Supporting Information

A practical and economic route for regioselective cyclization of β-phenoxyl ynones to flavonoid derivatives

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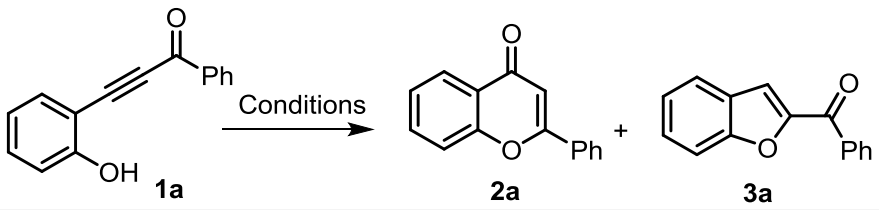
1. General Information

Anhydrous tetrahydrofuran (THF) and toluene (PhMe) were heated over sodium under N_2 for at least four hours before distilled to use. Anhydrous DCM was heated over calcium hydride for two hours before distilled to use. The reactions that sensitive to air or moisture were conducted under nitrogen atmosphere in dry solvents. All glassware and stir bars were washed with aqua regia prior to use. Purification of products was performed by forced-flow chromatograph (silca gel, 200-300 mesh). All reactions were monitored by using thin layer chromatograph (TLC, silica gel GF 254), and were first observed by ultraviolet (254 and 365 nm), then were stained with phosphomolybdic acid solutions (10%) or aqueous potassium permanganate, subsequently toasted by a heat gun. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Advance spectrometer at 500 MHz and 125 MHz or 400 MHz and 100 MHz, respectively. Chemical shift values are reported in δ (ppm) relative to $CDCl_3$ (^{1}H NMR, δ = 7.26; ^{13}C NMR, δ = 77.16), CD_3COCD_3 (^{1}H NMR, δ = 2.05; ^{13}C NMR, δ = 29.84, δ = 206.26), MeOH (1H NMR, δ = 3.31; 13C NMR, δ = 49.00), or DMSO-d_6 (^{1}H NMR, δ = 2.50; ^{13}C NMR, δ = 39.52). Signal shapes are shown as s (singlet), d (doublet), t (triplet), q (quartet), p (quintet), dd (doublet of doublets), td (triplet double), dt (doublet of triplets), m (multiplet), hept (heptet). High resolution mass spectra (HRMS) were performed on Thermo Scientific Q Exactive or waters G2-Xs qtof by using an electrospray ionization (ESI) ionization source analyzed. Among them, **1b-1ad**, **2b, 2d-2e, 2g-2l, 2o-2p, 2r, 2u, 2x-2ad, 2a', 3a, 4-10** were analyzed by Thermo Scientific LTQ Orbitrap XL. **2a, 2c, 2f, 2m-2n, 2q, 2s-2t, 2v-2w** were analyzed by Thermo Scientific Q Exactive.

2. Screening conditions

Table 1 Optimization of Reaction Conditions[a]



