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# Scandium-Catalyzed Highly Selective $N^2$-Alkylation of Benzotriazoles with Cyclohexanones

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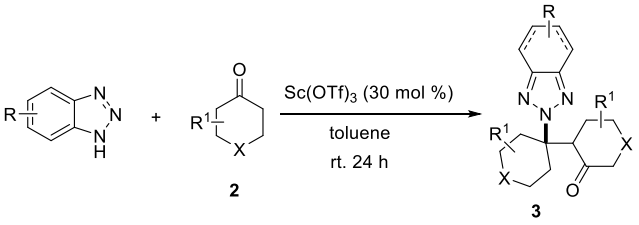
**General information**

All reactions were carried out using oven-dried glassware with magnetic stirring under argon atmosphere unless otherwise noted. Anhydrous solvents were dried prior to use. For column chromatography, 200-300 mesh silica gel was used. Thin layer chromatography (TLC) was performed on Silicycle 250μm silica gel 60Å plates. Visualization was accomplished with UV light (254 nm), Iodine, or Potassium Permanganate. $^{1}$H NMR and $^{13}$C NMR spectra were recorded on a Bruker 300 MHz (300 MHz for $^{1}$H; 282 MHz for $^{19}$F; 75 MHz for $^{13}$C) or 400 MHz (400 MHz for $^{1}$H; 376 MHz for $^{19}$F; 100 MHz for $^{13}$C) spectrometers at ambient temperature. The chemical shifts (δ) are given in parts per million relative to $CDCl_3$ (7.26 ppm for $^{1}$H) or TMS (0 ppm for $^{1}$H) and $CDCl_3$ (77.16 ppm for $^{13}$C). Coupling constants (*J*) are reported in Hz, and multiplicity is described using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, or combinations thereof. HRMS were performed on Agilent 6540 Q-TOF mass spectrometer (ESI). Melting points were determined on a SGW X-4B melting point apparatus.

The substrates of benzotriazoles (**1a**-**1k**) are all known compounds and prepared according to the literature procedures.[1] 4-aryl-triazoles (**4a**-**4f**) prepared according to the literature procedures.[2] Ketones were all commercially available and purchased from Aladdin and Energy Chemical.

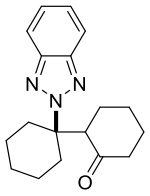
**General procedure for the synthesis and experiment data of compound 3**.




A dried tube was charged with triazoles **1** (0.2 mmol, 1 eq.), ketones **2** (3.2 mmol, 16 eq.) and freshly distilled toluene (2.0 mL). Then $Sc(OTf)_3$ (30.0 mg, 0.06 mmol, 30 mol %) were added. The tube was sealed and stirred at rt. After the reaction was complete (monitored by TLC), the solution was quenched with water (10 mL) and extracted with DCM (3 x 10 mL). The organic layers dried over $Na_2SO_4$ and concentrated by rotary evaporation. The crude product was analyzed by GC, the purified by silica gel column chromatography (EtOAc/PE = 1/10-1/40 or $CH_2Cl_2$/PE =

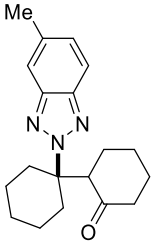
1/1-1/3) to afford the desired products (**3a**-**3s**).

**2-[1-(2*H*-benzotriazol-2-yl)cyclohexyl]cyclohexanone (3a)**



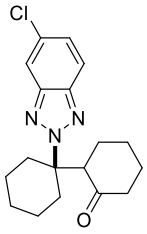
The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), white solid, 54.5 mg, 92% yield, m.p. 148-151$^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.38 (dd, *J* = 6.4, 3.0 Hz, 2H), 3.07 (dd, *J* = 12.6, 4.8 Hz, 1H), 2.88-2.65 (m, 2H), 2.62-2.40 (m, 2H), 2.36-2.24 (m, 2H), 1.98 (d, *J* = 14.4 Hz, 1H), 1.85-1.52 (m, 7H), 1.48-1.29 (m, 2H), 0.97-0.94 (m, 1H), 0.87-0.77 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.3, 143.8, 126.1, 118.4, 71.9, 60.8, 44.2, 34.4, 28.8, 28.3, 28.0, 25.4, 25.0, 22.2, 22.0. HRMS (ESI) calculated for $C_{18}H_{24}N_3O$ $[M+H]^+$: 298.1919, found: 298.1918.

**2-[1-(2*H*-(5-methylbenzotriazol)-2-yl)cyclohexyl]cyclohexanone (3b)**



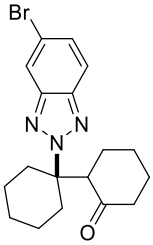
The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), colorless solid, 52.2 mg, 84% yield, m.p. 115-118 $^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.8 Hz, 1H), 7.64 (s, 1H), 7.22 (d, *J* = 8.8 Hz, 1H), 3.04 (dd, *J* = 12.6, 4.8 Hz, 1H), 2.79-2.66 (m, 2H), 2.57-2.41 (m, 2H), 3.50 (s, 3H, -$CH_3$), 2.37-2.25 (m, 2H), 1.98-1.92 (m, 1H), 1.76-1.25 (m, 9H), 0.99-0.91 (m, 1H), 0.86-0.76 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.5, 144.2, 142.4, 136.2, 129.0, 117.9, 116.7, 71.7, 60.8, 44.1, 34.4, 28.8, 28.3, 28.0, 25.4, 25.0, 22.20, 22.18, 22.0. HRMS (ESI) calculated for $C_{19}H_{26}N_3O$ $[M+H]^+$: 312.2076, found: 312.2076.

**2-[1-(2H-(5-chlorobenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3c**)



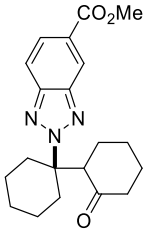
The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 15/1), white solid, 60.4 mg, 91% yield, m.p. 108-100 $^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.81 (d, *J* = 9.0 Hz, 1H), 7.31 (d, *J* = 9.0 Hz, 1H), 3.02 (dd, *J* = 12.4, 4.8 Hz, 1H), 2.70 (t, *J* = 15.8 Hz, 2H), 2.54-2.38 (m, 2H), 2.39-2.24 (m, 2H), 2.04-1.90 (m, 1H), 1.79-1.24 (m, 9H), 0.96-0.93 (m, 1H), 0.80 (td, *J* = 13.2, 3.4 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.0, 144.1, 142.2, 131.9, 127.6, 119.6, 117.5, 72.4, 60.6, 44.1, 34.2, 28.8, 28.2, 28.1, 25.4, 24.9, 22.1, 21.9. HRMS (ESI) calculated for $C_{18}H_{23}N_3OCl$ $[M+H]^+$: 332.1530, found: 332.1532.

**2-[1-(2*H*-(5-bromobenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3d**)



The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 15/1), colorless solid, 60.1 mg, 80% yield, m.p. 110-114 $^{o}$C. $^{1}$H NMR (300 MHz, $CDCl_3$) δ 8.07 (dd, *J* = 1.8, 0.6 Hz, 1H), 7.77 (dd, *J* = 9.0, 0.6 Hz, 1H), 7.45 (dd, *J* = 9.0, 1.8 Hz, 1H), 3.03 (dd, *J* = 12.6, 4.8 Hz, 1H), 2.71 (t, *J* = 13.2 Hz, 2H), 2.56-2.24 (m, 4H), 2.05- 1.94 (m, 1H), 1.79-1.23 (m, 9H), 0.99-0.73 (m, 2H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 210.1, 144.7, 142.4, 130.0, 120.9, 119.9, 72.4, 60.7, 44.1, 34.2, 28.8, 28.3, 28.2, 25.4, 24.9, 22.2, 21.9. HRMS (ESI) calculated for $C_{18}H_{23}N_3OBr$ $[M+H]^+$: 376.1024, found: 376.1028.

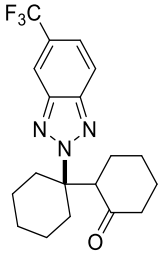
**2-[1-(2*H*-(5-methyloxycarbonylbenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3e**)



The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 4/1), colorless oil, 41.2 mg, 58% yield, m.p. 123-127 $^{o}$C.$^{1}$H NMR (400 MHz, $CDCl_3$)

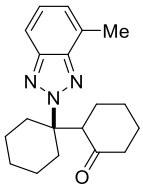
δ 8.70 (s, 1H), 8.01 (d, *J* = 9.0 Hz, 1H), 7.90 (d, *J* = 9.0 Hz, 1H), 3.96 (s, 3H), 3.06 (dd, *J* = 12.6, 4.4 Hz, 1H), 2.74 (t, *J* = 15.8 Hz, 2H), 2.50-2.44 (m, 2H), 2.31 (t, *J* = 13.8 Hz, 2H), 2.05-1.91 (m, 1H), 1.86-1.19 (m, 9H), 0.96 (d, *J* = 12.0 Hz, 1H), 0.81 (q, *J* = 12.8 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.0, 167.0, 145.7, 143.2, 128.1, 126.2, 122.2, 118.4, 72.8, 60.7, 52.5, 44.1, 34.2, 28.8, 28.2, 25.4, 24.9, 22.1, 21.9. HRMS (ESI) calculated for $C_{20}H_{26}N_3O_3$ $[M+H]^+$: 356.1974, found: 356.1979.

**2-[1-(2*H*-(5-Trifuloromethylbenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3f**)




The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 49.8 mg, 68% yield, m.p. 100-104 $^{o}$C. $^{1}$H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, *J* = 0.6 Hz, 1H), 8.00 (dd, *J* = 9.0, 0.6 Hz, 1H), 7.56 (dd, *J* = 9.0, 1.5 Hz, 1H), 3.07 (dd, *J* = 12.6, 4.8 Hz, 1H), 2.74 (t, *J* = 12.6 Hz, 2H), 2.51-2.45 (m, 2H), 2.37-2.23 (m, 2H), 2.04-1.85 (m, 1H), 1.78-1.23 (m, 9H), 1.04-0.94 (m, 1H), 0.89-0.75 (m, 1H). $^{19}$F NMR (282 MHz, $CDCl_3$) δ -62.18. $^{13}$C NMR (75 MHz, $CDCl_3$) δ 209.9, 144.7, 142.6, 128.4 (q, *J* = 32.3 Hz), 124.3 (q, *J* = 272.3 Hz), 122.3 (q, *J* = 2.9 Hz), 119.7, 117.2 (q, *J* = 4.9 Hz), 72.9, 60.7, 44.1, 34.2, 28.9, 28.3, 28.3, 25.5, 24.9, 22.1, 21.9. HRMS (ESI) calculated for $C_{19}H_{23}F_3N_3O$ $[M+H]^+$: 366.1793, found: 366.1790.

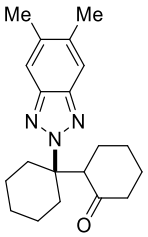
**2-[1-(2*H*-(4-methylbenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3g**)




The title compound was prepared via general procedure, silica gel column chromatography (PE/DCM = 4/1), white solid, 35.0 mg, 56% yield, m.p. 170-172 $^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.6 Hz, 1H), 7.27 (t, *J* = 7.8 Hz, 1H), 7.11 (d, *J* = 6.8 Hz, 1H), 3.08 (dd, *J* = 12.6, 4.8 Hz, 1H), 2.84-2.67 (m, 2H), 2.67 (s, 3H), 2.57-2.41 (m, 2H), 2.38-2.30 (m, 2H), 2.05-1.93 (m, 1H), 1.78-1.22 (m, 9H), 0.97 (d, *J* = 12.4 Hz, 1H), 0.88-0.74 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ

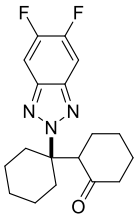
210.6, 144.3, 143.7, 129.2, 126.2, 125.0, 115.5, 71.7, 60.7, 44.2, 34.4, 28.8, 28.3, 28.0, 25.4, 25.0, 22.2, 22.0, 17.4. HRMS (ESI) calculated for $C_{19}H_{26}N_3O$ $[M+H]^+$: 312.2076, found: 312.2074.

**2-[1-(2*H*-(5,6-Dimethylbenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3h**)



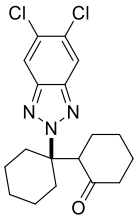

The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 44.9 mg, 69% yield, m.p. 146-149 $^oC$. $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.63 (s, 2H), 3.02 (dd, *J* = 12.6, 4.8 Hz, 1H), 2.72 (t, *J* = 14.1 Hz, 2H), 2.57-2.37 (m, 2H), 2.40 (s, 6H, $-CH_3$), 2.34-2.27 (m, 2H), 2.02-1.89 (m, 1H), 1.77-1.49 (m, 7H), 1.47-1.24 (m, 2H), 0.98-0.68 (m, 2H). $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 210.5, 143.1, 136.5, 116.9, 71.4, 60.8, 44.1, 34.4, 28.7, 28.3, 28.0, 25.4, 25.1, 22.2, 22.0, 21.0. HRMS (ESI) calculated for $C_{20}H_{28}N_3O$ $[M+H]^+$: 326.2232, found: 326.2234.

**2-[1-(2*H*-(5,6-Dimethylbenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3i**)



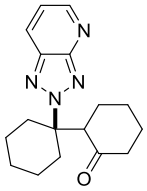

The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), colorless solid, 46.7 mg, 70% yield, m.p. 170-173 $^oC$. $^1H$ NMR (400 MHz, $CDCl_3$) δ 7.60 (t, *J* = 8.4 Hz, 2H), 3.01 (dd, *J* = 12.8, 4.8 Hz, 1H), 2.68 (t, *J* = 15.2 Hz, 2H), 2.51-2.37 (m, 2H), 2.37-2.21 (m, 2H), 2.02-1.91 (m, 1H), 1.79-1.22 (m, 9H), 0.95 (d, *J* = 13.2 Hz, 1H), 0.90-0.72 (m, 1H). $^{19}F$ NMR (282 MHz, $CDCl_3$) δ -134.16. $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 210.0, 151.3 (dd, $J_{C\text{-}F1,C\text{-}F2}$ = 251.4, 19.1 Hz), 139.5 (d, *J* = 6.0 Hz), 103.9 (dd, $J_{C\text{-}F1,C\text{-}F2}$= 15.4, 6.9 Hz), 72.4, 60.7, 44.1, 34.2, 28.8, 28.2, 25.4, 24.9, 22.1, 21.9. HRMS (ESI) calculated for $C_{18}H_{22}F_2N_3O$ $[M+H]^+$: 334.1731, found: 334.1732.

**2-[1-(2*H*-(5,6-Dichlorobenzotriazol)-2-yl)cyclohexyl]cyclohexanone** (**3j**)



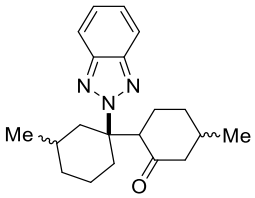
The title compound was prepared via general procedure, silica gel column chromatography ($^t$BuOMe/PE = 1/5), colorless solid, 55.5 mg, 76% yield, m.p. 131-135 $^o$C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 2H), 3.02 (dd, *J* = 12.8, 4.8 Hz, 1H), 2.69 (t, *J* = 14.4 Hz, 2H), 2.52-2.44 (m, 2H), 2.36-2.17 (m, 2H), 1.98 (d, *J* = 13.2 Hz, 1H), 1.81-1.25 (m, 9H), 0.96 (d, *J* = 13.2 Hz, 1H), 0.90-0.71 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 209.9, 142.6, 130.9, 119.4, 72.8, 60.6, 44.1, 34.2, 28.8, 28.3, 28.2, 25.4, 24.9, 22.1, 21.9. HRMS (ESI) calculated for $C_{18}H_{22}N_3OCl_2$ $[M+H]^+$: 366.1140, found: 366.1148.

**2-[1-(2*H*-(triazolopyridine)-2-yl)cyclohexyl]cyclohexanone** (**3k**)



The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 37.1 mg, 62% yield, m.p. 113-116 $^o$C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, *J* = 4.0 Hz, 1H), 8.27 (d, *J* = 8.4 Hz, 1H), 7.36 (dd, *J* = 8.4, 4.0 Hz, 1H), 3.13 (dd, *J* = 12.8, 4.8 Hz, 1H), 2.78 (t, *J* = 13.2 Hz, 2H), 2.60-2.49 (m, 2H), 2.36-2.28 (m, 2H), 2.05-1.93 (m, 1H), 1.81-1.25 (m, 9H), 1.04-0.99 (m, 1H), 0.91-0.78 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.0, 155.4, 151.7, 135.7, 127.5, 121.9, 73.1, 60.6, 44.1, 34.0, 28.8, 28.3, 28.0, 25.4, 24.9, 22.1, 21.9. HRMS (ESI) calculated for $C_{17}H_{23}N_4O$ $[M+H]^+$: 299.1872, found: 299.1873.

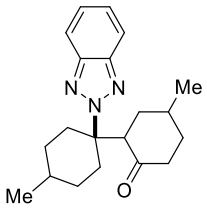
**2-[1-(2*H*-benzotriazol-2-yl)-3-methyl-cyclohexyl]-5-methyl-cyclohexanone** (**3l**)



The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 33.8 mg, 52% yield, m.p. 152-155 $^o$C. dr = 1:1, $^1$H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.46-7.31 (m,2H), 3.04 (dd, *J* = 13.2, 4.4 Hz, 1H),

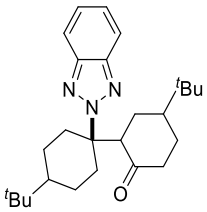
2.84-2.59 (m, 2H), 2.55-2.26 (m, 2H), 2.24-1.98 (m, 2H), 1.86-1.46 (m, 6H), 1.26-0.82 (m, 10H). $^{13}$C NMR (10 MHz, $CDCl_3$) δ 209.62, 209.55, 143.72, 126.10, 118.44, 72.59, 72.54, 60.09, 60.00, 52.34, 52.32, 42.48, 36.25, 36.17, 36.05, 34.14, 34.12, 33.88, 33.86, 33.81, 28.19, 27.91, 27.79, 27.71, 27.22, 22.74, 22.39, 22.27, 22.05, 22.02. HRMS (ESI) calculated for $C_{20}H_{26}N_3O$ $[M+H]^+$: 326.2232, found: 326.2230.

**2-[1-(2*H*-benzotriazol-2-yl)-4-methyl-cyclohexyl]-4-methyl-cyclohexanone (3m)**



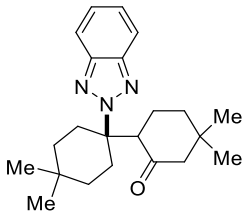

The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), colorless solid, 49.6 mg, 76% yield, m.p. 166-167 $^{o}$C. dr = 11:1, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 6.4, 2.8 Hz, 2H), 7.38 (dd, *J* = 6.4, 2.8 Hz, 2H), 3.16 (dd, *J* = 13.2, 4.4 Hz, 1H), 2.89-2.66 (m, 2H), 2.63-2.22 (m, 4H), 1.99-1.83 (m, 1H), 1.77-1.44 (m, 4H), 1.43-1.17 (m, 3H), 0.95-0.84 (m, 1H), 0.76 (d, *J* = 6.4 Hz, 3H,-$CH_3$), 0.73 (d, *J* = 6.3 Hz, 3H, -$CH_3$), 0.50-0.40 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.5, 143.7, 126.1, 118.5, 71.5, 59.3, 43.4, 37.0, 36.8, 34.2, 32.2, 31.5, 30.7, 27.8, 22.4, 21.2. HRMS (ESI) calculated for $C_{20}H_{26}N_3O$ $[M+H]^+$: 326.2232, found: 326.2230.

**2-[1-(2*H*-benzotriazol-2-yl)-4-tert-butyl-cyclohexyl]-4-tert-butyl-cyclohexanone (3n)**



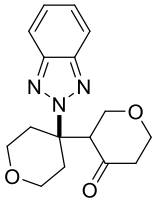

The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 59.0 mg, 72% yield, m.p. 232-234 $^{o}$C. $^{1}$H NMR (300 MHz, $CDCl_3$) δ 7.94-7.85 (m, 2H), 7.41-7.32 (m, 2H), 3.09 (dd, *J* = 12.3, 4.5 Hz, 1H), 2.89-2.78 (m, 2H), 2.59-2.37 (m, 2H), 2.35-2.27 (m, 2H), 1.98 (dd, *J* = 7.8, 4.2 Hz, 1H), 1.85-1.74 (m, 1H), 1.69-1.57 (m, 1H), 1.50-1.21 (m, 6H), 0.96-0.82 (m, 1H), 0.72 (s, 9H), 0.63 (s, 9H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 210.7, 143.7, 126.1, 118.3, 71.8, 59.7, 47.2, 46.8, 43.1, 34.4, 32.4, 32.4, 29.7, 28.9, 28.6, 27.5, 27.4, 23.2, 23.0. HRMS (ESI) calculated for $C_{26}H_{40}N_3O$ $[M+H]^+$: 410.3171, found: 410.3177.

**2-[1-(2*H*-benzotriazol-2-yl)-4-dimethylcyclohexyl]-5-dimethyl-cyclohexanone** (**3o**)



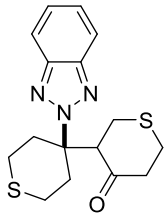

The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), colorless solid, 49.5 mg, 70% yield, m.p. 173-174 $^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.38 (dd, *J* = 6.4, 3.0 Hz, 2H), 3.30 (dd, *J* = 13.8, 4.4 Hz, 1H), 2.79 (td, *J* = 13.8, 3.6 Hz, 1H), 2.64-2.42 (m, 4H), 2.21 (dt, *J* = 13.8, 3.6 Hz, 1H), 1.67-1.52 (m, 4H), 1.45-1.32 (m, 1H), 1.33-1.18 (m, 1H), 1.14 (s, 3H), 0.87 (s, 3H), 0.80 (s, 3H), 0.76 (s, 3H), 0.77-0.66 (m, 1H), 0.42 (d, *J* = 13.2 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 211.1, 143.7, 126.1, 118.5, 71.4, 55.8, 41.2, 40.3, 40.3, 35.05, 35.01, 32.5, 31.3, 30.8, 30.6, 29.8, 24.7, 24.2, 24.0. HRMS (ESI) calculated for $C_{22}H_{32}N_3O$ $[M+H]^+$: 354.2545, found: 354.2540.

**2-[1-(2*H*-benzotriazol-2-yl)-4-oxocyclohexanyl]-4-oxocyclohexanone** (**3p**)



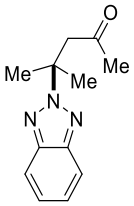

The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 3/1), colorless solid, 49.4 mg, 82% yield, m.p. 127-130 $^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.43 (dd, *J* = 6.4, 3.0 Hz, 2H), 4.04-3.93 (m, 2H), 3.88 (dd, *J* = 12.0, 3.6 Hz, 1H), 3.81 (dd, *J* = 11.6, 8.4 Hz, 1H), 3.74-3.64 (m, 1H), 3.55 (t, *J* = 11.6 Hz, 1H), 3.34 (dd, *J* = 11.6, 5.6 Hz, 1H), 3.16 (dd, *J* = 12.0, 6.0 Hz, 1H), 3.09 (d, *J* = 11.8 Hz, 1H), 2.86 (d, *J* = 14.0 Hz, 2H), 2.79-2.53 (m, 3H), 2.47 (dt, *J* = 14.4, 4.4 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 205.0, 144.0, 126.8, 118.5, 68.29, 68.27, 67.6, 63.99, 63.86, 60.3, 43.8, 34.4, 31.1. HRMS (ESI) calculated for $C_{16}H_{20}N_3O_3$ $[M+H]^+$: 302.1505, found: 302.1501.

**2-[1-(2*H*-benzotriazol-2-yl)-4-thioheterocyclohexanyl]-4-thioheterocyclohexanone** (**3q**)



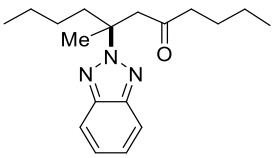
The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 3/1), colorless solid, 49.0 mg, 77% yield, m.p. 133-137 $^{o}$C. $^{1}$H NMR (300 MHz, $CDCl_3$) δ 8.00-7.76 (m, 2H), 7.53-7.34 (m, 2H), 3.40 (dd, *J* = 11.5, 4.2 Hz, 1H), 3.18-2.55 (m, 11H), 2.49-2.29 (m, 2H), 2.08 (ddd, *J* = 13.6, 4.1, 2.6 Hz, 1H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 207.46, 144.02, 126.83, 118.50, 70.59, 63.71, 45.89, 35.12, 31.04, 30.99, 30.69, 24.05, 23.99. HRMS (ESI) calculated for $C_{16}H_{20}N_3S_2O_1$ $[M+H]^+$: 333.0970, found: 333.0975.

**4-[2*H*-benzotriazol-2-yl)-4-methylpentan-2-one (3r)**



The title compound was prepared via general procedure, acetone (2.0 mL) instead of toluene as solvent, silica gel column chromatography (PE/EtOAc = 8/1), colorless solid, 29.1 mg, 67% yield, m.p. 50-52 $^{o}$C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 6.4, 3.2 Hz, 2H), 7.36 (dd, *J* = 6.4, 3.2 Hz, 2H), 3.35 (s, 1H), 2.04 (s, 3H), 1.91 (s, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 205.2, 143.9, 126.2, 118.2, 65.6, 53.7, 31.3, 28.2. HRMS (ESI) calculated for $C_{12}H_{16}N_3O$ $[M+H]^+$: 218.1293, found: 218.1290.

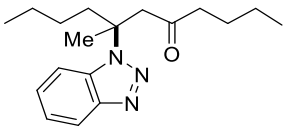
**7-[2*H*-benzotriazol-2-yl)-7-methyl-undecan-5-one** (**3s**)



The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 5/1), afford the colorless oil **3s** (32.2 mg, 50% yield) and **3s'** (13.1 mg, 20% yield) **3y**: $^{1}$H NMR (300 MHz, $CDCl_3$) δ 8.01-7.72 (m, 2H), 7.43-7.27 (m, 2H), 3.51 (d, *J* = 16.5 Hz, 1H), 3.15 (d, *J* = 16.5 Hz, 1H), 2.36-2.06 (m, 4H), 1.97 (s, 3H), 1.52-1.40 (m, 2H), 1.28-1.07 (m, 5H), 0.95-0.85 (m, 1H), 0.83 (t, *J* = 7.2 Hz, 3H), 0.81 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$)

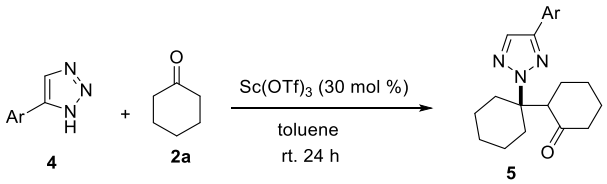
δ 207.7, 143.8, 126.1, 118.3, 68.4, 51.5, 43.9, 41.9, 25.8, 25.7, 24.5, 22.8, 22.3, 14.0, 13.9. HRMS (ESI) calculated for $C_{18}H_{28}N_3O$ $[M+H]^+$: 302.2232, found: 302.2230.

**7-[1*H*-benzotriazol-1-yl)-7-methyl-undecan-5-one (3s')**



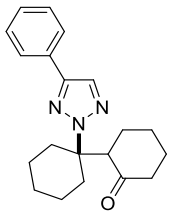

**3s'**: $^1$H NMR (300 MHz, $CDCl_3$) δ 8.06 (dt, *J* = 8.1, 0.9 Hz, 1H), 7.78-7.68 (m, 1H), 7.44 (ddd, *J* = 8.4, 6.9, 1.2 Hz, 1H), 7.34 (ddd, *J* = 8.0, 6.9, 1.0 Hz, 1H), 3.58 (d, *J* = 16.5 Hz, 1H), 3.13 (d, *J* = 16.5 Hz, 1H), 2.40-2.24 (m, 3H), 2.19-2.09 (m, 1H), 2.06 (s, 3H), 1.51-1.37 (m, 2H), 1.31-1.14 (m, 4H), 1.08-0.92 (m, 2H), 0.84 (t, *J* = 7.2 Hz, 3H), 0.78 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 207.8, 146.9, 132.2, 126.9, 123.6, 120.5, 112.0, 64.9, 51.2, 44.0, 40.7, 25.7, 24.8, 22.7, 22.2, 13.94, 13.91. HRMS (ESI) calculated for $C_{18}H_{28}N_3O$ $[M+H]^+$: 302.2232, found: 302.2230.

**General procedure for the synthesis and experiment data of compound 5**.




A dried tube was charged with triazoles **4** (0.2 mmol, 1 eq.), cyclohexanone **2a** (3.2 mmol, 16 eq.) and freshly distilled toluene (2.0 mL). Then $Sc(OTf)_3$ (30.0 mg, 0.06 mmol, 30 mol %) were added. The tube was sealed and stirred at rt. After the reaction was complete (monitored by TLC), the solution was quenched with water (10 mL) and extracted with DCM (3 x 10 mL). The organic layers dried over $Na_2SO_4$ and concentrated by rotary evaporation. The crude product was analyzed by GC, the purified by silica gel column chromatography (PE/EtOAc = 20/1-6/1) to afford the desired products (**5a-5f**).

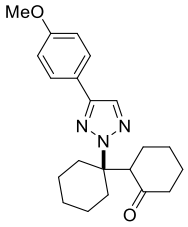
**2-[1-(2*H*-(4-Phenyl-1,2,3-triazole)-2-yl)cyclohexyl]cyclohexanone** (**5a**)




The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 51.8 mg, 80% yield, m.p. 108-111 $^o$C. $^1$H NMR (400 MHz,

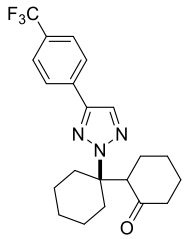
$CDCl_3$) δ 7.87 (s, 1H), 7.82 (d, *J* = 7.8 Hz, 2H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 1H), 2.92 (dd, *J* = 12.0, 4.8 Hz, 1H), 2.67-2.52 (m, 2H), 2.45-2.24 (m, 4H), 1.98-1.96 (d, *J* = 11.2 Hz, 1H), 1.73-1.52 (m, 7H), 1.47-1.36 (m, 2H), 1.16 (d, *J* = 12.4 Hz, 1H), 0.95 (q, *J* = 12.8 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 210.8, 146.8, 131.0, 130.2, 128.9, 128.3, 126.0, 69.8, 60.5, 44.1, 34.0, 28.7, 28.2, 28.0, 25.3, 25.1, 22.2, 21.9. HRMS (ESI) calculated for $C_{20}H_{26}N_3O$ $[M+H]^+$: 324.2076, found: 324.2082.

**2-[1-(2*H*-(4-(4-methoxyphenyl-1,2,3-triazole)-2-yl)cyclohexyl]cyclohexanone** (**5b**)




The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 6/1), colorless solid, 46.1 mg, 65% yield, m.p. 82-85 $^o$C. $^1$H NMR (300 MHz, $CDCl_3$) δ 7.79 (s, 1H), 7.75 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.7 Hz, 2H), 3.85 (s, 3H), 2.91 (dd, *J* = 12.0, 5.1 Hz, 1H), 2.59 (t, *J* = 14.7 Hz, 2H), 2.41-2.28 (m, 4H), 2.00-1.87 (m, 1H), 1.67-1.53 (m, 7H), 1.48-1.34 (m, 2H), 1.21-1.06 (m, 1H), 1.00-0.85 (m, 1H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 210.9, 159.8, 146.7, 129.7, 127.3, 123.8, 114.3, 69.6, 60.6, 55.5, 44.1, 34.0, 28.7, 28.3, 28.0, 25.3, 25.2, 22.2, 21.9. HRMS (ESI) calculated for $C_{21}H_{28}N_3O_2$ $[M+H]^+$: 354.2182, found: 354.2188.

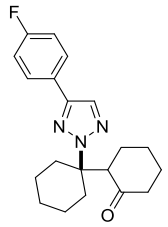
**2-[1-(2*H*-(4-(4-trifuloromethylphenyl-1,2,3-triazole)-2-yl)cyclohexyl]cyclohexanone** (**5c**)




The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 10/1), colorless solid, 56.4 mg, 72% yield, m.p. 111-114 $^o$C. $^1$H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.1 Hz, 2H), 7.93 (s, 1H), 7.68 (d, *J* = 8.1 Hz, 2H), 2.93 (dd, *J* = 12.3, 4.8 Hz, 1H), 2.60 (t, *J* = 14.4 Hz, 2H), 2.47-2.27 (m, 4H), 2.06-1.93 (m, 1H), 1.75-1.35 (m, 9H), 1.22-1.08 (m, 1H), 1.03-0.88 (m, 1H), $^{19}$F NMR (282 MHz, $CDCl_3$) δ -62.57. $^{13}$C NMR (75 MHz, $CDCl_3$) δ 210.6, 145.5, 134.5, 130.7, 130.1 (q, *J* = 32.4 Hz), 126.2, 125.9 (q, *J* = 3.8 Hz), 124.2 (q, $J_{CF3}$ = 271.8 Hz), 70.2, 60.5, 44.1, 33.9, 28.7, 28.2, 28.1, 25.4, 25.1, 22.1, 21.9. HRMS (ESI)

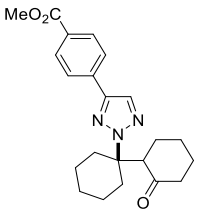
calculated for $C_{21}H_{25}F_3N_3O$ $[M+H]^+$: 392.1950, found: 392.1953.

**2-[1-(2*H*-(4-(*p*-fulorophenyl-1,2,3-triazole)-2-yl)cyclohexyl]cyclohexanone (5d)**



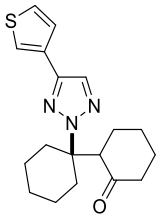
The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), colorless solid, 51.2 mg, 75% yield, m.p. 136-138 $^oC$. $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.82-7.76 (m, 2H), 7.24-6.96 (m, 2H), 2.91 (dd, *J* = 12.1, 4.9 Hz, 1H), 2.59 (t, *J* = 14.7 Hz, 2H), 2.44-2.26 (m, 4H), 2.03-1.88 (m, 1H), 1.72-1.53 (m, 7H), 1.50-1.37 (m, 2H), 1.22-1.12 (m, 1H), 1.02-0.85 (m, 1H). $^{19}F$ NMR (282 MHz, $CDCl_3$) δ -113.51. $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 210.8, 162.8 (d, $J_{CF}$ = 247.3 Hz), 146.0, 130.0, 127.7 (d, *J* = 8.1 Hz), 127.2 (d, *J* = 3.3 Hz), 115.9 (d, *J* = 21.7 Hz), 69.8, 60.5, 44.1, 33.9, 28.7, 28.3, 28.0, 25.4, 25.1, 22.1, 21.9. HRMS (ESI) calculated for $C_{20}H_{25}FN_3O$ $[M+H]^+$: 342.1982, found: 342.1985.

**2-[1-(2*H*-(4-(*p*-methoxycarbonylphenyl-1,2,3-triazole)-2-yl)-cyclohexyl]-cyclohexanone** (**5e**)



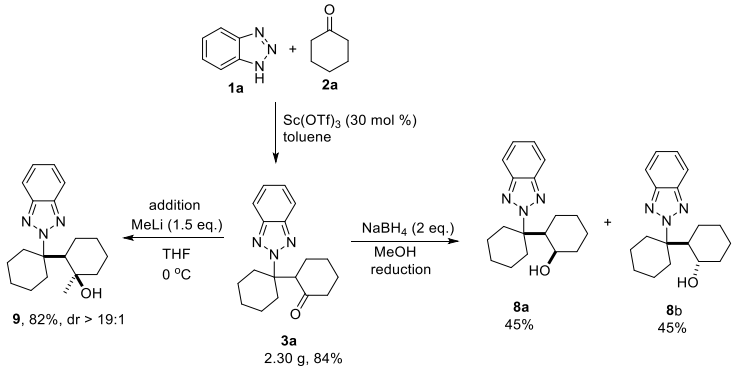
The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 6/1), colorless solid, 49.8 mg, 65% yield, m.p. 114-115 $^oC$. $^1H$ NMR (300 MHz, $CDCl_3$) δ 8.15-8.03 (m, 2H), 7.94 (s, 1H), 7.92-7.86 (m, 2H), 3.95 (s, 3H), 2.93 (dd, *J* = 12.3, 4.8 Hz, 1H), 2.60 (t, *J* = 14.7 Hz, 2H), 2.46-2.24 (m, 4H), 2.10-1.88 (m, 1H), 1.73-1.54 (m, 7H), 1.50-1.33 (m, 2H), 1.29-1.11 (m, 1H), 1.01-0.88 (m, 1H). $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 210.7, 166.9, 145.8, 135.4, 130.9, 130.3, 129.7, 125.8, 70.2, 60.5, 52.3, 44.1, 33.9, 28.7, 28.3, 28.1, 25.4, 25.1, 22.1, 21.9. HRMS (ESI) calculated for $C_{22}H_{28}N_3O_3$ $[M+H]^+$: 382.2131, found: 382.2133.

**2-[1-(2*H*-(4-(2-thiophene-1,2,3-triazole)-2-yl)cyclohexyl]cyclohexanone** (**5f**)



The title compound was prepared via general procedure, silica gel column chromatography (PE/EtOAc = 20/1), yellow oil, 50.0 mg, 76% yield. $^{1}$H NMR (300 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.63 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.48 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.39 (dd, *J* = 5.1, 3.0 Hz, 1H), 2.91 (dd, *J* = 12.0, 5.1 Hz, 1H), 2.58 (t, *J* = 14.7 Hz, 2H), 2.43-2.27 (m, 4H), 2.03-1.86 (m, 1H), 1.68-1.35 (m, 9H), 1.22-1.11 (m, 1H), 1.00-0.85 (m, 1H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 210.8, 143.1, 132.4, 130.3, 126.4, 126.2, 121.3, 69.7, 60.5, 44.1, 33.9, 28.7, 28.3, 27.9, 25.3, 25.1, 22.1, 21.9. HRMS (ESI) calculated for $C_{18}H_{24}N_3OS$ $[M+H]^+$: 330.1640, found: 330.1636.

