Electronic Supporting Information:

Photo-mediated controllable alkylation/difunctionalization of *N*-heteroaromatics *via* nucleohomolytic substitution of alkylboronic esters with *N*-nitrosamines

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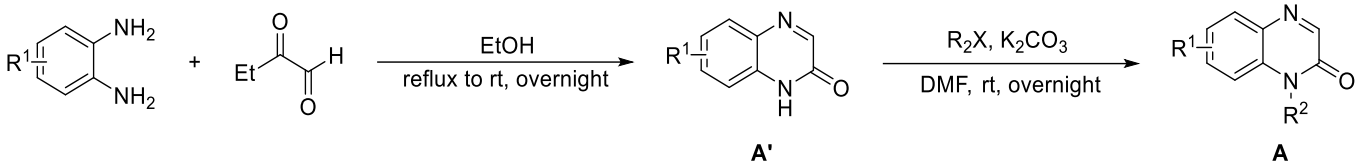
Contents

1. General information.

Commercial reagents were used without purification and specific 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 (δ = 7.26 ppm for ^{1}H, 77.16 ppm for ^{13}C), DMSO-d_6 (δ = 2.50 ppm for ^{1}H, 39.52 ppm for ^{13}C), CD_3OD (δ = 3.31 ppm for ^{1}H, 49.00 ppm for ^{13}C) as internal references. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, dq = doublet of quartets, tt = triplet of triplets, 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). 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.

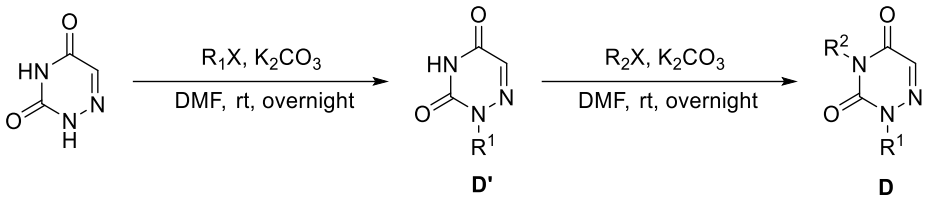
2.1. Synthesis of quinoxalinone substrates.[1]



To a solution of *o*-phenylenediamine (10 mmol, 1.0 equiv.) in EtOH (20 mL), ethyl glyoxalate (1.2 equiv.) was added. The mixture was heated at 85 °C for 1 h, then allowed to cool to room temperature and stirred overnight. After the reaction was complete, the product was collected by filtration. The crude product was washed by EtOH for 3 times and then dried to obtain **A'**.

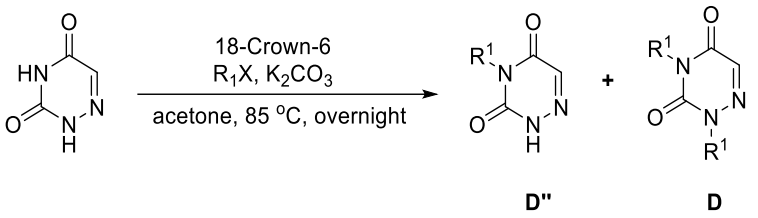
Compound **A'** (10 mmol, 1.0 equiv.) and K_2CO_3 (1.5 equiv.) were dissolved in DMF (20 mL) and placed in a round-bottom flask equipped with a magnetic stir bar, then organic halide (R_2X, 2.0 equiv.) was added. The reaction mixture was stirred at room temperature overnight. Upon completion of the reaction, it was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 10/1–3/1, v/v) to afford product **A**.

2.2. Synthesis of azauracil substrates.[2-3]



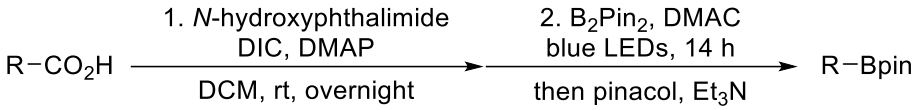
6-Azauracil (10 mmol, 1.0 equiv.) and K_2CO_3 (0.5 equiv.) were dissolved in DMF (20 mL) and placed in a round-bottom flask equipped with a magnetic stir bar, then organic halide (R_1X, 1.0 equiv.) was added. The reaction mixture was stirred at room temperature overnight. Upon completion of the reaction, it was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 10/1–1/1, v/v) to afford product **D'**.

D' (5 mmol, 1.0 equiv.) and K_2CO_3 (0.5 equiv.) were dissolved in DMF (10 mL) and placed in a round-bottom flask equipped with a magnetic stir bar, then organic halide (R_2X, 1.0 equiv.) was added. The reaction mixture was stirred at room temperature overnight. Upon completion of the reaction, it was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 10/1–3/1, v/v) to afford product **D**.

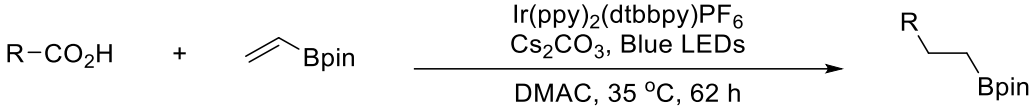


6-Azauracil (10 mmol, 1.0 equiv.), 18-crown-6-ether (0.1 equiv.) and K_2CO_3 (1.0 equiv.) were dissolved in acetone (30 mL) and placed in a round-bottom flask equipped with a magnetic stir bar, then organic halide (R_1X, 1.0 equiv.) was added. The reaction mixture was stirred at 85 °C overnight. Upon completion of the reaction, it was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 10/1–1/1, v/v) to afford product **D** and **D"**.

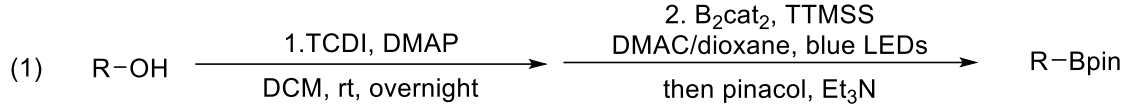
2.3. Synthesis of alkylboronic pinacol esters.

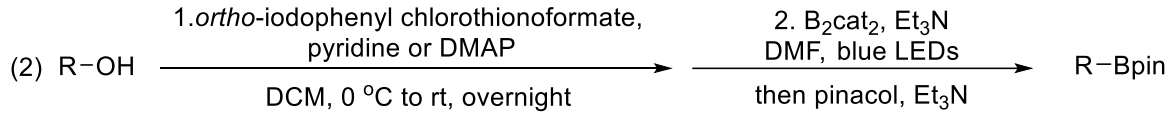


The alkylboronic pinacol esters from alkyl acids were prepared following the procedures reported in the literature.[4]

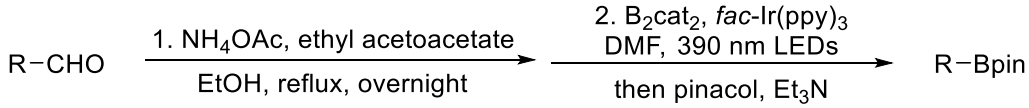


The alkylboronic pinacol esters from alkyl acids were prepared following the procedures reported in the literature.[5]

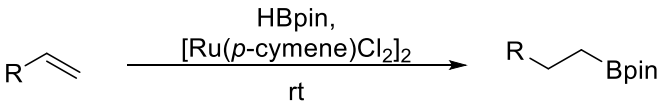




The alkylboronic pinacol esters from alkyl alcohols were prepared following the procedures reported in the literature.[6]



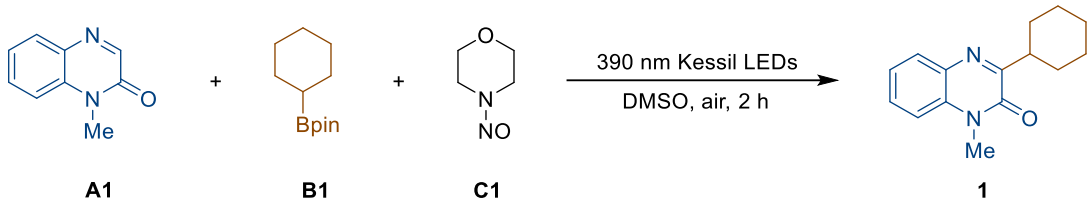
The alkylboronic pinacol esters from alkyl aldehydes were prepared following the procedures reported in the literature.[7]



The alkylboronic pinacol esters from alkyl alkenes were prepared following the procedures reported in the literature.[8]

3. General experimental procedure.

3.1. Procedure A:



A1 **B1** **C1** **1**

The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (16.0 mg, 0.1 mmol), **B1** (42.0 mg, 0.2 mmol, 2.0 equiv.) and **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.) were sequentially added in DMSO (1.0 mL). The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (100% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). Upon completion, the reaction mixture was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 8/1, v/v) to afford product **1** as a yellowish powder (19.4 mg, 80% yield). Unless otherwise noted, **15**–**29**, **48**–**58** were synthesized under the same reaction conditions.

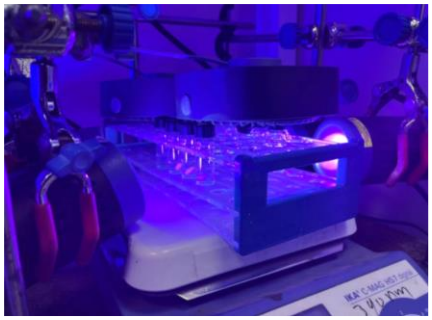


Figure S1. Representative pictures of reaction set-up.

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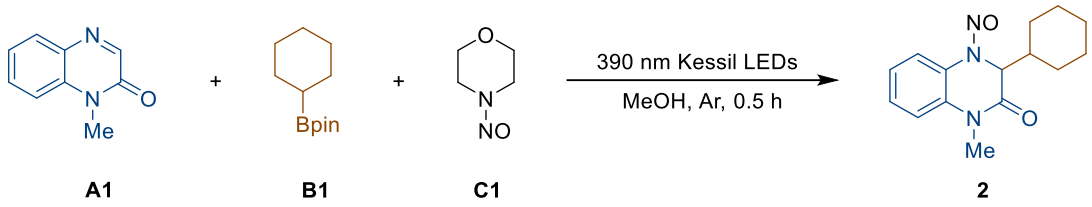
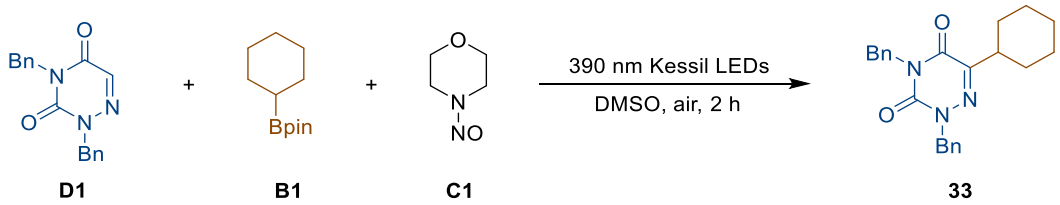
3.2. Procedure B:



A1 **B1** **C1** **2**

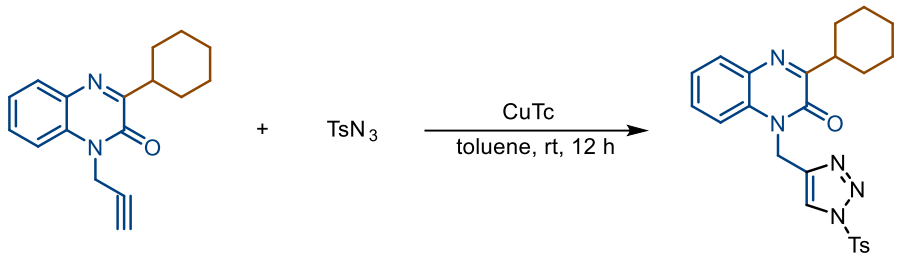
The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (32.0 mg, 0.2 mmol), **B1** (21.0 mg, 0.1 mmol) and **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.) were sequentially added in MeOH (1.0 mL) under argon atmosphere. The mixture was stirred at room temperature with cooling fans under irradiation of 390 nm LED lamps (25% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). Upon completion, the reaction mixture was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 8/1, v/v) to afford product **2** as a yellowish powder (22.9 mg, 84% yield). Unless otherwise noted, **2**–**14** were synthesized under the same reaction conditions. *Note: these compounds are prone to decomposition in light, air and acidic environments. To ensure a high yield, column chromatography should be done swiftly to prevent decomposition of the product on silica. The product should not be exposed to light, air and acidic conditions. These compounds showed slight decomposition in* $CDCl_3$.

3.3. Procedure C:



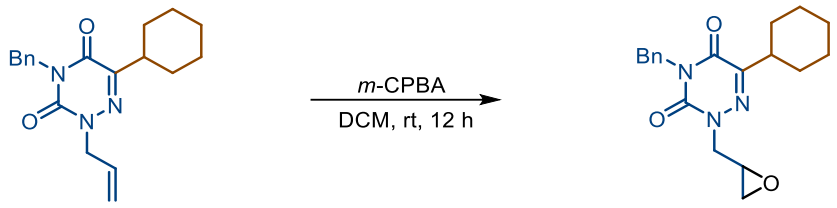
The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **D1** (29.5 mg, 0.1 mmol), **B1** (63.0 mg, 0.3 mmol, 3.0 equiv.) and **C1** (35.5 mg, 0.3 mmol, 3.0 equiv.) were sequentially added in DMSO (0.5 mL). The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (100% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). Upon completion, the reaction mixture was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 10/1, v/v) to afford product **33** as a yellowish powder (32.3 mg, 86% yield). Unless otherwise noted, **30**–**47** were synthesized under the same reaction conditions.

4. Synthetic applications.



18 **59**

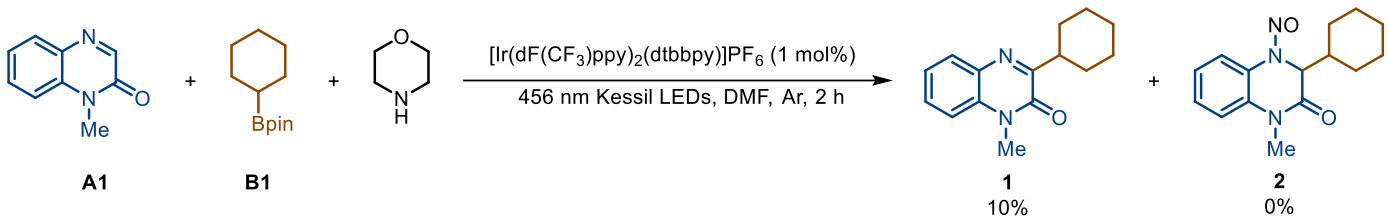
TsN_3 (1.3 equiv.) was slowly added to a solution of **18** (26.6 mg, 0.1 mmol) and CuTc (5 mol%) in toluene (1 mL) under argon atmosphere at room temperature. The reaction was stirred for 12 h. Upon completion, the reaction mixture was quenched by adding water. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 5/1, v/v) to afford product **59** as a white powder (39.4 mg, 85% yield).[9]



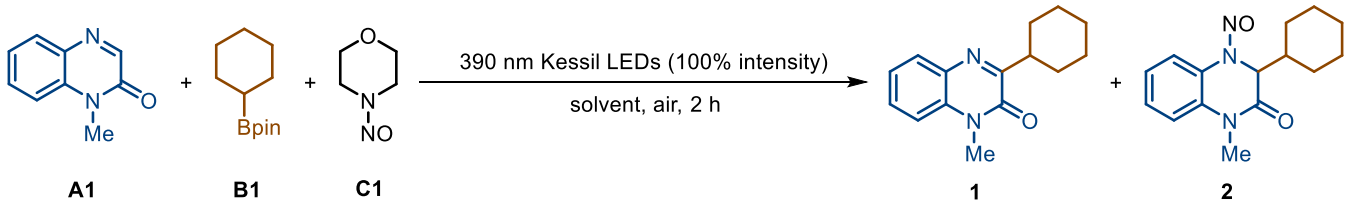
35 **60**

m-CPBA (2.5 equiv.) was slowly added to a solution of **35** (32.6 mg, 0.1 mmol) in DCM (1 mL) at room temperature. The reaction was stirred for 12 h. Upon completion, the reaction mixture was quenched by adding saturated $NaHCO_3$ solution. The mixture was then extracted with EtOAc, 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 purified by flash column chromatography using a PE/EtOAc system (PE/EtOAc, 8/1, v/v) to afford product **60** as a colorless oil (24.2 mg, 71% yield).[9]

5. Optimization of the reaction conditions.

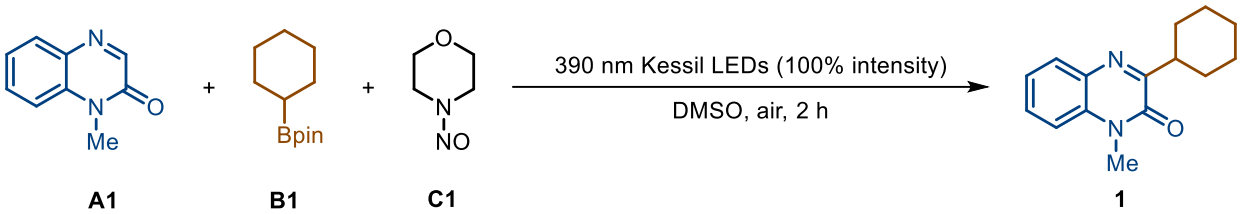


Reaction conditions: **A1** (0.1 mmol), **B1** (0.15 mmol), morpholine (0.15 mmol), [Ir] (1 mol%), 456 nm Kessil LEDs, DMF (1 mL) at room temperature under argon atmosphere with cooling fan for 2 h. The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.[10]



entry[a]	solvent	yield 1/2 (%)[b]
1	DCM	40/10
2	DCE	35/9
3	THF	38/12
4	MeCN	35/10
5	acetone	32/10
6	DMSO	69/0
7	Et_2O	29/8
8	EA	20/10
9	PhMe	24/9
10	MeOH	40/23
11	DMSO/H_2O = 1:1	8/0
12	DMF	8/0

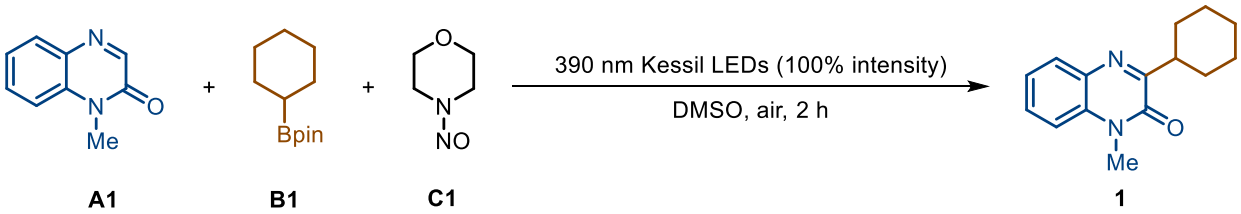
[a] Reaction conditions: **A1** (0.1 mmol), **B1** (0.15 mmol), **C1** (0.15 mmol), 390 nm Kessil LEDs, solvents (1 mL) at room temperature with cooling fan for 2 h. [b]The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



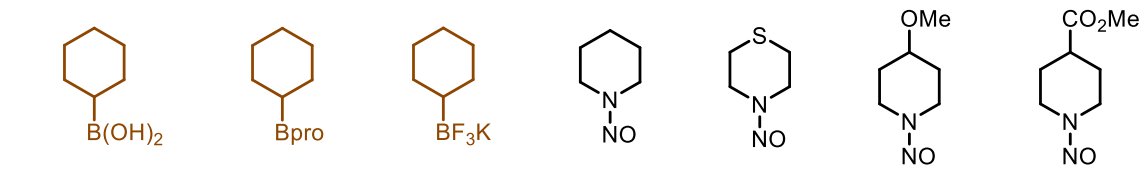
A1 **B1** **C1** **1**

entry[a]	variation	yield (%)[b]
1	none	82
2	0.5 mL	77
3	2 mL	79
4	427 nm	63
5	456 nm	46
6	**A1**/**B1**/**C1** = 2/1/2	81
7	without **C1**	ND

[a] Reaction conditions: **A1** (0.1 mmol), **B1** (0.2 mmol), **C1** (0.2 mmol), 390 nm Kessil LEDs, DMSO (1 mL) at room temperature with cooling fan for 2 h. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.

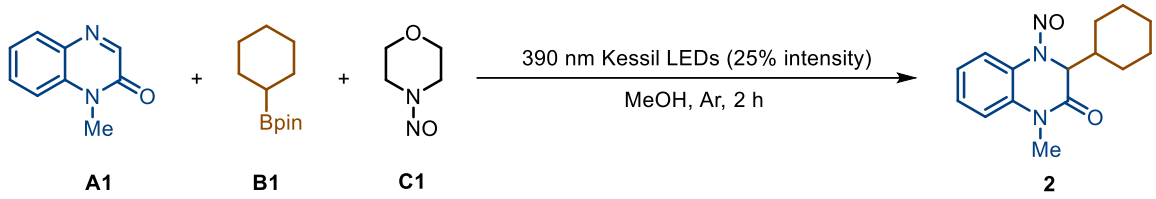


A1 **B1** **C1** **1**



B2	**B3**	**B4**	**C2**	**C3**	**C4**	**C5**
40%	79%	8%	76%	40%	73%	69%

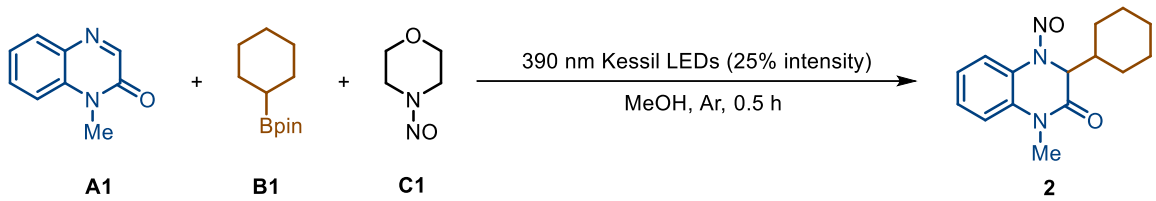
[a] Reaction conditions: **A1** (0.1 mmol), **B** (0.2 mmol), **C** (0.2 mmol), 390 nm Kessil LEDs, DMSO (1 mL) at room temperature with cooling fan for 2 h. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



A1 **B1** **C1** **2**

entry[a]	variation	yield (%)[b]
1	none	61
2	**A1**/**B1**/**C1** = 1/2/2	73
3	30 min, **A1**/**B1**/**C1** = 2/1/2	87

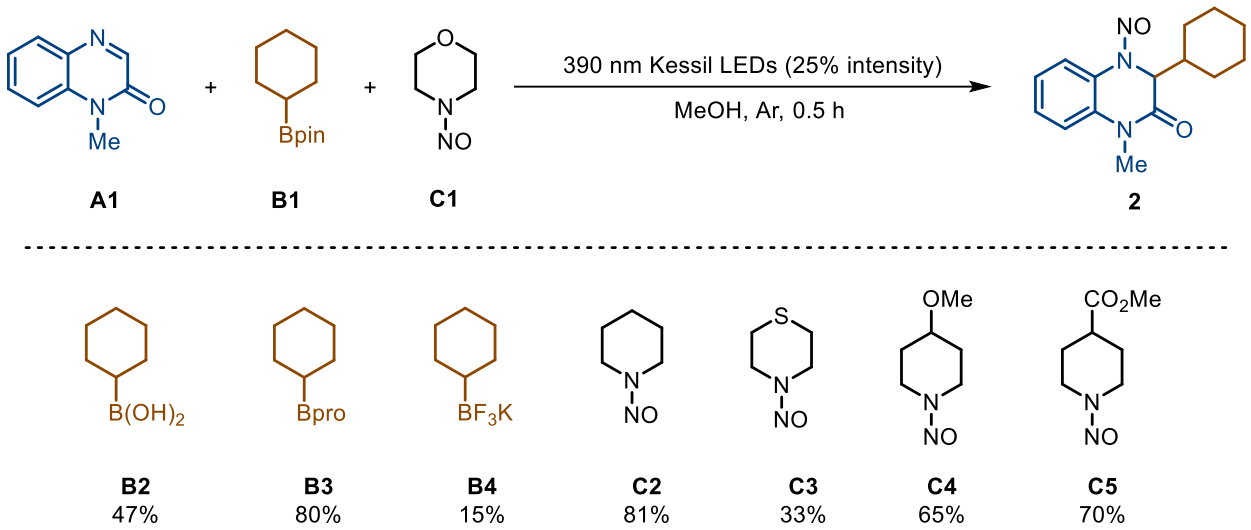
[a] Reaction conditions: **A1** (0.1 mmol), **B1** (0.15 mmol), **C1** (0.15 mmol), 390 nm Kessil LEDs, MeOH (1 mL) at room temperature with cooling fan. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



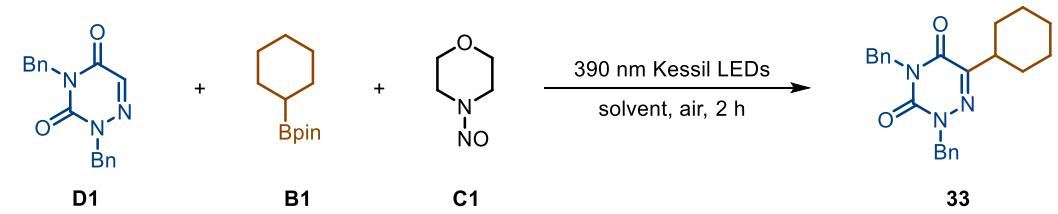
A1 **B1** **C1** **2**

entry[a]	variation	yield (%)[b]
1	none	87
2	EtOH	60
3	*t*-BuOH	50
4	*n*-BuOH	41
5	*i*-PrOH	45
6	*n*-PrOH	46
7	isopentanol	10
8	HFPI	7

[a] Reaction conditions: **A1** (0.2 mmol), **B1** (0.1 mmol), **C1** (0.2 mmol), 390 nm Kessil LEDs, solvent (1 mL) at room temperature with cooling fan under argon atmosphere for 0.5 h. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



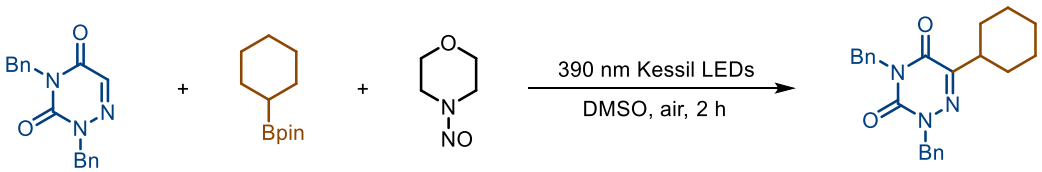
[a] Reaction conditions: **A1** (0.2 mmol), **B1** (0.1 mmol), **C1** (0.2 mmol), 390 nm Kessil LEDs, solvent (1 mL) at room temperature with cooling fan under argon atmosphere for 0.5 h. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



D1 **B1** **C1** **33**

entry[a]	solvent	yield (%)[b]
1	DCM	38
2	DCE	35
3	THF	33
4	MeCN	30
5	acetone	25
6	Et_2O	25
7	EA	29
8	DMC	30
9	PhMe	26
10	MeOH	28
11	DMSO	54
12	DMF	39
13	NMP	45
14	DMAC	36

[a] Reaction conditions: **D1** (0.1 mmol), **B1** (0.15 mmol), **C1** (0.15 mmol), 390 nm Kessil LEDs, solvent (1 mL) at room temperature with cooling fan for 2 h. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



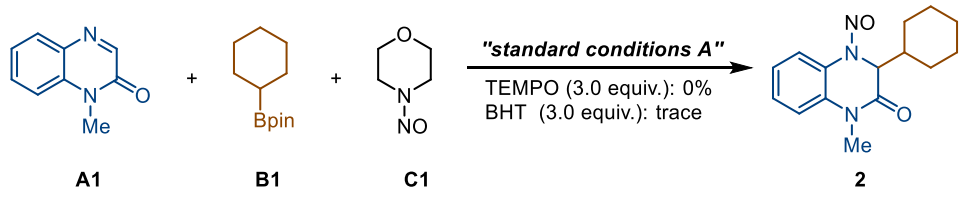
D1 **B1** **C1** **33**

entry[a]	variation	yield (%)[b]
1	none	54
2	**D1**/**B1**/**C1** = 1/2/2	65
3	**D1**/**B1**/**C1** = 1/3/3	70
4	0.5 mL	60
5	2 mL	38
6	0.5 mL DMSO + **D1**/**B1**/**C1** = 1/3/3	90
7	without **C1**	ND

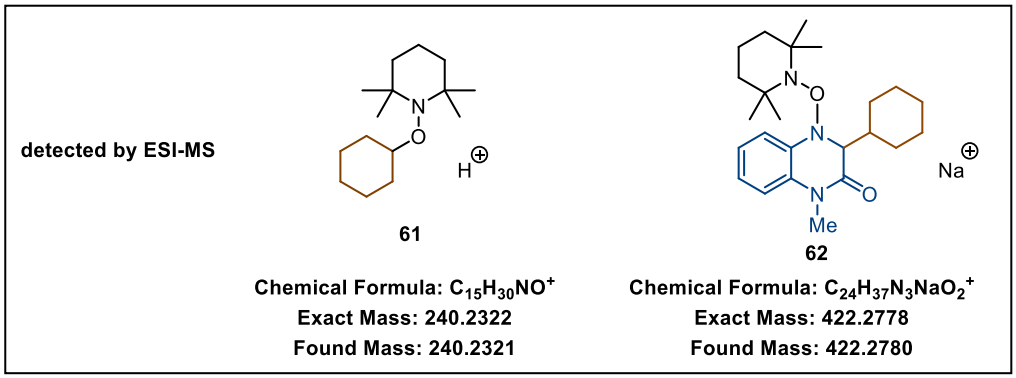
[a]Reaction conditions: **D1** (0.1 mmol), **B1** (0.15 mmol), **C1** (0.15 mmol), 390 nm Kessil LEDs, DMSO (1 mL) at room temperature with cooling fan for 2 h. [b] The yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.

6. Mechanistic investigations.

6.1. Radical trappling experiments.

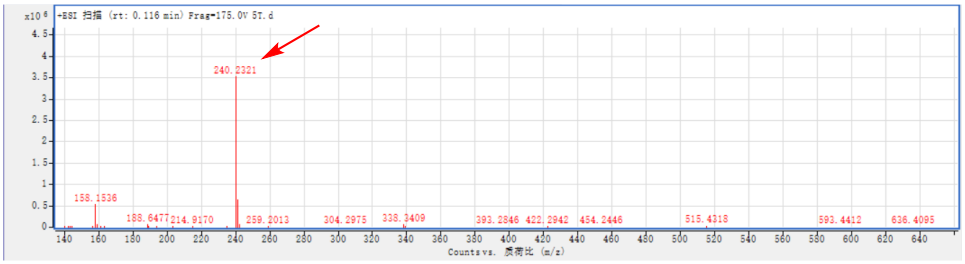


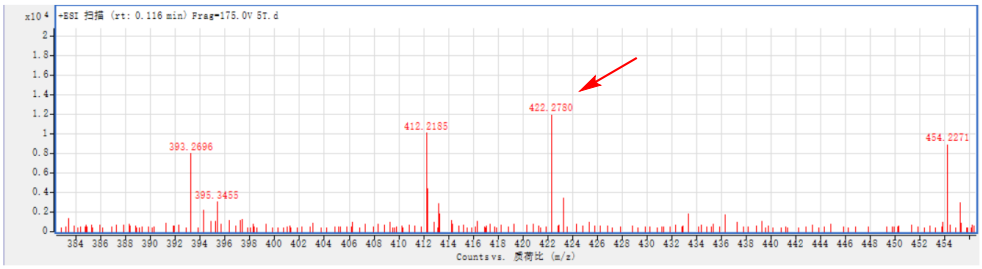
A1 B1 C1 2



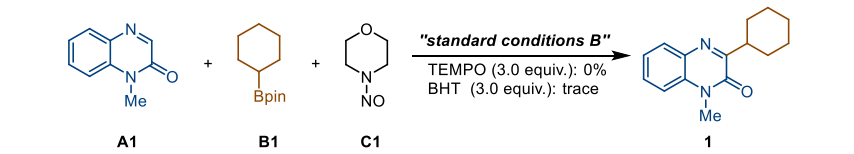
Chemical Formula: $C_{15}H_{30}NO^+$
Exact Mass: 240.2322
Found Mass: 240.2321

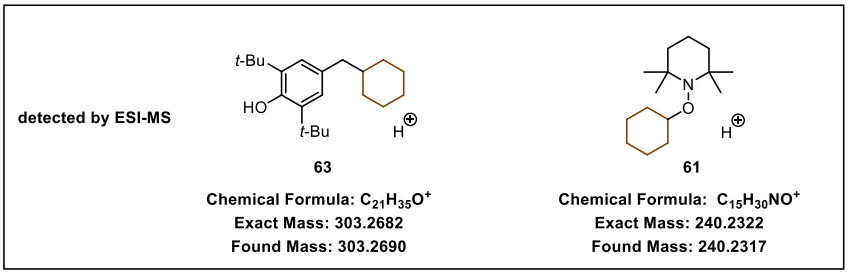
Chemical Formula: $C_{24}H_{37}N_3NaO_2^+$
Exact Mass: 422.2778
Found Mass: 422.2780

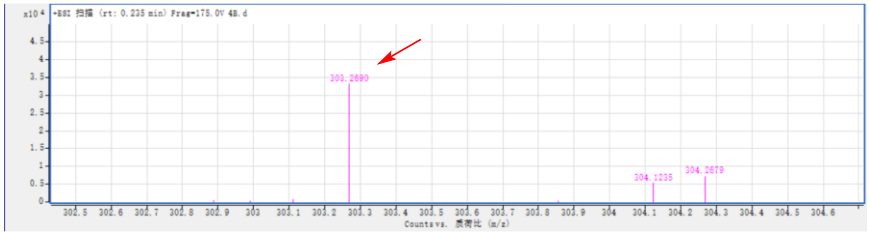


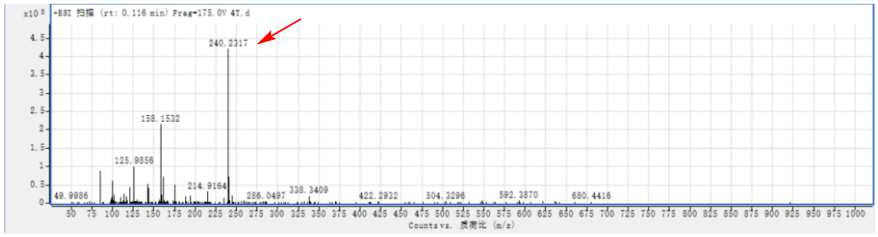


The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (32.0 mg, 0.1 mmol), **B1** (21.0 mg, 0.1 mmol), **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.), TEMPO (47.0 mg, 3.0 equiv.) or BHT (66.2 mg, 3.0 equiv.) were sequentially added in MeOH (1.0 mL) under argon atmosphere. The mixture was stirred at room temperature with cooling fans under irradiation of 390 nm LED lamps (25% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). Upon completion, the crude mixture was analyzed by ESI-MS.



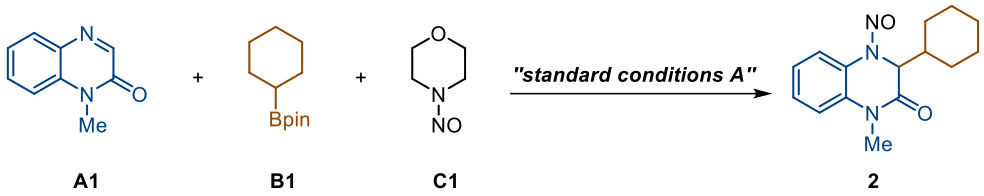






The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (16.0 mg, 0.1 mmol), **B1** (42.0 mg, 0.2 mmol, 2.0 equiv.), **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.), TEMPO (47.0 mg, 3.0 equiv.) or BHT (66.2 mg, 3.0 equiv.) were sequentially added in DMSO (1.0 mL). The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (100% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). Upon completion, the crude mixture was analyzed by ESI-MS.

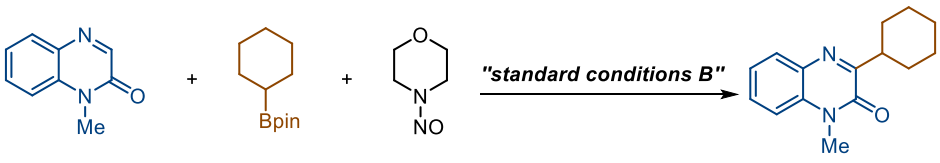
6.2. Studies with radical initiators.



A1 B1 C1 2

deviation from standard conditions		yield (%)
adding radical intiator, 70 °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** (32.0 mg, 0.2 mmol), **B1** (21.0 mg, 0.1 mmol), **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.) were dissolved in MeOH (1 mL) under argon atmosphere. Radical initiator was added and the tube was sealed. The reaction mixture was stirred at 70 °C for 0.5 h (oil bath as the heat source). Upon completion, the yield was determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.



A1 B1 C1 1

deviation from standard conditions		yield (%)
adding radical intiator, 100 °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** (16.0 mg, 0.1 mmol), **B1** (42.0 mg, 0.2 mmol, 2.0 equiv.), **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.) were dissolved in DMSO (1 mL) under argon atmosphere. Radical initiator was added and the tube was sealed. The reaction mixture was stirred at 100 °C for 2 h (oil bath as the heat source). Upon completion, the yield was determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.

6.3. Ultraviolet-visible absorption experiments.

UV-vis absorption spectra of **A1**, **B1**, **C1**, **B1+C1**, **A1+C1** and **A1+B1+C1** in DMSO 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.

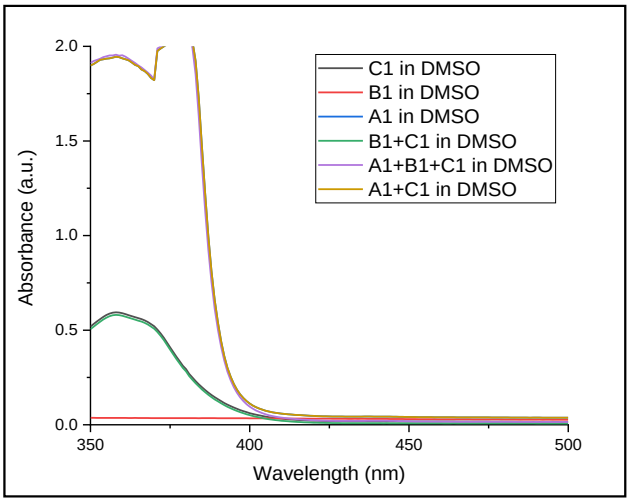


Figure S2. UV-vis absorption spectroscopy.

UV-vis absorption spectra of **A1**, **B1**, **C1**, **B1+C1**, **A1+C1** and **A1+B1+C1** in MeOH 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.

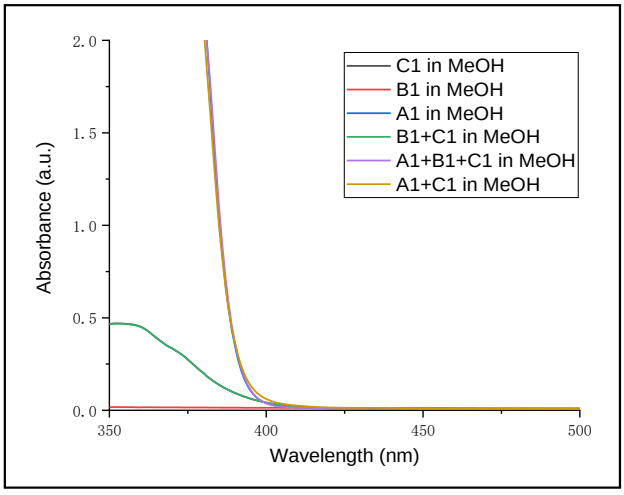


Figure S3. UV-vis absorption spectroscopy.

6.4. ^{11}B NMR experiments.

The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **A1** (16.0 mg, 0.1 mmol), **B1** (42.0 mg, 0.2 mmol, 2.0 equiv.) and **C1** (23.5 mg, 0.2 mmol, 2.0 equiv.) were sequentially added in DMSO-d_6 (1.0 mL) or CD_3OD (1.0 mL). The reaction mixture was stirred for 2 h and then transferred to an ^{11}B NMR tube for analysis.

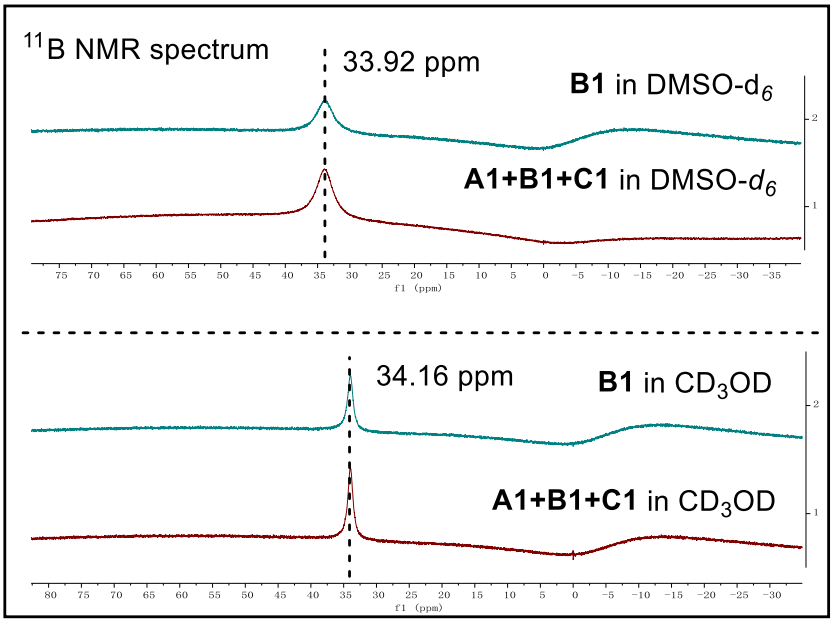
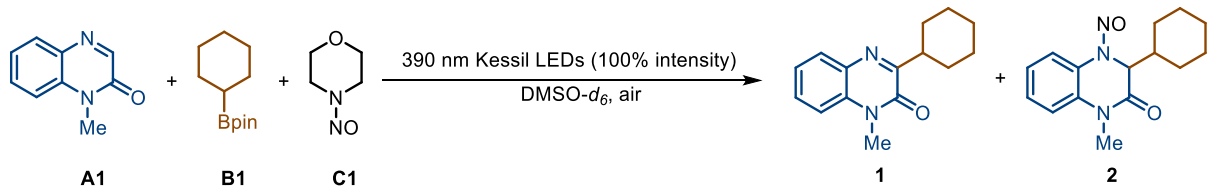
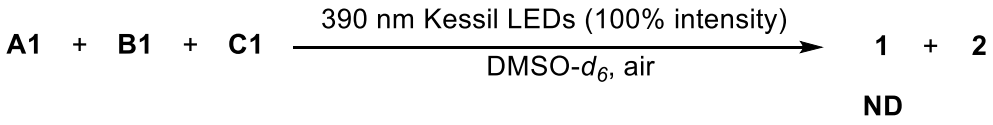


Figure S4. ^{11}B NMR experiments.

6.5. On-off experiments.



The reaction was conducted in an oven-dried 10-mL vial equipped with a stir bar. **A1** (48.0 mg, 0.3 mmol), **B1** (126.0 mg, 2.0 equiv.), **C1** (70.0 mg, 2.0 equiv.) and internal standard (1,3,5-trimethoxybenzene, 0.75 mmol, 12.6 mg) were sequentially added in DMSO-d_6 (3.0 mL). The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (100% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). An aliquot of the reaction mixture then taken at the indicated times and the yield was determined by ^{1}H NMR.



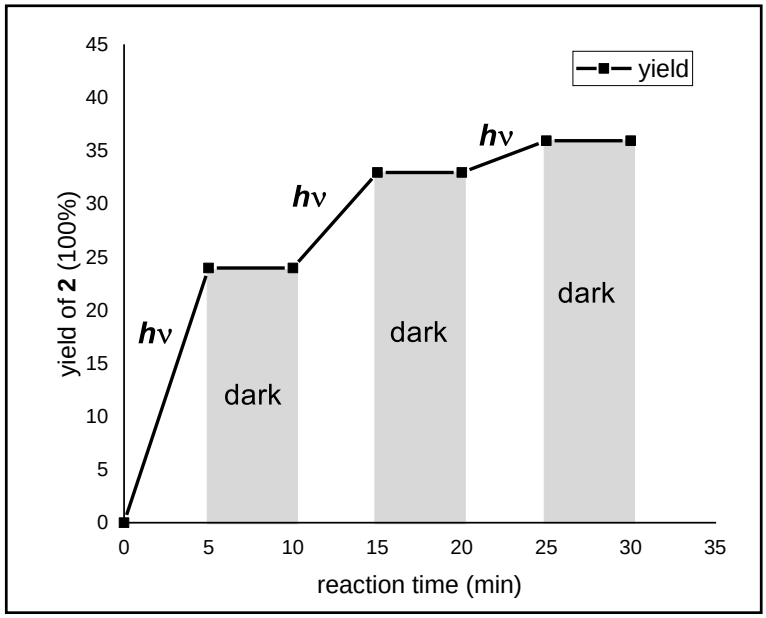
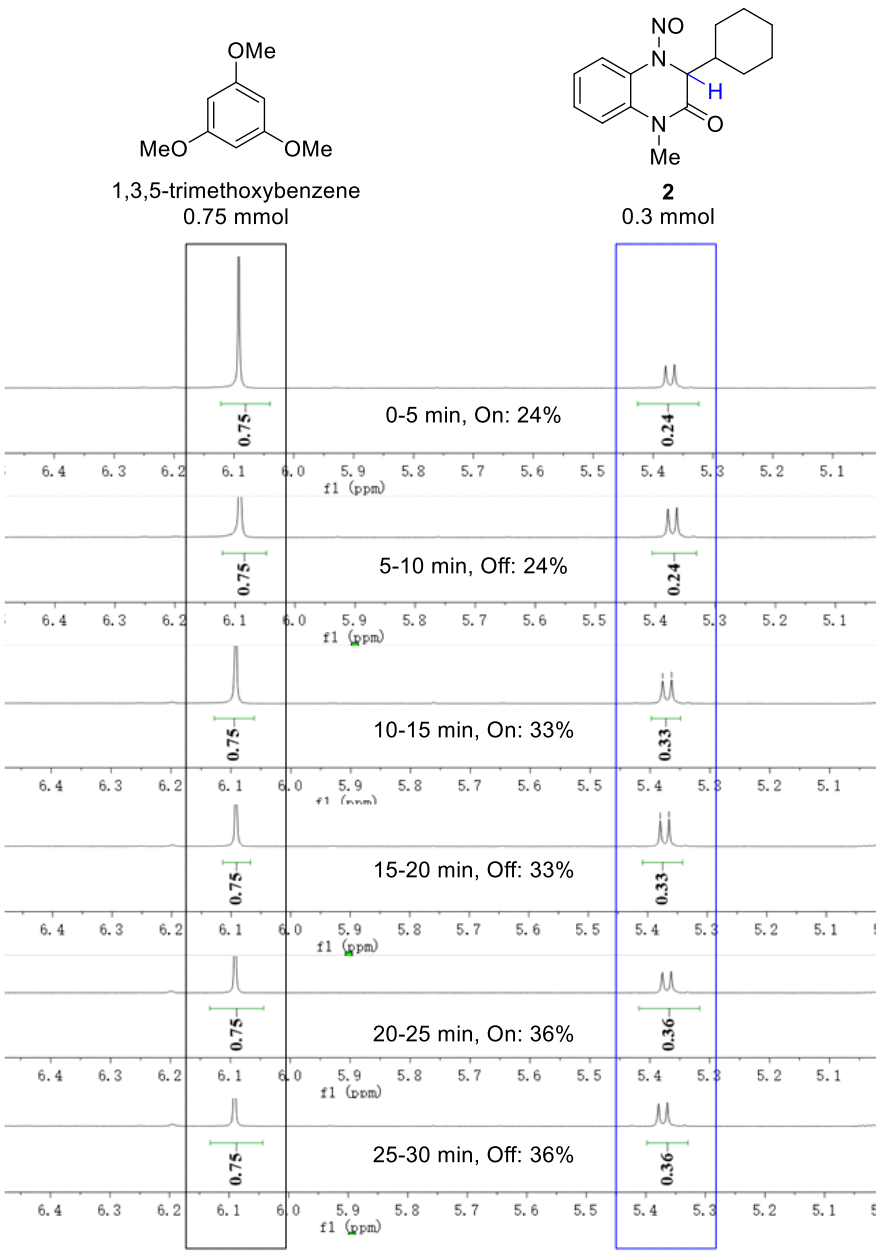


Figure S5. On-off Experiments.


OMe
MeO
OMe
1,3,5-trimethoxybenzene
0.75 mmol
NO
N
H
N
O
Me
2
0.3 mmol
0.75
0.24
0-5 min, On: 24%
6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1
f1 (ppm)
0.75
0.24
5-10 min, Off: 24%
6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1
f1 (ppm)
0.75
0.33
10-15 min, On: 33%
6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1
0.75
0.33
15-20 min, Off: 33%
6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1
f1 (ppm)
0.75
0.36
20-25 min, On: 36%
6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1
f1 (ppm)
0.75
0.36
25-30 min, Off: 36%
6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1
f1 (ppm)

6.6. 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).[11-13] A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate trihydrate (1.5 mmol) in 0.20 M aqueous H_2SO_4 (10.0 mL). A buffered solution of 1,10-phenanthroline (0.15 M) was prepared by dissolving NaOAc (15.0 mmol) and 1,10-phenanthroline (3.0 mmol) in 0.20 M aqueous H_2SO_4 (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 μL) 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:

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

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 $L \cdot mol^{-1} \cdot cm^{-1}$)[11].

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 λ = 405 nm and λ = 385 nm),[11-12] t is the irradiation time (300 s), and f is the fraction of light absorbed at λ = 390 nm by the ferrioxalate actinometer. This value was calculated using the following equation where A (390 nm) is the absorption of the ferrioxalate solution at 390 nm.

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

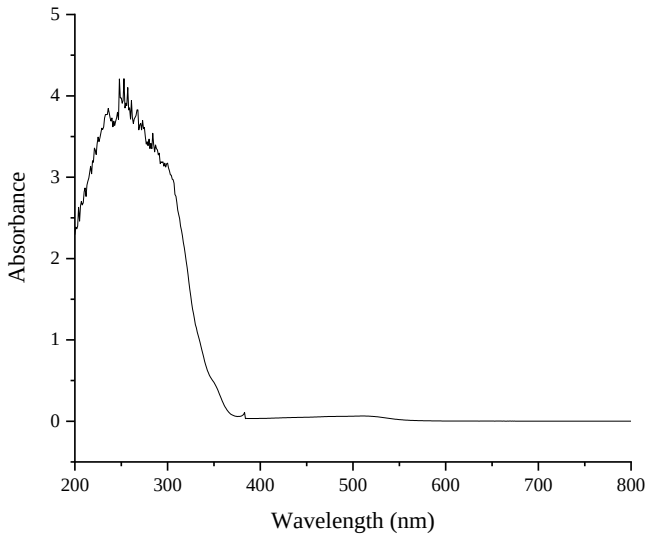


Figure S6. Absorbance of the ferrioxalate actinometer solution

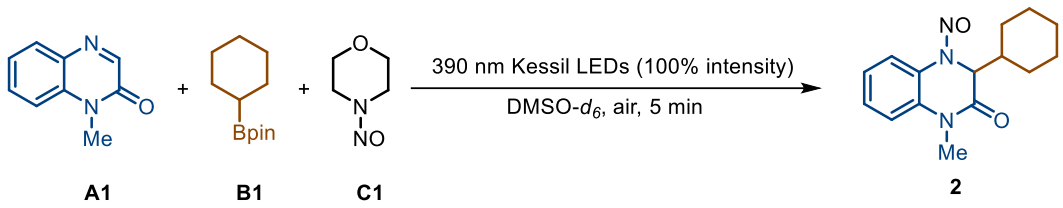
The average photon flux was thus calculated to be 2.06×10^{-7} einstein s^{-1}.

Sample calculation:

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

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

Determination of quantum yield:



The reaction was conducted in an oven-dried 10-mL vial equipped with a stir bar. **A1** (48.0 mg, 0.3 mmol), **B1** (126.0 mg, 2.0 equiv.), **C1** (70.0 mg, 2.0 equiv.) and internal standard (1,3,5-trimethoxybenzene, 0.75 mmol, 12.6 mg) were sequentially added in DMSO-d_6 (3.0 mL). The mixture was stirred at room temperature with cooling fans under irradiation of 40 W 390 nm LED lamps (100% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away). The reaction mixture then taken at the 5 min and the yield was determined by ^{1}H NMR.

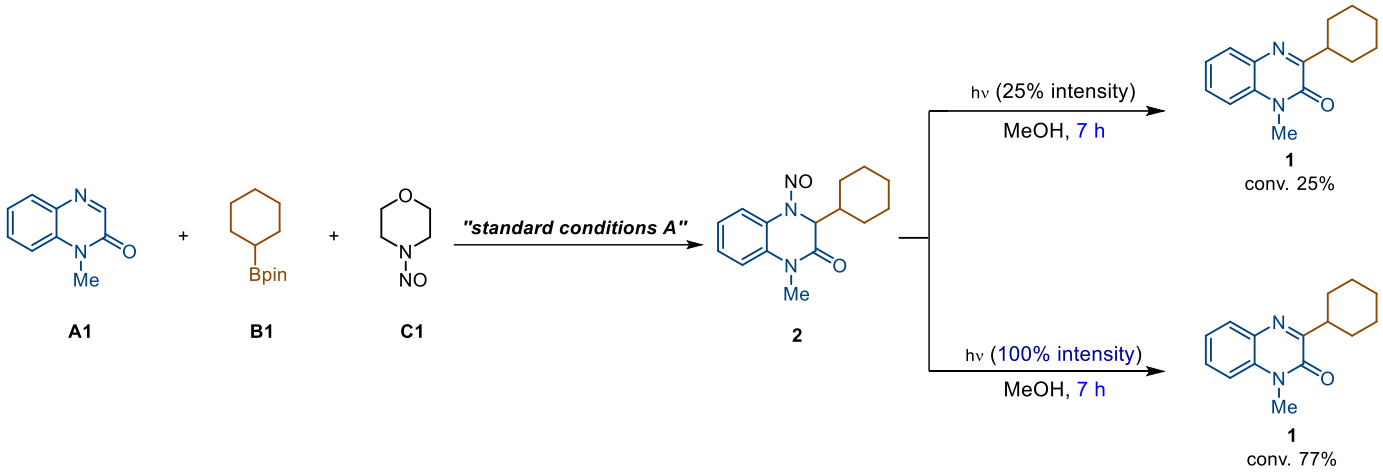
The quantum yield was determined using eq 4.

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

Sample quantum yield calculation:

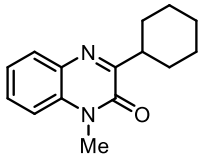
$$\phi = \frac{0.3 \text{ x } 10^{-3}\text{mol} \times 0.24}{2.06 \times 10^{-7} \times 300\text{s} \times 0.89731} = 1.30$$

6.7. Conversion of 2 to 1.



The reaction was conducted in an oven-dried 3-mL vial equipped with a stir bar. **2** (27.3 mg, 0.1 mmol) was added in MeOH (1.0 mL) under argon atmosphere. Continue to stir the mixture at room temperature with cooling fans under irradiation of 390 nm LED lamps (25% intensity or 100% intensity; Kessil KSPR160–390 LED Grow Light; 5-6 cm away) for 7 h. Upon completion, the conversion rate was determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.

7. Characterization data of new compounds.



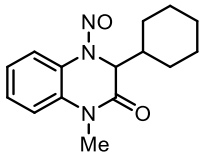
1

3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **1** (19.4 mg, 80% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 1H), 7.51–7.47 (m, 1H), 7.32–7.25 (m, 2H), 3.68 (s, 3H), 3.33 (tt, *J* = 11.6, 3.3 Hz, 1H), 1.97–1.83 (m, 4H), 1.78–1.73 (m, 1H), 1.60–1.41 (m, 4H), 1.34–1.25 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.4, 154.6, 133.0, 132.9, 129.8, 129.5, 123.5, 113.6, 40.9, 30.6, 29.2, 26.4, 26.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{19}N_2O^+$ 243.1492, found 243.1490.



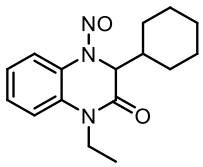
2

3-cyclohexyl-1-methyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **2** (22.9 mg, 84% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 1H), 7.43–7.40 (m, 1H), 7.25–7.21 (m, 1H), 7.16–7.14 (m, 1H), 5.57 (d, *J* = 7.4 Hz, 1H), 3.42 (s, 3H), 1.70–1.50 (m, 5H), 1.39–1.35 (m, 1H), 1.12–0.85 (m, 5H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.0, 130.5, 128.4, 127.4, 124.2, 118.5, 115.5, 58.6, 41.1, 29.6, 29.5, 29.1, 25.9, 25.8, 25.7 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{20}N_3O_2^+$ 274.1550, found 274.1555.



3

3-cyclohexyl-1-ethyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **3** (23.5 mg, 82% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 1H), 7.42–7.39 (m, 1H), 7.23–7.16 (m, 2H), 5.54 (d, *J* = 7.4 Hz, 1H), 4.13 (dq, *J* = 14.4, 7.2 Hz, 1H), 3.92 (dq, *J* = 14.4, 7.2 Hz, 1H), 1.68–1.52 (m, 5H), 1.39–1.35 (m, 1H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.10–0.86 (m, 5H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.4, 129.4, 128.4, 127.5, 124.1, 118.8, 115.2, 58.5, 40.9, 37.6, 29.4, 29.0, 25.8, 25.8, 25.7, 12.8 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{22}N_3O_2^+$ 288.1707, found 288.1710.

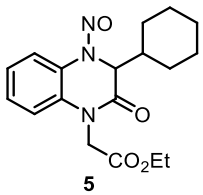
4

1-benzyl-3-cyclohexyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **4** (29.3 mg, 84% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.86–7.84 (m, 1H), 7.38–7.32 (m, 2H), 7.31–7.20 (m, 5H), 7.11–7.09 (m, 1H), 5.70 (d, *J* = 8.4 Hz, 1H), 5.49 (d, *J* = 16.2 Hz, 1H), 4.95 (d, *J* = 16.2 Hz, 1H), 1.83–1.58 (m, 5H), 1.47–1.42 (m, 1H), 1.26–0.94 (m, 5H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.0, 136.1, 129.9, 129.1, 128.3, 127.7, 127.3, 126.5, 124.4, 118.7, 116.3, 58.6, 46.3, 40.6, 29.6, 29.1, 25.8, 25.7, 25.6 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{24}N_3O_2^+$ 350.1863, found 350.1861.



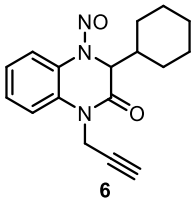
5

ethyl 2-(3-cyclohexyl-4-nitroso-2-oxo-3,4-dihydroquinoxalin-1(2*H*)-yl)acetate: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **5** (27.6 mg, 80% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.82 (m, 1H), 7.39–7.36 (m, 1H), 7.26–7.22 (m, 1H), 6.90–6.88 (m, 1H), 5.62 (d, J = 7.4 Hz, 1H), 5.11 (d, J = 17.6 Hz, 1H), 4.27–4.22 (m, 3H), 1.73–1.45 (m, 6H), 1.27 (t, J = 7.2 Hz, 3H), 1.13–1.00 (m, 4H), 0.92–0.85 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.9, 165.3, 129.6, 128.5, 127.4, 124.6, 118.9, 115.0, 62.1, 58.4, 44.2, 40.8, 29.3, 28.9, 25.8, 25.8, 25.9, 14.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{24}N_3O_4^+$ 346.1761, found 346.1755.



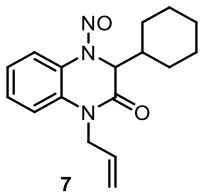
6

3-cyclohexyl-4-nitroso-1-(prop-2-yn-1-yl)-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **6** (22.9 mg, 77% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.85–7.83 (m, 1H), 7.46–7.43 (m, 1H), 7.37–7.35 (m, 1H), 7.28–7.25 (m, 1H), 5.60 (d, J = 7.2 Hz, 1H), 5.01 (dd, J = 17.7, 2.5 Hz, 1H), 4.46 (dd, J = 17.7, 2.5 Hz, 1H), 2.28 (t, J = 2.5 Hz, 1H), 1.69–1.52 (m, 5H), 1.42–1.28 (m, 1H), 1.12–0.98 (m, 4H), 0.93–0.85 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.5, 128.8 128.4, 127.5, 124.7, 118.7, 115.9, 77.6, 72.8, 58.3, 41.2, 31.9, 29.4, 29.0, 25.8, 25.7, 25.7 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{20}N_3O_2^+$ 298.1550, found 298.1557.



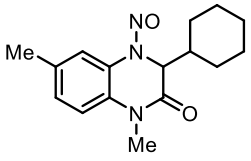
7

1-allyl-3-cyclohexyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **7** (23.6 mg, 79% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82–7.80 (m, 1H), 7.39–7.35 (m, 1H), 7.23–7.20 (m, 1H), 7.15–7.14 (m, 1H), 5.92–5.85 (m, 1H), 5.58 (d, J = 8.0 Hz, 1H), 5.27–5.17 (m, 2H), 4.89–4.84 (m, 1H), 4.32–4.27 (m, 1H), 1.71–1.52 (m, 5H), 1.40–1.36 (m, 1H), 1.13–0.87 (m, 5H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.6, 131.8, 129.8, 128.3, 127.3, 124.3, 118.7, 117.3, 116.1, 58.5, 45.1, 40.7, 29.5, 29.1, 25.8, 25.7 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{22}N_3O_2^+$ 300.1707, found 300.1704.



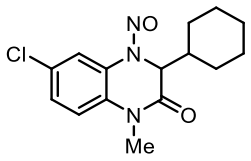
8

3-cyclohexyl-1,6-dimethyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **8** (21.5 mg, 75% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.69–7.63 (m, 1H), 7.22–6.95 (m, 2H), 7.23–7.20 (m, 1H), 5.57–5.54 (m, 1H), 3.40 and 3.39 (s, 3H), 2.44 and 2.42 (s, 3H), 1.70–1.52 (m, 5H), 1.38–1.35 (m, 1H), 1.07–1.00 (m, 4H), 0.94–0.85 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.2, 164.9, 138.6, 134.2, 130.6, 130.4, 129.0, 128.1, 127.2, 125.1, 124.9, 118.9, 118.4, 116.1, 115.3, 58.7, 41.1, 30.7, 29.6, 29.5, 29.1, 26.5, 25.9, 25.8, 25.7, 21.7, 20.9 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{22}N_3O_2^+$ 288.1707, found 288.1715.



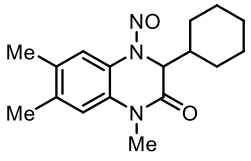
9

6-chloro-3-cyclohexyl-1-methyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **9** (26.1 mg, 85% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.82 (m, 1H), 7.38–7.35 (m, 1H), 7.08–7.06 (m, 1H), 5.51 (d, J = 7.2 Hz, 1H), 3.40 (s, 3H), 1.68–1.53(m, 5H), 1.37–1.33 (m, 1H), 1.10–0.98 (m, 4H), 0.98–0.86 (m, 4H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.6, 129.6, 129.1, 128.3, 128.0, 118.3, 116.5, 58.5, 41.3, 29.6, 29.5, 29.1, 25.9, 25.7 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{19}ClN_3O_2^+$ 308.1160, found 308.1161.



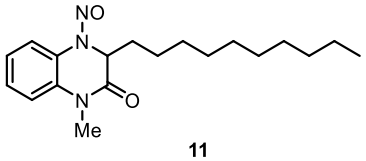
10

3-cyclohexyl-1,6,7-trimethyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **10** (21.7 mg, 72% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.57 (s, 1H), 6.91 (s, 1H), 5.55 (d, J = 7.2 Hz, 1H), 3.39 (s, 3H), 2.34 (s, 3H), 2.31 (s, 3H), 1.69–1.52 (m, 5H), 1.39–1.35 (m, 1H), 1.07–0.98 (m, 4H), 0.93–0.88 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.0, 137.1, 132.8, 128.2, 125.0, 119.4, 116.6, 58.8, 41.1, 29.6, 29.4, 29.1, 25.9, 25.8, 25.7, 20.1, 19.4 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{24}N_3O_2^+$ 302.1863, found 302.1860.



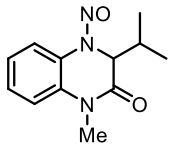
11

3-decyl-1-methyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 10/1, v/v). The product **11** (21.2 mg, 64% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.86–7.84 (m, 1H), 7.44–7.40 (m, 1H), 7.25–7.22 (m, 1H), 7.17–7.16 (m, 1H), 5.73 (dd, J = 8.6, 5.6 Hz, 1H), 3.42 (s, 3H), 1.27–1.15 (m, 18H), 0.86 (t, J = 6.9 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.2, 130.2, 128.4, 126.4, 124.4, 118.6, 115.5, 32.0, 31.4, 29.6, 29.5, 29.5, 29.4, 29.3, 29.1, 25.4, 22.8, 14.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{19}H_{30}N_3O_2^+$ 332.2333, found 332.2330.



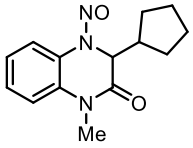
12

3-isopropyl-1-methyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **12** (17.5 mg, 75% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 1H), 7.43–7.39 (m, 1H), 7.24–7.21 (m, 1H), 7.16–7.14 (m, 1H), 5.54 (d, J = 7.4 Hz, 1H), 3.42 (s, 3H), 1.97–1.90 (m, 1H), 0.92 (d, J = 6.8 Hz, 3H), 0.74 (d, J = 6.8 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.2, 130.5, 128.4, 127.4, 124.3, 118.5, 115.5, 59.0, 32.0, 29.5, 19.1, 19.0 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{16}N_3O_2^+$ 234.1237, found 234.1240.



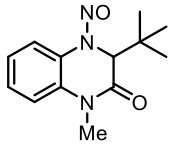
13

3-cyclopentyl-1-methyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **13** (20.7 mg, 80% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82–7.80 (m, 1H), 7.44–7.40 (m, 1H), 7.25–7.21 (m, 1H), 7.17–7.15 (m, 1H), 5.67 (d, J = 8.9 Hz, 1H), 3.41 (s, 3H), 2.04–1.96 (m, 1H), 1.72–1.24 (m, 7H), 1.15–1.07 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.5, 130.5, 128.5, 127.0, 124.2, 118.7, 115.5, 56.9, 42.5, 29.5, 29.4, 29.0, 24.7, 24.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{18}N_3O_2^+$ 260.1394, found 260.1395.



