Supporting Information

Metal-free aminofluorination of α-diazo 2H-benzopyran-4-one: Convenient access to β-fluoramides

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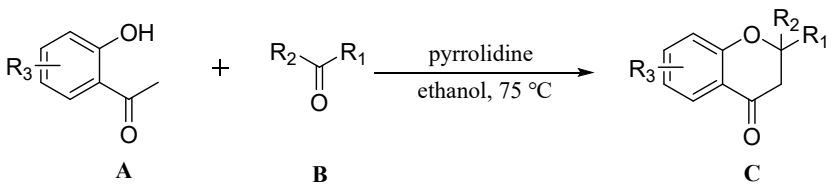
General Information

All solvents were dried according to known methods and distilled prior to use. Diazo compound were synthesized using known literature procedure[1-3]. Other reagents were commercially available and used as purchased. Purification of products was accomplished by chromatography using silica gel (200~300 mesh).

^{1}H and ^{13}C NMR spectra were recorded on a Bruker advance Ⅲ 400 spectrometer in $CDCl_3$ with TMS as internal standard. The chemical shifts (δ) were measured in ppm and all coupling constants are expressed in Hz. X-ray Single-crystal diffraction was measured on a Supemova spectrometer. IR spectra were recorded on a Bruker VERTEX 70V FT-IR spectrometer. HRMS were recorded on Bruker Apex II mass spectrometer with ESI resource.

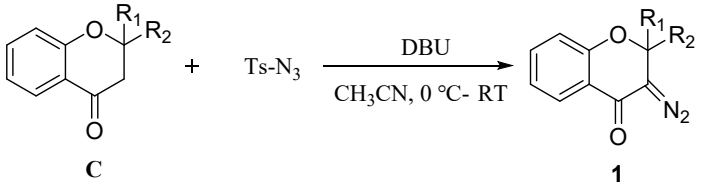
Experimental Procedures

General Procedure for the Preparation of 4-Chromanone



At room temperature, the pyrrolidine (1.5 mmol, 0.15 equiv) was added to the mixture of **A** (10 mmol, 1.0 equiv) and **B** (15 mmol, 1.5 equiv) in 50 ml of ethanol solution. Then the reaction was heated to 75 °C and refluxed for 4 hours. The reaction was monitored by TLC. The reaction mixture was extracted three times with EtOAc and saturated salt water. The combined organic layer was washed with water and saturated aqueous brine and dried over $MgSO_4$. The filtrate was concentrated in vacuo. Further purification by a short silica gel column using petroleum ether (Ⅱ, 60-90 °C) / EtOAc as eluent gave the corresponding product **C**.

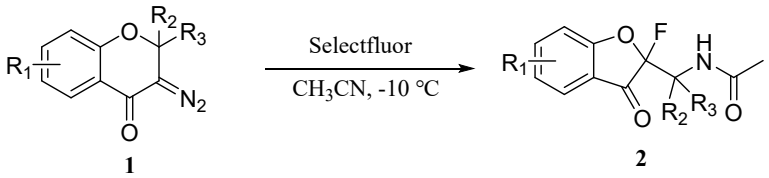
General Procedure for the Preparation of 3-diazochroman-4-one



C (1 mmol, 1.0 equiv) and Ts-N_3 (1.5 mmol, 1.5 equiv) were dissolved in CH_3CN (10 ml) and cooled to 0 °C in an ice/water bath. Then DBU (1.5 mmol, 1.5 equiv) was added dropwise. The reaction was monitored by TLC. The solution was stirred untill full conversion of the substrate ketone and quenched with saturated aqueous NH_4Cl (5 mL) and water (2 mL) and extracted with EtOAc three times (50 mL). The combined organic layer was washed with water (20 mL) and saturated aqueous brine (20 mL) and dried over $MgSO_4$. The filtrate was filtered and concentrated in vacuo. Further purification by a short silica gel column using petroleum ether (Ⅱ, 60-90 °C) /

EtOAc as eluent gave the corresponding product **1**.

General Procedure for the Preparation of 2

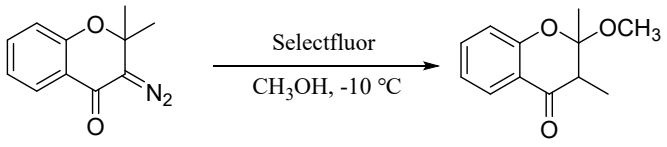


1 **2**

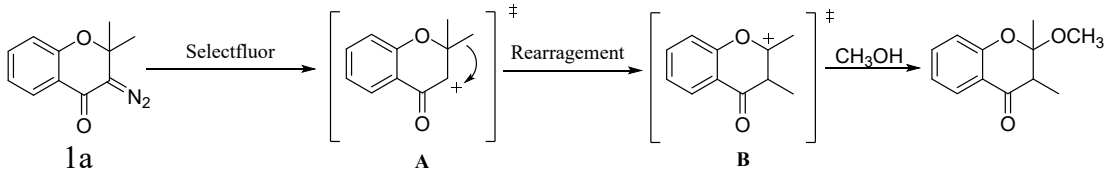
Under an air atmosphere, addition of **1** (0.2 mmol) in of 3ml acetonitrile over 6h (using a syringe pump) to a stirred solution of selectfluor (0.2 mmol) in 2ml acetonitrile at -10℃. Then the reaction mixture stirred until the reaction was completed by TLC analysis. The reaction mixture was extracted three times with EtOAc, and the combined organic phases were dried over $MgSO_4$, filtered and concentrated in vacuo. Further purification by a short silica gel column using petroleum ether /EtOAc (4:1) as eluent gave the corresponding product **2a**-**2p**.

General Procedure for the Preparation of 2-methoxy-2,3-dimethylchroman-4-one

Under optimal reaction conditions, we toke 1a as model and replaced the reaction solvent with methanol. Although the fluoride product was not obtained, we got 2-methoxy-2,3-dimethylchroman-4-one. We speculated that OCH_3 captured rearrangement carbocation (B). As shown in Scheme 1, secondary carbocation (A) is less stable than tertiary carbocation (B).
Under air atmosphere, addition of 1a (0.2 mmol) in of 3ml methanol over 6h (using a syringe pump) to a stirred solution of methanol (0.2 mmol) in 2ml methanol at -10 ℃. Then the reaction mixture stirred until the reaction was completed by TLC analysis. The reaction mixture was extracted three times with EtOAc, and the combined organic phases were dried over $MgSO_4$, filtered and concentrated in vacuo. Further purification by a short silica gel column using petroleum ether /EtOAc (15:1) as eluent gave the corresponding product 2-methoxy-2,3-dimethylchroman-4-one.



1a



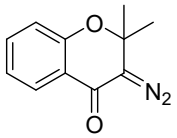
1a **A** **B**

Scheme 1 The proposed mechanism.

Characterization Data for Products.

Characterization Data for Products 1.

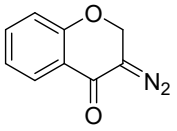
1a[3] 3-Diazo-2, 2-dimethyl-chroman-4-one



^{1}H NMR (400MHz, $CDCl_3$): δ 7.88 (dd, J = 7.8, 1.7 Hz, 1H), 7.41 (ddd, J = 8.3, 7.3, 1.8 Hz, 1H), 7.07 – 7.00 (m, 1H), 6.91 (dd, J = 8.3, 0.8 Hz, 1H), 1.68 (s, 6H);

^{13}C NMR (100MHz, $CDCl_3$): δ 178.4, 157.1, 135.0, 125.9, 121.8, 121.1, 118.2, 75.0, 27.2.

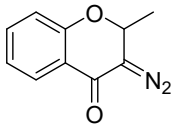
1b[3] 3-diazochroman-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.48 – 7.39 (m, 1H), 7.13 – 7.05 (m, 1H), 6.97 (dd, *J* = 8.3, 1.2 Hz, 1H), 5.20 (s, 2H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 178.63, 159.44, 134.89, 126.39, 122.47, 117.74, 62.30.

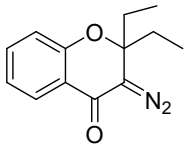
1c[3] 3-diazo-2-methylchroman-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.44 (ddd, *J* = 8.2, 7.2, 1.8 Hz, 1H), 7.08 (ddd, *J* = 7.5, 7.4, 1.0 Hz, 1H), 6.95 (dd, *J* = 8.3, 1.2 Hz, 1H), 5.41 (q, *J* = 6.3, 1.0 Hz, 1H), 1.71 (d, *J* = 6.3, 1.0 Hz, 3H)

^{13}C NMR (101 MHz, $CDCl_3$): δ 179.05, 158.74, 134.92, 126.28, 122.28, 117.77, 68.83, 18.44.

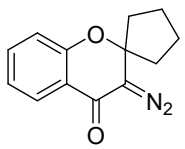
1d[2] 3-diazo-2,2-diethylchroman-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.42 (ddd, *J* = 8.3, 7.3, 1.8 Hz, 1H), 7.03 (ddd, *J* = 7.6, 7.5, 1.1 Hz, 1H), 6.92 (dd, *J* = 8.3, 1.1 Hz, 1H), 2.10 – 1.80 (m, 5H), 1.03 (t, *J* = 7.4 Hz, 7H)

^{13}C NMR (101 MHz, $CDCl_3$): δ 178.07, 156.60, 134.05, 124.82, 120.52, 79.73, 30.05, 7.06.

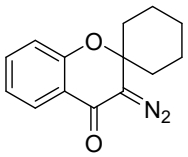
1e[2] 3-diazospiro[chromane-2,1'-cyclopentan]-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.42 (ddd, *J* = 8.7, 7.3, 1.8 Hz, 1H), 7.06 (ddd, *J* = 7.6, 7.5, 1.1 Hz, 1H), 6.91 (dd, *J* = 8.3, 1.0 Hz, 1H), 2.39 – 2.31 (m, 2H), 2.03 – 1.94 (m, 2H), 1.88 – 1.78 (m, 4H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 178.18, 156.32, 133.84, 125.07, 120.92, 117.37, 83.89, 37.06, 22.13.

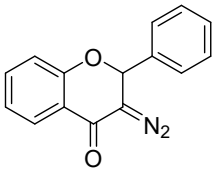
1f[3] 3-diazospiro[chromane-2,1'-cyclohexan]-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.44 (ddd, *J* = 8.7, 7.3, 1.7 Hz, 1H), 7.06 (ddd, *J* = 7.6, 7.5, 1.1 Hz, 1H), 6.98 (dd, *J* = 8.2, 1.1 Hz, 1H), 2.32 – 2.24 (m, 2H), 1.90 – 1.72 (m, 4H), 1.68 – 1.54 (m, 4H), 1.38 – 1.23 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 177.98, 155.75, 133.91, 125.00, 120.85, 120.72, 117.28, 74.63, 67.84, 34.17, 23.70, 20.31.

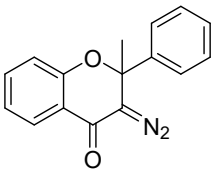
1g[2] 3-diazo-2-phenylchroman-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.44 (ddd, *J* = 8.7, 7.3, 1.7 Hz, 1H), 7.06 (ddd, *J* = 7.5, 7.5, 1.1 Hz, 1H), 6.98 (dd, *J* = 8.2, 1.1 Hz, 1H), 2.32 – 2.24 (m, 2H), 1.90 – 1.72 (m, 4H), 1.68 – 1.54 (m, 4H), 1.38 – 1.23 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 177.53, 157.52, 150.47, 134.16, 134.06, 129.13, 128.23, 126.44, 125.26, 121.51, 121.07, 116.95, 74.50.

1h 3-diazo-2-methyl-2-phenylchroman-4-one

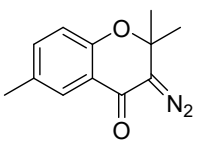


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.42 – 7.38 (m, 2H), 7.32 (ddd, *J* = 8.2, 7.2, 1.7 Hz, 1H), 7.28 – 7.23 (m, 2H), 7.22 – 7.18 (m, 1H), 6.94 – 6.89 (m, 2H), 1.93 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 177.61, 156.35, 141.86, 134.07, 127.70, 127.63, 125.11, 124.56, 121.16, 120.73, 117.39, 78.05, 67.28, 27.17.

HRMS (ESI): m/z=287.0791 calcd for $C_{16}H_{12}N_2O_2$+Na $[M+ Na]^+$, found: 287.0793.

1i 3-diazo-2,2,6-trimethylchroman-4-one

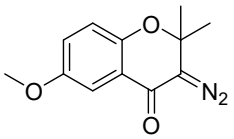


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 7.9 Hz, 1H), 7.24 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.82 (d, *J* = 8.3 Hz, 1H), 2.32 (s, 3H), 1.68 (s, 6H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 177.76, 154.05, 134.94, 130.42, 124.76, 119.75, 117.07, 73.83, 26.16, 19.44.

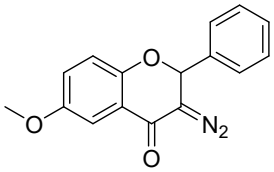
HRMS (ESI): m/z=239.0791 calcd for $C_{12}H_{12}N_2O_2$+Na $[M+ Na]^+$, found: 239.0793.

1j[2] 3-diazo-6-methoxy-2,2-dimethylchroman-4-one



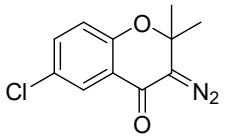
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (dd, *J* = 3.2, 1.2 Hz, 1H), 7.02 (ddd, *J* = 9.0, 3.2, 1.2 Hz, 1H), 6.86 (dd, *J* = 8.9, 1.1 Hz, 1H), 3.82 (d, *J* = 1.3 Hz, 3H), 1.68 (s, 6H);
^{13}C NMR (101 MHz, $CDCl_3$): δ 177.53, 153.53, 150.32, 122.32, 120.19, 118.49, 106.43, 73.80, 54.82, 26.07.

1k 3-diazo-6-methoxy-2-phenylchroman-4-one



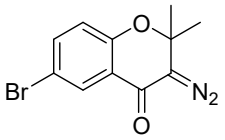
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.8 Hz, 1H), 7.56 (m, 2H), 7.48 (m, 3H), 6.68 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.48 (d, *J* = 2.4 Hz, 1H), 6.26 (s, 1H), 3.83 (s, 3H);
^{13}C NMR (101 MHz, $CDCl_3$): δ 176.83, 164.30, 159.36, 134.18, 129.11, 128.21, 126.91, 126.44, 114.56, 109.23, 100.65, 74.88, 54.62.
HRMS (ESI): m/z=303.0740 calcd for $C_{16}H_{12}N_2O_3$+Na $[M+ Na]^+$, found: 303.0742.

1l[2] 6-chloro-3-diazo-2,2-dimethylchroman-4-one



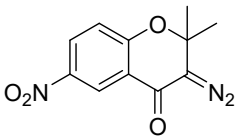
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 2.7 Hz, 1H), 7.38 (dd, *J* = 8.8, 2.7 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 1H), 1.70 (s, 6H);
^{13}C NMR (101 MHz, $CDCl_3$): δ 176.29, 154.52, 133.74, 126.47, 124.50, 120.98, 118.91, 74.49, 26.21.

1m[2] 6-bromo-3-diazo-2,2-dimethylchroman-4-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (d, *J* = 2.6 Hz, 1H), 7.94 (dd, *J* = 8.8, 2.5 Hz, 1H), 7.26 (d, *J* = 8.7 Hz, 1H), 2.12 (s, 6H).;
^{13}C NMR (101 MHz, $CDCl_3$): δ 176.15, 155.02, 136.58, 127.52, 121.41, 119.28, 113.61, 74.51, 26.22.

1n[2] 3-diazo-2,2-dimethyl-6-nitrochroman-4-one

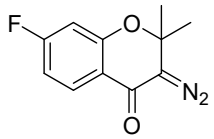


^{1}H NMR (400 MHz, $CDCl_3$): δ 8.81 (d, *J* = 2.8 Hz, 1H), 8.31 (dd, *J* = 9.1, 2.9 Hz, 1H), 7.07 (d, *J*

= 9.0 Hz, 1H), 1.76 (s, 6H).;

^{13}C NMR (101 MHz, $CDCl_3$): δ 175.08, 160.39, 128.61, 121.55, 119.69, 118.39, 26.53.

1o 3-diazo-7-fluoro-2,2-dimethylchroman-4-one

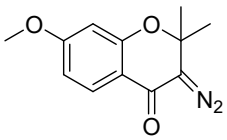


^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, *J* = 8.7, 6.7 Hz, 1H), 6.67 (dd, *J* = 8.5, 2.4 Hz, 1H), 6.60 (m, 1H), 1.45 (s, 6H);

^{13}C NMR (101 MHz, $CDCl_3$): δ 190.01, 167.99, 165.45, 160.63, 128.06, 116.11 (d, *J* = 2.5 Hz), 108.14, 103.86, 79.11, 25.56.

HRMS (ESI): m/z=243.0540 calcd for $C_{12}H_{12}N_2F_1O_2$+Na $[M+ Na]^+$, found: 243.0542.

1p 3-diazo-7-methoxy-2,2-dimethylchroman-4-one



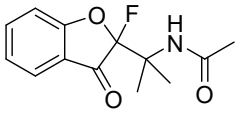
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, *J* = 8.7, 1.3 Hz, 1H), 6.62 (ddd, *J* = 8.7, 2.5, 1.3 Hz, 1H), 6.40 (dd, *J* = 2.2, 1.1 Hz, 1H), 3.83 (d, *J* = 1.1 Hz, 3H), 1.69 (d, *J* = 1.0 Hz, 6H).;

^{13}C NMR (101 MHz, $CDCl_3$): δ 176.75, 164.36, 158.01, 126.65, 113.71, 108.56, 101.00, 74.47, 54.56, 26.21.

HRMS (ESI): m/z=255.0740 calcd for $C_{12}H_{12}N_2O_3$+Na $[M+ Na]^+$, found: 255.0743.

Characterization Data for Products 2.

2a A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 48%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (ddd, *J* = 7.2, 4.2, 2.7 Hz, 2H), 7.18 (m, 2H), 6.43 (s, 1H), 1.96 (s, 3H), 1.69 (s, 3H), 1.45 (s, 3H).

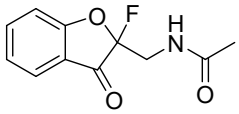
^{19}F NMR (376 MHz, $CDCl_3$): δ -130.58 (s，1F).

^{13}C NMR (101 MHz, $CDCl_3$):δ 193.7 (d, *J* = 20.2 Hz), 170.3 , 169.8 , 139.6 , 125.4 , 123.9 , 119.1 , 113.1 , 110.0(d, *J* = 245.4 Hz), 57.9 (d, *J* = 25.7 Hz), 24.5 , 20.7 (d, *J* = 3.8 Hz), 19.9 (d, *J* = 3.3 Hz).

HRMS (ESI): m/z=274.0857 calcd for $C_{13}H_{14}F_1N_1O_3$+Na [M+ Na]$^+$, found: 274.0850;

IR (cm^{-1}):3295, 1737, 1666, 1614, 1080.

2b A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 25%).



^{1}H NMR (400 MHz, CDCl3): δ 7.70 (ddd, *J* = 7.3, 4.1, 1.4 Hz, 2H), 7.22 – 7.13 (m, 2H), 5.96 (s, 1H), 4.04 (ddd, *J* = 16.3, 15.0, 6.8 Hz, 1H), 3.84 (td, *J* = 14.8, 5.5 Hz, 1H), 2.02 (s, 3H).

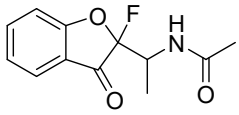
^{19}F NMR (376 MHz, CDCl3): δ -123.01 (dd, *J* = 24.9, 10.1 Hz，1F)

^{13}C NMR (101 MHz, CDCl3):δ 192.6 (d, *J* = 18.9 Hz), 170.4 , 170.3 , 139.6, 125.6, 124.0, 118.3, 113.5, 108.3 (d, *J* = 238.9 Hz), 40.2 (d, *J* = 31.7 Hz), 23.1.

HRMS (ESI)： m/z=246.0537 calcd for $C_{11}H_{10}F_1N_1O_3$+Na [M+ Na]$^+$, found: 246.0541;

IR (cm^{-1}):3290, 1736, 1656, 1616, 1076.

2c A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 35%, dr >20:1).



^{1}H NMR (400 MHz, CDCl3): δ 8.66 – 8.63 (m, 2H), 7.70 (ddd, *J* = 7.6, 5.9, 1.4 Hz, 2H), 7.23 – 7.00 (m, 1H), 5.82 (d, *J* = 9.0 Hz, 1H), 4.74 (ddd, *J* = 13.2, 9.5, 6.9 Hz, 1H), 2.05 (s, 3H), 1.24 (d, *J* = 6.9 Hz, 3H).

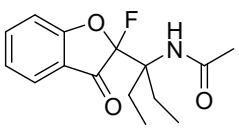
^{19}F NMR (376 MHz, CDCl3): δ -128.19 (d, *J* = 13.1 Hz，1F).

^{13}C NMR (101 MHz, CDCl3): δ 192.3 (d, *J* = 19.2 Hz), 170.2, 169.9, 139.4, 125.6, 123.9, 118.8, 113.2, 109.6 (d, *J* = 242.6 Hz), 46.2 (d, *J* = 27.2 Hz), 23.3, 14.74 (d, *J* = 5.0 Hz).

HRMS (ESI): m/z=260.0693 calcd for $C_{12}H_{12}F_1N_1O_3$+Na [M+ Na]$^+$, found: 260.0699;

IR (cm^{-1}):3275, 1738, 1658, 1615, 1051.

2d A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 25%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 – 7.66 (m, 2H), 7.22 – 7.09 (m, 2H), 6.14 (s, 1H), 2.39 – 2.29 (m, 1H), 2.21 (dd, *J* = 14.6, 7.4 Hz, 1H), 2.15 – 2.07 (m, 1H), 1.96 (s, 3H), 1.93 – 1.84 (m, 1H), 1.00 (dd, *J* = 7.5, 6.3 Hz, 3H), 0.81 (t, *J* = 7.5 Hz, 3H).

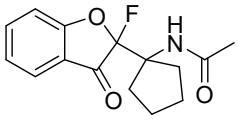
^{19}F NMR (376 MHz, $CDCl_3$): δ -123.30 (s，1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 193.6 (d, *J* = 21.0 Hz), 169.7, 169.6, 139.2, 125.5, 123.8, 119.0, 113.1, 111.1 (d, *J* = 245.2 Hz), 63.6 (d, *J* = 24.4 Hz), 25.5 (d, *J* = 3.8 Hz), 25.1, 24.3, 8.8 (d, *J* = 3.9 Hz), 8.8.

HRMS (ESI): m/z=302.1163 calcd for $C_{15}H_{18}F_1N_1O_3$+Na $[M+ Na]^+$, found: 302.1166;

IR (cm^{-1}):3309, 1741, 1663, 1616, 1080.

2e A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 20%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 – 7.58 (m, 2H), 7.21 – 7.06 (m, 2H), 5.98 (s, 1H), 2.45 – 2.35 (m, 1H), 2.33 – 2.24 (m, 1H), 2.15 – 2.03 (m, 1H), 1.98 – 1.89 (m, 1H), 1.88 (s, 3H), 1.84 (d, J = 4.7 Hz, 1H), 1.82 – 1.71 (m, 2H), 1.71 – 1.63 (m, 1H).

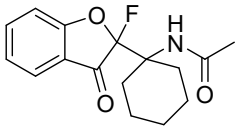
^{19}F NMR (376 MHz, $CDCl_3$): δ -125.43 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): 193.7 (d, *J* = 20.5 Hz), 170.12, 169.7, 139.3, 125.5, 123.7, 119.5, 113.1, 110.5 (d, *J* = 244.8 Hz), 67.2 (d, *J* = 27.1 Hz), 33.9 (d, *J* = 3.3 Hz), 32.5, 25.4, 25.2, 24.4.

HRMS (ESI): m/z=300.1006 calcd for $C_{15}H_{16}F_1N_1O_3$+Na $[M+ Na]^+$, found: 300.1011;

IR (cm^{-1}):3300, 1742, 1665, 1615, 1024.

2f A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 25%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 – 7.54 (m, 2H), 7.16 – 7.04 (m, 2H), 5.13 (s, 1H), 2.82 – 2.69 (m, 1H), 2.17 (d, *J* = 13.3 Hz, 1H), 1.86 (s, 3H), 1.76 (td, *J* = 13.5, 3.6 Hz, 1H), 1.65 (d, *J* = 11.2 Hz, 3H), 1.59 – 1.46 (m, 1H), 1.40 (ddd, *J* = 13.3, 9.8, 3.1 Hz, 2H), 1.22 (ddd, *J* = 12.8, 7.0, 3.5 Hz, 1H).

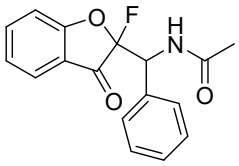
^{19}F NMR (376 MHz, $CDCl_3$): δ -129.33 (s，1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 193.2 (d, J = 20.1 Hz), 170.0, 167.0, 138.8, 125.2, 123.3, 119.7, 113.0, 111.2 (d, J = 246.1 Hz), 60.1 (d, J = 27.4 Hz), 28.7 (d, J = 3.6 Hz), 27.4, 25.0, 24.5, 20.9 (d, J = 6.3 Hz).

HRMS (ESI): m/z=292.1343 calcd for $C_{16}H_{18}F_1N_1O_3$+H $[M+H]^+$, found: 292.1338;

IR (cm^{-1}):3296, 1738, 1669, 1615, 1028.

2g A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 52%, dr >20:1).



^{1}H NMR (400 MHz, CDCl3): δ 7.59 (t, *J* = 7.3 Hz, 2H), 7.35 (d, *J* = 7.1 Hz, 2H), 7.22 (m, 3H), 7.13 – 7.03 (m, 2H), 6.90 (d, *J* = 8.9 Hz, 1H), 5.87 (dd, *J* = 9.0, 6.9 Hz, 1H), 2.03 (s, 3H).

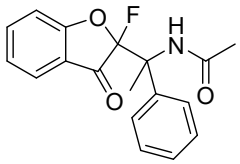
^{19}F NMR (376 MHz, CDCl3): δ -120.15 (d, *J* = 6.8 Hz,1F).

^{13}C NMR (101 MHz, CDCl3): δ 194.0 (d, *J* = 19.7 Hz), 171.0, 169.3, 139.6, 134.1, 134.0, 128.5, 128.4, 128.1, 125.3, 123.8, 118.7, 113.2, 108.1 (d, *J* = 245.5 Hz), 54.5 (d, *J* = 30.4 Hz), 23.3.

HRMS (ESI): m/z=322.0850 calcd for $C_{17}H_{14}F_1N_1O_3$+Na [M+ Na]$^+$, found: 322.0857;

IR (cm^{-1}):3284, 1739, 1663, 1616, 1031.

2h A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 55%, dr >20:1)



^{1}H NMR (400 MHz, CDCl3): δ 7.67 – 7.60 (m, 2H), 7.47 (d, J = 8.0 Hz, 2H), 7.30 (t, J = 7.4 Hz, 2H), 7.23 (dd, J = 6.0, 3.5 Hz, 1H), 7.19 – 7.13 (m, 2H), 7.12 – 7.07 (m, 1H), 2.11 (s, 3H), 1.97 (d, J = 1.4 Hz, 3H).

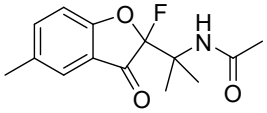
^{19}F NMR (376 MHz, CDCl3): δ -129.37 (s, 1F).

^{13}C NMR (101 MHz, CDCl3): δ 193.4 (d, *J* = 20.3 Hz), 170.3, 169.0, 139.7, 137.0, 127.9, 127.8, 127.4, 125.4, 123.9, 118.9, 113.0, 109.0 (d, *J* = 248.5 Hz), 62.0(d, *J* = 23.2 Hz), 24.2, 19.0.

HRMS (ESI): m/z=336.1006 calcd for $C_{18}H_{16}F_1N_1O_3$+Na [M+ Na]$^+$, found: 336.1013;

IR (cm^{-1}):3402, 1732, 1685, 1615, 1081.

2i A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 53%).



^{1}H NMR (400 MHz, CDCl3): δ 7.50 (dd, J = 12.9, 4.5 Hz, 2H), 7.06 (d, J = 8.4 Hz, 1H), 6.45 (s, 1H), 2.37 (s, 3H), 1.97 (s, 3H), 1.69 (s, 3H), 1.44 (s, 3H).

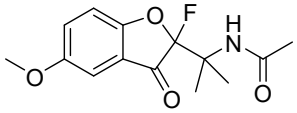
^{19}F NMR (376 MHz, CDCl3): δ -130.70 (s, 1F).

^{13}C NMR (101 MHz, CDCl3): δ 194.0 (d, *J* = 20.4 Hz), 169.8, 169.0, 140.8, 133.8, 124.9, 119.0, 112.8, 110.2 (d, *J* = 245.2 Hz), 58.0 (d, *J* = 25.5 Hz), 24.6, 20.7 (d, *J* = 3.7 Hz), 20.6, 19.8 (d, *J* = 3.3 Hz).

HRMS (ESI): m/z=288.1006 calcd for $C_{14}H_{16}F_1N_1O_3$+Na [M+ Na]$^+$, found: 288.1011;

IR (cm^{-1}):3307, 1739, 1670, 1620, 1080.

2j A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 58%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (dd, J = 9.0, 2.7 Hz, 1H), 7.12 – 7.04 (m, 2H), 6.40 (s, 1H), 3.81 (s, 3H), 1.96 (s, 3H), 1.68 (s, 3H), 1.45 (s, 3H).

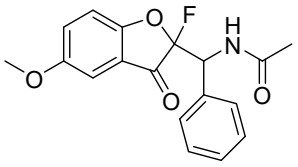
^{19}F NMR (376 MHz, $CDCl_3$): δ -130.62 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 194.0 (d, *J* = 20.4 Hz), 169.8, 165.6, 156.2, 128.9, 119.2, 114.0, 110.7 (d, *J* = 244.8 Hz), 105.6, 57.9 (d, *J* = 25.8 Hz), 56.0, 24.5, 20.8 (d, *J* = 3.8 Hz), 19.9 (d, *J* = 3.3 Hz).

HRMS (ESI): m/z=304.0956 calcd for $C_{14}H_{16}F_1N_1O_4$+Na [M+ Na]$^+$, found: 304.0961;

IR (cm^{-1}):3309, 1741, 1668, 1610, 1081.

2k A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 65%, dr >20:1).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (d, *J* = 7.0 Hz, 2H), 7.26 – 7.17 (m, 4H), 6.98 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.8 Hz, 1H), 5.85 (dd, *J* = 8.7, 7.0 Hz, 1H), 3.76 (s, 3H), 2.04 (s, 3H).

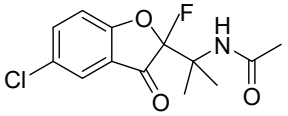
^{19}F NMR (376 MHz, $CDCl^3$): δ -120.04 (d, J = 6.6 Hz, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 194.3 (d, J = 20.0 Hz), 169.3, 166.0, 156.1, 134.2, 129.0, 128.5, 128.5, 128.1, 118.8, 114.1, 108.8 (d, J = 245.0 Hz), 105.4, 55.9, 54.5 (d, J = 30.5 Hz), 23.4.

HRMS (ESI): m/z=352.0956 calcd for $C_{18}H_{16}F_1N_1O_4$+Na [M+ Na]$^+$, found: 352.0960;

IR (cm^{-1}):3290, 1738, 1661, 1607, 1073.

2l A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 42%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 – 7.61 (m, 2H), 7.12 (d, J = 8.7 Hz, 1H), 6.16 (s, 1H), 1.94 (s, 3H), 1.68 (s, 3H), 1.47 (d, J = 1.3 Hz, 3H).

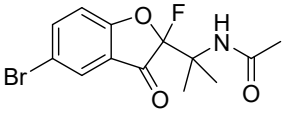
^{19}F NMR (376 MHz, $CDCl_3$): δ -128.72 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 192.1 (d, *J* = 20.8 Hz), 169.8, 168.3, 139.1, 129.5, 124.8, 120.5, 114.4, 110.8(d, *J* = 246.1 Hz), 57.7 (d, *J* = 26.3 Hz), 24.4, 21.1 (d, *J* = 4.2 Hz), 20.1 (d, *J* = 2.9 Hz).

HRMS (ESI): m/z=308.0460 calcd for $C_{13}H_{13}Cl_1F_1N_1O_3$+Na [M+ Na]$^+$, found: 308.0465;

IR (cm^{-1}):3290, 1748, 1663, 1612, 1078.

2m A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 38%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, J = 2.0 Hz, 1H), 7.76 (dd, J = 8.7, 2.1 Hz, 1H), 7.07 (d, J = 8.7 Hz, 1H), 6.17 (s, 1H), 1.93 (s, 3H), 1.67 (s, 3H), 1.47 (d, J = 1.0 Hz, 3H).

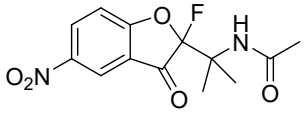
^{19}F NMR (376 MHz, $CDCl_3$): δ -128.64 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 191.8 (d, *J* = 20.7 Hz), 169.8, 168.7, 141.8, 127.9, 121.0, 116.4, 114.8, 110.6 (d, *J* = 246.2 Hz), 57.6 (d, *J* = 26.4 Hz), 24.4, 21.1 (d, *J* = 4.2 Hz), 20.1(d, *J* = 2.8 Hz).

HRMS (ESI): m/z=351.9955 calcd for $C_{13}H_{13}Br_1F_1N_1O_3$+Na [M+ Na]$^+$, found: 351.9963;

IR (cm^{-1}):3327, 1728, 1606, 1550, 1071.

2n A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 33%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 8.60 (d, J = 2.3 Hz, 1H), 8.56 (dd, J = 8.9, 2.5 Hz, 1H), 7.28 (s, 1H),

5.84 (s, 1H), 1.88 (s, 3H), 1.69 (s, 3H), 1.53 (d, J = 2.0 Hz, 3H).

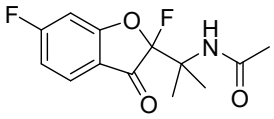
^{19}F NMR (376 MHz, $CDCl_3$): δ -125.39 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 189.8 (d, *J* = 21.3 Hz), 172.0, 169.9, 144.1, 133.5, 121.8, 120.4, 113.7, 111.8 (d, *J* = 247.0 Hz), 57.1 (d, *J* = 28.2 Hz), 23.9, 22.0 (d, *J* = 5.6 Hz), 20.6.

HRMS (ESI): 319.0701 calcd for $C_{13}H_{13}F_1N_2O_5$+Na $[M+ Na]^+$, found: 319.0706;

IR (cm^{-1}):3299, 1757, 1625, 1610, 1065.

2o A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 31%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (dd, J = 8.5, 5.6 Hz, 1H), 6.91 (td, J = 8.7, 2.1 Hz, 1H), 6.85 (dd, J = 8.6, 2.0 Hz, 1H), 6.32 (s, 1H), 1.95 (s, 3H), 1.68 (s, 3H), 1.46 (d, J = 1.1 Hz, 3H).

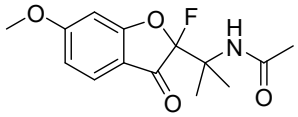
^{19}F NMR (376 MHz, $CDCl_3$): δ -92.58 (td, J = 8.7, 6.5 Hz, 1F), -129.10 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ191.4 (d, J = 20.4 Hz), 171.8 (d, J = 15.0Hz),169.9(d,J=260.6Hz)，169.8, 127.5 (d, J = 12.2 Hz), 115.8, 112.5 (d, J = 24.1 Hz), 110.9 (d, J = 247.0 Hz), 101.2 (d, J = 26.7 Hz), 57.8 (d, J = 25.8 Hz), 24.4, 20.9 (d, J = 3.9 Hz), 20.0 (d, J = 3.3 Hz).

HRMS (ESI): m/z=292.0756 calcd for $C_{13}H_{13}F_2N_1O_3$+Na $[M+ Na]^+$, found: 292.0760;

IR (cm^{-1}):3306, 1740, 1670, 1619, 1078.

2p A white solid after purification by flash column chromatography (petroleum ether/ethyl acetate = 4/1, yield 25%).



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, J = 8.6 Hz, 1H), 6.72 (dd, J = 8.6, 2.1 Hz, 1H), 6.63 (s, 1H), 6.60 (d, J = 2.0 Hz, 1H), 3.92 (s, 3H), 1.98 (s, 3H), 1.70 (s, 3H), 1.43 (s, 3H).

^{19}F NMR (376 MHz, $CDCl_3$): δ -130.29 (s, 1F).

^{13}C NMR (101 MHz, $CDCl_3$): δ 191.4 (d, *J* = 20.1 Hz), 173.2, 169.9, 169.7, 126.7, 112.0, 111.9,110.8 (d, *J* = 24.4 Hz), 96.7, 58.1 (d, *J* = 25.2 Hz), 56.2, 24.6, 20.6 (d, *J* = 3.5 Hz), 19.8(d, *J* = 3.9 Hz).

HRMS (ESI): m/z=304.0956 calcd for $C_{14}H_{16}F_1N_1O_4$+Na $[M+ Na]^+$, found: 304.0960;

IR (cm^{-1}):3313, 1725, 1672, 1618, 1080.

Theoretical section

All computations were performed using the Gaussion09 program[4]. The method RB3LYP with the basis set of 6-31g were performed for geometry optimization and energy calculation. The total energy values of α-fluorine benzofuranone (Fig. S1a) and α-fluorine benzopyranone (Fig. S1b) are obtained by subtracting the lowest values in the same calculation methods and CH_3CN solution. The result shows that five-membered ring of α-fluorine benzofuranone is more stable and that the energy difference between the two molecules is 36.97 kJ/mol.

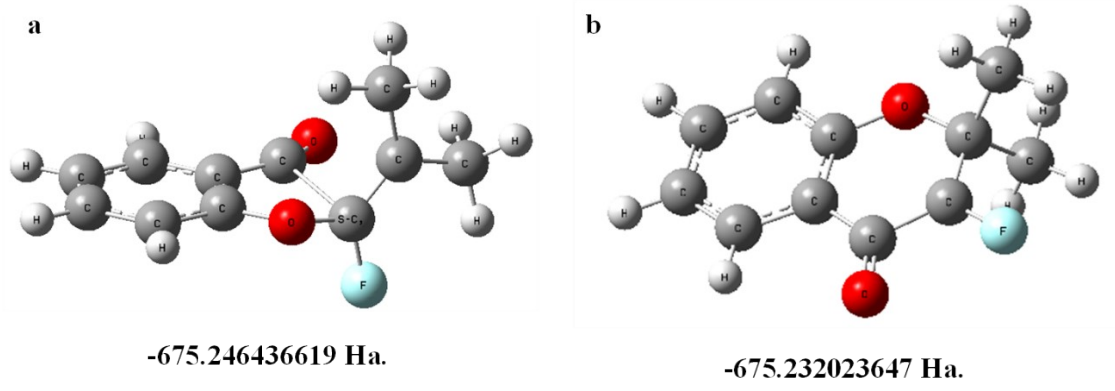
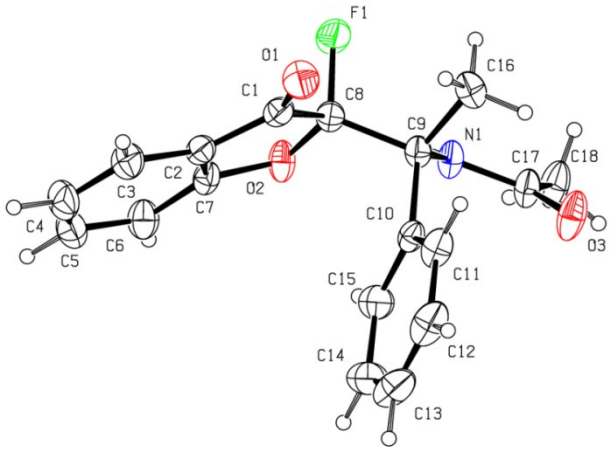


Fig. S1 Optimized molecular structures of α-fluorine benzofuraone (a) and α-fluorine benzopyranone (b) at the RB3LYP/6-31g level of calculations.

Absolute Configuration and X-Ray Analysis Data of Product 2h and 2j

X-ray structure of compound 2h



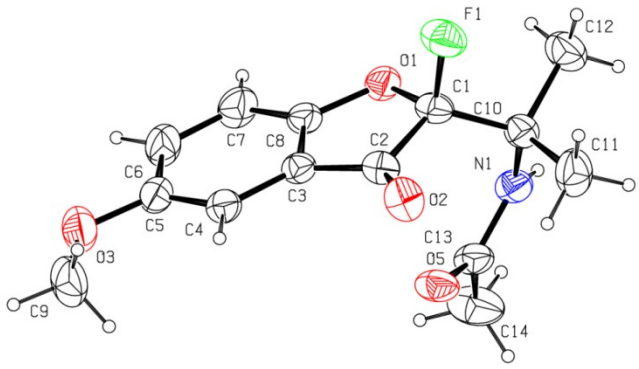
Crystal Data for $C_{14}H_{16}FNO_4$ (M =281.28 g/mol): trigonal, space group R-3 (no. 148), a = 26.022(3) Å, c = 12.7042(8) Å, V = 7450.0(16) Å^3, Z = 18, T = 290.83(10) K, μ(MoKα) = 0.090 mm^{-1}, *Dcalc* = 1.128 g/cm^3, 6612 reflections measured (6.664° ≤ 2Θ ≤ 52.034°), 3254 unique (R_{int} = 0.0441, R_{sigma} = 0.1028) which were used in all calculations. The final R_1 was 0.0856 (I > 2σ(I)) and wR_2 was 0.2635 (all data).

Table 1 Crystal data and structure refinement for zhangwt_0317.

Identification code	zhangwt_0317
Empirical formula	$C_{14}H_{16}FNO_4$
Formula weight	281.28
Temperature/K	290.83(10)
Crystal system	trigonal
Space group	R-3
a/Å	26.022(3)
b/Å	26.022(3)
c/Å	12.7042(8)
α/°	90
β/°	90
γ/°	120
Volume/Å^3	7450.0(16)
Z	18
ρ_{calc}g/cm^3	1.128
μ/mm^{-1}	0.090
F(000)	2664.0

Crystal size/mm^3	0.31 × 0.25 × 0.24
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.664 to 52.034
Index ranges	-14 ≤ h ≤ 34, -25 ≤ k ≤ 33, -17 ≤ l ≤ 9
Reflections collected	6612
Independent reflections	3254 [R_{int} = 0.0441, R_{sigma} = 0.1028]
Data/restraints/parameters	3254/0/185
Goodness-of-fit on F^2	1.004
Final R indexes [I>=2σ (I)]	R_1 = 0.0856, wR_2 = 0.2086
Final R indexes [all data]	R_1 = 0.1938, wR_2 = 0.2635
Largest diff. peak/hole / e Å^{-3}	0.61/-0.24

X-ray structure of compound 2j



Crystal Data for $C_{14}H_{16}FNO_4$ (M =281.28 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 11.6384(6) Å, b = 10.1354(4) Å, c = 23.7540(13) Å, β = 93.521(5)°, V = 2796.7(2) Å^3, Z = 8, T = 293.23(10) K, μ(MoKα) = 0.106 mm^{-1}, *Dcalc* = 1.336 g/cm^3, 10998 reflections measured (6.88° ≤ 2Θ ≤ 52.04°), 5483 unique (R_{int} = 0.0507, R_{sigma} = 0.0793) which were used in all calculations. The final R_1 was 0.0726 (>2sigma(I)) and wR_2 was 0.2293 (all data).

Table 1 Crystal data and structure refinement for mabaochun_0626.