**Gram scale for synthesis of 3a and it transformation**



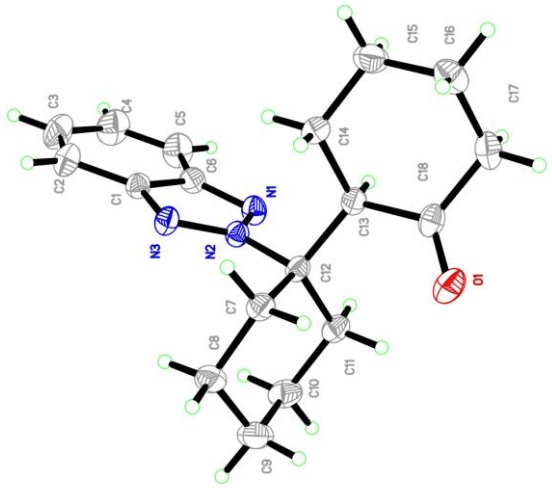

The procedure of gram scale: A dried flask was charged with benzotriazoles **1a** (10 mmol, 1.20 g, 1 eq.), cyclohexanone **2a** (160 mmol, 15.5 g, 16 eq.) and freshly distilled toluene (60 mL). Then $Sc(OTf)_3$ (1.47 g, 3 mmol, 30 mol %) were added. The tube was sealed and stirred at rt for 48 h. After the reaction was complete (monitored by TLC), the solution was quenched with water (10 mL) and extracted with DCM (3 x 10 mL). The organic layers dried over $Na_2SO_4$ and concentrated by rotary evaporation. The crude product was purified by silica gel column chromatography (EtOAc/PE = 1/20-1/3) to afford the **3a** (2.30 g, 84% yiled). $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.2 Hz, 1H), 7.76 (d, *J* = 8.6 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 3.07-2.89 (m, 3H), 2.40 (t, *J* = 11.8 Hz, 2H), 2.32-2.19 (m, 2H), 2.05-1.96 (m, 1H), 1.83-1.58 (m, 4H), 1.59-1.25 (m, 5H), 1.08-0.83 (m, 2H). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 210.5, 146.7, 127.1, 123.5, 120.4, 113.1, 68.5, 59.9, 44.1, 29.9, 29.8, 29.2, 28.3, 25.6, 25.2, 22.0, 21.9. HRMS (ESI) calculated for $C_{18}H_{24}N_3O$ $[M+H]^+$: 298.1919, found: 298.1916.

Compound **8** was prepared as follows: To a solution of **3a** (60 mg, 0.2 mmol) in dry methanol (5 mL) was added sodium borohydride (3 equiv). The reaction was stirred at r.t for 1 h. The reaction was then quenched with sat. $NH_4Cl$ (10 mL) and extracted with ethyl acetate (10 mL x 3). The organic layers were combined, dried over $Na_2SO_4$, concentrated by rotary evaporation. The dr ratio was determined by $^1H$ NMR of the crude reaction mixture. Then the residue was purified by silica gel column chromatography (petroleum ether /ethyl acetate = 3/1) to afford product **8a** (27.1 mg, 45%, Rf = 0.35) and **8b** (26.8 mg, 45%, Rf = 0.26). **8a**: $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.95-7.72 (m, 2H), 7.49-7.35 (m, 2H), 3.85 (s, 2H), 3.15 (dd, *J* = 14.2, 2.3 Hz, 1H), 2.94 (d, *J* = 11.7 Hz, 1H), 2.07 (td, *J* = 13.8, 3.6 Hz, 1H), 1.89-1.64 (m, 6H), 1.59-1.25 (m, 7H), 1.21-1.04 (m, 2H), 0.87 (qd, *J* = 12.8, 3.4 Hz, 1H). $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 143.6, 126.5, 118.3, 72.4, 66.0, 52.7, 34.8, 34.5, 34.0, 26.6, 25.2, 22.4, 22.2, 21.3, 19.8. **8b**: $^1H$ NMR (300 MHz, $CDCl_3$) δ 8.13-7.80 (m, 2H), 7.48-7.31 (m, 2H), 3.20 (d, *J* = 4.2 Hz, 2H), 3.03-2.87 (m, 1H), 2.74 (ddd, *J* = 13.8, 10.5, 3.7 Hz, 1H), 2.16-1.99 (m, 2H), 1.92-1.75 (m, 2H), 1.72-1.47 (m, 6H), 1.41-1.17 (m, 4H), 1.13-0.88 (m, 3H). $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 143.7, 126.3, 118.4, 72.7, 72.5, 55.2, 36.1, 34.3, 26.0, 25.8, 25.1, 24.8, 22.5, 22.4. HRMS (ESI) calculated for $C_{18}H_{26}N_3O$ $[M+H]^+$: 300.2076, found: 300.2072.

Compound **9** was prepared as follows: To a solution of **3a** (60 mg, 0.2 mmol) in dry THF (5 mL) was added methyl lithium (3M in $Et_2O$，0.3 mmol, 1.5 equiv). The reaction was then stirred at r.t for 1.5 h. The reaction was then quenched with sat. $NH_4Cl$ (10 mL) and extracted with ethyl acetate (10 mL x 3). The organic layers were combined, dried over $Na_2SO_4$, concentrated by rotary evaporation. The dr ratio was determined by $^1H$ NMR of the crude reaction mixture. Then the residue was purified by silica gel column chromatography (petroleum ether /ethyl acetate = 10/1) to afford product **9** as a yellow oil, 57.1 mg, 82% yield. $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 6.5, 3.1 Hz, 2H), 7.36 (dd, *J* = 6.5, 3.0 Hz, 2H), 3.12 (dd, *J* = 14.2, 2.4 Hz, 1H), 2.92 (d, *J* = 13.7 Hz, 1H), 2.54 (s, 1H), 2.13 (td, *J* = 13.3, 3.6 Hz, 1H), 2.05-1.92 (m, 2H), 1.72-1.13 (m, 13H), 0.85 (s, 3H), 0.81-0.72 (m, 1H). $^{13}C$ NMR (126 MHz, $CDCl_3$) δ 143.47, 126.21, 118.27, 77.41, 77.16, 76.91, 74.06, 72.62, 56.39, 44.11, 37.16, 34.15, 30.40, 26.48, 25.16, 24.29, 22.41, 22.01, 21.84.

**X-ray of 3a** (method of crystallization: In a 5 mL tube, compound **3a** (30 mg) was dissolved in a mixture n-hexane/$CH_2Cl_2$ (5 mL: 1 mL). The tube was closed with a septum and a needle was

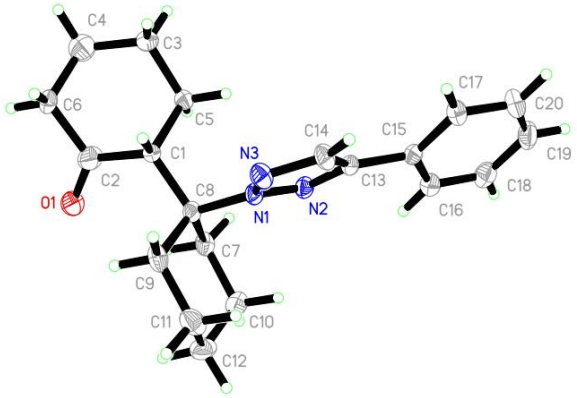
inserted into the septum in order to slowly evaporation of $CH_2Cl_2$). Thermal ellipsoids are shown at the 50% level.




**Crystallographic data of 3a**

| | |
|---|---|
| Empirical formula | $C_{18}H_{23}N_3O$ |
| Formula weigh | 297.39 |
| Crystal system | monoclinic |
| Space group | P21/n |
| *a* (Å) | 15.744(9) |
| *b* (Å) | 7.962(5) |
| *c* (Å) | 12.994(7) |
| α (°) | 90.00 |
| β (°) | 91.481(9) |
| γ (°) | 90.00 |
| *V* (Å3) | 1628.3(16) |
| *Z* | 4 |
| Temperature/K | 296(2) |
| *F* (000) | 640 |
| Crystal size/$mm^3$ | 0.120 ×0.110 ×0.09 |
| $\theta$ min, $\theta$ max (deg) | 1.294, 28.338 |
| Reflections collected | 4032 |
| Independent reflections | 4032 |
| Data/restraints/parameters | 4032/0/199 |
| Goodness-of-fit on F2 | 1.058 |
| Final R indexes [I>=2σ (I)] | R1 = 0.0484, wR2 = 0.1455 |
| Final R indexes [all data] | R1 = 0.0681, wR2 = 0.1607 |
| Largest diff. peak and hole/ e Å-3 | 0.155/-0.172 |

**X-ray of 5a** (method of crystallization: In a 5 mL tube, compound **5a** (30 mg) was dissolved in a mixture n-hexane/$CH_2Cl_2$ (5 mL: 1 mL). The tube was closed with a septum and a needle was inserted into the septum in order to slowly evaporation of $CH_2Cl_2$). Thermal ellipsoids are shown at the 50% level.



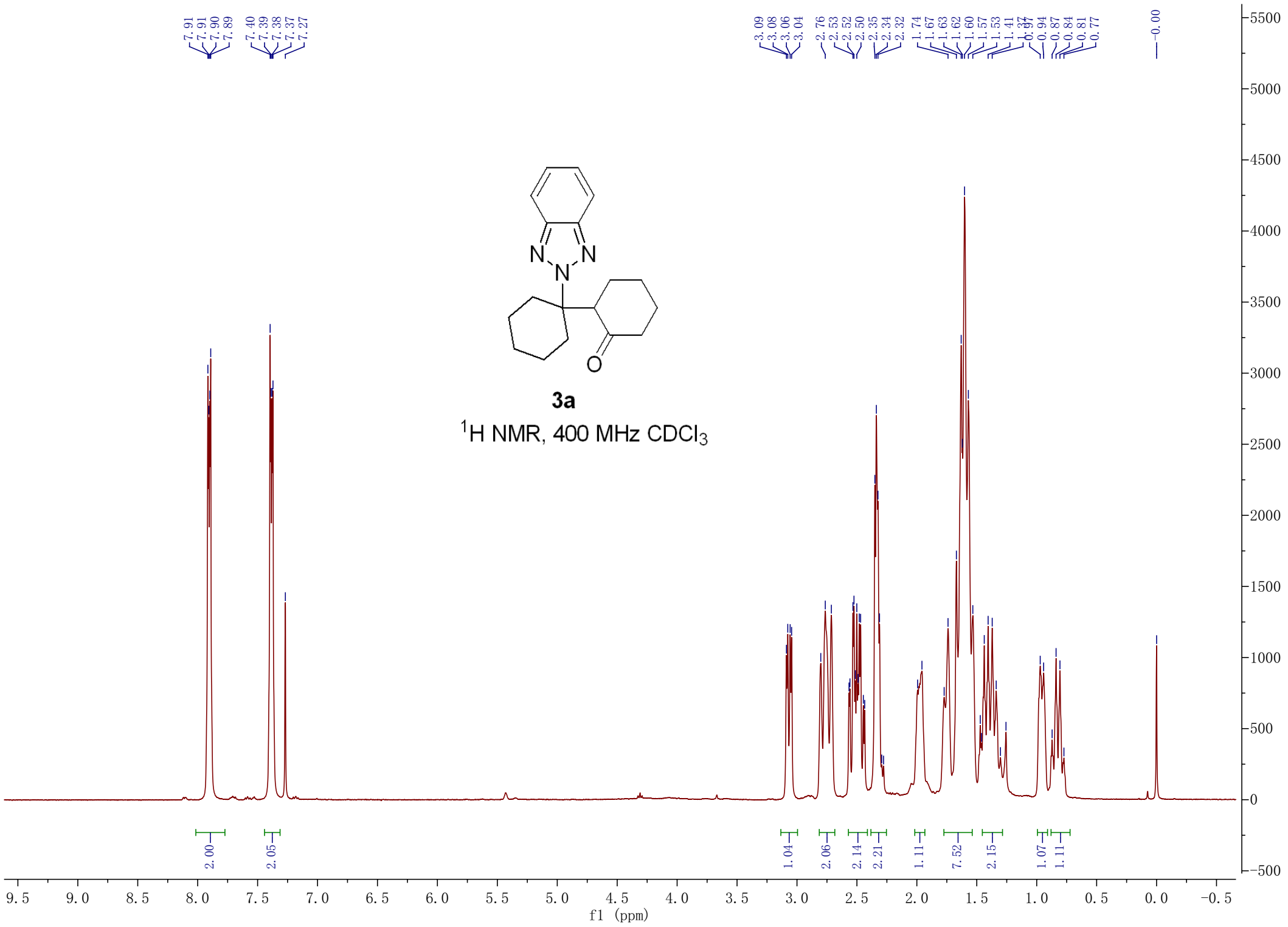

**Crystallographic data of 5a**

| | |
|---|---|
| Empirical formula | $C_{20}H_{25}N_3O$ |
| Formula weigh | 323.43 |
| Crystal system | monoclinic |
| Space group | P21/n |
| *a* (Å) | 9.436(3) |
| *b* (Å) | 18.526(7) |
| *c* (Å) | 10.349(5) |
| α (°) | 90.00 |
| β (°) | 102.799(13) |
| γ (°) | 90.00 |
| *V* (Å3) | 1764.3(13) |
| *Z* | 4 |
| Temperature/K | 210(2) |
| *F* (000) | 696 |
| Crystal size/$mm^3$ | 0.170 ×0.140 ×0.08 |
| $\theta$ min, $\theta$ max (deg) | 3.81, 53.73 |
| Reflections collected | 3228 |
| Independent reflections | 3203 |
| Data/restraints/parameters | 3203/12/254 |
| Goodness-of-fit on F2 | 1.091 |
| Final R indexes [I>=2σ (I)] | R1 = 0.0830, wR2 = 0.1632 |
| Final R indexes [all data] | R1 = 0.0754, wR2 = 0.1672 |

**References**

(1) (a) Wang, K.; Chen, P.; Ji, D.; Zhang, X.; Xu, G.; Sun, J. *Angew*. *Chem*. *Int*. *Ed*. **2018**, *57*, 12489. (b) Wang, Y.; Wu, Y.; Li, Y.; Tang, Y. *Chem. Sci.* **2017**, *8*, 3852.