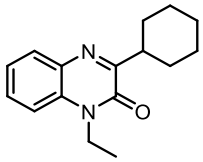
14

3-(*tert*-butyl)-1-methyl-4-nitroso-3,4-dihydroquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure B (eluent: PE/EtOAc, 8/1, v/v). The product **14** (18.8 mg, 76% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.79–7.77 (m, 1H), 7.43–7.39 (m, 1H), 7.25–7.22 (m, 1H), 7.14–7.12 (m, 1H), 5.57 (s, 1H), 3.42 (s, 3H), 0.84 (s, 9H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.7, 131.2, 128.5, 128.3, 124.2, 118.8, 115.2, 61.0, 38.3, 29.4, 27.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{13}H_{18}N_3O_2^+$ 248.1394, found 248.1399.



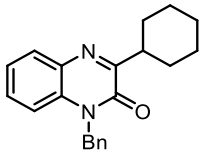
15

3-cyclohexyl-1-ethylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **15** (20.5 mg, 80% yield) was obtained as a yellowish powder.[15]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.82 (m, 1H), 7.50–7.47 (m, 1H), 7.31–7.28 (m, 2H), 4.30 (q, *J* = 7.2 Hz, 2H), 3.34 (tt, *J* = 11.6, 3.3 Hz, 1H), 1.97–1.92 (m, 2H), 1.87–1.83 (m, 2H), 1.77–1.73 (m, 1H), 1.60–1.41 (m, 4H), 1.36 (t, *J* = 7.2 Hz, 3H), 1.34–1.26 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.4, 154.1, 133.3, 131.8, 130.1, 129.4, 123.3, 113.4, 40.7, 37.4, 30.7, 26.5, 26.3, 12.5 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{21}N_2O^+$ 257.1648, found 257.1650.



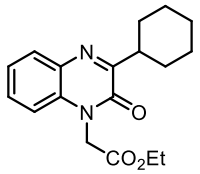
16

1-benzyl-3-cyclohexylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **16** (26.1 mg, 82% yield) was obtained as a white powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.82 (m, 1H), 7.38–7.20 (m, 8H), 5.48 (s, 2H), 3.40 (tt, *J* = 11.6, 3.3 Hz, 1H), 2.02–1.99 (m, 2H), 1.90–1.85 (m, 2H), 1.80–1.74 (m, 1H), 1.64–1.56 (m, 2H), 1.52–1.43 (m, 2H), 1.36–1.25 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.5, 154.7, 135.6, 133.2, 132.3, 130.0, 129.5, 129.0, 127.7, 127.0, 123.6, 114.3, 46.0, 40.9, 30.7, 26.4, 26.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{23}N_2O^+$ 319.1805, found 319.1803.



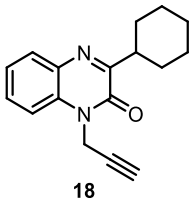
17

ethyl 2-(3-cyclohexyl-2-oxoquinoxalin-1(2*H*)-yl)acetate: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **17** (23.6 mg, 75% yield) was obtained as a yellowish powder.[15]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.83 (m, 1H), 7.47–7.43 (m, 1H), 7.32–7.29 (m, 1H), 7.04–7.02 (m, 1H), 5.00 (s, 2H), 4.23 (q, *J* = 7.1 Hz, 2H), 3.31 (tt, *J* = 11.6, 3.3 Hz, 1H), 1.97–1.94 (m, 2H), 1.87–1.82 (m, 2H), 1.76–1.73 (m, 1H), 1.60–1.52 (m, 2H), 1.48–1.39 (m, 2H), 1.33–1.30 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.3, 164.1, 154.2, 133.0, 132.1, 130.2, 129.6, 123.8, 113.0, 62.1, 43.7, 40.9, 30.6, 26.4, 26.2, 14.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{23}N_2O_3^+$ 315.1703, found 315.1704.



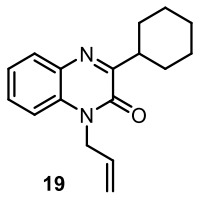
18

3-cyclohexyl-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **18** (21.6 mg, 81% yield) was obtained as a white powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.85–7.83 (m, 1H), 7.54–7.51 (m, 1H), 7.43–7.41 (m, 1H), 7.35–7.32 (m, 1H), 5.04 (d, J = 2.5 Hz, 2H), 3.32 (tt, J = 11.6, 3.3 Hz, 1H), 2.28 (t, J = 2.5 Hz, 1H), 1.98–1.93 (m, 2H), 1.88–1.83 (m, 2H), 1.77–1.73 (m, 1H), 1.60–1.52 (m, 2H), 1.49–1.40 (m, 2H), 1.34–1.25 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.2, 153.6, 133.2, 131.4, 130.0, 129.6, 123.9, 114.0, 76.9, 73.2, 40.9, 31.6, 30.6, 26.4, 26.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{19}N_2O^+$ 267.1492, found 267.1497.



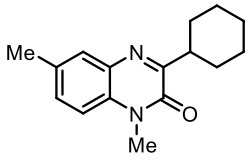
19

1-allyl-3-cyclohexylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **19** (20.6 mg, 77% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.82 (m, 1H), 7.47–7.44 (m, 1H), 7.31–7.24 (m, 2H), 5.97–5.89 (m, 1H), 5.26–5.14 (m, 2H), 4.90–4.88 (m, 2H), 3.34 (tt, *J* = 11.6, 3.3 Hz, 1H), 1.98–1.94 (m, 2H), 1.88–1.82 (m, 2H), 1.78–1.73 (m, 1H), 1.61–1.53 (m, 2H), 1.50–1.41 (m, 2H), 1.35–1.26 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.4, 154.2, 133.1, 132.1, 130.9, 129.9, 129.4, 123.5, 118.1, 114.1, 44.6, 40.8, 30.6, 26.4, 26.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{21}N_2O^+$ 269.1648, found 269.1650.



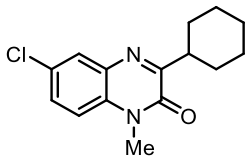
20

3-cyclohexyl-1,6-dimethylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **20** (20.0 mg, 78% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.70–7.63 (m, 1H), 7.31–7.05 (m, 2H), 3.66 (s, 3H), 3.35–3.27 (m, 1H), 2.48 and 2.42 (s, 3H), 1.96–1.92 (m, 2H), 1.87–1.82 (m, 2H), 1.77–1.72 (m, 1H), 1.59–1.40 (m, 4H), 1.34–1.25 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.3, 163.1, 154.8, 154.6, 140.1, 133.3, 132.9, 132.8, 131.2, 130.7, 130.6, 129.7, 129.5, 124.7, 113.7, 113.3, 40.8, 30.7, 30.6, 29.2, 29.1, 26.4, 26.3, 22.1, 20.7 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{21}N_2O^+$ 257.1648, found 257.1640.



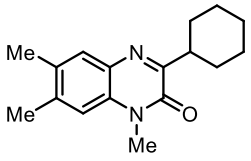
21

6-chloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **21** (19.9 mg, 72% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82–7.81 (m, 1H), 7.45–7.42 (m, 1H), 7.20–7.18 (m, 1H), 3.66 (s, 3H), 3.34–3.28 (m, 1H), 1.96–1.91 (m, 2H), 1.87–1.82 (m, 2H), 1.78–1.72 (m, 1H), 1.56–1.40 (m, 4H), 1.34–1.24 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.8, 154.3, 133.5, 131.7, 129.4, 129.2, 128.8, 114.7, 40.9, 30.6, 29.4, 26.4, 26.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{18}ClN_2O^+$ 277.1102, found 277.1110.



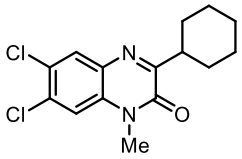
22

3-cyclohexyl-1,6,7-trimethylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **22** (20.5 mg, 76% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.58 (s, 1H), 7.02 (s, 1H), 3.65 (s, 3H), 3.30 (tt, J = 11.6, 3.3 Hz, 1H), 2.38 (s, 3H), 2.32 (s, 3H), 1.95–1.91 (m, 2H), 1.88–1.82 (m, 2H), 1.77–1.72 (m, 1H), 1.58–1.40 (m, 4H), 1.33–1.24 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 163.1, 154.7, 139.1, 132.3, 131.4, 130.9, 130.0, 114.2, 40.7, 30.7, 29.1, 26.5, 26.3, 20.6, 19.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{23}N_2O^+$ 271.1805, found 271.1810.



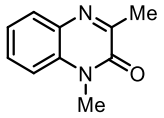
23

6,7-dichloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **23** (23.3 mg, 75% yield) was obtained as a yellow powder.[15]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.88 (s, 1H), 7.33 (s, 1H), 3.63 (s, 3H), 3.28 (tt, J = 11.6, 3.3 Hz, 1H), 1.92–1.89 (m, 2H), 1.86–1.82 (m, 2H), 1.77–1.72 (m, 1H), 1.53–1.39 (m, 4H), 1.31–1.23 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.9, 154.0, 133.4, 132.4, 132.0, 130.7, 127.2, 115.0, 41.0, 30.6, 29.4, 26.3, 26.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{15}H_{17}Cl_2N_2O^+$ 311.0712, found 311.0711.



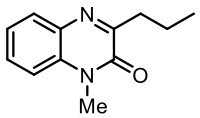
24

1,3-dimethylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **24** (9.4 mg, 54% yield) was obtained as a light orange powder.[16]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.81–7.79 (m, 1H), 7.54–7.50 (m, 1H), 7.35–7.28 (m, 2H), 3.70 (s, 3H), 2.59 (s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 158.5, 155.3, 133.4, 132.8, 129.7, 129.6, 123.8, 113.8, 29.2, 21.8 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{10}H_{11}N_2O^+$ 175.0866, found 175.0870.



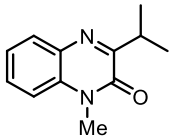
25

1-methyl-3-propylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **25** (12.7 mg, 63% yield) was obtained as a light pink powder.[16]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 1H), 7.53–7.49 (m, 1H), 7.34–7.27 (m, 2H), 3.69 (s, 3H), 2.93–2.90 (m, 2H), 1.84–1.79 (m, 2H), 1.04 (t, J = 7.4 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.3, 155.0, 133.2, 132.8, 129.7, 129.6, 123.6, 113.7, 36.4, 29.2, 20.4, 14.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{15}N_2O^+$ 203.1179, found 203.1177.



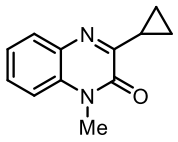
26

3-isopropyl-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **26** (15.8 mg, 78% yield) was obtained as a yellow powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.85–7.83 (m, 1H), 7.52–7.48 (m, 1H), 7.34–7.27 (m, 2H), 3.69 (s, 3H), 3.65–3.59 (m, 1H), 1.31 (d, J = 6.7 Hz, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 165.1, 154.6, 133.1, 132.9, 129.9, 129.6, 123.5, 113.6, 31.3, 29.2, 20.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{15}N_2O^+$ 203.1179, found 203.1178.



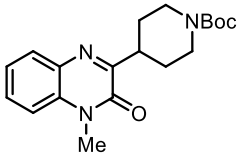
27

3-cyclopropyl-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **27** (14.0 mg, 70% yield) was obtained as a yellow powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = **3-cyclopropyl-1-methylquinoxalin-2(1*H*)-one** ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.6, 155.4, 133.0, 132.5, 129.3, 128.9, 123.6, 113.6, 29.3, 12.6, 11.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{12}H_{13}N_2O^+$ 201.1022, found 201.1025.



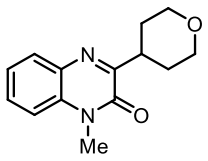
28

***tert*-butyl 4-(4-methyl-3-oxo-3,4-dihydroquinoxalin-2-yl)piperidine-1-carboxylate:** This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 4/1, v/v). The product **28** (27.8 mg, 81% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82–7.80 (m, 1H), 7.53–7.50 (m, 1H), 7.34–7.27 (m, 2H), 4.23 (brs, 2H), 3.69 (s, 3H), 3.45 (tt, J = 11.6, 3.3 Hz, 1H), 2.90 (brs, 2H), 1.93–1.89 (m, 2H), 1.79–1.70 (m, 2H), 1.46 (s, 9H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 162.3, 154.9, 154.5, 133.0, 132.8, 130.0, 129.9, 123.7, 113.7, 79.4, 43.6, 39.1, 29.5, 29.2, 28.6 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{19}H_{26}N_3O_3^+$ 344.1969, found 344.1970.



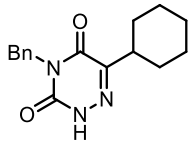
29

1-methyl-3-(tetrahydro-2*H*-pyran-4-yl)quinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 4/1, v/v). The product **29** (18.3 mg, 75% yield) was obtained as a yellowish powder.[14]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.86–7.84 (m, 1H), 7.54–7.51 (m, 1H), 7.36–7.28 (m, 2H), 4.11–4.07 (m, 2H), 3.70 (s, 3H), 3.64–3.52 (m, 3H), 1.99–1.86 (m, 4H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 162.1, 154.5, 133.0, 132.9, 130.1, 129.9, 123.7, 113.6, 68.0, 38.2, 30.2, 29.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{14}H_{17}N_2O_2^+$ 245.1285, found 245.1293.



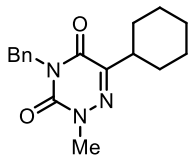
30

4-benzyl-6-cyclohexyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 3/1, v/v). The product **30** (19.4 mg, 68% yield) was obtained as a white powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 10.05 (brs, 1H), 7.50–7.48 (m, 2H), 7.34–7.27 (m, 3H), 5.08 (s, 2H), 2.89–2.84 (m, 1H), 1.88–1.69 (m, 5H), 1.41–1.19 (m, 5H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.9, 149.1, 148.8, 135.9, 129.6, 128.6, 128.1, 44.2, 39.5, 38.5, 30.5, 26.3, 26.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{16}H_{20}N_3O_2^+$ 286.1550, found 286.1547.



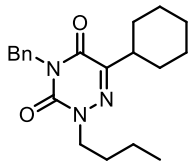
31

4-benzyl-6-cyclohexyl-2-methyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **31** (26.0 mg, 87% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51–7.49 (m, 2H), 7.33–7.26 (m, 3H), 5.09 (s, 2H), 3.59 (s, 3H), 2.90–2.84 (m, 1H), 1.87–1.70 (m, 5H), 1.42–1.31 (m, 4H), 1.26–1.18 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.9, 149.1, 148.8, 135.9, 129.6, 128.6, 128.1, 44.2, 39.5, 38.5, 30.5, 26.3, 26.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{17}H_{22}N_3O_2^+$ 300.1707, found 300.1714.



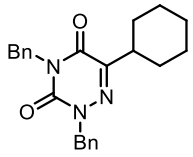
32

4-benzyl-2-butyl-6-cyclohexyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **32** (26.3 mg, 77% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51–7.48 (m, 2H), 7.33–7.26 (m, 3H), 5.09 (s, 2H), 3.95–3.92 (m, 2H), 2.90–2.85 (m, 1H), 1.87–1.79 (m, 4H), 1.74–1.70 (m, 3H), 1.42–1.31 (m, 6H), 1.27–1.18 (m, 1H), 0.95 (t, J = 7.4 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.8, 148.9, 148.6, 136.0, 129.6, 128.6, 128.0, 51.4, 44.2, 38.5, 30.6, 30.3, 26.3, 26.1, 19.8, 13.8 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{28}N_3O_2^+$ 342.2176, found 342.2175.



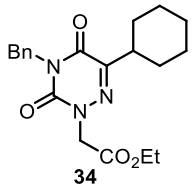
33

2,4-dibenzyl-6-cyclohexyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **33** (32.3 mg, 86% yield) was obtained as a yellowish powder.[2]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51–7.49 (m, 2H), 7.43–7.41 (m, 2H), 7.38–7.28 (m, 6H), 5.09 (s, 2H), 5.08 (s, 2H), 2.91–2.86 (m, 1H), 1.90–1.80 (m, 4H), 1.76–1.72 (m, 1H), 1.43–1.34 (m, 4H), 1.29–1.22 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.8, 149.1, 148.9, 136.0, 135.9, 129.6, 128.8, 128.7, 128.6, 128.2, 128.1, 55.4, 44.3, 38.5, 30.6, 26.2, 26.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{23}H_{26}N_3O_2^+$ 376.2020, found 376.2025.



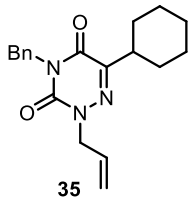
34

ethyl 2-(4-benzyl-6-cyclohexyl-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)acetate: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **34** (30.8 mg, 83% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.47–7.44 (m, 2H), 7.32–7.25 (m, 3H), 5.09 (s, 2H), 4.66 (s, 2H), 4.23 (q, J = 7.1 Hz, 2H), 2.90–2.85 (m, 1H), 1.88–1.68 (m, 5H), 1.38–1.32 (m, 4H), 1.27 (t, J = 7.1 Hz, 3H), 1.24–1.18 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.6, 155.7, 149.6, 149.2, 135.7, 129.4, 128.6, 128.1, 61.9, 52.9, 44.3, 38.5, 30.4, 26.2, 26.0, 14.2 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{26}N_3O_4^+$ 372.1918, found 372.1911.



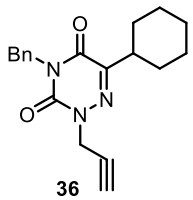
35

2-allyl-4-benzyl-6-cyclohexyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **35** (26.7 mg, 82% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51–7.48 (m, 2H), 7.34–7.26 (m, 3H), 5.98–5.90 (m, 1H), 5.29–5.23 (m, 2H), 5.10 (s, 2H), 4.54 (dt, J = 6.0, 1.4 Hz, 2H), 2.92–2.83 (m, 1H), 1.88–1.78 (m, 4H), 1.74–1.70 (m, 1H), 1.41–1.31 (m, 4H), 1.26–1.20 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.8, 149.1, 148.8, 135.9, 131.7, 129.6, 128.6, 128.1, 118.9, 54.1, 44.2, 38.6, 30.5, 26.2, 26.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{19}H_{24}N_3O_2^+$ 326.1863, found 326.1866.



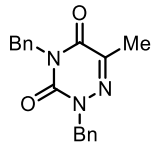
36

4-benzyl-6-cyclohexyl-2-(prop-2-yn-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **36** (27.1 mg, 84% yield) was obtained as a yellowish powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.52–7.50 (m, 2H), 7.34–7.26 (m, 3H), 5.09 (s, 2H), 4.71 (d, J = 2.5 Hz, 2H), 2.90–2.87 (m, 1H), 2.35 (t, J = 2.5 Hz, 1H), 1.90–1.79 (m, 4H), 1.74–1.71 (m, 1H), 1.42–1.32 (m, 4H), 1.27–1.21 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.7, 149.7, 148.5, 135.7, 129.7, 128.6, 128.2, 77.3, 73.3, 44.3, 41.5, 38.7, 30.4, 26.2, 26.0 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{19}H_{22}N_3O_2^+$ 324.1707, found 324.1710.



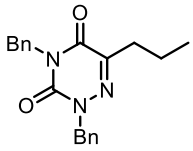
37

2,4-dibenzyl-6-methyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **37** (20.9 mg, 68% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.50–7.48 (m, 2H), 7.42–7.28 (m, 8H), 5.09 (s, 2H), 5.08 (s, 2H), 2.24(s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 156.4, 149.3, 143.0, 136.0, 135.7, 129.6, 128.8, 128.7, 128.7, 128.3, 128.2, 55.3, 44.3, 17.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{18}H_{18}N_3O_2^+$ 308.1394, found 308.1342.



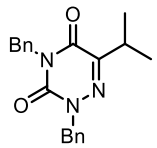
38

2,4-dibenzyl-6-propyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **38** (24.5 mg, 73% yield) was obtained as a colorless oil.[17]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.50–7.47 (m, 2H), 7.42–7.27 (m, 8H), 5.09 (s, 2H), 5.08 (s, 2H), 2.60–2.57 (m, 2H), 1.68–1.64 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 156.2, 149.1, 145.7, 136.0, 135.8, 129.5, 128.8, 128.7, 128.3, 128.1, 55.3, 44.3, 32.4, 19.7, 13.8 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{22}N_3O_2^+$ 336.1707, found 336.1710.



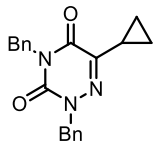
39

2,4-dibenzyl-6-isopropyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **39** (26.8 mg, 80% yield) was obtained as a colorless oil.[17]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51–7.49 (m, 2H), 7.43–7.27 (m, 8H), 5.10 (s, 2H), 5.09 (s, 2H), 3.22–3.16 (m, 1H), 1.21 (d, J = 6.8 Hz, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.7, 149.6, 149.0, 136.0, 135.9, 129.6, 128.9, 128.8, 128.7, 128.2, 128.1, 55.3, 44.2, 29.3, 20.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{22}N_3O_2^+$ 336.1707, found 336.1709.



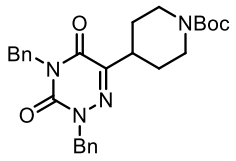
40

2,4-dibenzyl-6-cyclopropyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **40** (26.0 mg, 78% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.52–7.50 (m, 2H), 7.39–7.28 (m, 8H), 5.11 (s, 2H), 5.04 (s, 2H), 2.31–2.25 (m, 1H), 1.00–0.93 (m, 4H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 156.2, 148.9, 146.7, 135.9, 135.8, 129.5, 128.7, 128.6, 128.2, 128.1, 55.2, 44.3, 9.9, 9.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{20}N_3O_2^+$ 334.1550, found 334.1555.



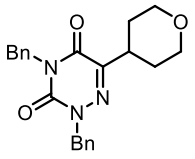
41

***tert*-butyl 4-(2,4-dibenzyl-3,5-dioxo-2,3,4,5-tetrahydro-1,2,4-triazin-6-yl)piperidine-1-carboxylate:** This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 4/1, v/v). The product **41** (41.9 mg, 88% yield) was obtained as a colorless oil.[17]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.49–7.47 (m, 2H), 7.41–7.28 (m, 8H), 5.08 (s, 2H), 5.07 (s, 2H), 4.19 (brs, 2H), 3.01 (tt, J = 11.6, 3.3 Hz, 1H), 2.82 (brs, 2H), 1.87–1.83 (m, 2H), 1.57–1.54 (m, 2H), 1.48 (s, 9H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.6, 154.8, 148.8, 147.1, 135.7, 135.7, 129.6, 128.9, 128.8, 128.6, 128.3, 128.1, 79.6, 55.4, 44.3, 43.6, 36.8, 29.4, 28.5 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{33}N_4O_4^+$ 477.2496, found 477.2500.



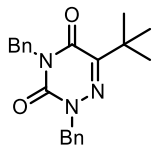
42

2,4-dibenzyl-6-(tetrahydro-2*H*-pyran-4-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 4/1, v/v). The product **42** (32.1 mg, 85% yield) was obtained as a white powder.[17]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51–7.48 (m, 2H), 7.43–7.27 (m, 8H), 5.10 (s, 2H), 5.08 (s, 2H), 4.06–4.02 (m, 2H), 3.54–3.49 (m, 2H), 3.15–3.09 (m, 1H), 1.83–1.71 (m, 4H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.6, 148.8, 147.1, 135.7, 135.7, 129.6, 128.9, 128. 8, 128.7, 128.3, 128.2, 67.8, 55.4, 44.3, 35.9, 30.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{22}H_{24}N_3O_3^+$ 378.1812, found 378.1811.



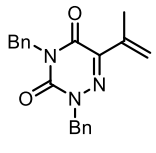
43

2,4-dibenzyl-6-(*tert*-butyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **43** (26.9 mg, 77% yield) was obtained as a white powder.[17]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.50–7.42 (m, 4H), 7.38–7.29 (m, 6H), 5.08 (s, 4H), 1.34 (s, 9H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 154.9, 150.4, 149.1, 136.1, 135.9, 129.5, 129.1, 128.8, 128.6, 128.3, 128.0, 55.3, 44.2, 37.4, 27.9 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{24}N_3O_2^+$ 350.1863, found 350.1866.



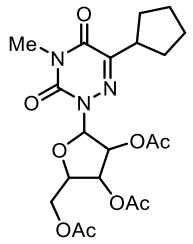
44

2,4-dibenzyl-6-(prop-1-en-2-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 10/1, v/v). The product **44** (16.7 mg, 50% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.50–7.42 (m, 4H), 7.38–7.28 (m, 6H), 6.53–6.52 (m, 1H), 5.58–5.57 (m, 1H), 5.14 (s, 2H), 5.12 (s, 2H), 2.04 (s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.2, 148.7, 141.0, 136.8, 135.8, 135.7, 129.5, 129.0, 128.8, 128.8, 128.7, 128.4, 128.1, 122.4, 55.7, 44.4, 21.4 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{20}N_3O_2^+$ 334.1550, found 334.1547.



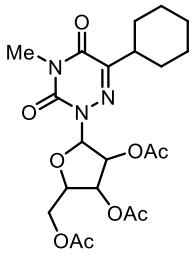
45

2-(acetoxymethyl)-5-(6-cyclopentyl-4-methyl-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl diacetate: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 3/1, v/v). The product **45** (25.4 mg, 56% yield) was obtained as a yellowish oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.33–6.32 (m, 1H), 5.63–5.61 (m, 1H), 5.48–5.45 (m, 1H), 4.36–4.32 (m, 2H), 4.16–4.12 (m, 1H), 3.33–3.29 (m, 4H), 2.11 (s, 3H), 2.09 (s, 3H), 2.06 (s, 3H), 2.03–1.97 (m, 2H), 1.75–1.66 (m, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.7, 169.7, 169.7, 155.8, 149.8, 149.0, 89.1, 78.8, 73.2, 70.6, 63.7, 40.5, 30.7, 30.4, 27.4, 25.4, 20.8, 20.7, 20.6 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{20}H_{28}N_3O_9^+$ 454.1820, found 454.1815.



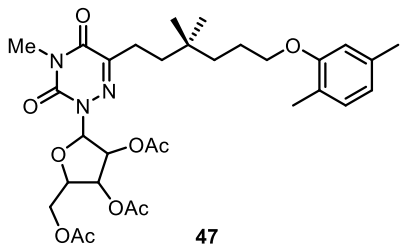
46

2-(acetoxymethyl)-5-(6-cyclohexyl-4-methyl-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl diacetate: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 3/1, v/v). The product **46** (35.0 mg, 75% yield) was obtained as a yellowish oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.32–6.31 (m, 1H), 5.63–5.61 (m, 1H), 5.49–5.47 (m, 1H), 4.35–4.31 (m, 2H), 4.16–4.11 (m, 1H), 3.30 (s, 3H), 2.90–2.85 (m, 1H), 2.10 (s, 3H), 2.08 (s, 3H), 2.05 (s, 3H), 1.95–1.88 (m, 2H), 1.84–1.78 (m, 2H), 1.74–1.69 (m, 1H), 1.41–1.29 (m, 4H), 1.28–1.17 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.6, 169.7, 169.6, 155.4, 150.3, 148.9, 89.0, 78.8, 73.2, 70.6, 63.8, 38.9, 30.7, 30.6, 27.4, 26.2, 26.0, 20.8, 20.6, 20.6 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{21}H_{30}N_3O_9^+$ 468.1977, found 468.1980.



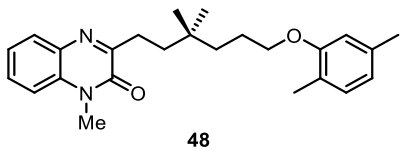
47

2-(acetoxymethyl)-5-(6-(6-(2,5-dimethylphenoxy)-3,3-dimethylhexyl)-4-methyl-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl diacetate: This compound was prepared by using the general procedure C (eluent: PE/EtOAc, 2/1, v/v). The product **47** (24.7 mg, 40% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.69–6.68 (m, 1H), 6.65–6.62 (m, 2H), 6.32 (d, J = 2.9 Hz, 1H), 5.65 (dd, J = 5.5, 2.9 Hz, 1H), 5.51–5.48 (m, 1H), 4.37–4.33 (m, 2H), 4.18–4.14 (m, 1H), 3.92 (t, J = 6.4 Hz, 2H), 3.33 (s, 3H), 2.66–2.62 (m, 2H), 2.30 (s, 3H), 2.17 (s, 3H), 2.12 (s, 3H), 2.07 (s, 3H), 2.05 (s, 3H), 1.80–1.74 (m, 2H), 1.64–1.53 (m, 2H), 1.45–1.42 (m, 2H), 0.98 (s, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.5, 169.6, 169.5, 157.2, 155.8, 149.0, 147.5, 136.5, 130.3, 123.6, 120.6, 112.1, 89.3, 79.1, 73.2, 70.7, 68.6, 63.7, 38.0, 37.7, 32.6, 27.4, 27.1, 25.8, 24.4, 21.5, 20.8, 20.6, 20.6, 15.9 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{31}H_{44}N_3O_{10}^+$ 618.3021, found 618.3026.



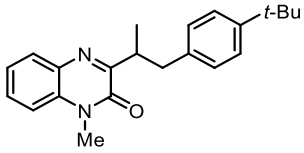
48

3-(6-(2,5-dimethylphenoxy)-3,3-dimethylhexyl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/DCM, 1/1, v/v). The product **48** (21.6 mg, 55% yield) was obtained as a white powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.85–7.83 (m, 1H), 7.53–7.50 (m, 1H), 7.35–7.28 (m, 2H), 6.99 (d, J = 7.4 Hz, 1H), 6.65–6.63 (m, 2H), 3.94 (t, J = 6.4 Hz, 2H), 3.70 (s, 3H), 2.96–2.92 (m, 2H), 2.31 (s, 3H), 2.19 (s, 3H), 1.86–1.70 (m, 4H), 1.51–1.47 (m, 2H),1.03 (s, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.9, 157.2, 155.0, 136.5, 133.2, 132.9, 130.3, 129.7, 129.6, 123.7, 123.6, 120.6, 113.7, 112.1, 68.7, 38.4, 38.2, 32.9, 29.7, 29.1, 27.2, 24.4, 21.5, 16.0 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{25}H_{33}N_2O_2^+$ 393.2537, found 393.2535.



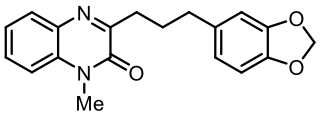
49

3-(1-(4-(*tert*-butyl)phenyl)propan-2-yl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **49** (18.0 mg, 54% yield) was obtained as a white powder.[18]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.89–7.87 (m, 1H), 7.54–7.50 (m, 1H), 7.36–7.33 (m, 4H), 7.23–7.21 (m, 2H), 3.87–3.82 (m, 1H), 3.70 (s, 3H), 3.26 (dd, J = 13.5, 5.9 Hz, 1H), 2.73 (dd, J = 13.5, 8.7 Hz, 1H), 1.30 (s, 9H), 1.27 (d, J = 6.9 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.1, 154.7, 148.7, 137.8, 133.1, 132.9, 129.9, 129.7, 129.1, 125.2, 123.6, 113.6, 40.0, 38.2, 34.5, 31.5, 29.2, 18.0 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{22}H_{27}N_2O^+$ 335.2118, found 335.2122.



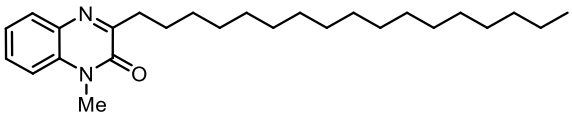
50

3-(3-(benzo[*d*][1,3]dioxol-5-yl)propyl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/DCM, 1/2, v/v). The product **50** (16.8 mg, 52% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.81 (m, 1H), 7.54–7.50 (m, 1H), 7.35–7.28 (m, 2H), 6.73–6.66 (m, 3H), 5.89 (s, 2H), 3.69 (s, 3H), 2.99–2.95 (m, 2H), 2.71–2.67 (m, 2H), 2.13–2.06 (m, 2H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 160.9, 155.0, 147.6, 145.6, 136.1, 133.2, 132.8, 129.8, 129.7, 123.7, 121.4, 113.7, 109.2, 108.2, 100.8, 35.5, 33.8, 29.2, 28.6 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{19}H_{19}N_2O_3^+$ 323.1390, found 323.1399.



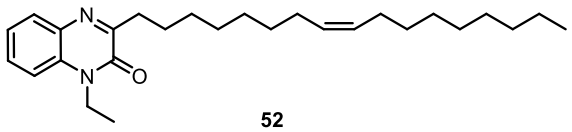
51

3-heptadecyl-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 10/1, v/v). The product **51** (23.9 mg, 60% yield) was obtained as a yellowish powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83–7.82 (m, 1H), 7.53–7.49 (m, 1H), 7.34–7.28 (m, 2H), 3.70 (s, 3H), 2.95–2.91 (m, 2H), 1.80–1.74 (m, 2H), 1.45–1.24 (m, 28H), 0.87 (t, J = 6.8 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.5, 155.1, 133.2, 132.9, 129.7, 129.6, 123.7, 113.7, 34.5, 32.1, 29.8, 29.8, 29.8, 29.7, 29.6, 29.5, 29.2, 27.0, 22.8, 14.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{26}H_{43}N_2O^+$ 399.3370, found 399.3377.



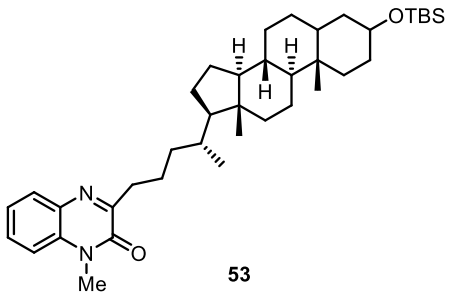
52

(*Z*)-1-ethyl-3-(heptadec-8-en-1-yl)quinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 10/1, v/v). The product **52** (20.9 mg, 51% yield) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.82 (m, 1H), 7.53–7.49 (m, 1H), 7.33–7.30 (m, 2H), 5.38–5.31 (m, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 2.95–2.91 (m, 2H), 2.02–1.98 (m, 4H), 1.80–1.76 (m, 2H), 1.46–1.22 (m, 23H), 0.87 (t, *J* =6.8 Hz, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.5, 154.5, 133.2, 132.1, 130.0, 130.0, 129.6, 123.4, 113.5, 37.4, 34.4, 32.0, 29.9, 29.8, 29.7, 29.5, 29.5, 29.4, 27.4, 27.0, 22.8, 14.3, 12.6 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{27}H_{43}N_2O^+$ 411.3370, found 411.3375.



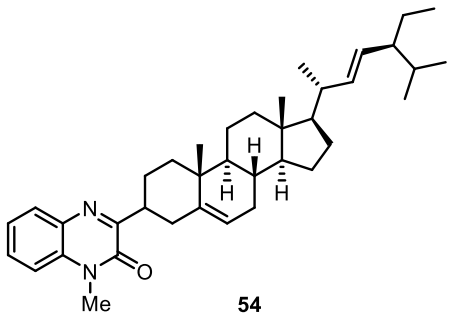
53

3-((4*R*)-4-((8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3-((*tert*-butyldimethylsilyl)oxy)-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentyl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 8/1, v/v). The product **53** (35.2 mg, 57% yield) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.81–7.79 (m, 1H), 7.50–7.47 (m, 1H), 7.32–7.25 (m, 2H), 3.67 (s, 3H), 3.59–3.53 (m, 1H), 3.02–2.96 (m, 1H), 2.82–2.76 (m, 1H), 1.97–1.72 (m, 7H), 1.57–1.00 (m, 24H), 0.88–0.87 (m, 12H), 0.63 (s, 3H), 0.04 (s, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 162.0, 155.0, 133.2, 132.8, 129.7, 129.5, 123.6, 113.6, 72.9, 56.5, 56.2, 42.9, 42.4, 40.3, 40.3, 37.0, 36.0, 36.0, 35.7, 34.7, 33.0, 31.4, 31.1, 29.1, 28.4, 27.4, 26.5, 26.1, 24.4, 23.5, 20.9, 18.7, 18.4, 12.2, -4.42 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{39}H_{63}N_2O_2Si^+$ 619.4653, found 619.4660.



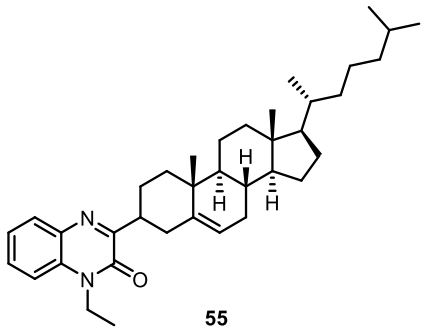
54

3-((8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-17-((2*R*,5*S*,*E*)-5-ethyl-6-methylhept-3-en-2-yl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 10/1, v/v). The product **54** (34.9 mg, 63% yield, dr = 1.5:1) was obtained as a light orange powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.85–7.78 (m, 1H), 7.50–7.45 (m, 1H), 7.32–7.22 (m, 2H), 5.40–4.98 (m, 3H), 3.68–3.30 (m, 4H), 2.80–2.25 (m, 2H), 2.13–0.95 (m, 29H), 0.87–0.78 (m, 9H), 0.71 and 0.69 (s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 163.5, 162.8, 154.5, 154.3, 142.4, 140.6, 138.5, 133.0, 132.6, 130.2, 129.9, 129.5, 129.3, 129.3, 123.5, 123.3, 121.0, 120.6, 113.5, 113.4, 57.1, 57.0, 56.2, 56.1, 51.3, 50.5, 50.1, 42.5, 42.3, 40.6, 39.9, 39.5, 37.3, 37.2, 37.2, 36.5, 35.1, 34.2, 32.1, 32.0, 31.9, 29.1, 29.1, 29.0, 26.7, 25.5, 24.5, 24.4, 24.1, 21.4, 21.2, 21.1, 20.9, 19.8, 19.8, 19.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{38}H_{55}N_2O^+$ 555.4309, found 555.4310.



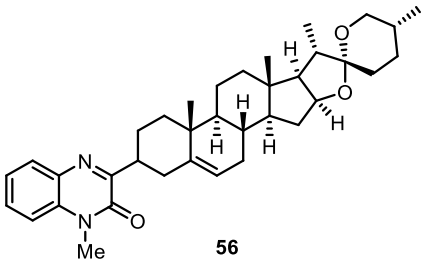
55

3-((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)-1-ethylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 10/1, v/v). The product **55** (39.0 mg, 72% yield, dr = 1.6:1) was obtained as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.86–7.79 (m, 1H), 7.51–7.46 (m, 1H), 7.31–7.27 (m, 2H), 5.40–5.21 (m, 1H), 4.33–4.25 (m, 2H), 3.65–3.62 (m, 1H), 2.83–2.77 and 2.28–2.2.24 (m, 1H), 2.66–2.55 (m, 1H), 1.65–0.84 (m, 41H), 0.69 and 0.67 (s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 163.6, 162.9, 154.0, 153.8, 142.4, 140.7, 133.3, 132.9, 131.9, 130.5, 130.2, 129.5, 129.3, 123.3, 123.1, 121.0, 120.5, 113.4, 113.3, 57.0, 56.9, 56.3, 56.3, 50.5, 50.1, 42.4, 40.0, 39.6, 39.5, 37.3, 37.2, 37.2, 37.2, 36.5, 36.3, 35.9, 35.9, 35.0, 34.3, 32.1, 32.0, 2.0, 28.4, 28.1, 26.8, 24.4, 24.3, 24.1, 24.0, 22.9, 22.7, 21.1, 20.9, 19.9, 19.8, 18.8, 12.6, 12.5, 12.0 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{37}H_{55}N_2O^+$ 543.4309, found 543.4305.



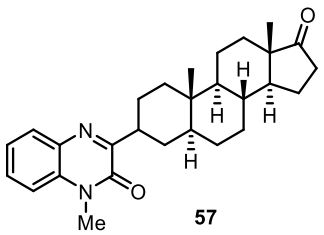
56

1-methyl-3-((5'*R*,6a*R*,6b*S*,8a*S*,8b*R*,9*S*,10*R*,11a*S*,12a*S*,12b*S*)-5',6a,8a,9-tetramethyl-1,3,3',4,4',5,5',6,6a,6b,6',7,8,8a,8b,9,11a,12,12a,12b-icosahydrospiro[naphtho[2',1':4,5]indeno[2,1-*b*]furan-10,2'-pyran]-4-yl)quinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 5/1, v/v). The product **56** (32.8 mg, 59% yield, dr = 1.9:1) was obtained as a yellow powder.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.84–7.78 (m, 1H), 7.51–7.46 (m, 1H), 7.33–7.24 (m, 2H), 5.39–5.20 (m, 1H), 4.44–4.34 (m, 1H), 3.69–3.60 (m, 3H), 3.49–3.28 (m, 3H), 2.82–2.24 (m, 2H), 2.02–1.41 (m, 20H), 1.17–0.95 (m, 8H), 0.81–0.76 (m, 6H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 163.5, 162.8, 154.6, 154.3, 142.4, 140.7, 133.0, 132.9, 132.6, 130.2, 129.9, 129.6, 129.4, 123.5, 123.3, 120.8, 120.3, 113.6, 113.5, 109.4, 81.0, 66.9, 62.2, 56.7, 50.4, 50.0, 42.5, 41.7, 40.4, 40.0, 39.5, 37.4, 37.3, 36.5, 35.0, 34.2, 32.2, 32.0, 31.9, 31.6, 31.5, 30.4, 29.1, 29.0, 28.9, 26.6, 24.1, 20.8, 20.7, 19.8, 19.8, 17.3, 16.4, 14.7 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{36}H_{49}N_2O_3^+$ 557.3738, found 557.3737.



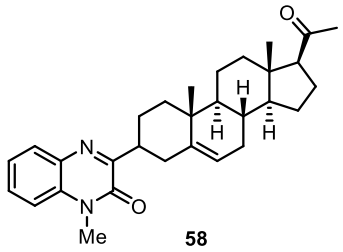
57

3-((5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-dimethyl-17-oxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 4/1, v/v). The product **57** (17.3 mg, 40% yield) was obtained as a yellowish oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.87–7.85 (m, 1H), 7.51–7.48 (m, 1H), 7.33–7.26 (m, 2H), 3.67 (s, 3H), 3.63–3.60 (m, 1H), 2.43–2.37 (m, 1H), 2.06–2.00 (m, 2H), 1.98–1.65 (m, 10H), 1.57–1.44 (m, 4H), 1.30–1.18 (m, 5H), 0.88 (s, 3H), 0.85 (s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 221.8, 163.8, 154.4, 133.1, 132.5, 130.2, 129.4, 123.4, 113.5, 54.7, 51.6, 47.9, 41.2, 36.2, 36.0, 35.5, 35.1, 34.8, 31.7, 30.9, 30.4, 29.1, 28.5, 23.7, 21.8, 20.2, 13.9, 11.9 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{28}H_{37}N_2O_2^+$ 433.2850, found 433.2857.



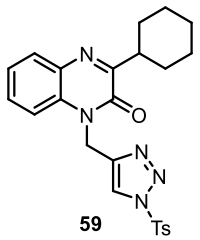
58

3-((8*S*,9*S*,10*R*,13*S*,14*S*,17*S*)-17-acetyl-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)-1-methylquinoxalin-2(1*H*)-one: This compound was prepared by using the general procedure A (eluent: PE/EtOAc, 4/1, v/v). The product **58** (17.0 mg, 37% yield) was obtained as a yellowish oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.80–7.78 (m, 1H), 7.51–7.47 (m, 1H), 7.32–7.25 (m, 2H), 5.22–5.20 (m, 1H), 3.67 (s, 3H), 3.66–3.61 (m, 1H), 2.82–2.77 (m, 1H), 2.58–2.47 (m, 2H), 2.18–1.80 (m, 10H), 1.67–1.38 (m, 7H), 1.24–1.00 (m, 6H), 0.61 (s, 3H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 209.8, 162.8, 154.3, 140.7, 133.0, 132.6, 130.1, 129.4, 123.3, 120.7, 113.5, 63.9, 57.1, 50.0, 44.2, 39.0, 37.3, 37.2, 35.1, 34.2, 31.9, 31.7, 29.1, 24.6, 24.1, 22.9, 20.9, 19.8, 13.3 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{30}H_{39}N_2O_2^+$ 459.3006, found 459.3010.



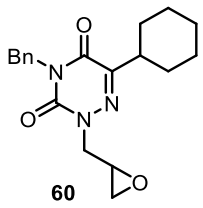
59

3-cyclohexyl-1-((1-tosyl-1*H*-1,2,3-triazol-4-yl)methyl)quinoxalin-2(1*H*)-one: This compound was prepared by using the procedure of synthetic application (eluent: PE/EtOAc, 5/1, v/v). The product **59** (39.4 mg, 85% yield) was obtained as a white powder.[19]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.20 (s, 1H), 7.96 (d, J = 8.1 Hz, 2H), 7.82–7.80 (m, 1H), 7.69–7.67 (m, 1H), 7.50–7.45 (m, 1H), 7.36–7.28 (m, 3H), 5.49 (s, 2H), 3.31 (tt, J = 11.6, 3.3 Hz, 1H), 2.42 (s, 3H), 1.96–1.93 (m, 4H), 1.89–1.85 (m, 1H), 1.60–1.42 (m, 4H), 1.35–1.24 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 164.0, 154.3, 147.6, 142.4, 133.2, 132.7, 131.7, 130.8, 130.0, 129.8, 129.0, 124.0, 123.6, 114.3, 40.9, 37.7, 30.7, 26.4, 26.2, 22.0 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{24}H_{26}N_5O_3S^+$ 464.1751, found 464.1746.



60

4-benzyl-6-cyclohexyl-2-(oxiran-2-ylmethyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione: This compound was prepared by using the procedure of synthetic application (eluent: PE/EtOAc, 7/1, v/v). The product **60** (24.2 mg, 71% yield) was obtained as a colorless oil.

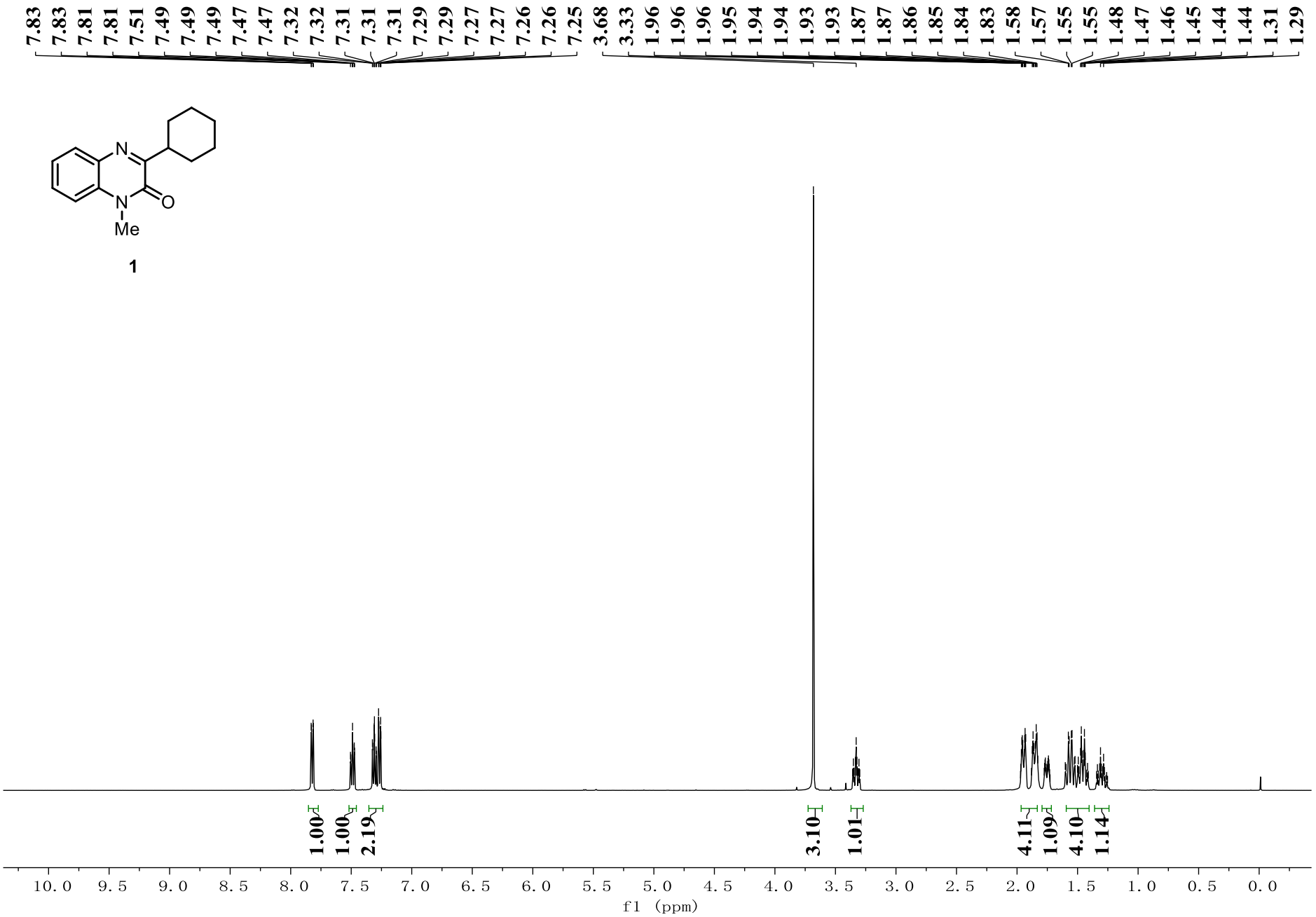
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.50–7.48 (m, 2H), 7.34–7.28 (m, 3H), 5.09 (s, 2H), 4.10–4.09 (m, 2H), 3.33–3.29 (m, 1H), 2.88–2.83 (m, 2H), 2.68–2.67 (m, 1H), 1.89–1.79 (m, 4H), 1.74–1.70 (m, 1H), 1.39–1.33 (m, 4H), 1.25–1.20 (m, 1H) ppm.

^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.7, 149.5, 149.2, 135.8, 129.7, 128.7, 128.2, 53.6, 49.2, 46.0, 44.4, 38.6, 30.5, 30.5, 26.2, 26.1 ppm.

HRMS (*m/z*): $[M+H]^+$ calcd for $C_{19}H_{24}N_3O_3^+$ 342.1812, found 342.1811.

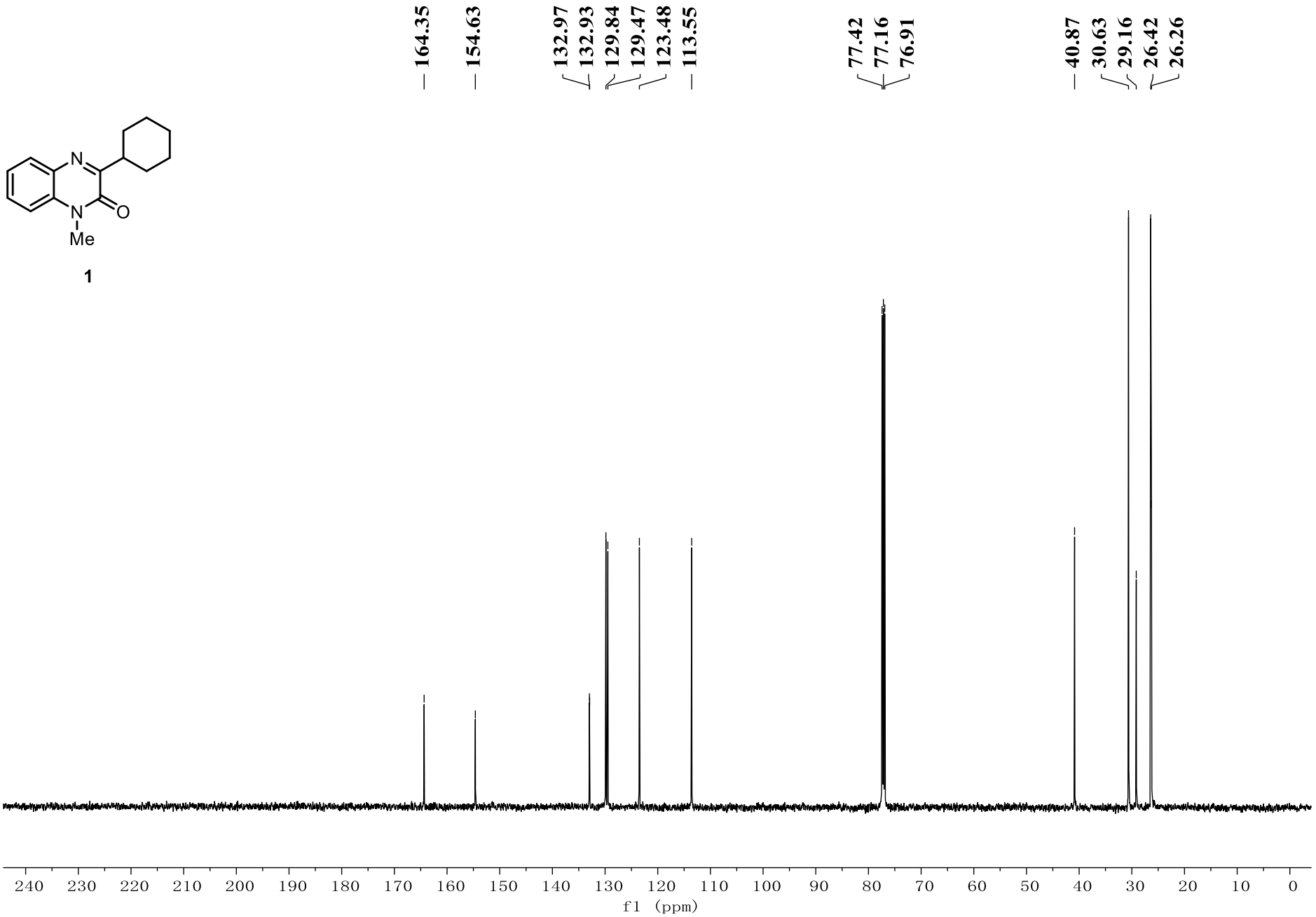
8. NMR spectra data.