Identification code	mabaochun_0626
Empirical formula	$C_{14}H_{16}FNO_4$
Formula weight	281.28
Temperature/K	293.23(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	11.6384(6)

b/Å	10.1354(4)
c/Å	23.7540(13)
α/°	90.00
β/°	93.521(5)
γ/°	90.00
Volume/Å^3	2796.7(2)
Z	8
ρ_{calc}g/cm^3	1.336
μ/mm^{-1}	0.106
F(000)	1184.0
Crystal size/mm^3	0.15 × 0.12 × 0.07
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.88 to 52.04
Index ranges	-14 ≤ h ≤ 14, -12 ≤ k ≤ 7, -29 ≤ l ≤ 16
Reflections collected	10998
Independent reflections	5483 [R_{int} = 0.0507, R_{sigma} = 0.0793]
Data/restraints/parameters	5483/0/369
Goodness-of-fit on F^2	1.049
Final R indexes [I>=2σ (I)]	R_1 = 0.0726, wR_2 = 0.1742
Final R indexes [all data]	R_1 = 0.1374, wR_2 = 0.2293
Largest diff. peak/hole / e Å^{-3}	0.21/-0.30

CheckCIF reports of 2h and 2j

CheckCIF report of **2h**

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) mabaochun_0626

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: mabaochun_0626

Bond precision: C-C = 0.0051 A Wavelength=0.71073

Cell: a=11.6384(6) b=10.1354(4) c=23.7540(13)
alpha=90 beta=93.521(5) gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	2796.7(2)	2796.7(2)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C14 H16 F N O4	C14 H16 F N O4
Sum formula	C14 H16 F N O4	C14 H16 F N O4
Mr	281.28	281.28
Dx,g cm-3	1.336	1.336
Z	8	8
Mu (mm-1)	0.106	0.106
F000	1184.0	1184.0
F000'	1184.73	
h,k,lmax	14,12,29	14,12,29
Nref	5498	5483
Tmin,Tmax	0.985,0.993	0.365,1.000
Tmin'	0.984	

Correction method= # Reported T Limits: Tmin=0.365 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997 Theta(max)= 26.020

R(reflections)= 0.0726(2784) wR2(reflections)= 0.2293(5483)
S = 1.049 Npar= 369

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT910_ALERT_3_B Missing # of FCF Reflection(s) Below Theta(Min). 13 Note

Alert level C

PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C13 Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00508 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 6.823 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 6 Report

Alert level G

PLAT005_ALERT_5_G No Embedded Refinement Details Found in the CIF Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 2 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 293 Check
PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 293 Check
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O1 . 107.5 Degree
PLAT398_ALERT_2_G Deviating C-O-C Angle From 120 for O5 . 107.4 Degree
PLAT793_ALERT_4_G Model has Chirality at C1 (Centro SPGR) S Verify
PLAT793_ALERT_4_G Model has Chirality at C15 (Centro SPGR) S Verify
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info

0 **ALERT level A** = Most likely a serious problem - resolve or explain
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2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
4 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

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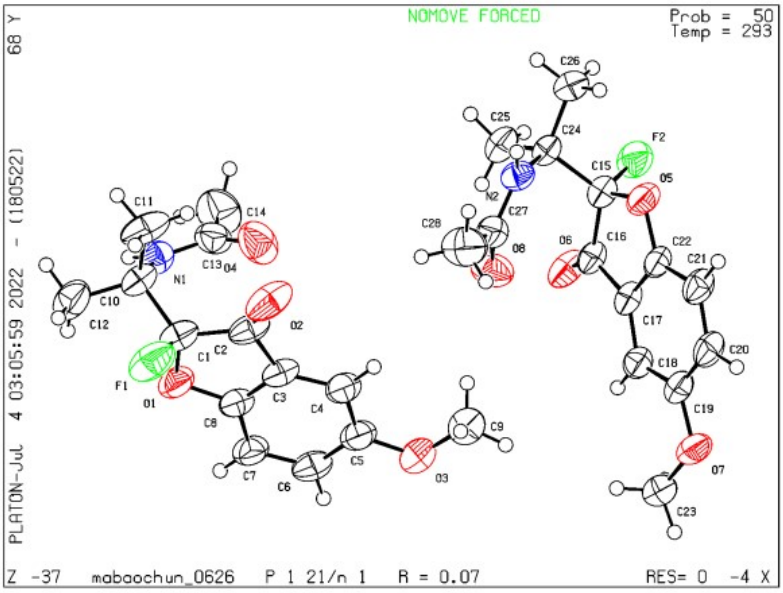
PLATON version of 18/05/2022; check.def file version of 17/05/2022

Datablock mabaochun_0626 - ellipsoid plot



CheckCIF report of **2j**

checkCIF/PLATON report

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Temperature: 293 K

	Calculated	Reported
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Moiety formula	C14 H16 F N O4	C14 H16 F N O4
Sum formula	C14 H16 F N O4	C14 H16 F N O4
Mr	281.28	281.28
Dx,g cm-3	1.336	1.336
Z	8	8
Mu (mm-1)	0.106	0.106
F000	1184.0	1184.0
F000'	1184.73	
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Nref	5498	5483
Tmin,Tmax	0.985,0.993	0.365,1.000
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PLAT793_ALERT_4_G Model has Chirality at C15 (Centro SPGR) S Verify
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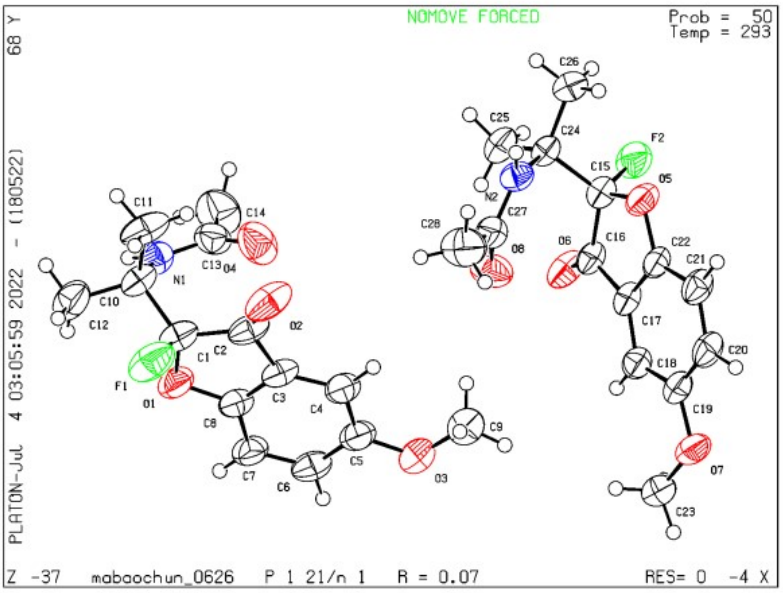
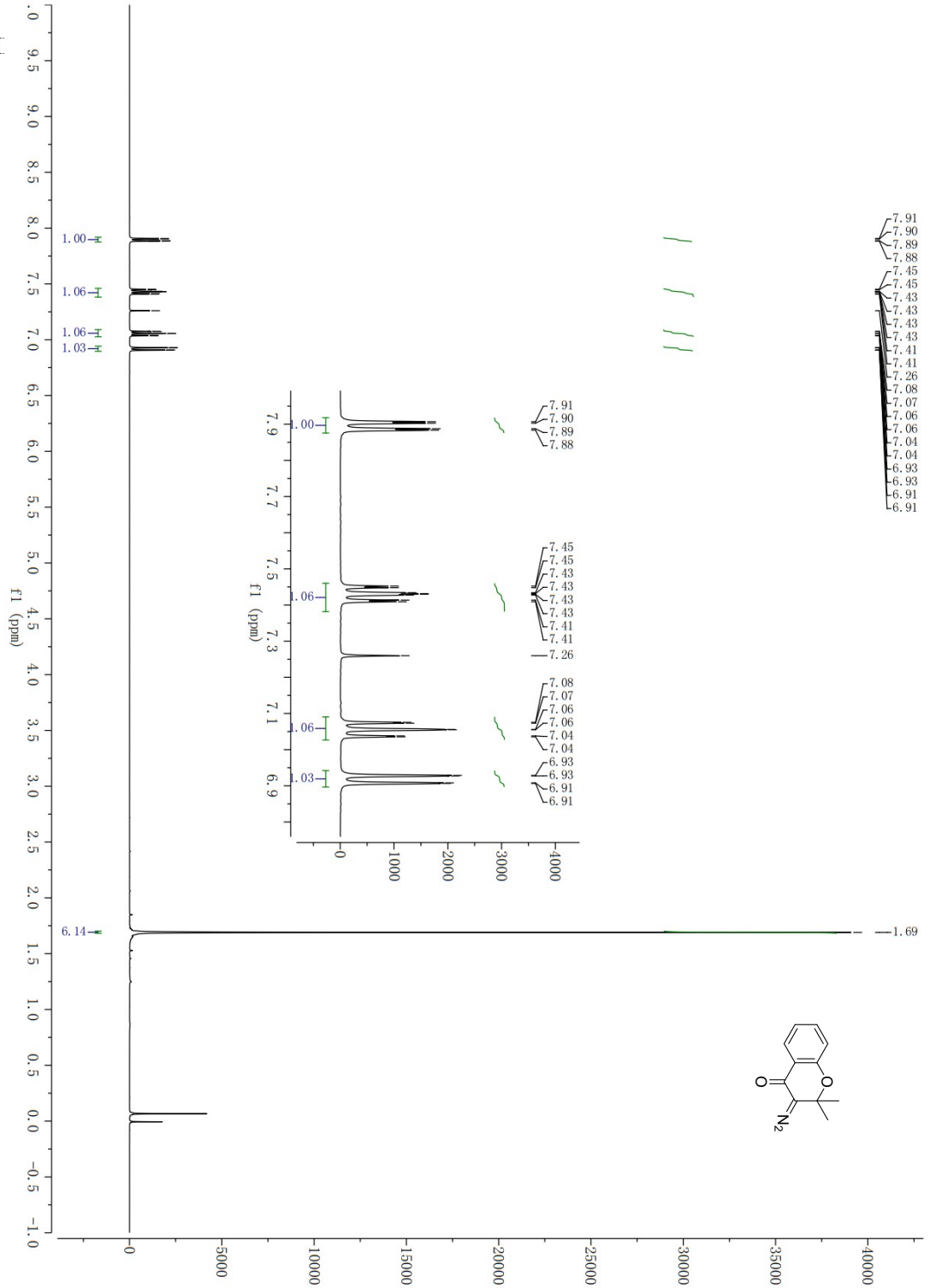


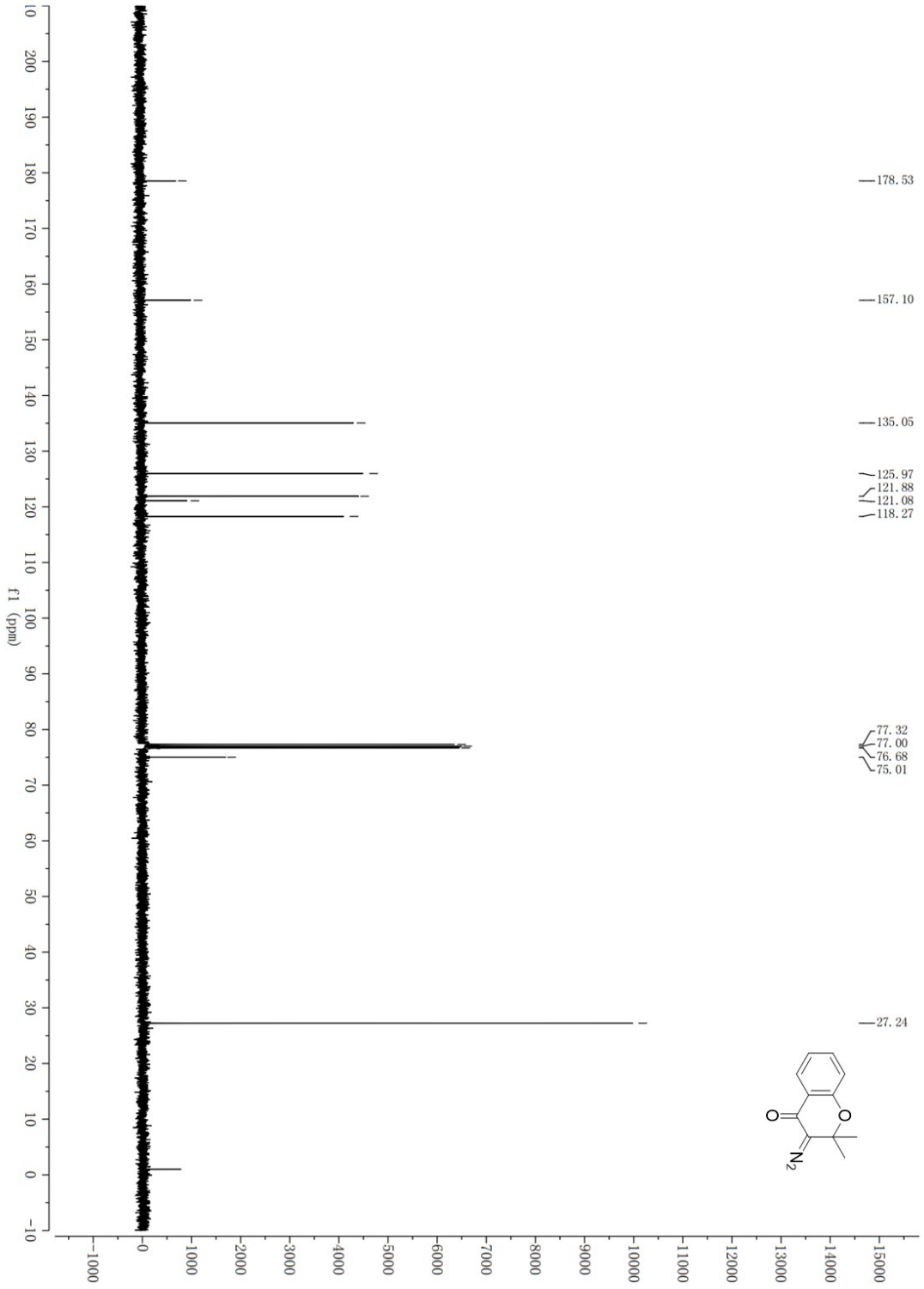
Spectrum of ^{1}H NM

(1a)

^{1}H NMR



^{13}C NMR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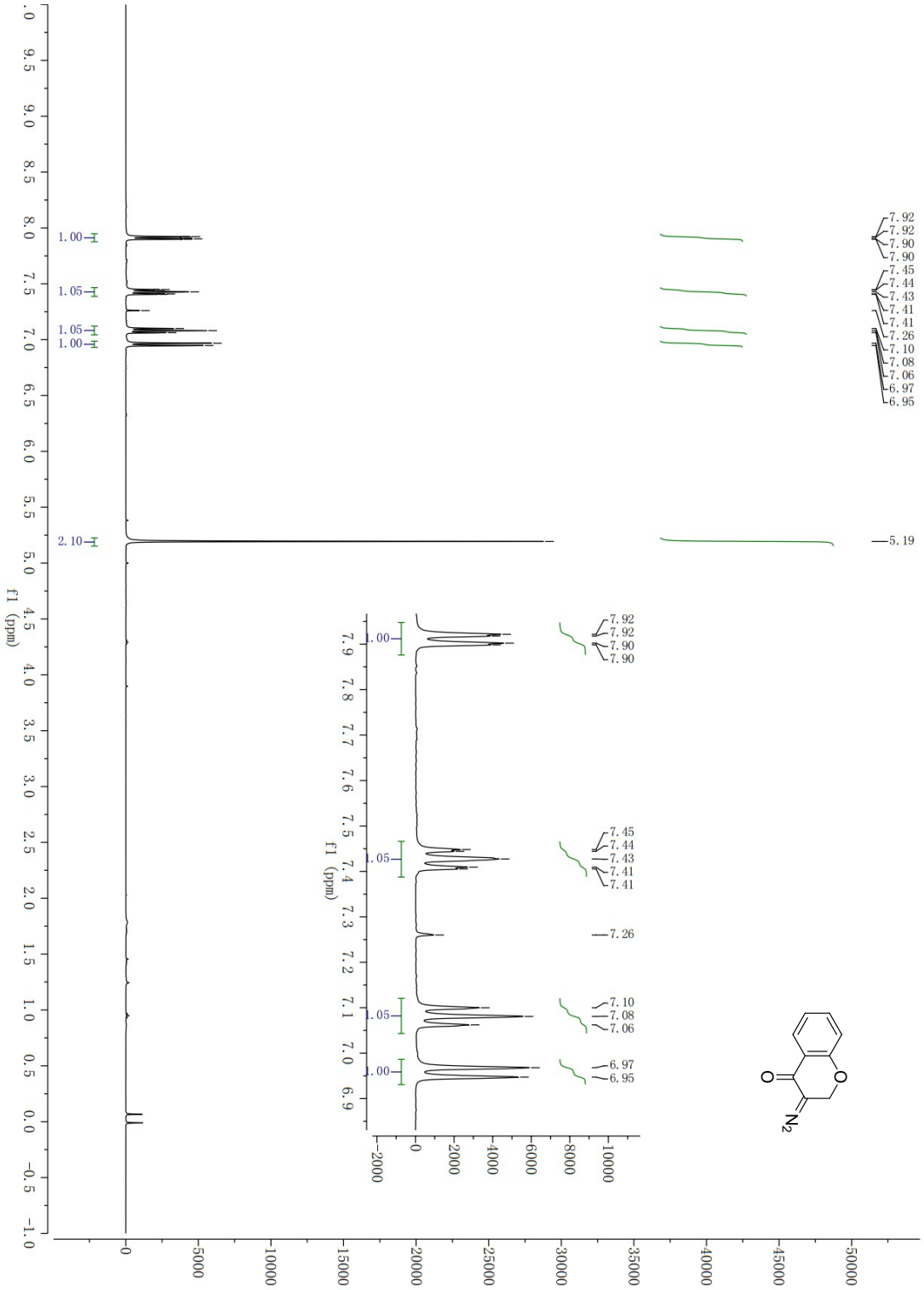
O
N2

(1b)

^{1}H NMR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7.41
7.41
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f1 (ppm)
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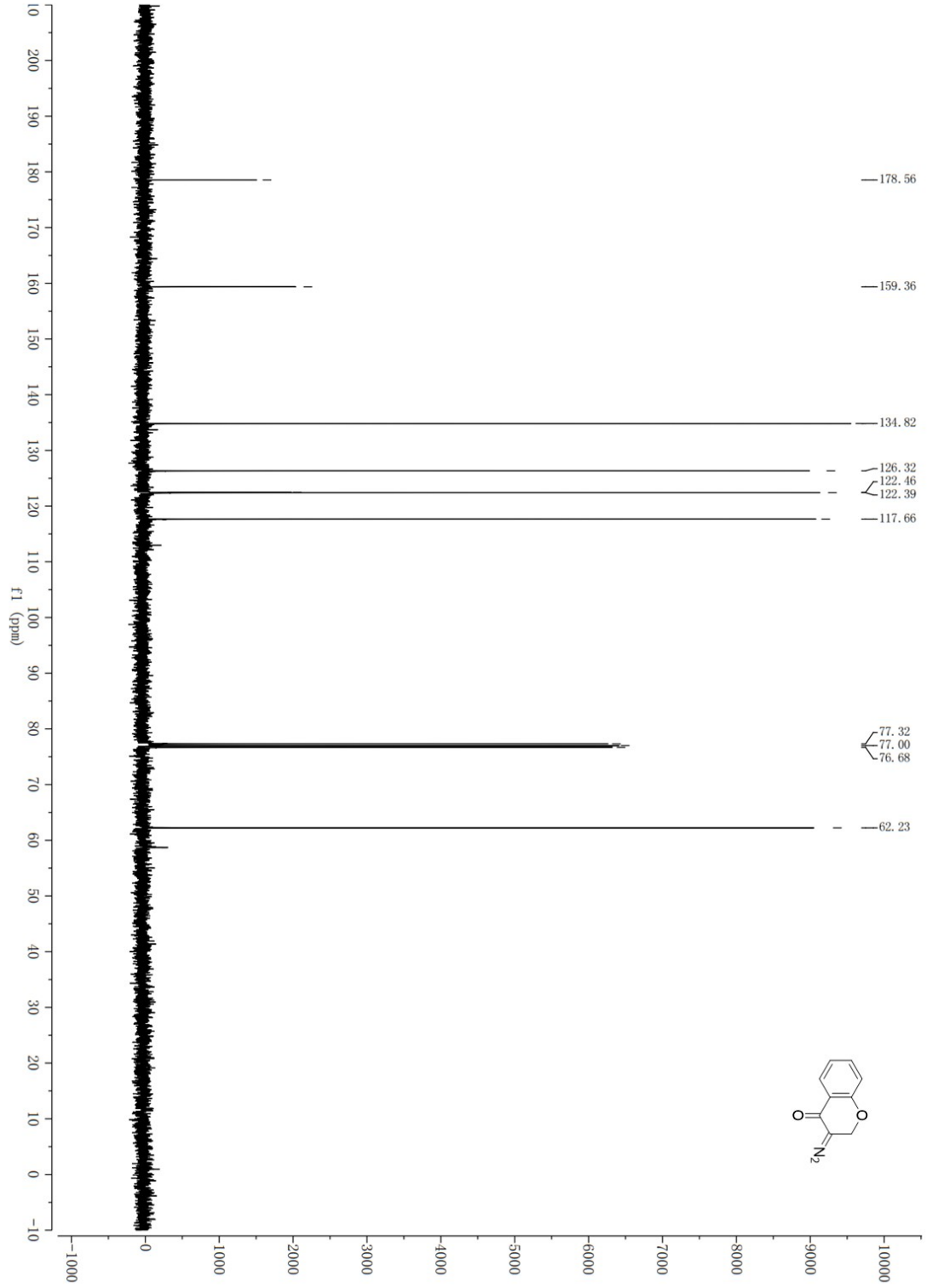
O
N2

^{13}C NMR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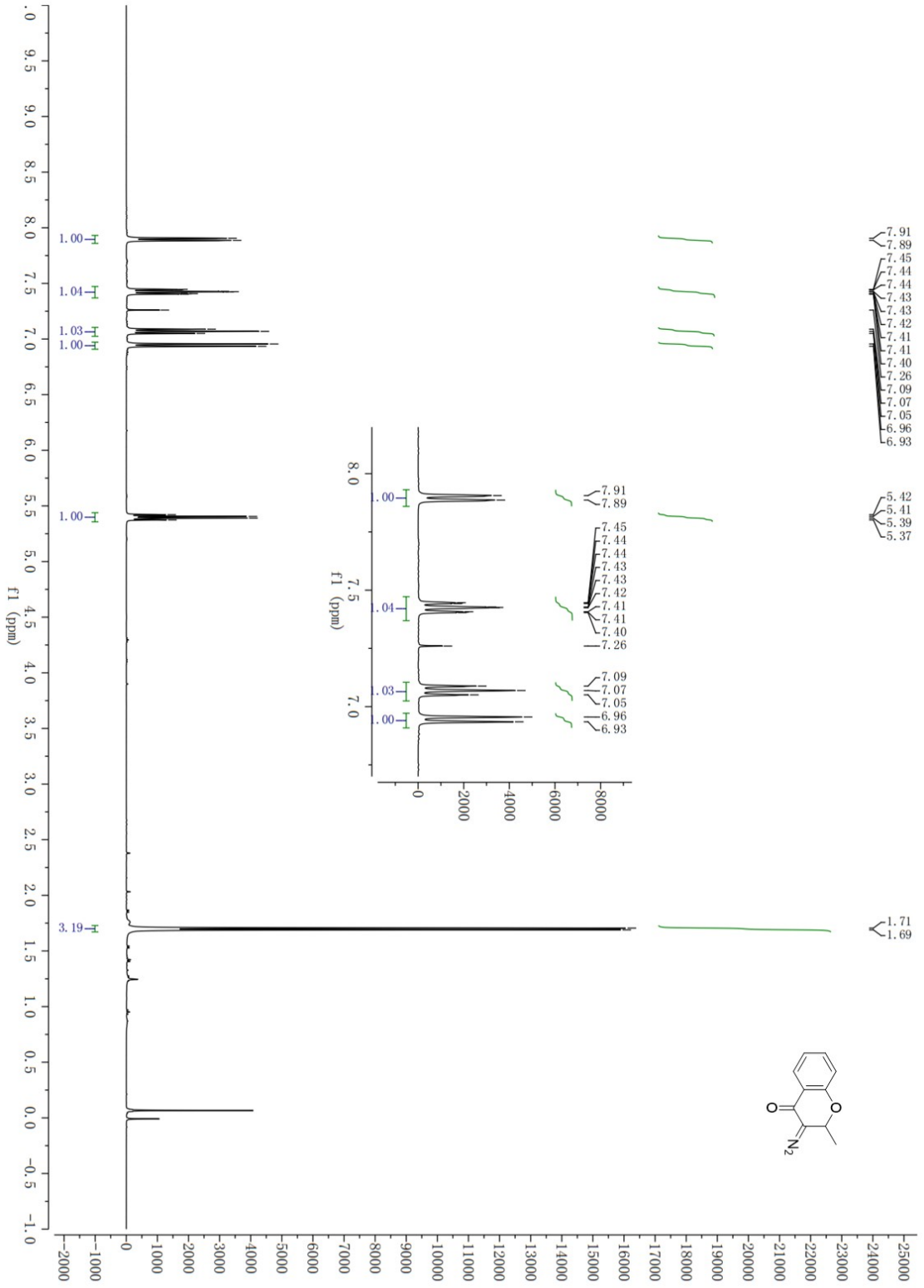
O
N2

(1c)

^{1}H NMR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7.26
7.09
7.07
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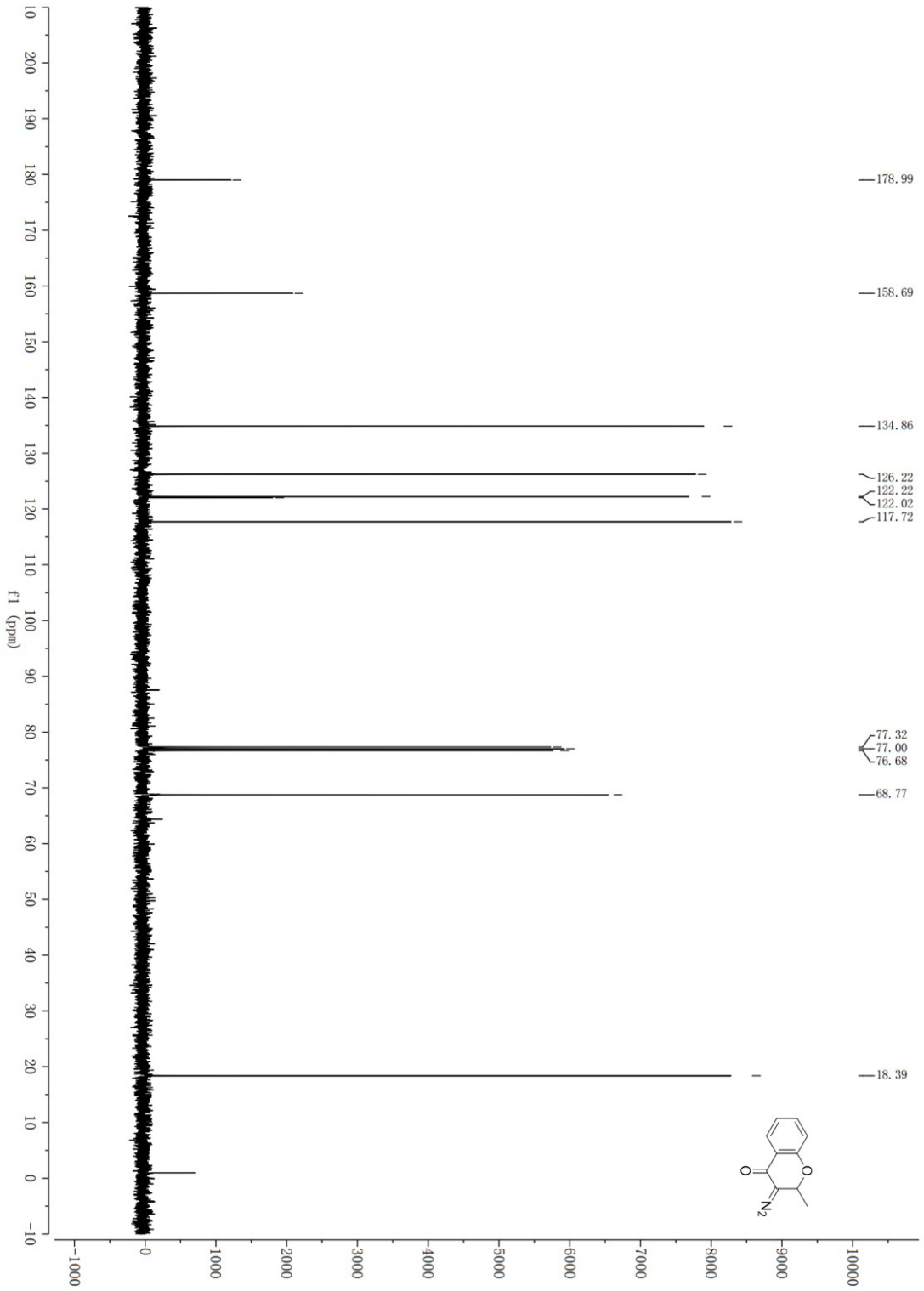
O
N2

^{13}C NMR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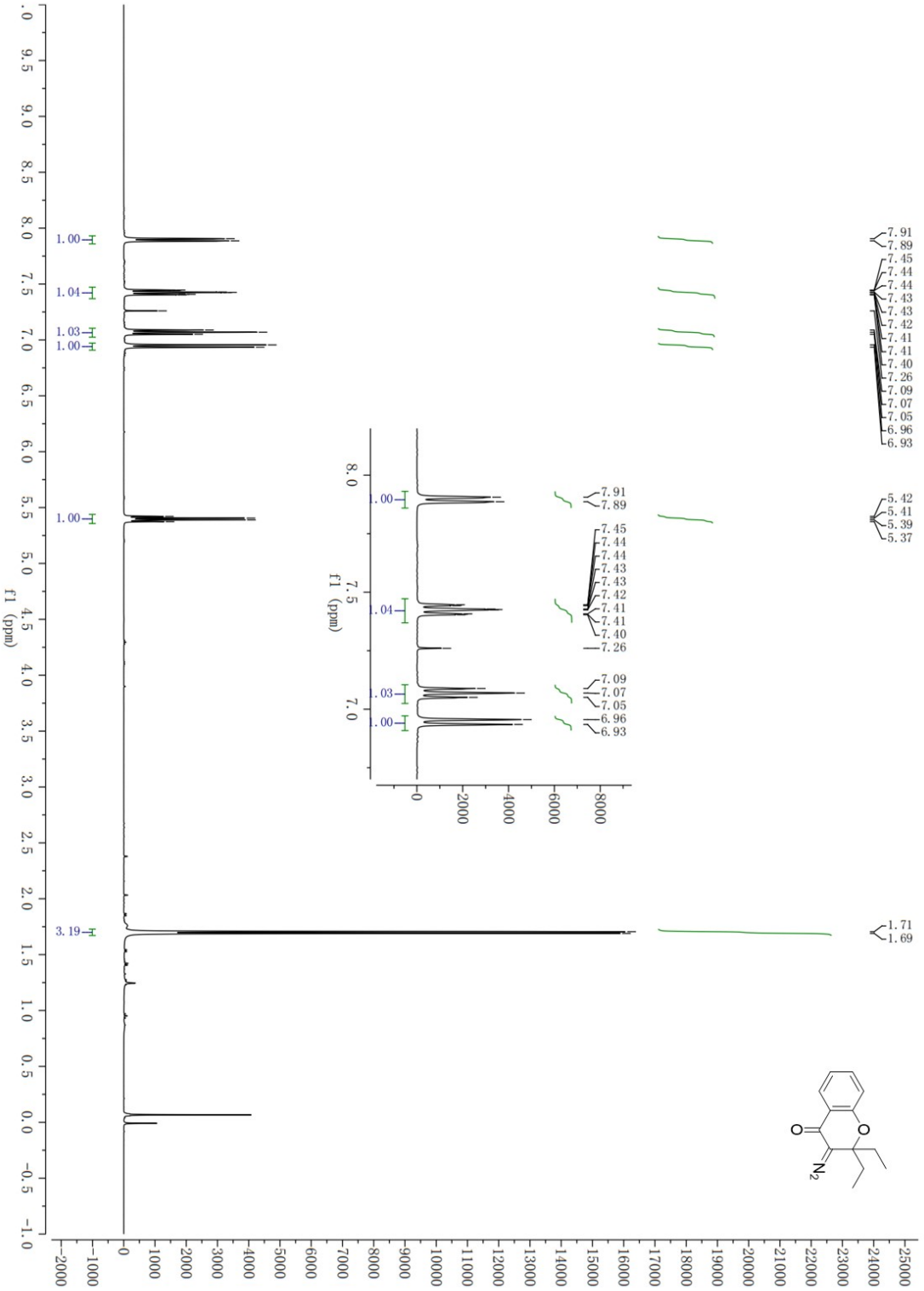
O
N2

(1d)

^{1}H NMR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7.43
7.43
7.42
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7.41
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7.07
7.05
6.96
6.93
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5.41
5.39
5.37
1.71
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1.03
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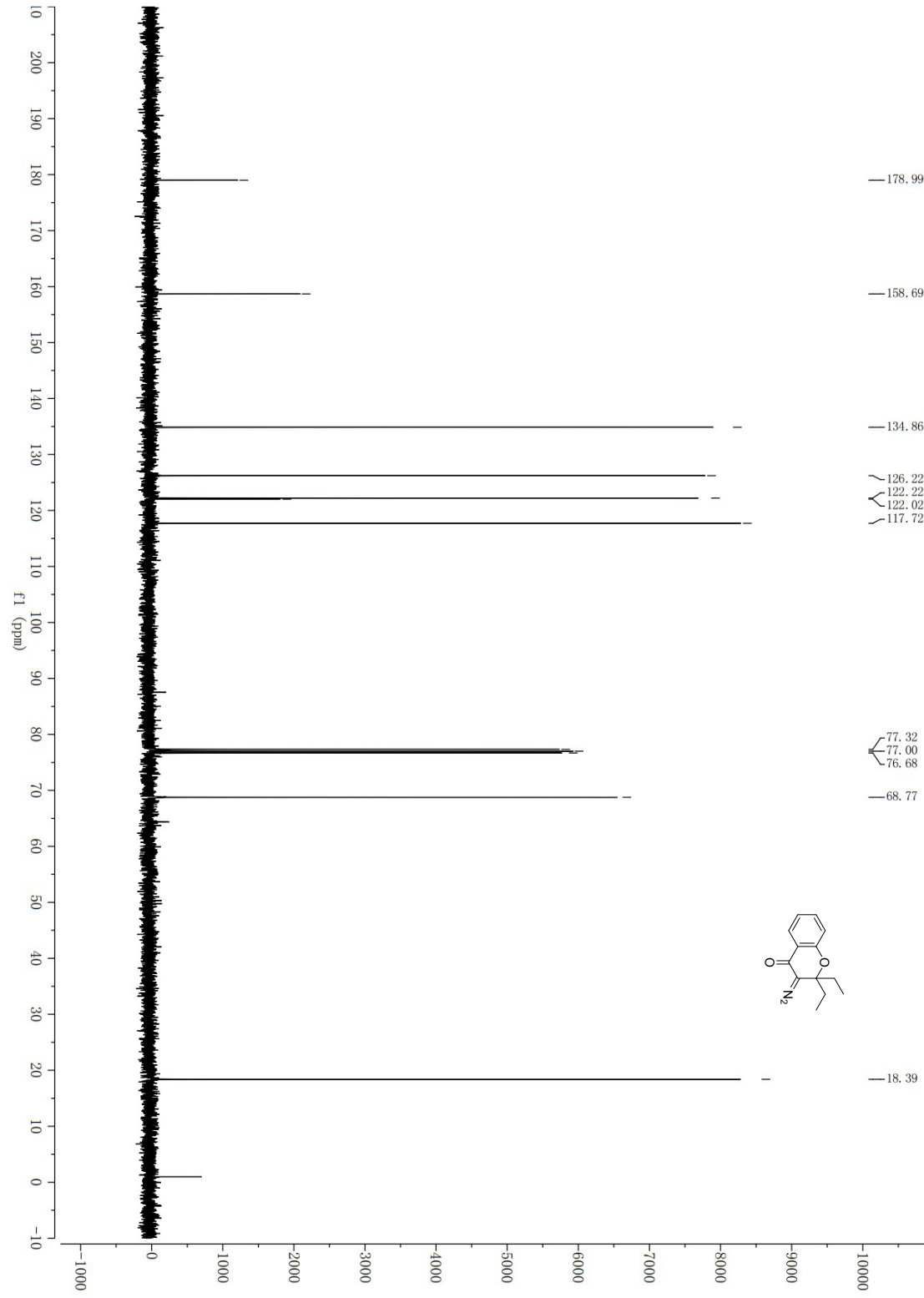
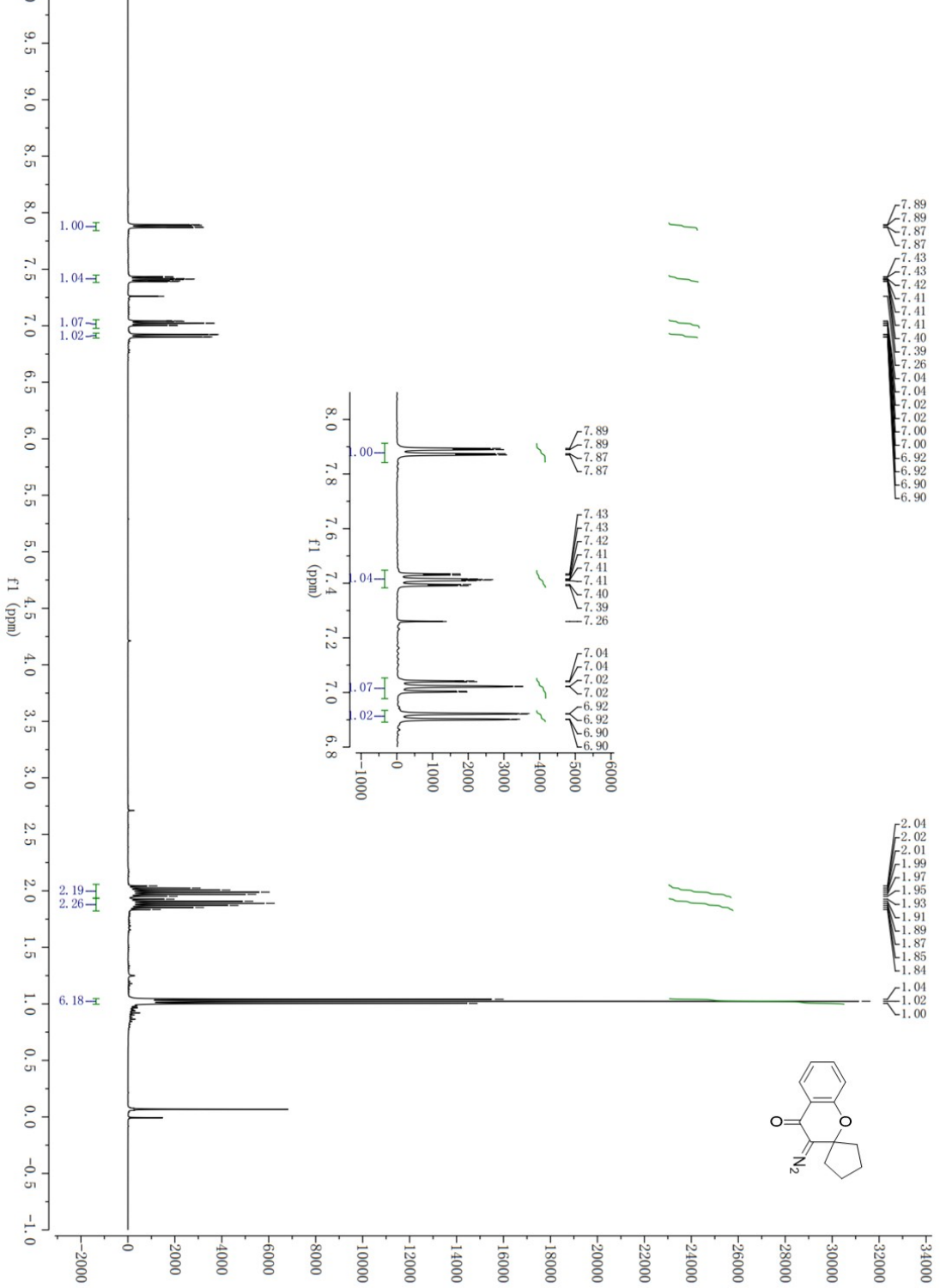
1.00
3.19
f1 (ppm)
O
O
N2

^{13}C NMR



(1e)

^{1}H NMR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7.40
7.39
7.26
7.04
7.02
6.92
6.90
2.04
2.02
2.01
1.99
1.97
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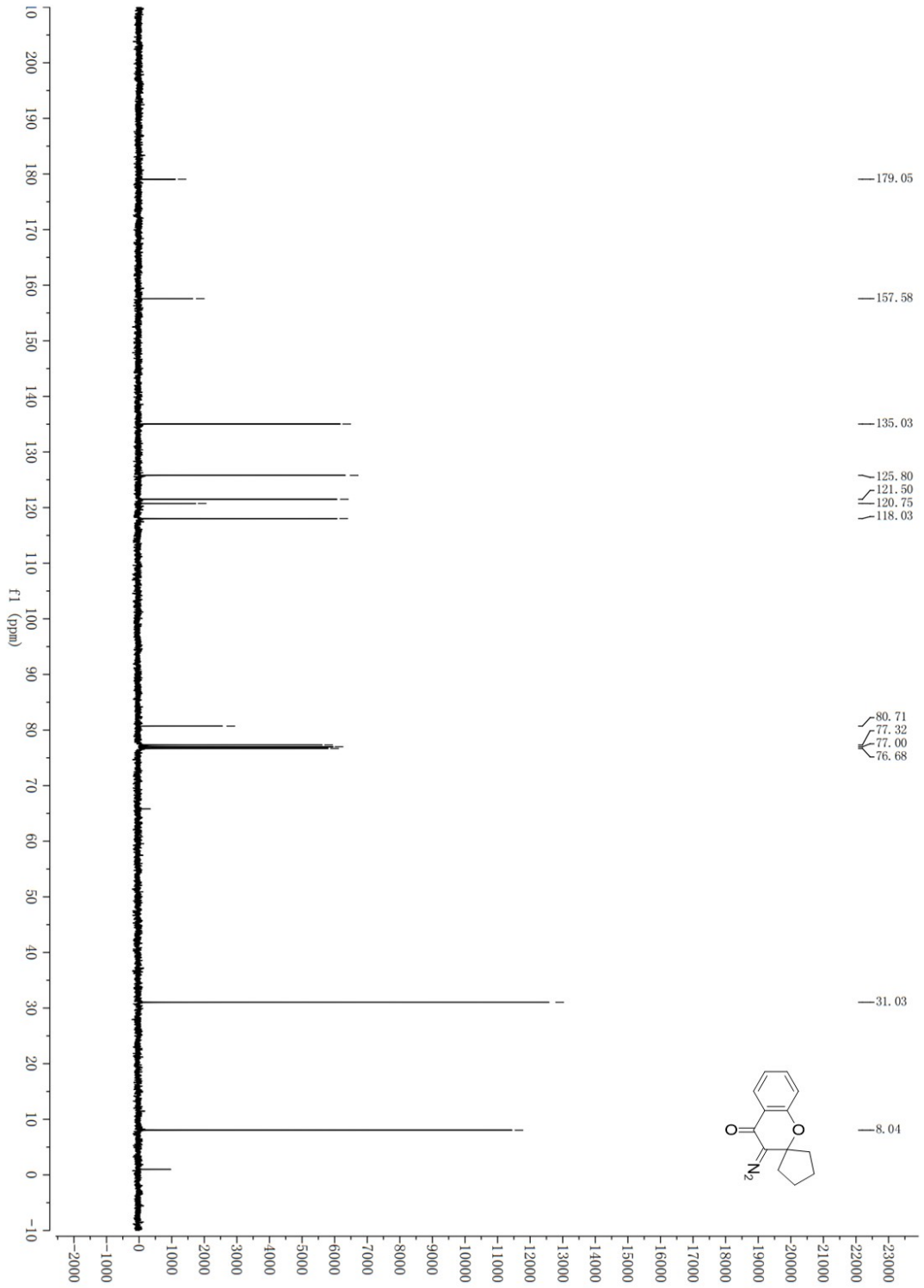
1.93
1.91
1.89
1.87
1.85
1.84
O
O
N2

^{13}C NMR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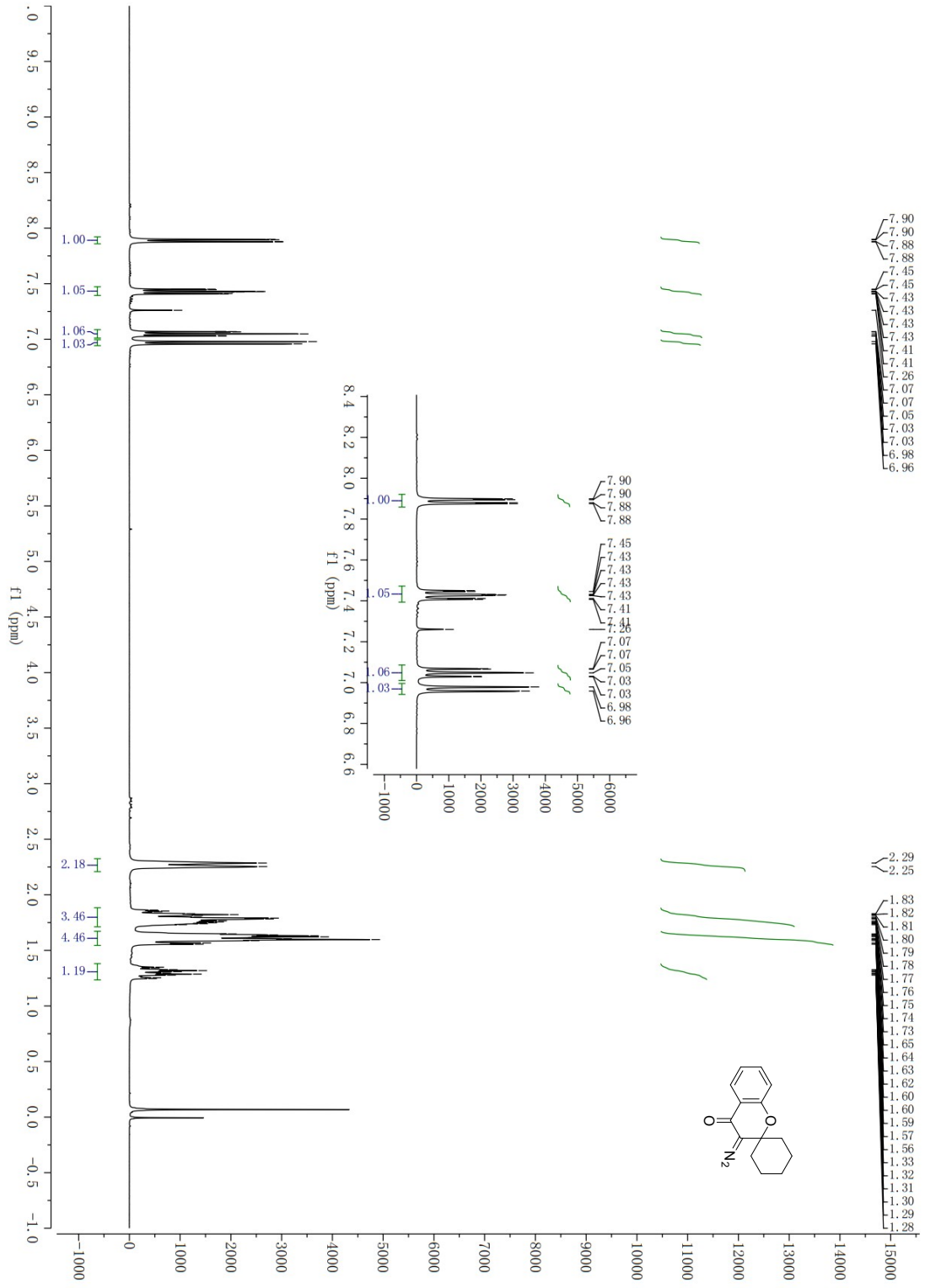
f1 (ppm)