(2) (a) Bhagat, U. K.; Peddinti, R. K. *J. Org. Chem.* **2018**, *83*, 793. (b) Zhu, L.; Tian, L.; Cai, B.; Liu, G.; Zhang, H.; Wang, Y. *Chem*. *Commun.* **2020**, *56*, 2979.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7.89
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7.39
7.38
7.37
7.27
3.09
3.08
3.06
3.04
2.76
2.53
2.52
2.50
2.35
2.34
2.32
1.74
1.67
1.63
1.62
1.60
1.57
1.53
1.41
0.94
0.87
0.84
0.81
0.77
0.00
N
N
N
O
3a
1H NMR, 400 MHz CDCl3
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2.05
1.04
2.06
2.14
2.21
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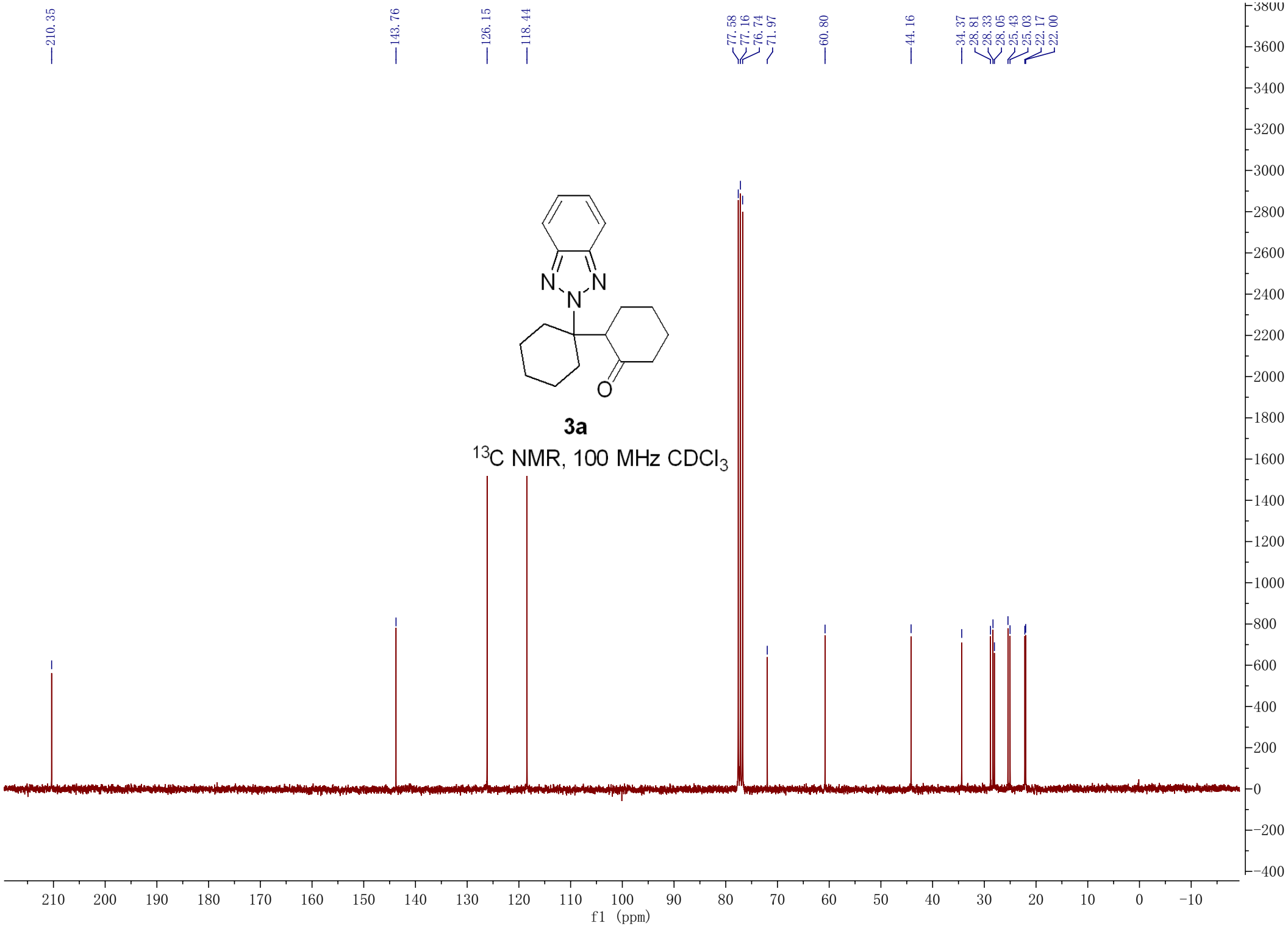
7.52
2.15
1.07
1.11
f1 (ppm)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71.97
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44.16
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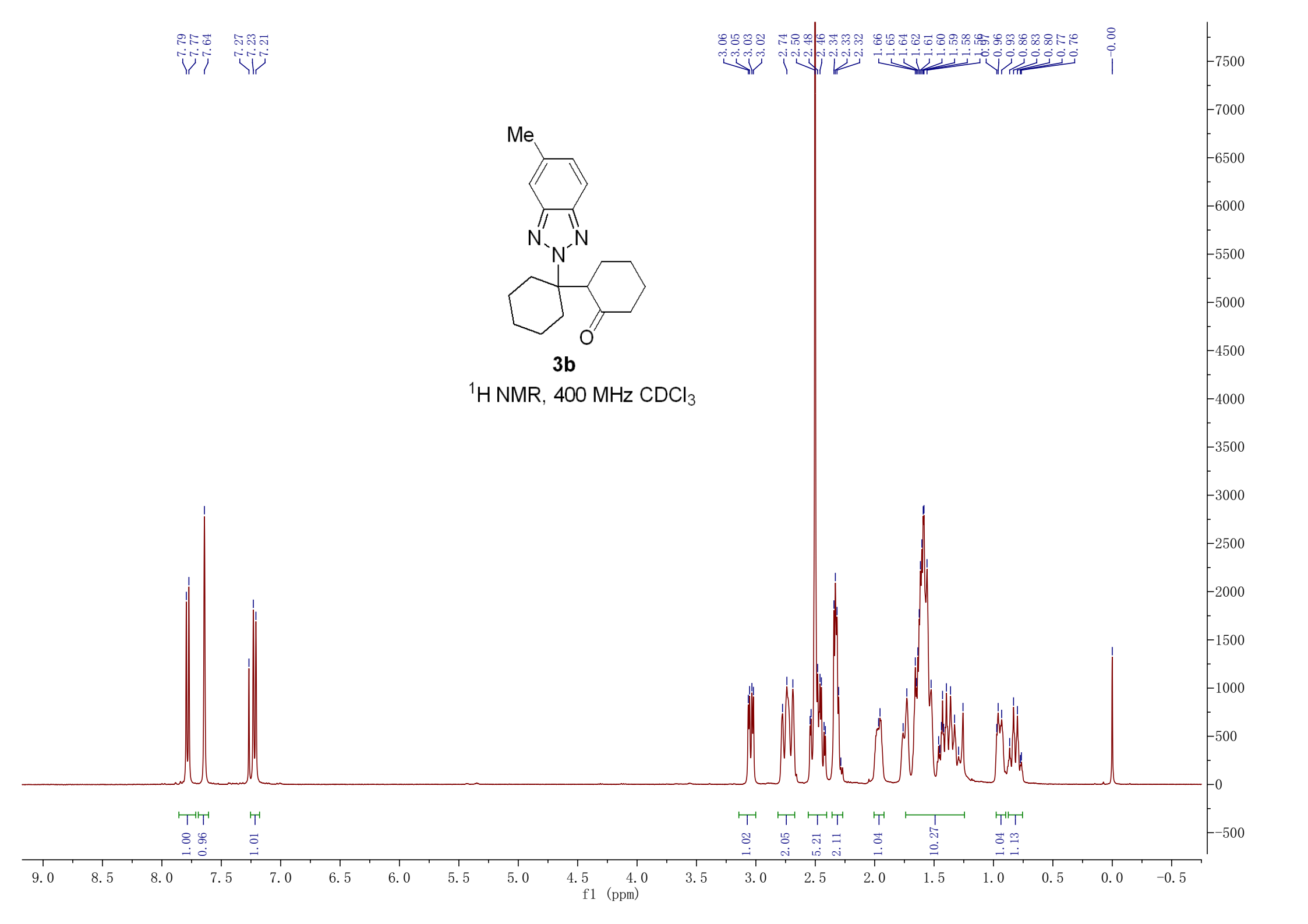
N
O
3a
13C NMR, 100 MHz CDCl3
f1 (ppm)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O
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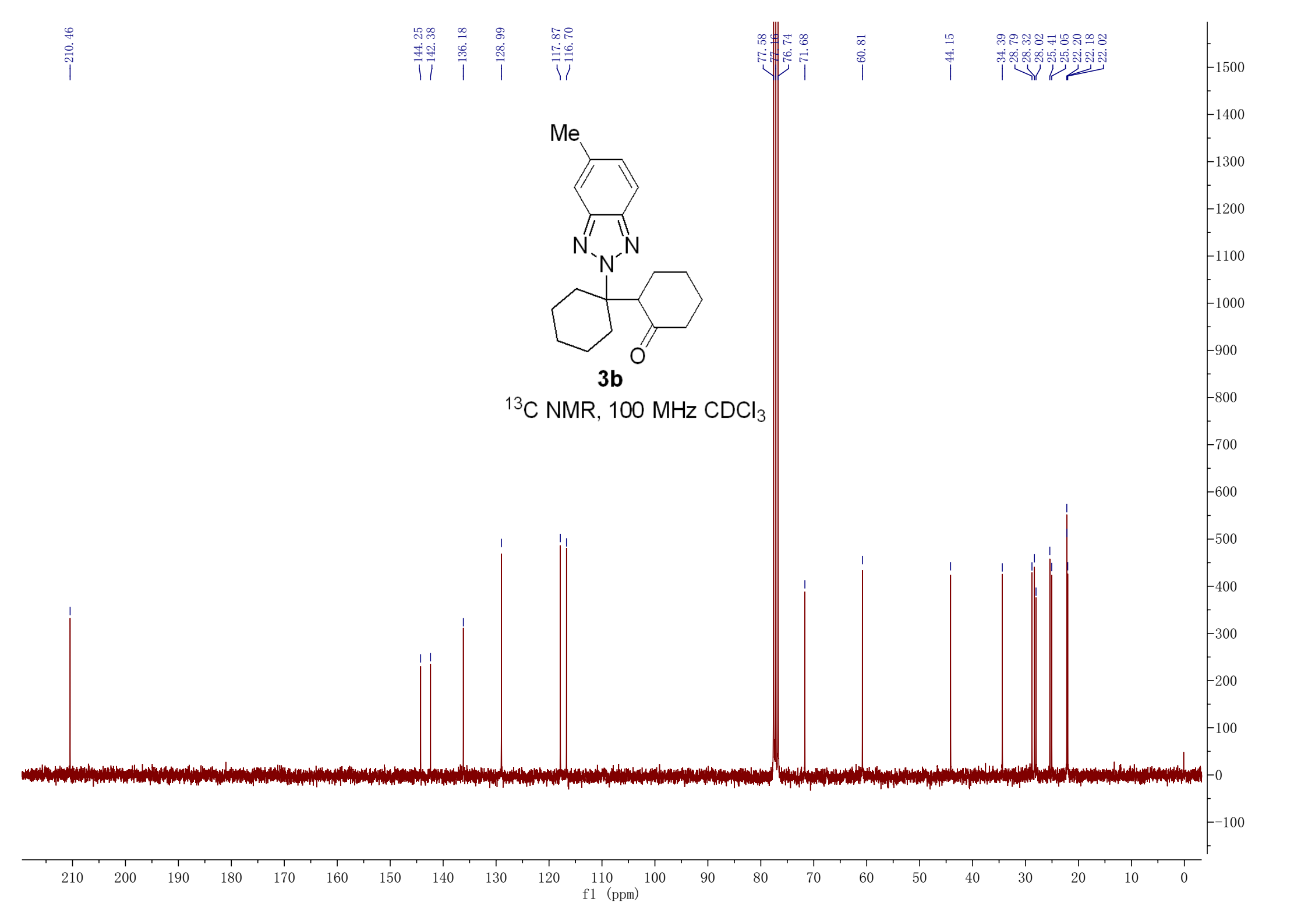
1H NMR, 400 MHz CDCl3
f1 (ppm)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44.15
34.39
28.79
28.32
28.02
25.41
25.05
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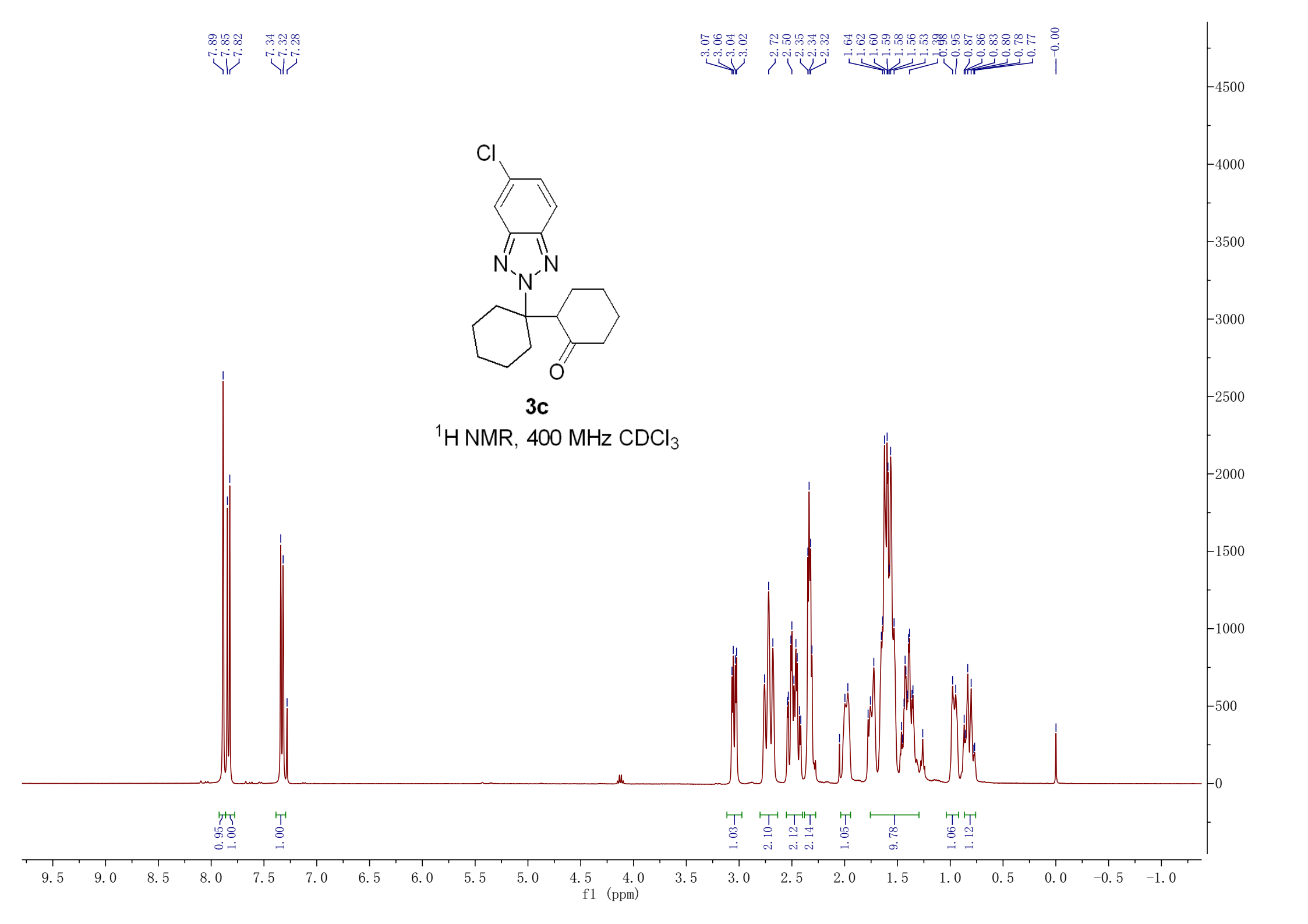
22.18
22.02
Me
N
N
N
O
3b
13C NMR, 100 MHz CDCl3
f1 (ppm)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7.82
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7.32
7.28
3.07
3.06
3.04
3.02
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2.50
2.35
2.34
2.32
1.64
1.62
1.60
1.59
1.58
1.56
1.53
0.95
0.87
0.86
0.83
0.80
0.78
0.77
0.00
Cl
N
N
N
O
3c
1H NMR, 400 MHz CDCl3
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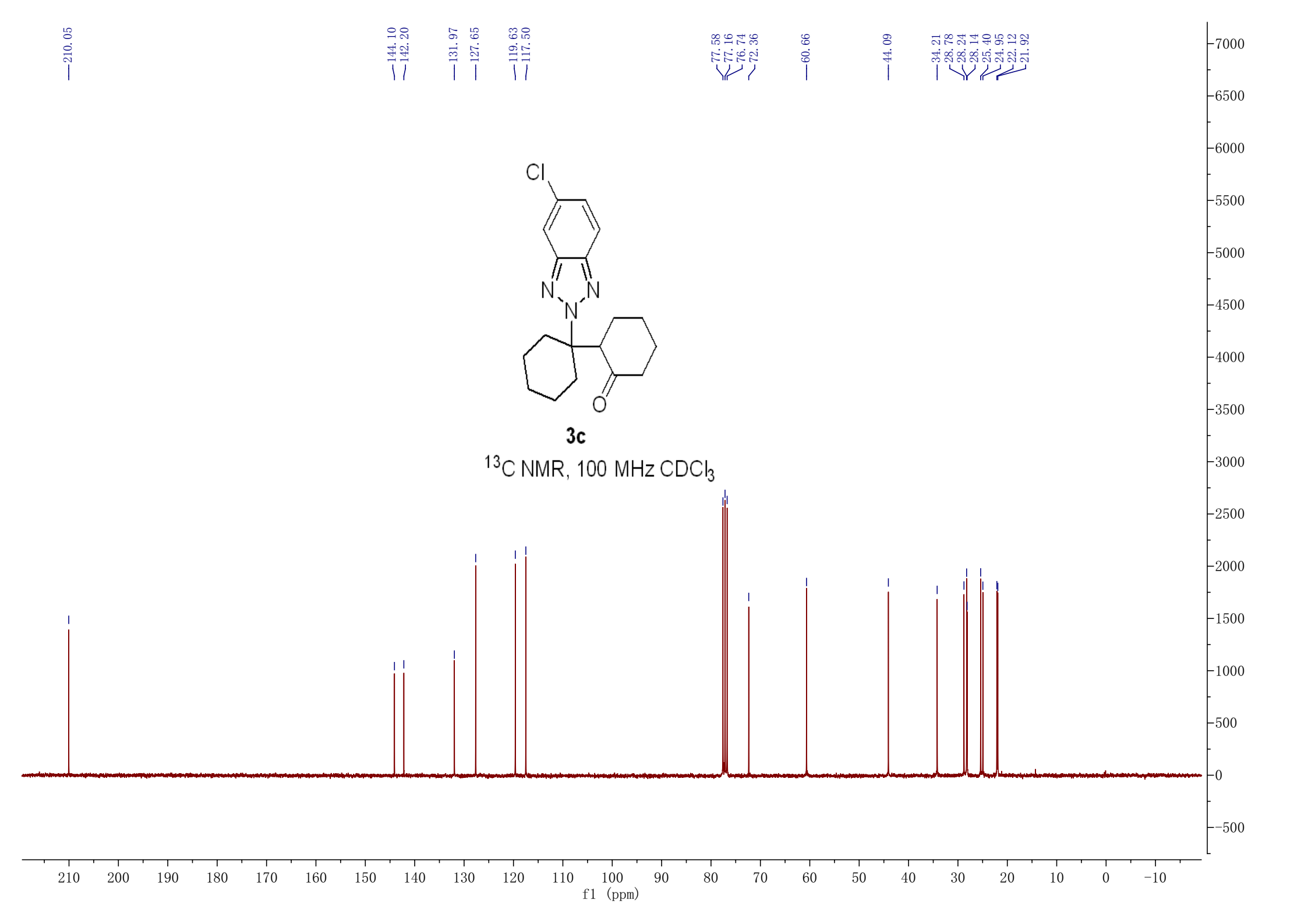
1.12
f1 (ppm)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34.21
28.78
28.24
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22.12
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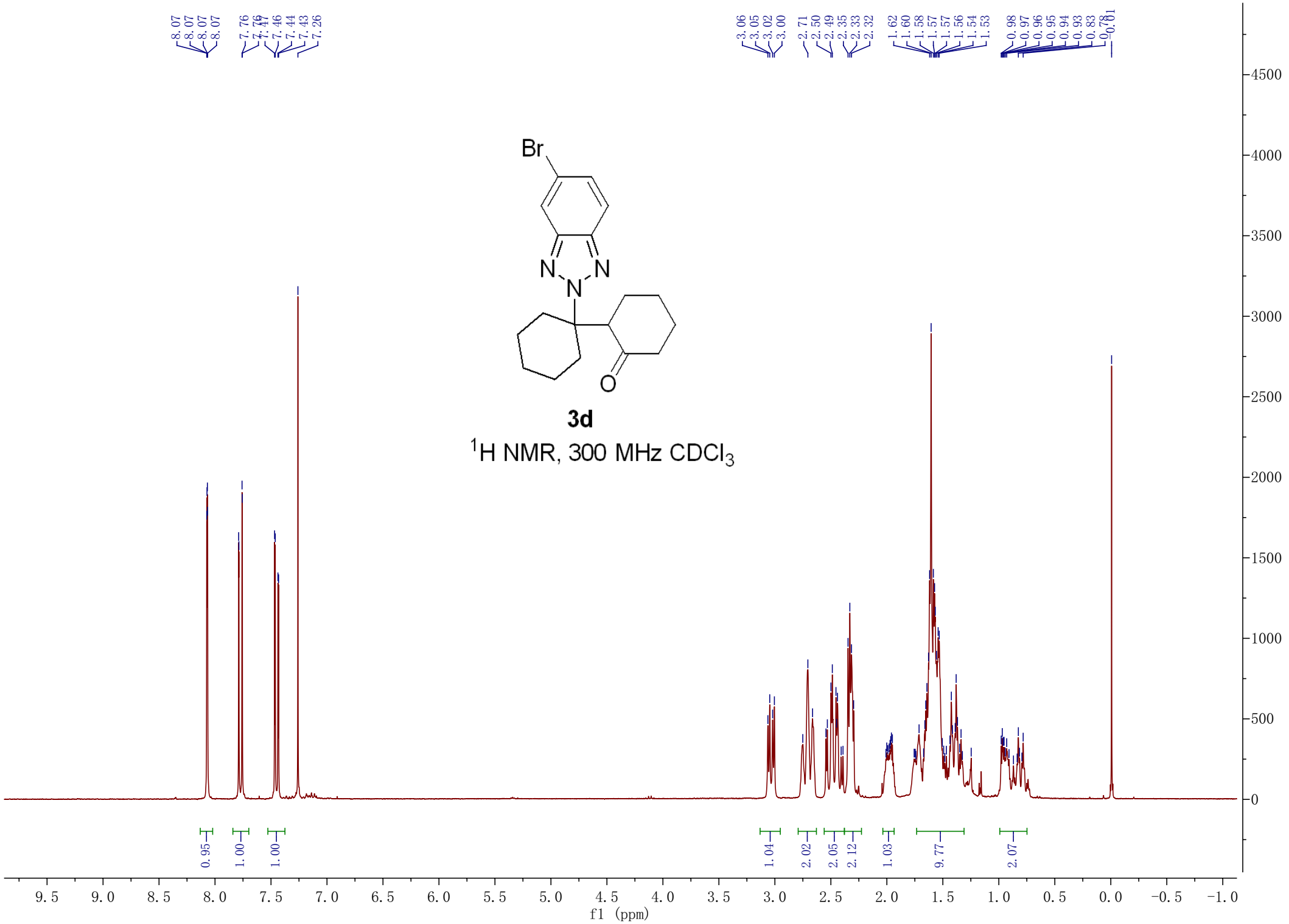
N
O
3c
13C NMR, 100 MHz CDCl3
f1 (ppm)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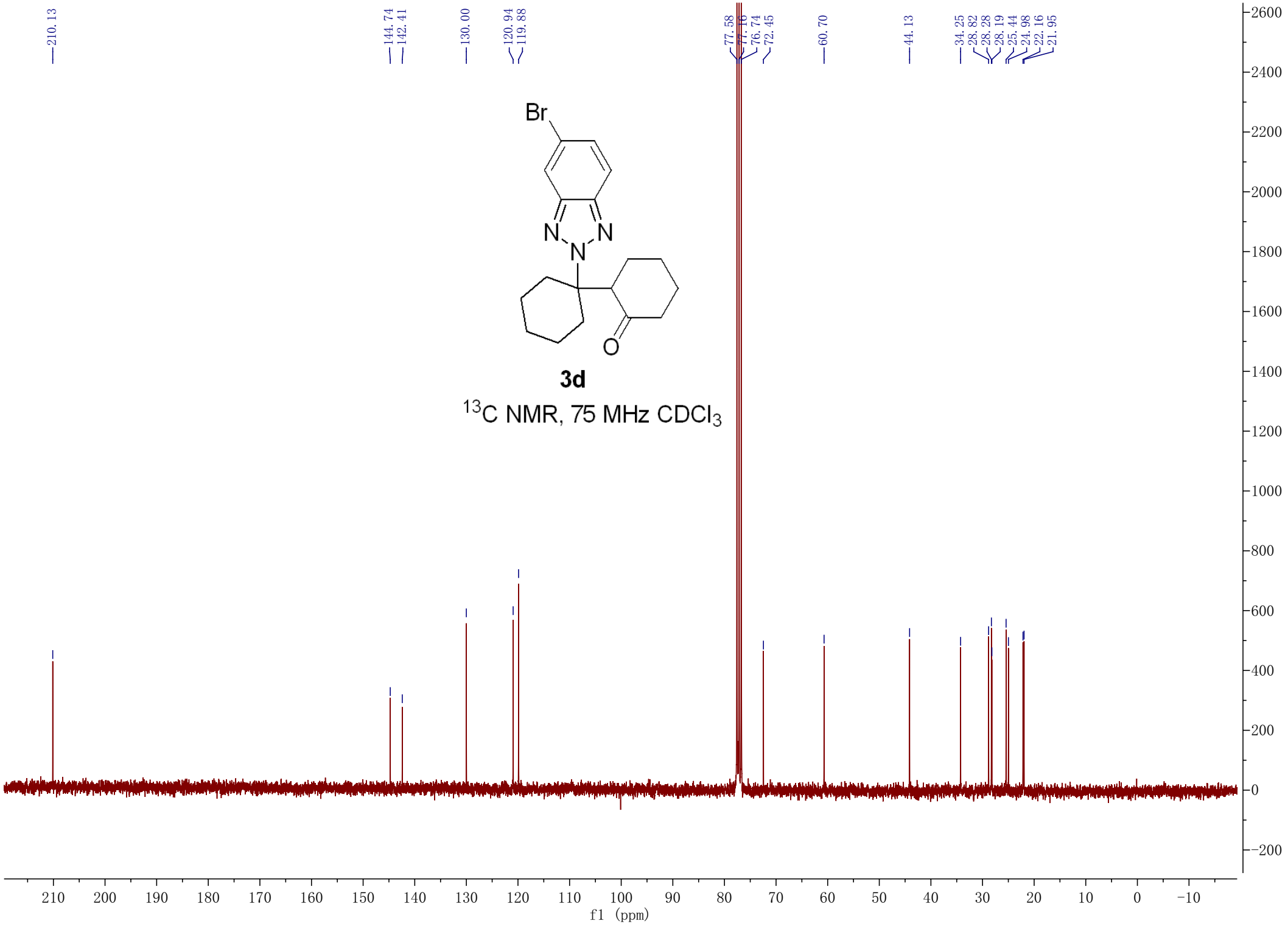
f1 (ppm)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28.28
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N
O
3d
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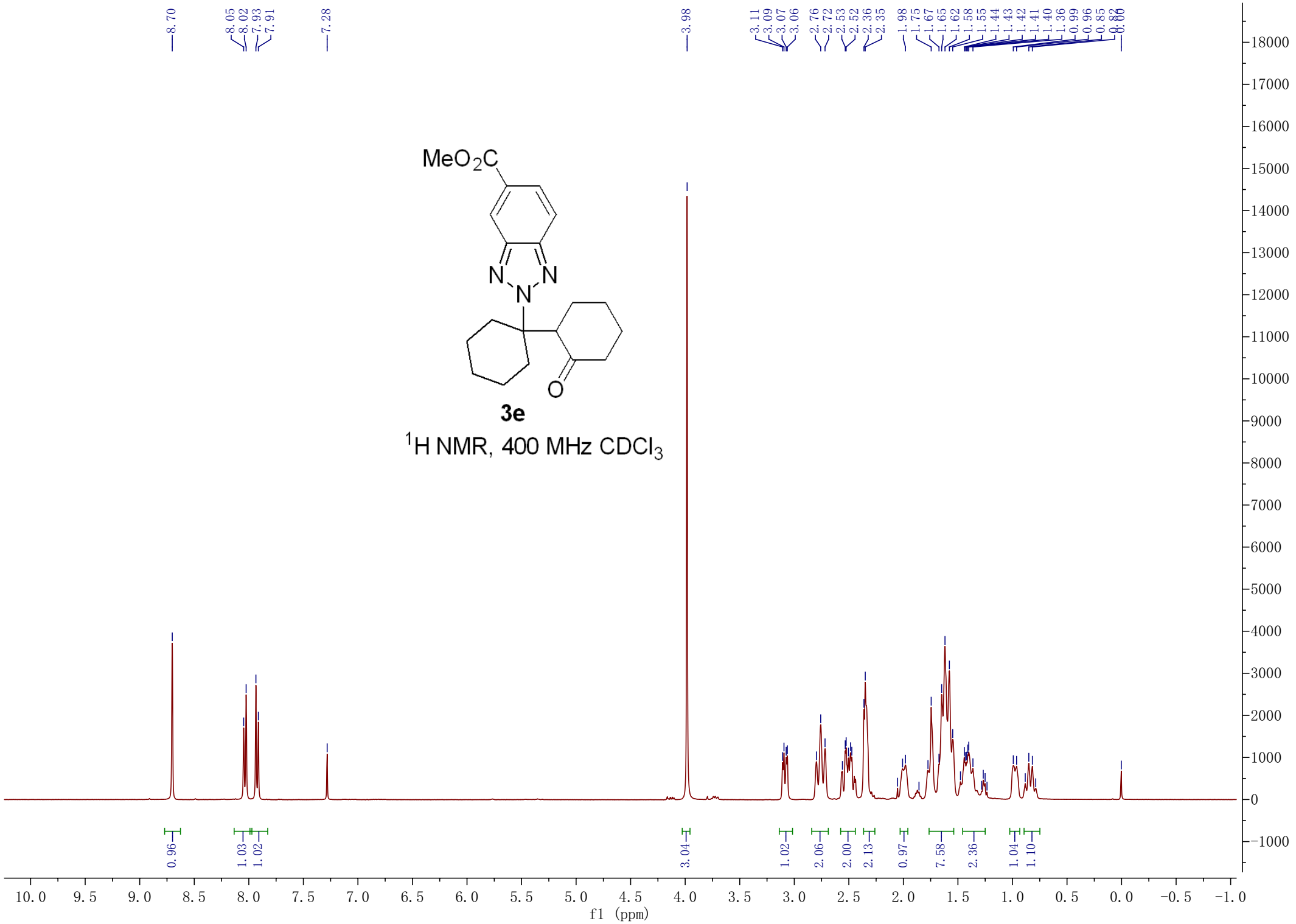
f1 (ppm)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1.75
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1.58
1.55
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1.43
1.42
1.41
1.40
1.36
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0.96
0.85
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N
N
O
3e
1H NMR, 400 MHz CDCl3
0.96
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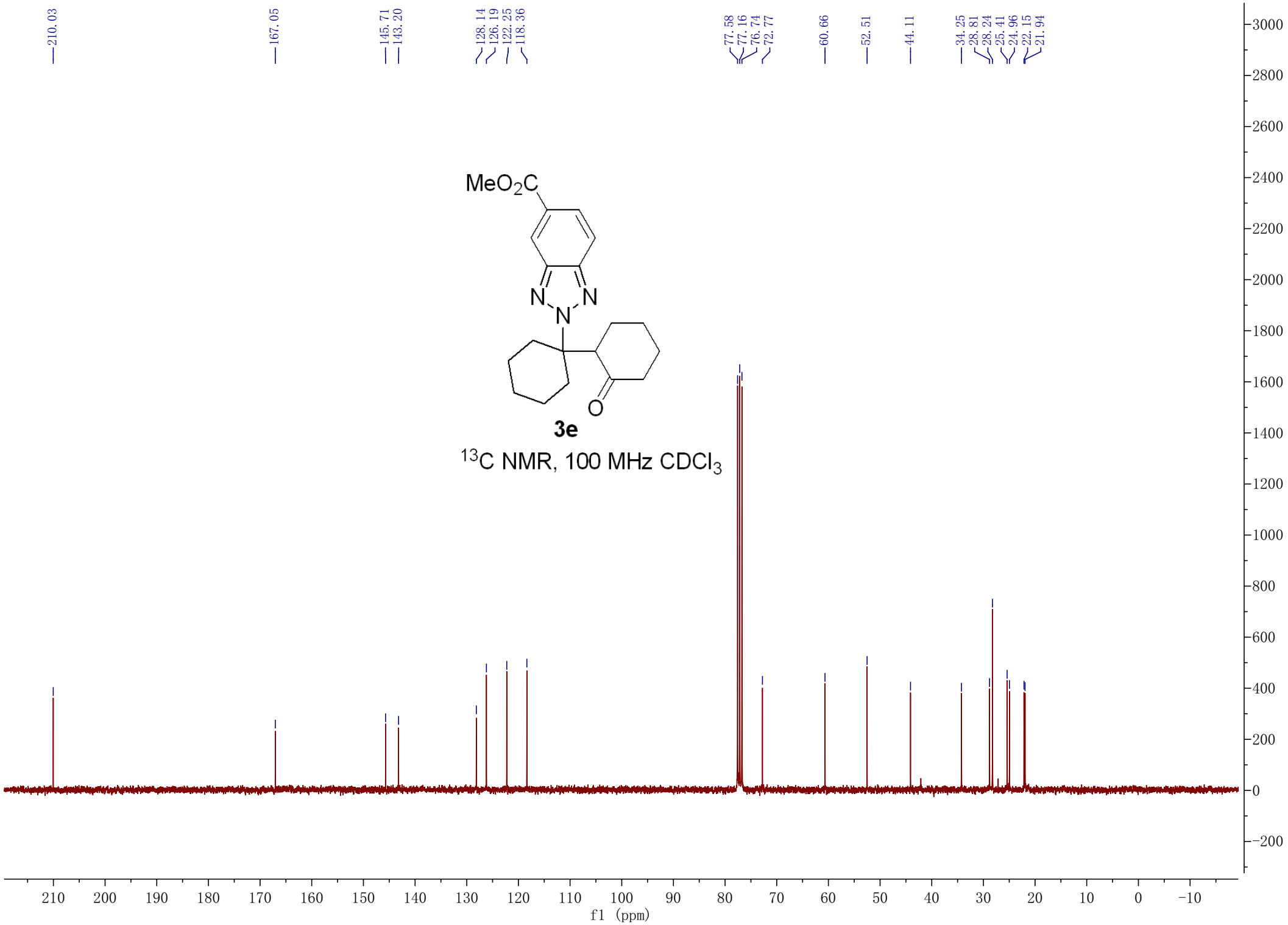
f1 (ppm)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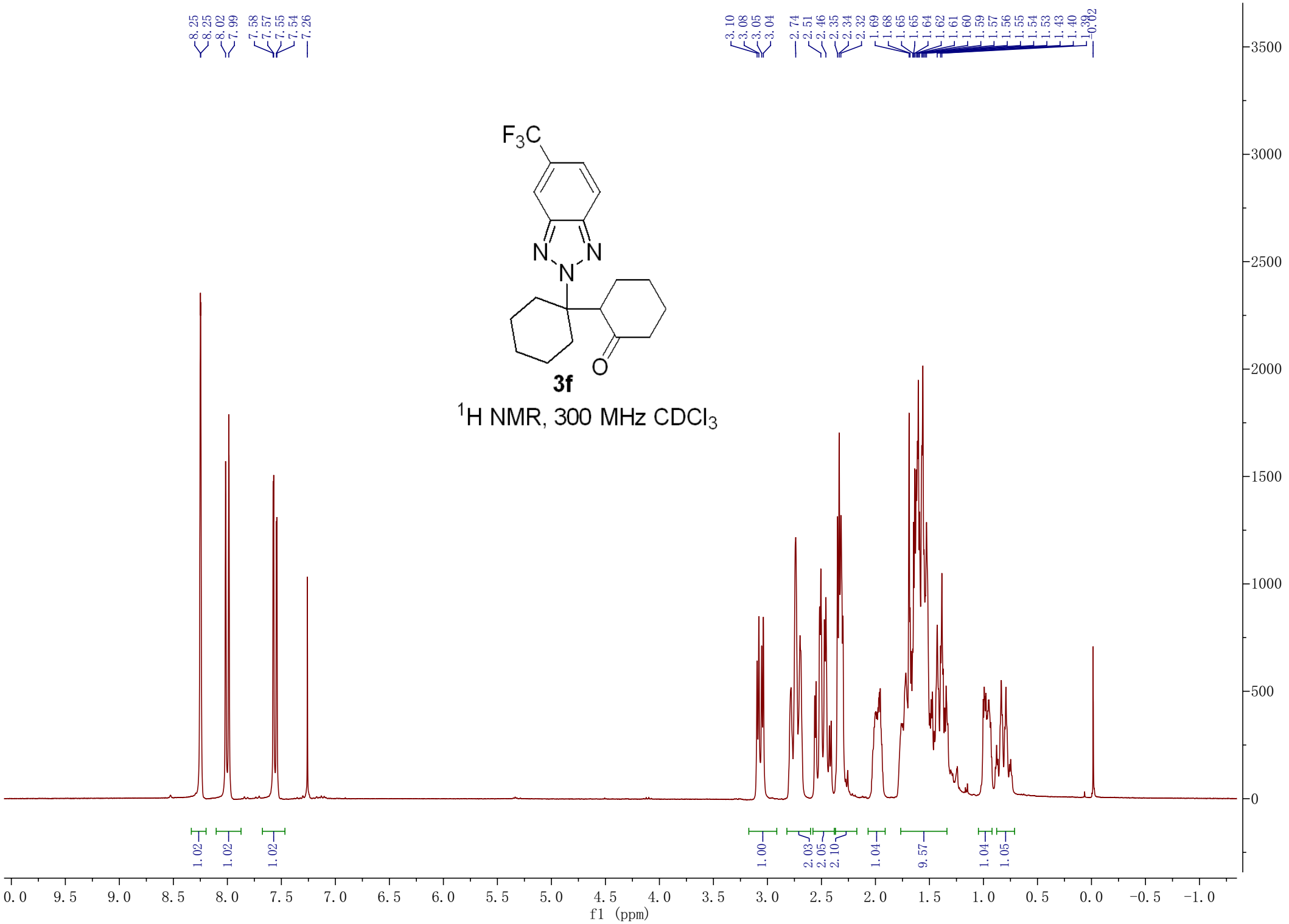
O
3e
13C NMR, 100 MHz CDCl3
f1 (ppm)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1.43
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N
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3f
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2.05
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2500
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4.0
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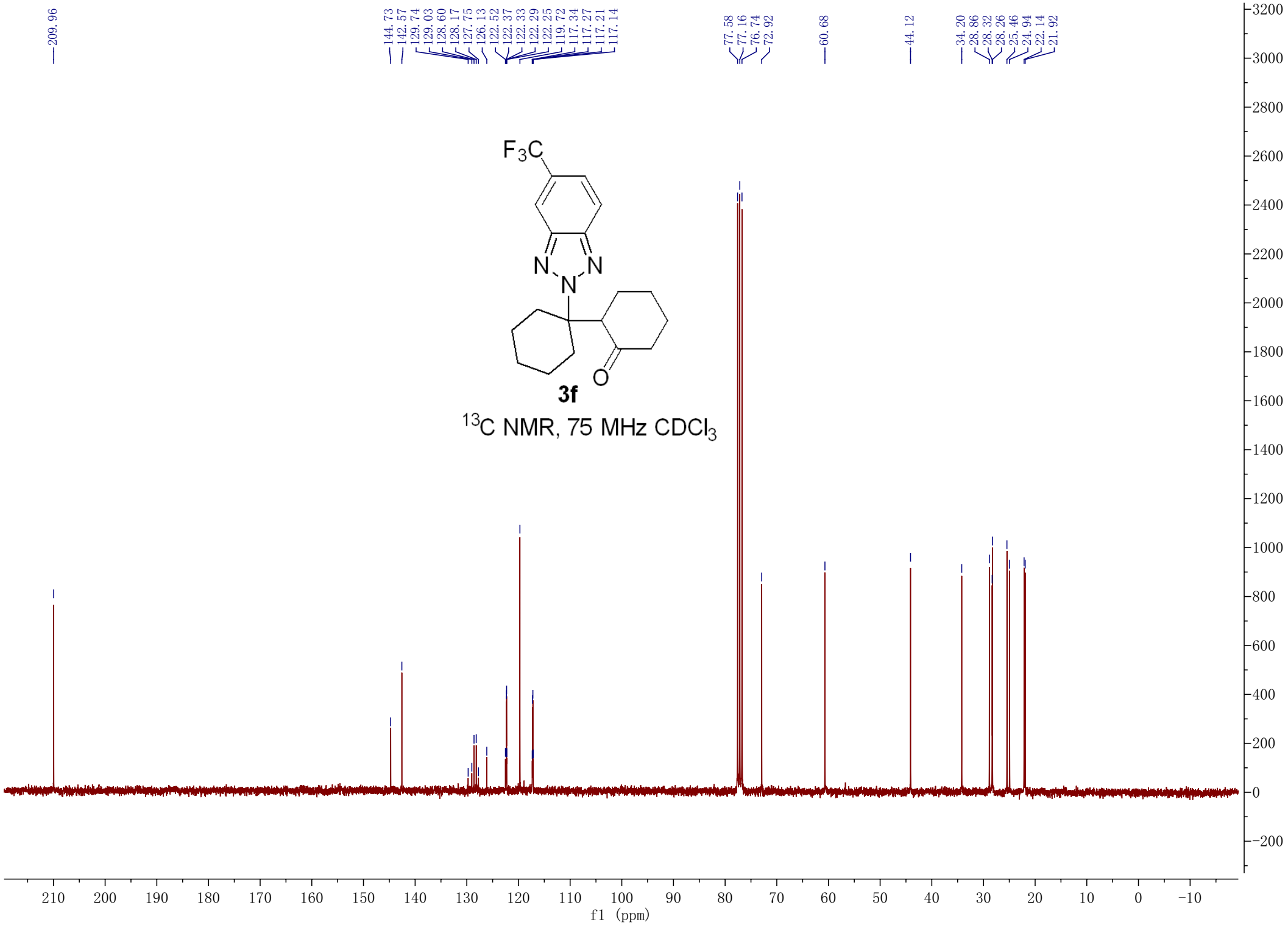
f1 (ppm)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72.92
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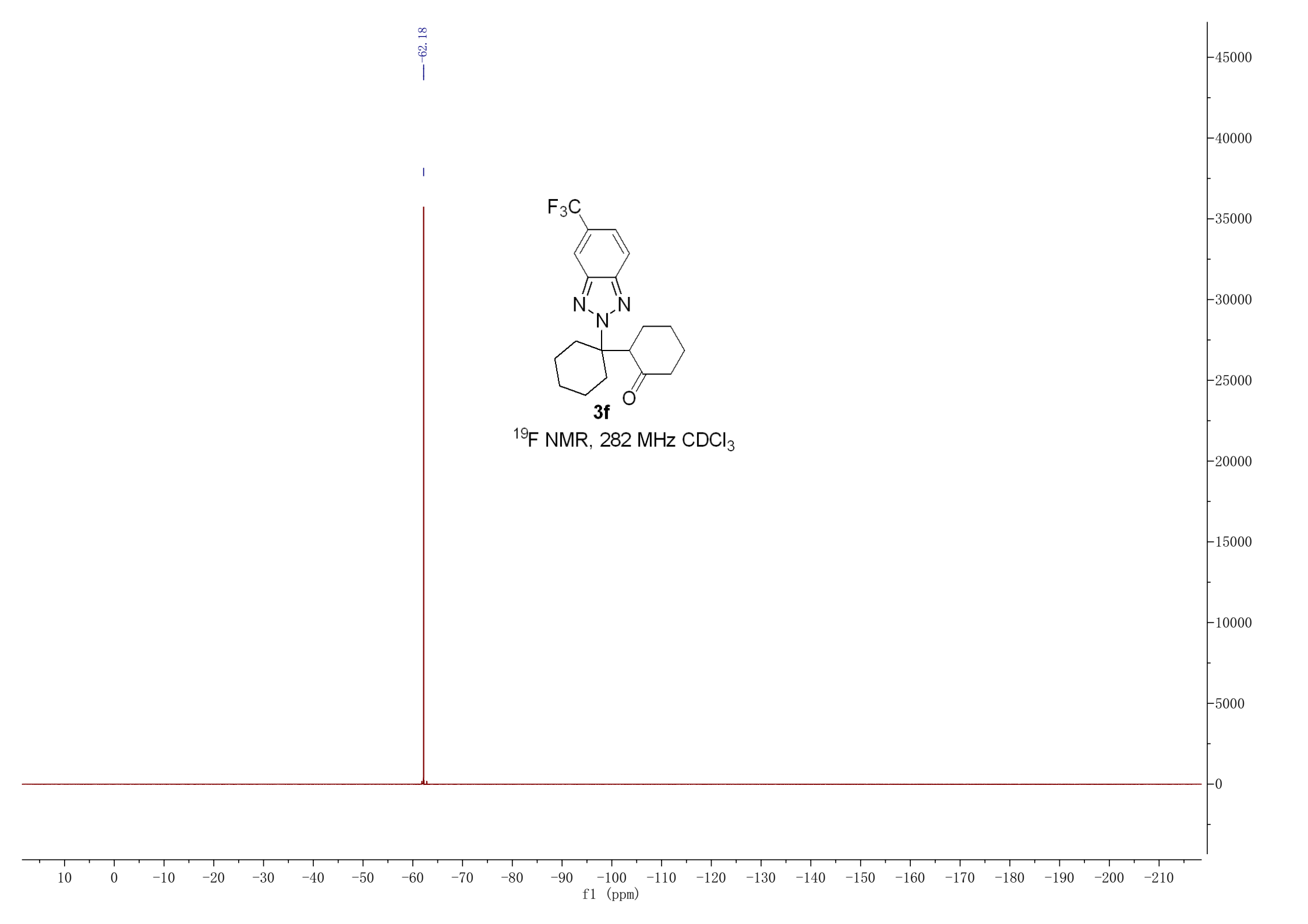
O
3f
13C NMR, 75 MHz CDCl3
f1 (ppm)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20000
15000
10000
5000
0
10
0
-10
-20
-30
-40
-50
-60
-70
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-110
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-140
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-180
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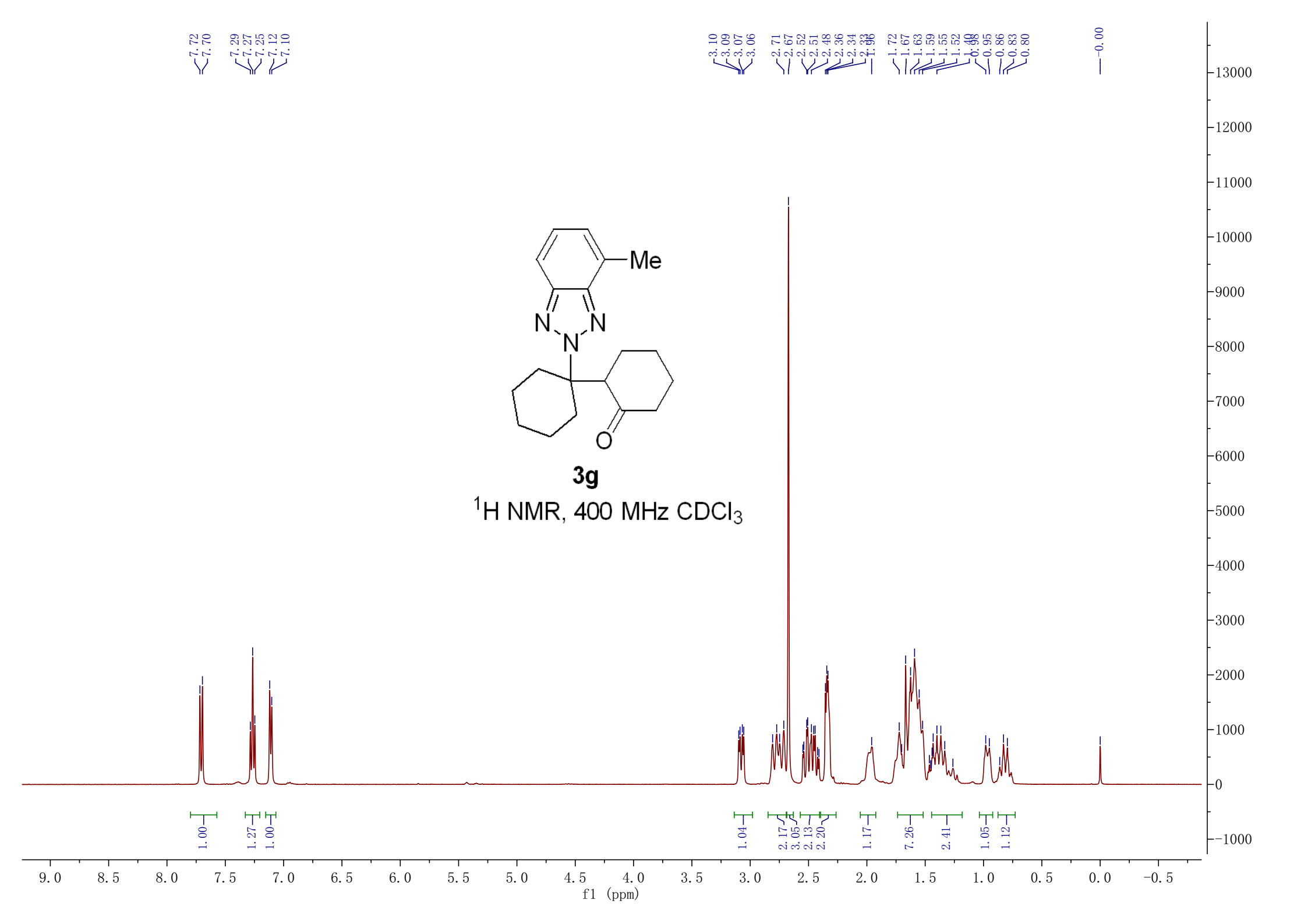
-210
f1 (ppm)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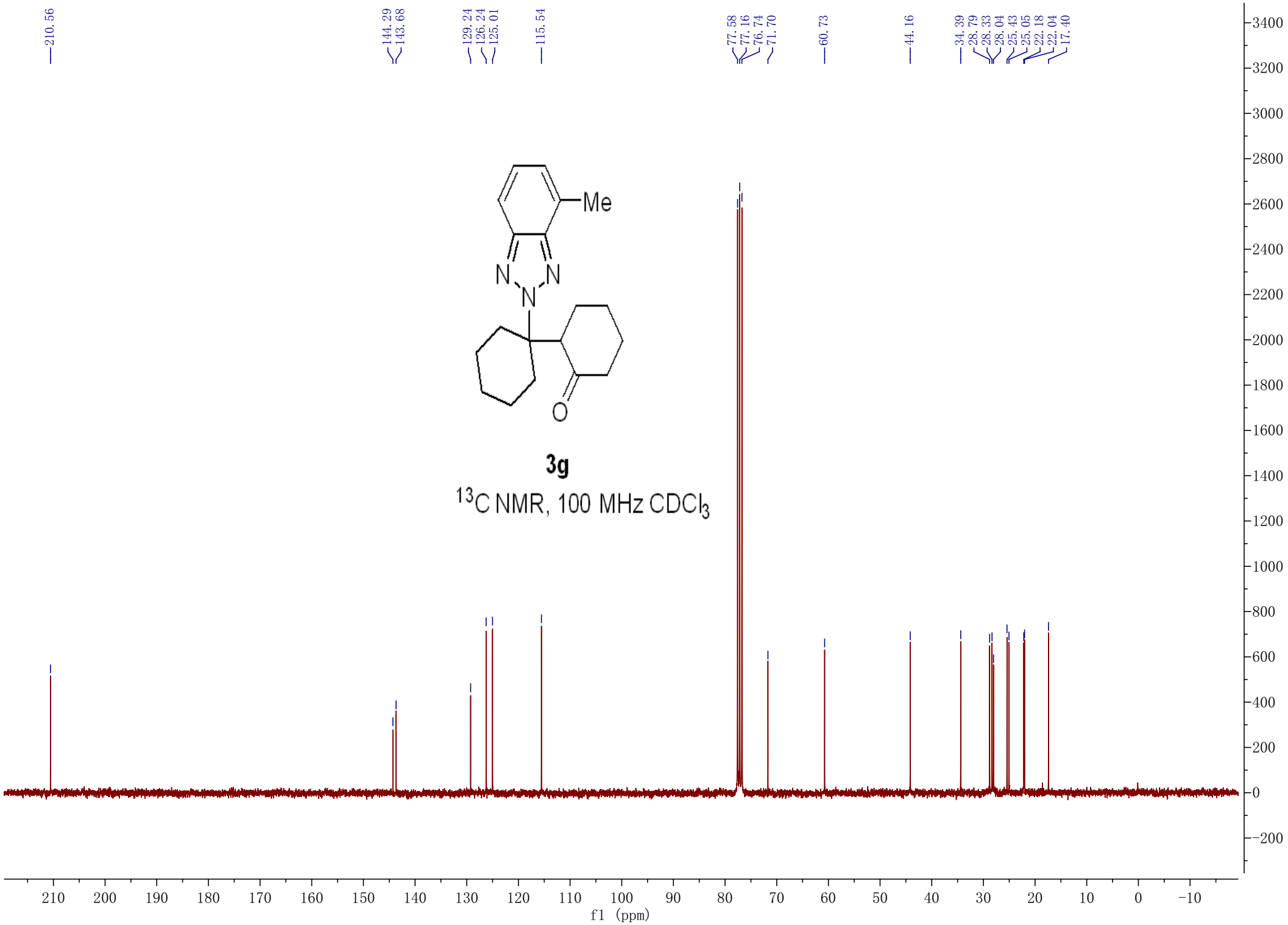
O
3g
1H NMR, 400 MHz CDCl3
f1 (ppm)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34.39
28.79
28.33
28.04
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Me
N
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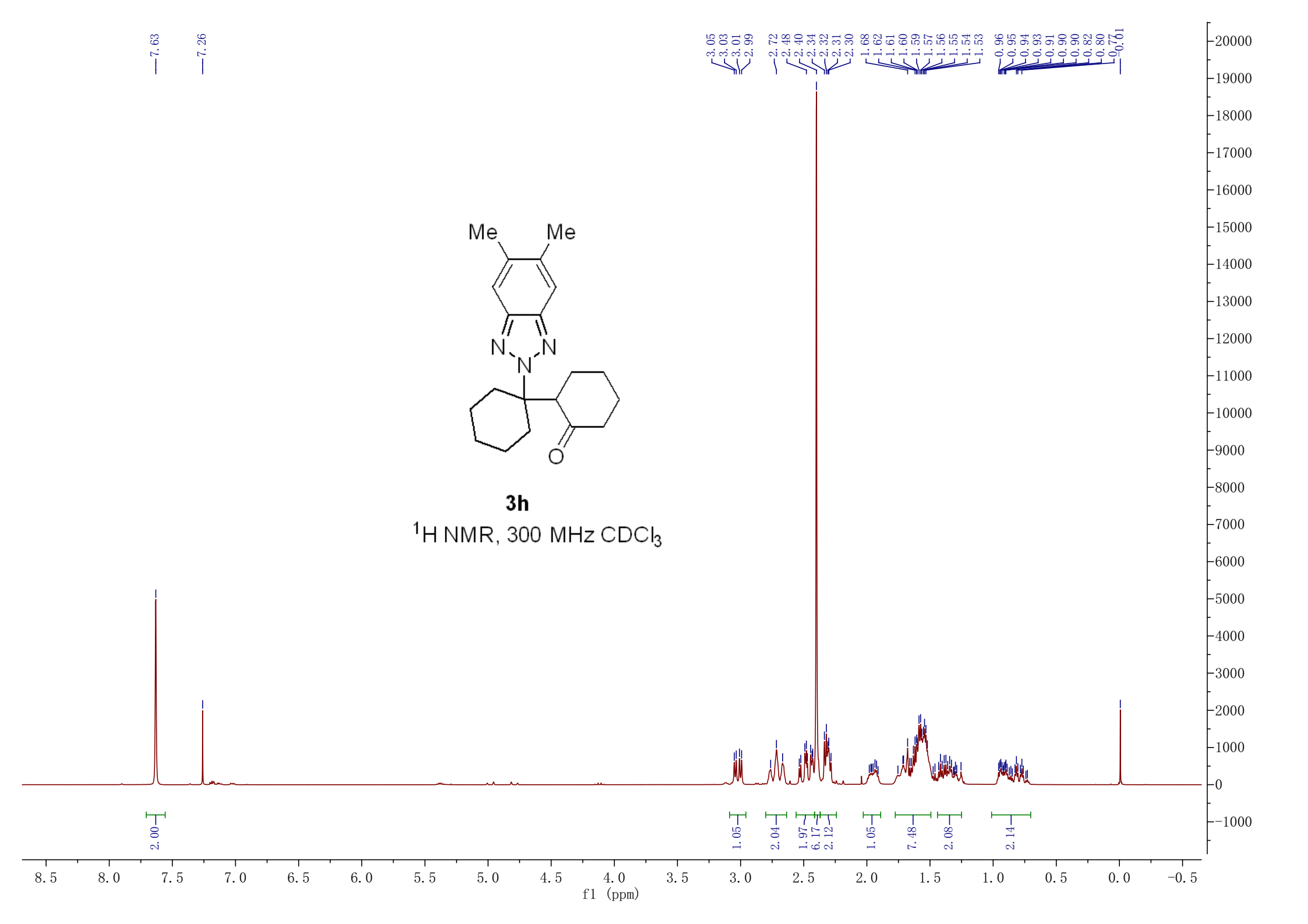
N
O
3g
13C NMR, 100 MHz CDCl3
f1 (ppm)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1.62
1.61
1.60
1.59
1.57
1.56
1.55
1.54
1.53
0.96
0.95
0.94
0.93
0.91
0.90
0.90
0.82
0.80
0.77
0.71
Me
Me
N
N
N
O
3h
1H NMR, 300 MHz CDCl3
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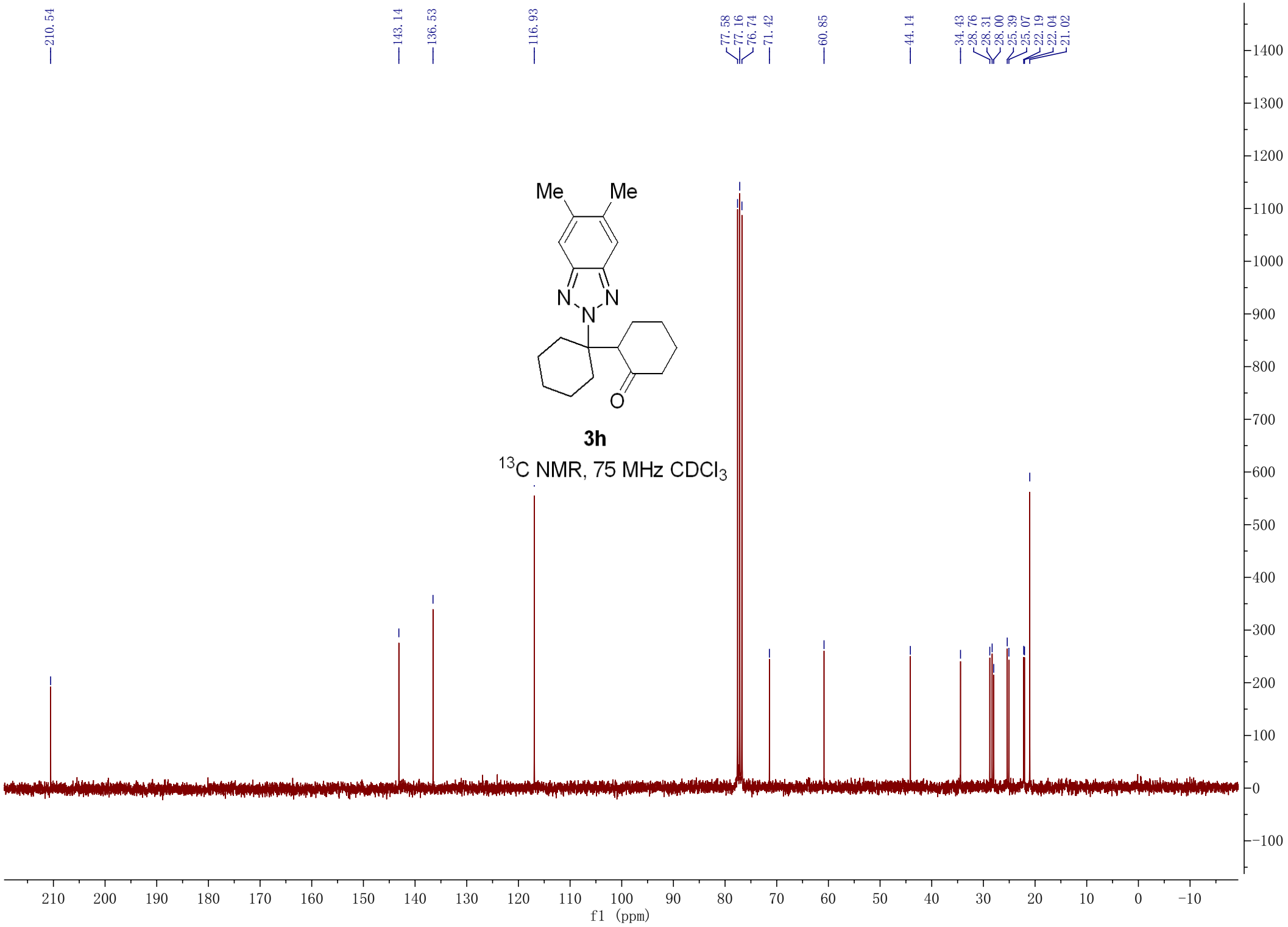
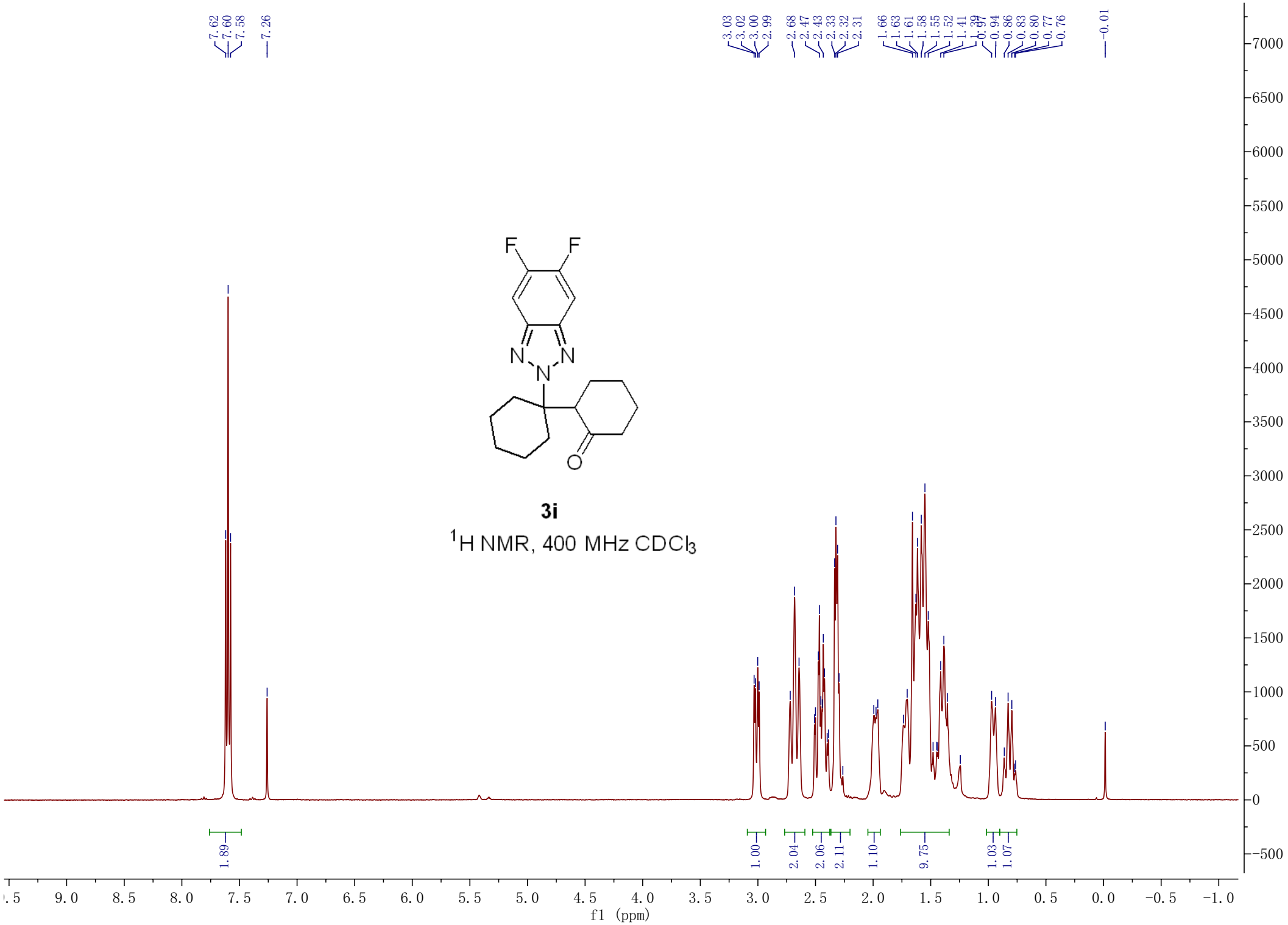
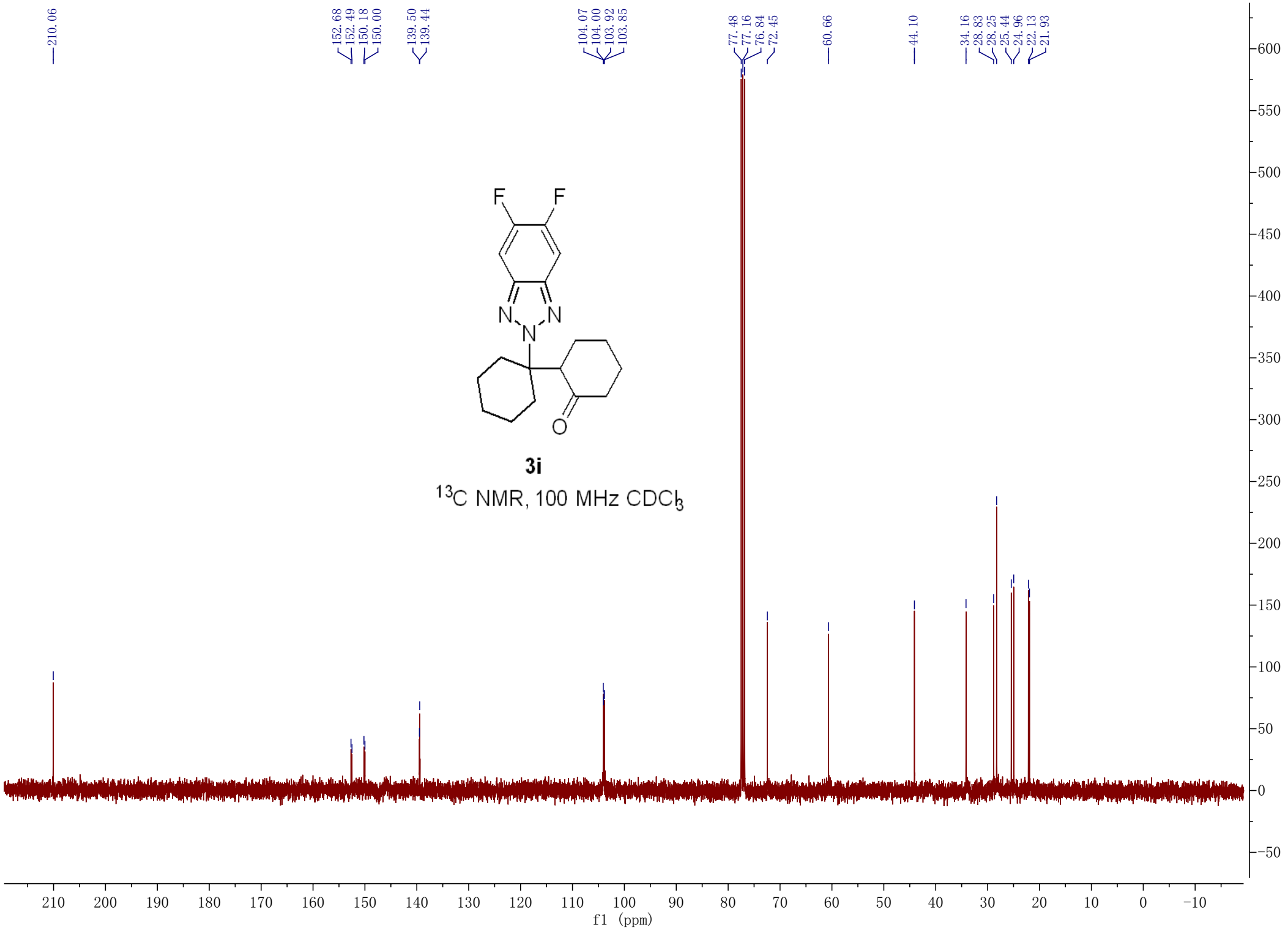
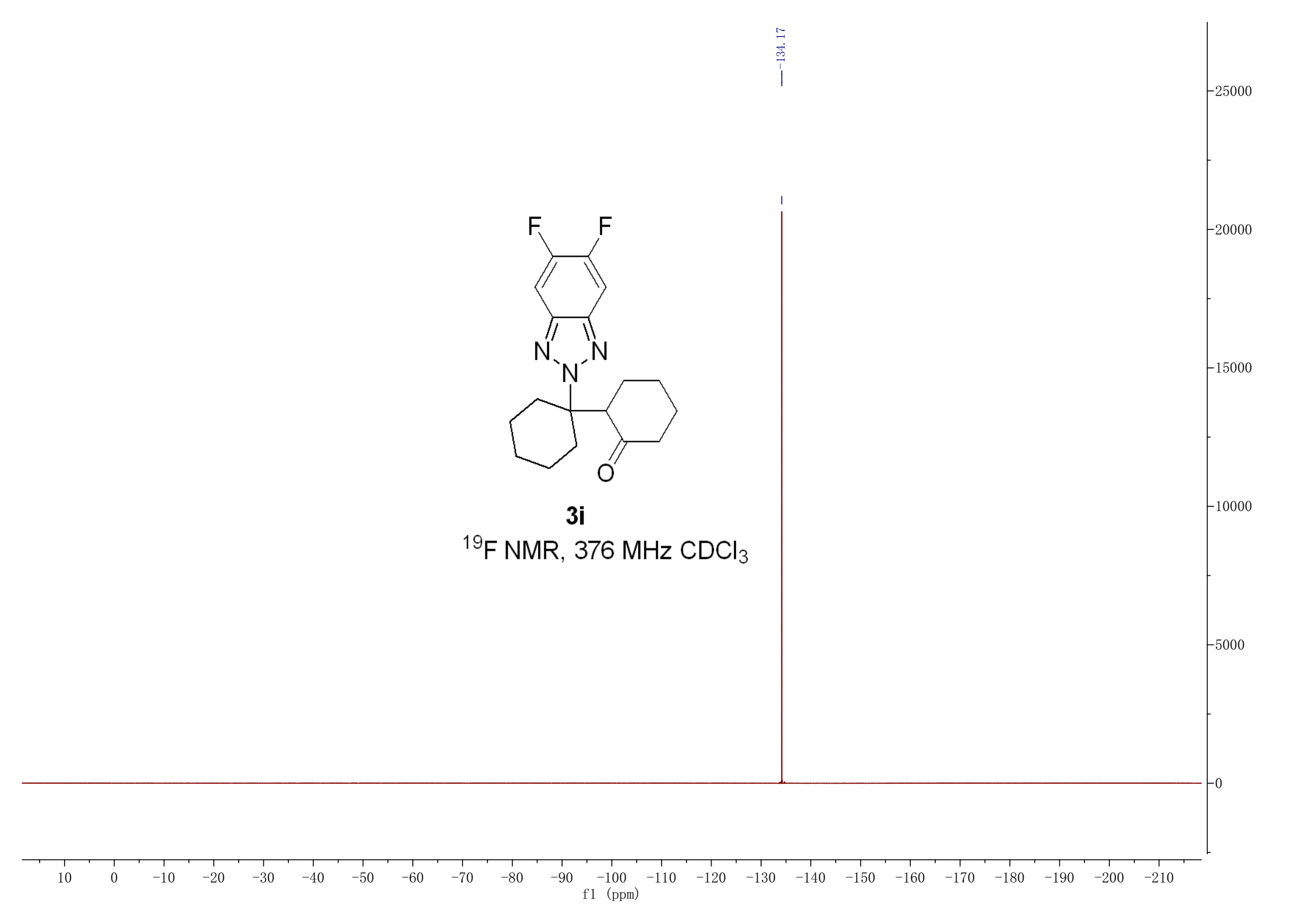
1.05
2.04
1.97
6.17
2.12
1.05
7.48
2.08
2.14
f1 (ppm)