^{1}H NMR (500 MHz, $CDCl_3$) for 1



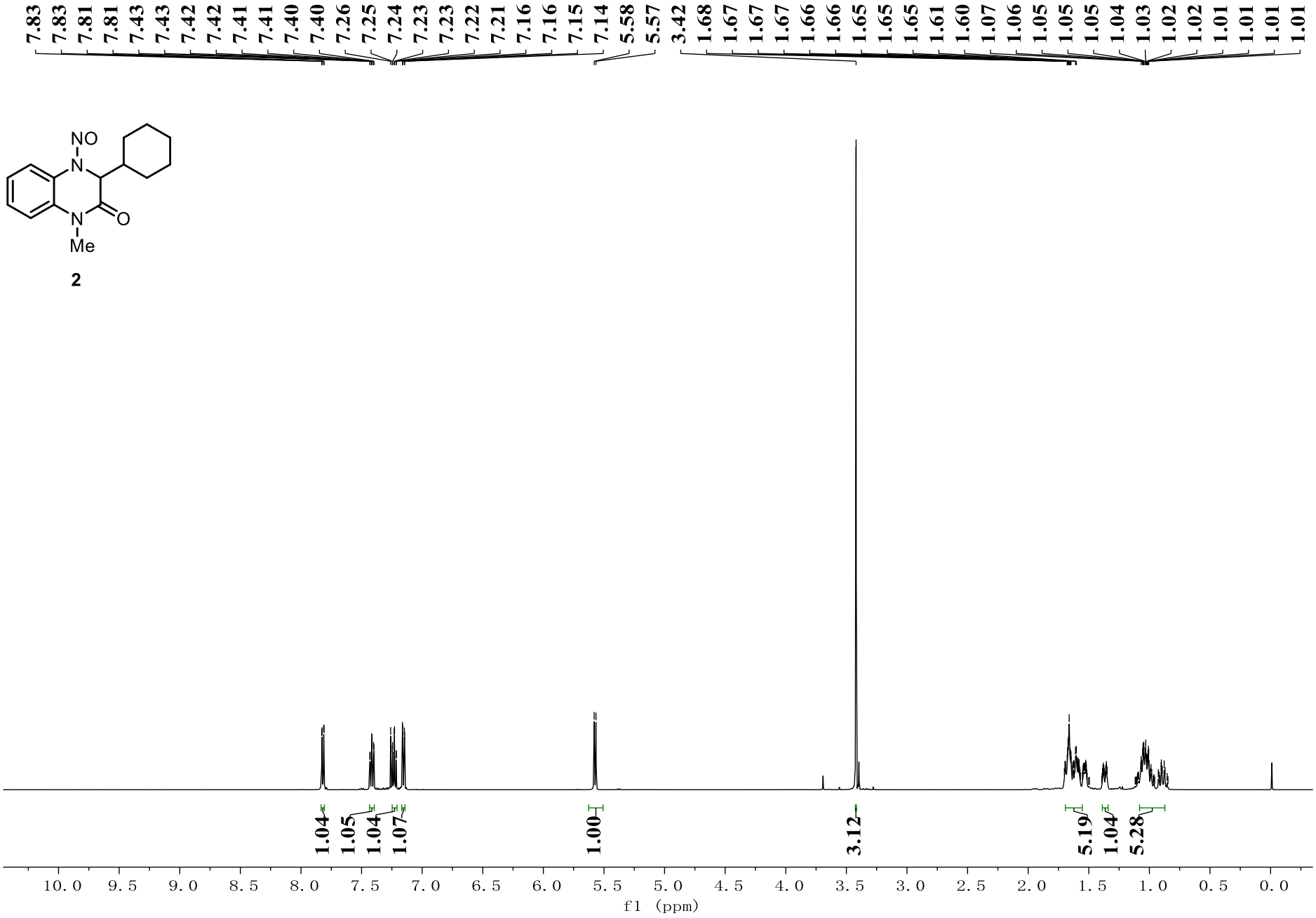
1

^{13}C NMR (125 MHz, $CDCl_3$) for 1



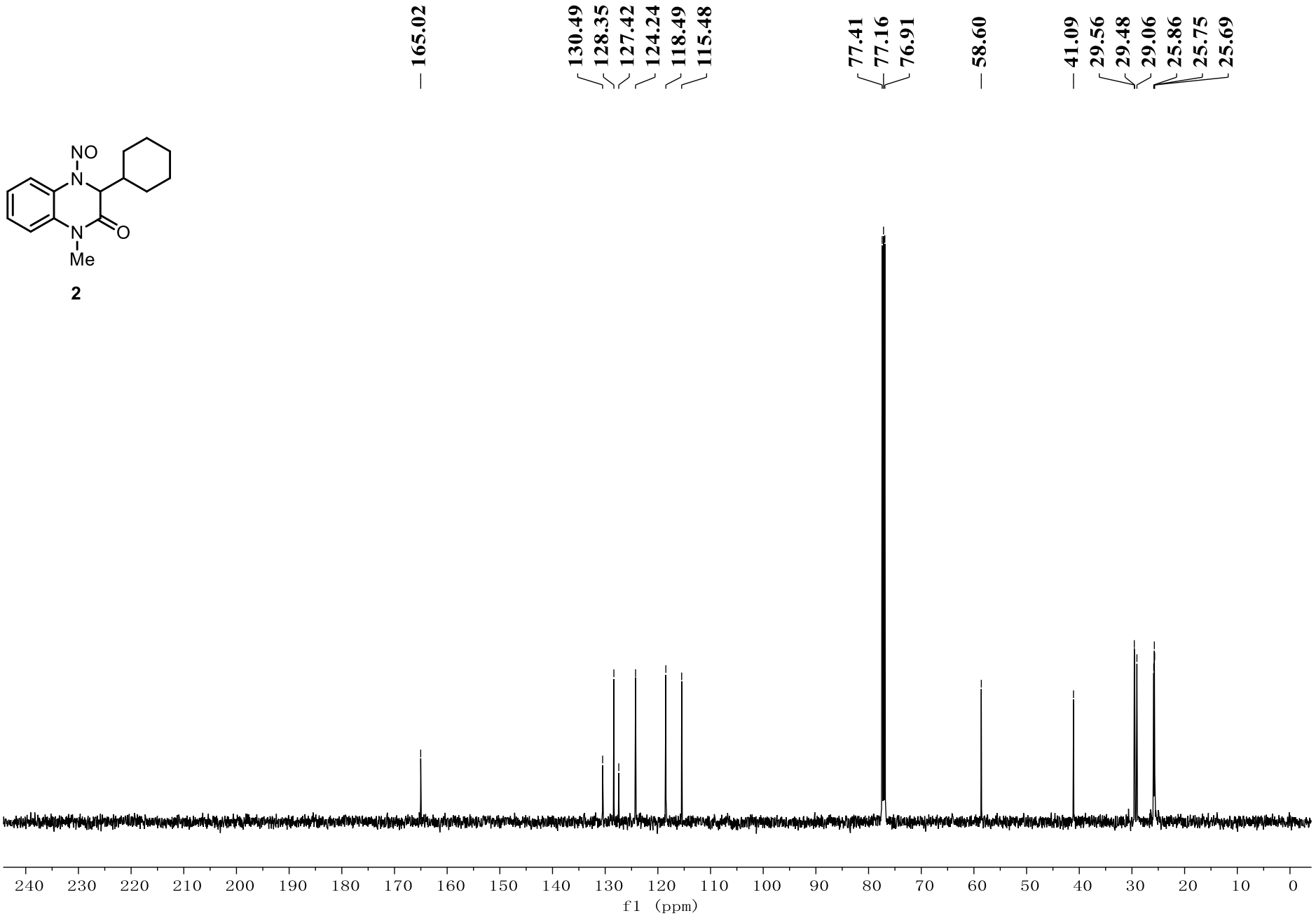
1

^{1}H NMR (500 MHz, $CDCl_3$) for 2

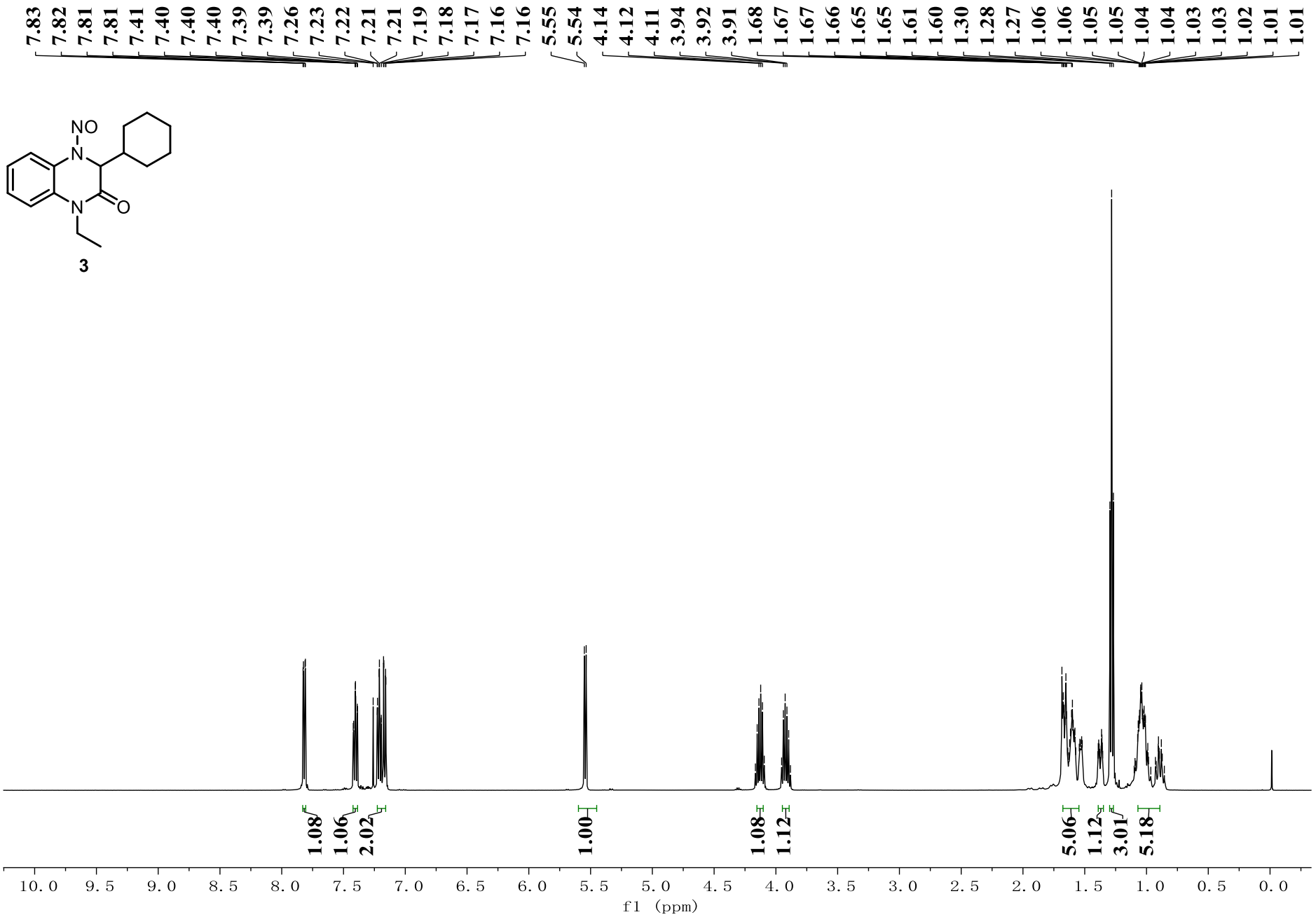


2

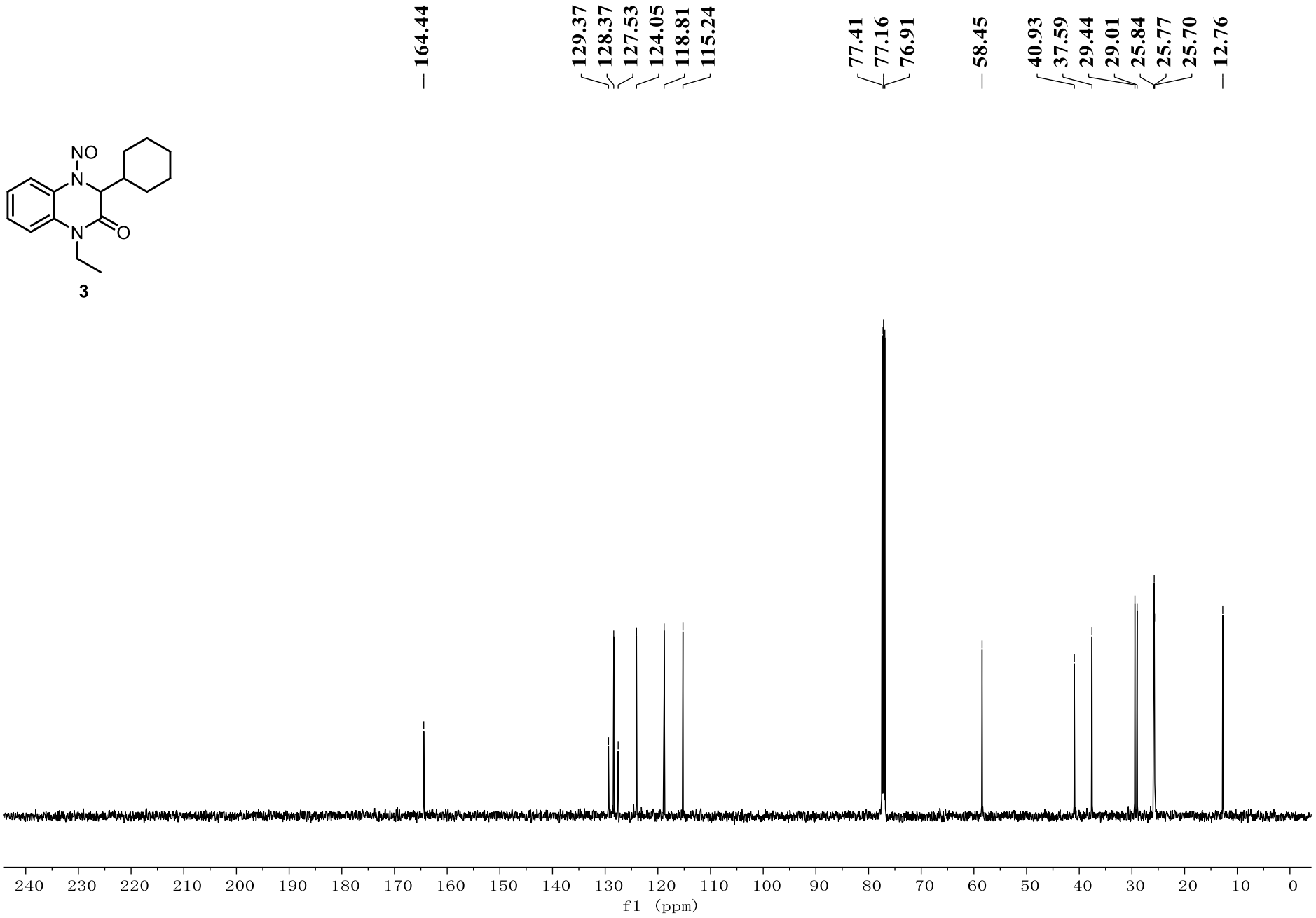
^{13}C NMR (125 MHz, $CDCl_3$) for 2



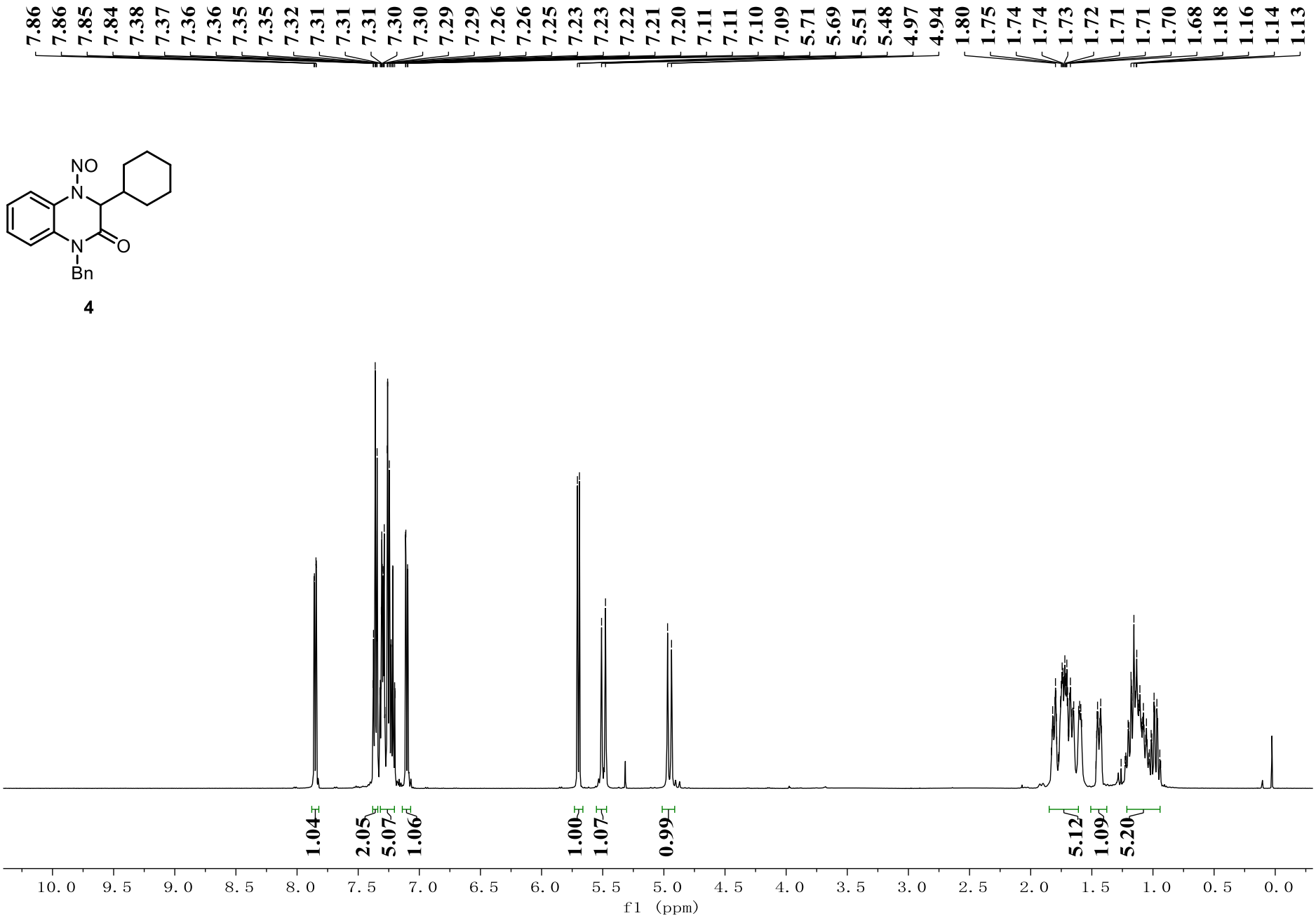
^{1}H NMR (500 MHz, $CDCl_3$) for 3



^{13}C NMR (125 MHz, $CDCl_3$) for 3

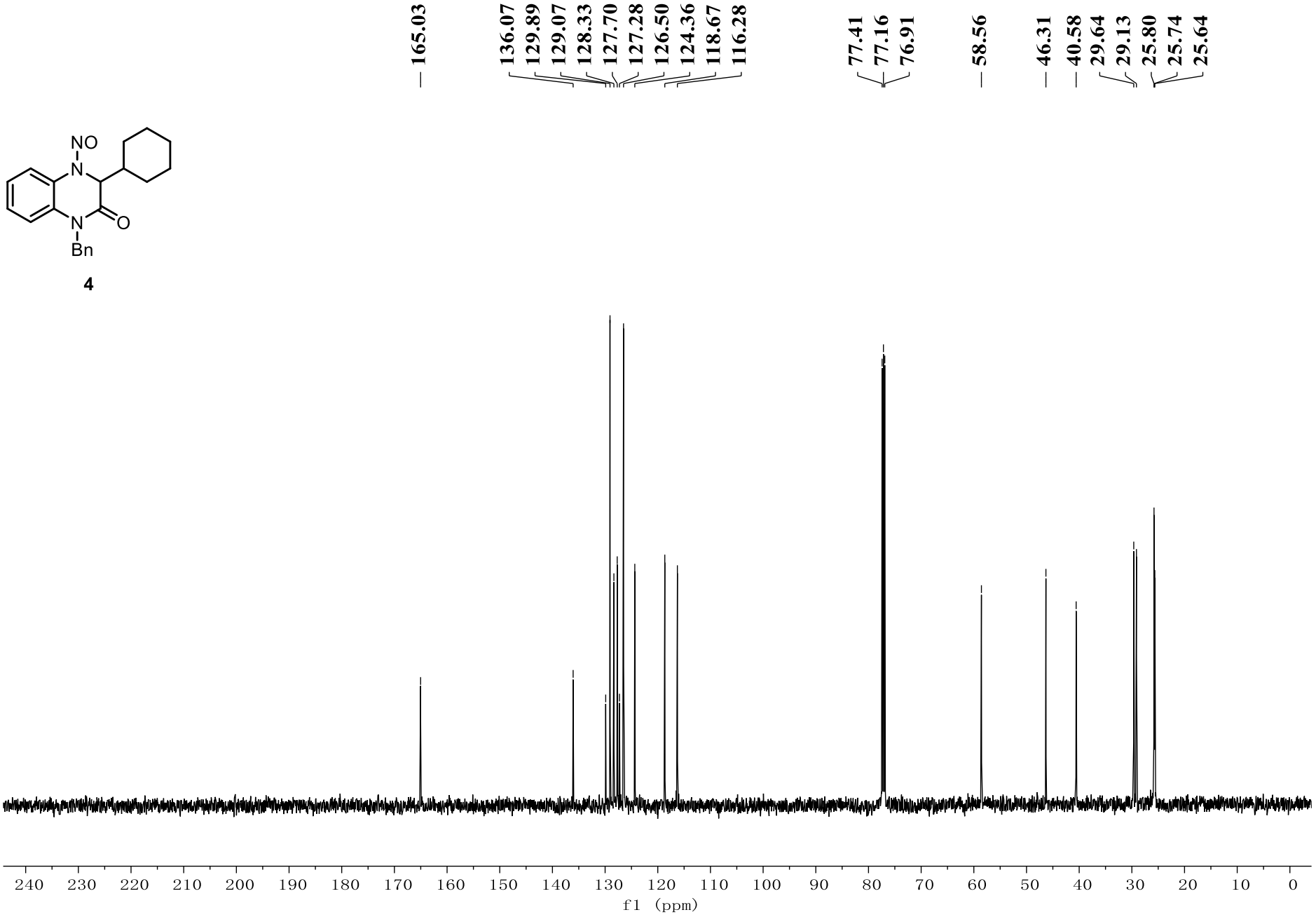


^{1}H NMR (500 MHz, $CDCl_3$) for 4



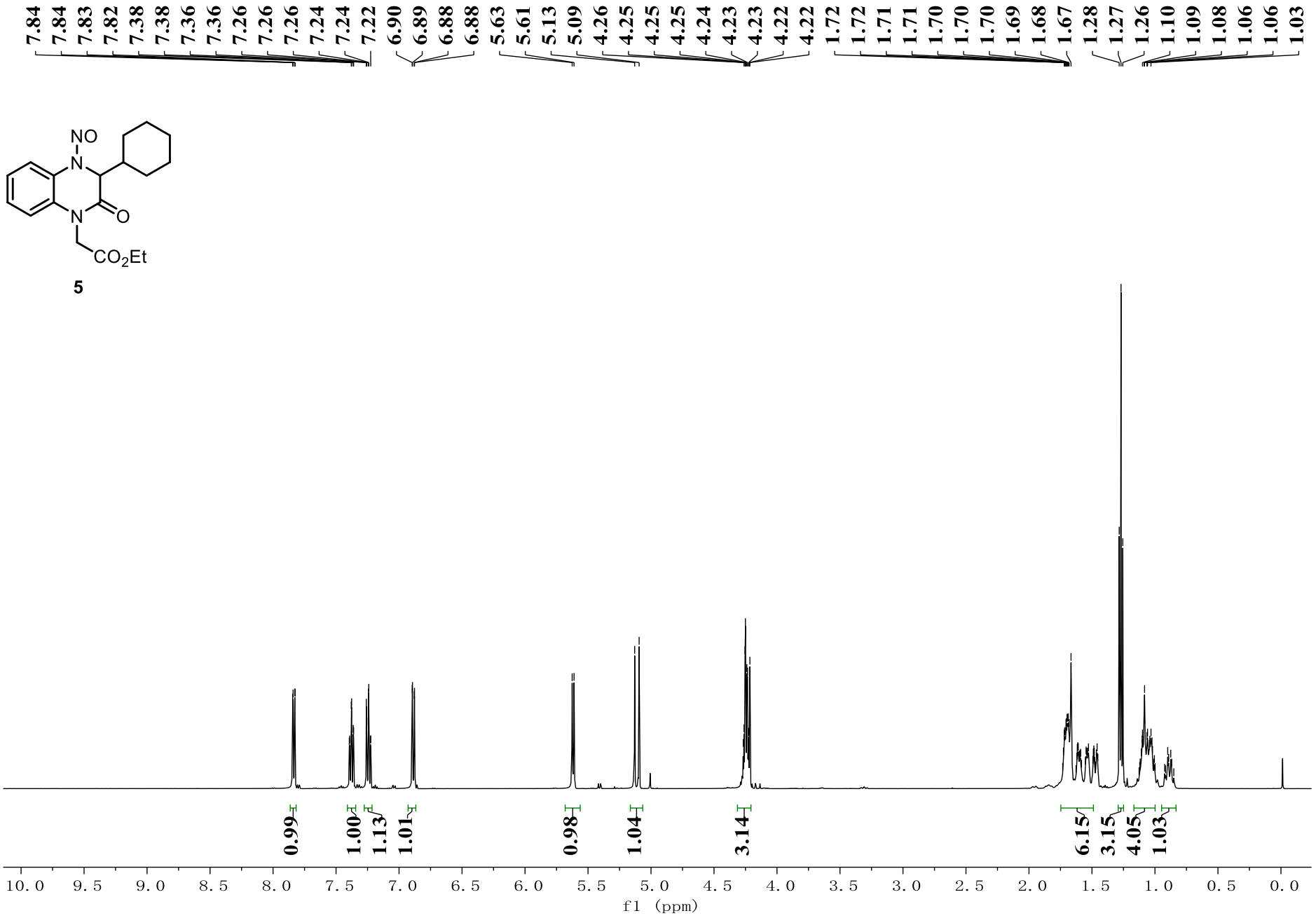
4

^{13}C NMR (125 MHz, $CDCl_3$) for 4

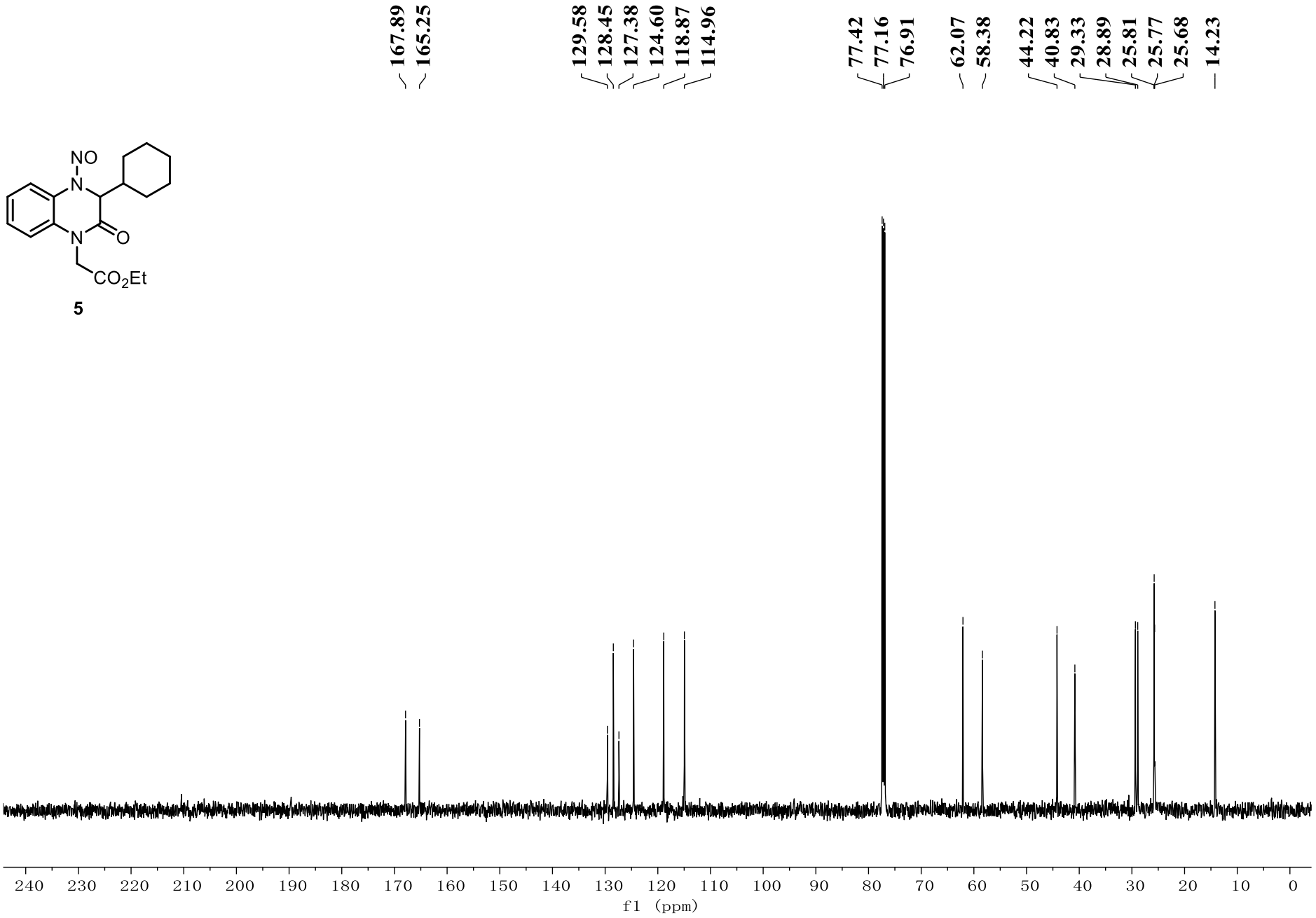


4

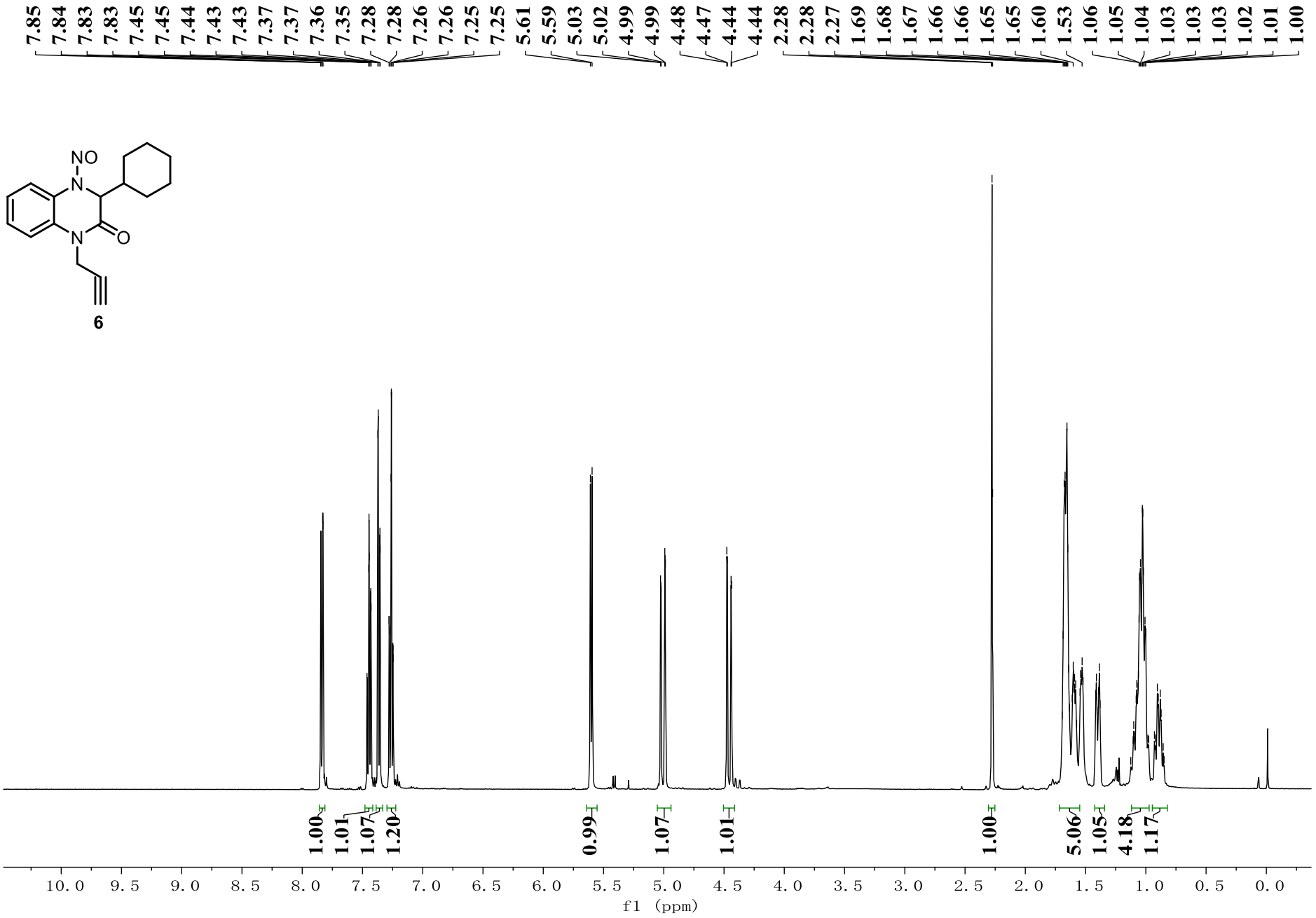
^{1}H NMR (500 MHz, $CDCl_3$) for 5



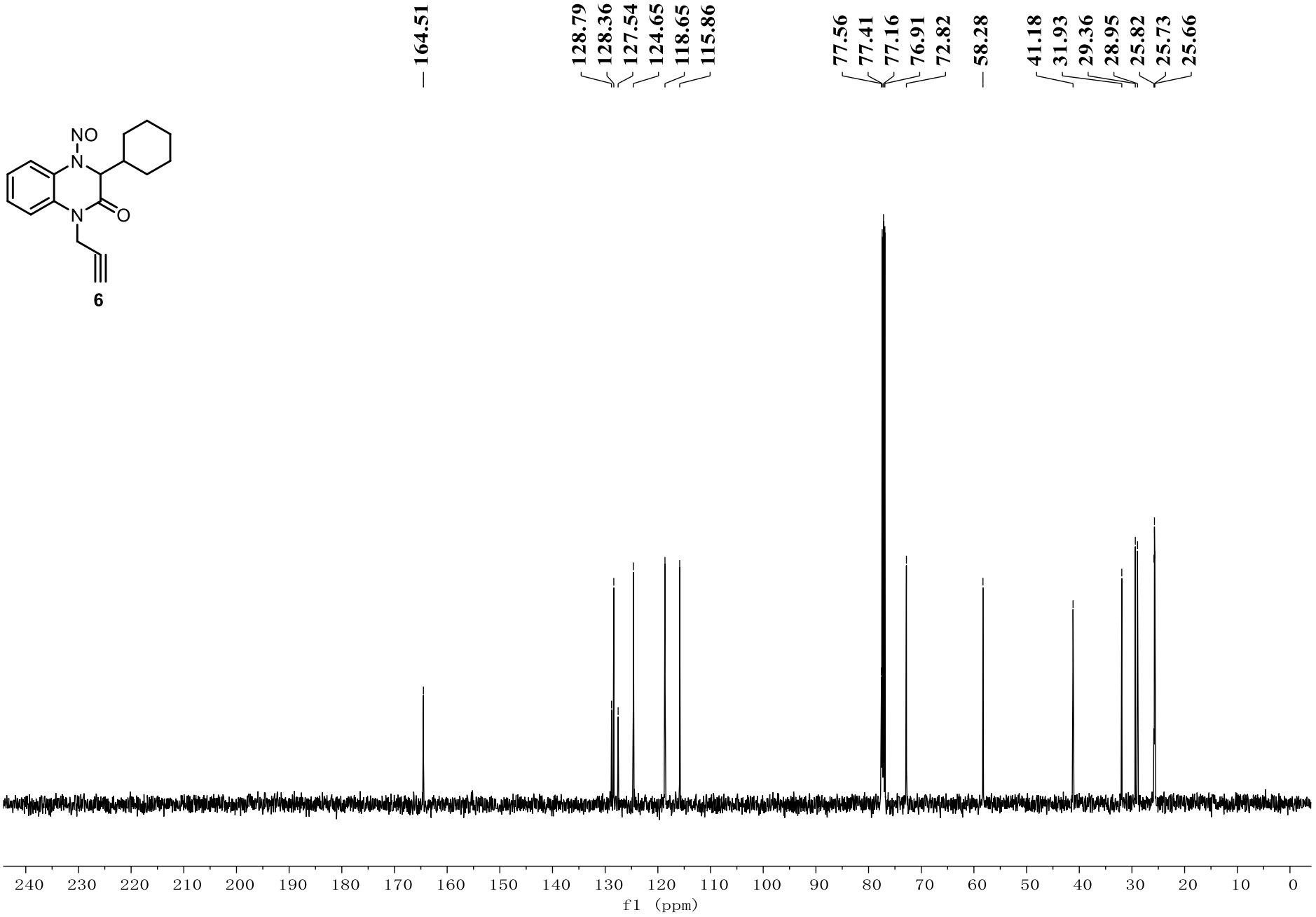
^{13}C NMR (125 MHz, $CDCl_3$) for 5



^{1}H NMR (500 MHz, $CDCl_3$) for 6

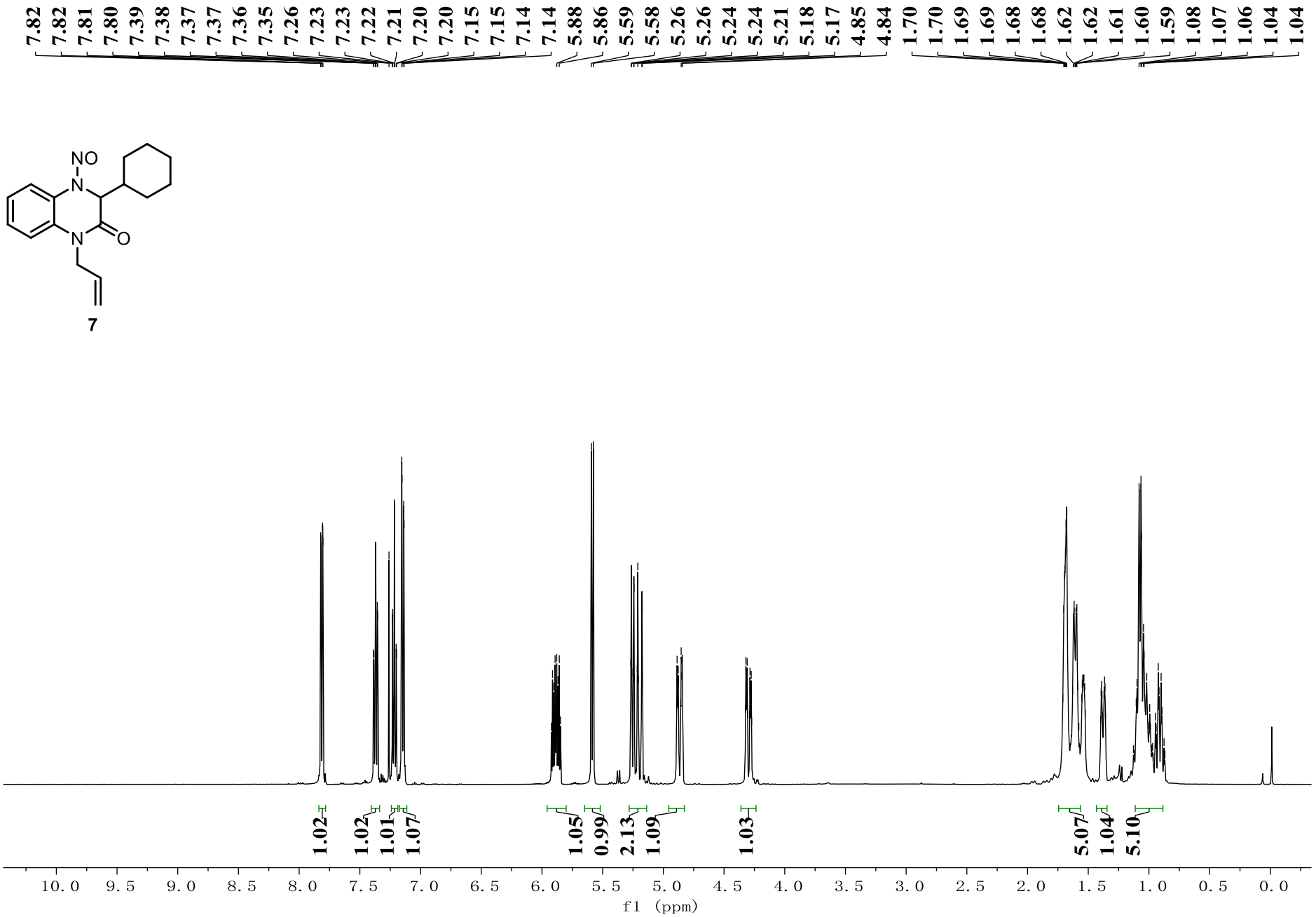


^{13}C NMR (125 MHz, $CDCl_3$) for 6



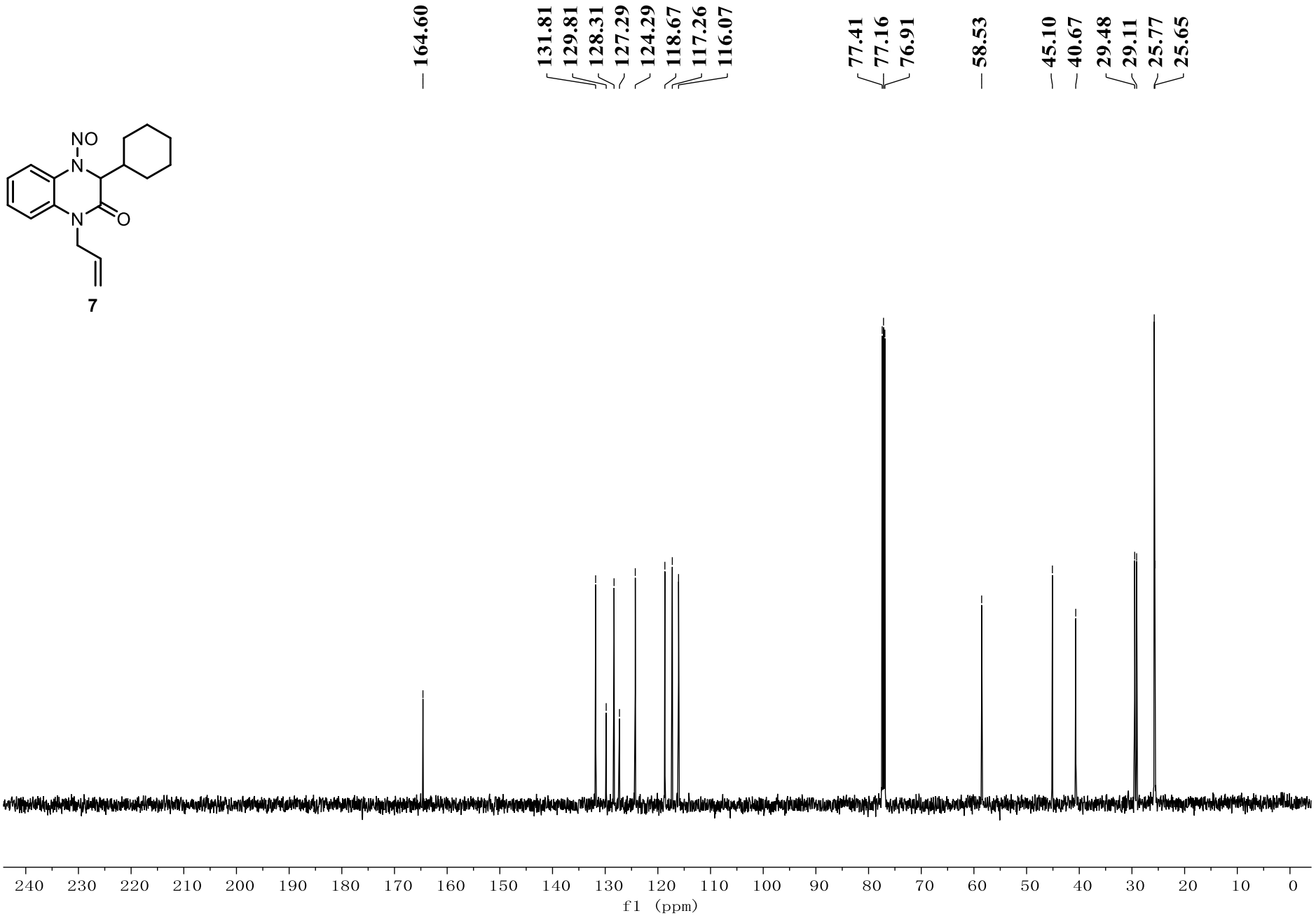
6

^{1}H NMR (500 MHz, $CDCl_3$) for 7

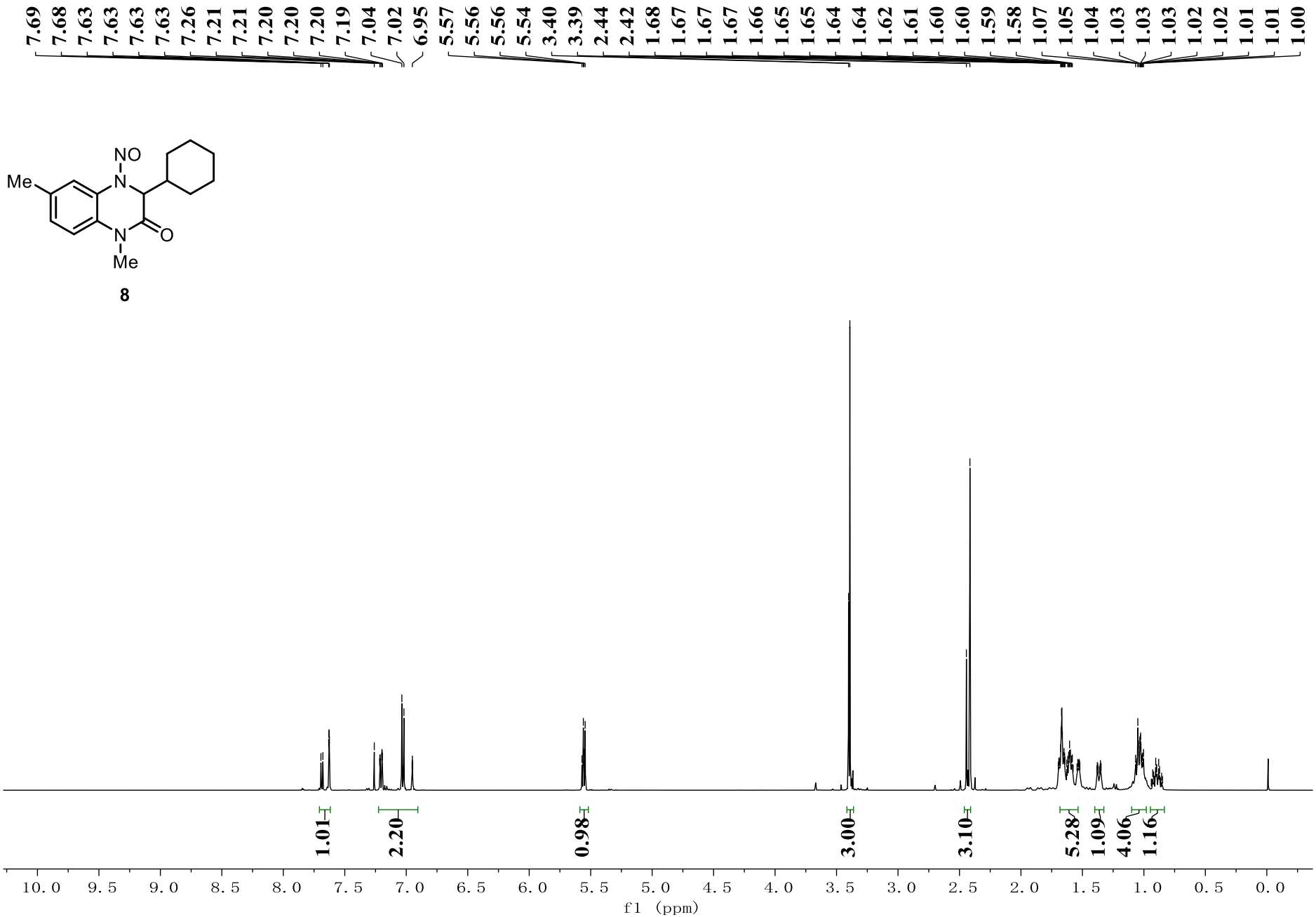


7

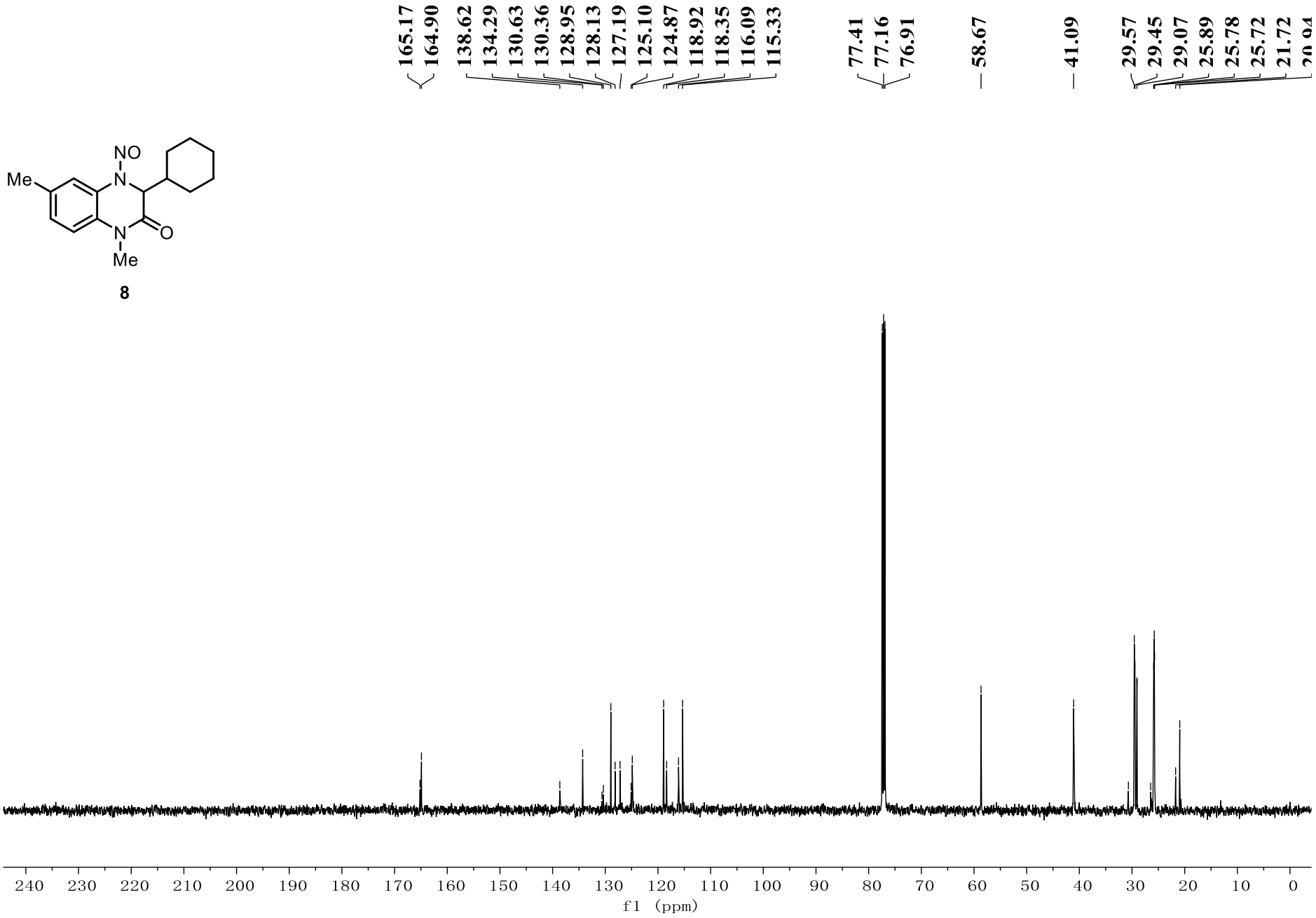
^{13}C NMR (125 MHz, $CDCl_3$) for 7



^{1}H NMR (500 MHz, $CDCl_3$) for 8

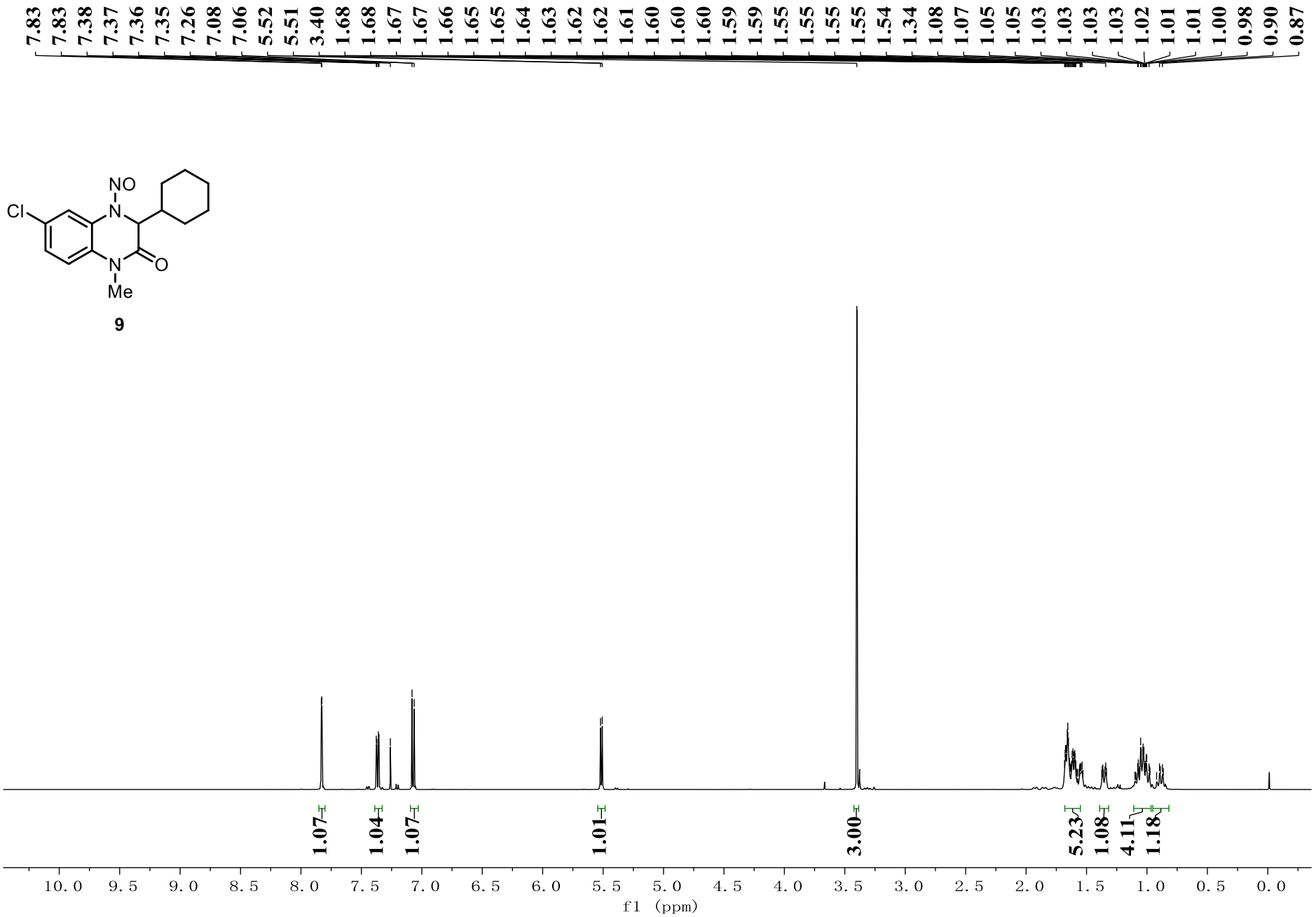


^{13}C NMR (125 MHz, $CDCl_3$) for 8

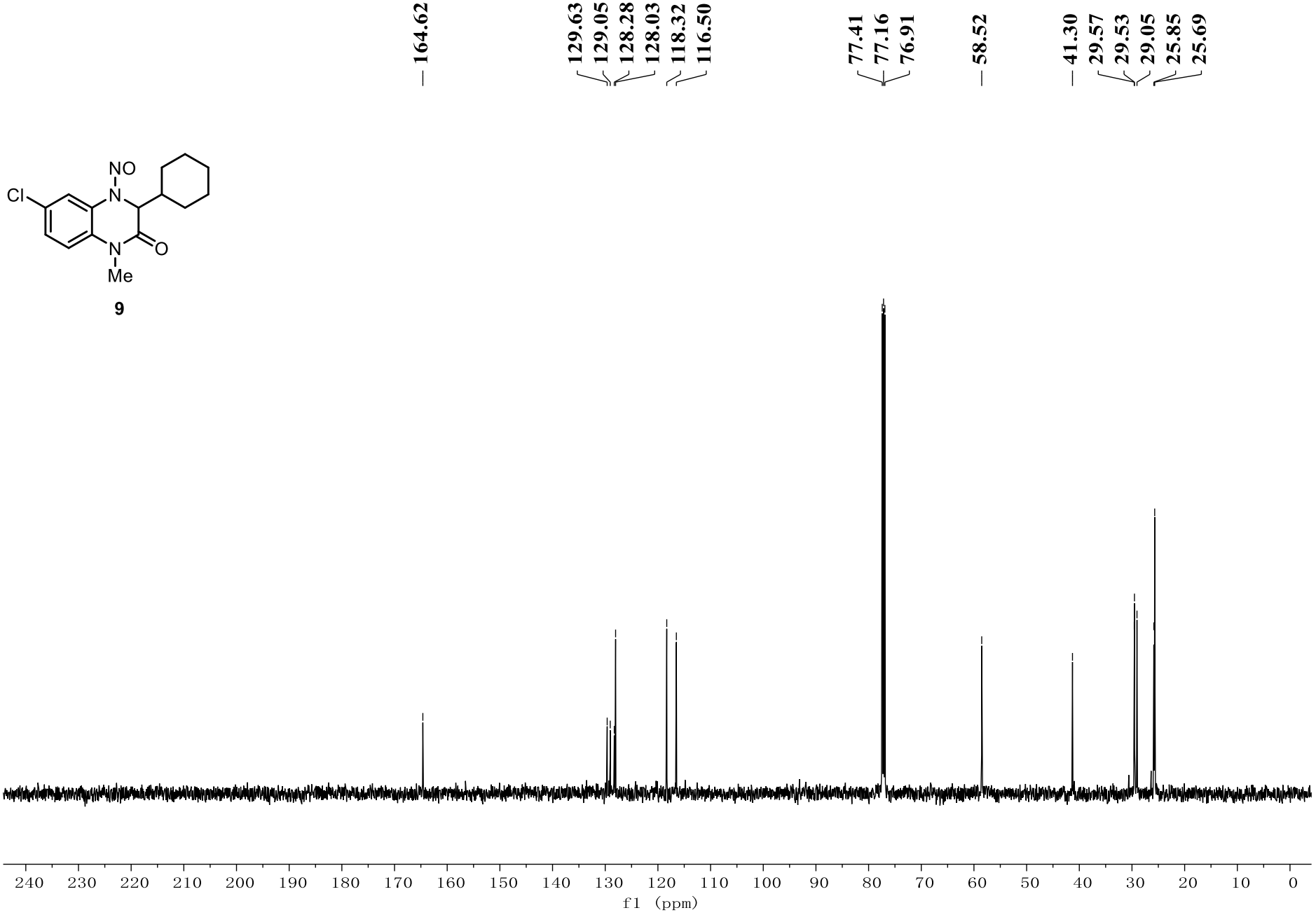


8

^{1}H NMR (500 MHz, $CDCl_3$) for 9

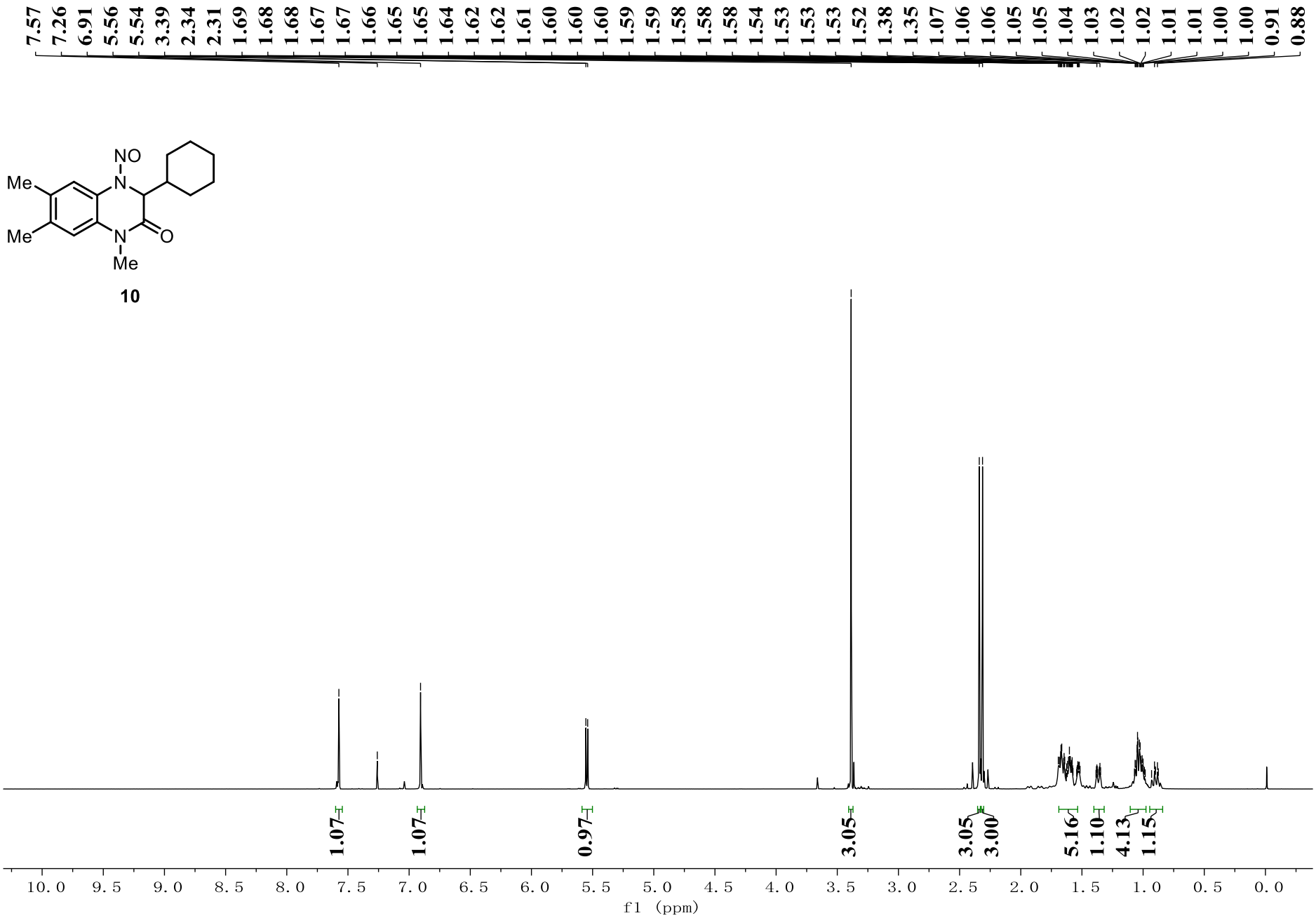


^{13}C NMR (125 MHz, $CDCl_3$) for 9

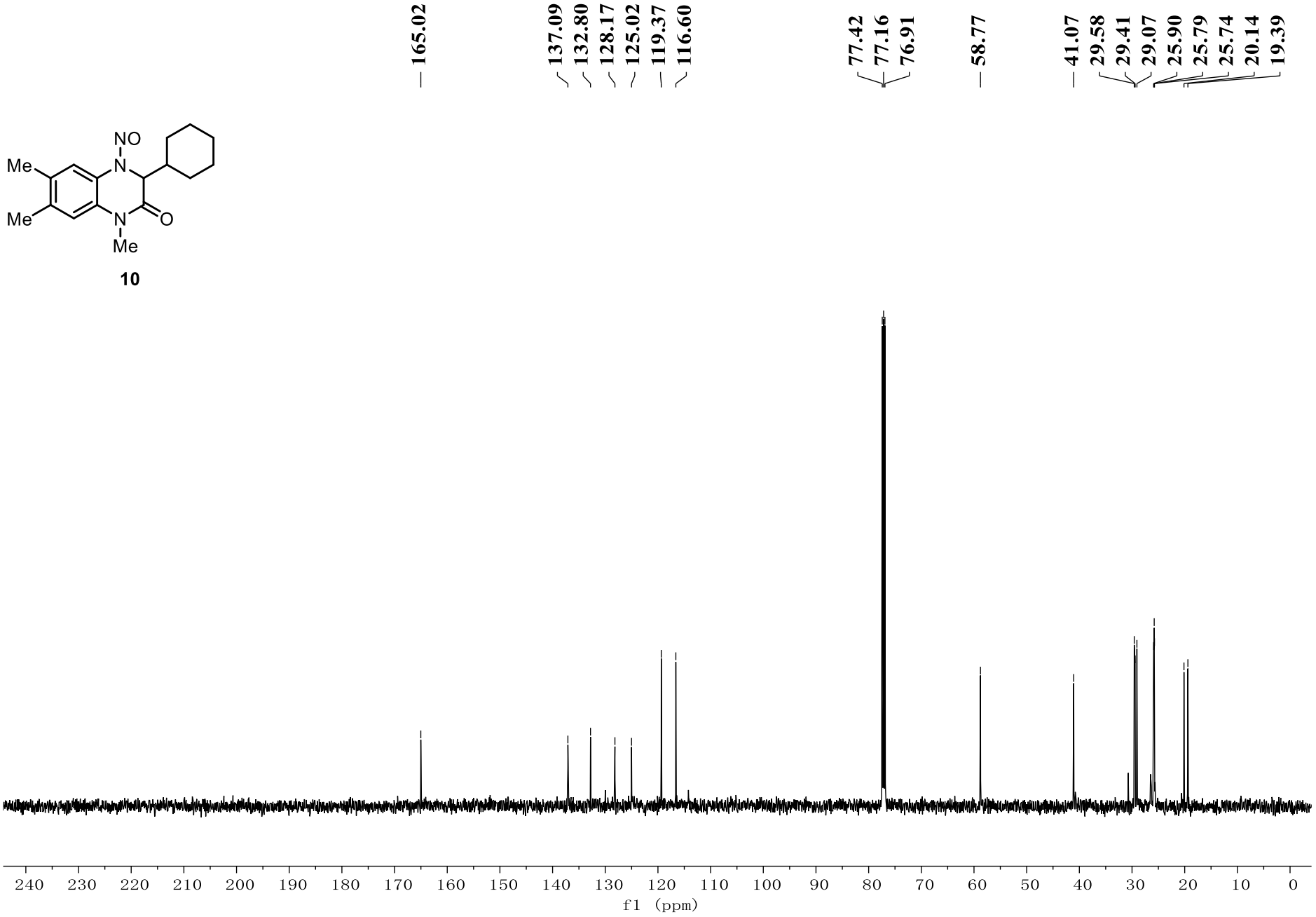


9

