bromide (38.2 mg, 1.5 equiv) was added dropwise. The reaction was slowly warmed to room temp. and then stirred for another 2 h. Quenched with sat. NH₄Cl aqueous solution (2.0 mL), the mixture was extracted with CH₂Cl₂ (3 × 5.0 mL). The organic layers were combined, washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The mixture was purified by column chromatography (PE / EA = 10 / 1) to give the double allylation product **2,5-diallyl-3-phenylisothiazolidine 1,1-dioxide 6** (39.0 mg, 70% yield) as a colorless oil. R_f = 0.5 (PE / EA = 5 / 1, KMnO₄).

¹H NMR (400 MHz, CDCl₃) δ 7.40 – 7.31 (m, 5H), 5.93 – 5.64 (m, 2H), 5.28 – 5.03 (m, 4H), 4.37 (dd, *J* = 7.6, 6.1 Hz, 1H), 3.94 (dd, *J* = 15.5, 4.8 Hz, 1H), 3.48 – 3.23 (m, 2H), 2.78 (dt, *J* = 13.1, 5.9 Hz, 1H), 2.44 (dt, *J* = 13.7, 7.6 Hz, 1H), 2.37 – 2.21 (m, 2H); **¹³C NMR** (100 MHz, CDCl₃) δ 140.1, 132.7, 132.3, 129.2, 128.4, 126.8, 120.0, 119.1, 58.0, 55.3, 45.2, 34.3, 32.3; **HRMS** (ESI⁺) calc'd for C₁₅H₁₉NNaO₂S [M+Na]⁺: 300.1029, found 300.1030.



To a solution of compound **6** (10.4 mg, 0.037 mmol) in CH_2Cl_2 (10.0 mL) was added Hoveyda-Grubbs second generation catalyst (1.7 mg, 5 mol %) and the mixture was stirred for 12 h at 45 °C. Then the reaction was cooled to room temp. and the solvent was removed under reduced pressure. The mixture was purified by flash column chromatography (PE / EA = 5 / 1) to give **8-phenyl-9-thia-1-azabicyclo[4.2.1]non-3-ene 9,9-dioxide 4** (7.3 mg, 78% yield) as a white solid. R_f = 0.3 (PE / EA = 5 / 1, KMnO_4)

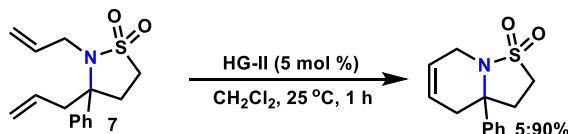
^1H NMR (400 MHz, CDCl_3) δ 7.46 (d, J = 7.5 Hz, 2H), 7.36 – 7.33 (m, 2H), 7.31 – 7.24 (m, 1H), 5.88 – 5.70 (m, 1H), 5.59 – 5.52 (m, 1H), 4.46 (t, J = 9.3 Hz, 1H), 4.26 – 4.04 (m, 1H), 3.73 (d, J = 3.8 Hz, 1H), 3.59 (dd, J = 18.7, 4.4 Hz, 1H), 2.76 (d, J = 18.5 Hz, 1H), 2.64 – 2.47 (m, 2H), 2.39 (dd, J = 18.6, 3.3 Hz, 1H); **^{13}C NMR** (100 MHz, CDCl_3) δ 142.0, 128.8, 127.7, 127.3, 127.0, 126.3, 61.6, 57.3, 51.2, 35.9, 29.1; **HRMS** (ESI+) calc'd for $\text{C}_{13}\text{H}_{15}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 272.0716, found 272.0716.



To a solution of **3a** (38.8 mg, 0.2 mmol) in THF (2.0 mL) at –15 °C was added allylmagnesium bromide (0.3 mL, 1 M in THF). The mixture was stirred for 6 h and quenched by sat. NH_4Cl aqueous solution (2.0 mL). The mixture was extracted with Et_2O (3×5.0 mL). The organic layers were combined, washed with brine, dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure. The crude mixture was directly used in the next step. A solution of the crude mixture in acetone (2.0 mL) was added K_2CO_3 (57.1 mg, 2.0 equiv) and heated to 65 °C, then 3-bromoprop-1-ene (48.6 mg, 2.0 equiv) was added dropwise. After stirring at 65 °C for 24 h, the mixture was filtered through a pad of celite, and the filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography (PE / EA = 5 / 1) to give **2,3-diallyl-3-phenylisothiazolidine 1,1-dioxide 7** (45.8 mg, 83% yield) as a colorless oil. R_f = 0.3 (PE / EA = 5 / 1, KMnO_4).

^1H NMR (400 MHz, CDCl_3) δ 7.49 – 7.37 (m, 4H), 7.33 – 7.29 (m, 1H), 6.08 – 5.95 (m, 1H), 5.95 – 5.84 (m, 1H), 5.32 – 5.25 (m, 2H), 5.17 – 5.08 (m, 2H), 3.90 (dd, J = 15.9, 5.9 Hz, 1H), 3.37 (dd, J = 15.9, 7.2 Hz, 1H), 3.28 – 3.06 (m, 2H), 2.90 (d, J = 7.3 Hz, 2H), 2.69 – 2.61 (m,

1H), 2.36 – 2.29 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.1, 134.3, 133.0, 129.1, 128.0, 126.5, 120.0, 118.4, 68.0, 45.7, 44.3, 41.9, 33.7; HRMS (ESI+) calc'd for $\text{C}_{15}\text{H}_{19}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 300.1029, found 300.1025.

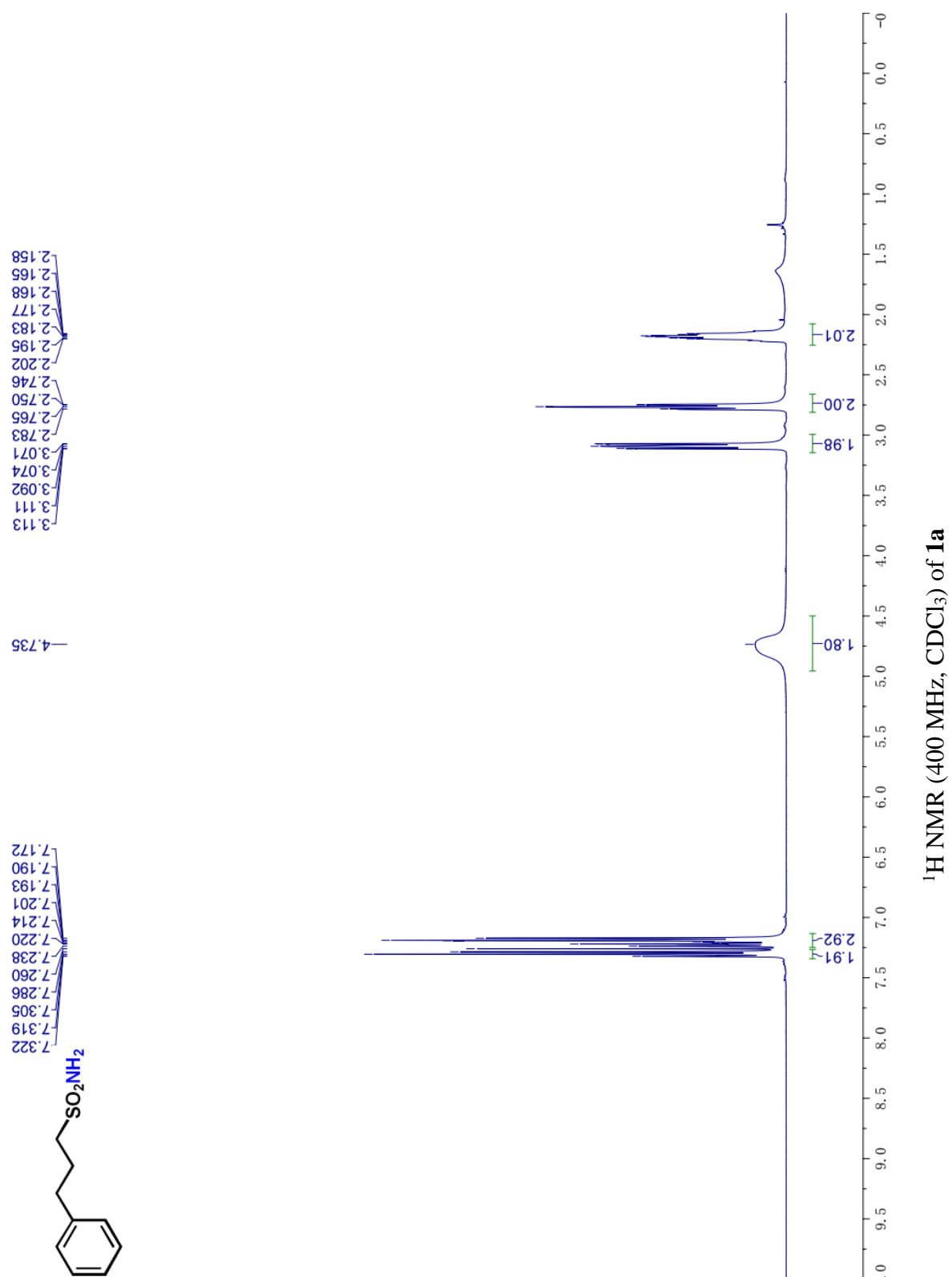


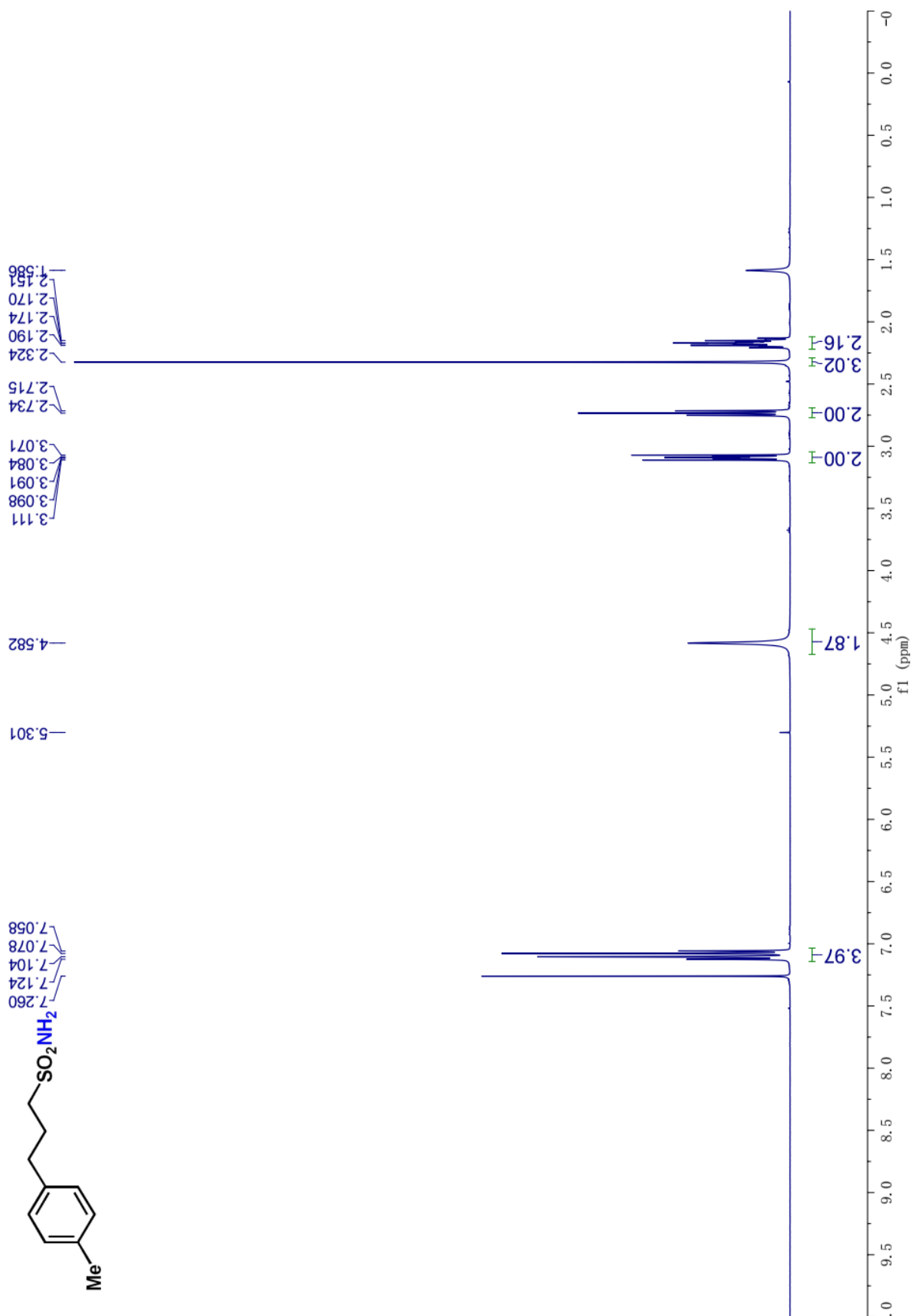
To a solution of **7** (10.1 mg, 0.037 mmol) in CH_2Cl_2 (10.0 mL) was added Hoveyda-Grubbs second generation catalyst (2.1 mg, 5 mol %). After stirring at room temp. for 1 h, the mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (PE / EA = 5 / 1) to give **3a-phenyl-3,3a,4,7-tetrahydro-2H-isothiazolo[2,3-a] pyridine 1,1-dioxide 5** (8.2 mg, 90% yield) as a white solid. R_f = 0.3 (PE / EA = 3 / 1, KMnO_4).

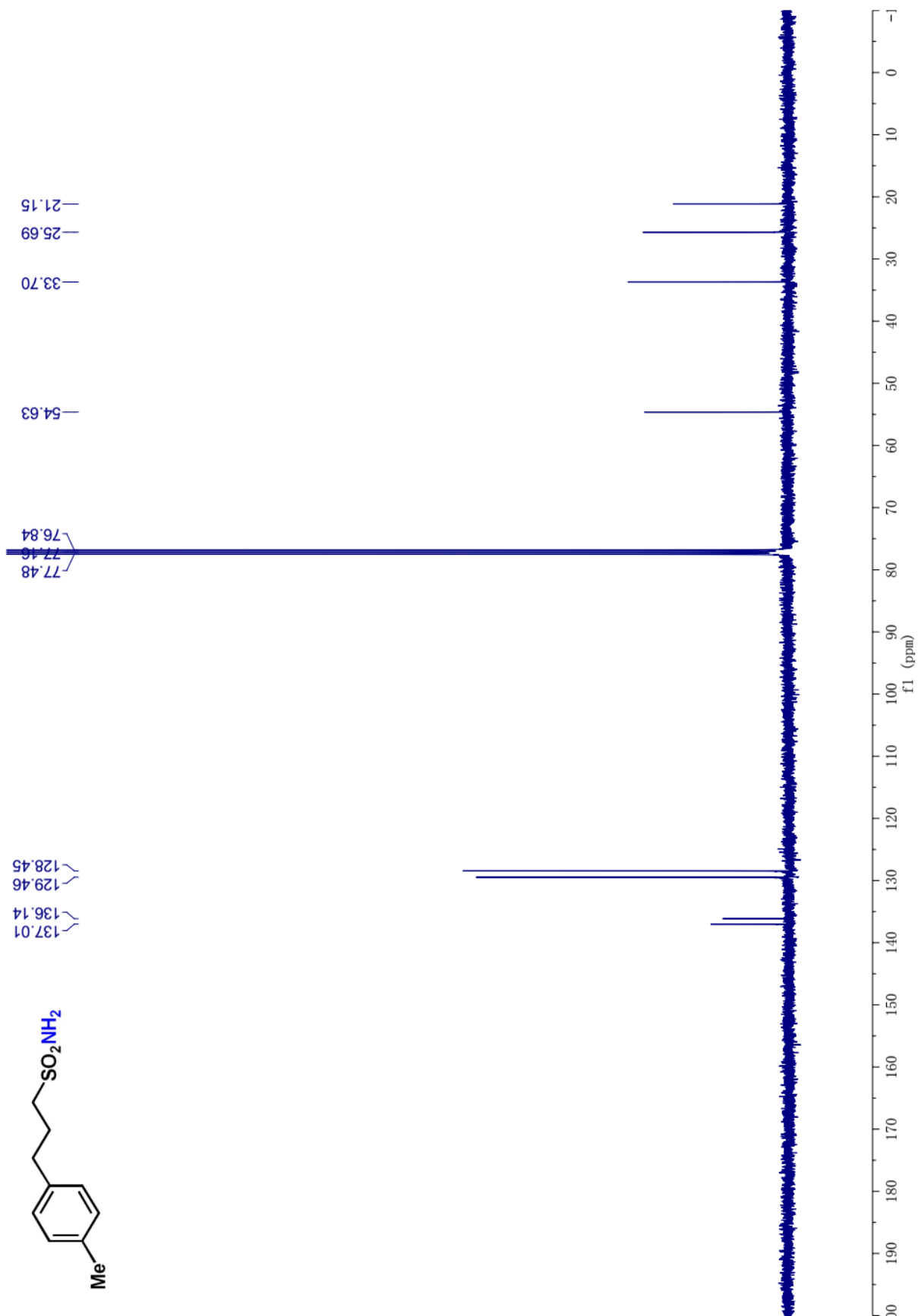
^1H NMR (400 MHz, CDCl_3) δ 7.47 – 7.42 (m, 2H), 7.40 – 7.36 (m, 2H), 7.31 – 7.28 (m, 1H), 5.91 – 5.73 (m, 1H), 5.64 – 5.50 (m, 1H), 4.11 – 4.04 (m, 1H), 3.49 – 3.43 (m, 1H), 3.36 – 3.13 (m, 2H), 2.96 – 2.89 (m, 1H), 2.71 – 2.56 (m, 1H), 2.50 – 2.46 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.5, 129.1, 127.9, 125.7, 123.6, 123.5, 61.7, 45.4, 38.1, 36.4, 32.3; HRMS (ESI+) calc'd for $\text{C}_{13}\text{H}_{15}\text{NNaO}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 272.0716, found 272.0718.

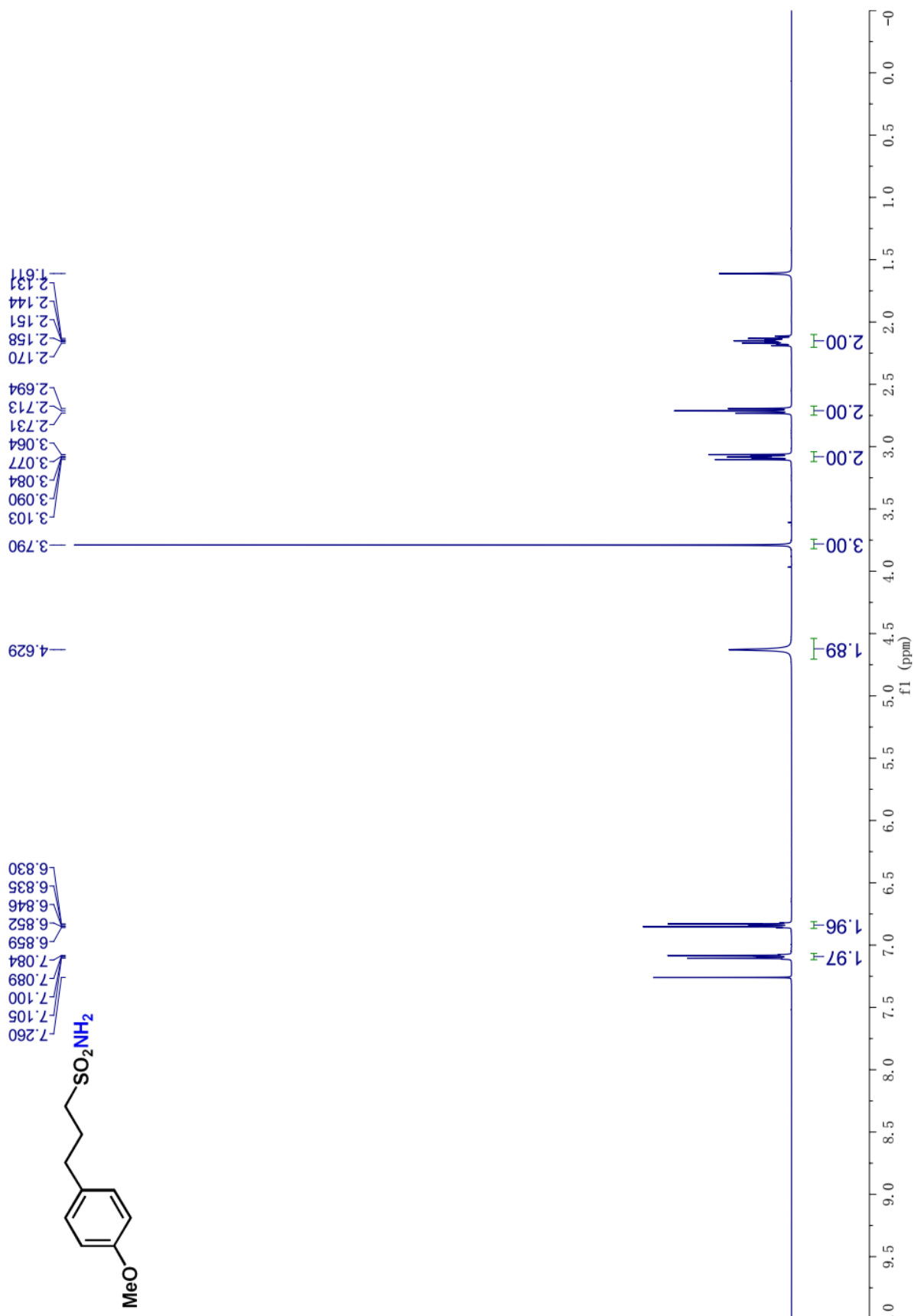
8. References

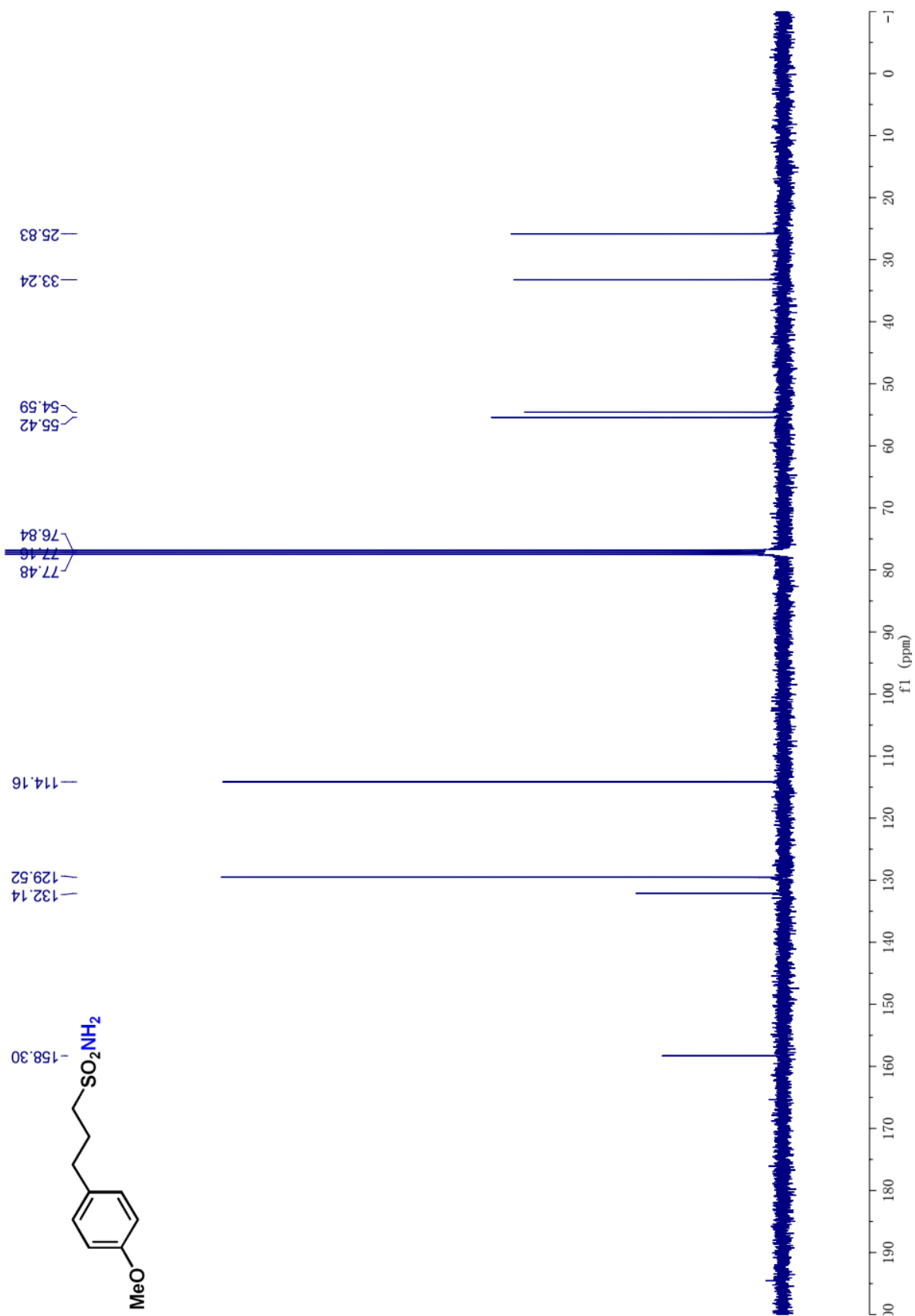
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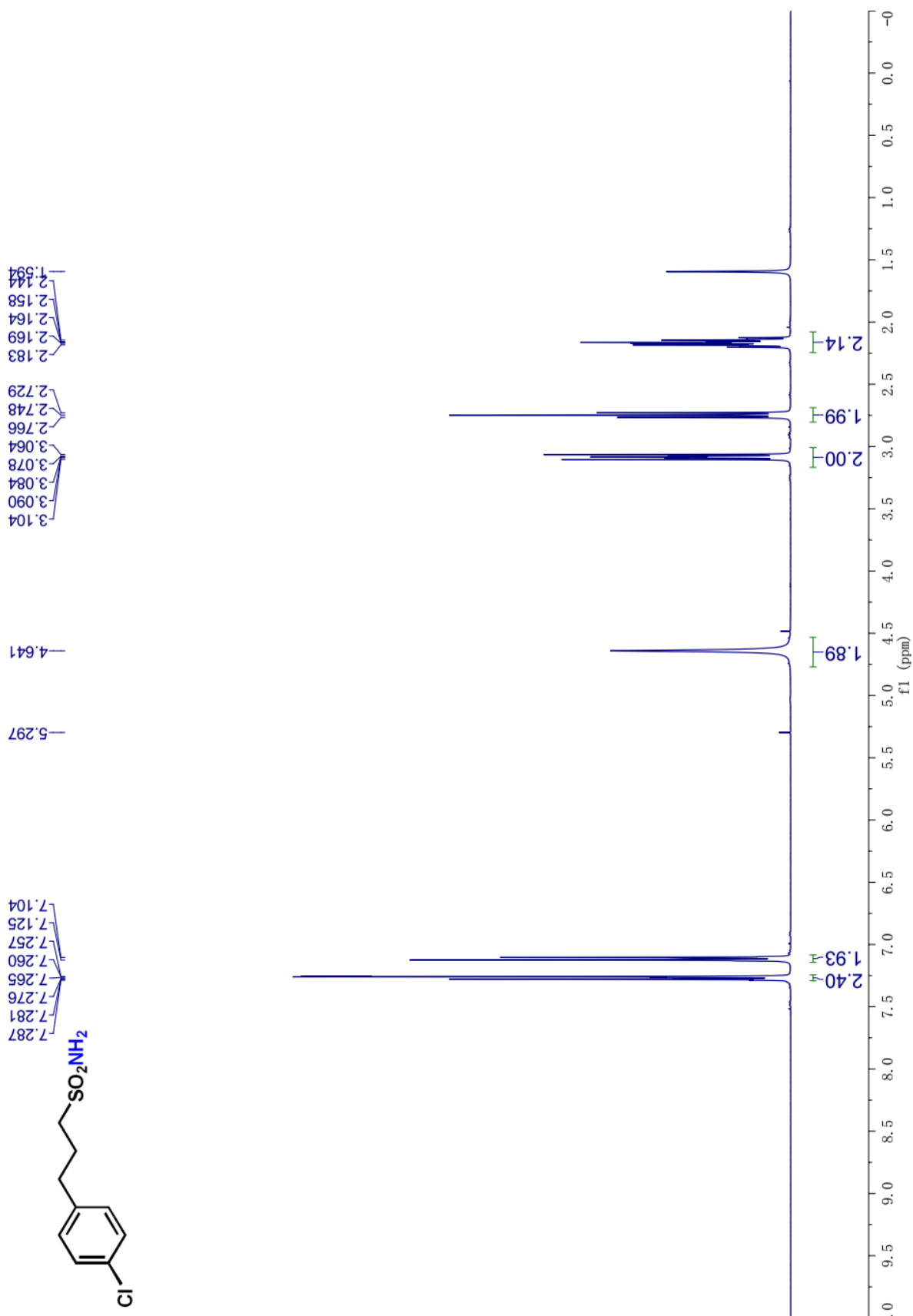
9. ^1H NMR, ^{13}C NMR and ^{19}F NMR Spectra for New Compounds

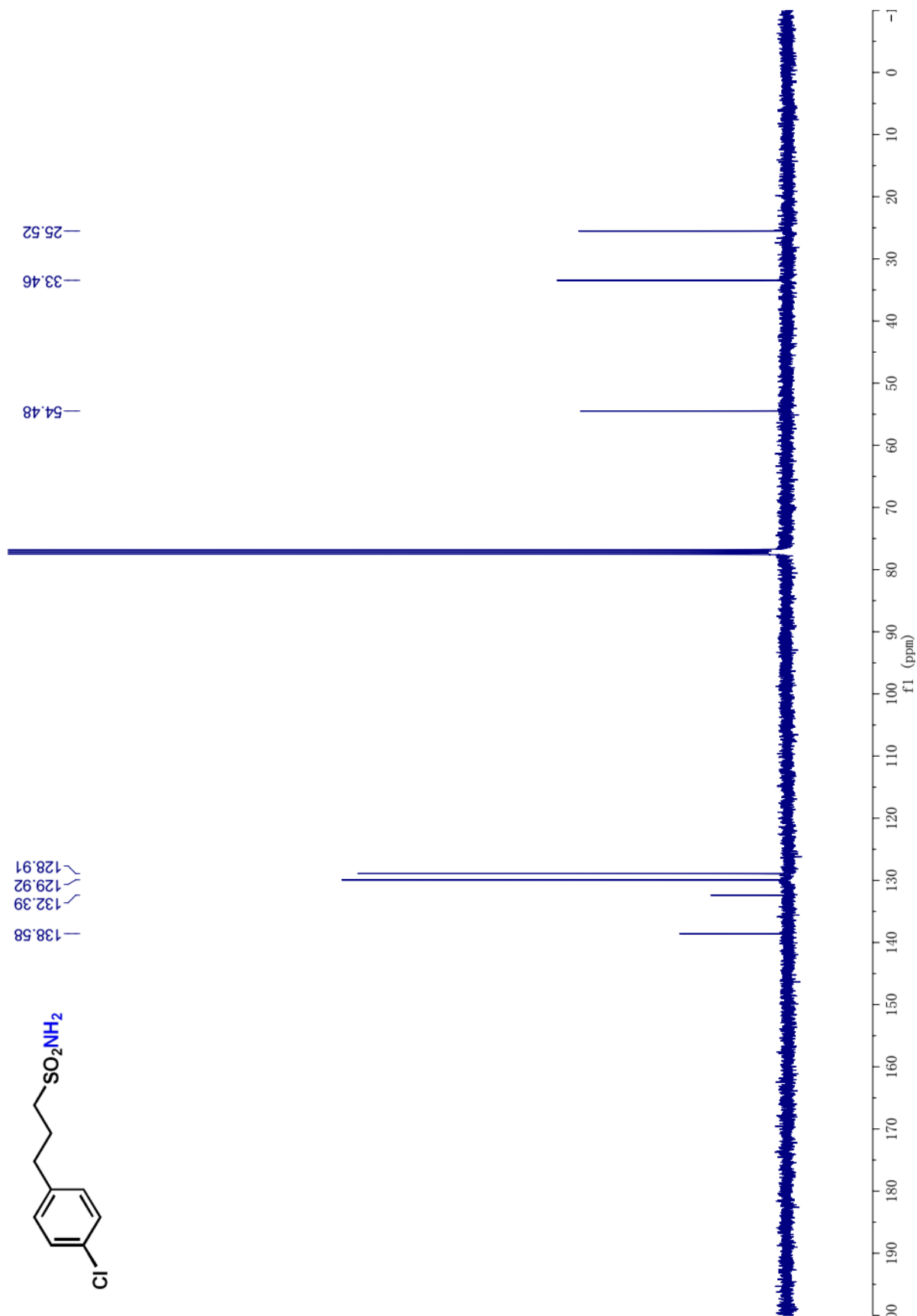
¹H NMR (400 MHz, CDCl₃) of **1b**

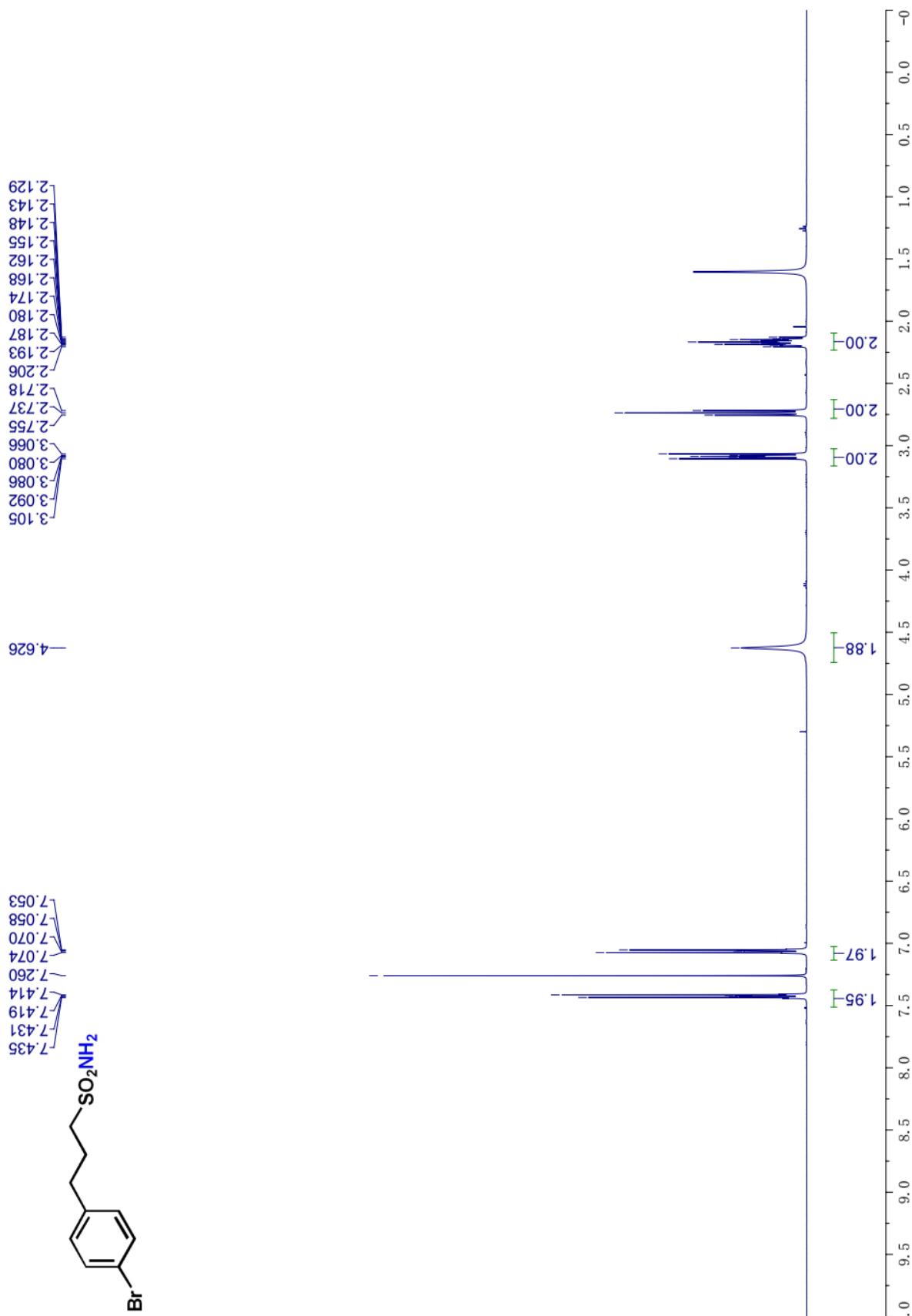
^{13}C NMR (100 MHz, CDCl_3) of **1b**

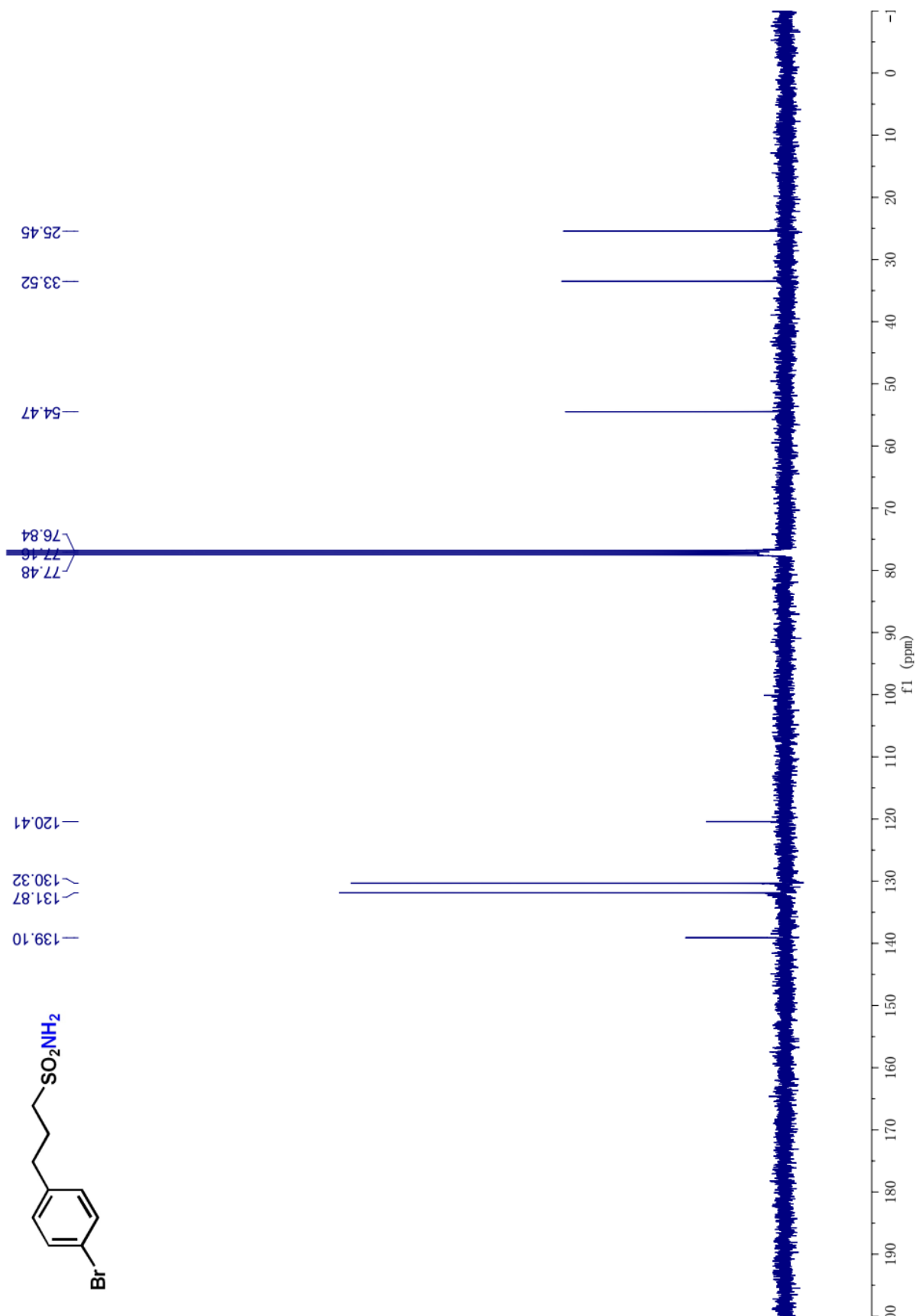
¹H NMR (400 MHz, CDCl₃) of **1c**

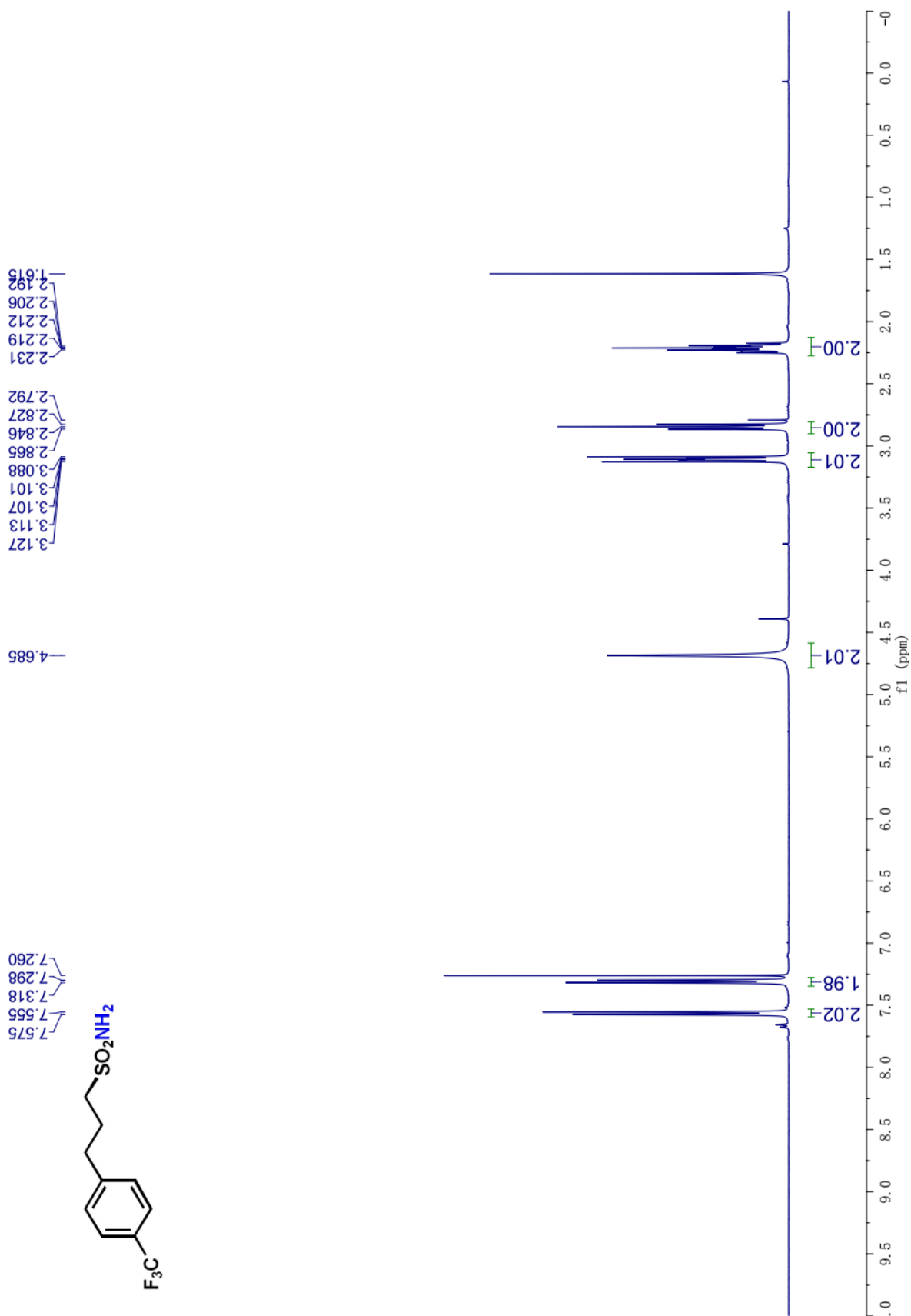
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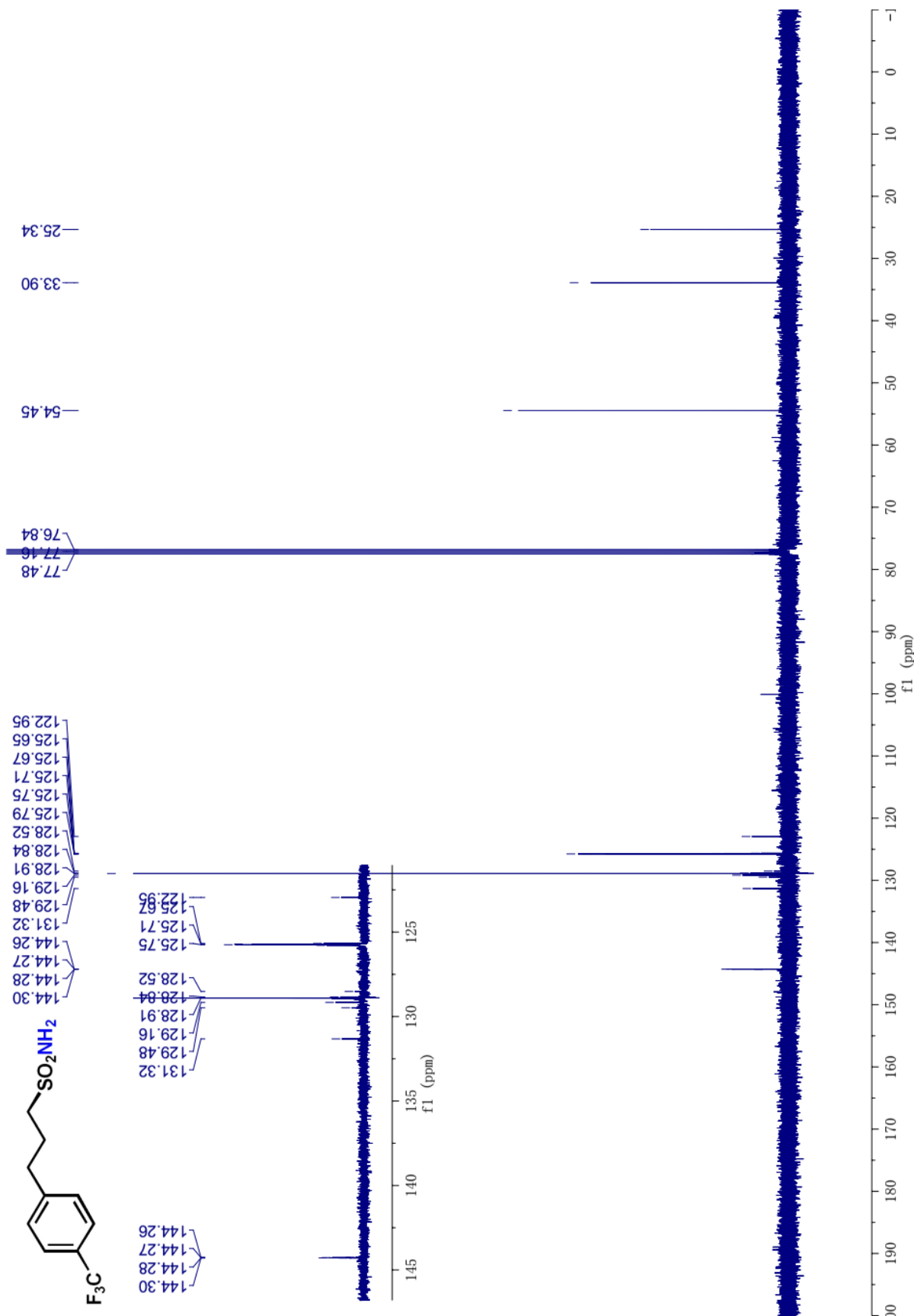
¹H NMR (400 MHz, CDCl₃) of **1d**

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

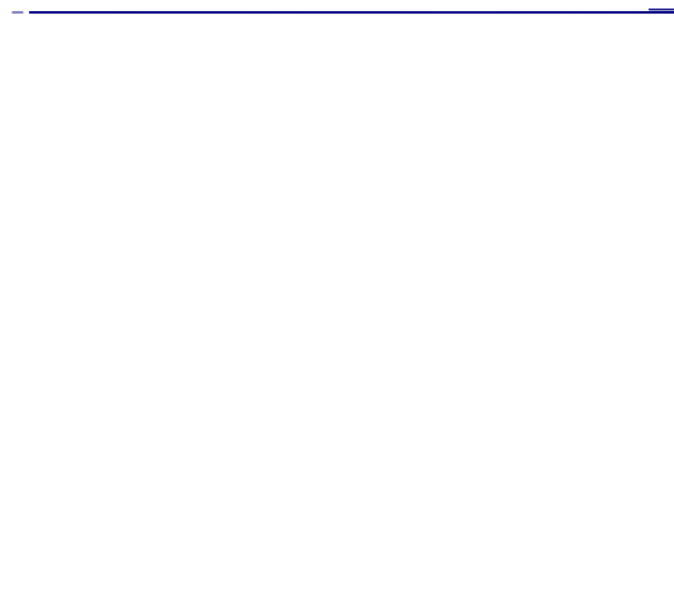
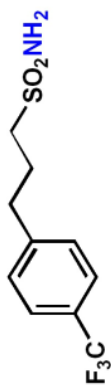
¹H NMR (400 MHz, CDCl₃) of **1e**

