Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information for:

Oxidation of 2-arylindoles for synthesis of 2-arylbenzoxazinones with oxone as the sole oxidant

Xiao-Li Lian, Hao Lei, Xue-Jing Quan, Zhi-Hui Ren, Yao-Yu Wang, Zheng-Hui Guan*

Key Laboratory of Synthetic and Natural Functional Molecule Chemistry of Ministry of Education, Department of Chemistry & Materials Science, Northwest University, Xi'an 710069, P. R. China

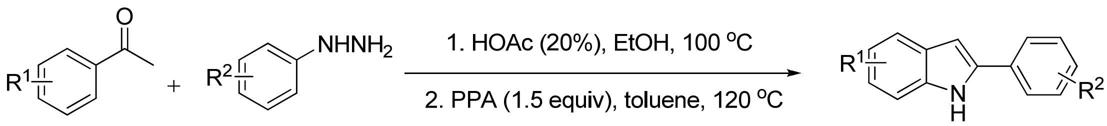
E-mail: guanzhh@nwu.edu.cn

CONTENTS

1. General Information

^{1}H and ^{13}CNMR spectra were recorded on Varian instrument (400 MHz) and (100 MHz). The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad. Coupling constants, J were reported in Hertz unit (Hz). Preparative TLC was performed on TLC plates, Analytical thin layer chromatography was performed on 10-25um silica gel GF254, visualization was carried out with UV light. Flash column chromatography was performed with SiO_2 (Silicycle Silica Gel 60 (200-300 mesh)). Unless otherwise stated, all reagents and solvents were purchased from commercial suppliers and used without further purification.

2. General procedure for the preparation of 2-arylindoles



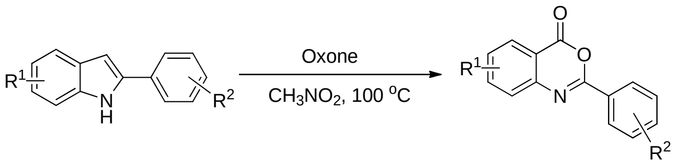
Acetophenone (10 mmol), phenylhydrazine (1.2 equiv), HOAc (20% mmol) and EtOH (6 mL) were charged in a 25 mL round bottom flask. Then, the reaction mixture was stirred at 100 °C. When the reaction was completed (detected by TLC), the mixture was cooled to room temperature. The EtOH was evaporated in vacuo, then recrystallized with EtOAc and hexane. Next, phenylhydrazone (10 mmol) were dissolved in toluene (6 mL). To this solution, 1.5 equiv PPA was added at one time and the solution was refluxed. After completion, the reaction mixture was cooled to room temperature, quenched with H_2O (10 mL) and extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and then evaporated in vacuo. The residue was purified by column chromatography on silica gel to afford the corresponding 2-arylindoles with ethyl acetate/hexane as the eluent.

(1) E. Fischer, F. Jourdan, *Ber. Dtsch. Chem. Ges,* **1883**, *16*, 2241-2245.

(2) B. Robinson, *The Fischer Indole Synthesis*, John Wiley & Sons Inc, New York, 1982.

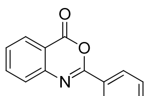
(3) Y. Wei, I. Deb and N. Yoshikai, *J. Am. Chem. Soc,* **2012**, *134*, 9098-9101.

3. Typical Procedure for Direct Oxidation of 2-arylindoles



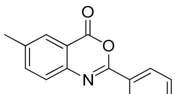
2-Aryllindoles (0.2 mmol), oxone (0.6 mmol, 369 mg) and CH_3NO_2 (2 mL) were charged in a 10 mL round bottom flask. Then, the reaction mixture was stirred at 100 °C. When the reaction was completed (detected by TLC), the mixture was cooled to room temperature. The reaction was quenched with H_2O (10 mL) and extracted with EtOAc (3 × 10 mL) or CH_2Cl_2 (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and then evaporated in vacuo. The residue was purified by column chromatography on silica gel to afford the corresponding 2-arylbenzoxazinones with ethyl acetate/hexane as the eluent.

4. Characterization Data of products



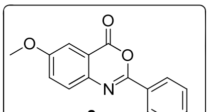
2a

2a: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.31 (d, J = 8.0 Hz, 2 H), 8.24 (d, J = 8.0 Hz, 1 H), 7.85-7.81 (t, J = 7.6 Hz, 1 H), 7.69 (d, J = 8.0 Hz, 1 H), 7.60-7.50 (m, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.5, 157.0, 146.9, 136.5, 132.6, 130.2, 128.7, 128.5, 128.3, 128.2, 127.2, 117.0, 77.3, 77.0, 76.9. HRMS Calcd (ESI) m/z for $C_{14}H_9NNaO_2$: $[M+Na]^+$ 246.0525, found: 246.0523.



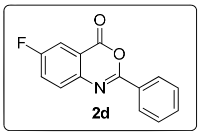
2b

2b: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.17 (d, J = 7.2 Hz, 2 H), 7.90 (s, 1 H), 7.51-7.37 (m, 5 H), 2.36 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.6, 144.6, 138.6, 137.7, 132.3, 130.2, 128.6, 128.0, 126.9, 116.6, 77.3, 77.0, 76.7, 21.2. HRMS Calcd (ESI) m/z for $C_{15}H_{11}NNaO_2$: $[M+Na]^+$ 260.0682, found: 260.0686.



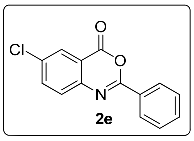
2c

2c: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.17 (d, J = 7.2 Hz, 2 H), 7.52 (d, J = 9.2 Hz, 2 H), 7.48-7.38 (m, 3 H), 7.30-7.27 (m, 1 H), 3.82 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.8, 159.2, 155.1, 141.0, 132.1, 130.2, 128.7, 128.6, 127.9, 125.9, 117.6, 108.5, 77.3, 77.0, 76.7, 55.9. HRMS Calcd (ESI) m/z for $C_{15}H_{11}NNaO_3$: $[M+Na]^+$ 276.0631, found: 276.0632.



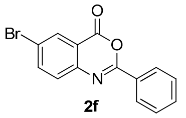
2d

2d: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.12 (d, J = 7.6 Hz, 2 H), 7.73-7.71 (m, 1 H), 7.57-7.54 (m, 1 H), 7.46-7.35 (m, 4 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.4, 157.4 (d, J_{CF} = 230.1 Hz), 143.4, 132.6, 129.7, 129.4 (d, J_{CF} =8.0 Hz), 128.6, 128.1, 124.6 (d, J_{CF} =23.5 Hz), 118.1 (d, J_{CF} =8.8 Hz), 113.7 (d, J_{CF} =24.0 Hz), 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8FNNaO_2$: $[M+Na]^+$ 264.0431, found: 264.0435.



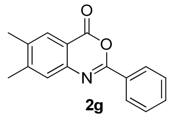
2e

2e: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.27 (d, J = 7.2 Hz, 2 H), 8.17 (s, 1 H), 7.74 (d, J = 8.0 Hz, 1 H), 7.63-7.50 (m, 4 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.4, 157.2, 145.4, 136.8, 133.8, 132.8, 129.7, 128.7, 128.7, 128.3, 127.9, 118.0, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{10}H_8N_2Na$: $[M+Na]^+$ 280.0136, found: 280.0126.



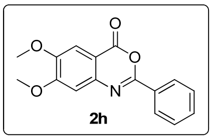
2f

2f: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.34 (s, 1 H), 8.28 (d, J = 7.2 Hz, 2 H), 7.90 (d, J = 8.4 Hz, 1 H), 7.59-7.49 (m, 4 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.2, 157.4, 145.8, 139.6, 132.9, 131.0, 129.8, 128.9, 128.8, 128.3, 121.4, 118.3, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8BrNNaO_2$: $[M+Na]^+$ 323.9631, found: 323.9617.

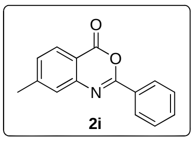


2g

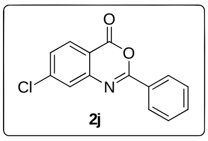
2g: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.24 (d, J = 7.2 Hz, 2 H), 7.89 (s, 1 H), 7.55-7.37 (m, 3 H), 7.37 (s, 1 H), 2.35 (s, 3 H), 2.32 (s, 1 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.4, 156.2, 146.9, 144.9, 137.7, 132.1, 130.2, 128.5, 128.2, 127.9, 127.3, 114.3, 77.3, 77.0, 76.7, 20.4, 19.5. HRMS Calcd (ESI) m/z for $C_{16}H_{13}NNaO_2$: $[M+Na]^+$ 274.0838, found: 274.0844.



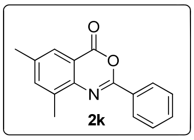
2h: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.25 (d, *J* = 7.6 Hz, 2 H), 7.55-7.48 (m, 4 H), 7.09 (s, 1 H), 4.02 (s, 3 H), 3.99 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.4, 156.5, 156.3, 149.6, 143.2, 132.2, 130.3, 128.6, 127.9, 109.5, 108.0, 107.4, 77.3, 77.0, 76.7, 56.4, 56.4. HRMS Calcd (ESI) m/z for $C_{16}H_{13}NNaO_4$: $[M+Na]^+$ 306.0737, found: 306.0735.



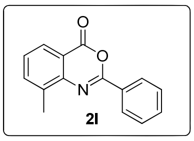
2i: ^{1}H NMR ($CDCl_3$, 400 MHz):δ 8.21 (d, *J* = 7.2 Hz, 2 H), 8.02 (d, *J* = 7.6 Hz, 1 H), 7.48-7.40 (m, 4 H), 7.23 (d, *J* = 7.6 Hz, 1 H), 2.42 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.5, 157.1, 148.0, 146.9, 132.5, 130.3, 129.6, 128.6, 128.4, 128.2, 127.1, 114.3, 77.3, 77.0, 76.7, 22.1. HRMS Calcd (ESI) m/z for $C_{15}H_{11}NNaO_2$: $[M+Na]^+$ 260.0682, found: 260.0691.



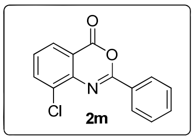
2j: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.20 (d, *J* = 7.6 Hz, 2 H), 8.07 (d, *J* = 8.4 Hz, 1 H), 7.60 (s, 1 H), 7.51-7.37 (m, 4 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.7, 158.2, 148.0, 142.9, 133.0, 129.8, 129.7, 128.8, 128.7, 128.4, 127.0, 115.3, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8ClNNaO_2$: $[M+Na]^+$ 280.0136, found: 280.0128.



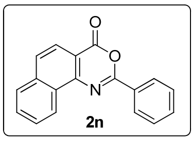
2k: ^{1}H NMR ($CDCl_3$, 400 MHz):δ 8.17 (d, *J* = 7.2 Hz, 2 H), 7.72 (s, 1 H), 7.44-7.23 (m, 3 H), 2.48 (s, 3 H), 2.30(s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 160.1, 154.9, 142.9, 138.5, 137.8, 135.8, 132.1, 130.5, 128.5, 128.0, 125.6, 116.5, 77.3, 77.0, 76.7, 21.2, 16.9. HRMS Calcd (ESI) m/z for $C_{16}H_{13}NNaO_2$: $[M+Na]^+$ 274.0838, found: 274.0845.



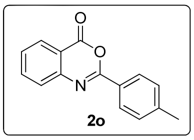
2l: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.30 (d, *J* = 7.6 Hz, 2 H), 8.03 (d, *J* = 7.6 Hz, 1 H), 7.63 (d, *J* = 7.2 Hz, 1 H), 7.53-7.47 (m, 3 H), 7.37-7.34 (t, *J* = 7.6 Hz, 1 H), 2.63 (s, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 160.0, 155.7, 145.1, 137.2, 136.1, 132.3, 130.4, 128.6, 128.1, 127.6, 126.1, 116.8, 77.3, 77.0, 76.7, 17.0. HRMS Calcd (ESI) m/z for $C_{15}H_{11}NNaO_2$: $[M+Na]^+$ 260.0682, found: 260.0678.



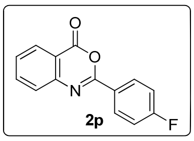
2m: [1]H NMR (CDCl3, 400 MHz): δ 8.26 (d, *J* = 7.6 Hz, 2 H), 8.05 (d, *J* = 8.0 Hz, 1 H), 7.78 (d, *J* = 7.6 Hz, 1 H), 7.52-7.49 (m, 1 H), 7.44-7.41 (m, 2 H), 7.35-7.31(m, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.8, 157.5, 143.7, 136.8, 133.0, 131.9, 129.8, 128.7, 128.6, 128.2, 127.1, 118.4, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8ClNNaO_2$: $[M+Na]^+$ 280.0136, found: 280.0147.



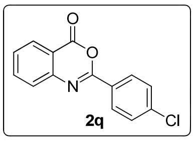
2n: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.81 (d, *J* = 6.0 Hz, 1 H), 8.27 (d, *J* = 6.4 Hz, 2 H), 7.93 (d, *J* = 8.4 Hz, 1 H), 7.72-7.67 (m, 2 H), 7.57 (s, 2 H), 7.48-7.42 (m, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.7, 157.9, 145.8, 137.0, 132.7, 130.3, 130.0, 128.7, 128.2, 127.8, 127.3, 125.2, 122.3, 112.8, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{18}H_{11}NNaO_2$: $[M+Na]^+$ 296.0682, found: 296.0687.



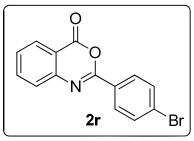
2o: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.09 (d, *J* = 8.0 Hz, 1 H), 8.05 (d, *J* = 8.0 Hz, 2 H), 7.70-7.66 (m,1 H), 7.53(d, *J* = 8.0 Hz, 1 H), 7.38-7.35 (m, 1 H), 7.17 (d, *J* = 8.0 Hz, 2 H) , 2.31 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.5, 157.1, 147.0, 143.2, 136.4, 129.4, 128.4, 128.2, 127.8, 127.3, 127.0, 116.8, 77.3, 77.0, 76.7, 21.6. HRMS Calcd (ESI) m/z for $C_{15}H_{11}NNaO_2$: $[M+Na]^+$ 260.0682, found: 260.0693.



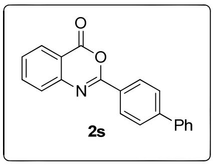
2p: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.31-8.28 (m, 2 H), 8.21 (d, *J* = 8.0 Hz, 1 H), 7.83-7.79 (t, *J* = 8.0 Hz, 1 H), 7.65 (d, *J* = 8.0 Hz, 1 H), 7.52-7.48 (t, *J* = 7.6 Hz, 1 H), 7.19-7.15 (t, *J* = 8.0 Hz, 2 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 165.5 (d, J_{CF}= 252.8 Hz), 159.3, 156.1, 146.8, 136.6, 130.6 (d, J_{CF}= 9.1 Hz), 128.5, 128.2, 127.1, 126.3, 116.7, 115.9 (d, J_{CF}= 22.0 Hz), 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8FNNaO_2$: $[M+Na]^+$ 264.0431, found: 264.0442.



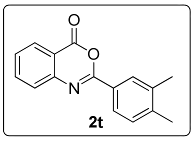
2q: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.25-8.23 (m, 2 H), 7.86-7.81 (m, 1 H), 7.68 (d, *J* = 8.4 Hz, 1 H), 7.55- 7.47 (m, 2 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.2, 156.2, 146.7, 139.0, 136.6, 129.6, 129.1, 128.6, 128.4, 127.2, 116.9, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8ClNNaO_2$: $[M+Na]^+$ 280.0136, found: 280.0129.


2r

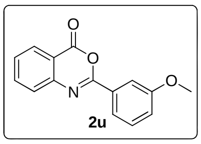
2r: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.12 (d, *J* = 7.6 Hz, 1 H), 8.04 (d, *J* = 8.0 Hz, 2 H), 7.75-7.71 (t, *J* = 7.6 Hz, 1 H), 7.57-7.52 (m, 3 H), 7.44-7.41 (m, 1 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.1, 156.2, 146.6, 136.6, 132.0, 129.6, 129.0, 128.6, 128.4, 127.6, 127.1, 116.8, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{14}H_8BrNNaO_2$: $[M+Na]^+$ 323.9631, found: 323.9637.


2s

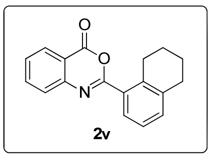
2s: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.36 (d, *J* = 8.4 Hz, 2 H), 8.23 (d, *J* = 7.6 Hz, 1 H), 7.84-7.80 (m, 2 H), 7.73-7.64 (m, 5 H), 7.50-7.40 (m, 3 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.5, 156.9, 147.0, 145.2, 139.7, 136.5, 128.9, 128.7, 128.5, 128.1, 128.1, 127.2, 127.1, 116.9, 77.3, 77.0, 76.7. HRMS Calcd (ESI) m/z for $C_{20}H_{13}NNaO_2$: $[M+Na]^+$ 322.0838, found: 322.0846.


2t

2t: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.18 (d, *J* = 8.0 Hz, 1 H), 8.00-7.96 (t, *J* = 10.8 Hz, 2 H), 7.79-7.75 (t, *J* = 7.6 Hz, 1 H), 7.62 (d, *J* =7.6 Hz, 1 H), 7.47-7.43 (t, *J* =7.6 Hz, 1 H), 7.20 (d, *J* = 8.0 Hz, 1 H), 2.30 (s, 3 H), 2.29 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.6, 157.3, 147.0, 142.0, 137.1, 136.4, 129.9, 129.1, 128.4, 127.8, 127.5, 126.9, 125.8, 116.8, 77.4, 77.0, 76.7, 20.0, 19.7. HRMS Calcd (ESI) m/z for $C_{16}H_{13}NNaO_2$: $[M+Na]^+$ 274.0838, found: 274.0847.

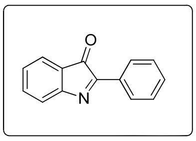

2u

2u: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.20 (d, *J* = 8.0 Hz, 1 H), 7.87 (d, *J* = 7.2 Hz, 1 H), 7.82-7.78 (m, 2 H), 7.66 (d, *J* = 8.0 Hz, 1 H), 7.51-7.47 (t, *J*=14.4 Hz, 1 H), 7.40-7.36 (t, *J* = 8.0 Hz, 1 H) ,7.10-7.08 (t, *J* = 8.0 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.7, 159.4, 156.8, 146.8, 136.5, 131.4, 129.7, 128.5, 128.2, 127.1, 120.7, 119.2, 116.9, 112.4, 77.4, 77.0, 76.7, 55.5. HRMS Calcd (ESI) m/z for $C_{15}H_{11}NNaO_3$: $[M+Na]^+$ 276.0631, found: 276.0637.


2v

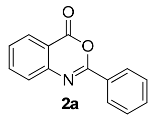
2v: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.20 (d, *J* = 8.0 Hz, 1 H), 7.98 (d, *J* = 5.6 Hz, 2 H), 7.81-7.77

(t, *J* = 8.0 Hz, 1 H), 7.64 (d, *J* = 8.4 Hz, 1 H),7.49-7.45(t, *J* = 7.2 Hz, 1 H), 7.16 (d, *J* = 7.6 Hz, 1 H), 2.84-2.81 (m, 4 H), 1.82 (s, 4 H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.7, 157.4, 147.1, 142.7, 137.6, 136.4, 129.5, 128.9, 128.4, 127.8, 127.1, 127.0, 125.2, 116.8, 77.3, 77.0, 76.7, 29.6, 29.3, 22.9, 22.8. HRMS Calcd (ESI) m/z for $C_{18}H_{15}NNaO_2$: $[M+Na]^+$ 300.0995, found: 300.1001.

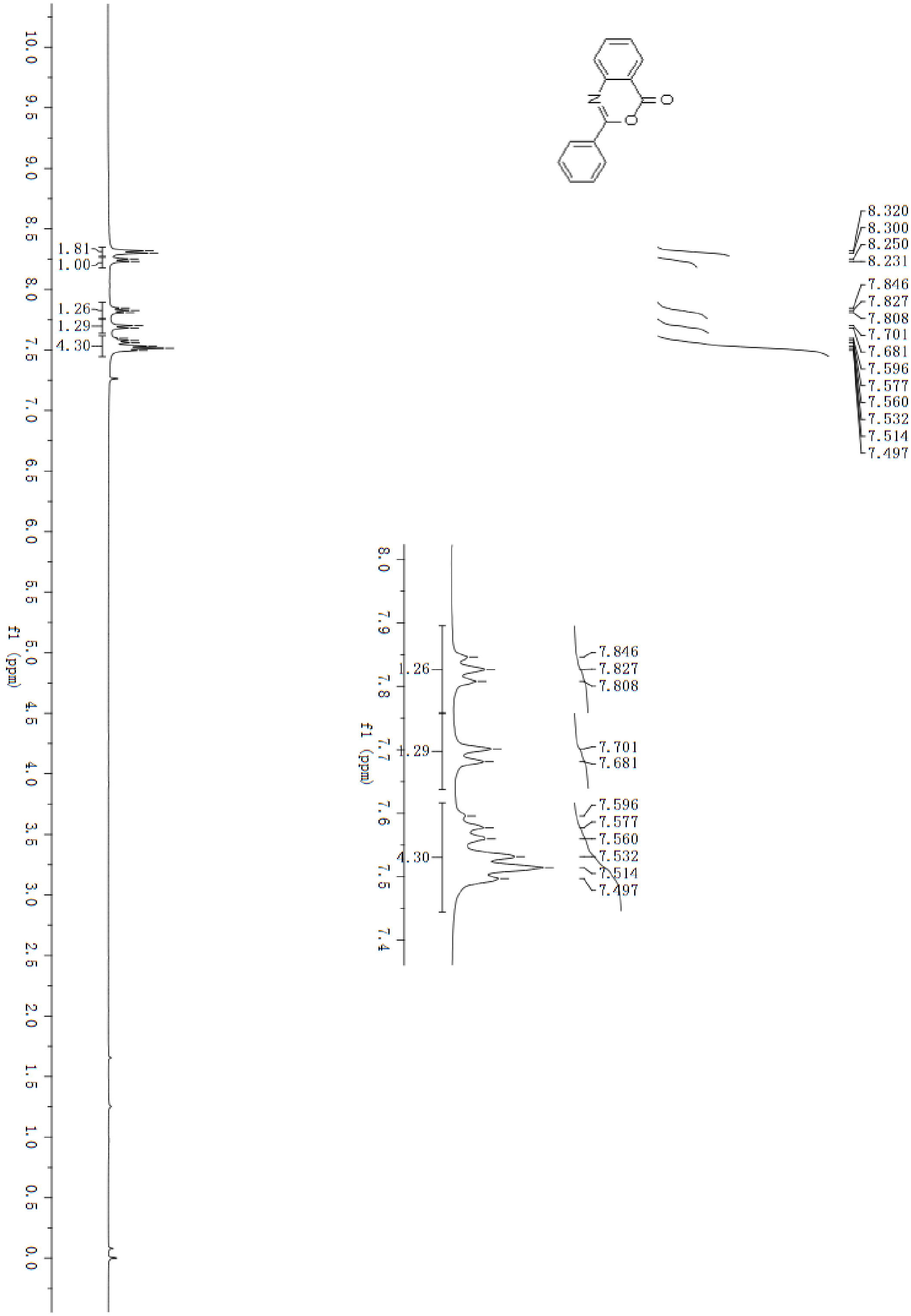


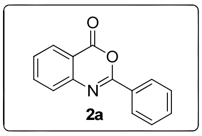
A: H NMR ($CDCl_3$, 400 MHz): δ 8.38 (d, *J* = 7.2 Hz, 2H), 7.57-7.47 (m, 5H), 7.42 (d, *J* = 7.2Hz, 1H), 7.27 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 193.4, 161.0, 159.7, 136.7, 132.1, 129.9, 129.2, 128.7, 128.3, 124.6, 123.0, 121.9. HRMS Calcd (ESI) m/z for $C_{14}H_9NO$: $[M+H]^+$ 208.0757, found: 208.0764

