Supplementary Information for

Nitrene-Triggered Carbocationic Rearrangement of Tetrasubstituted Alkenes Enables Divergent Access to Fused δ-Lactams

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1. General information

Reagents:

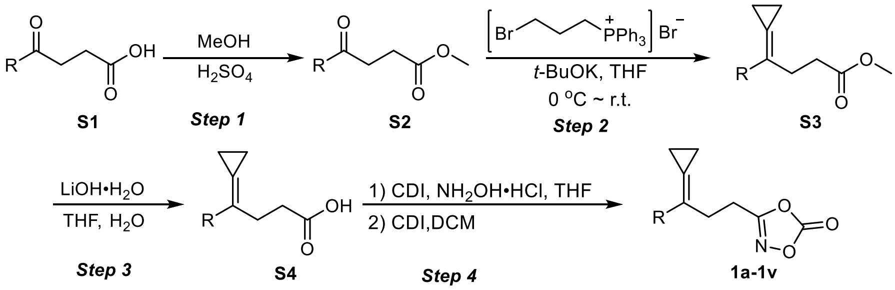
All commercial materials were used as received unless otherwise noted. Analytical thin layer chromatography (TLC) was performed on silica gel Huanghai HSGF254 plates and visualization of the developed chromatogram was performed by fluorescence quenching (λ_{max}=254 nm) or using iodine stain. Flash chromatography was performed using Silica gel (200-300 mesh) purchased from Qingdao Haiyang Chemical Co. China, and SiliCycle Inc. (SiliaFlash P60, 40-63 μm, 60 Å from Canada). $[Cp^*IrCl_2]_2$ was synthesized following a reported procedure using $IrCl_3 \cdot xH_2O$ (J&K Scientific). All starting materials were commercially available (from Le yan, Adamas-beta®, Energy Chemical, J&K Scientific, Macklin, Shanghai Bidepharm, Innochem, Aladdin, Meryer, Sigma-Aldrich, TCI, Alfa Aesar, and FUSION (Lenalidomide, No: FZ-0016359-1g)).

Instruments:

NMR spectra were recorded on Bruker AVANCE AV 400 instruments at **^{1}H NMR** (400 MHz), **^{13}C NMR** (101 MHz), **^{19}F NMR** (376 MHz) and all NMR spectra were processed using MestReNova software, reported in units, parts per million (ppm), using residual solvent peaks (chloroform (δ = 7.26 ppm) or DMSO (δ = 2.50 ppm) or CH_3OH (δ = 3.31 ppm) for **^{1}H NMR**, chloroform (δ = 77.16 ppm) or DMSO (δ 39.52 ppm) or CH_3OH (δ 49.00 ppm) for **^{13}C NMR**) as internal reference. All coupling constants (J values) were reported in Hertz (Hz). Multiplicities were recorded as: s = singlet, d = doublet, dd = doublet of doublet, ddd = doublet of doublet of doublet, dt = doublet of triplet, t = triplet, q = quartet, p = pentet, hept = heptet, m = Multiplet, br = broad. High-resolution ESI mass experiments were operated on a Q Exactive™ Focus Hybrid Quadrupole-Orbitrap™ Mass Spectrometer instrument.

2. Preparation of dioxazolone substrates

General procedure A for preparation of dioxazolone substrates **1a-1v**:



Supplementary Figure 1. The preparation of dioxazolone substrates

The starting substrates **S1** were prepared following the reported literature[1] procedure in a three steps sequence.

Step 1. A 100 mL flask was charged with **S1** (10 mmol, 1.0 equiv.), followed by the addition of MeOH (50 mL) under air. Then, H_2SO_4 (2 mL) was added to the mixture slowly. The reaction mixture was heated to 80 °C under reflux and stirred continuously for 4 hours. Upon completion of the reaction, the solvent was removed under reduced pressure. To the mixture was added DCM (30 mL), followed by quenching with saturated sodium bicarbonate solution until gas evolution ceased. The aqueous phase was extracted with DCM (30 mL × 3). The combined organic layers were washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product **S2** was obtained without further purification.

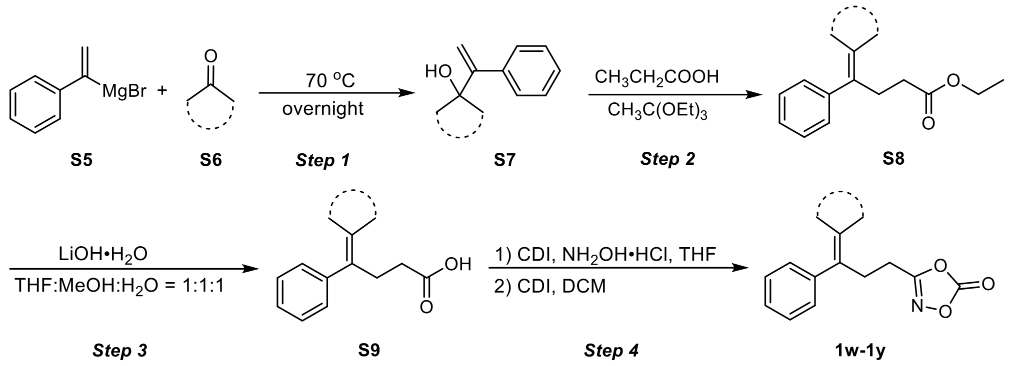
Step 2. A solution of 3-bromopropyltriphenylphosphonium bromide (12 mmol, 1.2 equiv.) and *t*-BuOK (25 mmol, 2.5 equiv.) in 100 mL THF was stirred at 75 °C under Ar for 1 hour. Afterward, a solution of compound **S2** (10 mmol, 1.0 equiv.) in 10 mL THF was added, and the reaction solution was stirred at 75 °C for 8 hours. Upon completion, the reaction was cooled to room temperature, and the mixture was filtered through a Celite. The filtrate was concentrated under reduced pressure, and the residue was purified by silica gel chromatography (EtOAc/hexane) to afford the corresponding products **S3** in 30% to 80% yields.

Note: 3-bromopropyltriphenylphosphonium bromide was commercially available (from *Cat No. 1030808, Leyan, Shanghai, China*).

Step 3. A mixture of **S3** (5.0 mmol) and LiOH·H_2O (25 mmol, 5.0 equiv.) in THF/H_2O (v:v = 1:1, 30 mL), and the reaction mixture was stirred at room temperature for 8 hours. Upon completion of the reaction, the mixture was diluted with ethyl acetate (10 mL) and extracted with water (20 mL × 3). The combined aqueous layers were acidified to pH 2 with 2 M hydrochloric acid and then extracted with dichloromethane (20 mL × 3). The organic extracts were dried over anhydrous Na_2SO_4, concentrated under reduced pressure, and afforded the product **S4** in high yield without further purification.

Step 4. The substrates **1a-1v** were prepared following the reported literature[2] procedure in a two-step sequence. 1,1'-Carbonyldiimidazole (CDI, 6 mmol, 1.5 equiv.) was added to a mixture of carboxylic acid **S4** (4 mmol, 1.0 equiv.) in dry THF (30 mL) at room temperature. The reaction mixture was stirred for 1-2 hours. Afterward, Hydroxylamine hydrochloride (NH_2OH·HCl, 8 mmol, 2.0 equiv.) was added. The resulting mixture was stirred overnight. The reaction mixture was diluted with 5% aq. $KHSO_4$ (20 mL) and extracted with ethyl acetate (15 mL × 2). The combined organic layers were washed with brine, dried over Na_2SO_4, filtrated and concentrated in vacuo. The crude material was purified by silica gel chromatography eluted with DCM/MeOH to afford hydroxamic acid. Then to a stirred solution of hydroxamic acid (1.0, mmol, 1.0 equiv.) in DCM (2 mL), CDI (1.2 mmol, 1.2 equiv.) was added at room temperature. After being stirred for 30 minutes, the reaction mixture was quenched with 1 N HCl (20 mL), and extracted with DCM (15 mL × 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtrated and concentrated. The crude material was purified by silica gel chromatography eluted with petroleum ether/EtOAc to afford product **1a-1v**.

General procedure B for preparation of dioxazolone substrates **1w-1y**:



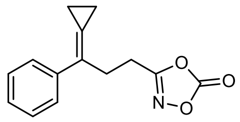
Step 1. Under an argon atmosphere, a 100 mL three-necked flask was charged with **S6** (20 mmol, 1.0 equiv.), followed by the addition of anhydrous THF (30 mL). The mixture was then heated to reflux at 70 °C, and **S5** (22 mmol, 1.1 equiv.) was added dropwise. The reaction mixture was stirred at this temperature overnight. Upon completion, the reaction was quenched with saturated aqueous ammonium chloride. The aqueous phase was extracted with ethyl acetate (30 mL × 3). The combined organic layers were washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (EtOAc/hexane) to afford the corresponding product **S7** in 40 - 60% yield.

Step 2. Under air, **S7** (10 mmol, 1.0 equiv.) was dissolved in triethyl orthoacetate (100 mmol, 10.0 equiv.), and a catalytic amount of propionic acid (1mmol, 0.1 equiv.) was added. The mixture was heated to 150 °C and stirred for approximately 5 hours. Upon completion of the reaction as monitored by TLC, the mixture was quenched with 1N HCl. The aqueous phase was extracted with ethyl acetate (30 mL × 3). The combined organic layers were washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (EtOAc/hexane) to afford the corresponding product **S8** in 30 - 50% yield.

Step 3. A mixture of **S8** (5.0 mmol) and $LiOH \cdot H_2O$ (25 mmol, 5.0 equiv.) in THF/MeOH/H_2O (v:v:v = 1:1:1, 45 mL), and the reaction mixture was stirred at 60 °C for 8 hours. Upon completion of the reaction, the mixture was diluted with ethyl acetate

(10 mL) and extracted with water (20 mL × 3). The combined aqueous layers were acidified to pH 2 with 2 M hydrochloric acid and then extracted with dichloromethane (20 mL × 3). The organic extracts were dried over anhydrous Na_2SO_4, concentrated under reduced pressure, and afforded the product **S9** in high yield without further purification.

Step 4. The substrates **1w-1y** were prepared following the reported literature[2] procedure in a two-step sequence. 1,1'-Carbonyldiimidazole (CDI, 6 mmol, 1.5 equiv.) was added to a mixture of carboxylic acid **S9** (4 mmol, 1.0 equiv.) in dry THF (30 mL) at room temperature. The reaction mixture was stirred for 1-2 hours. Afterward, Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$, 8 mmol, 2.0 equiv.) was added. The resulting mixture was stirred overnight. The reaction mixture was diluted with 5% aq. $KHSO_4$ (20 mL) and extracted with ethyl acetate (15 mL × 2). The combined organic layers were washed with brine, dried over Na_2SO_4, filtrated and concentrated in vacuo. The crude material was purified by silica gel chromatography eluted with DCM/MeOH to afford hydroxamic acid. Then to a stirred solution of hydroxamic acid (1 mmol, 1.0 equiv.) in DCM (2 mL), CDI (1.2 mmol, 1.2 equiv.) was added at room temperature. After being stirred for 30 minutes, the reaction mixture was quenched with 1N HCl (20 mL), and extracted with DCM (15 mL × 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtrated and concentrated. The crude material was purified by silica gel chromatography eluted with petroleum ether/EtOAc to afford product **1w-1y**.



1a

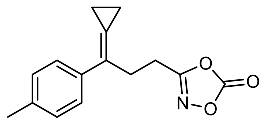
Compound **1a** is a previously known compound and was synthesized in 30% yield following the **general procedure A**.

White solid. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 (d, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 2H), 7.19 – 7.09 (m, 1H), 2.95 (t, *J* = 7.7 Hz, 2H), 2.75 (t, *J* = 7.6 Hz, 2H), 1.30 (t, *J* = 8.0 Hz, 2H), 1.08 (t, *J* = 8.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.51, 154.23, 138.65, 128.66, 127.30, 125.90, 124.29, 123.43, 29.00, 24.05, 4.81, 1.62.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{14}NO_3^+$[M+H]$^+$: 244.0968, found: 244.0970.



1b

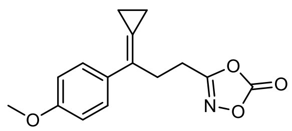
Compound **1b** is a previously unknown compound and was synthesized in 33% yield following the **general procedure A**.

White solid. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 7.9 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 3.08 (t, *J* = 7.7 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.38 (s, 3H), 1.43 (t, *J* = 8.0 Hz, 2H), 1.21 (t, *J* = 8.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.55, 154.24, 137.03, 135.74, 129.32, 125.76, 124.08, 122.33, 28.98, 24.02, 21.16, 4.72, 1.52.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{16}NO_3^+$[M+H]$^+$: 258.1125, found: 258.1128.



1c

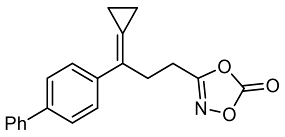
Compound **1c** is a previously unknown compound and was synthesized in 40% yield following the **general procedure A**.

Gray solid. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 3.82 (s, 3H), 3.04 (t, *J* = 7.9 Hz, 2H), 2.88 (t, *J* = 7.6 Hz, 2H), 1.40 (t, *J* = 8.0 Hz, 2H), 1.18 (t, *J* = 8.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.56, 158.86, 154.26, 131.17, 127.00, 123.65, 121.29, 114.02, 55.40, 29.07, 24.06, 4.72, 1.55.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{16}NO_4^+[M+H]^+$: 274.1074, found: 274.1073.



1d

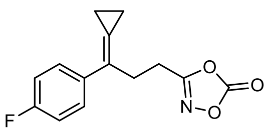
Compound **1d** is a previously unknown compound and was synthesized in 34% yield following the **general procedure A**.

White solid. (R_f = 0.40, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 – 7.58 (m, 6H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 7.3 Hz, 1H), 3.12 (t, *J* = 7.6 Hz, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 1.49 (t, *J* = 8.0 Hz, 2H), 1.24 (t, *J* = 7.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.51, 154.24, 140.67, 140.03, 137.56, 128.93, 127.47, 127.33, 127.05, 126.27, 123.91, 123.64, 28.95, 24.09, 4.94, 1.66.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{18}NO_3^+[M+H]^+$: 320.1281, found: 320.1279.



1e

Compound **1e** is a previously unknown compound and was synthesized in 26% yield following the **general procedure A**.

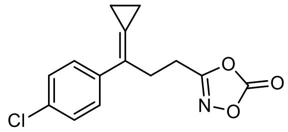
Yellow solid. (R_f = 0.40, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 – 7.48 (m, 2H), 7.09 – 7.01 (m, 2H), 3.05 (t, *J* = 8.0 Hz, 2H), 2.88 (t, *J* = 8.0 Hz, 2H), 1.40 (t, *J* = 8.0 Hz, 2H), 1.22 (t, *J* = 8.0 Hz, 2H).

^{13}**C NMR** (101 MHz, Chloroform-*d*) δ 166.43, 162.02 (d, J = 247.9 Hz), 154.18, 134.82, 127.47 (d, J = 8.0 Hz), 123.33, 123.16 (d, J = 1.9 Hz), 115.46 (d, J = 20.8 Hz), 29.01, 23.88, 4.62, 1.75.

^{19}**F NMR** (376 MHz, Chloroform-*d*) δ -115.13.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{13}FNO_3^+$[M+H]$^+$: 262.0874, found: 262.0872.



1f

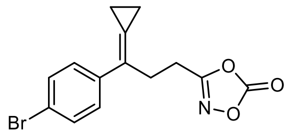
Compound **1f** is a previously unknown compound and was synthesized in 30% yield following the **general procedure A**.

Yellow solid. (R_f = 0.40, PE/EA = 10:1, v/v)

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.47 (d, J = 8.6 Hz, 2H), 7.33 (d, J = 8.6 Hz, 2H), 3.05 (t, J = 8.0 Hz, 2H), 2.88 (t, J = 8.0 Hz, 2H), 1.41 (t, J = 8.0 Hz, 2H), 1.22 (t, J = 8.0 Hz, 2H).

^{13}**C NMR** (101 MHz, Chloroform-*d*) δ 166.35, 154.16, 137.14, 133.00, 128.78, 127.14, 124.21, 123.30, 28.83, 23.90, 4.78, 1.78.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{13}ClNO_3^+$[M+H]$^+$: 278.0578, found: 278.0580.



1g

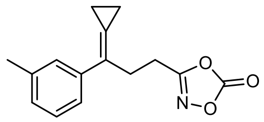
Compound **1g** is a previously unknown compound and was synthesized in 39% yield following the **general procedure A**.

Yellow solid. (R_f = 0.40, PE/EA = 10:1, v/v)

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.48 (d, J = 8.6 Hz, 2H), 7.41 (d, J = 8.6 Hz, 2H), 3.04 (t, J = 7.6 Hz, 2H), 2.88 (t, J = 7.6 Hz, 2H), 1.41 (t, J = 7.6 Hz, 2H), 1.21 (t, J = 7.6 Hz, 2H).

^{13}**C NMR** (101 MHz, Chloroform-*d*) δ 166.33, 154.14, 137.59, 131.73, 127.47, 124.35, 123.36, 121.16, 28.77, 23.89, 4.80, 1.80.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{13}BrNO_3^+$[M+H]$^+$: 322.0073, found: 322.0068.



1h

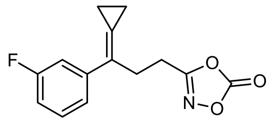
Compound **1h** is a previously unknown compound and was synthesized in 37% yield following the **general procedure A**.

White solid. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.31 (m, 2H), 7.25 – 7.20 (m, 1H), 7.07 (d, *J* = 7.5 Hz, 1H), 3.05 (t, *J* = 8.0 Hz, 2H), 2.86 (t, *J* = 7.6 Hz, 2H), 2.36 (s, 3H), 1.40 (t, *J* = 8.0 Hz, 2H), 1.18 (t, *J* = 8.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.55, 154.26, 138.63, 138.20, 128.56, 128.09, 126.58, 124.35, 123.18, 123.09, 29.08, 24.10, 21.72, 4.81, 1.63.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{16}NO_3^+$[M+H]$^+$: 258.1125, found: 258.1123.



1i

Compound **1i** is a previously unknown compound and was synthesized in 21% yield following the **general procedure A**.

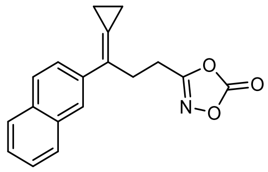
Yellow solid. (R_f = 0.40, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.23 (m, 3H), 6.95 (t, *J* = 8.3 Hz, 1H), 3.04 (t, *J* = 7.7 Hz, 2H), 2.88 (t, *J* = 7.6 Hz, 2H), 1.43 (t, *J* = 7.7 Hz, 2H), 1.23 (d, *J* = 7.9 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.36, 163.21 (d, *J* = 237.4 Hz), 154.18, 141.05, 130.13(d, *J* = 8.4 Hz), 124.97, 123.42(d, *J* = 2.9 Hz), 121.38 (d, *J* = 2.7Hz), 114.17(d, *J* = 21.3 Hz), 112.88 (d, *J* = 22.6 Hz), 28.90, 23.98, 4.89, 1.74.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.95.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{13}FNO_3^+$[M+H]$^+$: 262.0874, found: 262.0872.



1j

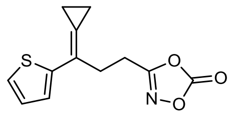
Compound **1j** is a previously unknown compound and was synthesized in 35% yield following the **general procedure A**.

White solid. (R_f = 0.30, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.54 – 9.38 (m, 5H), 9.14 – 9.06 (m, 2H), 4.81 (t, *J* = 7.7 Hz, 2H), 4.55 (t, *J* = 8.0 Hz, 2H), 3.12 (t, *J* = 8.0 Hz, 2H), 2.87 (d, *J* = 12.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.53, 154.24, 136.03, 133.54, 132.67, 128.23, 128.20, 127.68, 126.43, 126.09, 124.62, 124.33, 124.27, 124.00, 29.05, 24.11, 5.15, 1.72.

HRMS (ESI) *m/z* Calcd for $C_{18}H_{16}NO_3^+$[M+H]$^+$: 294.1125, found: 294.1128.



1k

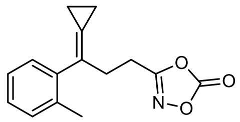
Compound **1k** is a previously unknown compound and was synthesized in 26% yield following the **general procedure A**.

Yellow solid. (R_f= 0.30, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.19 (m, 1H), 7.09 – 6.96 (m, 2H), 3.07 – 2.98 (m, 4H), 1.41 – 1.30 (m, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.38, 154.18, 144.53, 127.21, 124.92, 122.07, 122.04, 120.00, 28.80, 23.95, 5.00, 3.31.

HRMS (ESI) *m/z* Calcd for $C_{12}H_{12}NO_3S^+$[M+H]$^+$: 250.0532, found: 250.0528.



1l

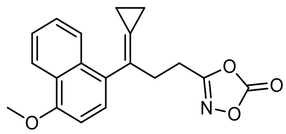
Compound **1l** is a previously unknown compound and was synthesized in 27% yield following the **general procedure A**.

White solid. (R_f= 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.16 – 7.05 (m, 3H), 7.05 – 6.97 (m, 1H), 2.84 (t, J = 7.5 Hz, 2H), 2.69 (t, J = 7.4 Hz, 2H), 2.14 (s, 3H), 1.16 (t, J = 8.4 Hz, 2H), 0.96 (t, J = 7.6 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.54, 154.19, 140.19, 135.76, 130.50, 128.50, 127.32, 125.82, 125.75, 123.65, 31.60, 23.63, 19.92, 3.29, 2.98.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{16}NO_3^+$[M+H]$^+$: 258.1125, found: 258.1134.



1m

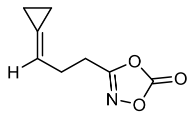
Compound **1m** is a previously unknown compound and was synthesized in 40% yield following the **general procedure A**.

Gray solid. (R_f = 0.50, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.39 – 8.32 (m, 1H), 7.80 – 7.72 (m, 1H), 7.52 (dt, J = 6.8, 3.4 Hz, 2H), 7.28 (s, 1H), 6.85 (d, J = 7.8 Hz, 1H), 4.05 (s, 3H), 3.10 (t, J = 7.5 Hz, 2H), 2.80 (t, J = 7.5 Hz, 2H), 1.37 (t, J = 8.0 Hz, 2H), 1.07 (t, J = 8.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.59, 155.00, 154.24, 132.28, 130.71, 126.57, 125.93, 125.72, 125.24, 125.23, 124.88, 124.62, 122.58, 103.34, 55.63, 32.32, 23.82, 3.38, 3.31.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{18}NO_4^+$[M+H]$^+$: 324.1230, found: 324.1237.



1n

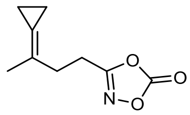
Compound **1n** is a previously unknown compound and was synthesized in 15% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.79 – 5.75 (m, 1H), 2.79 (t, *J* = 7.4 Hz, 2H), 2.61 – 2.56 (m, 2H), 1.05 (s, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.57, 154.30, 124.93, 114.04, 26.74, 24.65, 2.29, 2.15.

HRMS (ESI) *m/z* Calcd for $C_8H_{10}NO_3^+[M+H]^+$: 168.0655, found: 168.0647.



1o

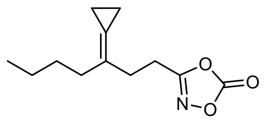
Compound **1o** is a previously unknown compound and was synthesized in 30% yield following the **general procedure A**.

Colorless oil. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.87 (t, *J* = 7.6 Hz, 2H), 2.55 (t, *J* = 7.7 Hz, 2H), 1.84 (s, 3H), 1.11 – 1.04 (m, 2H), 1.00 – 0.94 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.79, 154.33, 120.64, 118.09, 31.32, 23.19, 20.74, 3.32, 1.42.

HRMS (ESI) *m/z* Calcd for $C_9H_{12}NO_3^+[M+H]^+$: 182.0812, found: 182.0819.



1p

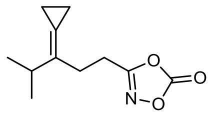
Compound **1p** is a previously unknown compound and was synthesized in 22% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.85 (t, *J* = 7.6 Hz, 2H), 2.52 (t, *J* = 7.8 Hz, 2H), 2.15 (t, *J* = 7.7 Hz, 2H), 1.48 – 1.40 (m, 2H), 1.27 (dt, *J* = 14.9, 7.4 Hz, 2H), 1.01 (d, *J* = 5.2 Hz, 4H), 0.88 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.88, 154.33, 124.75, 117.49, 34.88, 29.89, 29.68, 23.39, 22.58, 14.03, 2.46, 1.74.

HRMS (ESI) *m/z* Calcd for $C_{12}H_{18}NO_3^+$[M+H]$^+$: 224.1281, found: 224.1284.



1q

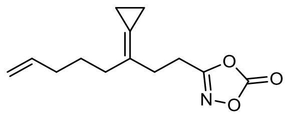
Compound **1q** is a previously unknown compound and was synthesized in 35% yield following the **general procedure A**.

Yellow solid. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.92 – 2.85 (m, 2H), 2.57 – 2.52 (m, 2H), 2.50 – 2.43 (m, 1H), 1.24 – 1.19 (m, 1H), 1.09 (s, 3H), 1.08 (s, 4H), 1.00 – 0.95 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.94, 154.36, 130.13, 115.48, 34.11, 28.07, 23.69, 21.49, 2.13, 1.15.

HRMS (ESI) *m/z* Calcd for $C_{11}H_{16}NO_3^+$[M+H]$^+$: 210.1125, found: 210.1116.



1r

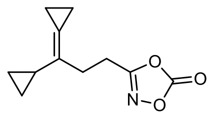
Compound **1r** is a previously unknown compound and was synthesized in 19% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.85 – 5.75 (m, 1H), 5.04 – 4.91 (m, 2H), 2.87 (t, *J* = 8.6 Hz, 2H), 2.55 (t, *J* = 8.0 Hz, 2H), 2.18 (t, *J* = 7.8 Hz, 2H), 2.03 (q, *J* = 7.1 Hz, 2H), 1.63 – 1.59 (m, 2H), 1.03 (s, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.81, 154.28, 138.64, 124.40, 117.79, 114.73, 34.57, 33.54, 29.70, 26.91, 23.35, 2.39, 1.81, 1.09.

HRMS (ESI) *m/z* Calcd for $C_{13}H_{18}NO_3^+$[M+H]$^+$: 236.1281, found: 236.1284.



1s

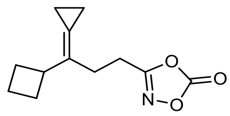
Compound **1s** is a previously unknown compound and was synthesized in 23% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.91 (t, *J* = 7.6 Hz, 2H), 2.54 (t, *J* = 7.7 Hz, 2H), 1.41 – 1.34 (m, 1H), 1.06 (t, *J* = 7.6 Hz, 2H), 0.92 (t, *J* = 7.6 Hz, 2H), 0.69 – 0.52 (m, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.80, 154.33, 125.43, 115.93, 30.15, 23.74, 15.15, 5.22, 2.05, 0.57.

HRMS (ESI) *m/z* Calcd for $C_{11}H_{14}NO_3^+$[M+H]$^+$: 208.0968, found: 208.0965.



1t

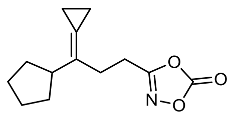
Compound **1t** is a previously unknown compound and was synthesized in 23% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.10 – 3.01 (m, 1H), 2.83 (t, *J* = 8.0 Hz, 2H), 2.49 (t, *J* = 7.8 Hz, 2H), 2.12 – 2.02 (m, 4H), 1.94 – 1.83 (m, 1H), 1.78 – 1.69 (m, 1H), 1.15 – 1.09 (m, 2H), 0.99 – 0.90 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.88, 154.34, 127.65, 116.00, 40.77, 28.39, 27.73, 23.50, 18.50, 2.22, 0.36.

HRMS (ESI) *m/z* Calcd for $C_{12}H_{16}NO_3^+$[M+H]$^+$: 222.1125, found: 222.1126.



1u

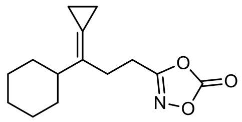
Compound **1u** is a previously unknown compound and was synthesized in 29% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.89 (t, *J* = 7.8 Hz, 2H), 2.55 (t, *J* = 7.8 Hz, 3H), 1.86 – 1.78 (m, 2H), 1.70 – 1.63 (m, 2H), 1.62 – 1.46 (m, 4H), 1.11 – 1.03 (m, 2H), 1.01 – 0.93 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.92, 154.32, 127.81, 115.81, 45.78, 31.26, 29.28, 25.13, 23.70, 2.50, 1.25.

HRMS (ESI) *m/z* Calcd for $C_{13}H_{18}NO_3^+$[M+H]$^+$: 236.1281, found: 236.1284.



1v

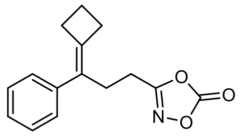
Compound **1v** is a previously unknown compound and was synthesized in 18% yield following the **general procedure A**.

Light yellow oil. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.87 (t, *J* = 7.7 Hz, 2H), 2.54 (t, *J* = 7.7 Hz, 2H), 2.09 – 2.00 (m, 1H), 1.79 – 1.70 (m, 4H), 1.70 – 1.64 (m, 1H), 1.36 – 1.19 (m, 5H), 1.08 (t, *J* = 8.0 Hz, 2H), 0.96 (t, *J* = 7.6 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.93, 154.35, 129.54, 115.96, 44.39, 31.89, 28.71, 26.79, 26.37, 23.79, 2.39, 1.14.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{20}NO_3^+$[M+H]$^+$: 250.1438, found: 250.1428.



1w

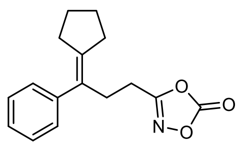
Compound **1w** is a previously known compound and was synthesized in 20% yield following the **general procedure B**.

Yellow liquid. (R_f = 0.60, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 (t, *J* = 8.6 Hz, 2H), 7.14 – 7.10 (m, 3H), 2.74 – 2.63 (m, 6H), 2.50 (t, *J* = 7.4 Hz, 1H), 1.90 (p, *J* = 7.8 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.39, 154.18, 141.30, 138.04, 128.48, 127.47, 127.26, 126.73, 31.72, 30.53, 25.91, 23.96, 16.88.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{16}NO_3^+$[M+H]$^+$: 258.1125, found: 258.1131.



1x

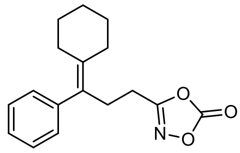
Compound **1x** is a previously known compound and was synthesized in 18% yield following the **general procedure B**.

Yellow liquid. (R_f = 0.70, PE/EA = 10:1, v/v)

^{1}H NMR (400 MHz, Chloroform-*d*) 7.20 (t, *J* = 7.5 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 7.04 (d, *J* = 6.9 Hz, 2H), 2.68 (t, *J* = 7.5 Hz, 2H), 2.45 (t, *J* = 7.5 Hz, 2H), 2.27 (t, *J* = 7.4 Hz, 2H), 2.05 (t, *J* = 7.2 Hz, 2H), 1.63 (p, *J* = 7.0 Hz, 2H), 1.47 (p, *J* = 7.0 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.50, 154.14, 143.51, 141.62, 128.42, 128.38, 128.06, 126.67, 32.56, 30.73, 29.95, 26.81, 26.45, 23.65.

HRMS (ESI) *m/z* Calcd for $C_{16}H_{18}NO_3^+$[M+H]$^+$: 272.1281, found: 272.1279.



1y

Compound **1y** is a previously unknown compound and was synthesized in 22% yield following the **general procedure B**.

Yellow liquid. (R_f = 0.70, PE/EA = 10:1, v/v)

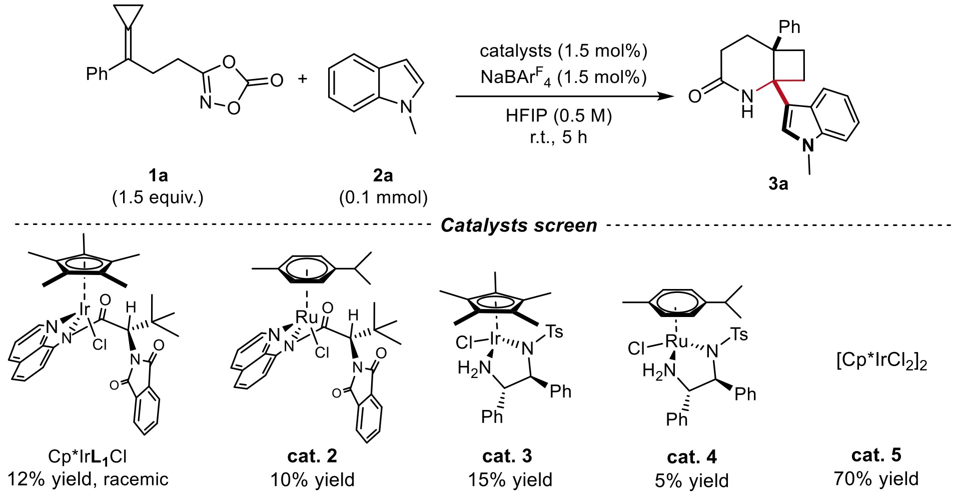
^{1}H NMR (400 MHz, Chloroform-*d*) 7.25 (t, *J* = 8.2 Hz, 2H), 7.19 – 7.13 (m, 1H), 7.03 – 6.97 (m, 2H), 2.71 (t, *J* = 7.4 Hz, 2H), 2.46 (t, *J* = 7.4 Hz, 2H), 2.20 (t, *J* = 5.5 Hz, 2H), 1.93 – 1.84 (m, 2H), 1.52 – 1.47 (m, 4H), 1.40 – 1.33 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.29, 154.12, 141.69, 139.50, 129.08, 128.37, 128.28, 126.65, 32.26, 30.55, 28.71, 28.50, 28.43, 26.69, 23.95.

HRMS (ESI) *m/z* Calcd for $C_{17}H_{20}NO_3^+$[M+H]$^+$: 286.1438, found: 286.1437.

3. Optimization of reaction conditions

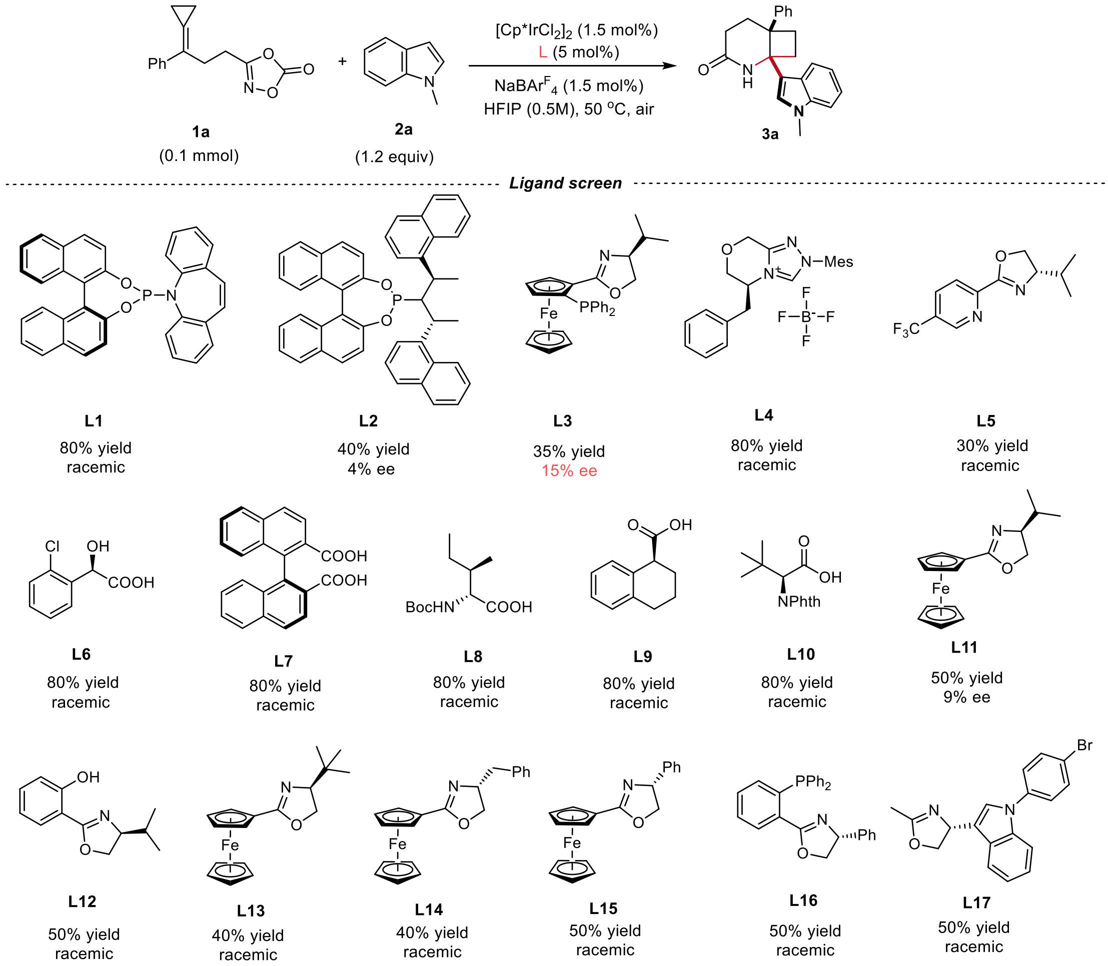
3.1 Screening of chiral catalysts



Supplementary Figure 2. Screening of chiral catalysts

Initially, the reaction between methylenecyclopropane-containing 1,4,2-dioxazolone **1a** and *N*-methylindole **2a** was examined in the presence of Cp*Ir**L1**Cl. Under these conditions, the cyclobutane-fused δ-lactam **3a** was obtained in 12% yield. Unfortunately, lactam **3a** was formed as a racemate. Subsequently, several alternative chiral catalysts were tested (**cat. 2**~**cat. 4**), which resulted in messy reaction systems and only low yields of the desired product. Notably, the use of the parent [Cp*IrCl$_2$]$_2$ as the catalyst led to a significant improvement, affording **3a** in 70% yield. Based on this observation, we proposed that the combination of [Cp*IrCl$_2$]$_2$ with a chiral ligand could potentially achieve the asymmetric synthesis of **3a**.

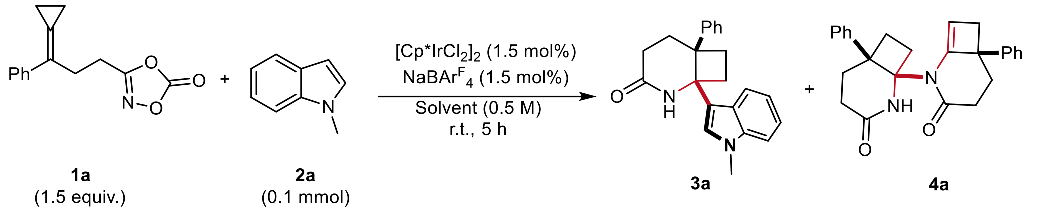
3.2 Screening of chiral ligands



Supplementary Figure 3. Screening of chiral ligands

Using the parent $[Cp^*IrCl_2]_2$ as the catalyst, we evaluated a range of chiral ligands. However, the introduction of chiral ligands led to complex reaction mixtures and a substantial reduction in product yield. Moreover, no enhancement in the enantiomeric excess of the product was achieved, with the product remaining essentially racemic. To date, an effective method for the asymmetric synthesis of **3a** has not been established.

3.3 Optimization of reaction conditions



Supplementary Figure 4. Optimization of reaction conditions

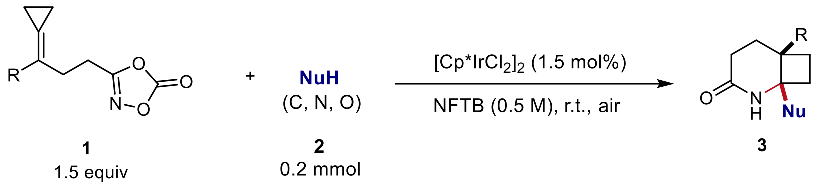
Entry	Catalyst	Solvent	Temperature	**3a** (%)	**4a** (%)
1	Cp*Ir**L1**Cl	HFIP	r.t.	12	ND
2	$[Cp^*IrCl_2]_2$	HFIP	r.t.	70	ND
3	$[Cp^*IrCl_2]_2$	$CHCl_3$	r.t.	41	20
4	$[Cp^*IrCl_2]_2$	TCE	r.t.	38[d]	20
5	$[Cp^*IrCl_2]_2$	DCM	r.t.	13	54
6	$[Cp^*IrCl_2]_2$	TFE	r.t.	76	ND
7	$[Cp^*IrCl_2]_2$	NFTB	r.t.	89	ND
8	PcFe	NFTB	r.t.	30	Trace
9	$FeCl_2$	NFTB	r.t.	ND	Trace
10	[Ru(*p*-cymene)$Cl_2]_2$	NFTB	r.t.	22	Trace
11	CuOAc	NFTB	r.t.	62	ND
12	$[Cp^*RhCl_2]_2$	NFTB	r.t.	ND	ND
13	w/o $[Cp^*IrCl_2]_2$	NFTB	r.t.	ND	ND
14	w/o $NaBAr^F{}_4$	NFTB	r.t.	97[b]	ND
15	$[Cp^*IrCl_2]_2$ (0.5 mol%)	NFTB	r.t.	96	ND
16	$[Cp^*IrCl_2]_2$, 20 min	NFTB	r.t.	87	ND
17	$[Cp^*IrCl_2]_2$	NFTB	0 °C	60	ND
18	$[Cp^*IrCl_2]_2$	NFTB	50 °C	98	ND
19	$[Cp^*IrCl_2]_2$	NFTB	r.t.	ND	60[b]
20	±$[Cp^*IrCl_2]_2$, using **4a** replace **1a**	NFTB	r.t.	ND	>95[c]

[a] Yields are based on ^{1}**H NMR** analysis of the reaction mixture on a 0.1 mmol scale using 1,1,2,2-tetrachloroethane as internal standard. [b] Isolated yield. [c] Isolated the recovered of **4a**. [d] Using dibromomethane as internal standard. TCE, 1,1,2,2-tetrachloroethane; DCM, dichloromethane; TFE, trifluoroethanol; HFIP, hexafluoro-2-propanol; NFTB, nonafluoro-tert-butanol; ND, not detected.

Supplementary Table 1. Evaluation of different reaction conditions

General experimental procedure: All reactions were performed on a 0.1 mmol scale. To an oven-dried screwed 4 mL glass vial equipped with a rod-like-shaped Teflon stir bar were added $[Cp^*IrCl_2]_2$ catalyst (0.0015 mmol, 1.5 mol%), $NaBAr^F_4$ (1.3 mg, 0.0015 mmol, 1.5 mol%), *N*-methyl-indole **2a**, solvent (200 μL, 0.5 M), and dioxazolone substrate **1a** under air atmosphere. The vial was then sealed. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 25 ^{o}C for 5 h. Water was added and the mixture was extracted with ethyl acetate (EA). The combined organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The resulting residue was dissolved in 0.5 mL of $CDCl_3$ along with 1,1,2,2-tetrachloroethane (16.8 mg, 0.1 mmol, 1.0 equiv., a singlet peak around 5.96 ppm) as internal standard.

4. General procedure for nitrene-triggered carbocationic rearrangement

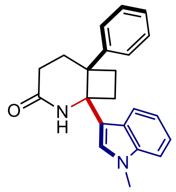


Supplementary Figure 5. General procedure for the reaction of **1** with **2**

Condition [A]: To an oven-dried screwed 4 mL glass vial equipped with a rod-like-shaped Teflon stir bar were added $[Cp^*IrCl_2]_2$ catalyst (2.4 mg, 0.003 mmol, 1.5 mol%), **Nu-H 2** (0.2 mmol), NFTB (400 μL, 0.5 M), and dioxazolone substrate **1** (0.3 mmol, 1.5 equiv.) under air atmosphere. The vial was then sealed. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 25 ^{o}C for 5 h. Water was added and the mixture was extracted with EA. The combined organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (3:1:0 to 2:1:0.2, v/v/v) to give the δ-lactam product.

Condition [B]: To an oven-dried screwed 4 mL glass vial equipped with a rod-like-shaped Teflon stir bar were added $[Cp^*IrCl_2]_2$ catalyst (3.6 mg, 0.0045 mmol, 1.5 mol%), NFTB (400 μL, 0.5 M), and dioxazolone substrate **1** (0.3 mmol, 1.0 equiv.) under air atmosphere. The vial was then sealed. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 25 ^{o}C for 5 h. Water was added and the mixture was extracted with EA. The combined organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (3:1:0 to 2:1:0.2, v/v/v) to give the δ-lactam product.

4.1 Nitrene-triggered carbocationic rearrangement of tetrasubstituted alkenes: the substrate scope of dioxazolones.



3a

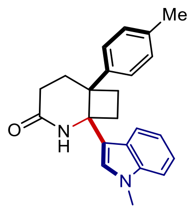
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3a** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (66.1 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.13 (m, 2H), 7.11 – 7.04 (m, 3H), 7.02 – 6.92 (m, 3H), 6.61 (s, 1H), 6.51 (s, 1H), 3.54 (s, 3H), 2.77 – 2.60 (m, 4H), 2.59 – 2.49 (m, 1H), 2.41 – 2.31 (m, 2H), 2.28 – 2.21 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.92, 144.78, 137.16, 127.43, 127.01, 126.45, 126.31, 125.90, 121.63, 120.56, 119.55, 116.21, 109.45, 62.56, 50.38, 35.52, 34.89, 32.71, 28.99, 25.64.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{23}N_2O^+$[M+H]$^+$: 331.1805, found: 331.1812.



3b

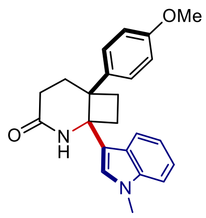
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3b** is a previously unknown compound and was synthesized in 84% yield, dr >20:1 (57.8 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 8.0 Hz, 1H), 7.16 (d, *J* = 4.2 Hz, 2H), 7.11 – 7.07 (m, 1H), 6.95 (d, *J* = 8.2 Hz, 2H), 6.80 (d, *J* = 7.9 Hz, 2H), 6.63 (s, 1H), 6.53 (d, *J* = 7.0 Hz, 1H), 3.54 (s, 3H), 2.75 – 2.62 (m, 3H), 2.62 – 2.49 (m, 2H), 2.40 – 2.35 (m, 1H), 2.34 – 2.29 (m, 1H), 2.25 – 2.17 (m, 1H), 2.13 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.91, 141.81, 137.12, 135.27, 128.15, 127.04, 126.36, 126.28, 121.55, 120.57, 119.51, 116.21, 109.42, 62.48, 49.99, 35.47, 34.99, 32.69, 28.86, 25.71, 20.78.

HRMS (ESI) *m/z* Calcd for $C_{23}H_{25}N_2O^+$[M+H]$^+$: 345.1961, found: 345.1968.



3c

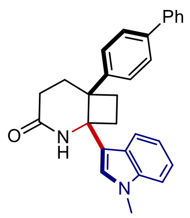
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3c** is a previously unknown compound and was synthesized in 84% yield, dr >20:1 (60.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 8.0 Hz, 1H), 7.15 (s, 2H), 7.09 – 7.05 (m, 1H), 6.97 (d, *J* = 7.9 Hz, 2H), 6.61 (s, 1H), 6.51 (d, *J* = 8.8 Hz, 2H), 6.48 (s, 1H), 3.61 (s, 3H), 3.56 (s, 3H), 2.74 – 2.65 (m, 3H), 2.61 – 2.46 (m, 2H), 2.37 – 2.19 (m, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.90, 157.57, 137.23, 136.85, 127.52, 127.10, 126.34, 121.64, 120.57, 119.57, 116.29, 112.84, 109.48, 62.63, 55.07, 49.80, 35.27, 34.76, 32.75, 29.07, 25.53.

HRMS (ESI) *m/z* Calcd for $C_{23}H_{25}N_2O_2^+$[M+H]$^+$: 361.1911, found: 361.1913.



3d

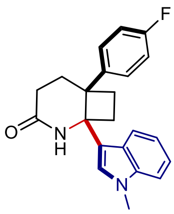
White solid. (R_f = 0.20, DCM/MeOH = 50:1, v/v)

Compound **3d** is a previously unknown compound and was synthesized in 98% yield, dr >20:1 (80.1 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 7.9 Hz, 1H), 7.37 – 7.34 (m, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.15 – 7.05 (m, 5H), 6.64 (s, 1H), 6.52 (s, 1H), 3.49 (s, 3H), 2.76 – 2.59 (m, 4H), 2.56 – 2.49 (m, 1H), 2.40 – 2.31 (m, 2H), 2.28 – 2.21 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.88, 143.93, 140.48, 138.46, 137.18, 128.64, 127.07, 126.89, 126.76, 126.37, 126.05, 121.68, 120.58, 119.65, 116.11, 109.54, 62.66, 50.30, 35.50, 34.81, 32.71, 28.98, 25.65.

HRMS (ESI) *m/z* Calcd for $C_{28}H_{27}N_2O^+$[M+H]$^+$: 407.2118, found: 407.2121.



3e

Yellow solid. (R_f= 0.40, DCM/MeOH = 50:1, v/v)

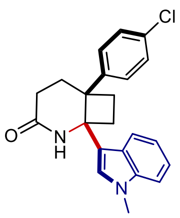
Compound **3e** is a previously unknown compound and was synthesized in 92% yield, dr >20:1 (64.0 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65 (d, *J* = 8.1 Hz, 1H), 7.15 (d, *J* = 3.1 Hz, 2H), 7.08 – 7.04 (m, 1H), 7.01 – 6.98 (m, 2H), 6.67 (s, 1H), 6.61 (dd, *J* = 18.2, 9.6 Hz, 3H), 3.57 (s, 3H), 2.76 – 2.70 (m, 1H), 2.68 – 2.62 (m, 2H), 2.60 – 2.47 (m, 2H), 2.39 – 2.26 (m, 2H), 2.23 – 2.17 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.92, 160.92 (d, *J* = 243.7 Hz), 140.46, 137.23, 127.9 (d, *J* = 7.7 Hz), 127.02, 126.18, 121.73, 120.47, 119.64, 116.01, 114.16 (d, *J* = 20.8 Hz), 109.54, 62.56, 50.02, 35.19, 34.70, 32.76, 29.00, 25.59.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -117.09.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{22}FN_2O^+$[M+H]$^+$: 349.1711, found: 349.1718.



3f

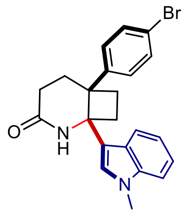
White solid. (R_f= 0.30, DCM/MeOH = 50:1, v/v)

Compound **3f** is a previously unknown compound and was synthesized in 80% yield, dr >20:1 (58.2 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 8.1 Hz, 1H), 7.17 (s, 2H), 7.10 – 7.06 (m, 1H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 8.5 Hz, 2H), 6.68 (s, 1H), 6.48 (s, 1H), 3.60 (s, 3H), 2.75 – 2.63 (m, 3H), 2.60 – 2.48 (m, 2H), 2.39 – 2.27 (m, 2H), 2.21 – 2.15 (m, 1H).

^{13}**C NMR** (101 MHz, Chloroform-*d*) δ 172.83, 143.44, 137.30, 131.67, 127.89, 127.60, 127.02, 126.23, 121.86, 120.50, 119.80, 115.99, 109.67, 62.60, 50.29, 35.42, 34.87, 32.86, 28.99, 25.67.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{22}ClN_2O^+$[M+H]$^+$: 365.1415, found: 365.1414.



3g

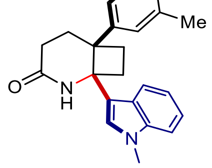
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3g** is a previously unknown compound and was synthesized in 86% yield, dr >20:1 (70.4 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 8.1 Hz, 1H), 7.19 – 7.14 (m, 2H), 7.10 – 7.05 (m, 3H), 6.90 (d, *J* = 8.3 Hz, 2H), 6.69 (s, 1H), 6.53 (s, 1H), 3.59 (s, 3H), 2.74 – 2.69 (m, 1H), 2.64 (t, *J* = 6.6 Hz, 2H), 2.59 – 2.48 (m, 2H), 2.39 – 2.28 (m, 2H), 2.20 – 2.13 (m, 1H).

^{13}**C NMR** (101 MHz, Chloroform-*d*) δ 172.81, 143.98, 137.24, 130.52, 128.24, 127.00, 126.21, 121.81, 120.46, 119.81, 119.78, 115.88, 109.67, 62.49, 50.29, 35.42, 34.84, 32.85, 28.88, 25.67.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{22}BrN_2O^+$[M+H]$^+$: 409.0910, found: 409.0914.



3h

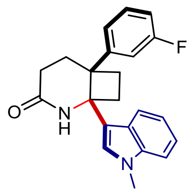
Light Yellow solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3h** is a previously unknown compound and was synthesized in 82% yield, dr >20:1 (56.6 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 8.0 Hz, 1H), 7.17 – 7.08 (m, 3H), 6.91 – 6.85 (m, 3H), 6.75 (d, *J* = 7.0 Hz, 1H), 6.64 (s, 1H), 6.55 (s, 1H), 3.54 (s, 3H), 2.73 – 2.53 (m, 5H), 2.40 – 2.31 (m, 2H), 2.25 – 2.18 (m, 1H), 1.99 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.02, 144.81, 137.10, 136.80, 127.43, 127.27, 126.95, 126.53, 126.49, 123.27, 121.58, 120.71, 119.48, 116.16, 109.39, 62.46, 50.37, 35.68, 35.11, 32.69, 28.92, 25.85, 21.11.

HRMS (ESI) *m/z* Calcd for $C_{23}H_{25}N_2O^+$[M+H]$^+$: 345.1961, found: 345.1968.



3i

White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

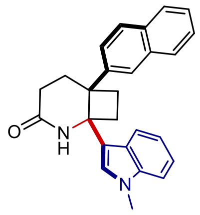
Compound **3i** is a previously unknown compound and was synthesized in 92% yield, dr >20:1 (64.1 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.66 (d, *J* = 8.1 Hz, 1H), 7.14 (d, *J* = 4.0 Hz, 2H), 7.09 – 7.03 (m, 1H), 6.90 – 6.74 (m, 3H), 6.69 (s, 1H), 6.60 (t, *J* = 8.0 Hz, 1H), 6.52 (s, 1H), 3.56 (s, 3H), 2.79 – 2.72 (m, 1H), 2.69 – 2.65 (m, 2H), 2.62 – 2.48 (m, 2H), 2.38 – 2.27 (m, 2H), 2.26 – 2.19 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.78, 162.18 (d, *J* = 243.8 Hz), 147.49 (d, *J* = 6.6 Hz), 137.26, 128.70 (d, *J* = 8.4 Hz), 126.97, 126.16, 122.23 (d, *J* = 2.5 Hz), 121.76, 120.47, 119.64, 115.89, 113.53(d, *J* = 22.0 Hz), 112.71 (d, *J* = 20.9 Hz), 109.50, 62.56, 50.44, 35.25, 34.56, 32.72, 29.02, 25.63.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.80.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{22}FN_2O^+$[M+H]$^+$: 349.1711, found: 349.1705.



3j

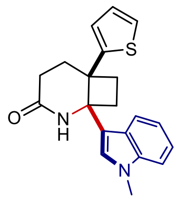
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3j** is a previously unknown compound and was synthesized in 89% yield, dr >20:1 (67.6 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 7.3 Hz, 1H), 7.59 (s, 2H), 7.51 (d, *J* = 7.4 Hz, 1H), 7.42 (d, *J* = 8.6 Hz, 1H), 7.35 – 7.31 (m, 2H), 7.13 (dd, *J* = 20.3, 7.3 Hz, 4H), 6.63 (d, *J* = 22.5 Hz, 2H), 3.43 (s, 3H), 2.81 – 2.58 (m, 5H), 2.49 – 2.39 (m, 2H), 2.34 – 2.27 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.96, 142.56, 137.13, 132.72, 131.70, 127.67, 127.23, 126.99, 126.95, 126.52, 125.71, 125.36, 125.31, 124.75, 121.69, 120.68, 119.68, 116.11, 109.55, 62.54, 50.81, 35.82, 35.26, 32.63, 29.00, 25.92.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}N_2O^+$[M+H]$^+$: 381.1961, found: 381.1969.



3k

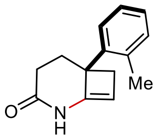
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3k** is a previously unknown compound and was synthesized in 50% yield, dr >20:1 (33.6 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.12 (m, 2H), 6.98 (t, *J* = 6.7 Hz, 1H), 6.84 (dd, *J* = 4.8, 1.4 Hz, 1H), 6.76 (s, 1H), 6.64 – 6.53 (m, 2H), 6.24 (s, 1H), 3.67 (s, 3H), 2.97 – 2.88 (m, 1H), 2.85 – 2.76 (m, 2H), 2.59 – 2.43 (m, 3H), 2.41 – 2.36 (m, 1H), 2.32 – 2.26 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.48, 148.85, 137.63, 127.74, 126.27, 125.96, 124.30, 123.66, 121.82, 120.88, 119.40, 115.38, 109.31, 63.77, 48.33, 33.56, 32.89, 32.70, 29.71, 27.60.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{21}N_2OS^+[M+H]^+$: 337.1369, found: 337.1370.



3l

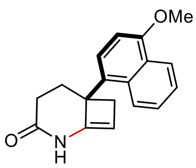
White solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3l** is a previously unknown compound and was synthesized in 98% yield (41.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.10 (s, 1H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.17 (d, *J* = 3.7 Hz, 2H), 7.13 – 7.10 (m, 1H), 4.99 (s, 1H), 2.75 (dd, *J* = 11.5, 1.4 Hz, 1H), 2.47 (d, *J* = 11.5 Hz, 1H), 2.42 (d, *J* = 13.7 Hz, 1H), 2.32 (s, 3H), 2.30 (d, *J* = 3.6 Hz, 1H), 2.18 (dd, *J* = 13.2, 4.5 Hz, 1H), 2.12 (d, *J* = 8.1 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.13, 141.16, 140.84, 135.51, 131.26, 126.94, 126.85, 125.64, 101.77, 48.79, 40.62, 30.61, 30.29, 19.41.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{16}NO^+[M+H]^+$: 214.1226, found: 214.1230.



3m

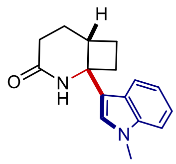
Gray solid. (R_f = 0.20, DCM/MeOH = 50:1, v/v)

Compound **3m** is a previously unknown compound and was synthesized in 97% yield (54.1 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.56 (s, 1H), 8.34 (d, *J* = 7.3 Hz, 1H), 7.85 (d, *J* = 8.5 Hz, 1H), 7.55 – 7.47 (m, 2H), 7.38 (d, *J* = 7.9 Hz, 1H), 6.70 (d, *J* = 7.9 Hz, 1H), 5.05 (s, 1H), 3.98 (s, 3H), 2.94 (d, *J* = 11.5 Hz, 1H), 2.64 – 2.59 (m, 2H), 2.45 – 2.39 (m, 1H), 2.32 – 2.12 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.75, 154.91, 141.45, 131.49, 130.57, 126.54, 126.31, 124.99, 124.34, 124.18, 123.18, 102.82, 102.33, 55.63, 48.32, 41.30, 31.52, 30.71.

HRMS (ESI) *m/z* Calcd for $C_{18}H_{18}NO_2^+$[M+H]$^+$: 280.1332, found: 280.1338.



3n

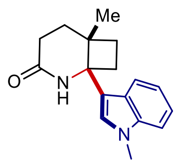
Light yellow oil. (R_f= 0.40, DCM/MeOH = 50:1, v/v)

Compound **3n** is a previously unknown compound and was synthesized in 42% yield, dr >20:1 (21.4 mg, 0.2 mmol scale) following the condition. *Note*: *the conditions are the same as* ***Condition [A]****, except that the reaction time is extended to 17 hours.*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 1H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.12 (t, *J* = 6.9 Hz, 1H), 7.01 (s, 1H), 6.12 (s, 1H), 3.77 (s, 3H), 3.05 – 2.98 (m, 1H), 2.89 – 2.82 (m, 1H), 2.65 – 2.57 (m, 1H), 2.52 (dt, *J* = 11.3, 5.2 Hz, 1H), 2.40 – 2.33 (m, 1H), 2.18 – 2.06 (m, 2H), 1.95 – 1.83 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.89, 138.02, 125.98, 125.31, 122.22, 119.91, 119.50, 118.84, 109.79, 57.33, 38.88, 34.87, 32.91, 28.82, 24.14, 19.31.

HRMS (ESI) *m/z* Calcd for $C_{16}H_{19}N_2O^+$[M+H]$^+$: 255.1492, found: 255.1498.



3o

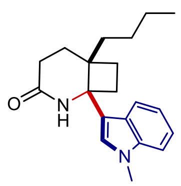
White solid. (R_f= 0.30, DCM/MeOH = 50:1, v/v)

Compound **3o** is a previously unknown compound and was synthesized in 83% yield, dr >20:1 (44.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 8.2 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 7.5 Hz, 1H), 6.99 (s, 1H), 6.01 (s, 1H), 3.77 (s, 3H), 2.81 – 2.74 (m, 1H), 2.67 – 2.60 (m, 2H), 2.24 – 2.17 (m, 1H), 2.09 – 2.02 (m, 1H), 1.92 (dd, *J* = 7.2, 5.0 Hz, 2H), 1.69 – 1.62 (m, 1H), 0.84 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.68, 137.90, 127.31, 126.47, 122.02, 120.83, 119.55, 115.97, 109.51, 61.15, 41.19, 32.93, 31.89, 31.75, 29.24, 25.57, 25.02.

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



3p

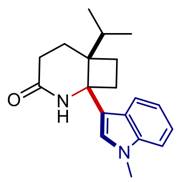
Light yellow oil. (R_f = 0.40, DCM/MeOH = 50:1, v/v)

Compound **3p** is a previously unknown compound and was synthesized in 96% yield, dr >20:1 (59.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 8.2 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.97 (s, 1H), 6.03 (s, 1H), 3.75 (s, 3H), 2.78 – 2.71 (m, 1H), 2.62 – 2.58 (m, 2H), 2.23 – 2.16 (m, 1H), 2.01 – 1.81 (m, 2H), 1.89 – 1.81 (m, 1H), 1.75 – 1.68 (m, 1H), 1.14 – 0.99 (m, 6H), 0.70 (t, *J* = 8.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.67, 137.83, 127.36, 126.69, 121.85, 120.88, 119.43, 115.52, 109.38, 61.47, 44.40, 36.44, 32.83, 32.28, 29.00, 28.36, 25.96, 23.45, 23.16, 14.11.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{27}N_2O^+$[M+H]$^+$: 311.2118, found: 311.2119.



3q

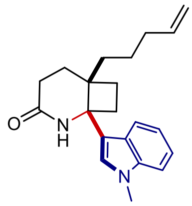
White solid. (R_f = 0.40, DCM/MeOH = 50:1, v/v)

Compound **3q** is a previously unknown compound and was synthesized in 89% yield, dr >20:1 (52.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 8.1 Hz, 1H), 7.29 (d, *J* = 8.2 Hz, 1H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.08 (t, *J* = 8.0 Hz, 1H), 6.99 (s, 1H), 6.27 (s, 1H), 3.77 (s, 3H), 2.79 – 2.72 (m, 1H), 2.67 – 2.60 (m, 2H), 2.22 – 2.16 (m, 1H), 2.00 – 1.91 (m, 2H), 1.89 – 1.85 (m, 1H), 1.78 – 1.65 (m, 2H), 0.71 (d, *J* = 6.9 Hz, 3H), 0.38 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.63, 137.58, 127.65, 126.57, 121.86, 121.16, 119.62, 115.94, 109.60, 61.78, 48.84, 34.48, 33.25, 32.95, 29.75, 25.19, 23.19, 17.82, 17.28.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{25}N_2O^+$[M+H]$^+$: 297.1961, found: 297.1970.



3r

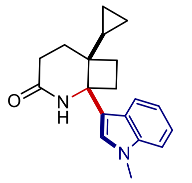
Light yellow oil. (R_f= 0.40, DCM/MeOH = 50:1, v/v)

Compound **3r** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (63.8 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.97 (s, 1H), 6.04 (s, 1H), 5.65 – 5.55 (m, 1H), 4.80 (d, *J* = 12.0 Hz, 2H), 3.75 (s, 3H), 2.78 – 2.71 (m, 1H), 2.61 (t, *J* = 8.0 Hz, 2H), 2.24 – 2.16 (m, 1H), 2.02 – 1.82 (m, 3H), 1.77 – 1.68 (m, 3H), 1.19 – 1.07 (m, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.60, 138.58, 137.83, 127.37, 126.66, 121.90, 120.86, 119.47, 115.42, 114.22, 109.40, 61.43, 44.38, 36.29, 34.06, 32.84, 32.30, 28.99, 28.37, 23.46, 23.19.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{27}N_2O^+$[M+H]$^+$: 323.2118, found: 323.2120.



3s

Light yellow solid. (R_f= 0.40, DCM/MeOH = 50:1, v/v)

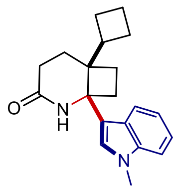
Compound **3s** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (57.6 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56 (d, *J* = 8.0 Hz, 1H), 7.24 (s, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.94 (s, 1H), 6.06 (s, 1H), 3.74 (s, 3H), 2.67 – 2.60 (m, 3H), 2.16 – 2.09 (m, 1H), 2.00 – 1.93 (m, 1H), 1.89 – 1.82 (m, 1H), 1.72 – 1.65 (m, 1H), 1.27 – 1.21 (m, 1H), 0.47 – 0.40 (m, 1H), 0.10 – 0.05 (m, 2H), -0.11 – -

0.16 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.46, 137.83, 127.38, 126.83, 121.76, 120.81, 119.30, 115.76, 109.32, 61.46, 44.58, 32.82, 32.07, 30.54, 29.01, 18.28, 16.20, 2.19, -0.36.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{23}N_2O^+[M+H]^+$: 295.1805, found: 295.1809.



3t

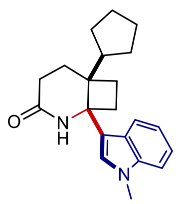
Light yellow solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **3t** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (59.8 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 4.0 Hz, 1H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 7.5 Hz, 1H), 6.89 (s, 1H), 5.97 (s, 1H), 3.74 (s, 3H), 2.73 – 2.67 (m, 1H), 2.63 (t, *J* = 6.7 Hz, 2H), 2.28 – 2.22 (m, 1H), 2.20 – 2.13 (m, 1H), 2.04 – 1.97 (m, 1H), 1.92 – 1.85 (m, 1H), 1.81 (t, *J* = 6.7 Hz, 2H), 1.72 – 1.63 (m, 1H), 1.47 – 1.40 (m, 4H), 0.90 – 0.82 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.61, 137.80, 127.11, 126.68, 121.83, 120.70, 119.32, 115.55, 109.29, 60.21, 46.24, 41.34, 32.77, 32.04, 28.78, 27.27, 24.43, 23.40, 19.16, 17.76.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{25}N_2O^+[M+H]^+$: 309.1961, found: 309.1967.



3u

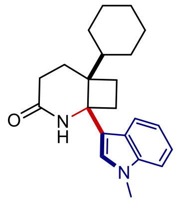
Light yellow oil. (R_f = 0.40, DCM/MeOH = 50:1, v/v)

Compound **3u** is a previously unknown compound and was synthesized in 98% yield, dr >20:1 (63.2 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.1 Hz, 1H), 7.24 (d, *J* = 1.5 Hz, 1H), 7.20 – 7.16 (m, 1H), 7.05 – 7.01 (m, 1H), 6.93 (s, 1H), 6.01 (s, 1H), 3.74 (s, 3H), 2.83 – 2.75 (m, 1H), 2.67 – 2.63 (m, 2H), 2.14 – 2.07 (m, 1H), 2.02 – 1.88 (m, 3H), 1.81 – 1.71 (m, 2H), 1.34 – 1.23 (m, 2H), 1.21 – 1.06 (m, 4H), 0.88 – 0.77 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.82, 137.76, 127.53, 126.64, 121.85, 121.03, 119.41, 116.50, 109.36, 61.67, 48.27, 45.32, 32.91, 32.87, 29.18, 27.82, 27.63, 26.08, 25.81, 21.93.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{27}N_2O^+[M+H]^+$: 323.2118, found: 323.2115.



3v

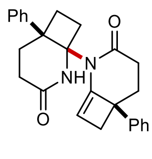
Light yellow oil. (R_f= 0.30, DCM/MeOH = 50:1, v/v)

Compound **3v** is a previously unknown compound and was synthesized in 87% yield, dr >20:1 (58.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 8.1 Hz, 1H), 7.23 (d, *J* = 4.4 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.04 (t, *J* = 7.6 Hz, 1H), 6.96 (s, 1H), 6.20 (s, 1H), 3.75 (s, 3H), 2.74 – 2.67 (m, 1H), 2.61 – 2.52 (m, 2H), 2.16 (dt, *J* = 12.5, 9.0 Hz, 1H), 2.02 – 1.94 (m, 1H), 1.84 (t, *J* = 8.4 Hz, 2H), 1.78 – 1.72 (m, 1H), 1.62 (d, *J* = 11.9 Hz, 1H), 1.49 (d, *J* = 12.5 Hz, 1H), 1.38 (d, *J* = 12.1 Hz, 1H), 1.17 (d, *J* = 12.5 Hz, 2H), 1.08 (d, *J* = 12.4 Hz, 1H), 0.97 – 0.90 (m, 1H), 0.88 – 0.77 (m, 2H), 0.73 – 0.63 (m, 1H), 0.33 – 0.22 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.45, 137.60, 127.56, 126.58, 121.83, 121.16, 119.61, 115.90, 109.52, 62.11, 48.42, 44.81, 34.70, 32.98, 29.90, 28.71, 27.17, 27.05, 26.64, 26.39, 25.11, 24.79.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{29}N_2O^+[M+H]^+$: 337.2274, found: 337.2274.



4a

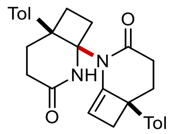
White solid. (R_f= 0.30, DCM/MeOH = 50:1, v/v)

Compound **4a** is a previously unknown compound and was synthesized in 83% yield, dr >20:1 (49.6 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65 (dd, *J* = 6.9, 2.9 Hz, 2H), 7.40 – 7.32 (m, 3H), 7.15 – 7.02 (m, 3H), 7.00 – 6.89 (m, 1H), 6.55 – 6.38 (m, 2H), 4.89 (s, 1H), 3.20 – 3.12 (m, 1H), 3.04 (q, *J* = 10.5 Hz, 1H), 2.74 – 2.70 (m, 2H), 2.56 (dd, *J* = 11.6, 1.3 Hz, 1H), 2.51 – 2.41 (m, 2H), 2.33 – 2.25 (m, 2H), 2.22 (d, *J* = 11.6 Hz, 1H), 2.15 – 2.01 (m, 2H), 1.87 – 1.78 (m, 1H), 1.75 – 1.69 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.76, 169.46, 142.86, 141.19, 140.60, 128.42, 127.58, 127.56, 126.20, 125.72, 103.96, 75.52, 50.25, 49.93, 40.95, 33.60, 32.35, 32.17, 28.38, 27.62, 22.43.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{27}N_2O_2^+$[M+H]$^+$: 399.2067, found: 399.2068.



4b

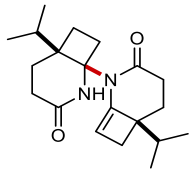
White solid. (R_f = 0.20, DCM/MeOH = 50:1, v/v)

Compound **4b** is a previously unknown compound and was synthesized in 76% yield, dr >20:1 (48.6 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.90 (s, 1H), 6.84 (d, *J* = 7.7 Hz, 2H), 6.39 (d, *J* = 7.8 Hz, 2H), 4.86 (s, 1H), 3.18 – 3.09 (m, 1H), 3.01 (q, *J* = 10.5 Hz, 1H), 2.73 – 2.69 (m, 2H), 2.53 (dd, *J* = 11.6, 1.3 Hz, 1H), 2.48 – 2.39 (m, 5H), 2.29 – 2.24 (m, 5H), 2.18 (d, *J* = 11.5 Hz, 1H), 2.13 – 2.09 (m, 1H), 2.07 – 2.00 (m, 1H), 1.89 – 1.80 (m, 1H), 1.71 – 1.65 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.72, 169.41, 141.26, 139.81, 137.50, 136.81, 135.53, 129.14, 128.84, 127.40, 125.62, 103.70, 75.35, 49.84, 49.59, 40.97, 33.43, 32.18, 28.33, 27.72, 22.23, 21.07.

HRMS (ESI) *m/z* Calcd for $C_{28}H_{31}N_2O_2^+$[M+H]$^+$: 427.2380, found: 427.2390.



4c

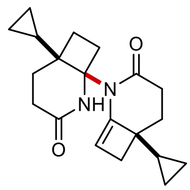
White solid. (R_f = 0.20, DCM/MeOH = 50:1, v/v)

Compound **4c** is a previously unknown compound and was synthesized in 98% yield, dr >20:1 (48.5 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.48 (t, *J* = 13.3 Hz, 1H), 4.86 (s, 1H), 2.57 (t, *J* = 12.0 Hz, 1H), 2.46 – 2.40 (m, 4H), 2.22 – 2.17 (m, 2H), 2.06 – 1.98 (m, 2H), 1.97 – 1.93 (m, 1H), 1.89 – 1.81 (m, 2H), 1.74 (dd, *J* = 12.3, 5.3 Hz, 2H), 1.68 – 1.63 (m, 1H), 1.55 – 1.50 (m, 1H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.91 (d, *J* = 6.7 Hz, 3H), 0.87 (d, *J* = 6.7 Hz, 3H), 0.75 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.75, 169.99, 142.40, 105.02, 75.27, 49.64, 48.31, 33.40, 31.78, 31.73, 31.17, 28.80, 27.71, 26.57, 23.86, 19.88, 18.25, 17.88, 17.55, 17.50.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{30}N_2NaO_2^+$[M+Na]$^+$:353.2199, found: 353.2197.



4d

Light yellow solid. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

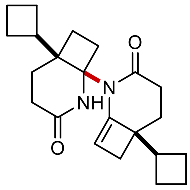
Compound **4d** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (47.9 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.31 (s, 1H), 4.75 (s, 1H), 2.85 – 2.75 (m, 1H), 2.51 – 2.38 (m, 5H), 2.10 – 2.04 (m, 2H), 1.99 – 1.90 (m, 3H), 1.77 – 1.69 (m, 1H), 1.46 – 1.41 (m, 1H), 0.96 (q, *J* = 8.1, 5.1 Hz, 2H), 0.86 (q, *J* = 7.2 Hz, 1H), 0.54 – 0.27 (m, 8H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.85, 170.01, 140.81, 104.10, 75.12, 45.94, 44.83, 36.82, 32.83, 32.44, 31.82, 27.88, 27.45, 16.38, 15.42, 14.01, 3.87, 2.52, -0.04,

-0.44.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{27}N_2O_2^+$[M+H]$^+$:327.2067, found: 327.2061.



4e

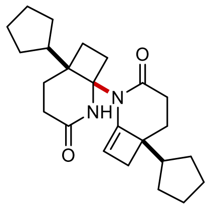
Light yellow solid. (R_f= 0.30, DCM/MeOH = 50:1, v/v)

Compound **4e** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (52.3 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.49 (s, 1H), 4.80 (s, 1H), 2.61 (s, 1H), 2.50 – 2.34 (m, 7H), 2.14 – 1.95 (m, 4H), 1.82 (s, 5H), 1.74 – 1.62 (m, 10H), 1.32 (d, *J* = 13.4 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.87, 169.62, 140.70, 103.81, 74.36, 47.74, 45.67, 39.79, 35.73, 32.94, 32.66, 31.73, 28.04, 27.57, 24.69, 23.95, 23.88, 23.71, 22.90, 18.16, 17.66, 17.24.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{30}N_2NaO_2^+$[M+Na]$^+$:377.2199, found: 377.2202.



4f

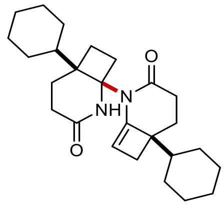
Light yellow solid. (R_f= 0.30, DCM/MeOH = 50:1, v/v)

Compound **4f** is a previously unknown compound and was synthesized in 98% yield, dr >20:1 (56.2 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.18 (s, 1H), 4.83 (s, 1H), 2.61 – 2.52 (m, 2H), 2.47 (t, *J* = 12.0 Hz, 3H), 2.33 – 2.09 (m, 4H), 2.03 – 1.93 (m, 3H), 1.90 – 1.78 (m, 4H), 1.63 – 1.48 (m, 12H), 1.40 – 1.29 (m, 2H), 1.12 (q, *J* = 9.6, 8.8 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.74, 169.84, 143.18, 104.17, 75.16, 48.52, 47.49, 44.10, 38.36, 33.40, 33.04, 31.67, 30.03, 29.33, 28.71, 27.87, 27.53, 27.11, 26.97, 26.59, 25.85, 25.63, 24.63, 18.37.

HRMS (ESI) *m/z* Calcd for $C_{24}H_{35}N_2O_2^+$[M+H]$^+$:383.2693, found: 383.2698.



4g

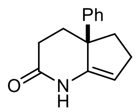
Light yellow oil. (R_f= 0.40, DCM/MeOH = 50:1, v/v)

Compound **4g** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (60.2 mg, 0.3 mmol scale) following the **Condition [B].**

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.35 (s, 1H), 4.83 (s, 1H), 2.55 (d, *J* = 10.8 Hz, 1H), 2.49 – 2.37 (m, 4H), 2.27 (d, *J* = 11.9 Hz, 1H), 2.19 (d, *J* = 8.7 Hz, 1H), 2.08 (d, *J* = 11.6 Hz, 1H), 1.98 (t, *J* = 12.0 Hz, 1H), 1.88 (d, *J* = 12.0 Hz, 1H), 1.77 – 1.68 (m, 10H), 1.62 – 1.53 (m, 5H), 1.43 (t, *J* = 12.0 Hz, 1H), 1.18 – 1.07 (m, 8H), 0.95 – 0.83 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.58, 170.02, 142.00, 104.71, 75.54, 49.42, 48.11, 41.82, 37.27, 33.58, 33.40, 31.76, 29.78, 28.42, 27.88, 27.72, 27.50, 27.10, 26.76, 26.72, 26.54, 26.46, 24.44, 18.64.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{38}N_2NaO_2^+$[M+Na]$^+$:433.2825, found: 433.2833.



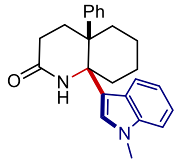
4h'

Yellow solid. (R_f = 0. 40, DCM/MeOH = 50:1, v/v)

Compound **4h'** is a previously known compound and was synthesized in 97% yield (41.3 mg, 0.2 mmol scale) following the **Condition [A]**.[3]

^{1}H NMR (400 MHz, Chloroform-*d*) 9.58 (s, 1H), 7.31 (s, 4H), 7.23 (s, 1H), 5.18 (s, 1H), 2.47 (d, *J* = 10.5 Hz, 1H), 2.35 (d, *J* = 15.6 Hz, 1H), 2.20 – 2.10 (m, 3H), 2.03 – 1.93 (m, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.90, 142.75, 141.58, 128.64, 126.62, 126.42, 105.29, 50.76, 41.33, 32.90, 29.49, 27.94.



4i

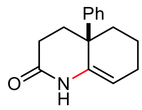
Yellow solid. (R_f = 0. 40, DCM/MeOH = 50:1, v/v)

Compound **4i** is a previously unknown compound and was synthesized in 47% yield, dr >20:1 (33.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) 7.36 – 7.30 (m, 2H), 7.26 (s, 1H), 7.25 – 7.18 (m, 4H), 7.13 – 7.09 (m, 1H), 6.83 – 6.76 (m, 3H), 3.80 (s, 3H), 2.53 – 2.41 (m, 2H), 2.39 – 2.31 (m, 1H), 2.26 – 2.09 (m, 2H), 1.85 – 1.78 (m, 1H), 1.74 – 1.68 (m, 2H), 1.57 – 1.47 (m, 2H), 1.34 – 1.28 (m, 1H), 1.15 (s, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.01, 144.26, 137.14, 128.05, 127.78, 127.19, 127.11, 126.39, 121.94, 120.88, 119.55, 117.02, 109.48, 68.53, 49.54, 35.37, 33.56, 33.09, 30.80, 28.42, 25.51, 24.99.

HRMS (ESI) *m/z* Calcd for $C_{24}H_{27}N_2O^+$[M+H]$^+$: 359.2118, found: 359.2126.



4i'

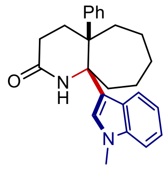
Yellow solid. (R_f = 0. 40, DCM/MeOH = 50:1, v/v)

Compound **4i'** is a previously unknown compound and was synthesized in 45% yield (20.4 mg, 0.2 mmol scale) following the condition. *Note: the conditions are the same as* ***Condition [B]****, except that HFIP (0.5 M) is used as the solvent.*

^{1}H NMR (400 MHz, Chloroform-*d*) 7.70 (s, 1H), 7.33 – 7.26 (m, 5H), 7.21 (d, *J* = 7.0 Hz, 1H), 5.04 (s, 1H), 3.02 (s, 2H), 2.24 – 2.18 (m, 2H), 2.04 – 1.97 (m, 2H), 1.75 (s, 4H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 180.40, 148.57, 144.44, 128.57, 126.78, 125.33, 98.34, 51.82, 37.79, 36.25, 34.40, 23.36.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{18}NO^+$[M+H]$^+$: 228.1383, found: 228.1387.



4j

Yellow liquid. (R_f = 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

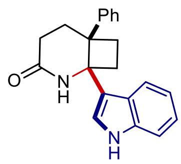
Compound **4j** is a previously unknown compound and was synthesized in 37% yield, dr >20:1 (28.9 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (s, 1H), 7.33 – 7.24 (m, 3H), 7.21 (d, J = 6.6 Hz, 3H), 7.09 (t, J = 7.6 Hz, 1H), 6.79 (t, J = 7.6 Hz, 1H), 6.71 (d, J = 8.2 Hz, 1H), 6.54 (s, 1H), 3.81 (s, 3H), 2.59 – 2.36 (m, 2H), 2.23 – 2.16 (m, 1H), 2.12 – 1.96 (m, 2H), 1.77 – 1.64 (m, 4H), 1.62 – 1.58 (m, 1H), 1.54 – 1.47 (m, 2H), 1.42 – 1.31 (m, 1H), 1.21 – 1.10 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.60, 143.74, 136.86, 128.94, 127.60, 127.33, 126.99, 126.64, 122.02, 121.15, 119.66, 116.41, 109.43, 70.00, 39.90, 33.08, 31.17, 29.06, 27.73, 25.97, 22.96, 22.06, 21.65.

HRMS (ESI) *m/z* Calcd for $C_{25}H_{29}N_2O^+$[M+H]$^+$: 373.2274, found: 373.2284.

4.2 Nitrene-triggered carbocationic rearrangement of tetrasubstituted alkenes: the substrate scope of nucleophiles.



5a

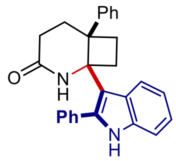
Gray solid. (R_f = 0.35, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5a** is a previously unknown compound and was synthesized in 78% yield, dr >20:1 (49.8 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-*d_6*) δ 10.76 (s, 1H), 7.97 (s, 1H), 7.54 (d, J = 7.9 Hz, 1H), 7.16 (d, J = 7.9 Hz, 1H), 7.04 (d, J = 2.6 Hz, 1H), 6.95 – 6.91(m, 3H), 6.90 – 6.86 (m, 1H), 6.84 – 6.76 (m, 3H), 2.67 – 2.61 (m, 1H), 2.56 – 2.46 (m, 1H), 2.41 – 2.32 (m, 3H), 2.30 – 2.16 (m, 2H), 2.05 – 1.94 (m, 1H).

^{13}C NMR (101 MHz, DMSO-*d_6*) δ 171.14, 145.56, 136.44, 127.10, 126.31, 126.29, 125.36, 123.39, 120.58, 120.39, 118.50, 117.22, 111.50, 61.85, 49.73, 34.85, 34.55, 28.56, 25.75.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{21}N_2O^+$[M+H]$^+$: 317.1648, found: 317.1652.



5b

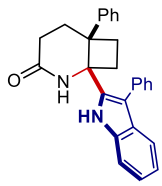
Light yellow solid. (R_f= 0.40, DCM/MeOH = 50:1, v/v)

Compound **5b** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (78.6 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.70 (s, 1H), 8.07 (s, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.27 – 7.21 (m, 4H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.93 (t, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.5 Hz, 2H), 6.85 – 6.72 (m, 5H), 2.68 – 2.58 (m, 1H), 2.52 – 2.38 (m, 2H), 2.30 – 2.20 (m, 1H), 2.14 – 2.07 (m, 1H), 1.89 – 1.84 (m, 1H), 1.73 – 1.51 (m, 2H).

^{13}C NMR (101 MHz, DMSO-*d*$_6$) δ 169.82, 143.42, 136.68, 135.47, 134.30, 128.12, 127.74, 127.40, 127.30, 126.98, 126.57, 125.69, 120.98, 120.51, 118.73, 113.23, 110.89, 63.76, 49.90, 36.12, 33.34, 29.99, 28.75.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{25}N_2O^+$[M+H]$^+$: 393.1961, found: 393.1957.



5c

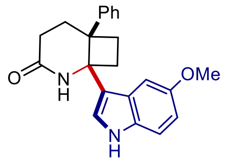
White solid. (R_f= 0.40, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5c** is a previously unknown compound and was synthesized in 54% yield, dr >20:1 (42.3 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Methanol-*d*$_4$) δ 7.34 (d, *J* = 8.1 Hz, 1H), 7.31 – 7.26 (m, 3H), 7.09 – 7.06 (m, 6H), 6.98 – 6.80 (m, 4H), 2.74 – 2.60 (m, 2H), 2.54 (dt, *J* = 17.9, 5.0 Hz, 1H), 2.43 – 2.26 (m, 3H), 2.07 – 2.00 (m, 1H), 1.93 – 1.87 (m, 1H).

^{13}C NMR (101 MHz, Methanol-*d*$_4$) δ 174.57, 144.47, 136.89, 136.33, 134.42, 132.38, 130.87, 129.86, 129.36, 128.73, 128.57, 127.69, 127.63, 127.30, 122.91, 120.05, 119.73, 117.83, 111.65, 65.45, 51.76, 33.49, 32.42, 29.46, 24.72.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{25}N_2O^+[M+H]^+$: 393.1961, found: 393.1964.



5d

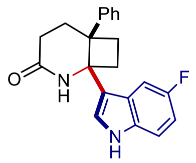
Brown green solid. (R_f = 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5d** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (68.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.25 (s, 1H), 7.10 (d, *J* = 8.8 Hz, 1H), 7.07 – 7.04 (m, 3H), 7.00 – 6.93 (m, 3H), 6.77 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.69 (d, *J* = 2.7 Hz, 1H), 6.46 (s, 1H), 3.80 (s, 3H), 2.76 – 2.64 (m, 3H), 2.63 – 2.56 (m, 1H), 2.53 – 2.46 (m, 1H), 2.39 – 2.28 (m, 2H), 2.28 – 2.17 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.24, 154.00, 144.70, 131.73, 127.56, 126.57, 126.20, 126.08, 123.08, 117.43, 112.30, 112.23, 102.36, 62.67, 56.05, 50.41, 35.17, 34.69, 29.12, 25.73.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{23}N_2O_2^+[M+H]^+$: 347.1754, found: 347.1755.



5e

White solid. (R_f = 0.25, PE/EA/MeOH = 2:1:0.2, v/v/v)

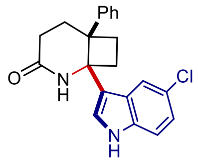
Compound **5e** is a previously unknown compound and was synthesized in 77% yield, dr >20:1 (51.4 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.86 (s, 1H), 8.05 (s, 1H), 7.22 – 7.07 (m, 3H), 6.91 – 6.89 (m, 2H), 6.87 – 6.72 (m, 4H), 2.63 – 2.57 (m, 1H), 2.51 – 2.44 (m, 1H), 2.40 – 2.29 (m, 3H), 2.26 – 2.18 (m, 2H), 2.02 – 1.95 (m, 1H).

^{13}C NMR (101 MHz, DMSO-*d*$_6$) δ 171.32, 156.38 (d, *J* = 230.7 Hz), 145.44, 133.04, 127.12, 126.42 (d, *J* = 9.9 Hz), 126.27, 125.39, 117.52 (d, *J* = 4.7 Hz), 112.30 (d, *J* = 11.0 Hz), 108.71 (d, *J* = 25.9 Hz), 105.00 (d, *J* = 23.4 Hz), 61.62, 49.72, 34.90, 34.42, 28.51, 25.83.

^{19}F NMR (376 MHz, DMSO-d_6) δ -124.88.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{20}FN_2O^+[M+H]^+$: 335.1554, found: 335.1562.



5f

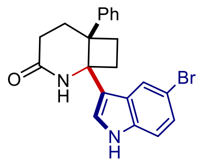
Grass green solid. (R_f= 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5f** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (69.3 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.06 (s, 1H), 8.20 (s, 1H), 7.57 (d, *J* = 2.1 Hz, 1H), 7.30 – 7.18 (m, 2H), 7.07 – 6.96 (m, 3H), 6.96 – 6.87 (m, 3H), 2.73 – 2.67 (m, 1H), 2.60 – 2.53 (m, 1H), 2.49 – 2.39 (m, 3H), 2.36 – 2.26 (m, 2H), 2.16 – 2.03 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.40, 145.32, 134.87, 127.37, 127.13, 126.27, 125.46, 125.20, 123.06, 120.53, 119.53, 117.21, 112.95, 61.64, 49.74, 34.99, 34.32, 28.59, 25.72.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{20}ClN_2O^+[M+H]^+$: 351.1259, found: 351.1262.



5g

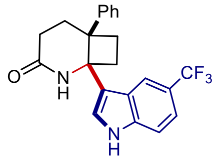
White solid. (R_f= 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5g** is a previously unknown compound and was synthesized in 73% yield, dr >20:1 (57.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.98 (s, 1H), 8.11 (s, 1H), 7.64 (s, 1H), 7.21 – 7.09 (m, 2H), 7.05 (d, *J* = 8.7 Hz, 1H), 6.92 (d, *J* = 7.3 Hz, 2H), 6.88 – 6.82 (m, 3H), 2.66 – 2.60 (m, 1H), 2.53 – 2.48 (m, 1H), 2.41 – 2.33 (m, 3H), 2.28 – 2.18 (m, 2H), 2.06 – 1.99 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.30, 145.25, 135.04, 128.04, 127.08, 126.23, 125.42, 125.00, 123.01, 122.51, 117.11, 113.36, 111.12, 61.62, 49.70, 34.93, 34.26, 28.60, 25.64.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{20}BrN_2O^+[M+H]^+$: 395.0754, found: 395.0762.



5h

White solid. (R_f= 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

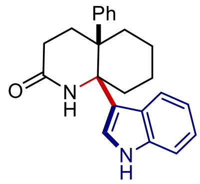
Compound **5h** is a previously unknown compound and was synthesized in 80% yield, dr >20:1 (61.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.23 (s, 1H), 8.17 (s, 1H), 7.81 (s, 1H), 7.34 (d, J = 8.5 Hz, 1H), 7.29 (d, J = 2.4 Hz, 1H), 7.21 (d, J = 8.7 Hz, 1H), 6.98 – 6.88 (m, 2H), 6.84 – 6.79 (m, 3H), 2.73 – 2.67 (m, 1H), 2.61 – 2.50 (m, 1H), 2.47 – 2.36 (m, 3H), 2.32 – 2.19 (m, 2H), 2.11 – 2.05 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.45, 145.03, 137.93, 126.99, 126.21, 125.69, 125.64 (q, J = 270.6 Hz), 125.42, 119.08 (q, J = 30.2 Hz), 118.36, 117.87 (q, J = 5.0 Hz), 116.89 (q, J = 3.6 Hz), 112.09, 61.65, 49.64, 34.81, 34.08, 28.75, 25.59.

^{19}F NMR (376 MHz, DMSO-d_6) δ -58.38.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{20}F_3N_2O^+[M+H]^+$: 385.1522, found: 385.1530.



5i

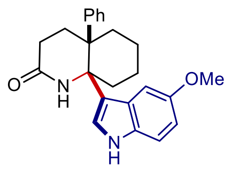
White solid. (R_f= 0.40, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5i** is a previously unknown compound and was synthesized in 41% yield, dr >20:1 (28.2 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.07 (s, 1H), 7.83 (s, 1H), 7.51 (d, J = 2.7 Hz, 1H), 7.38 – 7.28 (m, 3H), 7.28 – 7.17 (m, 3H), 7.03 – 6.92 (m, 2H), 6.71 (t, J = 7.7 Hz, 1H), 2.46 – 2.29 (m, 2H), 2.24 – 2.13 (m, 2H), 2.06 – 1.99 (m, 1H), 1.85 – 1.79 (m, 1H), 1.75 – 1.69 (m, 1H), 1.52 – 1.45 (m, 1H), 1.43 – 1.30 (m, 2H), 1.22 – 1.10 (m, 1H), 0.69 (s, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.40, 143.41, 136.32, 128.38, 127.20, 126.61, 125.88, 123.52, 120.97, 120.88, 118.37, 118.04, 111.39, 67.36, 48.77, 34.60, 34.03, 31.16, 28.37, 25.23, 24.61.

HRMS (ESI) *m/z* Calcd for $C_{23}H_{25}N_2O^+$[M+H]$^+$: 345.1961, found: 345.1966.



5j

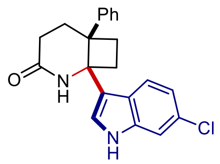
Brown solid. (R_f= 0.40, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5j** is a previously unknown compound and was synthesized in 50% yield, dr >20:1 (37.8 mg, 0.2 mmol scale) following the condition. *Note: the conditions are the same as **Condition [A]**, except that the reaction temperature of 50 ℃.*

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.91 (s, 1H), 7.94 (s, 1H), 7.47 (d, *J* = 2.7 Hz, 1H), 7.32 (d, *J* = 6.6 Hz, 2H), 7.28 – 7.18 (m, 4H), 6.63 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.33 (d, *J* = 2.6 Hz, 1H), 3.44 (s, 3H), 2.39 – 2.31 (m, 2H), 2.25 – 2.14 (m, 2H), 2.06 – 2.01 (m, 1H), 1.82 – 1.70 (m, 2H), 1.52 – 1.45 (m, 1H), 1.43 – 1.30 (m, 2H), 1.21 – 1.15 (m, 1H), 0.72 (s, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.46, 152.51, 143.52, 131.52, 128.50, 127.18, 126.52, 126.34, 124.17, 117.68, 111.80, 110.51, 103.54, 67.29, 55.14, 48.77, 34.67, 34.08, 31.14, 28.38, 25.18, 24.68.

HRMS (ESI) *m/z* Calcd for $C_{24}H_{27}N_2O_2^+$[M+H]$^+$: 375.2067, found: 375.2077.



5k

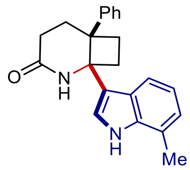
White solid. (R_f= 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5k** is a previously unknown compound and was synthesized in 85% yield, dr >20:1 (57.9 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.86 (s, 1H), 8.00 (s, 1H), 7.46 (d, J = 8.6 Hz, 1H), 7.15 (d, J = 1.9 Hz, 1H), 7.06 (d, J = 2.6 Hz, 1H), 6.88 – 6.83 (m, 3H), 6.82 – 6.74 (m, 3H), 2.66 – 2.55 (m, 1H), 2.50 – 2.41 (m, 1H), 2.36 – 2.27 (m, 3H), 2.24 – 2.10 (m, 2H), 2.02 – 1.92 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.32, 145.31, 136.85, 127.14, 126.27, 125.44, 125.32, 125.09, 124.55, 121.72, 118.73, 117.61, 111.01, 61.68, 49.66, 34.79, 34.30, 28.60, 25.63.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{20}ClN_2O^+$[M+H]$^+$: 351.1259, found: 351.1258.



5l

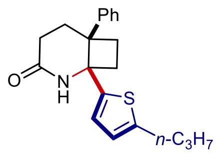
Yellow solid. (R_f = 0.45, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5l** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (90.3 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.37 (s, 1H), 7.54 (d, J = 8.1 Hz, 1H), 7.07 (d, J = 7.3 Hz, 2H), 7.03 – 6.86 (m, 5H), 6.66 (d, J = 2.7 Hz, 1H), 6.57 (s, 1H), 2.78 – 2.57 (m, 4H), 2.57 – 2.46 (m, 1H), 2.40 – 2.29 (m, 5H), 2.28 – 2.18 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 173.12, 144.85, 136.09, 127.57, 126.52, 126.00, 125.34, 122.57, 122.18, 120.69, 120.14, 118.06, 117.94, 62.60, 50.35, 35.48, 34.91, 29.01, 25.70, 16.64.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{23}N_2O^+$[M+H]$^+$: 331.1805, found: 331.1807.



5m

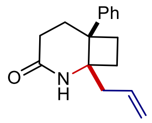
Pink oil. (R_f = 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5m** is a previously unknown compound and was synthesized in 35% yield, dr >20:1 (22.7 mg, 0.2 mmol scale) following the condition. *Note*: *the conditions are the same as* ***Condition [A]****, except that* ***Nu-H 2*** *(0.3 mmol, 1.5 equiv.), dioxazolone* *substrate **1** (0.2 mmol).*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.12 (t, *J* = 7.3 Hz, 2H), 7.06 (t, *J* = 7.1 Hz, 1H), 6.99 (d, *J* = 7.0 Hz, 2H), 6.65 (d, *J* = 3.6 Hz, 1H), 6.56 – 6.32 (m, 2H), 2.75 – 2.55 (m, 6H), 2.53 – 2.46 (m, 1H), 2.44 – 2.27 (m, 2H), 2.17 – 2.10 (m, 1H), 1.51 (h, *J* = 7.3 Hz, 2H), 0.81 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.83, 145.08, 144.70, 144.64, 127.72, 126.42, 126.07, 124.24, 123.68, 63.90, 50.80, 36.89, 35.54, 31.97, 28.66, 26.40, 24.80, 13.38.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{24}N_2OS^+[M+H]^+$: 326.1573, found: 326.1576.



5n

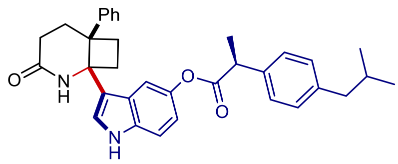
Yellow oil. (R_f = 0.45, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5n** is a previously unknown compound and was synthesized in 45% yield, dr >20:1 (21.7 mg, 0.2 mmol scale) following the **Condition [A]**. *Note: the conditions are the same as **Condition [A]**, except that HFIP (0.5 M) is used as the solvent.*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (t, *J* = 7.5 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 7.6 Hz, 2H), 6.18 (s, 1H), 5.78 – 5.68 (m, 1H), 5.18 (dd, *J* = 20.5, 13.5 Hz, 2H), 2.62 – 2.43 (m, 2H), 2.42 – 2.30 (m, 2H), 2.26 – 2.12 (m, 3H), 2.10 – 2.05 (m, 2H), 2.03 – 1.95 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.06, 144.44, 131.73, 128.31, 126.64, 126.53, 120.96, 59.13, 48.11, 41.60, 35.60, 31.32, 29.13, 25.36.

HRMS (ESI) *m/z* Calcd for $C_{16}H_{20}NO^+[M+H]^+$: 242.1539, found: 242.1547.



5o

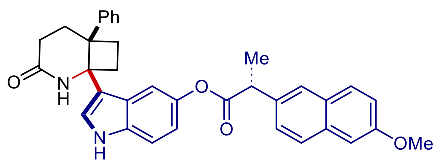
Colorless oil. (R_f = 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5o** is a previously unknown compound and was synthesized in 60% yield, dr >20:1 (61.3 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.65 – 8.62 (m, 1H), 7.43 – 7.33 (m, 2H), 7.19 (d, *J* = 7.2 Hz, 3H), 7.03 – 6.89 (m, 4H), 6.89 – 6.79 (m, 3H), 6.61 – 6.49 (m, 2H), 3.99 (q, *J* = 7.1 Hz, 1H), 2.76 – 2.53 (m, 4H), 2.50 (d, *J* = 7.2 Hz, 2H), 2.45 – 2.37 (m, 1H), 2.35 – 2.14 (m, 3H), 1.93 – 1.86 (m, 1H), 1.64 (dd, *J* = 7.1, 3.2 Hz, 3H), 0.94 (d, *J* = 6.8 Hz, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.50, 173.66, 144.42, 144.37, 144.17, 140.78, 140.75, 137.74, 137.69, 134.38, 129.60, 129.28, 127.53, 127.49, 127.36, 127.32, 126.40, 126.04, 126.01, 125.70, 125.67, 124.00, 117.18, 115.81, 112.14, 112.05, 111.92, 62.51, 62.48, 50.35, 45.33, 45.14, 35.06, 34.61, 34.43, 30.28, 30.20, 29.03, 25.28, 22.51, 22.47, 18.73.

HRMS (ESI) *m/z* Calcd for $C_{34}H_{37}N_2O_3^+$[M+H]$^+$: 521.2799, found: 521.2802.



5p

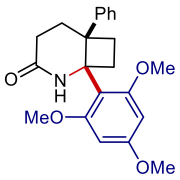
White solid. (R_f= 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5p** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (105.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.41 (t, *J* = 2.9 Hz, 1H), 7.84 – 7.73 (m, 3H), 7.55 (dt, *J* = 8.5, 1.9 Hz, 1H), 7.23 – 7.13 (m, 3H), 7.02 – 6.79 (m, 6H), 6.63 – 6.51 (m, 2H), 6.47 (d, *J* = 6.7 Hz, 1H), 4.14 (q, *J* = 7.1 Hz, 1H), 3.91 (s, 3H), 2.69 – 2.54 (m, 3H), 2.55 – 2.46 (m, 1H), 2.45 – 2.35 (m, 1H), 2.29 – 2.13 (m, 3H), 1.72 (dd, *J* = 7.1, 3.4 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.26, 173.28, 173.24, 157.73, 144.36, 144.31, 135.60, 135.57, 134.25, 133.83, 129.41, 129.39, 129.07, 127.47, 127.43, 126.37, 126.35, 126.32, 126.21, 125.97, 125.68, 123.79, 119.09, 117.55, 115.94, 115.86, 112.20, 112.08, 111.83, 105.69, 62.44, 62.40, 55.36, 50.34, 45.63, 45.61, 35.08, 34.53, 34.37, 29.11, 25.22, 25.15, 18.60.

HRMS (ESI) *m/z* Calcd for $C_{35}H_{33}N_2O_4^+$[M+H]$^+$: 545.2435, found: 545.2439.



5q

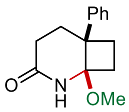
White solid. (R_f = 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **5q** is a previously unknown compound and was synthesized in 89% yield, dr >20:1 (64.9 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 (d, J = 7.5 Hz, 2H), 7.12 (t, J = 7.5 Hz, 2H), 7.04 (t, J = 7.2 Hz, 1H), 5.96 (s, 1H), 5.82 (s, 2H), 3.69 (s, 3H), 3.62 – 3.27 (m, 7H), 2.91 – 2.77 (m, 1H), 2.73 – 2.59 (m, 2H), 2.57 – 2.47 (m, 2H), 2.08 (m, 1H), 1.95 – 1.82 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.18, 160.71, 158.69, 146.44, 127.07, 125.49, 125.38, 110.70, 90.35, 64.91, 55.12, 54.72, 48.45, 37.78, 28.40, 27.23.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{26}NO_4^+$[M+H]$^+$: 368.1856, found: 368.1860.



6a

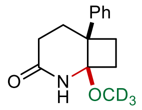
Yellow crystal. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6a** is a previously unknown compound and was synthesized in 73% yield, dr >20:1 (33.6 mg, 0.2 mmol scale) following the **Condition [A]**. *Note: the conditions are the same as **Condition [A]**, except that the reaction temperature of 50 °C.*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (s, 1H), 7.34 (d, J = 4.3 Hz, 4H), 7.25 – 7.22 (m, 1H), 3.12 (s, 3H), 2.56 – 2.46 (m, 2H), 2.40 – 2.31 (m, 2H), 2.30 – 2.19 (m, 2H), 2.17 – 2.09 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.62, 142.92, 128.02, 127.28, 126.51, 89.20, 50.10, 49.20, 34.57, 30.37, 28.66, 27.10.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{17}NNaO_2^+$[M+Na]$^+$: 254.1151, found: 254.1157.



6a-*d*

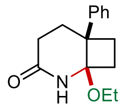
Yellow crystal. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6a**-*d* is a previously unknown compound and was synthesized in 84% yield, dr >20:1 (39.3 mg, 0.2 mmol scale) following the **Condition [A]**. *Note: the conditions are the same as* ***Condition [A]****, except that the reaction temperature of 50 ℃.*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 – 7.56 (m, 1H), 7.34 (d, *J* = 4.3 Hz, 4H), 7.25 – 7.22 (m, 1H), 2.55 – 2.46 (m, 2H), 2.42 – 2.30 (m, 2H), 2.30 – 2.19 (m, 2H), 2.16 – 2.09 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.70, 142.93, 128.00, 127.27, 126.48, 89.14, 49.17, 34.58, 30.32, 28.64, 27.09.

HRMS (ESI) *m/z* Calcd for $C_{14}H_{15}D_3NO_2^+$[M+H]$^+$: 235.1520, found: 235.1526.



6b

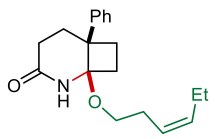
Yellow solid. (R_f = 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6b** is a previously unknown compound and was synthesized in 84% yield, dr >20:1 (42.2 mg, 0.2 mmol scale) following the **Condition [A]**. *Note: the conditions are the same as* ***Condition [A]****, except that the reaction temperature of 50 ℃.*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 – 7.36 (m, 3H), 7.33 (t, *J* = 7.7 Hz, 2H), 7.28 – 7.20 (m, 1H), 3.35 – 3.19 (m, 2H), 2.61 – 2.46 (m, 2H), 2.40 (dt, *J* = 17.1, 5.5 Hz, 1H), 2.35 – 2.22 (m, 2H), 2.19 (dd, *J* = 7.9, 5.1 Hz, 2H), 2.15 – 2.06 (m, 1H), 0.94 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.48, 142.97, 127.84, 127.31, 126.35, 88.82, 58.26, 49.25, 33.63, 31.77, 28.70, 26.65, 15.25.

HRMS (ESI) *m/z* Calcd for $C_{15}H_{20}NO_2^+$[M+H]$^+$: 246.1489, found: 246.1496.



6c

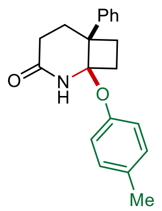
Yellow oil. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6c** is a previously unknown compound and was synthesized in 77% yield dr >20:1 (45.1 mg, 0.2 mmol scale) following the **Condition [A]**. *Note: the conditions are the same as **Condition [A]**, except that the reaction temperature of 50 ℃.*

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.36 (m, 2H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.26 – 7.17 (m, 2H), 5.39 – 5.31 (m, 1H), 5.14 – 5.07 (m, 1H), 3.27 – 3.15 (m, 2H), 2.60 – 2.45 (m, 2H), 2.40 (dt, *J* = 17.2, 5.4 Hz, 1H), 2.36 – 2.23 (m, 2H), 2.20 – 2.17 (m, 2H), 2.16 – 2.09 (m, 1H), 2.04 (p, *J* = 7.2 Hz, 2H), 1.86 (p, *J* = 7.3 Hz, 2H), 0.86 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.41, 142.91, 133.81, 127.87, 127.36, 126.39, 124.60, 88.80, 62.40, 49.34, 33.71, 31.74, 28.74, 27.76, 26.68, 20.51, 14.30.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{26}NO_2^+$[M+H]$^+$: 300.1958, found: 300.1966.



6d

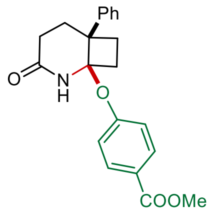
White solid. (R_f = 0.70, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6d** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (60.8 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 4.4 Hz, 4H), 7.29 (q, *J* = 4.4 Hz, 1H), 7.16 (s, 1H), 7.04 (d, *J* = 8.1 Hz, 2H), 6.65 (d, *J* = 8.1 Hz, 2H), 2.71 – 2.64 (m, 1H), 2.62 – 2.47 (m, 2H), 2.46 – 2.36 (m, 2H), 2.36 – 2.29 (m, 1H), 2.27 (s, 3H), 2.20 – 2.13 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.66, 150.92, 142.66, 132.26, 130.12, 127.92, 127.36, 126.44, 119.09, 89.29, 50.02, 35.47, 31.60, 28.54, 27.17, 20.58.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{22}NO_2^+$[M+H]$^+$: 308.1645, found: 308.1653.



6e

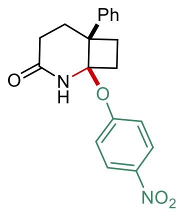
Pink oil. (R_f= 0.65, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6e** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (69.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.71 (s, 1H), 7.39 – 7.33 (m, 4H), 7.29 – 7.27 (m, 1H), 6.71 (d, *J* = 8.4 Hz, 2H), 3.86 (s, 3H), 2.72 – 2.61 (m, 1H), 2.61 – 2.47 (m, 3H), 2.46 – 2.27 (m, 2H), 2.23 – 2.17 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.22, 166.53, 157.29, 142.22, 131.52, 128.01, 127.24, 126.61, 124.00, 117.58, 89.36, 51.98, 50.10, 35.42, 31.90, 28.67, 27.44.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{22}NO_4^+$[M+H]$^+$: 352.1543, found: 352.1535.



6f

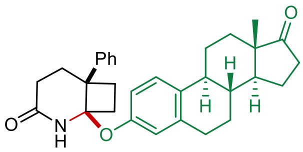
White solid. (R_f= 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6f** is a previously unknown compound and was synthesized in 93% yield, dr >20:1 (67.0 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (s, 1H), 8.08 (d, *J* = 9.2 Hz, 2H), 7.40 – 7.33 (m, 4H), 7.30 – 7.27 (m, 1H), 6.76 (d, *J* = 9.2 Hz, 2H), 2.66 – 2.53 (m, 4H), 2.45 (dt, *J* = 16.9, 4.3 Hz, 1H), 2.38 – 2.17 (m, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.04, 158.82, 142.36, 141.81, 128.16, 127.17, 126.87, 125.67, 117.91, 89.71, 50.17, 35.40, 31.98, 28.90, 27.66.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{19}N_2O_4^+$[M+H]$^+$: 339.1339, found: 339.1345.



6g

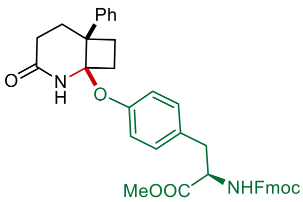
Yellow solid. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6g** is a previously unknown compound and was synthesized in 73% yield, dr >20:1 (68.3 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.33 (m, 4H), 7.29 – 7.27 (m, 1H), 7.14 (d, *J* = 8.5 Hz, 1H), 6.85 (d, *J* = 6.8 Hz, 1H), 6.53 (dd, *J* = 8.5, 2.7 Hz, 1H), 6.47 (d, *J* = 2.6 Hz, 1H), 2.91 – 2.79 (m, 2H), 2.75 – 2.69 (m, 1H), 2.58 – 2.44 (m, 4H), 2.40 (q, *J* = 3.7, 2.9 Hz, 1H), 2.39 – 2.31 (m, 2H), 2.26 – 2.17 (m, 2H), 2.17 – 2.11 (m, 2H), 2.10 – 2.02 (m, 1H), 2.01 – 1.95 (m, 2H), 1.69 – 1.58 (m, 1H), 1.57 – 1.49 (m, 2H), 1.47 (d, *J* = 9.5 Hz, 2H), 1.44 – 1.34 (m, 1H), 0.90 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 220.82, 172.45, 151.08, 142.69, 138.36, 134.36, 128.00, 127.40, 126.67, 126.51, 118.97, 118.87, 116.15, 116.09, 89.29, 89.27, 50.50, 50.06, 48.06, 44.05, 38.29, 35.94, 35.49, 31.91, 31.65, 29.64, 28.48, 27.26, 26.50, 25.92, 21.67, 13.94.

HRMS (ESI) *m/z* Calcd for $C_{31}H_{36}NO_3^+$[M+H]$^+$: 470.2690, found: 470.2696.



6h

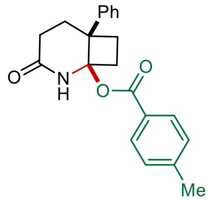
White solid. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6h** is a previously unknown compound and was synthesized in 79% yield, dr >20:1 (97.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 7.5 Hz, 2H), 7.56 – 7.51 (m, 2H), 7.42 – 7.30 (m, 6H), 7.27 (t, *J* = 7.4 Hz, 3H), 7.13 (d, *J* = 8.3 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 2H), 6.62 – 6.59 (m, 2H), 5.53 (dd, *J* = 20.2, 8.3 Hz, 1H), 4.59 (q, *J* = 6.4 Hz, 1H), 4.41 – 4.35 (m, 1H), 4.29 (dd, *J* = 10.7, 6.9 Hz, 1H), 4.18 – 4.13 (m, 1H), 3.67 (s, 3H), 3.06 – 2.94 (m, 2H), 2.62 – 2.50 (m, 2H), 2.47 – 2.34 (m, 3H), 2.29 – 2.25 (m, 1H), 2.15 – 2.10 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.67, 172.64, 171.96, 155.59, 152.28, 143.83, 143.78, 143.75, 142.46, 141.29, 130.49, 130.07, 127.89, 127.71, 127.28, 127.06, 126.45, 125.10, 119.97, 118.88, 118.81, 89.25, 89.21, 66.87, 54.85, 52.33, 50.01, 47.14, 37.28, 35.33, 31.59, 28.56, 27.08.

HRMS (ESI) *m/z* Calcd for $C_{38}H_{37}N_2O_6^+$[M+H]$^+$: 617.2646, found: 617.2648.



6i

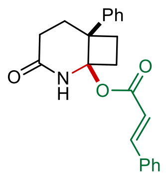
Pink oil. (R_f = 0.30, DCM/MeOH = 50:1, v/v)

Compound **6i** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (65.1 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63 – 7.47 (m, 3H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.35 – 7.32 (m, 3H), 7.10 (d, *J* = 7.9 Hz, 2H), 2.80 – 2.61 (m, 2H), 2.60 – 2.48 (m, 2H), 2.48 – 2.37 (m, 2H), 2.33 (s, 3H), 2.30 – 2.23 (m, 1H), 2.17 – 2.10 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.70, 166.15, 144.44, 142.46, 129.80, 129.21, 128.09, 127.20, 126.97, 126.74, 90.31, 48.76, 35.29, 32.93, 27.99, 27.81, 21.72.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{21}NNaO_3^+$[M+Na]$^+$: 358.1414, found: 358.1420.



6j

Yellow solid. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

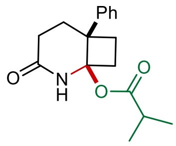
Compound **6j** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (68.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.42 (m, 2H), 7.39 (d, *J* = 3.1 Hz, 1H), 7.38 – 7.34 (m, 5H), 7.32 (td, *J* = 3.5, 1.2 Hz, 3H), 7.30 (t, *J* = 1.5 Hz, 1H), 6.21 (d, *J* = 16.0 Hz, 1H), 2.73 – 2.67 (m, 1H), 2.65 – 2.59 (m, 1H), 2.56 – 2.50 (m, 1H), 2.50 –

2.44 (m, 1H), 2.43 – 2.31 (m, 2H), 2.28 – 2.22 (m, 1H), 2.14 – 2.09 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.61, 166.40, 146.52, 142.55, 133.96, 130.79, 128.98, 128.30, 128.11, 127.23, 126.68, 117.63, 90.18, 48.70, 35.34, 32.89, 27.94, 27.75.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{21}NNaO_3{}^+$[M+Na]$^+$: 370.1414, found: 370.1416.



6k

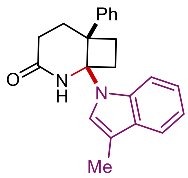
Yellow oil. (R_f= 0.70, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **6k** is a previously unknown compound and was synthesized in 81% yield, dr >20:1 (46.6 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (dd, *J* = 13.9, 6.1 Hz, 4H), 7.24 (t, *J* = 8.4 Hz, 2H), 2.72 – 2.57 (m, 1H), 2.56 – 2.43 (m, 3H), 2.43 – 2.32 (m, 3H), 2.26 – 2.19 (m, 1H), 2.16 – 2.05 (m, 1H), 0.99 (dd, *J* = 12.9, 7.0 Hz, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 176.94, 171.73, 142.34, 128.00, 127.07, 126.64, 89.91, 48.66, 35.21, 34.40, 33.01, 27.93, 27.68, 18.54, 18.40.

HRMS (ESI) *m/z* Calcd for $C_{17}H_{21}NNaO_3{}^+$[M+Na]$^+$: 310.1414, found: 310.1417.



7a

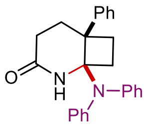
White solid. (R_f= 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7a** is a previously unknown compound and was synthesized in 56% yield, dr >20:1 (38.9 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 – 7.36 (m, 2H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.08 (t, *J* = 7.4 Hz, 1H), 7.03 – 6.99 (m, 1H), 6.96 (d, *J* = 4.2 Hz, 4H), 6.62 (s, 1H), 6.51 (s, 1H), 3.25 – 3.12 (m, 1H), 2.86 – 2.75 (m, 2H), 2.58 – 2.50 (m, 1H), 2.50 – 2.45 (m, 1H), 2.42 – 2.38 (m, 2H), 2.05 (s, 3H), 2.01 – 1.94 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.50, 140.18, 135.72, 130.65, 127.97, 126.98, 126.23, 123.19, 122.14, 119.58, 119.12, 112.58, 111.46, 76.29, 51.74, 34.19, 30.10, 28.85, 21.36, 9.39.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{23}N_2NaO^+$[M+H]$^+$: 311.1805, found: 311.1811.



7b

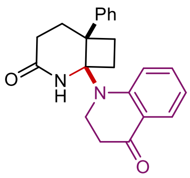
Pink solid. (R_f = 0.65, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7b** is a previously unknown compound and was synthesized in 96% yield, dr >20:1 (66.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 – 7.41 (m, 2H), 7.24 – 7.21 (m, 3H), 7.11 (t, *J* = 7.9 Hz, 4H), 7.05 (s, 1H), 6.93 (t, *J* = 7.3 Hz, 2H), 6.47 (d, *J* = 7.6 Hz, 4H), 2.84 – 2.73 (m, 2H), 2.73 – 2.65 (m, 1H), 2.37 – 2.24 (m, 1H), 2.23 – 2.07 (m, 2H), 2.01 – 1.94 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.46, 144.93, 141.50, 128.90, 128.10, 127.62, 127.06, 124.34, 122.87, 77.89, 50.83, 36.82, 29.02, 28.23, 21.55.

HRMS (ESI) *m/z* Calcd for $C_{25}H_{25}N_2O^+$[M+H]$^+$: 369.1961, found: 369.1965.



7c

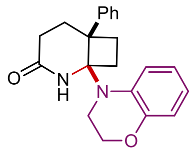
White solid. (R_f = 0.25, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7c** is a previously unknown compound and was synthesized in 82% yield, dr >20:1 (56.0 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.58 (d, *J* = 7.7 Hz, 2H), 7.39 – 7.33 (m, 3H), 7.30 (d, *J* = 7.3 Hz, 1H), 6.86 (t, *J* = 7.5 Hz, 1H), 6.71 (d, *J* = 8.4 Hz, 1H), 6.50 (s, 1H), 3.29 – 3.21 (m, 1H), 3.01 (ddd, *J* = 15.0, 12.1, 3.1 Hz, 1H), 2.90 – 2.72 (m, 3H), 2.67 – 2.45 (m, 3H), 2.22 – 2.05 (m, 2H), 1.90 (dt, *J* = 17.7, 2.7 Hz, 1H), 1.16 – 0.98 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 193.84, 172.36, 148.94, 143.03, 134.51, 129.38, 128.23, 127.47, 127.38, 122.85, 119.49, 114.72, 78.09, 47.96, 45.14, 38.70, 37.71, 28.52, 26.80, 26.11.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{22}N_2NaO_2^+$[M+Na]$^+$: 369.1573, found: 369.1581.



7d

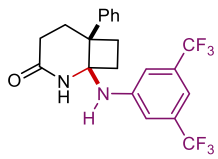
Pale yellow oil. (R_f= 0.45, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7d** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (66.1 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 7.8 Hz, 2H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.28 (d, *J* = 7.1 Hz, 1H), 6.82 – 6.70 (m, 3H), 6.69 – 6.62 (m, 1H), 6.52 (s, 1H), 3.68 (d, *J* = 10.2 Hz, 1H), 3.30 (q, *J* = 11.2 Hz, 1H), 2.95 (t, *J* = 8.9 Hz, 1H), 2.82 – 2.70 (m, 3H), 2.61 – 2.55 (m, 1H), 2.51 – 2.42 (m, 2H), 2.33 (d, *J* = 11.9 Hz, 1H), 2.16 – 2.14 (m, 1H), 2.10 – 1.97 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.42, 145.98, 143.05, 131.60, 128.00, 127.17, 127.10, 120.51, 120.15, 116.98, 114.29, 77.71, 65.51, 47.57, 40.80, 38.20, 28.59, 27.12, 26.50.

HRMS (ESI) *m/z* Calcd for $C_{21}H_{23}N_2O_2^+$[M+H]$^+$: 335.1754, found: 335.1762.



7e

White solid. (R_f= 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

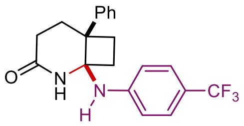
Compound **7e** is a previously unknown compound and was synthesized in 99% yield, dr >20:1 (84.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.76 (s, 1H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.36 (t, *J* = 7.3 Hz, 1H), 7.30 – 7.17 (m, 3H), 6.81 (s, 2H), 4.80 (s, 1H), 2.70 – 2.59 (m, 1H), 2.58 – 2.44 (m, 4H), 2.41 – 2.34 (m, 1H), 2.24 – 2.15 (m, 1H), 2.14 – 2.05 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.90, 142.74, 141.47, 132.50 (q, J = 33.1 Hz), 129.38, 127.73, 126.48, 123.42 (q, J = 273.7 Hz), 114.19 (q, J = 3.6 Hz), 111.83, 111.79, 111.75, 70.26, 49.78, 34.83, 32.22, 28.92, 25.79.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -63.12.

HRMS (ESI) *m/z* Calcd for: $C_{21}H_{19}F_6N_2O^+$[M+H]$^+$: 429.1396, found: 429.1395.



7f

White solid. (R_f = 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

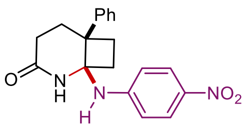
Compound **7f** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (69.9 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 (t, J = 7.5 Hz, 2H), 7.39 – 7.34 (m, 3H), 7.22 (d, J = 7.6 Hz, 2H), 6.79 (s, 1H), 6.46 (d, J = 8.3 Hz, 2H), 4.63 (s, 1H), 2.76 – 2.63 (m, 1H), 2.62 – 2.52 (m, 2H), 2.52 – 2.45 (m, 2H), 2.43 – 2.36 (m, 1H), 2.20 – 2.14 (m, 1H), 2.13 – 2.05 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.23, 145.30, 141.56, 129.33, 127.62, 126.87 (q, J = 3.8 Hz), 126.45, 124.66 (q, J = 271.0 Hz), 120.58 (q, J = 32.3 Hz), 113.84, 70.17, 49.57, 34.98, 32.49, 28.98, 25.91.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -61.32.

HRMS (ESI) *m/z* Calcd for $C_{20}H_{20}F_3N_2O^+$[M+H]$^+$: 361.1522, found: 361.1519.



7g

Yellow solid. (R_f = 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

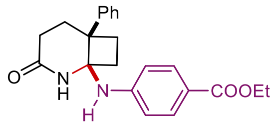
Compound **7g** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (65.5 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 (d, J = 9.1 Hz, 2H), 7.43 (t, J = 7.5 Hz, 2H), 7.37 – 7.30 (m, 1H), 7.24 – 7.17 (m, 2H), 7.11 (s, 1H), 6.41 (d, J = 9.2 Hz, 2H), 5.07

(s, 1H), 2.71 – 2.60 (m, 1H), 2.59 – 2.44 (m, 4H), 2.41 – 2.34 (m, 1H), 2.24 – 2.14 (m, 1H), 2.13 – 2.03 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.32, 148.31, 141.02, 139.27, 129.33, 127.72, 126.39, 126.04, 113.37, 70.07, 49.56, 34.64, 32.59, 28.84, 25.75.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{20}N_3O_3^+$[M+H]$^+$: 338.1499, found: 338.1496.



7h

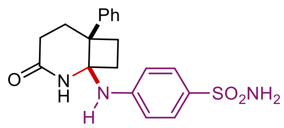
Colorless oil. (R_f = 0.45, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7h** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (70.7 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 (dd, *J* = 8.6, 3.1 Hz, 2H), 7.44 – 7.39 (m, 2H), 7.34 – 7.29 (m, 1H), 7.20 – 7.17 (m, 2H), 6.93 – 6.79 (m, 1H), 6.38 (dd, *J* = 8.7, 2.7 Hz, 2H), 4.70 (s, 1H), 4.29 – 4.23 (m, 2H), 2.69 – 2.61 (m, 1H), 2.59 – 2.42 (m, 4H), 2.42 – 2.27 (m, 1H), 2.20 – 1.99 (m, 2H), 1.31 (td, *J* = 7.1, 3.5 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.10, 166.38, 146.40, 141.43, 131.46, 129.19, 127.48, 126.37, 120.43, 113.36, 70.04, 60.37, 49.43, 34.83, 32.46, 28.84, 25.81, 14.37.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{25}N_2O_3^+$[M+H]$^+$: 365.1860, found: 365.1867.



7i

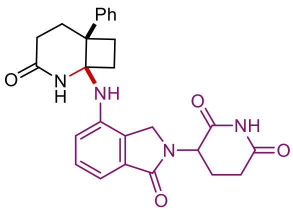
Brown solid. (R_f = 0.15, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7i** is a previously unknown compound and was synthesized in 82% yield, dr >20:1 (61.0 mg, 0.2 mmol scale) following the **Condition [A]**.

^{1}H NMR (400 MHz, Methanol-d_4) δ 7.55 – 7.51 (m, 2H), 7.45 – 7.36 (m, 2H), 7.35 – 7.26 (m, 2H), 7.25 – 7.16 (m, 1H), 6.49 (d, *J* = 8.9 Hz, 2H), 2.78 – 2.69 (m, 1H), 2.69 – 2.56 (m, 2H), 2.52 – 2.32 (m, 3H), 2.27 – 2.17 (m, 2H).

^{13}**C NMR** (101 MHz, Methanol-*d*$_4$) δ 175.86, 148.57, 143.14, 132.85, 129.43, 128.36, 128.13, 128.01, 115.12, 72.70, 50.99, 35.23, 32.89, 29.93, 25.63.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{22}N_3O_3S^+$[M+H]$^+$: 372.1376, found: 372.1383.



7j

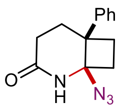
White solid. (R$_f$= 0.10, PE/EA/MeOH = 2:1:0.2, v/v/v)

Compound **7j** is a previously unknown compound and was synthesized in 88% yield, dr = 2:1 (81.1mg 0.2 mmol scale) following the **Condition [A]**.

^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 10.06 (d, *J* = 4.8 Hz, 0.95H), 9.69 (d, *J* = 9.5 Hz, 0.51H), 7.67 (s, 0.95H), 7.42 (q, *J* = 7.2 Hz, 2.61H), 7.39 – 7.31 (m, 2.43H), 7.29 – 7.14 (m, 5.14H), 7.08 (d, *J* = 7.5 Hz, 1.05H), 6.68 (dd, *J* = 12.5, 7.9 Hz, 1.47H), 5.10 – 4.99 (m, 1.47H), 4.14 (s, 0.50H), 4.00 (s, 1H), 3.65 – 3.61 (m, 2.27H), 3.37 (d, *J* = 15.6 Hz, 1.16H), 2.77 – 2.73 (m, 2.95H), 2.66 (d, *J* = 12.4 Hz, 1.64H), 2.60 (t, *J* = 6.7 Hz, 2.86H), 2.52 – 2.41 (m, 3.32H), 2.36 – 2.27 (m, 1.68H), 2.23 – 2.08 (m, 3.48H), 2.08 – 1.91 (m, 2.71H).

^{13}**C NMR** (101 MHz, Chloroform-*d*) δ 173.15, 172.86, 172.11, 170.68, 170.34, 169.58, 169.44, 141.70, 141.53, 137.62, 137.24, 132.51, 132.29, 129.18, 129.06, 128.91, 128.08, 127.83, 127.44, 127.36, 126.48, 126.42, 115.78, 115.43, 114.18, 70.33, 70.07, 51.73, 51.54, 49.74, 49.56, 44.35, 43.98, 34.18, 33.92, 32.82, 32.39, 31.51, 29.12, 28.95, 25.40, 25.11, 23.13.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{27}N_4O_4^+$[M+H]$^+$: 459.2027, found: 459.2033.



7k

Yellow solid. (R$_f$= 0.25, DCM/MeOH = 50:1, v/v)

Compound **7k** is a previously unknown compound and was synthesized in 95% yield, dr >20:1 (46.0 mg, 0.2 mmol scale) following the **Condition [A]**.

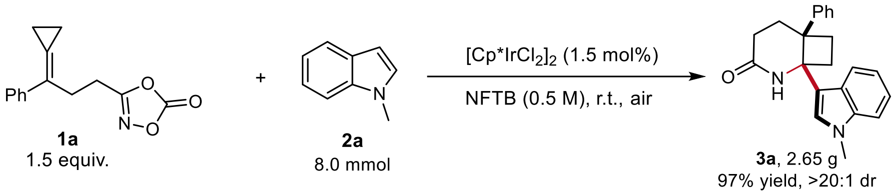
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (s, 1H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.33 – 7.24 (m, 3H), 2.68 – 2.46 (m, 4H), 2.45 – 2.32 (m, 2H), 2.28 – 2.22 (m, 1H), 2.13 – 2.07 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.17, 142.46, 128.28, 126.86, 126.59, 77.56, 50.17, 34.94, 32.40, 27.78, 26.69.

HRMS (ESI) *m/z* Calcd for $C_{13}H_{15}N_4O^+$[M+H]$^+$: 243.1240, found: 243.1246.

5. Transformations of δ-lactams

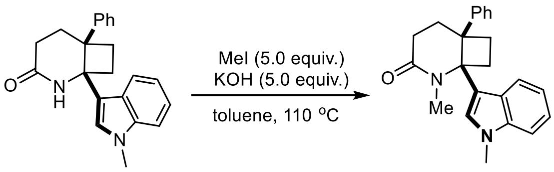
5.1 Gram-scale synthesis of 3a



Supplementary Figure 6. Gram-scale synthesis of **3a**

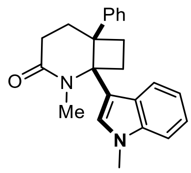
To a 25 mL round-bottom flask equipped with a rod-like-shaped Teflon stir bar were added $[Cp^*IrCl_2]_2$ catalyst (95.6 mg, 0.15 mmol, 1.5 mol%), **2a** (8.0 mmol), NFTB (8 mL, 1.0 M) at 25 °C, dioxazolone substrate **1a** (2.92 g, 12.0 mmol, 1.5 equiv.) was added under air atmosphere. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 25 °C for 5 h. Water was added and the mixture was extracted with ethyl acetate (EA). The combined organic layer was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (3:1:0 to 2:1:0.2, v/v/v) to give the δ-lactam **3a** product.

5.2 Access to *N*-methylated product



Supplementary Figure 7. *N*-methylation reaction of **3a**

To a solution of compound **3a** (66.1 mg, 0.2 mmol, 1.0 equiv.) in toluene (1 mL) in a 10 mL seal tube, MeI (65 μL, 1.0 mmol, 5.0 equiv.) and KOH (56.1 mg, 1.0 mmol, 5.0 equiv.) were added successively. The tube was sealed and heated to 110 °C for 12 hours. After being cooled to room temperature, the mixture was diluted with ethyl acetate (10 mL) and water (10 mL). The organic layer was separated, washed with brine, dried over anhydrous Na_2SO_4 and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (3:1:0 to 2:1:0.2, v/v/v) to give the target product.



8a

Pale yellow solid. (R_f= 0.5, PE/EA/MeOH = 2:1:0.2, v/v/v)

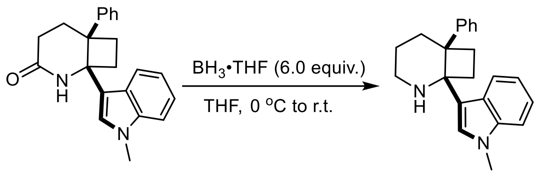
Compound **8a** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (66.8 mg, 0.2 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 7.6 Hz, 1H), 7.17 – 7.12 (m, 3H), 6.99 – 6.96 (m, 2H), 6.93 – 6.83 (m, 3H), 6.75 (s, 1H), 3.53 (s, 3H), 2.83 – 2.74 (m, 4H), 2.69 – 2.50 (m, 4H), 2.46 – 2.39 (m, 2H), 2.13 (dt, *J* = 13.6, 4.5 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.35, 145.33, 136.95, 127.28, 127.19, 126.45, 125.61, 121.49, 120.38, 120.09, 115.30, 109.35, 67.22, 51.28, 34.83, 32.70, 32.52, 30.01, 28.95, 26.78.

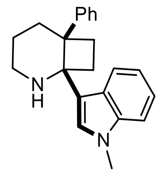
HRMS (ESI) *m/z* Calcd for $C_{23}H_{24}N_2NaO^+$[M+Na]$^+$: 367.1781, found: 367.1784.

5.3 Access to piperidine



Supplementary Figure 8. Reduction reaction of **3a**

To a solution of compound **3a** (66.1 mg, 0.2 mmol, 1.0 equiv.) in dry THF (3 mL) in a 10 mL seal tube, a solution of 1.2 mL of BH_3·THF complex was slowly added with cooling by ice and then stirred for 1 hours with cooling by ice and for 12 hours at room temperature. Then, methanol was slowly added the reaction solution and heated to 70 °C for 5 hours. After being cooled to room temperature, the mixture was diluted with ethyl acetate (10 mL) and water (10 mL). The organic layer was separated, washed with brine, dried over anhydrous Na_2SO_4 and concentrated in vacuo. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (3:1:0 to 2:1:0.2, v/v/v) to give the target product.



8b

Pale yellow solid. (R_f= 0.25, PE/EA/MeOH = 2:1:0.2, v/v/v)

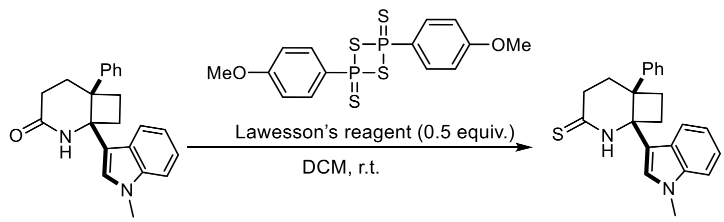
Compound **8b** is a previously unknown compound and was synthesized in 62% yield, dr >20:1 (39.0 mg, 0.2 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.1 Hz, 1H), 7.29 (d, *J* = 7.2 Hz, 2H), 7.18 (t, *J* = 7.3 Hz, 2H), 7.16 – 7.07 (m, 3H), 6.97 (ddd, *J* = 8.0, 6.3, 1.6 Hz, 1H), 5.99 (s, 1H), 3.47 (s, 3H), 3.34 – 3.15 (m, 2H), 2.99 (q, *J* = 10.0 Hz, 1H), 2.30 – 2.20 (m, 2H), 2.17 – 2.02 (m, 2H), 1.92 – 1.79 (m, 2H), 1.62 – 1.56 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 148.57, 136.92, 127.72, 127.34, 127.14, 126.60, 125.53, 122.12, 121.27, 118.37, 109.01, 61.80, 48.14, 42.02, 38.68, 32.66, 28.40, 27.11, 22.37.

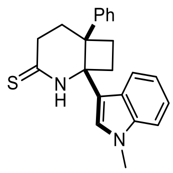
HRMS (ESI) *m/z* Calcd for $C_{22}H_{25}N_2^+$[M+H]$^+$: 317.2012, found: 317.2017.

5.4 Access to thioamide



Supplementary Figure 9. Oxygen-sulfur exchange reaction of **3a**

To a reaction vial was added **3a** (66.1 mg, 0.2 mmol), Lawesson's reagent (40.4 mg, 0.1 mmol) and anhydrous DCM (1.0 mL) was added under air atmosphere, and the reaction was stirred at room temperature for 12 h. The crude material was poured into deionized water, extracted with EA for three times, and dried under reduced pressure. The residue was purified by flash chromatography (PE/EA) to afford the desired product.



8c

White solid. (R_f= 0.80, PE/EA/MeOH = 2:1:0.2, v/v/v)

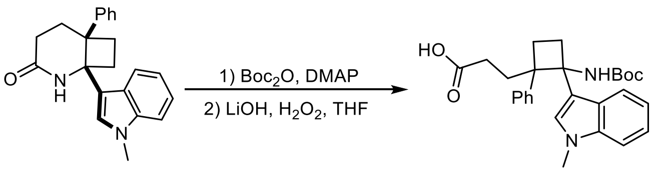
Compound **8c** is a previously unknown compound and was synthesized in 97% yield, dr >20:1 (67.2 mg, 0.2 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.76 (s, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.17 (d, *J* = 4.0 Hz, 2H), 7.14 – 7.09 (m, 1H), 7.07 – 7.01 (m, 2H), 7.01 – 6.92 (m, 3H), 6.66 (s, 1H), 3.57 (s, 3H), 3.29 – 3.12 (m, 2H), 2.80 – 2.71 (m, 1H), 2.70 – 2.56 (m, 2H), 2.42 – 2.33 (m, 1H), 2.32 – 2.24 (m, 1H), 2.21 – 2.14 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 202.89, 144.18, 137.19, 127.58, 127.30, 126.46, 126.16, 126.10, 121.92, 120.20, 120.07, 114.65, 109.61, 64.64, 49.64, 36.92, 34.46, 34.40, 32.87, 25.85.

HRMS (ESI) *m/z* Calcd for $C_{22}H_{23}N_2S^+[M+H]^+$: 347.1576, found: 347.1582.

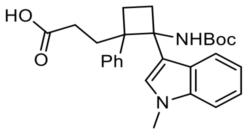
5.5 Access to Boc-protected *δ*-amino acid



Supplementary Figure 10. Hydrolysis of **3a**

To a solution of compound **3a** (132.17 mg, 0.4 mmol, 1.0 equiv.) in dry THF (10 mL) was added 4-dimethylaminopyridine (48.9 mg, 0.4 mmol, 1.0 equiv.) and Boc anhydride (1.0 mmol, 2.5 equiv.). The mixture was stirred at room temperature for 48 hours then concentrated in vacuo. Purification of the crude mixture by flash silica gel chromatography gave the Boc-protected amide in 85% yield.

A solution of Boc-protected amide in THF (5 mL) was cooled to 0 ºC, then 30% hydrogen peroxide (1.0 mmol, 5.0 equiv.) and lithium hydroxide monohydrate (1.0 mmol, 5 equiv.) were added. The reaction mixture was stirred at 0 ºC for 2.5 hours then quenched with 1.5 M aqueous sodium thiosulfate (1.2 mL) at 0 ºC. The organic solvent was removed under reduced pressure, and the residue was washed with dichloromethane (10 mL) twice. Then the aqueous phase was acidified to pH = 2 with 10% aqueous hydrochloric acid and extracted with ethyl acetate (10 mL) twice. The organic phase was dried over sodium sulfate and concentrated in vacuo. The resulting residue was purified by flash silica gel chromatography using PE/EA/MeOH eluent (3:1:0 to 2:1:0.2, v/v/v) to give compound **8d**.



8d

Green solid. (R_f = 0.60, PE/EA/MeOH = 2:1:0.2, v/v/v)

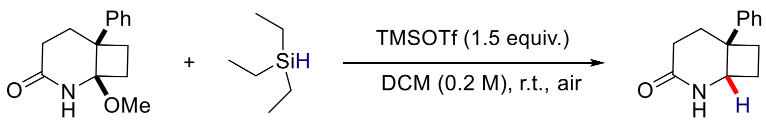
Compound **8d** is a previously unknown compound and was synthesized in 60% yield (54.5 mg, 0.2 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.72 (d, J = 8.0 Hz, 1H), 7.16 (d, J = 7.4 Hz, 2H), 7.04 (d, J = 4.2 Hz, 4H), 6.96 – 6.93 (m, 2H), 6.24 (s, 1H), 3.46 (s, 3H), 2.81 – 2.46 (m, 5H), 2.29 – 2.22 (m, 1H), 2.10 – 2.03 (m, 1H), 1.79 – 1.72 (m, 1H), 0.88 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.51, 158.26, 143.33, 136.85, 127.50, 127.20, 127.06, 126.27, 125.93, 121.36, 120.94, 118.65, 108.57, 80.62, 62.38, 55.69, 33.62, 33.38, 32.53, 30.26, 27.91, 23.56.

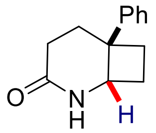
HRMS (ESI) *m/z* Calcd for $C_{27}H_{32}N_2NaO_4^+$[M+Na]$^+$: 471.2254, found: 471.2255.

5.6 Access to 6-phenyl-2-azabicyclo[4.2.0]octan-3-one



Supplementary Figure 11. Reduction reaction of **6a**

To an oven-dried screwed 4 mL glass vial equipped with a rod-like-shaped Teflon stir bar were added **6a** (46.3 mg, 0.2 mmol), triethylsilane (0.5 mmol, 2.5 equiv.), DCM (1 mL, 0.2 M), and TMSOTf (0.3 mmol, 1.5 equiv.) under air atmosphere. The vial was then sealed. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 25 °C overnight. Upon completion of the reaction, the mixture was diluted with DCM and quenched with sat. aq. $NaHCO_3$. The organic layer was separated, and the aqueous layer was extracted three more times with DCM. The combined organic layers were then dried with Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (2:1:0.2, v/v/v) to give the **8e**.



8e

White solid. (R_f = 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

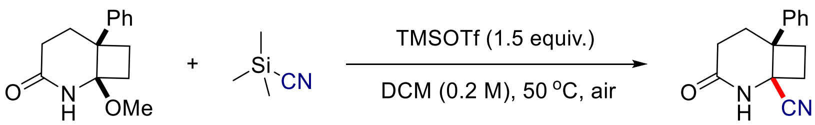
Compound **8e** is a previously unknown compound and was synthesized in 99% yield (43.2 mg, 0.2 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.32 (m, 2H), 7.26 – 7.18 (m, 2H), 7.18 – 7.12 (m, 2H), 4.16 (td, *J* = 7.3, 2.9 Hz, 1H), 2.43 (td, *J* = 12.9, 4.1 Hz, 1H), 2.37 – 2.25 (m, 2H), 2.24 – 2.13 (m, 2H), 2.13 – 2.03 (m, 2H), 1.97 (dt, *J* = 13.3, 4.3 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.51, 148.14, 128.56, 126.41, 125.26, 52.99, 44.40, 32.94, 28.43, 28.09, 27.70.

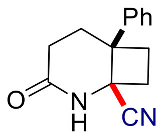
HRMS (ESI) *m/z* Calcd for $C_{13}H_{16}NO^{+}[M+H]^{+}$: 202.1226, found: 202.1226.

5.7 Access to 3-oxo-6-phenyl-2-azabicyclo[4.2.0]octane-1-carbonitrile



Supplementary Figure 12. Derivatization of **6a**

To an oven-dried screwed 4 mL glass vial equipped with a rod-like-shaped Teflon stir bar were added **6a** (46.3 mg, 0.2 mmol, 1.0 equiv.), TMSCN (0.5 mmol, 2.5 equiv.), DCM (1 mL, 0.2 M), and TMSOTf (0.3 mmol, 1.5 equiv.) under air atmosphere. The vial was then sealed. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 50 °C overnight. Upon completion of the reaction, the mixture was diluted with DCM and quenched with sat. aq. $NaHCO_3$. The organic layer was separated, and the aqueous layer was extracted three more times with DCM. The combined organic layers were then dried with Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (2:1:0.2, v/v/v) to give the **8f**.



8f

White solid. (R_f = 0.30, PE/EA/MeOH = 2:1:0.2, v/v/v)

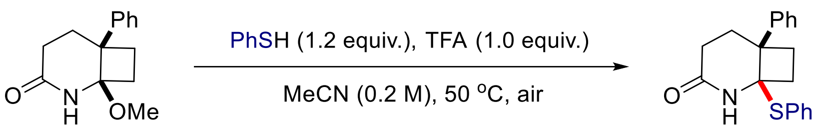
Compound **8f** is a previously unknown compound and was synthesized in 83% yield, dr >20:1 (38.6 mg, 0.2 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (s, 1H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.31 (t, *J* = 7.3 Hz, 1H), 7.24 – 7.12 (m, 2H), 2.84 – 2.69 (m, 1H), 2.64 – 2.47 (m, 3H), 2.46 – 2.24 (m, 3H), 2.12 – 1.99 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.88, 143.37, 128.79, 127.60, 125.50, 119.63, 55.81, 48.03, 34.31, 33.79, 27.30, 27.12.

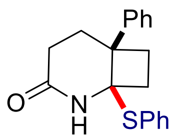
HRMS (ESI) *m/z* Calcd for $C_{14}H_{15}N_2O^+$[M+H]$^+$: 227.1179, found: 227.1188.

5.8 Access to 6-phenyl-1-(phenylthio)-2-azabicyclo[4.2.0]octan-3-one



Supplementary Figure 13. Derivatization of **6a**

To an oven-dried screwed 4 mL glass vial equipped with a rod-like-shaped Teflon stir bar were added **6a** (69.4 mg, 0.3 mmol, 1.0 equiv.), thiophenol (0.36 mmol, 1.2equiv.), CH_3CN (600 μL, 0.5 M), and TFA (0.3 mmol, 1.5 equiv.) under air atmosphere. The vial was then sealed. The reaction mixture was slowly stirred (200 rpm) in a metal heating plate at 25 °C overnight. Upon completion of the reaction, the mixture was diluted with DCM and quenched with sat. aq. $NaHCO_3$. The organic layer was separated, and the aqueous layer was extracted three more times with DCM. The combined organic layers were then dried with $NaSO_4$, filtered, and concentrated under reduced pressure. The resulting residue was purified by silica gel flash chromatography using PE/EA/MeOH eluent (2:1:0.2, v/v/v) to give the **8g**.



