

Electronic Supporting Information:

Photo-Mediated Radical Relay Oximinosulfonamidation of Alkenes with *N*-Nitrosamines Triggered by DABSO

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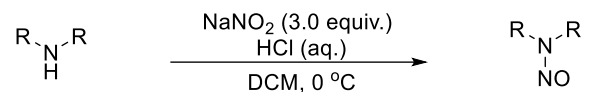
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1. General information.

Commercial reagents were used without purification and reactions were carried out under argon atmosphere with exclusion of moisture from reagents using standard techniques for manipulating air-sensitive compounds. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator using a water bath. Visualization of the developed chromatogram was performed by UV light or aqueous KMnO_4 stain. NMR-spectra were recorded on DRX-500 (500 MHz) spectrometer and calibrated by using residual undeuterated chloroform ($\delta = 7.26$ ppm for ^1H , 77.16 ppm for ^{13}C), $\text{DMSO-}d_6$ ($\delta = 2.50$ ppm for ^1H , 39.52 ppm for ^{13}C), CD_3OD ($\delta = 3.31$ ppm for ^1H , 49.00 ppm for ^{13}C) and CD_3CN ($\delta = 1.94$ ppm for ^1H , 1.32 and 118.26 ppm for ^{13}C) as internal references. Data for ^1H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, td = triplet of doublets, brs = broad singlet), coupling constant (Hz), and integration. High resolution mass spectra were obtained from the Agilent MSD-Trap-XCT. Kessil lamps were purchased from Tansoole, with precise wavelengths (390 nm). Single crystal X-ray crystallography data was obtained on Bruker D8 venture. Ultraviolet-visible absorption experiments were performed using a METASH UV-8000S (T) spectrophotometer. Thin-layer chromatography (TLC) was performed on 0.25 mm silica gel F-254 plates (Yantai Jiangyou).

2. Synthesis of starting materials.

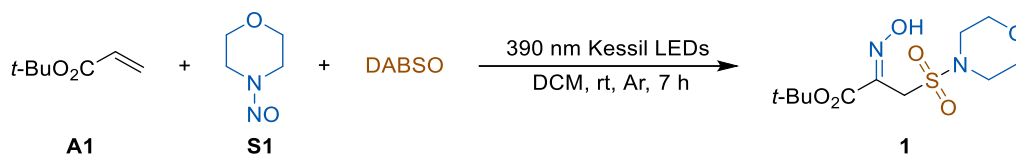
2.1. Synthesis of *N*-nitrosamines.^[1]



Secondary amine (10 mmol, 1.0 equiv.) and sodium nitrite (2.1 g, 30 mmol, 3.0 equiv.) were dissolved in DCM (50 mL, 0.2 M) at 0 °C in an ice bath, and concentrated hydrochloric acid was slowly added dropwise to this solution until the pH was adjusted to 1–2. The solution was stirred at 0 °C until the reaction was complete as indicated by TLC, and then quenched by adding water (100 mL). The crude mixture was extracted with EtOAc (50 mL × 3), and the combined organic layers were washed with saturated NaCl solution, dried over anhydrous Na₂SO₄, and concentrated under vacuum using a rotary evaporator. The resulting residue was purified by flash column chromatography using PE/EtOAc system (PE/EtOAc, 10/1–3/1, v/v).

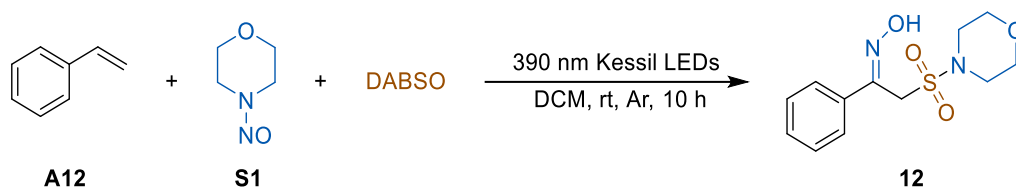
3. General procedure for coupling reaction.

3.1. Procedure A:



The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (25.7 mg, 0.2 mmol), **S1** (35.0 mg, 0.3 mmol, 1.5 equiv.) and DABSO (96.2 mg, 0.4 mmol, 2.0 equiv.) were sequentially added in DCM (2 mL) under argon atmosphere. The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (Kessil KSPR160–390 LED Grow Light; 5-6 cm away). Upon completion, the reaction mixture was filtered and subsequently concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using PE/EtOAc system (PE/EtOAc, 1/1, v/v) to afford product **1** as a white powder (44.4 mg, 72% yield). Unless otherwise noted, **1–49** were synthesized under the same reaction conditions.

3.2. Gram-scale synthesis of **12**.



The reaction was performed in an oven-dried 250-mL schlenk tube equipped with a stir bar. **A12** (520.3 mg, 5.0 mmol), **S1** (870.5 mg, 7.5 mmol, 1.5 equiv.) and DABSO (2.41 g, 10.0 mmol, 2.0 equiv.) were sequentially added in DCM (50 mL) under argon atmosphere. The mixture was stirred at room temperature with cooling fans under irradiation of four sets of 40 W 390 nm LED lamps (Kessil KSPR160–390 LED Grow Light; 8–10 cm away). The mixture until the starting materials were consumed as indicated by TLC analysis. Upon completion, the reaction mixture was filtered and subsequently concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using PE/EtOAc system (PE/EtOAc, 2/1, v/v) to afford product **1** as a white solid (1.24 g, 87% yield).

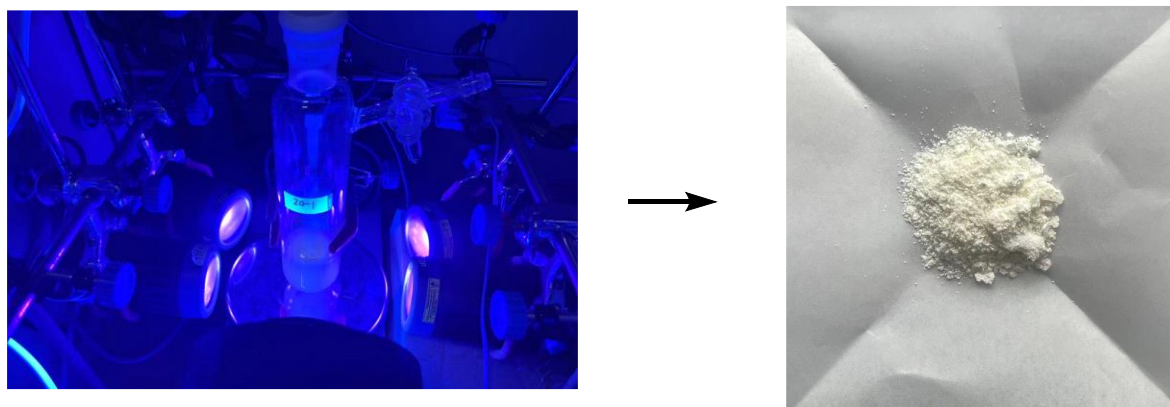
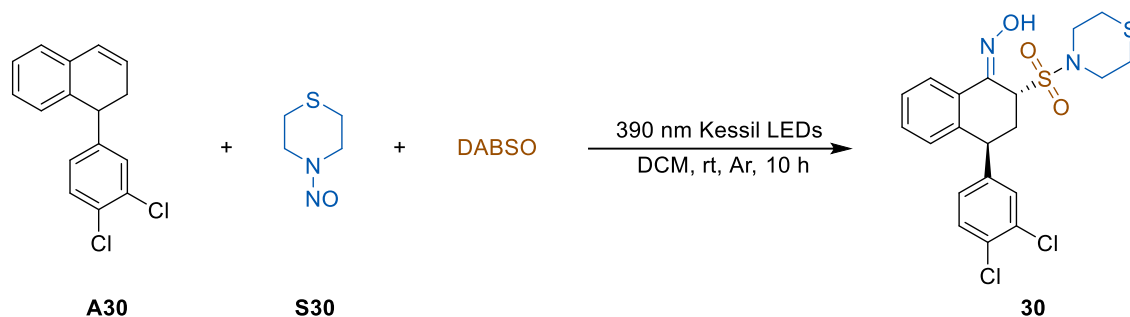


Figure S1. Gram-scale reaction set-up.

3.3. Gram-scale synthesis of 30.



The reaction was performed in an oven-dried 250-mL schlenk tube equipped with a stir bar. **A30** (1.1 g, 4.0 mmol), **S30** (792.2 mg, 6.0 mmol, 1.5 equiv.) and DABSO (1.93 g, 8.0 mmol, 2.0 equiv.) were sequentially added in DCM (40 mL) under argon atmosphere. The mixture was stirred at room temperature with cooling fans under irradiation of four sets of 40 W 390 nm LED lamps (Kessil KSPR160–390 LED Grow Light; 8–10 cm away). The mixture until the starting materials were consumed as indicated by TLC analysis. Upon completion, the reaction mixture was filtered and subsequently concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using PE/EtOAc system (PE/EtOAc, 2/1, v/v) to afford product **30** as a yellowish powder (1.32 g, 70% yield).

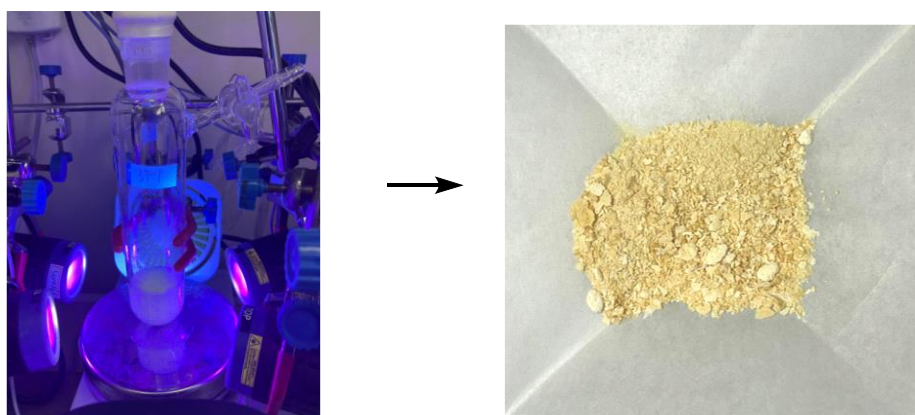
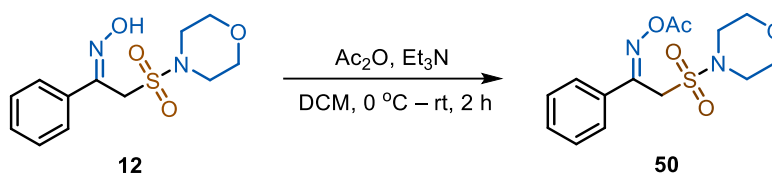


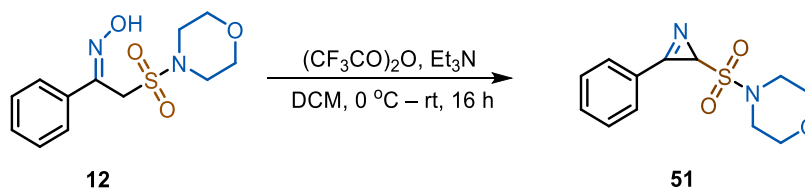
Figure S2. Gram-scale reaction set-up.

4. Synthetic applications.

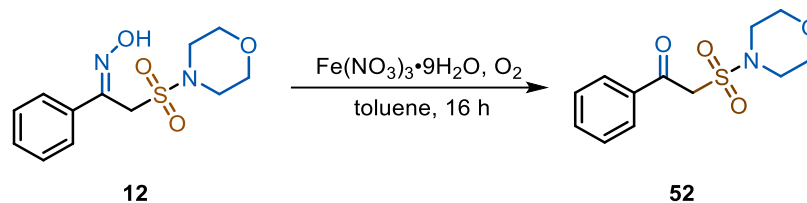
4.1. Follow-up chemistry.



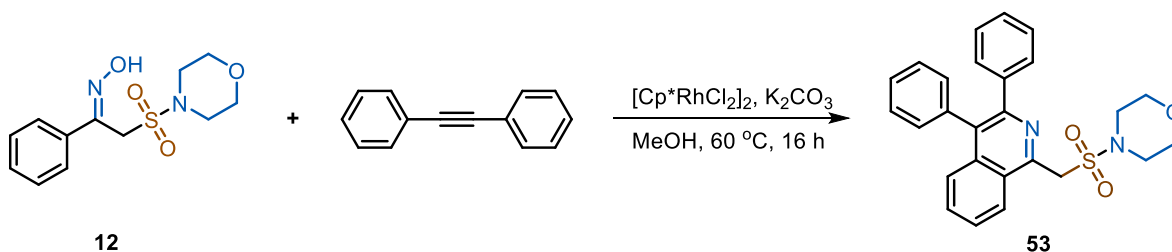
Acetic anhydride (28.2 μL , 0.3 mmol) was slowly added to a solution of **12** (56.8 mg, 0.2 mmol) and triethylamine (55.6 μL , 0.4 mmol) in DCM (2 mL) under argon atmosphere at 0 °C. The reaction was stirred for 2 h, and then quenched by adding saturated NaHCO_3 solution (5 mL). The crude mixture was extracted with EtOAc (5 mL \times 3), and the combined organic layers were washed with saturated NaCl solution, dried over anhydrous Na_2SO_4 , and concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 2/1, v/v) to afford product **50** as a white powder (62.0 mg, 95% yield, $Z/E = 7.7:1$).



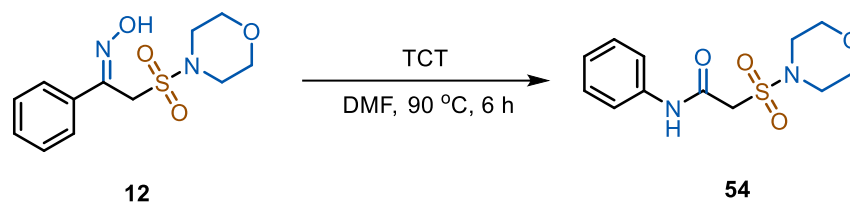
Trifluoroacetic anhydride (69.5 μL , 0.5 mmol) and triethylamine (111.2 μL , 0.8 mmol) were slowly added to a solution of **12** (56.8 mg, 0.2 mmol) in DCM (2 mL) under argon atmosphere at 0 °C. The reaction was stirred at for 16 h at room temperature, and then quenched by adding saturated NaHCO_3 solution (5 mL). The crude mixture was extracted with EtOAc (5 mL \times 3), and the combined organic layers were washed with saturated NaCl solution, dried over anhydrous Na_2SO_4 , and concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 3/1, v/v) to afford product **51** as white powder (32.0 mg, 60% yield).



$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (121.2 mg, 0.3 mmol) was added to a solution of **12** (56.8 mg, 0.2 mmol) in toluene (2 mL) under oxygen atmosphere, The reaction was stirred at for 16 h, and then quenched by adding water (5 mL). The crude mixture was extracted with EtOAc (5 mL \times 3), and the combined organic layers were washed with saturated NaCl solution, dried over anhydrous Na_2SO_4 , and concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 3/1, v/v) to afford product **52** as a white powder (33.9 mg, 63% yield).^[2]



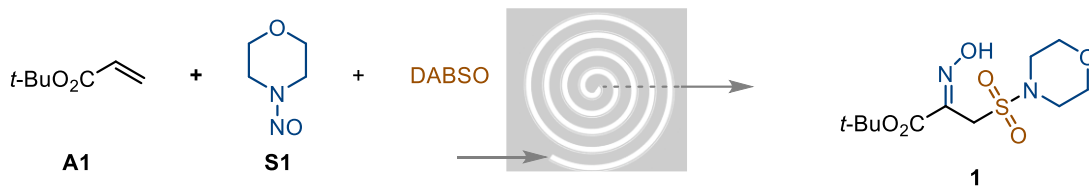
Diphenylacetylene (39 mg, 0.22 mmol, 1.1 equiv.), $[\text{Cp}^*\text{RhCl}_2]_2$ (2.5 mol%, 3 mg) and K_2CO_3 (30 mg, 0.2 mmol, 1.1 equiv.) were added to a solution of **12** (56.8 mg, 0.2 mmol) in MeOH (1 mL) under argon atmosphere, the reaction mixture was stirred in an oil bath at 60 °C for 16 h. The reaction was cooled down and diluted with EtOAc. Silica was added to the flask and concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 3/1, v/v) to afford product **53** as a yellowish powder (62.2 mg, 70% yield).^[3]



Cyanuric chloride (73.2 mg, 0.4 mmol, 2.0 equiv.) was added in DMF (0.2 mL) under oxygen atmosphere, after stirring the mixture for 30 minutes at room temperature, **12** (56.8 mg, 0.2 mmol) in DMF (0.2 mL) was added and the mixture was stirred at 90 °C for 6 h. The reaction was cooled down and diluted with EtOAc. Silica was added to the flask and concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 2/1, v/v) to afford product **54** as a white powder (47.7 mg, 84% yield).^[4]

4.2. Scale up in continuous-flow reactors.

$V = 1 \text{ mL}$, $OD = 1.6 \text{ mm}$, $ID = 0.8 \text{ mm}$, $\text{length} = 200 \text{ cm}$.



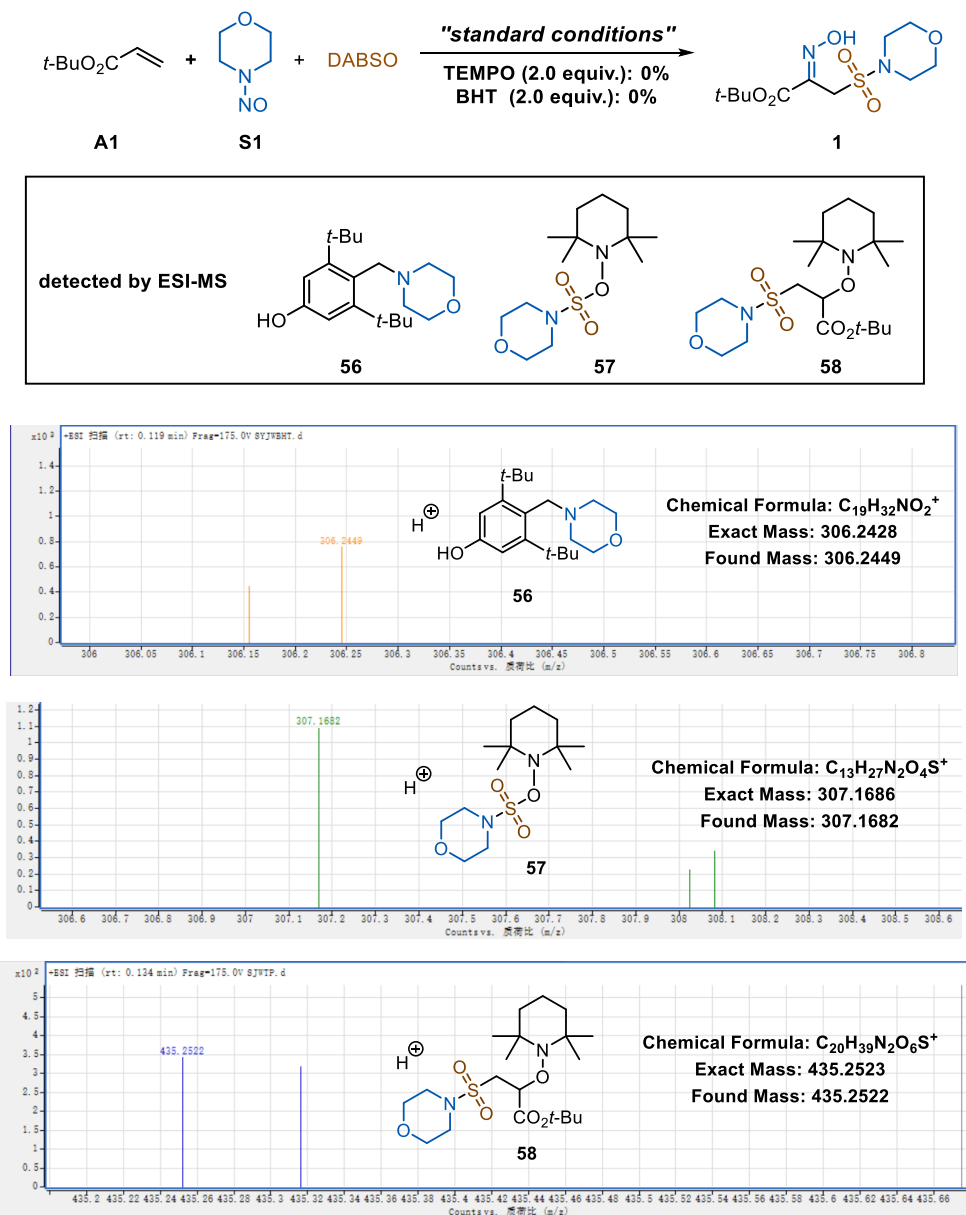
flow rate	collection time	residence time	conversion	yield	productivity
50 $\mu\text{L}/\text{min}$	200 min	20 min	100%	70%	0.3 mmol/h



The flow apparatus was purged with degassed DCM to remove the air first. A 20 mL Erlenmeyer flask was charged with **A1** (128.5 mg, 1 mmol), **S1** (175.0 mg, 1.5 mmol, 1.5 equiv.) and DABSO (481.0 mg, 2.0 mmol, 2.0 equiv.) in $\text{DCM}/\text{H}_2\text{O} = 4:1$ (10 mL) followed by sonication for 5 minutes, placed in a 20 mL disposable syringe and mounted on a syringe pump. The mixture liquid feed was pumped into the flow reactor. The tubing was then irradiated by a 40 W 390 nm Kessil lamp. The flow apparatus itself was set up with residence time = 20 min, flow rate = 50 $\mu\text{L}/\text{min}$. Upon completion, the reaction diluted with EtOAc and silica was added to the flask and concentrated under vacuum using a rotary evaporator. The resulting residue was then purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 3/1, v/v) to afford product **1** as a white powder (215.6 mg, 70% yield).

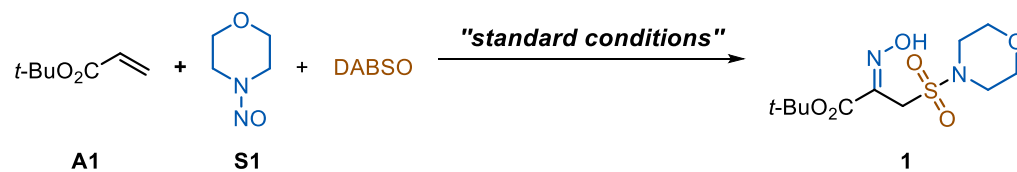
5. Mechanistic investigations.

5.1. Radical trapping experiments.



The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (25.7 mg, 0.2 mmol), **S1** (35.0 mg, 0.3 mmol, 1.5 equiv.), DABSO (96.2 mg, 0.4 mmol, 2.0 equiv.), TEMPO (31.3 mg, 0.2 mmol, 2.0 equiv.) or BHT (44.1 mg, 0.2 mmol, 2.0 equiv.) were sequentially added in DCM (2 mL) under argon atmosphere. The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (Kessil KSPR160–390 LED Grow Light; 5–6 cm away). Upon completion, the crude mixture was analyzed by ESI-MS.

5.2. Studies with radical initiators.



deviation from standard conditions		yield (%)
adding radical initiator, 60 °C, without light	AIBN (1.0 equiv.)	0
	DTBP (1.0 equiv.)	0
	BPO (1.0 equiv.)	0
	DLP (10 mol%)	0

The reaction was performed in a 15 mL pressure tube, **A1** (25.7 mg, 0.2 mmol), **S1** (35.0 mg, 0.3 mmol, 1.5 equiv.), **DABSO** (96.2 mg, 0.4 mmol, 2.0 equiv.) were dissolved in DCM (2 mL) under argon atmosphere. Radical initiator was added and the tube was sealed. The reaction mixture was stirred at 60 °C for 7 h (oil bath as the heat source). Upon completion, the yield was determined by ^1H NMR using 1,3,5-trimethoxybenzene as an internal standard.

5.3. Ultraviolet-visible absorption experiments.

UV-vis absorption spectra of **S1**, DABSO and **S1**+DABSO in DCM were recorded in screw-top 1.0 cm quartz cuvettes using a METASH UV-8000S (T) spectrophotometer. The concentration of each component was 5×10^{-4} M.

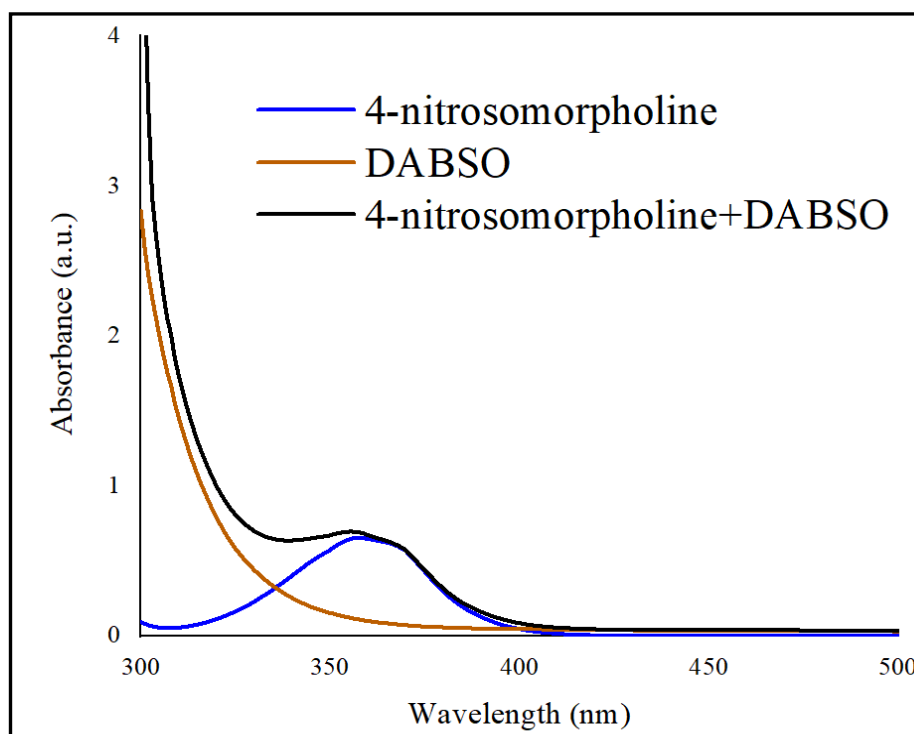


Figure S3. UV-vis absorption spectroscopy.

5.4. NMR experiments.

The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (25.7 mg, 0.2 mmol), **S1** (35.0 mg, 0.3 mmol, 1.5 equiv.) and DABSO (96.2 mg, 0.4 mmol, 2.0 equiv.) were sequentially added in DCM (2 mL) under argon atmosphere. The reaction mixture was stirred for 7 h and then transferred to an NMR tube for analysis.

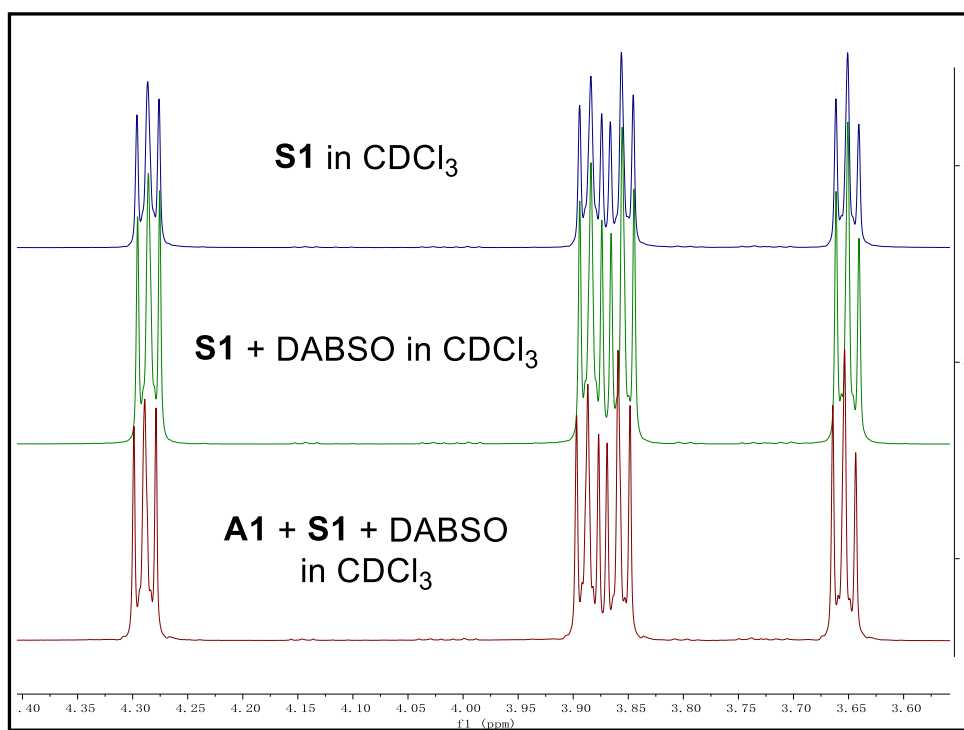
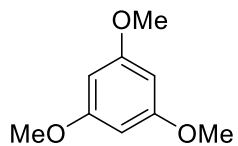
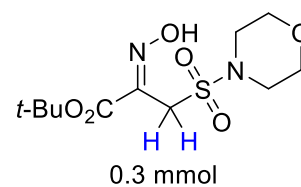


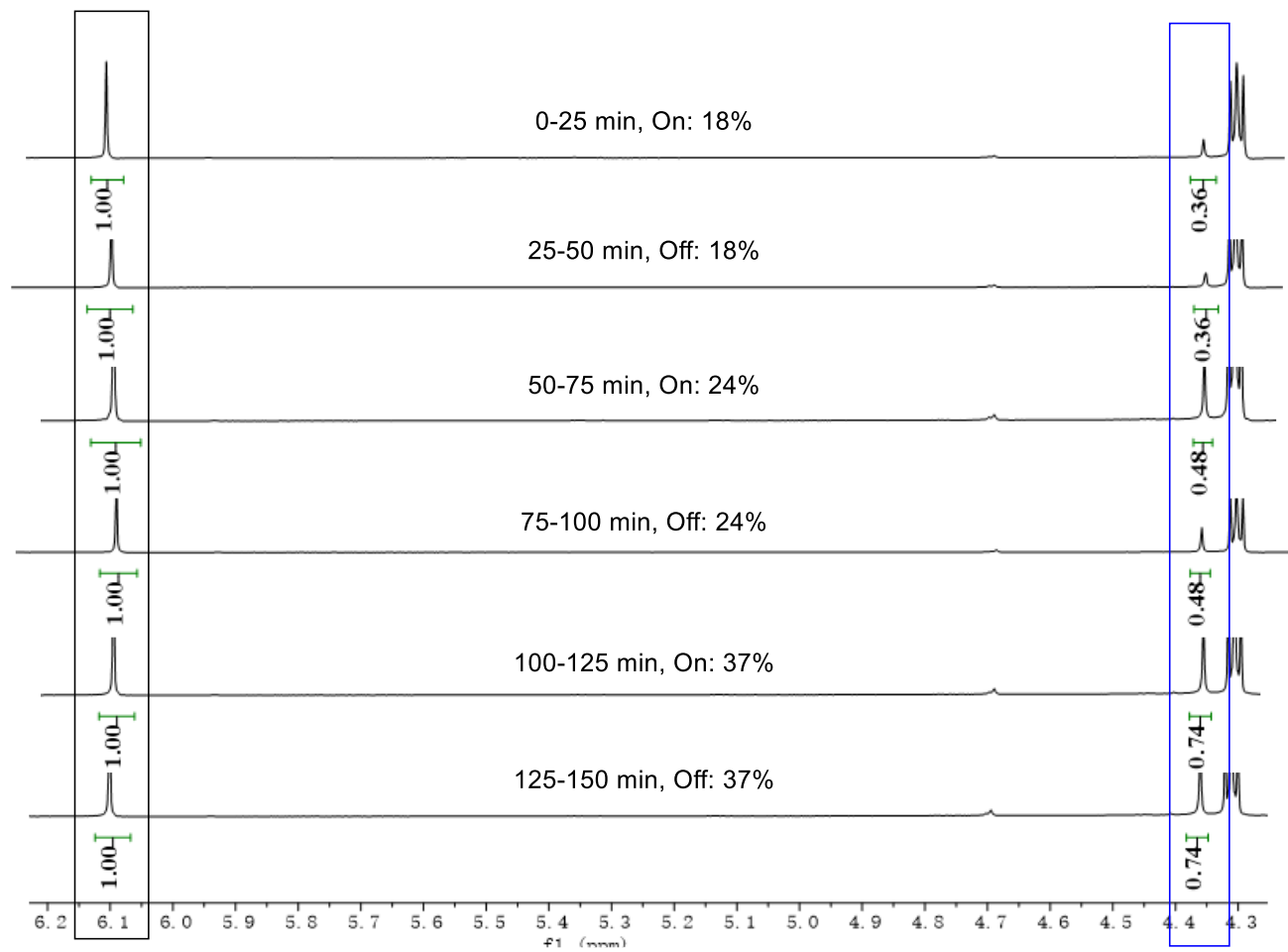
Figure S4. NMR experiments.



1,3,5-trimethoxybenzene
0.1 mmol



0.3 mmol



5.6. Calculation of quantum yield.

Determination of the light intensity at 390 nm:

Standard ferrioxalate actinometry was used to determine the photon flux of the LED lamp (40 W 390 nm).^[5-7] A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate trihydrate (646.5 mg, 1.5 mmol) in 0.20 M aqueous H₂SO₄ (10.0 mL). A buffered solution of 1,10-phenanthroline (0.15 M) was prepared by dissolving NaOAc (1.23 g, 15.0 mmol) and 1,10-phenanthroline (540.6 mg, 3.0 mmol) in 0.20 M aqueous H₂SO₄ (20 mL). To a 10 mL Schlenk tube was added the ferrioxalate solution (1.0 mL) and the tube was sealed and irradiated with a LED lamp (40 W 390 nm) for 300 s while maintaining the temperature at room temperature through cooling with a fan. The aqueous sulfuric acid (3.0 mL) and buffered solution (4.0 mL) were added immediately. The resulting mixture was then placed in the dark for 1 h to allow the formed ferrous ions to react completely with the 1,10-phenanthroline. An aliquot (25 μ L) of the resulting solution was diluted with 0.20 M aqueous sulfuric acid (3.0 mL), the solution was transferred to a cuvette ($l = 1.0$ cm) and the absorbance at a wavelength of 510 nm was measured by UV-vis spectrometry. The above procedure was repeated three times, and the average absorption was used for the calculation of the photon flux. A nonirradiated sample was also prepared and the absorbance at 510 nm was measured. The photon flux was calculated as follows:

$$\text{mol Fe}^{2+} = \frac{V \times \Delta A (510 \text{ nm}) \times 100}{l \times \epsilon} \quad (1)$$

Where V is the total volume (0.00325 L) of the solution that was analyzed, ΔA (0.48201) is the difference between the average absorption of irradiated and non-irradiated solutions at 510 nm, l is the path length (1.00 cm), and ϵ is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 L·mol⁻¹·cm⁻¹)^[5].

The photon flux was calculated as follows:

$$\text{photon flux} = \frac{\text{mol Fe}^{2+}}{\phi \times t \times f} \quad (2)$$

Where Φ is the quantum yield for the ferrioxalate actinometer (approximated as 0.783, which was the average value from the reported at $\lambda = 405$ nm and $\lambda = 385$ nm^[5-6]), t is the irradiation time (300 s), and f is the fraction of light absorbed at $\lambda = 390$ nm by the ferrioxalate actinometer. This value was calculated using the following equation where $A(390 \text{ nm})$ is the absorption of the ferrioxalate solution at 390 nm.

$$f = 1 - 10^{-A(390 \text{ nm})} = 0.23167 \quad (3)$$

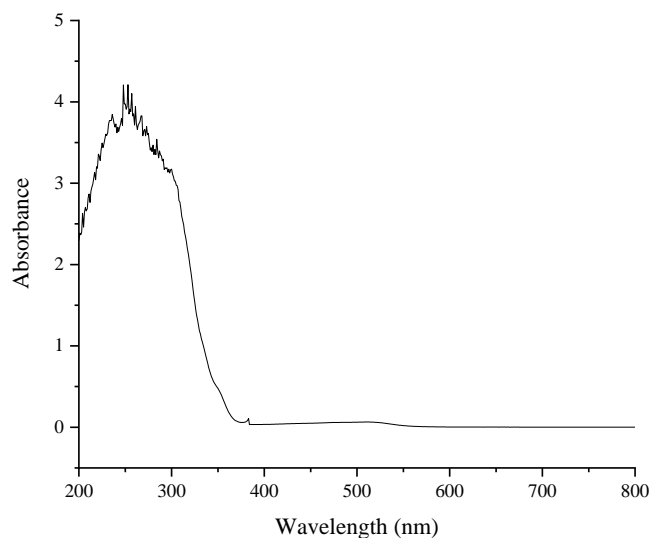


Figure S6. Absorbance of the ferrioxalate actinometer solution

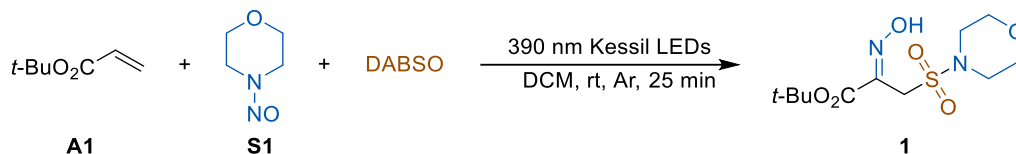
The average photon flux was thus calculated to be 2.06×10^{-7} einstein s^{-1} .

Sample calculation:

$$\text{mol Fe}^{2+} = \frac{V \times \Delta A(510 \text{ nm}) \times 100}{l \times \epsilon} = \frac{0.00325 \text{ L} \times 0.381121 \times 100}{1.000 \text{ cm} \times 11100 \text{ L mol}^{-1} \text{cm}^{-1}} = 1.12 \times 10^{-5} \text{ mol}$$

$$\text{photon flux} = \frac{\text{mol Fe}^{2+}}{\phi \times t \times f} = \frac{1.12 \times 10^{-5} \text{ mol}}{0.783 \times 300 \text{ s} \times 0.23167} = 2.06 \times 10^{-7} \text{ einstein s}^{-1}$$

Determination of quantum yield:



A1 (38.6 mg, 0.3 mmol), **S1** (52.5 mg, 0.45 mmol, 1.5 equiv.), DABSO (144.3 mg, 0.6 mmol, 2.0 equiv.) and internal standard (1,3,5-trimethoxybenzene, 0.1 mmol, 16.8 mg) were added into a dry 10 mL Schlenk tube equipped with a stir bar. The tube was evacuated and back filled with argon for three times, followed by the addition of DCM (3 mL) via syringe. The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (Kessil KSPR160–390 LED Grow Light; 5–6 cm away). The reaction mixture then taken at the 25 min and the yield was determined by ^1H NMR.

The quantum yield was determined using eq 4.

$$\phi = \frac{\text{mol product}}{\text{flux} \times t \times f} \quad (4)$$

Sample quantum yield calculation:

$$\phi = \frac{0.3 \times 10^{-3} \text{ mol} \times 0.18}{2.06 \times 10^{-7} \times 1500 \text{ s} \times 0.89731} = 0.15$$

7. The X-ray crystallographic data.

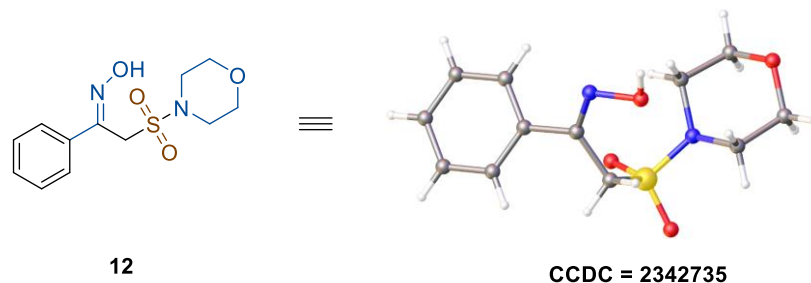


Figure 7. X-ray crystal structure of **12** (CCDC = 2342735).

To a 3-mL vial, the corresponding samples **12** (10.0 mg) were dissolved in CDCl_3 (2 mL). The vial was left uncovered and the sample was carefully setting in room temperature. The crystal was obtained by slow evaporation of **12** in CDCl_3 . Single crystal X-ray crystallography data was obtained on Bruker D8 venture.

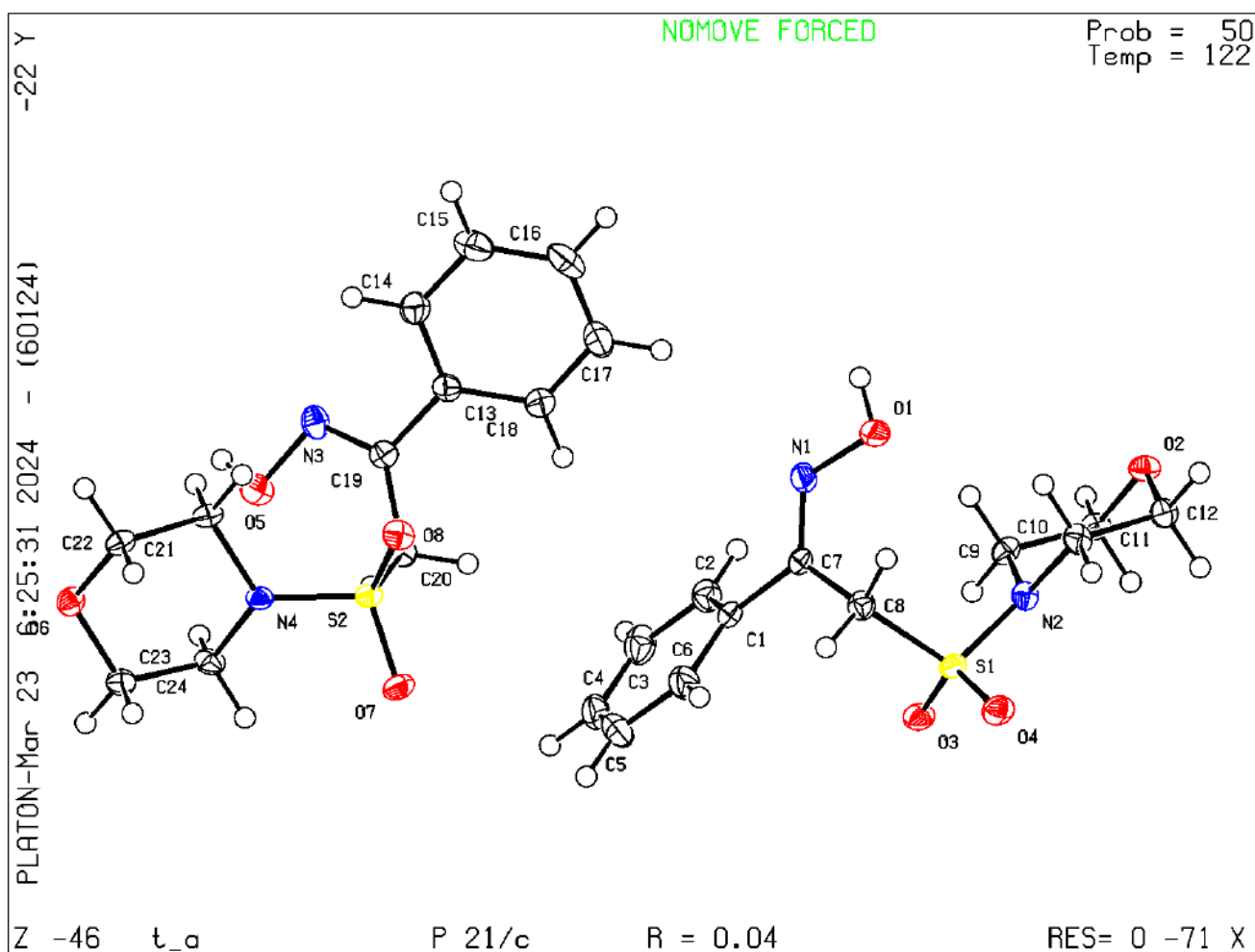
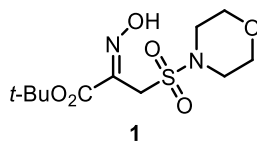


Table S1 Crystal data and structure refinement for 12.

Identification code	12
Empirical formula	C ₁₂ H ₁₆ N ₂ O ₄ S
Formula weight	284.33
Temperature/K	122(2)
Crystal system	monoclinic
Space group	P2 ₁ /c
<i>a</i> /Å	25.691(2)
<i>b</i> /Å	5.5958(5)
<i>c</i> /Å	19.8517(18)
α/°	90
β/°	112.73
γ/°	90
Volume/Å ³	2632.3(4)
<i>Z</i>	8
Density (calculated) mg/cm ³	1.435
Absorption coefficient mm ⁻¹	2.316
F(000)	1200
Crystal size/mm ³	0.180 × 0.160 × 0.140
2θ range for data collection/	1.864 to 68.916
Index ranges	-30 ≤ <i>h</i> ≤ 30, -6 ≤ <i>k</i> ≤ 6, -23 ≤ <i>l</i> ≤ 23
Reflections collected	33666
Independent reflections	4822 [R _{int} = 0.0416]
Data/restraints/parameters	4822/1/349
Goodness-of-fit on F ²	1.045
Final R indexes [<i>I</i> ≥ 2σ (<i>I</i>)]	R ₁ = 0.0358 wR ₂ = 0.0986
Final R indexes [all data]	R ₁ = 0.0409, wR ₂ = 0.1028
Largest diff. peak/hole / e Å ⁻³	0.771 and -0.490

8. Characterization data of new compounds.

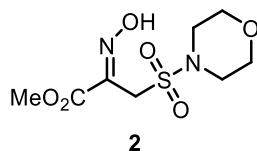


tert-butyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **1** (44.4 mg, 72% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 11.06 (s, 1H), 4.37 (s, 2H), 3.75–3.73 (m, 4H), 3.32–3.30 (m, 4H), 1.56 (s, 9H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 161.4, 143.6, 84.5, 66.8, 45.6, 44.0, 28.0 ppm.

HRMS (m/z): [M+Na]⁺ calcd for C₁₁H₂₀N₂NaO₆S⁺ 331.0934, found 331.0942.

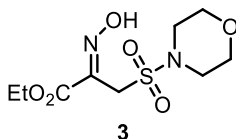


methyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **2** (37.3 mg, 70% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 11.11 (s, 1H), 4.42 (s, 2H), 3.93 (s, 3H), 3.77–3.75 (m, 4H), 3.33–3.31 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 163.1, 142.6, 66.8, 53.7, 45.5, 44.0 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₈H₁₅N₂O₆S⁺ 267.0645, found 267.0650.

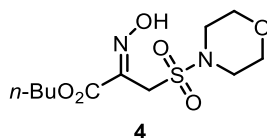


ethyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **3** (44.3 mg, 79% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 11.12 (s, 1H), 4.41–4.37 (m, 4H), 3.77–3.75 (m, 4H), 3.33–3.31 (m, 4H), 1.37 (t, *J* = 7.1 Hz, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.6, 142.8, 66.8, 63.0, 45.5, 44.0, 14.2 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₉H₁₇N₂O₆S⁺ 281.0802, found 281.0805.

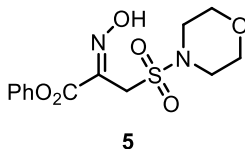


butyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **4** (42.5 mg, 69% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 10.98 (brs, 1H), 4.41 (s, 2H), 4.33 (t, *J* = 6.8 Hz, 2H), 3.77–3.75 (m, 4H), 3.33–3.31 (m, 4H), 1.75–1.69 (m, 2H), 1.45–1.38 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.7, 142.8, 66.9, 45.5, 44.0, 30.5, 19.1, 13.8 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₁H₂₁N₂O₆S⁺ 309.1115, found 309.1117.

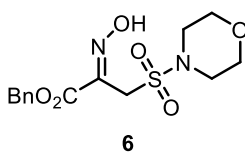


phenyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **5** (42.6 mg, 65% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, CDCl₃): δ = 11.14 (brs, 1H), 7.43–7.40 (m, 2H), 7.30–7.27 (m, 1H), 7.21–7.19 (m, 2H), 4.51 (s, 2H), 3.77–3.75 (m, 4H), 3.36–3.34 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 161.4, 150.3, 142.7, 129.8, 126.8, 121.5, 66.8, 45.7, 44.2 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₃H₁₇N₂O₆S⁺ 329.0802, found 329.0810.

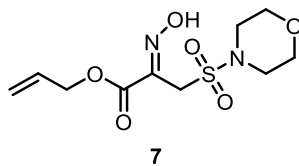


benzyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **6** (52.7 mg, 77% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 11.16 (brs, 1H), 7.41–7.33 (m, 5H), 5.33 (s, 2H), 4.38 (s, 2H), 3.70–3.69 (m, 4H), 3.26–3.24 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.5, 142.5, 134.7, 128.9, 128.8, 128.8, 68.5, 66.7, 45.5, 44.0 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₆S⁺ 343.0958, found 343.0962.

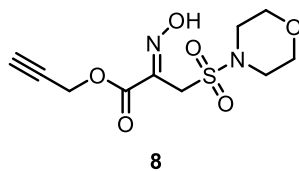


allyl (Z)-2-(hydroxyimino)-3-(morpholinylsulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **7** (36.2 mg, 62% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 10.94 (s, 1H), 6.01–5.94 (m, 1H), 5.41 (dd, *J* = 17.2, 1.5 Hz, 1H), 5.31 (dd, *J* = 10.4, 1.3 Hz, 1H), 4.82 (d, *J* = 5.9 Hz, 2H), 4.42 (s, 2H), 3.77–3.75 (m, 4H), 3.33–3.31 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.3, 142.7, 131.0, 120.0, 67.4, 66.8, 45.5, 44.0 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₀H₁₇N₂O₆S⁺ 293.0802, found 293.0807.

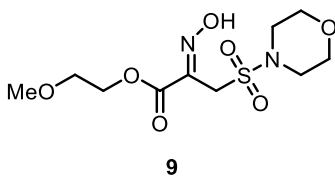


prop-2-yn-1-yl (Z)-2-(hydroxyimino)-3-(morpholinylsulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **8** (29.0 mg, 50% yield) was obtained as a white powder.

¹H NMR (500 MHz, CD₃CN): δ = 10.95 (brs, 1H), 4.85 (d, *J* = 2.4 Hz, 2H), 4.33 (s, 2H), 3.65–3.63 (m, 4H), 3.19–3.17 (m, 4H), 2.85 (t, *J* = 2.4 Hz, 1H) ppm.

¹³C NMR (125 MHz, CD₃CN): δ = 162.9, 142.9, 78.1, 77.0, 67.0, 54.1, 46.4, 44.3 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₀H₁₅N₂O₆S⁺ 291.0645, found 291.0644.

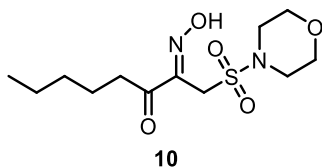


methyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **9** (45.9 mg, 74% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 11.50 (brs, 1H), 4.47–4.45 (m, 2H), 4.41 (s, 2H), 3.74–3.72 (m, 4H), 3.70–3.68 (m, 2H), 3.40 (s, 3H), 3.30–3.29 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.6, 142.3, 70.1, 66.8, 65.2, 59.0, 45.5, 44.0 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₀H₁₉N₂O₇S⁺ 311.0907, found 311.0910.

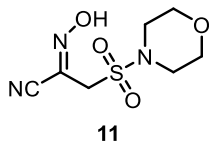


(Z)-2-(hydroxyimino)-1-(morpholinosulfonyl)octan-3-one: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **10** (39.2 mg, 64% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.97 (s, 1H), 4.38 (s, 2H), 3.79–3.77 (m, 4H), 3.33–3.31 (m, 4H), 2.81 (t, *J* = 7.5 Hz, 2H), 1.66–1.61 (m, 2H), 1.30–1.28 (m, 4H), 0.88 (t, *J* = 6.7 Hz, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 197.5, 148.7, 66.8, 45.6, 41.6, 37.5, 31.5, 23.7, 22.6, 14.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₂H₂₃N₂O₅S⁺ 307.1322, found 307.1325.

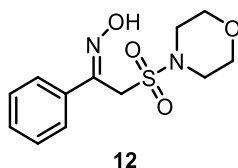


(Z)-N-hydroxy-2-(morpholinylsulfonyl)acetimidoyl cyanide: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/2, v/v). The product **11** (19.1 mg, 41% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 7.11 (s, 1H), 5.73 (s, 1H), 4.13–4.11 (m, 2H), 3.76–3.71 (m, 4H), 3.68–3.66 (m, 2H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 163.1, 160.2, 67.3, 66.8, 47.2, 43.6 ppm.

HRMS (*m/z*): [M+Na]⁺ calcd for C₇H₁₁N₃NaO₄S⁺ 256.0362, found 256.0370.



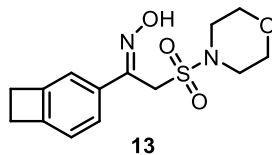
(Z)-2-(morpholinylsulfonyl)-1-phenylethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **12** (51.1 mg, 90% yield) was obtained as a white solid.

¹H NMR (500 MHz, CDCl₃): δ = 9.51 (s, 1H), 7.75–7.73 (m, 2H), 7.43–7.41 (m, 3H), 4.58 (s, 2H), 3.72–3.70 (m, 4H), 3.34–3.32 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 148.7, 133.8, 130.2, 128.8, 126.7, 66.9, 45.9, 45.6 ppm.

HRMS (*m/z*): [M+Na]⁺ calcd for C₁₂H₁₆N₂NaO₄S⁺ 307.0723, found 307.0730.

m.p. : 115.9–127.8 °C.

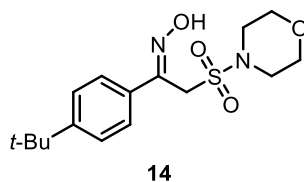


(Z)-1-(bicyclo[4.2.0]octa-1(6),2,4-trien-3-yl)-2-(morpholinylsulfonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **13** (47.7 mg, 77% yield, *Z/E* = 1.3:1) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 7.55–7.07 (m, 3H), 4.57 and 4.21 (s, 2H), 3.70–3.66 (m, 4H), 3.33–3.29 (m, 4H), 3.20–3.18 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 149.5, 149.4, 148.4, 148.1, 146.3, 145.9, 132.7, 130.0, 126.9, 125.7, 122.9, 122.7, 122.6, 120.9, 66.9, 66.8, 55.8, 46.2, 46.0, 45.6, 29.9, 29.8, 29.7, 29.6 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₄S⁺ 311.1060, found 311.1070.

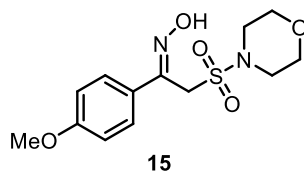


(Z)-1-(4-(tert-butyl)phenyl)-2-(morpholinylsulfonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **14** (61.9 mg, 91% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.53 (s, 1H), 7.69 (d, *J* = 8.6 Hz, 2H), 7.43 (d, *J* = 8.6 Hz, 2H), 4.58 (s, 2H), 3.72–3.70 (m, 4H), 3.35–3.34 (m, 4H), 1.32 (s, 9H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 153.5, 148.5, 130.9, 126.4, 125.8, 66.9, 45.9, 45.6, 34.9, 31.3 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₆H₂₅N₂O₄S⁺ 341.1530, found 341.1545.

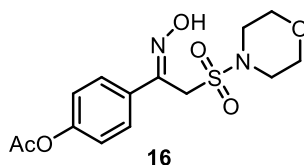


(Z)-1-(4-methoxyphenyl)-2-(morpholinylsulfonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **15** (47.7 mg, 76% yield) was obtained as a white powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.05 (s, 1H), 7.70–7.68 (m, 2H), 6.97–6.95 (m, 2H), 4.66 (s, 2H), 3.78 (s, 3H), 3.59–3.57 (m, 4H), 3.17–3.15 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 160.1, 146.1, 127.9, 127.3, 113.7, 65.9, 55.2, 45.3, 44.0 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₃H₁₉N₂O₅S⁺ 315.1009, found 315.1011.

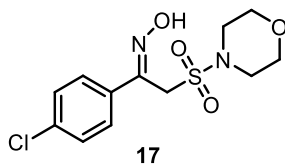


(Z)-4-(1-(hydroxyimino)-2-(morpholinylsulfonyl)ethyl)phenyl acetate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **16** (57.5 mg, 84% yield) was obtained as a white powder.

¹H NMR (500 MHz, CD₃CN): δ = 9.94 (s, 1H), 7.76 (d, *J* = 8.6 Hz, 2H), 7.14 (d, *J* = 8.6 Hz, 2H), 4.57 (s, 2H), 3.63–3.61 (m, 4H), 3.23–3.21 (m, 4H), 2.26 (s, 3H) ppm.

¹³C NMR (125 MHz, CD₃CN): δ = 170.3, 152.8, 148.0, 133.2, 128.7, 122.8, 67.1, 46.4, 45.1, 21.2 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₆S⁺ 343.0958, found 343.0977.

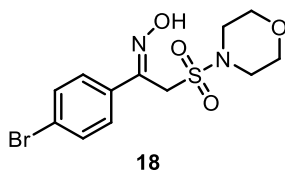


(Z)-1-(4-chlorophenyl)-2-(morpholinosulfonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **17** (56.6 mg, 89% yield) was obtained as a white powder.

¹H NMR (500 MHz, CD₃CN): δ = 10.02 (s, 1H), 7.69 (d, J = 8.6 Hz, 2H), 7.40 (d, J = 8.6 Hz, 2H), 4.53 (s, 2H), 3.61–3.59 (m, 4H), 3.20–3.18 (m, 4H) ppm.

¹³C NMR (125 MHz, CD₃CN): δ = 147.8, 135.8, 134.4, 129.4, 129.1, 67.1, 46.4, 45.1 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₂H₁₆ClN₂O₄S⁺ 319.0514, found 319.0520.

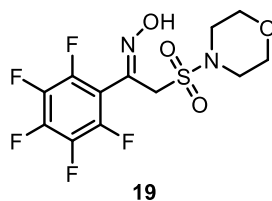


(Z)-1-(4-bromophenyl)-2-(morpholinosulfonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **18** (54.3 mg, 75% yield) was obtained as a white powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.43 (s, 1H), 7.71–7.69 (m, 2H), 7.62–7.60 (m, 2H), 4.70 (s, 2H), 3.59–3.57 (m, 4H), 3.17–3.15 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 145.9, 134.1, 131.3, 128.5, 122.6, 65.9, 45.3, 43.8 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₂H₁₆BrN₂O₄S⁺ 363.0009, found 363.0007.



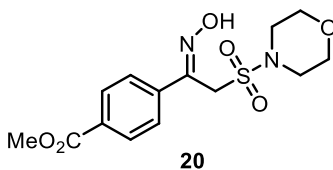
(Z)-2-(morpholinosulfonyl)-1-(perfluorophenyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **19** (69.6 mg, 93% yield, *Z/E* = 1.1:1) was obtained as a white powder.

¹H NMR (500 MHz, CD₃OD): δ = 4.51 and 4.31 (s, 2H), 3.71–3.36 (m, 4H), 3.35–3.31 (m, 4H) ppm.

¹³C NMR (125 MHz, CD₃OD): δ = 147.5, 145.8, 145.4, 144.0, 143.8, 142.1, 140.0, 139.8, 138.6, 138.0, 137.9, 137.0, 112.5, 109.3, 67.7, 54.6, 47.2, 46.7 ppm.

¹⁹F NMR (470 MHz, CD₃OD) δ = -138.7 – -138.8 (m), -143.3 – -143.4 (m), -155.6 – -155.9 (m), -164.8 – -165.1 (m) ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₂H₁₂F₅N₂O₄S⁺ 375.0432, found 375.0430.

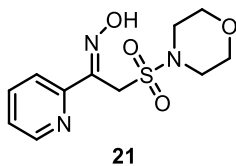


methyl (Z)-4-(1-(hydroxyimino)-2-(morpholinosulfonyl)ethyl)benzoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **20** (57.5 mg, 84% yield) was obtained as a white powder.

¹H NMR (500 MHz, CD₃CN): δ = 10.22 (s, 1H), 8.02 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 8.4 Hz, 2H), 4.60 (s, 2H), 3.87 (s, 3H), 3.63–3.61 (m, 4H), 3.28–3.20 (m, 4H) ppm.

¹³C NMR (125 MHz, CD₃CN): δ = 167.1, 148.2, 139.8, 131.8, 130.2, 127.7, 67.1, 52.8, 46.4, 45.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₆S⁺ 343.0958, found 343.0971.

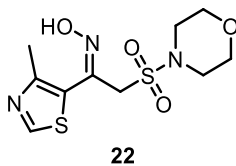


(Z)-2-(morpholinosulfonyl)-1-(pyridin-2-yl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **21** (47.9 mg, 84% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 10.92 (s, 1H), 8.65 (d, J = 4.5 Hz, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.76–7.73 (m, 1H), 7.33 (m, 1H), 4.89 (s, 2H), 3.72–3.70 (m, 4H), 3.34–3.32 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 152.1, 149.0, 148.6, 137.1, 124.5, 121.5, 66.9, 45.5, 43.9 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₁H₁₆N₃O₄S⁺ 286.0856, found 286.0850.

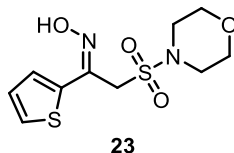


methyl (Z)-2-(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **22** (48.8 mg, 80% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.26 (s, 1H), 9.09 (s, 1H), 4.39 (s, 2H), 3.57–3.56 (m, 4H), 3.15–3.14 (m, 4H), 2.36 (s, 3H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 154.5, 154.5, 152.5, 139.8, 121.1, 65.8, 54.4, 45.4, 17.2 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₀H₁₆N₃O₄S₂⁺ 306.0577, found 306.0580.

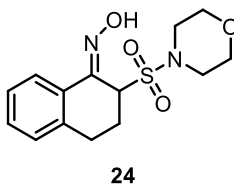


(Z)-2-(morpholinosulfonyl)-1-(thiophen-2-yl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **23** (41.8 mg, 72% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.53 (s, 1H), 7.78 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.70 (dd, *J* = 3.9, 1.0 Hz, 1H), 7.17 (dd, *J* = 5.0, 3.9 Hz, 1H), 4.58 (s, 2H), 3.60–3.58 (m, 4H), 3.21–3.19 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 140.6, 131.2, 130.8, 130.6, 125.7, 66.0, 52.7, 45.7 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₀H₁₅N₂O₄S₂⁺ 291.0468, found 291.0478.

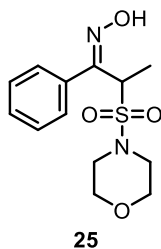


(Z)-2-(morpholinosulfonyl)-3,4-dihydronaphthalen-1(2H)-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **24** (50.8 mg, 82% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.33 (s, 1H), 7.85 (d, *J* = 7.9 Hz, 1H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.22–7.17 (m, 2H), 5.06 (dd, *J* = 5.1, 2.4 Hz, 1H), 3.72 (t, *J* = 4.8 Hz, 4H), 3.50–3.34 (m, 5H), 2.86–2.72 (m, 2H), 2.24–2.16 (m, 1H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 149.5, 138.1, 130.2, 129.3, 128.8, 126.5, 124.3, 67.1, 51.7, 45.5, 25.4, 24.4 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₄S⁺ 311.1060, found 311.1072.

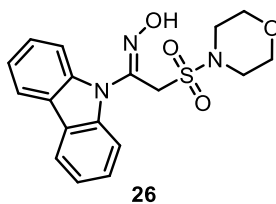


(Z)-2-(morpholinofonyl)-1-phenylpropan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **25** (37.5 mg, 63% yield, *Z/E* = 1.1:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.68 and 8.94 (s, 1H), 7.74–7.72 (m, 1H), 7.49–7.37 (m, 4H), 5.25 and 4.31 (q, *J* = 7.2, 1H), 3.74–3.67 (m, 4H), 3.48–3.34 (m, 4H), 1.63 and 1.54 (d, *J* = 7.2, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 154.5, 153.4, 133.2, 131.8, 129.6, 129.6, 128.7, 128.5, 128.5, 128.3, 67.1, 62.4, 53.1, 46.6, 46.0, 14.7, 14.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₃H₁₉N₂O₄S⁺ 299.1060, found 299.1061.

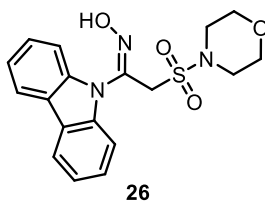


(E)-1-(9H-carbazol-9-yl)-2-(morpholinofonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **26** (67.1 mg, 90% yield, *Z/E* = 1:1) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.01 (s, 1H), 8.08 (d, *J* = 7.7 Hz, 2H), 7.49–7.42 (m, 4H), 7.32 (t, *J* = 7.4 Hz, 2H), 4.77 (s, 2H), 3.17–3.15 (m, 4H), 2.83–2.81 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 143.5, 139.5, 126.7, 124.3, 121.6, 120.8, 110.7, 66.0, 45.3 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₈H₂₀N₃O₄S⁺ 374.1169, found 374.1170.

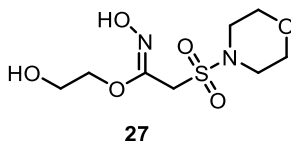


(Z)-1-(9H-carbazol-9-yl)-2-(morpholinosulfonyl)ethan-1-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **26** (67.1 mg, 90% yield, Z/E = 1:1) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.17 (s, 1H), 8.08 (d, *J* = 7.7 Hz, 2H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.34–7.26 (m, 4H), 4.50 (s, 2H), 3.03 (t, *J* = 4.5 Hz, 4H), 2.73 (t, *J* = 4.5 Hz, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 138.5, 138.2, 126.6, 124.2, 121.5, 120.7, 111.8, 65.9, 50.9, 45.4 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₈H₂₀N₃O₄S⁺ 374.1169, found 374.1170.

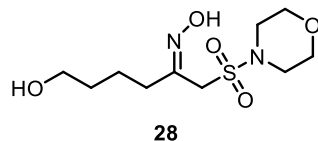


2-hydroxyethyl (Z)-N-hydroxy-2-(morpholinosulfonyl)acetimidate: This compound was prepared by using the general procedure (eluent: DCM/MeOH, 40/1, v/v). The product **27** (23.6 mg, 44% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 8.11 (s, 1H), 4.20–4.18 (m, 2H), 4.15 (s, 2H), 3.85–3.83 (m, 2H), 3.75–3.73 (m, 4H), 3.36–3.34 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 155.2, 69.6, 66.8, 60.9, 46.0, 45.6 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₈H₁₇N₂O₆S⁺ 269.0802, found 269.0820.