^{1}H NMR (500 MHz, $CDCl_3$) for 10

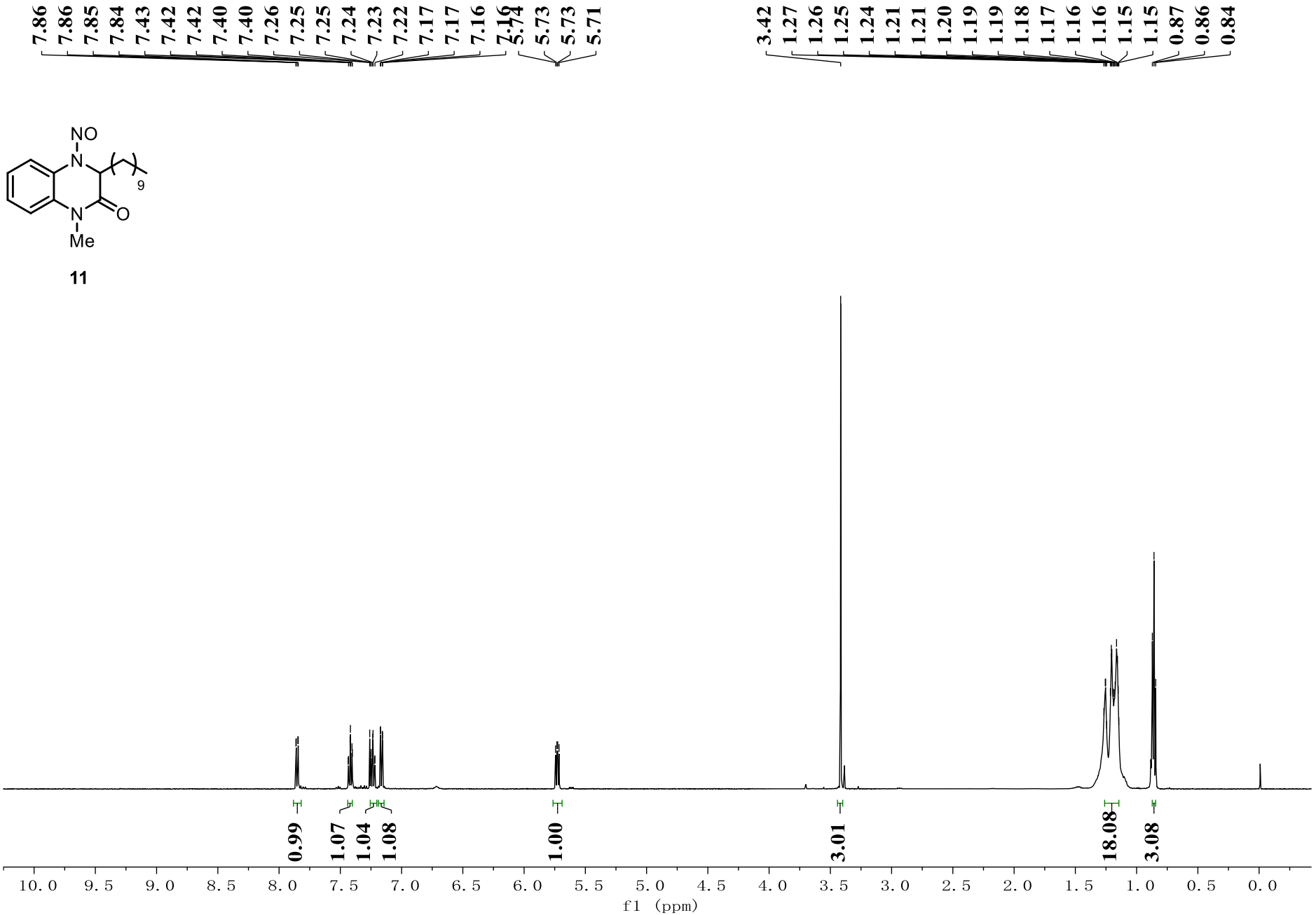


^{13}C NMR (125 MHz, $CDCl_3$) for 10



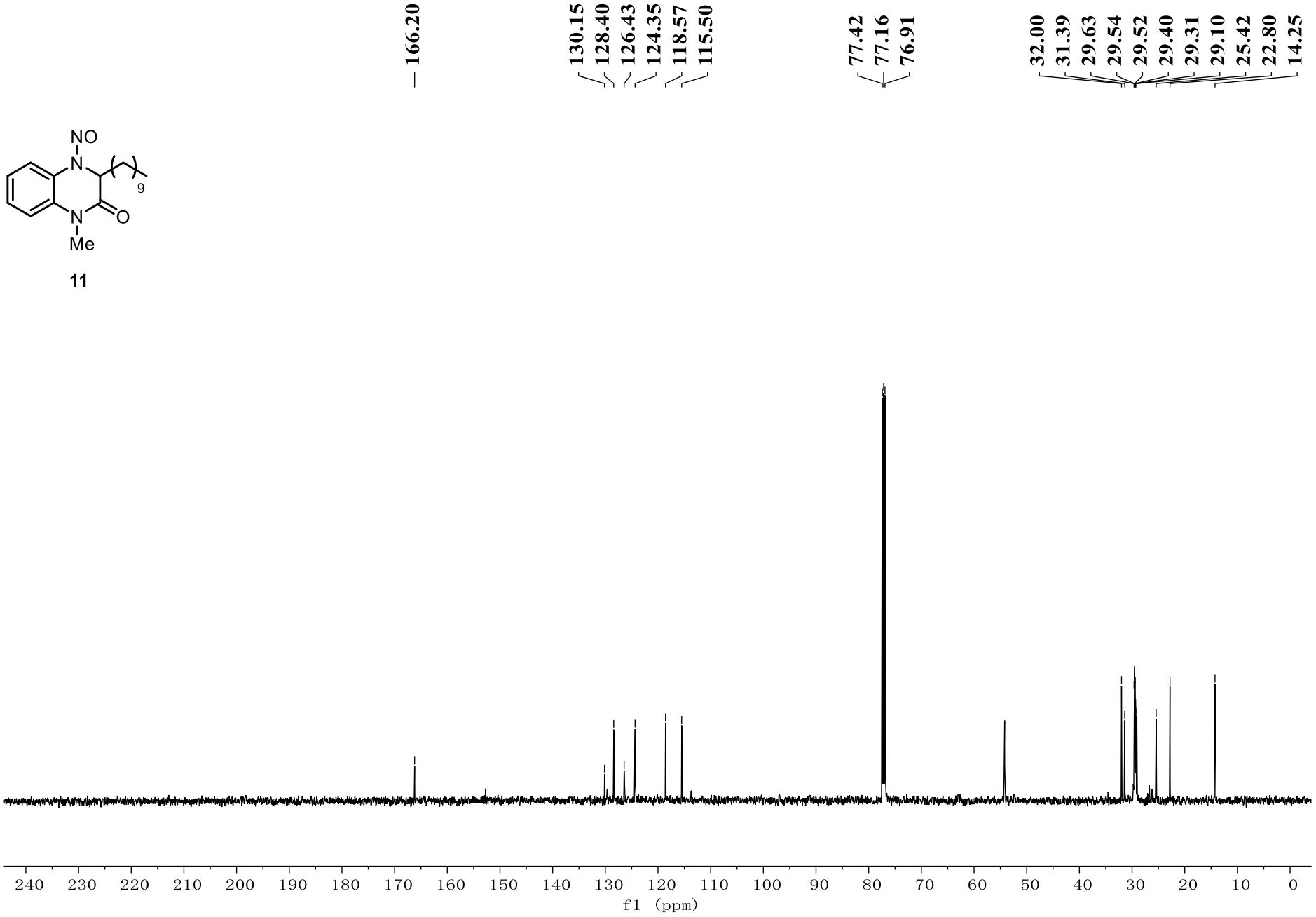
10

^{1}H NMR (500 MHz, $CDCl_3$) for 11



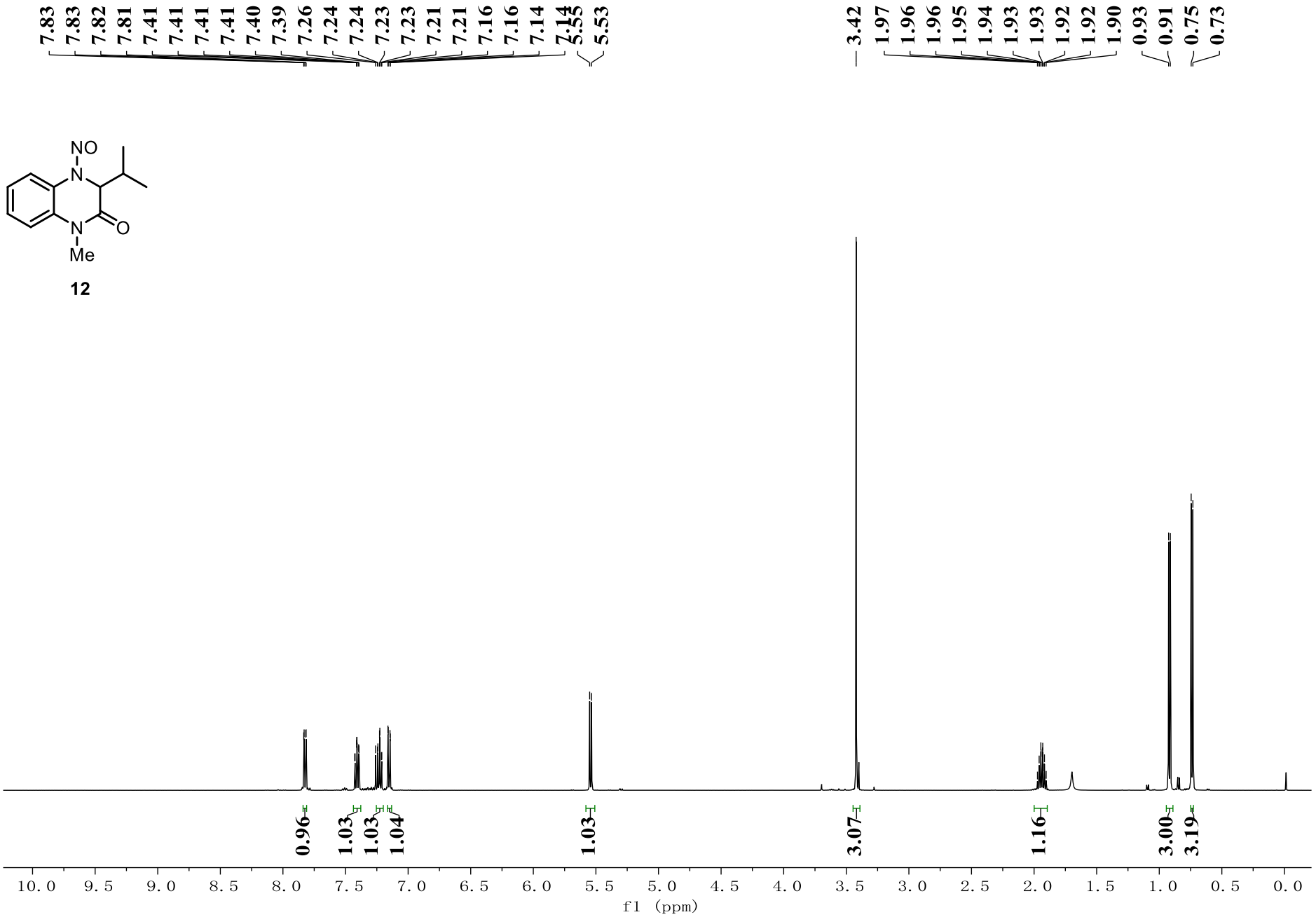
11

^{13}C NMR (125 MHz, $CDCl_3$) for 11



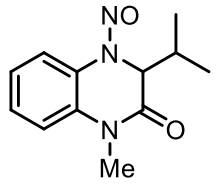
11

^{1}H NMR (500 MHz, $CDCl_3$) for 12



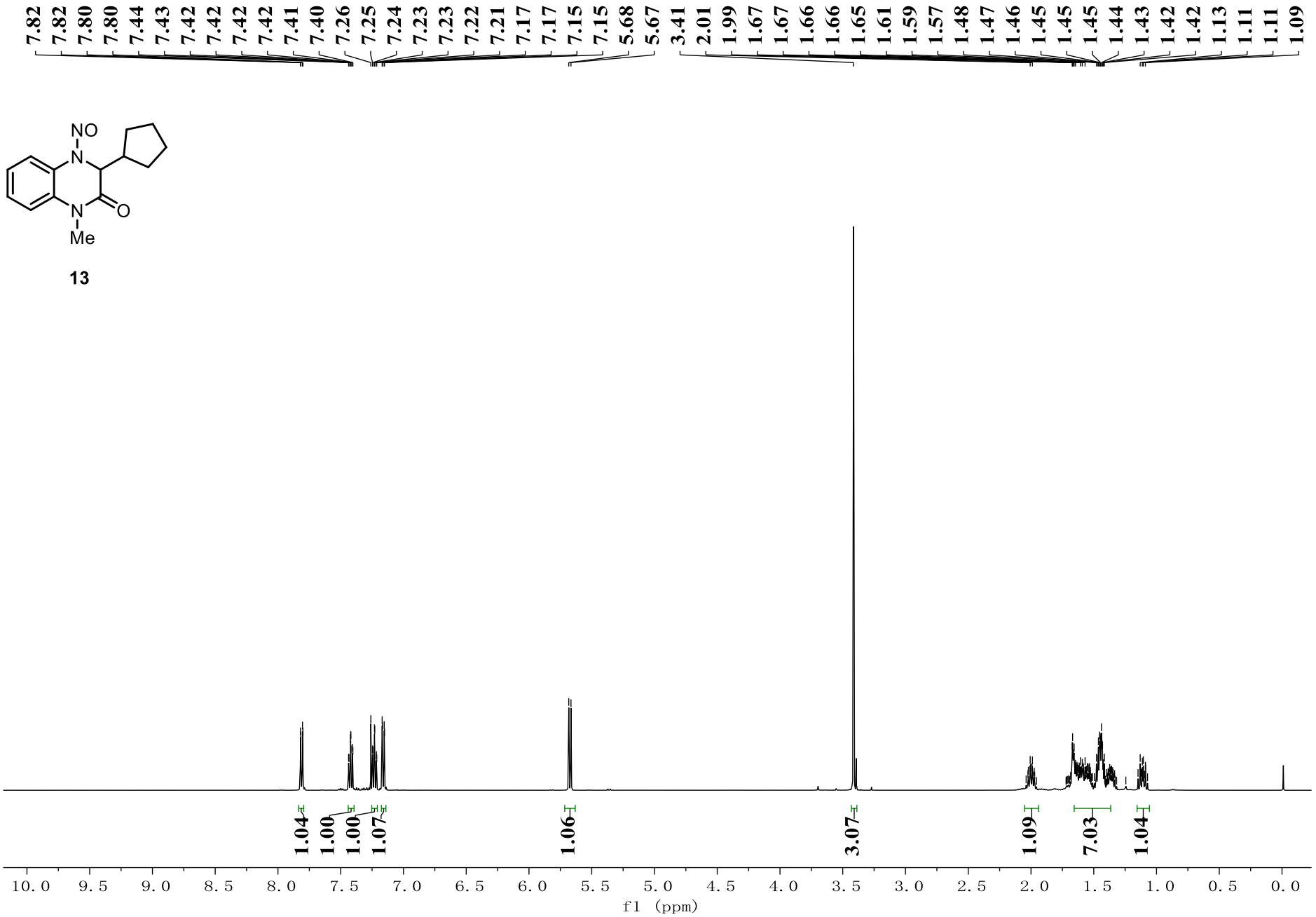
12

^{13}C NMR (125 MHz, $CDCl_3$) for 12

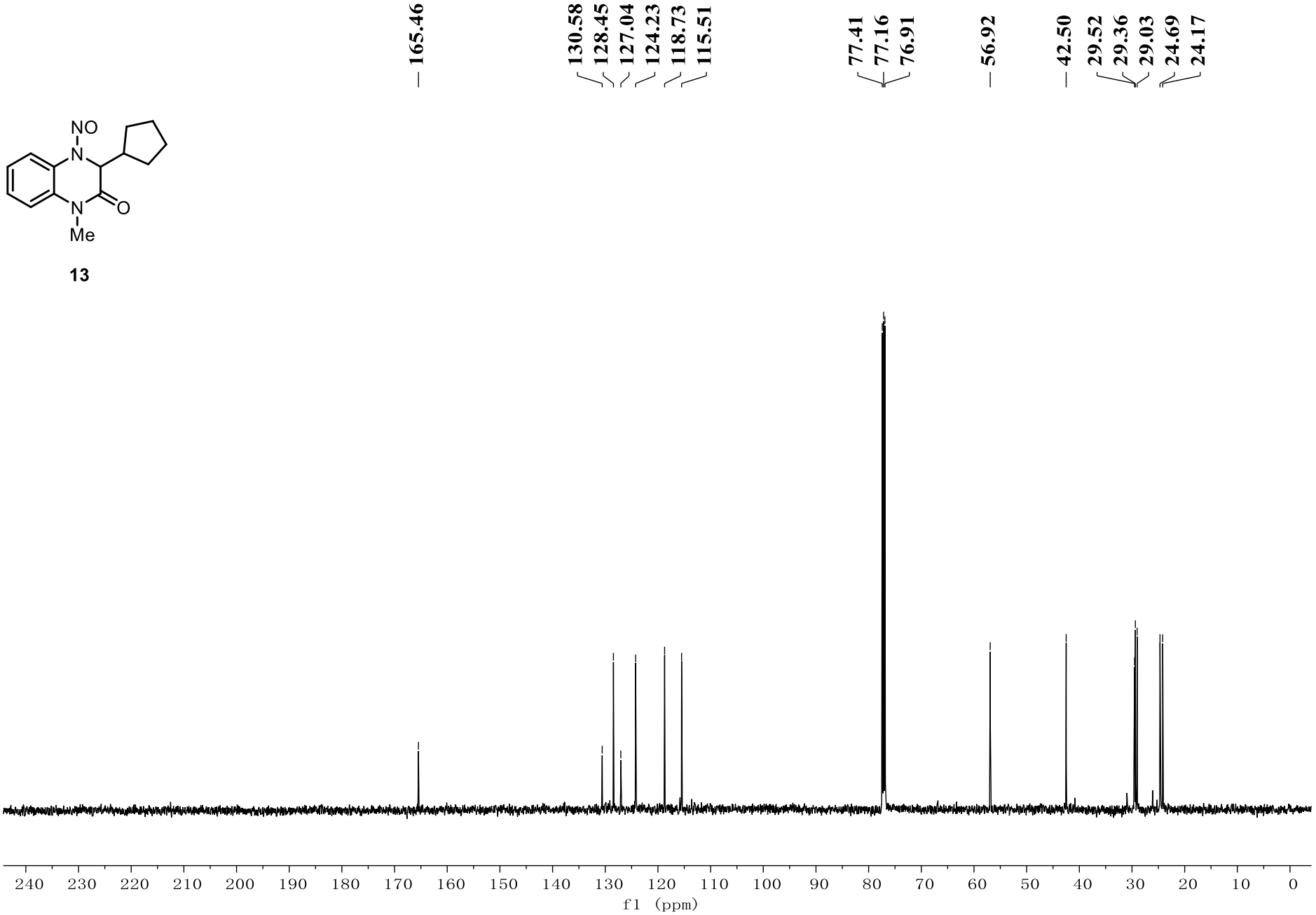


12

^{1}H NMR (500 MHz, $CDCl_3$) for 13

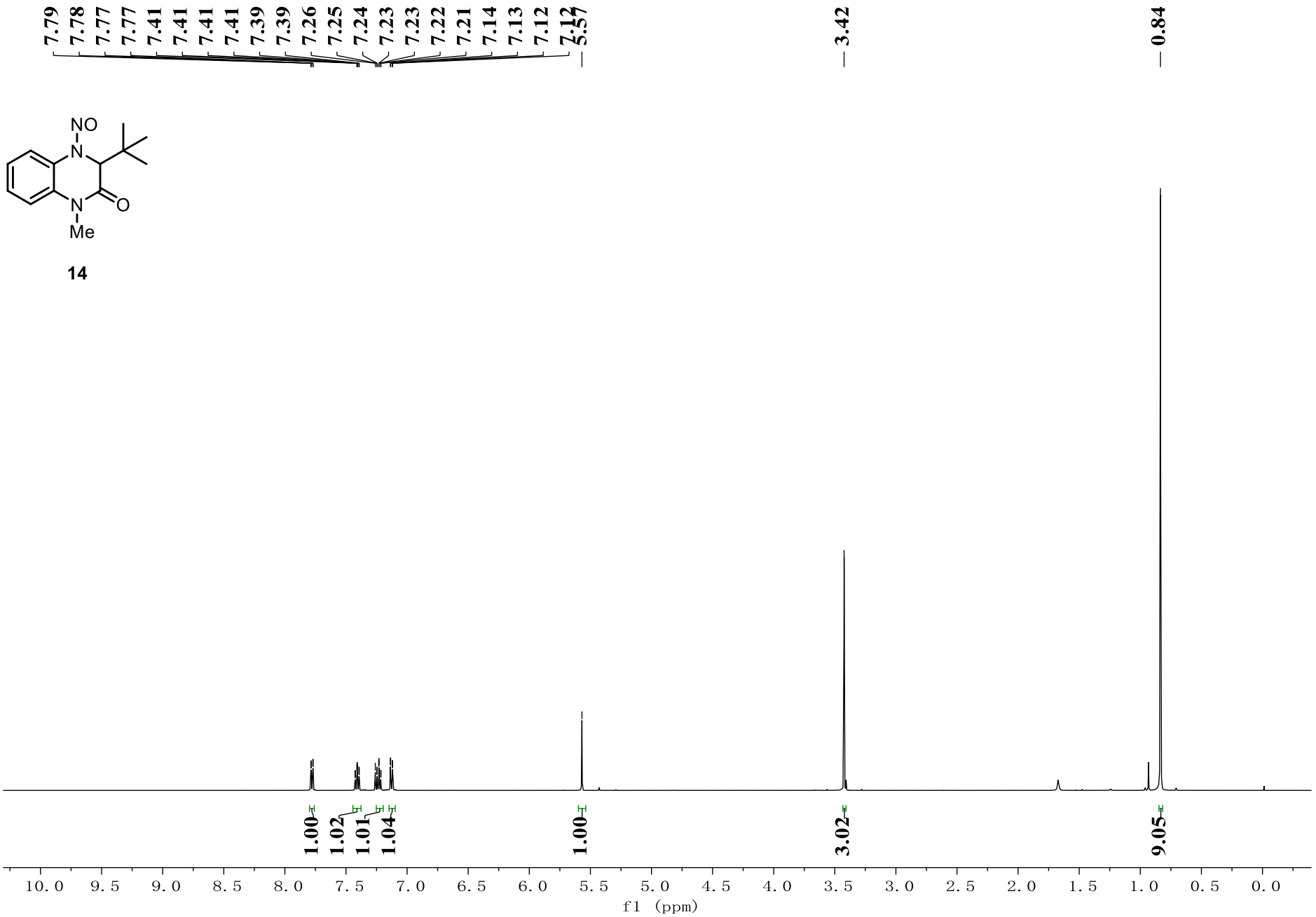


^{13}C NMR (125 MHz, $CDCl_3$) for 13



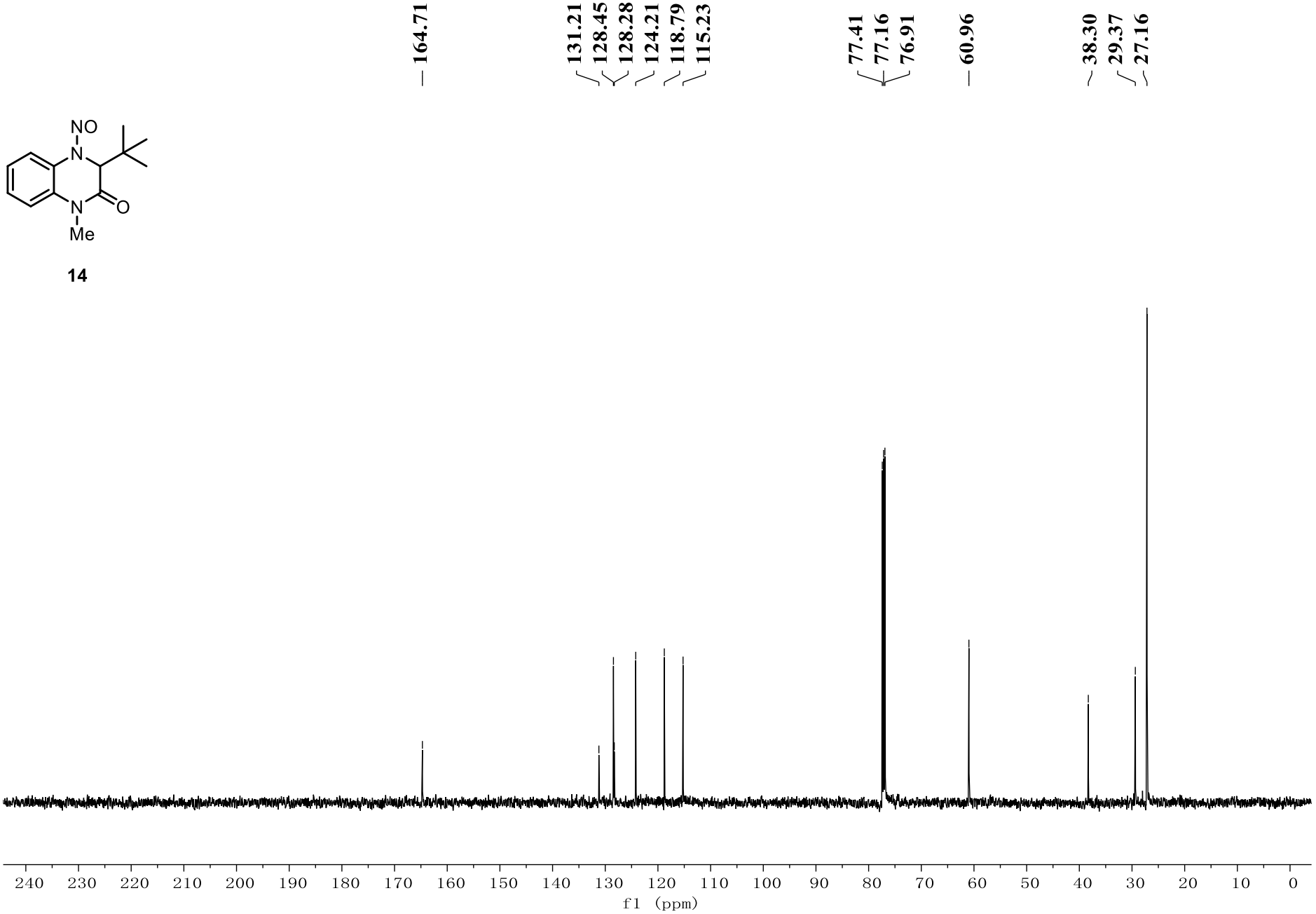
13

^{1}H NMR (500 MHz, $CDCl_3$) for 14



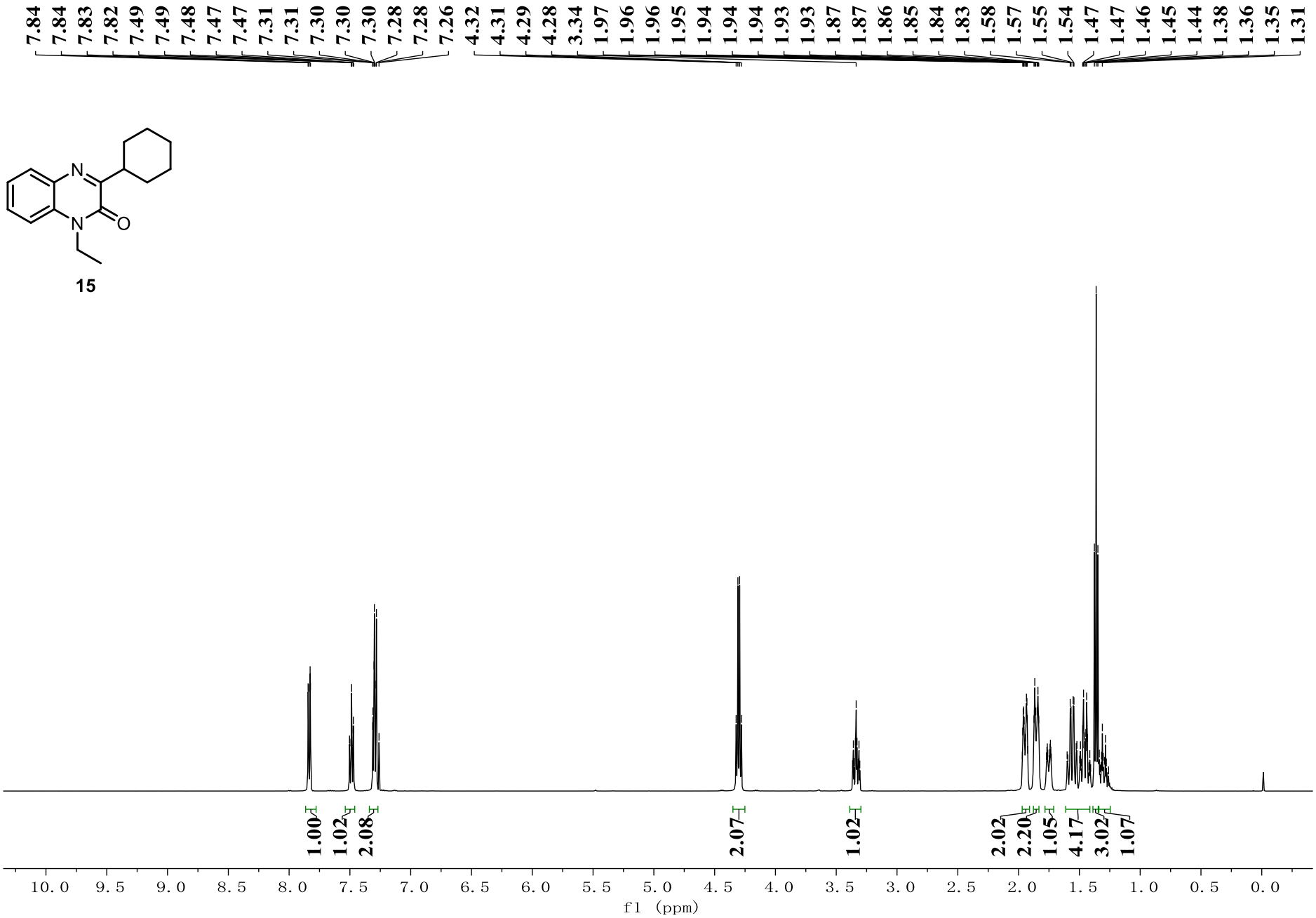
14

^{13}C NMR (125 MHz, $CDCl_3$) for 14

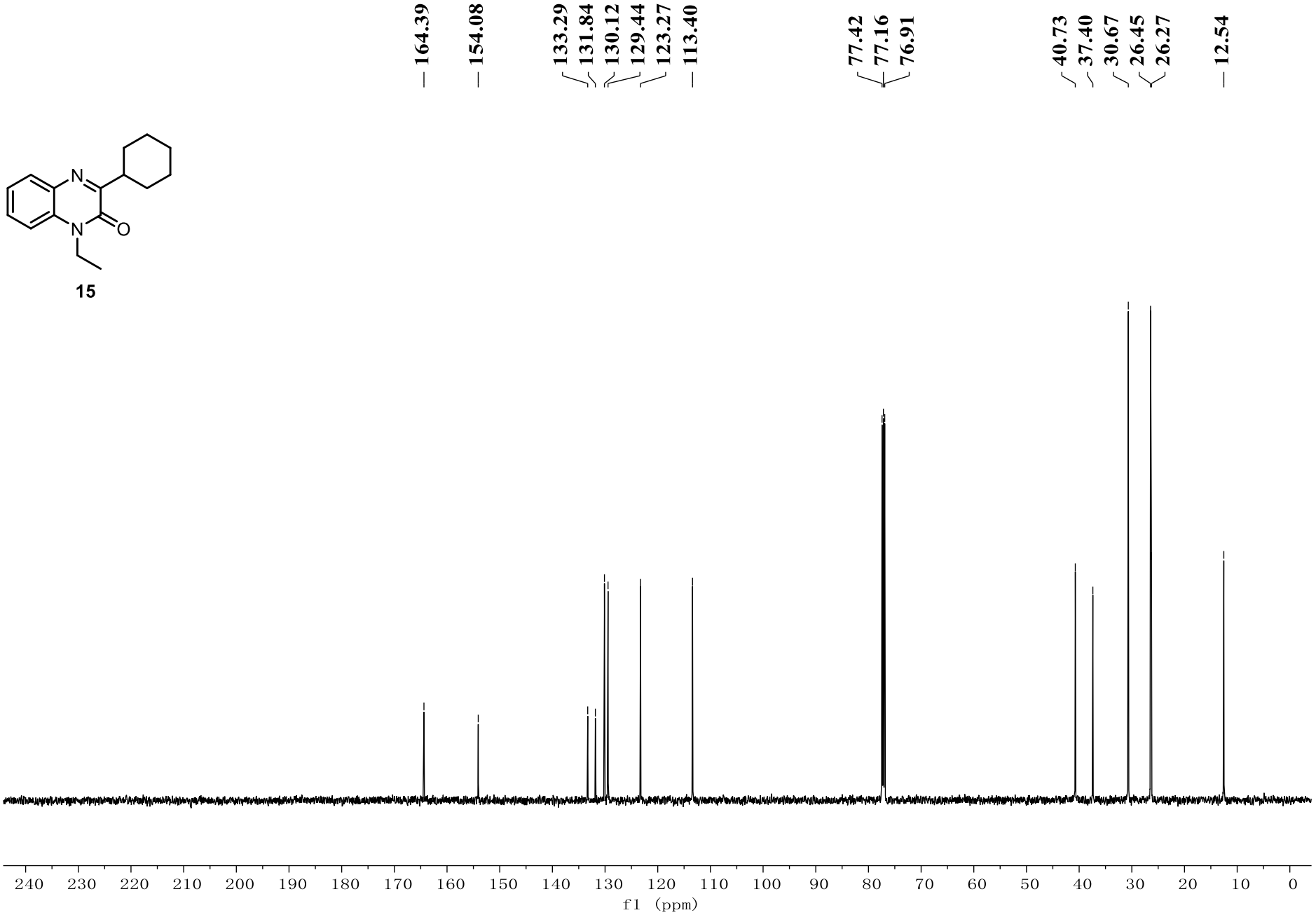


14

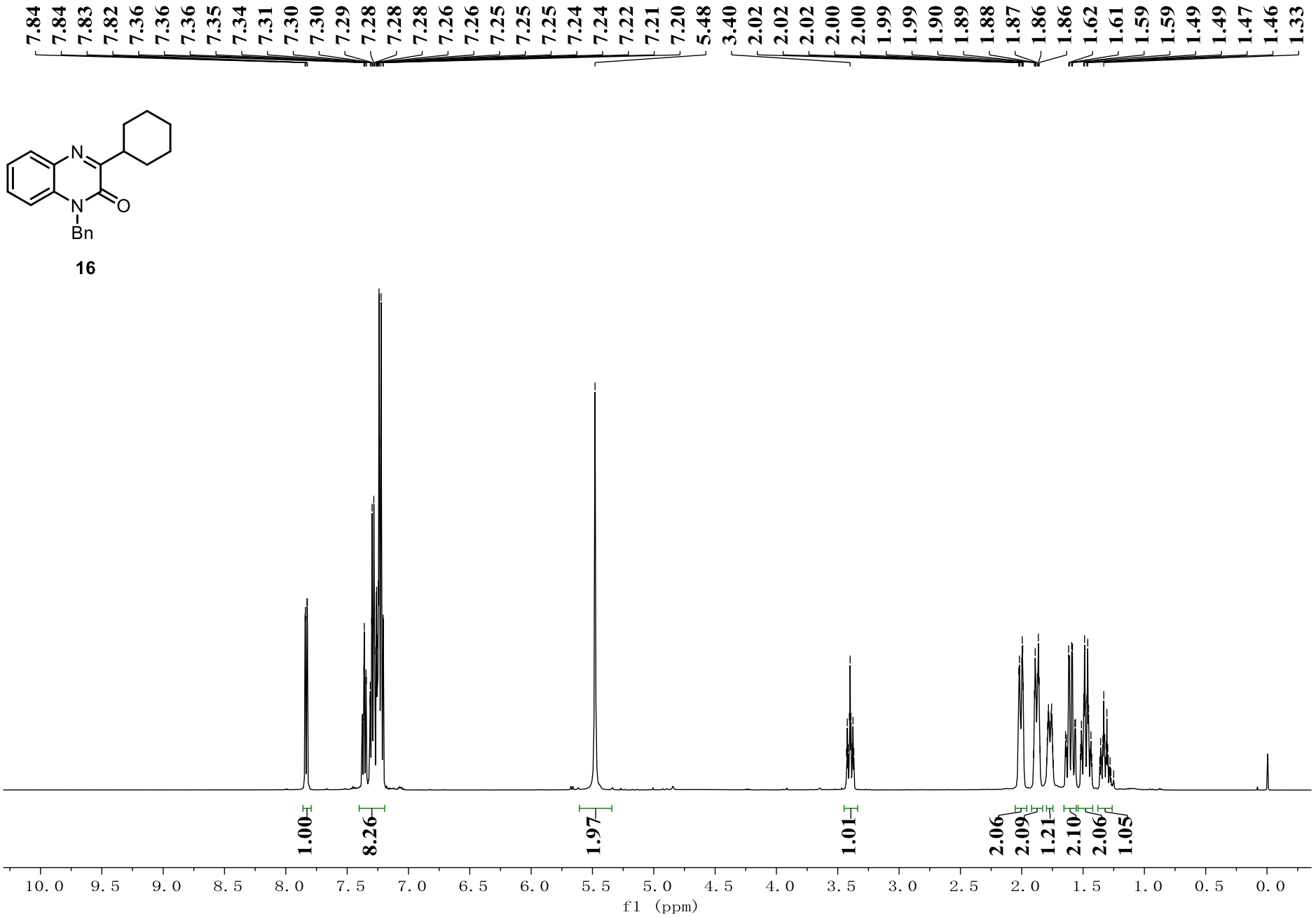
^{1}H NMR (500 MHz, $CDCl_3$) for 15



^{13}C NMR (125 MHz, $CDCl_3$) for 15

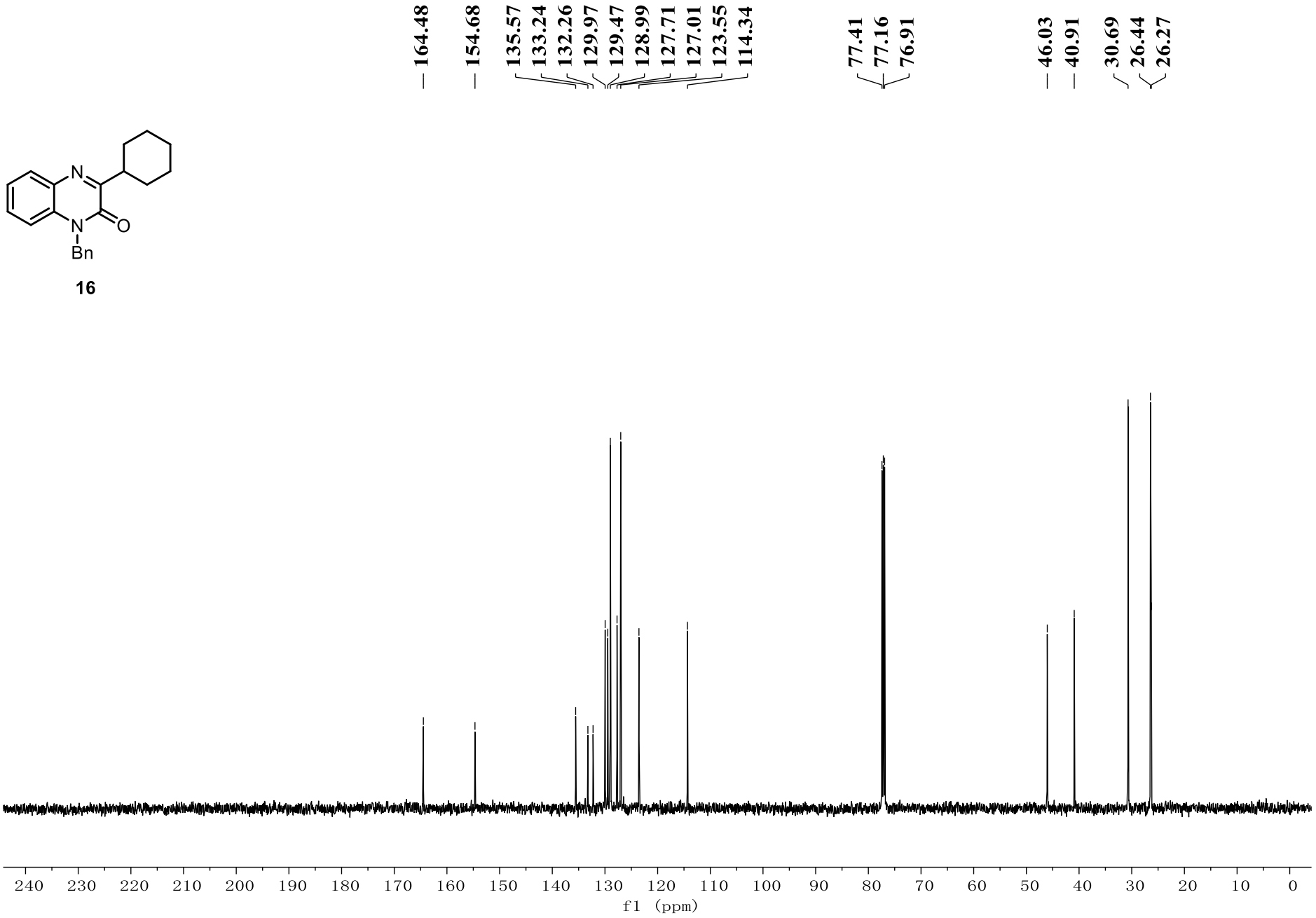


^{1}H NMR (500 MHz, $CDCl_3$) for 16

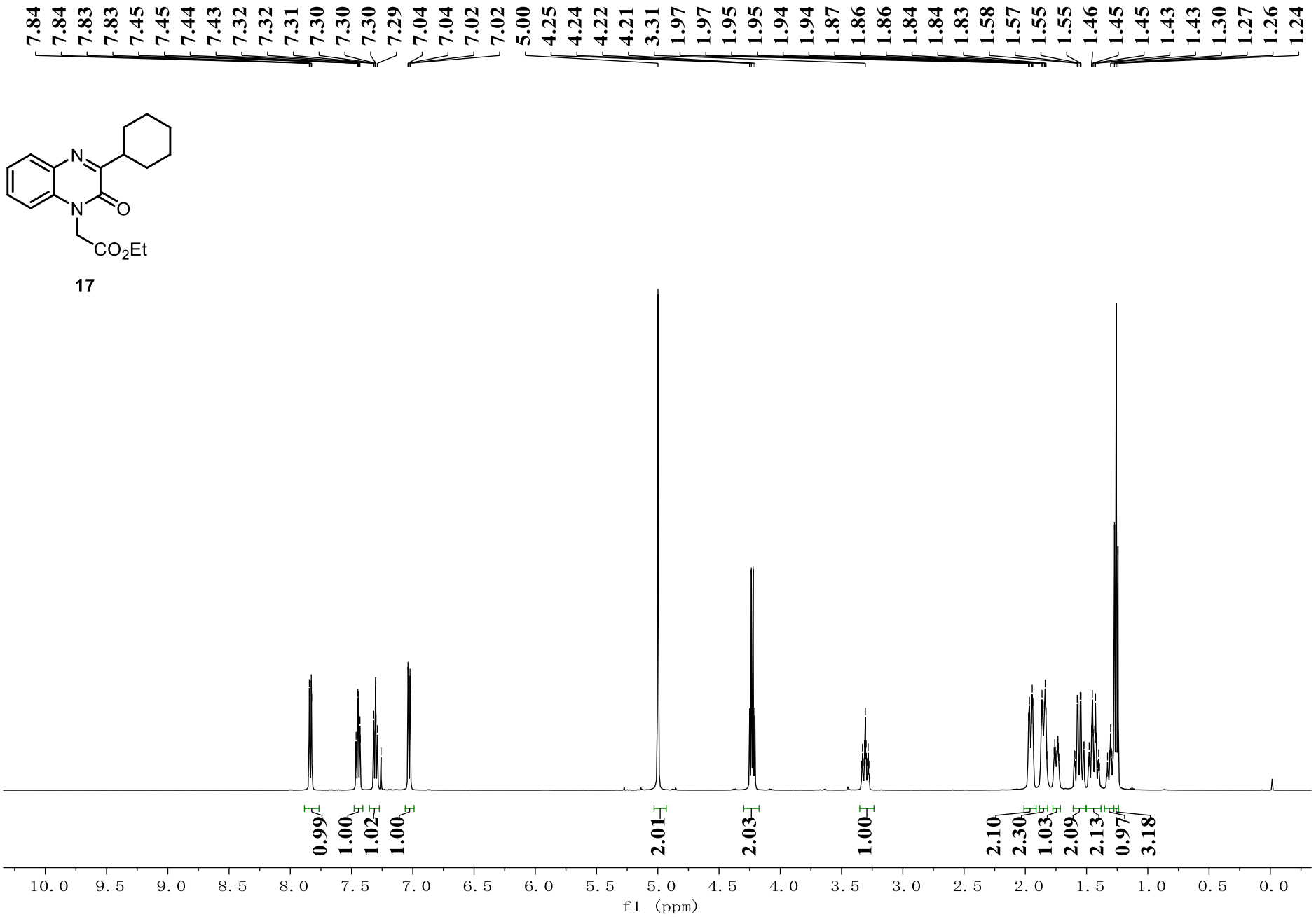


16

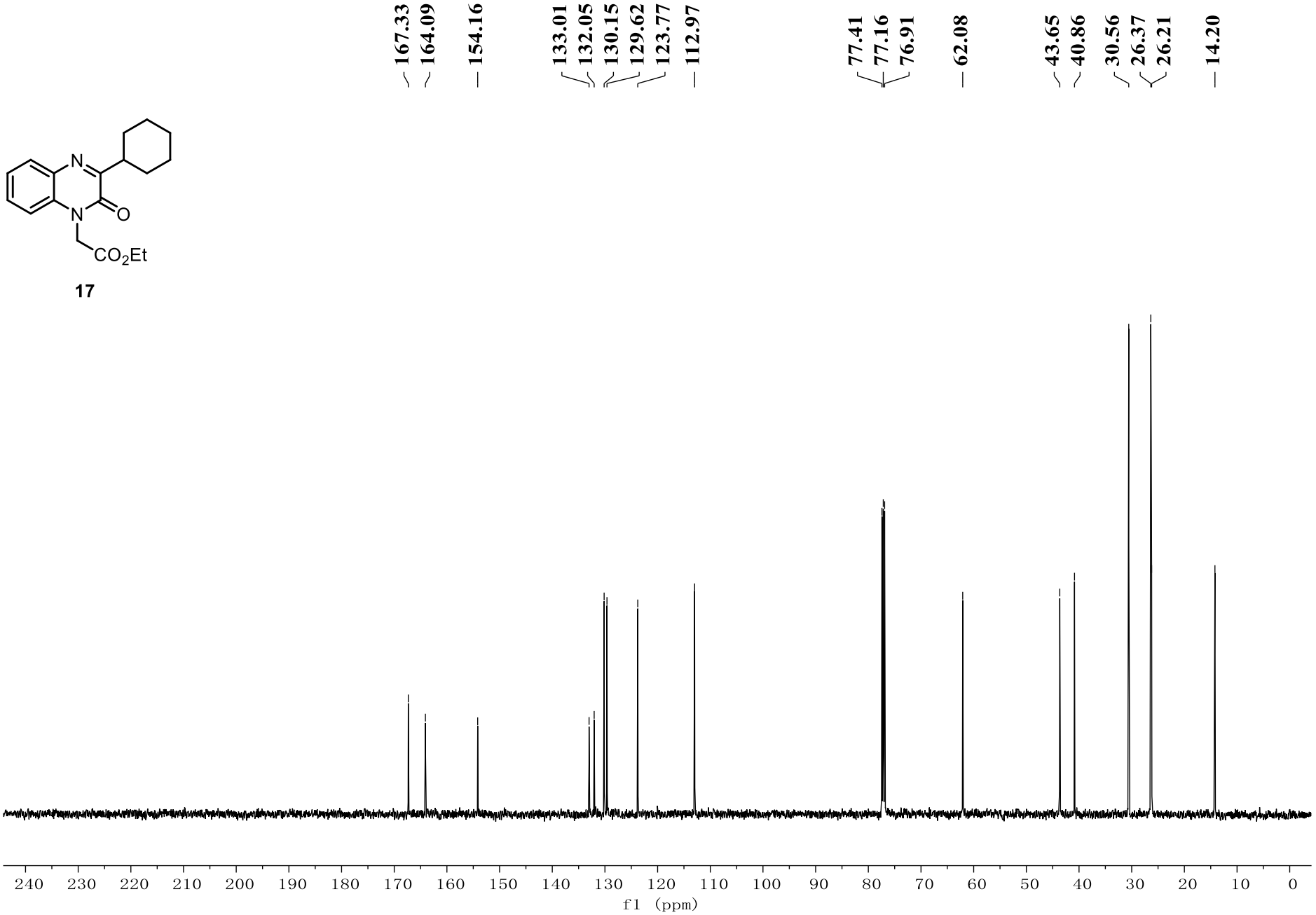
^{13}C NMR (125 MHz, $CDCl_3$) for 16



^{1}H NMR (500 MHz, $CDCl_3$) for 17

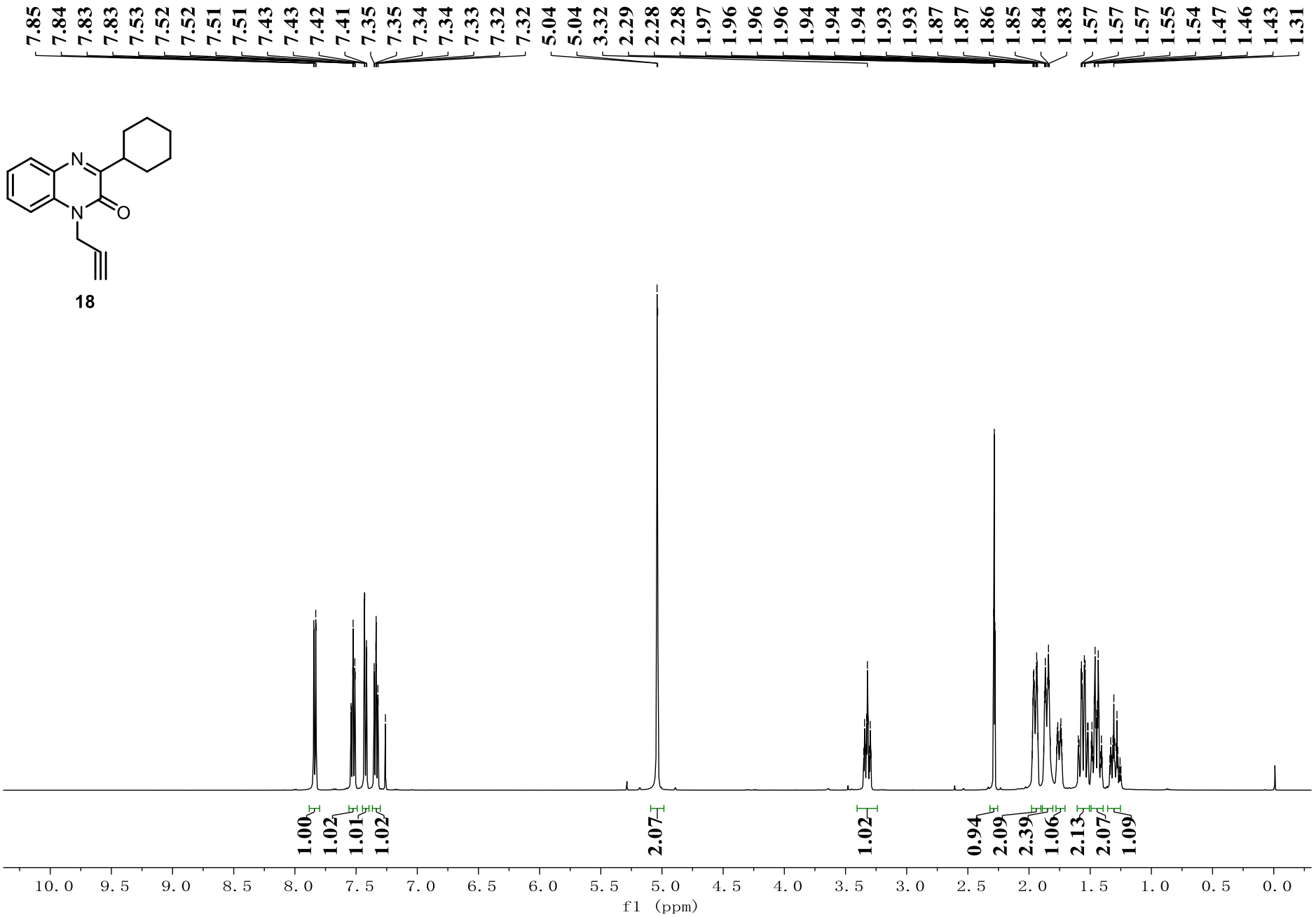


^{13}C NMR (125 MHz, $CDCl_3$) for 17

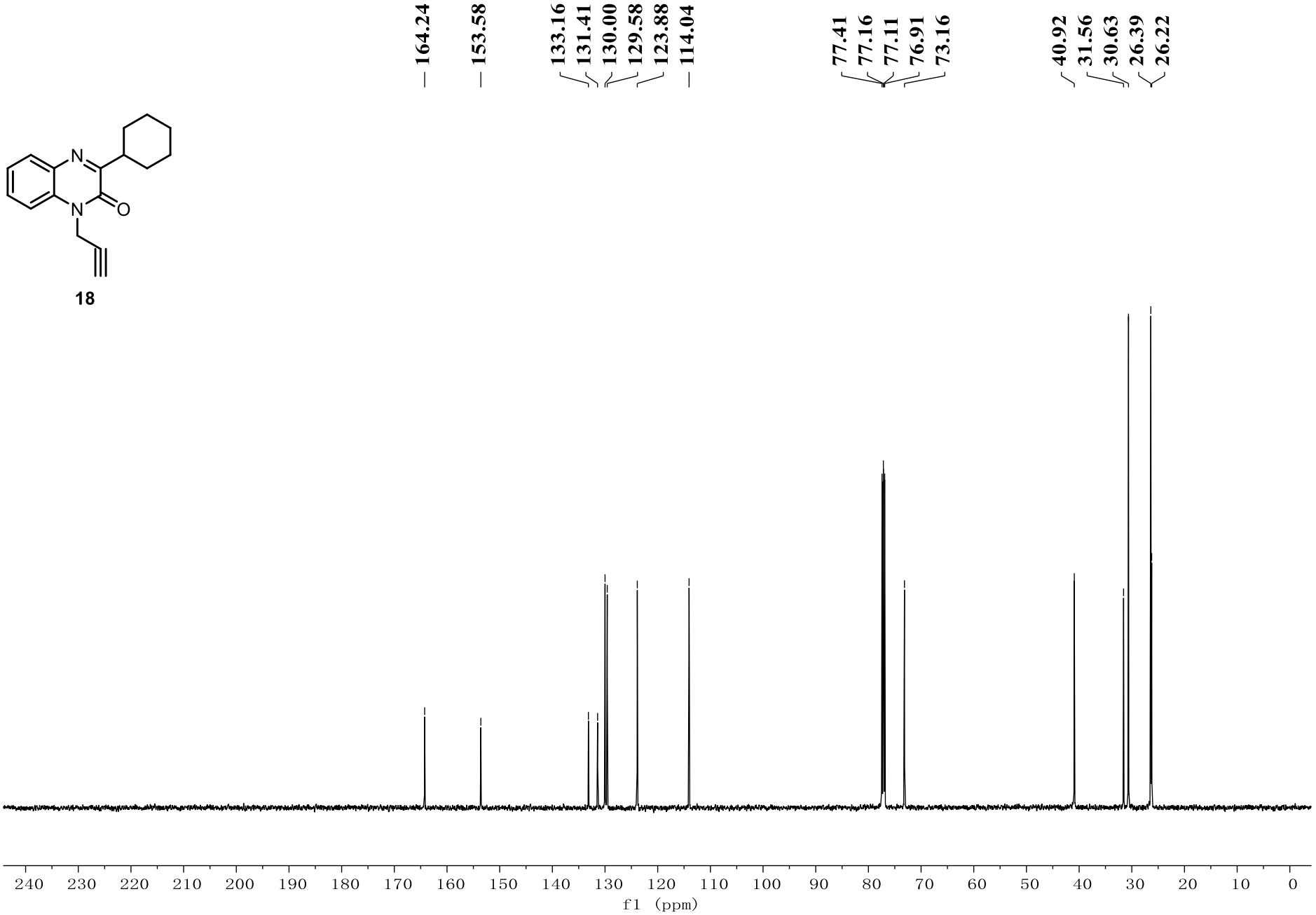


17

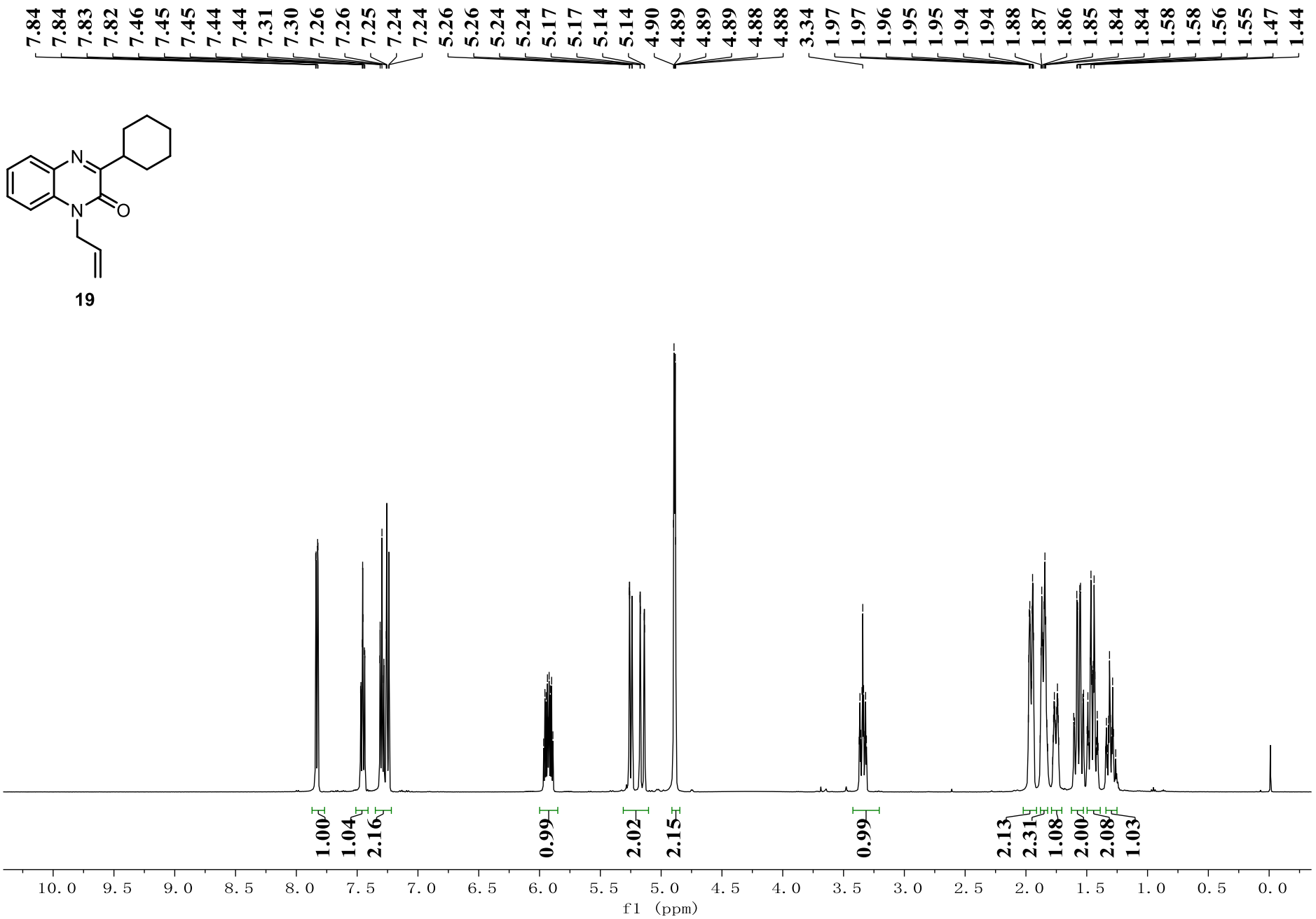
^{1}H NMR (500 MHz, $CDCl_3$) for 18



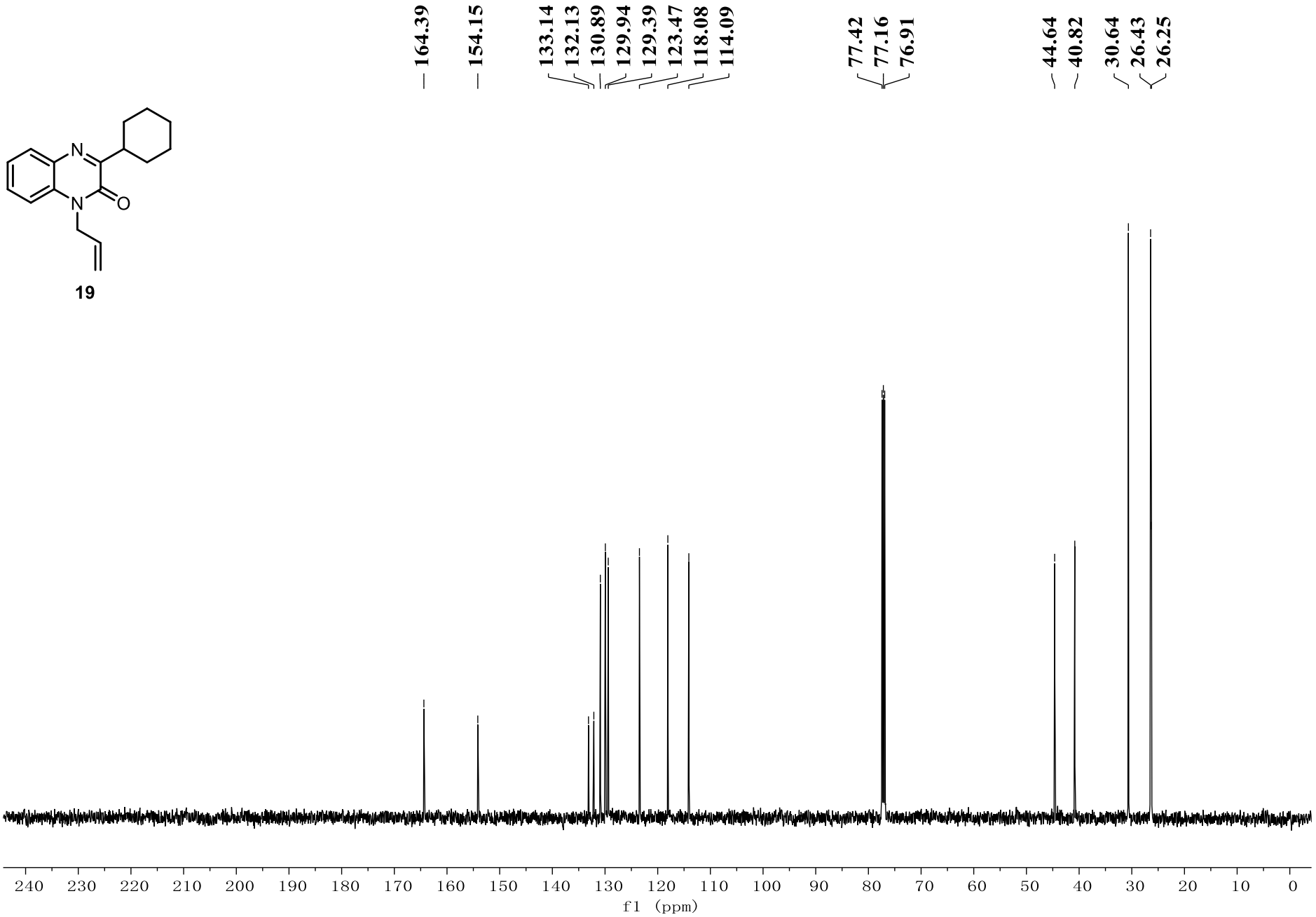
^{13}C NMR (125 MHz, $CDCl_3$) for 18