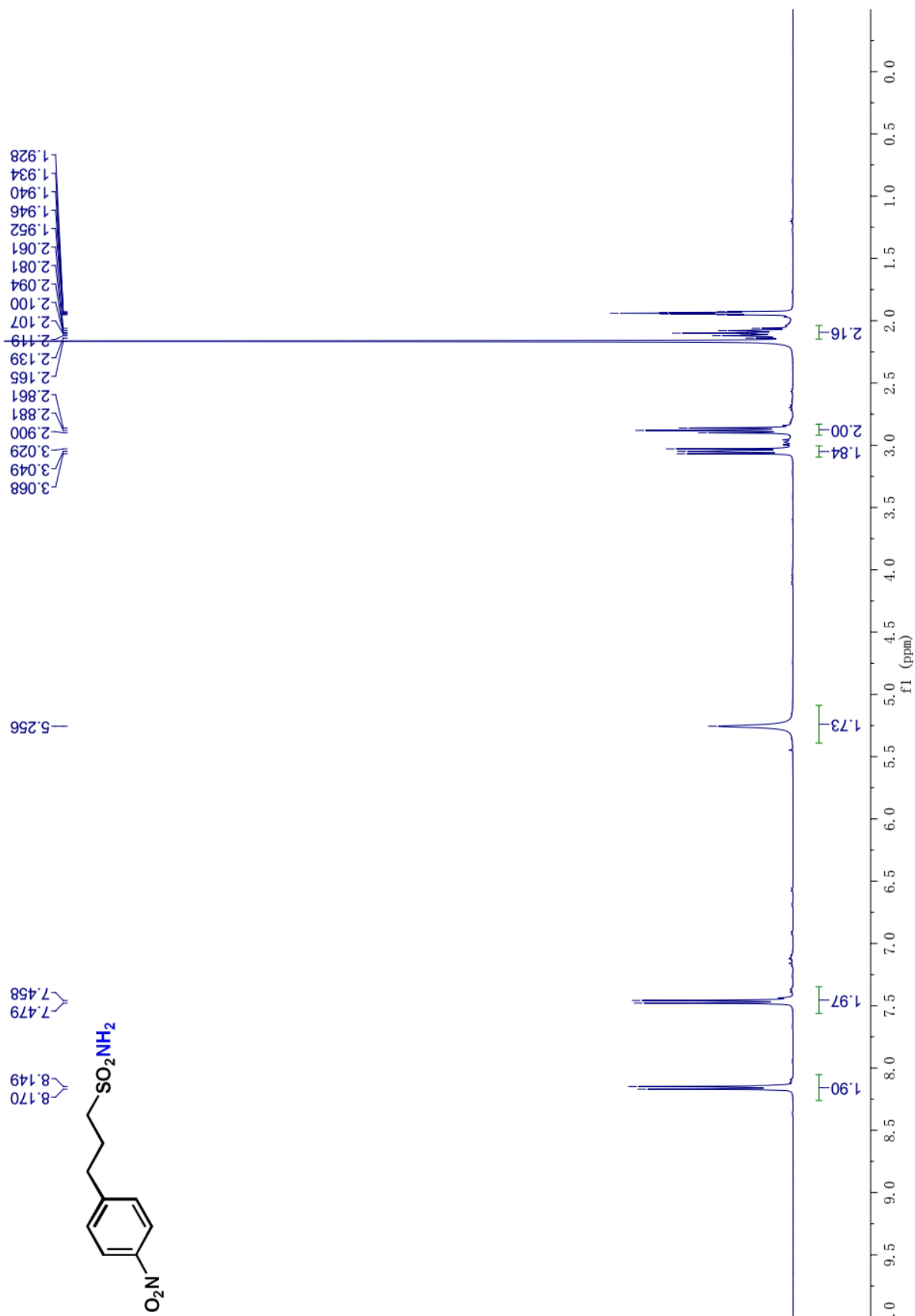
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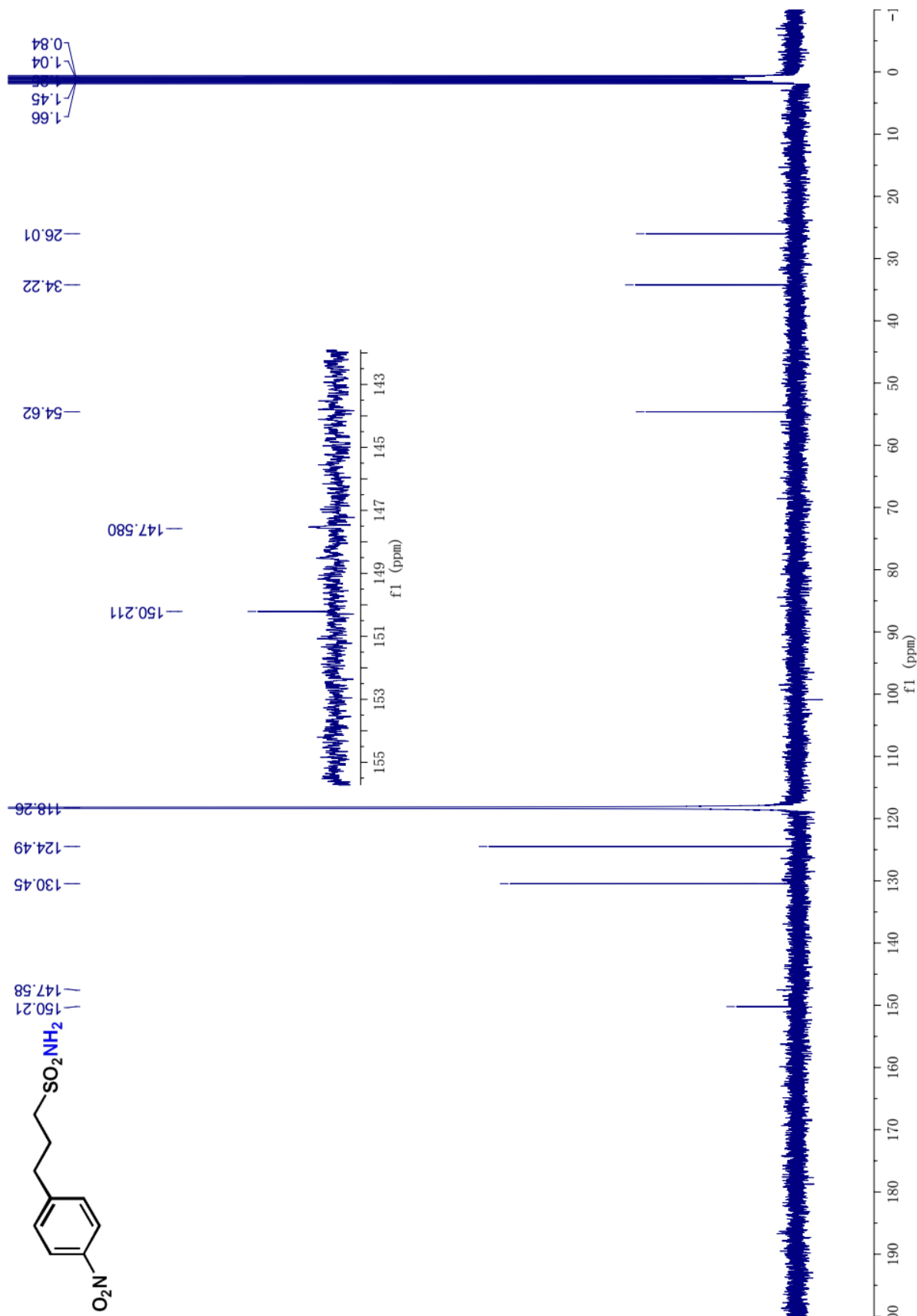
¹H NMR (400 MHz, CDCl₃) of **1f**

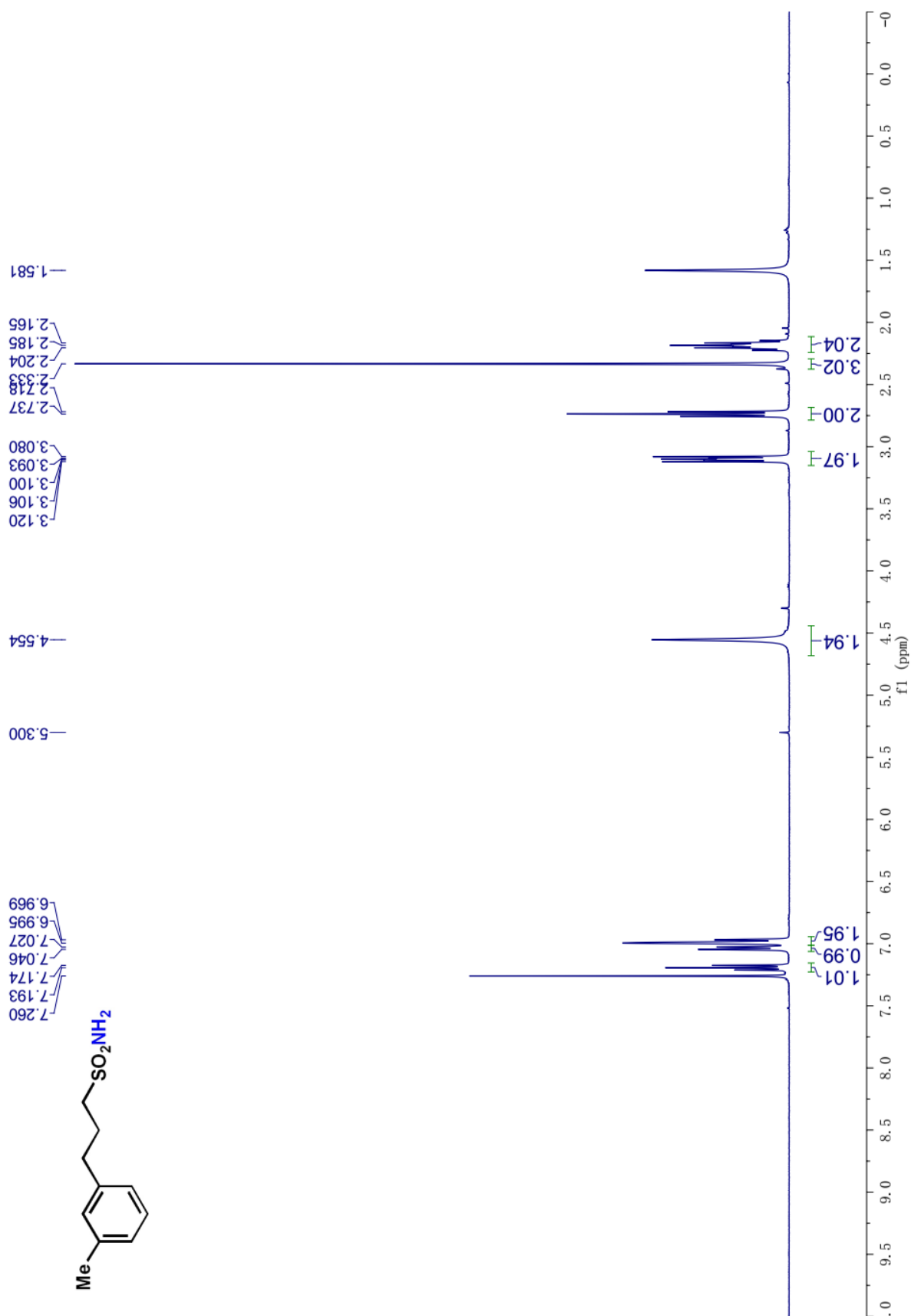
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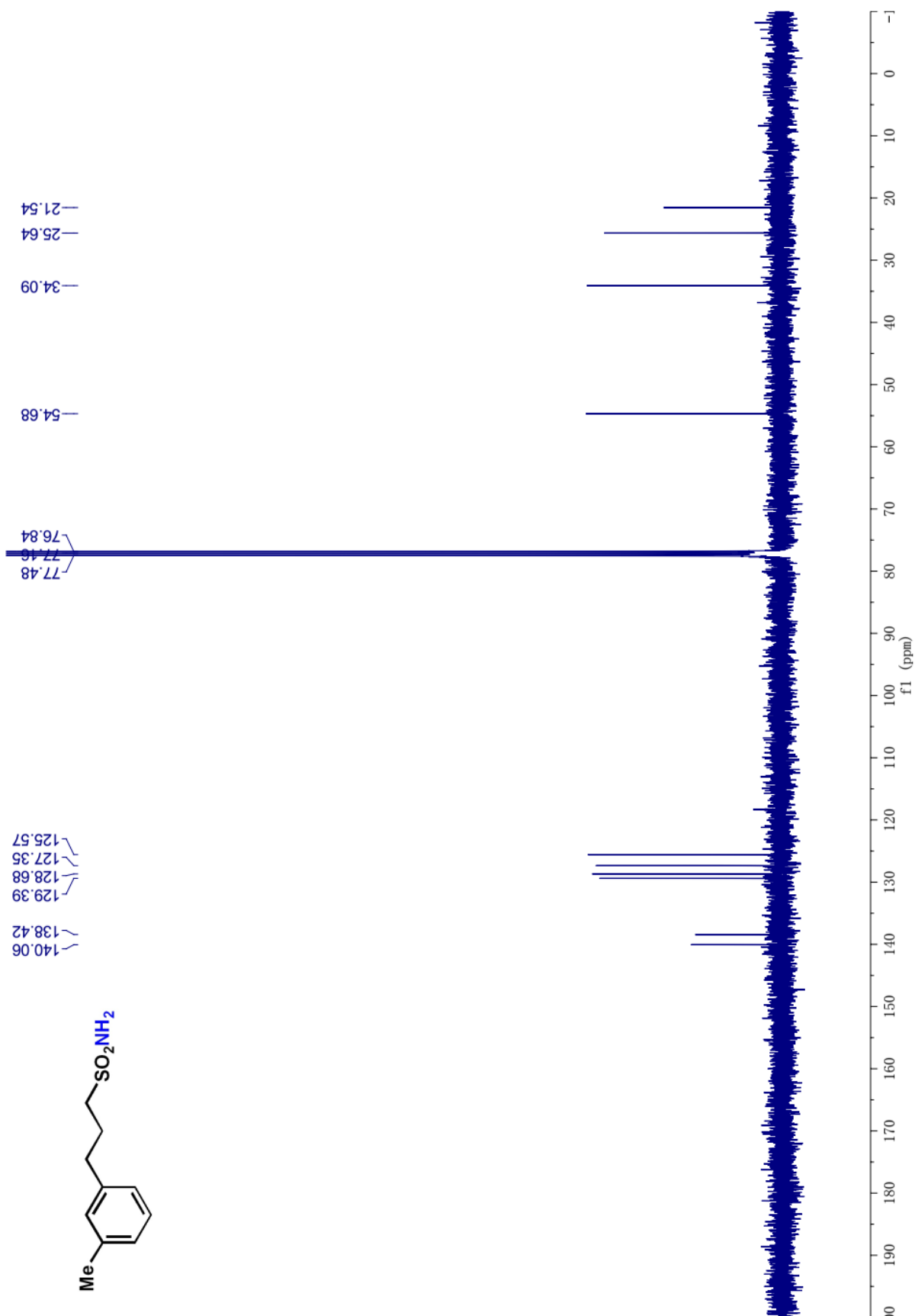
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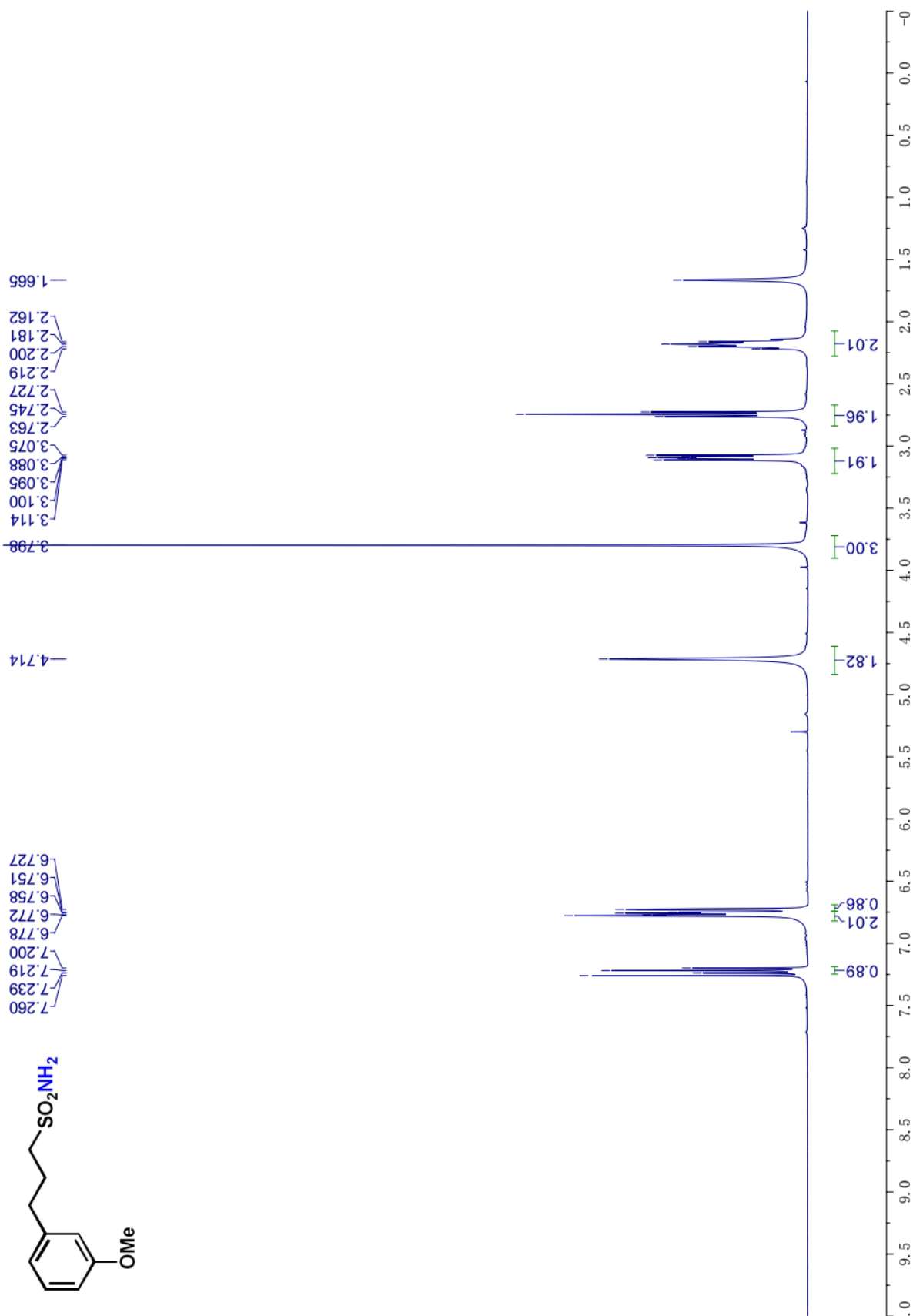
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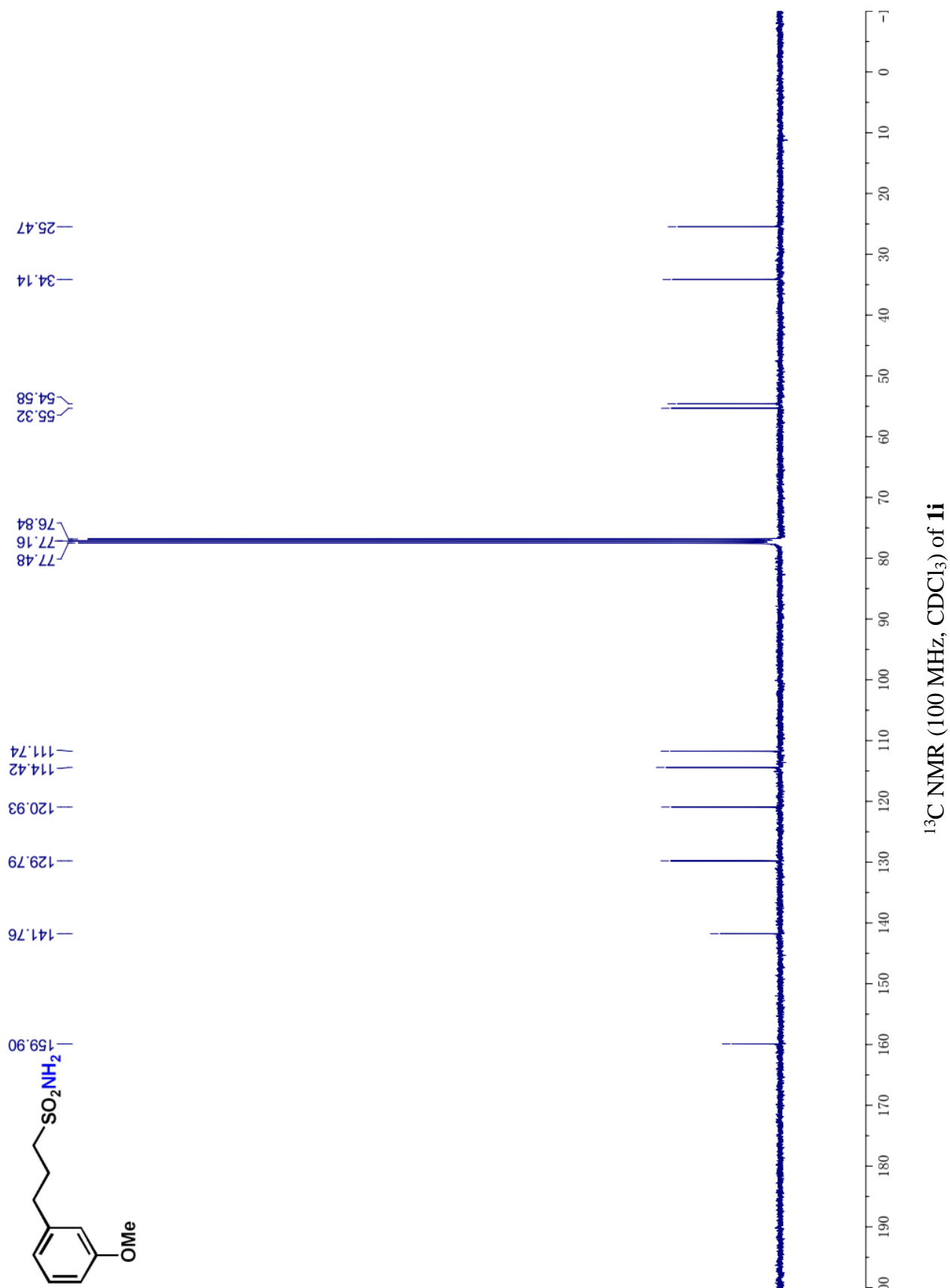
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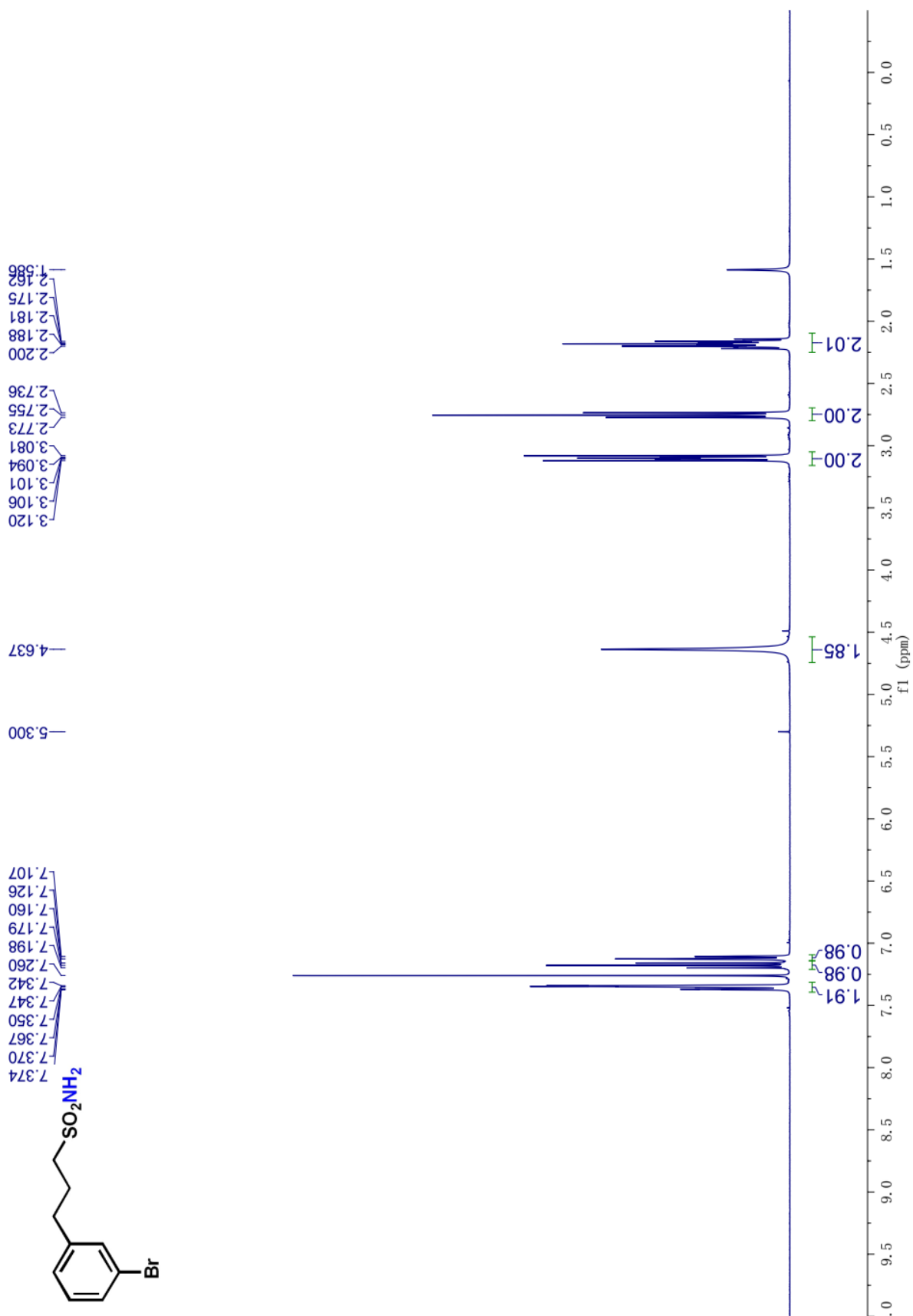
^{13}C NMR (100 MHz, CD_3CN) of **1g**

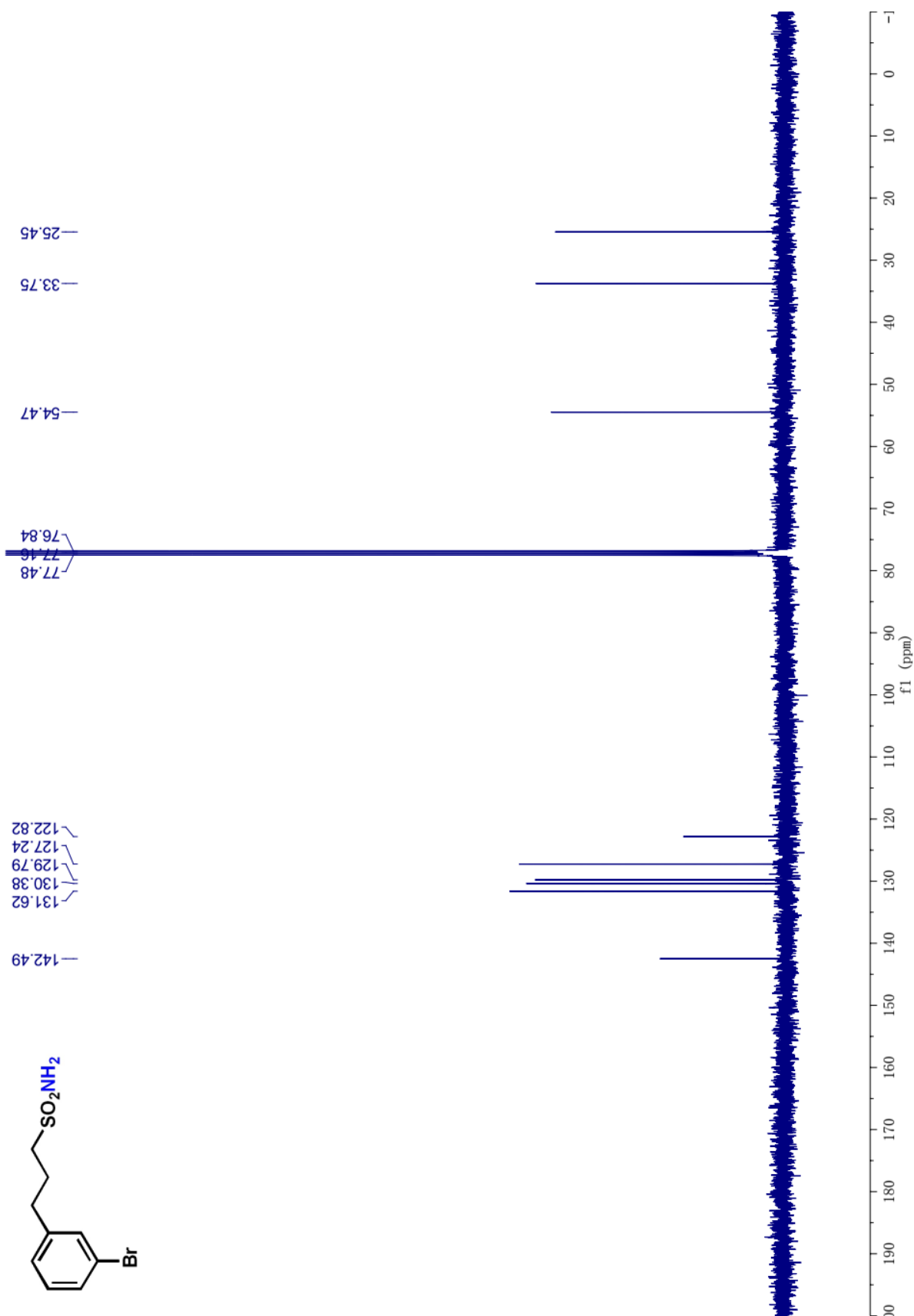
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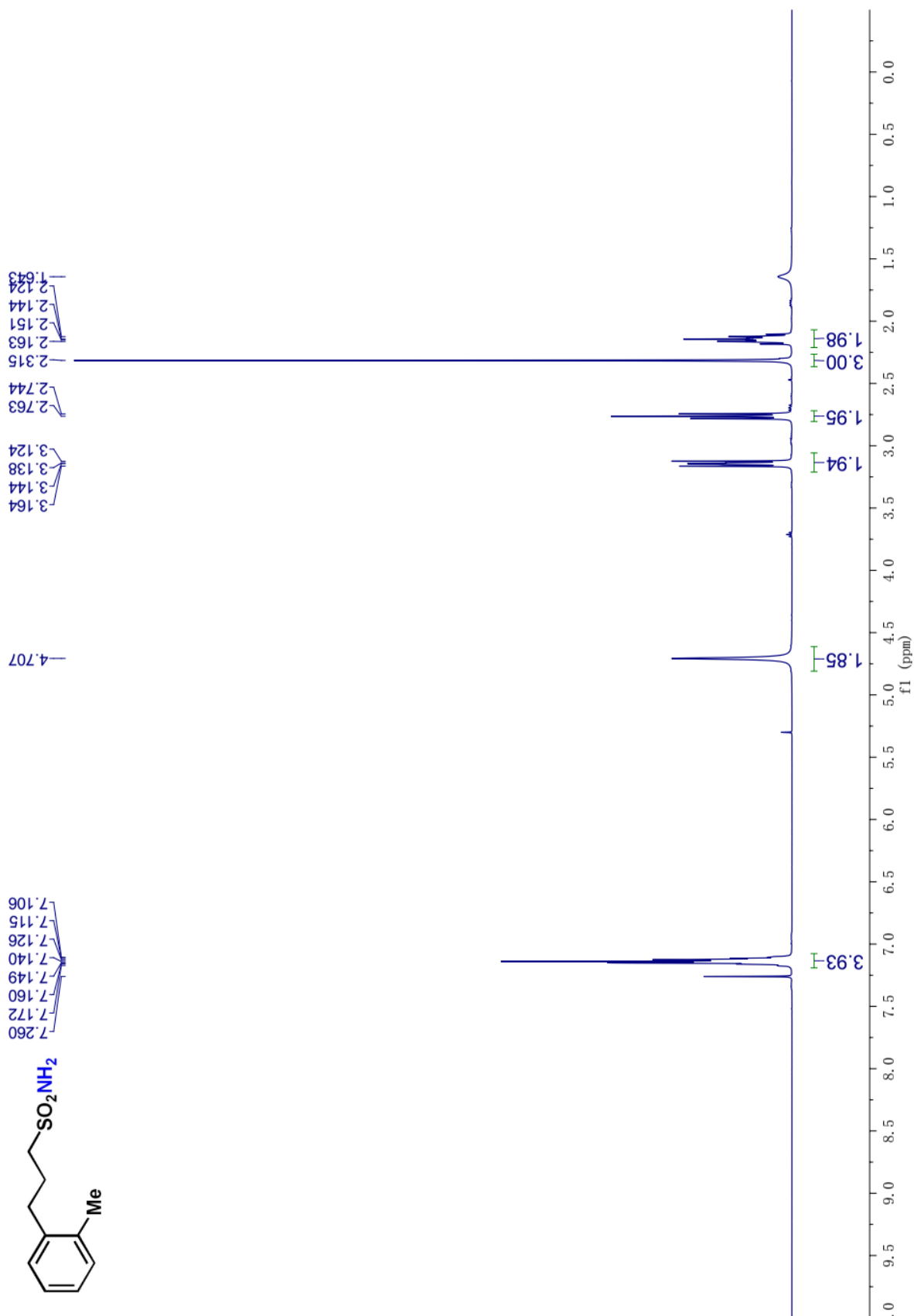
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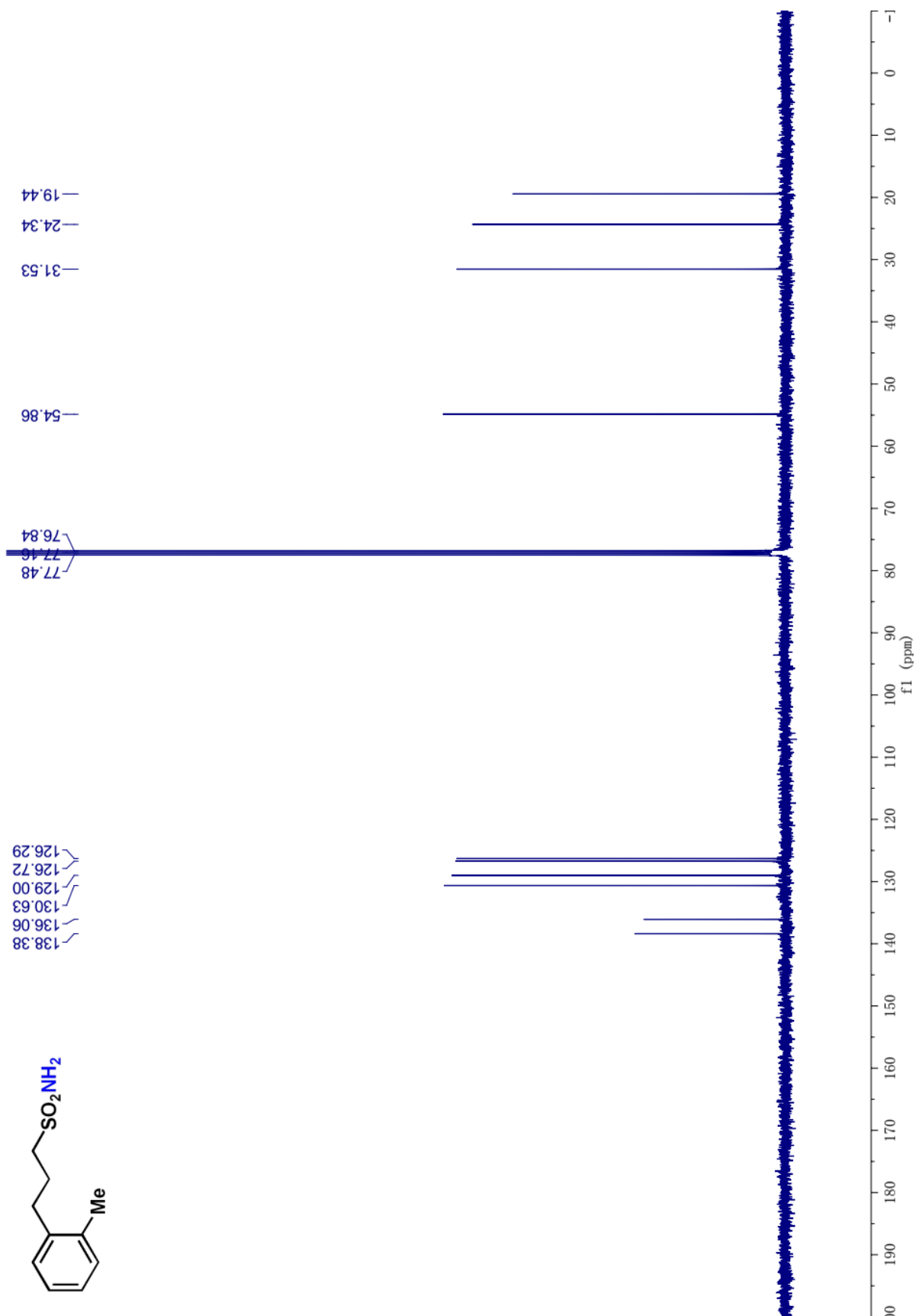
¹H NMR (400 MHz, CDCl₃) of **1i**

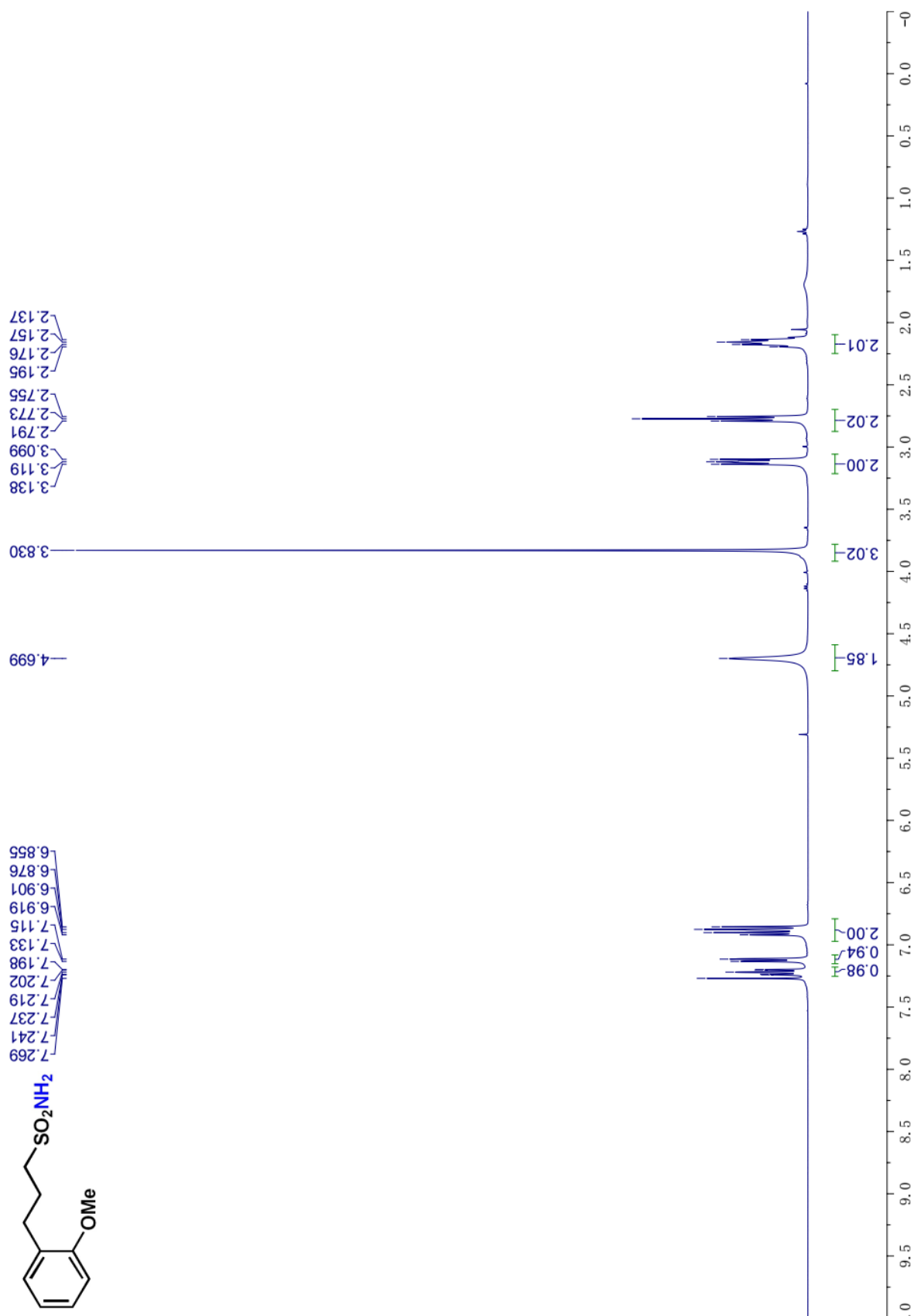


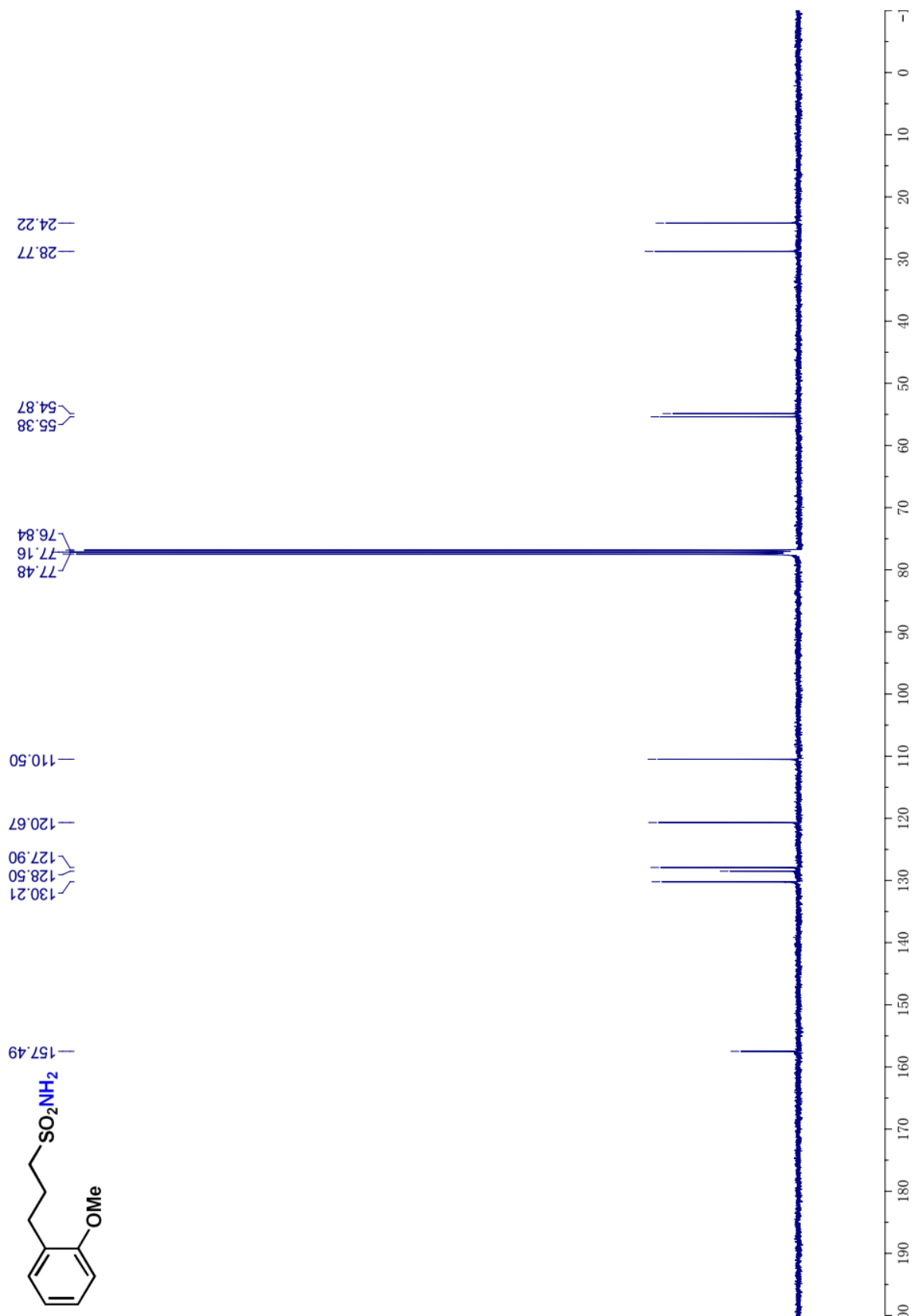
¹H NMR (400 MHz, CDCl₃) of **1j**

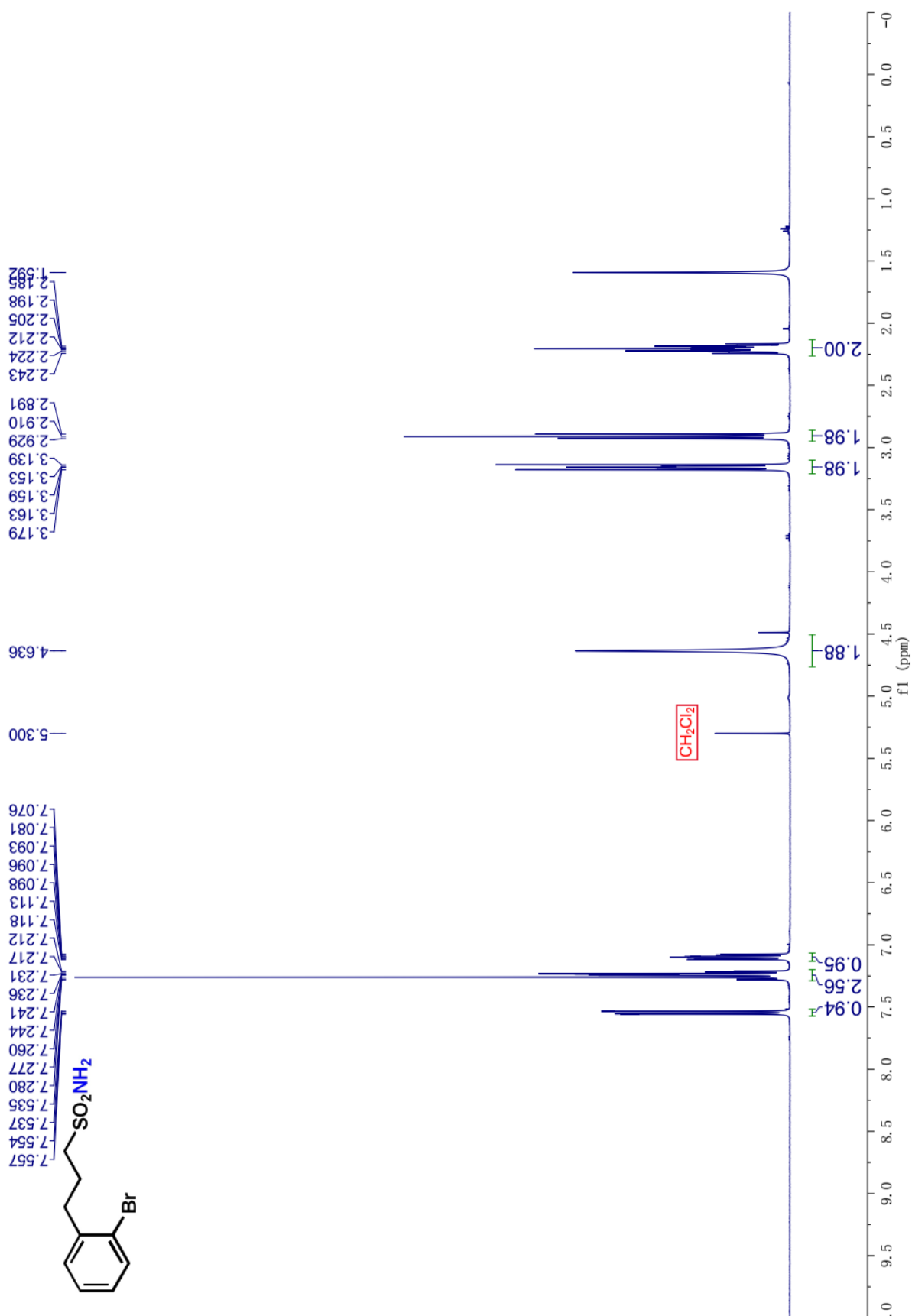
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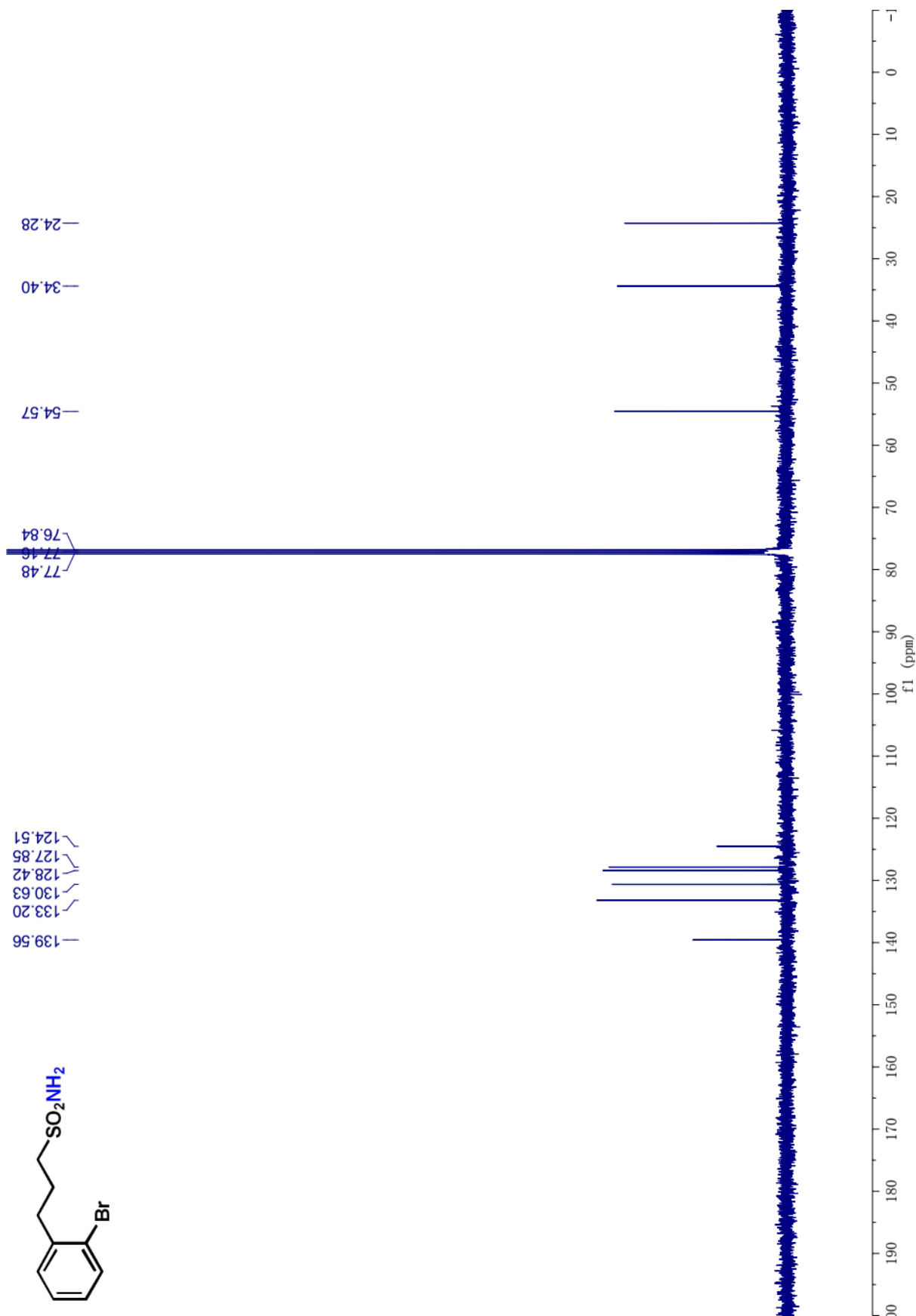
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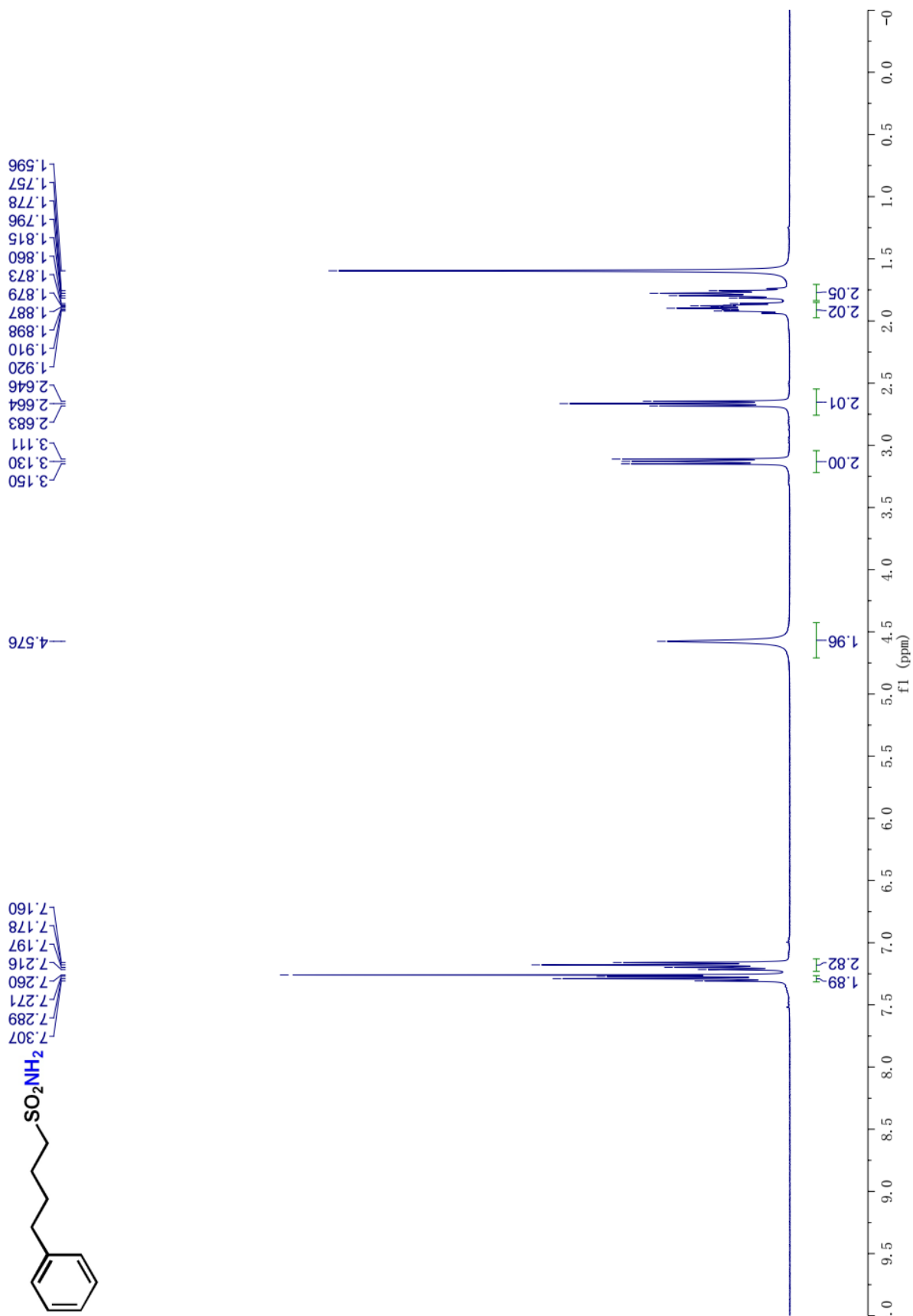