8g

White solid. (R_f = 0.50, PE/EA/MeOH = 2:1:0.2, v/v/v)

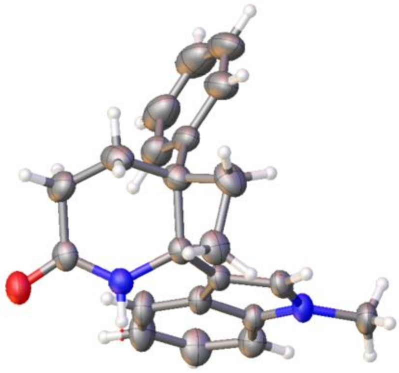
Compound **8g** is a previously unknown compound and was synthesized in 89% yield, dr >20:1 (87.8 mg, 0.3 mmol scale) following the general procedure.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (dd, *J* = 7.3, 2.0 Hz, 2H), 7.40 – 7.34 (m, 5H), 7.33 – 7.24 (m, 3H), 6.33 (s, 1H), 2.72 – 2.43 (m, 4H), 2.42 – 2.26 (m, 2H), 2.22 – 2.16 (m, 1H), 2.02 (dt, *J* = 13.3, 3.9 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.18, 143.34, 135.85, 131.19, 129.58, 129.32, 128.13, 127.12, 126.60, 69.26, 49.04, 34.81, 34.77, 27.89, 27.19.

HRMS (ESI) *m/z* Calcd for $C_{19}H_{20}NOS^+$[M+H]$^+$: 310.1260, found: 310.1265.

6. X-ray crystallographic data



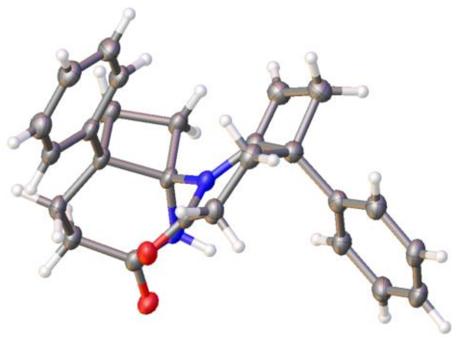
Supplementary Figure 14. X-ray structure of compound **3a**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **3a** in a mixture of DCM/heptane in 4 mL tube at room temperature. The X-ray data of **3a** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518236.

Supplementary Table 2. Crystal data and structure refinement for **3a**

Identification code	37-MR-20250417A_auto
Empirical formula	$C_{22}H_{22}N_2O$
Formula weight	330.41
Temperature/K	289(9)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	8.6158(3)
b/Å	17.4664(6)
c/Å	23.9834(9)
α/°	90
β/°	92.064(3)
γ/°	90
Volume/Å^3	3606.8(2)
Z	8
ρ_{calc} g/cm^3	1.217
μ/mm^{-1}	0.075
F(000)	1408.0
Crystal size/mm^3	0.23 × 0.2 × 0.14
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.964 to 50.698
Index ranges	$-10 \leq h \leq 9$, $-21 \leq k \leq 21$, $-28 \leq l \leq 28$
Reflections collected	26972
Independent reflections	6595 [R_{int} = 0.0302, R_{sigma} = 0.0254]

Data/restraints/parameters	6595/0/453
Goodness-of-fit on F^2	1.034
Final R indexes [I>=2σ (I)]	R_1 = 0.0697, wR_2 = 0.1895
Final R indexes [all data]	R_1 = 0.0933, wR_2 = 0.2051
Largest diff. peak/hole / e Å^{-3}	0.73/-0.19



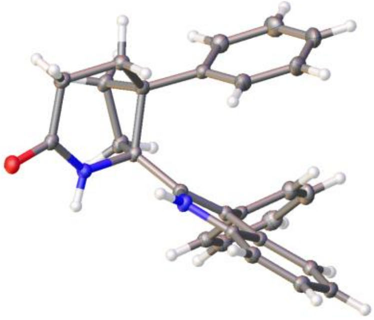
Supplementary Figure 15. X-ray structure of compound **4a**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **4a** in a mixture of $CHCl_3$/heptane in 4 mL tube at room temperature. The X-ray data of **4a** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518237.

Supplementary Table 3. Crystal data and structure refinement for **4a**.

Identification code	38-CR-20250904C_auto
Empirical formula	$C_{26.5}H_{26.5}Cl_{1.5}N_2O_2$
Formula weight	458.17
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	C2/c
a/Å	31.6097(7)
b/Å	9.6001(2)
c/Å	30.3578(6)
α/°	90
β/°	107.260(2)
γ/°	90
Volume/Å^3	8797.4(3)
Z	16
ρ_{calc} g/cm^3	1.384
μ/mm^{-1}	2.312
F(000)	3856.0
Crystal size/mm^3	0.2 × 0.18 × 0.14
Radiation	Cu Kα (λ = 1.54184)

2Θ range for data collection/°	5.856 to 152.746
Index ranges	-39 ≤ h ≤ 39, -11 ≤ k ≤ 8, -38 ≤ l ≤ 35
Reflections collected	34155
Independent reflections	8925 [R_{int} = 0.0494, R_{sigma} = 0.0453]
Data/restraints/parameters	8925/1/581
Goodness-of-fit on F^2	1.077
Final R indexes [I>=2σ (I)]	R_1 = 0.0591, wR_2 = 0.1708
Final R indexes [all data]	R_1 = 0.0666, wR_2 = 0.1775
Largest diff. peak/hole / e Å^{-3}	0.47/-1.03



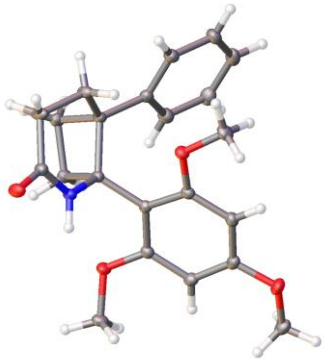
Supplementary Figure 16. X-ray structure of compound **5c**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **5c** in a mixture of $CHCl_3$/heptane in an NMR tube at room temperature. The X-ray data of **5c** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518231.

Supplementary Table 4. Crystal data and structure refinement for **5c**.

Identification code	38-CR-20250908F_auto
Empirical formula	$C_{28}H_{25}Cl_3N_2O$
Formula weight	511.85
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.5198(2)
b/Å	9.5021(2)
c/Å	16.2326(4)
α/°	96.133(2)
β/°	93.496(2)
γ/°	107.290(2)
Volume/Å^3	1241.60(5)
Z	2
ρ_{calc} g/cm^3	1.369

μ/mm^{-1}	3.528
F(000)	532.0
Crystal size/mm^3	0.18 × 0.13 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.834 to 149.038
Index ranges	-10 ≤ h ≤ 10, -7 ≤ k ≤ 11, -20 ≤ l ≤ 20
Reflections collected	20378
Independent reflections	4984 [R_{int} = 0.0292, R_{sigma} = 0.0190]
Data/restraints/parameters	4984/30/331
Goodness-of-fit on F^2	1.045
Final R indexes [I>=2σ (I)]	R_1 = 0.0343, wR_2 = 0.0912
Final R indexes [all data]	R_1 = 0.0352, wR_2 = 0.0919
Largest diff. peak/hole / e Å^{-3}	0.32/-0.43



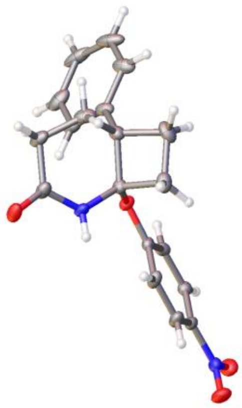
Supplementary Figure 17. X-ray structure of compound **5q**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **5q** in a mixture of DCM/heptane in an NMR tube at room temperature. The X-ray data of **5q** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518209.

Supplementary Table 5. Crystal data and structure refinement for **5q**.

Identification code	37-MR-20251210A_auto
Empirical formula	$C_{22}H_{25}NO_4$
Formula weight	367.43
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.39519(16)
b/Å	10.29504(19)
c/Å	11.8860(2)
α/°	109.1083(16)

β/°	104.1951(16)
γ/°	99.0491(15)
Volume/Å^3	908.86(3)
Z	2
ρ_{calc} g/cm^3	1.343
μ/mm^{-1}	0.092
F(000)	392.0
Crystal size/mm^3	0.34 × 0.24 × 0.22
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	5.182 to 59.148
Index ranges	$-11 \le h \le 11$, $-14 \le k \le 14$, $-16 \le l \le 16$
Reflections collected	22556
Independent reflections	5017 [R_{int} = 0.0323, R_{sigma} = 0.0275]
Data/restraints/parameters	5017/0/251
Goodness-of-fit on F^2	1.064
Final R indexes [I>=2σ (I)]	R_1 = 0.0395, wR_2 = 0.1051
Final R indexes [all data]	R_1 = 0.0465, wR_2 = 0.1104
Largest diff. peak/hole / e Å^{-3}	0.40/-0.23



Supplementary Figure 18. X-ray structure of compound **6f**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **6f** in a mixture of DCM/heptane in 4 mL tube at room temperature. The X-ray data of **6f** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518213.

Supplementary Table 6. Crystal data and structure refinement for **6f**.

Identification code	37-MR-20251208A
Empirical formula	$C_{19}H_{18}N_2O_4$
Formula weight	338.35
Temperature/K	100.00(10)

Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	7.2460(3)
b/Å	12.2378(4)
c/Å	18.5684(8)
α/°	90
β/°	100.138(4)
γ/°	90
Volume/Å^3	1620.85(11)
Z	4
ρ_{calc} g/cm^3	1.387
μ/mm^{-1}	0.098
F(000)	712.0
Crystal size/mm^3	0.23 × 0.2 × 0.13
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.456 to 52.744
Index ranges	$-9 \leq h \leq 9$, $-15 \leq k \leq 15$, $-23 \leq l \leq 20$
Reflections collected	12649
Independent reflections	3313 [R_{int} = 0.0333, R_{sigma} = 0.0286]
Data/restraints/parameters	3313/0/230
Goodness-of-fit on F^2	1.296
Final R indexes [I>=2σ (I)]	R_1 = 0.0852, wR_2 = 0.1930
Final R indexes [all data]	R_1 = 0.0892, wR_2 = 0.1946
Largest diff. peak/hole / e Å^{-3}	0.35/-0.36

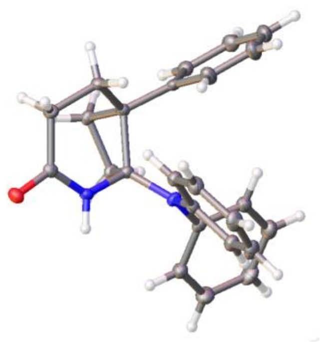
Supplementary Figure 19. X-ray structure of compound **7a**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **7a** in a mixture of $CDCl_3$/heptane in an NMR tube at room temperature. The X-ray data of **7a** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518207.

Supplementary Table 7. Crystal data and structure refinement for **7a**.

Identification code	38-CR-20251210A_auto

Empirical formula	$C_{22.5}H_{22}Cl_{1.5}D_{0.5}N_2O$
Formula weight	390.60
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1
a/Å	8.2063(3)
b/Å	10.2047(3)
c/Å	12.6923(4)
α/°	78.524(3)
β/°	77.279(3)
γ/°	69.660(3)
Volume/Å^3	963.41(6)
Z	2
ρ_{calc} g/cm^3	1.346
μ/mm^{-1}	2.501
F(000)	410.0
Crystal size/mm^3	0.22 × 0.2 × 0.14
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.204 to 150.472
Index ranges	$-10 \leq h \leq 10$, $-12 \leq k \leq 12$, $-14 \leq l \leq 15$
Reflections collected	14939
Independent reflections	3868 [R_{int} = 0.0259, R_{sigma} = 0.0230]
Data/restraints/parameters	3868/0/263
Goodness-of-fit on F^2	1.066
Final R indexes [I>=2σ (I)]	R_1 = 0.0397, wR_2 = 0.1051
Final R indexes [all data]	R_1 = 0.0425, wR_2 = 0.1071
Largest diff. peak/hole / e Å^{-3}	0.29/-0.35



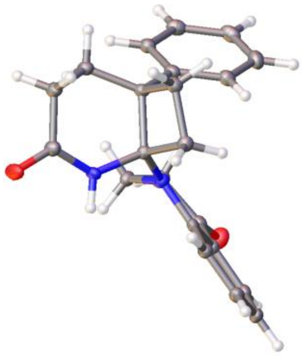
Supplementary Figure 20. X-ray structure of compound **7b**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **7b** in a mixture of $CDCl_3$/heptane in an NMR tube at room temperature. The

X-ray data of **7b** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518233.

Supplementary Table 8. Crystal data and structure refinement for **7b**.

Identification code	37-MR-20251210B_auto
Empirical formula	$C_{55}H_{48}Cl_{15}D_5N_4O_2$
Formula weight	1338.79
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	14.5636(2)
b/Å	19.4453(3)
c/Å	21.1345(2)
α/°	90
β/°	91.2433(11)
γ/°	90
Volume/Å^3	5983.74(13)
Z	4
ρ_{calc} g/cm^3	1.486
μ/mm^{-1}	0.734
F(000)	2728.0
Crystal size/mm^3	0.2 × 0.14 × 0.11
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	3.432 to 52.742
Index ranges	$-17 \leq h \leq 18$, $-24 \leq k \leq 24$, $-26 \leq l \leq 26$
Reflections collected	57297
Independent reflections	12220 [R_{int} = 0.0284, R_{sigma} = 0.0245]
Data/restraints/parameters	12220/307/760
Goodness-of-fit on F^2	1.034
Final R indexes [I>=2σ (I)]	R_1 = 0.0401, wR_2 = 0.1032
Final R indexes [all data]	R_1 = 0.0464, wR_2 = 0.1079
Largest diff. peak/hole / e Å^{-3}	0.83/-0.98



Supplementary Figure 21. X-ray structure of compound **7c**

Single crystals for X-ray studies were grown by slow evaporation of a solution of compound **7c** in a mixture of $CDCl_3$/heptane in an NMR tube at room temperature. The X-ray data of **7c** is deposited in the Cambridge Crystallographic Data Centre with a number of CCDC 2518223.

Supplementary Table 9. Crystal data and structure refinement for **7c**.

Identification code	38-CR-20251205B_auto
Empirical formula	$C_{44}H_{44}N_4O_4$
Formula weight	692.83
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	11.0004(2)
b/Å	11.4646(2)
c/Å	13.3602(2)
α/°	90
β/°	92.6991(16)
γ/°	90
Volume/Å^3	1683.06(6)
Z	2
ρ_{calc} g/cm^3	1.367
μ/mm^{-1}	0.700
F(000)	736.0
Crystal size/mm^3	0.2 × 0.16 × 0.11
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/ °	6.624 to 151.292
Index ranges	$-13 \le h \le 13$, $-14 \le k \le 14$, $-16 \le l \le 12$
Reflections collected	28172
Independent reflections	6557 [R_{int} = 0.0463, R_{sigma} = 0.0334]

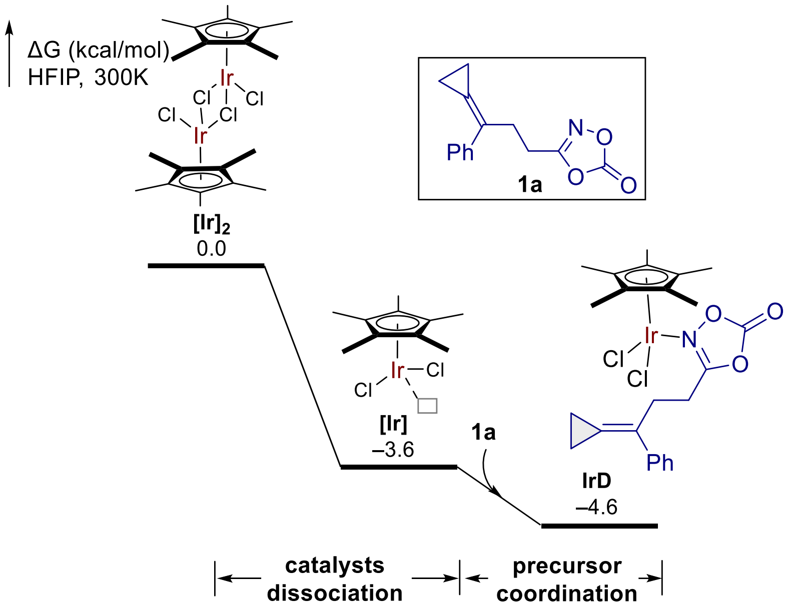
Data/restraints/parameters	6557/3/477
Goodness-of-fit on F^2	1.044
Final R indexes [I>=2σ (I)]	R_1 = 0.0322, wR_2 = 0.0853
Final R indexes [all data]	R_1 = 0.0331, wR_2 = 0.0860
Largest diff. peak/hole / e $Å^{-3}$	0.18/-0.20
Flack parameter	0.06(10)

7. Computational Details

All DFT calculations were carried out with Gaussian16-RevC.01 quantum chemical package.[4] Geometry optimizations were performed with B3LYP[5] functional with Grimme's D3BJ[6] dispersion correction. The iridium atom was represented using the Los Alamos LANL2DZ[7] basis set, which includes relativistic effective core potentials, and 6-31g*[8] for other atoms. For those structures having various conformations, the most stable conformer was searched and utilized using CREST[9]. Vibrational frequency calculations were carried out at the same level of theory as the geometry optimizations (No imaginary frequency for local minima and only one imaginary for transition states) and to provide the thermal corrections for Gibbs free energy determinations. Intrinsic reaction coordinate (IRC) calculations were done to confirm that the transition states proposed connected the appropriate reactants and products. The single-point calculations of the optimized geometries were performed with M06[10] functional with Grimme's D3(0)[5] dispersion correction and triple-zeta quality Def2-TZVPP[11] basis set for all atoms. We obtained solvation energies using the optimized gas phase structures with SMD[12] (HFIP[13]) model. Solvation calculations were carried out with the same level of single-point calculations employing HFIP as the model solvent. The raw energy data were processed and corrected employing the GoodVibes Python package (version 3.2)[14] to incorporate quasi-harmonic approximations for low-frequency vibrational modes and standard state corrections. Specifically, Gibbs free energies were adjusted to the standard state of 1.0 mol/L at 300.00 K and 1 atm. All reported energies are in kcal/mol unless otherwise specified. Electronic wavefunction analysis is performed by Multiwfn[15] software. Graphical structures are visualized with VMD[16], CYLview[17] and GaussView.

Note: as HFIP is not by default parametrized within the SMD model implemented in Gaussian, the following parameters were used following a report by Paton and coworkers[22]: eps = 16.7; epsinf = 1.625626; H Bond Acidity = 1.96; h bond basicity = 0.00; Surface Tension At Interface = 23.23; ElectronegativeHalogenicity = 0.6; CarbonAromaticity = 0.00.

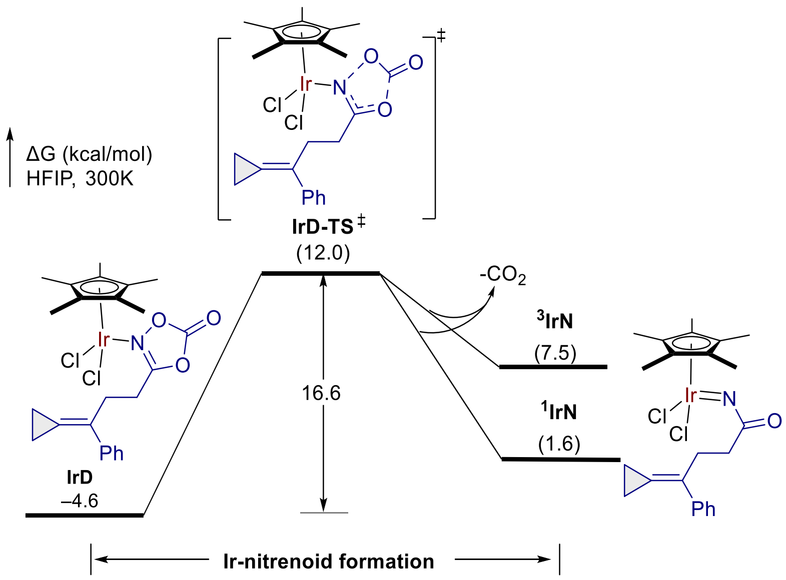
7.1 Dissociation Iridium Dimer and Dioxazolone Coordination



Supplementary Figure 22. Energy diagram of the reaction pathway (energies are relative to [Cp*IrCl₂]₂, in kcal/mol).

Computational studies reveal that the dissociation of [Cp*IrCl₂]₂ in solution is a thermally favorable process (-3.6 kcal/mol), leading to the formation of an **[Ir]** intermediate. This species subsequently coordinates with dioxazolone **1a** to yield a more stable **IrD** intermediate with a lower relative energy of -4.6 kcal/mol.

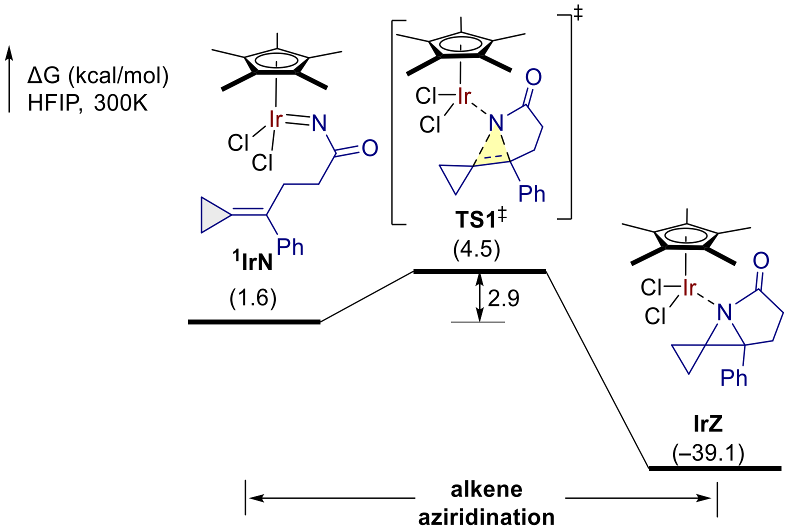
7.2 Energy diagram of Ir-nitrenoid formation



Supplementary Figure 23. Energy diagram of Ir-nitrenoid formation (energies are relative to [Cp*IrCl₂]₂, in kcal/mol).

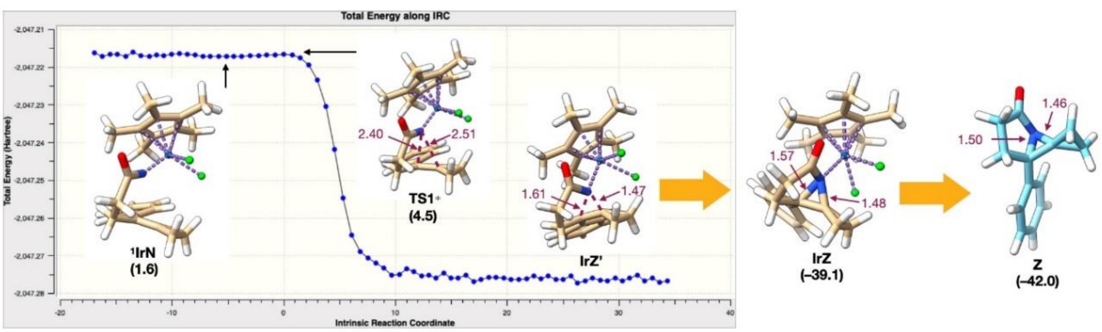
The **IrD** complex subsequently undergoes decarboxylation via transition state **IrD-TS‡** to generate the singlet Ir–nitrenoid species (**¹IrN**, 1.6 kcal/mol). This step entails an activation barrier of 16.6 kcal/mol, which is kinetically accessible at room temperature. Furthermore, the corresponding triplet species (**³IrN**, 7.5 kcal/mol) was found to lie significantly higher in energy.

7.3 Alkene aziridination



Supplementary Figure 24. Energy diagram of alkene aziridination (energies are relative to $[Cp^*IrCl_2]_2$, in kcal/mol).

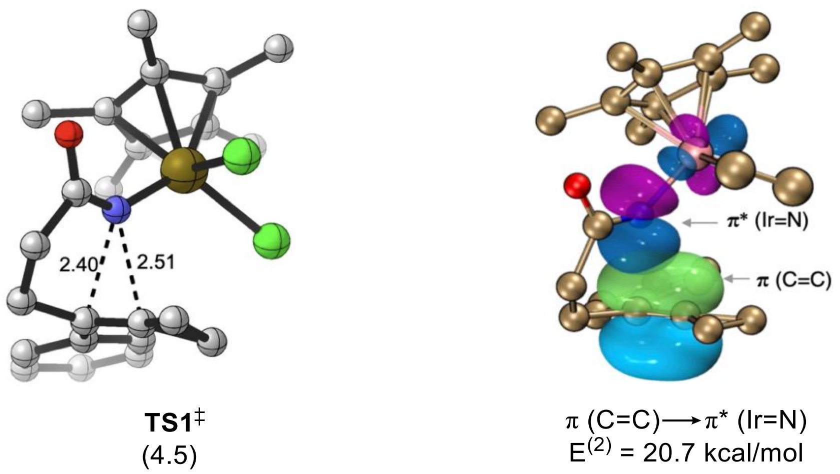
The singlet Ir–nitrenoid species (**¹IrN**) then undergoes an aziridination pathway to yield the aziridine complex **IrZ**. This process is characterized by a remarkably low activation barrier of only 2.9 kcal/mol. Based on geometric parameters and Intrinsic Reaction Coordinate (IRC) analysis, this transformation is identified as a *concerted asynchronous process*.



Supplementary Figure 25. IRC analysis of alkene aziridination

We conducted detailed DFT calculations to investigate the aziridination of substrate

¹IrN. Intrinsic Reaction Coordinate (IRC) analysis indicates that the process proceeds via a *concerted asynchronous mechanism.* Specifically, the carbon atom at the MCPs terminus preferentially attacks the Ir-nitrenoid nitrogen to initiate C–N bond formation, followed by the formation of a second C–N bond with the benzylic carbon. In the resulting intermediate **IrZ**, the two C–N bonds measure 1.57 Å and 1.48 Å, respectively—notably longer than those in the free aziridine **Z** (1.50 Å and 1.46 Å). This bond elongation in **IrZ** suggests a lower activation barrier for bond cleavage, thereby facilitating the subsequent ring-expansion and rearrangement processes. This mechanism is further supported by the geometric analysis of **TS1‡**, which exhibits two distinct C–N distances of 2.40 Å and 2.51 Å.

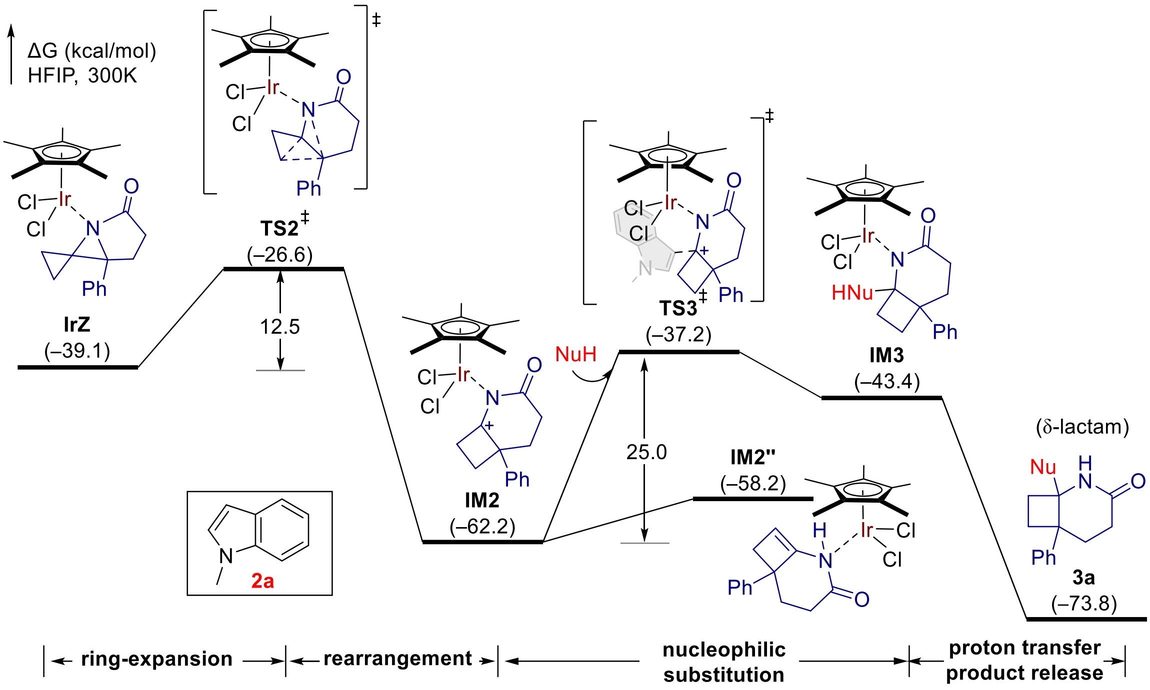


Supplementary Figure 26. Geometric and NBO analysis of alkene aziridination **TS1‡**

Furthermore, Natural Bond Orbital (NBO) analysis[18] reveals a significant hyperconjugative interaction between the π orbital of the alkene and the π* antibonding orbital of the Ir=N moiety ($E^{(2)}$ =20.7 kcal/mol), which facilitates the formation of the Ir-bound tricyclic aziridine intermediate **IrZ**.

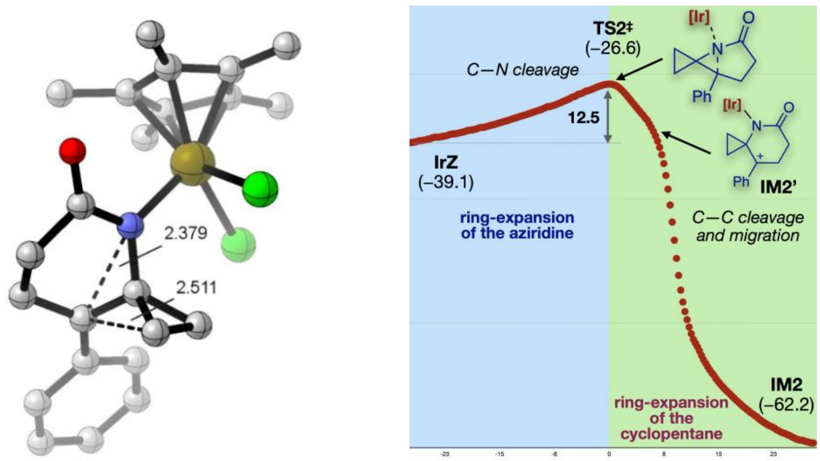
7.4 Comparison of the two possible pathways from IrZ to lactam 3a

7.4.1 From IrZ to lactam 3a: a favorable pathway



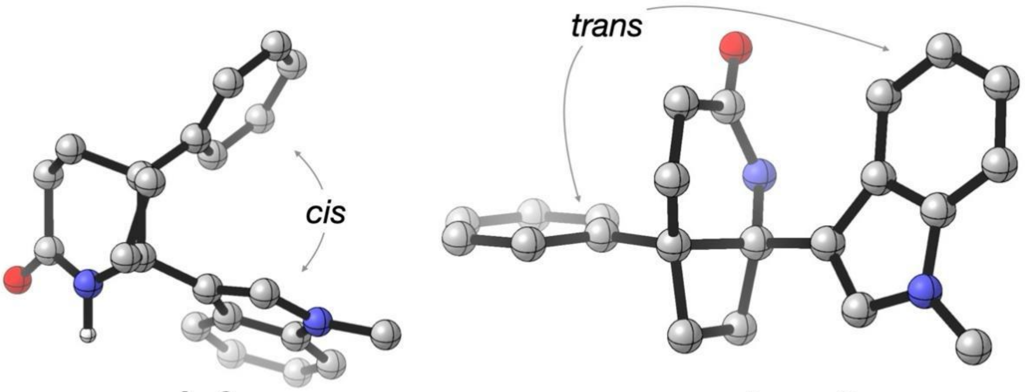
Supplementary Figure 27. Energy diagram of ring-expansion and rearrangement (energies are relative to $[Cp^*IrCl_2]_2$, in kcal/mol).

The resulting intermediate **IrZ** undergoes a more favorable direct ring-expansion rearrangement via transition state **TS2‡** to form the carbocation intermediate **IM2**. IRC analysis characterizes this ring-expansion and rearrangement as a *concerted asynchronous process*. Specifically, the process begins with the C–N bond cleavage of the aziridine to form a short-lived tertiary carbocation intermediate, **IM2'**. The energy of **IM2'** is closely comparable to that of **IrZ**, suggesting a dynamic equilibrium between these two species, mediated by an activation barrier of 12.5 kcal/mol. Subsequently, **IM2'** undergoes a rapid rearrangement to yield a thermodynamically more stable, nitrogen-stabilized carbocation **IM2** (-62.2 kcal/mol).Following this, **IM2** undergoes an S_N1-type nucleophilic substitution with *N*-methylindole **2a**, affording intermediate **IM3** via a transition state with a Gibbs free energy barrier of 25.0 kcal/mol. Notably, the nucleophilic attack is guided by multiple non-covalent interactions (NCIs) involving the chlorine atom on the catalyst (as supported by NCI analysis in Section 7.6). Finally, **IM3** undergoes proton transfer to yield the δ-lactam **3a**, while simultaneously regenerating the active [Ir] catalyst, thereby completing the catalytic cycle.



TS2‡ (-26.6 kcal/mol)

Supplementary Figure 28. TS and **IRC** analysis of ring-expansion and rearrangement (**TS2‡**)



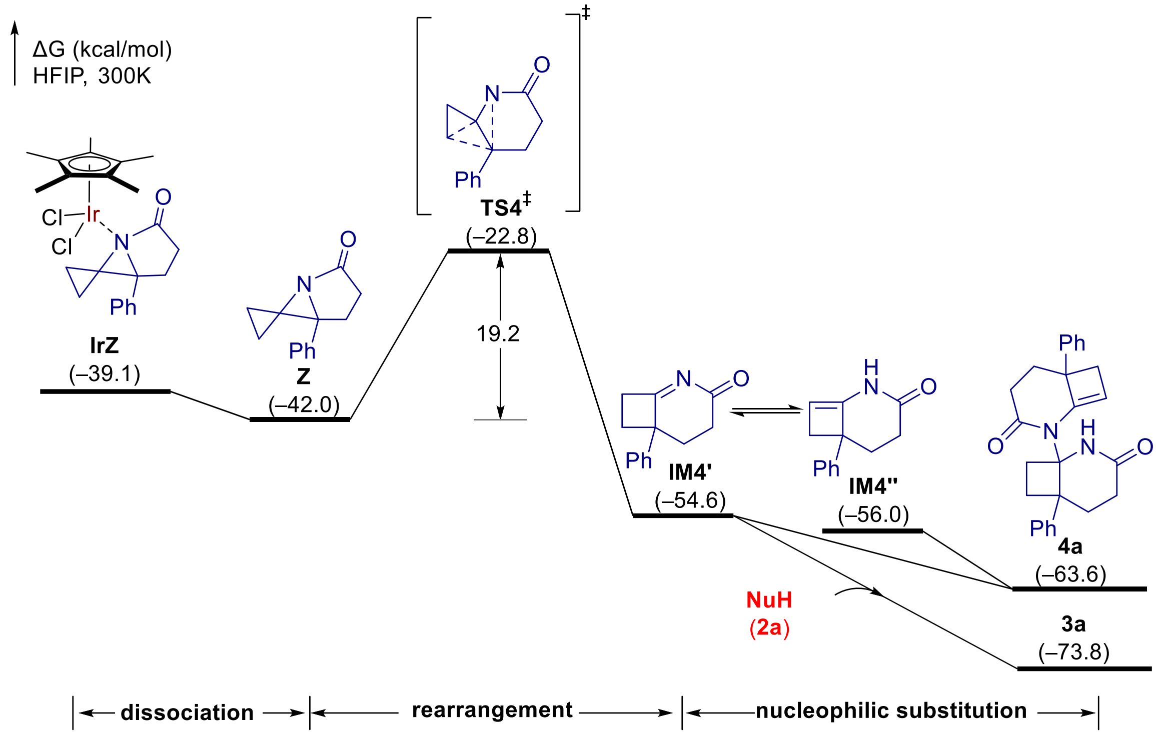
***cis*-3a**
(0.0)

***trans*-3a**
(24.1)

Supplementary Figure 29. Two distinct configurations of product **3a**

In our experiments, only the ***cis*-3a** isomer of product **3a** was observed, while the ***trans*-3a** isomer was not detected. Density functional theory (DFT) calculations indicate that the ***cis*-3a** configuration is more stable than the ***trans*-3a** configuration (0.0 kcal/mol *vs* 24.1 kcal/mol).

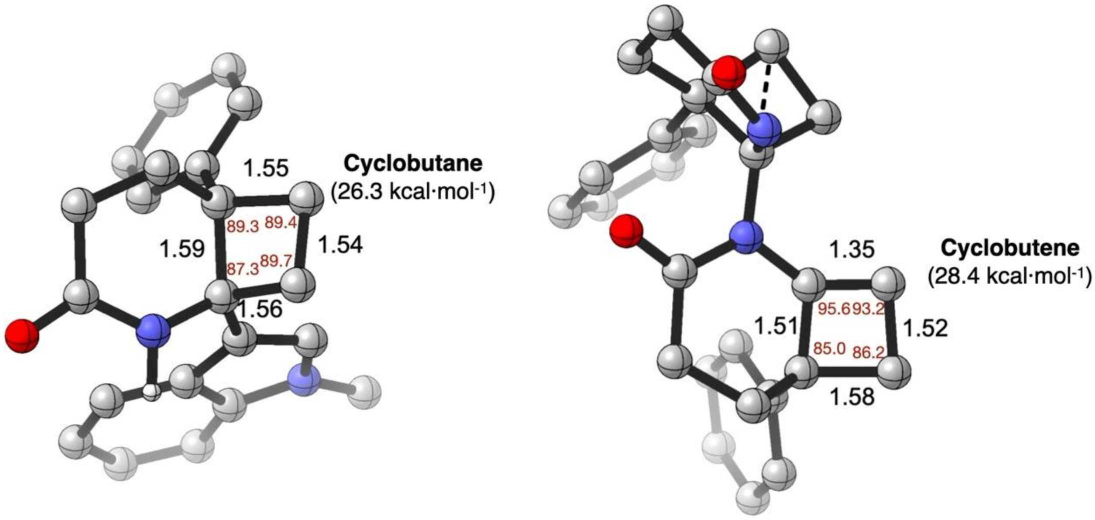
7.4.2 From IrZ to lactam 3a: a unfavorable pathway



Supplementary Figure 30. Energy diagram of catalyst dissociation and rearrangement (energies are relative to $[Cp^*IrCl_2]_2$, in kcal/mol).

The reaction pathway involves the dissociation of **IrZ** to release the free aziridine **Z**, which subsequently undergoes a ring-expansion rearrangement via transition state **TS4‡** (-22.8 kcal/mol, $\Delta G^‡$ = 19.2 kcal/mol relative to **Z**) to afford intermediate **IM4'**. In contrast, the iridium-coordinated ring-expansion/rearrangement via **TS2‡** is significantly more favorable, with a relative energy of -26.6 kcal/mol. Therefore, we conclude that the Ir-assisted pathway via **TS2‡** is the energetically preferred route. While the pathway involving **TS4‡** is considerably less favorable (unfavorable), its contribution to the overall reaction cannot be entirely ruled out. Nucleophilic capture of **IM4'** yields the δ-lactam **3a**, while an alternative rearrangement leads to the thermodynamically more stable species **IM4''**. Both **IM4'** and **IM4''** can undergo homocoupling, forming the dimeric product **4a**, consistent with experimental observations.

7.5 Structural analysis of 3a and 4a

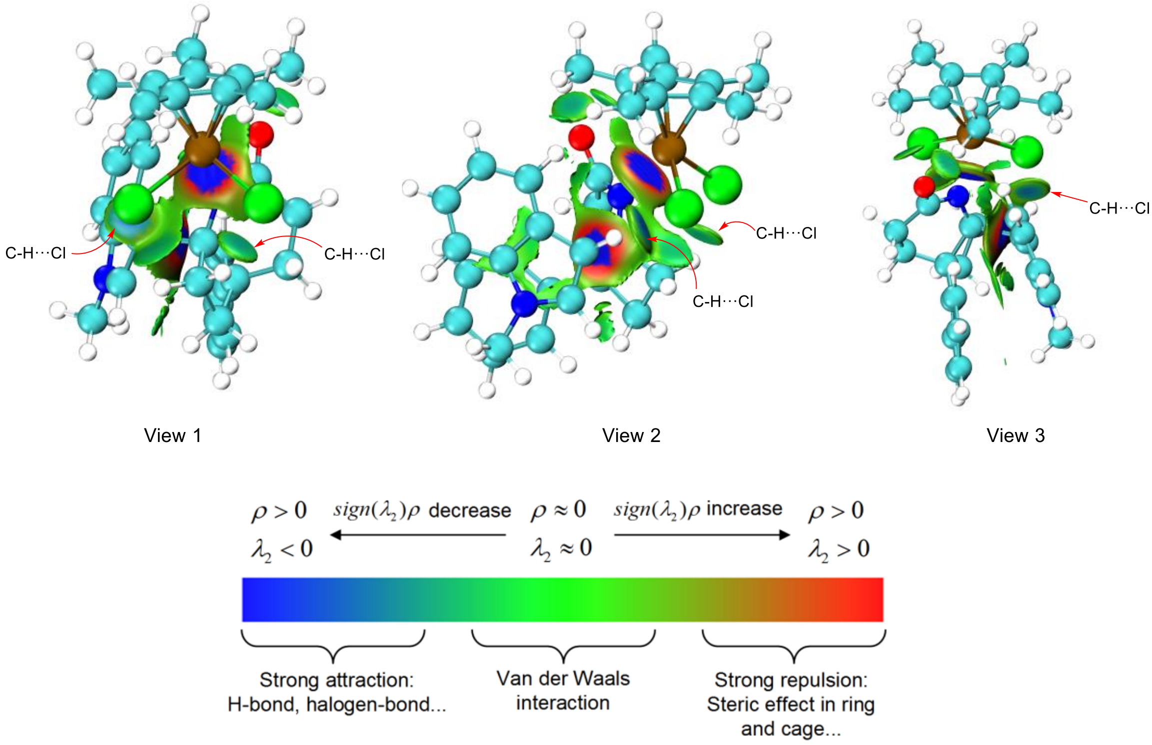


3a **4a**

Supplementary Figure 31. Structural Analysis of **3a** and **4a**

DFT-optimized structures were visualized using CYLview. For cyclobutane-fused δ-lactam **3a**, the four internal angles of the cyclobutane ring all fall within 88-90°, and the bond lengths range from 1.54 to 1.59 Å. Similarly, in the dimeric cyclobutene-fused δ-lactam **4a**, the cyclobutene fragment is markedly distorted, with the maximum bond angle reaching 95.6°, and the C1–C4 bond length significantly shortened to only 1.35 Å.

7.6 Non-covalent interaction (NCI) analysis



Supplementary Figure 32. Non-covalent interaction (NCI) analysis

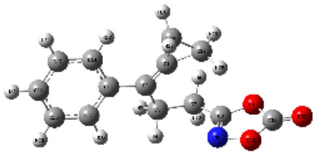
To investigate the underlying mechanism for the nucleophilic attack, we employed Multiwfn[15] to analyze weak interactions using Independent Gradient Model (IGM), coupled with graphical visualization using VMD[16]. This approach enable further investigation into the transition state of **IM3**, and provides a clearer depiction of the transition state from three distinct perspectives. Analysis indicates that characteristic C-H···Cl interactions are present in the transition state of **IM3**. The two chlorine atoms play two critical roles: one stabilizes the carbocation conformation, while the other directs the approach of the nucleophile.

7.7 Energy summary table

Structure	E_SPC	E	ZPE	H_SPC	T.S	T.qh-S	G(T)_SPC	qh-G(T)_SPC	Im.freq	ΔG
1a	-821.396322	-821.109762	0.24386	-821.136042	0.058695	0.055576	-821.194737	-821.191618		
1IrN	-2047.695067	-2047.217577	0.452859	-2047.209133	0.094774	0.08897	-2047.303907	-2047.298103		1.6
2a	-402.96536	-402.879689	0.157684	-402.798763	0.038756	0.038539	-402.837519	-402.837302		
3IrN	-2047.684598	-2047.214242	0.452621	-2047.198829	0.095571	0.089937	-2047.2944	-2047.288766		7.5
3a	-1035.950056	-1035.694034	0.394271	-1035.534341	0.067639	0.064488	-1035.601981	-1035.598829		-73.8
4a	-1265.911873	-1265.572339	0.468009	-1265.418643	0.07555	0.071659	-1265.494194	-1265.490302		-63.6
CO_2	-188.541722	-188.44468	0.011776	-188.526342	0.021414	0.021415	-188.547756	-188.547756		
IM2	-2047.802118	-2047.324615	0.456865	-2047.313472	0.09154	0.086364	-2047.405012	-2047.399836		-62.2
IM2"	-2047.79561	-2047.324123	0.45724	-2047.306562	0.092571	0.086874	-2047.399132	-2047.393436		-58.2
IM3	-2450.765785	-2450.202273	0.618674	-2450.107746	0.104896	0.099502	-2450.212641	-2450.207248		-43.4
IM4’	-632.926801	-632.750047	0.230837	-632.682859	0.049796	0.048033	-632.732654	-632.730892		-54.6
IM4”	-632.930252	-632.761474	0.231611	-632.685685	0.048784	0.04742	-632.734469	-632.733106		-56.0
Ir2	-2829.702993	-2829.104482	0.449674	-2829.216059	0.103292	0.097659	-2829.319351	-2829.313718		0.0
IrD	-2236.268688	-2235.68324	0.470042	-2235.763705	0.097345	0.092062	-2235.86105	-2235.855767		-4.6
IrD-TS	-2236.238689	-2235.65275	0.467056	-2235.736365	0.098735	0.093004	-2235.8351	-2235.829369	-183.11	12.0
Ir	-1414.844396	-1414.522032	0.223724	-1414.602027	0.063494	0.060647	-1414.665521	-1414.662673		-3.6
IrZ	-2047.765889	-2047.279034	0.456145	-2047.278209	0.088579	0.084909	-2047.366788	-2047.363118		-39.1
***trans*-3**	-1035.911975	-1035.655952	0.394218	-1035.496604	0.06636	0.063809	-1035.562964	-1035.560412		-49.7
TS1	-2047.692266	-2047.216705	0.452886	-2047.207328	0.090167	0.086153	-2047.297495	-2047.293481	-95.24	4.5
TS1_pdt	-2047.765855	-2047.279034	0.456147	-2047.278174	0.088564	0.084901	-2047.366739	-2047.363075		-39.1
TS2_IM2	-2047.790799	-2047.320089	0.457628	-2047.301572	0.090699	0.085724	-2047.392271	-2047.387296		-54.3
TS2	-2047.744505	-2047.258952	0.454545	-2047.258852	0.087659	0.084206	-2047.346511	-2047.343057	-106.98	-26.6
TS3-SM	-2450.783138	-2450.228014	0.616343	-2450.125146	0.114281	0.105629	-2450.239427	-2450.230775		-58.2
TS3	-2450.755627	-2450.1979	0.618388	-2450.098058	0.104286	0.099238	-2450.202344	-2450.197295	-251.5	-37.2
TS4	-632.872063	-632.666212	0.226522	-632.632469	0.04862	0.047652	-632.681089	-632.680121	-334.16	-22.8
Z	-632.904559	-632.714184	0.229057	-632.662026	0.050397	0.04876	-632.712423	-632.710786		-42.0

Supplementary Table 10. Energy summary table. Hcorr = Thermal correction to Enthalpy; Gcorr =Thermal correction to Gibbs Free Energy Gibbs (total) = E(M06) + Gcorr; Enthalpy (total) = E(M06) + Hcor (ΔG in kcal/mol at 300K)

7.8 Cartesian coordinates of the optimized geometries (XYZ formation)



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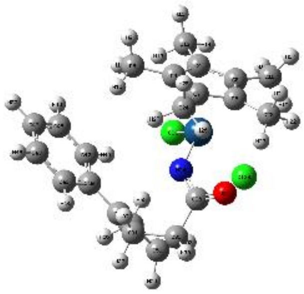
1a

C	0.299600	1.230114	0.285876
C	0.918153	0.052028	0.418765
C	0.191952	-1.196056	0.886734
H	0.149621	-1.926487	0.061706
H	0.795191	-1.671898	1.676543
N	-2.150261	-1.021791	-0.867851
C	-1.220871	-0.992698	1.431934
C	-2.243101	-0.747981	0.382243
H	-1.262943	-0.157088	2.145604
H	-1.536668	-1.891699	1.989348
O	-3.432653	-0.200444	0.733897
C	2.363180	-0.104753	0.098647
C	2.916384	-1.374847	-0.156090
C	3.229717	1.005689	0.042533
C	4.268957	-1.526674	-0.469519
H	2.286307	-2.264102	-0.128896
C	4.580176	0.853937	-0.264723
H	2.840301	1.999561	0.257187
C	5.109427	-0.414097	-0.526139
H	4.665661	-2.524659	-0.671391
H	5.227593	1.733837	-0.295245
H	6.168252	-0.532373	-0.767697
C	-0.938716	2.023811	0.315634
C	0.392470	2.632211	-0.129709
H	-1.691059	1.847268	-0.461079
H	-1.359442	2.347344	1.275019
H	0.531484	2.855699	-1.194265
H	0.867502	3.356970	0.542336
O	-3.387953	-0.613080	-1.413682
O	-5.276067	0.315775	-0.528751
C	-4.176761	-0.113658	-0.430506



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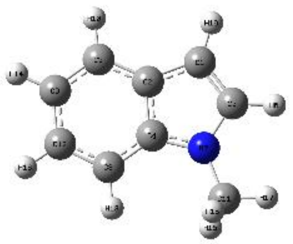
1IrN

Cl	0.356099	0.376294	-2.432959
Ir	0.943971	-0.070659	-0.169904
C	1.156040	2.054618	0.704161
C	2.159044	1.921945	-0.340899
C	3.006662	0.820425	-0.033755
C	2.551089	0.257362	1.239193
C	1.447887	1.079278	1.706654
C	0.091704	3.099382	0.762692
H	0.463720	4.007294	1.268798
H	-0.792197	2.740181	1.305773
H	-0.237802	3.382139	-0.244969
C	2.249673	2.821426	-1.524662
H	1.266147	2.949845	-1.996090
H	2.934049	2.426174	-2.284500
H	2.614622	3.810990	-1.200715
C	4.164795	0.304340	-0.823366
H	5.115990	0.532383	-0.314840
H	4.191726	0.748066	-1.826643
H	4.084850	-0.786107	-0.943304
C	3.222790	-0.838246	1.997952
H	3.868112	-0.410824	2.784331
H	3.842238	-1.448408	1.329197
H	2.470907	-1.498726	2.451734
C	0.711015	0.901321	2.990063
H	1.097293	1.615602	3.736769
H	0.830661	-0.119356	3.372703
H	-0.361972	1.091515	2.853397
Cl	1.572232	-2.247661	-0.935235
C	-2.664877	-1.653539	-1.171013
C	-2.952206	-1.081822	0.007199
C	-2.962208	-1.931989	1.268807
H	-3.009668	-1.283632	2.155303
H	-3.874526	-2.553547	1.290490

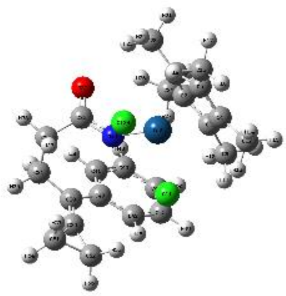
N	-0.347486	-0.909405	0.856206
C	-1.739165	-2.842084	1.404052
C	-0.436043	-2.077028	1.540556
H	-1.642030	-3.502637	0.529418
H	-1.809639	-3.482575	2.295597
O	0.443561	-2.410504	2.337209
C	-3.300270	0.356512	0.099446
C	-4.110826	0.853808	1.137540
C	-2.805136	1.276770	-0.846157
C	-4.422330	2.213102	1.224475
H	-4.521019	0.170047	1.882801
C	-3.118921	2.632186	-0.759887
H	-2.137423	0.928314	-1.631988
C	-3.927922	3.111667	0.276028
H	-5.060851	2.569511	2.036885
H	-2.717738	3.323450	-1.505982
H	-4.170478	4.175054	0.343334
C	-2.293338	-2.877281	-1.887292
C	-2.604127	-1.574676	-2.632400
H	-1.239455	-3.180925	-1.849982
H	-3.009072	-3.706376	-1.946655
H	-1.764310	-1.058295	-3.102829
H	-3.555581	-1.500148	-3.173557



19

2a

C	0.730563	1.891840	0.001282
C	-0.387754	0.988371	0.000216
C	1.866933	1.118682	-0.001850
C	0.154591	-0.332306	-0.003972
C	-1.785924	1.147189	0.002281
H	2.914161	1.416705	-0.002442
N	1.531447	-0.224326	-0.007840
C	-0.658807	-1.473627	-0.003166
C	-2.595902	0.015707	0.002087
H	-2.227353	2.147055	0.004242
C	2.449614	-1.337312	0.006970
C	-2.038279	-1.281254	-0.000495
H	-0.231123	-2.478363	-0.004474
H	-3.682664	0.128667	0.004283
H	2.329020	-1.944314	0.920314
H	2.292891	-1.993124	-0.865693
H	3.480142	-0.959517	-0.025221
H	-2.701062	-2.149925	0.000810
H	0.695647	2.979356	0.002947

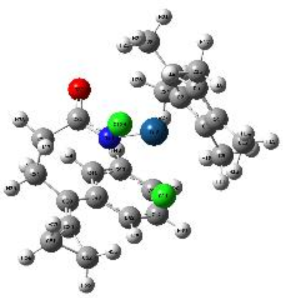


56

3IrN

Cl	0.015638	-0.752038	-2.258821
Ir	0.966426	-0.280677	-0.088574
C	1.763910	1.689495	-0.651753
C	2.807049	0.751914	-1.063127
C	3.258594	0.065165	0.089228
C	2.499042	0.543125	1.244684
C	1.637752	1.612828	0.785898
C	1.080745	2.643708	-1.573488
H	1.788008	3.416527	-1.919274
H	0.233887	3.135344	-1.080549
H	0.692108	2.107139	-2.450879
C	3.250645	0.571928	-2.476847
H	2.389987	0.307658	-3.110707
H	3.997297	-0.226908	-2.568437
H	3.689789	1.506905	-2.861590
C	4.291323	-1.007450	0.160038
H	5.080147	-0.724430	0.874865
H	4.752054	-1.190666	-0.818644
H	3.832402	-1.949252	0.501072
C	2.710116	0.110682	2.658993
H	3.496361	0.716382	3.141808
H	3.015394	-0.943860	2.695888
H	1.780629	0.211763	3.235550
C	0.780066	2.487744	1.640154
H	1.359465	3.371081	1.958305
H	0.429624	1.946327	2.527050

H	-0.105449	2.832294	1.090400
Cl	1.298255	-2.648367	0.235700
C	-3.502115	-1.511061	-0.836040
C	-3.407910	-0.658834	0.190767
C	-3.527952	-1.232099	1.590707
H	-4.004308	-0.516001	2.275725
H	-4.188631	-2.110077	1.554596
N	-0.620312	-0.405314	0.891435
C	-2.182446	-1.697847	2.196329
C	-1.094078	-0.646941	2.155327
H	-1.813793	-2.563093	1.622052
H	-2.314417	-1.996075	3.246956
O	-0.669837	-0.051078	3.138968
C	-3.131723	0.787312	0.002603
C	-3.175192	1.696019	1.078027
C	-2.786895	1.300489	-1.266181
C	-2.906253	3.054845	0.895536
H	-3.411814	1.347364	2.082965
C	-2.524435	2.654413	-1.449350
H	-2.691557	0.619727	-2.108315
C	-2.584775	3.546037	-0.370732
H	-2.950780	3.730870	1.753049
H	-2.259201	3.017987	-2.445280
H	-2.381218	4.609658	-0.517913
C	-3.663069	-2.914651	-1.226683
C	-3.456516	-1.804007	-2.266791
H	-2.828480	-3.603039	-1.046406
H	-4.654692	-3.378825	-1.157943
H	-2.484059	-1.747847	-2.768035
H	-4.314134	-1.518968	-2.888632



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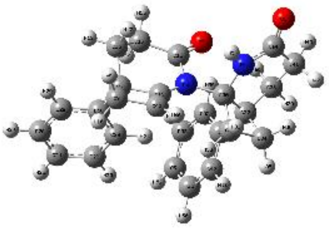
3

O	-1.560482	-3.641419	-1.010973
N	2.479526	0.847618	1.401881
N	-0.968970	-2.166641	0.595868
H	-0.255210	-2.842525	0.853805
C	2.752137	0.022299	0.330654
C	3.925481	-0.084881	-0.427570
H	4.800734	0.529577	-0.207580
C	3.934990	-1.000422	-1.475110
H	4.832683	-1.109063	-2.088154
C	2.800673	-1.790521	-1.754617
H	2.836147	-2.503102	-2.581783
C	1.636620	-1.687423	-0.998502
H	0.785157	-2.320965	-1.246710
C	1.589232	-0.765799	0.070967
C	0.592994	-0.366746	1.047548
C	1.190496	0.603077	1.826827
H	0.784769	1.166396	2.664040
C	-0.835527	-0.837072	1.166049
C	-1.423144	-0.644716	2.599710
H	-2.128266	-1.452704	2.836225
H	-0.691236	-0.563605	3.415117
C	-2.125893	0.645737	2.134464
H	-1.547409	1.548942	2.371662
H	-3.165179	0.800392	2.459373
C	-1.932259	0.201000	0.658399
C	-3.170759	-0.570999	0.155852
H	-3.634617	-1.104417	1.001742
H	-3.916863	0.148828	-0.214349
C	-2.802666	-1.582118	-0.921916
H	-3.666393	-2.183806	-1.239833
H	-2.430581	-1.077812	-1.828508
C	-1.734986	-2.562409	-0.467906
C	-1.523856	1.263753	-0.336205
C	-0.734542	0.972524	-1.459655
H	-0.348809	-0.033290	-1.611956
C	-0.405811	1.966419	-2.383828
H	0.221481	1.714283	-3.242154
C	-0.868026	3.273222	-2.209116
H	-0.609423	4.051224	-2.931396
C	-1.662906	3.575686	-1.100846
H	-2.033727	4.593054	-0.952563
C	-1.983479	2.579609	-0.175056
H	-2.604937	2.831900	0.687376
C	3.404481	1.793727	1.978333
H	4.307215	1.287854	2.359810
H	2.918119	2.314111	2.813837
H	3.716814	2.545556	1.234693



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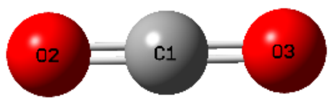
4

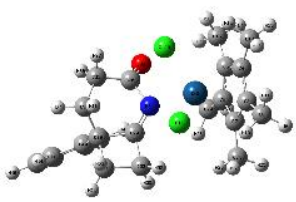
O	-4.650699	-2.193665	-0.636778
O	-1.311168	-0.568152	-2.095830
N	-0.464637	-0.882373	-0.005761
N	-2.762042	-1.403557	0.307828
H	-2.518379	-2.379946	0.454304
C	-1.047957	2.010647	0.136849
C	1.848467	-1.958867	0.194853
C	1.960619	-2.835957	1.506740
H	1.954907	-3.922300	1.313338
H	2.822484	-2.587099	2.145810
C	0.500706	-1.552503	0.741176
C	-0.404656	-0.984049	-1.399998
C	0.839627	-1.615214	-2.022403
H	1.511037	-0.773665	-2.267580
H	0.506196	-2.038395	-2.979787
C	1.591135	-2.635617	-1.156910
H	0.970724	-3.534502	-1.004148
H	2.519152	-2.951089	-1.657364
C	2.916351	-0.885600	0.097859
C	2.633857	0.479633	0.231064
H	1.610191	0.815543	0.398860
C	3.659809	1.427189	0.143513
H	3.416269	2.485212	0.249521
C	4.977160	1.025152	-0.079643
H	5.775710	1.767890	-0.147043
C	5.268920	-0.336351	-0.215871
H	6.297570	-0.663157	-0.387378
C	4.245765	-1.279832	-0.127680
H	4.481635	-2.343247	-0.225700
C	-1.721947	-0.443849	0.613795
C	-1.660234	-0.150019	2.134737
H	-1.983307	-0.972443	2.786372
H	-0.667084	0.197674	2.444048
C	-2.642005	1.012166	1.898544
H	-2.513031	1.926959	2.489540
H	-3.683366	0.684277	2.004852
C	-2.214163	1.052170	0.400592
C	-3.336532	1.233423	-0.625055
H	-3.748636	2.250077	-0.518126
H	-2.914706	1.151416	-1.631521
C	-4.447173	0.195627	-0.491340
H	-5.091397	0.406053	0.380216
H	-5.111198	0.205093	-1.366361
C	-3.971466	-1.234534	-0.321386
C	0.607425	-2.253851	1.889516
H	-0.049988	-2.426182	2.741118
C	-0.389475	2.697043	1.170810
H	-0.686187	2.550596	2.208330
C	0.652178	3.592190	0.909688
H	1.134662	4.111027	1.741478
C	1.070591	3.822767	-0.400403
H	1.879882	4.526324	-0.609214
C	0.441844	3.136430	-1.441488
H	0.761479	3.294445	-2.474274
C	-0.600751	2.247660	-1.176608
H	-1.061515	1.720375	-2.008586



3

CO_2

C	0.000000	0.000000	0.000000
O	0.000000	0.000000	1.258400
O	0.000000	0.000000	-1.258400



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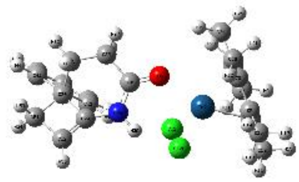
IM2

Cl	-1.174963	-2.538841	-0.279436
Ir	-1.394100	-0.119738	-0.088010

C	-3.235057	-0.215385	1.093411
C	-3.388599	0.759893	0.057468
C	-2.362575	1.785669	0.239188
C	-1.596984	1.426830	1.406426
C	-2.105438	0.172791	1.928812
C	-4.055781	-1.445224	1.306306
H	-4.640994	-1.355508	2.236538
H	-3.407184	-2.330441	1.376281
H	-4.750520	-1.611733	0.473411
C	-4.400259	0.769108	-1.041687
H	-4.951459	-0.178918	-1.087448
H	-3.904231	0.913754	-2.012473
H	-5.124411	1.586888	-0.889863
C	-2.204526	3.001342	-0.614356
H	-2.947113	3.772141	-0.344915
H	-2.338416	2.739610	-1.673306
H	-1.198299	3.426072	-0.512509
C	-0.453803	2.210891	1.967177
H	-0.824131	2.982969	2.662365
H	0.109655	2.701862	1.162977
H	0.237595	1.561935	2.522929
C	-1.661543	-0.546144	3.162178
H	-2.285107	-0.274938	4.032184
H	-0.617817	-0.306366	3.409981
H	-1.730184	-1.633736	3.019605
Cl	-1.277616	-0.013263	-2.532404
C	1.606857	-0.784219	0.250790
C	2.975214	-1.081173	-0.333524
C	2.788076	-1.056671	-1.864385
H	3.732999	-1.262197	-2.389351
H	2.069564	-1.845042	-2.135475
N	0.712852	-0.032861	-0.275679
C	2.222678	0.312028	-2.270438
C	1.252895	0.902623	-1.262350
H	1.649550	0.240628	-3.207971
H	3.010825	1.065708	-2.415244
O	0.981540	2.069507	-1.183658
C	4.122474	-0.211996	0.140970
C	5.440956	-0.639132	-0.082774
C	3.912828	1.014780	0.784771
C	6.522593	0.141882	0.323725
H	5.619832	-1.596309	-0.579962
C	4.996510	1.799491	1.193202
H	2.896980	1.371552	0.966177
C	6.303094	1.366576	0.964123
H	7.542294	-0.205709	0.141327
H	4.813751	2.754511	1.691298
H	7.149508	1.979379	1.282496
C	2.858264	-2.463325	0.415072
C	1.645379	-1.909879	1.233321
H	2.553078	-3.270585	-0.263473
H	3.740266	-2.762471	0.994260
H	0.729567	-2.507602	1.309705
H	1.962145	-1.540439	2.222946



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IM2''

Cl	-0.032147	1.200385	-1.422099
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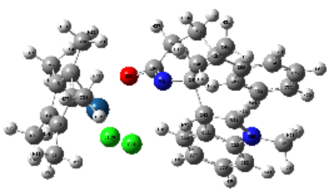
C	3.422520	0.875023	-0.651843
C	3.720655	-0.394546	-0.007683
C	3.183601	-0.362233	1.324821
C	2.599201	0.961774	1.535248
C	2.364949	3.146404	0.096552
H	3.252444	3.779111	-0.069157
H	1.804013	3.553725	0.947725
H	1.714023	3.204929	-0.788197
C	3.825469	1.297237	-2.027503
H	3.061474	1.953863	-2.465514
H	3.933847	0.426867	-2.688329
H	4.786212	1.840595	-2.009880
C	4.436734	-1.535056	-0.651437
H	5.525133	-1.357137	-0.623594
H	4.125127	-1.652000	-1.697544
H	4.224246	-2.481915	-0.139540
C	3.239293	-1.447259	2.350818
H	4.079315	-1.289270	3.049316
H	3.365363	-2.431620	1.880792
H	2.310393	-1.474032	2.937547
C	1.923339	1.389131	2.797208
H	2.653704	1.484136	3.617919

H	1.165615	0.648358	3.091425
H	1.415354	2.354137	2.672580
Cl	1.186027	-2.005825	-1.472833
C	-2.944858	-1.727338	-0.600150
C	-4.086159	-1.198257	0.225083
C	-3.711533	-1.464822	1.691608
H	-4.471940	-1.101272	2.399403
H	-3.598148	-2.550584	1.847057
N	-1.622648	-1.451694	-0.303395
C	-2.372385	-0.749319	1.951031
C	-1.290341	-0.941912	0.896906
H	-1.922532	-1.042935	2.911027
H	-2.529393	0.341374	2.006169
O	-0.121295	-0.612153	1.185710
C	-4.476491	0.252359	0.005484
C	-5.713410	0.706655	0.491036
C	-3.632051	1.162110	-0.644976
C	-6.099425	2.038007	0.330628
H	-6.384678	0.005322	0.995378
C	-4.015338	2.498564	-0.799160
H	-2.659804	0.848793	-1.029248
C	-5.247352	2.941256	-0.314763
H	-7.068636	2.372871	0.709019
H	-3.333956	3.190089	-1.300484
H	-5.546157	3.985118	-0.438412
C	-4.961918	-2.236269	-0.589595
C	-3.679598	-2.571899	-1.346076
H	-5.358057	-3.062491	0.025643
H	-5.787828	-1.782515	-1.159948
H	-3.443651	-3.291646	-2.130713
H	-0.842786	-1.666813	-0.950663

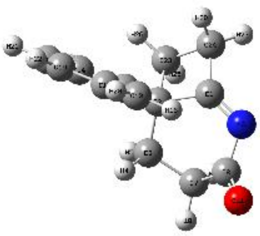


75

IM3

Cl	-2.311018	-2.500629	-0.659044
Ir	-1.957416	-0.132707	-0.189098
C	-4.111695	0.107358	0.149637
C	-3.655767	1.159895	-0.693653
C	-2.657776	1.932106	0.044637
C	-2.550638	1.366881	1.356674
C	-3.379969	0.177555	1.408638
C	-5.167298	-0.900277	-0.164481
H	-5.438427	-0.879278	-1.227720
H	-6.071679	-0.690639	0.431384
H	-4.812447	-1.914203	0.065277
C	-4.151590	1.484036	-2.065099
H	-4.585836	0.599789	-2.549874
H	-3.334383	1.840380	-2.704629
H	-4.927809	2.267098	-2.011385
C	-1.989755	3.165431	-0.472352
H	-2.689120	4.019449	-0.485662
H	-1.628775	2.997766	-1.497358
H	-1.123963	3.440293	0.144718
C	-1.860875	1.973628	2.534985
H	-1.239952	2.831774	2.247336
H	-1.250426	1.220709	3.046410
H	-2.628641	2.342188	3.237588
C	-3.654302	-0.665509	2.609823
H	-2.736200	-0.799500	3.193395
H	-4.009941	-1.659670	2.306174
H	-4.432449	-0.200324	3.241865
Cl	-1.064764	0.351912	-2.521368
O	-0.628830	-0.743803	2.653887
N	3.556832	1.268115	-1.662139
N	-0.002766	-0.851871	0.461726
C	3.075876	2.010336	-0.561574
C	3.644523	3.123230	0.054692
H	4.572527	3.572254	-0.301924
C	2.974608	3.630945	1.169363
H	3.382259	4.499199	1.691457
C	1.785654	3.036454	1.629589
H	1.288698	3.449339	2.510357
C	1.227941	1.926577	0.990733
H	0.320662	1.454331	1.353274
C	1.887846	1.399776	-0.120337
C	1.596148	0.252487	-1.024593
C	2.740547	0.256040	-1.914980
H	2.940102	-0.422448	-2.741615
C	1.061147	-1.134652	-0.468276
C	0.823470	-2.109024	-1.660645
H	-0.042332	-2.747294	-1.458069
H	0.659284	-1.640190	-2.638298

C	2.182518	-2.780288	-1.406105
H	2.969027	-2.459236	-2.103111
H	2.195265	-3.879733	-1.379864
C	2.255617	-2.094575	-0.016846
C	1.712811	-3.043965	1.060276
H	0.918420	-3.659108	0.609641
H	2.510011	-3.727772	1.393620
C	1.113825	-2.260217	2.208660
H	0.552448	-2.915978	2.890231
H	1.881619	-1.778033	2.832256
C	0.099154	-1.203150	1.767877
C	3.575515	-1.466831	0.377572
C	3.662677	-0.476102	1.373113
H	2.764443	-0.138684	1.882305
C	4.883602	0.113471	1.706140
H	4.909962	0.892482	2.471347
C	6.058228	-0.279860	1.060298
H	7.014213	0.180483	1.321764
C	5.997768	-1.281861	0.087412
H	6.910038	-1.617886	-0.412423
C	4.772392	-1.865873	-0.244599
H	4.748286	-2.653005	-0.999953
C	4.813307	1.545940	-2.335681
H	4.934280	0.853411	-3.177173
H	5.642849	1.404215	-1.627684
H	4.819602	2.580048	-2.709180
H	0.686815	0.521362	-1.690507



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IM4'

C	-1.210150	0.587547	-0.915040
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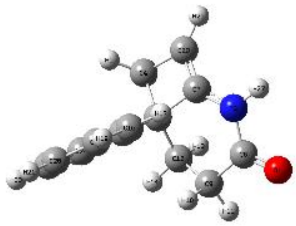
C	-0.982700	0.186970	1.594567
H	-0.384998	-0.660626	1.958127
H	-0.970369	0.950213	2.388511
N	-2.214066	-0.172263	-1.074211
C	-2.427677	-0.308466	1.346539
C	-2.501541	-1.037598	0.004696
H	-3.123472	0.545693	1.309008
H	-2.753734	-0.988469	2.144509
O	-2.788319	-2.201446	-0.132700
C	1.078681	0.170861	0.117292
C	1.274145	-0.992017	-0.642041
C	2.183750	0.755418	0.752436
C	2.547893	-1.552858	-0.769498
H	0.421985	-1.467227	-1.134123
C	3.456519	0.194227	0.628469
H	2.049054	1.656550	1.355910
C	3.643286	-0.961176	-0.136066
H	2.682533	-2.456660	-1.368477
H	4.306701	0.662096	1.130886
H	4.639323	-1.398503	-0.236712
C	-0.386264	2.282344	-0.035115
C	-0.830560	1.924560	-1.492804
H	-1.190665	2.782329	0.522909
H	0.547845	2.848015	0.069949
H	-1.636385	2.500981	-1.968070
H	0.036511	1.861383	-2.167638



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IM4''

O	-3.059150	-2.127495	0.012869
N	-2.117833	-0.293289	-0.932948
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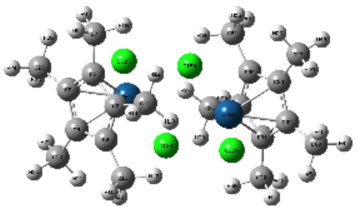
C	-0.273105	2.490468	-0.093650
H	-0.666488	3.151764	0.697593
H	0.713056	2.861554	-0.415916
C	-1.339747	0.837418	-0.782143
C	-2.284494	-1.200013	0.112341
C	-1.439898	-0.960727	1.368010
H	-0.569043	-1.633321	1.282533
H	-2.040804	-1.339390	2.206667
C	-0.955767	0.482708	1.600967
H	-1.812313	1.137797	1.832028
H	-0.269934	0.515990	2.461253
C	1.030254	0.255952	0.054080

C	1.216449	-0.663384	-0.986366
H	0.386171	-0.899581	-1.653849
C	2.454429	-1.286654	-1.175962
H	2.579139	-2.003147	-1.991596
C	3.525485	-0.997099	-0.329059
H	4.492417	-1.483513	-0.477673
C	3.352019	-0.077734	0.711036
H	4.185501	0.160098	1.376683
C	2.115341	0.539927	0.899140
C	-1.285300	2.125087	-1.174591
H	-1.826659	2.716626	-1.914391
H	-2.786271	-0.382460	-1.693942
H	1.989729	1.261898	1.710789



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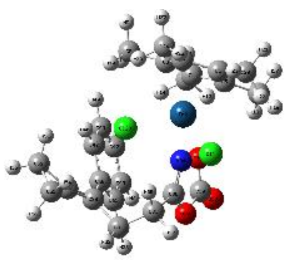
Ir2

Ir	1.840611	-0.209077	0.000028
C	3.525699	0.380805	1.171734
C	2.829130	1.584901	0.715182
C	2.828572	1.585770	-0.713840
C	3.524838	0.382225	-1.172326
C	3.986723	-0.331543	-0.000929
C	3.784397	0.012514	2.596305
H	4.671466	0.542316	2.984376
H	3.955892	-1.067151	2.698761
H	2.925381	0.275537	3.228679
C	2.198173	2.627218	1.579797
H	1.996296	2.239088	2.585904
H	1.237321	2.954988	1.157571
H	2.866605	3.500503	1.670446
C	2.197035	2.629115	-1.576800
H	2.865022	3.502869	-1.666185
H	1.236027	2.955765	-1.154042
H	1.995277	2.242412	-2.583480
C	3.782128	0.015635	-2.597592
H	4.668144	0.546780	-2.986221
H	2.922031	0.278415	-3.228598
H	3.954620	-1.063740	-2.701386
C	4.775777	-1.598435	-0.001879
H	5.855538	-1.371687	-0.000869
H	4.544866	-2.206134	-0.885920
H	4.543603	-2.208179	0.880428
Cl	1.433973	-2.586203	-0.000204
Cl	0.000007	0.000119	-1.682729
Ir	-1.840616	0.209093	-0.000104
C	-2.829260	-1.584750	-0.715382
C	-3.525973	-0.380573	-1.171508
C	-3.986730	0.331493	0.001454
C	-3.524534	-0.382529	1.172552
C	-2.828342	-1.585951	0.713638
C	-2.198454	-2.626859	-1.580355
H	-2.866968	-3.500060	-1.671210
H	-1.237607	-2.954833	-1.158283
H	-1.996614	-2.238453	-2.586364
C	-3.785084	-0.012007	-2.595933
H	-2.926396	-0.275244	-3.228661
H	-3.956223	1.067734	-2.698192
H	-4.672482	-0.541443	-2.983751
C	-4.775822	1.598362	0.002997
H	-5.855576	1.371580	0.003891
H	-4.545143	2.207634	-0.880022
H	-4.543451	2.206549	0.886326
C	-3.781425	-0.016232	2.597966
H	-4.667045	-0.547836	2.986869
H	-3.954384	1.063048	2.701954
H	-2.920957	-0.278699	3.228602
C	-2.196582	-2.629484	1.576213
H	-2.864331	-3.503463	1.665176
H	-1.995020	-2.243147	2.583071
H	-1.235445	-2.955694	1.153401
Cl	-1.434065	2.586234	-0.000034
Cl	-0.000014	-0.000076	1.682718



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IrD

Cl	1.555144	-0.593506	-2.544904

Ir	1.272885	-0.201377	-0.167482
C	1.921602	-0.348235	1.909525
C	1.866663	1.035659	1.547015
C	2.771185	1.256114	0.426346
C	3.424142	-0.005012	0.145383
C	2.879943	-1.007520	1.023047
C	1.179191	-1.019439	3.017161
H	1.862780	-1.264208	3.847494
H	0.717855	-1.949762	2.655996
H	0.381237	-0.374209	3.406270
C	1.057432	2.083712	2.240973
H	1.539790	2.352867	3.195787
H	0.041786	1.728210	2.457205
H	0.968041	2.991268	1.633374
C	3.079757	2.567083	-0.222081
H	3.879729	3.099964	0.320728
H	2.193213	3.215082	-0.243632
H	3.411774	2.419708	-1.258671
C	4.468029	-0.214053	-0.900470
H	5.435507	0.174704	-0.540520
H	4.198720	0.302060	-1.831223
H	4.585573	-1.277911	-1.140053
C	3.254878	-2.451990	1.099104
H	3.792140	-2.771077	0.196592
H	2.352425	-3.073336	1.184064
H	3.900856	-2.640995	1.973086
Cl	-0.107609	-2.218311	-0.008095
C	-3.446489	-2.388775	-0.093024
C	-3.399974	-1.103383	-0.444141
C	-3.317202	-0.749013	-1.913021
H	-3.649136	-1.608164	-2.511946
H	-3.975388	0.095838	-2.171279
N	-0.399851	0.920002	-0.827784
C	-1.865102	-0.423861	-2.354431
C	-1.352228	0.791910	-1.693011
H	-1.831357	-0.241893	-3.440643
H	-1.187490	-1.251182	-2.114544
O	-1.965916	1.976060	-1.931155
C	-3.285218	-0.021674	0.567508
C	-3.854440	1.248119	0.357724
C	-2.565412	-0.233219	1.759913
C	-3.713136	2.270118	1.300382
H	-4.420654	1.445606	-0.554058
C	-2.443391	0.779598	2.709950
H	-2.069199	-1.190345	1.909373
C	-3.008947	2.039520	2.484330
H	-4.156114	3.249484	1.106237
H	-1.891248	0.587815	3.633743
H	-2.898780	2.836148	3.223745
C	-3.405893	-3.778724	-0.550212
C	-3.395394	-3.417118	0.944477
H	-4.314426	-4.238723	-0.956670
H	-2.469632	-4.139809	-0.991752
H	-4.298397	-3.628968	1.529210
H	-2.453898	-3.551374	1.488125
O	-0.395481	2.279148	-0.447345
O	-1.672324	4.066565	-1.040942
C	-1.382872	2.922837	-1.122262

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IrD-TS

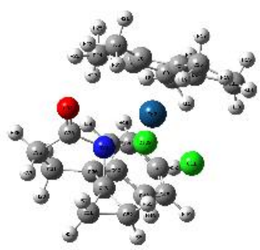
Cl	-2.421182	-0.903877	-2.090522
Ir	-1.269834	0.020479	-0.207873
C	-1.154189	1.004657	1.803580
C	-0.893051	-0.394618	1.932084
C	-2.080537	-1.103706	1.458349
C	-3.116689	-0.112025	1.190707
C	-2.552355	1.167577	1.383567
C	-0.246573	2.132828	2.159009
H	-0.486735	2.521937	3.163165
H	-0.363376	2.952236	1.436835
H	0.803331	1.814889	2.151034
C	0.351008	-1.027960	2.452771
H	0.189950	-1.342910	3.497575
H	1.196148	-0.331070	2.431133
H	0.619086	-1.909699	1.858226
C	-2.300000	-2.580822	1.456376
H	-2.761599	-2.891967	2.409791
H	-1.355594	-3.118143	1.315114
H	-2.972860	-2.867880	0.637076
C	-4.495843	-0.450299	0.733159
H	-4.997824	-1.088166	1.477784

H	-4.451094	-0.999502	-0.220565
H	-5.102977	0.450199	0.577420
C	-3.222785	2.488654	1.221698
H	-4.243239	2.381167	0.833839
H	-2.658167	3.122569	0.522142
H	-3.268870	3.003368	2.195449
Cl	-1.181045	2.106547	-1.384380
C	2.629957	2.529563	-0.858134
C	2.987211	1.240055	-0.863267
C	2.983763	0.534536	-2.209053
H	3.295873	1.256577	-2.978315
H	3.708582	-0.290924	-2.239873
N	0.244621	-0.822603	-0.856964
C	1.592150	0.025382	-2.627967
C	1.110075	-1.121024	-1.758797
H	1.619731	-0.382576	-3.650822
H	0.860615	0.842310	-2.594501
O	1.686020	-2.260641	-1.923371
C	3.335079	0.497530	0.373493
C	3.559736	-0.892442	0.362063
C	3.456938	1.162732	1.613449
C	3.866508	-1.591331	1.532084
H	3.493607	-1.456306	-0.566342
C	3.776405	0.468574	2.778924
H	3.313461	2.241666	1.656242
C	3.977956	-0.916659	2.748384
H	4.013760	-2.672570	1.480005
H	3.870723	1.014141	3.721389
H	4.222712	-1.460371	3.663657
C	2.201259	3.663724	-1.680312
C	2.352398	3.783965	-0.157871
H	2.939544	4.172916	-2.312077
H	1.182848	3.650576	-2.083801
H	3.193257	4.370762	0.231961
H	1.438272	3.861453	0.440382
O	0.416485	-2.924134	-0.112910
O	2.054581	-4.312223	-0.890262
C	1.339394	-3.364352	-0.804851

28

Ir

Ir	-0.012149	0.418191	-0.007978
C	0.683204	-1.315496	1.042669
C	1.246834	-1.354332	-0.275344
C	0.136907	-1.337542	-1.209104
C	-1.114117	-1.406974	-0.463483
C	-0.779208	-1.362179	0.926775
C	1.451661	-1.285235	2.322043
H	1.601594	-2.310725	2.701987
H	0.917309	-0.713125	3.092837
H	2.436900	-0.823002	2.178859
C	2.705336	-1.337967	-0.603764
H	3.215315	-0.524943	-0.066256
H	2.868880	-1.171335	-1.676367
H	3.179464	-2.294919	-0.330080
C	0.254138	-1.320504	-2.697073
H	0.292428	-2.352131	-3.087730
H	1.165176	-0.798022	-3.016885
H	-0.605256	-0.813870	-3.155843
C	-2.493186	-1.462398	-1.036433
H	-2.888770	-2.491552	-1.007767
H	-2.502298	-1.122383	-2.080310
H	-3.174496	-0.808596	-0.474544
C	-1.739366	-1.385678	2.069731
H	-1.903332	-2.421720	2.412958
H	-2.708757	-0.960444	1.779470
H	-1.357619	-0.803026	2.919281
Cl	-1.717146	2.007108	0.005304
Cl	1.616306	2.088123	-0.007154



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IrZ

Cl	0.334526	-1.009965	-2.130326
Ir	-0.871203	-0.105638	-0.222203
C	-0.946708	-1.864644	1.048514
C	-1.944386	-1.986866	-0.000701
C	-2.862142	-0.892644	0.117481

C	-2.468588	-0.084696	1.265783
C	-1.293899	-0.697218	1.832940
C	0.127657	-2.867579	1.318778
H	-0.305391	-3.822660	1.663579
H	0.822578	-2.513439	2.089224
H	0.714659	-3.063761	0.410256
C	-1.990173	-3.096533	-0.997750
H	-0.991969	-3.279426	-1.417116
H	-2.659097	-2.856954	-1.833987
H	-2.353107	-4.018368	-0.512007
C	-4.050889	-0.611885	-0.741703
H	-4.980028	-0.840222	-0.192535
H	-4.032299	-1.214511	-1.658827
H	-4.063885	0.446183	-1.038411
C	-3.243604	1.090774	1.764408
H	-4.230672	0.767843	2.137410
H	-3.396422	1.813344	0.949789
H	-2.716688	1.613900	2.569804
C	-0.571286	-0.249372	3.062821
H	-0.873627	-0.868057	3.924602
H	-0.790668	0.798693	3.297490
H	0.515877	-0.358392	2.943446
Cl	-1.674625	1.772611	-1.571442
C	1.676147	1.820131	-0.884726
C	2.325402	1.136425	0.225907
C	2.872464	2.083576	1.296235
H	3.147949	1.521518	2.198099
H	3.787401	2.567784	0.923365
N	0.770668	1.319321	0.174132
C	1.741814	3.066619	1.593511
C	0.467496	2.375483	1.139148
H	1.832638	4.007787	1.030565
H	1.640038	3.331265	2.654218
O	-0.634245	2.599595	1.542815
C	2.893481	-0.243062	0.171285
C	3.071444	-0.968928	1.359518
C	3.299825	-0.821487	-1.037528
C	3.635354	-2.244304	1.342422
H	2.758494	-0.539721	2.313538
C	3.869670	-2.095355	-1.057049
H	3.151371	-0.283155	-1.969124
C	4.036547	-2.811707	0.129745
H	3.757934	-2.797779	2.276316
H	4.172473	-2.534010	-2.010197
H	4.478953	-3.810607	0.111359
C	1.723595	3.153619	-1.541397
C	1.571492	1.879113	-2.353459
H	0.822517	3.767567	-1.452998
H	2.675378	3.694909	-1.549463
H	0.587619	1.671905	-2.776312
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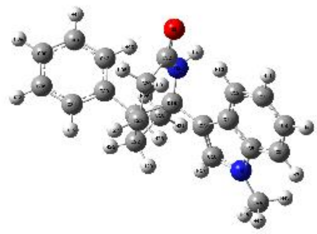

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***trans*-3**

O	-0.799268	3.068634	-0.698231
N	3.234362	-1.679342	-0.066560
N	-0.482421	0.893311	-1.235817
H	-0.160925	1.197373	-2.150699
C	3.482773	-0.332616	0.083130
C	4.696668	0.296619	0.388691
H	5.605189	-0.283827	0.560601
C	4.704734	1.684572	0.458791
H	5.632384	2.211052	0.694738
C	3.526027	2.416084	0.214885
H	3.554499	3.507275	0.256931
C	2.321560	1.787868	-0.088368
H	1.460597	2.418584	-0.289163
C	2.260249	0.375917	-0.146499
C	1.240467	-0.632372	-0.429085
C	1.906128	-1.845374	-0.381105
H	1.531487	-2.850600	-0.550296
C	-0.247521	-0.447141	-0.750447
C	-0.790361	-1.770606	-1.372704
H	-1.713408	-1.621514	-1.944545
H	-0.083152	-2.355062	-1.976640
C	-1.110526	-2.236637	0.084461
H	-0.265674	-2.729050	0.579715
H	-2.014642	-2.842192	0.227235
C	-1.241106	-0.721320	0.437516
C	-0.752420	0.089080	1.630350
H	-1.335937	-0.062013	2.549826
H	0.293078	-0.162293	1.855231

C	-0.880203	1.562965	1.163720
H	-1.870228	1.962765	1.429961
H	-0.149034	2.207954	1.673498
C	-0.727755	1.906290	-0.336902
C	-2.735676	-0.388279	0.211345
C	-3.595692	-0.761597	1.262216
H	-3.177168	-1.236795	2.152688
C	-4.972384	-0.554970	1.193977
H	-5.607056	-0.856664	2.030981
C	-5.537233	0.032401	0.057868
H	-6.615095	0.200486	-0.001542
C	-4.704233	0.397199	-0.998180
H	-5.125747	0.855984	-1.895941
C	-3.321573	0.190001	-0.923853
H	-2.700328	0.500106	-1.759884
C	4.218087	-2.727143	0.066316
H	5.034872	-2.601132	-0.663844
H	3.739557	-3.699301	-0.110845
H	4.657230	-2.733644	1.077739

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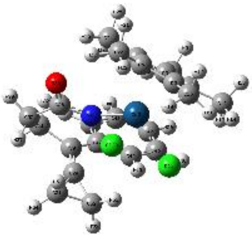
TS1_pdt

Cl	0.333246	-1.009592	-2.131180
Ir	-0.871205	-0.105354	-0.222008
C	-0.947051	-1.865525	1.046787
C	-1.945795	-1.986016	-0.001697
C	-2.862719	-0.891377	0.118550
C	-2.467440	-0.084755	1.267128
C	-1.292699	-0.698714	1.832724
C	0.126899	-2.869488	1.314734
H	-0.306260	-3.824013	1.660955
H	0.824233	-2.515860	2.083215
H	0.711306	-3.066526	0.404718
C	-1.993359	-3.094915	-0.999494
H	-0.996143	-3.276438	-1.421853
H	-2.664985	-2.855440	-1.833595
H	-2.353827	-4.017512	-0.513380
C	-4.052162	-0.608576	-0.739029
H	-4.980986	-0.830243	-0.186648
H	-4.038704	-1.215005	-1.653727
H	-4.060897	0.448434	-1.039813
C	-3.241450	1.090545	1.767699
H	-4.228376	0.767625	2.141123
H	-3.394638	1.814123	0.954048
H	-2.713700	1.612471	2.573331
C	-0.569154	-0.252577	3.062667
H	-0.875354	-0.868577	3.925020
H	-0.784035	0.796802	3.295720
H	0.517597	-0.366863	2.944568
Cl	-1.674501	1.773573	-1.570420
C	1.676157	1.819547	-0.885130
C	2.325907	1.136190	0.225434
C	2.873388	2.083782	1.295131
H	3.149443	1.522183	2.197103
H	3.788034	2.567968	0.921515
N	0.771026	1.319125	0.174083
C	1.742782	3.066844	1.592590
C	0.468313	2.375456	1.139090
H	1.833295	4.007887	1.029398
H	1.641465	3.331711	2.653286
O	-0.633199	2.599438	1.543477
C	2.893739	-0.243399	0.171149
C	3.072497	-0.968535	1.359741
C	3.298868	-0.822736	-1.037610
C	3.635984	-2.244097	1.343007
H	2.760570	-0.538527	2.313732
C	3.868147	-2.096879	-1.056777
H	3.149745	-0.285060	-1.969474
C	4.035853	-2.812481	0.130341
H	3.759289	-2.796963	2.277169
H	4.169897	-2.536286	-2.009913
H	4.477864	-3.811560	0.112225
C	1.724063	3.152910	-1.542061
C	1.571530	1.878310	-2.353872
H	0.823188	3.767179	-1.453856
H	2.676064	3.693818	-1.550134
H	0.587553	1.671418	-2.776636
H	2.427144	1.517350	-2.929309



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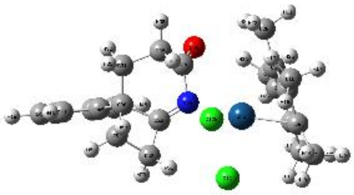
TS1

Cl	0.431147	0.888818	-2.387996
Ir	0.904554	-0.000873	-0.196922
C	0.928097	1.838828	1.137776
C	1.957806	2.012680	0.136454
C	2.879763	0.921428	0.222733
C	2.450510	0.069305	1.330814
C	1.267192	0.671415	1.905287
C	-0.207197	2.771911	1.403726
H	0.090046	3.554300	2.123984
H	-1.073873	2.238375	1.816020
H	-0.538850	3.265262	0.481634
C	2.024943	3.161824	-0.809961
H	1.030466	3.409654	-1.202044
H	2.673644	2.944967	-1.666444
H	2.423438	4.042567	-0.277274
C	4.090550	0.683189	-0.619089
H	5.010994	0.919537	-0.059745
H	4.067278	1.298079	-1.527791
H	4.129764	-0.369719	-0.933424
C	3.189629	-1.117358	1.849879
H	3.825992	-0.822503	2.702020
H	3.827578	-1.546234	1.067175
H	2.477747	-1.891960	2.162003
C	0.525475	0.200482	3.110727
H	0.812539	0.818377	3.978398
H	0.741718	-0.851441	3.324231
H	-0.558378	0.304654	2.963799
Cl	1.695672	-1.954603	-1.350811
C	-2.284302	-1.688803	-1.161246
C	-2.806502	-1.124036	-0.040688
C	-3.012839	-2.005754	1.173019
H	-3.059358	-1.380088	2.075400
H	-3.978438	-2.538246	1.108898
N	-0.467081	-1.134621	0.486114
C	-1.855084	-2.987394	1.318938
C	-0.509735	-2.280362	1.202342
H	-1.892169	-3.777060	0.554571
H	-1.860832	-3.490922	2.295843
O	0.466336	-2.677303	1.847053
C	-3.193863	0.299183	0.007695
C	-4.057800	0.787119	1.009076
C	-2.684510	1.219032	-0.930408
C	-4.399765	2.139103	1.069202
H	-4.483845	0.103688	1.744772
C	-3.022661	2.569142	-0.864455
H	-1.983378	0.887348	-1.691754
C	-3.880962	3.039471	0.134224
H	-5.078151	2.489974	1.850797
H	-2.599012	3.259102	-1.598526
H	-4.144264	4.098783	0.184871
C	-1.898736	-2.921719	-1.854004
C	-1.962164	-1.579952	-2.580370
H	-0.890836	-3.312657	-1.671968
H	-2.669628	-3.678673	-2.042227
H	-1.005764	-1.128517	-2.858831
H	-2.789203	-1.372709	-3.270906



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TS2_IM2

Cl	0.765061	-0.891059	-2.411369
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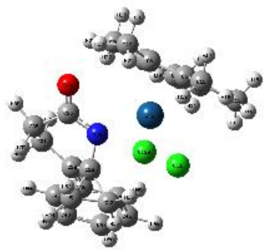
C	2.863335	1.202943	-1.023898
C	3.449248	-0.016746	-0.483314
C	3.198204	-0.022434	0.941077
C	2.410307	1.135240	1.273163
C	2.204390	1.893234	0.042593
C	2.977756	1.635401	-2.447554
H	4.026007	1.888610	-2.678407
H	2.355929	2.516198	-2.652737
H	2.650544	0.828326	-3.118699
C	4.250132	-1.008252	-1.264125
H	3.777281	-1.194632	-2.238292
H	4.309808	-1.968710	-0.735272

H	5.277179	-0.641761	-1.433969
C	3.689478	-1.053373	1.902035
H	4.712766	-0.799273	2.227604
H	3.699971	-2.048520	1.440403
H	3.044845	-1.113152	2.787782
C	2.048737	1.585737	2.651666
H	2.863127	2.197882	3.077455
H	1.891086	0.728729	3.321493
H	1.134604	2.191886	2.651500
C	1.516490	3.216043	-0.050591
H	2.182982	4.014327	0.319675
H	0.598358	3.219965	0.548798
H	1.247891	3.451135	-1.088883
Cl	0.829072	-2.328920	0.762558
C	-1.770231	-0.616079	-0.199332
C	-3.081475	-0.861744	0.531576
C	-2.977805	-0.650713	2.068787
H	-3.712611	0.104895	2.378631
H	-3.229981	-1.586666	2.588928
N	-0.799272	0.121685	0.207179
C	-1.567835	-0.177777	2.493990
C	-1.061515	0.791785	1.454012
H	-0.866696	-1.028807	2.508403
H	-1.590705	0.313640	3.475223
O	-0.902004	1.977658	1.603915
C	-4.244089	-0.074024	-0.055390
C	-5.546694	-0.588606	0.021717
C	-4.051022	1.193292	-0.622277
C	-6.632083	0.145756	-0.458745
H	-5.714849	-1.573210	0.465006
C	-5.137184	1.927821	-1.107427
H	-3.045608	1.615464	-0.683872
C	-6.430143	1.406926	-1.028254
H	-7.640452	-0.269739	-0.390927
H	-4.967864	2.911701	-1.551252
H	-7.278577	1.979980	-1.409310
C	-2.967029	-2.313989	-0.062315
C	-1.932956	-1.795687	-1.106296
H	-2.486256	-2.995877	0.651558
H	-3.895578	-2.750192	-0.450703
H	-1.026107	-2.373775	-1.304202
H	-2.407450	-1.506722	-2.055917



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TS2

Cl	0.289903	-2.037551	-0.962522
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C	-1.656792	-1.214958	1.660890
C	-2.561891	-1.663873	0.609123
C	-3.289062	-0.529459	0.141635
C	-2.860254	0.643061	0.891446
C	-1.875578	0.194267	1.857586
C	-0.755015	-2.112421	2.444981
H	-1.333652	-2.738643	3.145989
H	-0.027225	-1.532815	3.029358
H	-0.196261	-2.772695	1.765925
C	-2.700565	-3.078744	0.152332
H	-1.712501	-3.526221	-0.020917
H	-3.262713	-3.142006	-0.788306
H	-3.233169	-3.671667	0.915582
C	-4.332755	-0.507224	-0.925876
H	-5.317993	-0.282177	-0.484605
H	-4.395495	-1.470739	-1.447302
H	-4.094119	0.260398	-1.675580
C	-3.513461	1.982247	0.796697
H	-4.442958	1.998540	1.393805
H	-3.775089	2.202878	-0.247599
H	-2.836805	2.771307	1.137084
C	-1.258511	1.008985	2.947589
H	-1.806616	0.839067	3.890620
H	-1.278045	2.075606	2.703159
H	-0.211444	0.716168	3.113907
Cl	-1.512107	0.617334	-2.456180
C	1.722678	0.961874	-0.896215
C	2.810455	0.992986	0.059541
C	2.781280	2.180430	0.965072
H	2.609581	1.822223	1.997699
H	3.783559	2.642809	0.991810
N	0.462515	1.238210	-0.232901
C	1.703997	3.178942	0.556547

C	0.363219	2.462811	0.353990
H	1.991697	3.677070	-0.383220
H	1.555244	3.954464	1.317493
O	-0.667550	2.976255	0.769977
C	3.841840	-0.001863	0.174172
C	5.131390	0.320697	0.675510
C	3.561384	-1.351655	-0.166432
C	6.108382	-0.658091	0.794023
H	5.375776	1.352524	0.930529
C	4.542823	-2.326827	-0.017837
H	2.548774	-1.628297	-0.477933
C	5.815965	-1.986223	0.450527
H	7.104242	-0.392311	1.154791
H	4.305883	-3.364408	-0.262452
H	6.583996	-2.756118	0.557353
C	2.080722	1.760778	-2.216644
C	1.796081	0.309647	-2.247026
H	1.272528	2.444487	-2.492462
H	3.102472	2.127328	-2.352902
H	0.829674	0.002500	-2.648261
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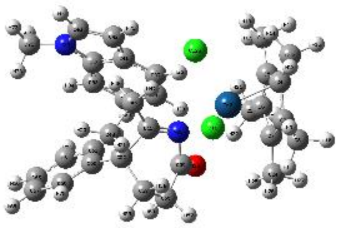

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TS3-SM

Cl	2.144291	1.878647	-1.722422
Ir	2.028711	-0.039588	-0.209148
C	4.068601	0.341655	0.453116
C	3.979635	-0.922437	-0.244285
C	3.120462	-1.793786	0.544872
C	2.641436	-1.053314	1.671107
C	3.220195	0.285256	1.614197
C	4.918022	1.497229	0.040541
H	4.937859	1.601952	-1.051296
H	5.948409	1.345694	0.405496
H	4.537444	2.440711	0.451232
C	4.710545	-1.319140	-1.486300
H	5.006357	-0.434830	-2.066134
H	4.065025	-1.934992	-2.127483
H	5.619994	-1.895313	-1.243445
C	2.834910	-3.222734	0.222387
H	3.742544	-3.829875	0.377394
H	2.514446	-3.322092	-0.824154
H	2.035395	-3.627251	0.856273
C	1.821956	-1.596105	2.794682
H	1.256701	-2.484182	2.484263
H	1.110675	-0.845708	3.154598
H	2.483711	-1.889382	3.628317
C	3.119786	1.331234	2.677145
H	2.153569	1.285556	3.193740
H	3.234849	2.338856	2.253327
H	3.917110	1.190860	3.428057
Cl	1.086250	-1.418359	-1.986279
O	0.034769	0.940397	2.443183
N	-4.532063	-2.565755	-0.986458
N	0.052448	0.834616	0.142249
C	-3.770910	-2.421792	0.155529
C	-4.163263	-2.378677	1.500269
H	-5.210253	-2.484045	1.792300
C	-3.166917	-2.182238	2.454033
H	-3.438177	-2.131578	3.511333
C	-1.812848	-2.038149	2.079476
H	-1.065516	-1.858996	2.852697
C	-1.420619	-2.105609	0.745247
H	-0.378414	-1.981344	0.449944
C	-2.405073	-2.300423	-0.241533
C	-2.381456	-2.389442	-1.677627
C	-3.685944	-2.541660	-2.084441
H	-4.097526	-2.643016	-3.087459
C	-0.810203	1.161099	-0.760358
C	-1.022955	0.906449	-2.215079
H	-0.128703	0.685558	-2.805529
H	-1.754047	0.091092	-2.339029
C	-1.694373	2.309384	-2.301005
H	-2.674452	2.347489	-2.791843
H	-1.021122	3.064070	-2.728132
C	-1.731227	2.373477	-0.732358
C	-0.910421	3.483983	-0.049500
H	-0.001037	3.656501	-0.644969
H	-1.477133	4.425567	0.006788
C	-0.525712	2.985795	1.342061
H	0.332536	3.543167	1.752254
H	-1.347555	3.100226	2.065487

C	-0.131189	1.513411	1.395661
C	-3.110455	2.210511	-0.127627
C	-3.452293	1.130157	0.694820
H	-2.730527	0.339277	0.905113
C	-4.733305	1.046490	1.253507
H	-4.973940	0.197120	1.893562
C	-5.684991	2.032146	0.991580
H	-6.683605	1.963529	1.429923
C	-5.353502	3.110229	0.162679
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C	-4.076633	3.196313	-0.389986
H	-3.822955	4.039260	-1.038598
C	-5.971768	-2.648892	-1.014249
H	-6.308877	-2.783033	-2.050696
H	-6.432205	-1.729636	-0.613243
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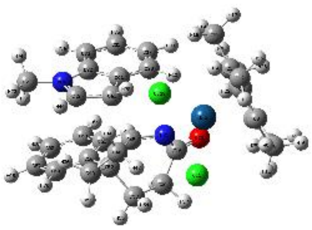
H	-1.493681	-2.346360	-2.303062



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TS3

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C	-4.105176	-0.059671	0.501650
C	-3.932074	0.802292	-0.625749
C	-2.968169	1.834599	-0.252304
C	-2.590341	1.612400	1.111076
C	-3.231659	0.394811	1.570973
C	-5.037656	-1.221186	0.596610
H	-5.346101	-1.569549	-0.396924
H	-5.938287	-0.926066	1.161941
H	-4.557474	-2.068003	1.102960
C	-4.646925	0.731740	-1.935711
H	-5.126109	-0.246183	-2.074952
H	-3.943423	0.877759	-2.766574
H	-5.427355	1.510220	-1.988467
C	-2.555289	2.968426	-1.133327
H	-3.362172	3.716983	-1.214818
H	-2.318721	2.595468	-2.139952
H	-1.658188	3.469734	-0.744866
C	-1.821273	2.551214	1.981716
H	-1.344844	3.350402	1.399416
H	-1.061508	2.009587	2.555314
H	-2.523123	3.028933	2.687552
C	-3.204763	-0.149864	2.961608
H	-2.208317	-0.029077	3.404018
H	-3.448814	-1.221217	2.961348
H	-3.944762	0.367449	3.598486
Cl	-1.399816	-0.001232	-2.569076
O	-0.159783	0.083435	2.549454
N	3.812025	1.026480	-1.786624
N	0.042015	-0.603325	0.365068
C	3.365776	1.894855	-0.775290
C	4.067431	2.872231	-0.071802
H	5.121587	3.077769	-0.263031
C	3.361582	3.566691	0.913549
H	3.871295	4.340494	1.491908
C	2.008372	3.282460	1.176461
H	1.487497	3.836798	1.960384
C	1.321713	2.299473	0.460862
H	0.286039	2.054987	0.674495
C	2.011309	1.593226	-0.525211
C	1.623352	0.478473	-1.400984
C	2.800035	0.234451	-2.170037
H	2.941013	-0.485440	-2.973429
C	1.003119	-1.057487	-0.506239
C	0.779439	-2.043348	-1.673694
H	-0.108946	-2.653523	-1.471903
H	0.659709	-1.614662	-2.673254
C	2.117860	-2.731785	-1.342111
H	2.934731	-2.467364	-2.026916
H	2.091920	-3.827376	-1.253304
C	2.173188	-1.971582	0.006581
C	1.541236	-2.831186	1.122474
H	0.690348	-3.383999	0.697114
H	2.276667	-3.564331	1.488845
C	1.030647	-1.935923	2.234354
H	0.297950	-2.466410	2.863031
H	1.827615	-1.597633	2.912219
C	0.280560	-0.706796	1.726234
C	3.502130	-1.406602	0.452473
C	3.601533	-0.332104	1.352351
H	2.706623	0.178438	1.701559

C	4.845676	0.121528	1.796669
H	4.888464	0.967191	2.486101
C	6.021926	-0.490711	1.358343
H	6.993895	-0.137305	1.710981
C	5.940405	-1.566413	0.468906
H	6.850200	-2.064339	0.123672
C	4.694380	-2.015517	0.024242
H	4.647192	-2.865459	-0.659374
C	5.189714	0.935702	-2.228479
H	5.255640	0.230392	-3.066117
H	5.822623	0.574419	-1.404041
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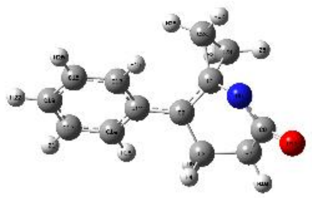

28

TS4

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C	-0.865081	-1.570706	0.272367
H	-0.621134	-2.155111	-0.634797
H	-0.430504	-2.153062	1.106487
N	-2.108387	0.686905	-0.729340
C	-2.369810	-1.419860	0.430347
C	-2.924011	-0.376710	-0.590343
H	-2.606205	-1.042591	1.439947
H	-2.896823	-2.370562	0.283843
O	-3.970789	-0.596663	-1.181729
C	1.285288	-0.212560	0.020672
C	1.875936	0.808821	-0.763260
C	2.130770	-1.201017	0.580552
C	3.252751	0.843773	-0.963017
H	1.233386	1.538553	-1.255457
C	3.508419	-1.145099	0.396635
H	1.702901	-1.996977	1.191471
C	4.075131	-0.123868	-0.375489
H	3.688756	1.626192	-1.588019
H	4.147572	-1.902670	0.855412
H	5.156545	-0.087730	-0.525351

C	-1.308698	1.512193	1.593118
C	-0.636391	2.291664	0.530589
H	-2.383307	1.639928	1.745014
H	-0.728858	1.169598	2.453492
H	-1.210848	3.053597	0.001421
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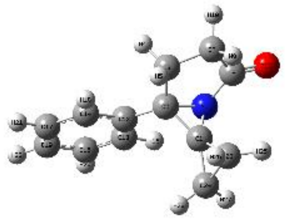

28

Z

C	1.078919	0.947556	-0.412332
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C	0.907547	-1.566563	-0.842135
H	0.298309	-2.425095	-0.524475
H	0.903738	-1.547686	-1.942372
N	1.190990	0.223223	0.845076
C	2.319527	-1.641843	-0.250001
C	2.379895	-0.580841	0.854195
H	3.109022	-1.388995	-0.975530
H	2.578687	-2.625506	0.167026
O	3.292329	-0.395763	1.609987
C	-1.172087	-0.207769	-0.025058
C	-1.692229	0.525726	1.051428
C	-2.057726	-0.835565	-0.912633
C	-3.071915	0.625935	1.235511
H	-0.996160	1.000070	1.745051
C	-3.439124	-0.730505	-0.729557
H	-1.669812	-1.409348	-1.757391
C	-3.950661	0.000262	0.345428
H	-3.464602	1.193932	2.082517
H	-4.117592	-1.224048	-1.429697
H	-5.030559	0.080041	0.491095
C	2.057201	1.563846	-1.335282
C	0.905422	2.375272	-0.725307
H	3.064770	1.760819	-0.955351
H	2.008965	1.308153	-2.398678
H	1.159809	3.099450	0.053590
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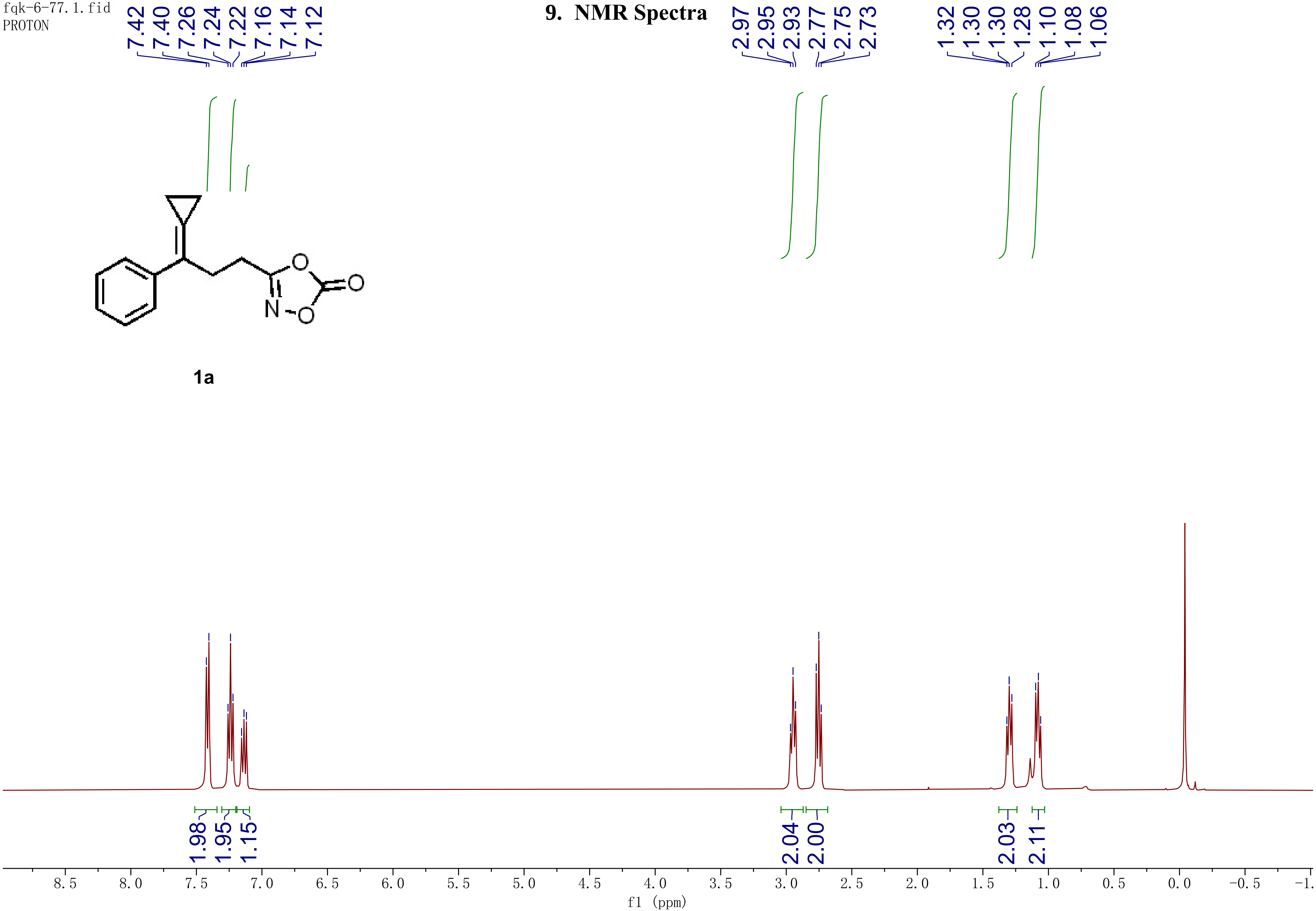
9. NMR Spectra



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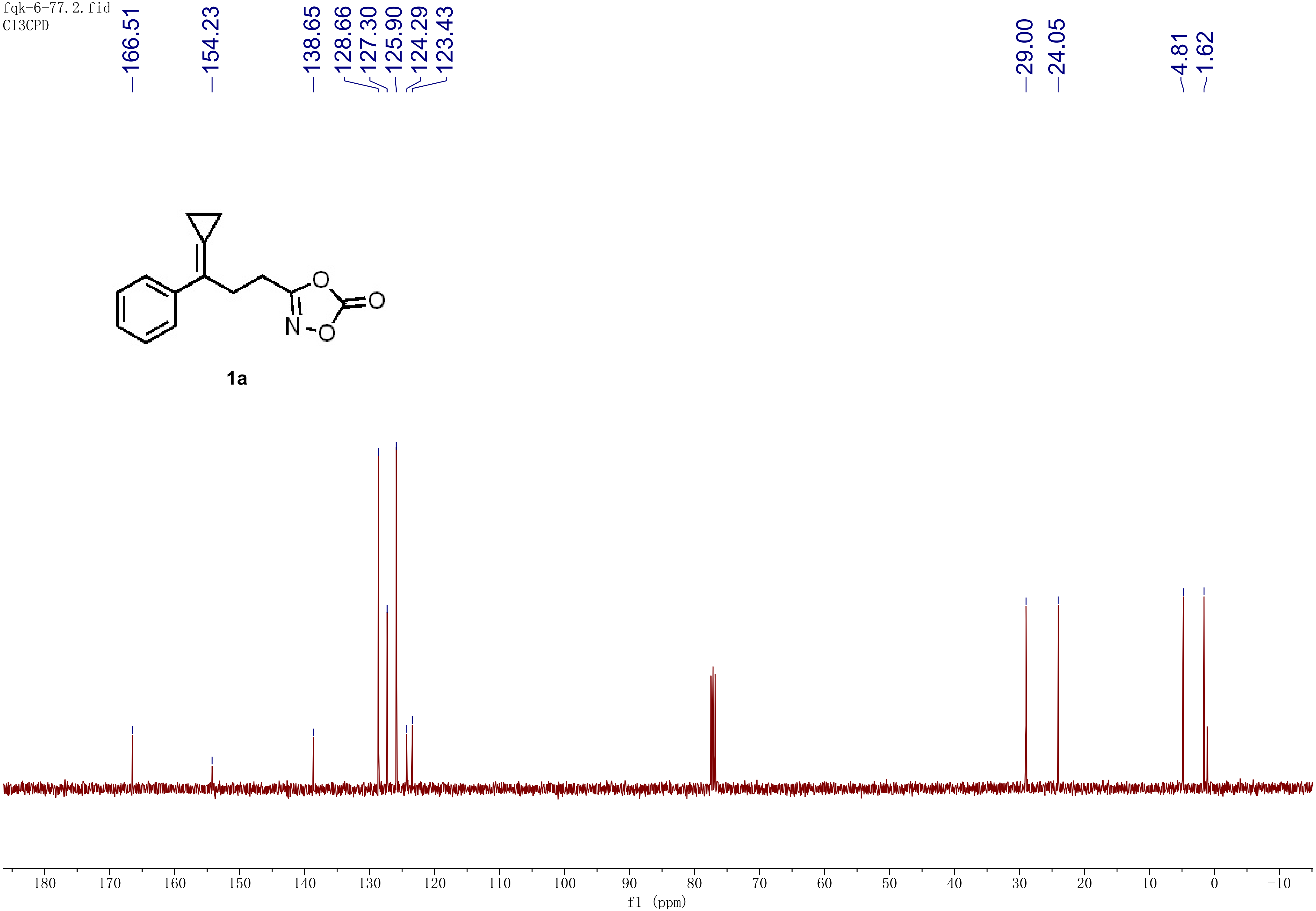

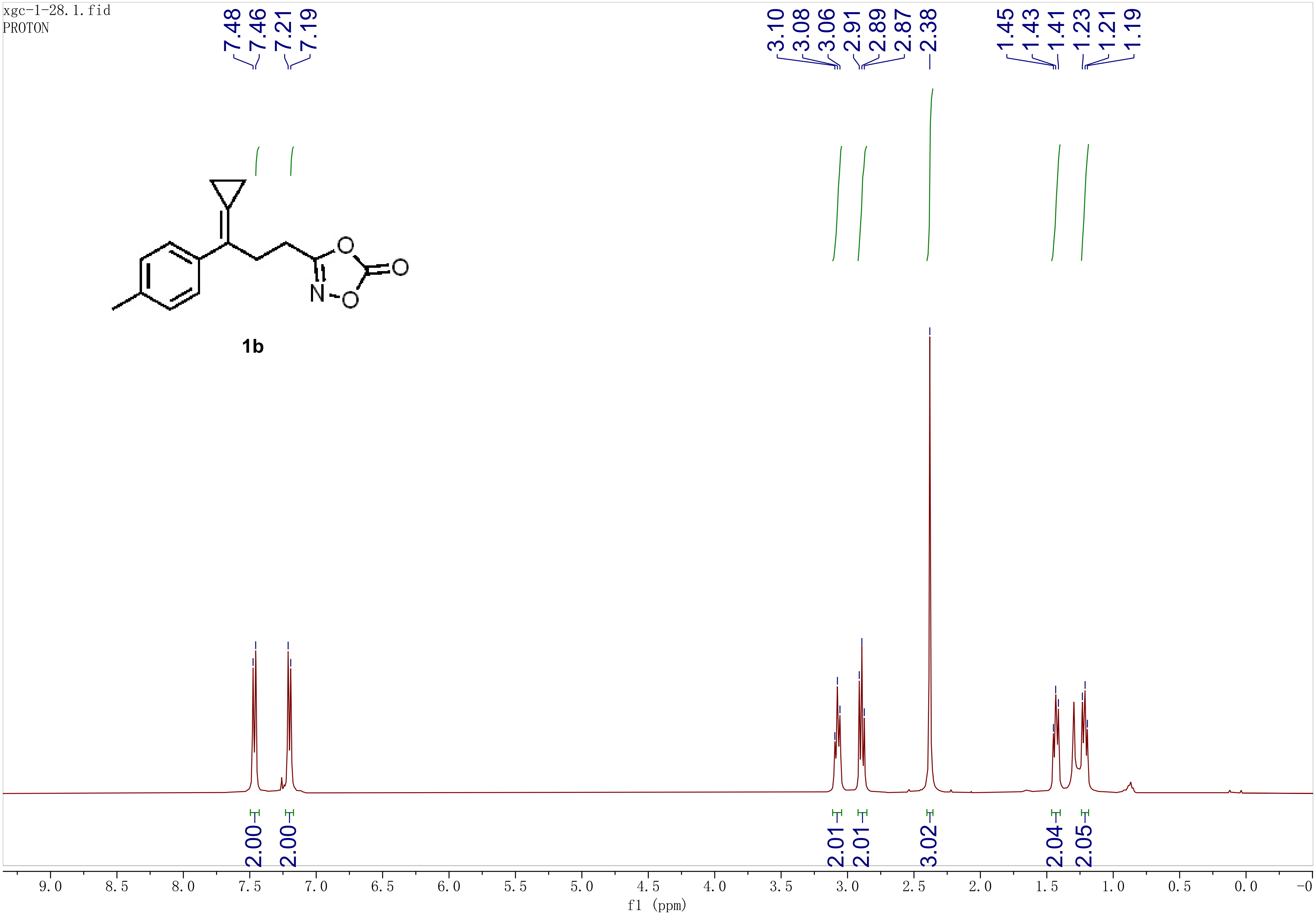
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3.06
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2.89
2.87
2.38
1.45
1.43
1.41
1.23
1.21
1.19
1b
2.00
2.00
2.01
2.01
3.02
2.04
2.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

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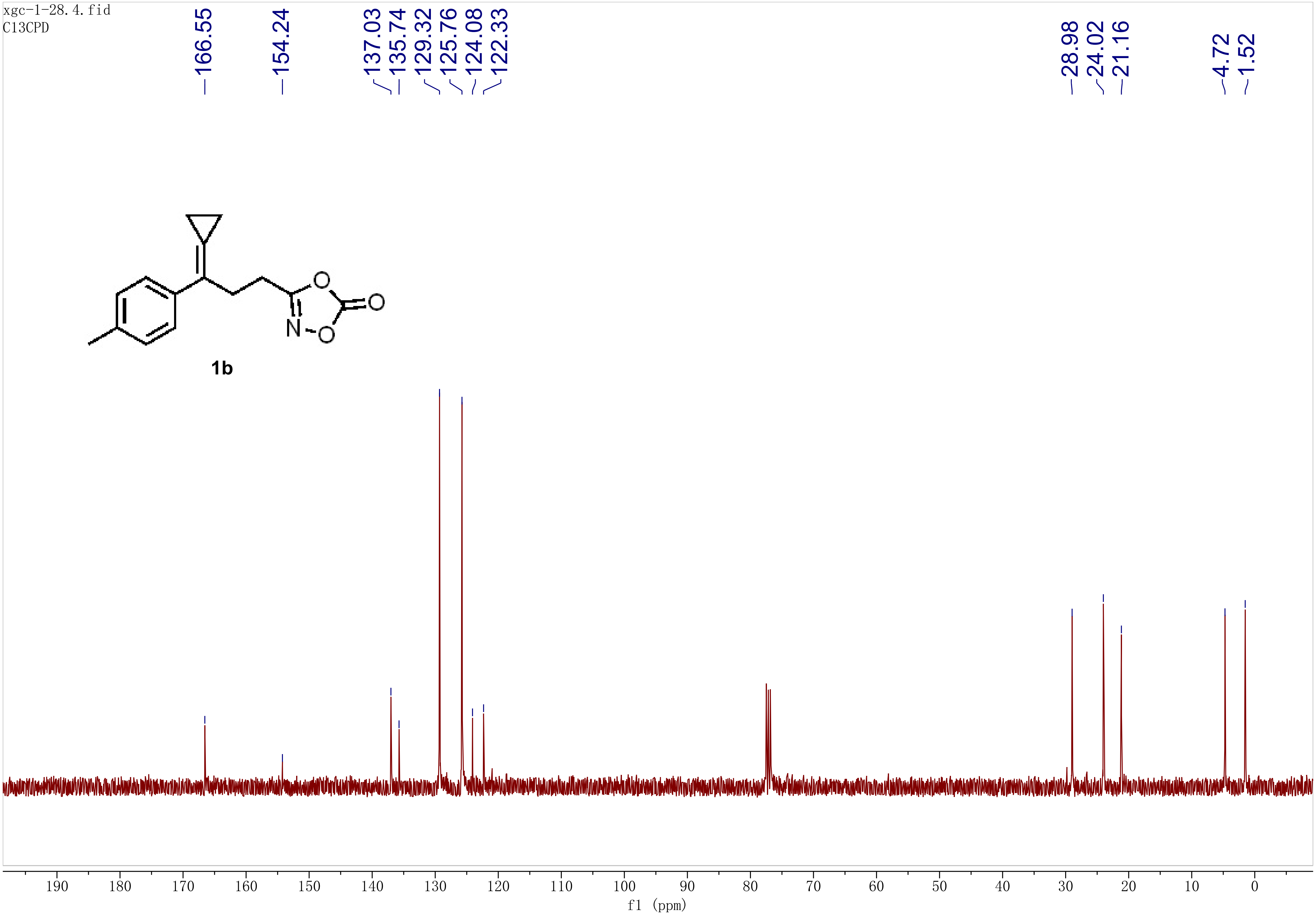
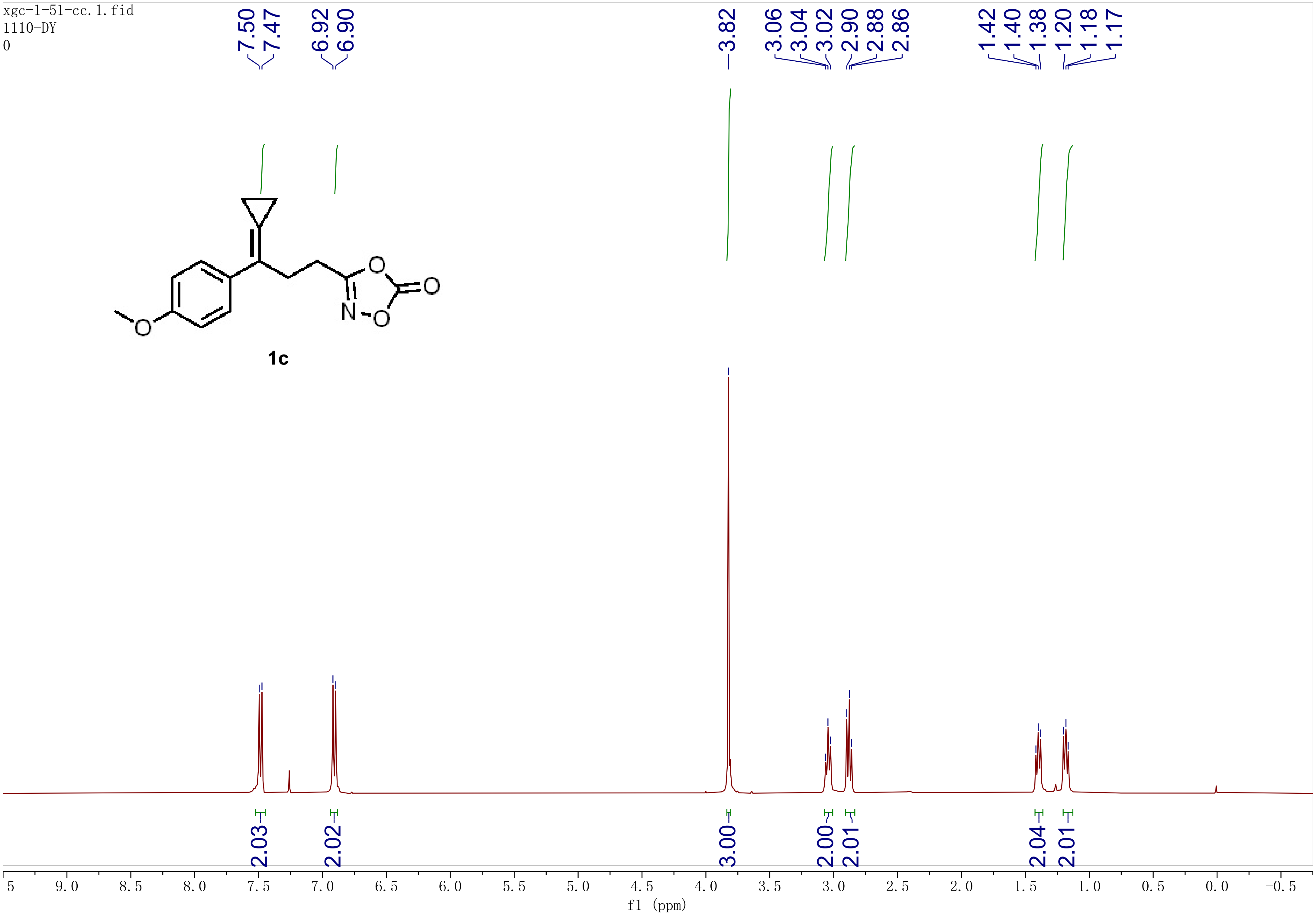


1b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3.06
3.04
3.02
2.90
2.88
2.86
1.42
1.40
1.38
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1.17
1c
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2.02
3.00
2.00
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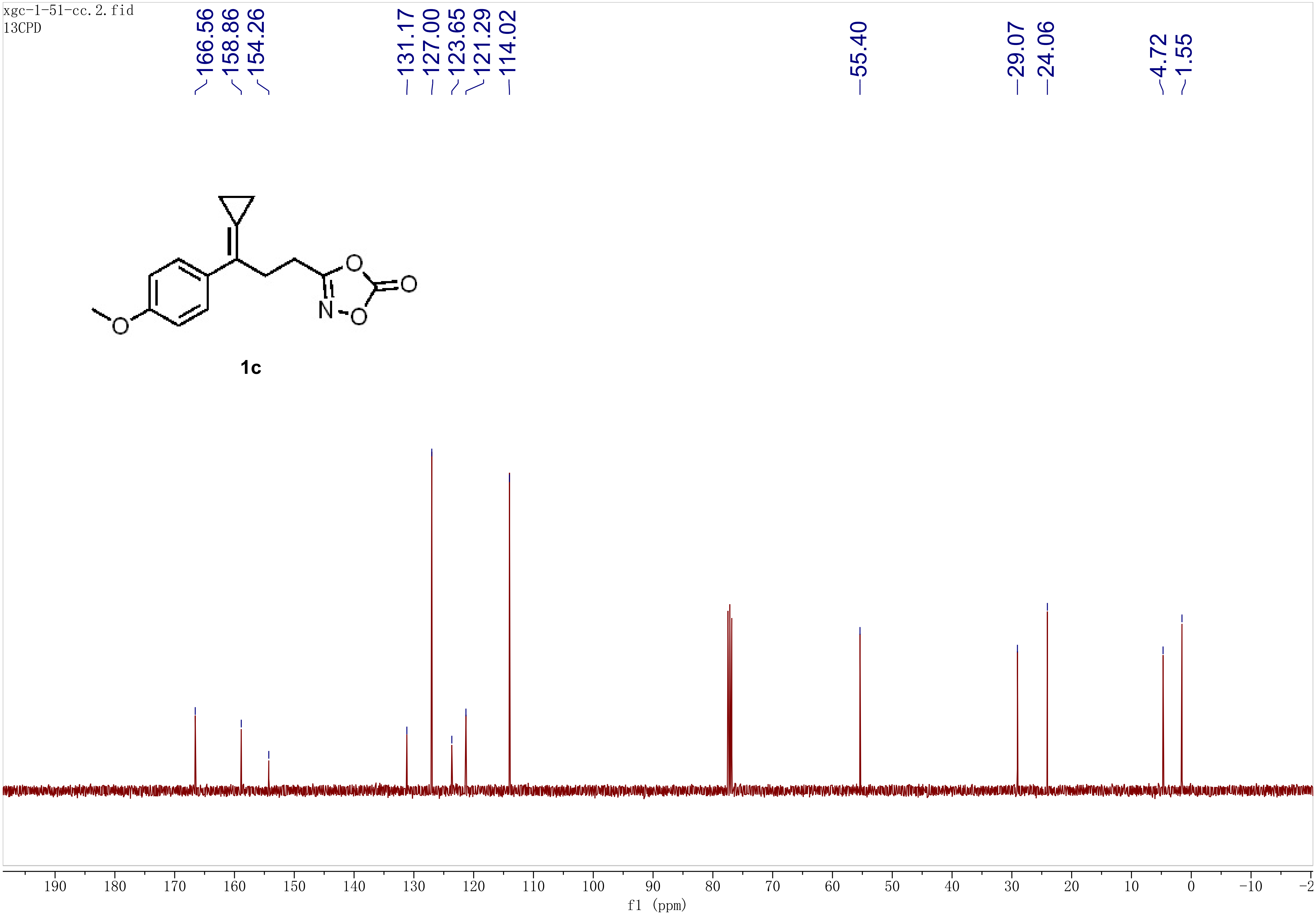
2.04
2.01
f1 (ppm)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29.07
24.06
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1.55
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O
O
O
O
1c
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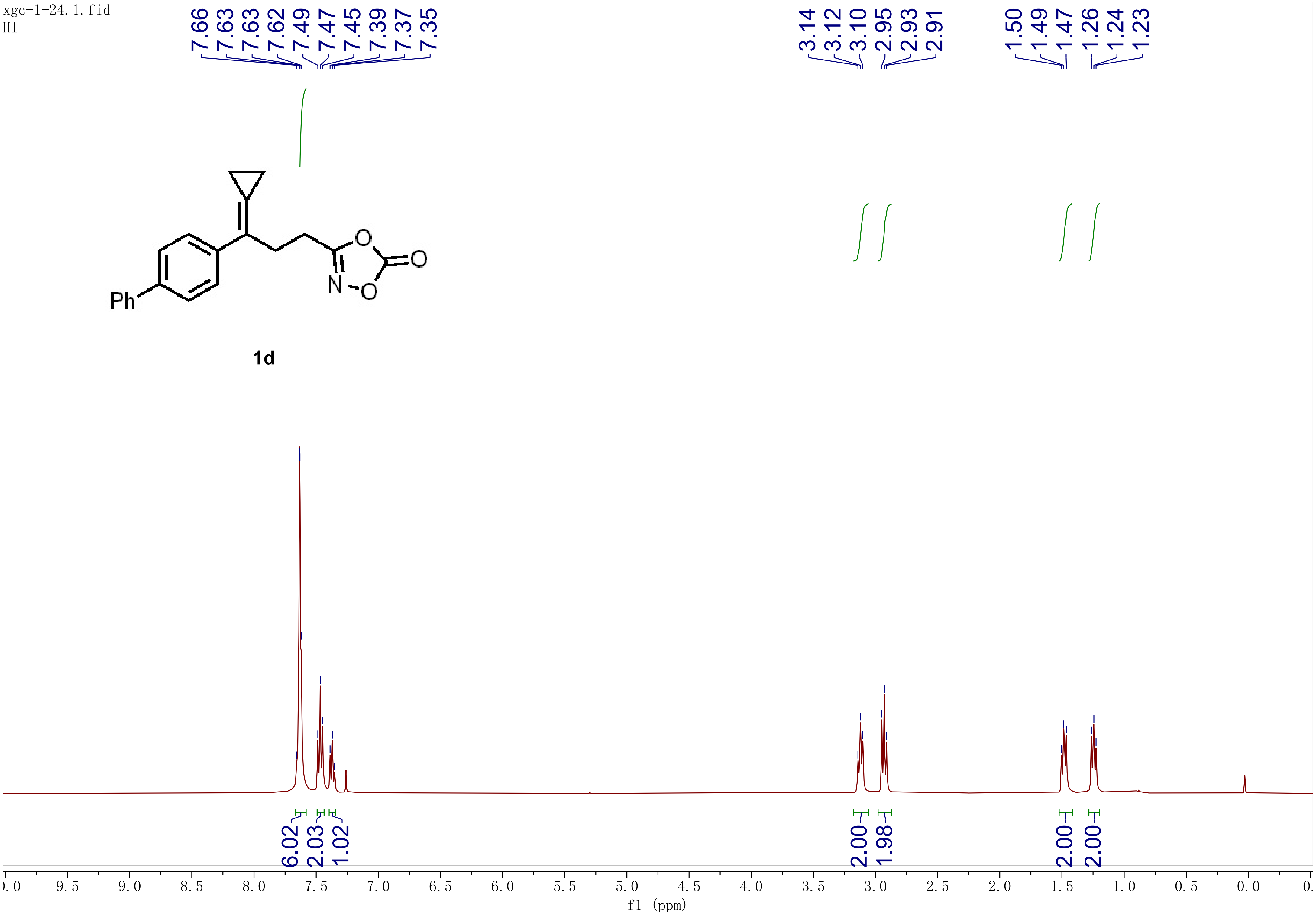
f1 (ppm)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1.50 1.49 1.47 1.26 1.24 1.23
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O
O
O
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1.98
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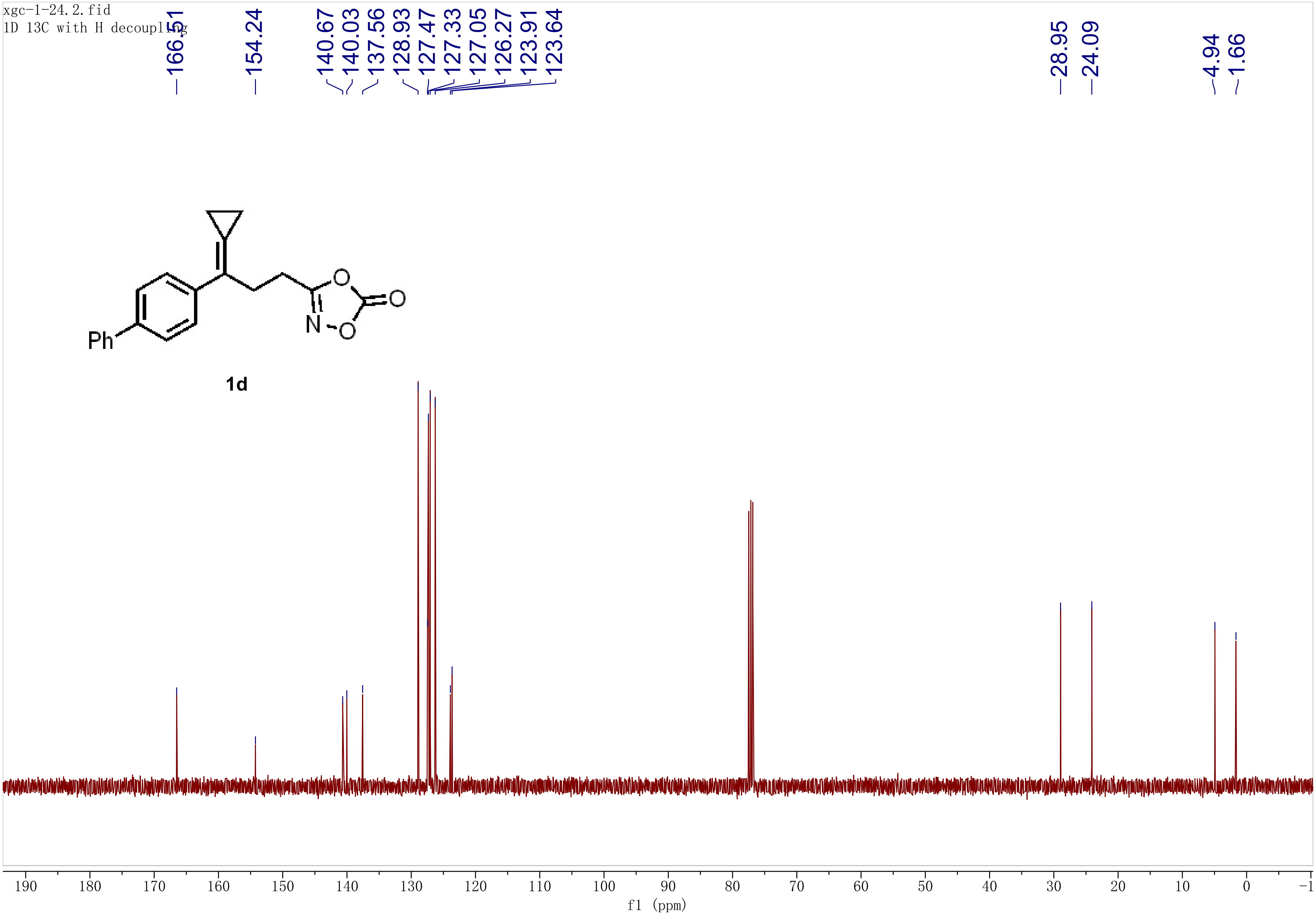
2.00
f1 (ppm)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127.05
126.27
123.91
123.64
28.95
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4.94
1.66
Ph
N
O
1d
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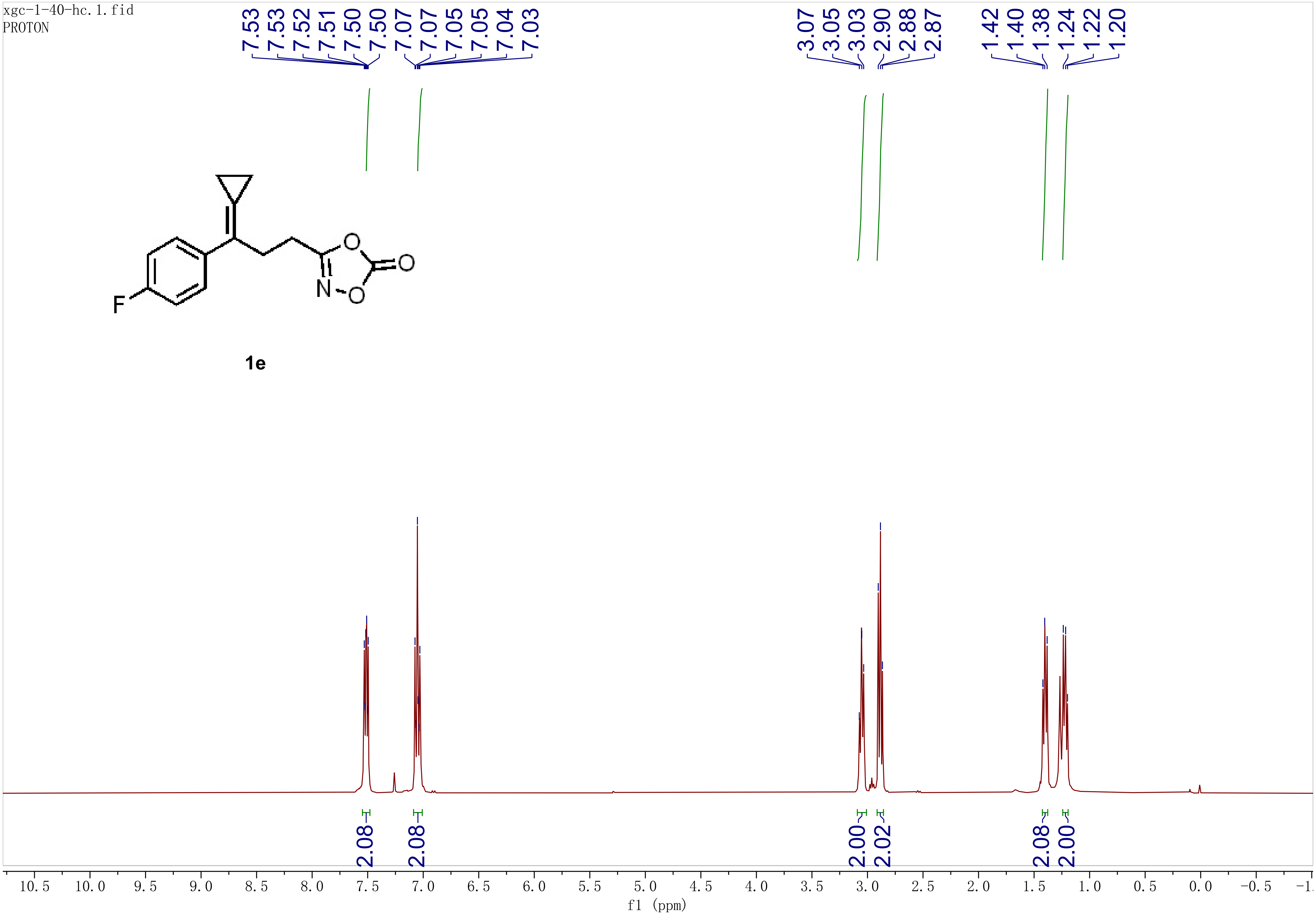
f1 (ppm)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1.42 1.40 1.38 1.24 1.22 1.20
F
O
O
N
O
1e
2.08 2.08 2.00 2.02 2.08 2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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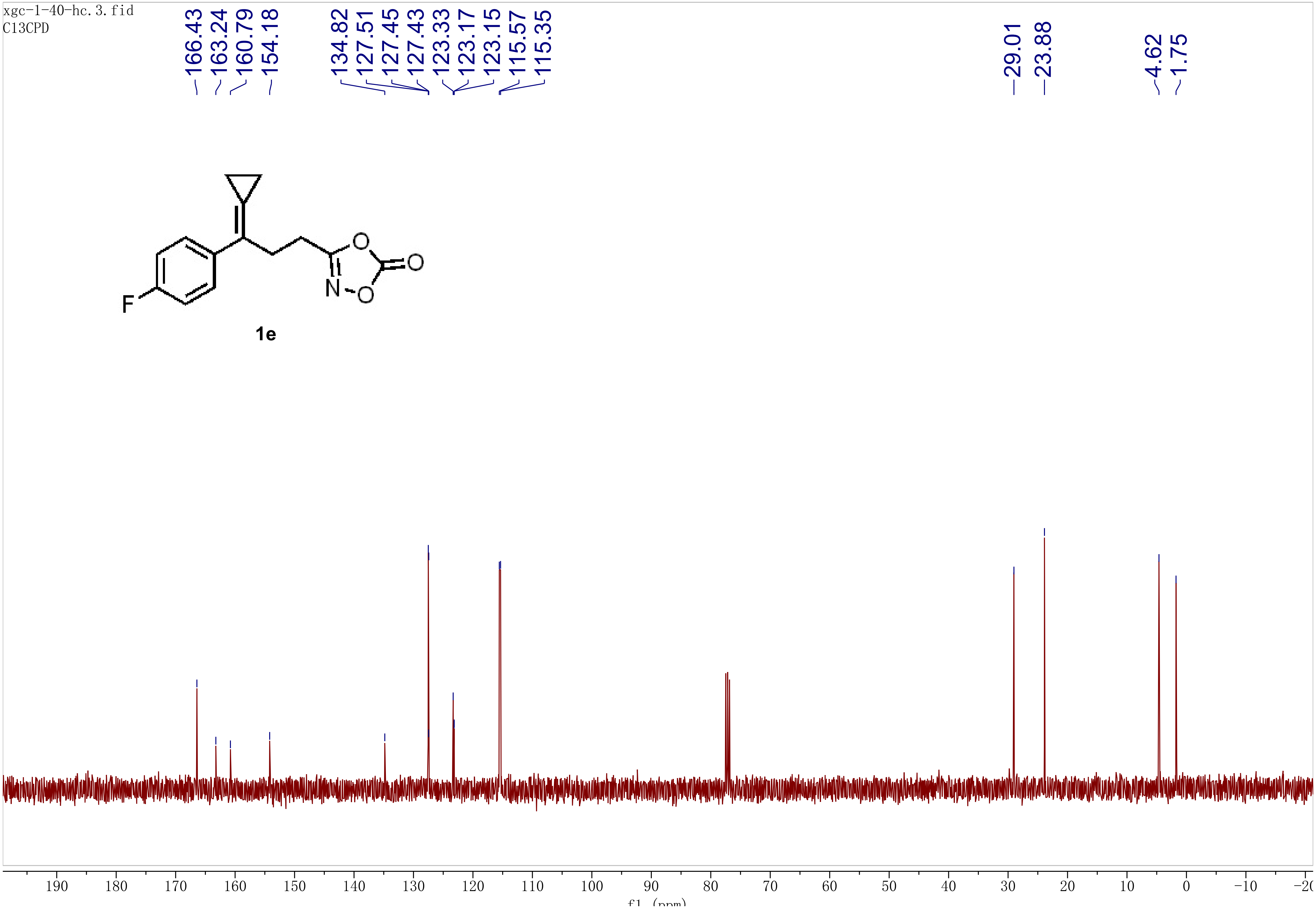
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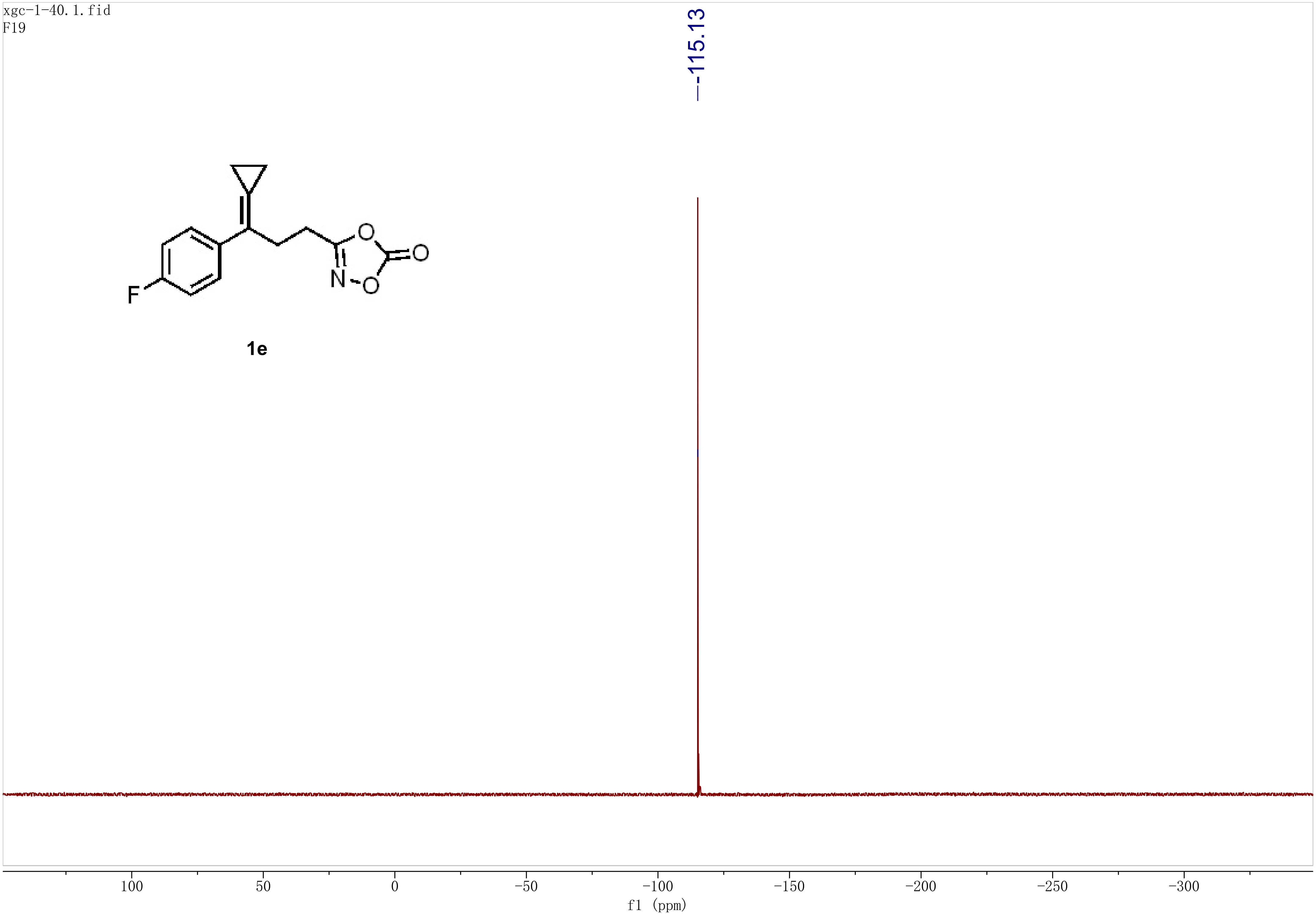
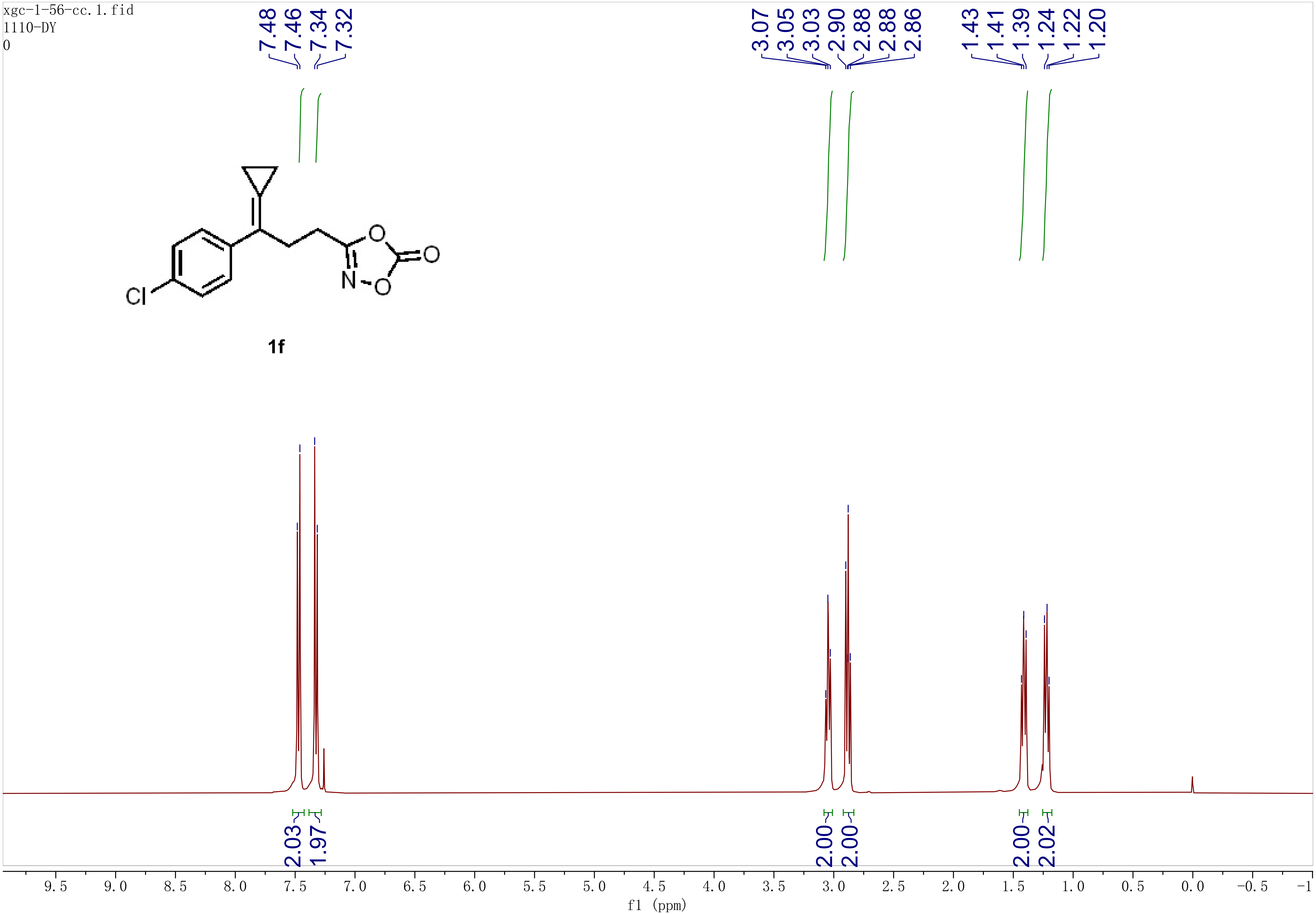
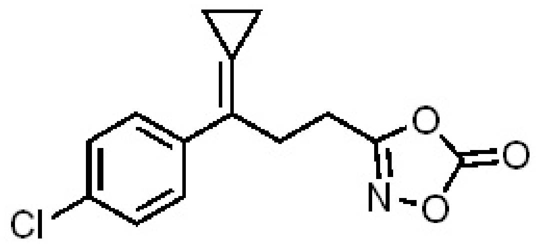
115.57
115.35
29.01
23.88
4.62
1.75
F
O
O
O
N
1e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