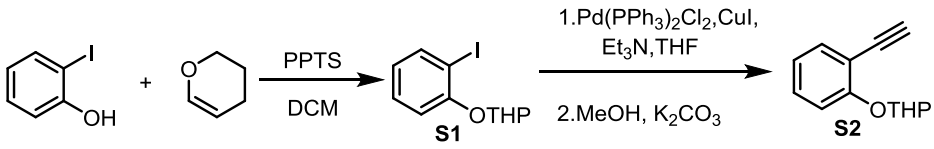
1a **2a** **3a**

entry	solvent	temp.(°C)	conc. [M]	time(h)	2a(%)[b]	3a(%)[b]
1	MeOH	reflux	0.5	12	50	10
2	EtOH	reflux	0.5	12	79	8
3	^{n}BuOH	100	0.5	12	–	–
4	^{i}PrOH	100	0.5	12	–	–
5[c]	HFIP	100	0.5	12	81	–
6[d]	EG	100	0.5	12	80	6
7[e]	1,3-PDO	100	0.5	12	86	5
8[f]	1,4-BDO	100	0.5	12	82	6
9	1,3-PDO	120	0.5	5	90	4
10[g]	dried 1,3-PDO	120	0.5	11	83	9
11[h]	***wet 1,3-PDO***	***120***	0.5	***3***	***95***	–
12	wet 1,3-PDO	120	0.3	3	95	–
13	DCM	reflux	0.5	12	–	–
14	THF	reflux	0.5	12	–	–
15	PhMe	100	0.5	12	–	–
16[i]	PhMe	100	0.5	12	78	–
17	DMSO	100	0.5	12	75	–

[a] Reaction conditions: The reactions was conducted with everything in a reaction tube under an air atmosphere. [**1a**]: Molar concentrations of the **1a** in solvent. [b] Isolated yield from column chromatography. [c] HFIP: Hexafluoroisopropanol. [d] EG: Ethylene glycol. [e] 1,3-PDO: 1,3-Propanediol (0.005% moisture). [f] 1,4-BDO: 1,4-Butanediol. [g] Dried 1,3-PDO: dry the 1,3-Propanediol in advance with $MgSO_4$ and add 1 eq. of $MgSO_4$ in the reaction. [h] Wet 1,3-PDO: 99% pure 1,3-Propanediol with 1% moisture. [i] PhMe: 95% pure toluene with 5% wet 1,3-PDO.

3. Experimental Procedures and Characterization Data for Substrates

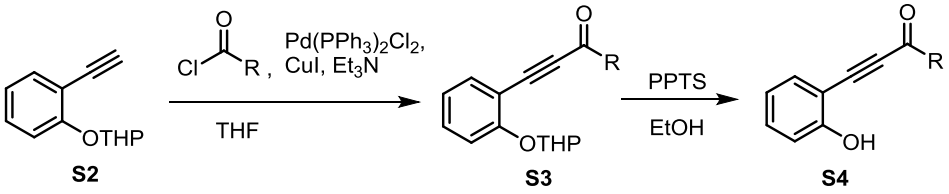
A. General procedures for compound S1, S2.



S1 were prepared according to the reported method[1].

To an anhydrous flask was added $Pd(PPh_3)_2Cl_2$ (70 mg, 0.1 mmol, 2 mol%) and CuI (38 mg, 0.2 mmol, 4 mol%), then the mixture was degassed and flushed with N_2 for three times at room temperature. THF (20 mL), Et_3N (2.02 g, 20 mmol, 4.0 equiv.), **S1** (1.52 g, 5 mmol, 1.0 equiv.) and trimethylsilylacetylene (541 mg, 5.5 mmol, 1.1 equiv.) were added successively and the mixture was allowed to stirred at room temperature for 1 hour. Upon completion, the mixture was quenched with saturated NH_4Cl solution (30 mL), extracted with EtOAc (3 × 10 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 20) to get the crude product (1.26 g, 4.6 mmol, 92%). The crude product bearing trimethylsilyl substituent was dissolved in MeOH (25 mL). Then K_2CO_3 (1.91 g, 13.8 mmol, 3.0 equiv.) was added and the resulting mixture was stirred at room temperature for 1 hour. Then, the mixture was filtered and concentrated to get the crude product which was purified by flash column chromatography to get the crude product **S2** (911 mg, 4.51 mmol, 98%, oily liquid).