210.54
143.14
136.53
116.93
77.58
77.16
76.74
71.42
60.85
44.14
34.43
28.76
28.31
28.00
25.39
25.07
22.19
22.04
21.02
Me
Me
N
N
N
O
3h
13C NMR, 75 MHz CDCl3
f1 (ppm)

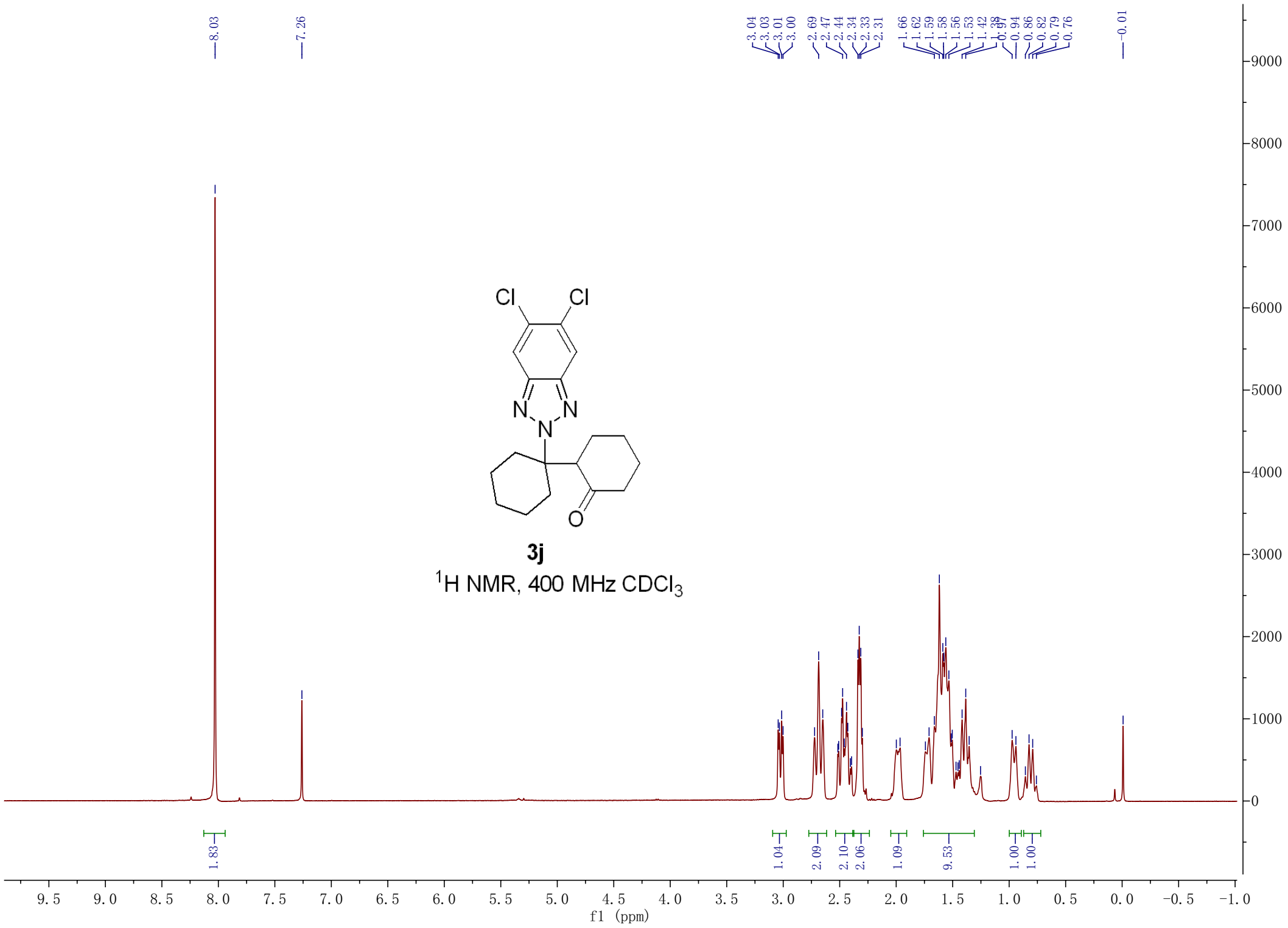

7.62
7.60
7.58
7.26
3.03
3.02
3.00
2.99
2.68
2.47
2.43
2.33
2.32
2.31
1.66
1.63
1.61
1.58
1.55
1.52
1.41
0.94
0.86
0.83
0.80
0.77
0.76
0.01
F
F
N
N
N
O
3i
1H NMR, 400 MHz CDCl3
1.89
1.00
2.04
2.06
2.11
1.10
9.75
1.03
1.07
f1 (ppm)

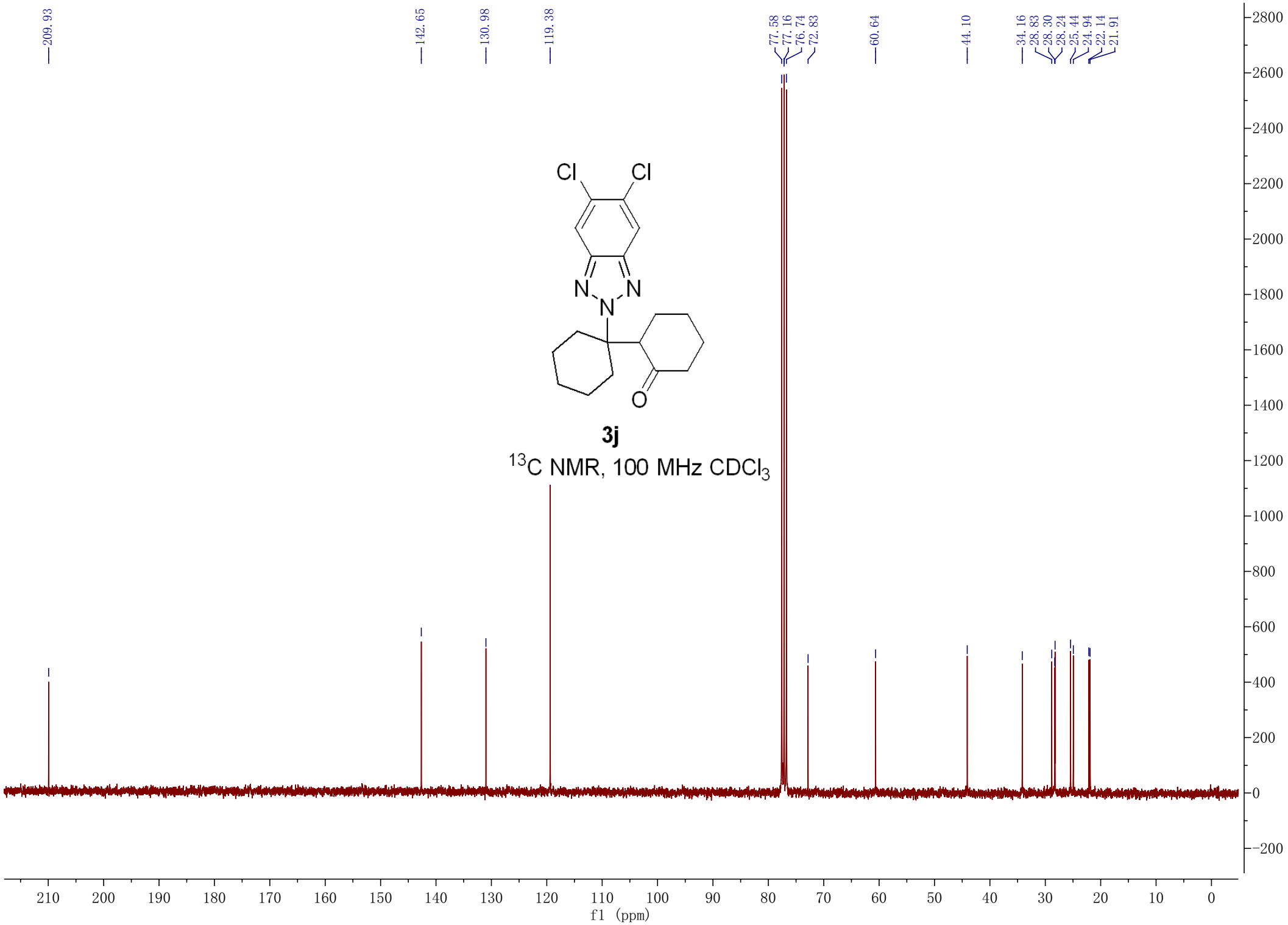

210.06
152.68
152.49
150.18
150.00
139.50
139.44
104.07
104.00
103.92
103.85
77.48
77.16
76.84
72.45
60.66
44.10
34.16
28.83
28.25
25.44
24.96
22.13
21.93
F
F
N
N
N
O
3i
13C NMR, 100 MHz CDCl3
f1 (ppm)

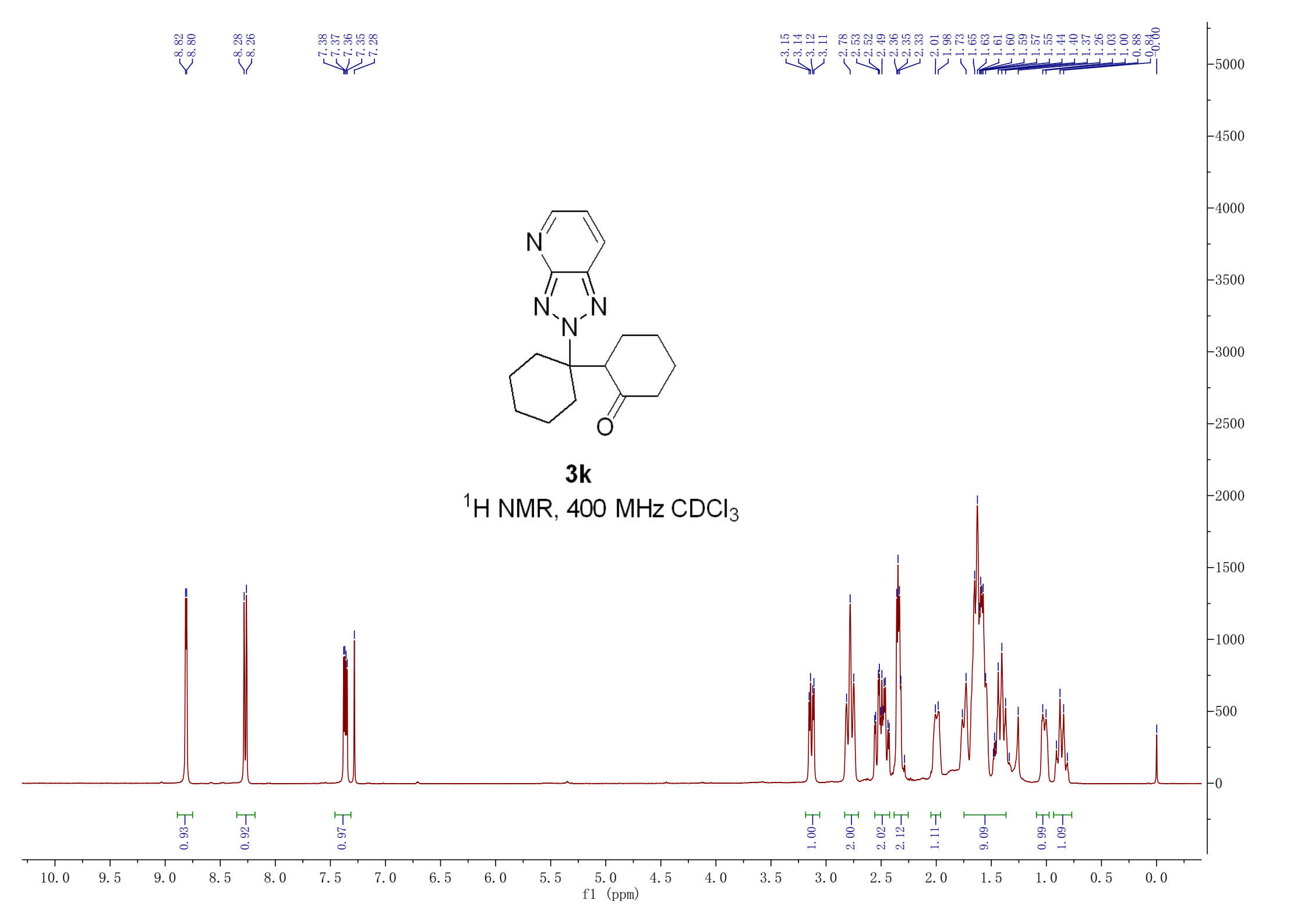


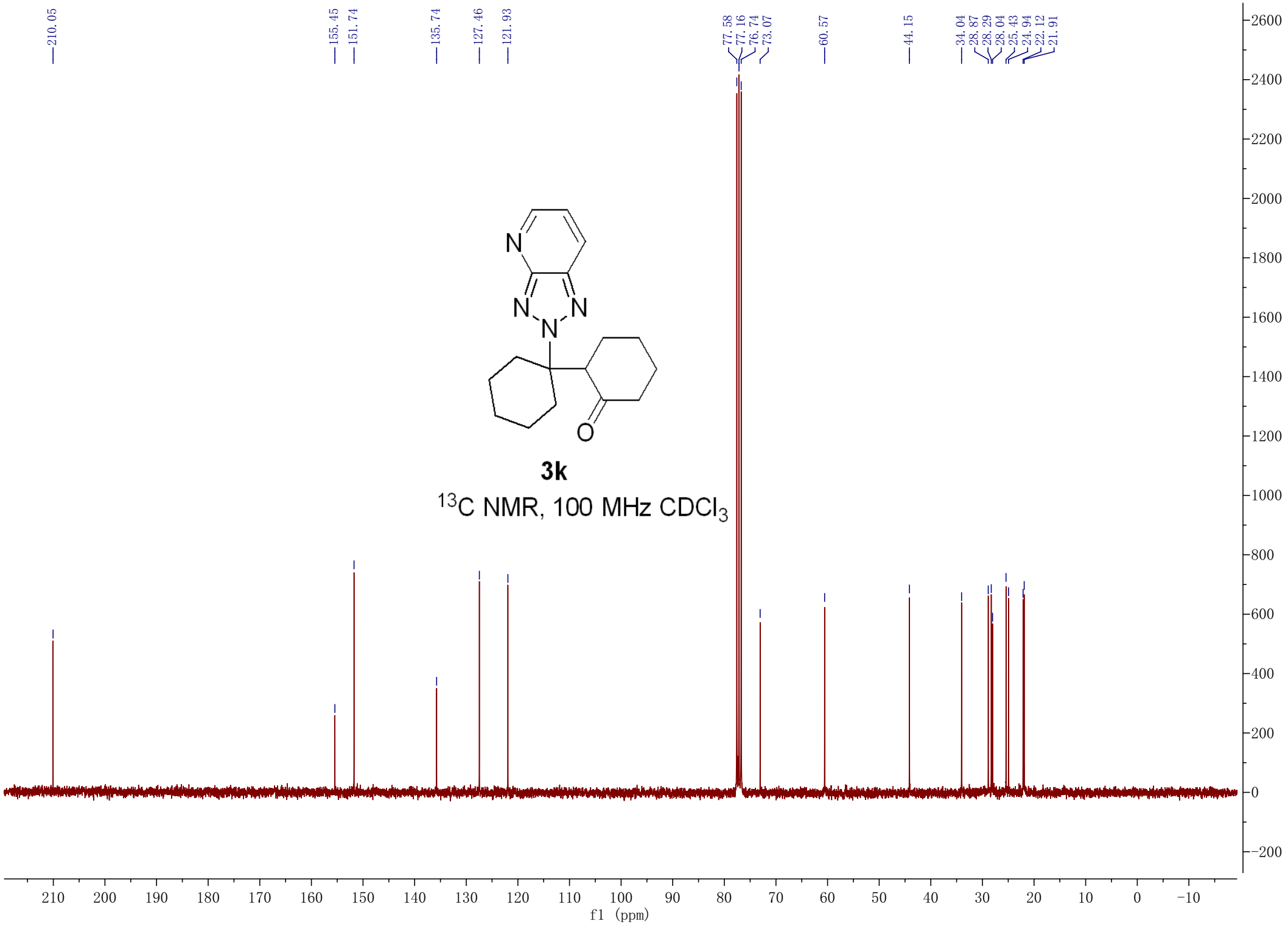

**3i**

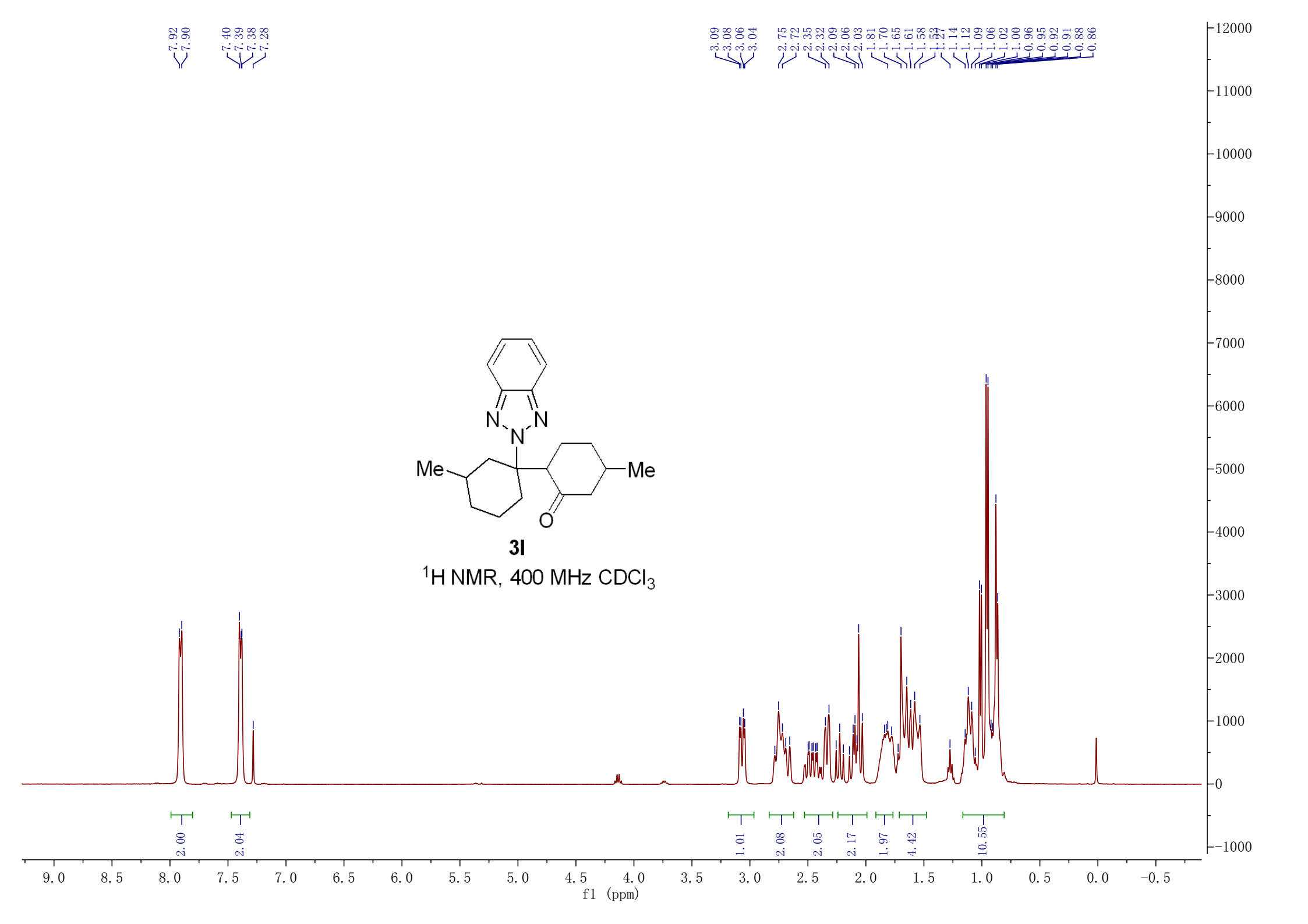
$^{19}$F NMR, 376 MHz $CDCl_3$

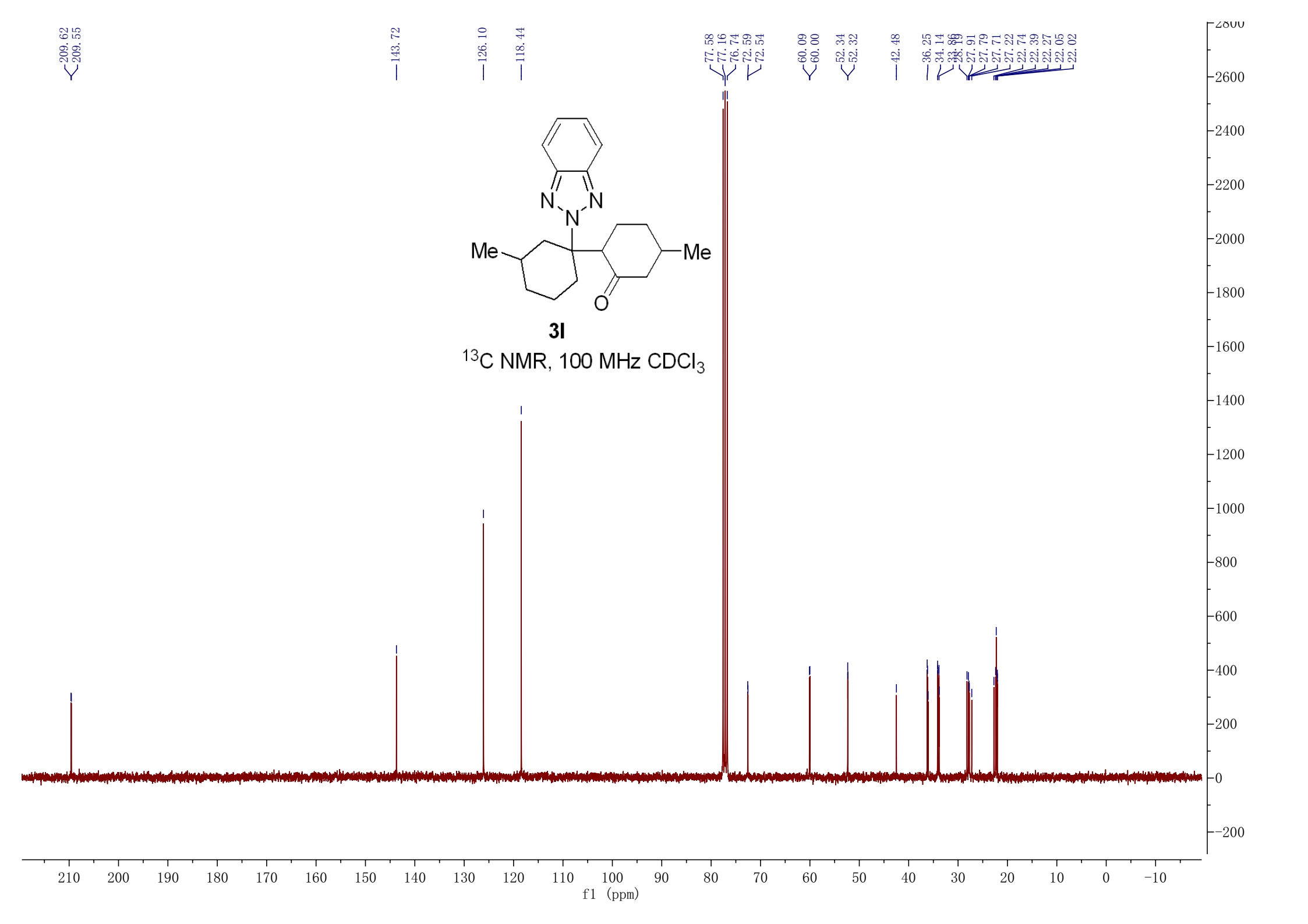

8.03
7.26
3.04
3.03
3.01
3.00
2.69
2.47
2.44
2.34
2.33
2.31
1.66
1.62
1.59
1.58
1.56
1.53
1.42
1.38
0.97
0.94
0.86
0.82
0.79
0.76
-0.01
Cl
Cl
N
N
N
O
3j
1H NMR, 400 MHz CDCl3
1.83
1.04
2.09
2.10
2.06
1.09
9.53
1.00
1.00
f1 (ppm)