5. Copies of ^{1}H and ^{13}C NMR Spectra

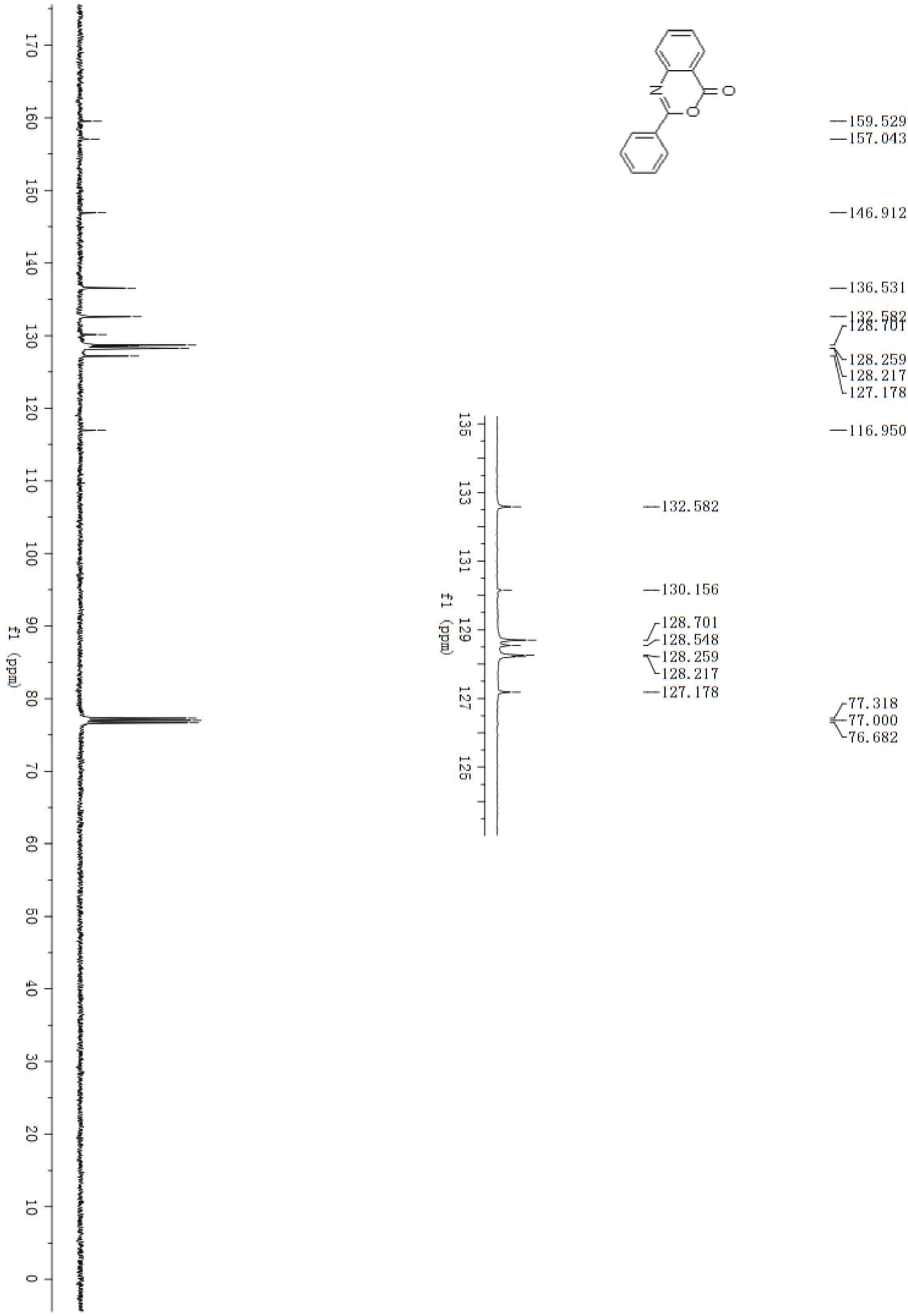


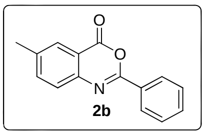
2a



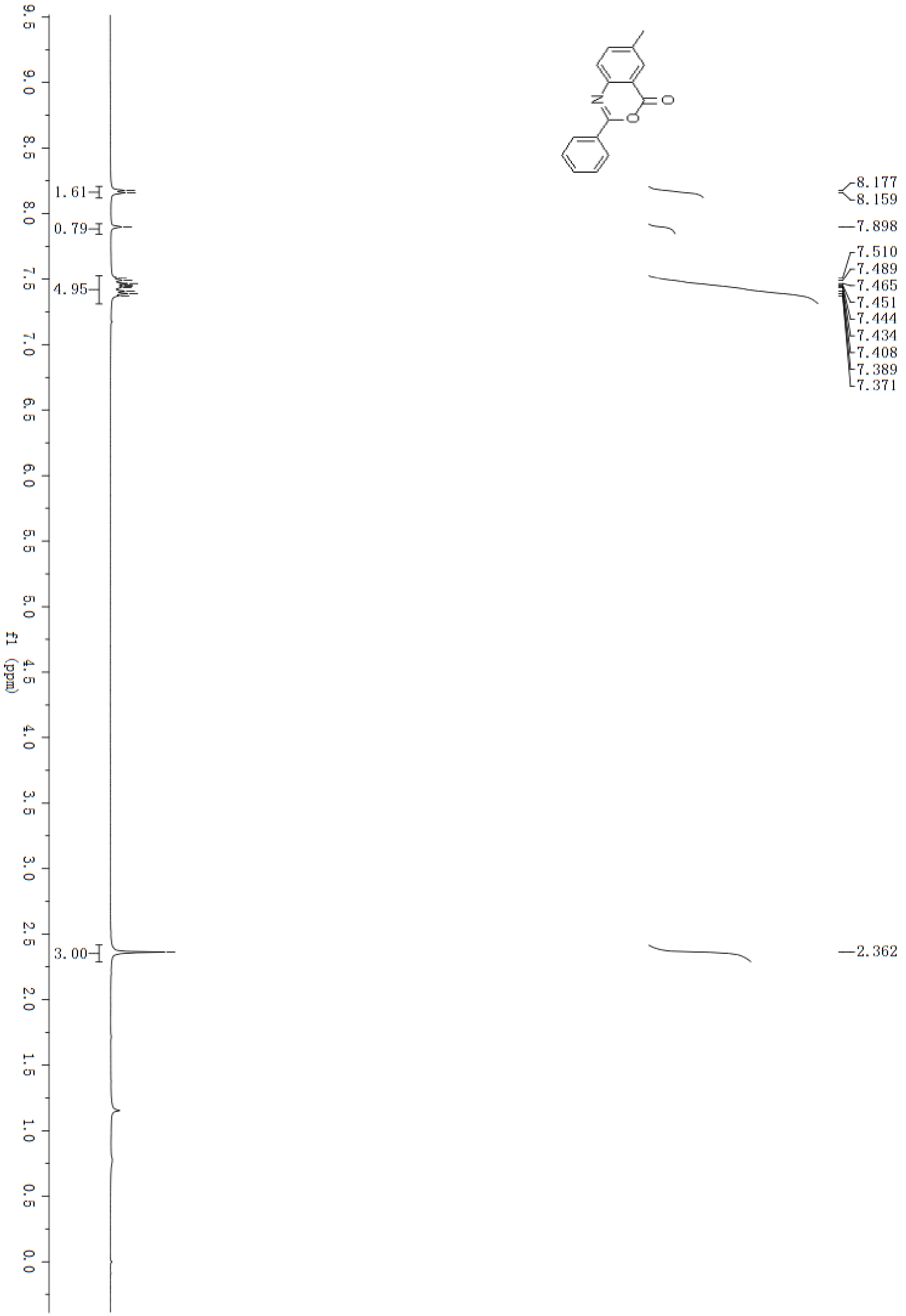


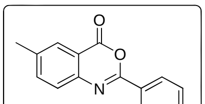
2a


159.529
157.043
146.912
136.531
132.582
128.701
128.259
128.217
127.178
116.950
132.582
130.156
128.701
128.548
128.259
128.217
127.178
77.318
77.000
76.682
f1 (ppm)
f1 (ppm)

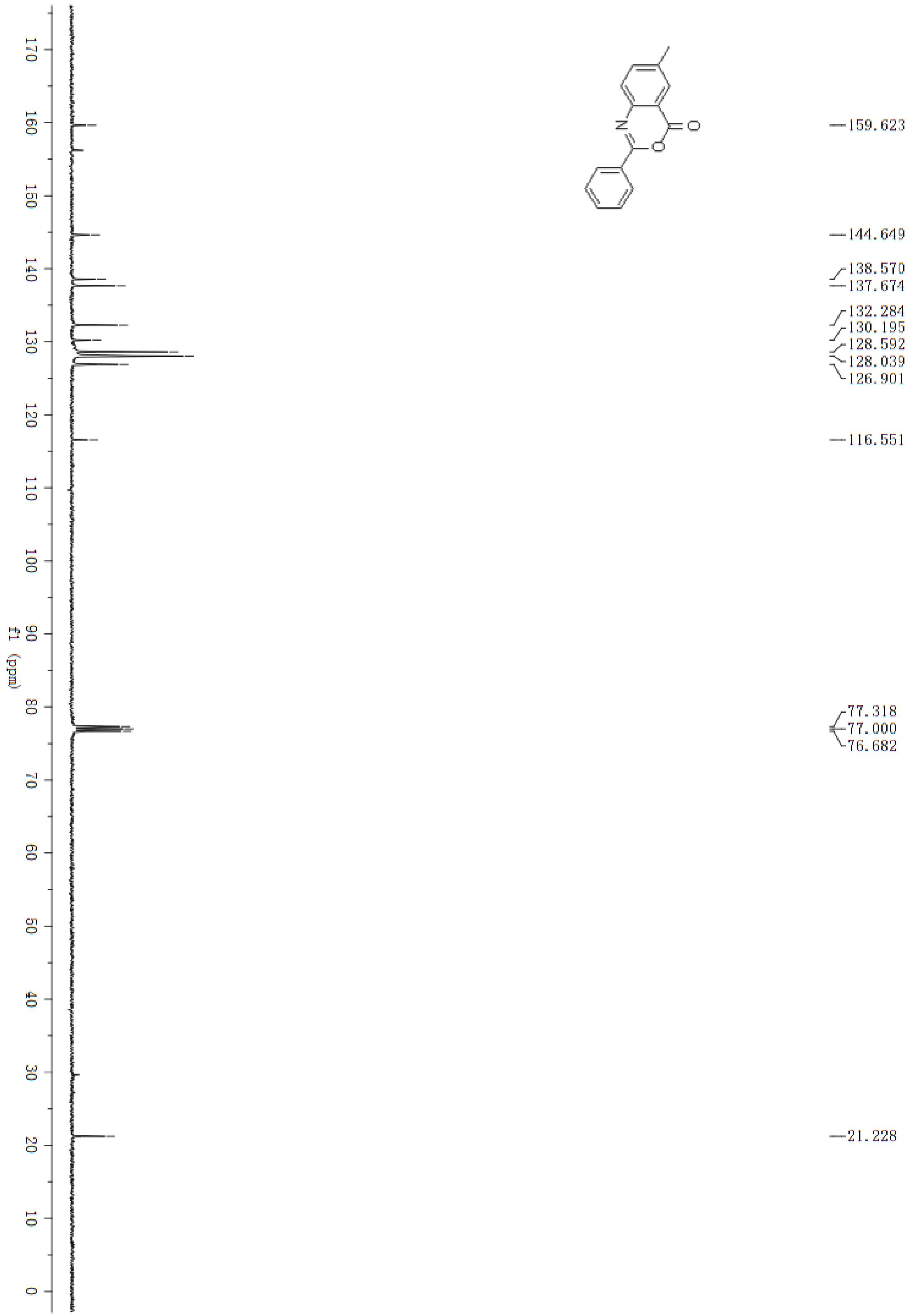


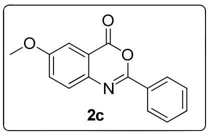
2b

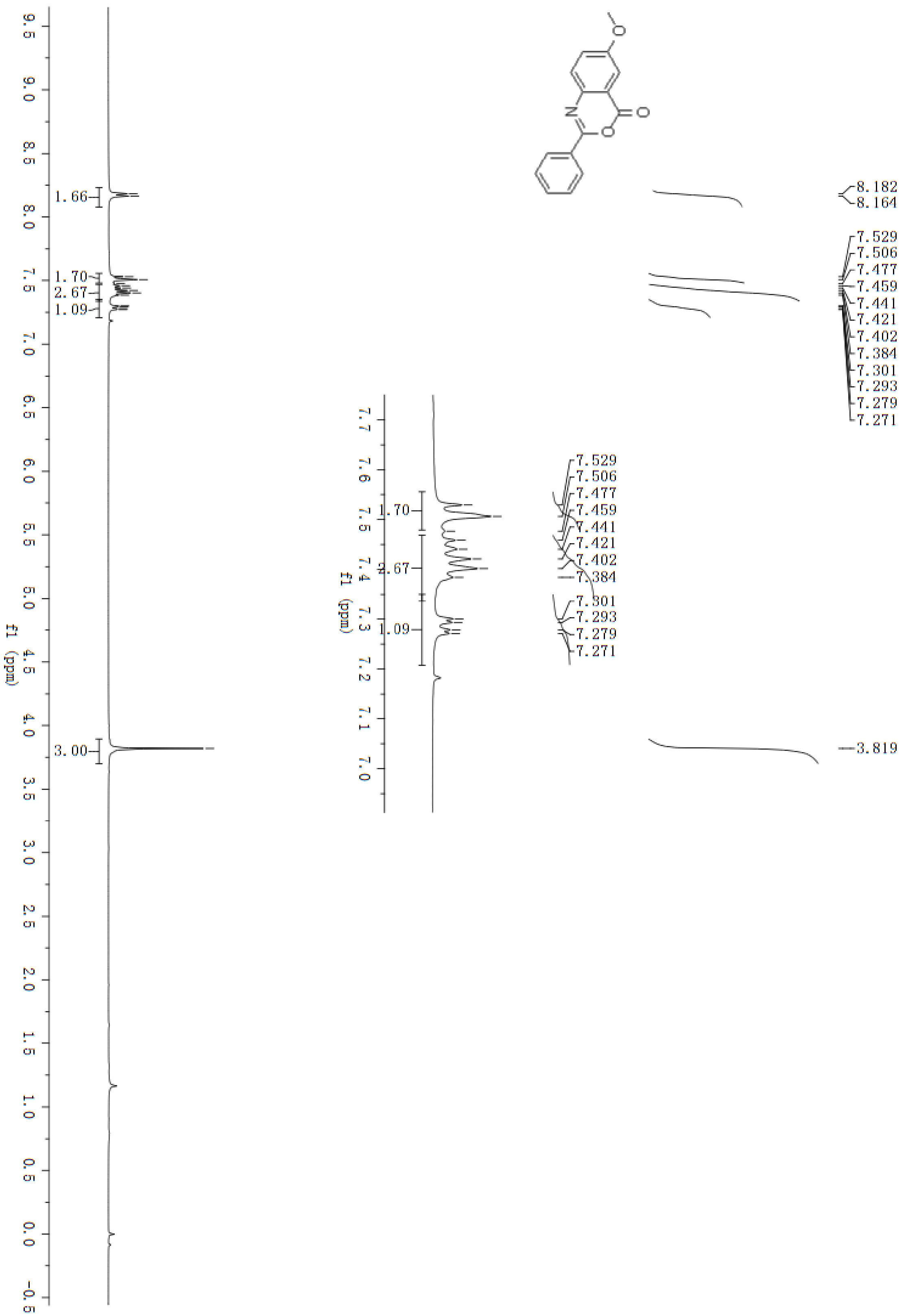

8.177
8.159
7.898
7.510
7.489
7.465
7.451
7.444
7.434
7.408
7.389
7.371
2.362
1.61
0.79
4.95
3.00
f1 (ppm)

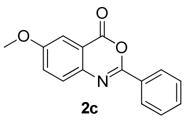

O
O
N

2b

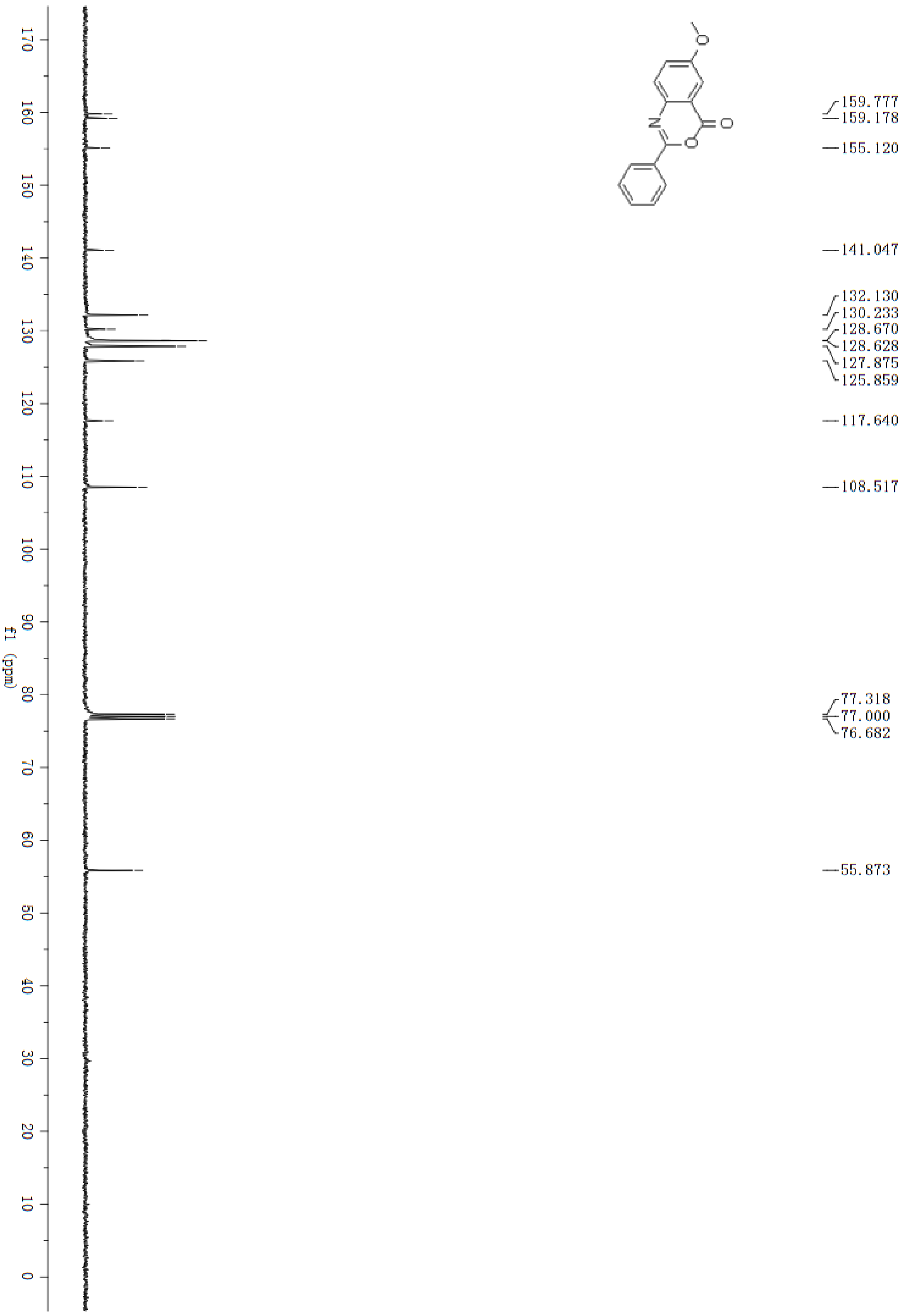

159.623
144.649
138.570
137.674
132.284
130.195
128.592
128.039
126.901
116.551
77.318
77.000
76.682
21.228
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
N
O
O

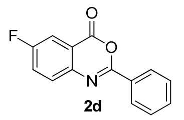

2c


8.182
8.164
7.529
7.506
7.477
7.459
7.441
7.421
7.402
7.384
7.301
7.293
7.279
7.271
3.819
1.66
1.70
2.67
1.09
3.00
f1 (ppm)

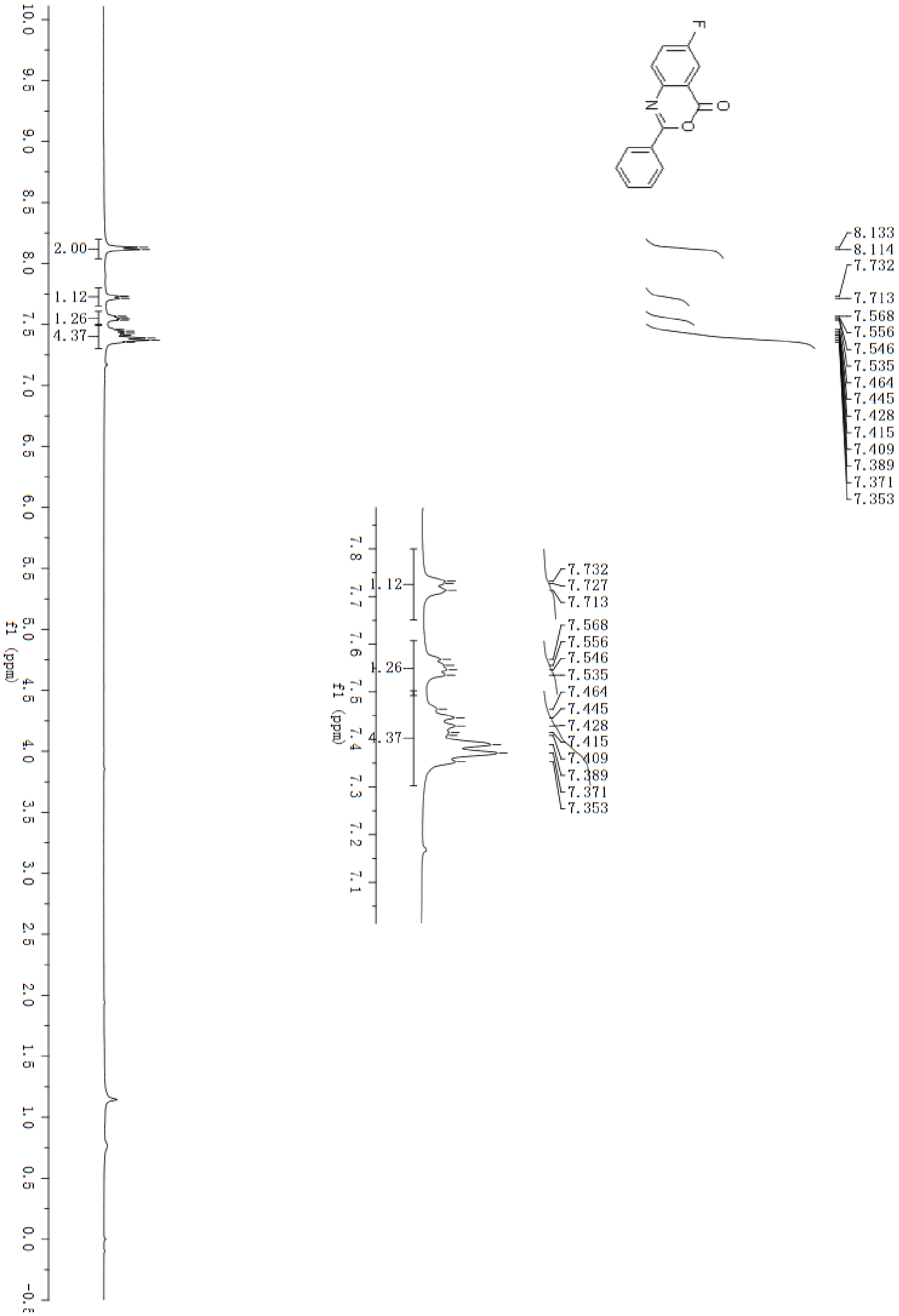


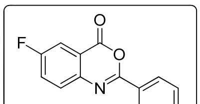
2c


159.777
159.178
155.120
141.047
132.130
130.233
128.670
128.628
127.875
125.859
117.640
108.517
77.318
77.000
76.682
55.873
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

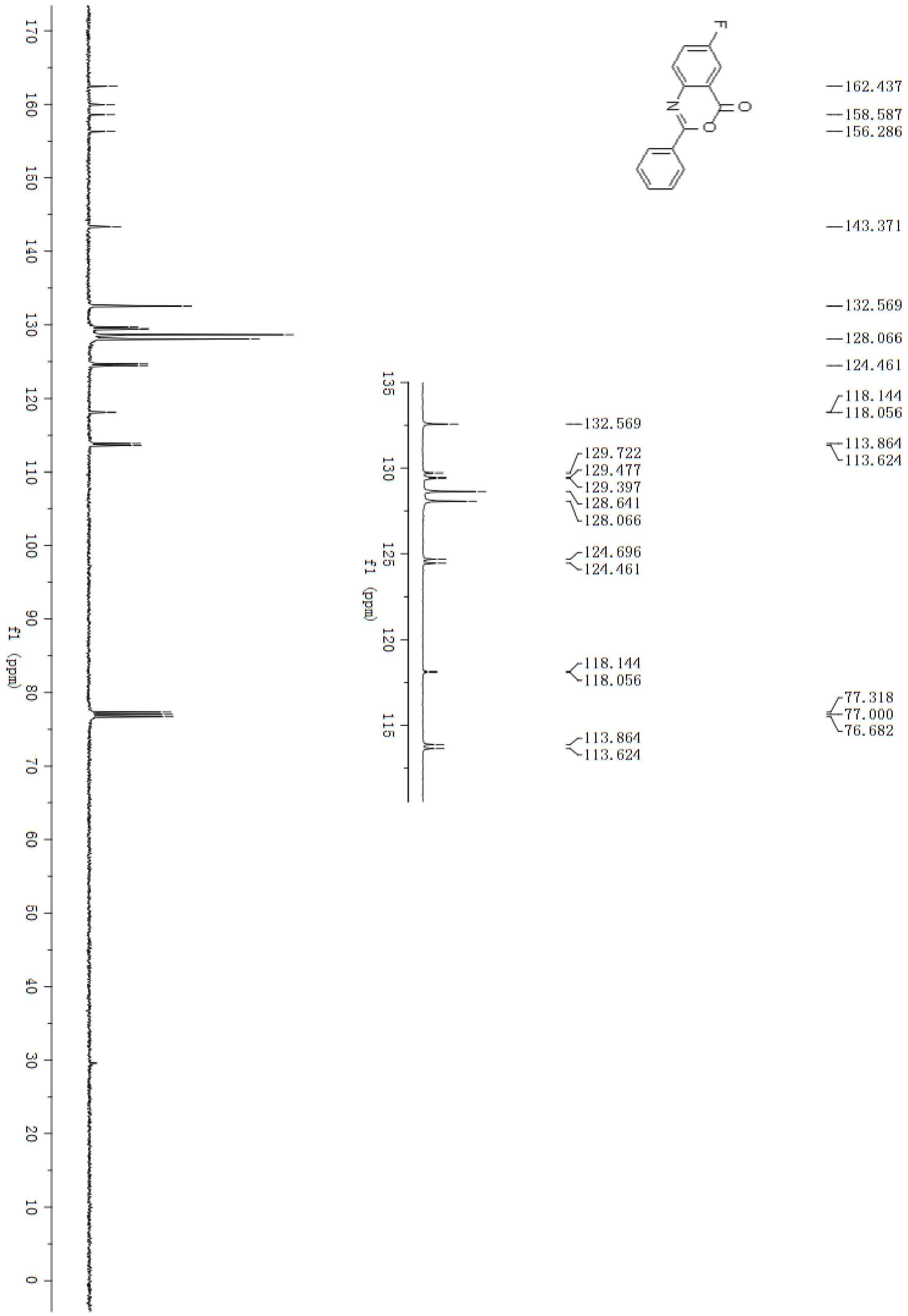


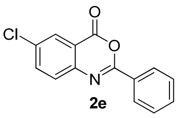
2d


8.133
8.114
7.732
7.713
7.568
7.556
7.546
7.535
7.464
7.445
7.428
7.415
7.409
7.389
7.371
7.353
2.00
1.12
1.26
4.37
f1 (ppm)