B. General procedures for compound S3, S4.

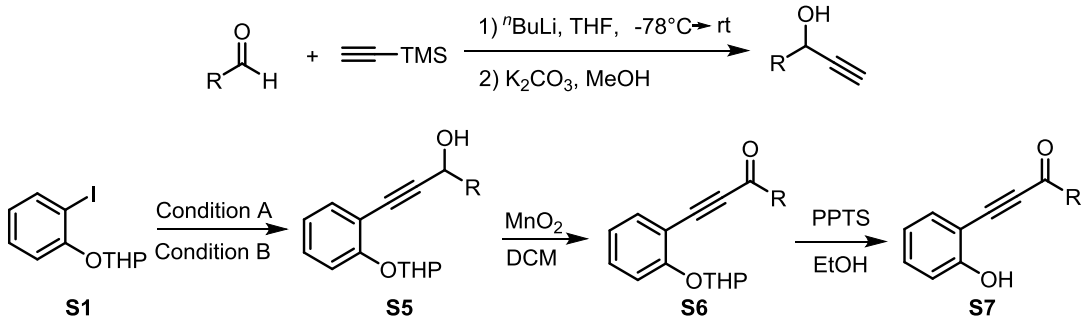


To an anhydrous flask was added $Pd(PPh_3)_2Cl_2$ (42.1 mg, 0.06 mmol, 2 mol%) and CuI (22.8 mg, 0.12 mmol, 4 mol%), then the mixture was degassed and flushed with N_2 for three times at room temperature. THF (15 mL), Et_3N (1.21 g, 12 mmol, 4.0 equiv.), **S2** (607 mg, 3 mmol, 1.0 equiv.) and benzoyl chloride (632 mg, 4.5 mmol, 1.5 equiv.) were added successively and the mixture was allowed to stirred at room temperature for 1 hour. Upon completion, the mixture was quenched with saturated NH_4Cl solution (15 mL), extracted with EtOAc (3 × 5 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 10) to get the pure product **S3** (653 mg, 2.1 mmol, 71%, yellow solid).

Place **S3** (613 mg, 2 mmol, 1.0 equiv.) in an anhydrous flask, add ethanol (6 mL), and Pyridinium p-Toluenesulfonate (151 mg, 0.6 mmol, 0.3 equiv.) is added to the mixture. Stir the mixture under 50 ℃ oil bath heating for 3 hours. After completion, concentrate and use EtOAc (3 × 5 mL) was extracted, washed with salt water, dried with Na_2SO_4, filtered and concentrated to obtain crude product. The crude product was purified by silica gel column chromatography (ethyl

acetate : petroleum ether = 1 : 5) to obtain pure product **S4-1a** (440 mg, 2.0 mmol, 99%, yellow solid).

C. General procedures for compound 1s-1u, 1aa.



Under N_2 atmosphere, to a solution of trimethylsilylacetylene (12 mmol, 1.2 equiv.) in THF (20 mL) at -78 ℃ was added ^{n}BuLi solution (2.5 M in hexanes, 4.4 mL, 11 mmol, 1.1 equiv.). The solution was allowed to warm to 0 ℃ over 1 hour and stirred at 0 ℃ for 30 minutes. Then the solution was cooled to -78 ℃ and aldehyde (10 mmol, 1.0 equiv.) was added. The reaction mixture was allowed to warm to room temperature over 1 hour and stirred at room temperature for 3 hours. NH_4Cl solution (20 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 20 mL). The combined organic layers were washed with H_2O (20 mL), saturated $NaHCO_3$ solution (20 mL), brine (20 mL), dried over Na_2SO_4, filtered and evaporated under reduced pressure to give the crude product. The crude propargylic alcohol bearing trimethylsilyl substituent was dissolved in MeOH (40 mL). Then K_2CO_3 (4.41 g, 30 mmol, 3 equiv.) was added and the resulting mixture was stirred at room temperature for 1 hour. The reaction mixture was filtered and evaporated. The residue was dissolved in EtOAc (30 mL) and washed with saturated NH_4Cl solution (10 mL) and brine (10 mL). The organic layer was dried over Na_2SO_4, filtered and evaporated under reduced pressure. Residue was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to give the propargylic alcohols in pure form.