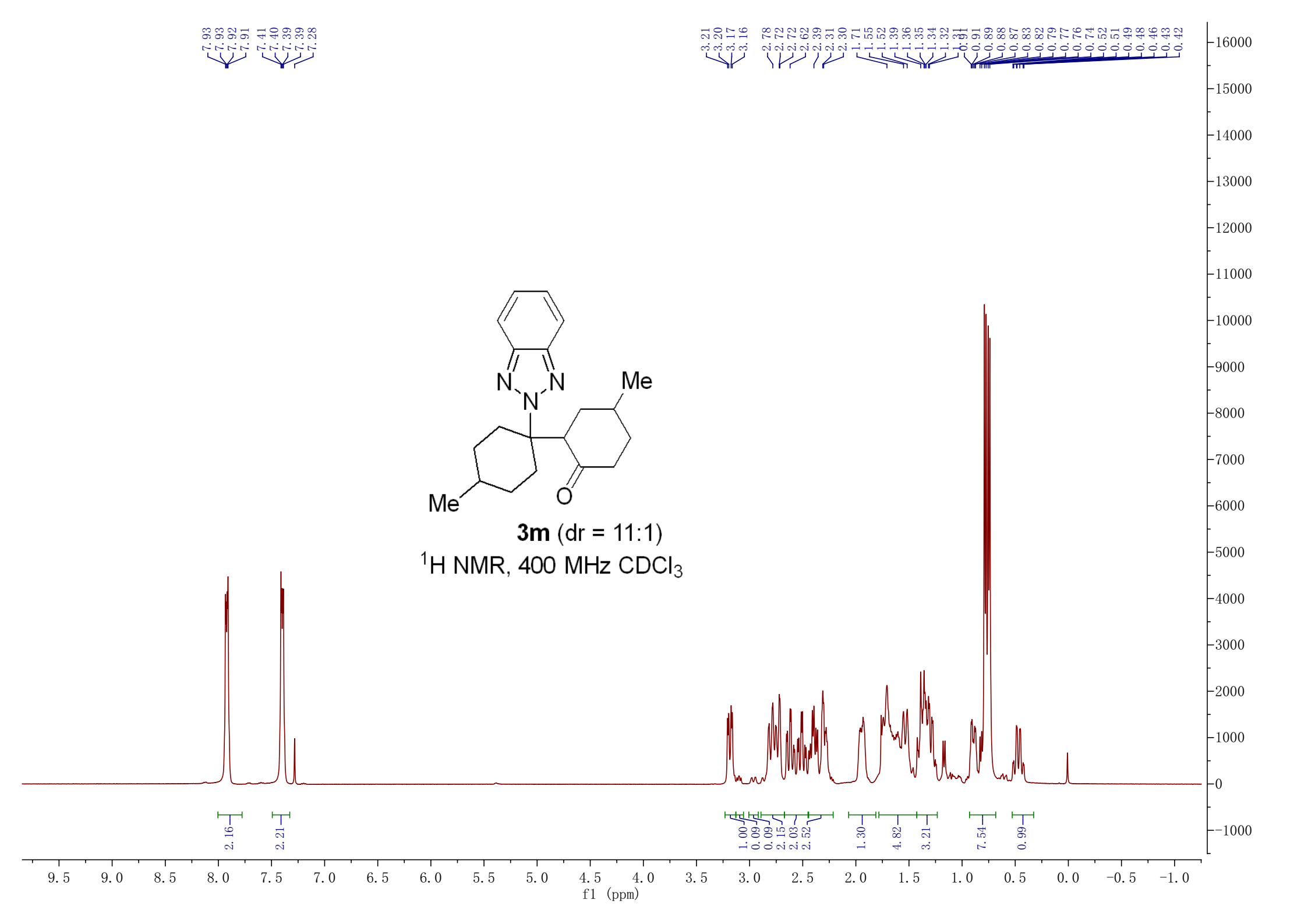

209.93
142.65
130.98
119.38
77.58
77.16
76.74
72.83
60.64
44.10
34.16
28.83
28.30
28.24
25.44
24.94
22.14
21.91
Cl
Cl
N
N
N
O
3j
13C NMR, 100 MHz CDCl3
f1 (ppm)

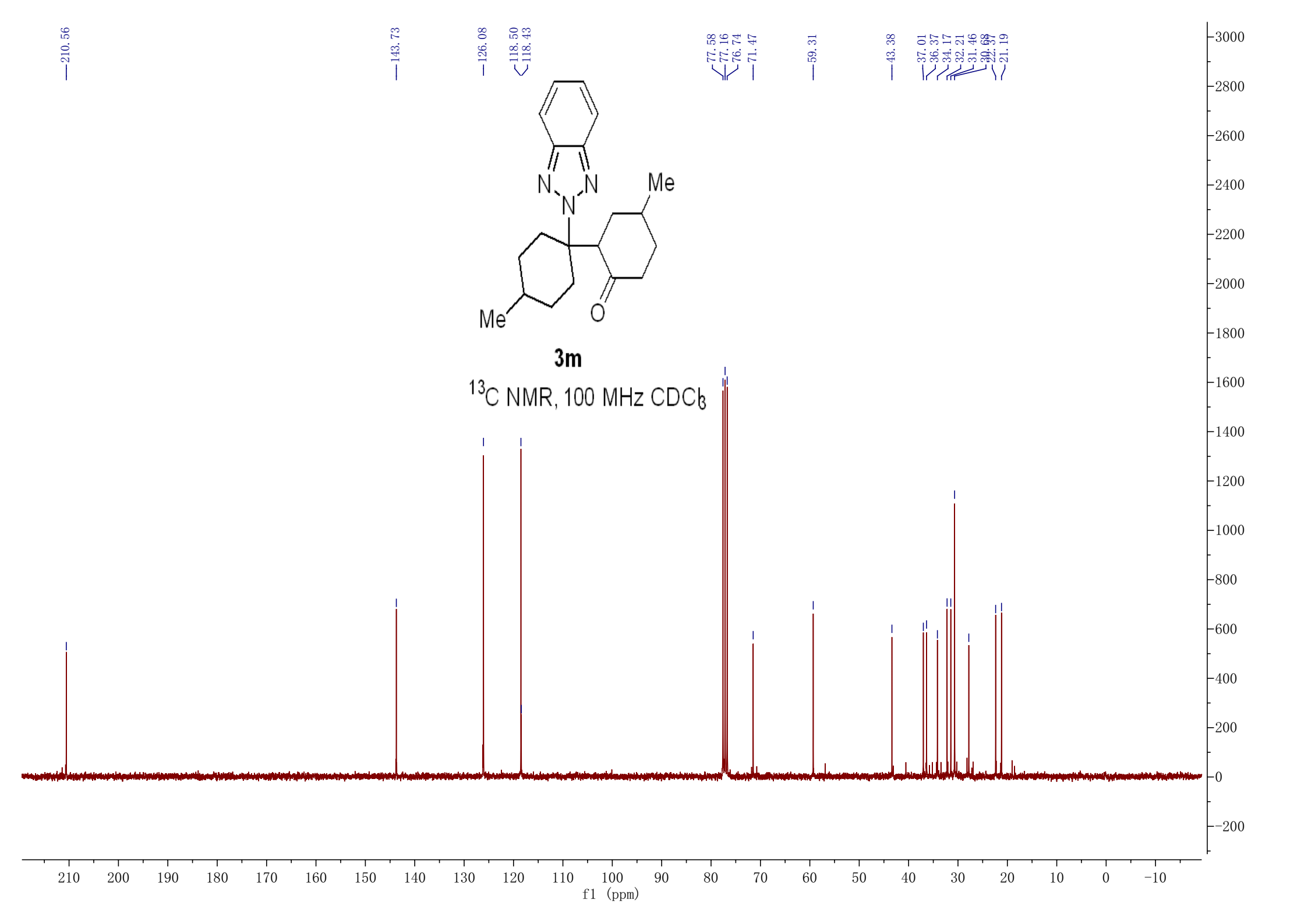

8.82
8.80
8.28
8.26
7.38
7.37
7.36
7.35
7.28
3.15
3.14
3.12
3.11
2.78
2.53
2.52
2.49
2.36
2.35
2.33
2.01
1.98
1.73
1.65
1.63
1.61
1.60
1.59
1.57
1.55
1.44
1.40
1.37
1.26
1.03
1.00
0.88
0.84
0.00
N
N
N
N
O
3k
1H NMR, 400 MHz CDCl3
0.93
0.92
0.97
1.00
2.00
2.02
2.12
1.11
9.09
0.99
1.09
f1 (ppm)

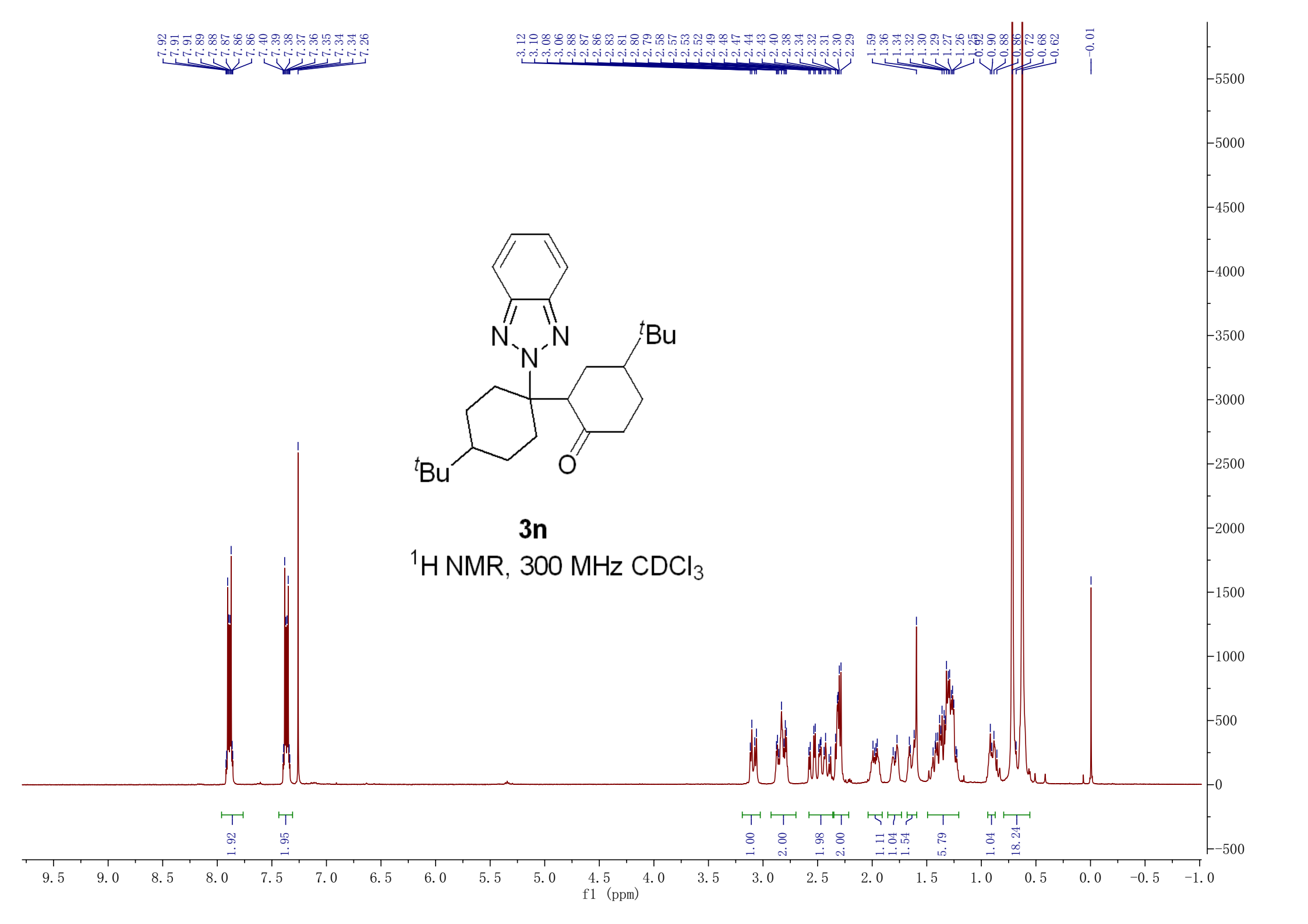

210.05
155.45
151.74
135.74
127.46
121.93
77.58
77.16
76.74
73.07
60.57
44.15
34.04
28.87
28.29
28.04
25.43
24.94
22.12
21.91
N
N
N
N
O
3k
13C NMR, 100 MHz CDCl3
f1 (ppm)

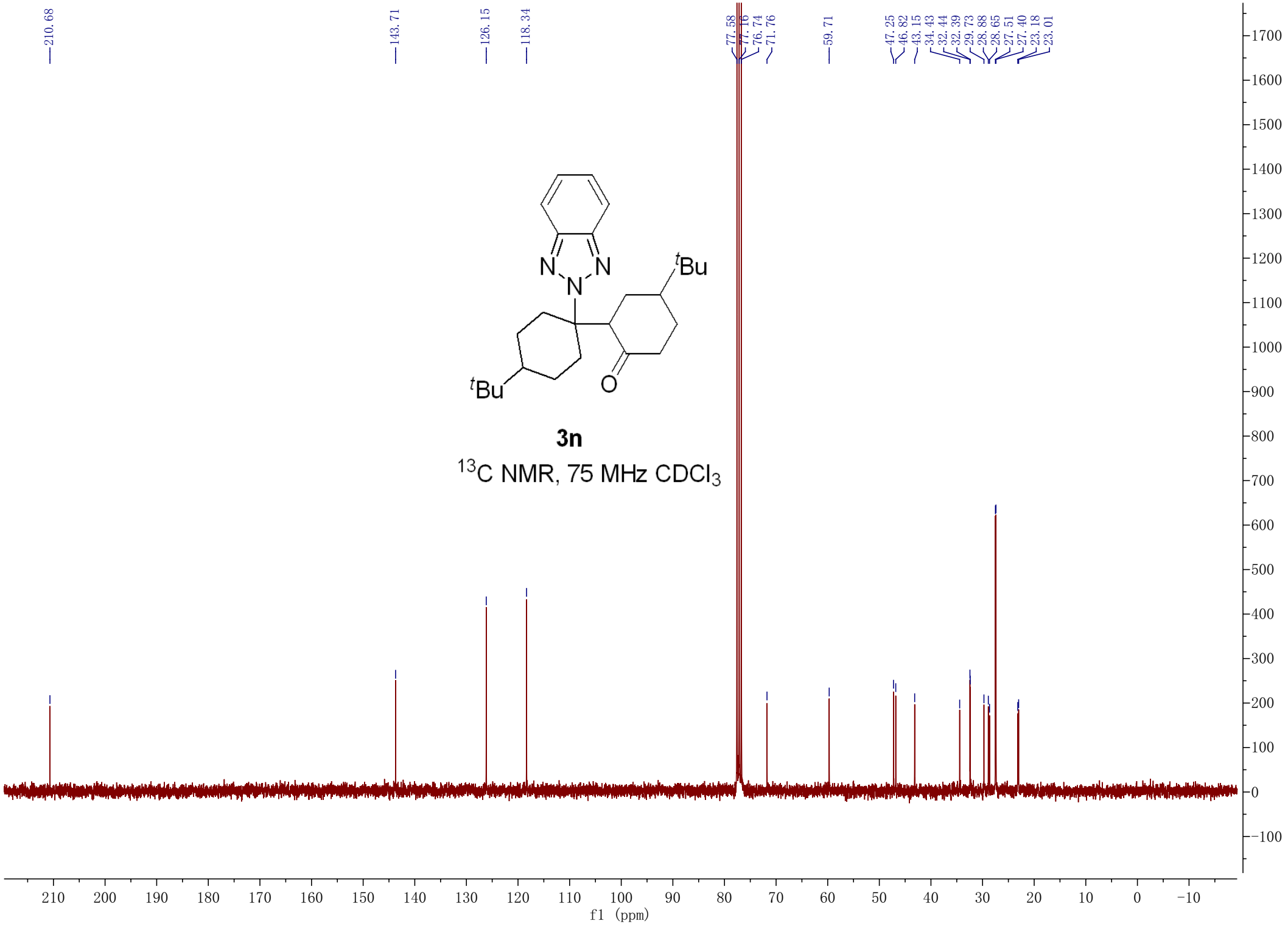

7.92
7.90
7.40
7.39
7.38
7.28
3.09
3.08
3.06
3.04
2.75
2.72
2.35
2.32
2.09
2.06
2.03
1.81
1.70
1.65
1.61
1.58
1.14
1.12
1.09
1.06
1.02
1.00
0.96
0.95
0.92
0.91
0.88
0.86
Me
Me
O
N
N
N
3l
1H NMR, 400 MHz CDCl3
2.00
2.04
1.01
2.08
2.05
2.17
1.97
4.42
10.55
f1 (ppm)

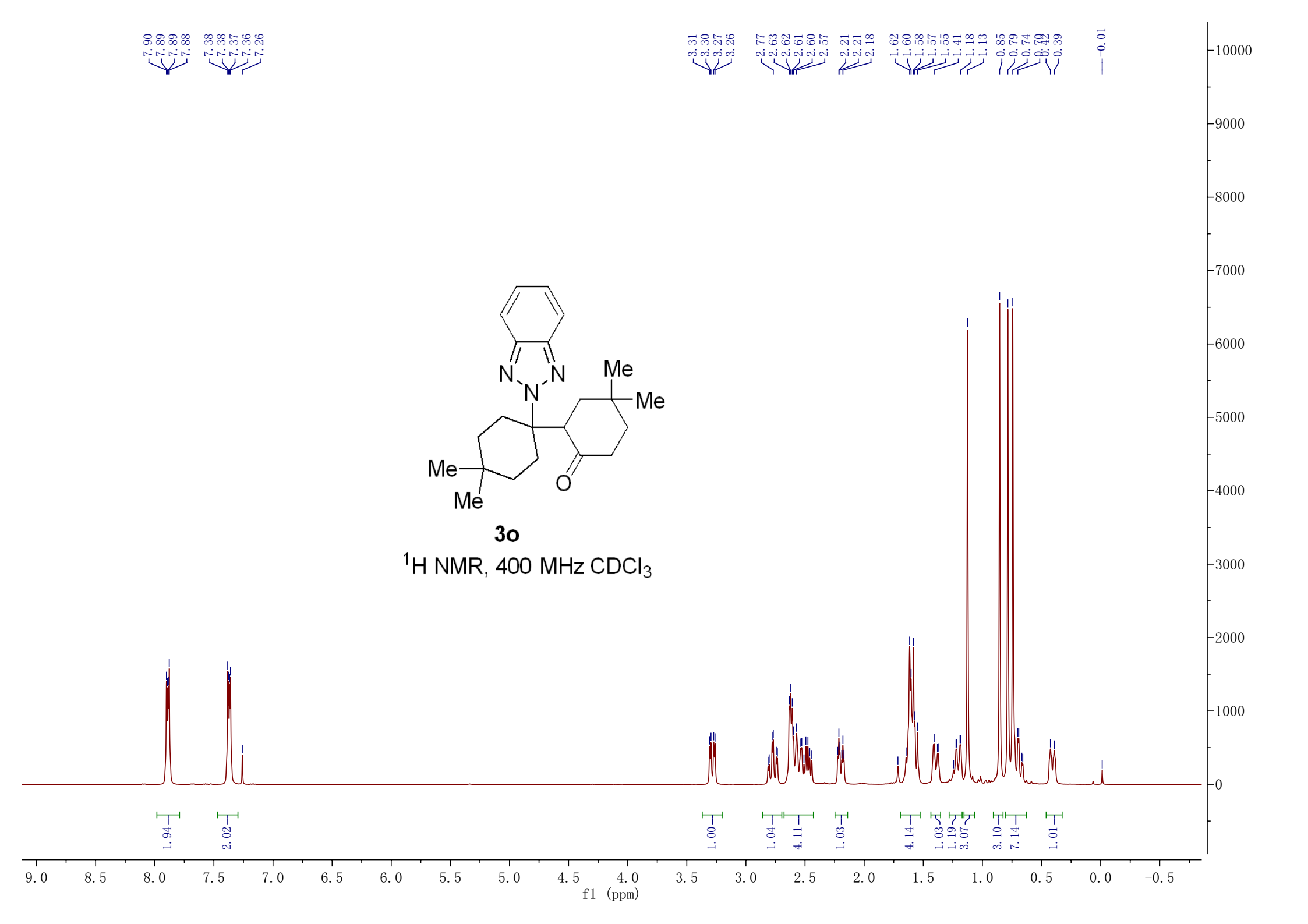

209.62
209.55
143.72
126.10
118.44
77.58
77.16
76.74
72.59
72.54
60.09
60.00
52.34
52.32
42.48
36.25
34.14
27.91
27.79
27.71
27.22
22.74
22.39
22.27
22.05
22.02
N
N
N
Me
Me
O
3l
13C NMR, 100 MHz CDCl3
f1 (ppm)


7.93
7.93
7.92
7.91
7.41
7.40
7.39
7.39
7.28
3.21
3.20
3.17
3.16
2.78
2.72
2.72
2.62
2.39
2.31
2.30
1.71
1.55
1.52
1.39
1.36
1.35
1.34
1.32
0.91
0.89
0.88
0.87
0.83
0.82
0.79
0.77
0.76
0.74
0.52
0.51
0.49
0.48
0.46
0.43
0.42
N
N
N
Me
Me
O
3m (dr = 11:1)
1H NMR, 400 MHz CDCl3
2.16
2.21
1.00
0.09
0.09
2.15
2.03
2.52
1.30
4.82
3.21
7.54
0.99
f1 (ppm)


210.56
143.73
126.08
118.50
118.43
77.58
77.16
76.74
71.47
59.31
43.38
37.01
36.37
34.17
32.21
31.46
21.19
N
N
N
Me
Me
O
3m
13C NMR, 100 MHz CDCl3
f1 (ppm)


7.92 7.91 7.91 7.89 7.88 7.87 7.86 7.86 7.40 7.39 7.38 7.37 7.36 7.35 7.34 7.34 7.26
3.12 3.10 3.08 3.06 2.88 2.87 2.86 2.83 2.81 2.80 2.79 2.58 2.57 2.53 2.52 2.49 2.48 2.47 2.44 2.43 2.40 2.38 2.34 2.32 2.31 2.30 2.29
1.59 1.36 1.34 1.32 1.30 1.29 1.27 1.26 0.92 0.90 0.88 0.86 0.72 0.68 0.62
-0.01
tBu
tBu
N
N
N
O
3n
1H NMR, 300 MHz CDCl3
1.92 1.95 1.00 2.00 1.98 2.00 1.11 1.04 1.54 5.79 1.04 18.24
f1 (ppm)


210.68
143.71
126.15
118.34
77.58
77.16
76.74
71.76
59.71
47.25
46.82
43.15
34.43
32.44
32.39
29.73
28.88
28.65
27.51
27.40
23.18
23.01
tBu
tBu
O
N
N
N
3n
13C NMR, 75 MHz CDCl3
f1 (ppm)


7.90
7.89
7.89
7.88
7.38
7.38
7.37
7.36
7.26
3.31
3.30
3.27
3.26
2.77
2.63
2.62
2.61
2.60
2.57
2.21
2.21
2.18
1.62
1.60
1.58
1.57
1.55
1.41
1.18
1.13
0.85
0.79
0.74
0.39
0.01
N
N
N
Me
Me
Me
Me
O
3o
1H NMR, 400 MHz CDCl3
1.94
2.02
1.00
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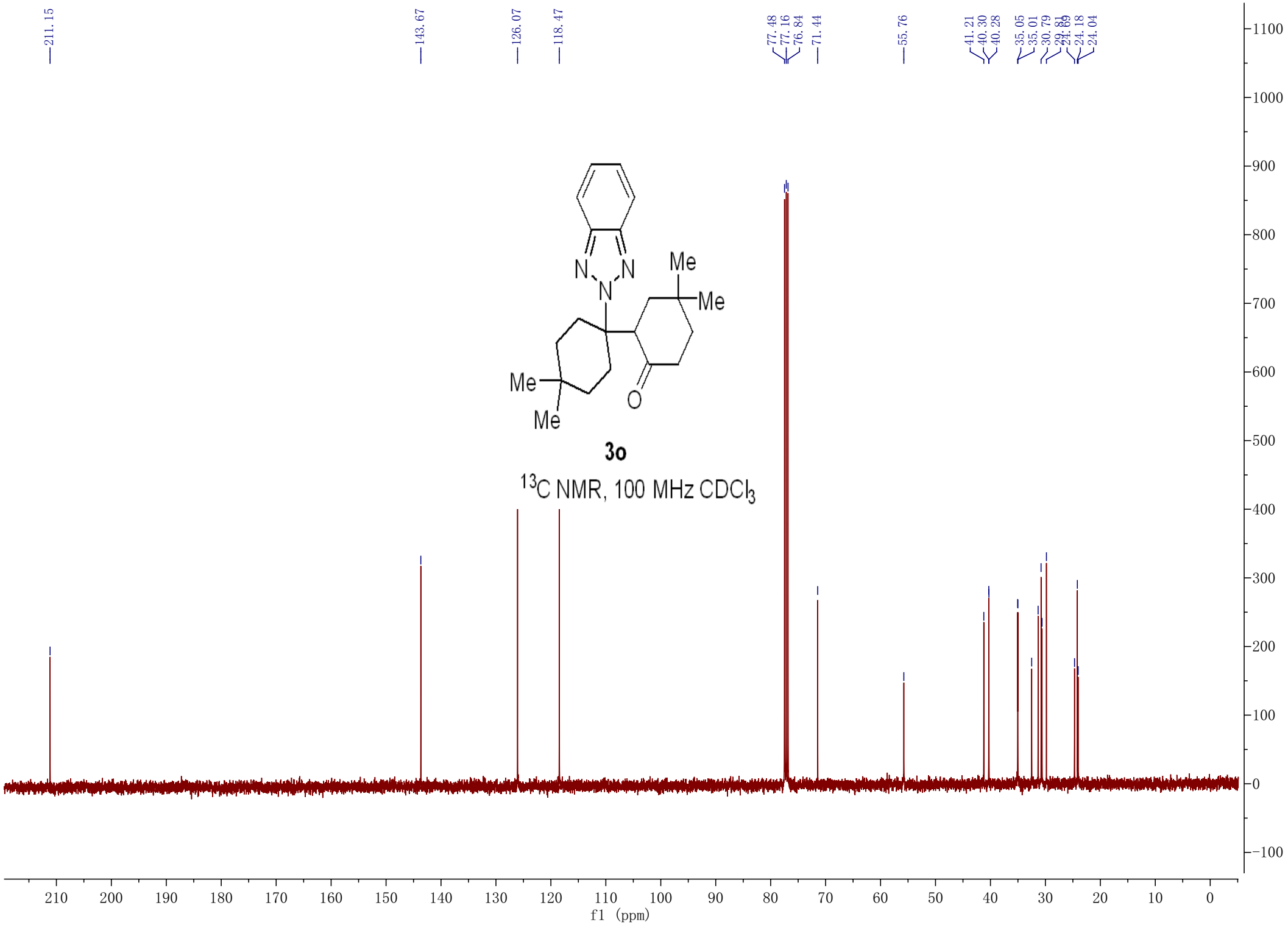
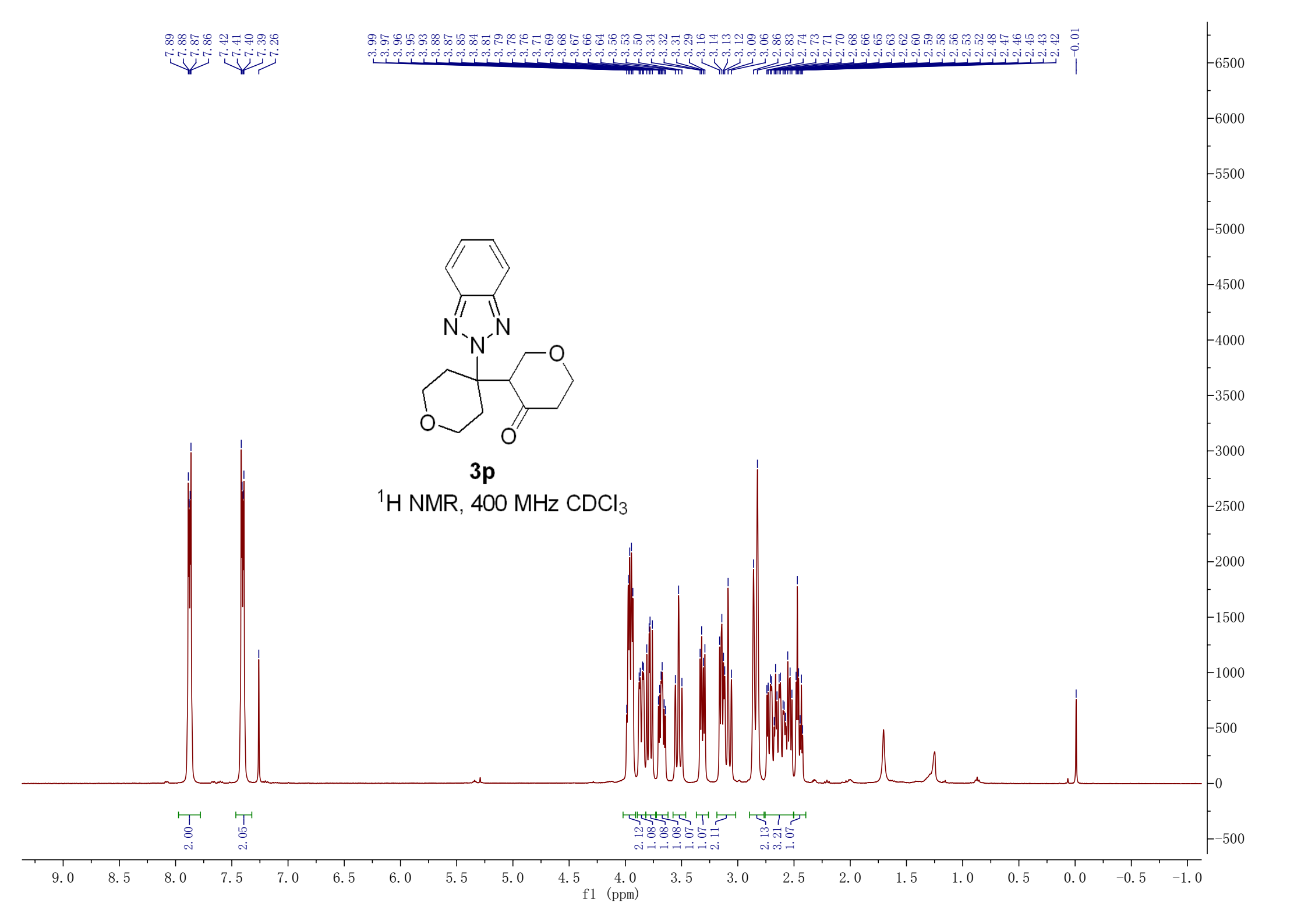
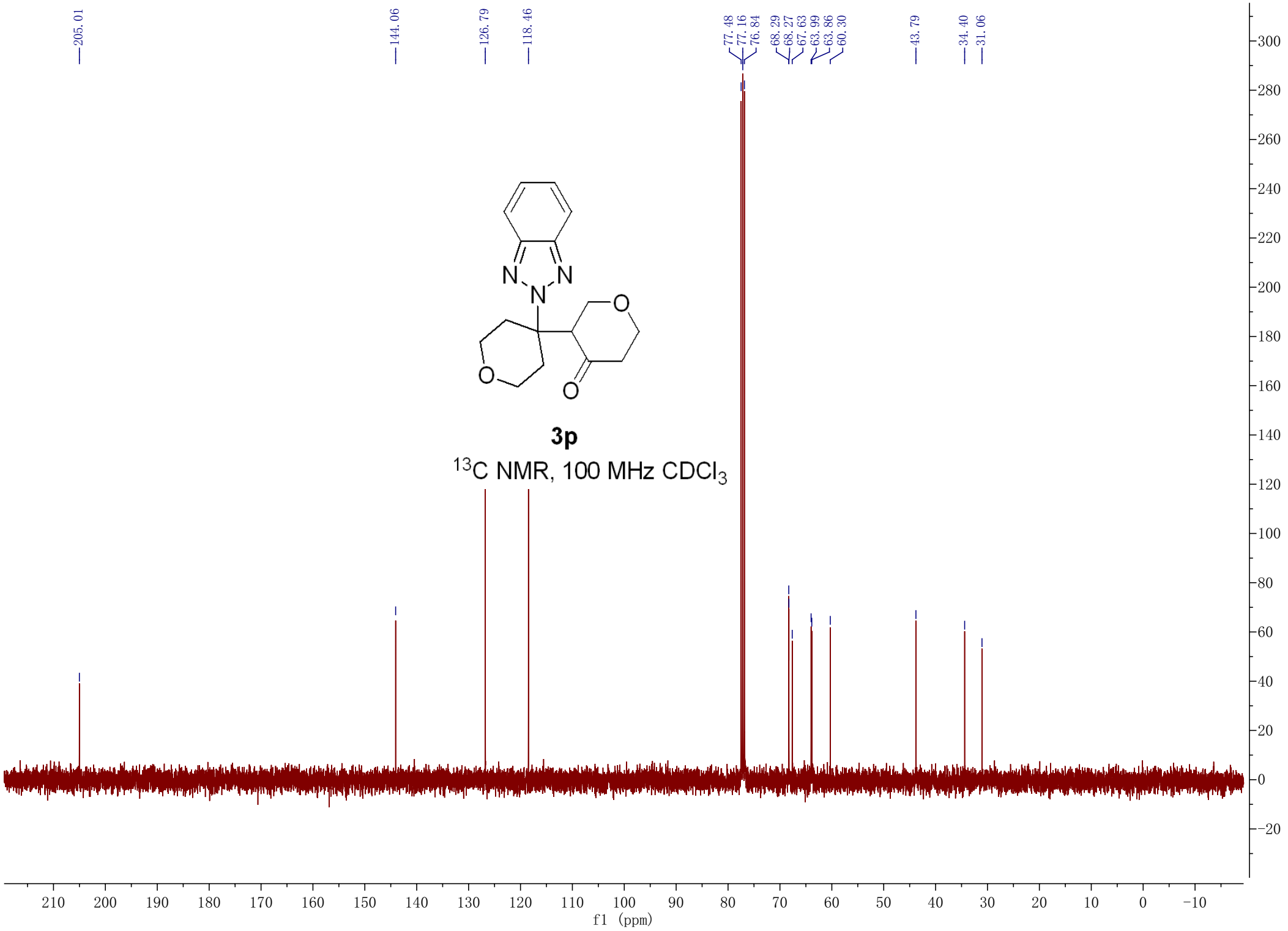
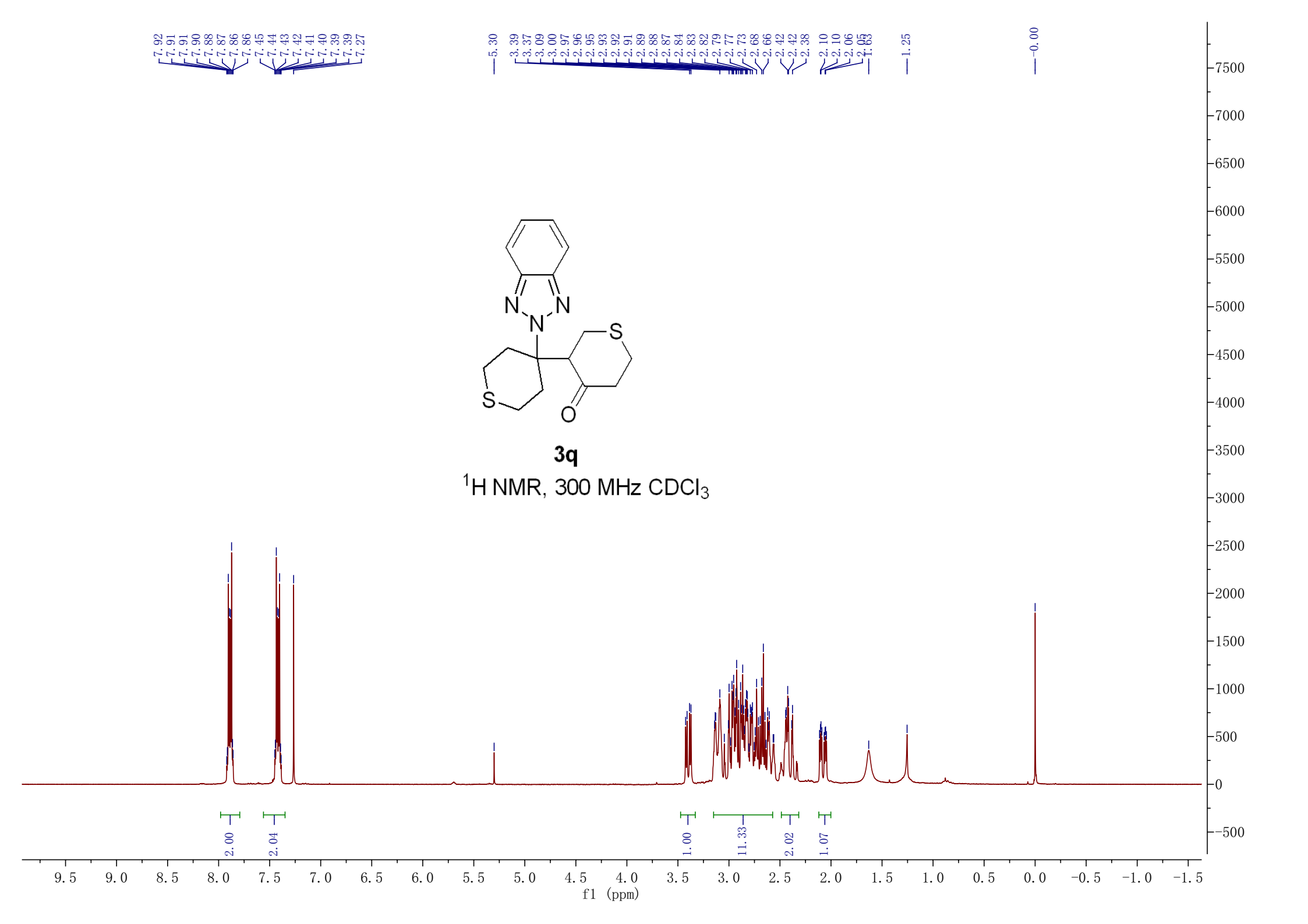
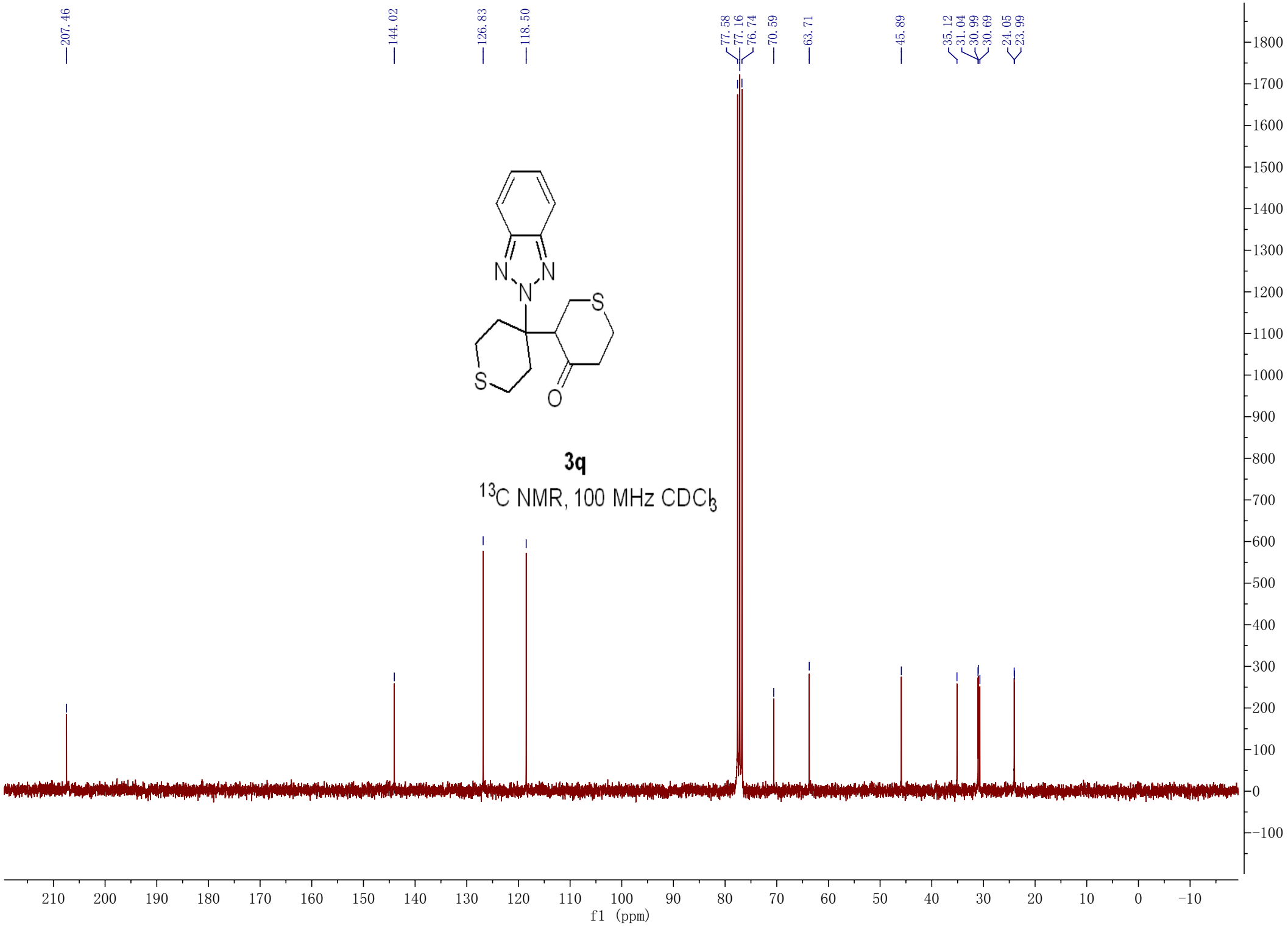
4.14
1.03
1.19
3.07
3.10
7.14
1.01
f1 (ppm)