(1f)

^{1}H NMR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7.07
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6.98
6.96
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1.77
1.76
1.75
1.74
1.73
1.65
1.64
1.63
1.62
1.60
1.59
1.57
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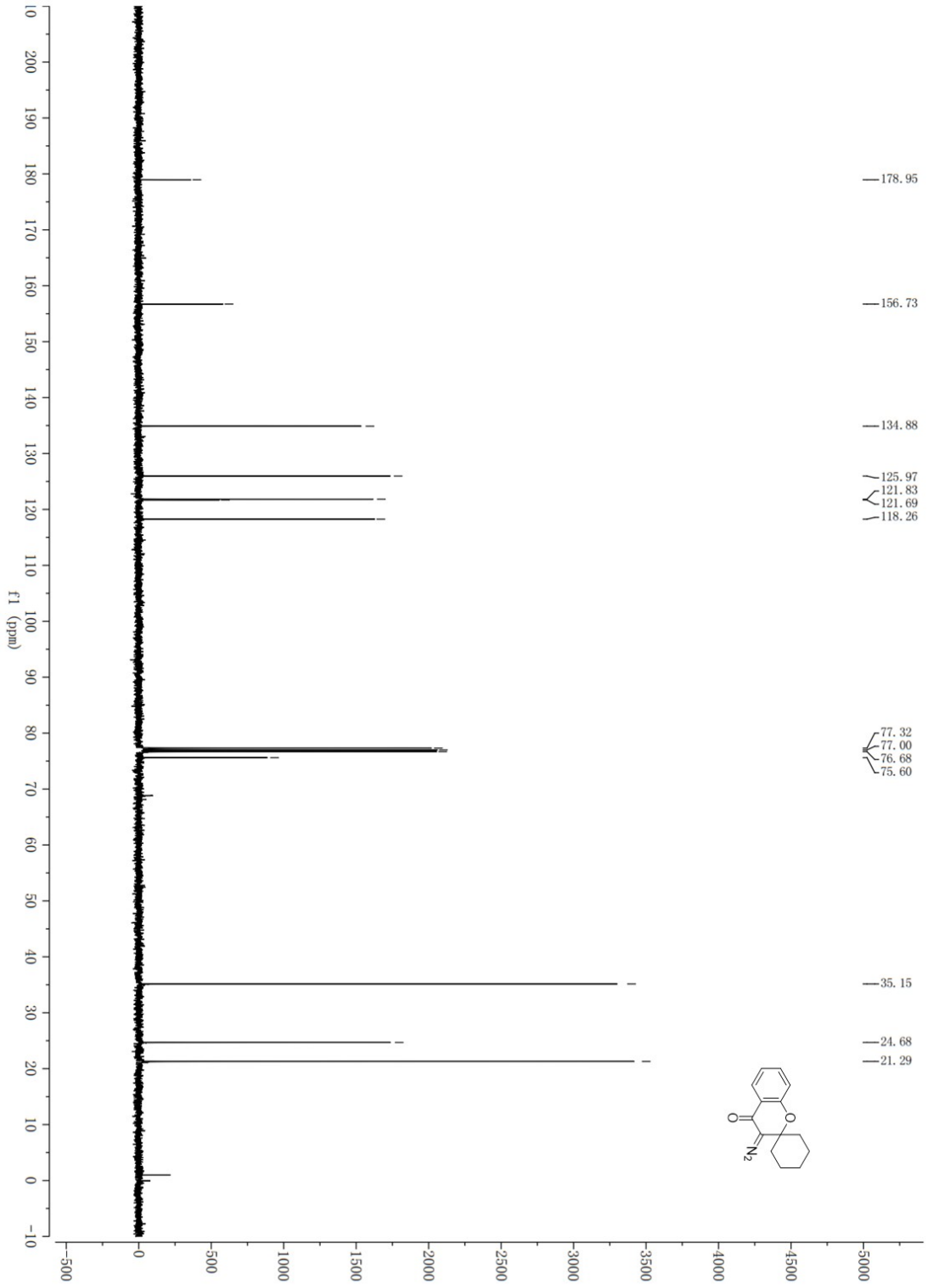
1.33
1.32
1.31
1.30
1.29
1.28
O
O
N2

^{13}C NMR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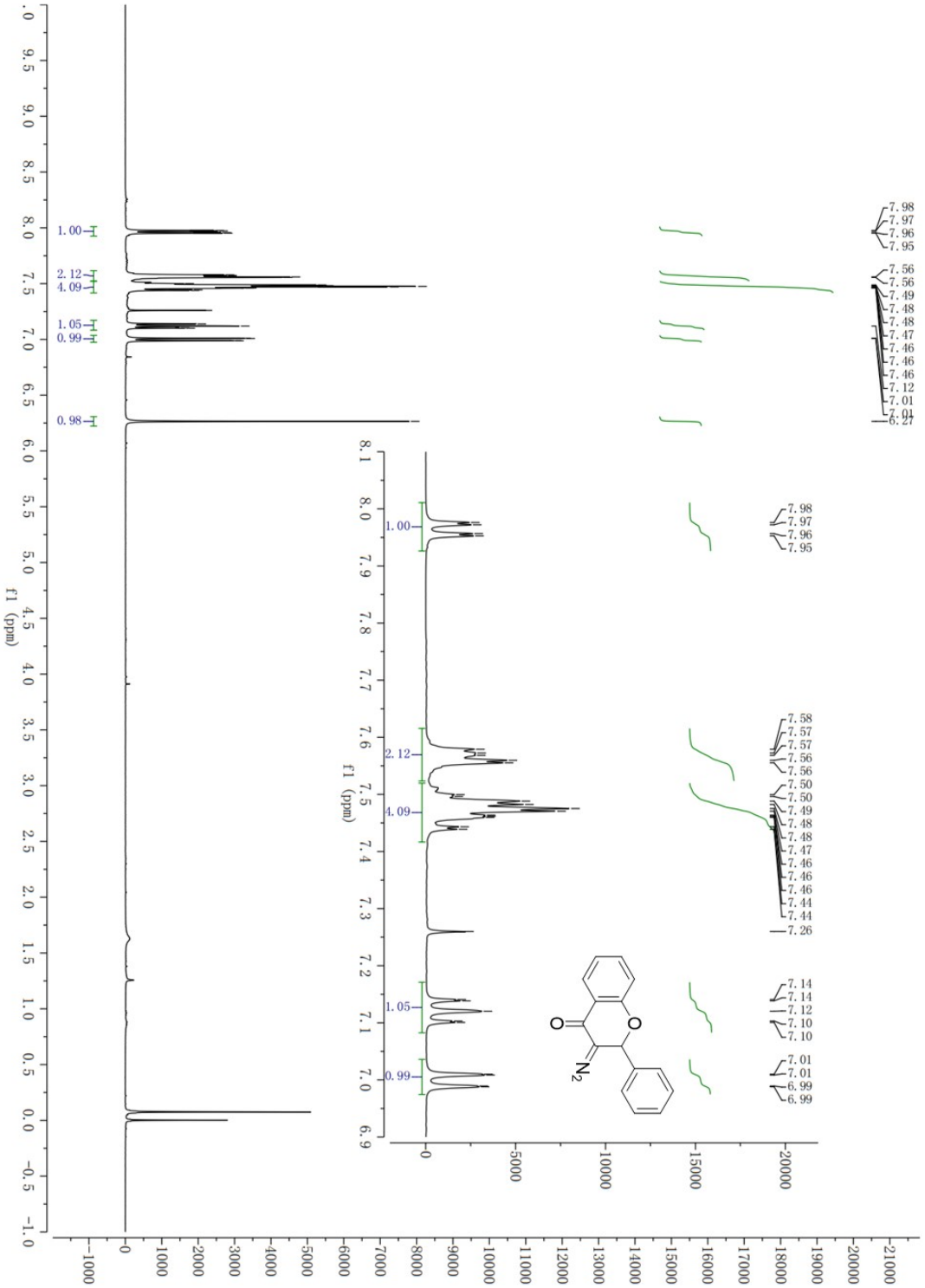
121.69
118.26
77.32
77.00
76.68
75.60
35.15
24.68
21.29
f1 (ppm)
O
O
N2

(1g)

^{1}H NMR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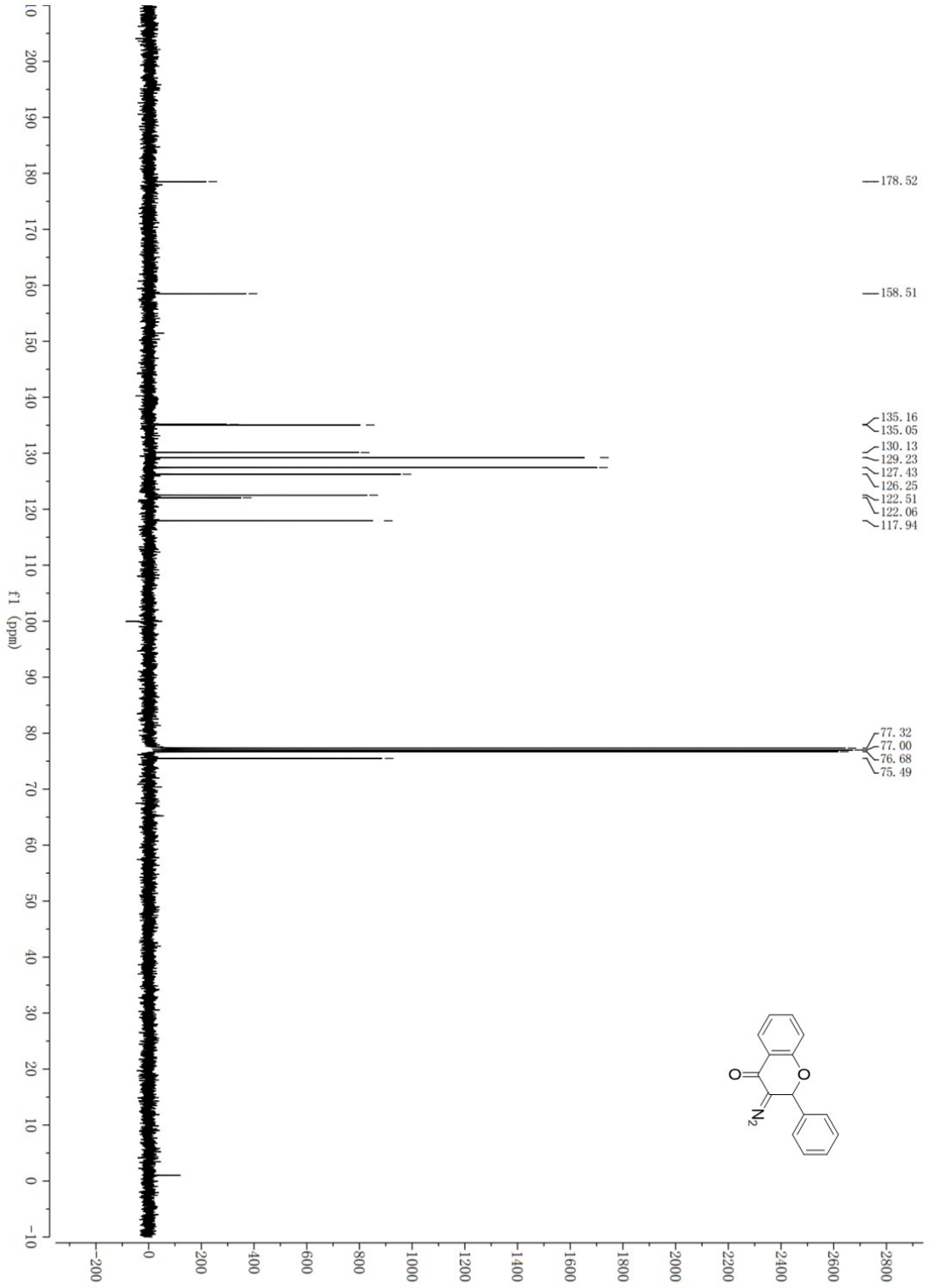
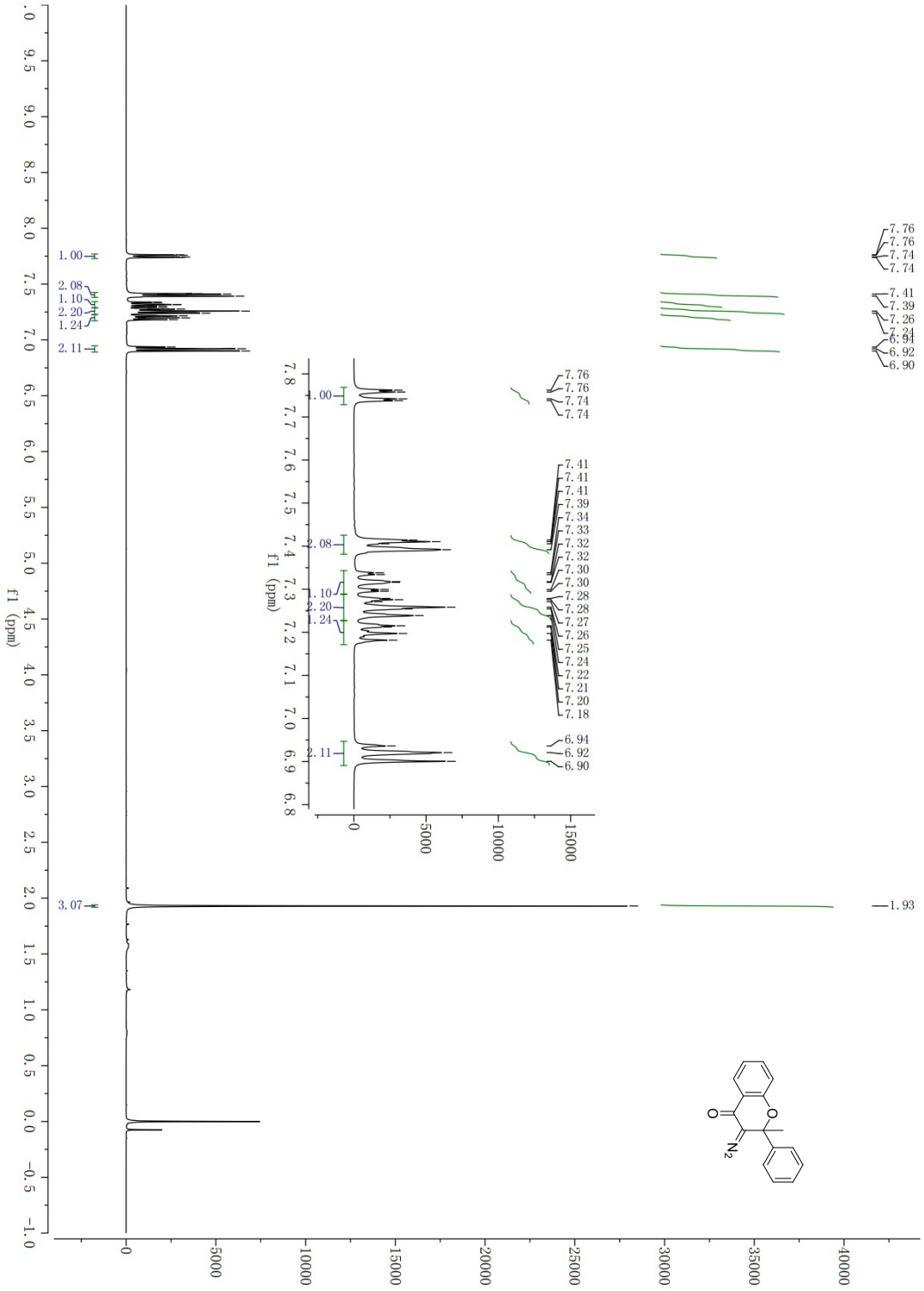
0.99
0.98
7.98
7.97
7.96
7.95
7.56
7.49
7.48
7.47
7.46
7.12
7.01
6.27
7.58
7.57
7.50
7.44
7.26
7.14
7.10
6.99
O
N2

^{13}C NMR

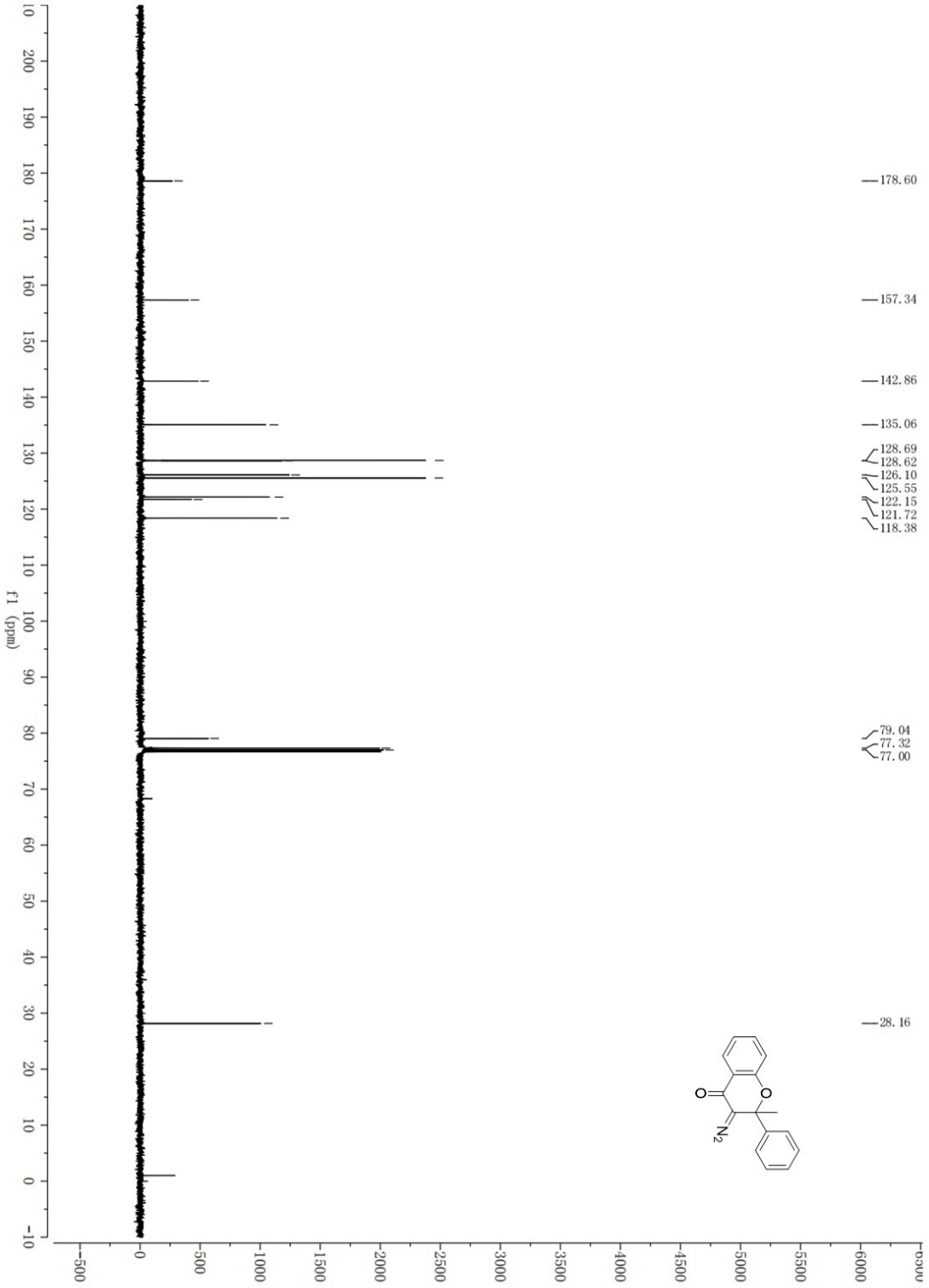

178.52
158.51
135.16
135.05
130.13
129.23
127.43
126.25
122.51
122.06
117.94
77.32
77.00
76.68
75.49
f1 (ppm)

(1h)

^{1}H NMR

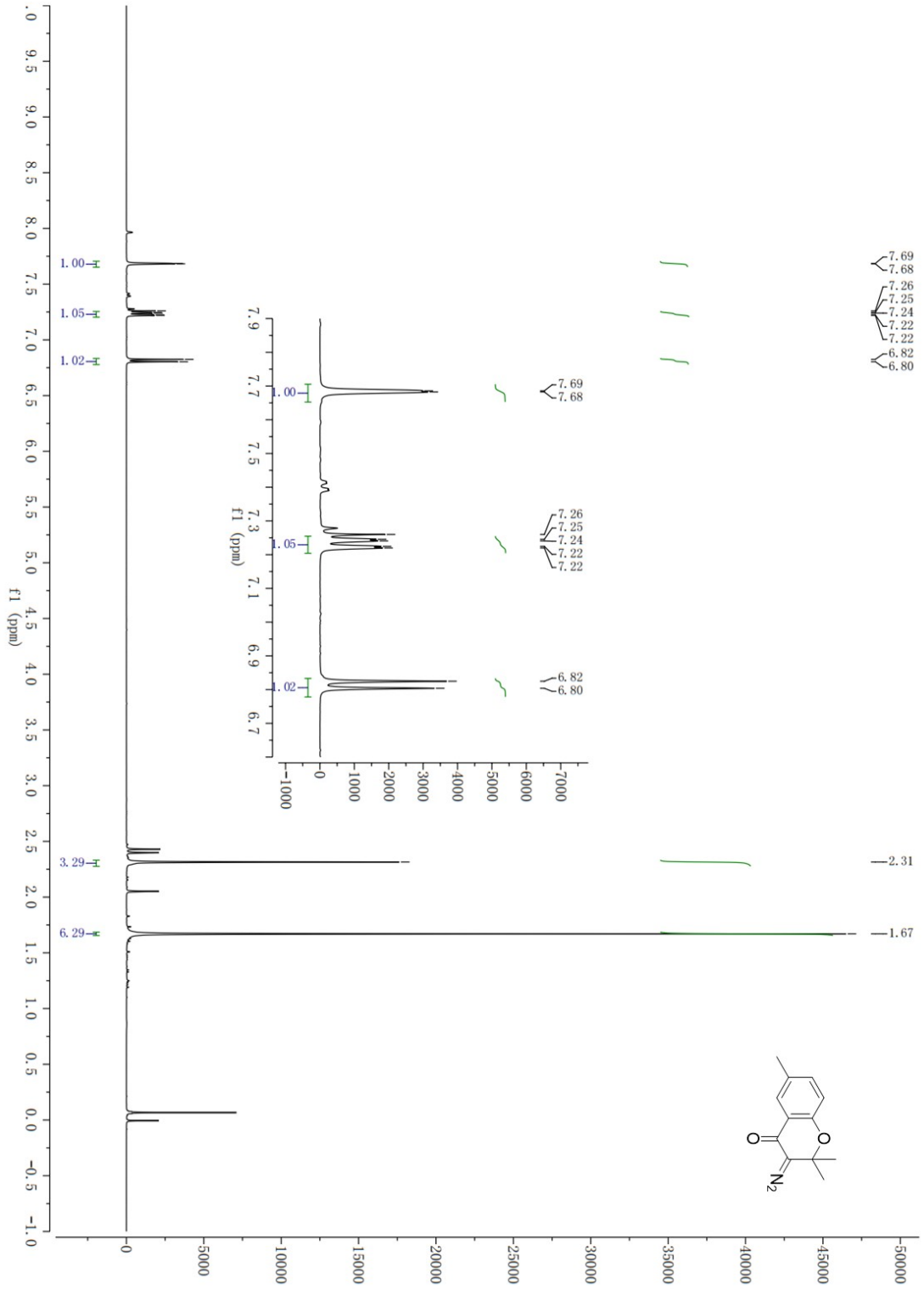

1.00
2.08
1.10
2.20
1.24
2.11
3.07
1.93
f1 (ppm)
7.76
7.74
7.41
7.39
7.26
6.94
6.92
6.90
O
O
N2

^{13}C NMR

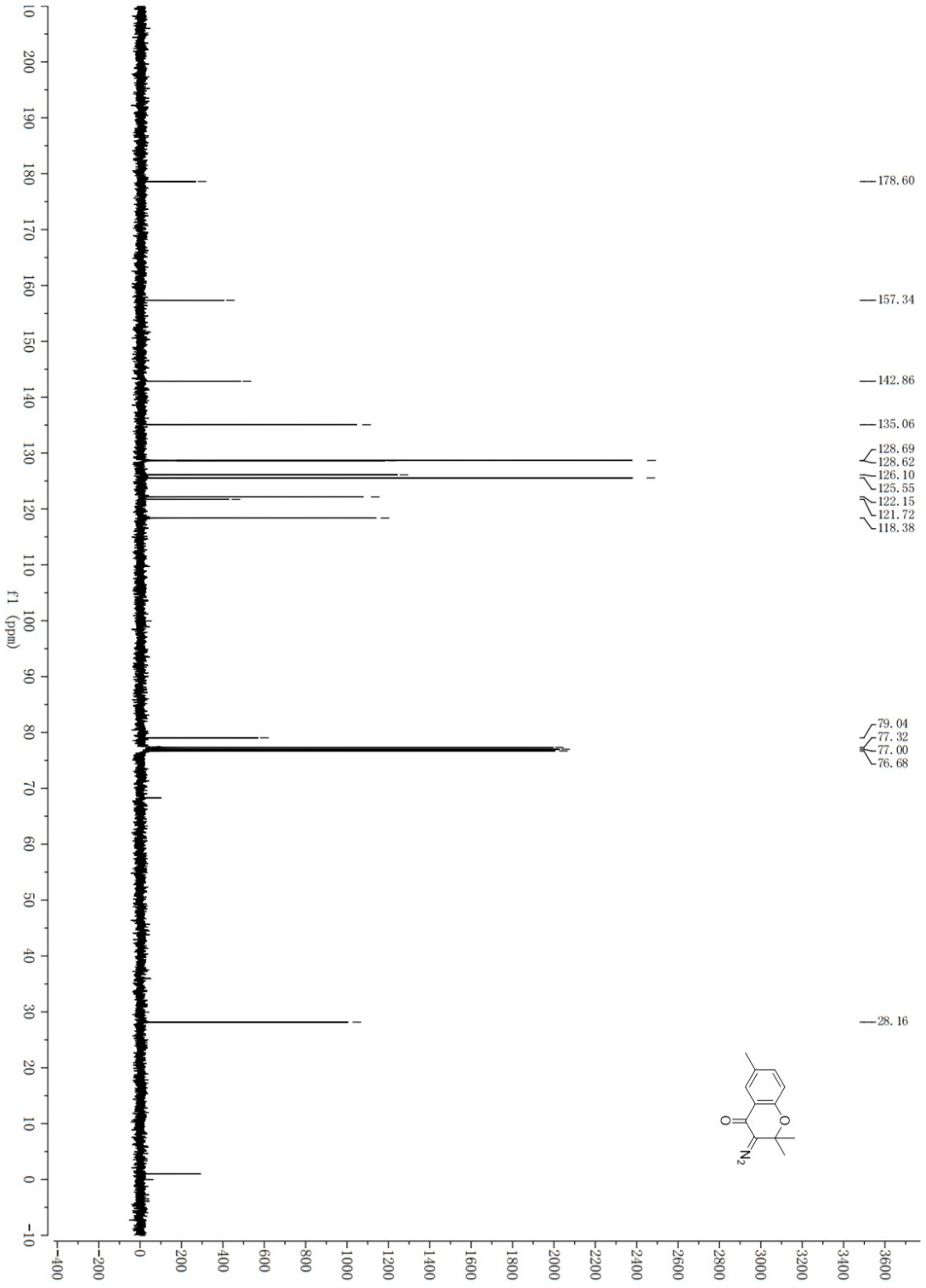

178.60
157.34
142.86
135.06
128.69
128.62
126.10
125.55
122.15
121.72
118.38
79.04
77.32
77.00
28.16
f1 (ppm)

(1i)

^{1}H NMR

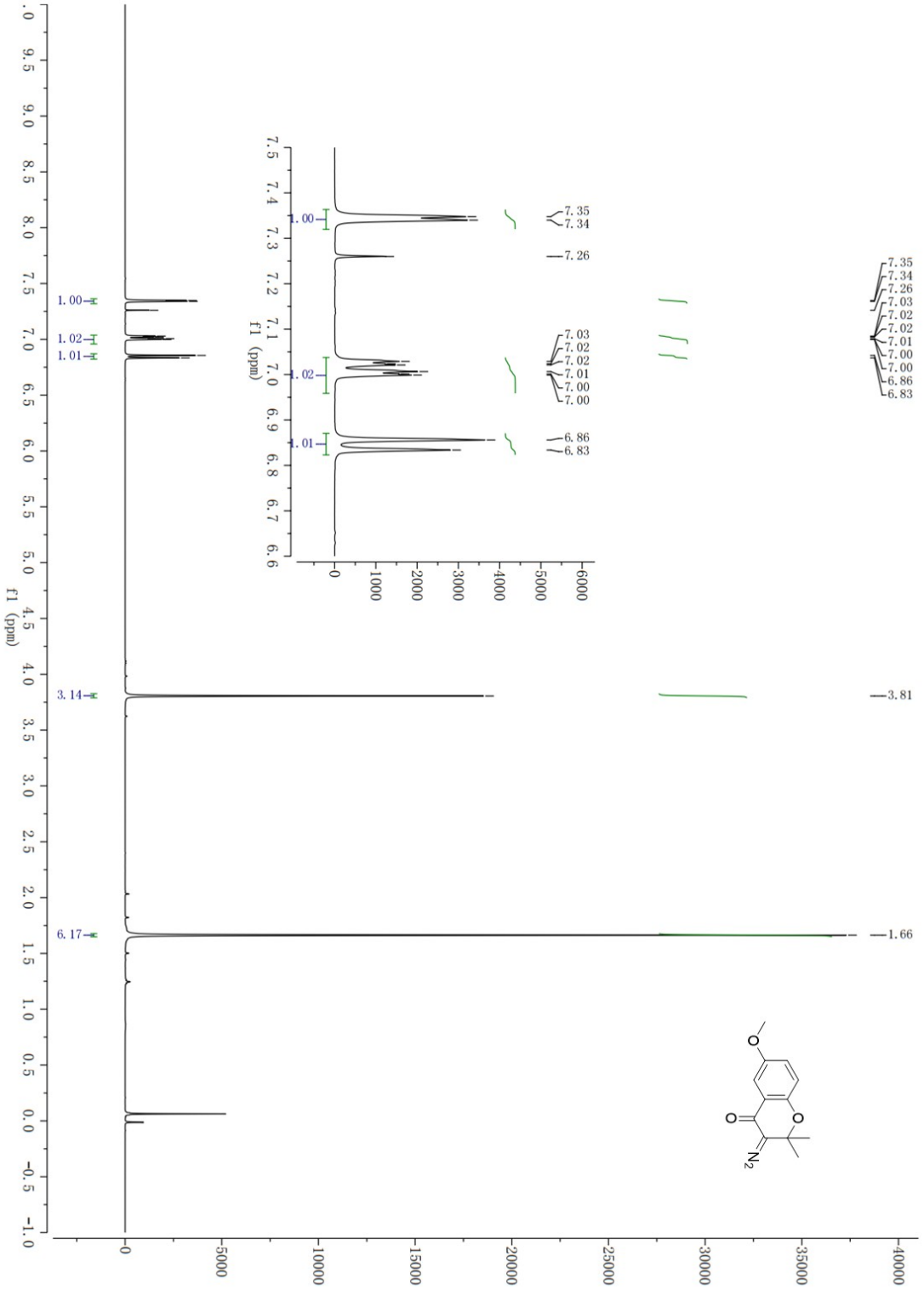

1.00
1.05
1.02
3.29
6.29
2.31
1.67
7.69
7.68
7.26
7.25
7.24
7.22
7.22
6.82
6.80
f1 (ppm)
O
O
N2

^{13}C NMR

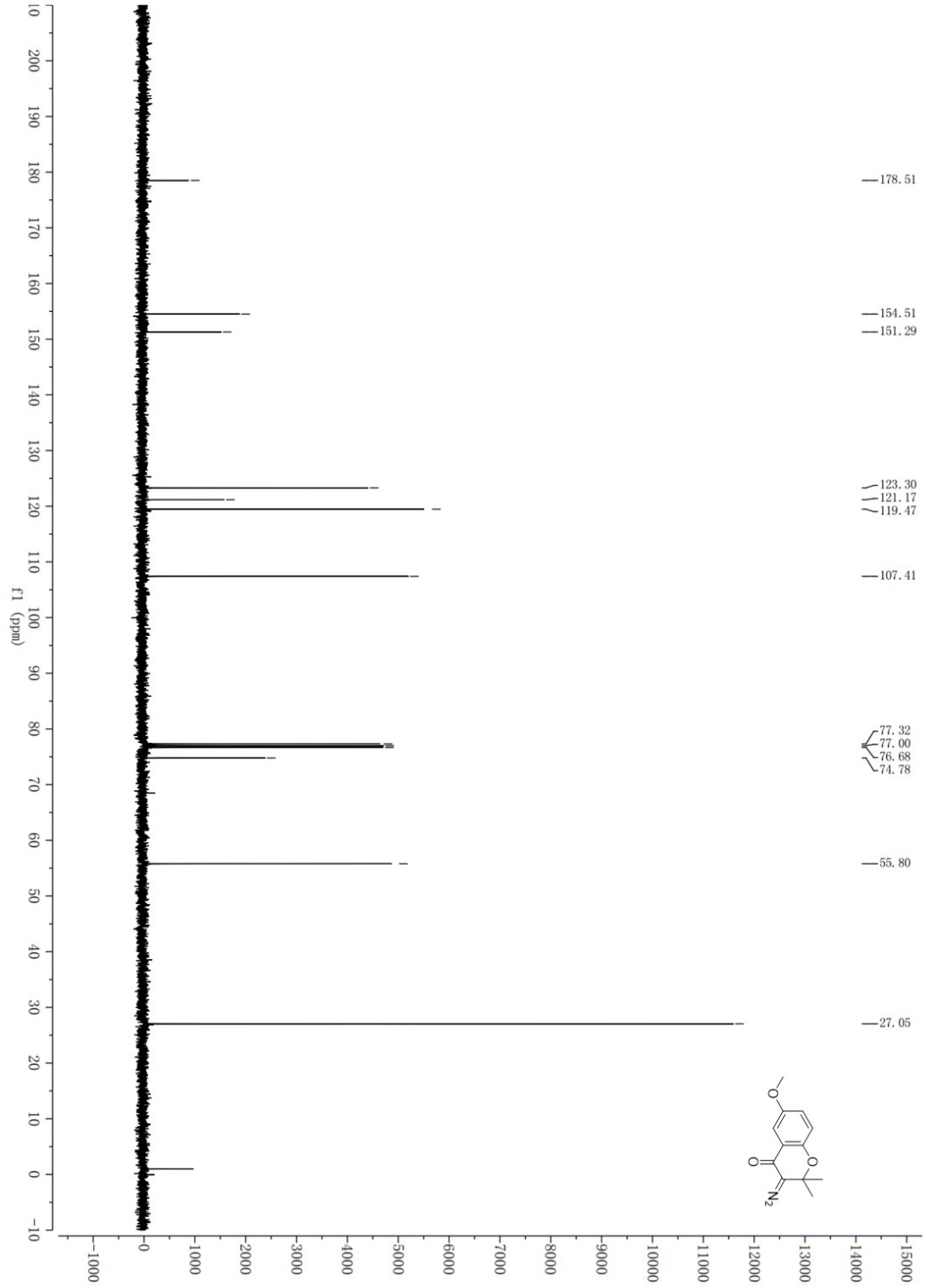

178.60
157.34
142.86
135.06
128.69
128.62
126.10
125.55
122.15
121.72
118.38
79.04
77.32
77.00
76.68
28.16
f1 (ppm)

(1j)

^{1}H NMR

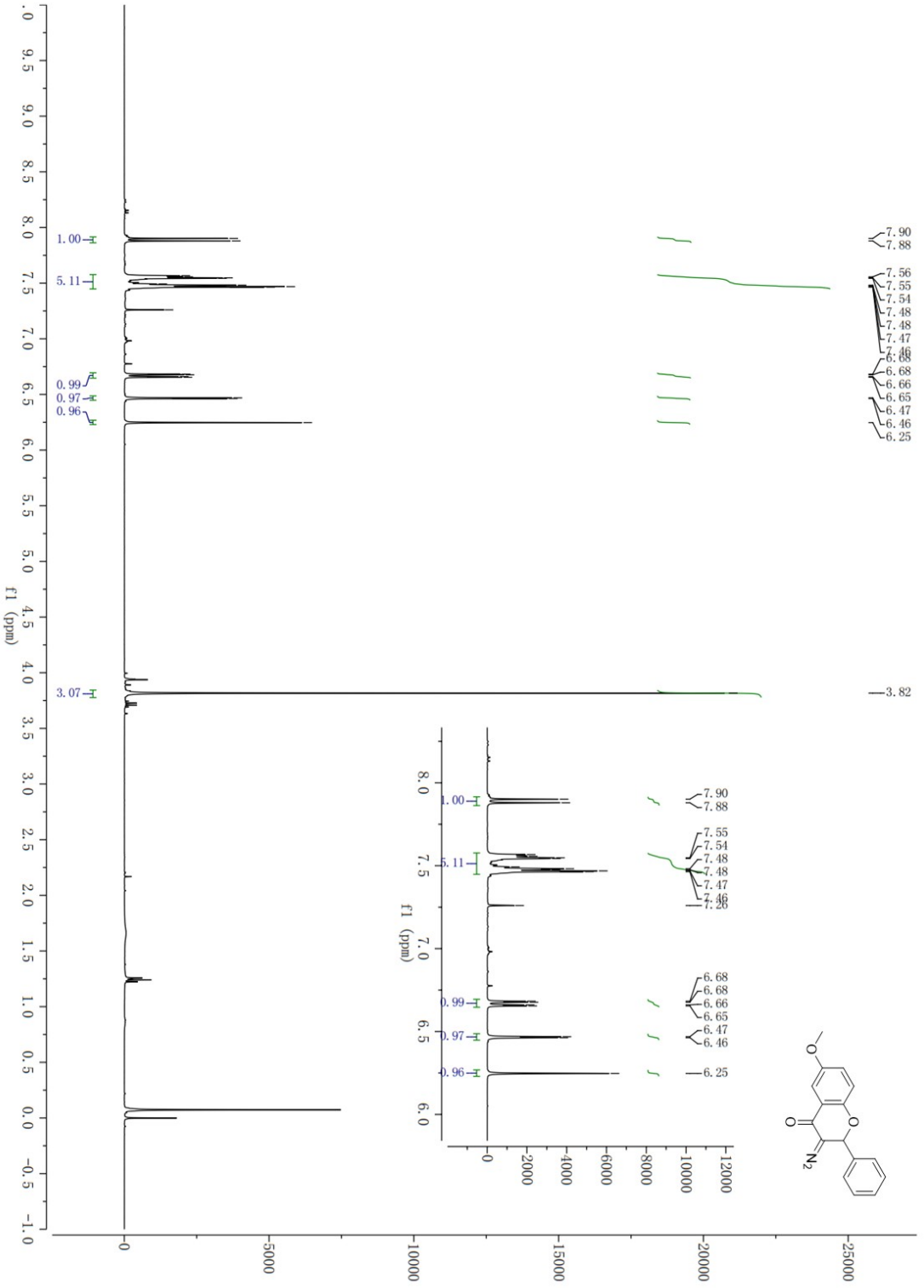

f1 (ppm)
1.00
1.02
1.01
3.14
6.17
7.35
7.34
7.26
7.03
7.02
7.02
7.01
7.00
7.00
6.86
6.83
3.81
1.66
O
O
O
N2

^{13}C NMR

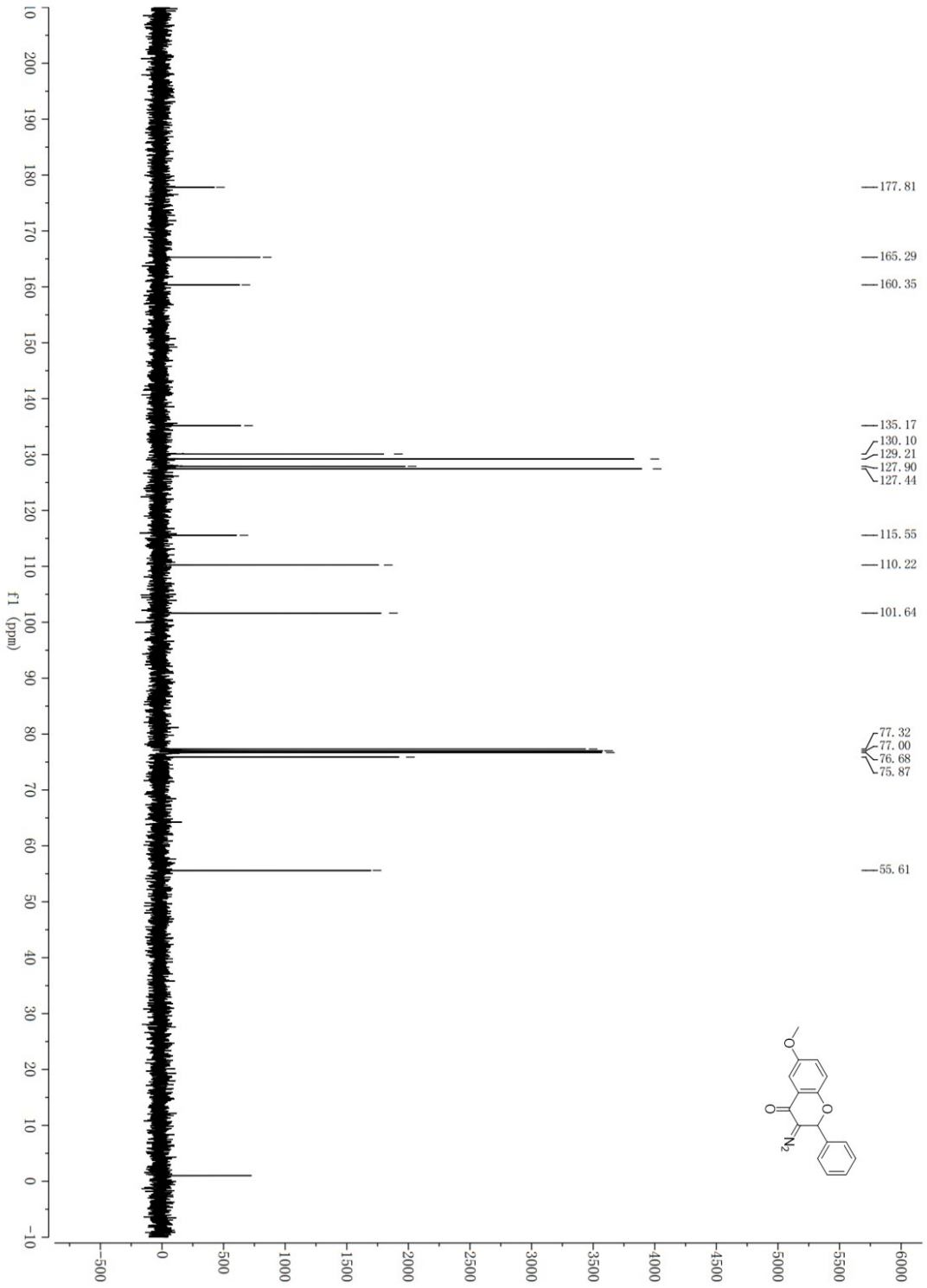

178.51
154.51
151.29
123.30
121.17
119.47
107.41
77.32
77.00
76.68
74.78
55.80
27.05
f1 (ppm)

(1k)