Condition A: To an anhydrous bottle was added $Pd(PPh_3)_2Cl_2$ (84.2 mg, 0.12 mmol, 2 mol%) and CuI (45.6 mg, 0.24 mmol, 4 mol%), then the mixture was degassed and flushed with N_2 for three times at room temperature. THF (25 mL), Et_3N (2.42 g, 24 mmol, 4.0 equiv.), **S1** (1.82 g, 6 mmol, 1.0 equiv.) and propargylic alcohols (1.26 g, 9 mmol, 1.5 equiv.) were added successively and the mixture was allowed to stirred at room temperature for 2 hours. Upon completion, the mixture was quenched with saturated NH_4Cl solution (30 mL), extracted with EtOAc (3 × 10 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 2) to get the pure product **S5** (1.54 g, 4.86 mmol, 81%, oily liquid).

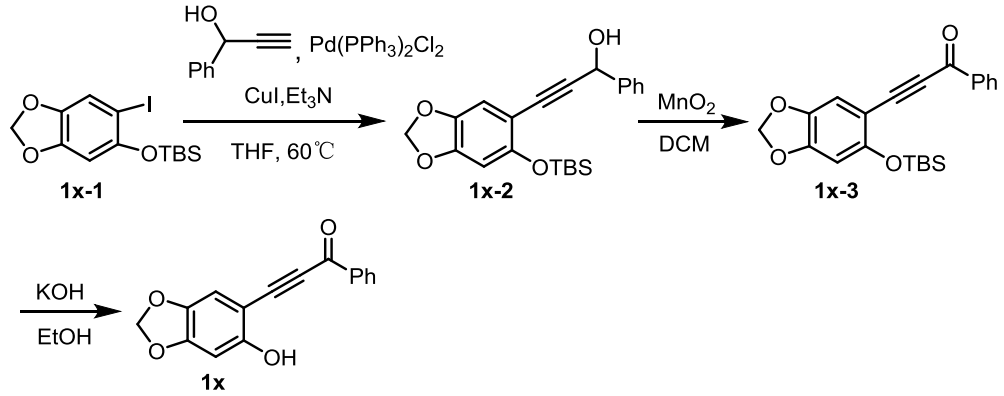
Condition B: To an anhydrous bottle was added $Pd(PPh_3)_2Cl_2$ (84.2 mg, 0.12 mmol, 2 mol%) and CuI (45.6 mg, 0.24 mmol, 4 mol%), then the mixture was degassed and flushed with N_2 for three times at room temperature. THF (25 mL), $(i\text{-Pr})_2NH$ (2.43 g, 24 mmol, 4.0 equiv.), **S1** (2.13 g, 6 mmol, 1.0 equiv.) and 1-phenylprop-2-yn-1-ol (1.19 g, 9 mmol, 1.5 equiv.) were added successively and the mixture was allowed to stirred at 50 ℃ for 2 hours. Upon completion, the mixture was quenched with saturated NH_4Cl solution (30 mL), extracted with EtOAc (3 × 10 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated to get the crude product which

was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 2) to get the pure product **S5** (1.85 g, 5.16 mmol, 86%, white solid).

Compound **S6** (633 mg, 2 mmol, 1.0 equiv.) was dissolved into DCM (10 mL) at room temperature, followed by MnO_2 (1.39 g, 16 mmol, 8 equiv.) was added. The resulting mixture was stirred at room temperature for 2 hours. When the reaction is completed, removing MnO_2 through filtration. Washing to colorless with DCM (3 × 10 mL). Then the organic layer was filtered and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to get the pure product **S6** (503.09 mg, 1.60 mmol, 80%, oily liquid).

Place **S6** (629 mg, 2 mmol, 1.0 equiv.) in an anhydrous flask, add ethanol (6 mL), and pyridinium *p*-toluenesulfonate (151 mg, 0.6 mmol, 0.3 equiv.) is added to the mixture. Stir the mixture under 50 ℃ oil bath heating for 3 hours. After completion, concentrate and use EtOAc (3 × 5 mL) was extracted, washed with salt water, dried with Na_2SO_4, filtered and concentrated to obtain crude product. The crude product was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 4) to obtain pure product **S7-1u** (456 mg, 2.0 mmol, 99%, yellow solid).

D. General procedures for compound 1x.



1x-1 were prepared according to the reported method[2].