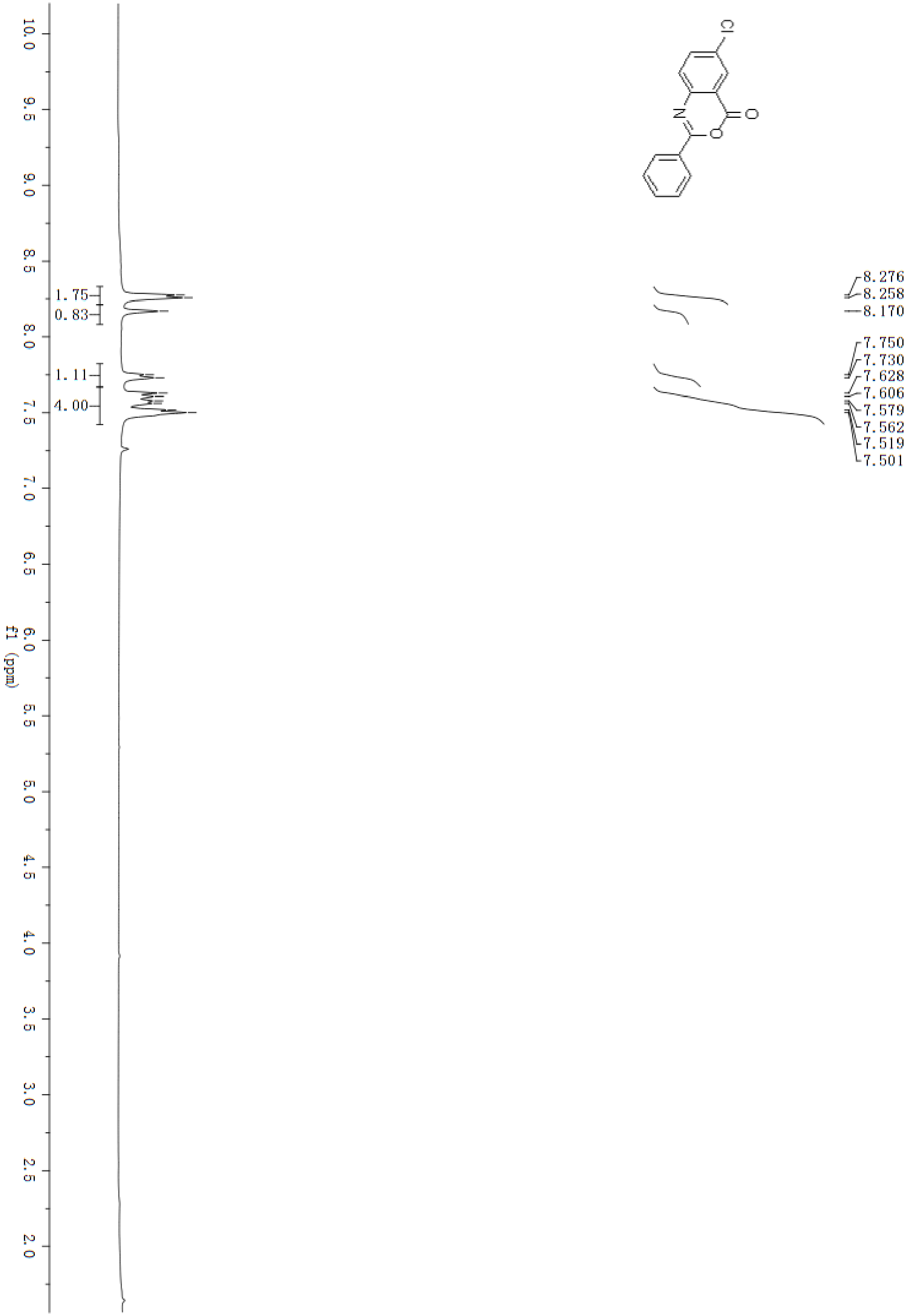


2d

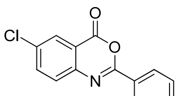

162.437
158.587
156.286
143.371
132.569
128.066
124.461
118.144
118.056
113.864
113.624
77.318
77.000
76.682
132.569
129.722
129.477
129.397
128.641
128.066
124.696
124.461
118.144
118.056
113.864
113.624
f1 (ppm)
f1 (ppm)



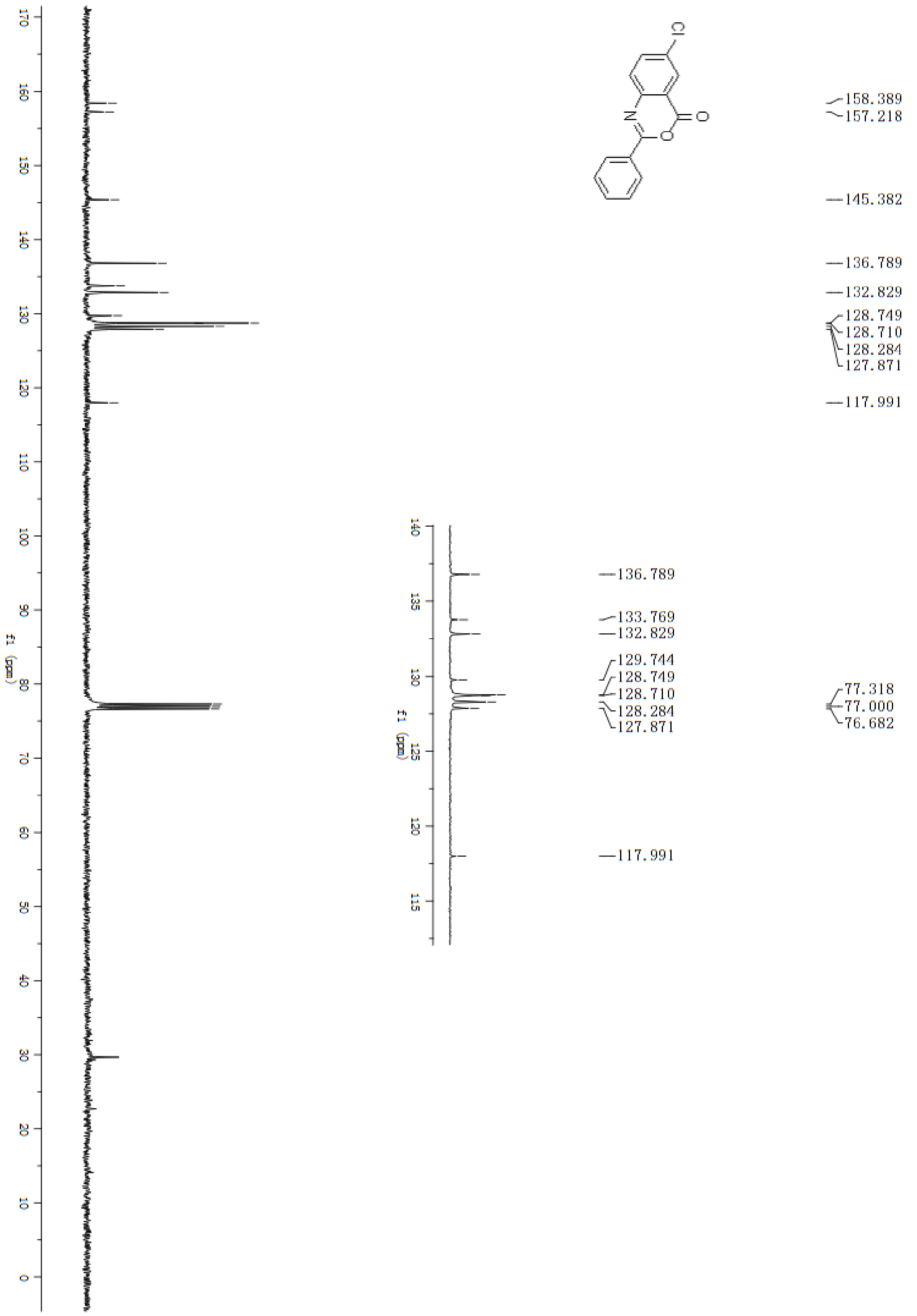
2e

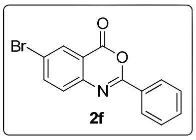


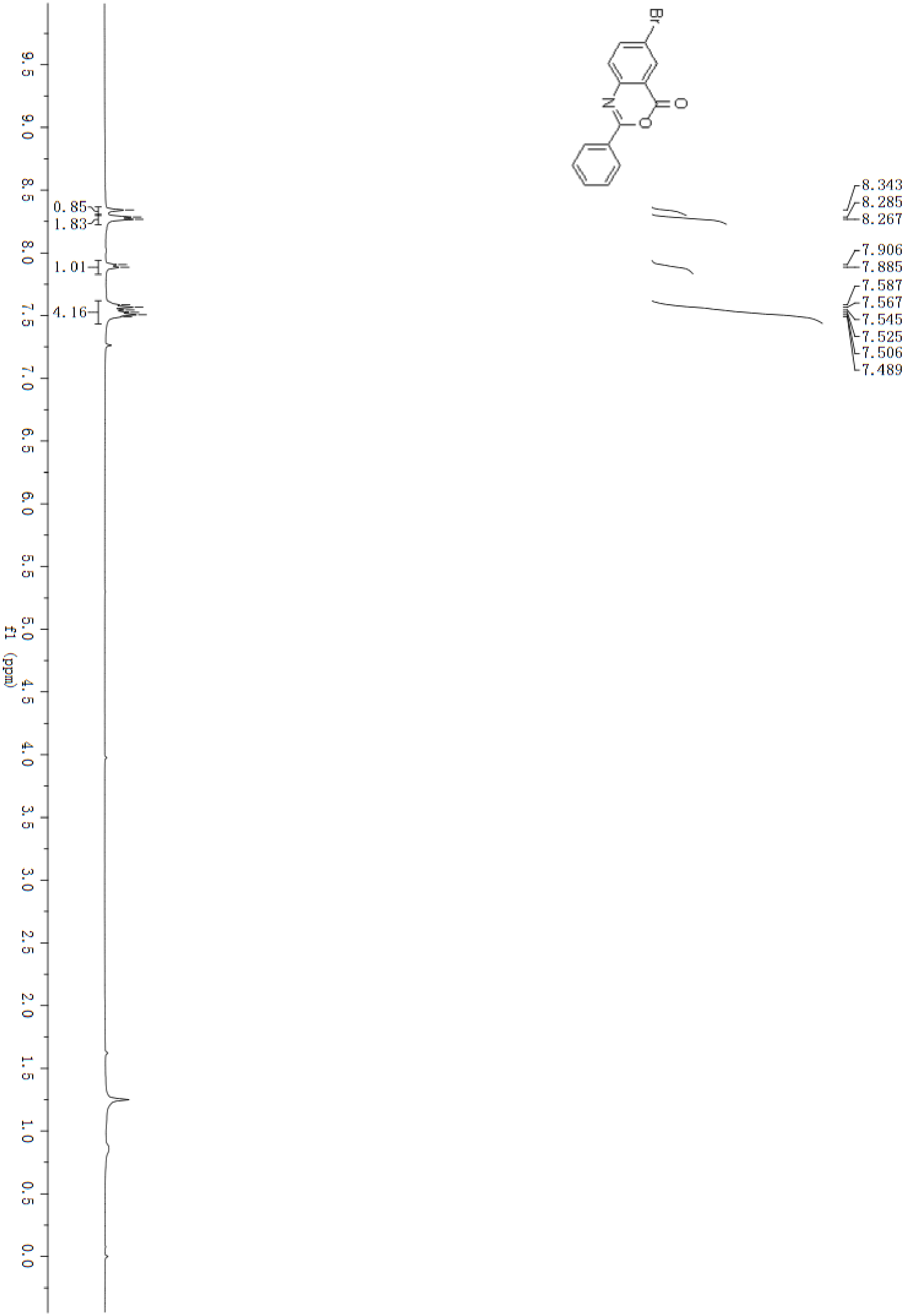
8.276
8.258
8.170
7.750
7.730
7.628
7.606
7.579
7.562
7.519
7.501
1.75
0.83
1.11
4.00
f1 (ppm)

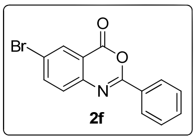

Cl
O
O
N

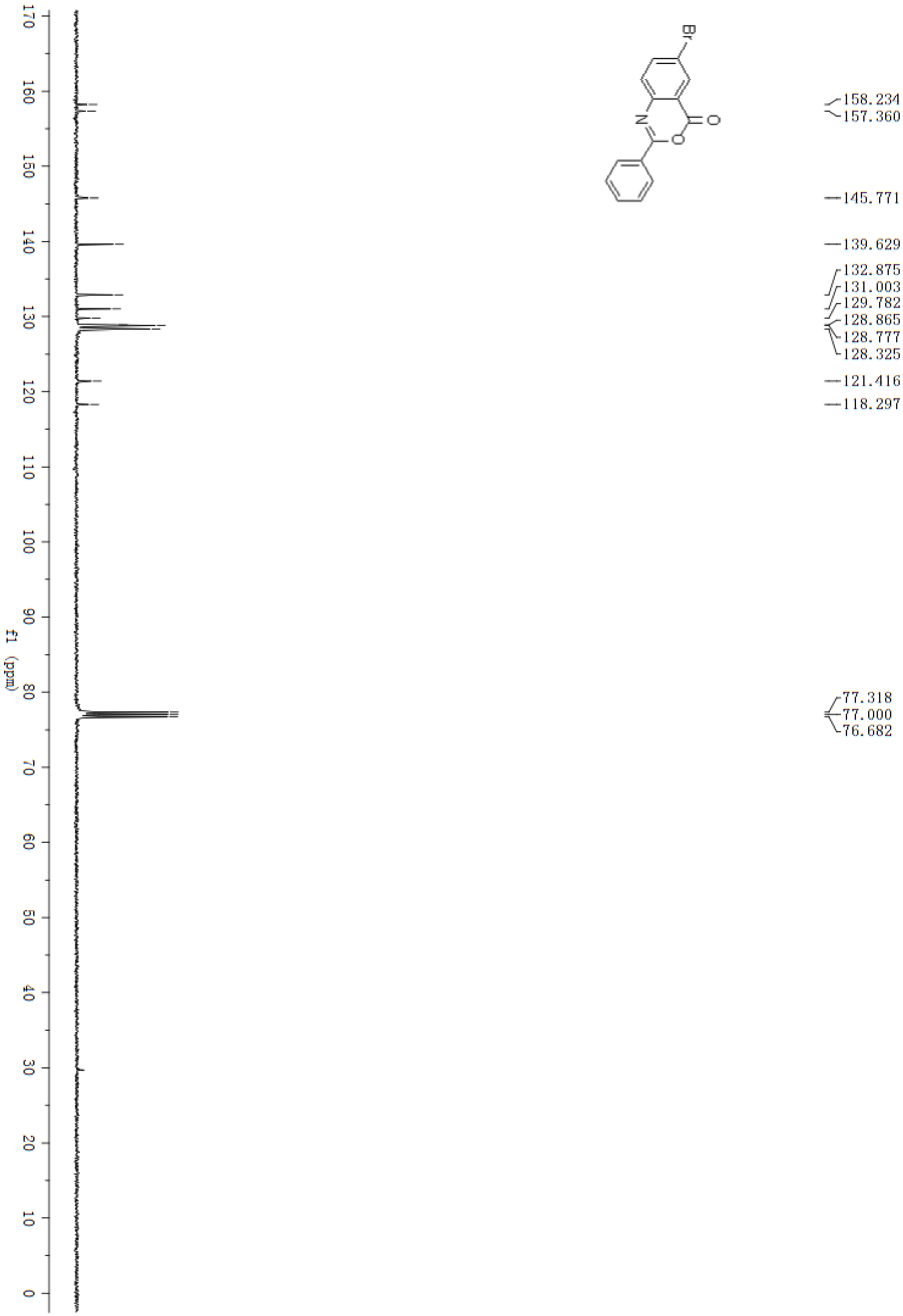
2e

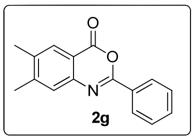

158.389
157.218
145.382
136.789
132.829
128.749
128.710
128.284
127.871
117.991
136.789
133.769
132.829
129.744
128.749
128.710
128.284
127.871
117.991
77.318
77.000
76.682
f1 (ppm)

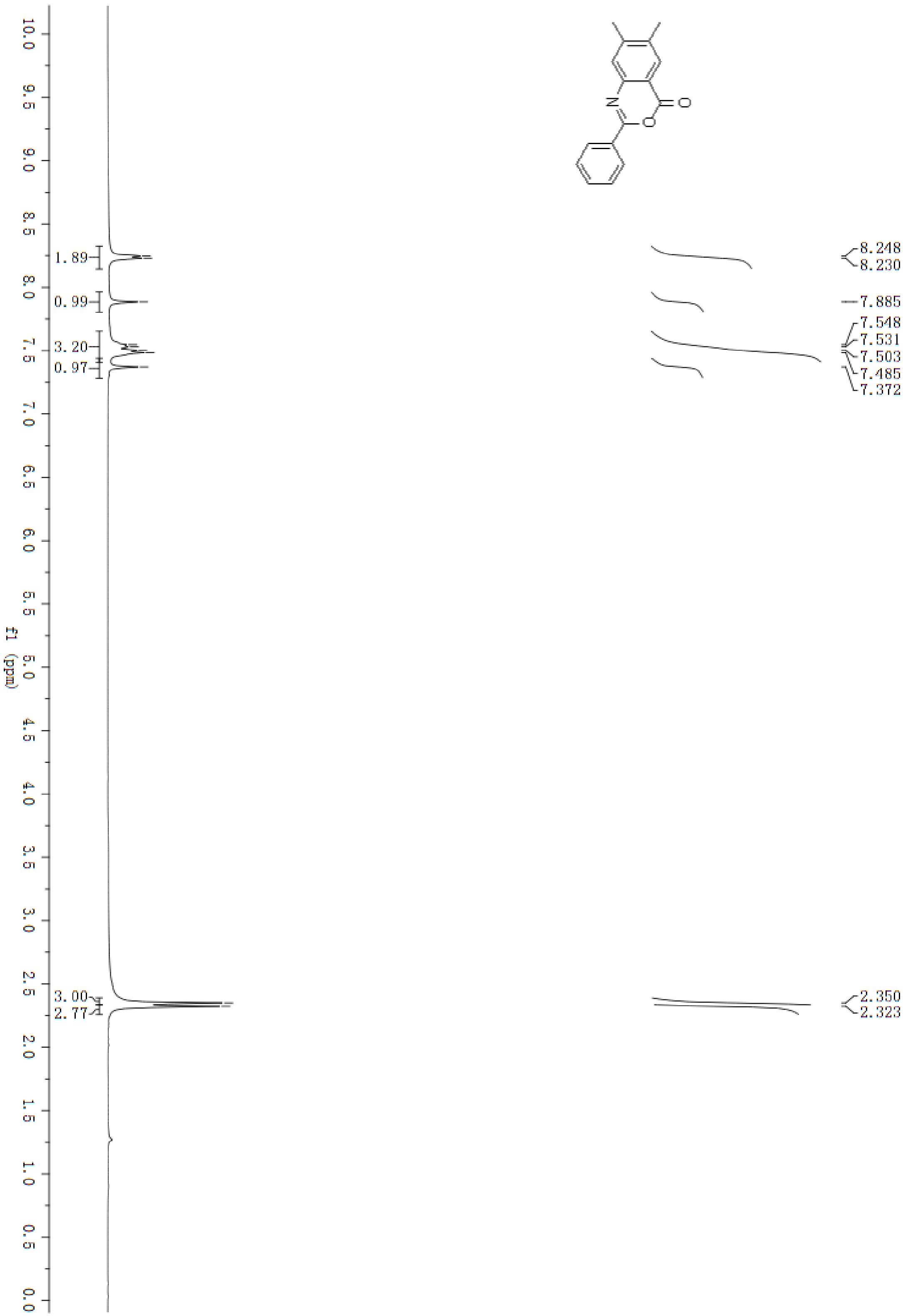

Br
O
O
N
2f

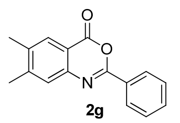

8.343
8.285
8.267
7.906
7.885
7.587
7.567
7.545
7.525
7.506
7.489
0.85
1.83
1.01
4.16
f1 (ppm)


Br
O
O
N
2f

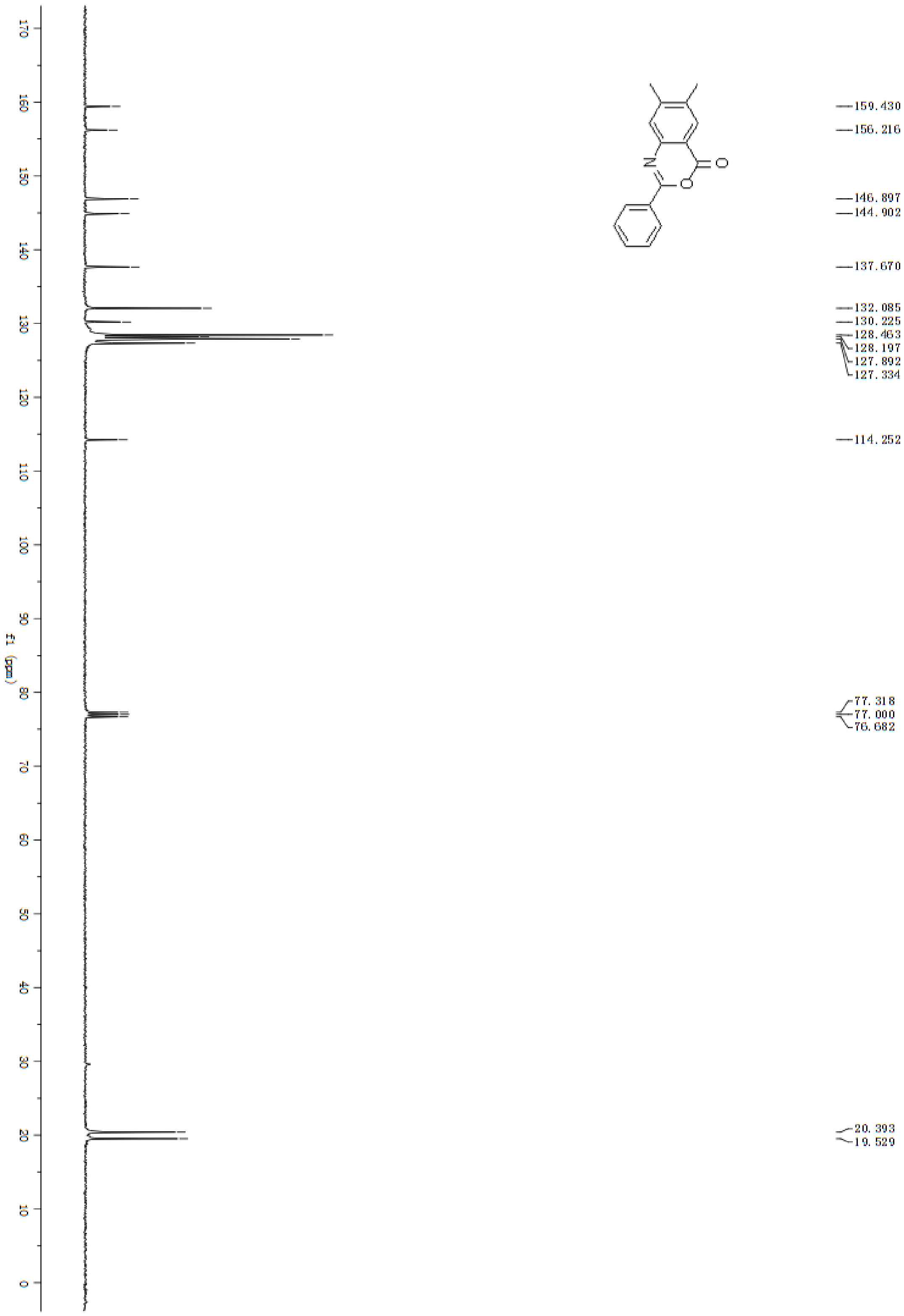

158.234
157.360
145.771
139.629
132.875
131.003
129.782
128.865
128.777
128.325
121.416
118.297
77.318
77.000
76.682
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Br
O
O
N

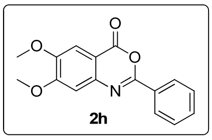

O
O
N
2g


8.248
8.230
7.885
7.548
7.531
7.503
7.485
7.372
2.350
2.323
1.89
0.99
3.20
0.97
3.00
2.77
f1 (ppm)