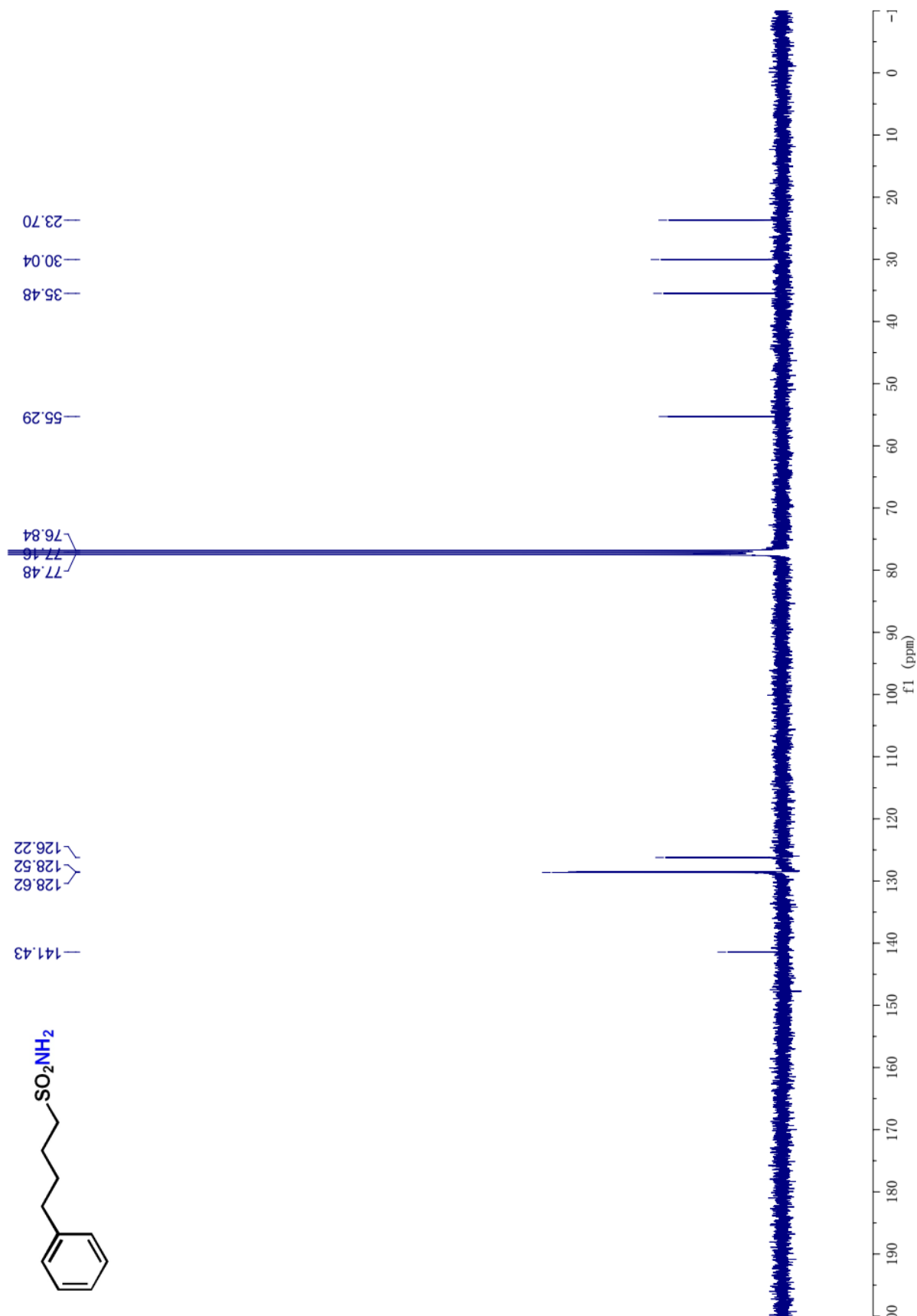
¹H NMR (400 MHz, CDCl₃) of **11**

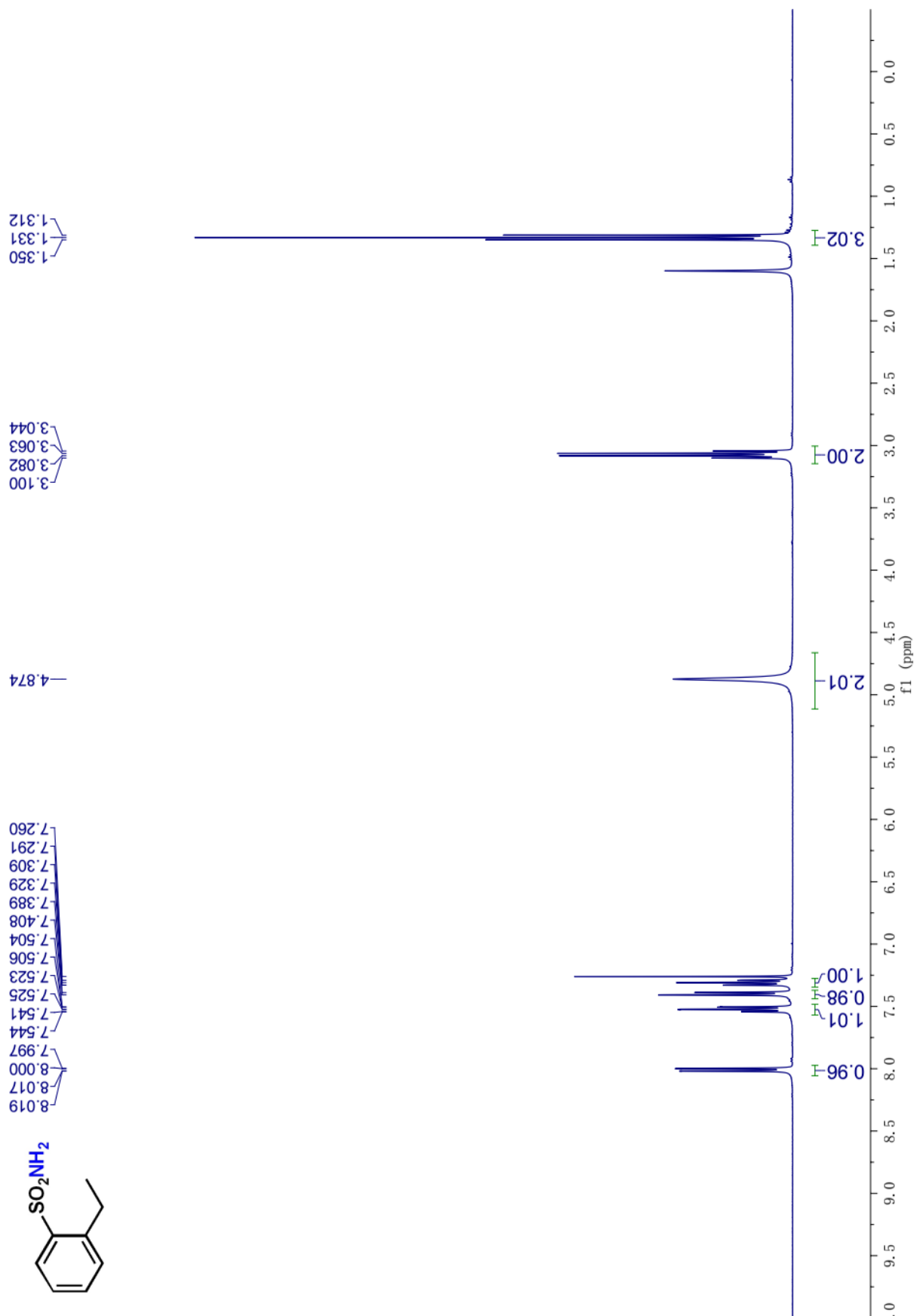
^{13}C NMR (100 MHz, CDCl_3) of **II**

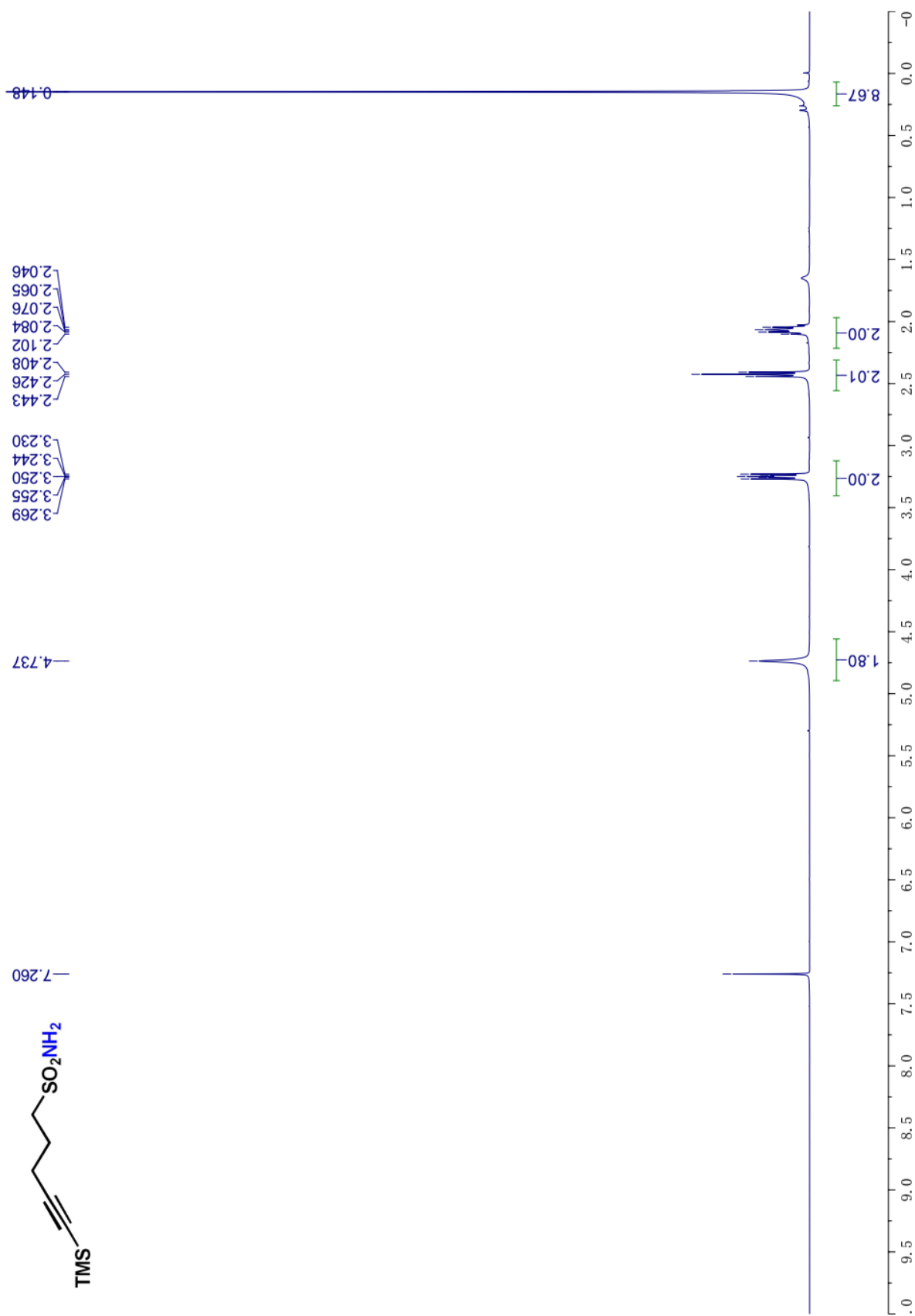
¹H NMR (400 MHz, CDCl₃) of **1m**

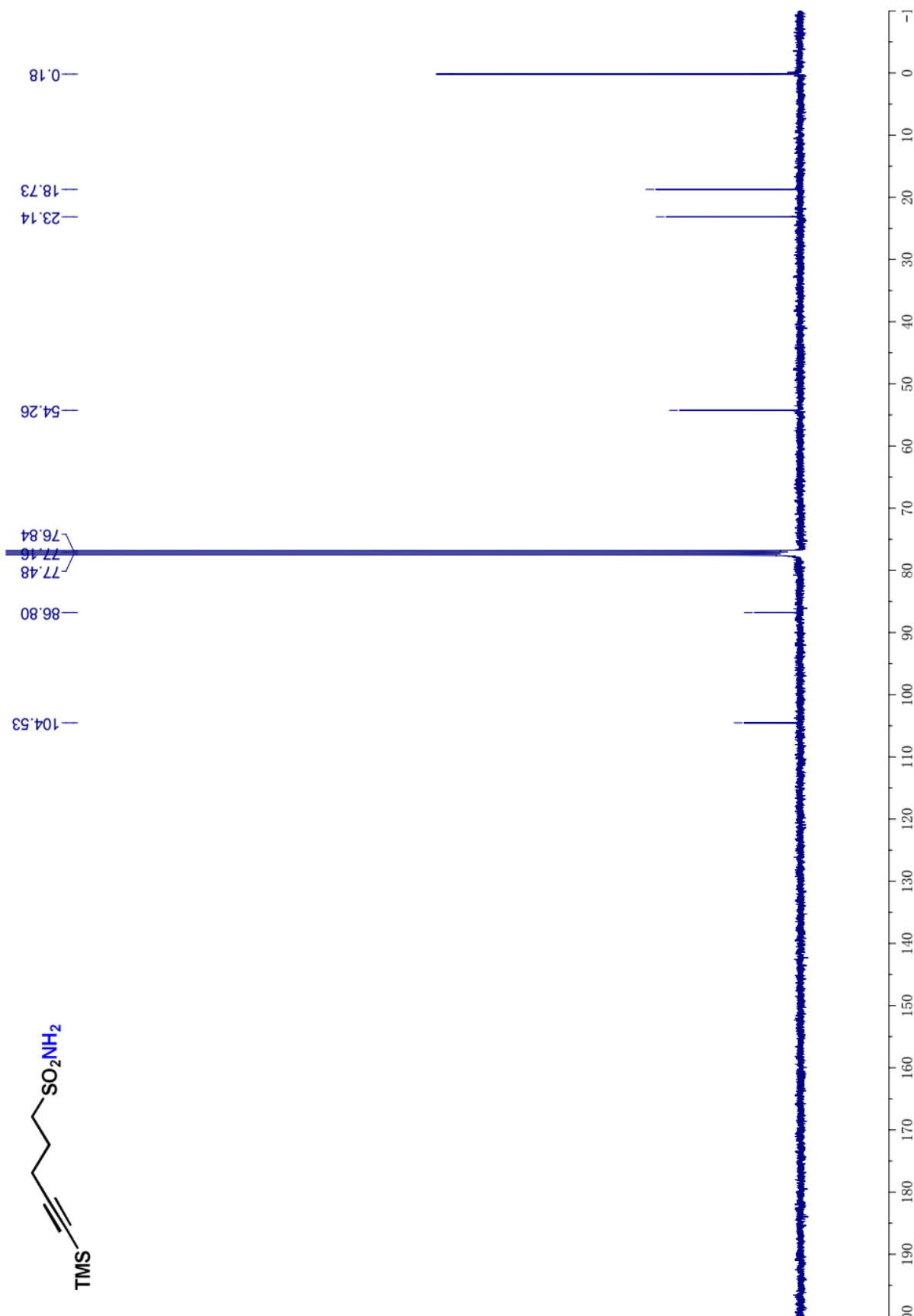
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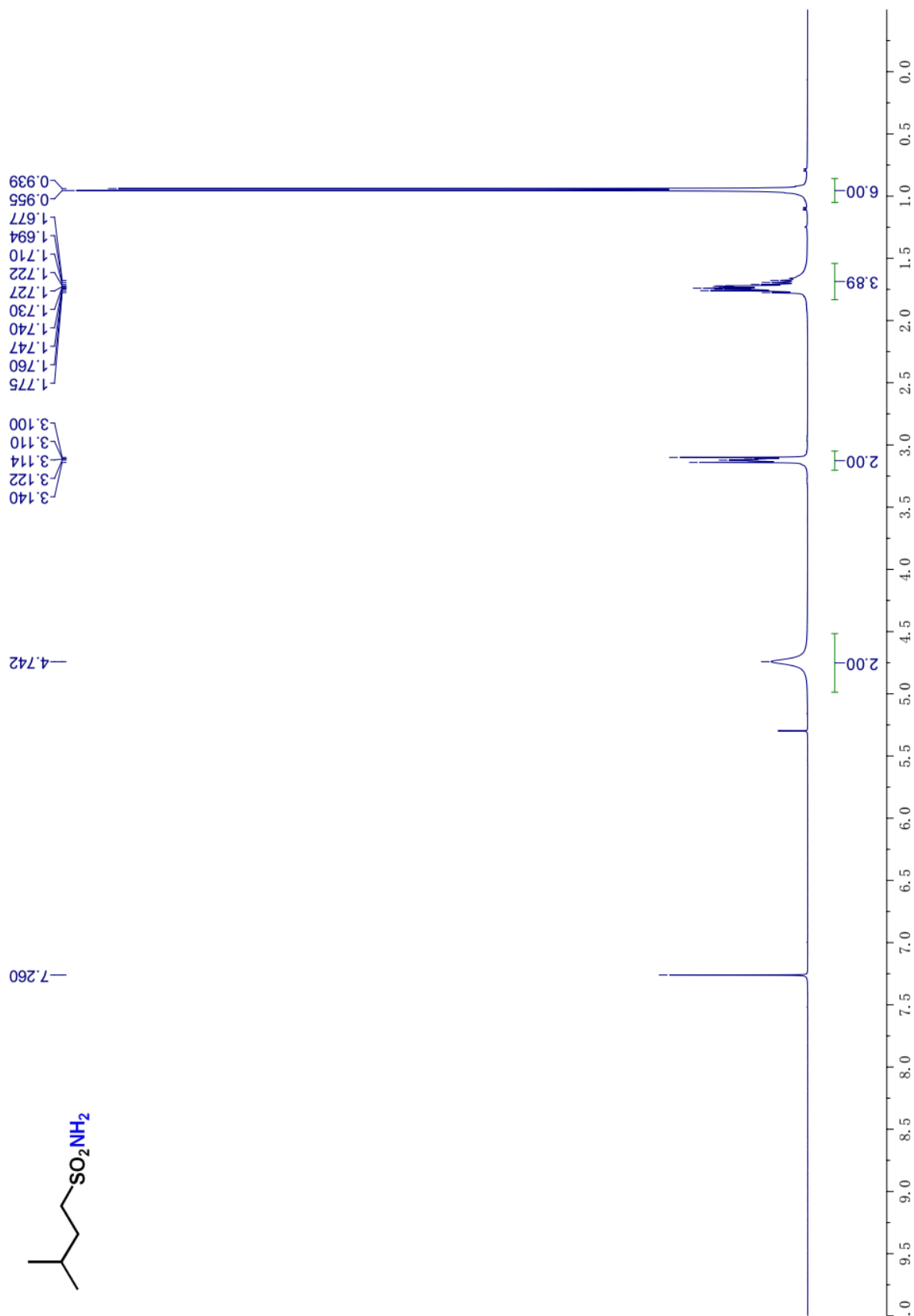
¹H NMR (400 MHz, CDCl₃) of **1n**

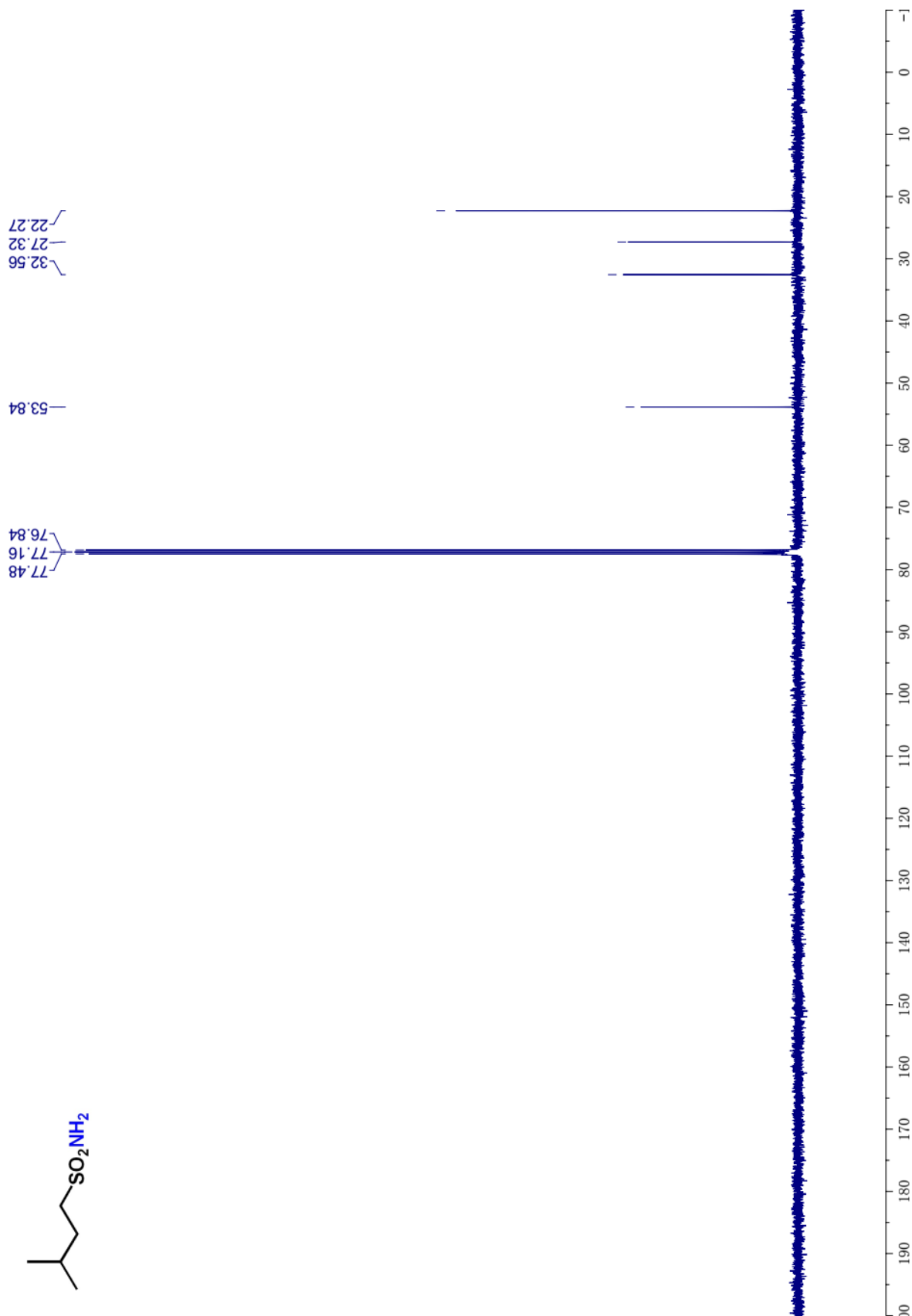
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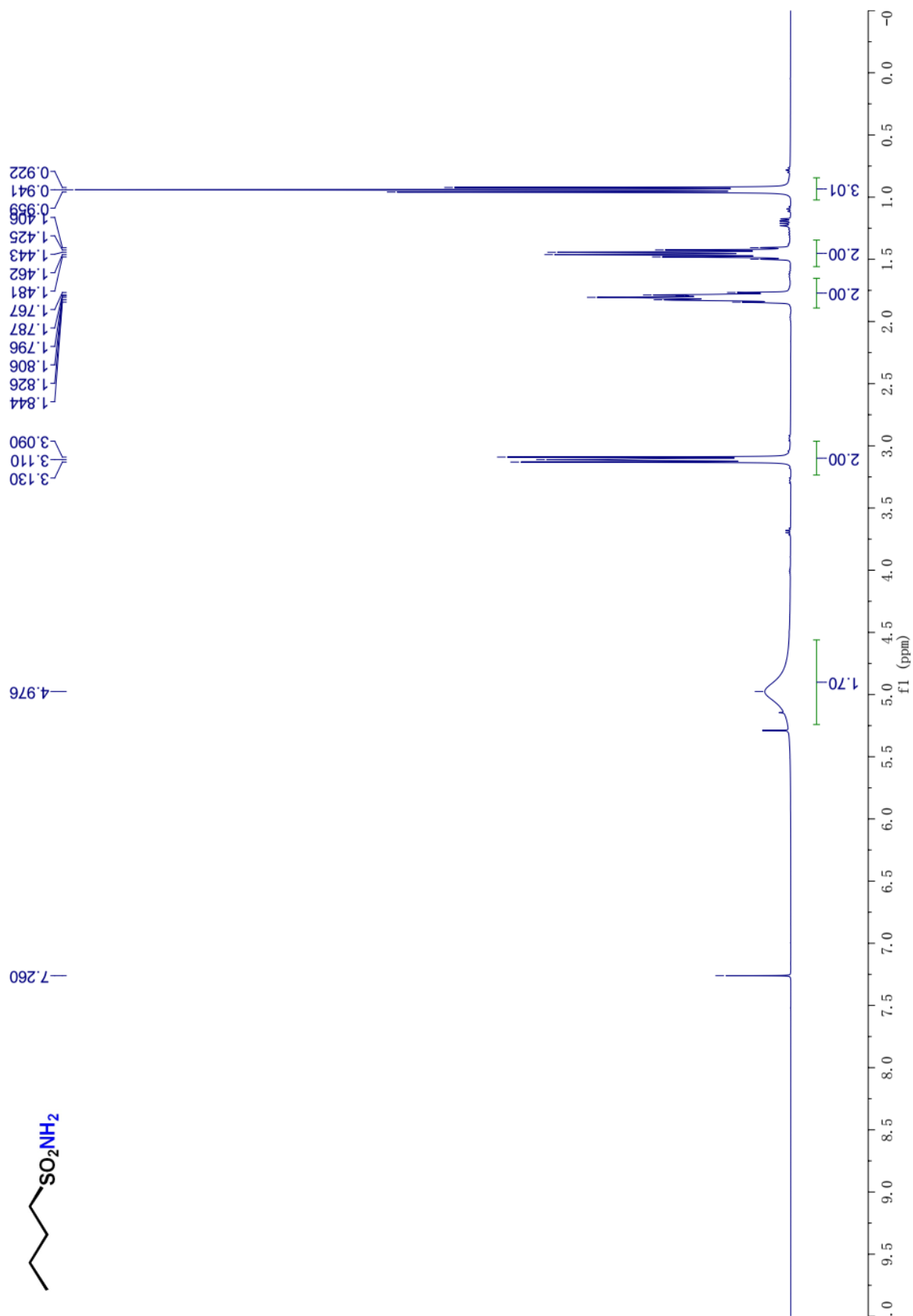
¹H NMR (400 MHz, CDCl₃) of **1p**