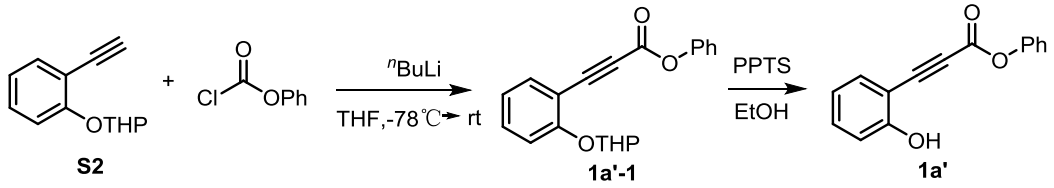
To an anhydrous bottle was added $Pd(PPh_3)_2Cl_2$ (84.2 mg, 0.12 mmol, 2 mol%) and CuI (45.6 mg, 0.24 mmol, 4 mol%), then the mixture was degassed and flushed with N_2 for three times at room temperature. THF (25 mL), Et_3N (2.42 g, 24 mmol, 4.0 equiv.), **1x-1** (2.27 g, 6 mmol, 1.0 equiv.) and 1-phenylprop-2-yn-1-ol (1.19 g, 9 mmol, 1.5 equiv.) were added successively and the mixture was allowed to stirred at 60 ℃ for 2 hours. Upon completion, the mixture was quenched with saturated NH_4Cl solution (30 mL), extracted with EtOAc (3 × 10 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to get the pure product **1x-2** (1.79 g, 4.68 mmol, 78%, yellow solid).

Compound **1x-2** (765 mg, 2 mmol, 1.0 equiv.) was dissolved into DCM (10 mL) at room temperature, followed by MnO_2 (1.39 g, 16 mmol, 8 equiv.) was added. The resulting mixture was stirred at room temperature for 2 hours. When the reaction is completed, removing MnO_2 through filtration. Washing to colorless with DCM (3 × 10 mL). Then the organic layer was filtered and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to get the pure product **1y-3** (548 mg, 1.44 mmol, 72%,

yellow solid).

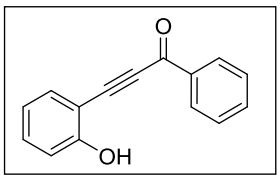
Compound **1x-3** (765 mg, 2 mmol, 1.0 equiv.) was dissolved into EtOH (10 mL) at room temperature, followed by KOH (1.39 g, 16 mmol, 8 equiv.) was added. The resulting mixture was stirred at room temperature for 2 hours. When the reaction is completed, removing KOH through filtration. Washing to colorless with DCM (3 × 10 mL). Then the organic layer was filtered and concentrated to get the crude product which was purified by silica gel column chromatography (dichloromethane : methano = 20 : 1) to get the pure product **1x** (522 mg, 1.96 mmol, 98%, yellow solid).

E. General procedures for compound 1a'.

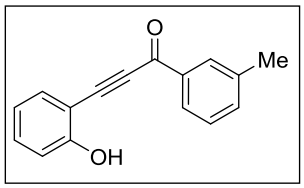


Add ^{n}BuLi solution (2.5 M in hexanes, 1 mL, 2.4 mmol) to the solution of **S2** (2.0 mmol) in THF (6 mL) at -78 ℃. Stir the mixture for 30 minutes at -78 ℃. Then add phenyl carbonochloridate (3 mmol) to the above reaction mixture. Allow the reaction mixture to warm to room temperature over 1 hour. Stir the reaction mixture at room temperature for 3 hours. Add NH_4Cl solution (10 mL) to quench the reaction. Extract the reaction mixture with EtOAc (3 × 10 mL). Wash the combined organic layers with H_2O (10 mL), saturated $NaHCO_3$ solution (10 mL), brine (10 mL). Dry the reaction mixture over Na_2SO_4. Filter and evaporate the reaction mixture under reduced pressure. Purify the residue by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 10) to get the pure product to obtain **1a'-1** (528 mg, 1.64 mmol, 82%, oily liquid).