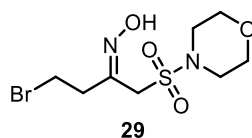


(Z)-6-hydroxy-1-(morpholinosulfonyl)hexan-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/3, v/v). The product **28** (25.2 mg, 45% yield, *Z/E* = 1.3:1) was obtained as a white powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 11.42 and 11.21 (s, 1H), 4.41–4.38 (m, 1H), 4.13 and 3.97 (s, 2H), 3.62–3.60 (m, 4H), 3.40–3.36 (m, 2H), 3.20–3.15 (m, 4H), 2.40–2.34 (m, 2H), 1.56–1.36 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 150.6, 147.9, 65.9, 60.5, 52.6, 45.7, 45.3, 44.9, 33.0, 32.6, 32.1, 27.2, 22.0, 21.3 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₀H₂₁N₂O₅S⁺ 281.1166, found 281.1171.

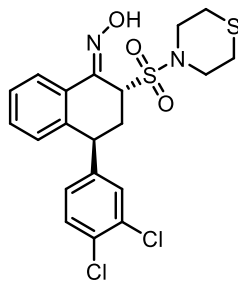


(Z)-4-bromo-1-(morpholinosulfonyl)butan-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **29** (25.7 mg, 41% yield, *Z/E* = 4.4:1) was obtained as a yellow powder.

¹H NMR (500 MHz, CDCl₃): δ = 9.22 (s, 1H), 4.42–4.38 (m, 2H), 4.08 and 4.06 (s, 2H), 3.75–3.69 (m, 4H), 3.33–3.28 (m, 4H), 3.17–3.03 (m, 2H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 150.1, 147.6, 69.9, 66.6, 66.5, 48.2, 46.1, 45.9, 45.5, 36.7, 36.5, 28.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₈H₁₆BrN₂O₄S⁺ 315.0009, found 315.0015.



30

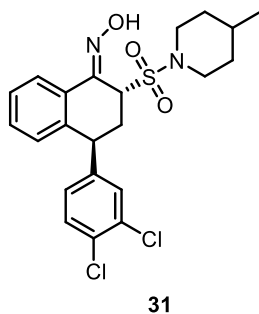
(2*R*,4*S*,*Z*)-4-(3,4-dichlorophenyl)-2-(thiomorpholinofonyl)-3,4-dihydronaphthalen-1(2*H*)-one

oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **30** (69.6 mg, 74% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.41 (s, 1H), 7.89–7.87 (m, 1H), 7.57 (d, *J* = 8.3 Hz, 1H), 7.51 (d, *J* = 2.1 Hz, 1H), 7.24–7.19 (m, 3H), 6.68–6.67 (m, 1H), 5.10 (dd, *J* = 5.1, 2.5 Hz, 1H), 4.52 (dd, *J* = 12.3, 5.5 Hz, 1H), 3.46–3.45 (m, 4H), 2.61–2.57 (m, 5H), 2.39–2.33 (m, 1H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.2, 146.0, 139.4, 131.3, 130.9, 130.1, 129.5, 129.3, 129.2, 126.7, 123.8, 51.7, 47.1, 40.8, 32.5, 27.2 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₀H₂₁Cl₂N₂O₃S₂⁺ 471.0365, found 471.0370.



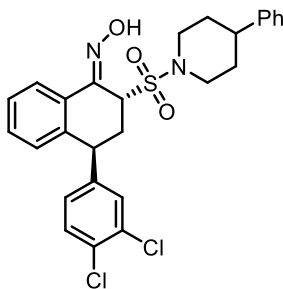
(2*R*,4*S*,*Z*)-4-(3,4-dichlorophenyl)-2-((4-methylpiperidin-1-yl)sulfonyl)-3,4-dihydronaphthalen-

1(2*H*)-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **31** (73.6 mg, 79% yield, dr = 1.8:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.32 and 11.92 (s, 1H), 8.40–6.66 (m, 7H), 5.10–4.32 (m, 2H), 3.63–3.42 (m, 2H), 3.00–2.79 (m, 2H), 2.61–2.31 (m, 2H), 1.66–1.56 (m, 2H), 1.45–1.39 (m, 1H), 1.12–1.00 (m, 2H), 0.90–0.88 (m, 3H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.8, 146.7, 145.9, 144.3, 140.5, 139.8, 131.8, 131.6, 131.3, 131.3, 131.1, 130.7, 130.2, 129.9, 129.8, 129.7, 129.6, 129.6, 129.5, 129.1, 128.1, 127.1, 126.8, 124.2, 61.0, 51.7, 46.8, 45.9, 45.6, 45.3, 42.0, 41.2, 34.3, 34.1, 33.9, 33.9, 33.1, 32.1, 30.4, 30.2, 21.9, 21.8 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₂H₂₅Cl₂N₂O₃S⁺ 467.0957, found 467.0966.



32

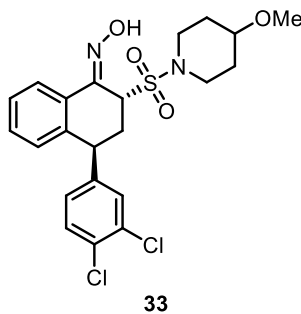
(2R,4S,Z)-4-(3,4-dichlorophenyl)-2-((4-phenylpiperidin-1-yl)sulfonyl)-3,4-dihydronaphthalen-

1(2H)-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **32** (81.3 mg, 77% yield, dr = 4.5:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.38 and 11.99 (s, 1H), 8.44–6.68 (m, 12H), 5.17–3.56 (m, 4H), 3.18–2.93 (m, 2H), 2.65–2.33 (m, 3H), 1.86–1.75 (m, 2H), 1.65–1.49 (m, 2H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.4, 146.3, 145.5, 145.3, 145.3, 143.9, 140.1, 139.4, 131.3, 131.2, 130.8, 130.8, 130.7, 130.3, 130.3, 129.8, 129.5, 129.3, 129.3, 129.2, 129.1, 129.0, 128.7, 128.4, 127.7, 126.8, 126.7, 126.7, 126.3, 123.8, 66.4, 60.8, 51.5, 48.6, 46.9, 45.9, 45.8, 45.4, 41.5, 41.5, 41.3, 40.8, 33.2, 32.9, 32.7, 32.7, 31.6 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₇H₂₇Cl₂N₂O₃S⁺ 529.1114, found 529.1115.



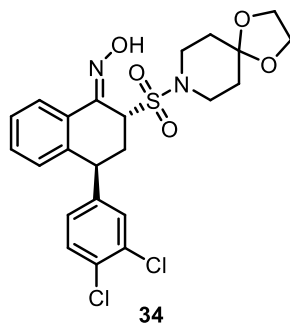
(2*R*,4*S*,*Z*)-4-(3,4-dichlorophenyl)-2-((4-methoxypiperidin-1-yl)sulfonyl)-3,4-dihydronaphthalen-

1(2*H*)-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **33** (68.4 mg, 71% yield, dr = 1.3:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.31 and 11.94 (s, 1H), 8.40–6.66 (m, 7H), 5.12–4.32 (m, 2H), 3.47–3.05 (m, 8H), 2.61–2.31 (m, 2H), 1.83–1.78 (m, 2H), 1.48–1.42 (m, 2H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.3, 146.2, 145.4, 143.9, 140.1, 139.4, 131.3, 131.2, 130.9, 130.8, 130.7, 130.2, 129.8, 129.5, 129.3, 129.2, 129.1, 129.0, 128.7, 127.6, 126.7, 126.3, 123.8, 74.3, 66.4, 60.7, 55.1, 54.9, 51.5, 43.2, 43.0, 42.5, 42.4, 41.5, 40.7, 34.2, 33.9, 32.6, 31.6, 30.4, 30.3 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₂H₂₅Cl₂N₂O₄S⁺ 483.0907, found 483.0915.



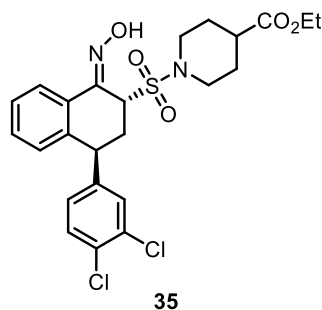
(2*R*,4*S*,*Z*)-2-((1,4-dioxo-8-azaspiro[4.5]decan-8-yl)sulfonyl)-4-(3,4-dichlorophenyl)-3,4-

dihydronaphthalen-1(2*H*)-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **34** (61.2 mg, 60% yield, dr = 3.4:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.34 and 11.97 (s, 1H), 8.39–6.66 (m, 7H), 5.13–4.34 (m, 2H), 3.89 (s, 4H), 3.37–3.26 (m, 4H), 2.61–2.32 (m, 2H), 1.64–1.60 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.3, 146.1, 145.5, 143.9, 140.0, 139.3, 131.3, 131.2, 130.9, 130.8, 130.7, 130.3, 130.2, 129.8, 129.5, 129.3, 129.3, 129.2, 129.2, 129.1, 129.0, 128.7, 127.6, 126.7, 126.3, 123.8, 105.8, 63.8, 61.0, 51.8, 44.1, 43.4, 41.5, 40.7, 34.9, 34.9, 32.6, 31.7 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₃H₂₅Cl₂N₂O₅S⁺ 511.0856, found 511.0859.

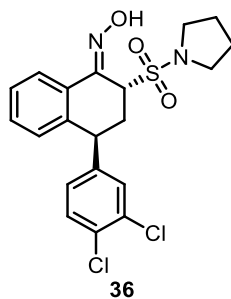


ethyl 1-(((2R,4S,Z)-4-(3,4-dichlorophenyl)-1-(hydroxyimino)-1,2,3,4-tetrahydronaphthalen-2-yl)sulfonyl)piperidine-4-carboxylate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **35** (87.0 mg, 83% yield, dr = 1:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.30 and 11.93 (s, 1H), 8.40–6.67 (m, 7H), 5.13–4.33 (m, 2H), 4.07 (q, *J* = 7.1 Hz, 2H), 3.62–3.43 (m, 2H), 3.17–2.32 (m, 5H), 1.90–1.82 (m, 2H), 1.57–1.43 (m, 2H), 1.18 (t, *J* = 7.1 Hz, 3H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 173.6, 146.3, 145.4, 143.8, 140.1, 139.4, 131.3, 131.2, 130.9, 130.7, 130.3, 130.3, 130.2, 129.8, 129.5, 129.3, 129.3, 129.3, 129.1, 129.0, 128.7, 127.6, 126.7, 126.4, 123.8, 60.7, 60.1, 51.5, 48.6, 45.4, 44.7, 44.4, 44.1, 41.5, 40.7, 32.6, 31.5, 28.1, 27.9, 27.8, 27.8, 14.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₄H₂₇Cl₂N₂O₅S⁺ 525.1012, found 525.1015.



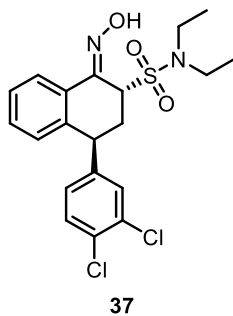
(2*R*,4*S*,*Z*)-4-(3,4-dichlorophenyl)-2-(pyrrolidin-1-ylsulfonyl)-3,4-dihydronaphthalen-1(2*H*)-one

oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **36** (56.9 mg, 65% yield, dr = 6.7:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.25 and 11.89 (s, 1H), 8.42–6.66 (m, 7H), 5.26–4.17 (m, 2H), 3.35–3.18 (m, 4H), 2.61–2.56 (m, 1H), 2.38–2.31 (m, 1H), 1.88–1.82 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.8, 146.3, 145.3, 144.0, 140.2, 139.5, 131.4, 131.2, 130.9, 130.8, 130.7, 130.3, 130.2, 129.8, 129.5, 129.3, 129.2, 129.2, 129.0, 128.7, 127.8, 126.7, 126.4, 123.7, 58.8, 50.4, 49.3, 48.1, 47.9, 47.9, 45.2, 41.6, 40.8, 32.7, 31.2, 25.4, 23.5, 22.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₀H₂₁Cl₂N₂O₃S⁺ 439.0644, found 439.0654.



(2*R*,4*S*,*Z*)-4-(3,4-dichlorophenyl)-*N,N*-diethyl-1-(hydroxyimino)-1,2,3,4-tetrahydronaphthalene-2-

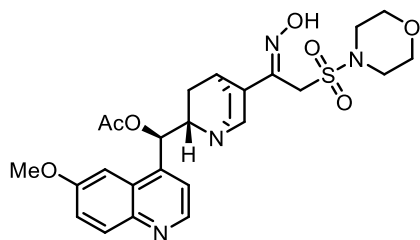
sulfonamide: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v).

The product **37** (47.5 mg, 54% yield, dr = 1.3:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.15 and 11.88 (s, 1H), 8.39–6.66 (m, 7H), 5.21–4.11 (m, 2H), 3.20–3.16 (m, 4H), 2.60–2.31 (m, 2H), 1.12–1.08 (m, 6H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.4, 146.3, 145.4, 143.8, 140.1, 139.5, 131.3, 131.2, 130.9, 130.8, 130.7, 130.3, 130.3, 129.7, 129.5, 129.4, 129.2, 129.2, 129.1, 129.0, 128.7, 127.7, 126.7, 126.4, 123.7, 60.6, 51.7, 43.5, 42.9, 41.6, 40.8, 32.6, 31.4, 15.8, 15.3 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₀H₂₃Cl₂N₂O₃S⁺ 441.0801, found 441.0795.



38

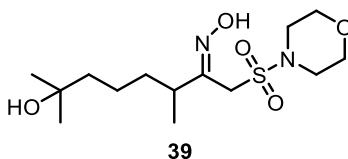
(1*R*)-((1*S*,2*S*,4*S*)-5-((*Z*)-1-(hydroxyimino)-2-(morpholinyl)ethyl)quinuclidin-2-yl)(6-

methoxyquinolin-4-yl)methyl acetate: This compound was prepared by using the general procedure (eluent: DCM/MeOH, 30/1, v/v). The product **38** (54.6 mg, 50% yield) was obtained as a white powder.

¹H NMR (500 MHz, CD₃CN): δ = 11.72 (s, 1H), 8.61 (d, J = 4.5 Hz, 1H), 7.96 (d, J = 9.2 Hz, 1H), 7.49 (d, J = 2.8 Hz, 1H), 7.35–7.32 (m, 2H), 6.36 (d, J = 8.0 Hz, 1H), 4.37 (d, J = 13.6 Hz, 1H), 3.90 (s, 3H), 3.74 (d, J = 13.6 Hz, 1H), 3.62–3.52 (m, 5H), 3.25–3.12 (m, 6H), 2.82–2.72 (m, 2H), 2.63–2.58 (m, 1H), 2.21–2.16 (m, 1H), 2.05 (s, 3H), 1.81–1.74 (m, 2H), 1.57–1.49 (m, 2H) ppm.

¹³C NMR (125 MHz, CD₃CN): δ = 171.0, 158.8, 150.8, 148.2, 146.4, 144.7, 131.8, 128.2, 122.9, 119.8, 102.7, 74.0, 67.1, 59.8, 56.4, 52.9, 46.3, 45.9, 43.2, 40.9, 27.9, 25.7, 25.4, 21.1 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₂₆H₃₅N₄O₇S⁺ 547.2221, found 547.2230.

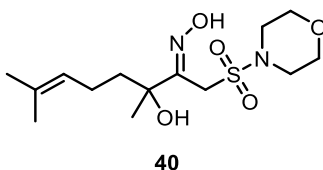


(Z)-7-hydroxy-3,7-dimethyl-1-(morpholinosulfonyl)octan-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/2, v/v). The product **39** (20.8 mg, 31% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 9.16 (s, 1H), 4.13–4.04 (m, 2H), 3.74–3.72 (m, 4H), 3.34–3.32 (m, 4H), 2.80–2.76 (m, 1H), 1.46–1.38 (m, 6H), 1.19 (s, 6H), 1.14 (d, J = 6.9 Hz, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 154.3, 71.2, 66.8, 45.8, 45.6, 43.6, 38.0, 34.6, 29.5, 29.3, 21.7, 18.4 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₄H₂₉N₂O₅S⁺ 337.1792, found 337.1790.

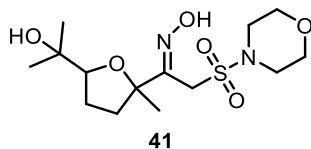


(Z)-3-hydroxy-3,7-dimethyl-1-(morpholinosulfonyl)oct-6-en-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **40** (19.4 mg, 29% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 4.95–4.94 (m, 1H), 4.74 (s, 1H), 3.76 (t, J = 4.7 Hz, 4H), 3.27–3.20 (m, 5H), 2.95 (s, 1H), 2.69–2.62 (m, 2H), 2.54–2.50 (m, 1H), 1.89–1.85 (m, 1H), 1.76 (s, 3H), 1.69–1.63 (m, 3H), 1.48 (s, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 144.8, 112.7, 79.1, 66.5, 50.6, 46.0, 45.5, 44.2, 39.6, 32.1, 26.8, 23.0 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₄H₂₇N₂O₅S⁺ 335.1635, found 335.1640.



(Z)-1-(5-(2-hydroxypropan-2-yl)-2-methyltetrahydrofuran-2-yl)-2-(morpholinosulfonyl)ethan-1-

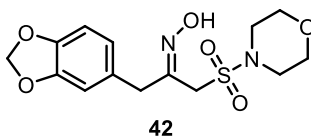
one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/2, v/v).

The product **41** (25.9 mg, 37% yield, *Z/E* = 1.2:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, CDCl₃): δ = 10.94 and 9.67 (brs, 1H), 4.24–3.29 (m, 12H), 2.44–2.30 (m, 1H), 2.02–1.81 (m, 3H), 1.48–1.08 (m, 9H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 154.4, 153.7, 87.0, 86.2, 84.0, 83.9, 71.6, 71.1, 66.7, 66.0, 45.7, 45.6, 45.2, 44.0, 37.6, 36.8, 28.3, 27.3, 27.2, 26.4, 26.2, 25.5, 25.1, 24.1 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₂₇N₂O₆S⁺ 351.1584, found 351.1592.

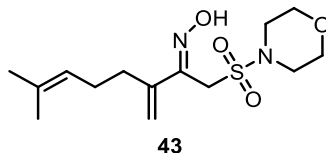


(Z)-1-(benzo[*d*][1,3]dioxol-5-yl)-3-(morpholinosulfonyl)propan-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **42** (39.0 mg, 57% yield, *Z/E* = 2:1) was obtained as a white powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 11.68 and 11.58 (s, 1H), 6.86–6.66 (m, 3H), 5.98 and 5.97 (m, 2H), 3.97–3.61 (m, 8H), 3.20–3.17 (m, 4H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 148.6, 147.4, 147.4, 146.1, 145.9, 130.2, 129.6, 122.2, 122.1, 109.4, 109.3, 108.4, 100.9, 100.9, 65.9, 51.9, 45.6, 45.2, 44.2, 38.9, 32.5 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₆S⁺ 343.0958, found 343.0962.

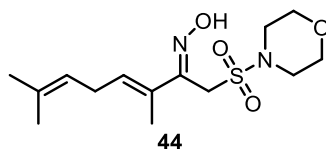


(Z)-7-methyl-3-methylene-1-(morpholinylsulfonyl)oct-6-en-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **43** (34.8 mg, 55% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 8.38 (brs, 1H), 8.04 (d, *J* = 10.2 Hz, 1H), 6.18 (d, *J* = 10.2 Hz, 1H), 5.08–5.05 (m, 1H), 3.74–3.72 (m, 6H), 3.30 (t, *J* = 4.7 Hz, 4H), 2.49 (t, *J* = 7.5 Hz, 2H), 2.15 (q, *J* = 7.5 Hz, 2H), 1.67 (s, 3H), 1.59 (s, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 147.7, 137.7, 133.8, 127.2, 122.4, 66.8, 57.2, 46.4, 46.3, 30.9, 27.1, 25.8, 17.9 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₂₅N₂O₄S⁺ 317.1530, found 317.1536.

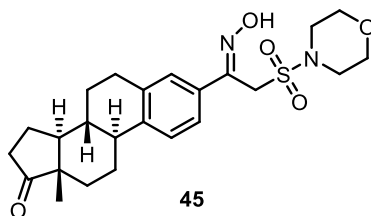


(2Z,3E)-3,7-dimethyl-1-(morpholinylsulfonyl)octa-3,6-dien-2-one oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **44** (27.8 mg, 44% yield, *Z/E* = 1.2:1) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 8.69 and 8.46 (brs, 1H), 8.09–6.15 (m, 2H), 4.88–4.85 (m, 1H), 3.71–3.60 (m, 5H), 3.37–3.27 (m, 4H), 2.80–2.58 (m, 2H), 1.98–1.64 (m, 9H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 147.8, 140.2, 138.1, 135.7, 135.7, 126.2, 118.1, 71.3, 71.0, 67.1, 67.0, 46.6, 46.5, 26.5, 26.4, 25.9, 18.2, 14.8, 14.7 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₂₅N₂O₄S⁺ 317.1530, found 317.1520.



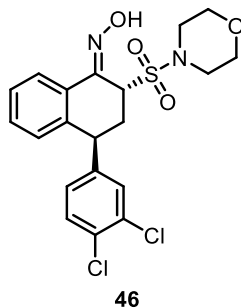
(8*R*,9*S*,13*S*,14*S*)-3-((*Z*)-1-(hydroxyimino)-2-(morpholinosulfonyl)ethyl)-13-methyl-

6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **45** (66.2 mg, 72% yield) was obtained as a white powder.

¹H NMR (500 MHz, CD₃CN): δ = 9.84 (s, 1H), 7.46–7.43 (m, 2H), 7.31 (d, J = 8.1 Hz, 1H), 4.53 (s, 2H), 3.62–3.61 (m, 4H), 3.22–3.20 (m, 4H), 2.91–2.88 (m, 2H), 2.46–2.37 (m, 2H), 2.29–2.24 (m, 1H), 2.10–1.98 (m, 3H), 1.86–1.83 (m, 1H), 1.64–1.39 (m, 6H), 0.86 (m, 3H) ppm.

¹³C NMR (125 MHz, CD₃CN): δ = 148.6, 142.6, 137.8, 132.9, 127.9, 126.4, 124.9, 67.2, 51.1, 48.6, 46.5, 45.2, 45.1, 38.7, 36.3, 32.4, 30.0, 27.0, 26.4, 22.1, 14.2 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₂₄H₃₃N₂O₅S⁺ 461.2105, found 461.2104.



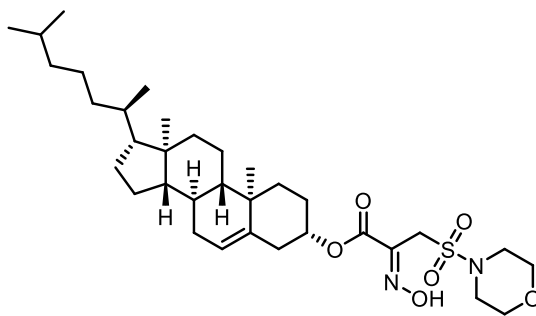
(2*R*,4*S*,*Z*)-4-(3,4-dichlorophenyl)-2-(morpholinofonyl)-3,4-dihydronaphthalen-1(2*H*)-one oxime:

This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **46** (68.1 mg, 75% yield, dr = 2.8:1) was obtained as a yellowish powder.

¹H NMR (500 MHz, DMSO-*d*₆): δ = 12.44 and 12.02 (s, 1H), 8.42–6.67 (m, 7H), 5.14–5.13 (m, 1H), 4.55–4.42 (m, 1H), 3.60–3.57 (m, 4H), 3.27–3.22 (m, 4H), 2.63–2.59 (m, 1H), 2.42–2.35 (m, 1H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆): δ = 146.3, 146.2, 145.3, 143.7, 140.1, 139.4, 131.3, 131.2, 130.9, 130.7, 130.3, 130.1, 129.9, 129.5, 129.4, 129.3, 129.2, 128.7, 127.6, 126.7, 126.4, 123.8, 66.1, 60.3, 51.0, 46.0, 45.1, 41.5, 40.8, 32.7, 31.5 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₂₀H₂₁Cl₂N₂O₄S⁺ 455.0594, found 455.0607.



47

(3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-

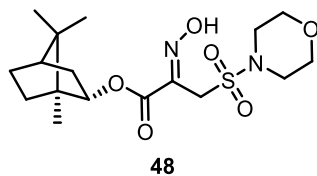
2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl (Z)-2-

(hydroxyimino)-3-(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **47** (74.4 mg, 60% yield) was obtained as a white powder.

¹H NMR (500 MHz, DMSO-*d*₆:CDCl₃ = 2:1): δ = 13.21 (s, 1H), 5.35–5.34 (m, 1H), 4.64–4.62 (m, 1H), 4.30 (s, 2H), 3.62–3.60 (m, 4H), 3.15–3.13 (m, 4H), 2.33 (d, *J* = 8.0 Hz, 2H), 1.98–0.92 (m, 29H), 0.88 (d, *J* = 6.4 Hz, 3H), 0.82 (dd, *J* = 6.6, 2.4 Hz, 6H), 0.64 (s, 3H) ppm.

¹³C NMR (125 MHz, DMSO-*d*₆:CDCl₃ = 2:1): δ = 161.7, 141.0, 138.9, 122.4, 74.9, 65.8, 56.1, 55.5, 49.4, 45.0, 43.0, 41.8, 38.9, 37.4, 36.4, 36.0, 35.6, 35.2, 31.3, 31.3, 27.7, 27.4, 27.2, 23.8, 23.2, 22.6, 22.3, 20.5, 18.9, 18.4, 11.5 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₃₄H₅₇N₂O₆S⁺ 621.3932, found 621.3940.



(1S,2S,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl

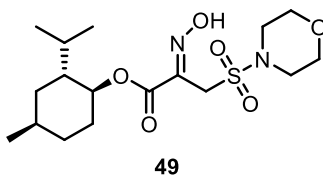
(Z)-2-(hydroxyimino)-3-

(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **48** (56.7 mg, 73% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 11.39 (s, 1H), 4.86 (dd, J = 7.6, 3.9 Hz, 1H), 4.40–4.30 (m, 2H), 3.75–3.73 (m, 4H), 3.31–3.29 (m, 4H), 1.92–1.67 (m, 4H), 1.60–1.54 (m, 1H), 1.19–1.06 (m, 2H), 1.00 (s, 3H), 0.89 (s, 3H), 0.84 (s, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.1, 143.0, 84.0, 66.9, 49.1, 47.1, 45.4, 45.1, 44.0, 38.6, 33.7, 27.0, 20.1, 20.0, 11.6 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₇H₂₉N₂O₆S⁺ 389.1741, found 389.1740.



(1S,2R,4R)-2-isopropyl-4-methylcyclohexyl

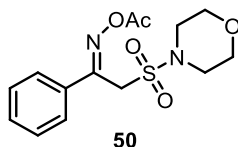
(Z)-2-(hydroxyimino)-3-

(morpholinosulfonyl)propanoate: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 1/1, v/v). The product **49** (53.1 mg, 68% yield) was obtained as a colorless oil.

¹H NMR (500 MHz, CDCl₃): δ = 11.07 (brs, 1H), 4.92 (td, J = 10.9, 4.4 Hz, 1H), 4.44–4.35 (m, 2H), 3.76–3.74 (m, 4H), 3.32–3.30 (m, 4H), 2.08–2.04 (m, 1H), 1.93–1.87 (m, 1H), 1.72–1.67 (m, 2H), 1.53–1.46 (m, 2H), 1.14–1.03 (m, 2H), 0.92–0.84 (m, 7H), 0.76 (d, J = 6.9 Hz, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 162.2, 142.9, 77.4, 66.8, 46.9, 45.5, 44.0, 40.5, 34.1, 31.5, 26.2, 23.4, 22.1, 20.8, 16.3 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₇H₃₁N₂O₆S⁺ 391.1897, found 391.1992.

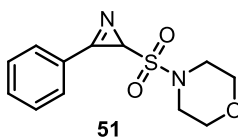


(Z)-2-(morpholinosulfonyl)-1-phenylethan-1-one O-acetyl oxime: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **50** (62.0 mg, 95% yield, *Z/E* = 7.7:1) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 7.82–7.43 (m, 5H), 4.58 and 4.35 (s, 2H), 3.67–3.60 (m, 4H), 3.30–3.19 (m, 4H), 2.33 and 2.09 (s, 3H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 168.3, 154.3, 132.5, 131.6, 130.7, 129.0, 128.6, 128.2, 127.7, 66.6, 66.5, 55.6, 47.9, 46.1, 45.8, 20.0, 19.5 ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₄H₁₉N₂O₅S⁺ 327.1009, found 327.1012.

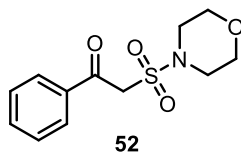


4-((3-phenyl-2H-azirin-2-yl)sulfonyl)morpholine: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **51** (31.9 mg, 60% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 7.99–7.97 (m, 2H), 7.72–7.68 (m, 1H), 7.63–7.60 (m, 2H), 3.77 (t, *J* = 4.7 Hz, 4H), 3.57 (s, 1H), 3.53–3.49 (m, 2H), 3.45–3.41 (m, 2H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 161.5, 135.0, 131.1, 129.7, 121.4, 66.7, 46.4, 43.3. ppm.

HRMS (*m/z*): [M+H]⁺ calcd for C₁₂H₁₅N₂O₃S⁺ 267.0798, found 267.0780.

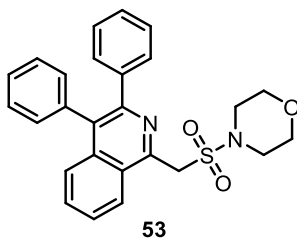


2-(morpholinylsulfonyl)-1-phenylethan-1-one: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **52** (33.9 mg, 63% yield) was obtained as a white powder.

¹H NMR (500 MHz, CDCl₃): δ = 8.04–8.02 (m, 2H), 7.67–7.63 (m, 1H), 7.52 (t, J = 7.8 Hz, 2H), 4.58 (s, 2H), 3.73–3.71 (m, 4H), 3.37–3.35 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 189.2, 135.8, 134.6, 129.5, 129.1, 66.7, 57.4, 46.3 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₁₂H₁₆NO₄S⁺ 270.0795, found 270.0800.

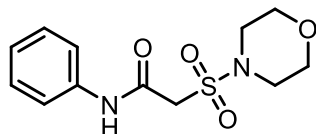


4-(((3,4-diphenylisoquinolin-1-yl)methyl)sulfonyl)morpholine: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 3/1, v/v). The product **53** (62.2 mg, 70% yield) was obtained as a yellowish powder.

¹H NMR (500 MHz, CDCl₃): δ = 8.39–8.37 (m, 1H), 7.73–7.63 (m, 3H), 7.40–7.36 (m, 5H), 7.26–7.21 (m, 5H), 5.13 (s, 2H), 3.64–3.63 (m, 4H), 3.28–3.26 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 149.9, 148.9, 140.2, 136.9, 136.9, 132.0, 131.3, 130.7, 130.2, 128.5, 127.9, 127.7, 127.5, 126.8, 126.5, 126.0, 66.7, 56.8, 46.2 ppm.

HRMS (m/z): [M+H]⁺ calcd for C₂₆H₂₅N₂O₃S⁺ 445.1580, found 445.1590.



54

2-(morpholinosulfonyl)-N-phenylacetamide: This compound was prepared by using the general procedure (eluent: PE/EtOAc, 2/1, v/v). The product **54** (47.7 mg, 84% yield) was obtained as a white powder.

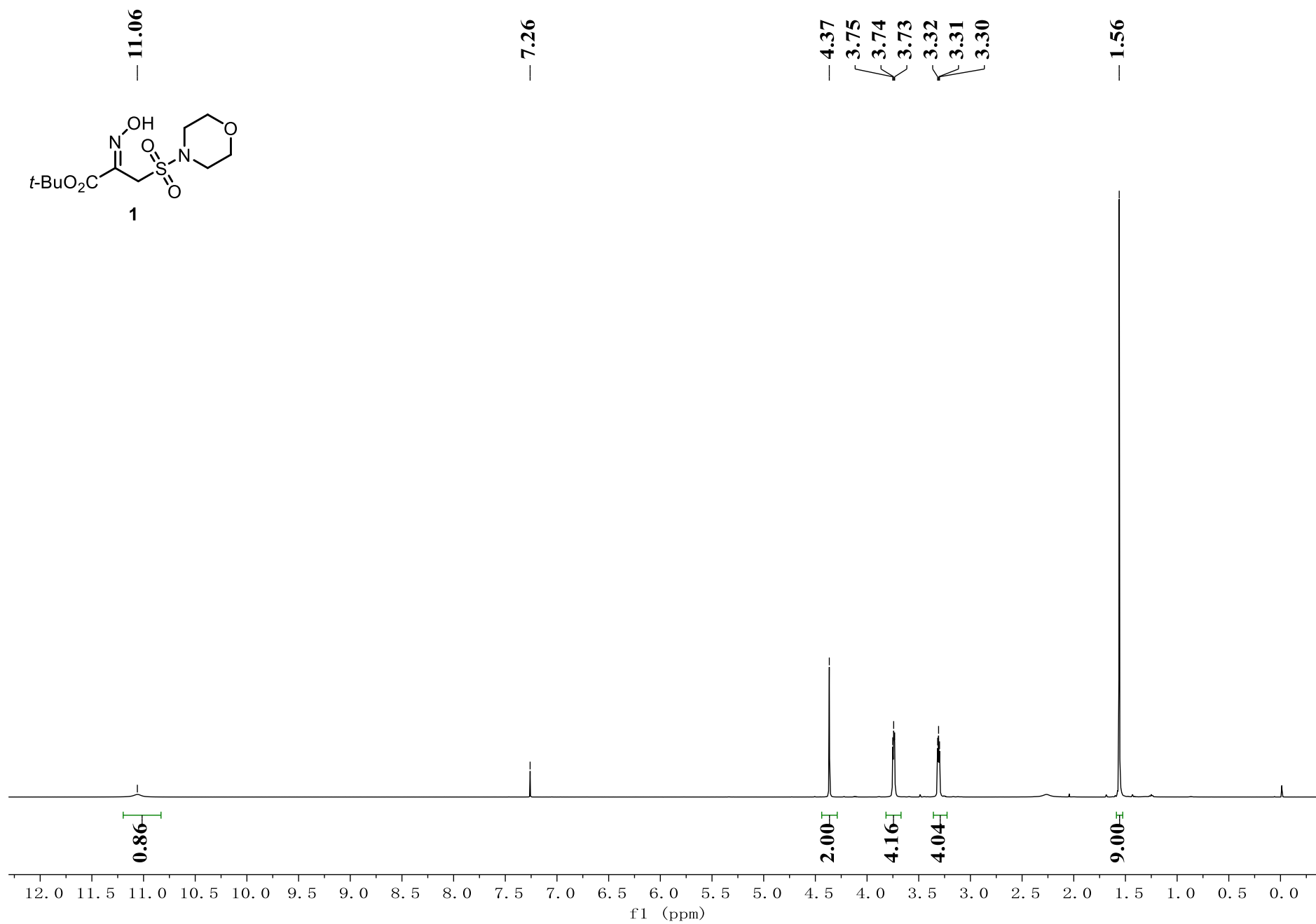
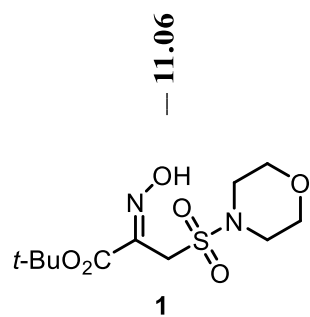
¹H NMR (500 MHz, CDCl₃): δ = 10.41 (s, 1H), 7.59–7.57 (m, 2H), 7.36–7.33 (m, 2H), 7.12–7.09 (m, 1H), 4.21 (s, 2H), 3.65–3.63 (m, 4H), 3.26–3.24 (m, 4H) ppm.

¹³C NMR (125 MHz, CDCl₃): δ = 160.3, 138.5, 129.0, 124.1, 119.3, 65.9, 55.7, 45.7 ppm.

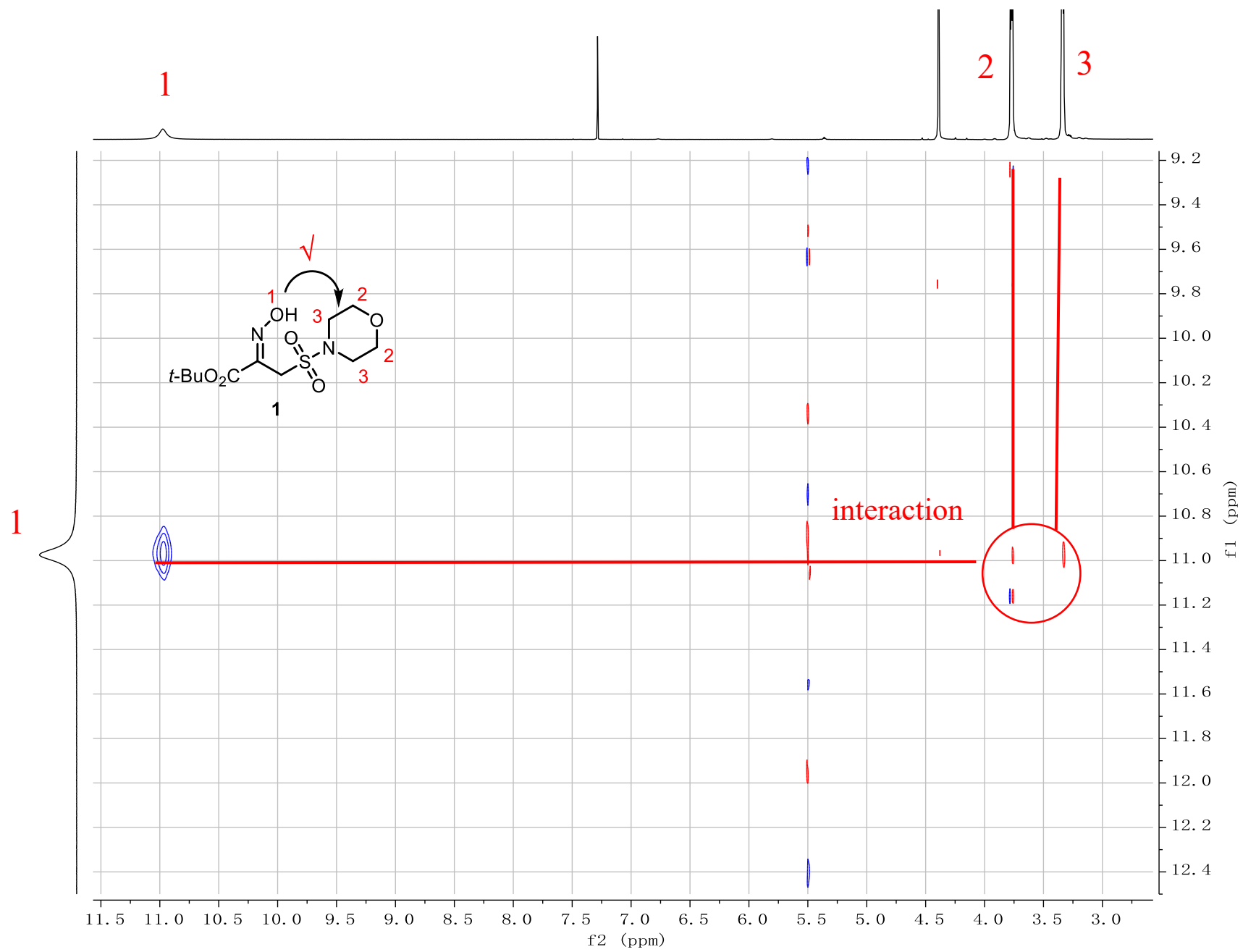
HRMS (m/z): [M+Na]⁺ calcd for C₁₂H₁₆N₂NaO₄S⁺ 307.0723, found 307.0721.

9. NMR spectra data.

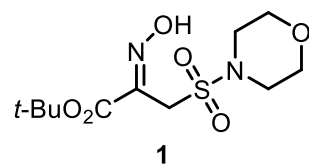
^1H NMR (500 MHz, CDCl_3) for **1**



NOESY of 1



¹³C NMR (125 MHz, CDCl₃) for 1



— 161.39

— 143.59

84.47

77.41

77.16

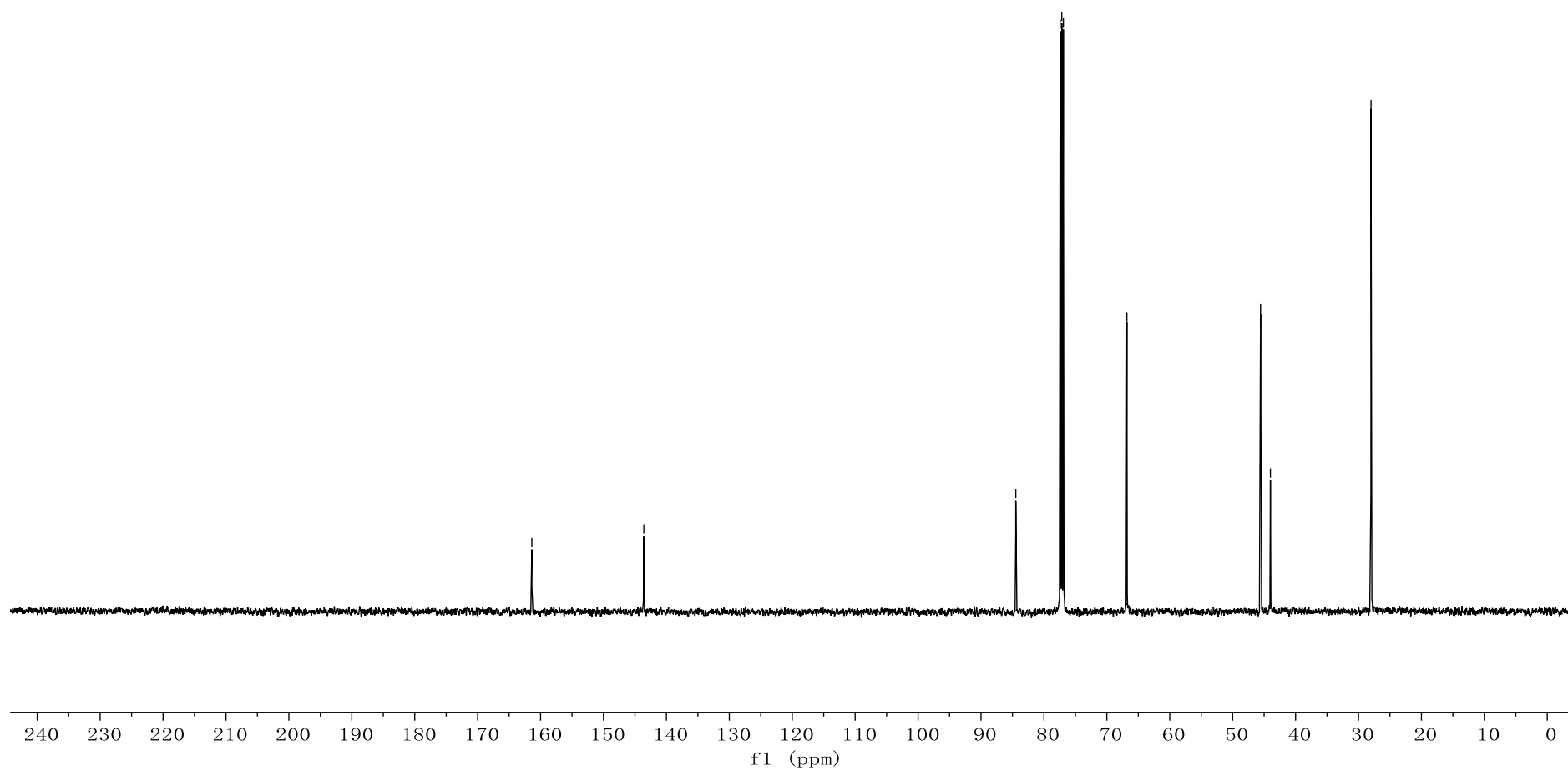
76.91

— 66.81

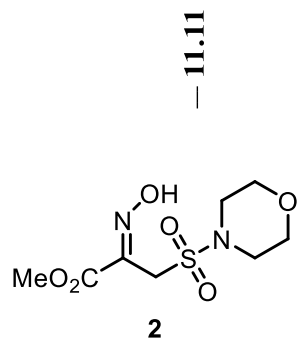
45.57

44.00

— 28.01



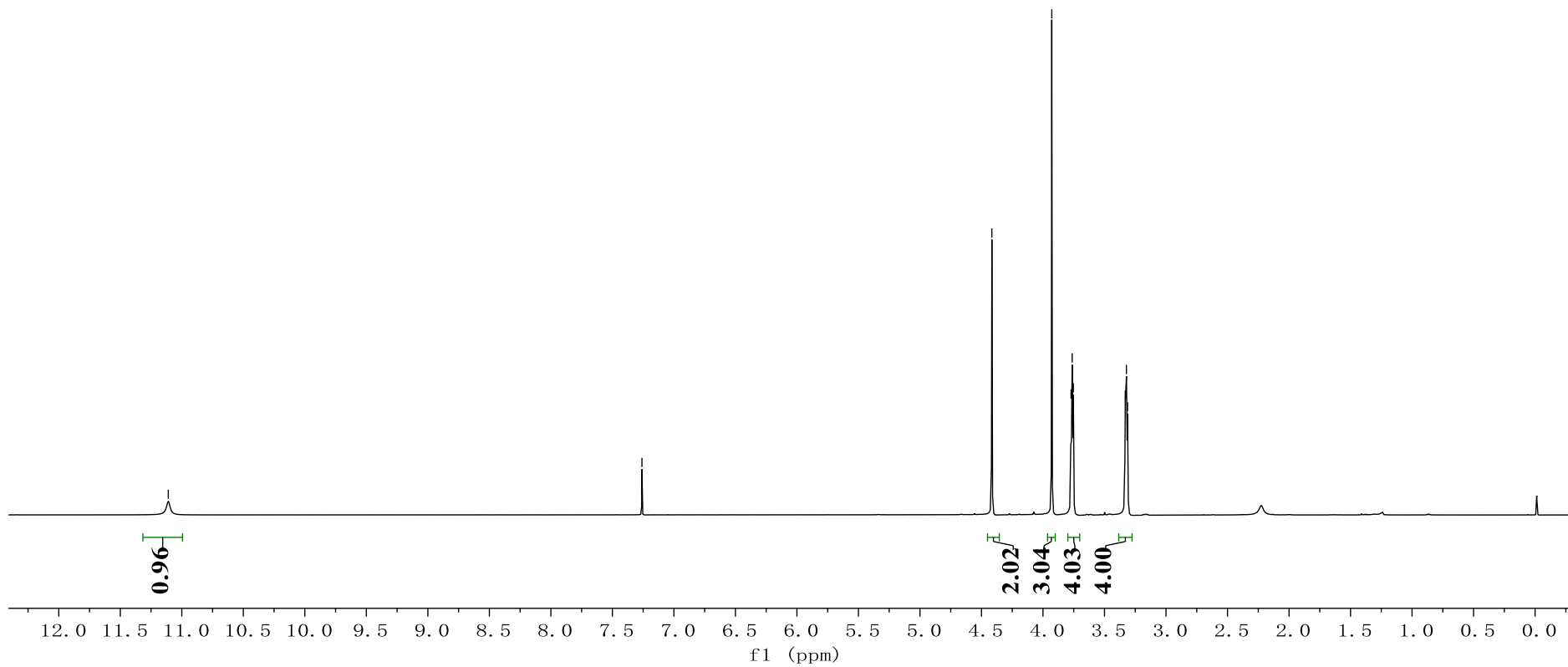
¹H NMR (500 MHz, CDCl₃) for 2



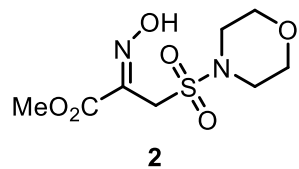
— 11.11

— 7.26

4.42
3.93
3.77
3.76
3.75
3.33
3.32
3.31



¹³C NMR (125 MHz, CDCl₃) for 2



— 163.13

— 142.62

{ 77.41

{ 77.16

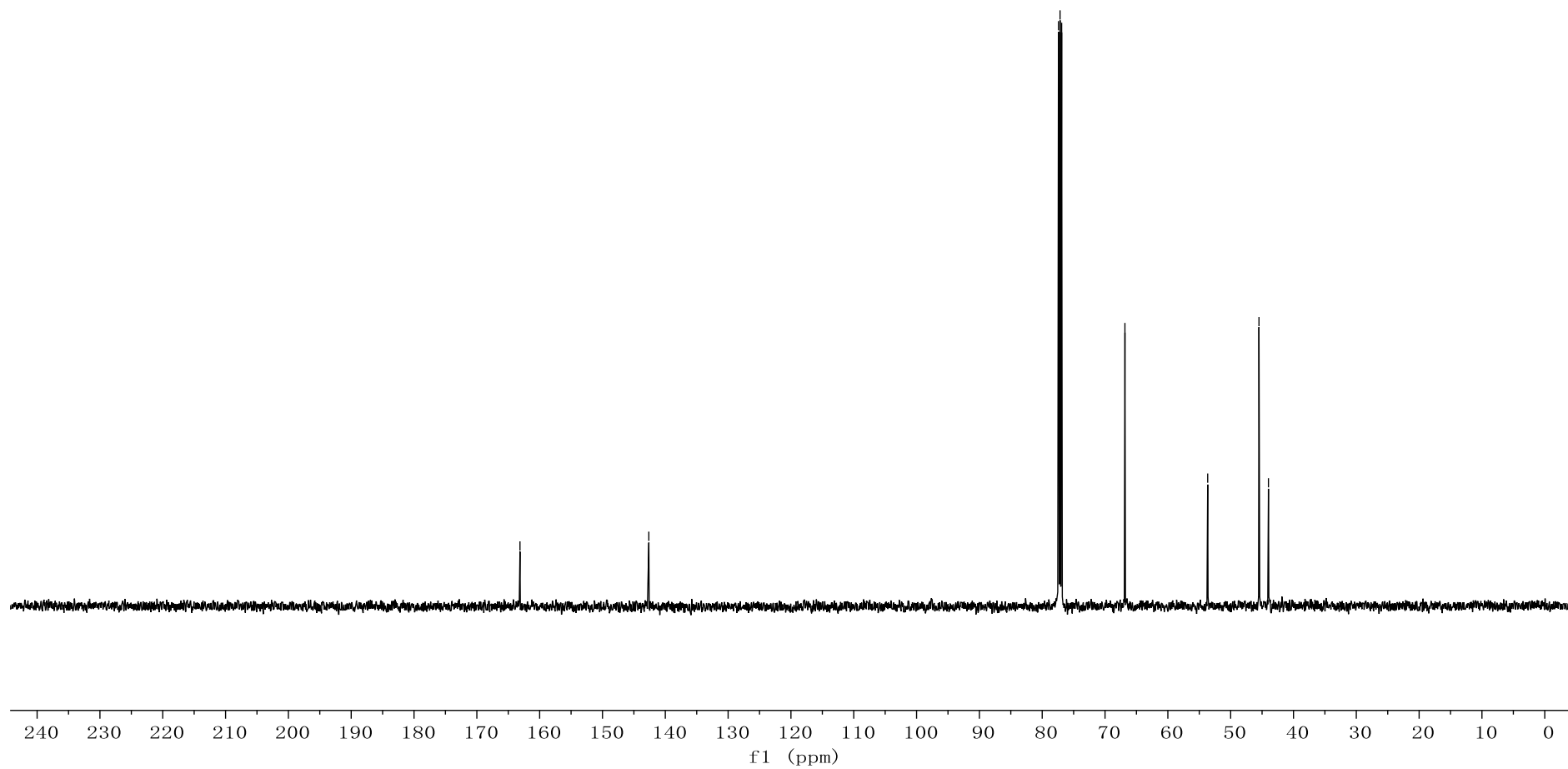
{ 76.91

— 66.83

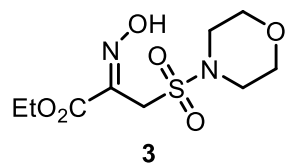
— 53.66

{ 45.49

{ 43.99



¹H NMR (500 MHz, CDCl₃) for 3

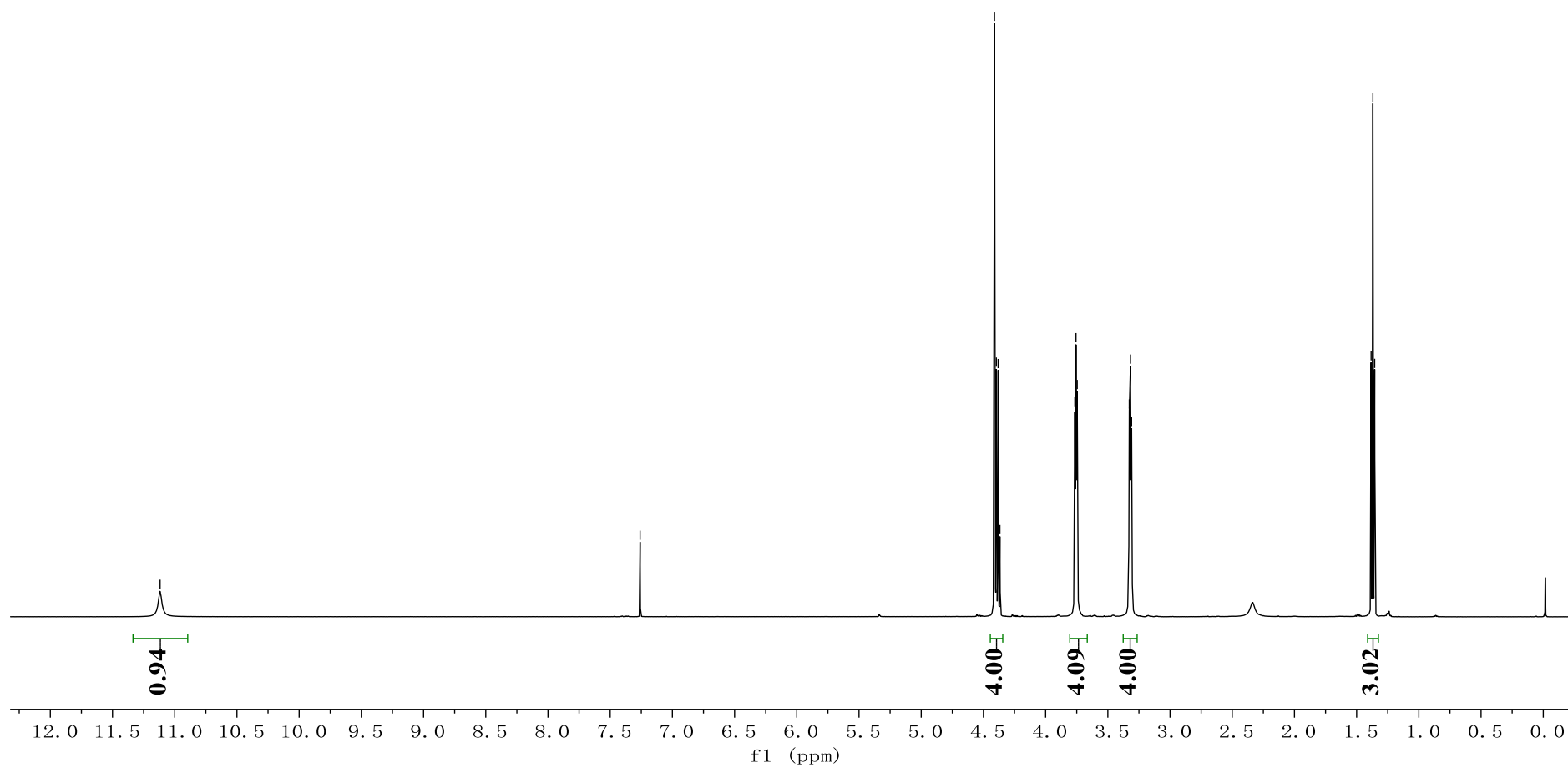


— 11.12

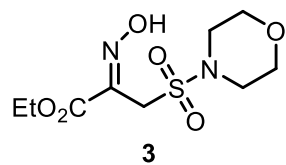
— 7.26

4.41
4.40
4.38
4.37
3.77
3.76
3.75
3.33
3.32
3.31

1.38
1.37
1.36



¹³C NMR (125 MHz, CDCl₃) for 3



— 162.62

— 142.76

77.41

77.16

76.91

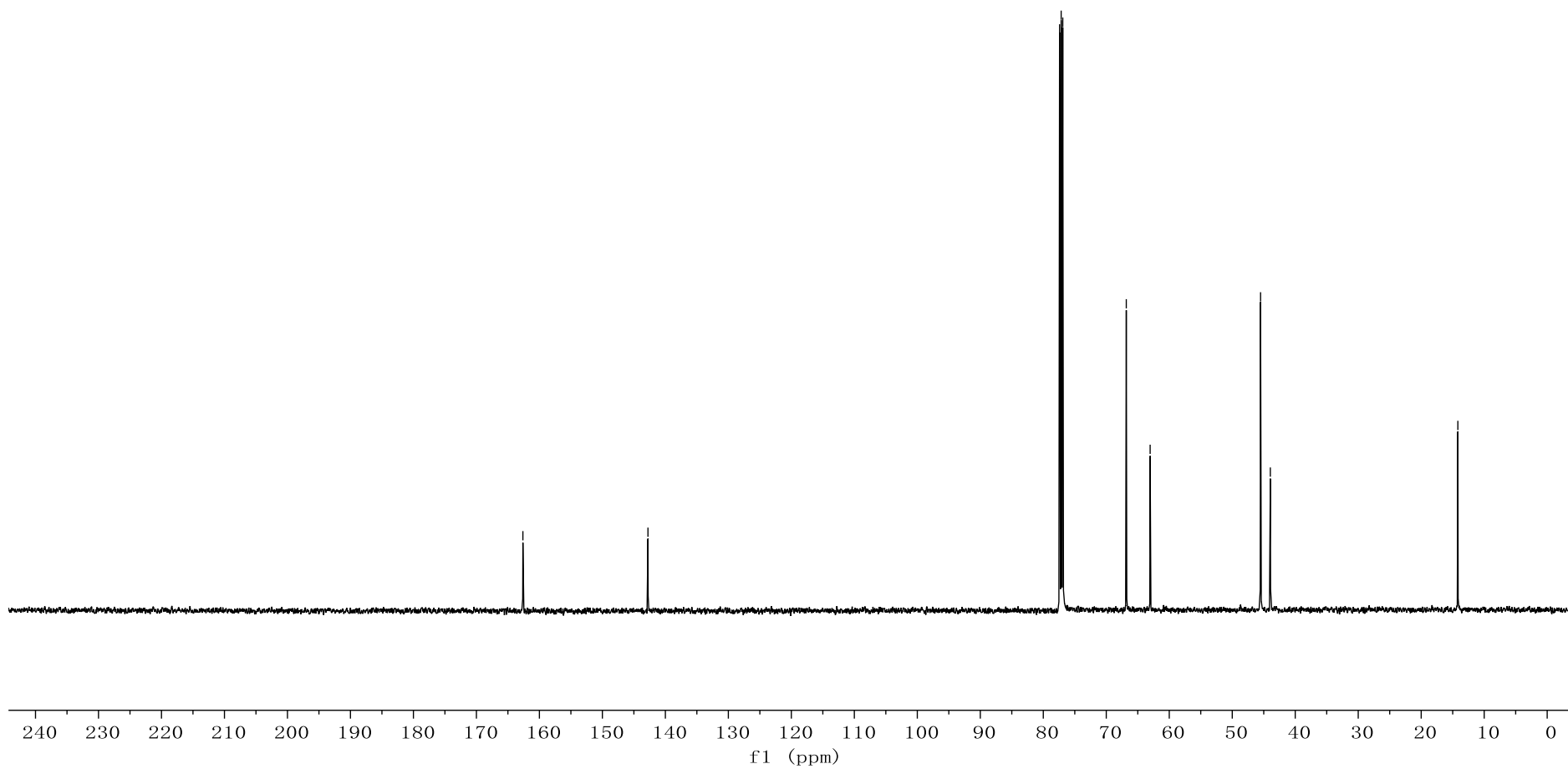
66.82

63.04

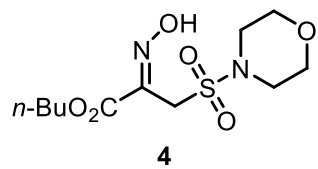
45.50

43.95

— 14.18



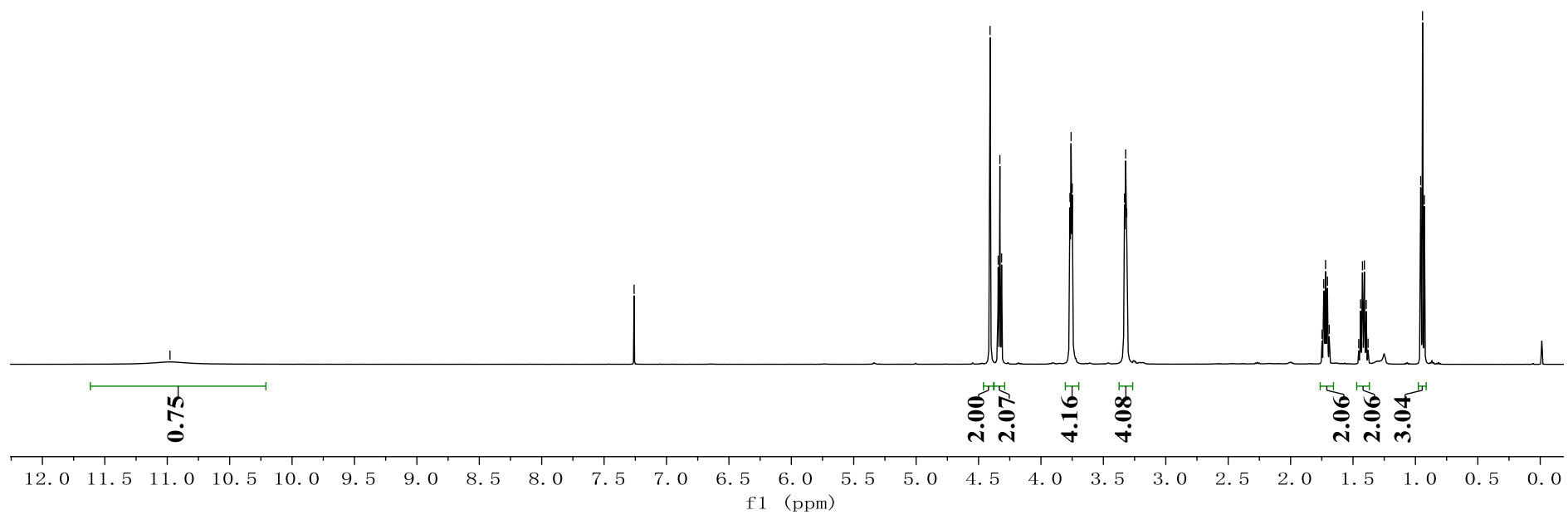
¹H NMR (500 MHz, CDCl₃) for 4



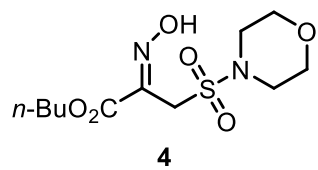
— 10.98

— 7.26

4.41
4.34
4.33
4.32
3.77
3.76
3.75
3.33
3.32
3.31
1.73
1.72
1.70
1.69
1.44
1.42
1.41
1.39
0.96
0.94
0.93