^{1}H NMR

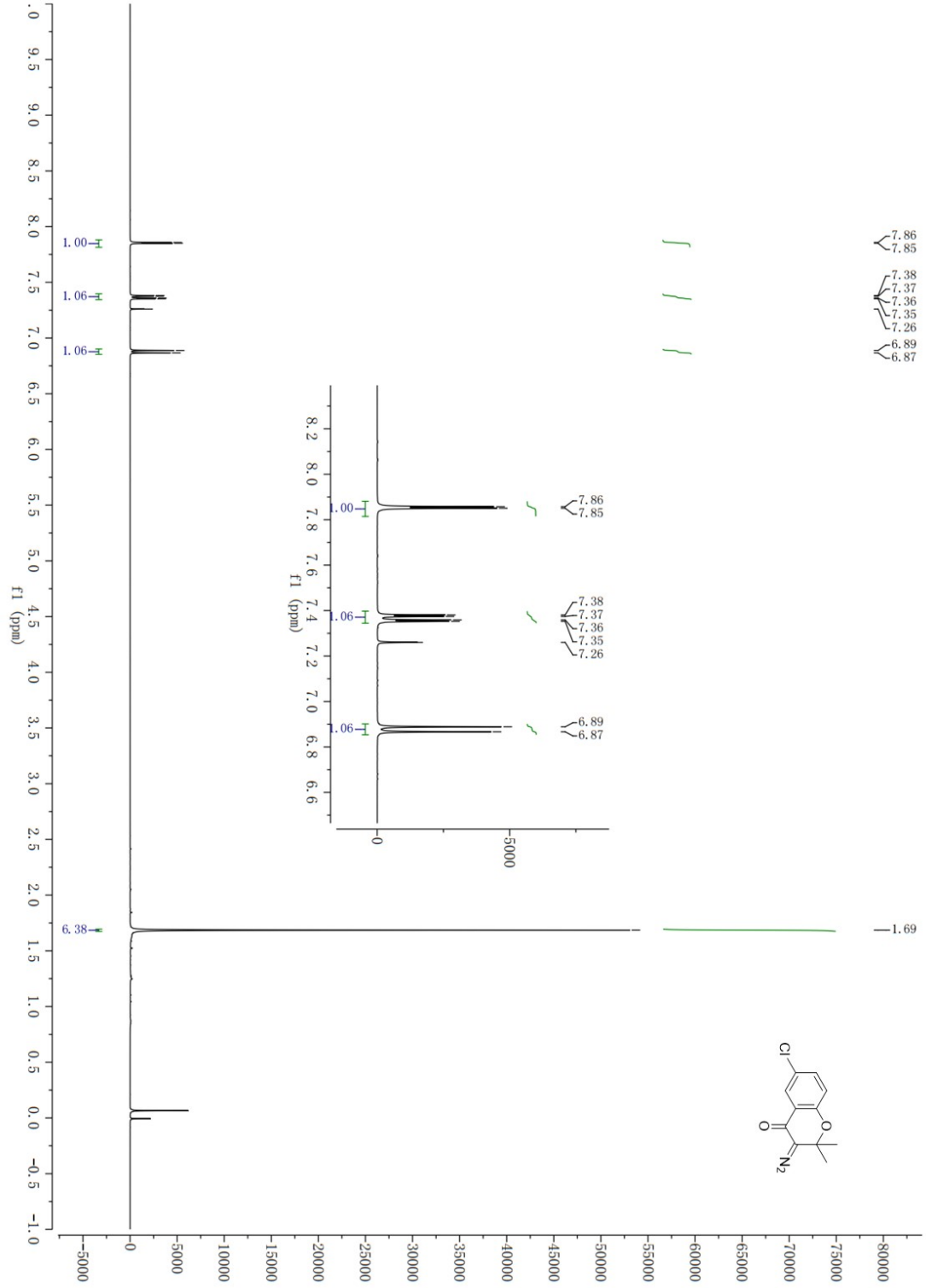

1.00
5.11
0.99
0.97
0.96
3.07
7.90
7.88
7.56
7.55
7.54
7.48
7.48
7.47
7.46
6.68
6.68
6.66
6.65
6.47
6.46
6.25
3.82
f1 (ppm)
O
O
N2

^{13}C NMR

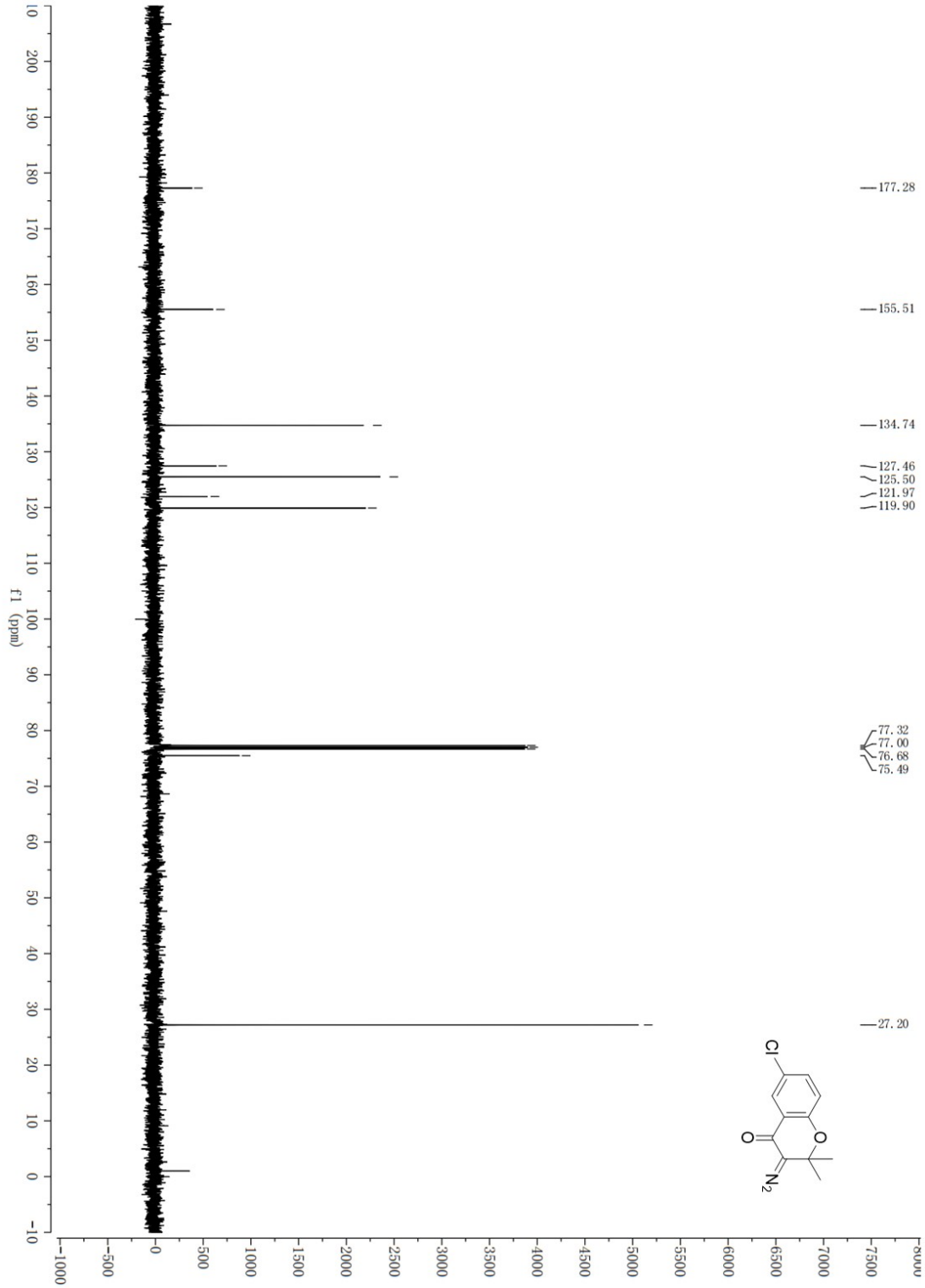

177.81
165.29
160.35
135.17
130.10
129.21
127.90
127.44
115.55
110.22
101.64
77.32
77.00
76.68
75.87
55.61
f1 (ppm)

(1l)

^{1}H NMR

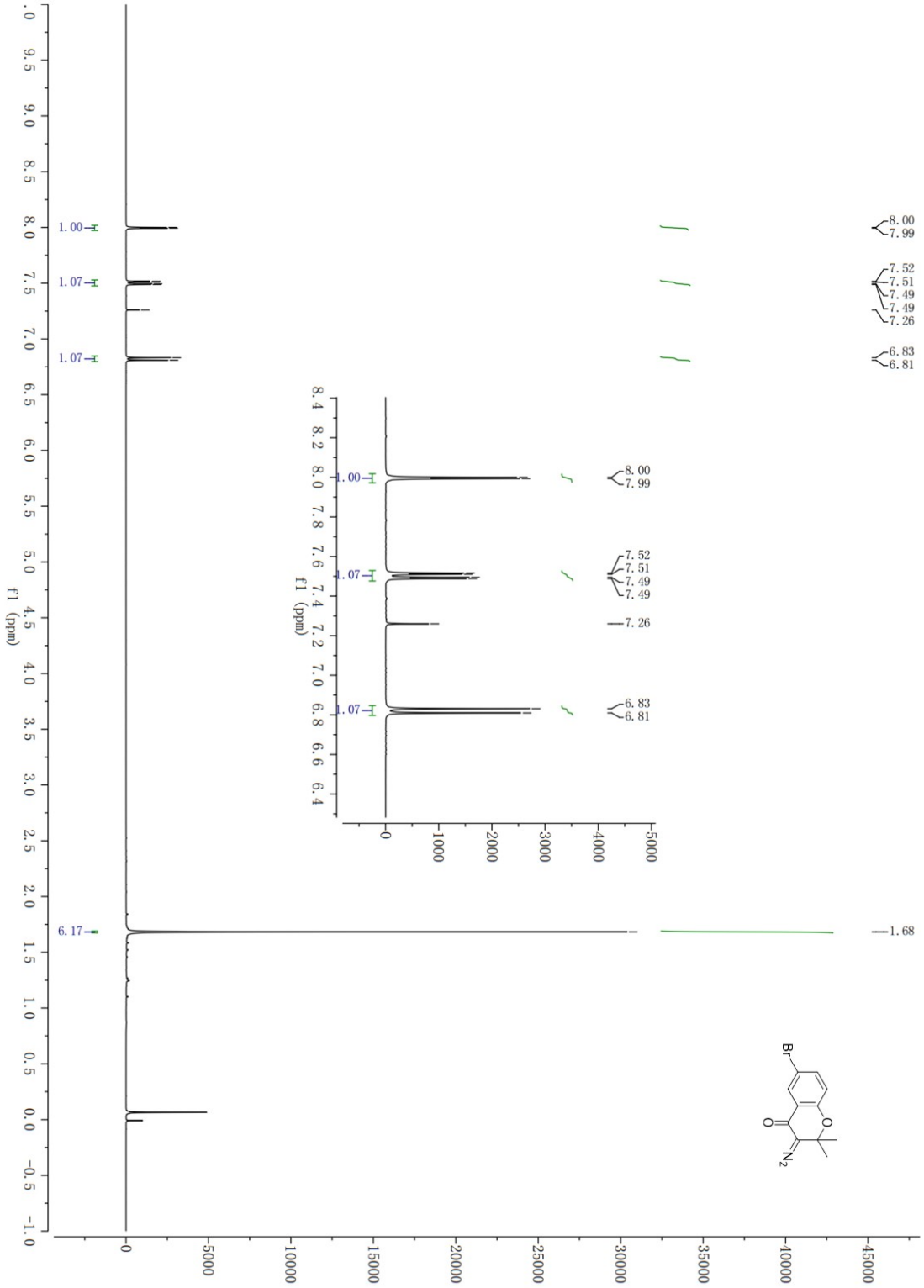

1.00
1.06
1.06
6.38
1.69
7.86
7.85
7.38
7.37
7.36
7.35
7.26
6.89
6.87
f1 (ppm)
Cl
O
O
N2

^{13}C NMR

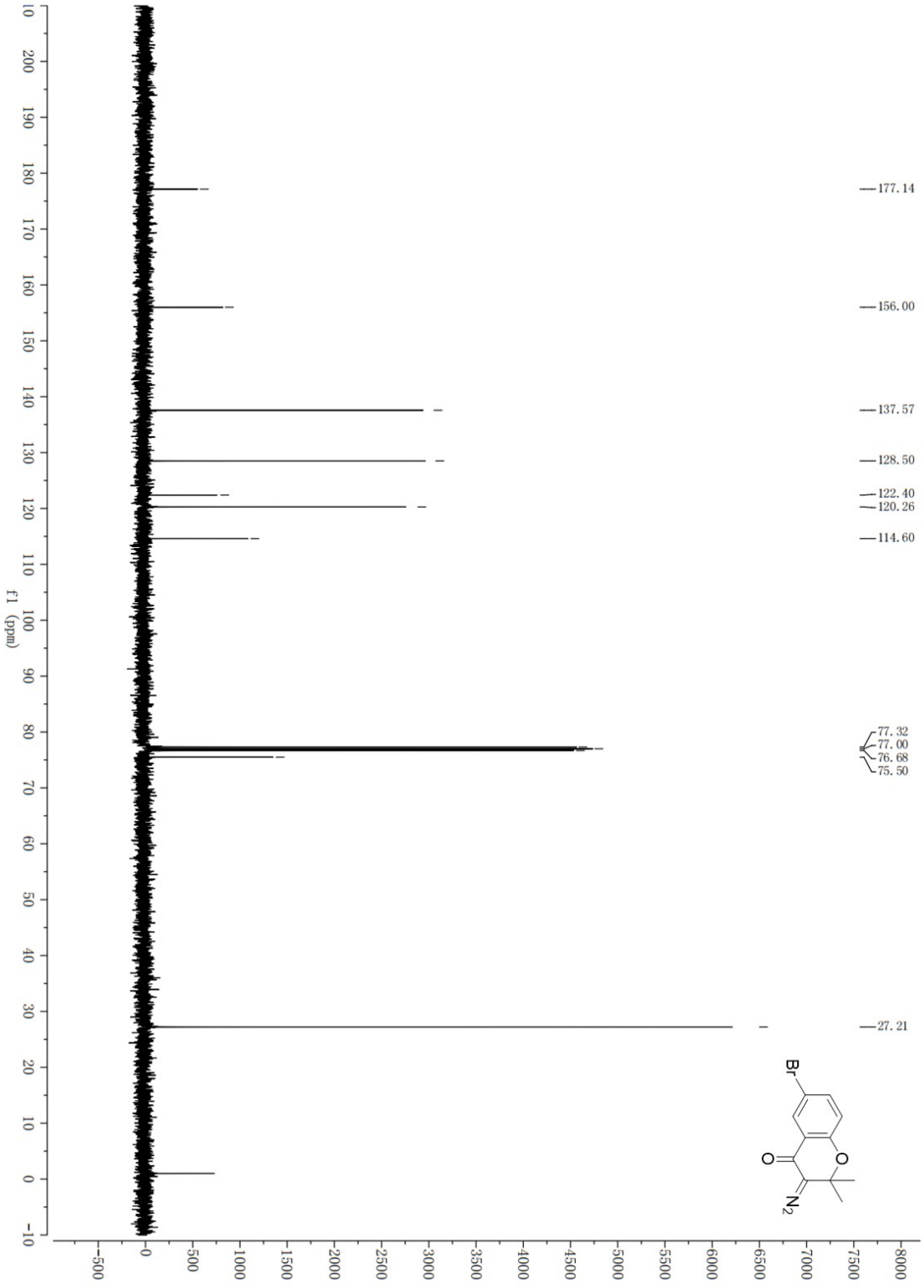

177.28
155.51
134.74
127.46
125.50
121.97
119.90
77.32
77.00
76.68
75.49
27.20
f1 (ppm)
Cl
O
O
N2

(1m)

^{1}H NMR

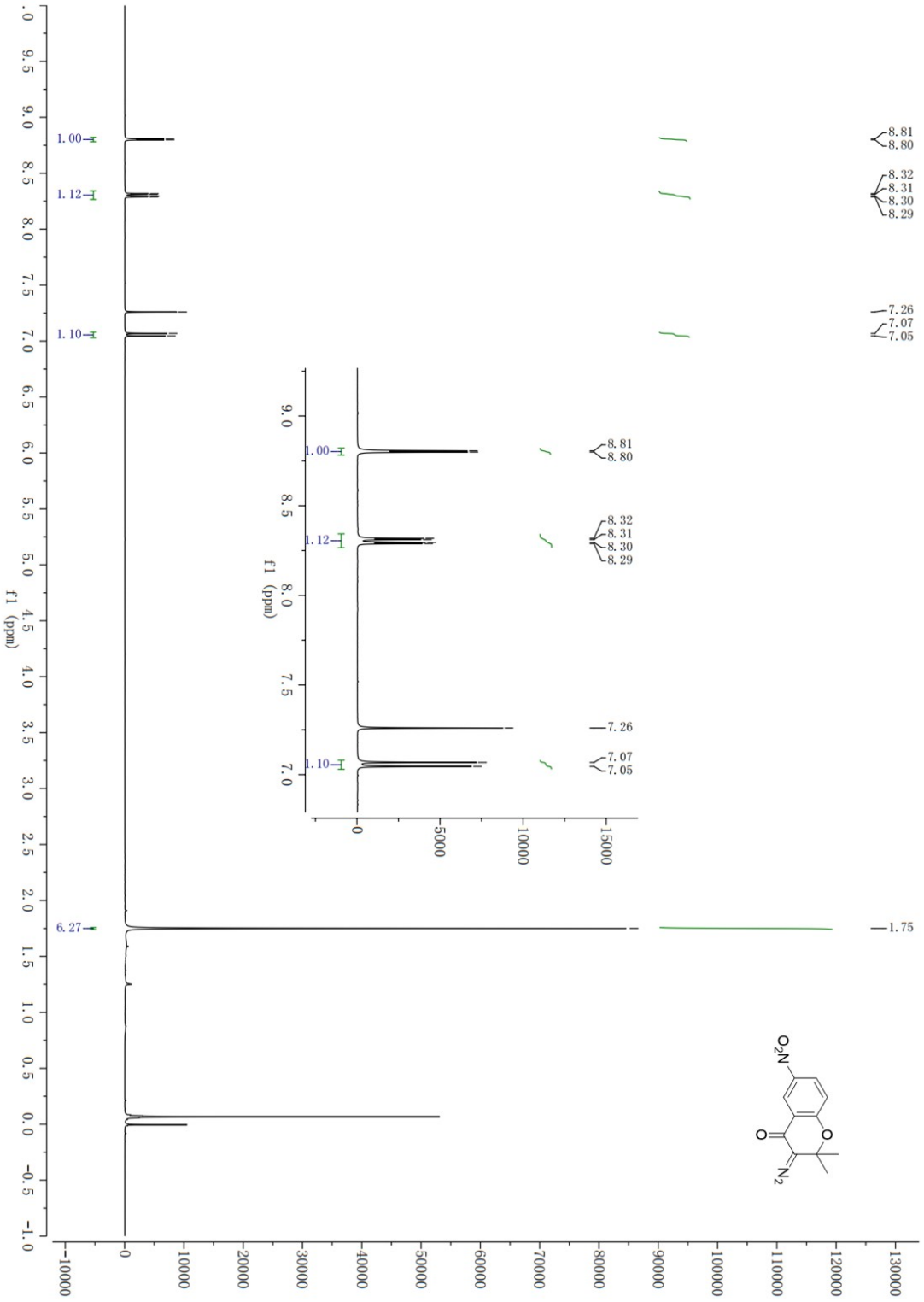

f1 (ppm)
1.00
1.07
1.07
6.17
8.00
7.99
7.52
7.51
7.49
7.49
7.26
6.83
6.81
1.68
Br
O
O
N2

^{13}C NMR

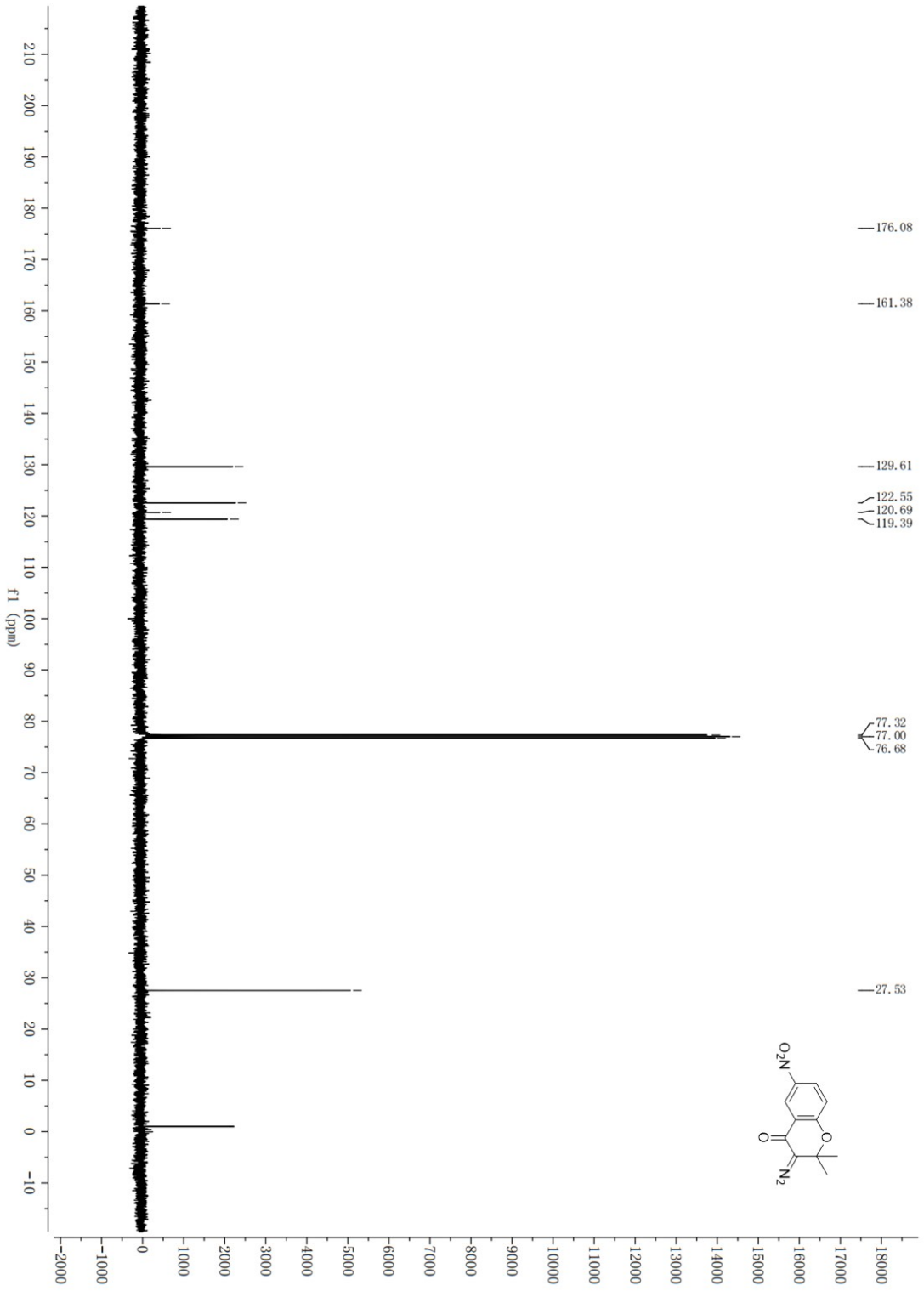

177.14
156.00
137.57
128.50
122.40
120.26
114.60
77.32
77.00
76.68
75.50
27.21
f1 (ppm)
Br
O
O
N2

(1n)

^{1}H NMR

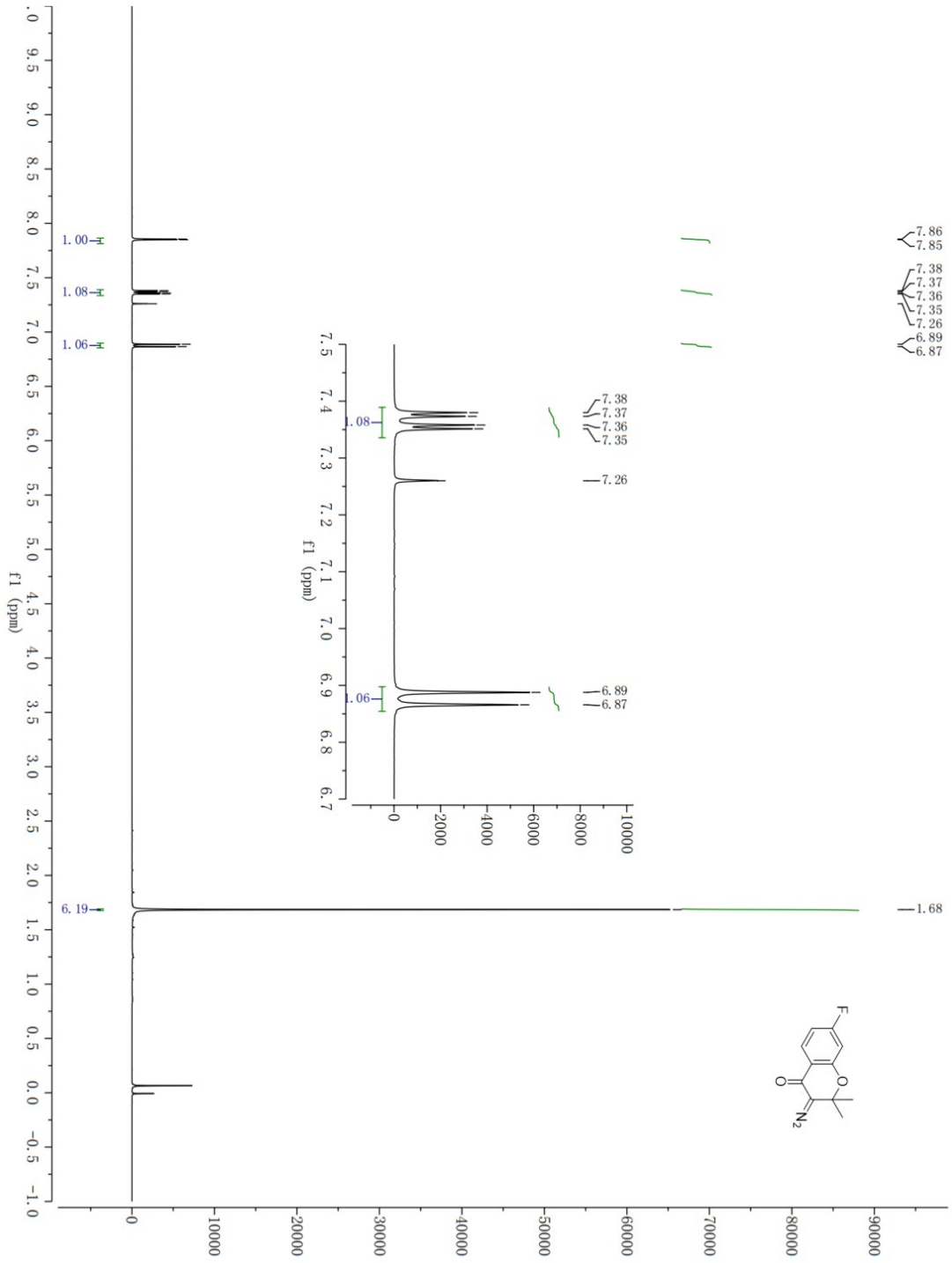


^{13}C NMR

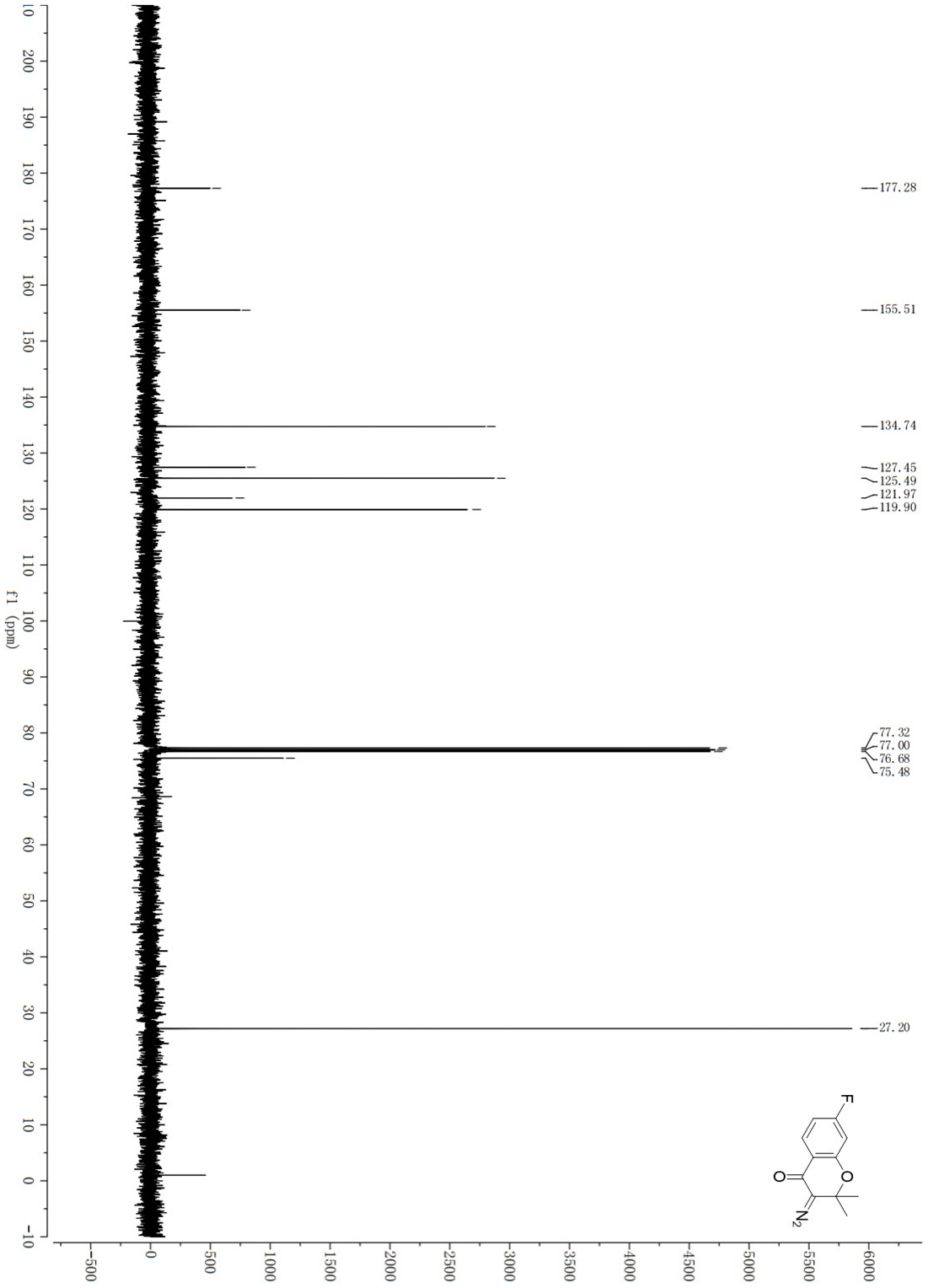

176.08
161.38
129.61
122.55
120.69
119.39
77.32
77.00
76.68
27.53
f1 (ppm)
O2N
O
O
N2

(1o)

^{1}H NMR

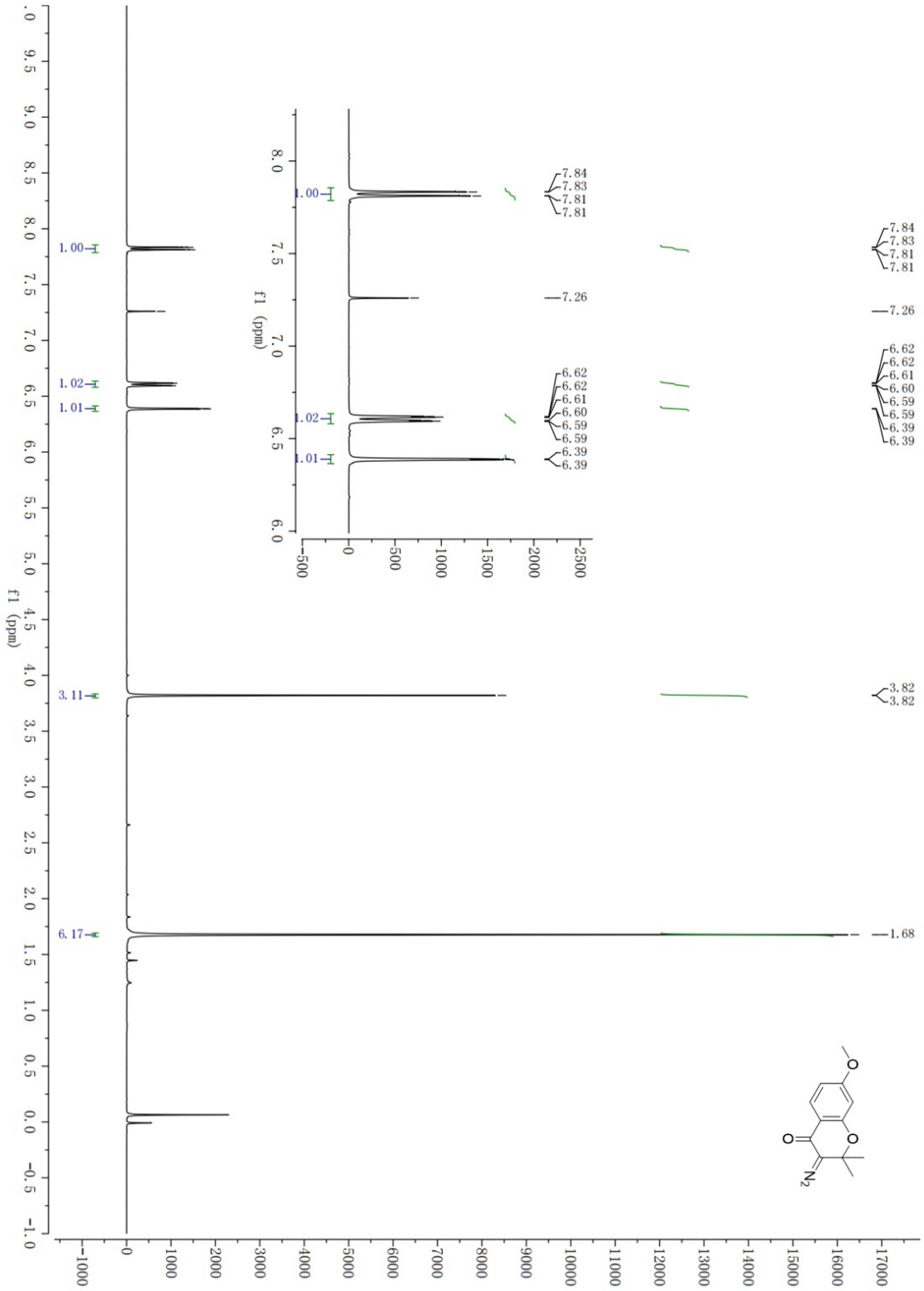

1.00
1.08
1.06
6.19
1.68
7.86
7.85
7.38
7.37
7.36
7.35
7.26
6.89
6.87
f1 (ppm)
F
O
O
N2

^{13}C NMR

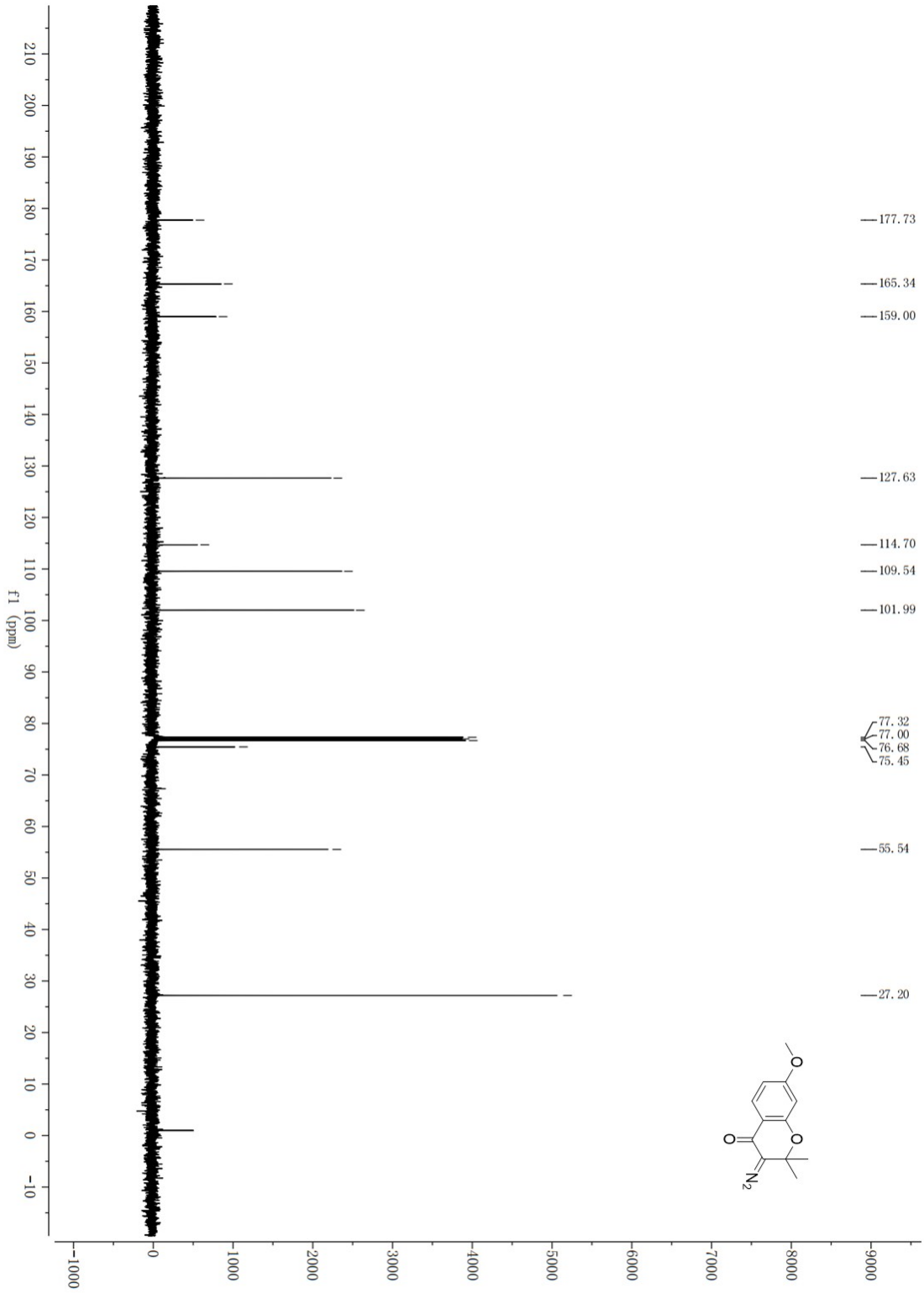

177.28
155.51
134.74
127.45
125.49
121.97
119.90
77.32
77.00
76.68
75.48
27.20
f1 (ppm)
F
O
O
N2

(1p)

^{1}H NMR

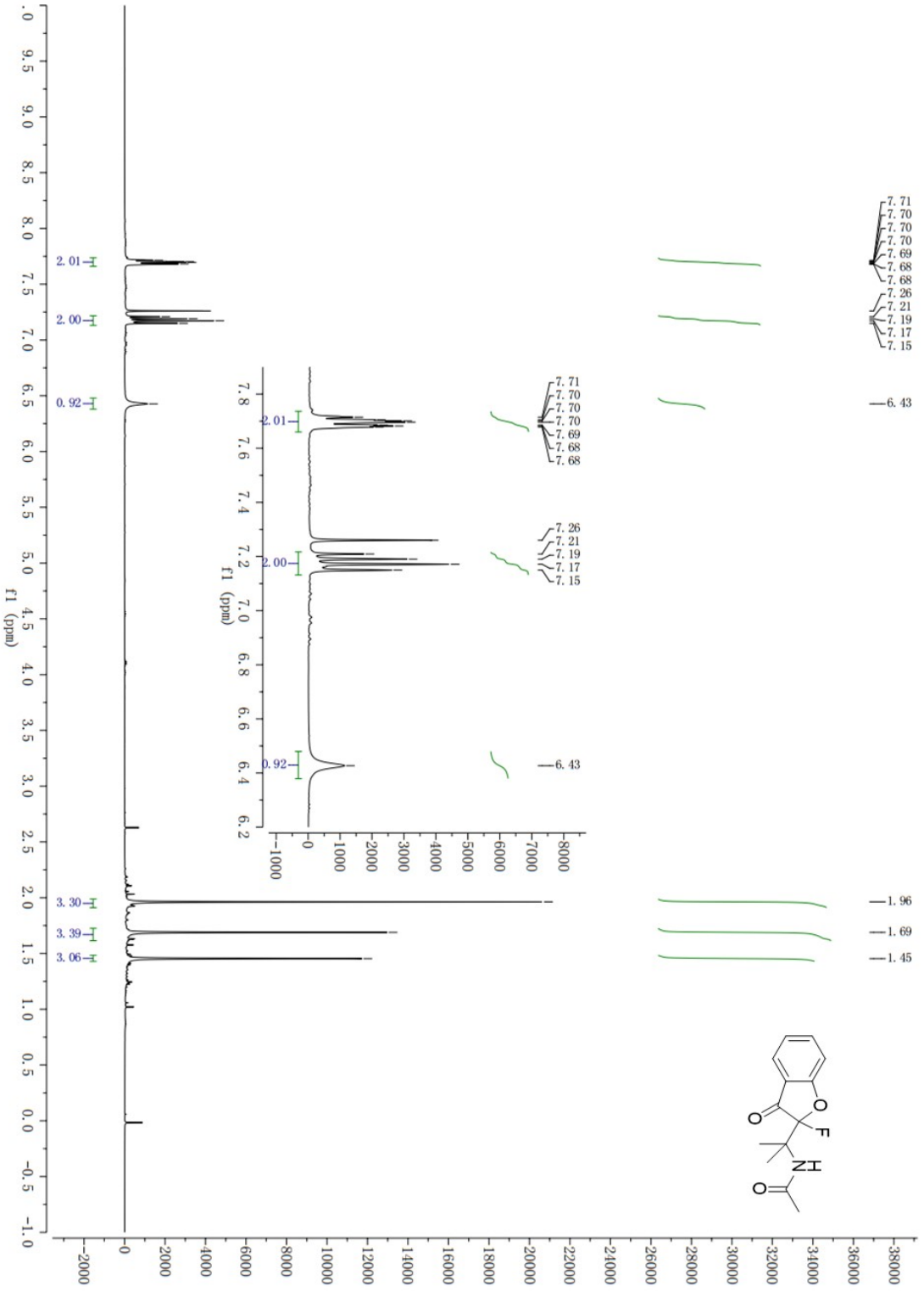

7.84
7.83
7.81
7.81
7.26
6.62
6.62
6.61
6.60
6.59
6.59
6.39
6.39
3.82
3.82
1.68
1.00
1.02
1.01
3.11
6.17
f1 (ppm)

^{13}C NMR

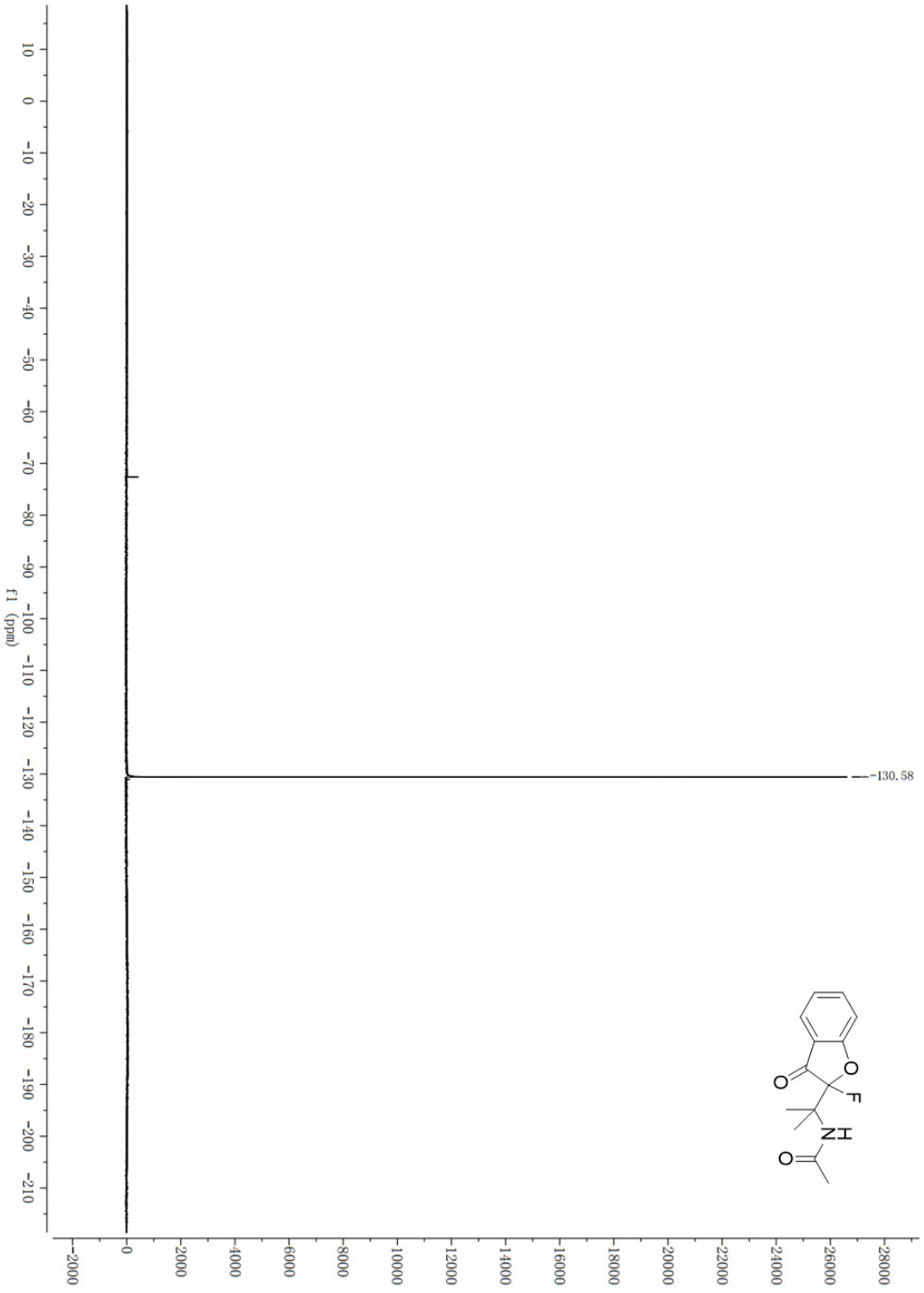

177.73
165.34
159.00
127.63
114.70
109.54
101.99
77.32
77.00
76.68
75.45
55.54
27.20
f1 (ppm)

(2a)