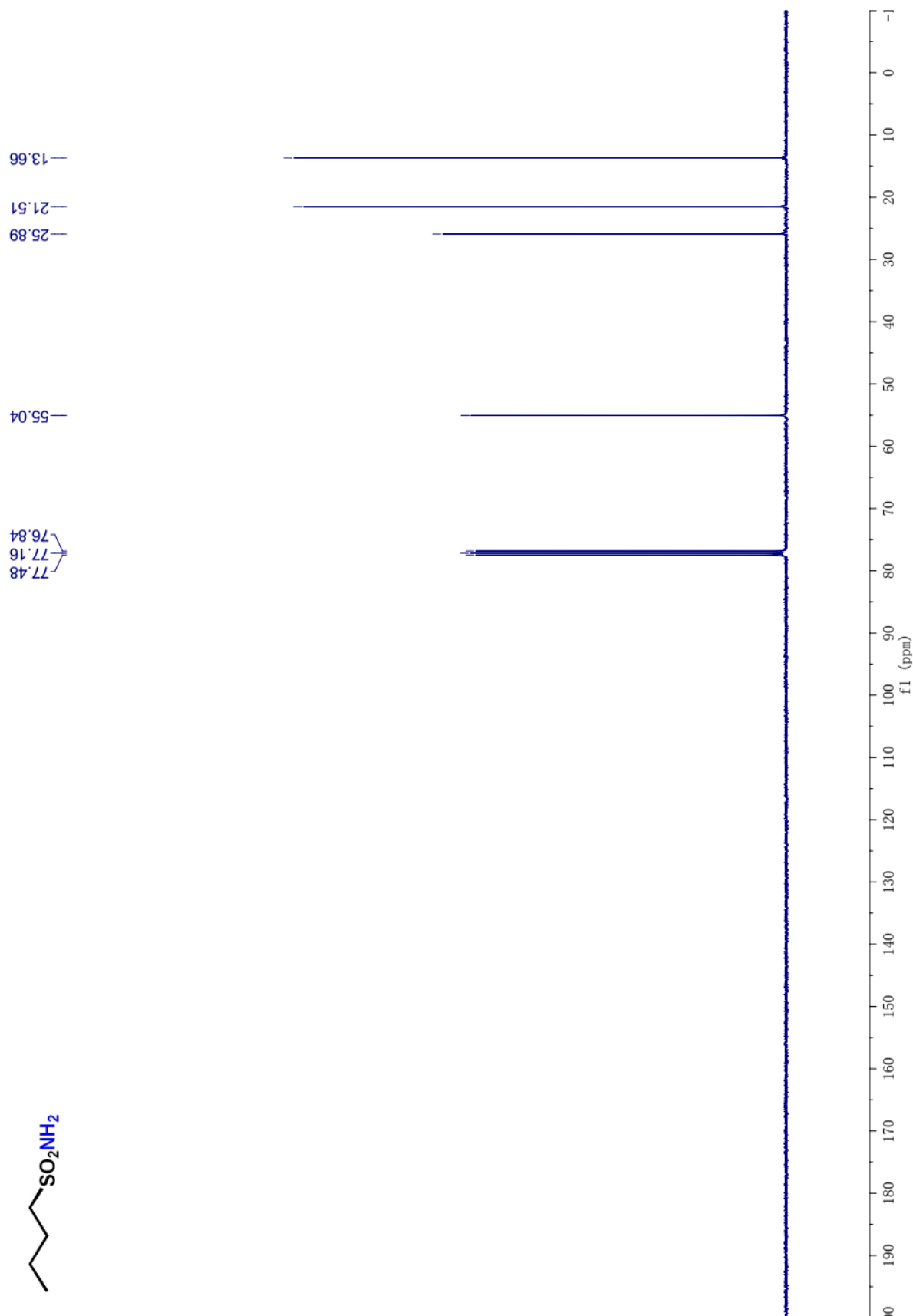
¹H NMR (400 MHz, CDCl₃) of **1q**

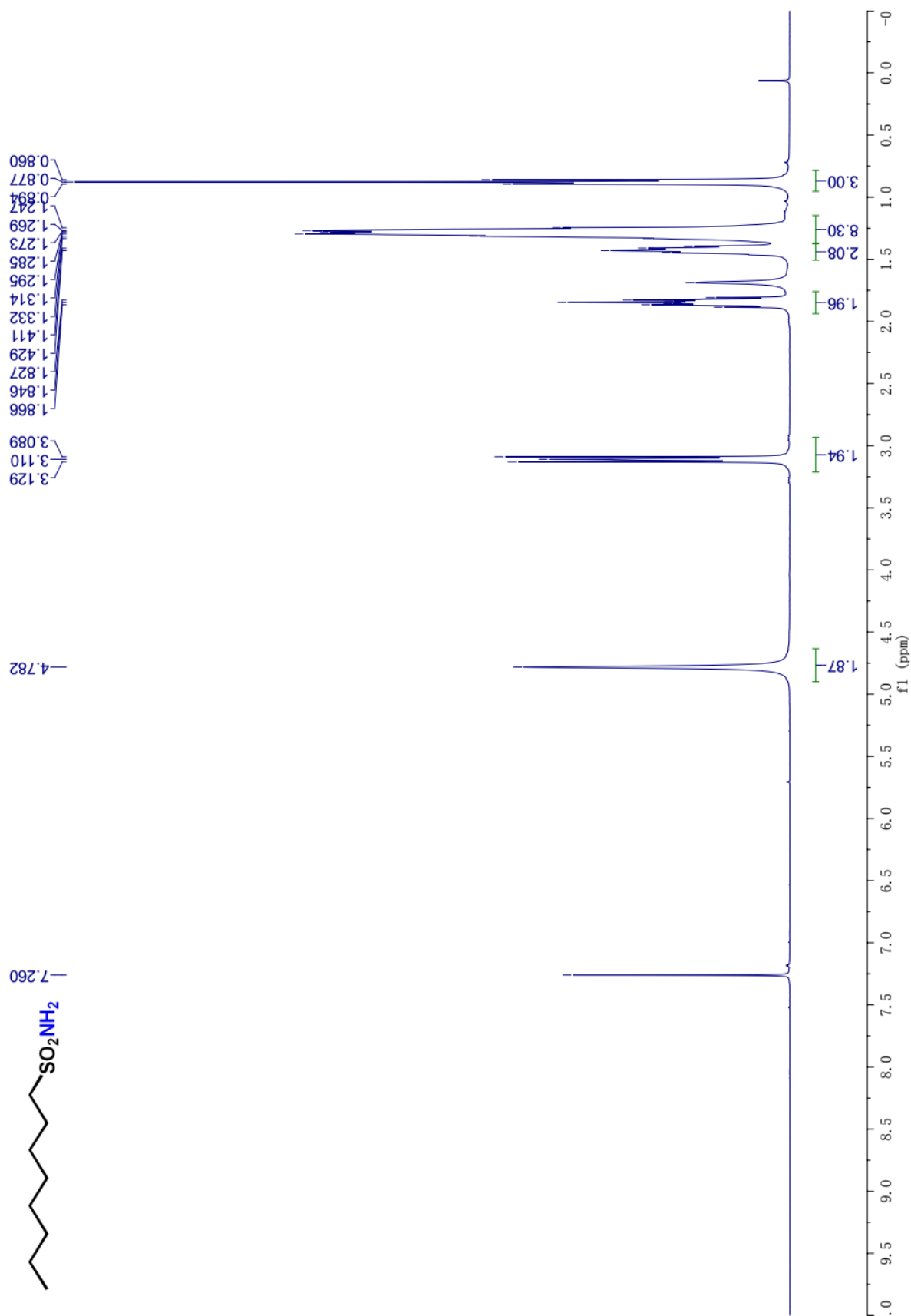
^{13}C NMR (100 MHz, CDCl_3) of **1q**

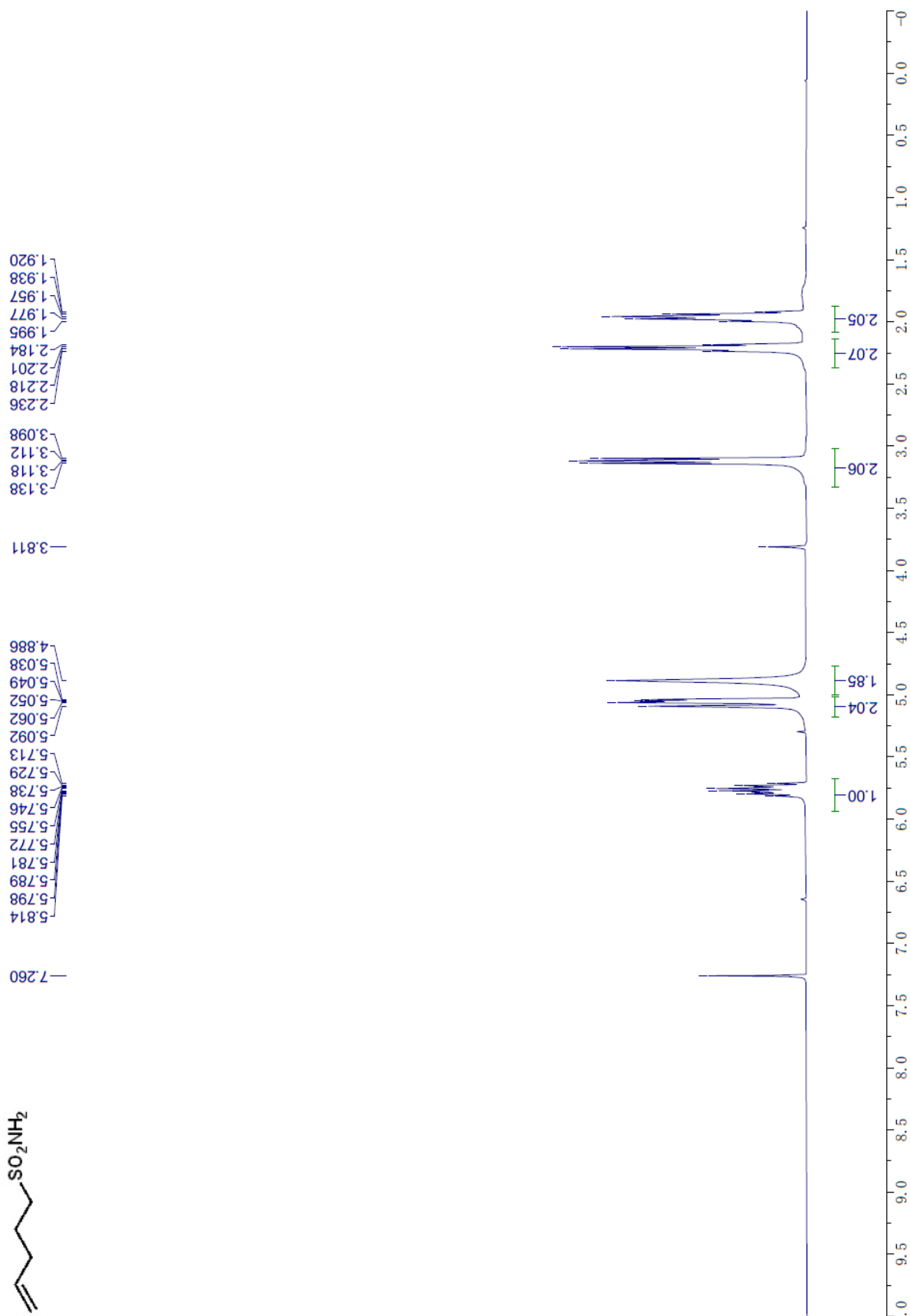
¹H NMR (400 MHz, CDCl₃) of **1r**

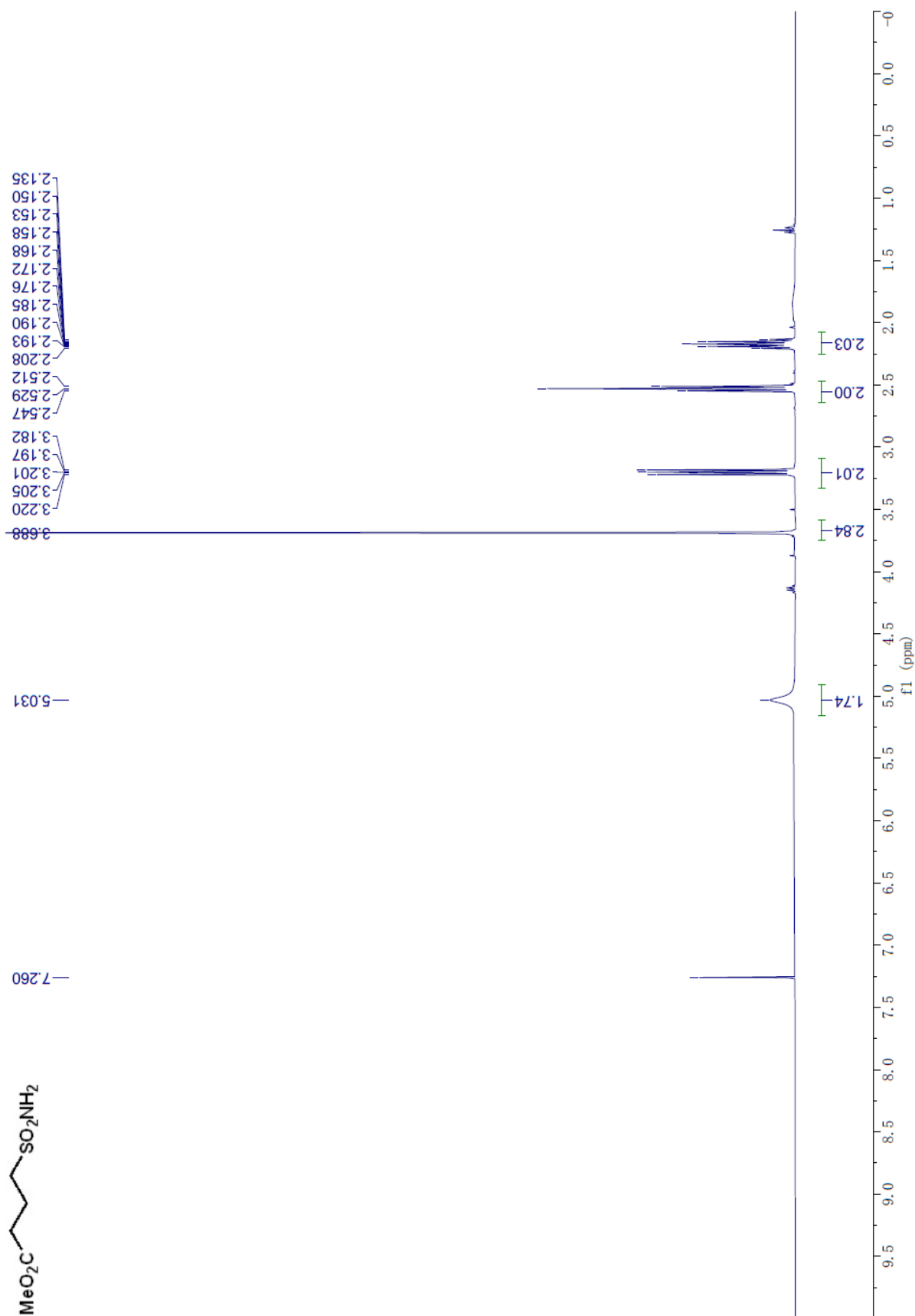


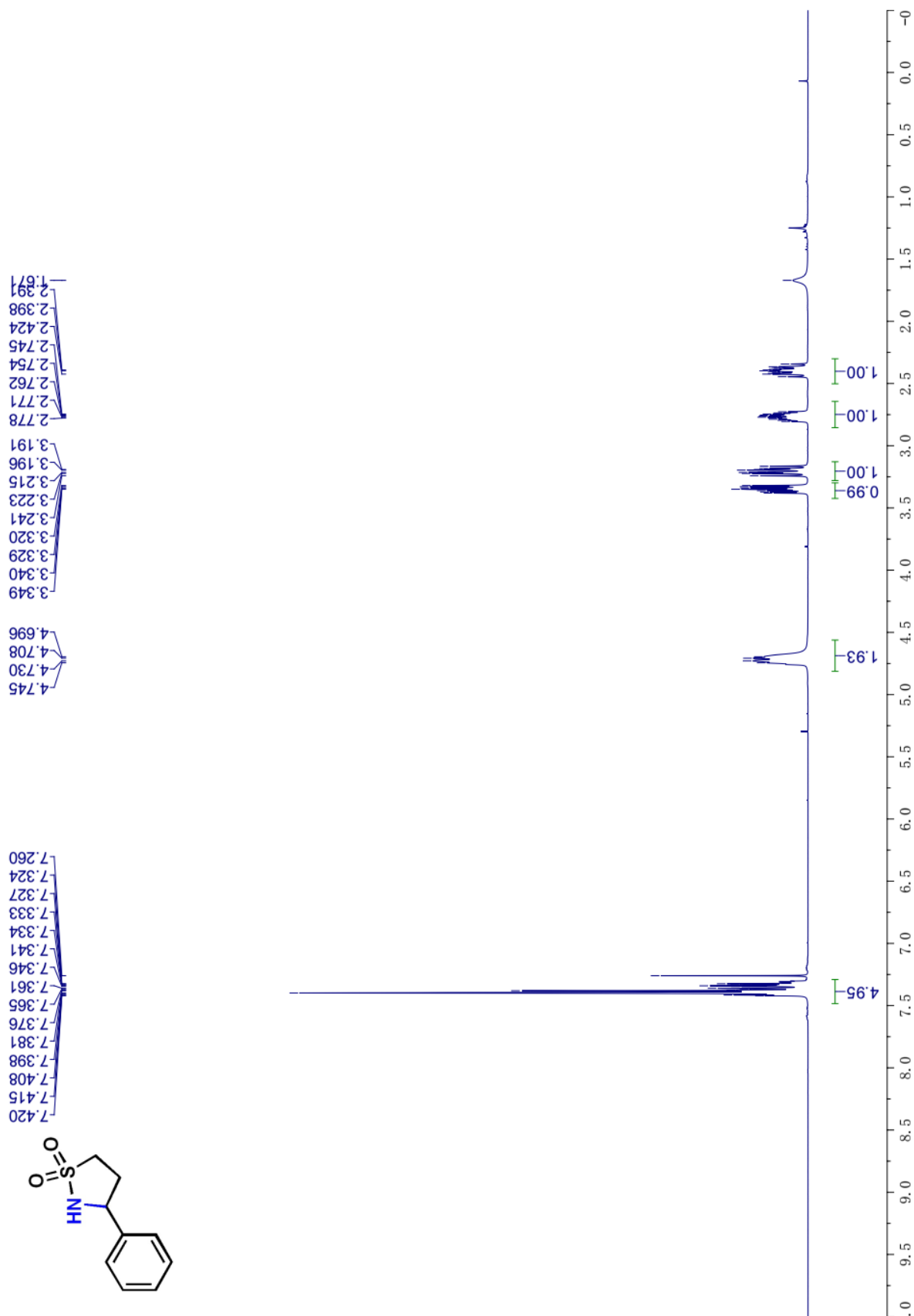
¹H NMR (400 MHz, CDCl₃) of **1s**

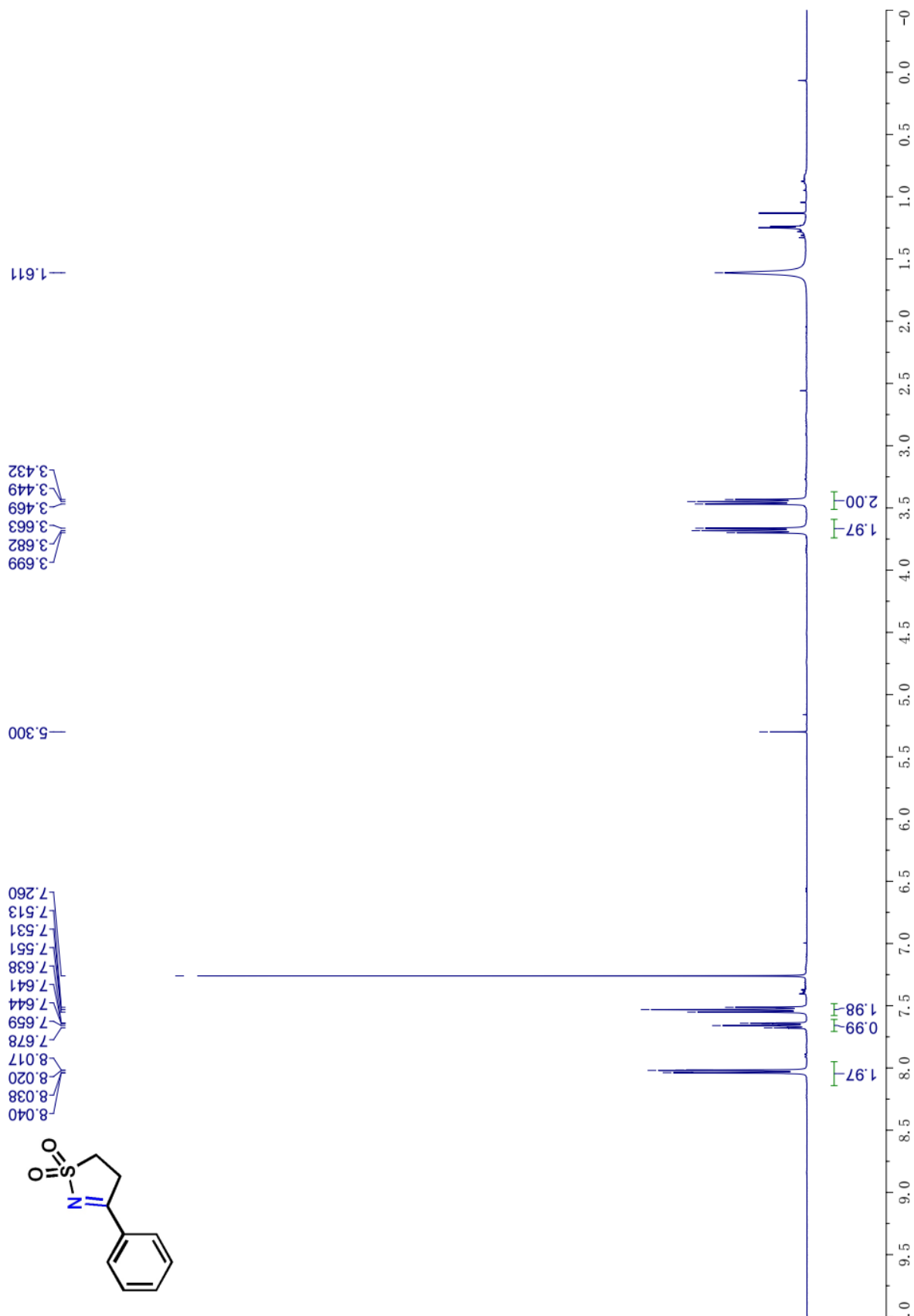
^{13}C NMR (100 MHz, CDCl_3) of **1s**

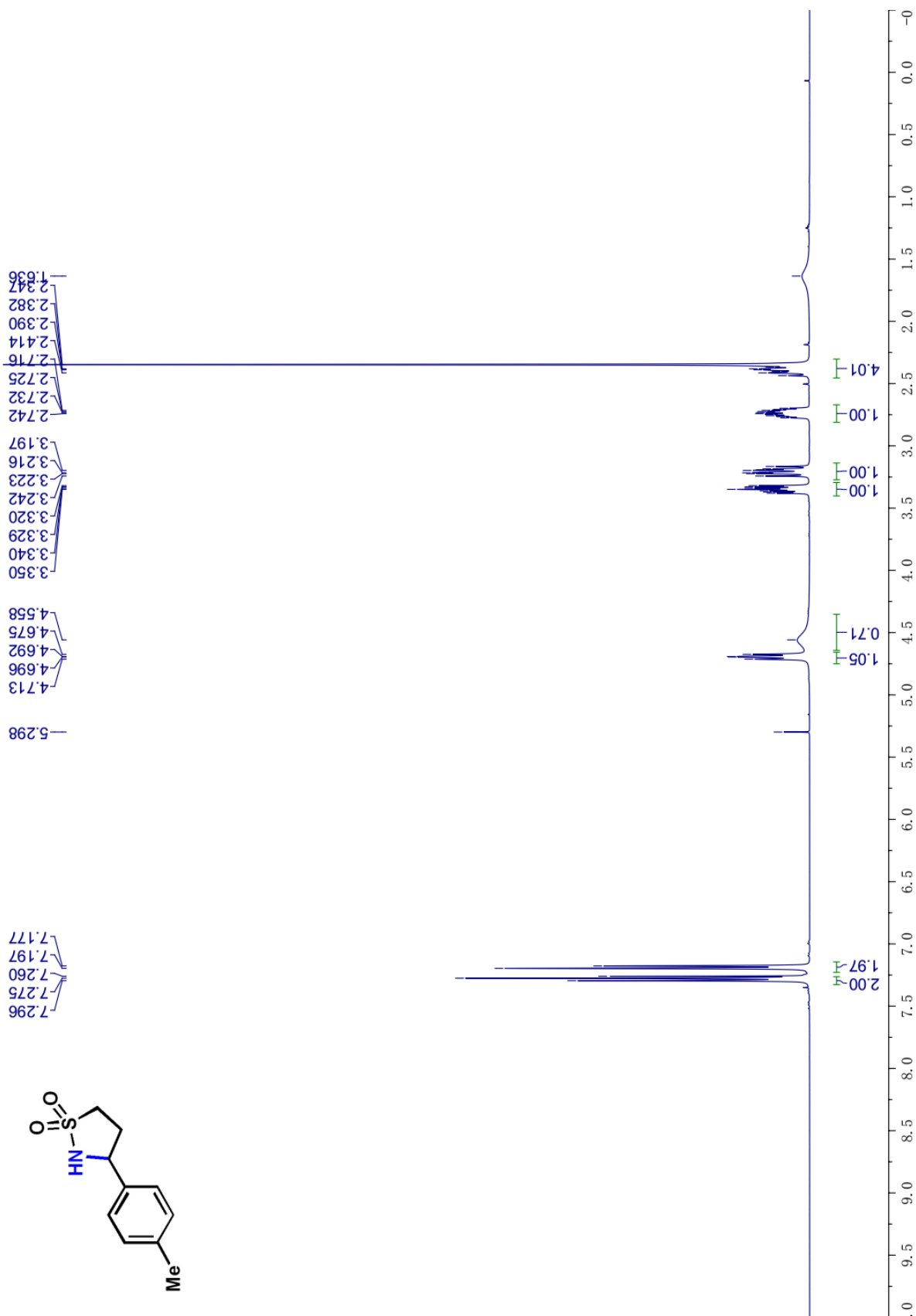
¹H NMR (400 MHz, CDCl₃) of **1t**

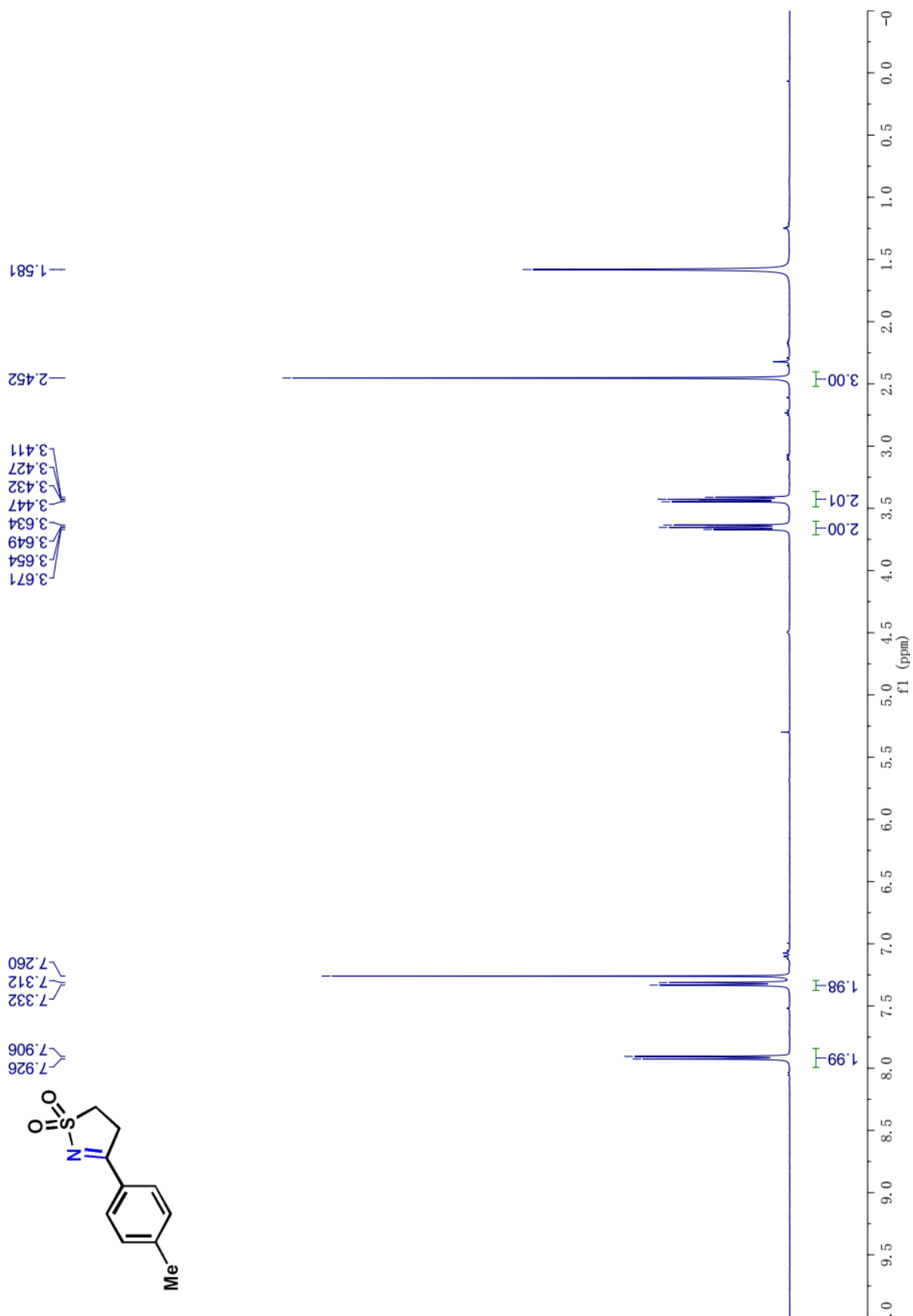
¹H NMR (400 MHz, CDCl₃) of **1u**

¹H NMR (400 MHz, CDCl₃) of **1v**

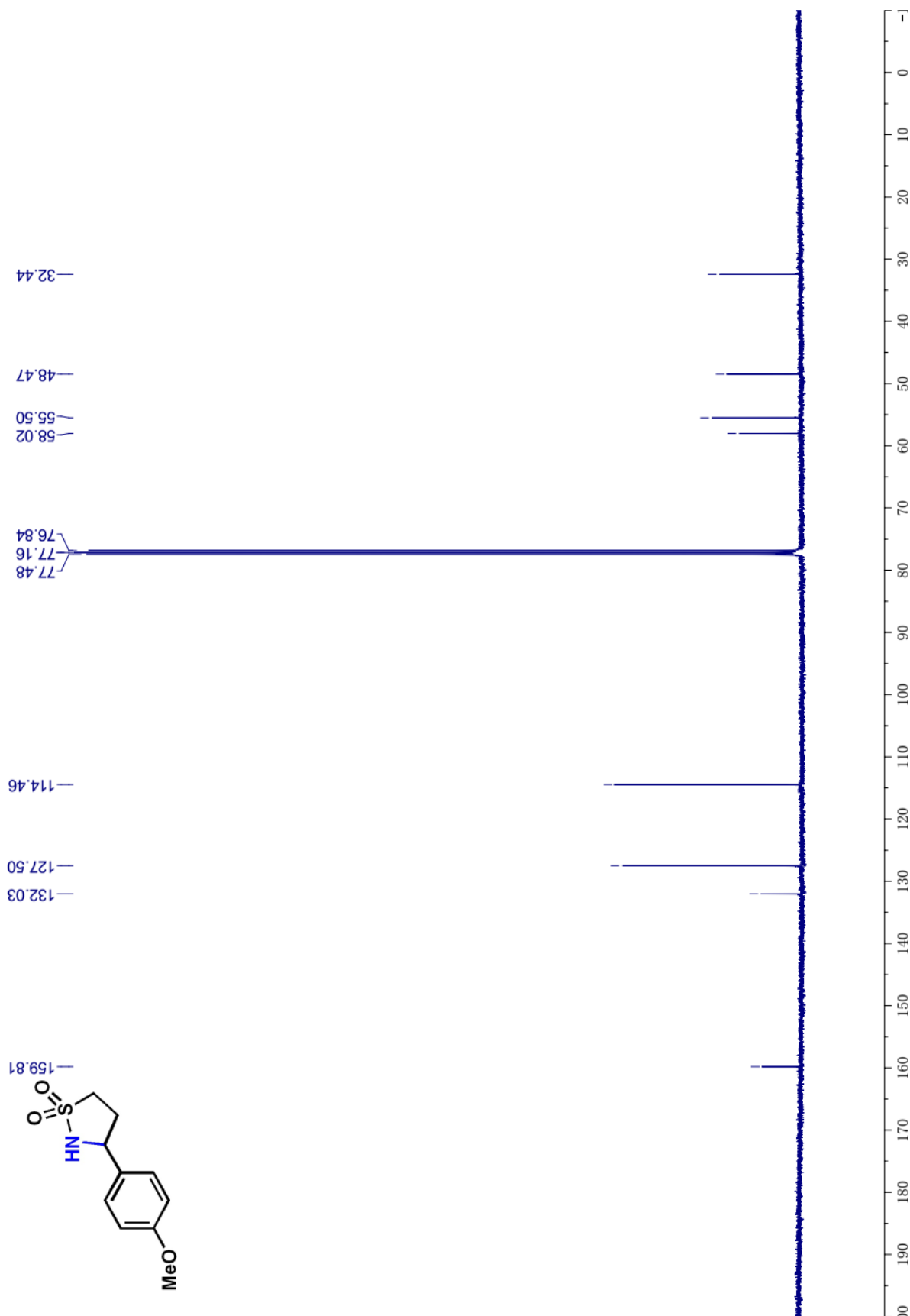
¹H NMR (400 MHz, CDCl₃) of **2a**

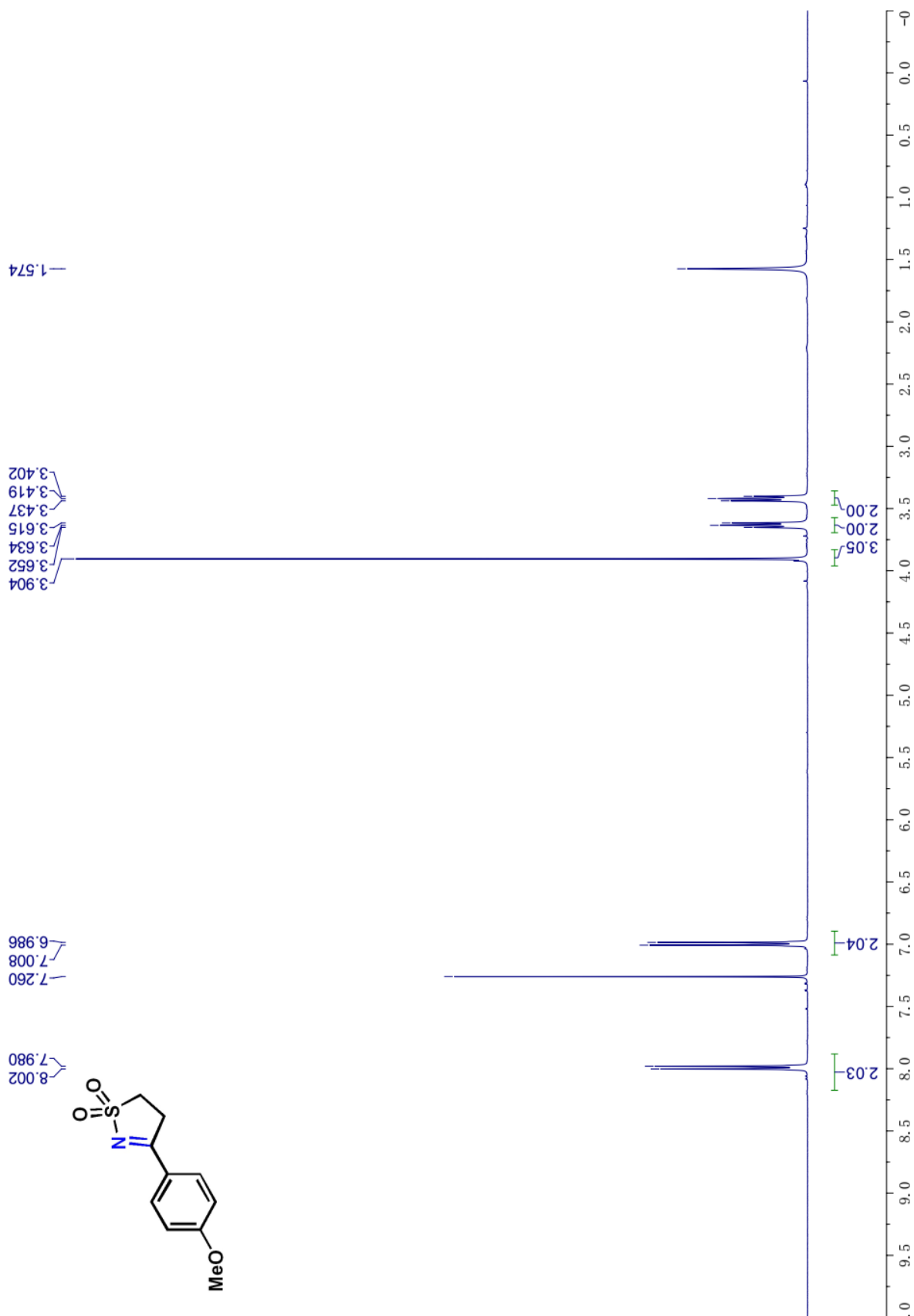
¹H NMR (400 MHz, CDCl₃) of **3a**

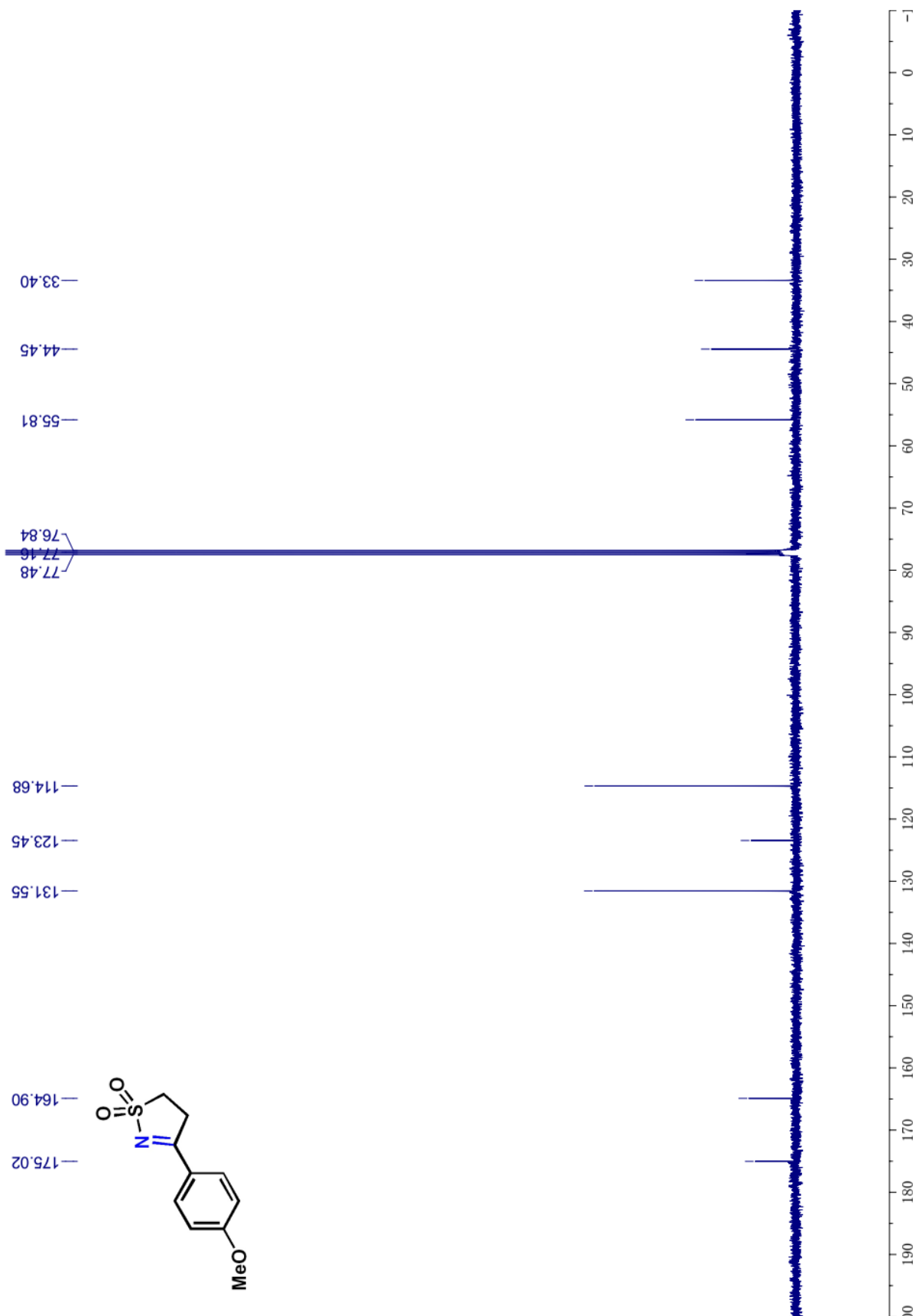
¹H NMR (400 MHz, CDCl₃) of **2b**

¹H NMR (400 MHz, CDCl₃) of **3b**

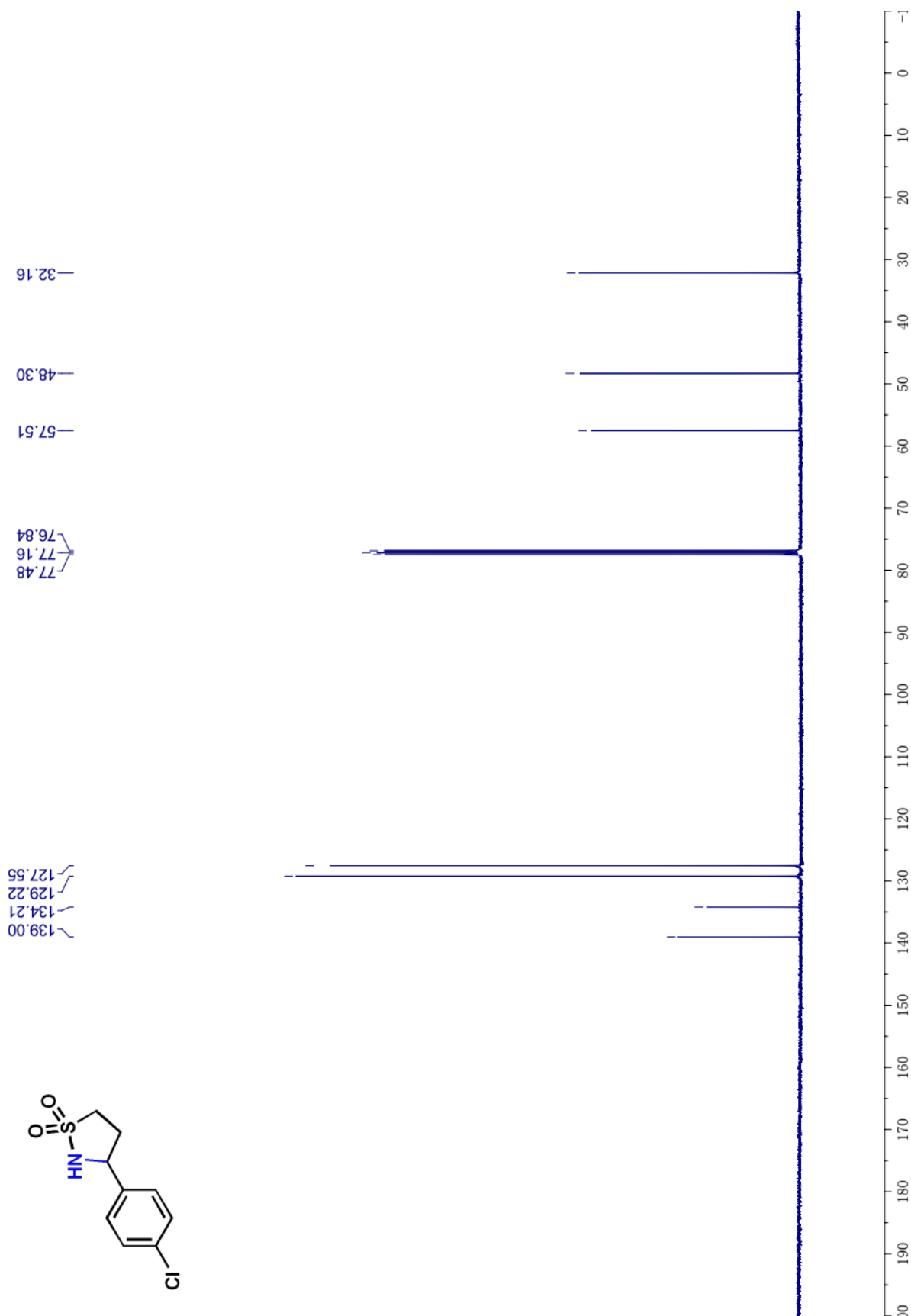
¹H NMR (400 MHz, CDCl₃) of **2c**

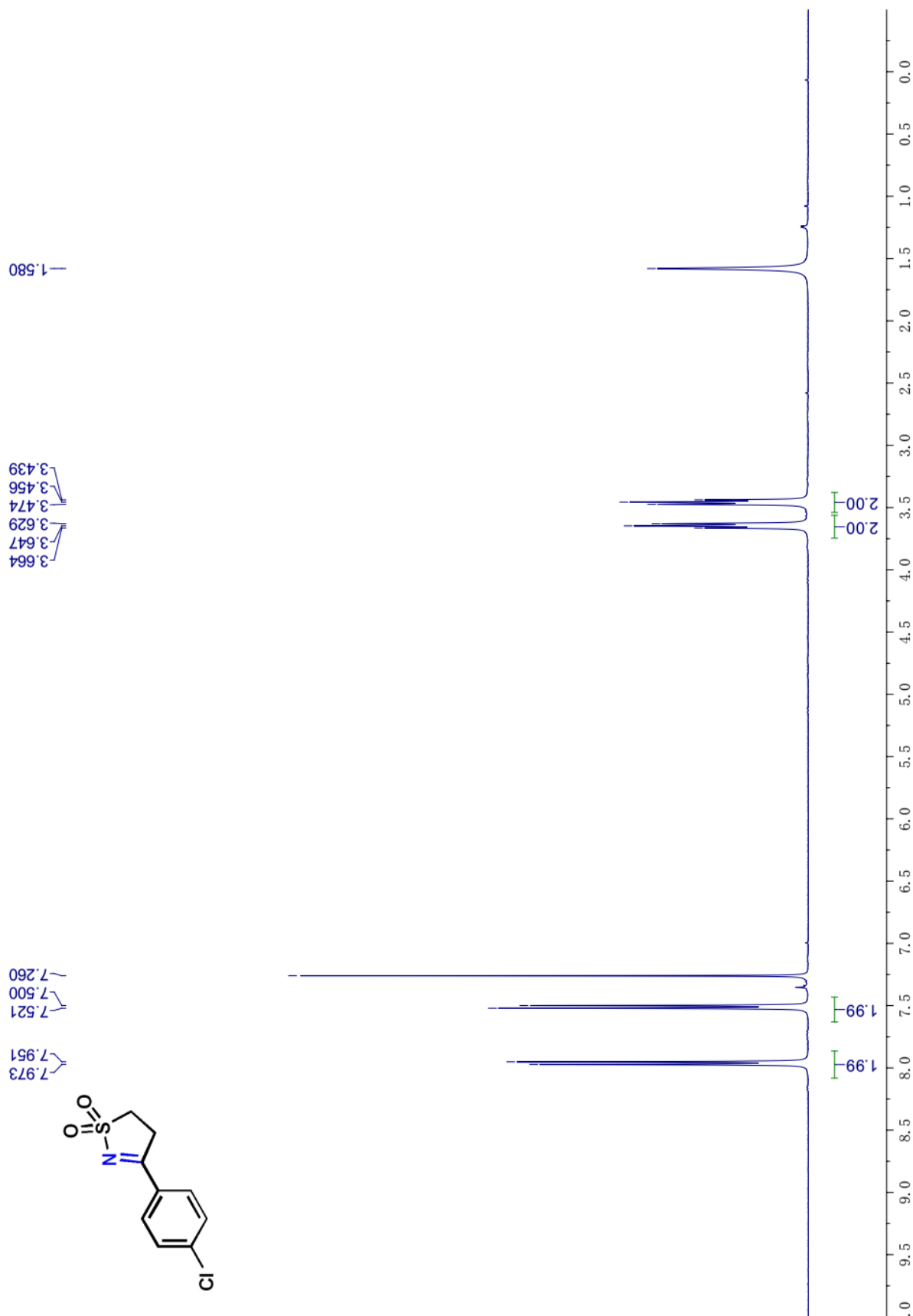
^{13}C NMR (100 MHz, CDCl_3) of **2c**

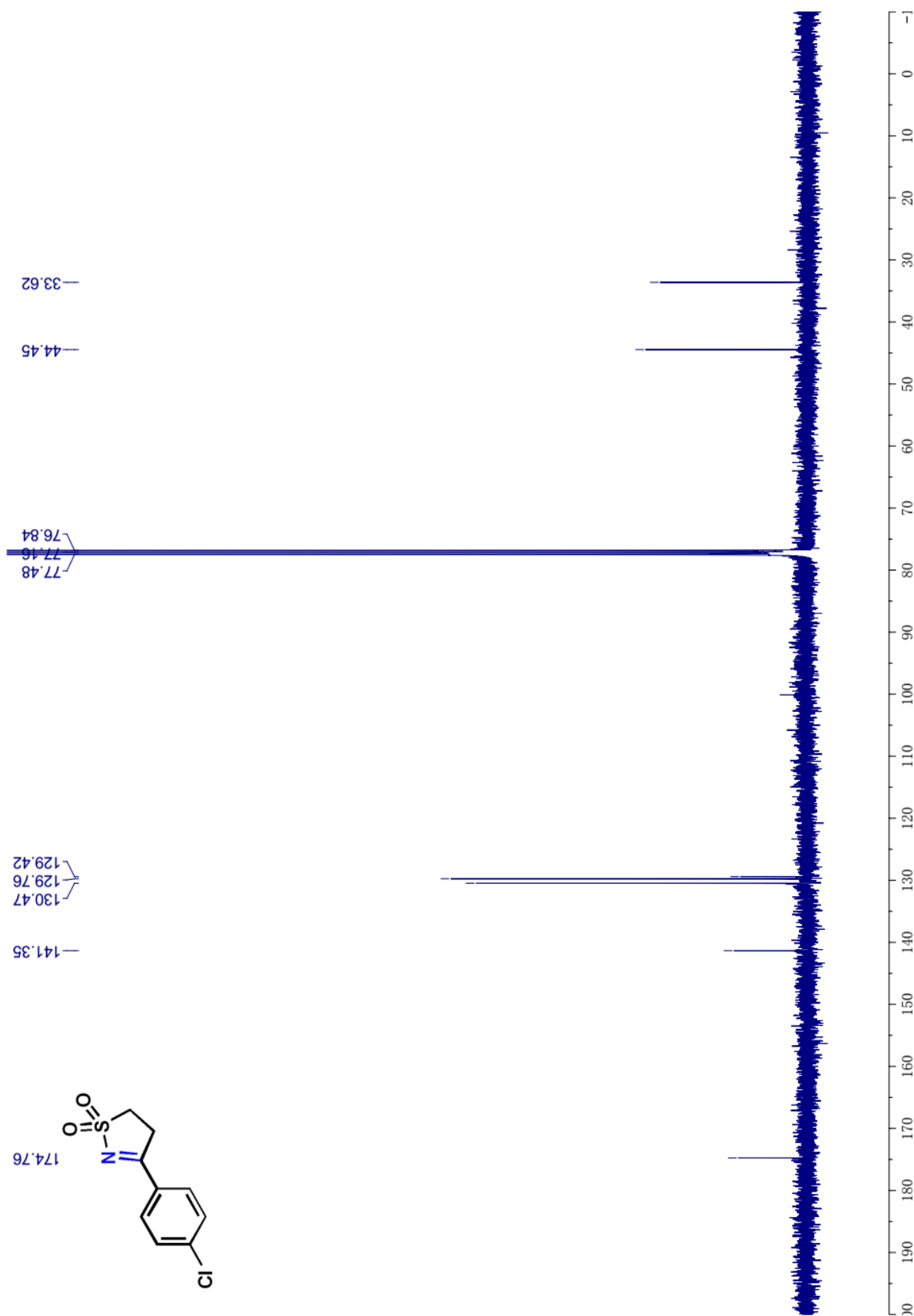
¹H NMR (400 MHz, CDCl₃) of **3c**

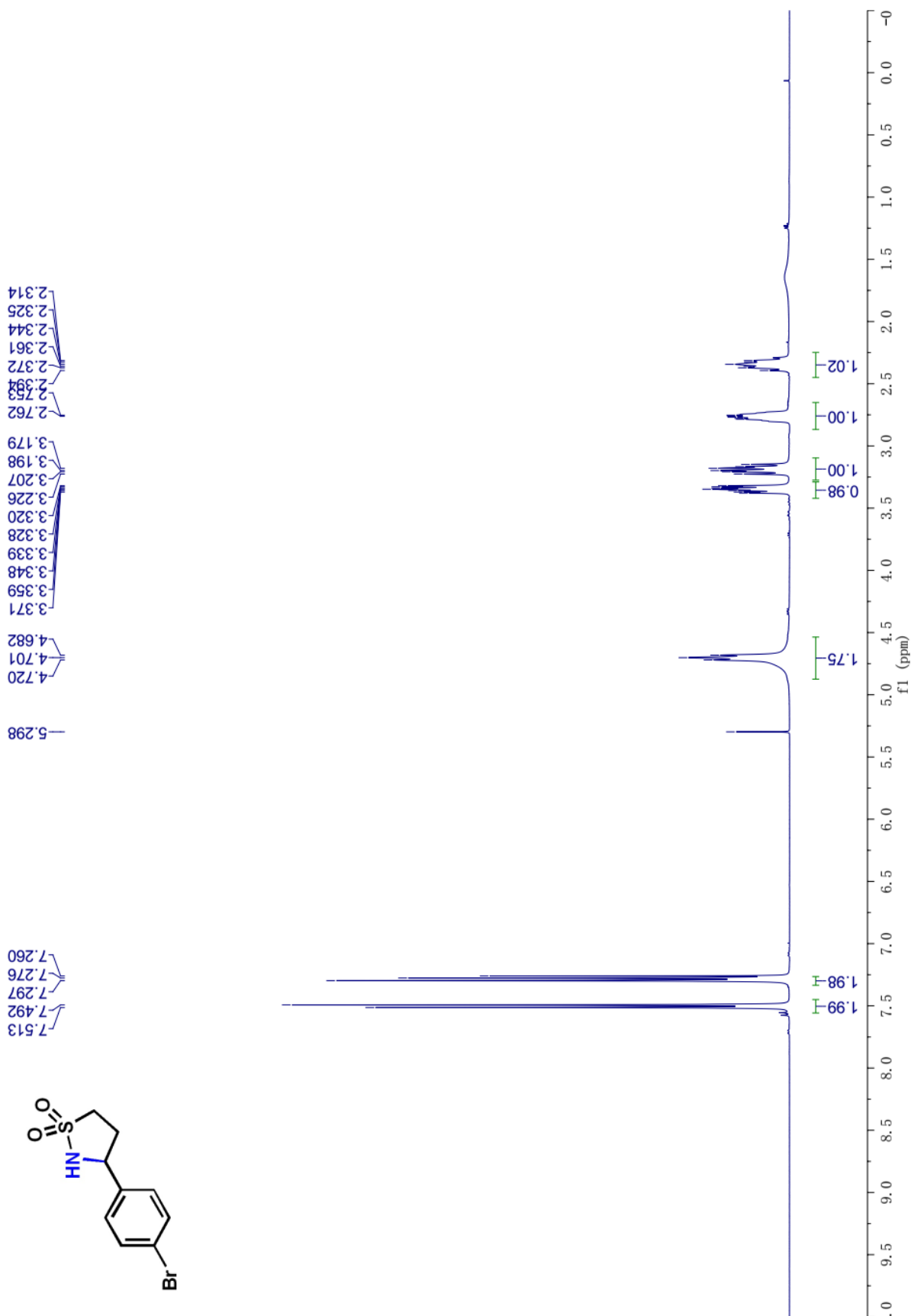
^{13}C NMR (100 MHz, CDCl_3) of **3c**

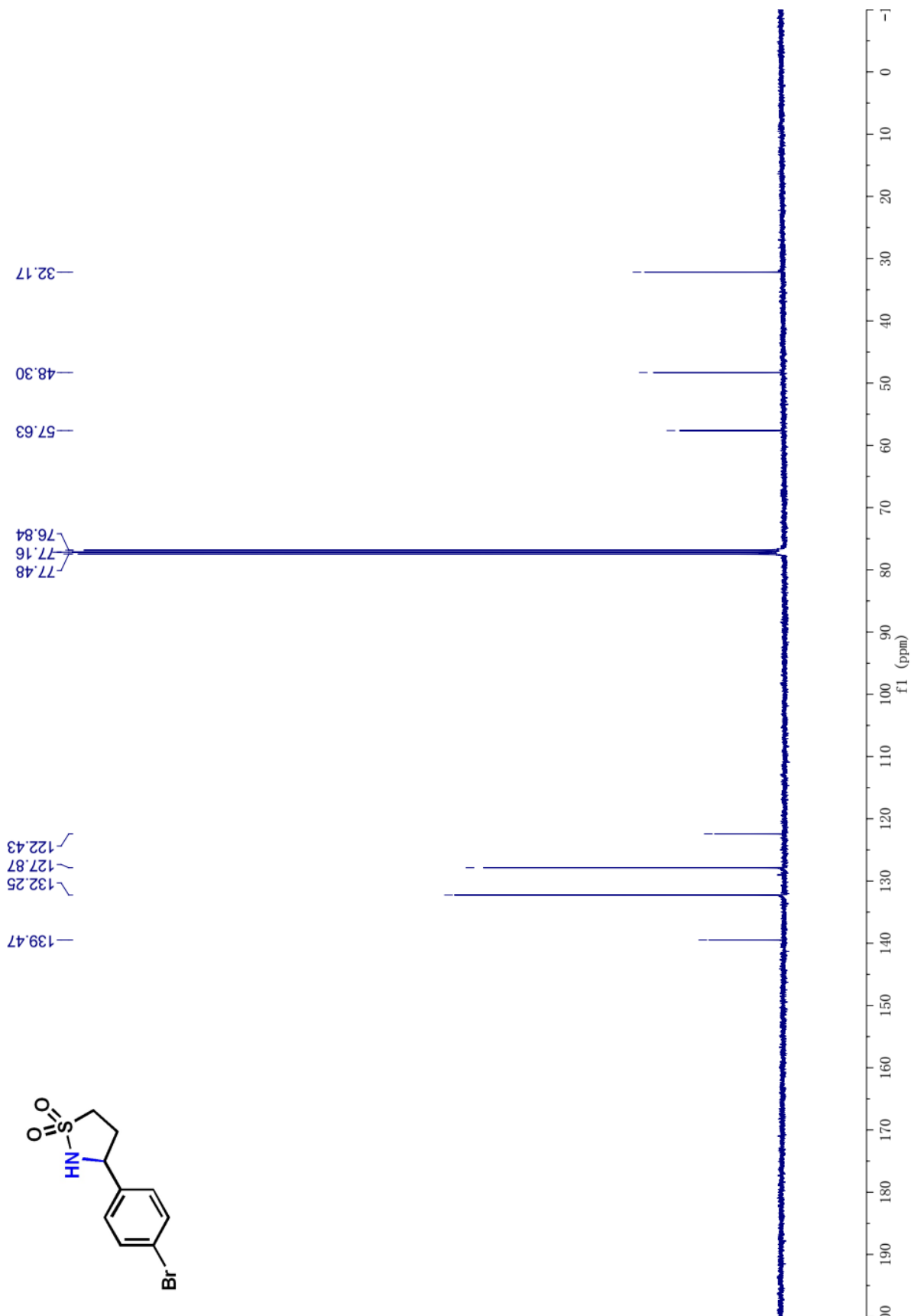
¹H NMR (400 MHz, CDCl₃) of **2d**

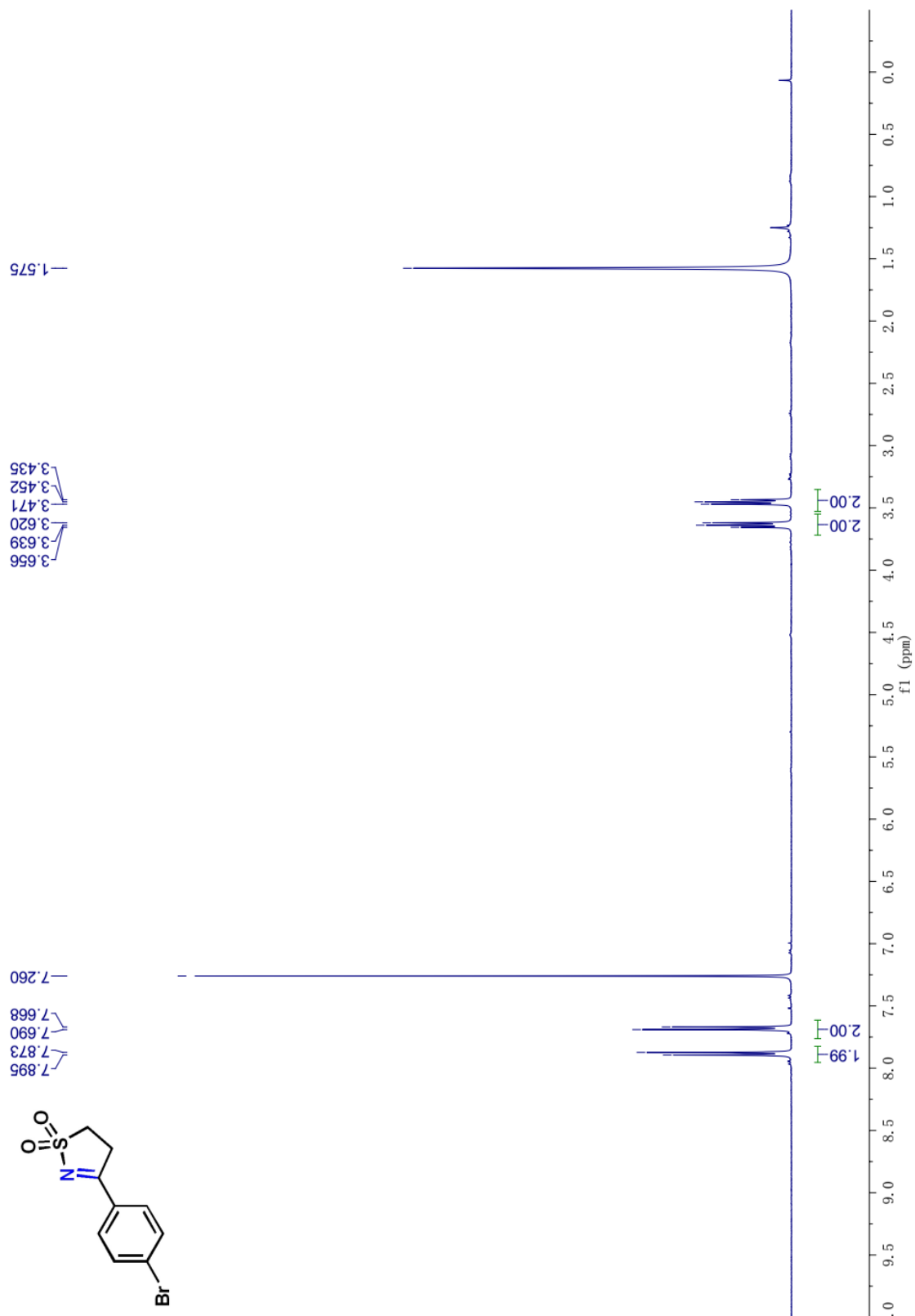
^{13}C NMR (100 MHz, CDCl_3) of **2d**

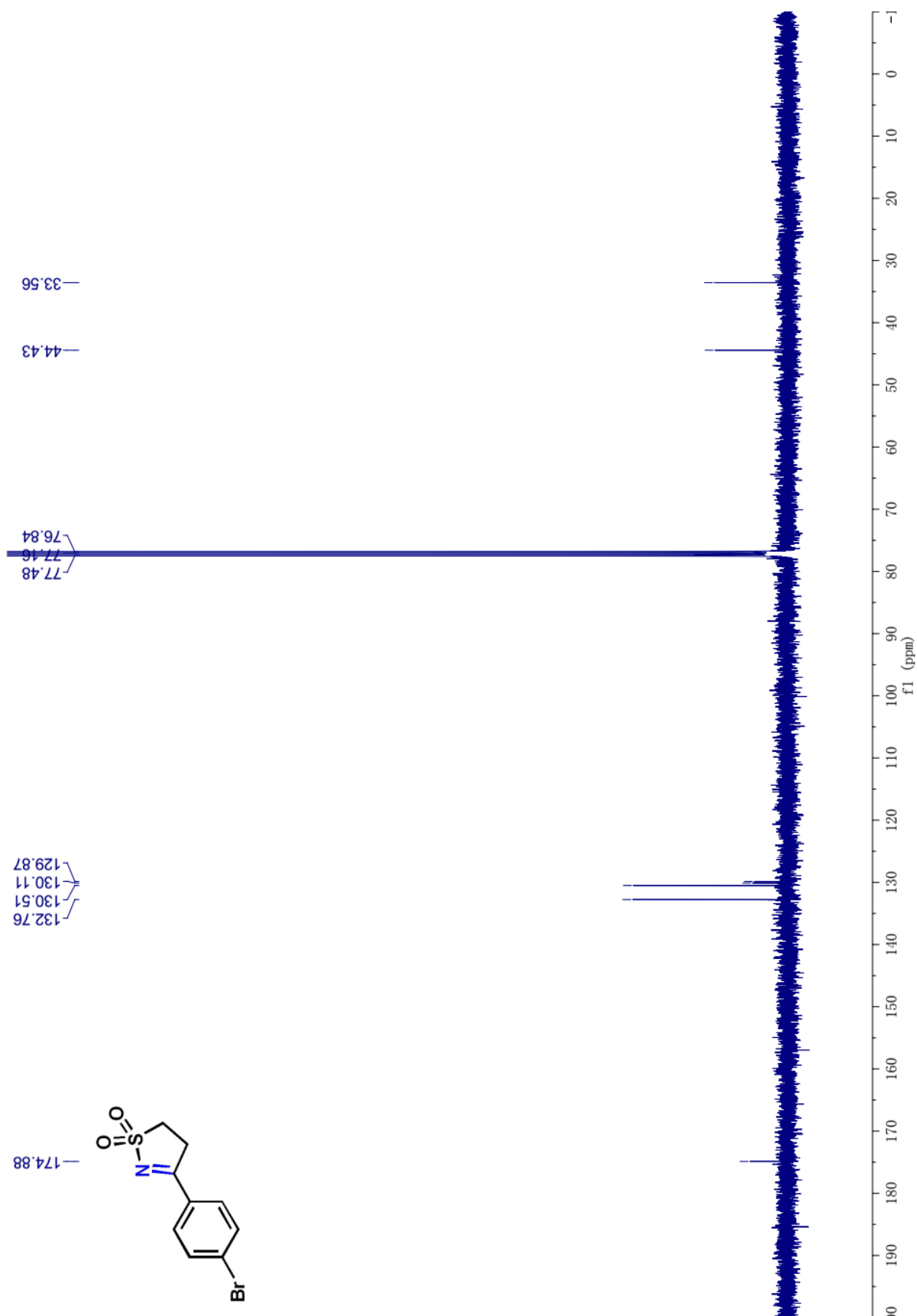
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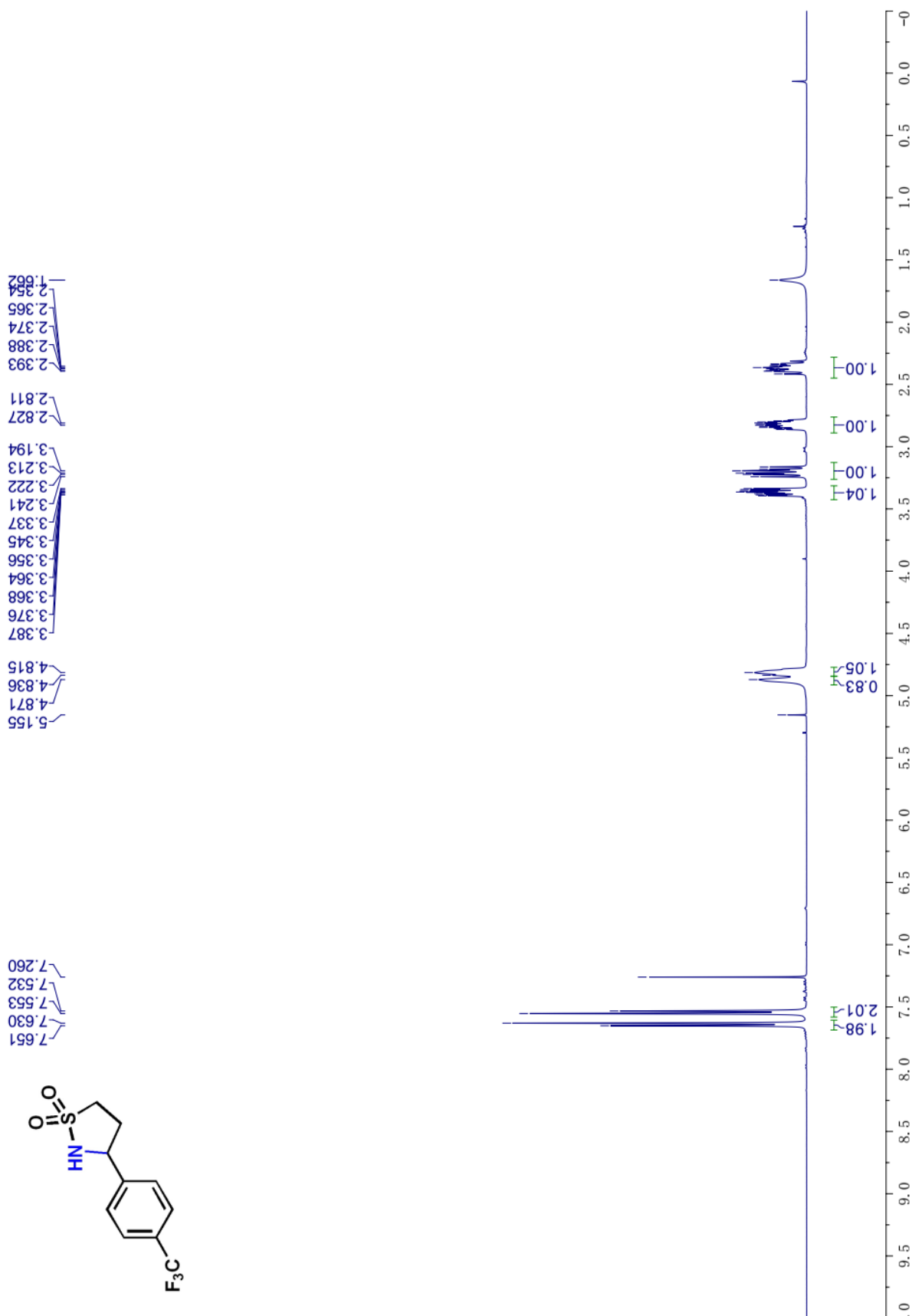
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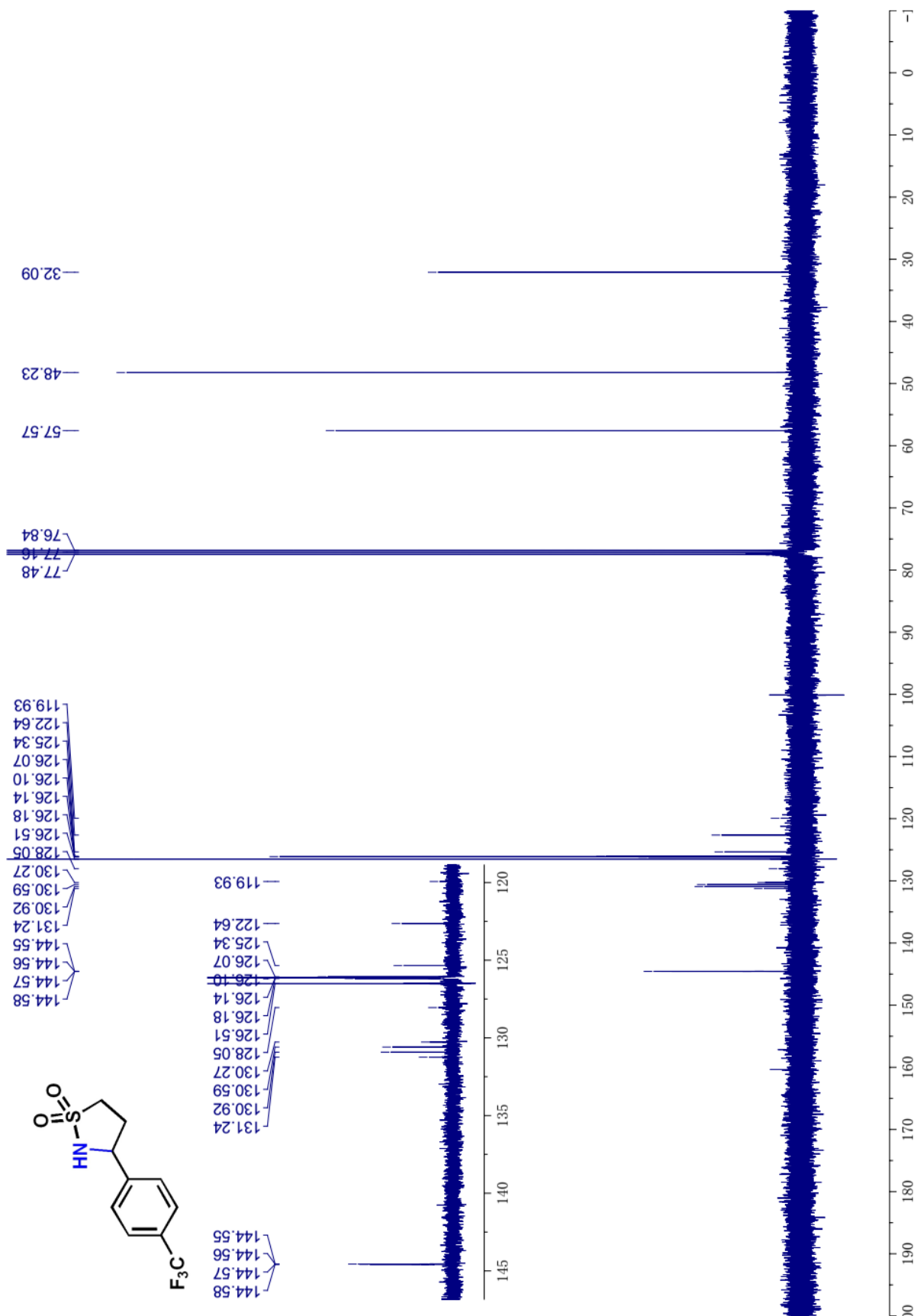
¹H NMR (400 MHz, CDCl₃) of **2e**