¹³C NMR (125 MHz, CDCl₃) for 4



— 162.69

— 142.84

77.41

77.16

76.91

— 66.85

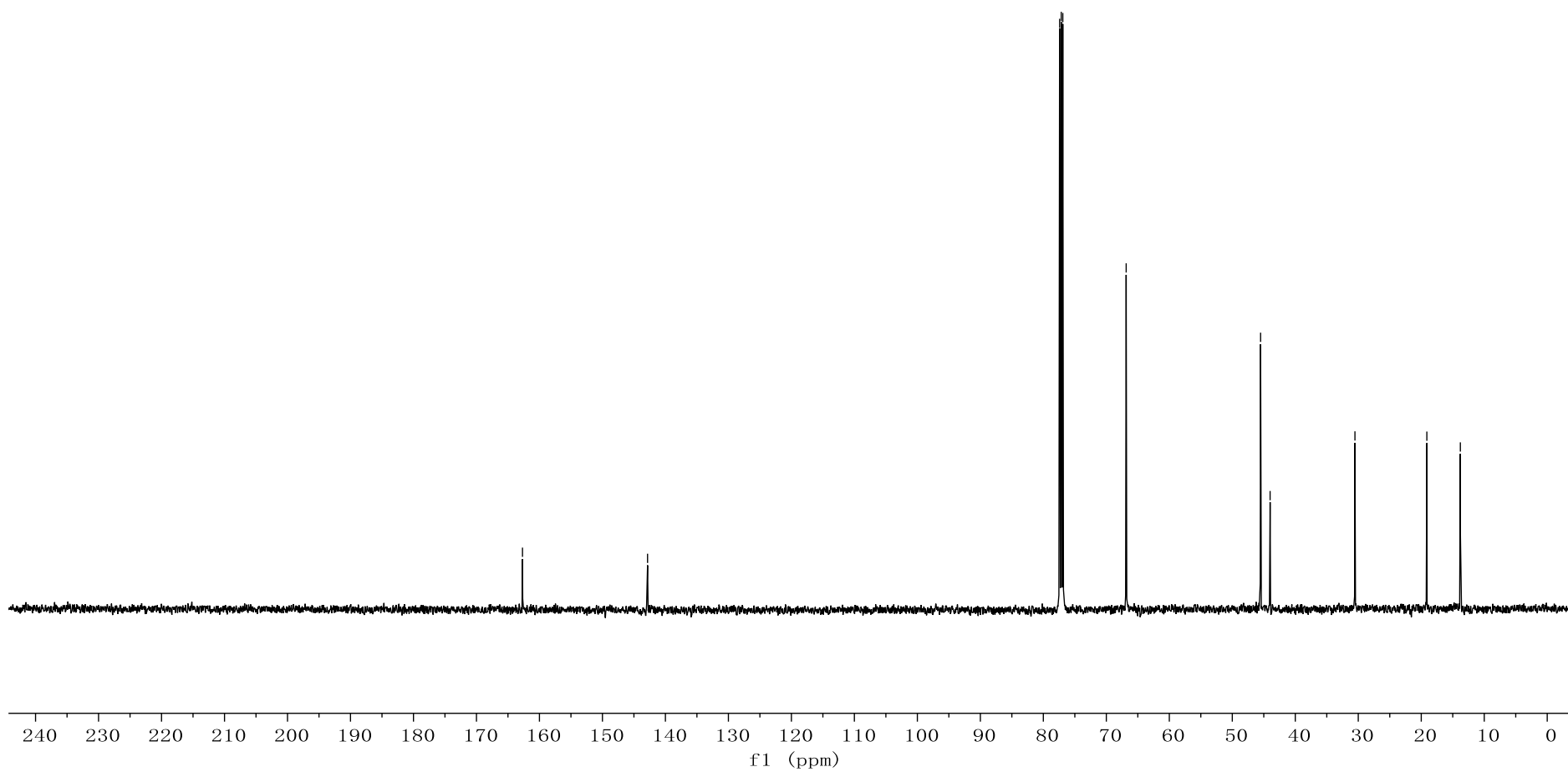
45.51

44.00

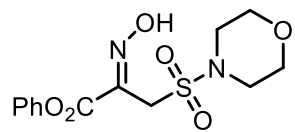
— 30.53

— 19.11

— 13.79



¹H NMR (500 MHz, CDCl₃) for 5

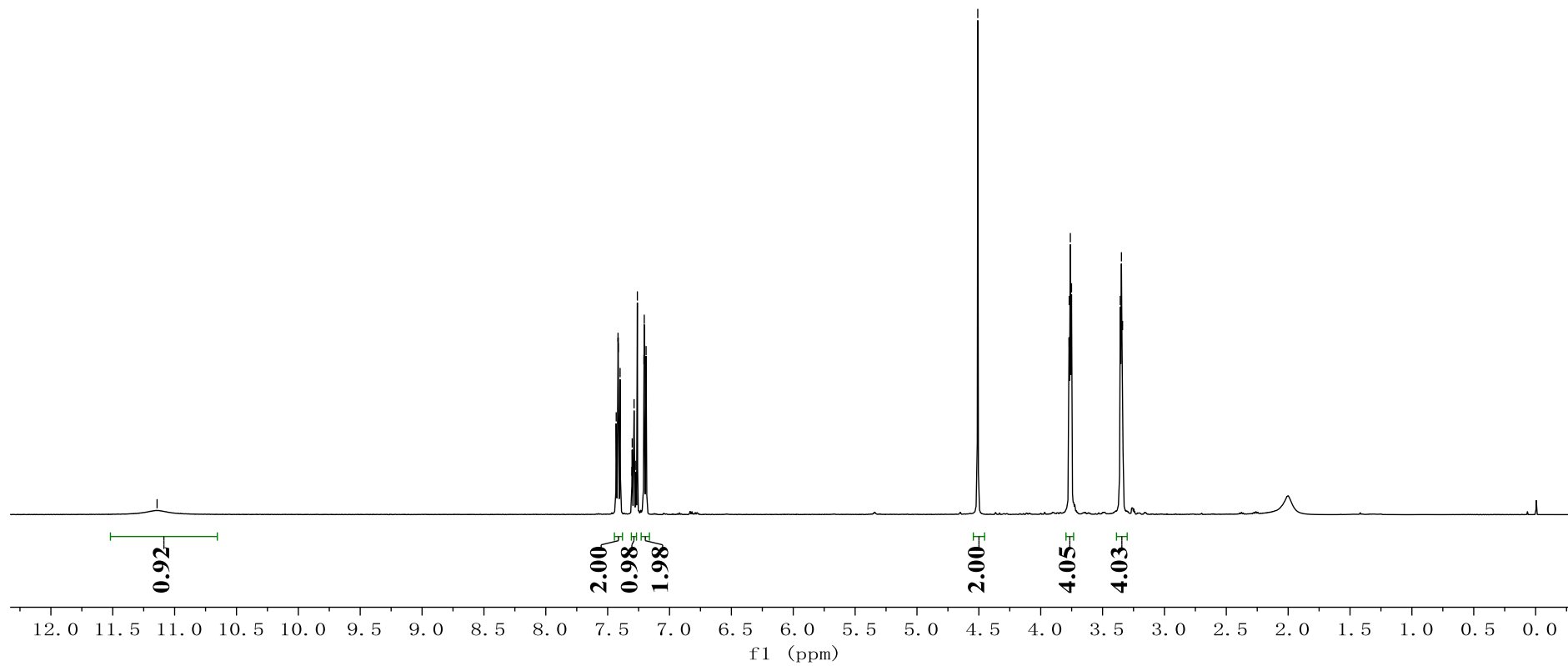


5

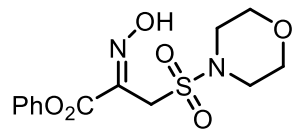
11.14

7.43 7.43 7.42 7.41 7.40 7.40 7.30 7.30 7.30 7.29 7.27 7.27 7.26 7.21 7.20 7.19 7.19 7.19

4.51 3.77 3.76 3.75 3.36 3.35 3.34



¹³C NMR (125 MHz, CDCl₃) for 5

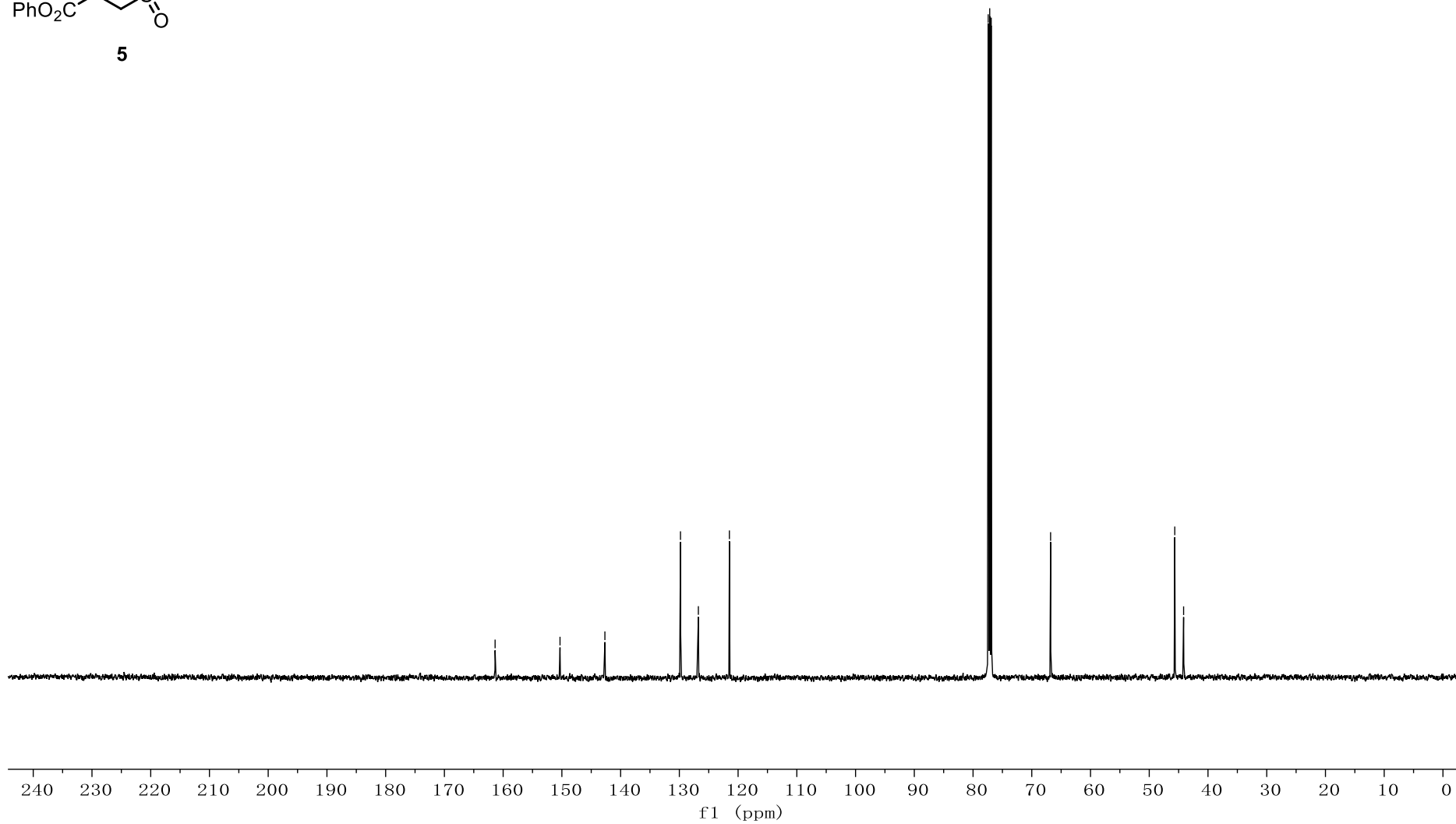


5

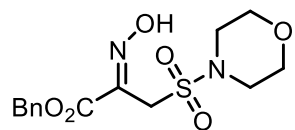
— 161.37
— 150.32
— 142.67
~ 129.81
~ 126.76
~ 121.48

77.41
77.16
76.91
— 66.80

45.66
44.15



¹H NMR (500 MHz, CDCl₃) for 6



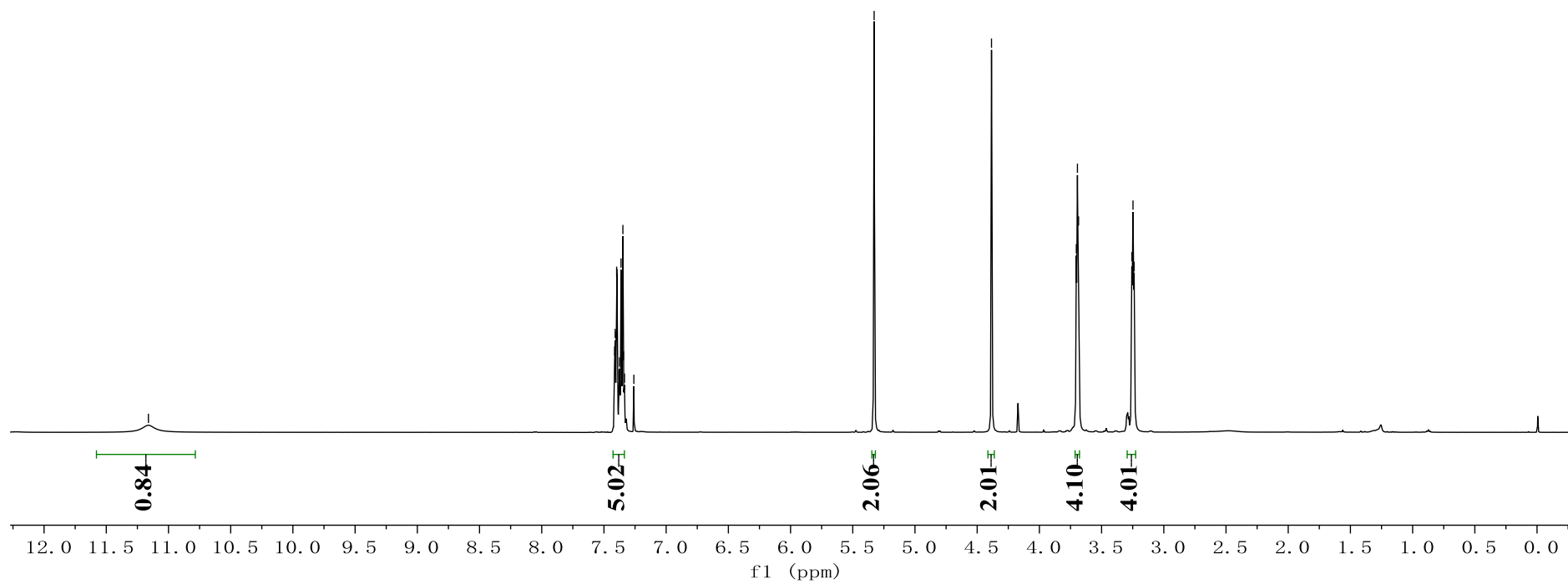
6

11.16

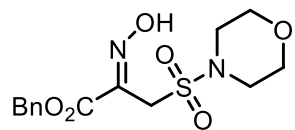
7.41
7.41
7.40
7.40
7.39
7.38
7.37
7.36
7.35
7.35
7.34
7.33
7.26

5.33

4.38
3.70
3.69
3.69
3.26
3.25
3.24

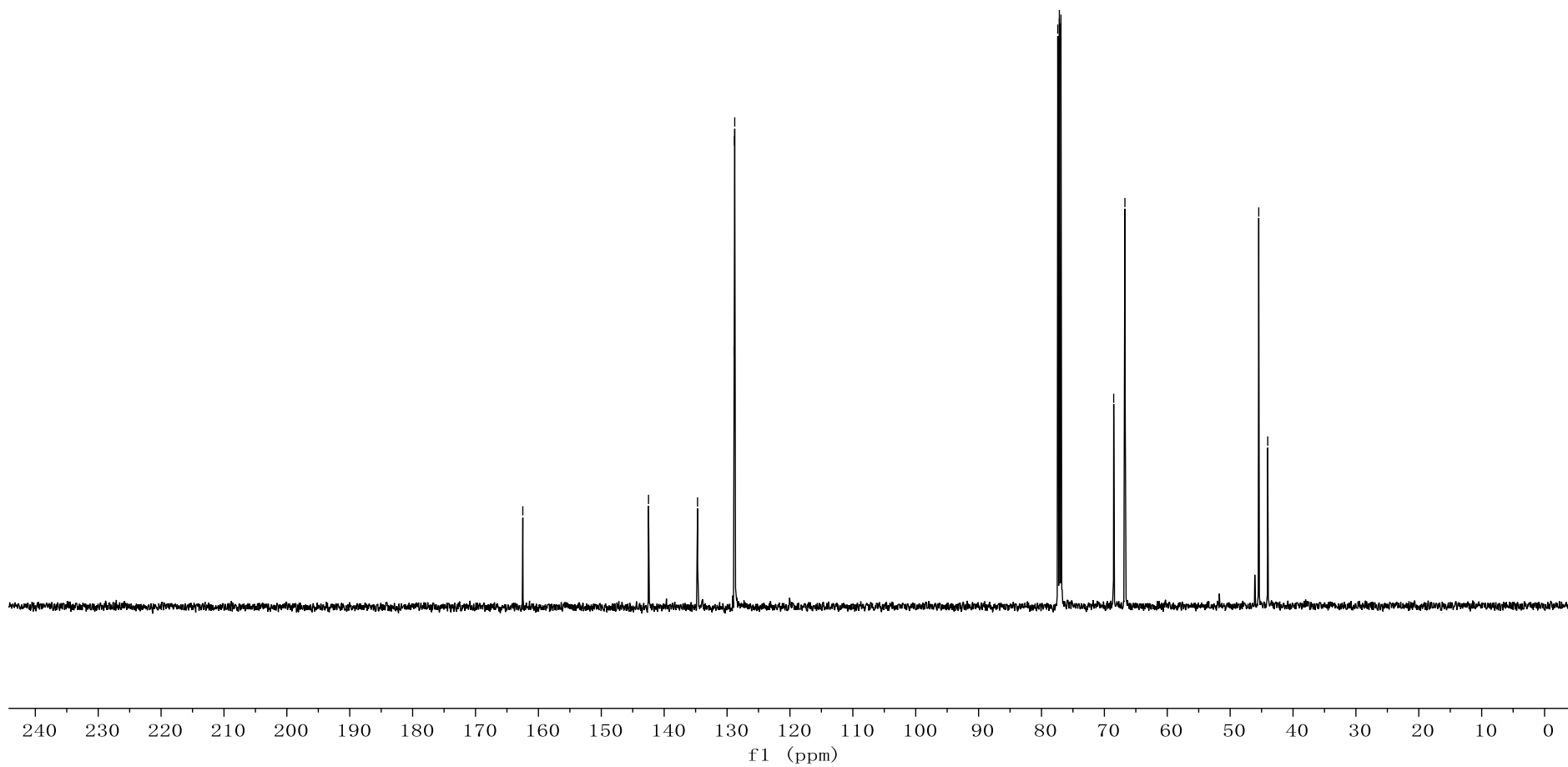


¹³C NMR (125 MHz, CDCl₃) for 6

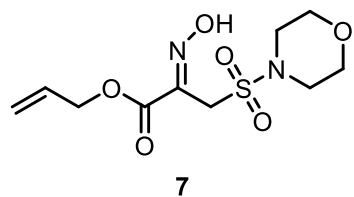


6

162.48
142.49
134.69
128.87
128.82
128.78
77.41
77.16
76.91
68.52
66.74
45.45
44.02



¹³C NMR (125 MHz, CDCl₃) for 7



— 162.33

— 142.70

— 131.04

— 120.00

{ 77.41

{ 77.16

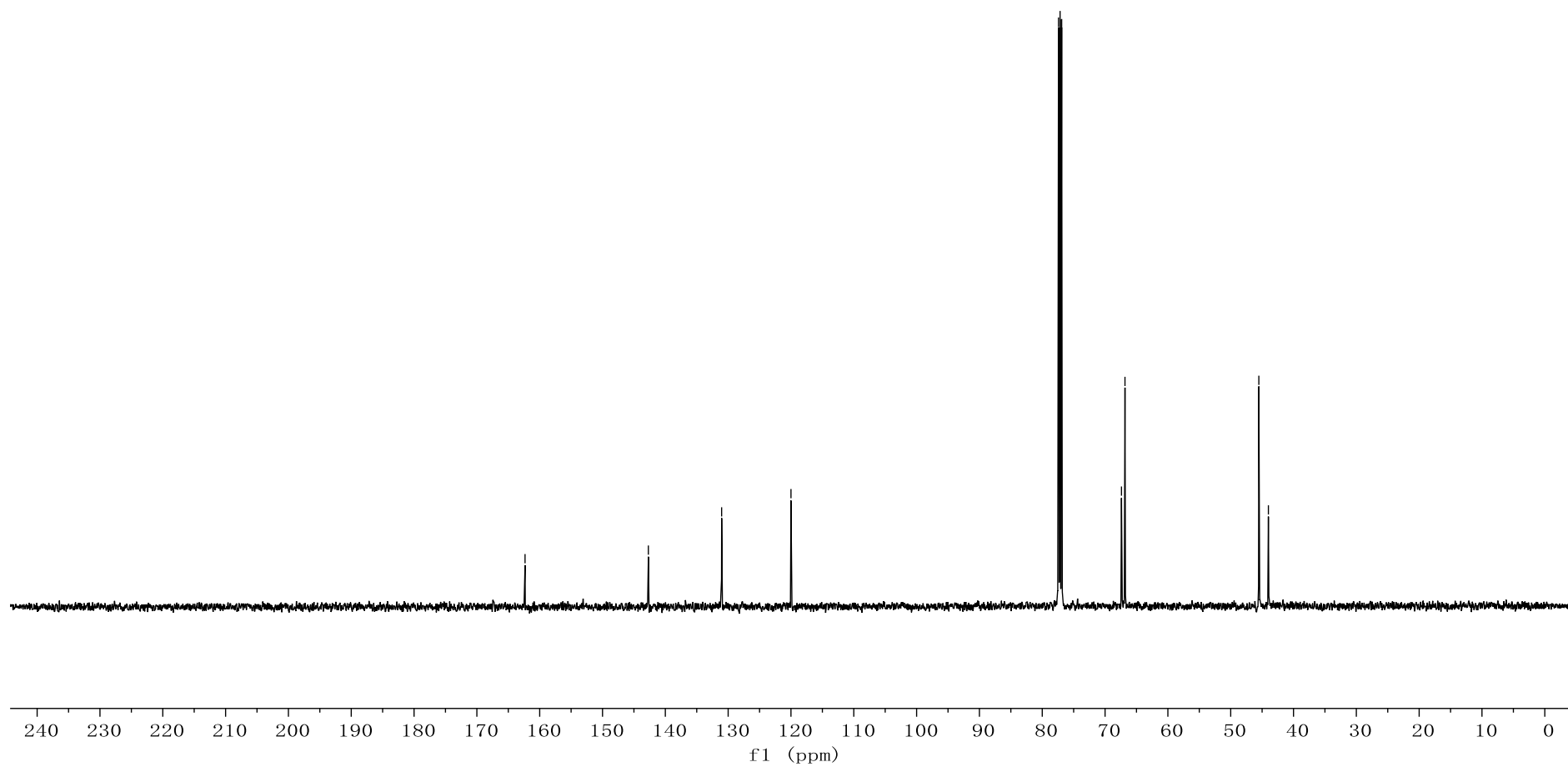
{ 76.91

{ 67.39

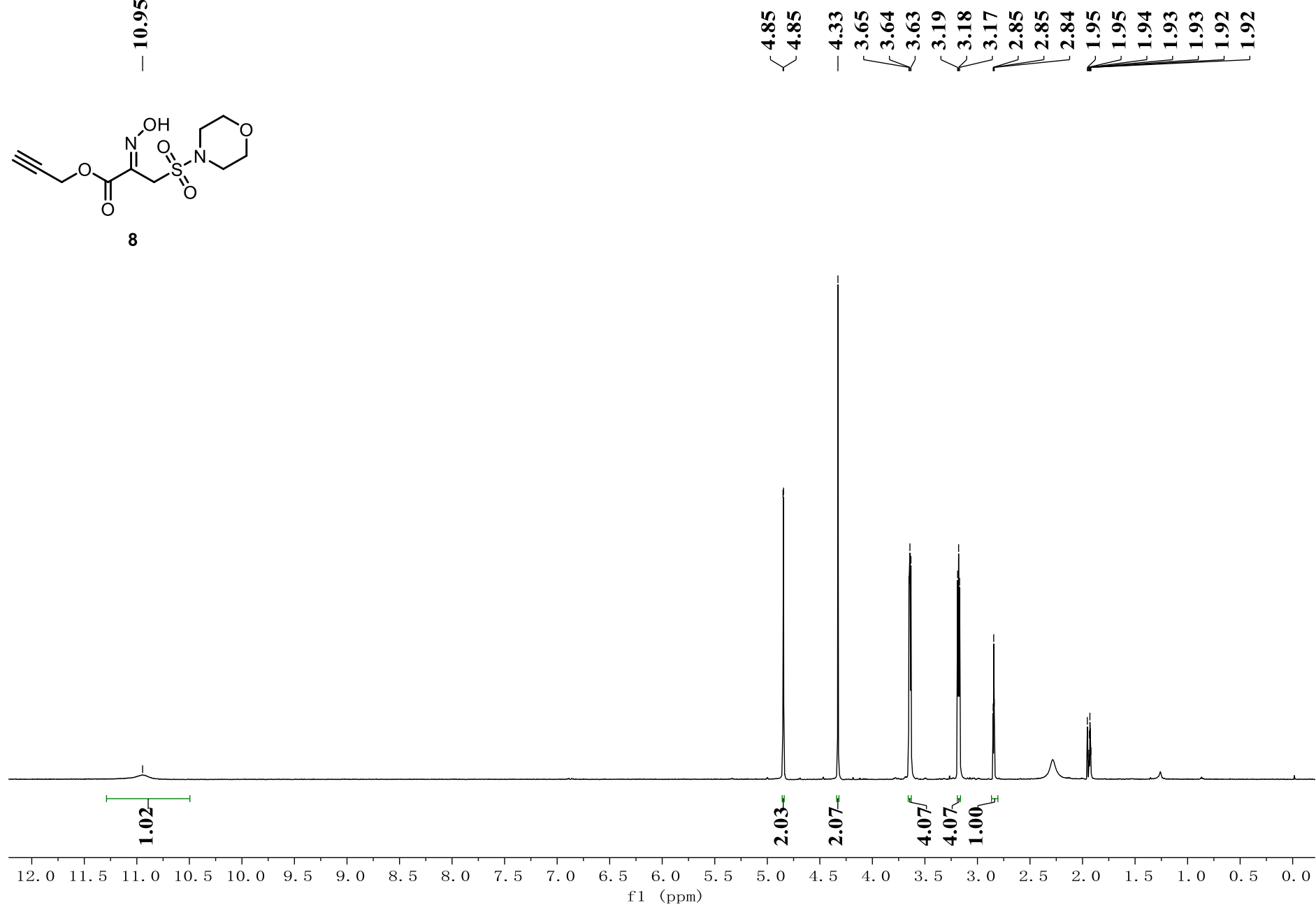
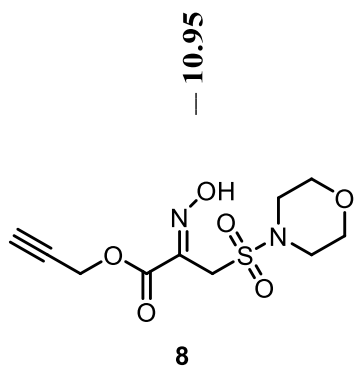
{ 66.83

{ 45.52

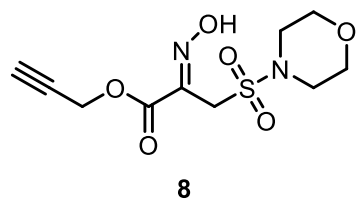
{ 44.00



¹H NMR (500 MHz, CD₃CN) for 8



¹³C NMR (125 MHz, CD₃CN) for 8



— 162.94

— 142.88

— 118.26

∩ 78.05

∩ 76.96

— 67.02

— 54.11

∩ 46.38

— 44.26

∩ 1.76

∩ 1.59

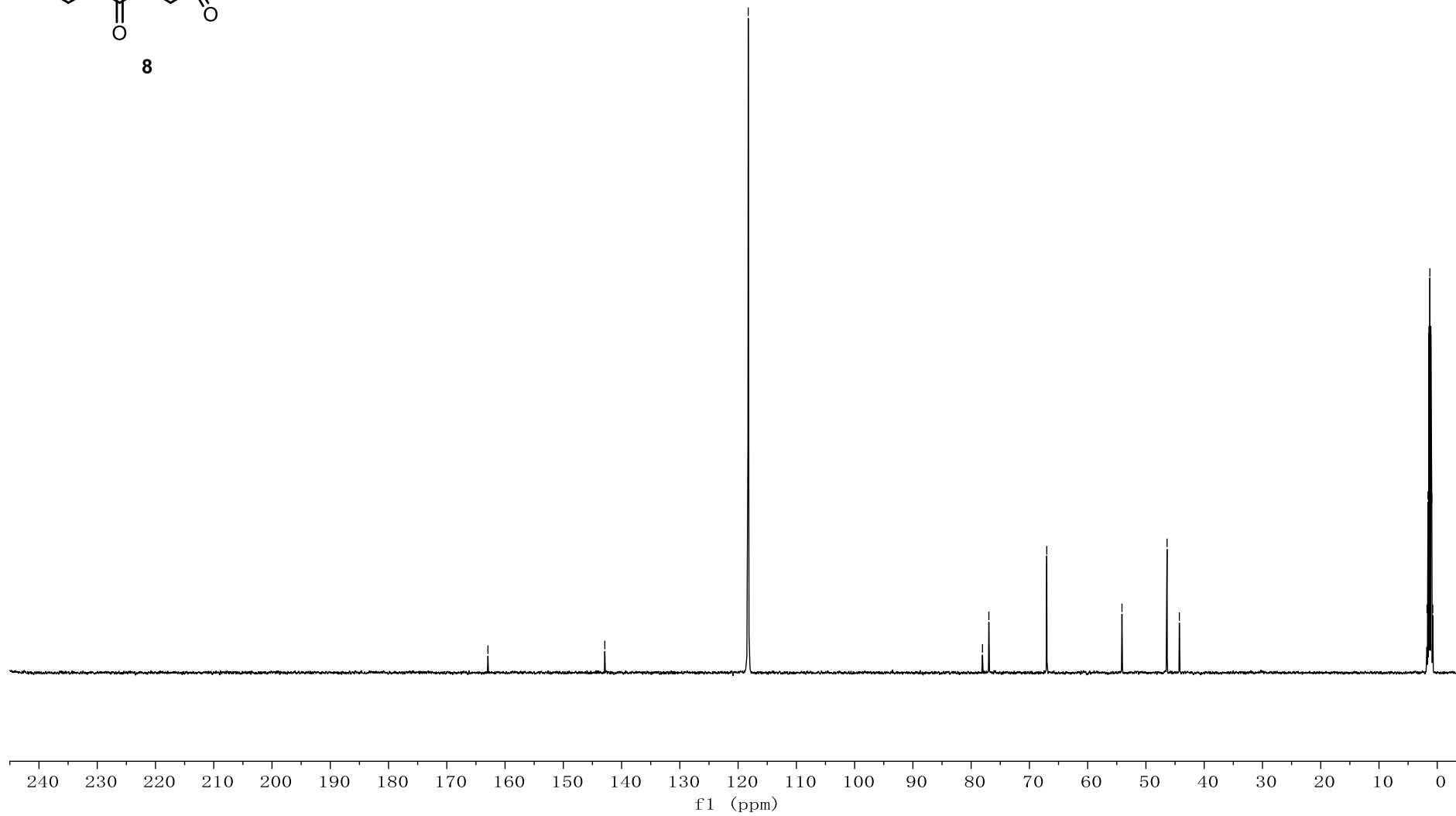
∩ 1.43

∩ 1.26

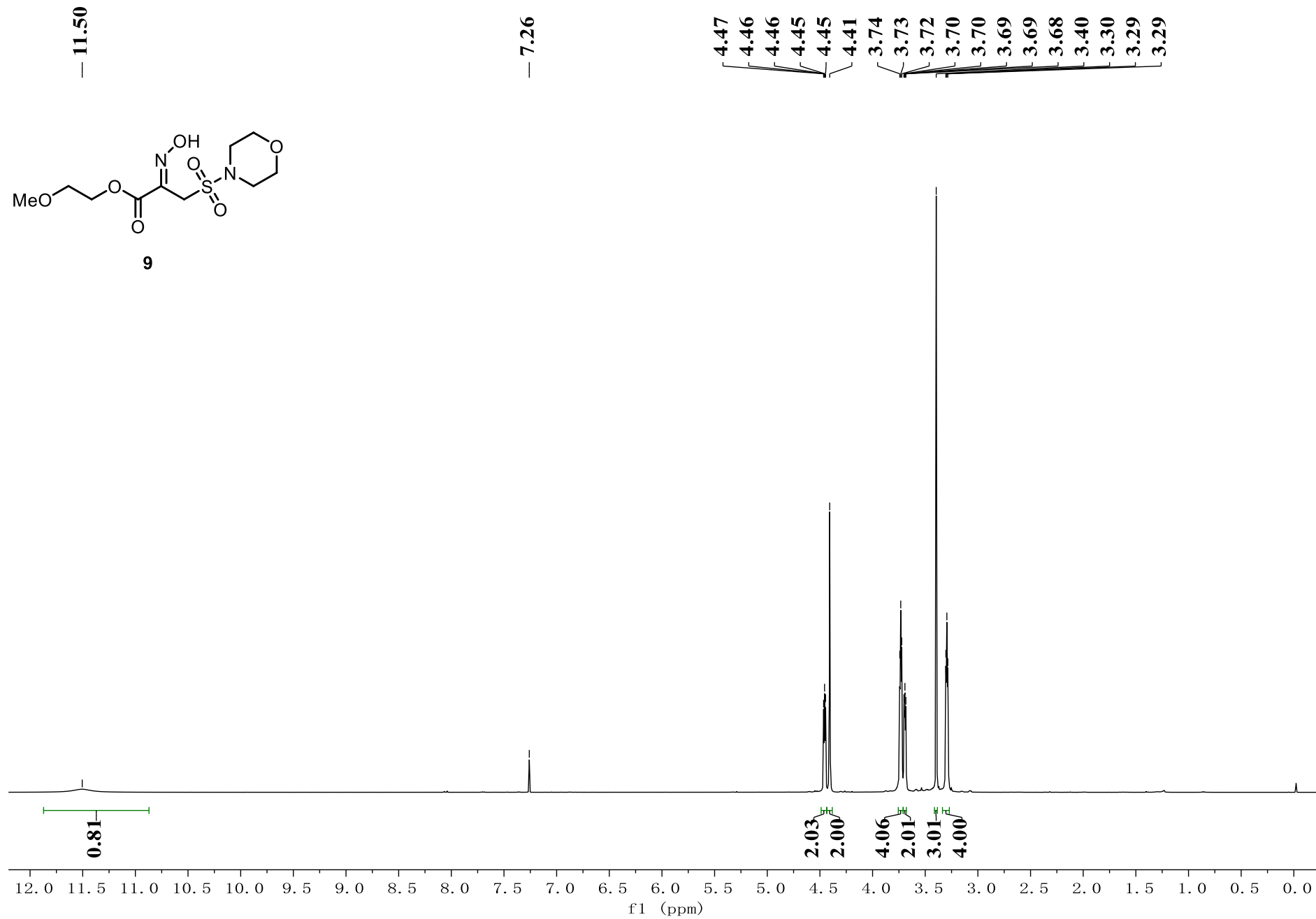
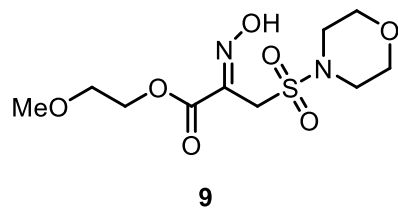
∩ 1.10

∩ 0.93

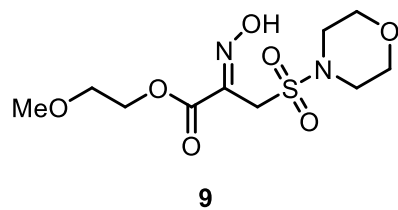
∩ 0.76



¹H NMR (500 MHz, CDCl₃) for 9



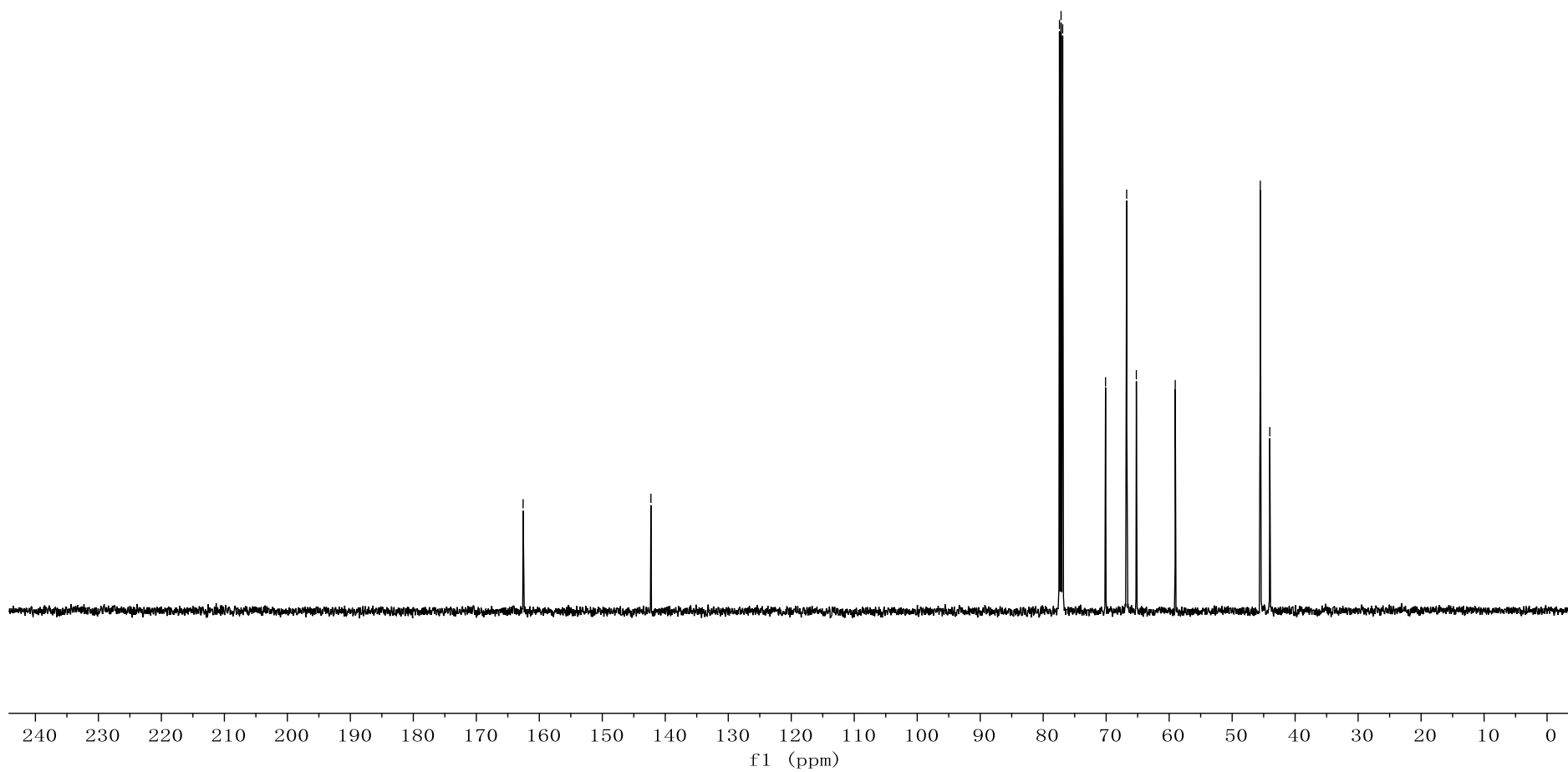
¹³C NMR (125 MHz, CDCl₃) for 9



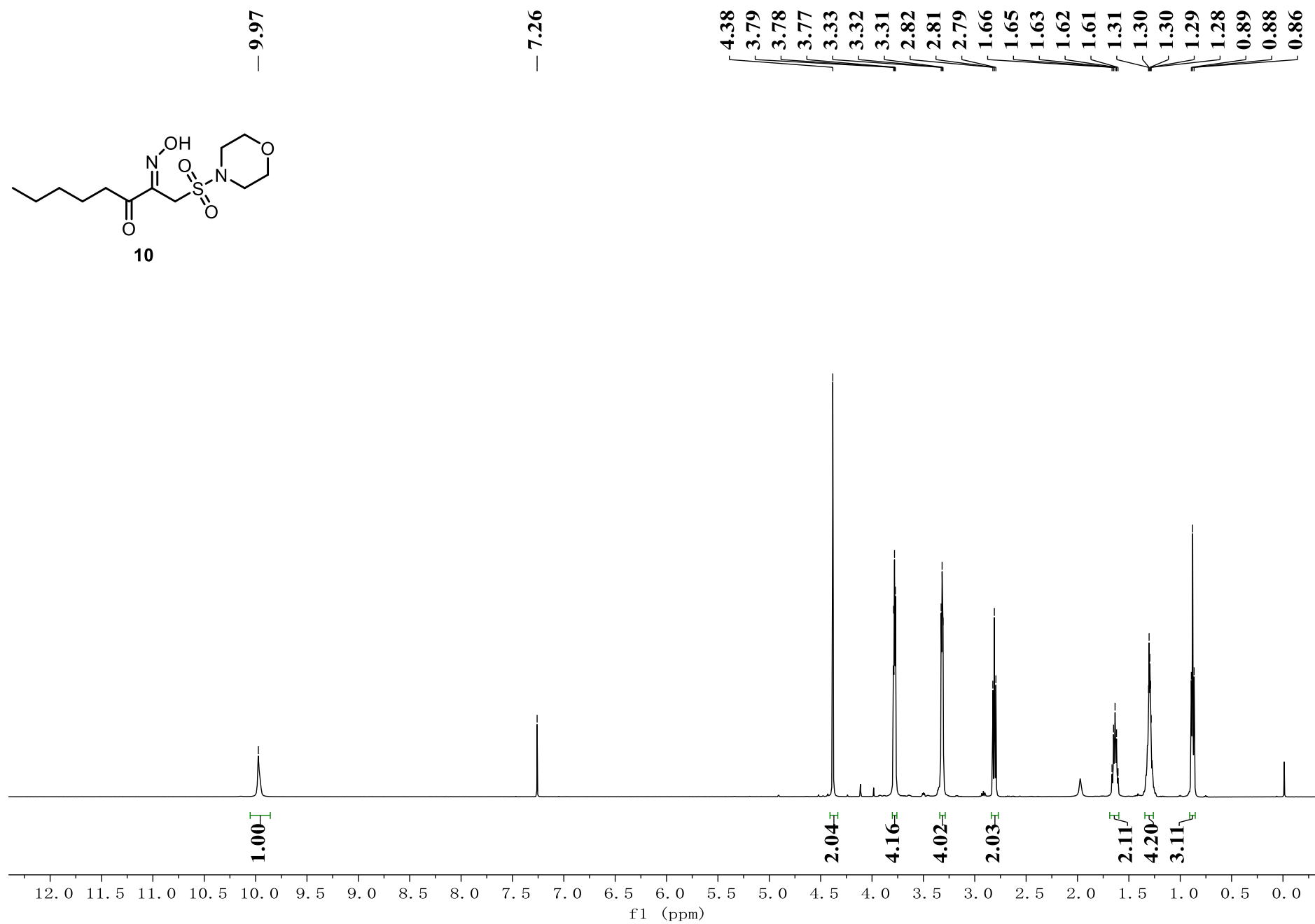
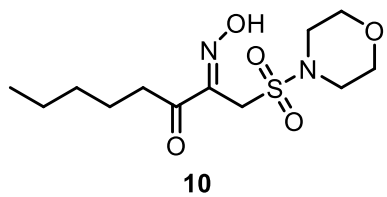
— 162.57

— 142.28

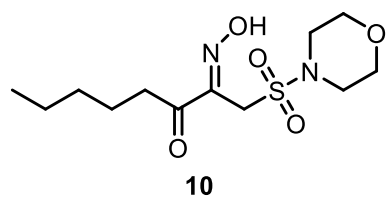
77.41
77.16
76.91
70.10
66.75
65.21
59.03
45.53
44.02



¹H NMR (500 MHz, CDCl₃) for 10



¹³C NMR (125 MHz, CDCl₃) for 10

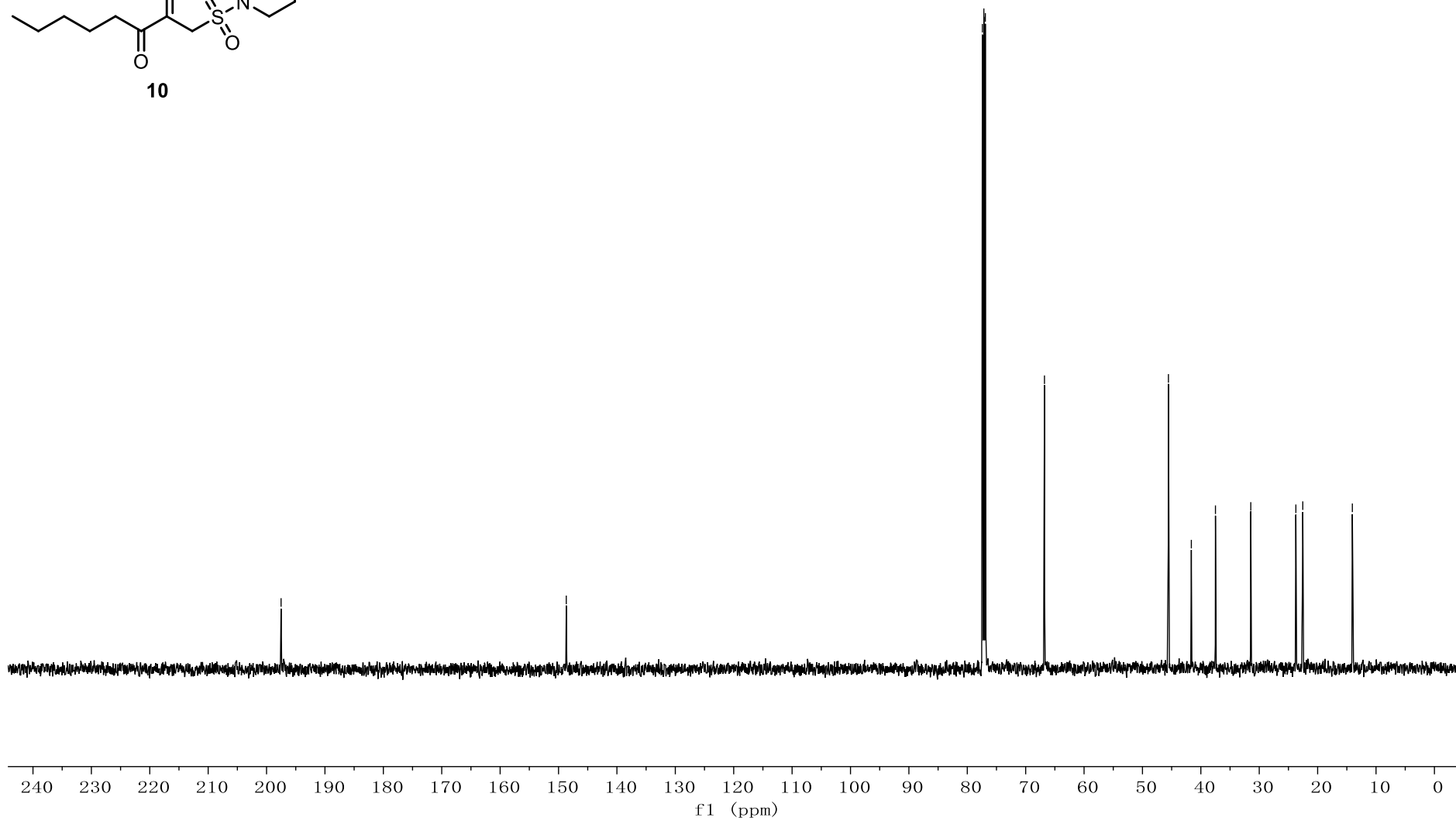


— 197.51

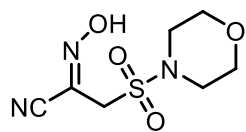
— 148.67

{ 77.41
77.16
76.91
— 66.78

~ 45.55
~ 41.63
~ 37.50
~ 31.45
~ 23.73
~ 22.55
~ 14.06



¹H NMR (500 MHz, CDCl₃) for 11

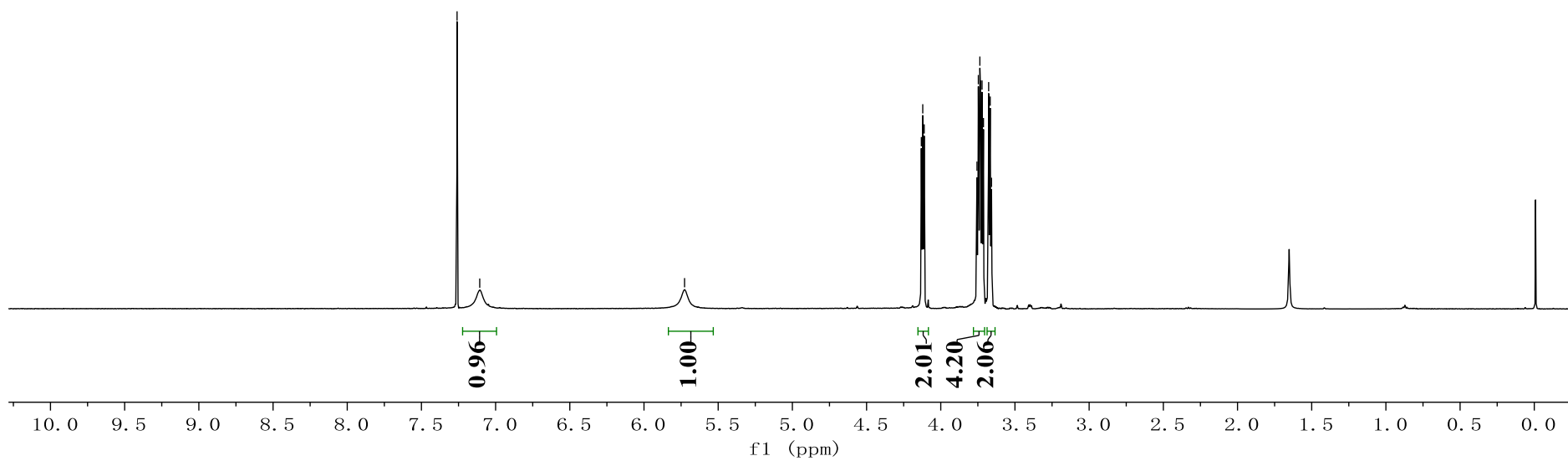


11

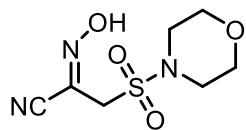
~ 7.26
~ 7.11

— 5.73

4.13
4.12
4.11
3.76
3.75
3.74
3.74
3.73
3.72
3.71
3.68
3.67
3.66



¹³C NMR (125 MHz, CDCl₃) for 11

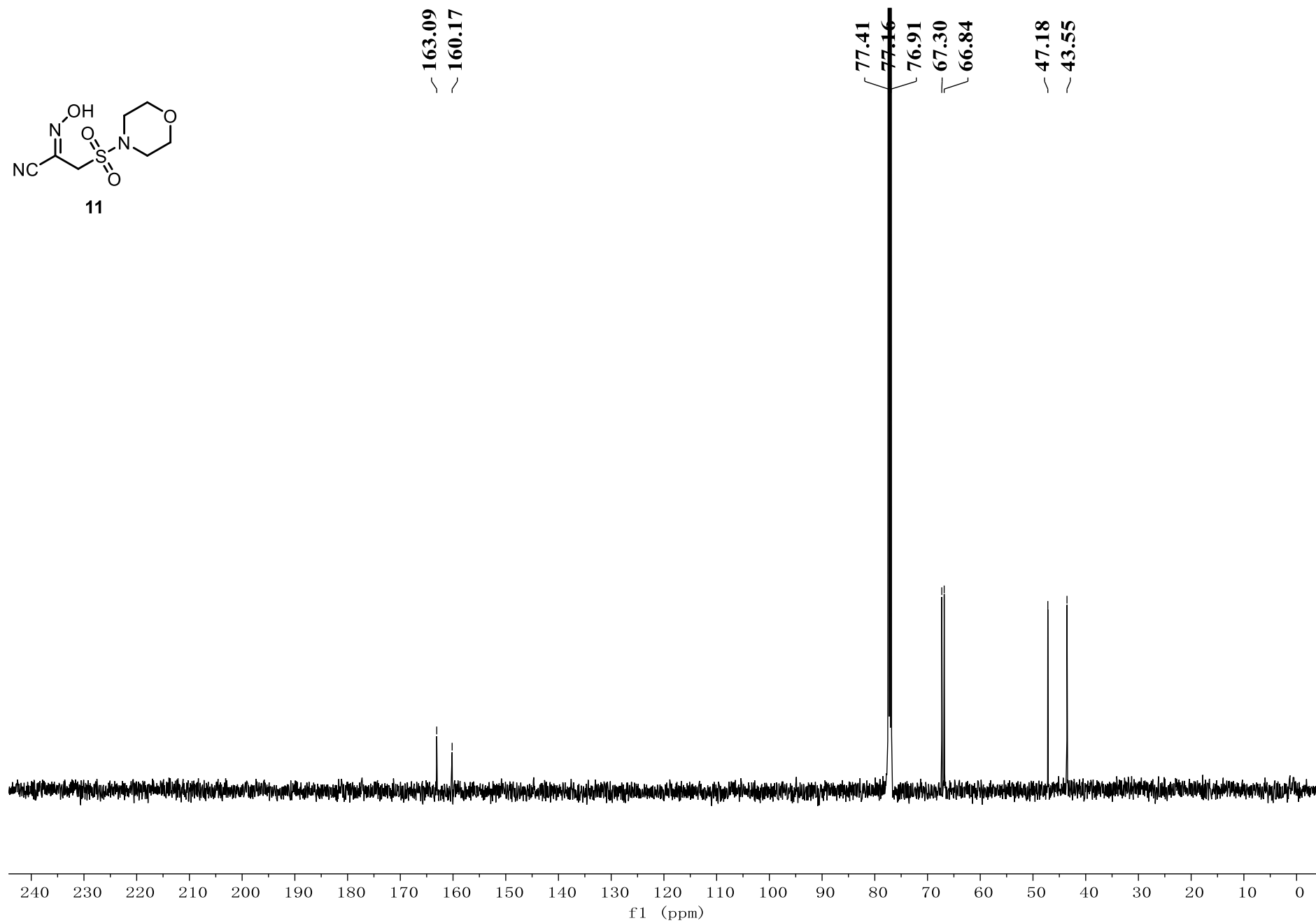


11

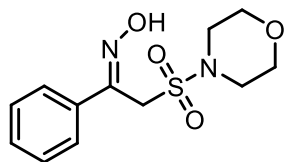
~ 163.09
~ 160.17

77.41
77.16
76.91
67.30
66.84

~ 47.18
~ 43.55



¹H NMR (500 MHz, CDCl₃) for 12



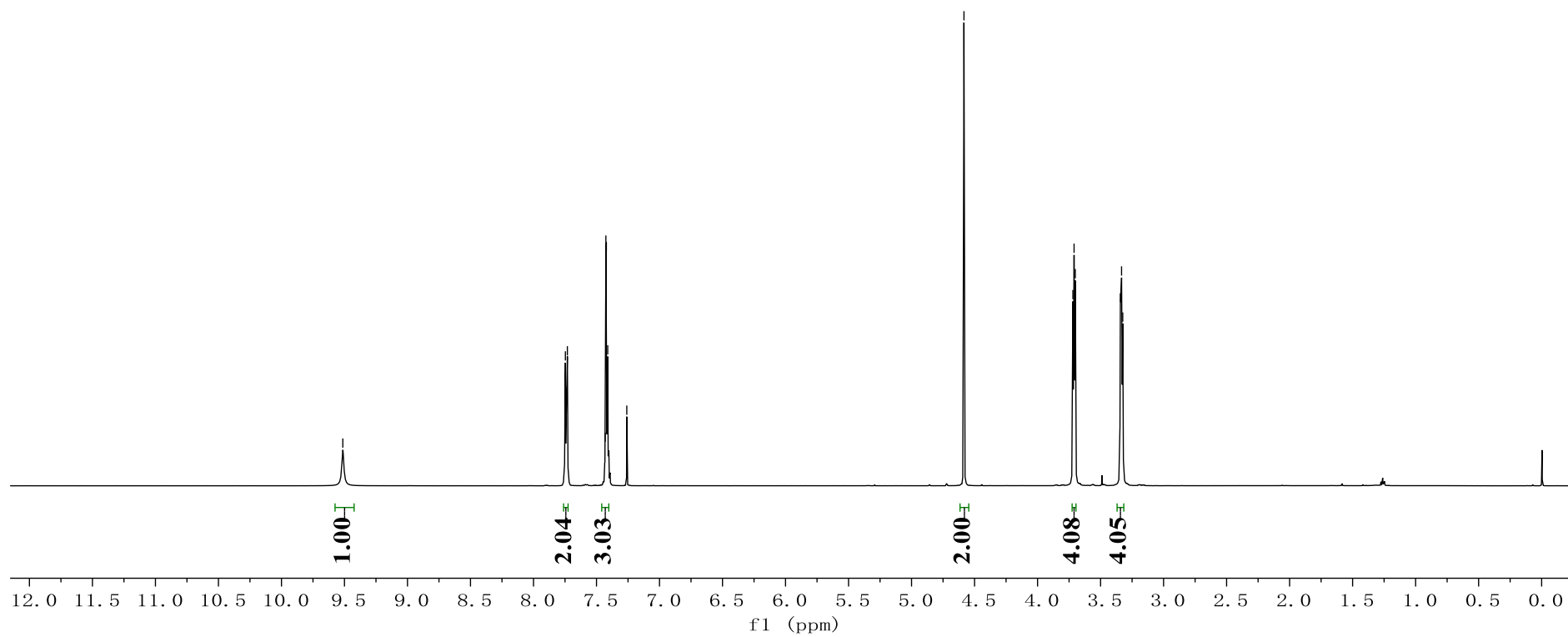
12

9.51

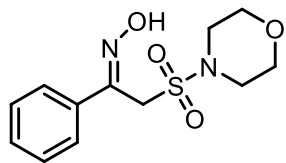
7.75
7.75
7.74
7.74
7.73
7.43
7.43
7.42
7.42
7.41
7.26

4.58

3.72
3.71
3.70
3.34
3.33
3.32



¹³C NMR (125 MHz, CDCl₃) for 12

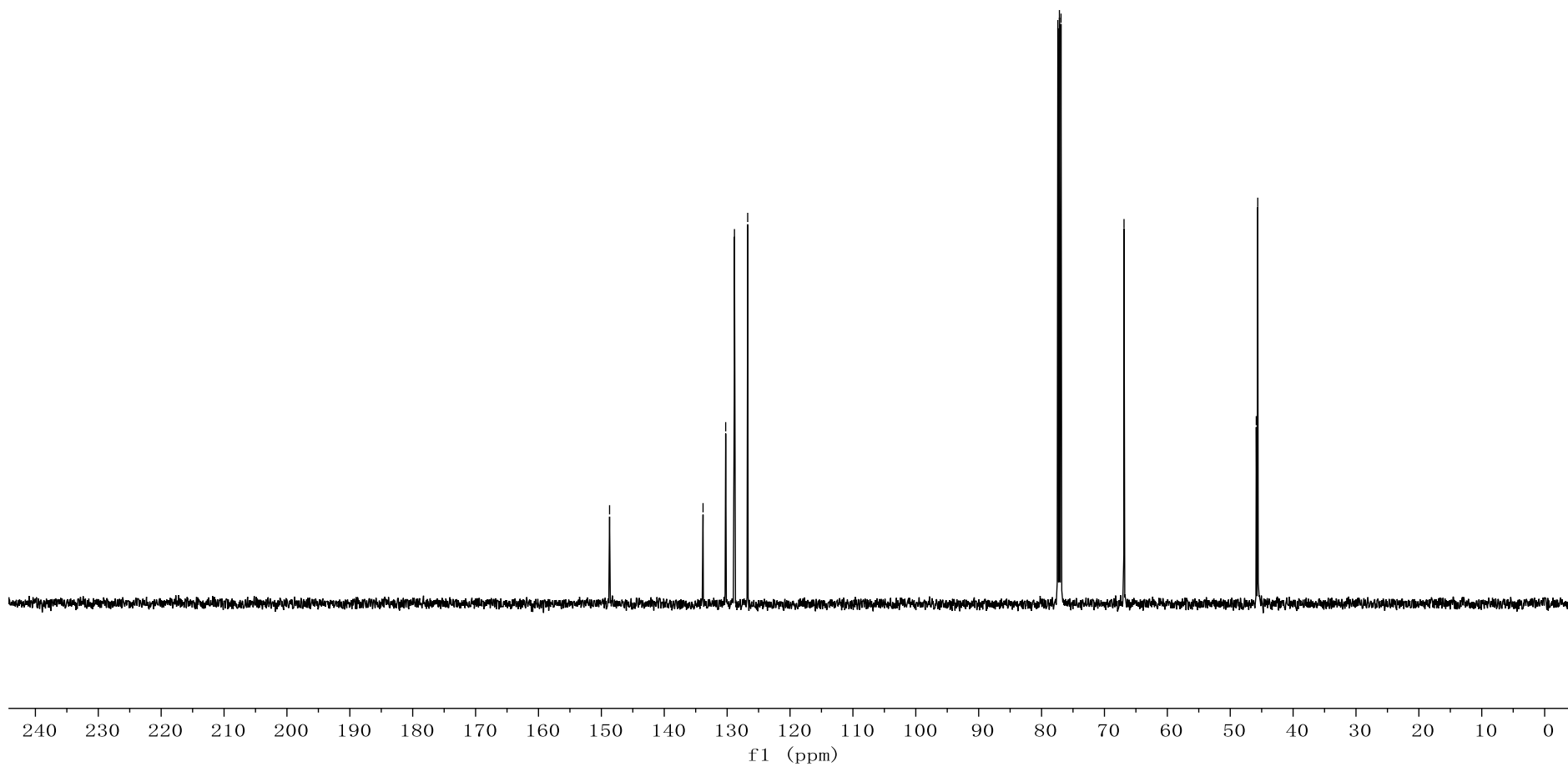


12

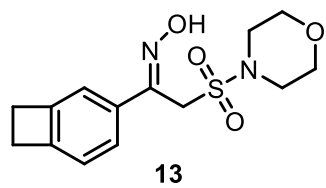
— 148.70
/ 133.83
/ 130.23
/ 128.84
/ 126.73

77.41
/ 77.16
/ 76.91
— 66.88

45.86
/ 45.62

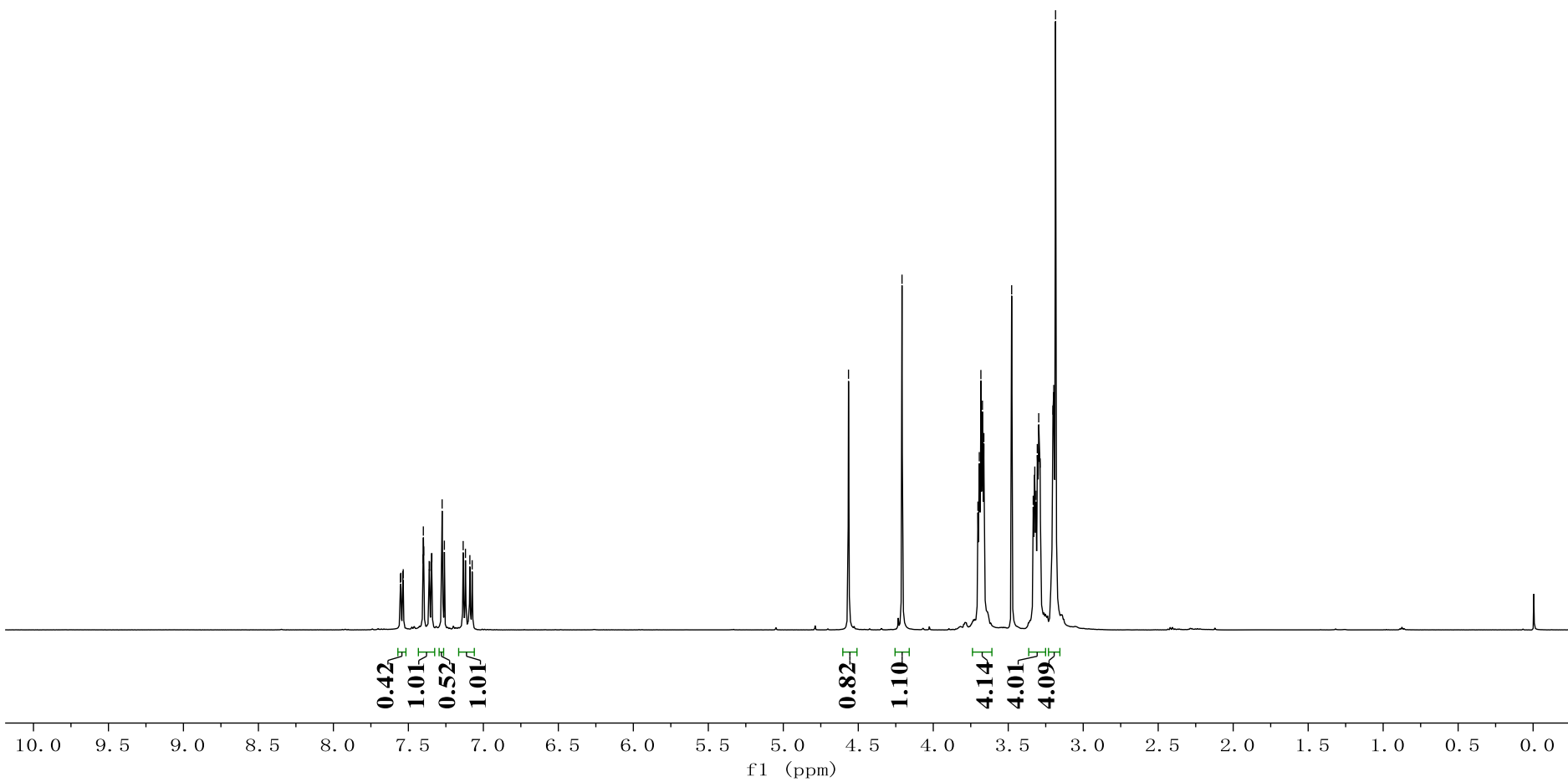


¹H NMR (500 MHz, CDCl₃) for 13

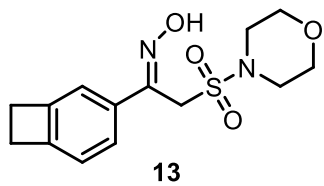


7.55
7.55
7.54
7.53
7.40
7.40
7.40
7.36
7.36
7.35
7.34
7.27
7.26
7.13
7.12
7.09
7.07

4.57
4.21
3.70
3.69
3.68
3.67
3.66
3.48
3.33
3.33
3.32
3.31
3.31
3.30
3.30
3.29
3.29
3.20
3.20
3.18