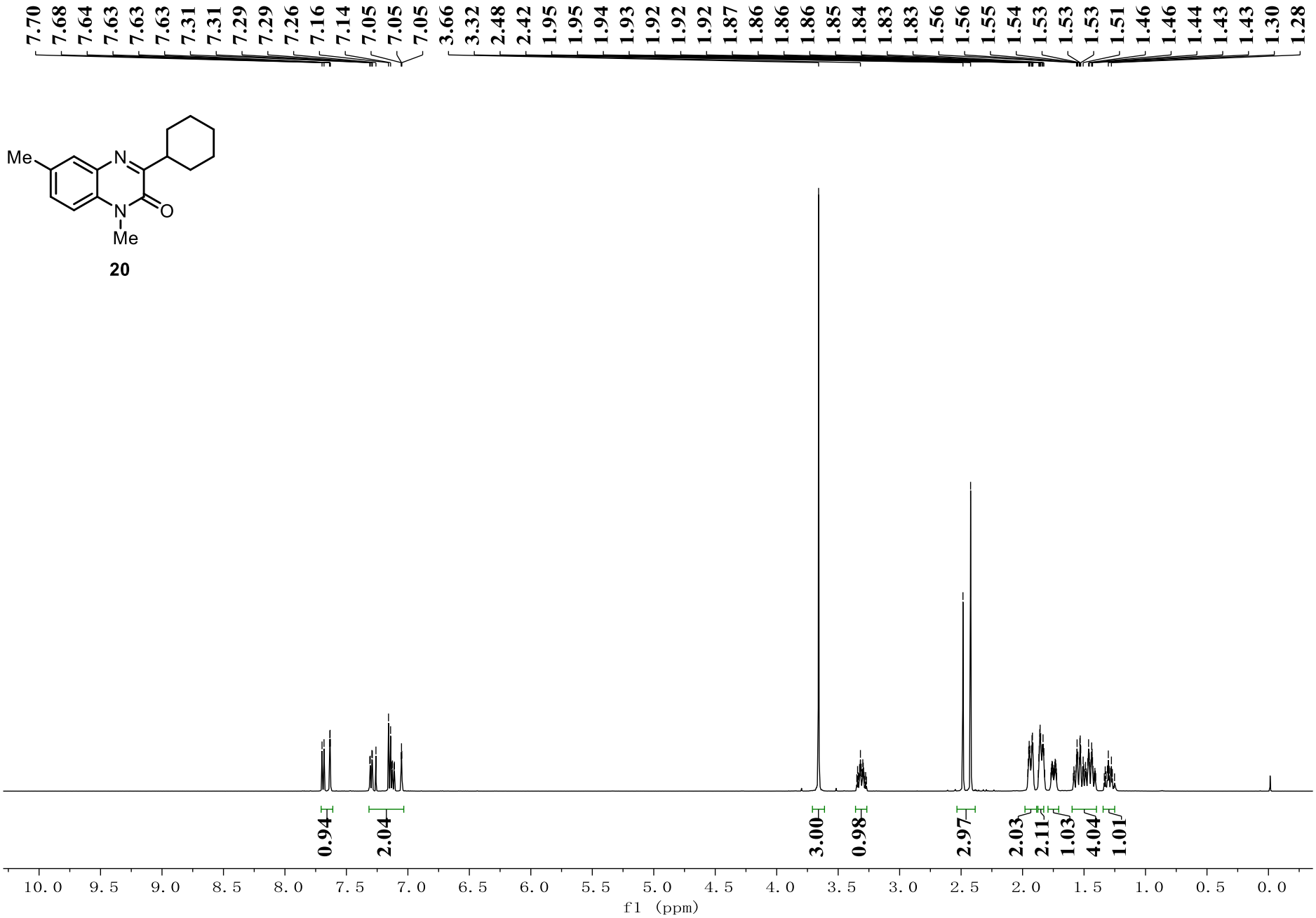
^{1}H NMR (500 MHz, $CDCl_3$) for 19



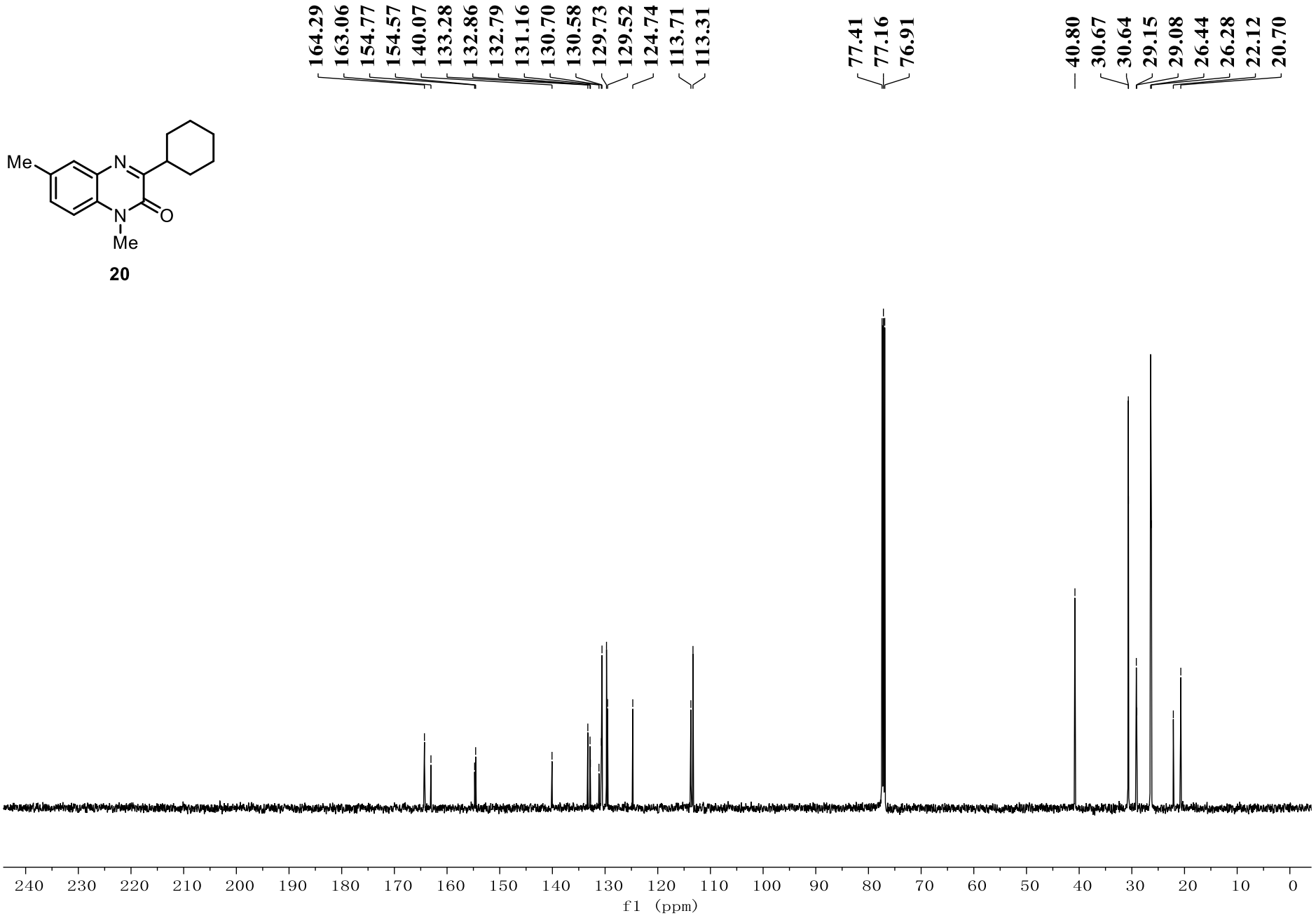
^{13}C NMR (125 MHz, $CDCl_3$) for 19



^{1}H NMR (500 MHz, $CDCl_3$) for 20

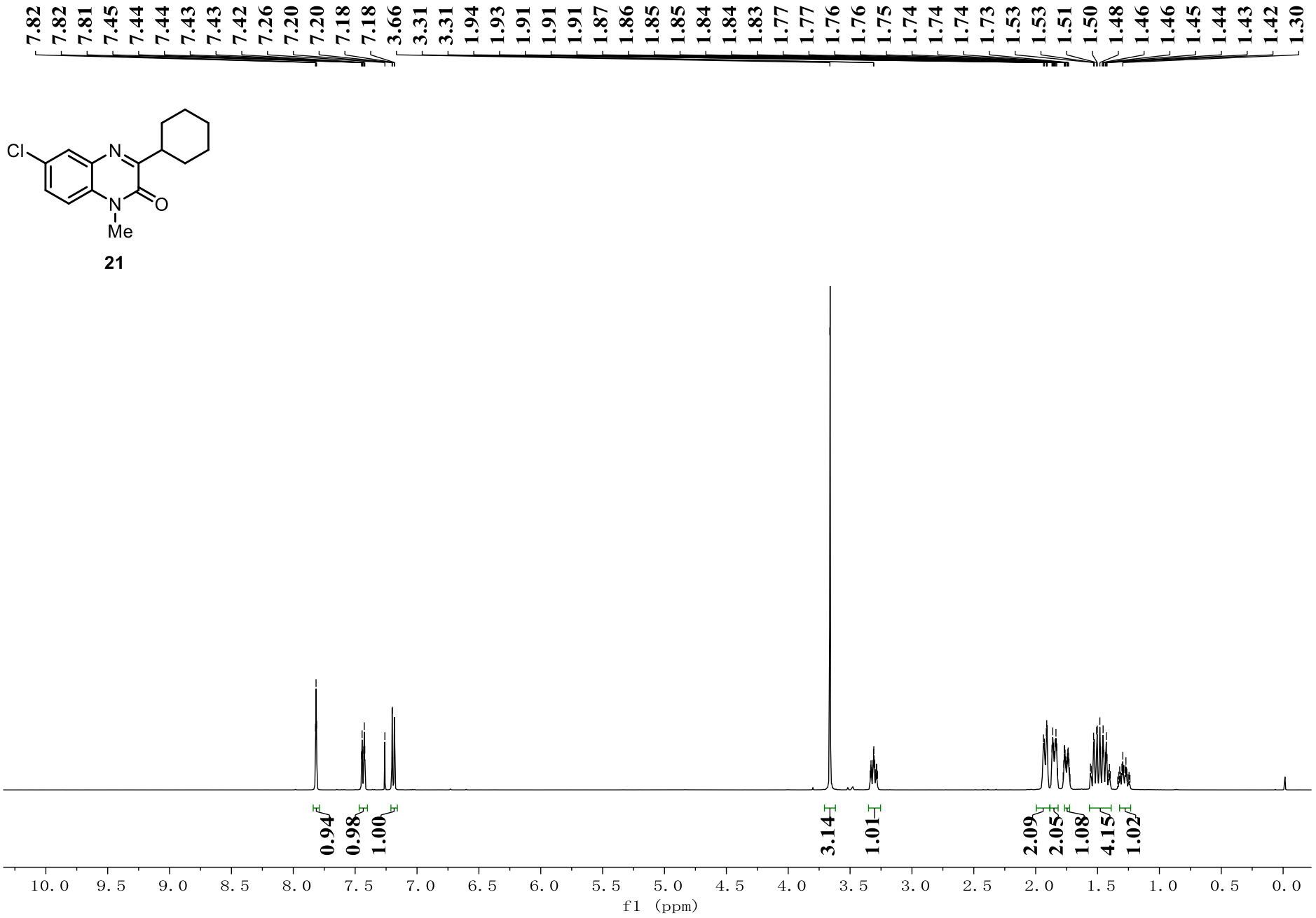


^{13}C NMR (125 MHz, $CDCl_3$) for 20

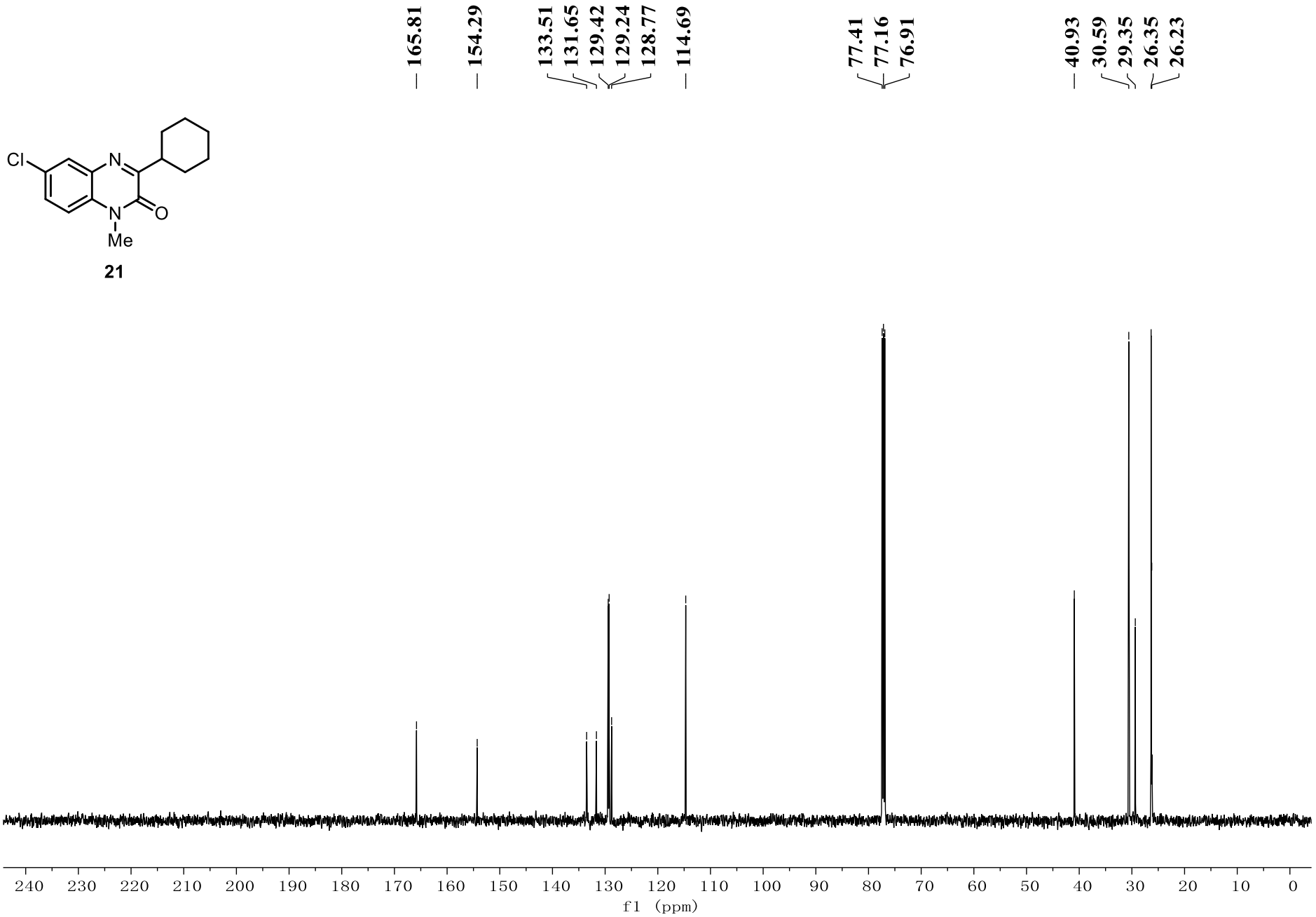


20

^{1}H NMR (500 MHz, $CDCl_3$) for 21

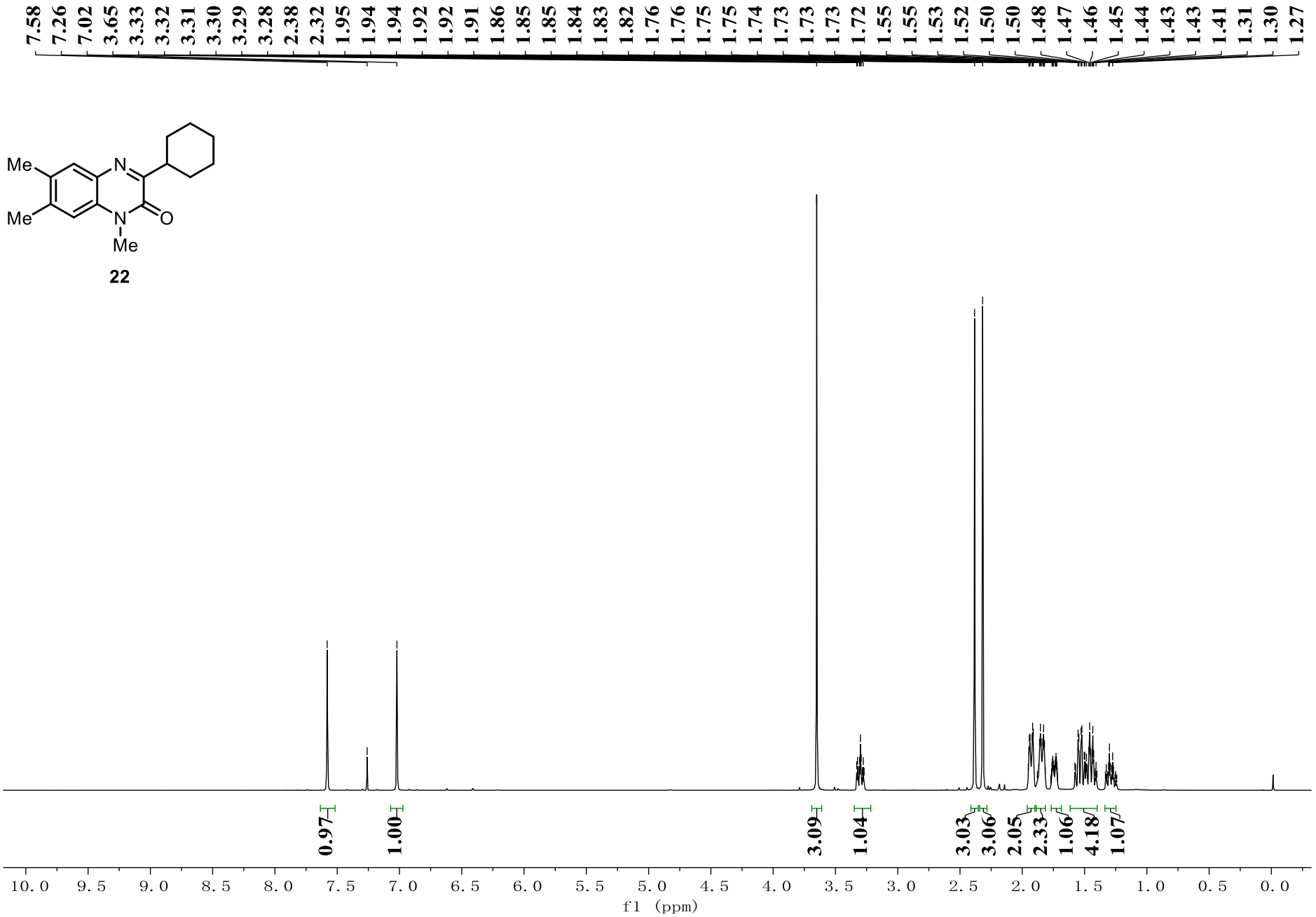


^{13}C NMR (125 MHz, $CDCl_3$) for 21

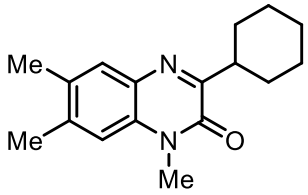


21

^{1}H NMR (500 MHz, $CDCl_3$) for 22

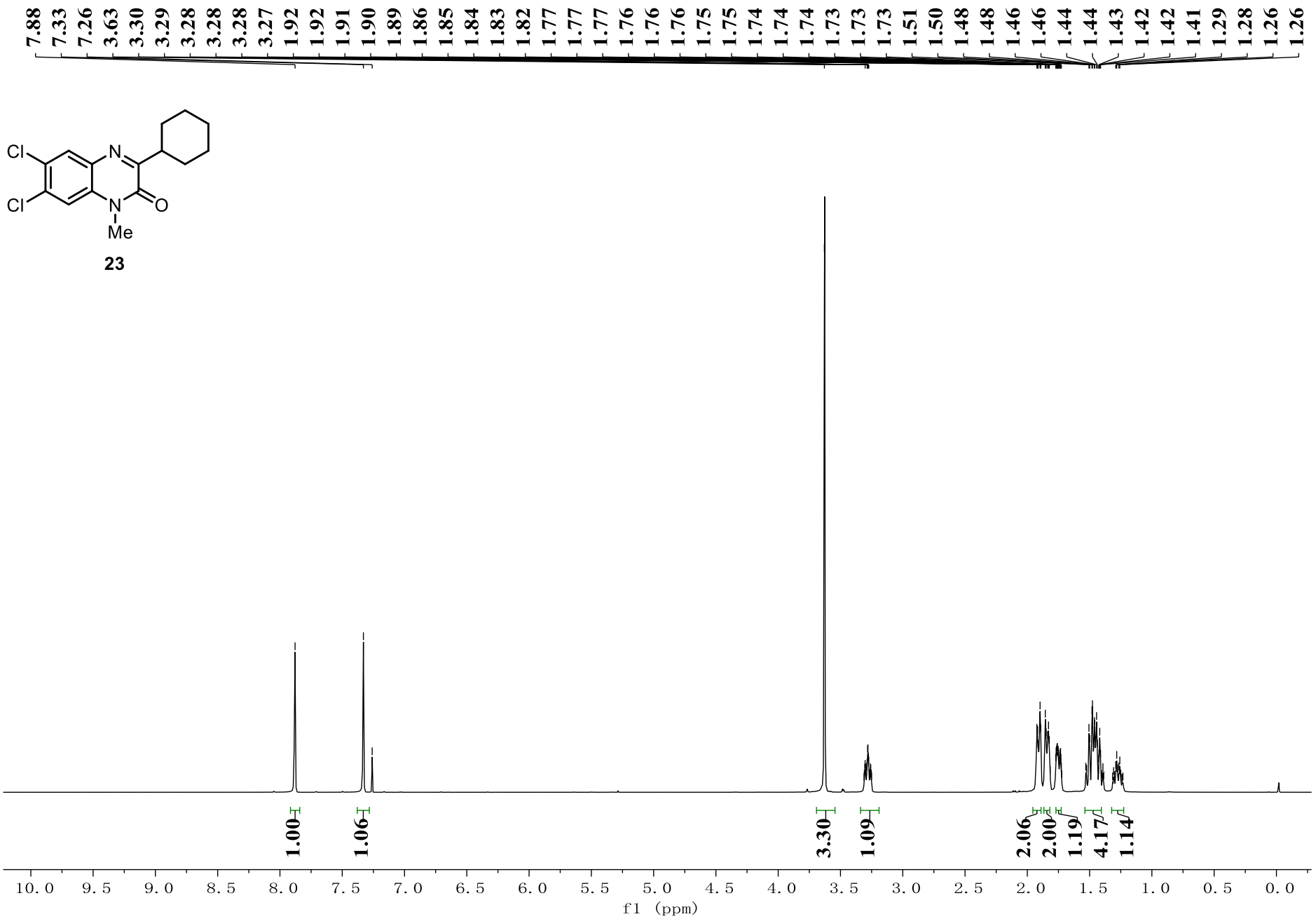


^{13}C NMR (125 MHz, $CDCl_3$) for 22

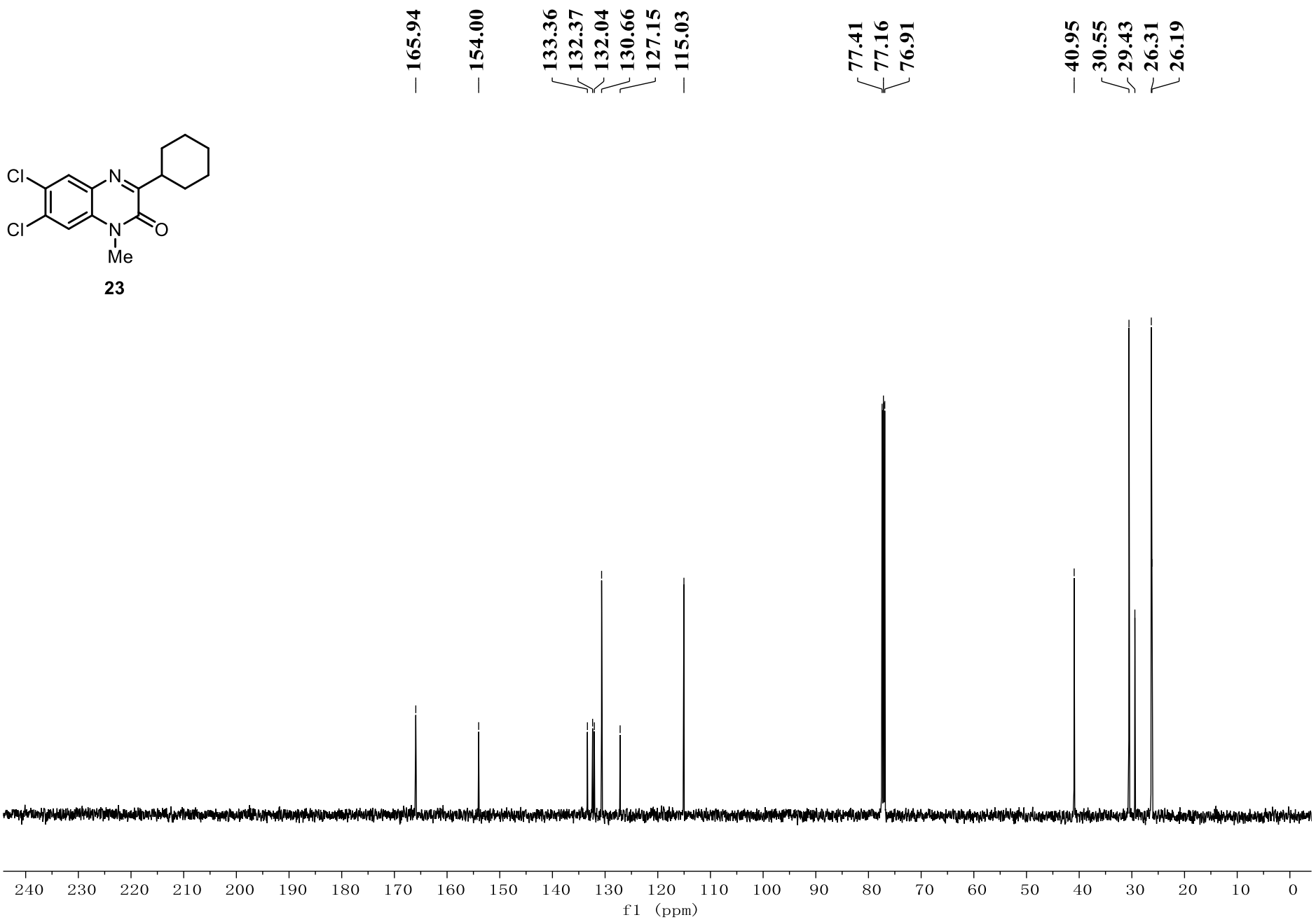


22

^{1}H NMR (500 MHz, $CDCl_3$) for 23

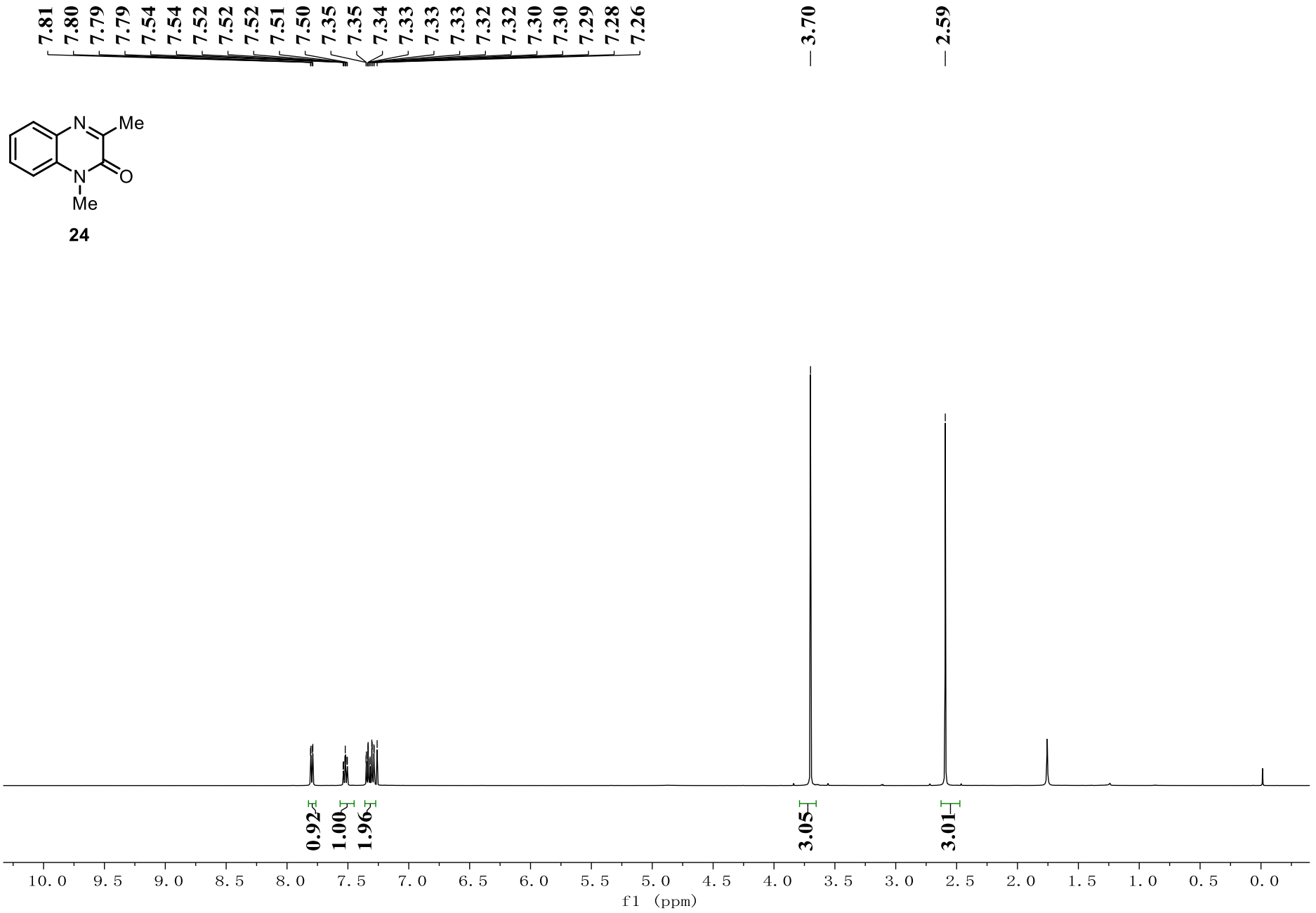


^{13}C NMR (125 MHz, $CDCl_3$) for 23



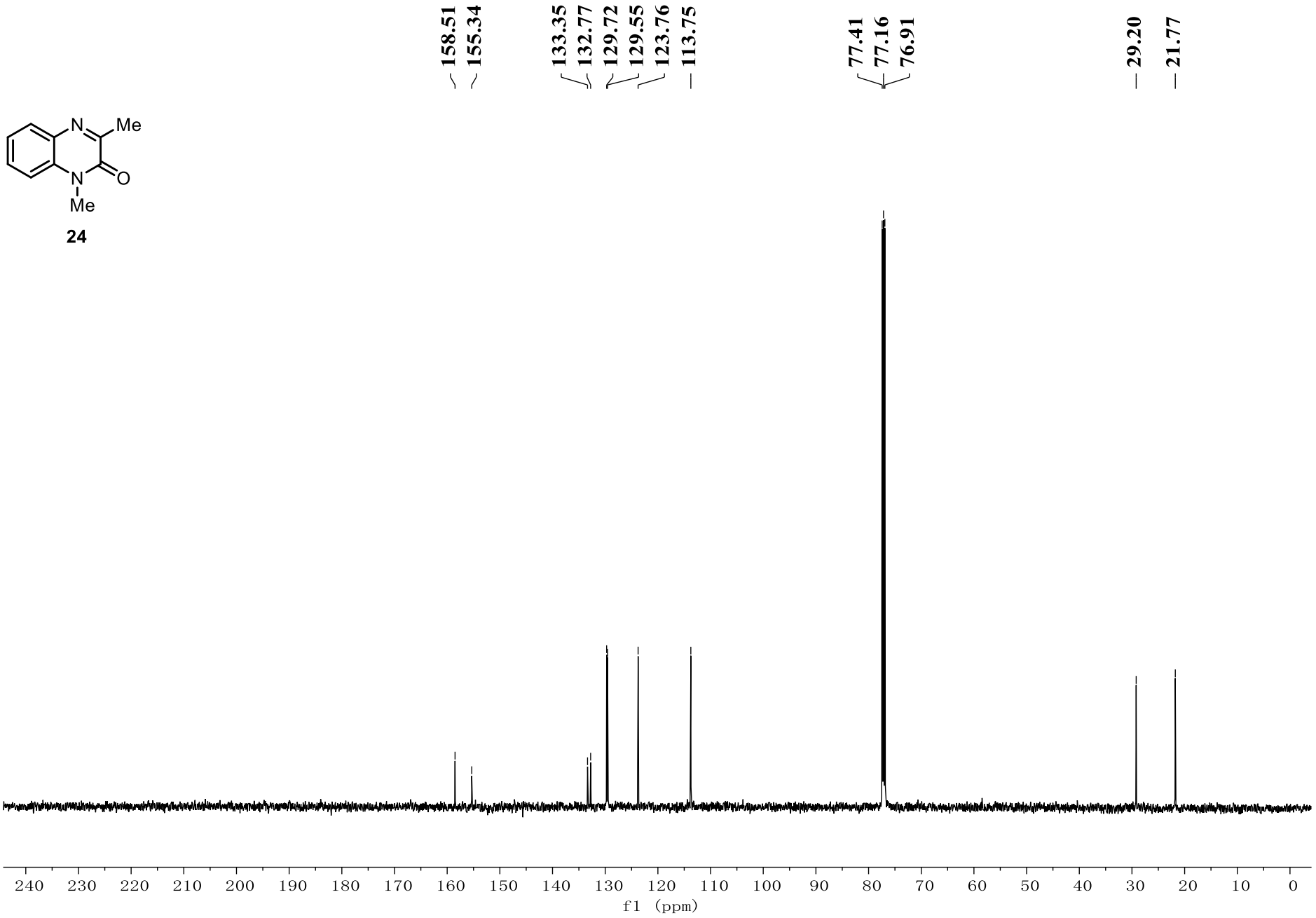
23

^{1}H NMR (500 MHz, $CDCl_3$) for 24



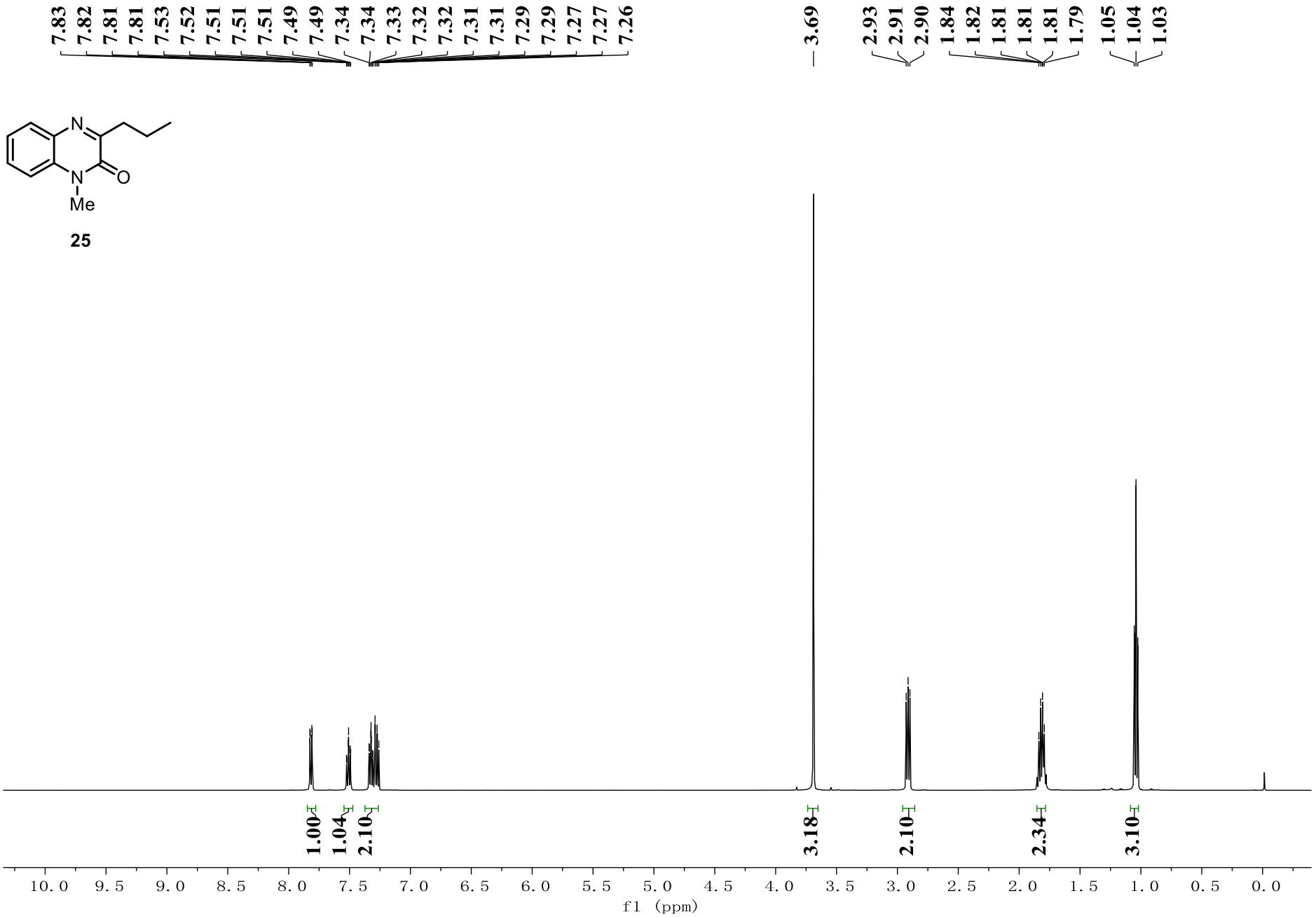
24

^{13}C NMR (125 MHz, $CDCl_3$) for 24



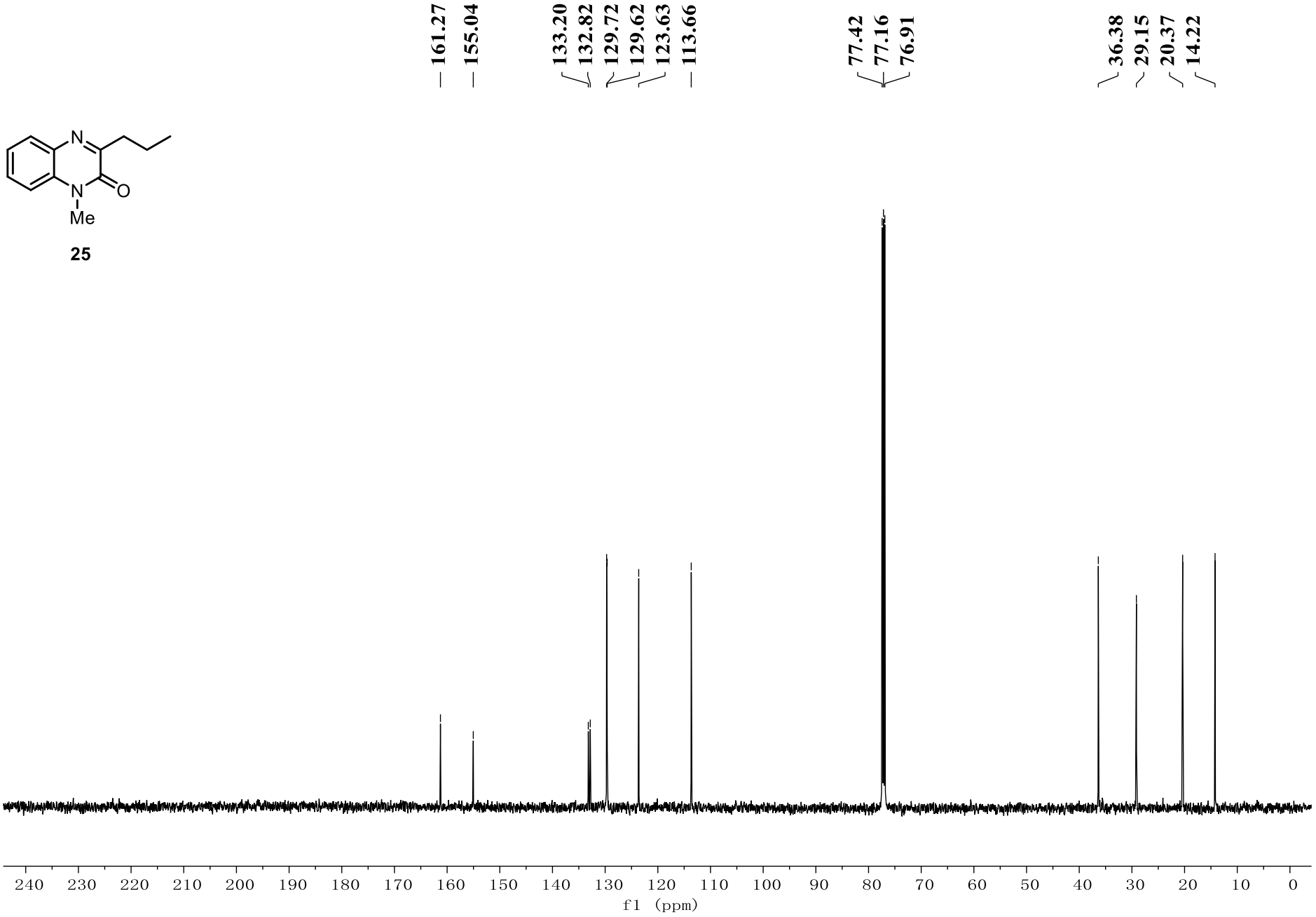
24

^{1}H NMR (500 MHz, $CDCl_3$) for 25



25

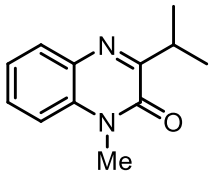
^{13}C NMR (125 MHz, $CDCl_3$) for 25



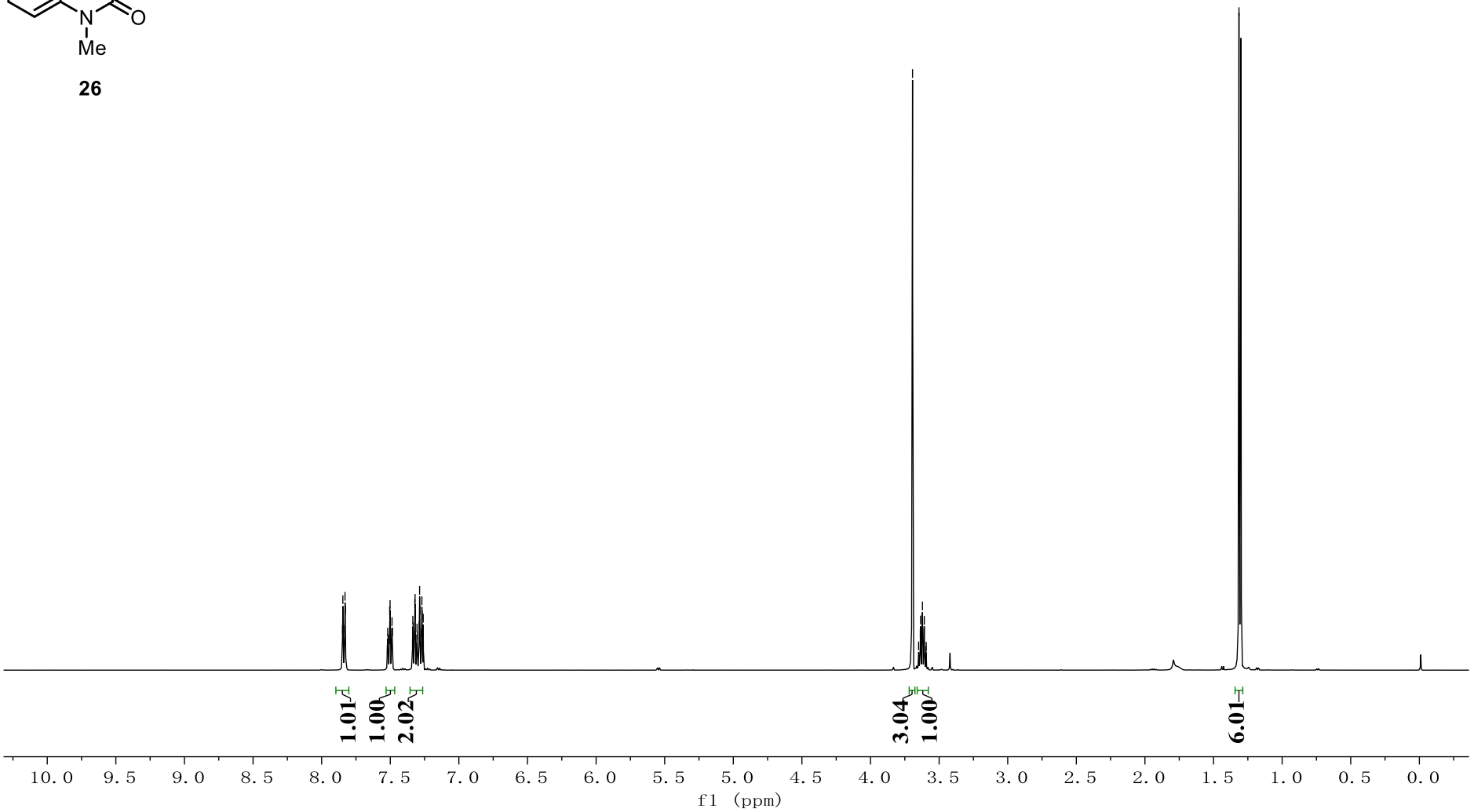
25

^{1}H NMR (500 MHz, $CDCl_3$) for 26

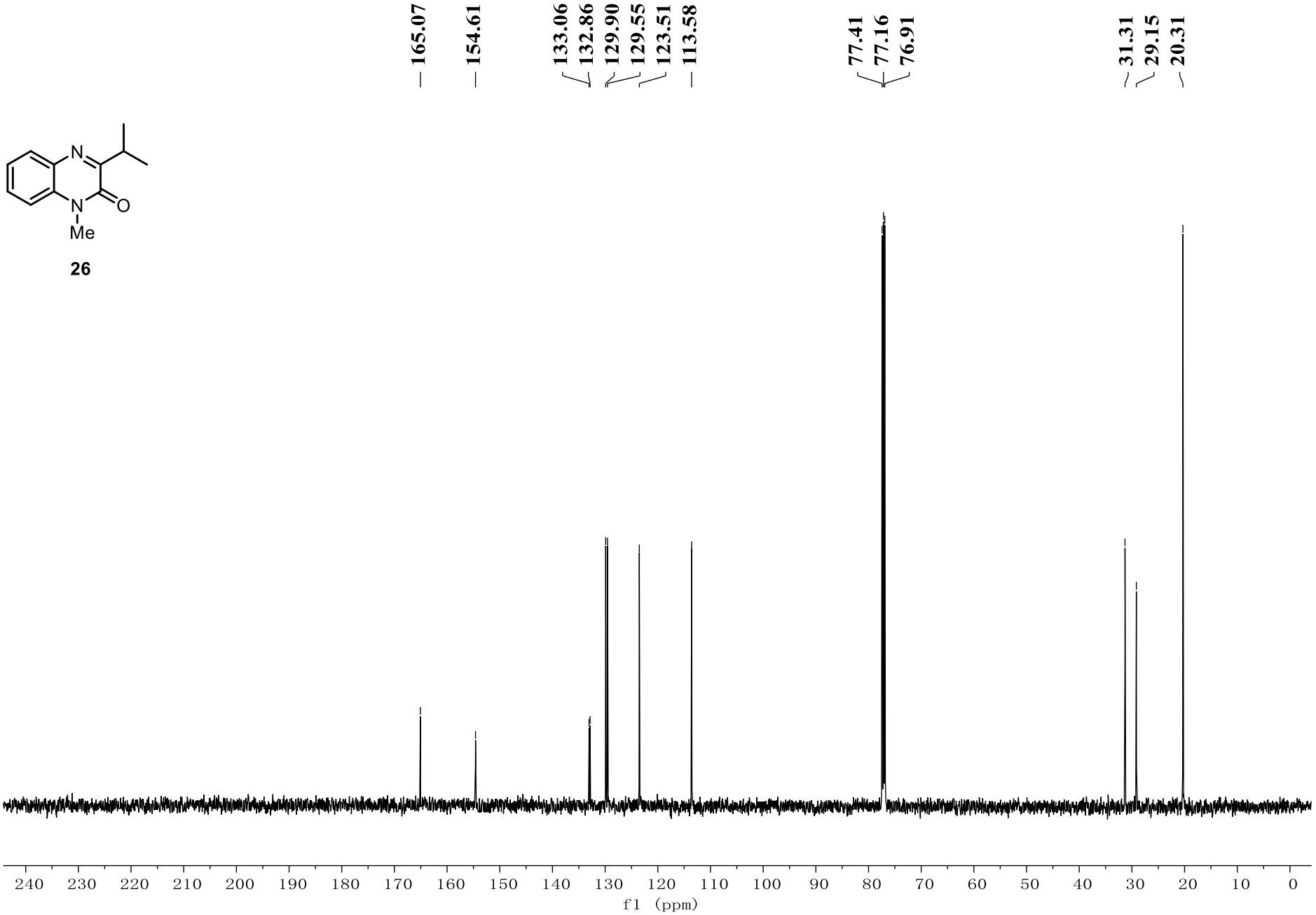
7.85 7.85 7.84 7.83 7.83 7.83 7.52 7.52 7.51 7.50 7.50 7.50 7.49 7.49 7.48 7.34 7.34 7.33 7.32 7.32 7.32 7.32 7.31 7.30 7.30 7.29 7.29 7.28 7.27 7.27 7.27 7.26 3.69 3.65 3.64 3.64 3.62 3.62 3.61 3.61 3.59 1.31 1.30