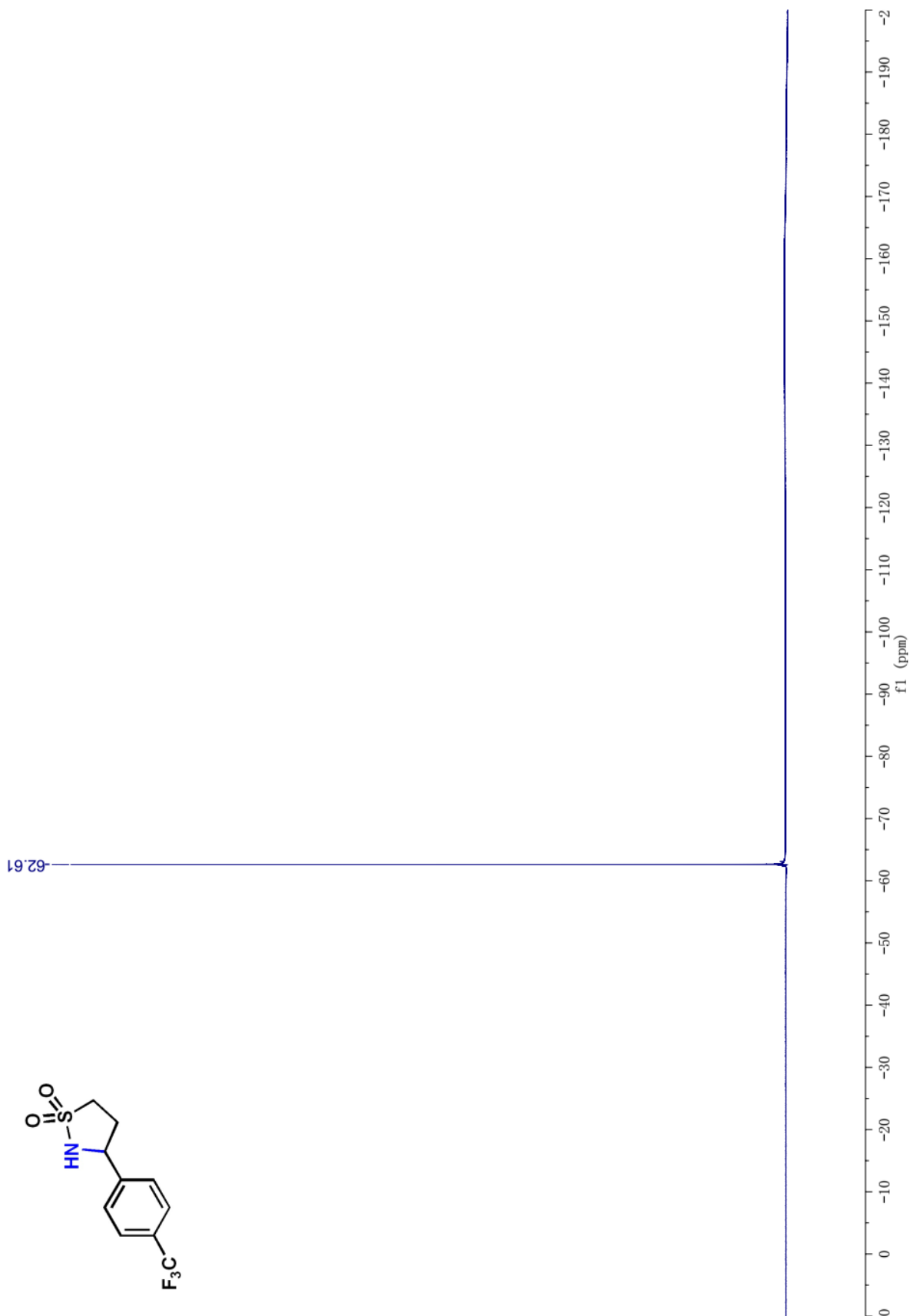
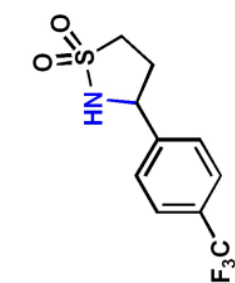
^{13}C NMR (100 MHz, CDCl_3) of **2e**