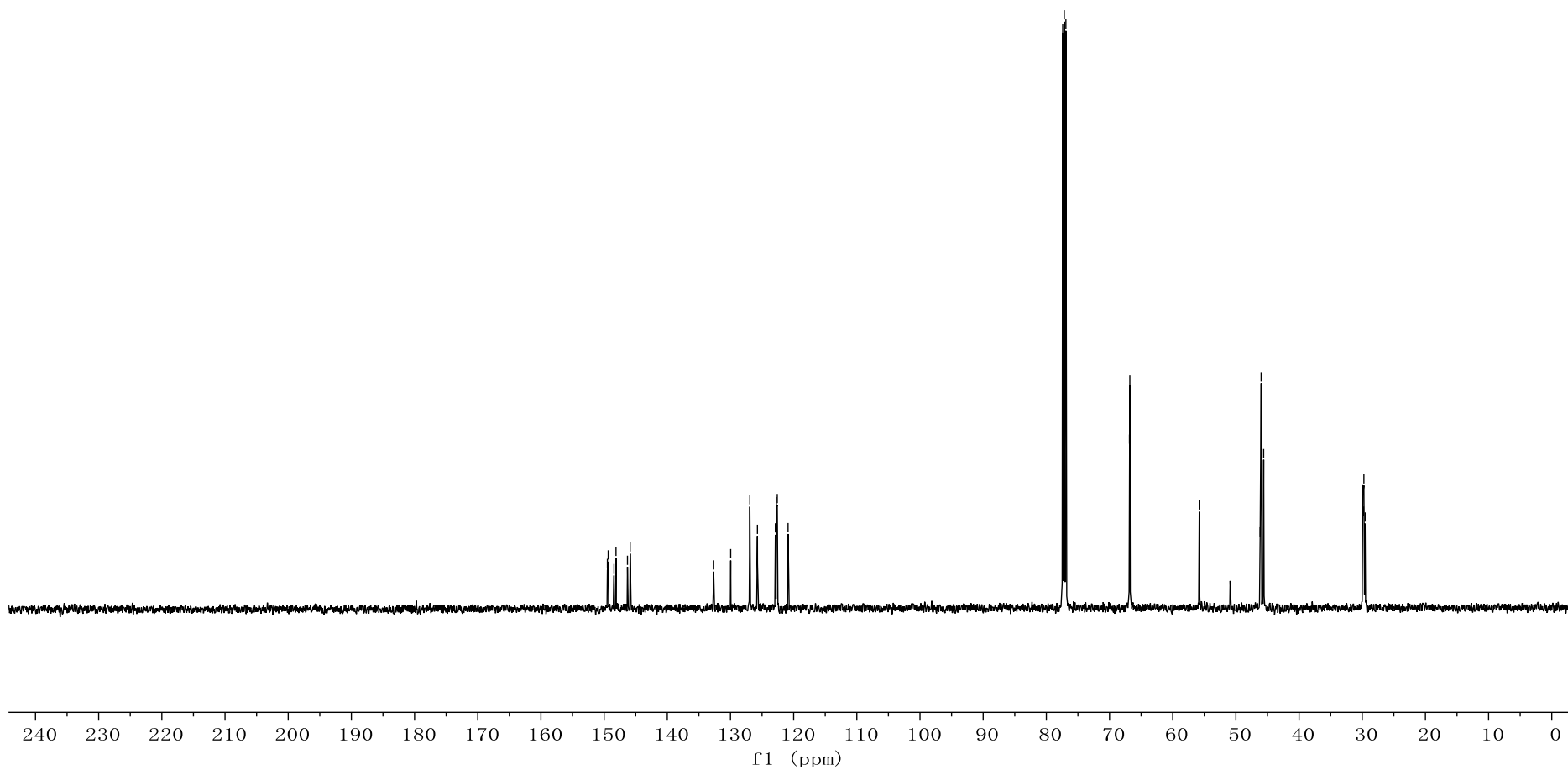


¹³C NMR (125 MHz, CDCl₃) for 13

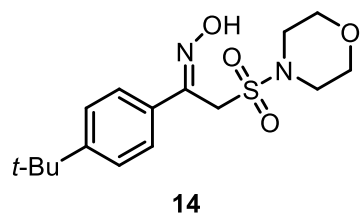


149.48
149.35
148.44
148.12
146.30
145.88
132.66
129.96
126.93
125.74
122.89
122.73
122.59
120.88

77.41
77.16
76.91
66.85
66.79
55.80
46.16
46.00
45.60
29.92
29.79
29.74
29.55



¹H NMR (500 MHz, CDCl₃) for 14



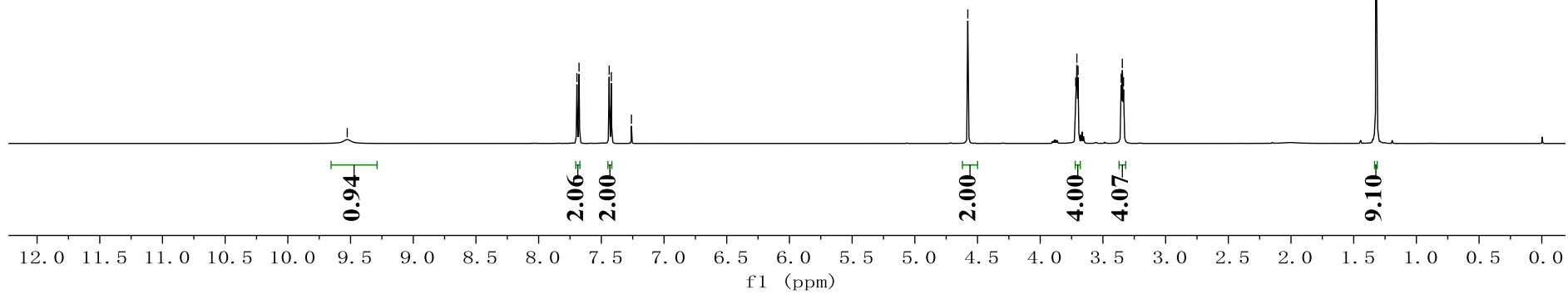
— 9.53

7.69
7.68
7.44
7.42
7.26

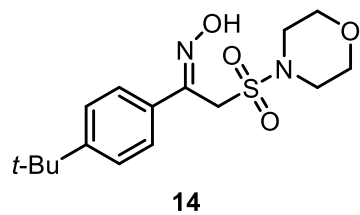
— 4.58

3.72
3.71
3.70
3.35
3.35
3.34

— 1.32



¹³C NMR (125 MHz, CDCl₃) for 14

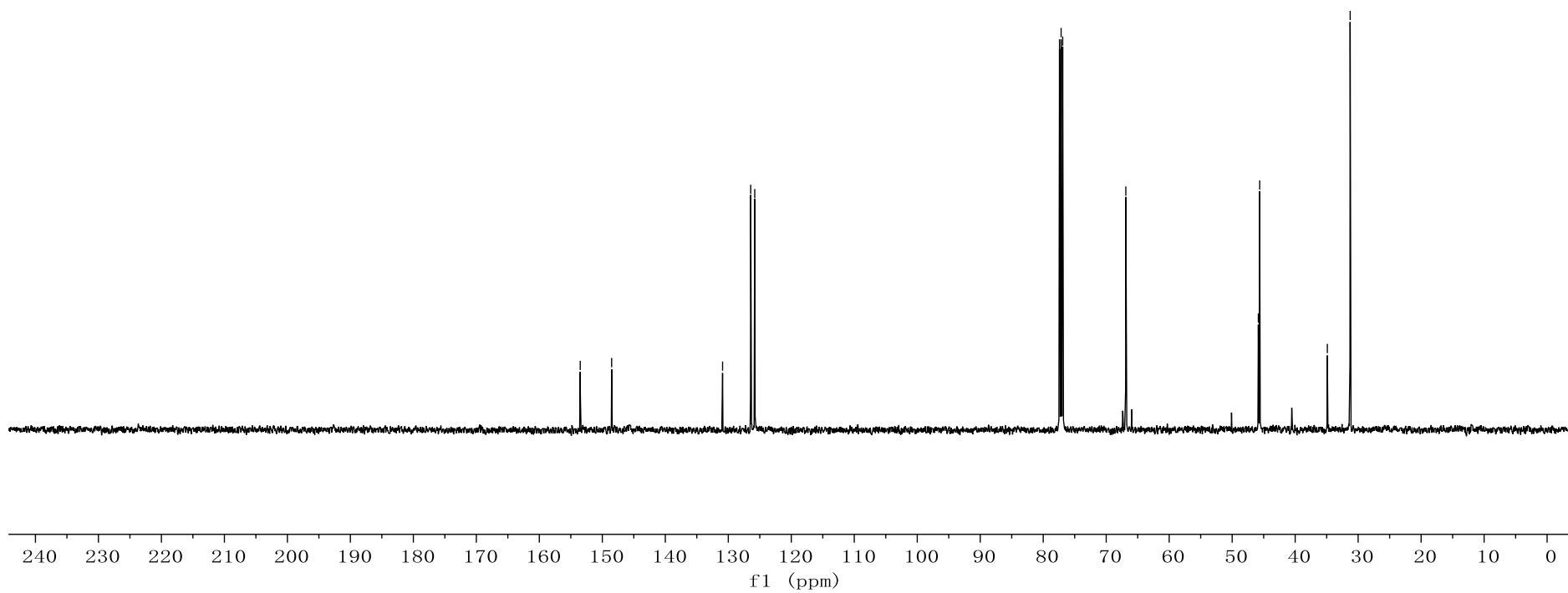


— 153.51
— 148.52

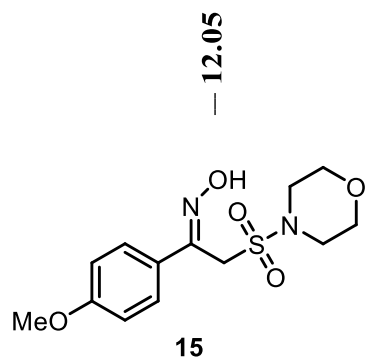
∩ 130.91
∩ 126.44
∩ 125.80

∩ 77.41
∩ 77.16
∩ 76.91
— 66.88

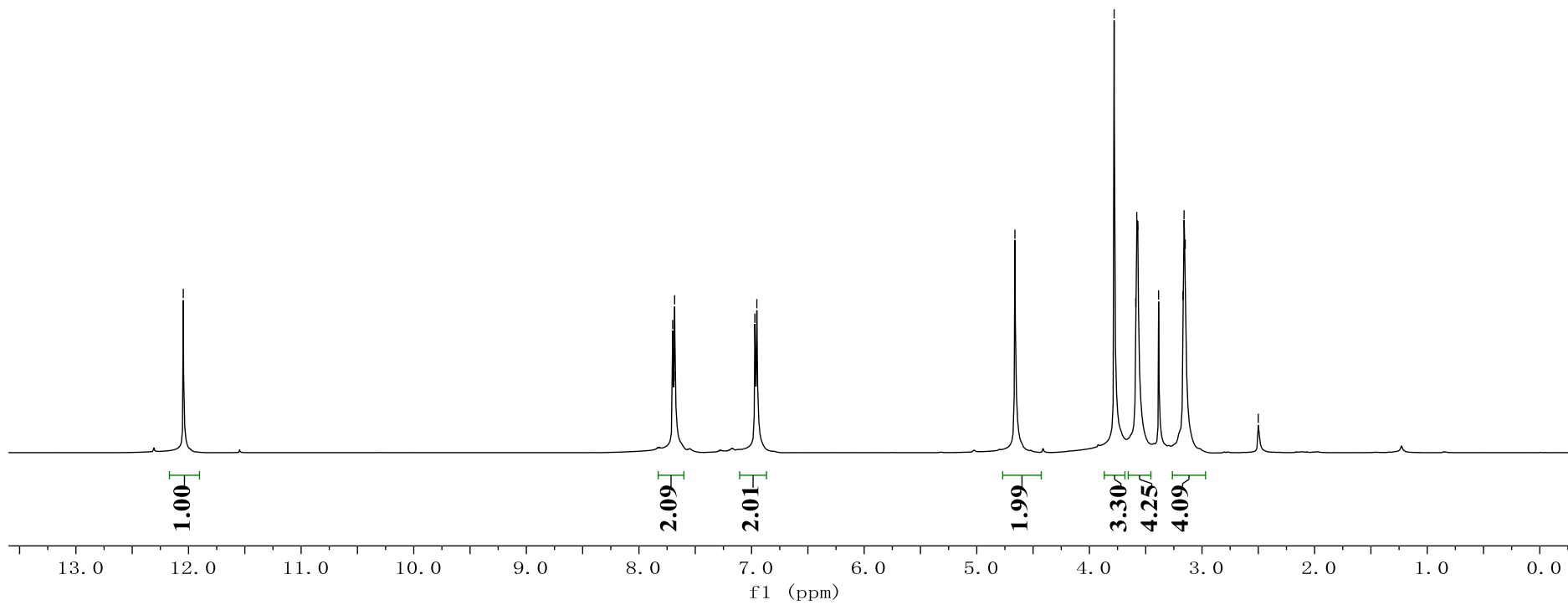
∩ 45.85
∩ 45.63
— 34.89
— 31.27



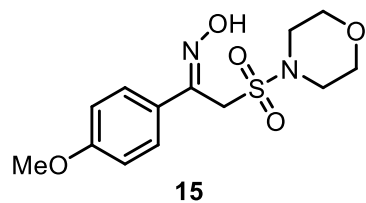
¹H NMR (500 MHz, DMSO-*d*₆) for 15



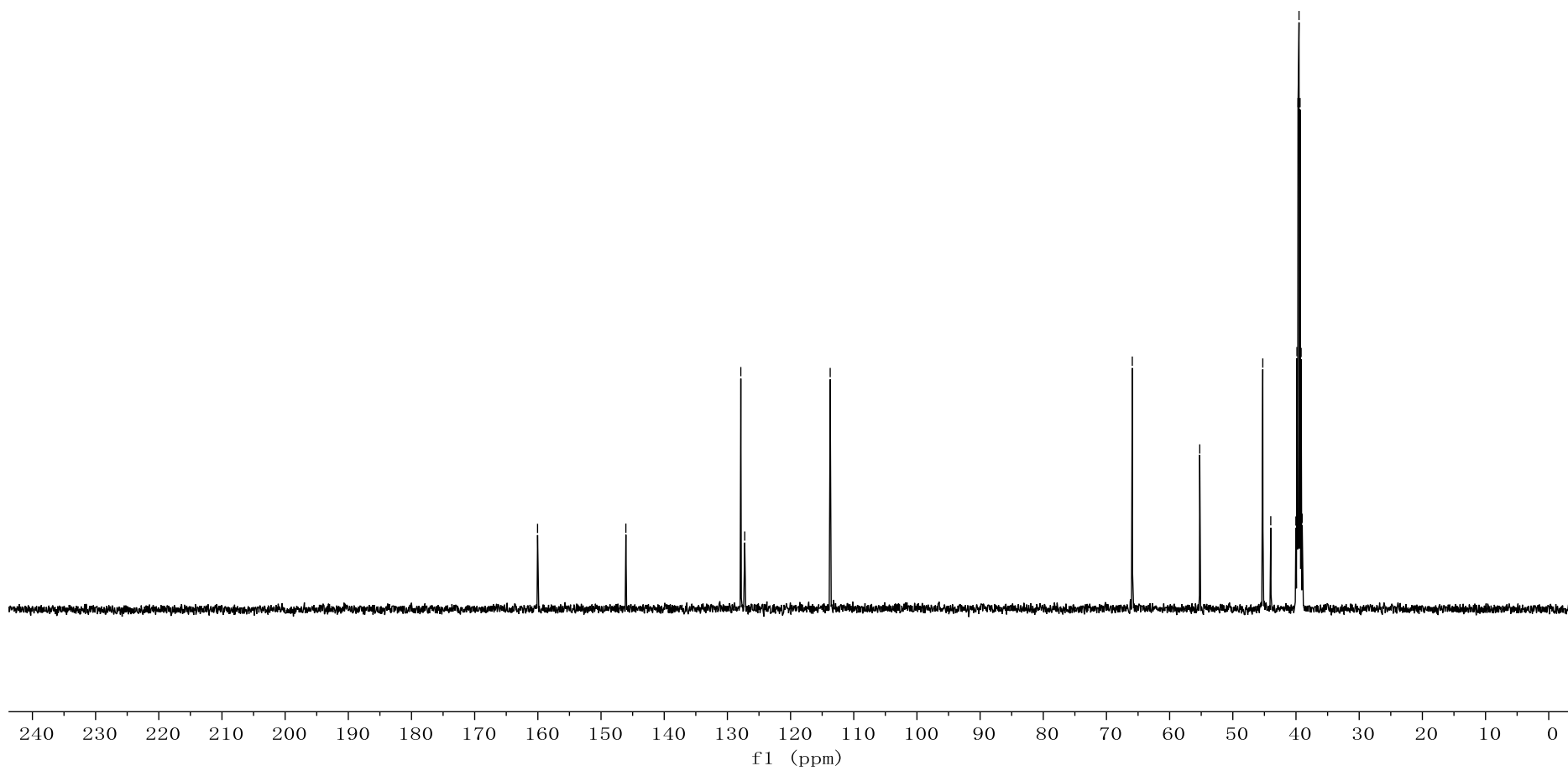
12.05
7.70 7.70 7.68 7.68 6.97 6.97 6.95
4.66 3.78 3.59 3.58 3.57 3.39 3.17 3.16 3.15 2.50



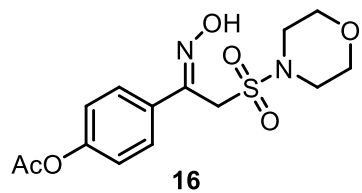
¹³C NMR (125 MHz, DMSO-*d*₆) for 15



- 160.05
- 146.07
- { 127.87
- { 127.25
- 113.73
- 65.91
- 55.24
- ↙ 45.26
- ↘ 43.99
- 40.02
- 39.85
- 39.69
- 39.52
- 39.35
- 39.19
- 39.02



¹H NMR (500 MHz, CD₃CN) for 16



— 9.94

{ 7.77
7.75

{ 7.15
7.13

— 4.57

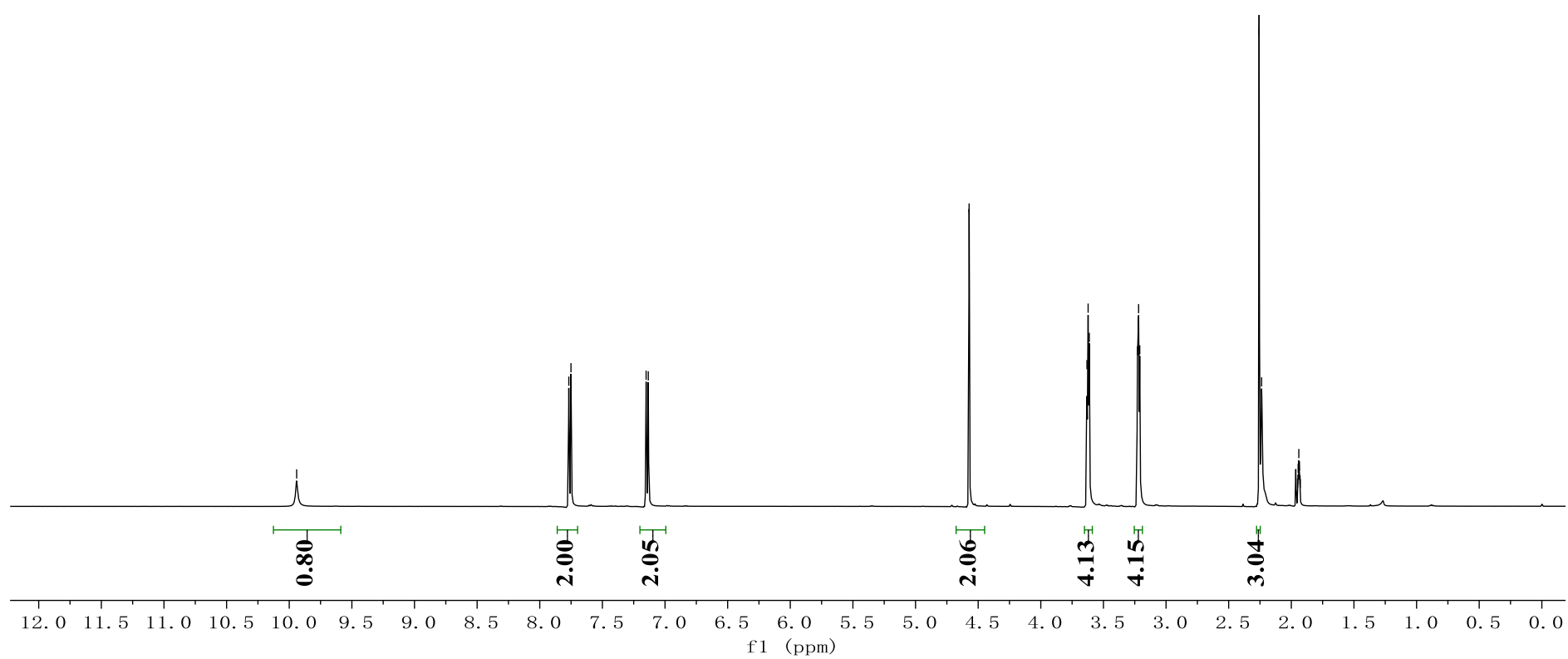
{ 3.63
3.62
3.61

{ 3.23
3.22
3.21

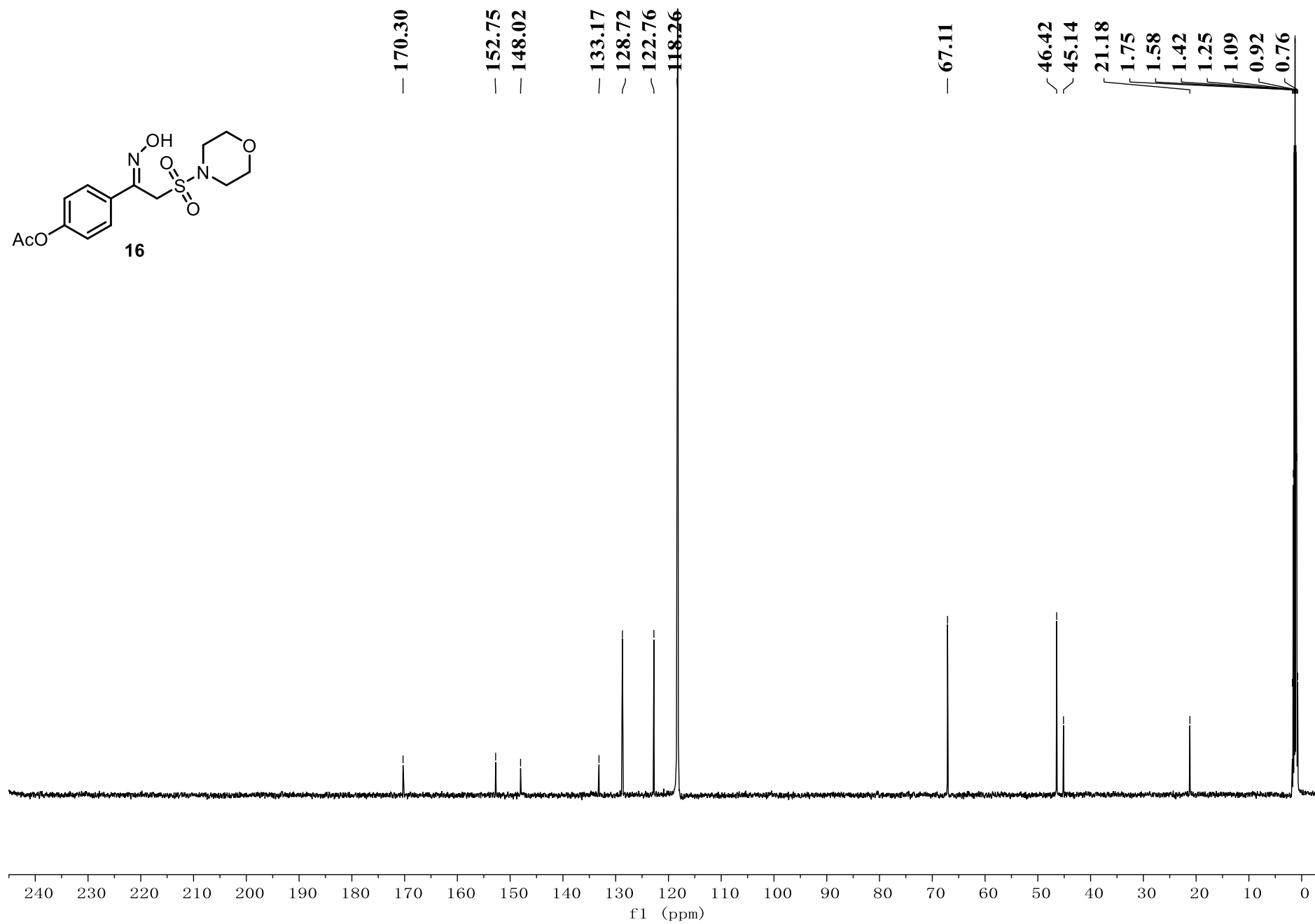
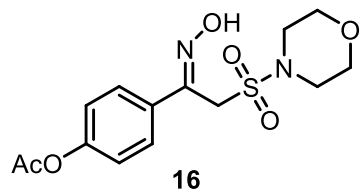
{ 2.26
2.24

{ 1.95
1.95

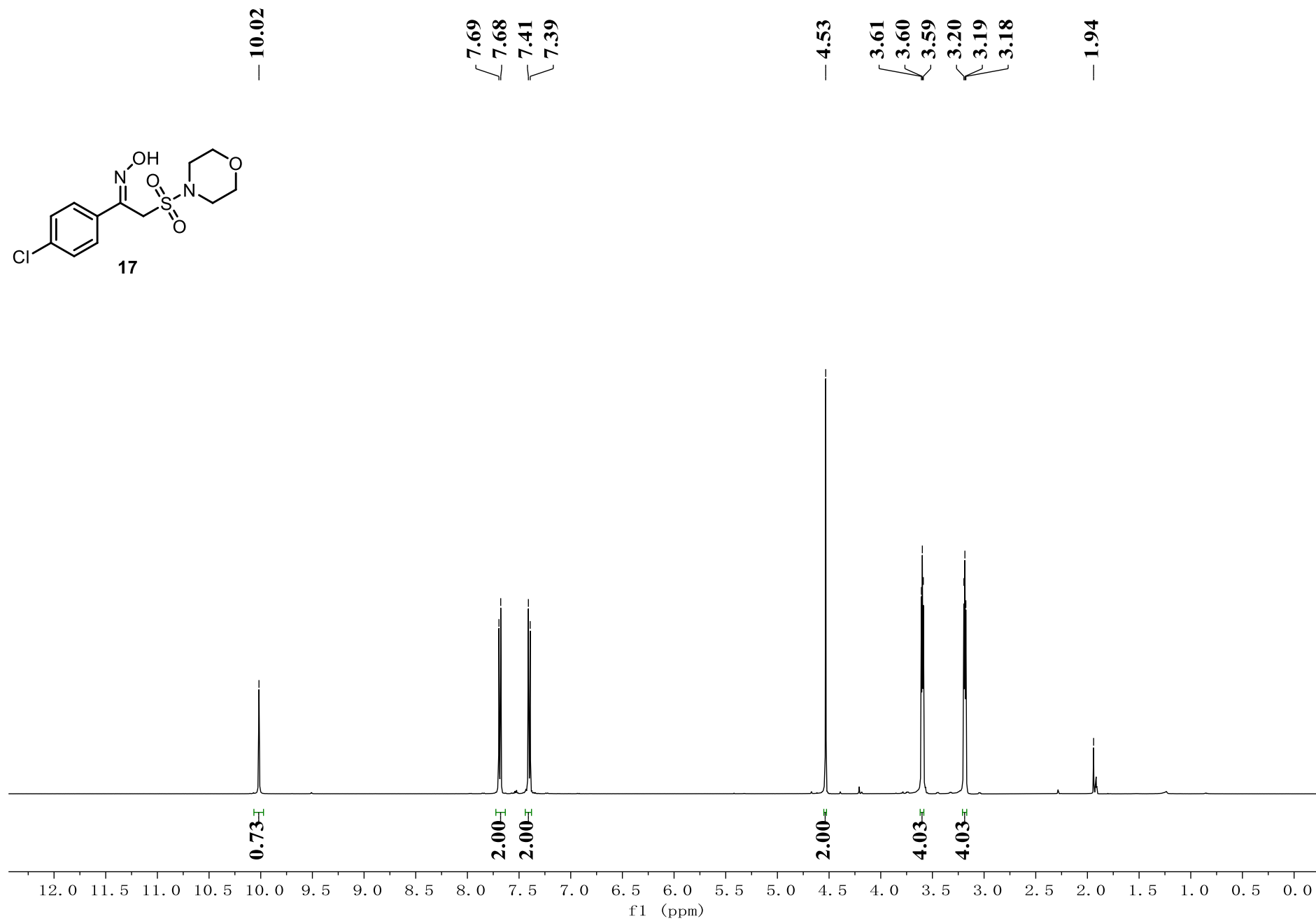
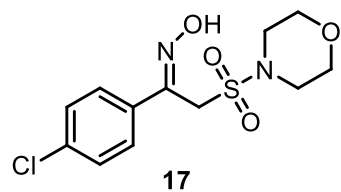
{ 1.94
1.93
1.93
1.93



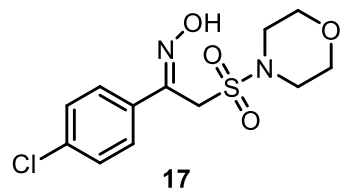
¹³C NMR (125 MHz, CD₃CN) for 16



¹H NMR (500 MHz, CD₃CN) for 17



¹³C NMR (125 MHz, CD₃CN) for 17

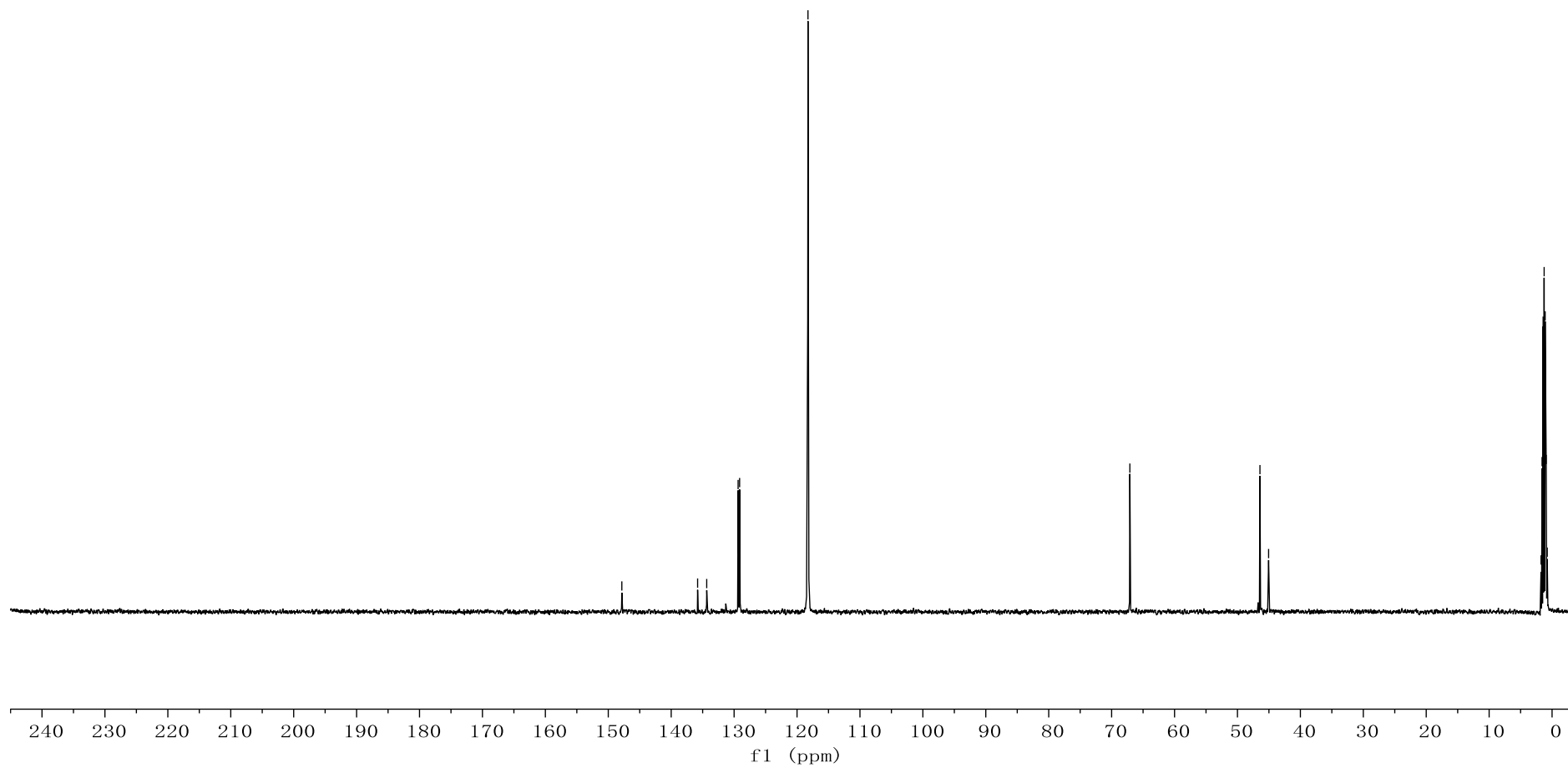


— 147.84
└ 135.80
└ 134.35
└ 129.36
└ 129.13
— 118.26

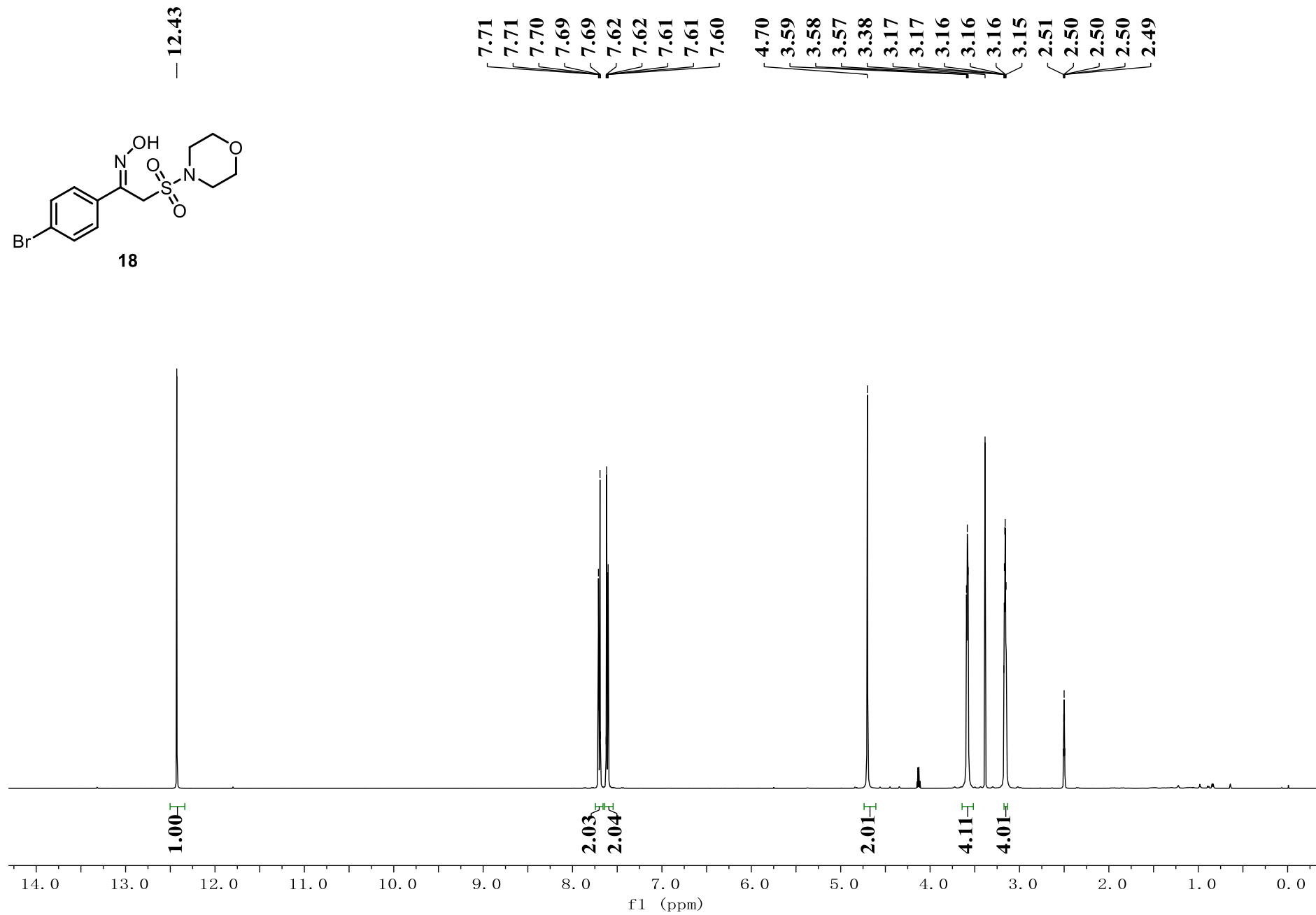
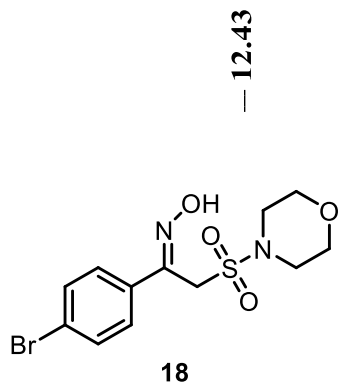
— 67.10

└ 46.43
└ 45.05

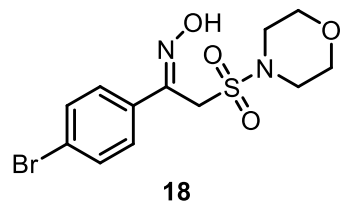
└ 1.76
└ 1.59
└ 1.42
└ 1.26
└ 1.09
└ 0.93
└ 0.76



¹H NMR (125 MHz, DMSO-d₆) for 18



¹³C NMR (125 MHz, DMSO-*d*₆) for 18



— 145.86

~ 134.08

~ 131.29

~ 128.51

~ 122.62

— 65.88

45.25

43.79

40.02

39.86

39.78

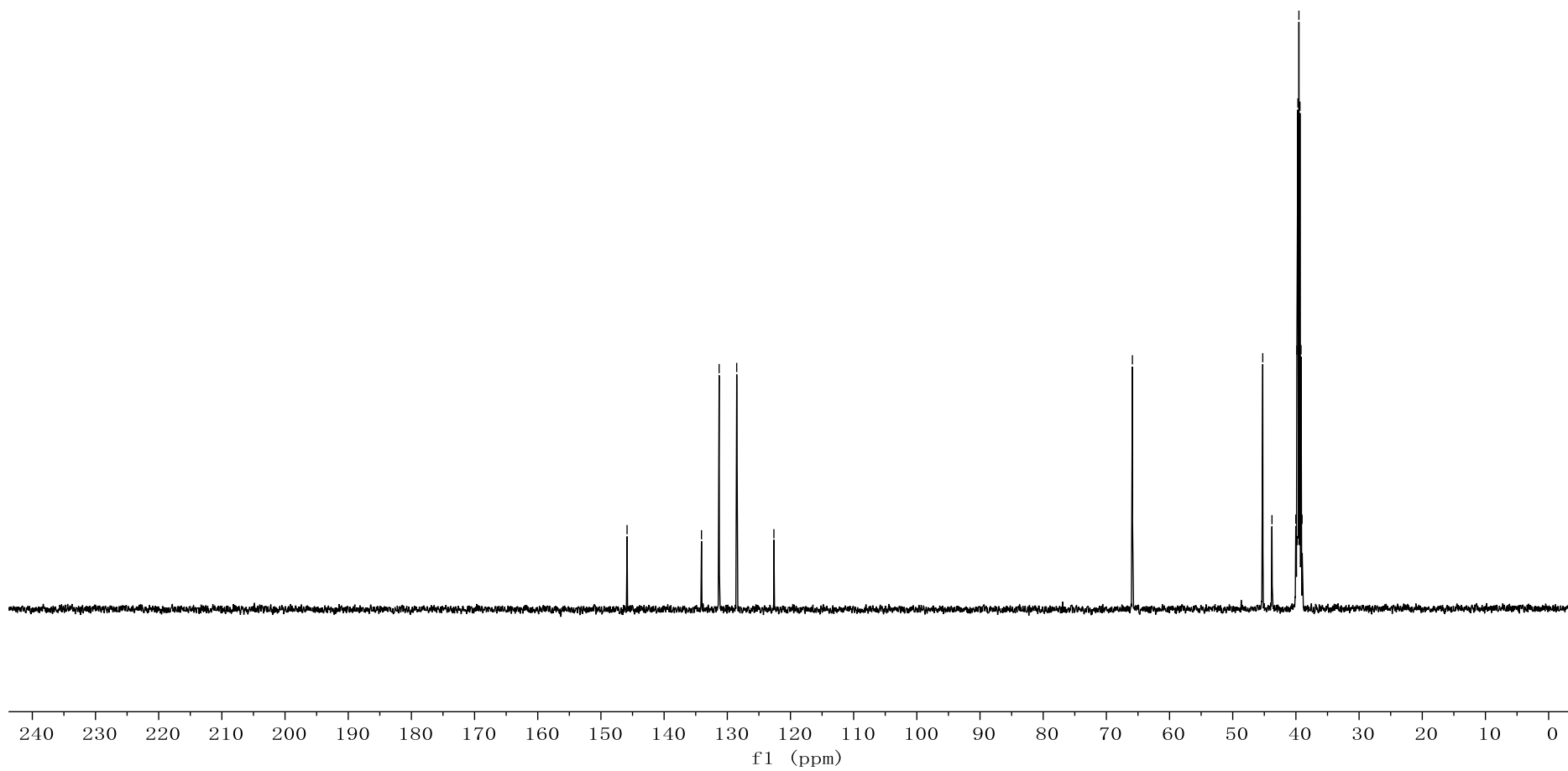
39.69

39.52

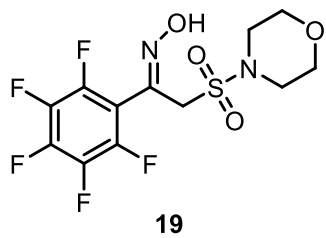
39.36

39.19

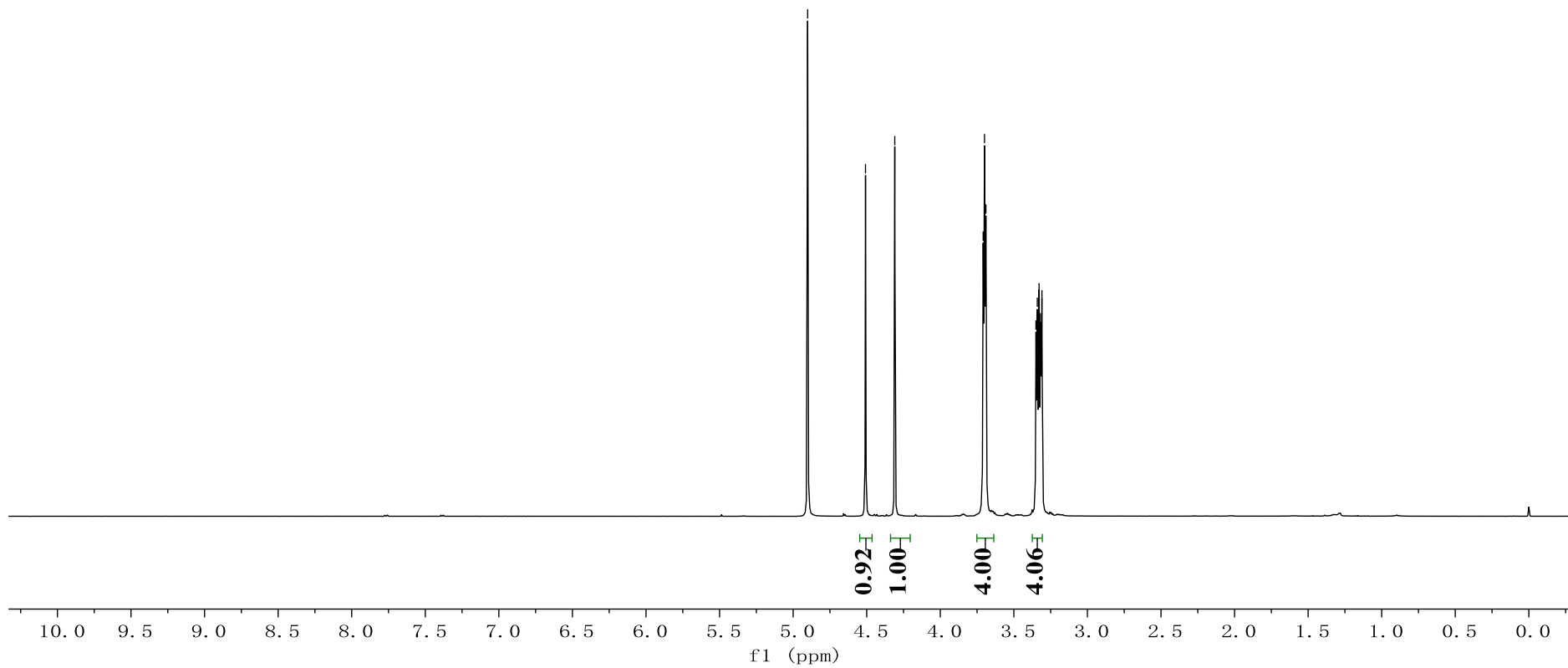
39.02



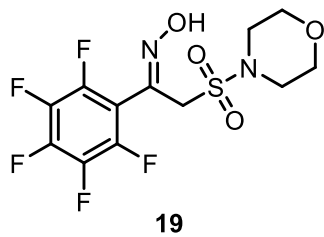
¹H NMR (500 MHz, CD₃OD) for 19



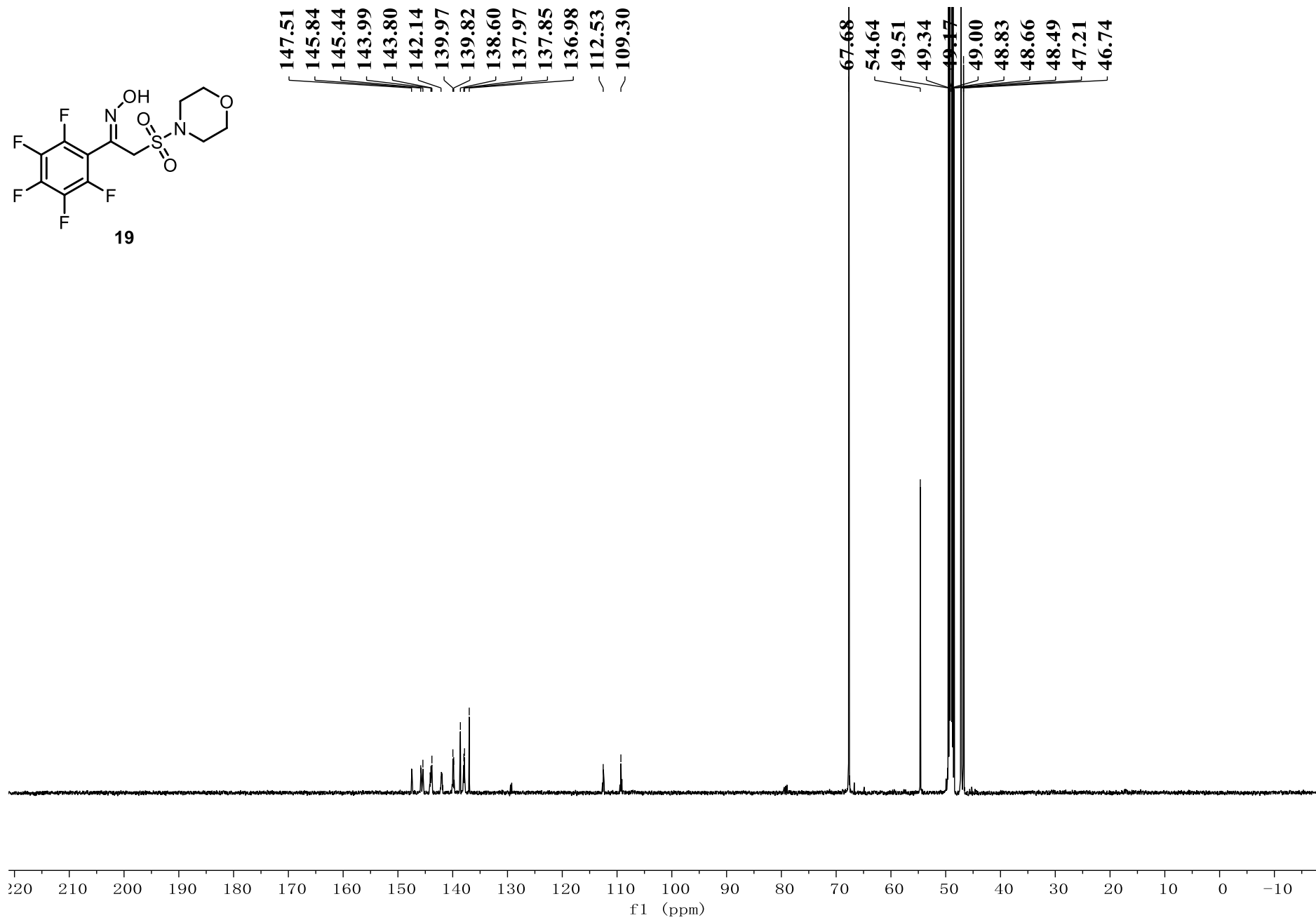
4.90
4.51
4.31
3.71
3.70
3.69
3.35
3.34
3.33
3.33
3.32
3.32
3.32
3.31



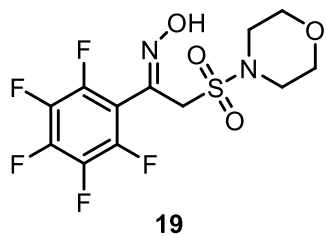
¹³C NMR (125 MHz, CD₃OD) for 19



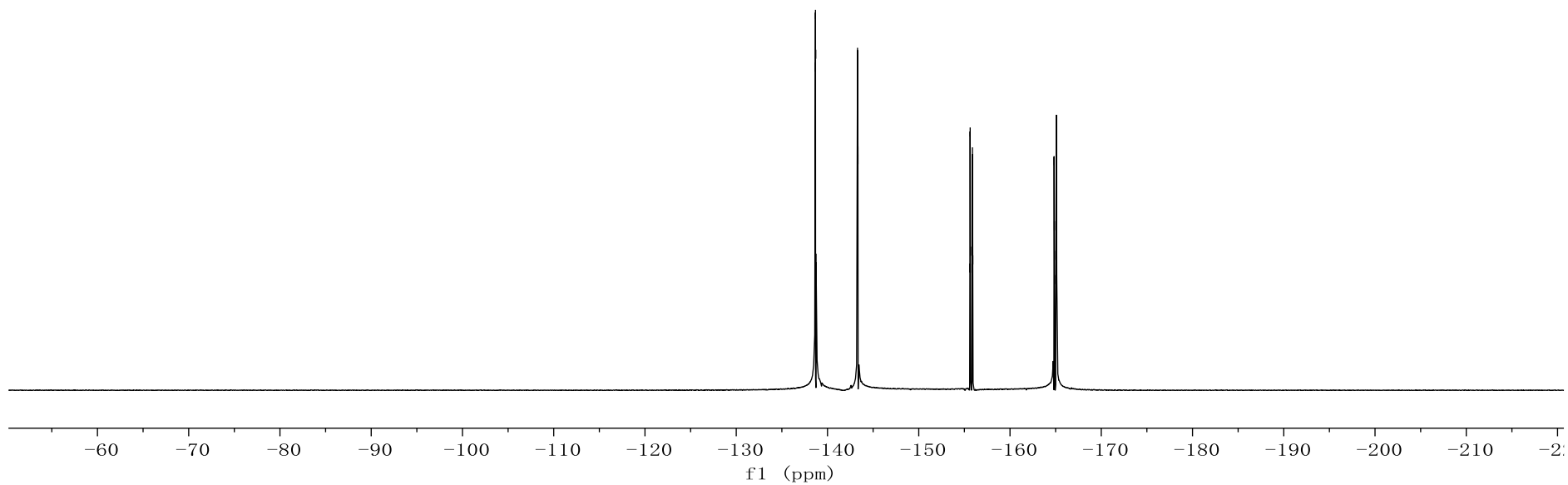
- 147.51
- 145.84
- 145.44
- 143.99
- 143.80
- 142.14
- 139.97
- 139.82
- 138.60
- 137.97
- 137.85
- 136.98
- 112.53
- 109.30



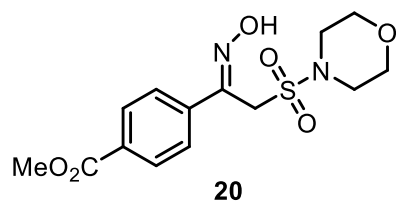
¹⁹F NMR (470 MHz, CD₃OD) for 19



-138.67
-138.68
-138.71
-138.75
-138.75
-143.27
-143.29
-143.32
-143.34
-143.34
-155.59
-155.63
-155.68
-155.84
-155.88
-155.93
-164.84
-164.87
-164.88
-165.03
-165.06
-165.08
-165.12



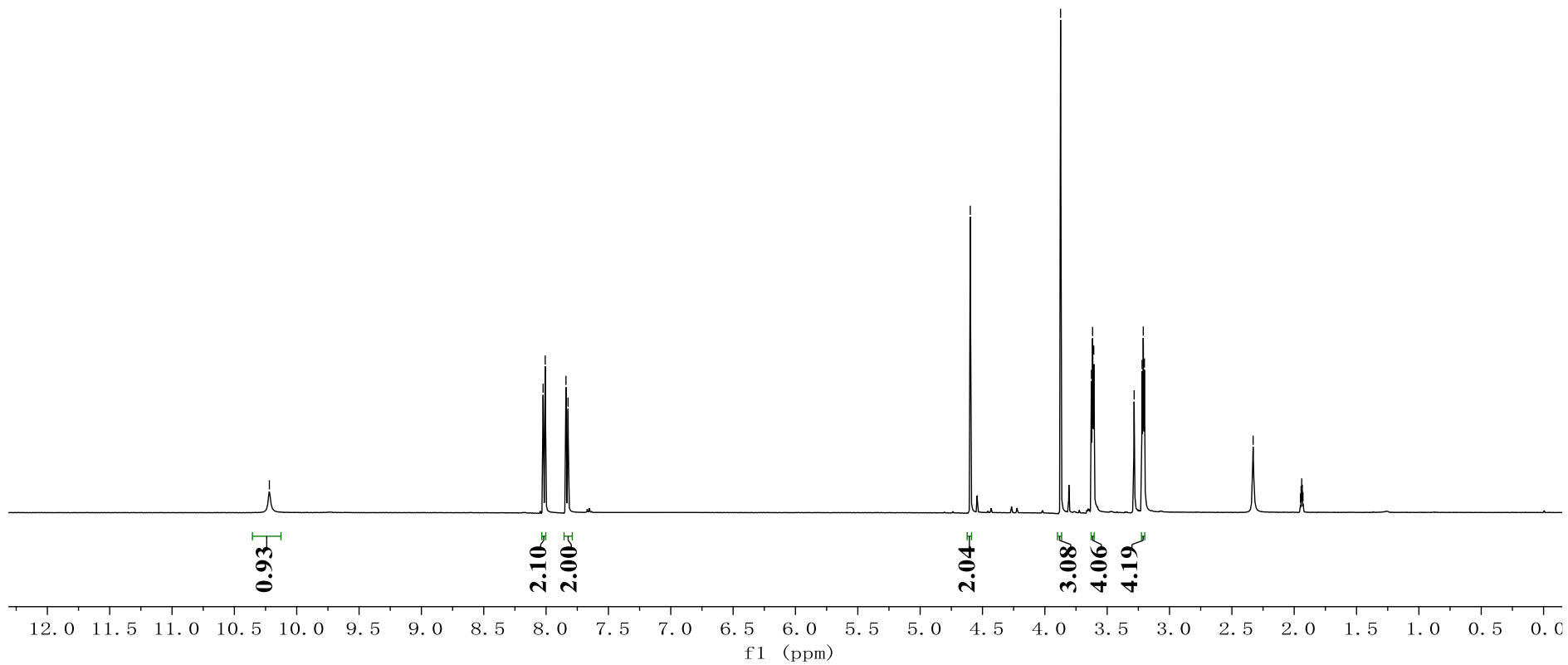
¹H NMR (500 MHz, CD₃CN) for 20



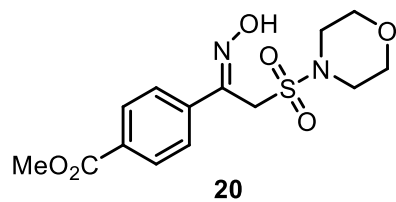
— 10.22

8.02
8.01
7.84
7.82

— 4.60
3.87
3.63
3.62
3.61
3.28
3.22
3.21
3.20
2.33
1.95
1.94
1.94
1.93
1.93



¹³C NMR (125 MHz, CD₃CN) for 20



— 167.13

~ 148.15

~ 139.80

~ 131.78

~ 130.17

~ 127.65

~ 118.26

— 67.09

~ 52.76

~ 46.42

~ 45.09

1.75

1.58

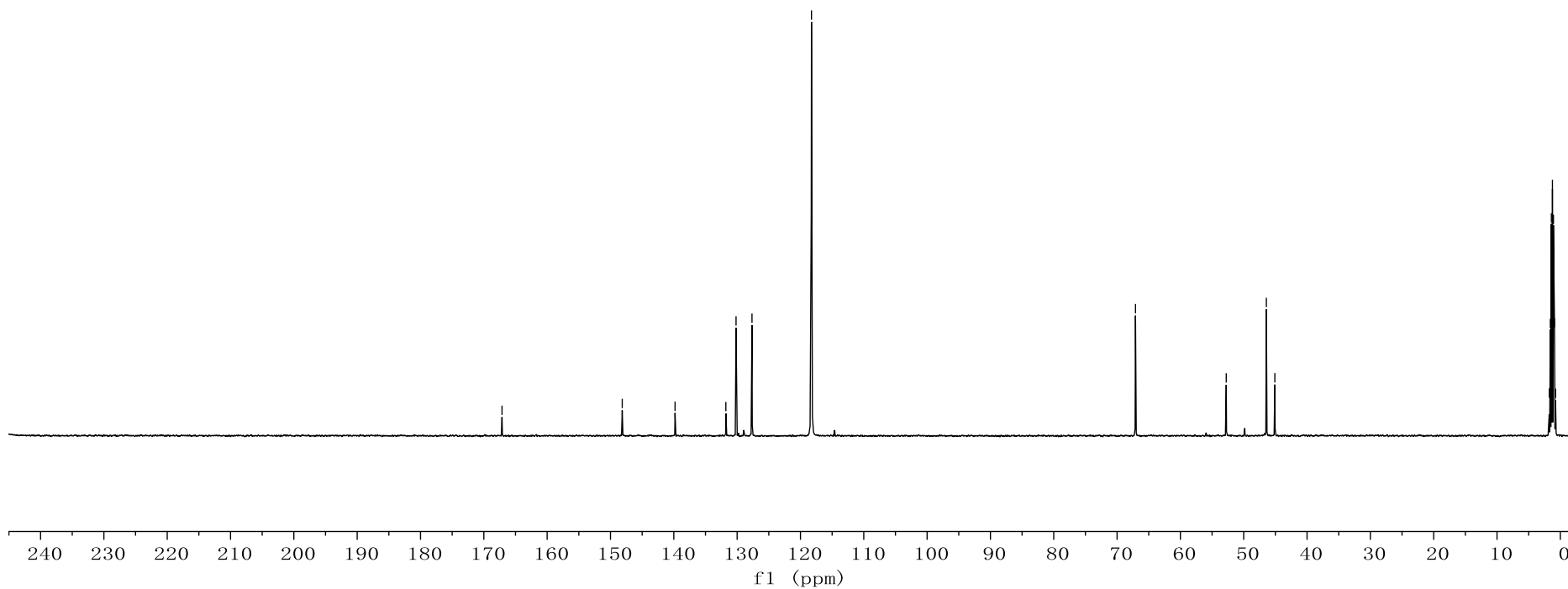
1.42

1.25

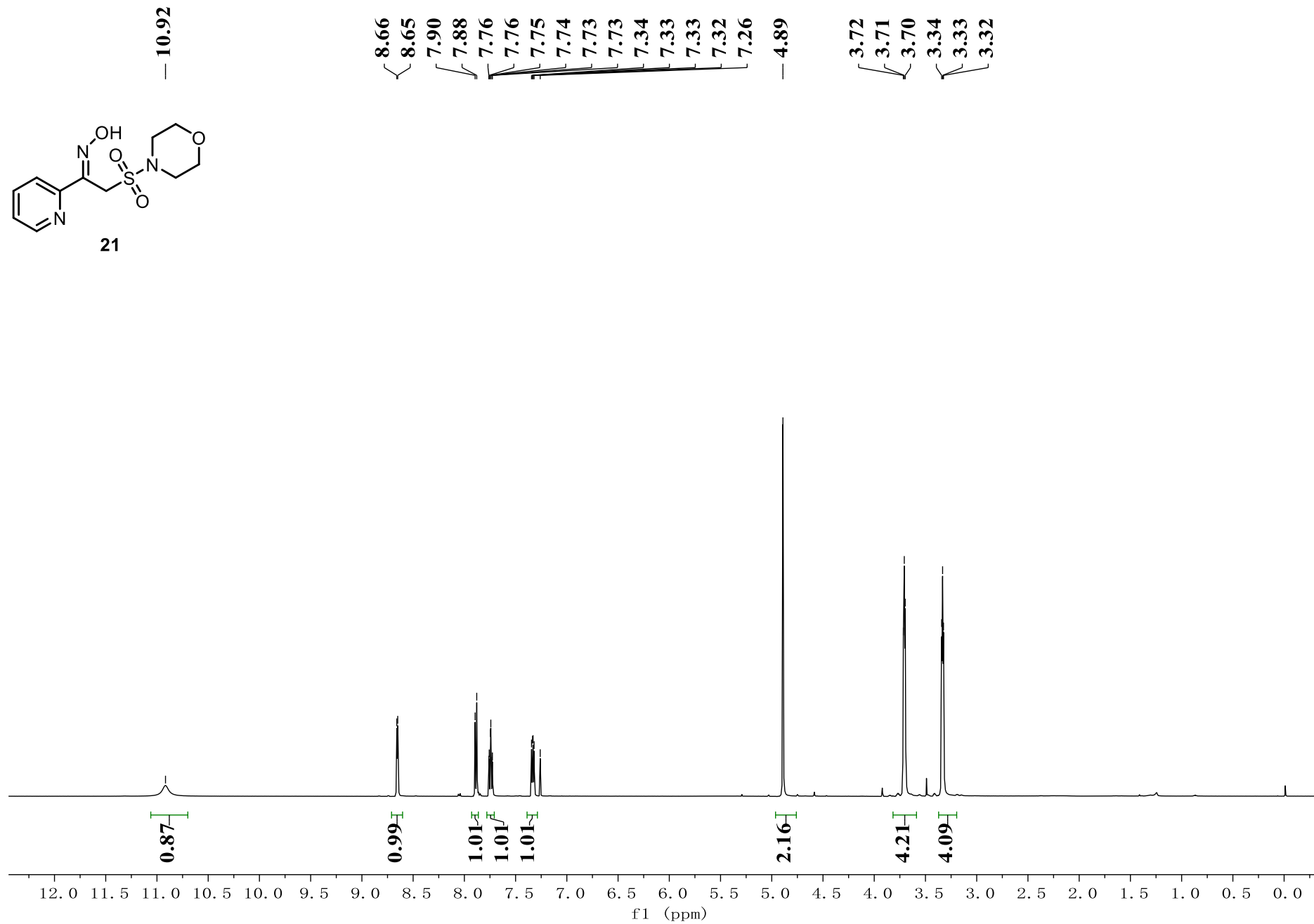
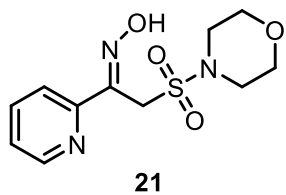
1.09

0.92

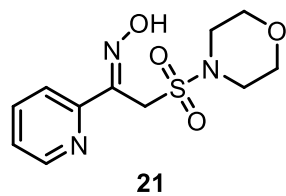
0.76



¹H NMR (500 MHz, CDCl₃) for 21



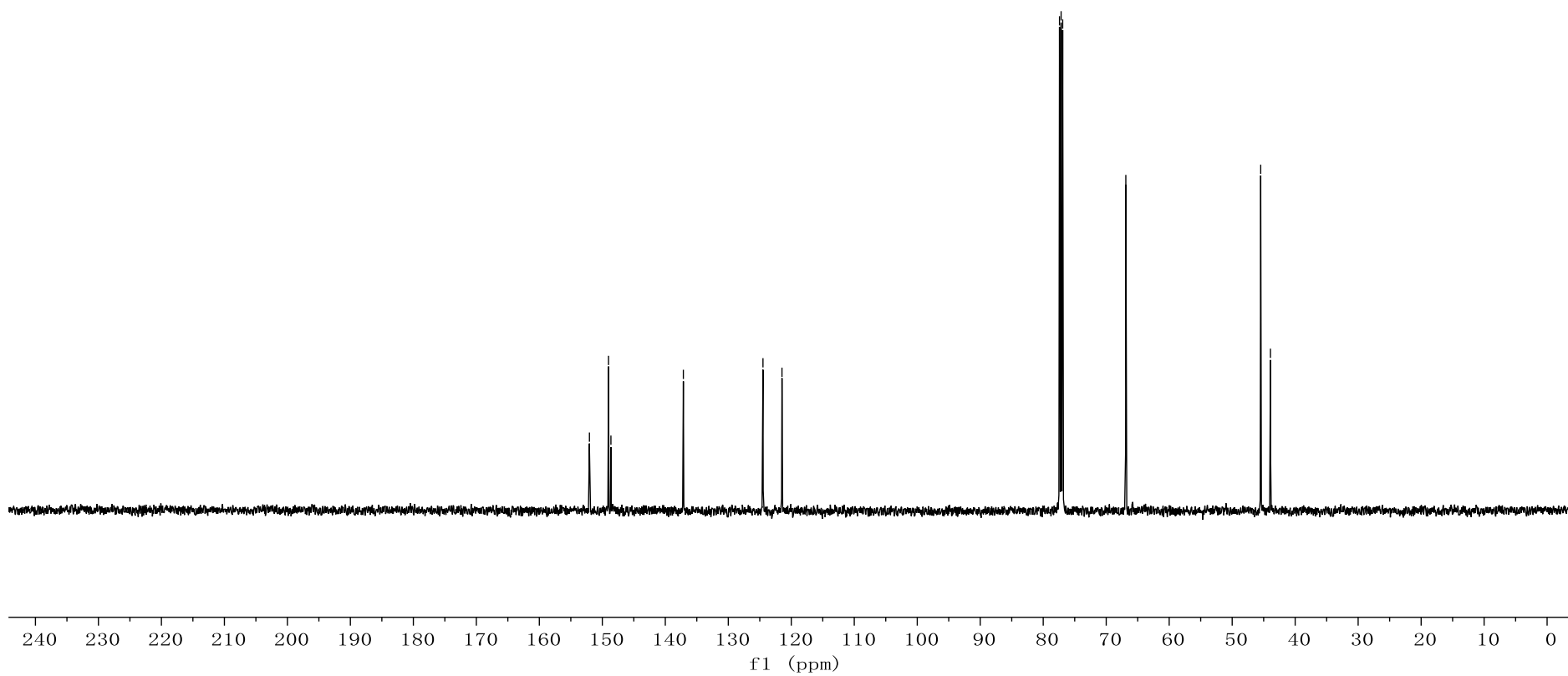
¹³C NMR (125 MHz, CDCl₃) for 21



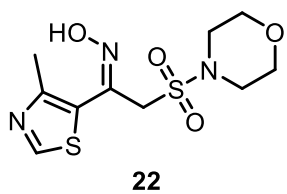
152.05
149.02
148.63
— 137.13
~ 124.49
~ 121.48