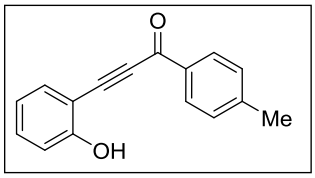
Place **1a'-1** (645 mg, 2 mmol, 1.0 equiv.) in an anhydrous flask, add ethanol (6 mL), and Pyridinium *p*-Toluenesulfonate (151 mg, 0.6 mmol, 0.3 equiv.) is added to the mixture. Stir the mixture under 50 ℃ oil bath heating for 3 hours. After completion, concentrate and use EtOAc (3 × 5 mL) was extracted, washed with salt water, dried with Na_2SO_4, filtered and concentrated to obtain crude product. The crude product was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to obtain pure product **1a'** (472 mg, 2.0 mmol, 99%, white solid).



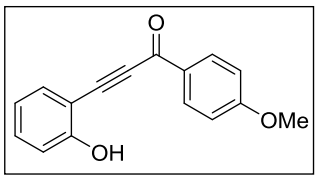
3-(2-hydroxyphenyl)-1-phenylprop-2-yn-1-one **1a.** Known compound[3]. General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 71% yield (653 mg), yellow solid. **^{1}H NMR (500 MHz, Acetone-d_6)** δ 9.51 (s, 1H), 8.37 – 8.27 (m, 2H), 7.69 (t, *J* = 7.29 Hz, 1H), 7.63 (dd, *J* = 7.77, 1.78 Hz, 1H), 7.58 (t, *J* = 7.81 Hz, 2H), 7.41 (td, *J* = 8.72, 1.81 Hz, 1H), 7.07 (d, *J* = 8.25 Hz, 1H), 6.96 (t, *J* = 7.51 Hz, 1H). **^{13}C NMR (125 MHz, Acetone-d_6)** δ 177.9, 161.2, 138.0, 135.3, 134.8, 133.7, 130.1, 129.5, 120.8, 116.8, 108.1, 91.6, 91.2.



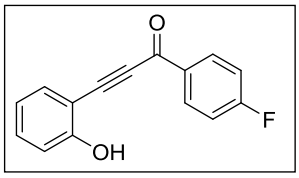
3-(2-hydroxyphenyl)-1-(m-tolyl)prop-2-yn-1-one **1b.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 85% yield (817 mg), brown solid. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.13 (s, 1H), 8.08 – 7.99 (m, 2H), 7.54 (dd, *J* = 7.74, 1.70 Hz, 1H), 7.45 – 7.29 (m, 3H), 7.06 (d, *J* = 8.30 Hz, 1H), 6.92 (t, *J* = 7.54 Hz, 1H), 2.40 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 179.0, 159.8, 138.6, 136.5, 135.3, 134.0, 133.3, 130.1, 128.6, 127.4, 120.5, 116.4, 106.9, 92.8, 91.1, 21.3. **HRMS (ESI)** m/z calcd for $C_{16}H_{12}NaO_2^+$ $(M+Na)^+$ 259.0730, found 259.0730.



3-(2-hydroxyphenyl)-1-(p-tolyl)prop-2-yn-1-one **1c.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 49% yield (471 mg), orange solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.41 (s, 1H), 8.20 (d, *J* = 8.27 Hz, 2H), 7.61 (dd, *J* = 7.77, 1.72 Hz, 1H), 7.43 – 7.38 (m, 1H), 7.36 (d, *J* = 7.95 Hz, 2H), 7.07 (d, *J* = 8.11 Hz, 1H), 6.95 (t, *J* = 7.53 Hz, 1H), 2.41 (s, 3H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 177.4, 161.0, 145.6, 135.5, 135.1, 133.4, 130.1, 129.9, 120.6, 116.5, 107.9, 91.5, 90.5, 21.5. **HRMS (ESI)** m/z calcd for $C_{16}H_{12}NaO_2^+$ $(M+Na)^+$ 259.0730, found 259.0730.