¹H NMR (400 MHz, CDCl₃) of **3e**

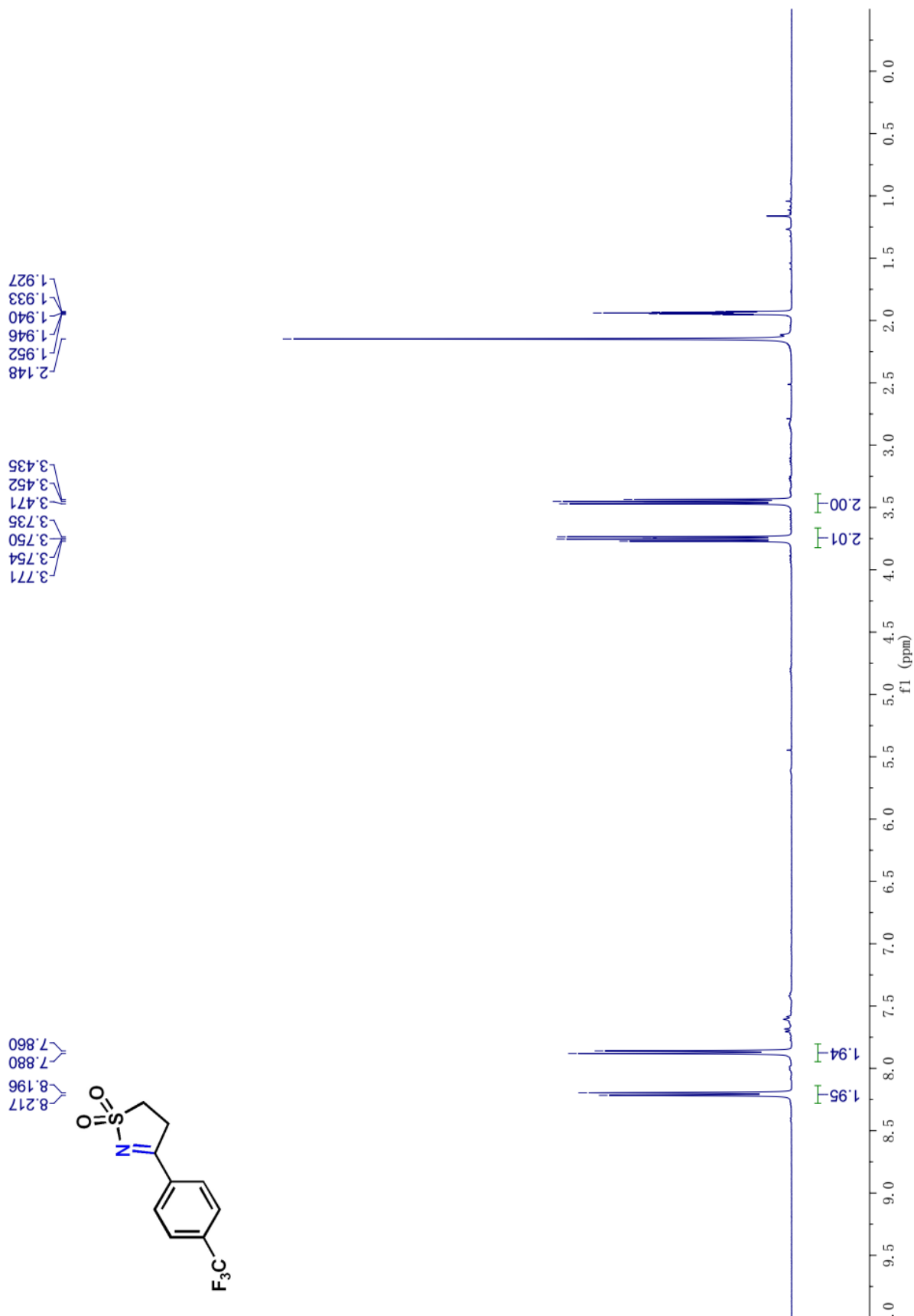
^{13}C NMR (100 MHz, CDCl_3) of **3e**

¹H NMR (400 MHz, CDCl₃) of **2f**

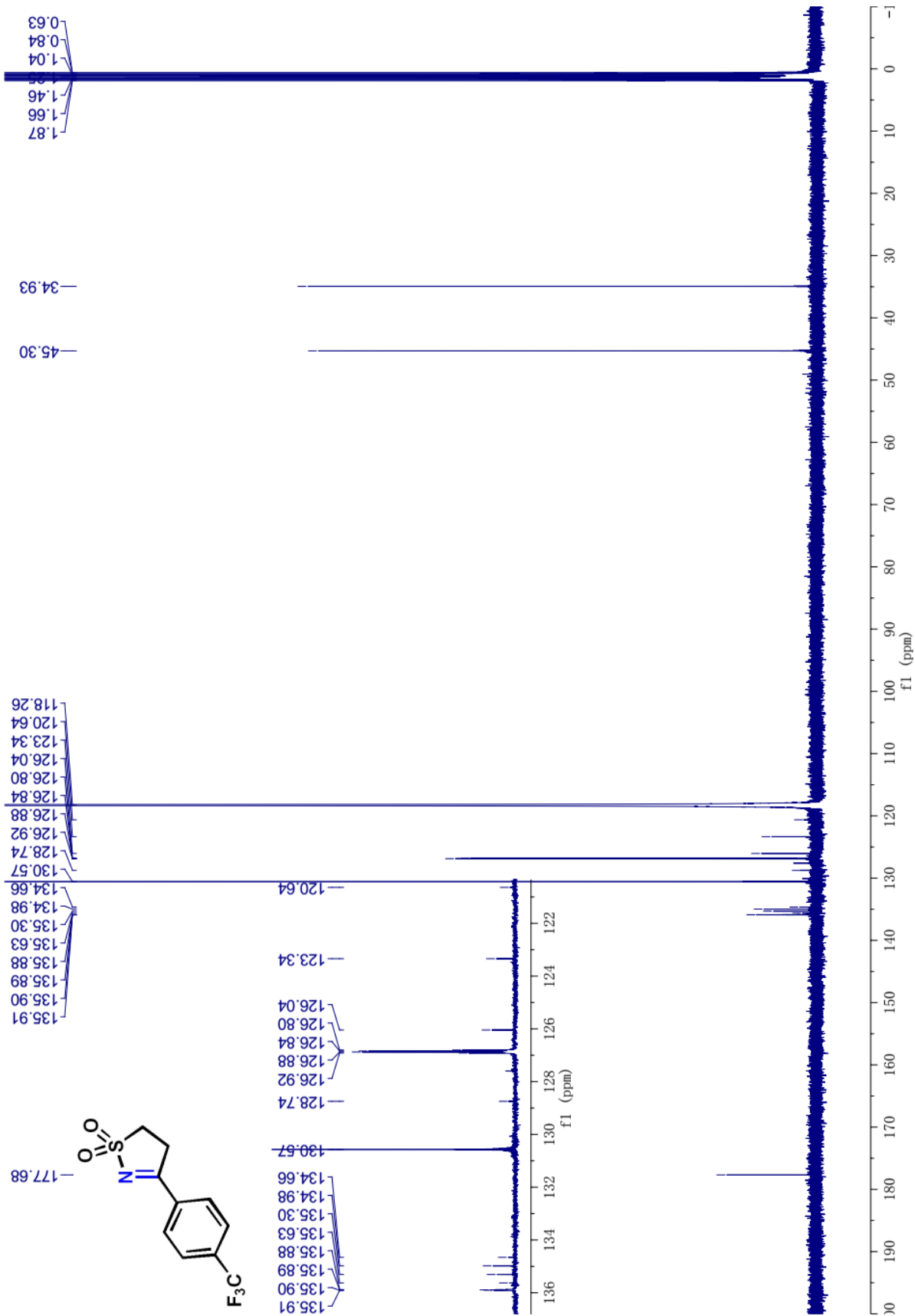
^{13}C NMR (100 MHz, CDCl_3) of **2f**

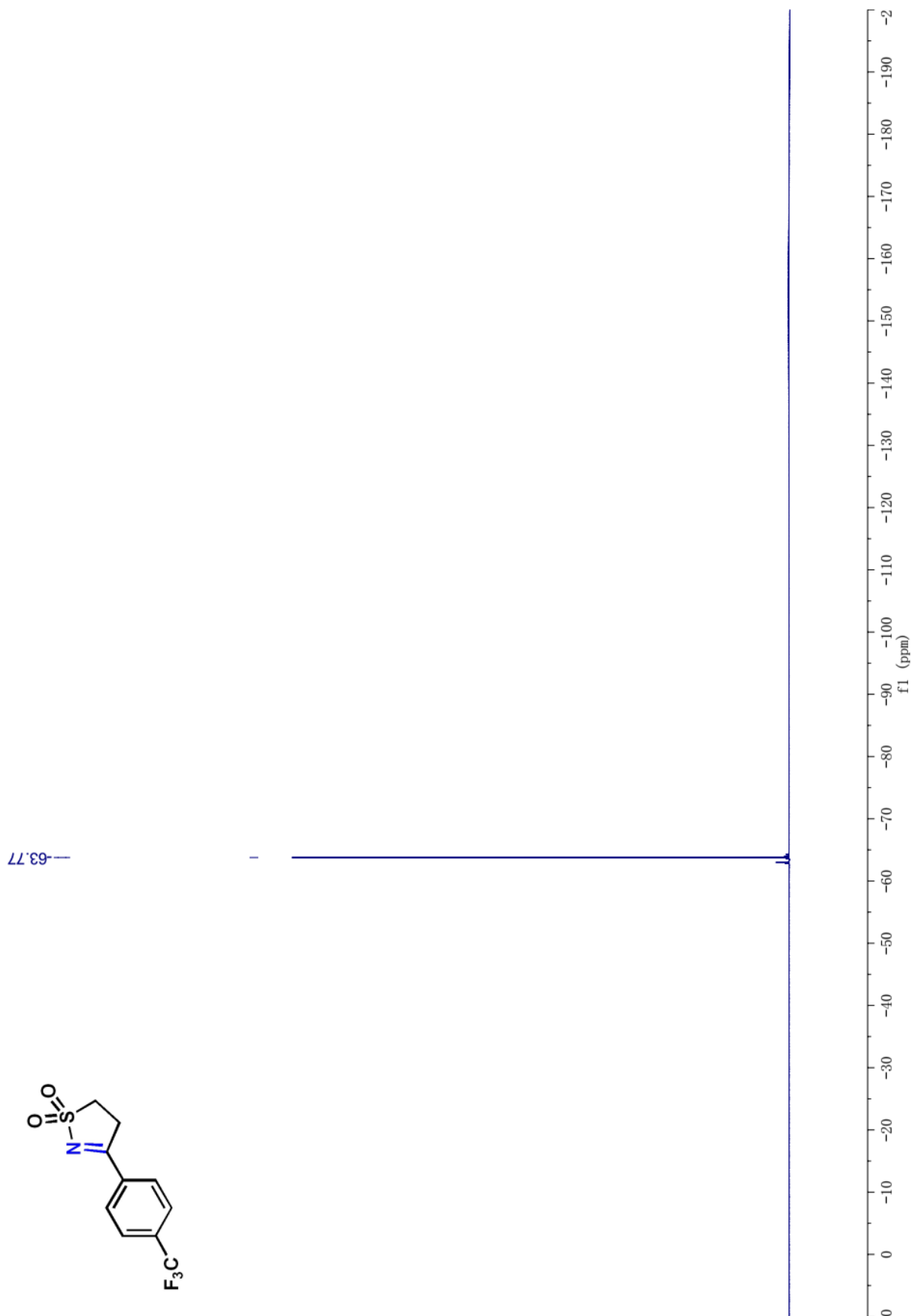


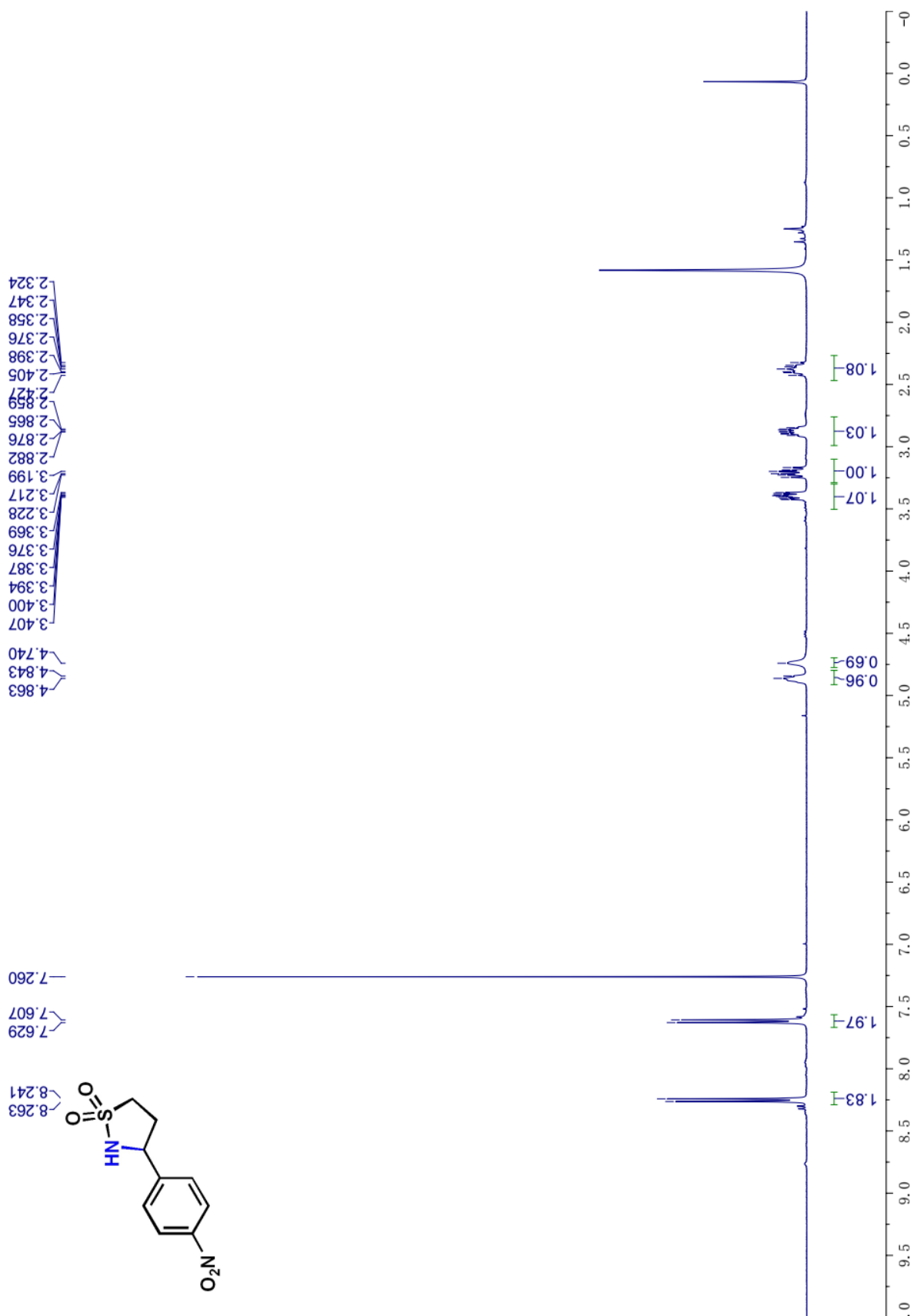
¹⁹F NMR (376 MHz, CDCl₃) of **2f**

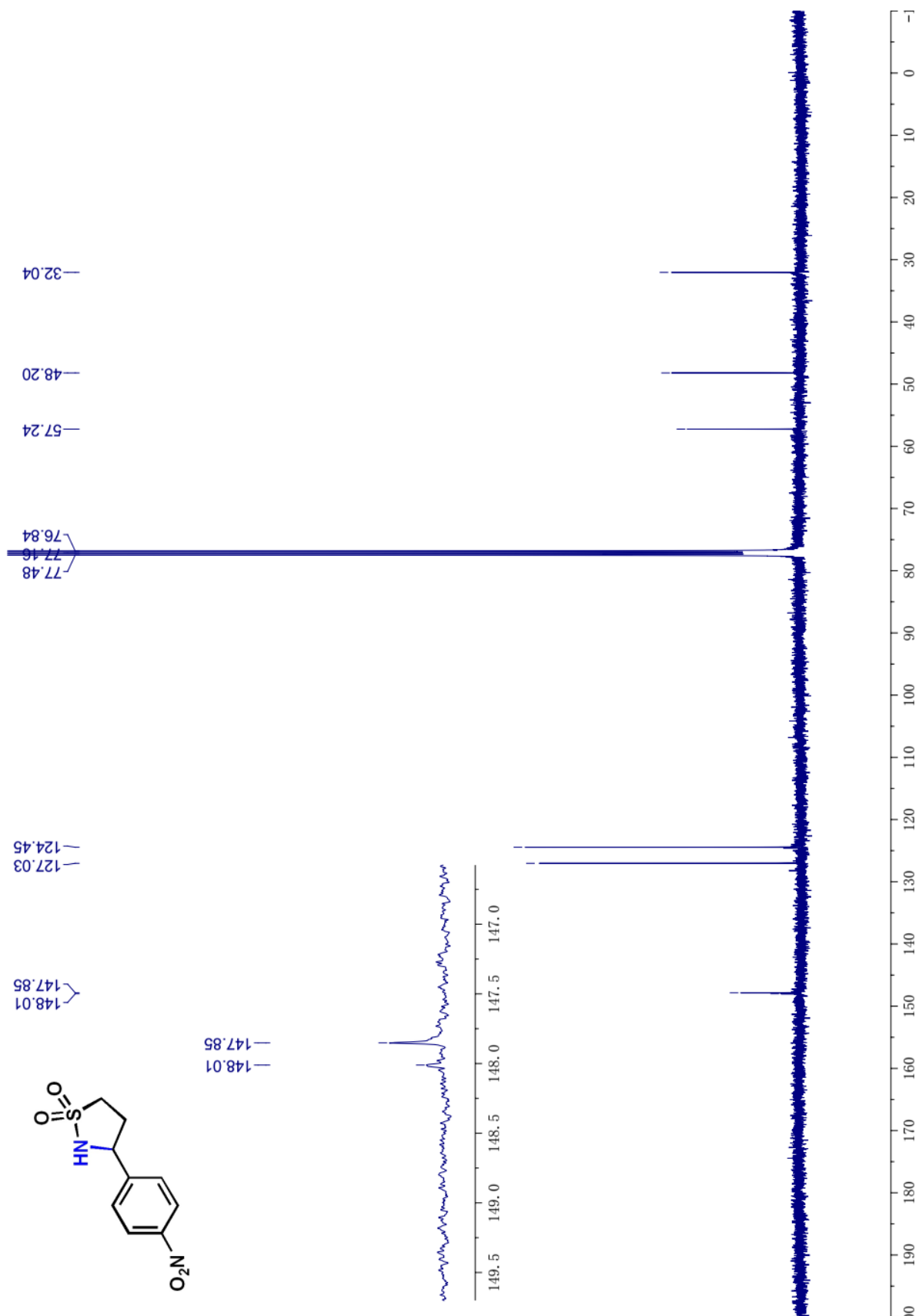
¹H NMR (400 MHz, CD₃CN) of **3f**

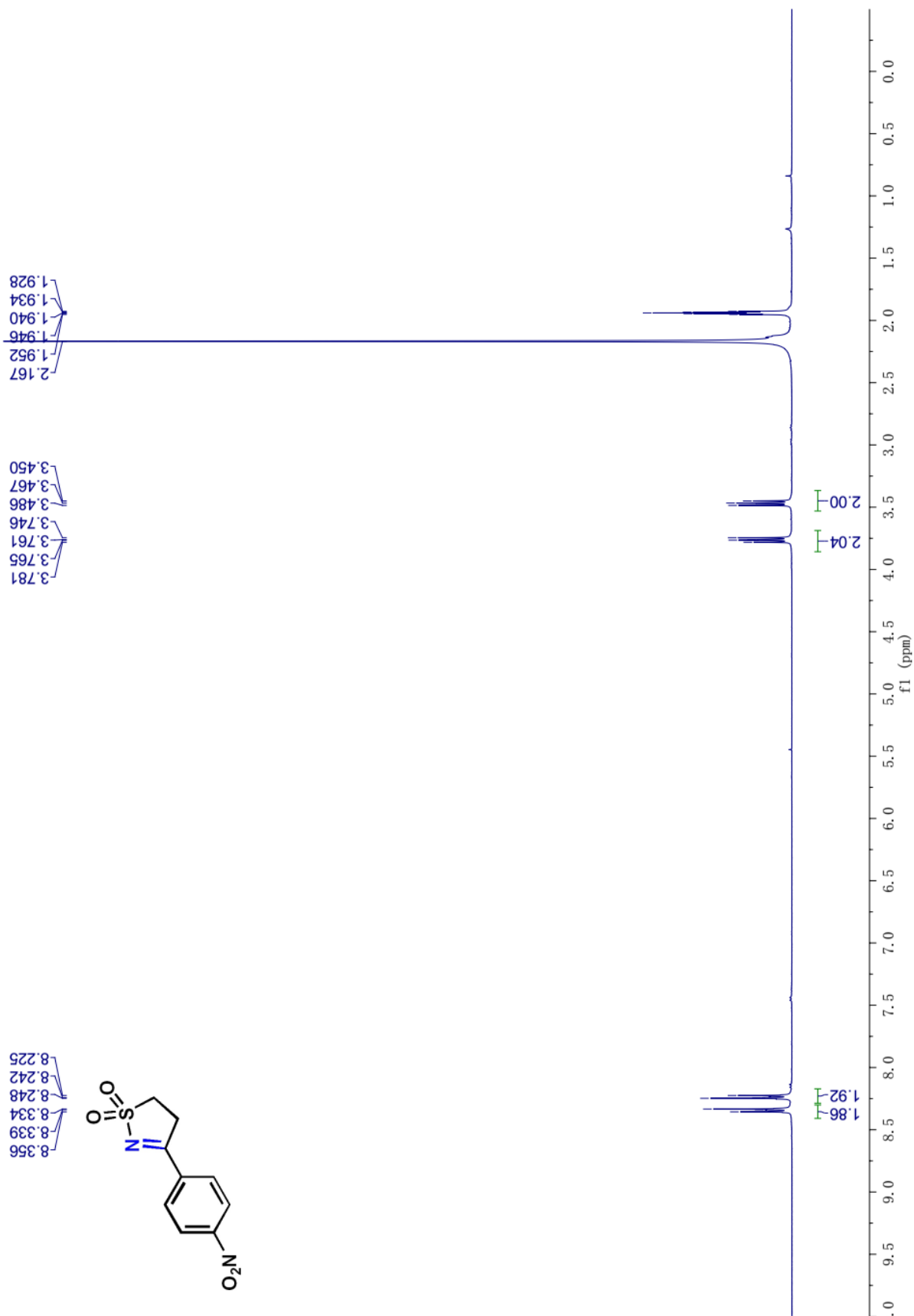
¹³C NMR (100 MHz, CD₃CN) of **3f**

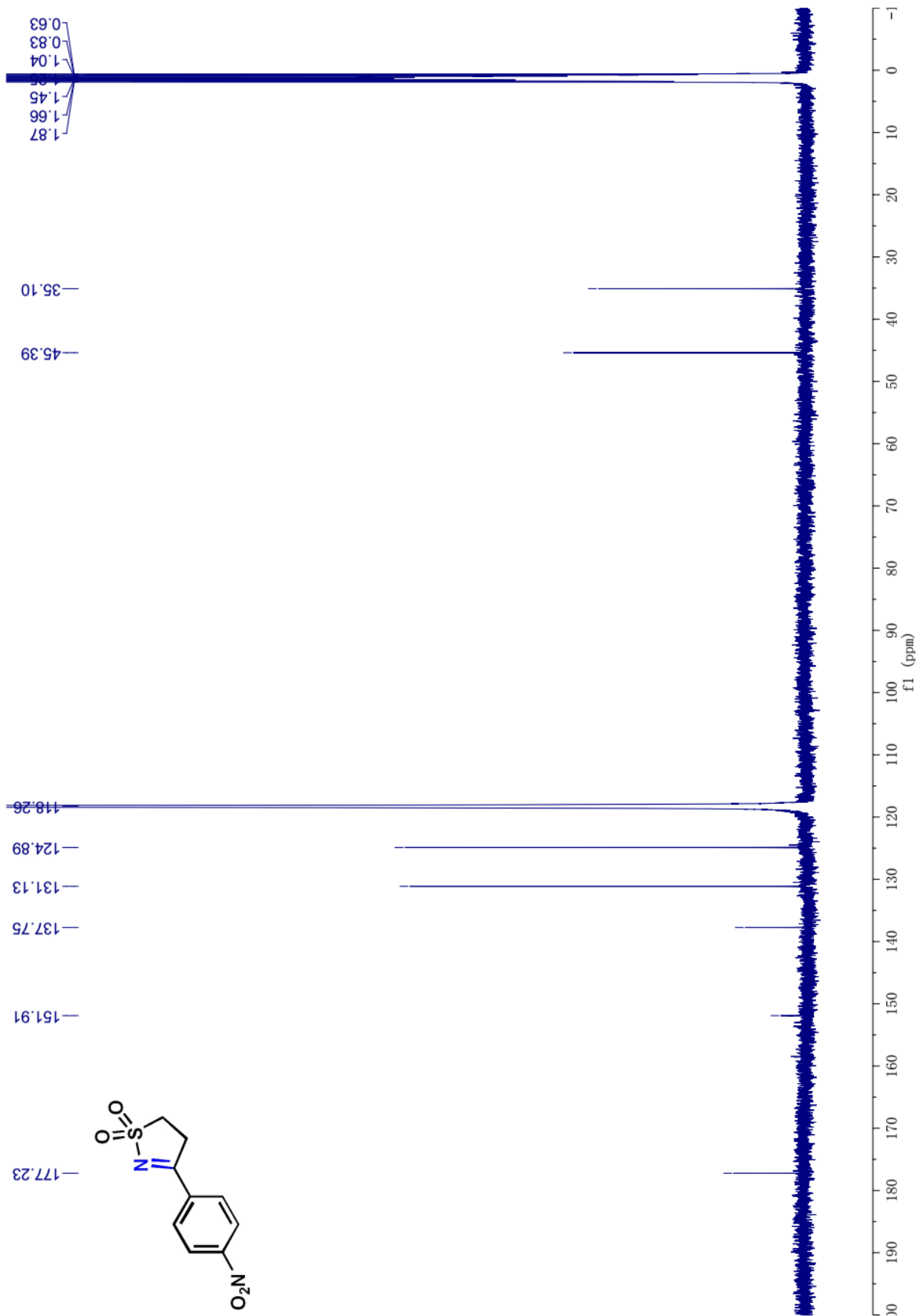


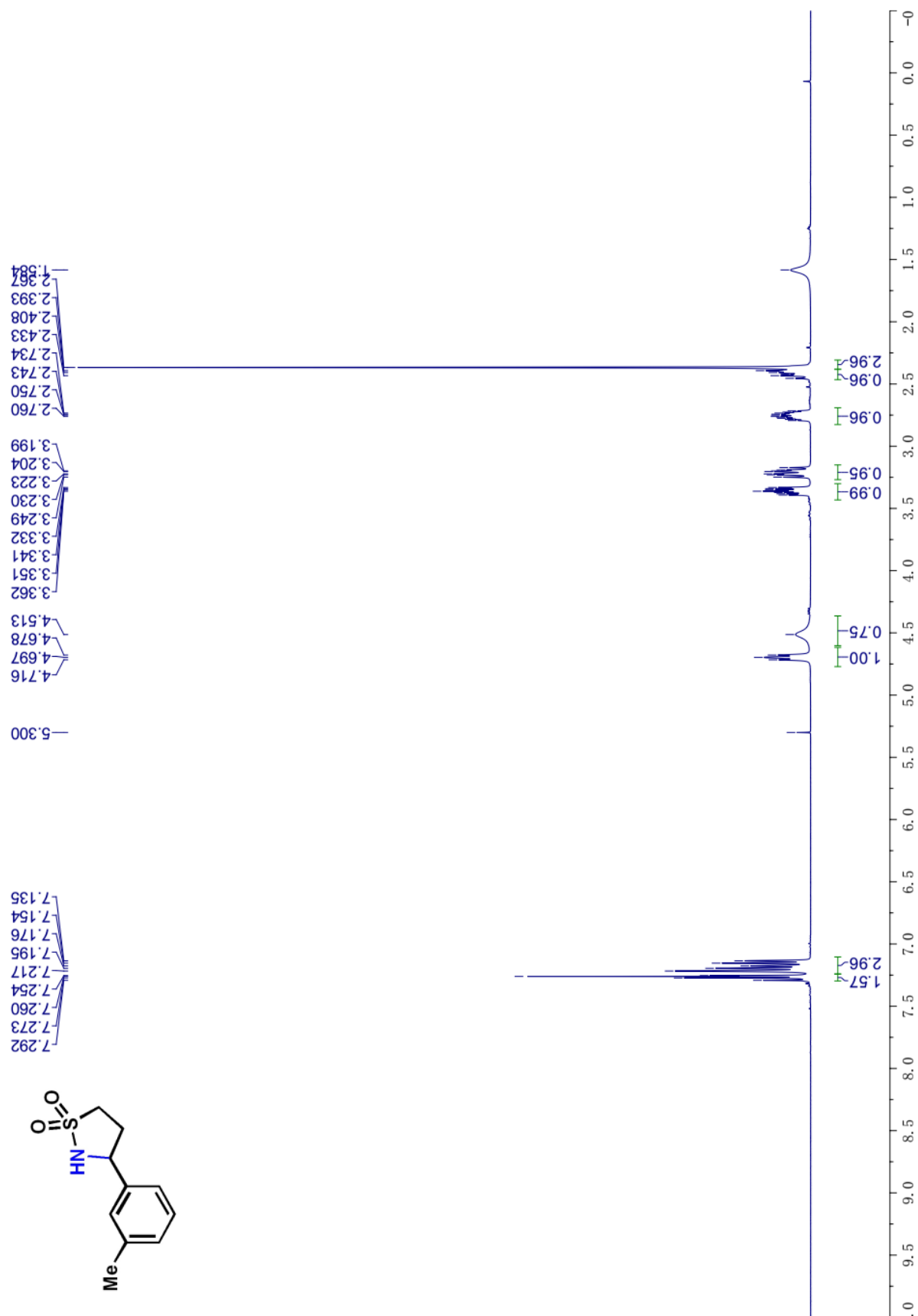


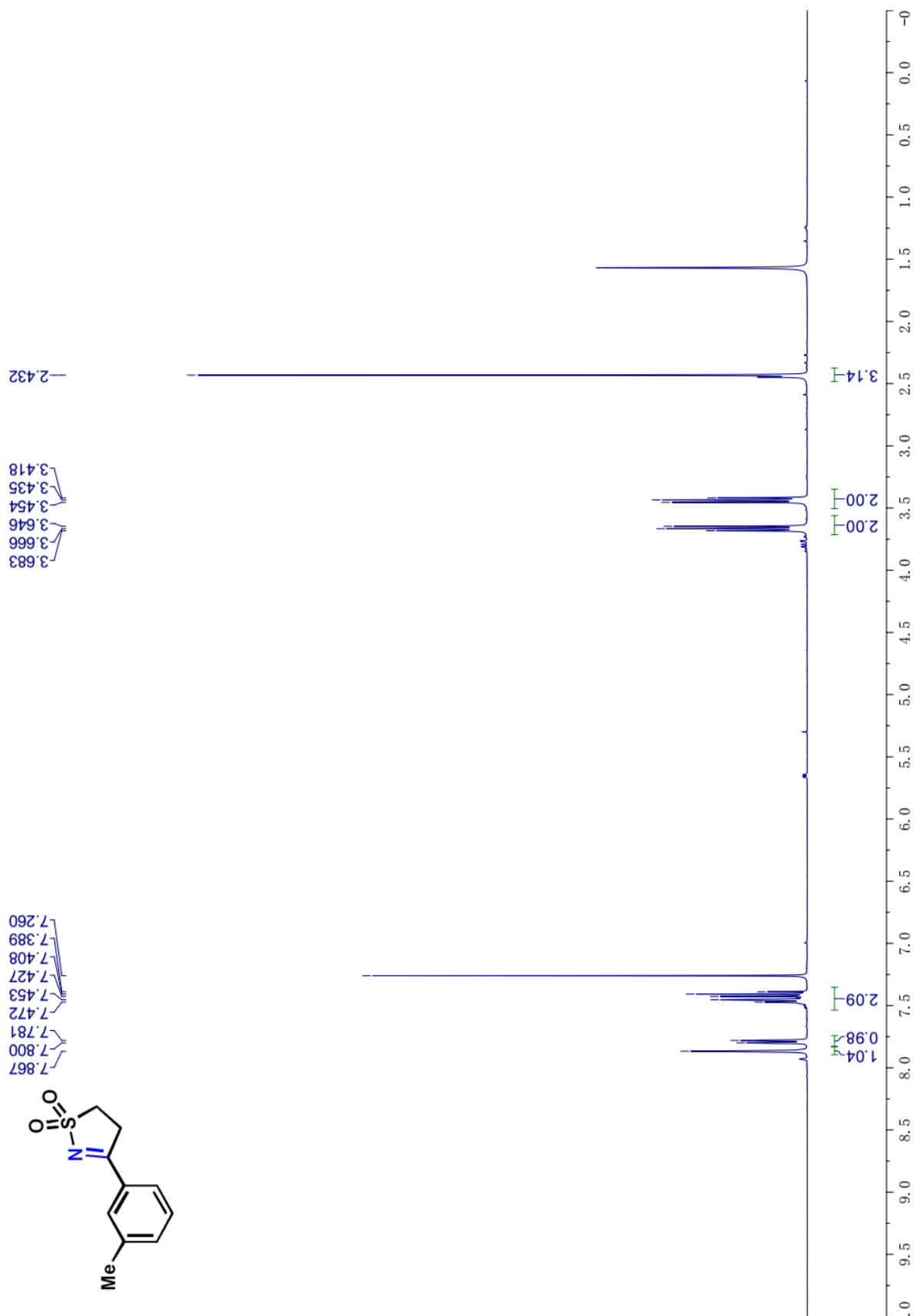
¹H NMR (400 MHz, CDCl₃) of **2g**

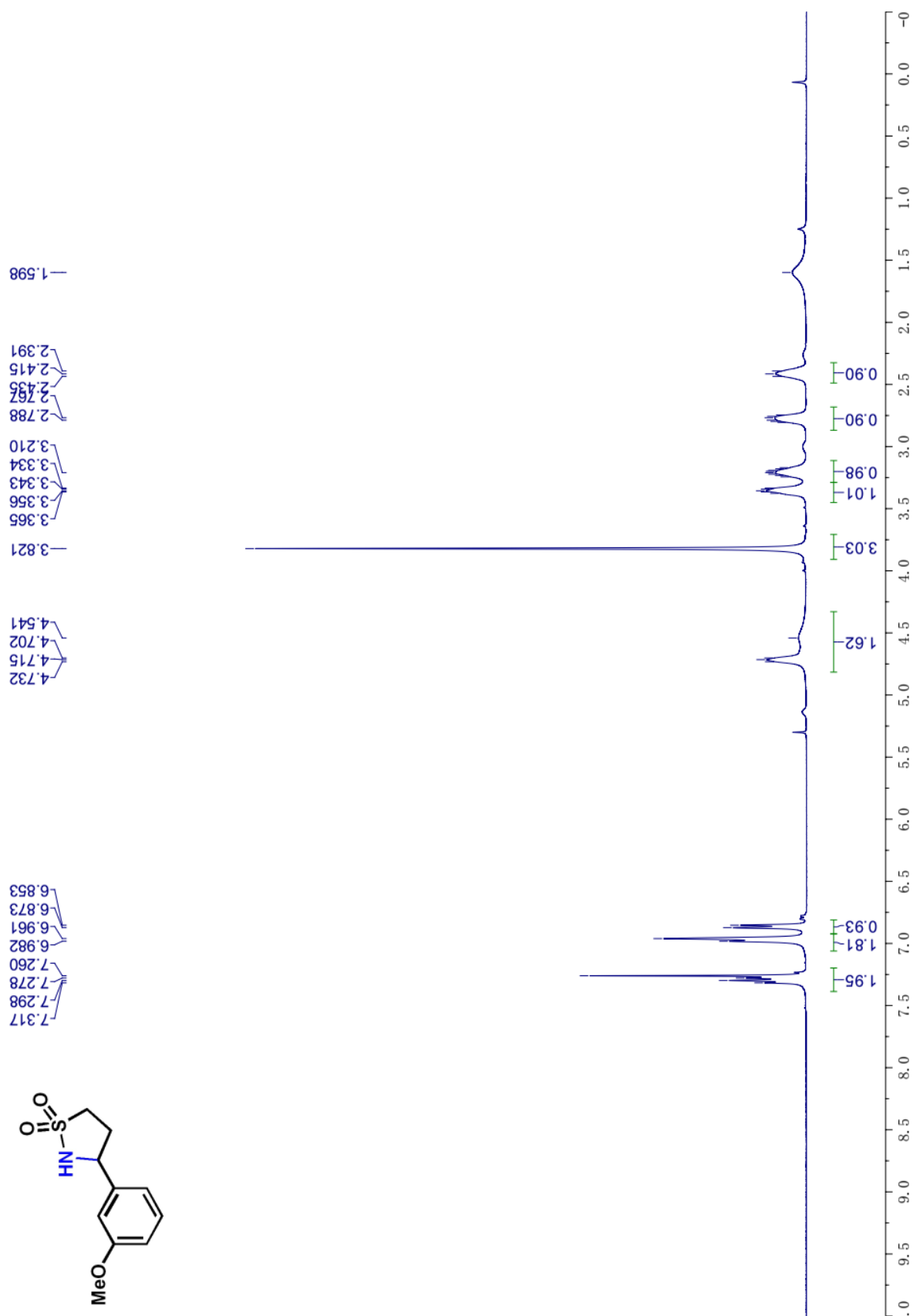
^{13}C NMR (100 MHz, CDCl_3) of **2g**

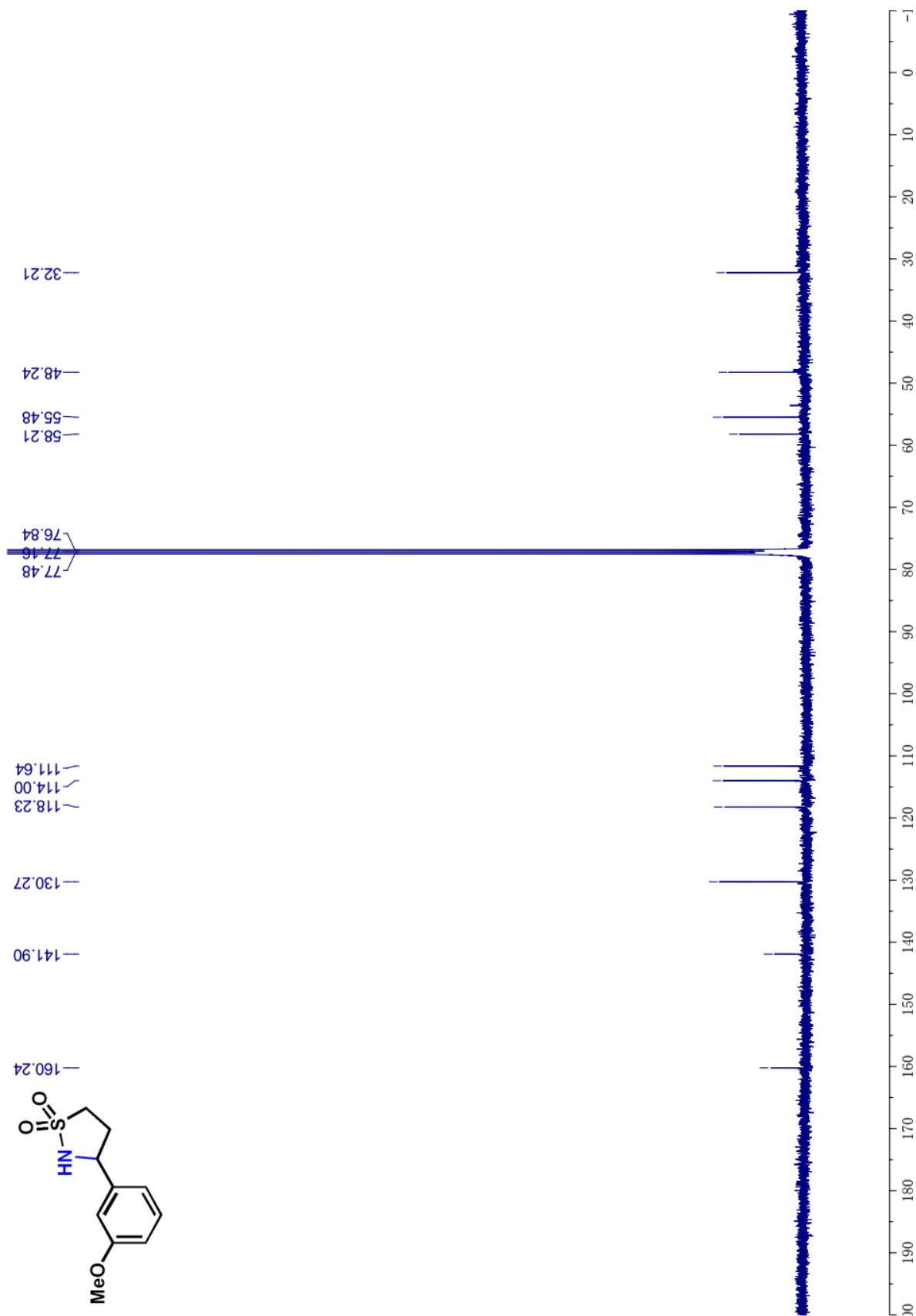
¹H NMR (400 MHz, CD₃CN) of **3g**

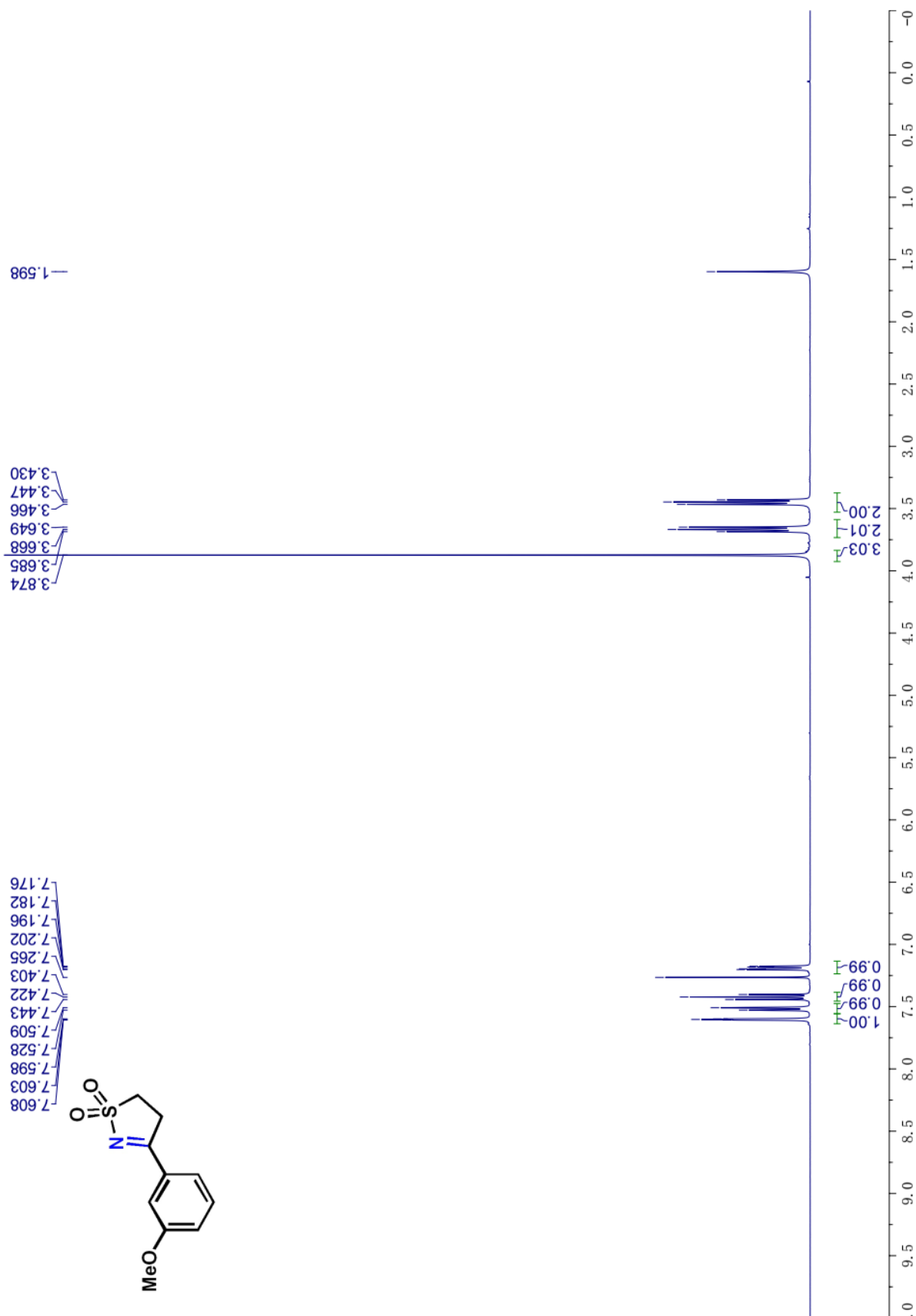
^{13}C NMR (100 MHz, CD_3CN) of **3g**

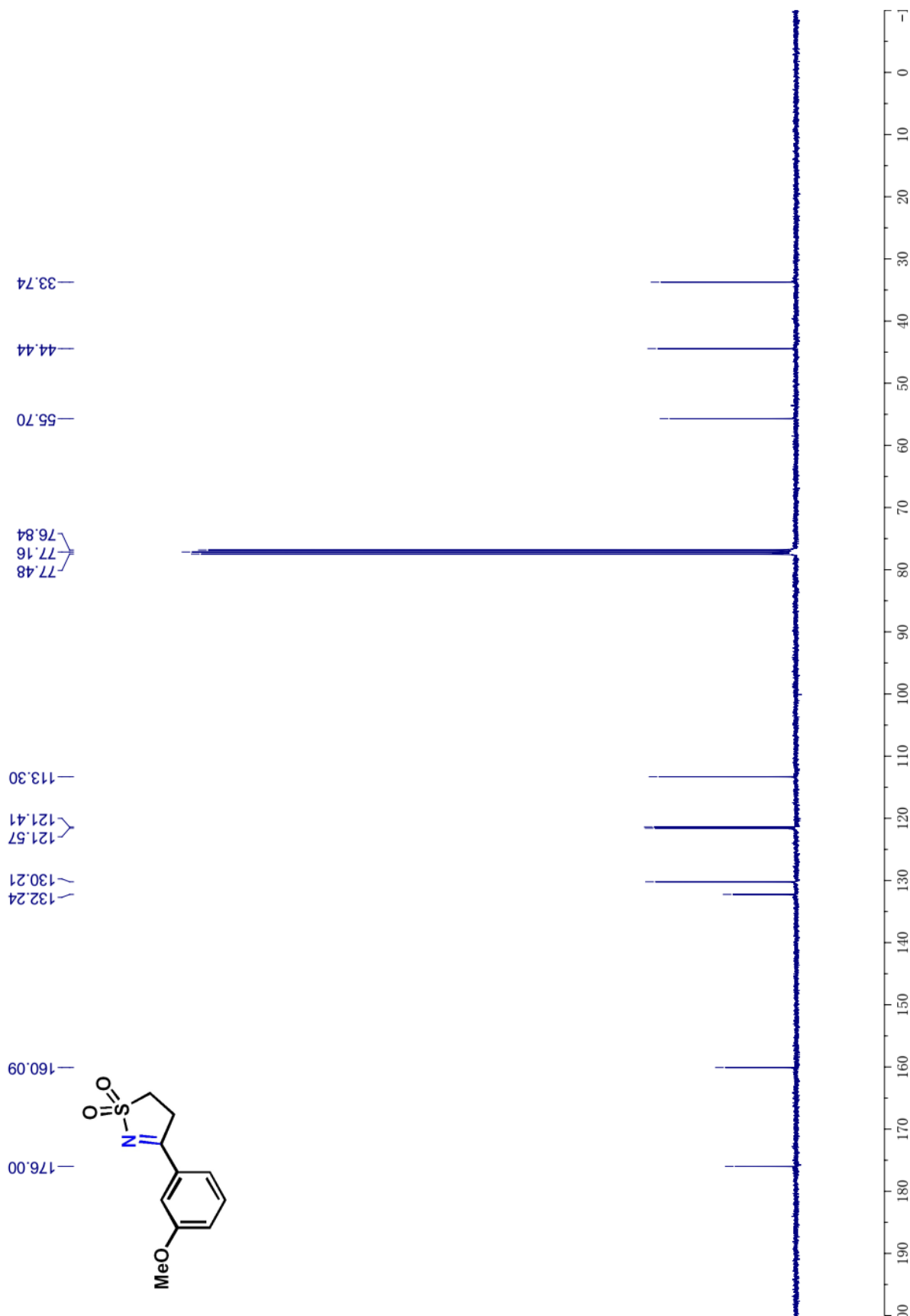
¹H NMR (400 MHz, CDCl₃) of **2h**

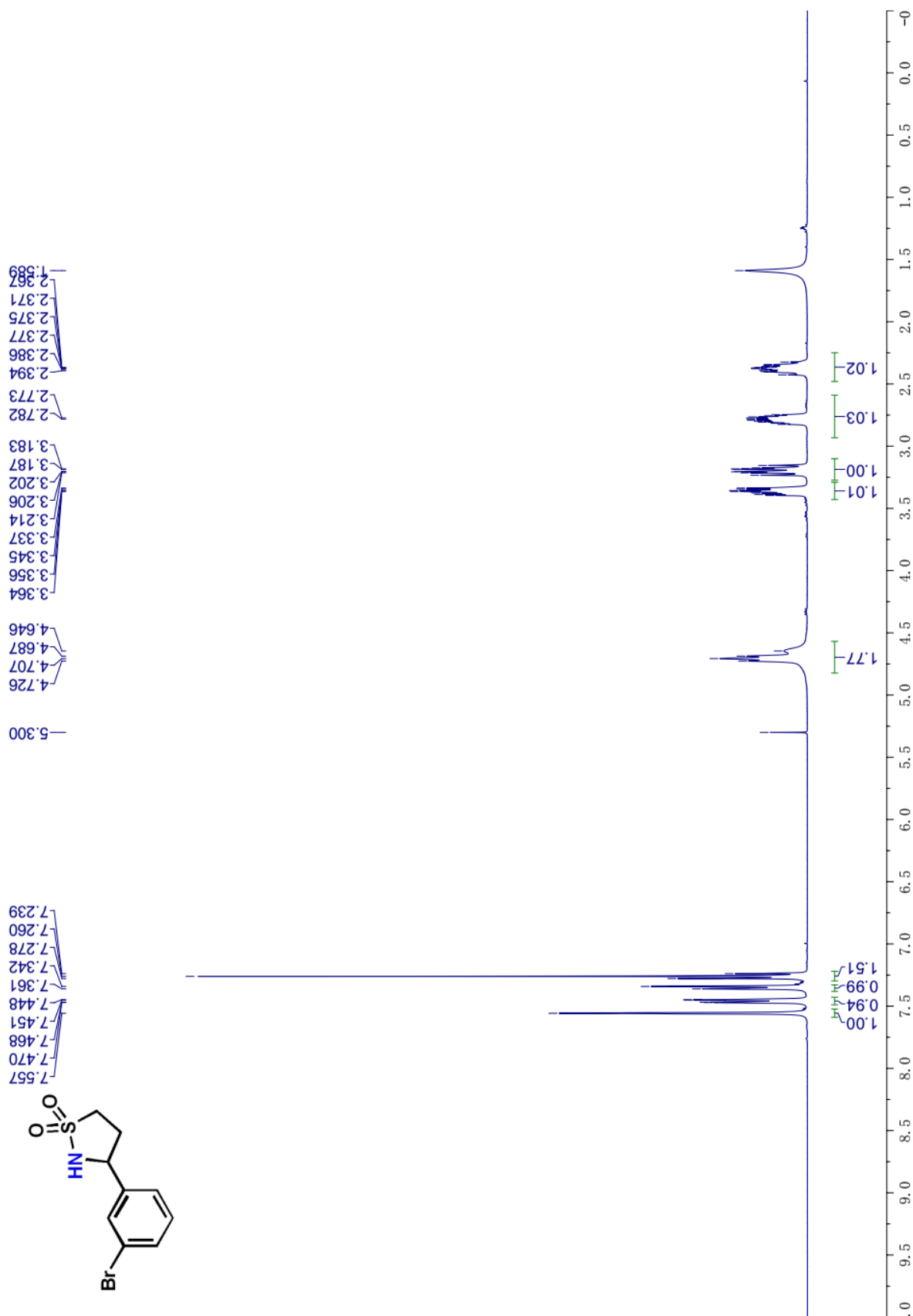
¹H NMR (400 MHz, CDCl₃) of **3h**

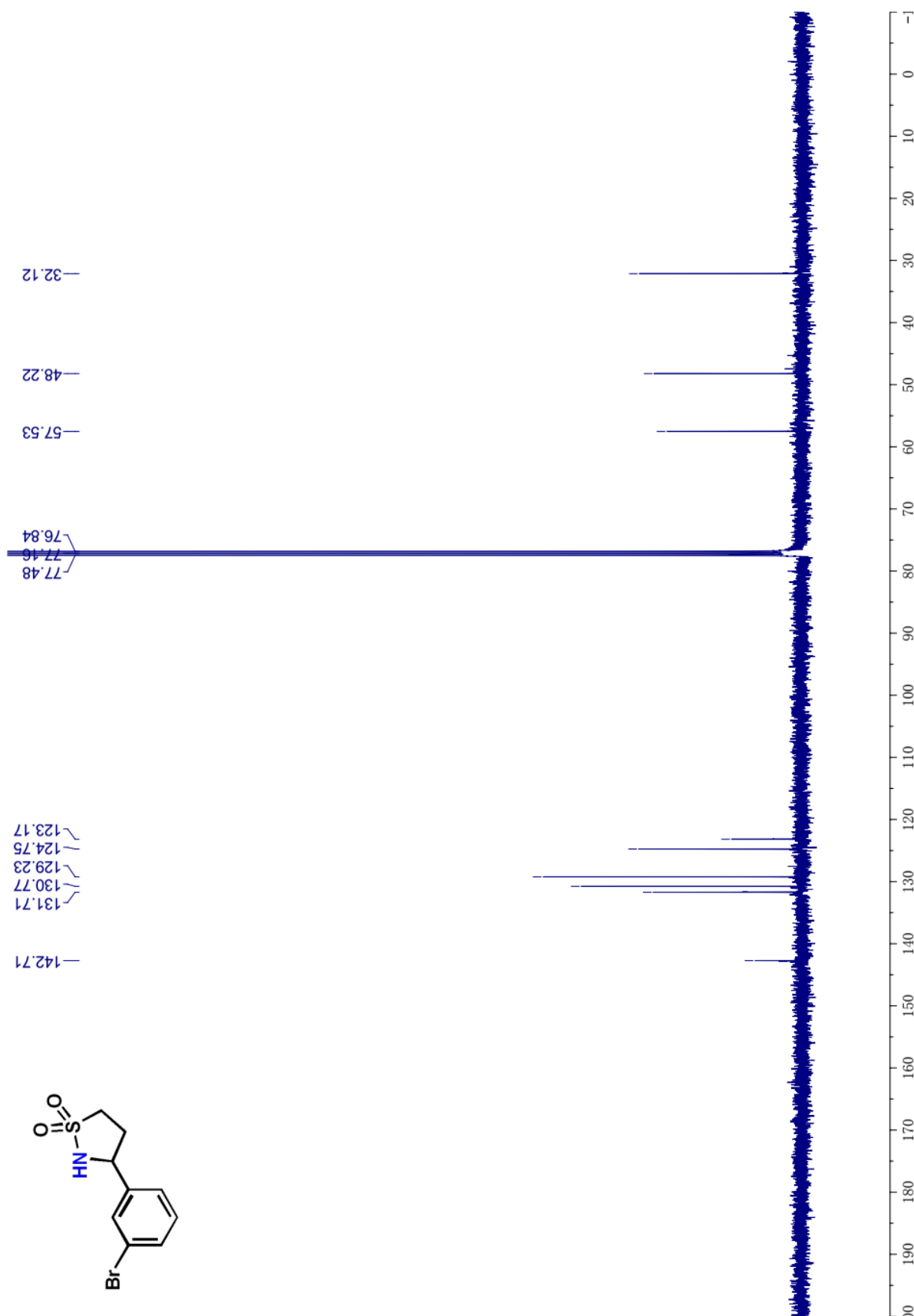
¹H NMR (400 MHz, CDCl₃) of **2i**

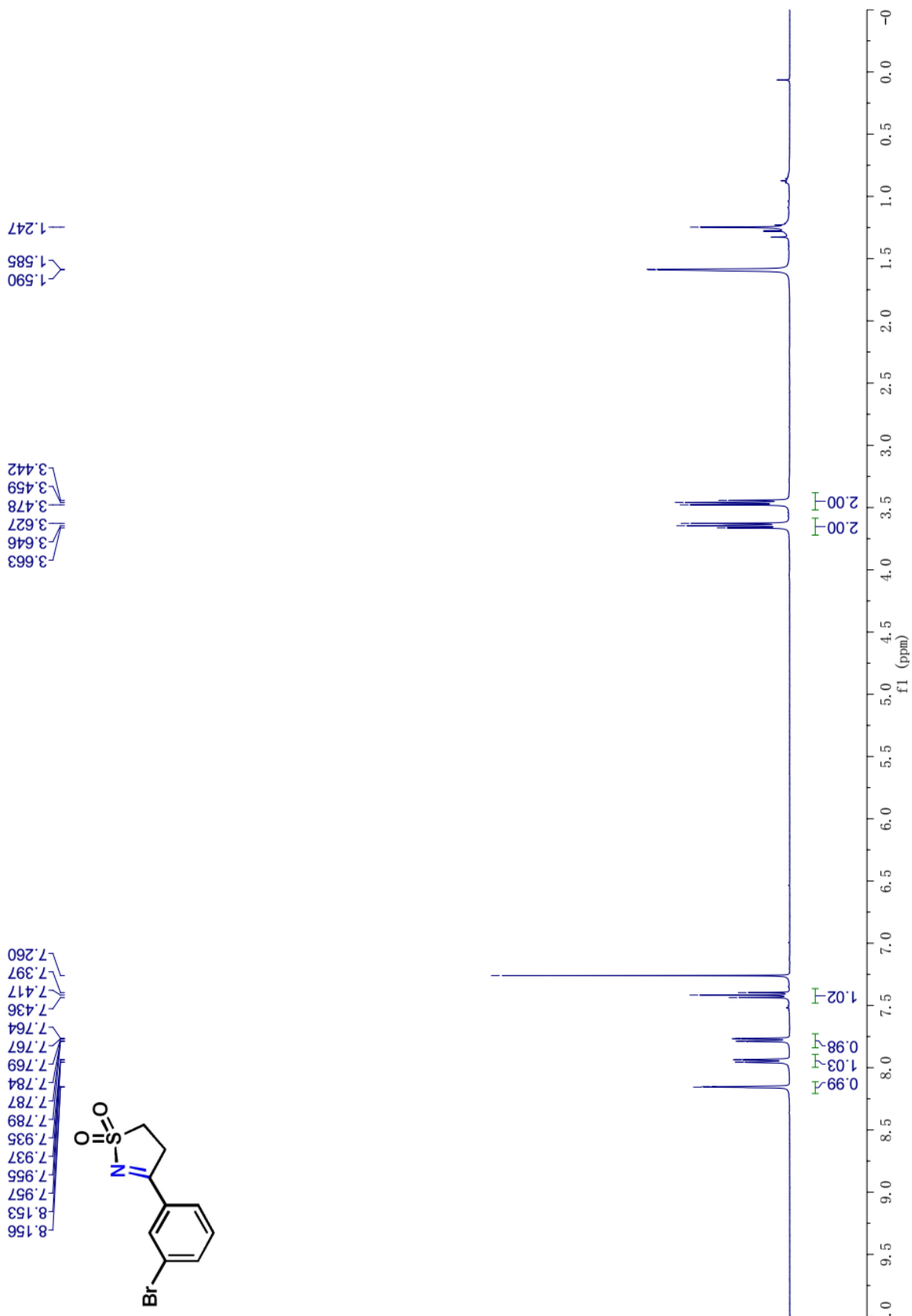
^{13}C NMR (100 MHz, CDCl_3) of **2i**

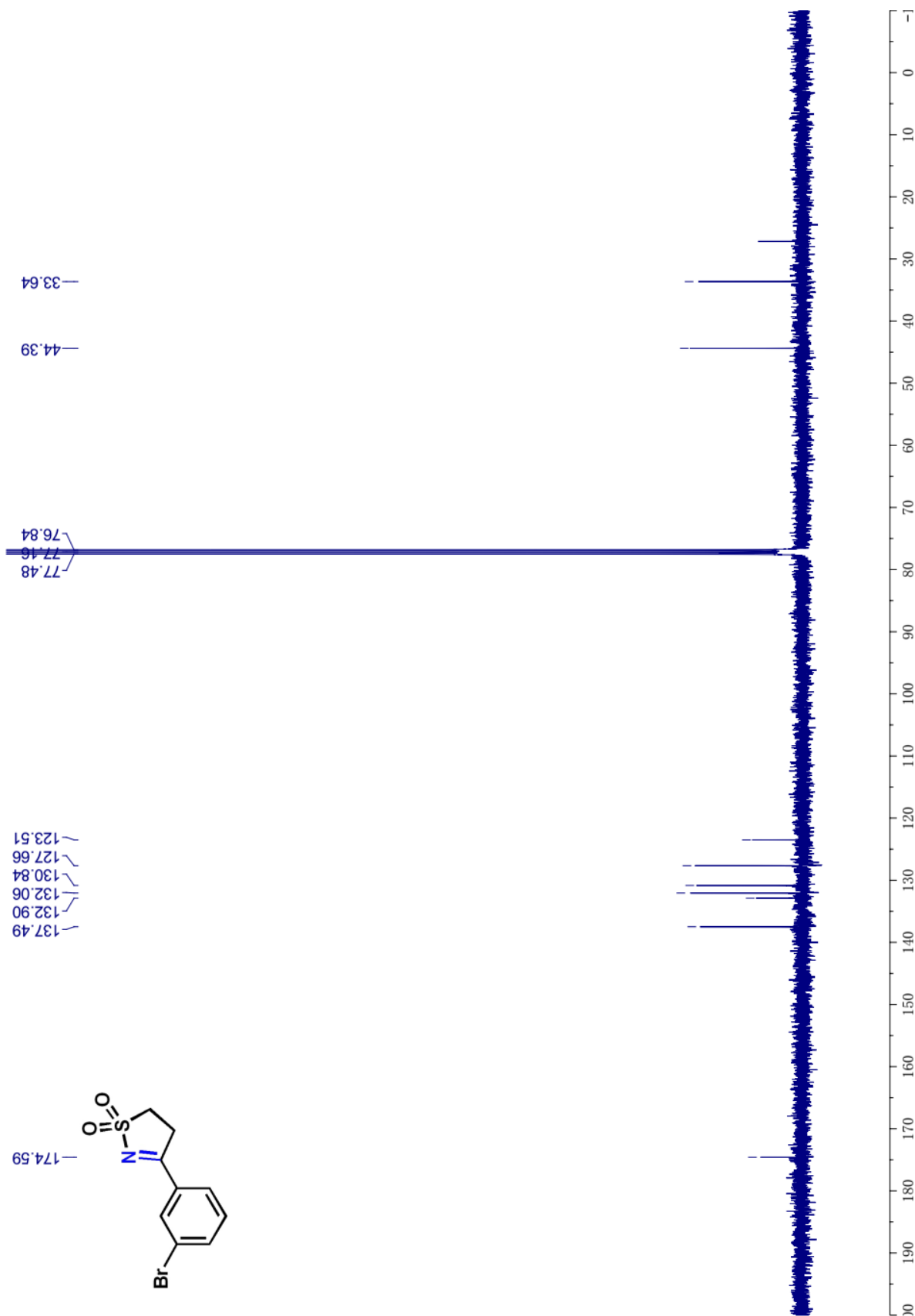
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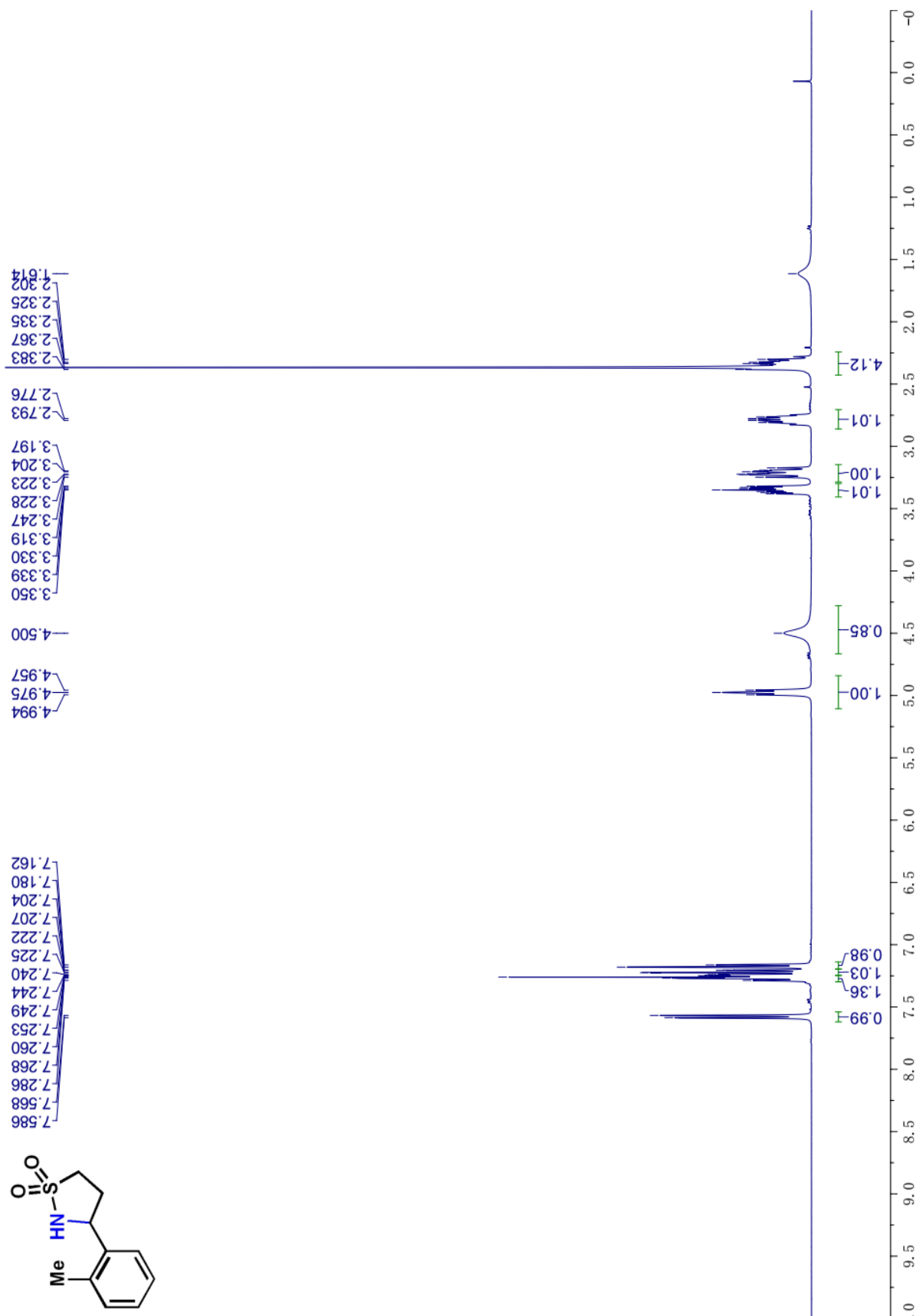
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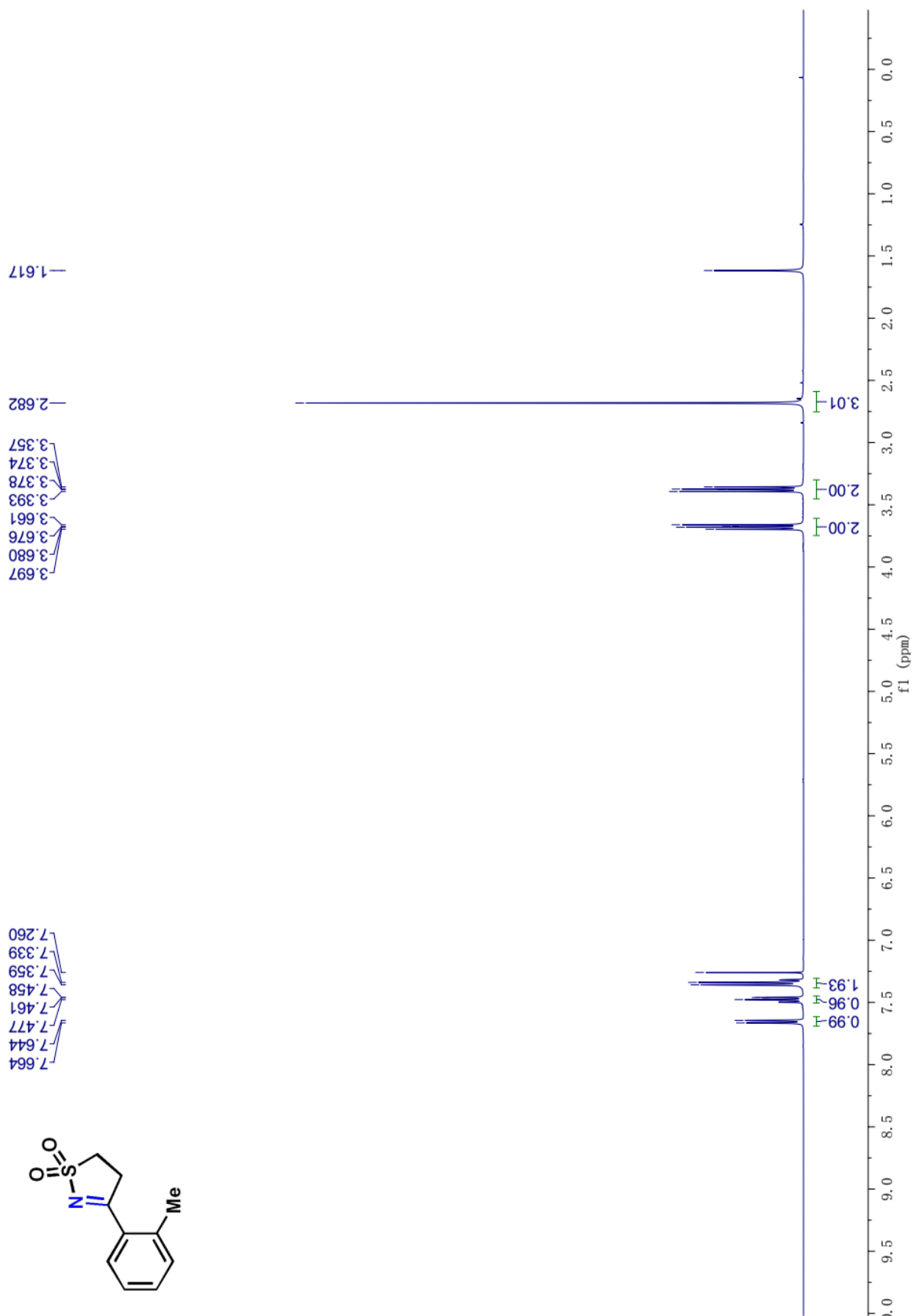