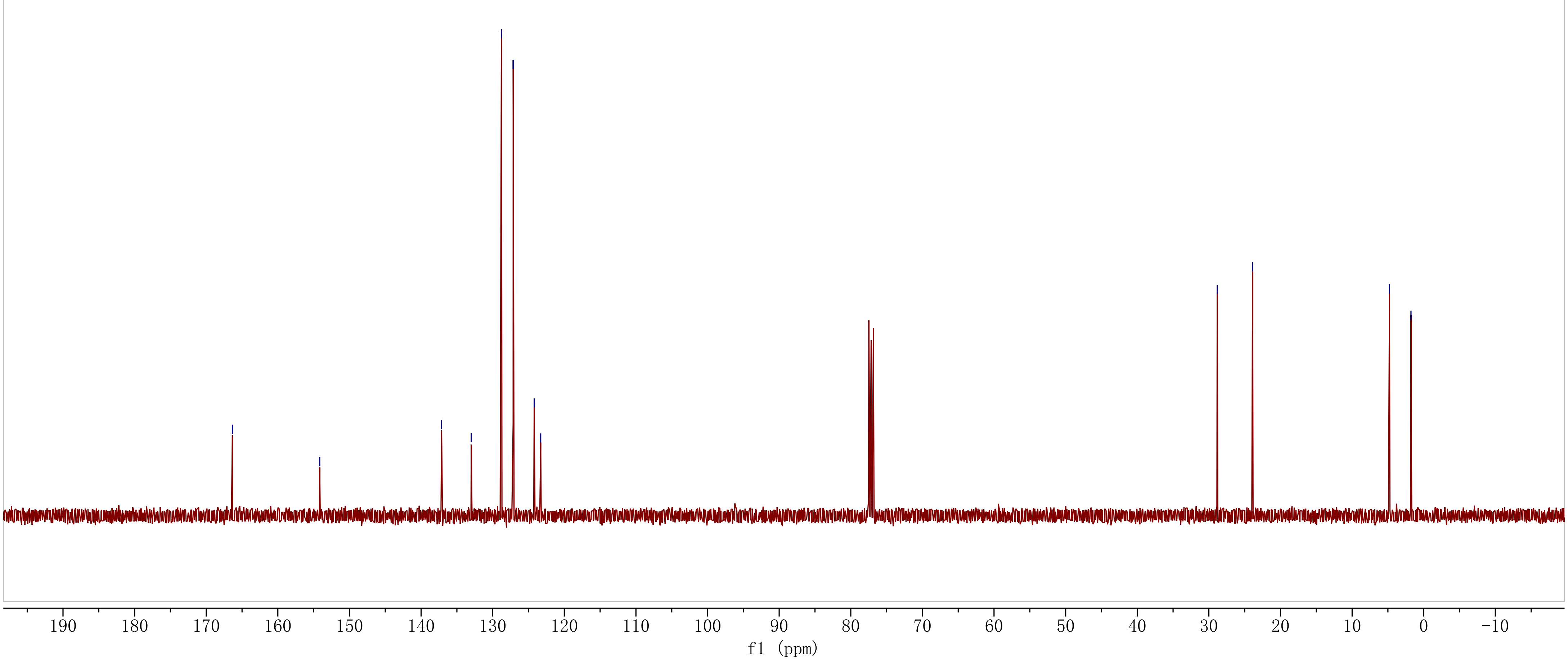

xgc-1-40.1.fid
F19
-115.13
F
N
O
O
O
1e
100
50
0
−50
−100
−150
−200
−250
−300
f1 (ppm)

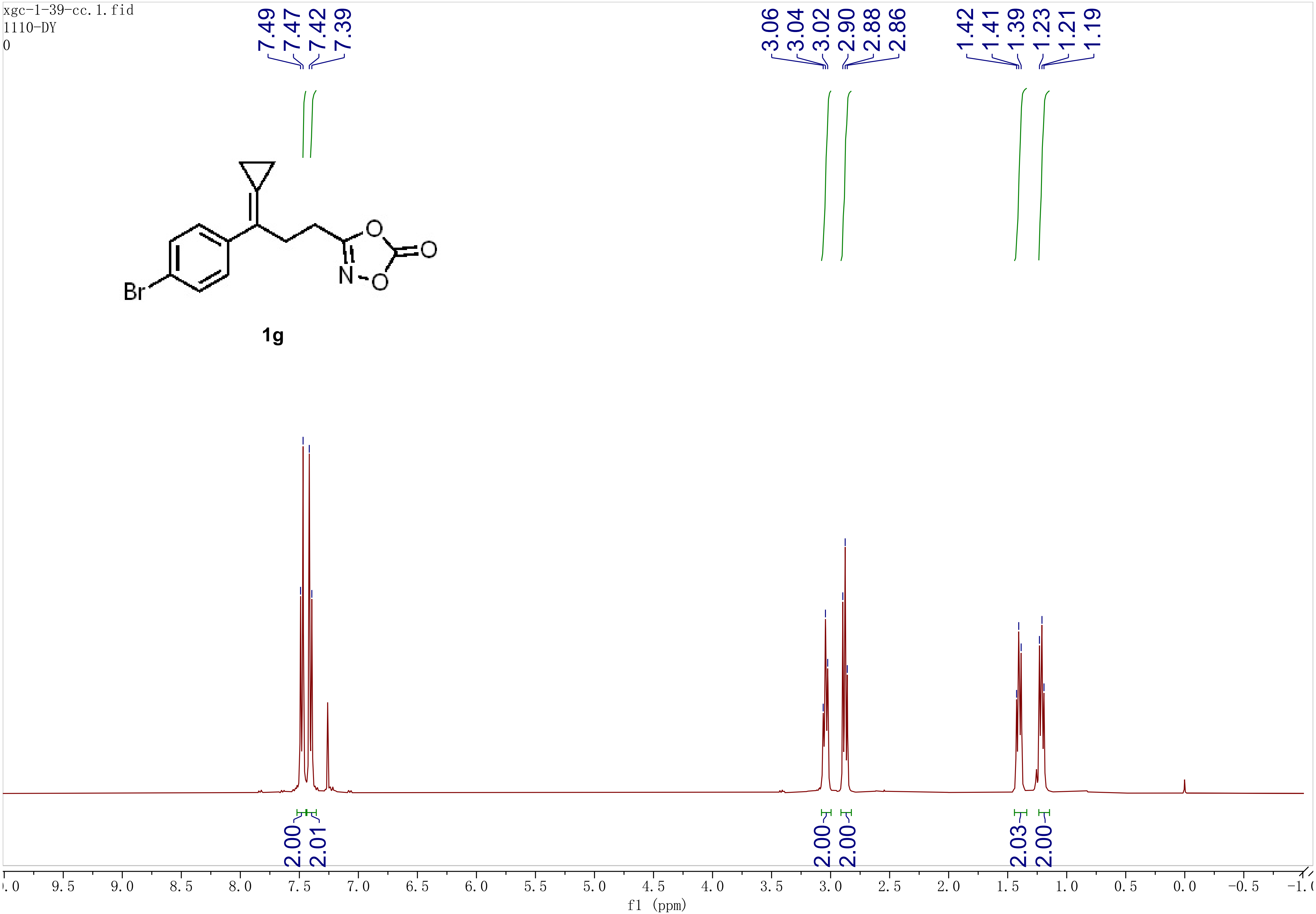

xgc-1-56-cc.1.fid
1110-DY
0
7.48
7.46
7.34
7.32
3.07
3.05
3.03
2.90
2.88
2.88
2.86
1.43
1.41
1.39
1.24
1.22
1.20
Cl
N
O
O
O
1f
2.03
1.97
2.00
2.00
2.00
2.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

xgc-1-56-cc.2.fid
13CPD

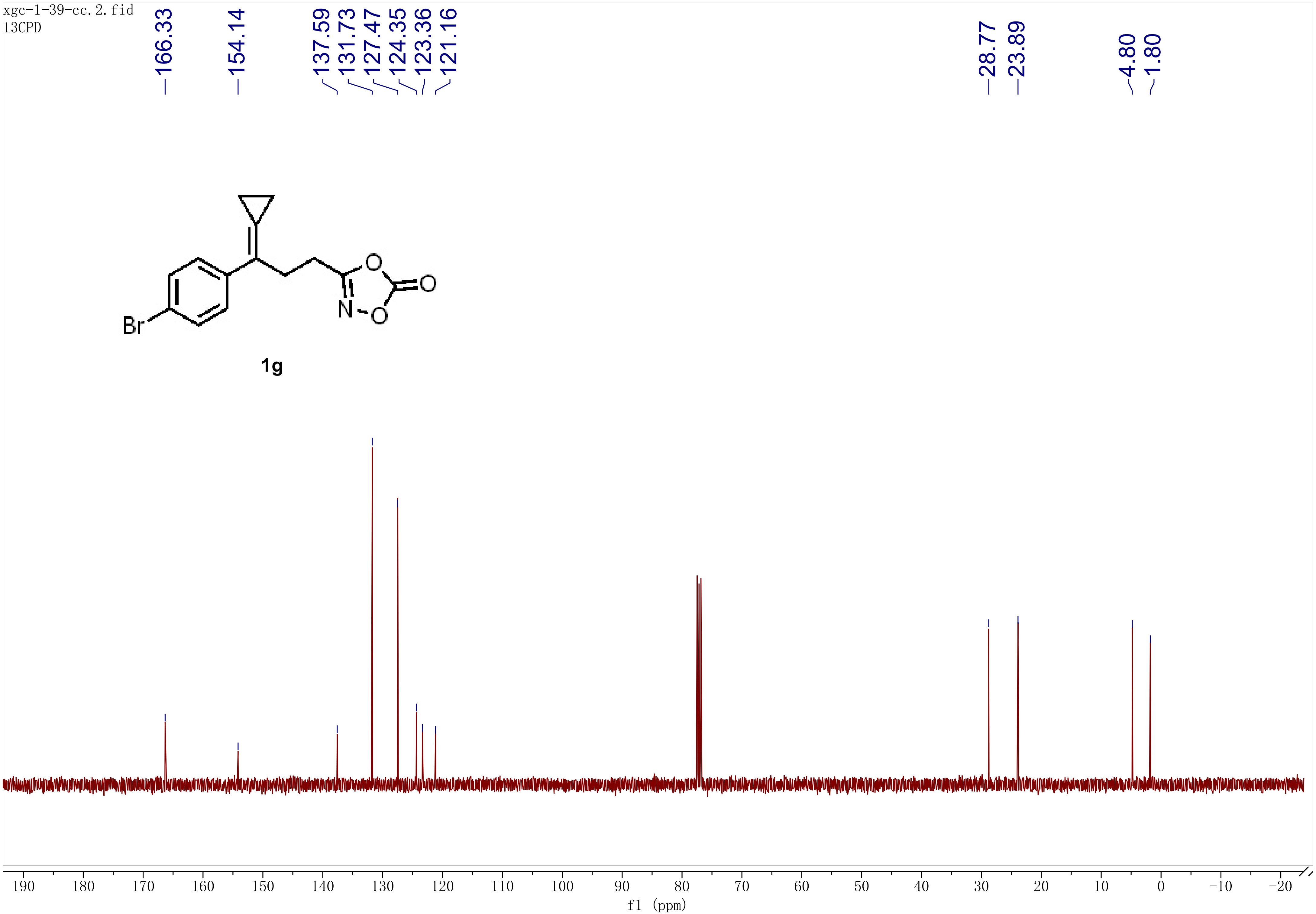


1f

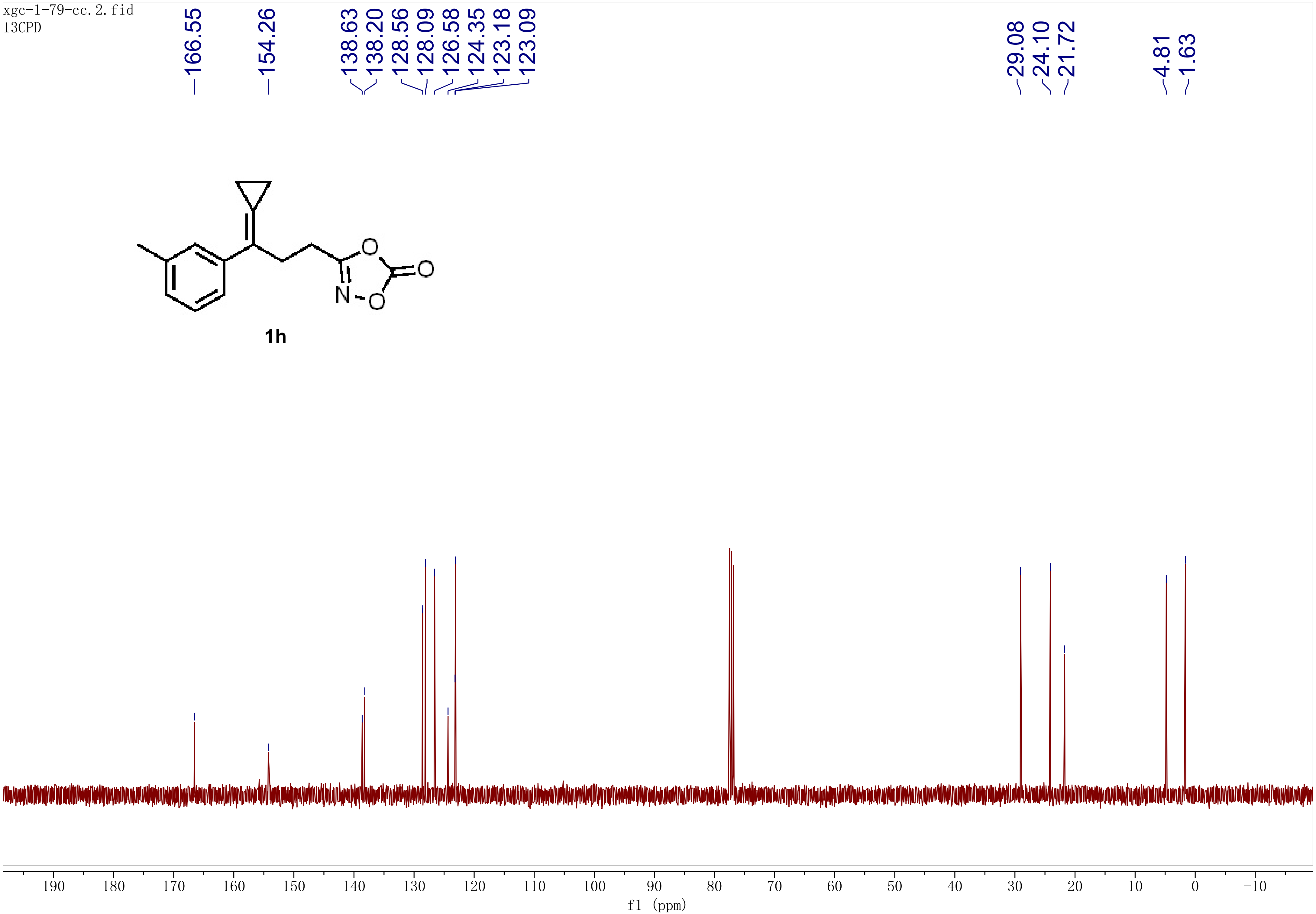


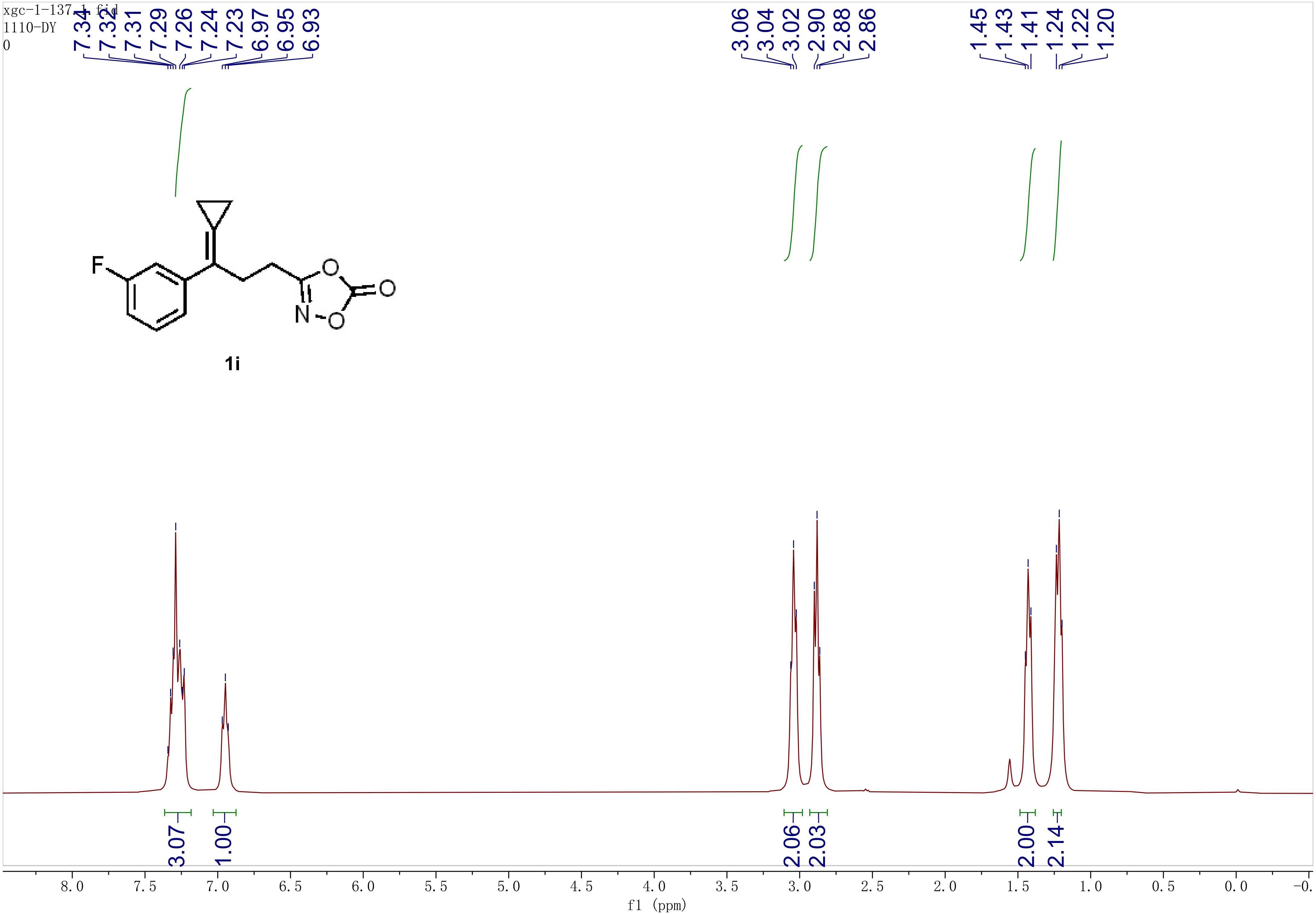


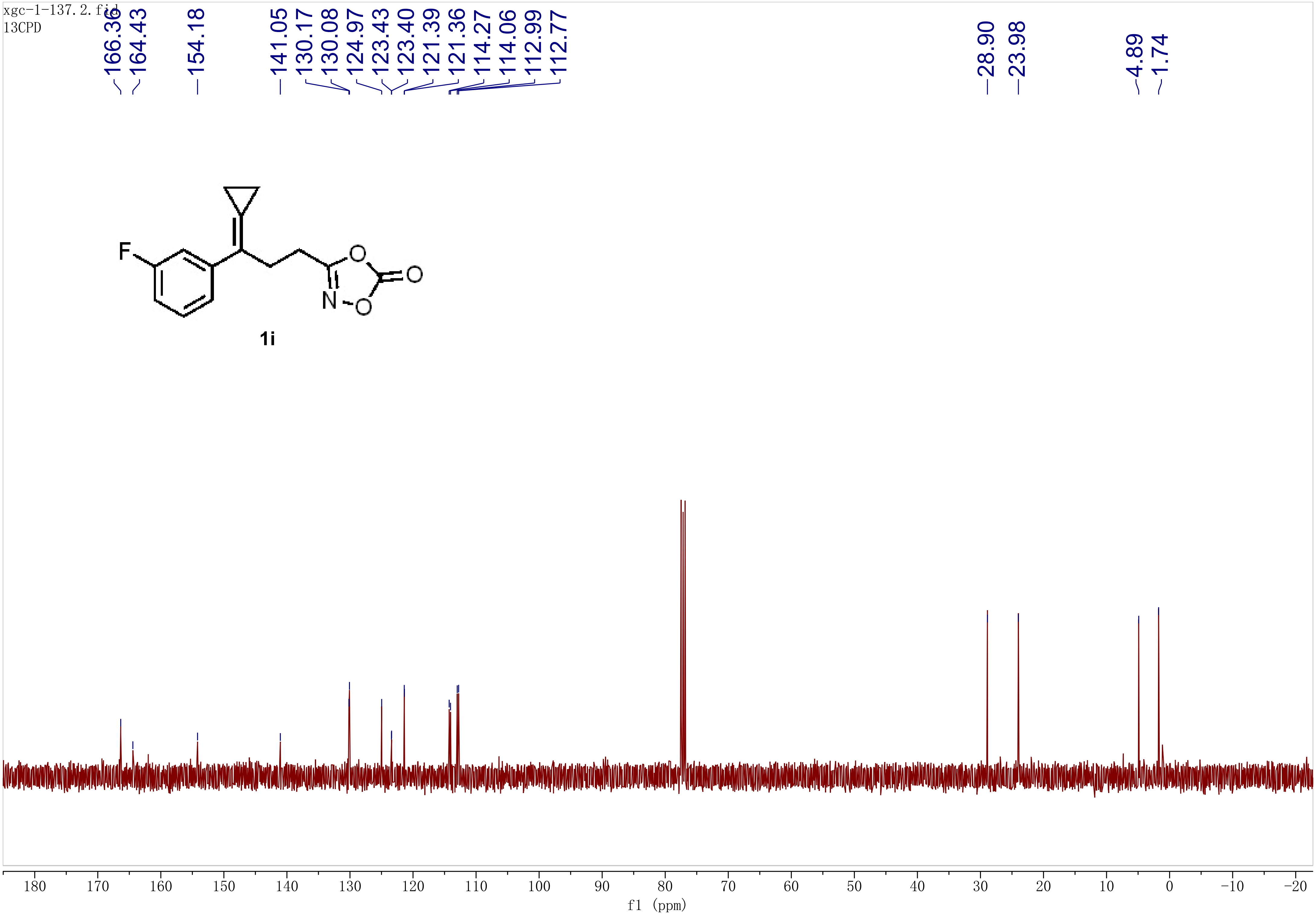
xgc-1-39-cc.1.fid
1110-DY
0
7.49
7.47
7.42
7.39
3.06
3.04
3.02
2.90
2.88
2.86
1.42
1.41
1.39
1.23
1.21
1.19
Br
O
N
1g
2.00
2.01
2.00
2.00
2.03
2.00
f1 (ppm)


xgc-1-39-cc.2.fid
13CPD
166.33
154.14
137.59
131.73
127.47
124.35
123.36
121.16
28.77
23.89
4.80
1.80
Br
O
N
O
O
1g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

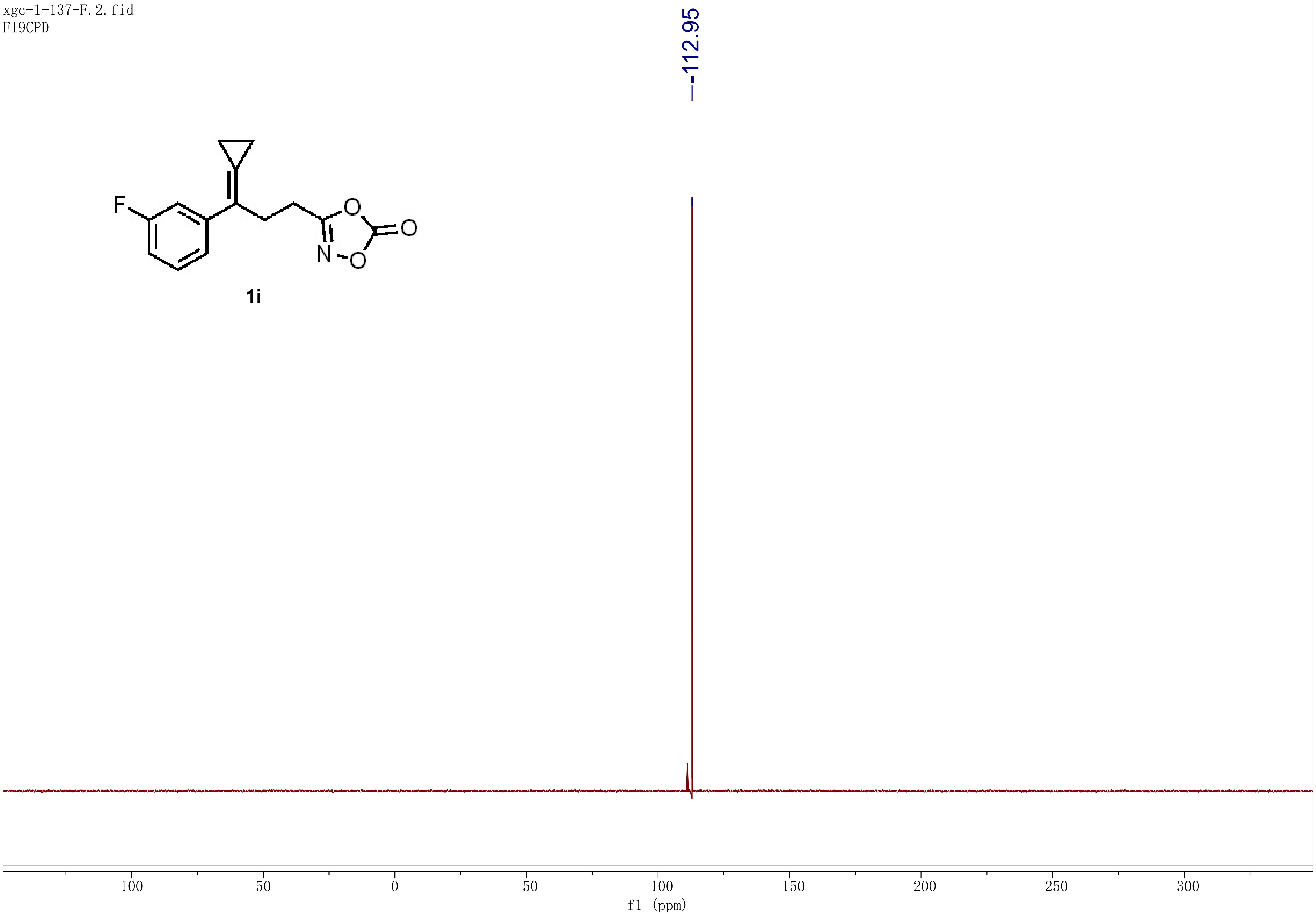

xgc-1-79-cc.1.fid
1110-DY
0
7.33 7.32 7.32 7.31 7.24 7.23 7.22 7.08 7.06
3.07 3.05 3.03 2.88 2.86 2.84 2.36
1.42 1.40 1.38 1.20 1.18 1.16
1h
2.03 0.99 0.97
2.00 2.04
3.00
2.01 2.03
f1 (ppm)

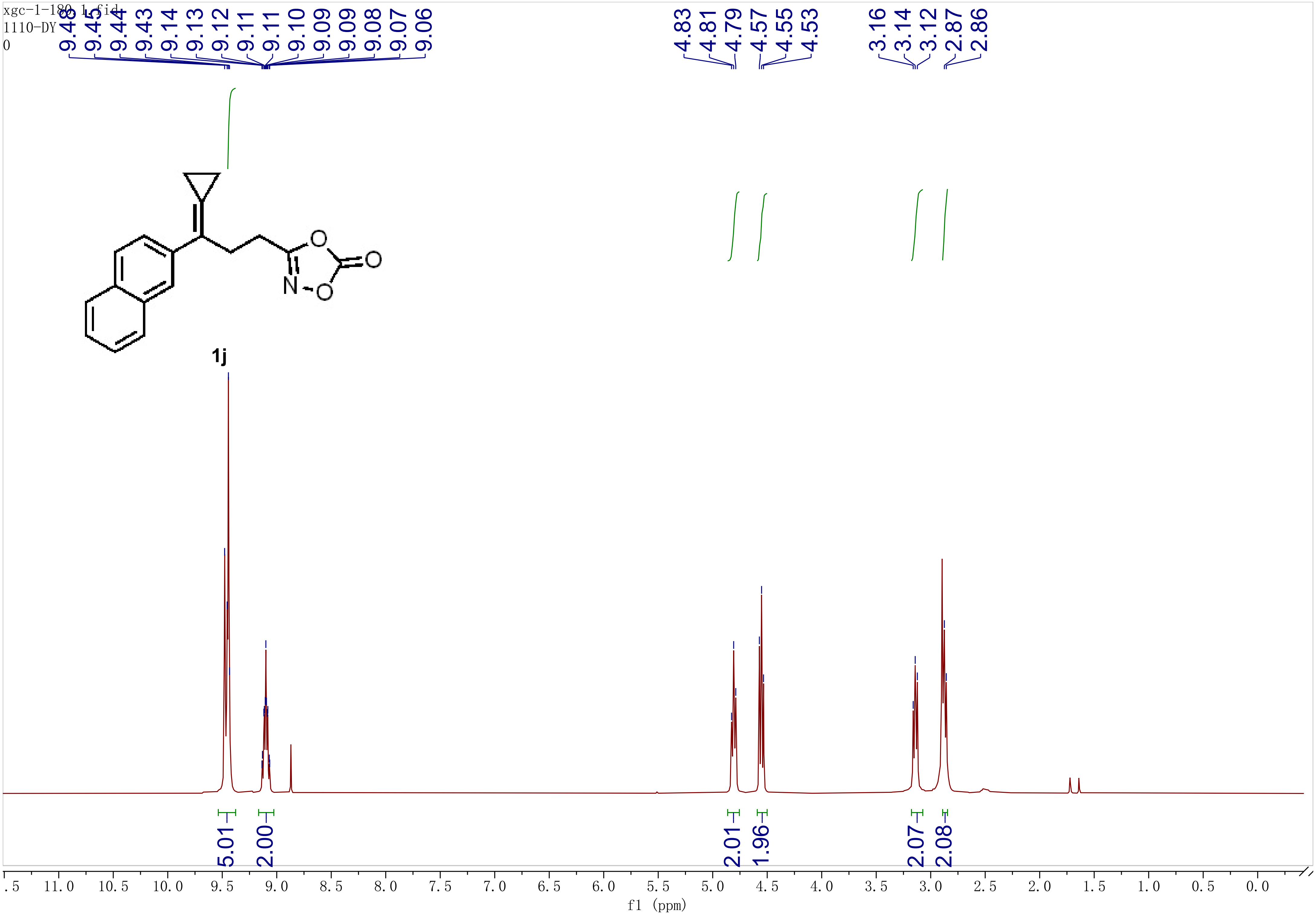

xgc-1-79-cc.2.fid
13CPD
166.55
154.26
138.63
138.20
128.56
128.09
126.58
124.35
123.18
123.09
29.08
24.10
21.72
4.81
1.63
O
O
O
N
1h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


xgc-1-137.1.fid
1110-DY
0
7.34 7.32 7.31 7.29 7.26 7.24 7.23 6.97 6.95 6.93
3.06 3.04 3.02 2.90 2.88 2.86
1.45 1.43 1.41 1.24 1.22 1.20
F
O
O
N
O
1i
3.07
1.00
2.06
2.03
2.00
2.14
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

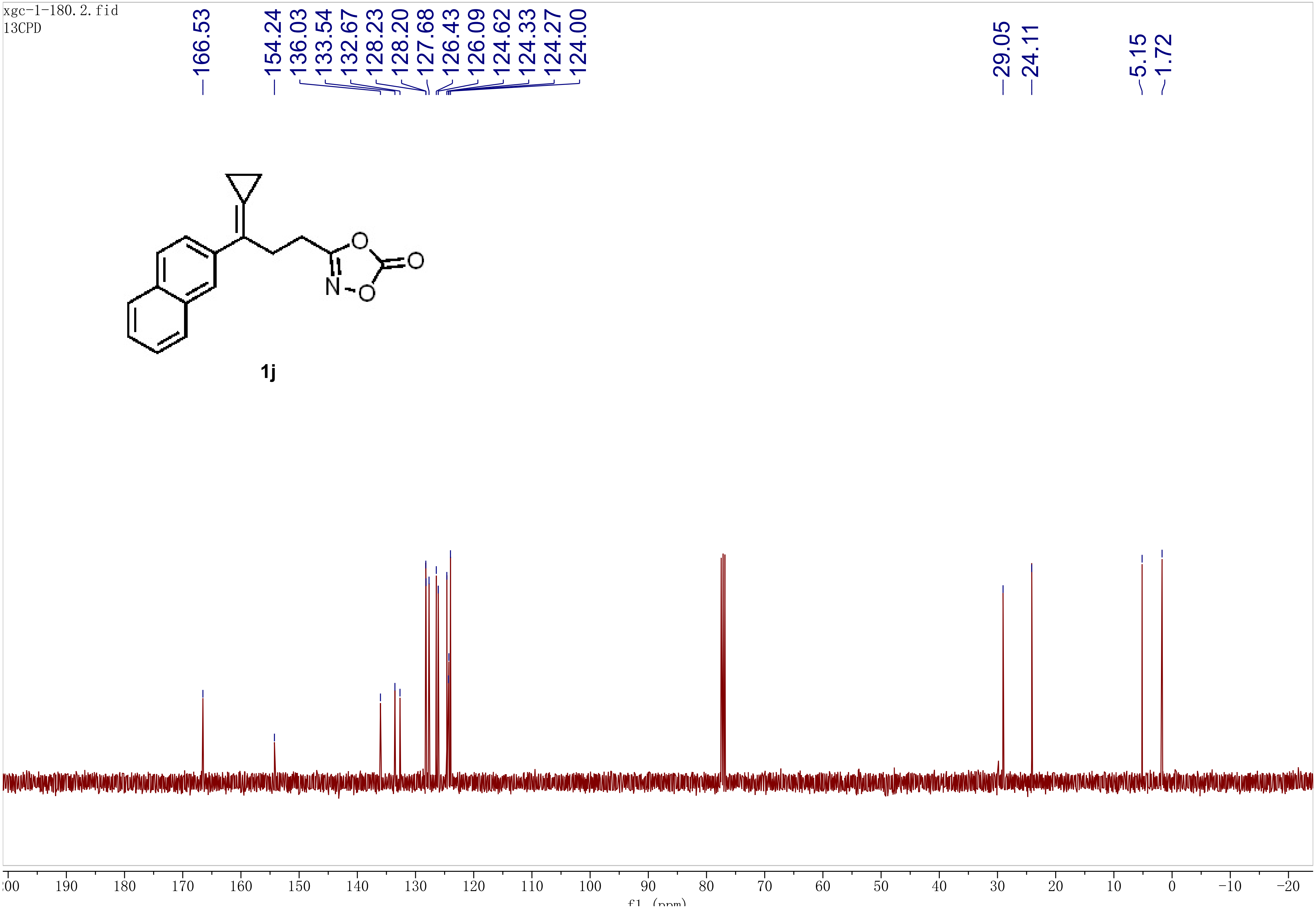

xgc-1-137.2.fid
13CPD
166.36
164.43
154.18
141.05
130.17
130.08
124.97
123.43
123.40
121.39
121.36
114.27
114.06
112.99
112.77
28.90
23.98
4.89
1.74
F
O
O
N
O
1i
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

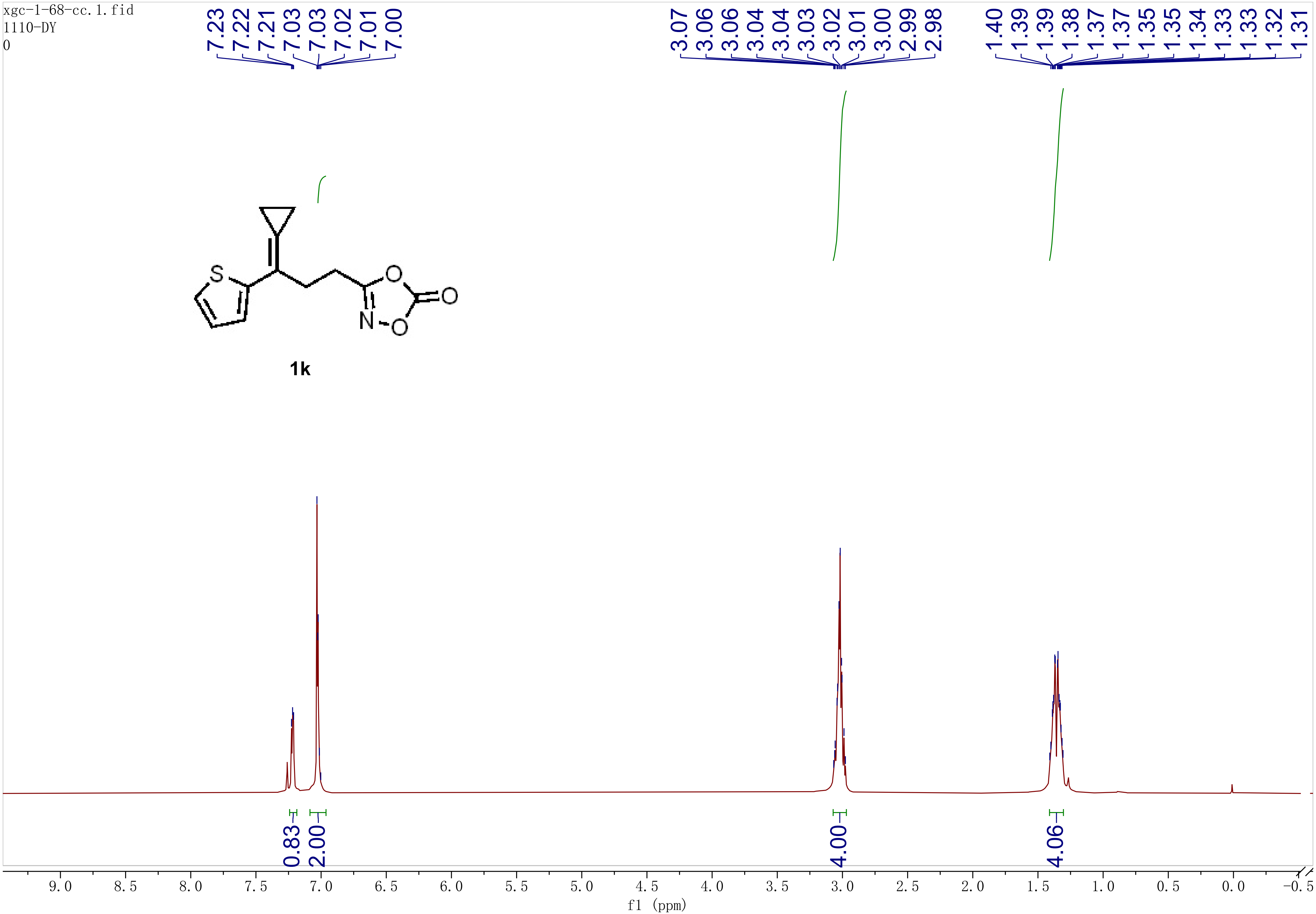
xgc-1-137-F.2.fid
F19CPD

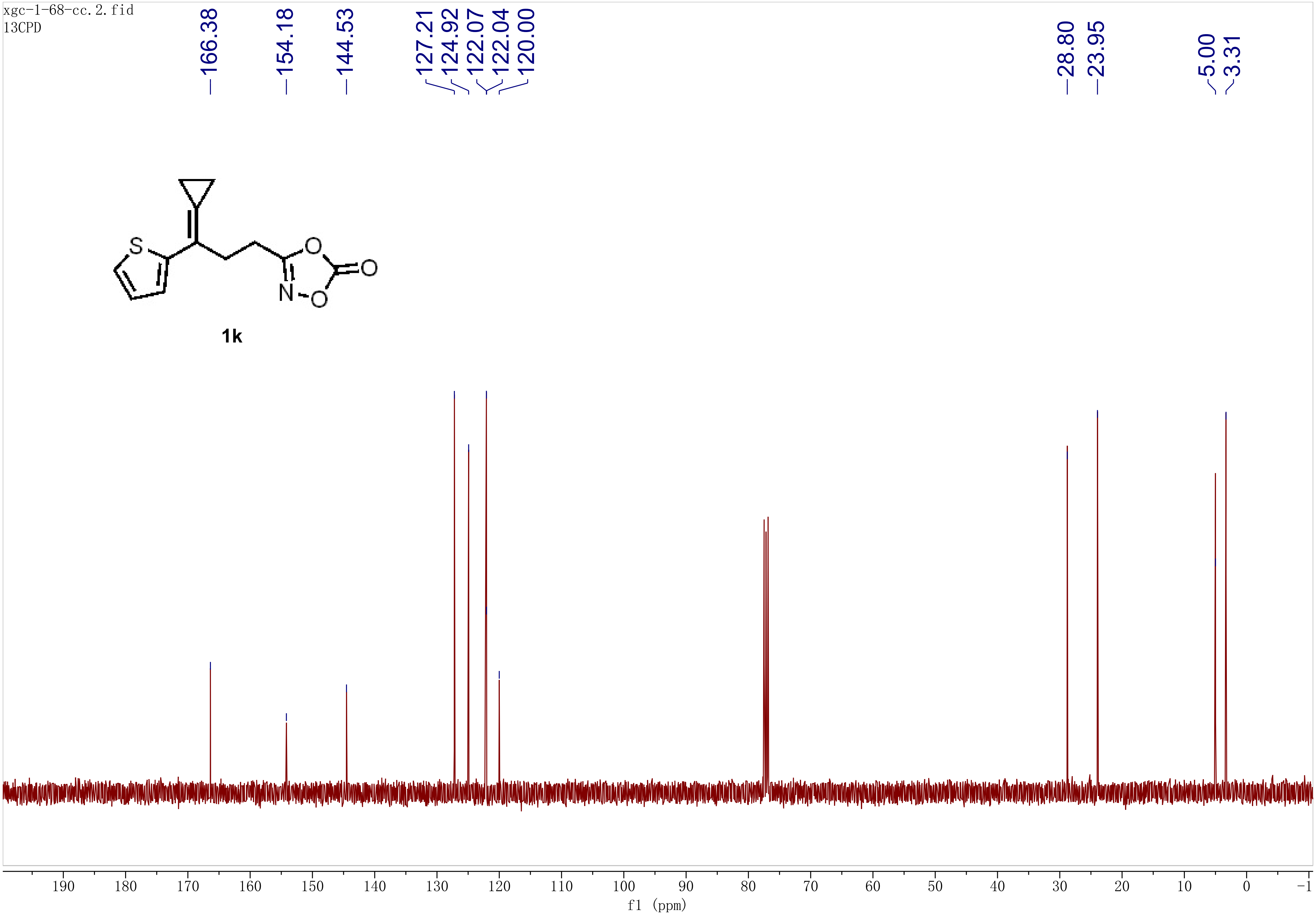


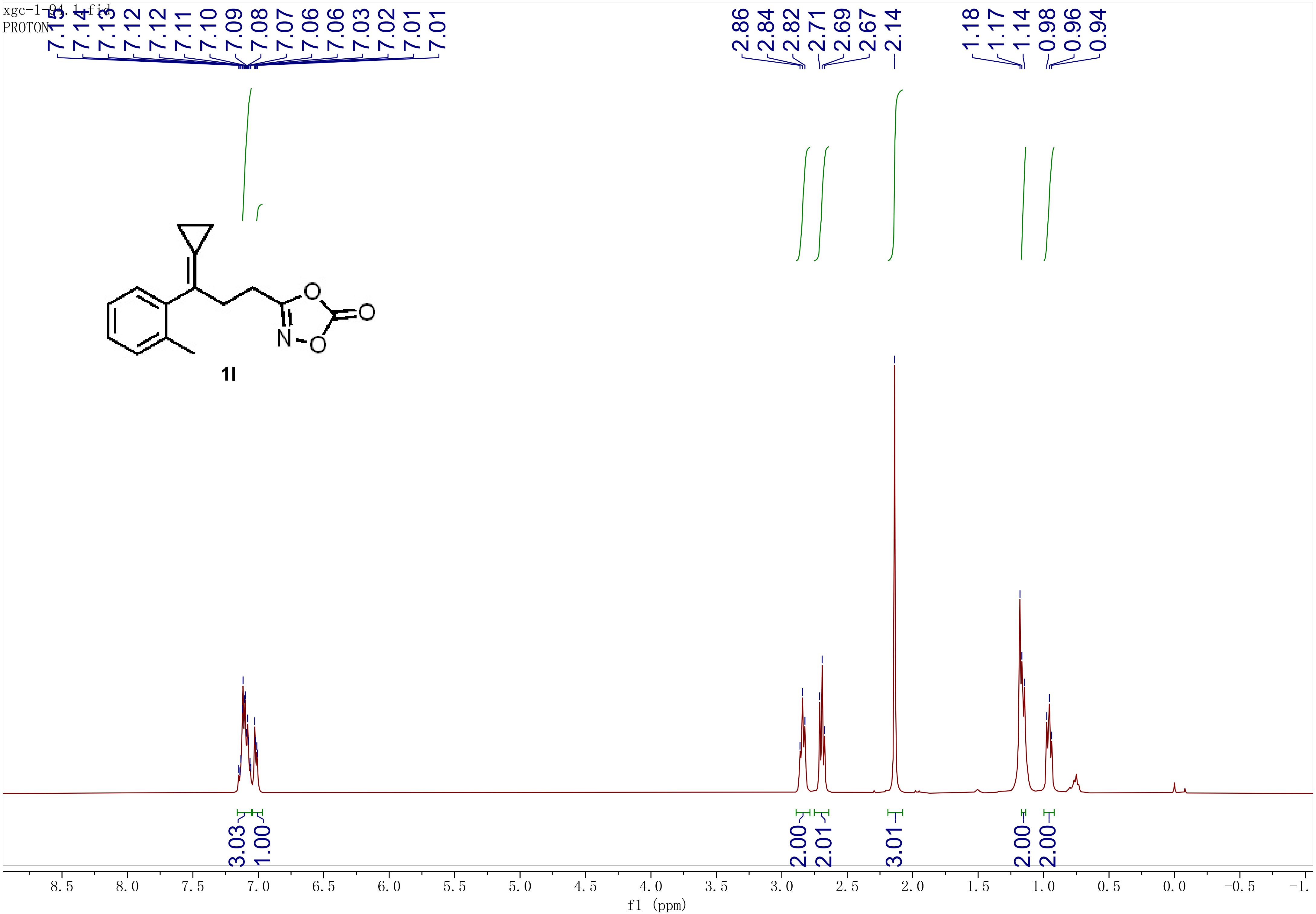


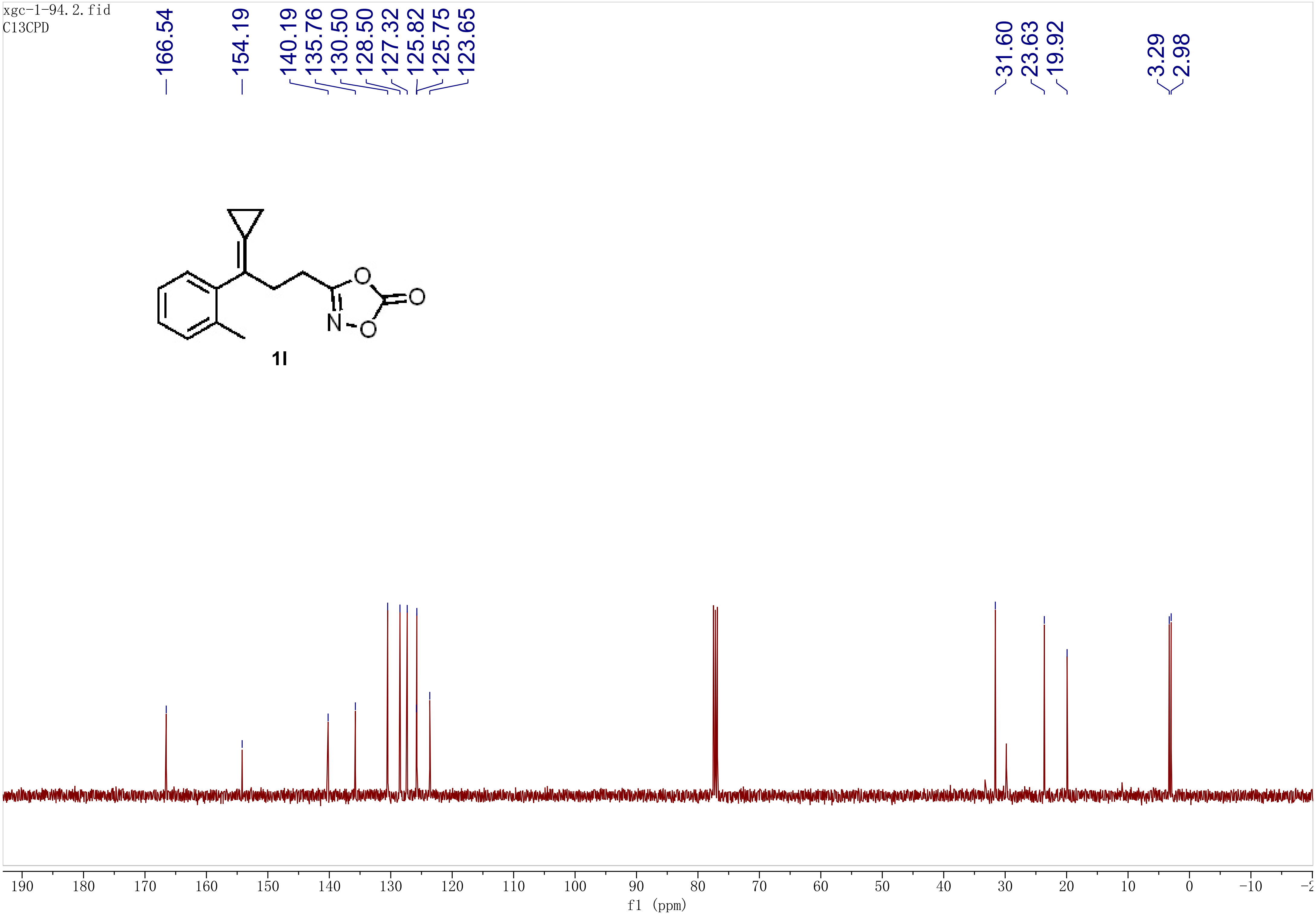
xgc-1-180.1.fid
1110-DY
0
9.48
9.45
9.44
9.43
9.14
9.13
9.12
9.11
9.11
9.10
9.09
9.09
9.08
9.07
9.06
4.83
4.81
4.79
4.57
4.55
4.53
3.16
3.14
3.12
2.87
2.86
1j
5.01
2.00
2.01
1.96
2.07
2.08
.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

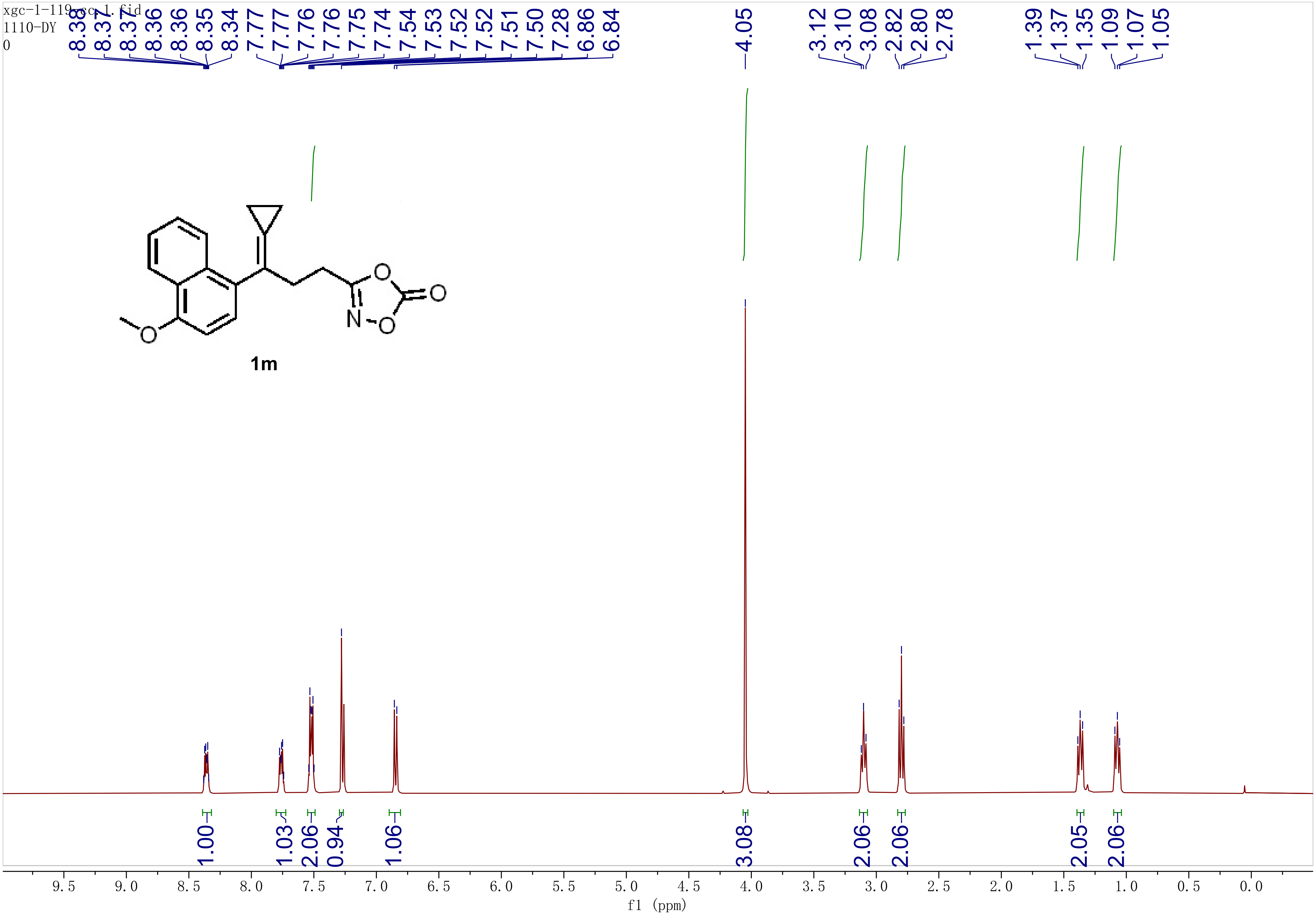

xgc-1-180.2.fid
13CPD
166.53
154.24
136.03
133.54
132.67
128.23
128.20
127.68
126.43
126.09
124.62
124.33
124.27
124.00
29.05
24.11
5.15
1.72
1j
f1 (ppm)

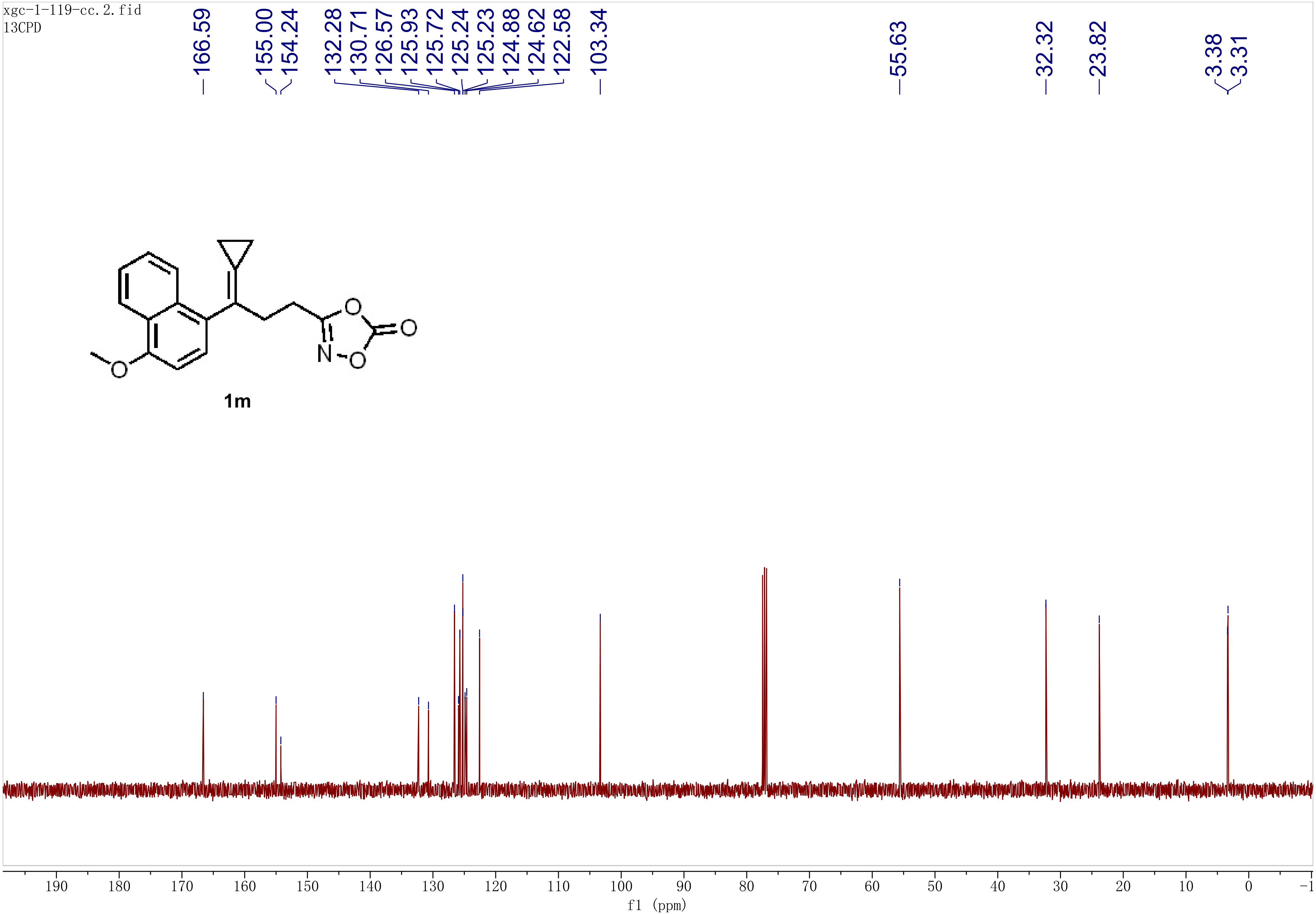

xgc-1-68-cc.1.fid
1110-DY
0
7.23
7.22
7.21
7.03
7.03
7.02
7.01
7.00
3.07
3.06
3.06
3.04
3.04
3.03
3.02
3.01
3.00
2.99
2.98
1.40
1.39
1.39
1.38
1.37
1.37
1.35
1.35
1.34
1.33
1.33
1.32
1.31
S
O
O
N
O
1k
0.83
2.00
4.00
4.06
f1 (ppm)

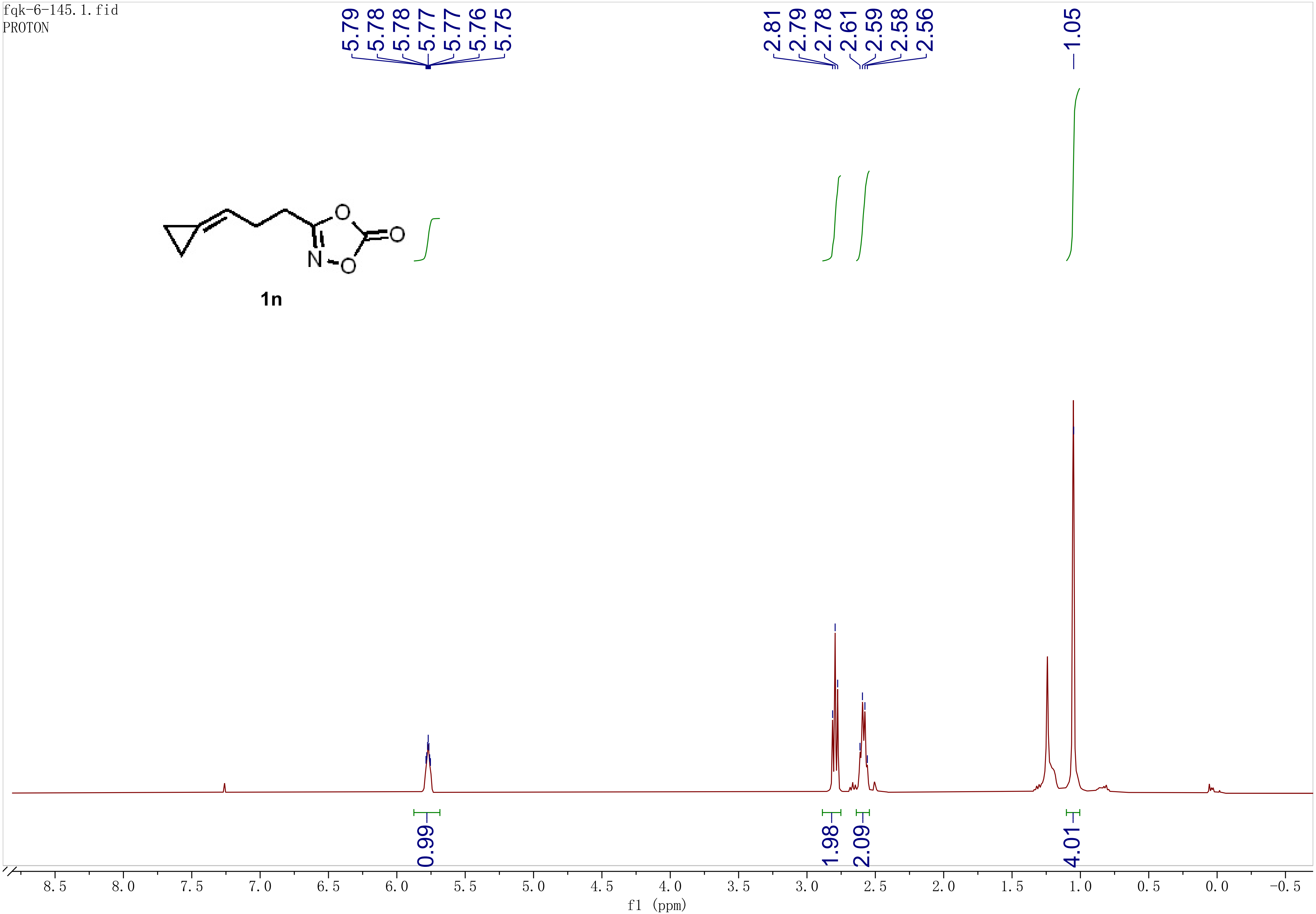

xgc-1-68-cc.2.fid
13CPD
166.38
154.18
144.53
127.21
124.92
122.07
122.04
120.00
28.80
23.95
5.00
3.31
1k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −1
f1 (ppm)

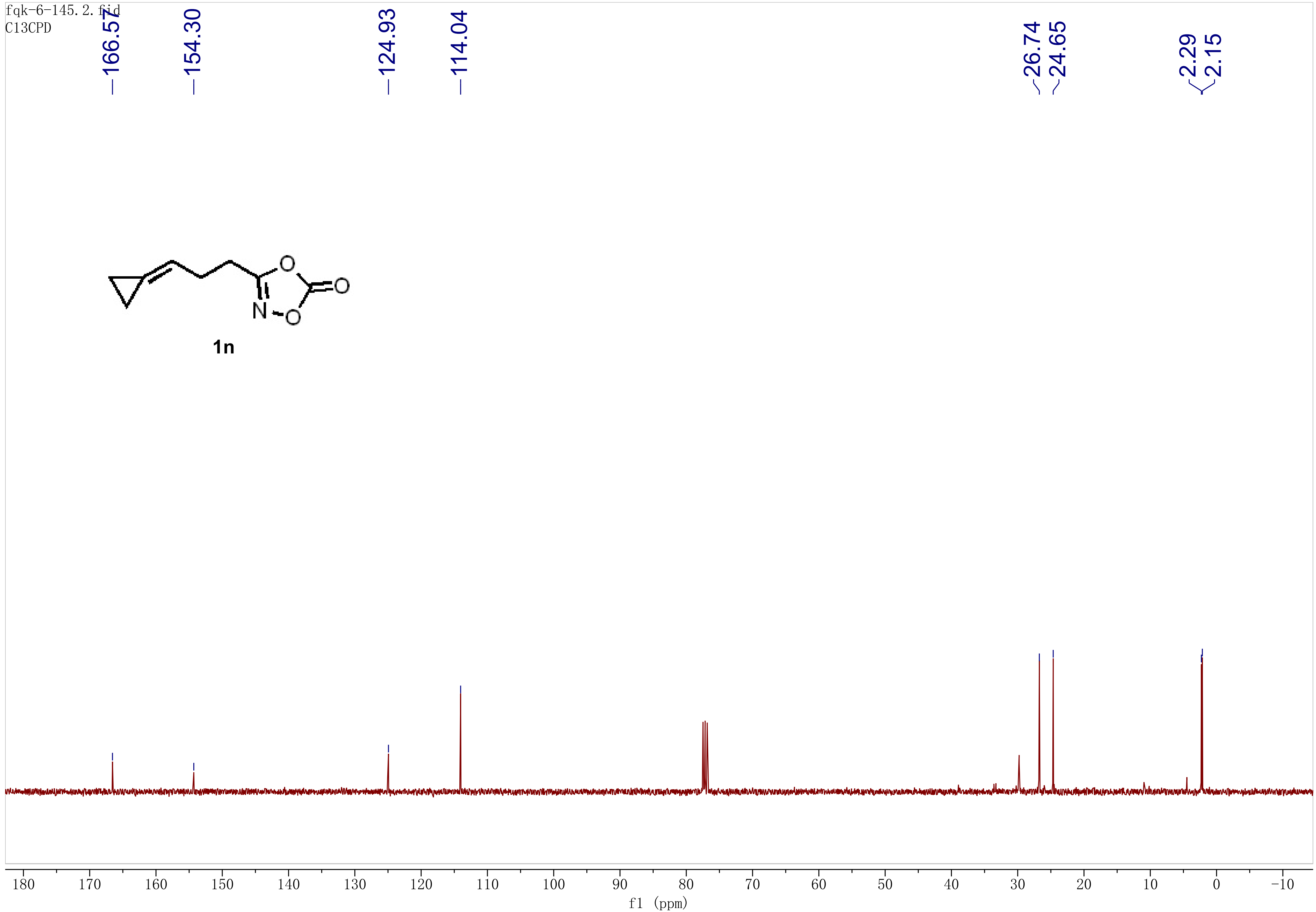

xgc-1-94.1.fid
PROTON
7.15 7.14 7.13 7.12 7.12 7.11 7.10 7.09 7.08 7.07 7.06 7.06 7.03 7.02 7.01 7.01
2.86 2.84 2.82 2.71 2.69 2.67 2.14
1.18 1.17 1.14 0.98 0.96 0.94
1l
3.03 1.00 2.00 2.01 3.01 2.00 2.00
f1 (ppm)

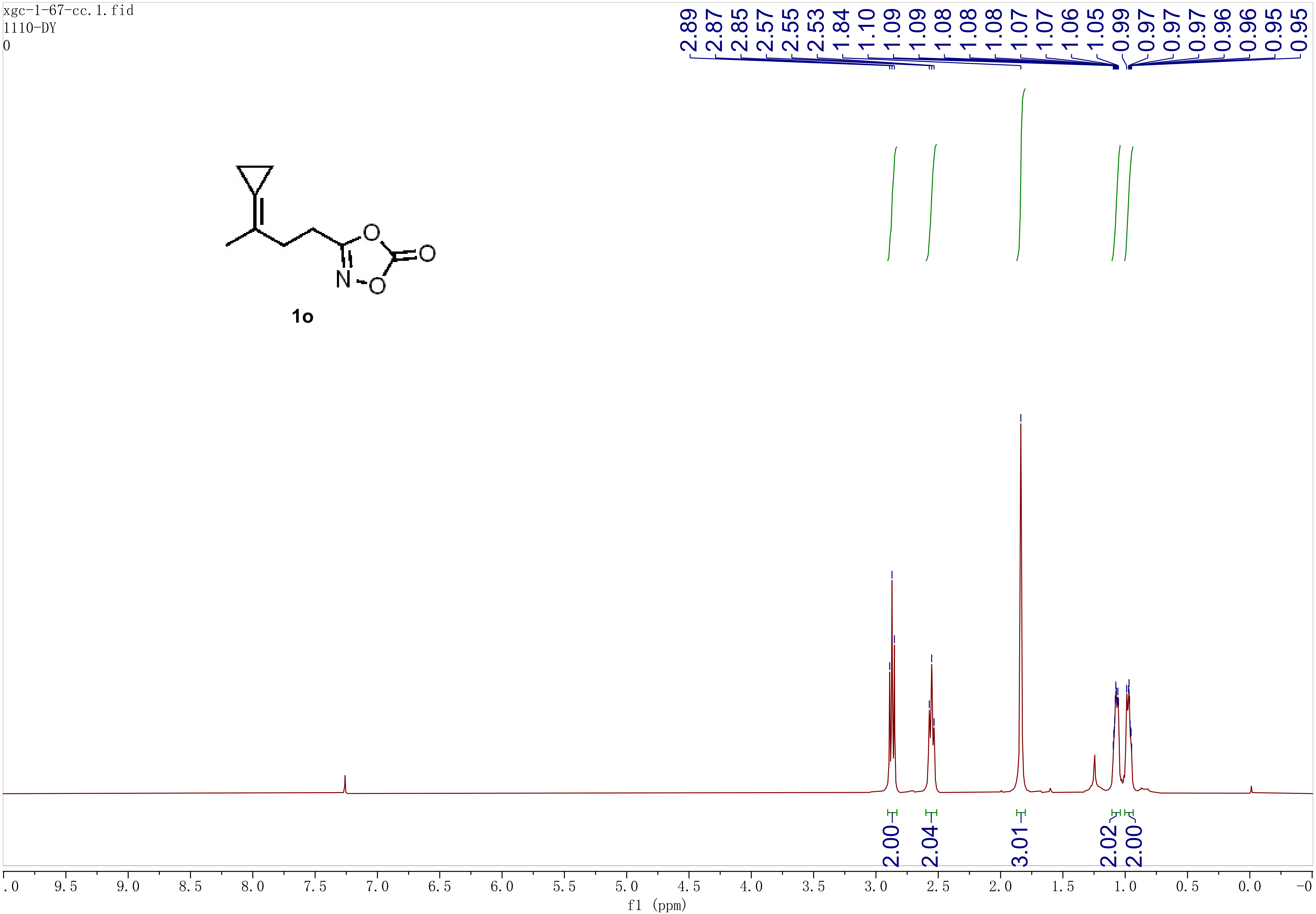

xgc-1-94.2.fid
C13CPD
166.54
154.19
140.19
135.76
130.50
128.50
127.32
125.82
125.75
123.65
31.60
23.63
19.92
3.29
2.98
1l
f1 (ppm)

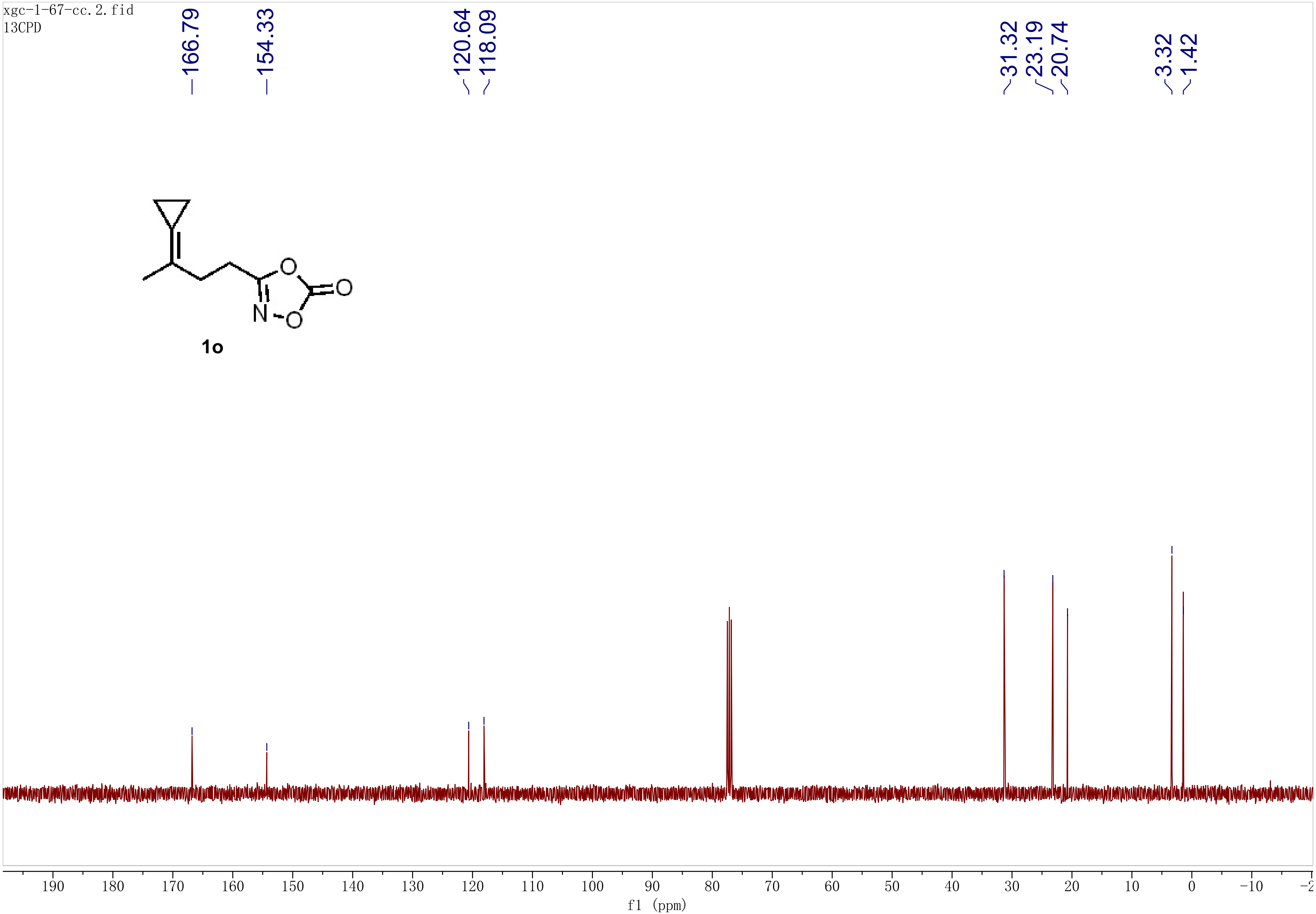

xgc-1-119-cc-1.fid
1110-DY
0
8.38 8.37 8.37 8.36 8.36 8.35 8.34 7.77 7.77 7.76 7.76 7.75 7.74 7.54 7.53 7.52 7.52 7.51 7.50 7.28 6.86 6.84
4.05
3.12 3.10 3.08 2.82 2.80 2.78
1.39 1.37 1.35 1.09 1.07 1.05
O
O
N
O
O
1m
1.00 1.03 2.06 0.94 1.06 3.08 2.06 2.06 2.05 2.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

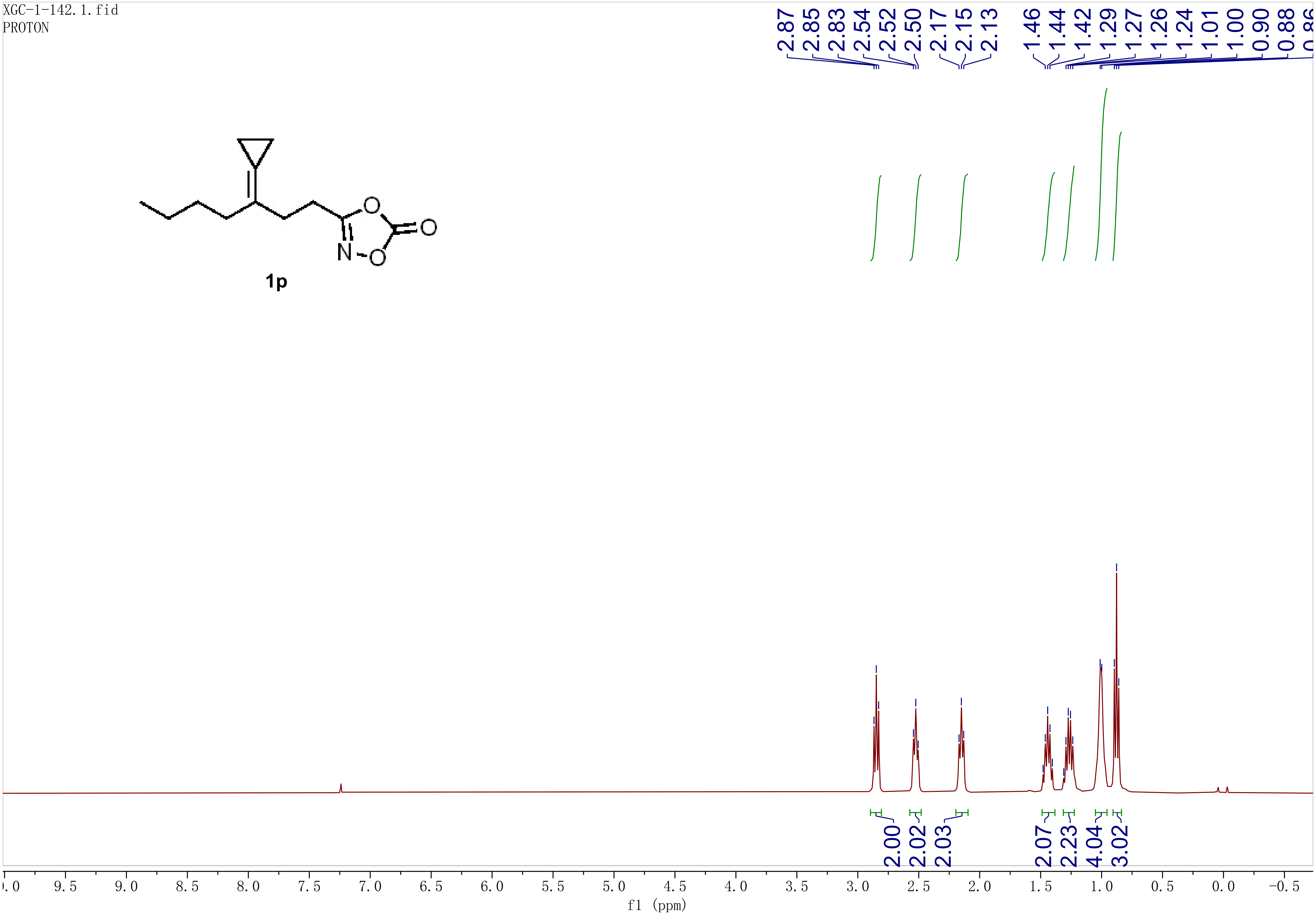

xgc-1-119-cc.2.fid
13CPD
166.59
155.00
154.24
132.28
130.71
126.57
125.93
125.72
125.24
125.23
124.88
124.62
122.58
103.34
55.63
32.32
23.82
3.38
3.31
1m
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

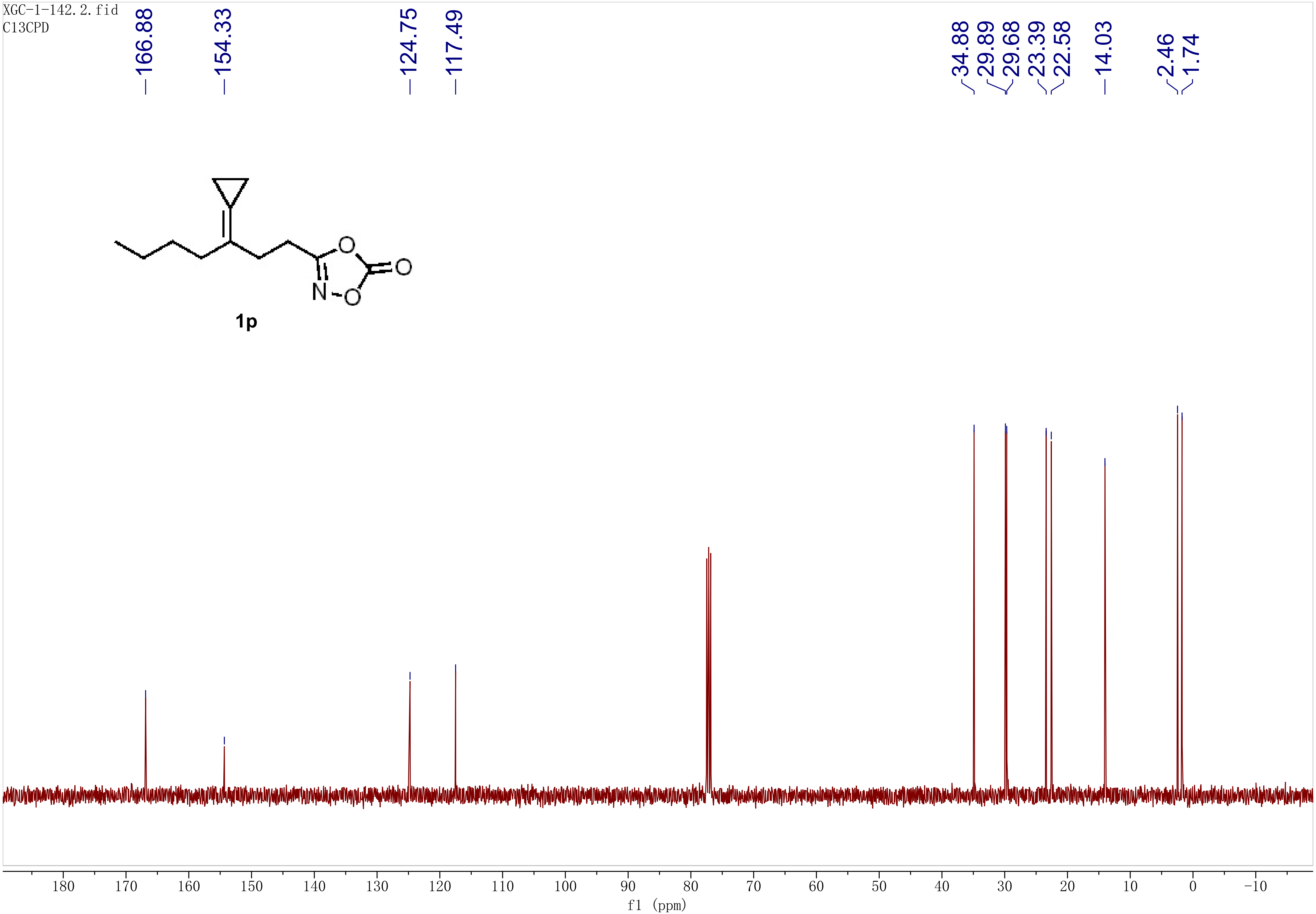

fqk-6-145.1.fid
PROTON
5.79
5.78
5.78
5.77
5.77
5.76
5.75
2.81
2.79
2.78
2.61
2.59
2.58
2.56
1.05
O
O
O
N
1n
0.99
1.98
2.09
4.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


fqk-6-145.2.fid
C13CPD
166.57
154.30
124.93
114.04
26.74
24.65
2.29
2.15
O
O
N
O
1n
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

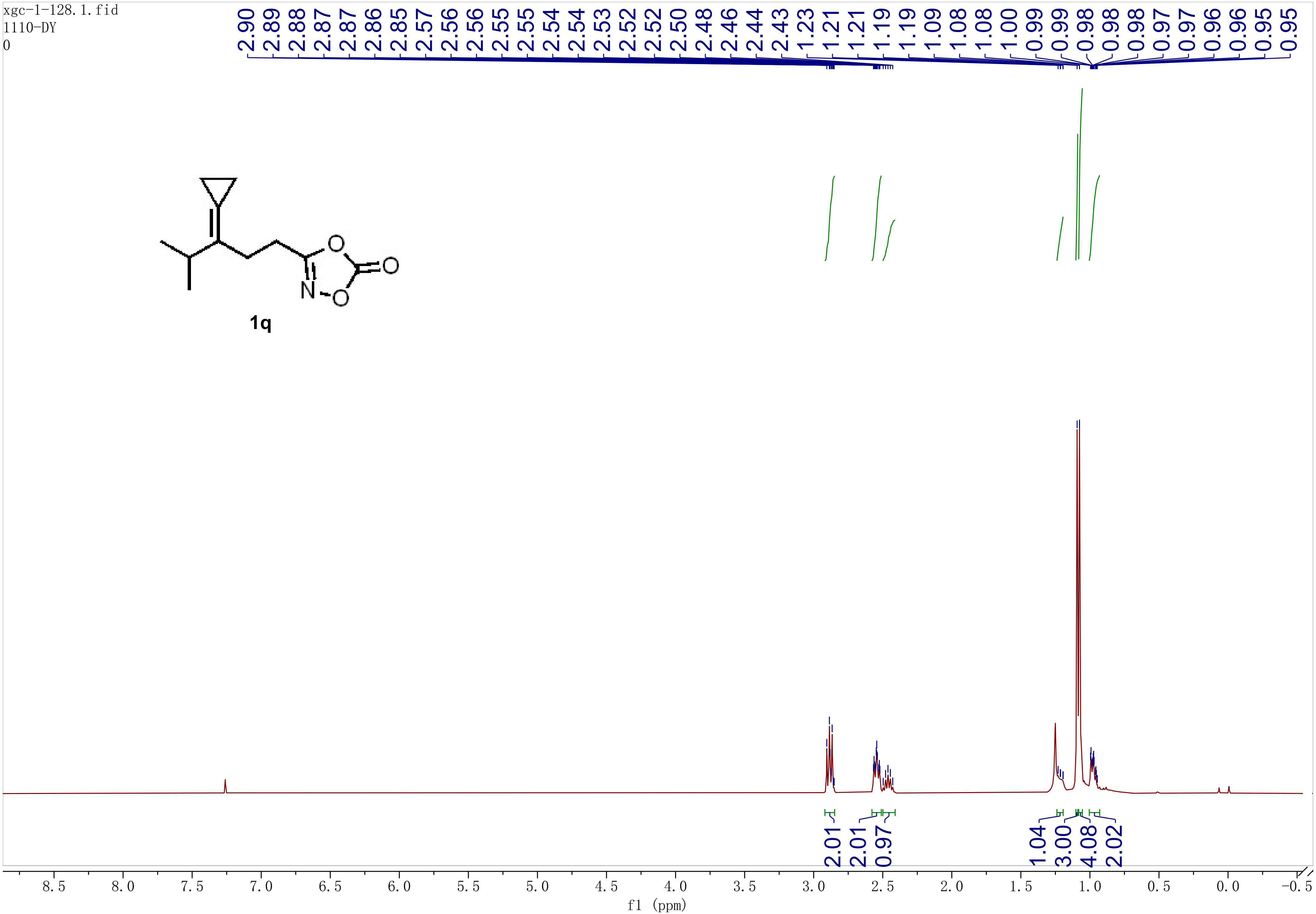

xgc-1-67-cc.1.fid
1110-DY
0
2.89
2.87
2.85
2.57
2.55
2.53
1.84
1.10
1.09
1.09
1.08
1.08
1.08
1.07
1.07
1.06
1.05
0.99
0.97
0.97
0.97
0.96
0.96
0.95
0.95
N
O
O
O
1o
2.00
2.04
3.01
2.02
2.00
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

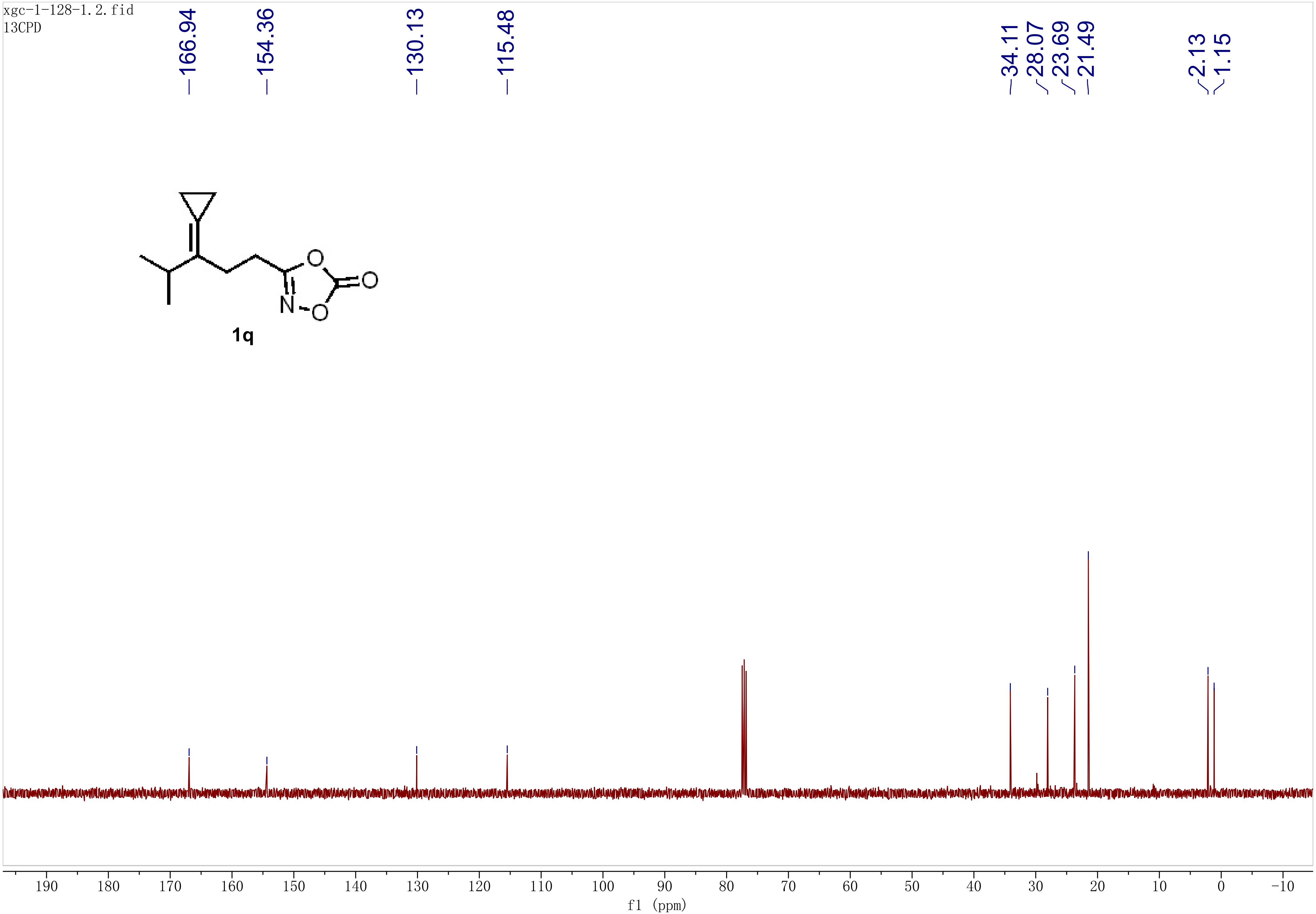

xgc-1-67-cc.2.fid
13CPD
166.79
154.33
120.64
118.09
31.32
23.19
20.74
3.32
1.42
1o
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

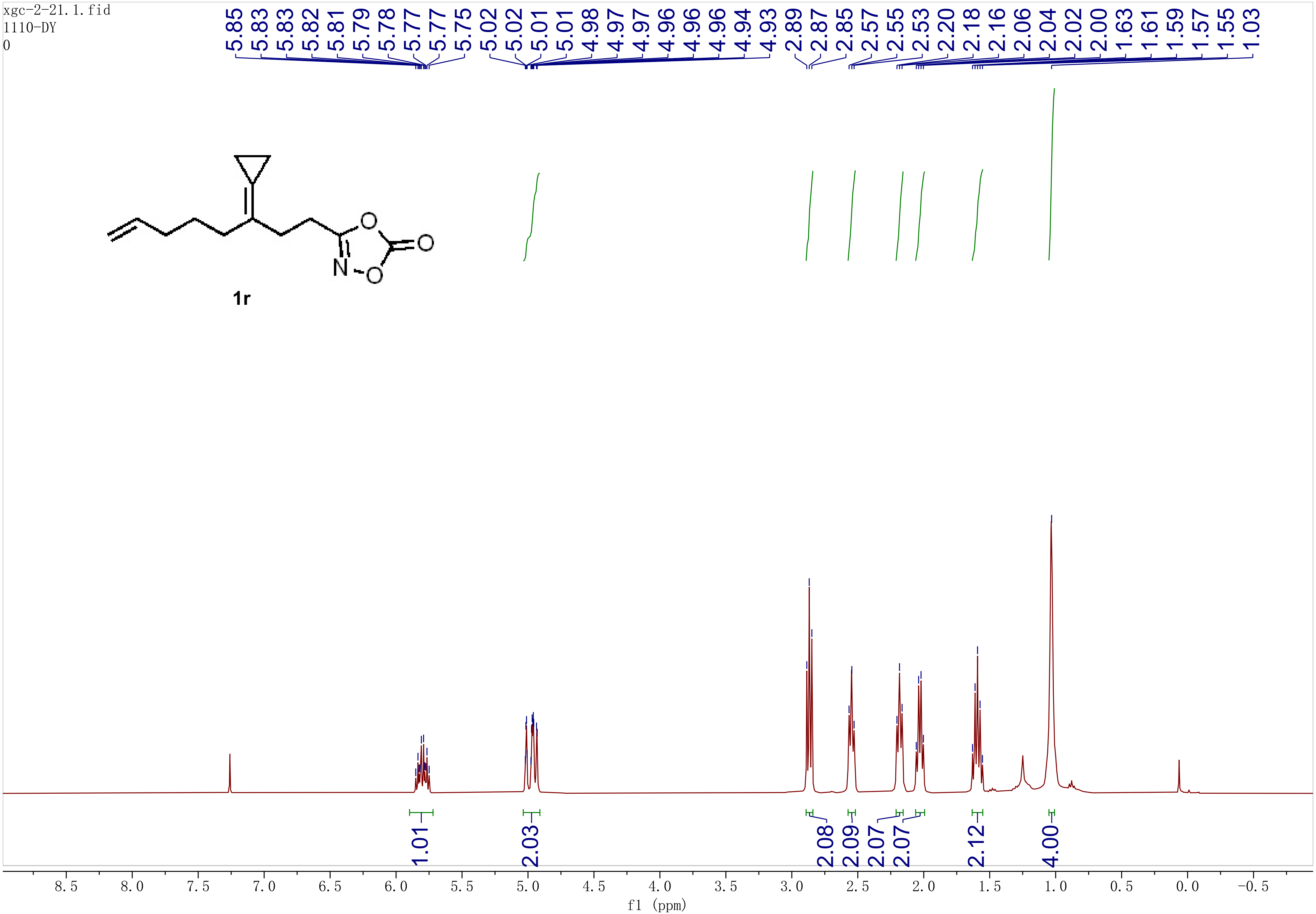

XGC-1-142.1.fid
PROTON
2.87
2.85
2.83
2.54
2.52
2.50
2.17
2.15
2.13
1.46
1.44
1.42
1.29
1.27
1.26
1.24
1.01
1.00
0.90
0.88
1p
2.00
2.02
2.03
2.07
2.23
4.04
3.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

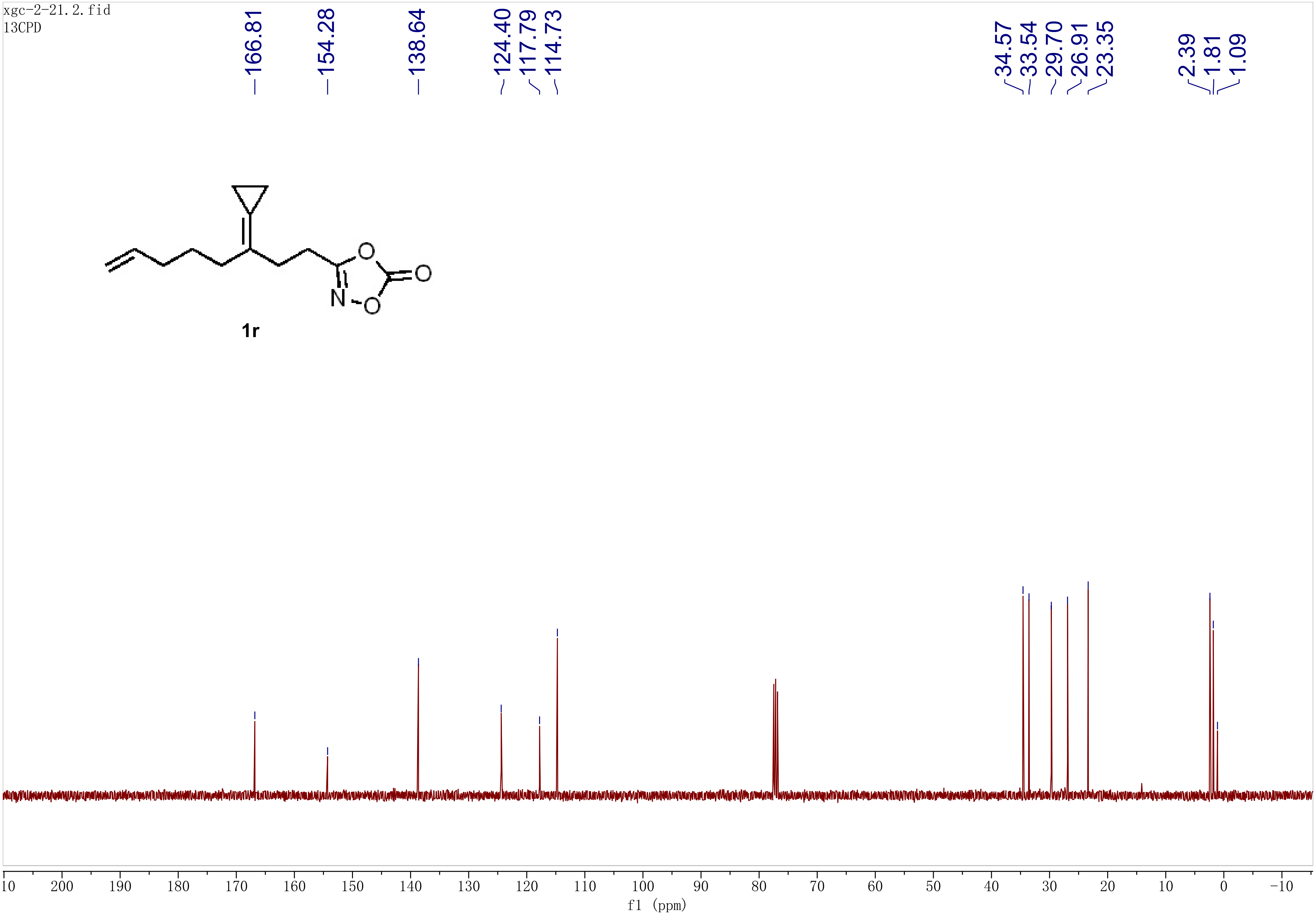


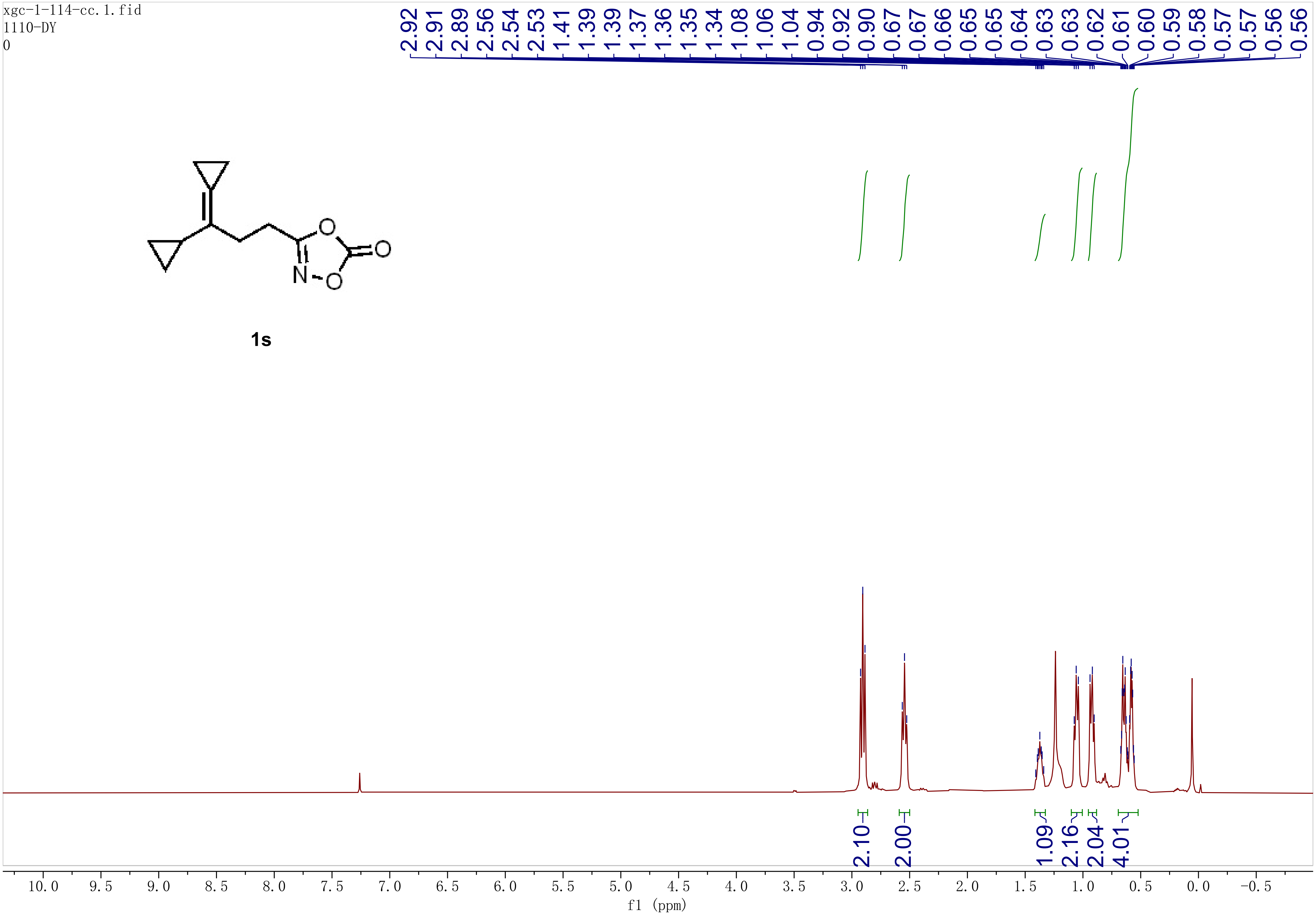
XGC-1-142.2.fid
C13CPD
166.88
154.33
124.75
117.49
34.88
29.89
29.68
23.39
22.58
14.03
2.46
1.74
1p
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

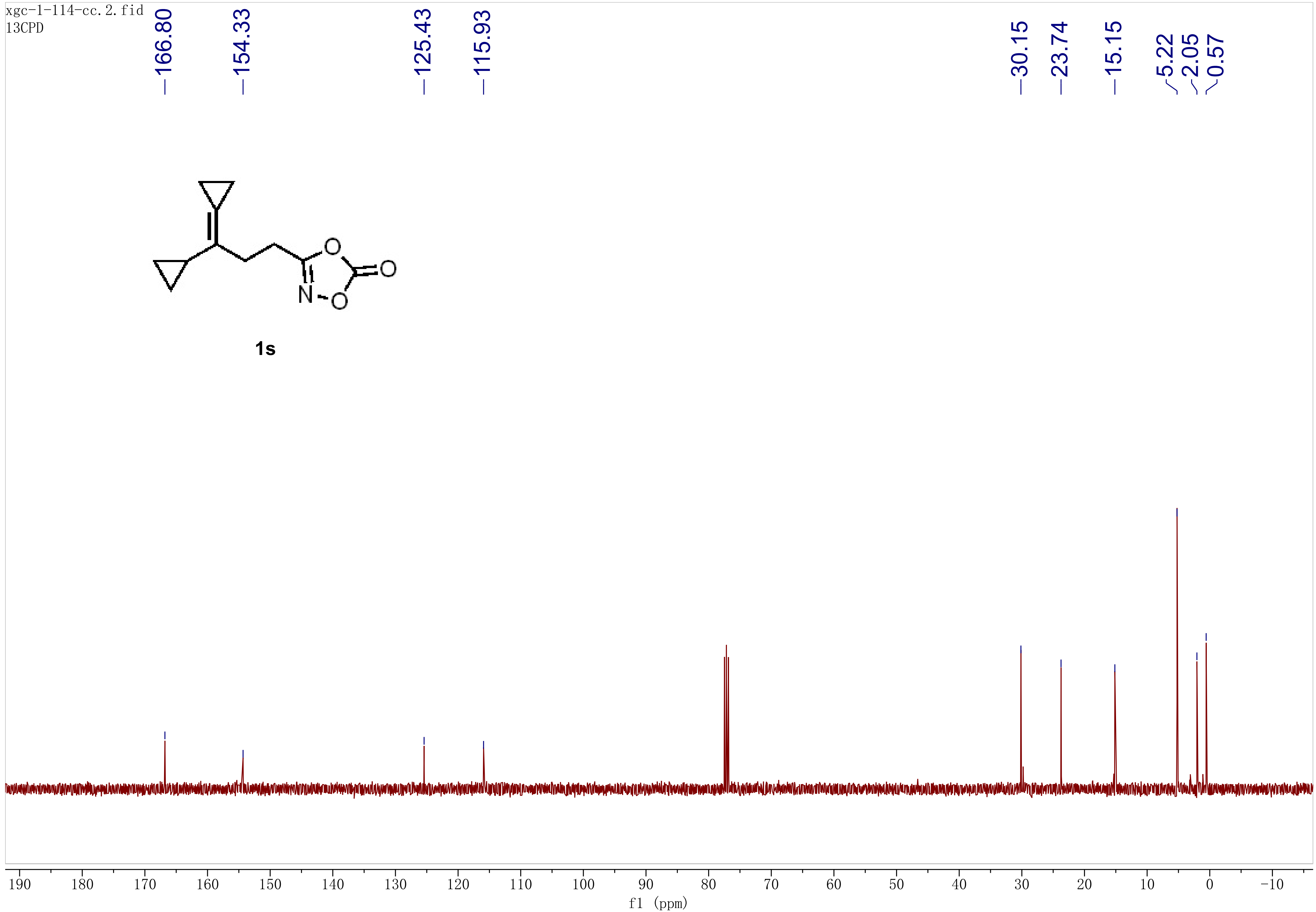

xgc-1-128.1.fid
1110-DY
0
1q
2.90 2.89 2.88 2.87 2.87 2.86 2.85 2.57 2.56 2.56 2.55 2.55 2.54 2.54 2.53 2.52 2.52 2.50 2.48 2.46 2.44 2.43 1.23 1.21 1.21 1.19 1.19 1.09 1.08 1.08 1.00 0.99 0.99 0.98 0.98 0.98 0.97 0.97 0.96 0.96 0.95 0.95
2.01 2.01 0.97 1.04 3.00 4.08 2.02
f1 (ppm)

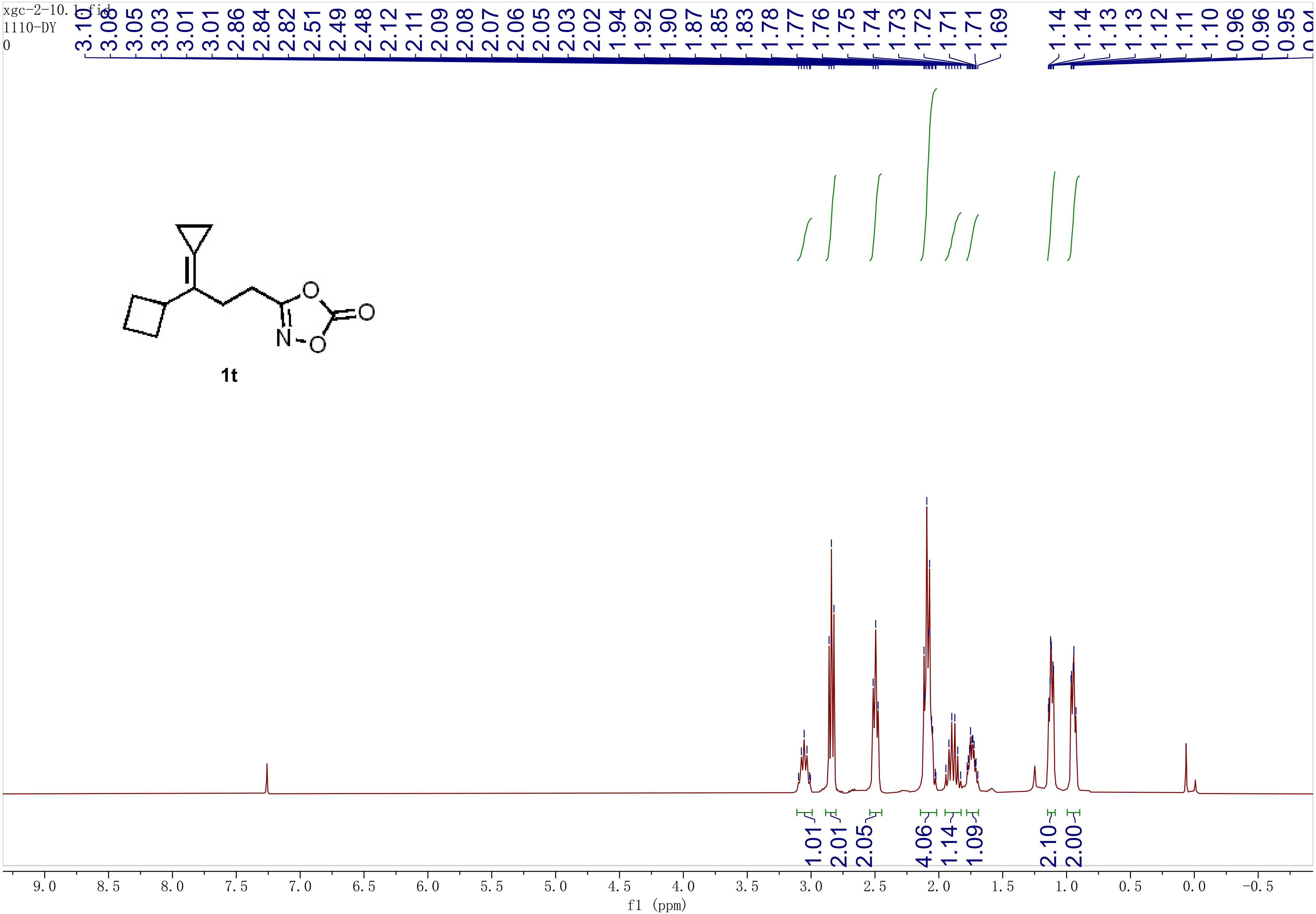

xgc-1-128-1.2.fid
13CPD
166.94
154.36
130.13
115.48
34.11
28.07
23.69
21.49
2.13
1.15
O
N
O
O
1q
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


xgc-2-21.1.fid
1110-DY
0
5.85 5.83 5.83 5.82 5.81 5.79 5.78 5.77 5.77 5.75 5.02 5.02 5.01 5.01 4.98 4.97 4.97 4.96 4.96 4.96 4.94 4.93 2.89 2.87 2.85 2.57 2.55 2.53 2.20 2.18 2.16 2.06 2.04 2.02 2.00 1.63 1.61 1.59 1.57 1.55 1.03
1r
1.01
2.03
2.08
2.09
2.07
2.07
2.12
4.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


xgc-2-21.2.fid
13CPD
166.81
154.28
138.64
124.40
117.79
114.73
34.57
33.54
29.70
26.91
23.35
2.39
1.81
1.09
O
O
N
O
1r
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

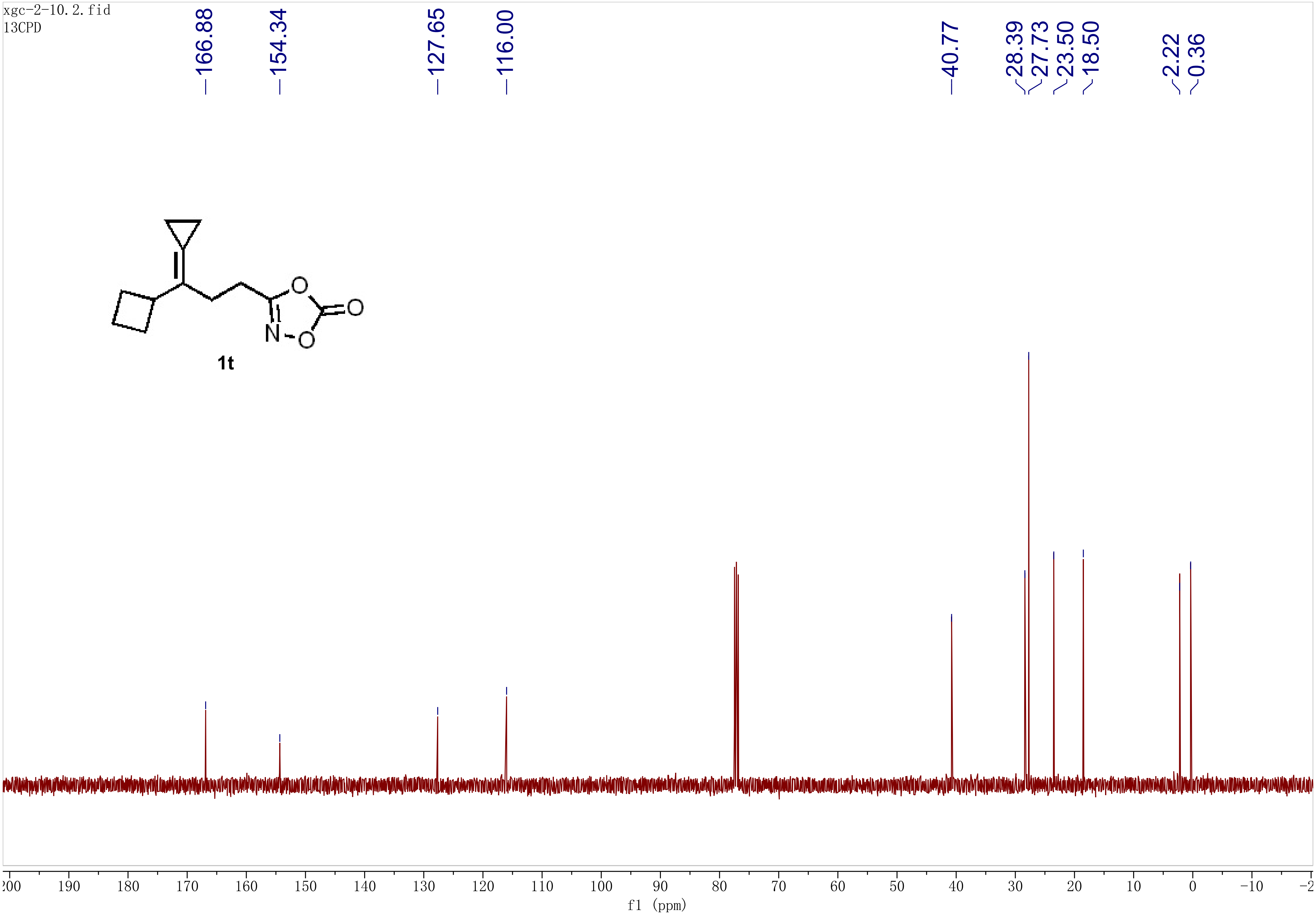

xgc-1-114-cc.1.fid
1110-DY
0
2.92 2.91 2.89 2.56 2.54 2.53 1.41 1.39 1.39 1.37 1.36 1.35 1.34 1.08 1.06 1.04 0.94 0.92 0.90 0.67 0.67 0.66 0.65 0.65 0.64 0.63 0.63 0.62 0.62 0.61 0.60 0.59 0.58 0.57 0.57 0.56 0.56
N
O
O
O
1s
2.10
2.00
1.09
2.16
2.04
4.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

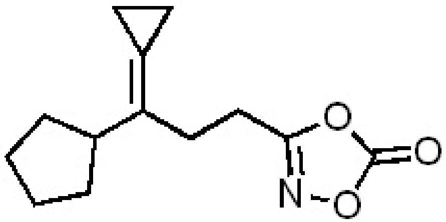

xgc-1-114-cc.2.fid
13CPD
166.80
154.33
125.43
115.93
30.15
23.74
15.15
5.22
2.05
0.57
1s
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



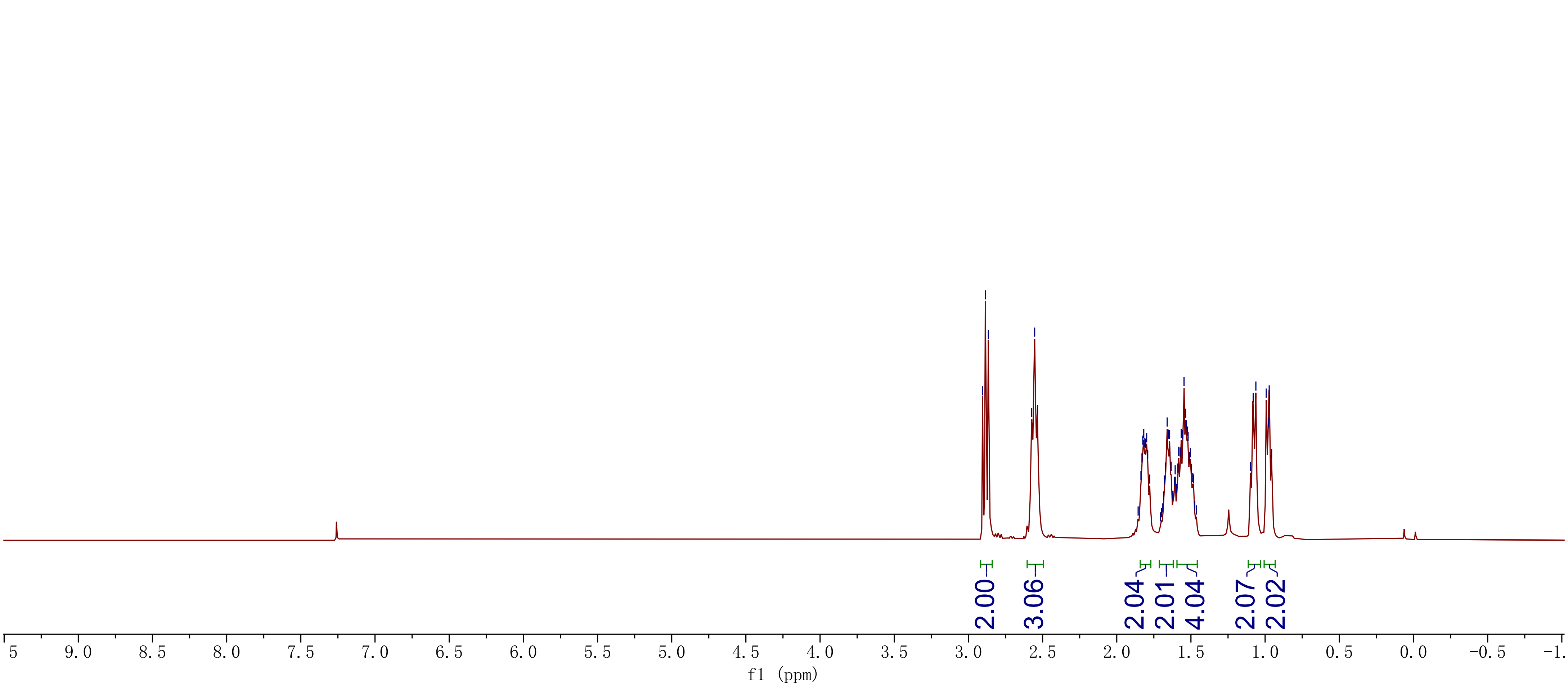
xgc-2-10.1.fid
1110-DY
0
3.10 3.08 3.05 3.03 3.01 3.01 2.86 2.84 2.82 2.51 2.49 2.48 2.12 2.11 2.09 2.08 2.07 2.06 2.05 2.03 2.02 1.94 1.92 1.90 1.87 1.85 1.83 1.78 1.77 1.76 1.75 1.74 1.73 1.72 1.71 1.71 1.69 1.14 1.14 1.13 1.13 1.12 1.11 1.10 0.96 0.96 0.95
1.01 2.01 2.05 4.06 1.14 1.09 2.10 2.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

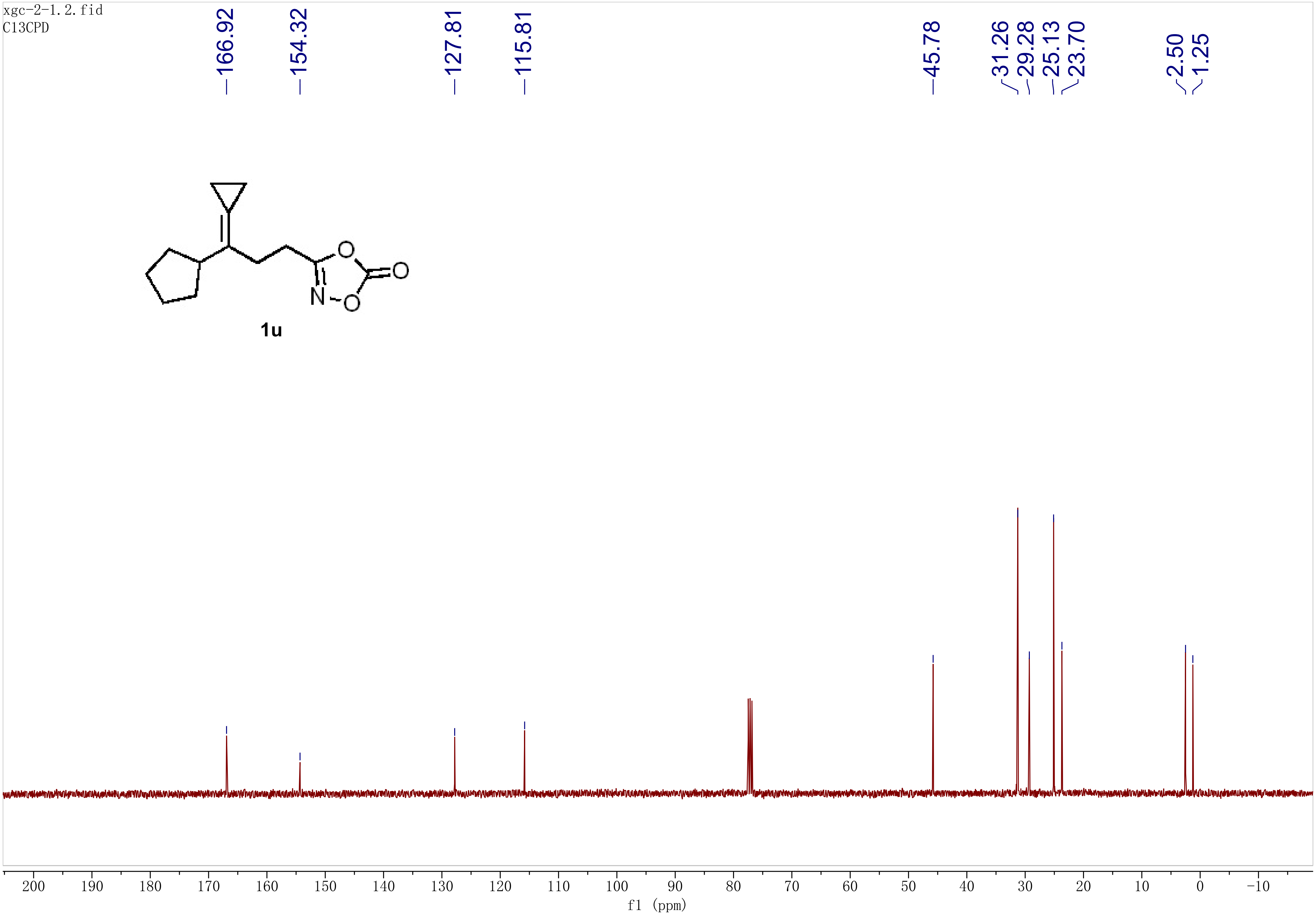
1t

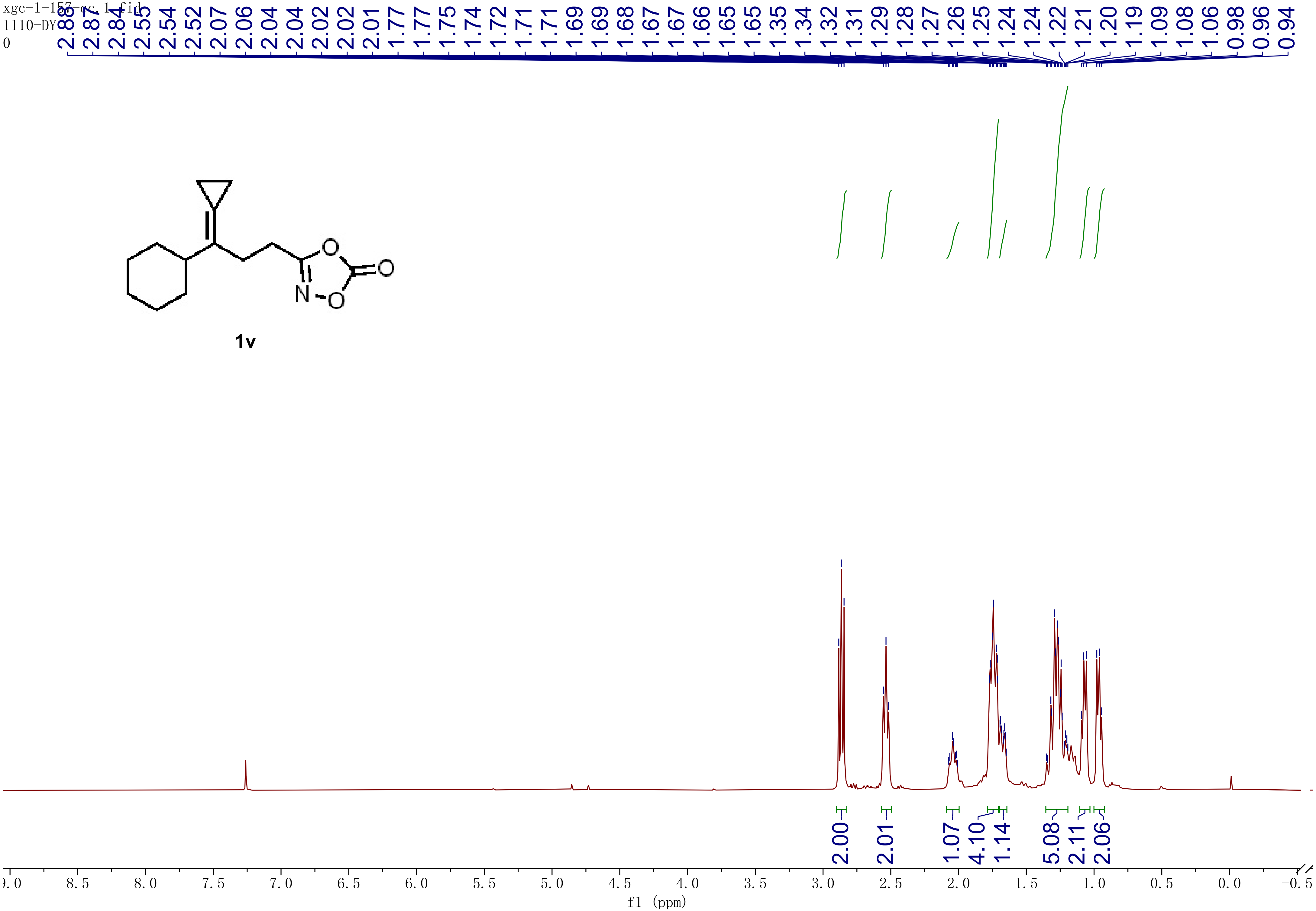

xgc-2-10.2.fid
13CPD
166.88
154.34
127.65
116.00
40.77
28.39
27.73
23.50
18.50
2.22
0.36
1t
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −2
f1 (ppm)



1u

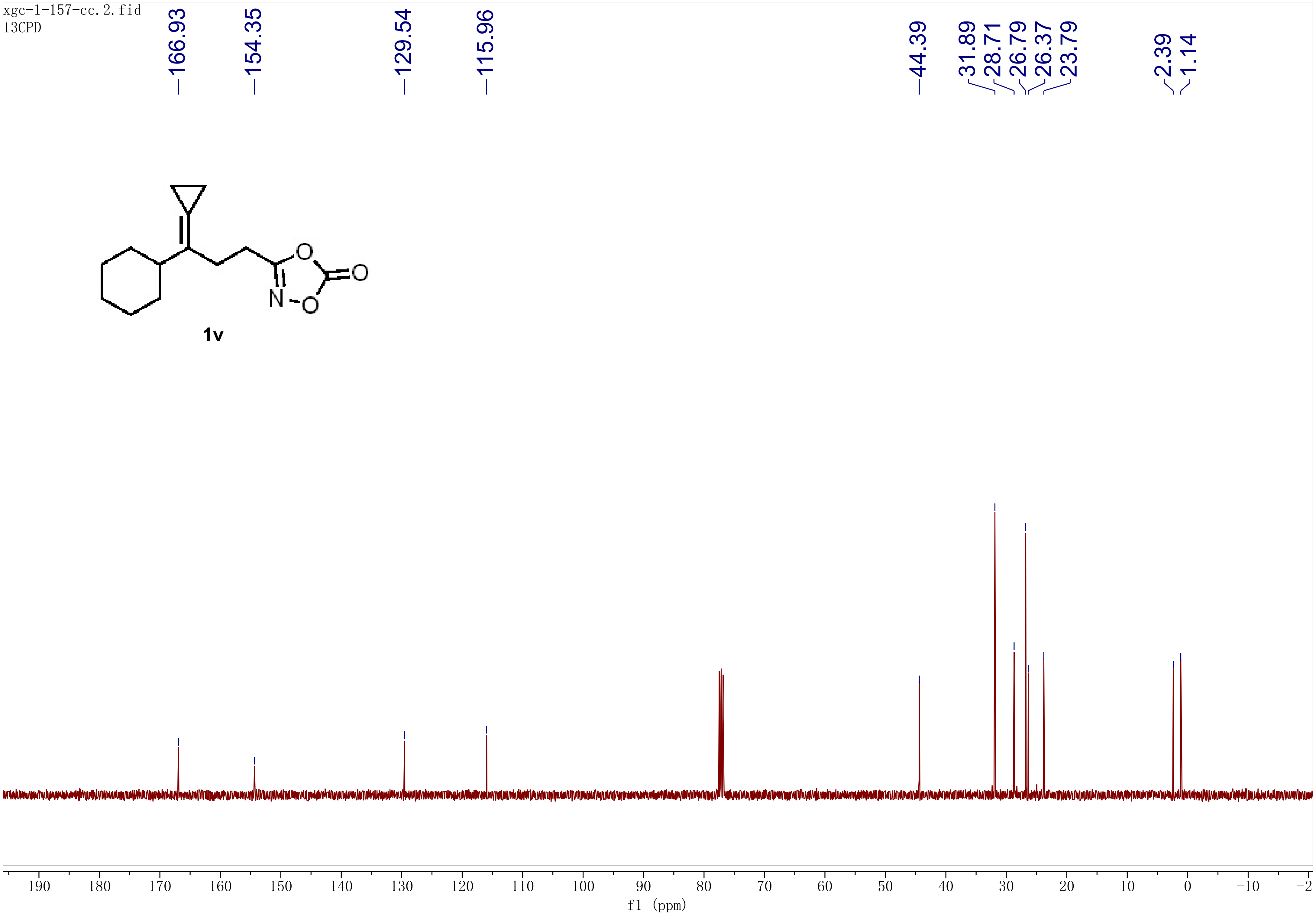

xgc-2-1.1.fid
PROTON
2.90 2.89 2.87 2.57 2.55 2.53 1.84 1.83 1.82 1.82 1.81 1.80 1.80 1.79 1.78 1.69 1.68 1.68 1.67 1.66 1.65 1.64 1.63 1.62 1.61 1.61 1.60 1.59 1.58 1.57 1.57 1.56 1.55 1.54 1.53 1.52 1.52 1.51 1.50 1.50 1.49 1.48 1.47 1.10 1.08 1.06 0.99 0.98 0.97 0.97 0.96
2.00 3.06 2.04 2.01 4.04 2.07 2.02
5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.
f1 (ppm)

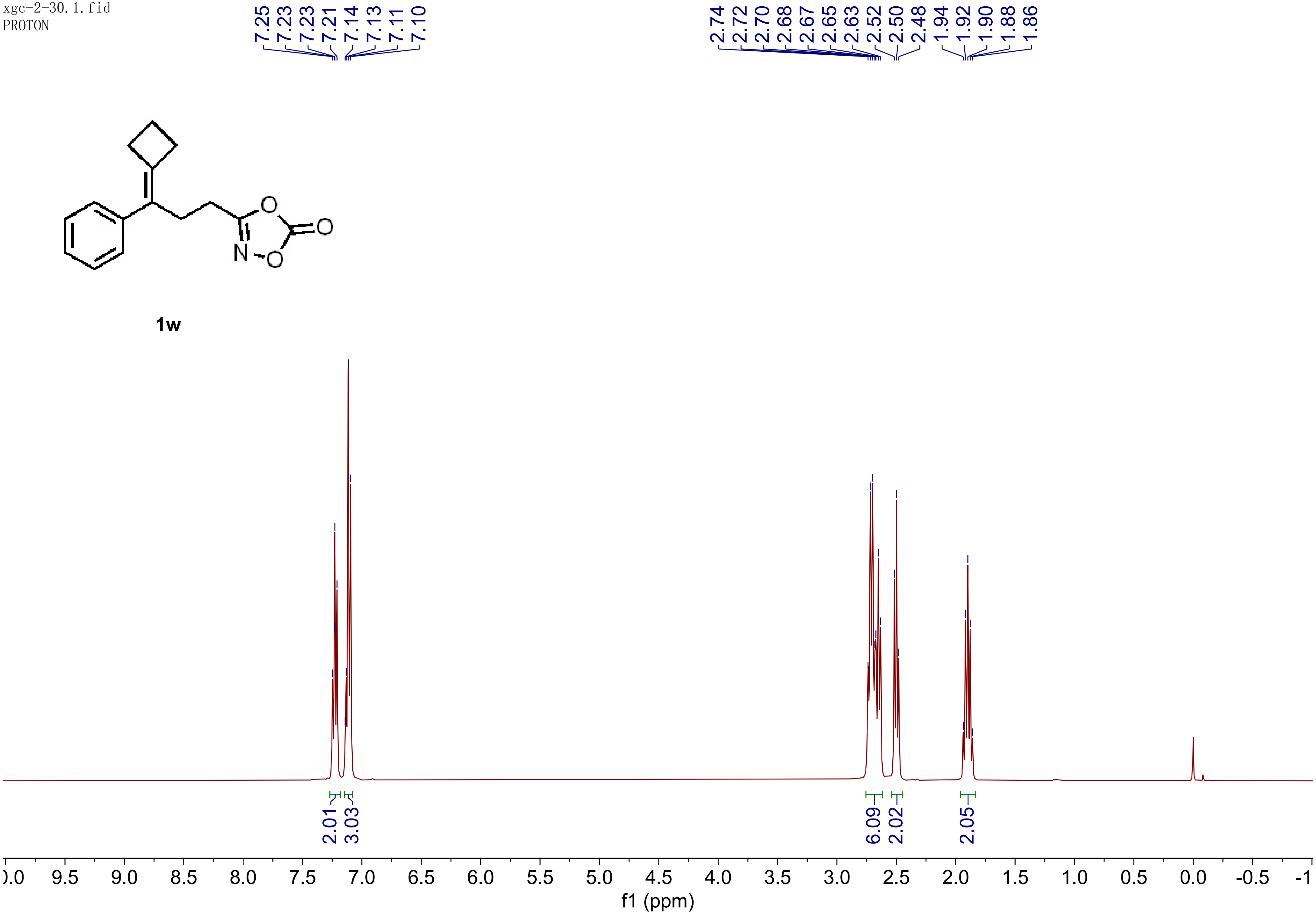

xgc-2-1.2.fid
C13CPD
166.92
154.32
127.81
115.81
45.78
31.26
29.28
25.13
23.70
2.50
1.25
1u
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

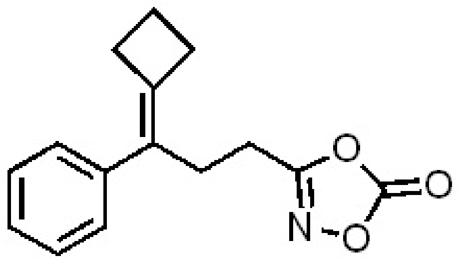


xgc-1-157-qc.1.fid
1110-DY
0
2.88 2.87 2.84 2.55 2.54 2.52 2.07 2.06 2.04 2.04 2.02 2.02 2.01 1.77 1.77 1.75 1.74 1.72 1.71 1.71 1.69 1.69 1.68 1.67 1.67 1.66 1.65 1.65 1.35 1.34 1.32 1.31 1.29 1.28 1.27 1.26 1.25 1.24 1.24 1.22 1.21 1.20 1.19 1.09 1.08 1.06 0.98 0.96 0.94
2.00
2.01
1.07
4.10
1.14
5.08
2.11
2.06
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

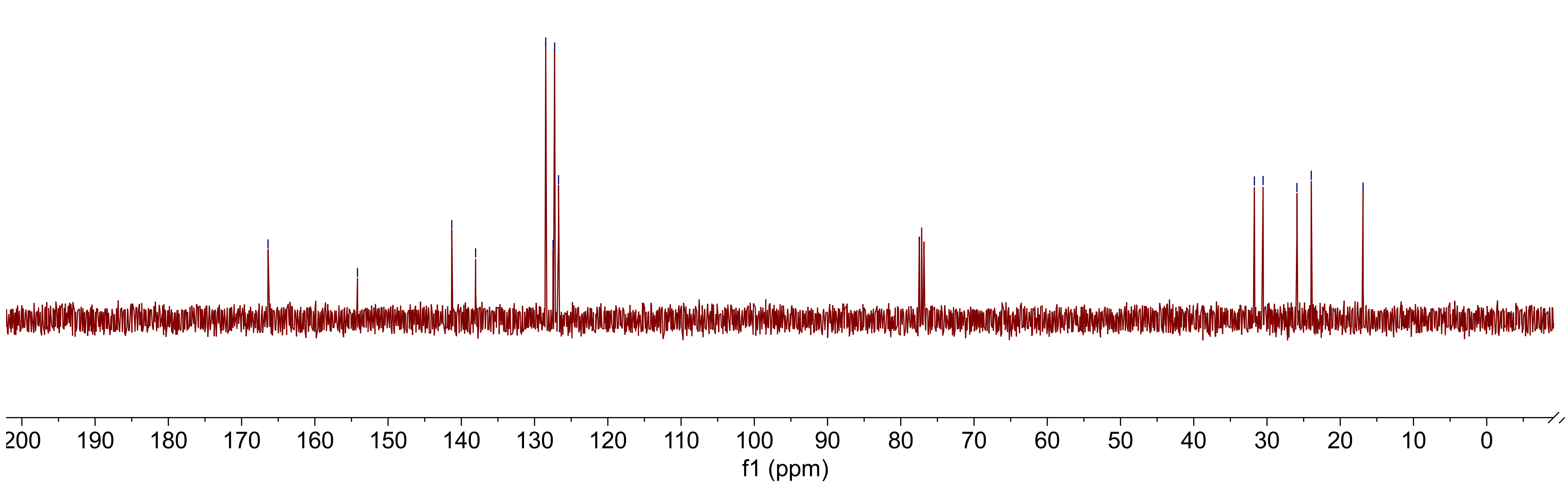
1v


xgc-1-157-cc.2.fid
13CPD
166.93
154.35
129.54
115.96
44.39
31.89
28.71
26.79
26.37
23.79
2.39
1.14
1v
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2
f1 (ppm)

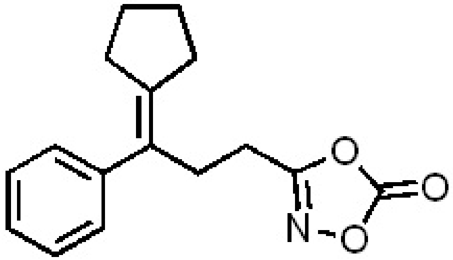

xgc-2-30.1.fid
PROTON
7.25
7.23
7.23
7.21
7.14
7.13
7.11
7.10
2.74
2.72
2.70
2.68
2.67
2.65
2.63
2.52
2.50
2.48
1.94
1.92
1.90
1.88
1.86
O
O
O
N
1w
2.01
3.03
6.09
2.02
2.05
f1 (ppm)