77.41
77.16
76.91
— 66.89

45.47
43.92



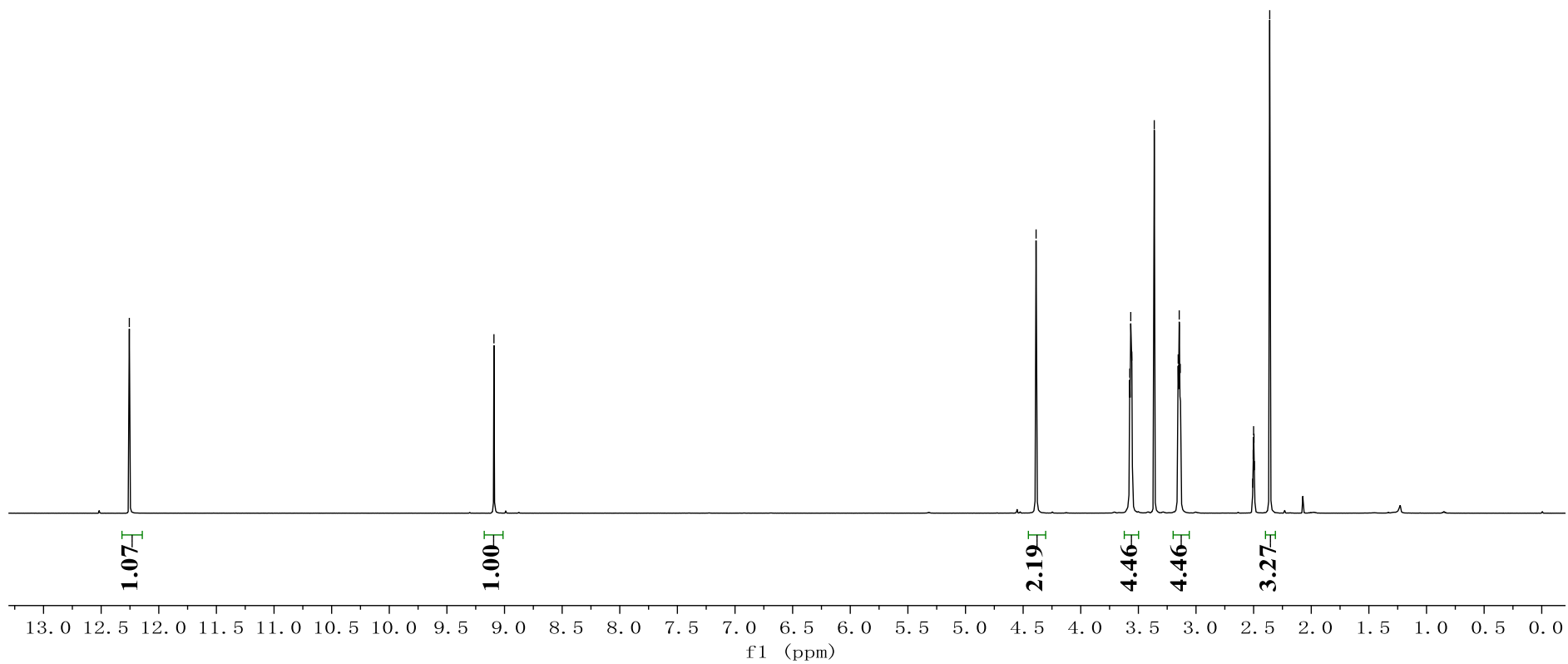
¹H NMR (500 MHz, DMSO-d₆) for 22



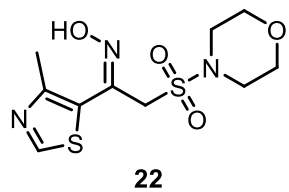
— 12.26

— 9.09

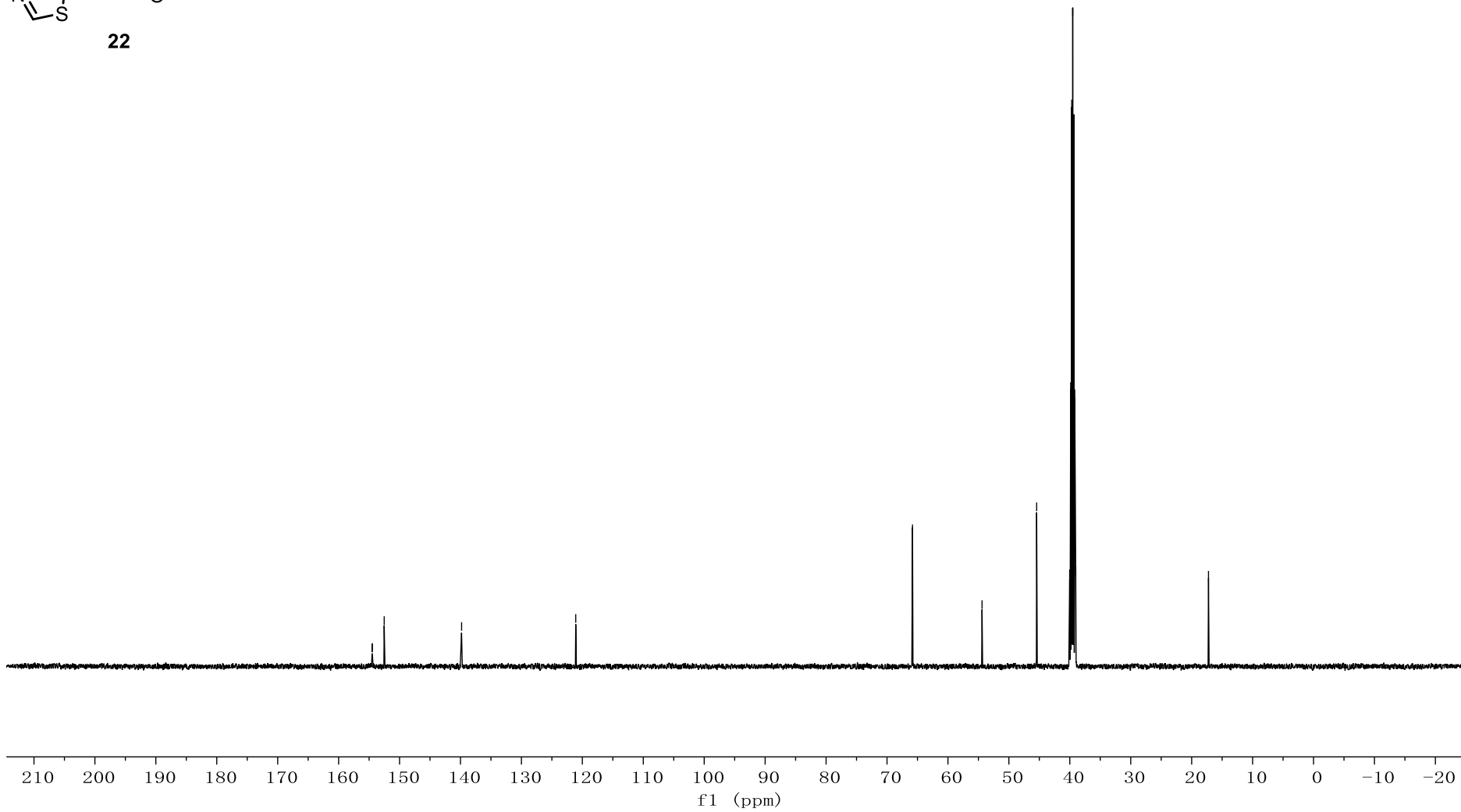
— 4.39
— 3.57
— 3.57
— 3.56
— 3.36
— 3.15
— 3.15
— 3.14
— 2.51
— 2.50
— 2.50
— 2.50
— 2.49
— 2.36



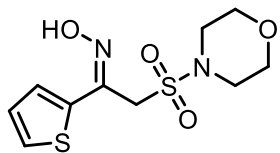
¹³C NMR (125 MHz, DMSO-*d*₆) for 22



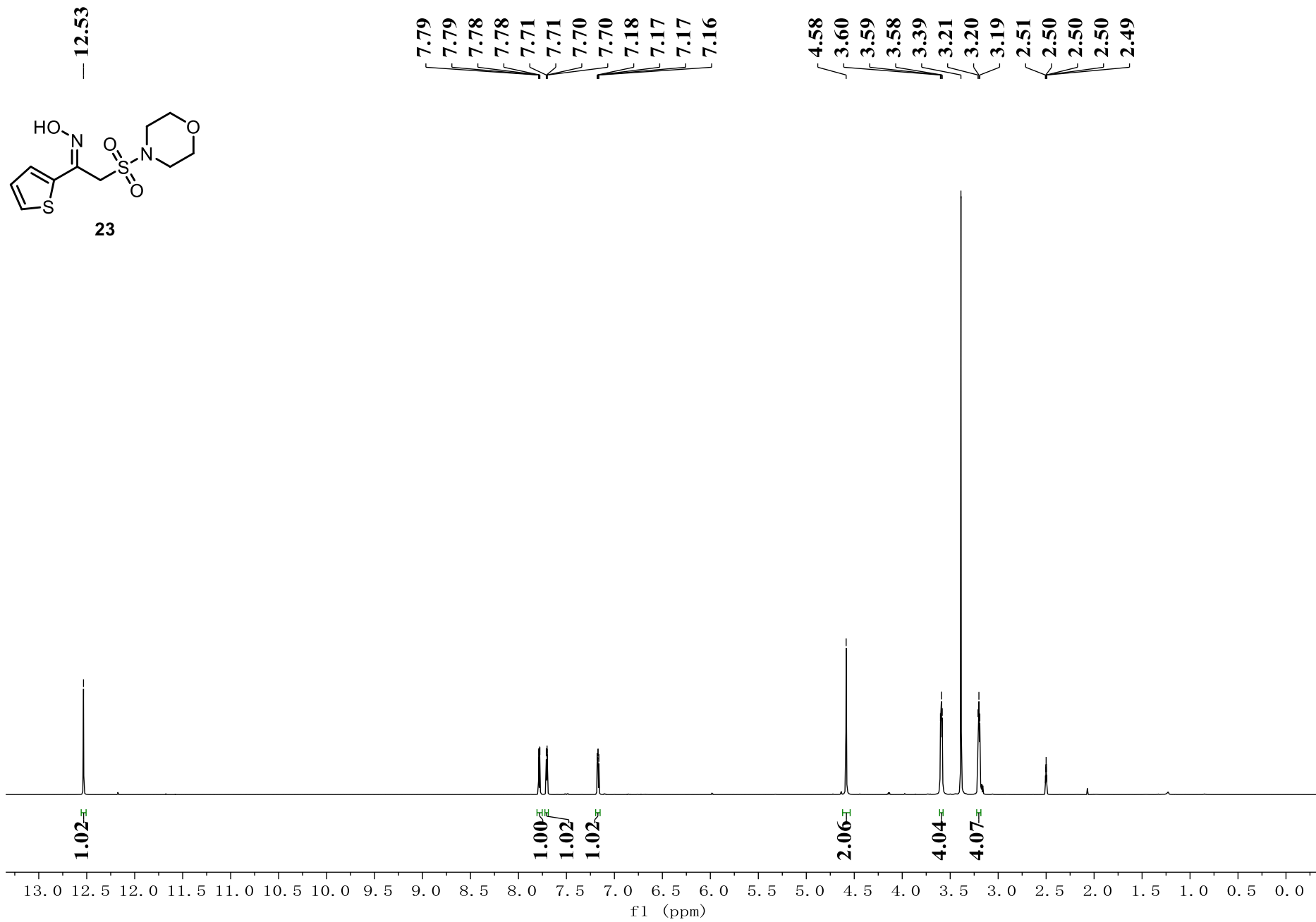
154.52
154.46
152.52
— 139.82
— 121.08
— 65.81
54.40
45.44
40.02
39.85
39.69
39.52
39.35
39.19
39.02
— 17.24



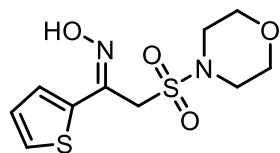
¹H NMR (500 MHz, DMSO-d₆) for 23



23



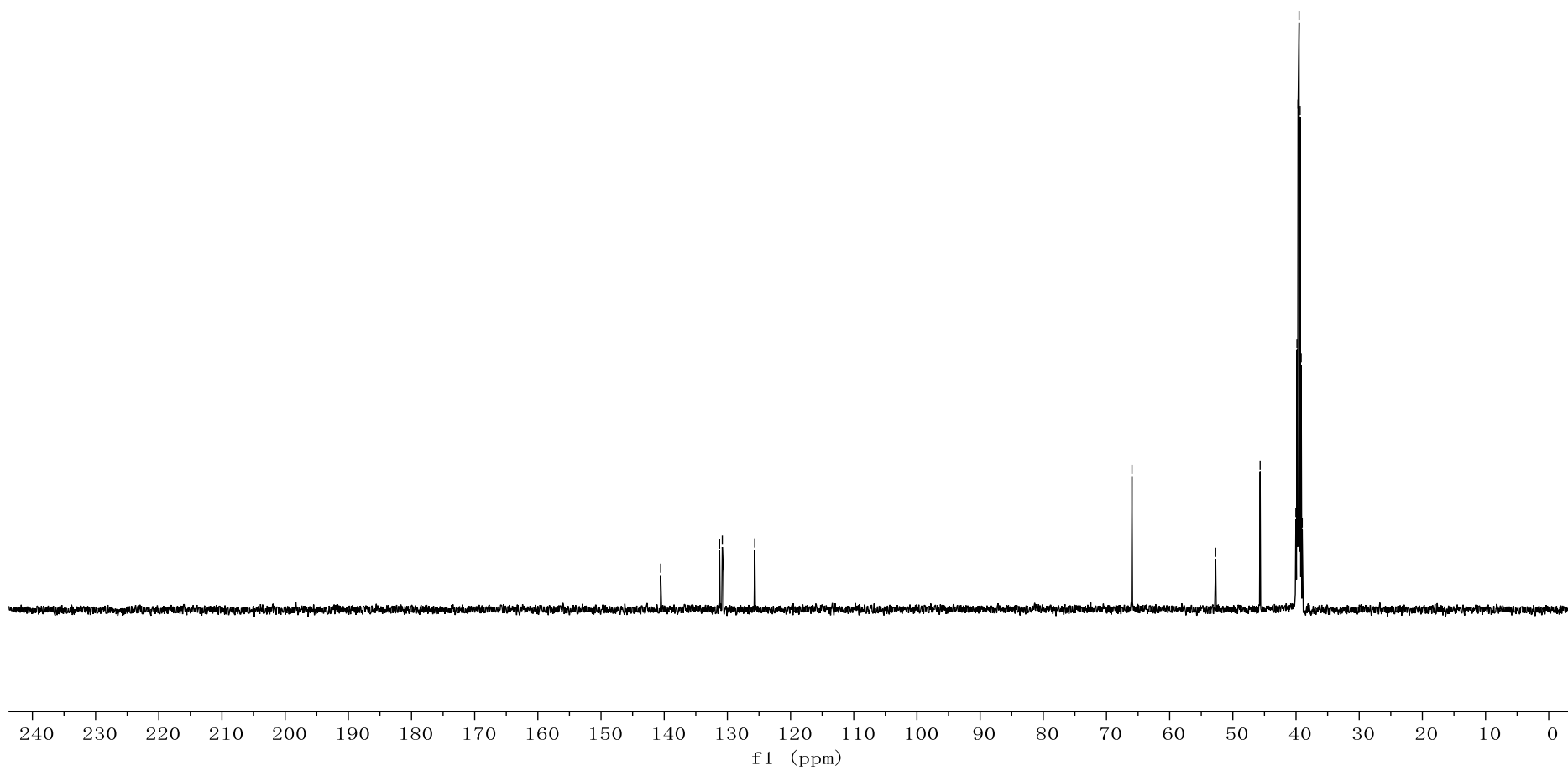
¹³C NMR (125 MHz, DMSO-*d*₆) for 23



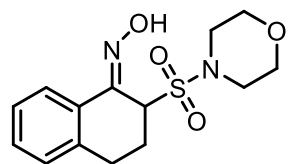
23

140.56
131.23
130.80
130.62
125.67

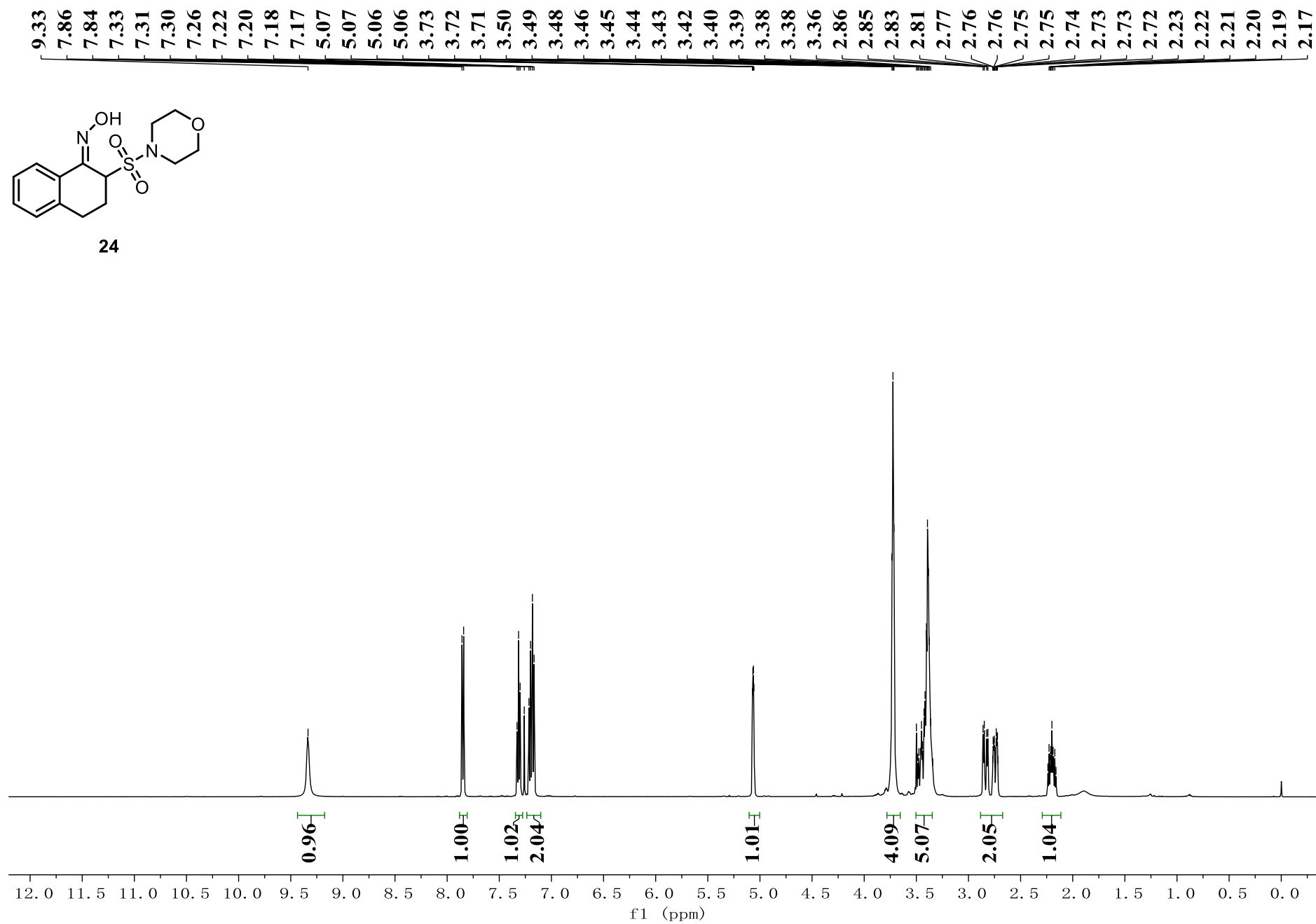
65.97
52.72
45.68
40.02
39.85
39.69
39.52
39.35
39.19
39.02



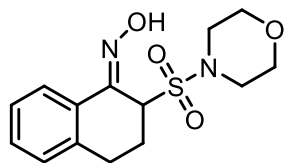
¹H NMR (500 MHz, CDCl₃) for 24



24



¹³C NMR (125 MHz, CDCl₃) for 24



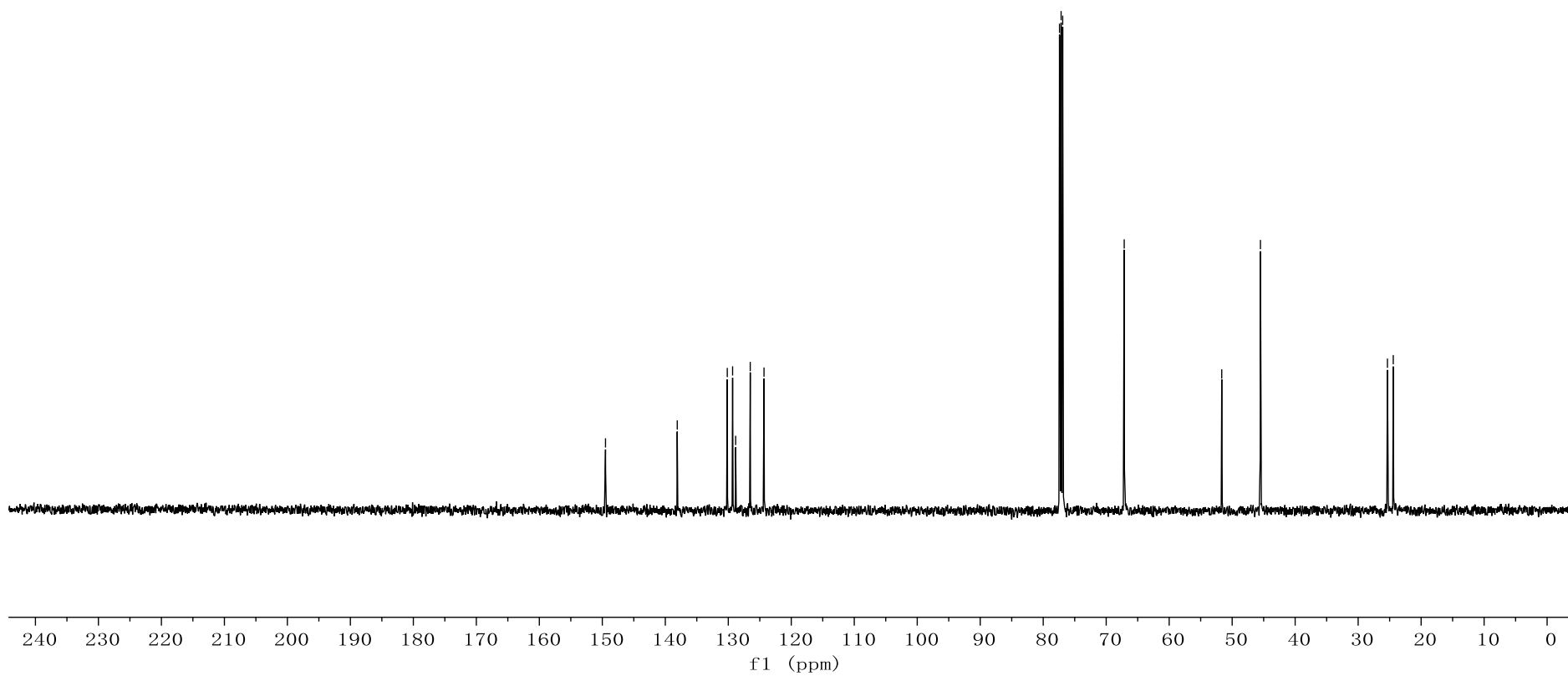
24

— 149.49
— 138.10
— 130.16
— 129.31
— 128.83
— 126.51
— 124.33

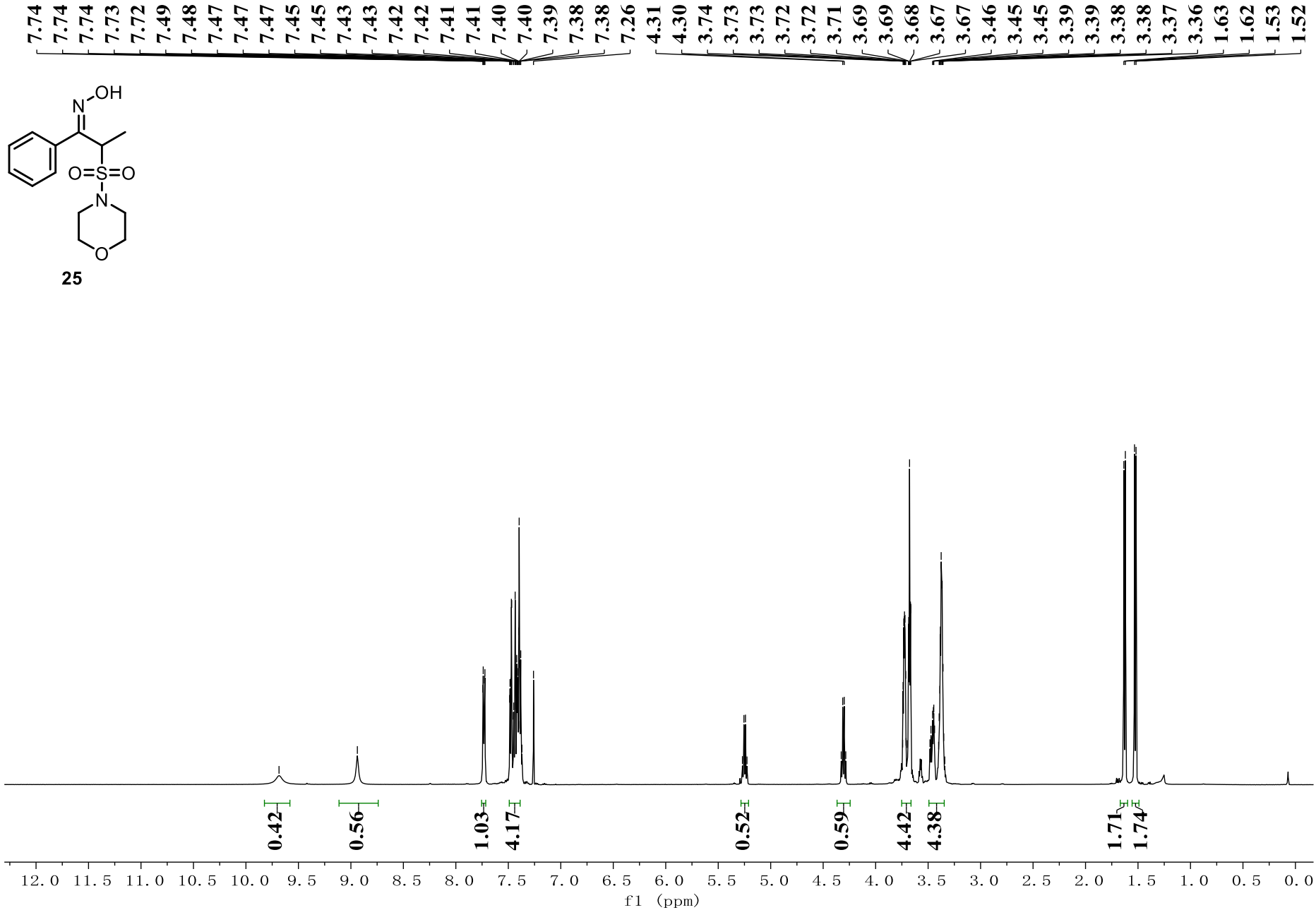
— 77.41
— 77.16
— 76.91
— 67.14

— 51.65
— 45.51

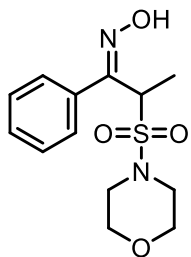
— 25.35
— 24.42



¹H NMR (500 MHz, CDCl₃) for 25



¹³C NMR (125 MHz, CDCl₃) for 25

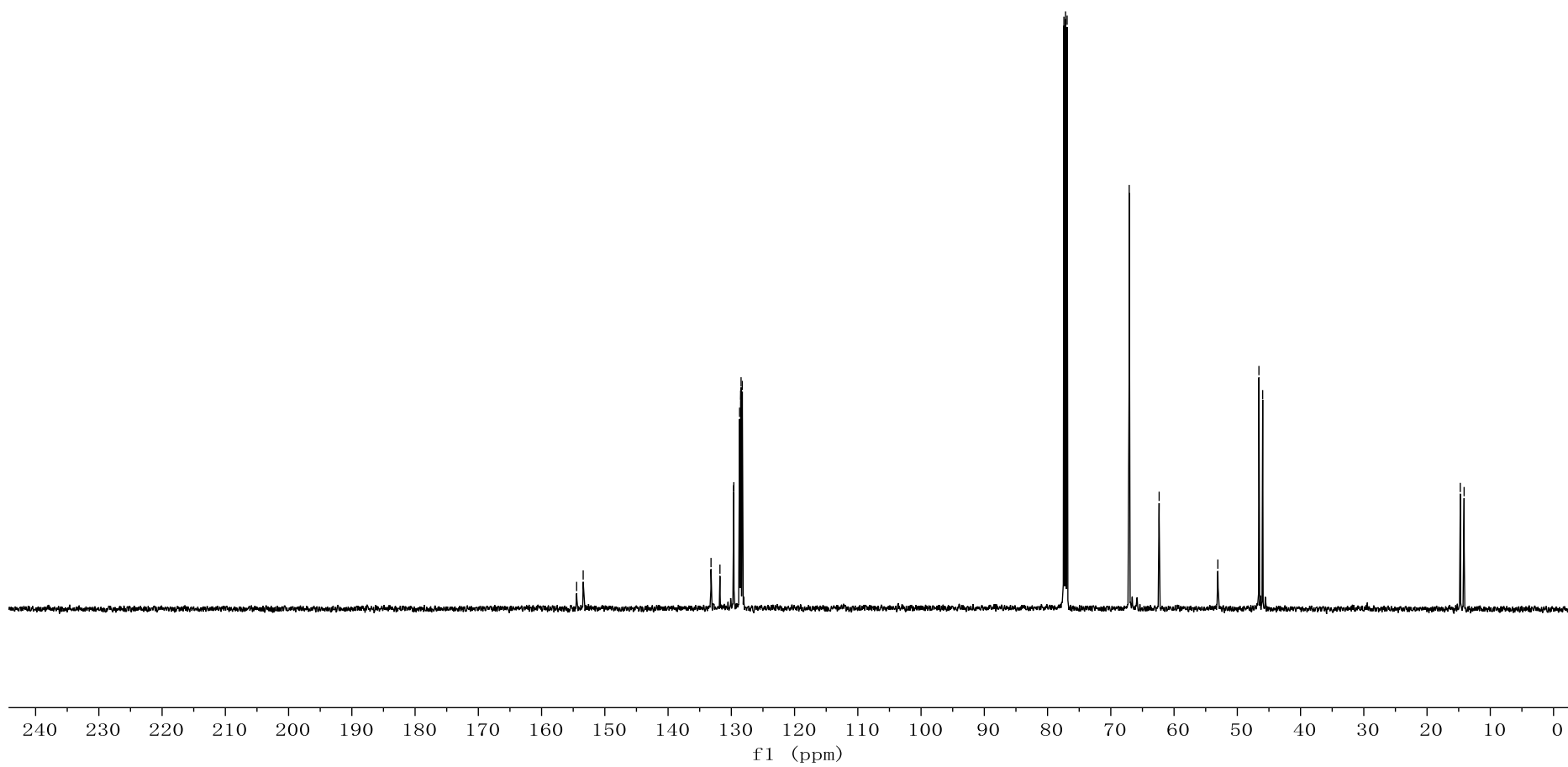


25

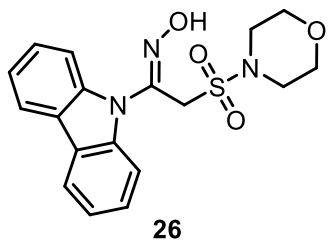
154.46
153.42
133.21
131.80
129.64
129.61
128.70
128.53
128.46
128.26

77.41
77.16
76.91
67.09
62.36
53.07
46.58
45.99

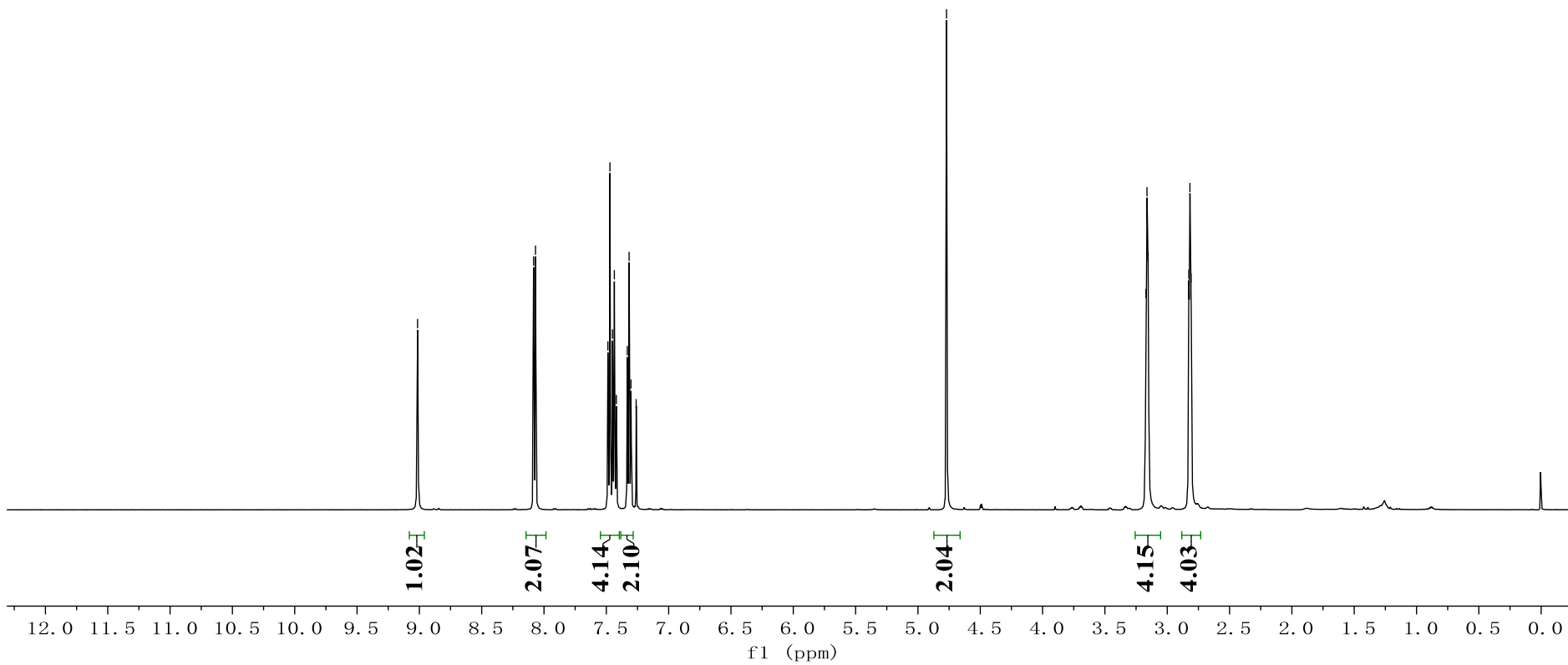
14.74
14.14



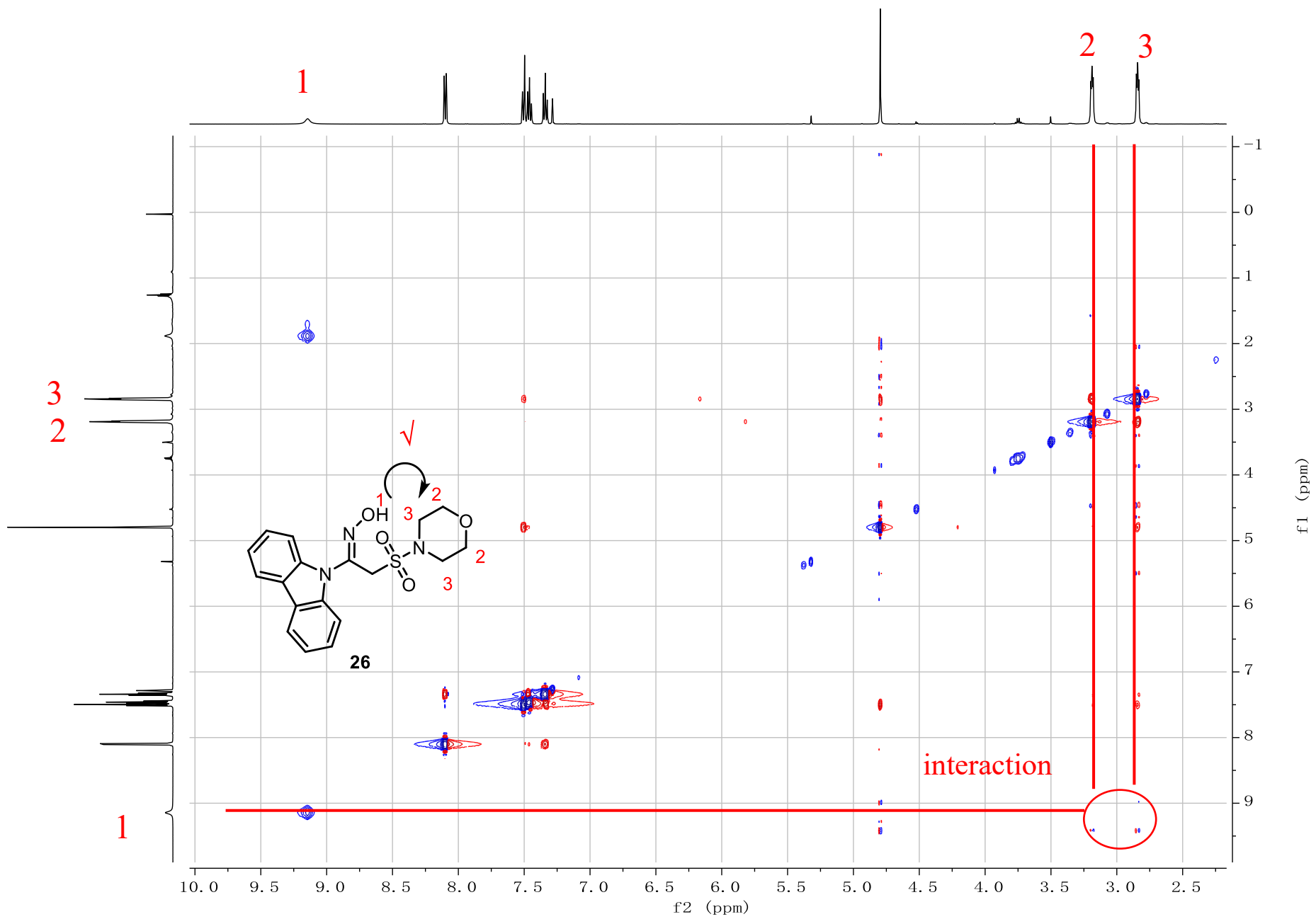
¹H NMR (500 MHz, CDCl₃) for *E*-26



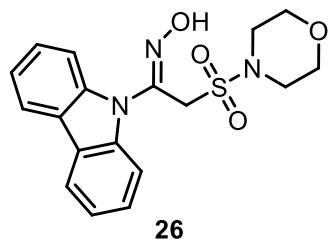
— 9.01
8.08
8.07
7.49
7.47
7.45
7.44
7.42
7.33
7.32
7.30
7.26
— 4.77
3.17
3.16
3.15
2.83
2.82
2.81



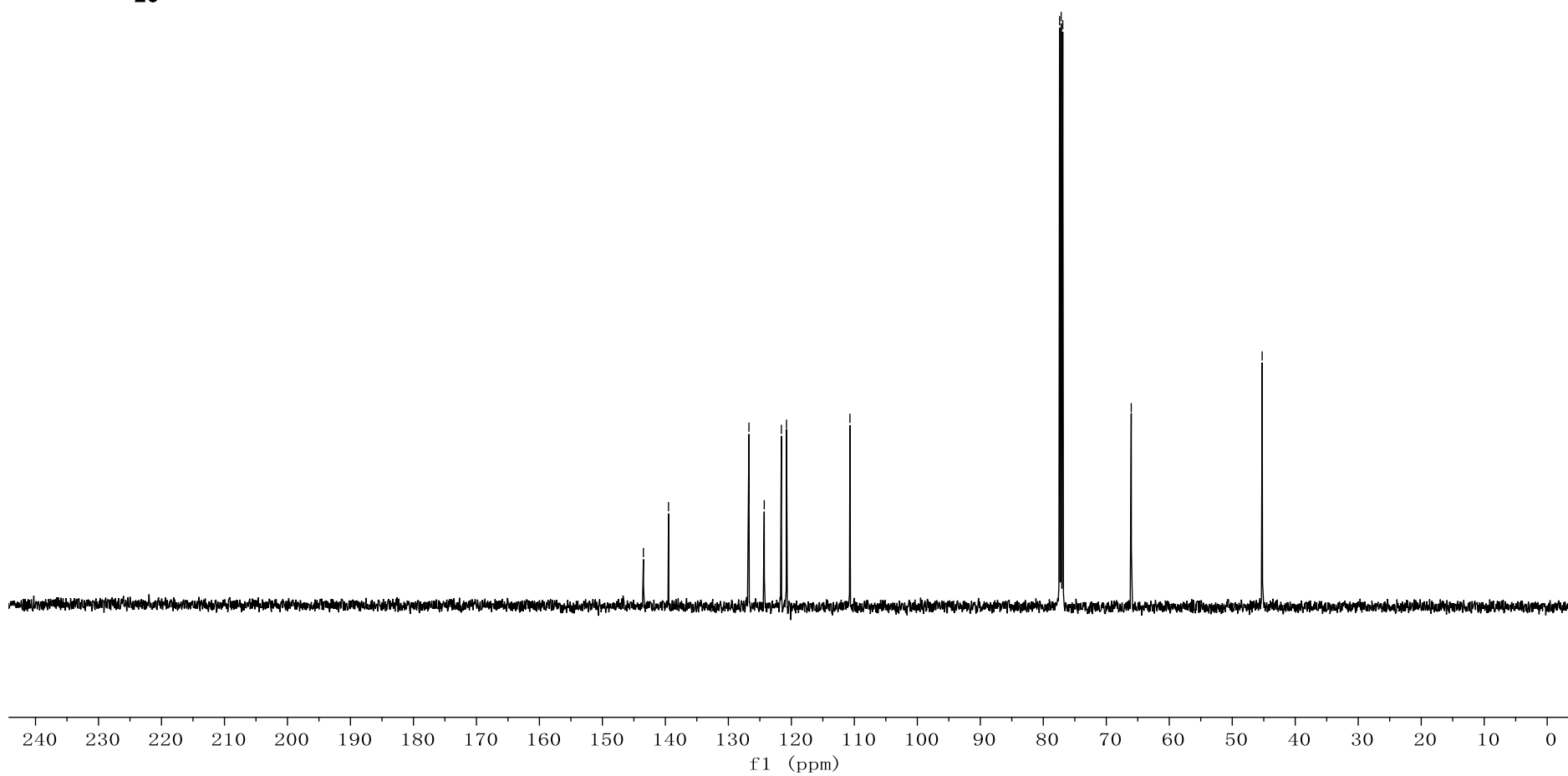
NOESY of E-26



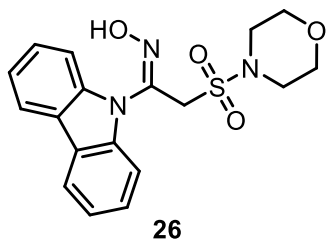
¹³C NMR (125 MHz, CDCl₃) for *E*-26



143.49
139.51
126.73
124.30
121.59
120.76
110.70
77.42
77.16
76.91
66.04
45.25



¹H NMR (500 MHz, CDCl₃) for Z-26

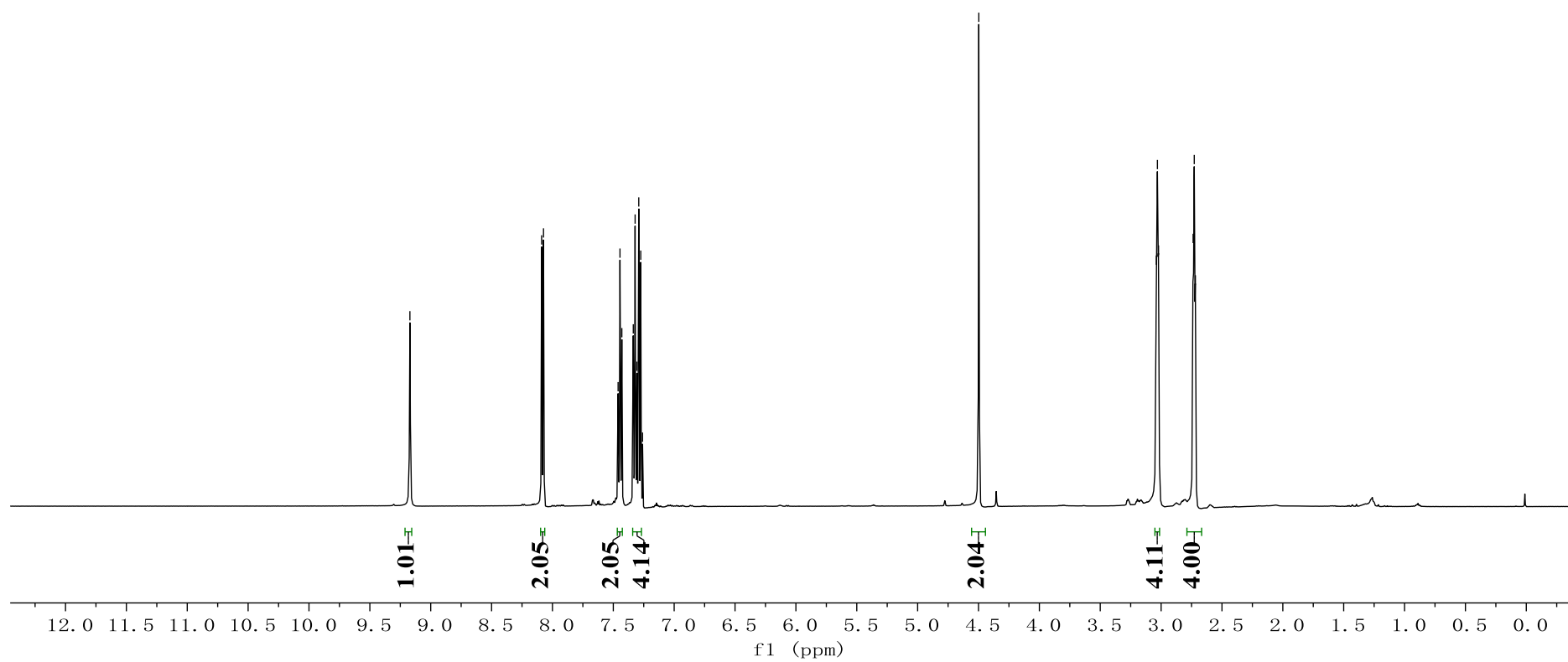


— 9.17

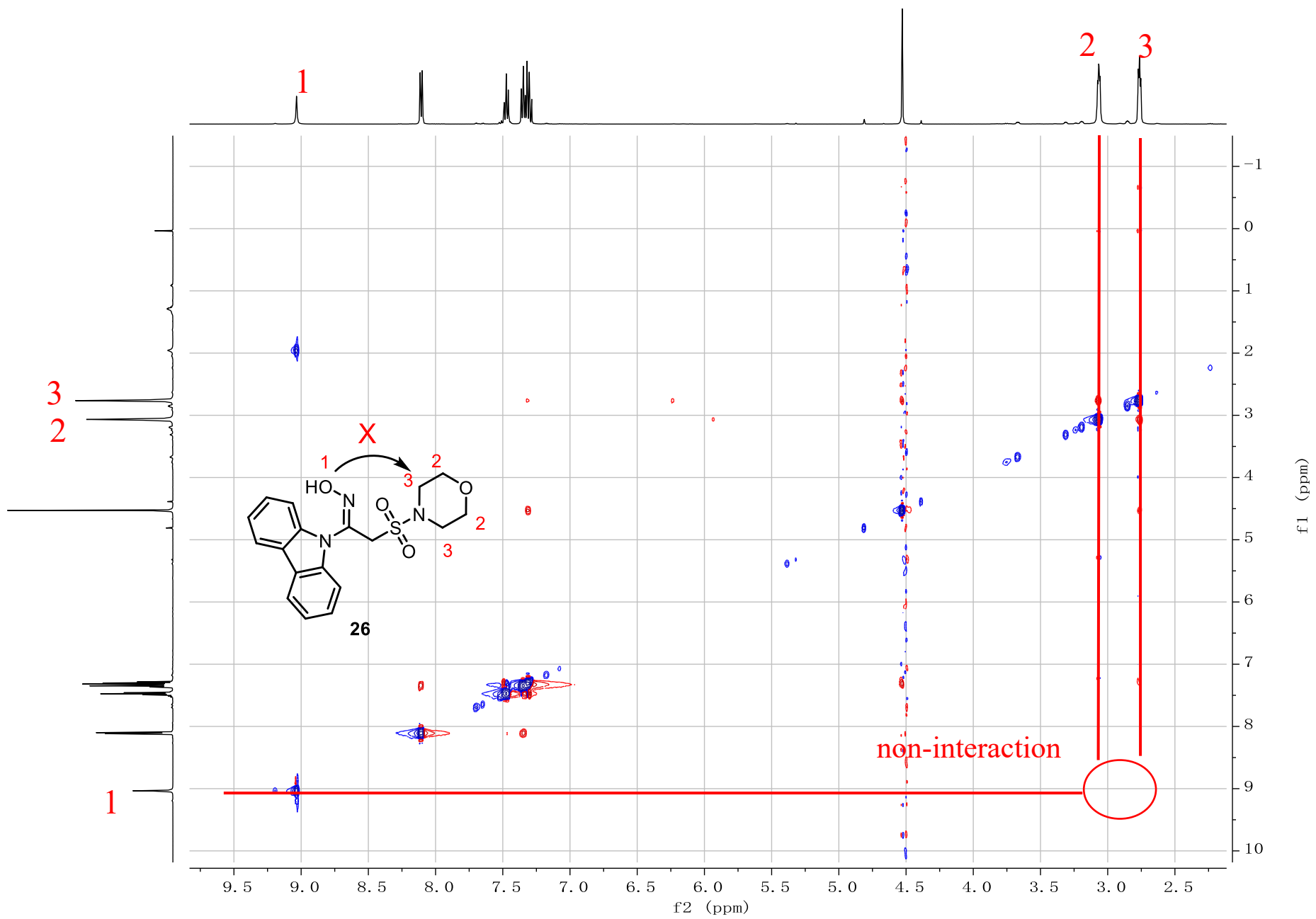
8.09
8.07
7.46
7.45
7.43
7.34
7.32
7.31
7.29
7.27
7.26

— 4.50

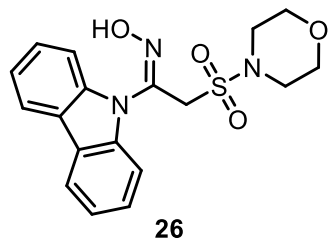
3.04
3.03
3.02
2.74
2.73
2.72



NOESY of Z-26



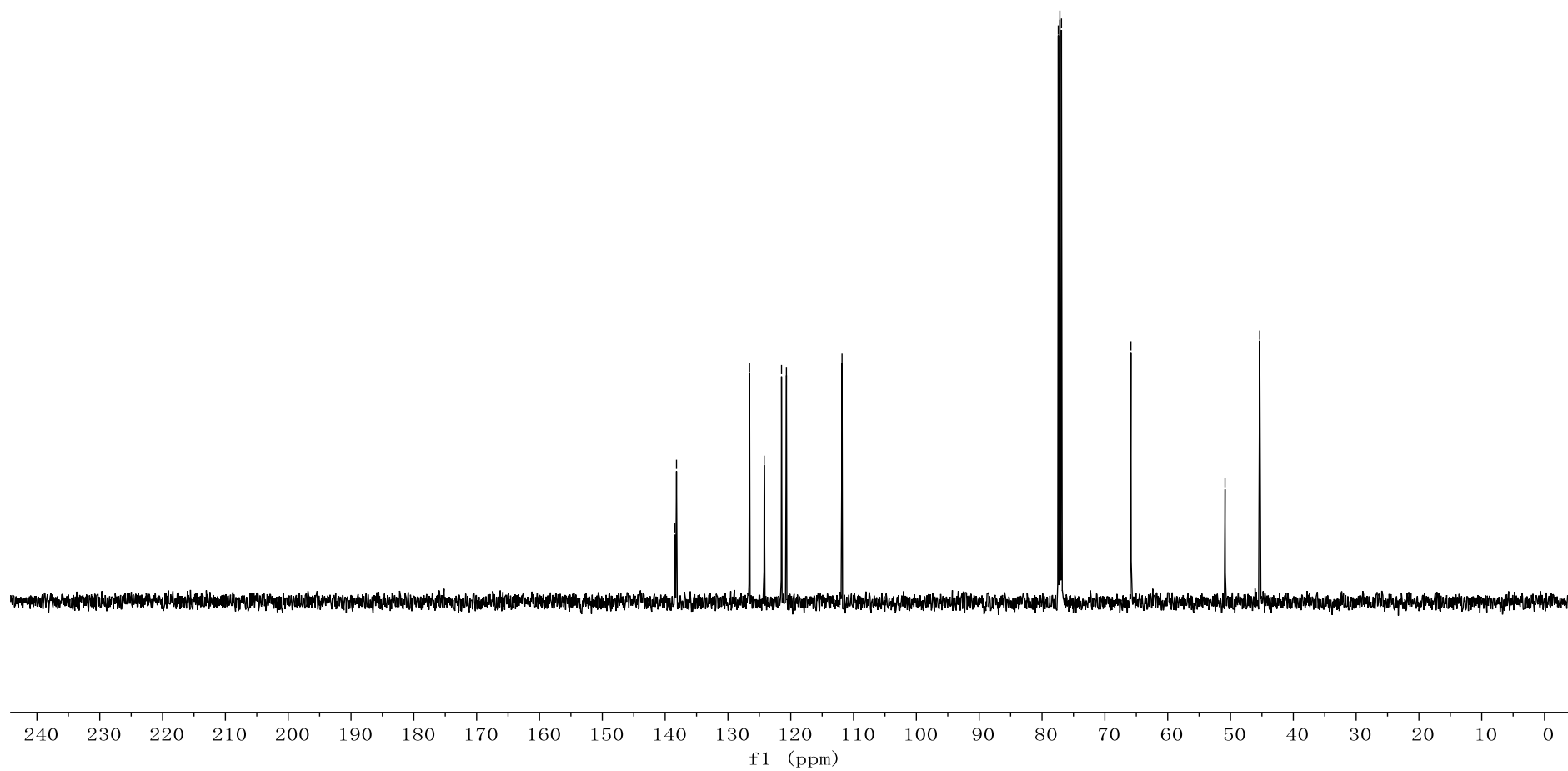
¹³C NMR (125 MHz, CDCl₃) for Z-26



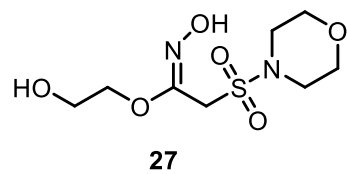
138.45
138.19
126.56
124.23
121.47
120.69
111.84

77.41
77.16
76.91
65.85

50.87
45.35



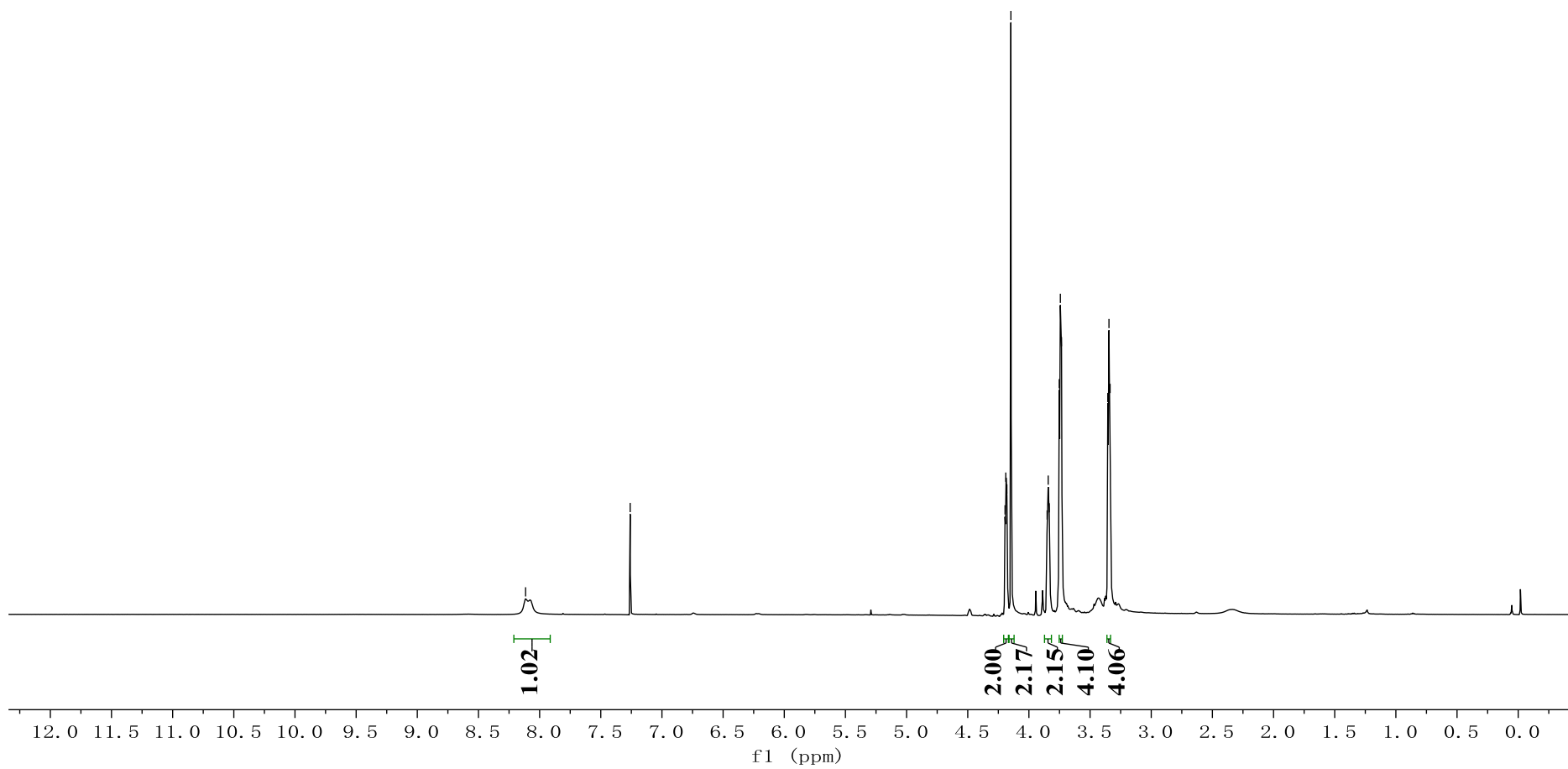
¹H NMR (500 MHz, CDCl₃) for 27



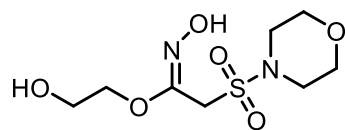
— 8.11

— 7.26

4.20
4.19
4.18
4.15
3.85
3.84
3.83
3.75
3.74
3.73
3.36
3.35
3.34



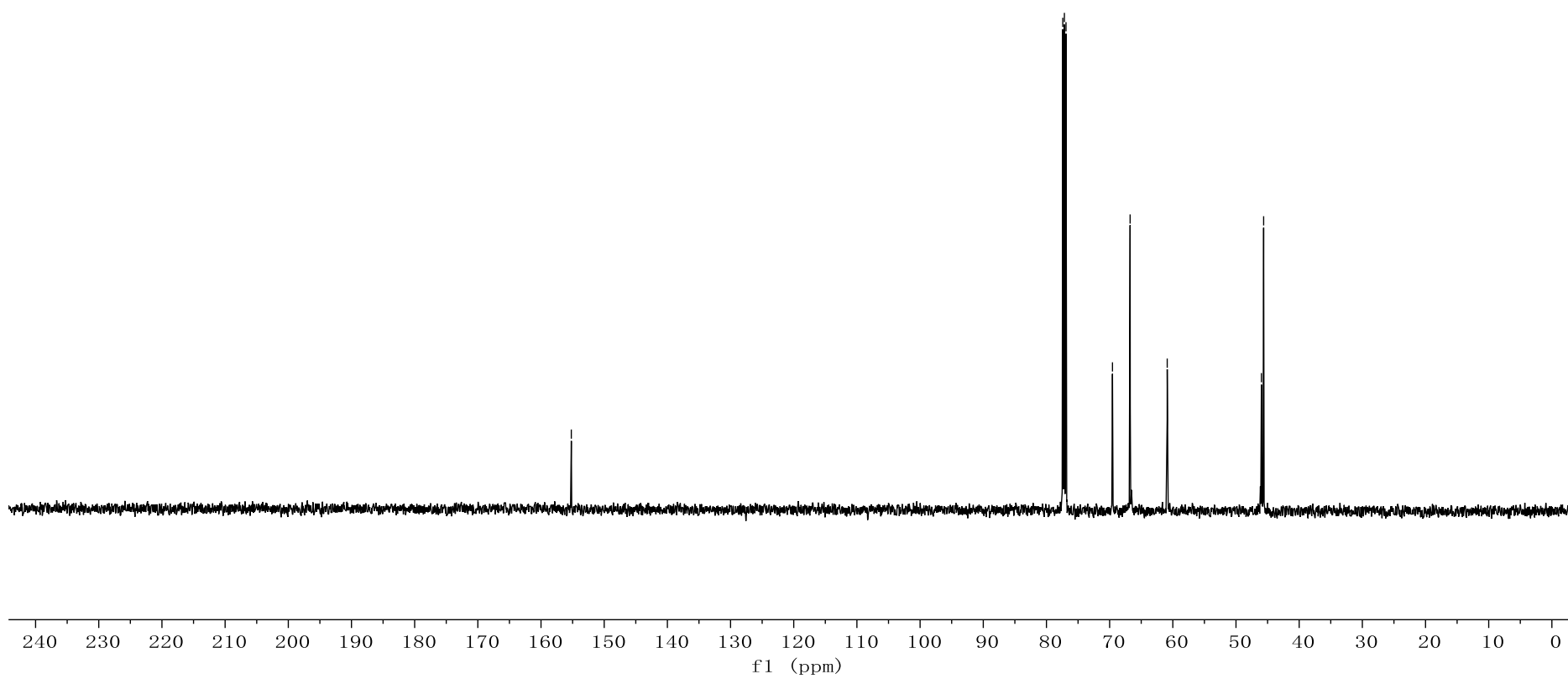
¹³C NMR (125 MHz, CDCl₃) for 27



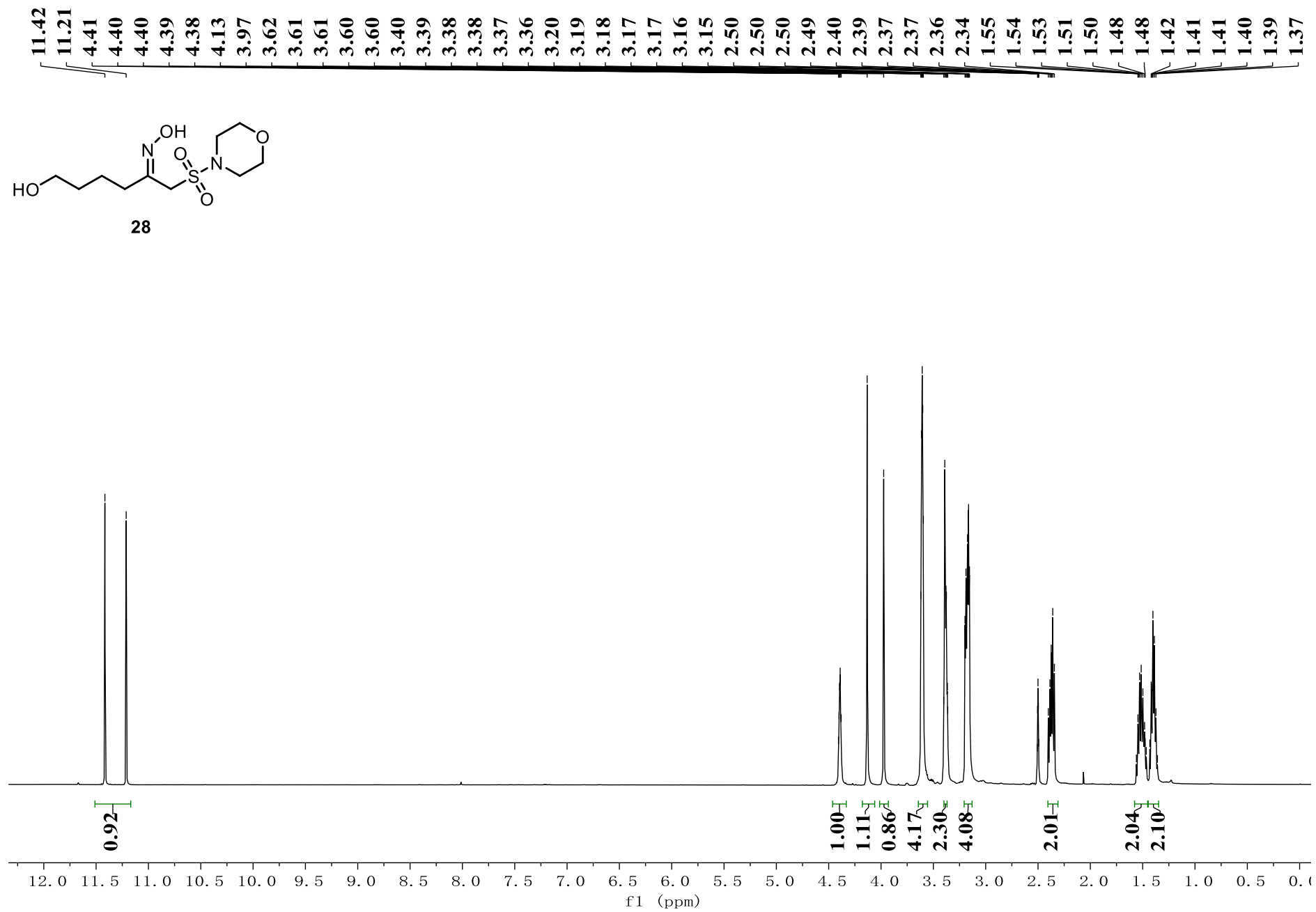
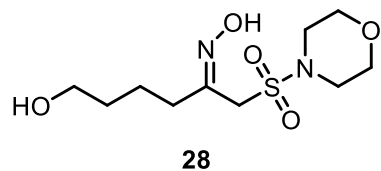
27