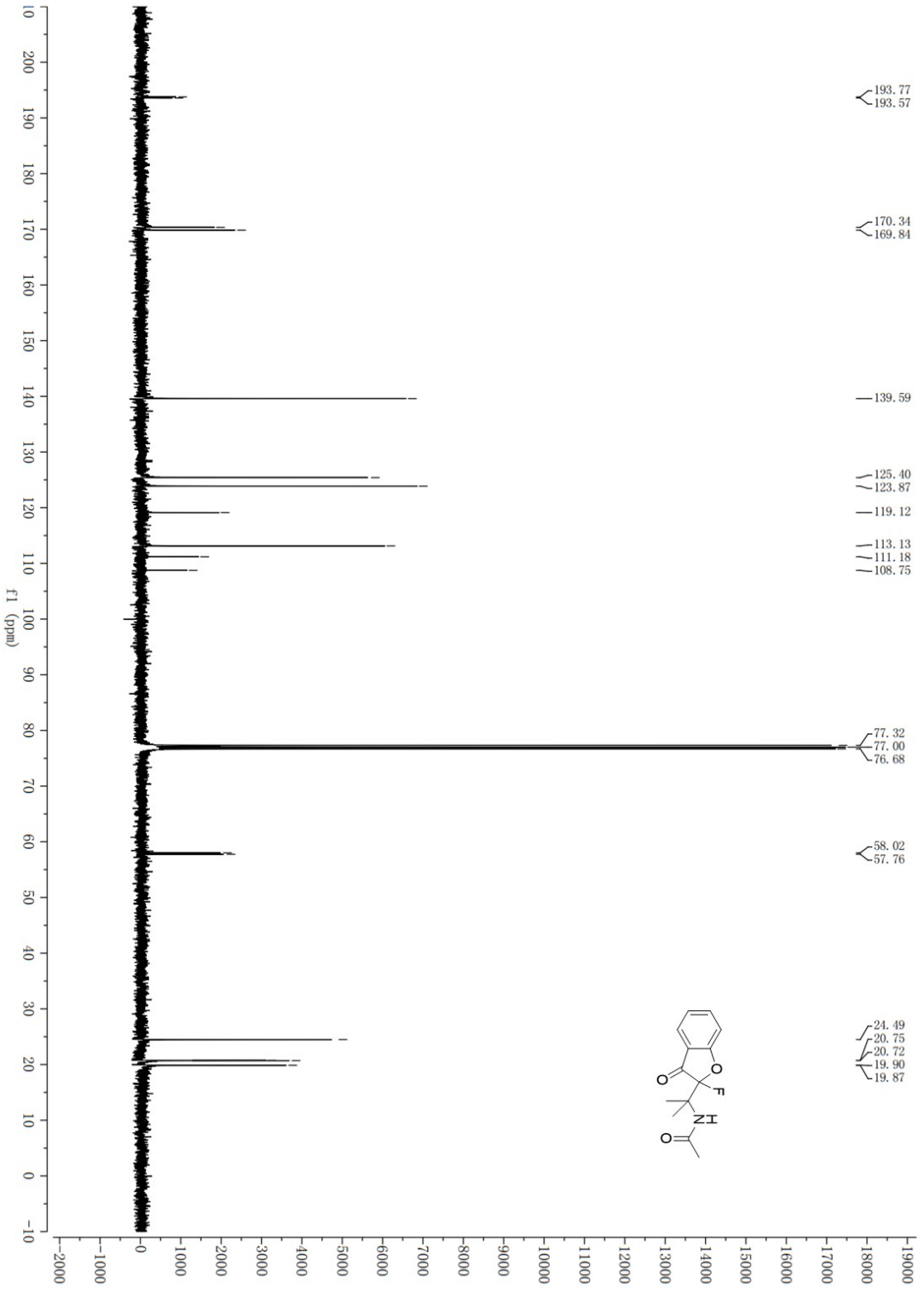
^{1}H NMR


f1 (ppm)
2.01
2.00
0.92
3.30
3.39
3.06
7.71
7.70
7.70
7.70
7.69
7.68
7.68
7.26
7.21
7.19
7.17
7.15
6.43
1.96
1.69
1.45
O
O
F
NH
O

^{19}F NMR

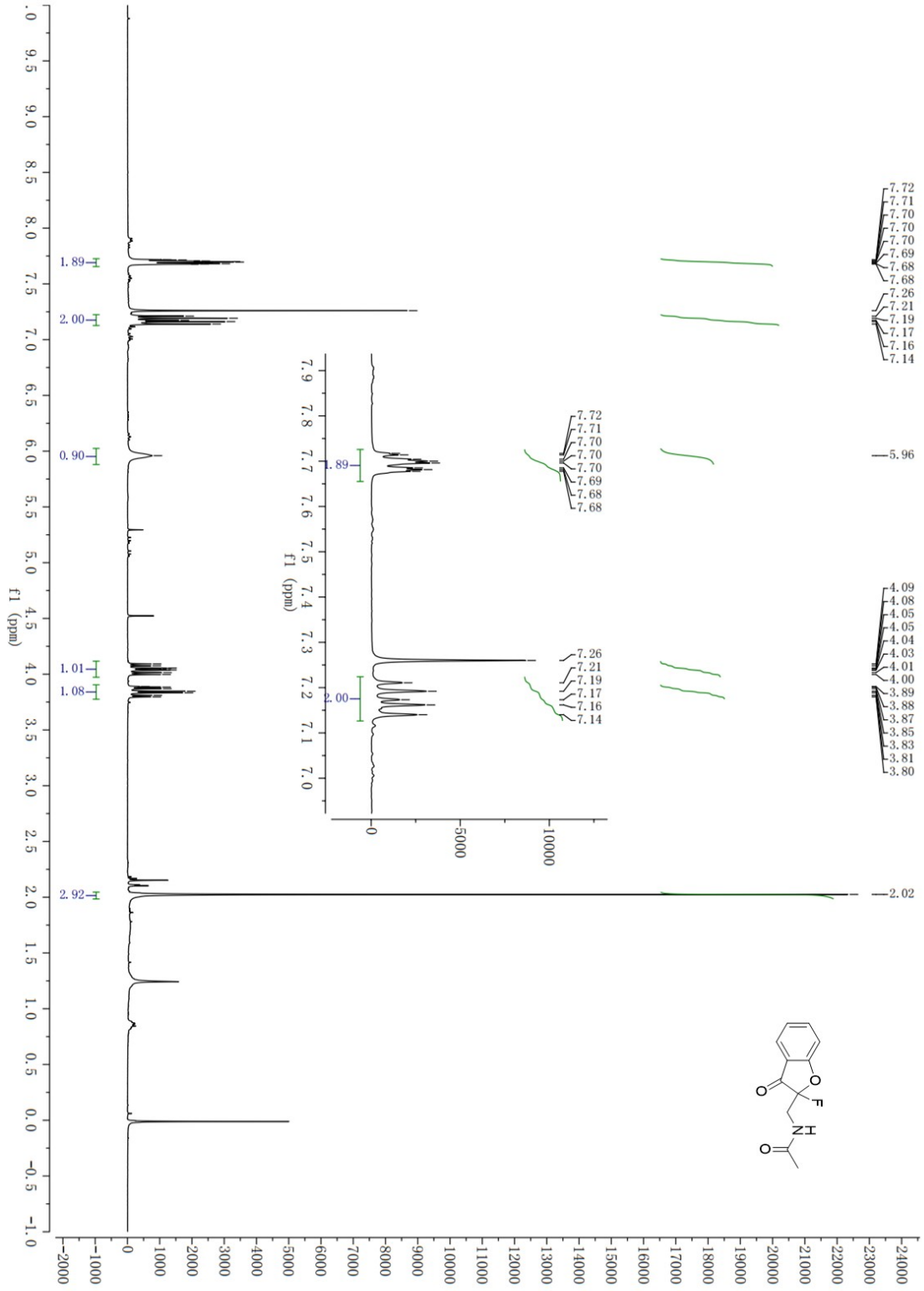

-130.58
f1 (ppm)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
-2000 0 2000 4000 6000 8000 10000 12000 14000 16000 18000 20000 22000 24000 26000 28000
O
F
O
N
H
O

^{13}C NMR

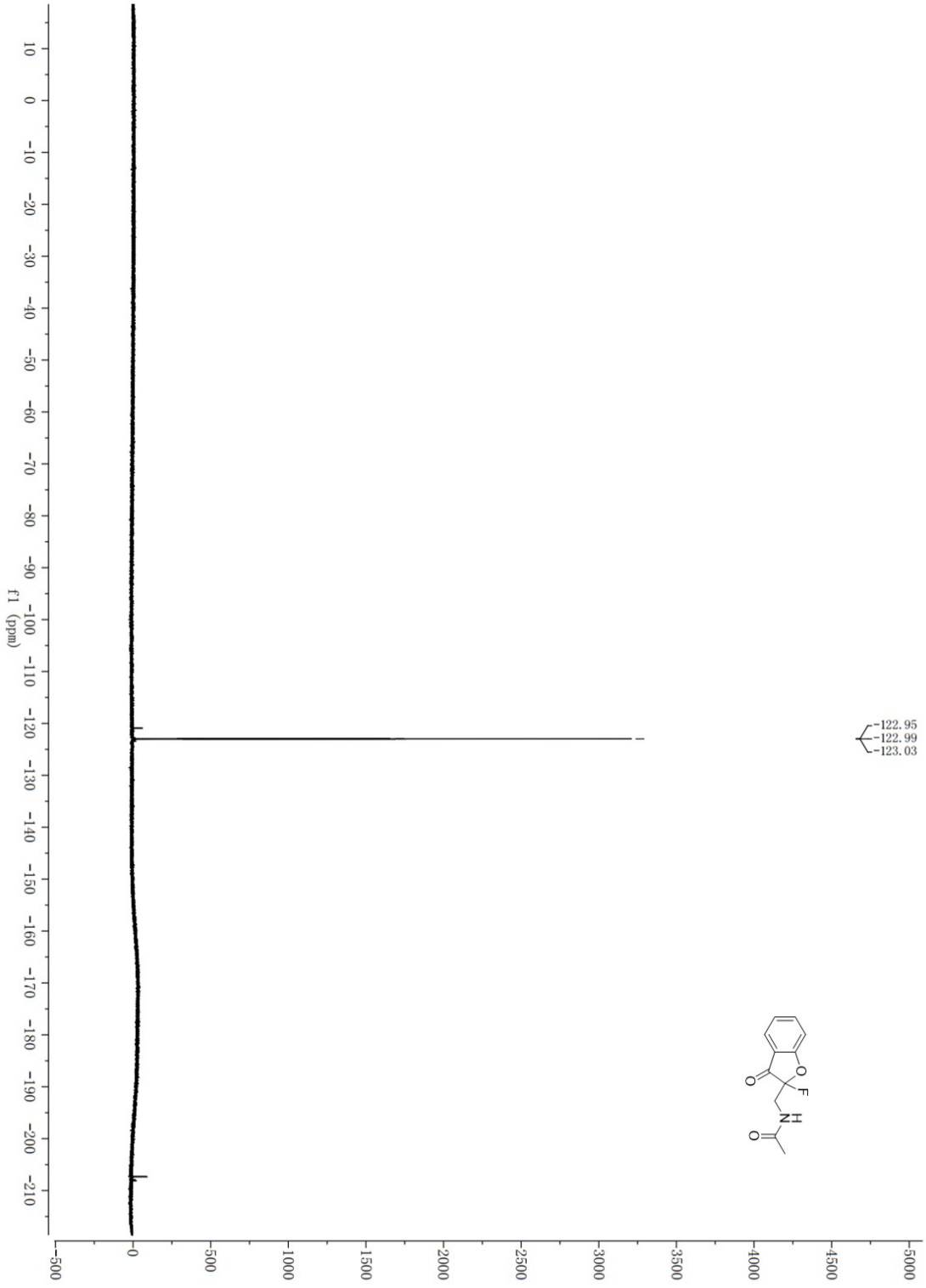

193.77
193.57
170.34
169.84
139.59
125.40
123.87
119.12
113.13
111.18
108.75
77.32
77.00
76.68
58.02
57.76
24.49
20.75
20.72
19.90
19.87
f1 (ppm)

(2b)

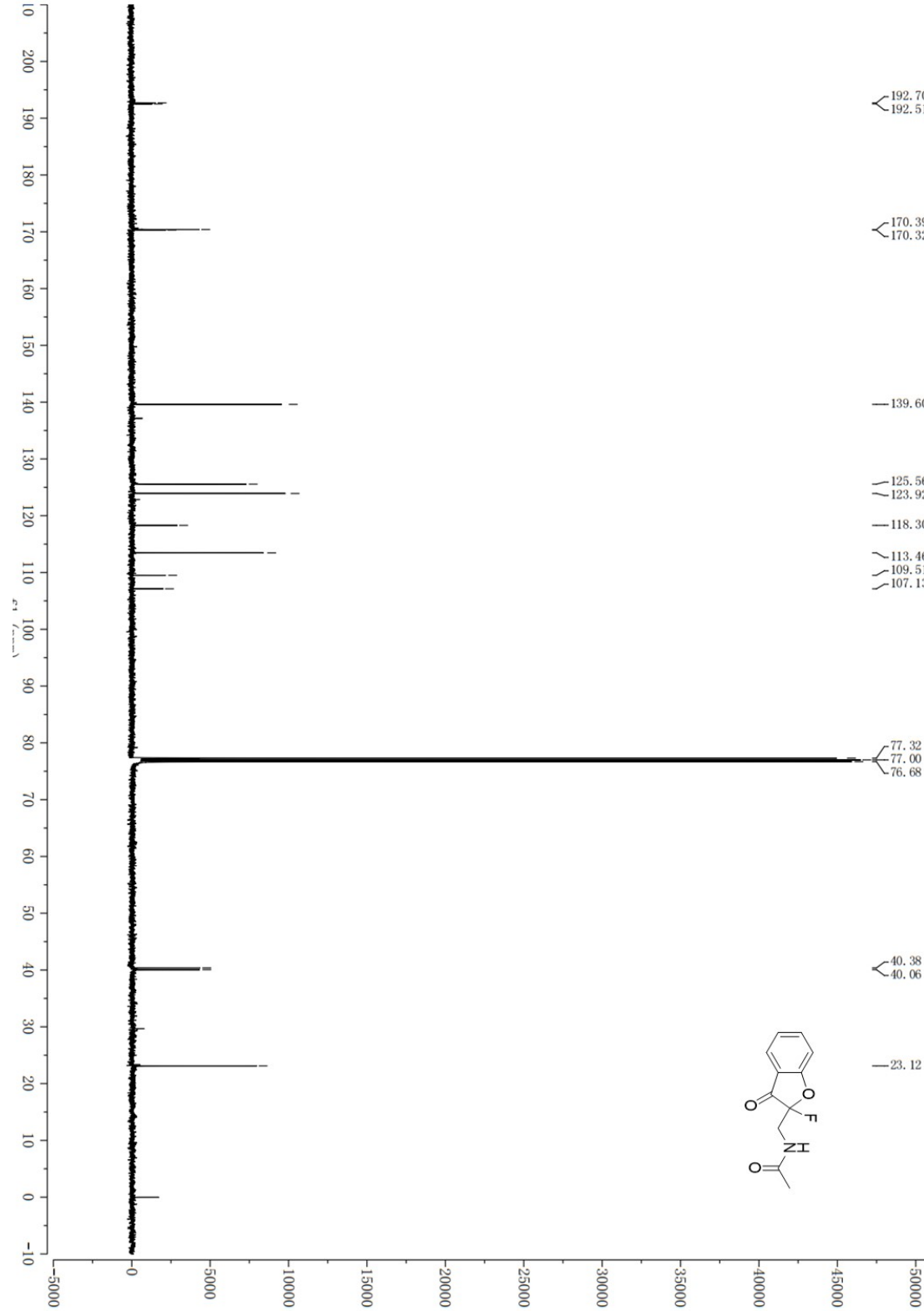
^{1}H NMR


f1 (ppm)
1.89
2.00
0.90
1.01
1.08
2.92
7.72
7.71
7.70
7.70
7.70
7.69
7.68
7.68
7.26
7.21
7.19
7.17
7.16
7.14
5.96
4.09
4.08
4.05
4.05
4.04
4.03
4.01
4.00
3.89
3.88
3.87
3.85
3.83
3.81
3.80
2.02
O
O
F
NH
O

^{19}F NMR

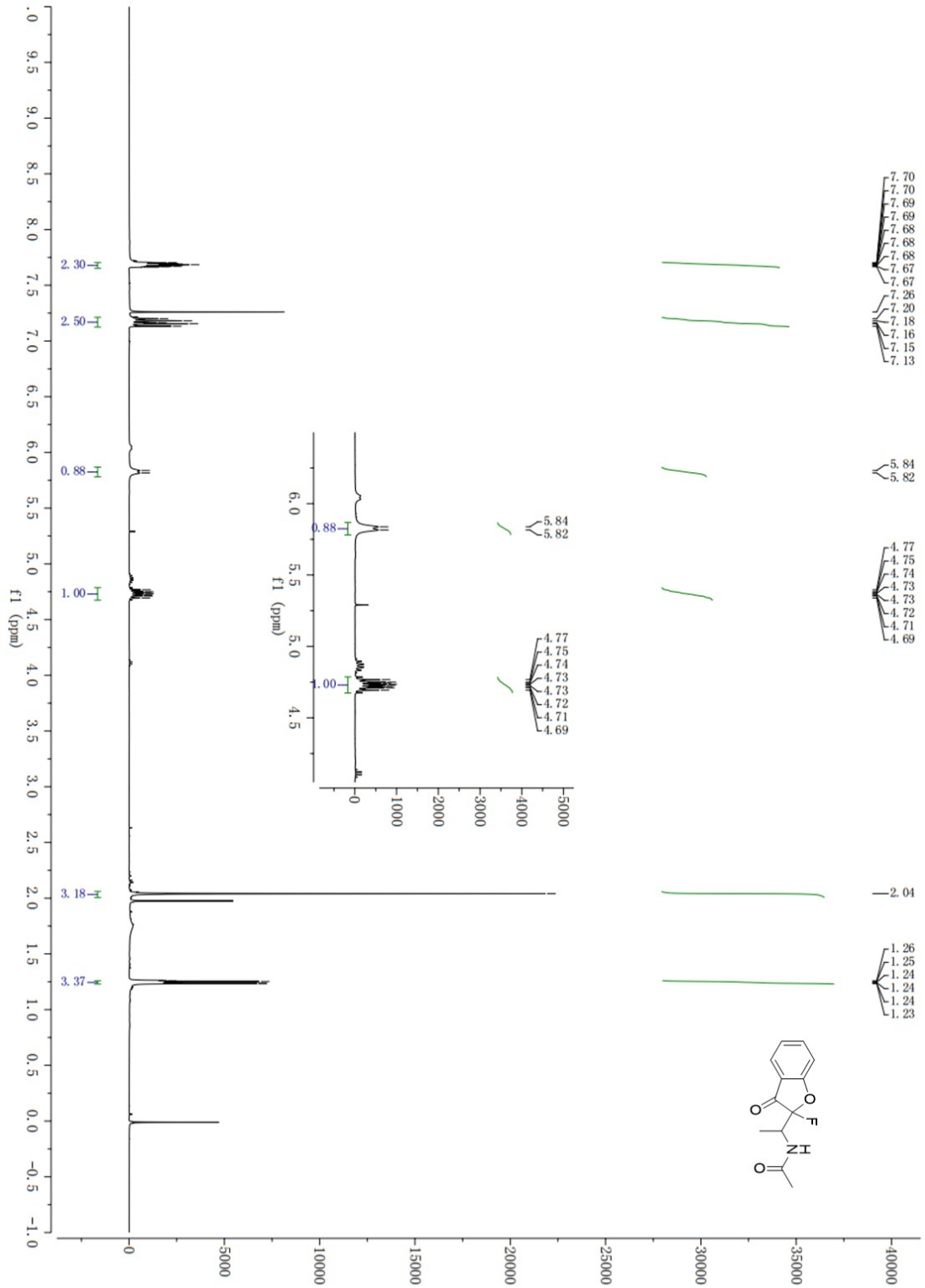


^{13}C NMR

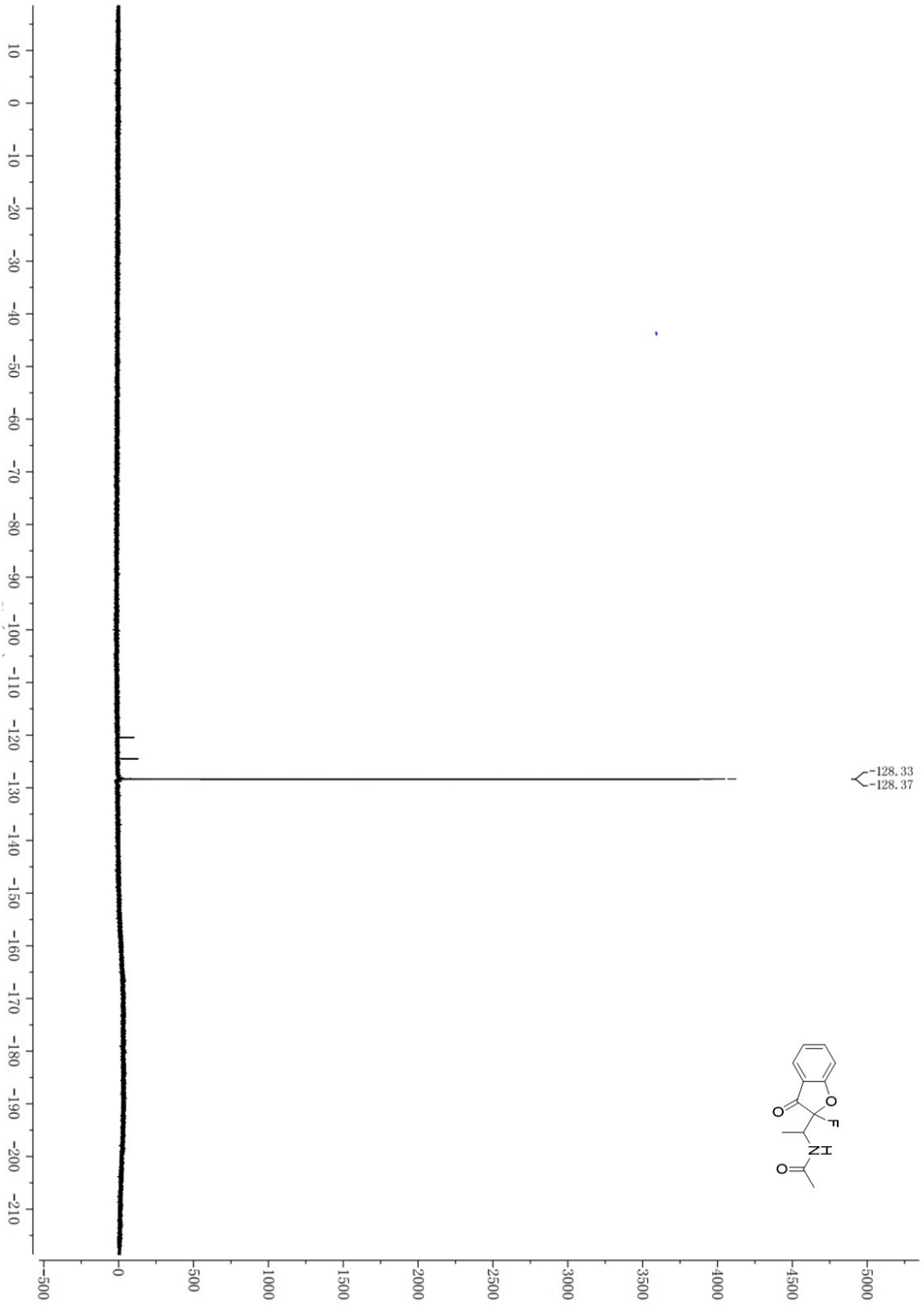

192.70
192.51
170.39
170.32
139.66
125.56
123.92
118.30
113.46
109.51
107.13
77.32
77.00
76.68
40.38
40.06
23.12
O
F
NH
O

(2c)

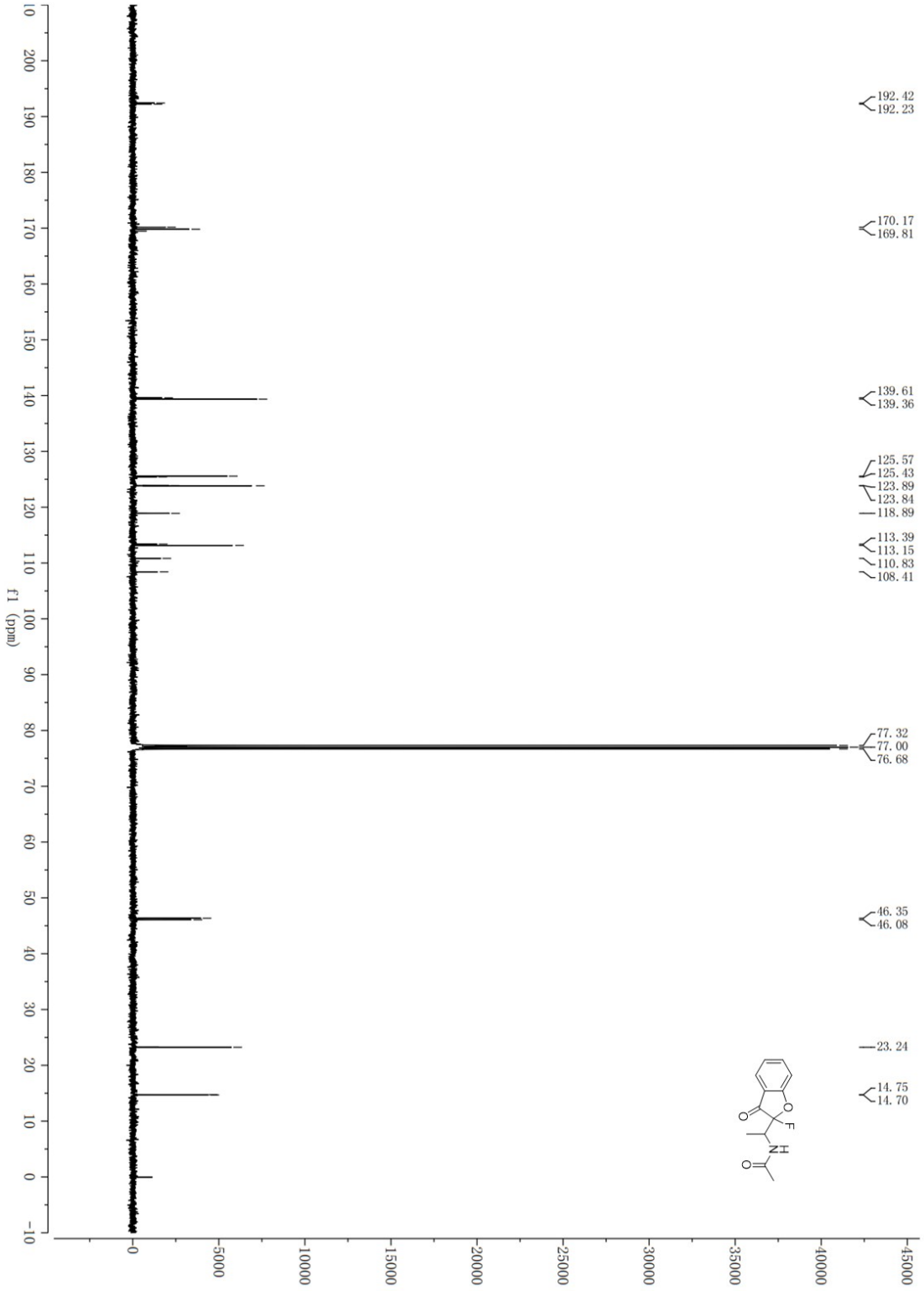
^{1}H NMR


f1 (ppm)
2.30
2.50
0.88
1.00
3.18
3.37
7.70
7.70
7.69
7.69
7.68
7.68
7.68
7.67
7.67
7.26
7.20
7.18
7.16
7.15
7.13
5.84
5.82
4.77
4.75
4.74
4.73
4.73
4.72
4.71
4.69
2.04
1.26
1.25
1.24
1.24
1.24
1.23
O
O
F
NH
O

^{19}F NMR

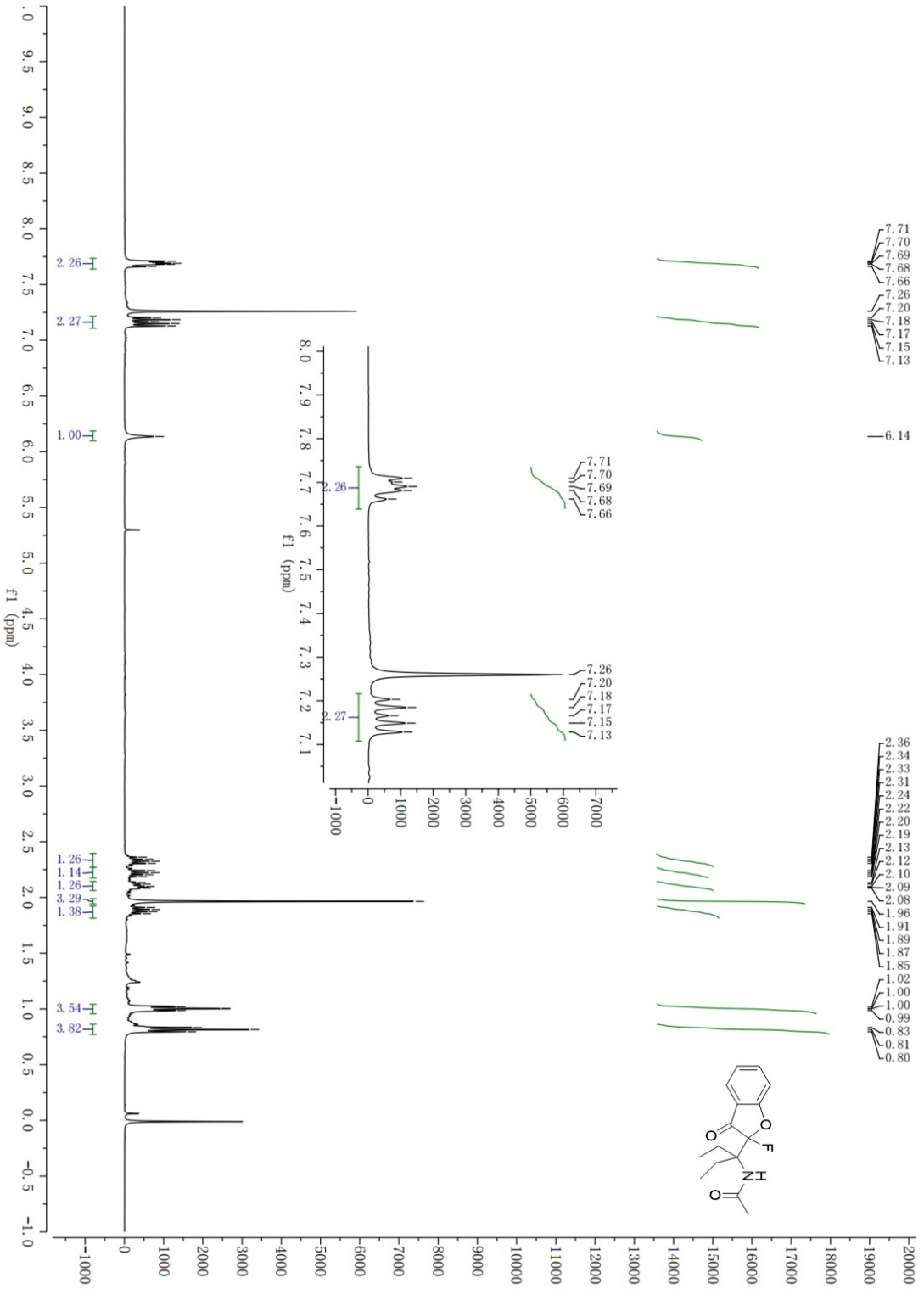


^{13}C NMR

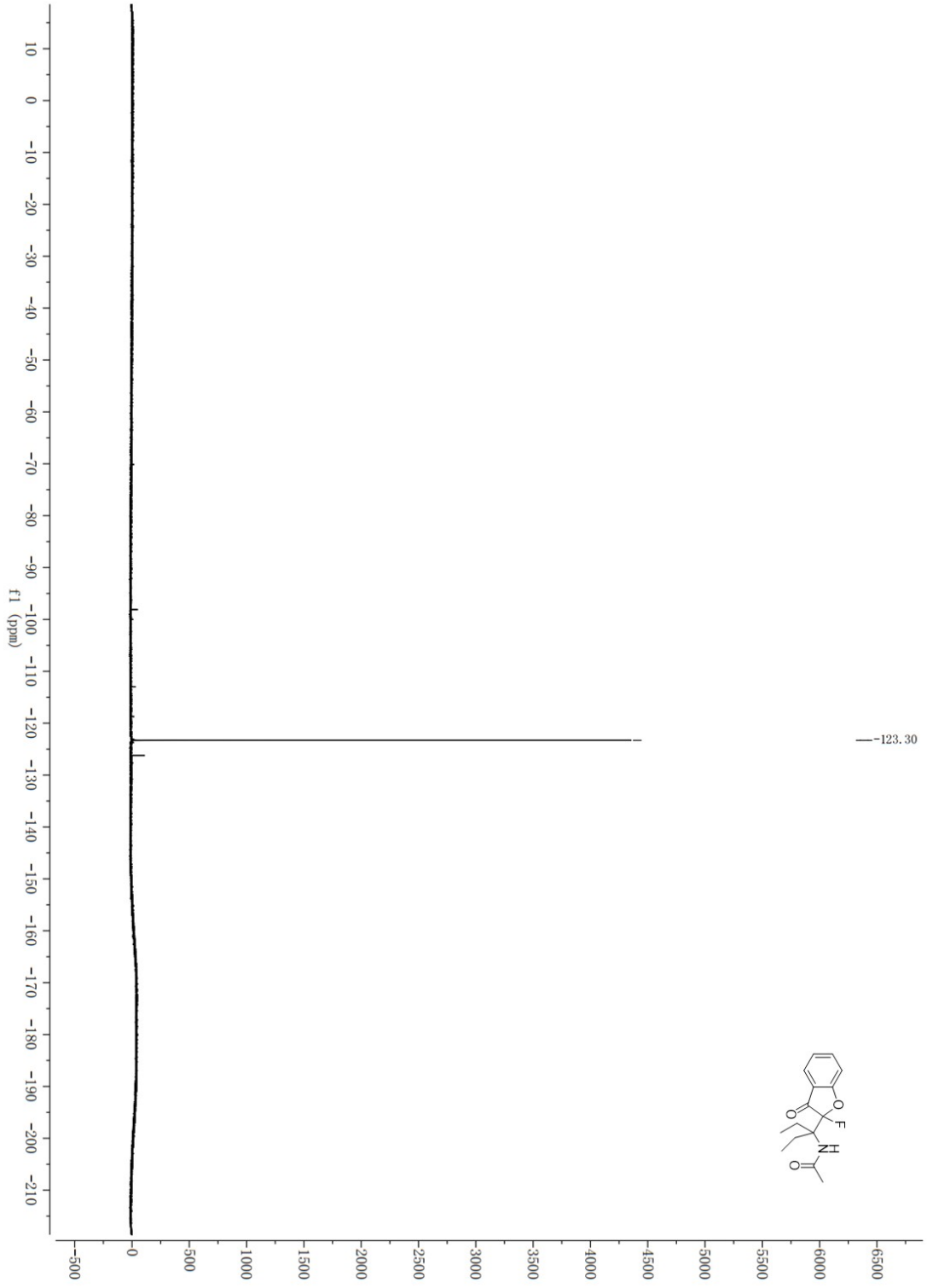

192.42
192.23
170.17
169.81
139.61
139.36
125.57
125.43
123.89
123.84
118.89
113.39
113.15
110.83
108.41
77.32
77.00
76.68
46.35
46.08
23.24
14.75
14.70
f1 (ppm)

(2d)

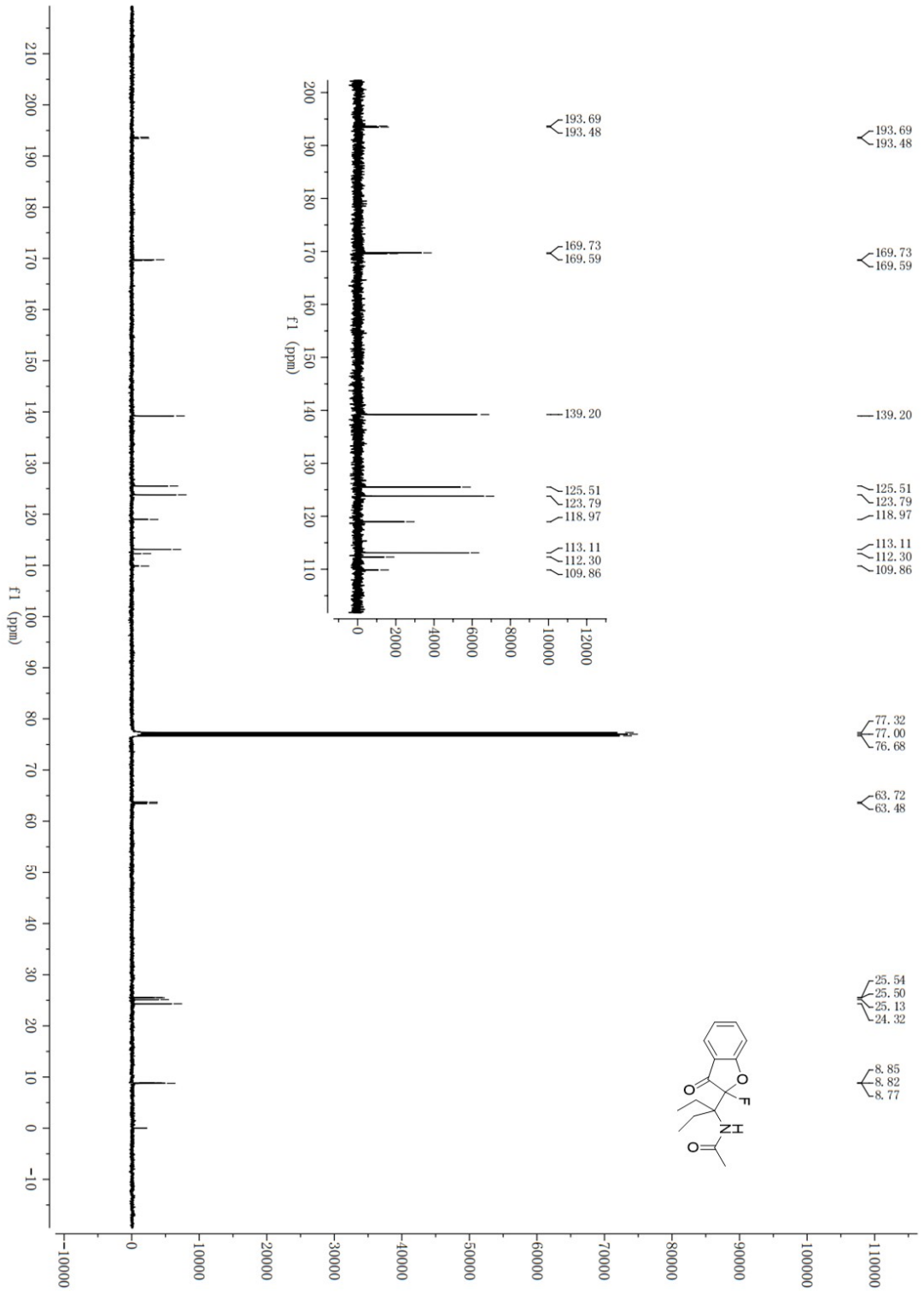
^{1}H NMR


f1 (ppm)
2.26
2.27
1.00
1.26
1.14
1.26
3.29
1.38
3.54
3.82
7.71
7.70
7.69
7.68
7.66
7.26
7.20
7.18
7.17
7.15
7.13
6.14
2.36
2.34
2.33
2.31
2.24
2.22
2.20
2.19
2.13
2.12
2.10
2.09
2.08
1.96
1.91
1.89
1.87
1.85
1.02
1.00
1.00
0.99
0.83
0.81
0.80
O
O
F
N
H
O

^{19}F NMR

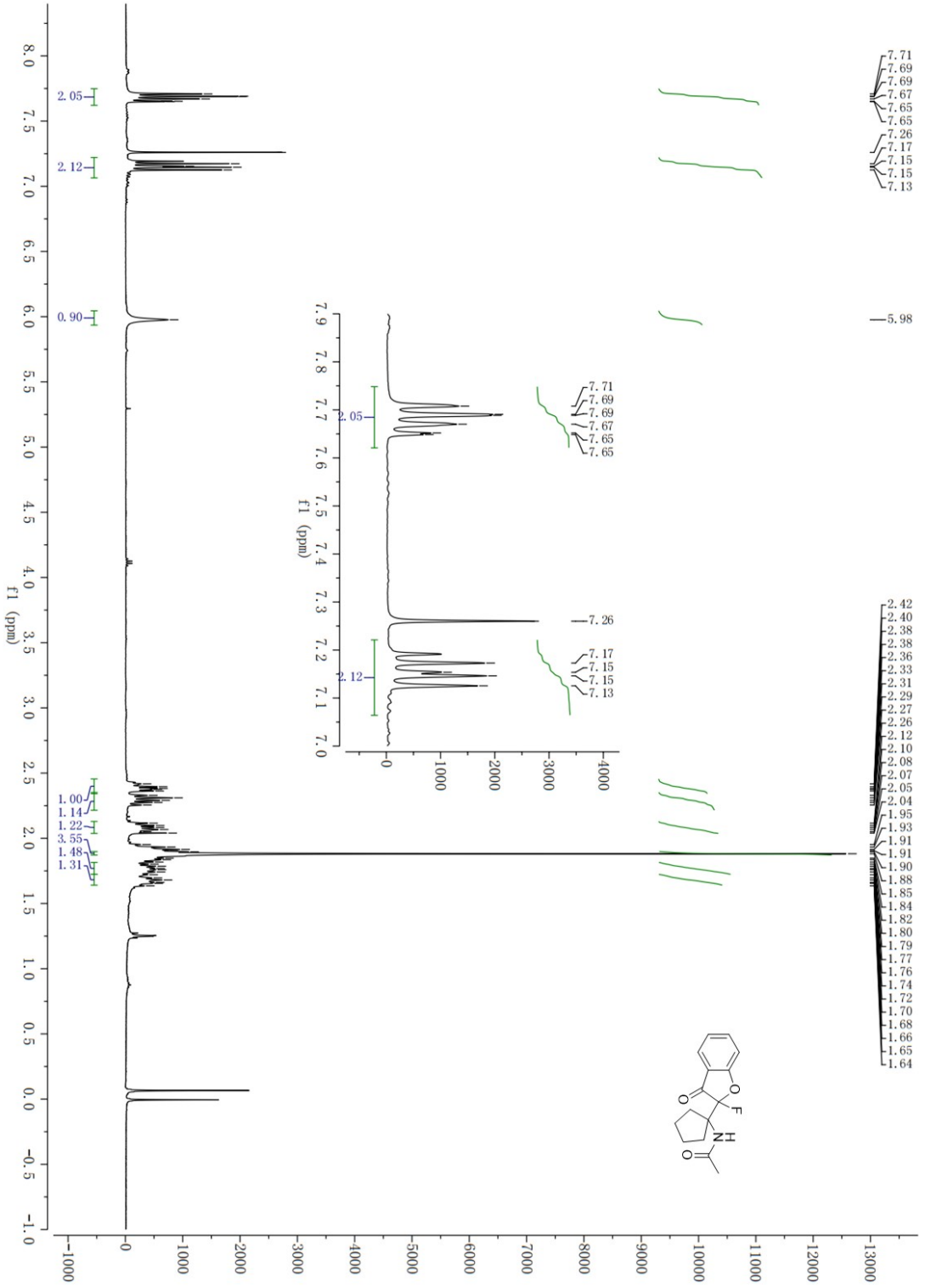


^{13}C NMR

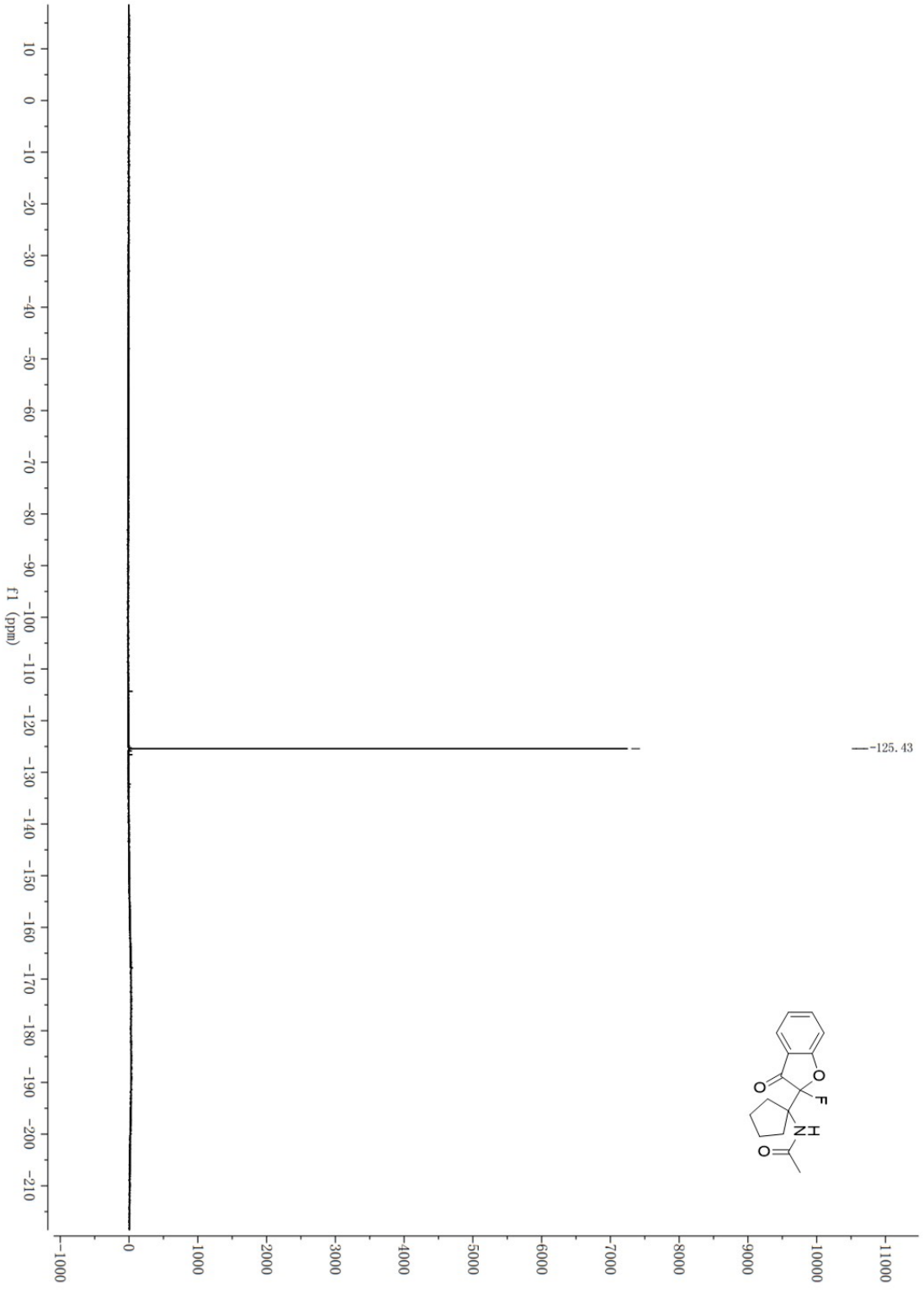

193.69
193.48
169.73
169.59
139.20
125.51
123.79
118.97
113.11
112.30
109.86
77.32
77.00
76.68
63.72
63.48
25.54
25.50
25.13
24.32
8.85
8.82
8.77
f1 (ppm)