26

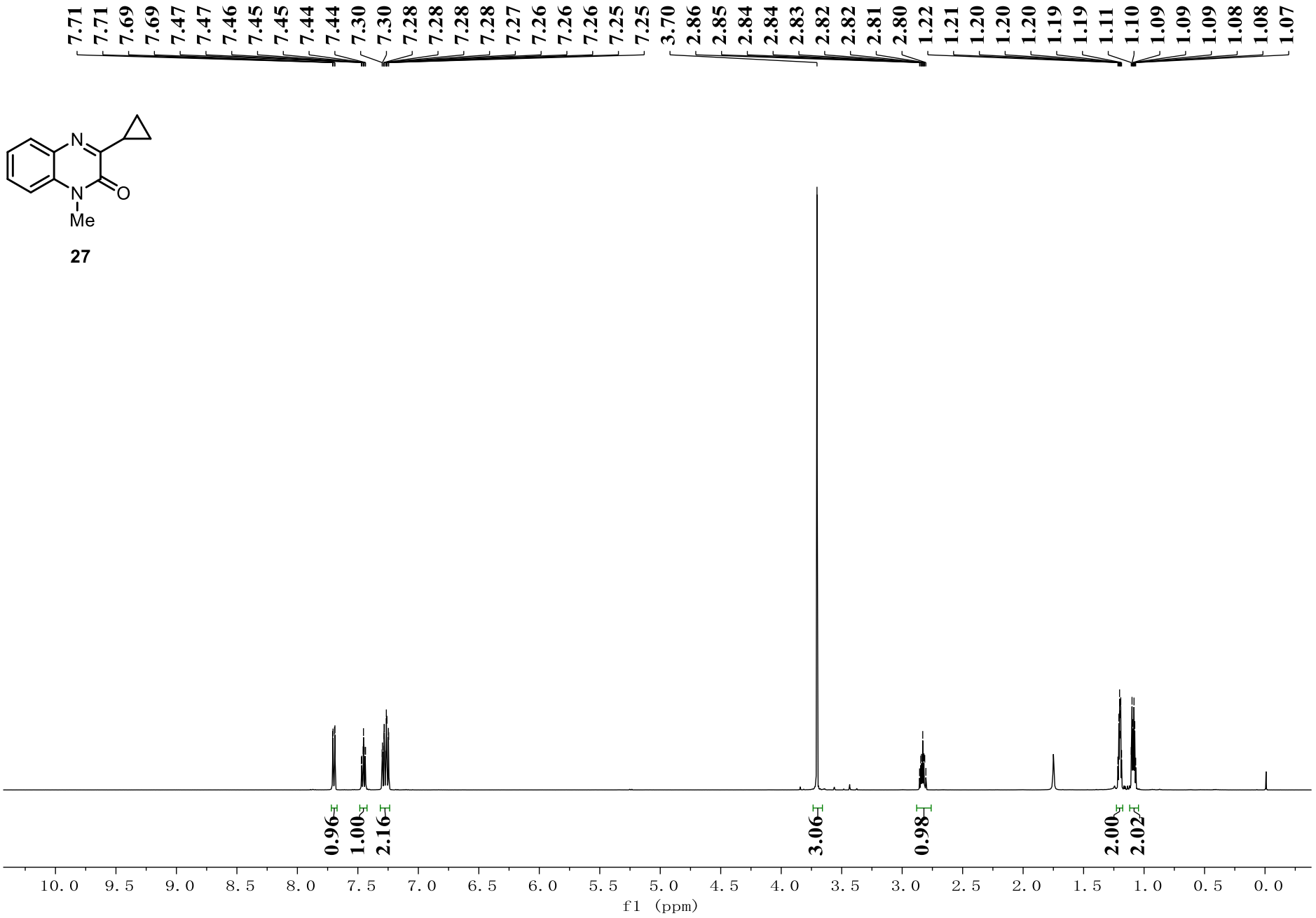


^{13}C NMR (125 MHz, $CDCl_3$) for 26



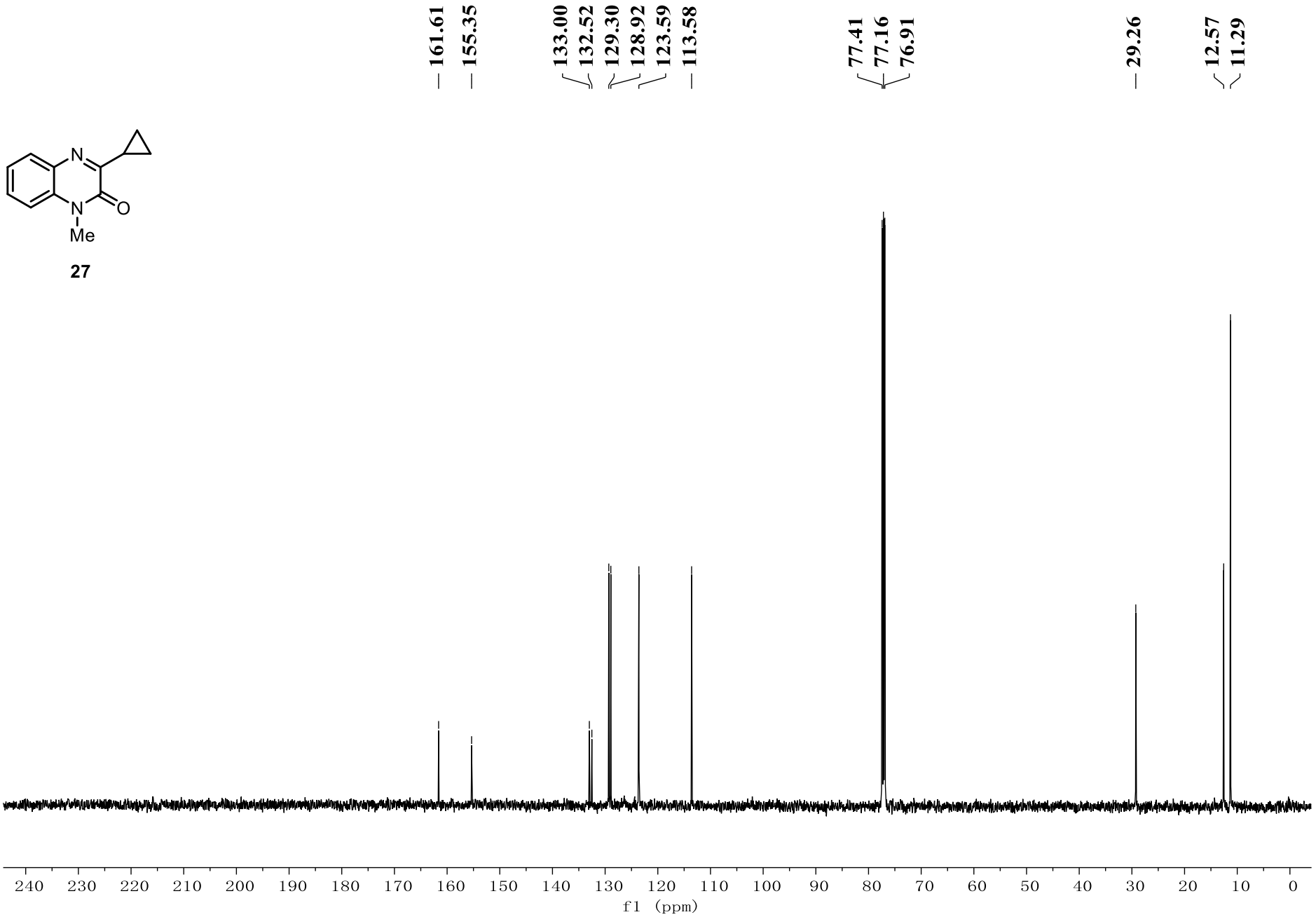
26

^{1}H NMR (500 MHz, $CDCl_3$) for 27



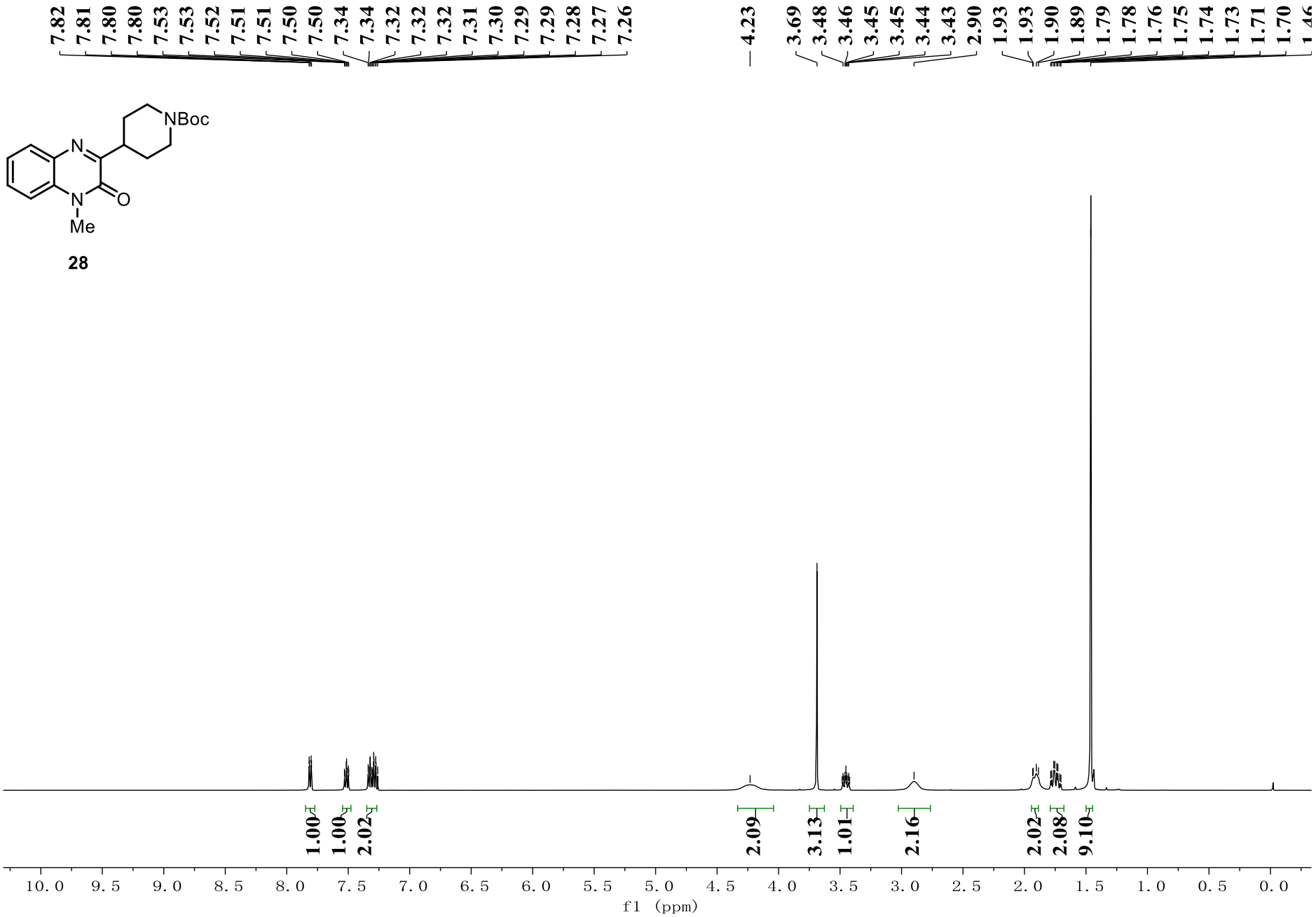
27

^{13}C NMR (125 MHz, $CDCl_3$) for 27

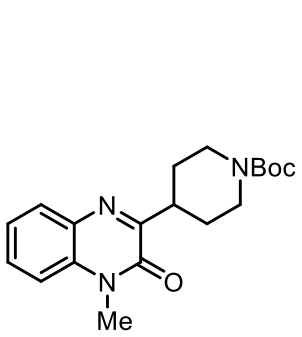


27

^{1}H NMR (500 MHz, $CDCl_3$) for 28

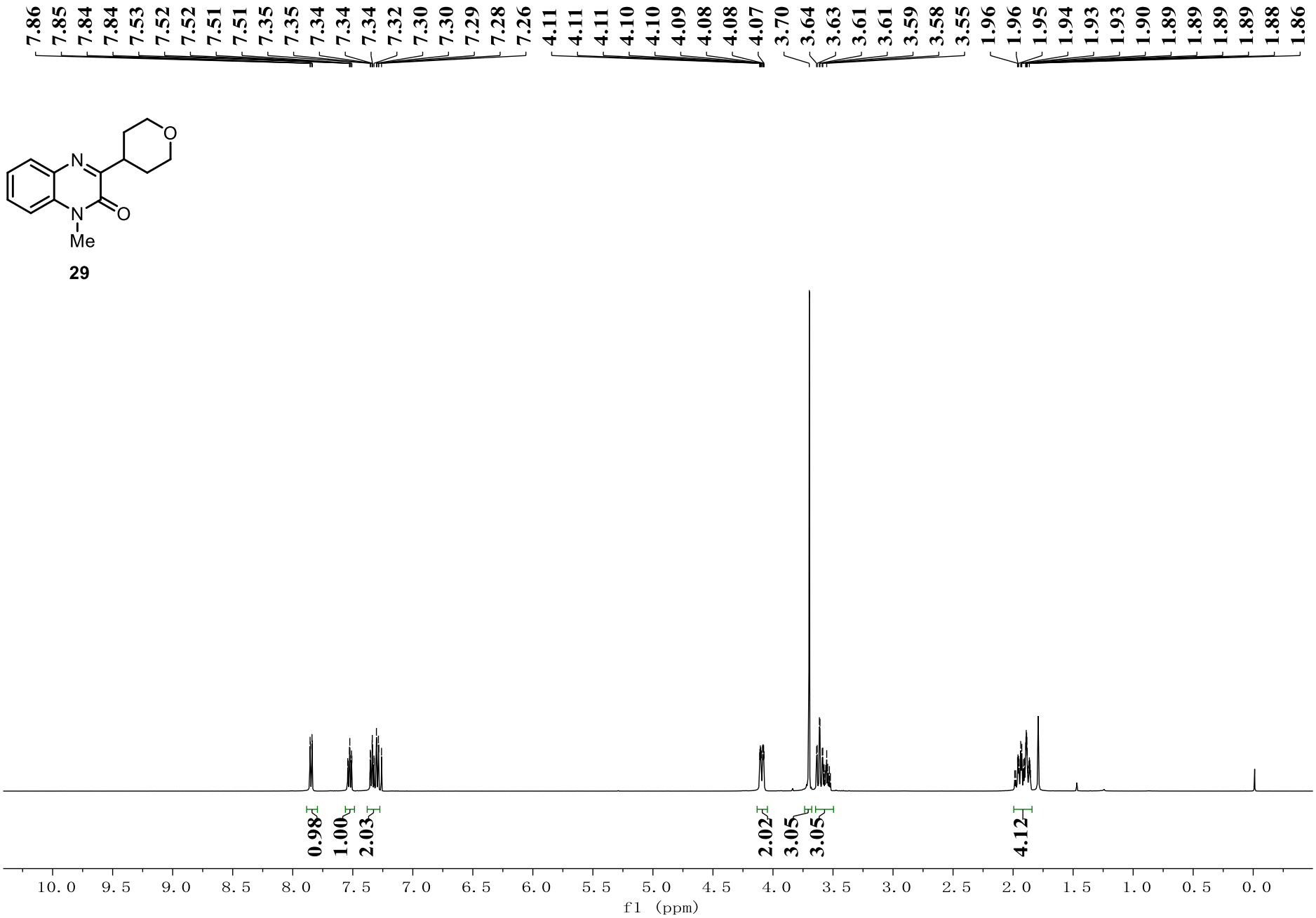


^{13}C NMR (125 MHz, $CDCl_3$) for 28



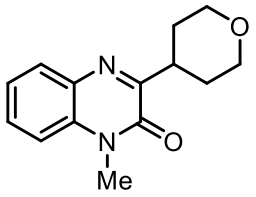
28

^{1}H NMR (500 MHz, $CDCl_3$) for 29



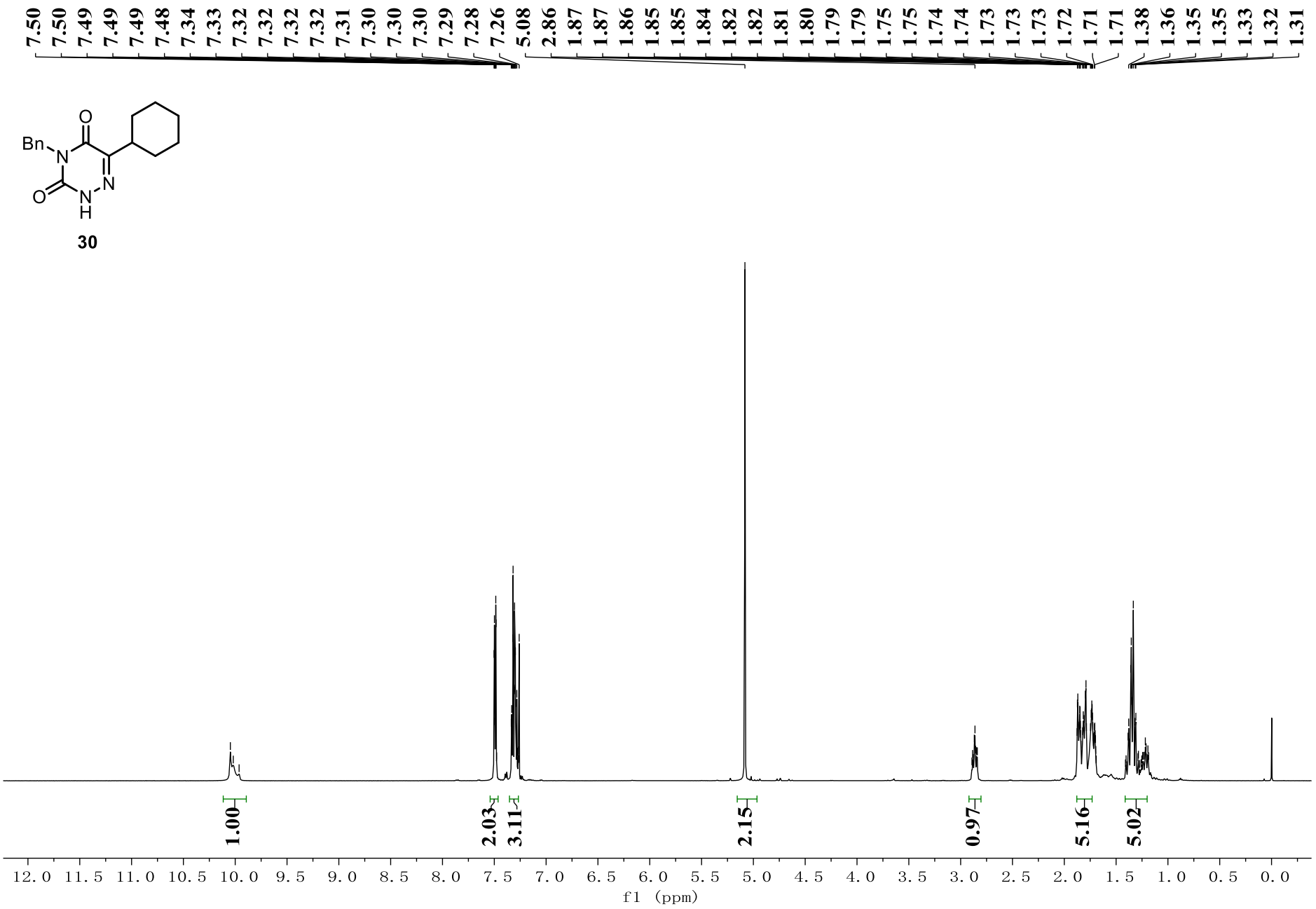
29

^{13}C NMR (125 MHz, $CDCl_3$) for 29

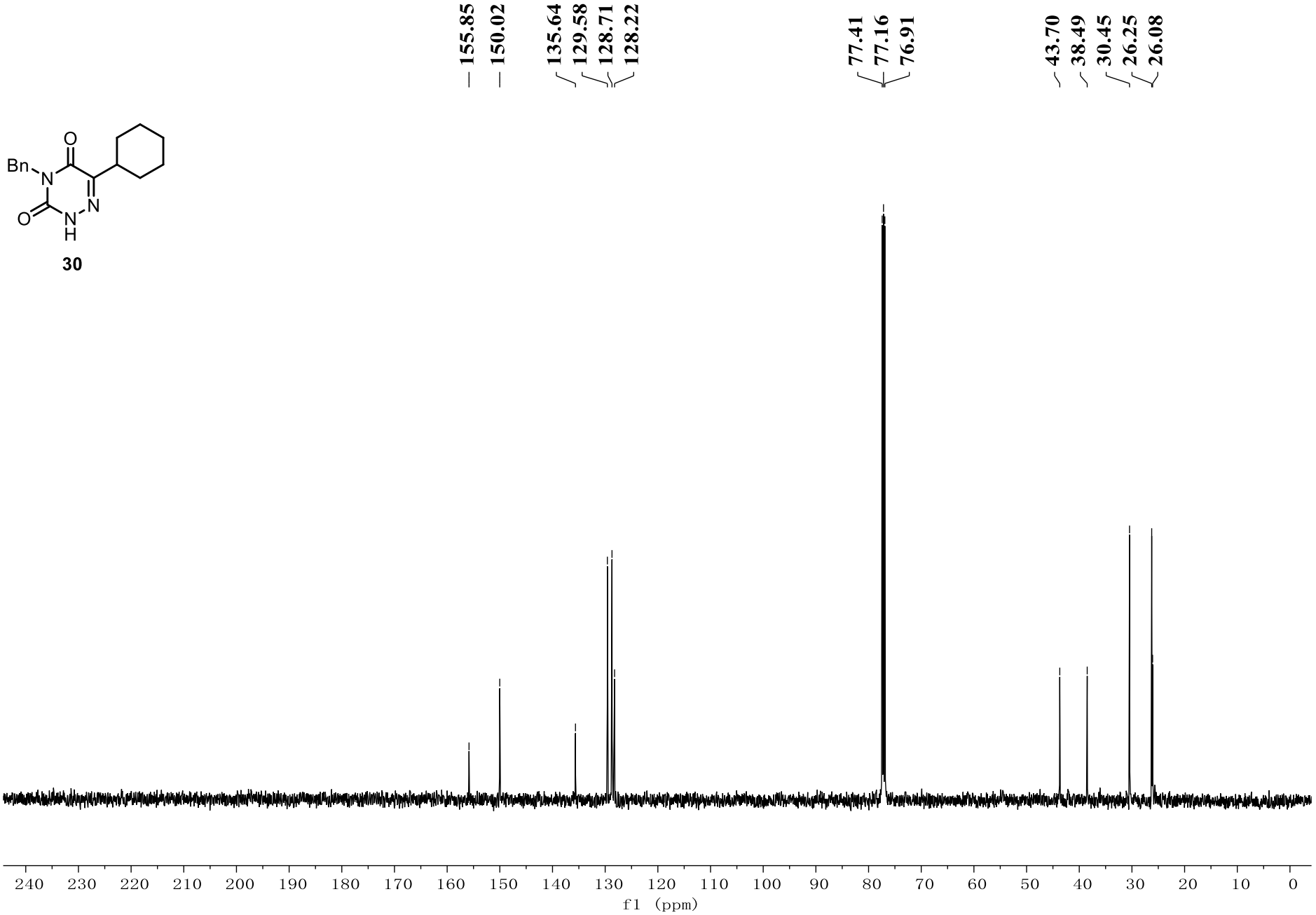


29

^{1}H NMR (500 MHz, $CDCl_3$) for 30

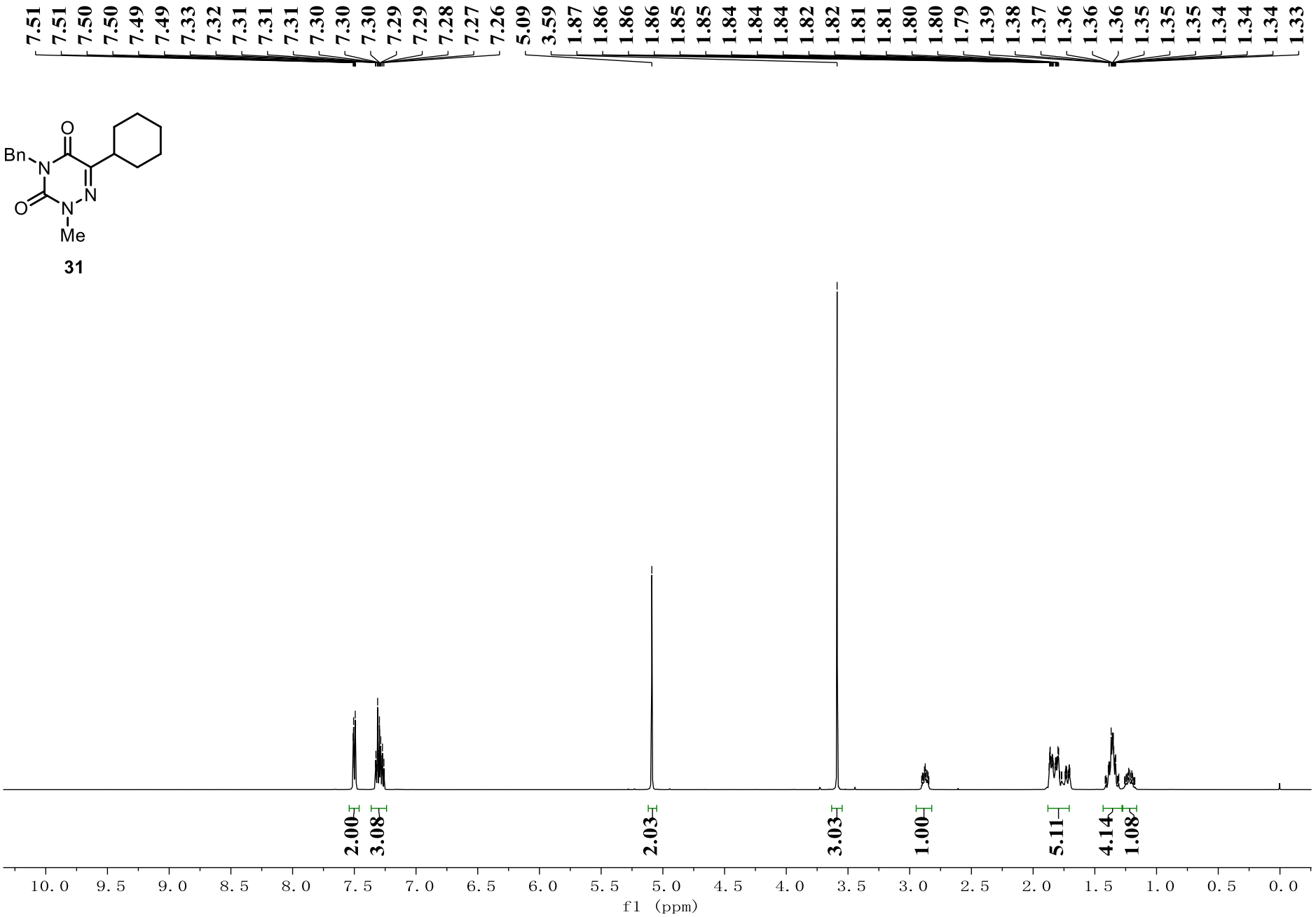


^{13}C NMR (125 MHz, $CDCl_3$) for 30



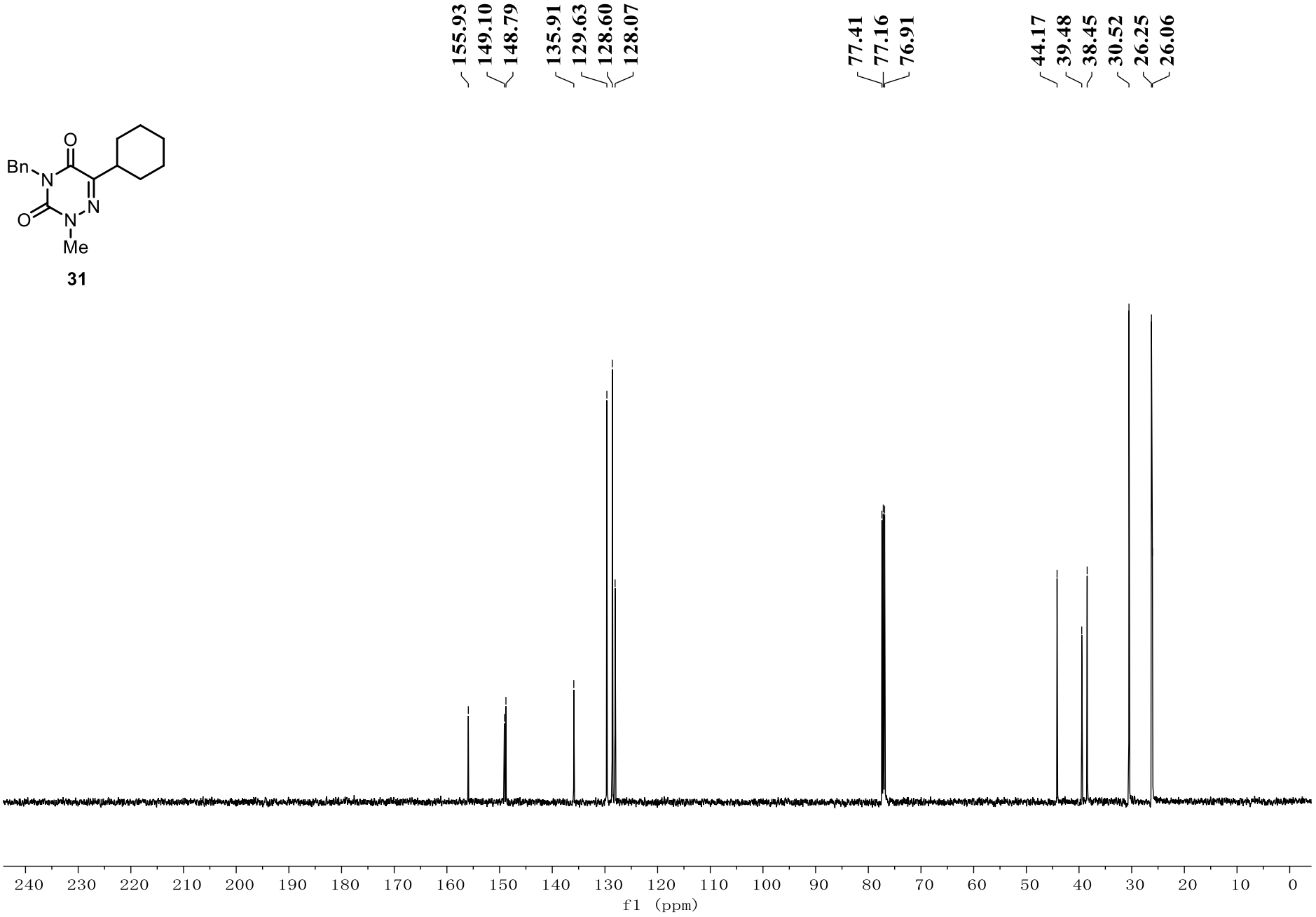
30

^{1}H NMR (500 MHz, $CDCl_3$) for 31



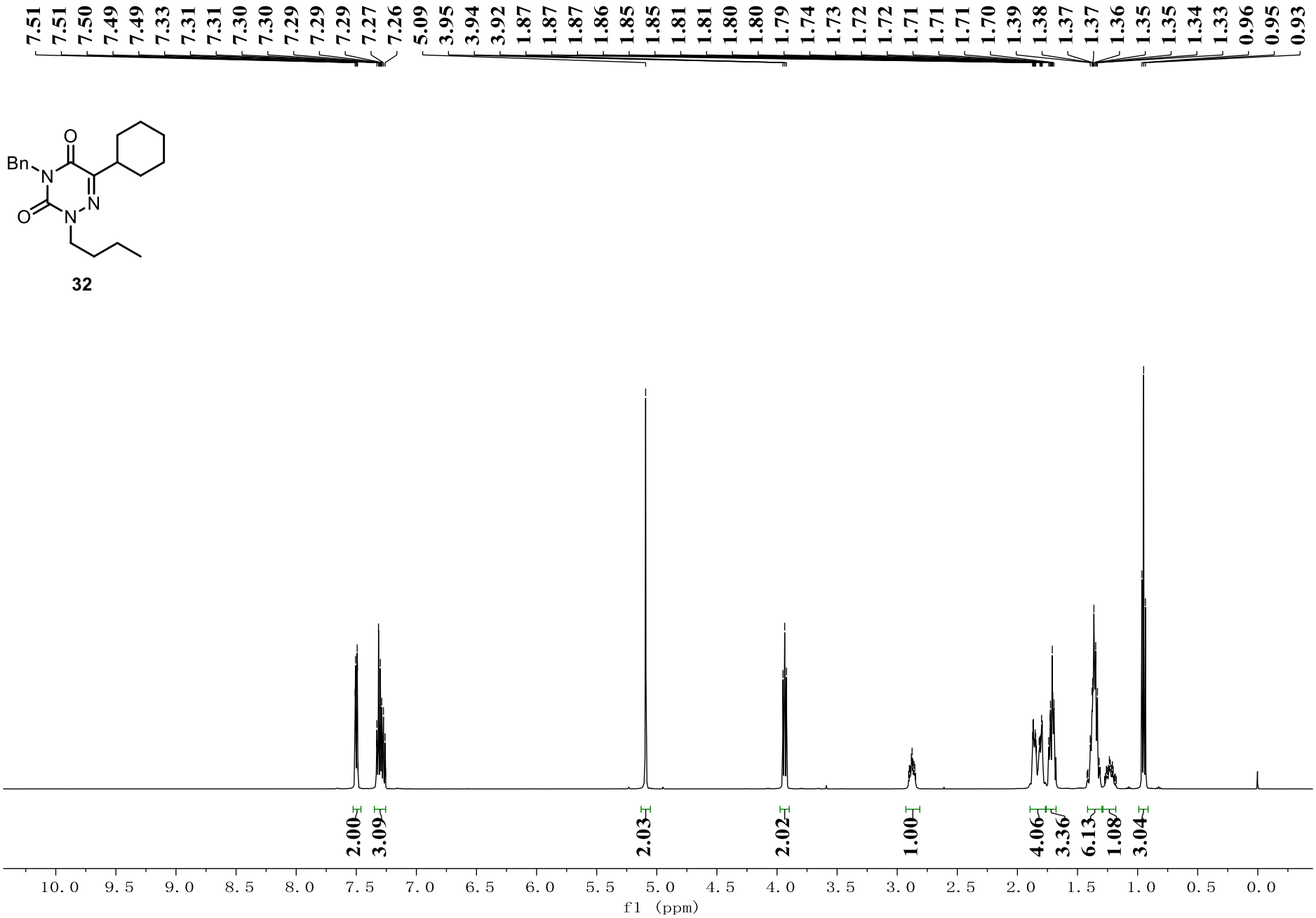
31

^{13}C NMR (125 MHz, $CDCl_3$) for 31



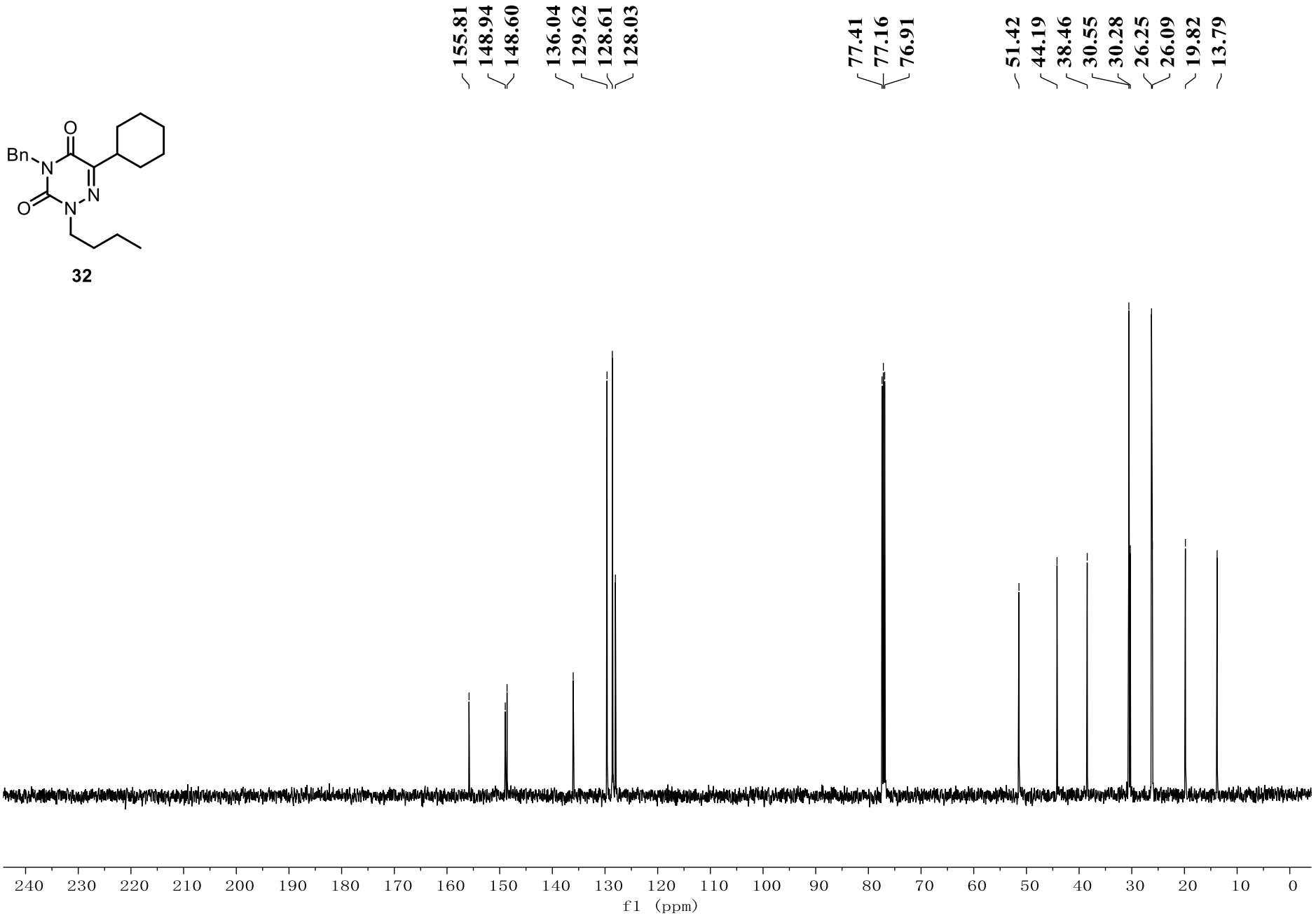
31

^{1}H NMR (500 MHz, $CDCl_3$) for 32



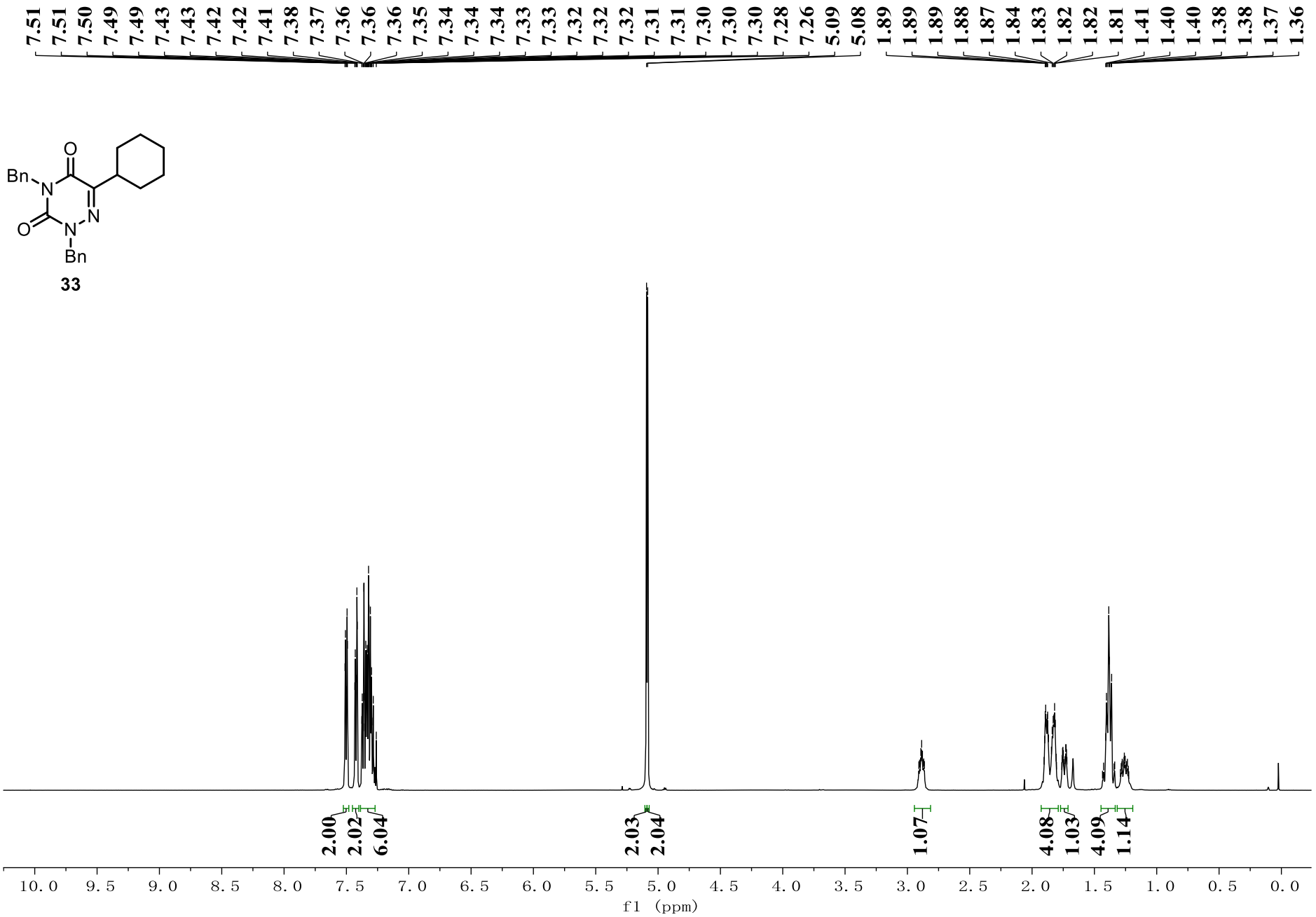
32

^{13}C NMR (125 MHz, $CDCl_3$) for 32

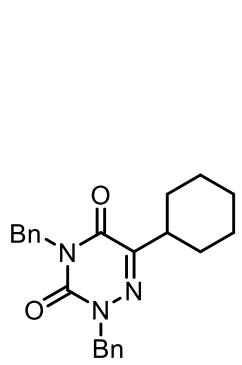


32

^{1}H NMR (500 MHz, $CDCl_3$) for 33

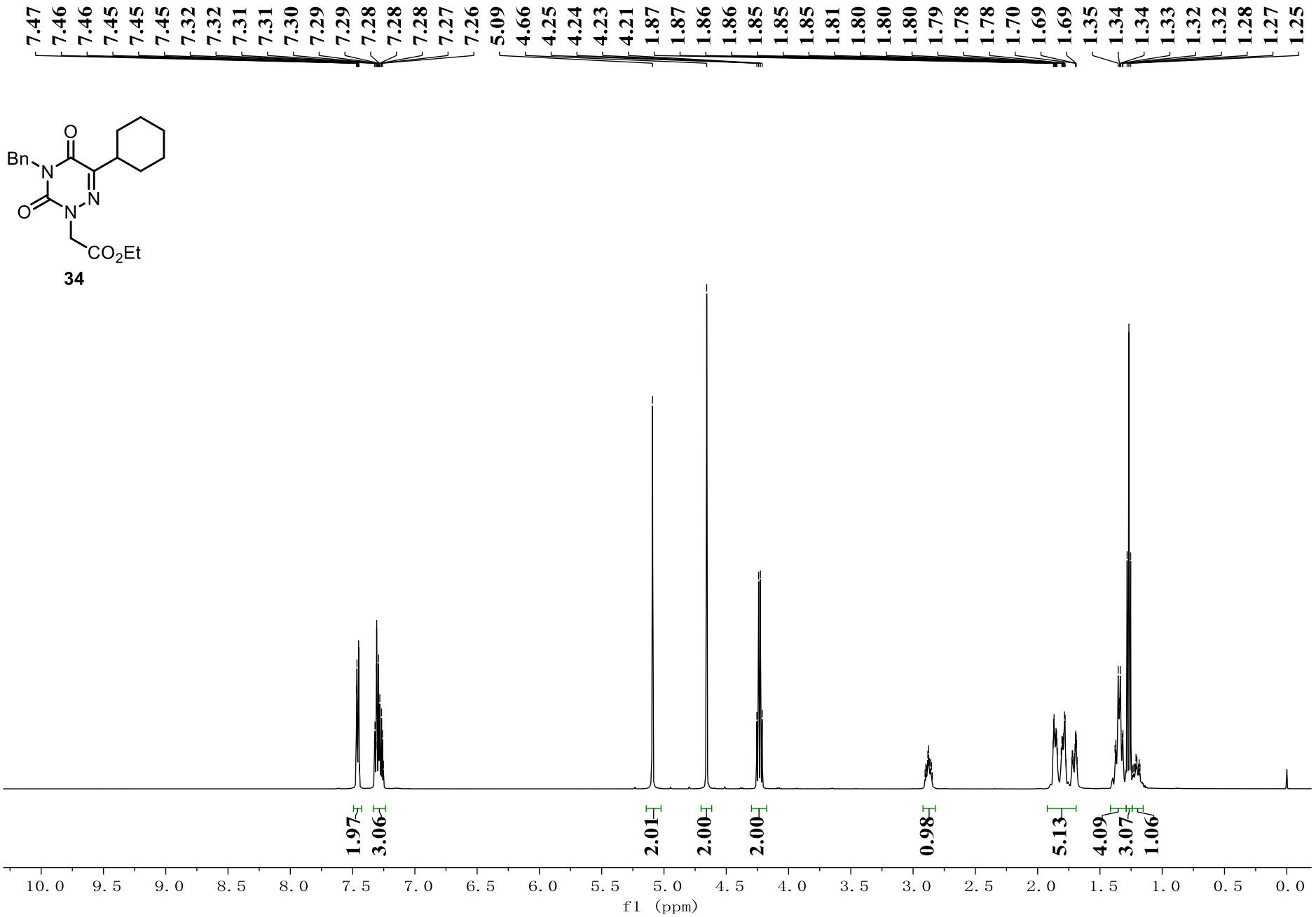


^{13}C NMR (125 MHz, $CDCl_3$) for 33



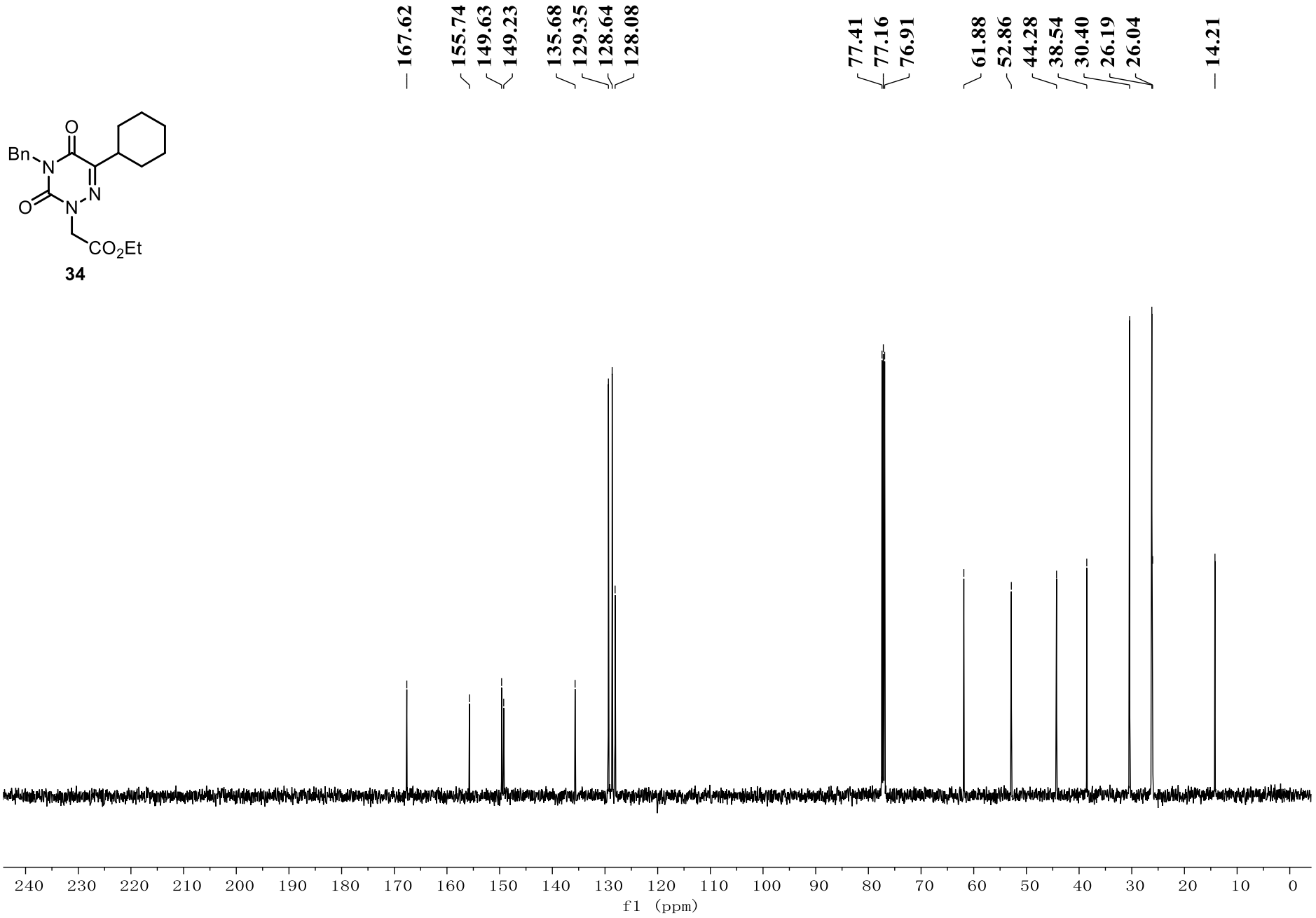
33

^{1}H NMR (500 MHz, $CDCl_3$) for 34

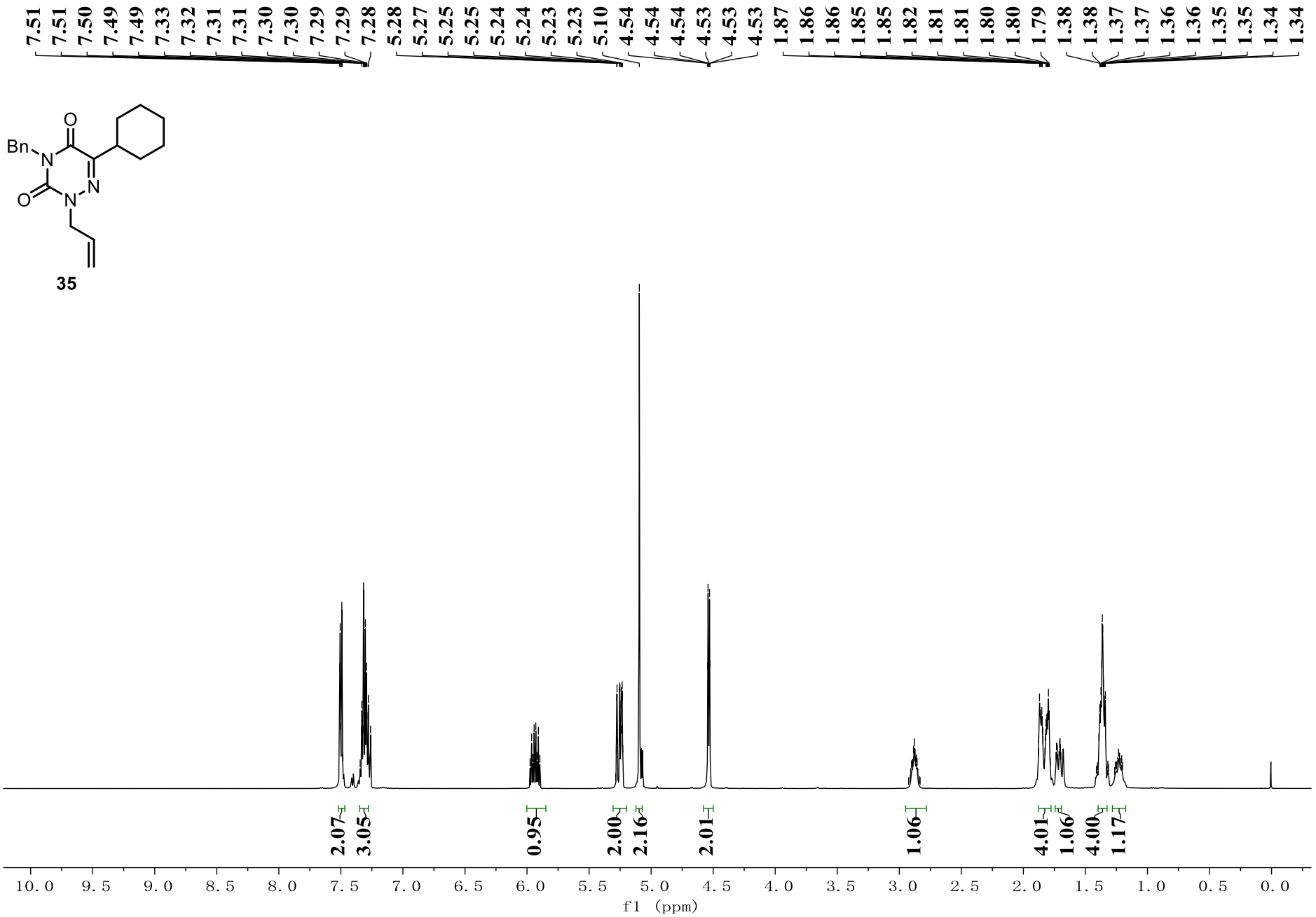


34

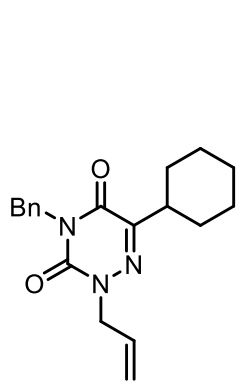
^{13}C NMR (125 MHz, $CDCl_3$) for 34



^{1}H NMR (500 MHz, $CDCl_3$) for 35

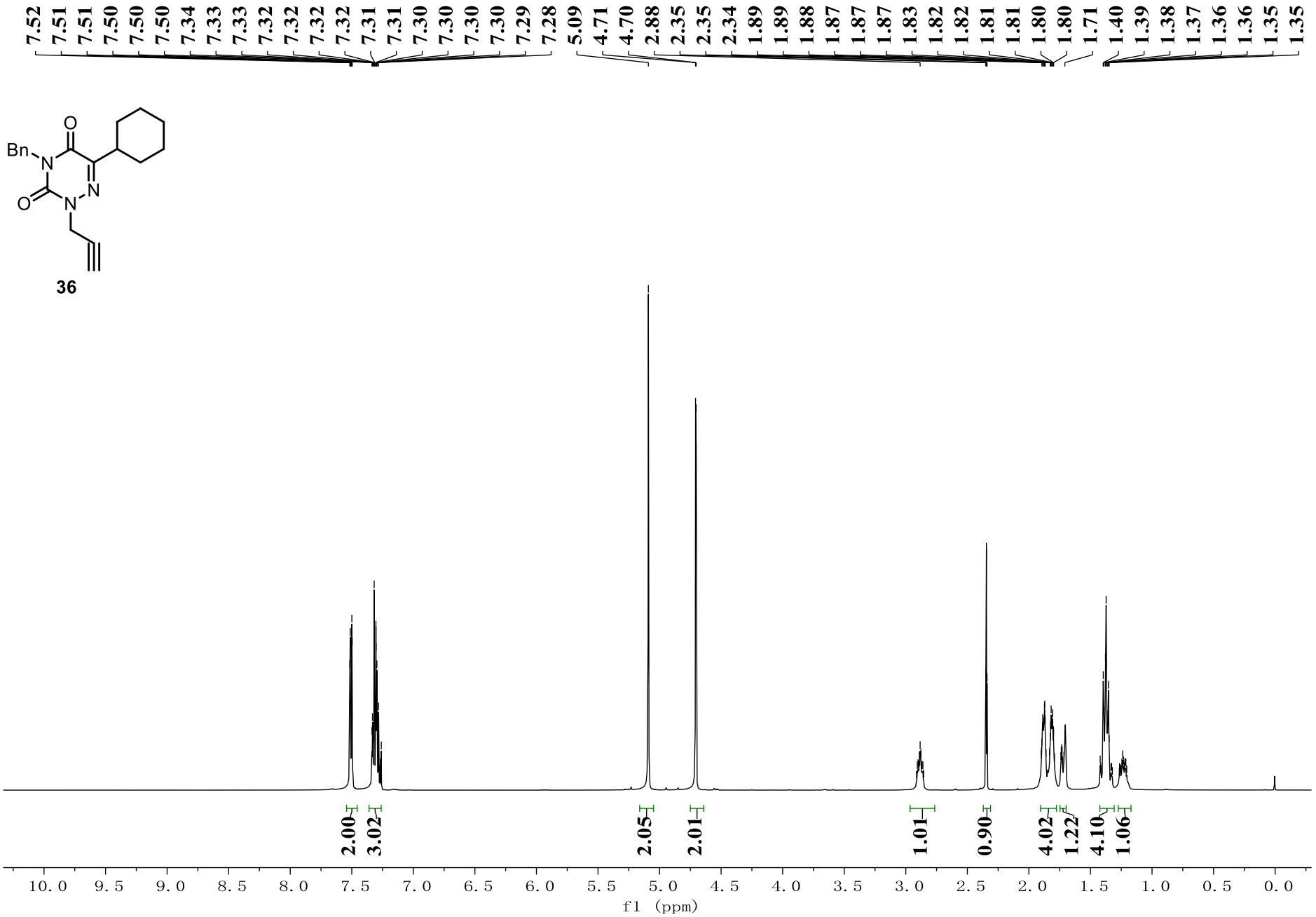


^{13}C NMR (125 MHz, $CDCl_3$) for 35

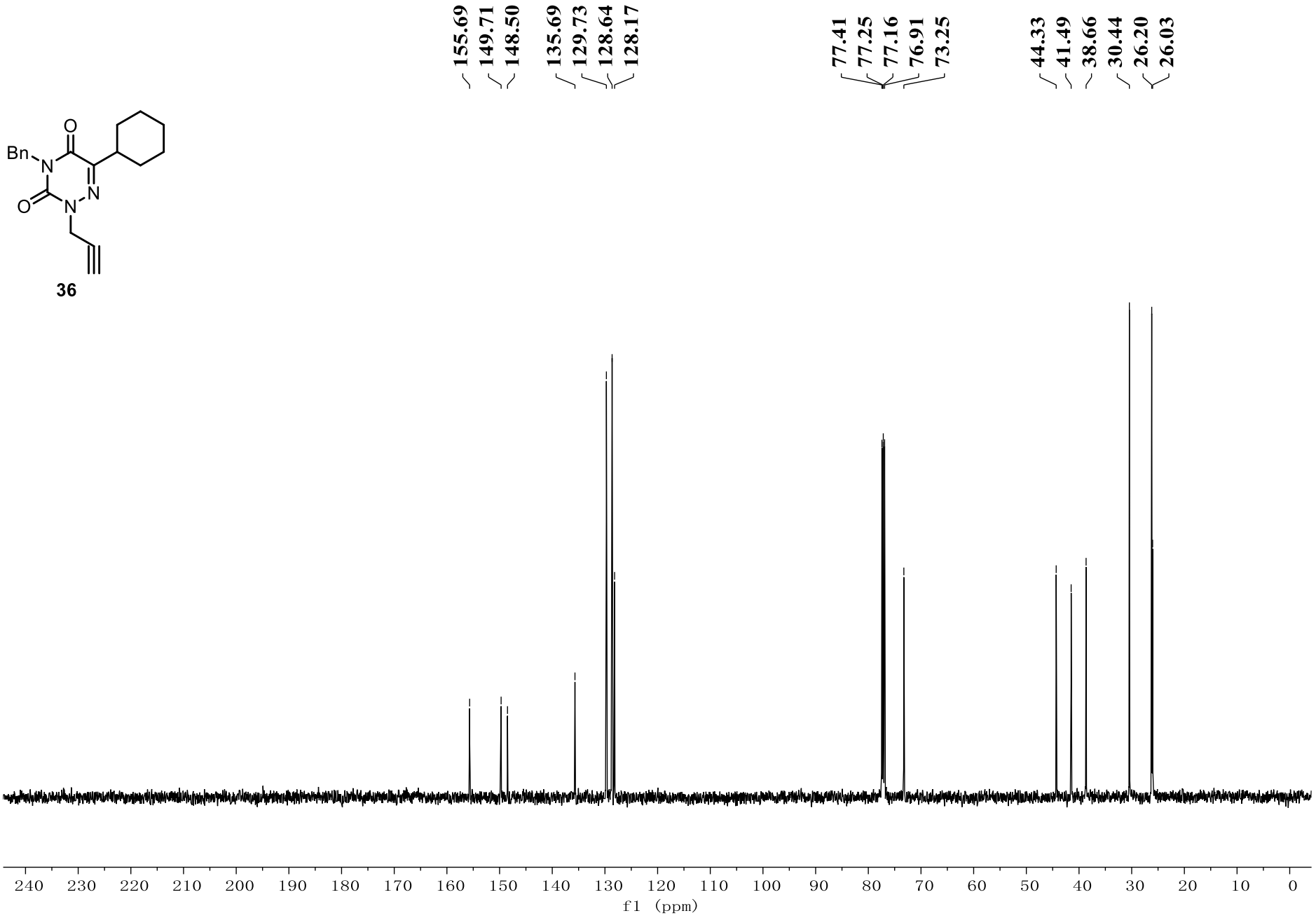


35

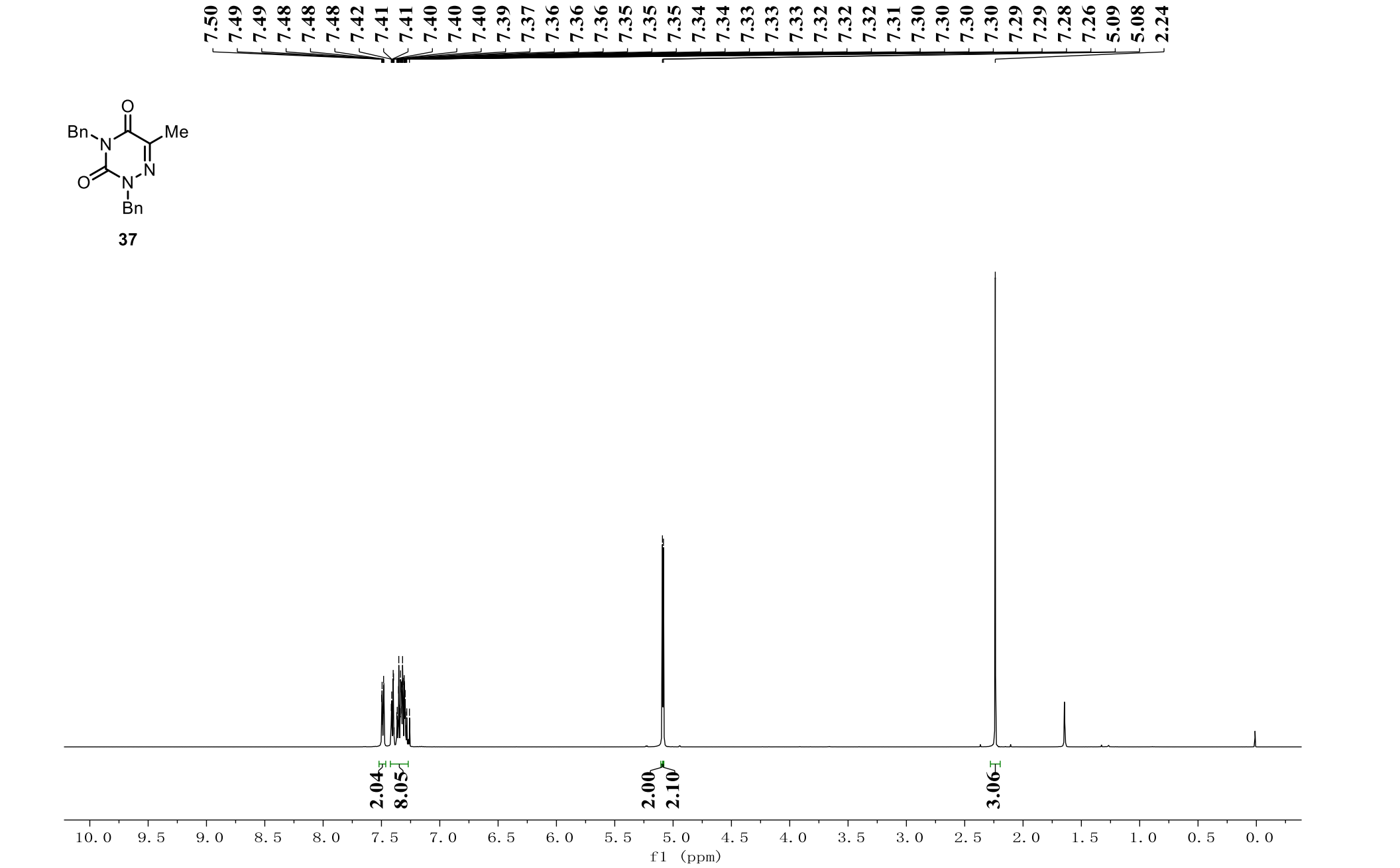
^{1}H NMR (500 MHz, $CDCl_3$) for 36



^{13}C NMR (125 MHz, $CDCl_3$) for 36

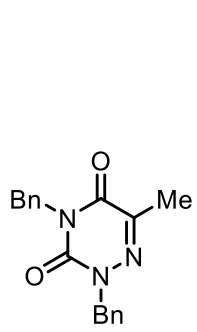


^{1}H NMR (500 MHz, $CDCl_3$) for 37



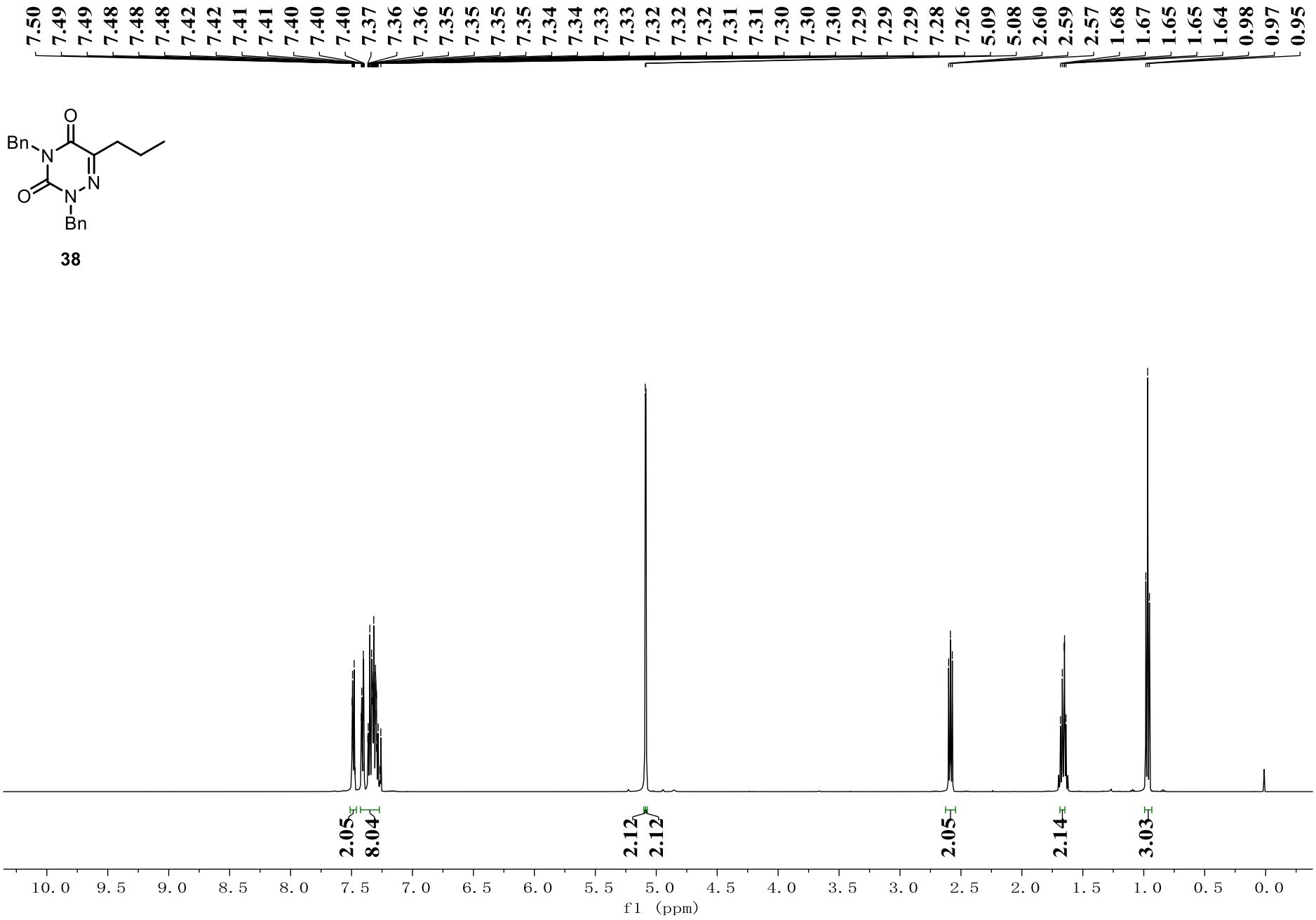
37

^{13}C NMR (125 MHz, $CDCl_3$) for 37

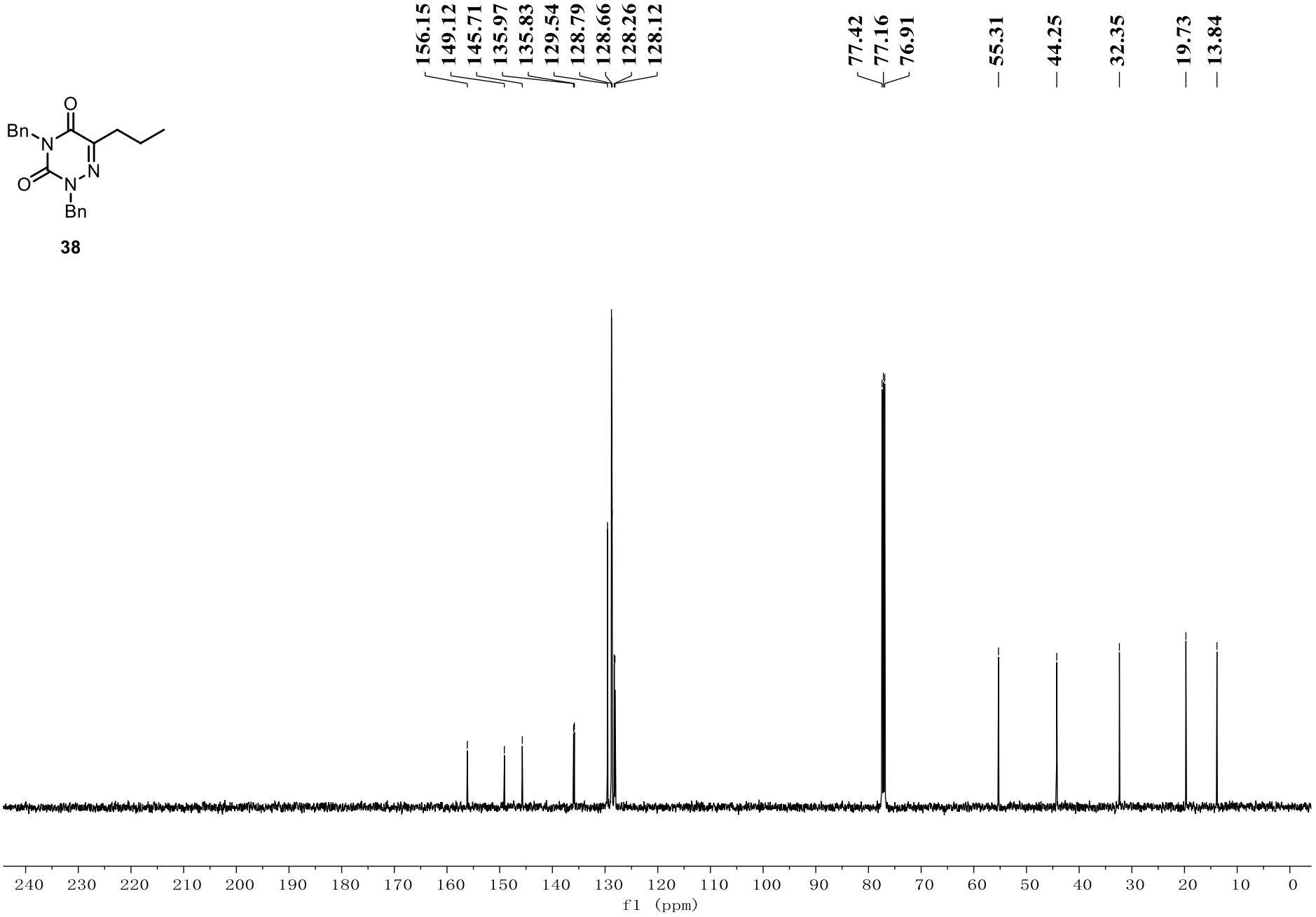


37

^{1}H NMR (500 MHz, $CDCl_3$) for 38

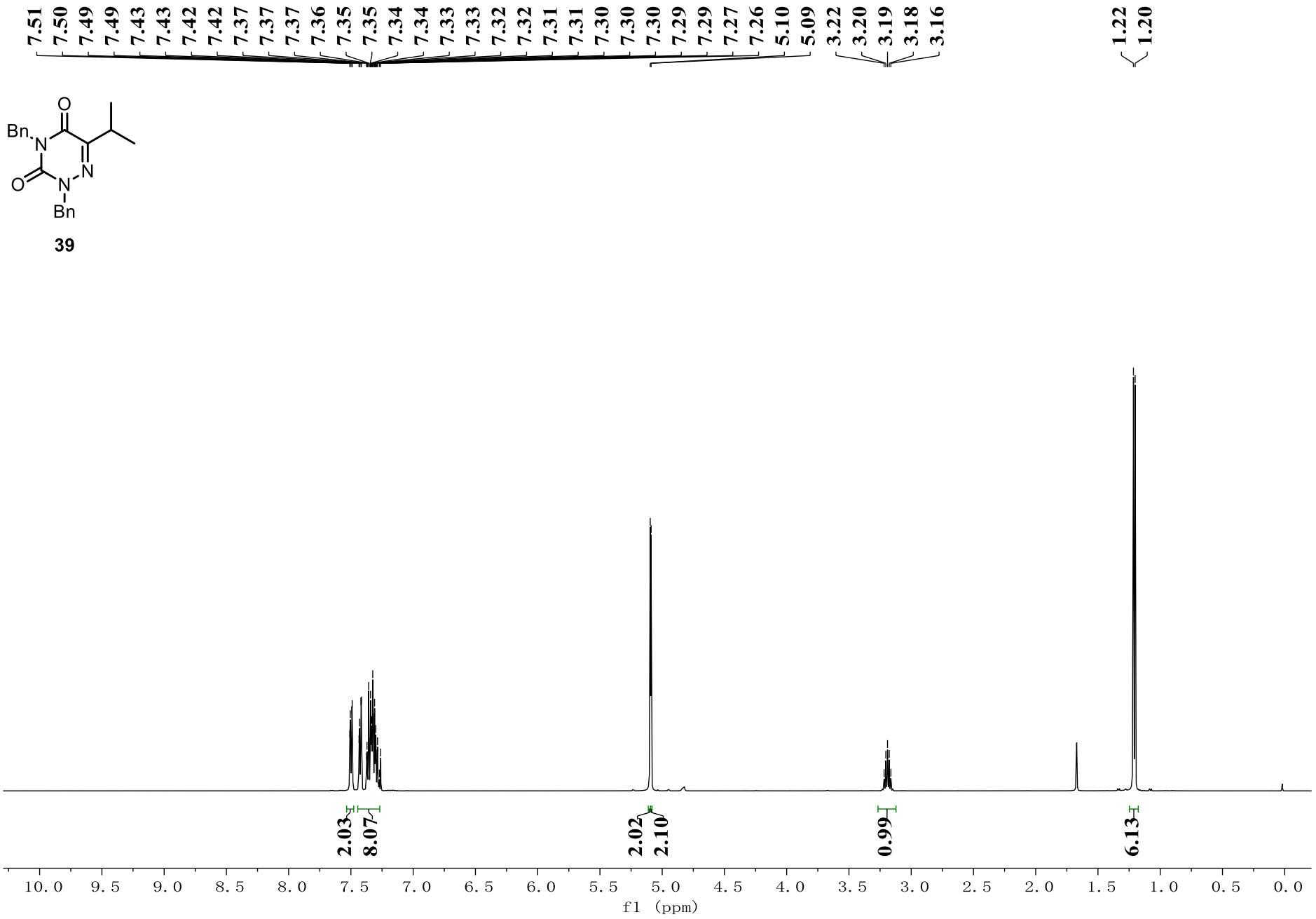


^{13}C NMR (125 MHz, $CDCl_3$) for 38



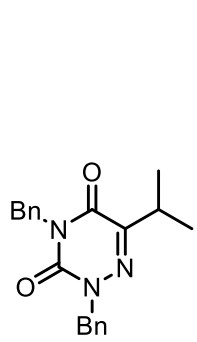
38

^{1}H NMR (500 MHz, $CDCl_3$) for 39



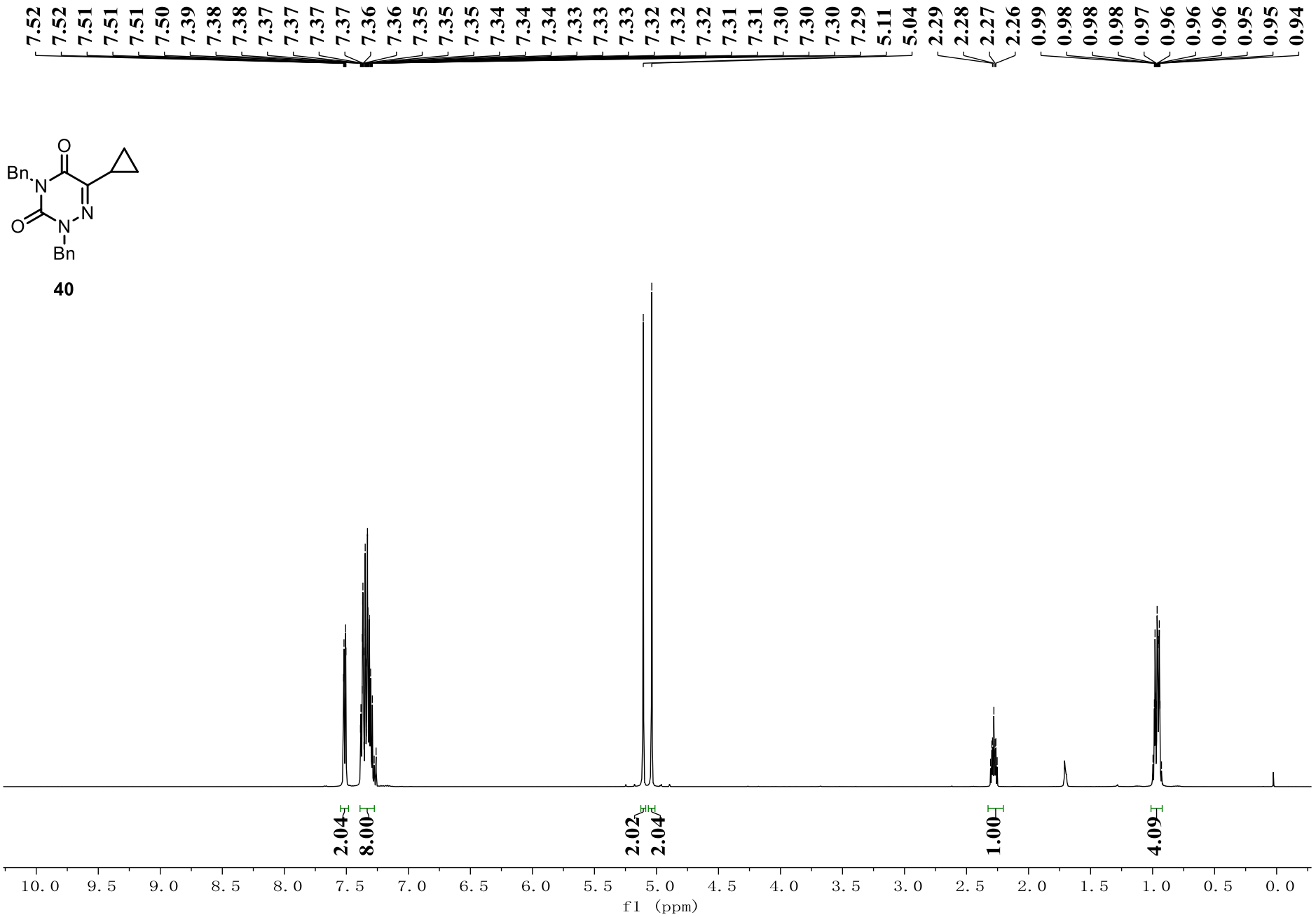
39

^{13}C NMR (125 MHz, $CDCl_3$) for 39