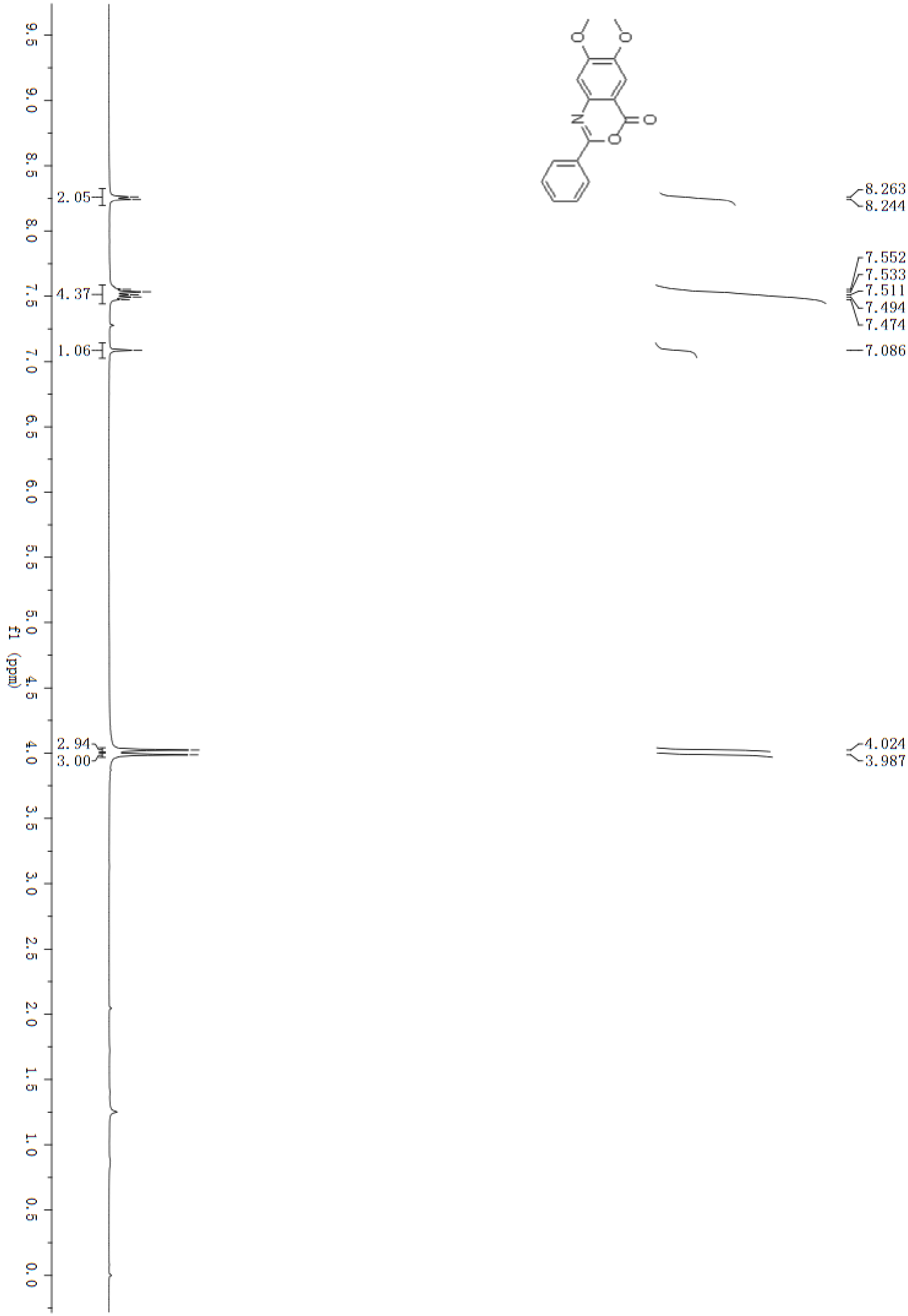


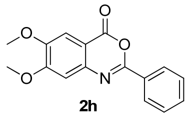
2g


159.430
156.216
146.897
144.902
137.670
132.085
130.225
128.463
128.197
127.892
127.334
114.252
77.318
77.000
76.682
20.393
19.529
f1 (ppm)

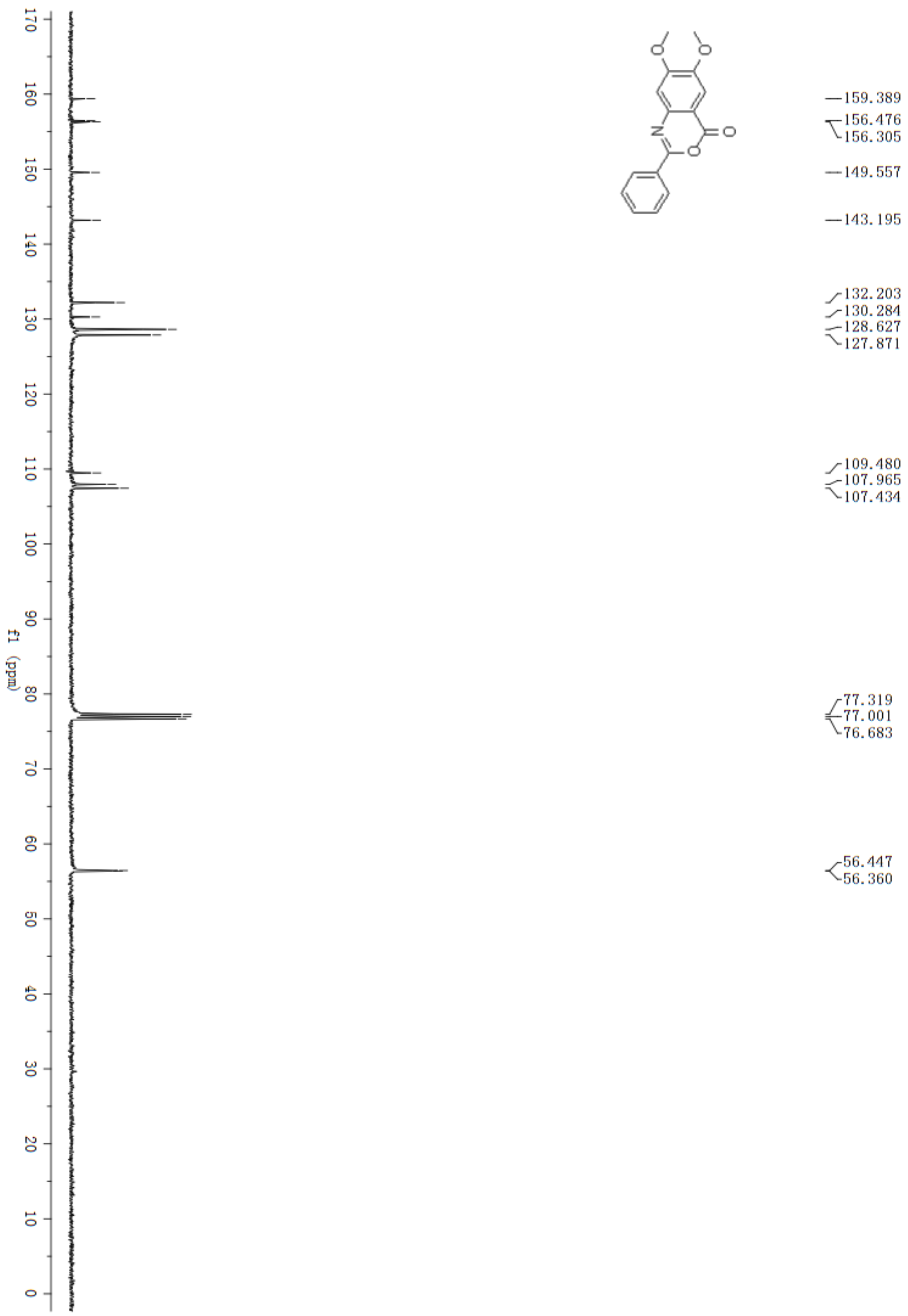


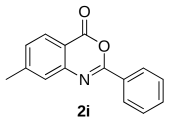
2h


8.263
8.244
7.552
7.533
7.511
7.494
7.474
7.086
4.024
3.987
2.05
4.37
1.06
2.94
3.00
f1 (ppm)

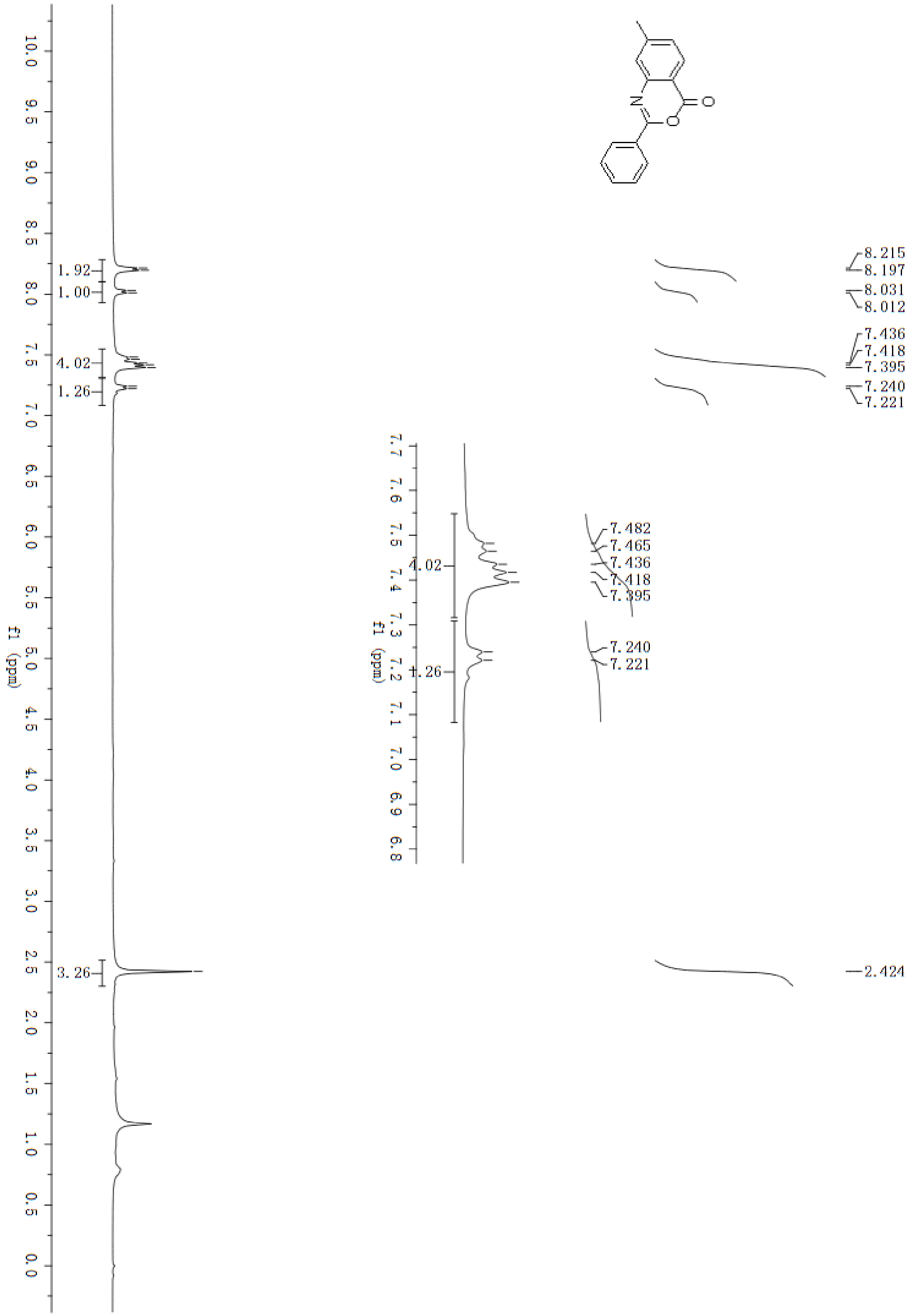


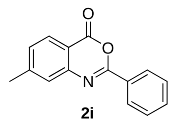
2h


159.389
156.476
156.305
149.557
143.195
132.203
130.284
128.627
127.871
109.480
107.965
107.434
77.319
77.001
76.683
56.447
56.360
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

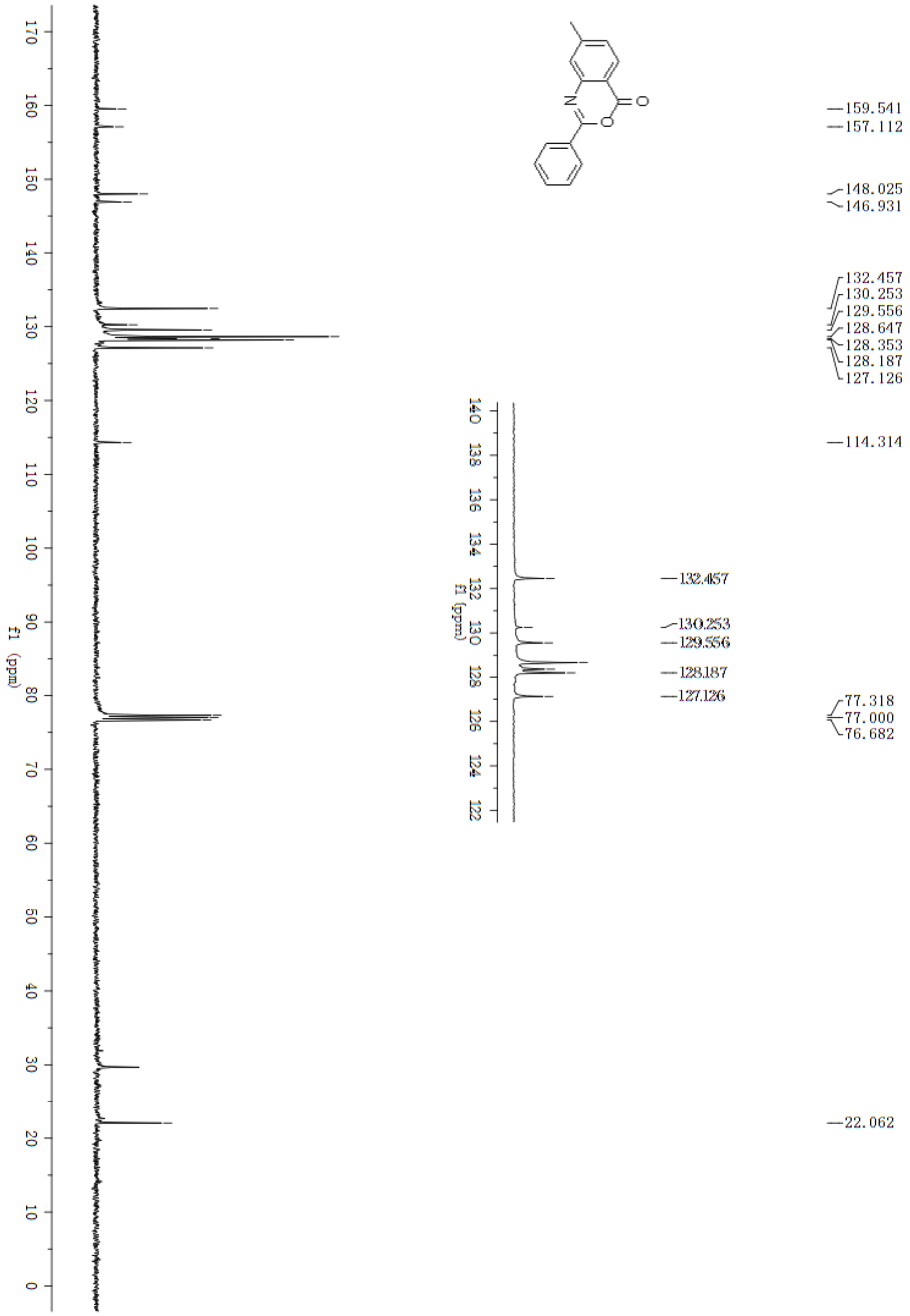


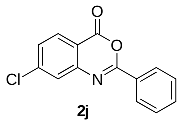
2i


8.215
8.197
8.031
8.012
7.436
7.418
7.395
7.240
7.221
2.424
7.482
7.465
7.436
7.418
7.395
7.240
7.221
1.92
1.00
4.02
1.26
3.26
4.02
1.26
f1 (ppm)
f1 (ppm)

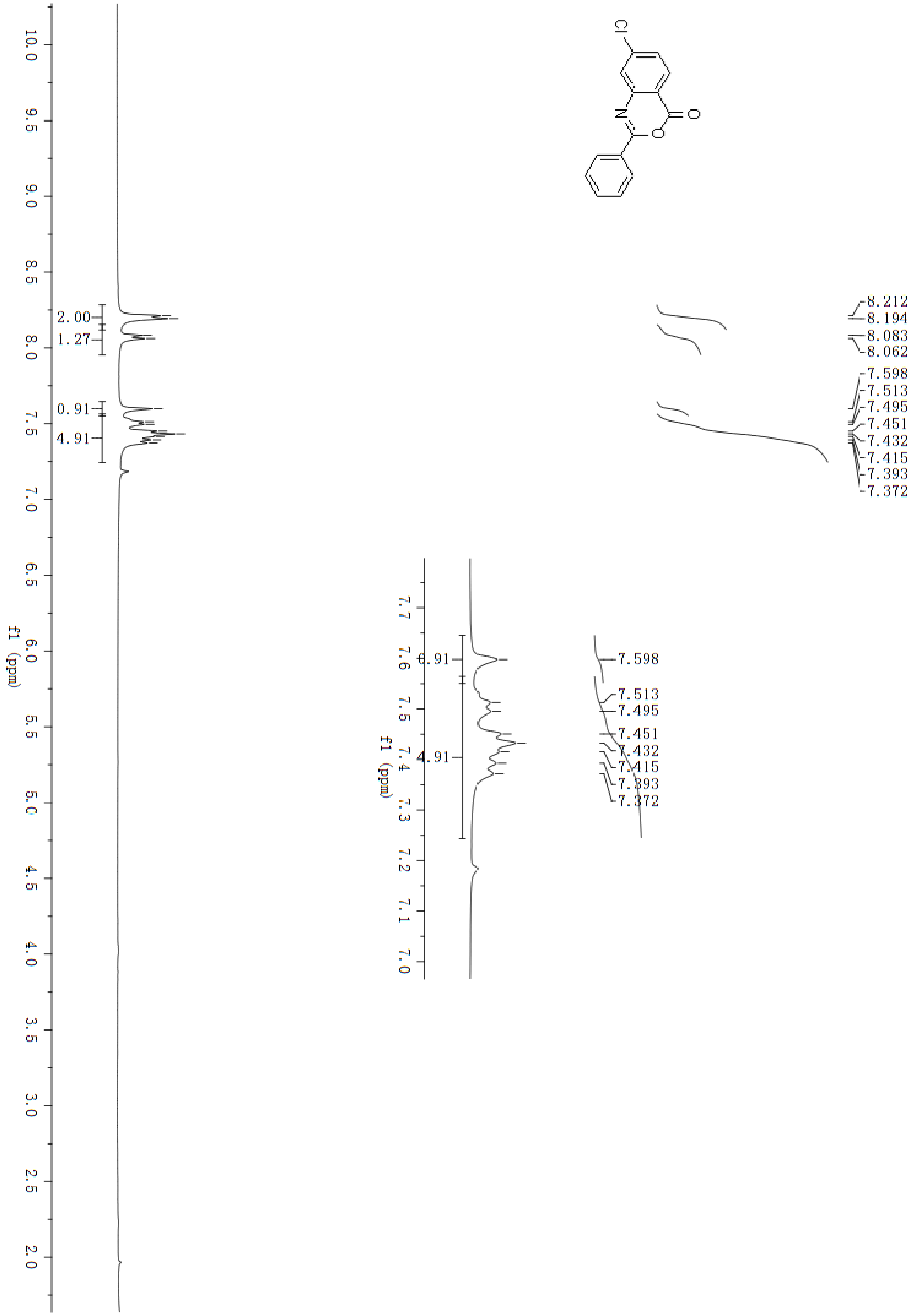


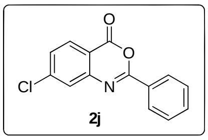
2i

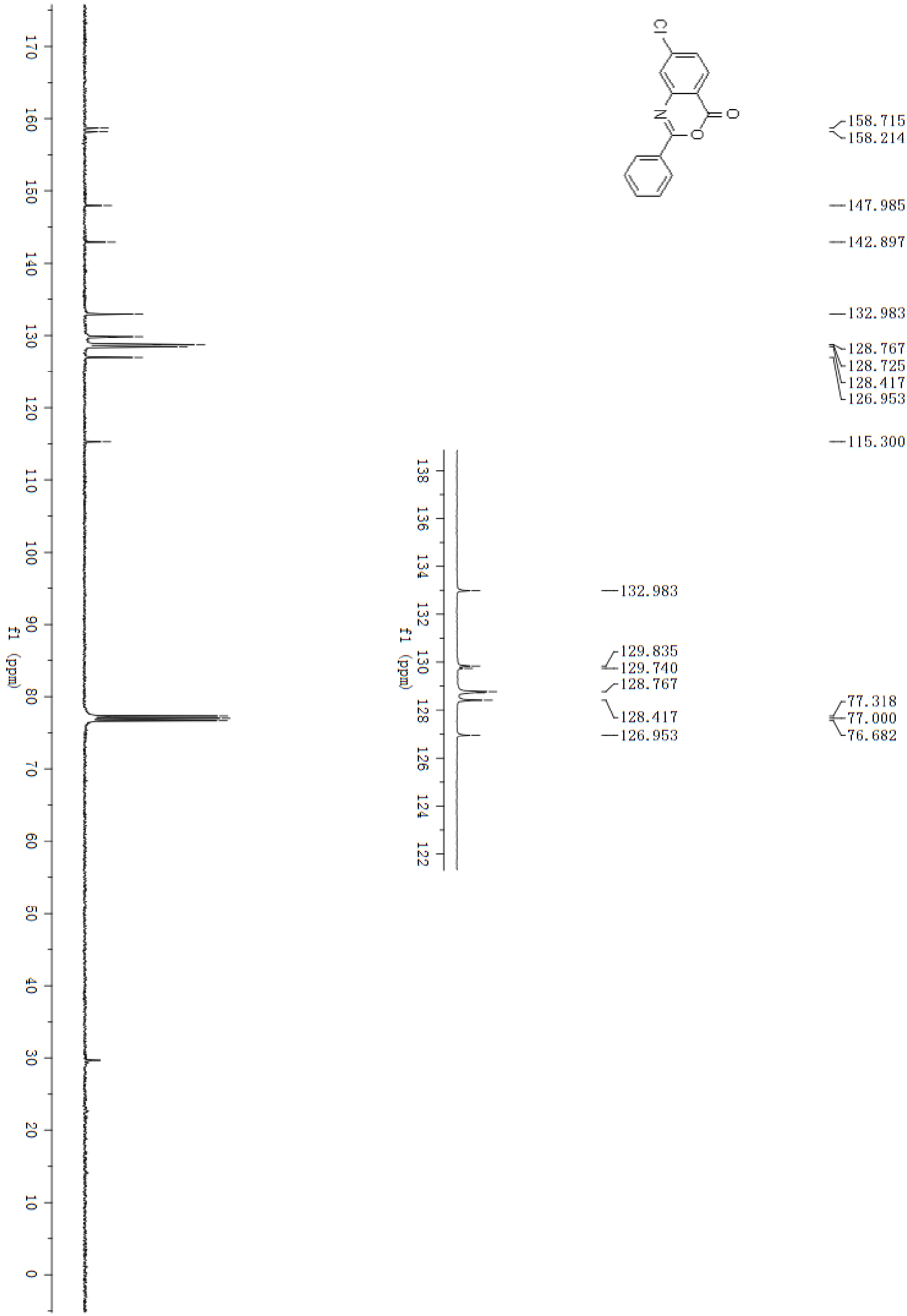

159.541
157.112
148.025
146.931
132.457
130.253
129.556
128.647
128.353
128.187
127.126
114.314
77.318
77.000
76.682
22.062
132.457
130.253
129.556
128.187
127.126
f1 (ppm)
f1 (ppm)

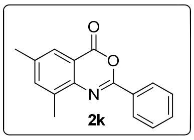


2j

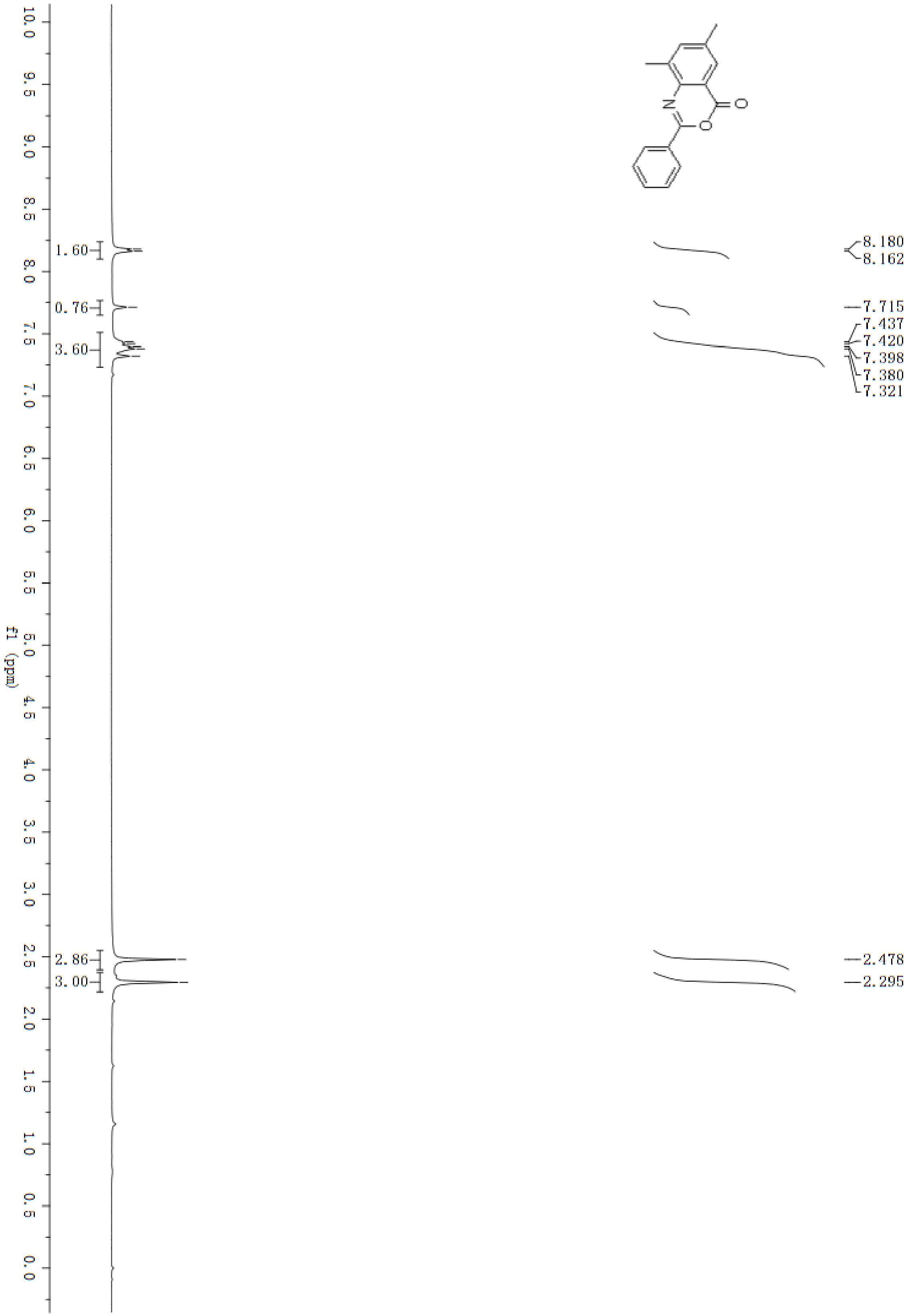

8.212
8.194
8.083
8.062
7.598
7.513
7.495
7.451
7.432
7.415
7.393
7.372
2.00
1.27
0.91
4.91
f1 (ppm)