— 155.21

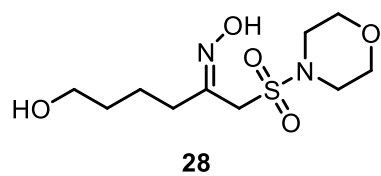
77.42
77.16
76.91
69.55
66.76
60.88
45.98
45.62



¹H NMR (500 MHz, DMSO-d₆) for 28

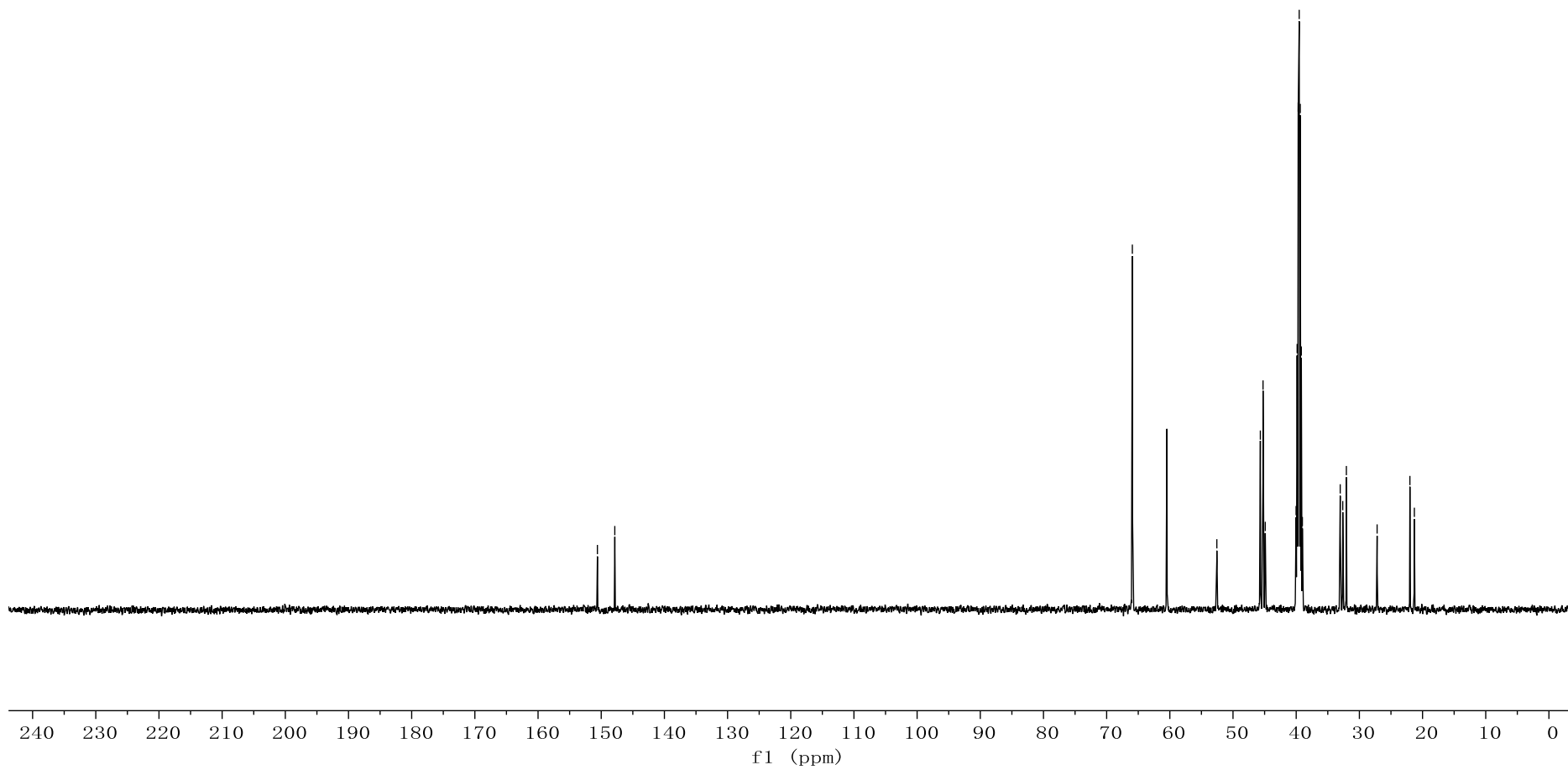


¹³C NMR (125 MHz, DMSO-*d*₆) for 28

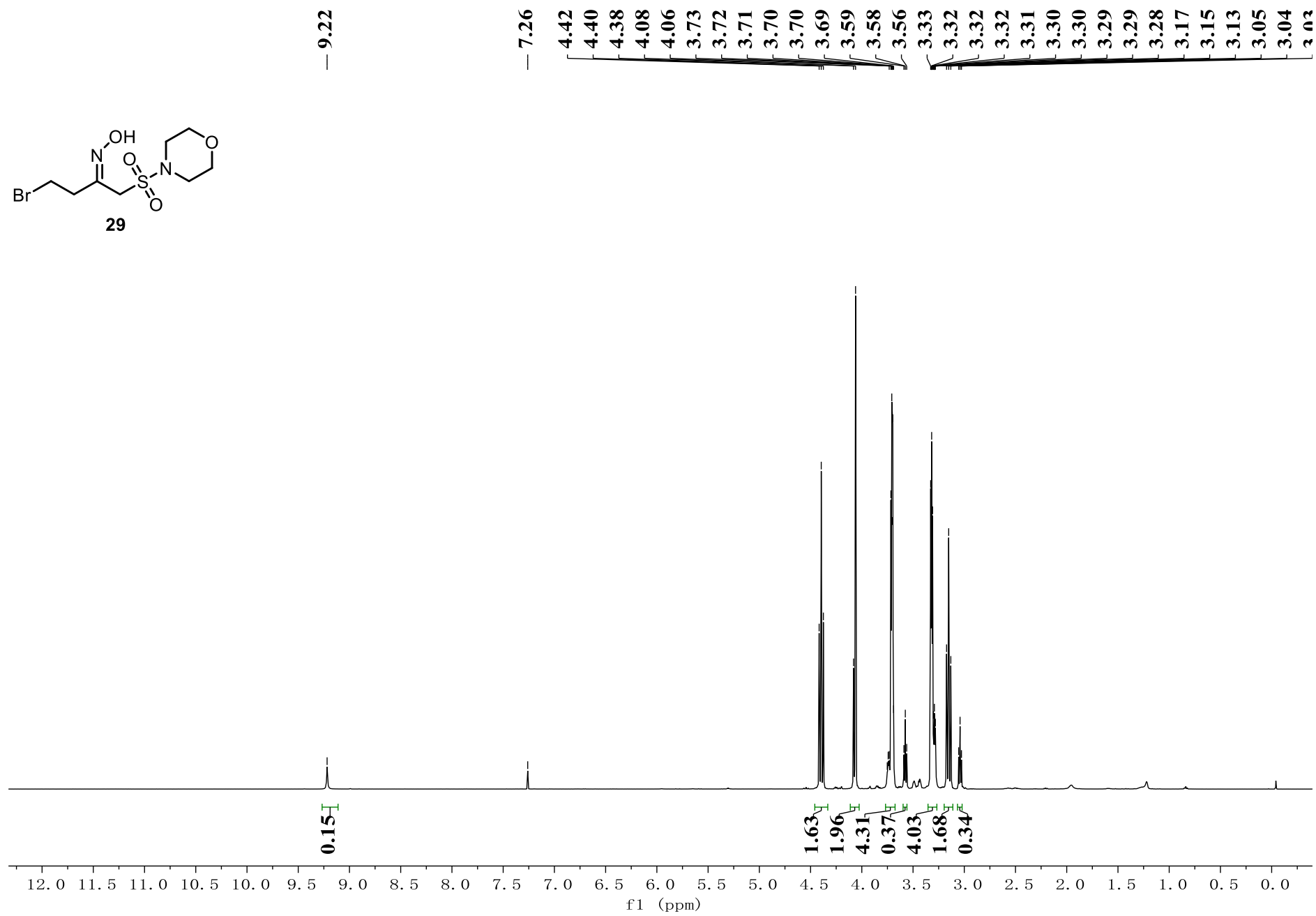
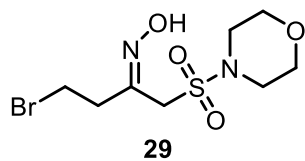


~ 150.60
~ 147.85

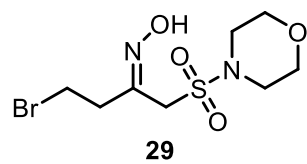
65.93
60.47
52.56
45.68
45.25
44.89
40.02
39.85
39.69
39.52
39.35
39.19
39.02
33.03
32.63
32.06
27.19
22.00
21.30



¹H NMR (500 MHz, CDCl₃) for 29

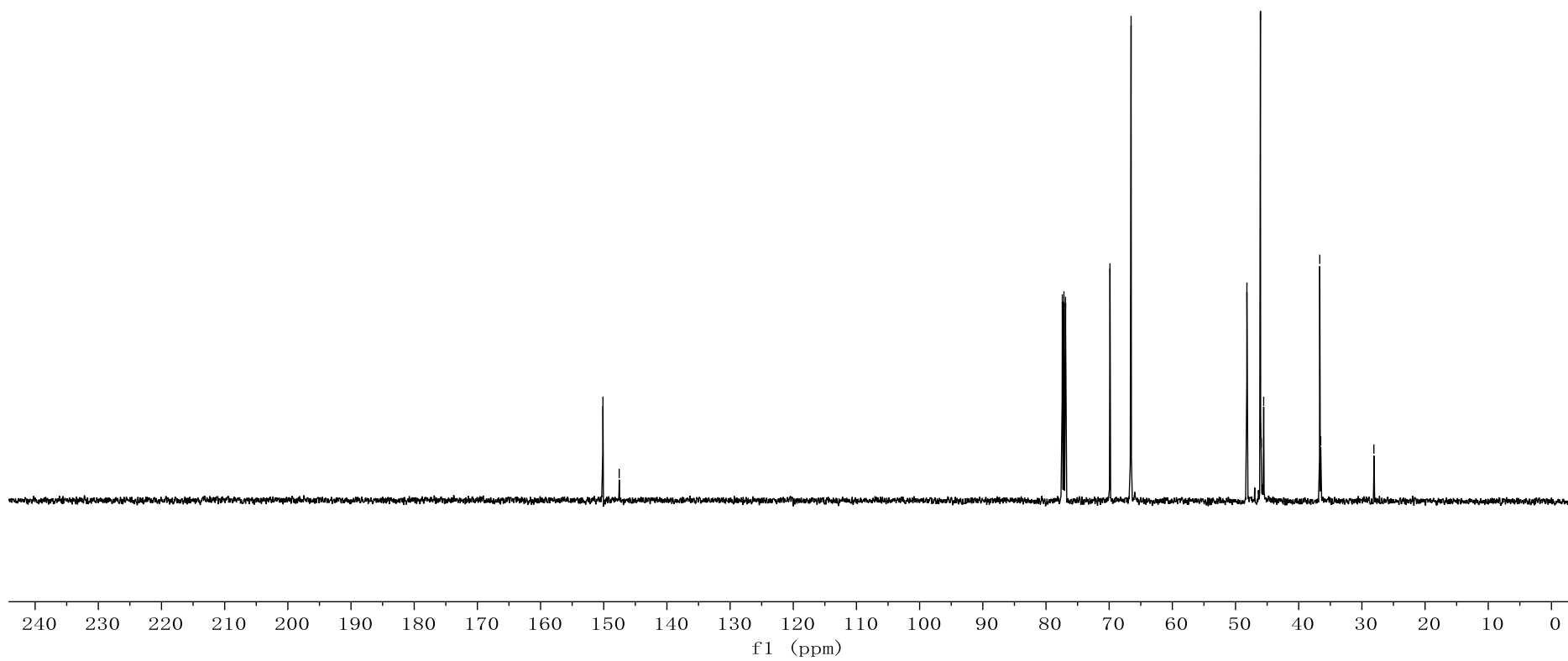


¹³C NMR (125 MHz, CDCl₃) for 29

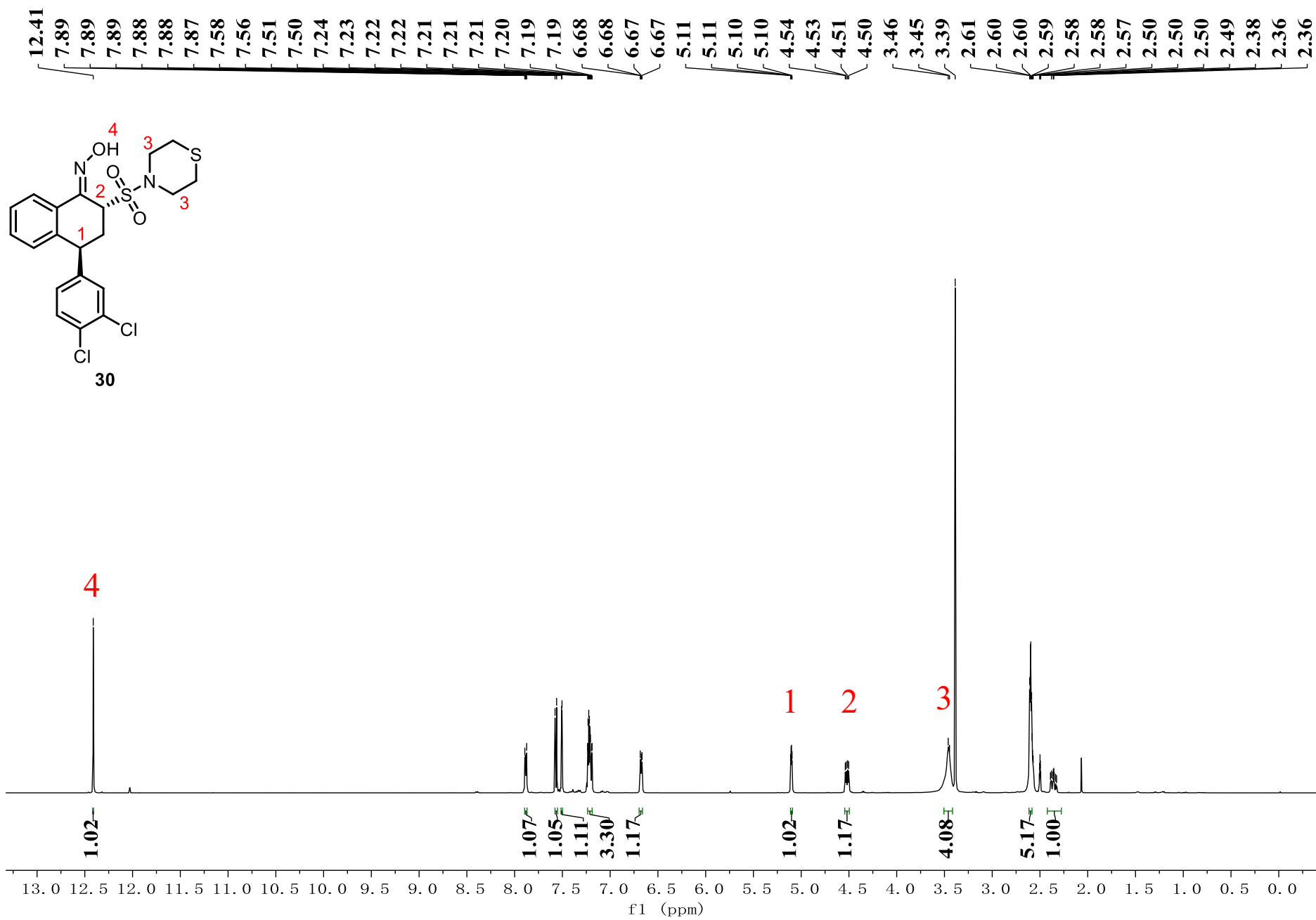


~ 150.13
~ 147.55

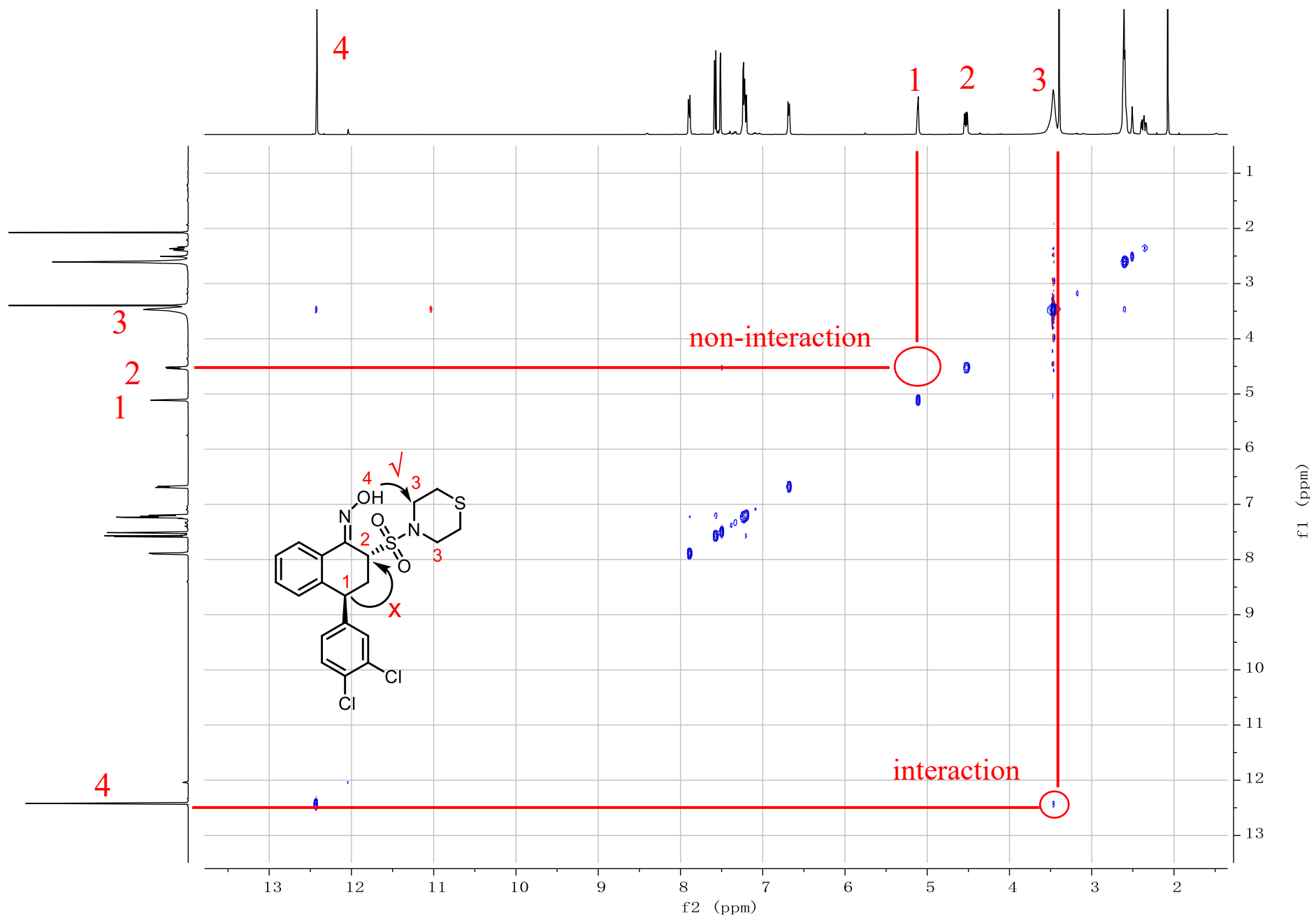
77.42
77.16
76.91
69.87
66.63
66.53
48.20
46.06
45.92
45.54
36.67
36.52
28.10



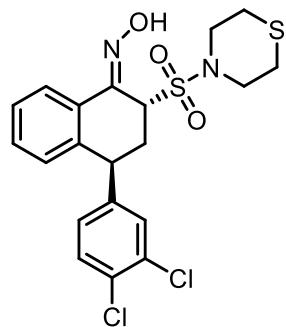
¹H NMR (500 MHz, DMSO-d₆) for 30



NOESY of 30



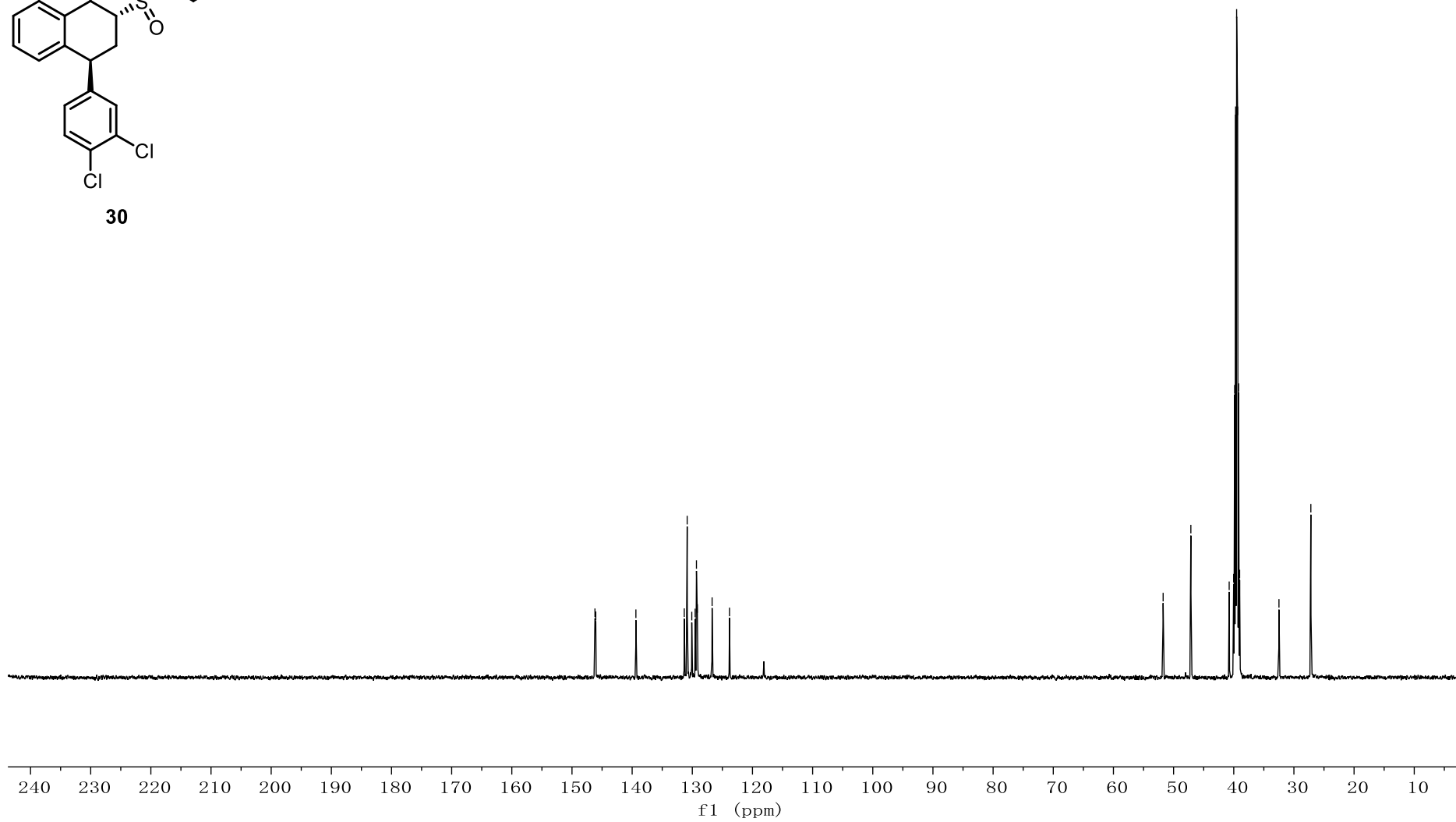
¹³C NMR (125 MHz, DMSO-*d*₆) for 30



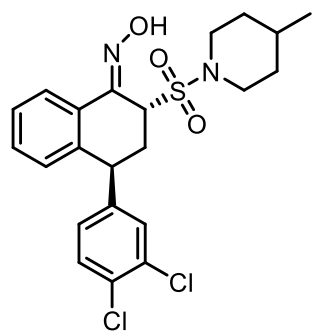
30

146.18
146.04
139.38
131.34
130.85
130.09
129.53
129.31
129.16
126.71
123.81

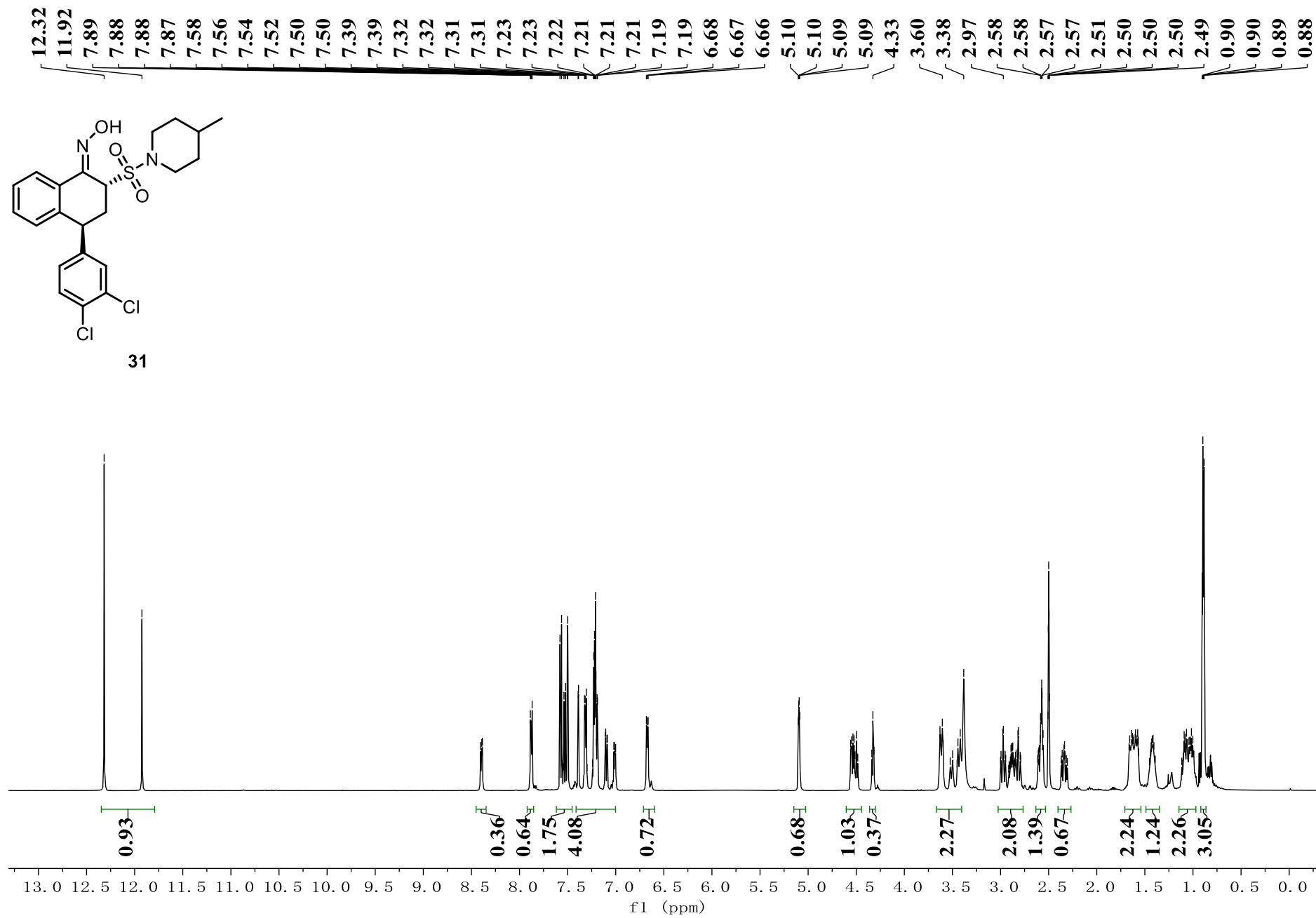
51.74
47.13
40.76
40.02
39.85
39.69
39.52
39.35
39.19
39.02
32.48
27.19



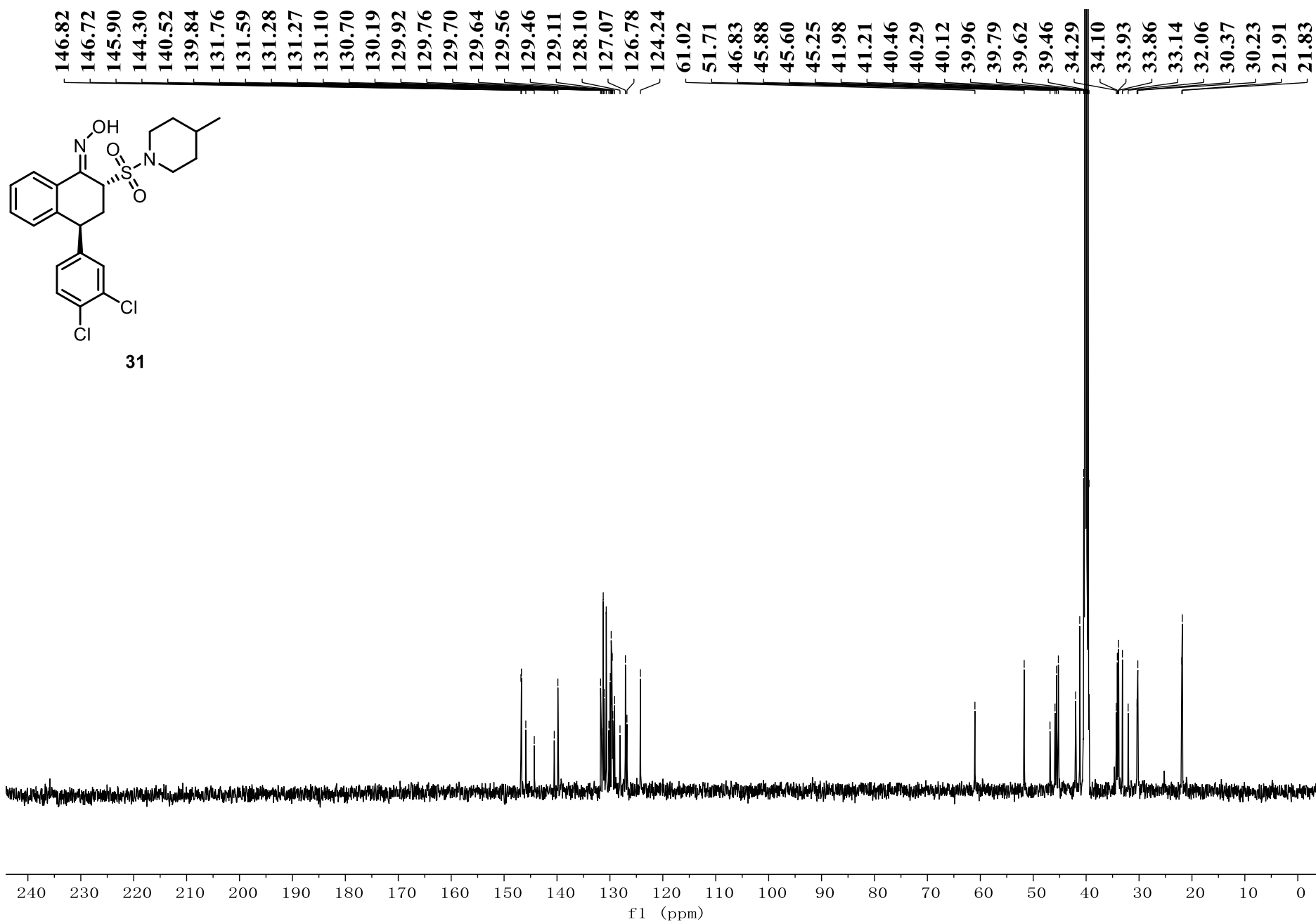
¹H NMR (500 MHz, DMSO-d₆) for 31



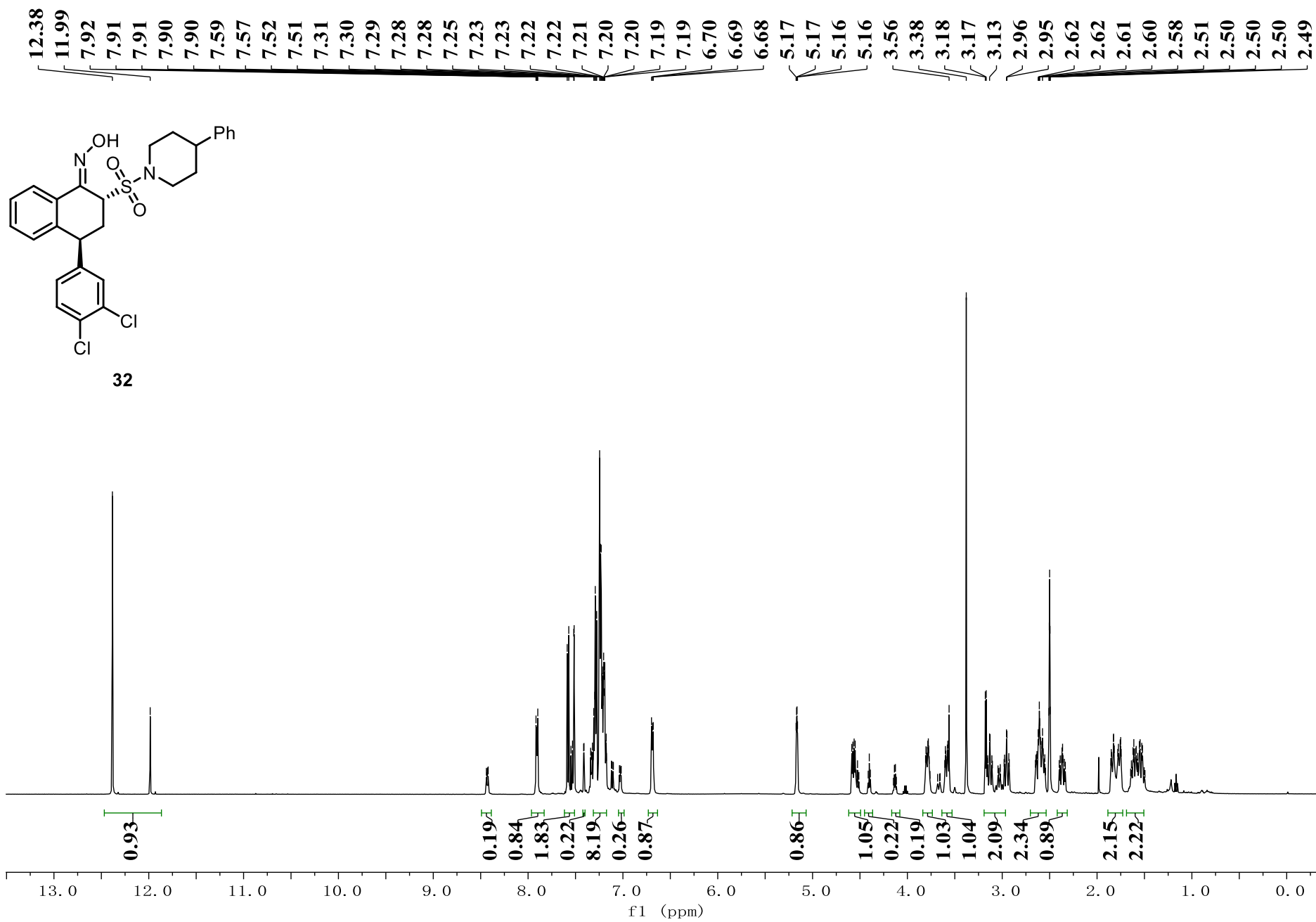
31



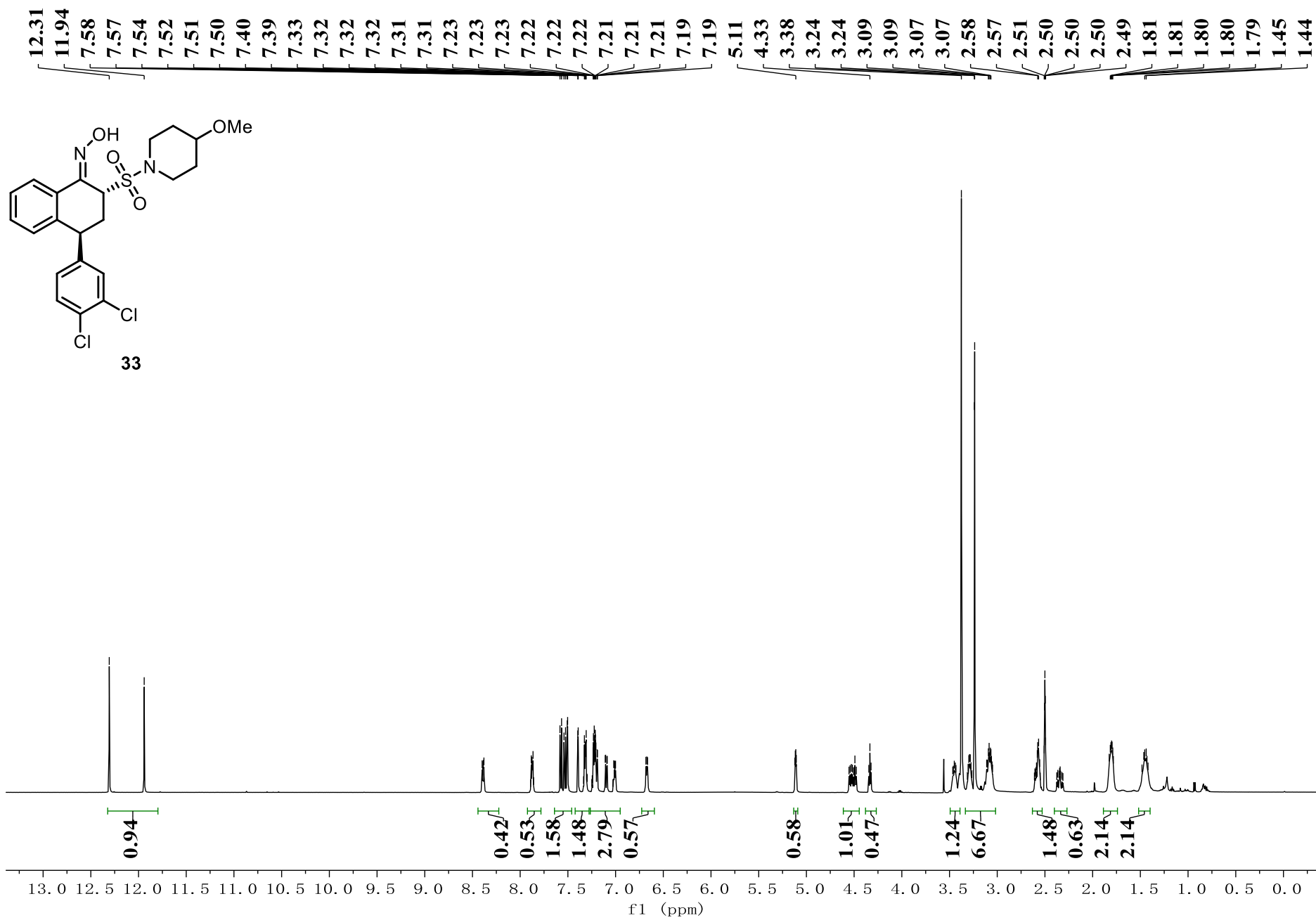
¹³C NMR (125 MHz, DMSO-*d*₆) for 31



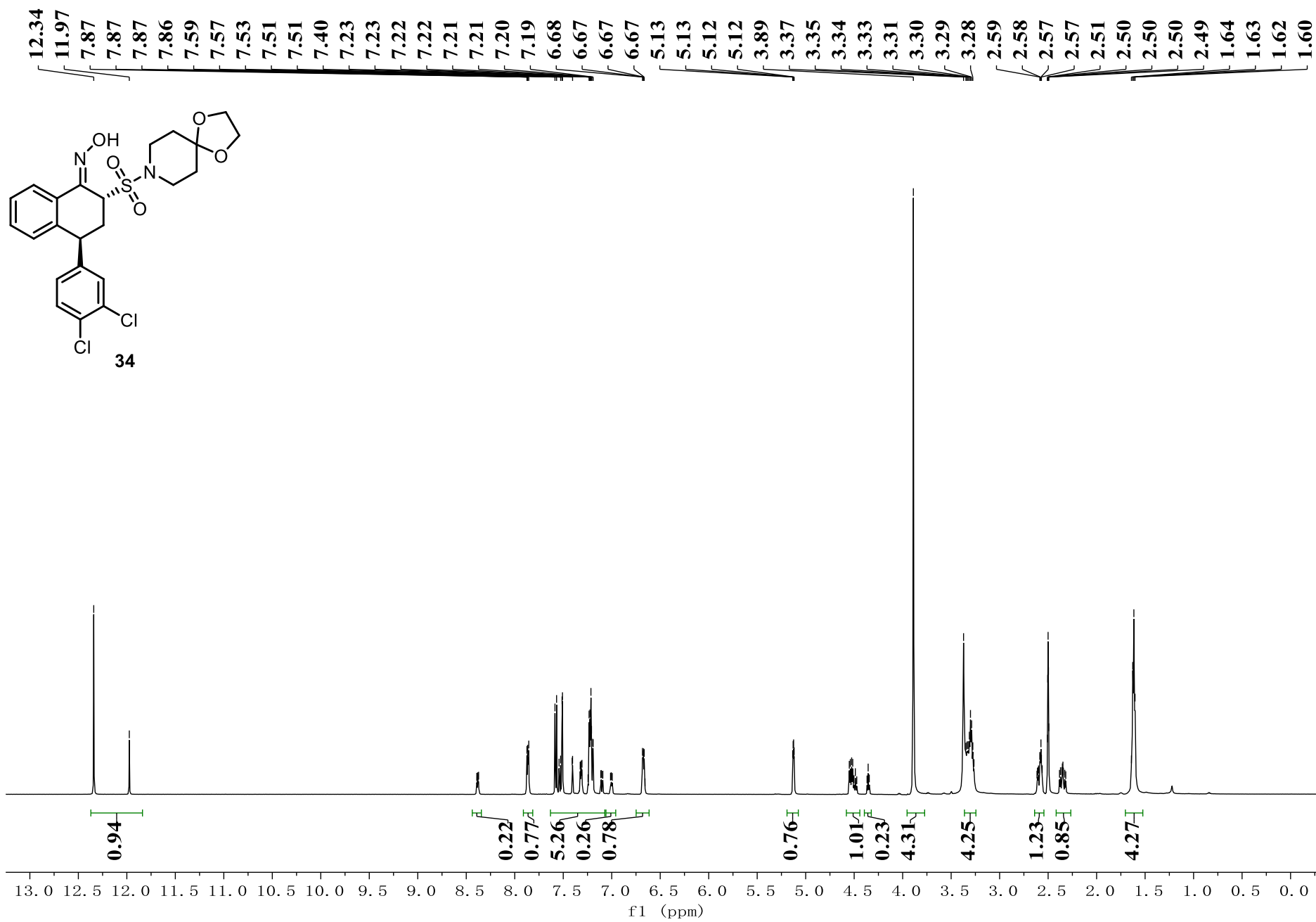
¹H NMR (500 MHz, DMSO-*d*₆) for 32



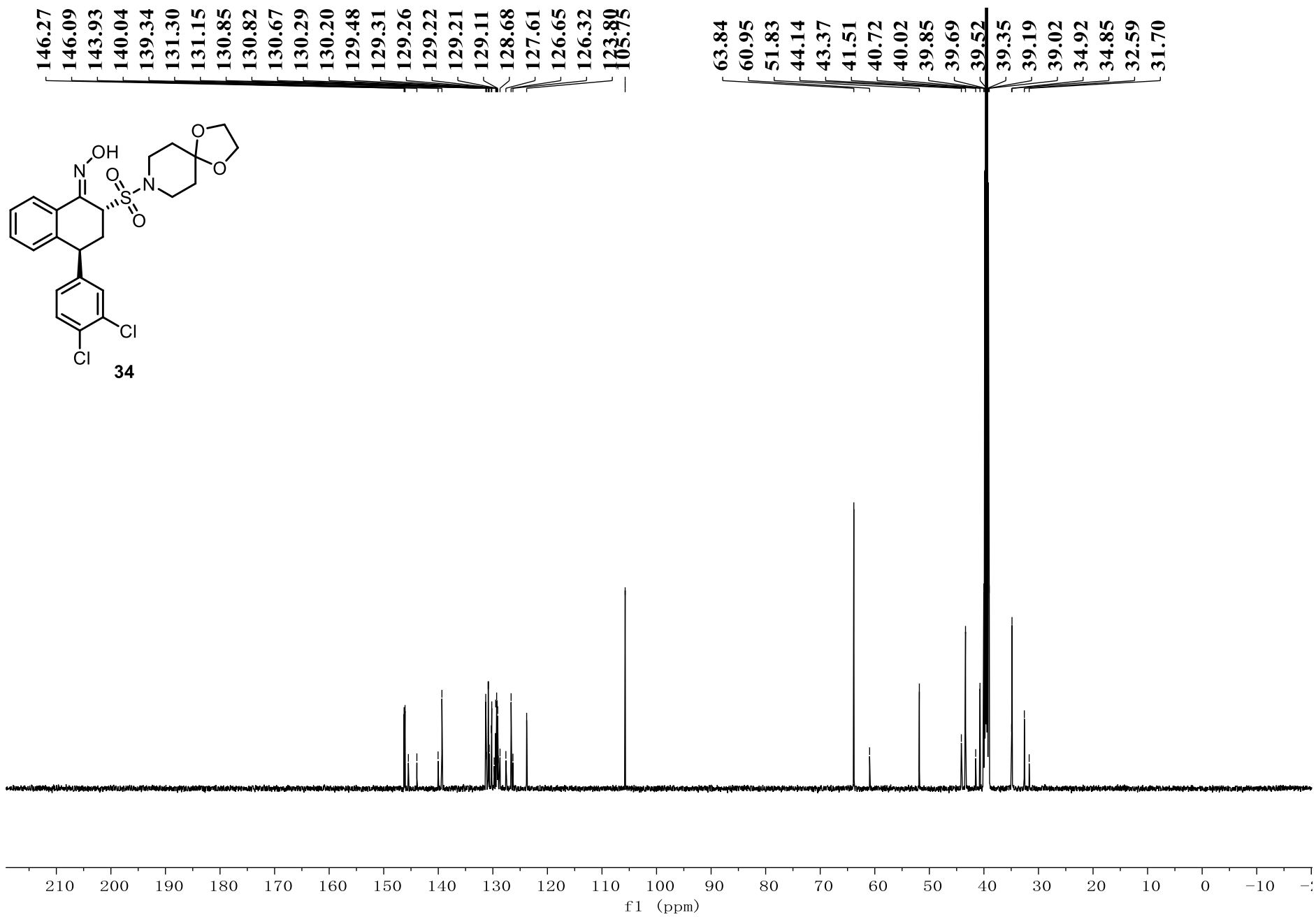
¹H NMR (500 MHz, DMSO-d₆) for 33



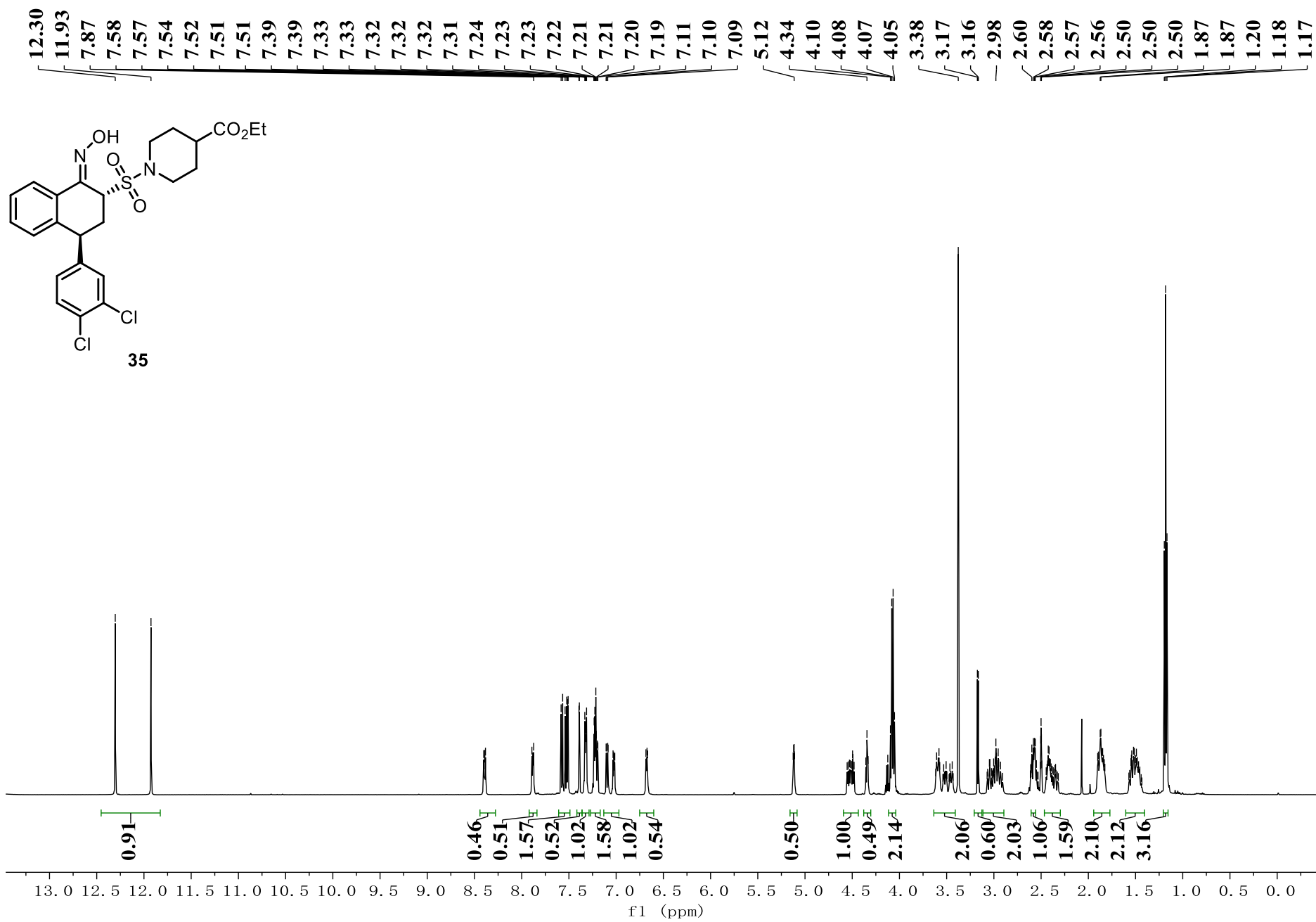
¹H NMR (500 MHz, DMSO-d₆) for 34



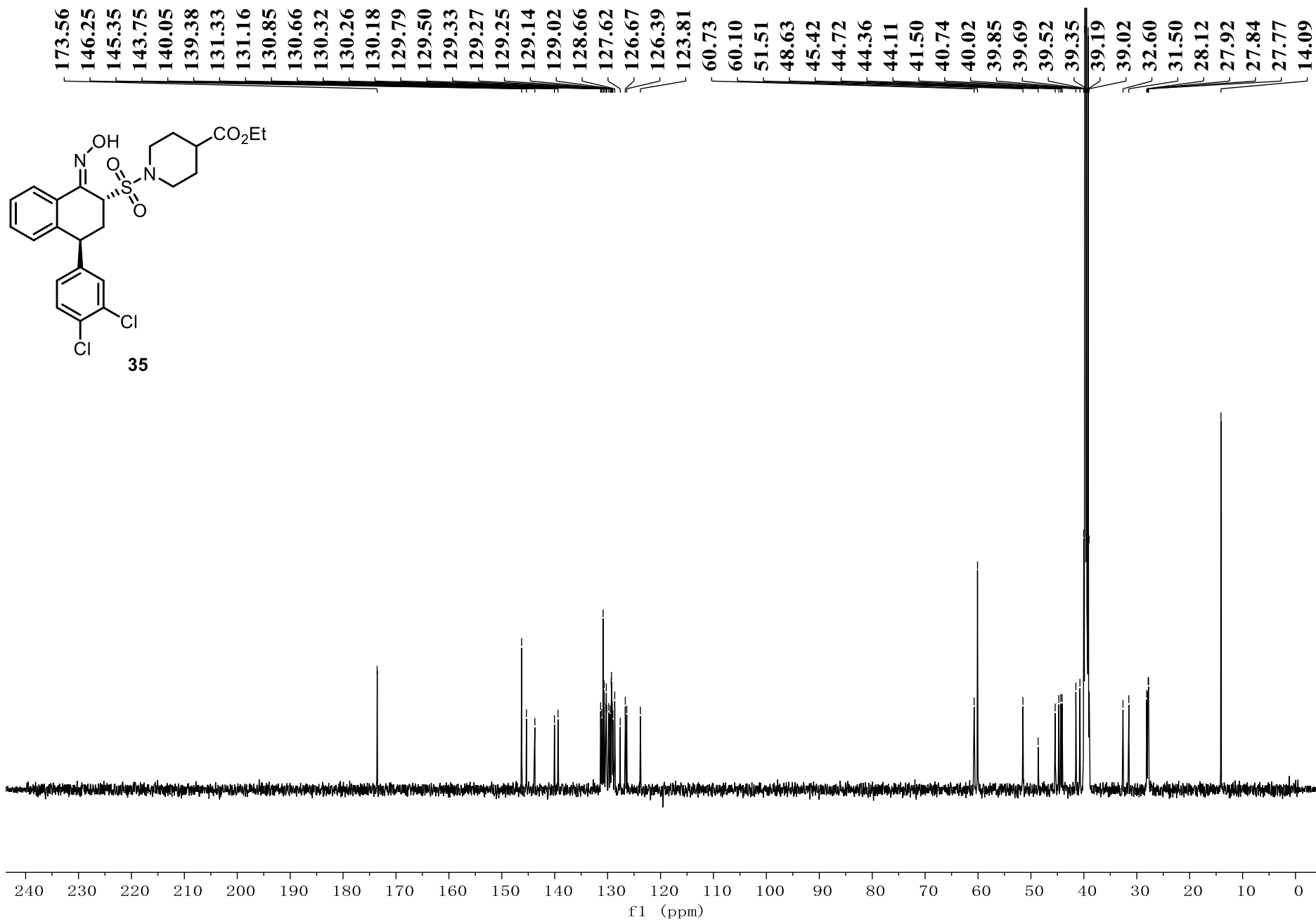
¹³C NMR (125 MHz, DMSO-*d*₆) for 34



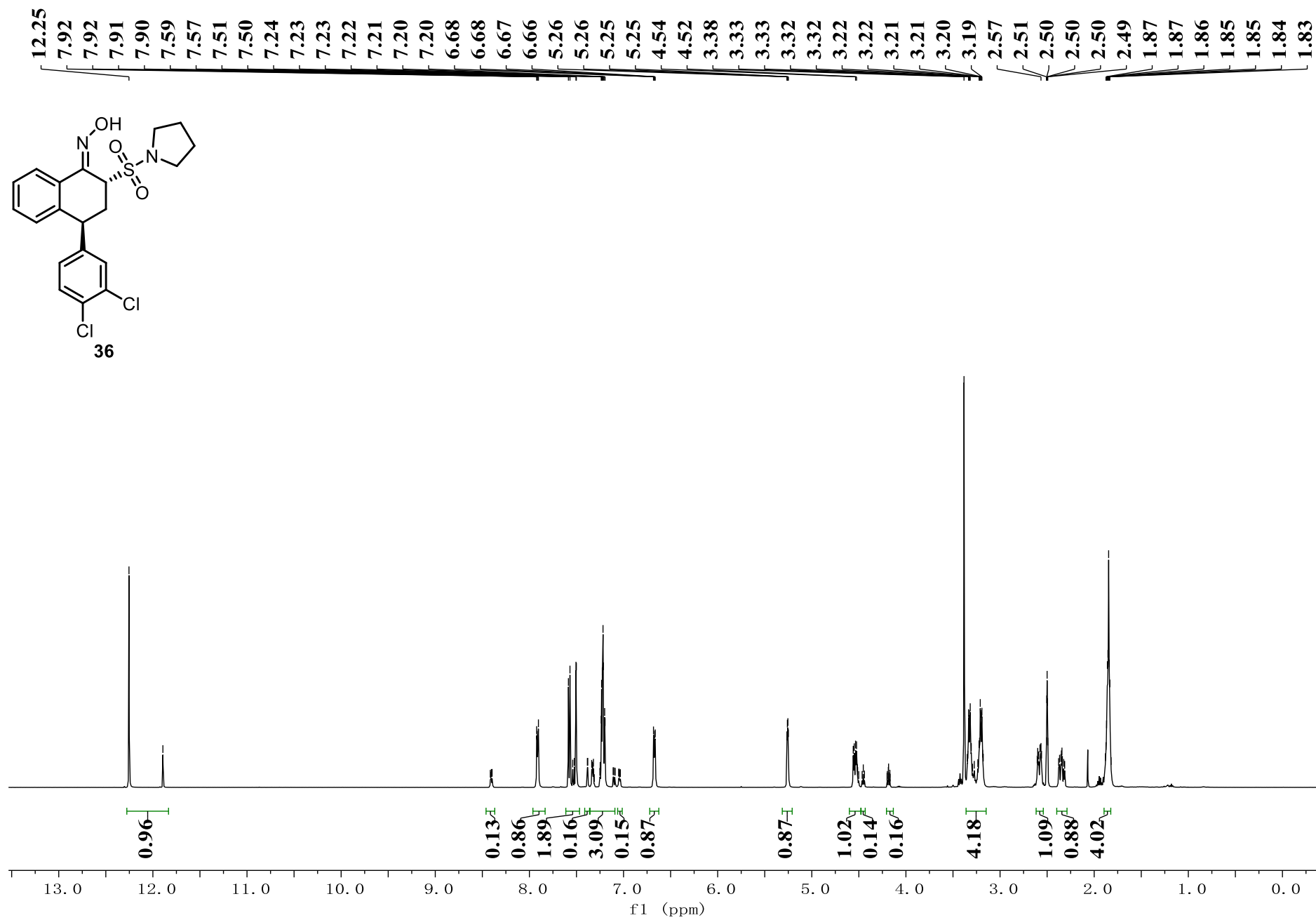
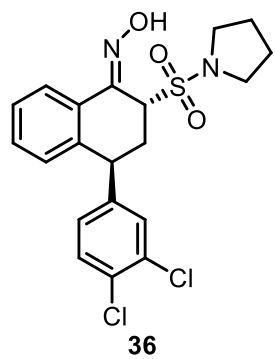
¹H NMR (500 MHz, DMSO-d₆) for 35



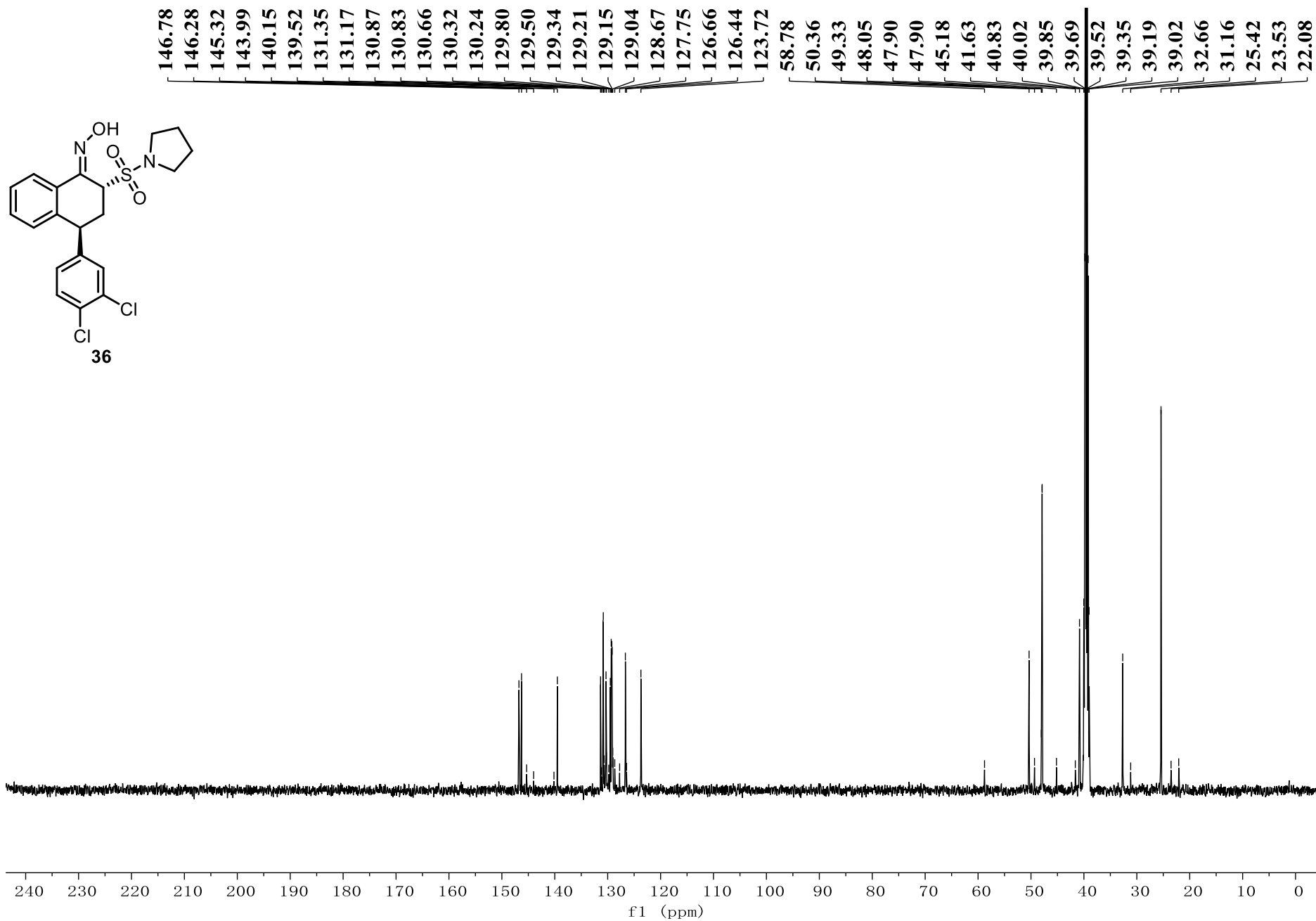
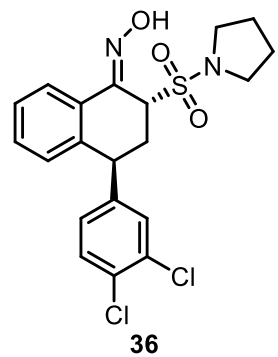
¹³C NMR (125 MHz, DMSO-*d*₆) for 35



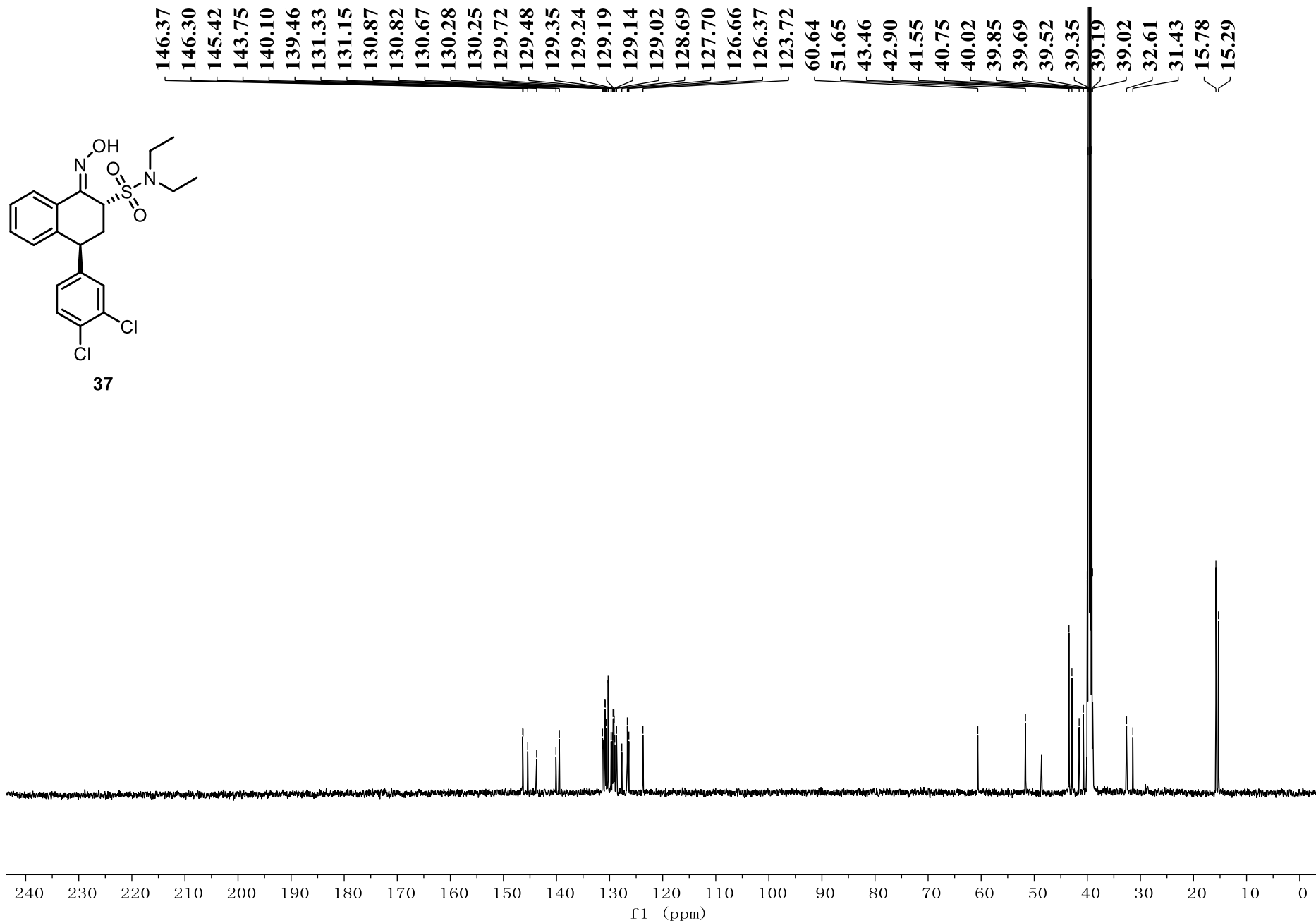
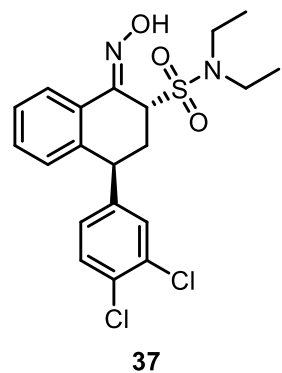
¹H NMR (500 MHz, DMSO-d₆) for 36