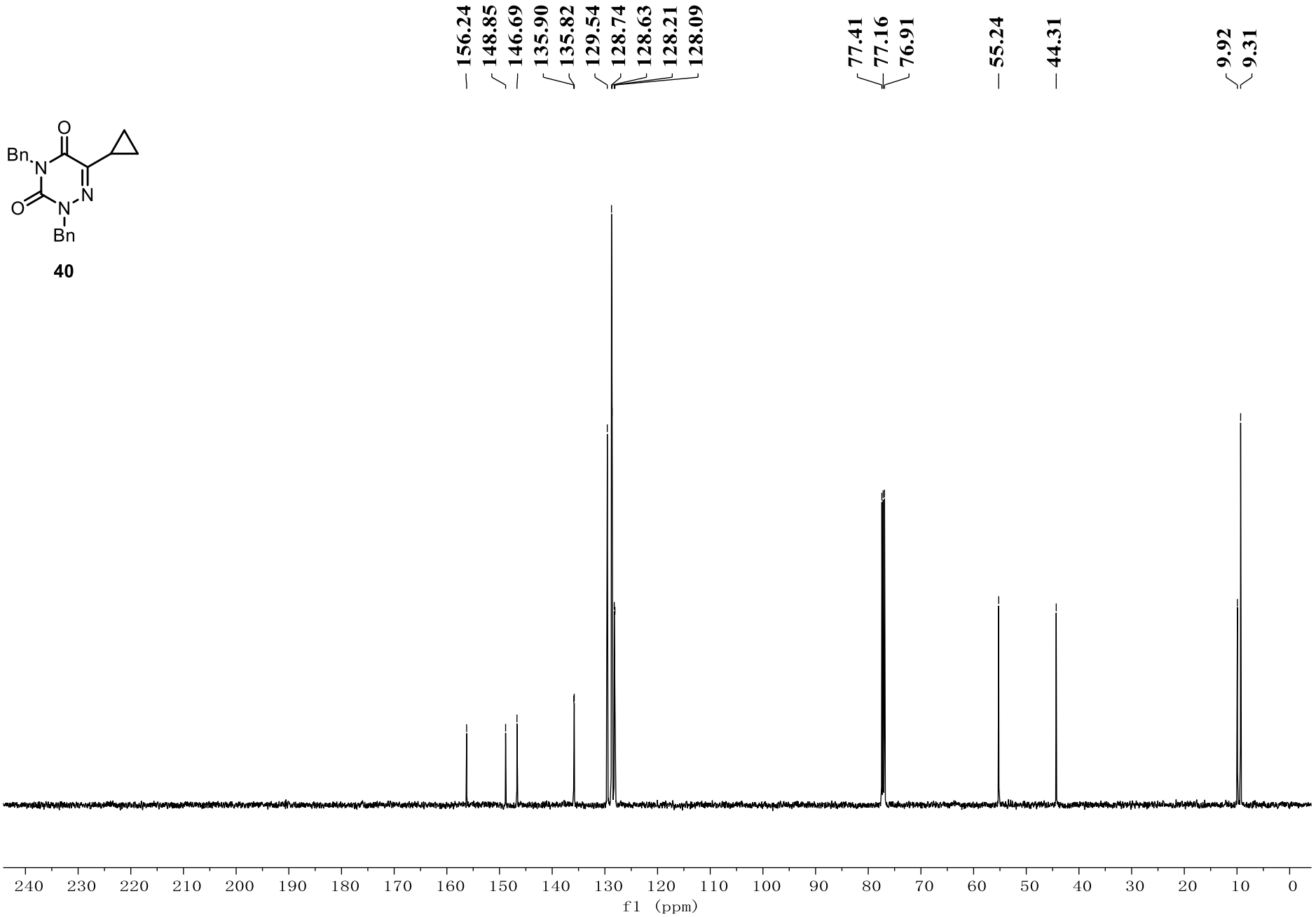
39

^{1}H NMR (500 MHz, $CDCl_3$) for 40



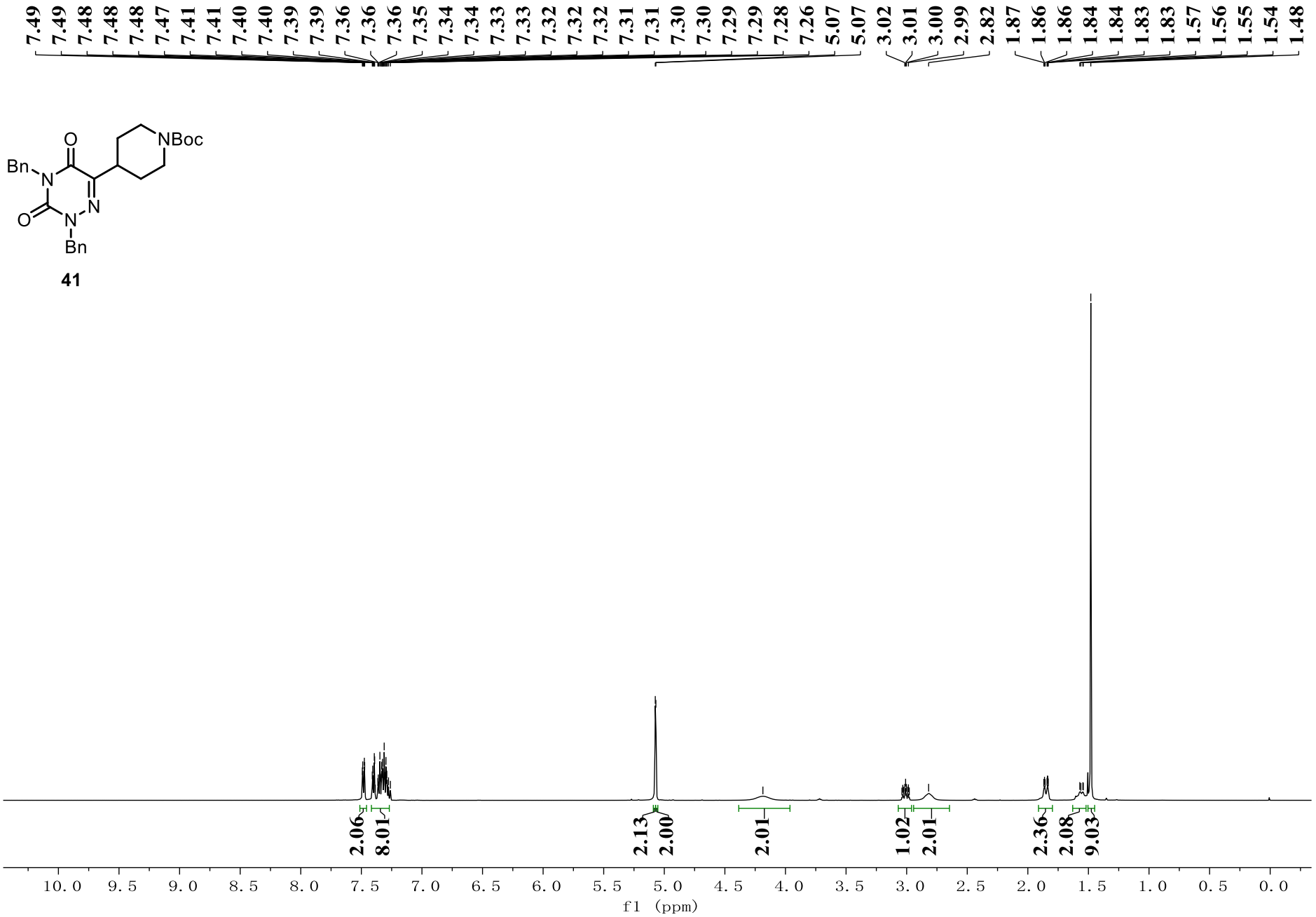
40

^{13}C NMR (125 MHz, $CDCl_3$) for 40



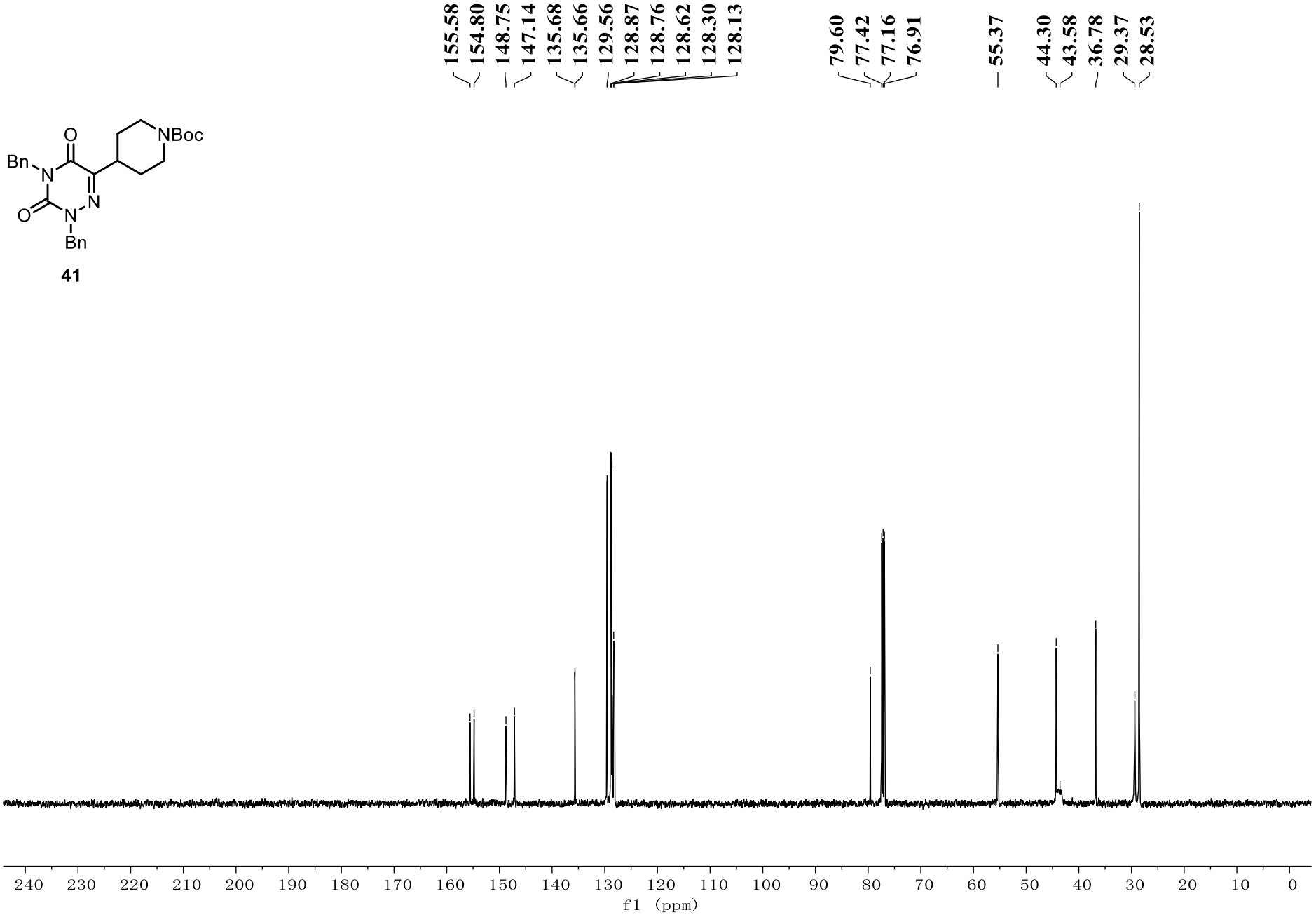
40

^{1}H NMR (500 MHz, $CDCl_3$) for 41



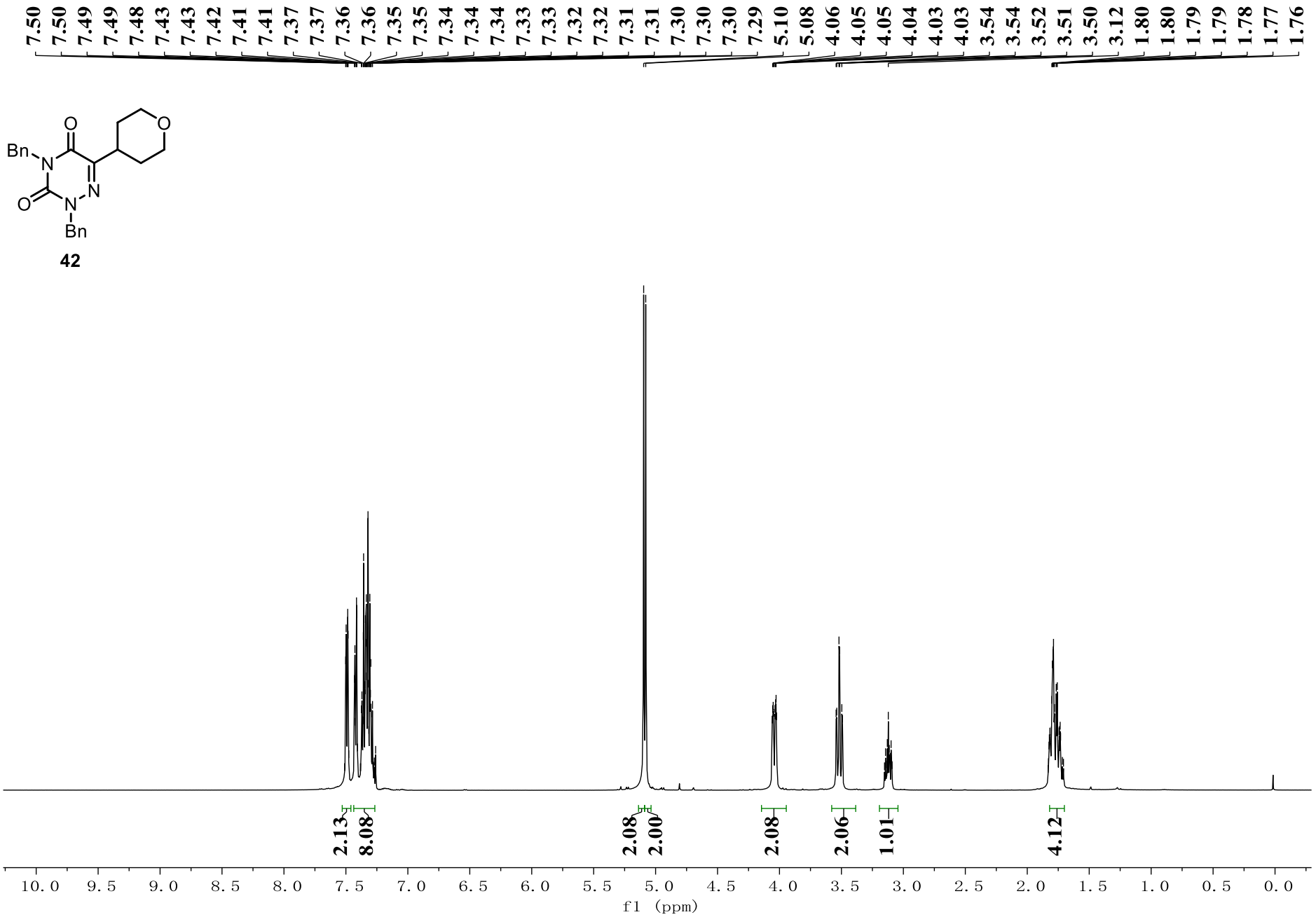
41

^{13}C NMR (125 MHz, $CDCl_3$) for 41

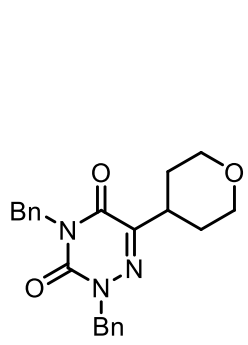


41

^{1}H NMR (500 MHz, $CDCl_3$) for 42

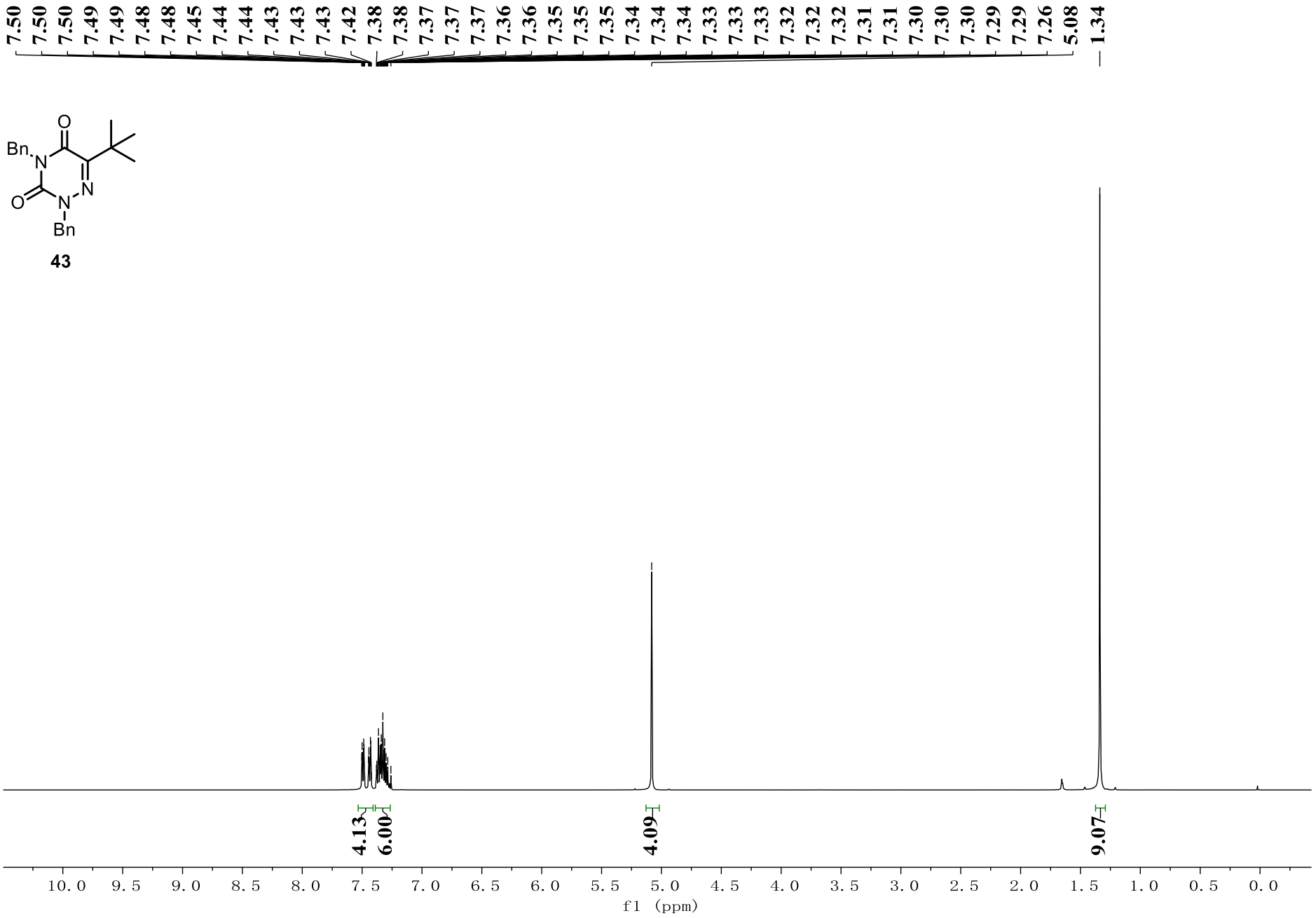


^{13}C NMR (125 MHz, $CDCl_3$) for 42



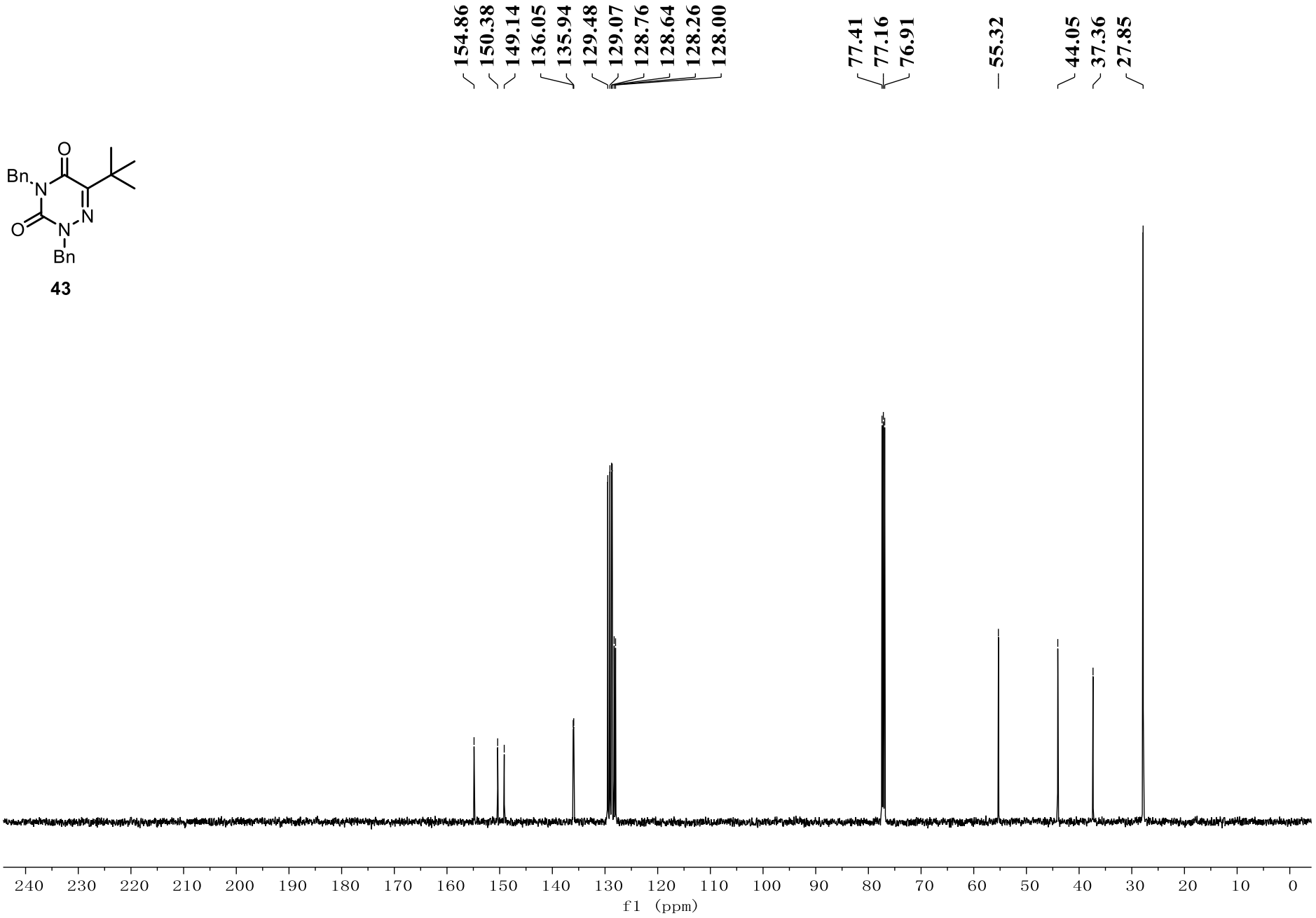
42

^{1}H NMR (500 MHz, $CDCl_3$) for 43



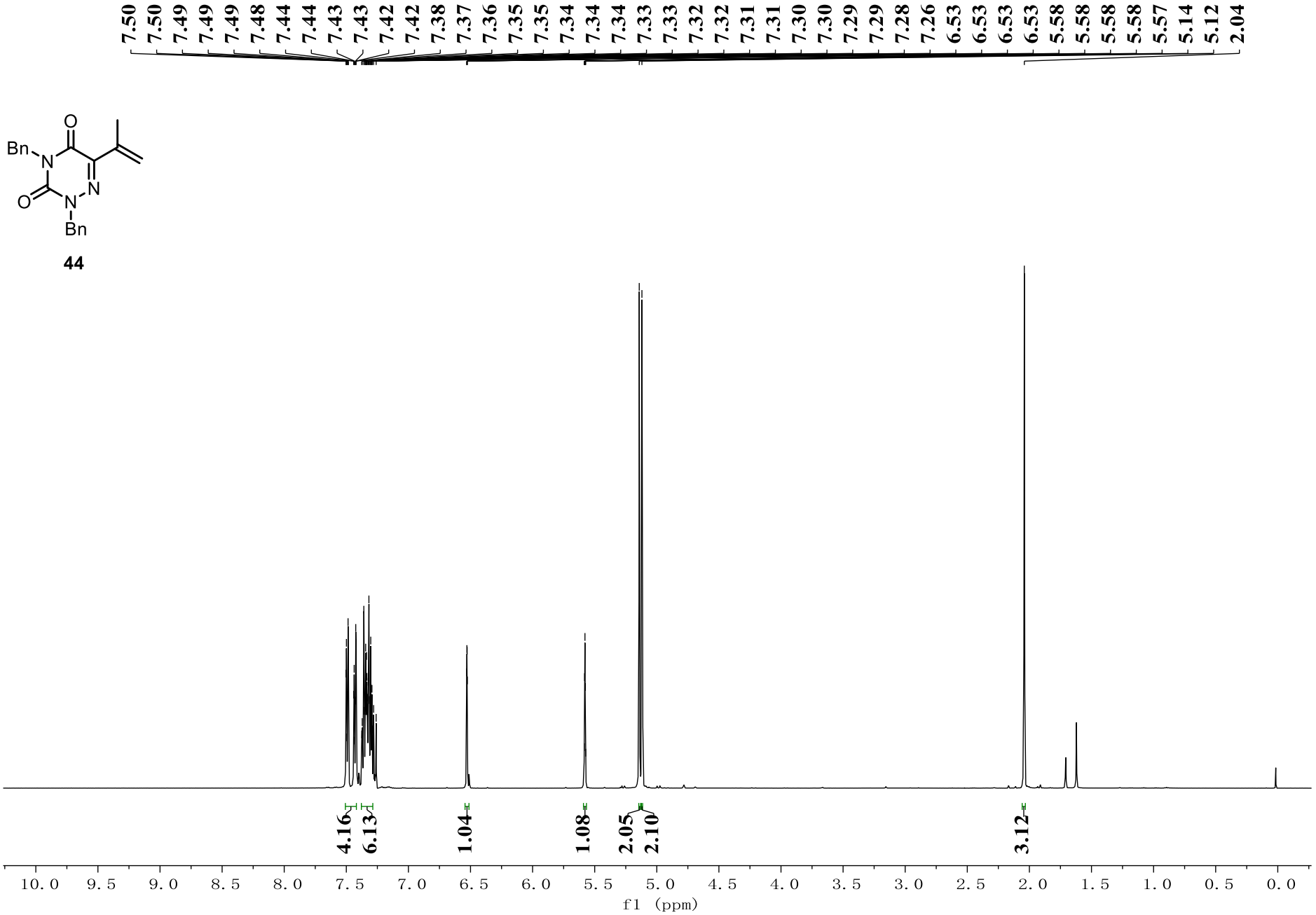
43

^{13}C NMR (125 MHz, $CDCl_3$) for 43



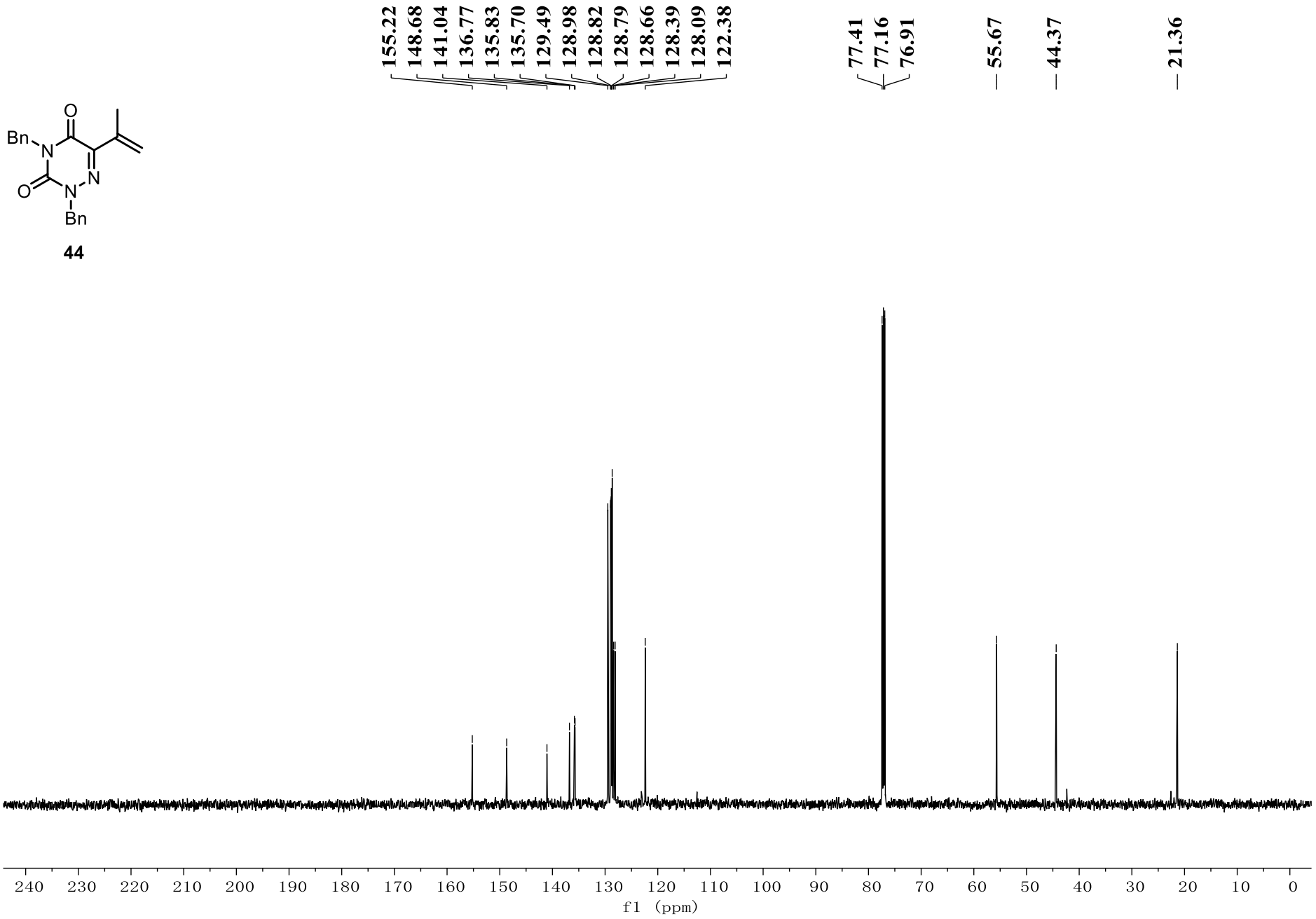
43

^{1}H NMR (500 MHz, $CDCl_3$) for 44



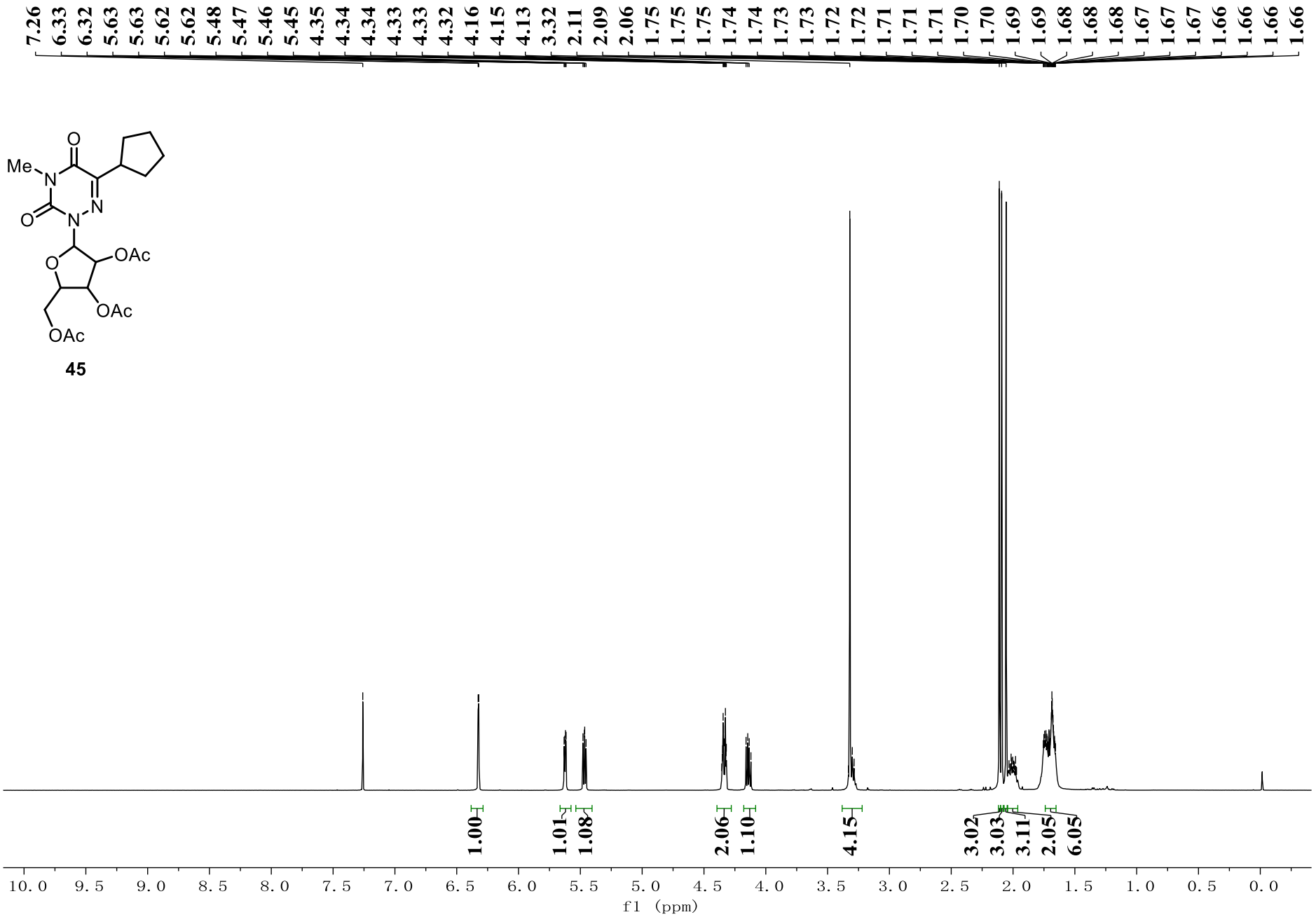
44

^{13}C NMR (125 MHz, $CDCl_3$) for 44



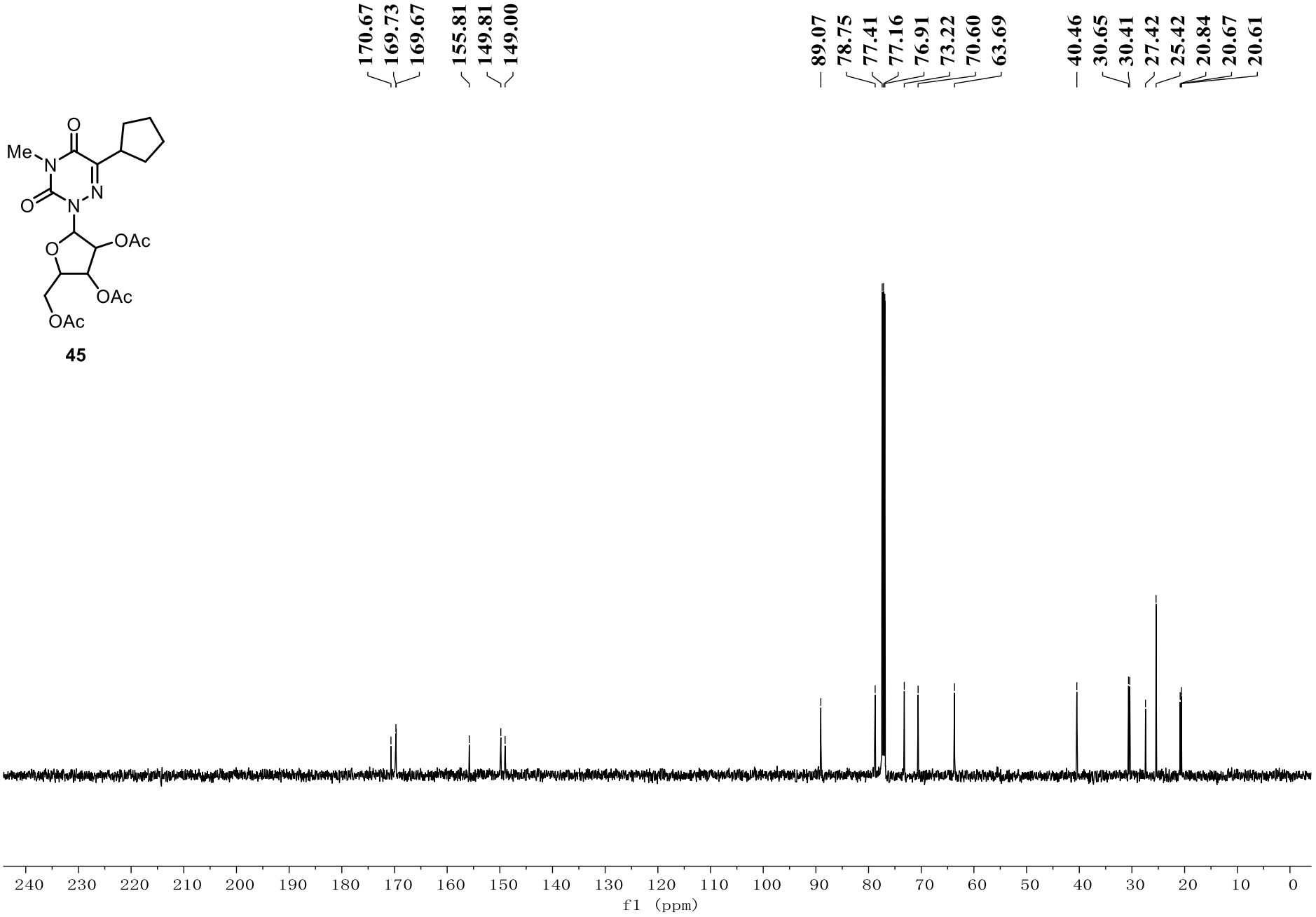
44

^{1}H NMR (500 MHz, $CDCl_3$) for 45



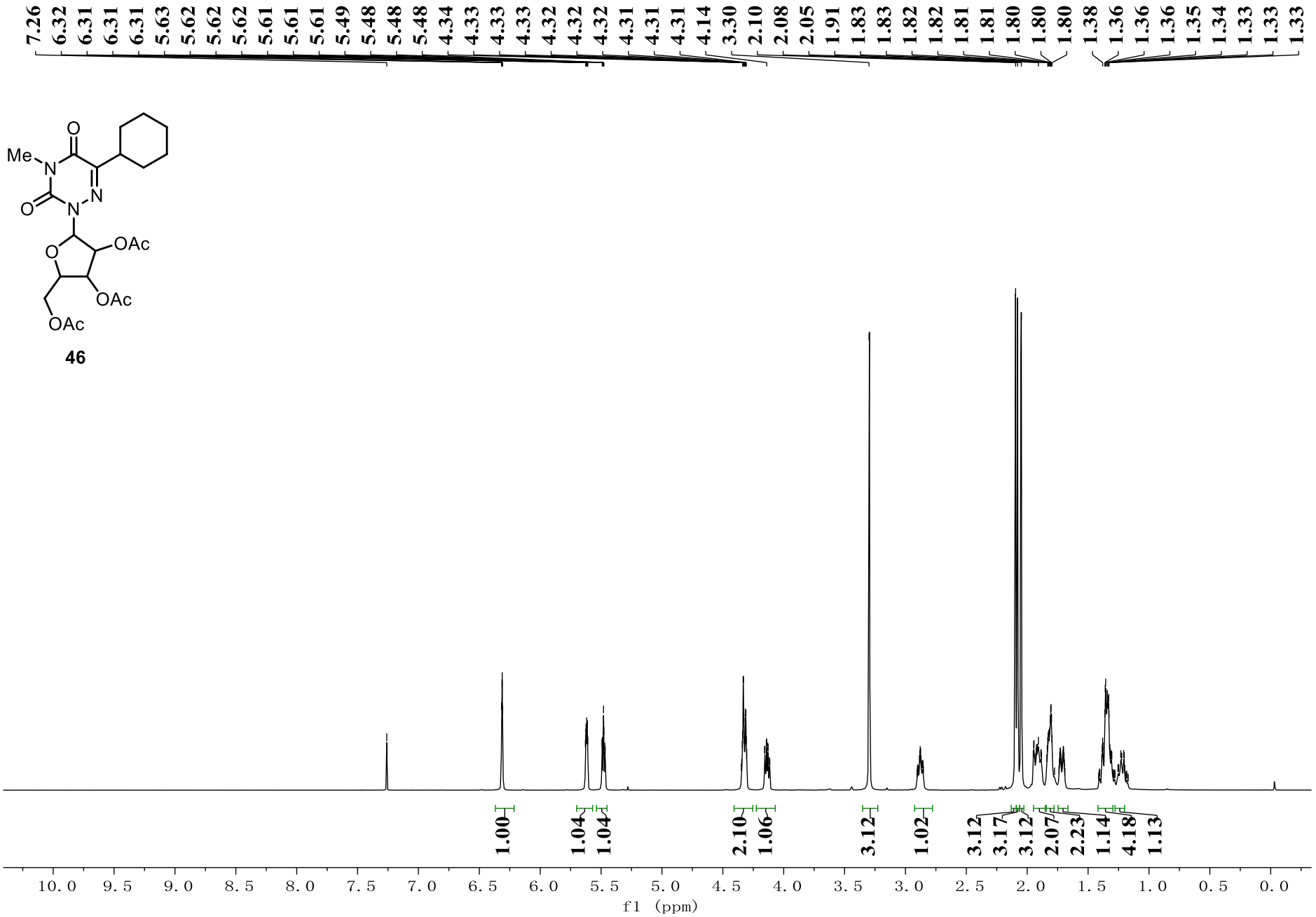
45

^{13}C NMR (125 MHz, $CDCl_3$) for 45



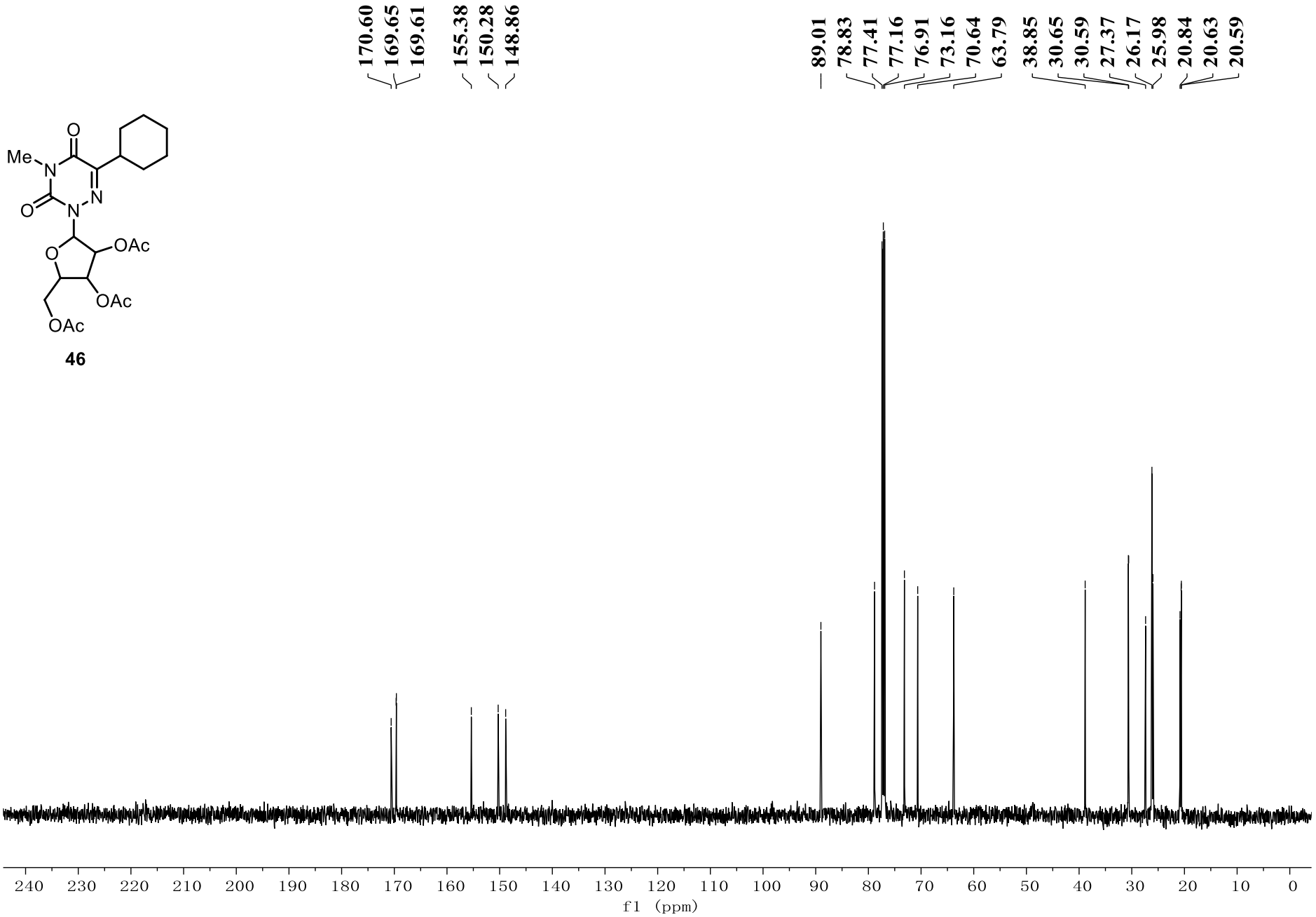
45

^{1}H NMR (500 MHz, $CDCl_3$) for 46



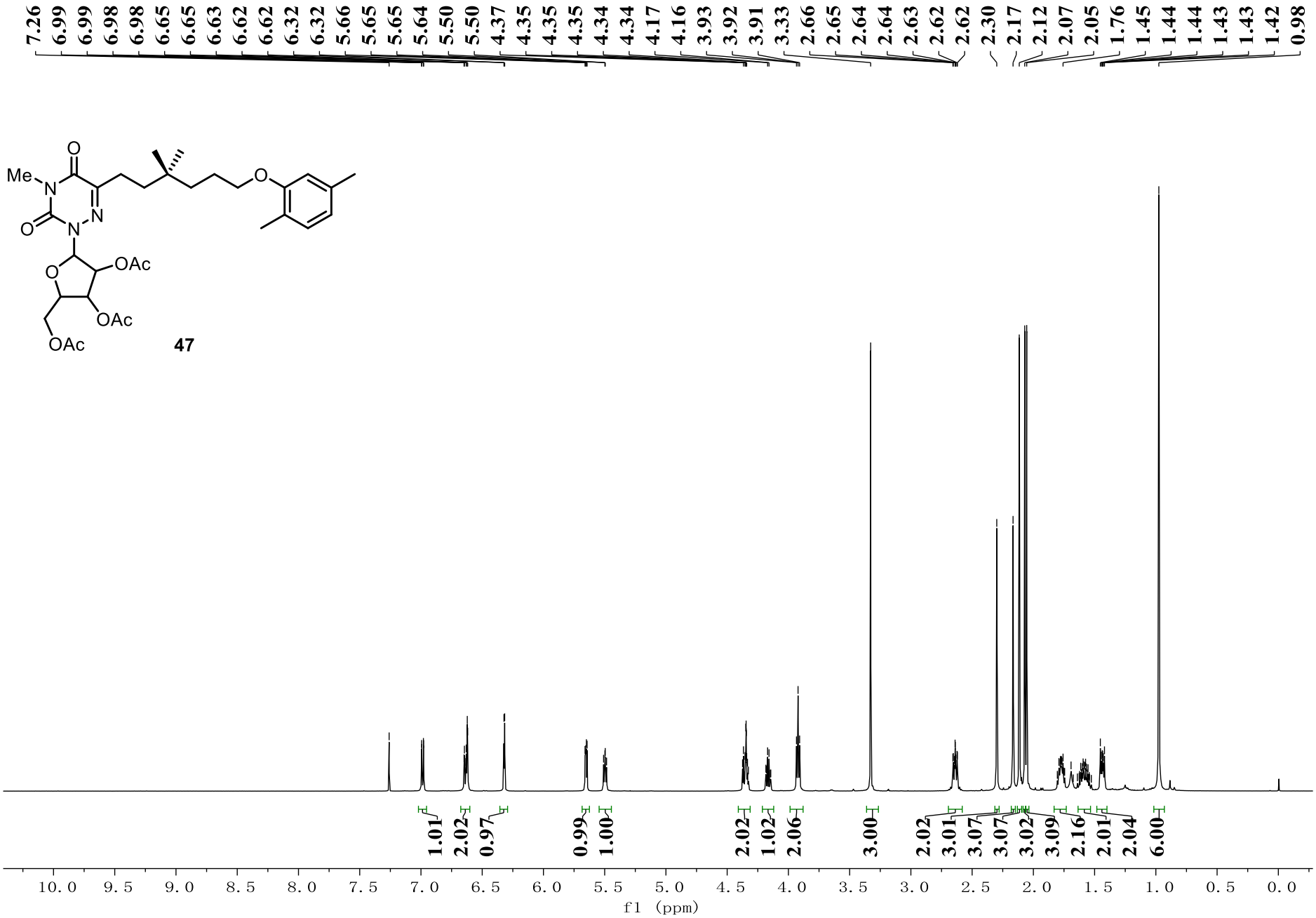
46

^{13}C NMR (125 MHz, $CDCl_3$) for 46

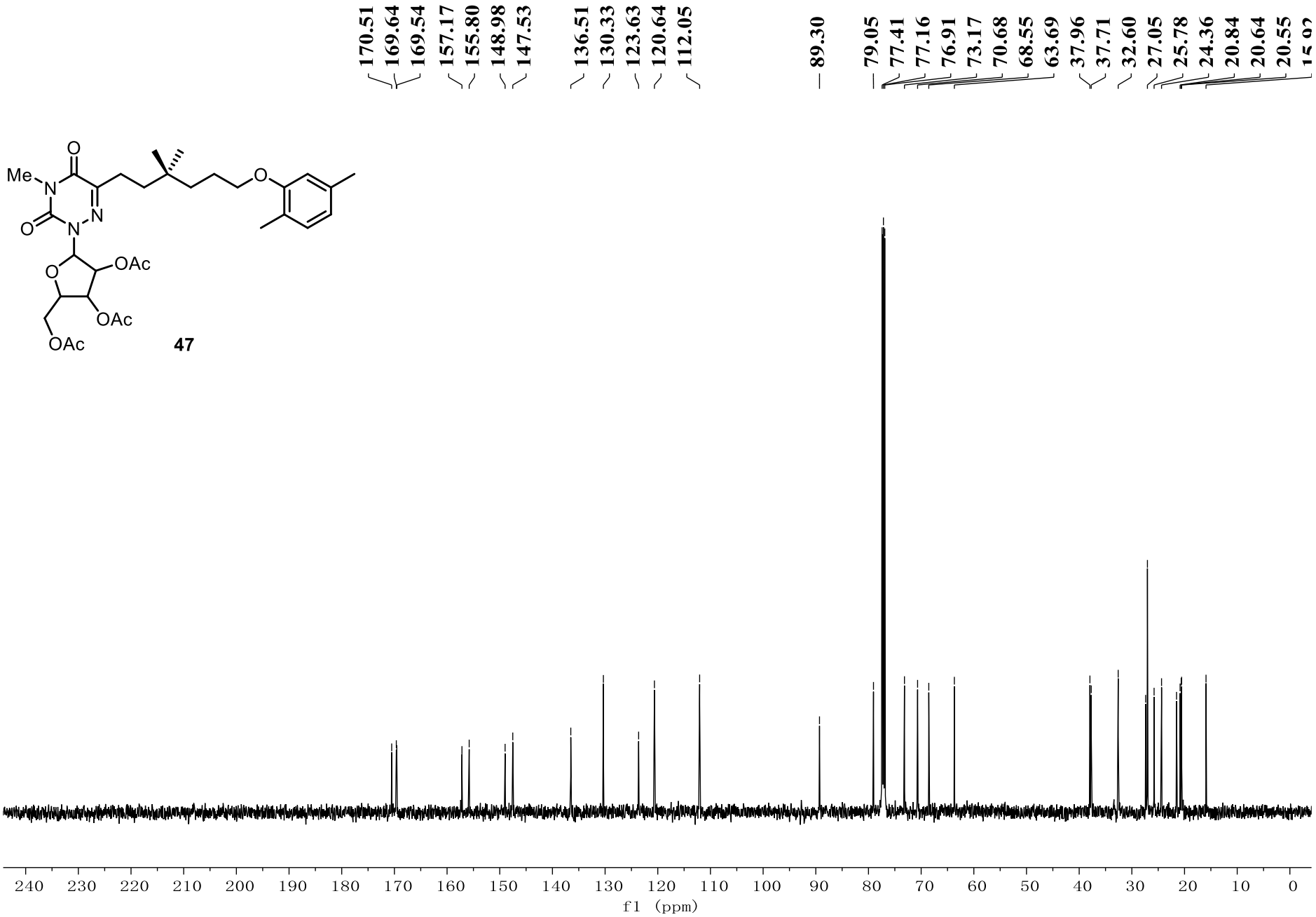


46

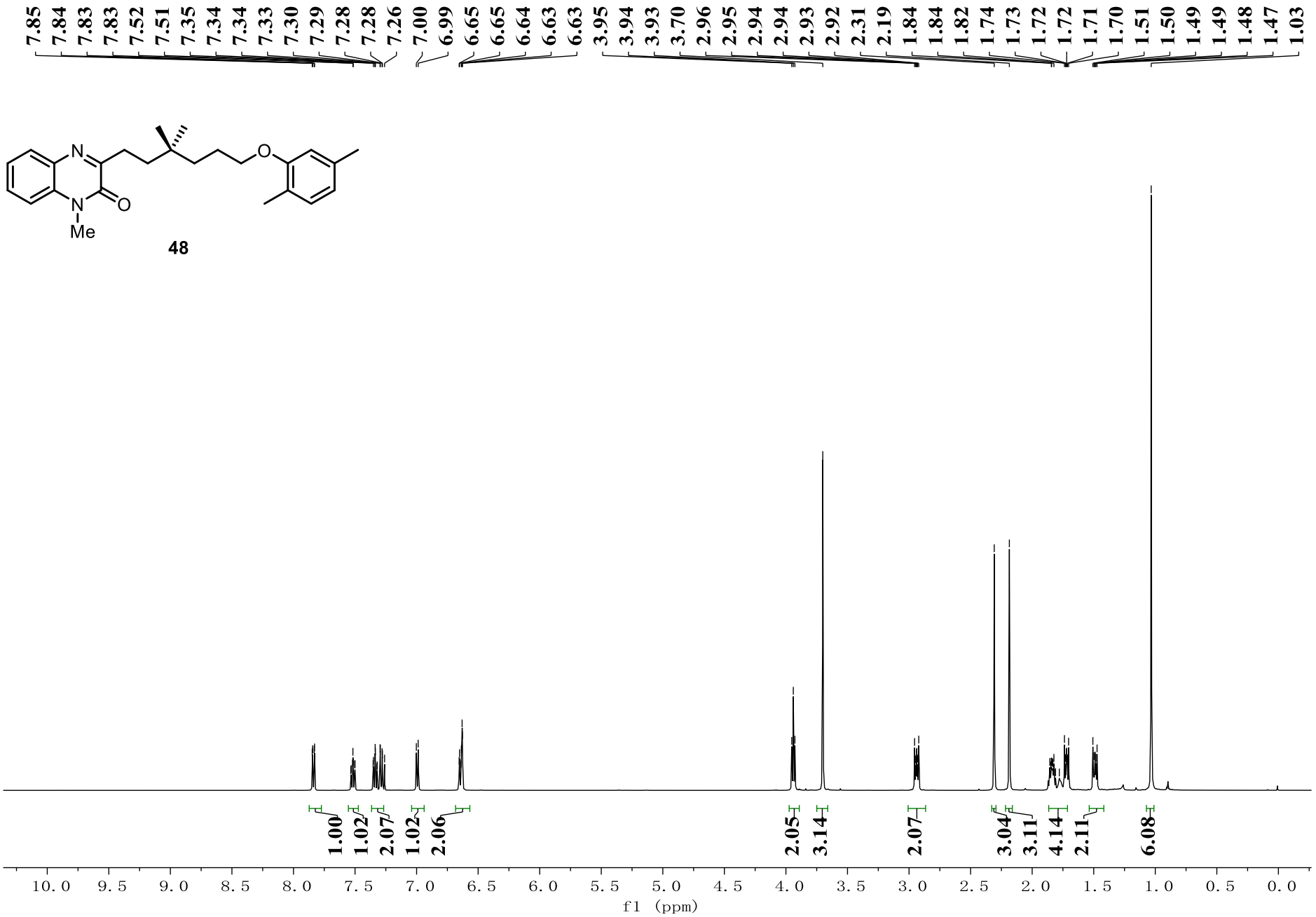
^{1}H NMR (500 MHz, $CDCl_3$) for 47



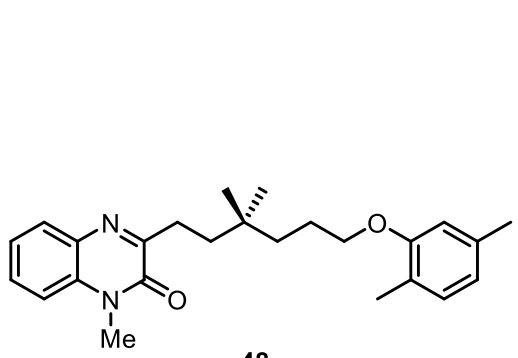
^{13}C NMR (125 MHz, $CDCl_3$) for 47



^{1}H NMR (500 MHz, $CDCl_3$) for 48

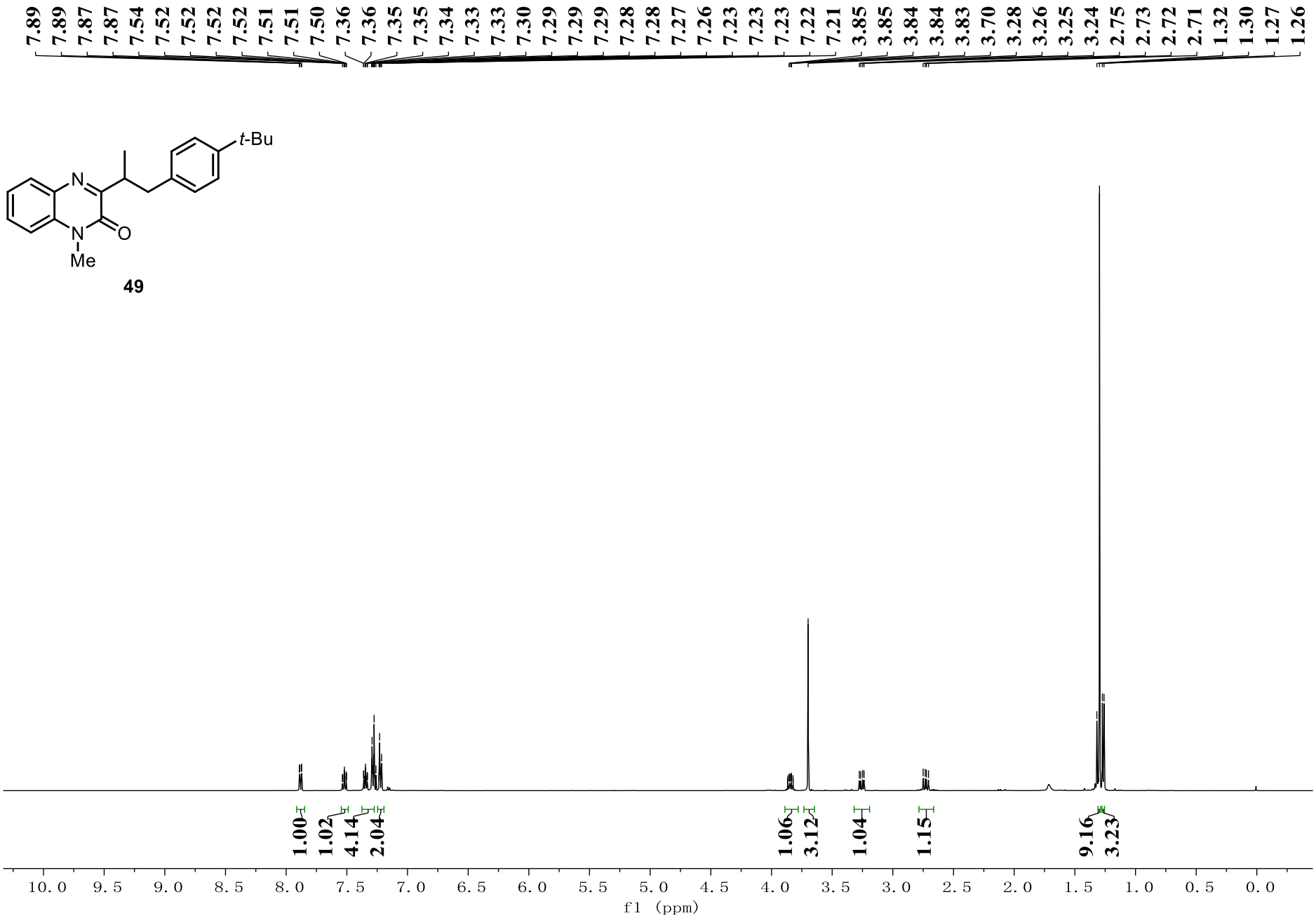


^{13}C NMR (125 MHz, $CDCl_3$) for 48



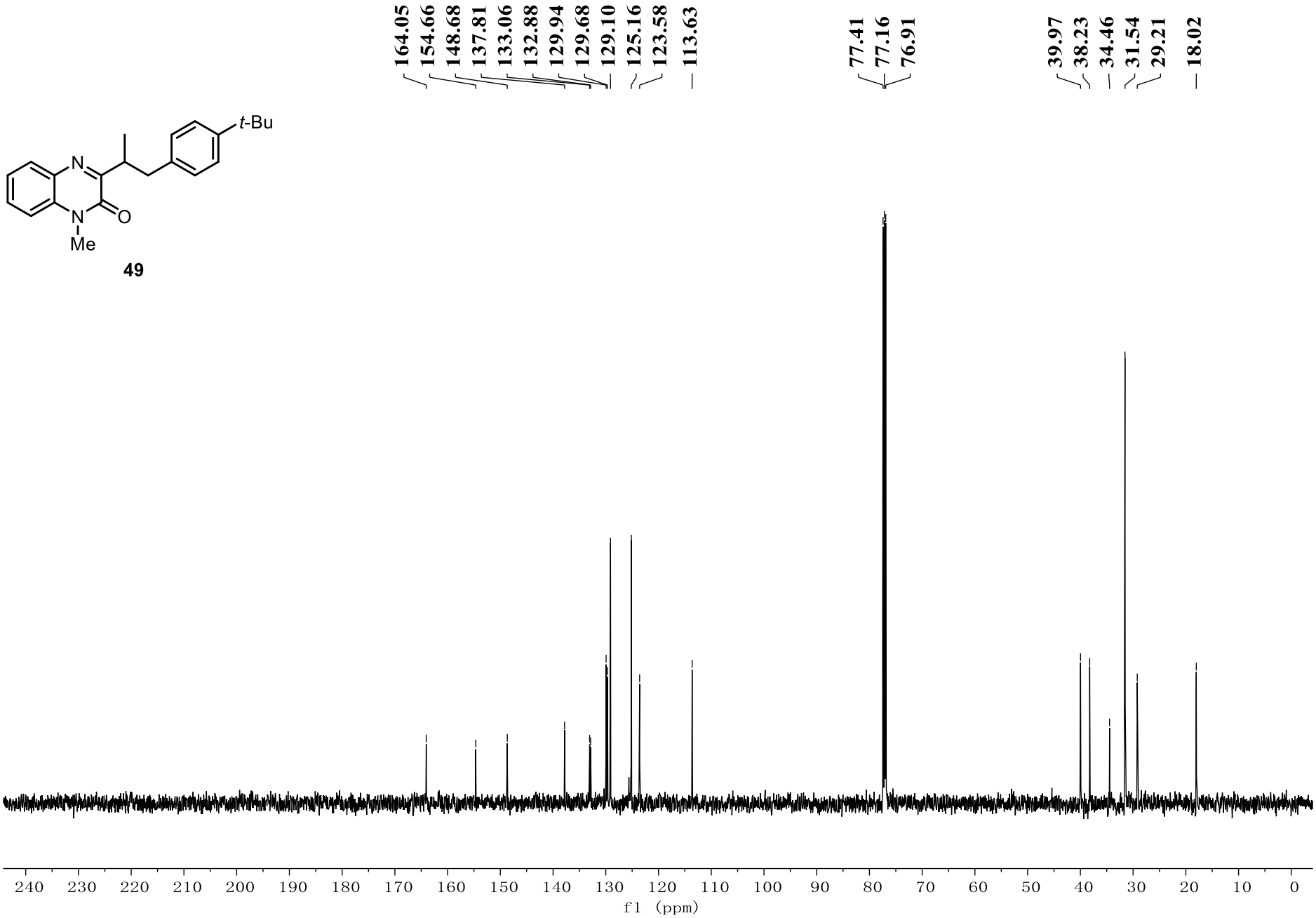
48

^{1}H NMR (500 MHz, $CDCl_3$) for 49

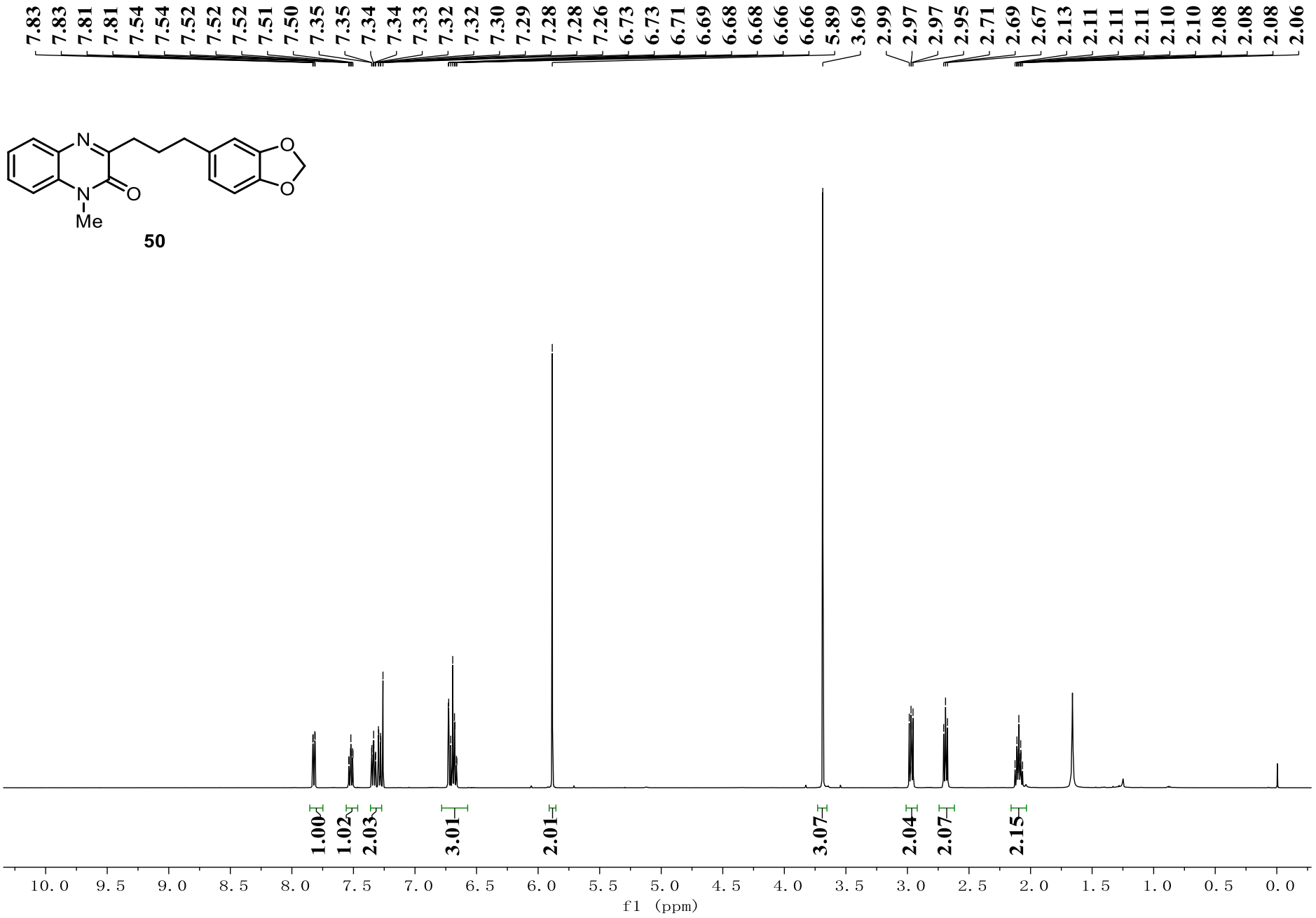


49

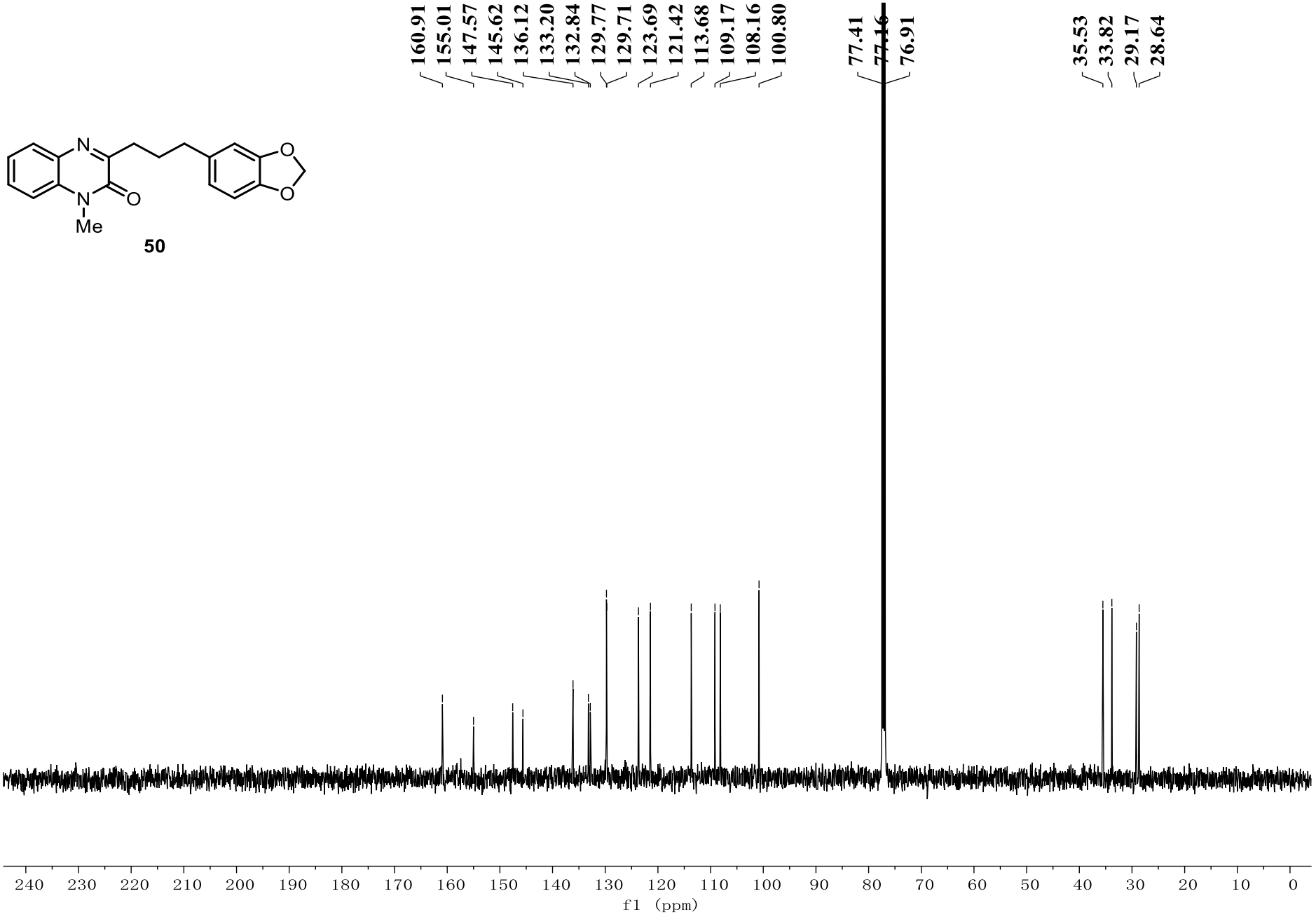
^{13}C NMR (125 MHz, $CDCl_3$) for 49



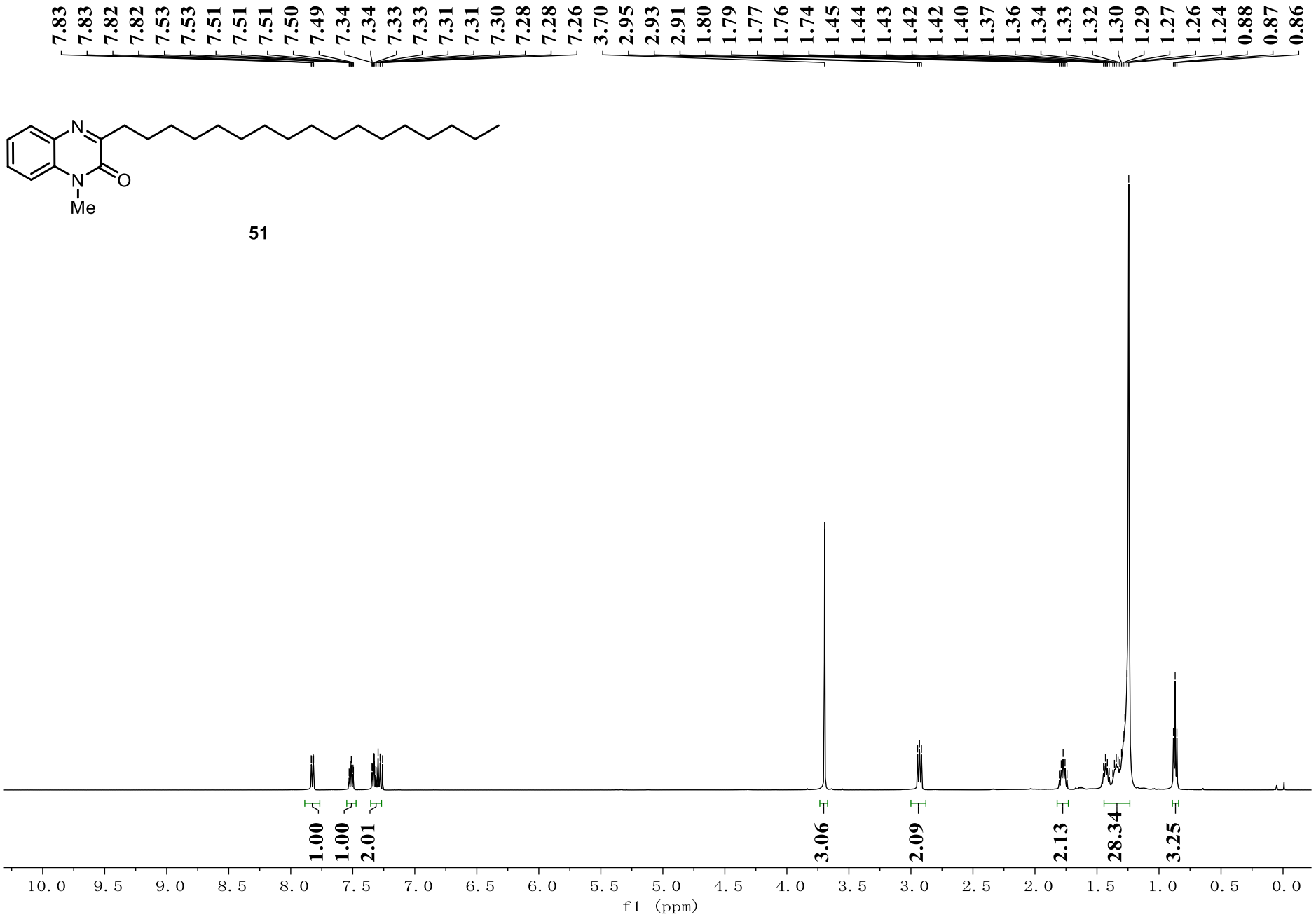
^{1}H NMR (500 MHz, $CDCl_3$) for 50



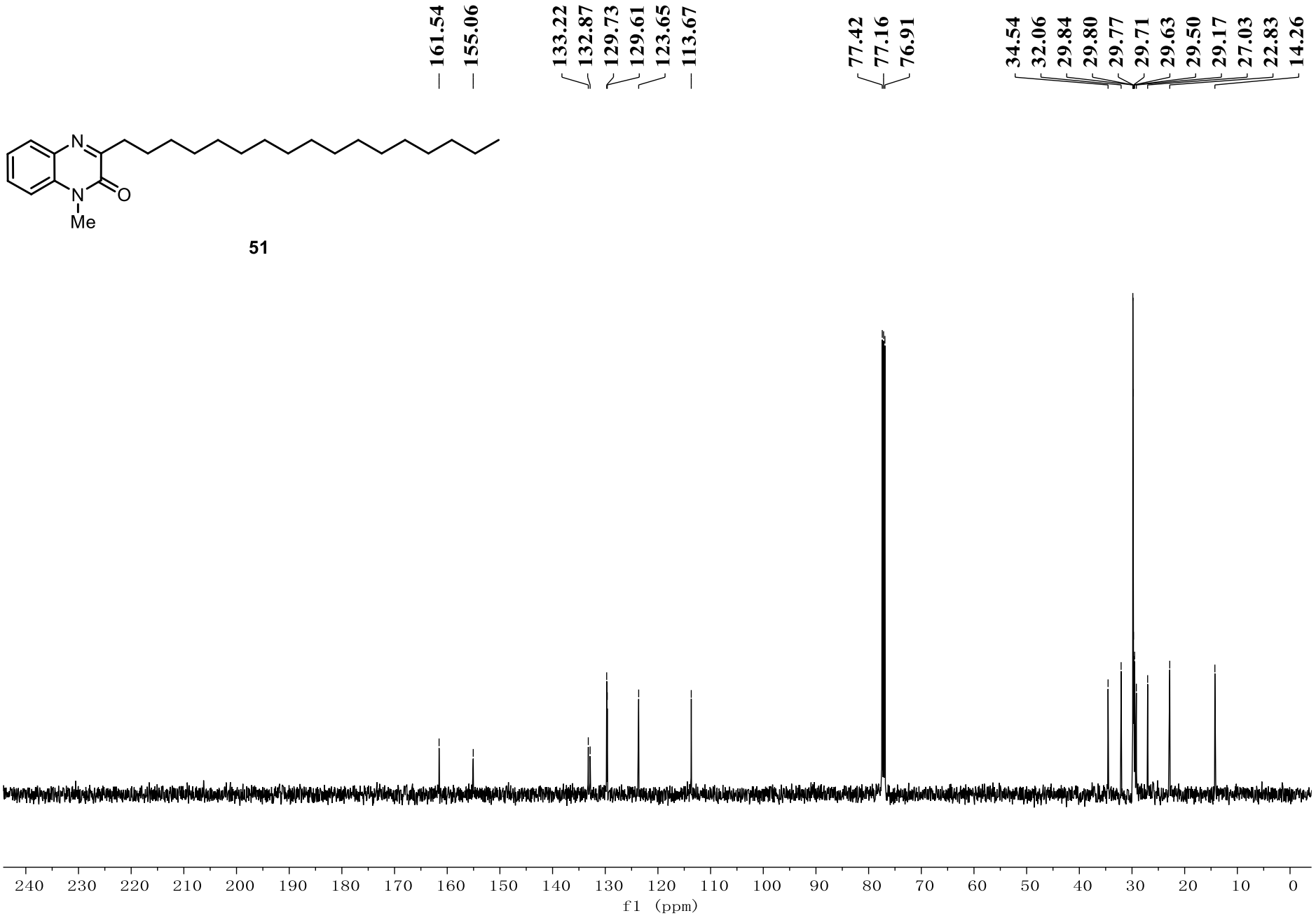
^{13}C NMR (125 MHz, $CDCl_3$) for 50



^{1}H NMR (500 MHz, $CDCl_3$) for 51

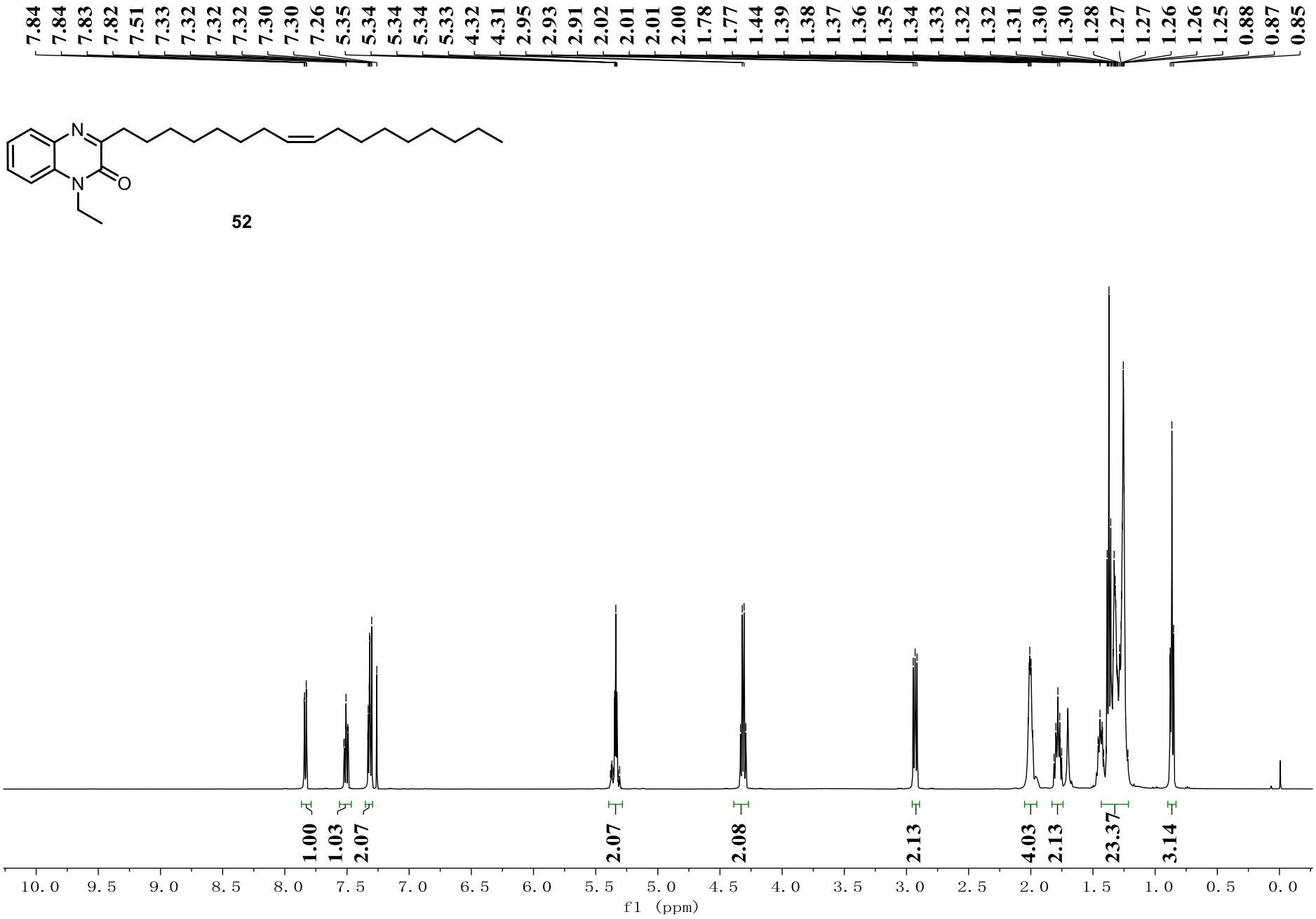


^{13}C NMR (125 MHz, $CDCl_3$) for 51

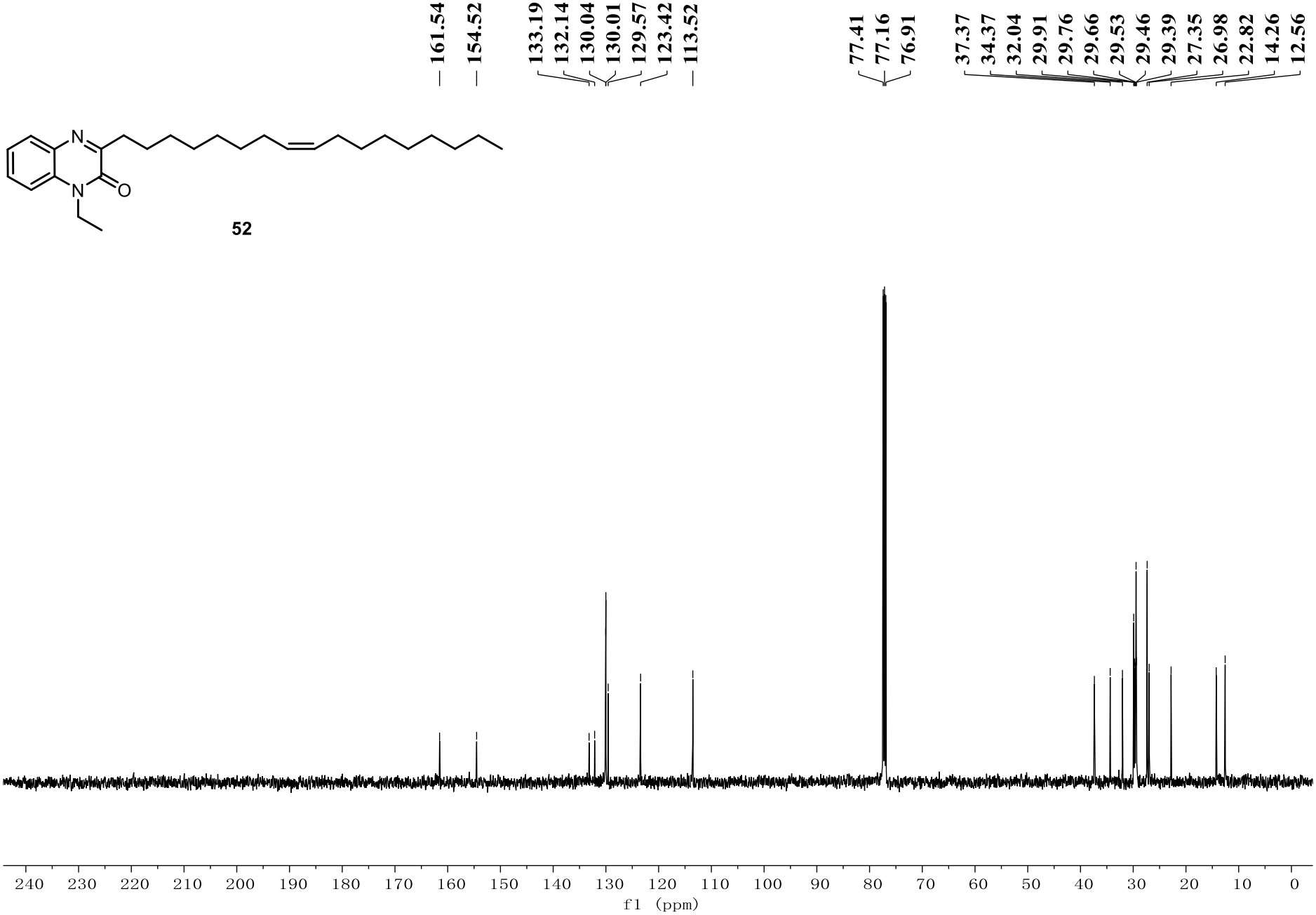


51

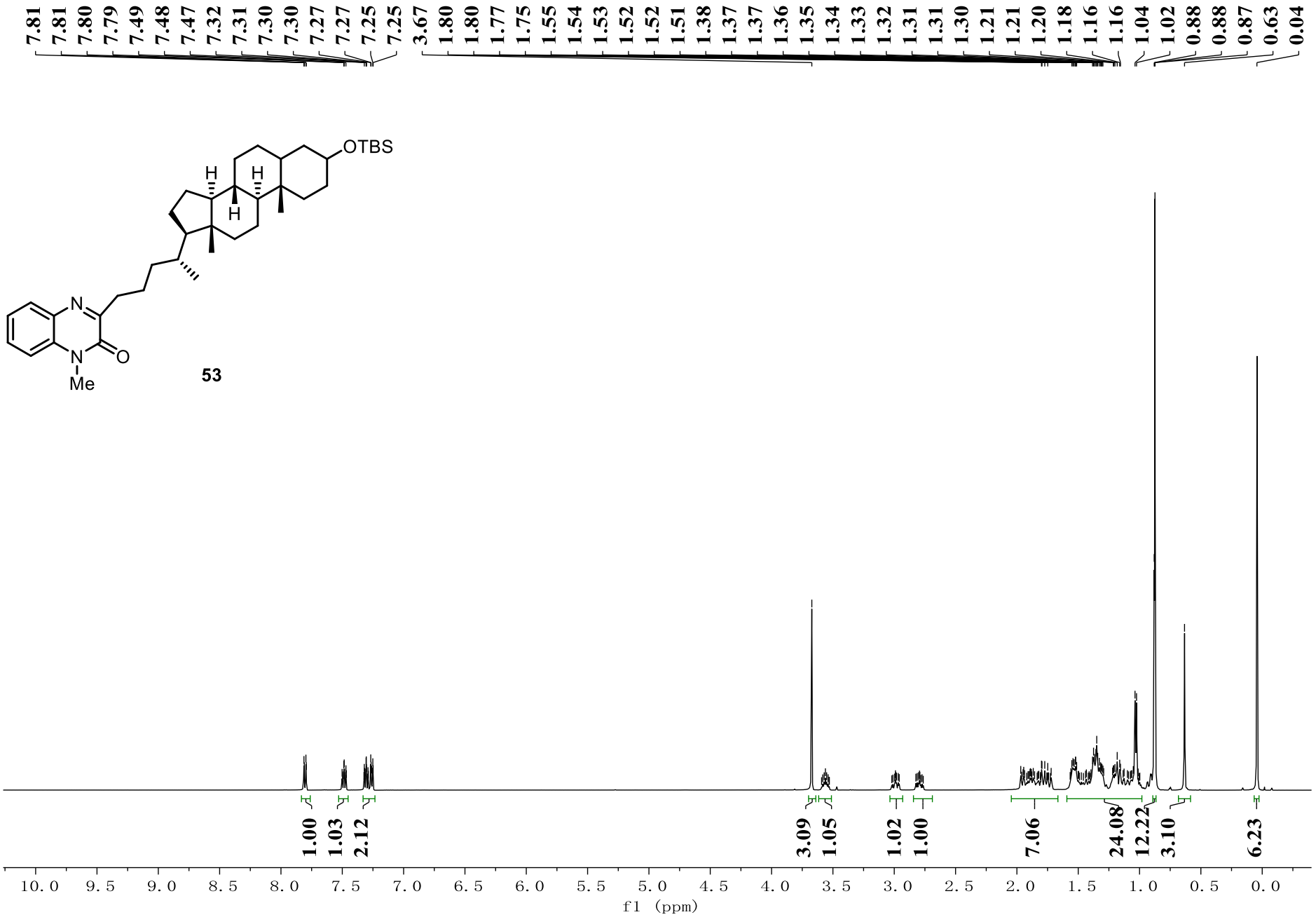
^{1}H NMR (500 MHz, $CDCl_3$) for 52



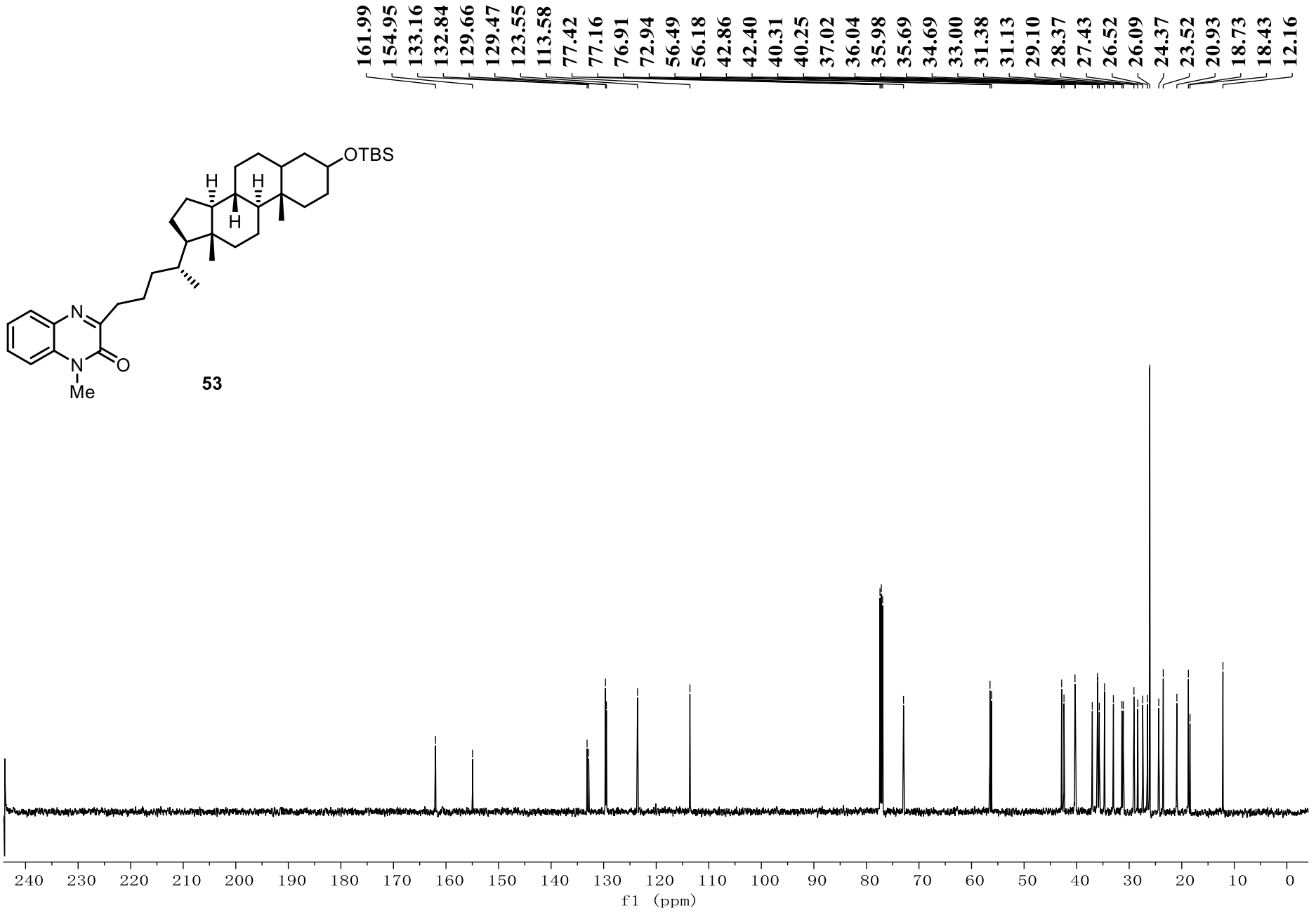
^{13}C NMR (125 MHz, $CDCl_3$) for 52



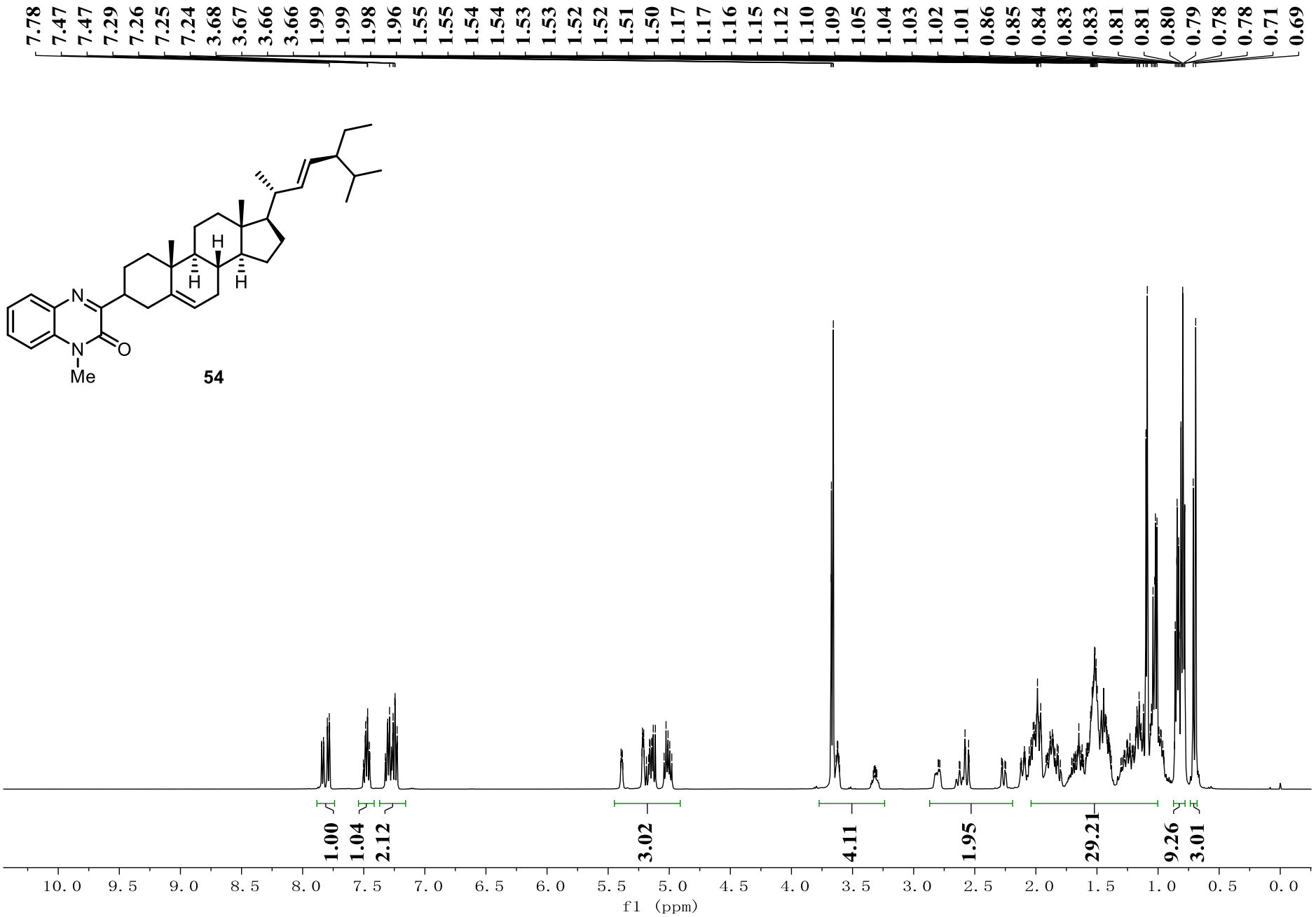