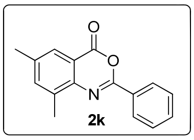

2j

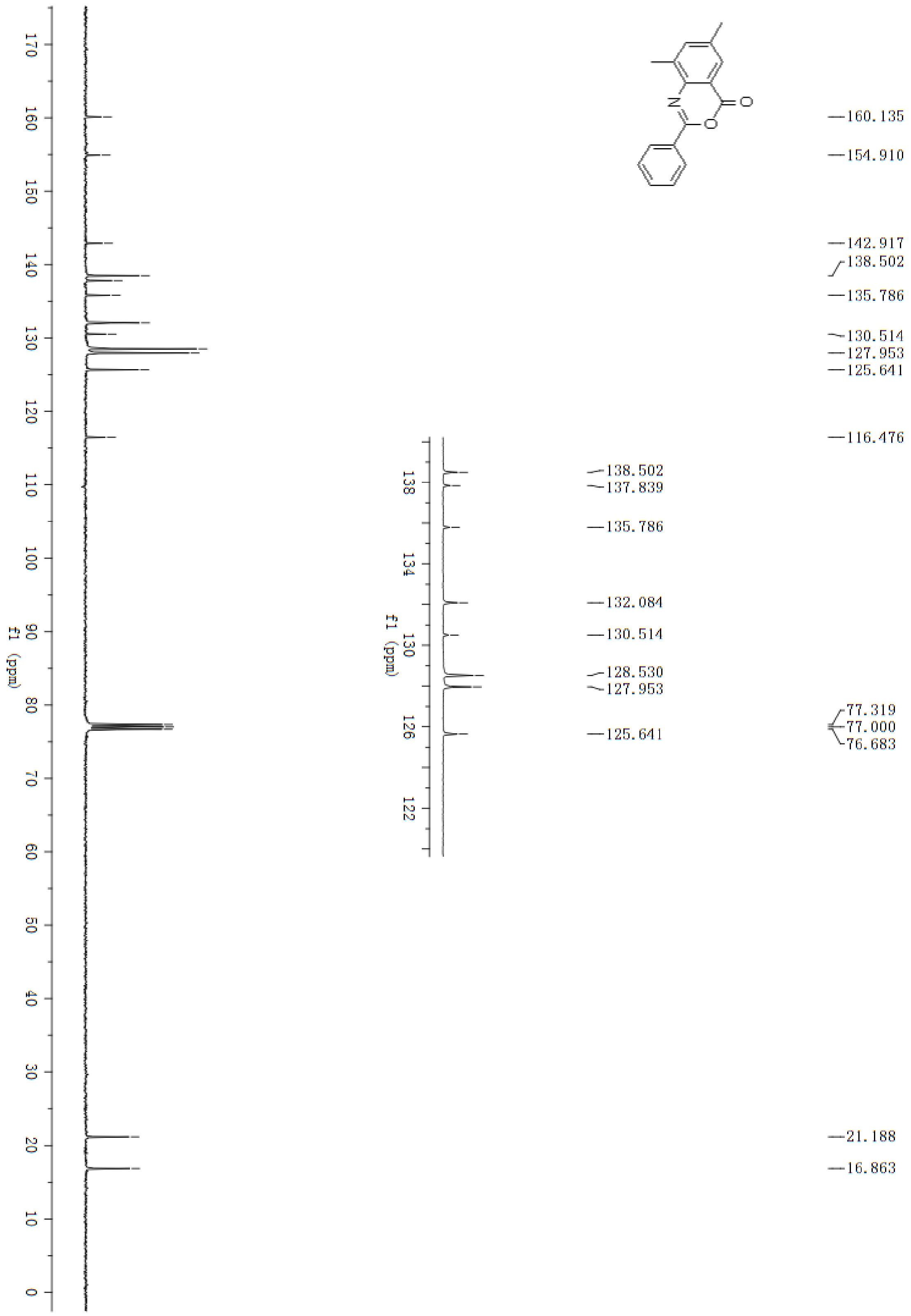

158.715
158.214
147.985
142.897
132.983
128.767
128.725
128.417
126.953
115.300
132.983
129.835
129.740
128.767
128.417
126.953
77.318
77.000
76.682
f1 (ppm)
f1 (ppm)

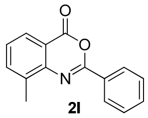


2k

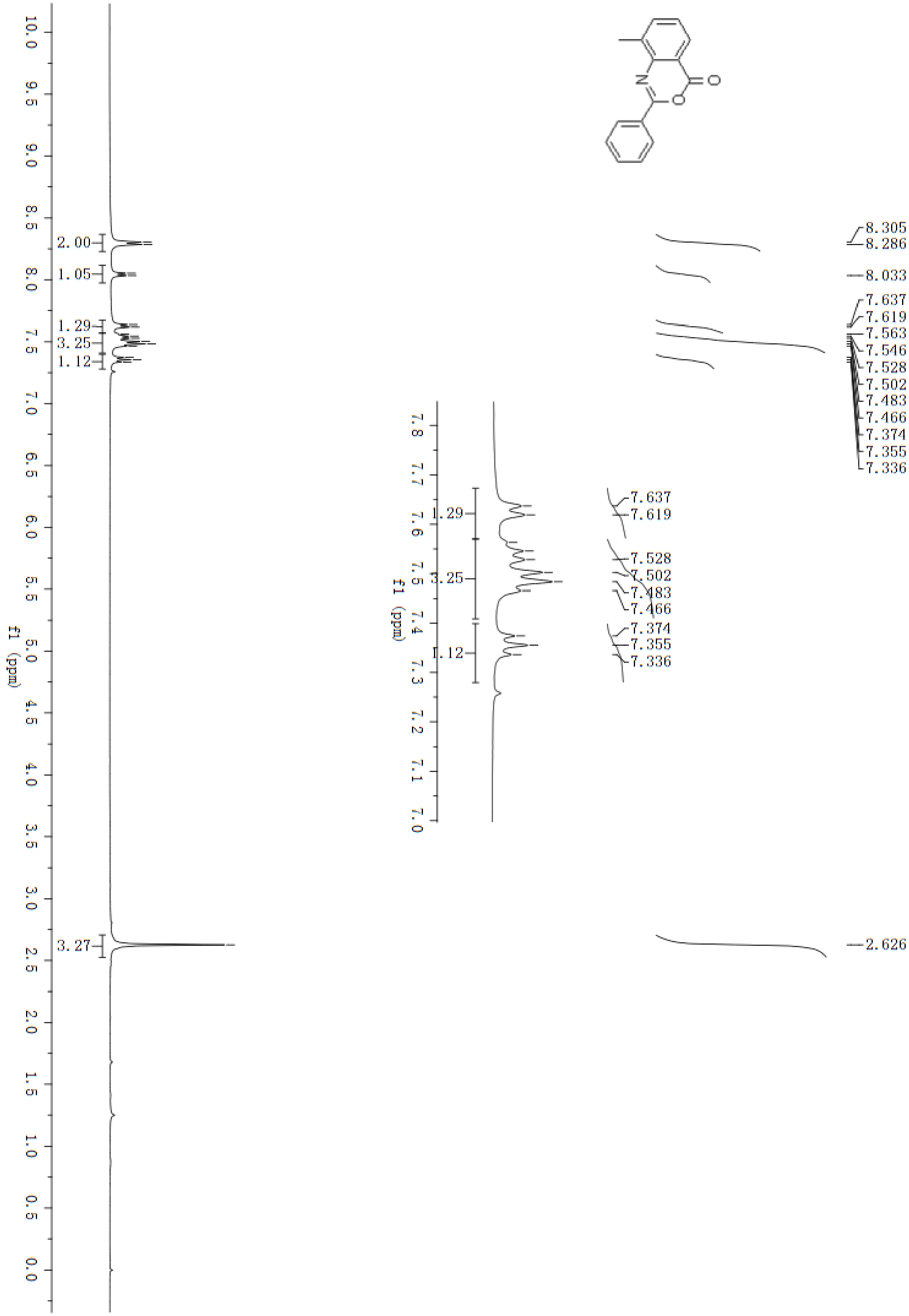

8.180
8.162
7.715
7.437
7.420
7.398
7.380
7.321
2.478
2.295
1.60
0.76
3.60
2.86
3.00
f1 (ppm)

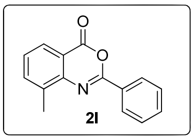

2k


160.135
154.910
142.917
138.502
135.786
130.514
127.953
125.641
116.476
77.319
77.000
76.683
21.188
16.863
138.502
137.839
135.786
132.084
130.514
128.530
127.953
125.641
f1 (ppm)

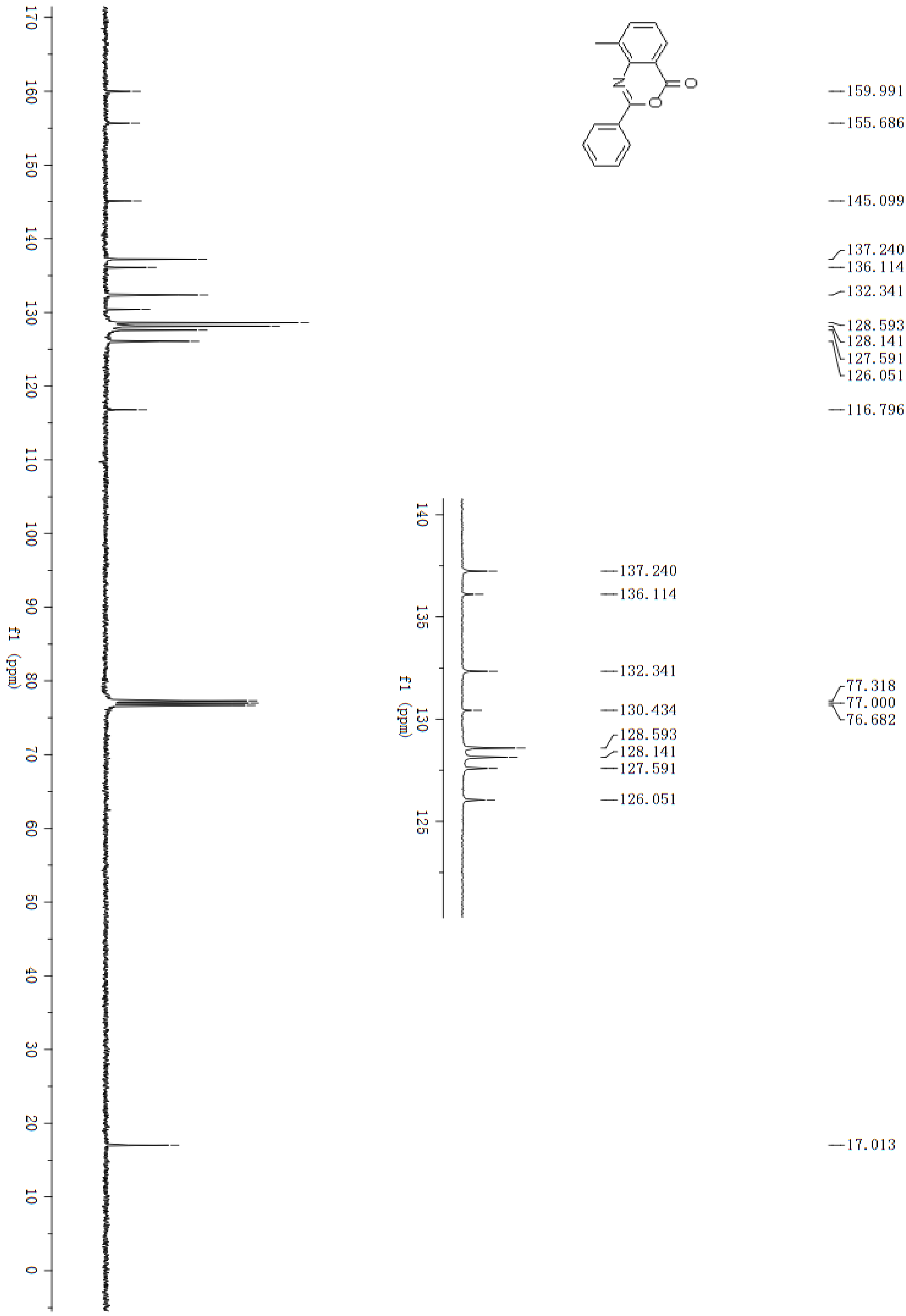


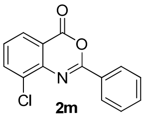
2l


8.305
8.286
8.033
7.637
7.619
7.563
7.546
7.528
7.502
7.483
7.466
7.374
7.355
7.336
2.626
2.00
1.05
1.29
3.25
1.12
3.27
f1 (ppm)

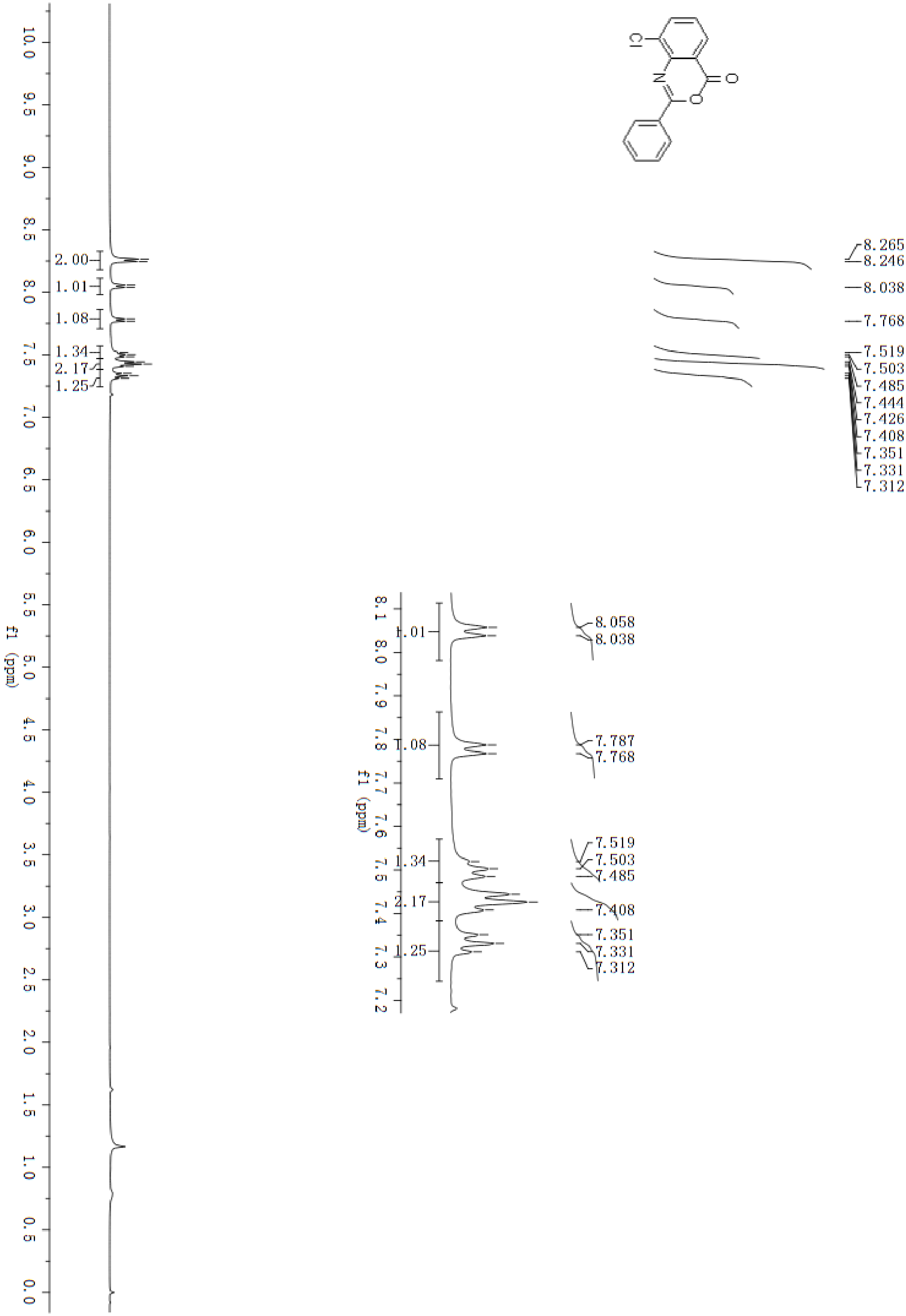

O
O
N

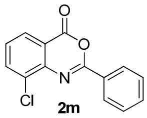
2l


159.991
155.686
145.099
137.240
136.114
132.341
128.593
128.141
127.591
126.051
116.796
137.240
136.114
132.341
130.434
128.593
128.141
127.591
126.051
77.318
77.000
76.682
17.013
f1 (ppm)
f1 (ppm)

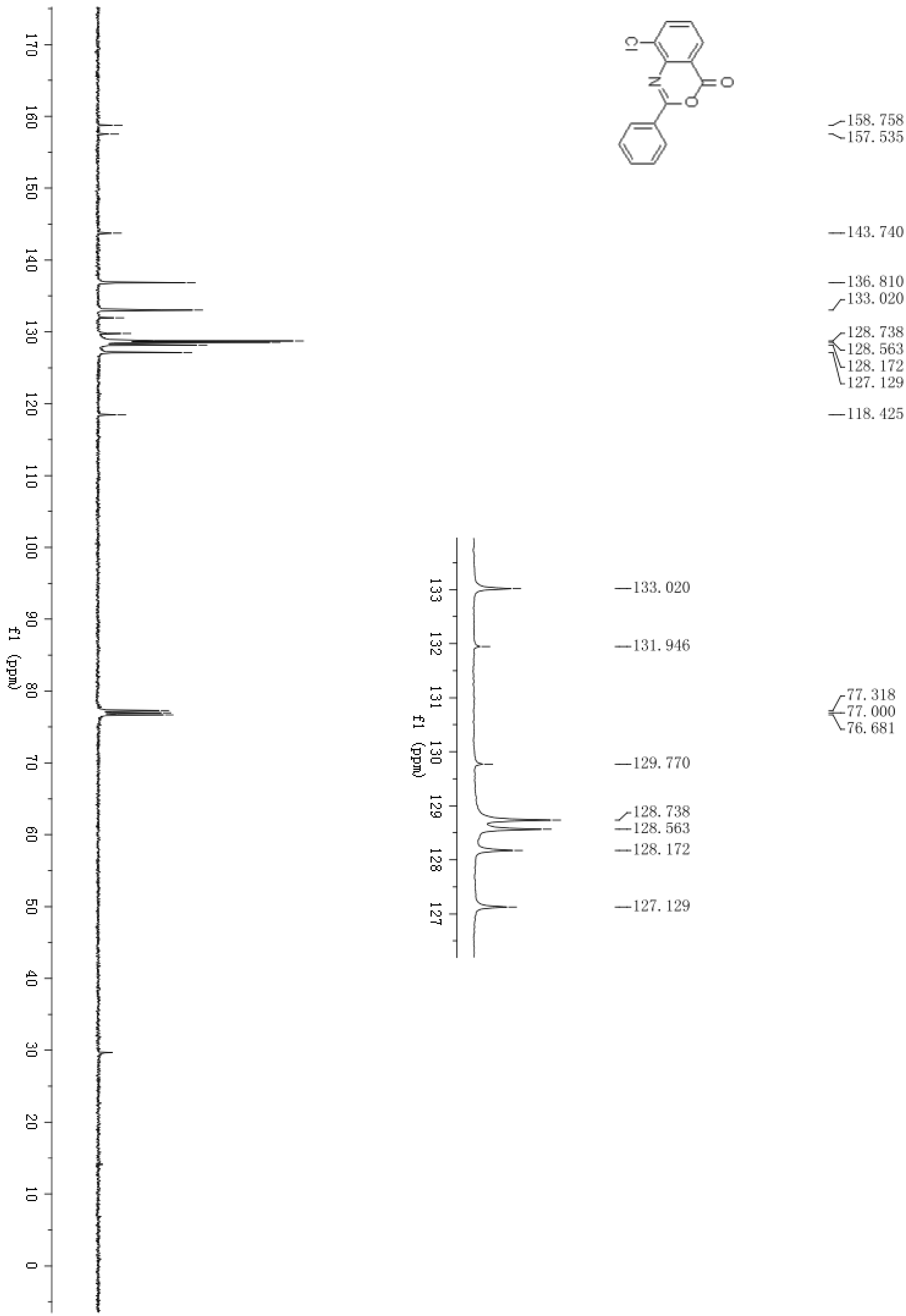


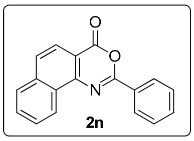
2m

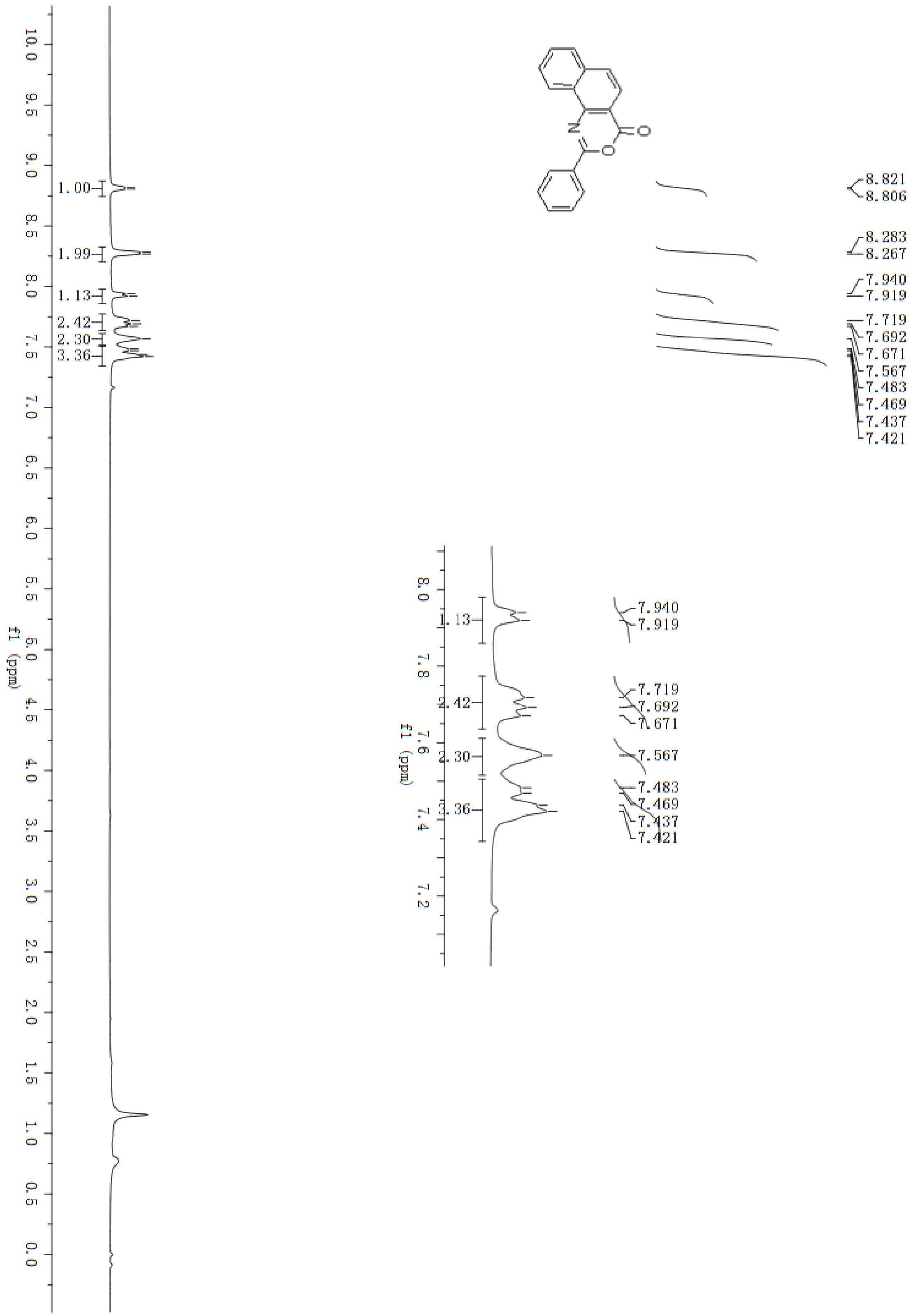

8.265
8.246
8.038
7.768
7.519
7.503
7.485
7.444
7.426
7.408
7.351
7.331
7.312
2.00
1.01
1.08
1.34
2.17
1.25
f1 (ppm)
8.058
8.038
7.787
7.768
7.519
7.503
7.485
7.408
7.351
7.331
7.312