(2e)

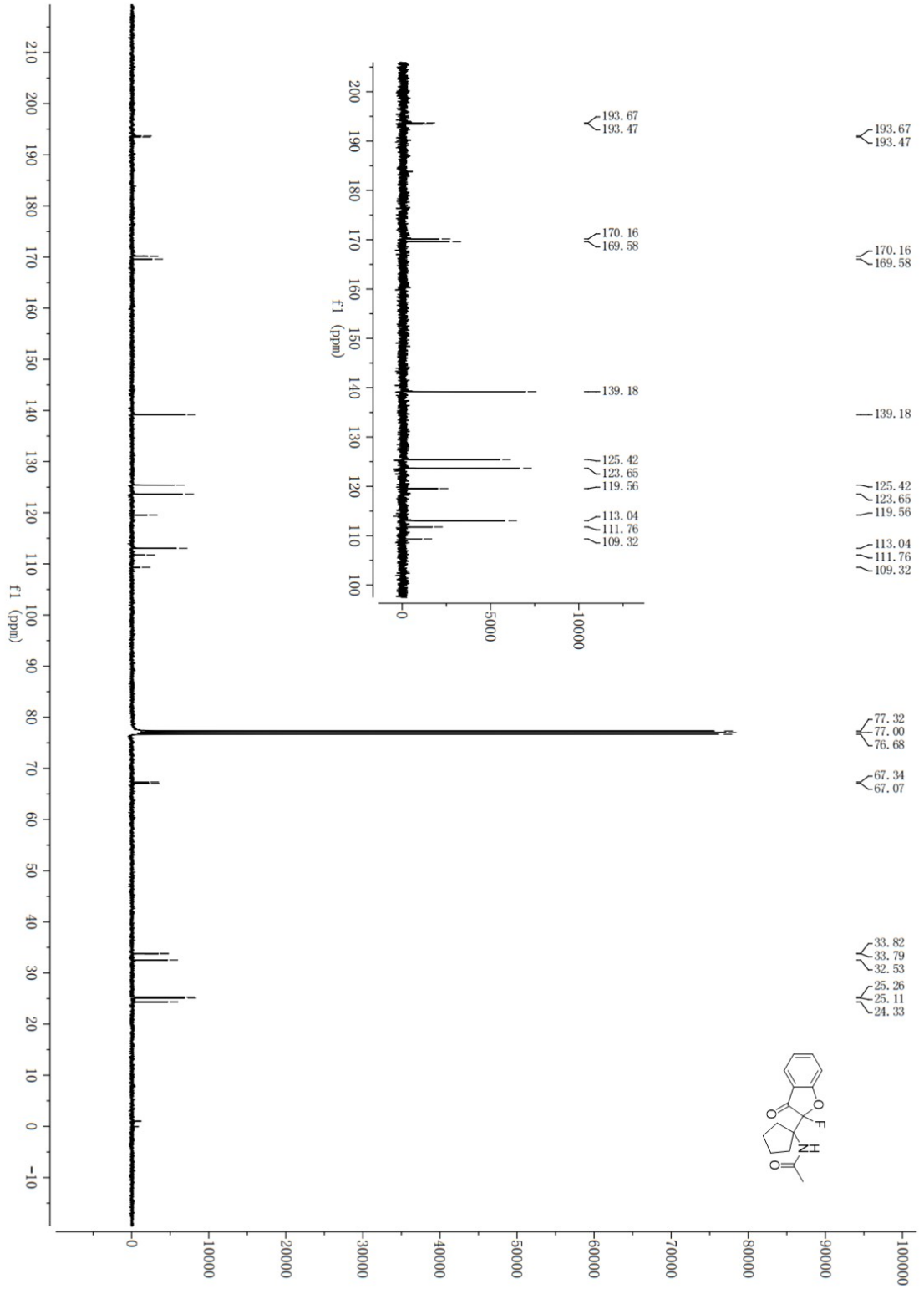
^{1}H NMR



^{19}F NMR

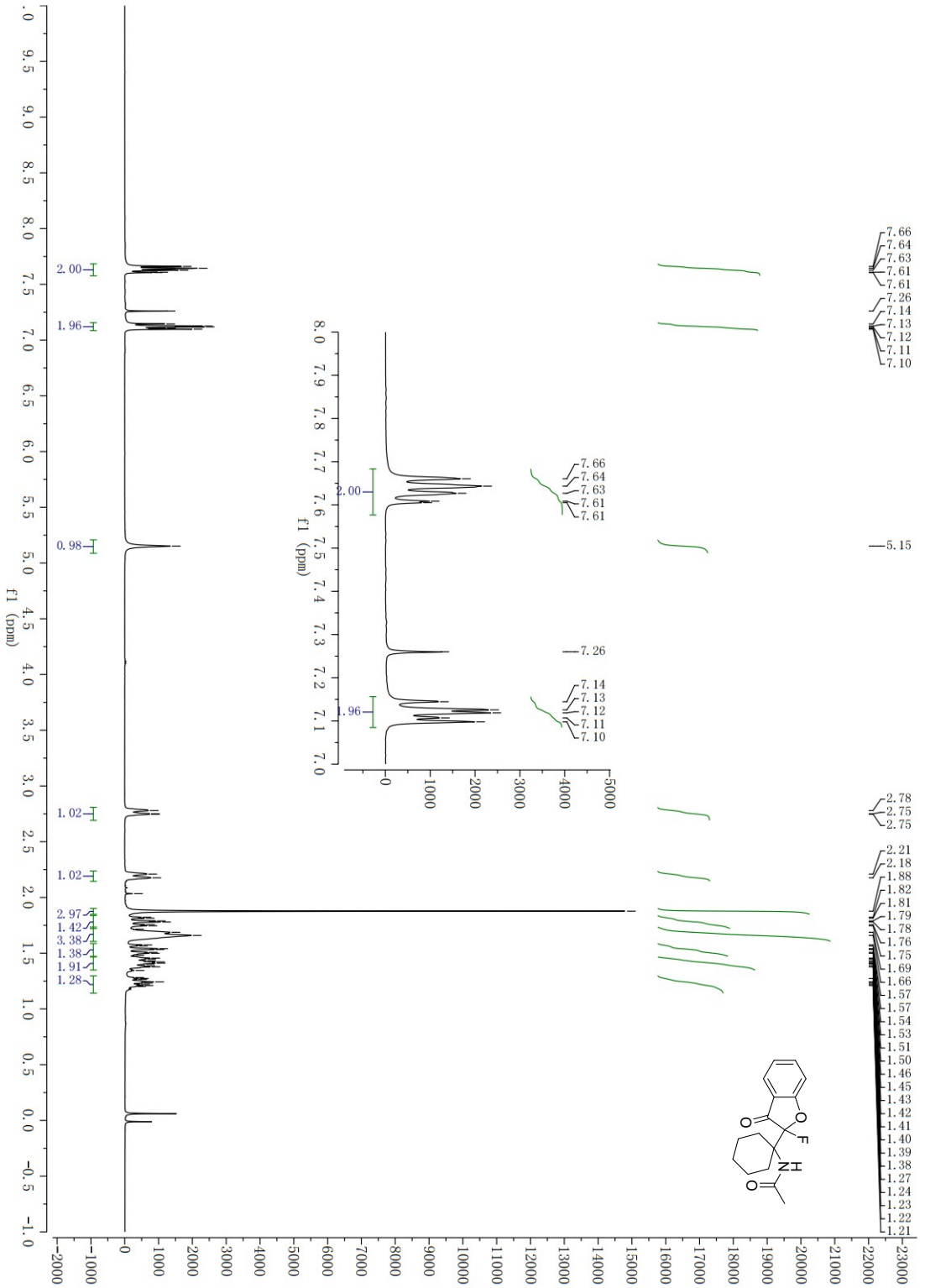


^{13}C NMR

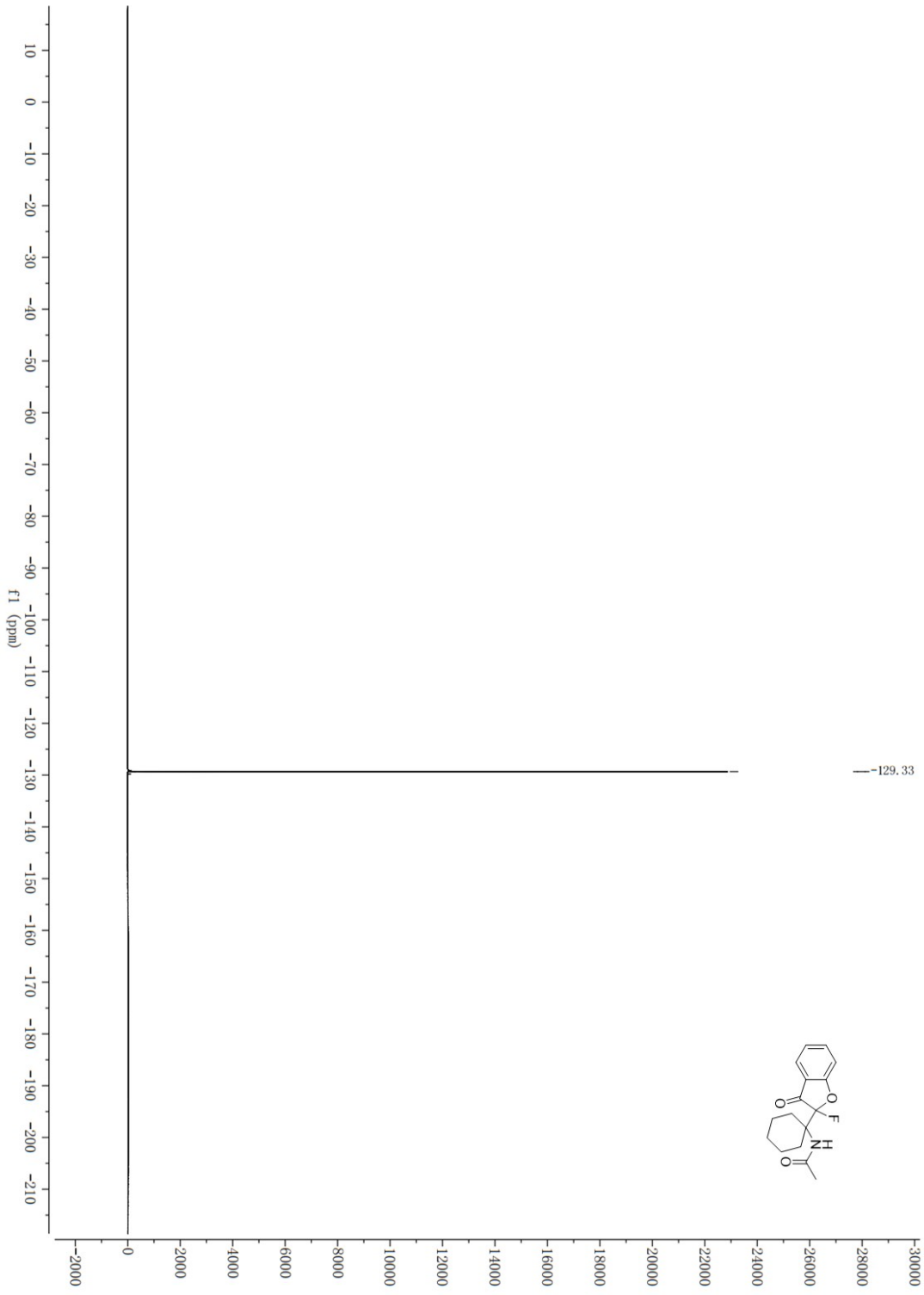

193.67
193.47
170.16
169.58
139.18
125.42
123.65
119.56
113.04
111.76
109.32
77.32
77.00
76.68
67.34
67.07
33.82
33.79
32.53
25.26
25.11
24.33
f1 (ppm)

(2f)

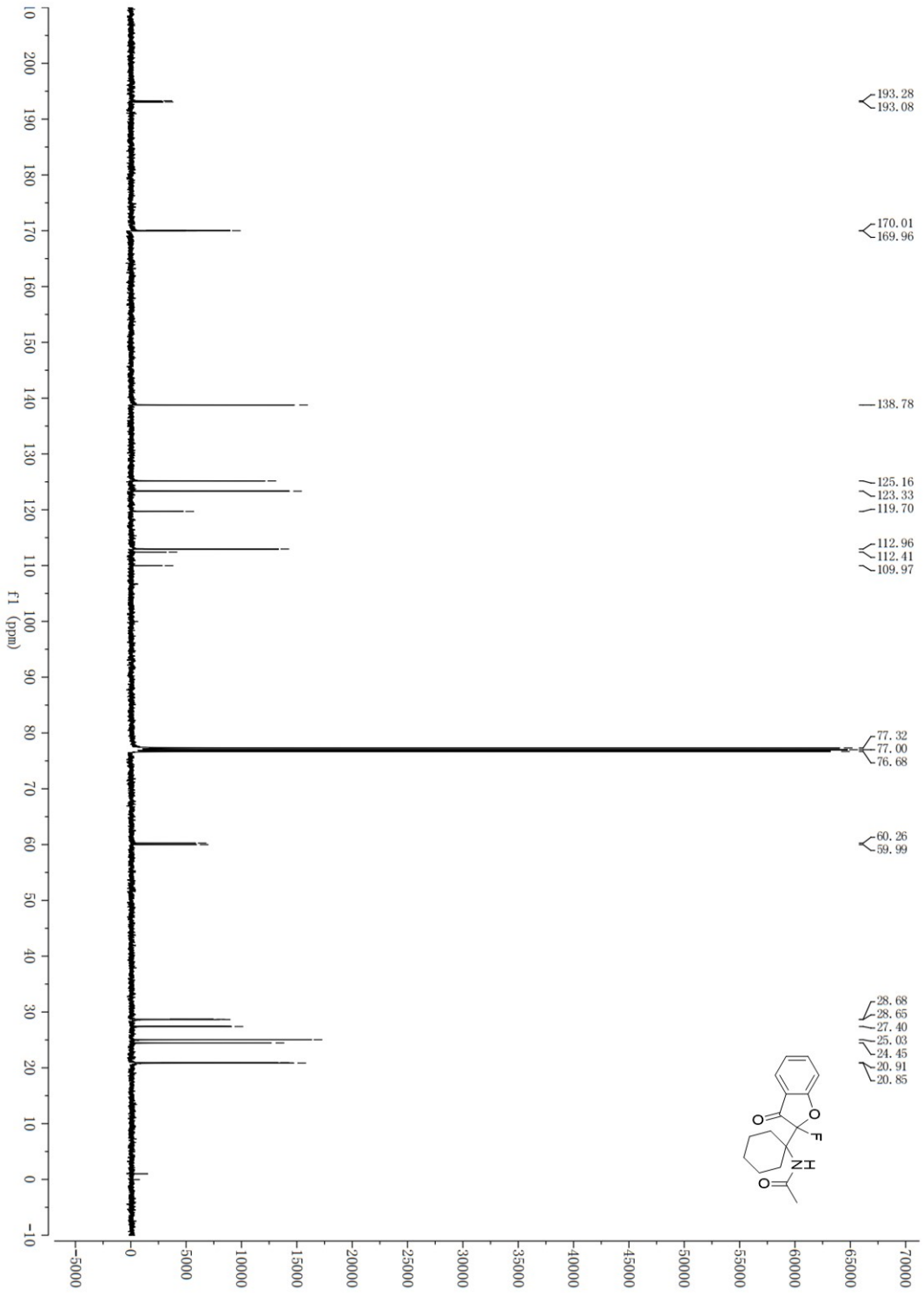
^{1}H NMR


f1 (ppm)
2.00
1.96
0.98
1.02
1.02
2.97
1.42
3.38
1.38
1.91
1.28
7.66
7.64
7.63
7.61
7.61
7.26
7.14
7.13
7.12
7.11
7.10
5.15
2.78
2.75
2.75
2.21
2.18
1.88
1.82
1.81
1.79
1.78
1.76
1.75
1.69
1.66
1.57
1.57
1.54
1.53
1.51
1.50
1.46
1.45
1.43
1.42
1.41
1.40
1.39
1.38
1.27
1.24
1.23
1.22
1.21
O
O
F
NH
O

^{19}F NMR

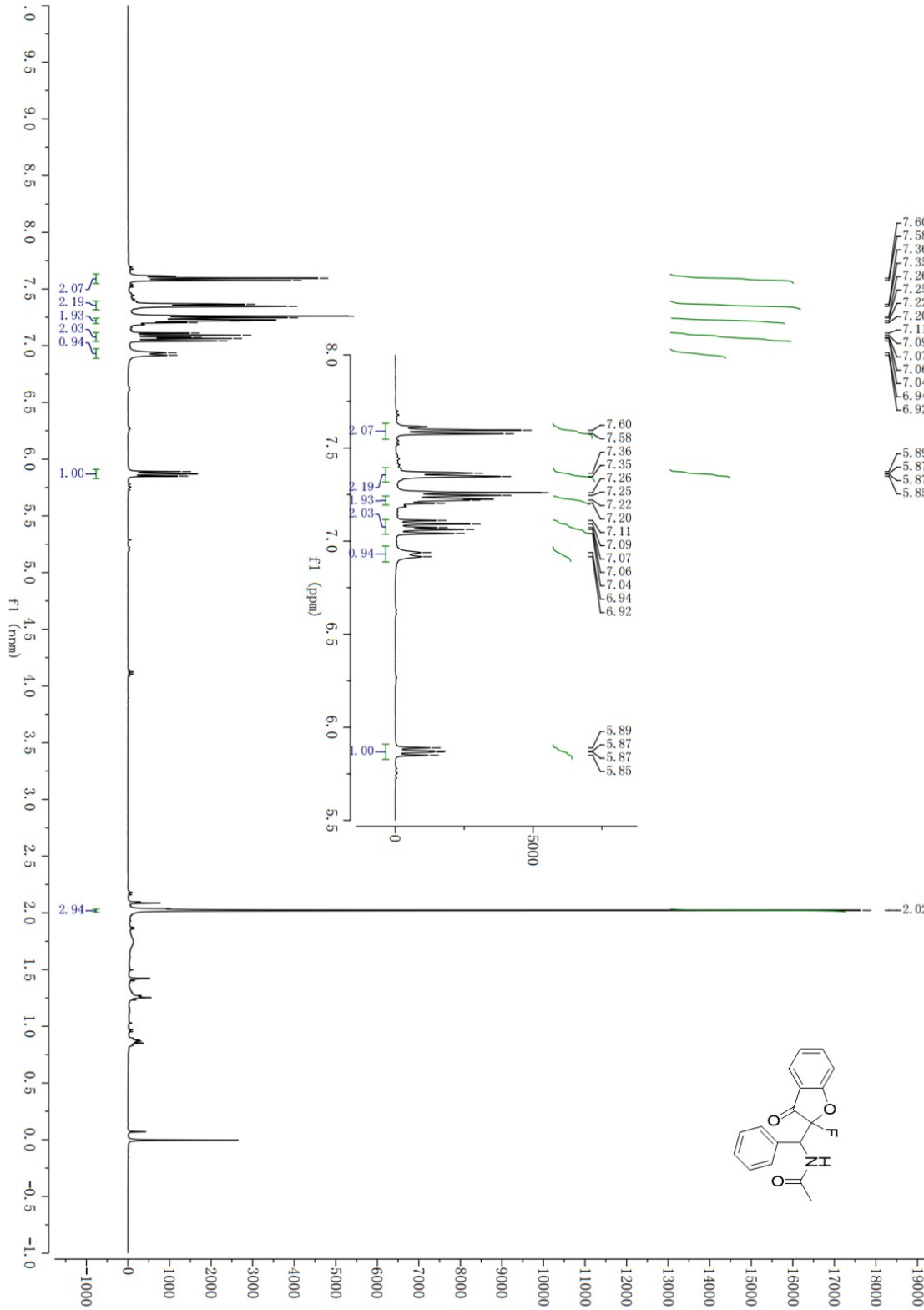

-129.33
f1 (ppm)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
-2000 0 2000 4000 6000 8000 10000 12000 14000 16000 18000 20000 22000 24000 26000 28000 30000
O
F
O
H
N
O

^{13}C NMR

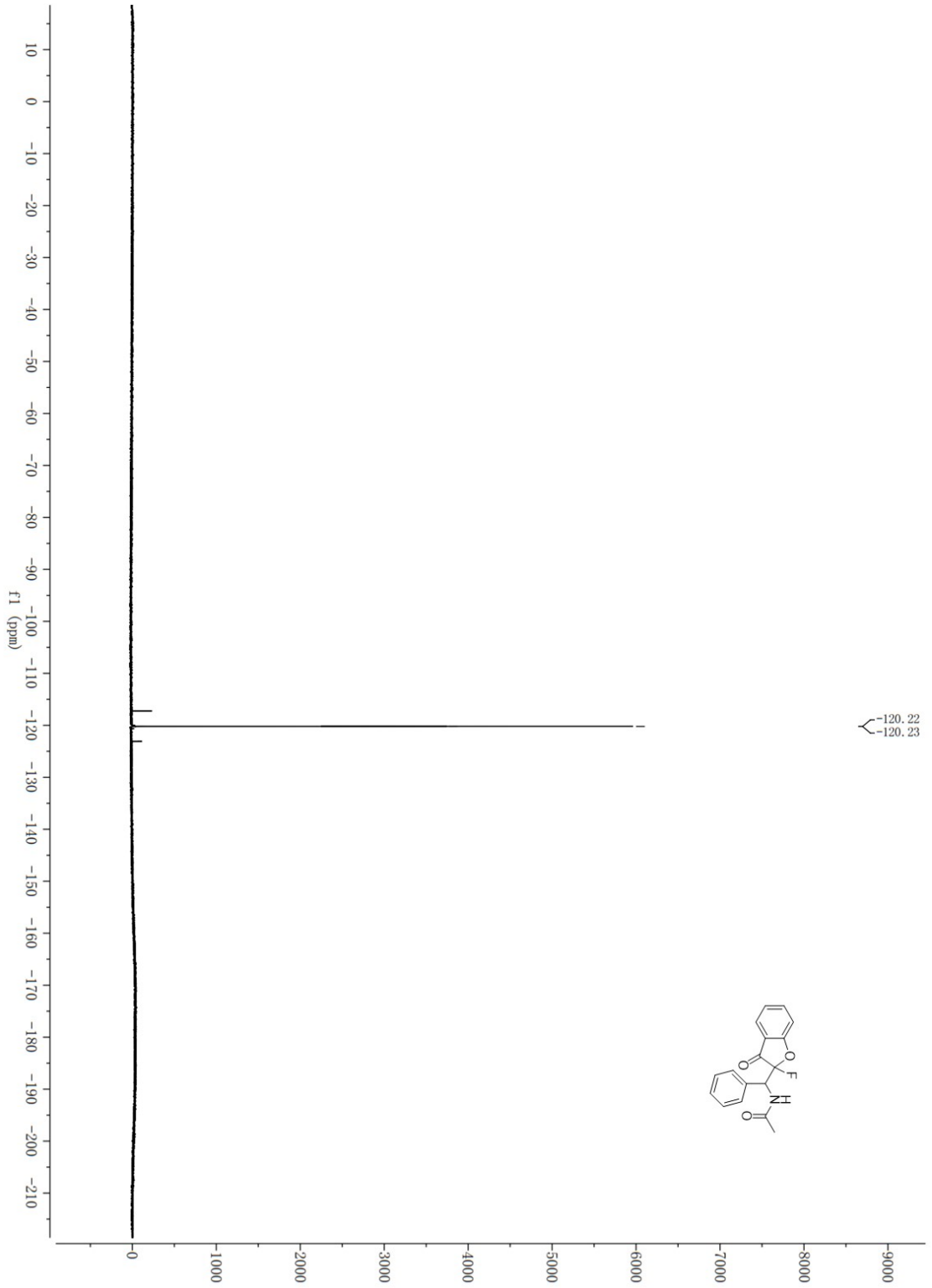

193.28
193.08
170.01
169.96
138.78
125.16
123.33
119.70
112.96
112.41
109.97
77.32
77.00
76.68
60.26
59.99
28.68
28.65
27.40
25.03
24.45
20.91
20.85
f1 (ppm)

(2g)

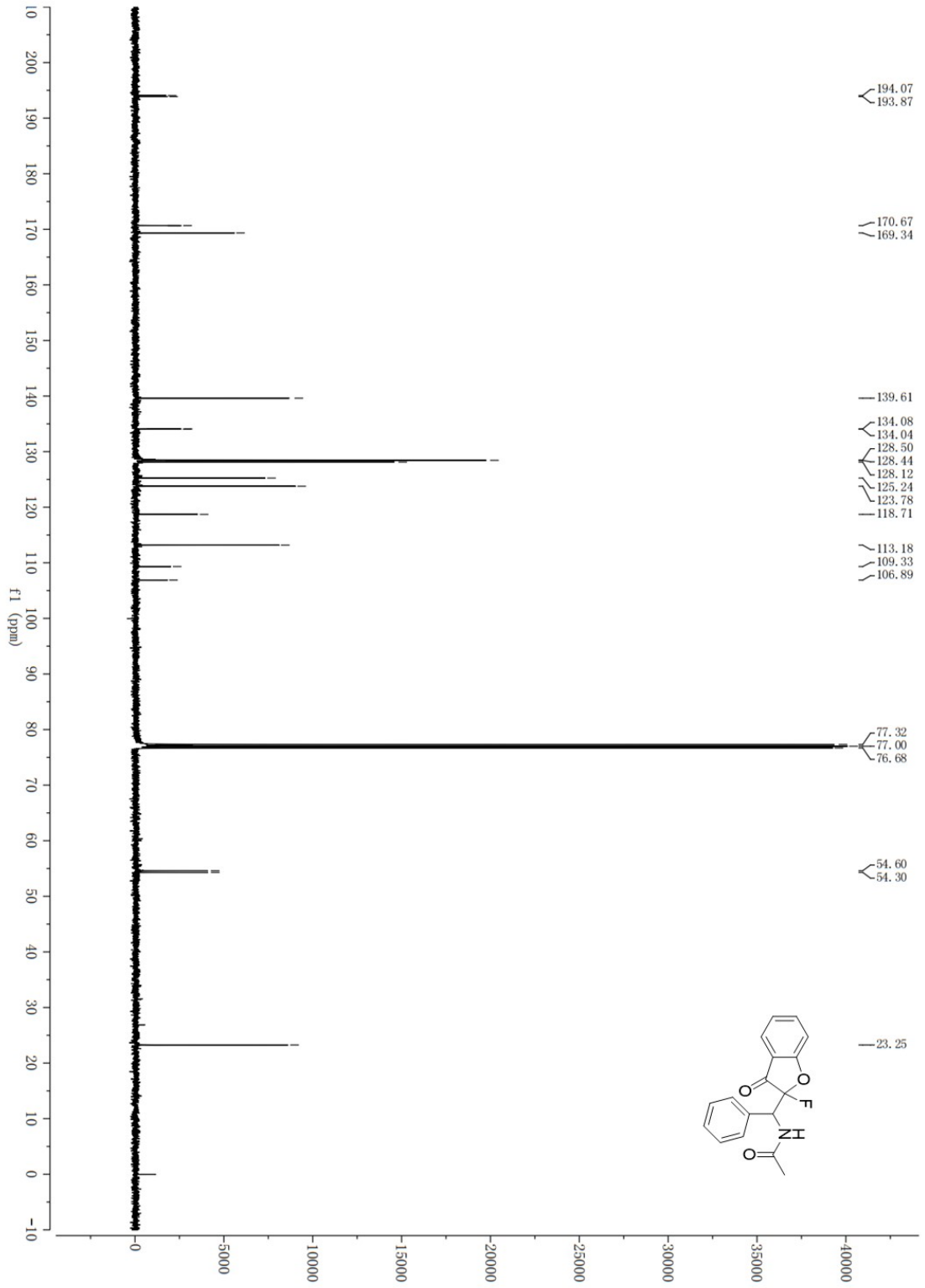
^{1}H NMR


2.07
2.19
1.93
2.03
0.94
1.00
2.94
f1 (ppm)
7.60
7.58
7.36
7.35
7.26
7.25
7.22
7.20
7.11
7.09
7.07
7.06
7.04
6.94
6.92
5.89
5.87
5.87
5.85
O
F
N
H
O

^{19}F NMR



^{13}C NMR


194.07
193.87
170.67
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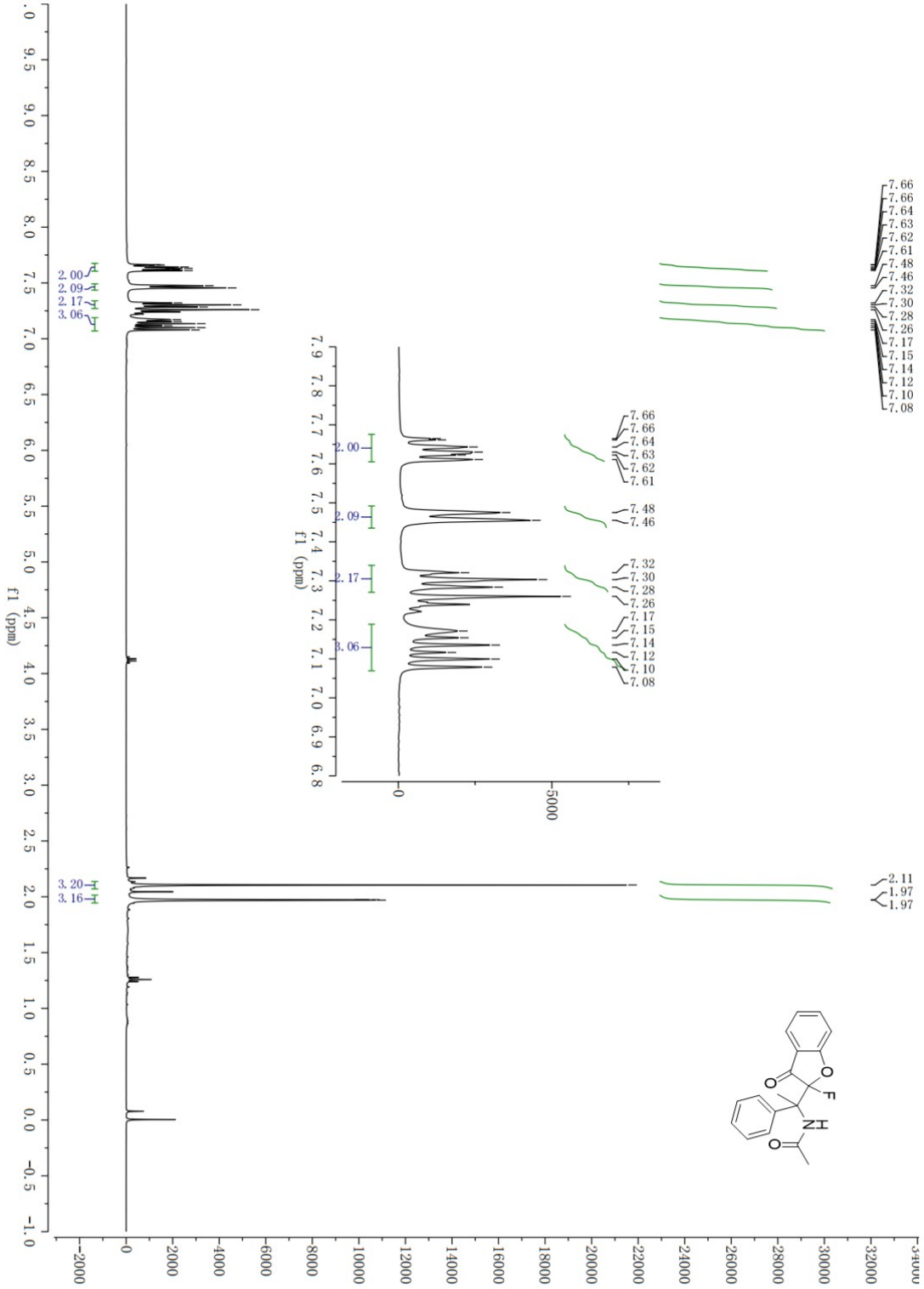
139.61
134.08
134.04
128.50
128.44
128.12
125.24
123.78
118.71
113.18
109.33
106.89
77.32
77.00
76.68
54.60
54.30
23.25
f1 (ppm)
O
F
NH
O

(2h)

^{1}H NMR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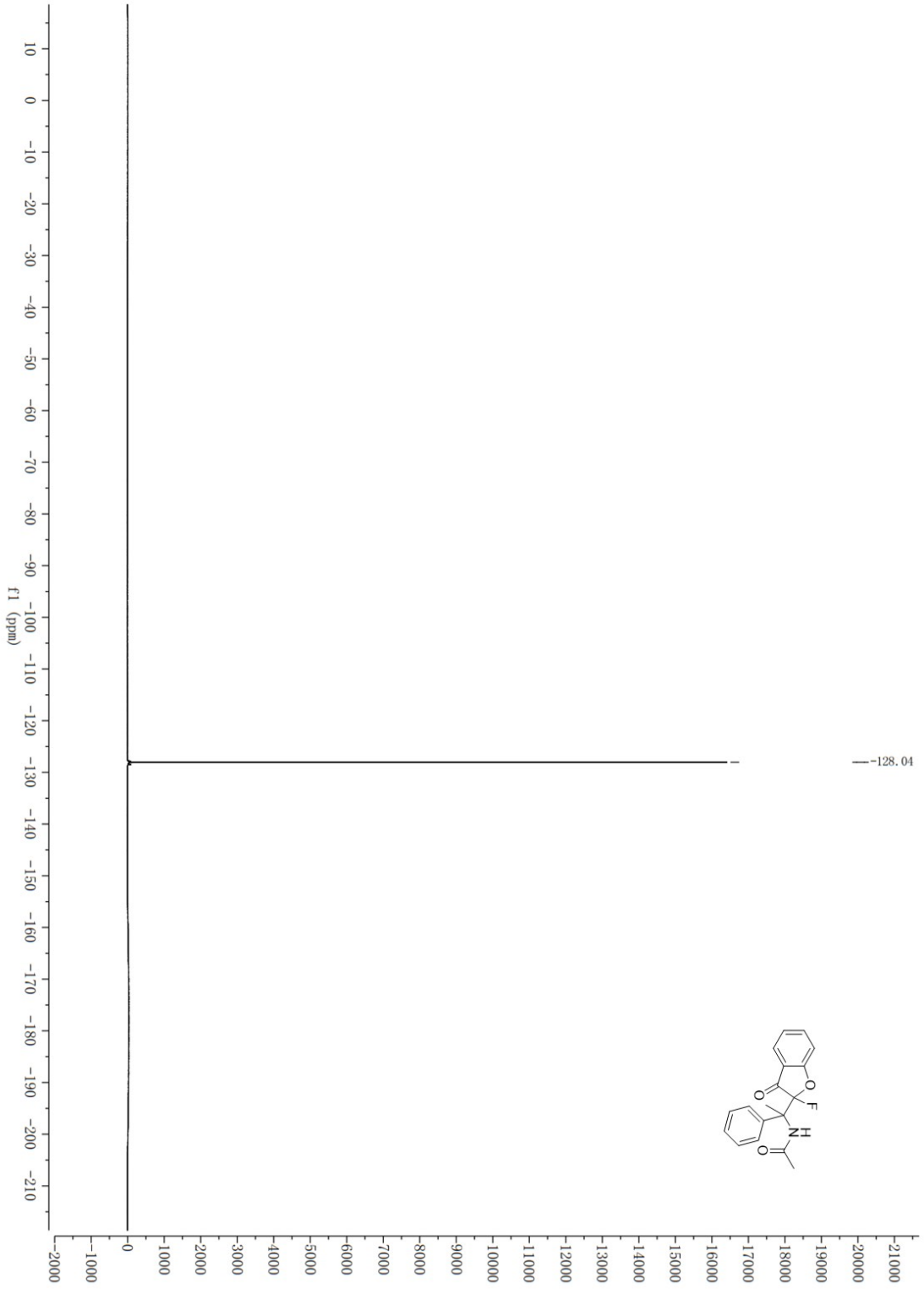
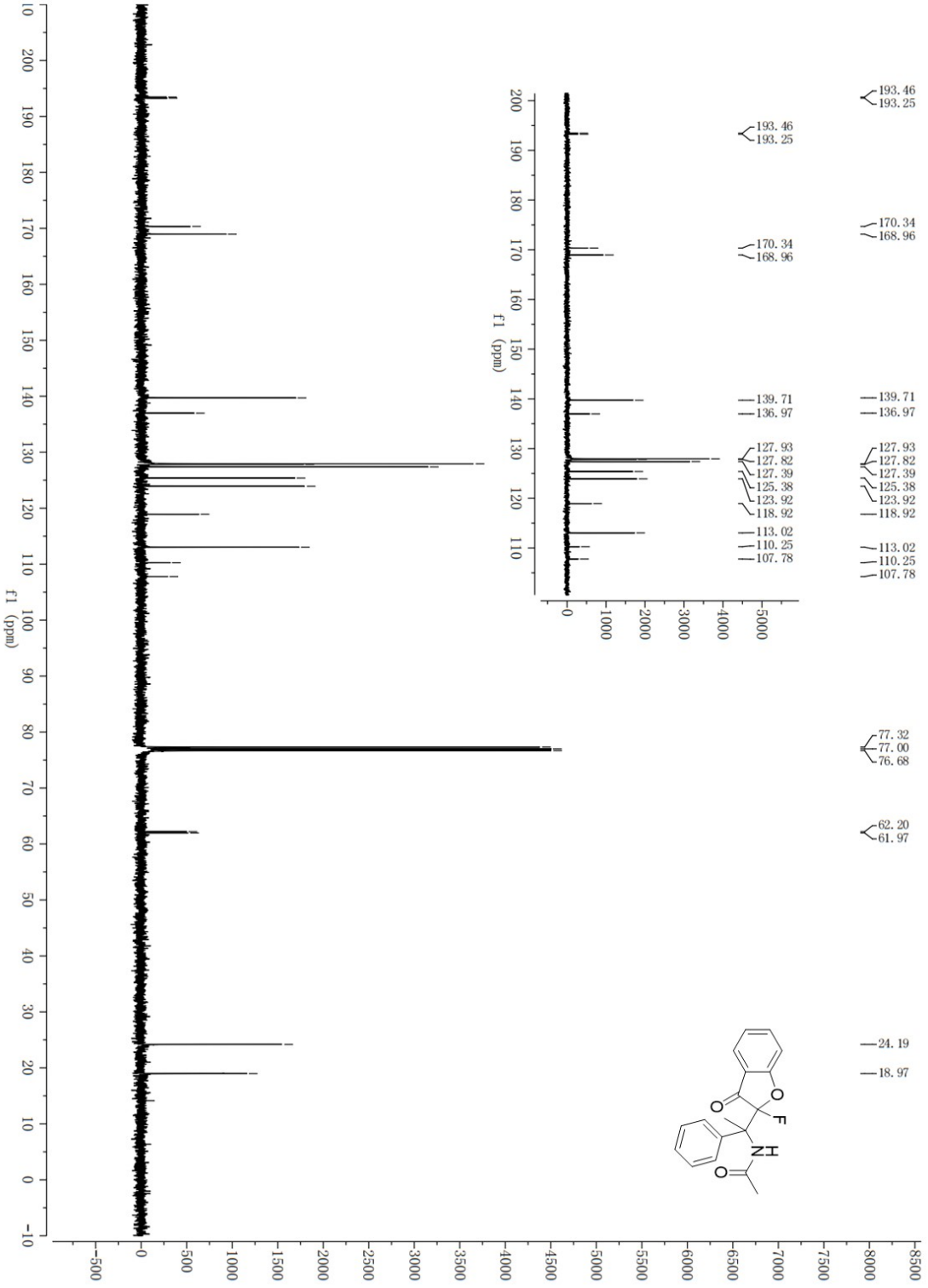
2.09
2.17
3.06
3.20
3.16
7.66
7.66
7.64
7.63
7.62
7.61
7.48
7.46
7.32
7.30
7.28
7.26
7.17
7.15
7.14
7.12
7.10
7.08
2.11
1.97
1.97
f1 (ppm)

^{19}F NMR

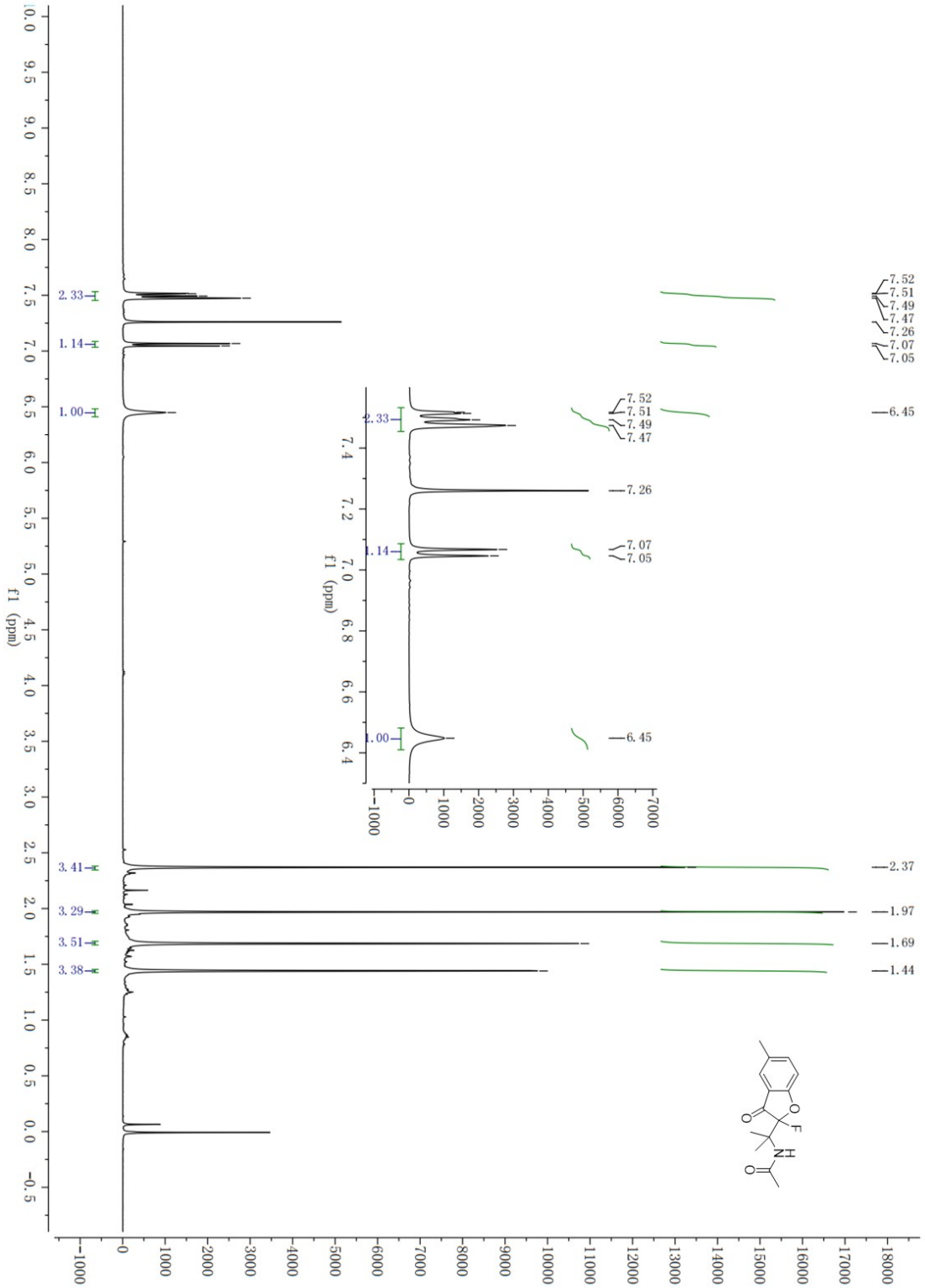


^{13}C NMR

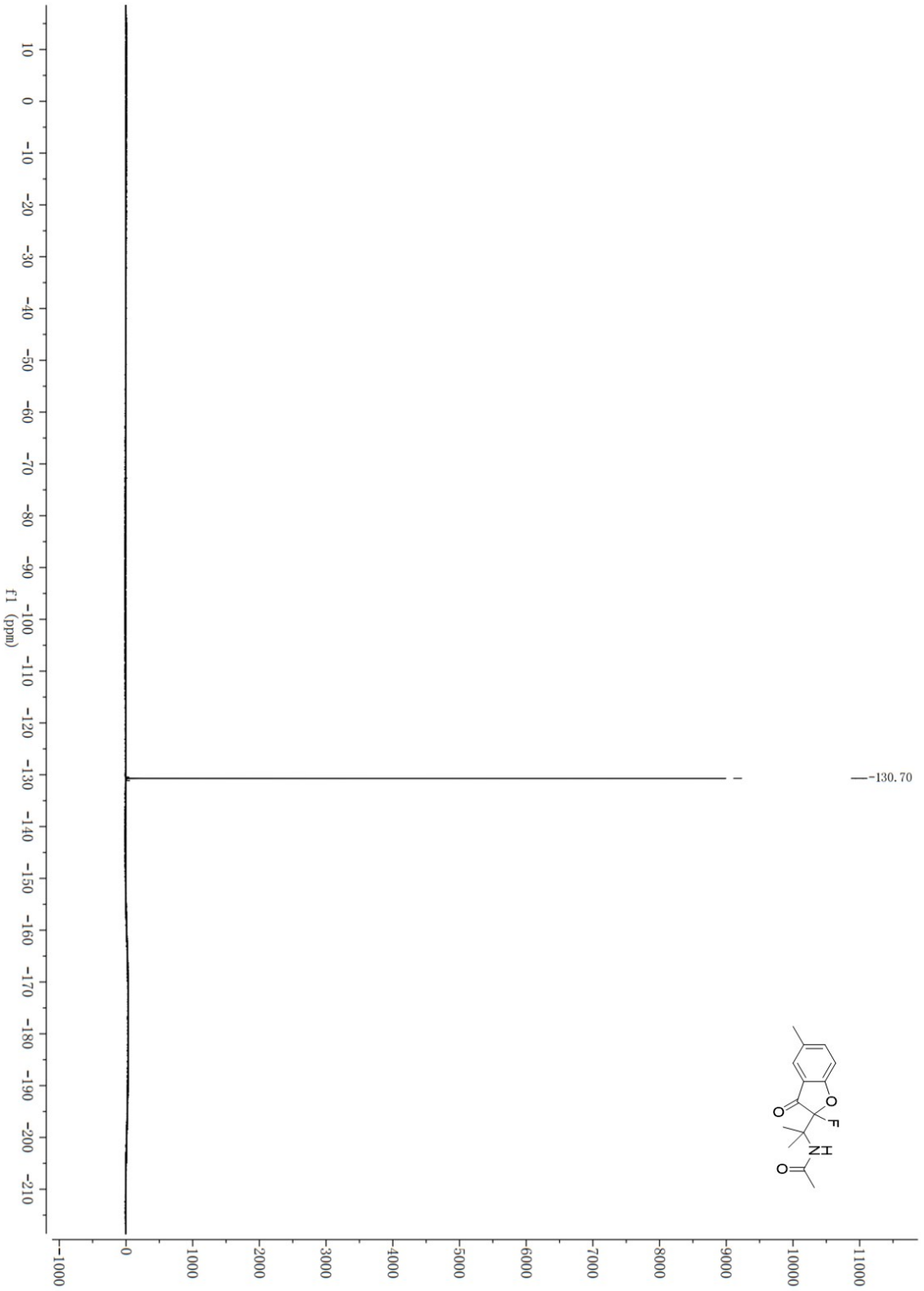

193.46
193.25
170.34
168.96
139.71
136.97
127.93
127.82
127.39
125.38
123.92
118.92
113.02
110.25
107.78
77.32
77.00
76.68
62.20
61.97
24.19
18.97
f1 (ppm)