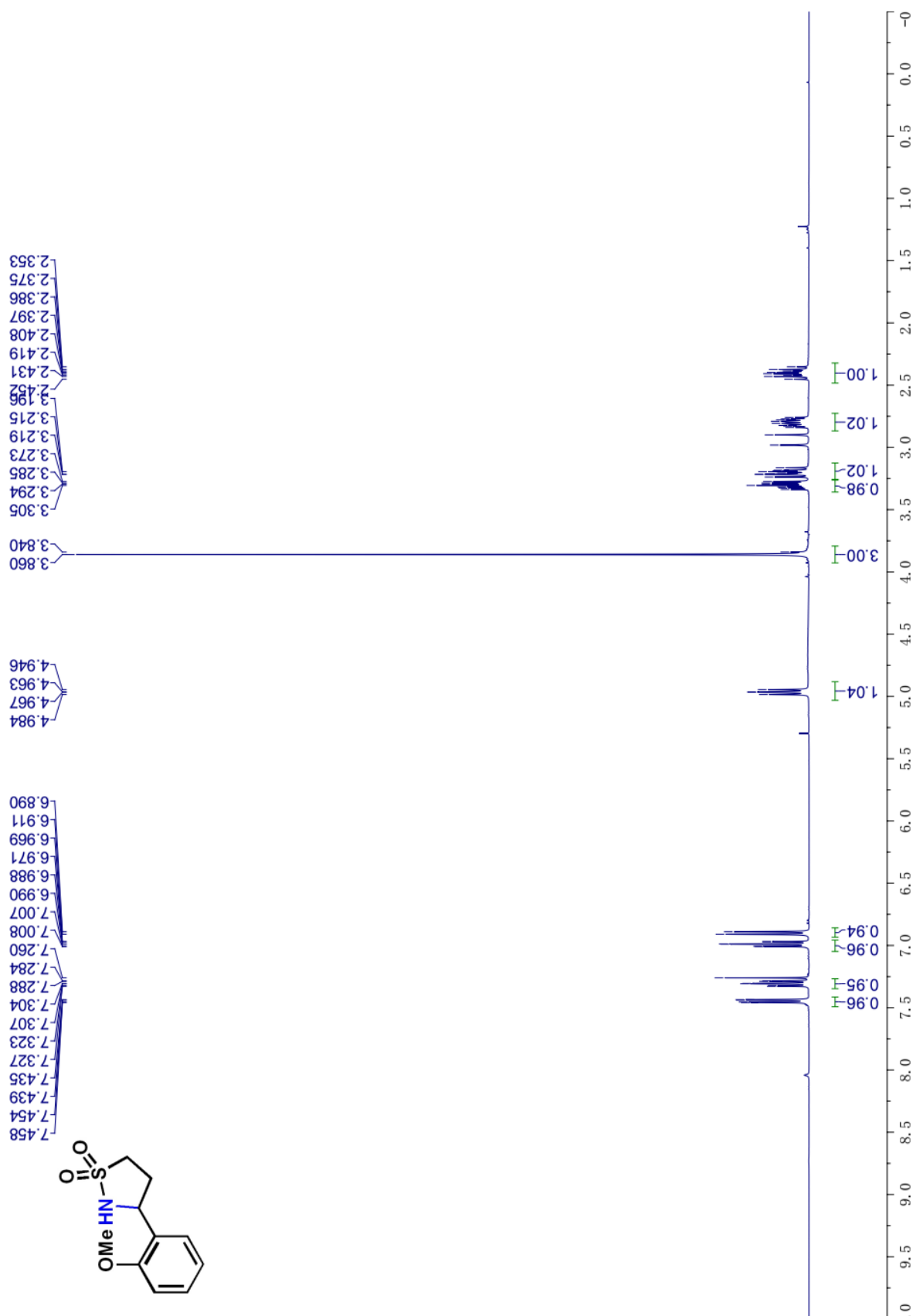
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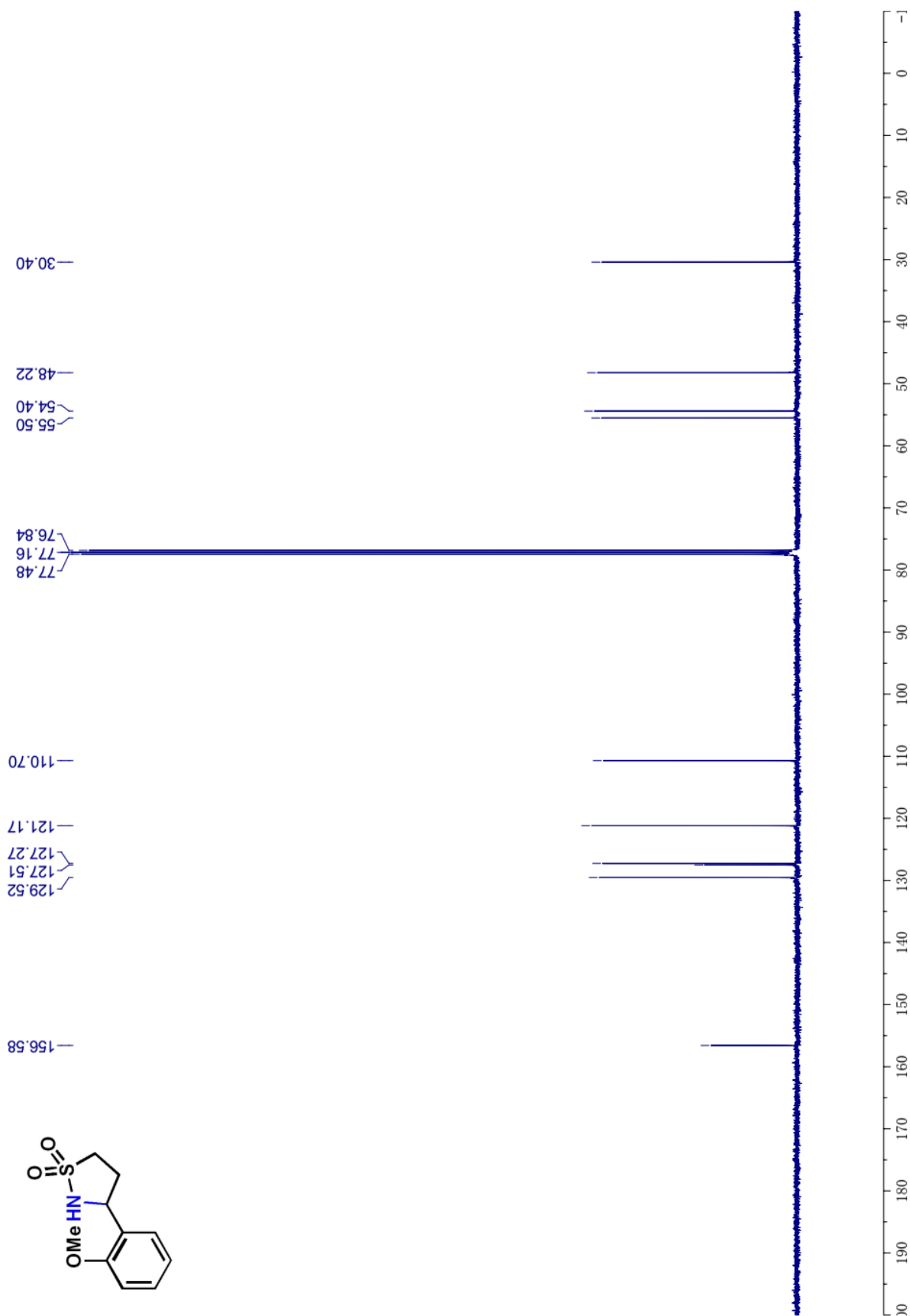
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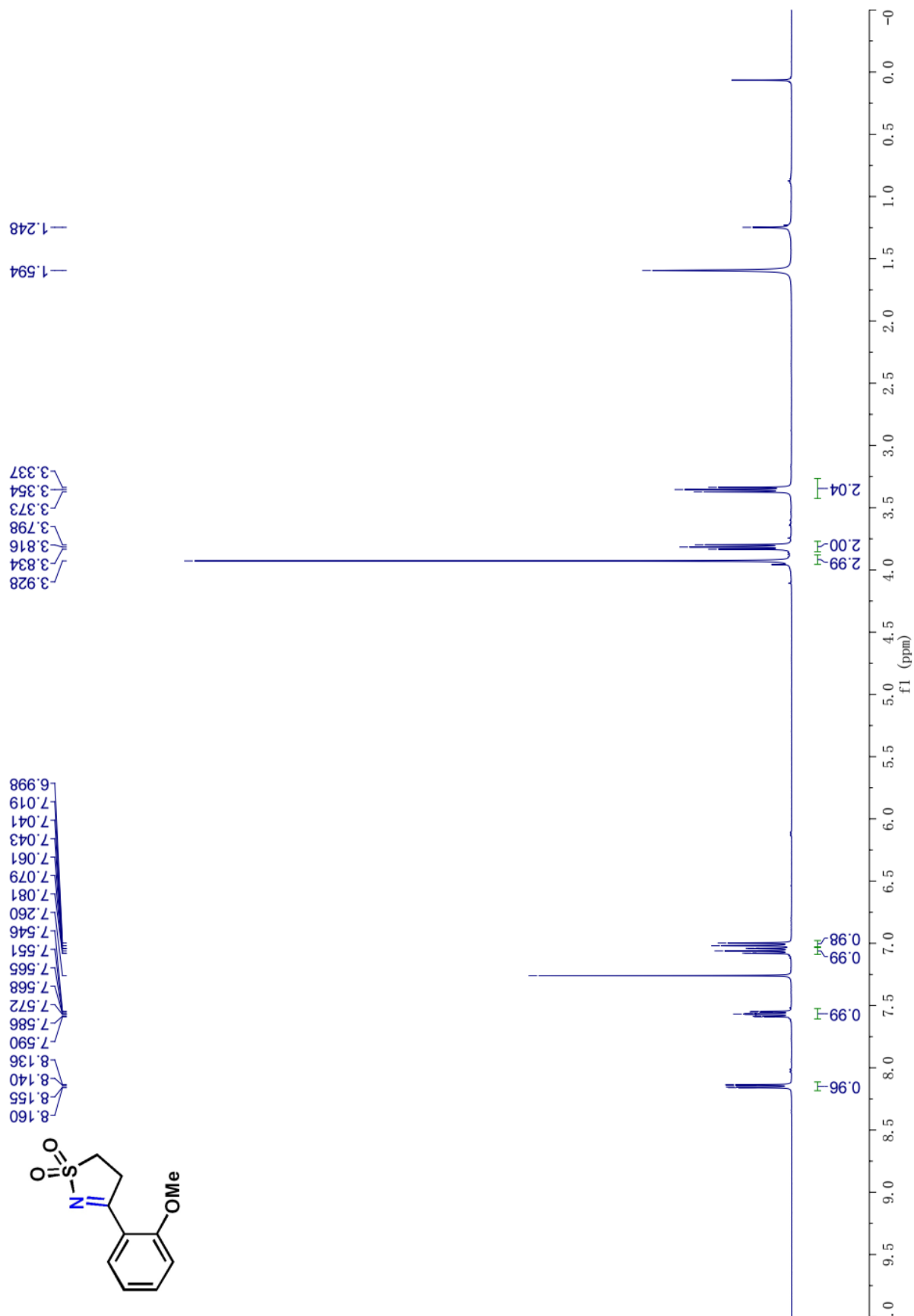
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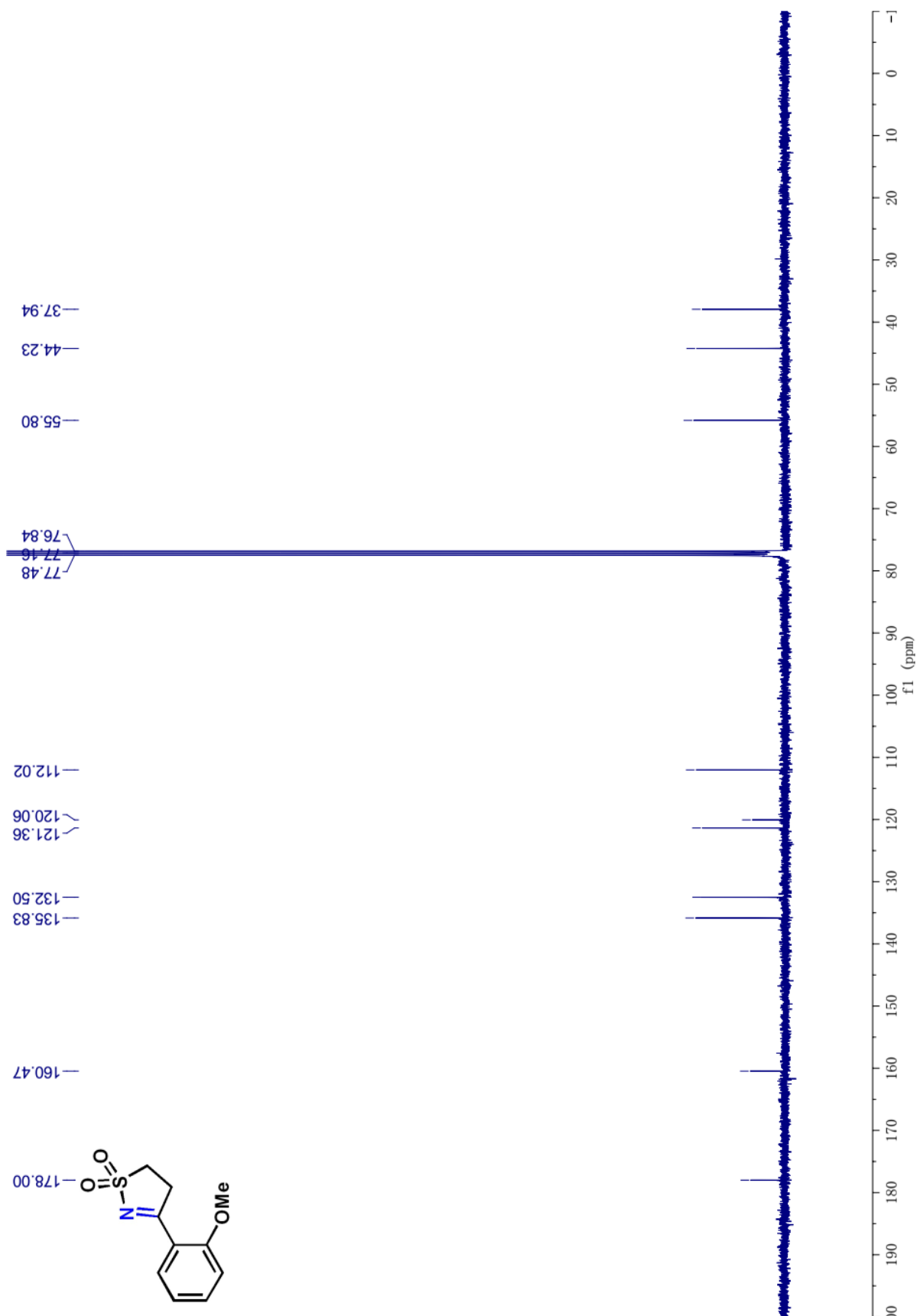
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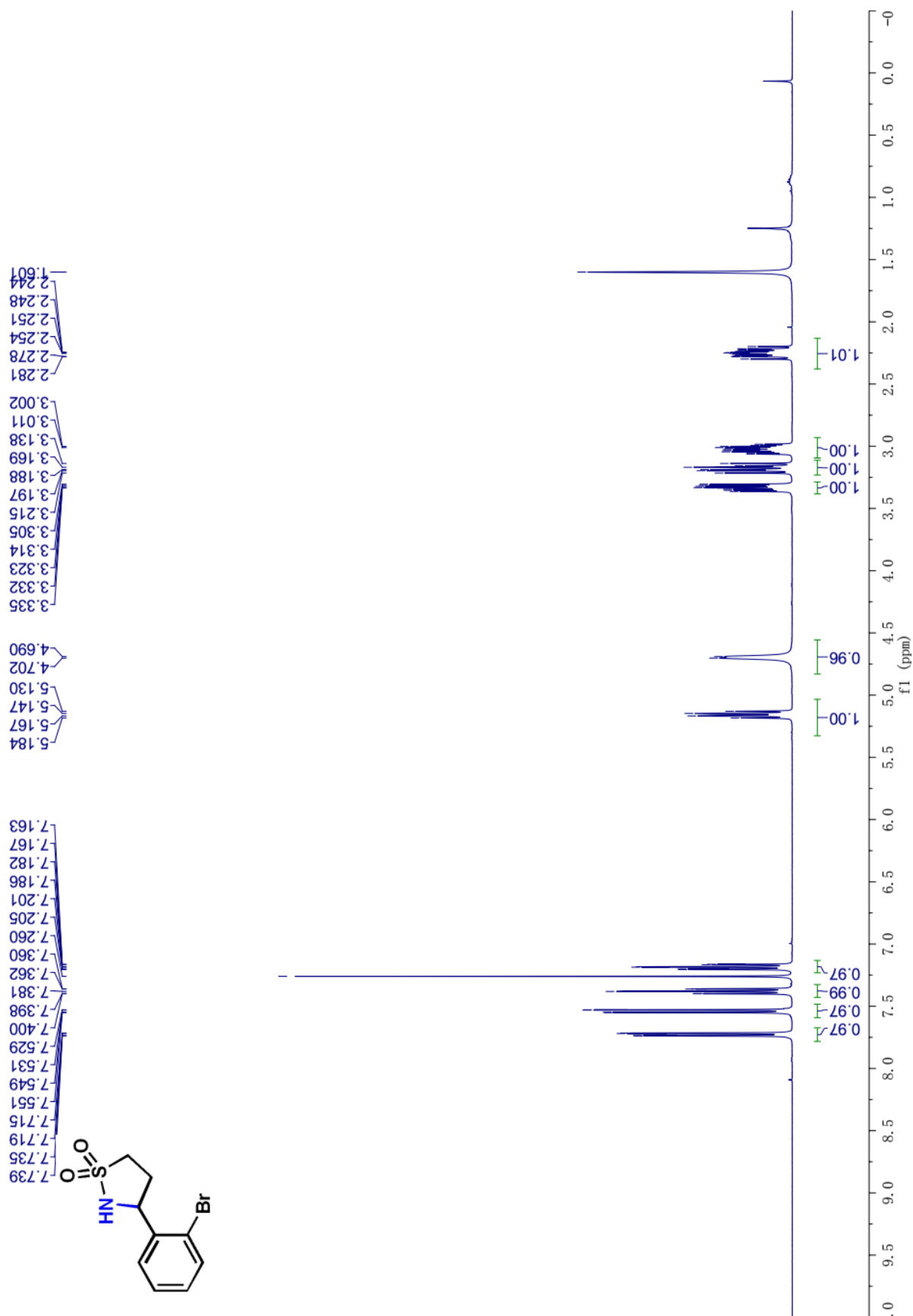
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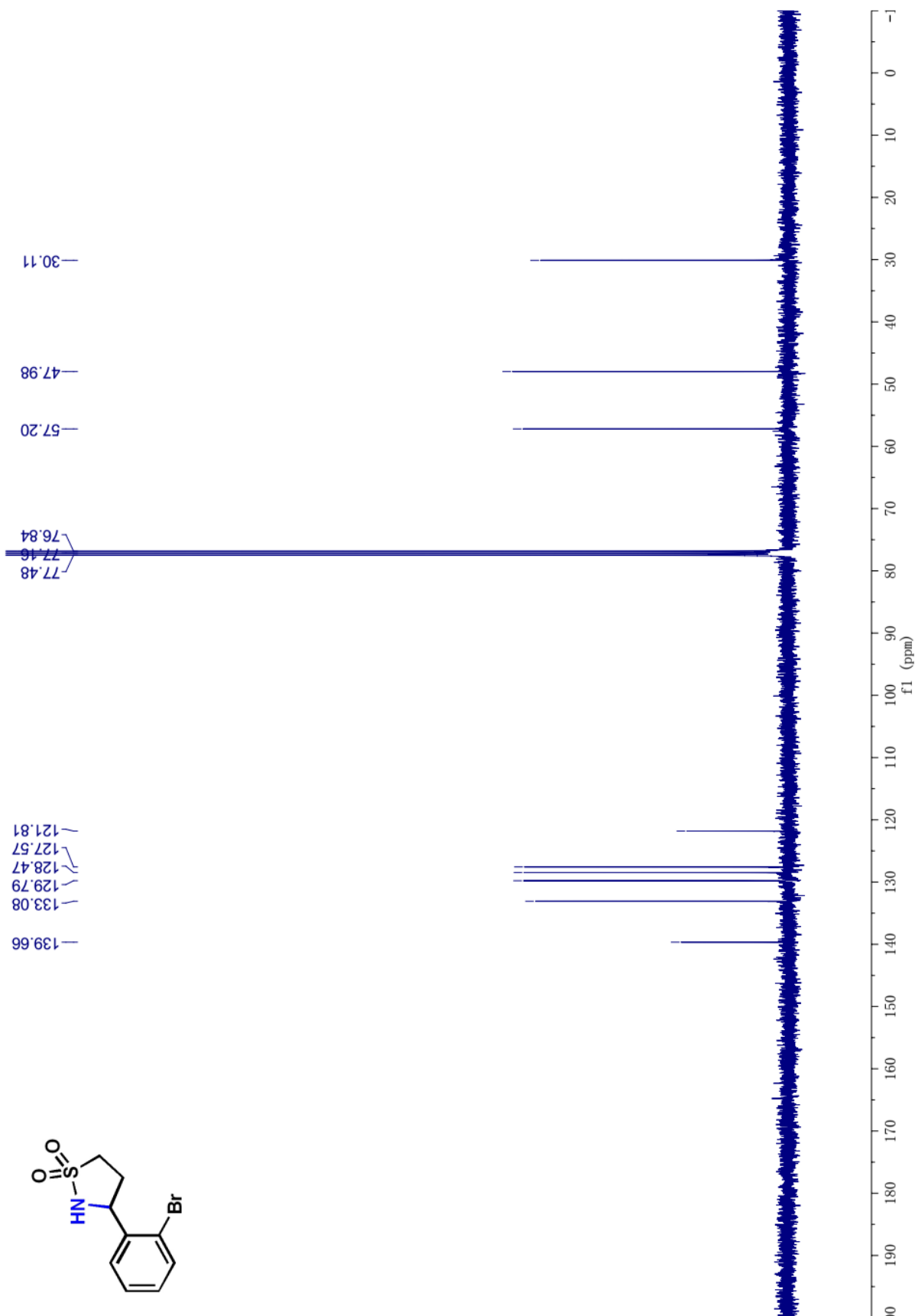
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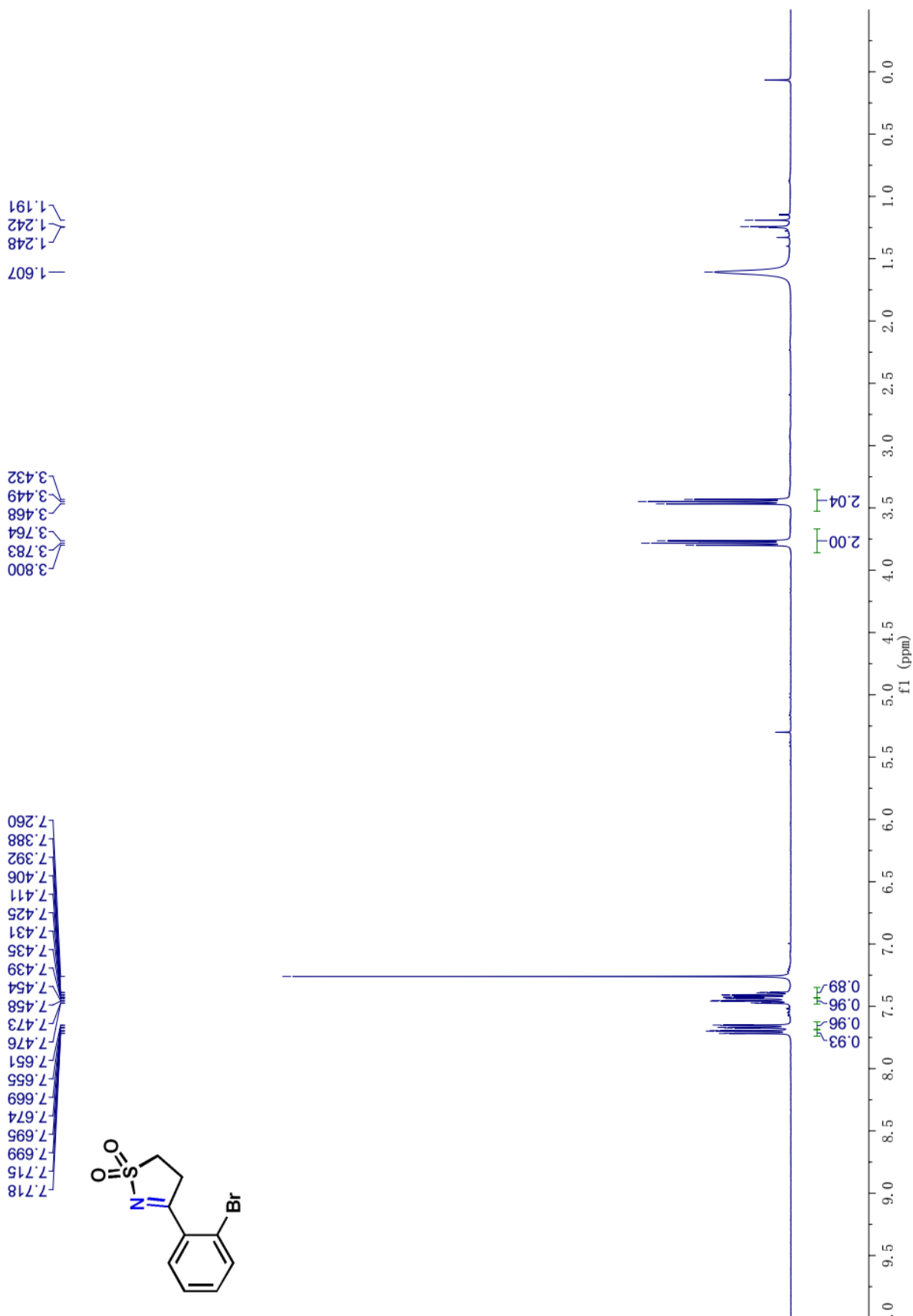
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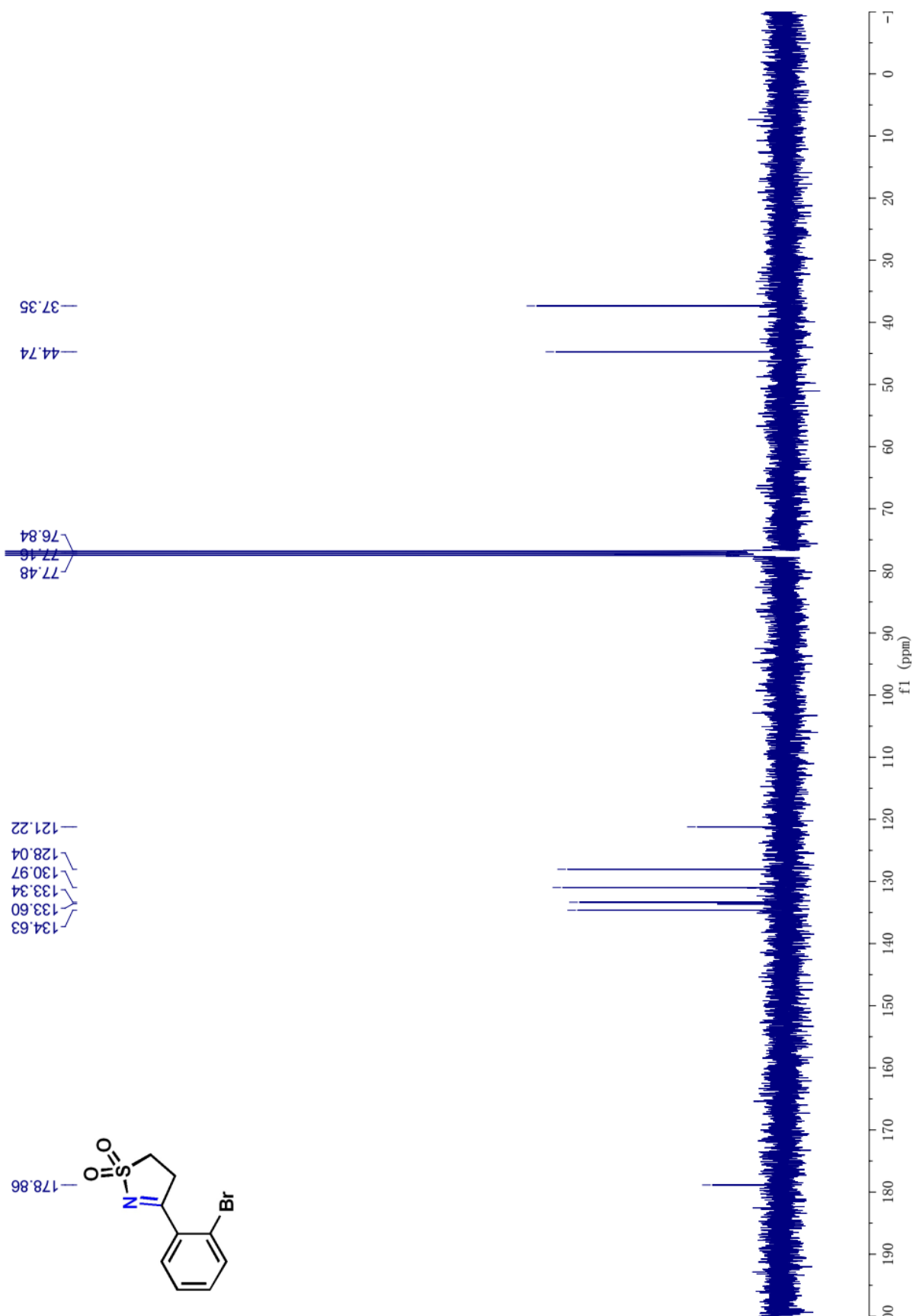
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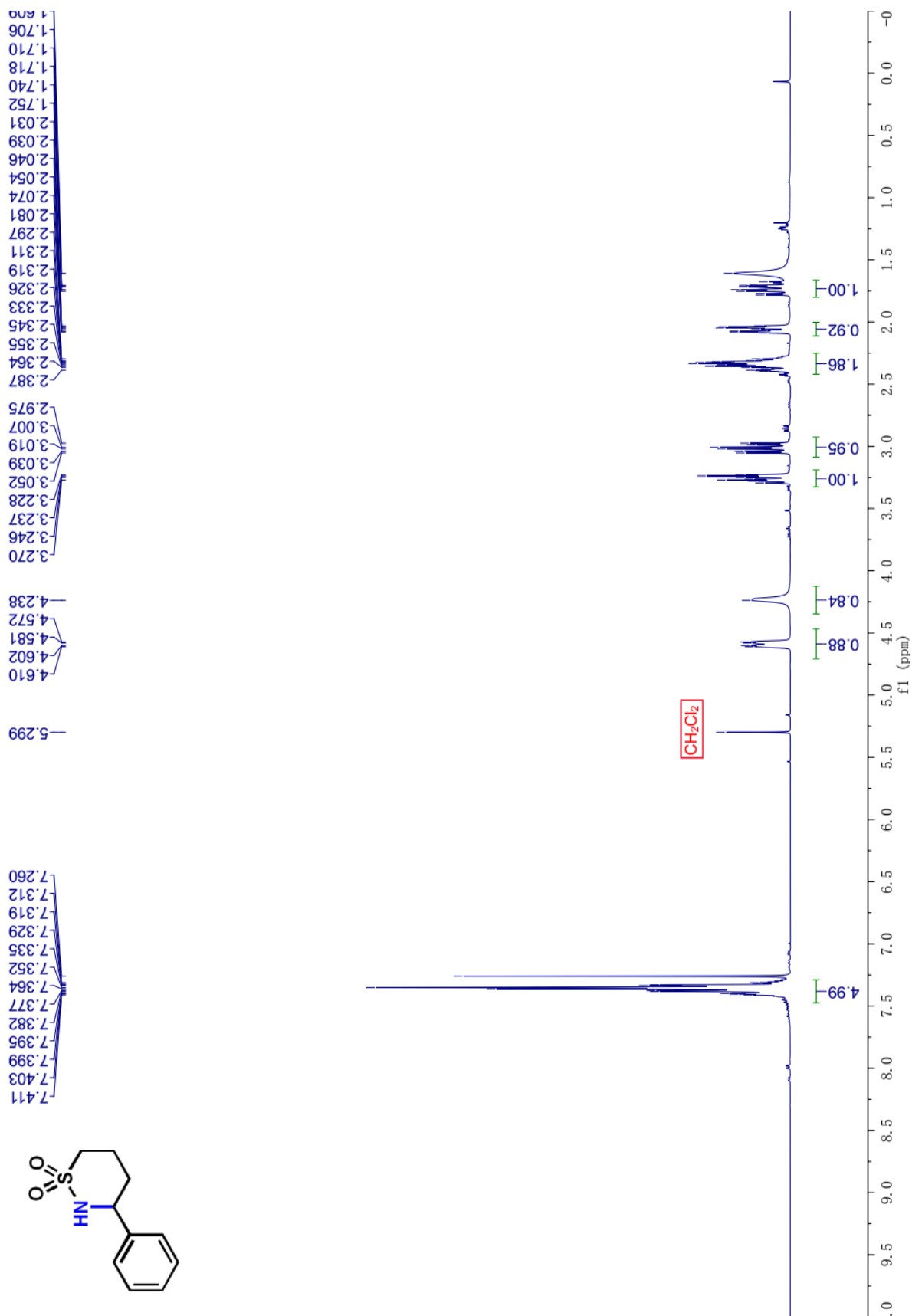
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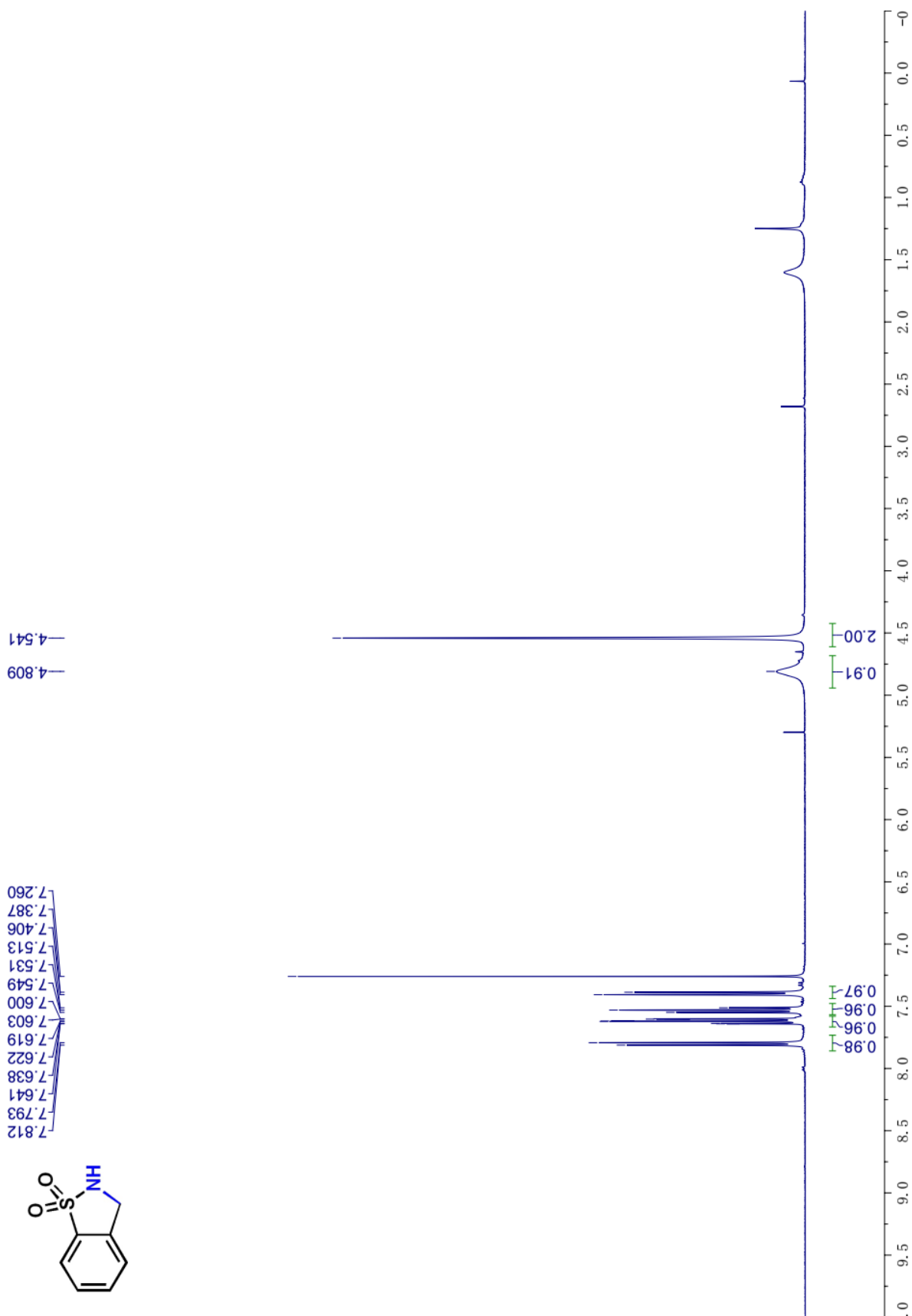
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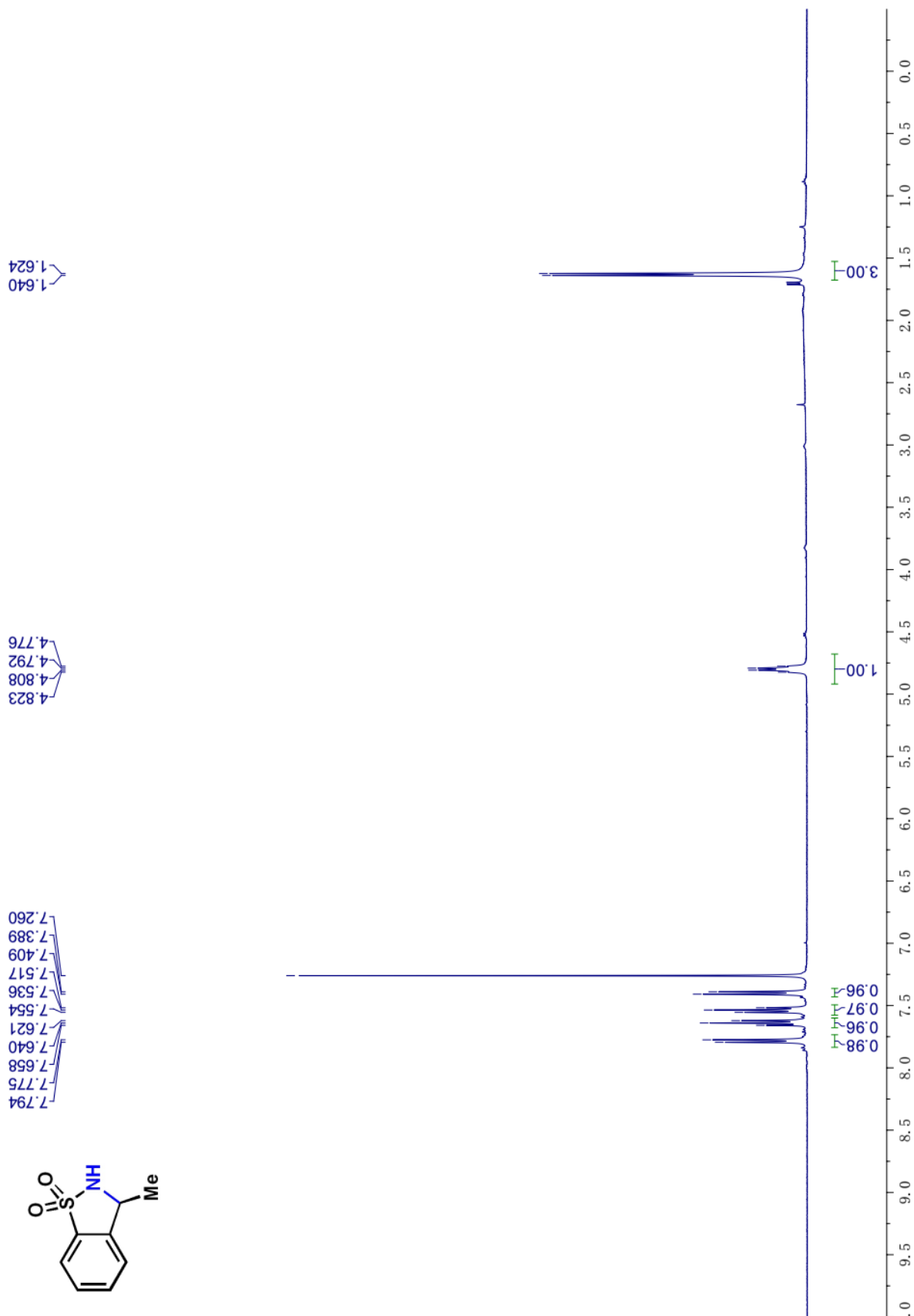
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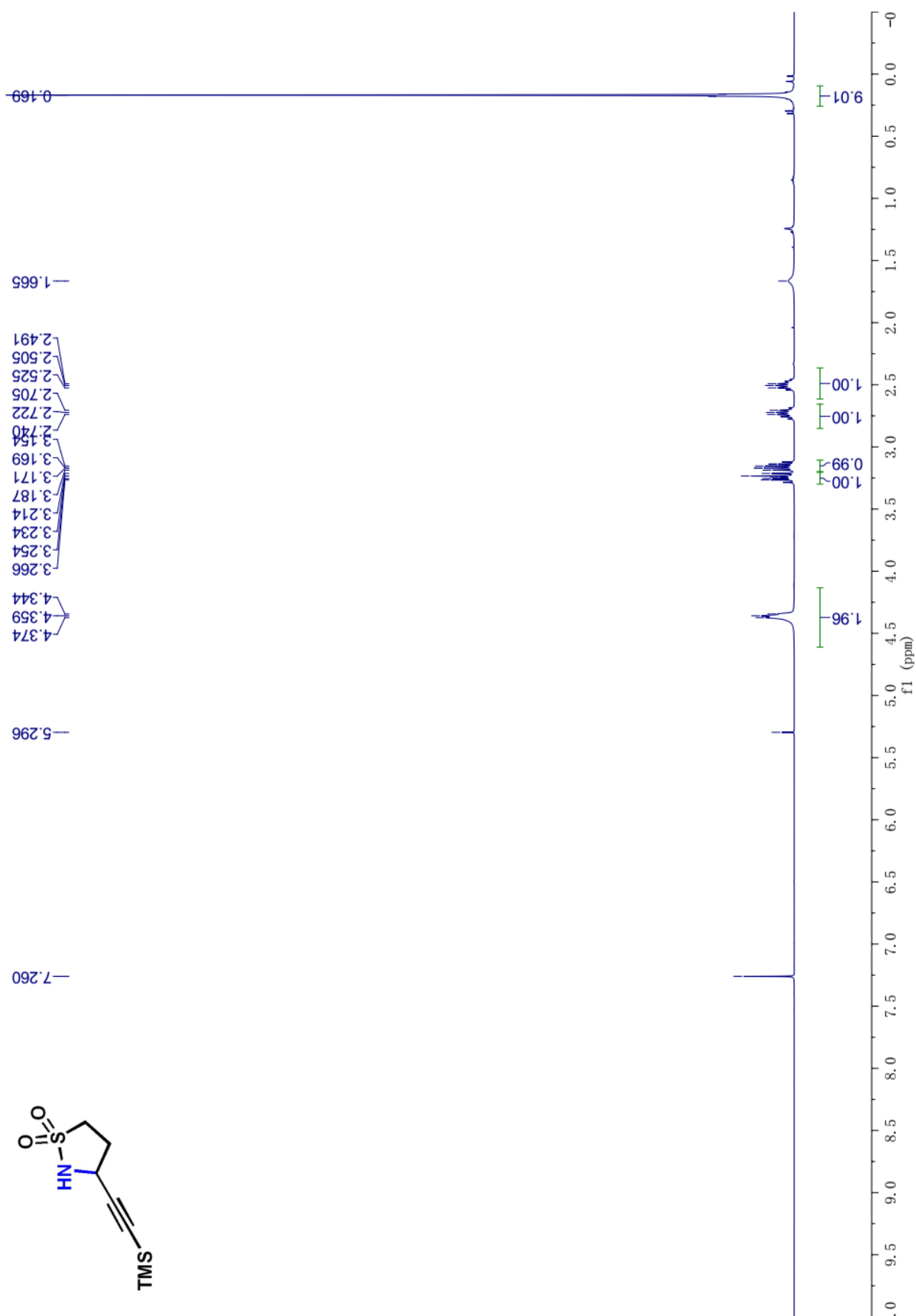
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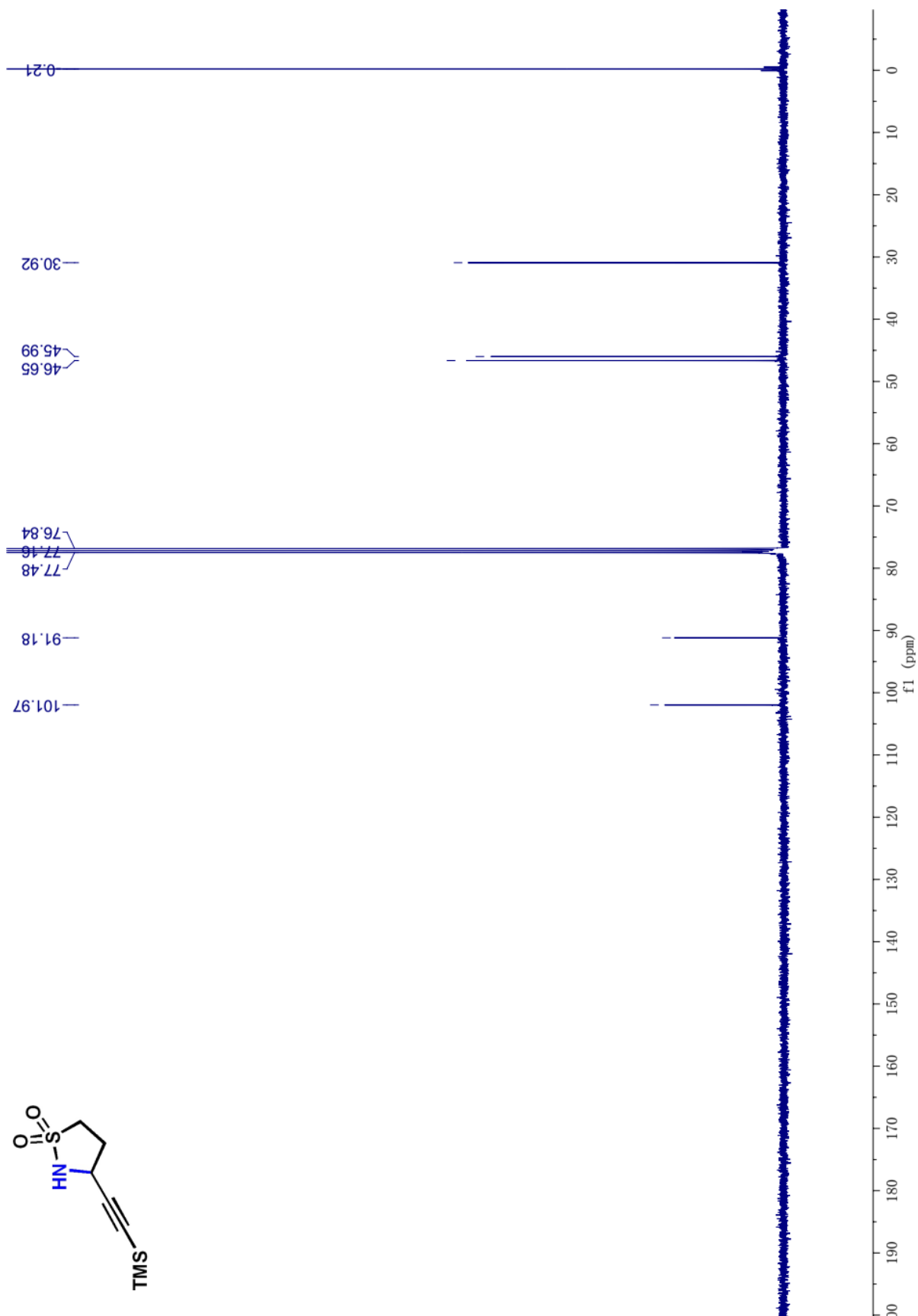
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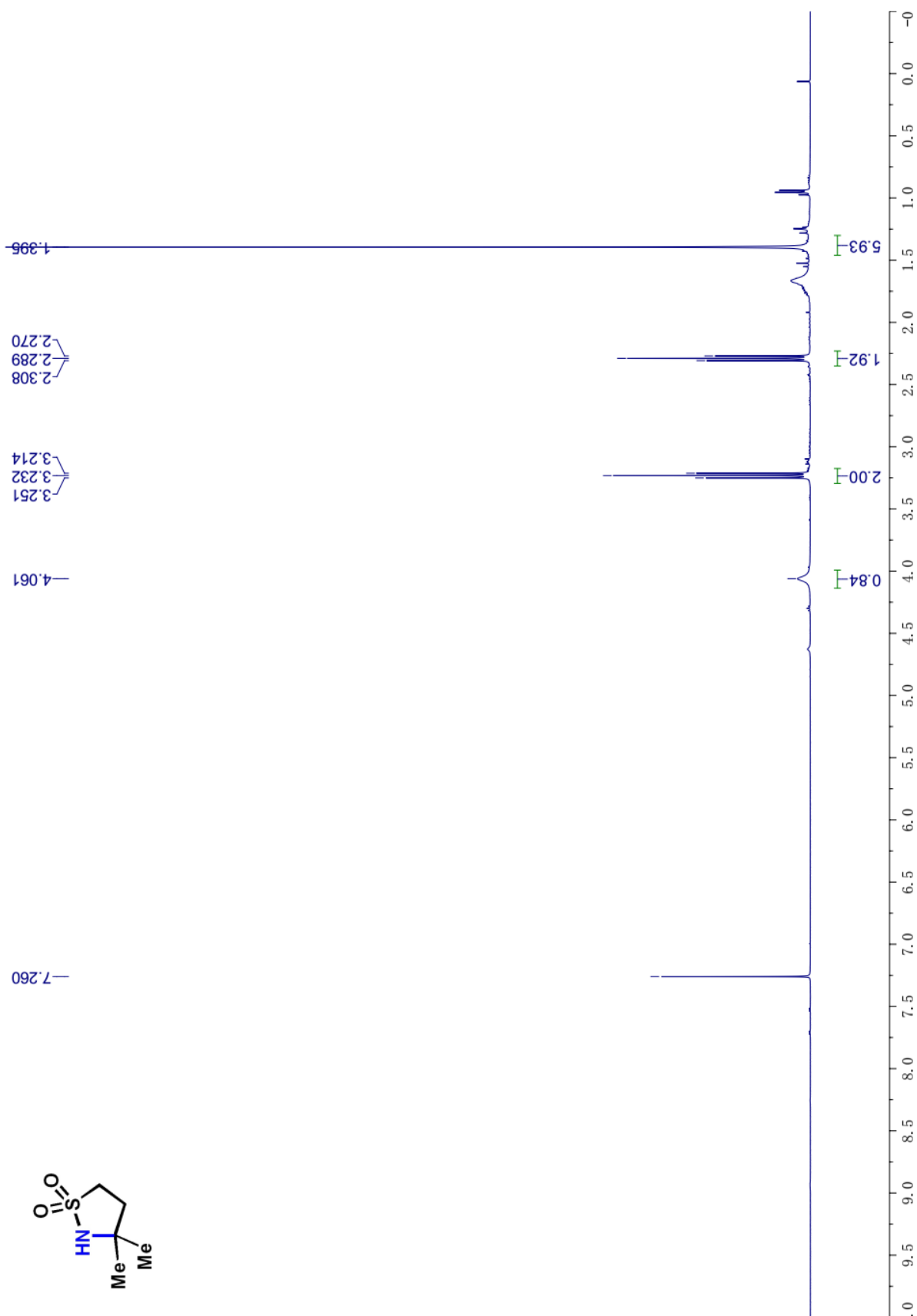
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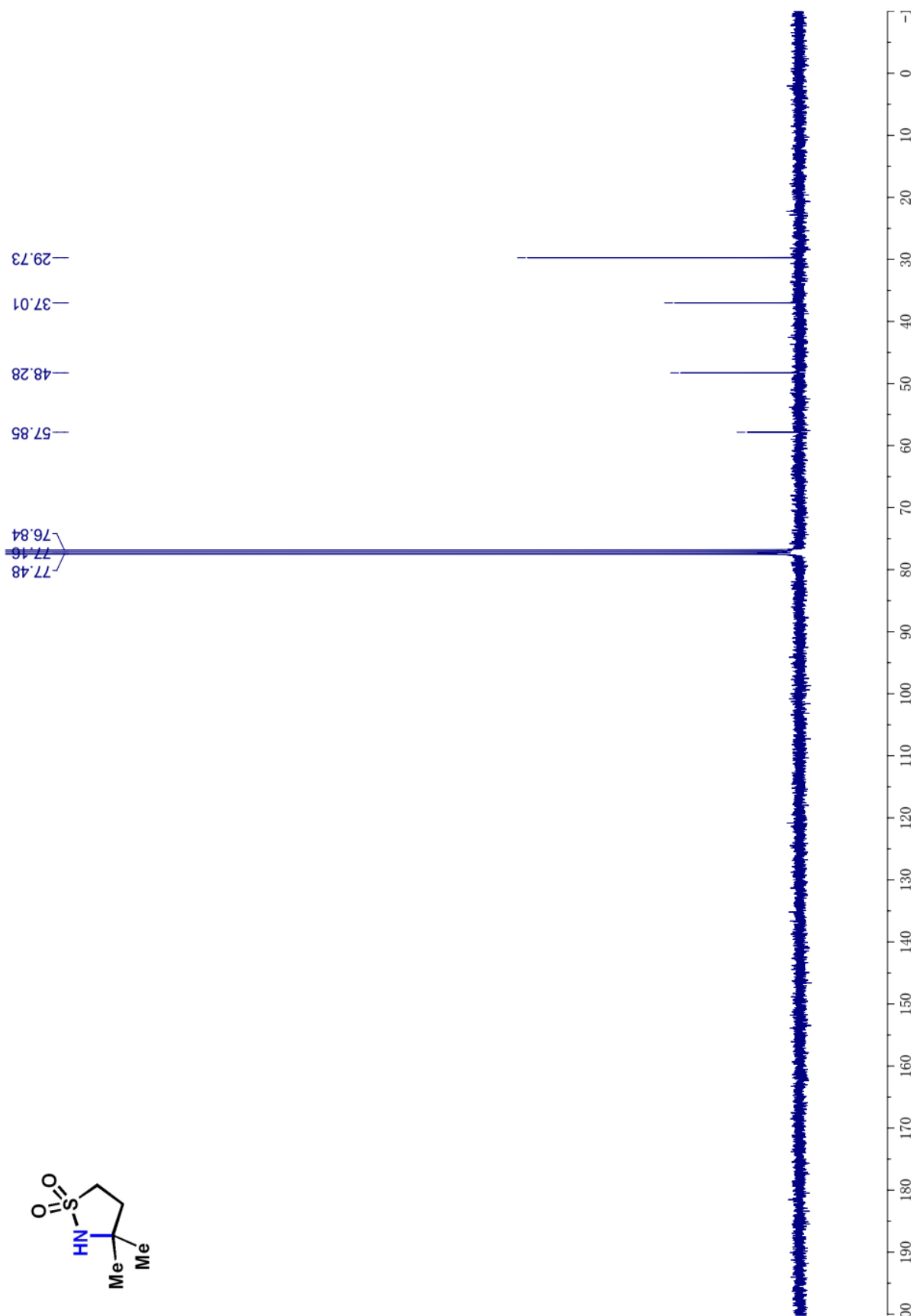
¹H NMR (400 MHz, CDCl₃) of **2o**

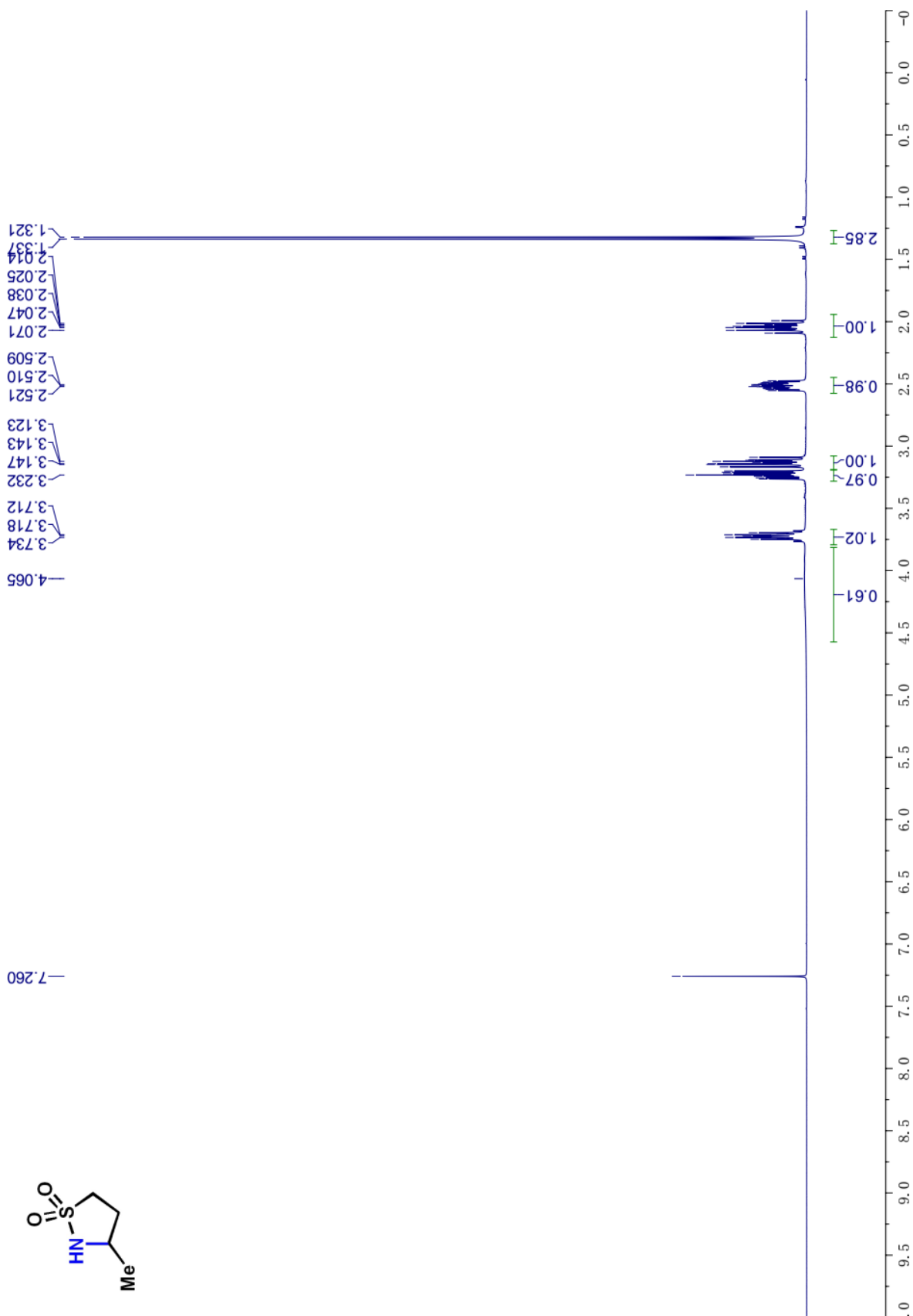
¹H NMR (400 MHz, CDCl₃) of **2p**

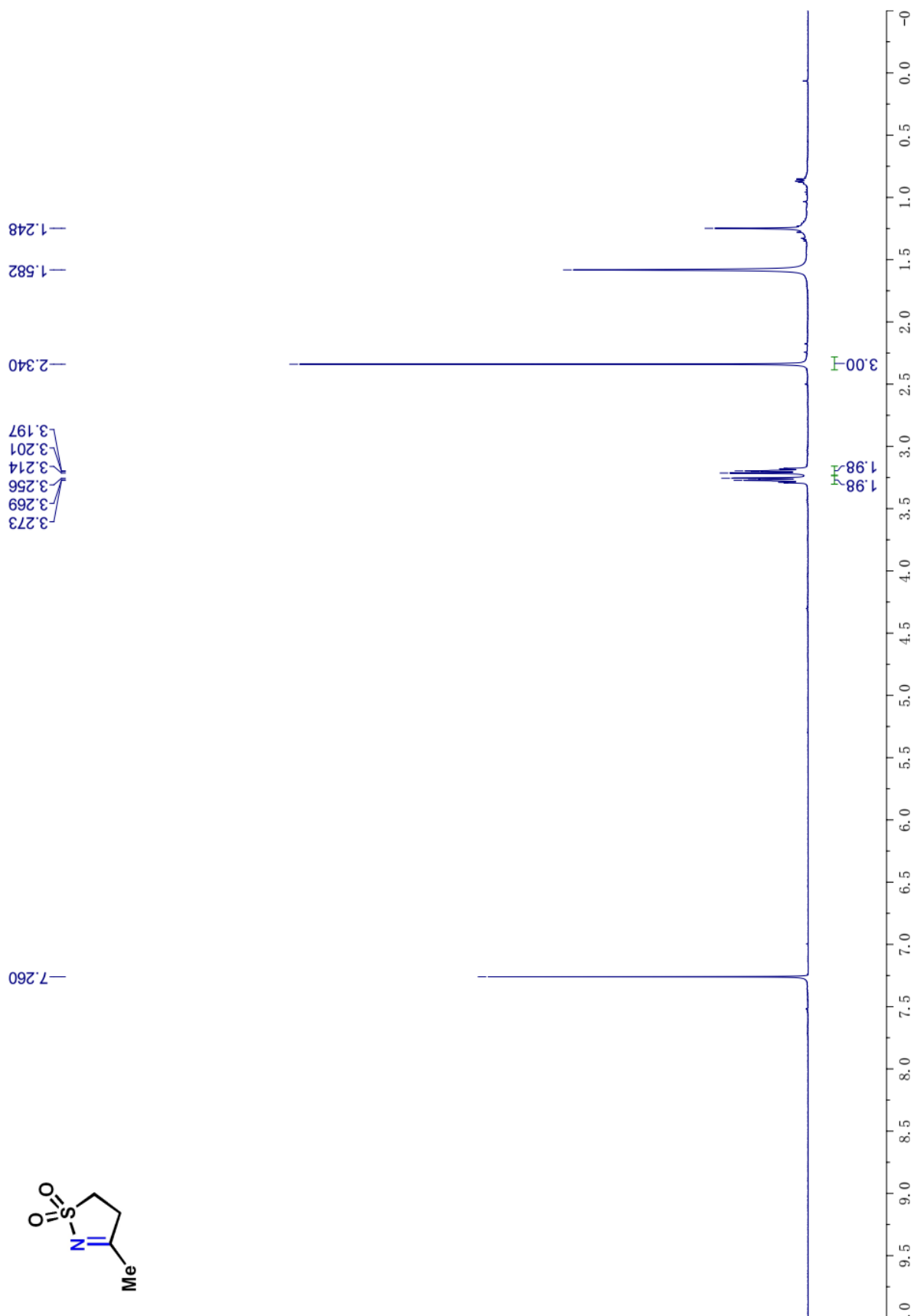
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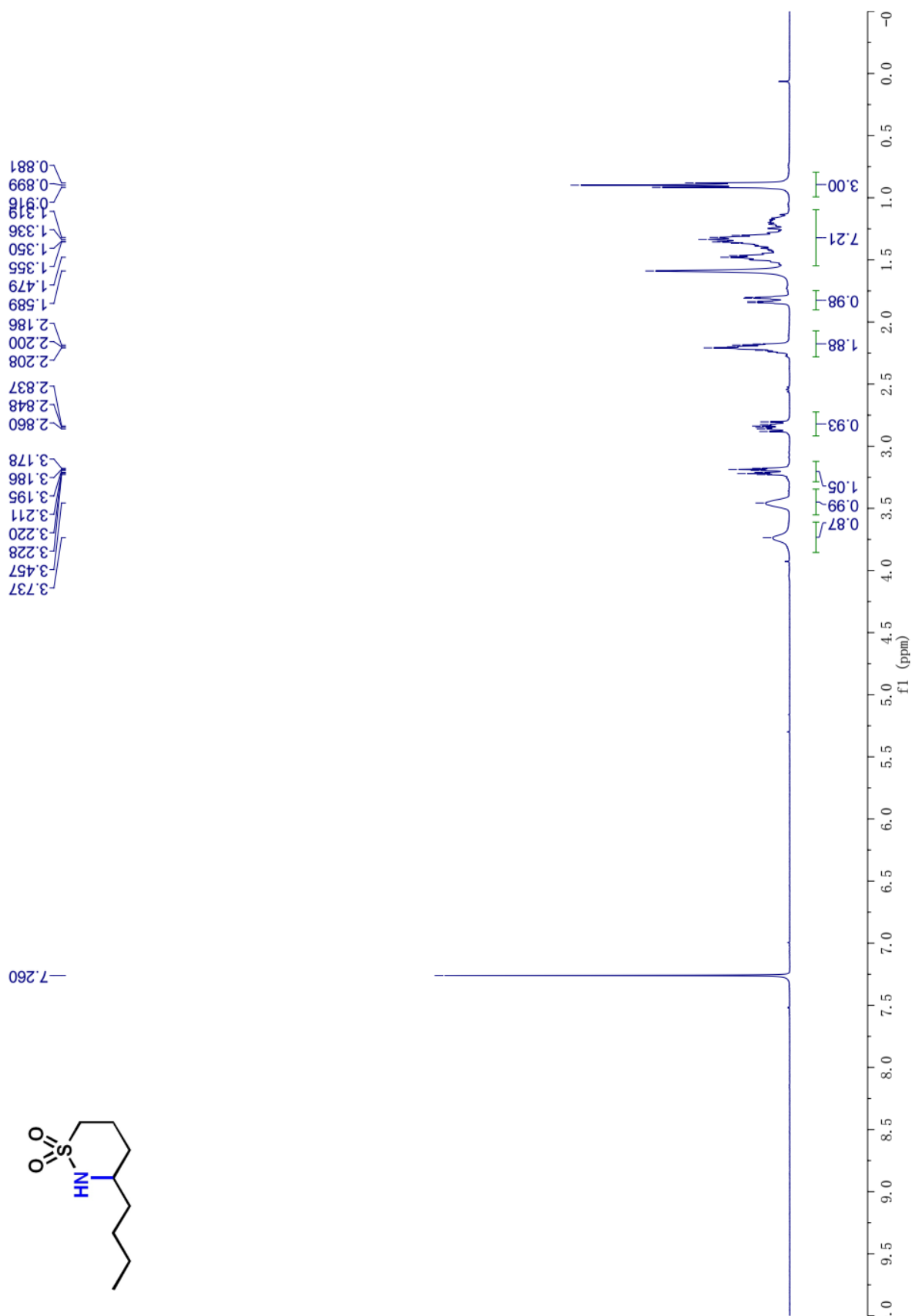
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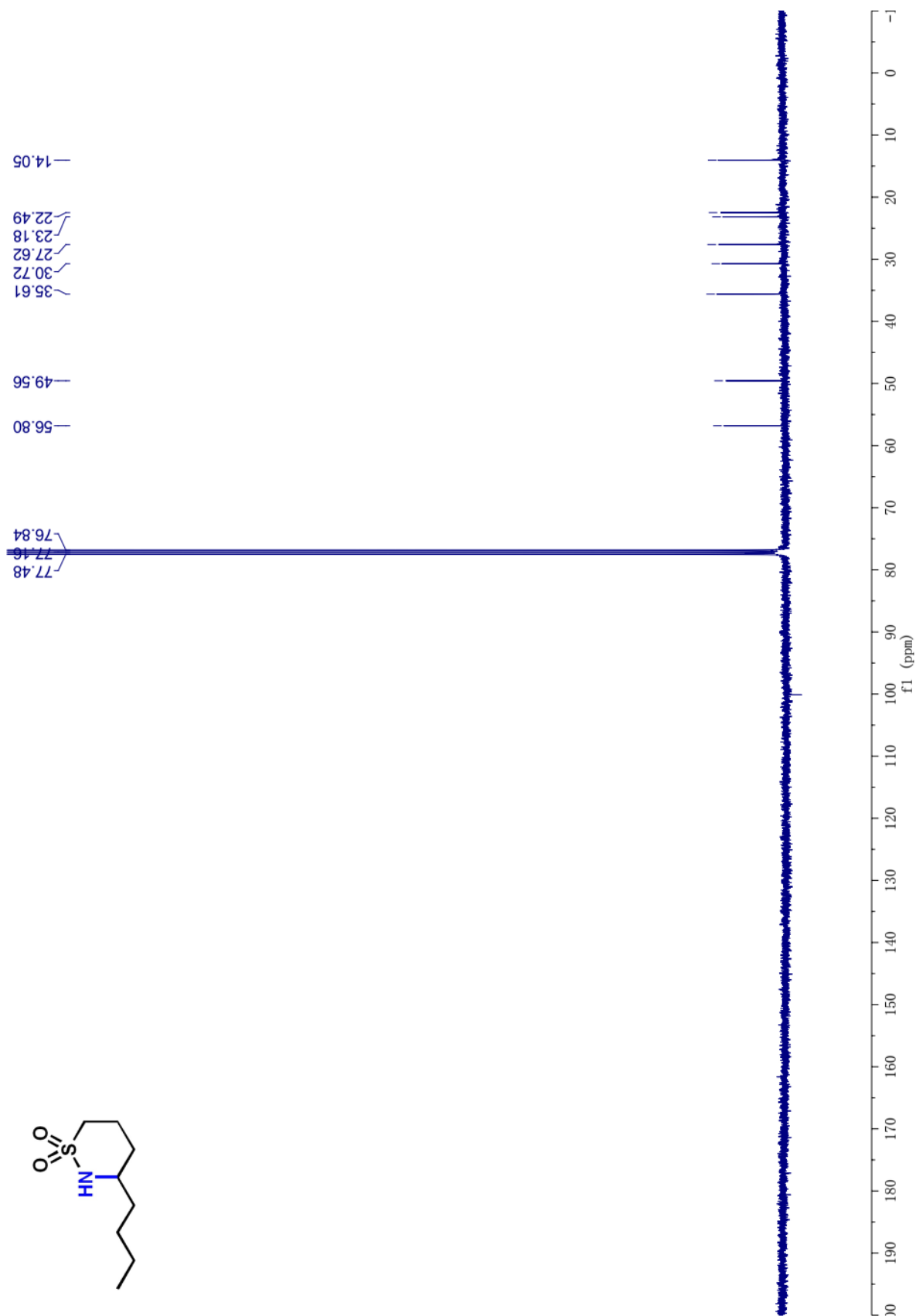
¹H NMR (400 MHz, CDCl₃) of **2r**