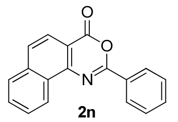


2m

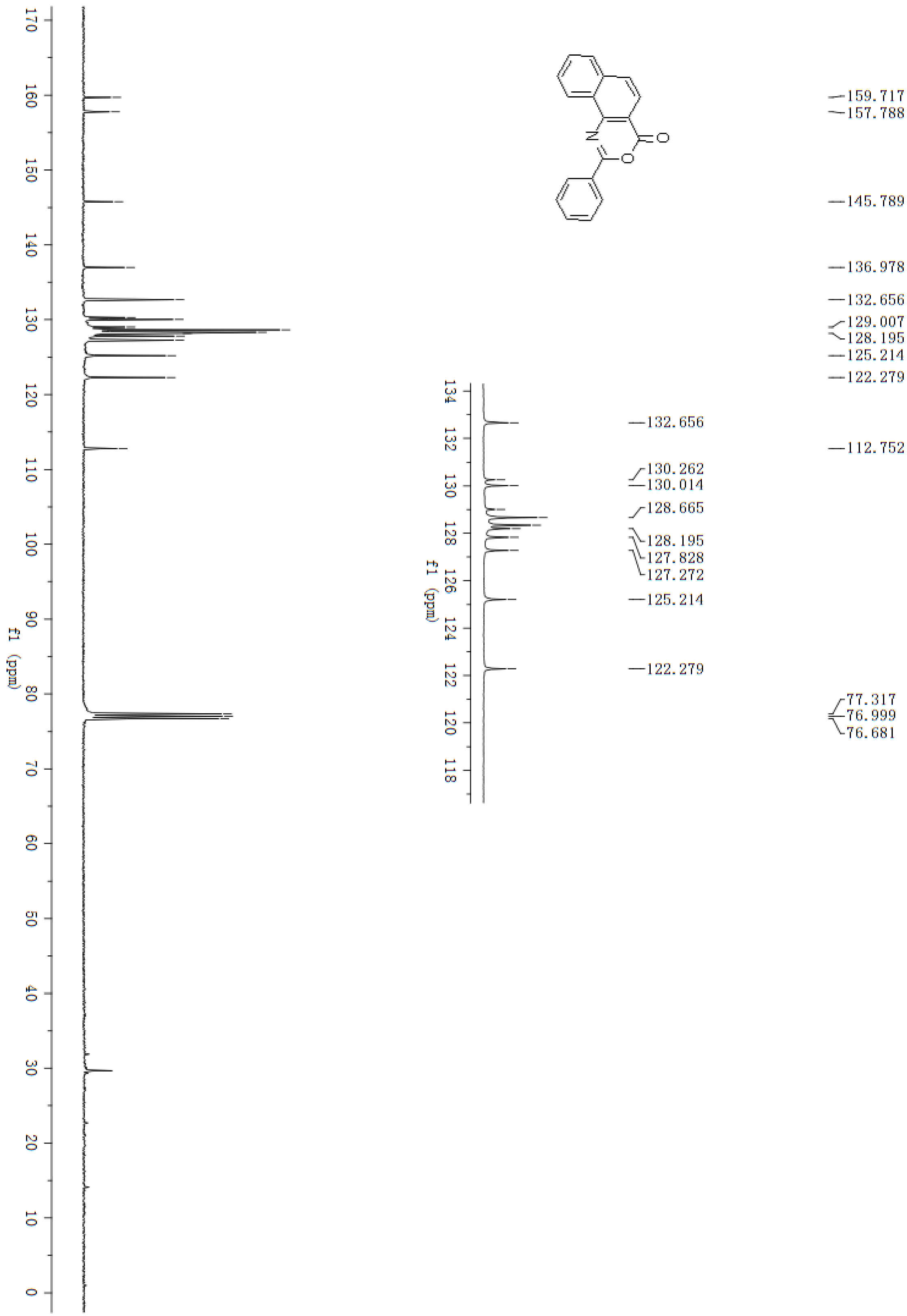

158.758
157.535
143.740
136.810
133.020
128.738
128.563
128.172
127.129
118.425
77.318
77.000
76.681
133.020
131.946
129.770
128.738
128.563
128.172
127.129
f1 (ppm)
f1 (ppm)

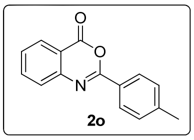

2n


8.821
8.806
8.283
8.267
7.940
7.919
7.719
7.692
7.671
7.567
7.483
7.469
7.437
7.421
1.00
1.99
1.13
2.42
2.30
3.36
f1 (ppm)

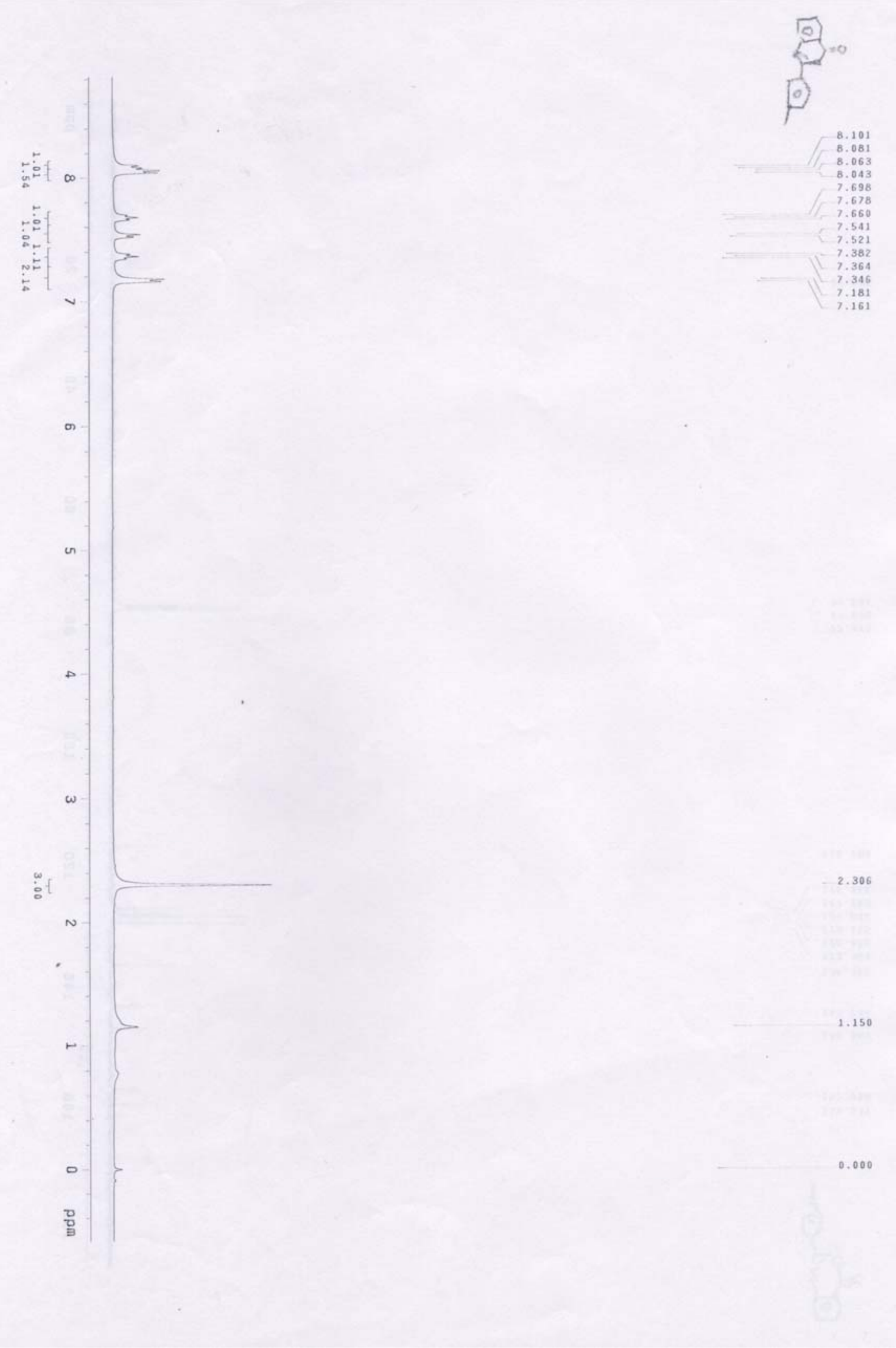


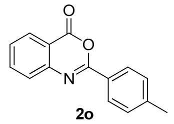
2n


159.717
157.788
145.789
136.978
132.656
129.007
128.195
125.214
122.279
112.752
77.317
76.999
76.681
132.656
130.262
130.014
128.665
128.195
127.828
127.272
125.214
122.279
f1 (ppm)
f1 (ppm)

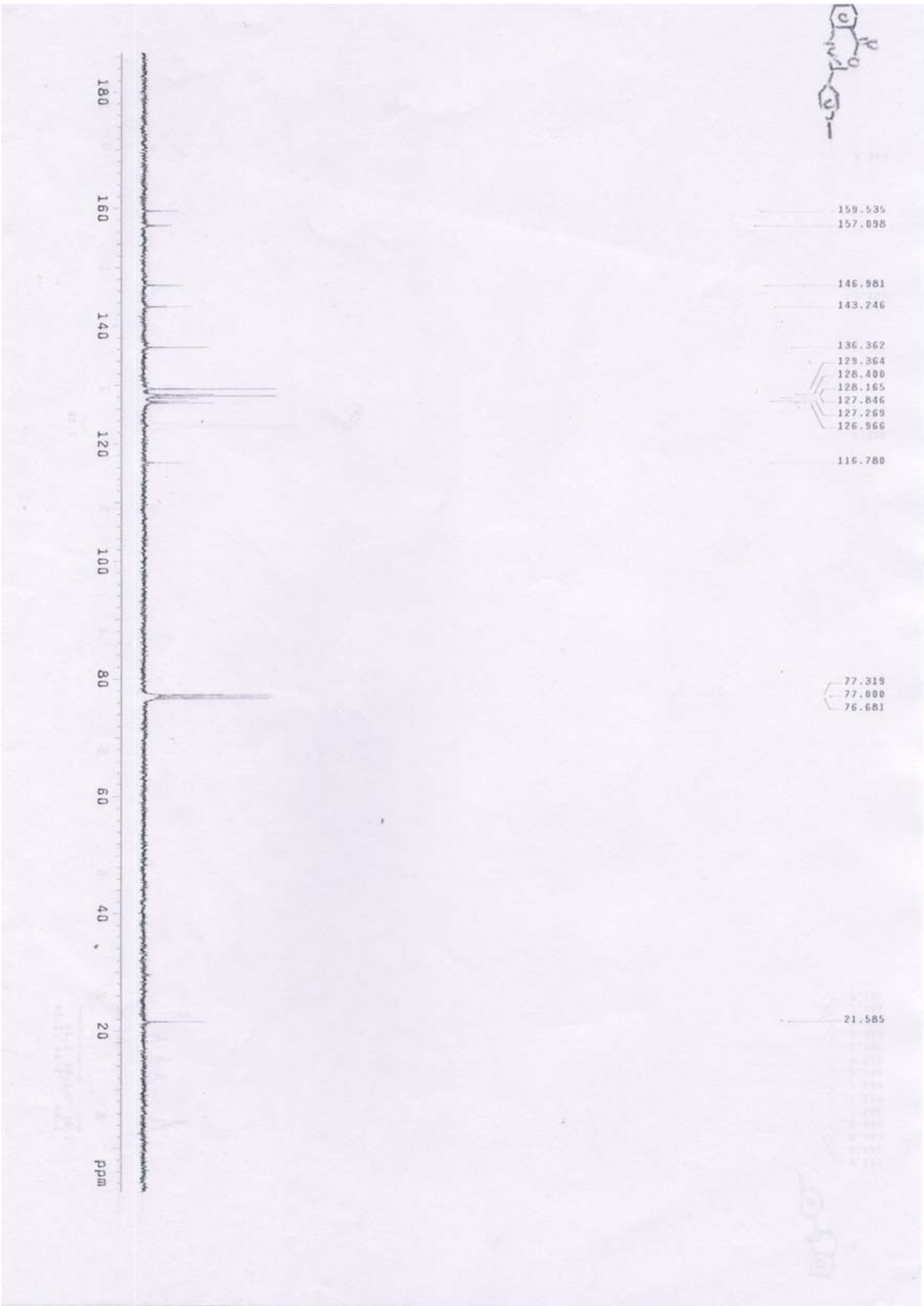


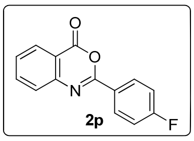
2o

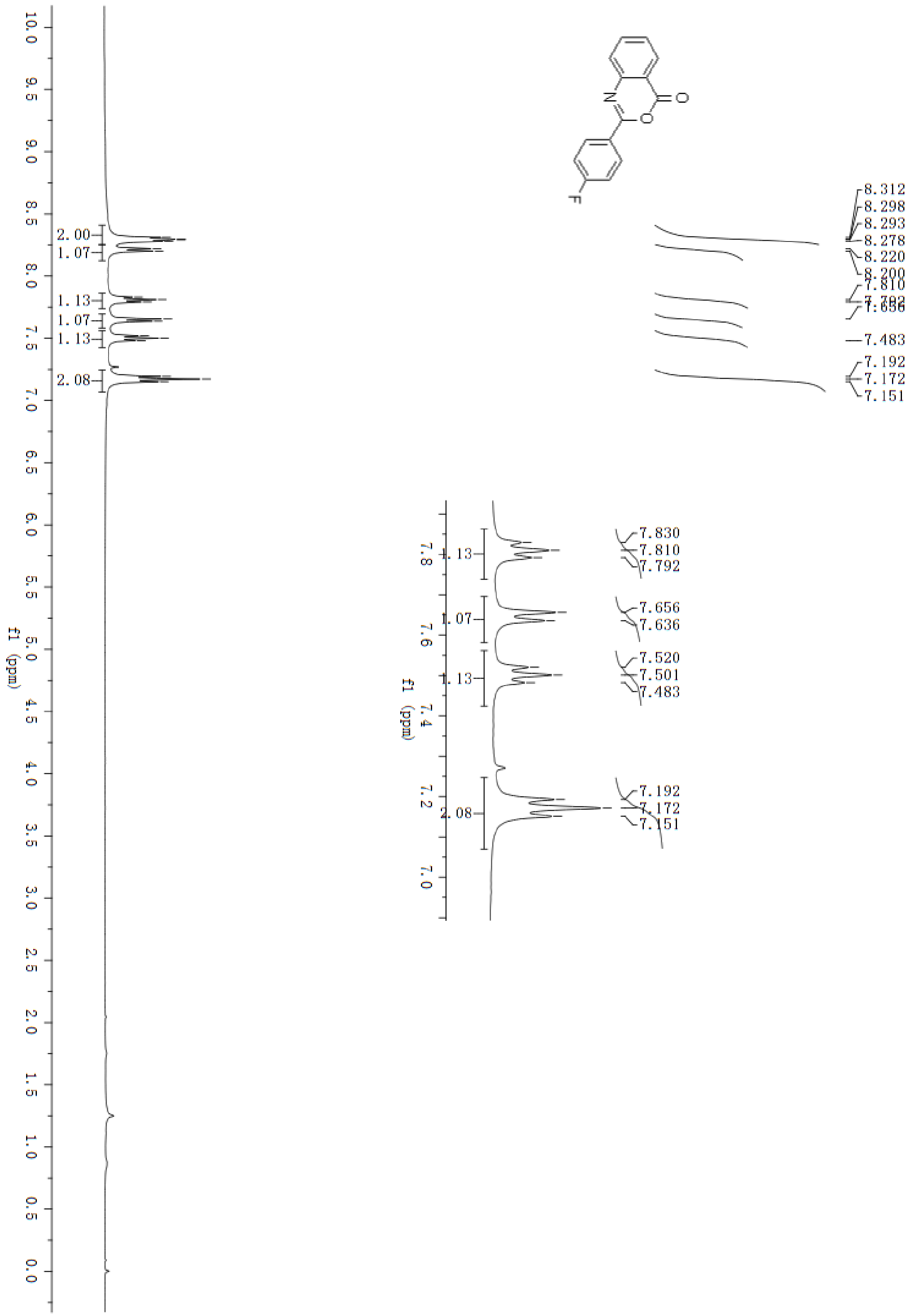


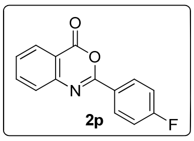


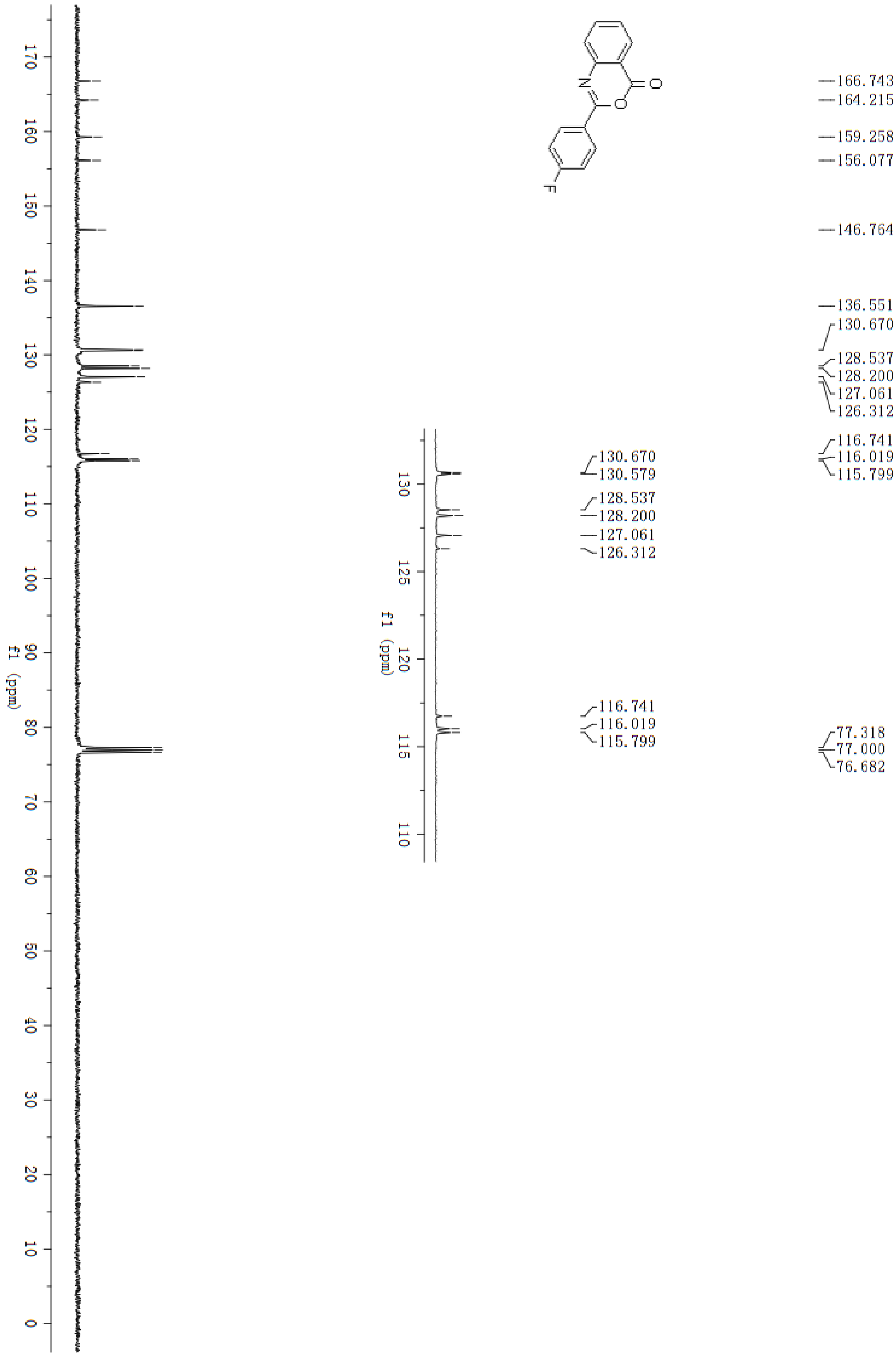
2o

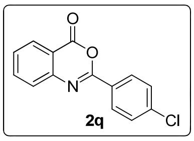

159.535
157.098
146.981
143.246
136.362
129.364
128.400
128.165
127.846
127.269
126.966
116.780
77.319
77.000
76.681
21.585
180
160
140
120
100
80
60
40
20
ppm

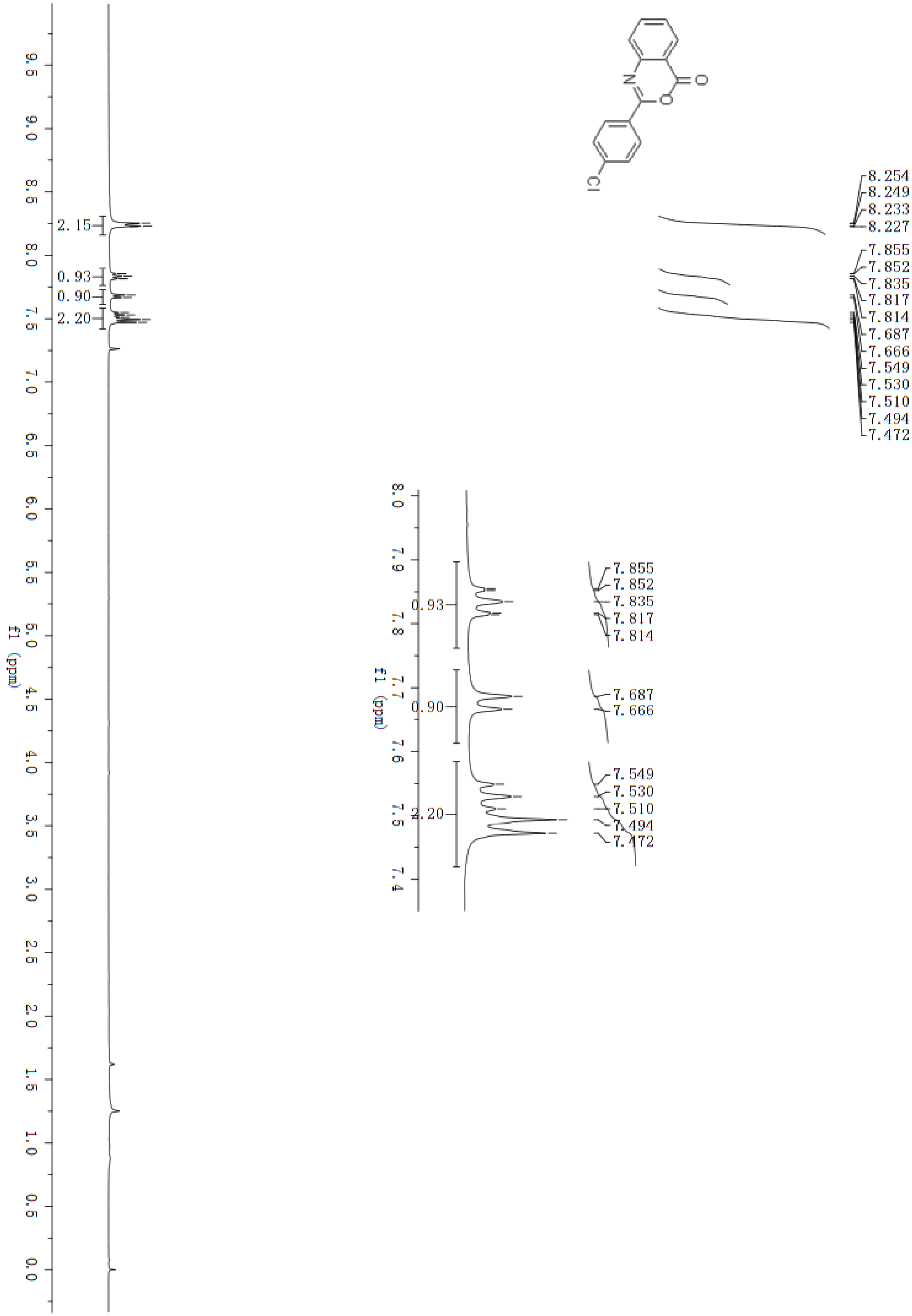

2p

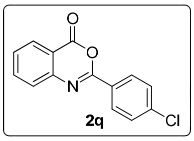

8.312
8.298
8.293
8.278
8.220
8.200
7.810
7.483
7.192
7.172
7.151
7.830
7.810
7.792
7.656
7.636
7.520
7.501
7.483
7.192
7.172
7.151
f1 (ppm)