(2i)

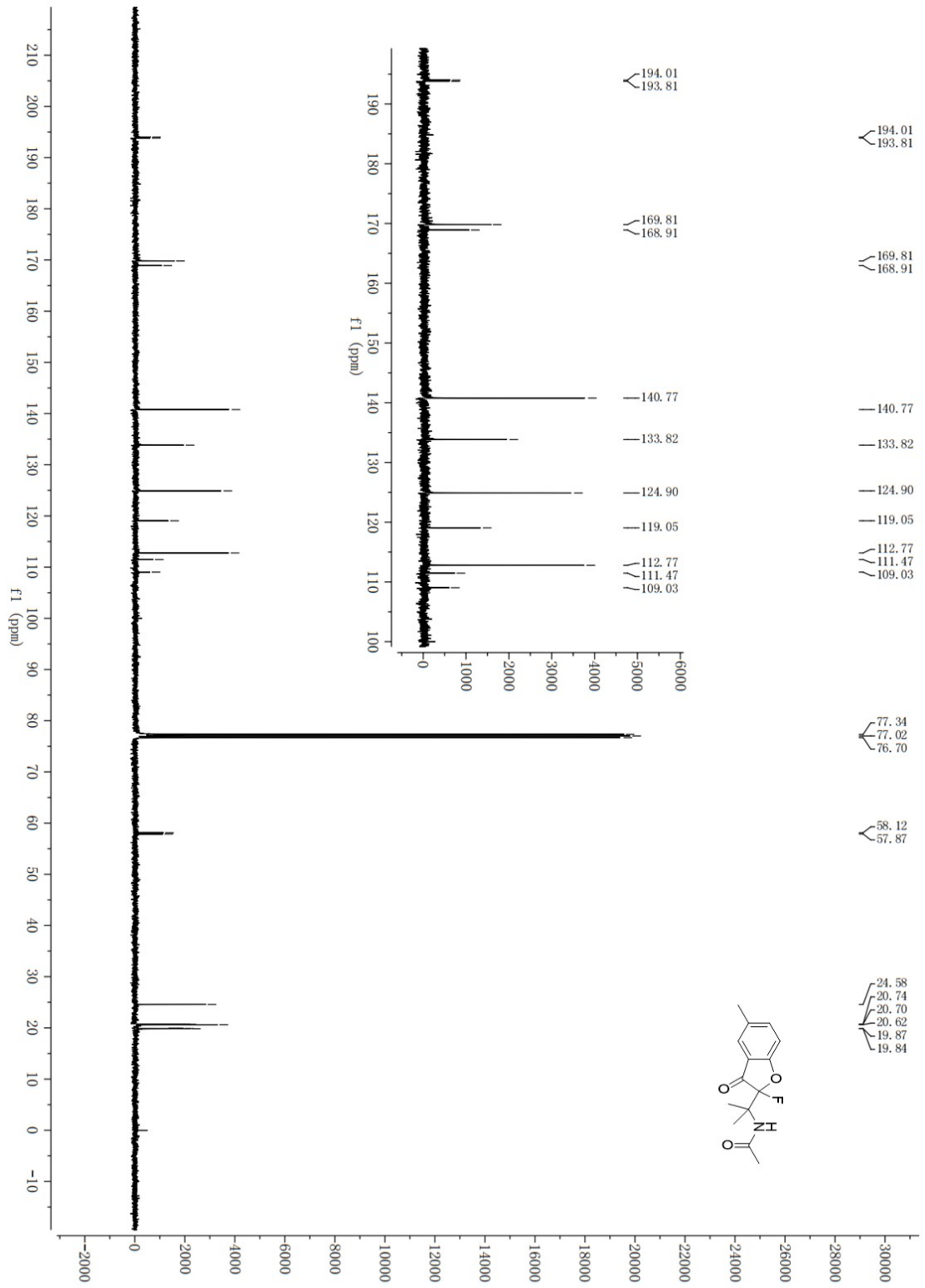
^{1}H NMR


2.33
1.14
1.00
3.41
3.29
3.51
3.38
7.52
7.51
7.49
7.47
7.26
7.07
7.05
6.45
2.37
1.97
1.69
1.44
f1 (ppm)

^{19}F NMR

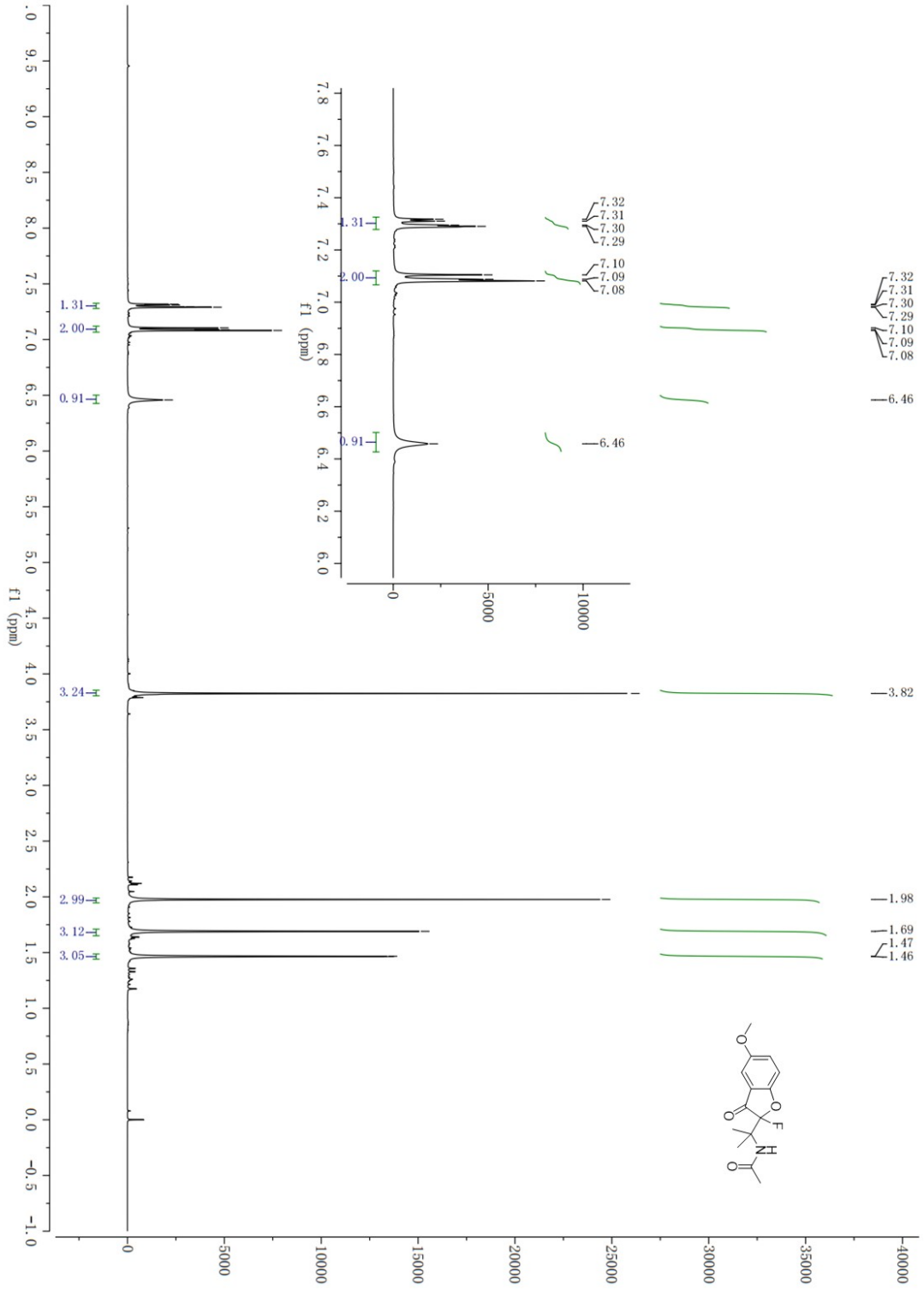


^{13}C NMR


194.01
193.81
169.81
168.91
140.77
133.82
124.90
119.05
112.77
111.47
109.03
77.34
77.02
76.70
58.12
57.87
24.58
20.74
20.70
20.62
19.87
19.84
f1 (ppm)
O
F
NH
O

(2j)

^{1}H NMR


1.31
2.00
0.91
3.24
2.99
3.12
3.05
7.32
7.31
7.30
7.29
7.10
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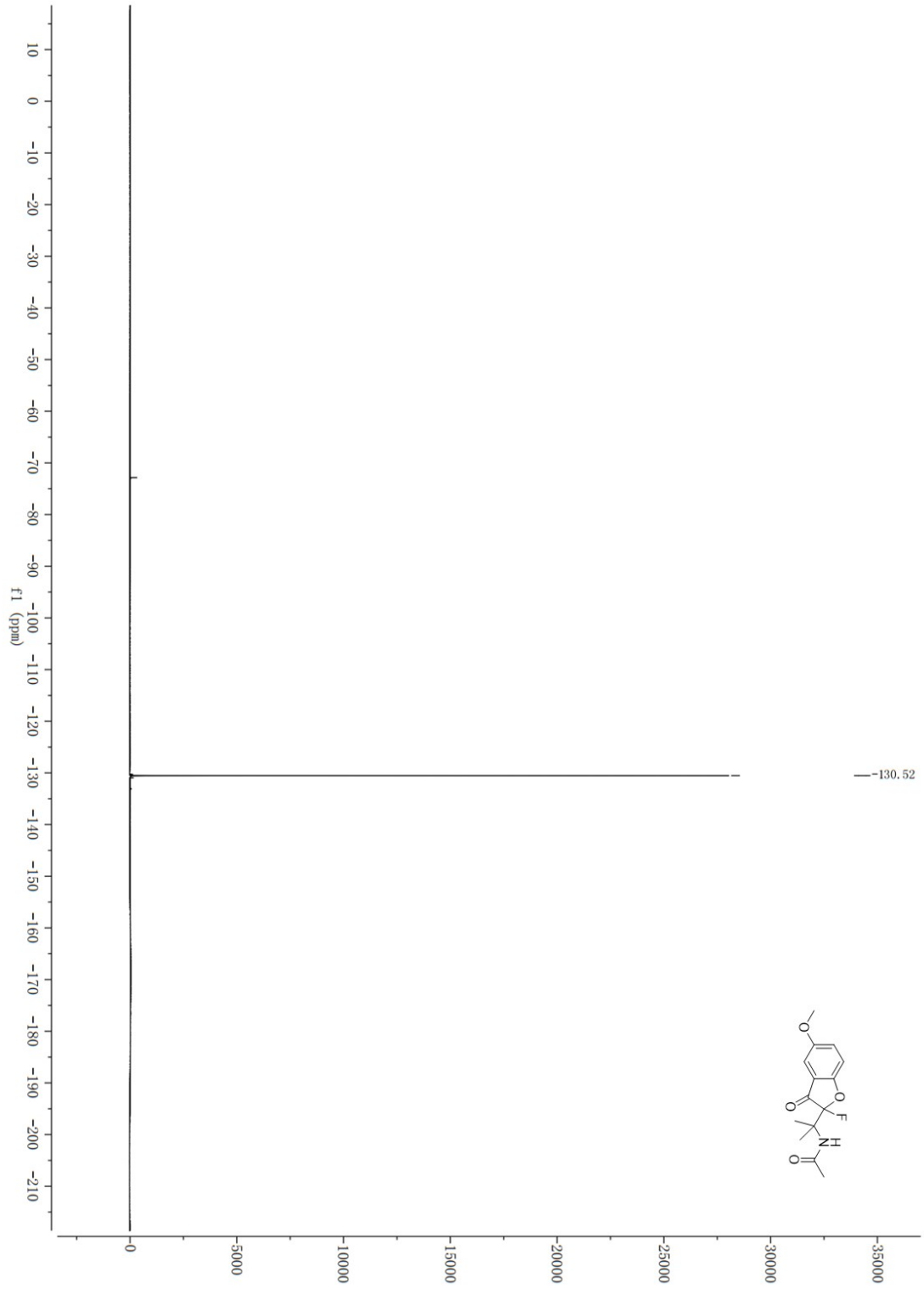
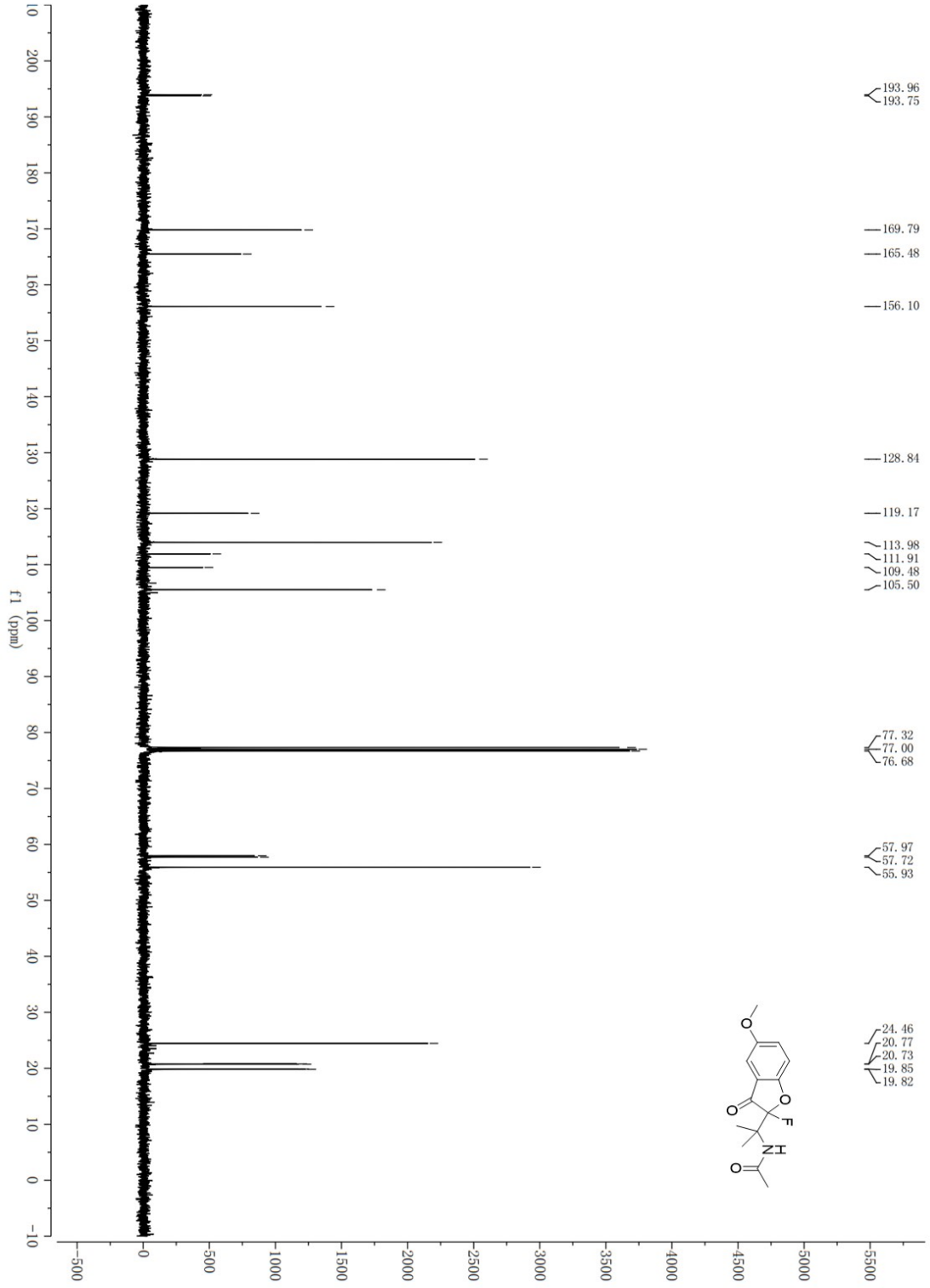
7.08
6.46
3.82
1.98
1.69
1.47
1.46
f1 (ppm)

^{19}F NMR

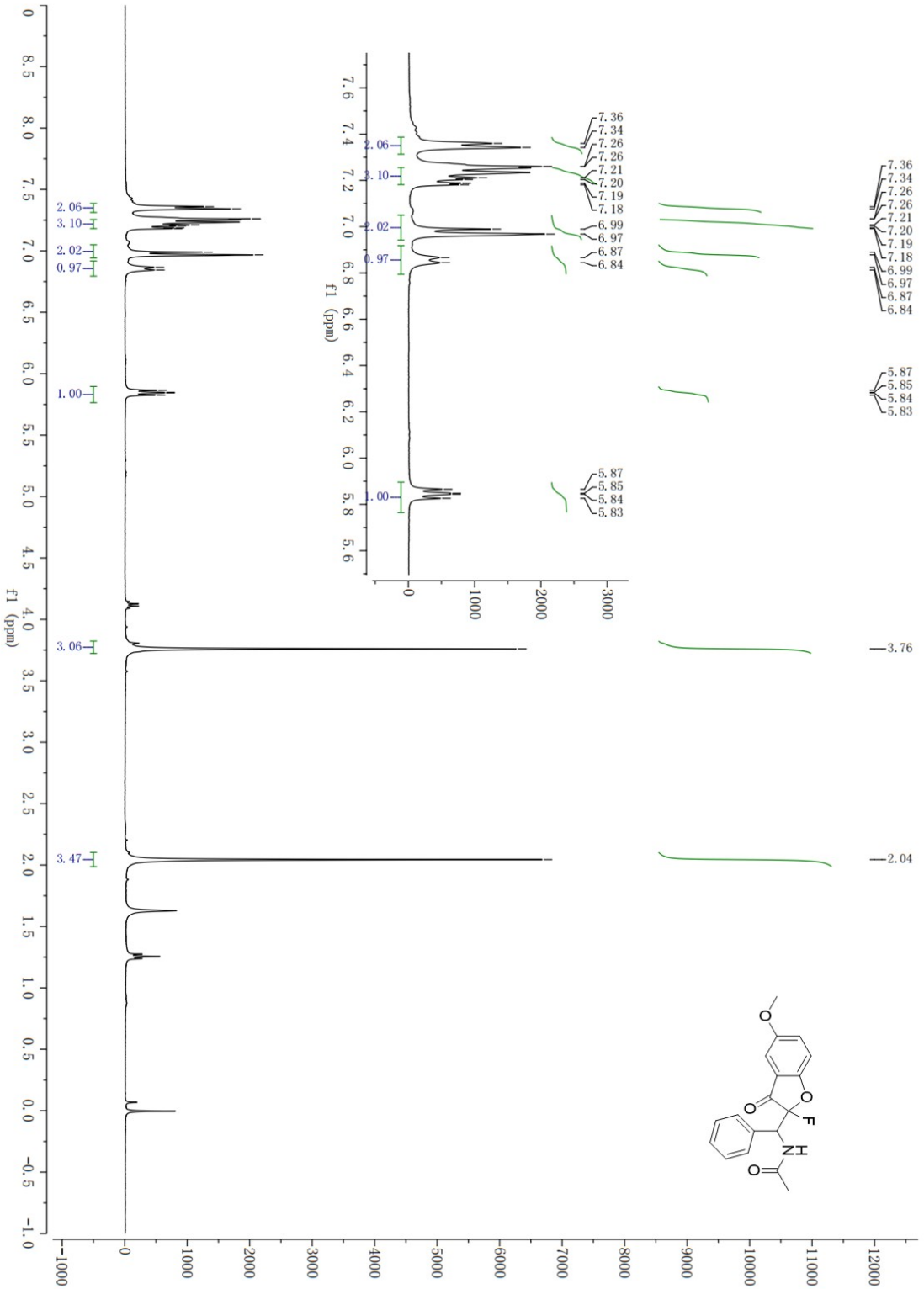

-130.52
f1 (ppm)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
0
5000
10000
15000
20000
25000
30000
35000
O
F
H
N
O
O

^{13}C NMR

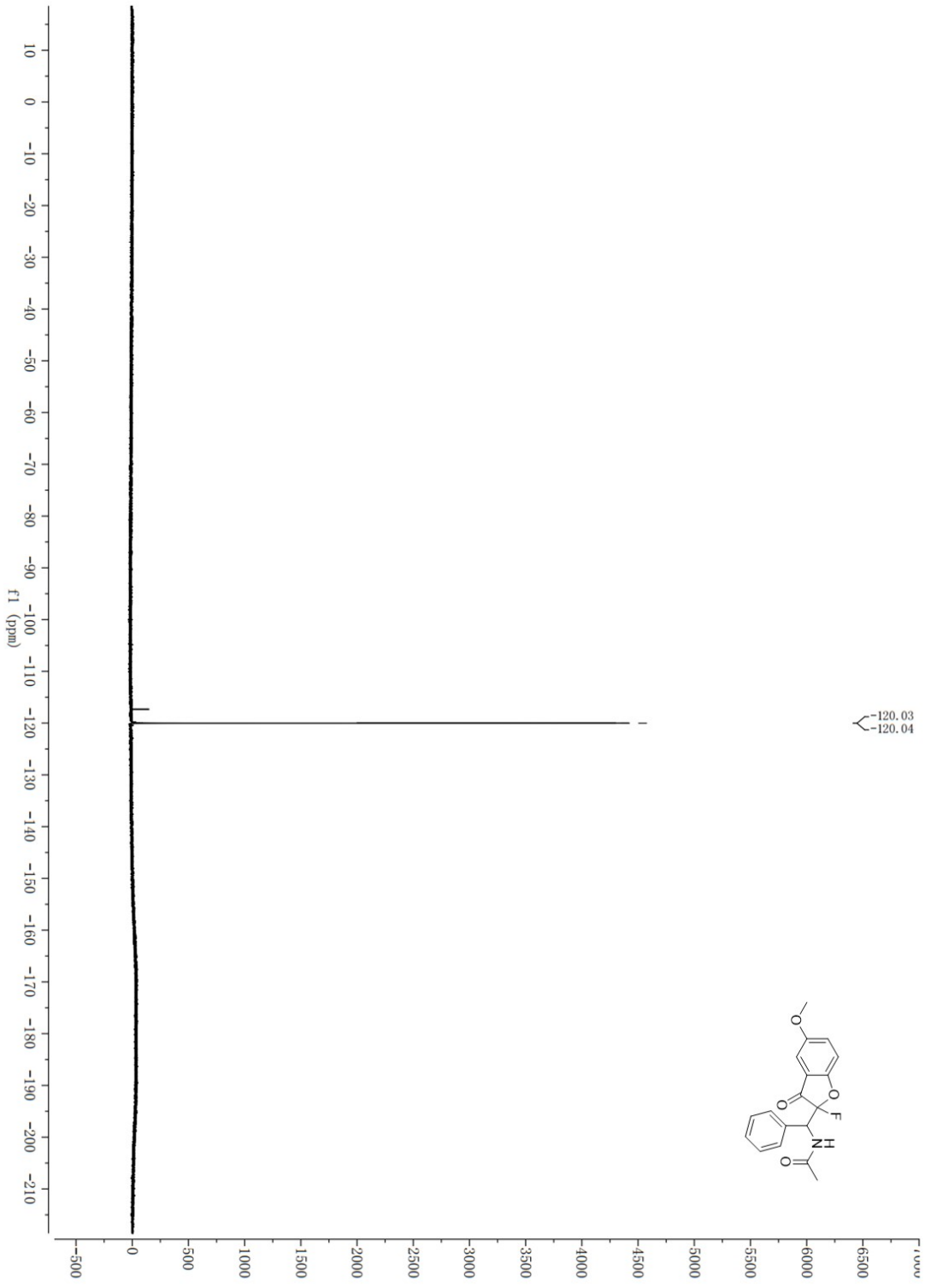

193.96
193.75
169.79
165.48
156.10
128.84
119.17
113.98
111.91
109.48
105.50
77.32
77.00
76.68
57.97
57.72
55.93
24.46
20.77
20.73
19.85
19.82
f1 (ppm)

(2k)

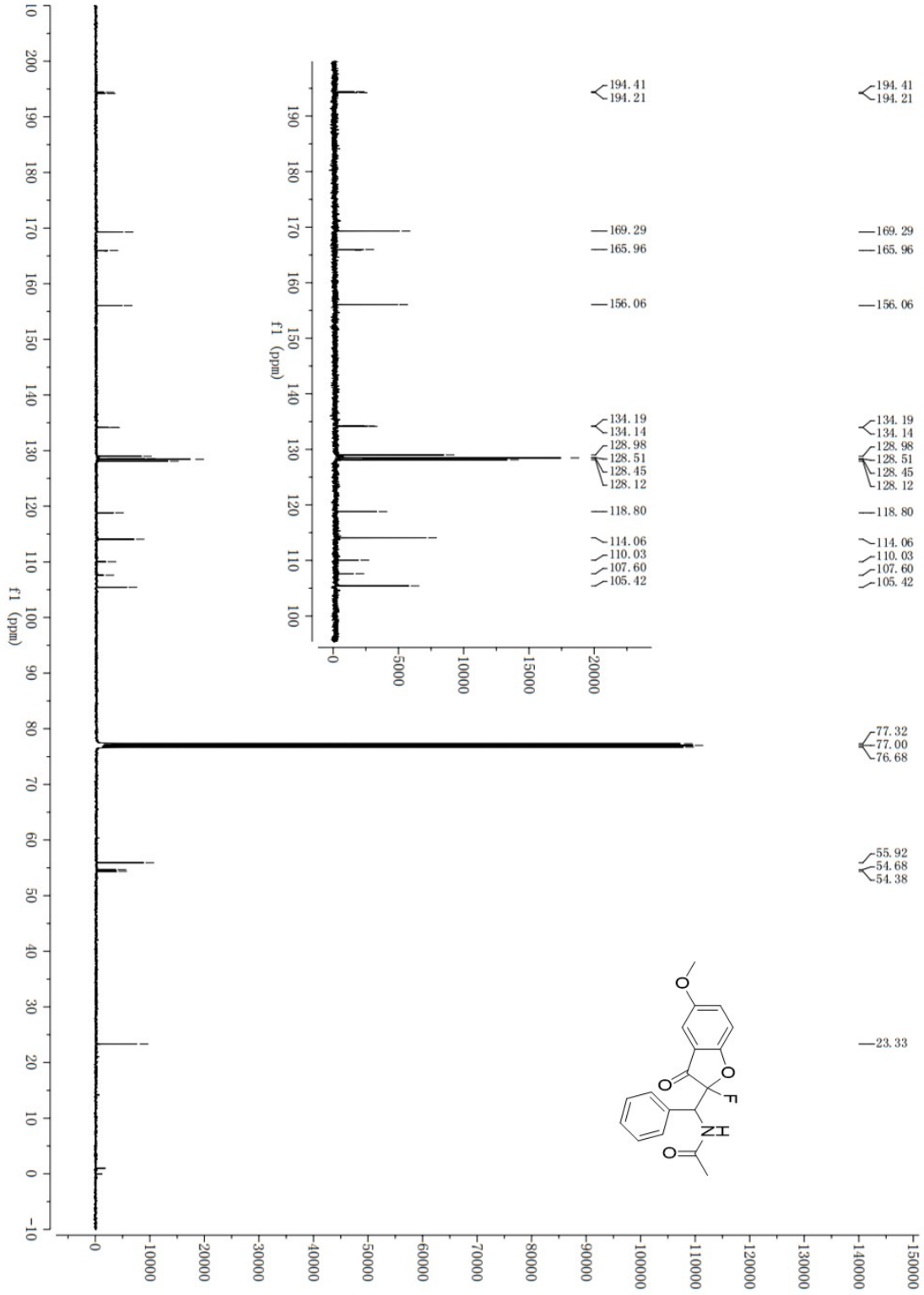
^{1}H NMR


f1 (ppm)
2.06
3.10
2.02
0.97
1.00
3.06
3.47
7.36
7.34
7.26
7.26
7.21
7.20
7.19
7.18
6.99
6.97
6.87
6.84
5.87
5.85
5.84
5.83
3.76
2.04
O
O
F
NH
O

19F NMR

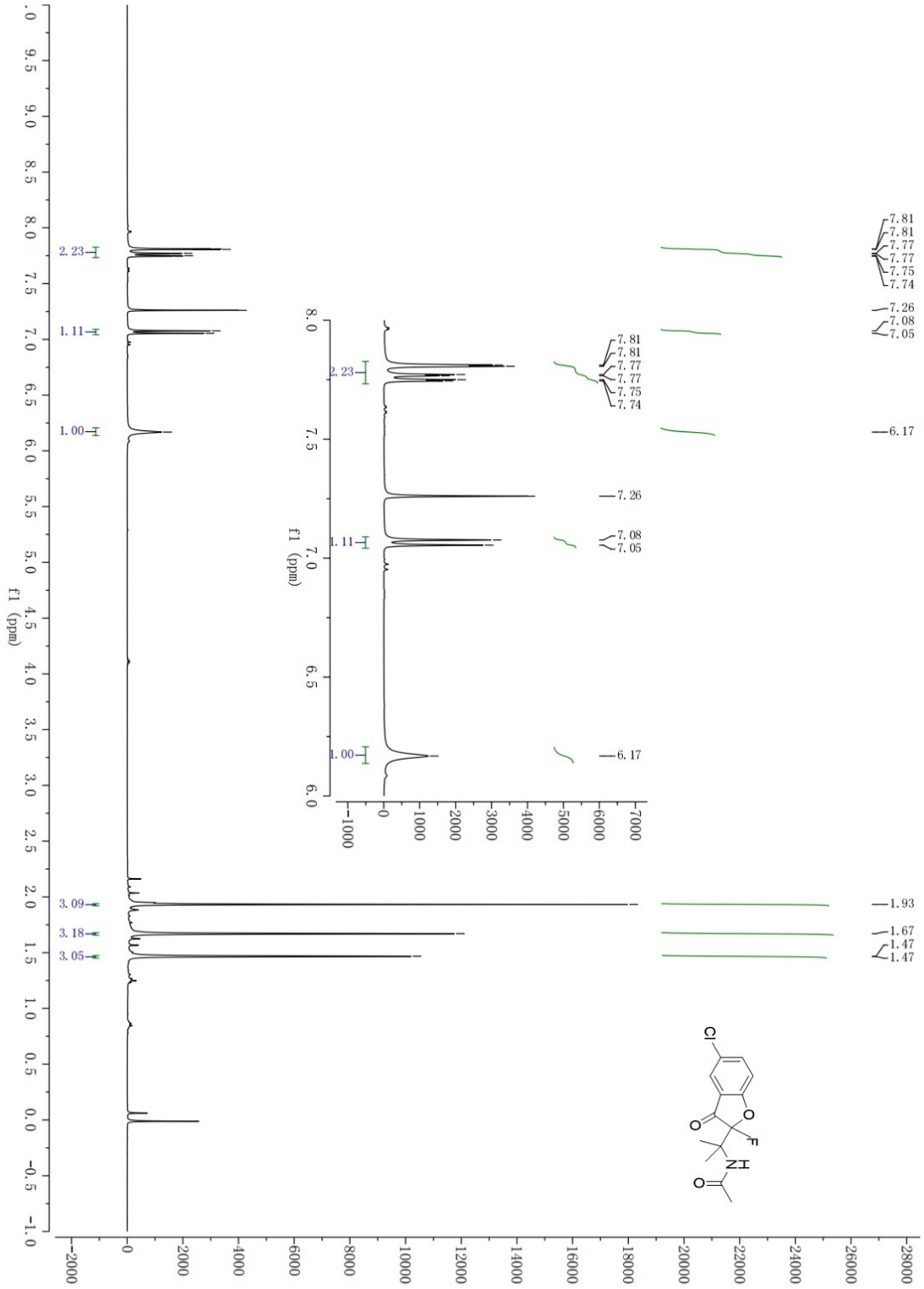


^{13}C NMR

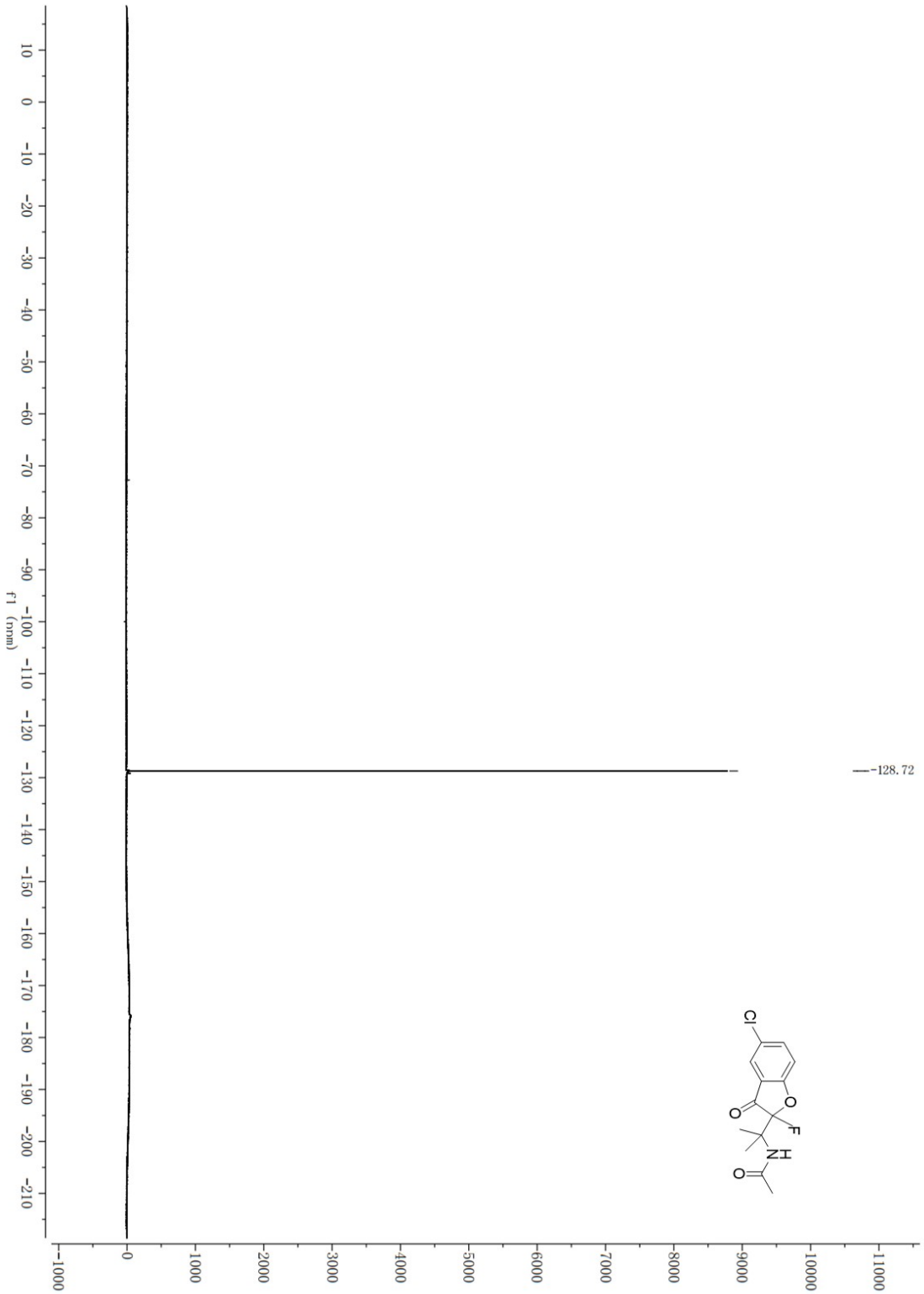

194.41
194.21
169.29
165.96
156.06
134.19
134.14
128.98
128.51
128.45
128.12
118.80
114.06
110.03
107.60
105.42
77.32
77.00
76.68
55.92
54.68
54.38
23.33
f1 (ppm)
O
O
F
NH
O

(2l)

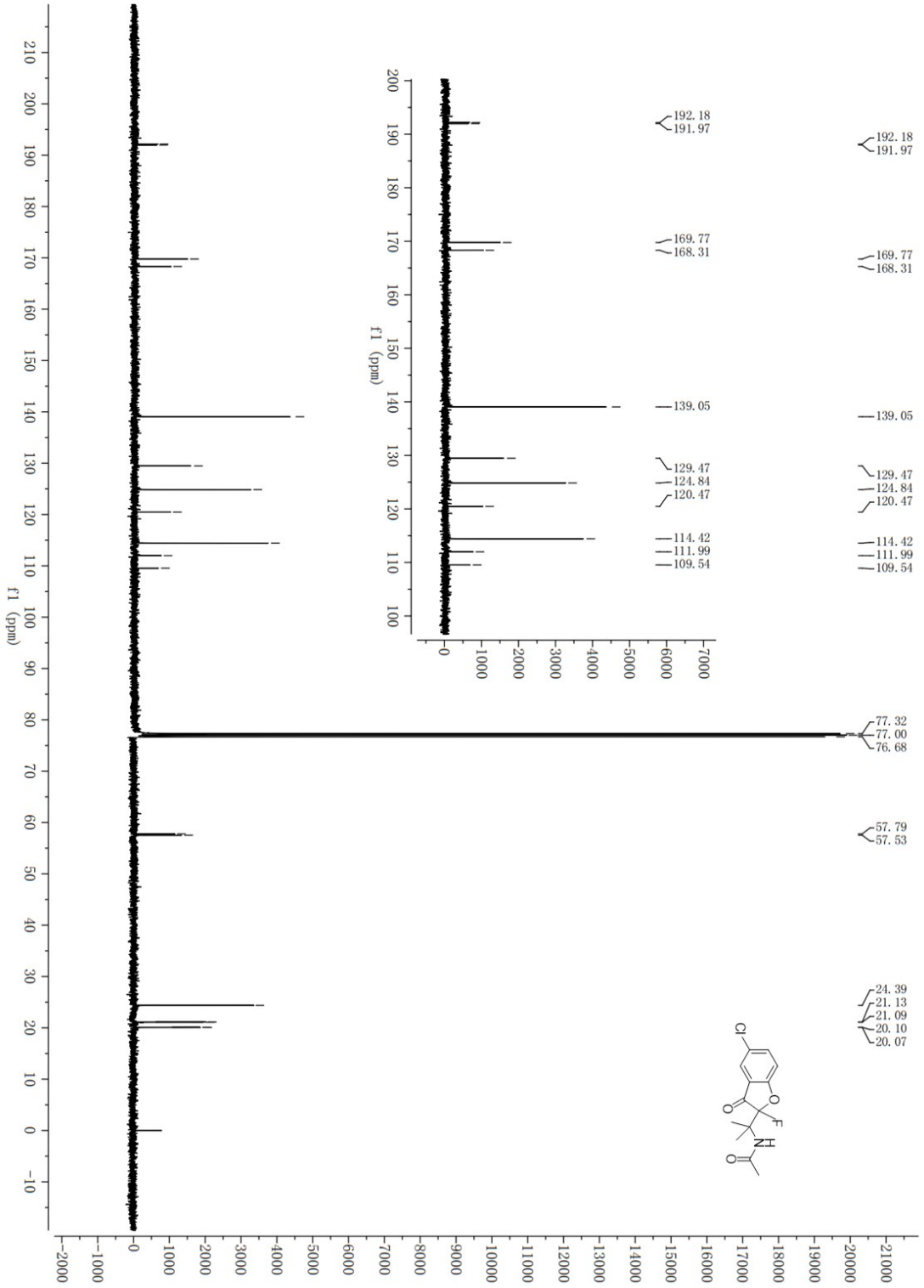
^{1}H NMR


2.23
1.11
1.00
3.09
3.18
3.05
7.81
7.81
7.77
7.77
7.75
7.74
7.26
7.08
7.05
6.17
1.93
1.67
1.47
1.47
f1 (ppm)
Cl
O
F
N
H
O
O