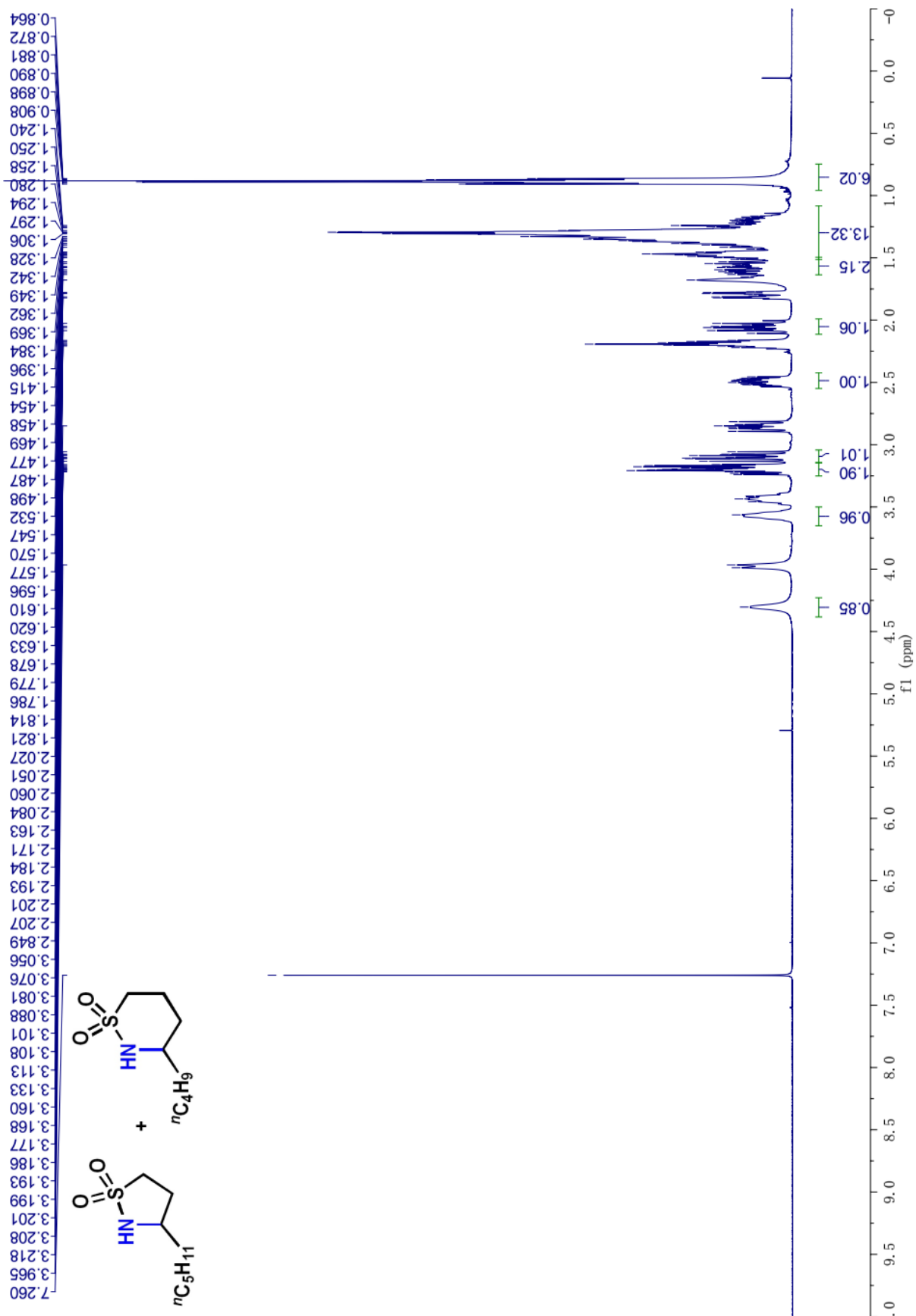


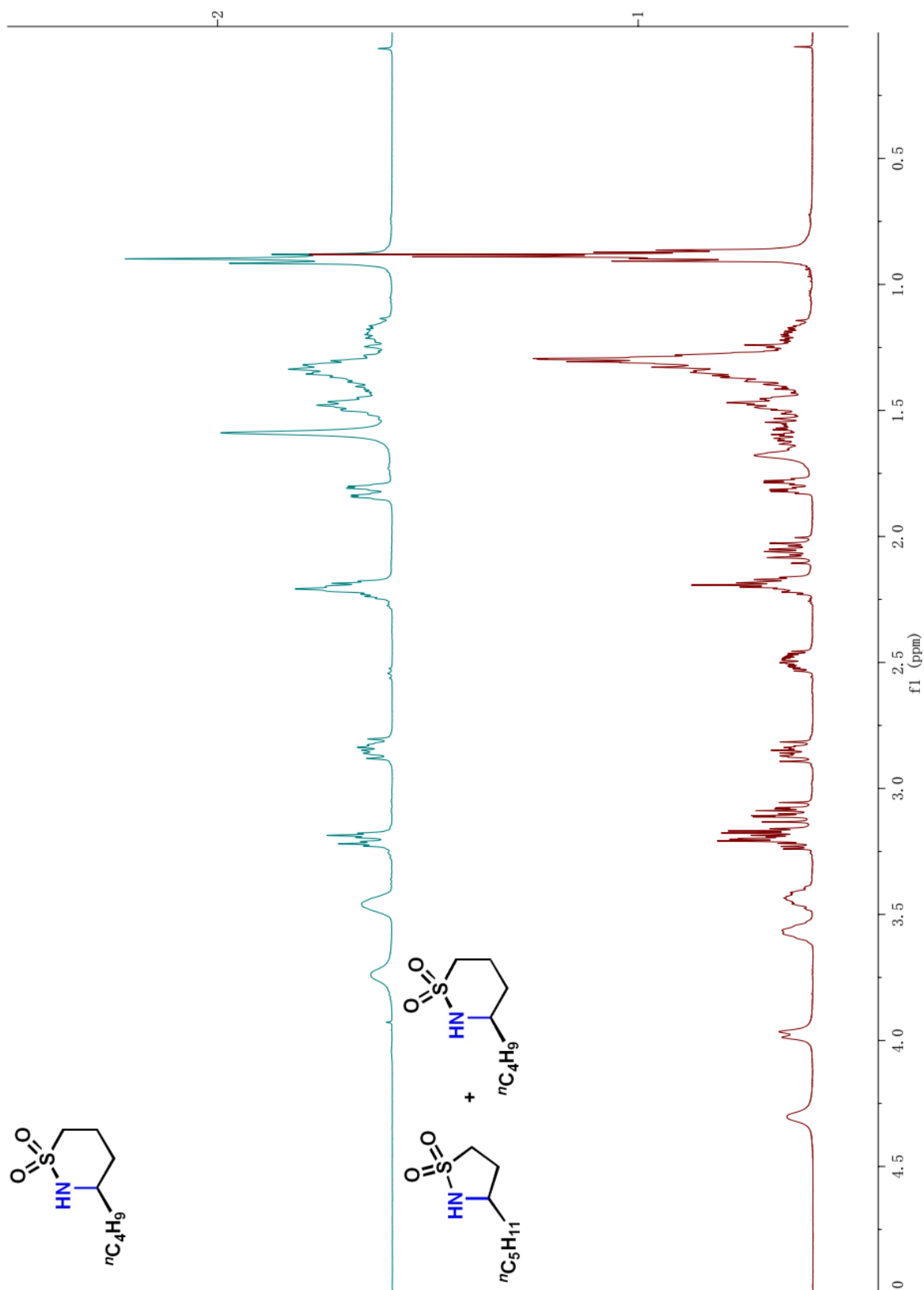
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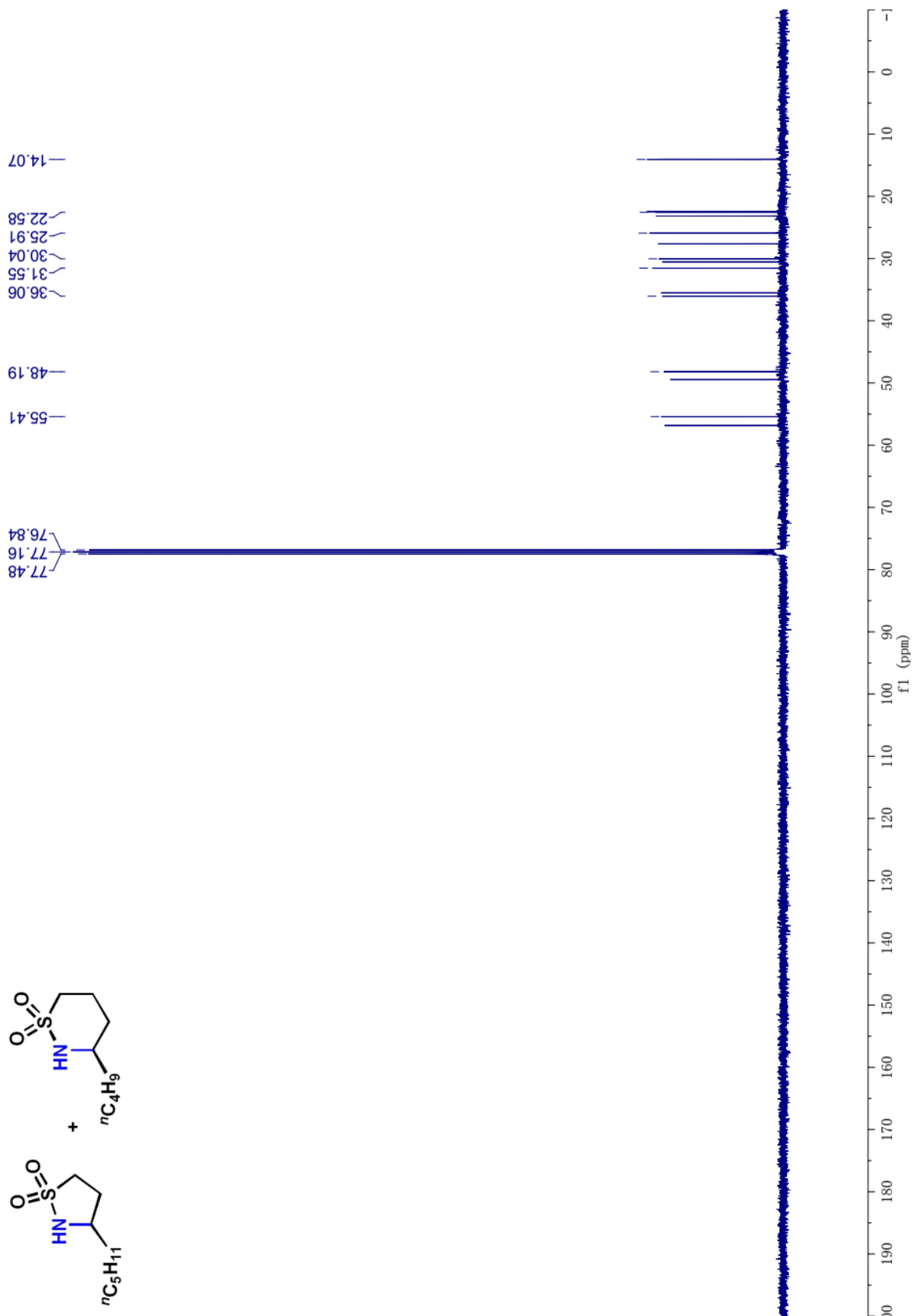
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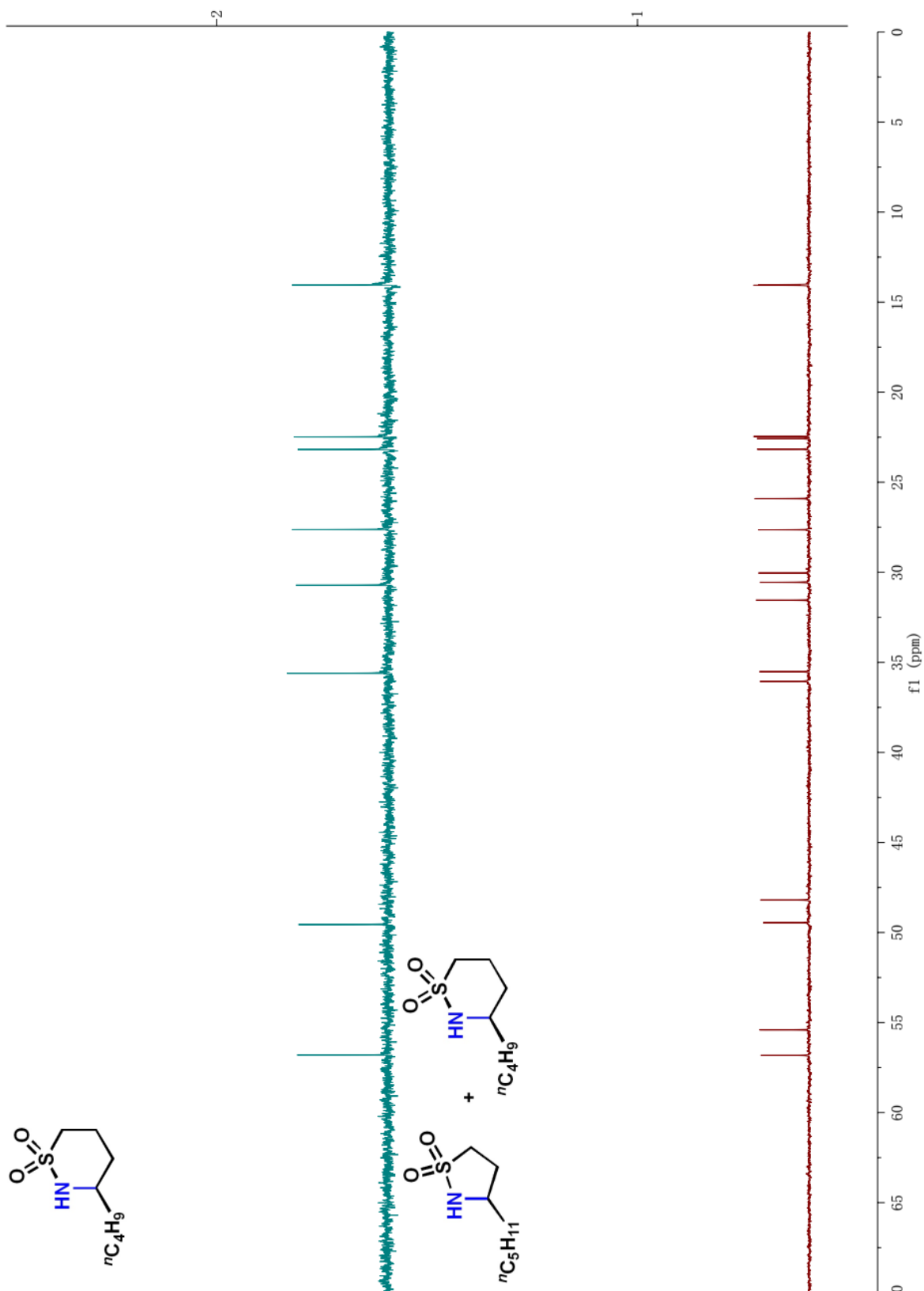
¹H NMR (400 MHz, CDCl₃) of **2t'**

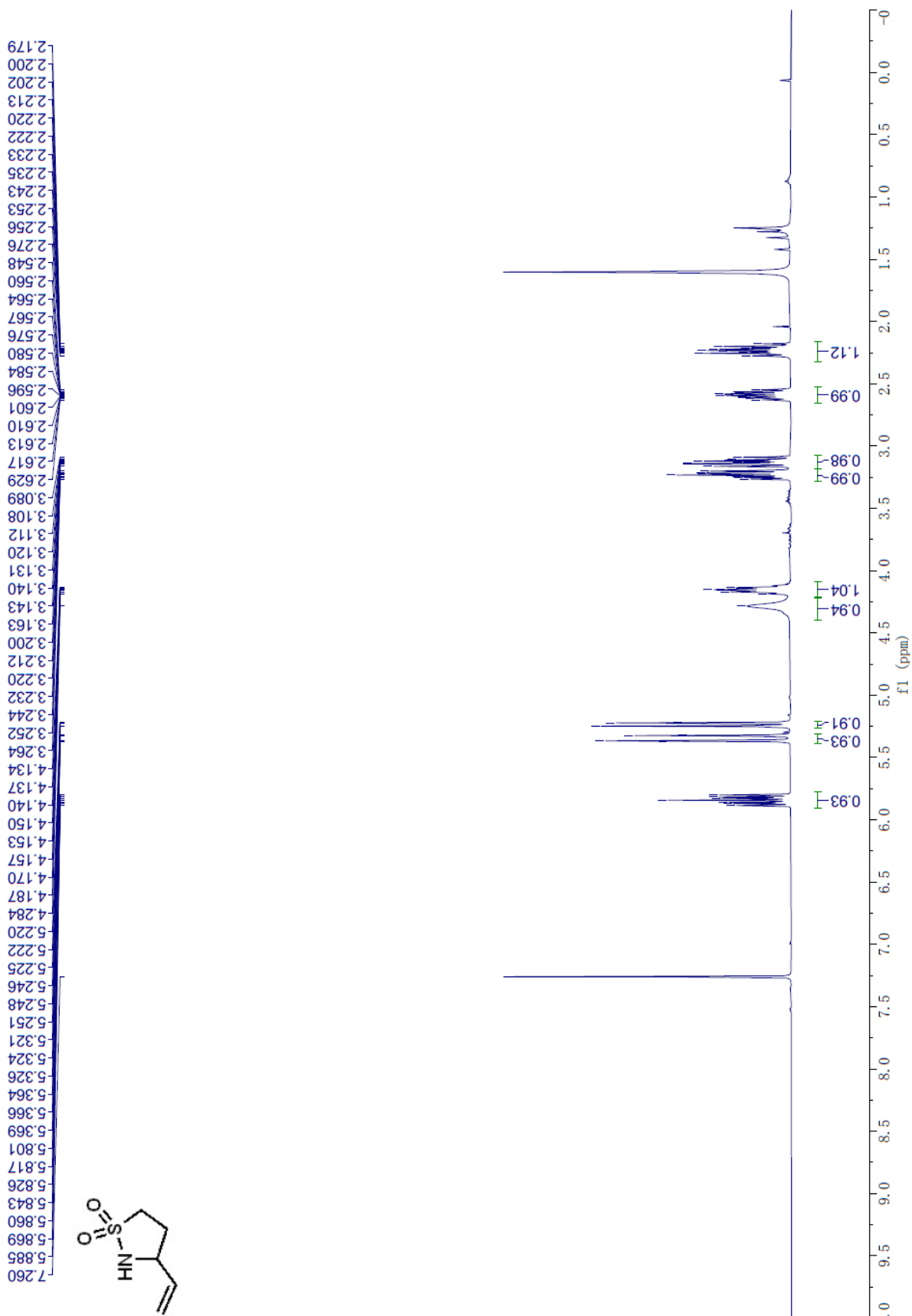
^{13}C NMR (100 MHz, CDCl_3) of **2t'**

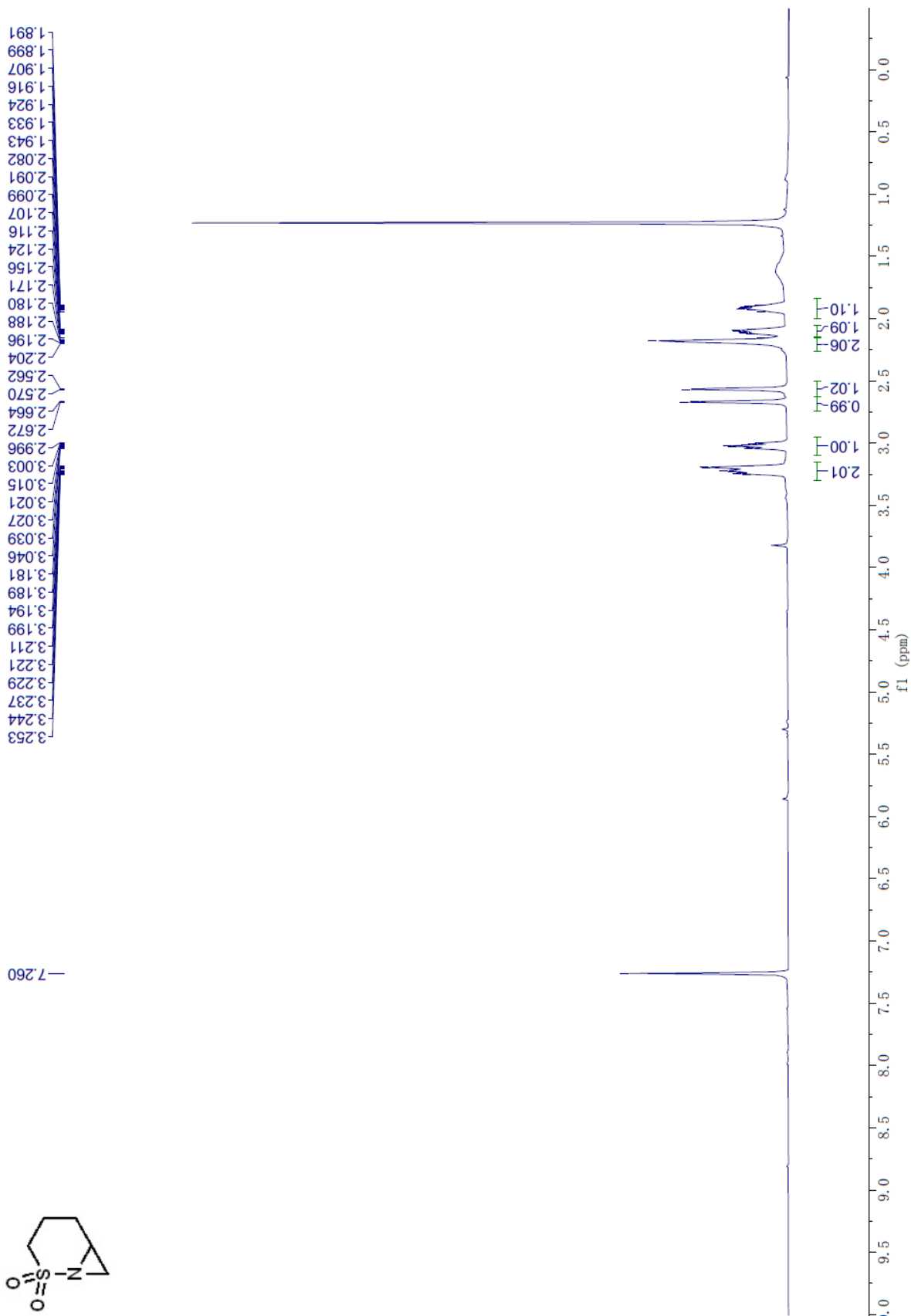
¹H NMR (400 MHz, CDCl₃) of **2t** and **2t'**

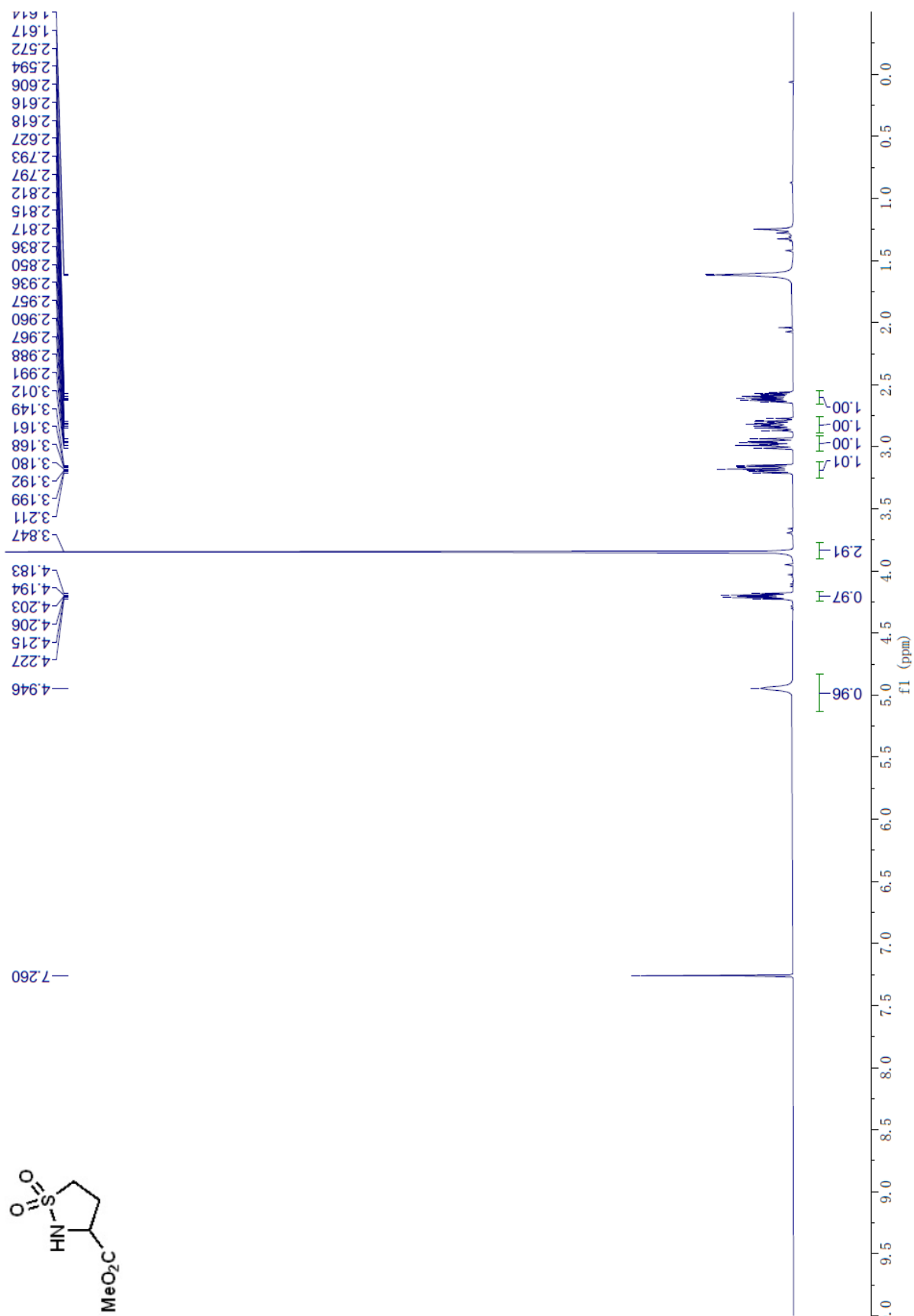


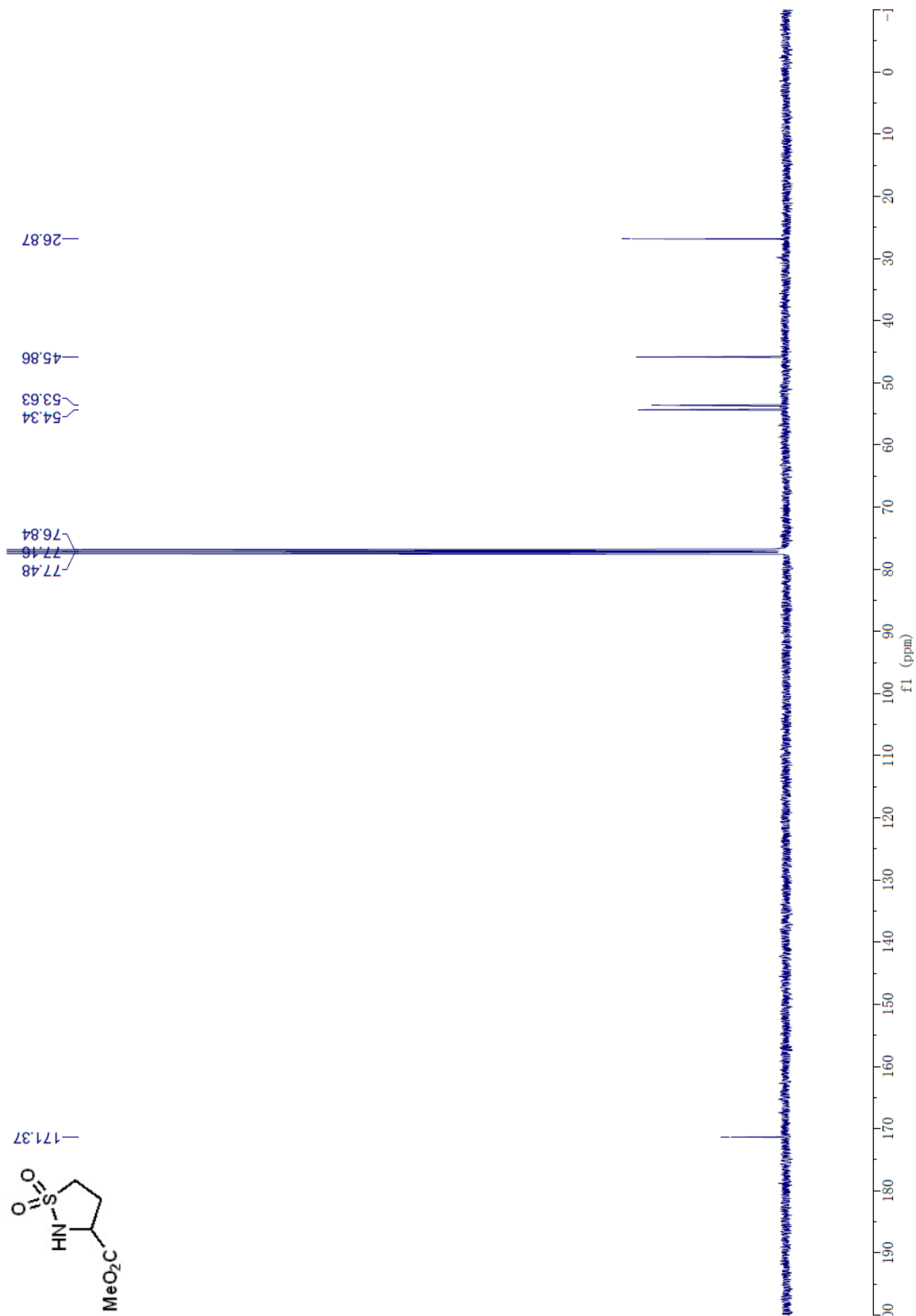
^{13}C NMR (100 MHz, CDCl_3) of **2t** and **2t**

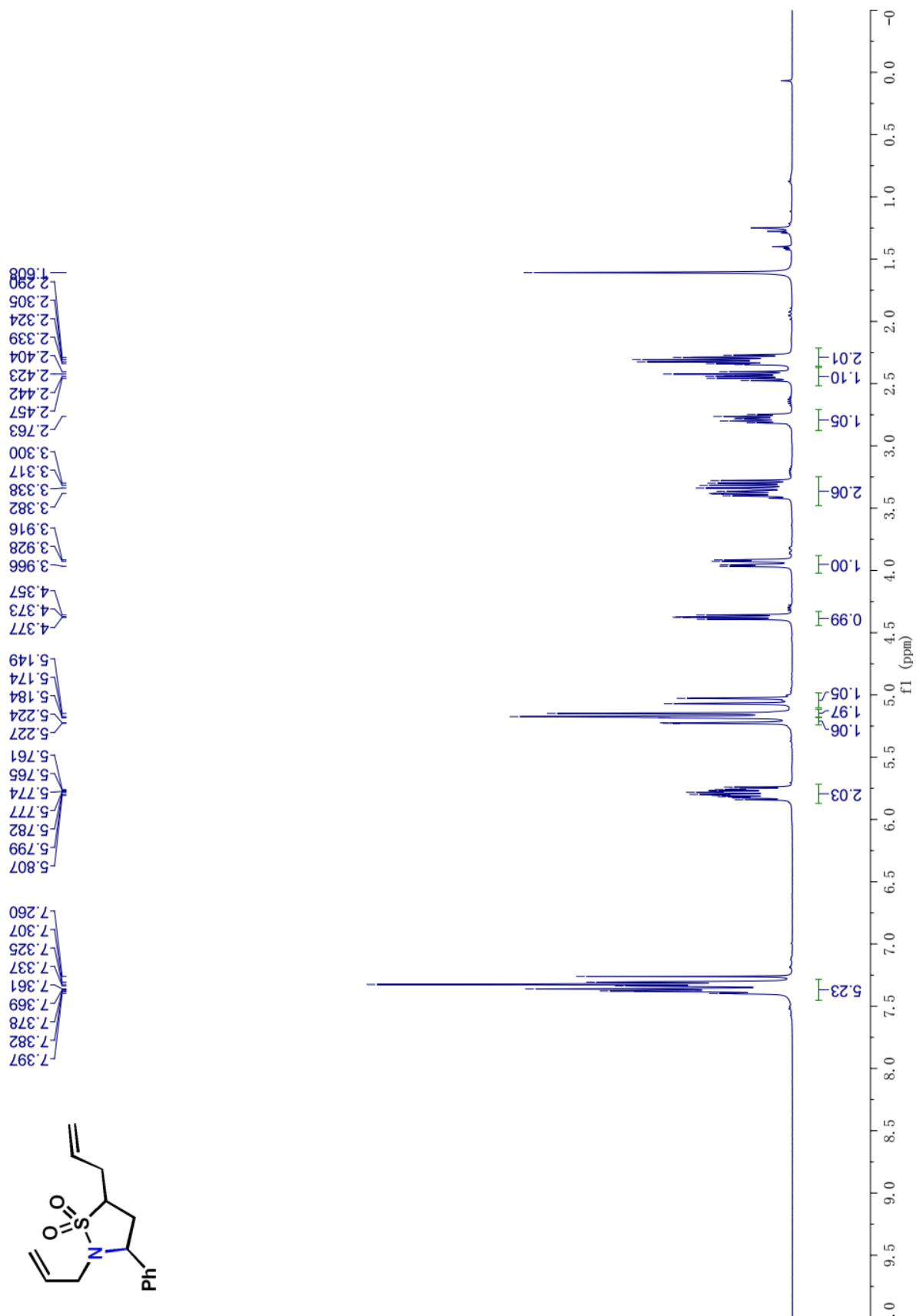


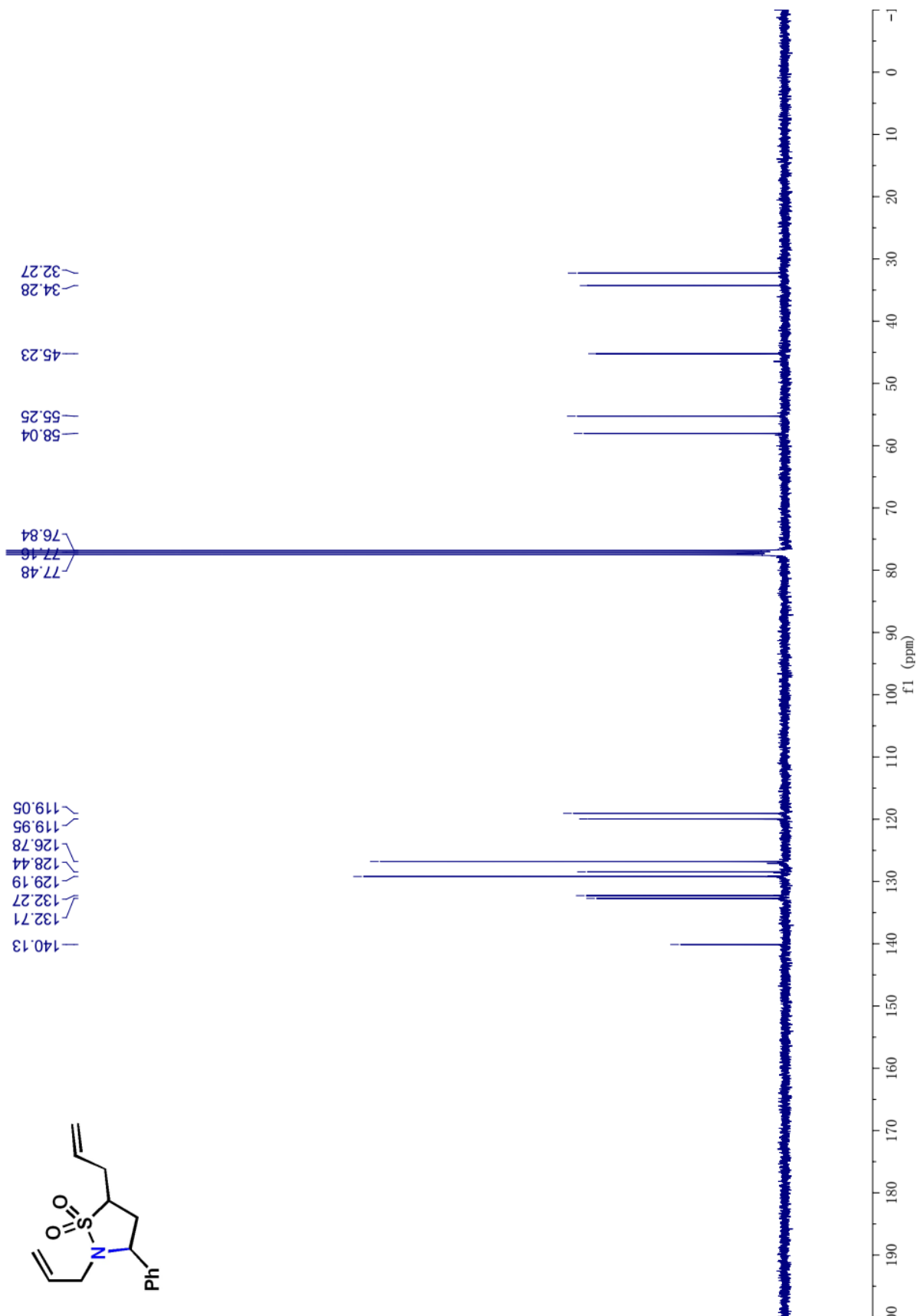
¹H NMR (400 MHz, CDCl₃) of **2u**

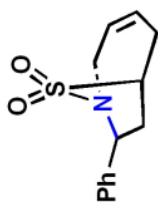
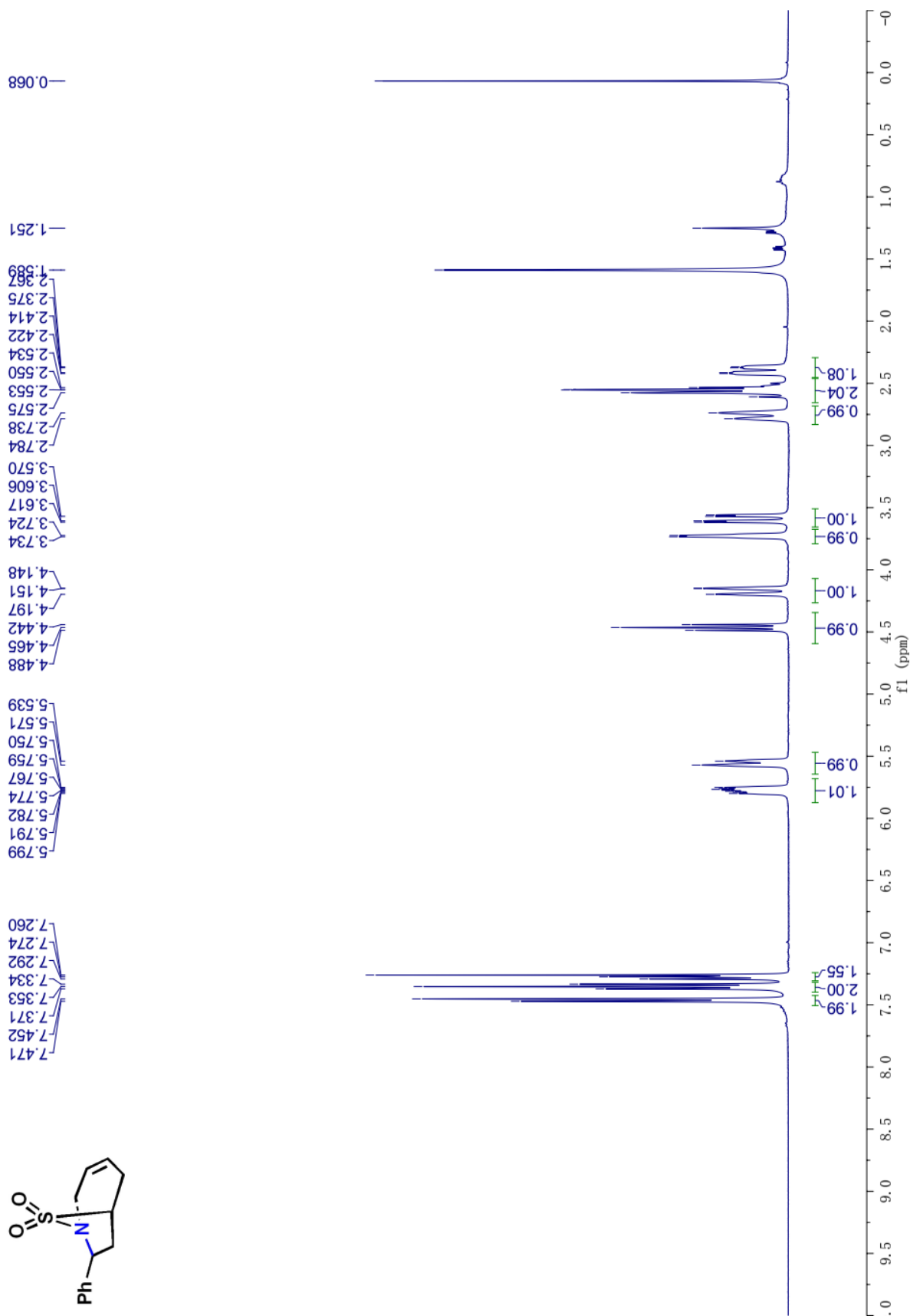
¹H NMR (400 MHz, CDCl₃) of **2u'**

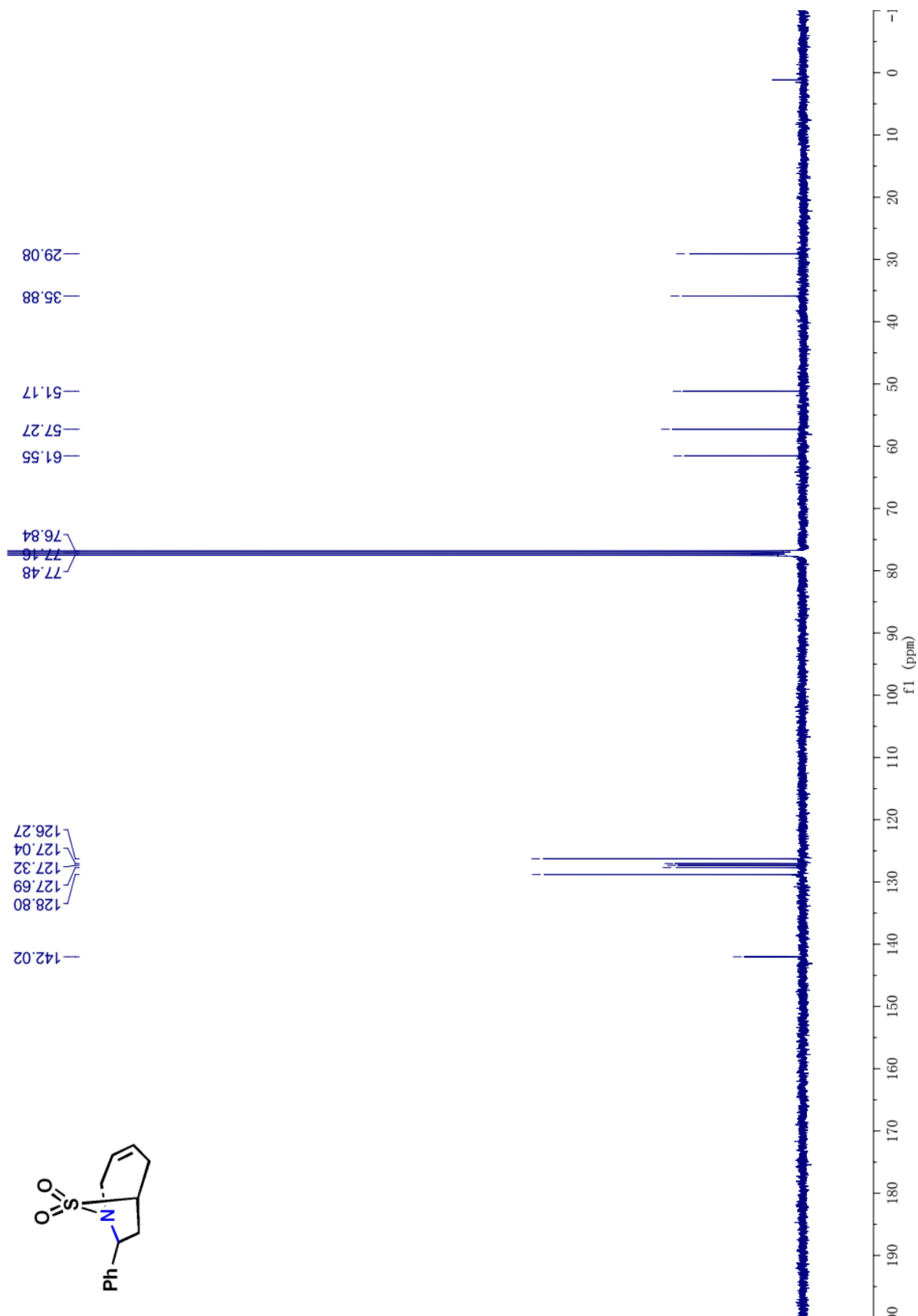
¹H NMR (400 MHz, CDCl₃) of **2v**

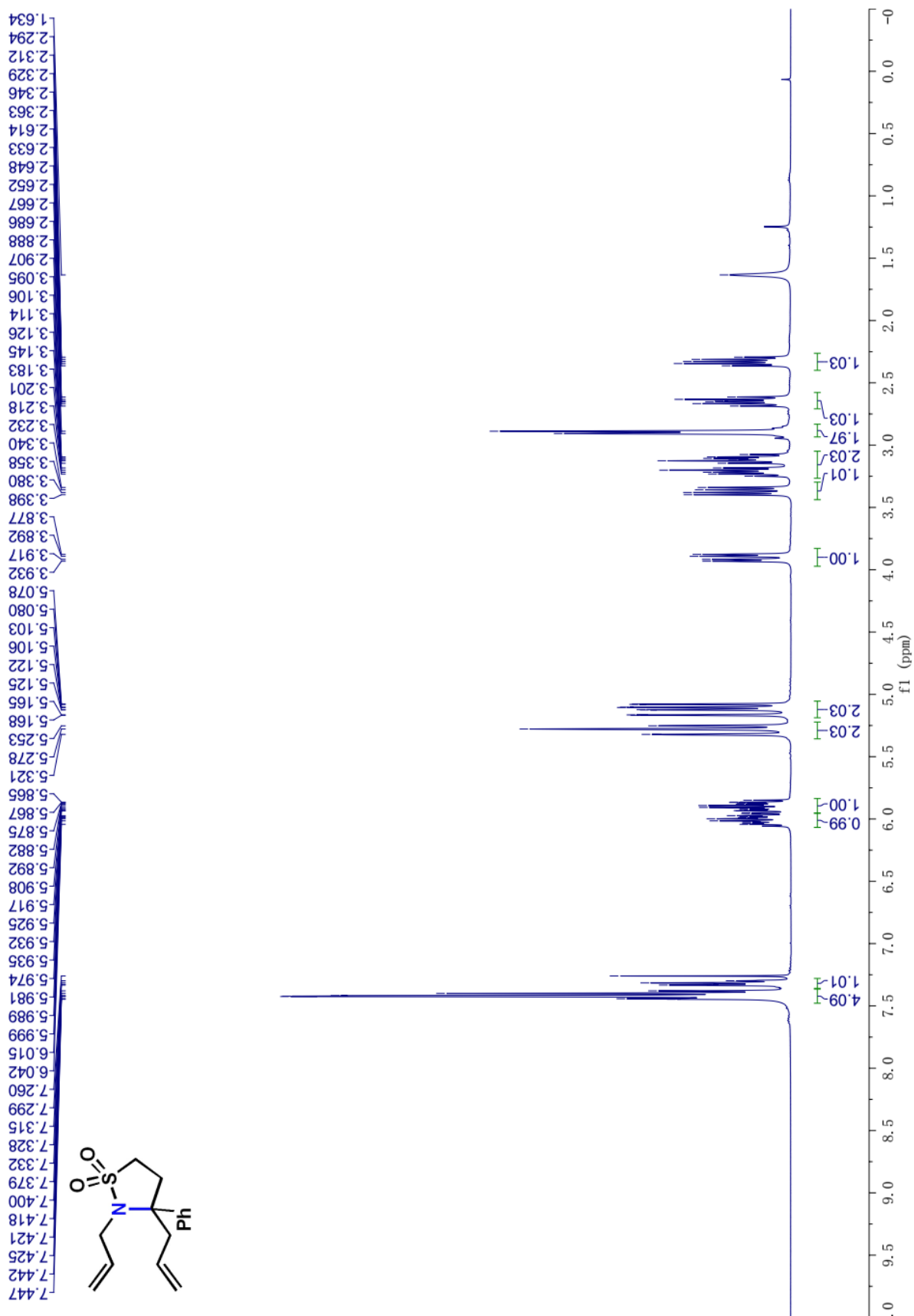
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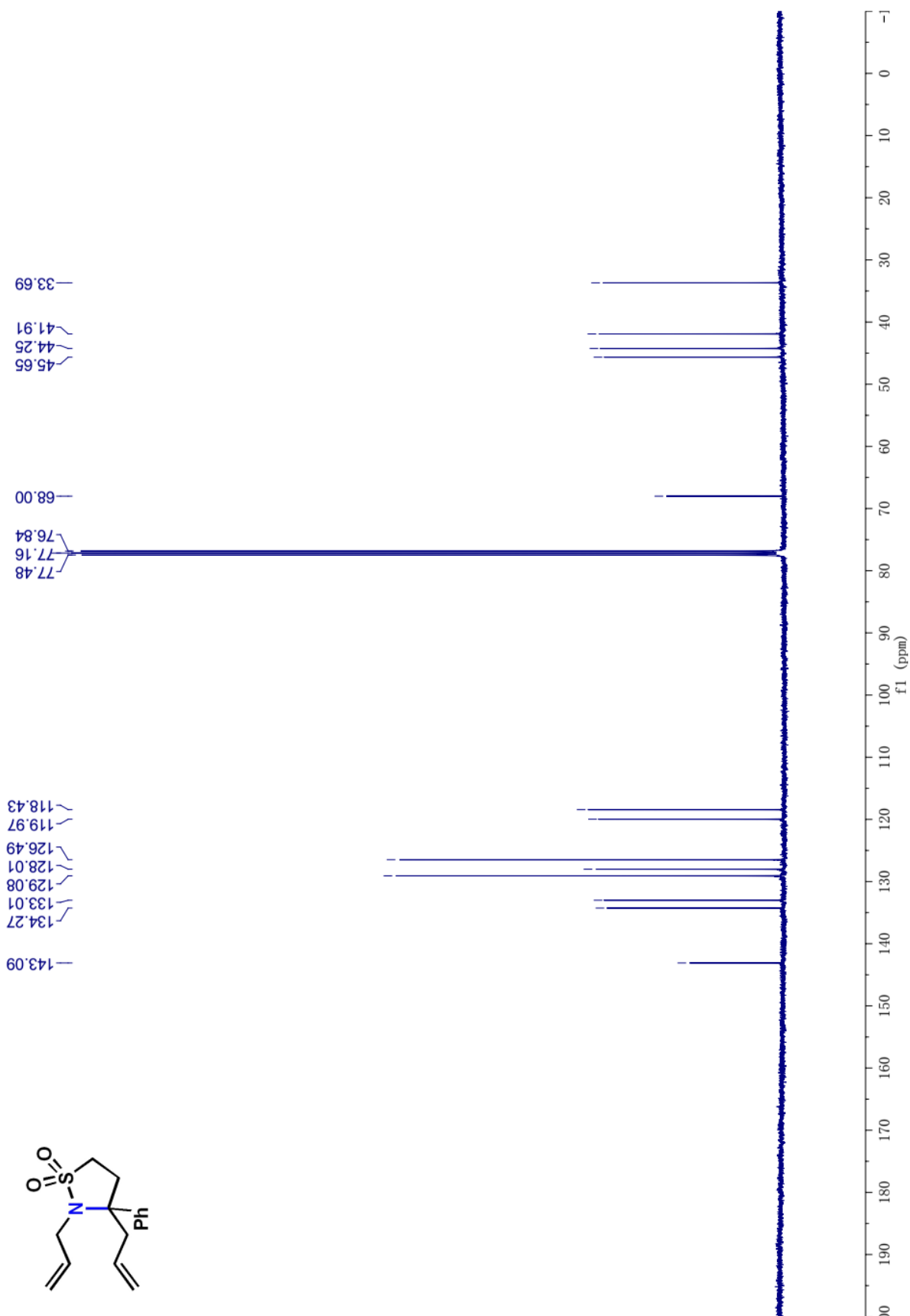
¹H NMR (400 MHz, CDCl₃) of **6**

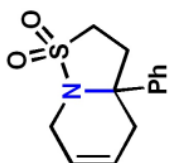
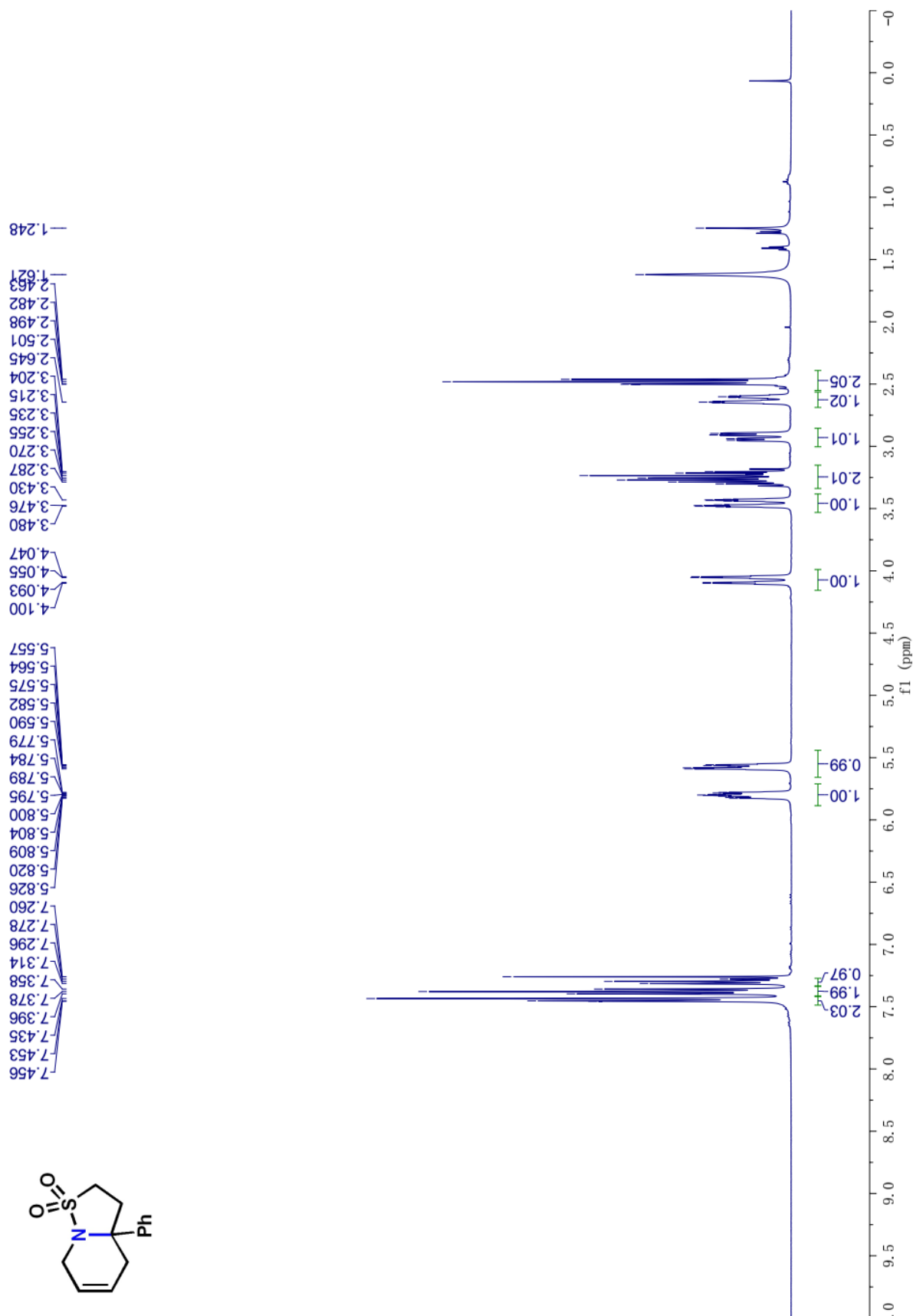
^{13}C NMR (100 MHz, CDCl_3) of **6**

¹H NMR (400 MHz, CDCl₃) of **4**

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

¹H NMR (400 MHz, CDCl₃) of **7**

^{13}C NMR (100 MHz, CDCl_3) of **7**

¹H NMR (400 MHz, CDCl₃) of **5**

^{13}C NMR (100 MHz, CDCl_3) of **5**