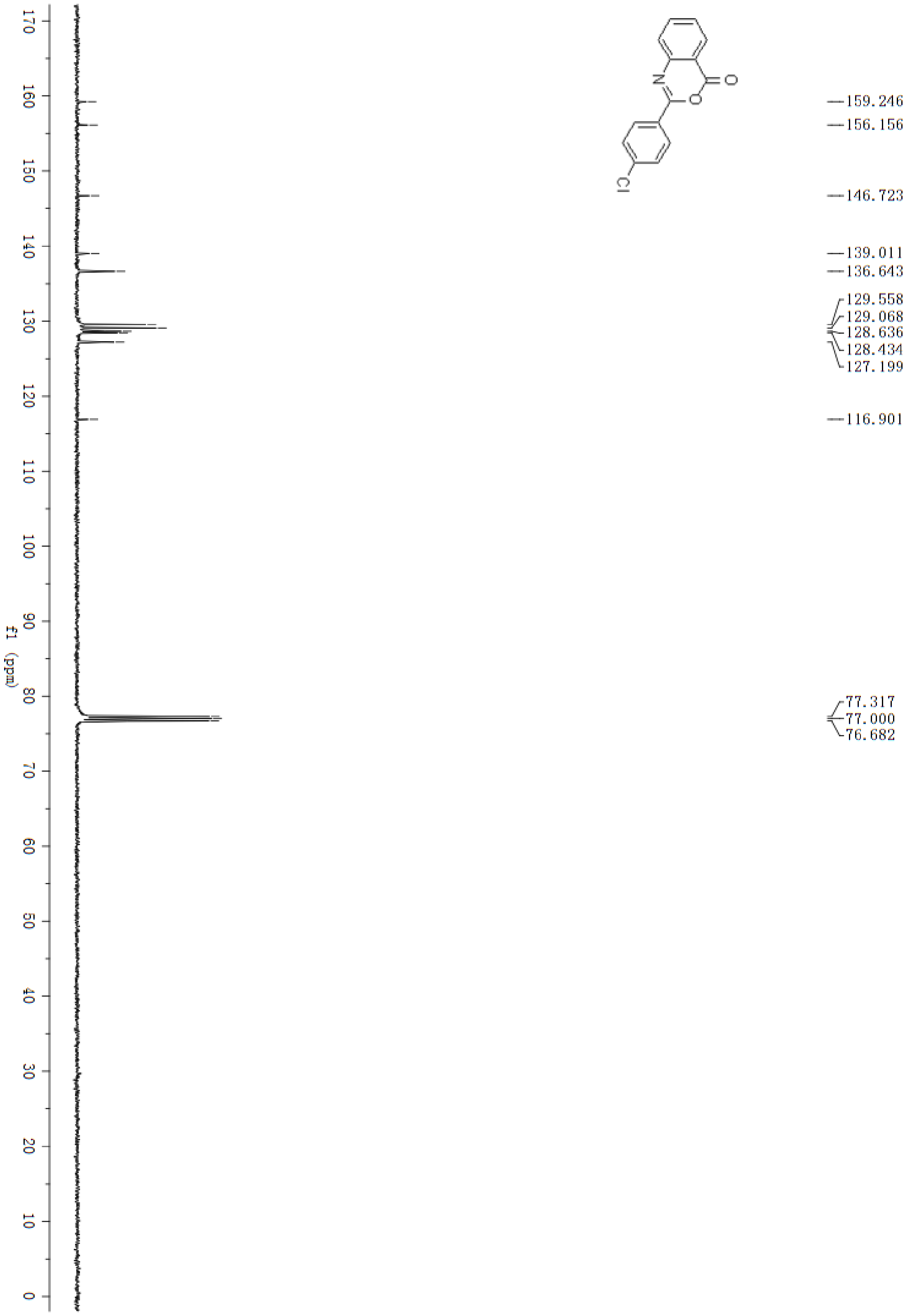


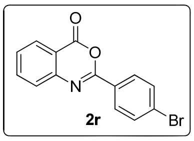

166.743
164.215
159.258
156.077
146.764
136.551
130.670
128.537
128.200
127.061
126.312
116.741
116.019
115.799
130.670
130.579
128.537
128.200
127.061
126.312
116.741
116.019
115.799
77.318
77.000
76.682
f1 (ppm)


O
O
N
Cl
2q

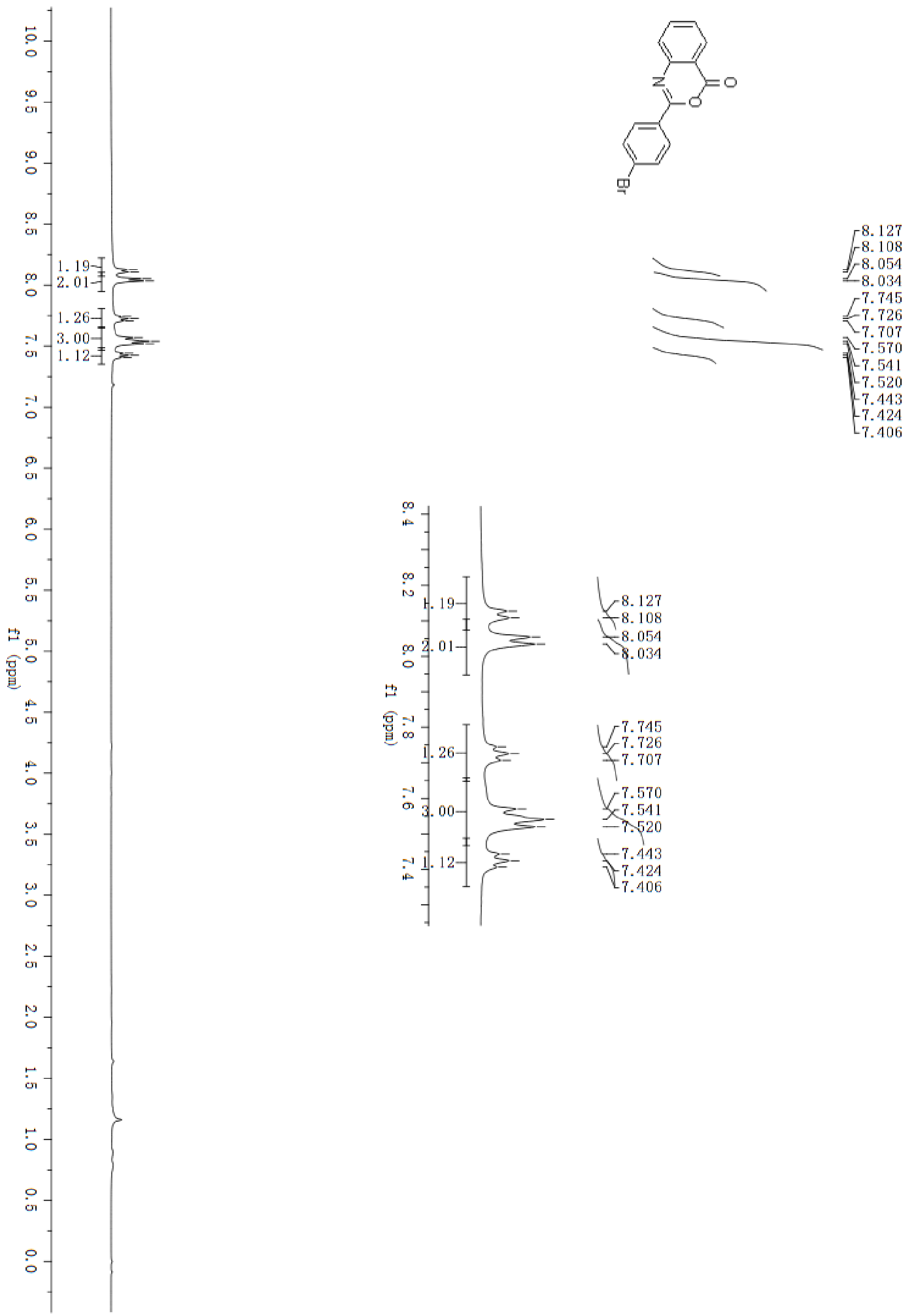

8.254
8.249
8.233
8.227
7.855
7.852
7.835
7.817
7.814
7.687
7.666
7.549
7.530
7.510
7.494
7.472
2.15
0.93
0.90
2.20
f1 (ppm)
Cl
N
O
O

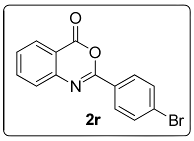

O
O
N
2q
Cl


159.246
156.156
146.723
139.011
136.643
129.558
129.068
128.636
128.434
127.199
116.901
77.317
77.000
76.682
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

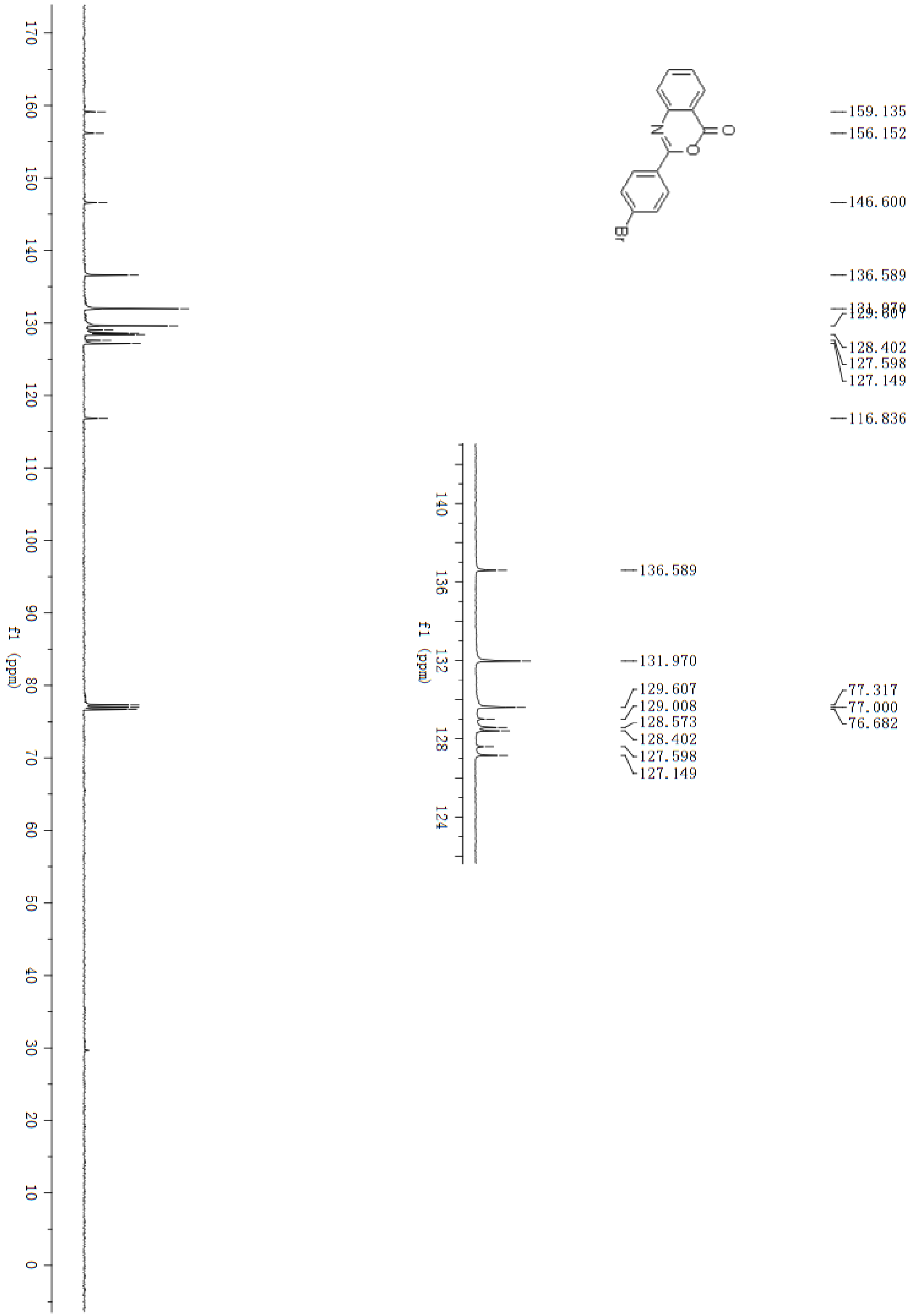


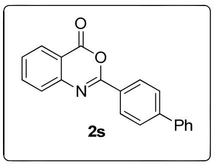
2r

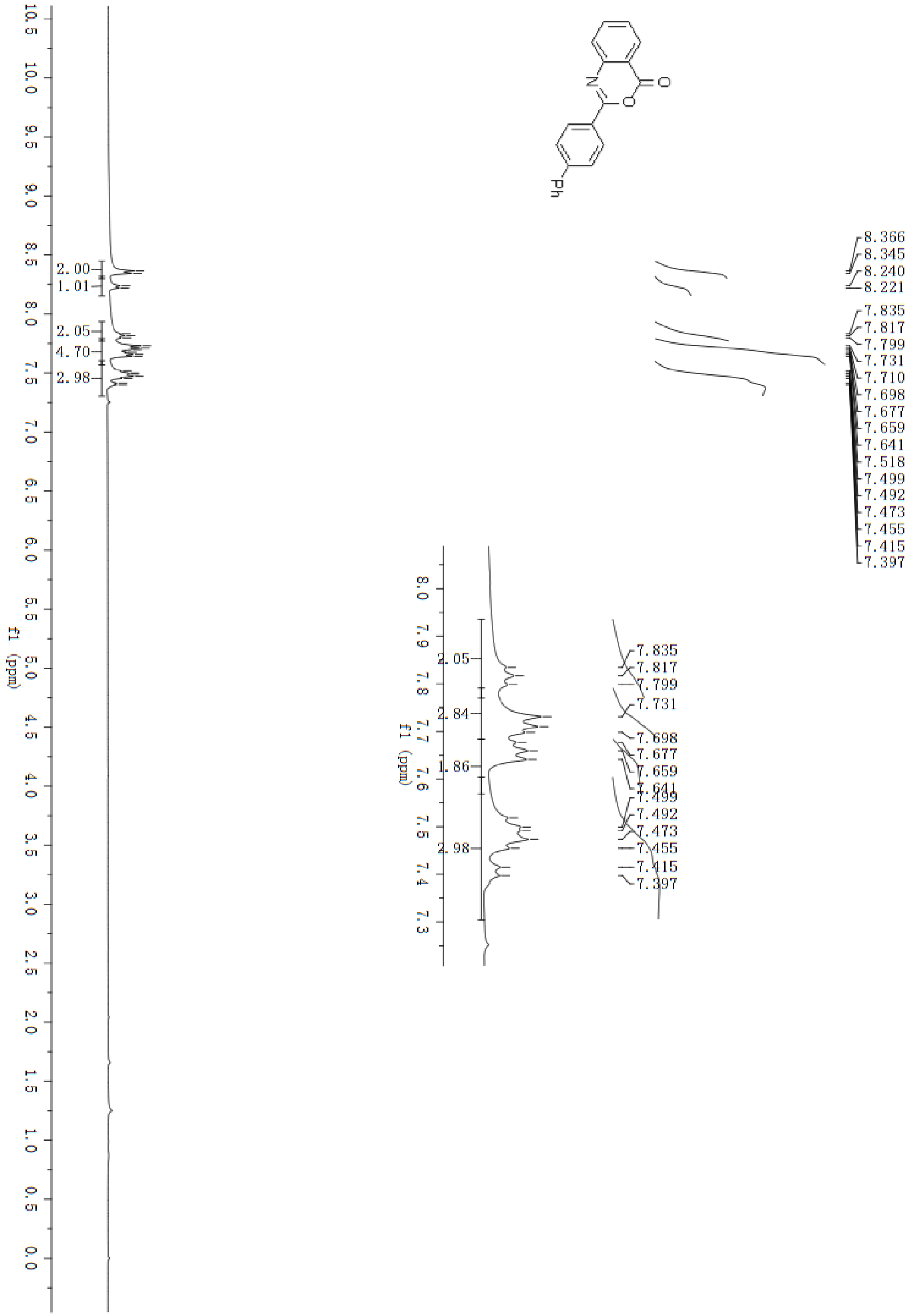

8.127
8.108
8.054
8.034
7.745
7.726
7.707
7.570
7.541
7.520
7.443
7.424
7.406
1.19
2.01
1.26
3.00
1.12
f1 (ppm)

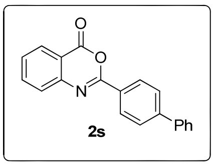


2r

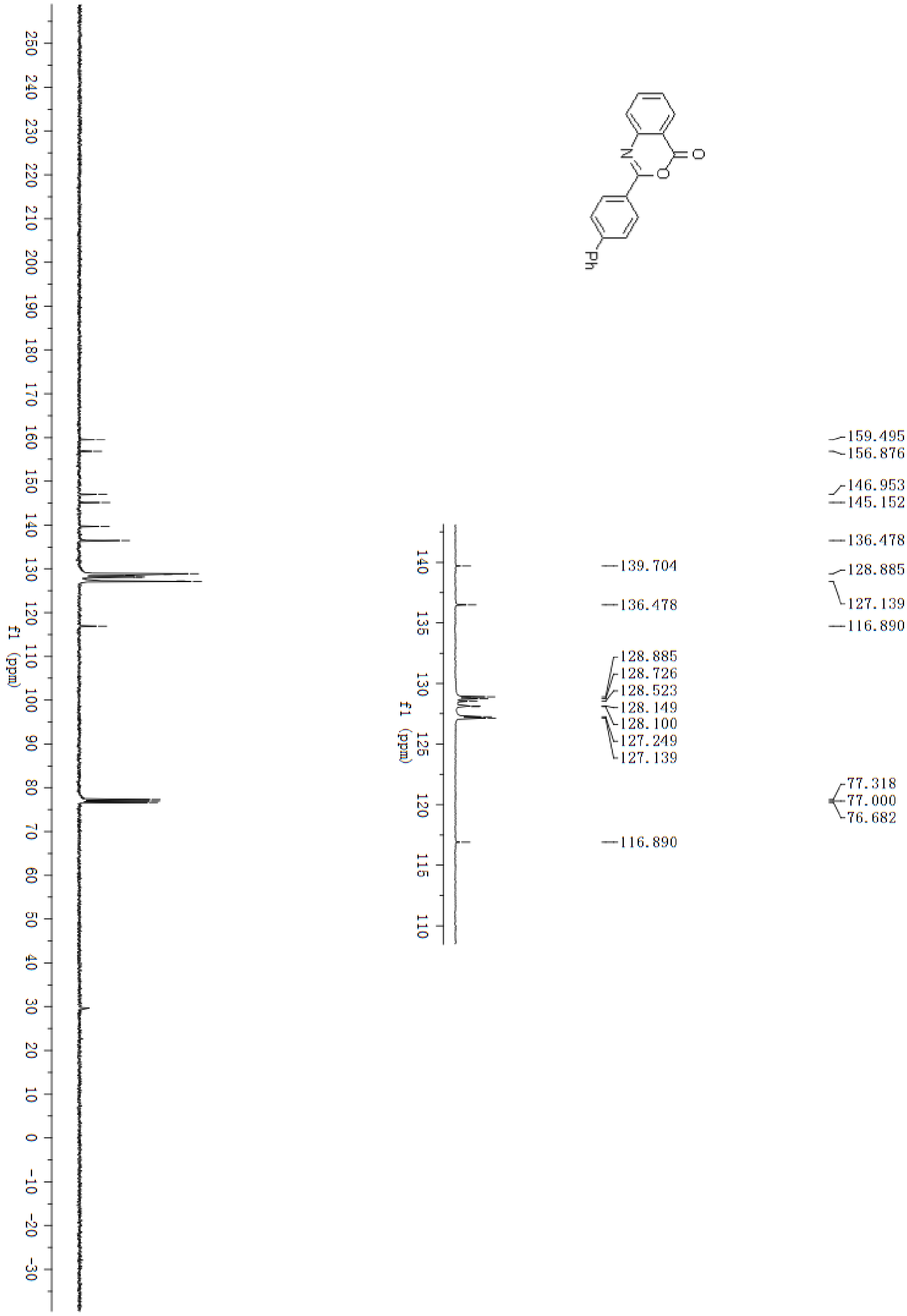

159.135
156.152
146.600
136.589
131.970
129.607
129.008
128.573
128.402
127.598
127.149
116.836
77.317
77.000
76.682
f1 (ppm)

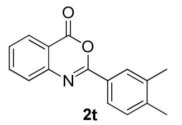

2s
Ph


8.366
8.345
8.240
8.221
7.835
7.817
7.799
7.731
7.710
7.698
7.677
7.659
7.641
7.518
7.499
7.492
7.473
7.455
7.415
7.397
2.00
1.01
2.05
4.70
2.98
2.84
1.86
f1 (ppm)
Ph

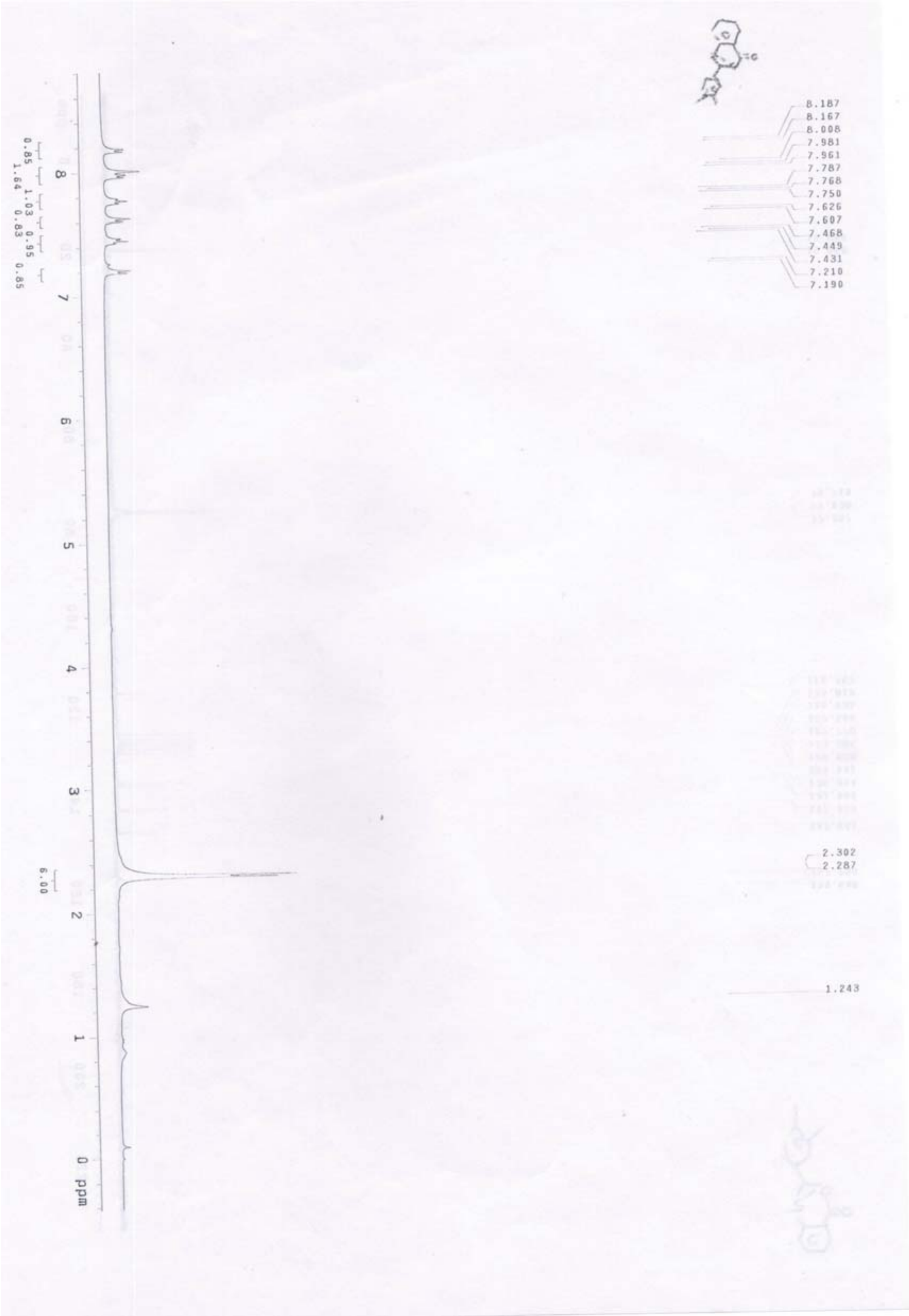

O
O
N
Ph

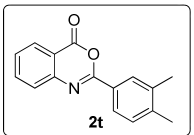
2s


159.495
156.876
146.953
145.152
136.478
128.885
127.139
116.890
77.318
77.000
76.682
139.704
136.478
128.885
128.726
128.523
128.149
128.100
127.249
127.139
116.890
f1 (ppm)
f1 (ppm)

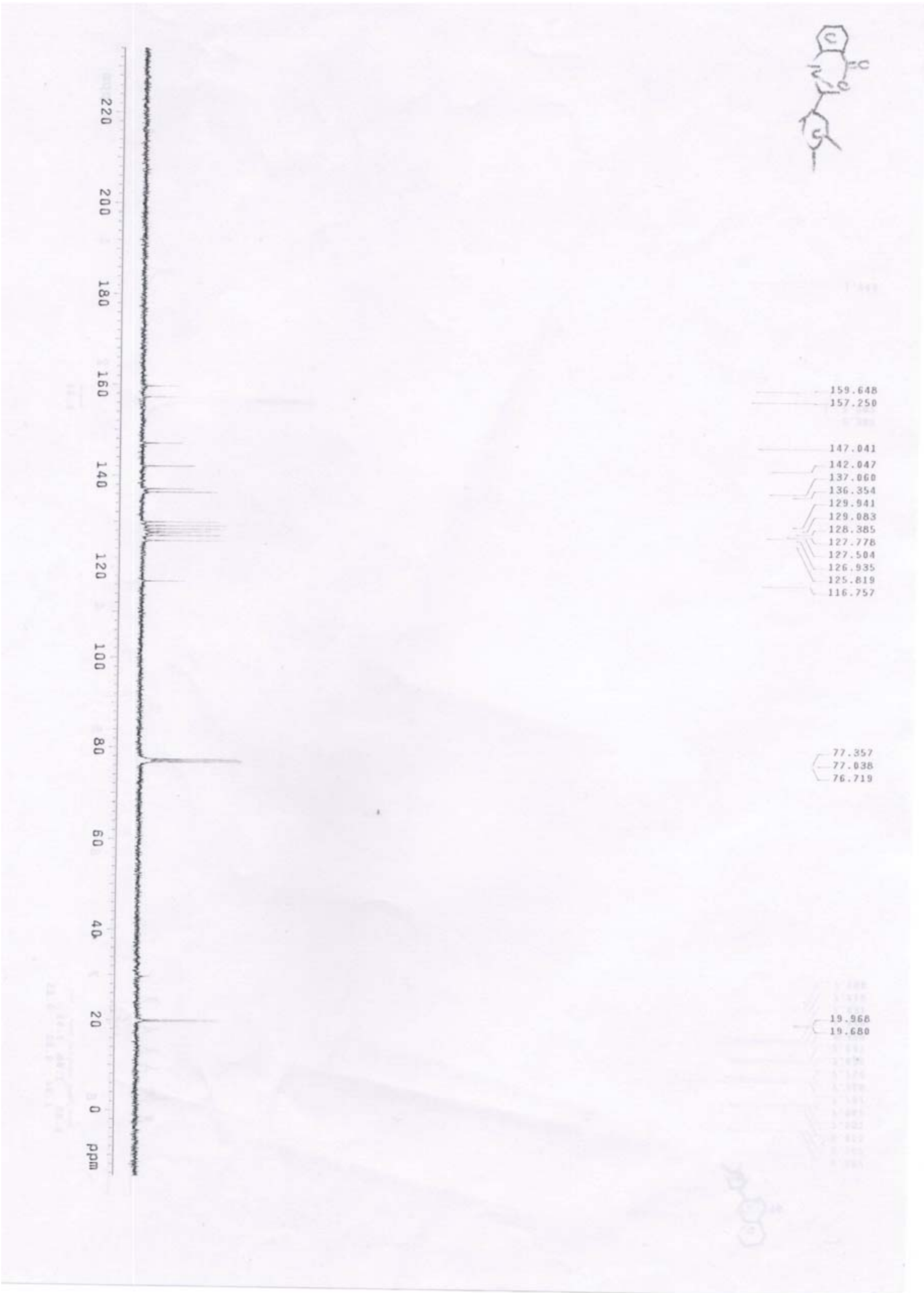


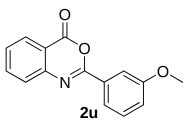
2t


8.187
8.167
8.008
7.981
7.961
7.787
7.768
7.750
7.626
7.607
7.468
7.449
7.431
7.210
7.190
2.302
2.287
1.243
0.85
1.64
1.03
0.83
0.95
0.85
6.00
8
7
6
5
4
3
2
1
0
ppm