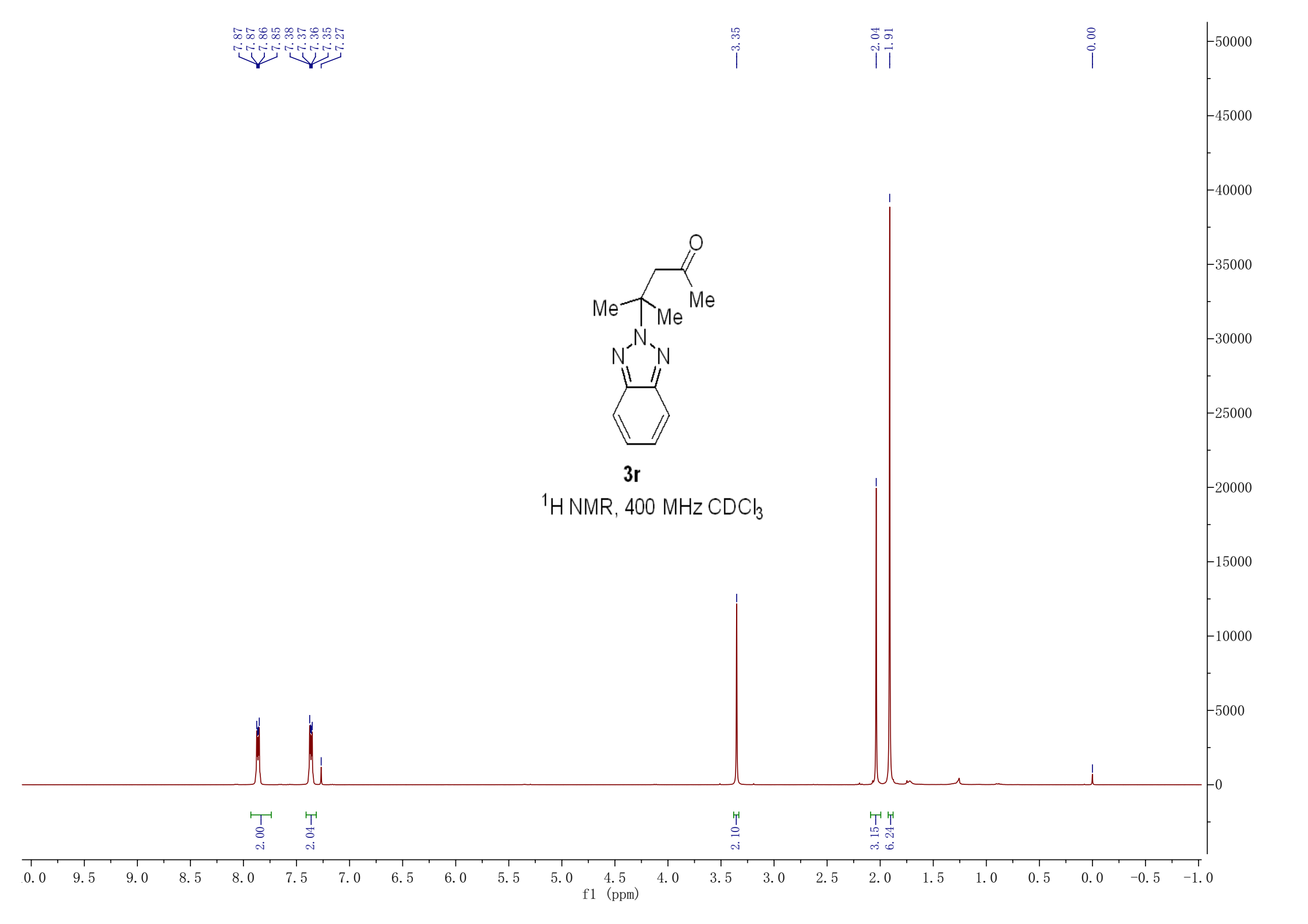

211.15
143.67
126.07
118.47
77.48
77.16
76.84
71.44
55.76
41.21
40.30
40.28
35.05
35.01
30.79
24.69
24.18
24.04
Me
Me
Me
Me
N
N
N
O
3o
13C NMR, 100 MHz CDCl3
f1 (ppm)

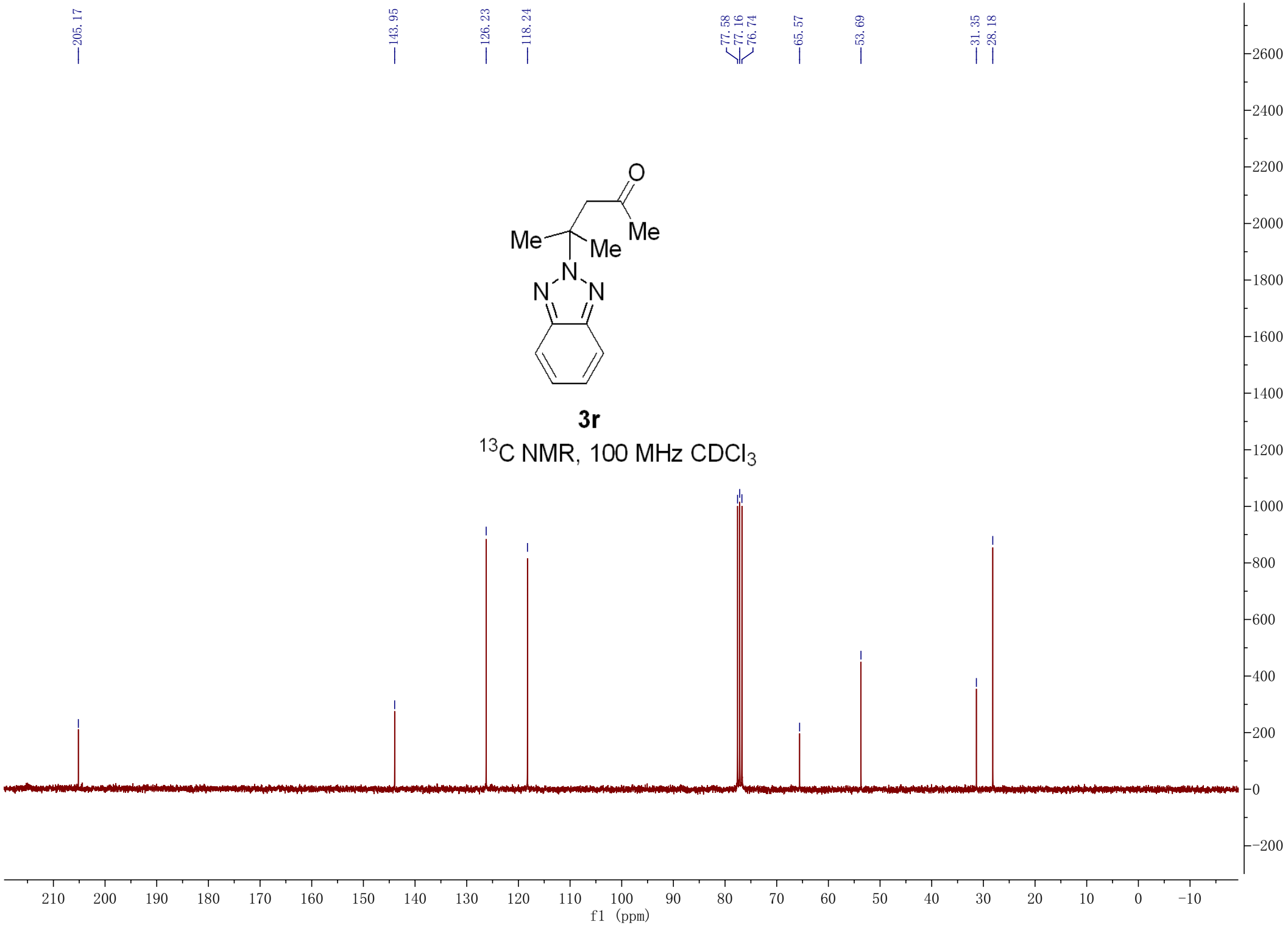

7.89
7.88
7.87
7.86
7.42
7.41
7.40
7.39
7.26
3.99
3.97
3.96
3.95
3.93
3.88
3.87
3.85
3.84
3.81
3.79
3.78
3.76
3.71
3.69
3.68
3.67
3.66
3.64
3.56
3.53
3.50
3.34
3.32
3.31
3.29
3.16
3.14
3.13
3.12
3.09
3.06
2.86
2.83
2.74
2.73
2.71
2.70
2.68
2.66
2.65
2.63
2.62
2.60
2.59
2.58
2.56
2.53
2.52
2.48
2.47
2.46
2.45
2.43
2.42
-0.01
N
N
N
O
O
O
3p
1H NMR, 400 MHz CDCl3
2.00
2.05
2.12
1.08
1.08
1.08
1.07
1.07
2.11
2.13
3.21
1.07
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
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)

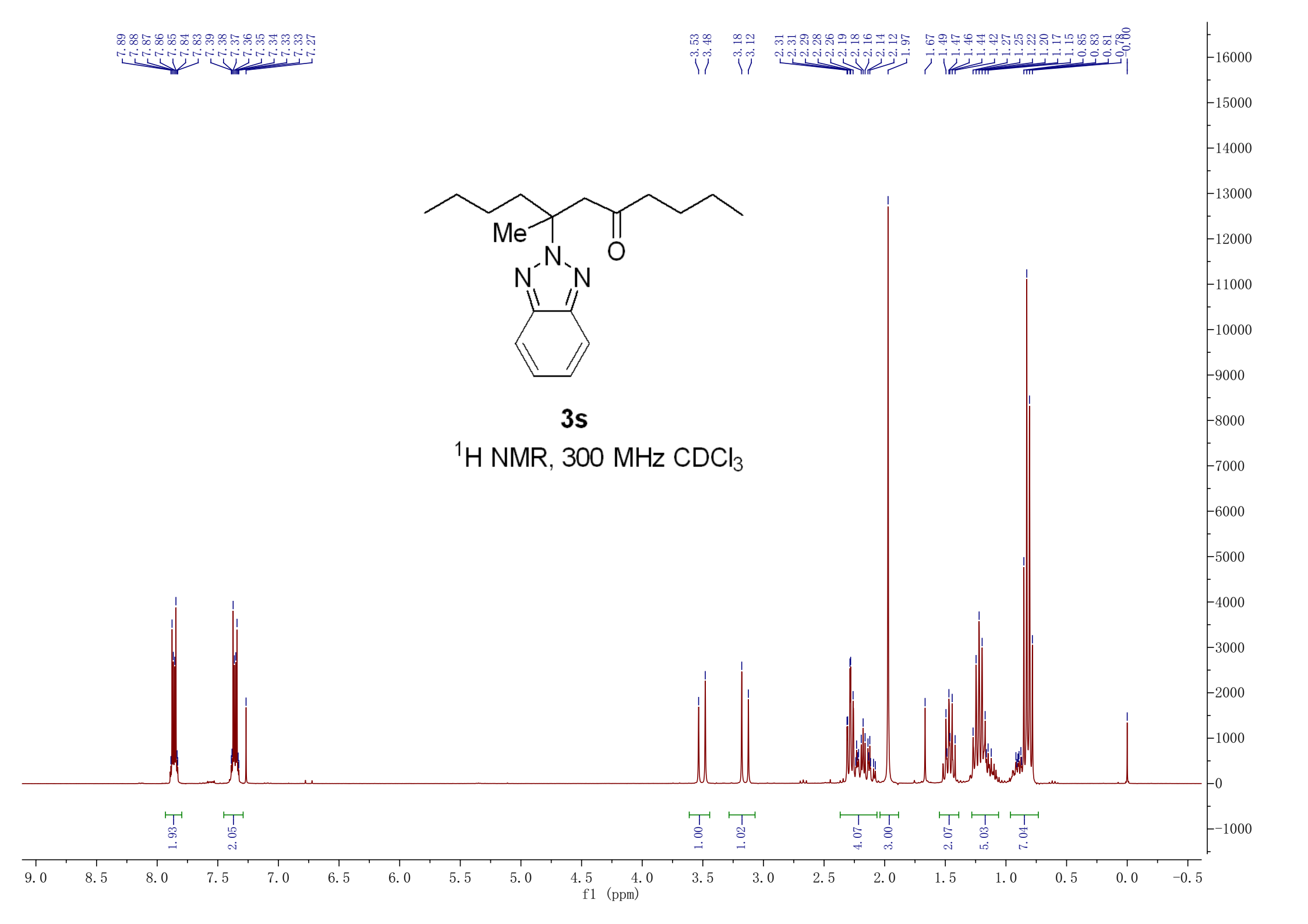

205.01
144.06
126.79
118.46
77.48
77.16
76.84
68.29
68.27
67.63
63.99
63.86
60.30
43.79
34.40
31.06
N
N
N
O
O
O
3p
13C NMR, 100 MHz CDCl3
f1 (ppm)

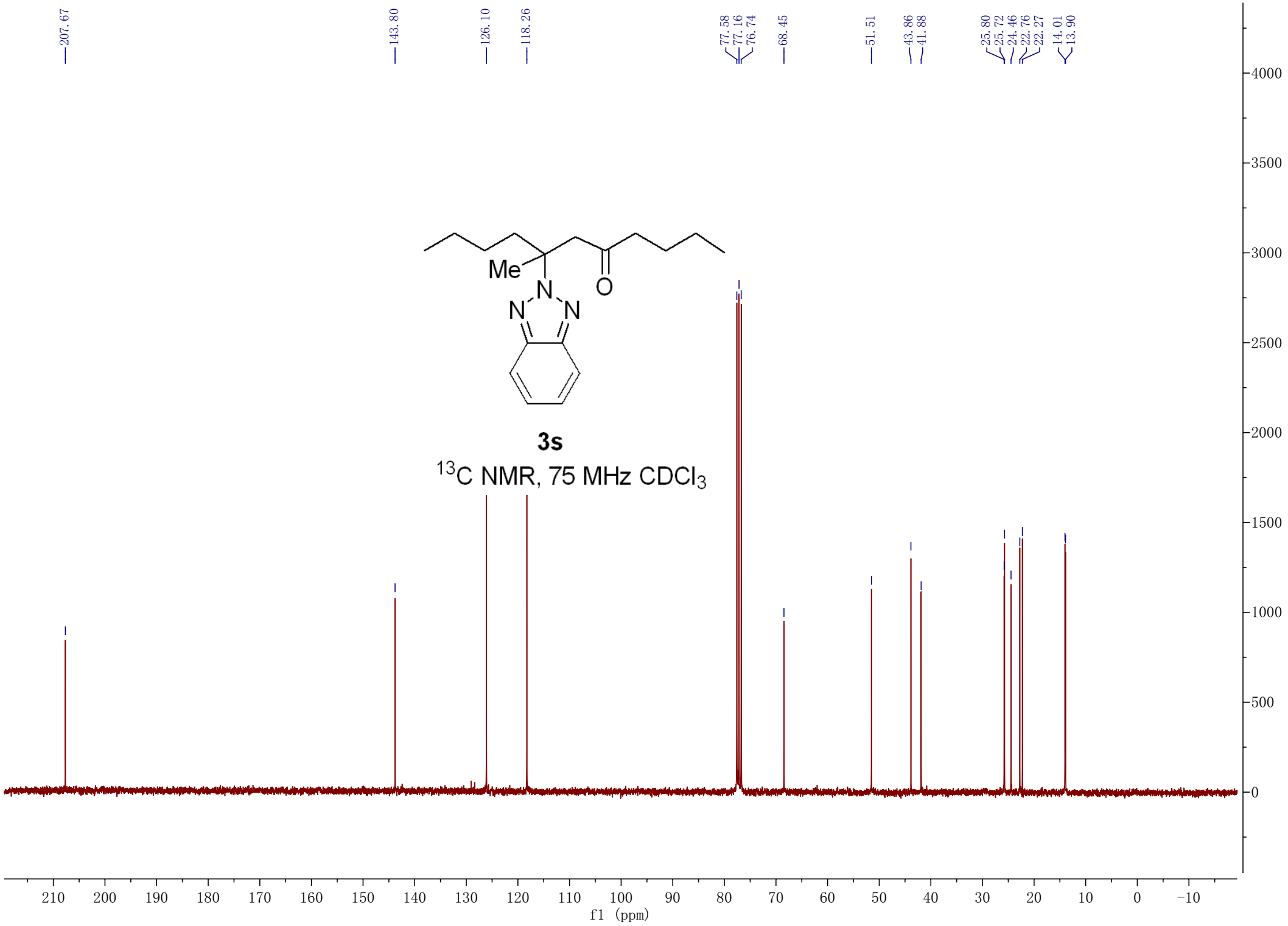

7.92
7.91
7.91
7.90
7.88
7.87
7.86
7.86
7.45
7.44
7.43
7.42
7.41
7.40
7.39
7.39
7.27
5.30
3.39
3.37
3.09
3.00
2.97
2.96
2.95
2.93
2.92
2.91
2.89
2.88
2.87
2.84
2.83
2.82
2.79
2.77
2.73
2.68
2.66
2.42
2.42
2.38
2.10
2.10
2.06
1.63
1.25
0.00
N
N
N
S
S
O
3q
1H NMR, 300 MHz CDCl3
2.00
2.04
1.00
11.33
2.02
1.07
f1 (ppm)

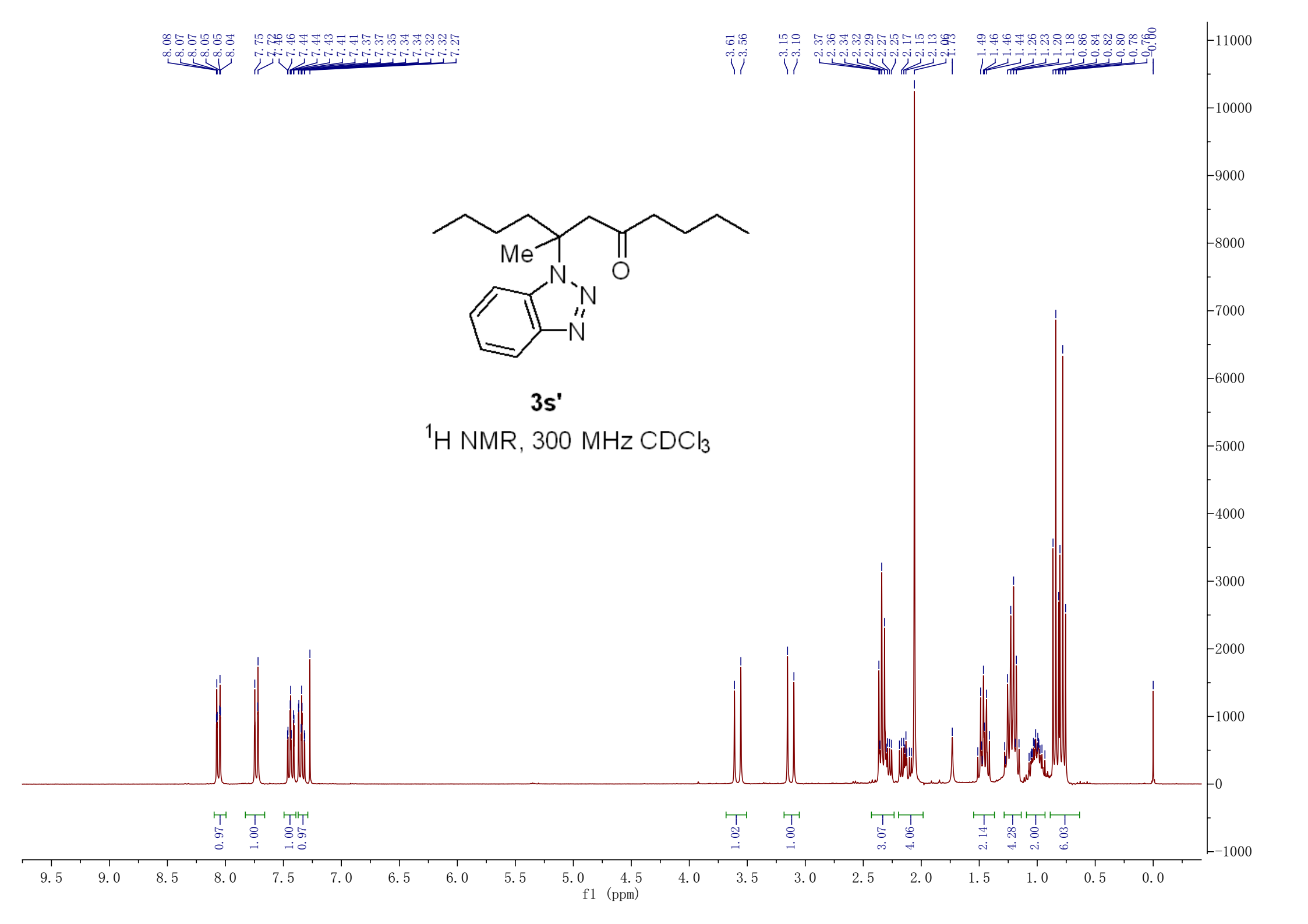

207.46
144.02
126.83
118.50
77.58
77.16
76.74
70.59
63.71
45.89
35.12
31.04
30.99
30.69
24.05
23.99
N
N
N
S
S
O
3q
13C NMR, 100 MHz CDCl3
f1 (ppm)


7.87
7.87
7.86
7.85
7.38
7.37
7.36
7.35
7.27
3.35
2.04
1.91
0.00
O
Me
Me
Me
N
N
N
3r
1H NMR, 400 MHz CDCl3
2.00
2.04
2.10
3.15
6.24
f1 (ppm)


205.17
143.95
126.23
118.24
77.58
77.16
76.74
65.57
53.69
31.35
28.18
O
Me
Me
Me
N
N
N
3r
13C NMR, 100 MHz CDCl3
f1 (ppm)

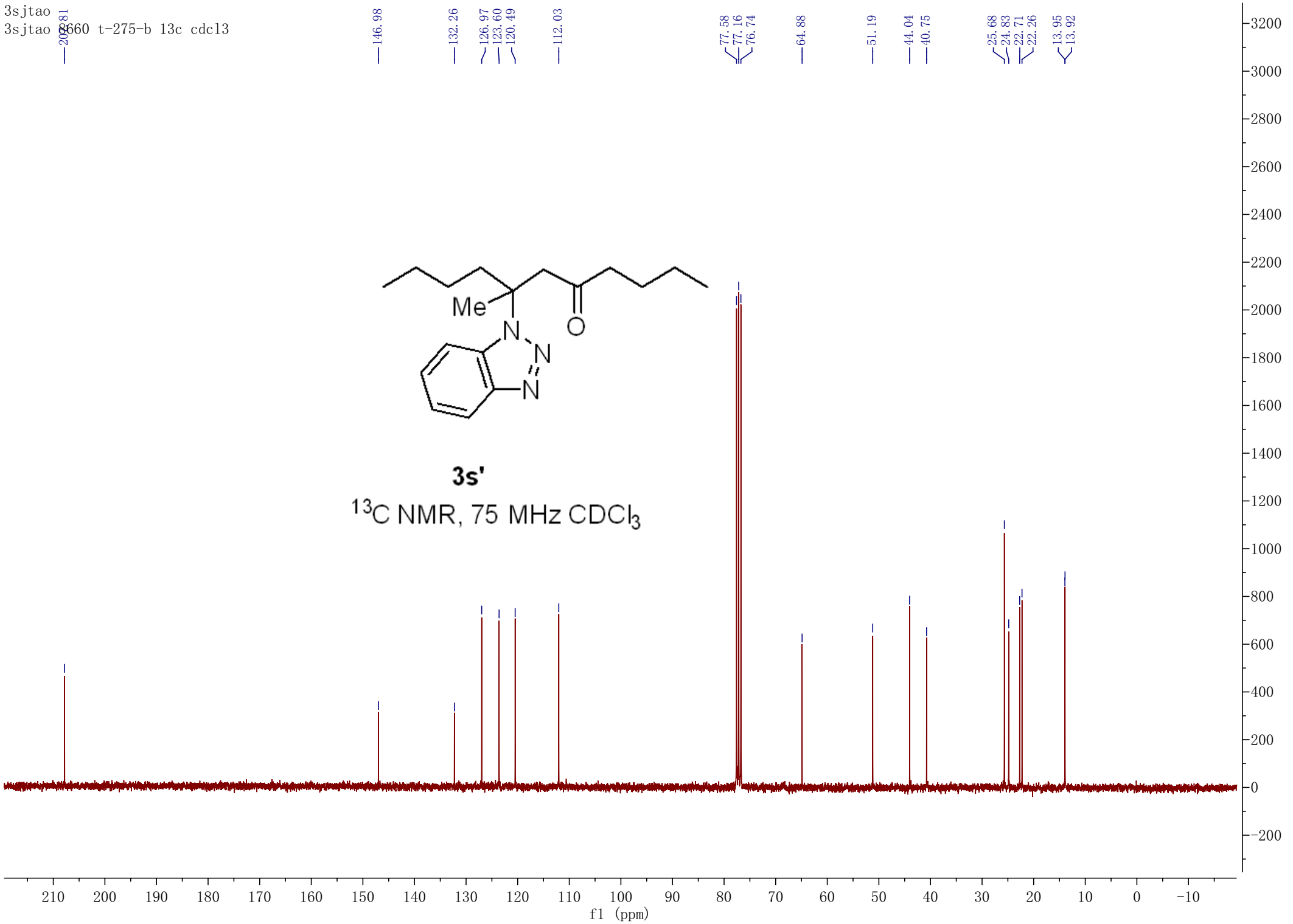

7.89
7.88
7.87
7.86
7.85
7.84
7.83
7.39
7.38
7.37
7.36
7.35
7.34
7.33
7.33
7.27
3.53
3.48
3.18
3.12
2.31
2.31
2.29
2.28
2.26
2.19
2.18
2.16
2.14
2.12
1.97
1.67
1.49
1.47
1.46
1.44
1.42
1.27
1.25
1.22
1.20
1.17
1.15
0.85
0.83
0.81
0.78
0.00
Me
N
N
N
O
3s
1H NMR, 300 MHz CDCl3
1.93
2.05
1.00
1.02
4.07
3.00
2.07
5.03
7.04
f1 (ppm)

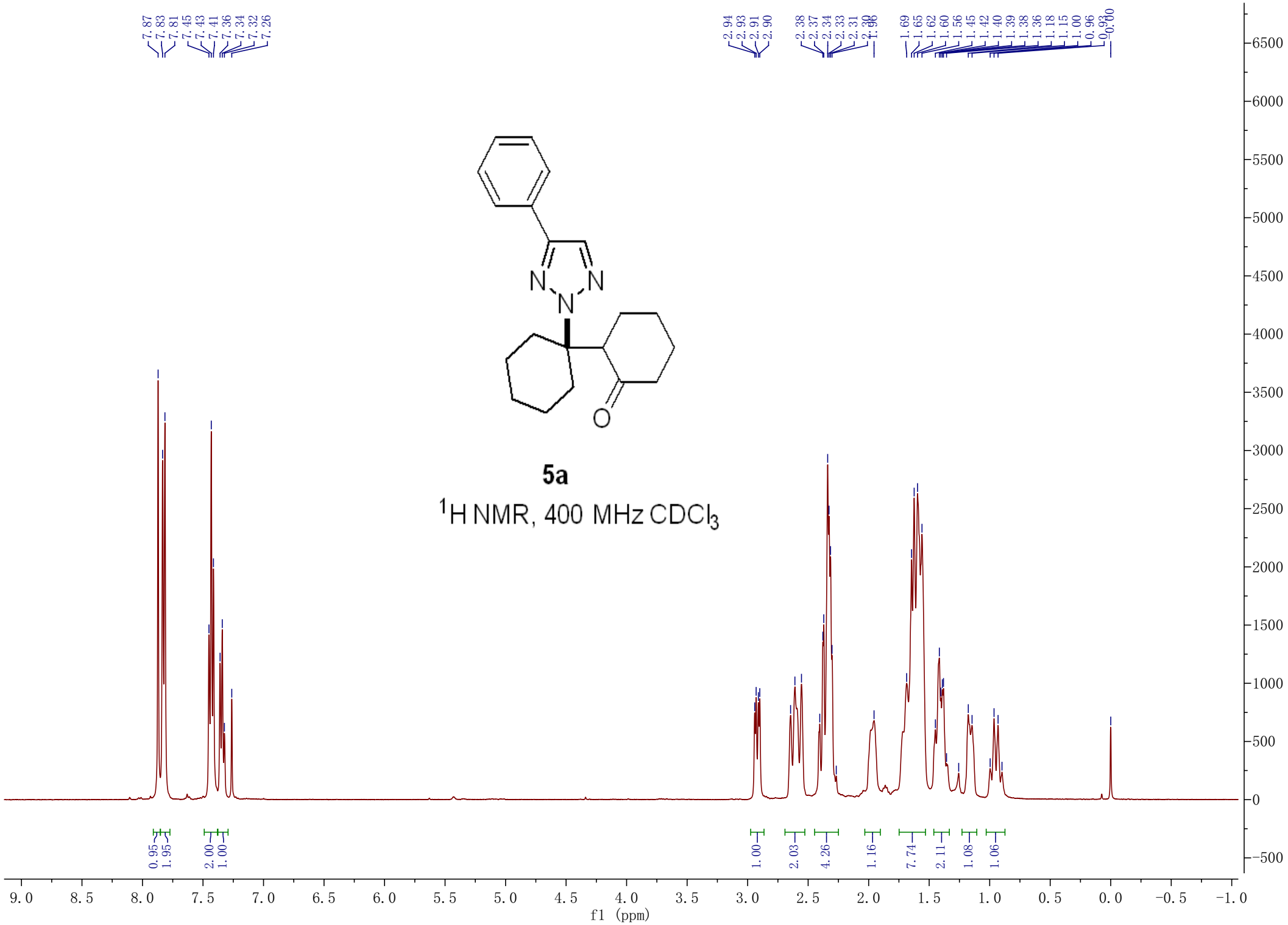

207.67
143.80
126.10
118.26
77.58
77.16
76.74
68.45
51.51
43.86
41.88
25.80
25.72
24.46
22.76
22.27
14.01
13.90
Me
N
N
N
O
3s
13C NMR, 75 MHz CDCl3
4000
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

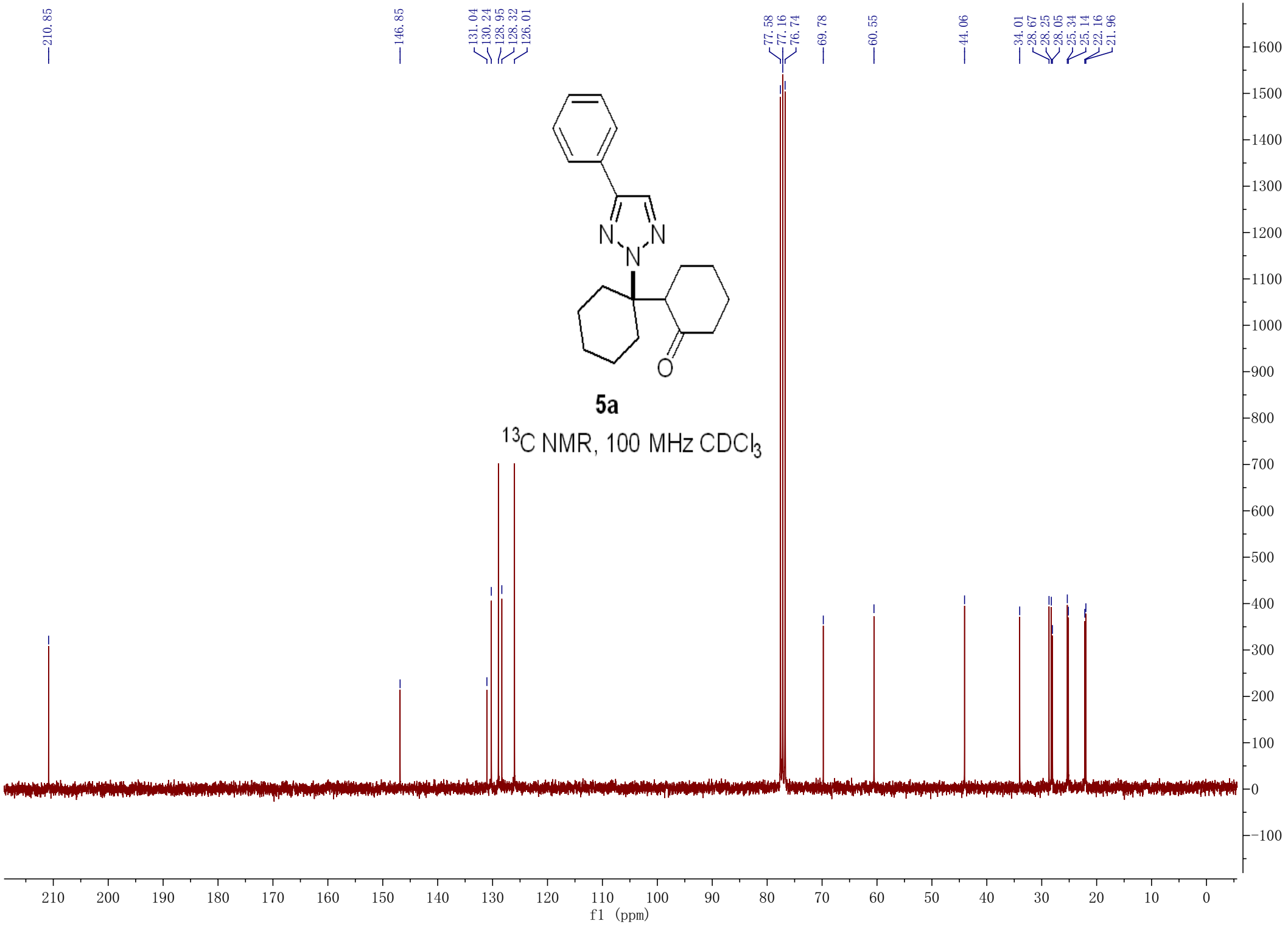

Me
N
N
N
O
3s'
1H NMR, 300 MHz CDCl3
f1 (ppm)