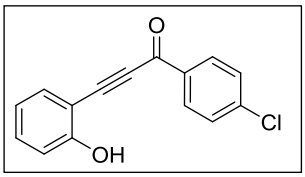


3-(2-hydroxyphenyl)-1-(4-methoxyphenyl)prop-2-yn-1-one **1d**. General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 3/1). 72% yield (727 mg), yellow solid. **^{1}H NMR (400 MHz, DMSO-d_6)** δ 10.71 (s, 1H), 8.20 (d, *J* = 8.84 Hz, 2H), 7.58 (dd, *J* = 7.67, 1.69 Hz, 1H), 7.46 – 7.33 (m, 1H), 7.11 (d, *J* = 8.88 Hz, 2H), 7.01 (d, *J* = 7.38 Hz, 1H), 6.89 (t, *J* = 7.50 Hz, 1H), 3.87 (s, 3H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 175.7, 164.2, 160.8, 134.5, 133.0, 131.6, 129.9, 119.5, 115.9, 114.3, 106.6, 90.7, 90.6, 55.7. **HRMS (ESI)** m/z calcd for $C_{16}H_{12}NaO_3^+$ $(M+Na)^+$ 275.0679, found 275.0678.

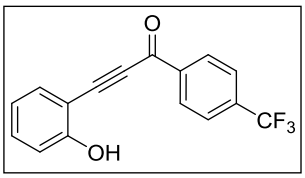


1-(4-fluorophenyl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1e.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 81% yield (788 mg),

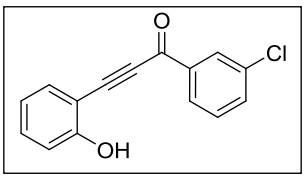
brown solid. **^{1}H NMR (400 MHz, DMSO-d_6)** δ 10.78 (s, 1H), 8.42 – 8.19 (m, 2H), 7.61 (dd, *J* = 7.69, 1.71 Hz, 1H), 7.50 – 7.32 (m, 3H), 7.01 (d, *J* = 8.38 Hz, 1H), 6.90 (t, *J* = 7.53 Hz, 1H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 175.5, 165.8(d, *J* = 253.0 Hz), 161.0, 134.6, 133.4, 133.3, 132.2(d, *J* = 10.0 Hz), 119.5, 116.2 (d, *J* =22.0 Hz) , 115.9, 106.2, 92.1, 90.4. **HRMS (ESI)** m/z calcd for $C_{15}H_9NaFO_2^+$ $(M+Na)^+$ 263.0479, found 263.0478.



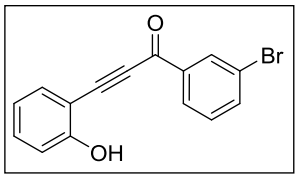
1-(4-bromophenyl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1f.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 57% yield (583 mg), brown solid. **^{1}H NMR (500 MHz, Acetone-d_6)** δ 9.54 (s, 1H), 8.30 (d, *J* = 8.77 Hz, 2H), 7.61 (d, *J* = 8.34 Hz, 1H), 7.59 (d, *J* = 8.78 Hz, 2H), 7.41 (t, *J* = 7.38 Hz, 1H), 7.07 (d, *J* = 8.22 Hz, 1H), 6.96 (t, *J* = 7.54 Hz, 1H). **^{13}C NMR (125 MHz, Acetone-d_6)** δ 176.6, 161.3, 140.6, 136.6, 135.3, 133.8, 131.7, 129.7, 120.8, 116.7, 107.8, 91.9, 91.3. **HRMS (ESI)** m/z calcd for $C_{15}H_9NaClO_2^+$ $(M+Na)^+$ 279.0183, found 279.0187.