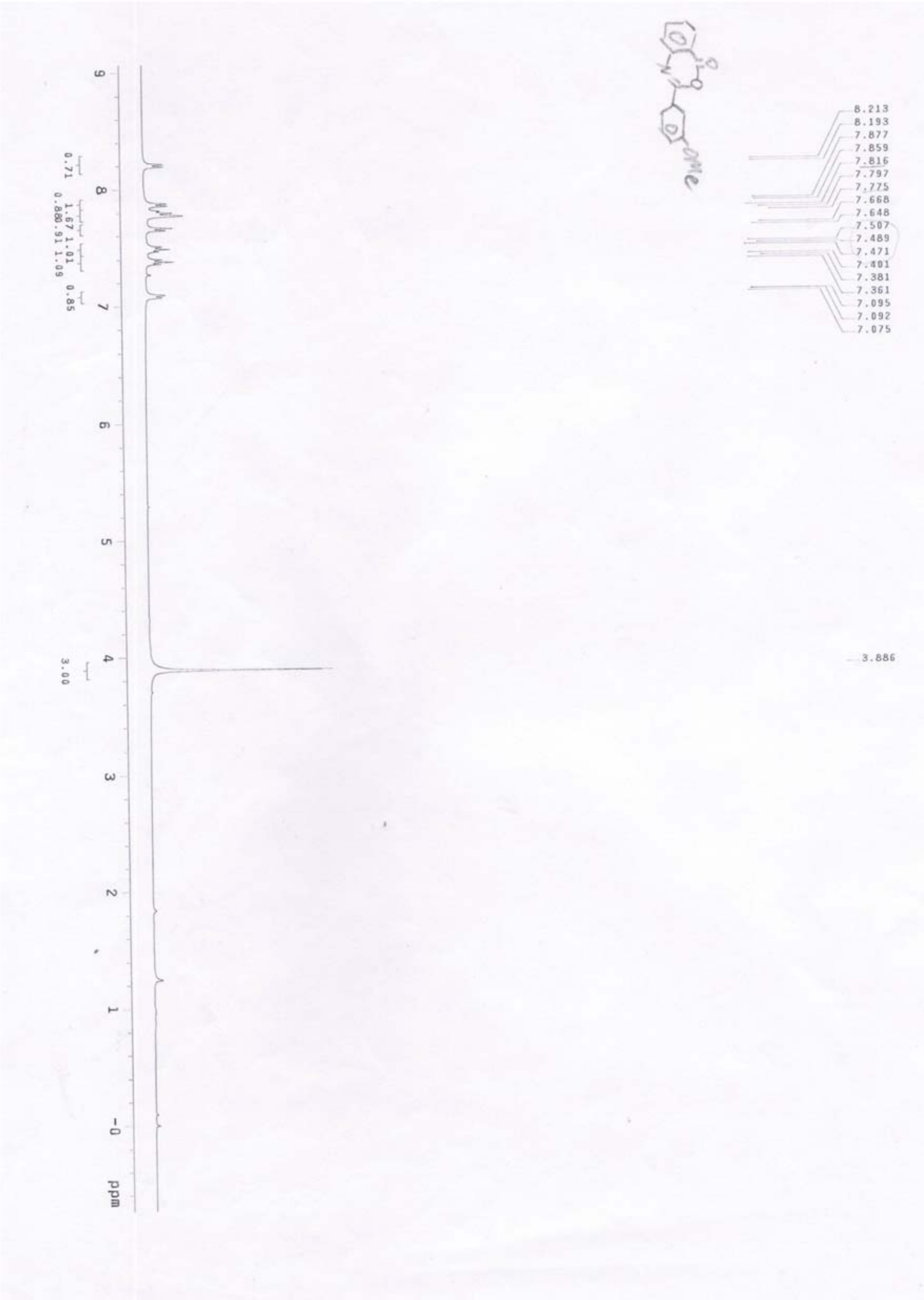


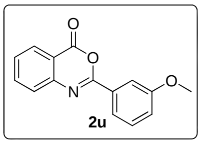
2t


159.648
157.250
147.041
142.047
137.060
136.354
129.941
129.083
128.385
127.778
127.504
126.935
125.819
116.757
77.357
77.038
76.719
19.968
19.680
220
200
180
160
140
120
100
80
60
40
20
0
ppm

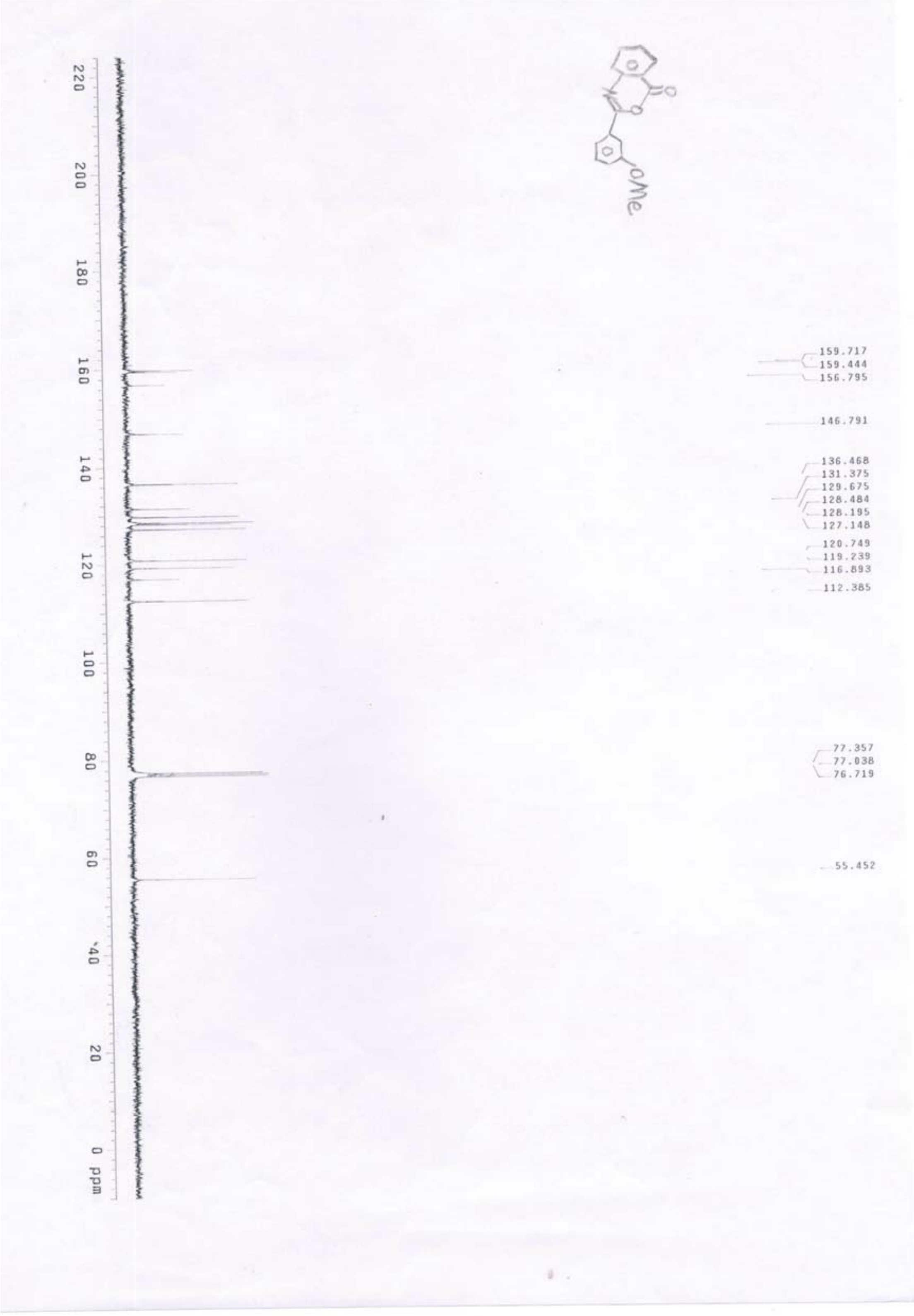


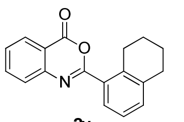
2u


8.213
8.193
7.877
7.859
7.816
7.797
7.775
7.668
7.648
7.507
7.489
7.471
7.401
7.381
7.361
7.095
7.092
7.075
3.886
ppm
0.71
1.67
1.01
0.88
0.91
1.09
0.85
3.00

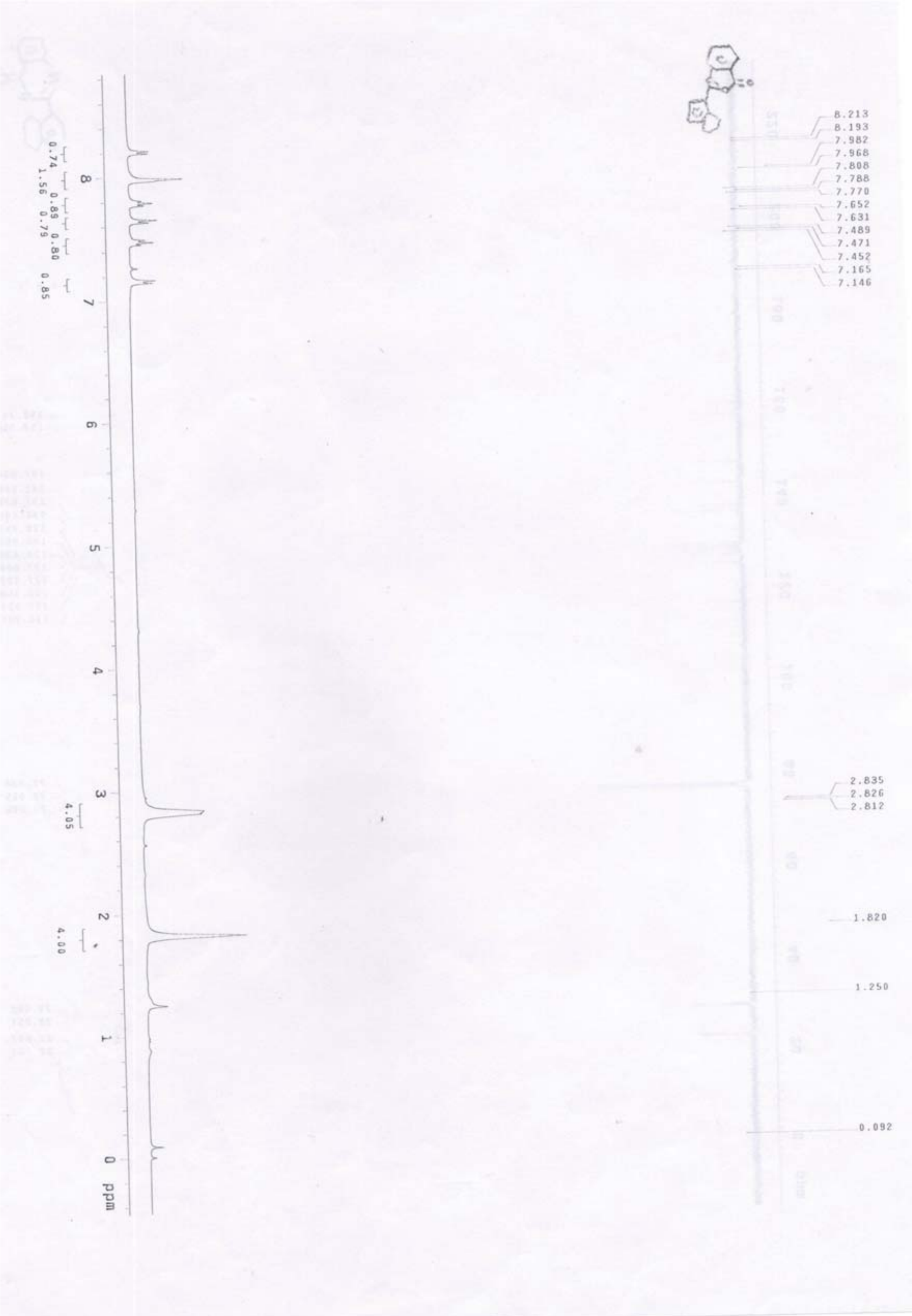


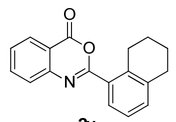
2u



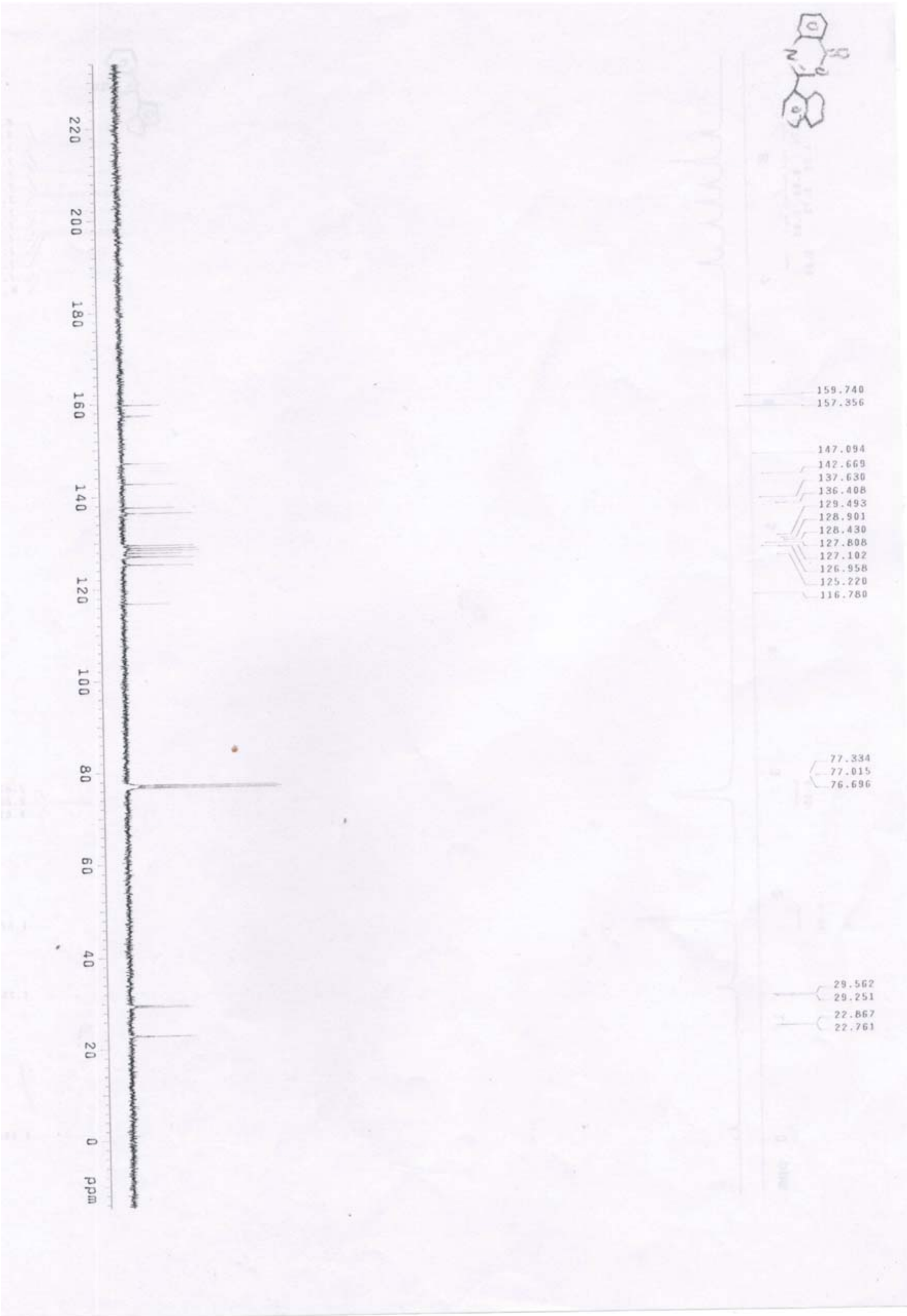


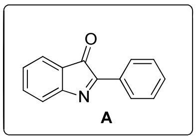
2v

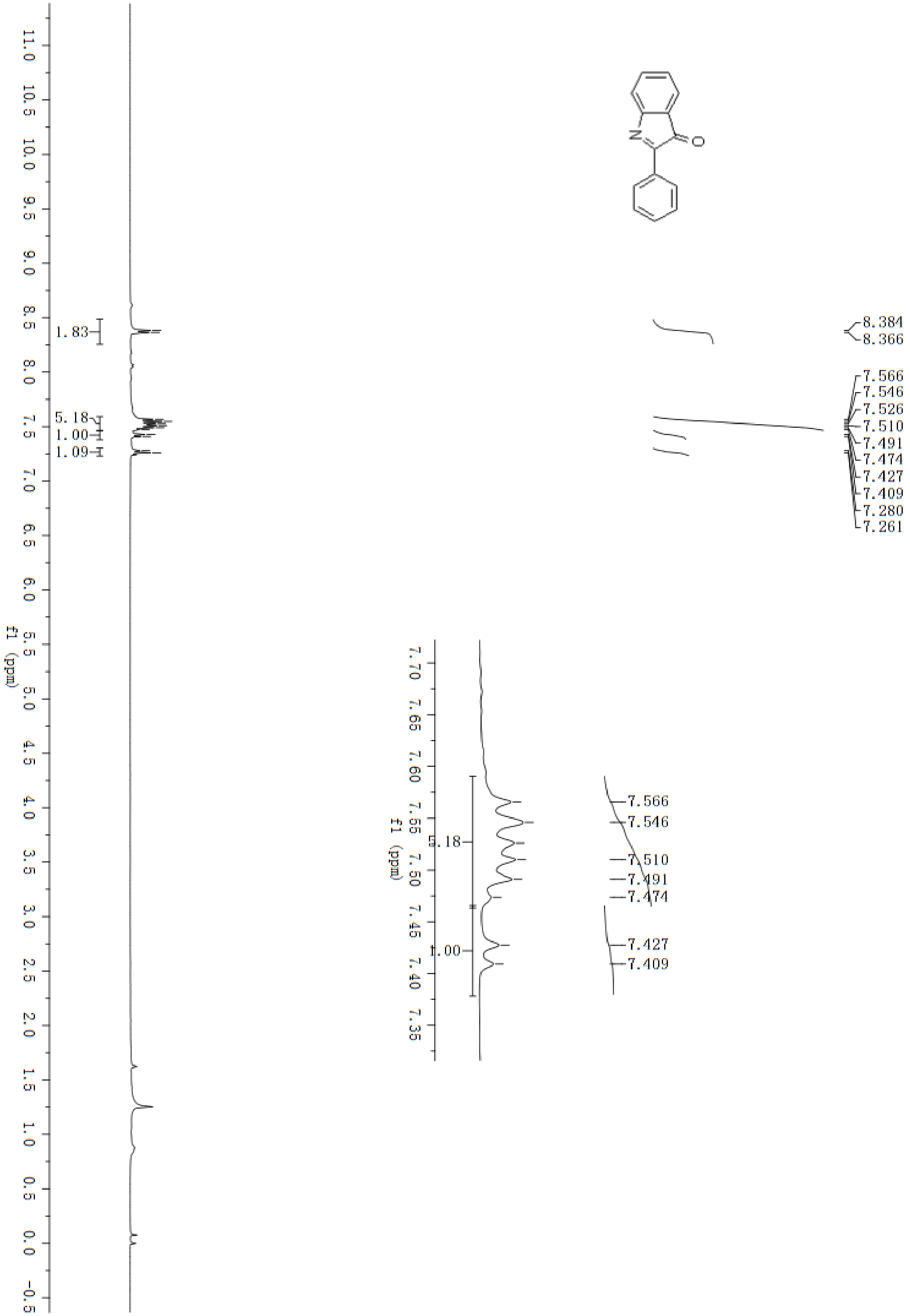

8.213
8.193
7.982
7.968
7.808
7.788
7.770
7.652
7.631
7.489
7.471
7.452
7.165
7.146
2.835
2.826
2.812
1.820
1.250
0.092

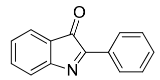


2v

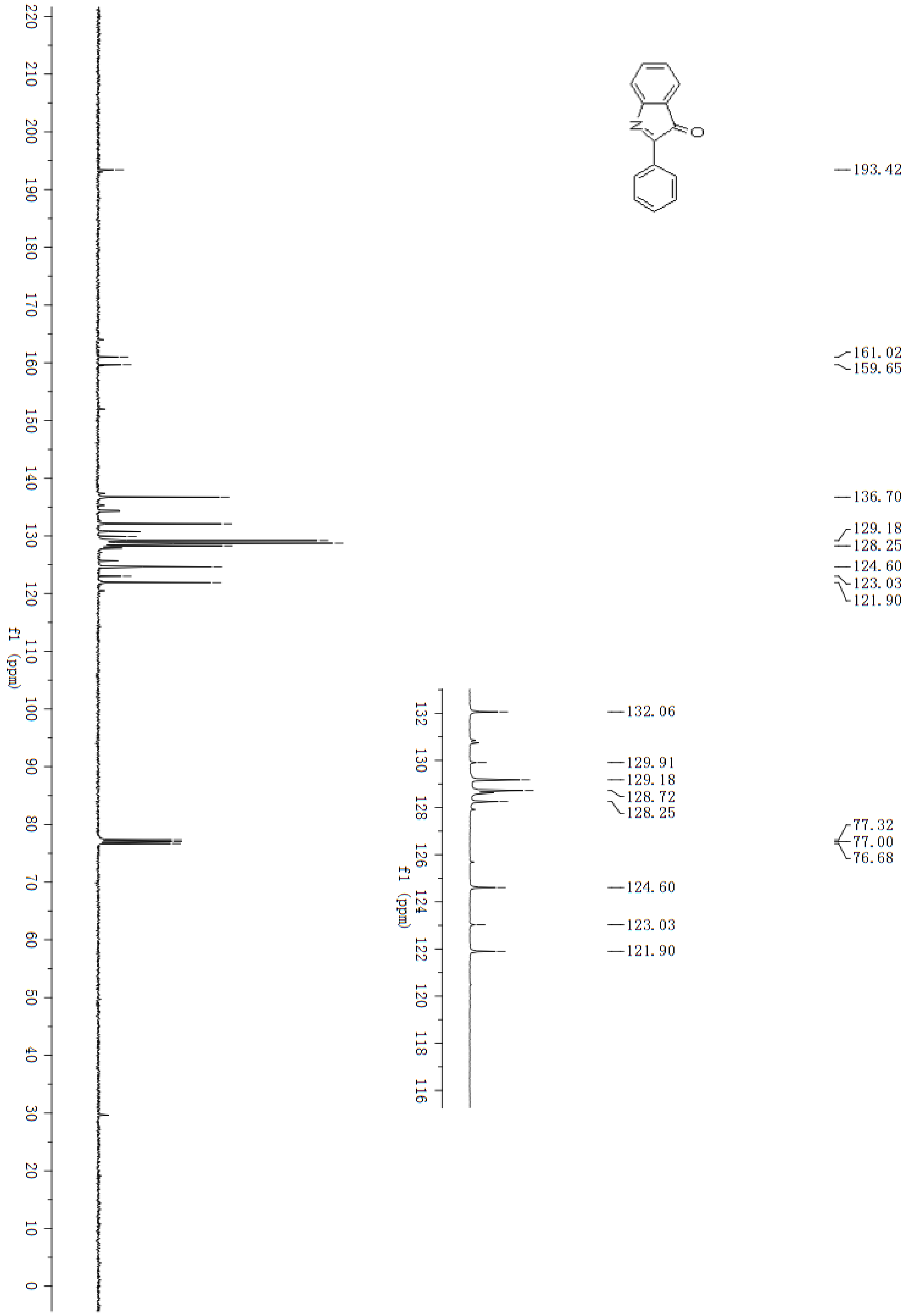

159.740
157.356
147.094
142.669
137.630
136.408
129.493
128.901
128.430
127.808
127.102
126.958
125.220
116.780
77.334
77.015
76.696
29.562
29.251
22.867
22.761
220 200 180 160 140 120 100 80 60 40 20 0 ppm


O
N
A


8.384
8.366
7.566
7.546
7.526
7.510
7.491
7.474
7.427
7.409
7.280
7.261
1.83
5.18
1.00
1.09
f1 (ppm)



A


193.42
161.02
159.65
136.70
129.18
128.25
124.60
123.03
121.90
77.32
77.00
76.68
132.06
129.91
129.18
128.72
128.25
124.60
123.03
121.90
f1 (ppm)
f1 (ppm)

6. X-Ray Structure of 2a