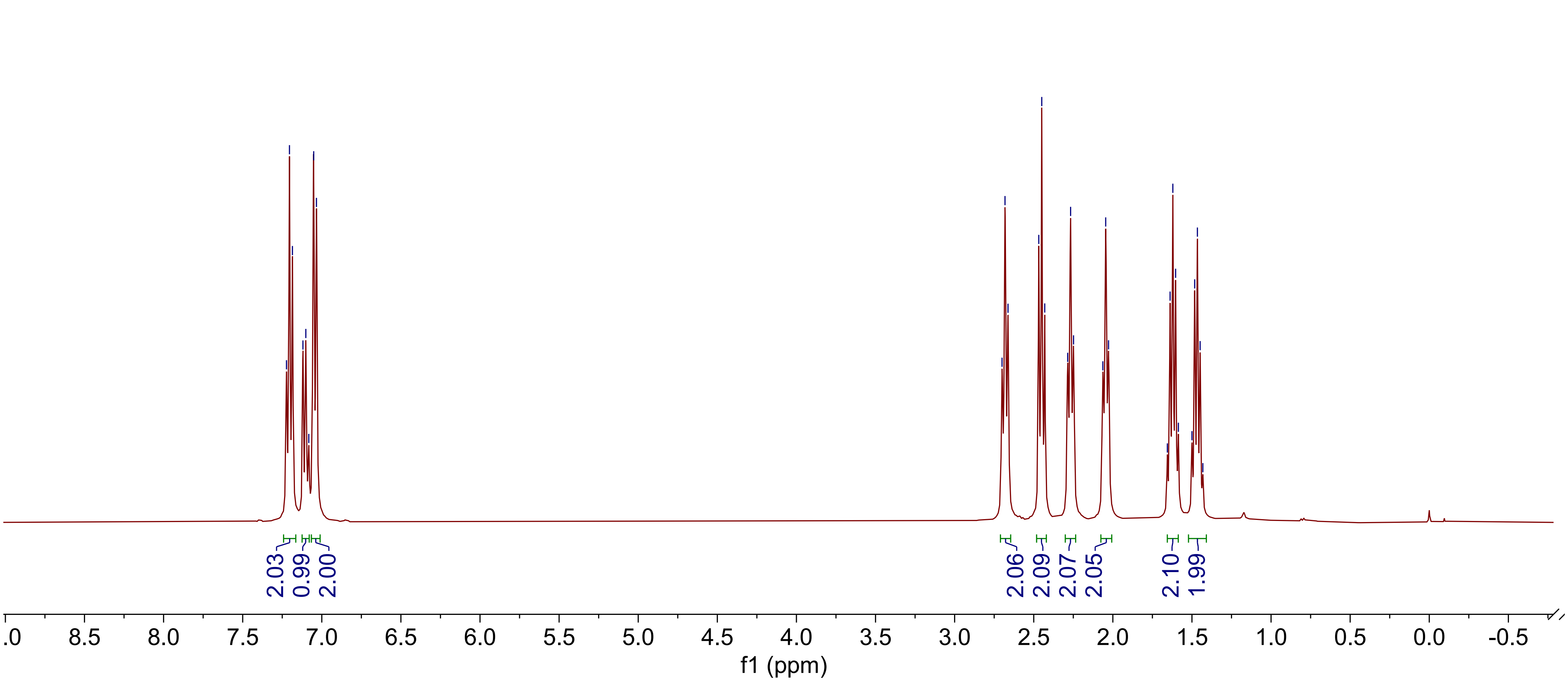
1w


xgc-2-30.3.fid
C13CPD
166.39
154.18
141.30
138.04
128.48
127.47
127.26
126.73
31.72
30.53
25.91
23.96
16.88
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

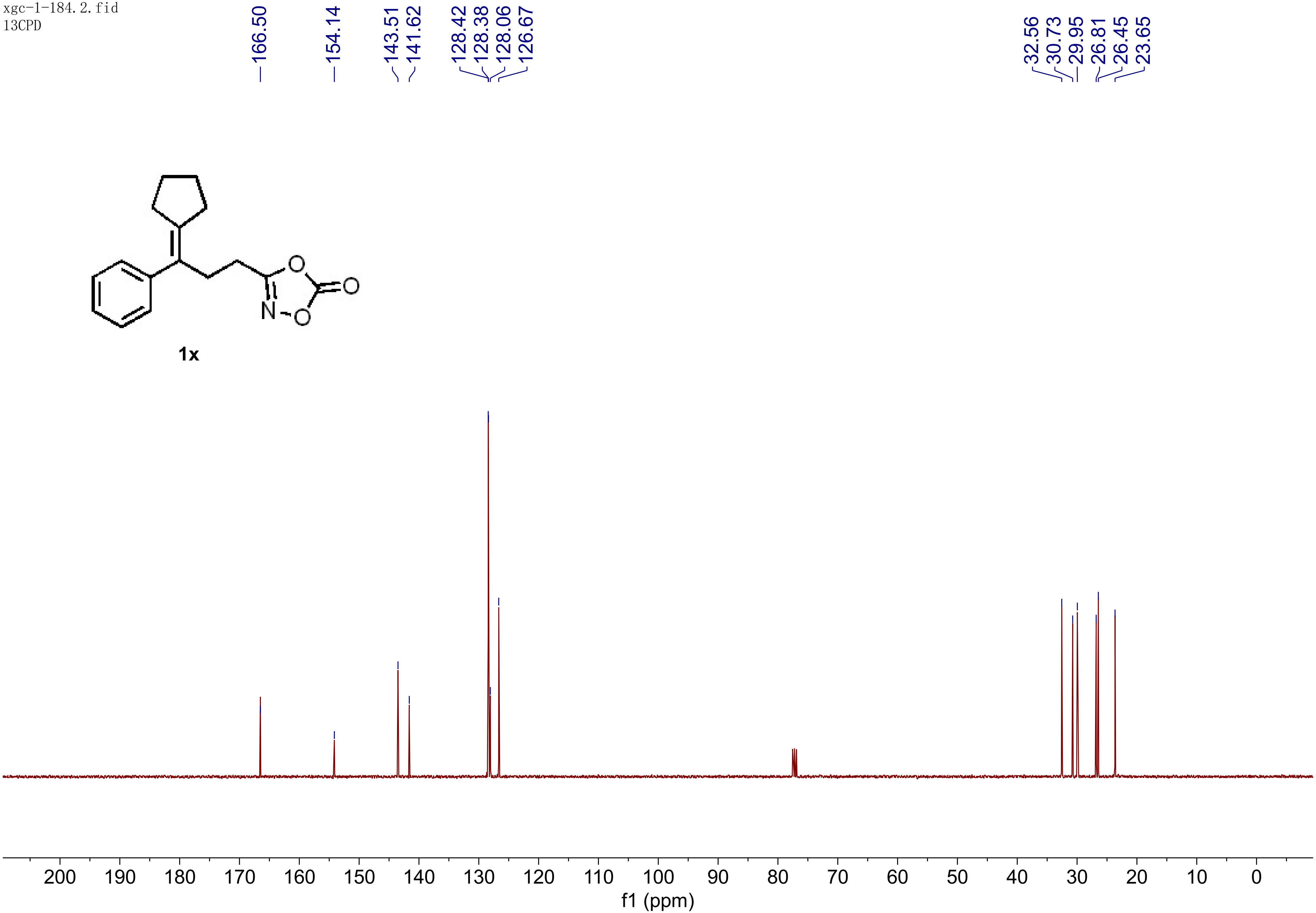
xgc-1-184.1.fid
1110-DY
0

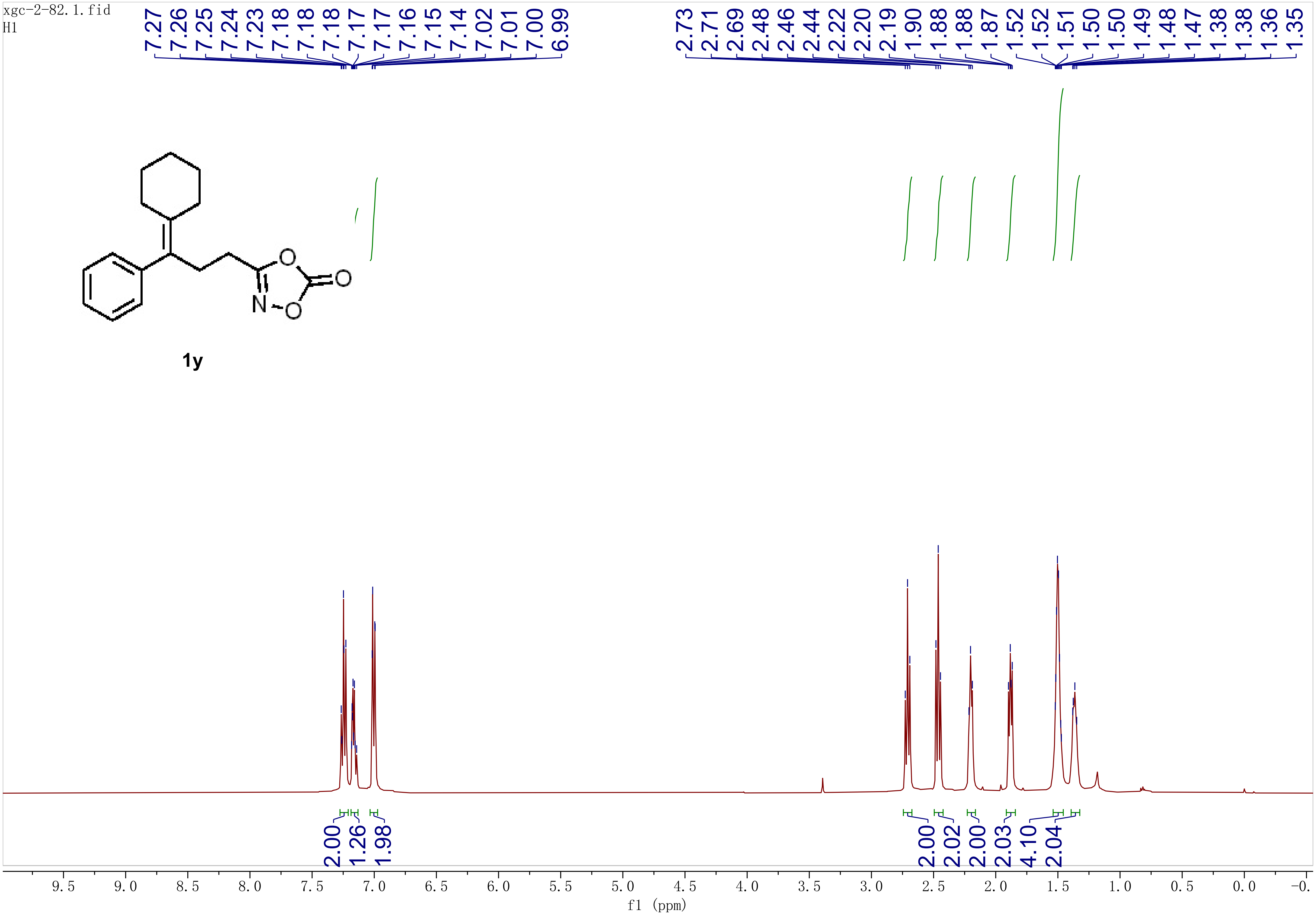


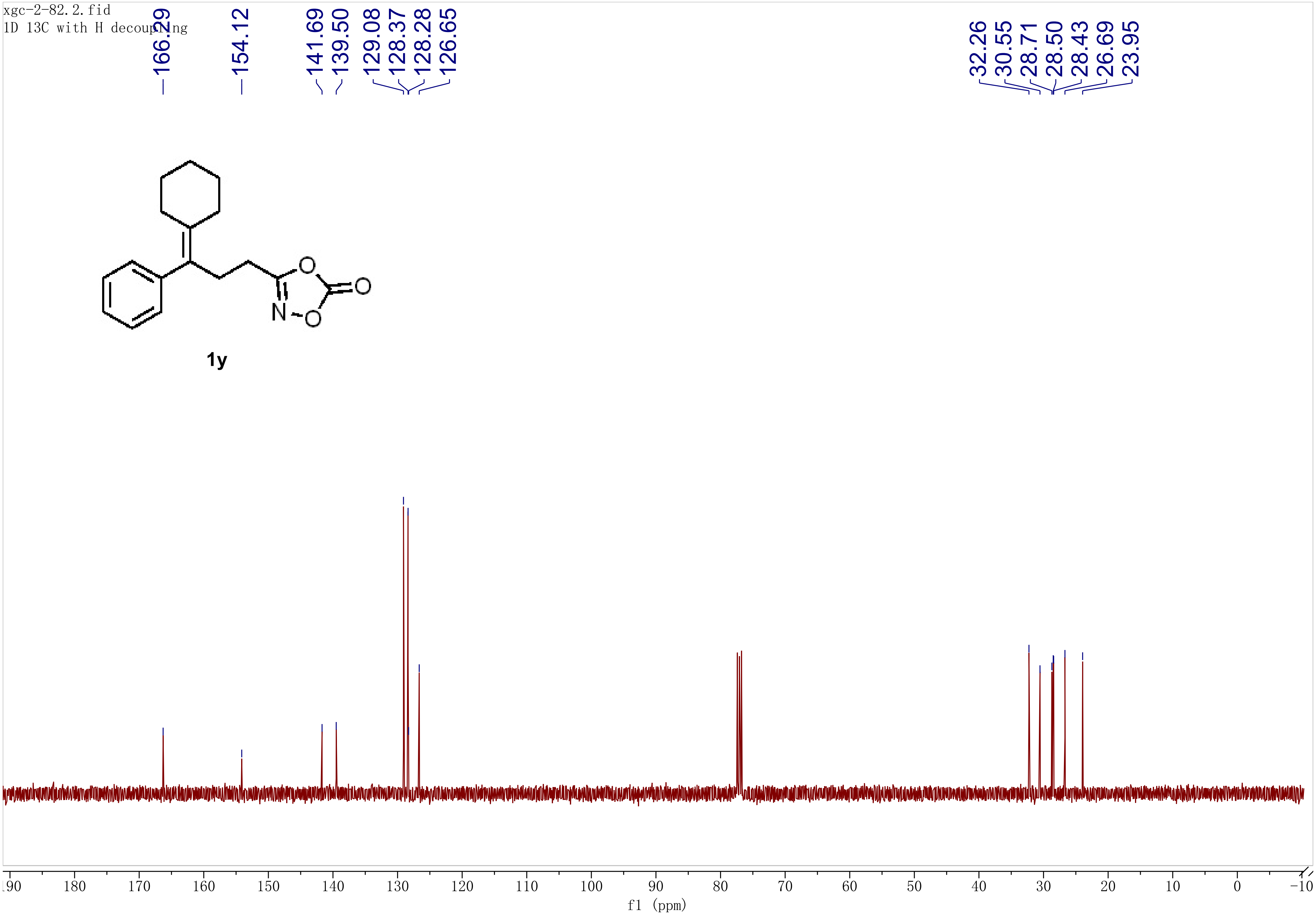
1x

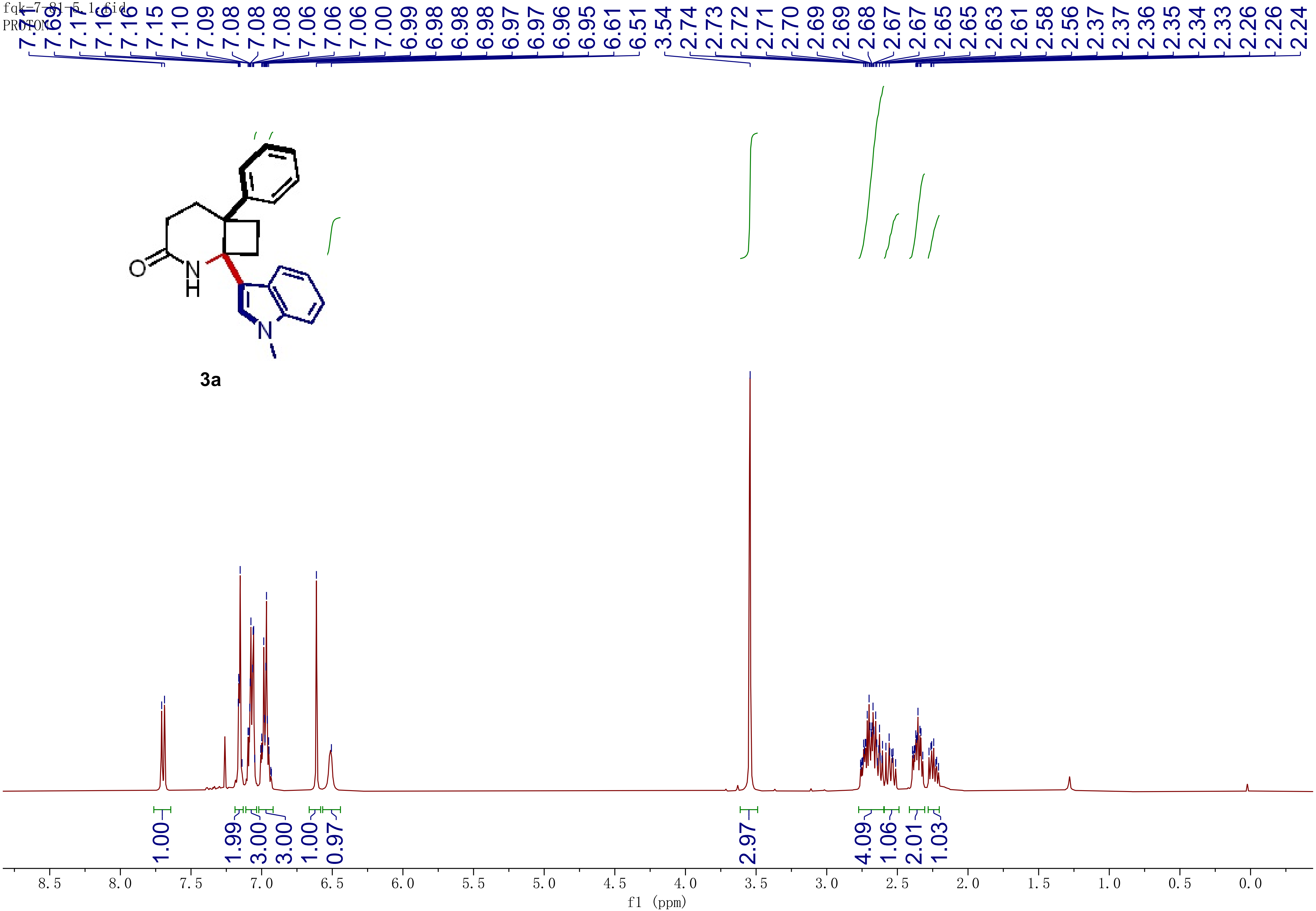


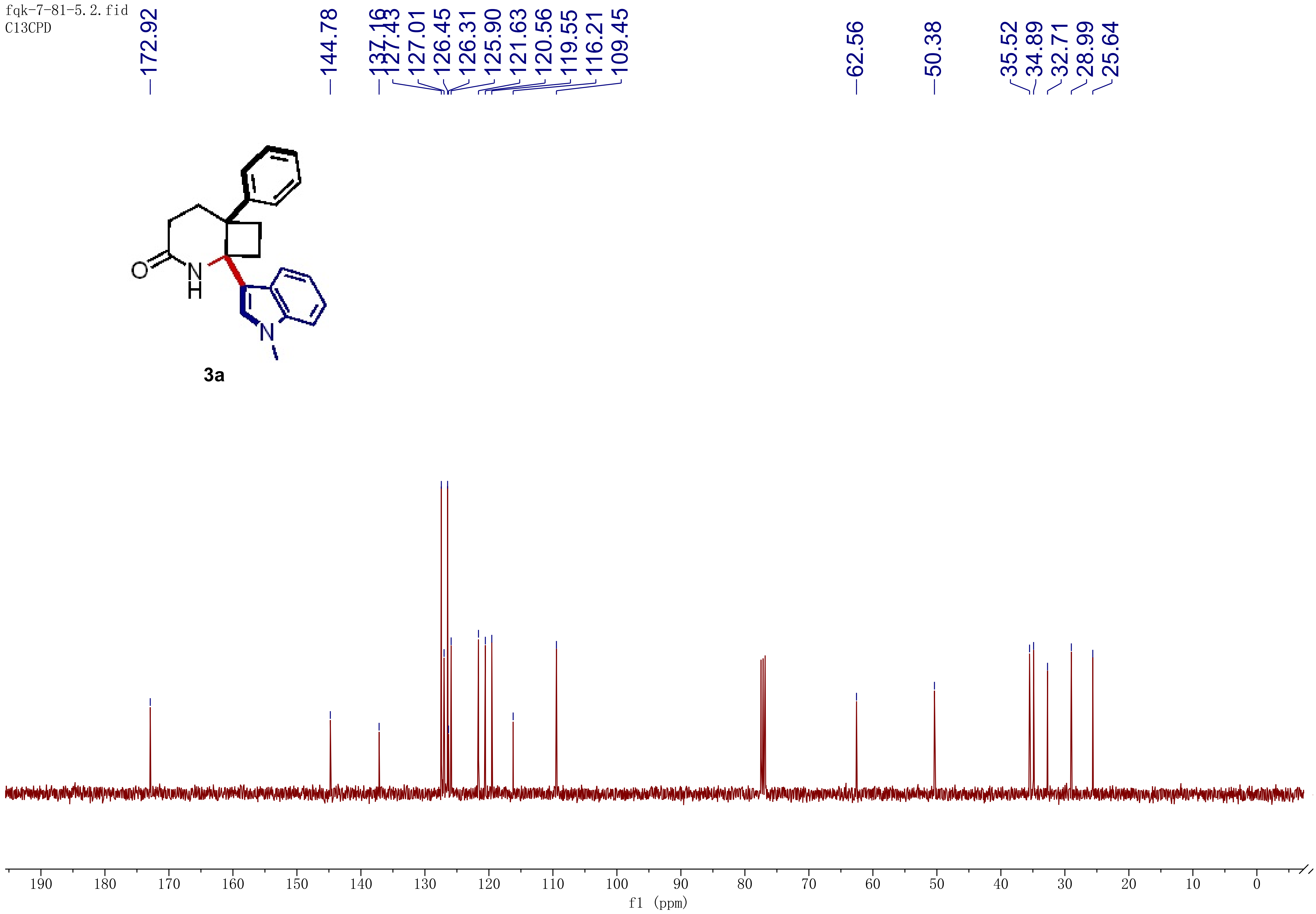
xgc-1-184.2.fid
13CPD

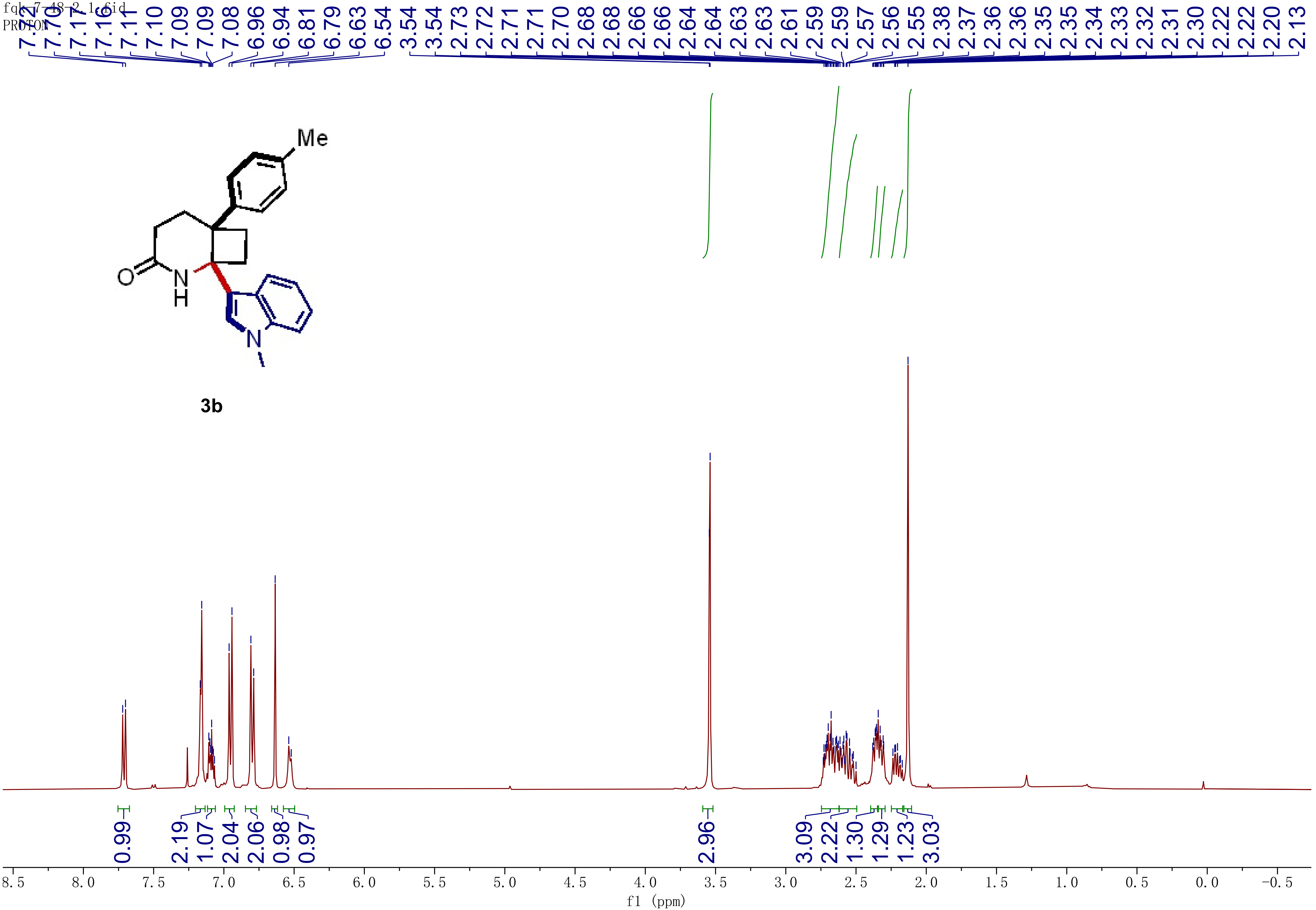


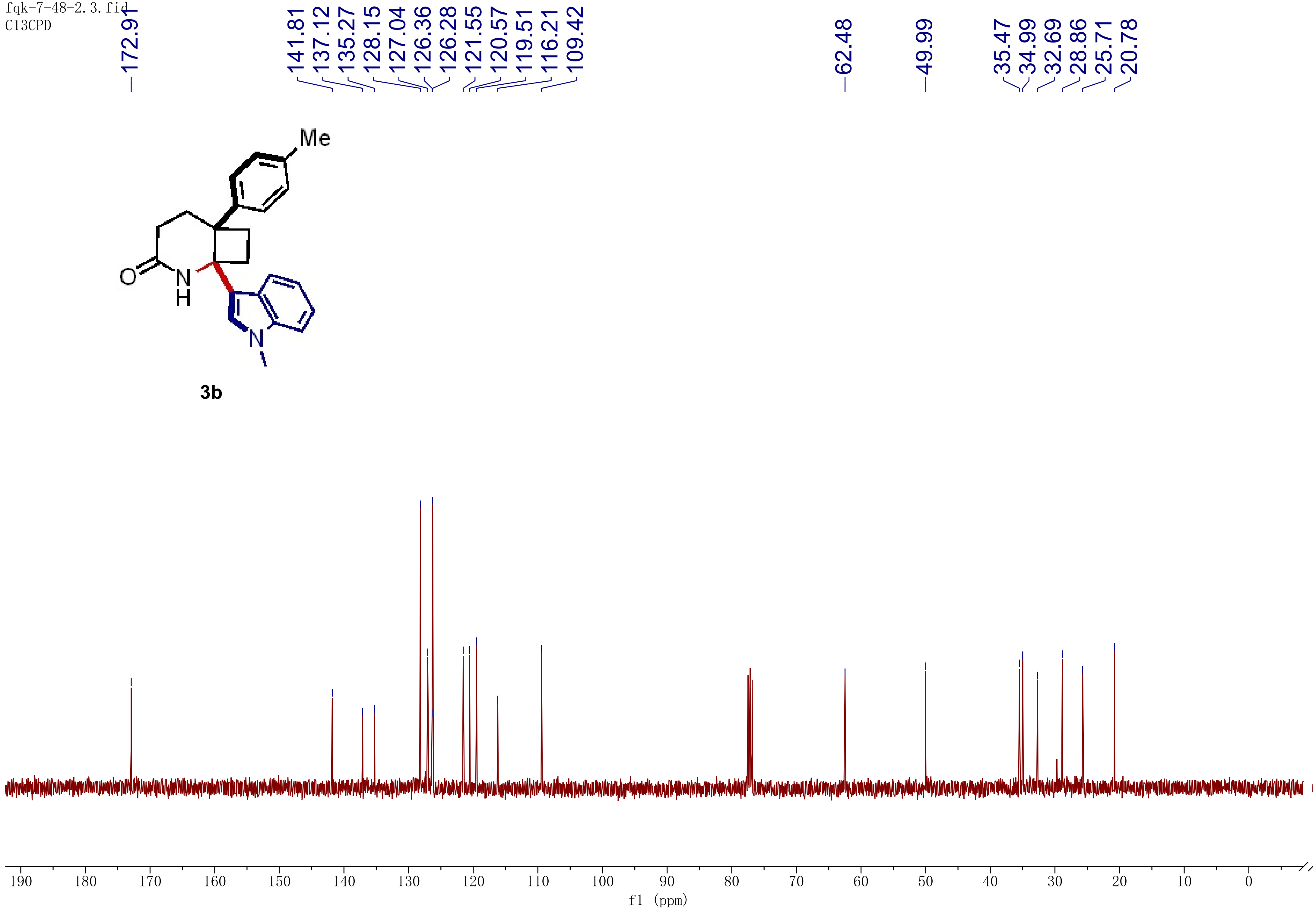

xgc-2-82.1.fid
H1
7.27
7.26
7.25
7.24
7.23
7.18
7.18
7.18
7.17
7.17
7.16
7.15
7.14
7.02
7.01
7.00
6.99
2.73
2.71
2.69
2.48
2.46
2.44
2.22
2.20
2.19
1.90
1.88
1.88
1.87
1.52
1.52
1.51
1.50
1.50
1.49
1.48
1.47
1.38
1.38
1.36
1.35
1y
2.00
1.26
1.98
2.00
2.02
2.00
2.03
4.10
2.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


xgc-2-82.2.fid
1D 13C with H decoupling
166.29
154.12
141.69
139.50
129.08
128.37
128.28
126.65
32.26
30.55
28.71
28.50
28.43
26.69
23.95
1y
O
O
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


fqk-7-81-5.1.fid
PROTON
7.69
7.16
7.16
7.15
7.10
7.09
7.08
7.08
7.08
7.06
7.06
7.00
6.99
6.98
6.98
6.98
6.97
6.97
6.96
6.95
6.61
6.51
3.54
2.74
2.73
2.72
2.71
2.70
2.69
2.69
2.68
2.67
2.67
2.65
2.65
2.63
2.61
2.58
2.56
2.37
2.37
2.36
2.35
2.34
2.33
2.26
2.26
2.24
3a
1.00
1.99
3.00
3.00
1.00
0.97
2.97
4.09
1.06
2.01
1.03
f1 (ppm)


fqk-7-81-5.2.fid
C13CPD
172.92
144.78
137.16
127.43
127.01
126.45
126.31
125.90
121.63
120.56
119.55
116.21
109.45
62.56
50.38
35.52
34.89
32.71
28.99
25.64
O
N
H
N
3a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

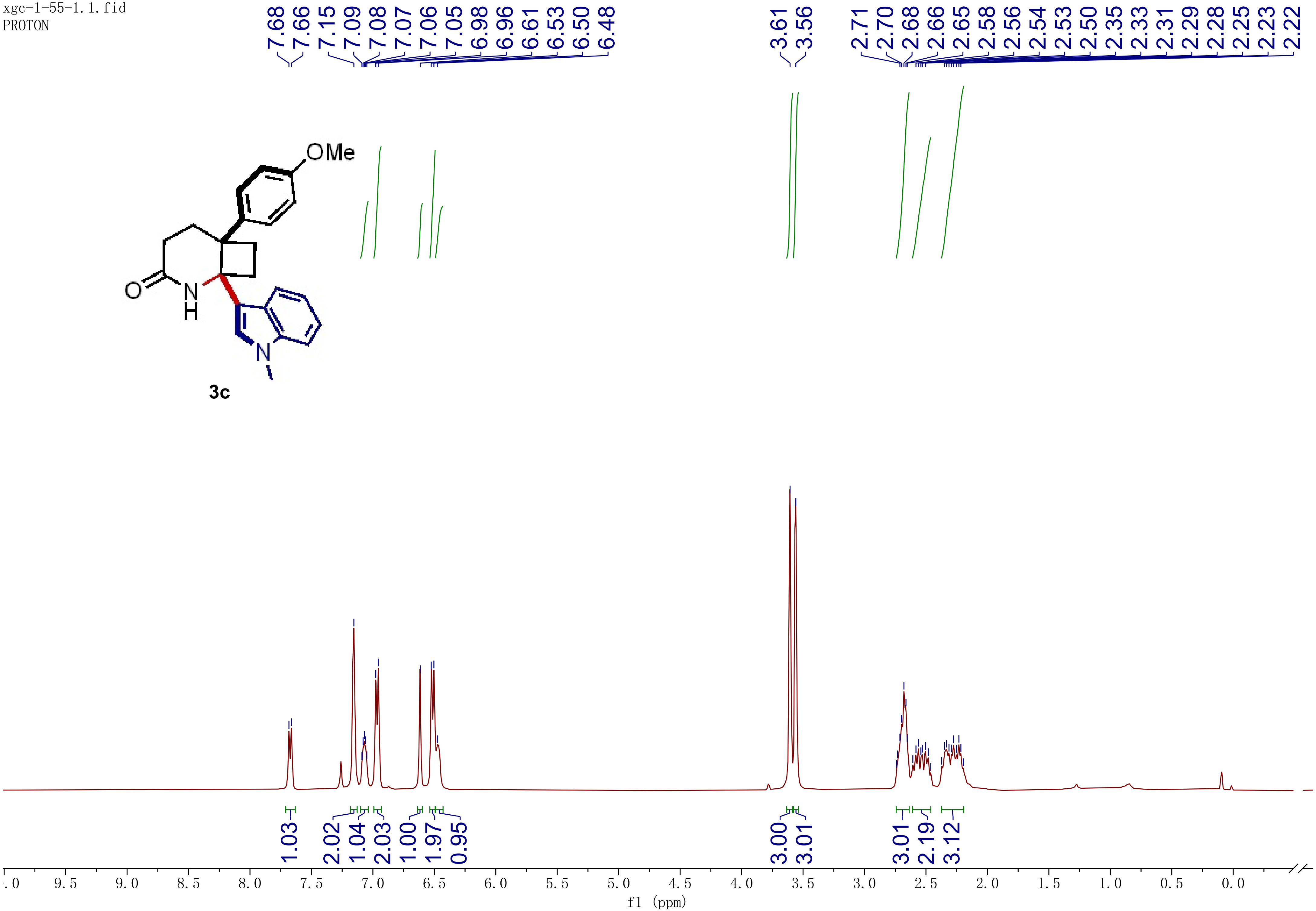

fqk-7-48-2.1.fid
PROTON
7.72
7.70
7.17
7.16
7.11
7.10
7.09
7.09
7.08
6.96
6.94
6.81
6.79
6.63
6.54
3.54
3.54
2.73
2.72
2.71
2.71
2.70
2.68
2.68
2.66
2.66
2.64
2.64
2.63
2.63
2.61
2.59
2.59
2.57
2.56
2.55
2.38
2.37
2.36
2.36
2.35
2.35
2.34
2.33
2.32
2.31
2.30
2.22
2.22
2.20
2.13
Me
O
N
H
N
3b
0.99
2.19
1.07
2.04
2.06
0.98
0.97
2.96
3.09
2.22
1.30
1.29
1.23
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)



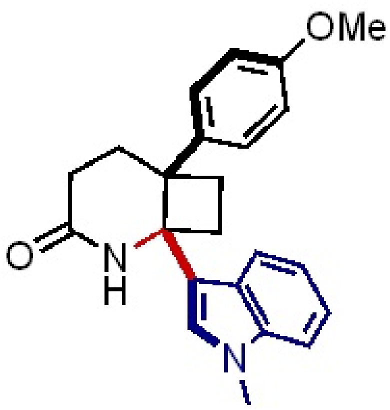
fqk-7-48-2.3.fid
C13CPD
172.91
141.81
137.12
135.27
128.15
127.04
126.36
126.28
121.55
120.57
119.51
116.21
109.42
62.48
49.99
35.47
34.99
32.69
28.86
25.71
20.78
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
Me
O
N
H
N

3b

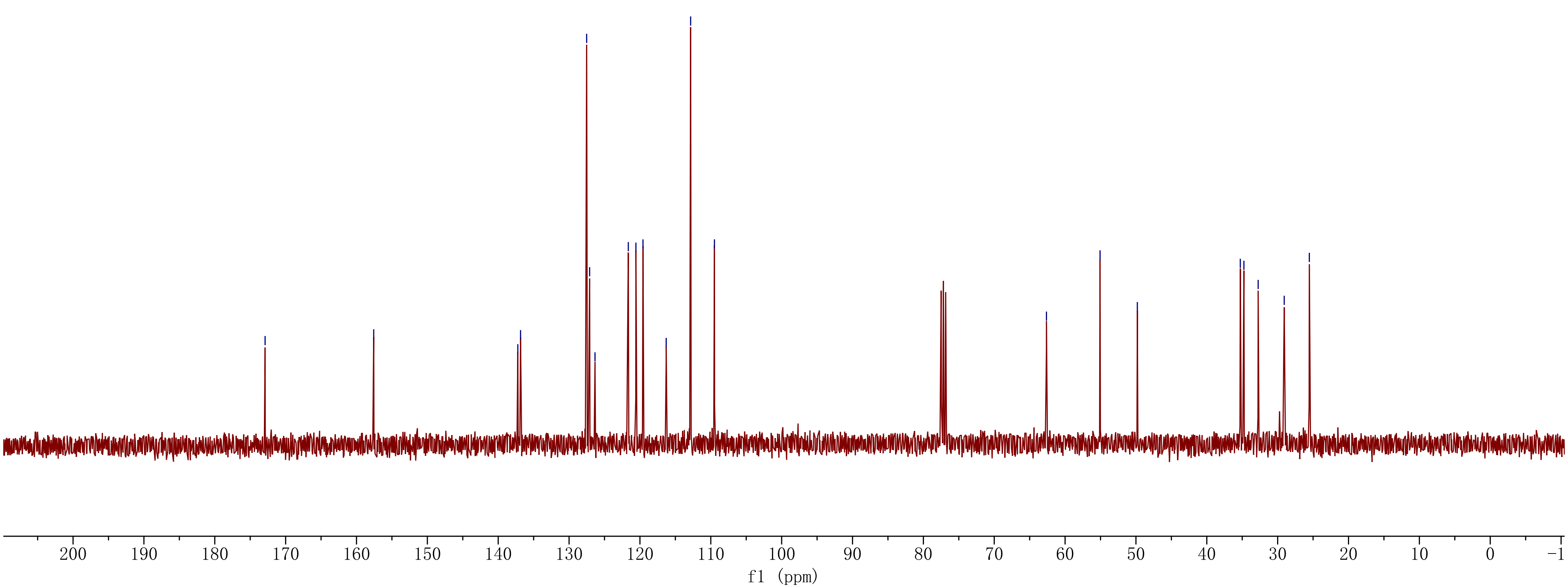
xgc-1-55-1.1.fid
PROTON

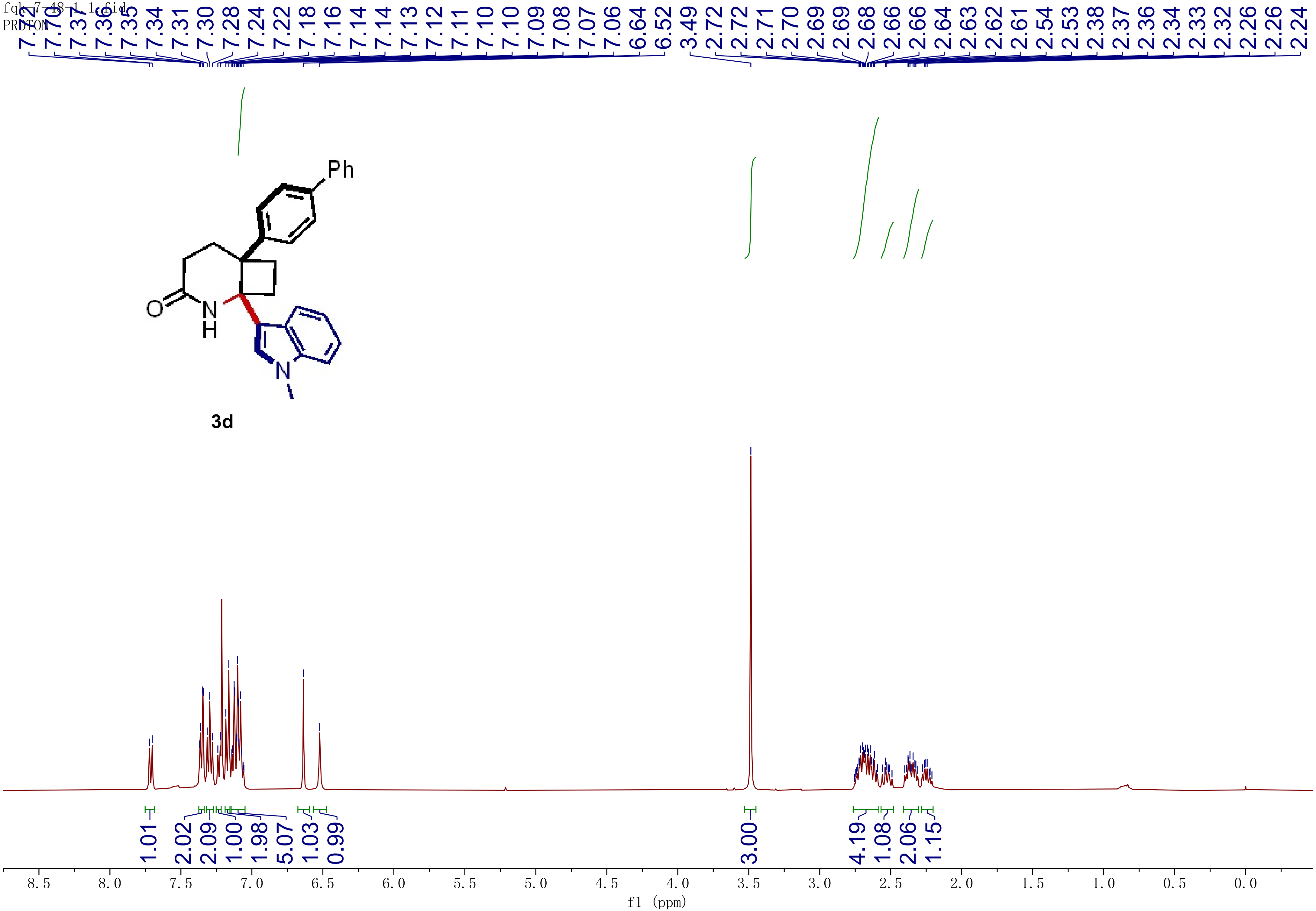


xgc-1-55-1.2.fid
C13CPD



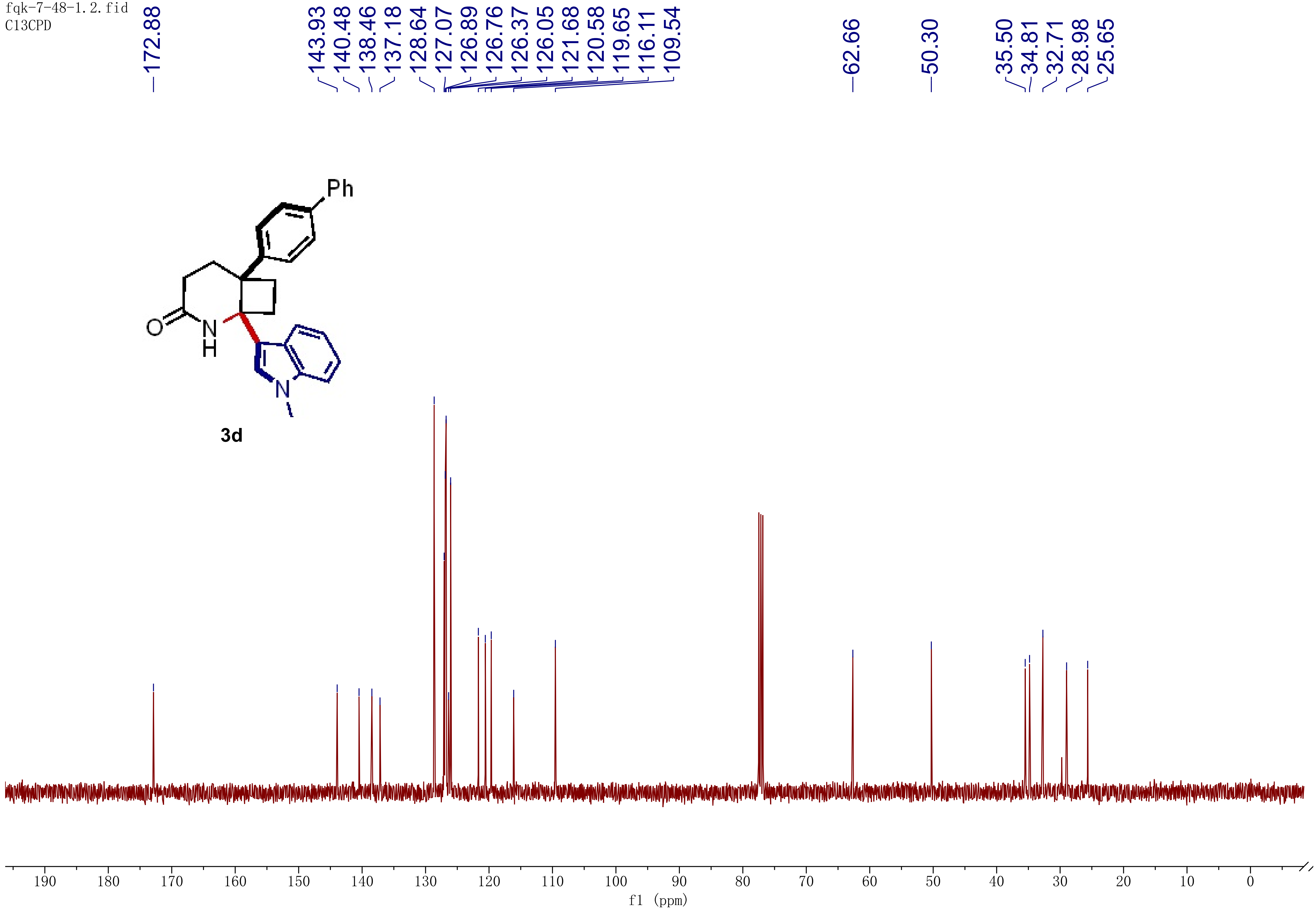
3c

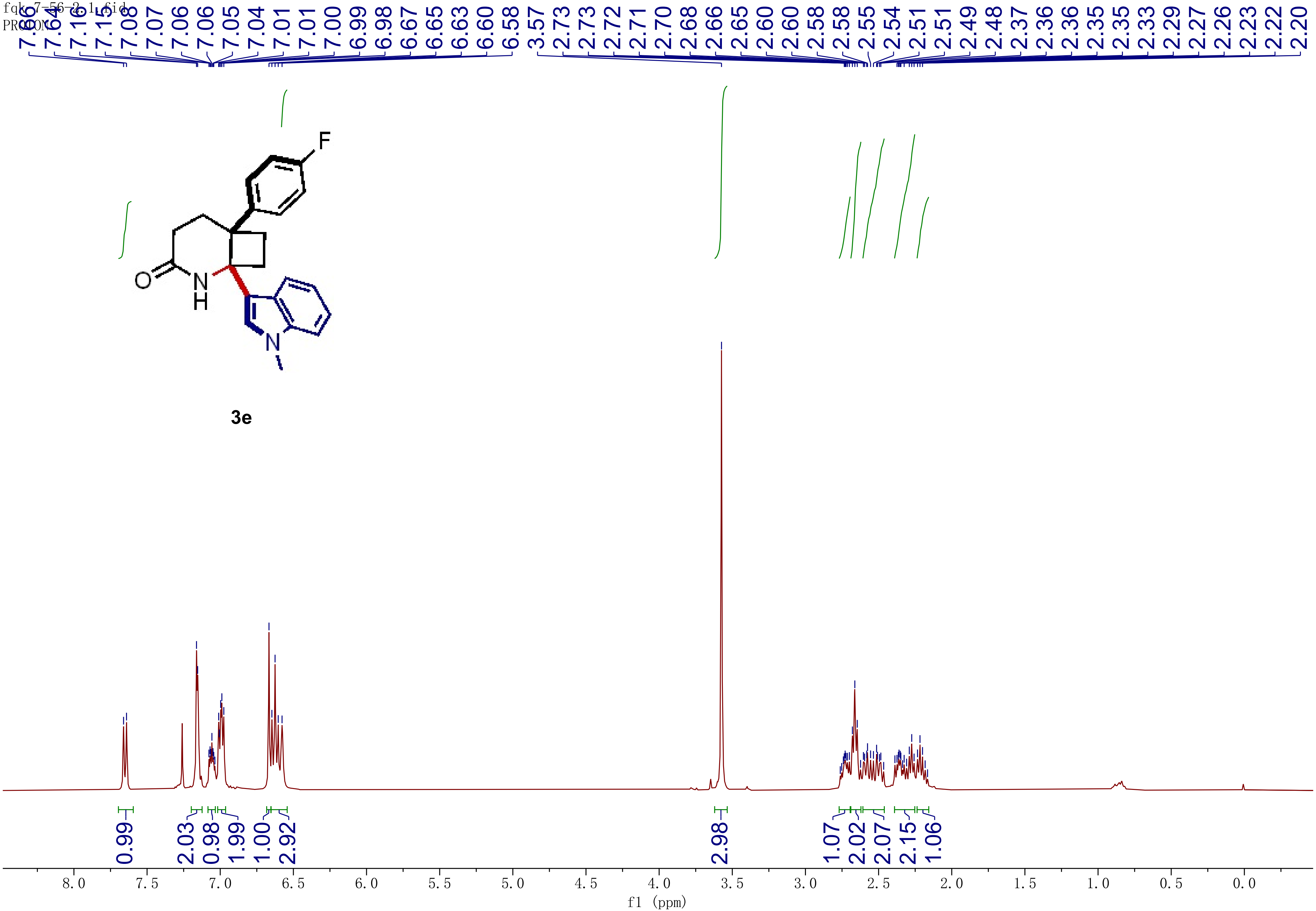


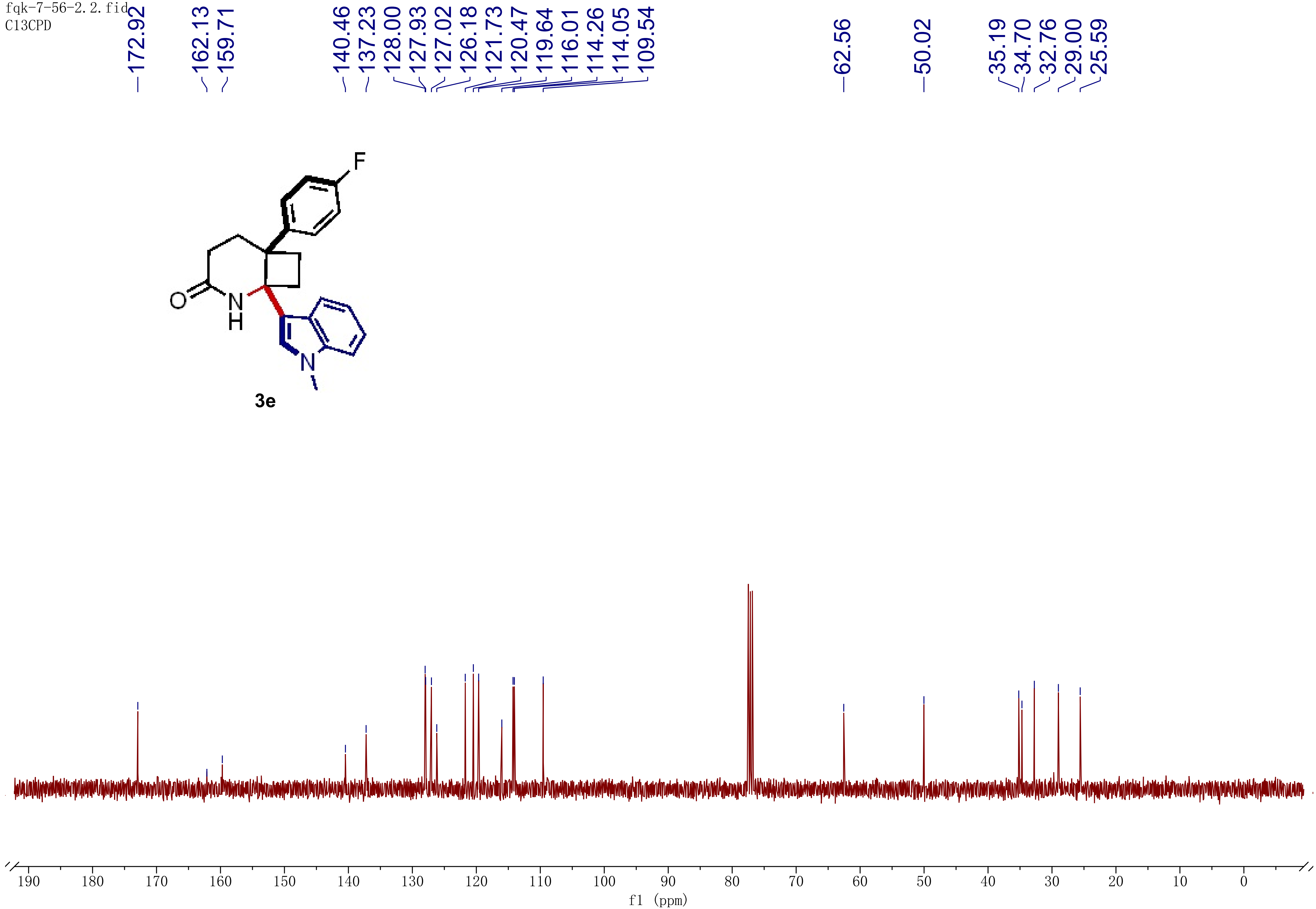


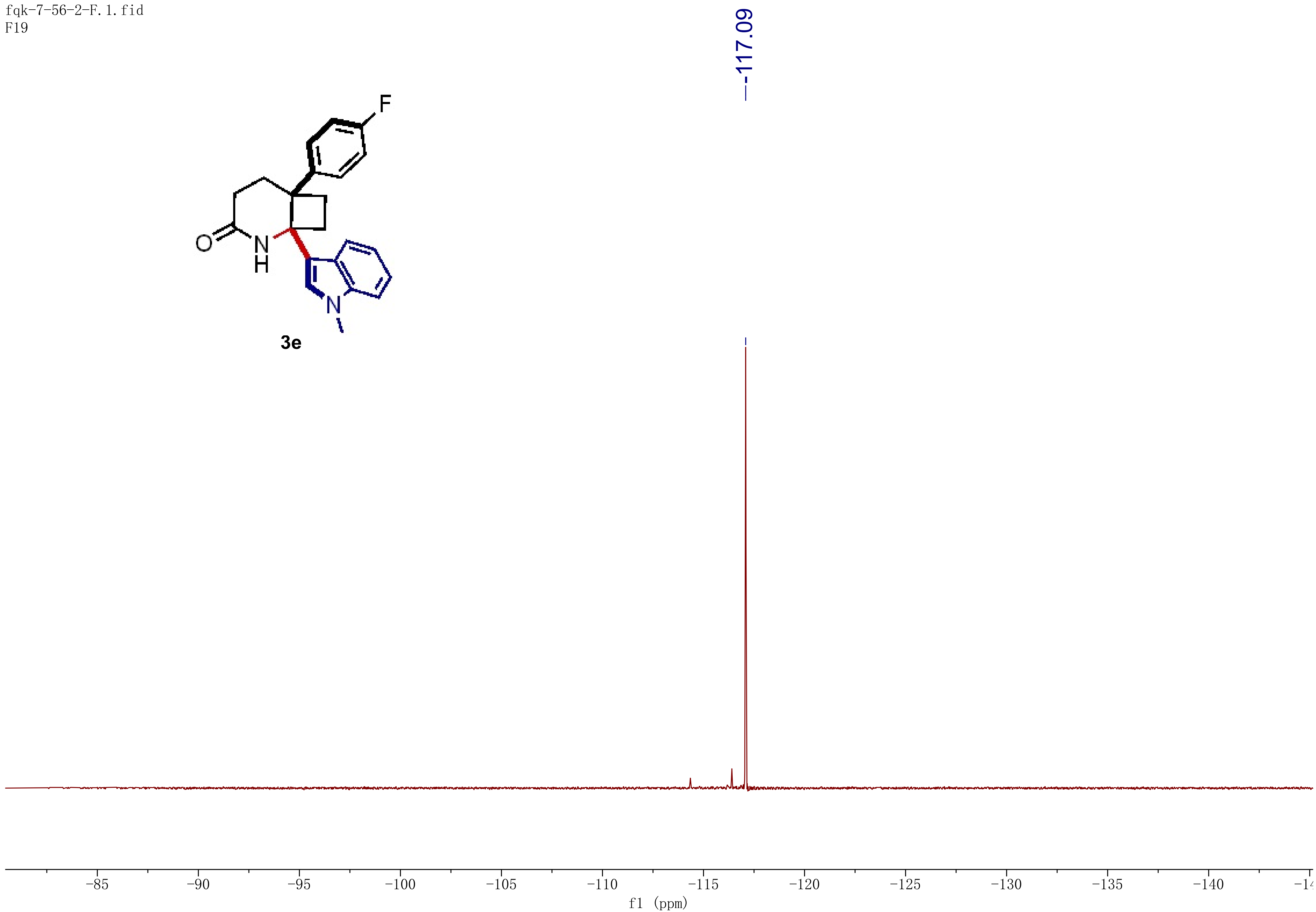
fqk-7-48-1.1.fid
PROTON
7.72 7.70 7.37 7.36 7.35 7.34 7.31 7.30 7.28 7.24 7.22 7.18 7.16 7.14 7.14 7.13 7.12 7.11 7.10 7.10 7.09 7.08 7.07 7.06 6.64 6.52 3.49 2.72 2.72 2.71 2.70 2.69 2.69 2.68 2.66 2.66 2.64 2.63 2.62 2.61 2.54 2.53 2.38 2.37 2.36 2.34 2.33 2.32 2.26 2.26 2.24
Ph
O
N
H
N
1.01 2.02 2.09 1.00 1.98 5.07 1.03 0.99 3.00 4.19 1.08 2.06 1.15
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

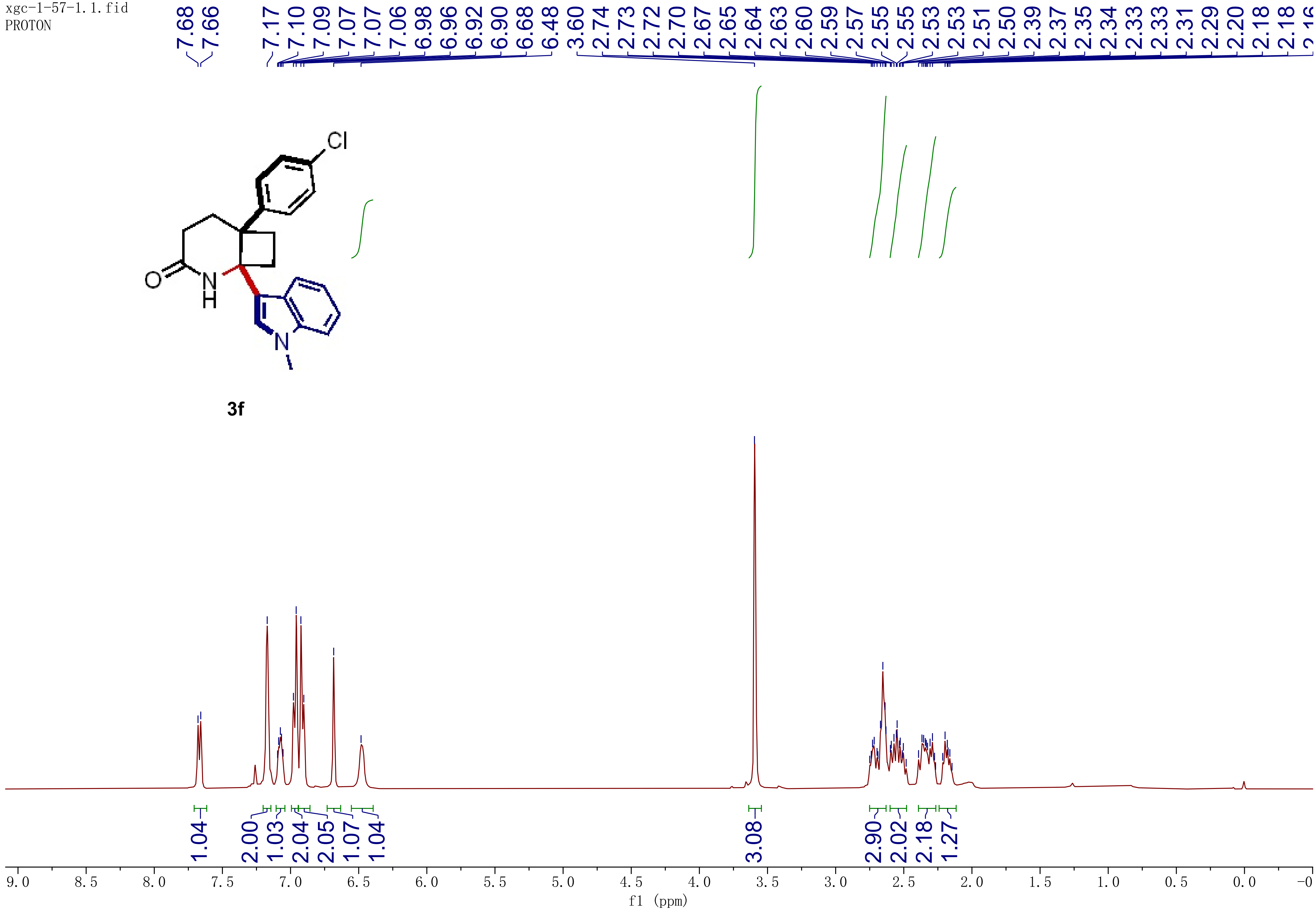
3d


fqk-7-48-1.2.fid
C13CPD
172.88
143.93
140.48
138.46
137.18
128.64
127.07
126.89
126.76
126.37
126.05
121.68
120.58
119.65
116.11
109.54
62.66
50.30
35.50
34.81
32.71
28.98
25.65
Ph
O
N
H
N
3d
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

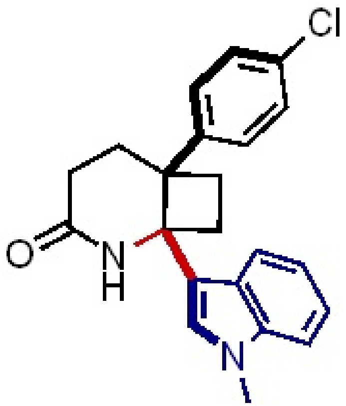

fqk-7-56-2.1.fid
PROTON
7.66 7.64 7.16 7.15 7.08 7.07 7.06 7.06 7.05 7.04 7.01 7.01 7.00 6.99 6.98 6.67 6.65 6.63 6.60 6.58 3.57 2.73 2.73 2.72 2.71 2.70 2.68 2.66 2.65 2.60 2.60 2.58 2.58 2.55 2.54 2.51 2.51 2.49 2.48 2.37 2.36 2.36 2.35 2.35 2.33 2.29 2.27 2.26 2.23 2.22 2.20
3e
0.99 2.03 0.98 1.99 1.00 2.92 2.98 1.07 2.02 2.07 2.15 1.06
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


fqk-7-56-2.2.fid
C13CPD
172.92
162.13
159.71
140.46
137.23
128.00
127.93
127.02
126.18
121.73
120.47
119.64
116.01
114.26
114.05
109.54
62.56
50.02
35.19
34.70
32.76
29.00
25.59
F
O
N
H
N
3e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

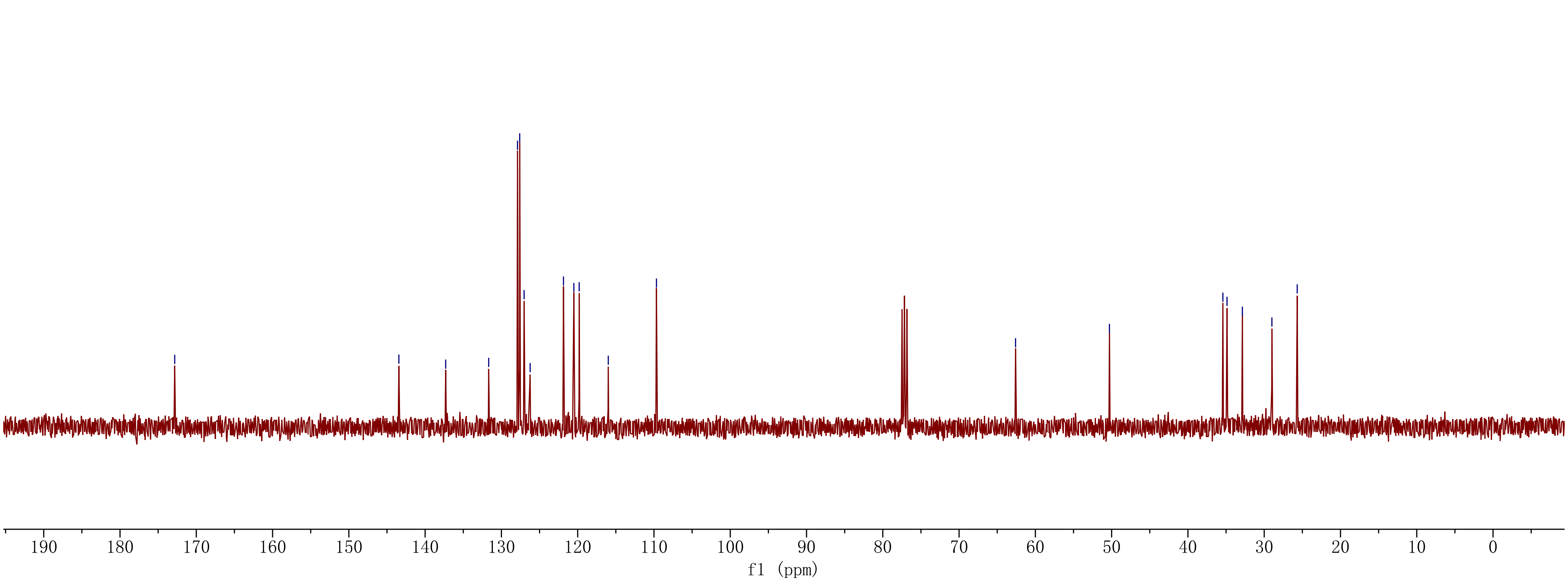

fqk-7-56-2-F.1.fid
F19
-117.09
F
O
N
H
N
3e
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
-140
f1 (ppm)


xgc-1-57-1.1.fid
PROTON
7.68 7.66 7.17 7.10 7.09 7.07 7.07 7.06 6.98 6.96 6.92 6.90 6.68 6.48 3.60 2.74 2.73 2.72 2.70 2.67 2.65 2.64 2.63 2.60 2.59 2.57 2.55 2.55 2.53 2.53 2.51 2.50 2.39 2.37 2.35 2.34 2.33 2.33 2.31 2.29 2.20 2.18 2.18
Cl
O
N
H
N
3f
1.04 2.00 1.03 2.04 2.05 1.07 1.04 3.08 2.90 2.02 2.18 1.27
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

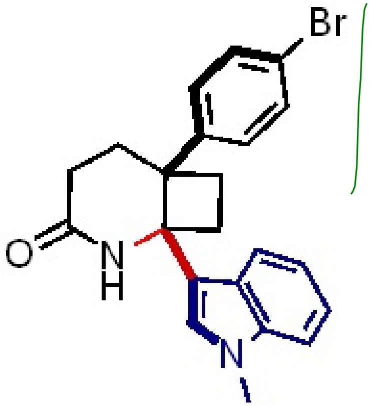
xgc-1-57-1.2.fid
C13CPD



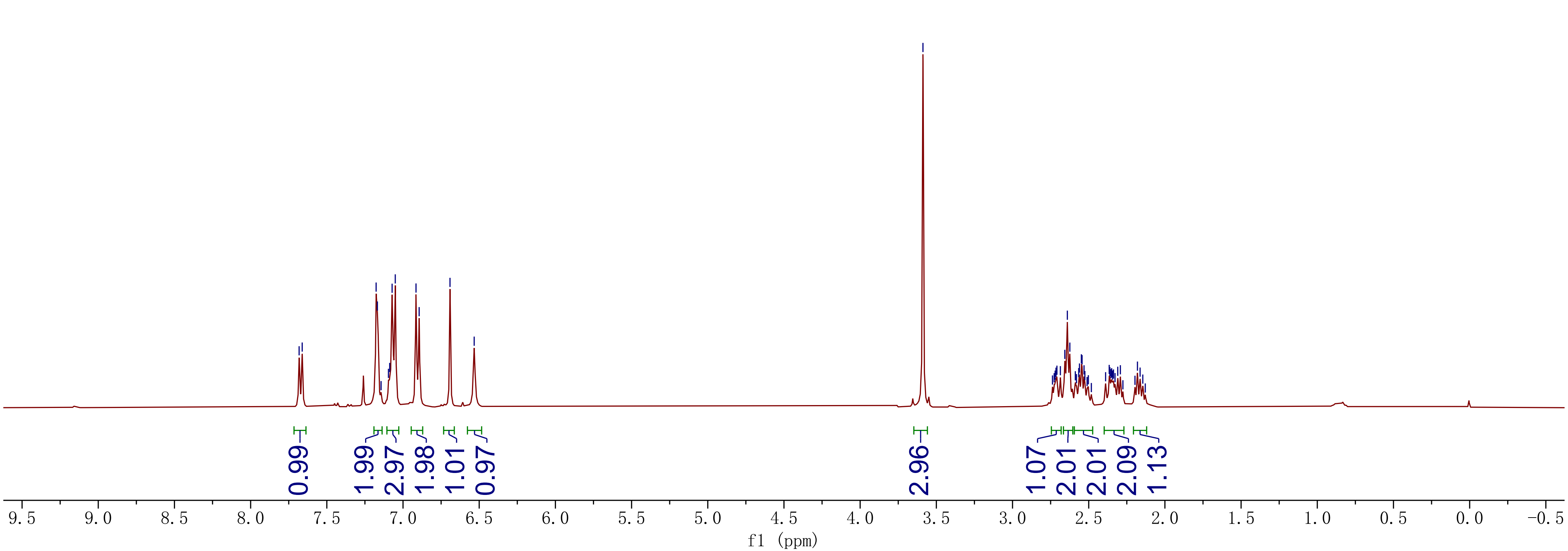
3f



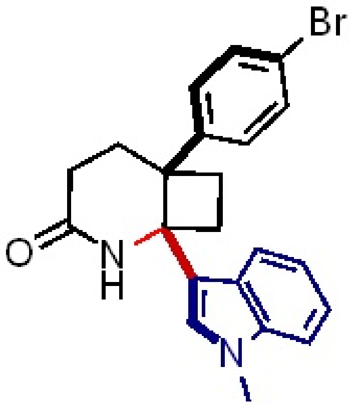
fqk-7-56-3.1.fid
PROTON



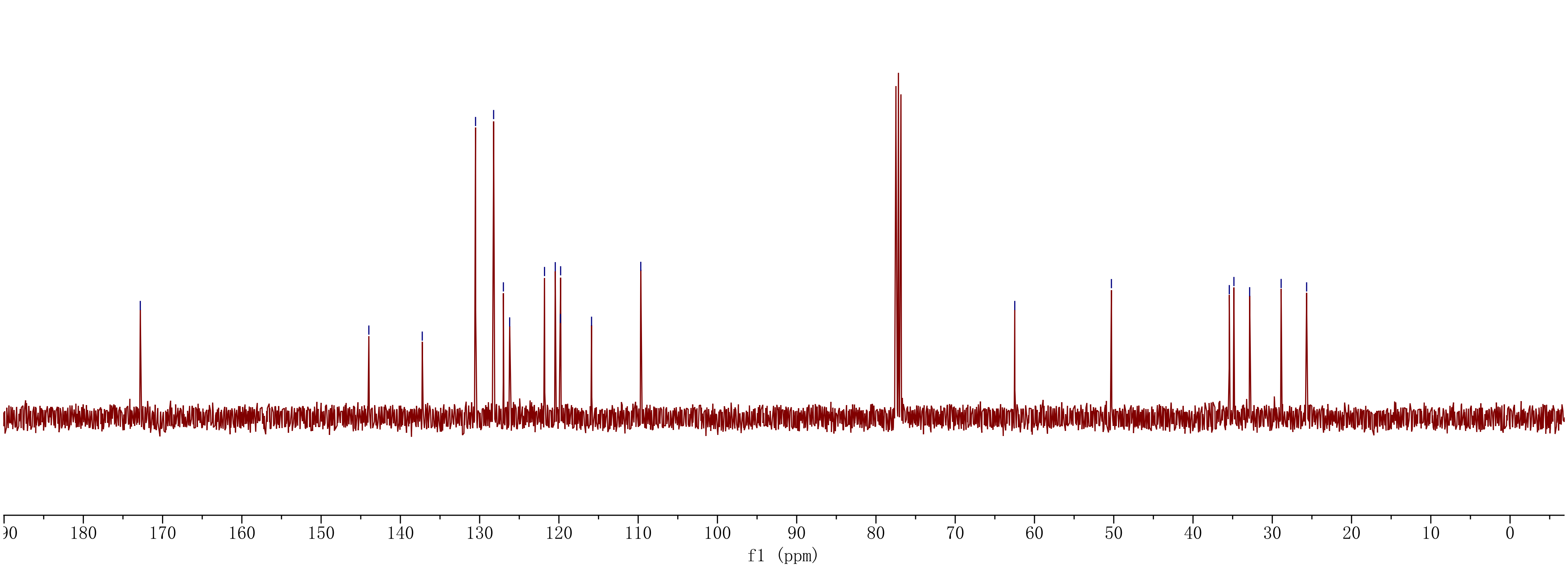
3g

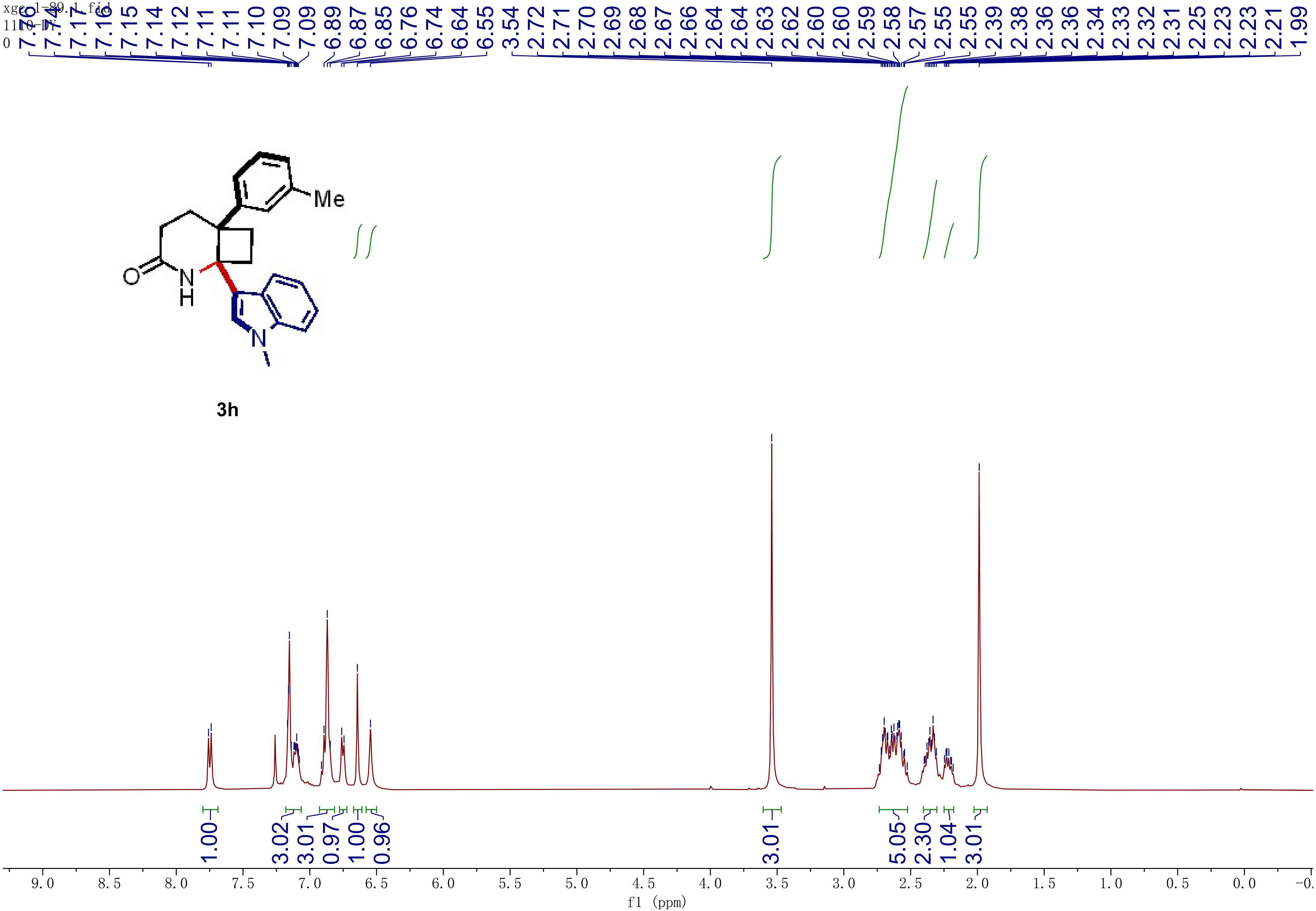


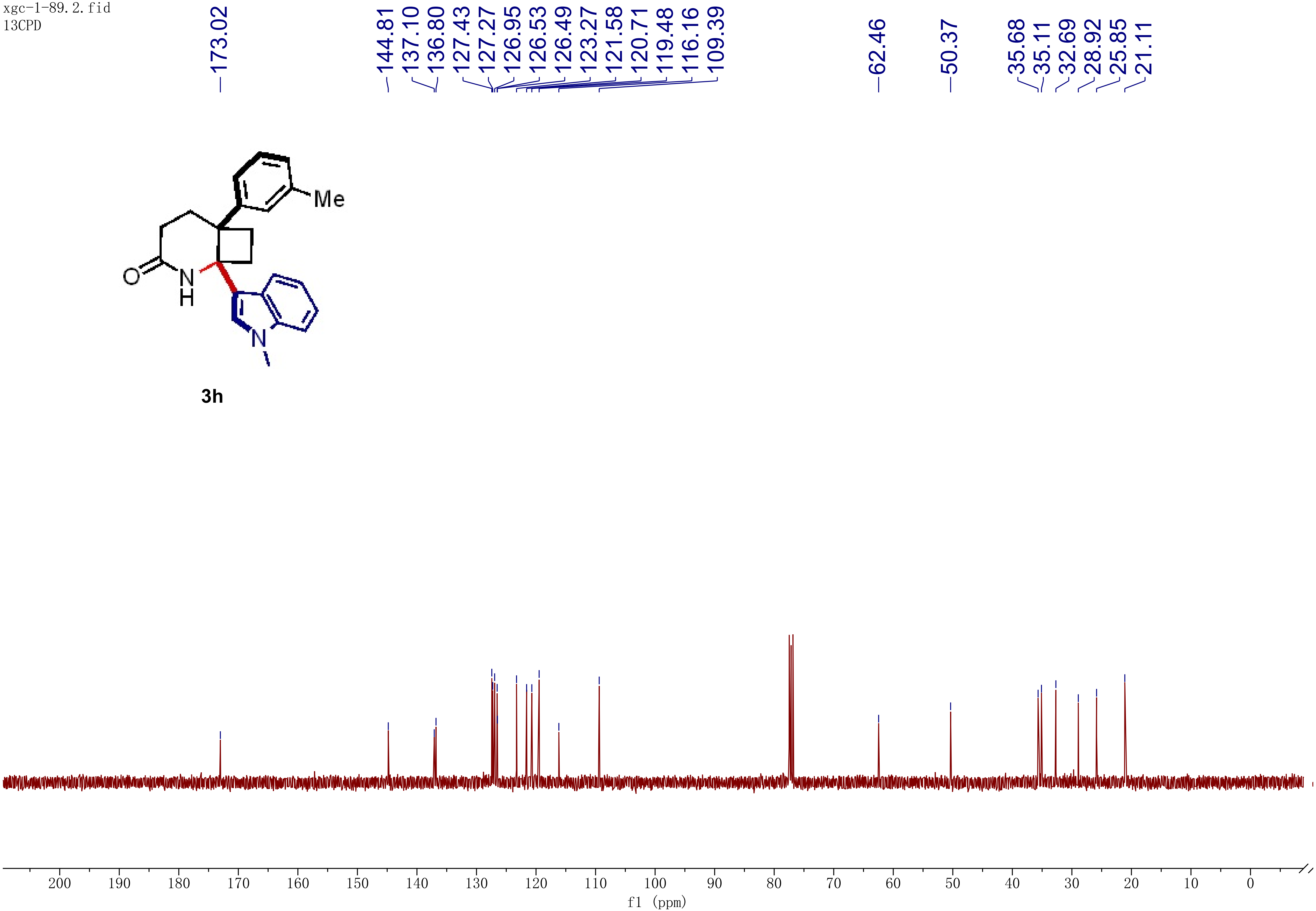
fqk-7-56-3.2.fid
C13CPD



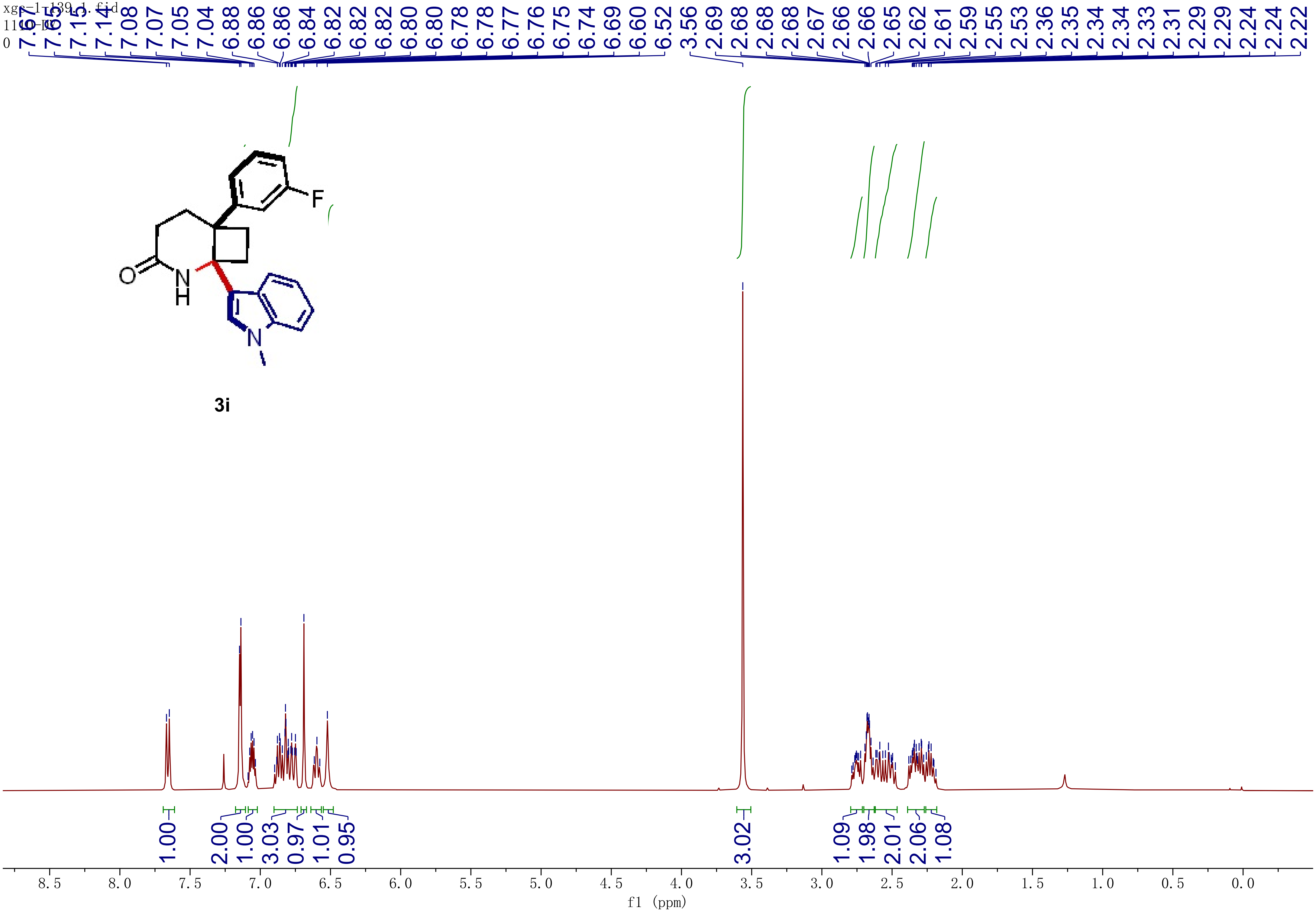
3g

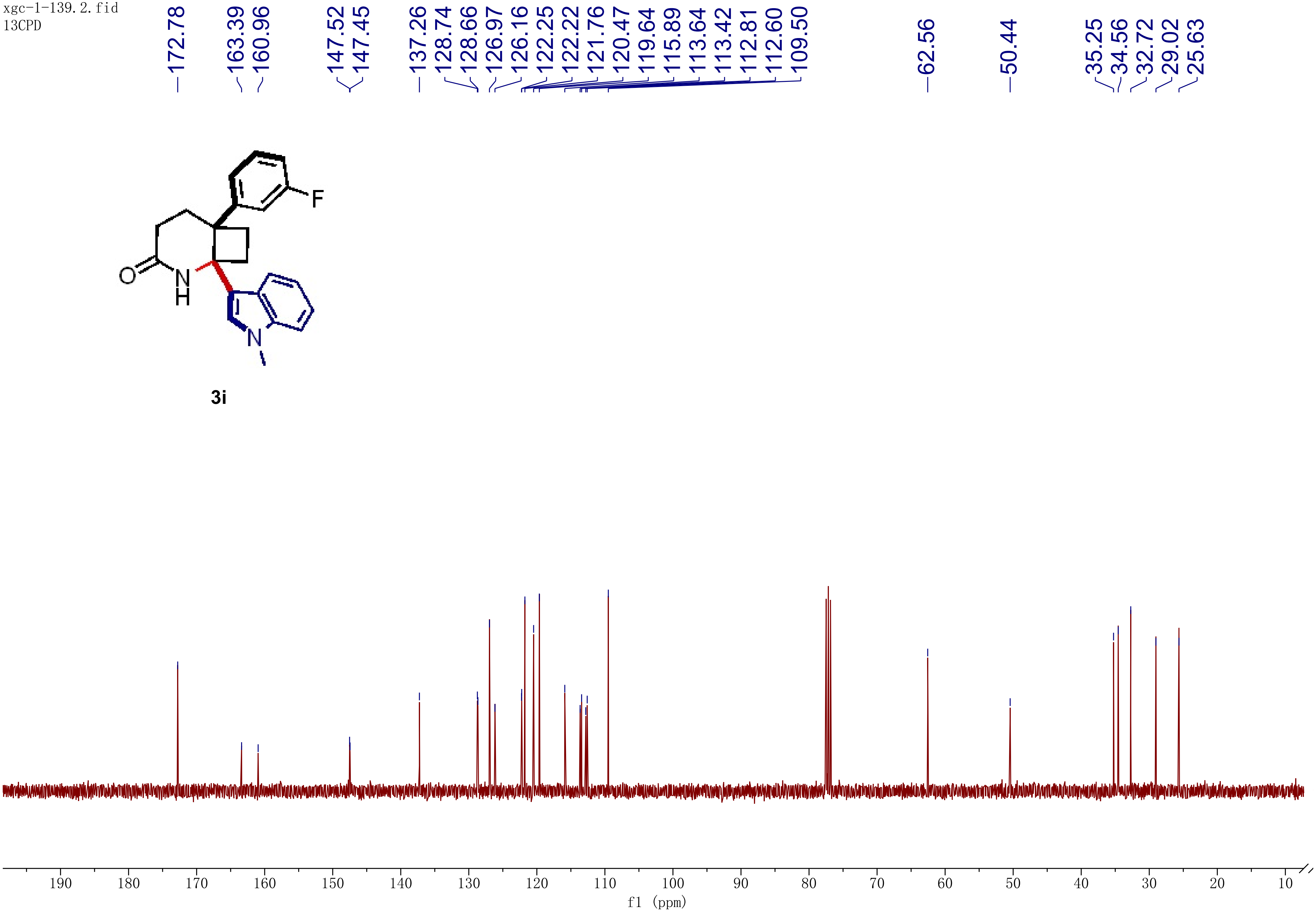



xge-1-89.1.fid
1110-DY
7.76 7.74 7.17 7.16 7.15 7.14 7.12 7.11 7.11 7.10 7.09 7.09 6.89 6.87 6.85 6.76 6.74 6.64 6.55 3.54 2.72 2.71 2.70 2.69 2.68 2.67 2.66 2.64 2.64 2.63 2.62 2.60 2.60 2.59 2.58 2.57 2.55 2.55 2.39 2.38 2.36 2.36 2.34 2.33 2.32 2.31 2.25 2.23 2.23 2.21 1.99
Me
O
N
H
N
3h
1.00 3.02 3.01 0.97 1.00 0.96 3.01 5.05 2.30 1.04 3.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

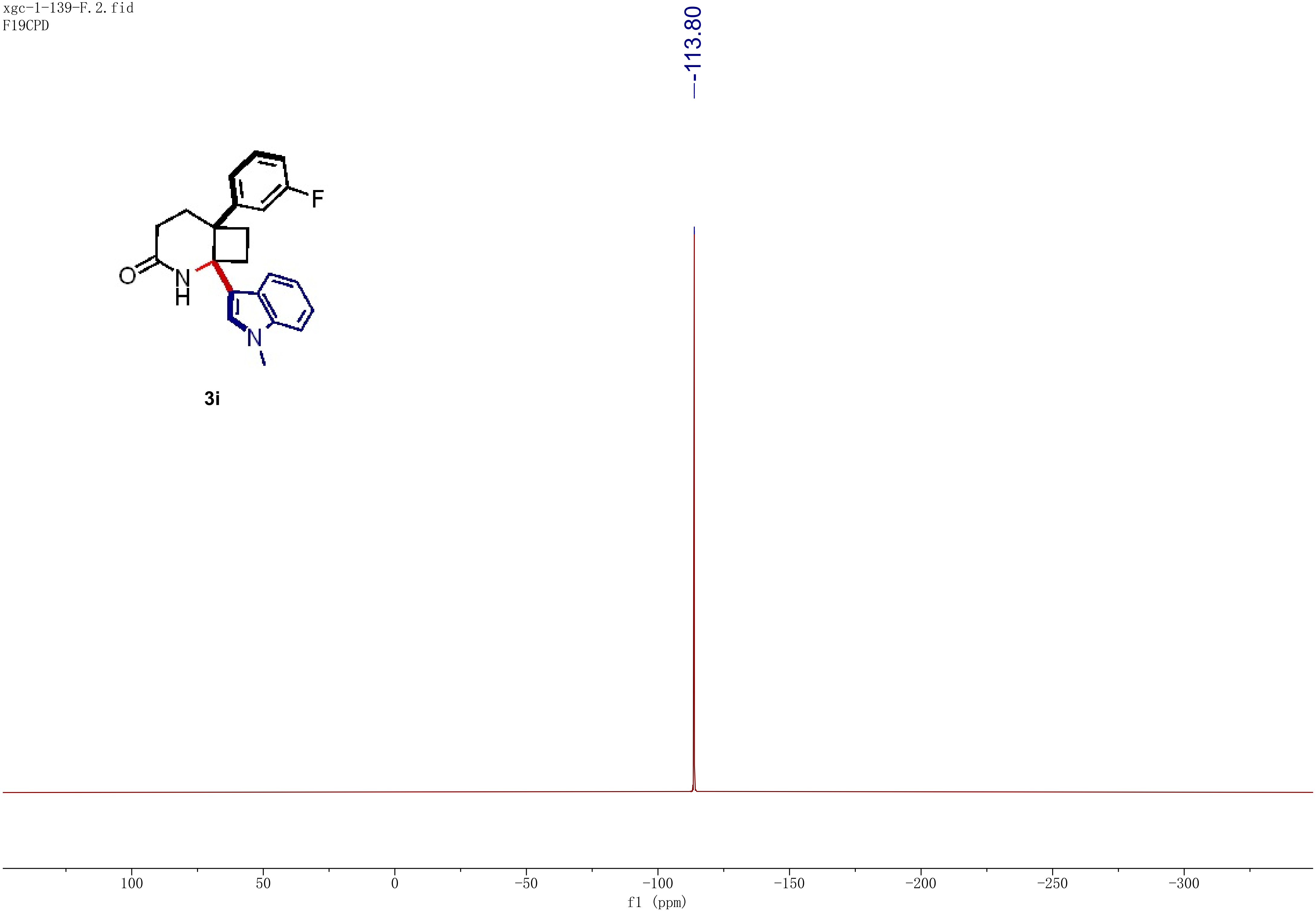

xgc-1-89.2.fid
13CPD
173.02
144.81
137.10
136.80
127.43
127.27
126.95
126.53
126.49
123.27
121.58
120.71
119.48
116.16
109.39
62.46
50.37
35.68
35.11
32.69
28.92
25.85
21.11
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
Me
O
N
H
N

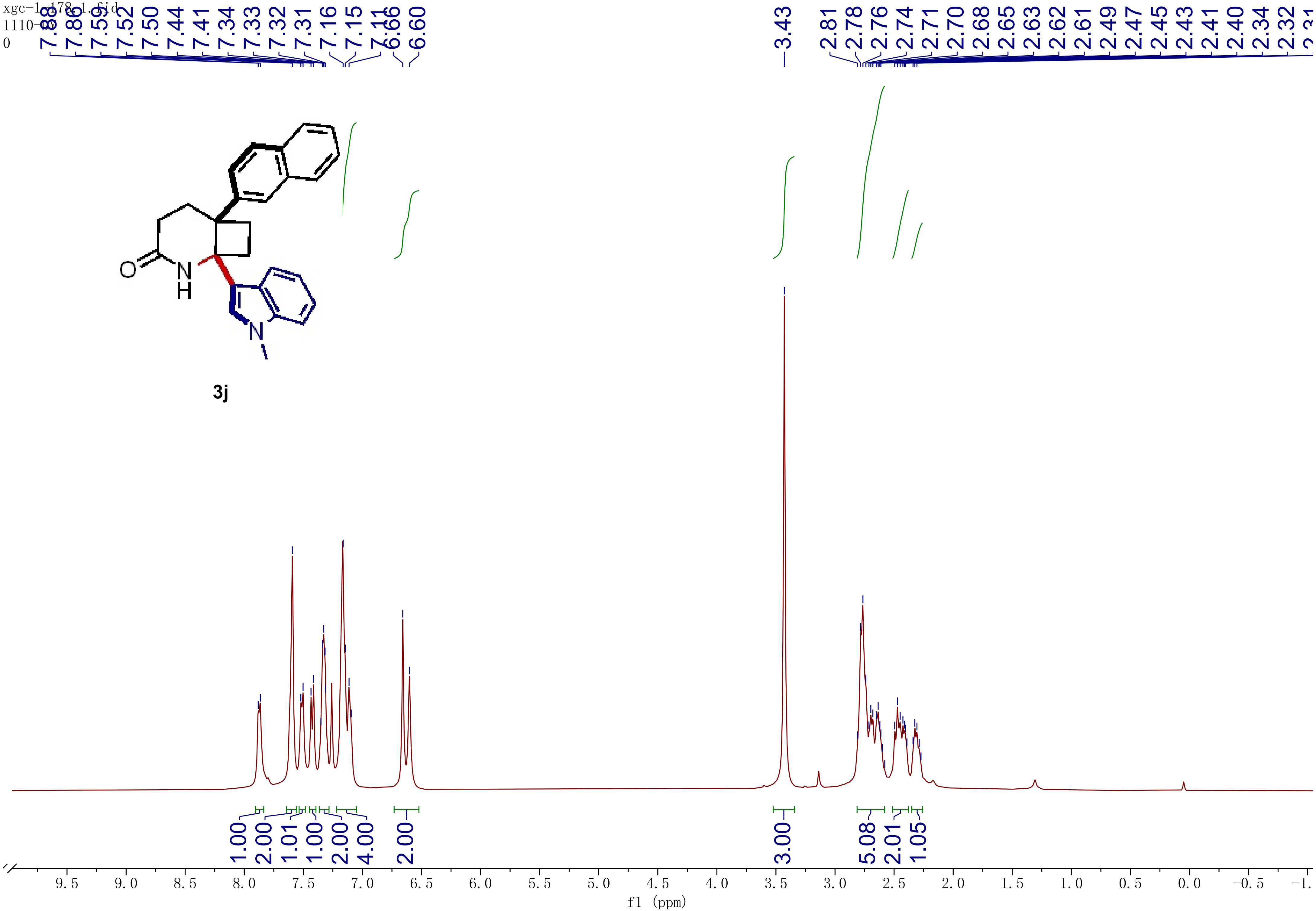
3h


xgs-1-139.1.fid
1100-H
0
7.67 7.65 7.15 7.14 7.08 7.07 7.05 7.04 6.88 6.86 6.86 6.84 6.82 6.82 6.82 6.80 6.80 6.78 6.78 6.77 6.76 6.75 6.74 6.69 6.60 6.52 3.56 2.69 2.68 2.68 2.68 2.67 2.66 2.66 2.65 2.62 2.61 2.59 2.55 2.53 2.36 2.35 2.34 2.34 2.33 2.31 2.29 2.29 2.24 2.24 2.22
F
O
N
H
N
3i
1.00 2.00 1.00 3.03 0.97 1.01 0.95 3.02 1.09 1.98 2.01 2.06 1.08
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

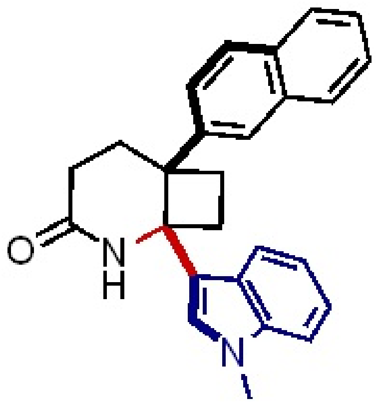

xgc-1-139.2.fid
13CPD
172.78
163.39
160.96
147.52
147.45
137.26
128.74
128.66
126.97
126.16
122.25
122.22
121.76
120.47
119.64
115.89
113.64
113.42
112.81
112.60
109.50
62.56
50.44
35.25
34.56
32.72
29.02
25.63
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)
F
O
N
H
N

3i

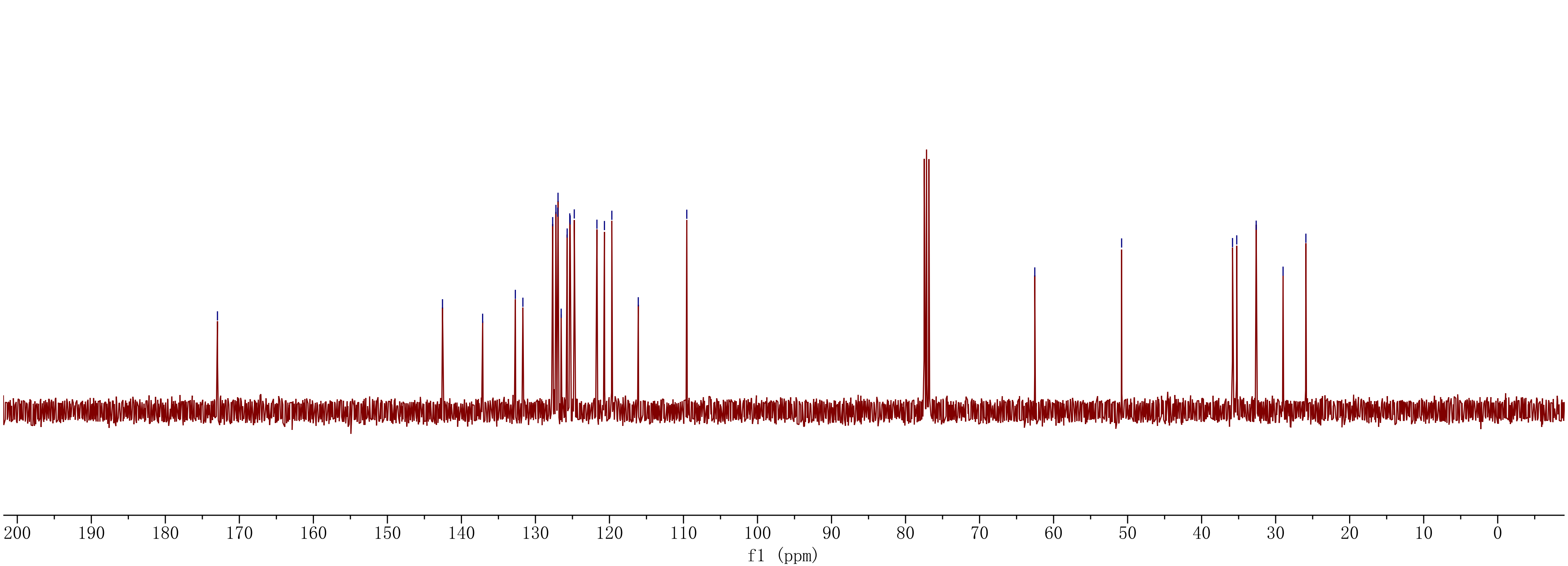

xgc-1-139-F.2.fid
F19CPD
-113.80
F
O
N
H
N
3i
100
50
0
−50
−100
−150
−200
−250
−300
f1 (ppm)

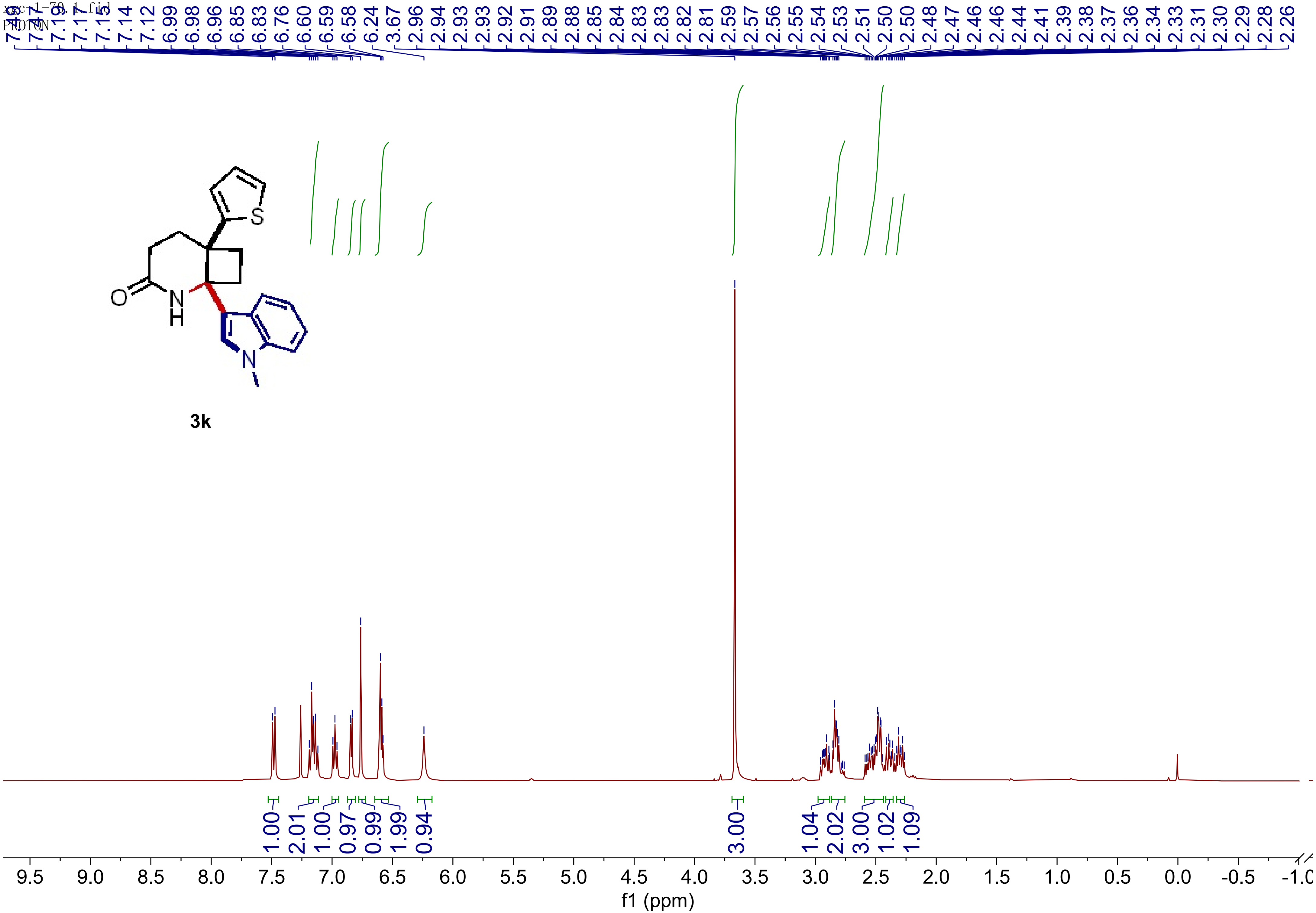

xgc-1-178-1.fid
1110-00
0
7.88
7.86
7.59
7.52
7.50
7.44
7.41
7.34
7.33
7.32
7.31
7.16
7.15
7.11
6.66
6.60
3.43
2.81
2.78
2.76
2.74
2.71
2.70
2.68
2.65
2.63
2.62
2.61
2.49
2.47
2.45
2.43
2.41
2.40
2.34
2.32
O
N
H
N
3j
1.00
2.00
1.01
1.00
2.00
4.00
2.00
3.00
5.08
2.01
1.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

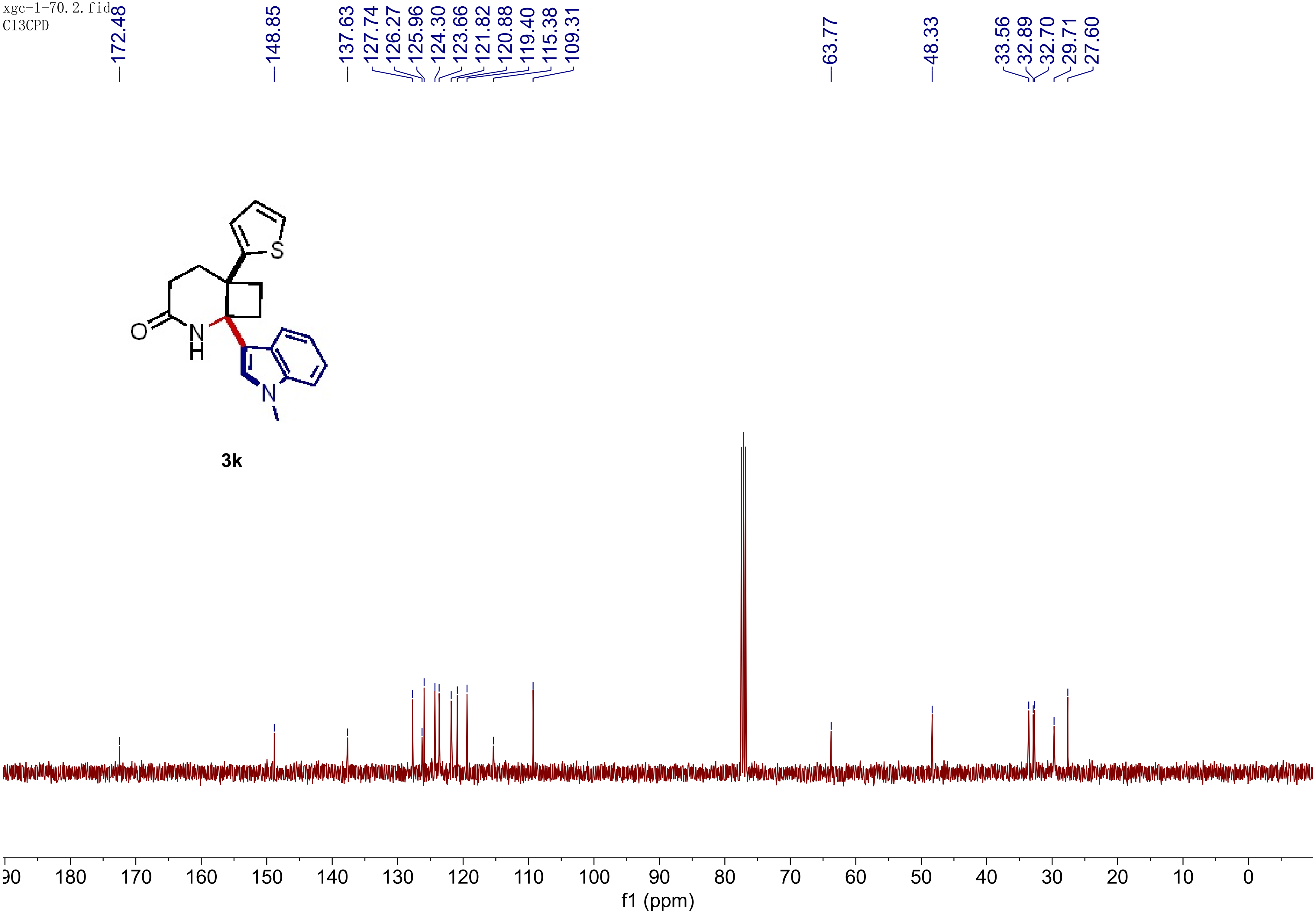
xgc-1-178.2.fid
13CPD



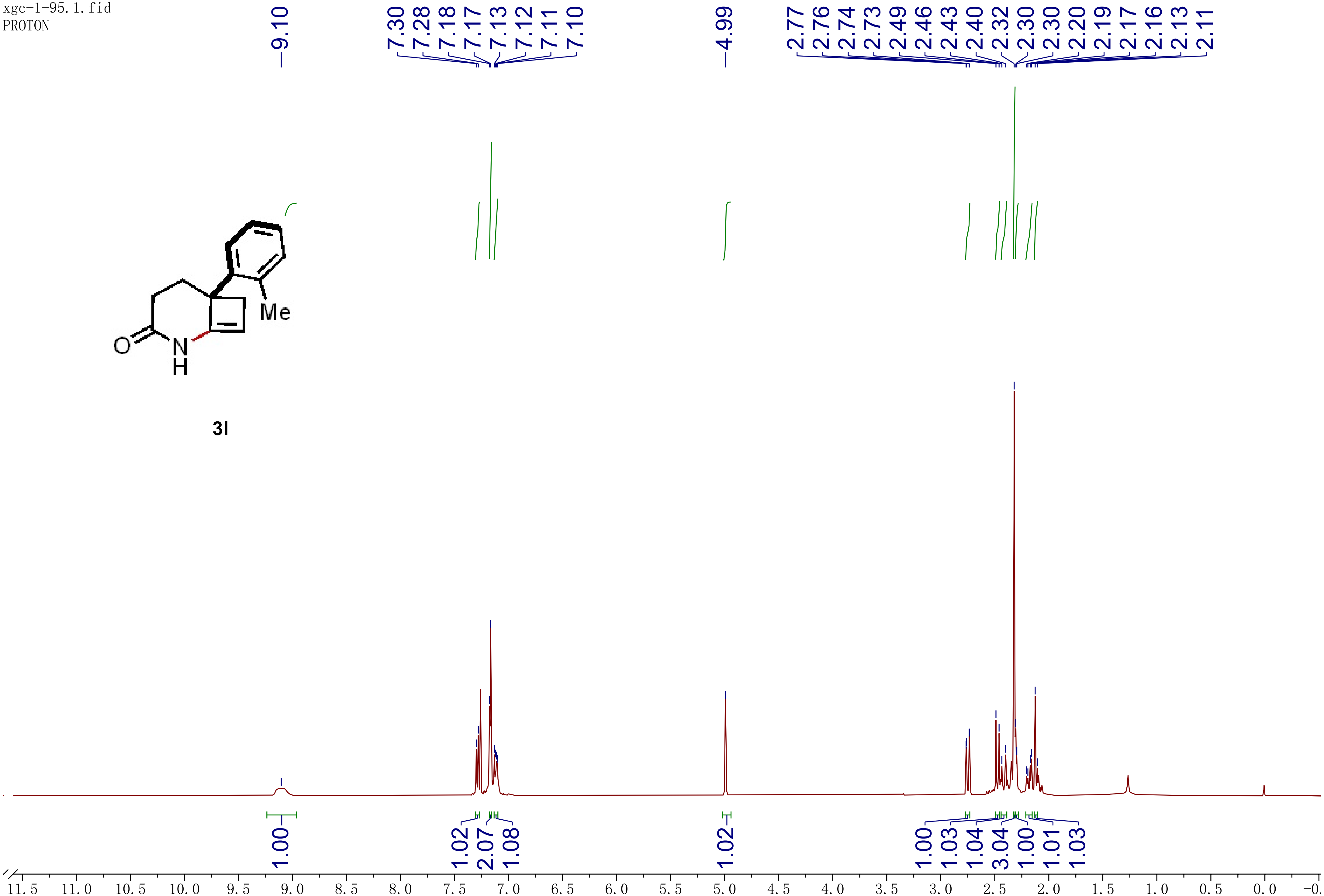
3j



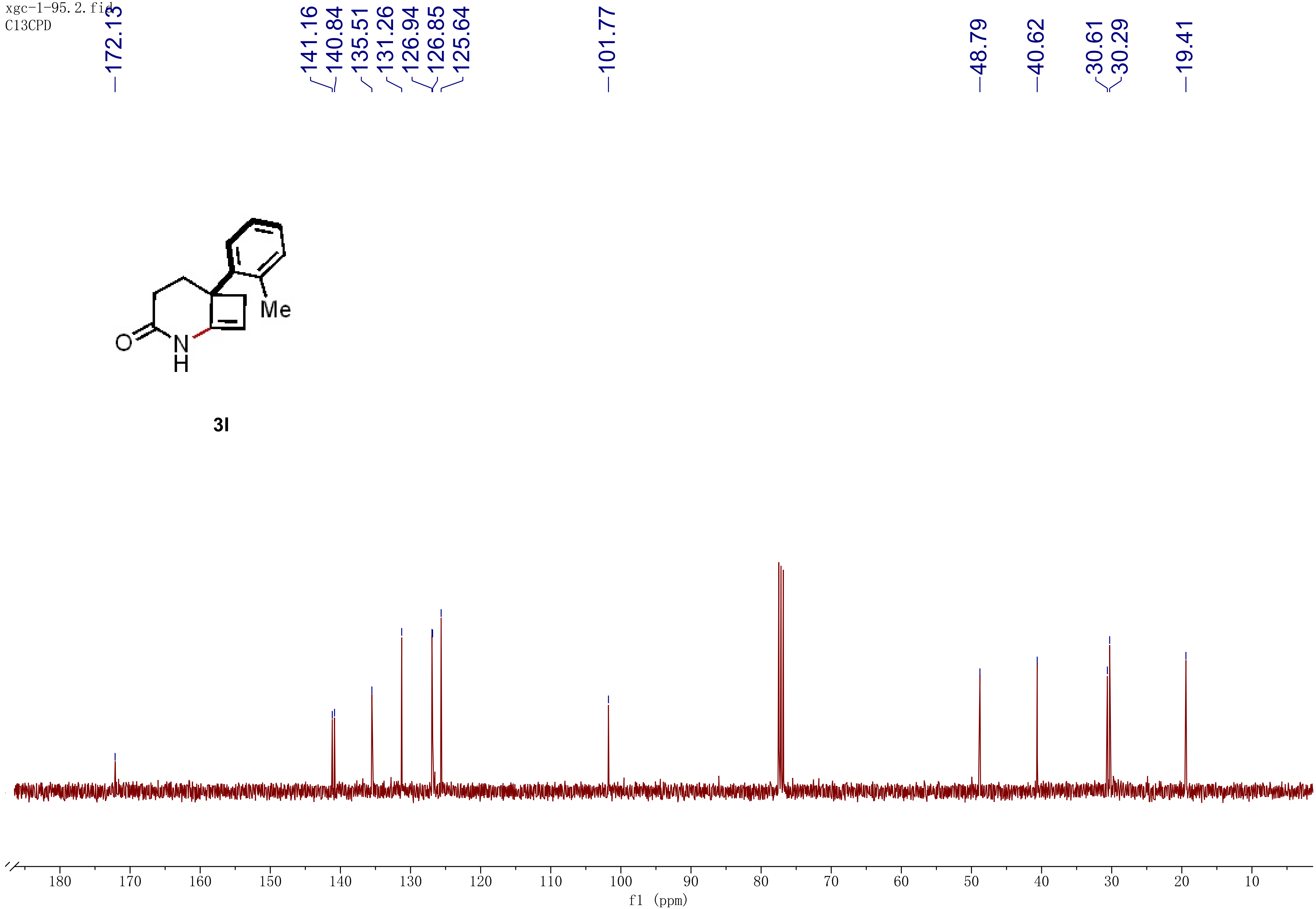

PROTON
7.15 7.14 7.12 6.99 6.98 6.96 6.85 6.83 6.76 6.60 6.59 6.58 6.24 3.67 2.96 2.94 2.93 2.93 2.92 2.91 2.89 2.88 2.85 2.84 2.83 2.83 2.82 2.81 2.59 2.57 2.56 2.55 2.54 2.53 2.51 2.50 2.50 2.48 2.47 2.46 2.46 2.44 2.41 2.39 2.38 2.37 2.36 2.34 2.33 2.31 2.30 2.29 2.28 2.26
S
O
N
H
N
3k
1.00 2.01 1.00 0.97 0.99 1.99 0.94 3.00 1.04 2.02 3.00 1.02 1.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

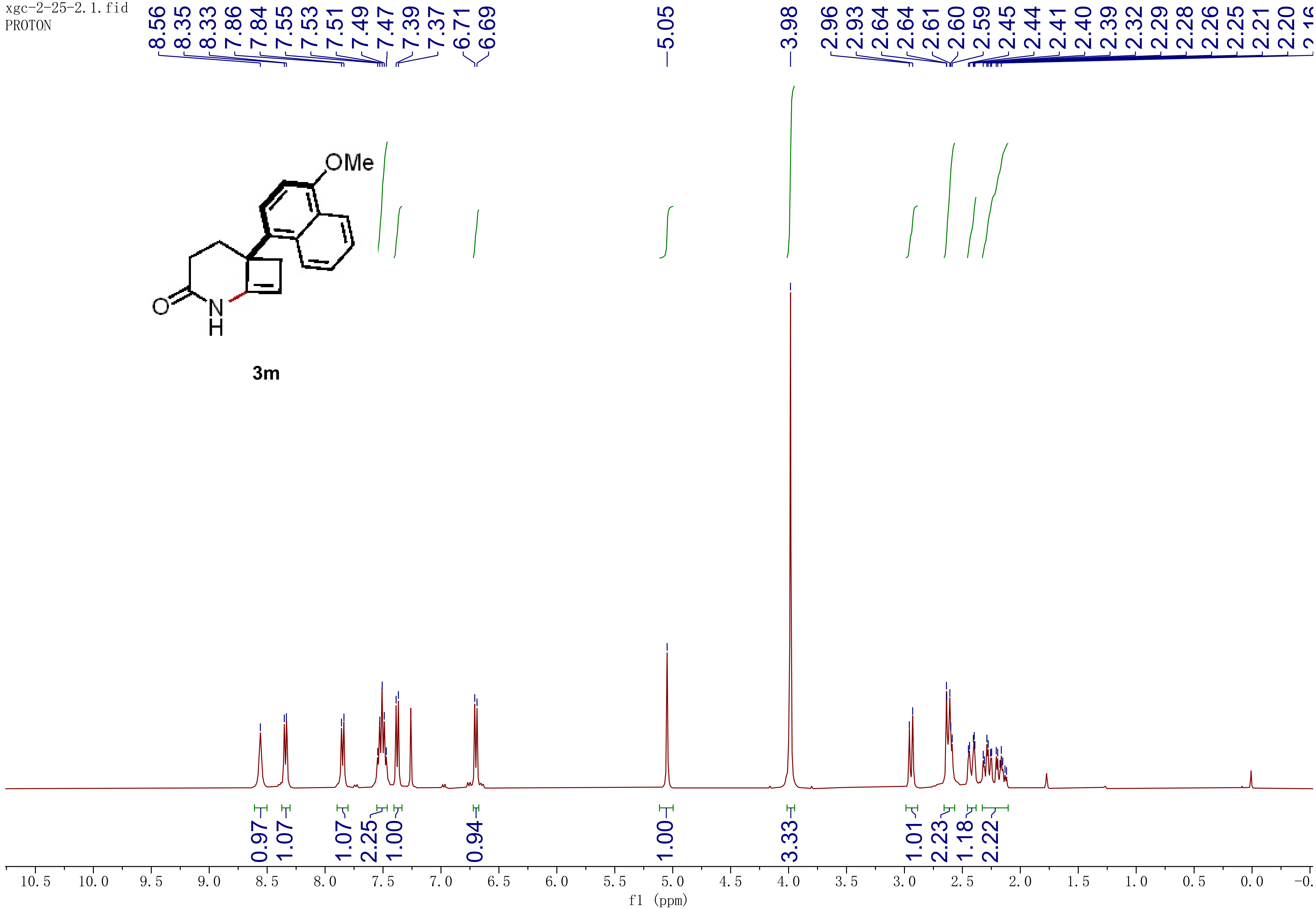

xgc-1-70.2.fid
C13CPD
172.48
148.85
137.63
127.74
126.27
125.96
124.30
123.66
121.82
120.88
119.40
115.38
109.31
63.77
48.33
33.56
32.89
32.70
29.71
27.60
3k
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

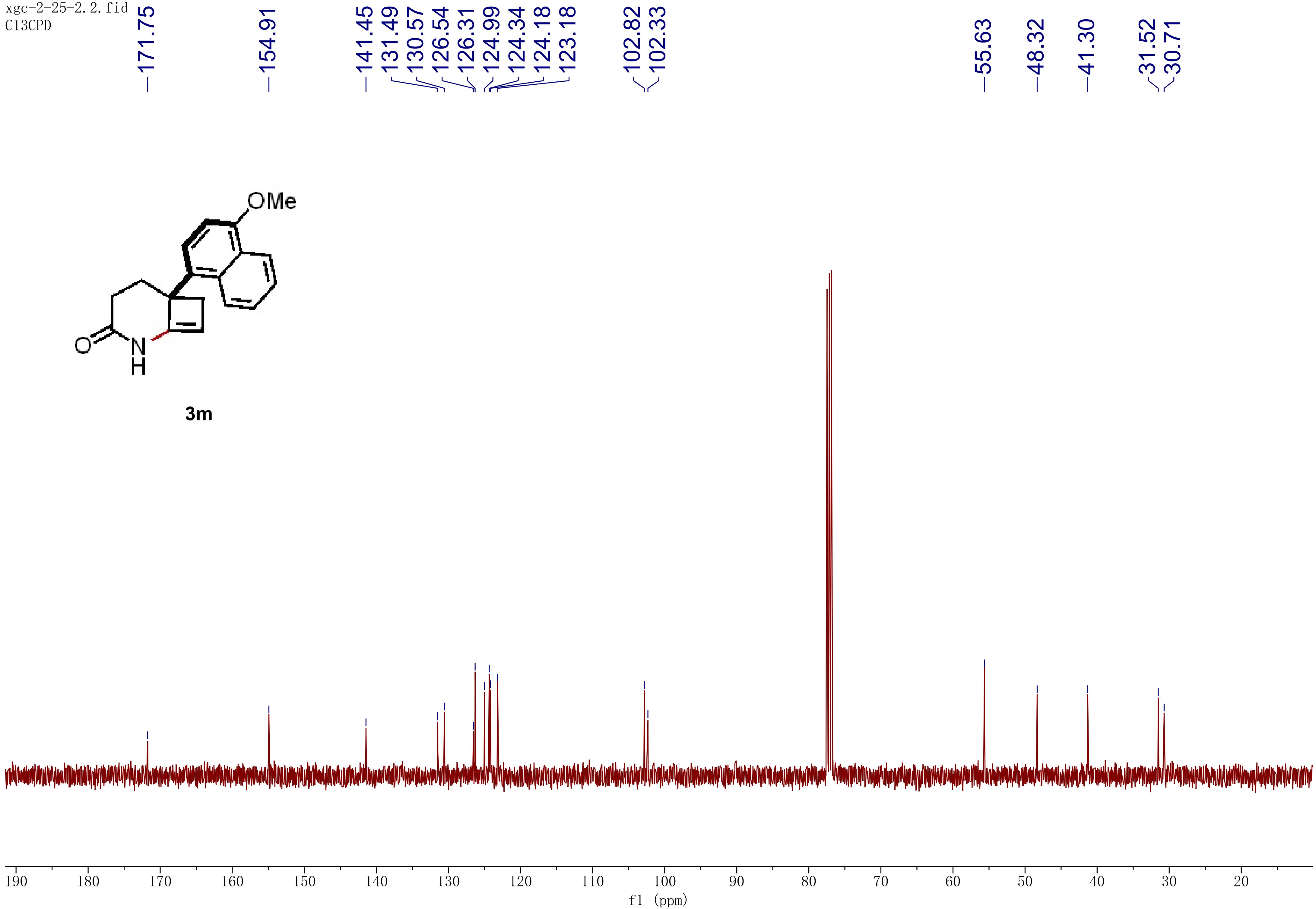
xgc-1-95.1.fid
PROTON

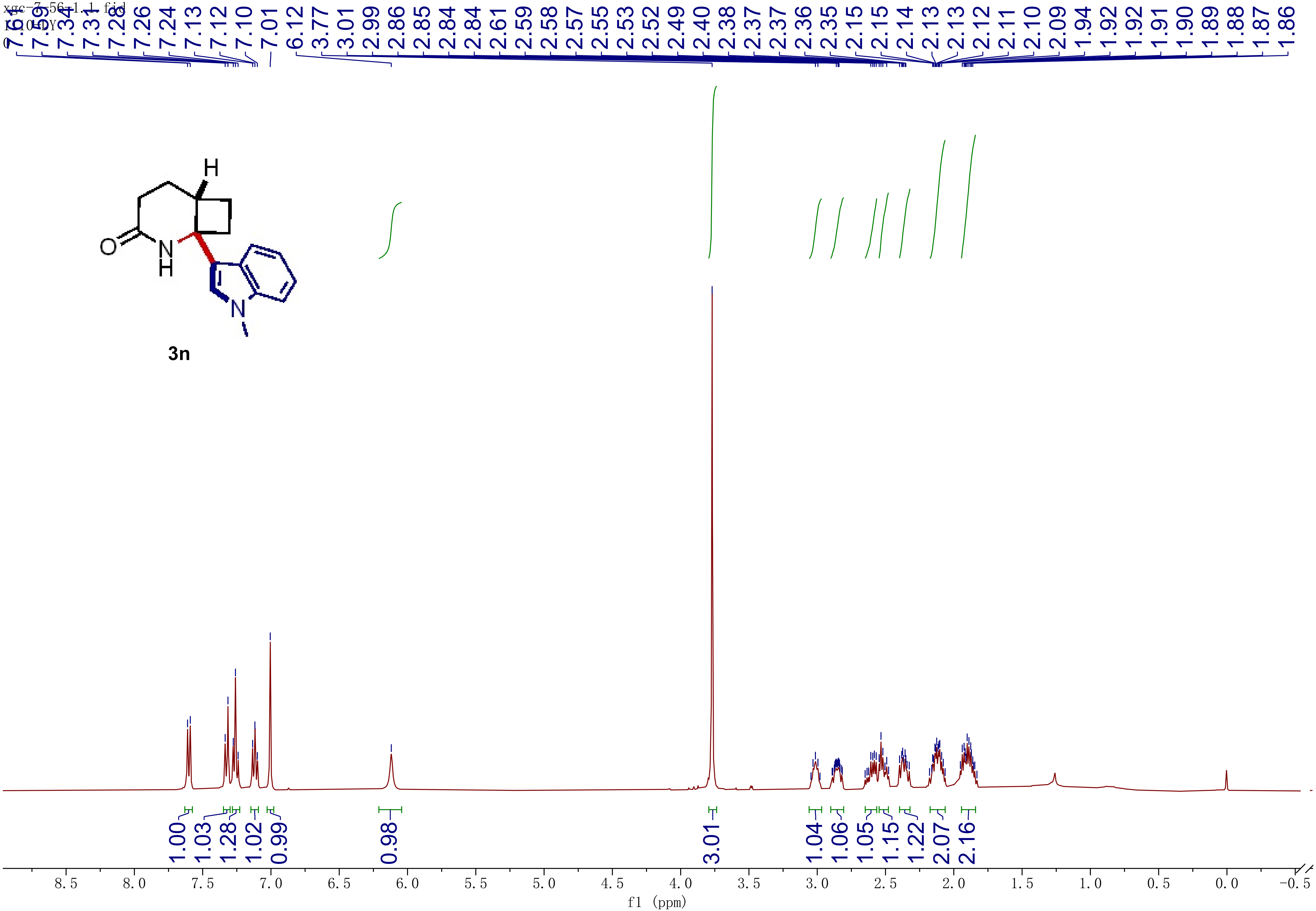


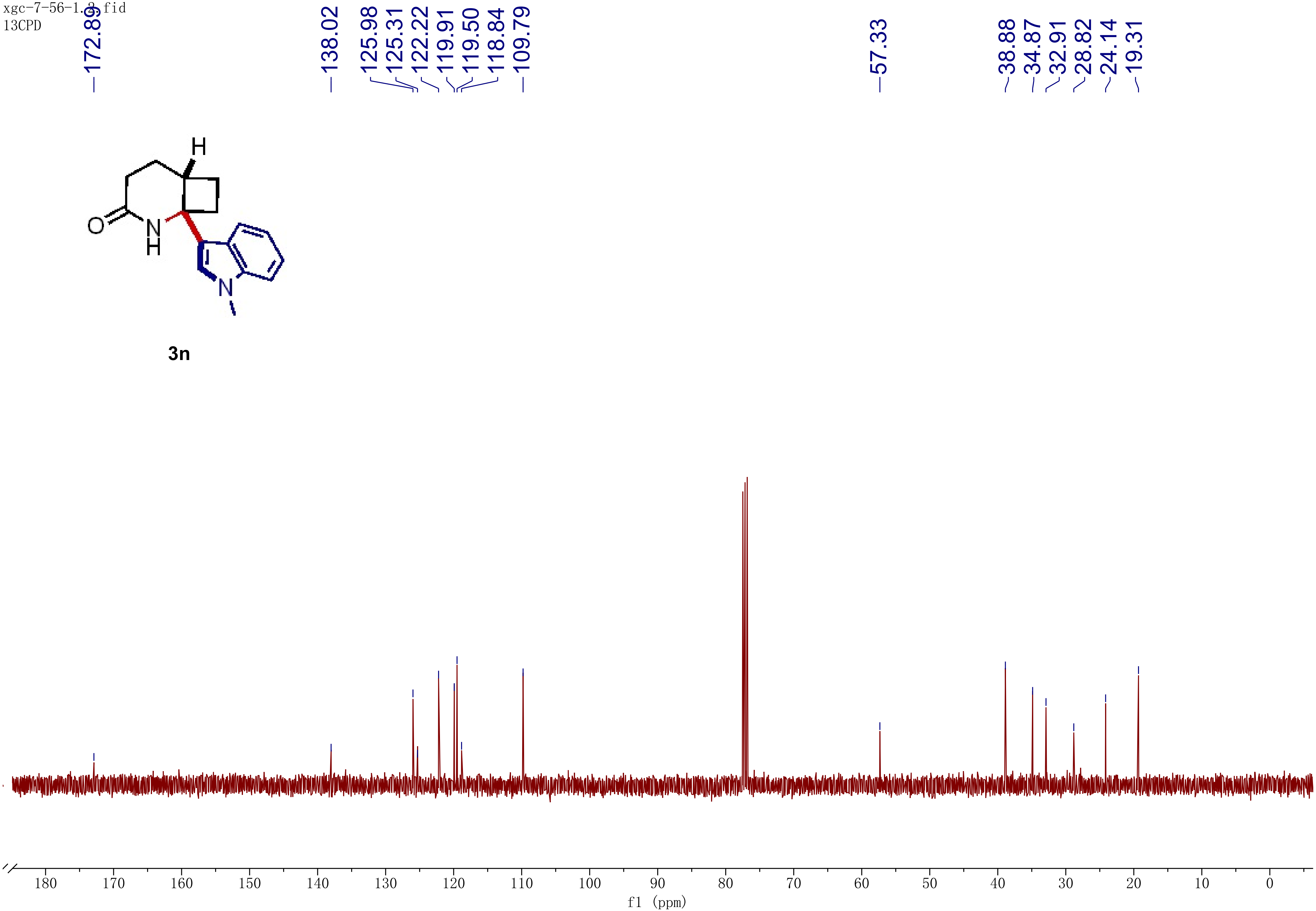
3l


xgc-1-95.2.fid
C13CPD
172.13
141.16
140.84
135.51
131.26
126.94
126.85
125.64
101.77
48.79
40.62
30.61
30.29
19.41
Me
O
N
H
3l
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

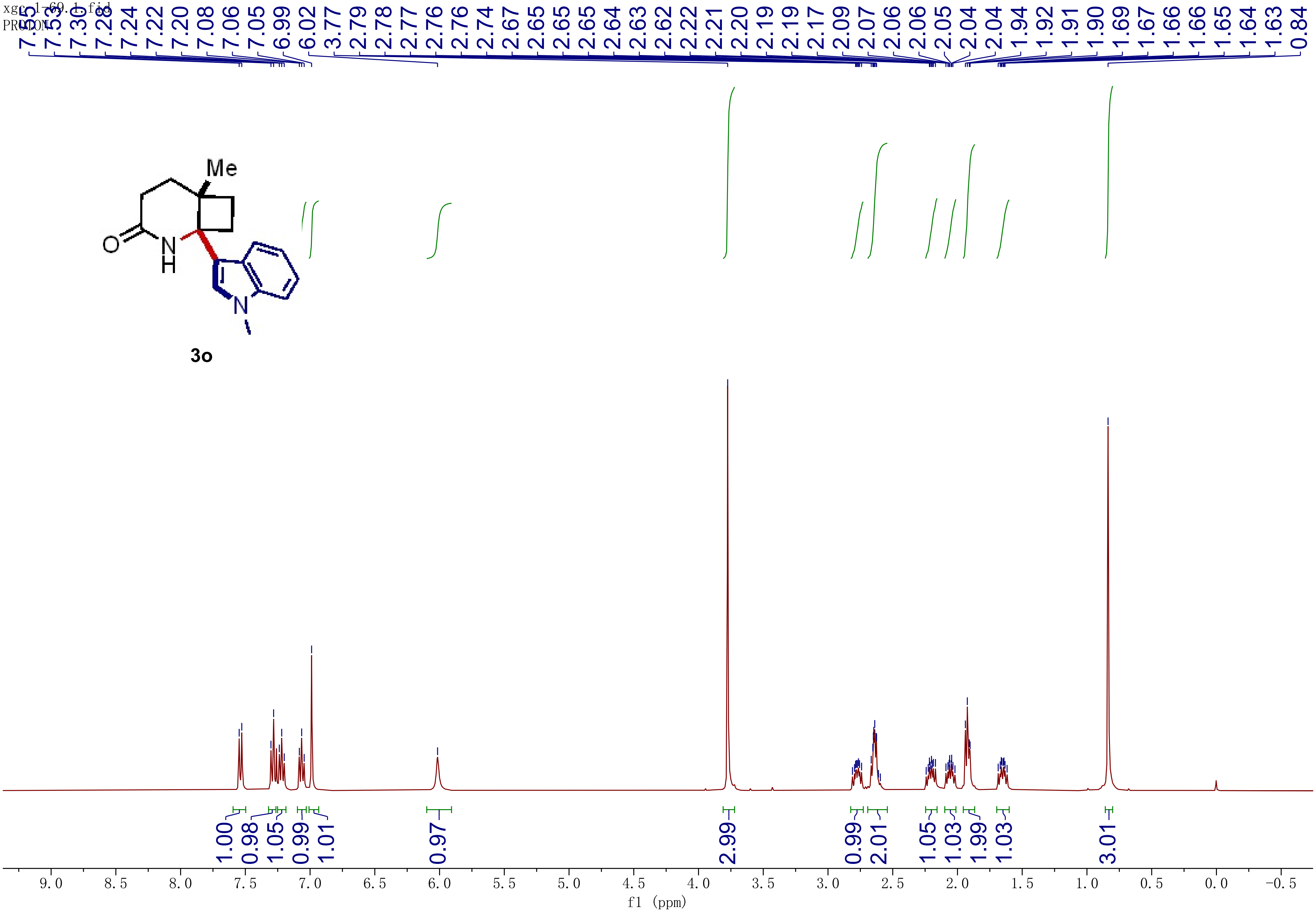

xgc-2-25-2.1.fid
PROTON
8.56
8.35
8.33
7.86
7.84
7.55
7.53
7.51
7.49
7.47
7.39
7.37
6.71
6.69
5.05
3.98
2.96
2.93
2.64
2.64
2.61
2.60
2.59
2.45
2.44
2.41
2.40
2.39
2.32
2.29
2.28
2.26
2.25
2.21
2.20
OMe
O
N
H
3m
0.97
1.07
1.07
2.25
1.00
0.94
1.00
3.33
1.01
2.23
1.18
2.22
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


xgc-2-25-2.2.fid
C13CPD
171.75
154.91
141.45
131.49
130.57
126.54
126.31
124.99
124.34
124.18
123.18
102.82
102.33
55.63
48.32
41.30
31.52
30.71
OMe
O
N
H
3m
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

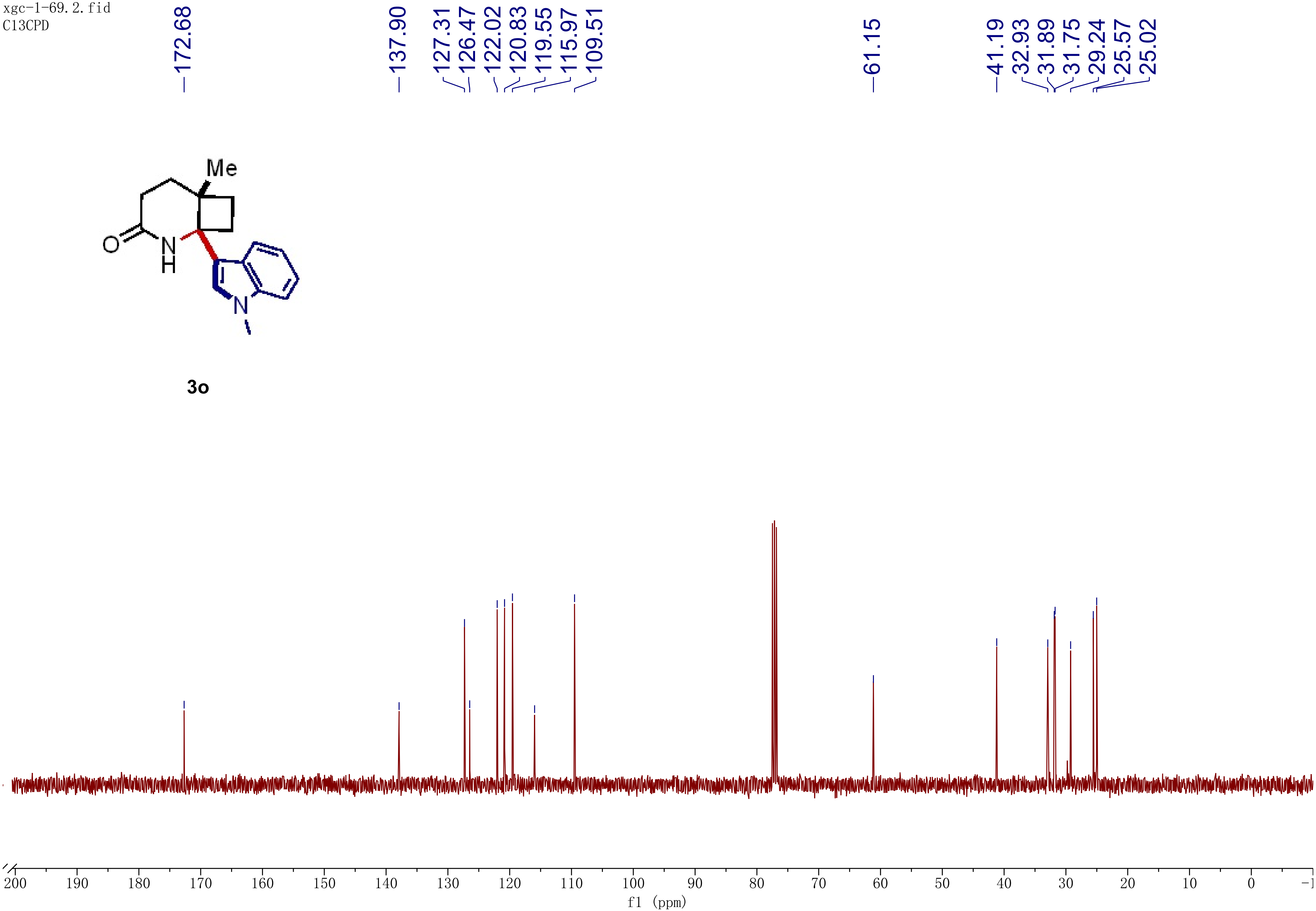

xgc-7-56-1.1.fid
7.60 7.59 7.34 7.31 7.28 7.26 7.24 7.13 7.12 7.10 7.01 6.12 3.77 3.01 2.99 2.86 2.85 2.84 2.84 2.61 2.59 2.58 2.57 2.55 2.53 2.52 2.49 2.40 2.38 2.37 2.37 2.36 2.35 2.15 2.15 2.14 2.13 2.13 2.12 2.11 2.10 2.09 1.94 1.92 1.92 1.91 1.90 1.89 1.88 1.87 1.86
H
O
N
H
N
3n
1.00 1.03 1.28 1.02 0.99 0.98 3.01 1.04 1.06 1.05 1.15 1.22 2.07 2.16
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)



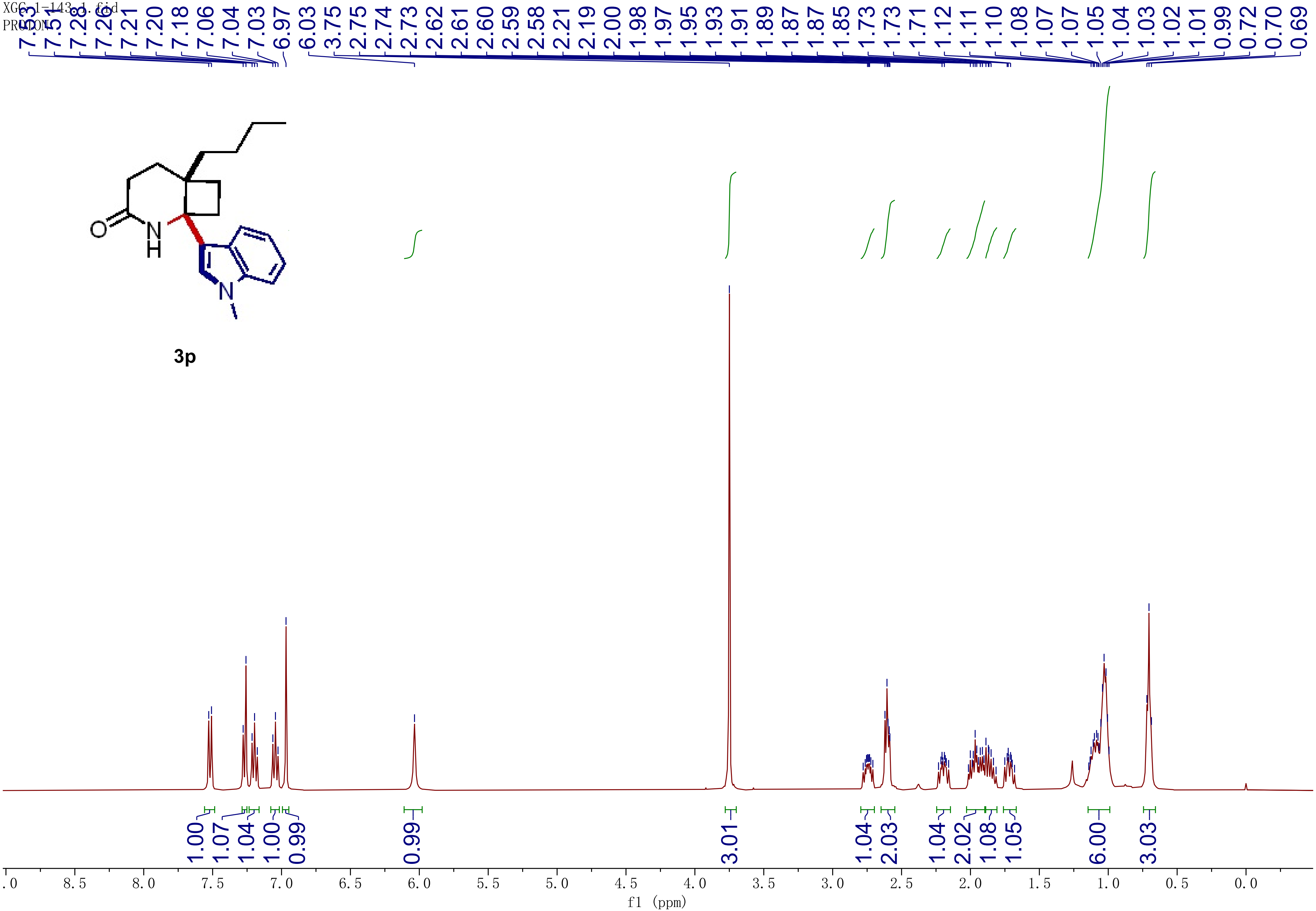
xgc-7-56-1.2.fid
13CPD
172.89
138.02
125.98
125.31
122.22
119.91
119.50
118.84
109.79
57.33
38.88
34.87
32.91
28.82
24.14
19.31
O
N
H
3n
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