3sjtao

3sjtao 8660 t-275-b 13c cdcl3




7.87
7.83
7.81
7.45
7.43
7.41
7.36
7.34
7.32
7.26
2.94
2.93
2.91
2.90
2.38
2.37
2.34
2.33
2.31
1.69
1.65
1.62
1.60
1.56
1.45
1.42
1.40
1.39
1.38
1.36
1.18
1.15
1.00
0.96
0.93
0.90
N
N
N
O
5a
1H NMR, 400 MHz CDCl3
0.95
1.95
2.00
1.00
1.00
2.03
4.26
1.16
7.74
2.11
1.08
1.06
f1 (ppm)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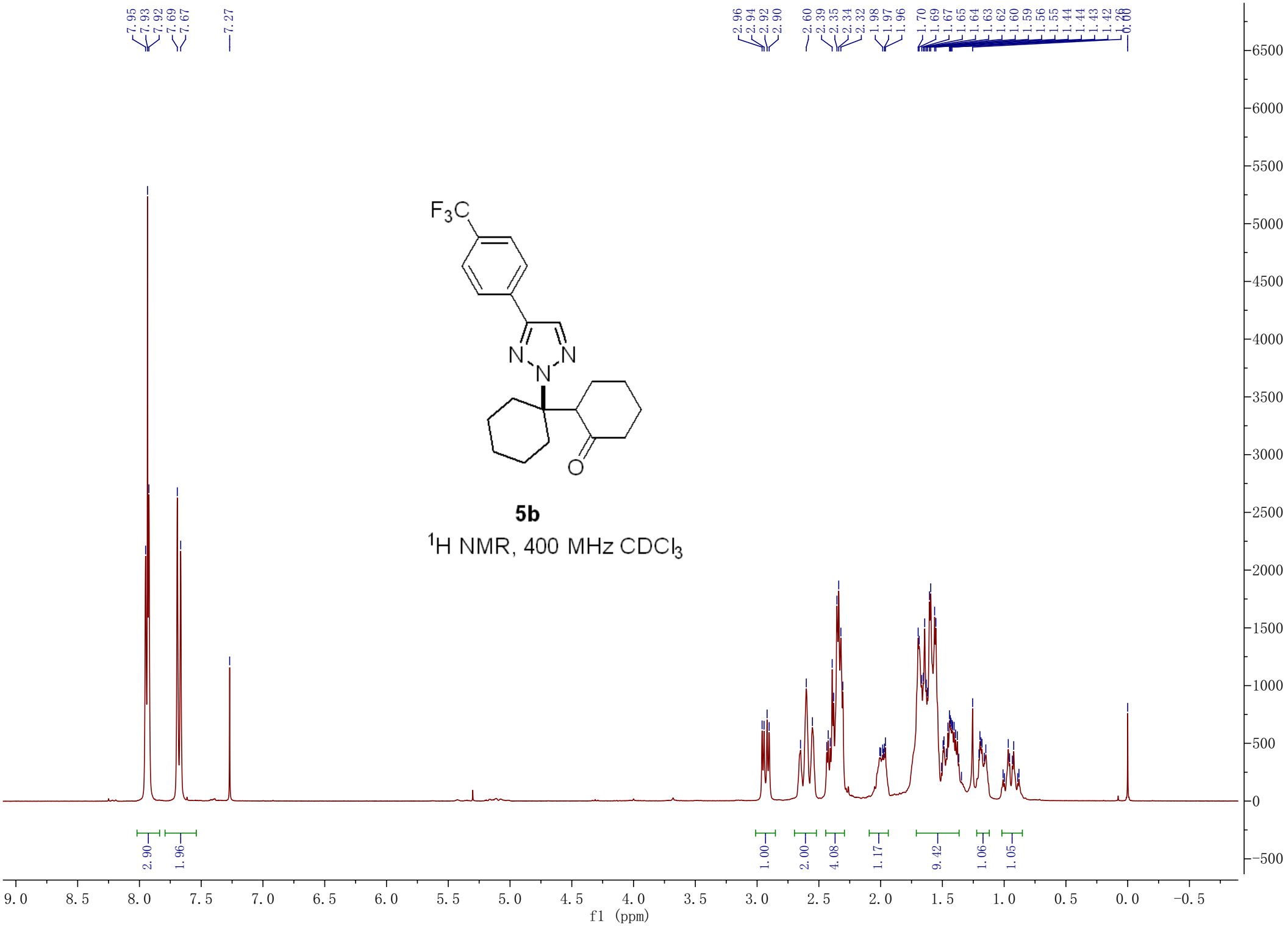
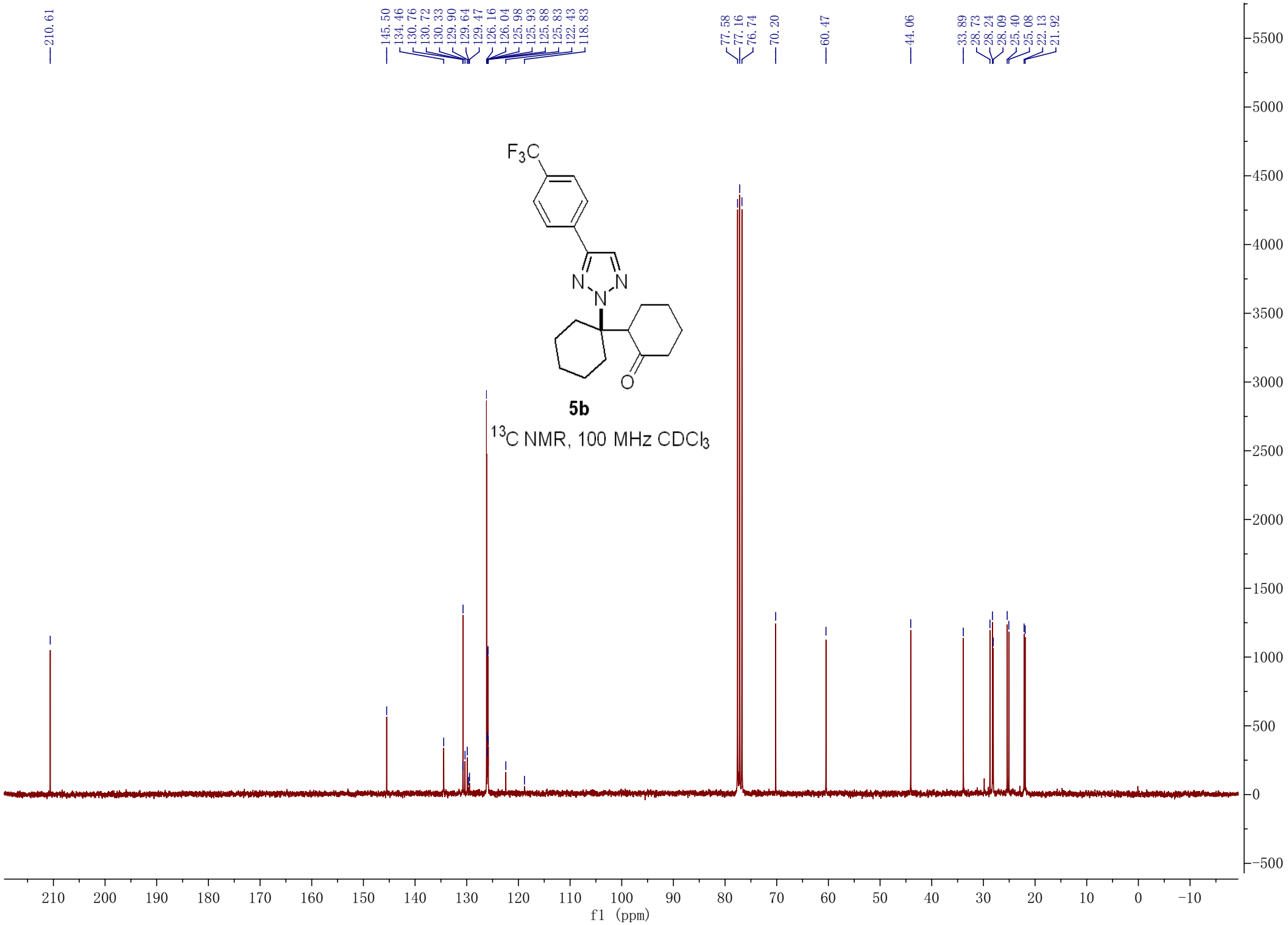
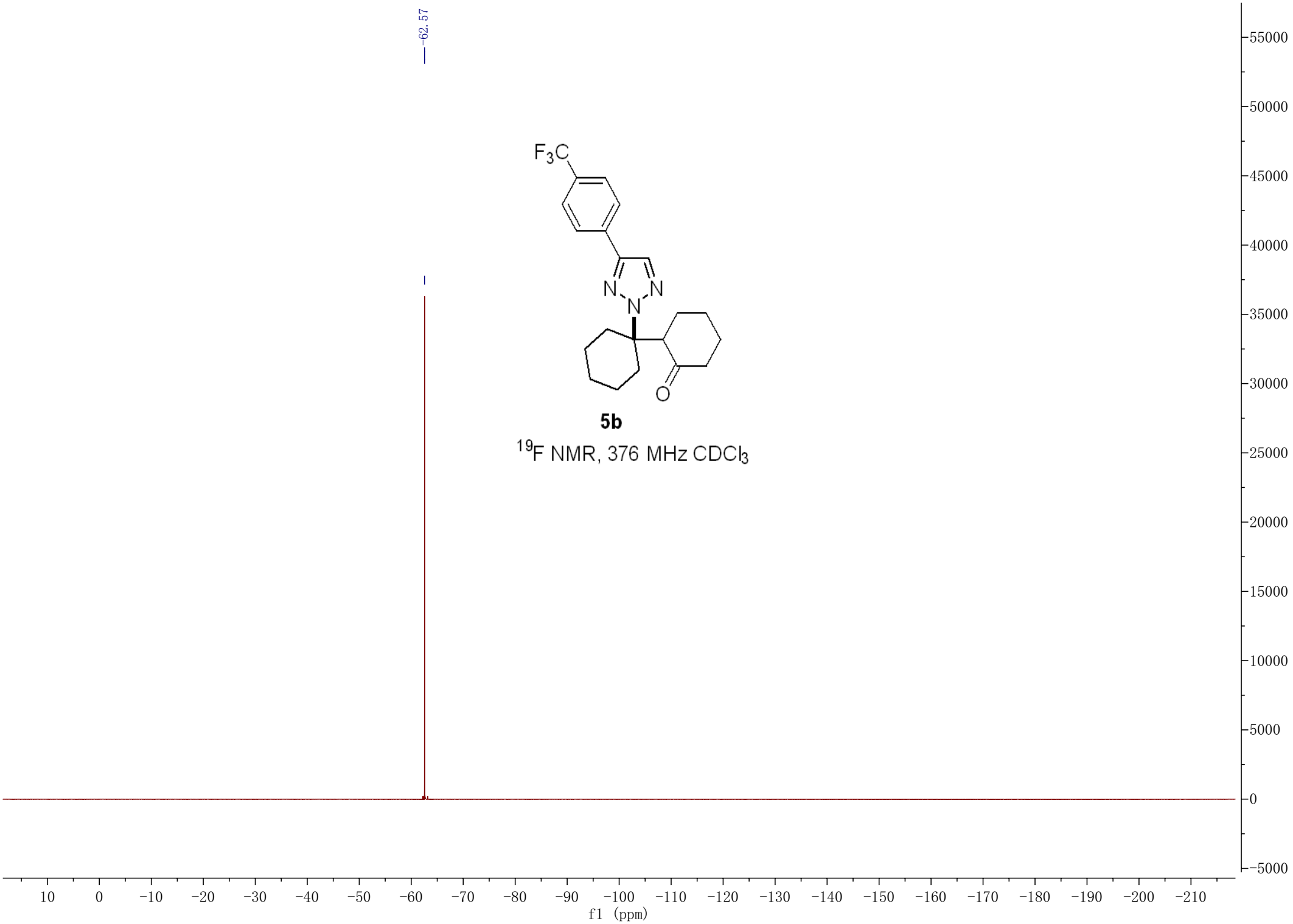
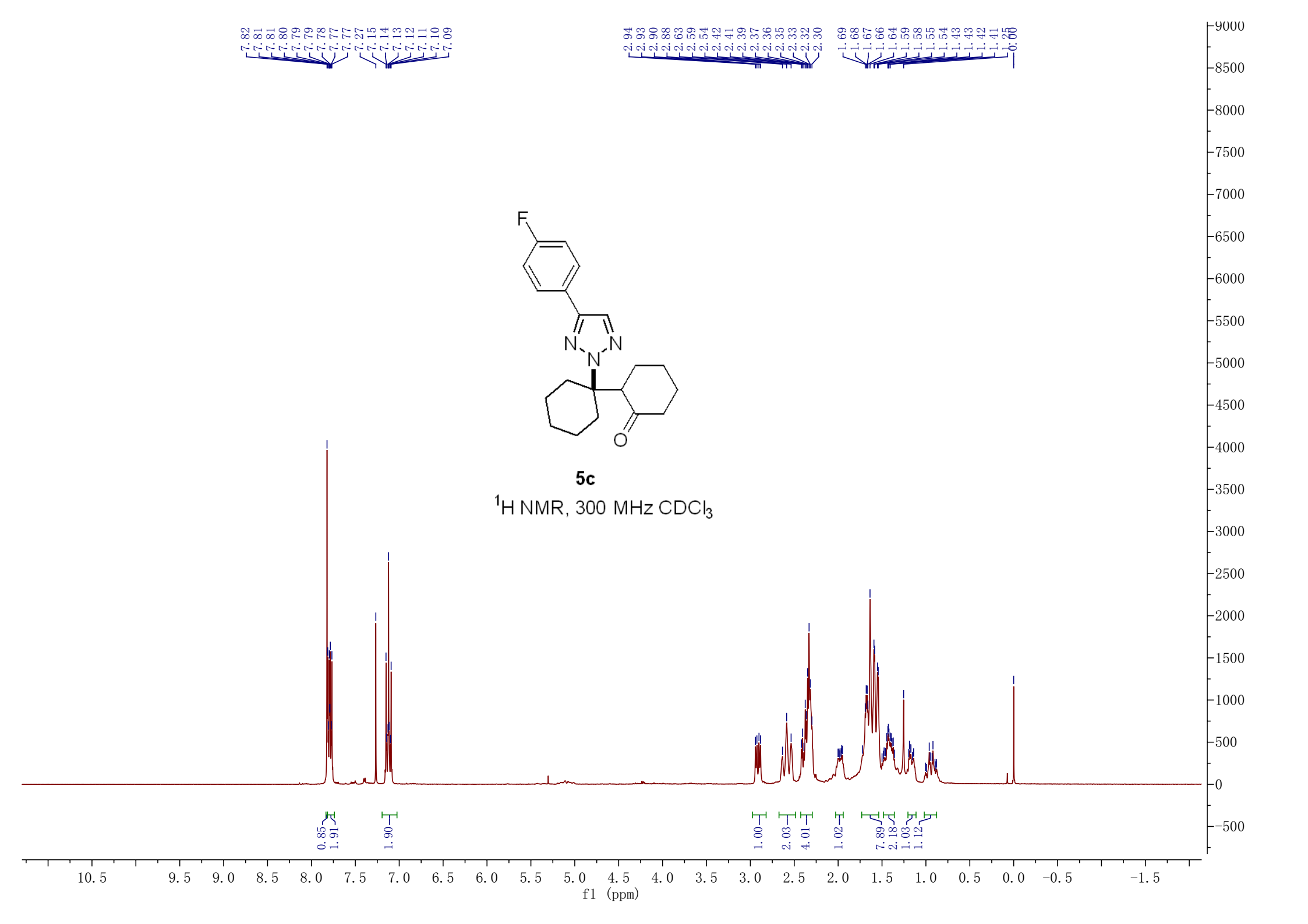
146.85
131.04
130.24
128.95
128.32
126.01
77.58
77.16
76.74
69.78
60.55
44.06
34.01
28.67
28.25
28.05
25.34
25.14
22.16
21.96
N
N
N
O
5a
13C NMR, 100 MHz CDCl3
f1 (ppm)


7.95
7.93
7.92
7.69
7.67
7.27
2.96
2.94
2.92
2.90
2.60
2.39
2.35
2.34
2.32
1.98
1.97
1.96
1.70
1.69
1.67
1.65
1.64
1.63
1.62
1.60
1.59
1.56
1.55
1.44
1.44
1.43
1.42
1.26
0.00
F3C
N
N
N
O
5b
1H NMR, 400 MHz CDCl3
2.90
1.96
1.00
2.00
4.08
1.17
9.42
1.06
1.05
f1 (ppm)


210.61
145.50
134.46
130.76
130.72
130.33
129.90
129.64
129.47
126.16
126.04
125.98
125.93
125.88
125.83
122.43
118.83
77.58
77.16
76.74
70.20
60.47
44.06
33.89
28.73
28.24
28.09
25.40
25.08
22.13
21.92
F3C
N
N
N
O
5b
13C NMR, 100 MHz CDCl3
f1 (ppm)


-62.57
F3C
N
N
N
O
5b
19F NMR, 376 MHz CDCl3
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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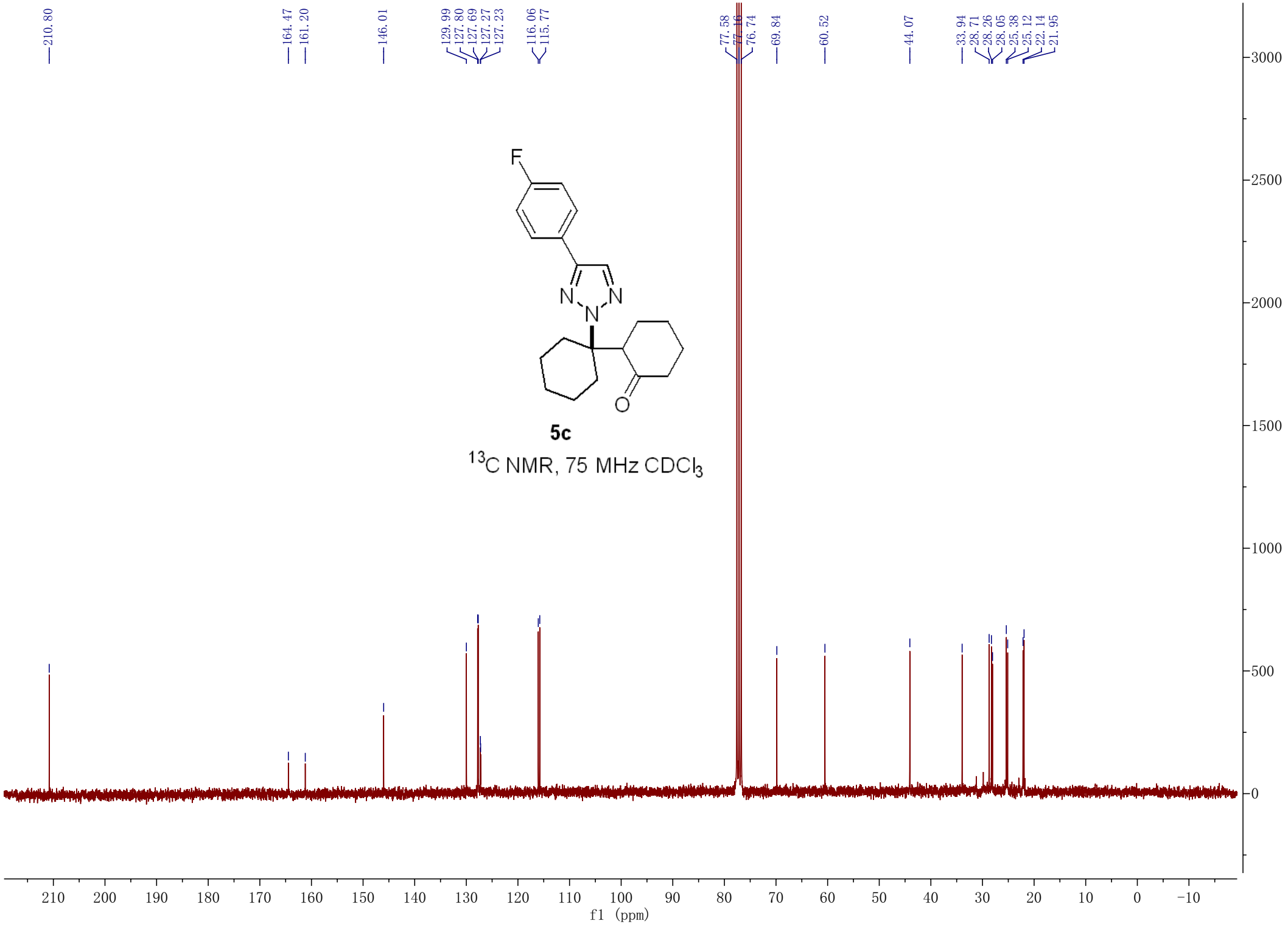
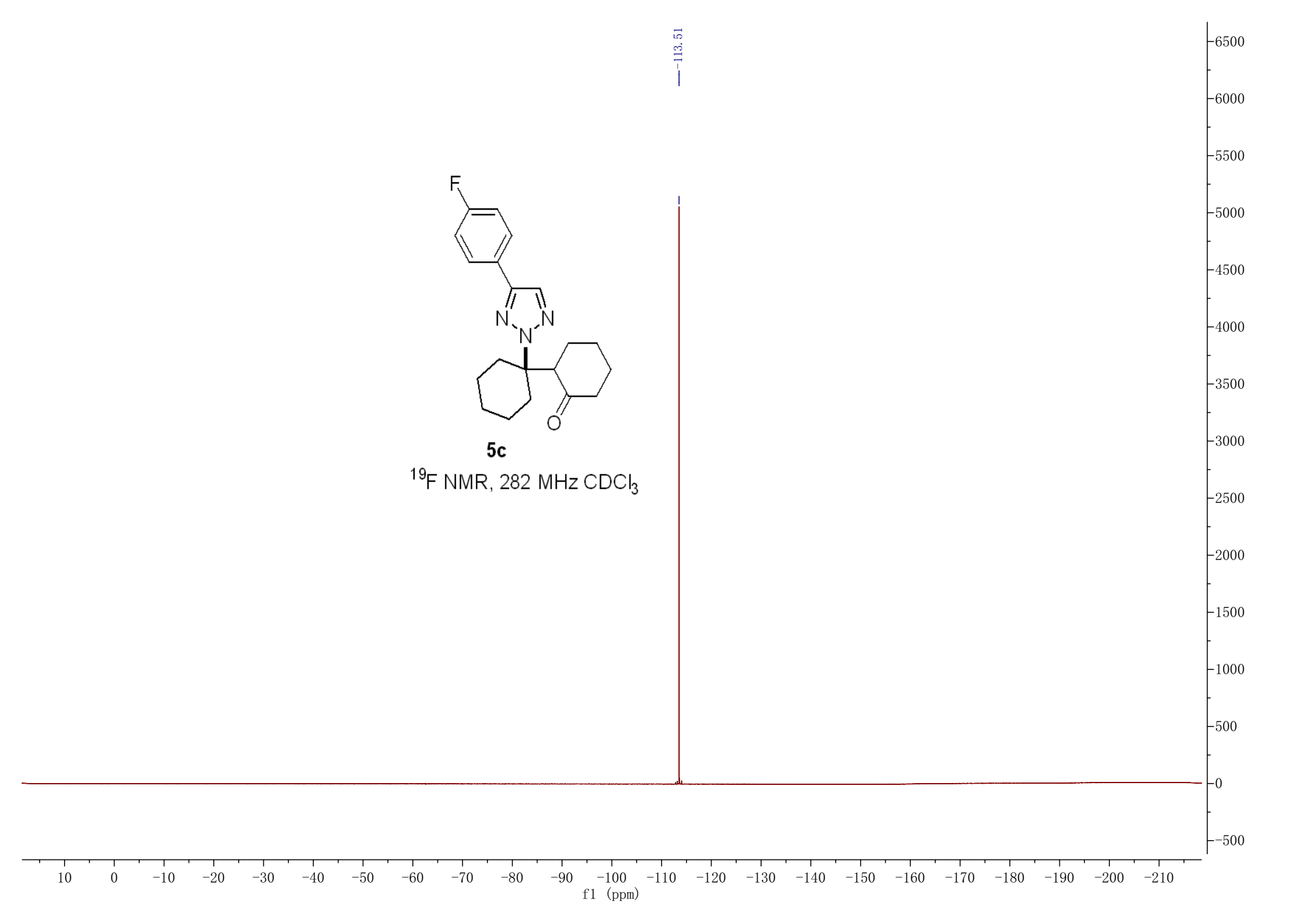
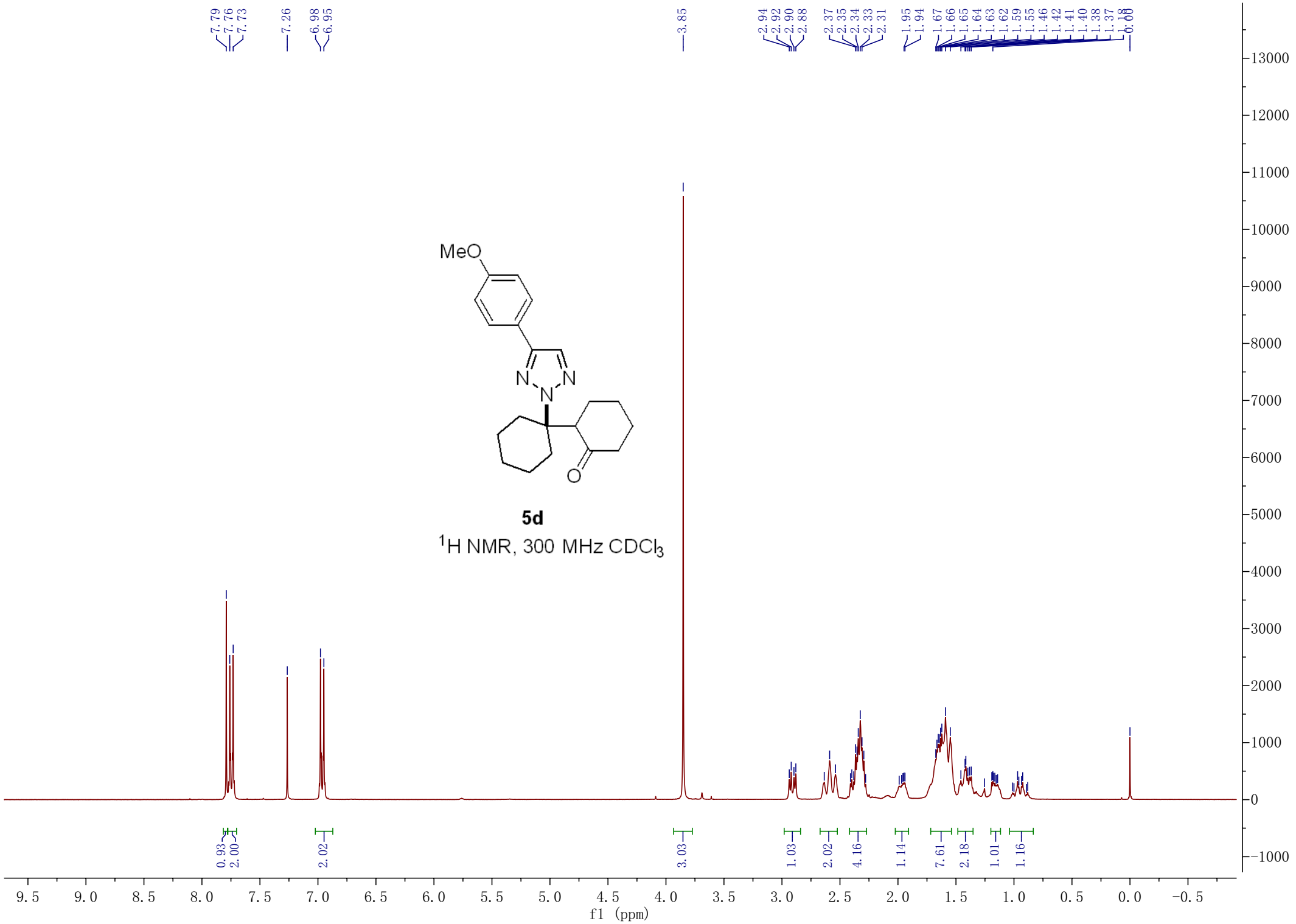
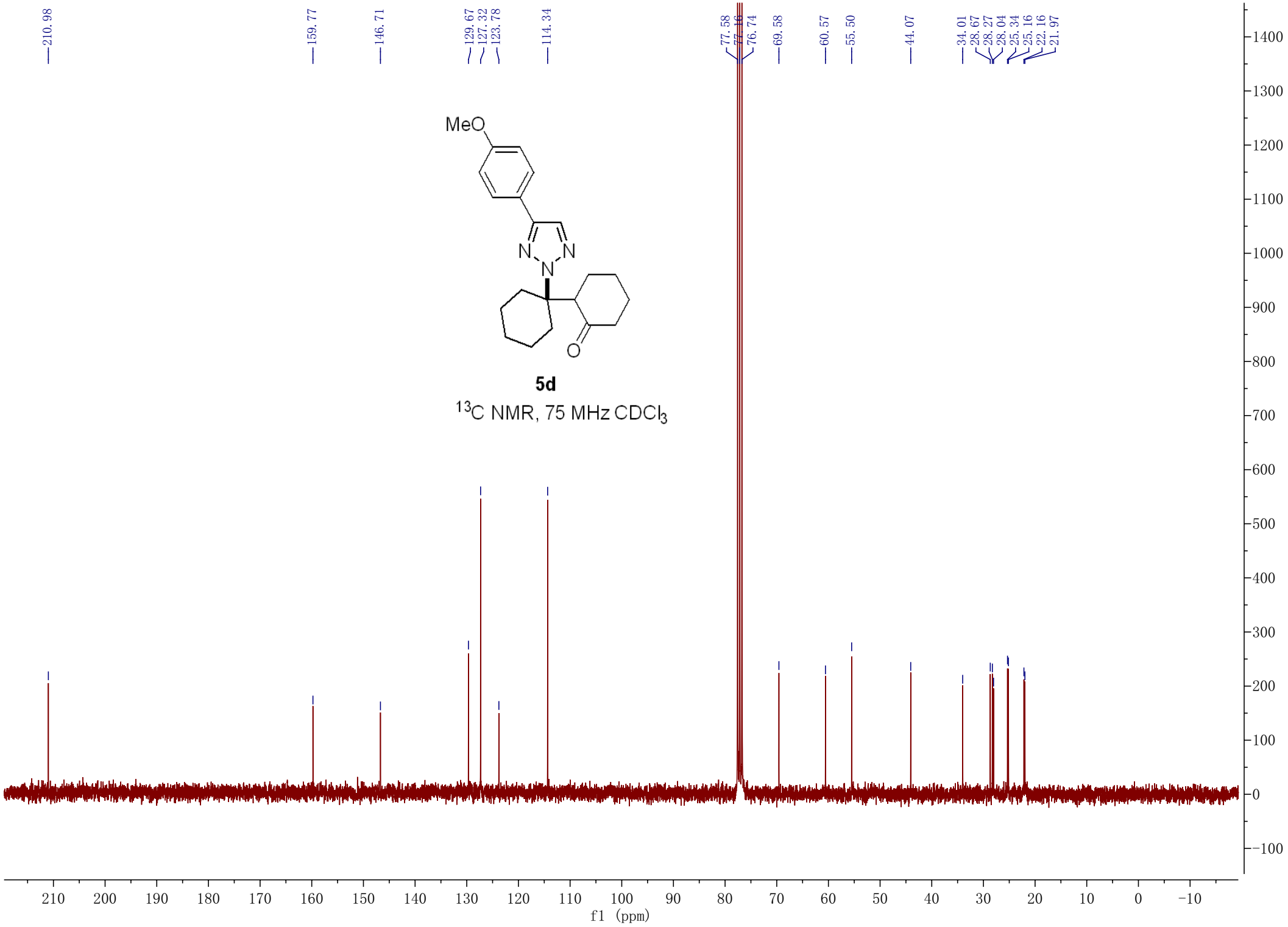
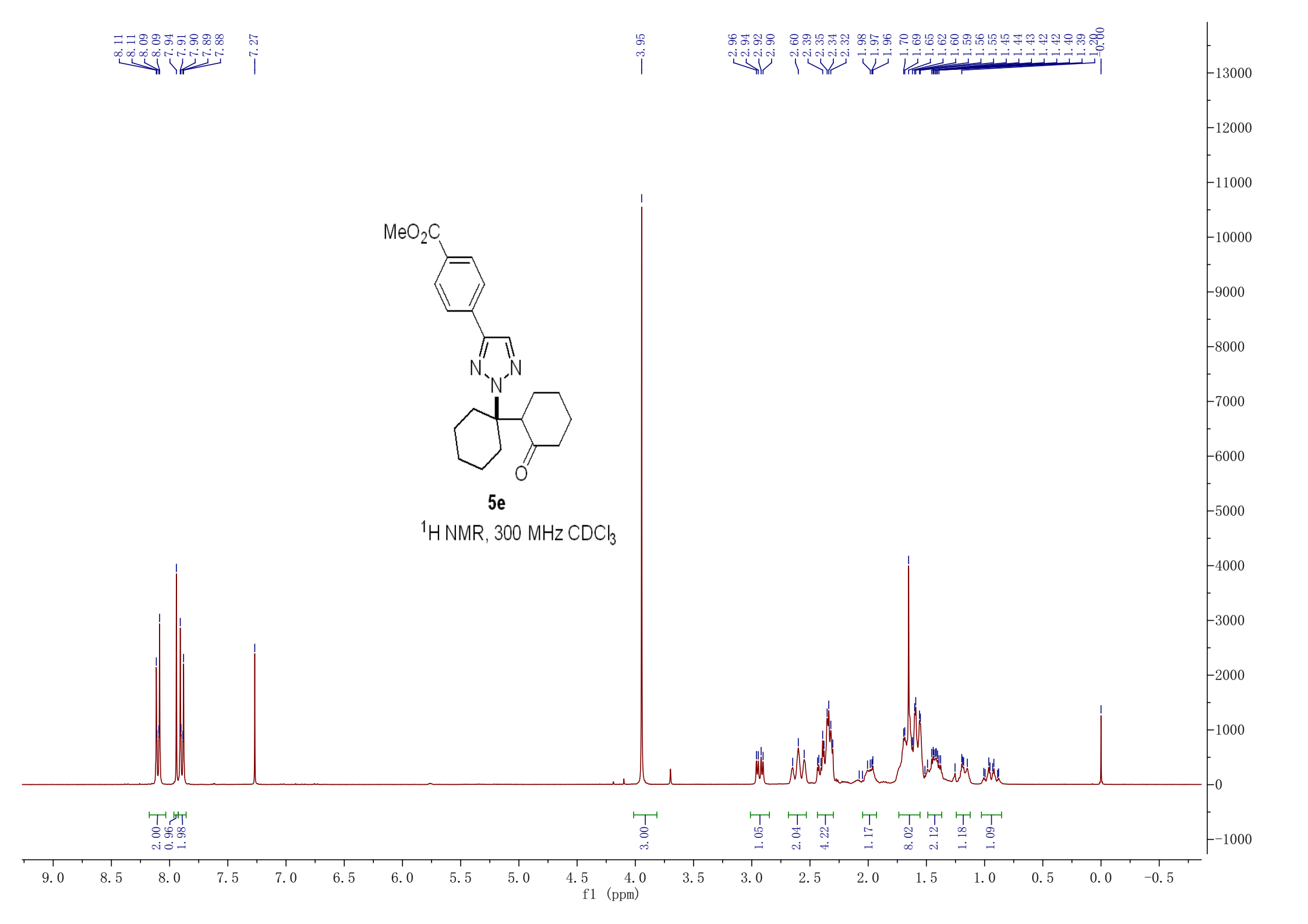
7.81
7.81
7.80
7.79
7.79
7.78
7.77
7.77
7.27
7.15
7.14
7.13
7.12
7.11
7.10
7.09
2.94
2.93
2.90
2.88
2.63
2.59
2.54
2.42
2.41
2.39
2.37
2.36
2.35
2.33
2.32
2.30
1.69
1.68
1.67
1.66
1.64
1.59
1.58
1.55
1.54
1.43
1.43
1.42
1.41
F
N
N
N
O
5c
1H NMR, 300 MHz CDCl3
0.85
1.91
1.90
1.00
2.03
4.01
1.02
7.89
2.18
1.03
1.12
f1 (ppm)