^{1}H NMR (500 MHz, $CDCl_3$) for 53



^{13}C NMR (125 MHz, $CDCl_3$) for 53

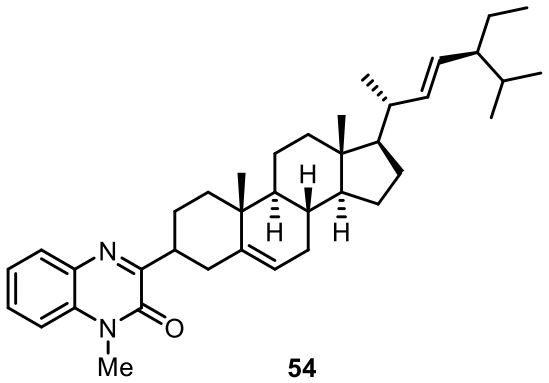


^{1}H NMR (500 MHz, $CDCl_3$) for 54

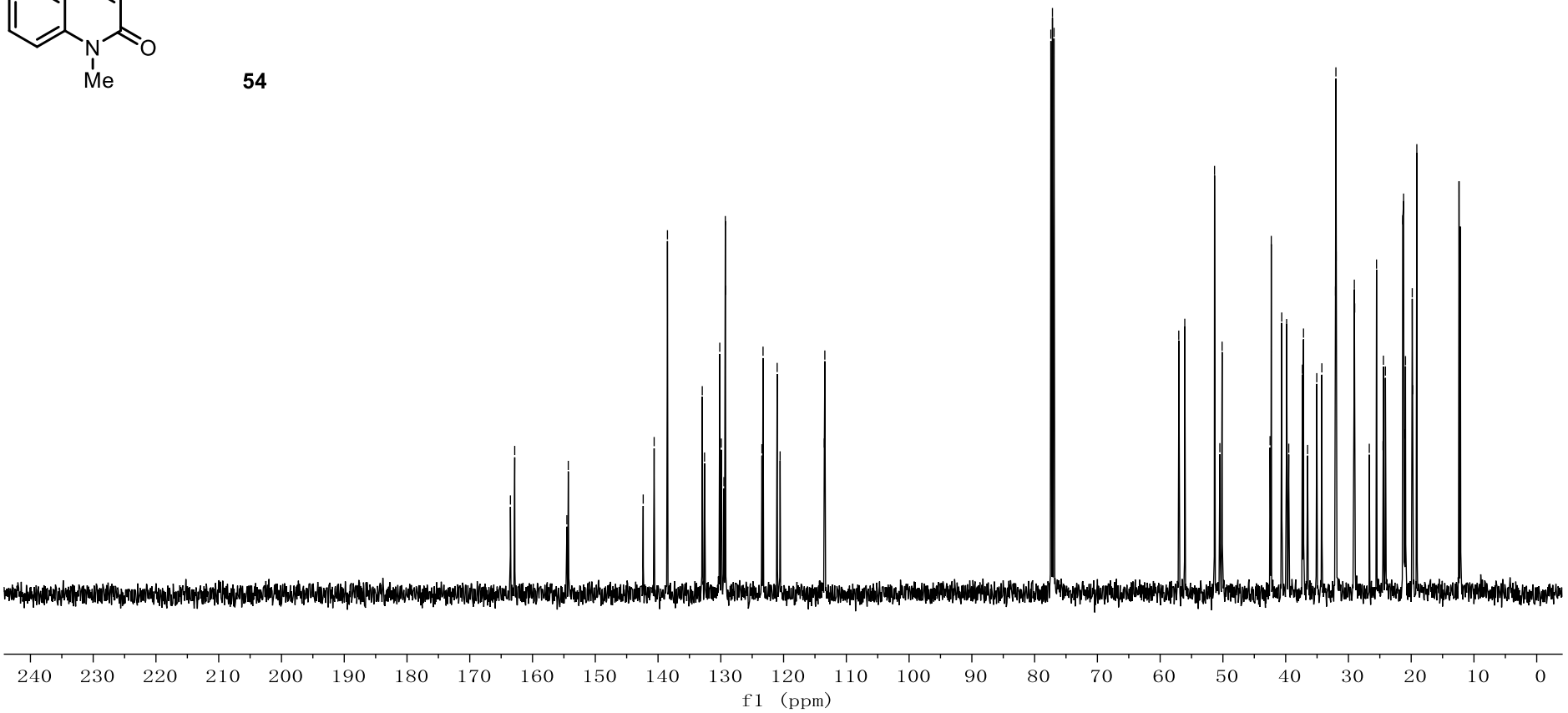


^{13}C NMR (125 MHz, $CDCl_3$) for 54

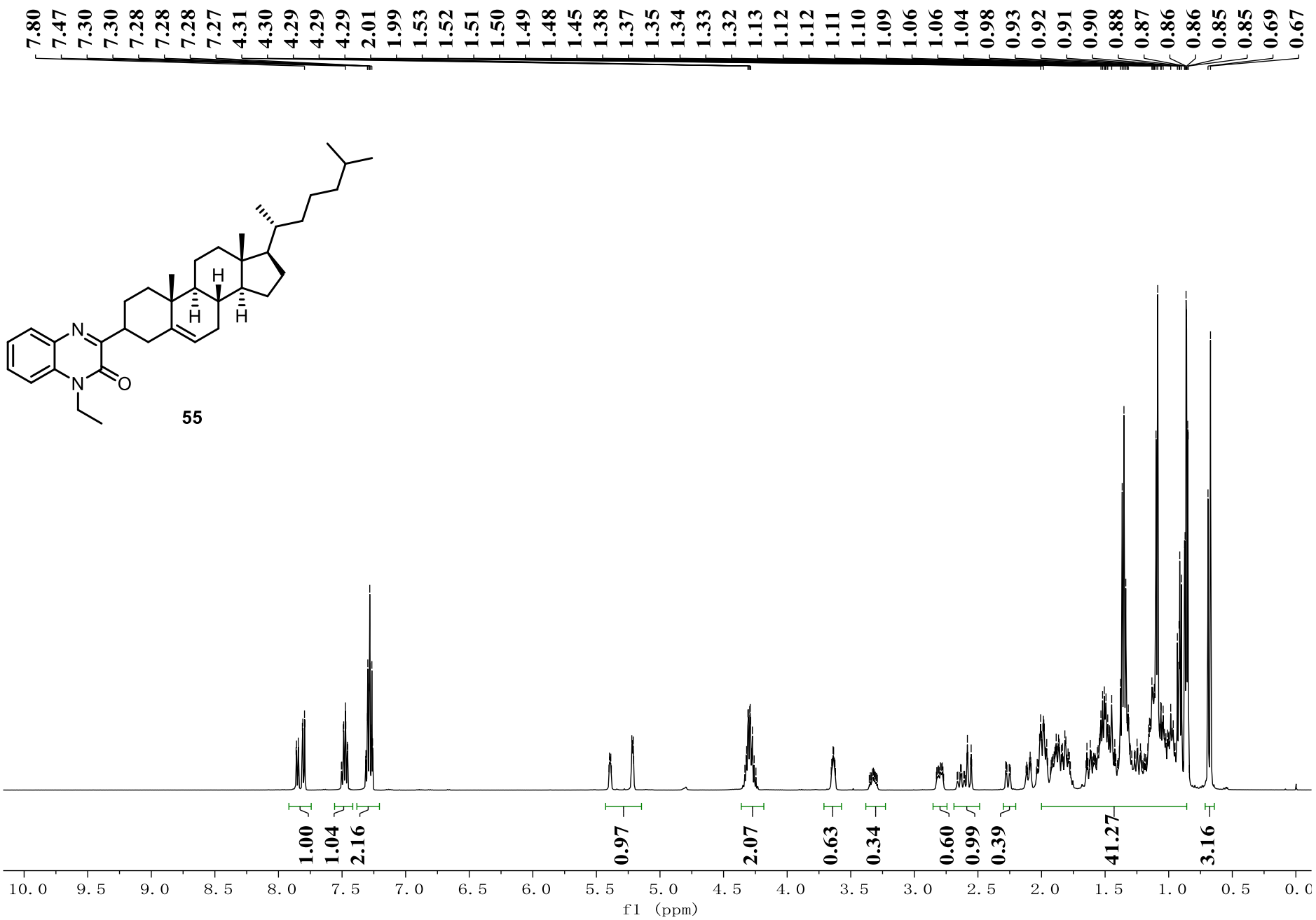
140.62 138.50 132.96 130.17 129.92 129.28 129.25 123.45 123.27 121.02 113.51 113.43 77.42 77.16 76.91 57.03 56.99 56.08 56.05 51.33 50.49 50.13 42.49 42.29 40.63 39.85 39.51 37.34 37.24 37.16 35.05 34.23 32.06 31.99 31.95 29.10 29.06 29.00 26.67 25.52 24.49 24.43 24.11 21.36 21.21 21.05 20.91 19.83 19.77 19.10



54

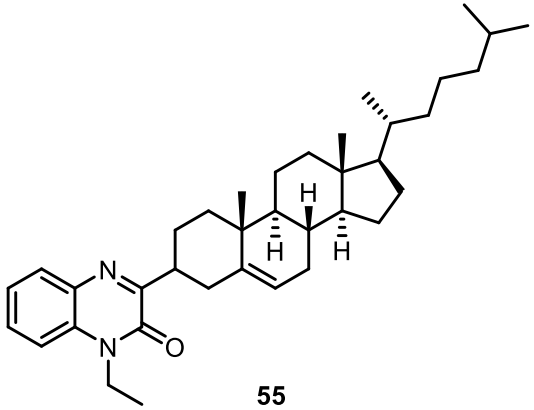


^{1}H NMR (500 MHz, $CDCl_3$) for 55

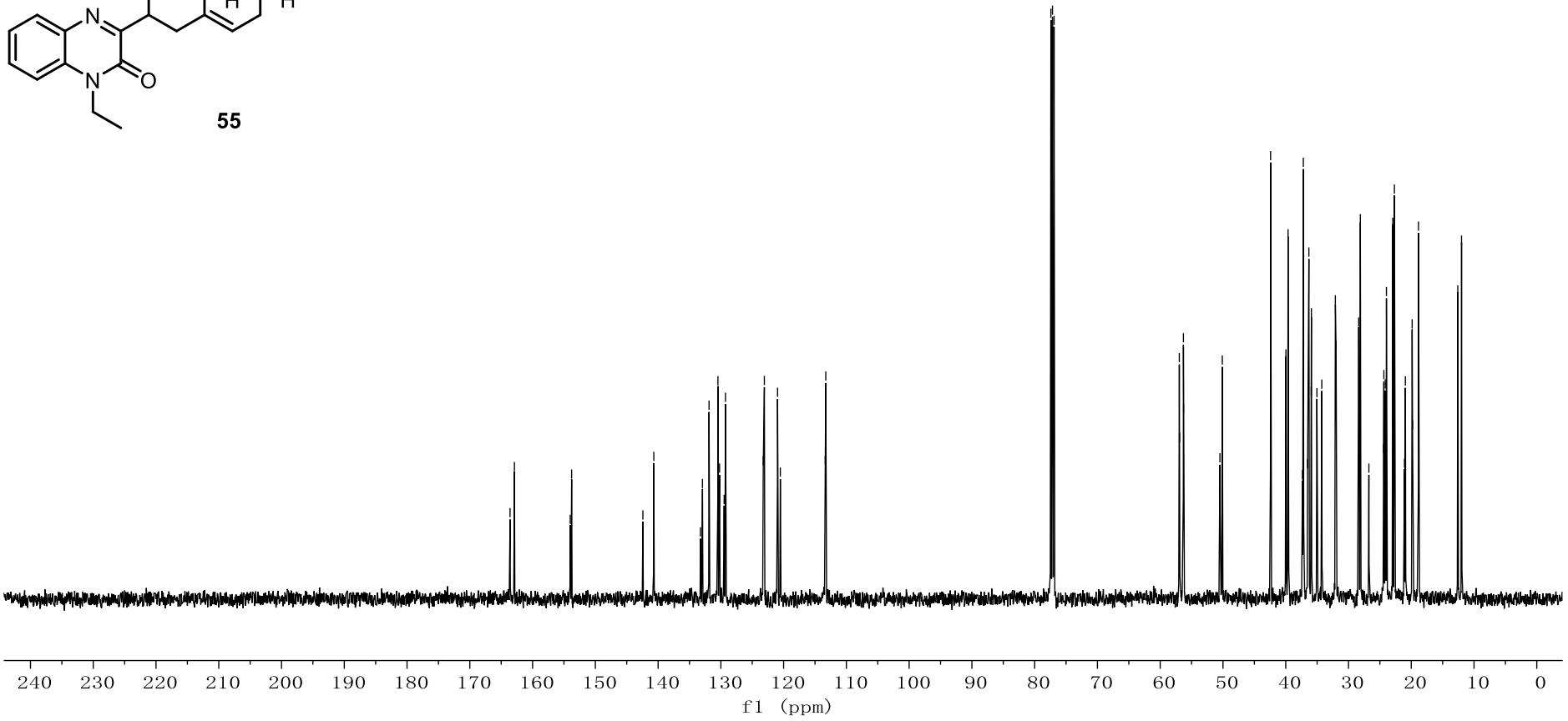


^{13}C NMR (125 MHz, $CDCl_3$) for 55

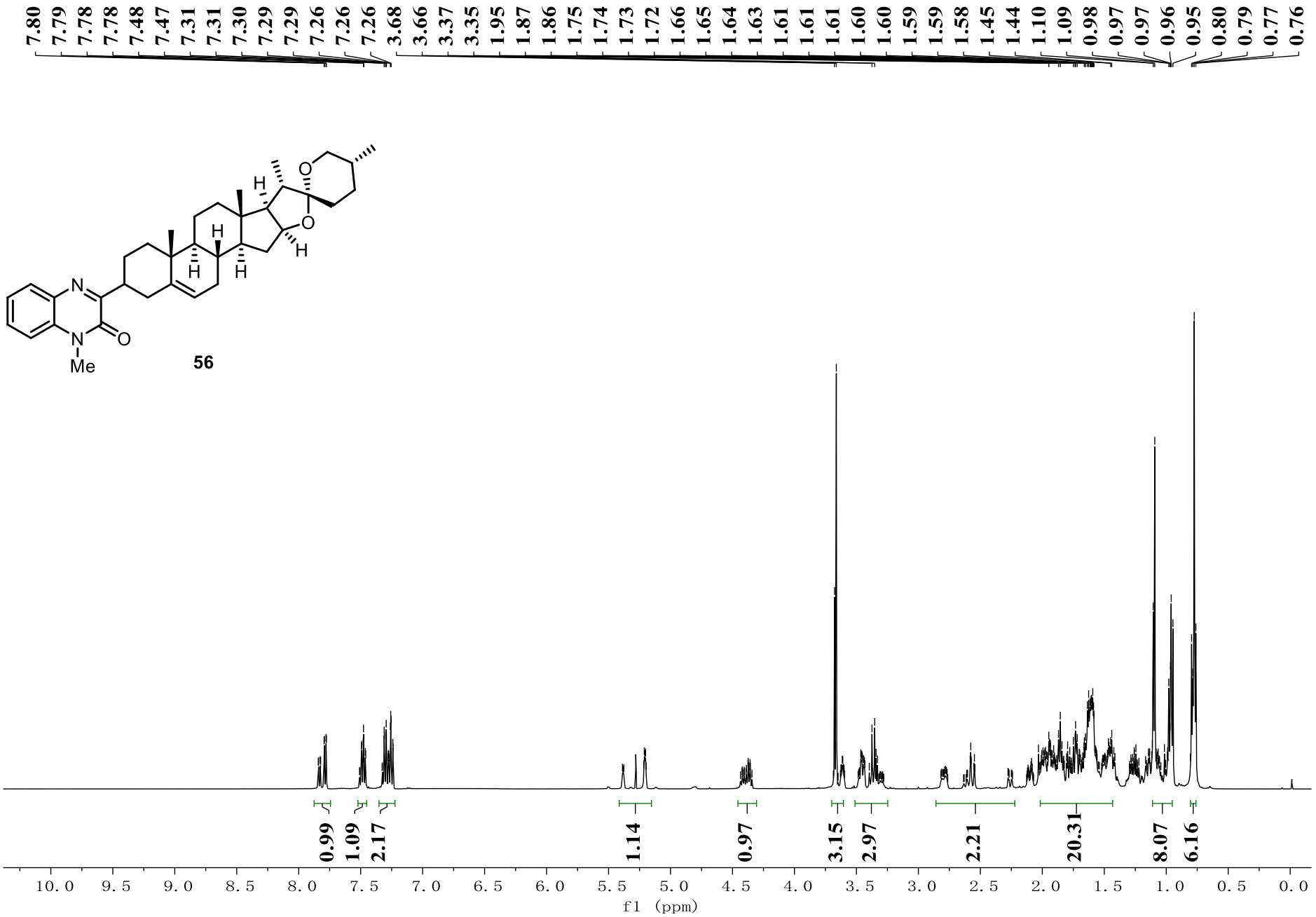
140.68
131.89
130.47
129.27
123.25
123.07
120.97
113.37
113.28
77.42
77.16
76.91
56.96
56.90
56.30
56.27
50.48
50.11
42.41
39.97
39.63
39.55
37.24
37.20
37.16
36.52
36.31
35.93
35.91
35.03
34.25
32.09
32.02
31.97
28.37
28.11
24.42
24.36
24.14
23.95
22.94
22.69
21.08
20.94
19.86
19.79
18.84
12.59
12.56
11.99



55

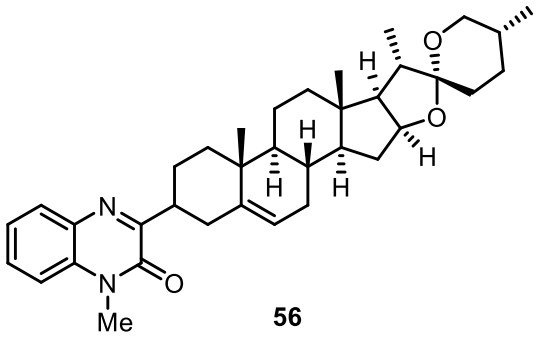


^{1}H NMR (500 MHz, $CDCl_3$) for 56



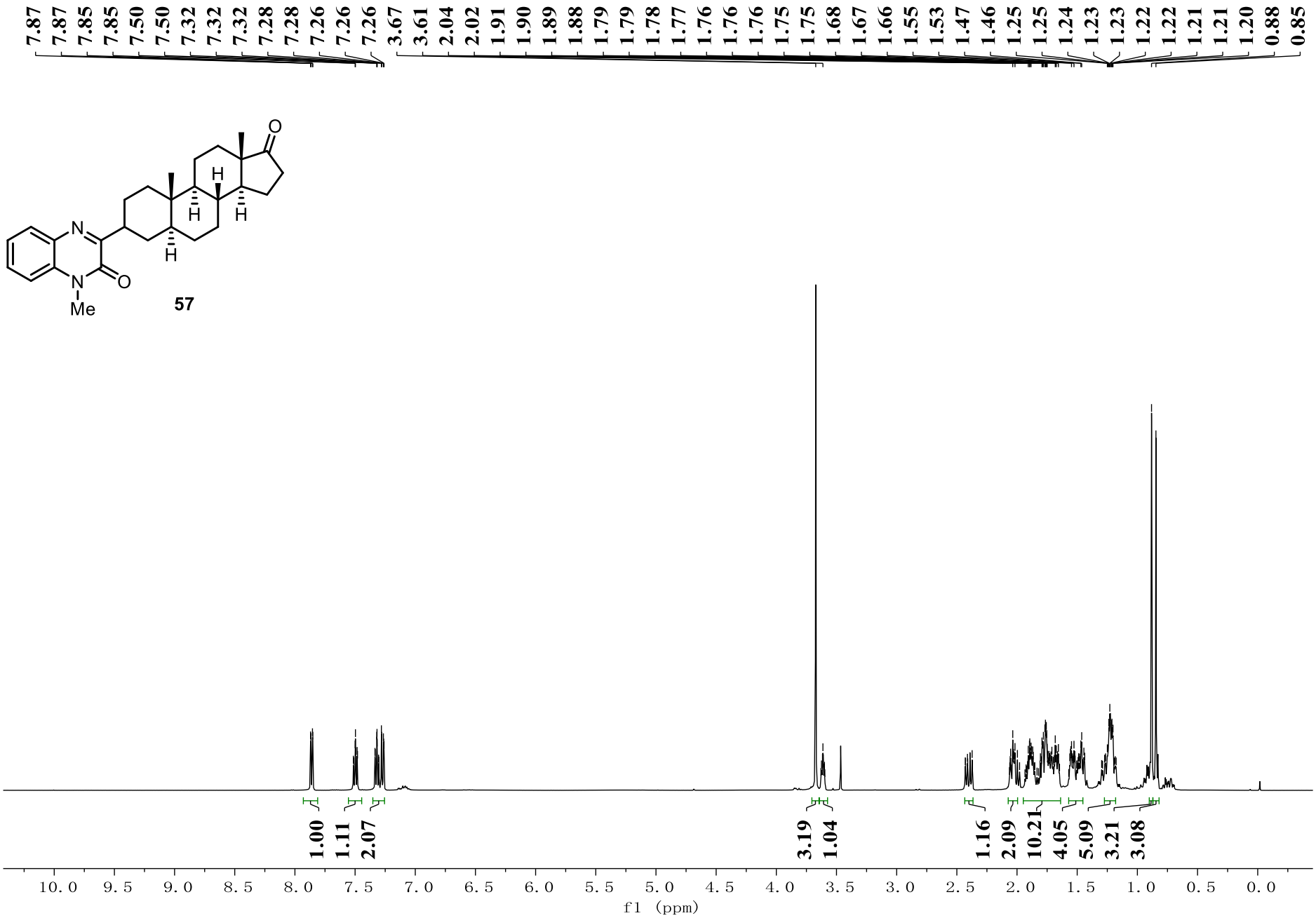
^{13}C NMR (125 MHz, $CDCl_3$) for 56

162.78 154.32 140.69 132.97 132.59 130.18 129.92 129.35 123.50 123.32 120.76 120.29 113.55 113.47 109.36 80.98 77.41 77.16 76.91 66.92 62.20 56.66 50.38 50.03 42.46 41.69 40.35 39.95 37.38 37.30 35.03 34.20 32.21 31.96 31.91 31.55 31.49 30.39 29.13 29.04 28.90 26.63 24.08 20.84 20.69 19.82 19.79 17.25 16.42 14.65

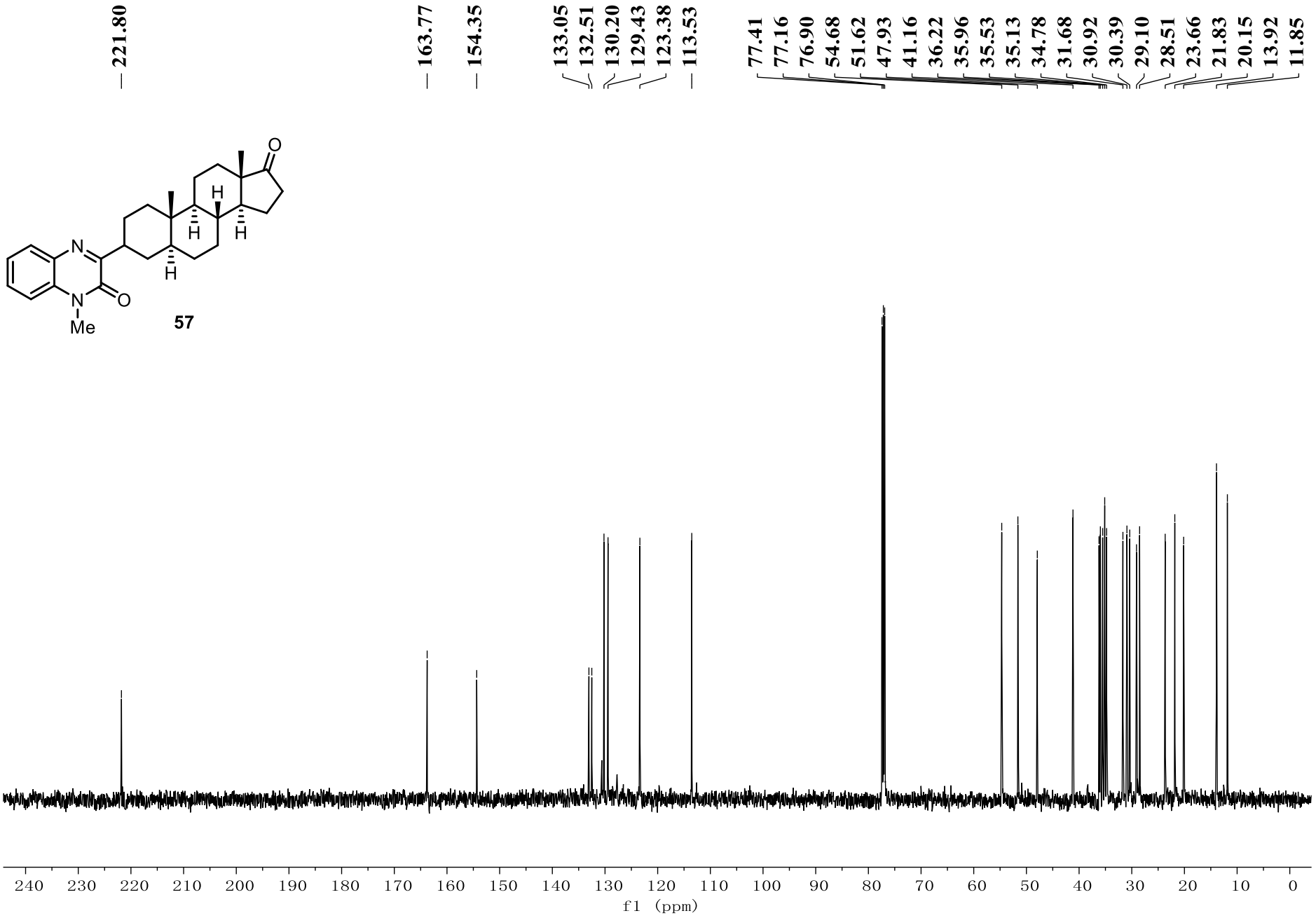


56

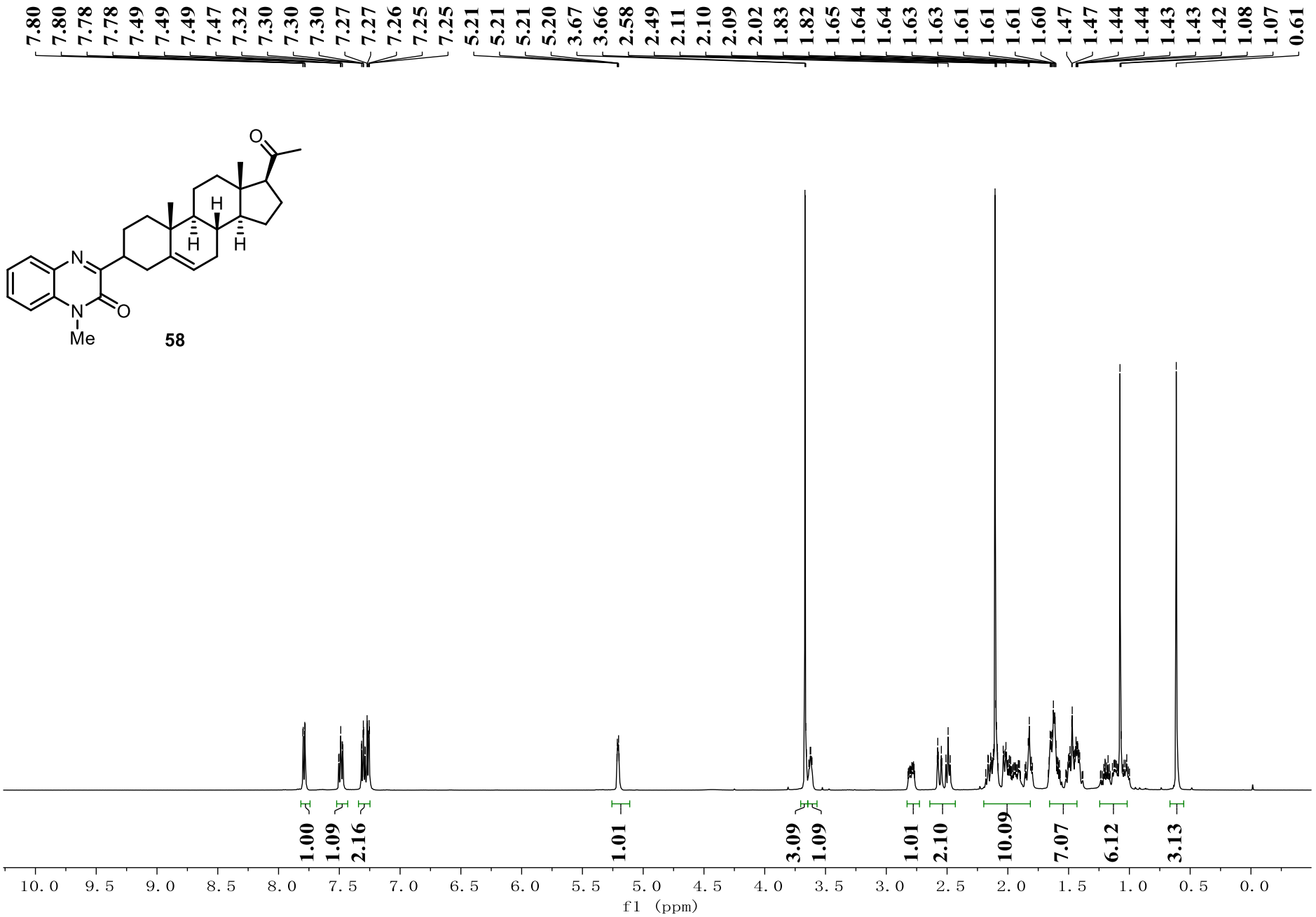
^{1}H NMR (500 MHz, $CDCl_3$) for 57



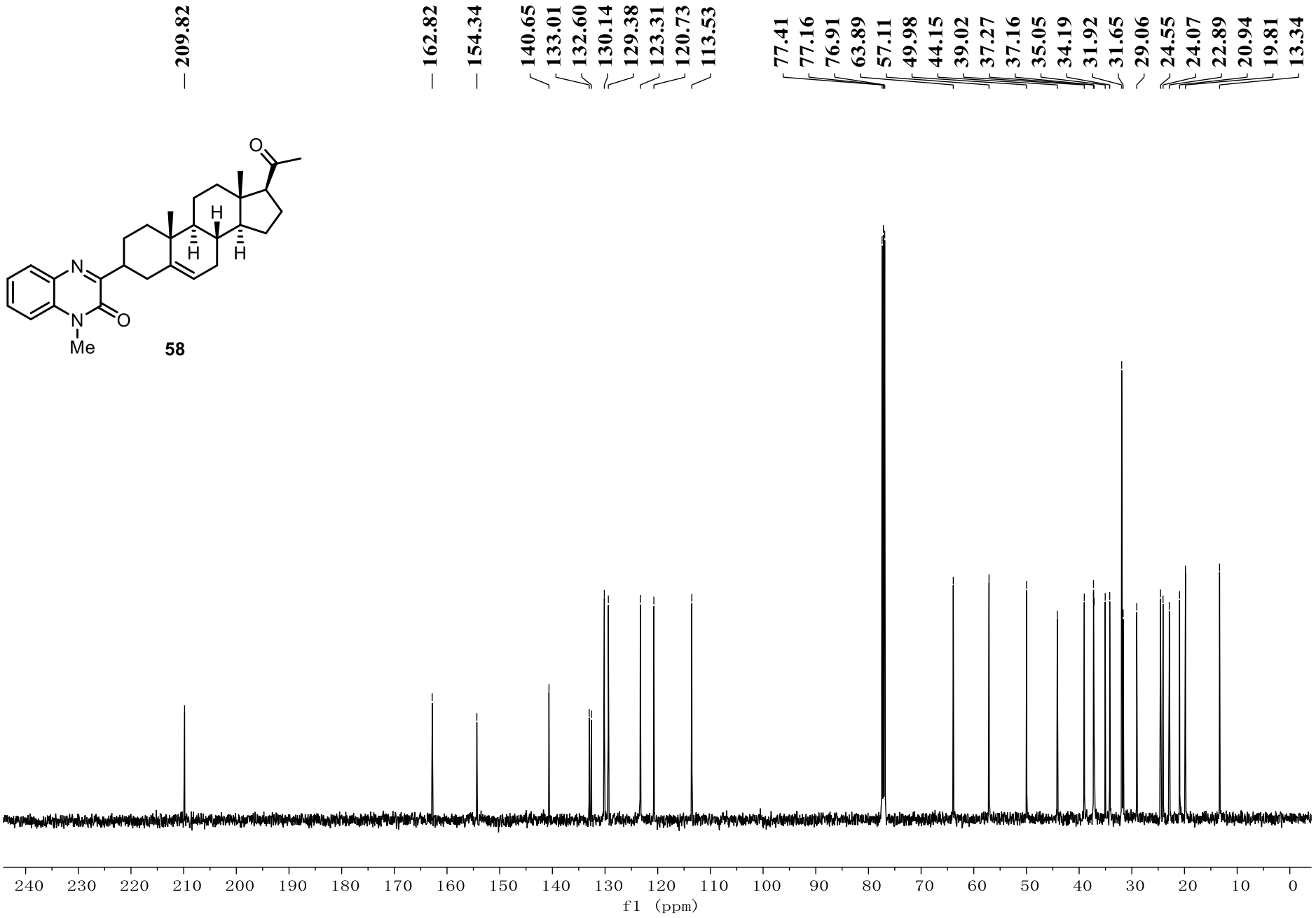
^{13}C NMR (125 MHz, $CDCl_3$) for 57



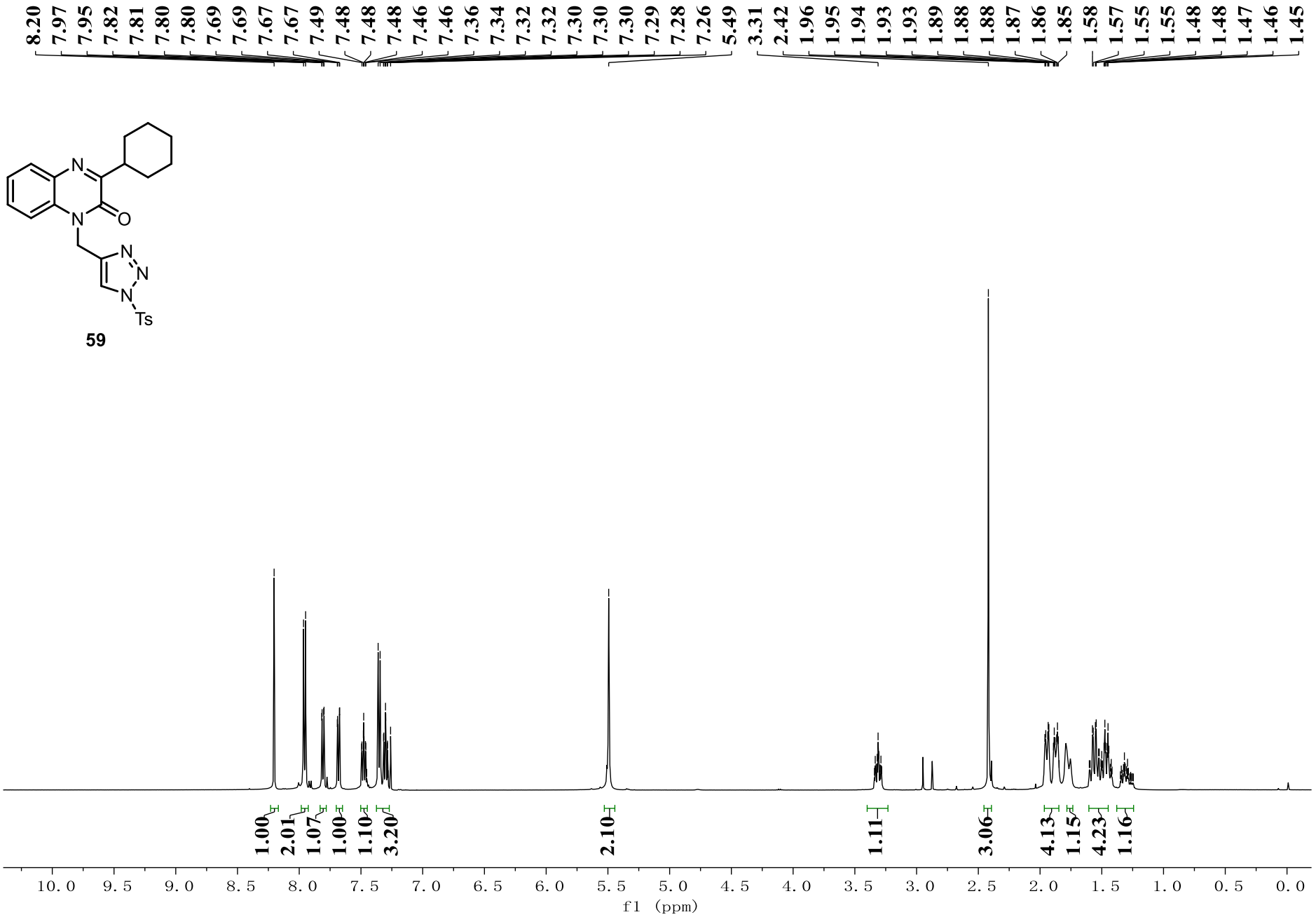
^{1}H NMR (500 MHz, $CDCl_3$) for 58



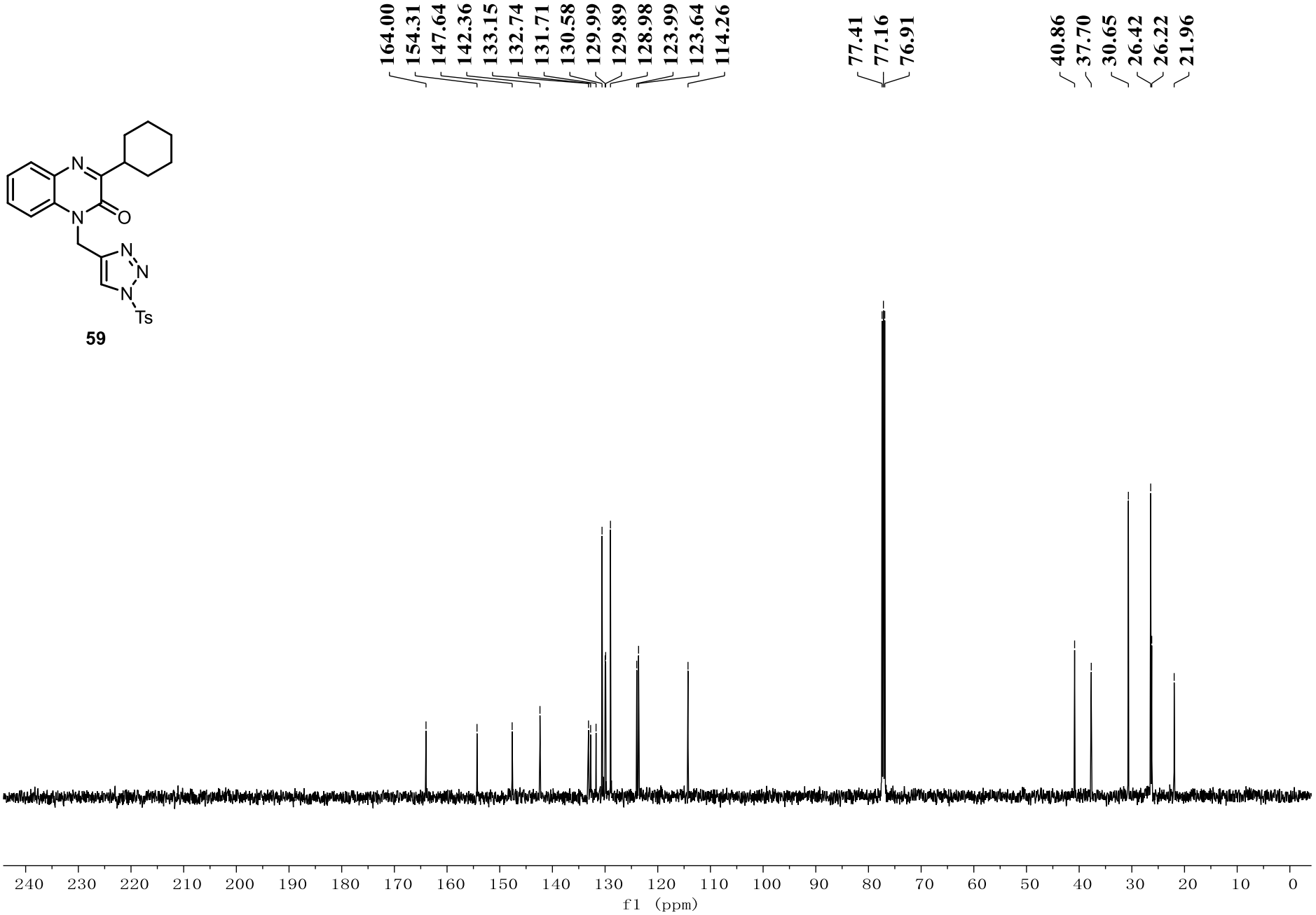
^{13}C NMR (125 MHz, $CDCl_3$) for 58



^{1}H NMR (500 MHz, $CDCl_3$) for 59

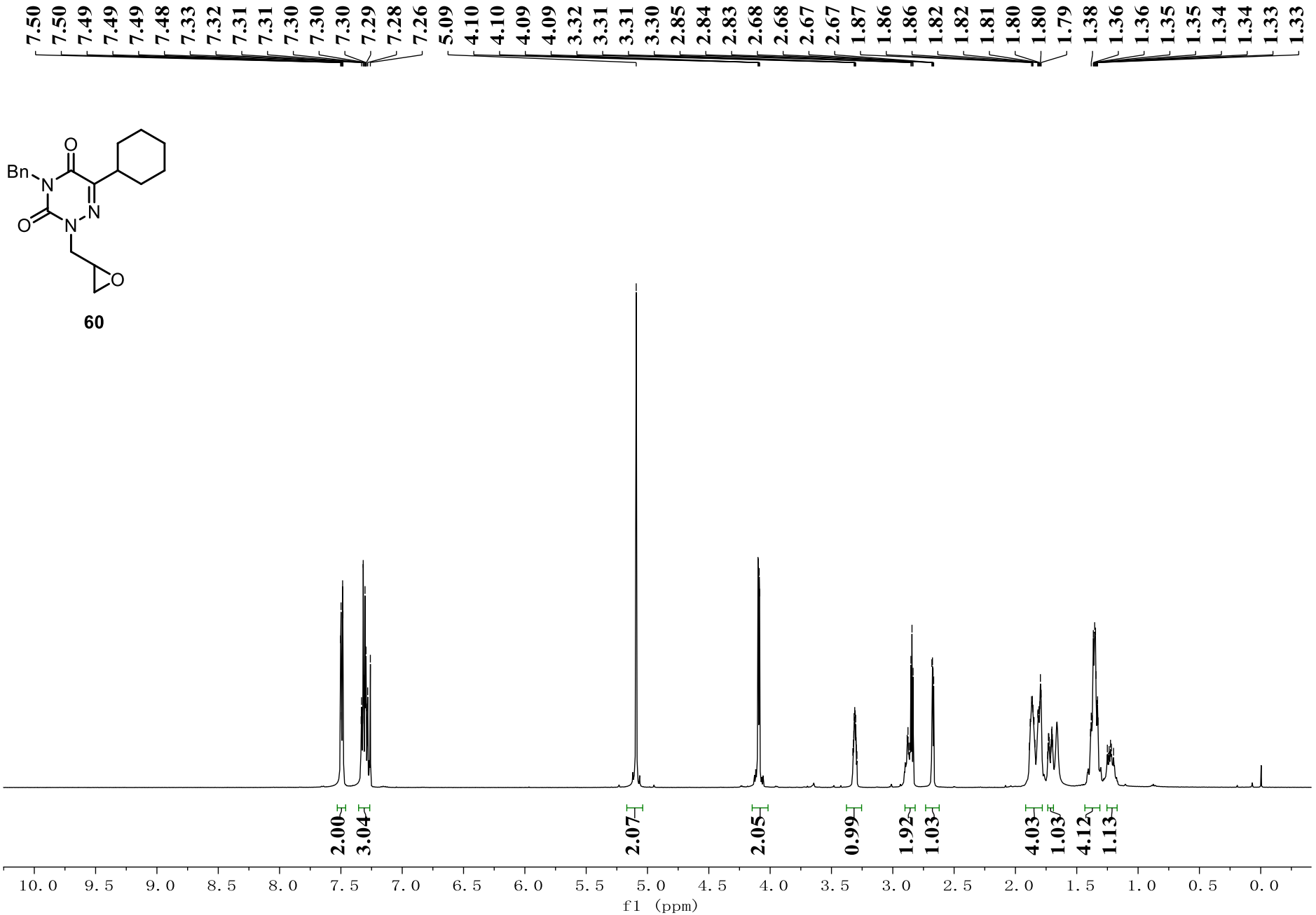


^{13}C NMR (125 MHz, $CDCl_3$) for 59

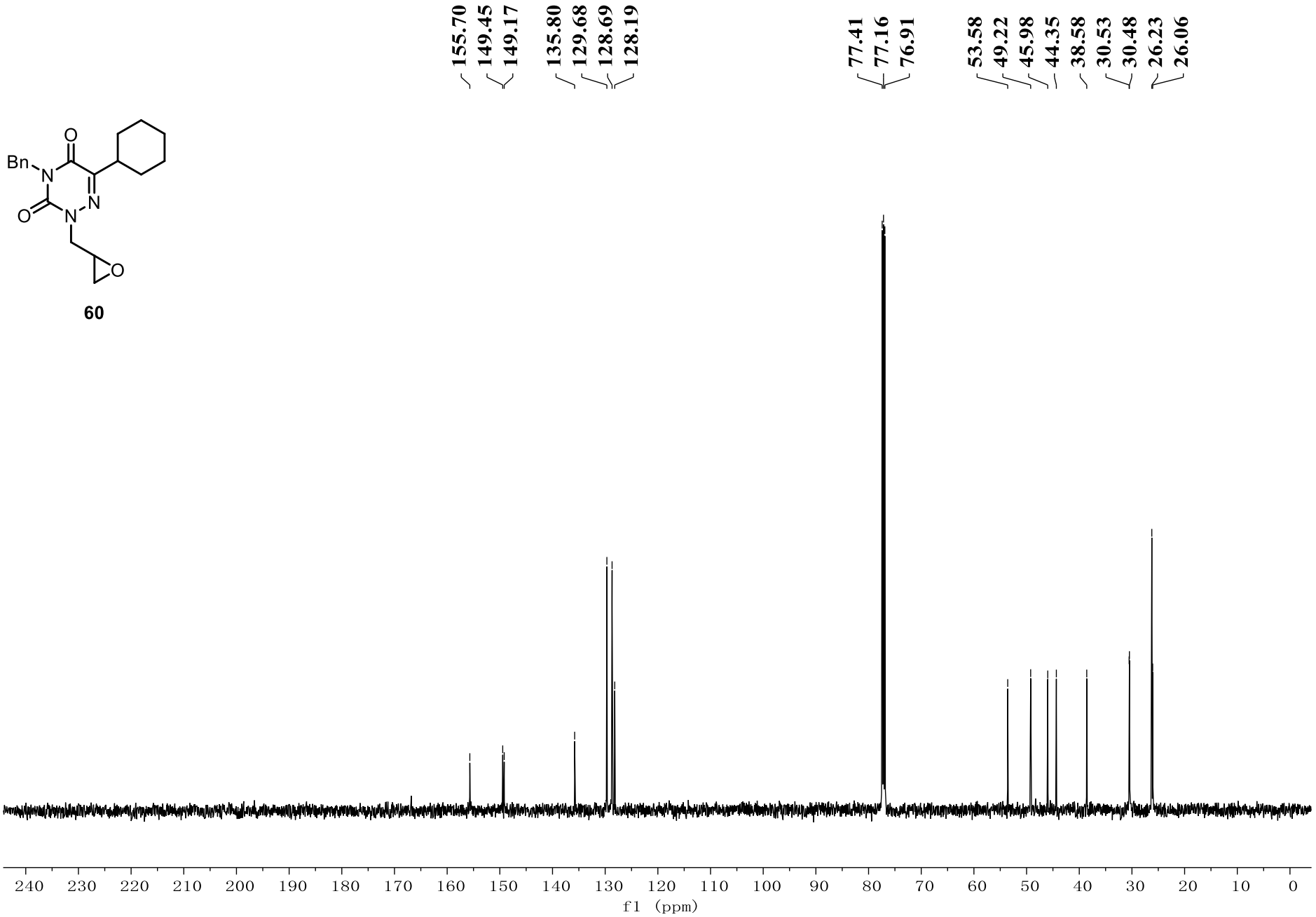


59

^{1}H NMR (500 MHz, $CDCl_3$) for 60



^{13}C NMR (125 MHz, $CDCl_3$) for 60



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