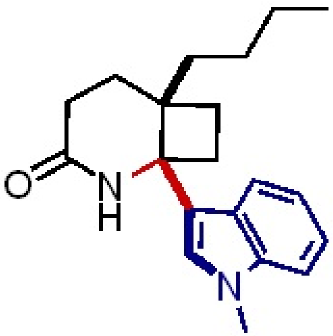
xgc-1-69.1.fid
PROTON
7.30 7.28 7.24 7.22 7.20 7.08 7.06 7.05 6.99 6.02 3.77 2.79 2.78 2.77 2.76 2.76 2.74 2.67 2.65 2.65 2.65 2.64 2.63 2.62 2.22 2.21 2.20 2.19 2.19 2.17 2.09 2.07 2.06 2.06 2.05 2.04 2.04 1.94 1.92 1.91 1.90 1.69 1.67 1.66 1.66 1.65 1.64 1.63 0.84
Me
O
N
H
N
3o
1.00 0.98 1.05 0.99 1.01 0.97 2.99 0.99 2.01 1.05 1.03 1.99 1.03 3.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


xgc-1-69.2.fid
C13CPD
172.68
137.90
127.31
126.47
122.02
120.83
119.55
115.97
109.51
61.15
41.19
32.93
31.89
31.75
29.24
25.57
25.02
Me
O
N
H
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

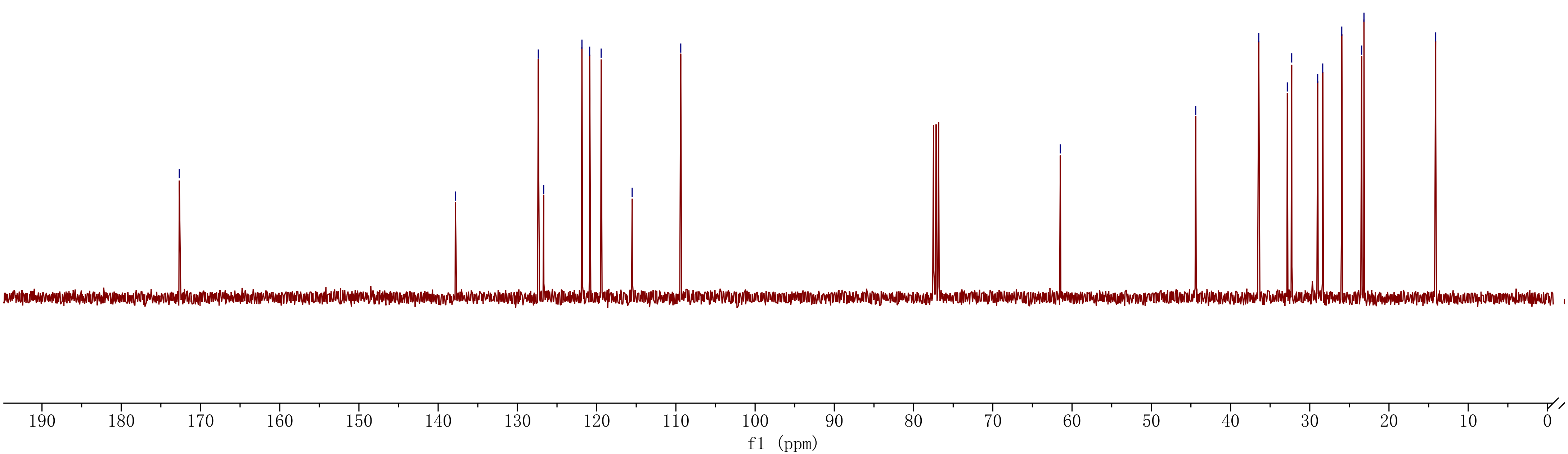
3o

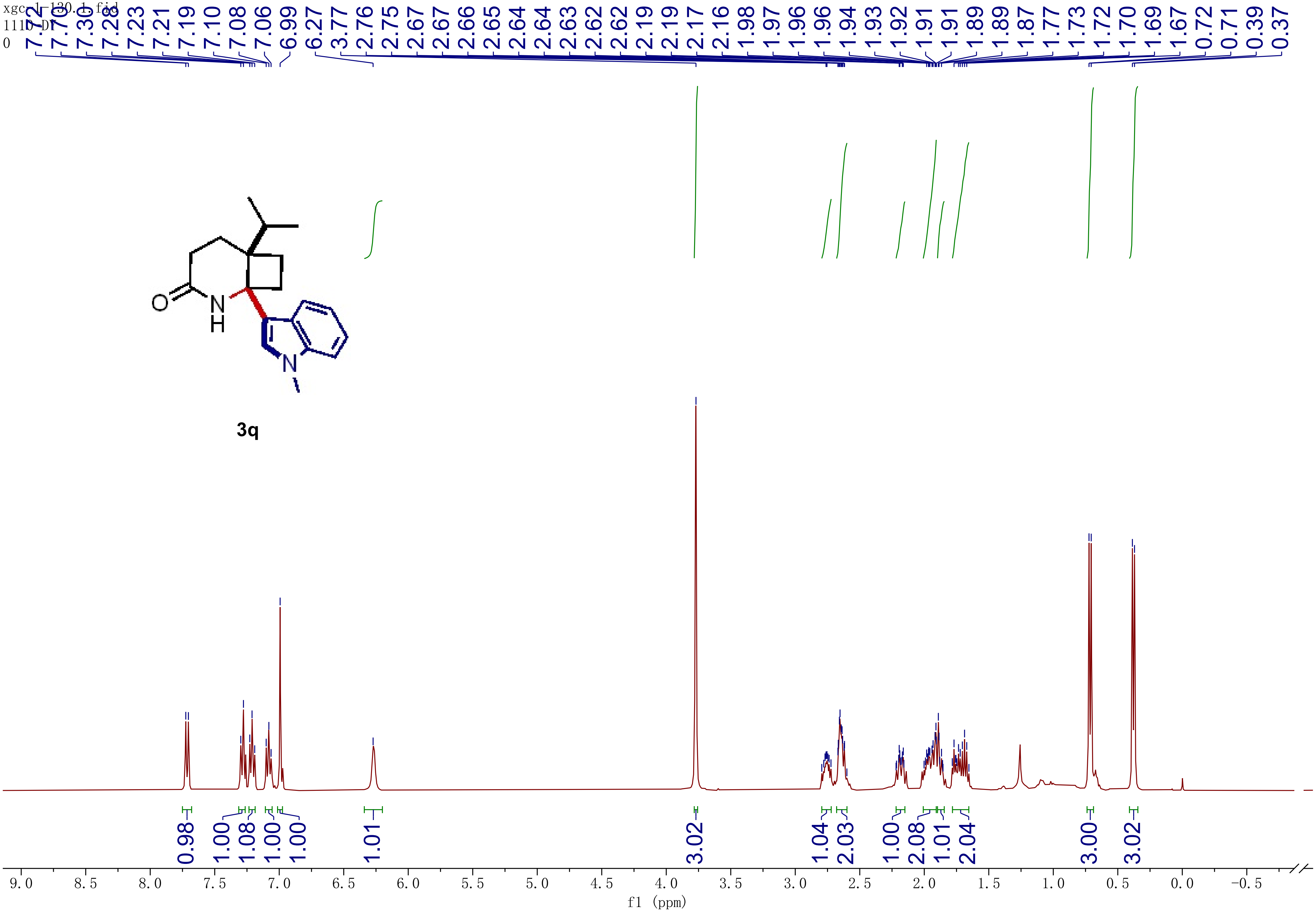

XGC-1-143.1.fid
PROTON
7.53 7.51 7.28 7.26 7.21 7.20 7.18 7.06 7.04 7.03 6.97 6.03 3.75 2.75 2.74 2.73 2.62 2.61 2.60 2.59 2.58 2.21 2.19 2.00 1.98 1.97 1.95 1.93 1.91 1.89 1.87 1.87 1.85 1.73 1.73 1.71 1.12 1.11 1.10 1.08 1.07 1.07 1.05 1.04 1.03 1.02 1.01 0.99 0.72 0.70 0.69
O
N
H
N
3p
1.00 1.07 1.04 1.00 0.99 0.99 3.01 1.04 2.03 1.04 2.02 1.08 1.05 6.00 3.03
f1 (ppm)

XGC-1-143.2.fid
C13CPD

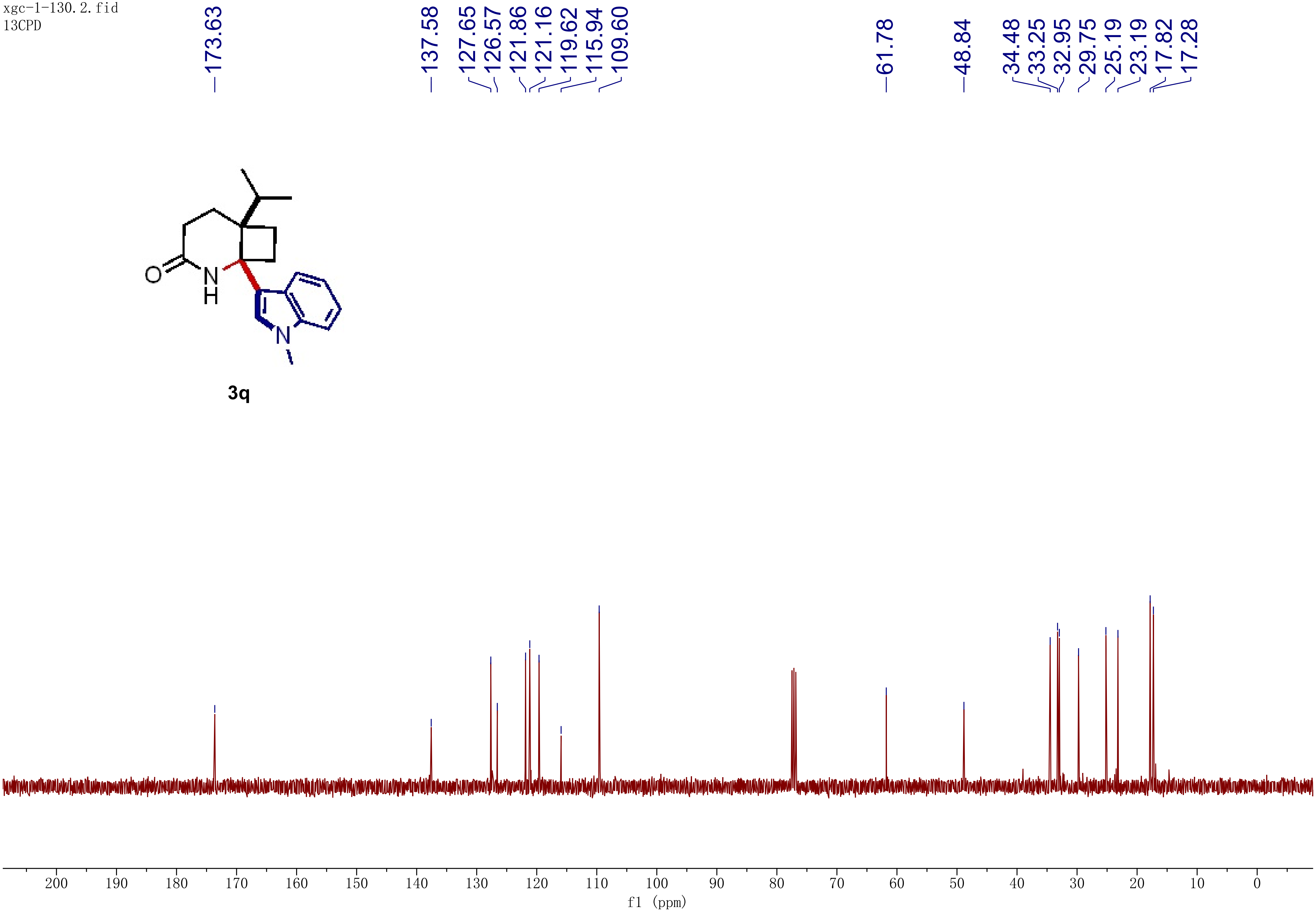


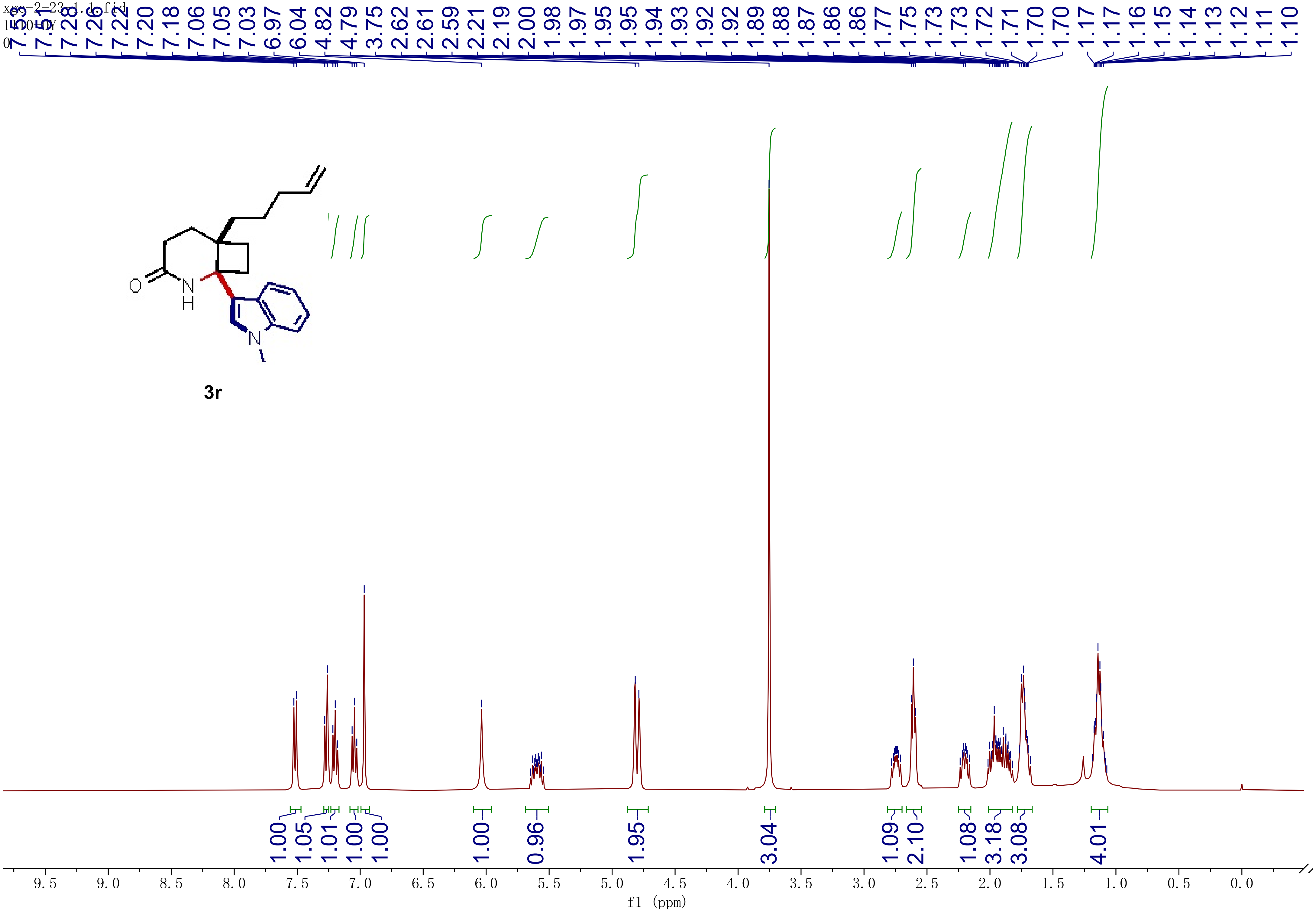
3p

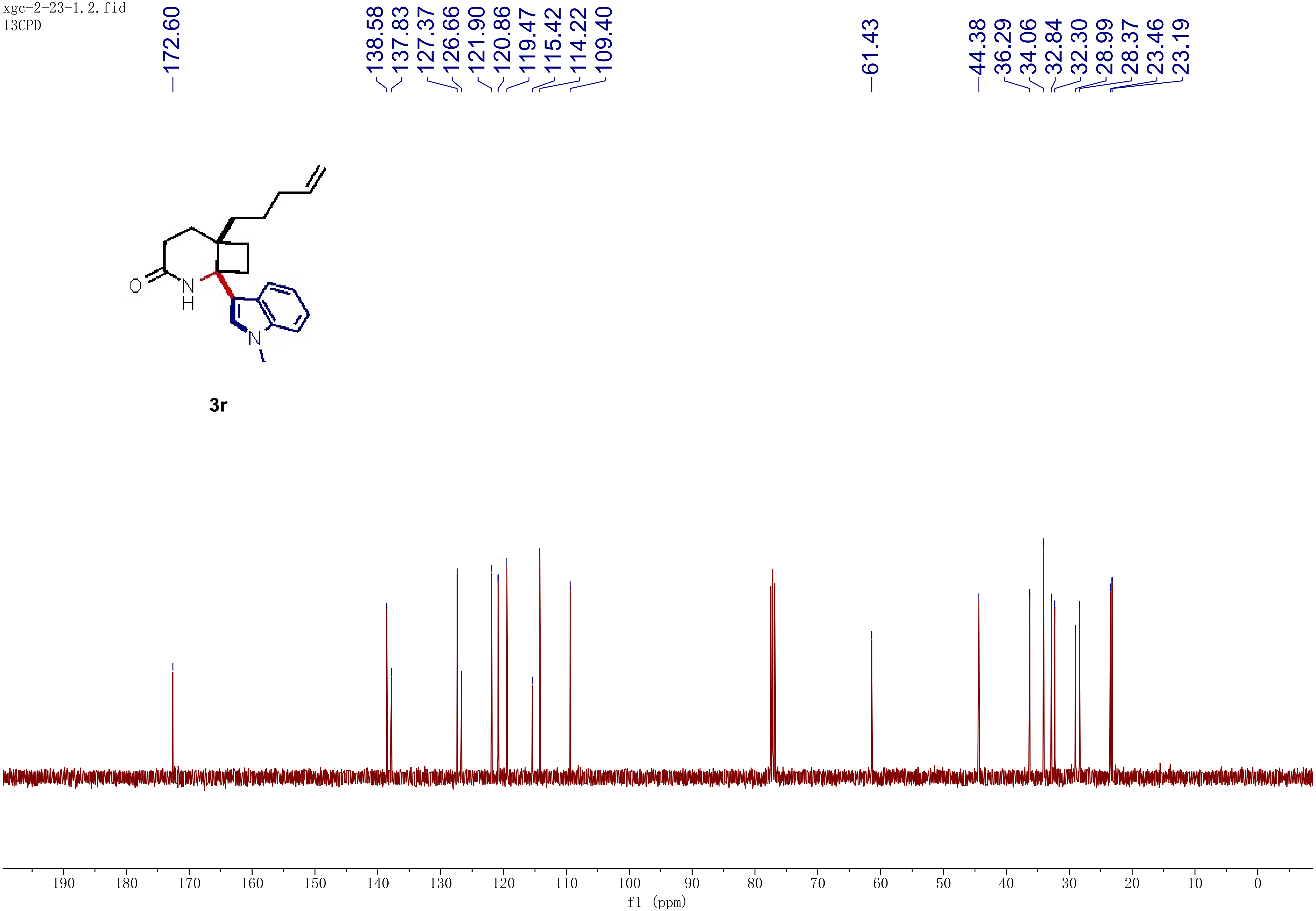


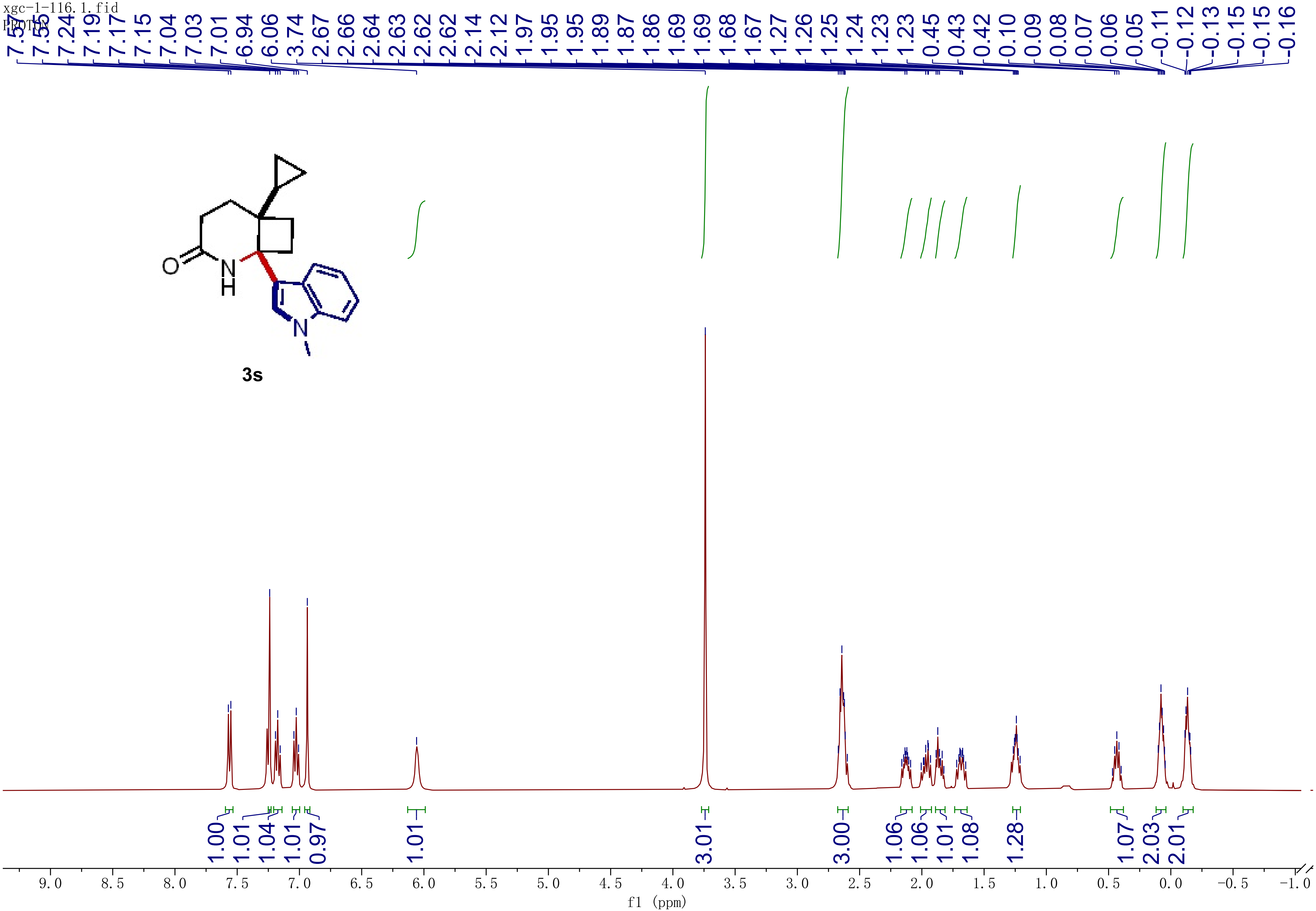

xgc-1-130.1.fid
1110-DT
0
7.72 7.70 7.30 7.28 7.23 7.21 7.19 7.10 7.08 7.06 6.99 6.27 3.77 2.76 2.75 2.67 2.67 2.66 2.65 2.64 2.64 2.63 2.62 2.62 2.19 2.19 2.17 2.16 1.98 1.97 1.96 1.96 1.94 1.93 1.92 1.91 1.91 1.89 1.89 1.87 1.77 1.73 1.72 1.70 1.69 1.67 0.72 0.71 0.39 0.37
O
N
H
N
3q
0.98 1.00 1.08 1.00 1.00 1.01 3.02 1.04 2.03 1.00 2.08 1.01 2.04 3.00 3.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

xgc-1-130.2.fid
13CPD

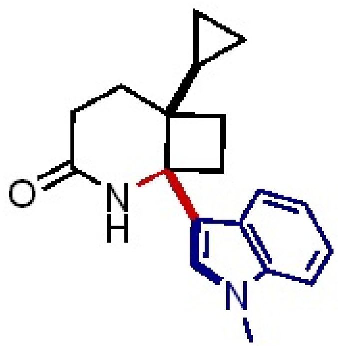



3r

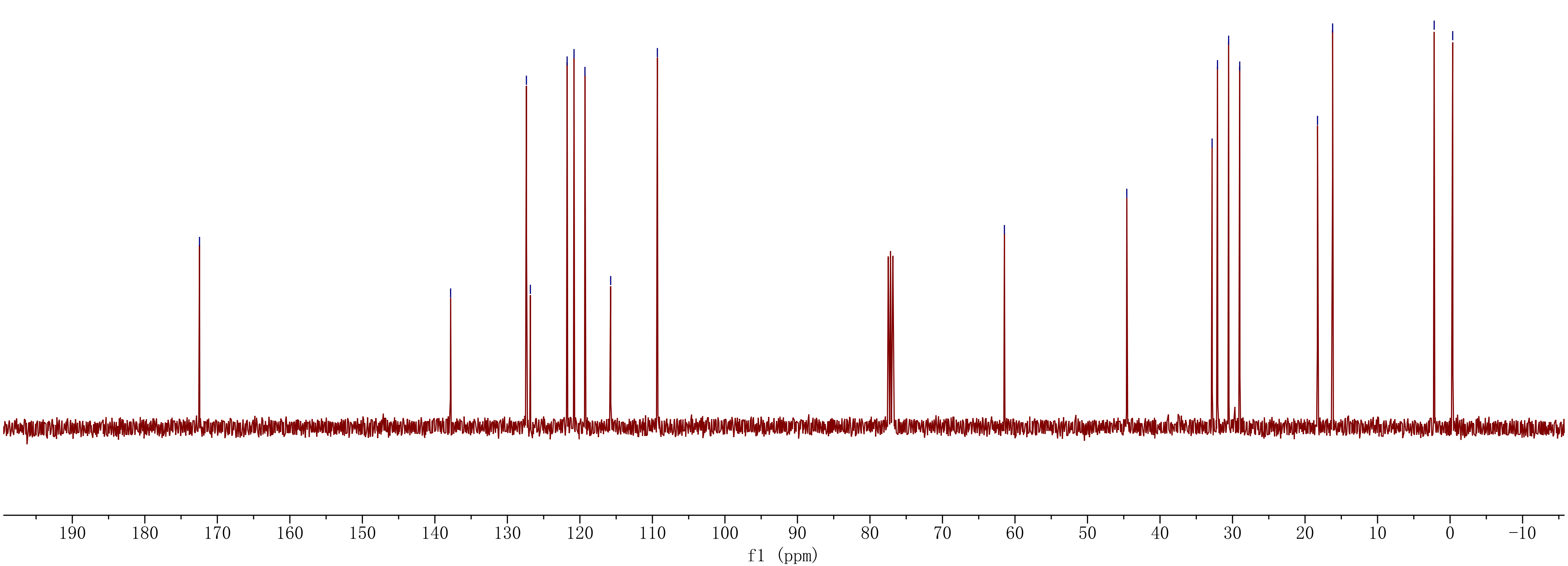

xgc-2-23-1.2.fid
13CPD
172.60
138.58
137.83
127.37
126.66
121.90
120.86
119.47
115.42
114.22
109.40
61.43
44.38
36.29
34.06
32.84
32.30
28.99
28.37
23.46
23.19
3r
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

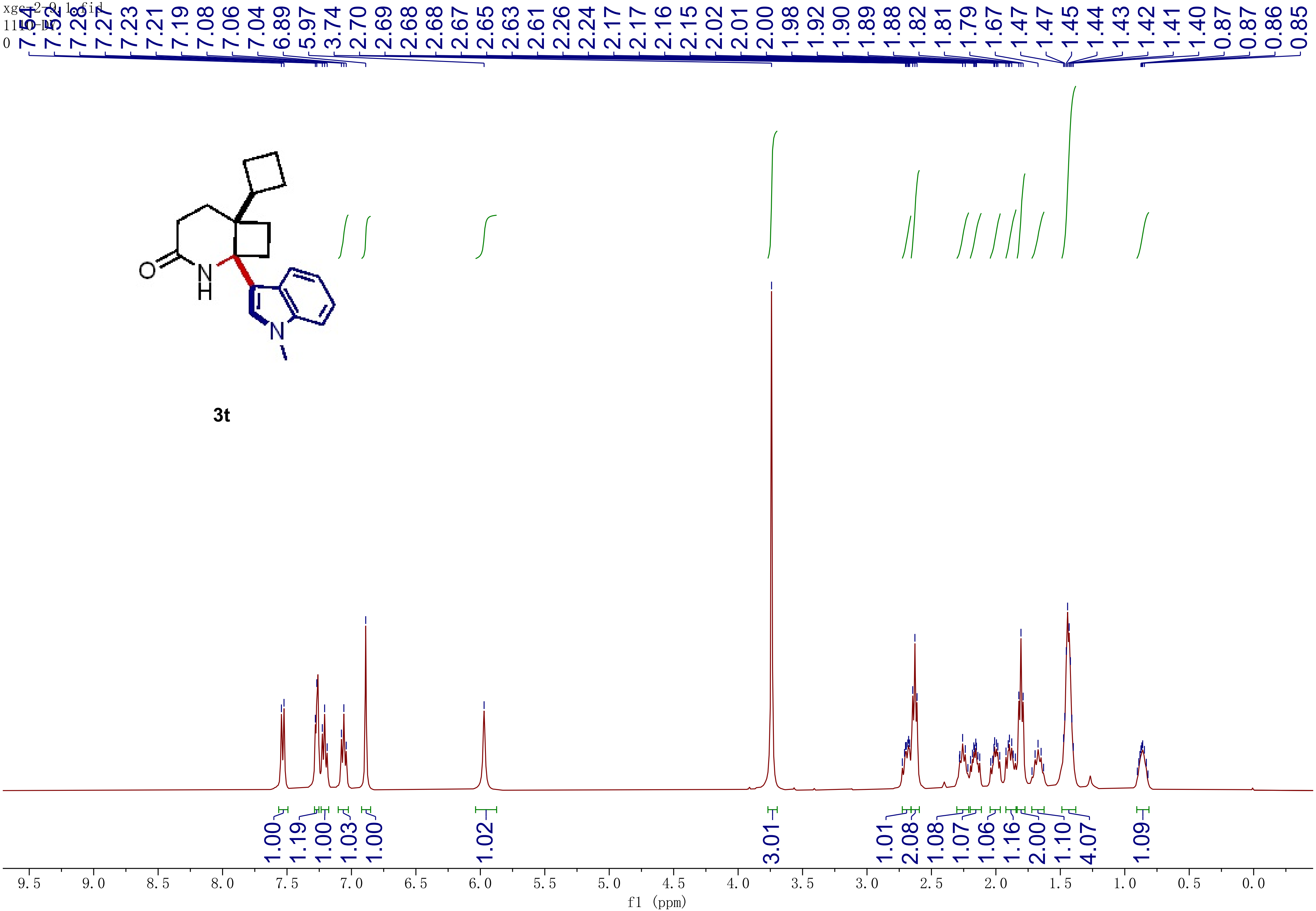

xgc-1-116.1.fid
PROTON
7.55
7.55
7.24
7.19
7.17
7.15
7.04
7.03
7.01
6.94
6.06
3.74
2.67
2.66
2.64
2.63
2.62
2.62
2.14
2.12
1.97
1.95
1.95
1.89
1.87
1.86
1.69
1.69
1.68
1.67
1.27
1.26
1.25
1.24
1.23
1.23
0.45
0.43
0.42
0.10
0.09
0.08
0.07
0.06
0.05
-0.11
-0.12
-0.13
-0.15
-0.15
-0.16
O
N
H
N
3s
1.00
1.01
1.04
1.01
0.97
1.01
3.01
3.00
1.06
1.06
1.01
1.08
1.28
1.07
2.03
2.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

xgc-1-116.2.fid
C13CPD

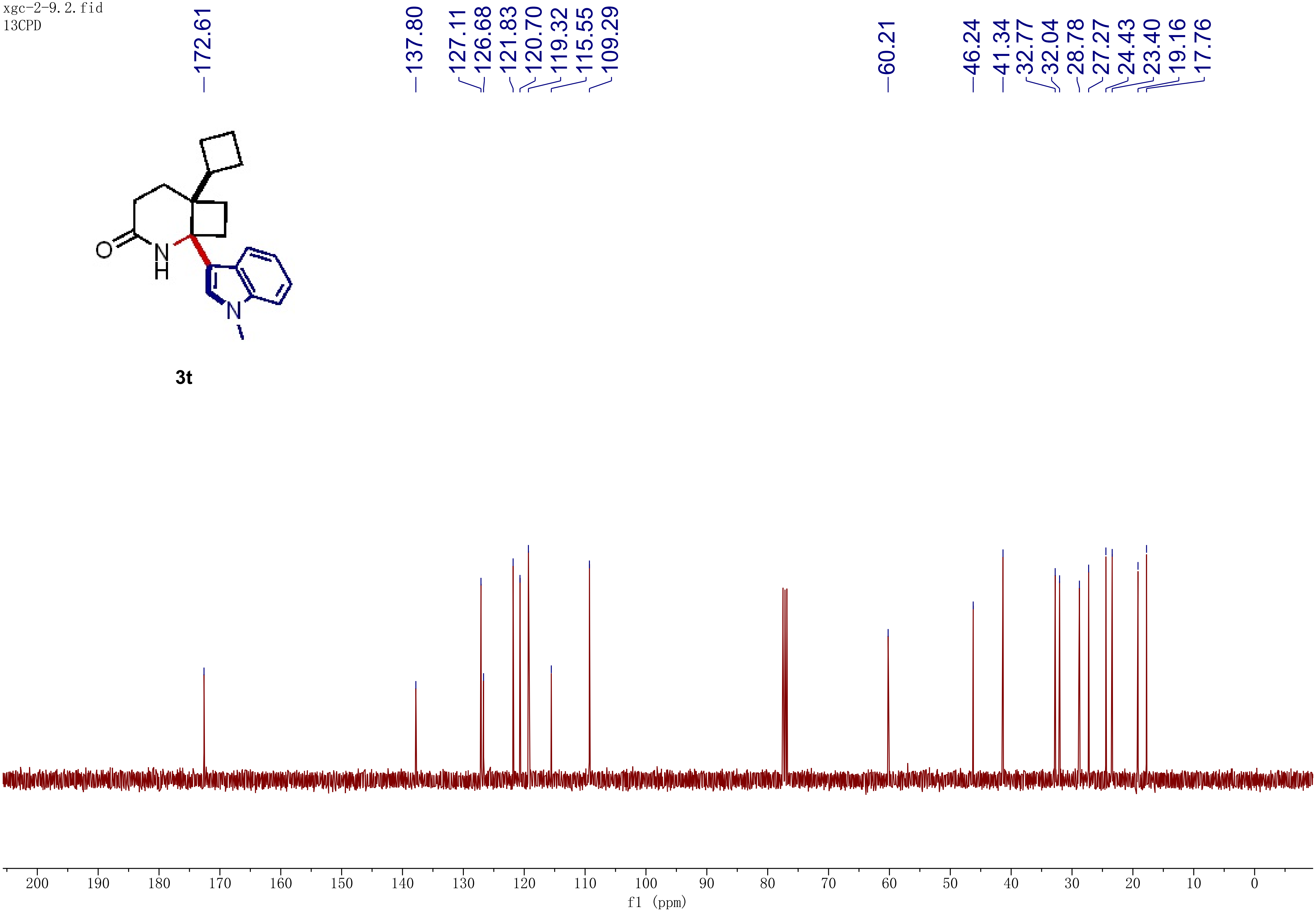


3s

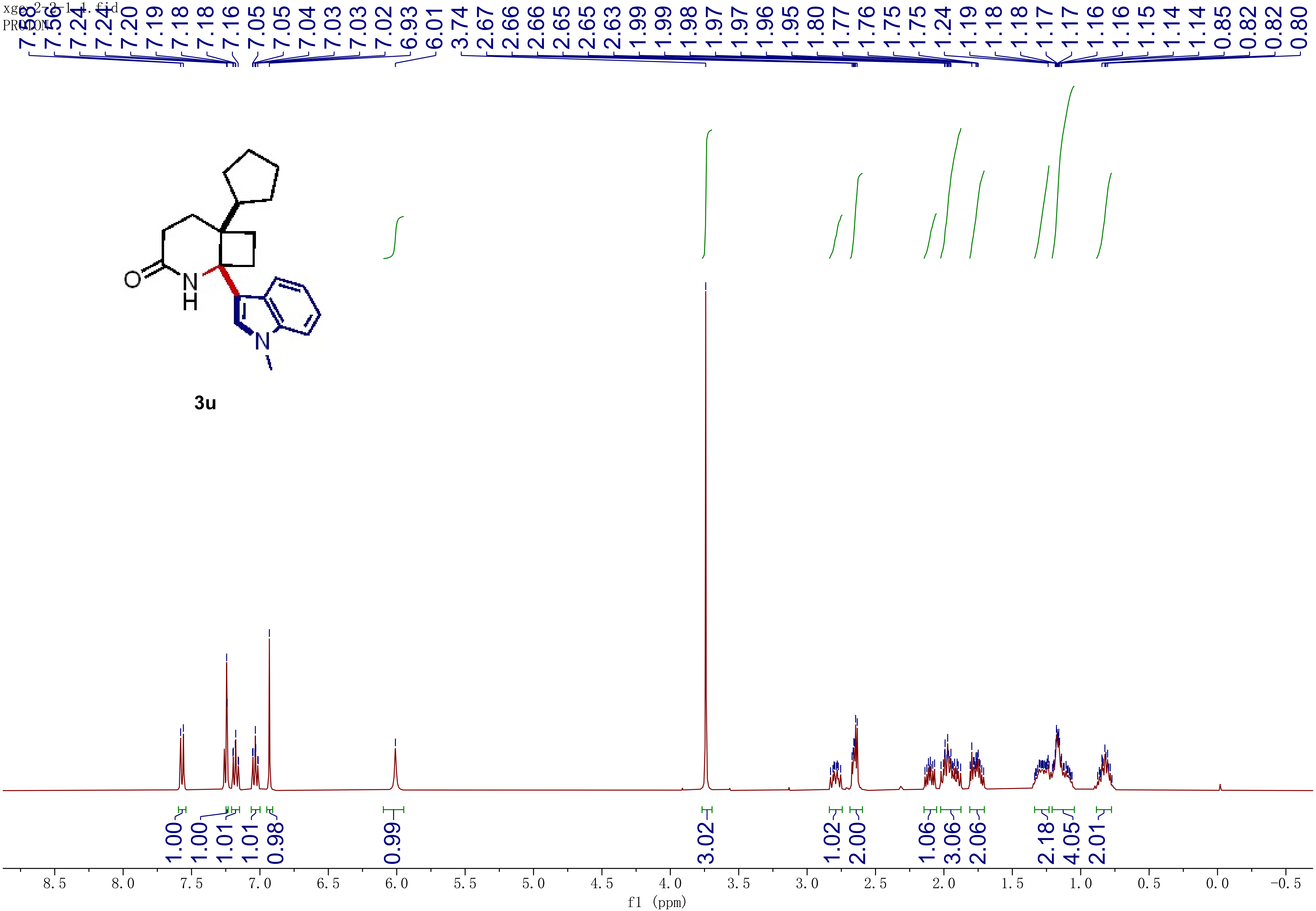


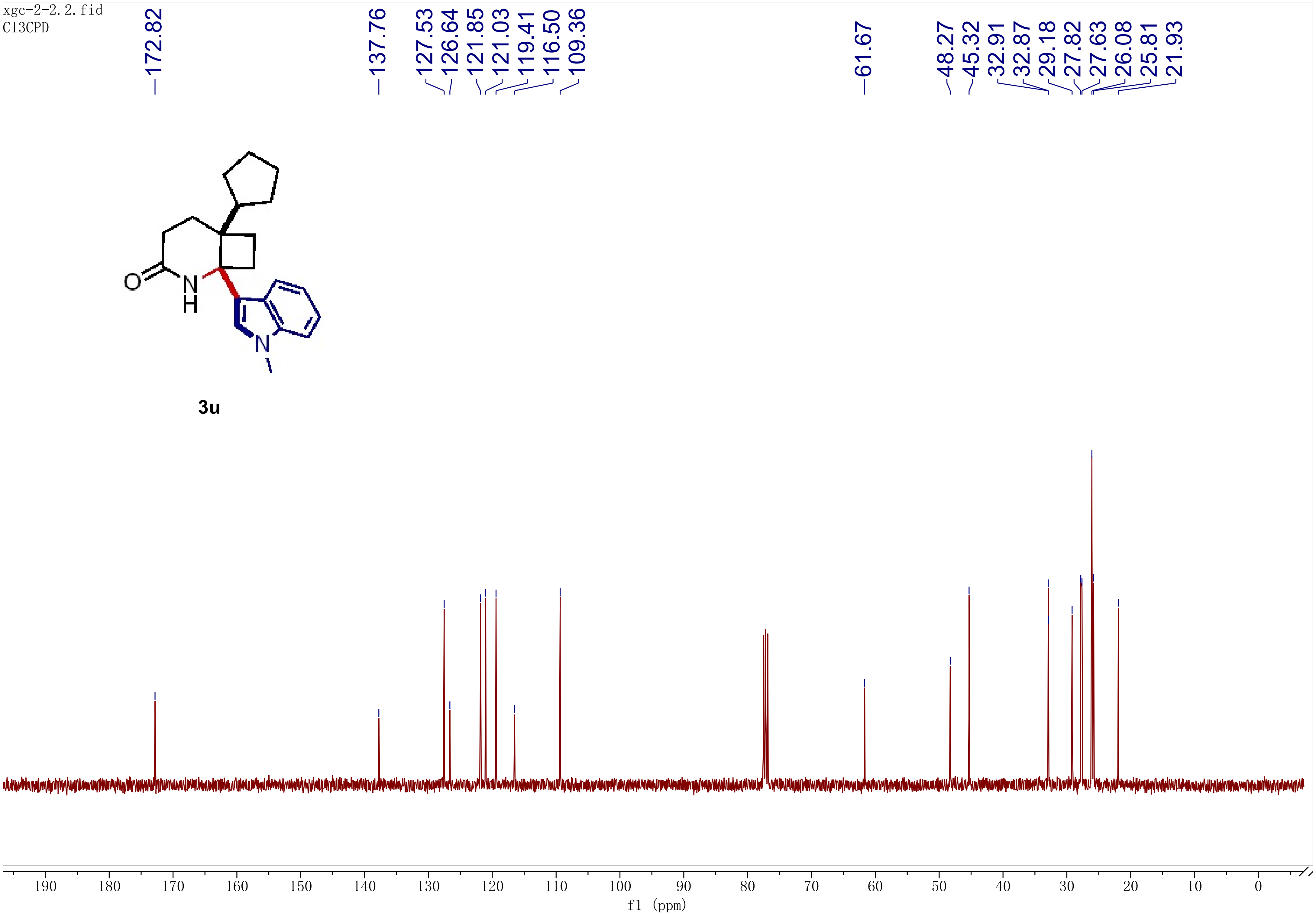


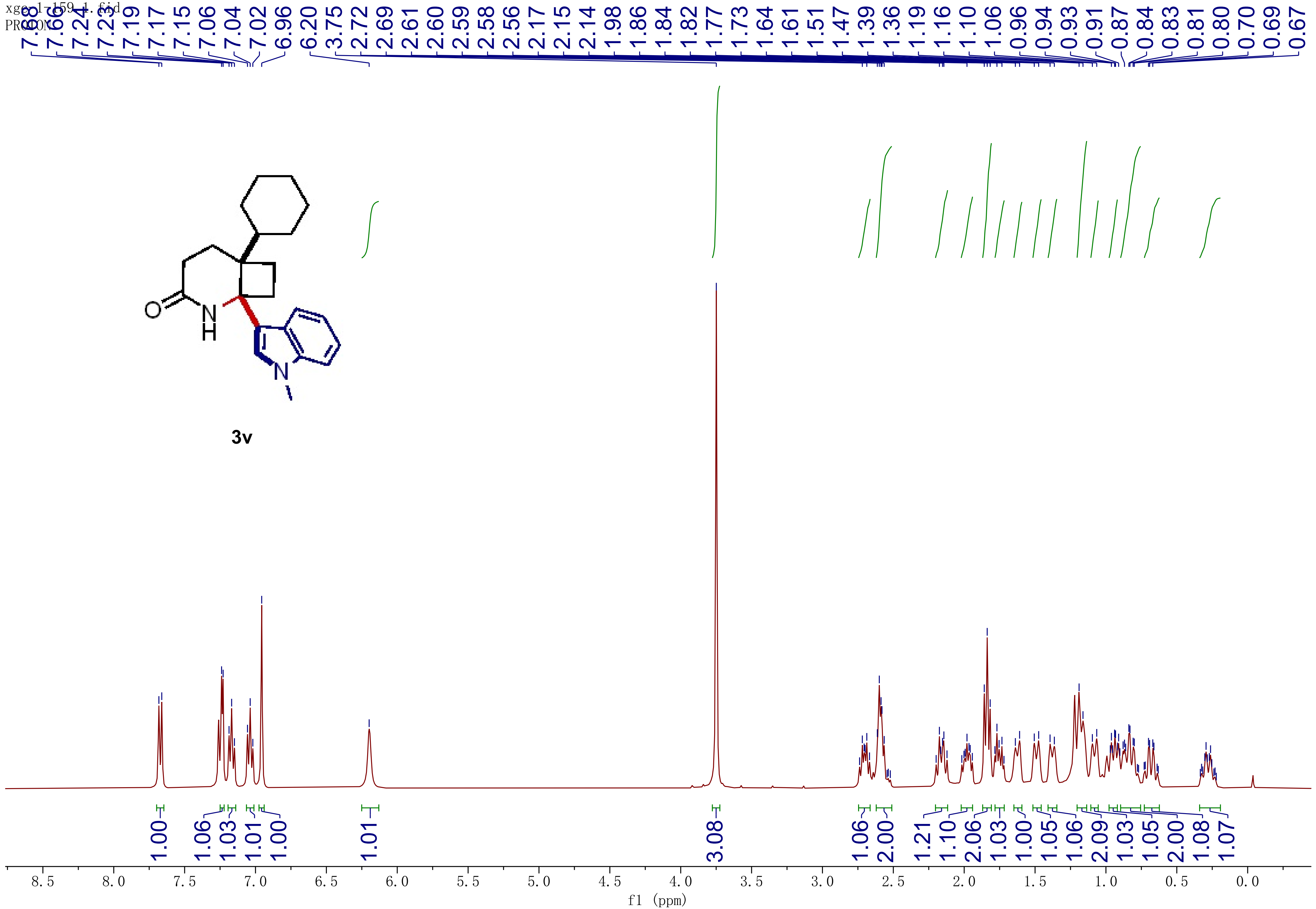
7.54 7.52 7.28 7.27 7.23 7.21 7.19 7.08 7.06 7.04 6.89 5.97 3.74 2.70 2.69 2.68 2.68 2.67 2.65 2.63 2.61 2.26 2.24 2.17 2.17 2.16 2.15 2.02 2.01 2.00 1.98 1.92 1.90 1.89 1.88 1.82 1.81 1.79 1.67 1.47 1.47 1.45 1.44 1.43 1.42 1.41 1.40 0.87 0.87 0.86 0.85
O
N
H
N
3t
1.00 1.19 1.00 1.03 1.00 1.02 3.01 1.01 2.08 1.08 1.07 1.06 1.16 2.00 1.10 4.07 1.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


xgc-2-9.2.fid
13CPD
172.61
137.80
127.11
126.68
121.83
120.70
119.32
115.55
109.29
60.21
46.24
41.34
32.77
32.04
28.78
27.27
24.43
23.40
19.16
17.76
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

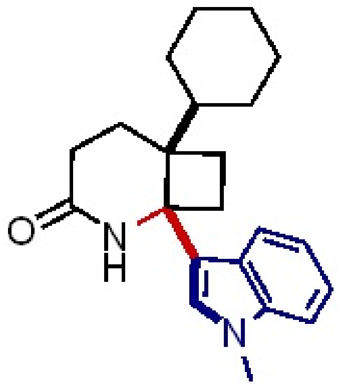
3t


xge-2-2-1-1.fid
PROTON
7.58
7.56
7.24
7.24
7.20
7.19
7.18
7.18
7.16
7.05
7.05
7.04
7.03
7.03
7.02
6.93
6.01
3.74
2.67
2.66
2.66
2.65
2.65
2.63
1.99
1.99
1.98
1.97
1.97
1.96
1.95
1.80
1.77
1.76
1.75
1.75
1.24
1.19
1.18
1.18
1.17
1.17
1.16
1.16
1.15
1.14
1.14
0.85
0.82
0.82
0.80
O
N
H
N
3u
1.00
1.00
1.01
1.01
0.98
0.99
3.02
1.02
2.00
1.06
3.06
2.06
2.18
4.05
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

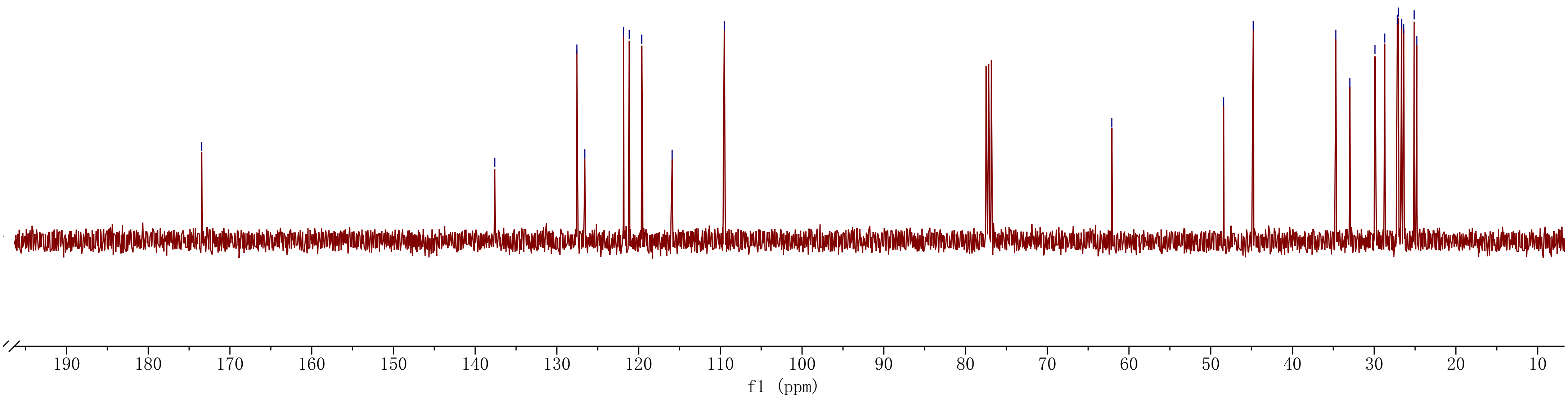

xgc-2-2.2.fid
C13CPD
172.82
137.76
127.53
126.64
121.85
121.03
119.41
116.50
109.36
61.67
48.27
45.32
32.91
32.87
29.18
27.82
27.63
26.08
25.81
21.93
O
N
H
N
3u
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

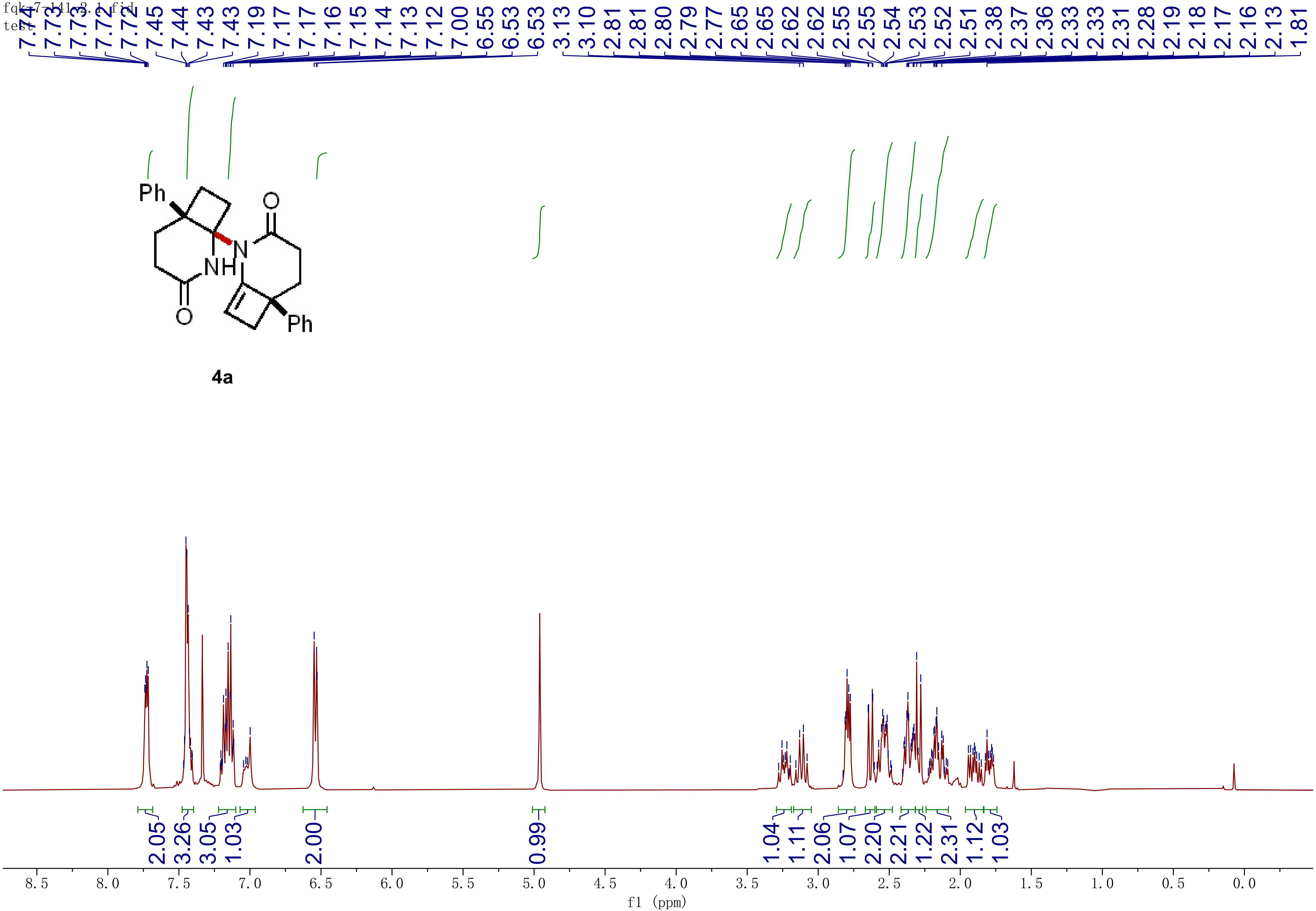

xge-1-159-1.fid
PROTON
7.68 7.66 7.24 7.23 7.19 7.17 7.15 7.06 7.04 7.02 6.96 6.20 3.75 2.72 2.69 2.61 2.60 2.59 2.58 2.56 2.17 2.15 2.14 1.98 1.86 1.84 1.82 1.77 1.73 1.64 1.61 1.51 1.47 1.39 1.36 1.19 1.16 1.10 1.06 0.96 0.94 0.93 0.91 0.87 0.84 0.83 0.81 0.80 0.70 0.69 0.67
O
N
H
N
3v
1.00 1.06 1.03 1.01 1.00 1.01 3.08 1.06 2.00 1.21 1.10 2.06 1.03 1.00 1.05 1.06 2.09 1.03 1.05 2.00 1.08 1.07
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

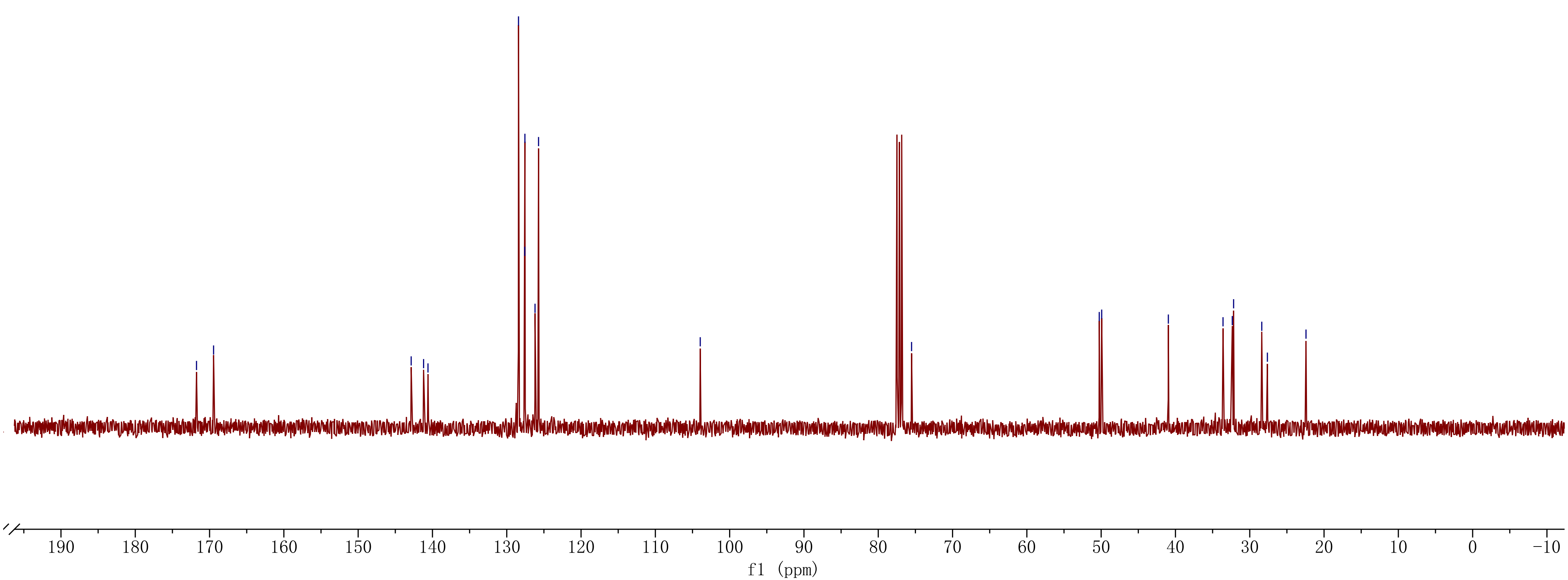
xgc-1-159.2.fid
C13CPD

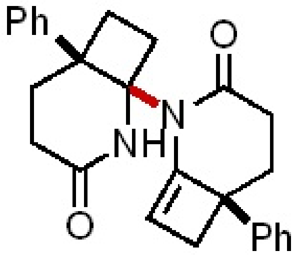


3v

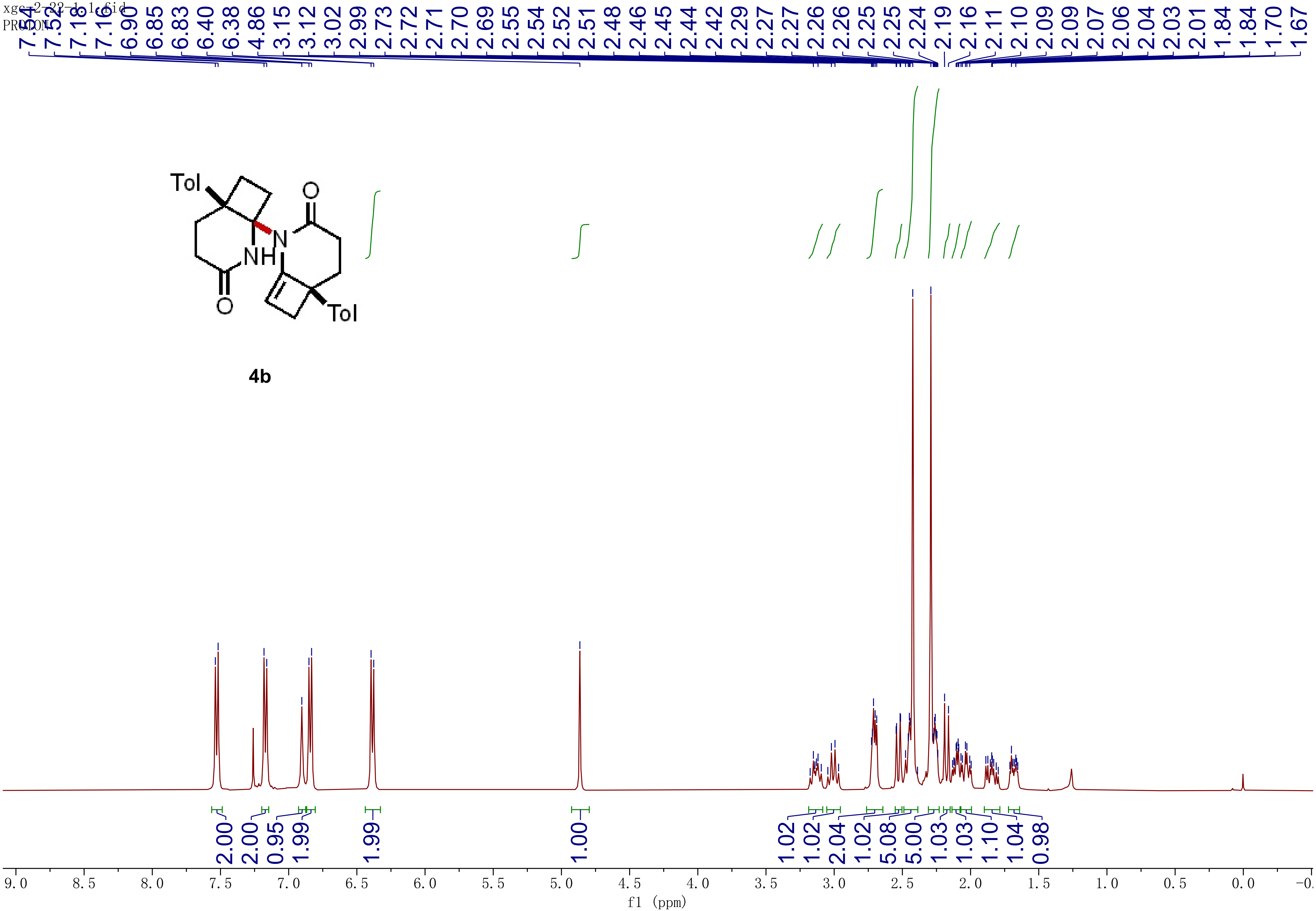


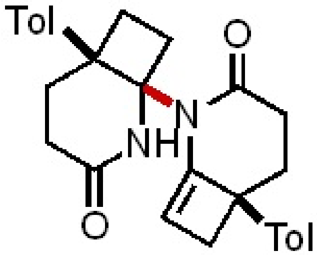

fqk-7-141-2.1.fid
test
7.74 7.73 7.73 7.72 7.72 7.45 7.44 7.43 7.43 7.19 7.17 7.17 7.16 7.15 7.14 7.13 7.12 7.00 6.55 6.53 6.53 3.13 3.10 2.81 2.81 2.80 2.79 2.77 2.65 2.65 2.62 2.62 2.55 2.55 2.54 2.53 2.52 2.51 2.38 2.37 2.36 2.33 2.33 2.31 2.28 2.19 2.18 2.17 2.16 2.13 1.81
Ph
O
NH
N
O
Ph
4a
2.05 3.26 3.05 1.03 2.00 0.99 1.04 1.11 2.06 1.07 2.20 2.21 1.22 2.31 1.12 1.03
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


fqk-7-141-2.2.fid
test
171.76
169.46
142.86
141.19
140.60
128.42
127.58
127.56
126.20
125.72
103.96
75.52
50.25
49.93
40.95
33.60
32.35
32.17
28.38
27.62
22.43
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

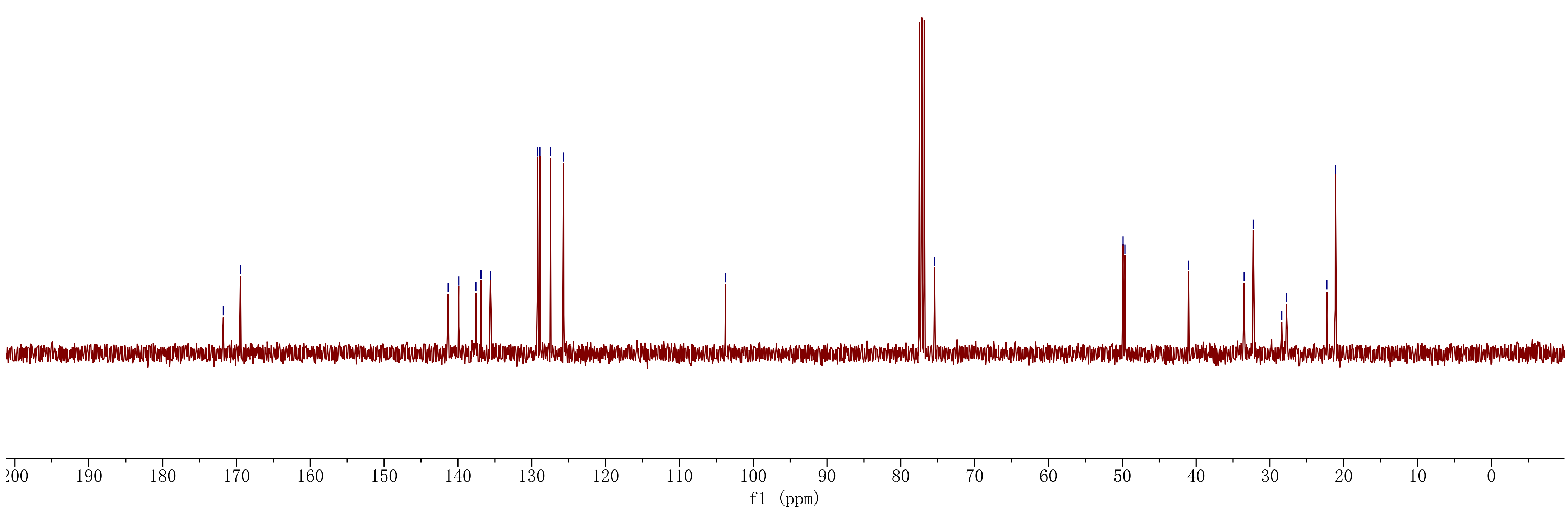

Ph
O
N
NH
O
Ph

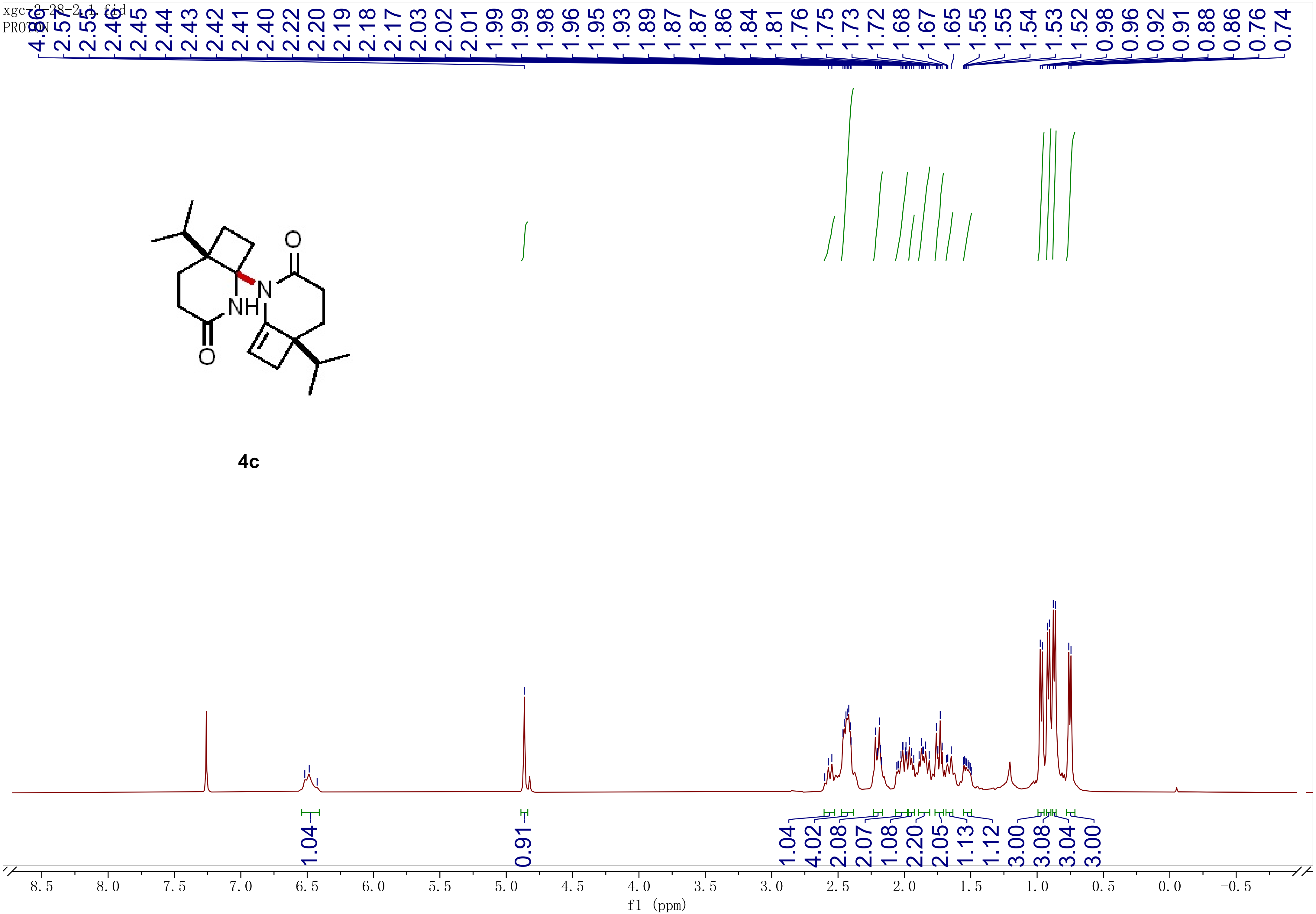
4a

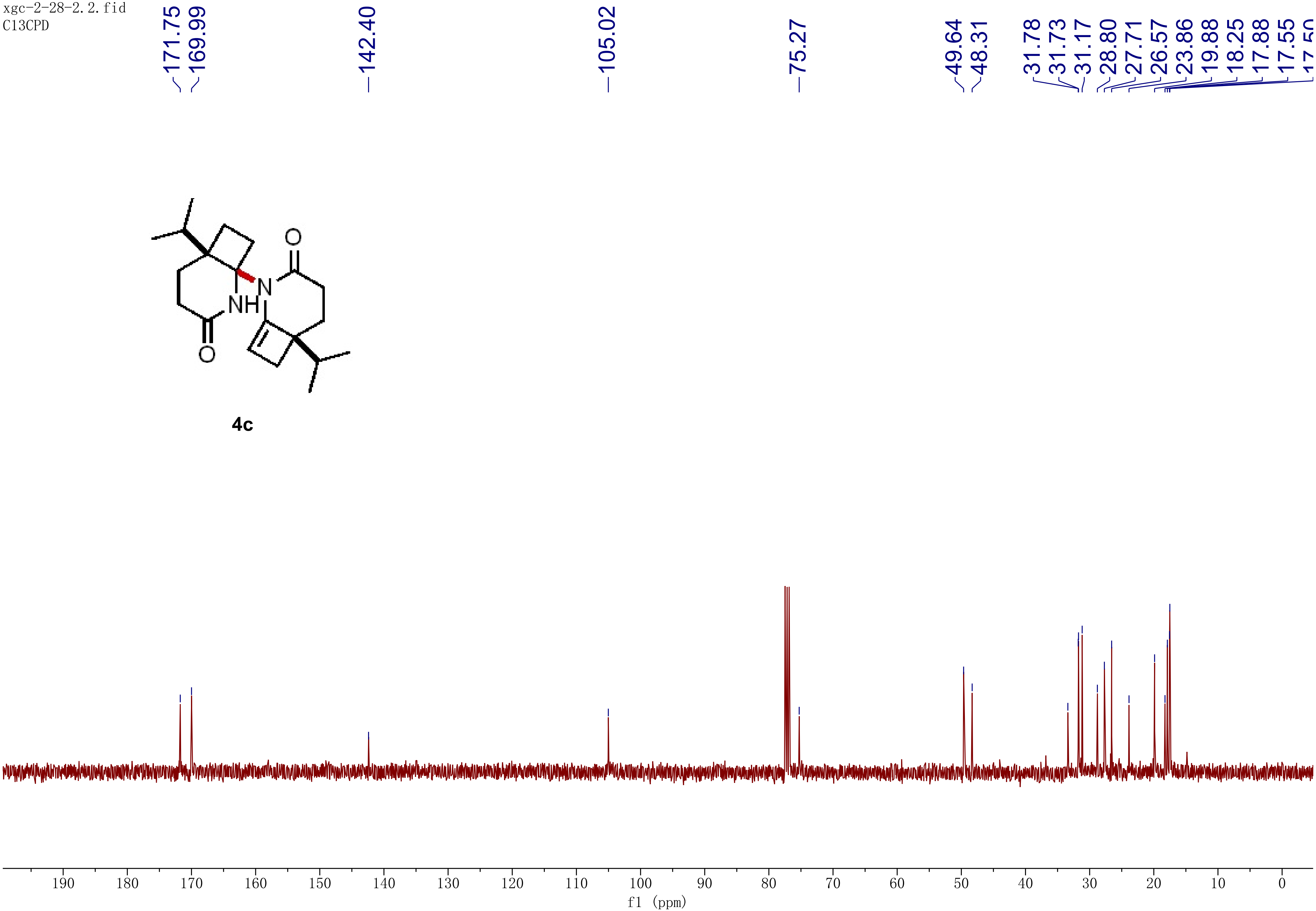

xge-2-22-1.1.fid
PROTON
7.54 7.52 7.18 7.16 6.90 6.85 6.83 6.40 6.38 4.86 3.15 3.12 3.02 2.99 2.73 2.72 2.71 2.70 2.69 2.55 2.54 2.52 2.51 2.48 2.46 2.45 2.44 2.42 2.29 2.27 2.27 2.26 2.26 2.25 2.25 2.24 2.19 2.16 2.11 2.10 2.09 2.09 2.07 2.06 2.04 2.03 2.01 1.84 1.84 1.70 1.67
Tol
O
NH
N
O
Tol
4b
2.00 2.00 0.95 1.99 1.99 1.00 1.02 1.02 2.04 1.02 5.08 5.00 1.03 1.03 1.10 1.04 0.98
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

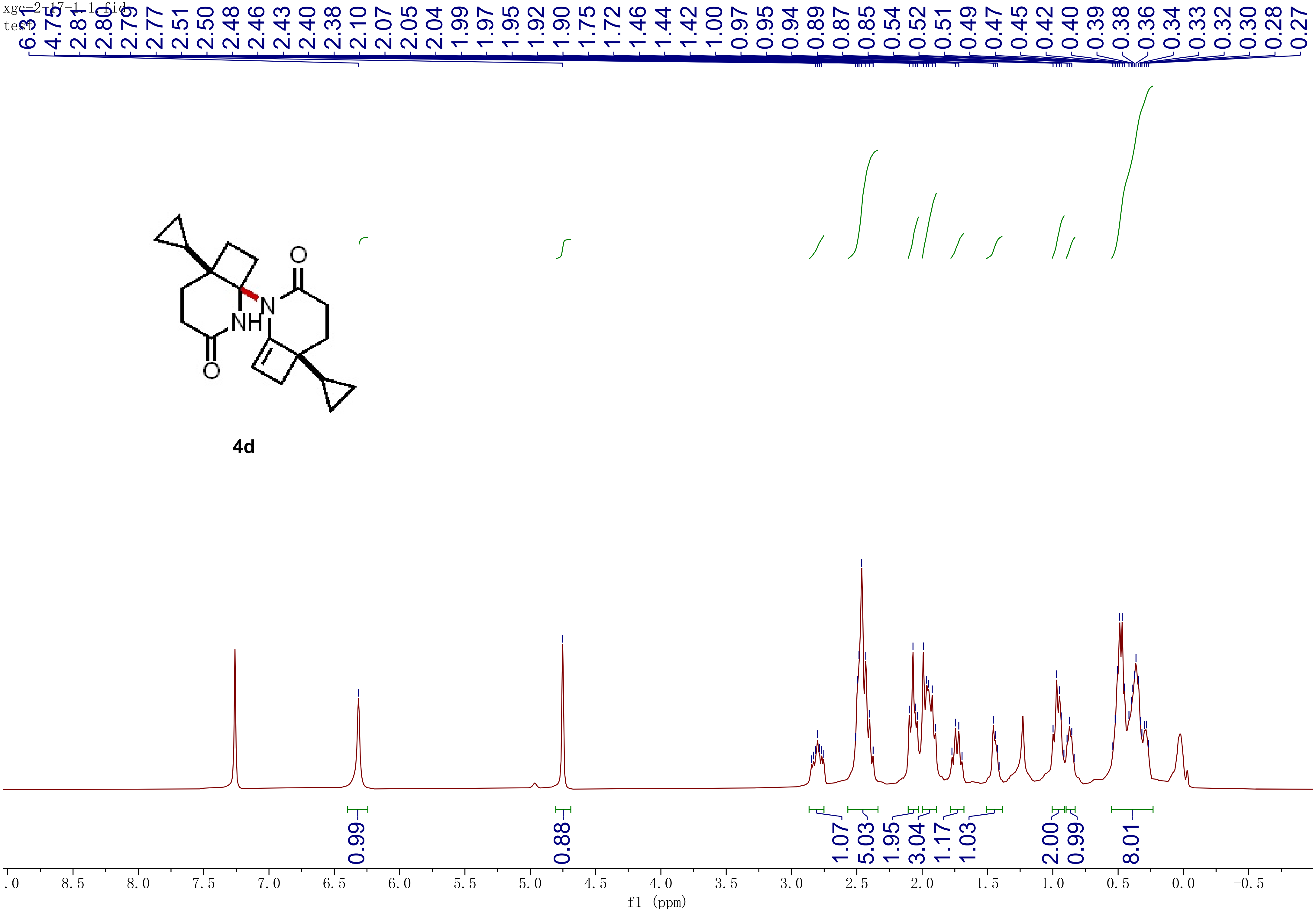

Tol
NH
N
O
O
Tol

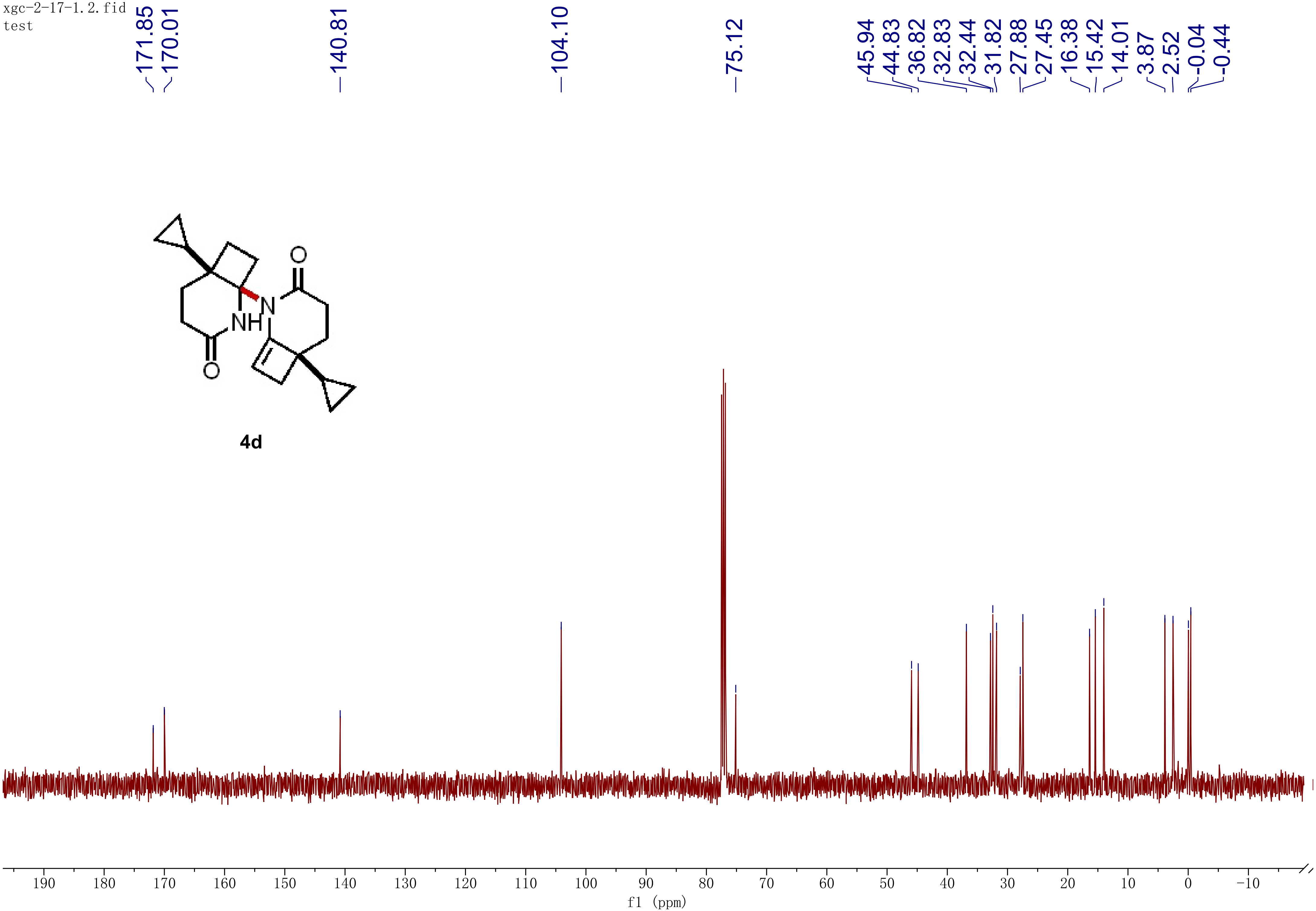
4b

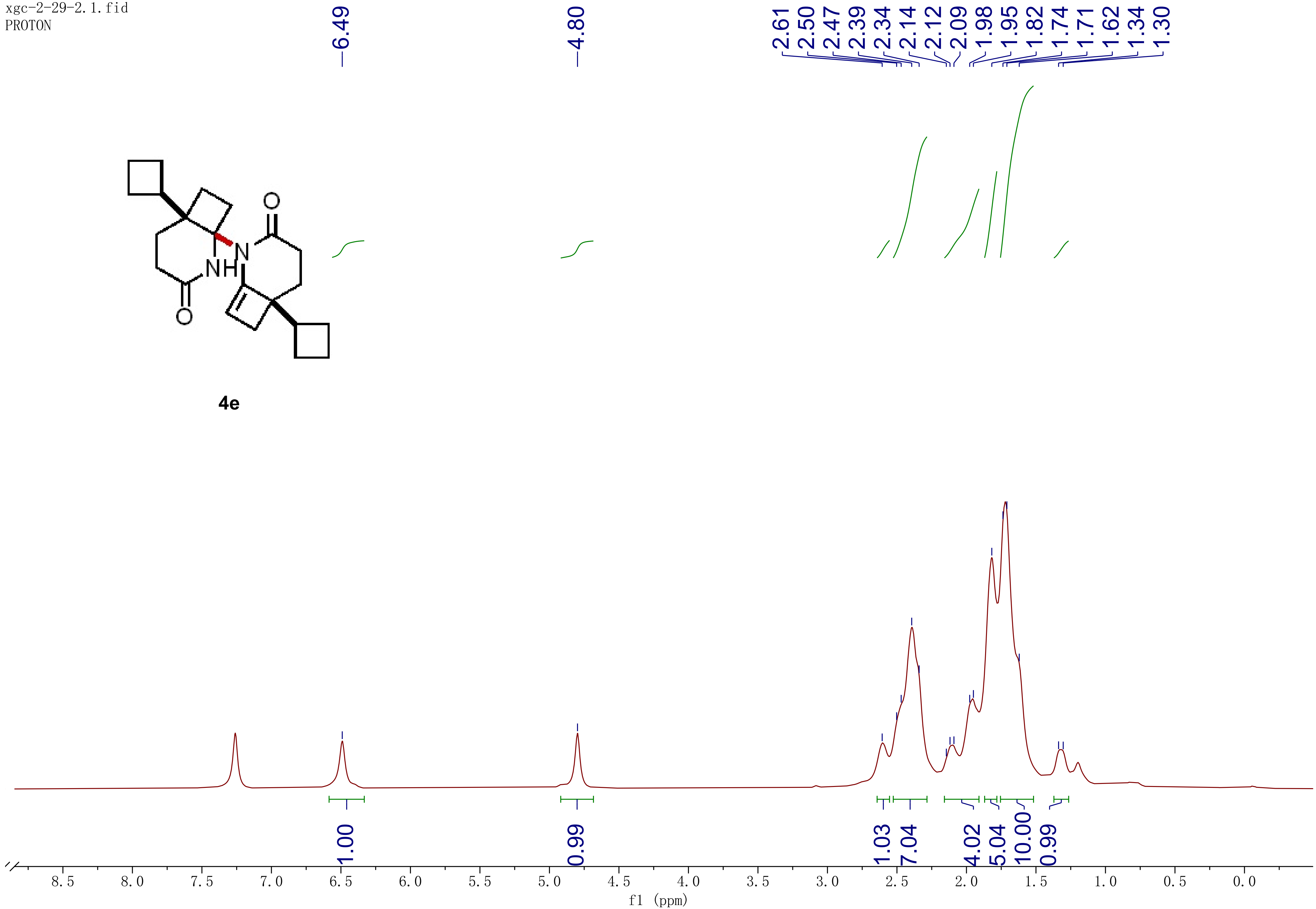

xgc-2-22-1.4.fid
C13CPD
171.78
169.47
141.32
139.88
137.57
136.87
135.59
129.20
128.90
127.46
125.68
103.77
75.42
49.90
49.65
41.03
33.49
32.25
28.40
27.78
22.29
21.14
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


xgc-2-28-2.1.fid
PROTON
4.86 2.57 2.55 2.46 2.45 2.44 2.43 2.42 2.41 2.40 2.22 2.20 2.19 2.18 2.17 2.03 2.02 2.01 1.99 1.99 1.98 1.96 1.95 1.93 1.89 1.87 1.87 1.86 1.84 1.81 1.76 1.75 1.73 1.72 1.68 1.67 1.65 1.55 1.55 1.54 1.53 1.52 0.98 0.96 0.92 0.91 0.88 0.86 0.76 0.74
O
N
NH
O
4c
1.04
0.91
1.04 4.02 2.08 2.07 1.08 2.20 2.05 1.13 1.12 3.00 3.08 3.04 3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

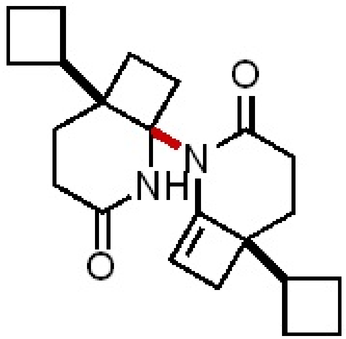

xgc-2-28-2.2.fid
C13CPD
171.75
169.99
142.40
105.02
75.27
49.64
48.31
31.78
31.73
31.17
28.80
27.71
26.57
23.86
19.88
18.25
17.88
17.55
17.50
O
NH
N
O
4c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


xgc-2-17-1-1.fid
test
6.31
4.75
2.81
2.80
2.79
2.77
2.51
2.50
2.48
2.46
2.43
2.40
2.38
2.10
2.07
2.05
2.04
1.99
1.97
1.95
1.92
1.90
1.75
1.72
1.46
1.44
1.42
1.00
0.97
0.95
0.94
0.89
0.87
0.85
0.54
0.52
0.51
0.49
0.47
0.45
0.42
0.40
0.39
0.38
0.36
0.34
0.33
0.32
0.30
0.28
0.27
NH
N
O
O
4d
0.99
0.88
1.07
5.03
1.95
3.04
1.17
1.03
2.00
0.99
8.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

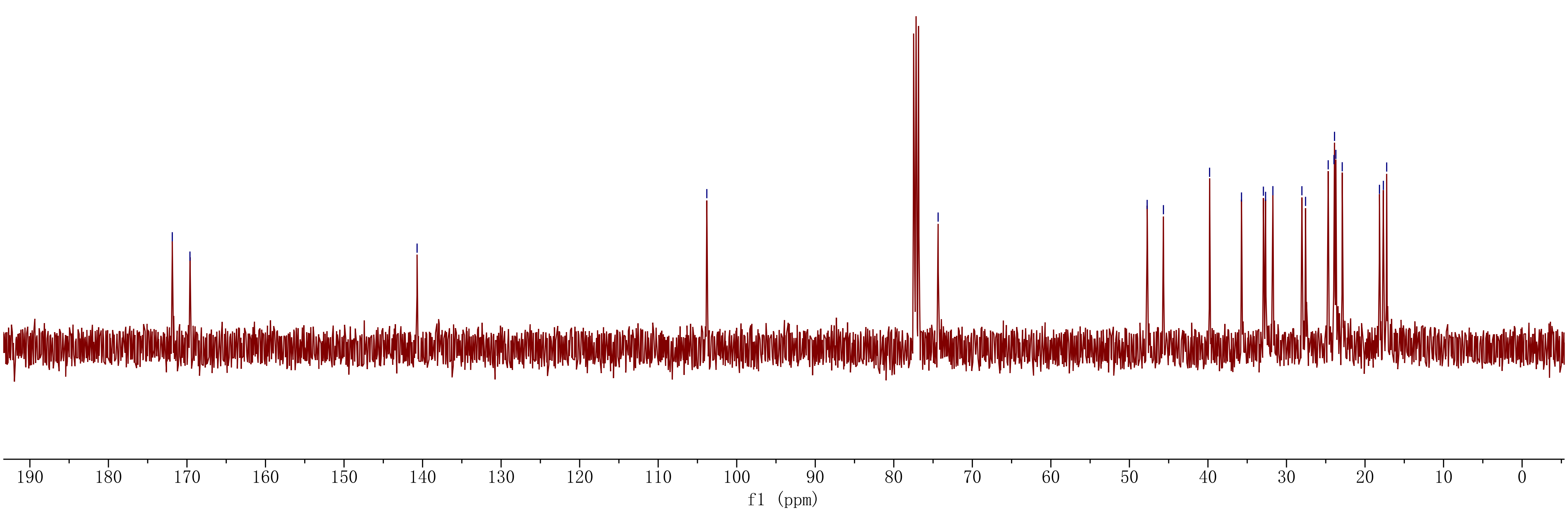

xgc-2-17-1.2.fid
test
171.85
170.01
140.81
104.10
75.12
45.94
44.83
36.82
32.83
32.44
31.82
27.88
27.45
16.38
15.42
14.01
3.87
2.52
-0.04
-0.44
NH
N
O
O
4d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

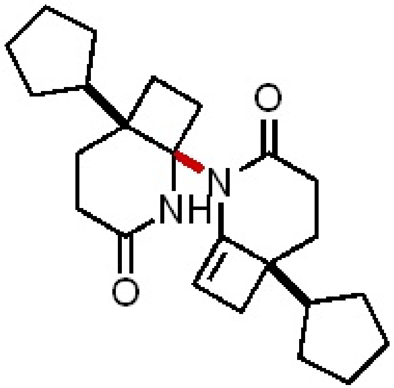

xgc-2-29-2.1.fid
PROTON
6.49
4.80
2.61 2.50 2.47 2.39 2.34 2.14 2.12 2.09 1.98 1.95 1.82 1.74 1.71 1.62 1.34 1.30
O
N
NH
O
4e
1.00
0.99
1.03
7.04
4.02
5.04
10.00
0.99
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

xgc-2-29-2.3.fid
C13CPD

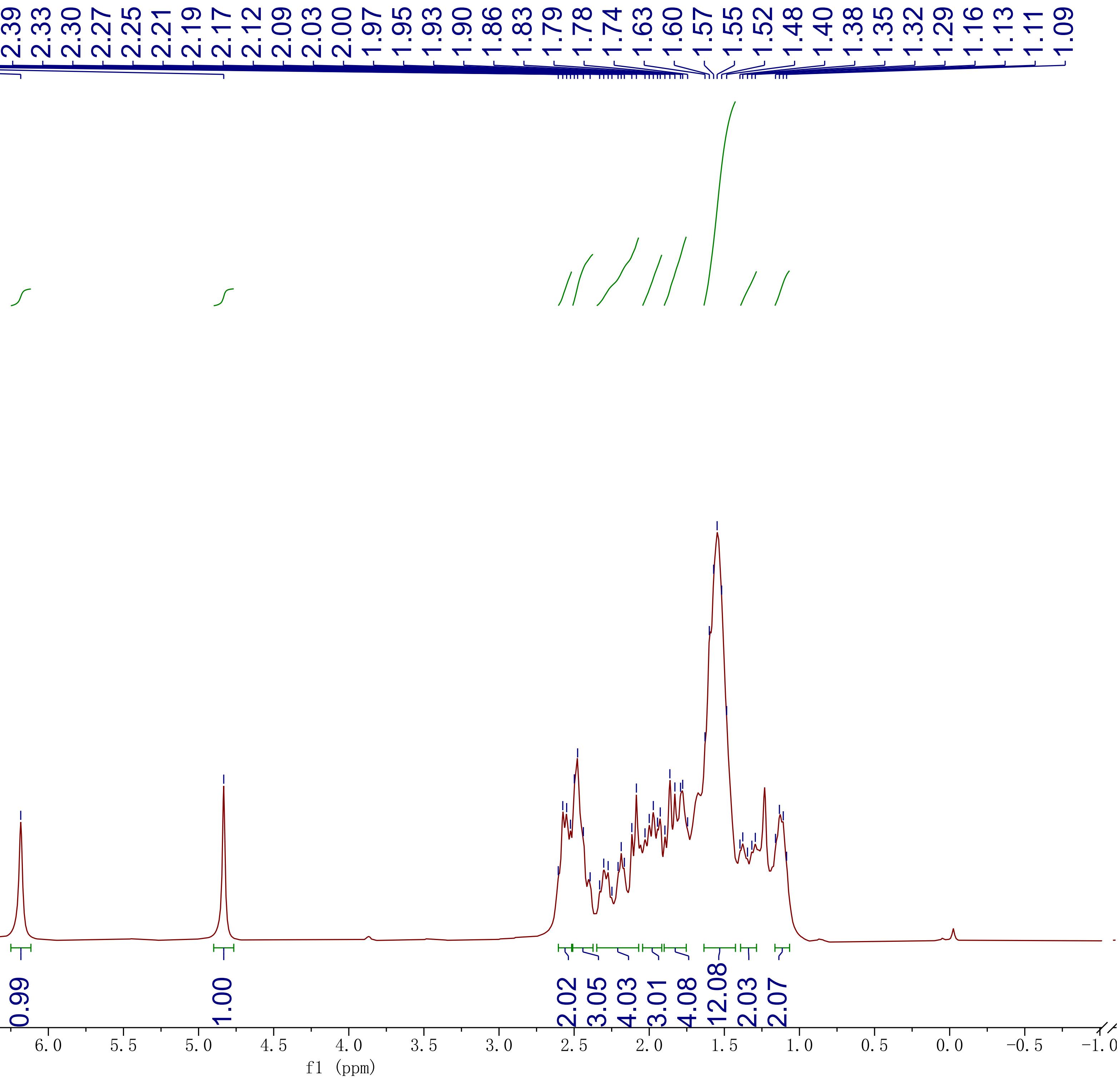


4e

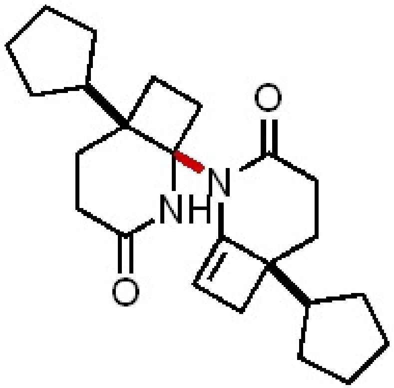



O
NH
N
O

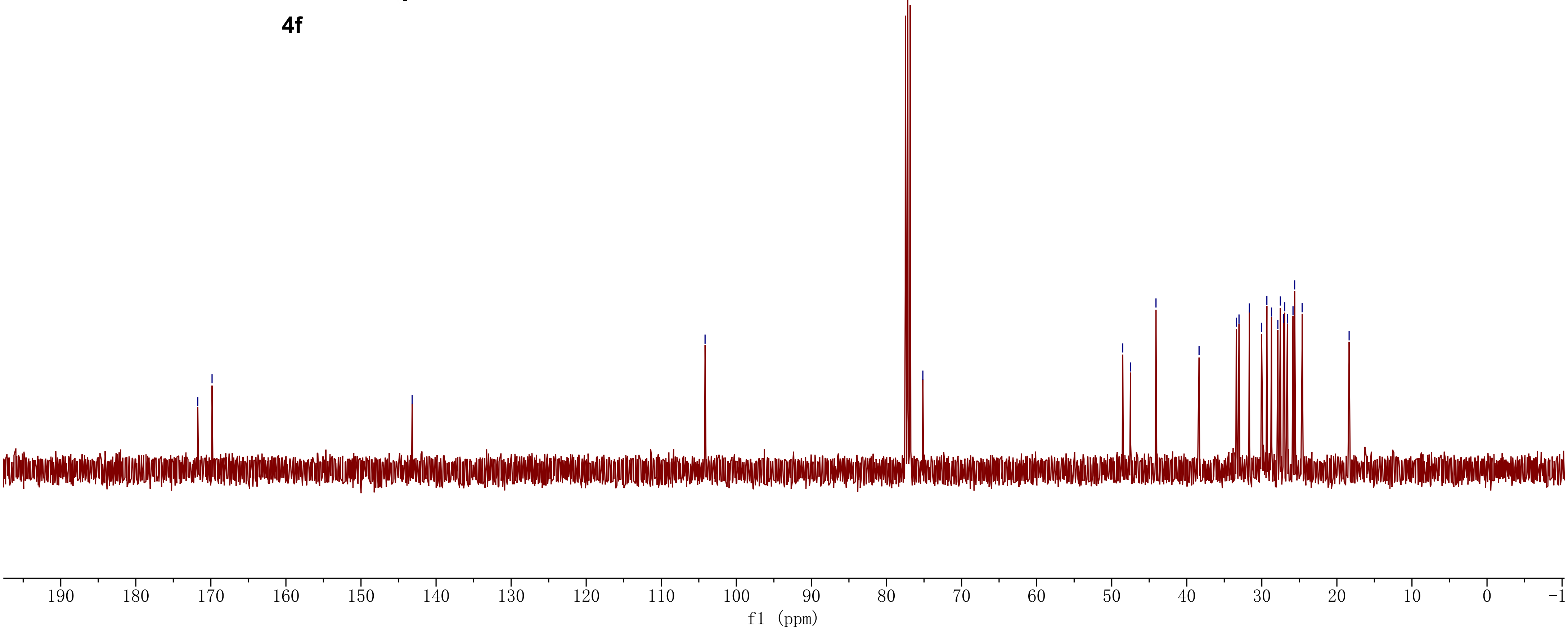
4f

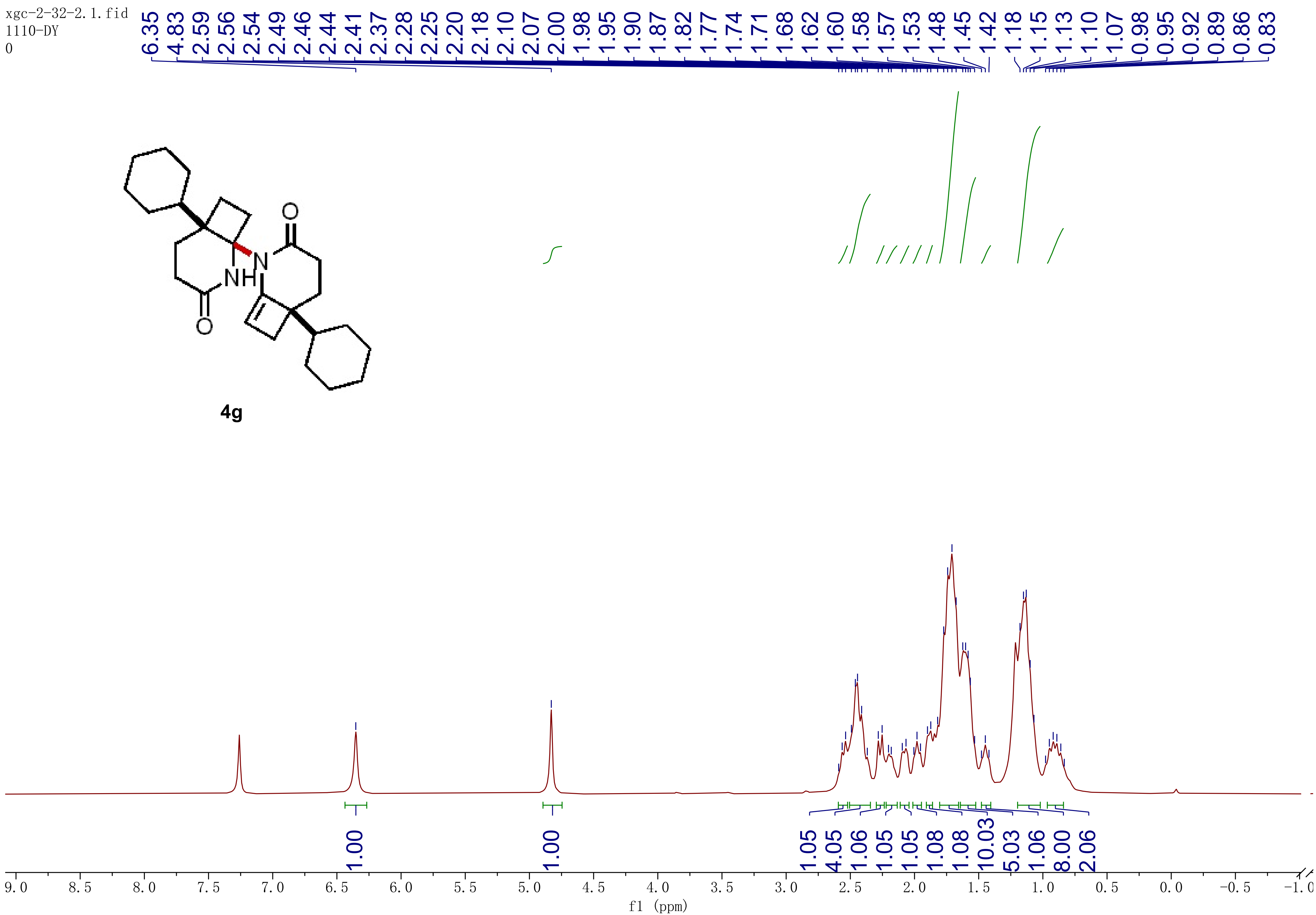

xgc-2-32-1.1.fid
1110-DY
0
6.18 4.83 2.61 2.58 2.55 2.52 2.50 2.48 2.44 2.39 2.33 2.30 2.27 2.25 2.21 2.19 2.17 2.12 2.09 2.03 2.00 1.97 1.95 1.93 1.90 1.86 1.83 1.79 1.78 1.74 1.63 1.60 1.57 1.55 1.52 1.48 1.40 1.38 1.35 1.32 1.29 1.16 1.13 1.11 1.09
0.99
1.00
2.02
3.05
4.03
3.01
4.08
12.08
2.03
2.07
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

xgc-2-32-1-C.1.fid
13CPD

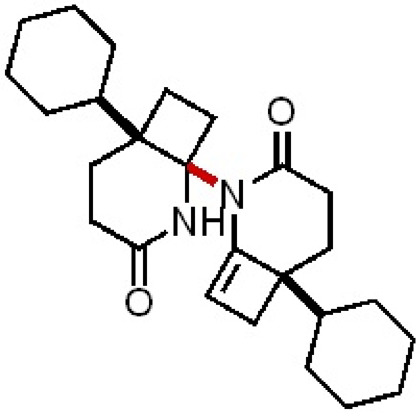


4f

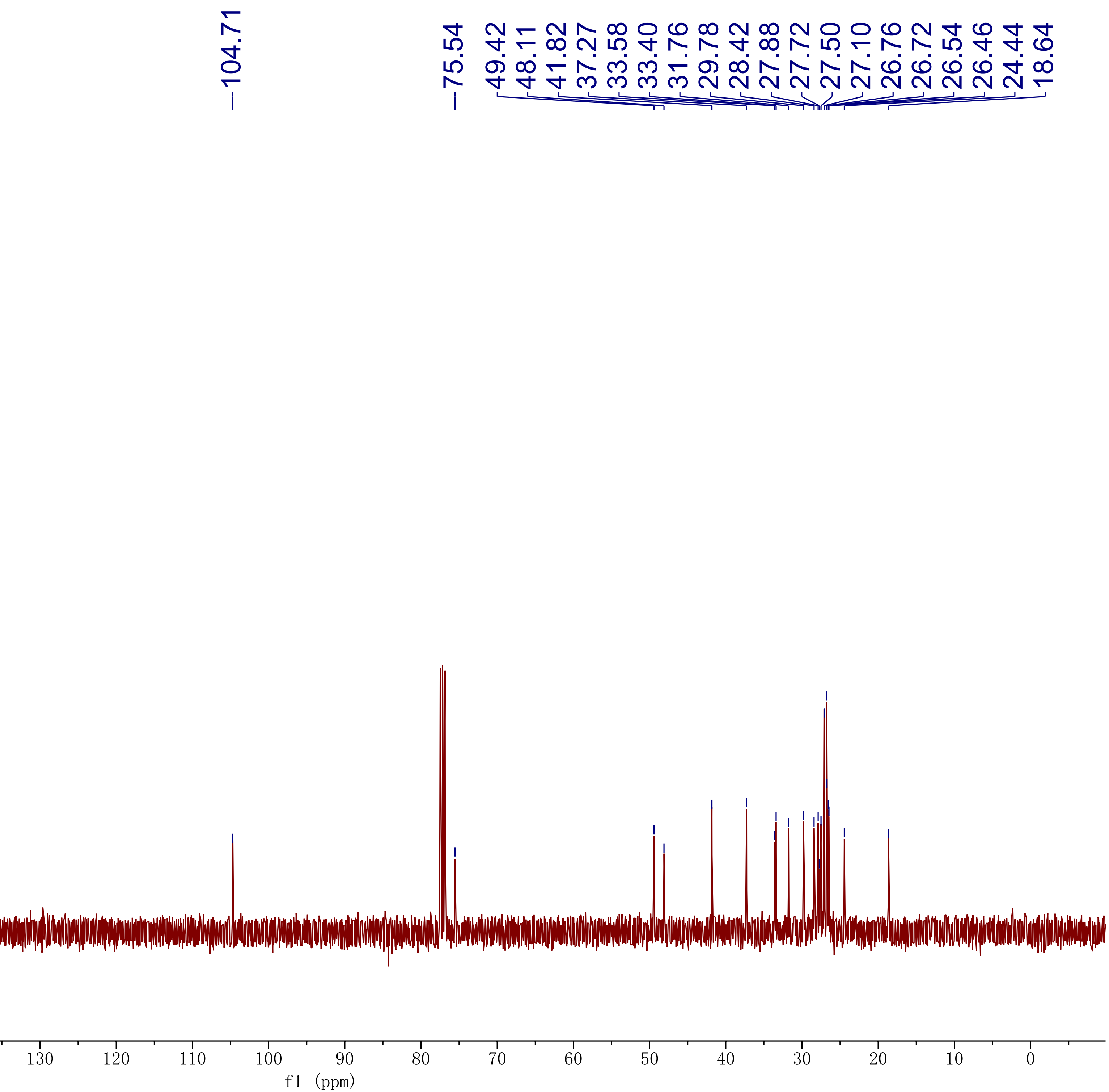


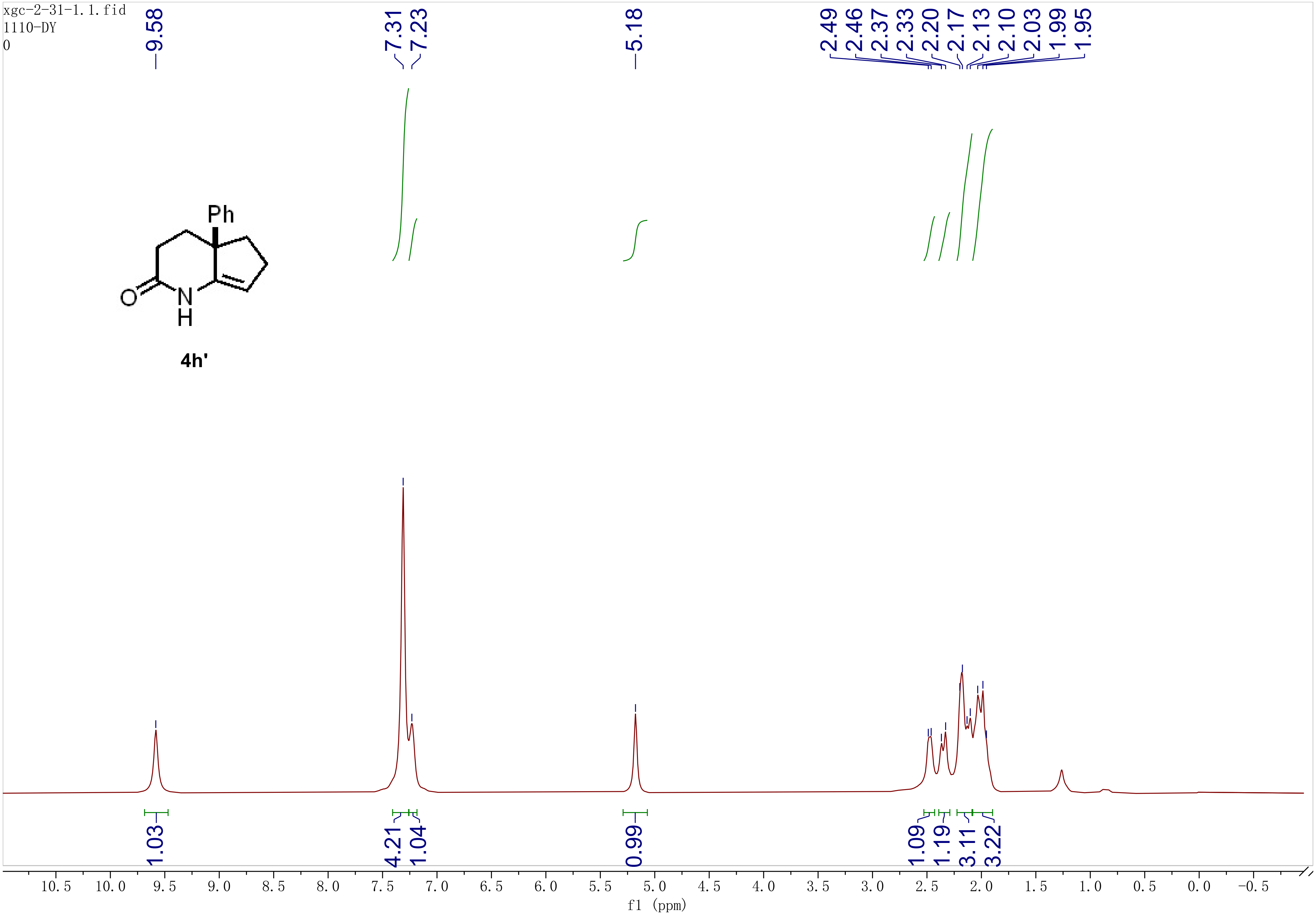

xgc-2-32-2.1.fid
1110-DY
0
6.35 4.83 2.59 2.56 2.54 2.49 2.46 2.44 2.41 2.37 2.28 2.25 2.20 2.18 2.10 2.07 2.00 1.98 1.95 1.90 1.87 1.82 1.77 1.74 1.71 1.68 1.62 1.60 1.58 1.57 1.53 1.48 1.45 1.42 1.18 1.15 1.13 1.10 1.07 0.98 0.95 0.92 0.89 0.86 0.83
4g
1.00 1.00 1.05 4.05 1.06 1.05 1.05 1.08 1.08 10.03 5.03 1.06 8.00 2.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

xgc-2-32-2.3.fid
13CPD

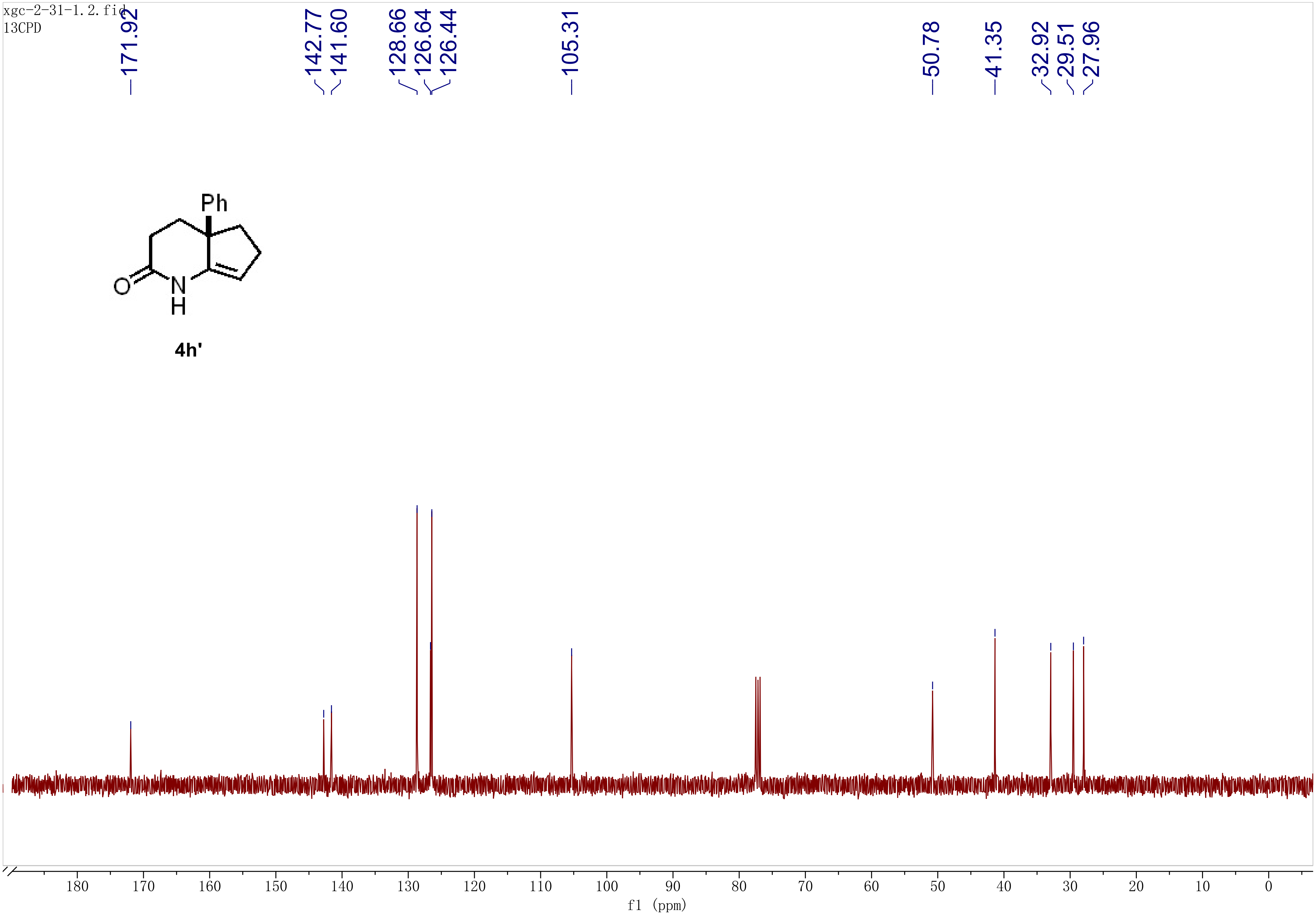


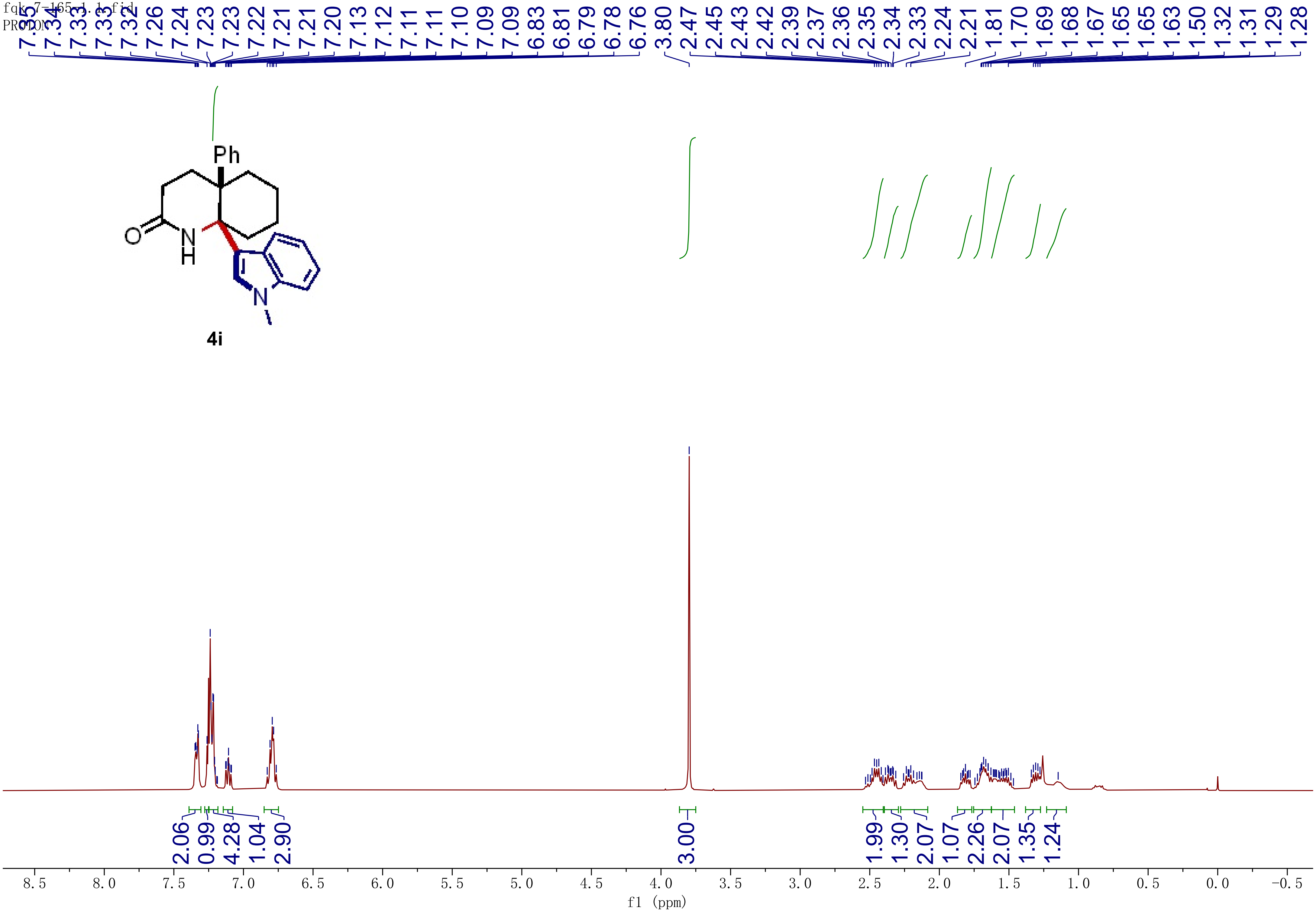
4g

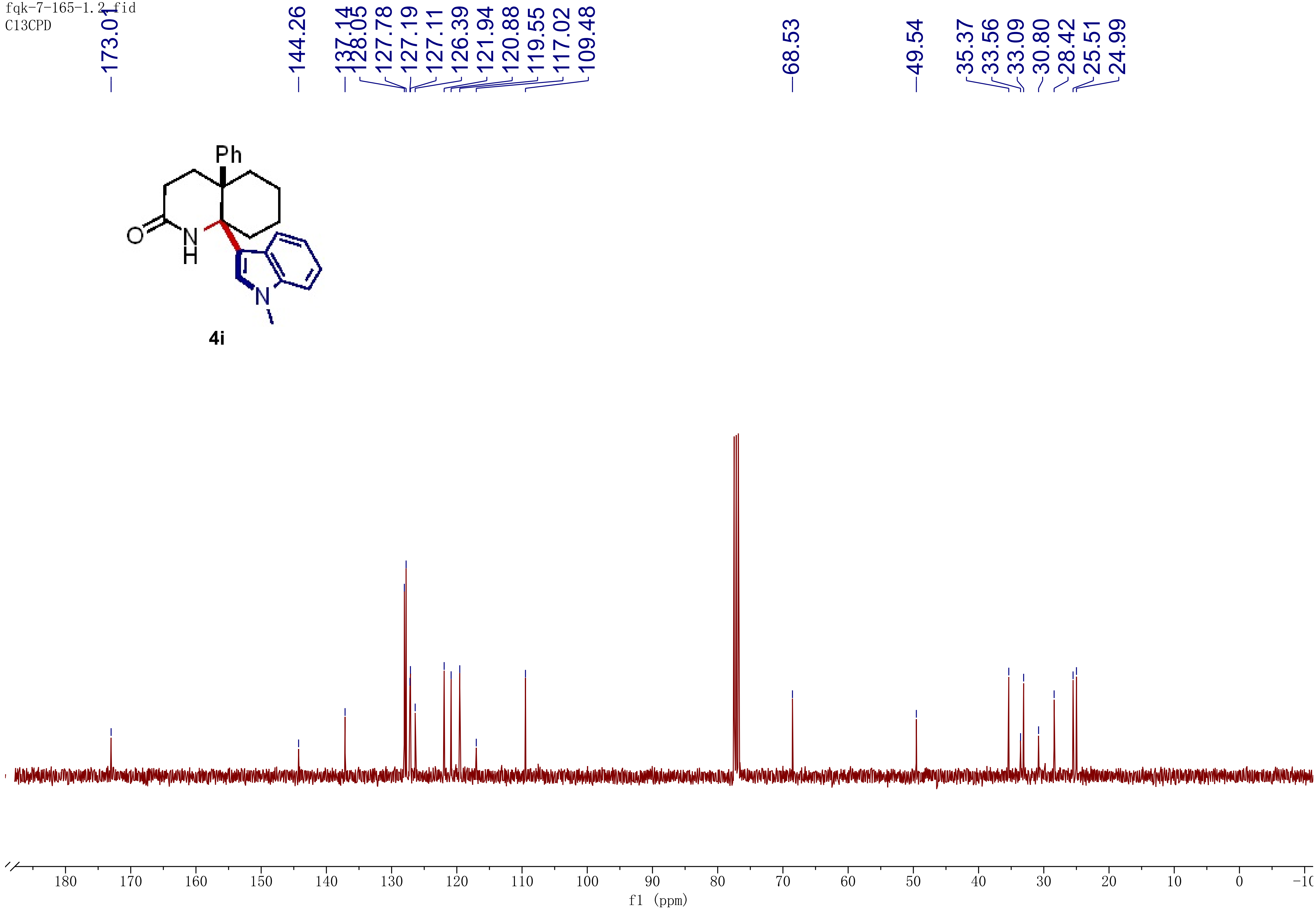


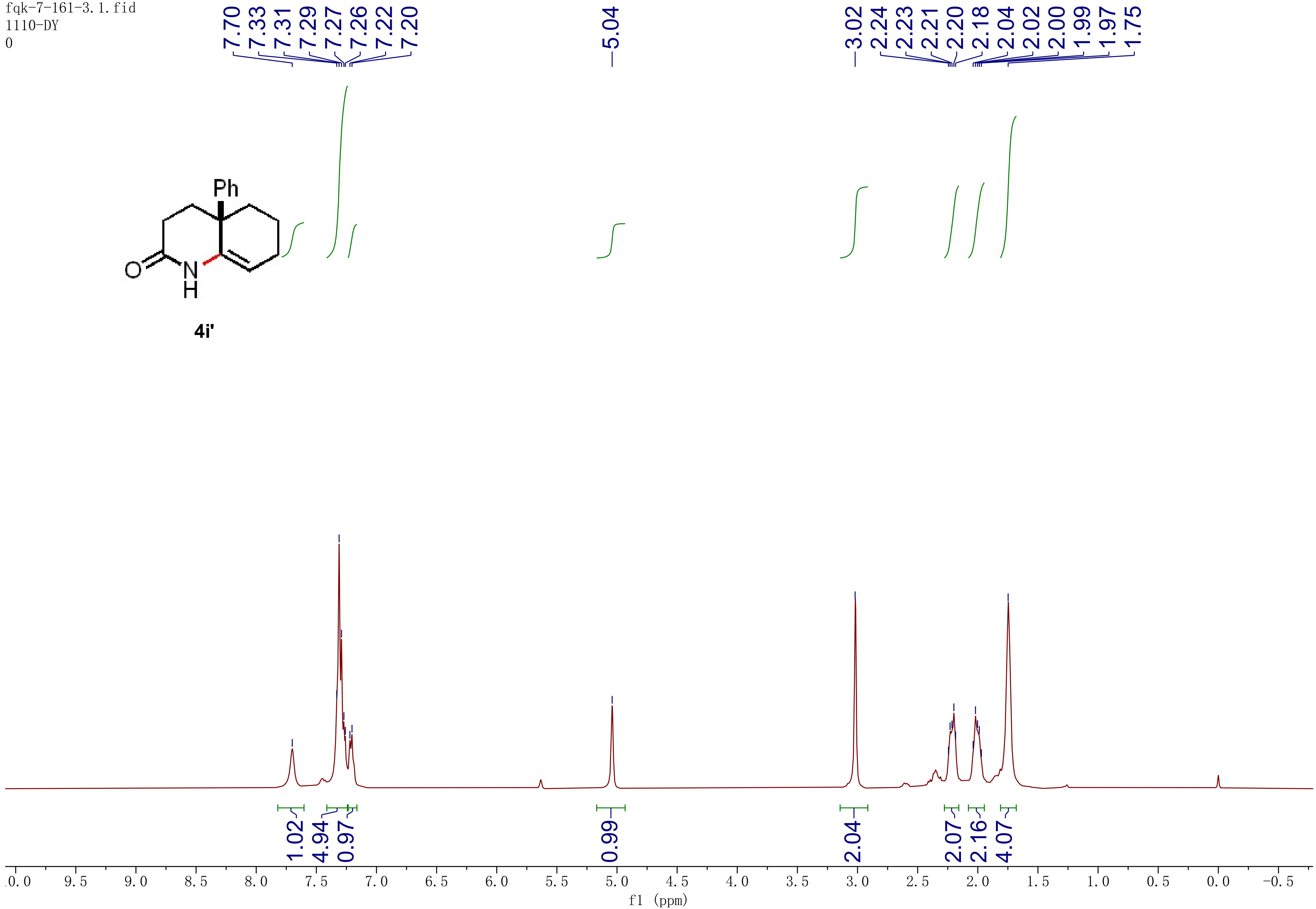


xgc-2-31-1.1.fid
1110-DY
0
9.58
7.31
7.23
5.18
2.49
2.46
2.37
2.33
2.20
2.17
2.13
2.10
2.03
1.99
1.95
Ph
O
N
H
4h'
1.03
4.21
1.04
0.99
1.09
1.19
3.11
3.22
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

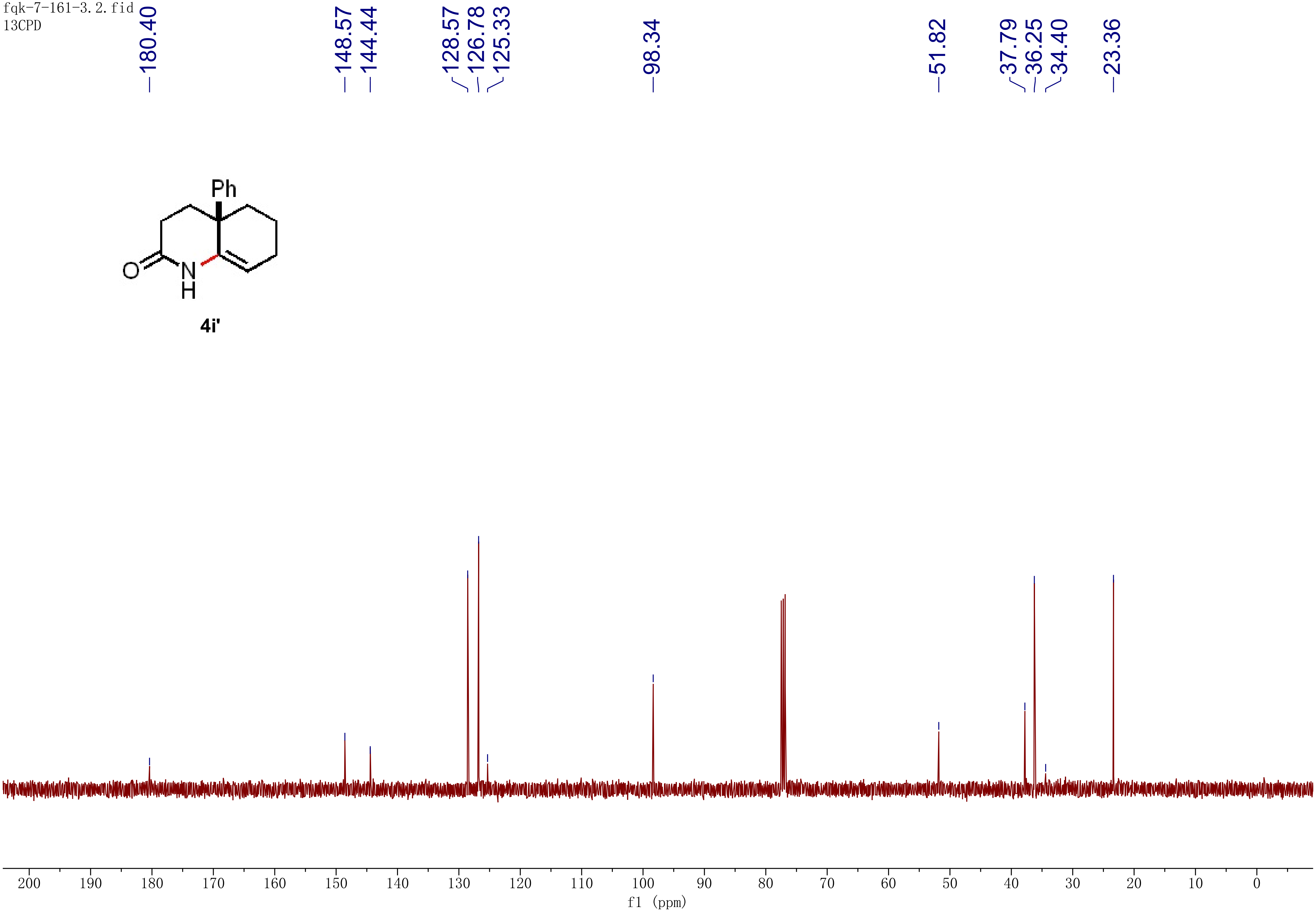

xgc-2-31-1.2.fid
13CPD
171.92
142.77
141.60
128.66
126.64
126.44
105.31
50.78
41.35
32.92
29.51
27.96
Ph
O
N
H
4h'
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

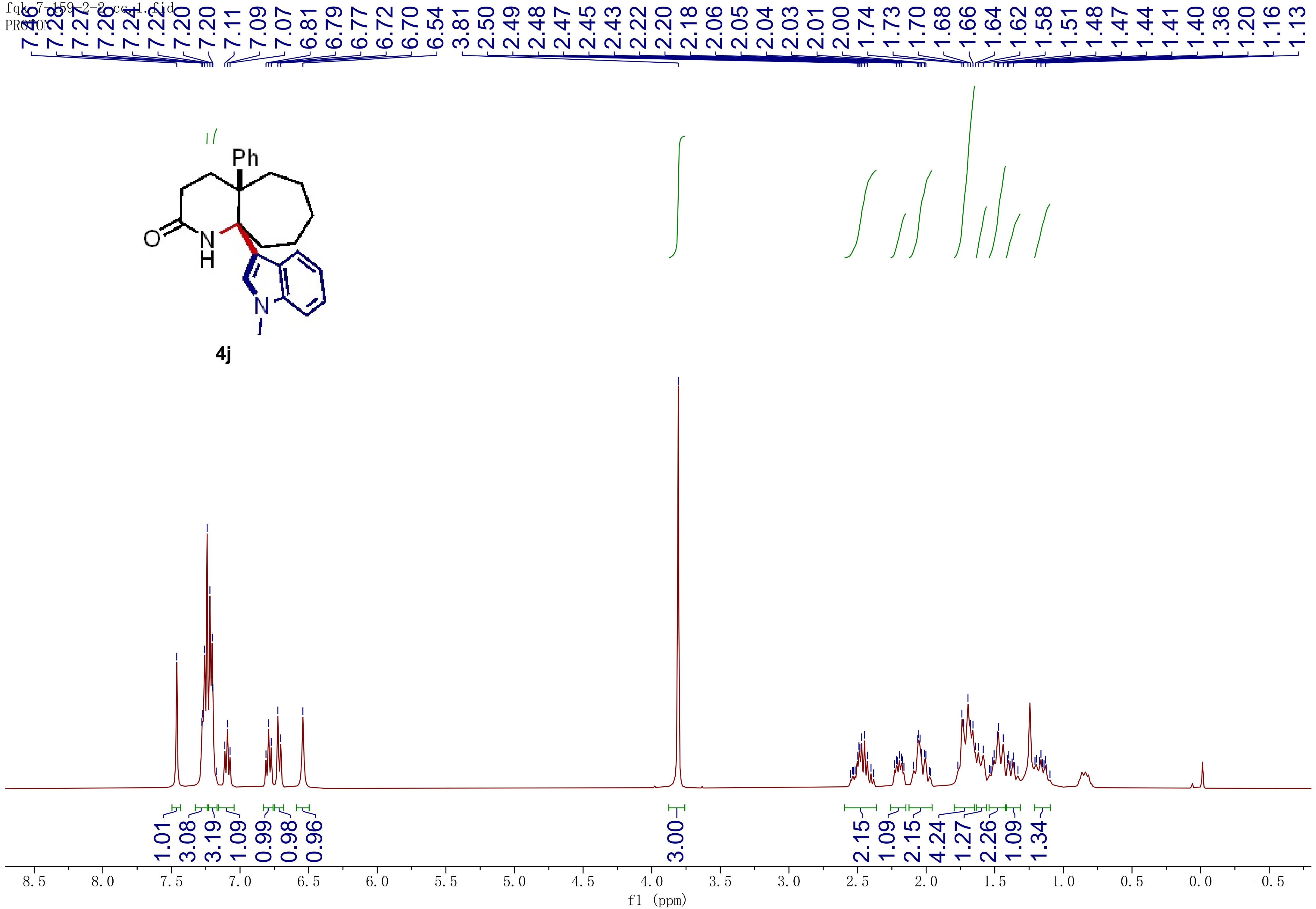

fqk-7-165-1.1.fid
PROTON
7.34
7.33
7.33
7.32
7.26
7.24
7.23
7.23
7.22
7.21
7.21
7.20
7.13
7.12
7.11
7.11
7.10
7.09
7.09
6.83
6.81
6.79
6.78
6.76
3.80
2.47
2.45
2.43
2.42
2.39
2.37
2.36
2.35
2.34
2.33
2.24
2.21
1.81
1.70
1.69
1.68
1.67
1.65
1.65
1.63
1.50
1.32
1.31
1.29
1.28
Ph
O
N
H
N
4i
2.06
0.99
4.28
1.04
2.90
3.00
1.99
1.30
2.07
1.07
2.26
2.07
1.35
1.24
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


fqk-7-165-1.2.fid
C13CPD
173.01
144.26
137.14
128.05
127.78
127.19
127.11
126.39
121.94
120.88
119.55
117.02
109.48
68.53
49.54
35.37
33.56
33.09
30.80
28.42
25.51
24.99
Ph
O
N
H
N
4i
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

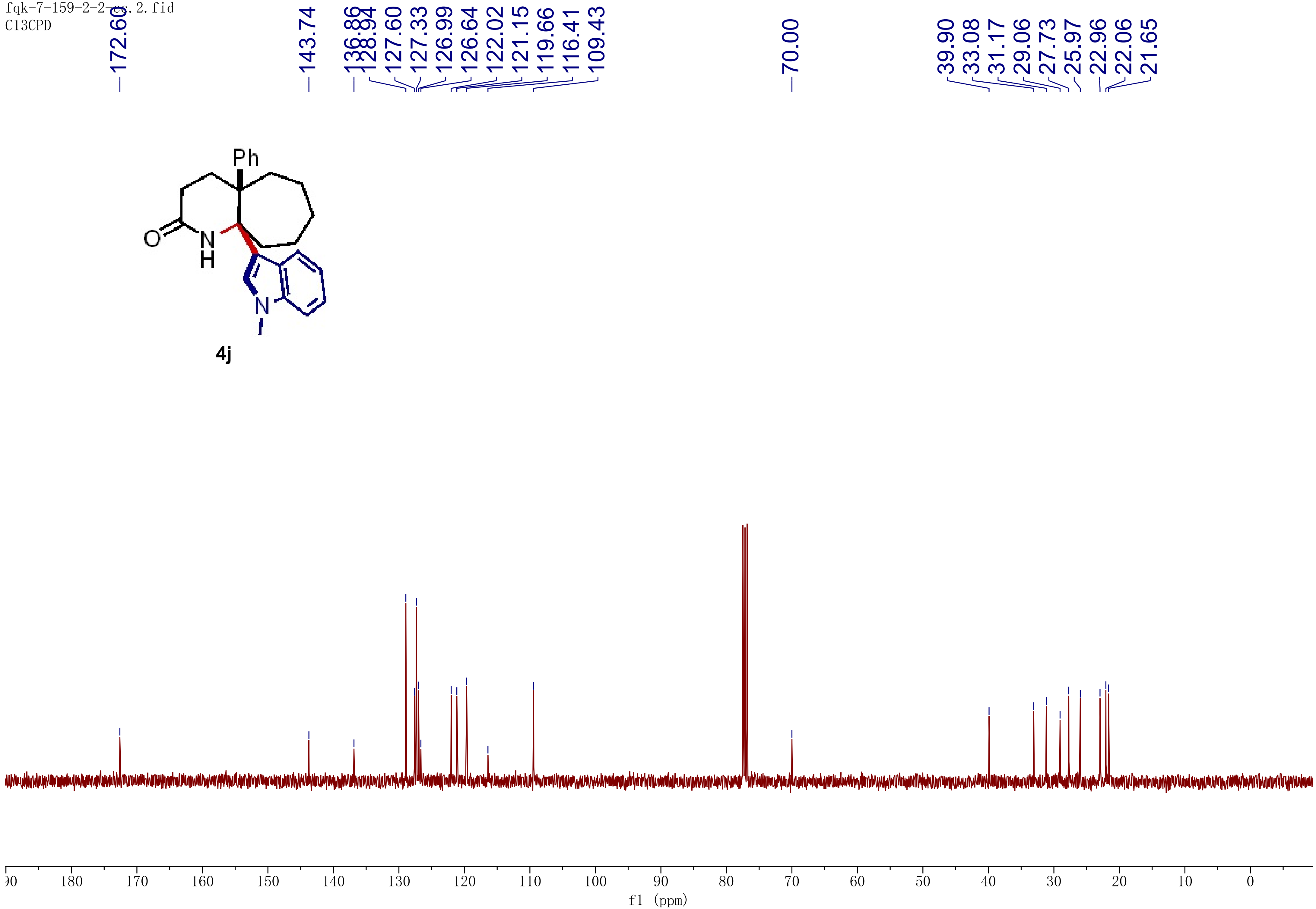

fqk-7-161-3.1.fid
1110-DY
0
7.70
7.33
7.31
7.29
7.27
7.26
7.22
7.20
5.04
3.02
2.24
2.23
2.21
2.20
2.18
2.04
2.02
2.00
1.99
1.97
1.75
Ph
O
N
H
4i'
1.02
4.94
0.97
0.99
2.04
2.07
2.16
4.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

fqk-7-161-3.2.fid
13CPD

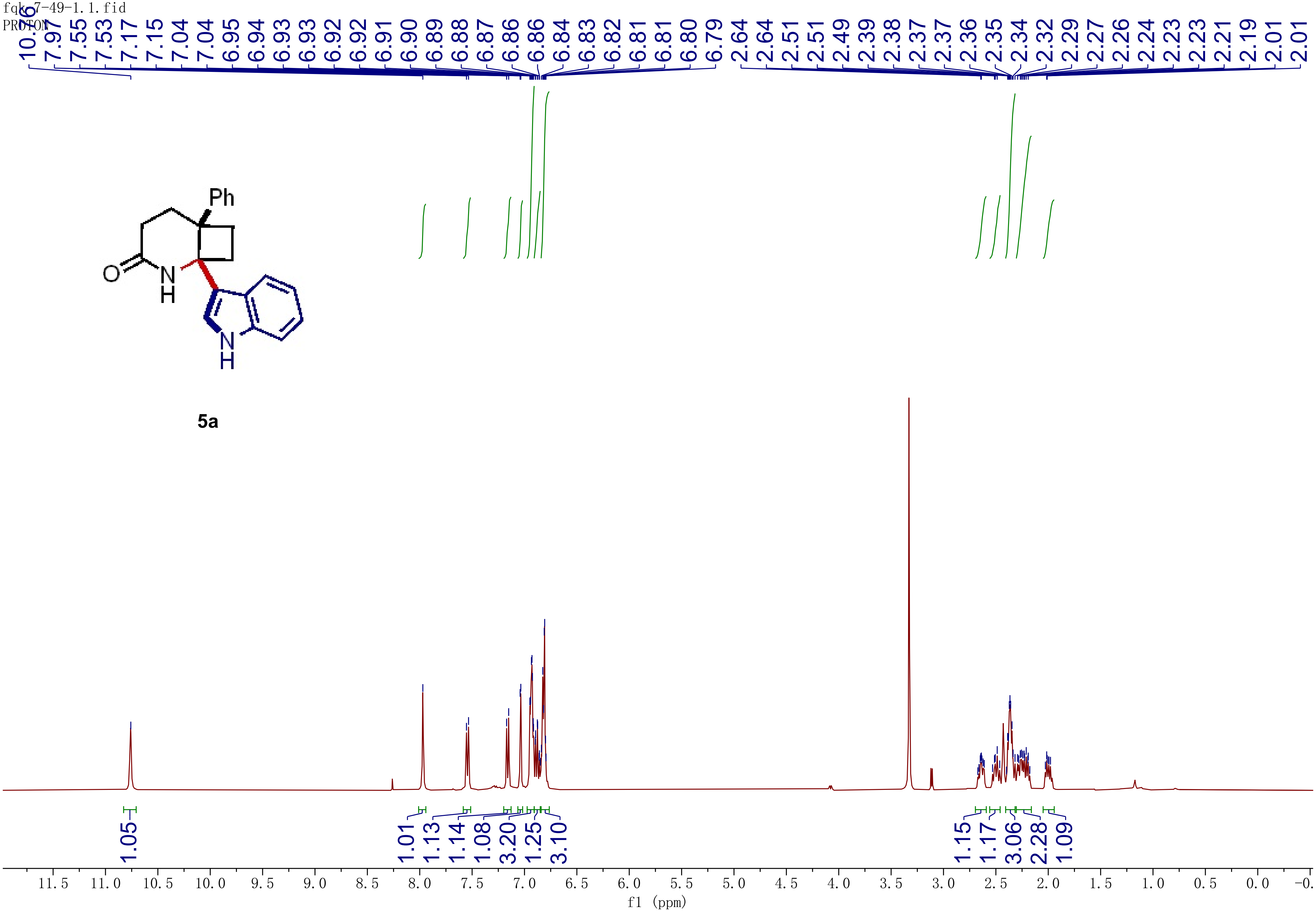


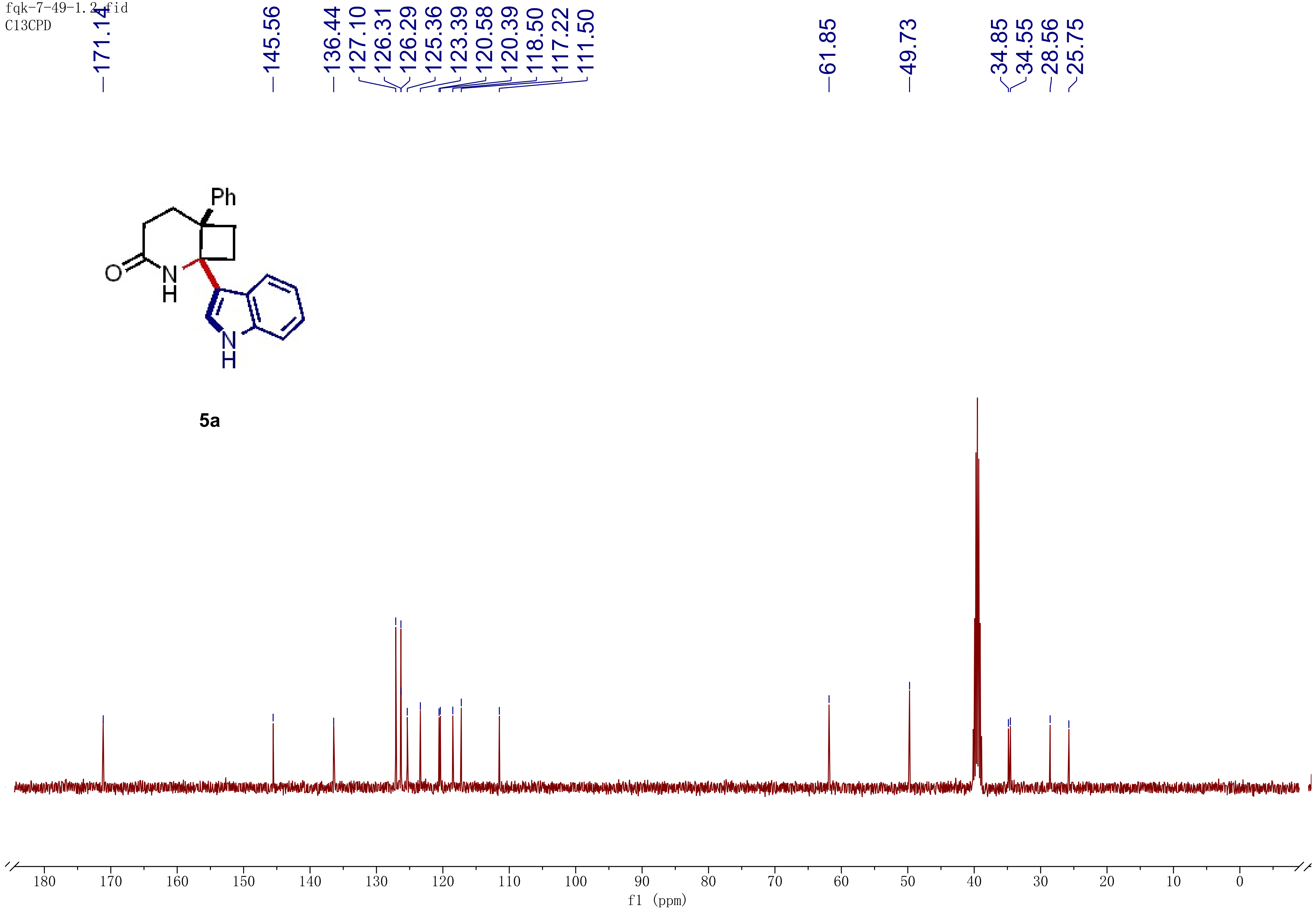

fqk-7-159-2-2-cc-1.fid
PROTON
7.46 7.28 7.27 7.26 7.24 7.22 7.20 7.20 7.11 7.09 7.07 6.81 6.79 6.77 6.72 6.70 6.54 3.81 2.50 2.49 2.48 2.47 2.45 2.43 2.22 2.20 2.18 2.06 2.05 2.04 2.03 2.01 2.00 1.74 1.73 1.70 1.68 1.66 1.64 1.62 1.58 1.51 1.48 1.47 1.44 1.41 1.40 1.36 1.20 1.16 1.13
Ph
O
N
H
N
4j
1.01 3.08 3.19 1.09 0.99 0.98 0.96 3.00 2.15 1.09 2.15 4.24 1.27 2.26 1.09 1.34
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