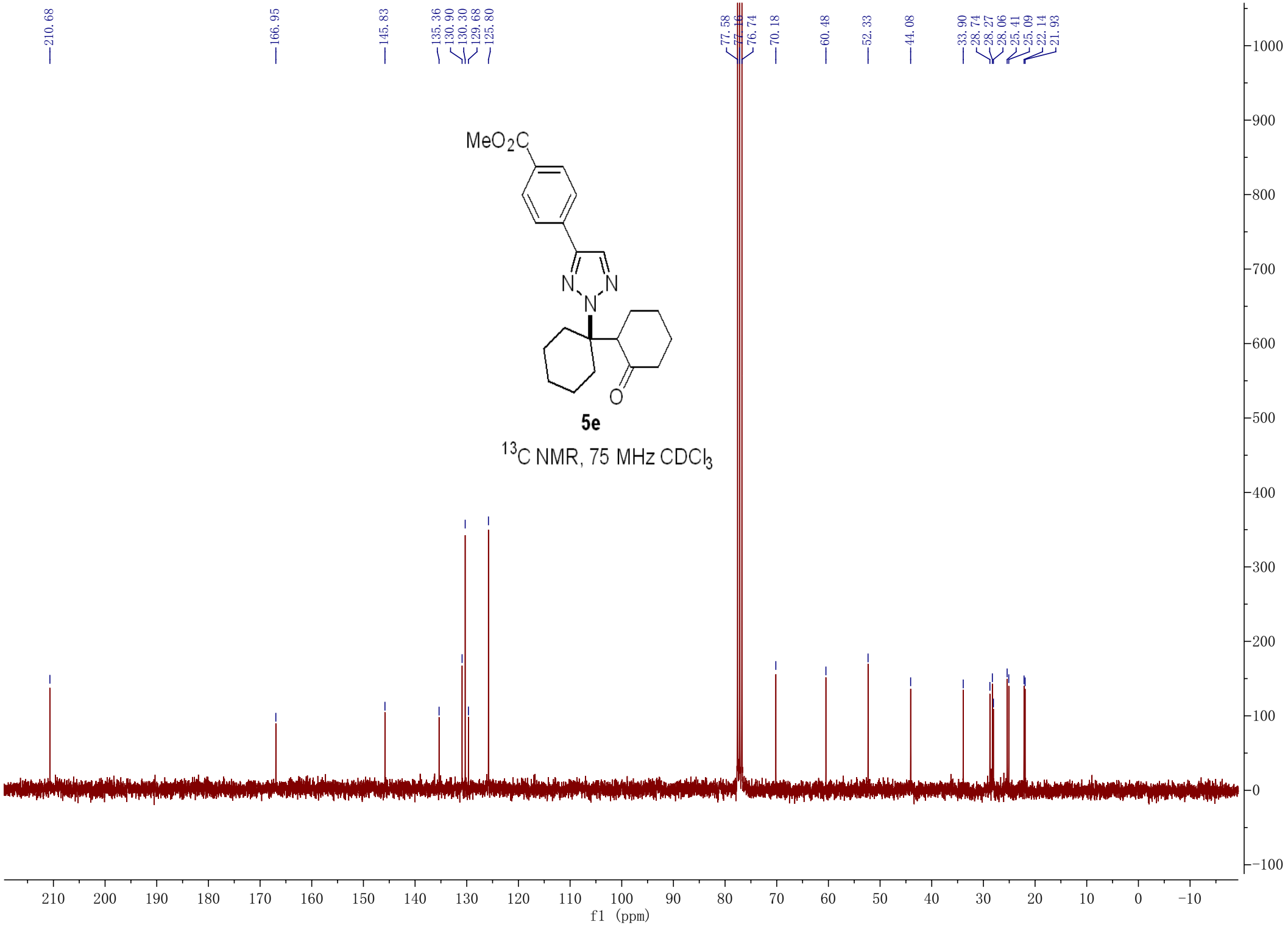

210.80
164.47
161.20
146.01
129.99
127.80
127.69
127.27
127.23
116.06
115.77
77.58
77.16
76.74
69.84
60.52
44.07
33.94
28.71
28.26
28.05
25.38
25.12
22.14
21.95
F
N
N
N
O
5c
13C NMR, 75 MHz CDCl3
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


-113.51
F
N
N
N
O
5c
19F NMR, 282 MHz CDCl3
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


MeO
N
N
N
O
5d
1H NMR, 300 MHz CDCl3
f1 (ppm)


210.98
159.77
146.71
129.67
127.32
123.78
114.34
77.58
77.16
76.74
69.58
60.57
55.50
44.07
34.01
28.67
28.27
28.04
25.34
25.16
22.16
21.97
MeO
N
N
N
O
5d
13C NMR, 75 MHz CDCl3
f1 (ppm)


8.11
8.11
8.09
8.09
7.94
7.91
7.90
7.89
7.88
7.27
3.95
2.96
2.94
2.92
2.90
2.60
2.39
2.35
2.34
2.32
1.98
1.97
1.96
1.70
1.69
1.65
1.62
1.60
1.59
1.56
1.55
1.45
1.44
1.43
1.42
1.42
1.40
1.39
1.20
0.00
MeO2C
N
N
N
O
5e
1H NMR, 300 MHz CDCl3
2.00
0.96
1.98
3.00
1.05
2.04
4.22
1.17
8.02
2.12
1.18
1.09
f1 (ppm)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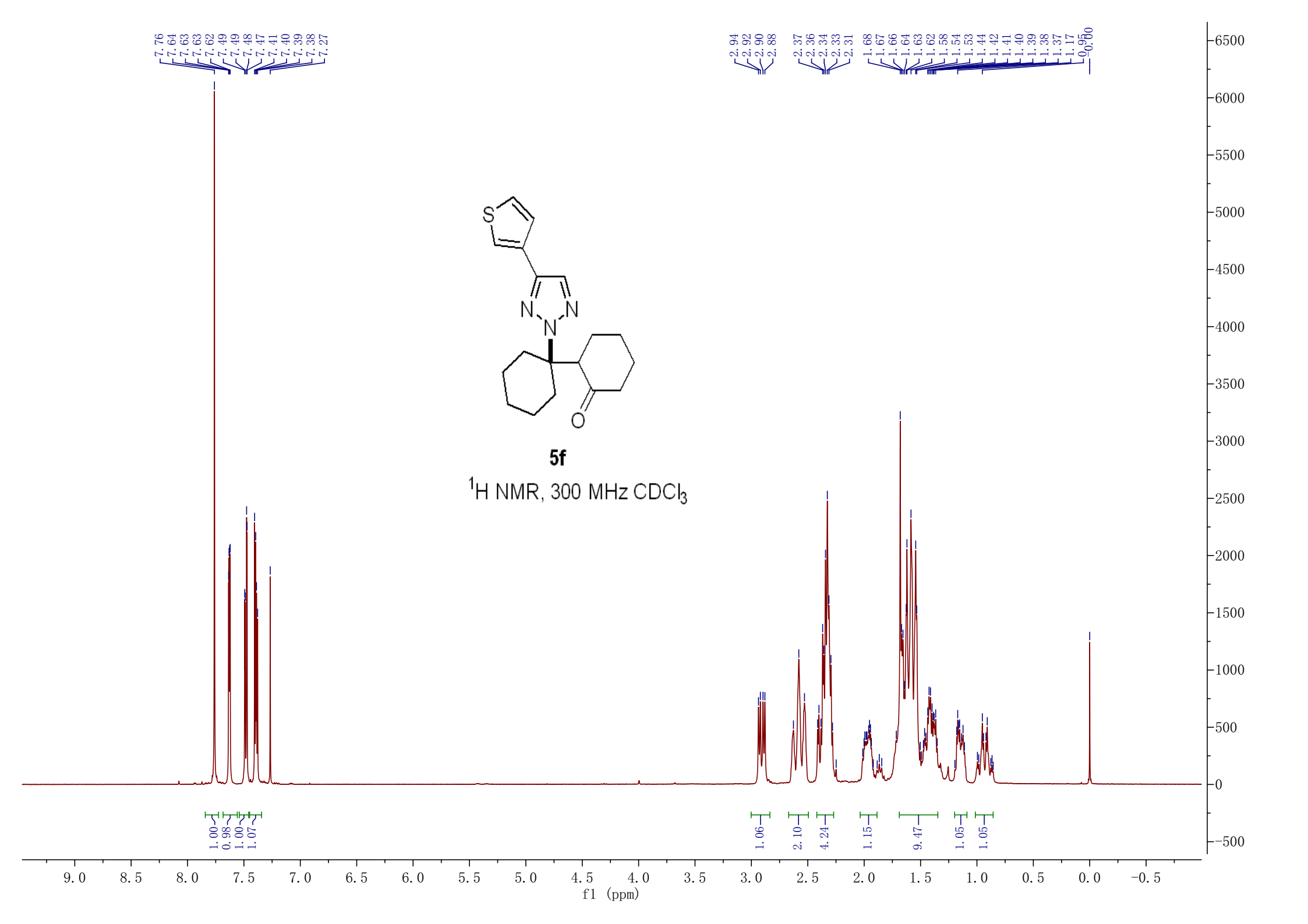
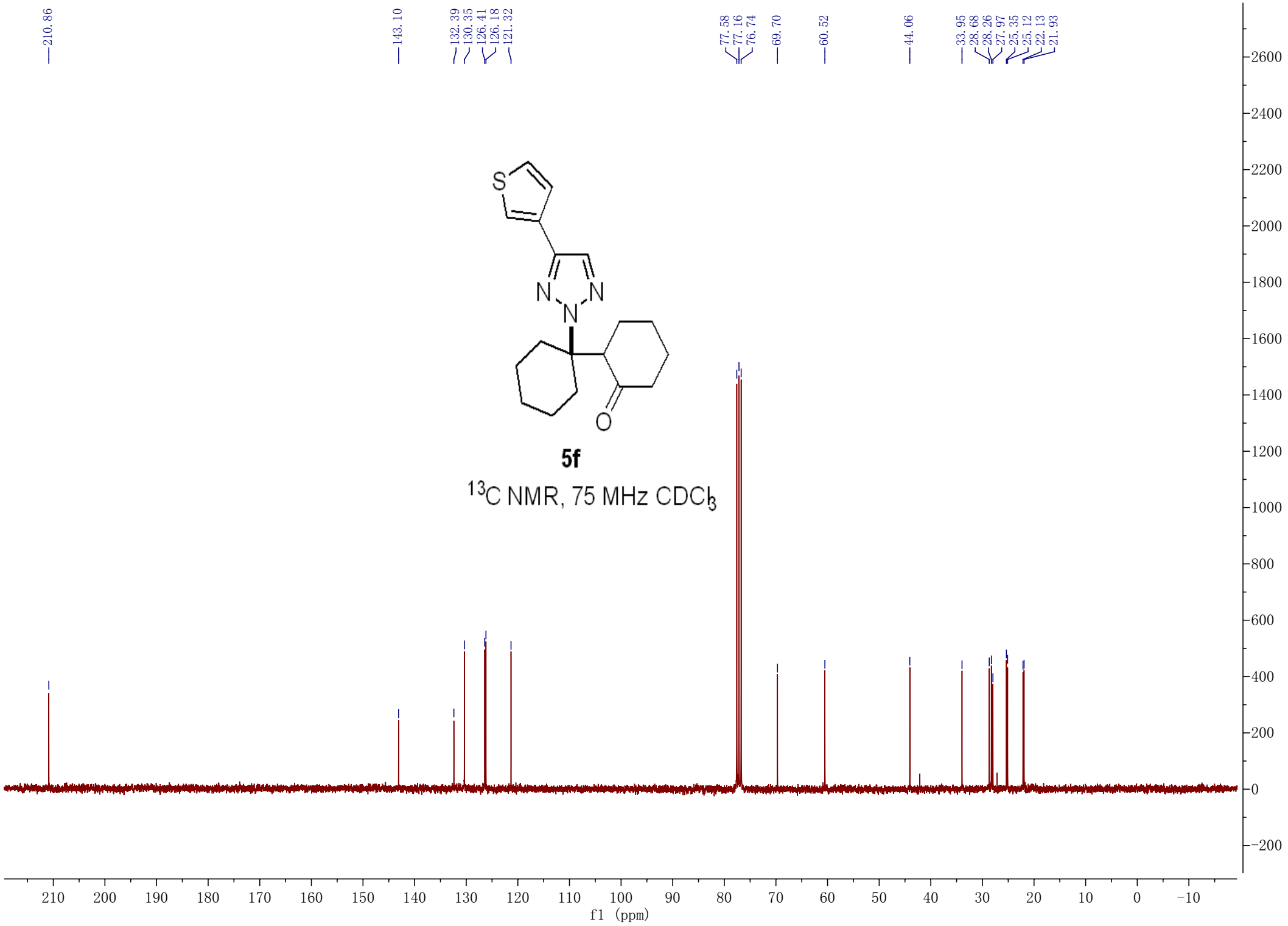
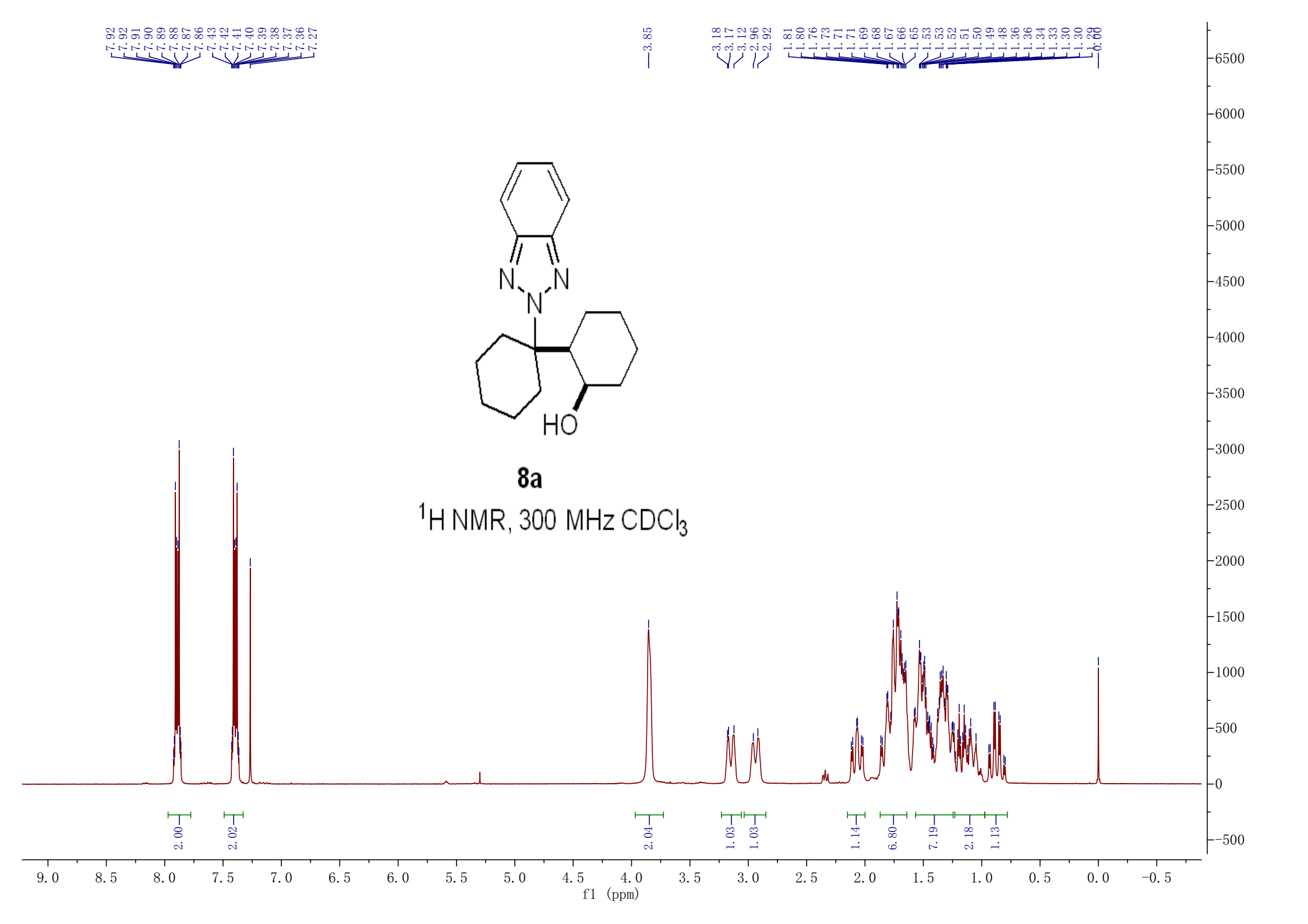
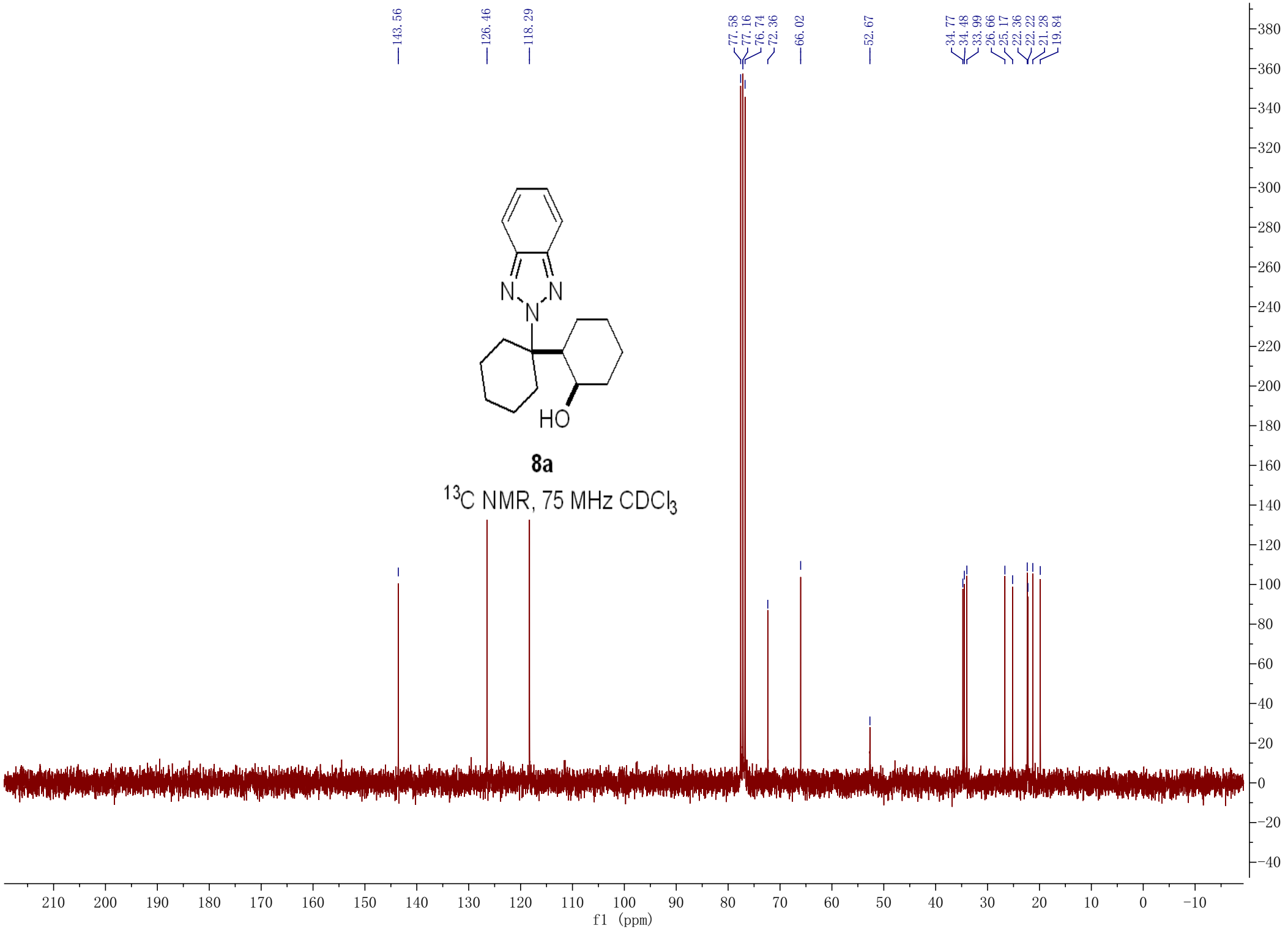
166.95
145.83
135.36
130.90
130.30
129.68
125.80
77.58
77.16
76.74
70.18
60.48
52.33
44.08
33.90
28.74
28.27
28.06
25.41
25.09
22.14
21.93
MeO2C
N
N
N
O
5e
13C NMR, 75 MHz CDCl3
f1 (ppm)


7.76
7.64
7.63
7.63
7.62
7.49
7.49
7.48
7.47
7.41
7.40
7.39
7.38
7.27
2.94
2.92
2.90
2.88
2.37
2.36
2.34
2.33
2.31
1.68
1.67
1.66
1.64
1.63
1.62
1.58
1.54
1.53
1.44
1.42
1.41
1.40
1.39
1.38
1.37
1.17
0.95
0.80
S
N
N
N
O
5f
1H NMR, 300 MHz CDCl3
1.00
0.98
1.00
1.07
1.06
2.10
4.24
1.15
9.47
1.05
1.05
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
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)


210.86
143.10
132.39
130.35
126.41
126.18
121.32
77.58
77.16
76.74
69.70
60.52
44.06
33.95
28.68
28.26
27.97
25.35
25.12
22.13
21.93
S
N
N
N
O
5f
13C NMR, 75 MHz CDCl3
f1 (ppm)


7.92
7.92
7.91
7.90
7.89
7.88
7.87
7.86
7.43
7.42
7.41
7.40
7.39
7.38
7.37
7.36
7.27
3.85
3.18
3.17
3.12
2.96
2.92
1.81
1.80
1.76
1.73
1.71
1.71
1.69
1.68
1.67
1.66
1.65
1.53
1.53
1.52
1.51
1.50
1.49
1.48
1.36
1.34
1.33
1.30
1.29
0.00
N
N
N
HO
8a
1H NMR, 300 MHz CDCl3
2.00
2.02
2.04
1.03
1.03
1.14
6.80
7.19
2.18
1.13
f1 (ppm)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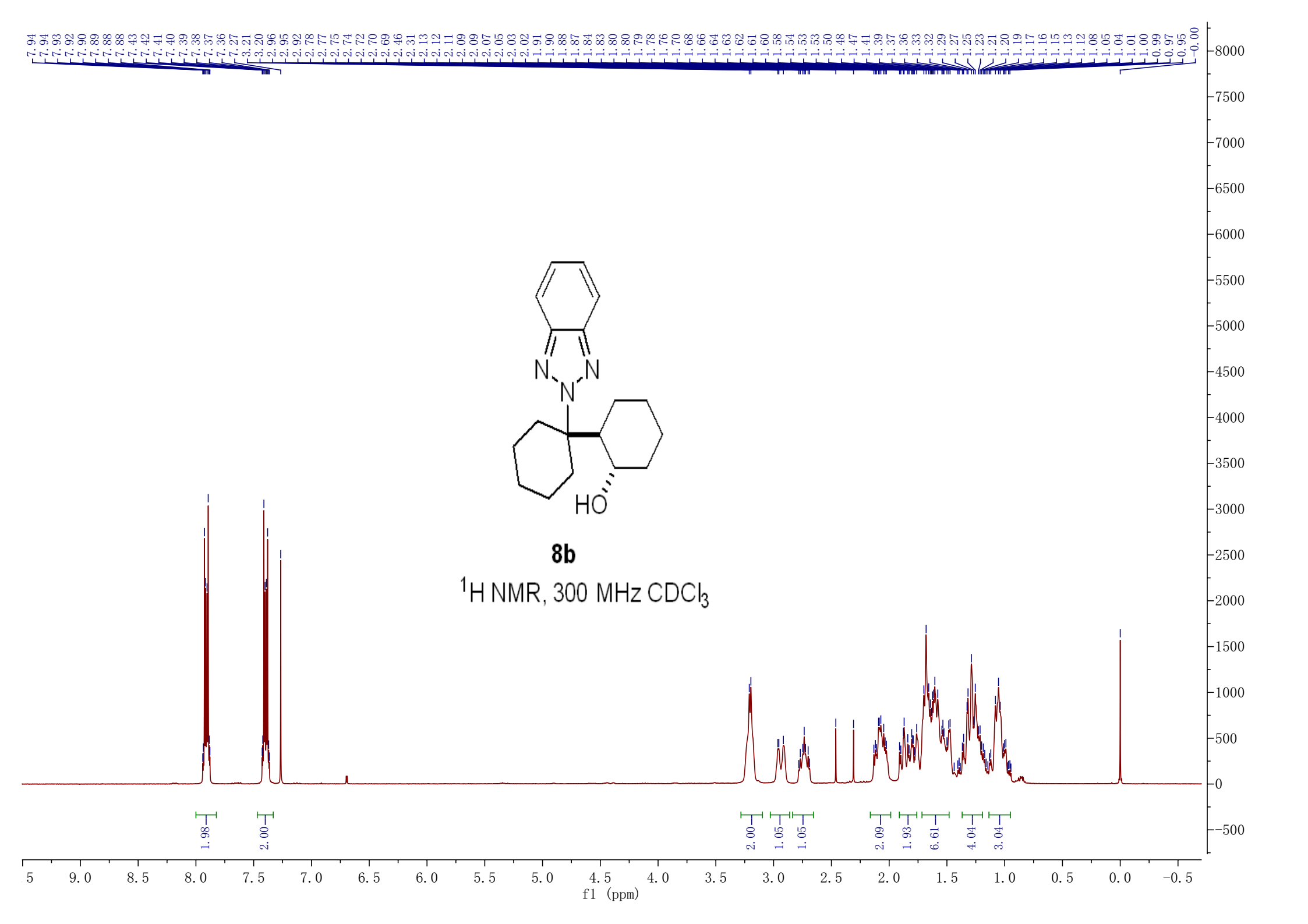
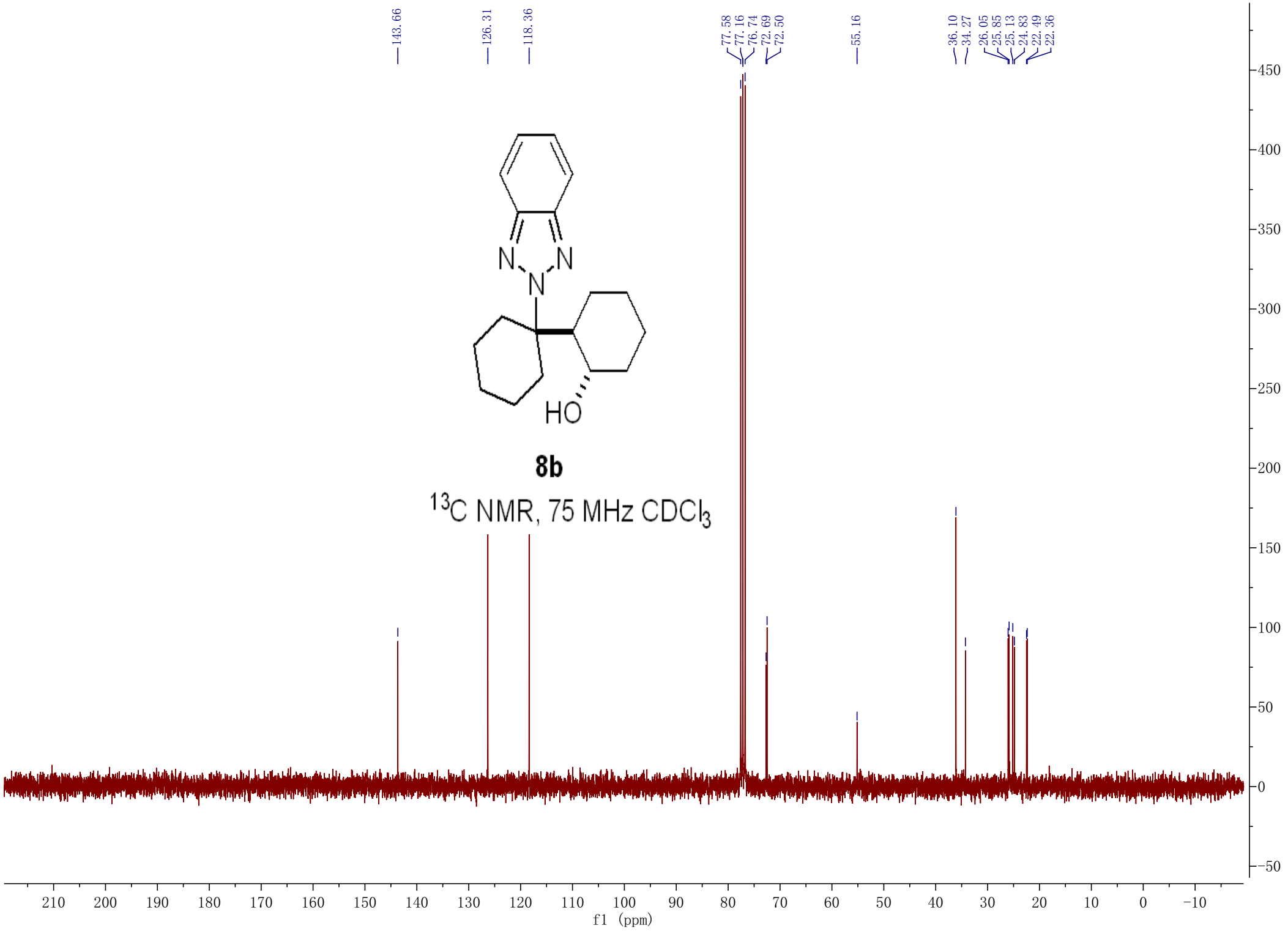
126.46
118.29
77.58
77.16
76.74
72.36
66.02
52.67
34.77
34.48
33.99
26.66
25.17
22.36
22.22
21.28
19.84
N
N
N
HO
8a
13C NMR, 75 MHz CDCl3
f1 (ppm)


HO
8b
1H NMR, 300 MHz CDCl3
f1 (ppm)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126.31
118.36
77.58
77.16
76.74
72.69
72.50
55.16
36.10
34.27
26.05
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22.49
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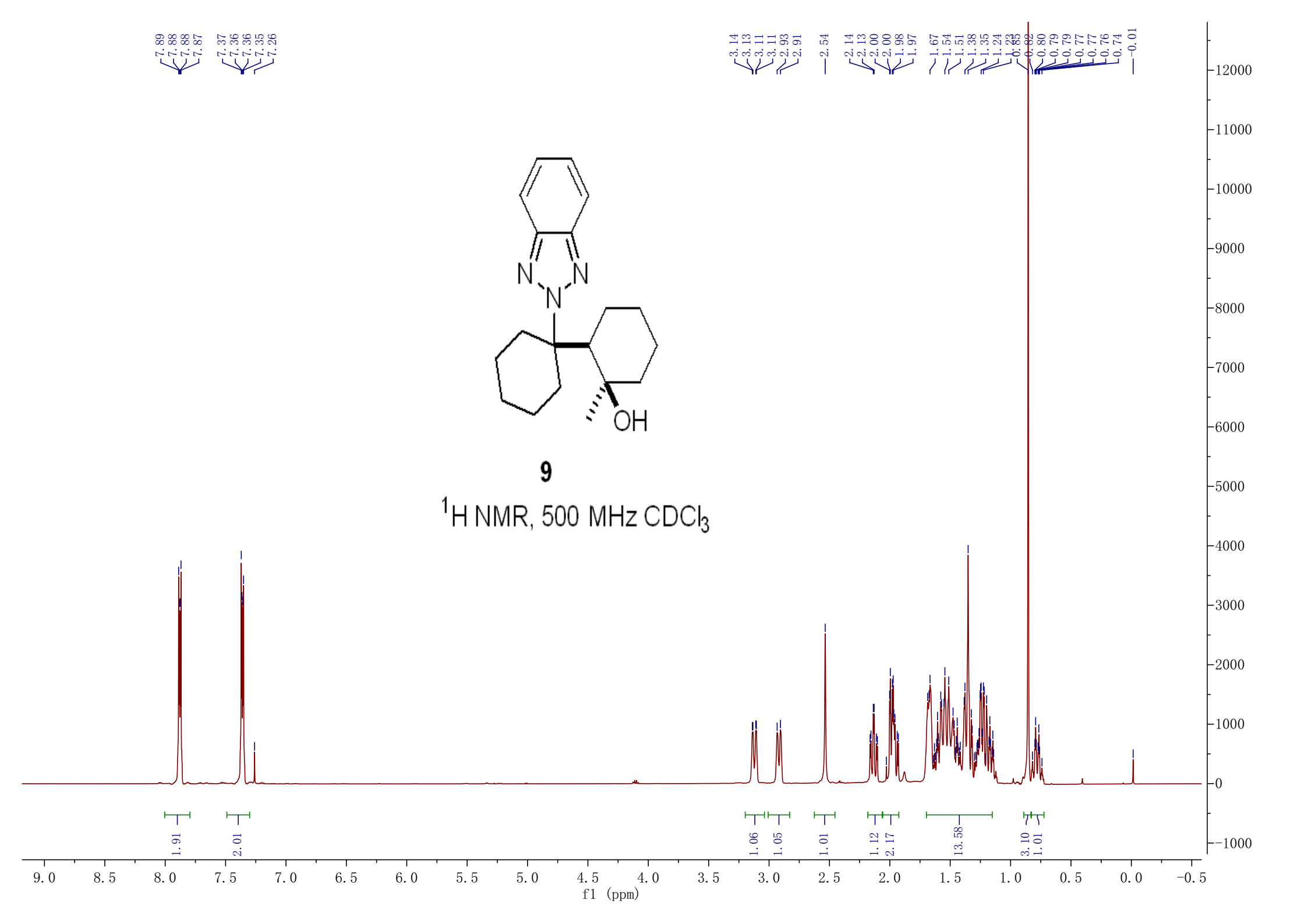
N
HO
8b
13C NMR, 75 MHz CDCl3
f1 (ppm)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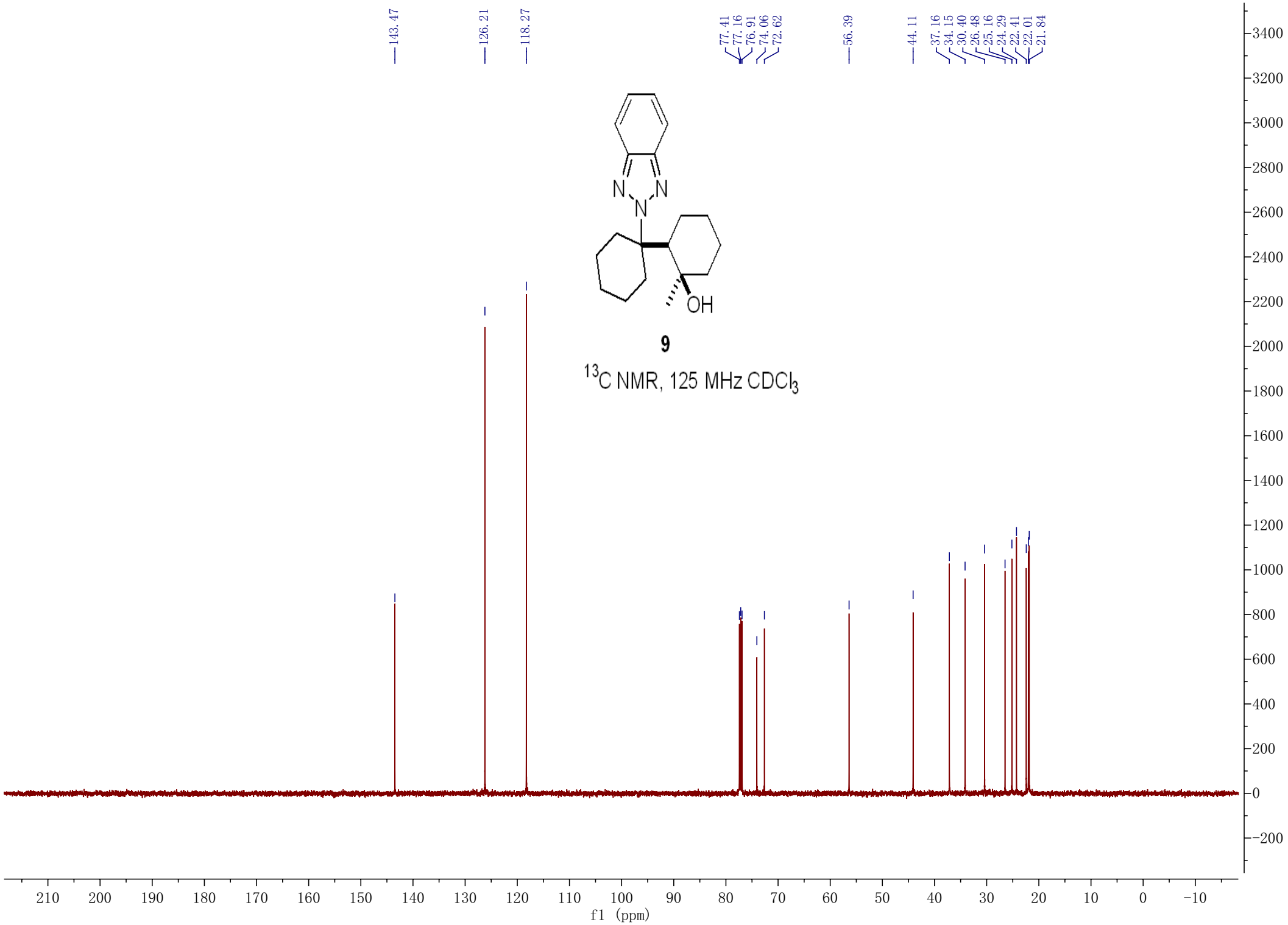
7.88
7.88
7.87
7.37
7.36
7.36
7.35
7.26
3.14
3.13
3.11
3.11
2.93
2.91
2.54
2.14
2.13
2.00
2.00
1.98
1.97
1.67
1.54
1.51
1.38
1.35
1.24
0.82
0.80
0.79
0.79
0.77
0.77
0.76
0.74
-0.01
N
N
N
OH
9
1H NMR, 500 MHz CDCl3
1.91
2.01
1.06
1.05
1.01
1.12
2.17
13.58
3.10
1.01
f1 (ppm)


143.47
126.21
118.27
77.41
77.16
76.91
74.06
72.62
56.39
44.11
37.16
34.15
30.40
26.48
25.16
24.29
22.41
22.01
21.84
N
N
N
OH
9
13C NMR, 125 MHz CDCl3
f1 (ppm)