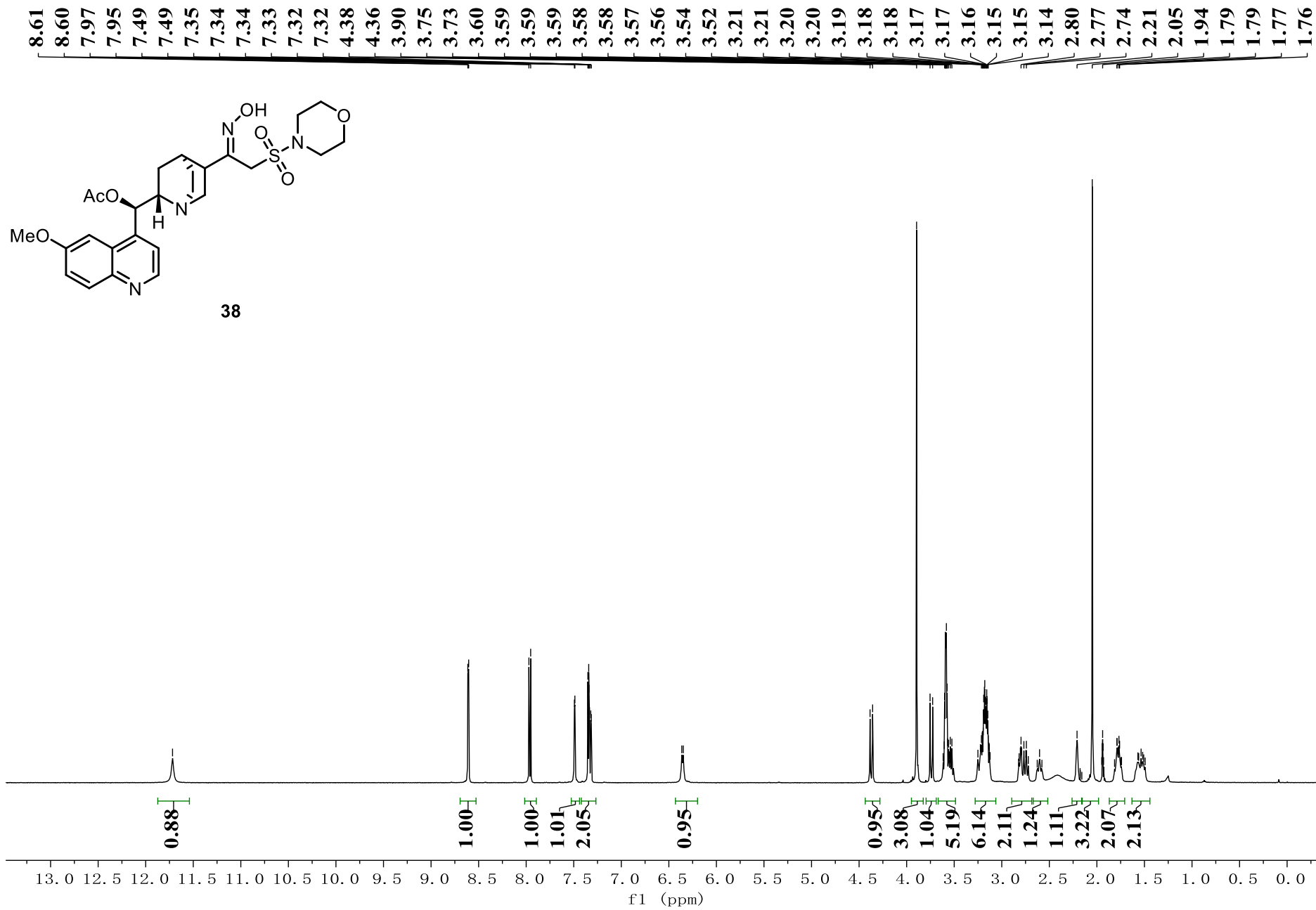
¹³C NMR (125 MHz, DMSO-*d*₆) for 36



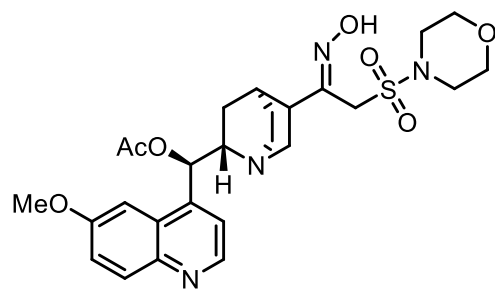
¹H NMR (500 MHz, DMSO-d₆) for 37



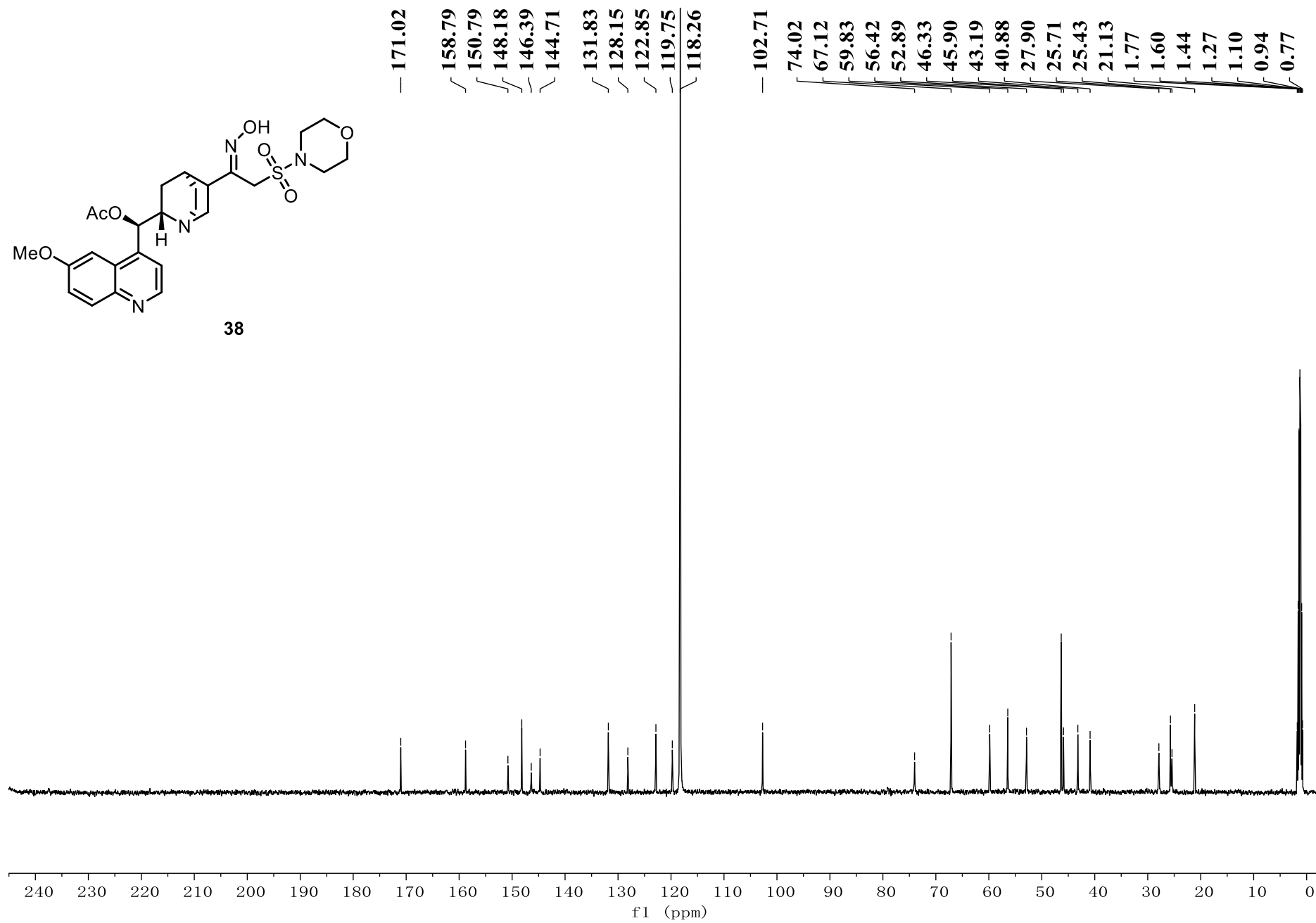
¹H NMR (500 MHz, CD₃CN) for 38



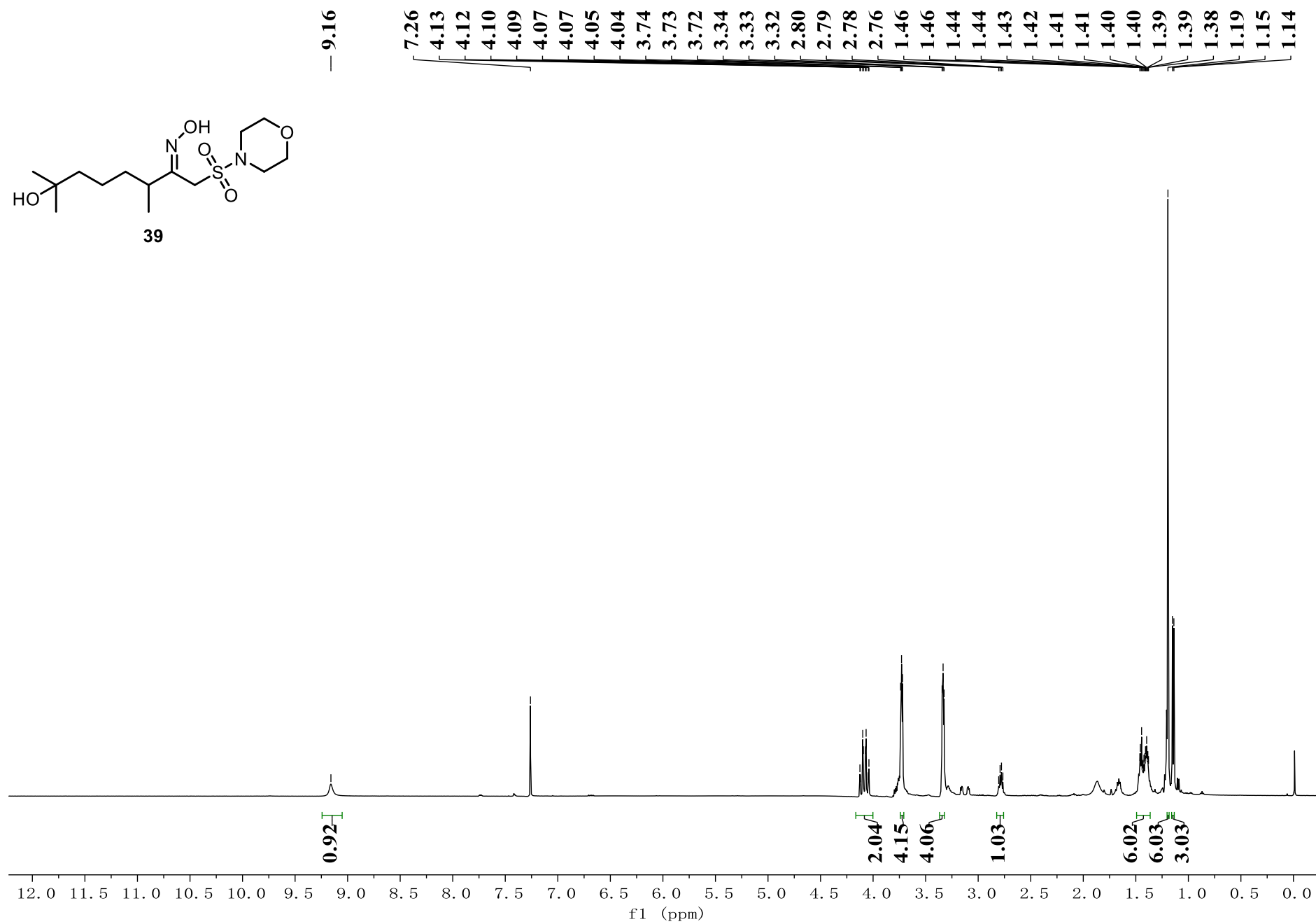
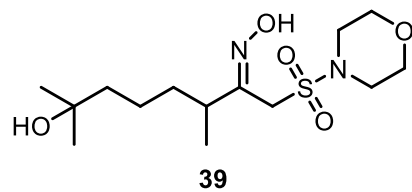
¹³C NMR (125 MHz, CD₃CN) for 38



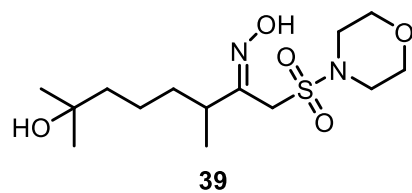
38



¹H NMR (500 MHz, CDCl₃) for 39

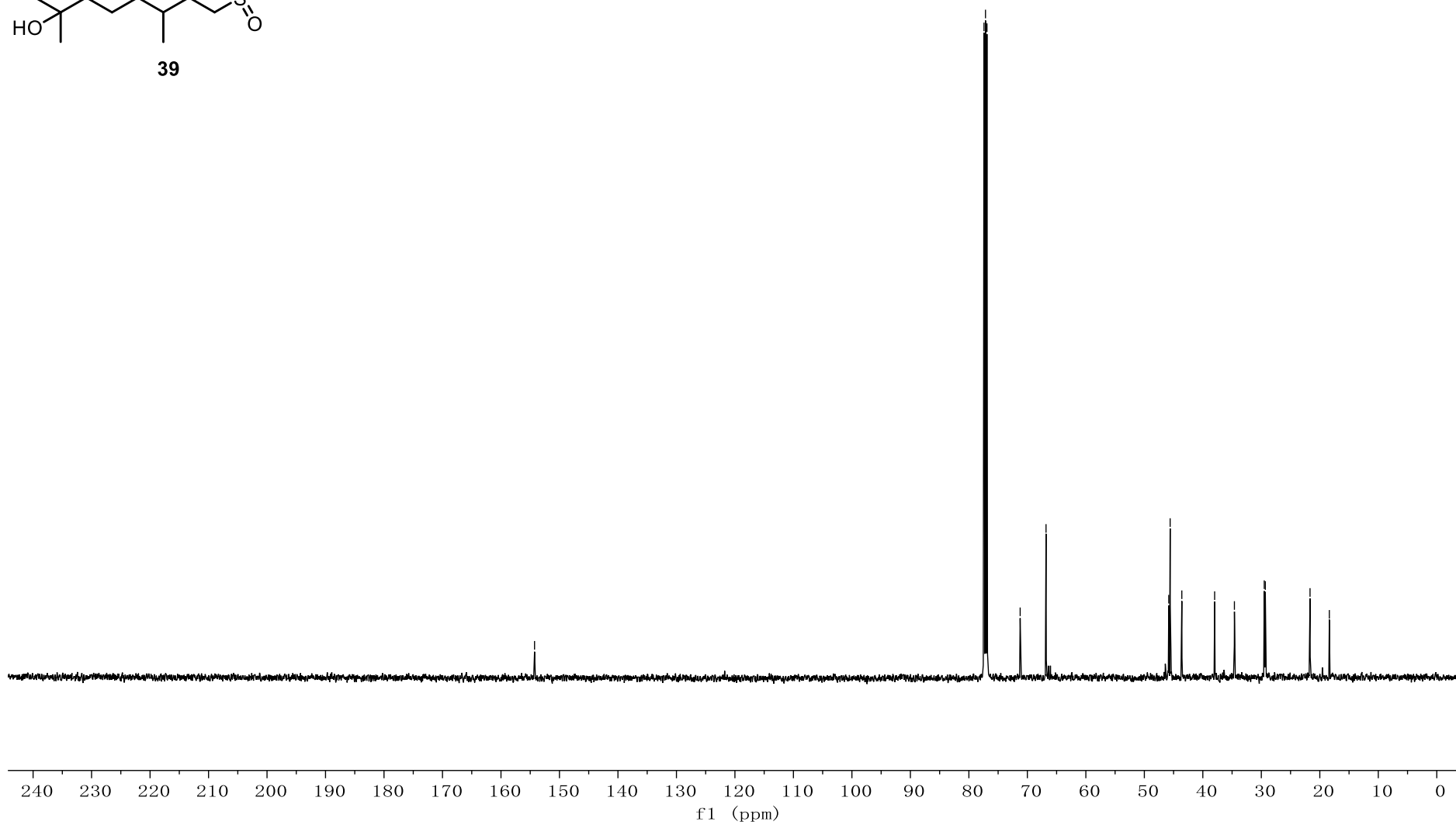


¹³C NMR (125 MHz, CDCl₃) for 39



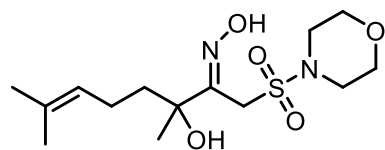
— 154.26

77.41
77.16
76.91
71.23
66.81
45.83
45.59
43.59
37.98
34.60
29.50
29.32
21.67
18.37

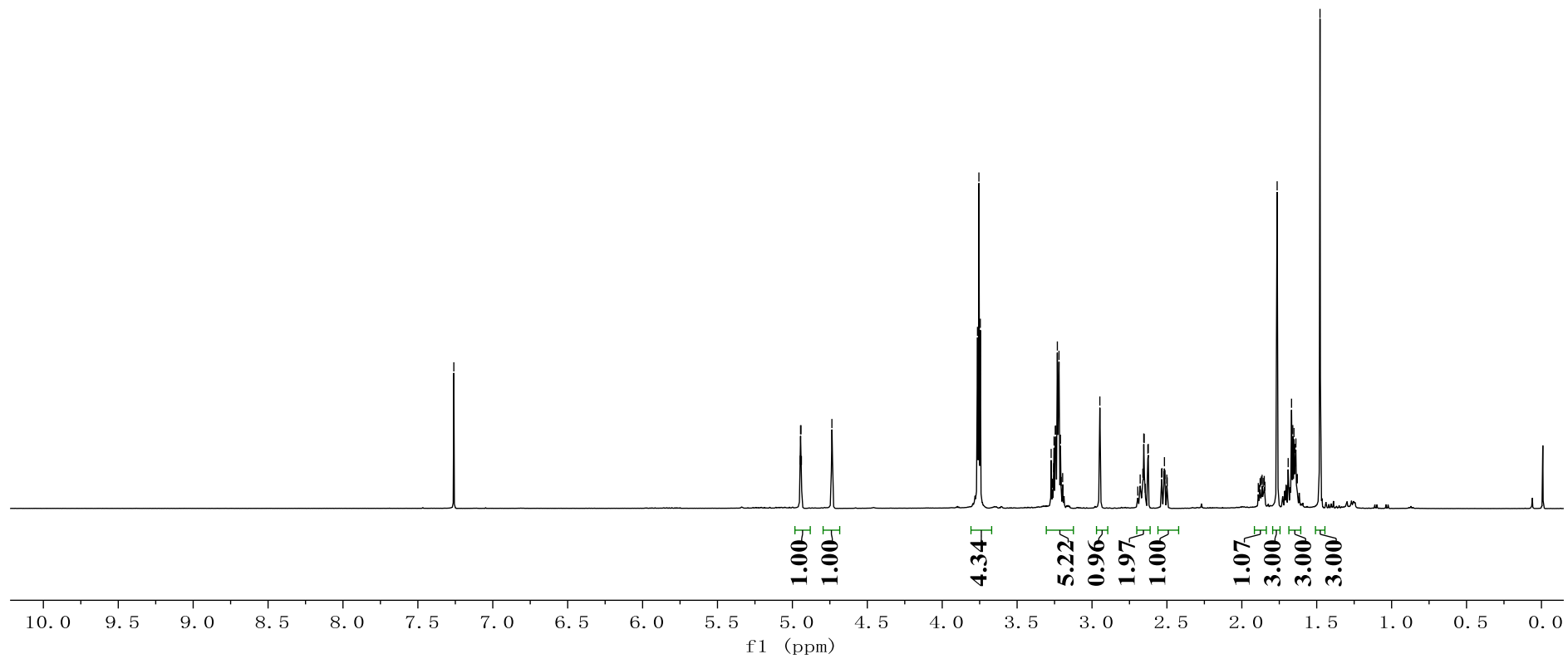


¹H NMR (500 MHz, CDCl₃) for 40

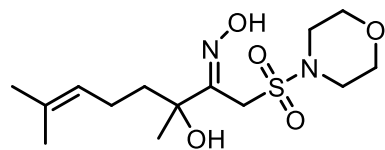
7.26
4.95
4.95
4.94
4.94
4.74
3.76
3.76
3.75
3.27
3.26
3.26
3.25
3.25
3.24
3.23
3.23
3.22
3.21
3.21
3.20
2.95
2.68
2.66
2.65
2.65
2.63
2.62
2.54
2.53
2.52
2.52
2.51
2.50
2.50
1.88
1.87
1.86
1.86
1.85
1.76
1.69
1.67
1.66
1.65
1.65
1.64
1.63
1.63
1.48



40



¹³C NMR (125 MHz, CDCl₃) for 40



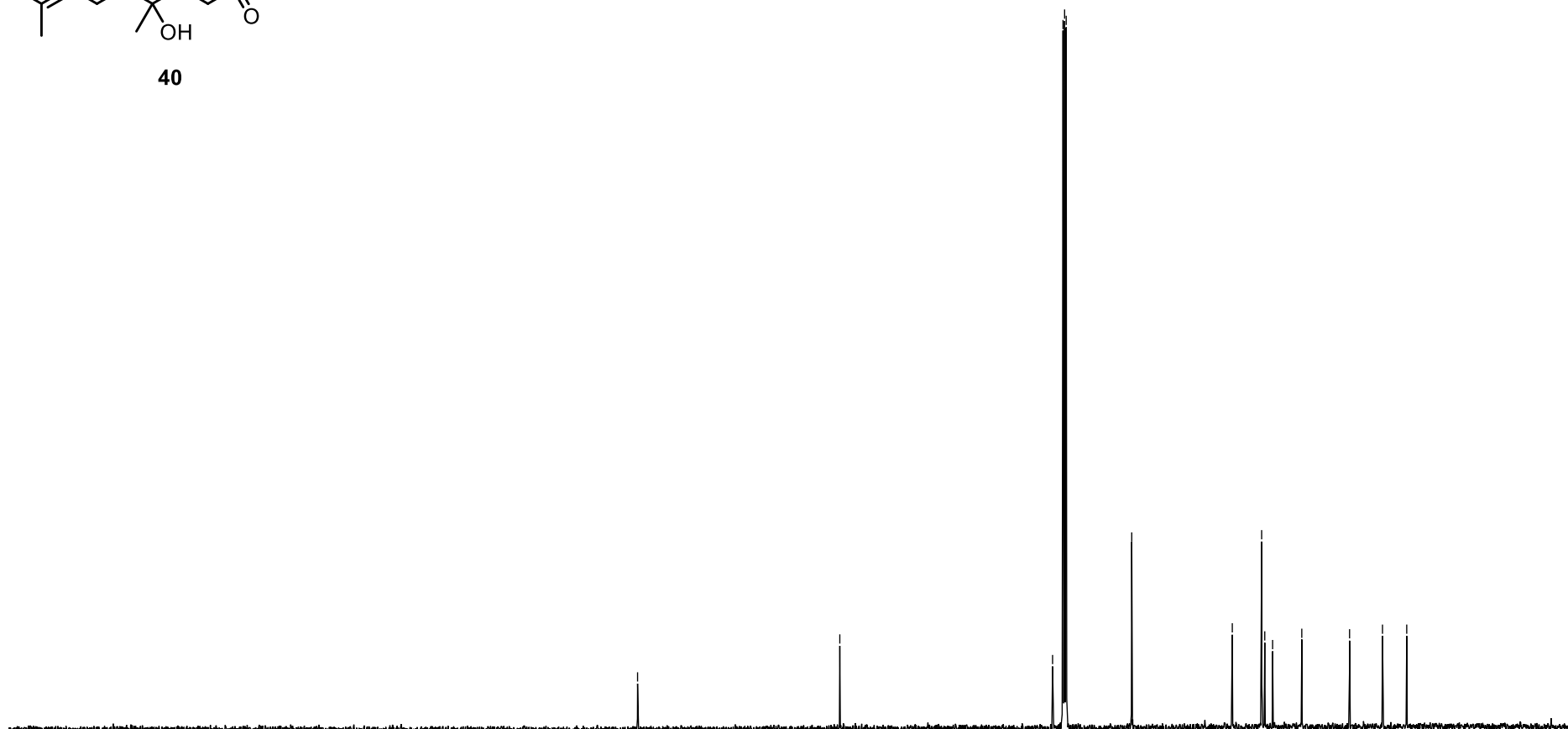
40

— 144.75

— 112.74

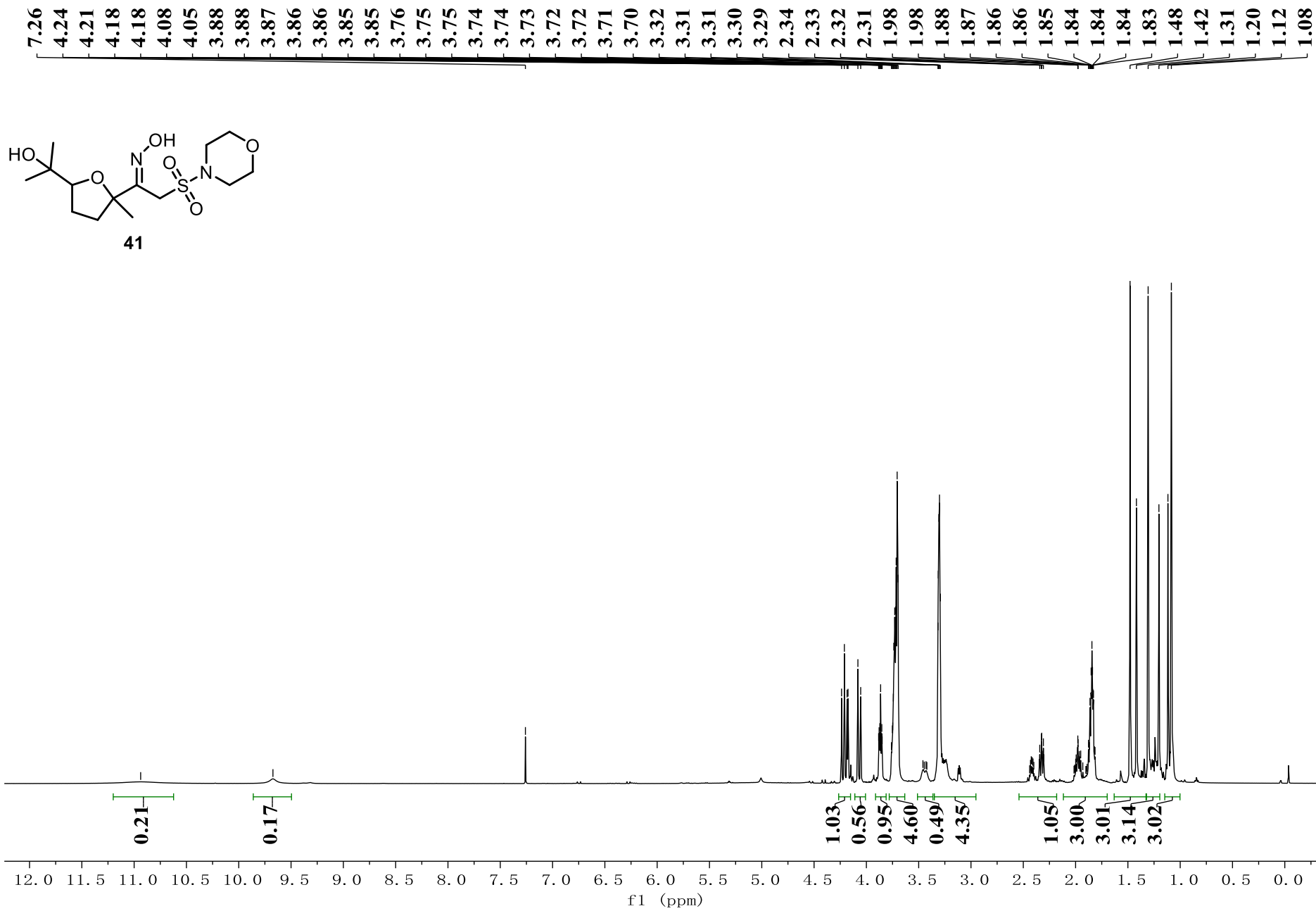
79.06
77.41
77.16
76.91
— 66.54

50.62
45.97
45.48
44.24
39.62
32.06
26.84
23.01

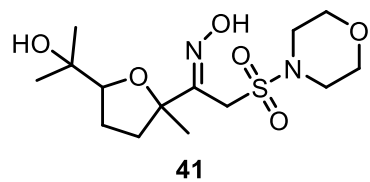


f1 (ppm)

¹H NMR (500 MHz, CDCl₃) for 41

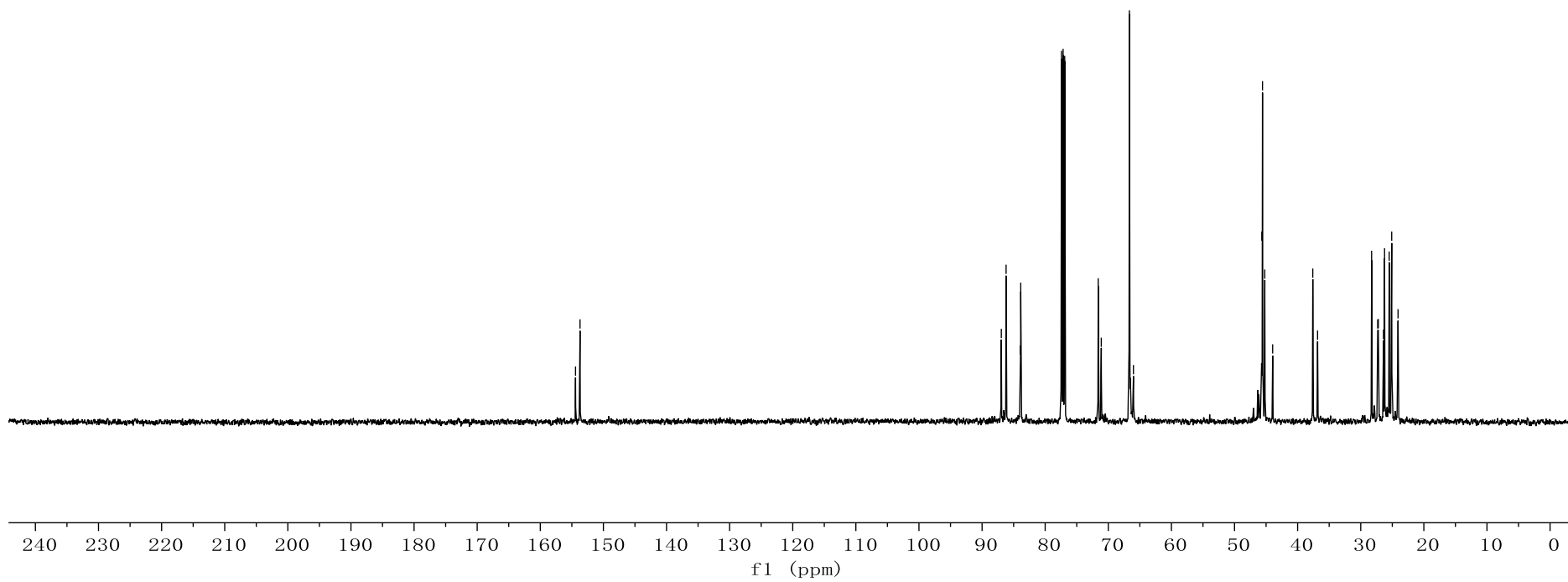


¹³C NMR (125 MHz, CDCl₃) for 41

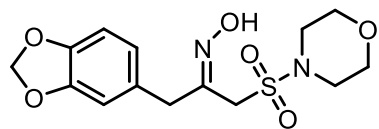


154.42
153.70

86.95
86.19
83.96
83.87
77.42
77.16
76.91
71.60
71.11
66.66
66.02
45.68
45.57
45.21
37.60
28.28
27.31
27.22
26.40
26.23
25.49
25.09
24.00



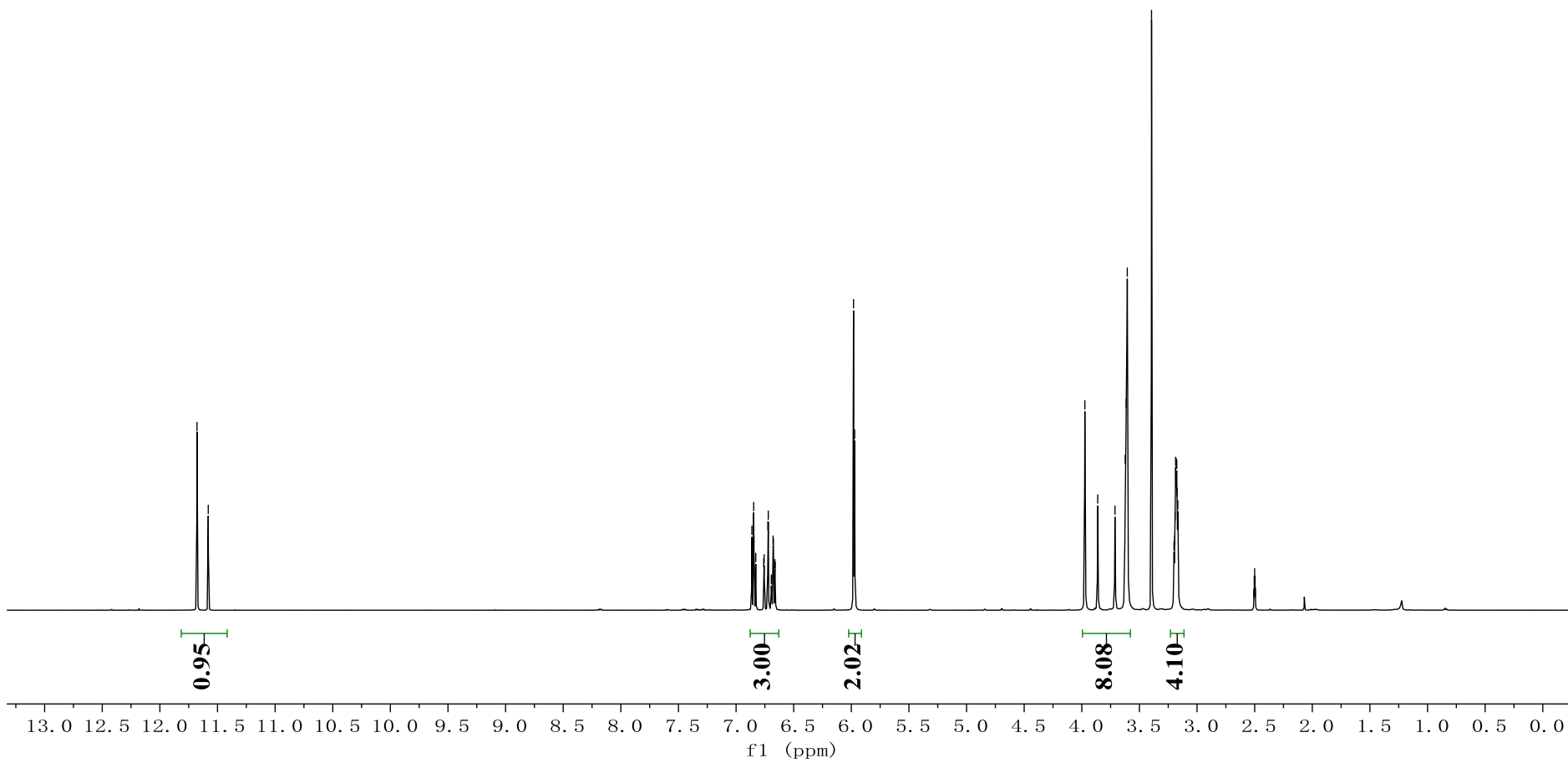
¹H NMR (500 MHz, DMSO-d₆) for 42



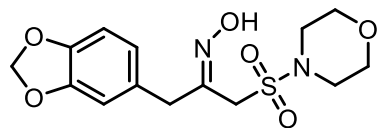
42

11.68
11.58

6.86
6.85
6.85
6.83
6.76
6.76
6.72
6.72
6.69
6.69
6.68
6.68
6.66
6.66
5.98
5.97
3.97
3.86
3.71
3.62
3.61
3.61
3.39
3.20
3.19
3.19
3.19
3.18
3.18
3.17
3.17
2.51
2.50
2.50
2.50
2.49



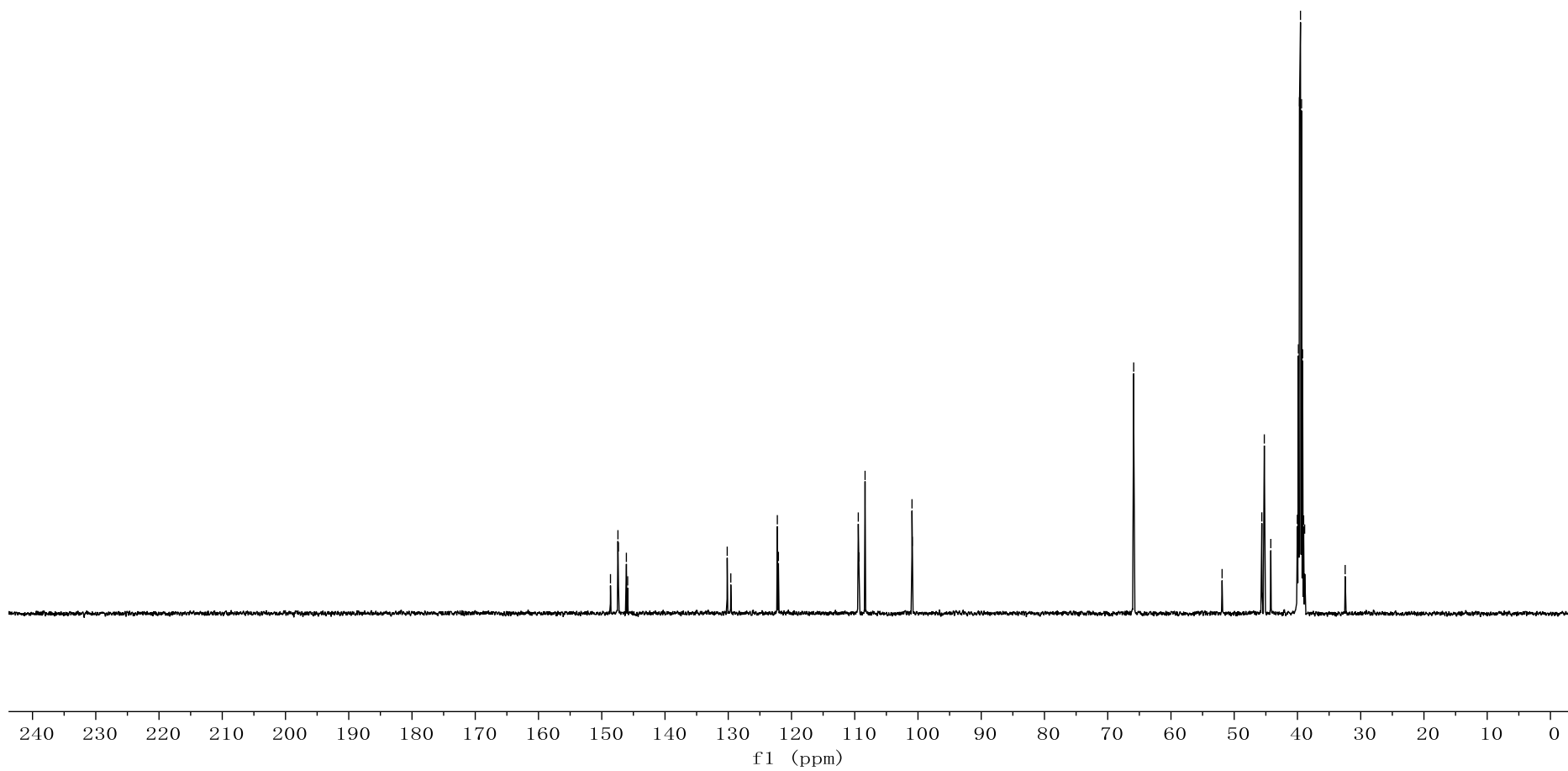
¹³C NMR (125 MHz, DMSO-*d*₆) for 42



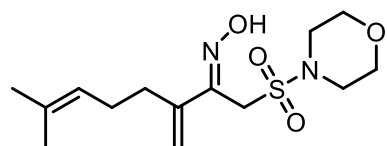
42

148.62
147.44
147.37
146.11
145.90
130.16
129.60
122.24
122.07
109.44
109.34
108.37
100.94
100.91

65.89
51.92
45.64
45.24
44.21
40.02
39.85
39.69
39.52
39.35
39.19
39.02
38.87
32.46



¹H NMR (500 MHz, CDCl₃) for 43



43

8.38
8.05
8.03

7.26

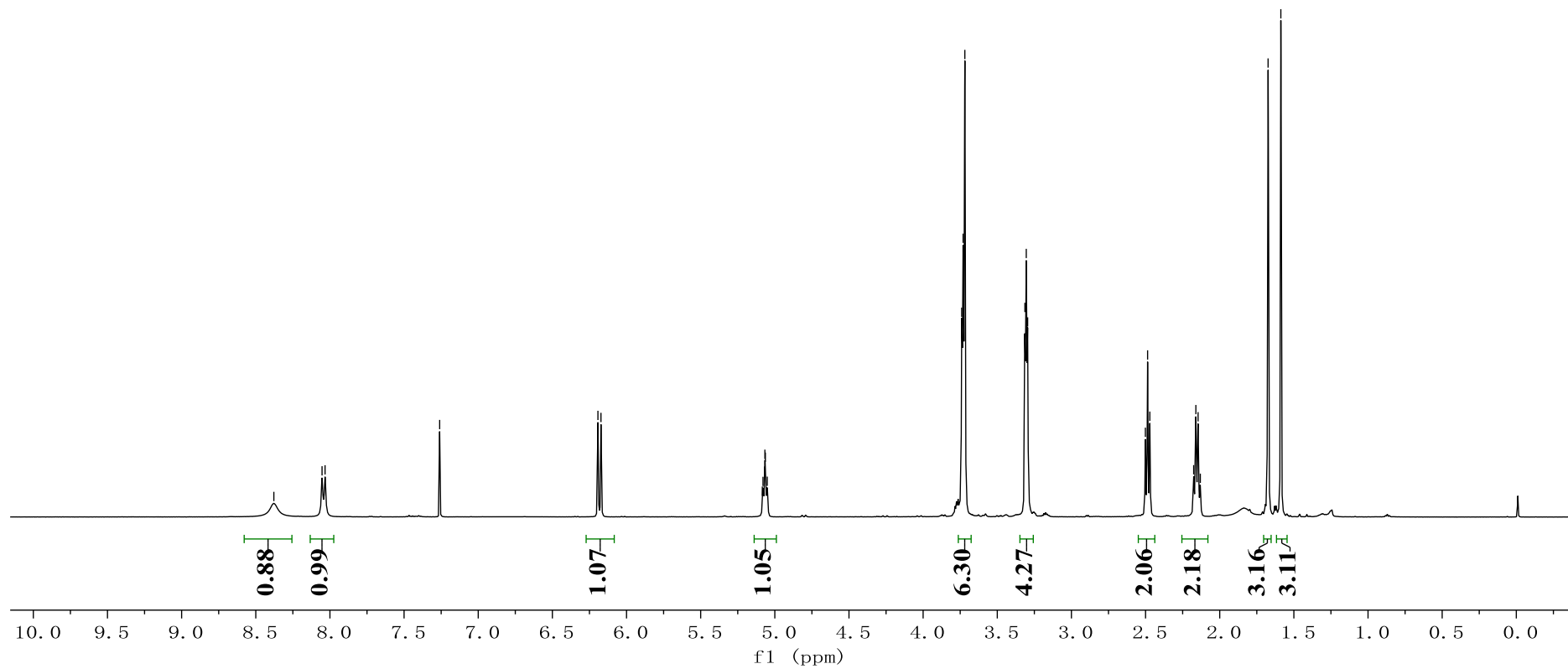
6.19
6.17

5.08
5.08
5.07
5.07
5.06
5.05

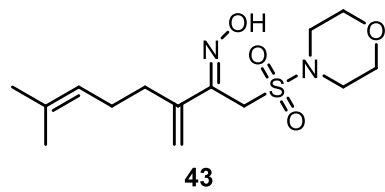
3.74
3.73
3.72
3.31
3.30
3.30

2.50
2.49
2.47

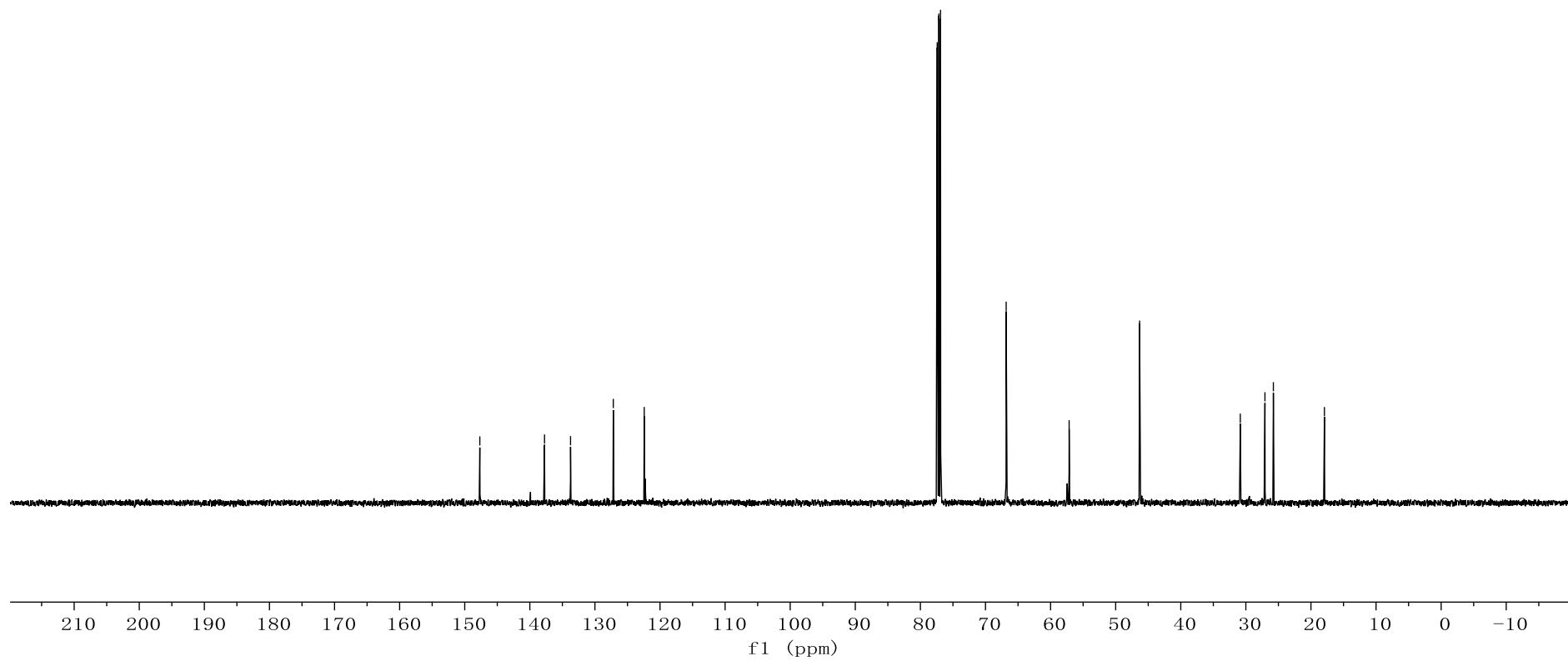
2.18
2.16
2.15
2.13
1.67
1.59



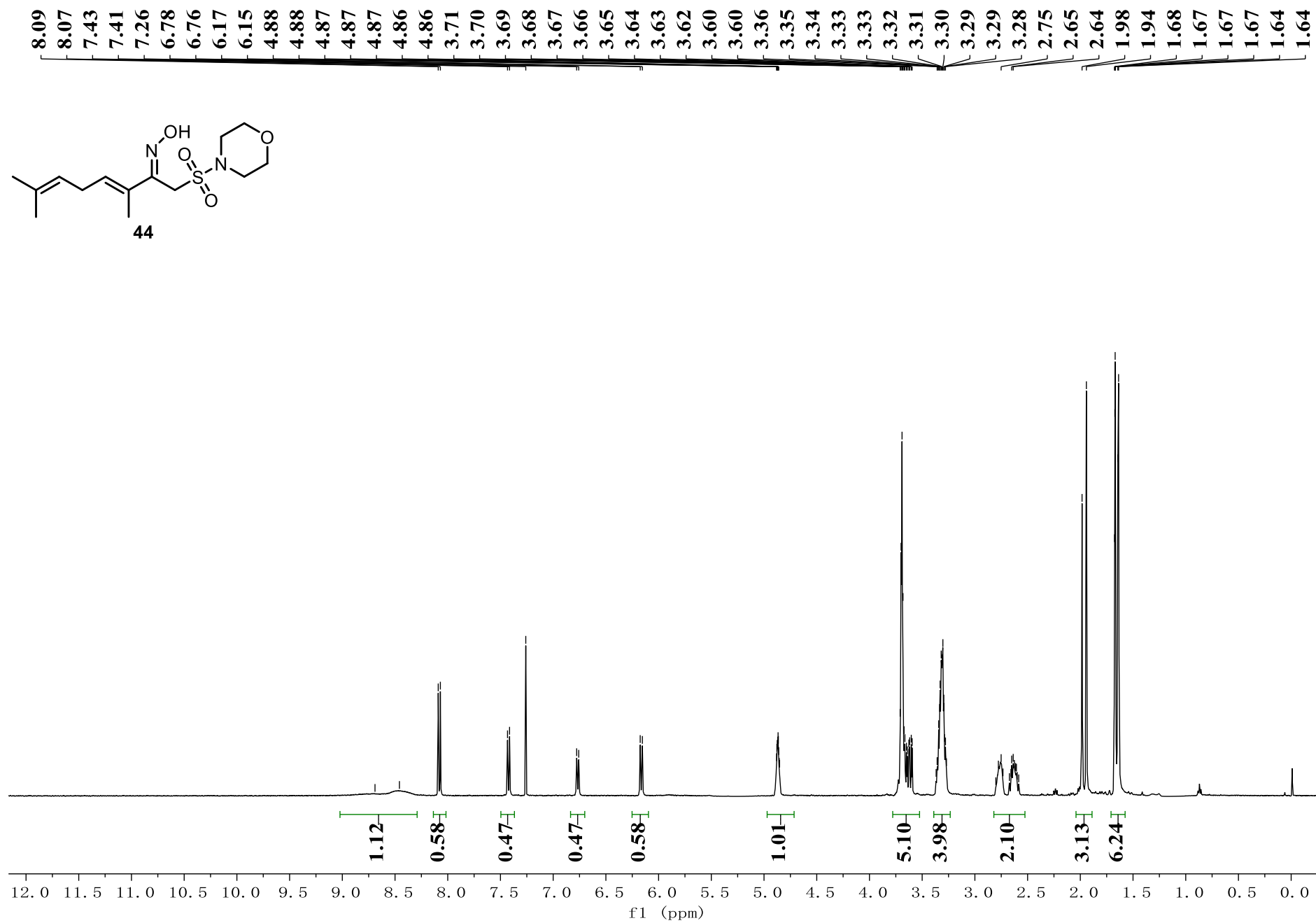
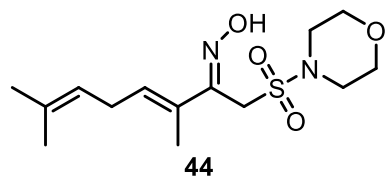
¹³C NMR (125 MHz, CDCl₃) for 43



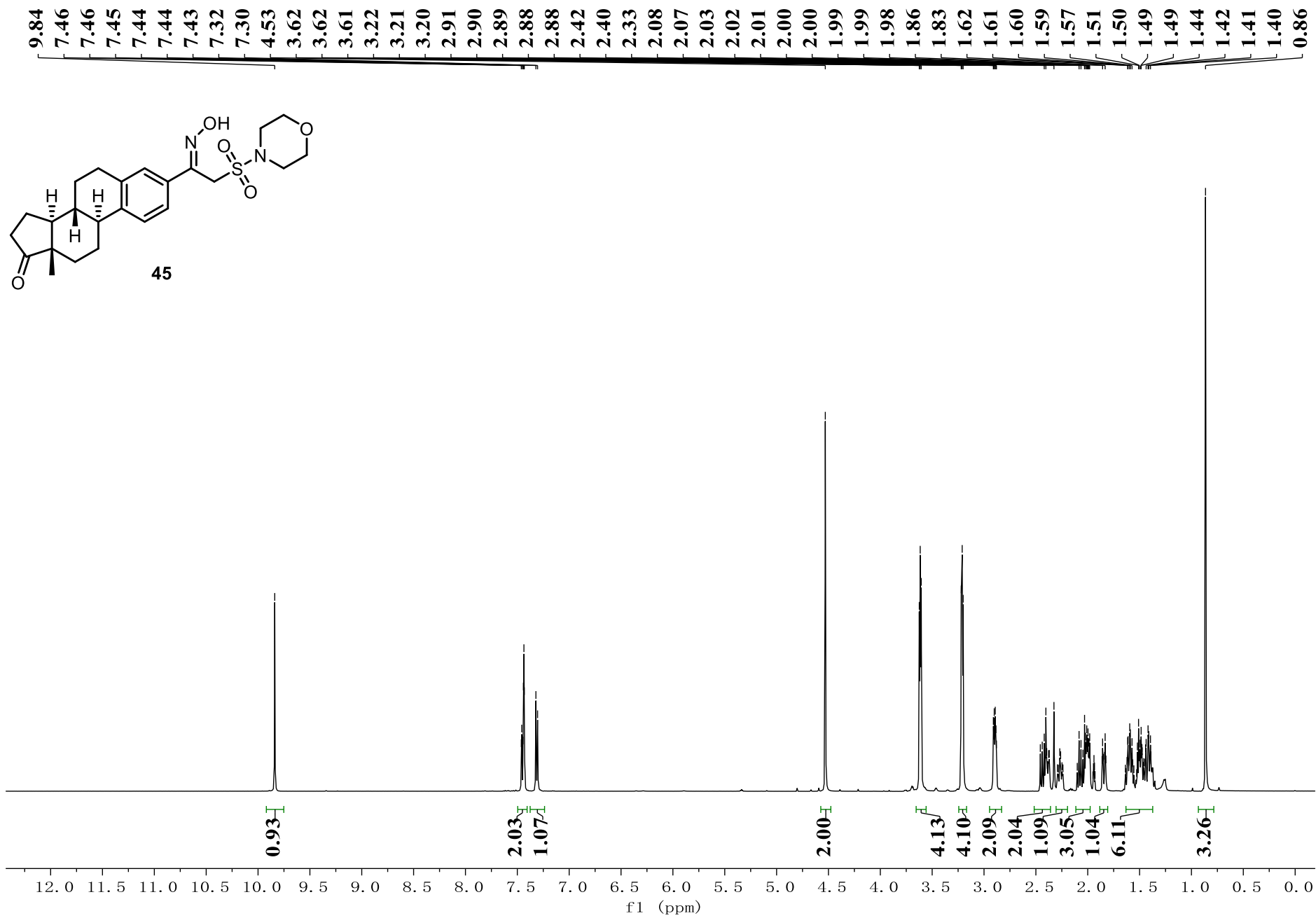
- 147.68
- ~ 137.74
- ~ 133.75
- ~ 127.18
- ~ 122.42
- 77.42
- 77.16
- 76.91
- 66.82
- 57.15
- 46.36
- 46.32
- ~ 30.85
- ~ 27.07
- ~ 25.77
- ~ 17.93



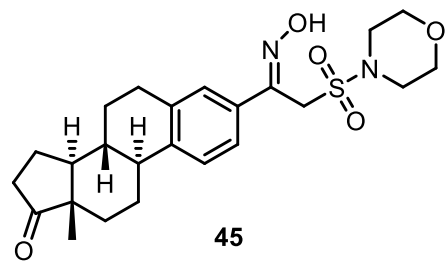
¹H NMR (500 MHz, CDCl₃) for 44



¹H NMR (500 MHz, CD₃CN) for 45

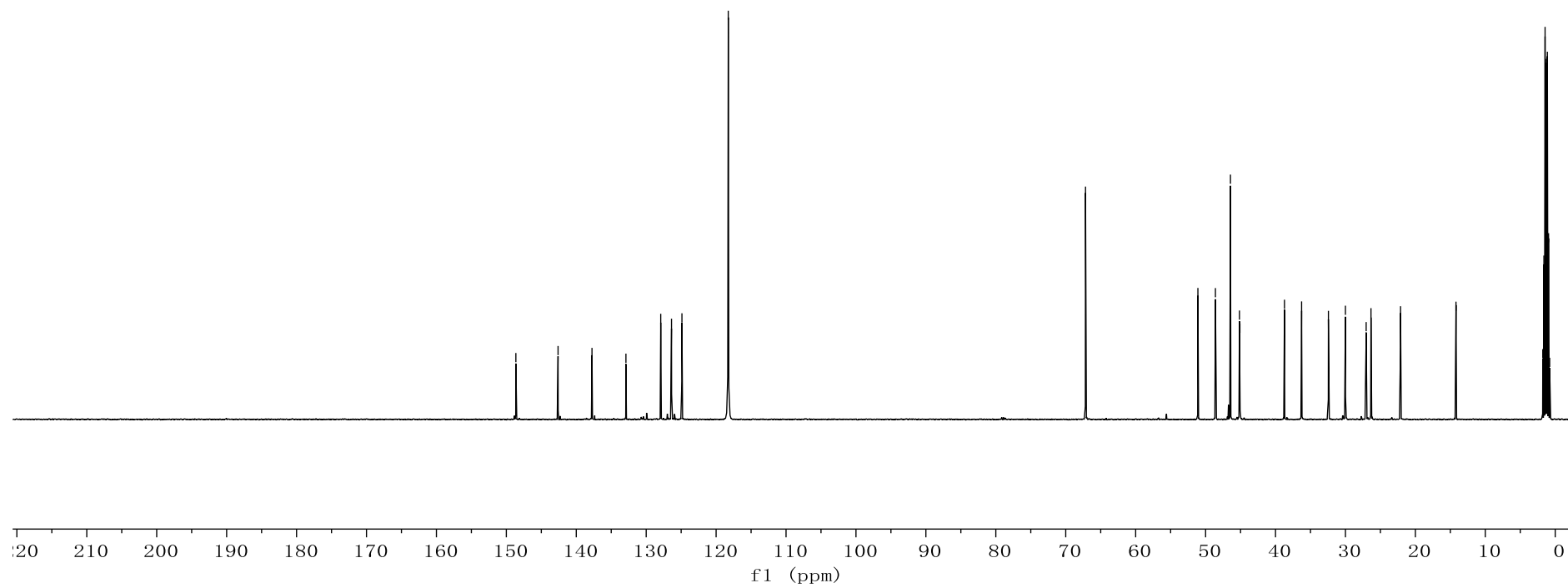


¹³C NMR (125 MHz, CD₃CN) for 45

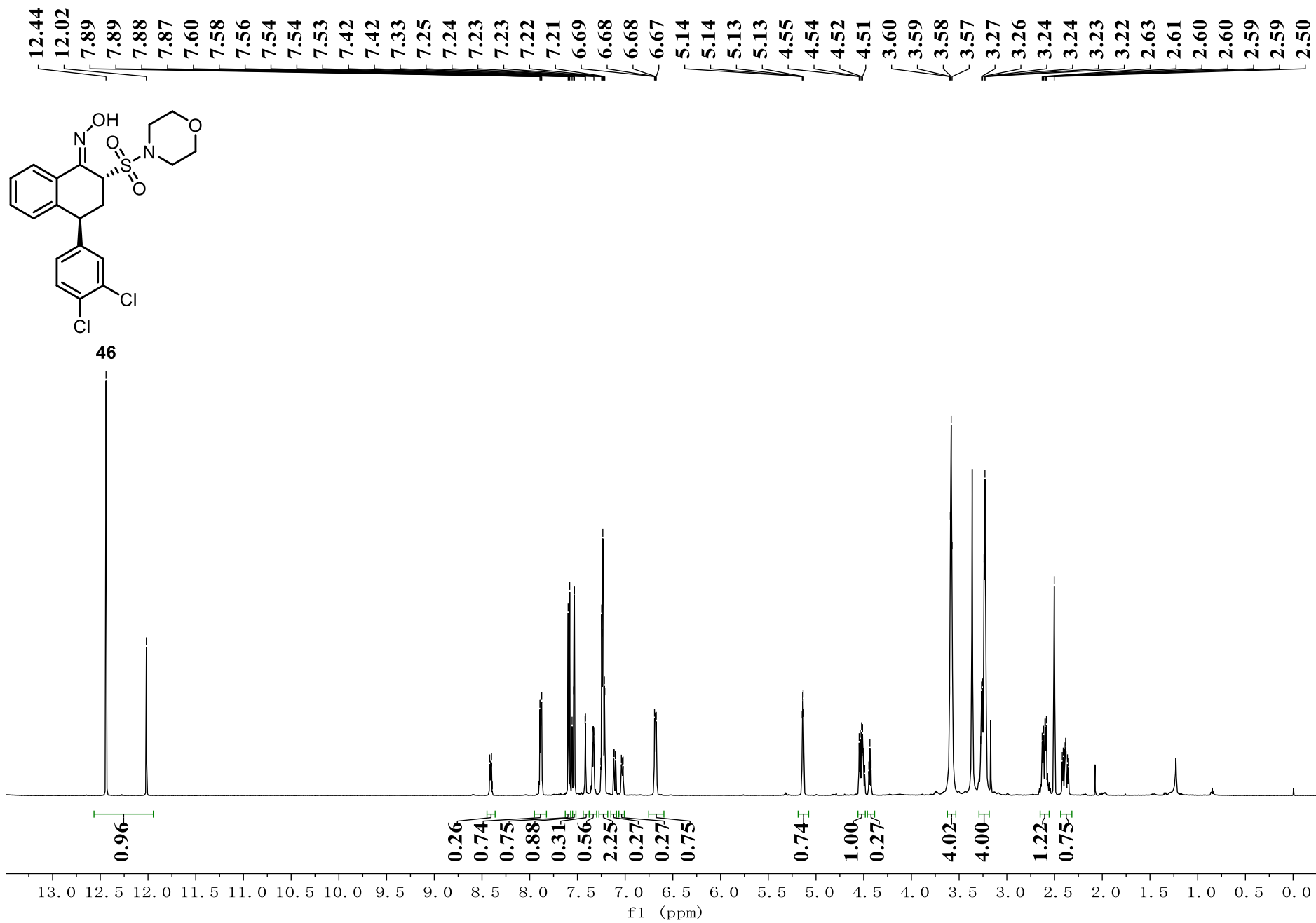


148.64
142.60
137.75
132.90
127.92
126.38
124.89
118.26

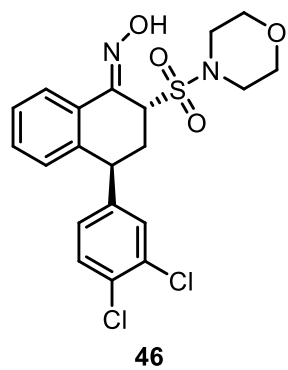
67.17
51.10
48.59
46.45
45.16
45.09
38.72
36.27
32.44
30.00
27.04
26.35
22.12
14.19
1.78
1.62
1.45
1.28
1.12
0.95
0.79