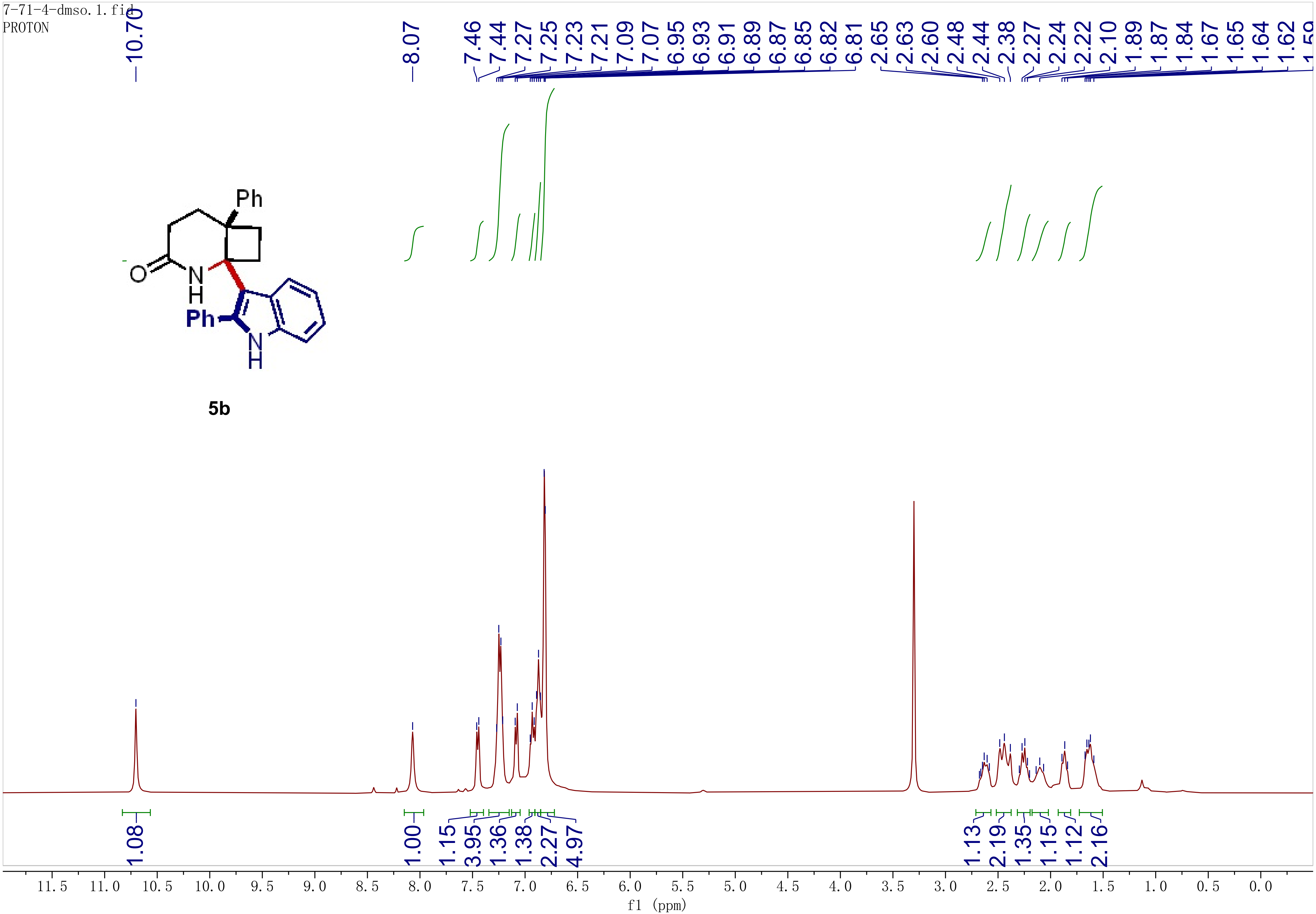
fqk-7-159-2-2-cs.2.fid
C13CPD

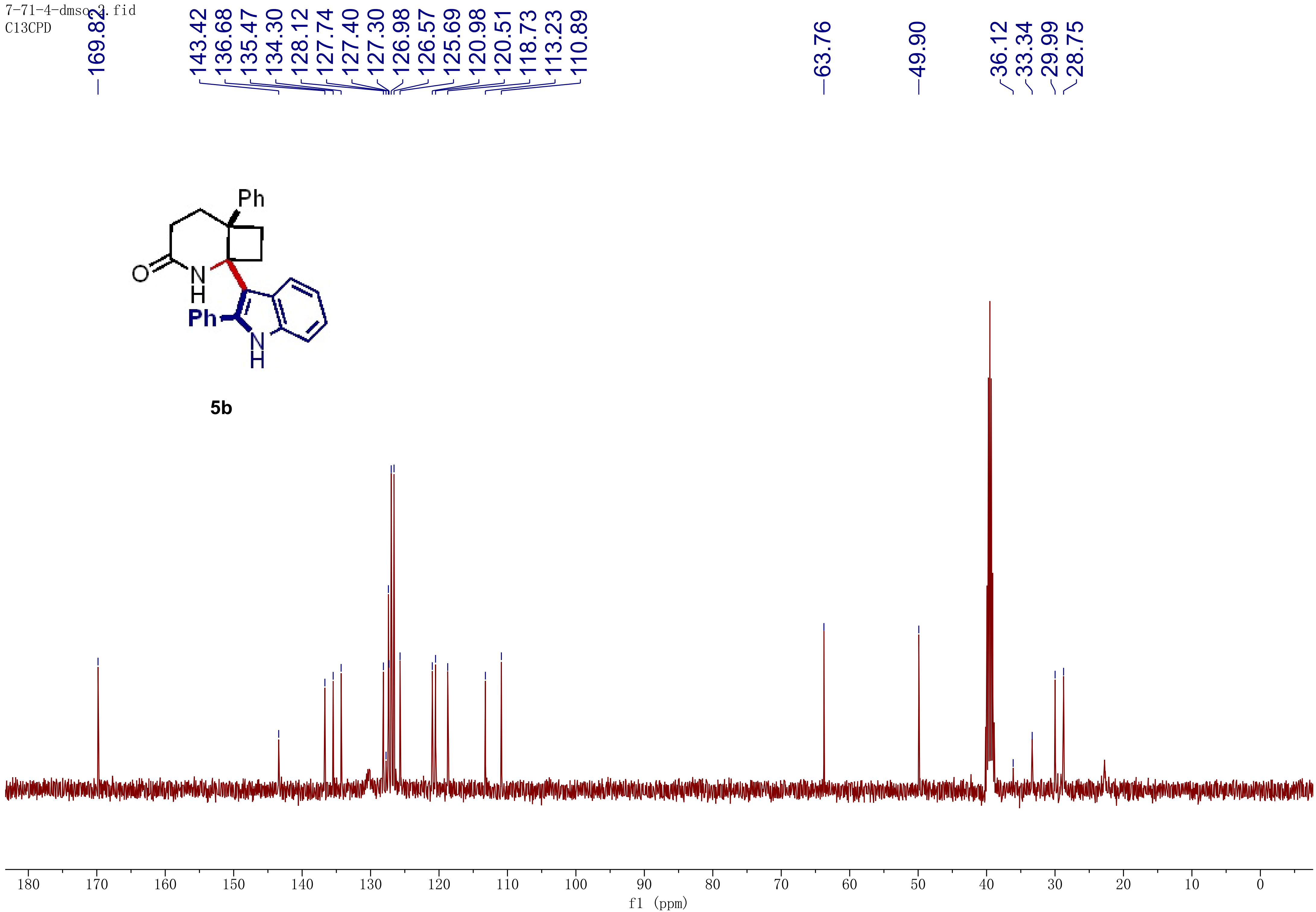


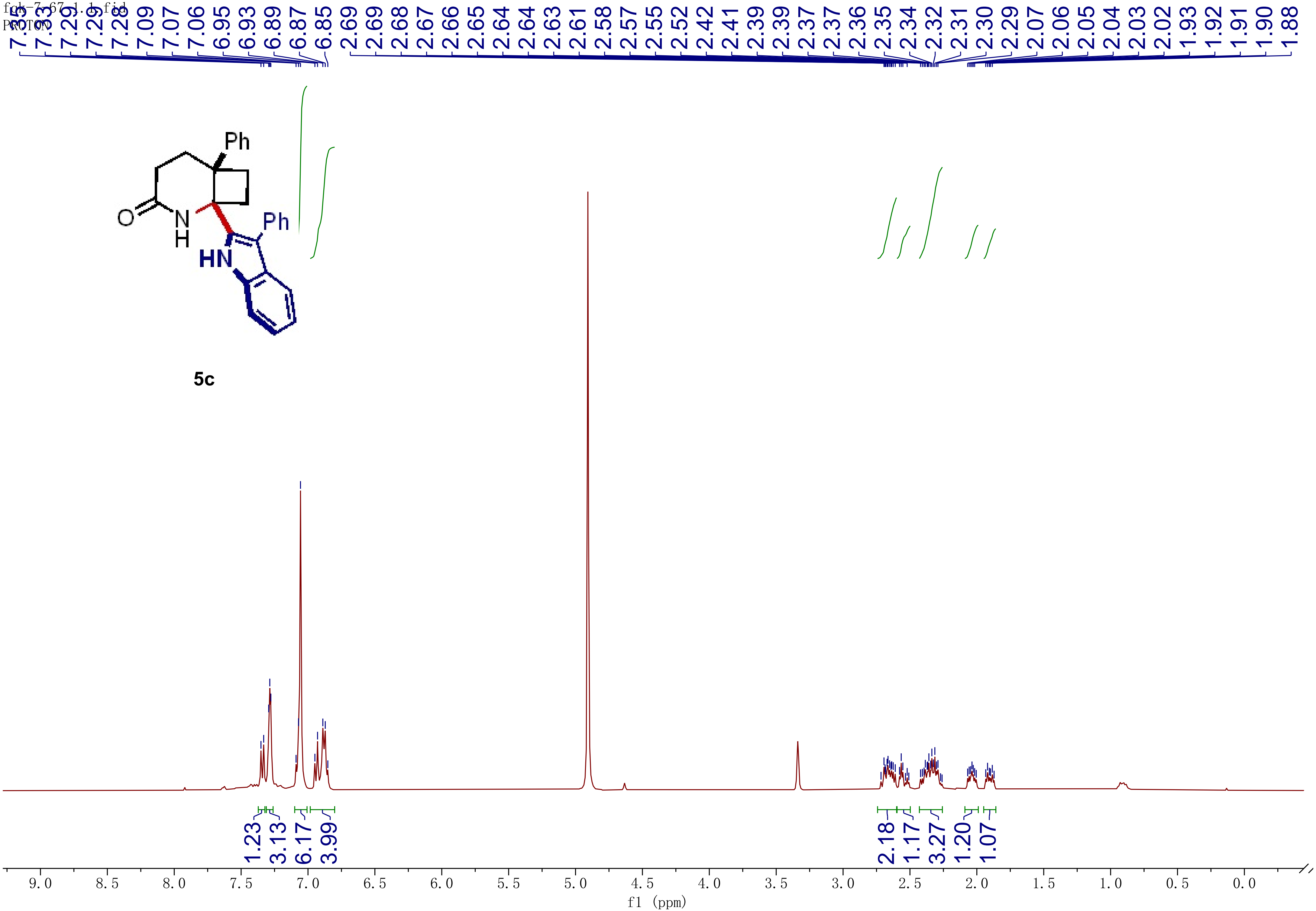
4j

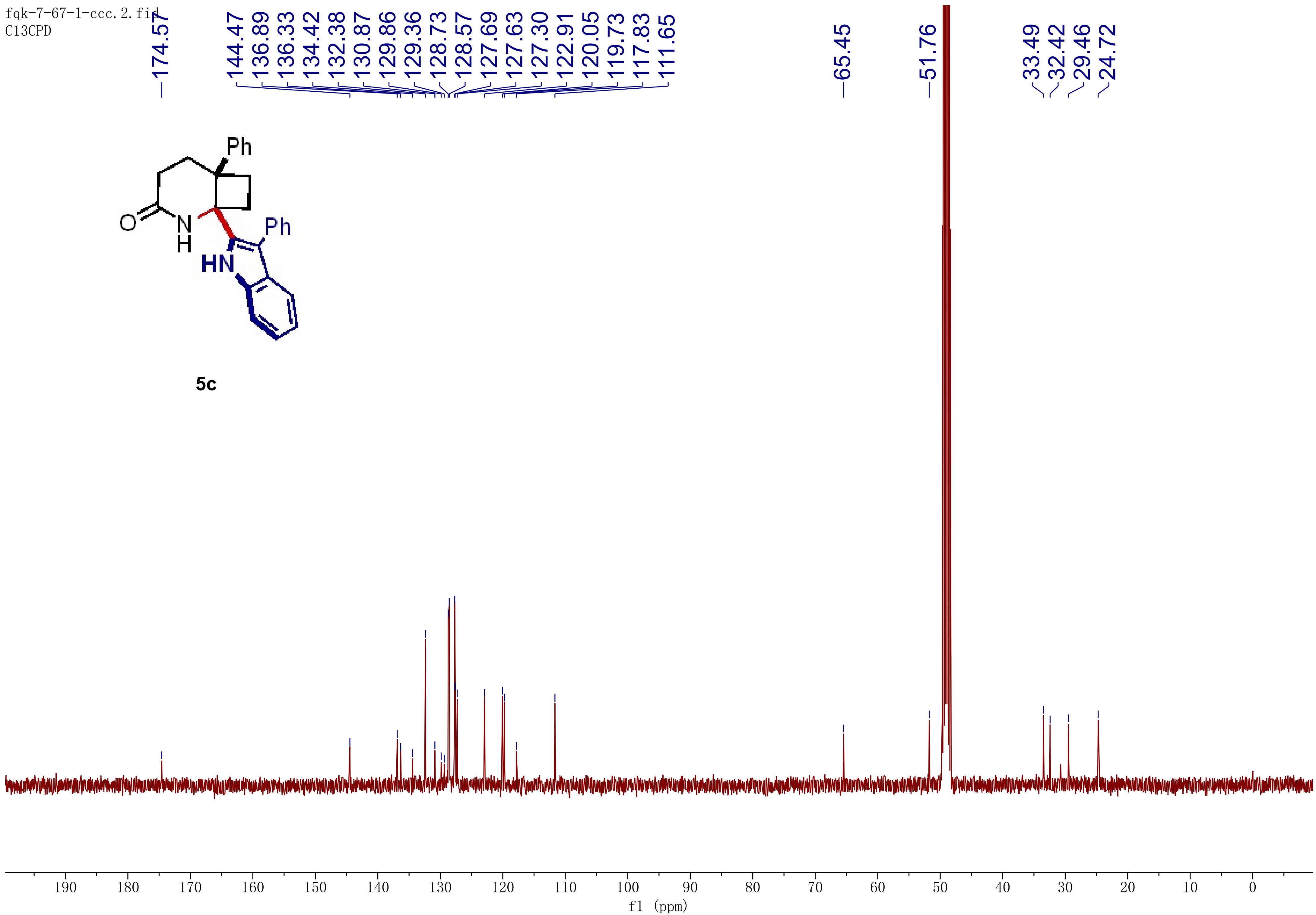

fqk-7-49-1.1.fid
PROTON
10.76
7.97
7.55
7.53
7.17
7.15
7.04
7.04
6.95
6.94
6.93
6.93
6.92
6.92
6.91
6.90
6.89
6.88
6.87
6.86
6.86
6.84
6.83
6.82
6.81
6.81
6.80
6.79
2.64
2.64
2.51
2.51
2.49
2.39
2.38
2.37
2.37
2.36
2.35
2.34
2.32
2.29
2.27
2.26
2.24
2.23
2.23
2.21
2.19
2.01
2.01
Ph
O
N
H
N
H
5a
1.05
1.01
1.13
1.14
1.08
3.20
1.25
3.10
1.15
1.17
3.06
2.28
1.09
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


fqk-7-49-1.2.fid
C13CPD
171.14
145.56
136.44
127.10
126.31
126.29
125.36
123.39
120.58
120.39
118.50
117.22
111.50
61.85
49.73
34.85
34.55
28.56
25.75
Ph
O
N
H
N
H
5a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

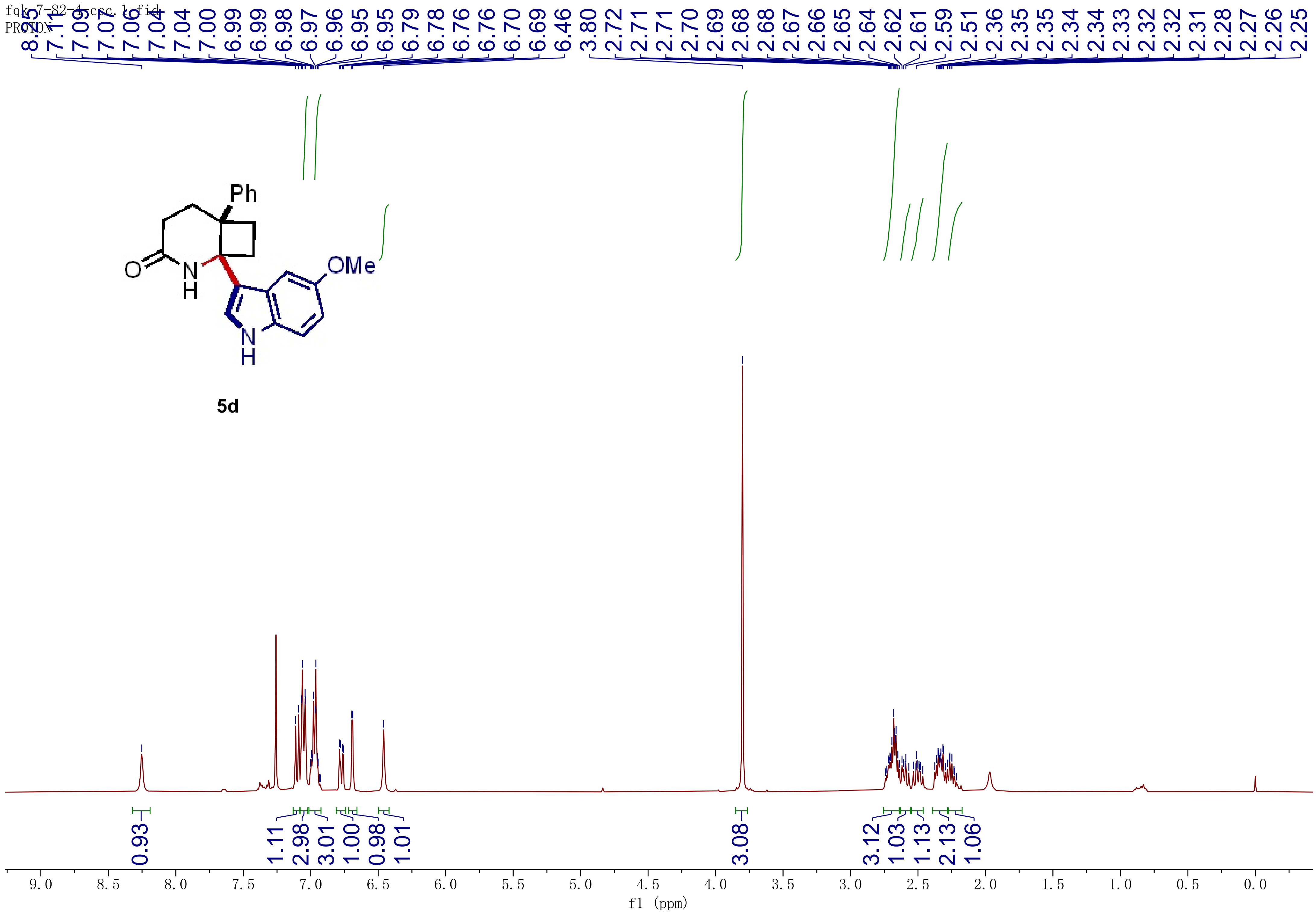

7-71-4-dmso.1.fid
PROTON
10.70
8.07
7.46
7.44
7.27
7.25
7.23
7.21
7.09
7.07
6.95
6.93
6.91
6.89
6.87
6.85
6.82
6.81
2.65
2.63
2.60
2.48
2.44
2.38
2.27
2.24
2.22
2.10
1.89
1.87
1.84
1.67
1.65
1.64
1.62
Ph
O
N
H
Ph
N
H
5b
1.08
1.00
1.15
3.95
1.36
1.38
2.27
4.97
1.13
2.19
1.35
1.15
1.12
2.16
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


7-71-4-dmso 2.fid
C13CPD
169.82
143.42
136.68
135.47
134.30
128.12
127.74
127.40
127.30
126.98
126.57
125.69
120.98
120.51
118.73
113.23
110.89
63.76
49.90
36.12
33.34
29.99
28.75
Ph
O
N
H
Ph
N
H
5b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

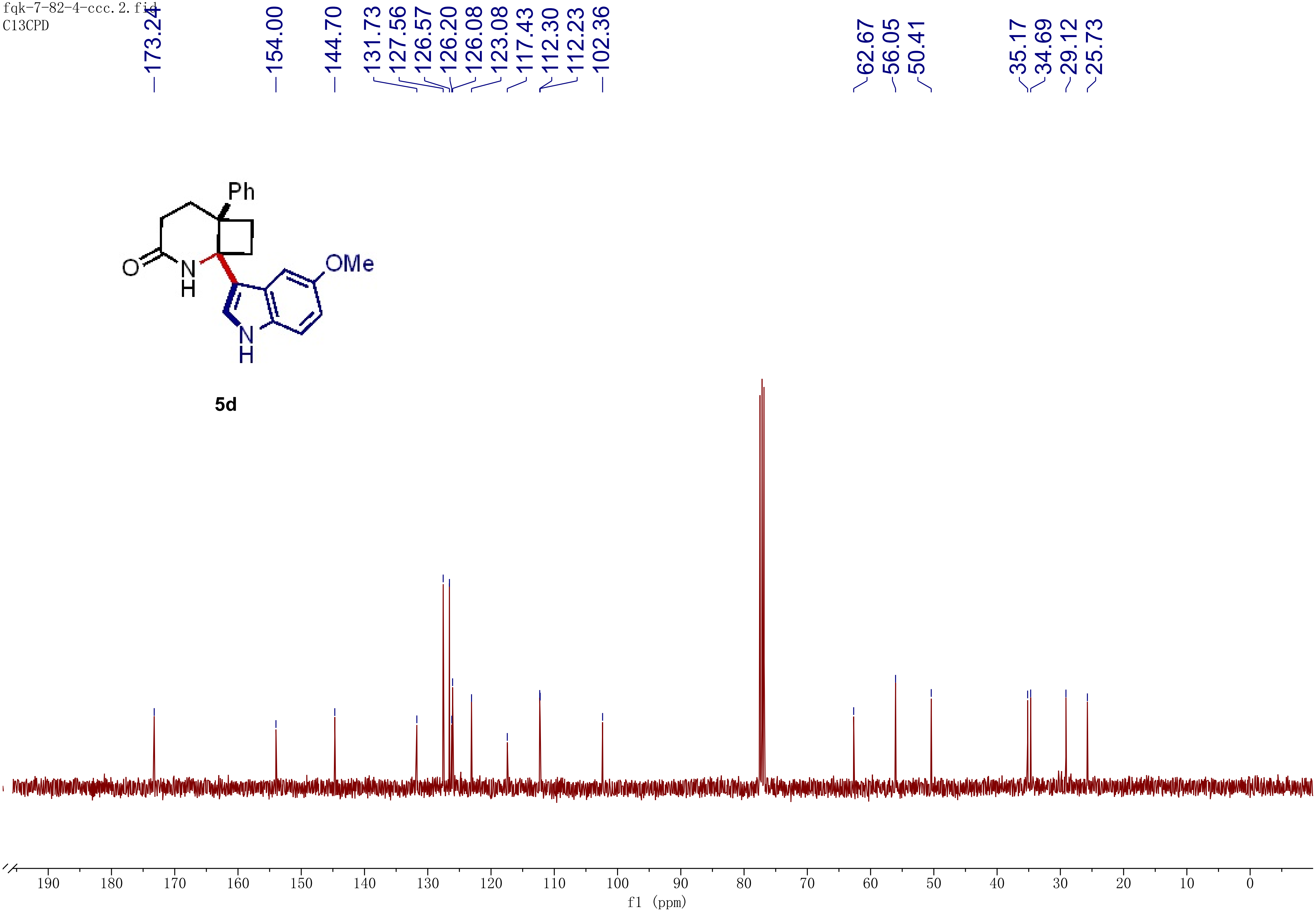

fgk-7-67-1.1.fid
PROTON
7.33
7.29
7.29
7.28
7.09
7.07
7.06
6.95
6.93
6.89
6.87
6.85
2.69
2.69
2.68
2.67
2.66
2.65
2.64
2.64
2.63
2.61
2.58
2.57
2.55
2.52
2.42
2.41
2.39
2.39
2.37
2.37
2.36
2.35
2.34
2.32
2.31
2.30
2.29
2.07
2.06
2.05
2.04
2.03
2.02
1.93
1.92
1.91
1.90
1.88
Ph
O
N
H
Ph
HN
5c
1.23
3.13
6.17
3.99
2.18
1.17
3.27
1.20
1.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



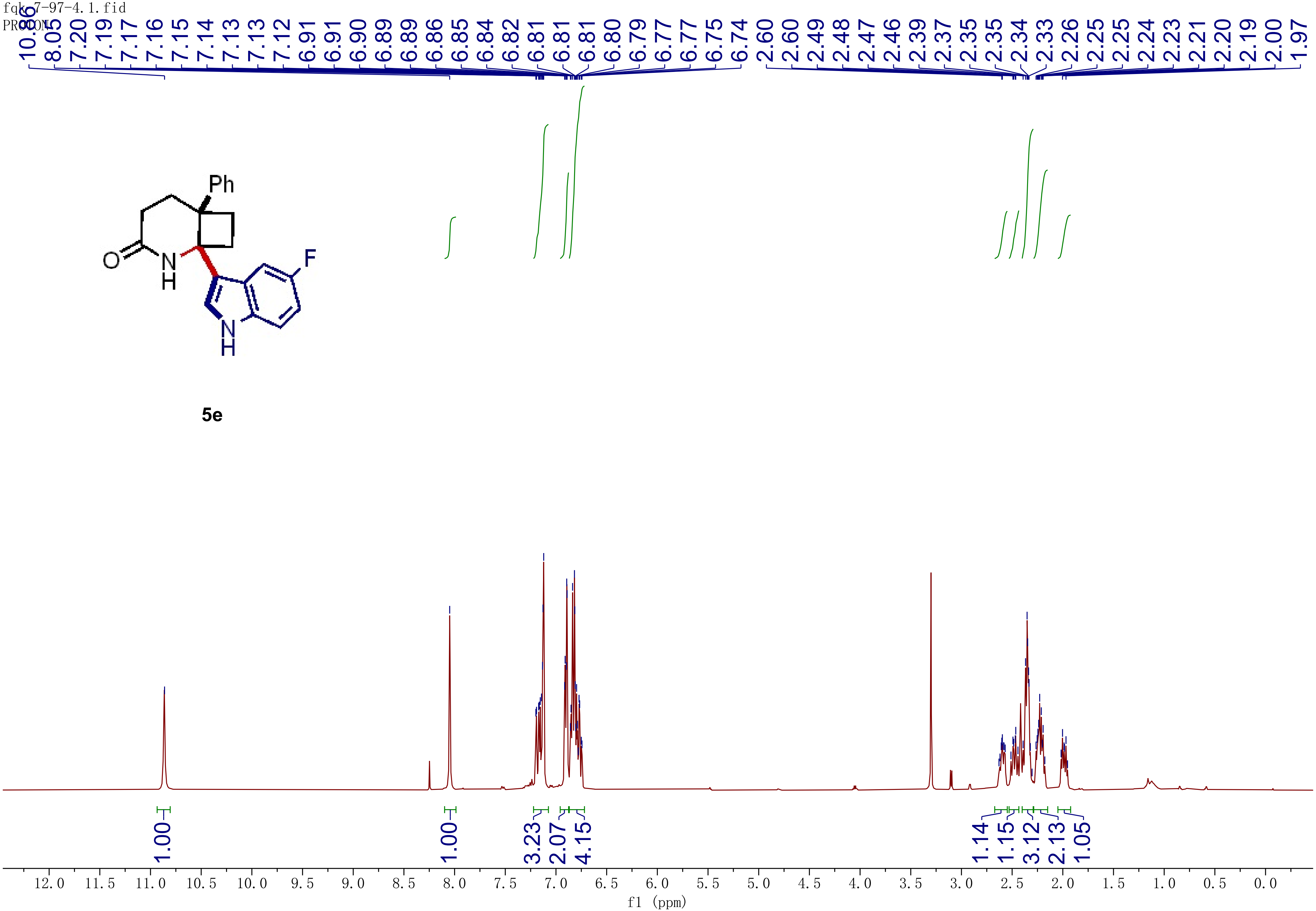
fqk-7-67-1-ccc.2.fid
C13CPD
174.57
144.47
136.89
136.33
134.42
132.38
130.87
129.86
129.36
128.73
128.57
127.69
127.63
127.30
122.91
120.05
119.73
117.83
111.65
65.45
51.76
33.49
32.42
29.46
24.72
Ph
O
N
H
Ph
HN
5c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

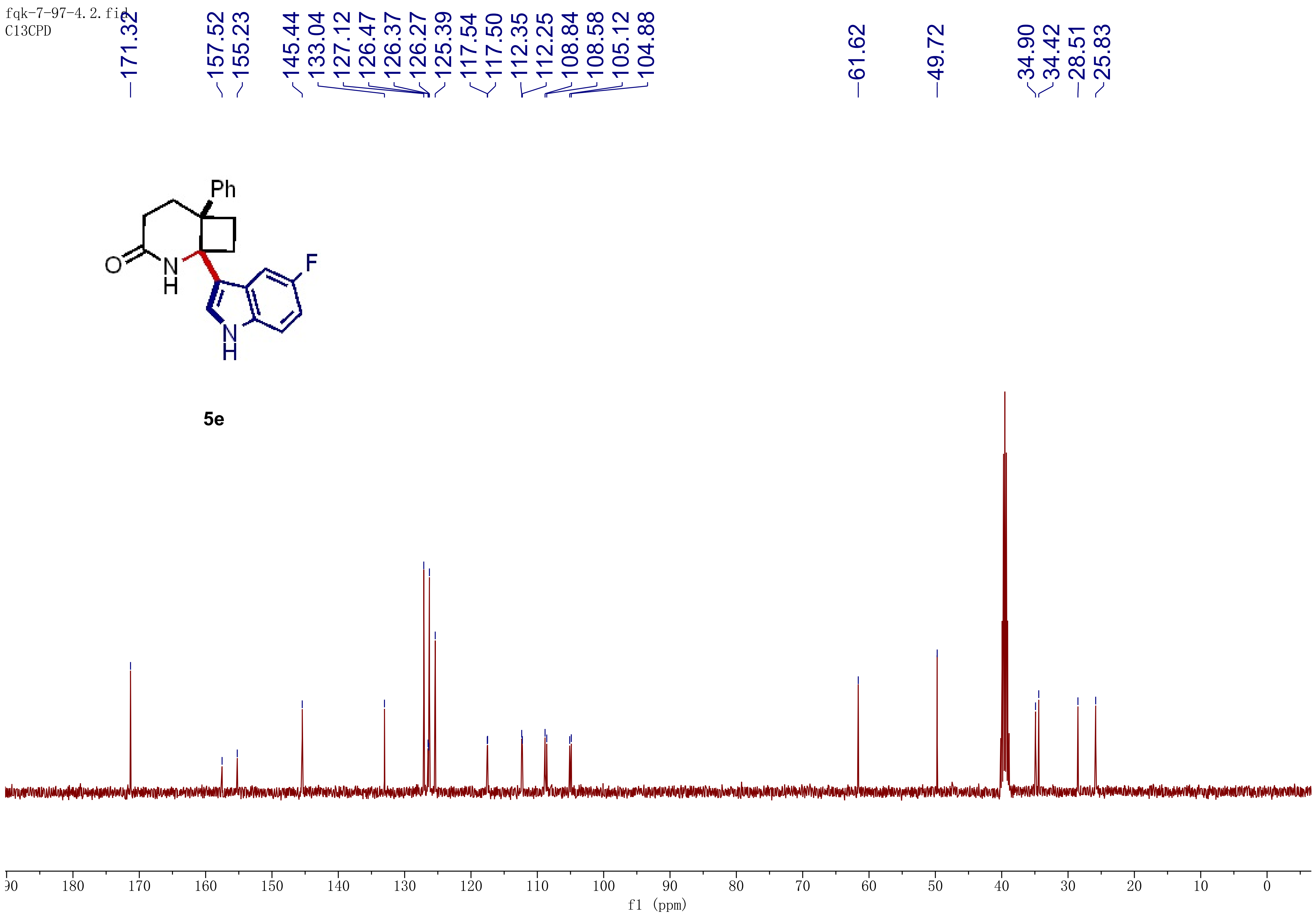

fqk-7-82-4-csc.1.fid
PROTON
8.25 7.11 7.09 7.07 7.06 7.04 7.04 7.00 6.99 6.99 6.98 6.97 6.96 6.95 6.95 6.79 6.78 6.76 6.76 6.70 6.69 6.46 3.80 2.72 2.71 2.71 2.70 2.69 2.68 2.68 2.67 2.66 2.65 2.64 2.62 2.61 2.59 2.51 2.36 2.35 2.35 2.34 2.34 2.33 2.32 2.32 2.31 2.28 2.27 2.26 2.25
Ph
O
N
H
OMe
N
H
0.93
1.11
2.98
3.01
1.00
0.98
1.01
3.08
3.12
1.03
1.13
2.13
1.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

5d

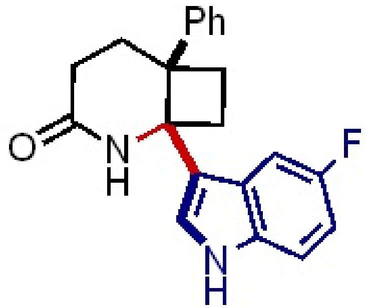

fqk-7-82-4-ccc.2.fid
C13CPD
173.24
154.00
144.70
131.73
127.56
126.57
126.20
126.08
123.08
117.43
112.30
112.23
102.36
62.67
56.05
50.41
35.17
34.69
29.12
25.73
Ph
O
N
H
OMe
N
H
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

5d

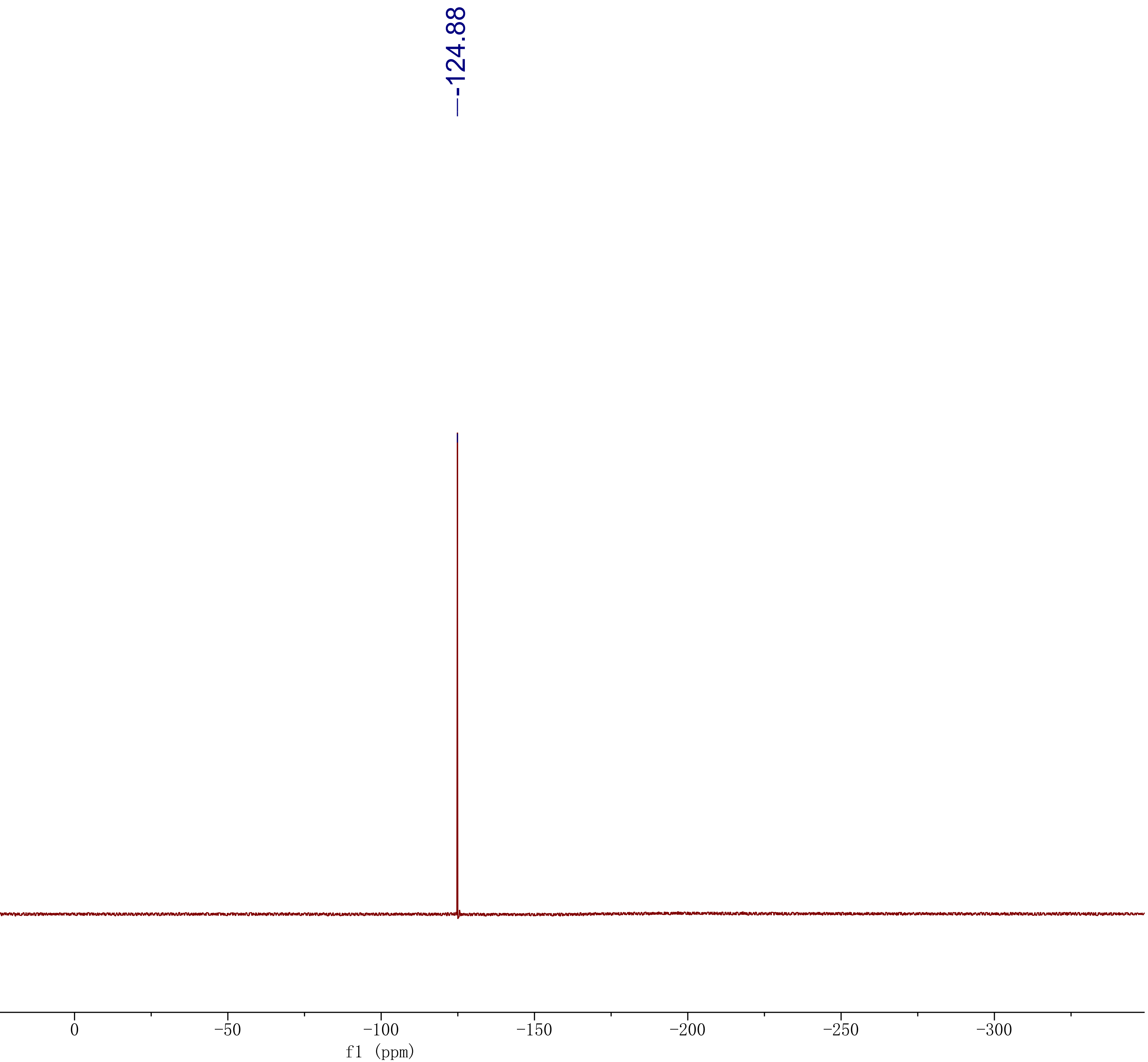

fqk-7-97-4.1.fid
PROTON
10.86 8.05 7.20 7.19 7.17 7.16 7.15 7.14 7.13 7.13 7.12 6.91 6.91 6.90 6.89 6.89 6.86 6.85 6.84 6.82 6.81 6.81 6.81 6.80 6.79 6.77 6.77 6.75 6.74 2.60 2.60 2.49 2.48 2.47 2.46 2.39 2.37 2.35 2.35 2.34 2.33 2.26 2.25 2.25 2.24 2.23 2.21 2.20 2.19 2.00 1.97
Ph
O
N
H
F
N
H
5e
1.00
1.00
3.23
2.07
4.15
1.14
1.15
3.12
2.13
1.05
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

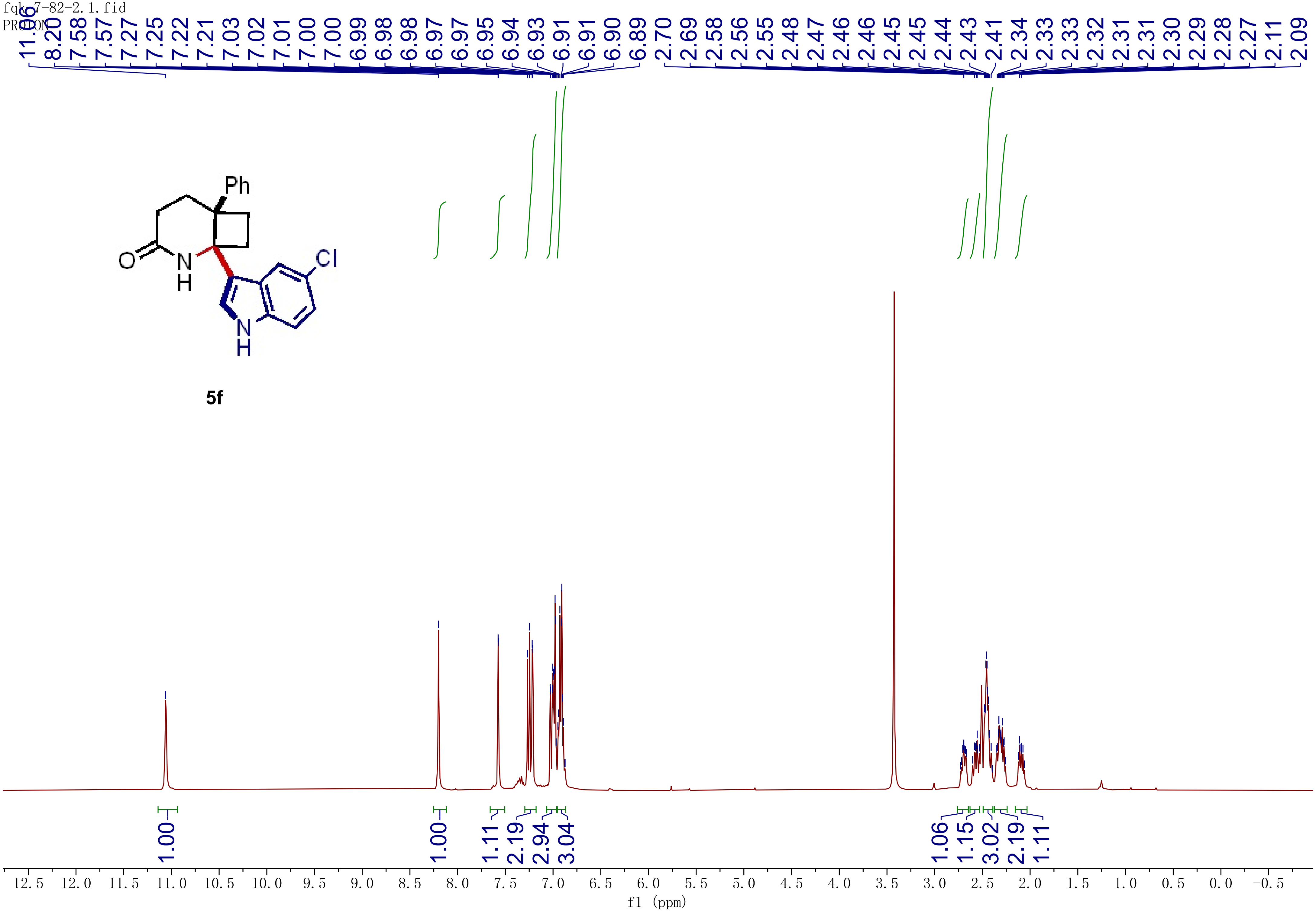

fqk-7-97-4.2.fid
C13CPD
171.32
157.52
155.23
145.44
133.04
127.12
126.47
126.37
126.27
125.39
117.54
117.50
112.35
112.25
108.84
108.58
105.12
104.88
61.62
49.72
34.90
34.42
28.51
25.83
Ph
O
N
H
F
N
H
5e
f1 (ppm)

fqk-7-97-4.1.fid
F19

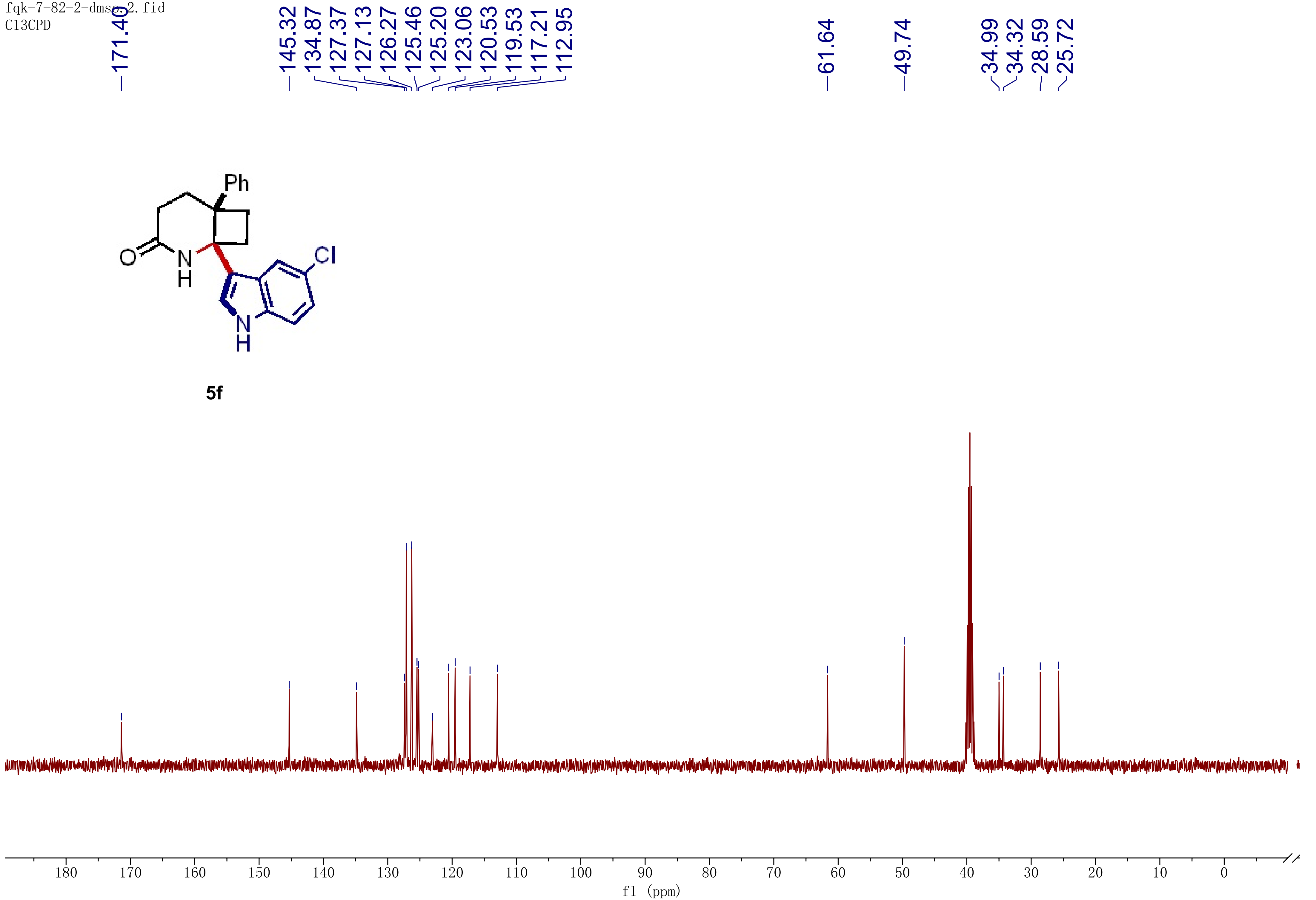


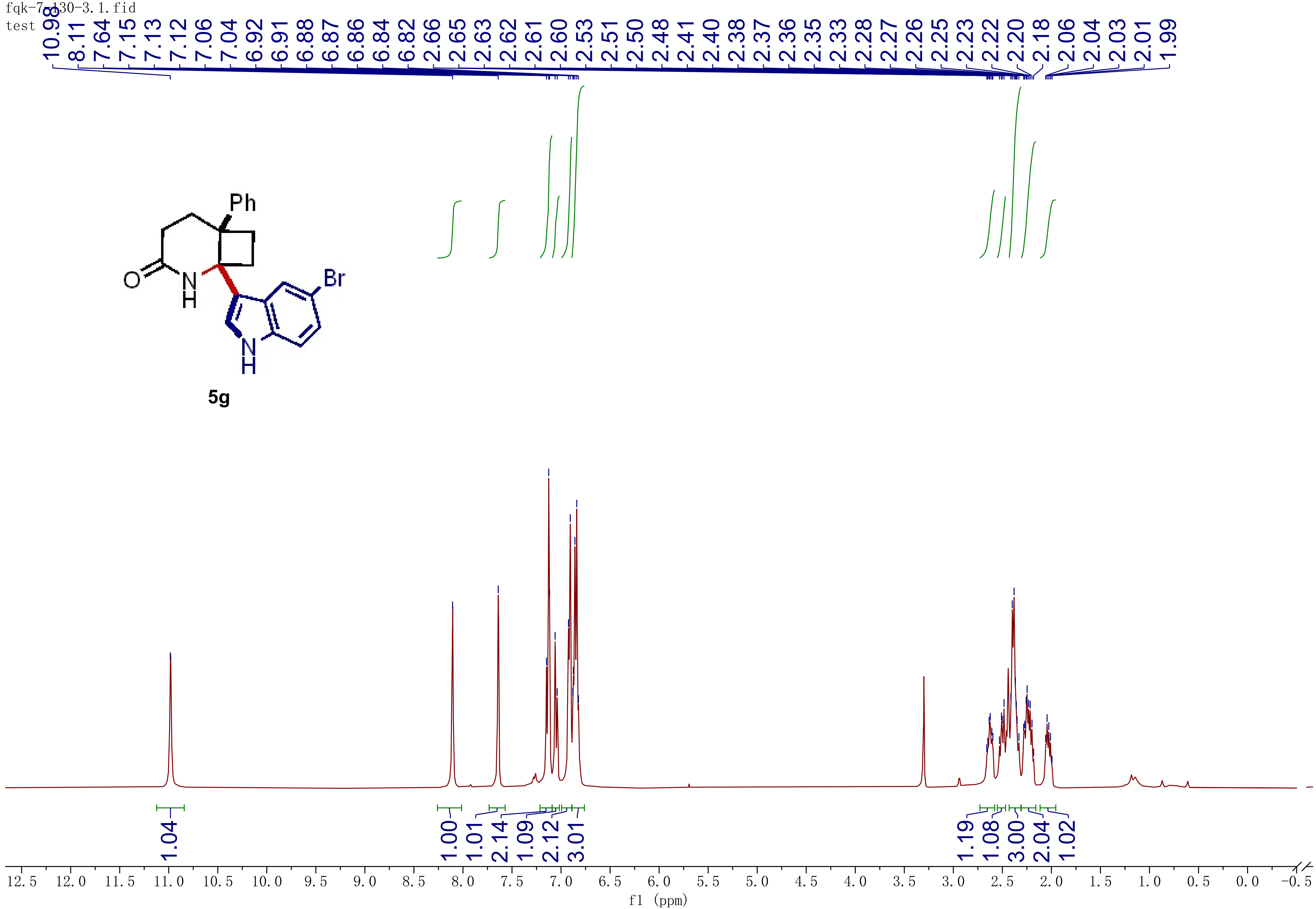
5e

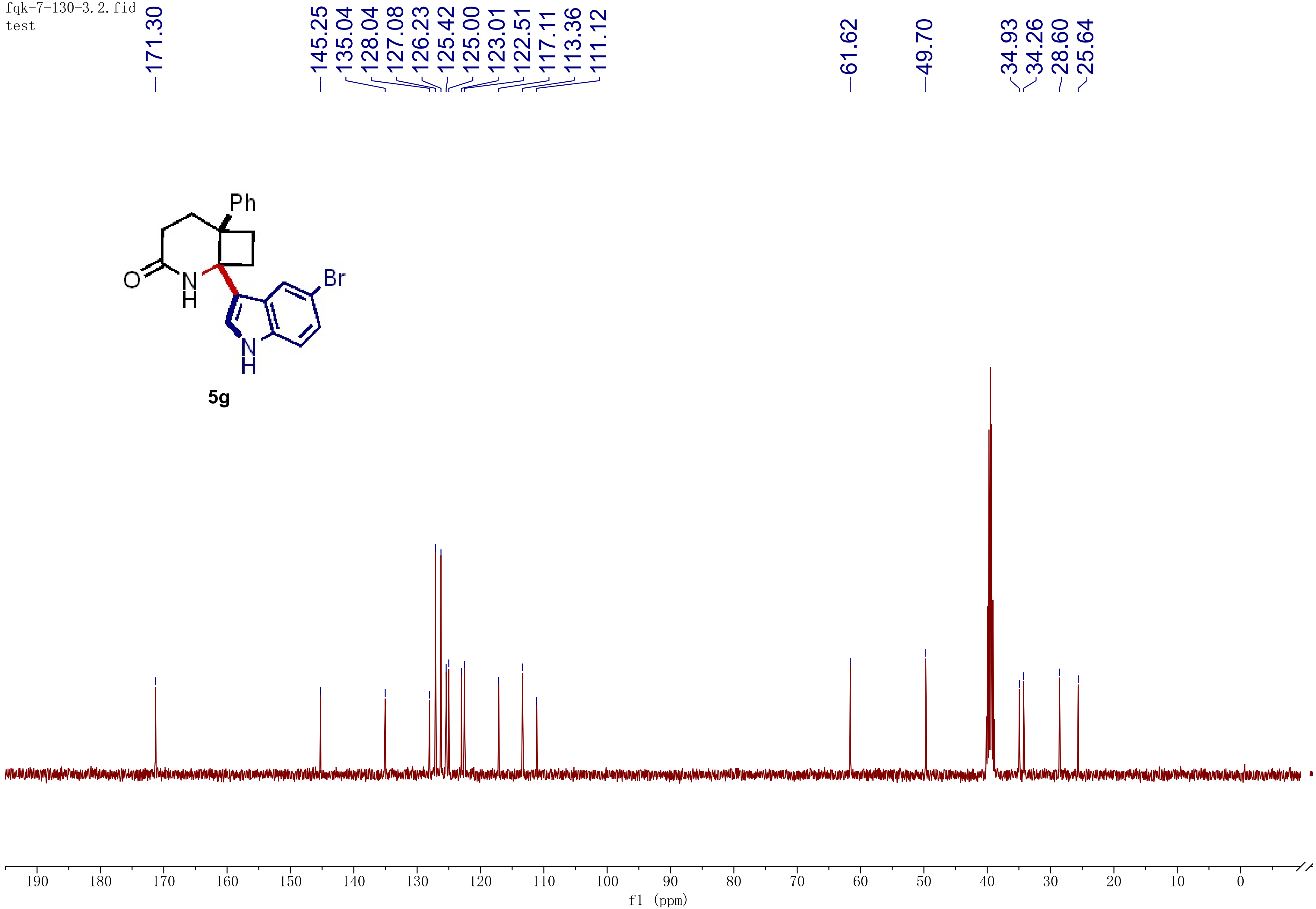


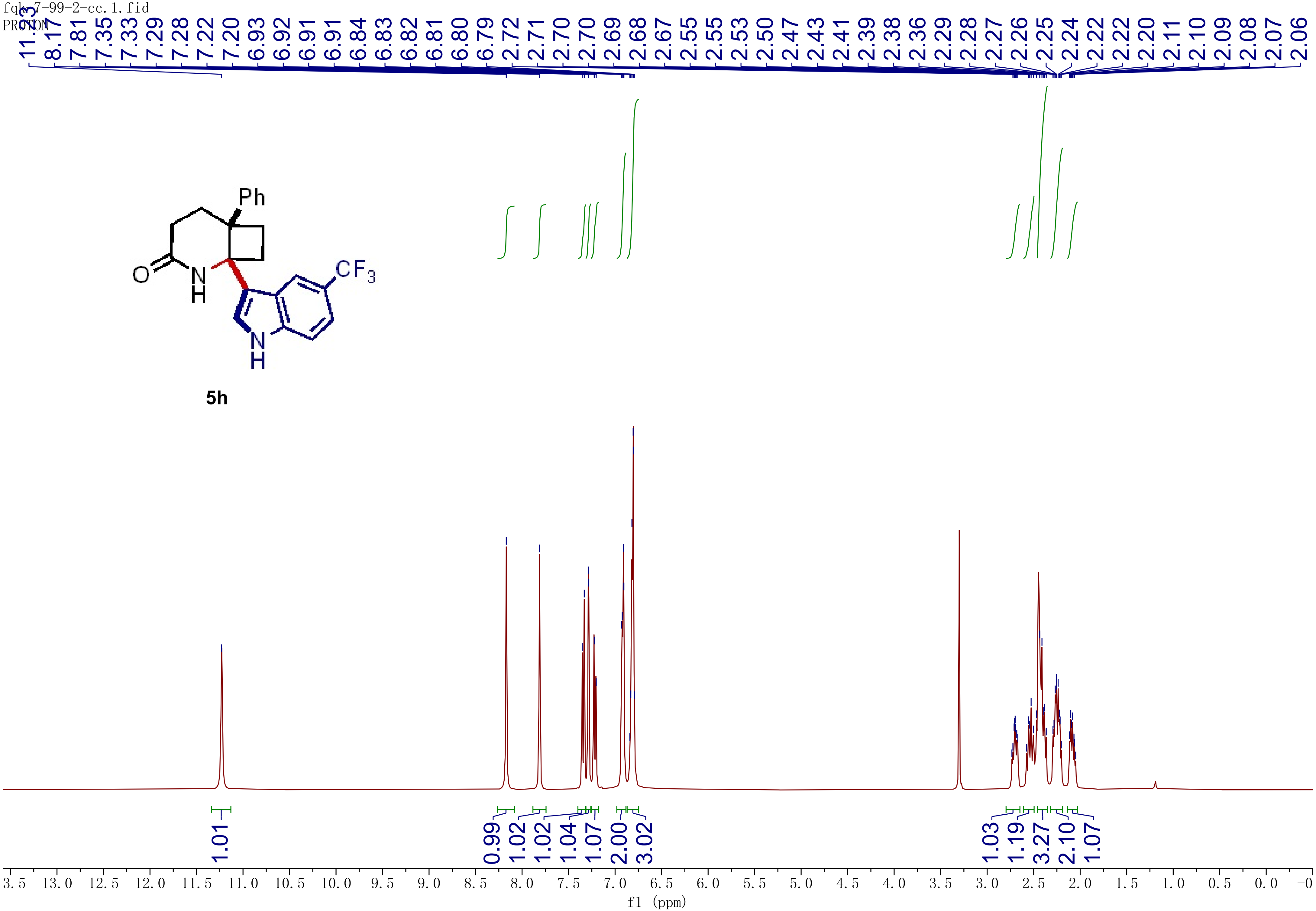

fqk-7-82-2.1.fid
PROTON
11.06
8.20
7.58
7.57
7.27
7.25
7.22
7.21
7.03
7.02
7.01
7.00
7.00
6.99
6.98
6.98
6.97
6.97
6.95
6.94
6.93
6.91
6.91
6.90
6.89
2.70
2.69
2.58
2.56
2.55
2.48
2.47
2.46
2.46
2.45
2.45
2.44
2.43
2.41
2.34
2.33
2.33
2.32
2.31
2.31
2.30
2.29
2.28
2.27
2.11
2.09
Ph
O
N
H
Cl
N
H
5f
1.00
1.00
1.11
2.19
2.94
3.04
1.06
1.15
3.02
2.19
1.11
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

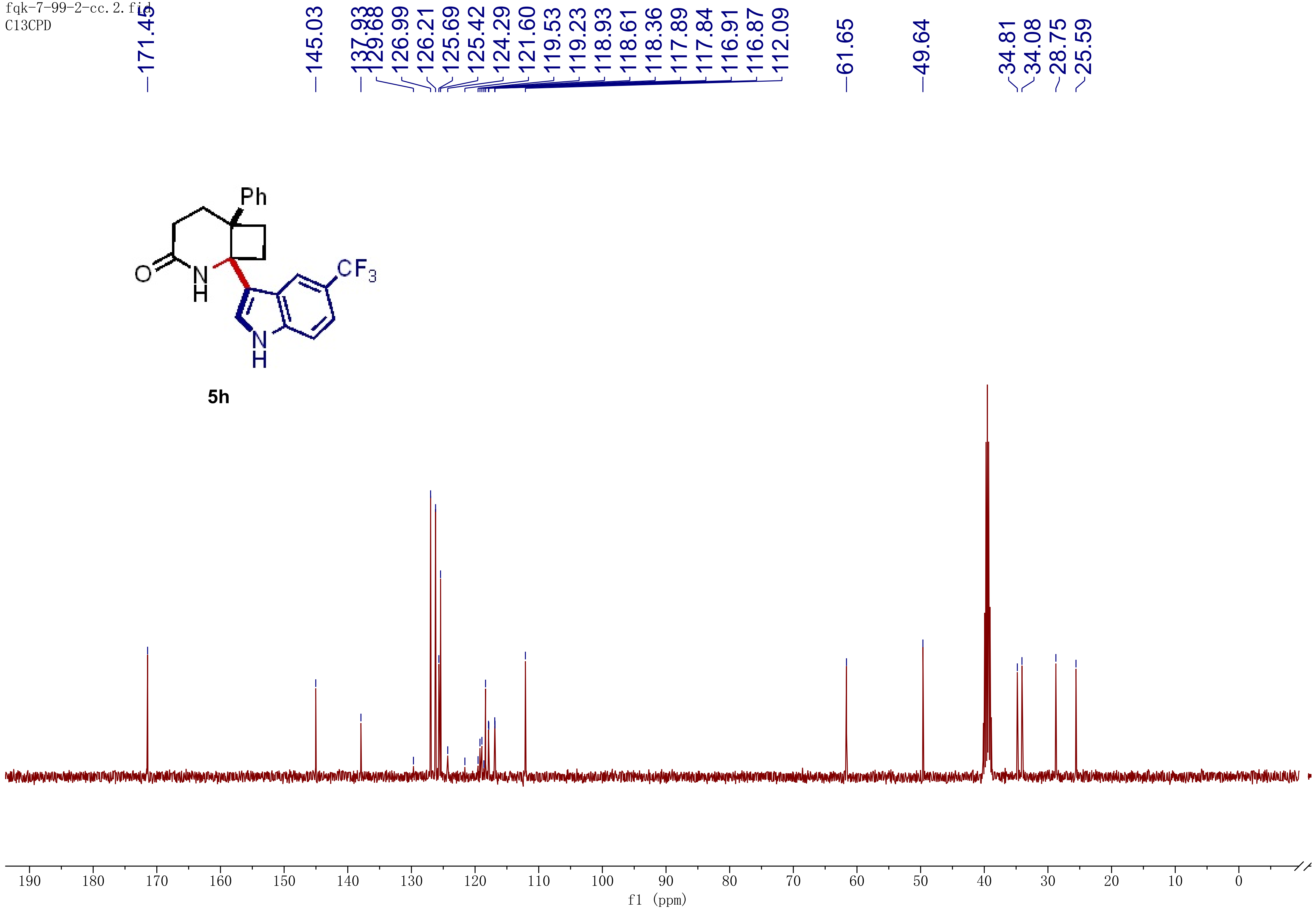
fqk-7-82-2-dmso.2.fid
C13CPD

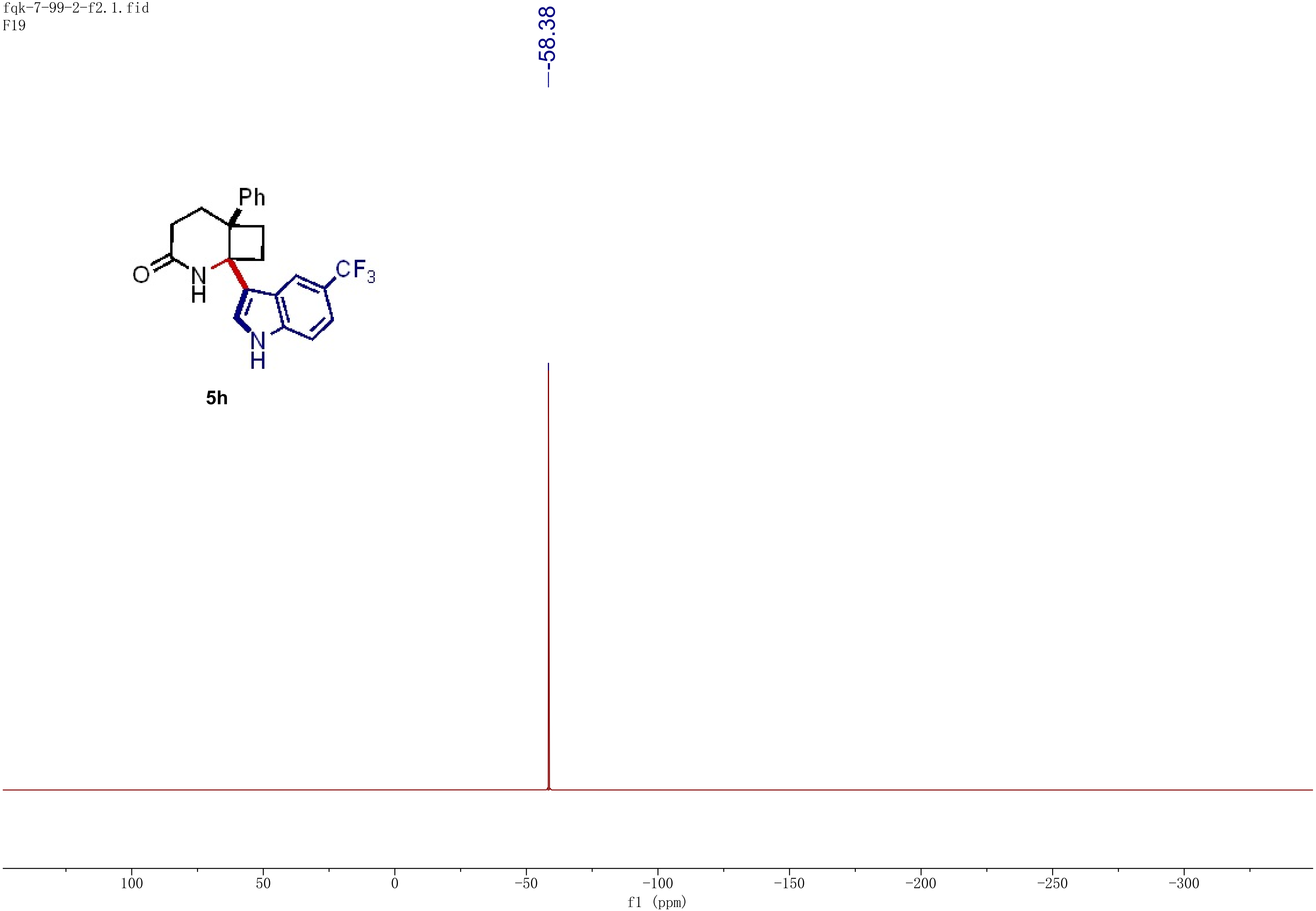



fqk-7-130-3.1.fid
test
10.98 8.11 7.64 7.15 7.13 7.12 7.06 7.04 6.92 6.91 6.88 6.87 6.86 6.84 6.82 2.66 2.65 2.63 2.62 2.61 2.60 2.53 2.51 2.50 2.48 2.41 2.40 2.38 2.37 2.36 2.35 2.33 2.28 2.27 2.26 2.25 2.23 2.22 2.20 2.18 2.06 2.04 2.03 2.01 1.99
Ph
O
N
H
Br
N
H
5g
1.04
1.00
1.01
2.14
1.09
2.12
3.01
1.19
1.08
3.00
2.04
1.02
f1 (ppm)

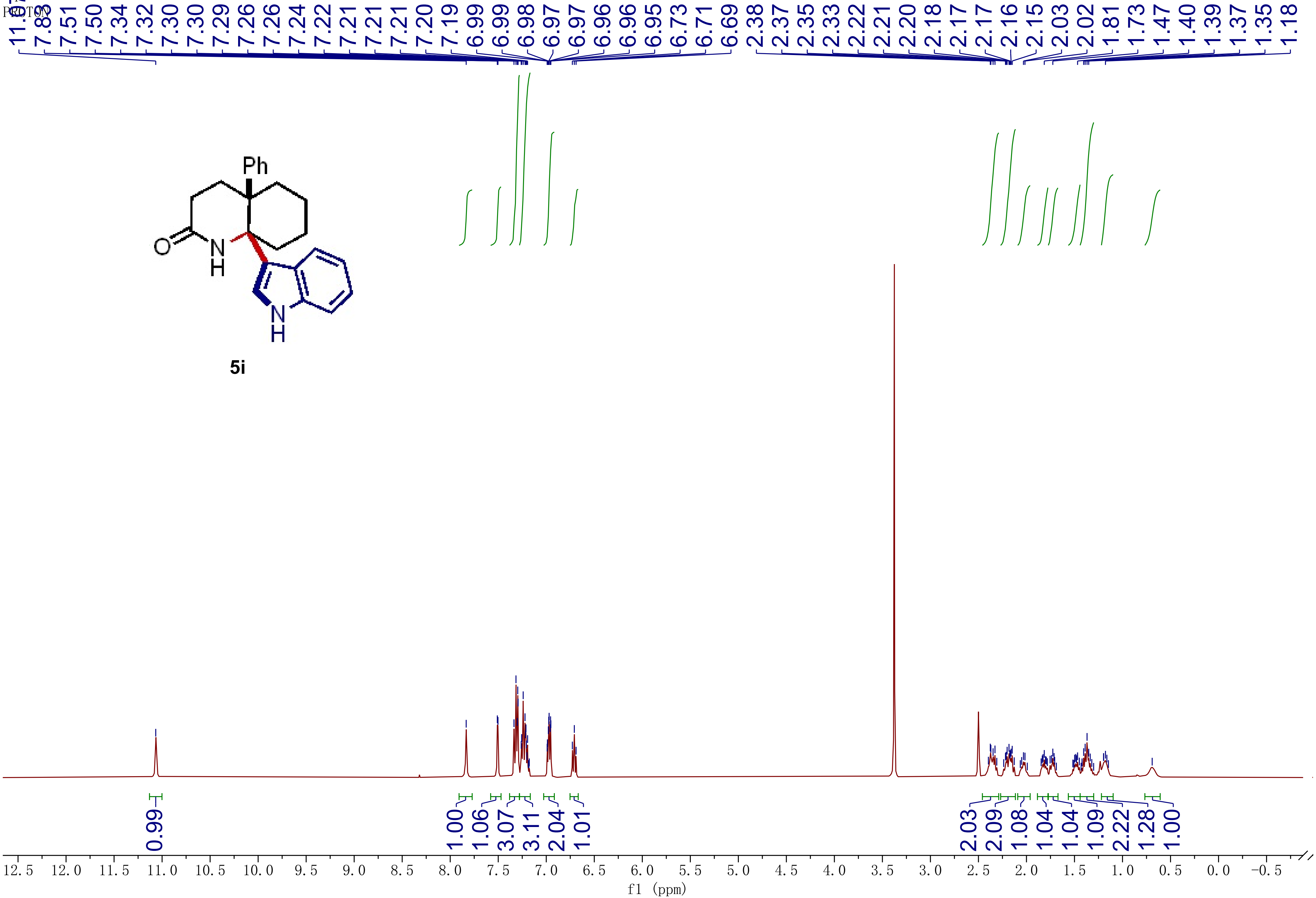

fqk-7-130-3.2.fid
test
171.30
145.25
135.04
128.04
127.08
126.23
125.42
125.00
123.01
122.51
117.11
113.36
111.12
61.62
49.70
34.93
34.26
28.60
25.64
Ph
Br
O
N
H
N
H
5g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


fqk-7-99-2-cc.1.fid
PROTON
8.17 7.81 7.35 7.33 7.29 7.28 7.22 7.20 6.93 6.92 6.91 6.91 6.84 6.83 6.82 6.81 6.80 6.79 2.72 2.71 2.70 2.70 2.69 2.68 2.67 2.55 2.55 2.53 2.50 2.47 2.43 2.41 2.39 2.38 2.36 2.29 2.28 2.27 2.26 2.25 2.24 2.22 2.22 2.20 2.11 2.10 2.09 2.08 2.07 2.06
Ph
O
N
H
CF3
5h
1.01
0.99 1.02 1.02 1.04 1.07 2.00 3.02
1.03 1.19 3.27 2.10 1.07
f1 (ppm)


fqk-7-99-2-cc.2.fid
C13CPD
171.45
145.03
137.93
129.68
126.99
126.21
125.69
125.42
124.29
121.60
119.53
119.23
118.93
118.61
118.36
117.89
117.84
116.91
116.87
112.09
61.65
49.64
34.81
34.08
28.75
25.59
Ph
O
N
H
CF3
N
H
5h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

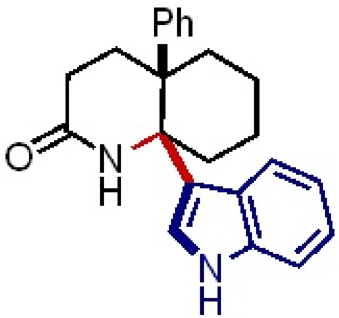

fqk-7-99-2-f2.1.fid
F19
-58.38
Ph
O
N
H
CF3
N
H
5h
100
50
0
−50
−100
−150
−200
−250
−300
f1 (ppm)

fpk-7-161-1-2-ccc.1.fid
PROTON

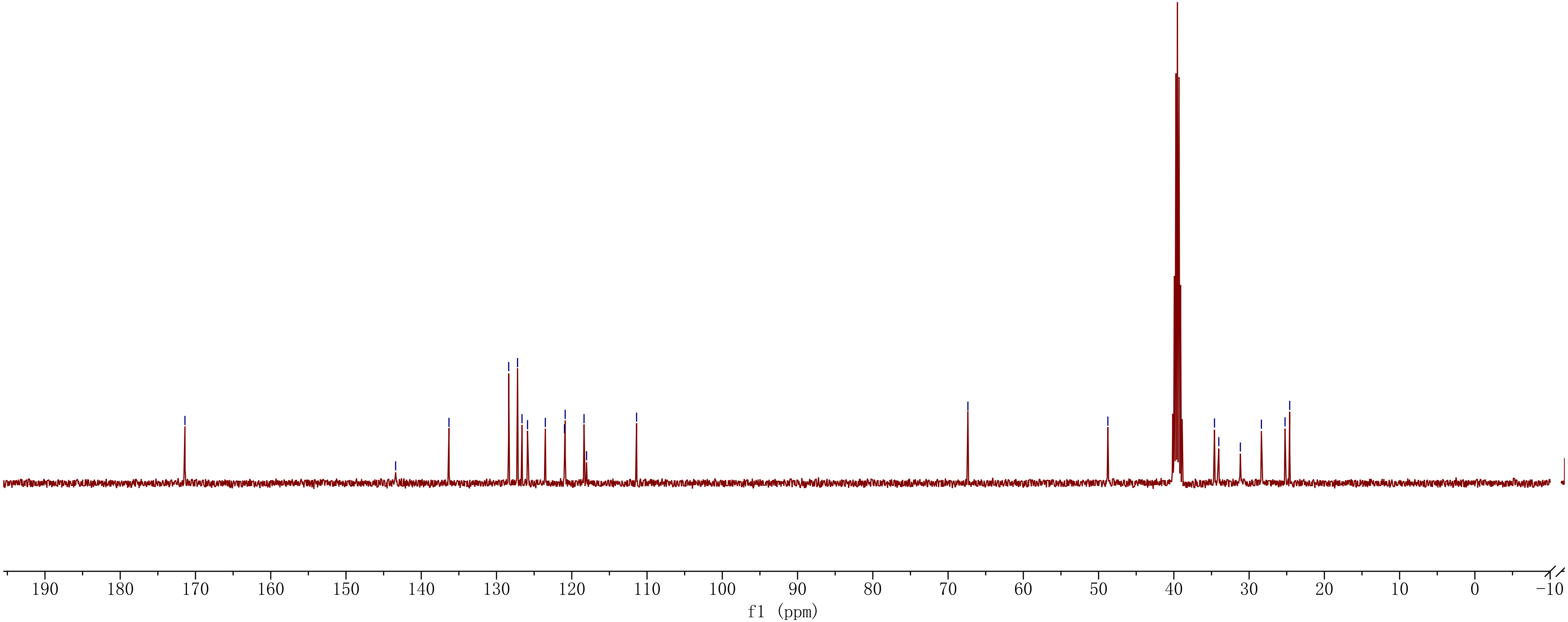


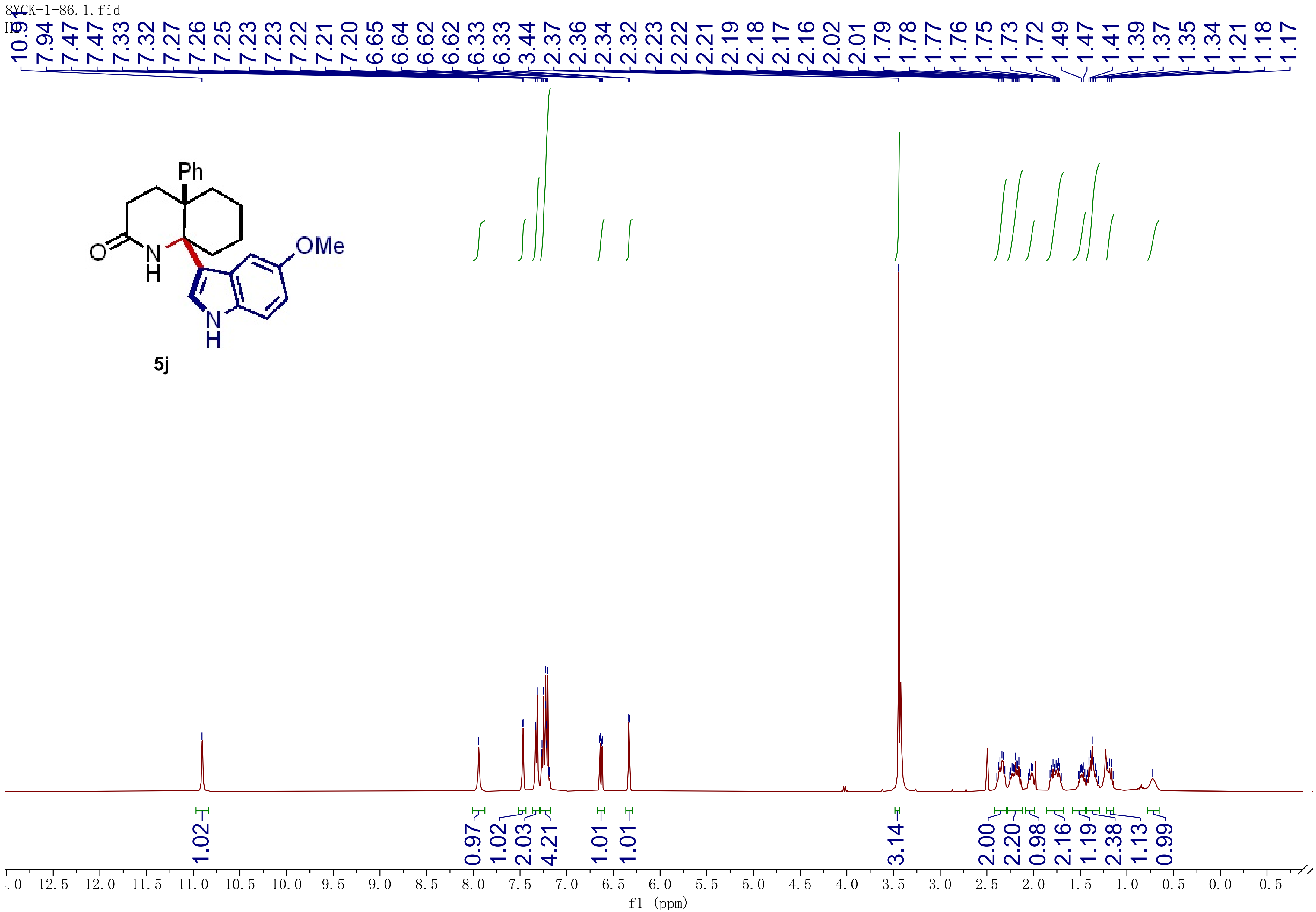
5i

fqk-7-161-1-2-ccc.2.fid
C13CPD



5i




8VCK-1-86.1.fid
H
10.91
7.94
7.47
7.47
7.33
7.32
7.27
7.26
7.25
7.23
7.23
7.22
7.21
7.20
6.65
6.64
6.62
6.62
6.33
6.33
3.44
2.37
2.36
2.34
2.32
2.23
2.22
2.21
2.19
2.18
2.17
2.16
2.02
2.01
1.79
1.78
1.77
1.76
1.75
1.73
1.72
1.49
1.47
1.41
1.39
1.37
1.35
1.34
1.21
1.18
1.17
Ph
OMe
N
H
O
N
H
5j
1.02
0.97
1.02
2.03
4.21
1.01
1.01
3.14
2.00
2.20
0.98
2.16
1.19
2.38
1.13
0.99
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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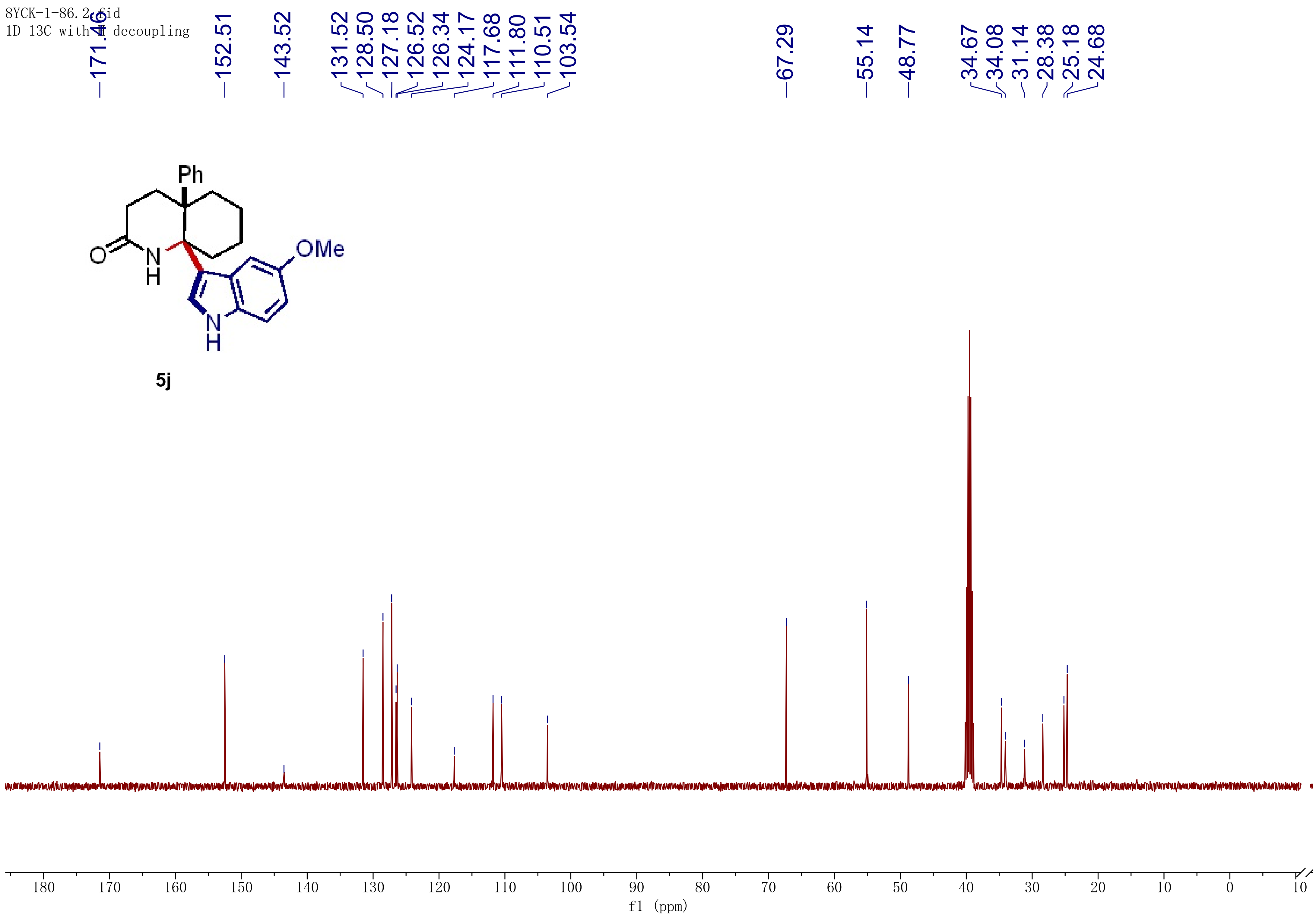
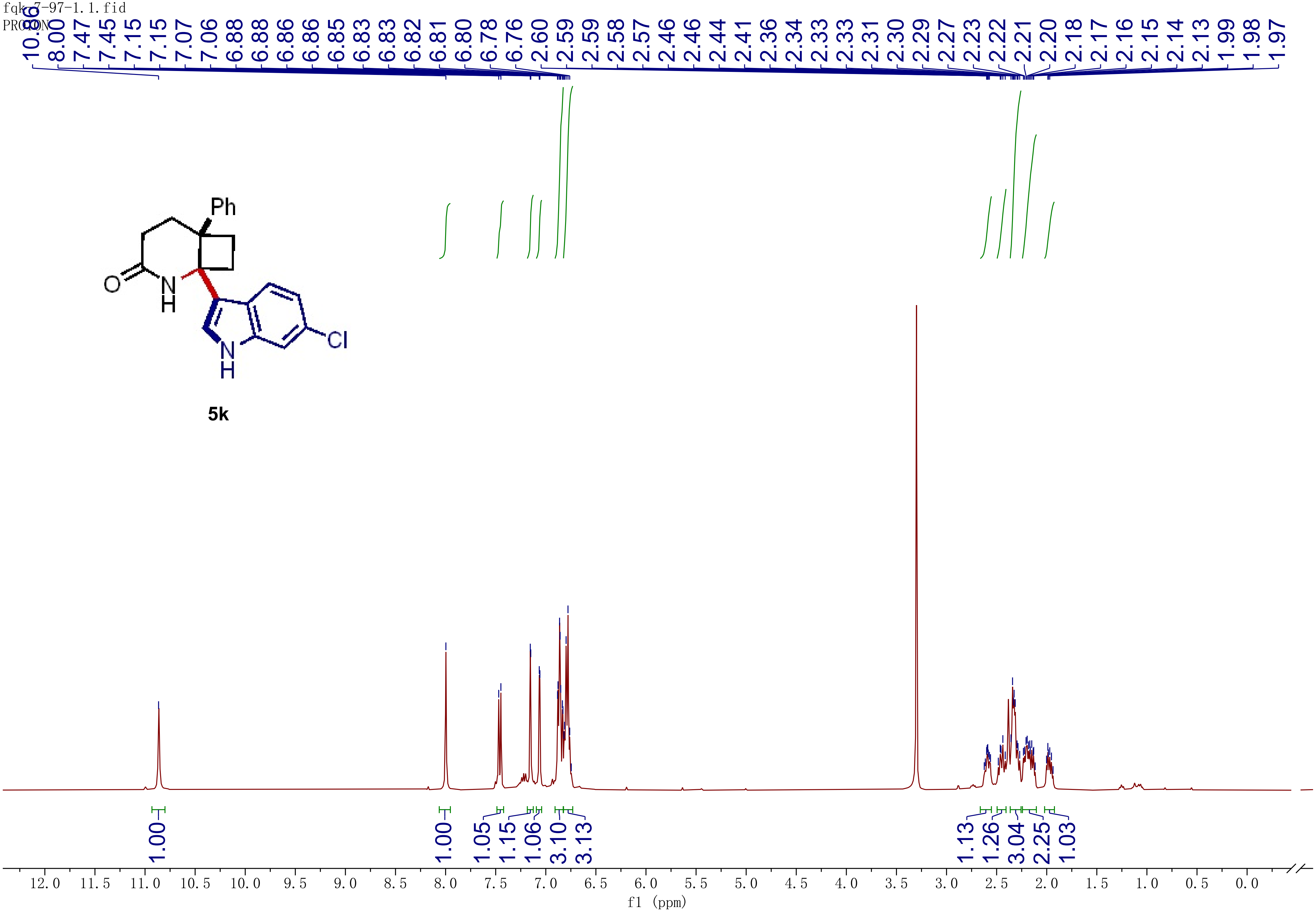
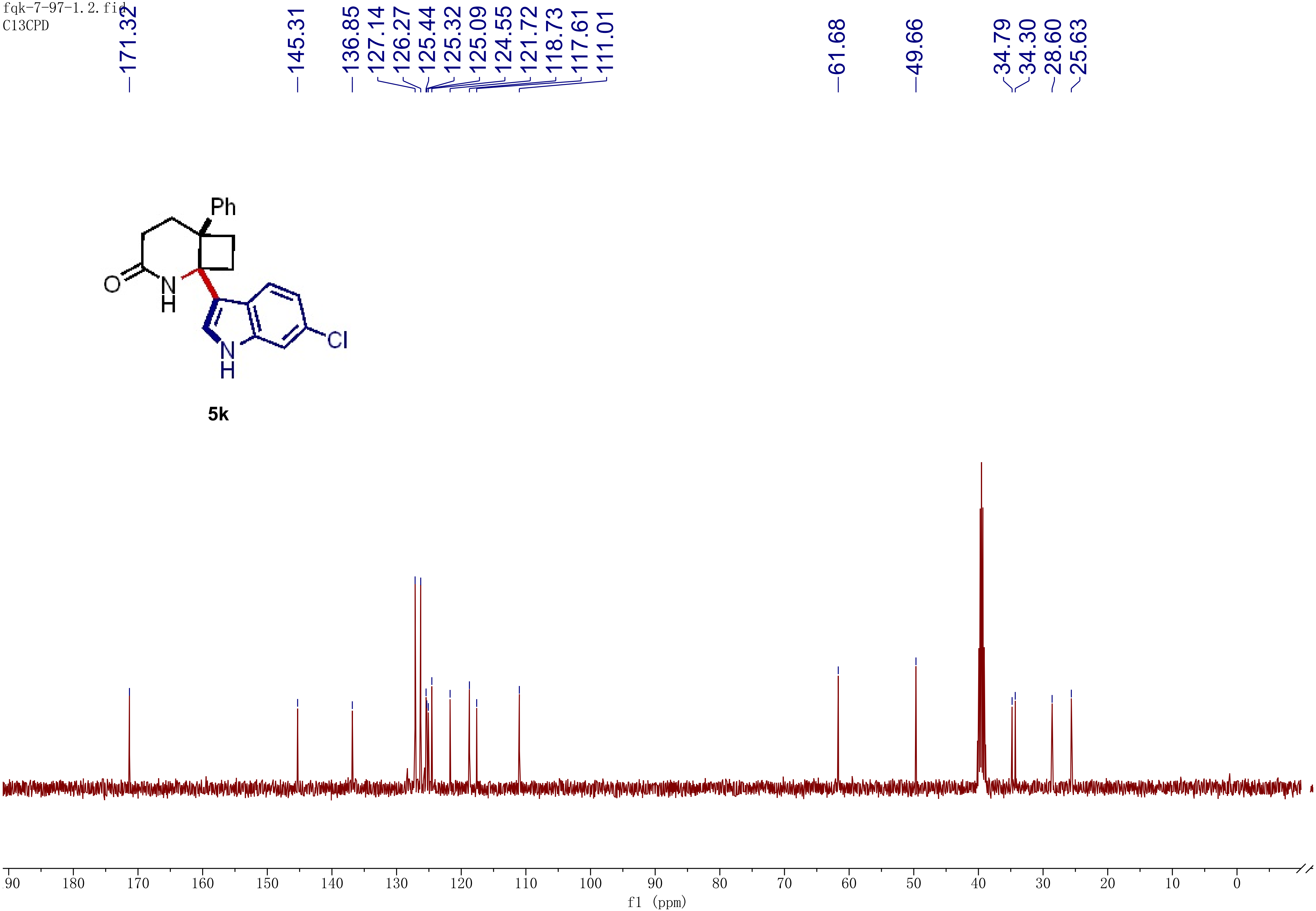
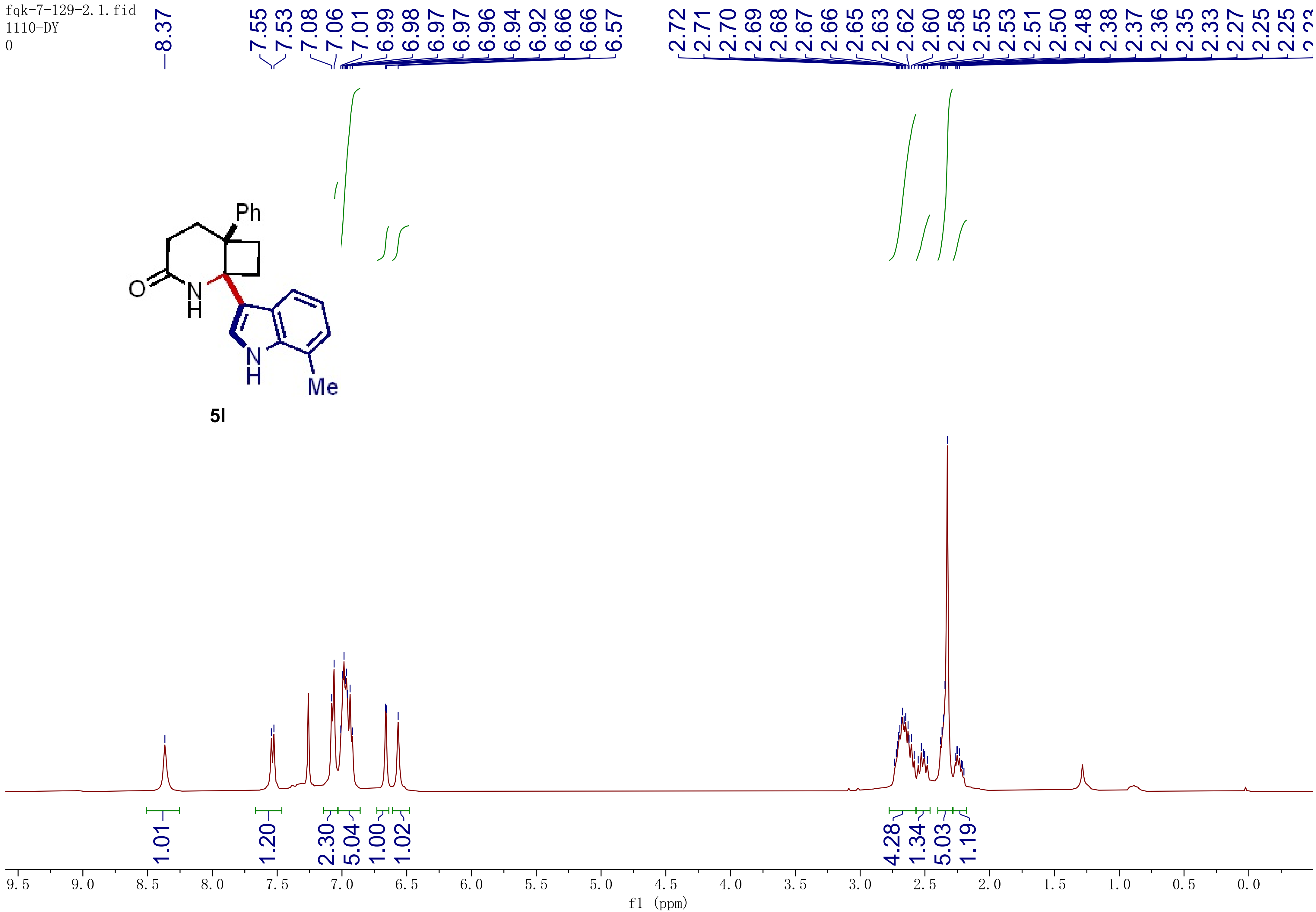
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

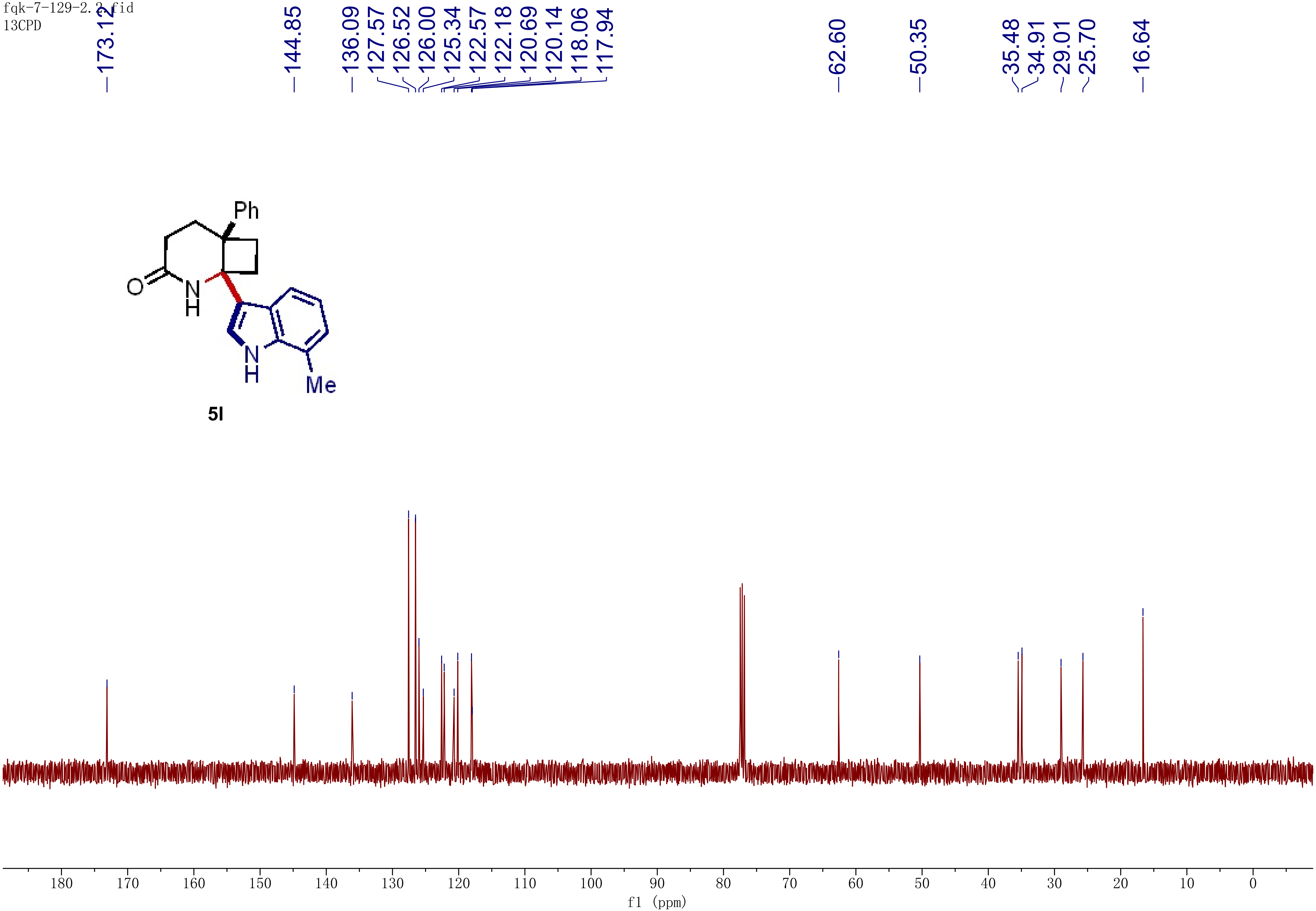

8YCK-1-86.2.fid
1D 13C with H decoupling
171.46
152.51
143.52
131.52
128.50
127.18
126.52
126.34
124.17
117.68
111.80
110.51
103.54
67.29
55.14
48.77
34.67
34.08
31.14
28.38
25.18
24.68
Ph
OMe
O
N
H
5j
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

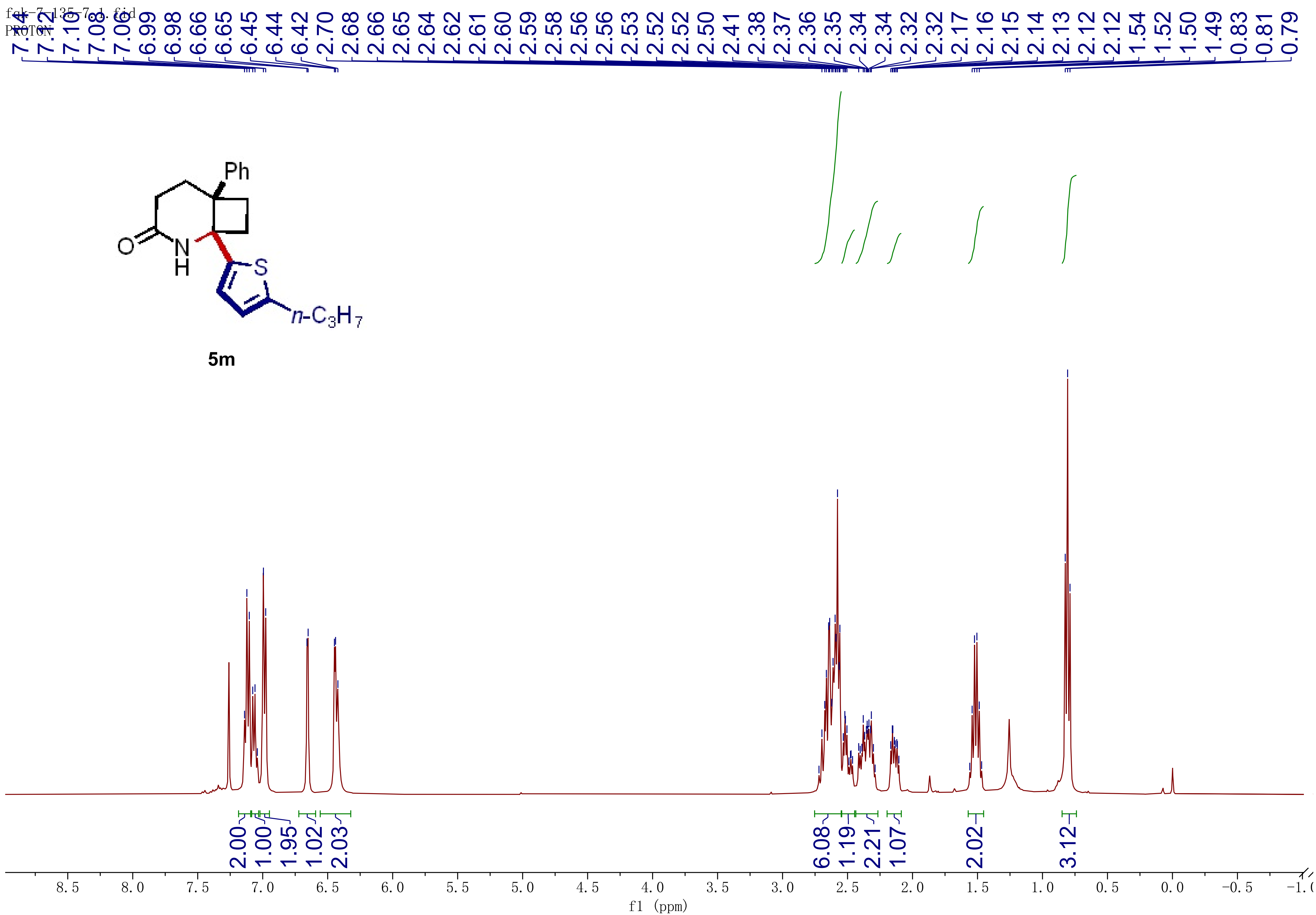

fqk-7-97-1.1.fid
PROTON
10.86 8.00 7.47 7.45 7.15 7.15 7.07 7.06 6.88 6.88 6.86 6.86 6.85 6.83 6.83 6.82 6.81 6.80 6.78 6.76 2.60 2.59 2.59 2.58 2.57 2.46 2.46 2.44 2.41 2.36 2.34 2.33 2.33 2.31 2.30 2.29 2.27 2.23 2.22 2.21 2.20 2.18 2.17 2.16 2.15 2.14 2.13 1.99 1.98 1.97
Ph
O
N
H
N
H
Cl
5k
1.00
1.00
1.05
1.15
1.06
3.10
3.13
1.13
1.26
3.04
2.25
1.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


fqk-7-97-1.2.fid
C13CPD
171.32
145.31
136.85
127.14
126.27
125.44
125.32
125.09
124.55
121.72
118.73
117.61
111.01
61.68
49.66
34.79
34.30
28.60
25.63
Ph
O
N
H
N
H
Cl
5k
f1 (ppm)

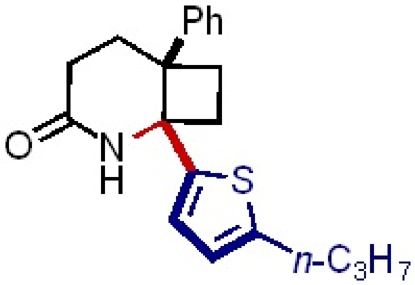


fqk-7-129-2.1.fid
1110-DY
0
8.37
7.55 7.53 7.08 7.06 7.01 6.99 6.98 6.97 6.97 6.96 6.94 6.92 6.66 6.66 6.57
2.72 2.71 2.70 2.69 2.68 2.67 2.66 2.65 2.63 2.62 2.60 2.58 2.55 2.53 2.51 2.50 2.48 2.38 2.37 2.36 2.35 2.33 2.27 2.25 2.25
Ph
O
N
H
N
H
Me
5l
1.01
1.20
2.30
5.04
1.00
1.02
4.28
1.34
5.03
1.19
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

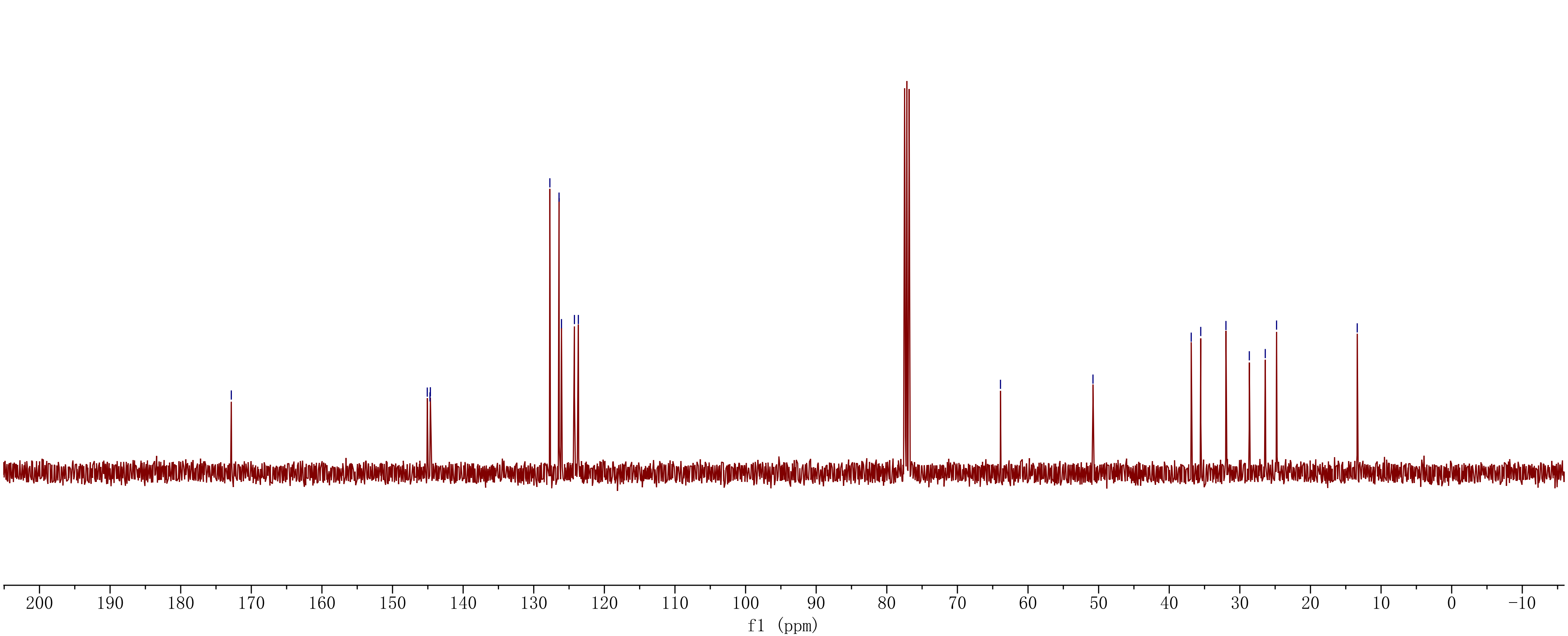

fqk-7-129-2.2.fid
13CPD
173.12
144.85
136.09
127.57
126.52
126.00
125.34
122.57
122.18
120.69
120.14
118.06
117.94
62.60
50.35
35.48
34.91
29.01
25.70
16.64
Ph
O
N
H
N
H
Me
5l
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

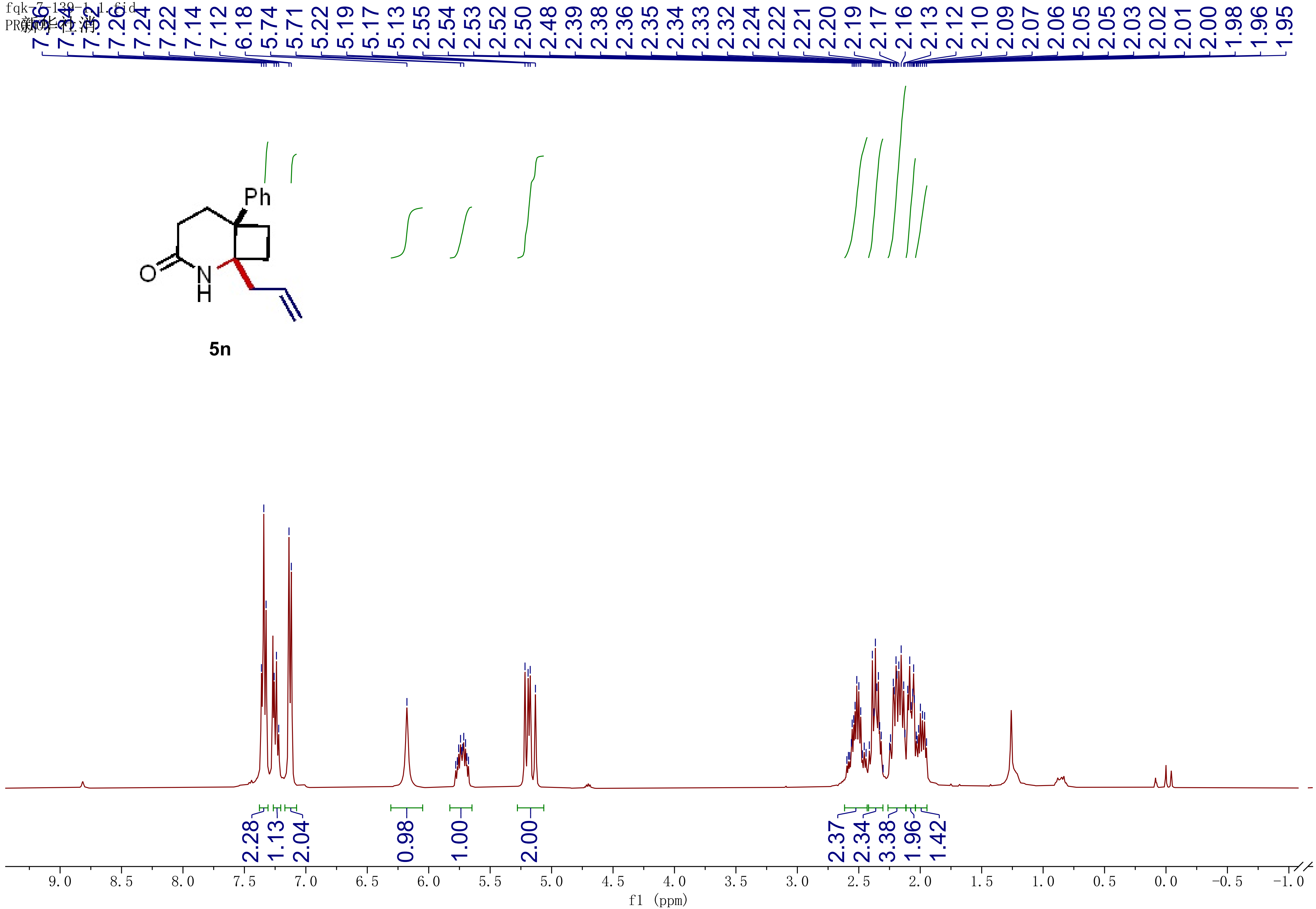

7.10
7.08
7.06
6.99
6.98
6.66
6.65
6.45
6.44
6.42
2.70
2.68
2.66
2.65
2.64
2.62
2.61
2.60
2.59
2.58
2.56
2.56
2.53
2.52
2.52
2.50
2.41
2.38
2.37
2.36
2.35
2.34
2.34
2.32
2.32
2.17
2.16
2.15
2.14
2.13
2.12
2.12
1.54
1.52
1.50
1.49
0.83
0.81
0.79
Ph
O
N
H
S
n-C3H7
5m
2.00
1.00
1.95
1.02
2.03
6.08
1.19
2.21
1.07
2.02
3.12
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

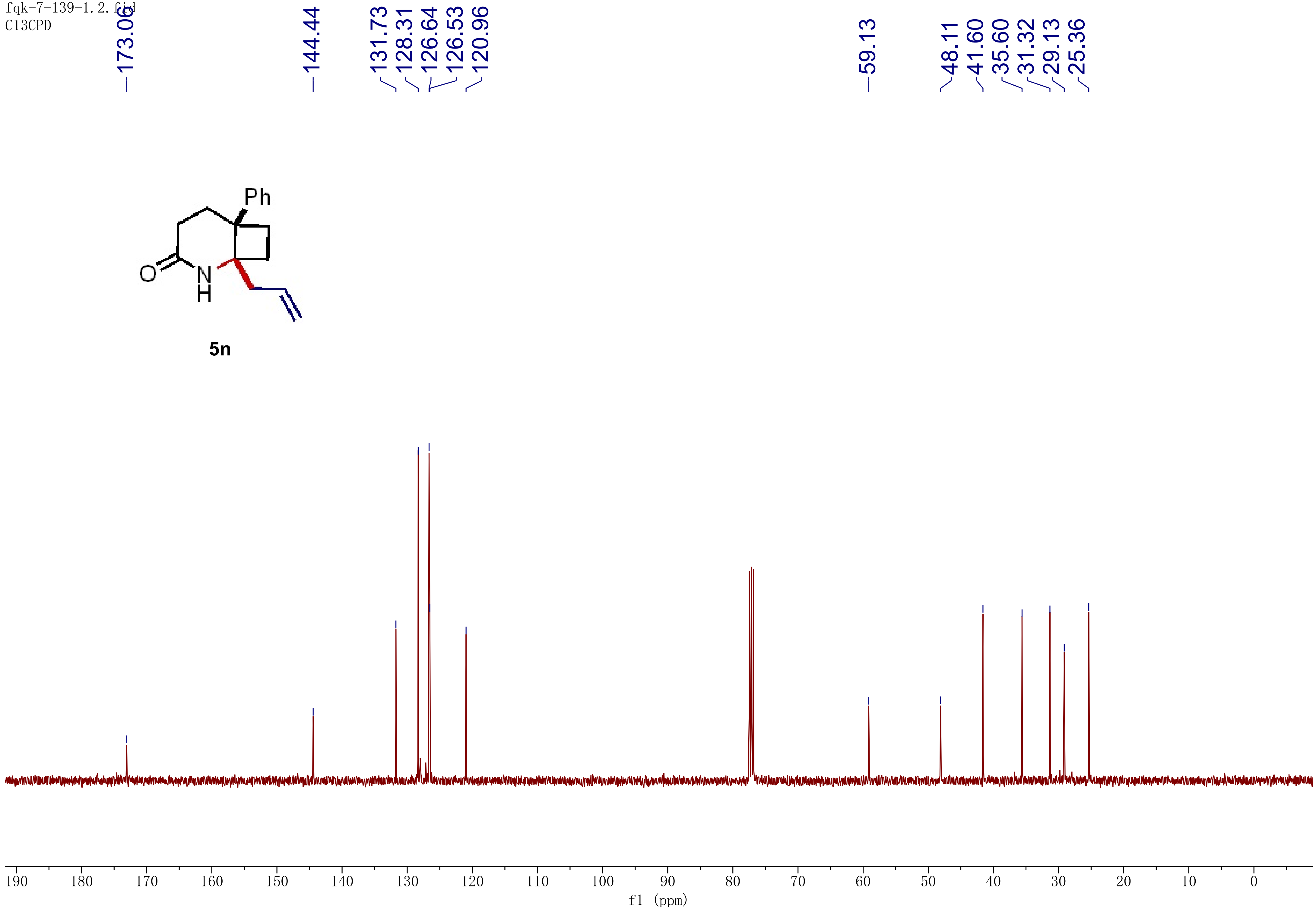
fqk-7-135-7.2.fid
C13CPD

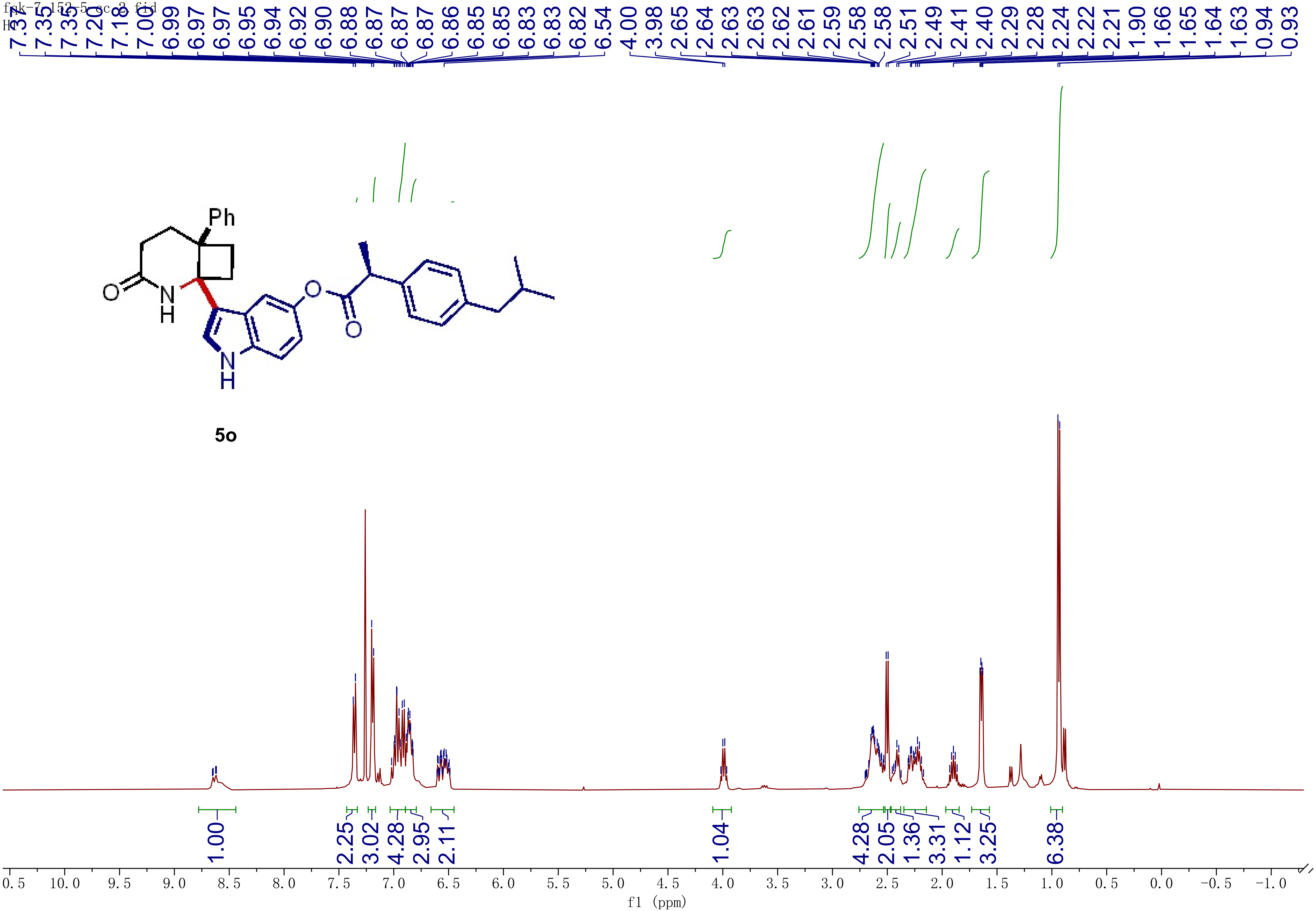


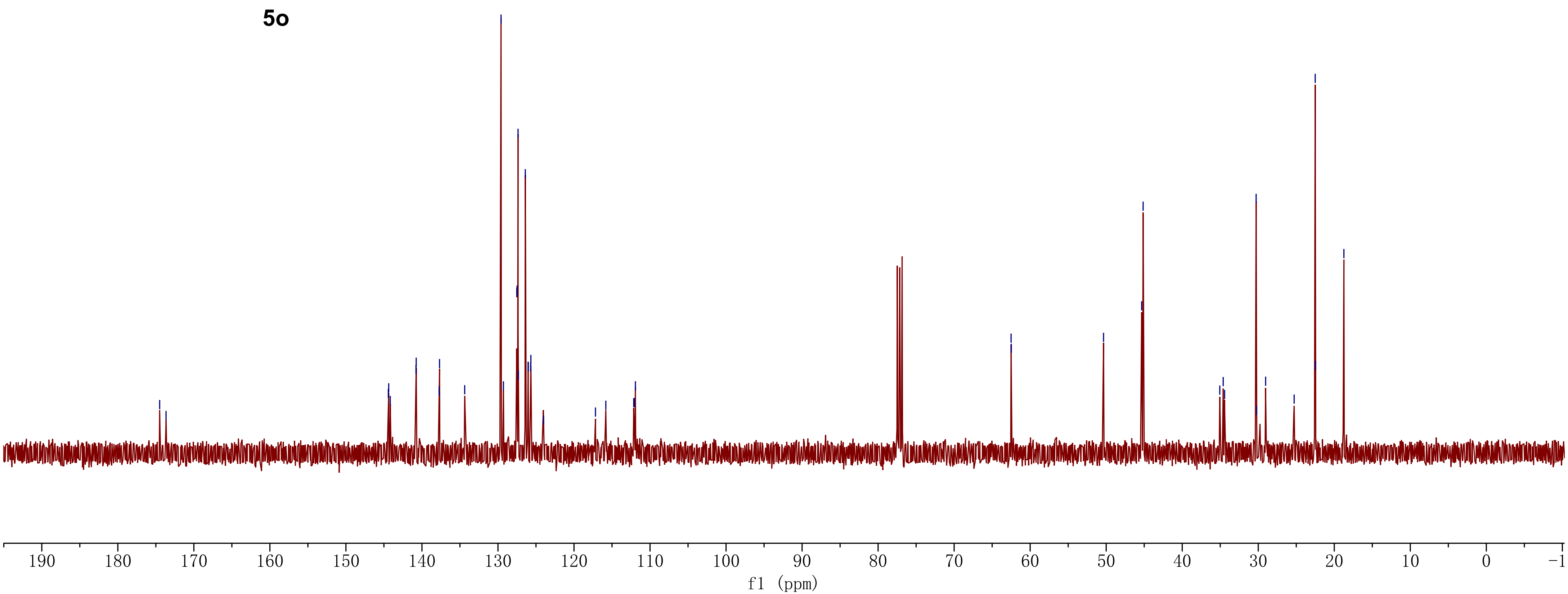
5m

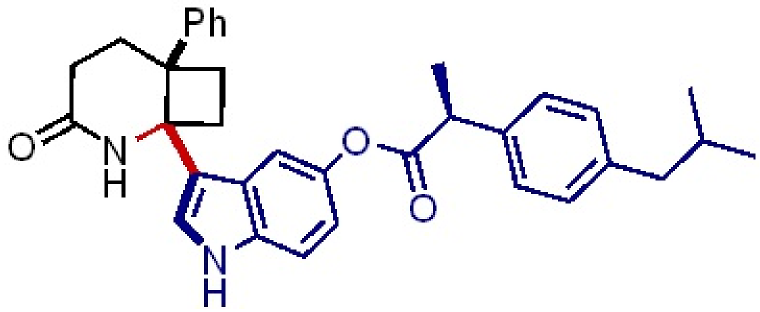



fqk-7-139-1.1.fid
7.36
7.34
7.32
7.26
7.24
7.22
7.14
7.12
6.18
5.74
5.71
5.22
5.19
5.17
5.13
2.55
2.54
2.53
2.52
2.50
2.48
2.39
2.38
2.36
2.35
2.34
2.33
2.32
2.24
2.22
2.21
2.20
2.19
2.17
2.16
2.13
2.12
2.10
2.09
2.07
2.06
2.05
2.05
2.03
2.02
2.01
2.00
1.98
1.96
1.95
Ph
O
N
H
5n
2.28
1.13
2.04
0.98
1.00
2.00
2.37
2.34
3.38
1.96
1.42
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

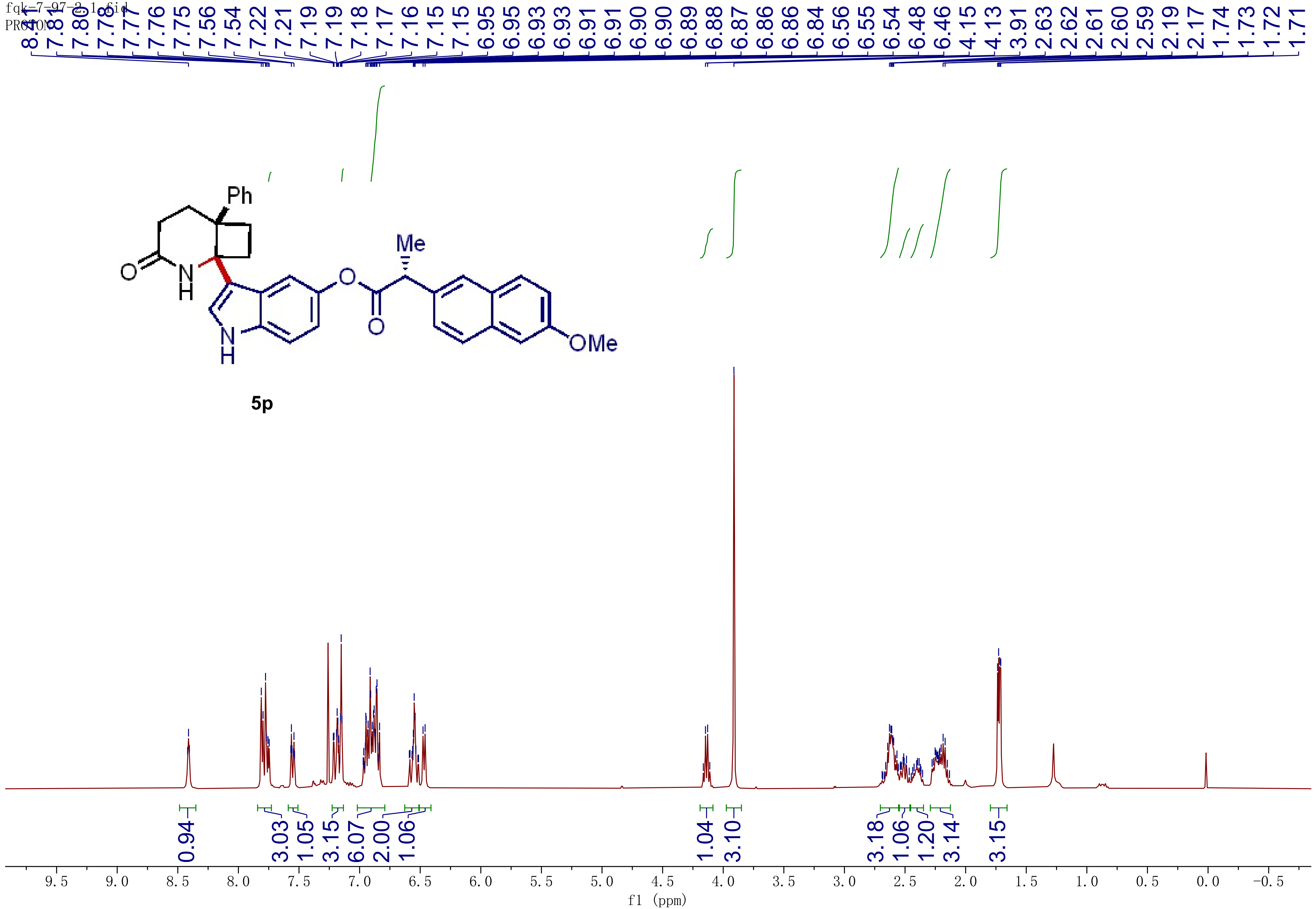

fqk-7-139-1.2.fid
C13CPD
173.06
144.44
131.73
128.31
126.64
126.53
120.96
59.13
48.11
41.60
35.60
31.32
29.13
25.36
Ph
O
N
H
5n
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


fpk-7-152-5-cc-2.fid
7.35 7.35 7.20 7.18 7.00 6.99 6.97 6.97 6.95 6.94 6.92 6.90 6.88 6.87 6.87 6.87 6.86 6.85 6.85 6.83 6.83 6.82 6.54 4.00 3.98 2.65 2.64 2.63 2.63 2.62 2.61 2.59 2.58 2.58 2.51 2.49 2.41 2.40 2.29 2.28 2.24 2.22 2.21 1.90 1.66 1.65 1.64 1.63 0.94 0.93
Ph
O
N
H
O
O
N
H
5o
1.00 2.25 3.02 4.28 2.95 2.11 1.04 4.28 2.05 1.36 3.31 1.12 3.25 6.38
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

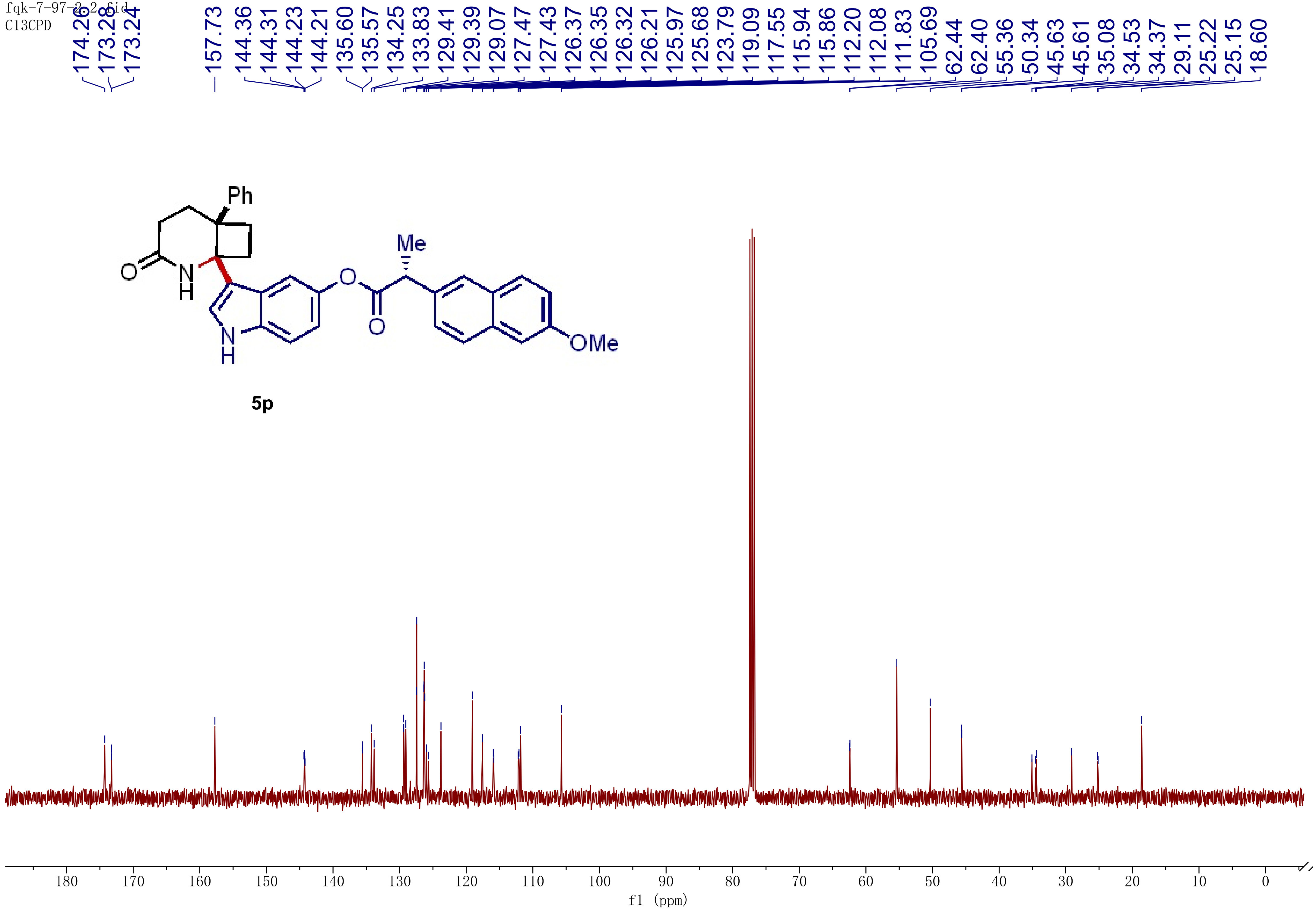

fqk-7-152-5-cc.4.fid
1D 13C with H decoupling
174.50
173.66
144.42
144.37
144.17
140.78
140.75
137.74
137.69
134.38
129.60
129.28
127.53
127.49
127.36
127.32
126.40
126.04
126.01
125.70
125.67
124.00
117.18
115.81
112.14
112.05
111.92
62.51
62.48
50.35
45.33
45.14
35.06
34.61
34.43
30.28
30.20
29.03
25.28
22.51
22.47
18.73
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

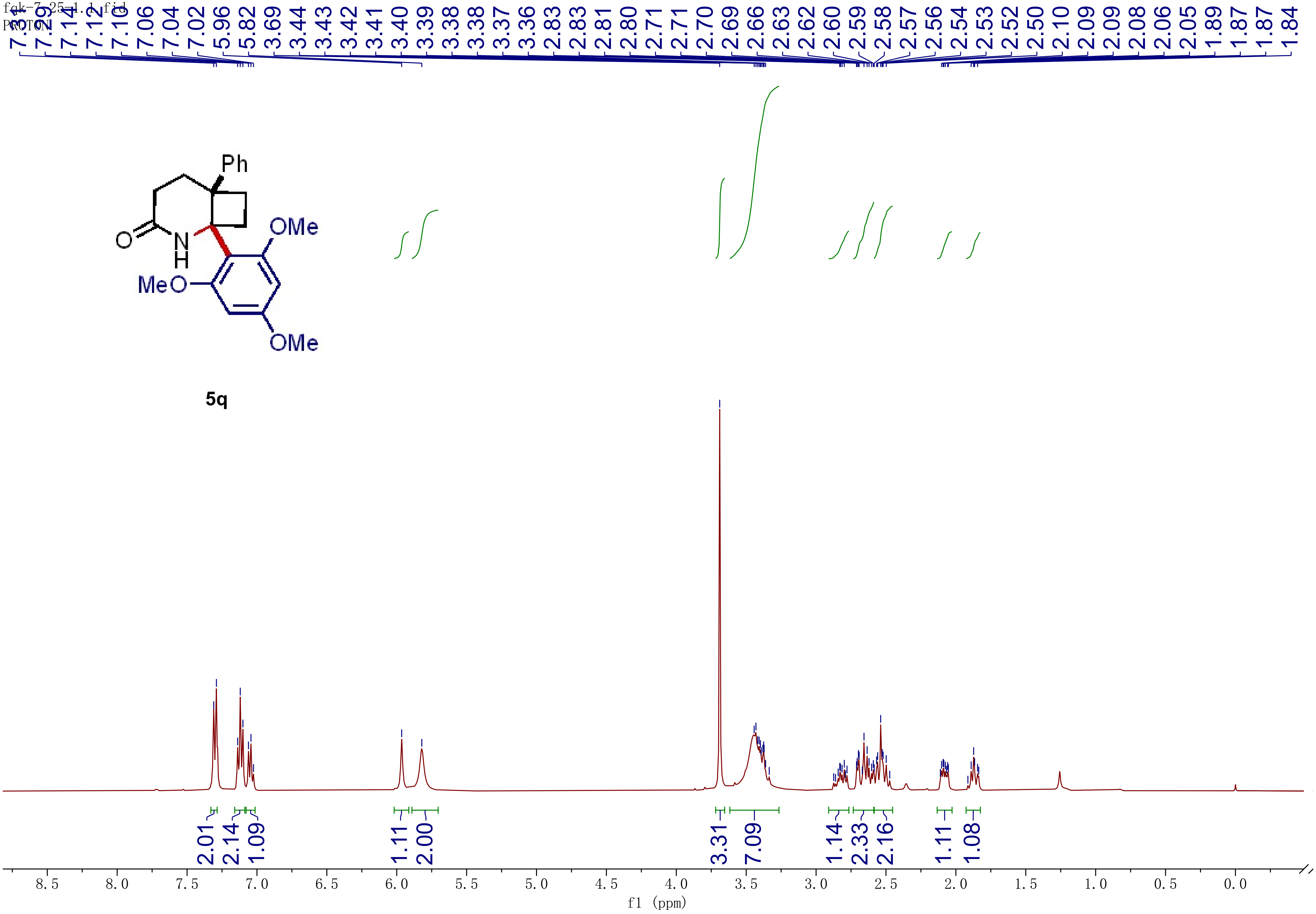

Ph
O
N
H
O
O
N
H

5o

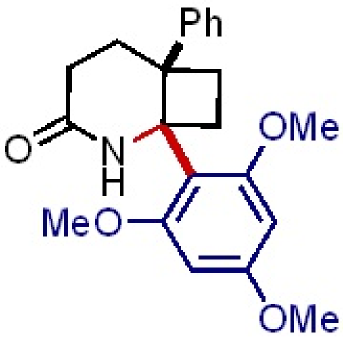

fqk-7-97-2.1.fid
PROTON
8.41 7.81 7.80 7.78 7.77 7.76 7.75 7.56 7.54 7.22 7.21 7.19 7.19 7.18 7.17 7.16 7.15 7.15 6.95 6.95 6.93 6.93 6.91 6.91 6.90 6.90 6.89 6.88 6.87 6.86 6.86 6.84 6.56 6.55 6.54 6.48 6.46 4.15 4.13 3.91 2.63 2.62 2.61 2.60 2.59 2.19 2.17 1.74 1.73 1.72 1.71
Ph
Me
O
N
H
OMe
0.94 3.03 1.05 3.15 6.07 2.00 1.06 1.04 3.10 3.18 1.06 1.20 3.14 3.15
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

5p

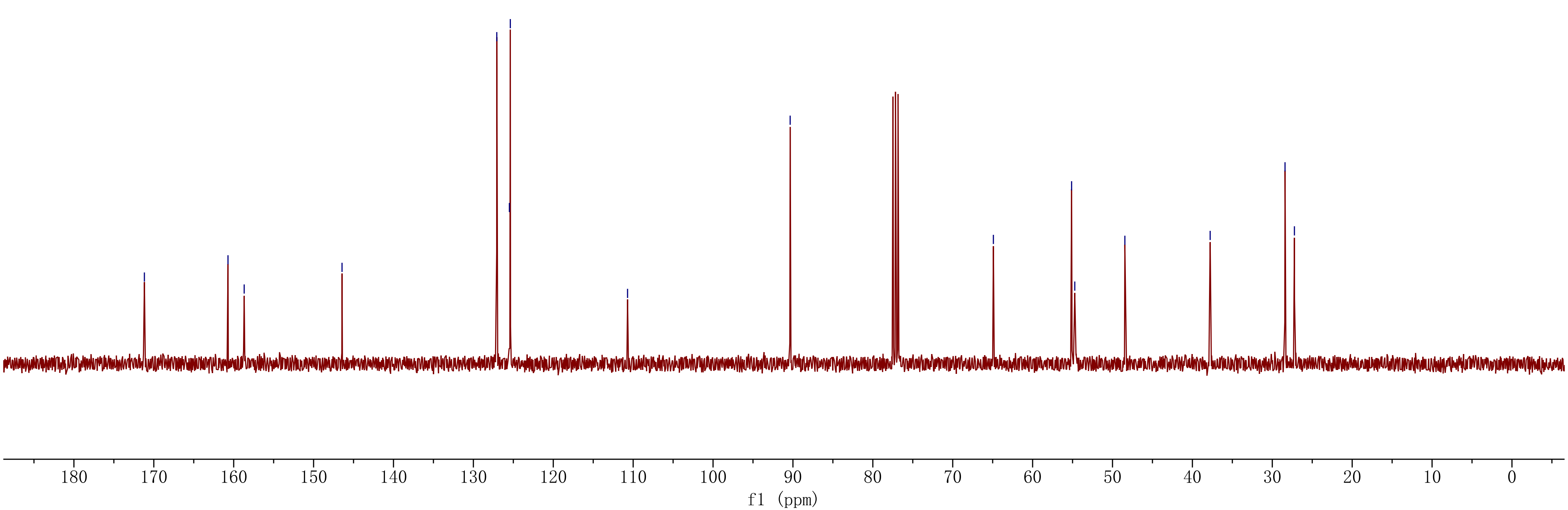

fqk-7-97-2.2.fid
C13CPD
174.26
173.28
173.24
157.73
144.36
144.31
144.23
144.21
135.60
135.57
134.25
133.83
129.41
129.39
129.07
127.47
127.43
126.37
126.35
126.32
126.21
125.97
125.68
123.79
119.09
117.55
115.94
115.86
112.20
112.08
111.83
105.69
62.44
62.40
55.36
50.34
45.63
45.61
35.08
34.53
34.37
29.11
25.22
25.15
18.60
Ph
Me
O
N
H
OMe
5p
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


fqk-7-25-1.1.fid
PROTON
7.14 7.12 7.10 7.06 7.04 7.02 5.96 5.82 3.69 3.44 3.43 3.42 3.41 3.40 3.39 3.38 3.38 3.37 3.36 2.83 2.83 2.81 2.80 2.71 2.71 2.70 2.69 2.66 2.63 2.62 2.60 2.59 2.58 2.57 2.56 2.54 2.53 2.52 2.50 2.10 2.09 2.09 2.08 2.06 2.05 1.89 1.87 1.87 1.84
Ph
OMe
O
N
H
MeO
OMe
5q
2.01 2.14 1.09 1.11 2.00 3.31 7.09 1.14 2.33 2.16 1.11 1.08
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