^{19}F NMR

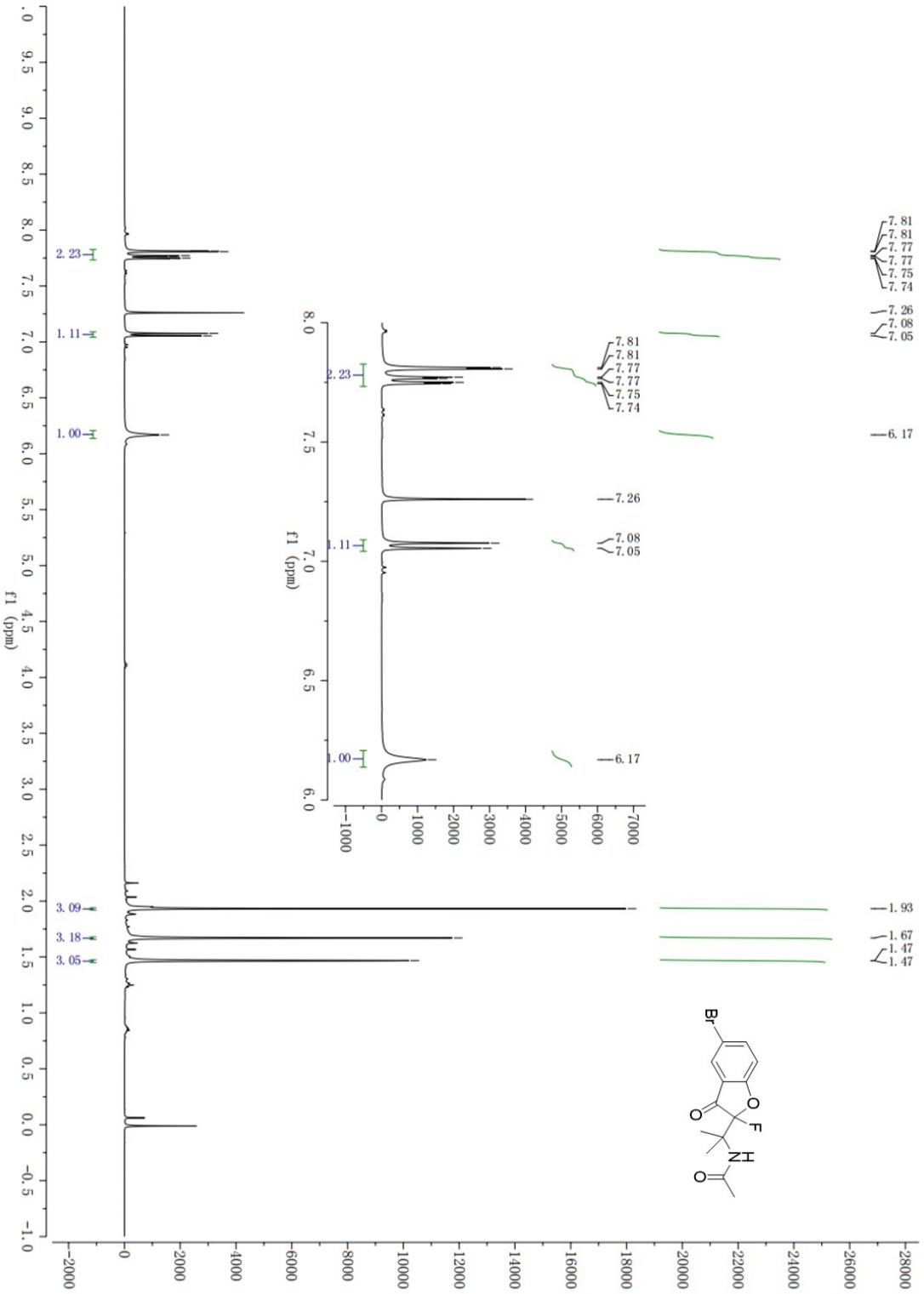


^{13}C NMR

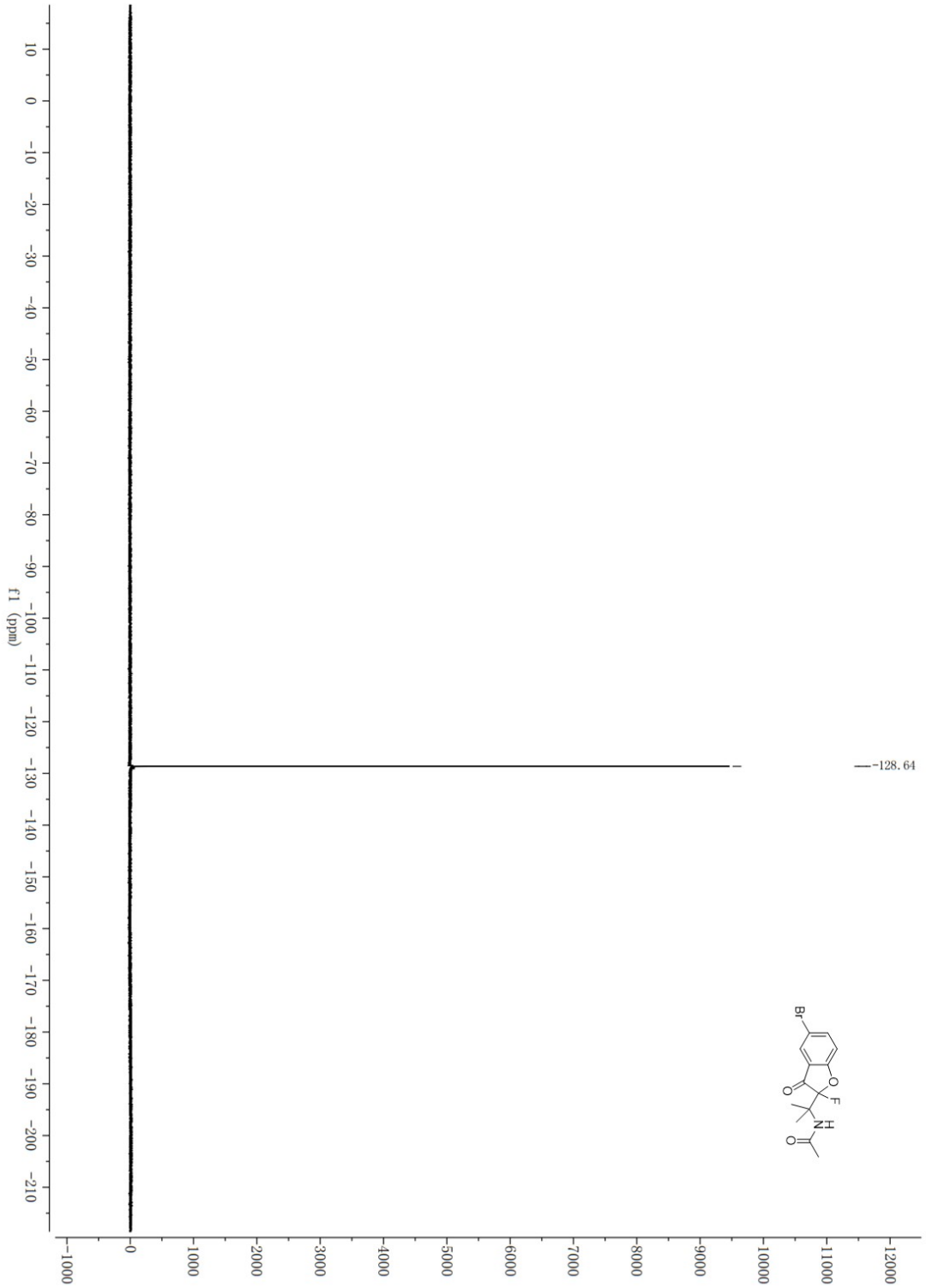

192.18
191.97
169.77
168.31
139.05
129.47
124.84
120.47
114.42
111.99
109.54
77.32
77.00
76.68
57.79
57.53
24.39
21.13
21.09
20.10
20.07
f1 (ppm)
Cl
O
F
N
H
O

(2m)

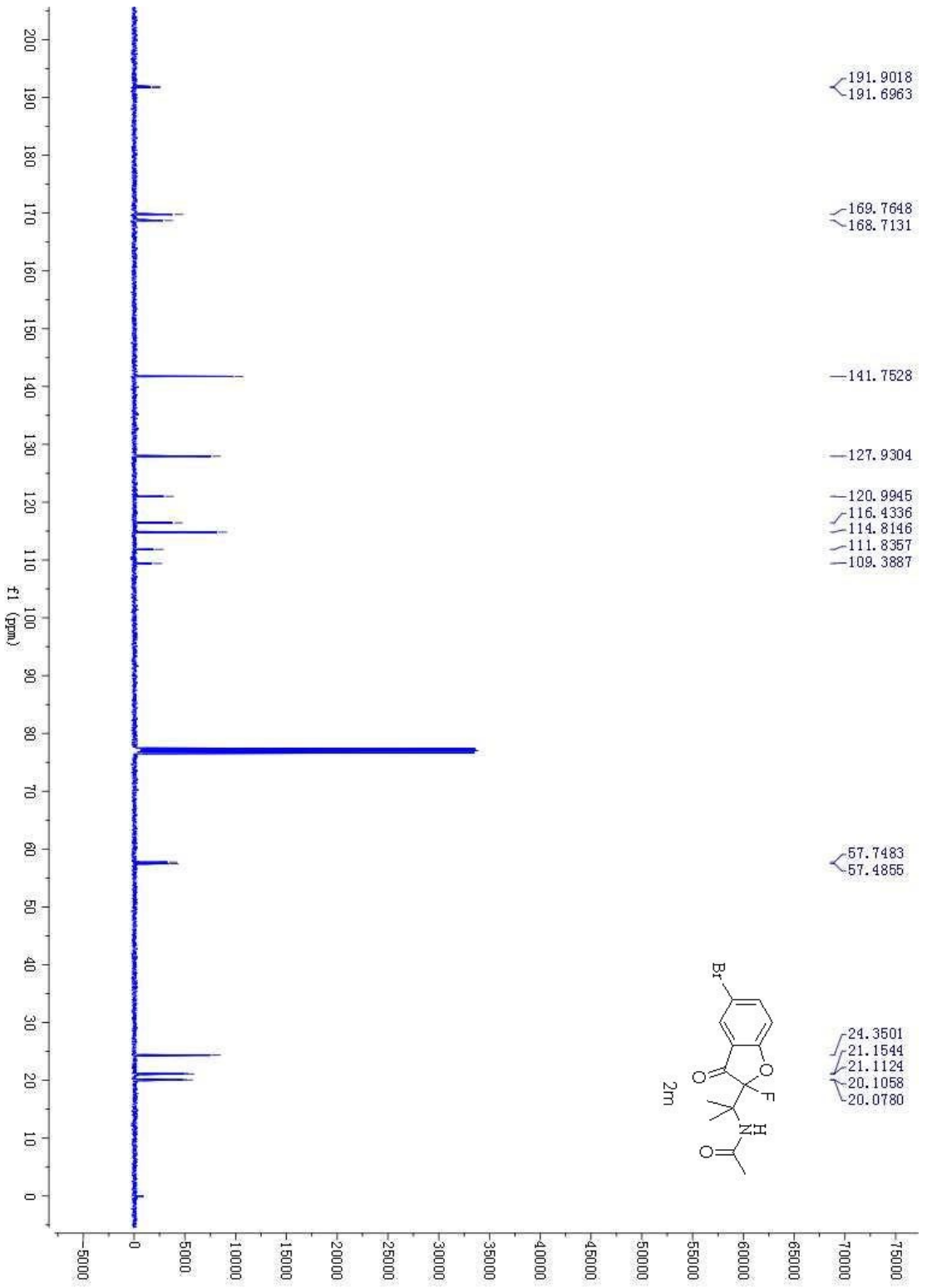
^{1}H NMR


f1 (ppm)
2.23
1.11
1.00
3.09
3.18
3.05
7.81
7.81
7.77
7.77
7.75
7.74
7.26
7.08
7.05
6.17
1.93
1.67
1.47
1.47
Br
O
F
N
H
O

^{19}F NMR

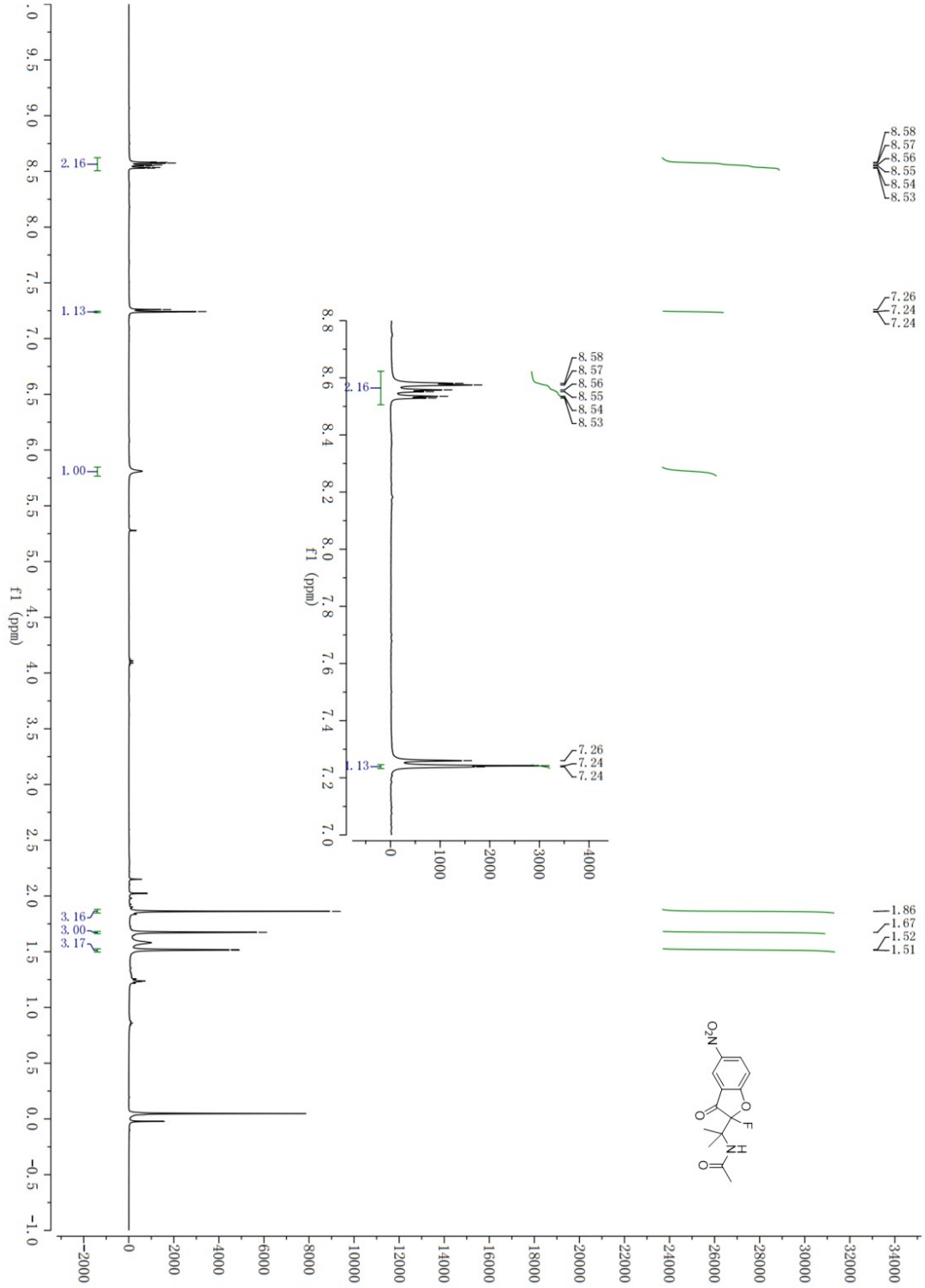


^{13}C NMR

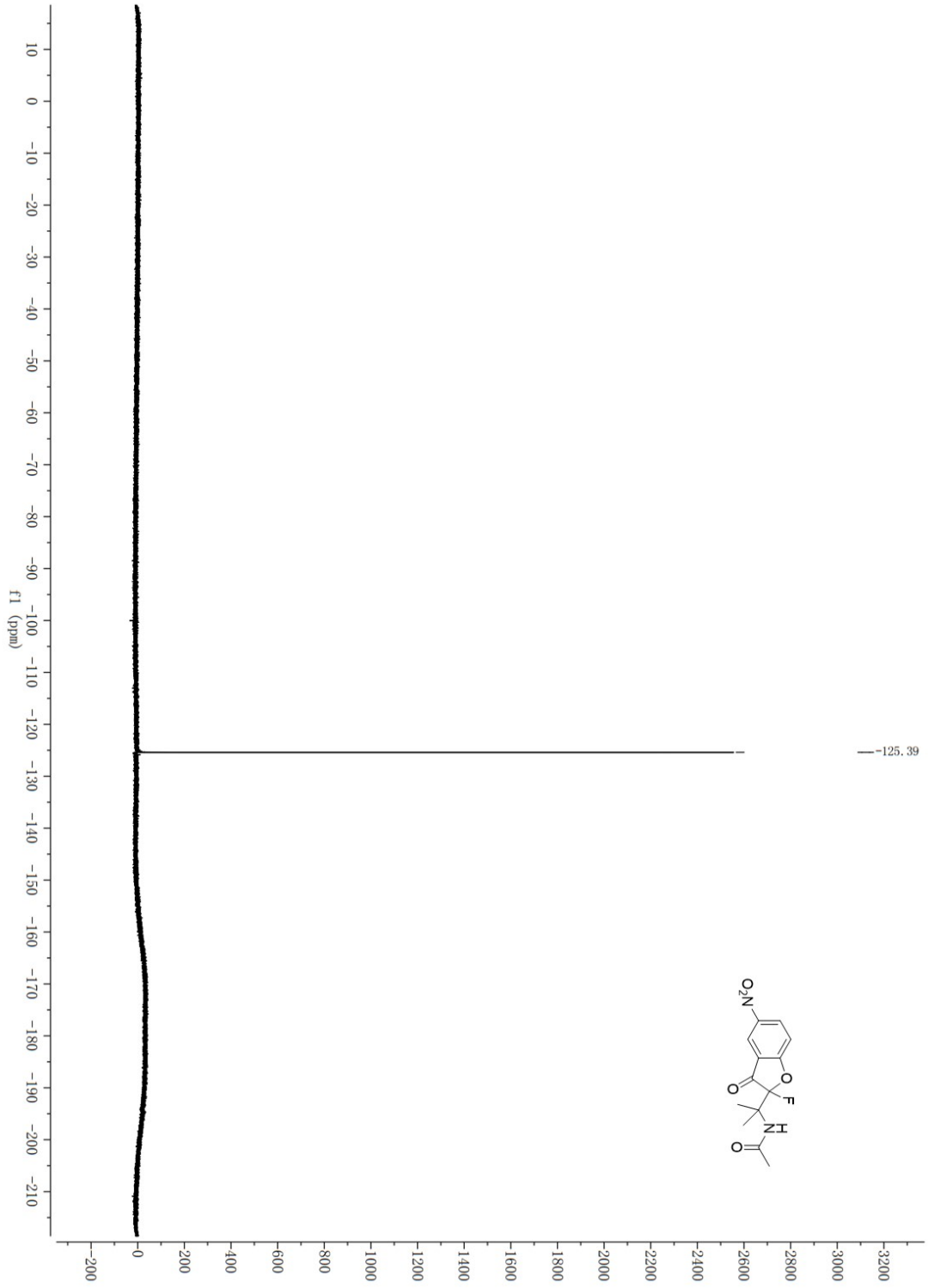

191.9018
191.6963
169.7648
168.7131
141.7528
127.9304
120.9945
116.4336
114.8146
111.8357
109.3887
57.7483
57.4855
24.3501
21.1544
21.1124
20.1058
20.0780
f1 (ppm)
2m

(2n)

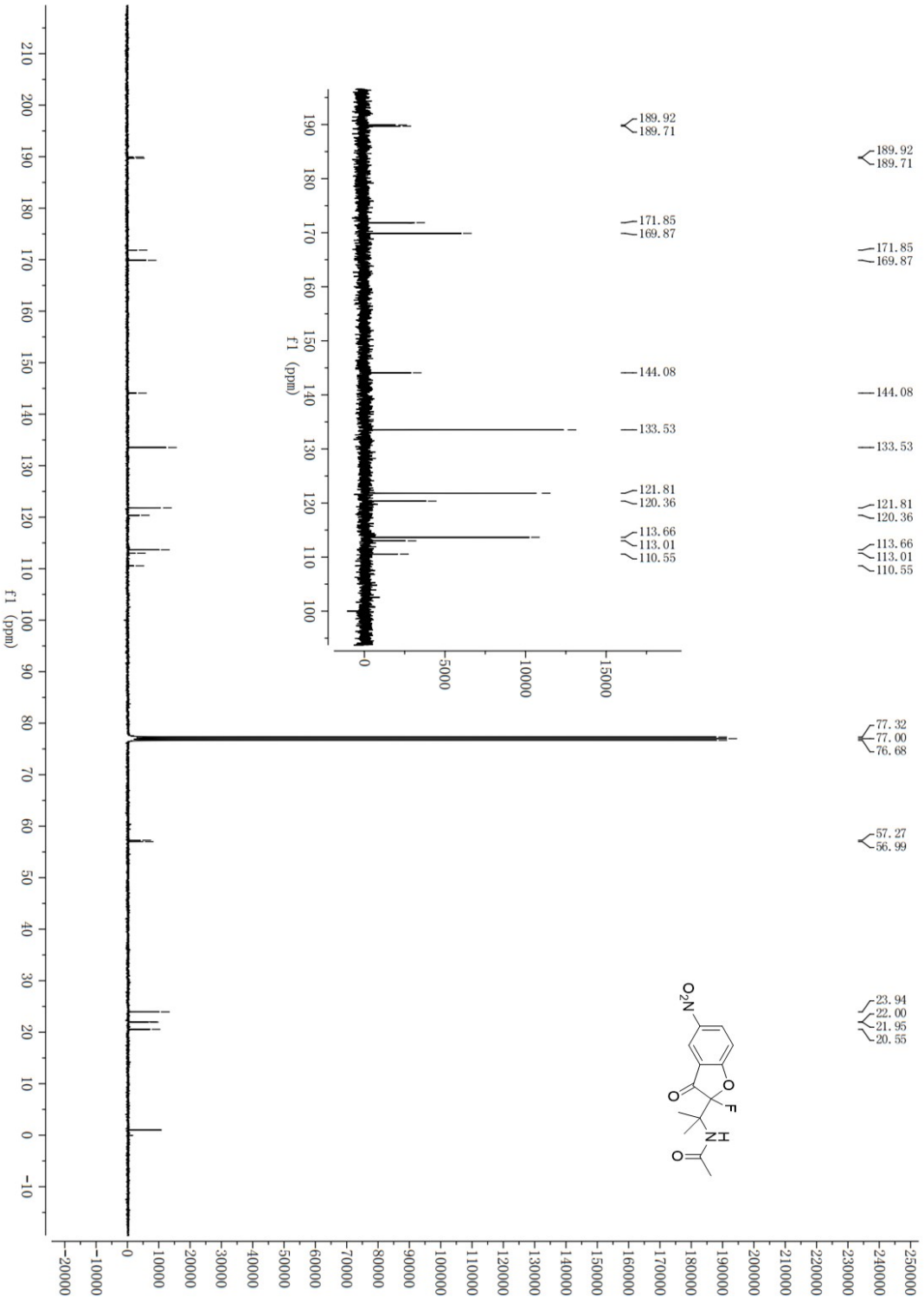
^{1}H NMR


2.16
1.13
1.00
3.16
3.00
3.17
f1 (ppm)
8.58
8.57
8.56
8.55
8.54
8.53
7.26
7.24
7.24
1.86
1.67
1.52
1.51
O2N
O
F
H
N
O

^{19}F NMR

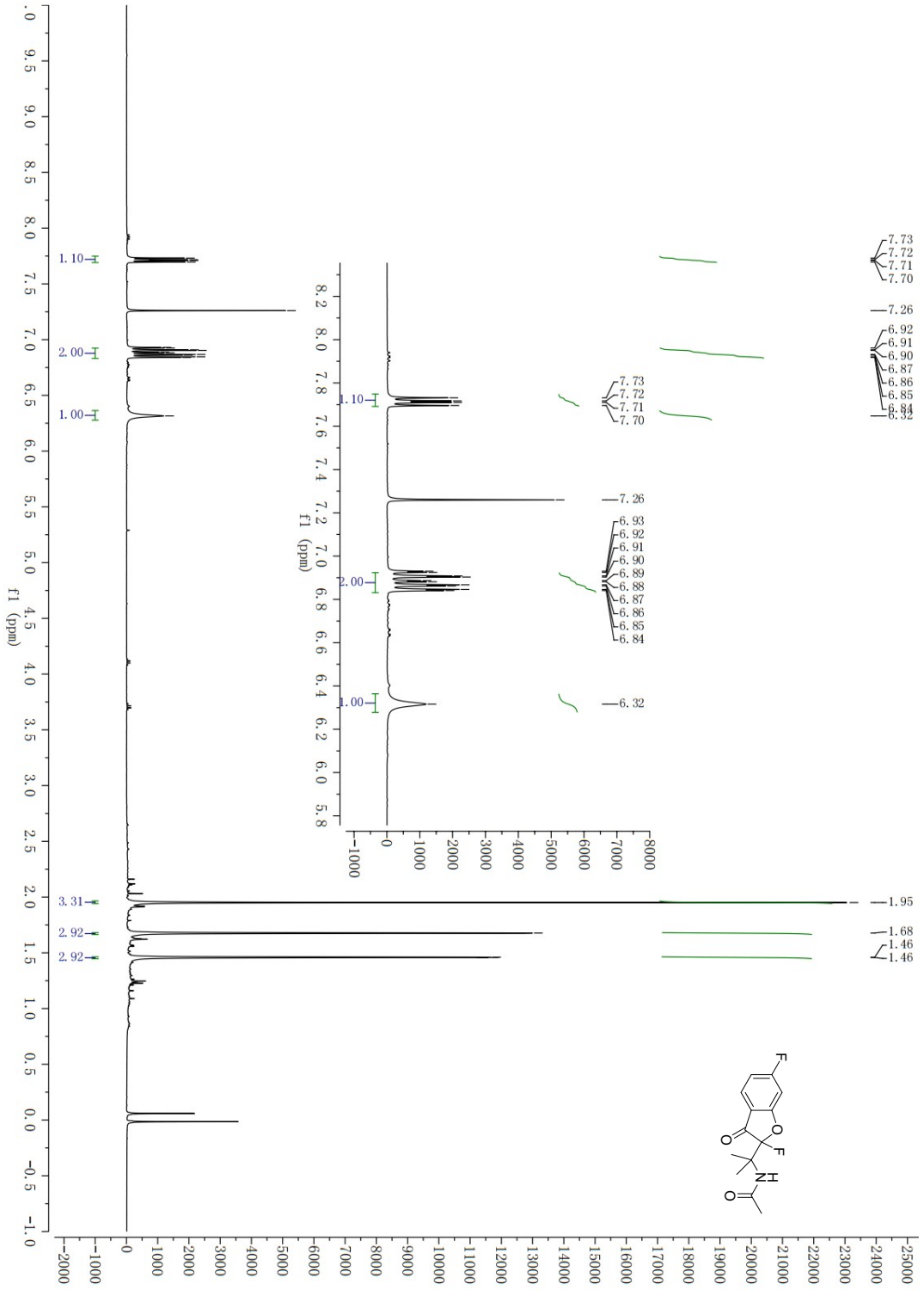


^{13}C NMR

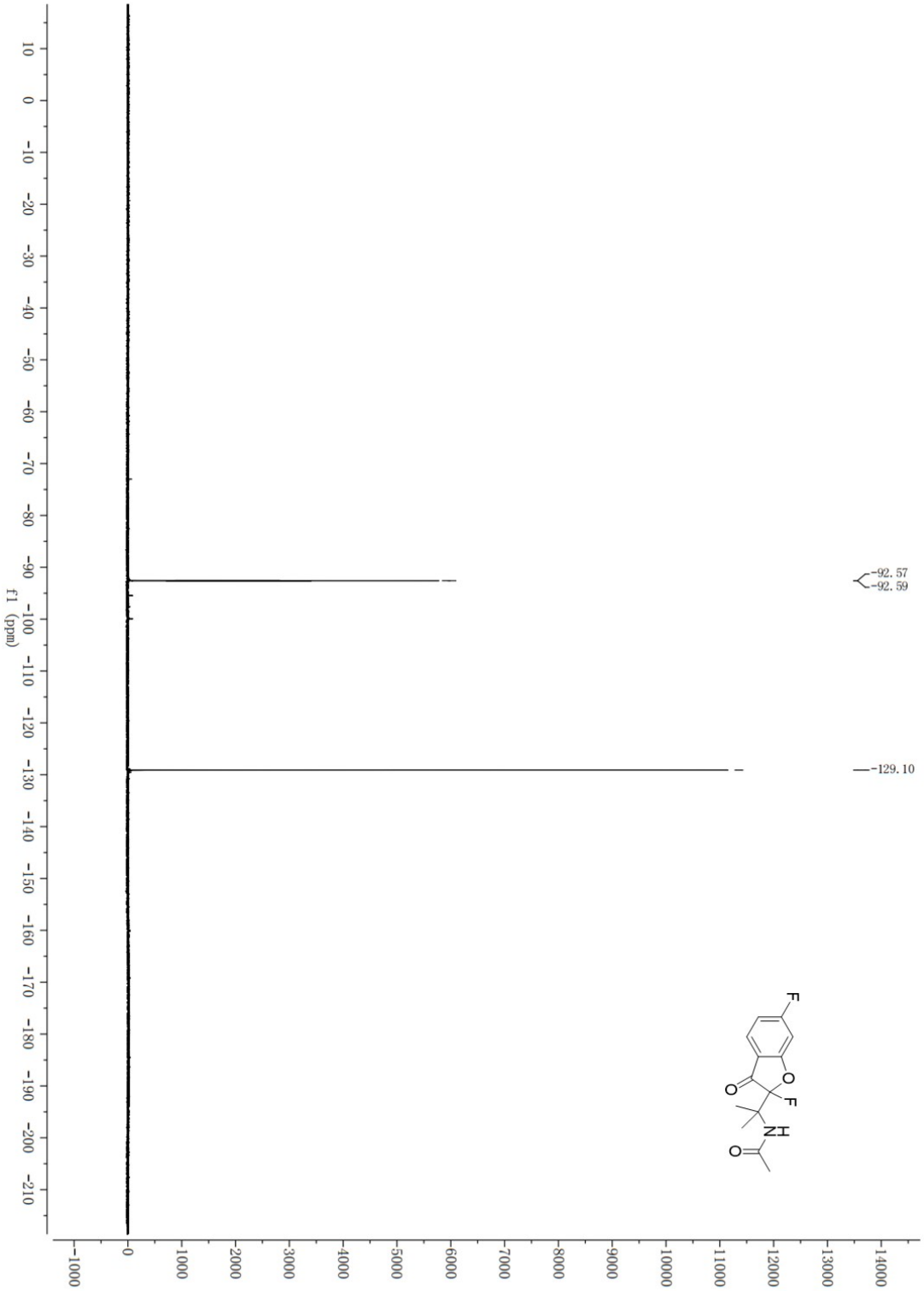

189.92
189.71
171.85
169.87
144.08
133.53
121.81
120.36
113.66
113.01
110.55
77.32
77.00
76.68
57.27
56.99
23.94
22.00
21.95
20.55
f1 (ppm)

(2o)

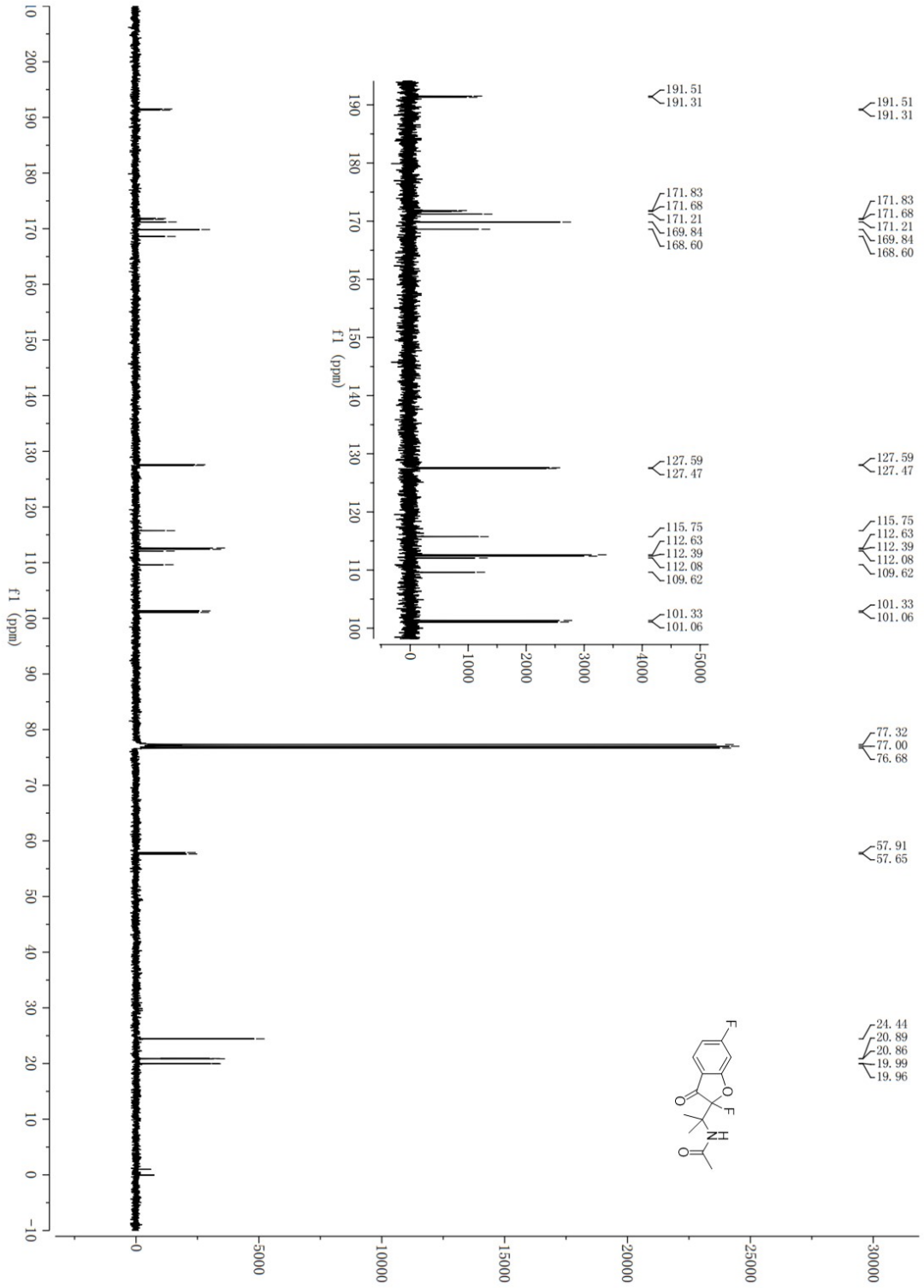
^{1}H NMR


7.73
7.72
7.71
7.70
7.26
6.92
6.91
6.90
6.87
6.86
6.85
6.84
6.32
1.95
1.68
1.46
1.46
f1 (ppm)

^{19}F NMR



^{13}C NMR


191.51
191.31
171.83
171.68
171.21
169.84
168.60
127.59
127.47
115.75
112.63
112.39
112.08
109.62
101.33
101.06
77.32
77.00
76.68
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24.44
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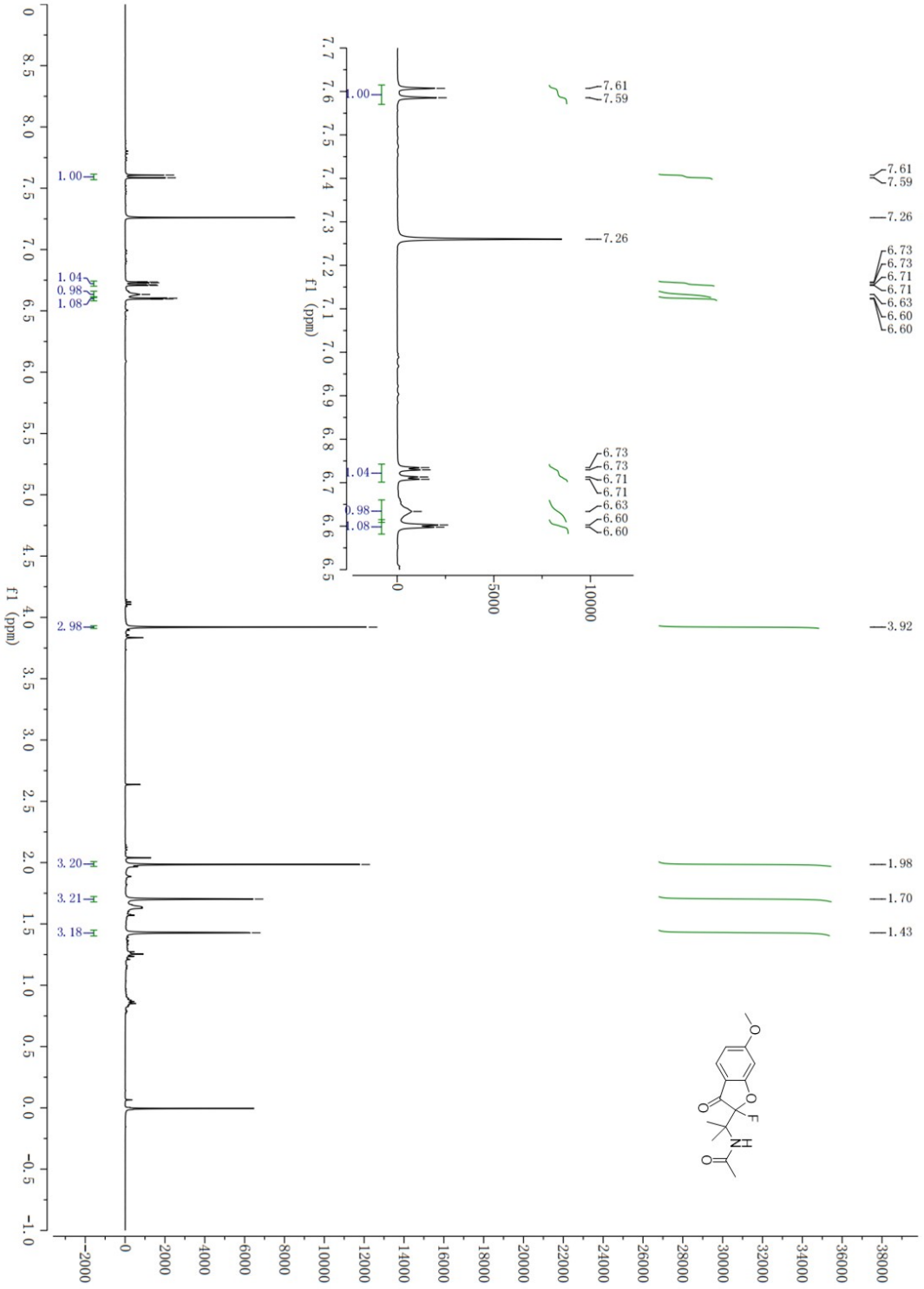
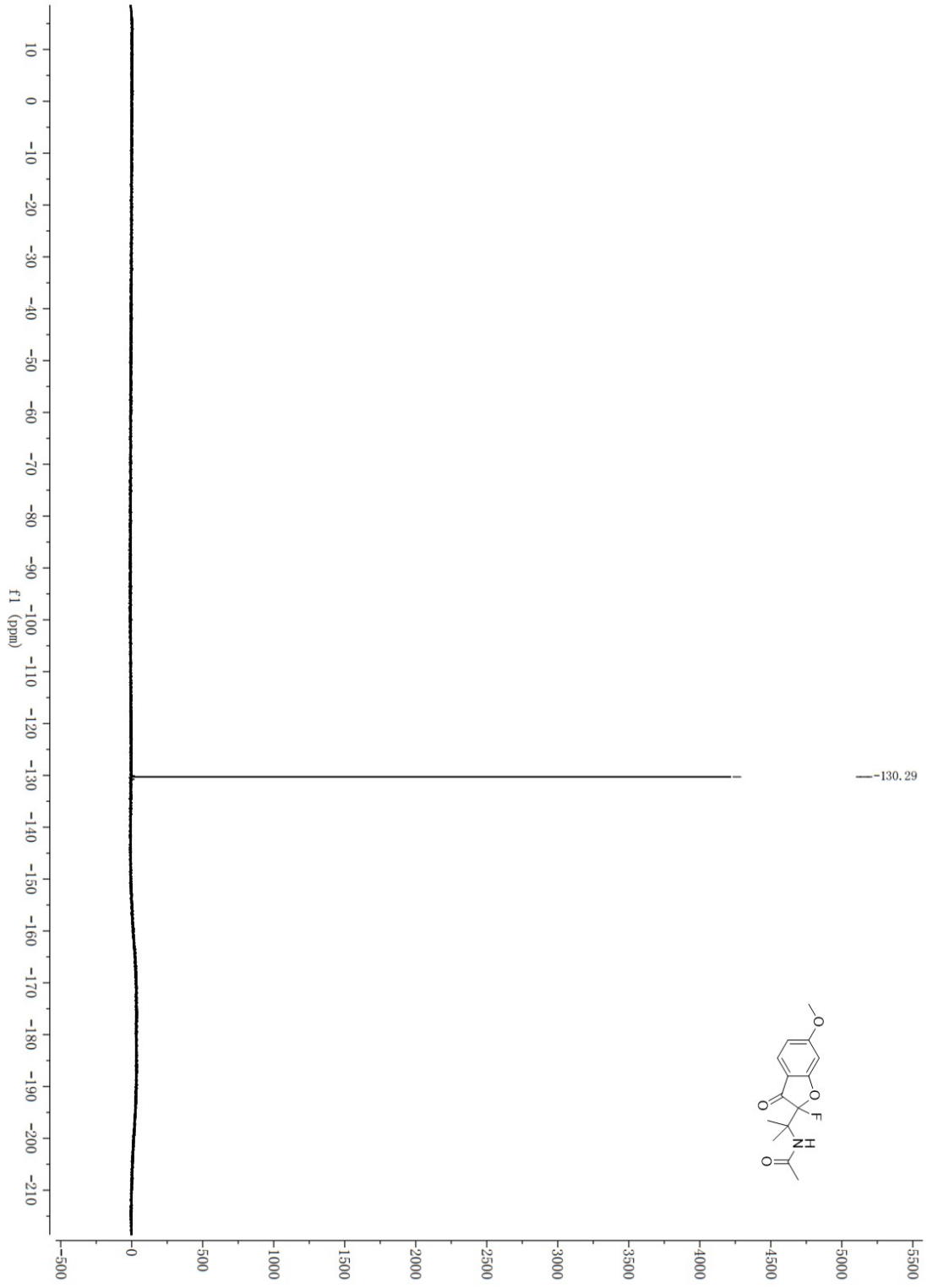
20.86
19.99
19.96
f1 (ppm)

(2p)

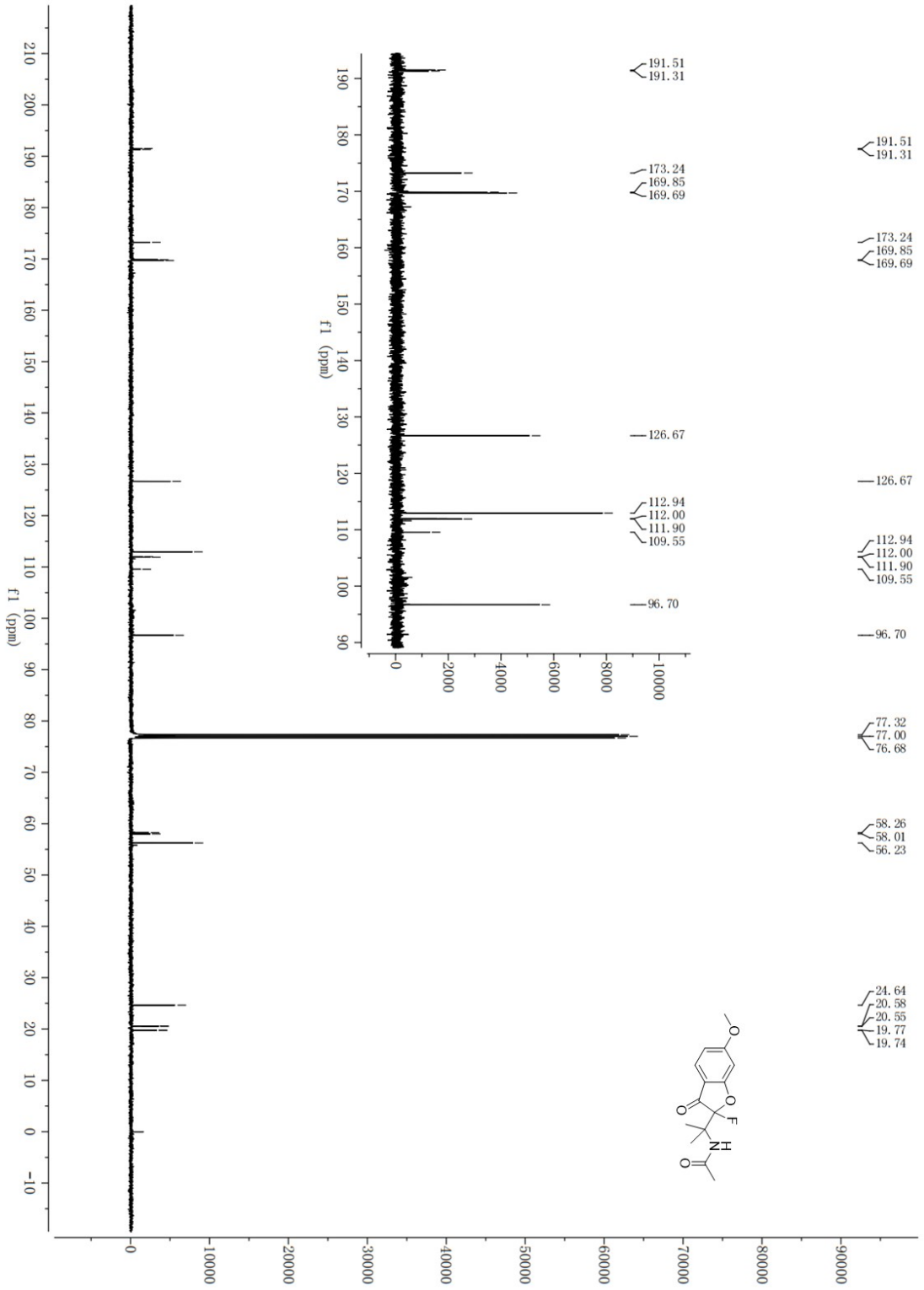
^{1}H NMR


f1 (ppm)
1.00
1.04
0.98
1.08
2.98
3.20
3.21
3.18
7.61
7.59
7.26
6.73
6.73
6.71
6.71
6.63
6.60
6.60
3.92
1.98
1.70
1.43
F
O
N
H

^{19}F NMR



^{13}C NMR


191.51
191.31
173.24
169.85
169.69
126.67
112.94
112.00
111.90
109.55
96.70
77.32
77.00
76.68
58.26
58.01
56.23
24.64
20.58
20.55
19.77
19.74
f1 (ppm)

References

[1] Baum,J.S.;Shook,D.A.;Davies,H.M.L.;Smith,H.D.*Synth.Commun.*,**1987**,*17(14)*,1709-1716.

[2] M. Xiao, F. Zhang, Z. Du and B. Ma, *Adv. Synth. Catal.*, 2018, **360**, 911-916.

[3] J. Gong, Z. Zhao, F. Zhang, S. Wu, G. Yan, Y. Quan, B. Ma, Org. Lett., 16 (2014) 5524-5527.

[4] Margetic, D, Trošelj, P, Ishikawa, T, Kumamoto, T. *Bull. Chem. Soc. Jpn.* 2010, ***83***, 1055-1057.