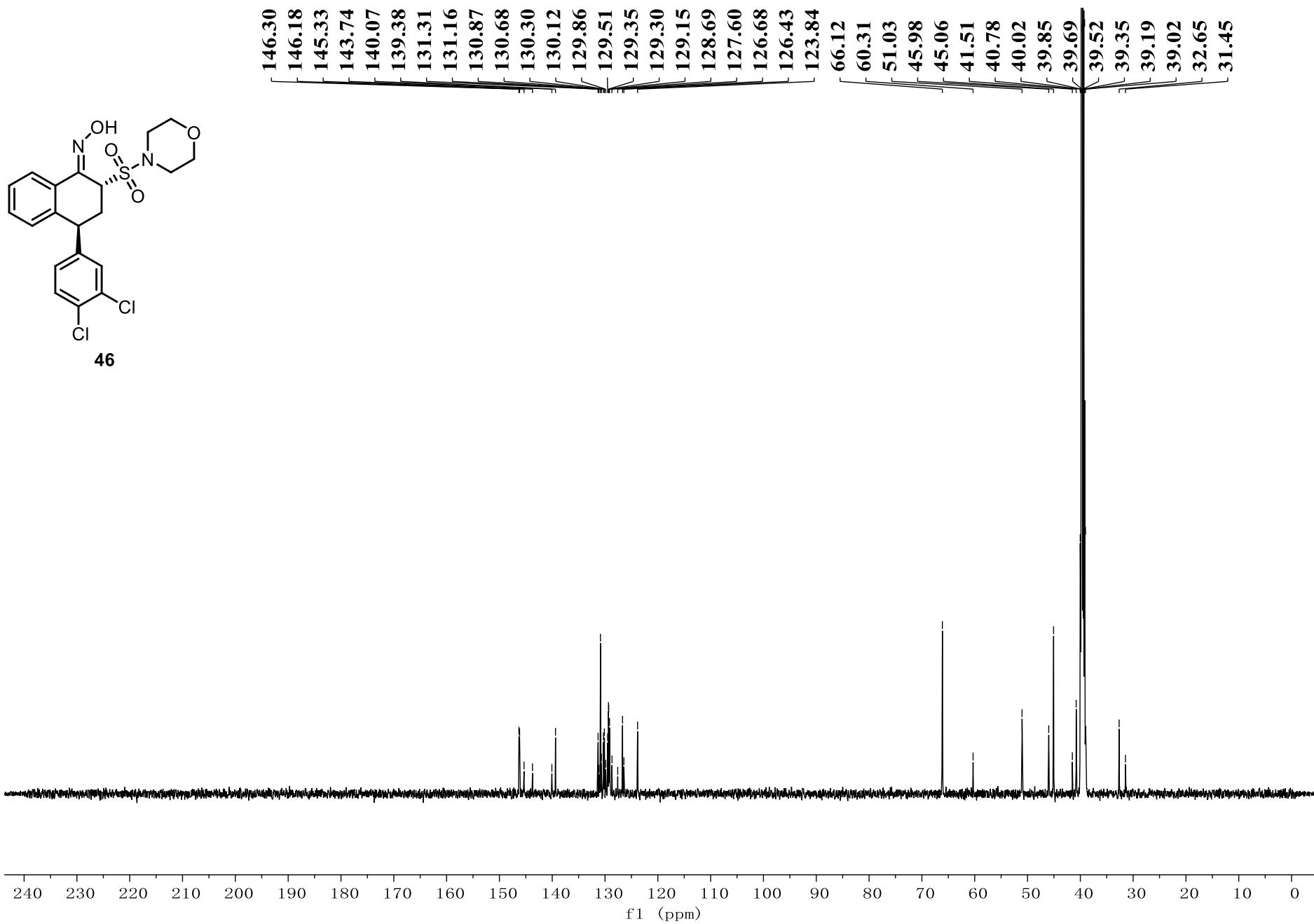
¹H NMR (500 MHz, DMSO-d₆) for 46



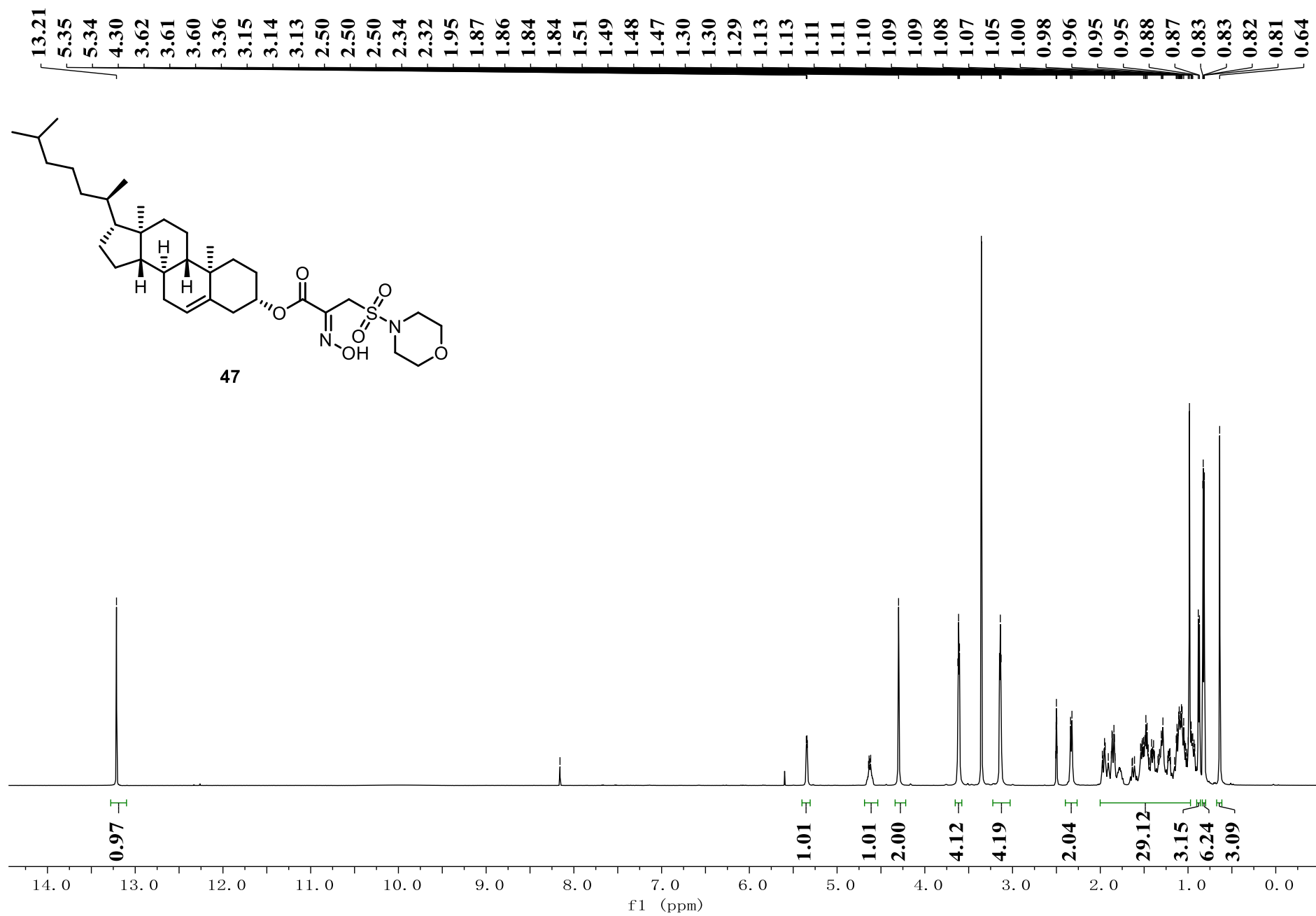
¹³C NMR (125 MHz, DMSO-*d*₆) for 46



146.30
146.18
145.33
143.74
140.07
139.38
131.31
131.16
130.87
130.68
130.30
130.12
129.86
129.51
129.35
129.30
129.15
128.69
127.60
126.68
126.43
123.84
66.12
60.31
51.03
45.98
45.06
41.51
40.78
40.02
39.85
39.69
39.52
39.35
39.19
39.02
32.65
31.45

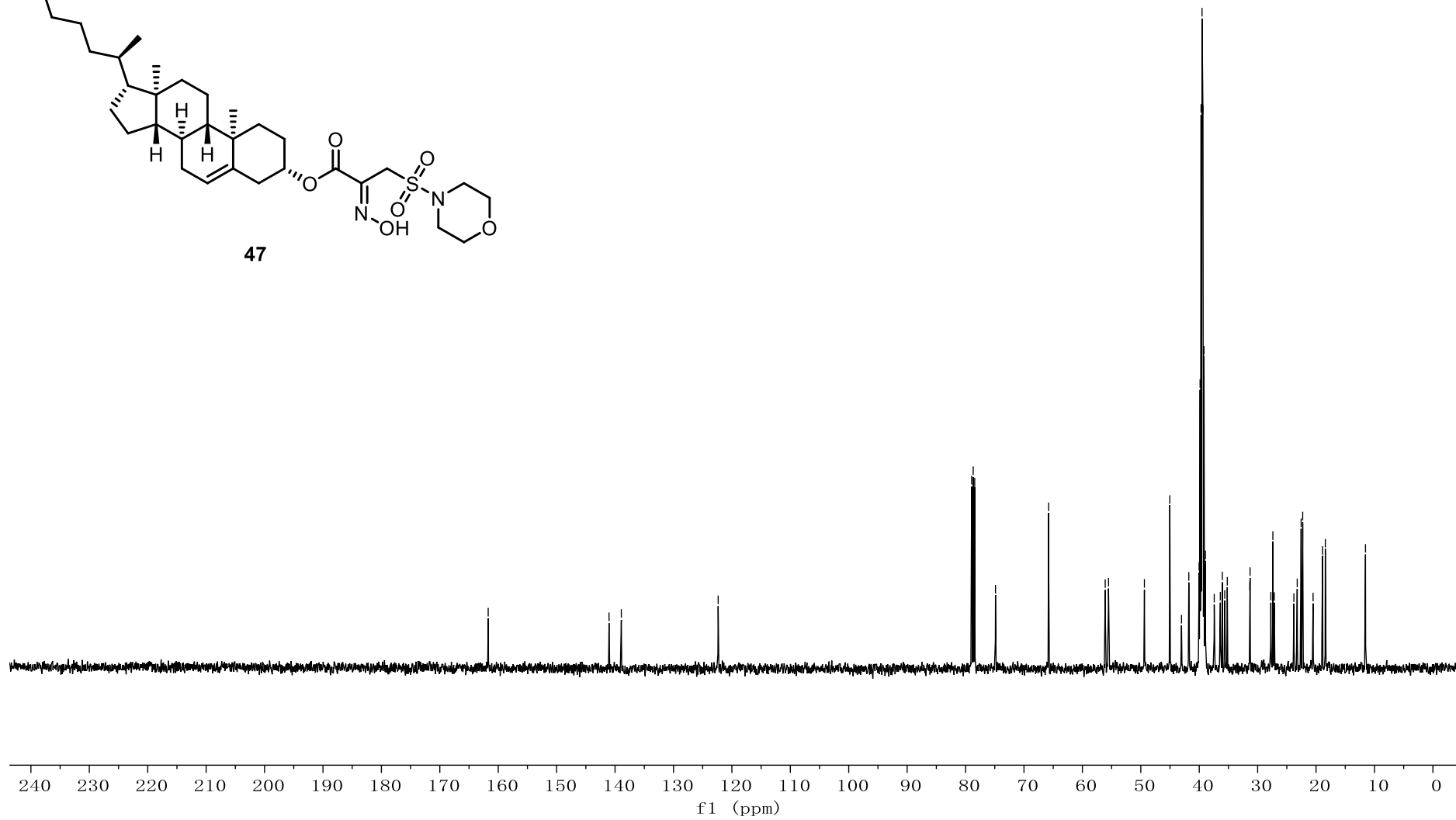
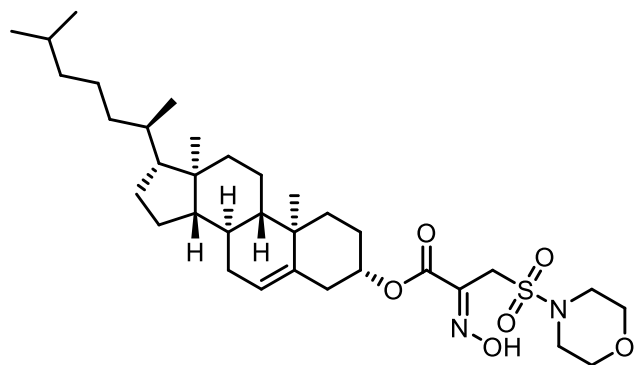


¹H NMR (500 MHz, DMSO-d₆:CDCl₃ = 2:1) for 47

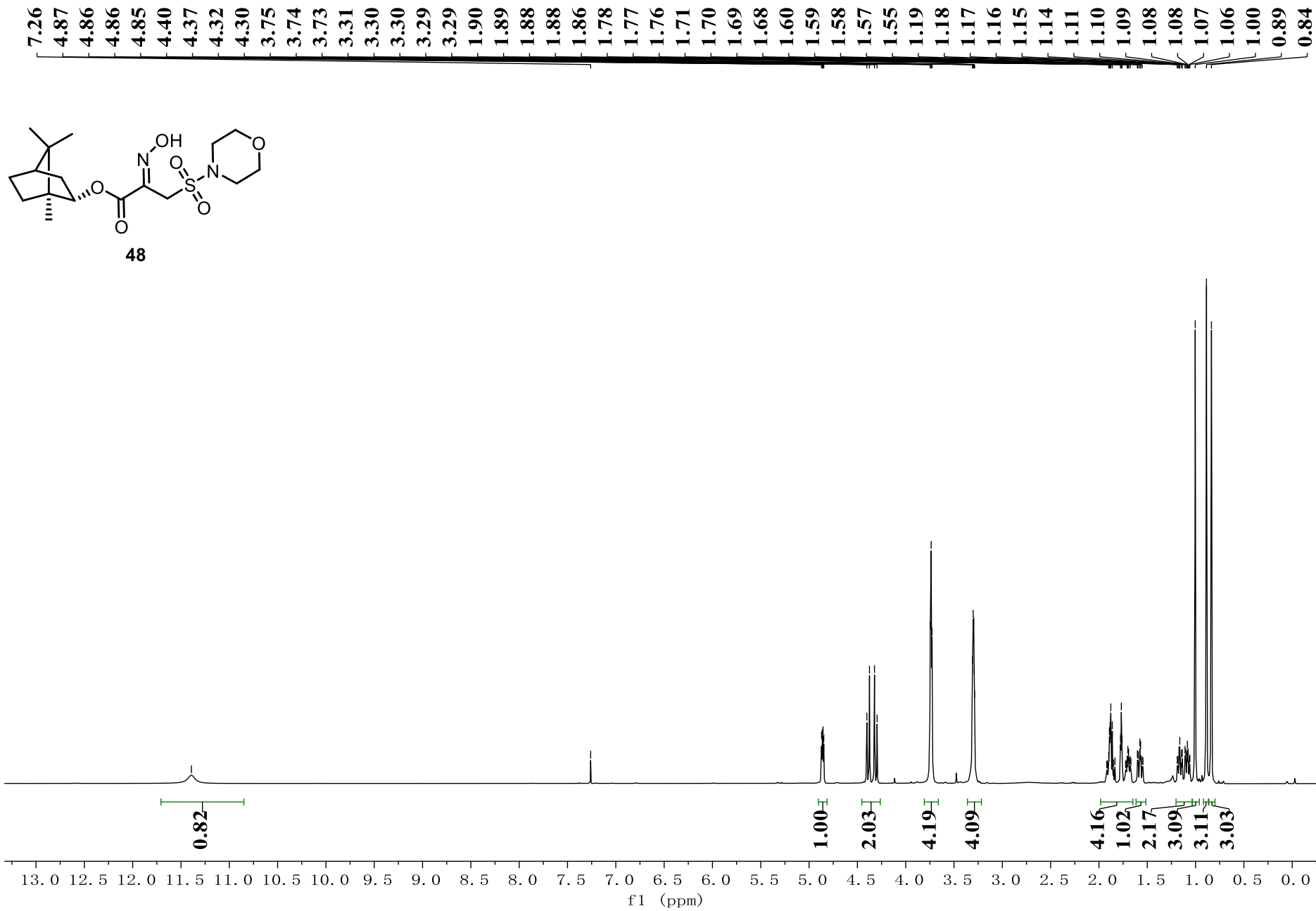


¹³C NMR (125 MHz, DMSO-*d*₆:CDCl₃ = 2:1) for 47

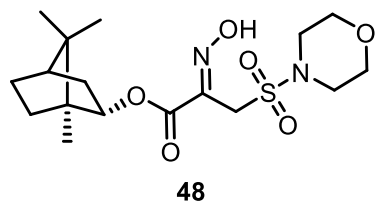
161.72
141.03
138.93
122.35
78.96
78.70
78.43
74.88
65.78
56.09
55.53
49.38
45.03
43.03
41.79
40.02
39.85
39.69
39.52
39.35
39.18
39.02
38.94
37.43
36.39
36.04
35.63
35.19
31.34
31.29
27.74
27.39
27.16
23.79
23.23
22.56
22.29
20.51
18.89
18.41
11.54



¹H NMR (500 MHz, CDCl₃) for 48



¹³C NMR (125 MHz, CDCl₃) for 48



— 162.14

— 142.99

84.03

77.42

77.16

76.91

— 66.85

49.10

47.10

45.43

45.05

44.03

38.57

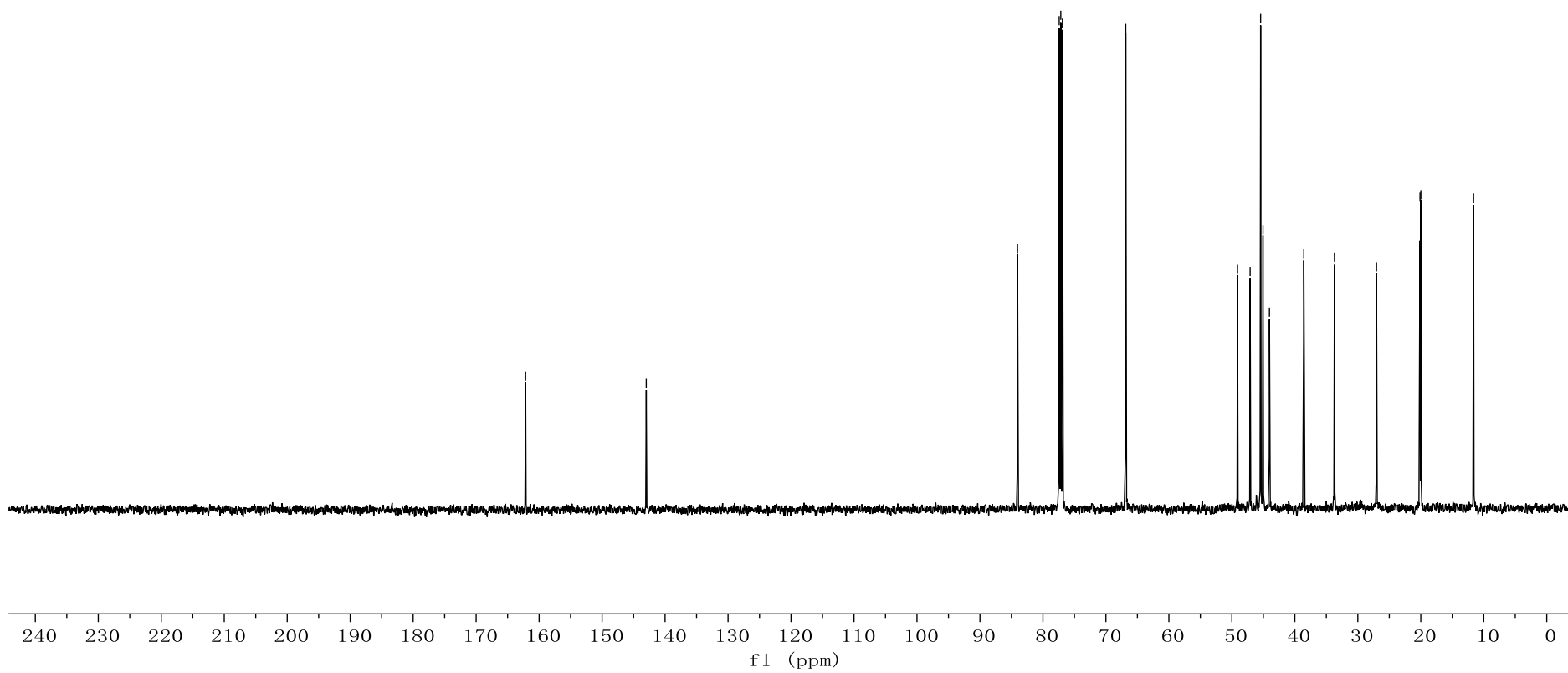
33.71

27.03

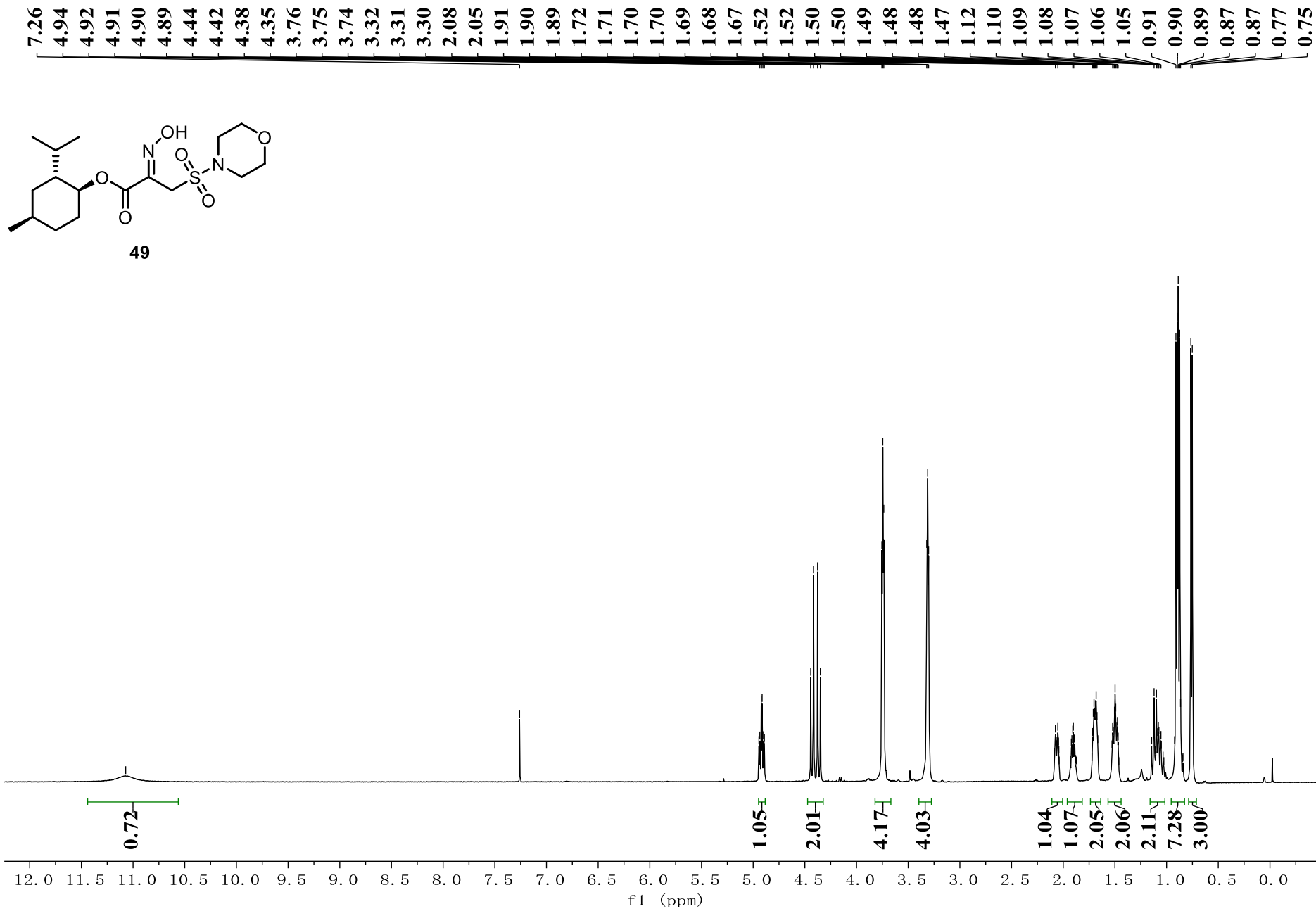
20.13

19.99

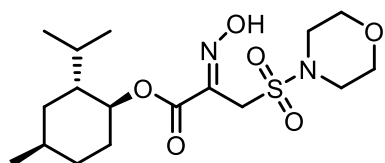
11.62



¹H NMR (500 MHz, CDCl₃) for 49



¹³C NMR (125 MHz, CDCl₃) for 49



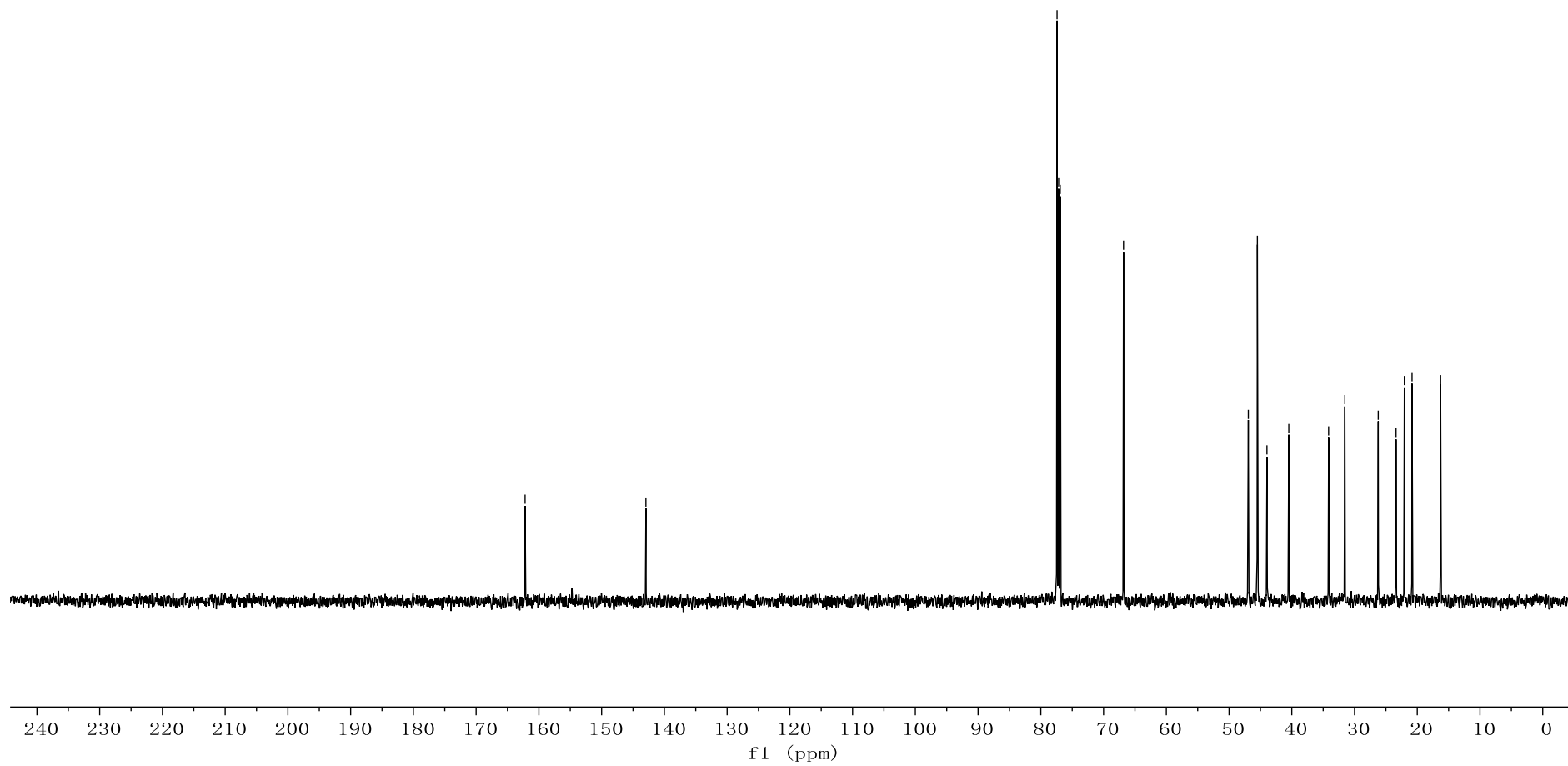
49

— 162.20

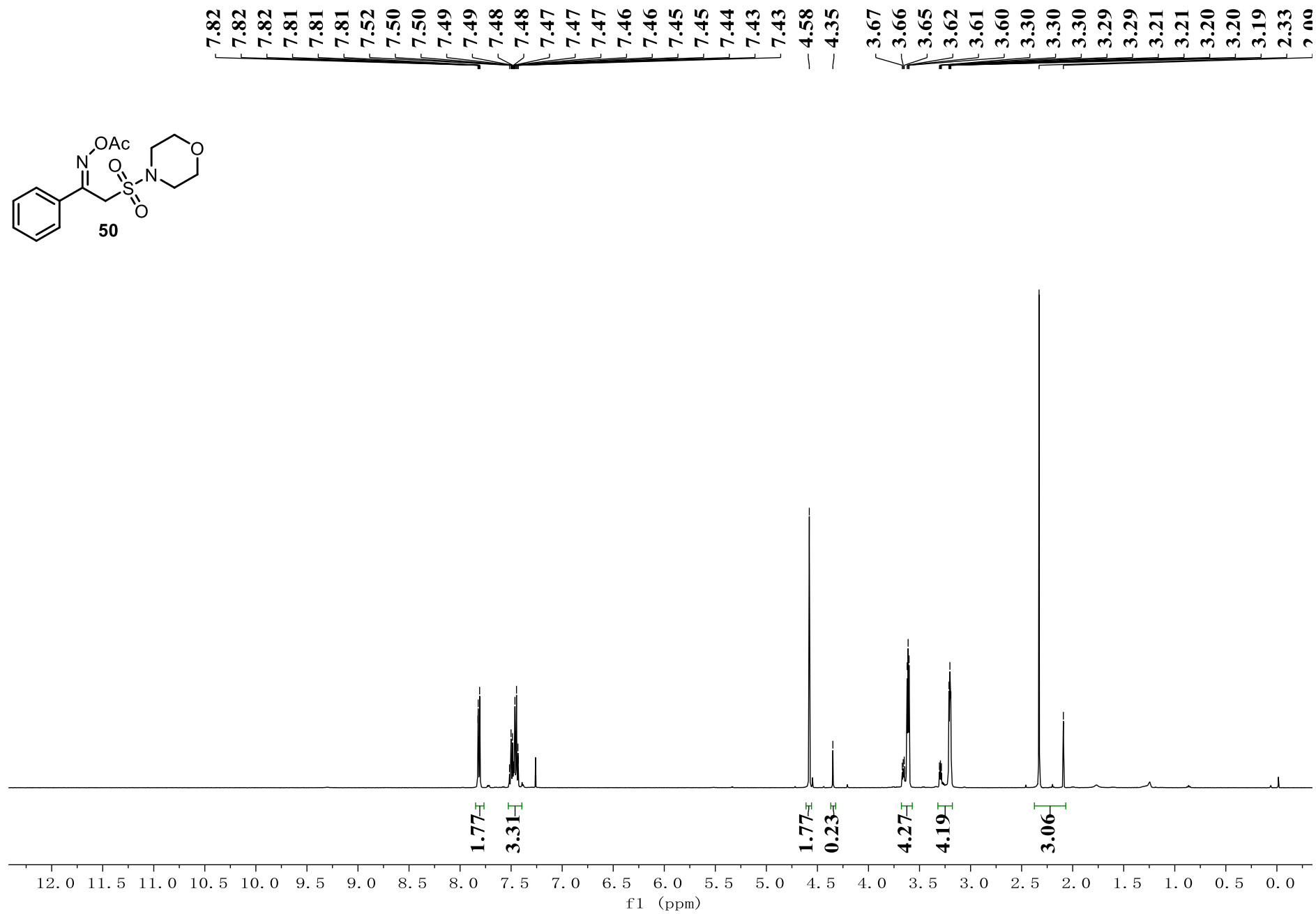
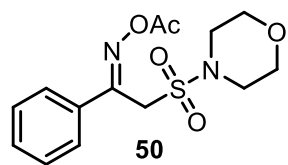
— 142.94

77.41
77.16
76.91
— 66.82

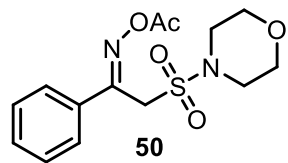
46.93
45.49
43.96
40.48
34.13
31.54
26.22
23.38
22.06
20.83
16.28



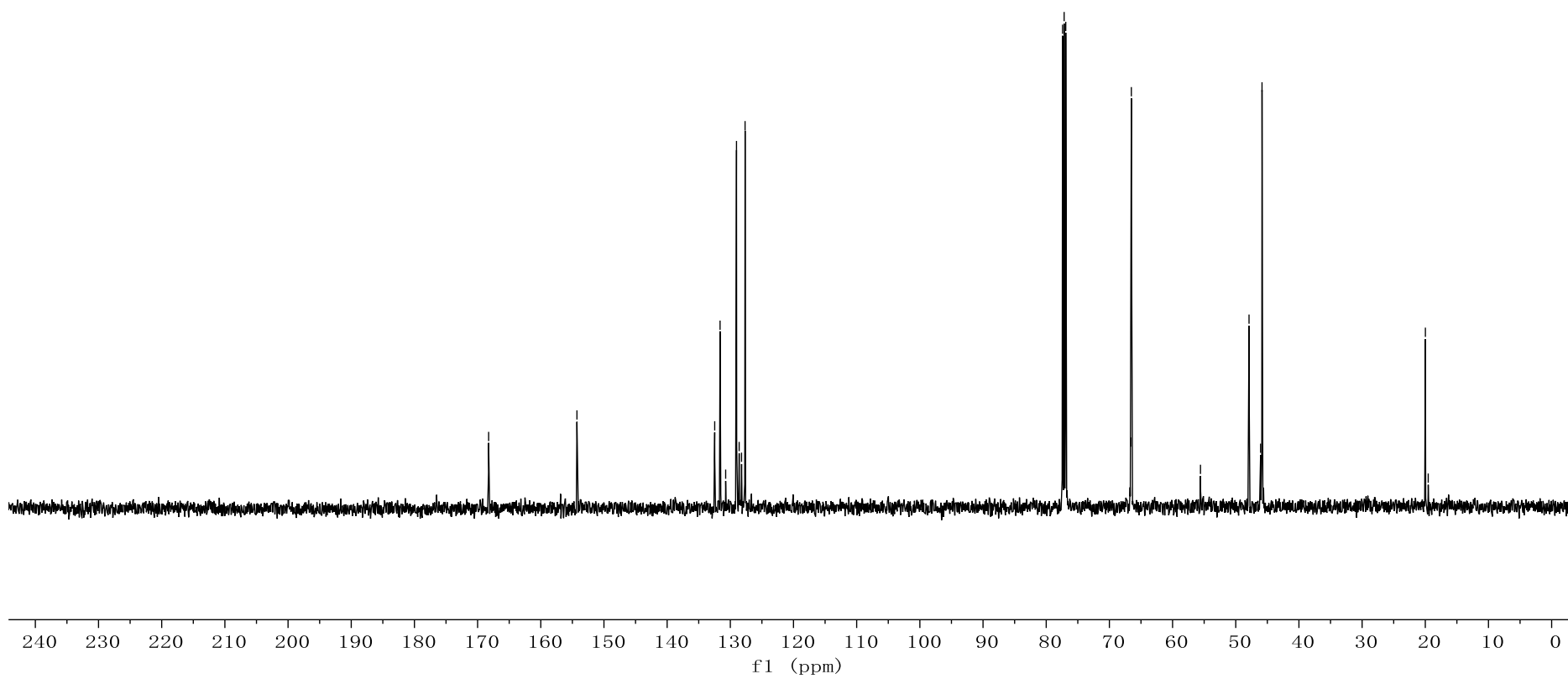
¹H NMR (500 MHz, CDCl₃) for 50



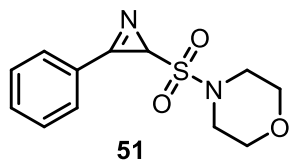
¹³C NMR (125 MHz, CDCl₃) for 50



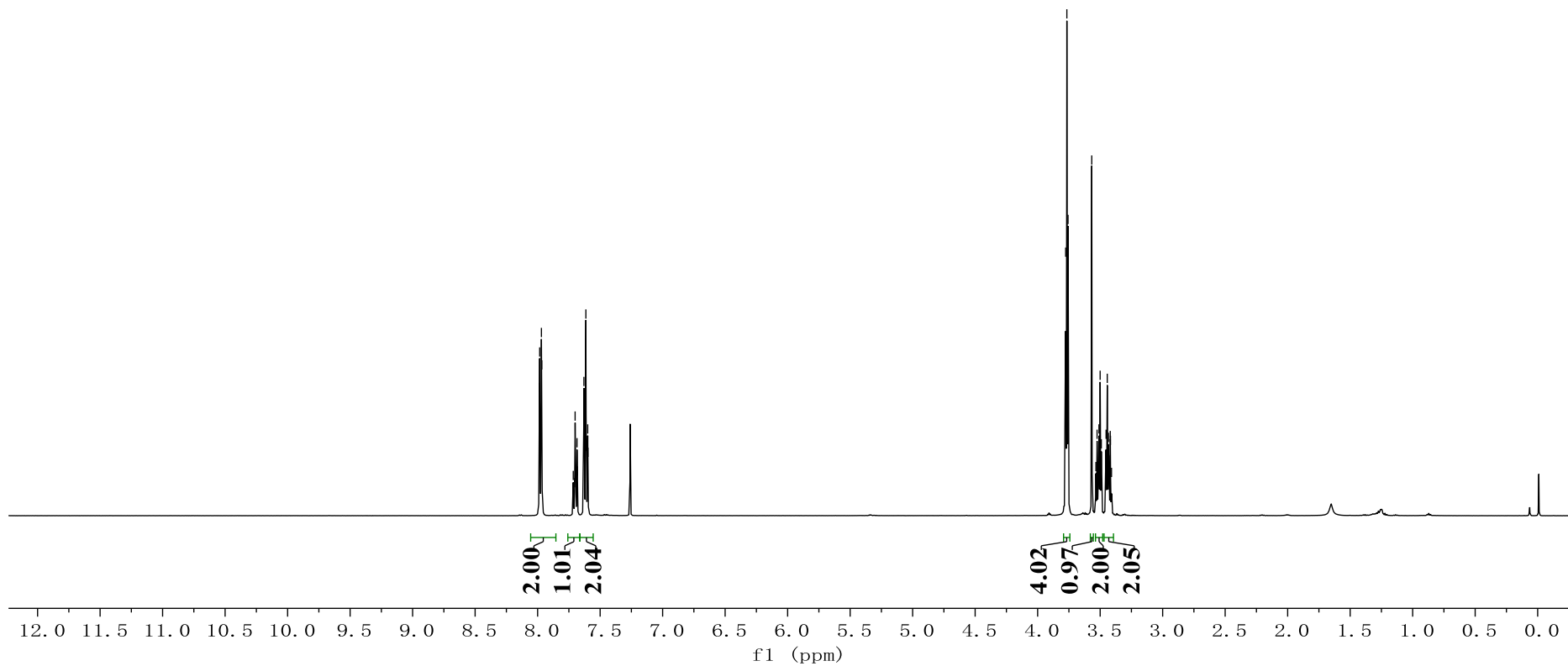
— 168.25
— 154.28
132.47
131.62
130.74
129.02
128.60
128.22
127.66
77.41
77.16
76.91
66.63
66.52
55.58
47.90
46.07
45.84
19.98
19.54



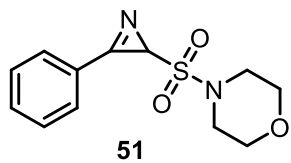
¹H NMR (500 MHz, CDCl₃) for 51



7.99
7.98
7.98
7.97
7.97
7.72
7.71
7.71
7.70
7.70
7.70
7.69
7.68
7.68
7.63
7.63
7.62
7.61
7.61
7.60
7.60
7.60
3.78
3.77
3.76
3.57
3.53
3.52
3.52
3.51
3.51
3.50
3.49
3.45
3.44
3.43
3.43
3.43
3.42
3.42
3.41



¹³C NMR (125 MHz, CDCl₃) for 51



— 161.48

~ 135.01

~ 131.09

~ 129.68

~ 121.44

{ 77.41

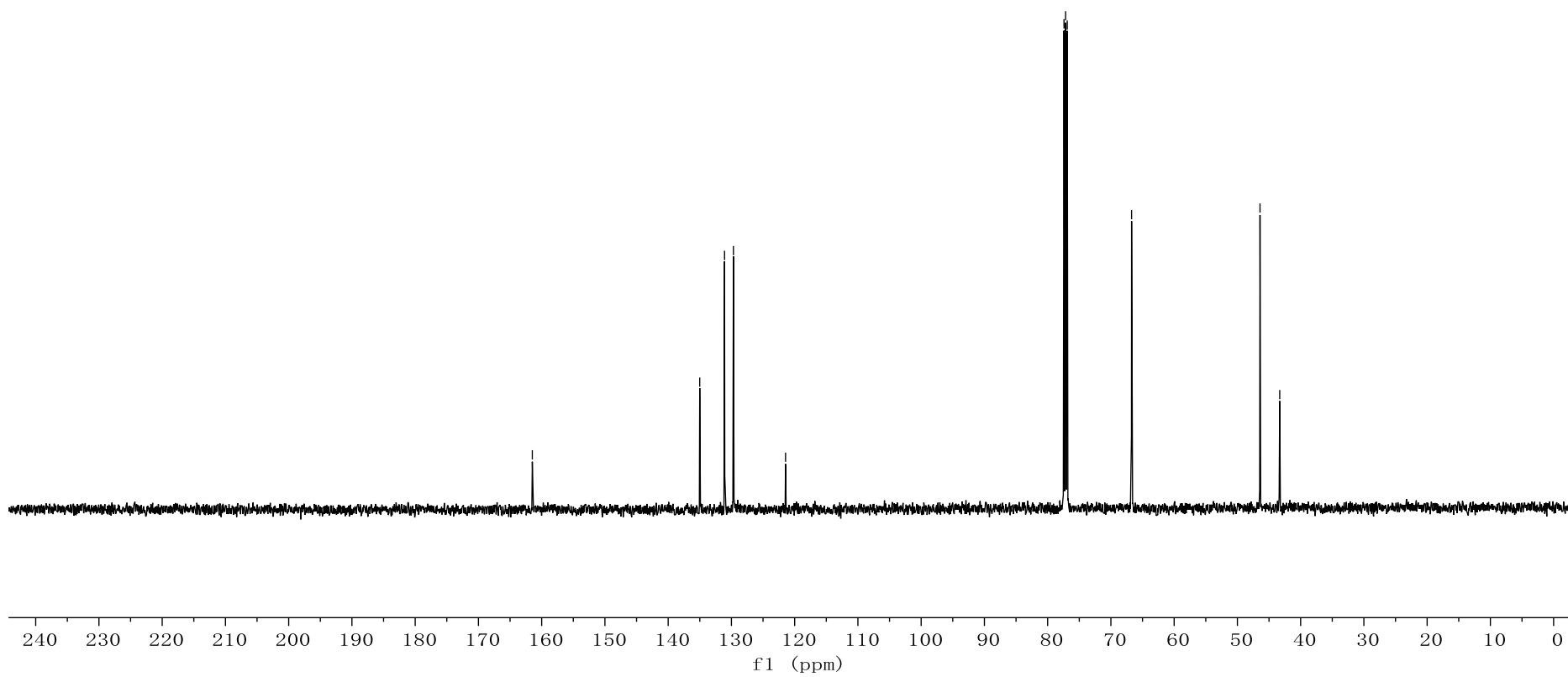
{ 77.16

{ 76.91

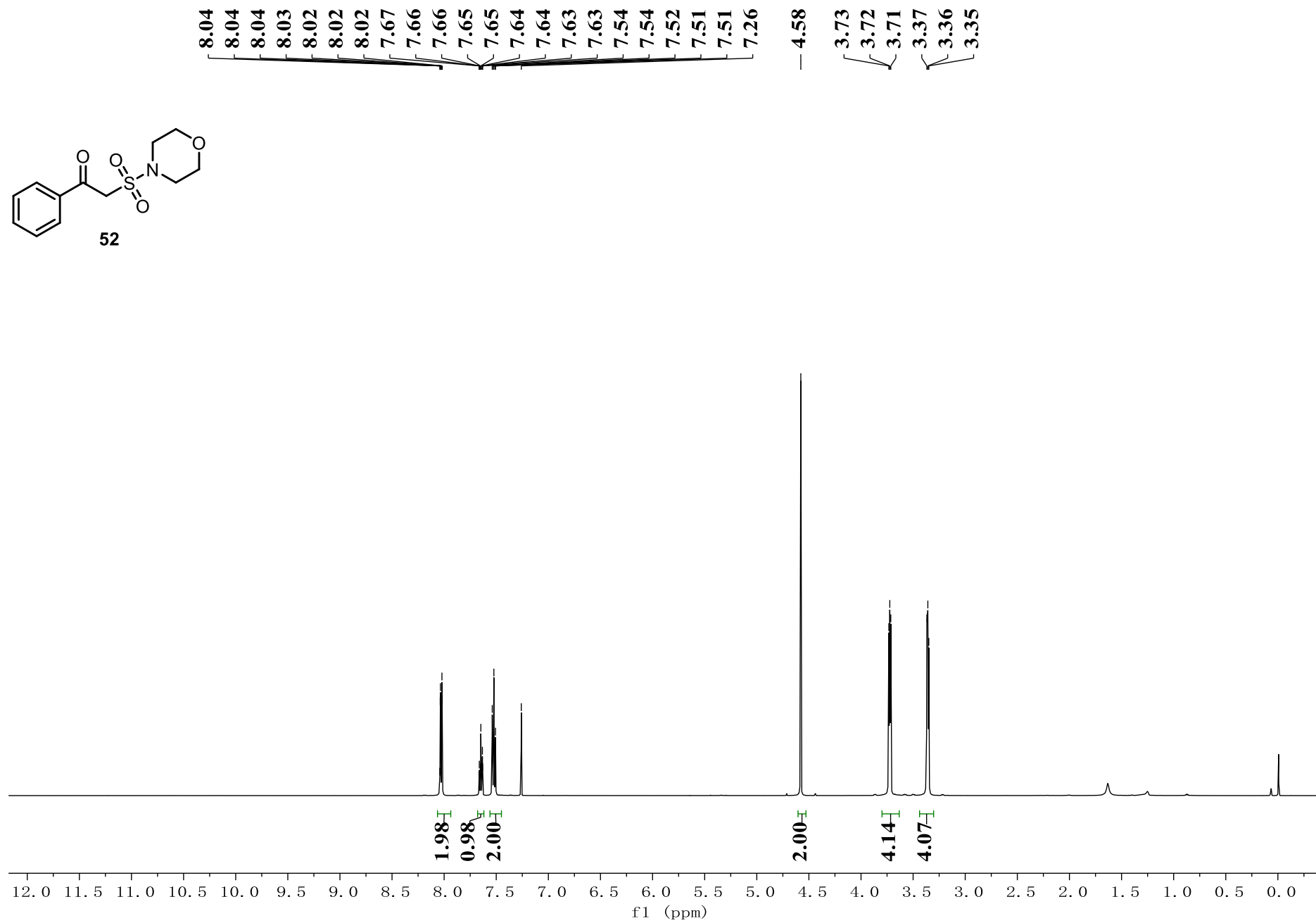
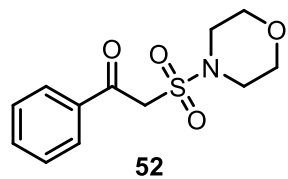
— 66.73

~ 46.42

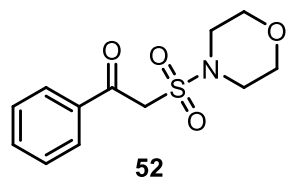
~ 43.31



¹H NMR (500 MHz, CDCl₃) for 52



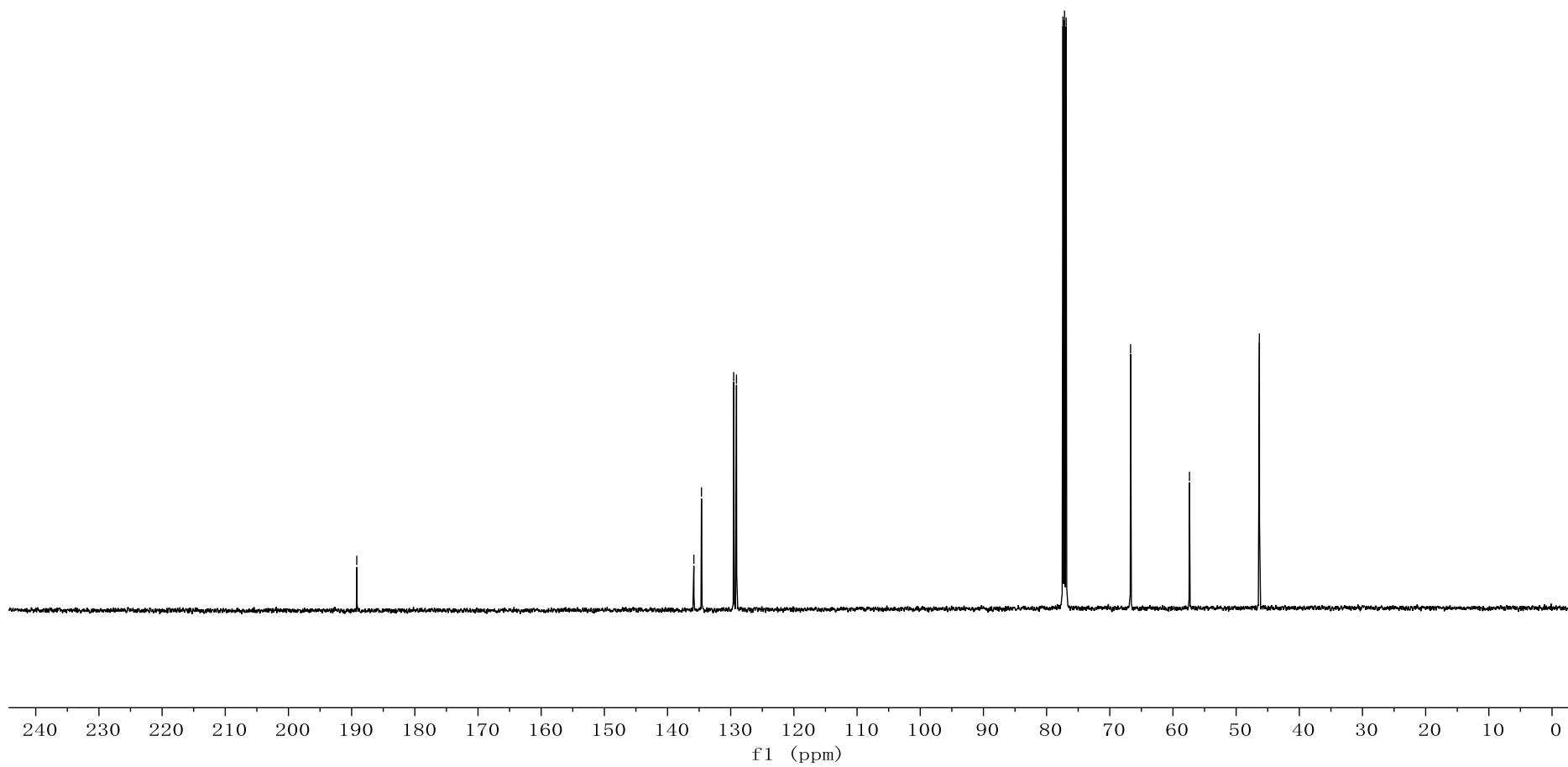
¹³C NMR (125 MHz, CDCl₃) for 52



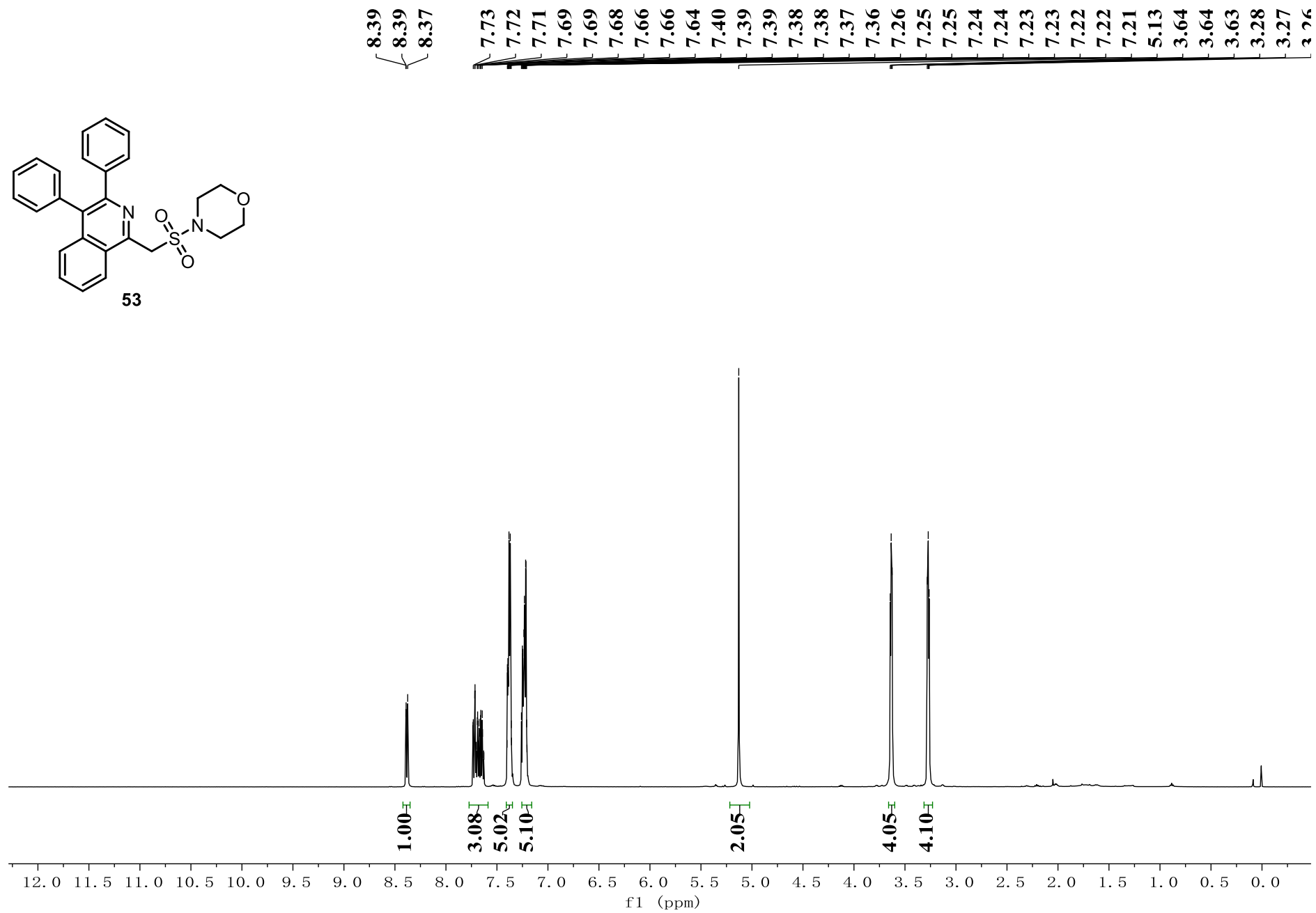
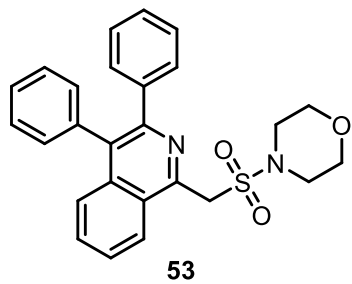
— 189.18

└ 135.83
└ 134.63
└ 129.53
└ 129.09

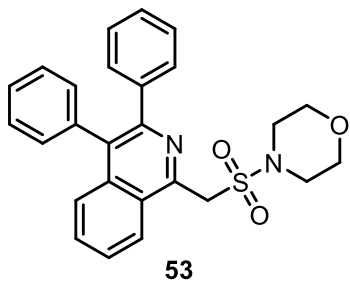
└ 77.41
└ 77.16
└ 76.91
— 66.70
— 57.39
— 46.32



¹H NMR (500 MHz, CDCl₃) for 53

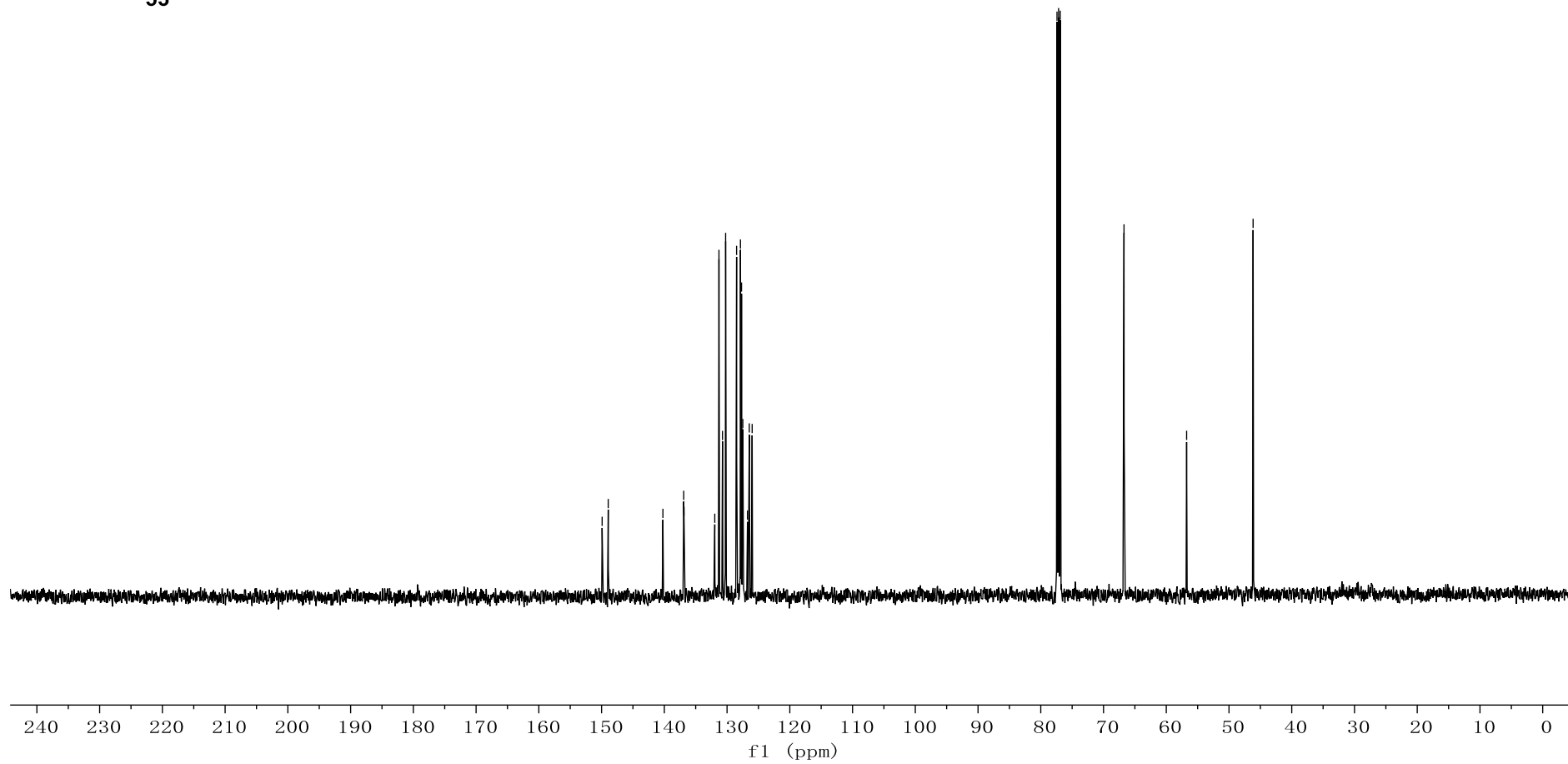


¹³C NMR (125 MHz, CDCl₃) for 53

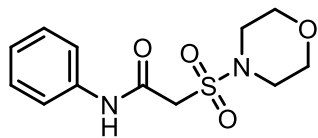


149.91
148.94
140.22
136.91
136.86
131.96
131.29
130.73
130.23
128.48
127.87
127.69
127.49
126.75
126.46
126.00

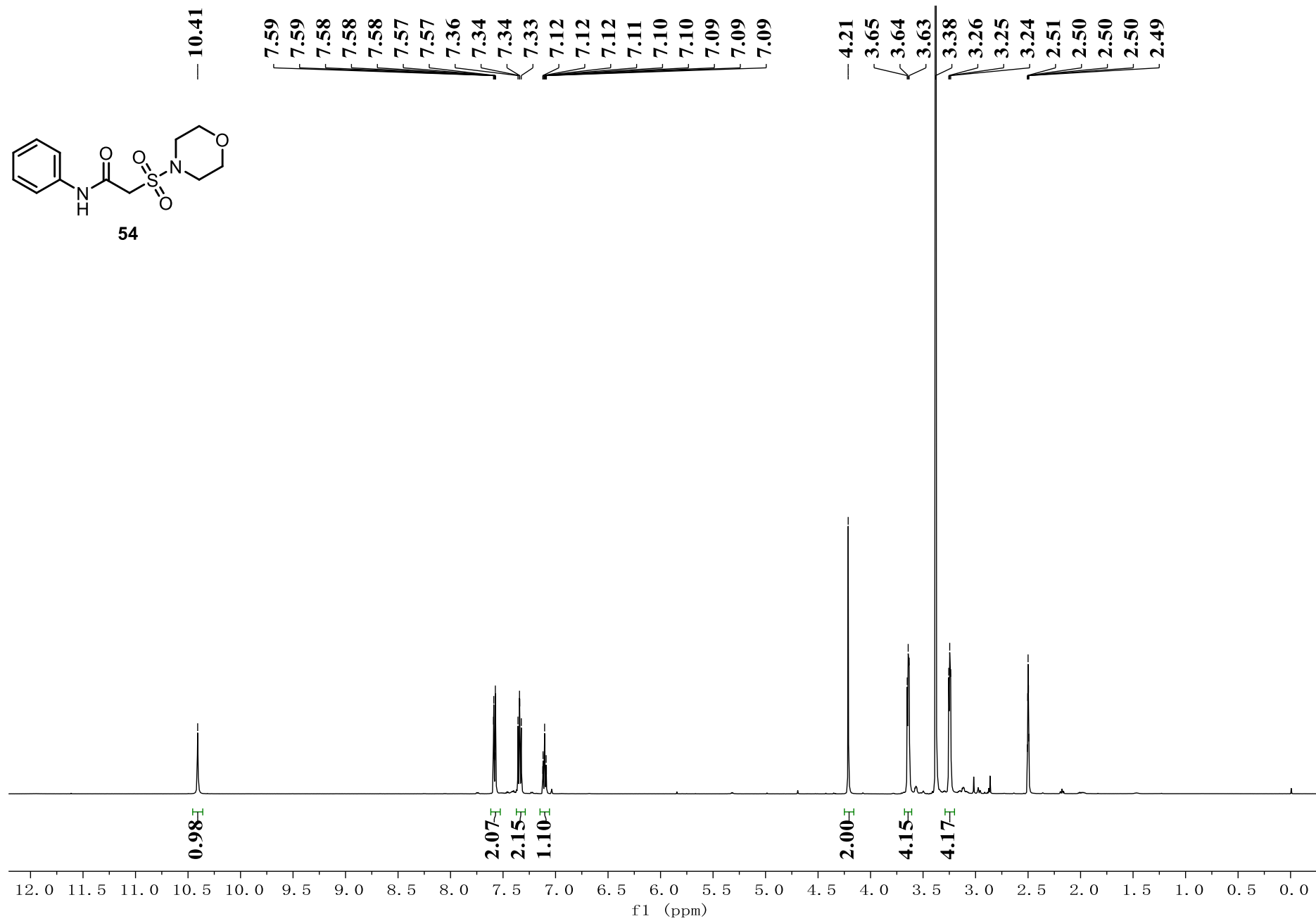
77.41
77.16
76.91
66.73
56.75
46.16



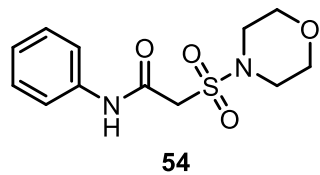
¹H NMR (500 MHz, DMSO-d₆) for 54



54

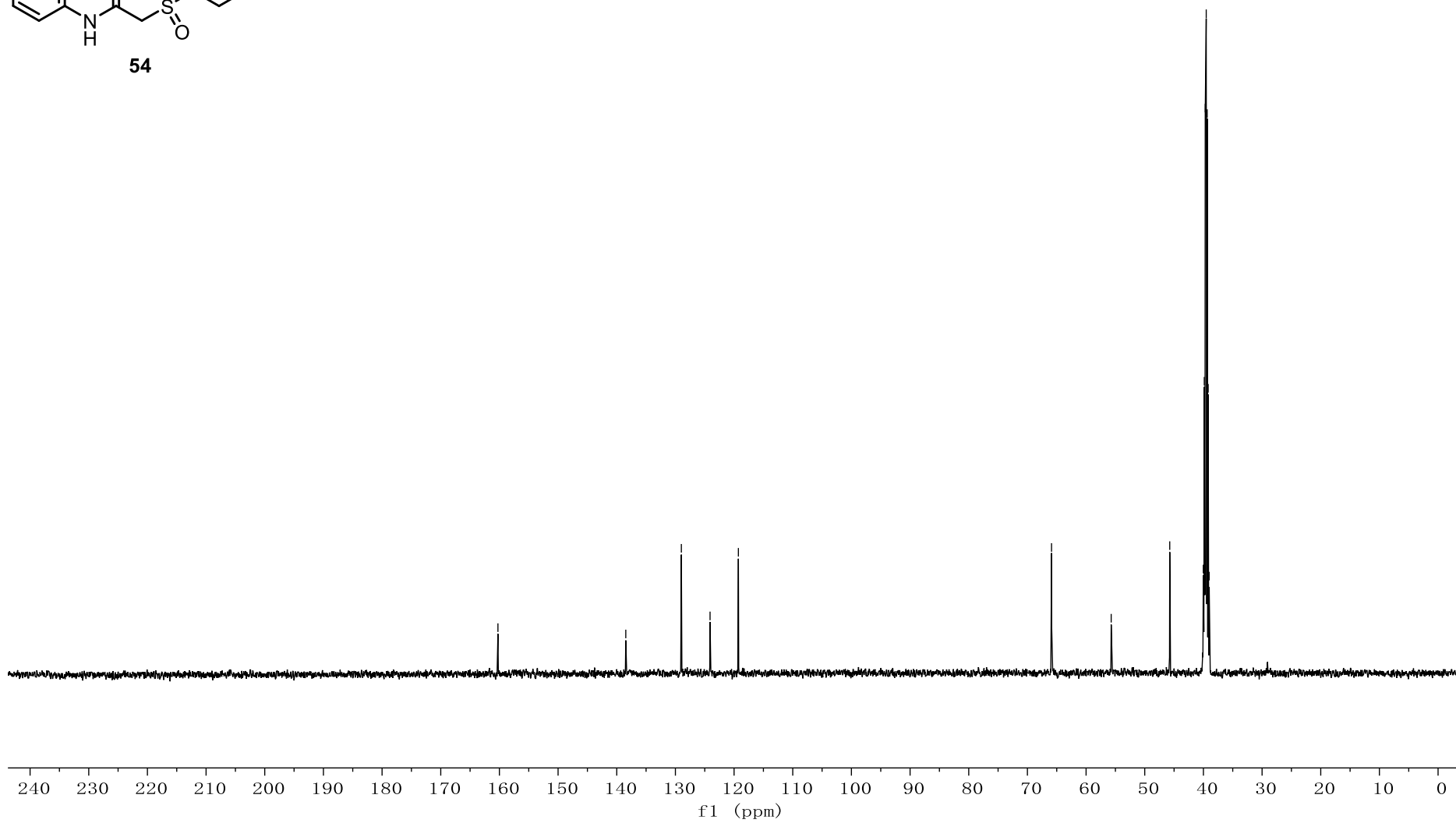


¹³C NMR (125 MHz, DMSO-*d*₆) for 54



— 160.25
— 65.89
— 55.72
— 45.74
— 40.02
— 39.85
— 39.69
— 39.52
— 39.35
— 39.19
— 39.02

138.46
128.99
124.10
119.26



10. Reference.

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