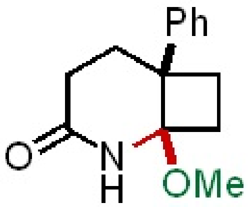
fqk-7-25-1.2.fid
C13CPD



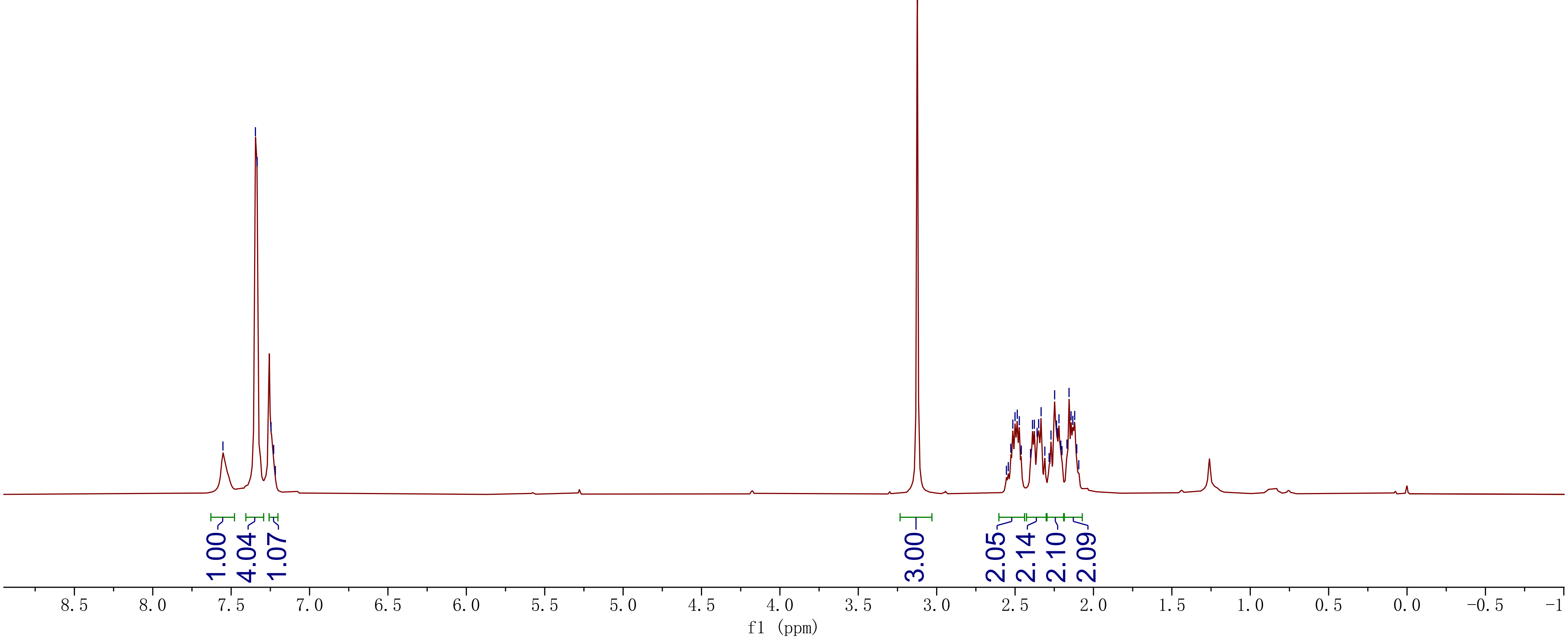
5q

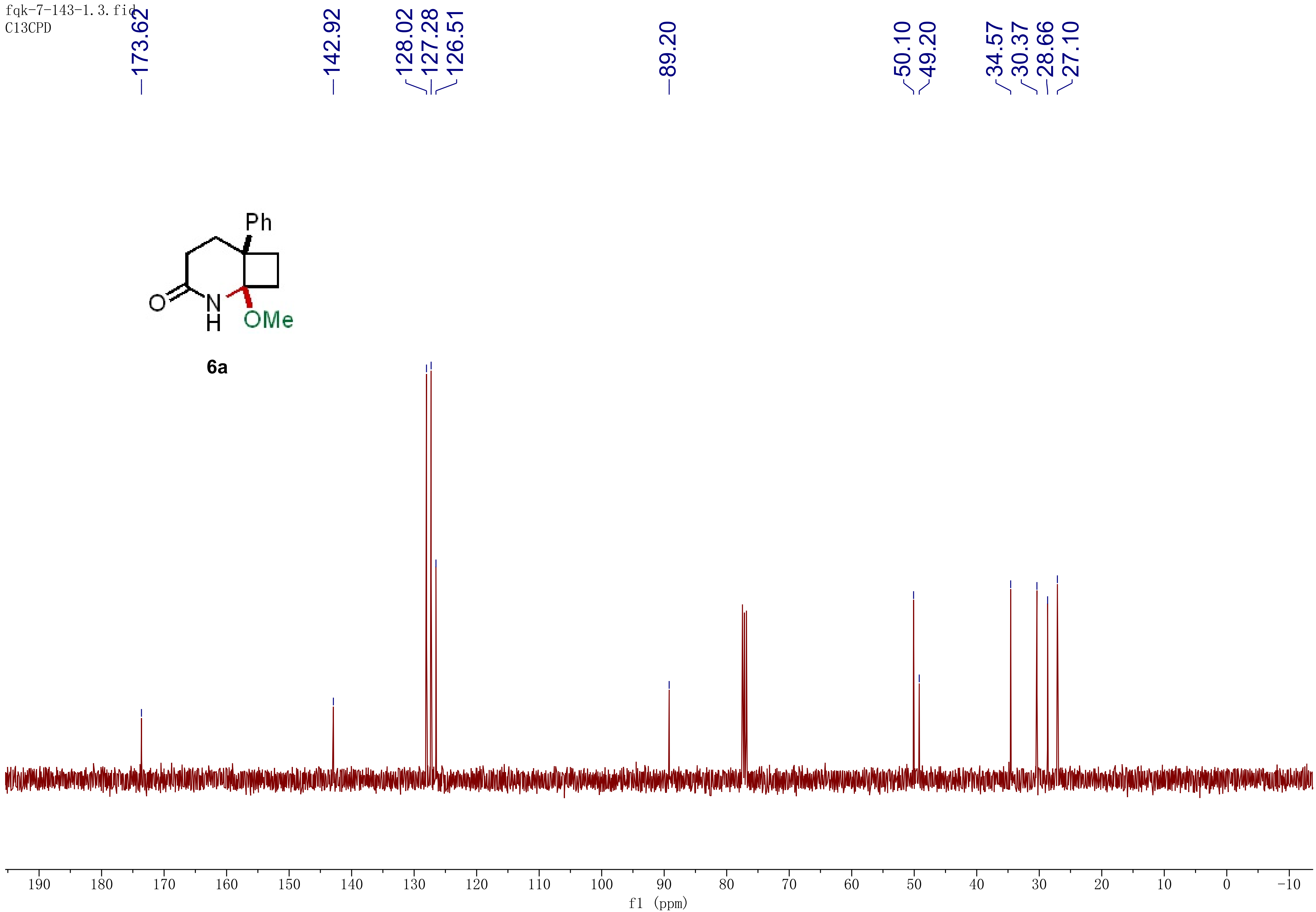


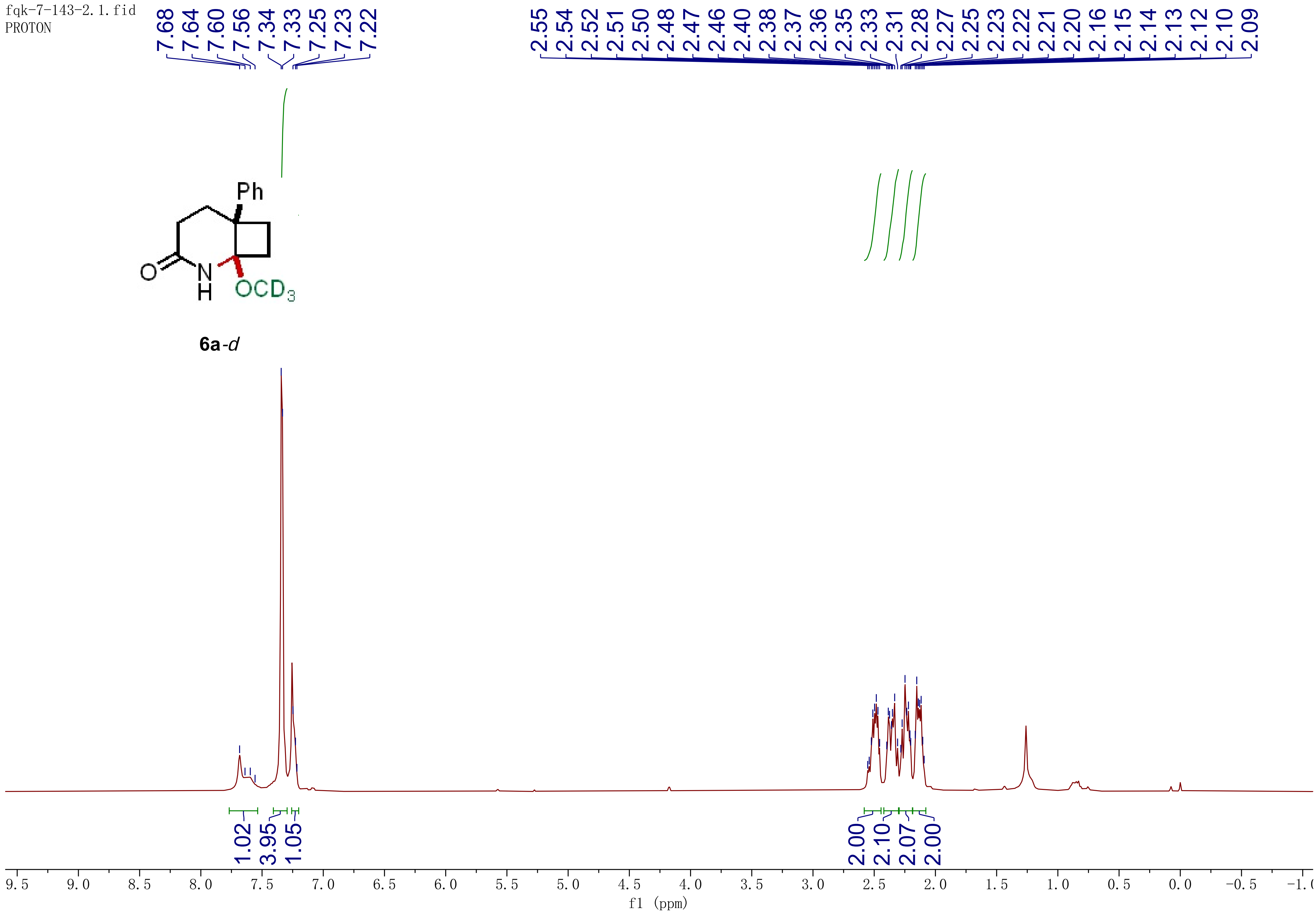
fqk-7-143-1.1.fid
PROTON

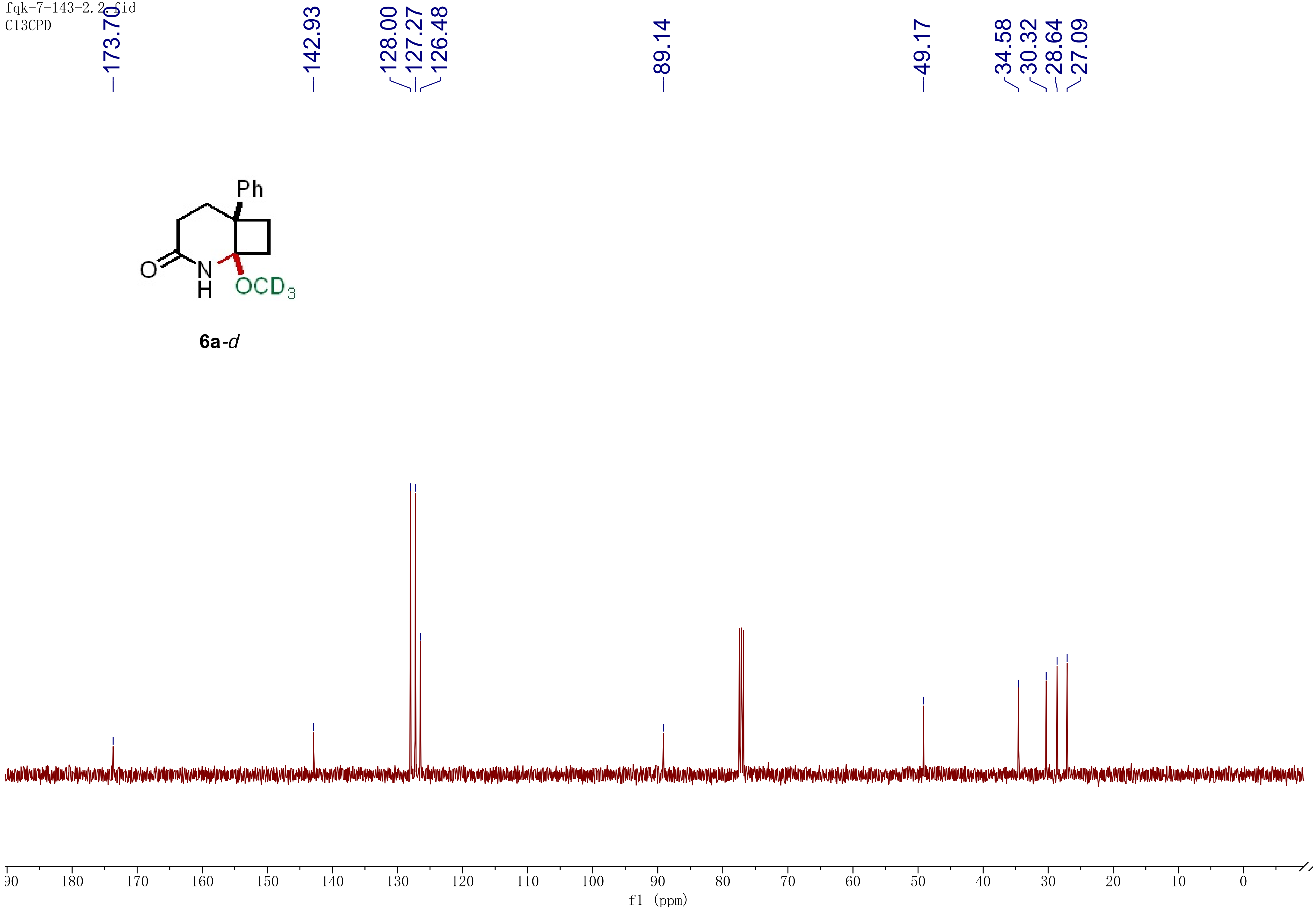


6a




fqk-7-143-1.3.fid
C13CPD
173.62
142.92
128.02
127.28
126.51
89.20
50.10
49.20
34.57
30.37
28.66
27.10
Ph
O
N
H
OMe
6a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


fqk-7-143-2.1.fid
PROTON
7.68
7.64
7.60
7.56
7.34
7.33
7.25
7.23
7.22
2.55
2.54
2.52
2.51
2.50
2.48
2.47
2.46
2.40
2.38
2.37
2.36
2.35
2.33
2.31
2.28
2.27
2.25
2.23
2.22
2.21
2.20
2.16
2.15
2.14
2.13
2.12
2.10
2.09
Ph
O
N
H
OCD3
6a-d
1.02
3.95
1.05
2.00
2.10
2.07
2.00
f1 (ppm)


fqk-7-143-2.2.fid
C13CPD
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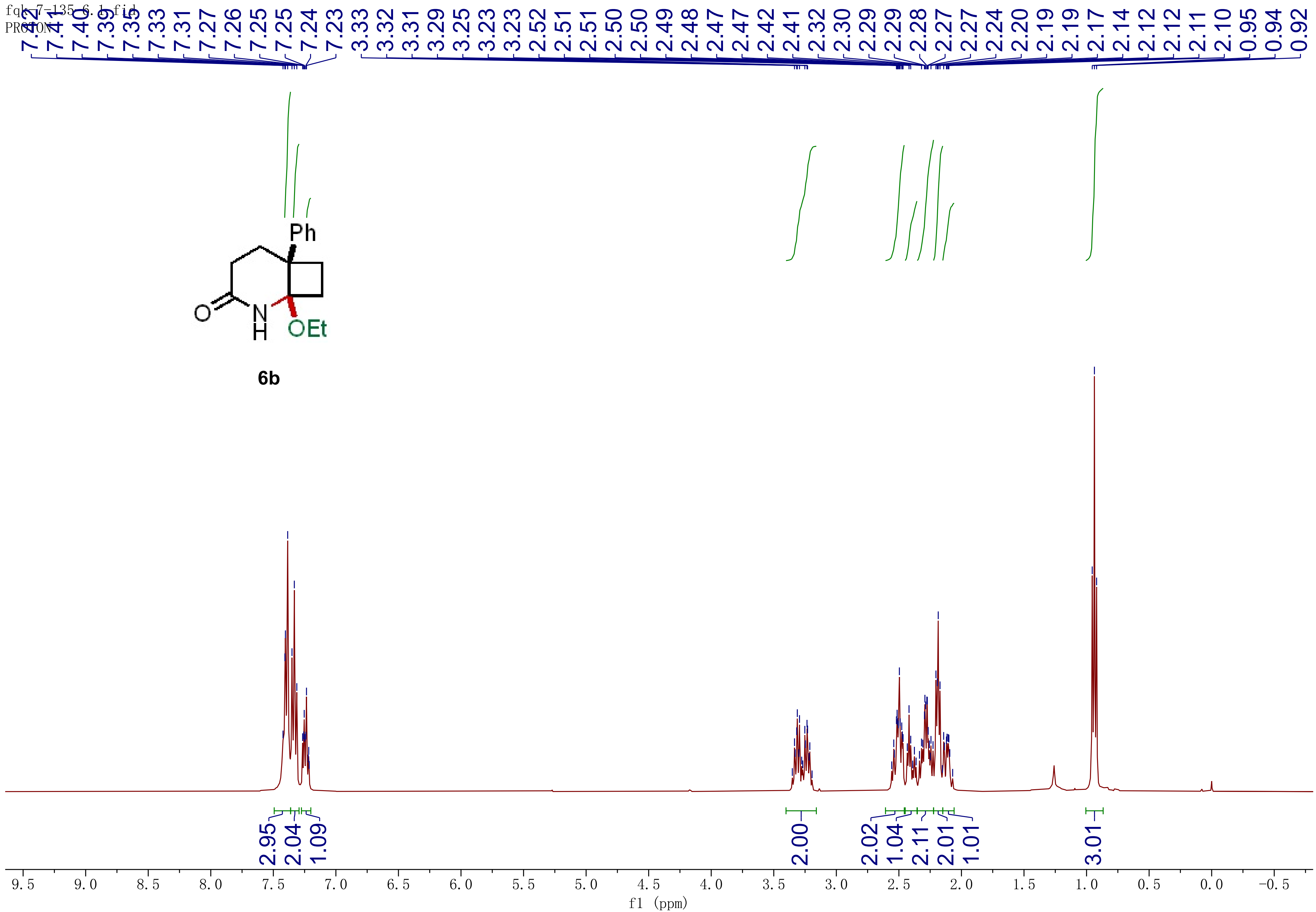
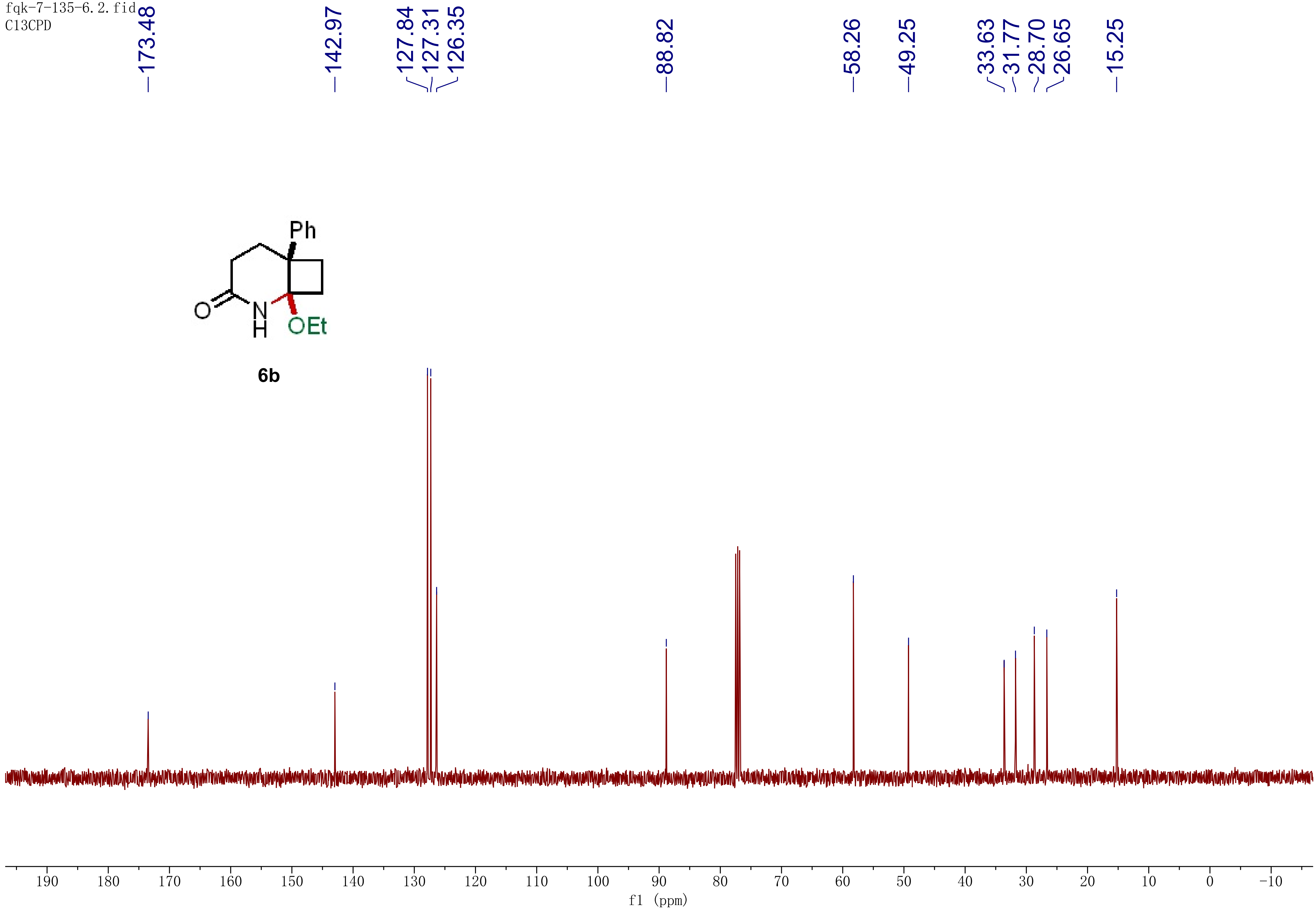
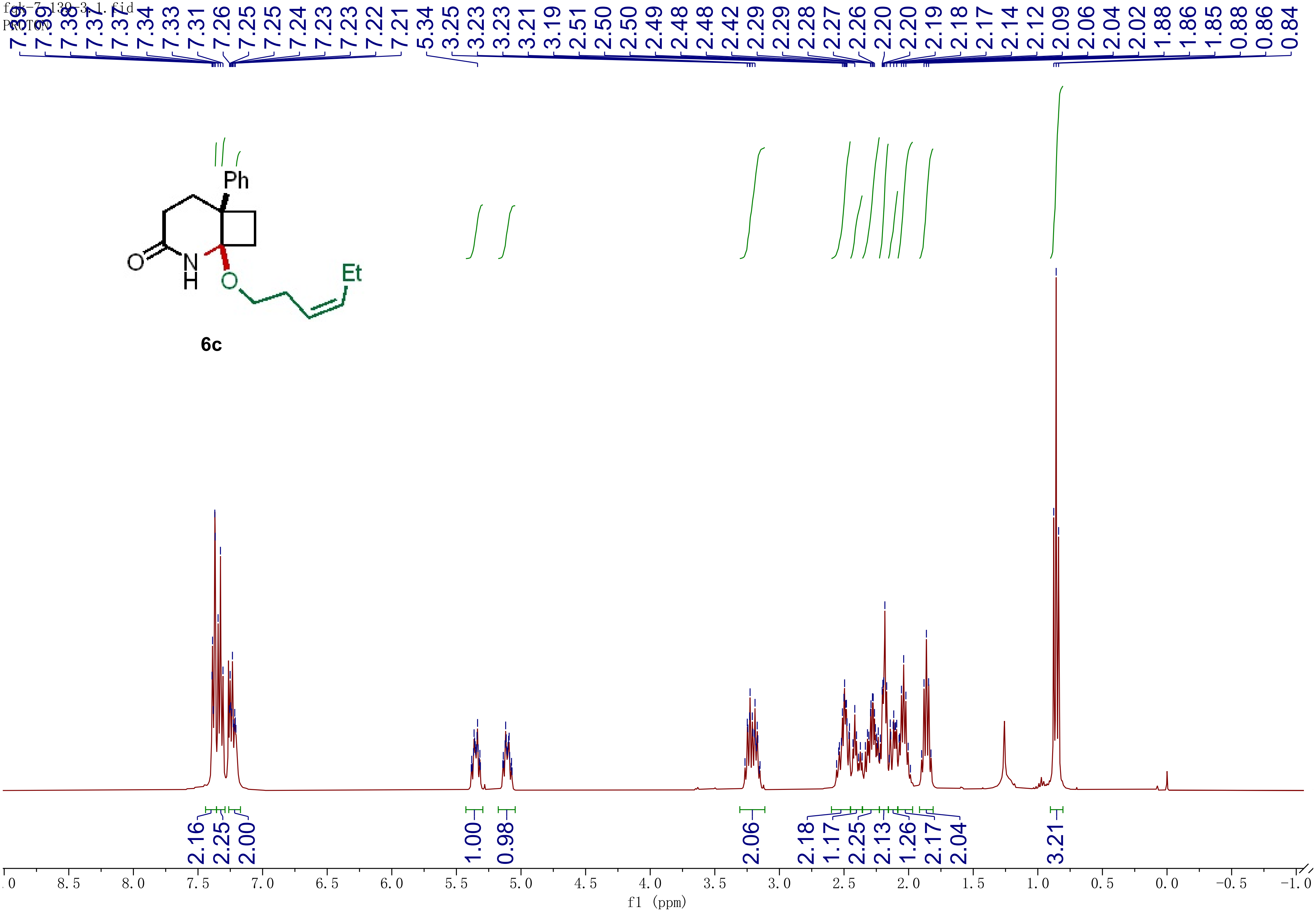
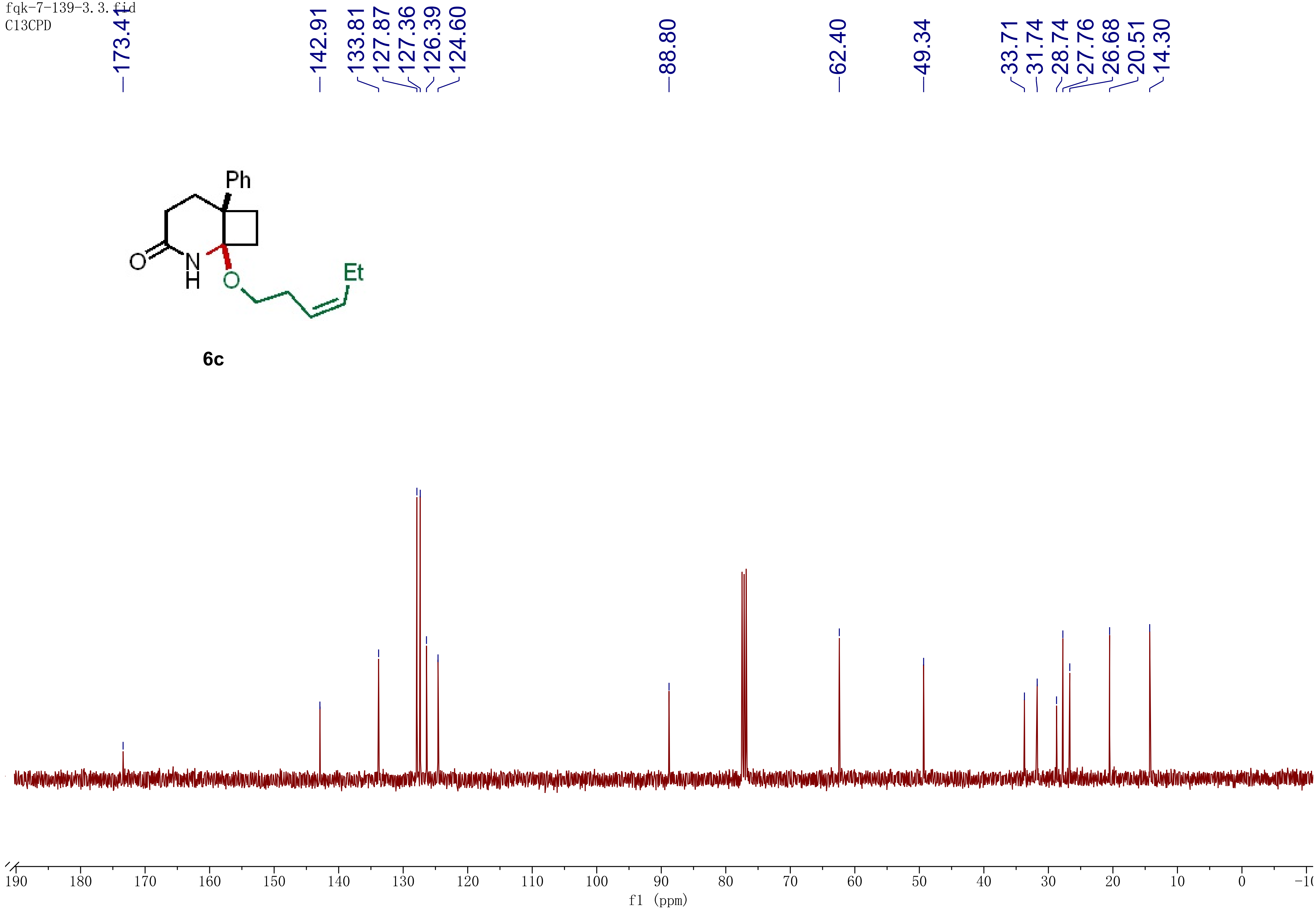
142.93
128.00
127.27
126.48
89.14
49.17
34.58
30.32
28.64
27.09
Ph
O
N
H
OCD3
6a-d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

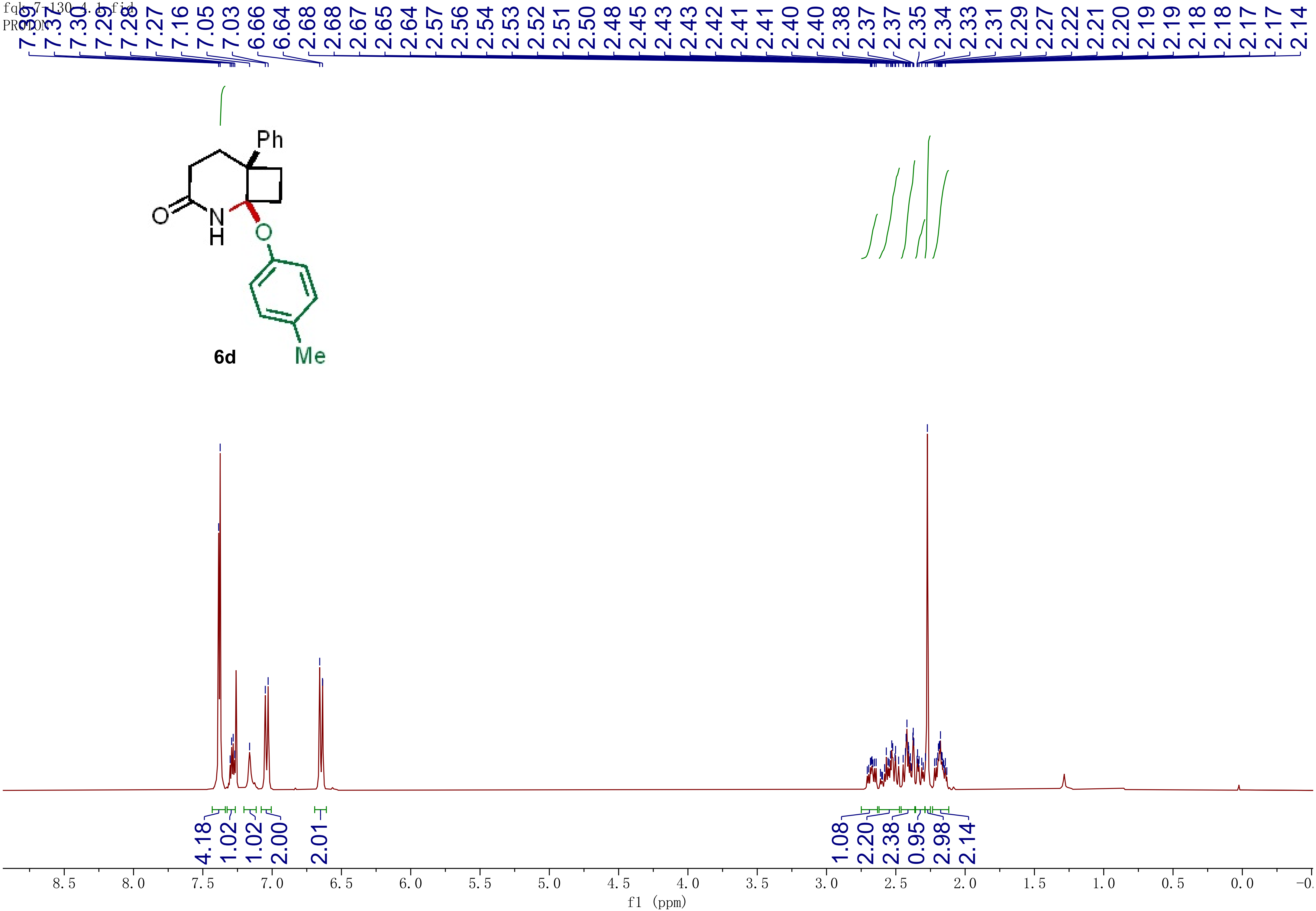

fqk-7-135 6.1.fid
PROTON
7.40
7.39
7.35
7.33
7.31
7.27
7.26
7.25
7.25
7.24
7.23
3.33
3.32
3.31
3.29
3.25
3.23
3.23
2.52
2.51
2.51
2.50
2.50
2.49
2.48
2.47
2.47
2.42
2.41
2.32
2.30
2.29
2.29
2.28
2.27
2.27
2.24
2.20
2.19
2.19
2.17
2.14
2.12
2.12
2.11
2.10
0.95
0.94
0.92
Ph
O
N
H
OEt
6b
2.95
2.04
1.09
2.00
2.02
1.04
2.11
2.01
1.01
3.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


fqk-7-135-6.2.fid
C13CPD
173.48
142.97
127.84
127.31
126.35
88.82
58.26
49.25
33.63
31.77
28.70
26.65
15.25
Ph
O
N
H
OEt
6b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

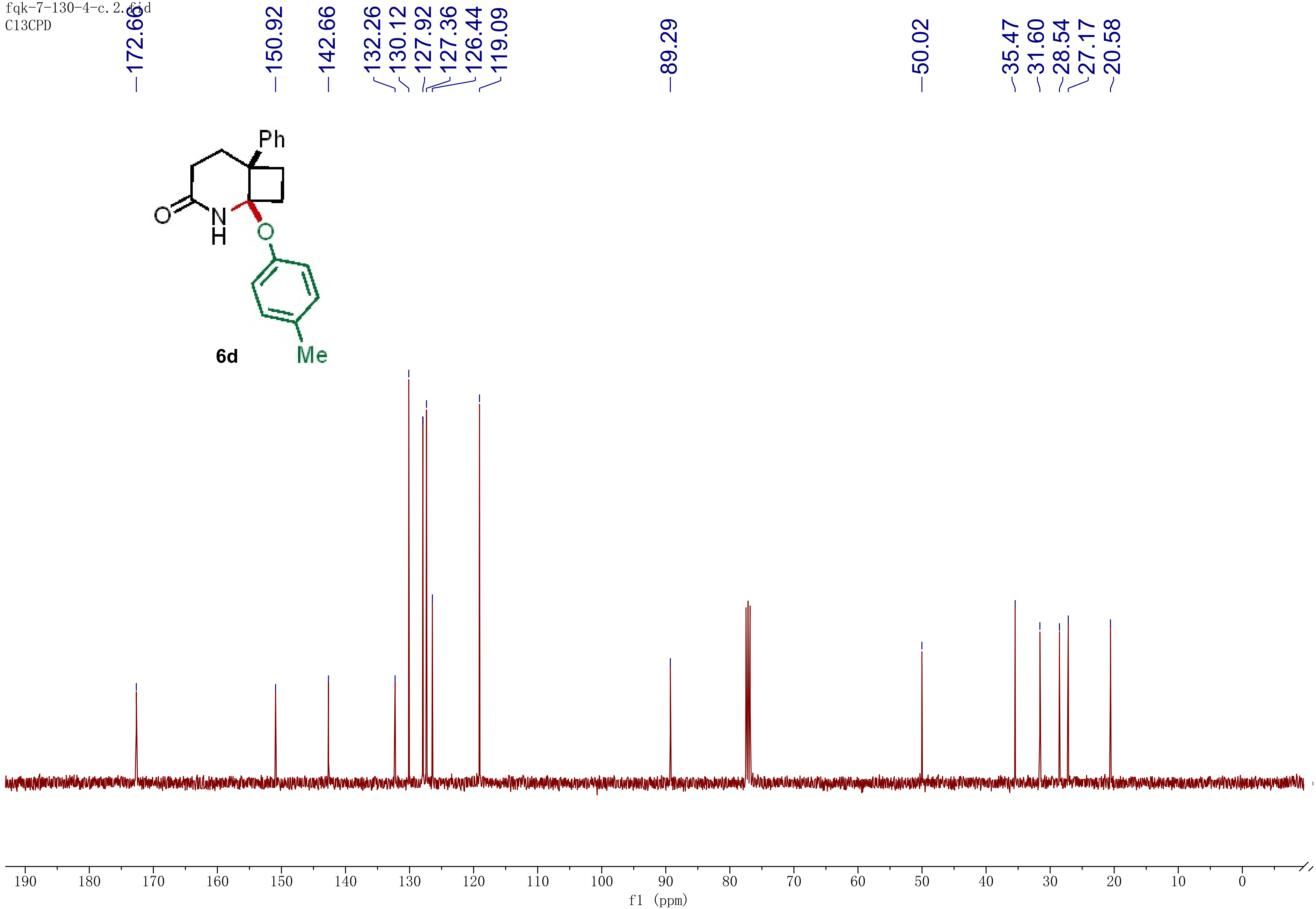


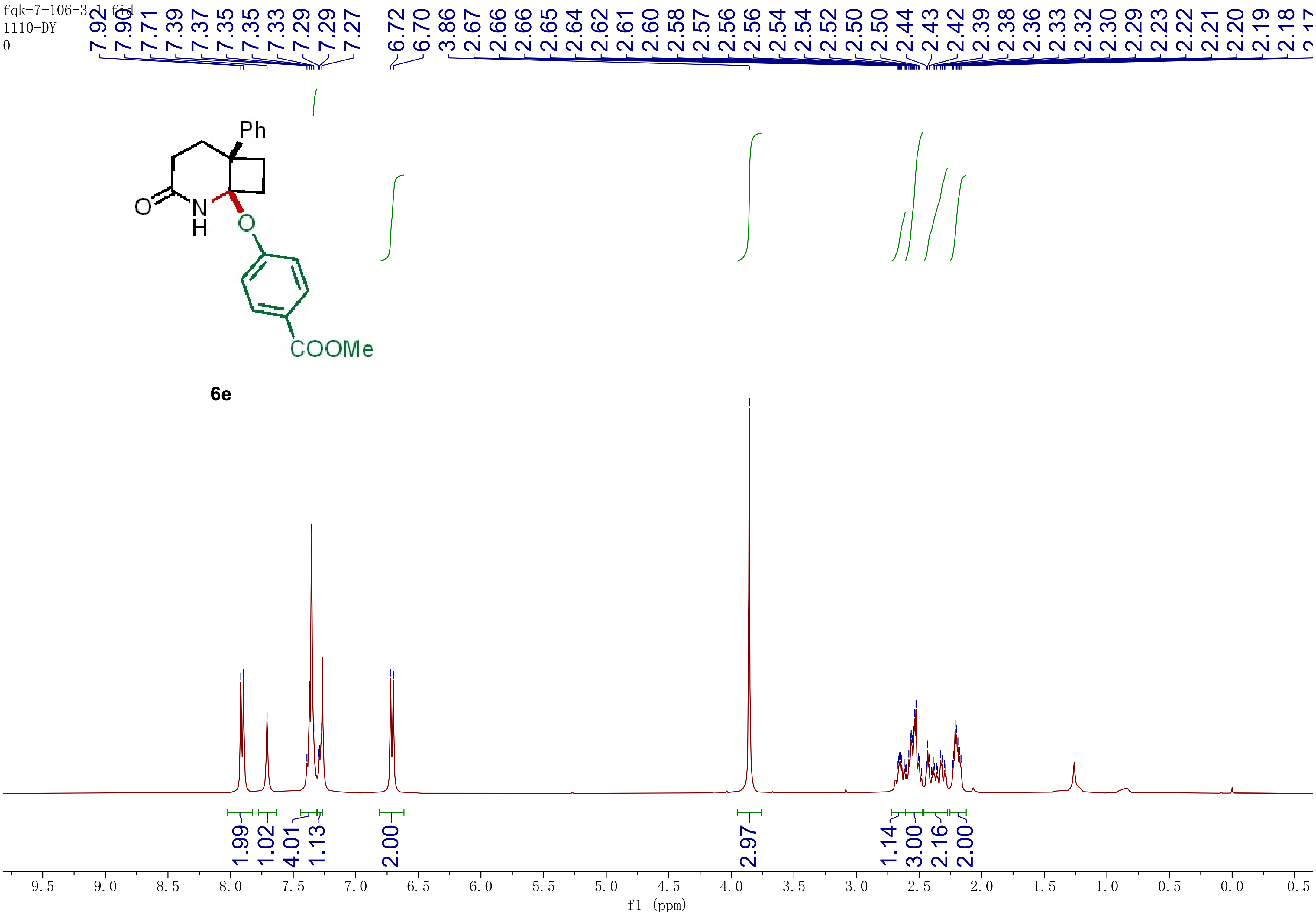
fqk-7-139-3.1.fid
PROTON
7.39 7.39 7.38 7.37 7.37 7.34 7.33 7.31 7.26 7.25 7.25 7.24 7.23 7.23 7.22 7.21 5.34 3.25 3.23 3.23 3.21 3.19 2.51 2.50 2.50 2.49 2.48 2.48 2.42 2.29 2.29 2.28 2.27 2.26 2.20 2.20 2.19 2.18 2.17 2.14 2.12 2.09 2.06 2.04 2.02 1.88 1.86 1.85 0.88 0.86 0.84
Ph
O
N
H
O
Et
6c
2.16 2.25 2.00 1.00 0.98 2.06 2.18 1.17 2.25 2.13 1.26 2.17 2.04 3.21
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

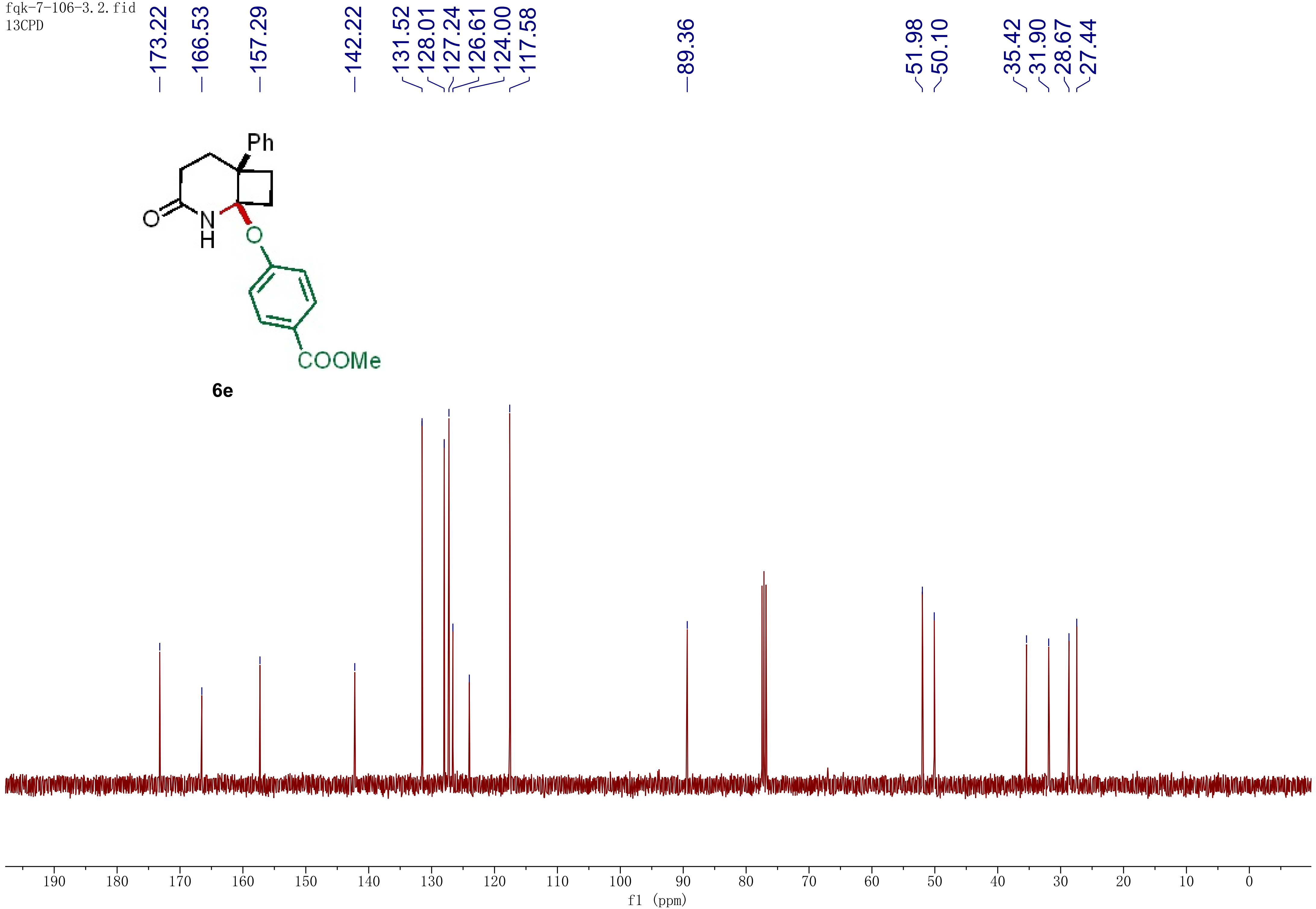

fqk-7-139-3.3.fid
C13CPD
173.41
142.91
133.81
127.87
127.36
126.39
124.60
88.80
62.40
49.34
33.71
31.74
28.74
27.76
26.68
20.51
14.30
Ph
O
N
H
O
Et
6c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

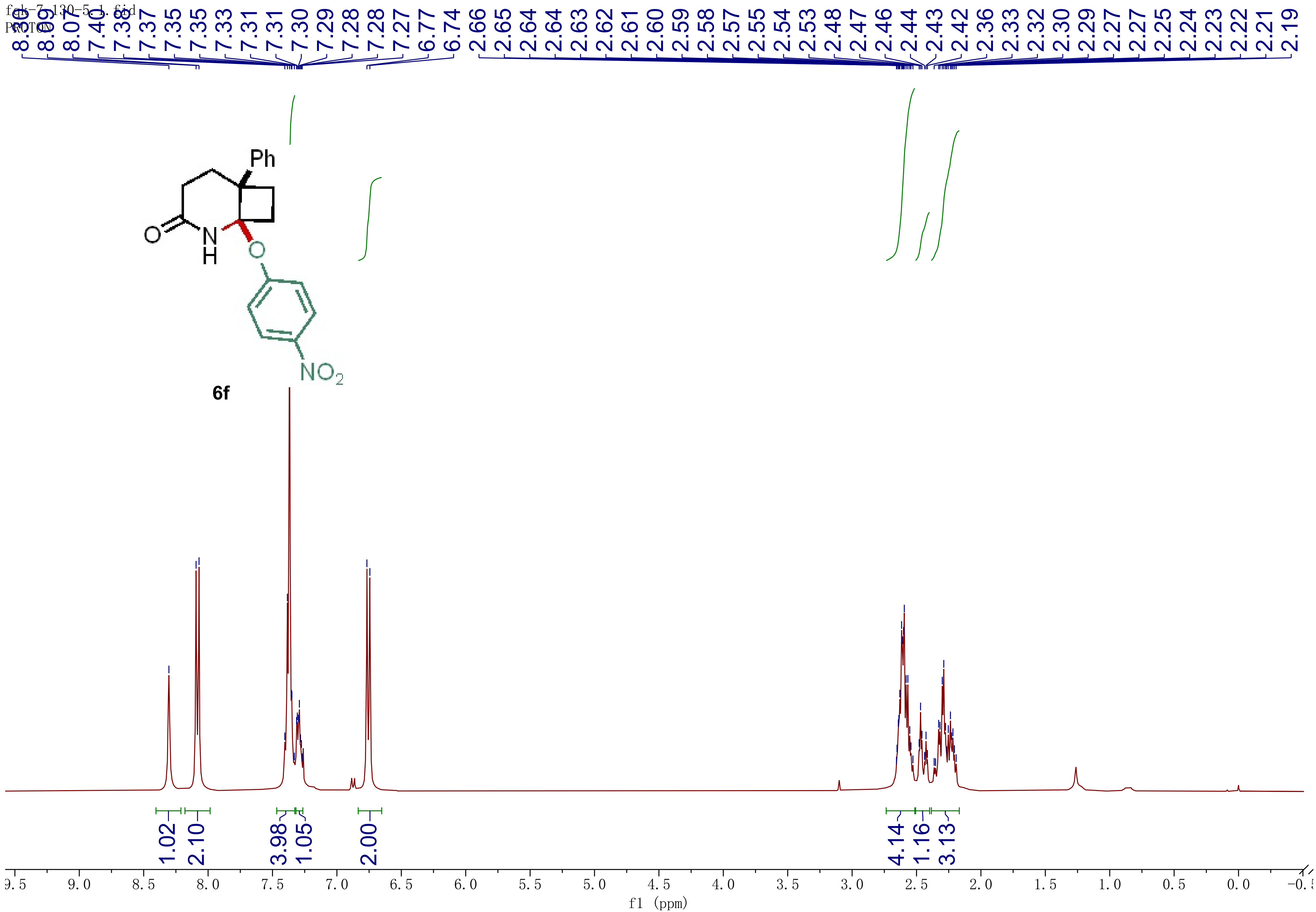


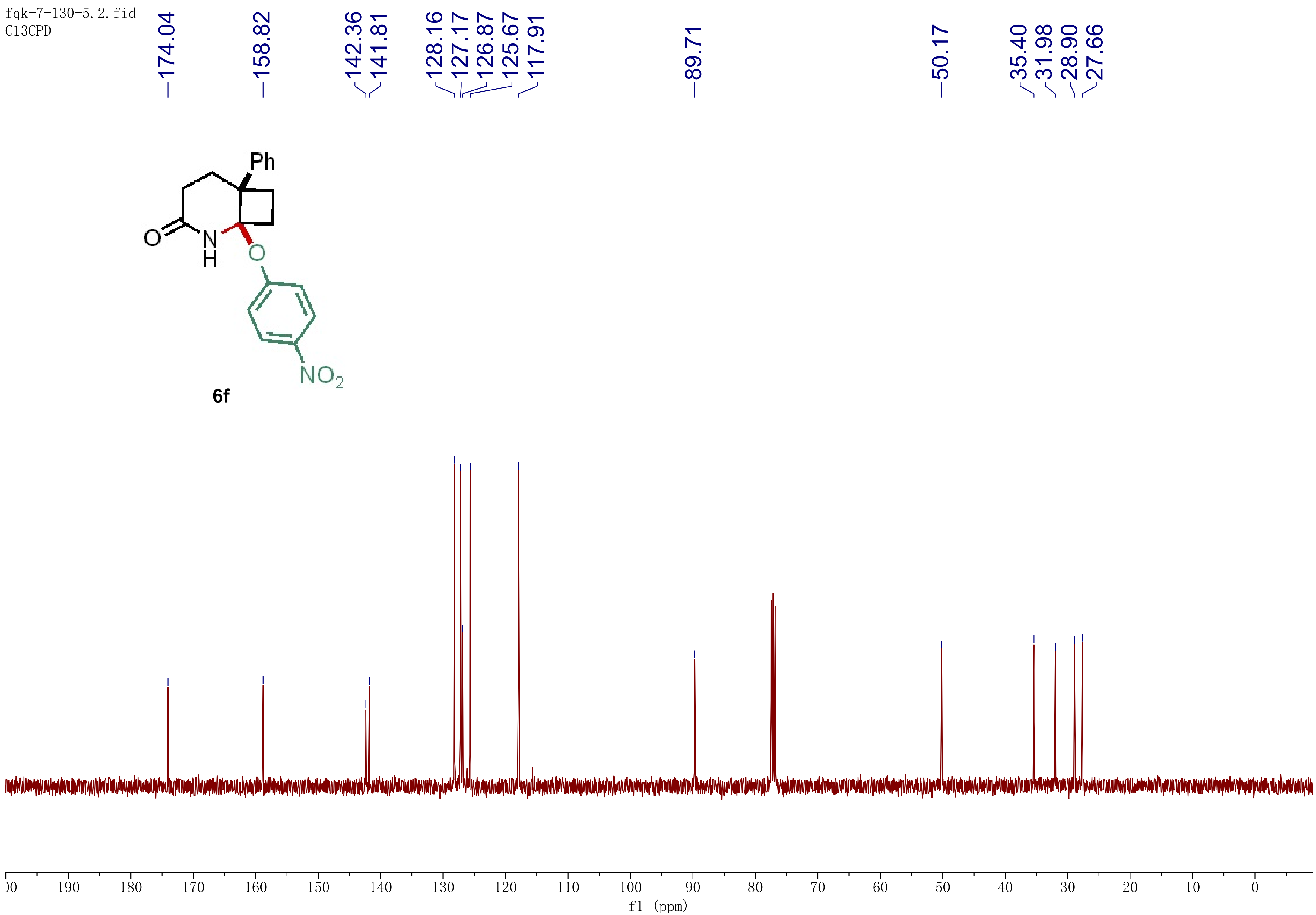
fqk-7-130-4.1.fid
PROTON
7.37
7.30
7.29
7.28
7.27
7.16
7.05
7.03
6.66
6.64
2.68
2.68
2.67
2.65
2.64
2.57
2.56
2.54
2.53
2.52
2.51
2.50
2.48
2.45
2.43
2.43
2.42
2.41
2.41
2.40
2.40
2.38
2.37
2.37
2.35
2.34
2.33
2.31
2.29
2.27
2.22
2.21
2.20
2.19
2.19
2.18
2.18
2.17
2.17
2.14
Ph
O
N
H
O
Me
6d
4.18
1.02
1.02
2.00
2.01
1.08
2.20
2.38
0.95
2.98
2.14
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

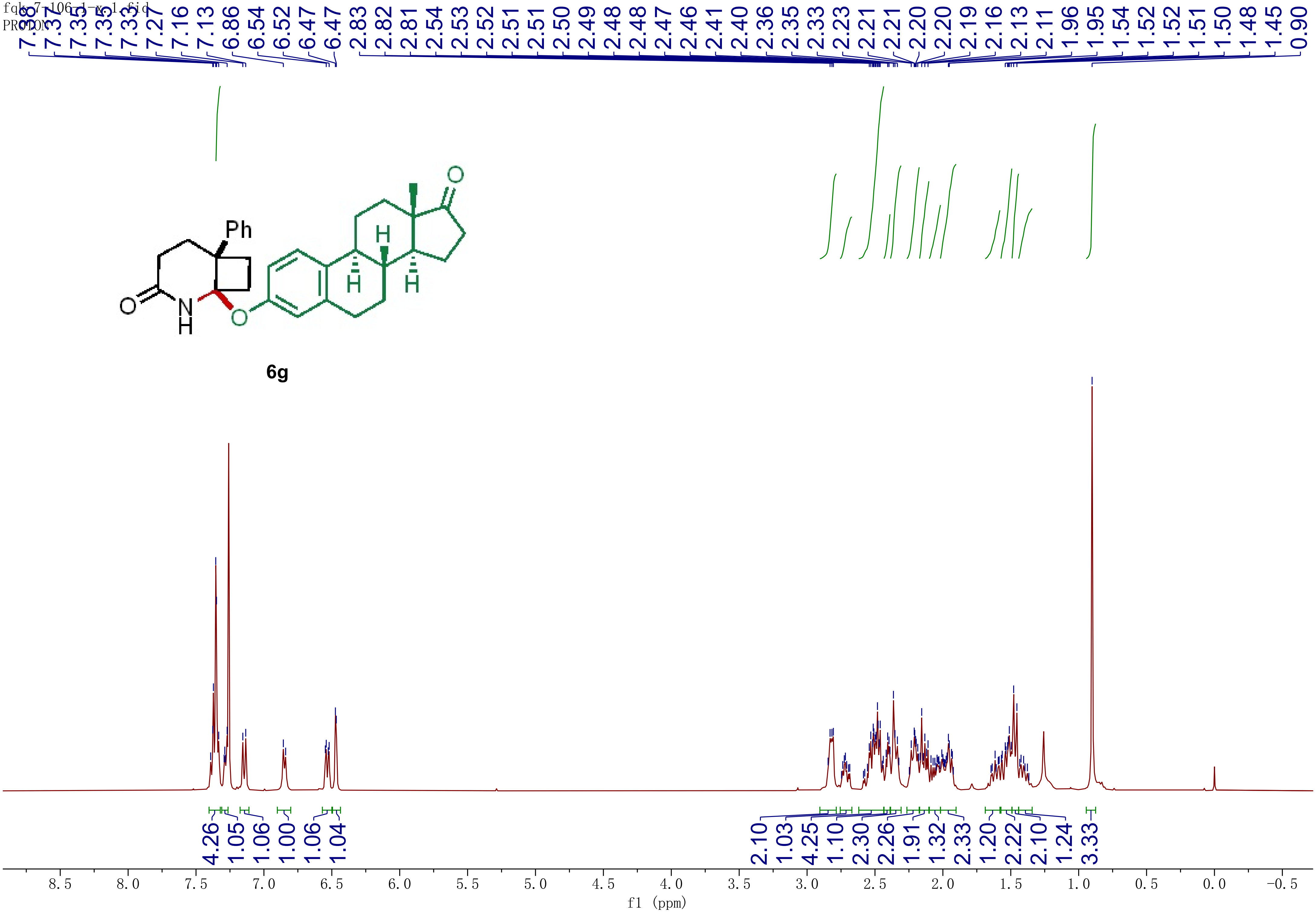

fqk-7-130-4-c.2.fid
C13CPD
172.66
150.92
142.66
132.26
130.12
127.92
127.36
126.44
119.09
89.29
50.02
35.47
31.60
28.54
27.17
20.58
Ph
O
N
H
O
Me
6d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

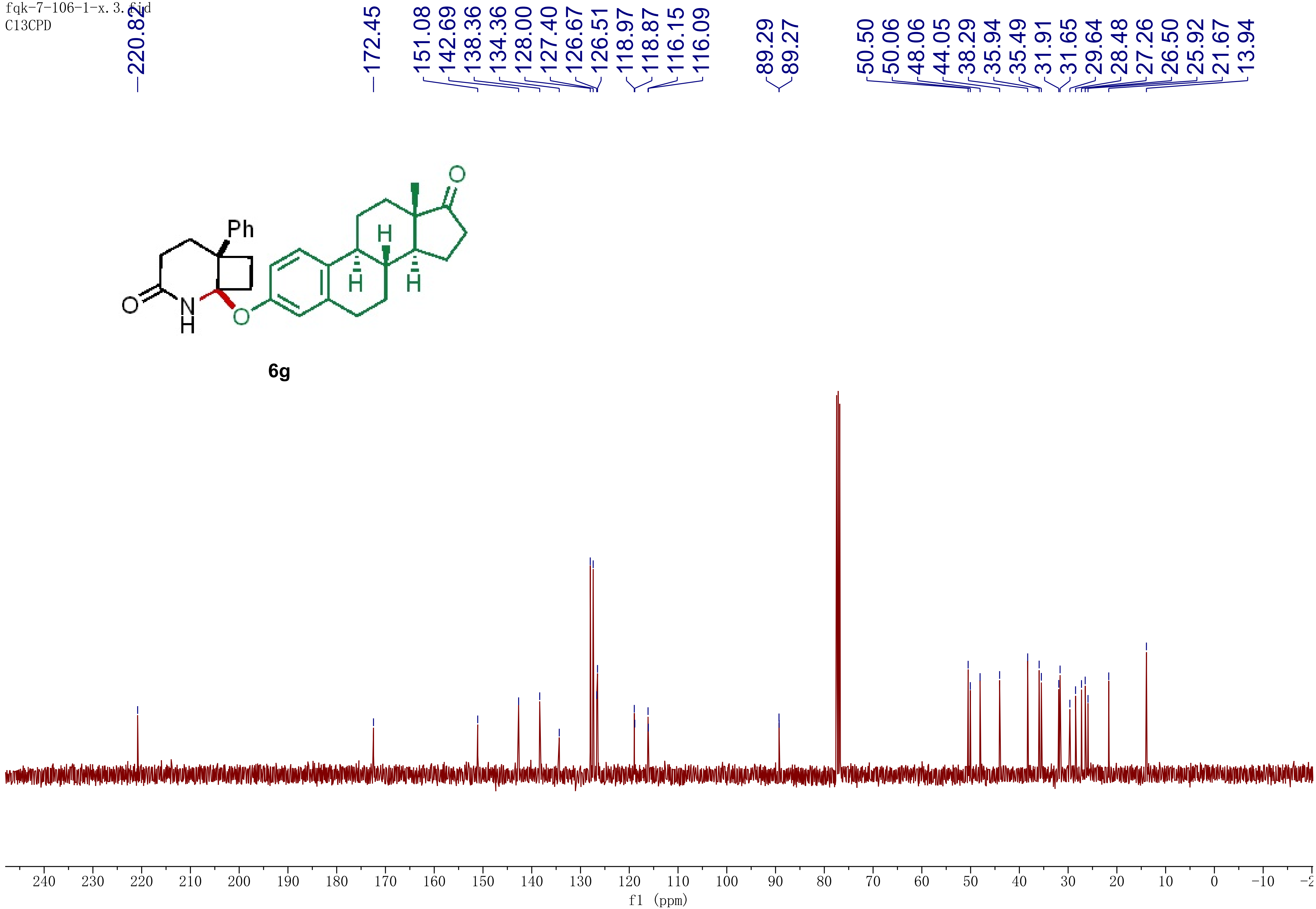

fqk-7-106-3.1.fid
1110-DY
0
7.92 7.90 7.71 7.39 7.37 7.35 7.35 7.33 7.29 7.29 7.27 6.72 6.70 3.86 2.67 2.66 2.66 2.65 2.64 2.62 2.61 2.60 2.58 2.57 2.56 2.56 2.54 2.54 2.52 2.50 2.50 2.44 2.43 2.42 2.39 2.38 2.36 2.33 2.32 2.30 2.29 2.23 2.22 2.21 2.20 2.19 2.18
Ph
O
N
H
O
COOMe
6e
1.99 1.02 4.01 1.13 2.00 2.97 1.14 3.00 2.16 2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

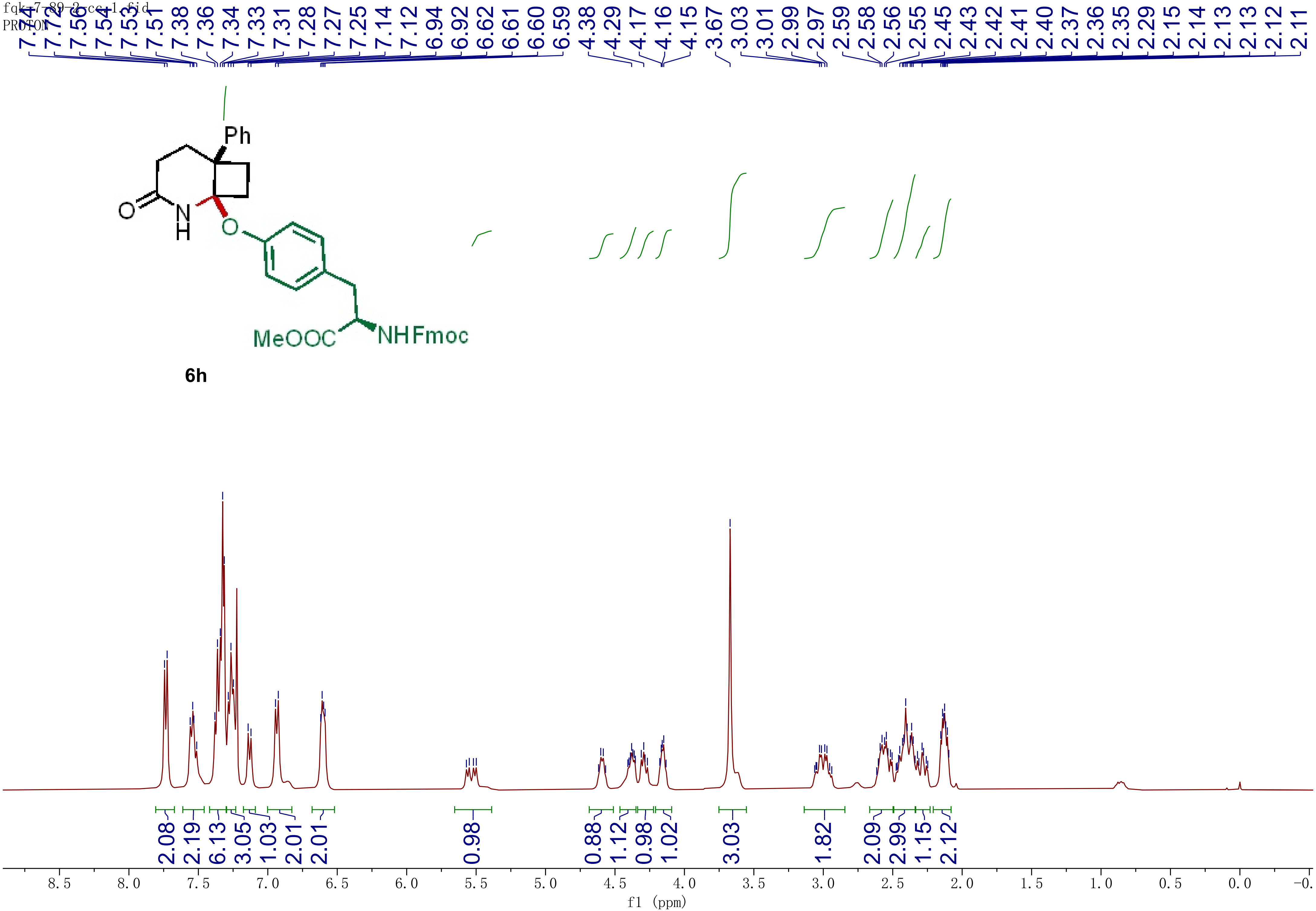

fqk-7-106-3.2.fid
13CPD
173.22
166.53
157.29
142.22
131.52
128.01
127.24
126.61
124.00
117.58
89.36
51.98
50.10
35.42
31.90
28.67
27.44
Ph
O
N
H
O
COOMe
6e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

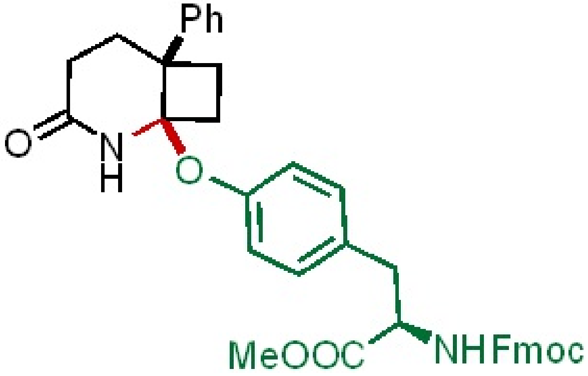

fqk-7-130-5.1.fid
PROTON
8.30 8.09 8.07 7.40 7.38 7.37 7.35 7.35 7.33 7.31 7.31 7.30 7.29 7.28 7.28 7.27 6.77 6.74 2.66 2.65 2.64 2.64 2.63 2.62 2.61 2.60 2.59 2.58 2.57 2.55 2.54 2.53 2.48 2.47 2.46 2.44 2.43 2.42 2.36 2.33 2.32 2.30 2.29 2.27 2.27 2.25 2.24 2.23 2.22 2.21 2.19
Ph
O
N
H
O
NO_2
6f
1.02 2.10 3.98 1.05 2.00 4.14 1.16 3.13
f1 (ppm)


fqk-7-130-5.2.fid
C13CPD
174.04
158.82
142.36
141.81
128.16
127.17
126.87
125.67
117.91
89.71
50.17
35.40
31.98
28.90
27.66
Ph
O
N
H
O
NO_2
6f
f1 (ppm)

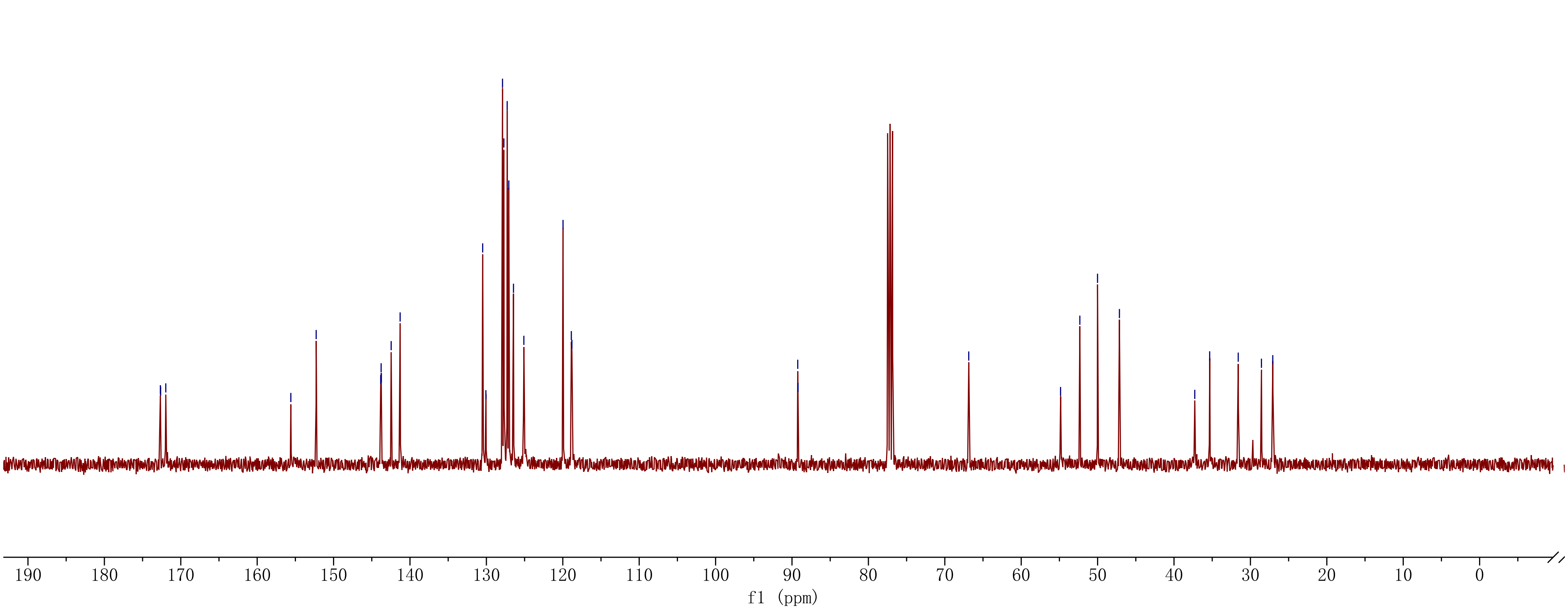

fqk-7-106-1-x_1.fid
PROTON
7.38 7.37 7.35 7.35 7.33 7.27 7.16 7.13 6.86 6.54 6.52 6.47 6.47 2.83 2.82 2.81 2.54 2.53 2.52 2.51 2.51 2.50 2.49 2.48 2.48 2.47 2.46 2.41 2.40 2.36 2.35 2.33 2.23 2.21 2.21 2.20 2.20 2.19 2.16 2.13 2.11 1.96 1.95 1.54 1.52 1.52 1.51 1.50 1.48 1.45 0.90
Ph
O
N
H
O
H
H
H
O
6g
4.26 1.05 1.06 1.00 1.06 1.04 2.10 1.03 4.25 1.10 2.30 2.26 1.91 1.32 2.33 1.20 2.22 2.10 1.24 3.33
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

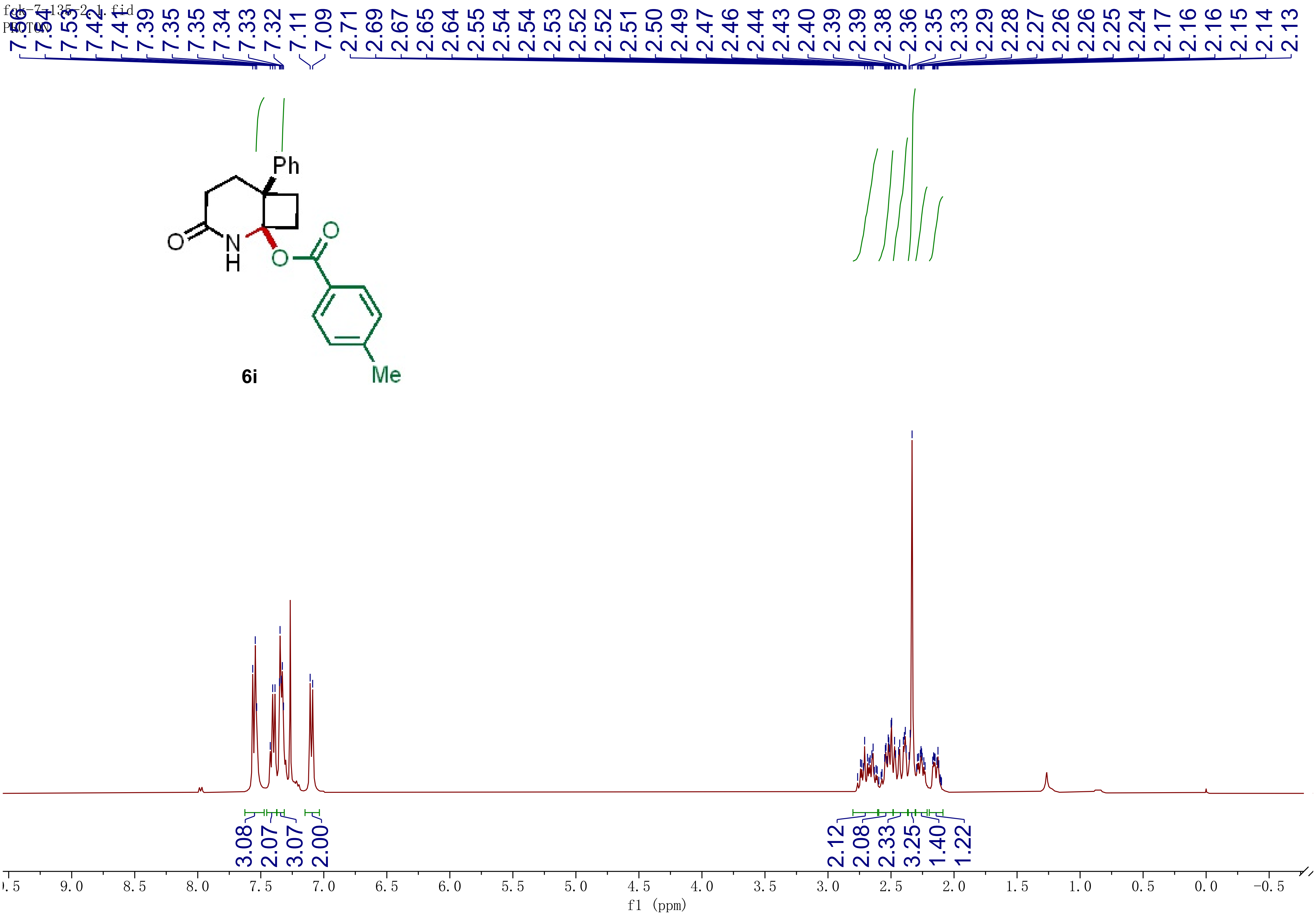

fqk-7-106-1-x.3.fid
C13CPD
220.82
172.45
151.08
142.69
138.36
134.36
128.00
127.40
126.67
126.51
118.97
118.87
116.15
116.09
89.29
89.27
50.50
50.06
48.06
44.05
38.29
35.94
35.49
31.91
31.65
29.64
28.48
27.26
26.50
25.92
21.67
13.94
Ph
O
N
H
H
6g
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2
f1 (ppm)

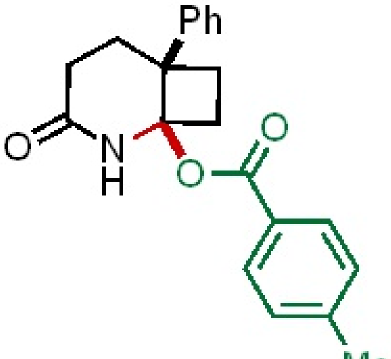

fqk-7-89-2-ce-1.fid
PROTON
7.74 7.72 7.56 7.54 7.53 7.51 7.38 7.36 7.34 7.33 7.31 7.28 7.27 7.25 7.14 7.12 6.94 6.92 6.62 6.61 6.60 6.59 4.38 4.29 4.17 4.16 4.15 3.67 3.03 3.01 2.99 2.97 2.59 2.58 2.56 2.55 2.45 2.43 2.42 2.41 2.40 2.37 2.36 2.35 2.29 2.15 2.14 2.13 2.13 2.12 2.11
Ph
O
N
H
O
MeOOC
NHFmoc
6h
2.08 2.19 6.13 3.05 1.03 2.01 2.01 0.98 0.88 1.12 0.98 1.02 3.03 1.82 2.09 2.99 1.15 2.12
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)


Ph
O
N
H
O
MeOOC
NHFmoc

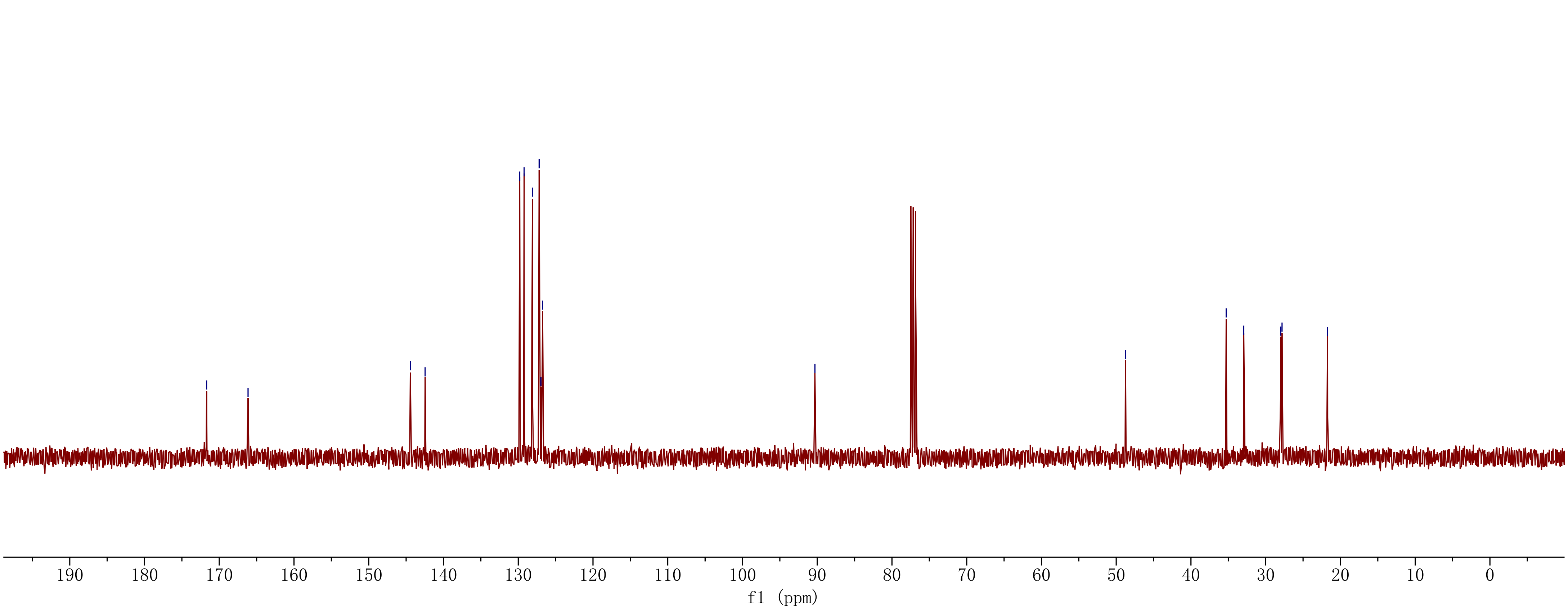
6h

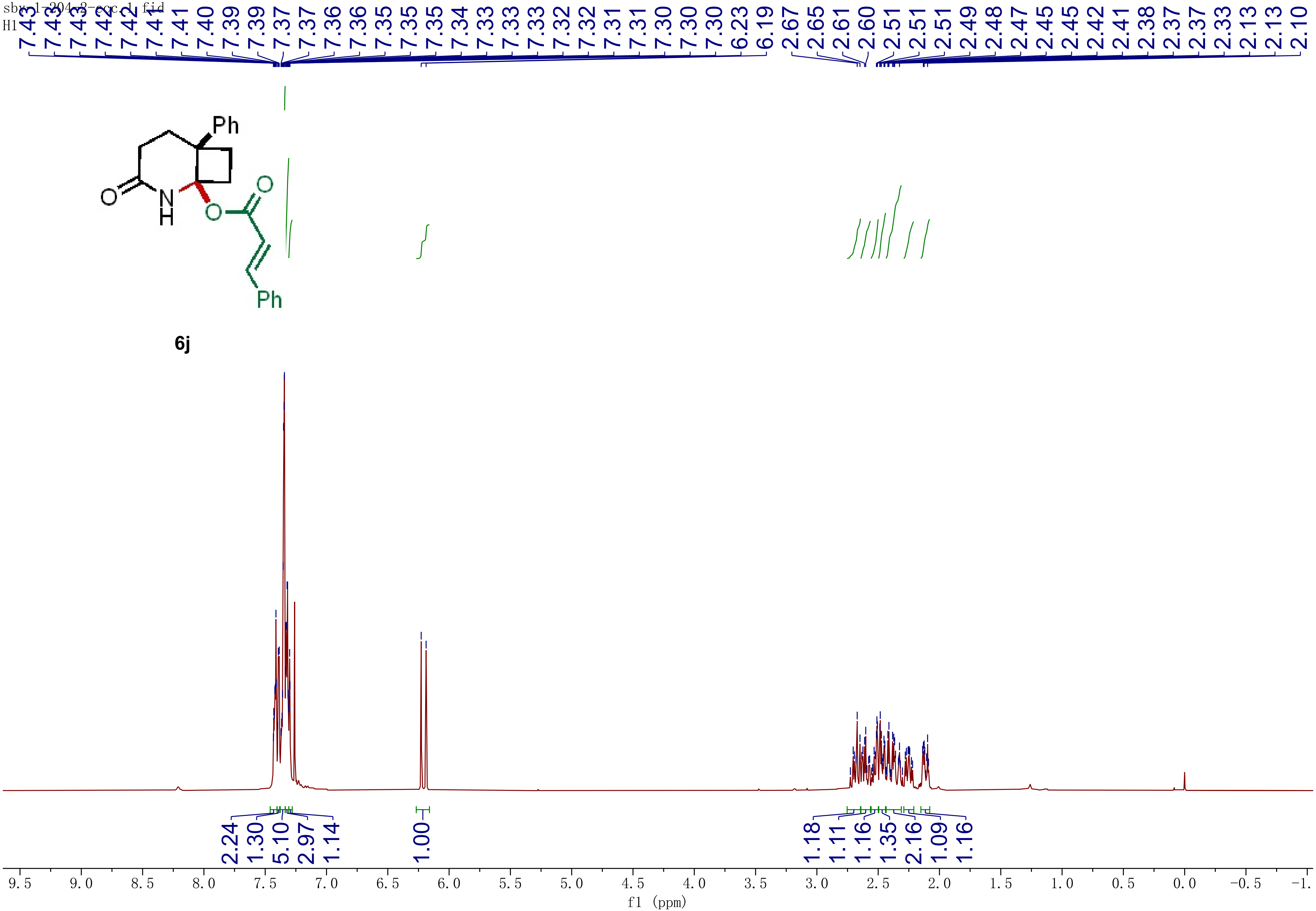

fqk-7-89-2-cq 2.fid
C13CPD
172.67
172.64
171.96
155.59
152.28
143.83
143.78
143.75
142.46
141.29
130.49
130.07
127.89
127.71
127.28
127.06
126.45
125.10
119.97
118.88
118.81
89.25
89.21
66.87
54.85
52.33
50.01
47.14
37.28
35.33
31.59
28.56
27.08
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

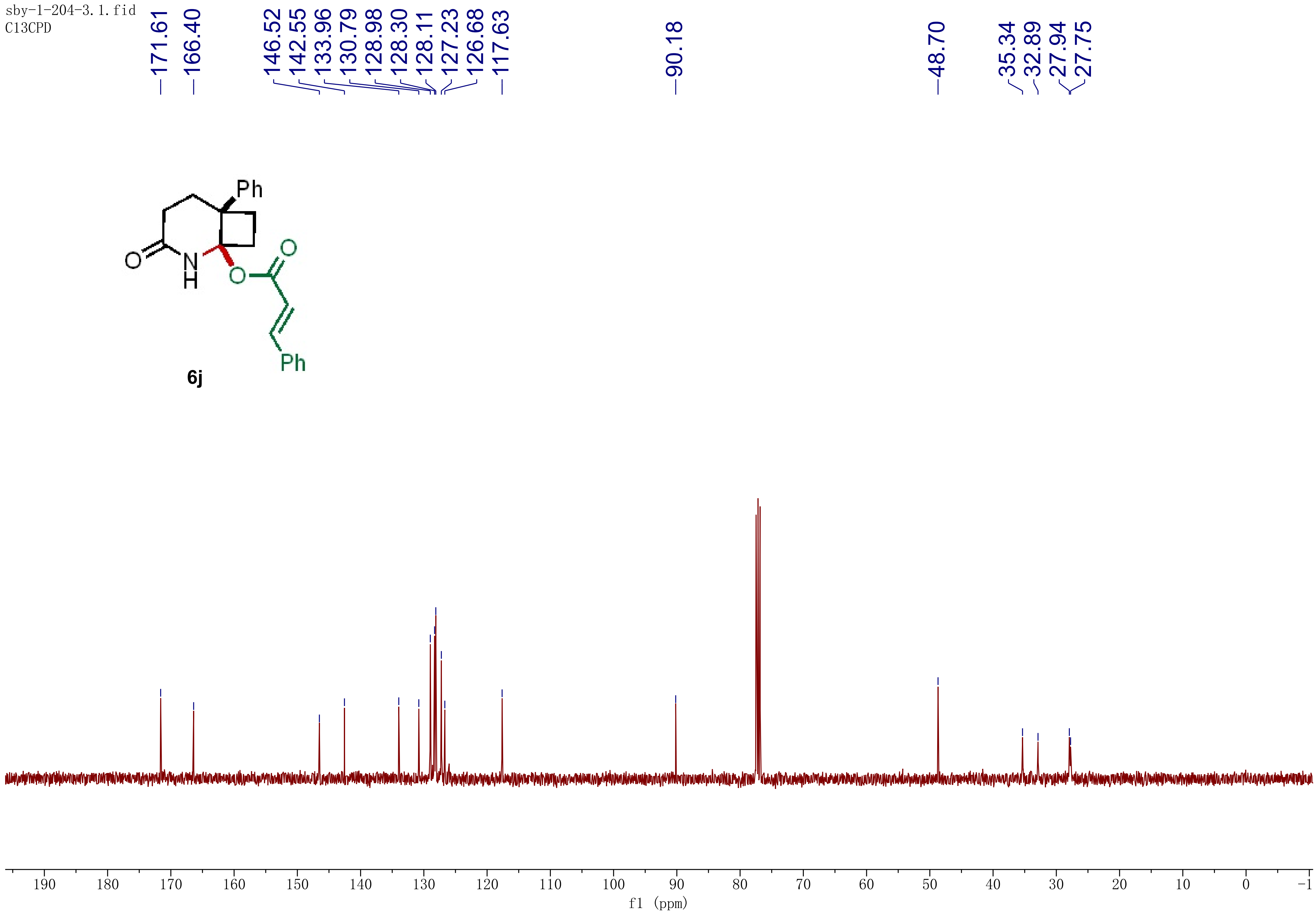

fqk-7-135-2.1.fid
PROTON
7.56 7.54 7.53 7.42 7.41 7.39 7.35 7.35 7.34 7.33 7.32 7.11 7.09 2.71 2.69 2.67 2.65 2.64 2.55 2.54 2.54 2.53 2.52 2.52 2.51 2.50 2.49 2.47 2.46 2.44 2.43 2.40 2.39 2.39 2.38 2.36 2.35 2.33 2.29 2.28 2.27 2.26 2.26 2.25 2.24 2.17 2.16 2.16 2.15 2.14 2.13
Ph
O
N
H
O
O
Me
6i
3.08 2.07 3.07 2.00
2.12 2.08 2.33 3.25 1.40 1.22
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

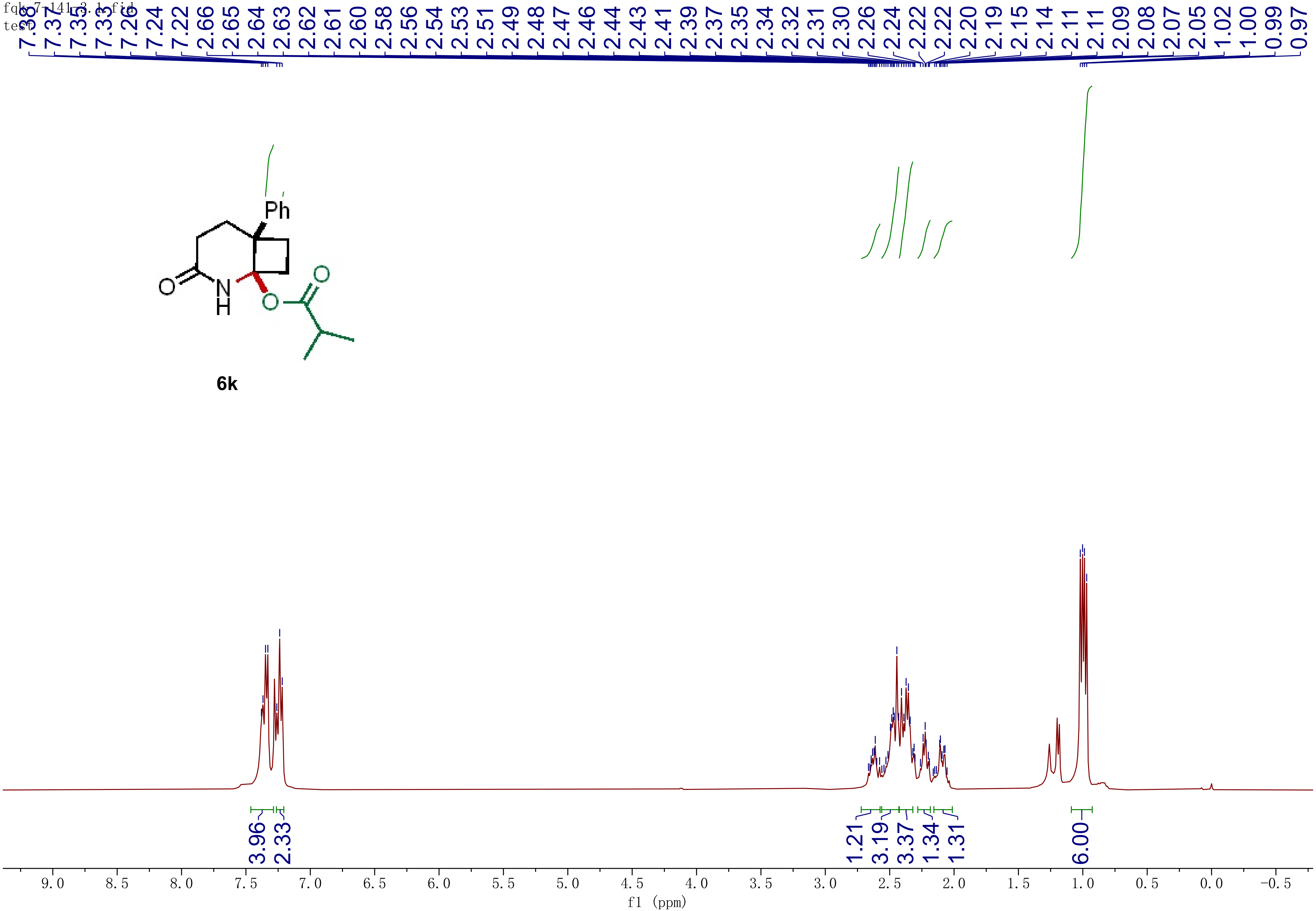

Ph
O
N
H
O
O
Me

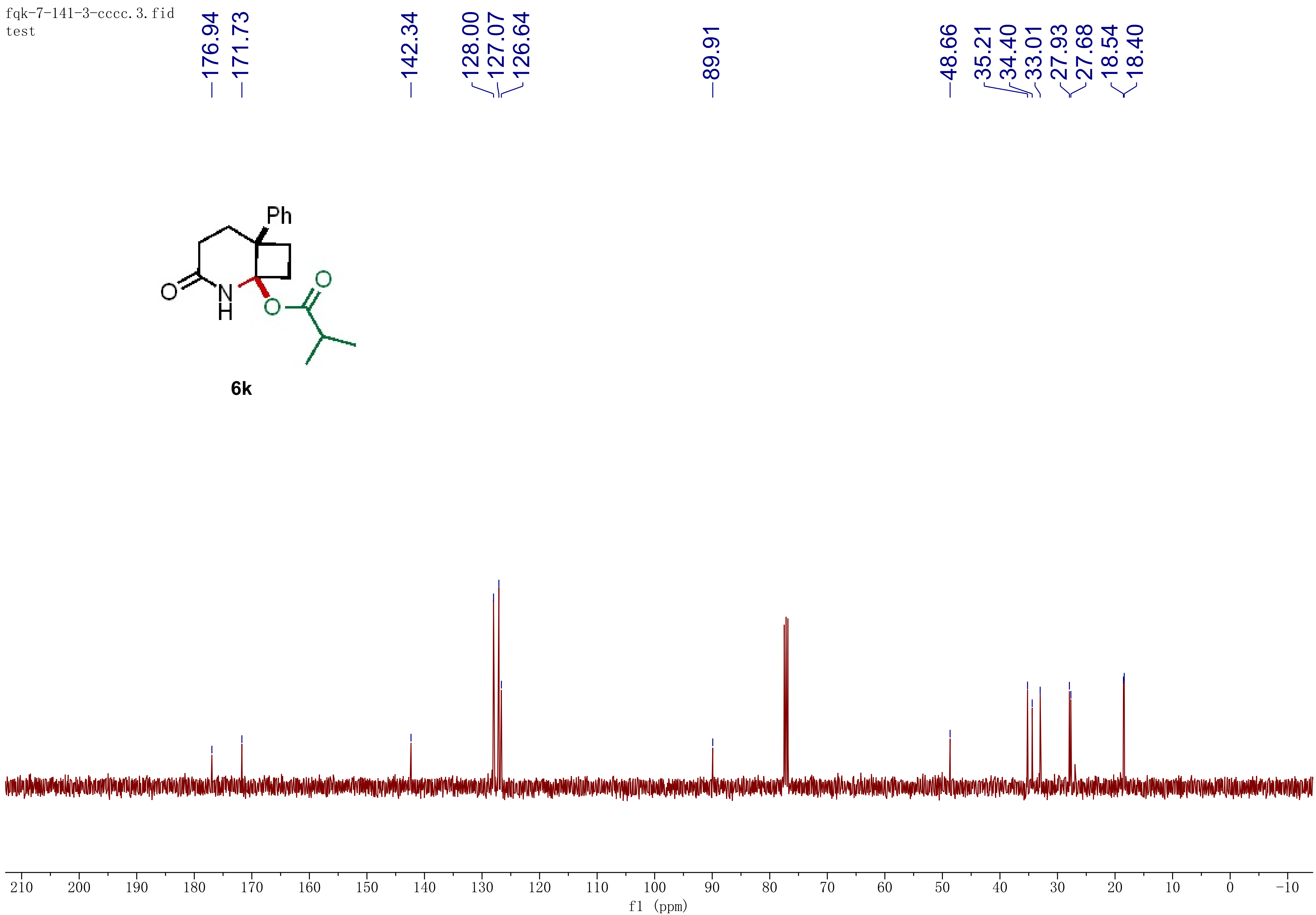
6i

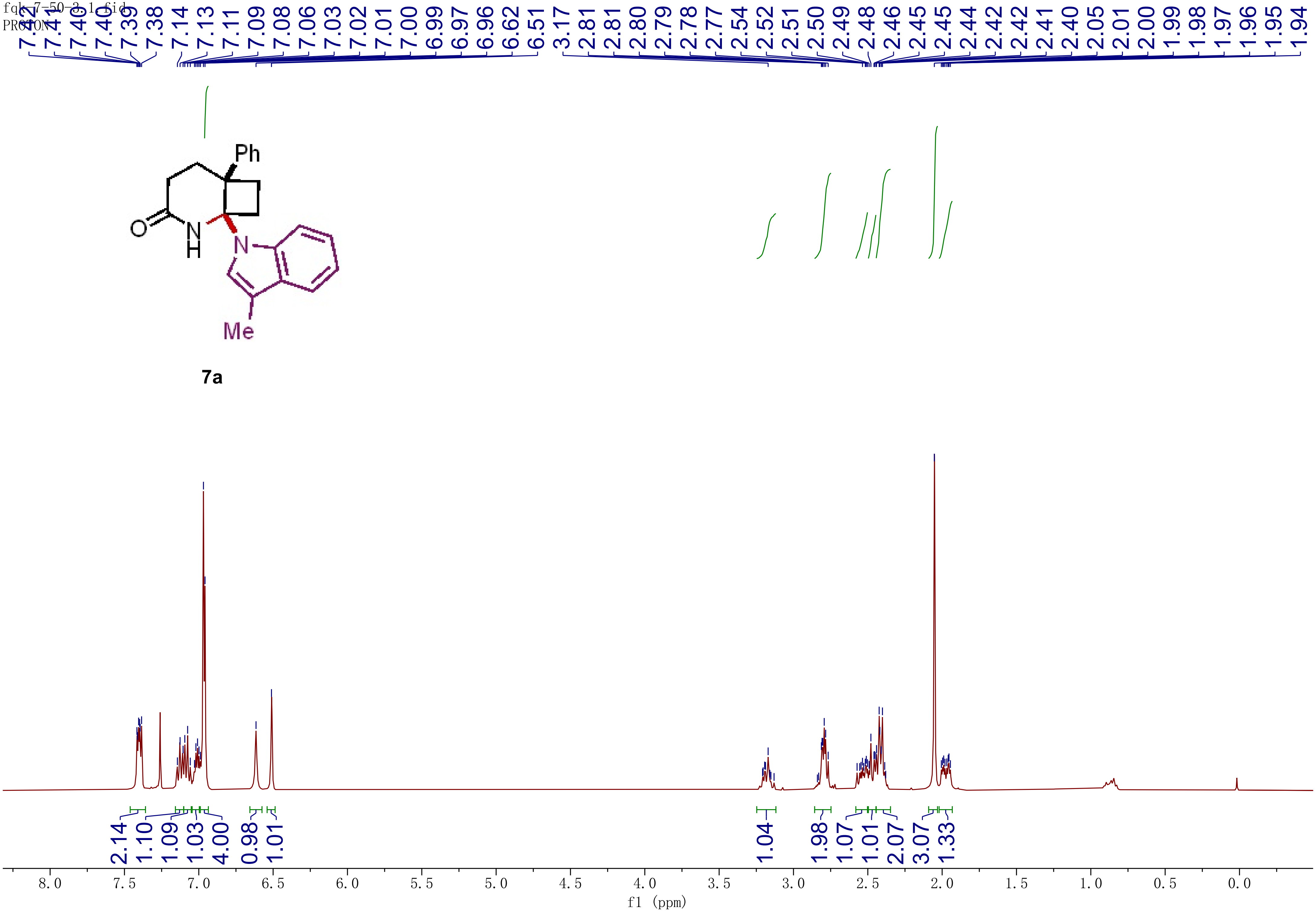

fqk-7-135-2-cc.2.fid
C13CPD
171.70
166.15
144.44
142.46
129.80
129.21
128.09
127.20
126.97
126.74
90.31
48.76
35.29
32.93
27.99
27.81
21.72
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


sby-1-204-2-ccc.1.fid
H1
7.43 7.43 7.43 7.42 7.42 7.41 7.41 7.40 7.39 7.39 7.37 7.37 7.36 7.36 7.35 7.35 7.35 7.34 7.33 7.33 7.33 7.32 7.32 7.31 7.31 7.30 7.30 7.30 6.23 6.19 2.67 2.65 2.61 2.60 2.51 2.51 2.51 2.49 2.48 2.47 2.45 2.45 2.42 2.41 2.38 2.37 2.37 2.33 2.13 2.13 2.10
Ph
O
N
H
O
O
Ph
6j
2.24 1.30 5.10 2.97 1.14
1.00
1.18 1.11 1.16 1.35 2.16 1.09 1.16
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

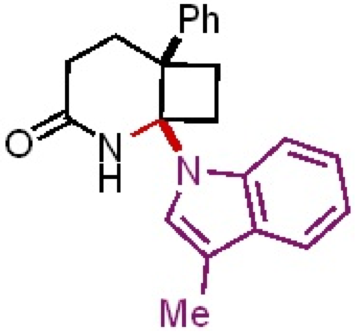

sby-1-204-3.1.fid
C13CPD
171.61
166.40
146.52
142.55
133.96
130.79
128.98
128.30
128.11
127.23
126.68
117.63
90.18
48.70
35.34
32.89
27.94
27.75
Ph
O
N
H
O
O
Ph
6j
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −1
f1 (ppm)


fqk-7-141-3.1.fid
test
7.38 7.37 7.35 7.33 7.26 7.24 7.22 2.66 2.65 2.64 2.63 2.62 2.61 2.60 2.58 2.56 2.54 2.53 2.51 2.49 2.48 2.47 2.46 2.44 2.43 2.41 2.39 2.37 2.35 2.34 2.32 2.31 2.30 2.26 2.24 2.22 2.22 2.20 2.19 2.15 2.14 2.11 2.11 2.09 2.08 2.07 2.05 1.02 1.00 0.99 0.97
Ph
O
N
H
O
O
6k
3.96
2.33
1.21
3.19
3.37
1.34
1.31
6.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

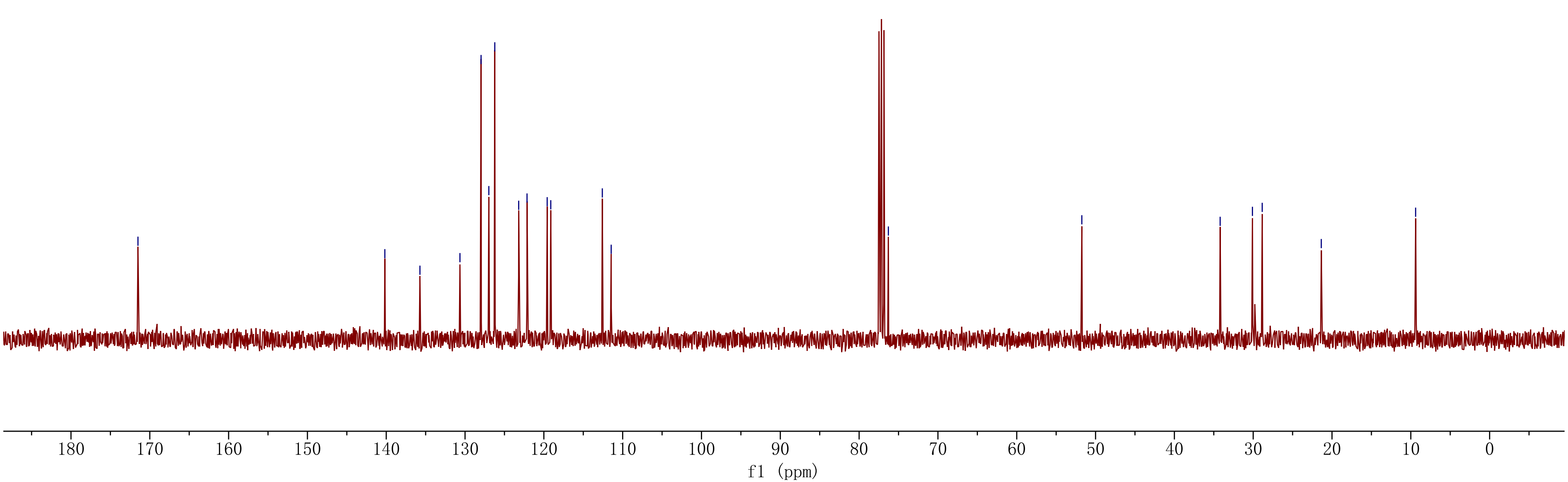

fqk-7-141-3-cccc.3.fid
test
176.94
171.73
142.34
128.00
127.07
126.64
89.91
48.66
35.21
34.40
33.01
27.93
27.68
18.54
18.40
Ph
O
N
H
O
O
6k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

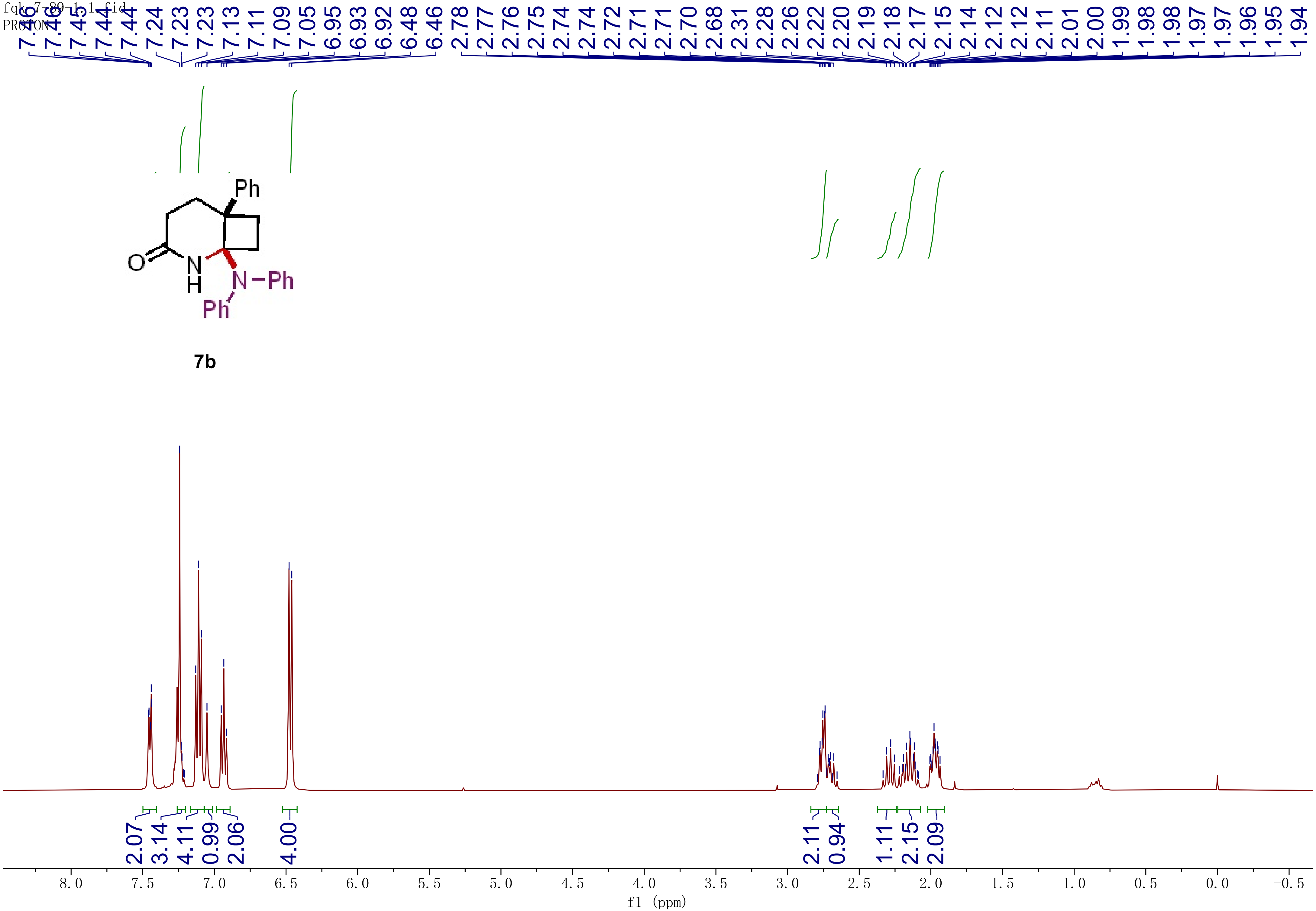

fqk-7-50-3.1.fid
PROTON
7.42 7.41 7.40 7.40 7.39 7.38 7.14 7.13 7.11 7.09 7.08 7.06 7.03 7.02 7.01 7.00 6.99 6.97 6.96 6.62 6.51 3.17 2.81 2.81 2.80 2.79 2.78 2.77 2.54 2.52 2.51 2.50 2.49 2.48 2.46 2.45 2.45 2.44 2.42 2.42 2.41 2.40 2.05 2.01 2.00 1.99 1.98 1.97 1.96 1.95 1.94
Ph
O
N
H
N
Me
7a
2.14 1.10 1.09 1.03 4.00 0.98 1.01 1.04 1.98 1.07 1.01 2.07 3.07 1.33
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

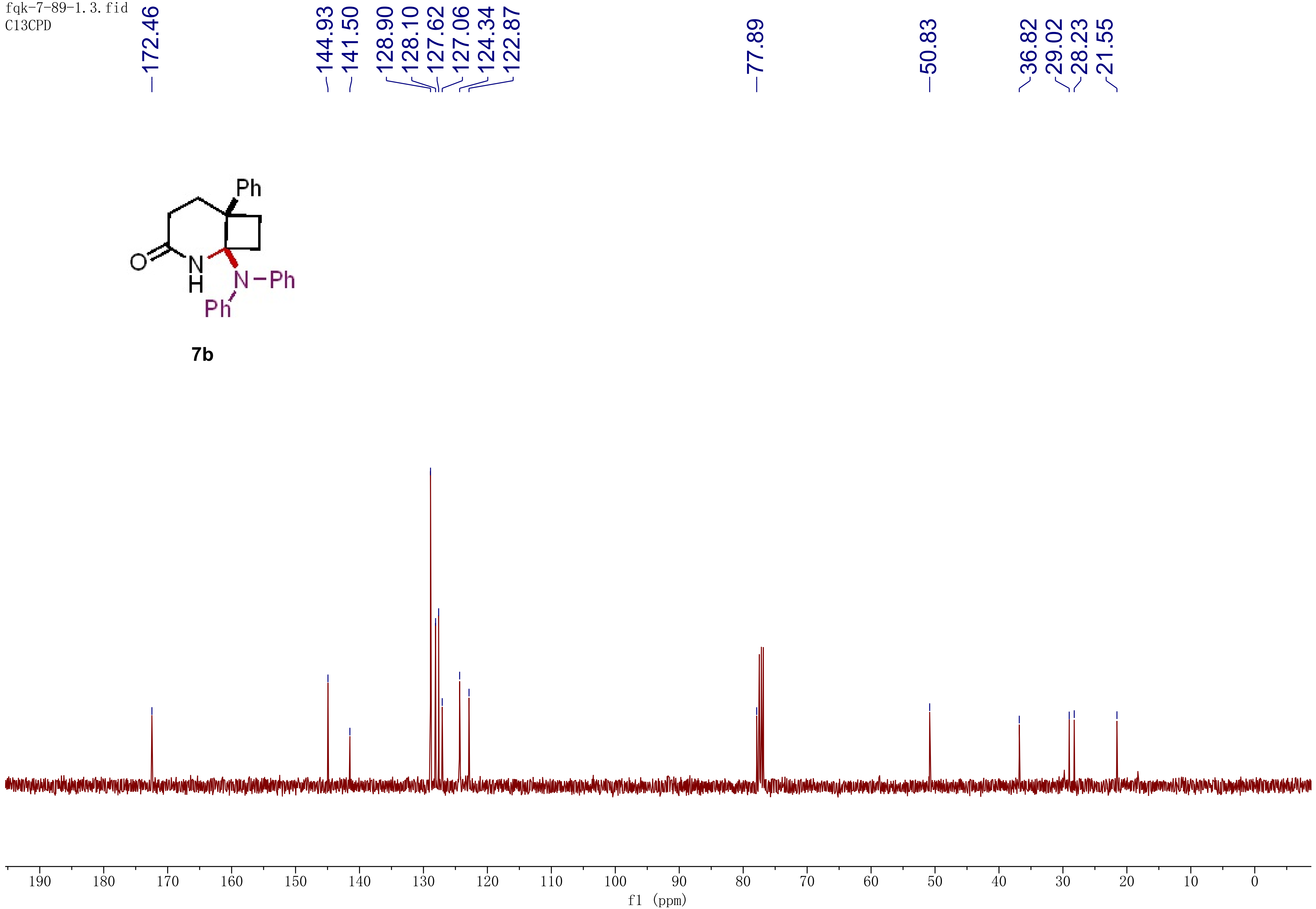
fqk-7-50-3.2.fid
C13CPD

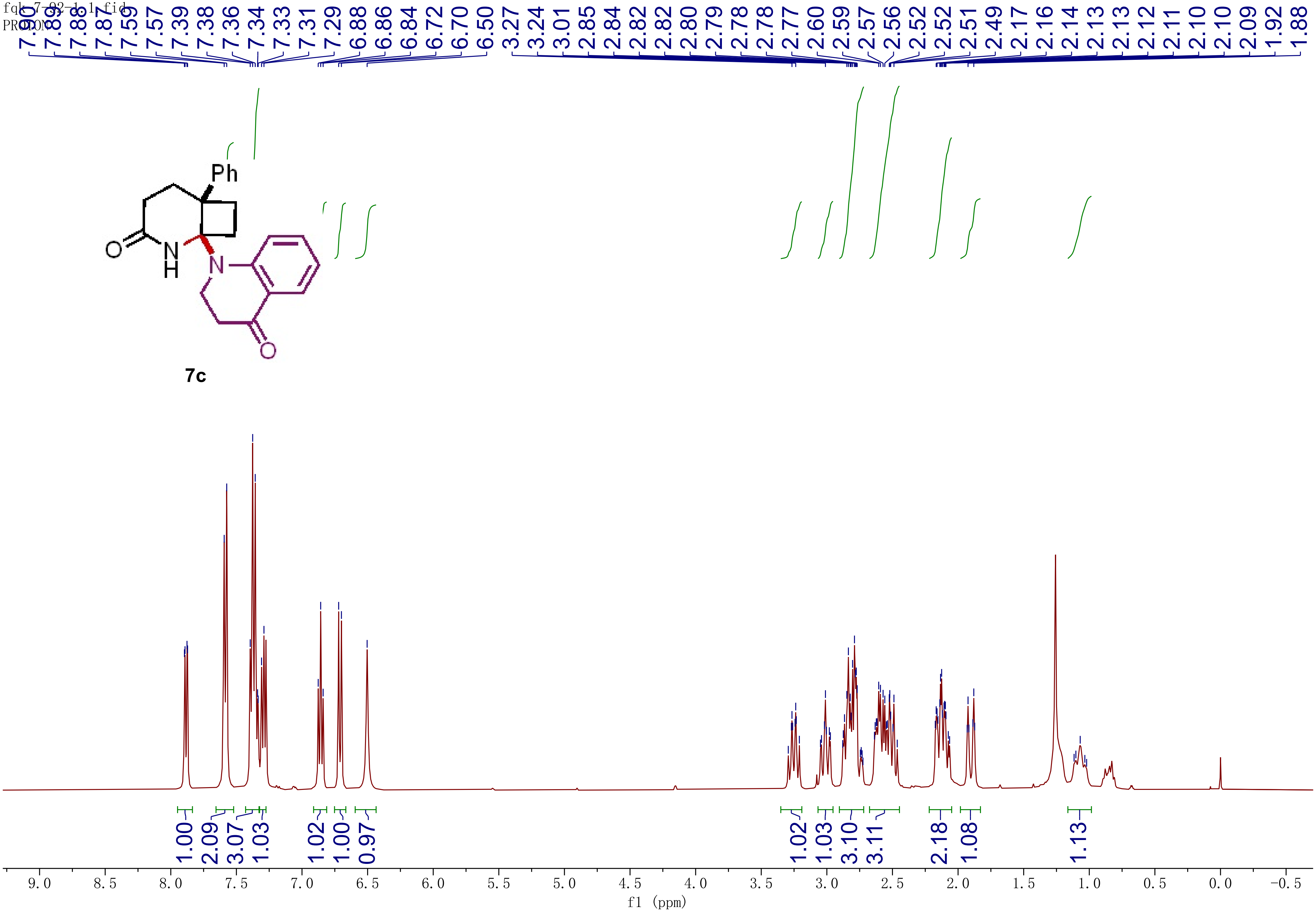


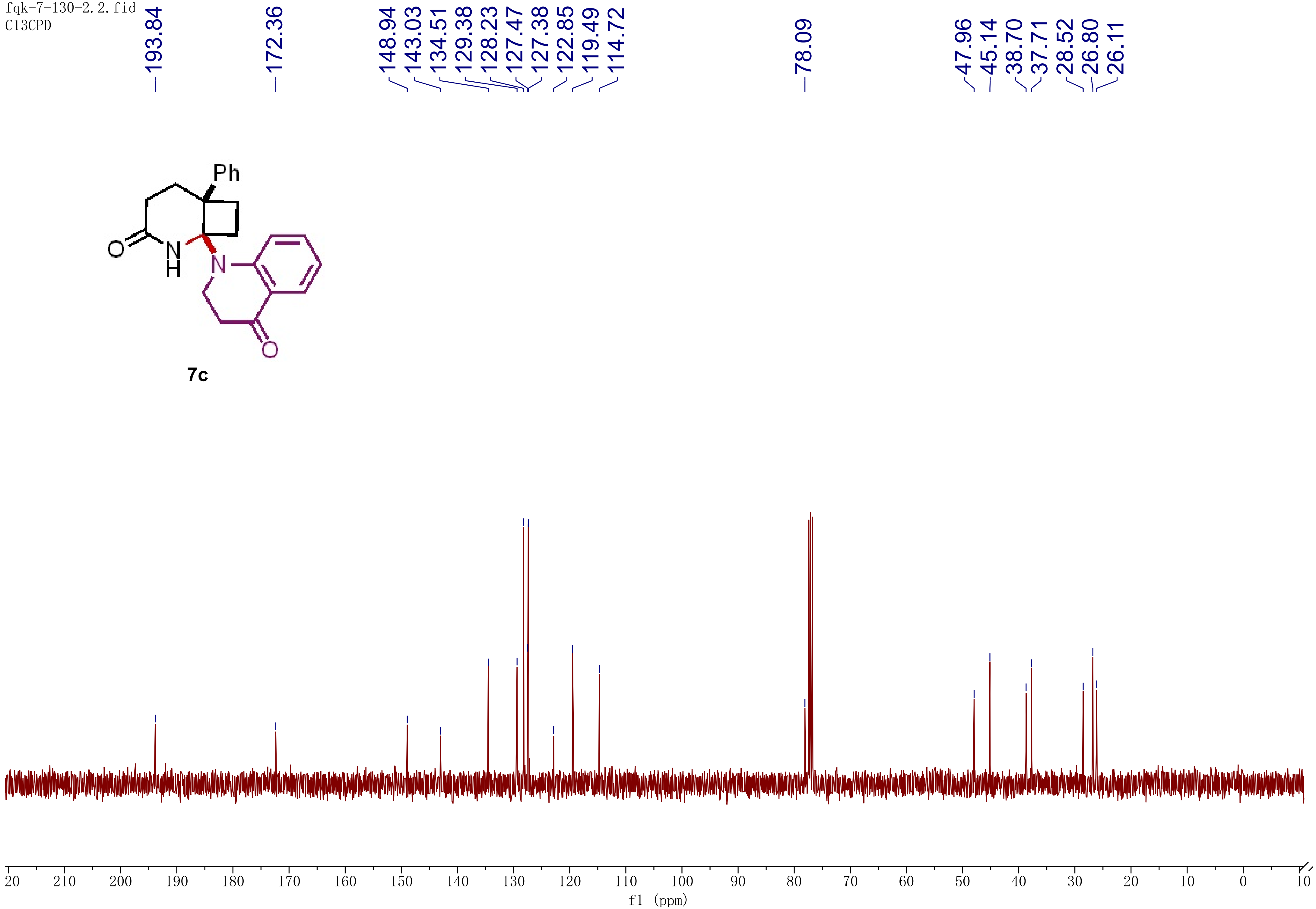
7a



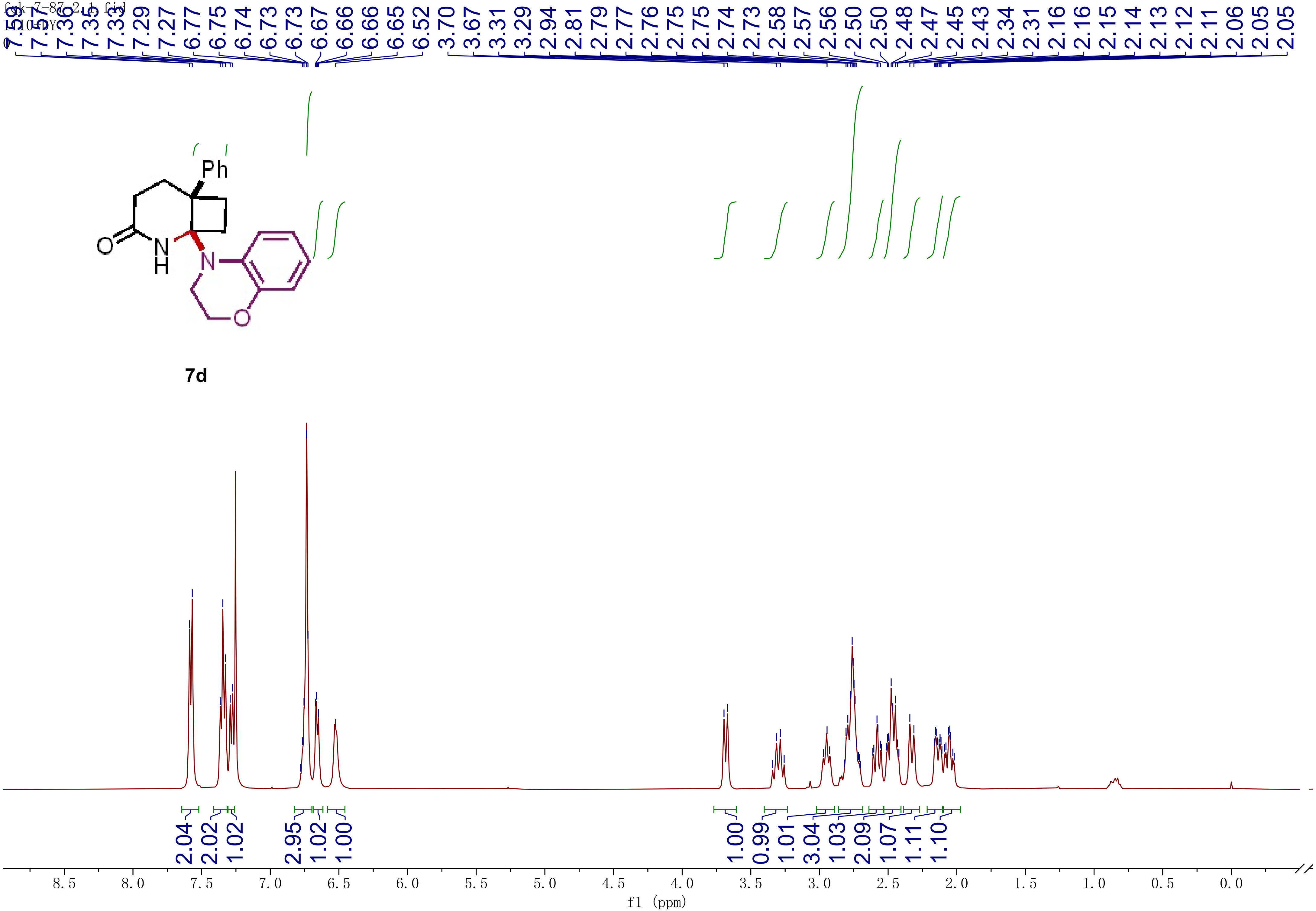

fqk-7-89-1.1.fid
PROTON
7.45
7.44
7.44
7.24
7.23
7.23
7.13
7.11
7.09
7.05
6.95
6.93
6.92
6.48
6.46
2.78
2.77
2.76
2.75
2.74
2.74
2.72
2.71
2.71
2.70
2.68
2.31
2.28
2.26
2.22
2.20
2.19
2.18
2.17
2.15
2.14
2.12
2.12
2.11
2.01
2.00
1.99
1.98
1.98
1.97
1.97
1.96
1.95
1.94
Ph
O
N
H
N−Ph
Ph
7b
2.07
3.14
4.11
0.99
2.06
4.00
2.11
0.94
1.11
2.15
2.09
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


fqk-7-89-1.3.fid
C13CPD
172.46
144.93
141.50
128.90
128.10
127.62
127.06
124.34
122.87
77.89
50.83
36.82
29.02
28.23
21.55
Ph
O
N
H
N-Ph
Ph
7b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


fqk-7-92-1.1.fid
PROTON
7.90
7.89
7.88
7.87
7.59
7.57
7.39
7.38
7.36
7.34
7.33
7.31
7.29
6.88
6.86
6.84
6.72
6.70
6.50
3.27
3.24
3.01
2.85
2.84
2.82
2.82
2.80
2.79
2.78
2.78
2.77
2.60
2.59
2.57
2.56
2.52
2.52
2.51
2.49
2.17
2.16
2.14
2.13
2.13
2.12
2.11
2.10
2.10
2.09
1.92
1.88
Ph
O
N
H
N
O
7c
1.00
2.09
3.07
1.03
1.02
1.00
0.97
1.02
1.03
3.10
3.11
2.18
1.08
1.13
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

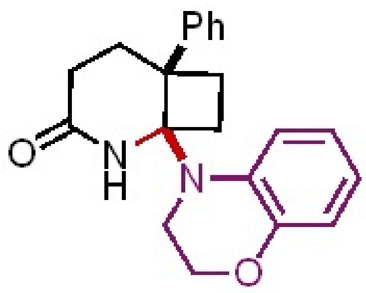

fqk-7-130-2.2.fid
C13CPD
193.84
172.36
148.94
143.03
134.51
129.38
128.23
127.47
127.38
122.85
119.49
114.72
78.09
47.96
45.14
38.70
37.71
28.52
26.80
26.11
f1 (ppm)
Ph
O
NH
N
O

7c

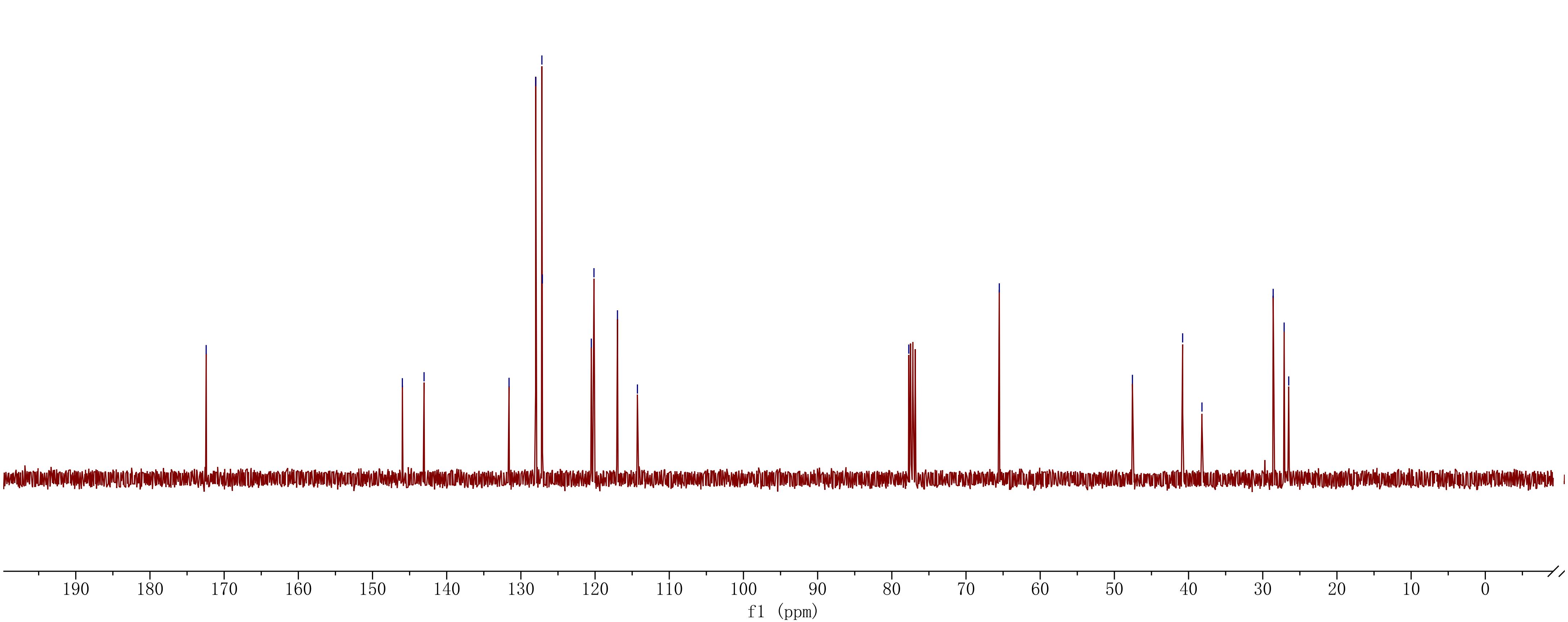


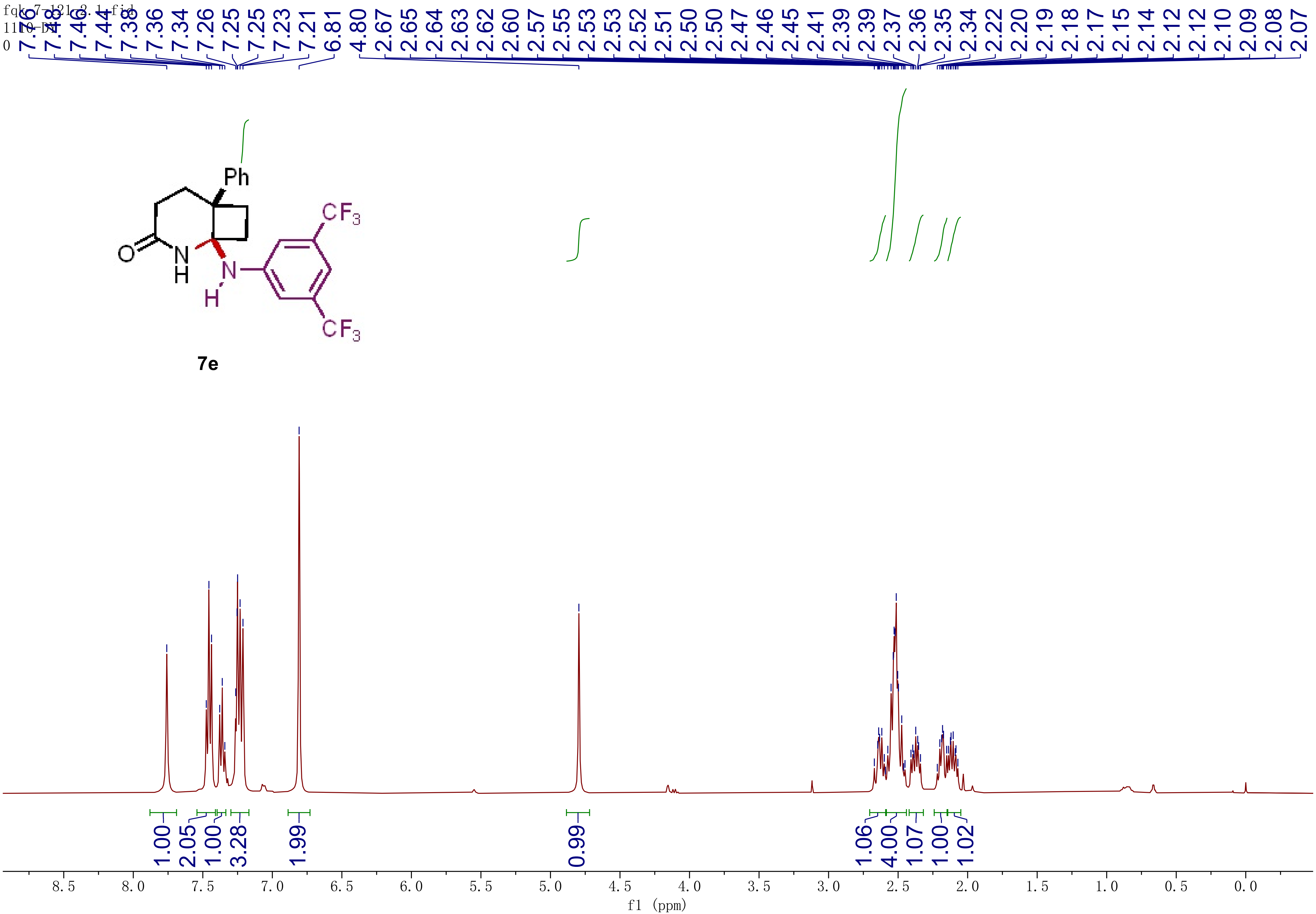
7d

fqk-7-87-2.2.fid
13CPD

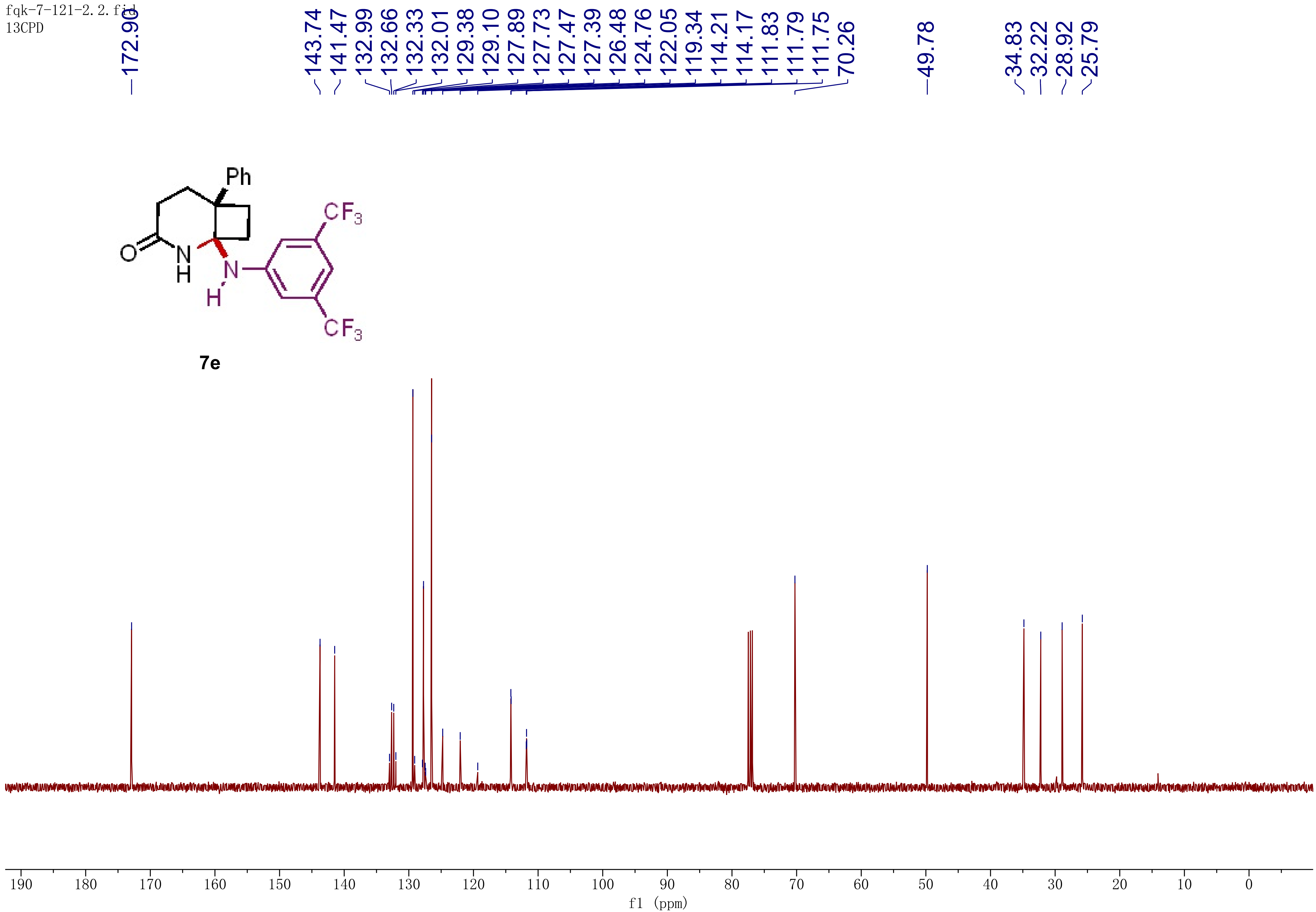


7d

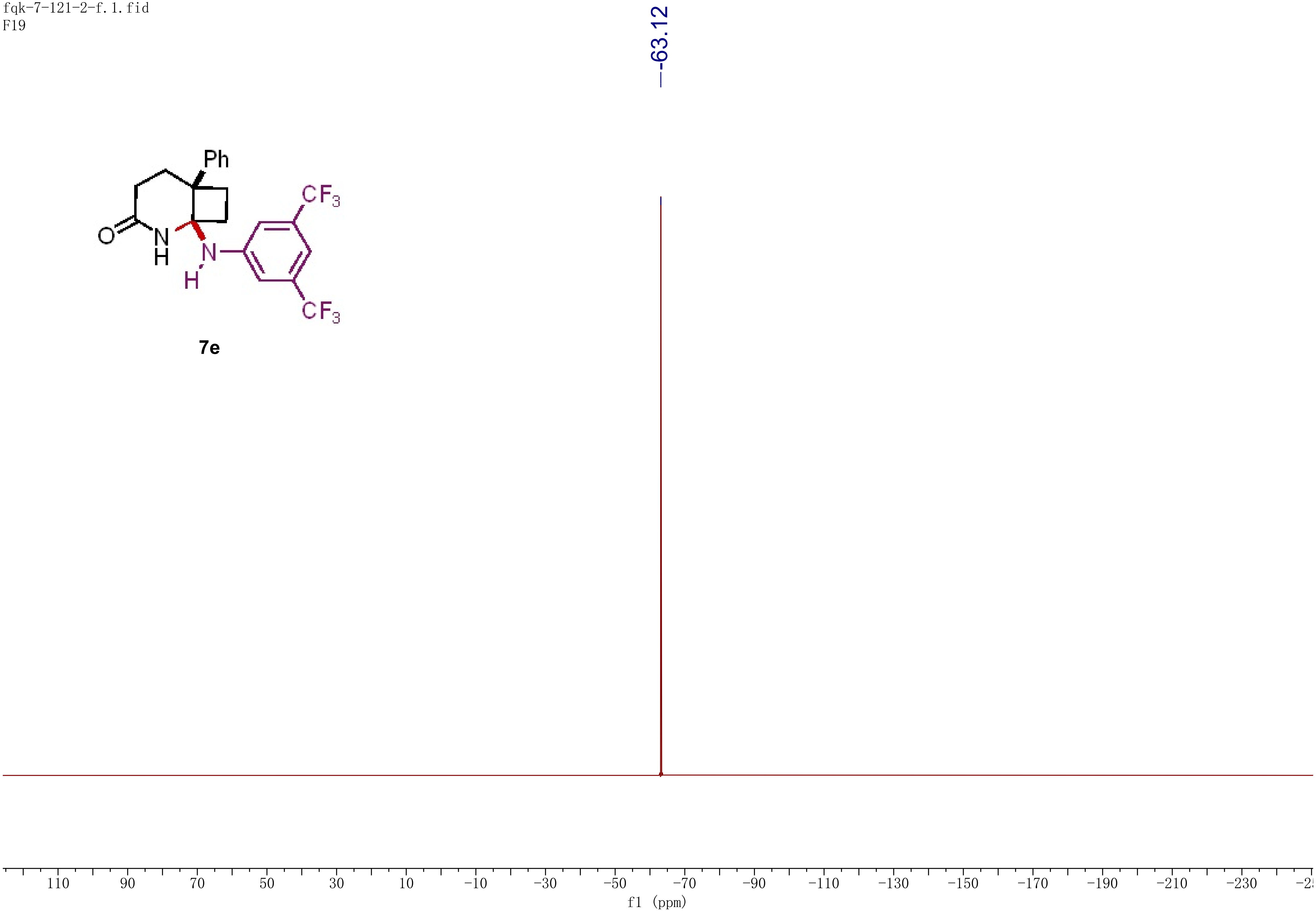


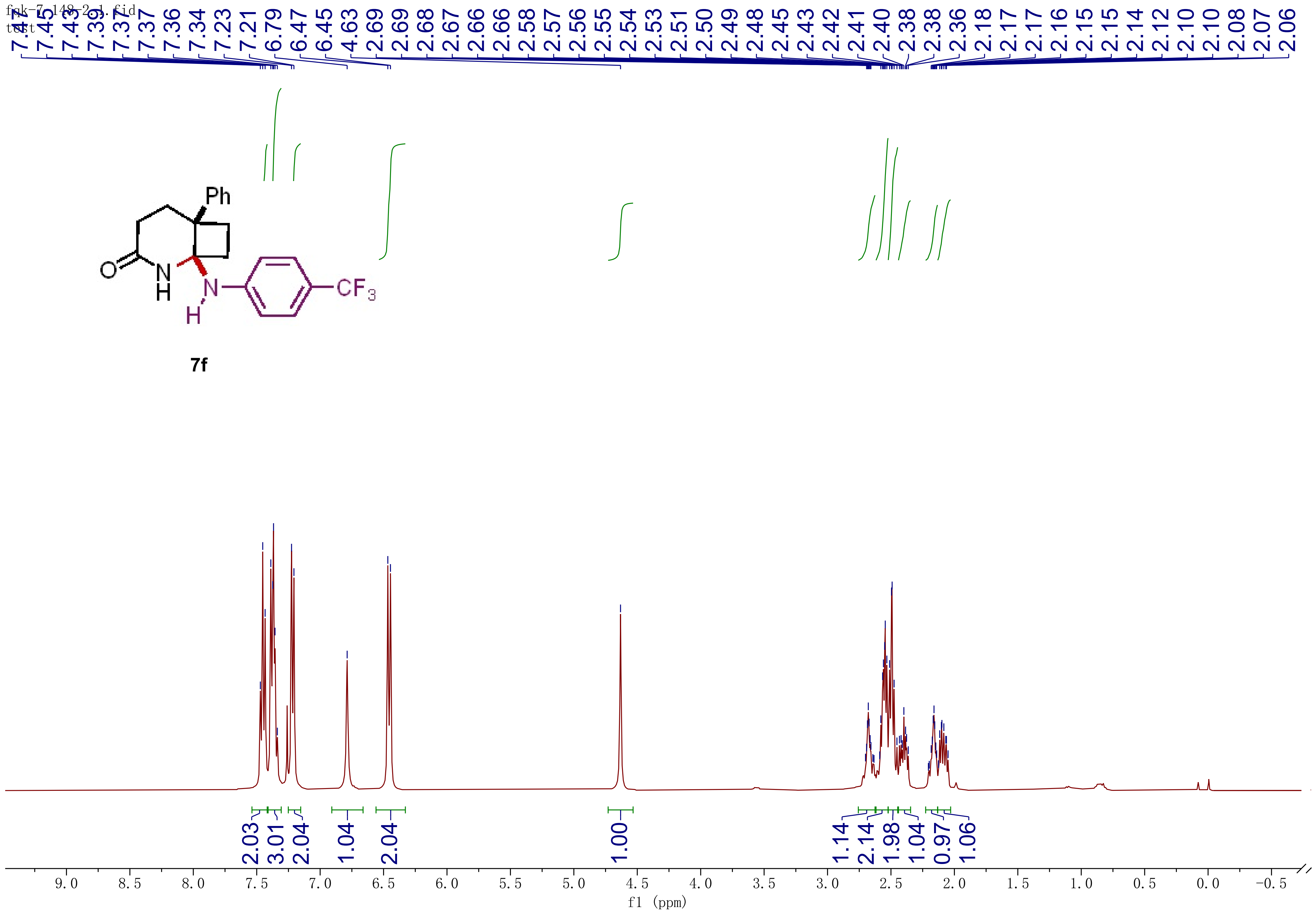


7.76 7.48 7.46 7.44 7.38 7.36 7.34 7.26 7.25 7.25 7.23 7.21 6.81 4.80 2.67 2.65 2.64 2.63 2.62 2.60 2.57 2.55 2.53 2.53 2.52 2.51 2.50 2.50 2.47 2.46 2.45 2.41 2.39 2.39 2.37 2.36 2.35 2.34 2.22 2.20 2.19 2.18 2.17 2.15 2.14 2.12 2.12 2.10 2.09 2.08 2.07
Ph
CF3
O
N
H
N
H
CF3
7e
1.00 2.05 1.00 3.28 1.99 0.99 1.06 4.00 1.07 1.00 1.02
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

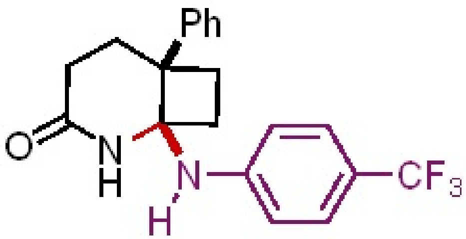

fqk-7-121-2.2.fid
13CPD
172.90
143.74
141.47
132.99
132.66
132.33
132.01
129.38
129.10
127.89
127.73
127.47
127.39
126.48
124.76
122.05
119.34
114.21
114.17
111.83
111.79
111.75
70.26
49.78
34.83
32.22
28.92
25.79
Ph
CF3
O
N
H
N
H
CF3
7e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

fqk-7-121-2-f.1.fid
F19

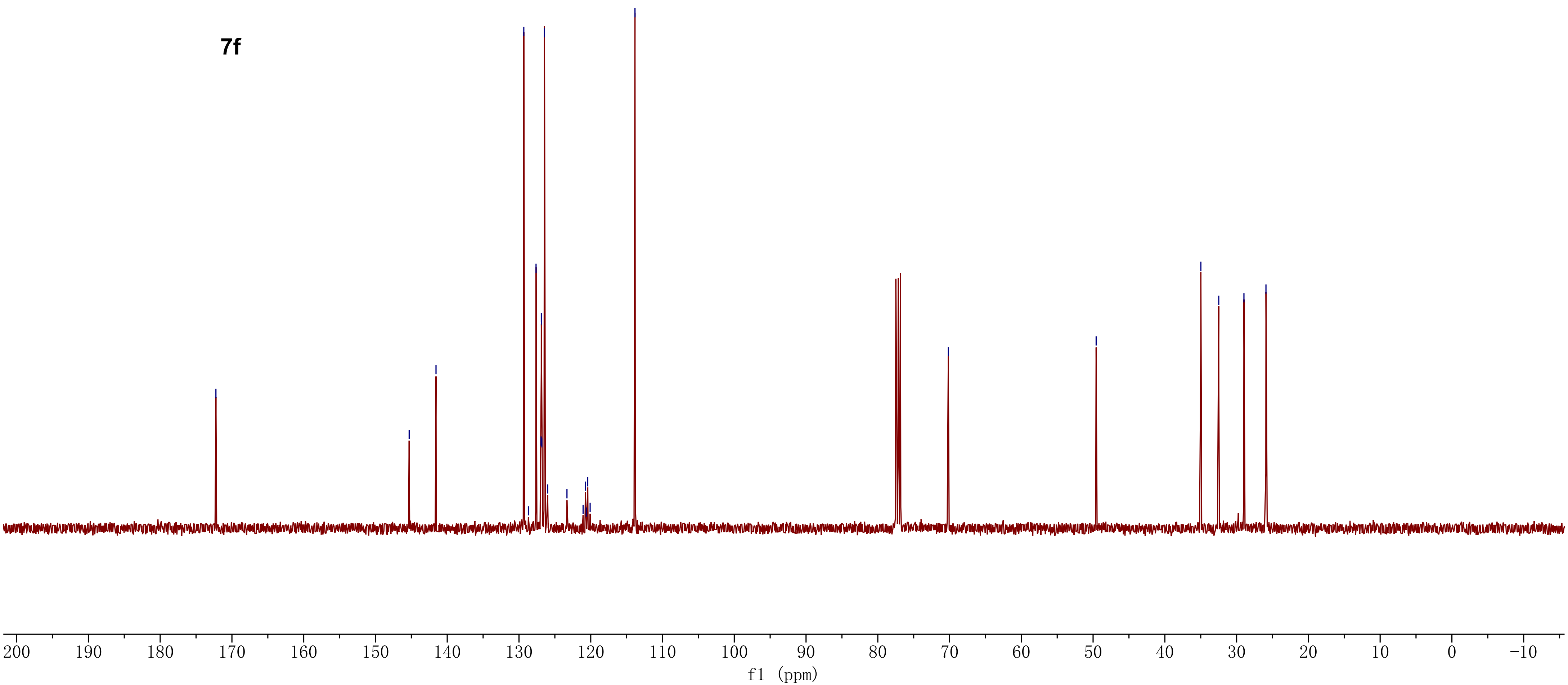


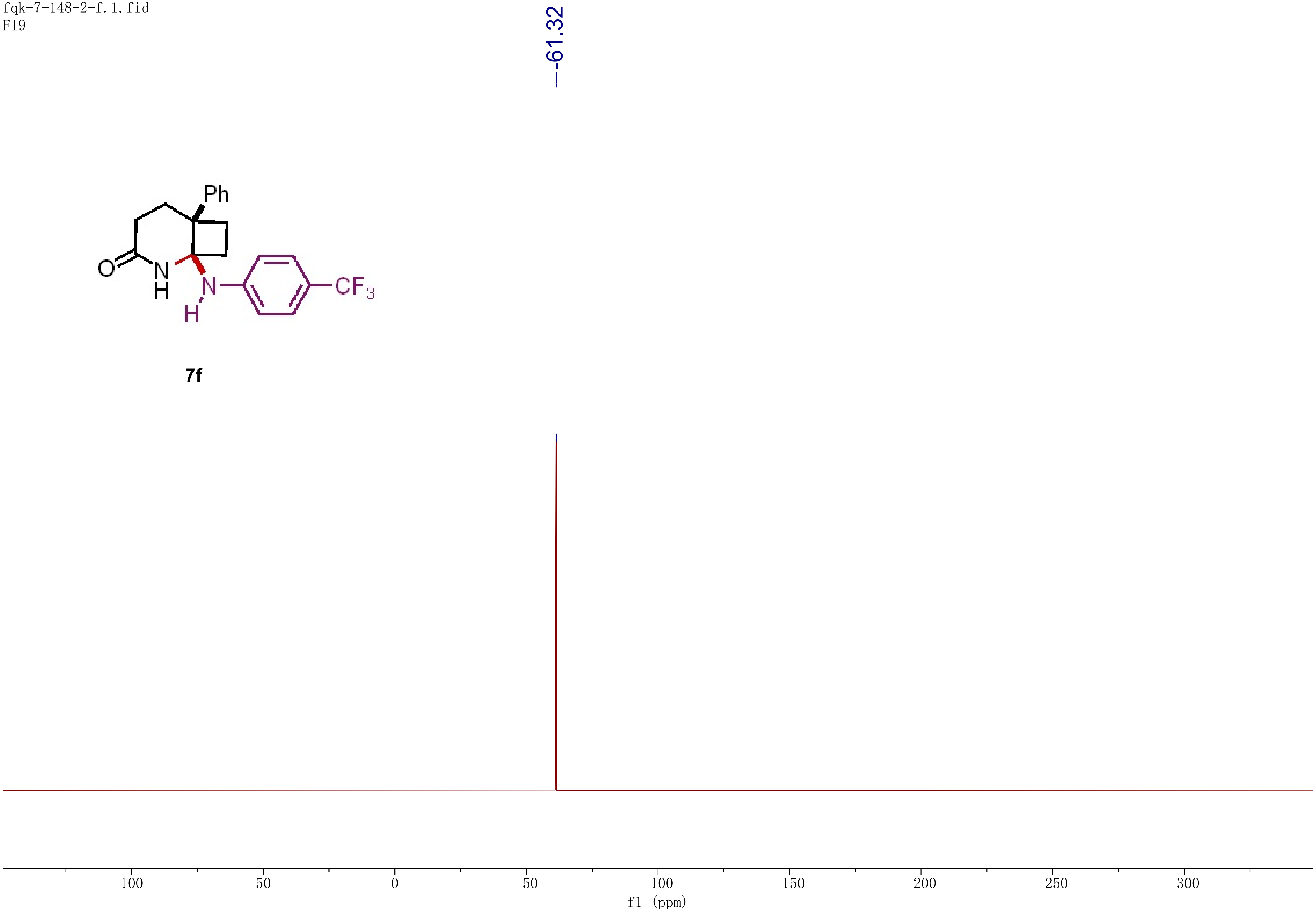

fpk-7-148-2.1.fid
test
7.47 7.45 7.43 7.39 7.37 7.37 7.36 7.34 7.23 7.21 6.79 6.47 6.45 4.63 2.69 2.69 2.68 2.67 2.66 2.66 2.58 2.57 2.56 2.55 2.54 2.53 2.51 2.50 2.49 2.48 2.45 2.43 2.42 2.41 2.40 2.38 2.38 2.36 2.18 2.17 2.17 2.16 2.15 2.15 2.14 2.12 2.10 2.10 2.08 2.07 2.06
Ph
O
N
H
N
H
CF3
7f
2.03 3.01 2.04 1.04 2.04 1.00 1.14 2.14 1.98 1.04 0.97 1.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

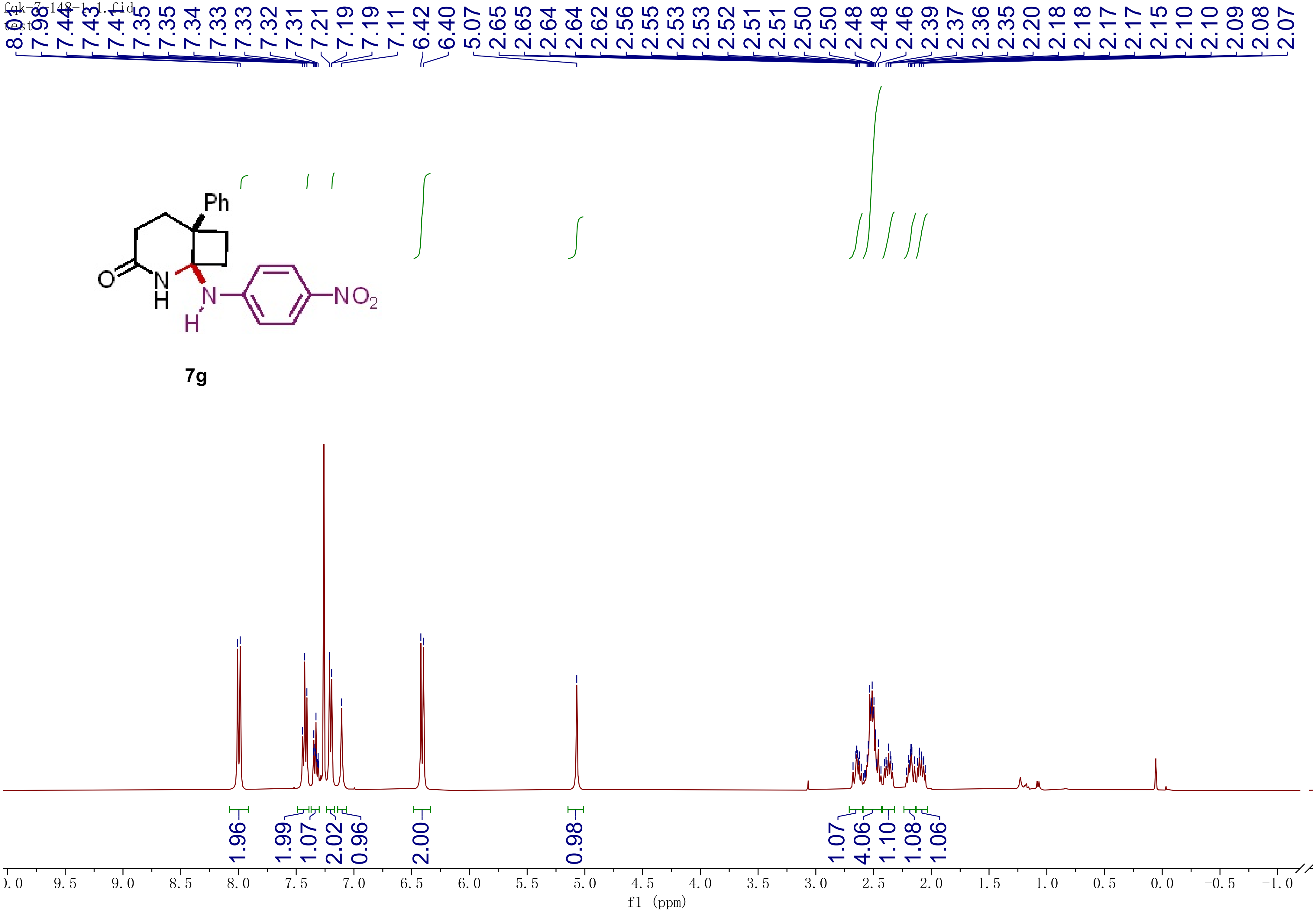
fqk-7-148-2.2.fid
test

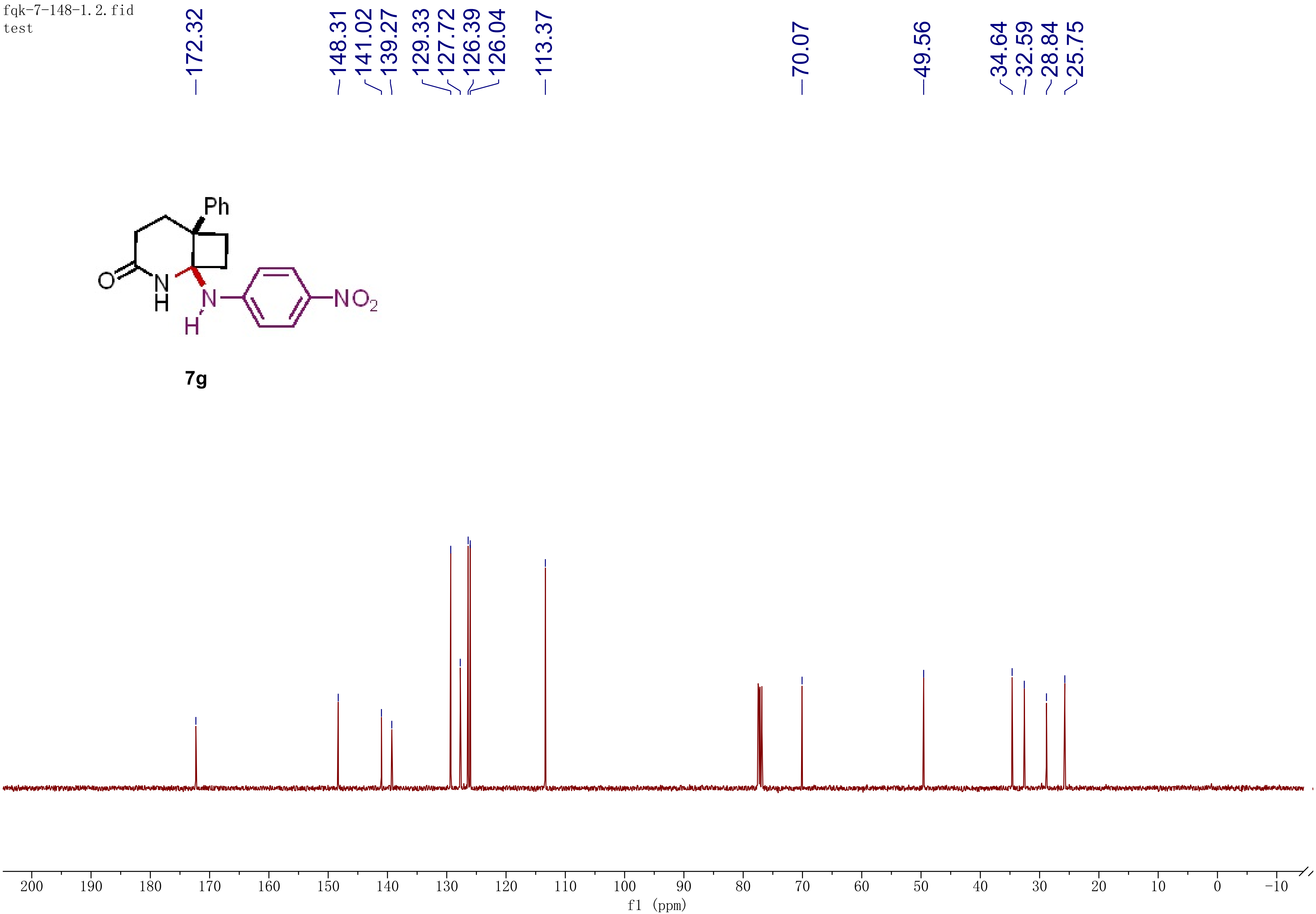


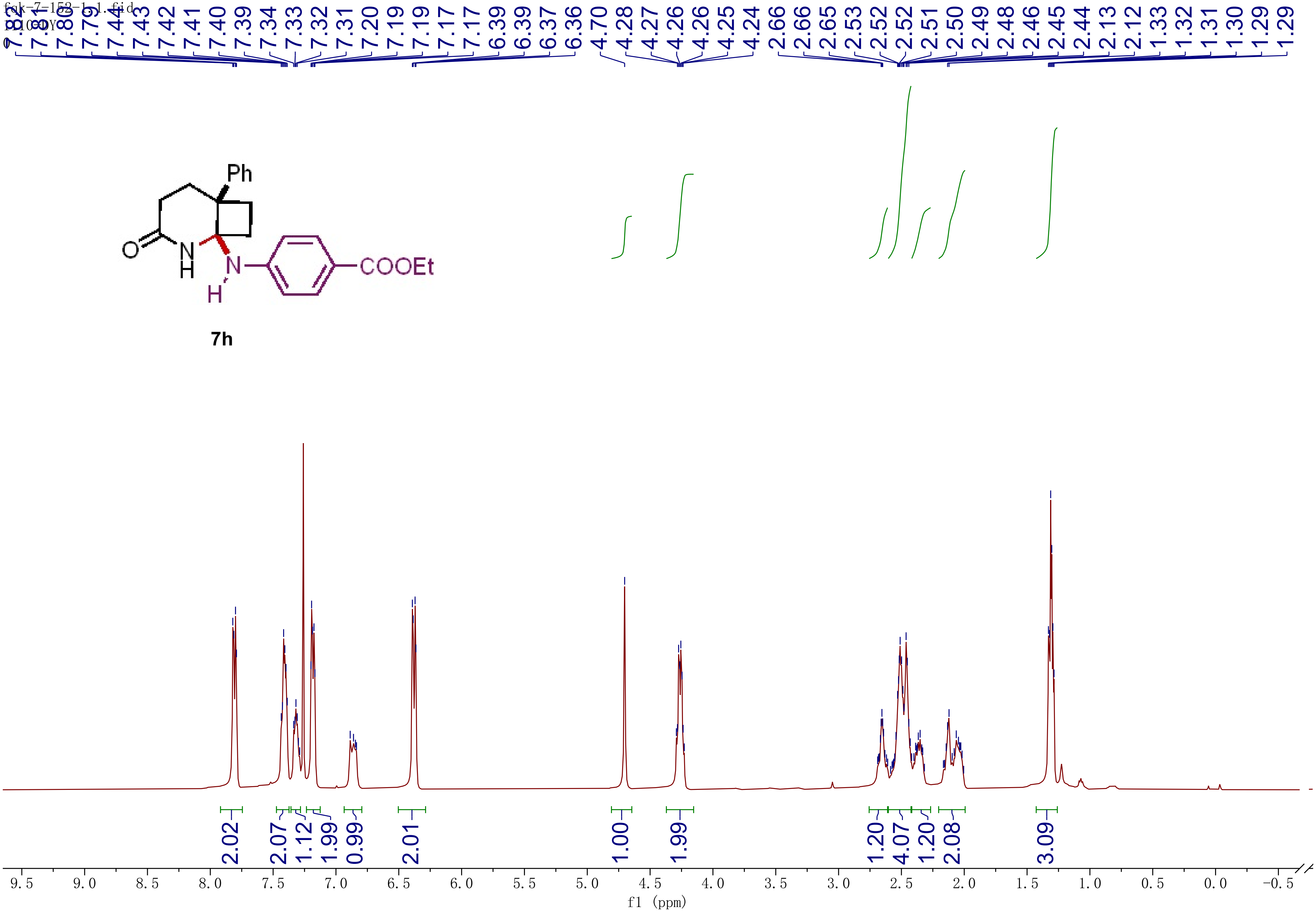
7f

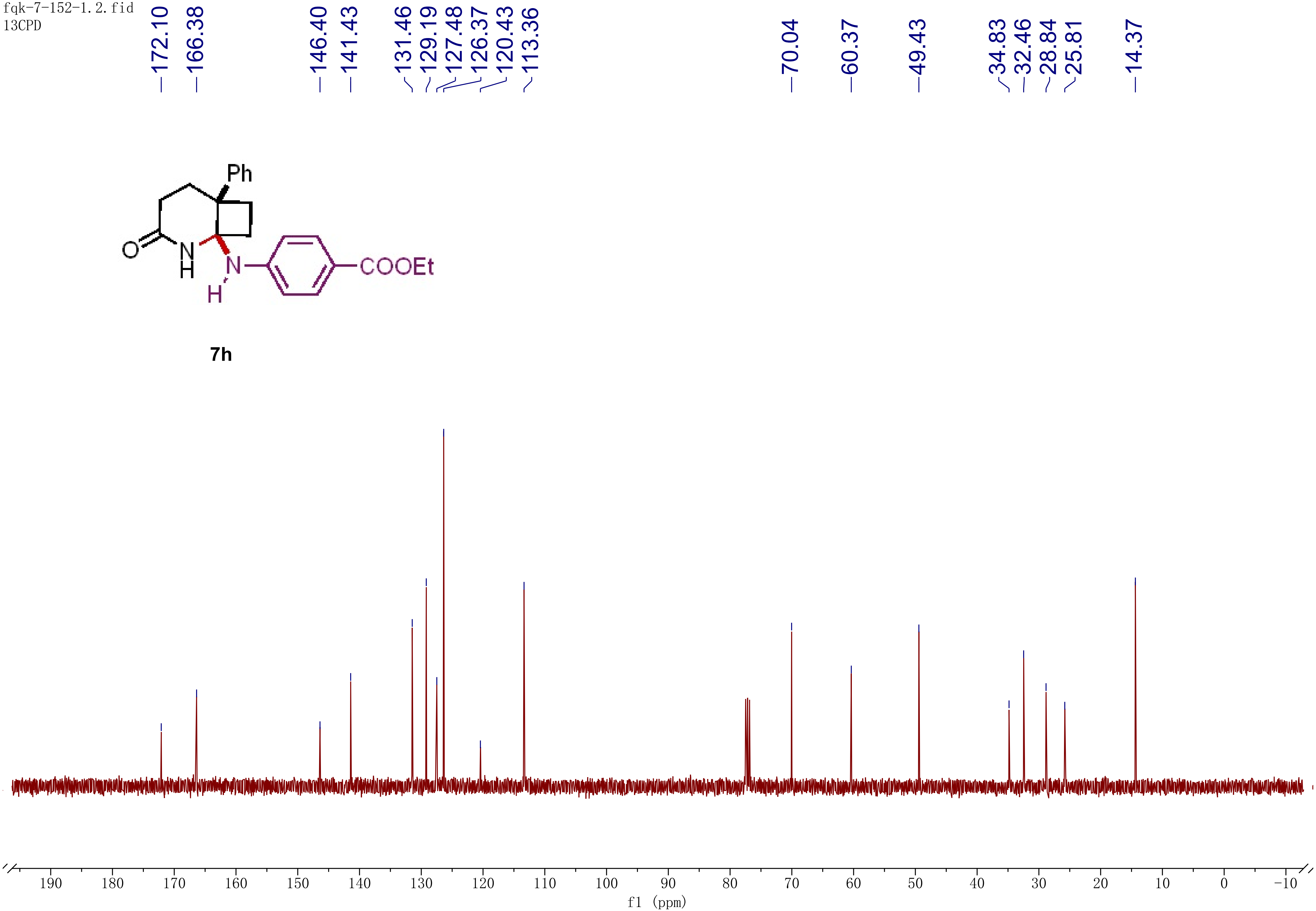



fqk-7-148-2-f.1.fid
F19
-61.32
Ph
O
N
H
N
H
CF3
7f
100
50
0
−50
−100
−150
−200
−250
−300
f1 (ppm)

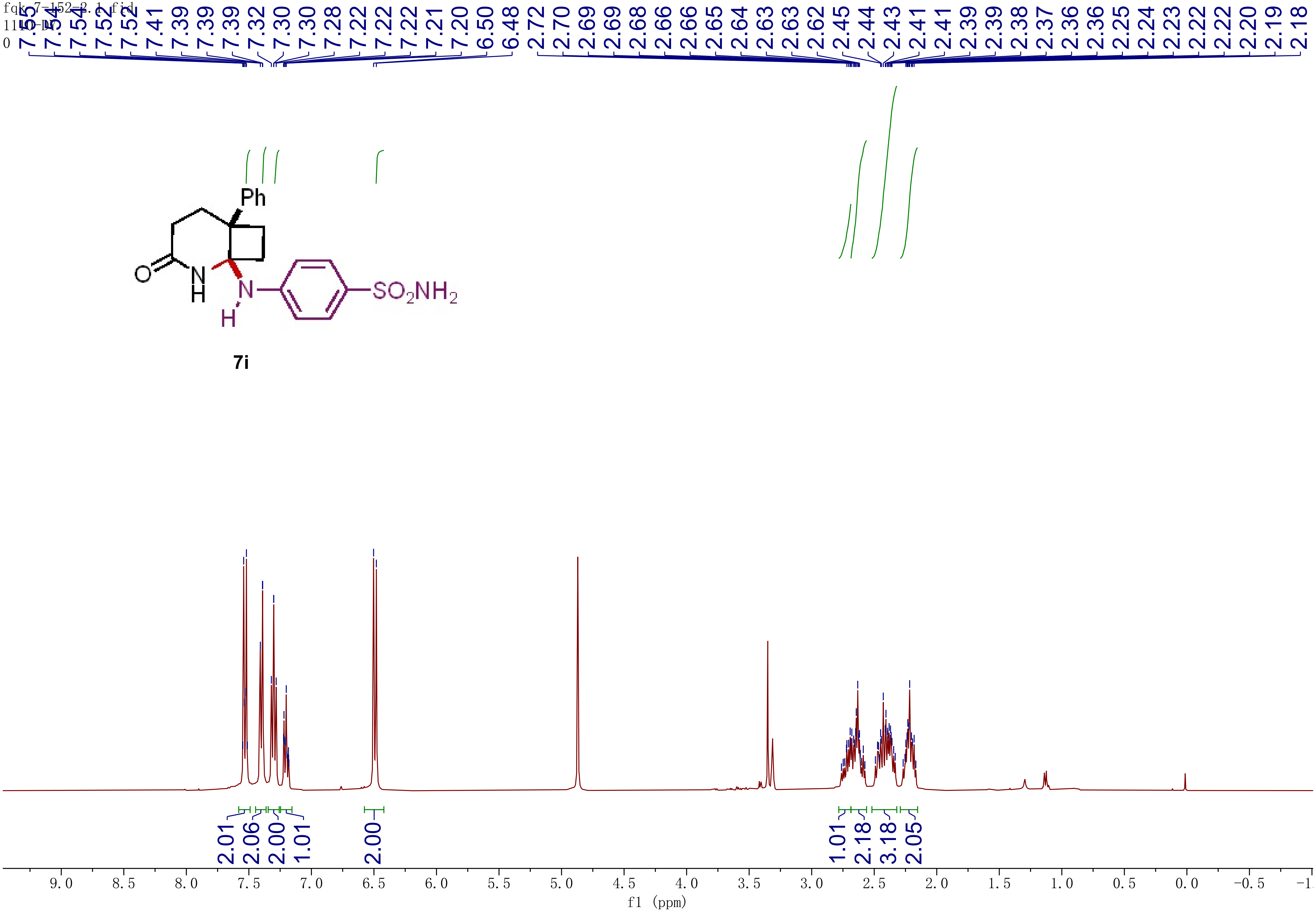

fqk-7-148-1-1.fid
test
8.01 7.98 7.44 7.43 7.41 7.35 7.35 7.34 7.33 7.33 7.32 7.31 7.21 7.19 7.19 7.11 6.42 6.40 5.07 2.65 2.65 2.64 2.64 2.62 2.56 2.55 2.53 2.53 2.52 2.51 2.51 2.50 2.50 2.48 2.48 2.46 2.39 2.37 2.36 2.35 2.20 2.18 2.18 2.17 2.17 2.15 2.10 2.10 2.09 2.08 2.07
Ph
O
N
H
N
H
NO_2
7g
1.96 1.99 1.07 2.02 0.96 2.00 0.98 1.07 4.06 1.10 1.08 1.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

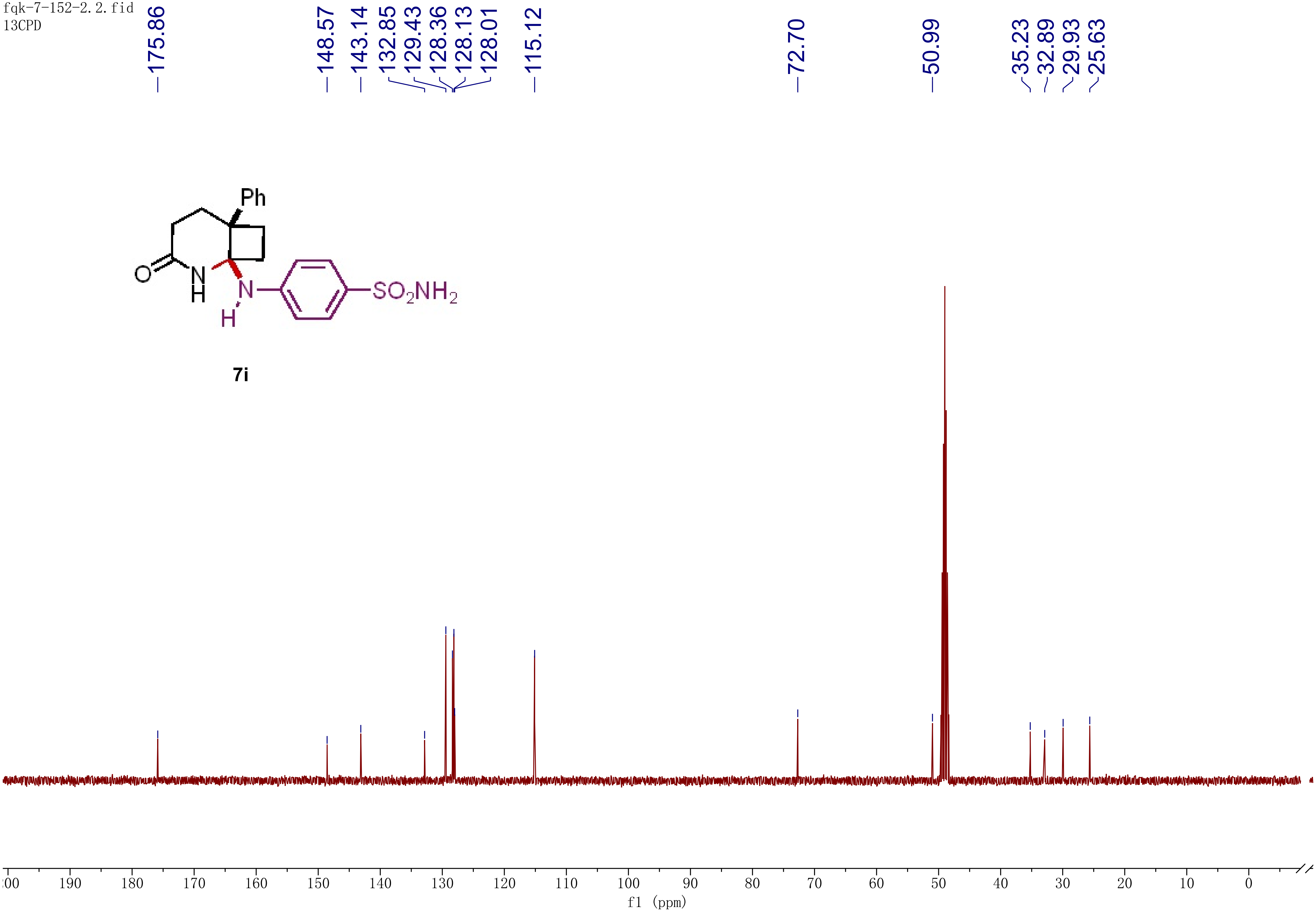

fqk-7-148-1.2.fid
test
172.32
148.31
141.02
139.27
129.33
127.72
126.39
126.04
113.37
70.07
49.56
34.64
32.59
28.84
25.75
Ph
O
N
H
N
H
NO2
7g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


fqk-7-152-1.1.fid
Ph
O
N
H
N
H
COOEt
7h
7.82
7.81
7.80
7.79
7.44
7.43
7.42
7.41
7.40
7.39
7.34
7.33
7.32
7.31
7.20
7.19
7.19
7.17
7.17
6.39
6.39
6.37
6.36
4.70
4.28
4.27
4.26
4.26
4.25
4.24
2.66
2.66
2.65
2.53
2.52
2.52
2.51
2.50
2.49
2.48
2.46
2.45
2.44
2.13
2.12
1.33
1.32
1.31
1.30
1.29
1.29
2.02
2.07
1.12
1.99
0.99
2.01
1.00
1.99
1.20
4.07
1.20
2.08
3.09
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

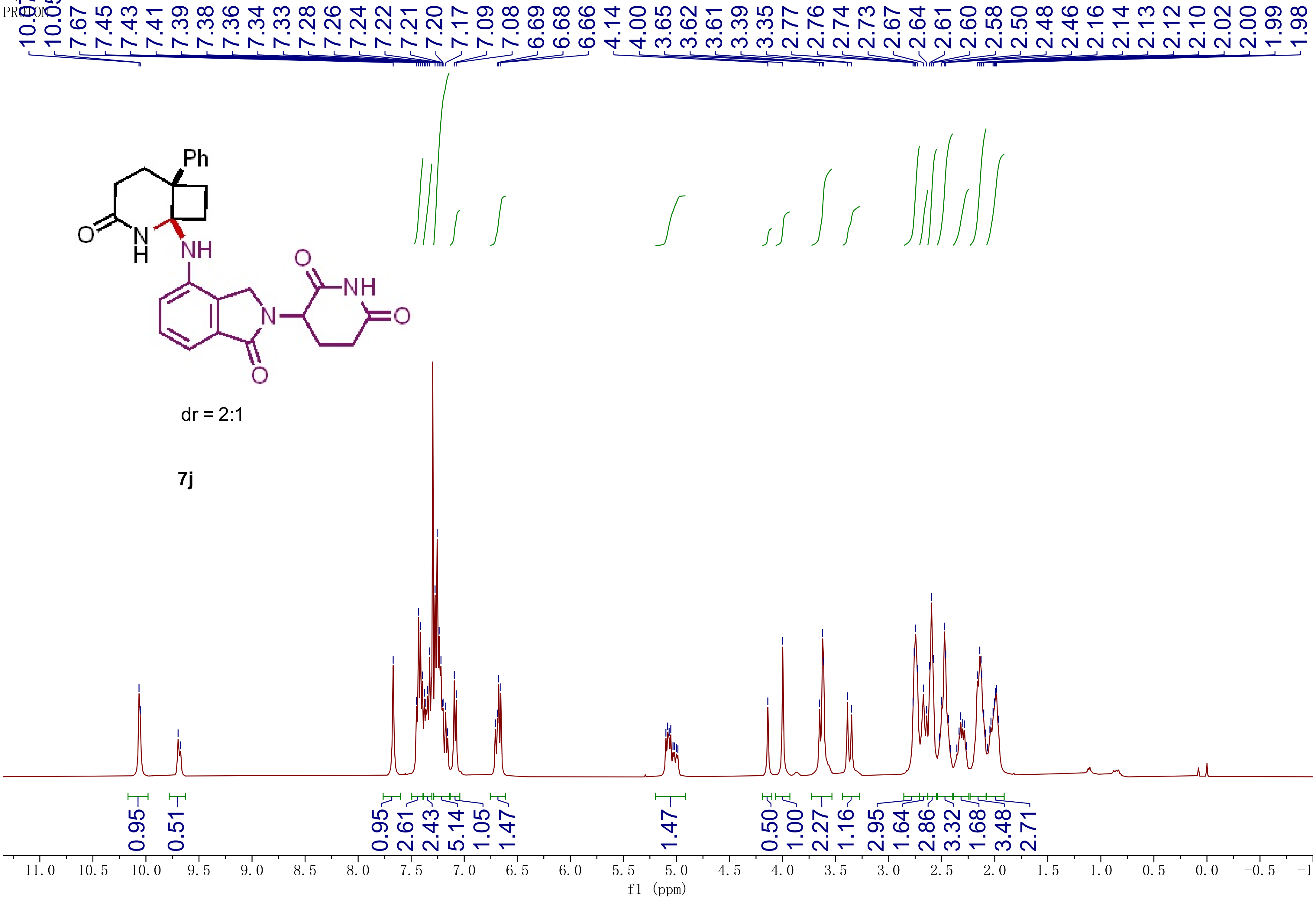

fqk-7-152-1.2.fid
13CPD
172.10
166.38
146.40
141.43
131.46
129.19
127.48
126.37
120.43
113.36
70.04
60.37
49.43
34.83
32.46
28.84
25.81
14.37
Ph
O
N
H
N
H
COOEt
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

7h


fqk 7-152-2.1.fid
7.55 7.54 7.54 7.52 7.52 7.41 7.39 7.39 7.39 7.32 7.30 7.30 7.28 7.22 7.22 7.22 7.21 7.20 6.50 6.48 2.72 2.70 2.69 2.69 2.68 2.66 2.66 2.65 2.64 2.63 2.63 2.62 2.45 2.44 2.43 2.41 2.41 2.39 2.39 2.38 2.37 2.36 2.36 2.25 2.24 2.23 2.22 2.22 2.20 2.19 2.18
Ph
O
N
H
N
H
SO2NH2
7i
2.01 2.06 2.00 1.01 2.00 1.01 2.18 3.18 2.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

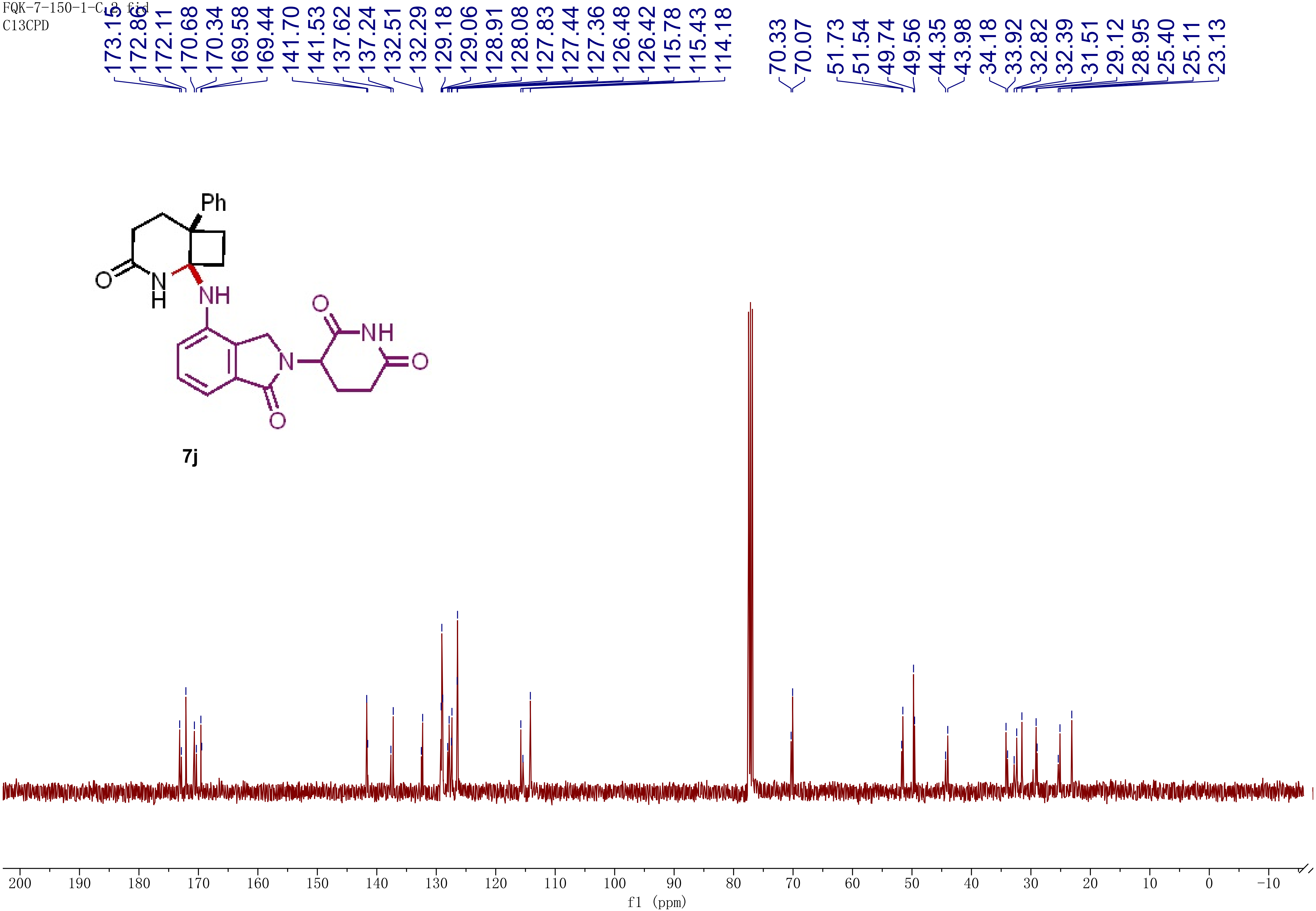

fqk-7-152-2.2.fid
13CPD
175.86
148.57
143.14
132.85
129.43
128.36
128.13
128.01
115.12
72.70
50.99
35.23
32.89
29.93
25.63
Ph
O
N
H
N
H
SO2NH2
7i
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

FQK-7-150-1-C.1.fid
PROTON



dr = 2:1

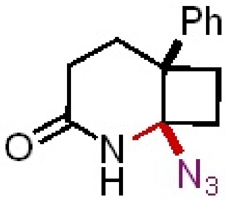
7j



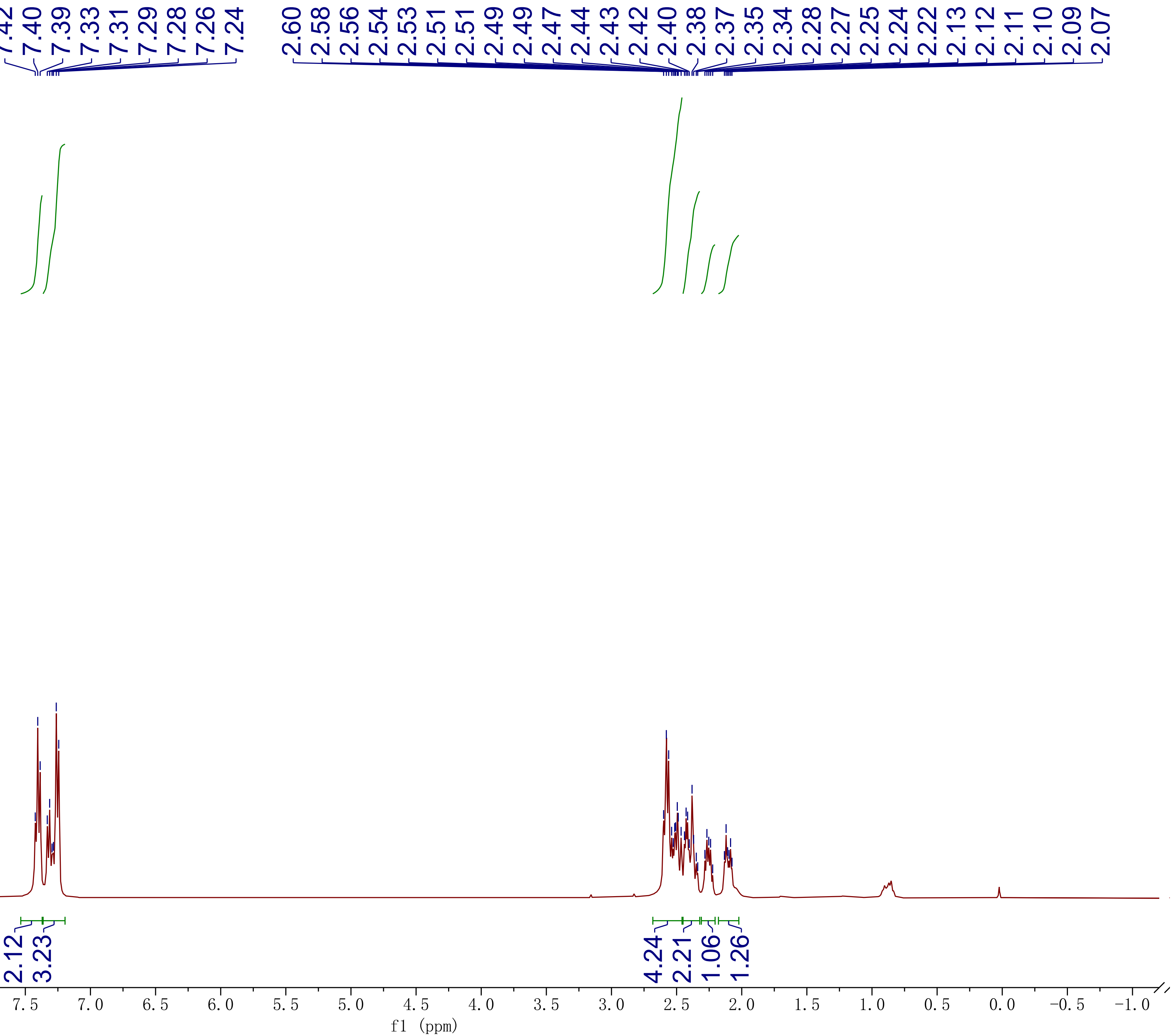
FQK-7-150-1-C.2.fid
C13CPD
173.15
172.86
172.11
170.68
170.34
169.58
169.44
141.70
141.53
137.62
137.24
132.51
132.29
129.18
129.06
128.91
128.08
127.83
127.44
127.36
126.48
126.42
115.78
115.43
114.18
70.33
70.07
51.73
51.54
49.74
49.56
44.35
43.98
34.18
33.92
32.82
32.39
31.51
29.12
28.95
25.40
25.11
23.13
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
Ph
O
N
H
NH
O
NH
N
O
O

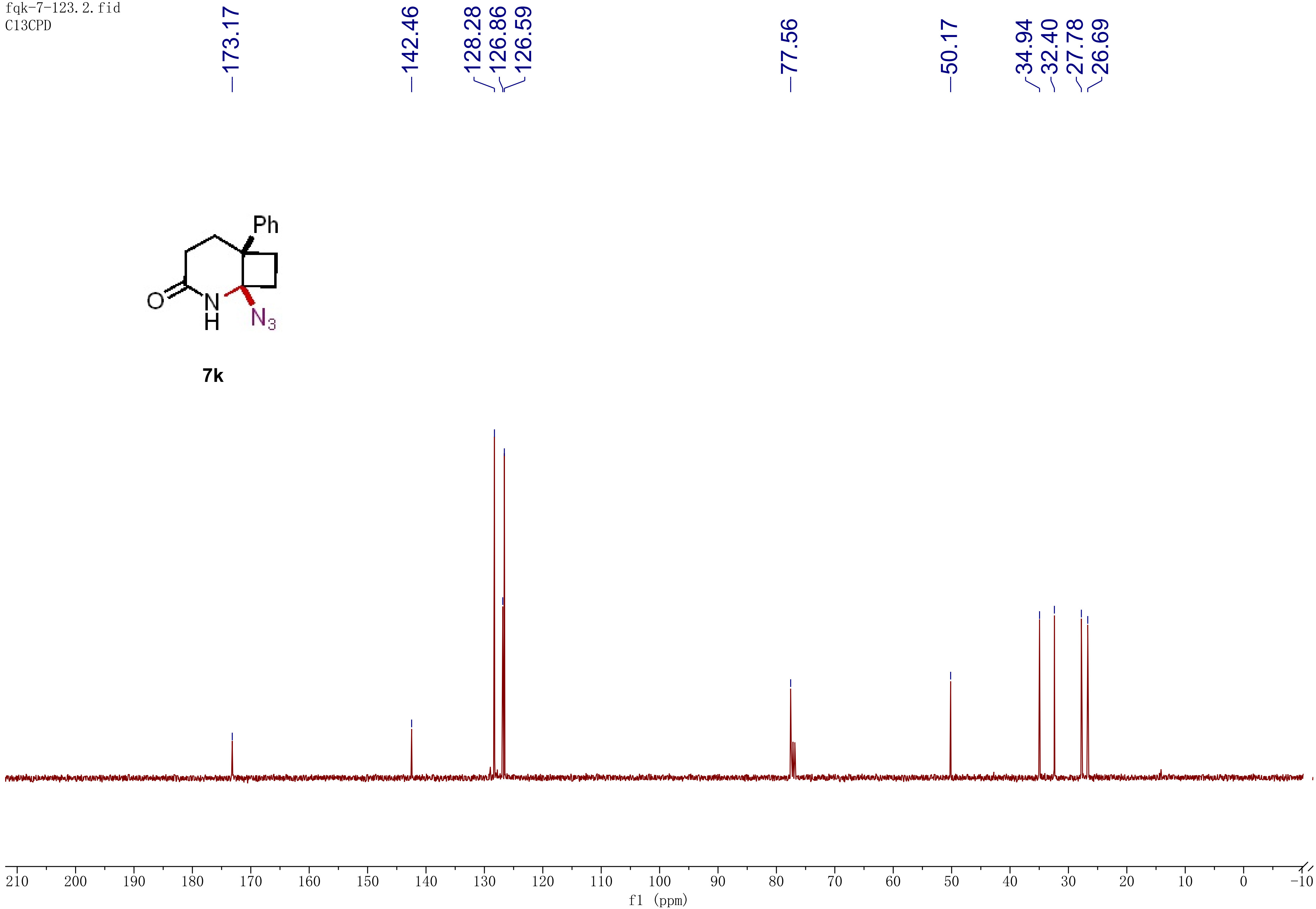
7j

fqk-7-34-1.1.fid
test

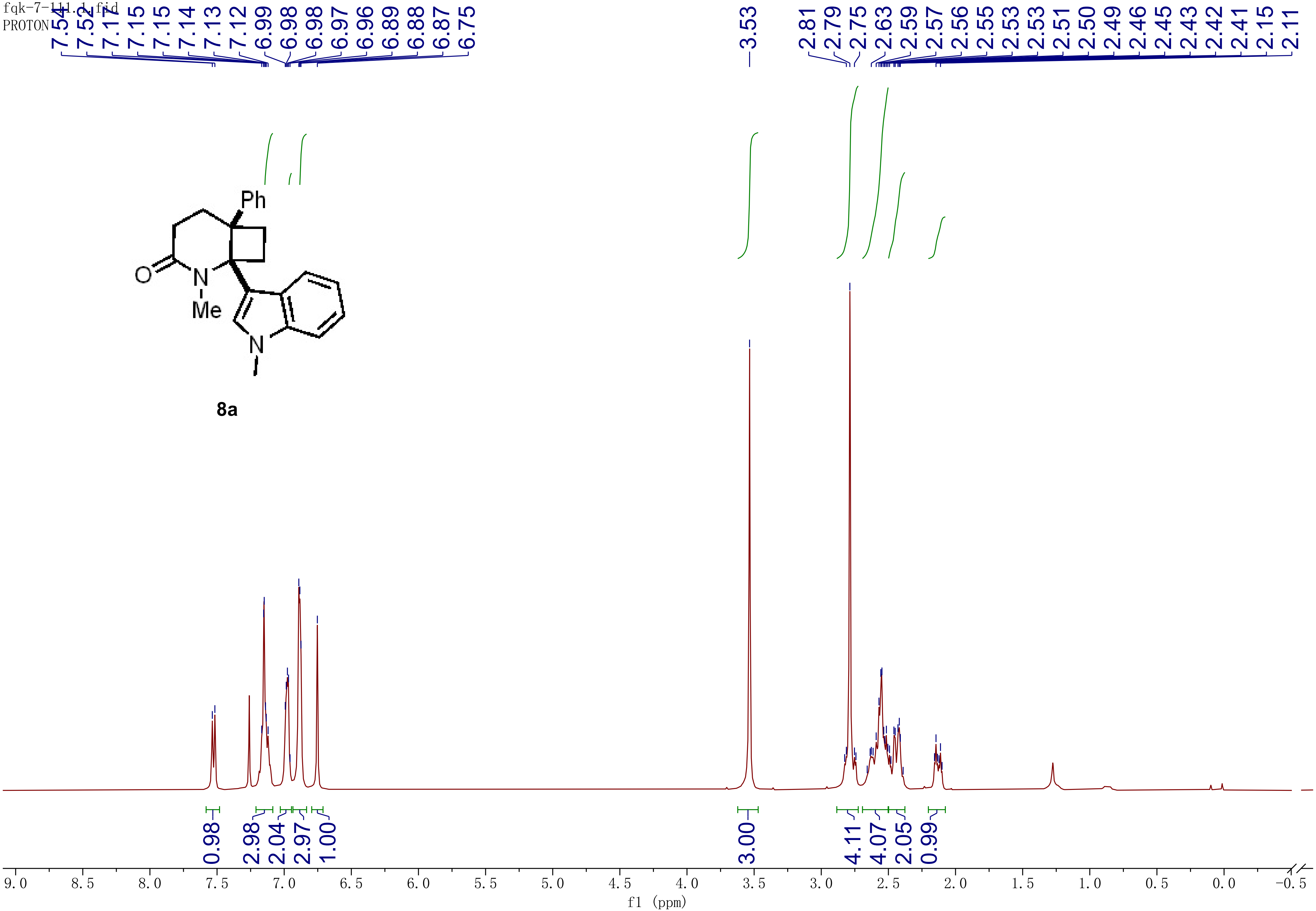


7k

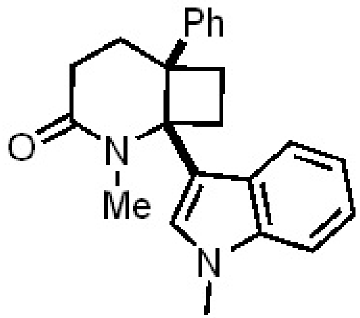



fqk-7-123.2.fid
C13CPD
173.17
142.46
128.28
126.86
126.59
77.56
50.17
34.94
32.40
27.78
26.69
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
Ph
O
N
H
N_3

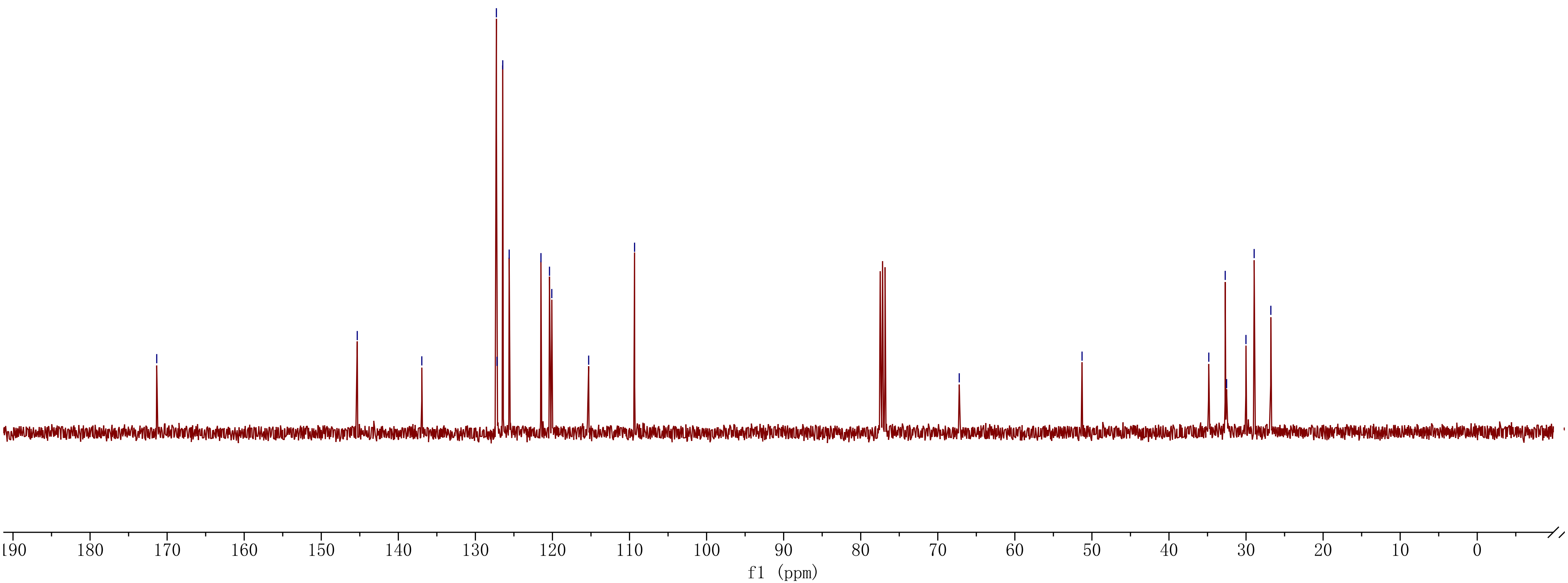
7k

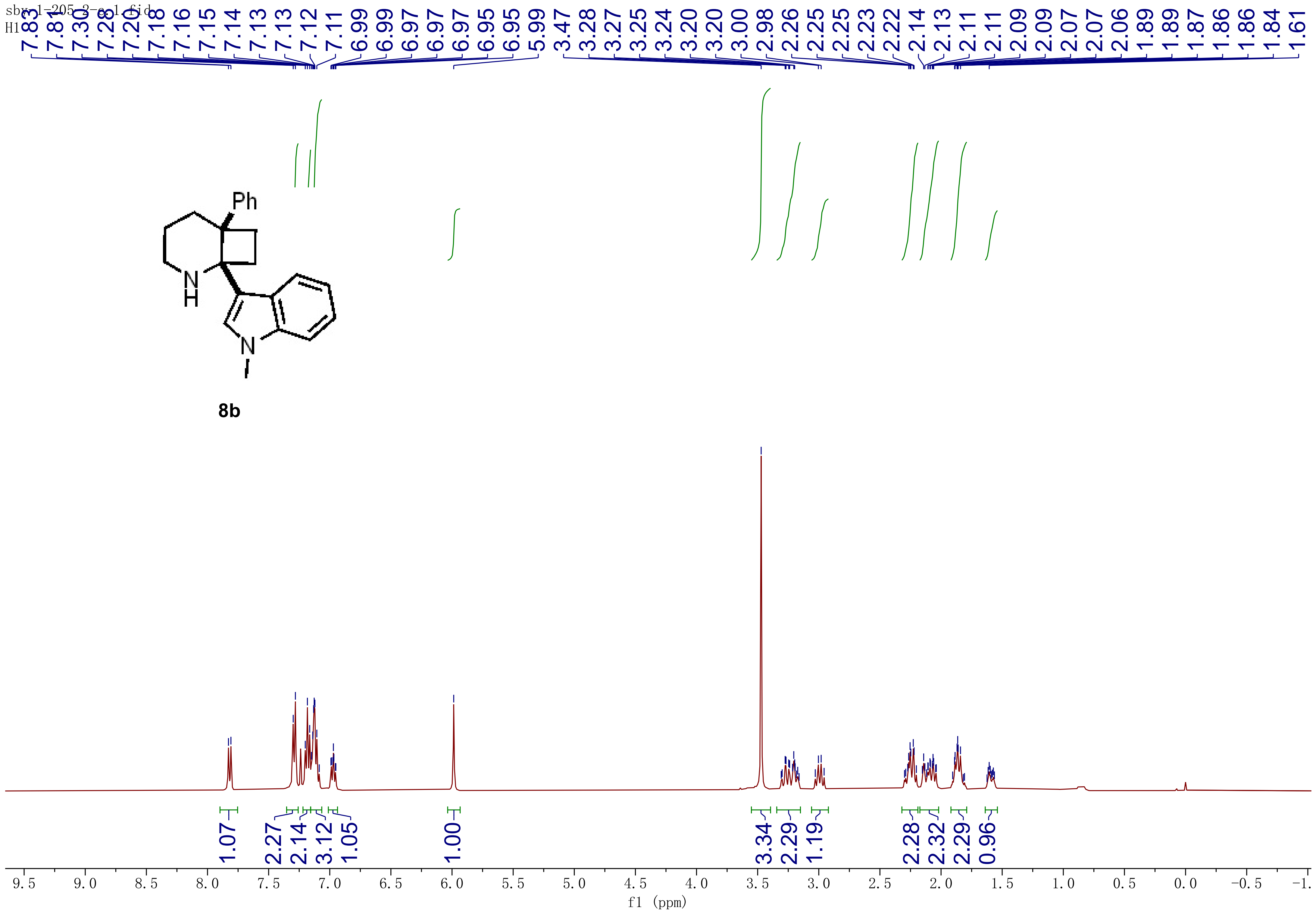

fqk-7-111.1.fid
PROTON
7.54
7.52
7.17
7.15
7.15
7.14
7.13
7.12
6.99
6.98
6.98
6.97
6.96
6.89
6.88
6.87
6.75
3.53
2.81
2.79
2.75
2.63
2.59
2.57
2.56
2.55
2.53
2.53
2.51
2.50
2.49
2.46
2.45
2.43
2.42
2.41
2.15
2.11
Ph
O
N
Me
N
8a
0.98
2.98
2.04
2.97
1.00
3.00
4.11
4.07
2.05
0.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

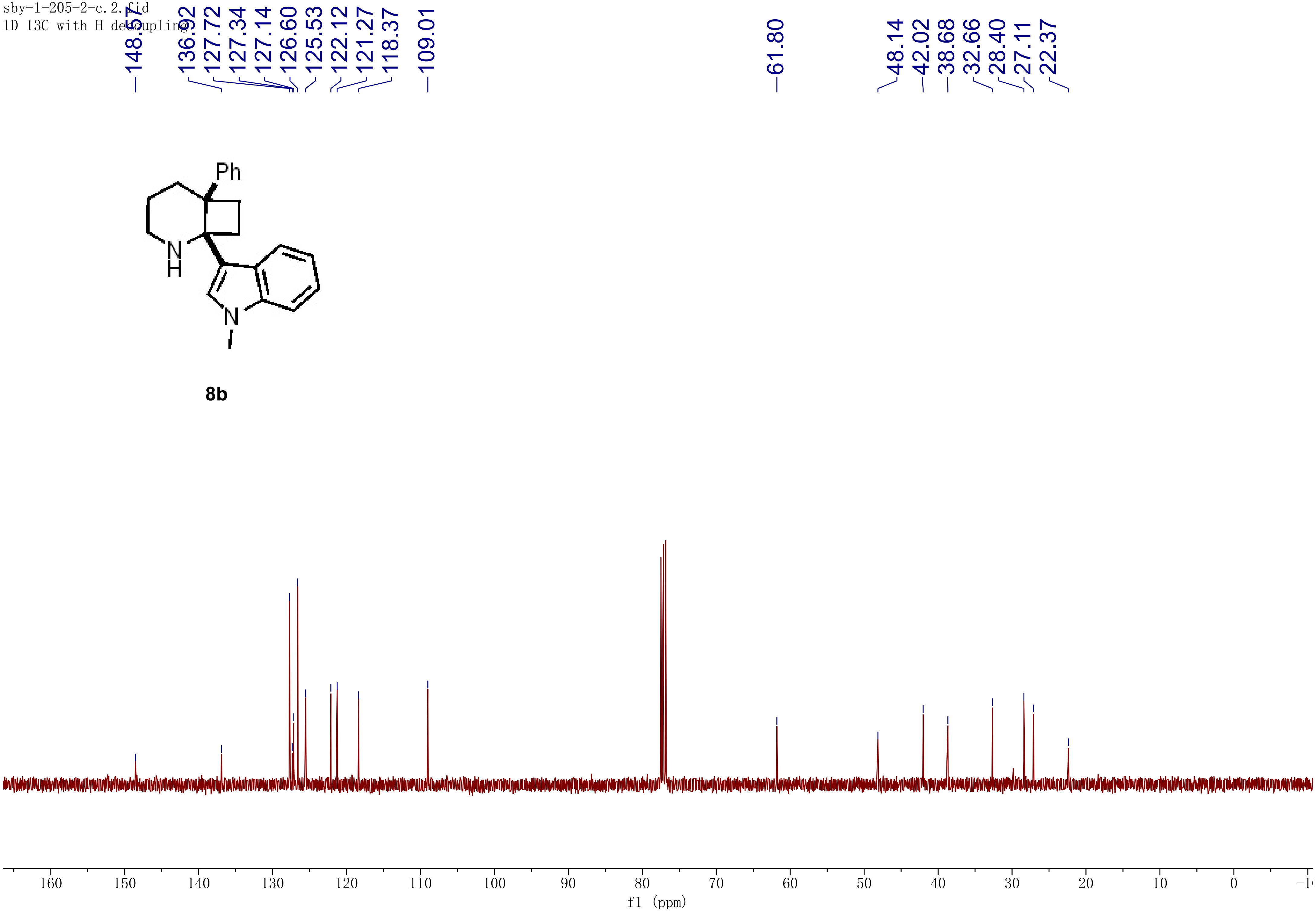
fqk-7-111.2.fid
C13CPD



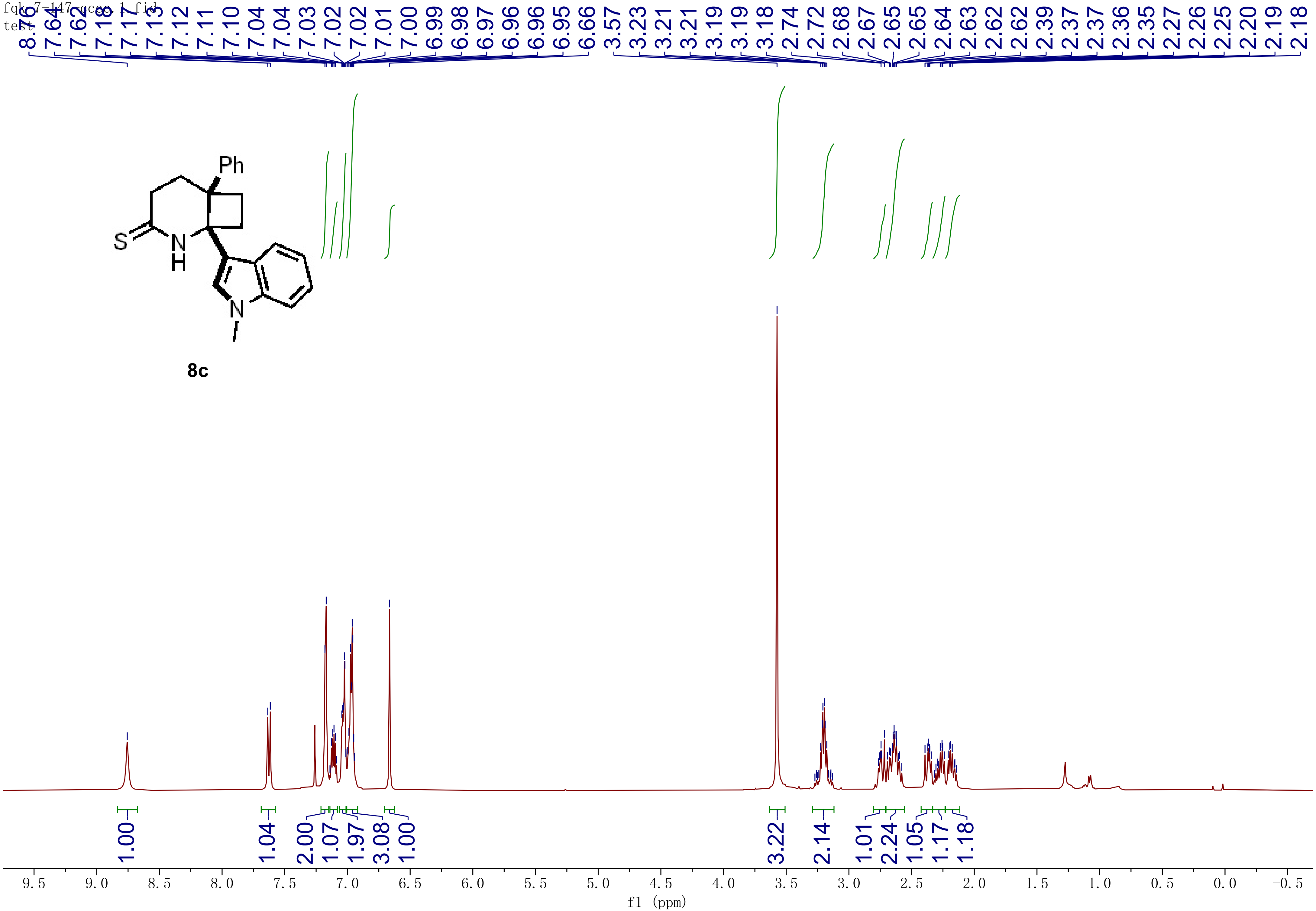
8a

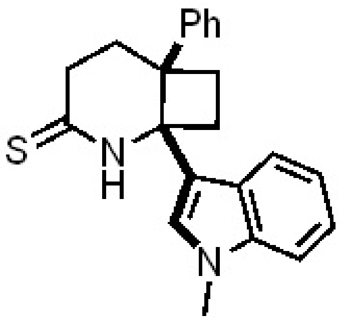



sby-1-205-2-c.1.fid
H1
7.83 7.81 7.30 7.28 7.20 7.18 7.16 7.15 7.14 7.13 7.13 7.12 7.11 6.99 6.99 6.97 6.97 6.97 6.95 6.95 5.99 3.47 3.28 3.27 3.25 3.24 3.20 3.20 3.00 2.98 2.26 2.25 2.25 2.23 2.22 2.14 2.13 2.11 2.11 2.09 2.09 2.07 2.07 2.06 1.89 1.89 1.87 1.86 1.86 1.84 1.61
Ph
N
H
N
8b
1.07 2.27 2.14 3.12 1.05 1.00 3.34 2.29 1.19 2.28 2.32 2.29 0.96
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)


sby-1-205-2-c.2.fid
1D 13C with H decoupling
148.57
136.92
127.72
127.34
127.14
126.60
125.53
122.12
121.27
118.37
109.01
61.80
48.14
42.02
38.68
32.66
28.40
27.11
22.37
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
Ph
N
H
N

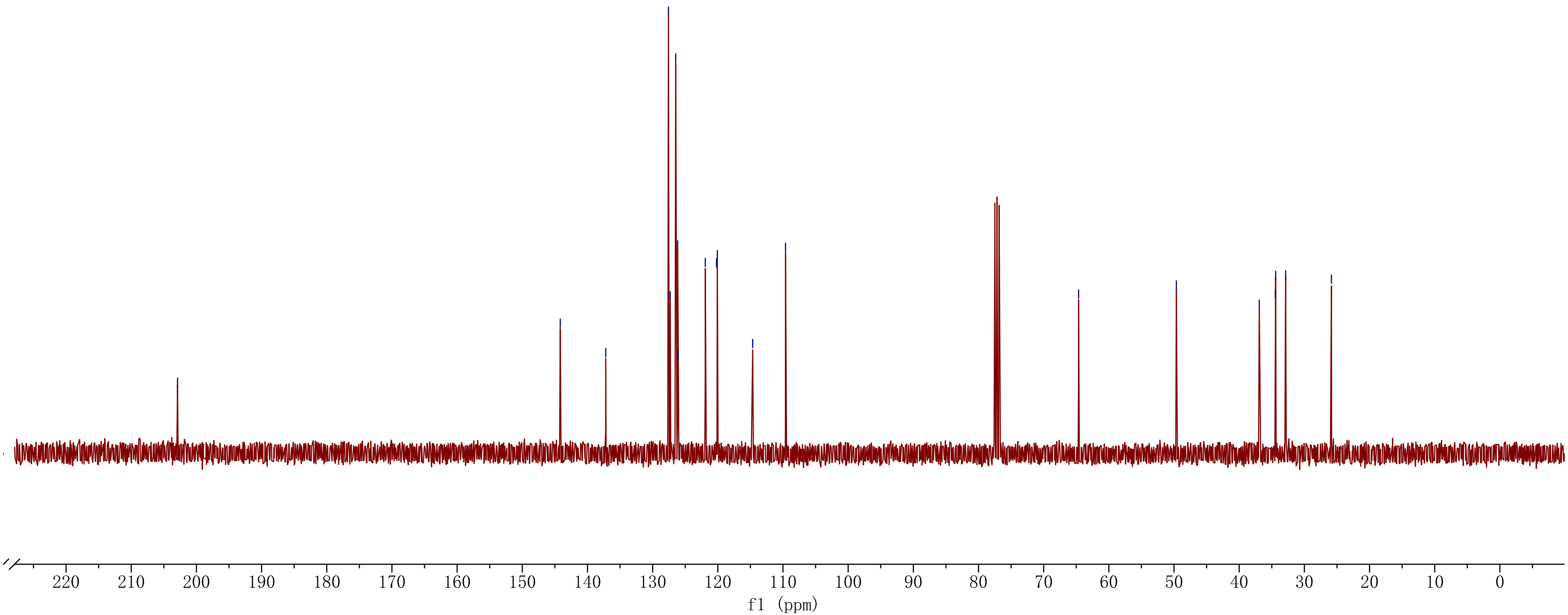
8b


8c
fqk-7-147-ccee.1.fid
test
8.76 7.64 7.62 7.18 7.17 7.13 7.12 7.11 7.10 7.04 7.04 7.03 7.02 7.02 7.01 7.00 6.99 6.98 6.97 6.96 6.96 6.95 6.66 3.57 3.23 3.21 3.21 3.19 3.19 3.18 2.74 2.72 2.68 2.67 2.65 2.65 2.64 2.63 2.62 2.62 2.39 2.37 2.37 2.36 2.35 2.27 2.26 2.25 2.20 2.19 2.18
1.00 1.04 2.00 1.07 1.97 3.08 1.00 3.22 2.14 1.01 2.24 1.05 1.17 1.18
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)



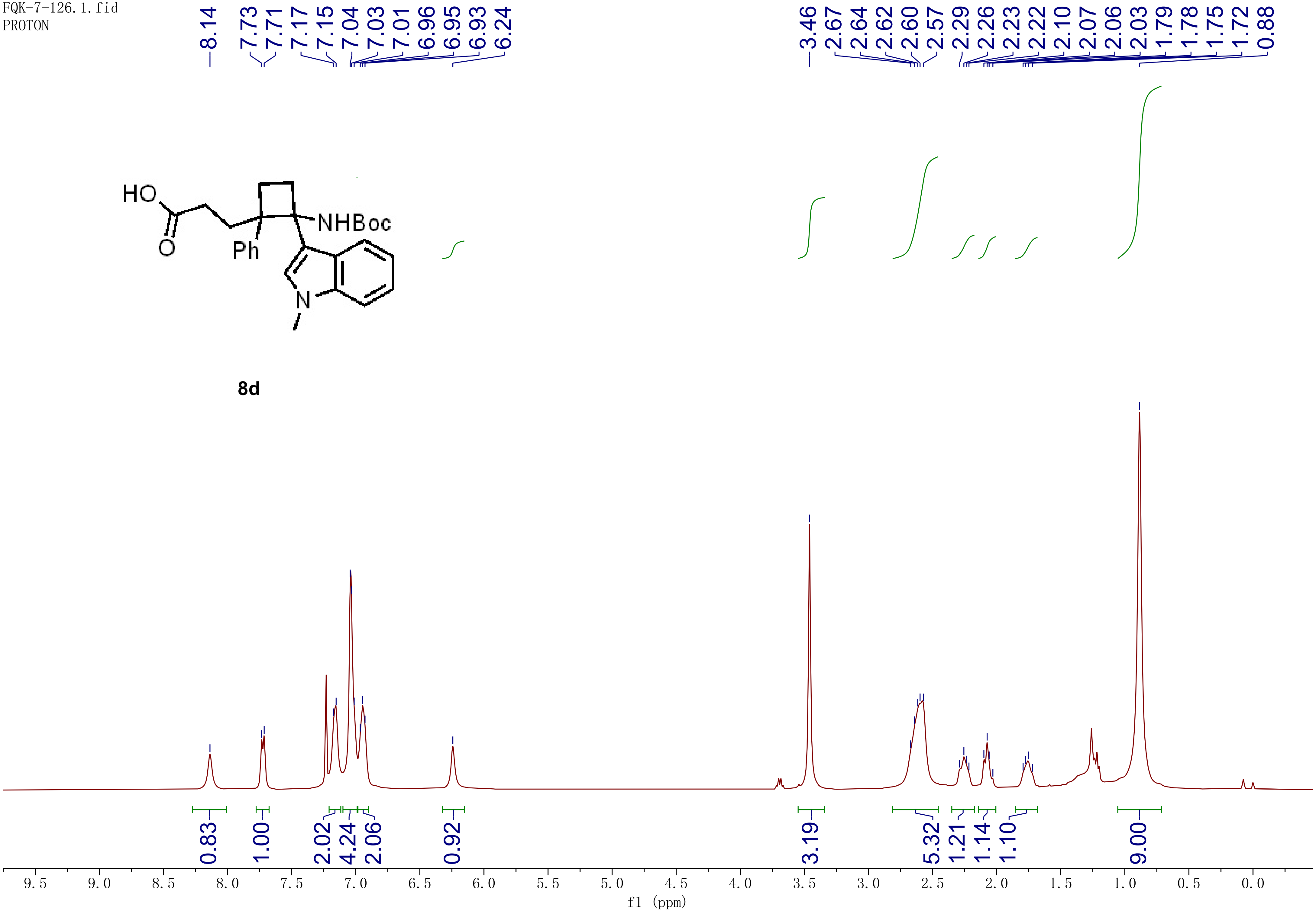
Ph
S
N
H
N

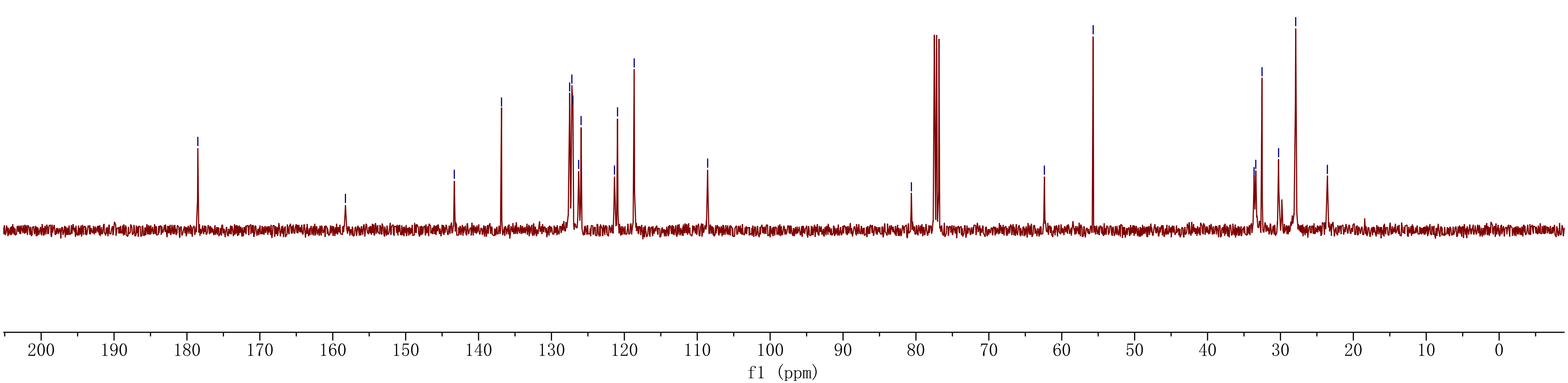
8c

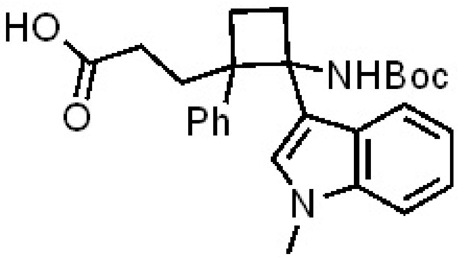


fqk-7-147-cccccc.2.fid
1D 13C with H decoupling
202.89
144.18
137.19
127.58
127.30
126.46
126.16
126.10
121.92
120.20
120.07
114.65
109.61
64.64
49.64
36.92
34.46
34.40
32.87
25.85
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

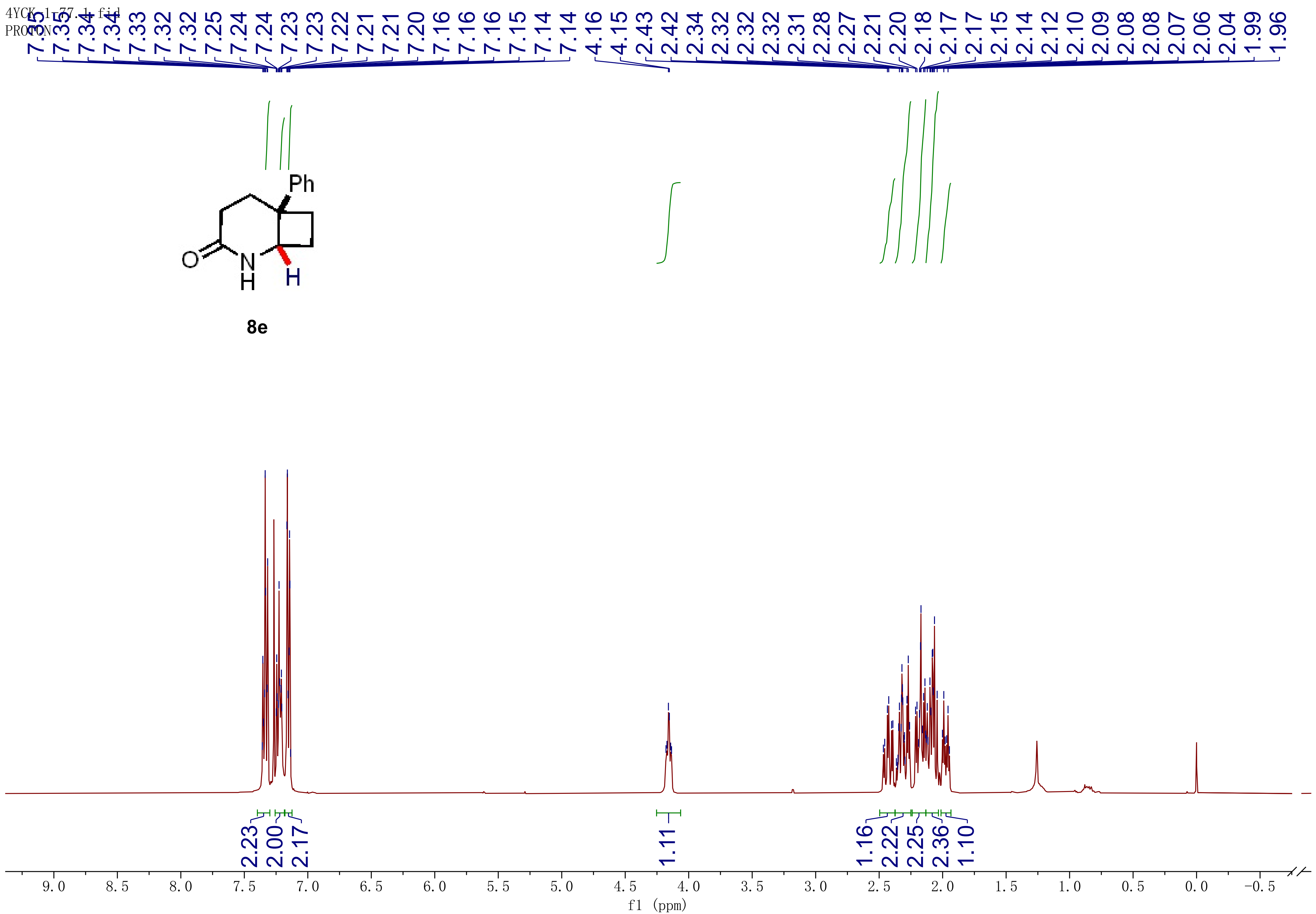
FQK-7-126.1.fid
PROTON

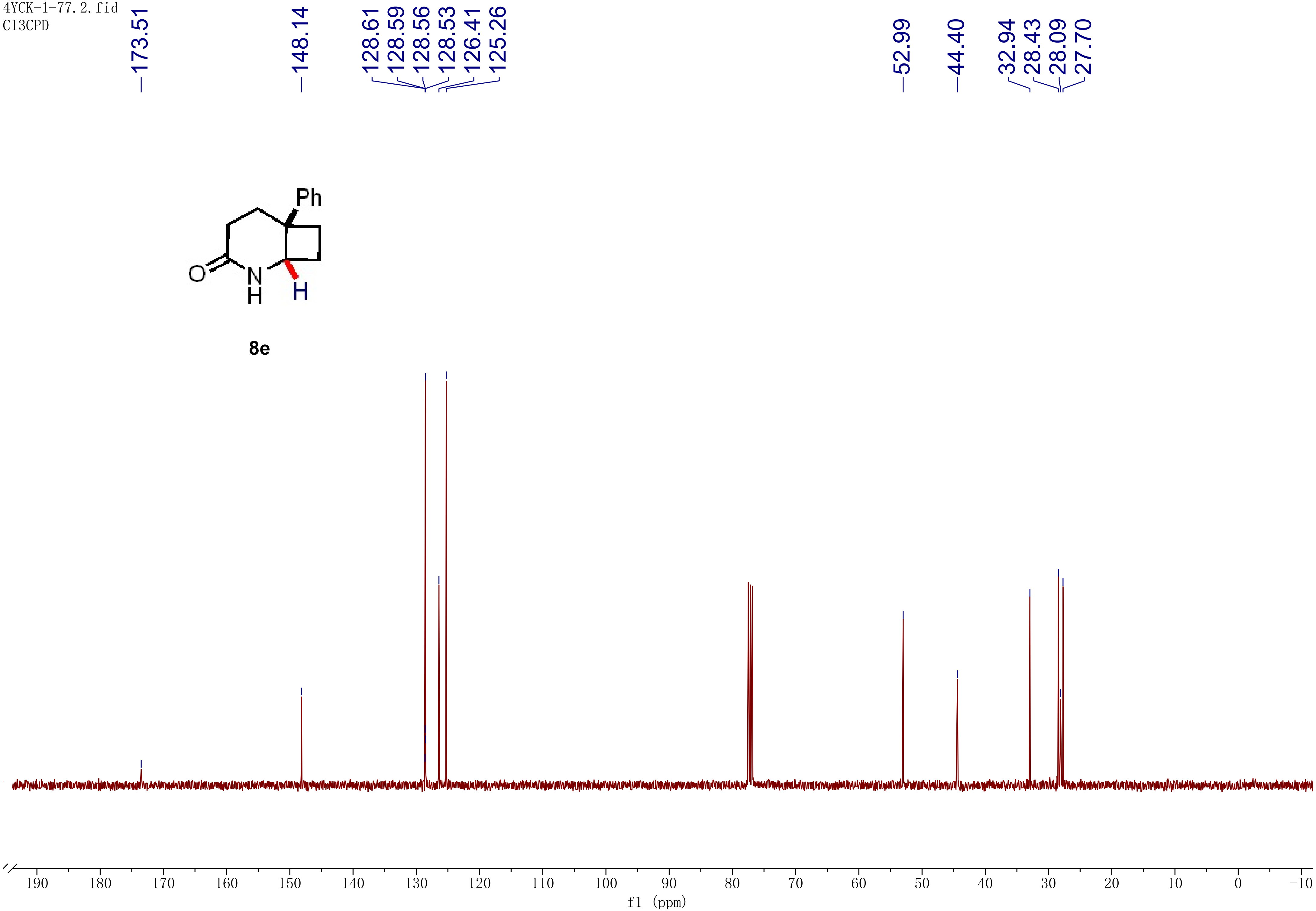


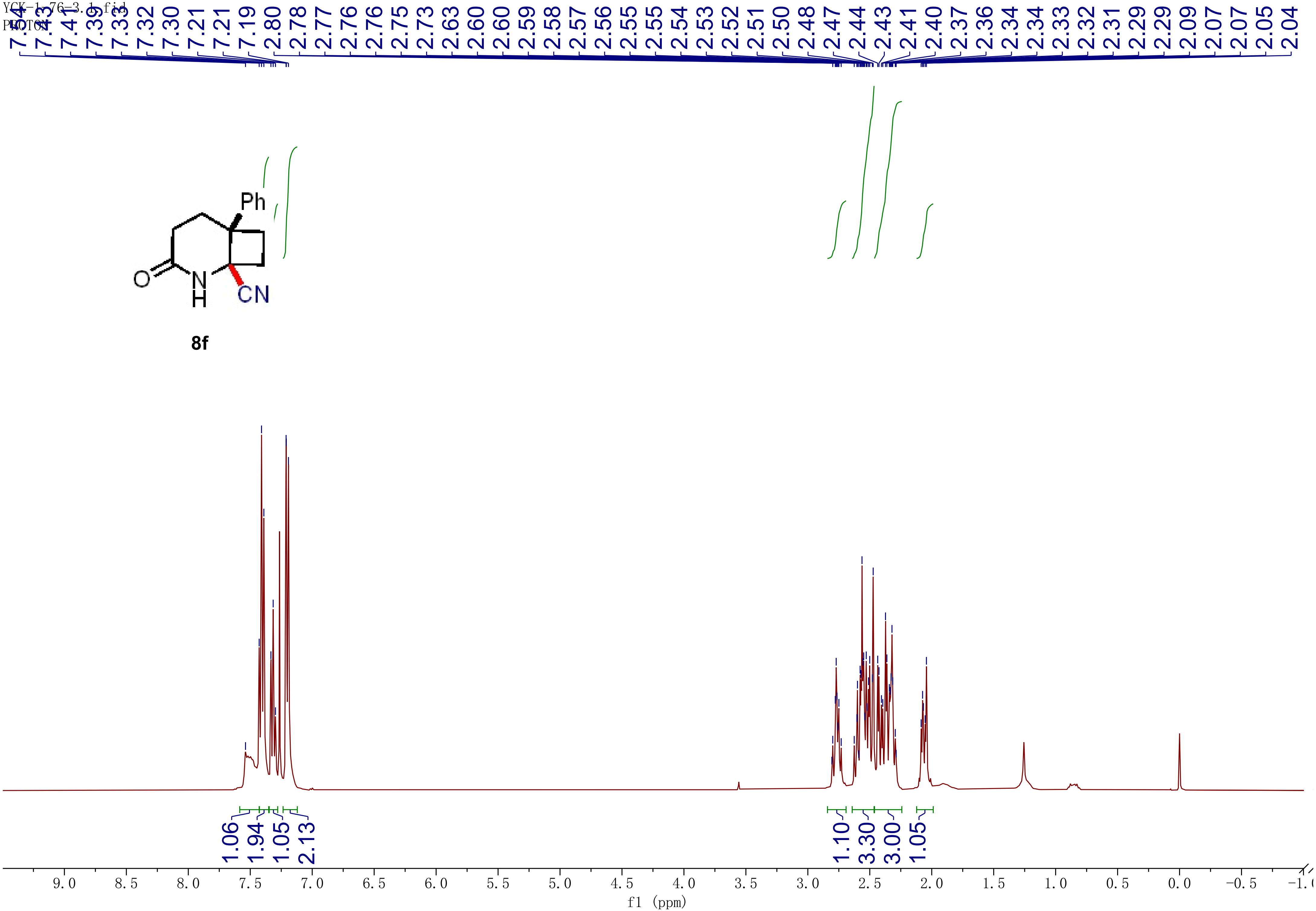

FQK-7-126.2.fid
C13CPD
178.51
158.26
143.33
136.85
127.50
127.20
127.06
126.27
125.93
121.36
120.94
118.65
108.57
80.62
62.38
55.69
33.62
33.38
32.53
30.26
27.91
23.56
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


HO
O
Ph
NHBoc
N

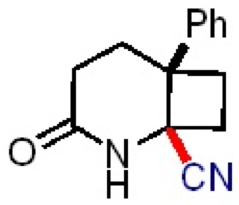
8d


4YCK-1-77.1.fid
PROTON
8e
f1 (ppm)

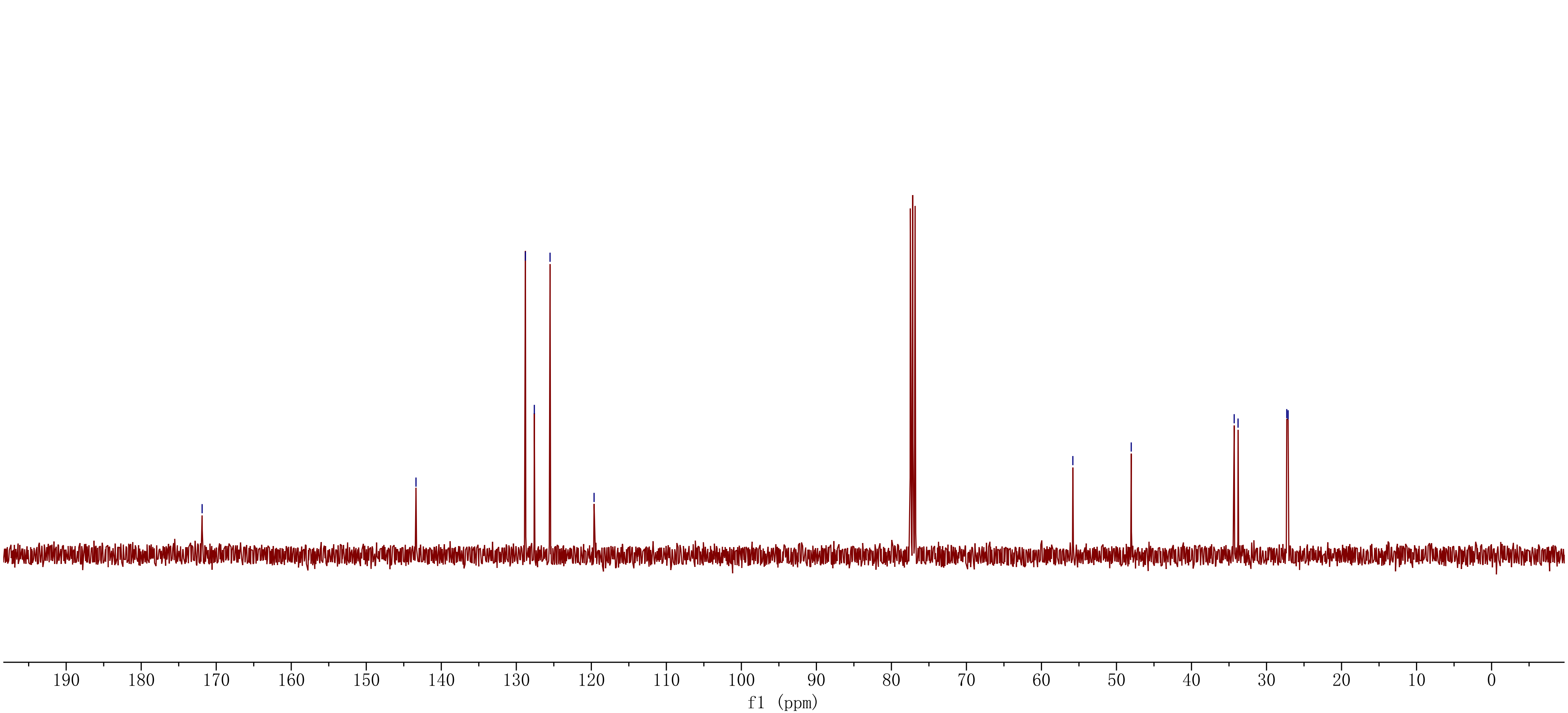

4YCK-1-77.2.fid
C13CPD
173.51
148.14
128.61
128.59
128.56
128.53
126.41
125.26
52.99
44.40
32.94
28.43
28.09
27.70
Ph
O
N
H
H
8e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


YCK-1-76-3.1.fid
PROTON
7.54 7.43 7.41 7.39 7.33 7.32 7.30 7.21 7.21 7.19 2.80 2.78 2.77 2.76 2.76 2.75 2.73 2.63 2.60 2.60 2.59 2.58 2.57 2.56 2.55 2.55 2.54 2.53 2.52 2.51 2.50 2.48 2.47 2.44 2.43 2.41 2.40 2.37 2.36 2.34 2.34 2.33 2.32 2.31 2.29 2.29 2.09 2.07 2.07 2.05 2.04
Ph
O
N
H
CN
8f
1.06
1.94
1.05
2.13
1.10
3.30
3.00
1.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

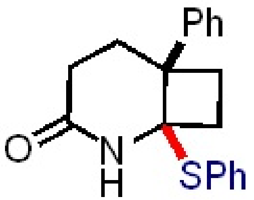
YCK-1-76-3.2.fid
C13CPD



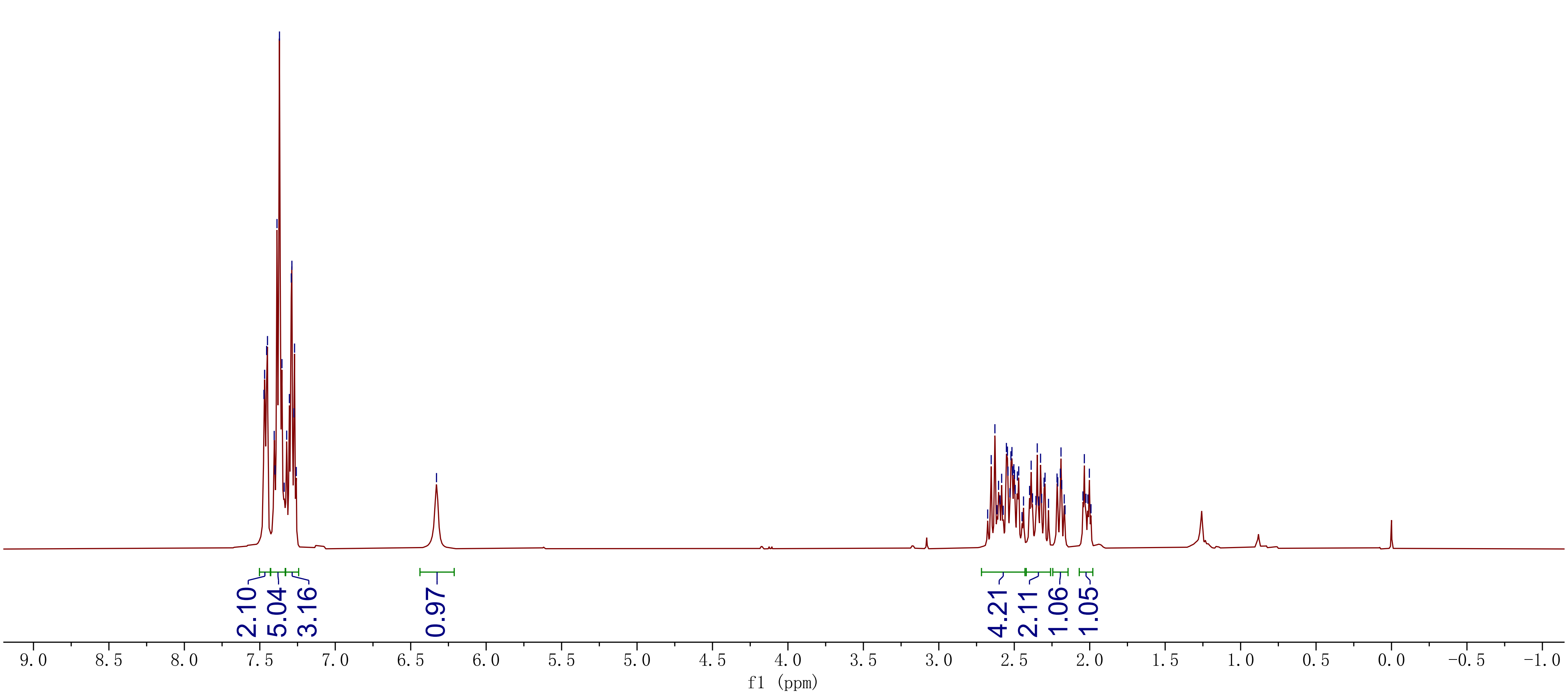
8f



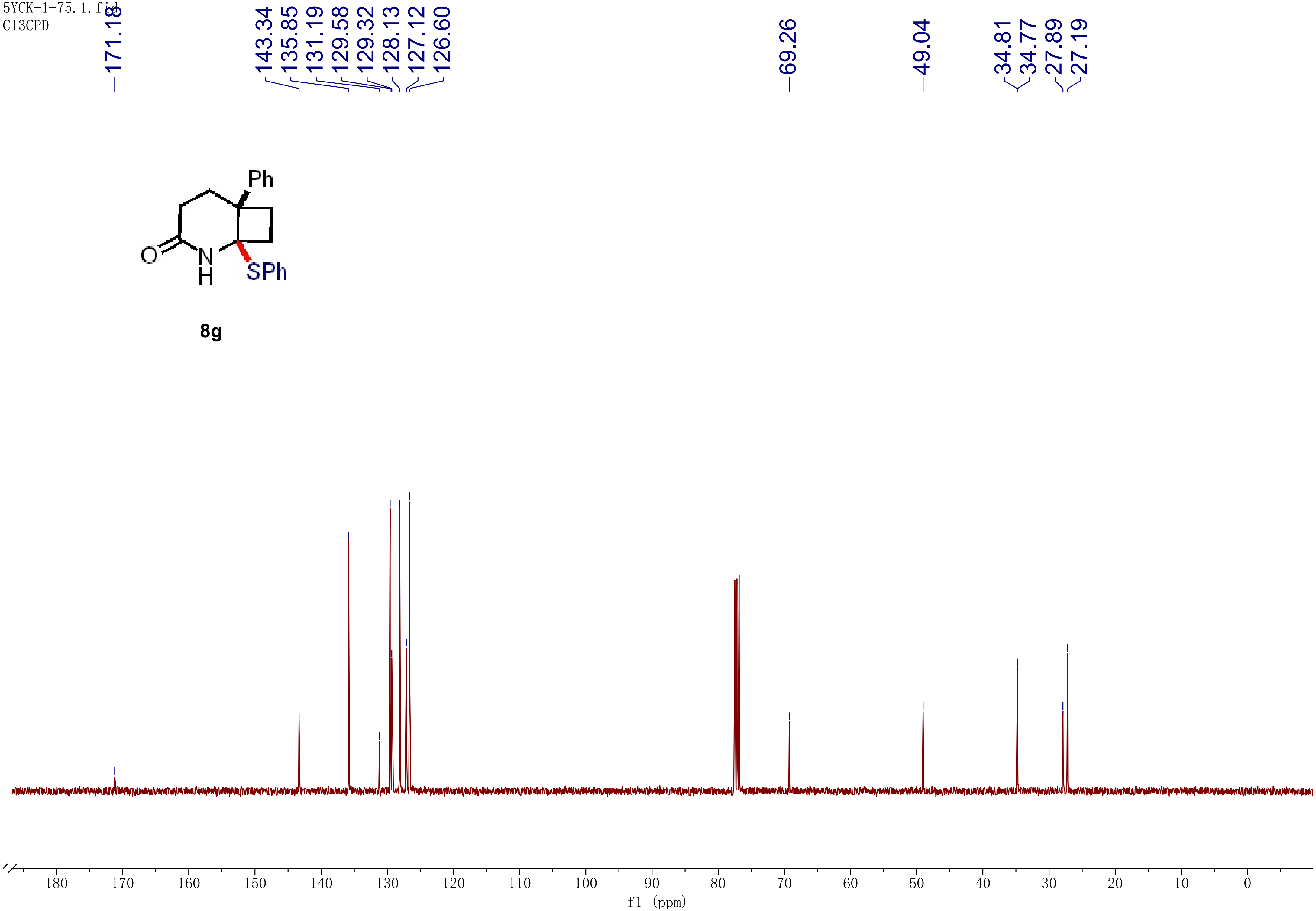
YCK-1-75.1.fid
PROTON



8g



5YCK-1-75.1.fid
C13CPD



8g

10. Checkcif of compound 3a、4a、5c、5q、6f、7a、7b、7c

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 37-mr-20250417a_auto

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: 37-mr-20250417a_auto

```
Bond precision:   C-C = 0.0043 A            Wavelength=0.71073

Cell:             a=8.6158(3)     b=17.4664(6)       c=23.9834(9)
                  alpha=90        beta=92.064(3)     gamma=90
Temperature:      289 K

                Calculated                  Reported
Volume          3606.9(2)                   3606.8(2)
Space group     P 21/n                      P 1 21/n 1
Hall group      -P 2yn                      -P 2yn
Moiety formula  C22 H22 N2 O                C22 H22 N2 O
Sum formula     C22 H22 N2 O                C22 H22 N2 O
Mr              330.42                      330.41
Dx,g cm-3       1.217                       1.217
Z               8                           8
Mu (mm-1)       0.075                       0.075
F000            1408.0                      1408.0
F000'           1408.52
h,k,lmax        10,21,28                    10,21,28
Nref            6611                        6595
Tmin,Tmax       0.983,0.990                 0.507,1.000
Tmin'           0.983

Correction method= # Reported T Limits: Tmin=0.507 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.998          Theta(max)= 25.349

R(reflections)= 0.0697( 5012)          wR2(reflections)=
                                       0.2051( 6595)
S = 1.034                 Npar= 453
```

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 C

```
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
            The relevant atom site should be identified.
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ....       3.79 Report
PLAT097_ALERT_2_C Large Reported Max.  (Positive) Residual Density       0.73 eA-3
PLAT220_ALERT_2_C NonSolvent   Resd 2  C   Ueq(max)/Ueq(min) Range        3.7 Ratio
PLAT234_ALERT_4_C Large Hirshfeld Difference C50      --C51      .       0.17 Ang.
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ...............     0.0043 Ang.
PLAT410_ALERT_2_C Short Intra H...H Contact  H37A     ..H47      .       1.90 Ang.
                                                    x,y,z  =      1_555 Check
PLAT601_ALERT_2_C Unit Cell Contains Solvent Accessible VOIDS   <=         81 Ang**3
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     15.816 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      3.135 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         12 Report
              -1  0  1,  -1  7  3,  -9  7  4,   2  0 10,  -9  0 15,  -8  1 15,
              -7  1 15,  -8  0 16,  -8  1 16,  -8  2 16,  -8  1 17,  -8  2 17,
```

Alert level G

```
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          2 Report
              H17   H42
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.11 Report
PLAT793_ALERT_4_G Model has Chirality at C10         (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C13         (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C35         (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C38         (Centro SpGr)          R Verify
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          4 Note
               0  2  0,   0  1  1,   0  0  2,   0  1  2,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          1 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File         11 Note
              -9  0 15,  -9  1 15,  -8  0 16,  -8  1 15,  -8  1 16,  -8  1 17,
              -8  2 16,  -8  2 17,  -7  1 15,  -1  7  3,   2  0 10,
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        4.1 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      8.368 Note
              Predicted wR2: Based on SigI**2  2.45 or SHELX Weight 19.84
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          7 Info
```

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
11 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
12 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
8 ALERT type 2 Indicator that the structure model may be wrong or deficient
6 ALERT type 3 Indicator that the structure quality may be low
6 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

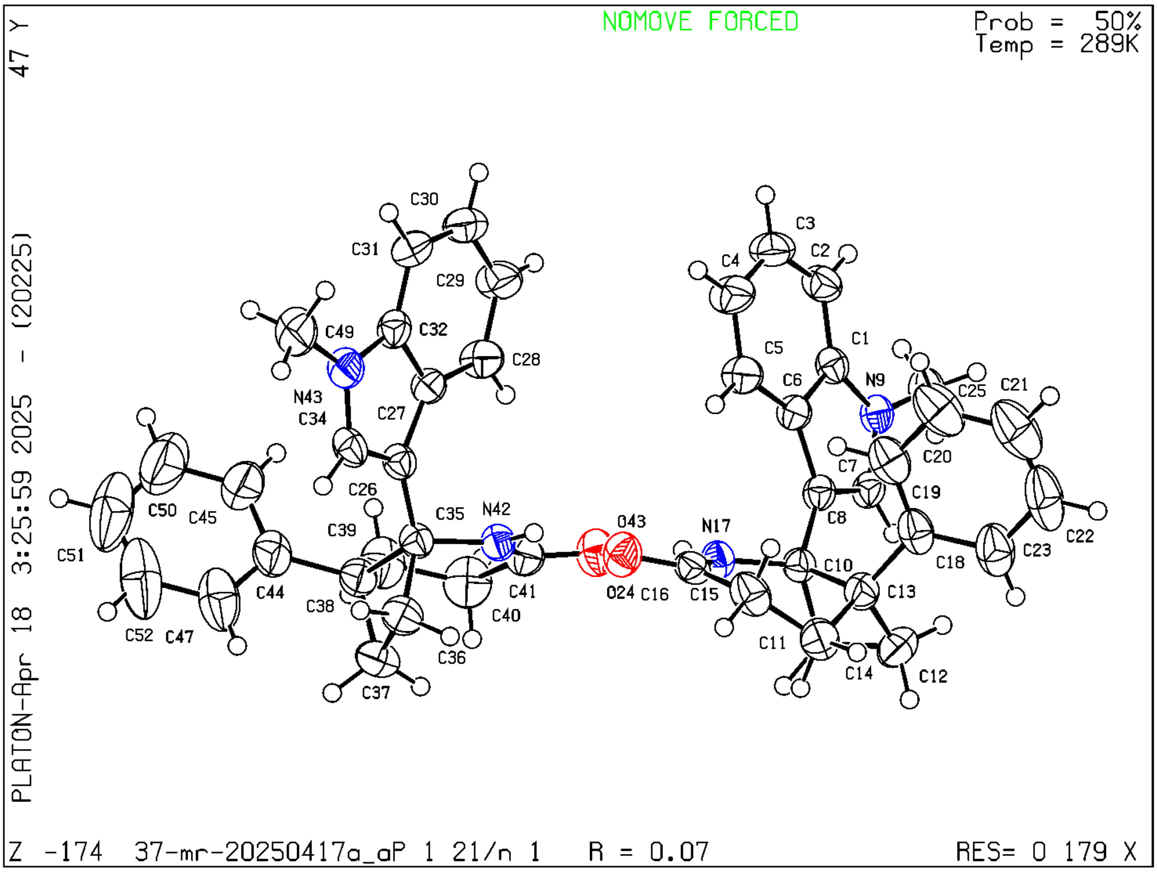
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 02/02/2025; check.def file version of 02/02/2025

Datablock 37-mr-20250417a_auto - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 38-cr-20250904c_auto

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 38-cr-20250904c_auto

```
Bond precision:    C-C = 0.0033 A            Wavelength=1.54184

Cell:              a=31.6097(7)     b=9.6001(2)         c=30.3578(6)
                   alpha=90         beta=107.260(2)     gamma=90
Temperature:       100 K

                  Calculated                    Reported
Volume            8797.4(3)                     8797.4(3)
Space group       C 2/c                         C 1 2/c 1
Hall group        -C 2yc                        -C 2yc
Moiety formula    2(C26 H26 N2 O2), C H Cl3     C26 H26 N2 O2, 0.5(C H Cl3)
Sum formula       C53 H53 Cl3 N4 O4             C26.50 H26.50 Cl1.50 N2 O2
Mr                916.34                        458.17
Dx,g cm-3         1.384                         1.384
Z                 8                             16
Mu (mm-1)         2.312                         2.312
F000              3856.0                        3856.0
F000'             3874.50
h,k,lmax          39,12,38                      39,11,38
Nref              9220                          8925
Tmin,Tmax         0.661,0.723                   0.628,1.000
Tmin'             0.600

Correction method= # Reported T Limits: Tmin=0.628 Tmax=1.000
AbsCorr = MULTI-SCAN
```

Data completeness= 0.968   Theta(max)= 76.373

R(reflections)= 0.0591(7688)   wR2(reflections)= 0.1775(8925)

S = 1.077   Npar= 581

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 C

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ     Please Check
              Calc: C53 H53 Cl3 N4 O4
              Rep.: C26.50 H26.50 Cl1.50 N2 O2
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ     Please Check
              Calc: 2(C26 H26 N2 O2), C H Cl3
              Rep.: C26 H26 N2 O2, 0.5(C H Cl3)
PLAT244_ALERT_4_C Low    'Solvent' Ueq as Compared to Neighbors of         C2 Check
PLAT250_ALERT_2_C Large U3/U1 Ratio for <U(i,j)> Tensor(Resd    1)        2.1 Note
PLAT250_ALERT_2_C Large U3/U1 Ratio for <U(i,j)> Tensor(Resd    2)        2.5 Note
PLAT410_ALERT_2_C Short Intra H...H Contact  H11B     ..H33      .       1.97 Ang.
                                                  x,y,z  =      1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact  H55A     ..H62      .       1.97 Ang.
                                                  x,y,z  =      1_555 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600        115 Report
                1 11  0,   3 11  0,   5 11  0,   6  0  0,  -5 11  1,  -3 11  1,
               -1 11  1,   1 11  1,   3 11  1, -17  9  2, -10 10  2,  -7 11  2,
               -5 11  2,  -3 11  2,  -1 11  2,   1 11  2,   3 11  2,   8 10  2,
               32  0  2, -25  7  3, -10 10  3,  -9 11  3,  -7 11  3,  -5 11  3,
               -3 11  3,  -1 11  3,   1 11  3,   3 11  3,   8 10  3, -25  7  4,
                   (   85 More Missing: see the .ckf listing file)
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite          2 Note
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms .............          1 Report
              H60
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...      0.500 Check
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large      17.26 Why ?
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records          1 Report
PLAT431_ALERT_2_G Short Inter HL..A Contact  Cl4      ..O68      .       2.96 Ang.
                                             -x,y,1/2-z  =      2_555 Check
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd.  #          3 Note
              C H Cl3
PLAT793_ALERT_4_G Model has Chirality at C9          (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C12         (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C21         (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C39         (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C53         (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C56         (Centro SpGr)          R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints ............          1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        160 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........        3.8 Low
```

```
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........      3.425 Note
              Predicted wR2: Based on SigI**2  5.18 or SHELX Weight 16.48
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.        13 Info
```

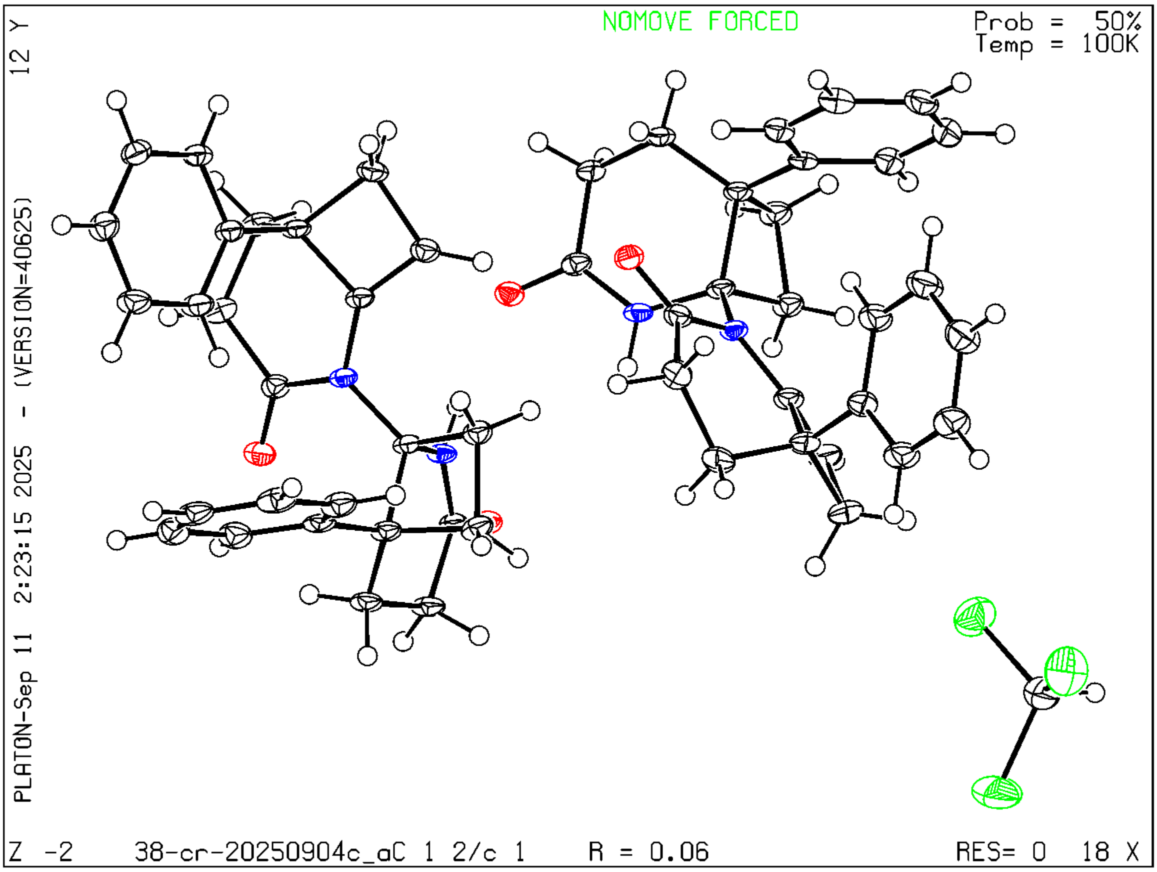
```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   8 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  18 ALERT level G = General information/check it is not something unexpected

   3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   8 ALERT type 2 Indicator that the structure model may be wrong or deficient
   3 ALERT type 3 Indicator that the structure quality may be low
  10 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 04/06/2025; check.def file version of 30/05/2025

Datablock 38-cr-20250904c_auto - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 38-cr-20250908f_auto

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: 38-cr-20250908f_auto

```
Bond precision:   C-C = 0.0019 A          Wavelength=1.54184

Cell:           a=8.5198(2)       b=9.5021(2)       c=16.2326(4)
                alpha=96.133(2)   beta=93.496(2)    gamma=107.290(2)
Temperature:    100 K

                 Calculated                  Reported
Volume           1241.60(5)                  1241.60(5)
Space group      P -1                        P -1
Hall group       -P 1                        -P 1
Moiety formula   C27 H24 N2 O, C H Cl3       C27 H24 N2 O, C H Cl3
Sum formula      C28 H25 Cl3 N2 O            C28 H25 Cl3 N2 O
Mr               511.85                      511.85
Dx,g cm-3        1.369                       1.369
Z                2                           2
Mu (mm-1)        3.528                       3.528
F000             532.0                       532.0
F000'            535.37
h,k,lmax         10,11,20                    10,11,20
Nref             5081                        4984
Tmin,Tmax        0.606,0.703                 0.721,1.000
Tmin'            0.505

Correction method= # Reported T Limits: Tmin=0.721 Tmax=1.000
AbsCorr = MULTI-SCAN
```

```
Data completeness= 0.981          Theta(max)= 74.519

R(reflections)= 0.0343( 4780)                          wR2(reflections)=
                                                       0.0919( 4984)
S = 1.045                  Npar= 331
```

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

```
PLAT410_ALERT_2_B Short Intra H...H Contact  H5B      ..H27       .       1.88 Ang.
                                                      x,y,z   =      1_555 Check
```

Alert level C

```
PLAT244_ALERT_4_C Low    'Solvent' Ueq as Compared to Neighbors of        C28 Check
PLAT420_ALERT_2_C D-H Bond Without Acceptor  N1       --H1        .    Please Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          39 Report
                1  1  0,  -5 10  0,  -4 10  0,  -5 11  0,  -4 11  0,  -3 11  0,
                3-11  1,   4-11  1,   5-11  1,   4-10  1,   6 -2  1,  -7 10  1,
               -6 10  1,  -5 10  1,  -4 10  1,  -5 11  1,  -4 11  1,  -3 11  1,
                3-11  2,   4-11  2,   5-11  2,  -6 10  2,  -5 10  2,  -4 10  2,
               -4 11  2,  -3 11  2,  -2 11  2,   4-11  3,   5-11  3,  -6 10  3,
                   (    9 More Missing: see the .ckf listing file)
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite          5 Note
PLAT003_ALERT_2_G Number of Uiso or U(i,j)  Restrained non-H-Atoms          4 Report
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms .............           1 Report
              H2
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.002 Degree
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records          2 Report
PLAT186_ALERT_4_G The CIF-Embedded .res File Contains ISOR Records          1 Report
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    2)        50% Note
PLAT793_ALERT_4_G Model has Chirality at C4          (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C7          (Centro SpGr)          S Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............         30 Note
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       4.92 Note
                0  0  1,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         57 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........         4.1 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........       5.140 Note
              Predicted wR2: Based on SigI**2  1.79 or SHELX Weight  8.80
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         21 Info
```

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
15 **ALERT level G** = General information/check it is not something unexpected

```
 1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 5 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
 7 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

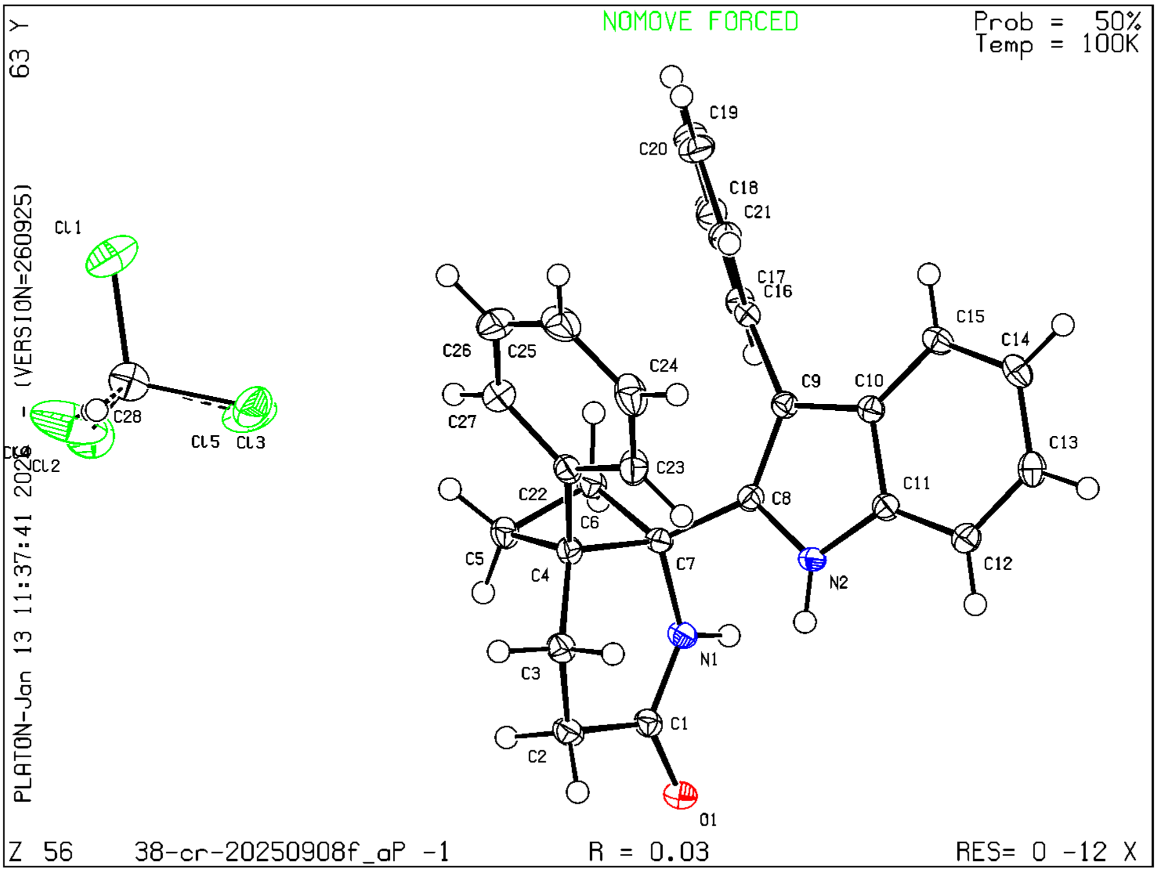
PLATON version of 26/09/2025; check.def file version of 20/09/2025

---

duplicate check

No duplication found

Datablock 38-cr-20250908f_auto - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 37-mr-20251210a_auto

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: 37-mr-20251210a_auto

```
Bond precision:   C-C = 0.0016 A          Wavelength=0.71073

Cell:          a=8.39519(16)       b=10.29504(19)     c=11.8860(2)
               alpha=109.1083(16)  beta=104.1951(16)  gamma=99.0491(15)
Temperature:   100 K
```

	Calculated	Reported
Volume	908.86(3)	908.86(3)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C22 H25 N O4	C22 H25 N O4
Sum formula	C22 H25 N O4	C22 H25 N O4
Mr	367.43	367.43
Dx,g cm-3	1.343	1.343
Z	2	2
Mu (mm-1)	0.092	0.092
F000	392.0	392.0
F000'	392.19	
h,k,lmax	11,14,16	11,14,16
Nref	5102	5017
Tmin,Tmax	0.974,0.980	0.695,1.000
Tmin'	0.969	

```
Correction method= # Reported T Limits: Tmin=0.695 Tmax=1.000
AbsCorr = MULTI-SCAN
```

Data completeness= 0.983   Theta(max)= 29.574

R(reflections)= 0.0395(4308)   wR2(reflections)= 0.1104(5017)

S = 1.064   Npar= 251

```
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 G

```
PLAT793_ALERT_4_G Model has Chirality at C4          (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C5          (Centro SpGr)          S Verify
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       2.59 Note
                0  1  0,   0 -1  1,   0  0  1,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         82 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          3 Note
                0 -1  1,   0  0  1,   0  1  0,
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........        4.5 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........      3.209 Note
              Predicted wR2: Based on SigI**2  3.44 or SHELX Weight 10.38
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         21 Info
```

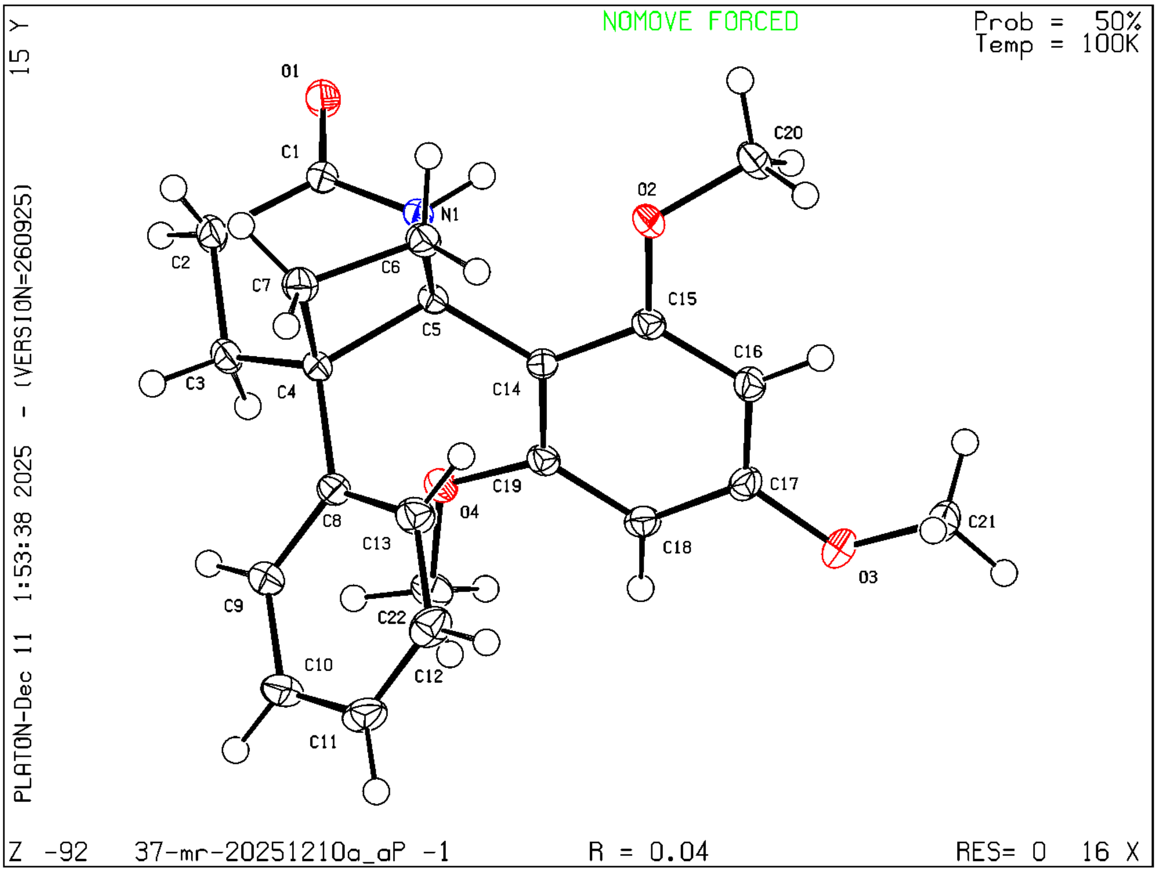
```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   0 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   8 ALERT level G = General information/check it is not something unexpected

   0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   2 ALERT type 2 Indicator that the structure model may be wrong or deficient
   2 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   1 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 26/09/2025; check.def file version of 20/09/2025

Datablock 37-mr-20251210a_auto - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 37-mr-20251208a

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: 37-mr-20251208a

Bond precision:	C-C = 0.0056 A		Wavelength=0.71073
Cell:	a=7.2460(3)	b=12.2378(4)	c=18.5684(8)
	alpha=90	beta=100.138(4)	gamma=90
Temperature:	100 K		

	Calculated	Reported
Volume	1620.85(11)	1620.85(11)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C19 H18 N2 O4	C19 H18 N2 O4
Sum formula	C19 H18 N2 O4	C19 H18 N2 O4
Mr	338.35	338.35
Dx,g cm-3	1.387	1.387
Z	4	4
Mu (mm-1)	0.098	0.098
F000	712.0	712.0
F000’	712.35	
h,k,lmax	9,15,23	9,15,23
Nref	3314	3313
Tmin,Tmax	0.978,0.987	0.515,1.000
Tmin’	0.978	

Correction method= # Reported T Limits: Tmin=0.515 Tmax=1.000
AbsCorr = MULTI-SCAN

```
Data completeness= 1.000            Theta(max)= 26.372

                                                        wR2(reflections)=
R(reflections)= 0.0852( 3068)                           0.1946( 3313)

S = 1.296                    Npar= 230
```

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 C

```
PLAT250_ALERT_2_C Large U3/U1 Ratio for <U(i,j)> Tensor(Resd    1)        2.1 Note
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ..............      0.0056 Ang.
PLAT411_ALERT_2_C Short Inter H...H Contact  H8B      ..H15       .      2.13 Ang.
                                            1-x,1/2+y,1/2-z  =      2_655 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     12.775 Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      3.373 Check
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .          1 Check
                0  0  2,
```

Alert level G

```
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large       6.30 Why ?
PLAT793_ALERT_4_G Model has Chirality at C7          (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C11         (Centro SpGr)          S Verify
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       2.23 Note
                0  1  1,
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          1 Note
                0  1  1,
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........         3.8 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........       5.448 Note
              Predicted wR2: Based on SigI**2  3.57 or SHELX Weight 15.01
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Info
```

---

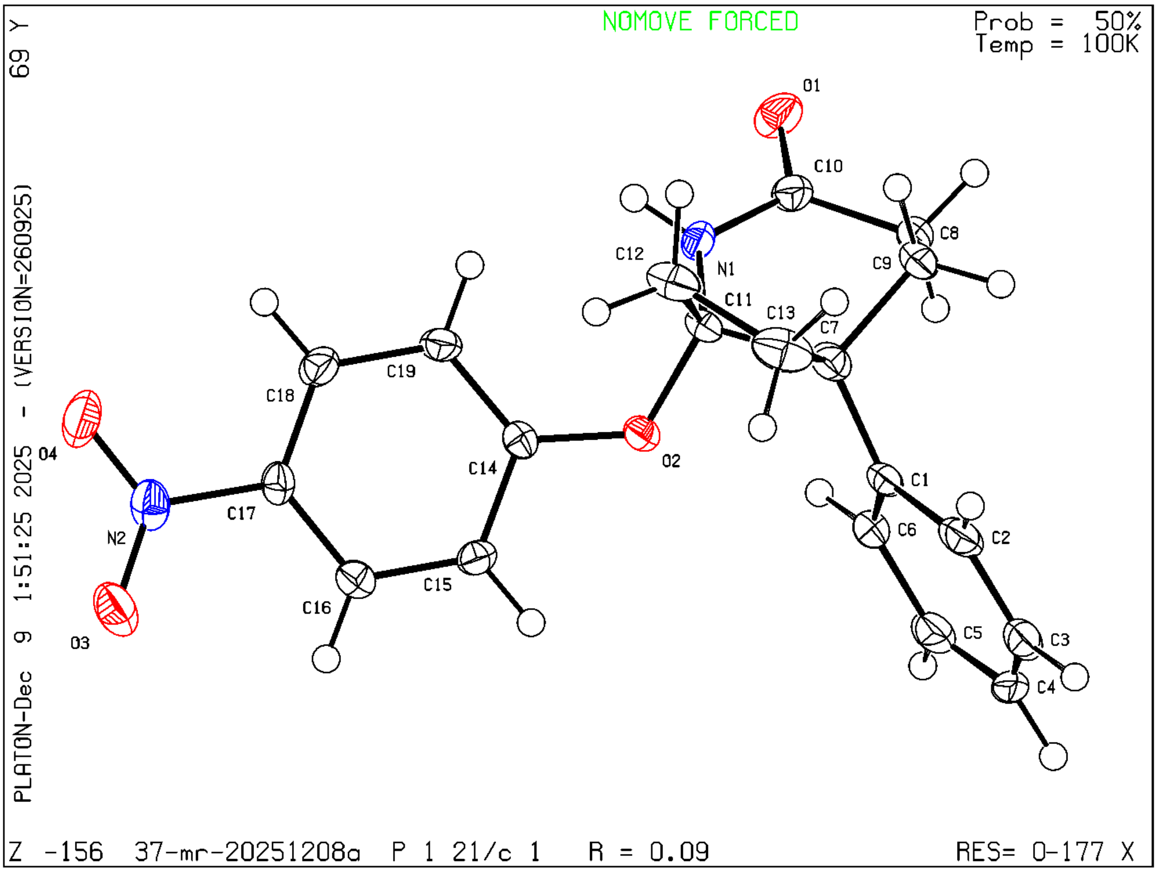
0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
6 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
8 **ALERT level G** = General information/check it is not something unexpected

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
5 ALERT type 2 Indicator that the structure model may be wrong or deficient
6 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
1 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 26/09/2025; check.def file version of 20/09/2025

Datablock 37-mr-20251208a - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 38-cr-20251210a_auto

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: 38-cr-20251210a_auto

```
Bond precision:   C-C = 0.0019 A          Wavelength=1.54184

Cell:            a=8.2063(3)        b=10.2047(3)       c=12.6923(4)
                 alpha=78.524(3)    beta=77.279(3)     gamma=69.660(3)
Temperature:     100 K
```

	Calculated	Reported
Volume	963.41(6)	963.41(6)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	2(C22 H22 N2 O), C D Cl3	C22 H22 N2 O, 0.5(C Cl3 D)
Sum formula	C45 H44 D Cl3 N4 O2	C22.50 H22 Cl1.50 D0.50 N2 O
Mr	781.20	390.60
Dx,g cm-3	1.347	1.346
Z	1	2
Mu (mm-1)	2.501	2.501
F000	410.0	410.0
F000’	412.08	
h,k,lmax	10,12,15	10,12,15
Nref	3986	3868
Tmin,Tmax	0.606,0.705	0.746,1.000
Tmin’	0.549	

```
Correction method= # Reported T Limits: Tmin=0.746 Tmax=1.000
AbsCorr = MULTI-SCAN
```

```
Data completeness= 0.970            Theta(max)= 75.236

R(reflections)= 0.0397( 3550)                          wR2(reflections)=
                                                       0.1071( 3868)
S = 1.066                   Npar= 263
```

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 C

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ     Please Check
              Calc: C45 H44 D Cl3 N4 O2
              Rep.: C22.50 H22 Cl1.50 D0.50 N2 O
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ     Please Check
              Calc: 2(C22 H22 N2 O), C D Cl3
              Rep.: C22 H22 N2 O, 0.5(C Cl3 D)
PLAT410_ALERT_2_C Short Intra H...H Contact  H14B     ..H18      .       1.92 Ang.
                                                     x,y,z  =      1_555 Check
PLAT790_ALERT_4_C Centre of Gravity not Within Unit-Cell: Resd.  #          1 Note
              C22 H22 N2 O
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         20 Report
               -1  0  1,   8  6  7,   1 -1  9,  -1  8 10,   0  9 10,   3  0 11,
               -1  7 11,   0  7 11,  -1  8 11,   0  8 11,   1  9 11,  -1  6 12,
               -1  7 12,   0  7 12,   1  8 12,   2  9 12,   0  6 13,   1  7 13,
                2  8 13,   1  6 14,
```

Alert level G

```
PLAT003_ALERT_2_G Number of Uiso or U(i,j)  Restrained non-H-Atoms          1 Report
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms .............          1 Report
              H2
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...      0.500 Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.003 Degree
PLAT299_ALERT_4_G Atom Site Occupancy Constrained at ............        0.5 Check
              Cl1     Cl2     Cl3     C23     D23
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    2)       100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    2)       2.50 Check
PLAT789_ALERT_4_G Atoms with Negative _atom_site_disorder_group  #          5 Check
PLAT793_ALERT_4_G Model has Chirality at C13         (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C16         (Centro SpGr)          R Verify
PLAT822_ALERT_4_G CIF-embedded .res Contains Negative PART Numbers          1 Check
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         98 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........        3.9 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........      4.353 Note
              Predicted wR2: Based on SigI**2  2.46 or SHELX Weight 10.05
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         11 Info
```

---

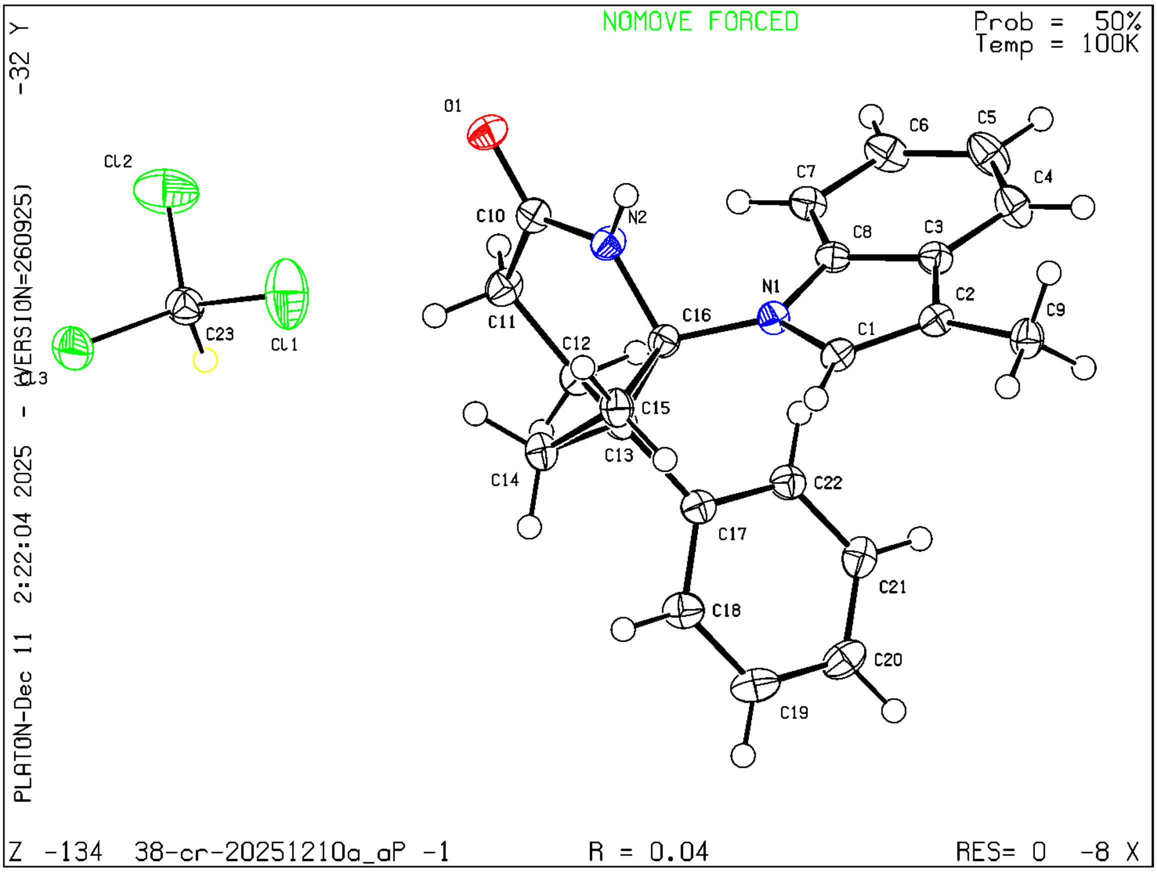
0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
5 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
15 **ALERT level G** = General information/check it is not something unexpected

```
4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
9 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 26/09/2025; check.def file version of 20/09/2025

Datablock 38-cr-20251210a_auto - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 37-mr-20251210b_auto

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 37-mr-20251210b_auto

```
Bond precision:   C-C = 0.0029 A          Wavelength=0.71073

Cell:            a=14.5636(2)     b=19.4453(3)         c=21.1345(2)
                 alpha=90         beta=91.2433(11)     gamma=90
Temperature:     100 K

                Calculated                  Reported
Volume          5983.75(14)                 5983.74(13)
Space group     P 21/n                      P 1 21/n 1
Hall group      -P 2yn                      -P 2yn
Moiety formula  2(C25 H24 N2 O), 5(C D Cl3) 2(C25 H24 N2 O), 5(C Cl3 D)
Sum formula     C55 H48 D5 Cl15 N4 O2       C55 H48 Cl15 D5 N4 O2
Mr              1338.77                     1338.79
Dx,g cm-3       1.486                       1.486
Z               4                           4
Mu (mm-1)       0.734                       0.734
F000            2728.0                      2728.0
F000'           2737.58
h,k,lmax        18,24,26                    18,24,26
Nref            12231                       12220
Tmin,Tmax       0.884,0.922                 0.571,1.000
Tmin'           0.863

Correction method= # Reported T Limits: Tmin=0.571 Tmax=1.000
AbsCorr = MULTI-SCAN
```

Data completeness= 0.999   Theta(max)= 26.371

R(reflections)= 0.0401(10776)   wR2(reflections)= 0.1079(12220)

S = 1.034   Npar= 760

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 C

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ     Please Check
              Calc: C55 H48 D5 Cl15 N4 O2
              Rep.: C55 H48 Cl15 D5 N4 O2
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ     Please Check
              Calc: 2(C25 H24 N2 O), 5(C D Cl3)
              Rep.: 2(C25 H24 N2 O), 5(C Cl3 D)
PLAT244_ALERT_4_C Low    'Solvent' Ueq as Compared to Neighbors of        C54 Check
PLAT410_ALERT_2_C Short Intra H...H Contact  H5B      ..H21      .       1.91 Ang.
                                                x,y,z  =     1_555 Check
PLAT410_ALERT_2_C Short Intra H...H Contact  H30A     ..H50      .       1.91 Ang.
                                                x,y,z  =     1_555 Check
PLAT790_ALERT_4_C Centre of Gravity not Within Unit-Cell: Resd.  #          1 Note
              C25 H24 N2 O
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          8 Report
                1  1  0,   0  2  0,   1  1  1,   0  0  2,   8  2  5,   3  9  7,
                3  8  8,  -4  6  9,
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) .          2 Check
                1  0  1,  -1  1  1,
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         12 Note
PLAT003_ALERT_2_G Number of Uiso or U(i,j)  Restrained non-H-Atoms         19 Report
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          2 Report
              H1      H3
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large       7.16 Why ?
PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing ..............    0.00020 Ang.
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records         13 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records          1 Report
PLAT186_ALERT_4_G The CIF-Embedded .res File Contains ISOR Records          1 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used     0.0100 Report
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    4)       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    8)       100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd    9)       100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    4)       2.80 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    8)       1.14 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd    9)       1.05 Check
PLAT431_ALERT_2_G Short Inter HL..A Contact  Cl6      ..O1       .       3.15 Ang.
                                              1+x,y,z  =     1_655 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  Cl3B     ..C37      .       3.10 Ang.
                                           -x,1-y,1-z  =     3_566 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact  Cl3B     ..C36      .       3.18 Ang.
```

```
                                                -x,1-y,1-z  =      3_566 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl2      ..Cl5B     .       3.26 Ang.
                                        -1/2+x,3/2-y,1/2+z  =      4_576 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl2      ..Cl15     .       3.37 Ang.
                                        -1/2+x,3/2-y,1/2+z  =      4_576 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl9      ..Cl14     .       3.34 Ang.
                                                      x,y,z  =      1_555 Check
PLAT790_ALERT_4_G Centre of Gravity not Within Unit-Cell: Resd.  #          2 Note
              C25 H24 N2 O
PLAT790_ALERT_4_G Centre of Gravity not Within Unit-Cell: Resd.  #          3 Note
              C D Cl3
PLAT790_ALERT_4_G Centre of Gravity not Within Unit-Cell: Resd.  #          7 Note
              C D Cl3
PLAT793_ALERT_4_G Model has Chirality at C4          (Centro SpGr)          S Verify
PLAT793_ALERT_4_G Model has Chirality at C7          (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C29         (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C32         (Centro SpGr)          S Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............        307 Note
PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]=       1.72 Note
               -1  0  1,   0  1  1,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          1 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          3 Note
                0  0  2,   0  2  0,   1  1  0,
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........        4.7 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........      3.684 Note
              Predicted wR2: Based on SigI**2  2.93 or SHELX Weight 10.44
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          5 Info
```

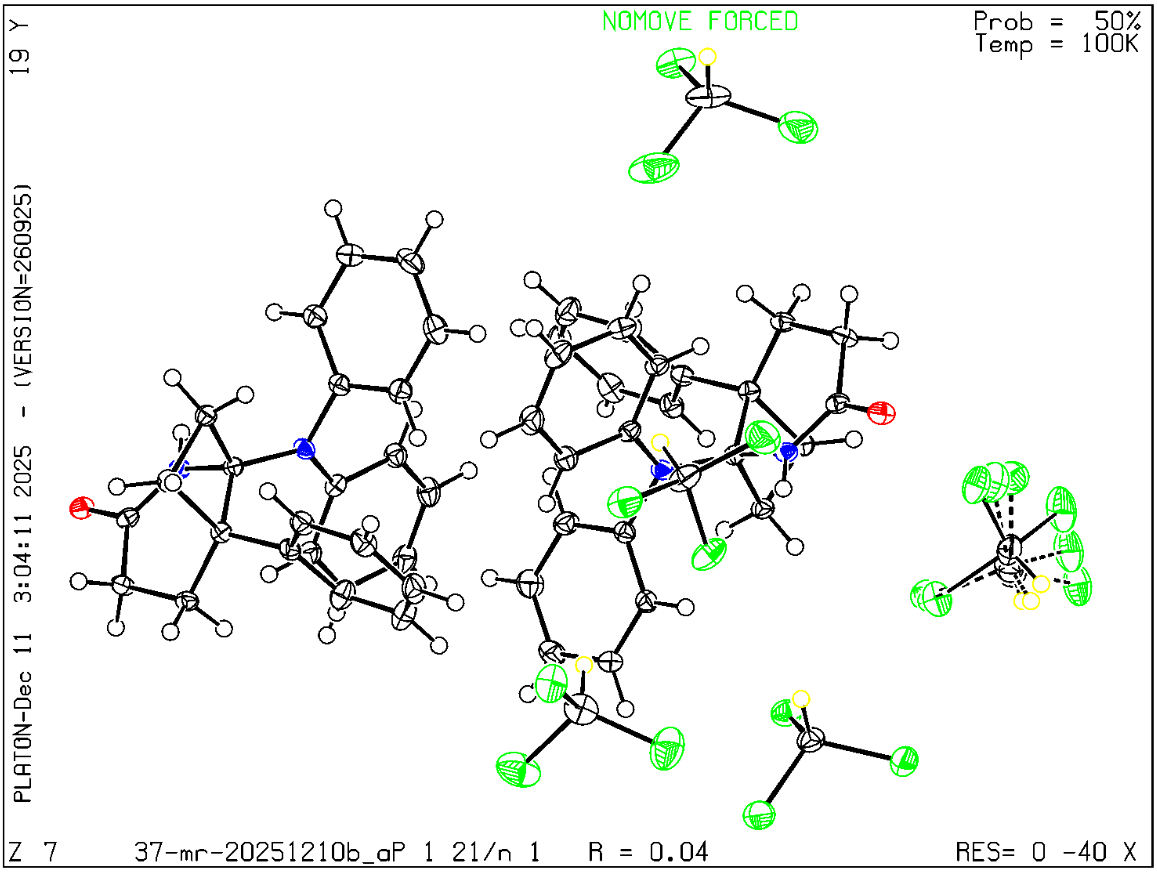
```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   8 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  35 ALERT level G = General information/check it is not something unexpected

   2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  13 ALERT type 2 Indicator that the structure model may be wrong or deficient
   6 ALERT type 3 Indicator that the structure quality may be low
  20 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 26/09/2025; check.def file version of 20/09/2025

Datablock 37-mr-20251210b_auto - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 38-cr-20251205b_auto

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: 38-cr-20251205b_auto

```
Bond precision:    C-C = 0.0030 A           Wavelength=1.54184

Cell:             a=11.0004(2)     b=11.4646(2)         c=13.3602(2)
                  alpha=90         beta=92.6991(16)     gamma=90
Temperature:      100 K

                 Calculated                     Reported
Volume           1683.06(5)                     1683.06(6)
Space group      P 21                           P 1 21 1
Hall group       P 2yb                          P 2yb
Moiety formula   C22 H22 N2 O2                  2(C22 H22 N2 O2)
Sum formula      C22 H22 N2 O2                  C44 H44 N4 O4
Mr               346.42                         692.83
Dx,g cm-3        1.367                          1.367
Z                4                              2
Mu (mm-1)        0.700                          0.700
F000             736.0                          736.0
F000'            738.11
h,k,lmax         13,14,16                       13,14,16
Nref             6999[ 3681]                    6557
Tmin,Tmax        0.874,0.926                    0.783,1.000
Tmin'            0.869

Correction method= # Reported T Limits: Tmin=0.783 Tmax=1.000
AbsCorr = MULTI-SCAN
```

```
Data completeness= 1.78/0.94       Theta(max)= 75.646

R(reflections)= 0.0322( 6368)                            wR2(reflections)=
                                                         0.0860( 6557)
S = 1.044                 Npar= 477
```

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 C

```
PLAT041_ALERT_1_C Calc. and Reported SumFormula    Strings  Differ     Please Check
              Calc: C22 H22 N2 O2
              Rep.: C44 H44 N4 O4
PLAT042_ALERT_1_C Calc. and Reported MoietyFormula Strings  Differ     Please Check
              Calc: C22 H22 N2 O2
              Rep.: 2(C22 H22 N2 O2)
PLAT411_ALERT_2_C Short Inter H...H Contact  H7       ..H37B     .        2.08 Ang.
                                           -x,-1/2+y,1-z  =      2_546 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          3 Report
                4  0 15,  -1  0 16,   0  0 16,
```

Alert level G

```
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite          4 Note
PLAT045_ALERT_1_G Calculated and Reported Z Differ by a Factor ...          2 Check
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records          1 Report
PLAT791_ALERT_4_G Model has Chirality at C10        (Sohncke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C14        (Sohncke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C32        (Sohncke SpGr)          R Verify
PLAT791_ALERT_4_G Model has Chirality at C33        (Sohncke SpGr)          S Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints ............           3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         75 Note
PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File          1 Note
               11  2  9,
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..........       2.953 Note
              Predicted wR2: Based on SigI**2  2.91 or SHELX Weight  8.23
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         20 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by          2 Check
```

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
4 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
13 **ALERT level G** = General information/check it is not something unexpected

3 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
2 ALERT type 3 Indicator that the structure quality may be low
6 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. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 26/09/2025; check.def file version of 20/09/2025

Datablock 38-cr-20251205b_auto - ellipsoid plot