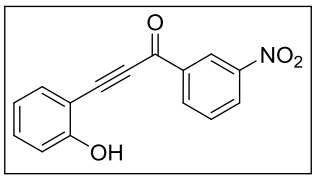
3-(2-hydroxyphenyl)-1-(4-(trifluoromethyl)phenyl)prop-2-yn-1-one **1g**. General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 93% yield (1.04 g), yellow solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.64 (s, 1H), 8.49 (d, *J* = 8.14 Hz, 2H), 7.91 (d, *J* = 8.16 Hz, 2H), 7.64 (dd, *J* = 7.76, 1.65 Hz, 1H), 7.48 – 7.38 (m, 1H), 7.08 (d, *J* = 7.44 Hz, 1H), 6.97 (t, *J* = 7.66 Hz, 1H). **13C NMR (100 MHz, Acetone-d_6)** δ 176.7, 161.5, 140.7, 135.4, 135.0 (d, *J* = 32.1 Hz), 134.1, 130.7, 126.5 (q, *J* = 3.7 Hz), 124.7 (d, *J* = 272.3 Hz), 120.8, 116.7, 107.6, 92.8, 91.3. **HRMS (ESI)** m/z calcd for $C_{16}H_9NaF_3O_2^+$ $(M+Na)^+$ 313.0447, found 313.0445.



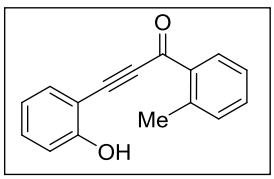
1-(3-chlorophenyl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1h.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 80% yield (818 mg), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.63 (s, 1H), 8.28 (d, *J* = 1.98 Hz, 1H), 8.21 (dd, *J* = 7.75, 1.50 Hz, 1H), 7.64 (d, *J* = 4.73 Hz, 1H), 7.62 – 7.52 (m, 2H), 7.40 (t, *J* = 7.81 Hz, 1H), 7.07 (d, *J* = 8.28 Hz, 1H), 6.94 (t, *J* = 7.51 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 175.9, 160.9, 138.9, 134.8, 134.6, 133.9, 133.4, 130.7, 129.3, 127.8, 120.2, 116.1, 107.1, 92.0, 90.8. **HRMS (ESI)** m/z calcd for $C_{15}H_9NaClO_2^+$ $(M+Na)^+$ 279.0183, found 279.0187.



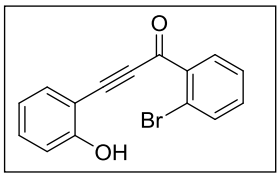
1-(3-bromophenyl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1i.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 76% yield (878 mg), yellow solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.69 (s, 1H), 8.46 (t, *J* = 1.86 Hz, 1H), 8.28 (d, *J* = 7.75 Hz, 1H), 7.84 (d, *J* = 7.27 Hz, 1H), 7.61 (dd, *J* = 7.74, 1.71 Hz, 1H), 7.53 (t, *J* = 7.88 Hz, 1H), 7.45 – 7.39 (m, 1H), 7.08 (d, *J* = 7.92 Hz, 1H), 6.96 (t, *J* = 7.53 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 176.2, 161.4, 139.6, 137.3, 135.2, 133.9, 132.7, 131.4, 128.6, 123.1, 120.7, 116.5, 107.5, 92.4, 91.1. **HRMS (ESI)** m/z calcd for $C_{15}H_9NaBrO_2^+$ $(M+Na)^+$ 322.9678, found 322.9680.



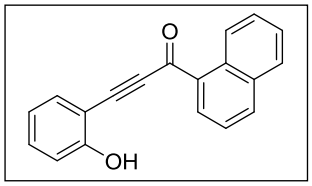
3-(2-hydroxyphenyl)-1-(3-nitrophenyl)prop-2-yn-1-one **1j.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 76% yield (801 mg), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.85 (s, 1H), 9.11 (t, *J* = 2.01 Hz, 1H), 8.67 (d, *J* = 7.84 Hz, 1H), 8.54 (d, *J* = 6.96 Hz, 1H), 7.91 (t, *J* = 7.95 Hz, 1H), 7.64 (dd, *J* = 7.69, 1.70 Hz, 1H), 7.50 – 7.42 (m, 1H), 7.11 (d, *J* = 8.40 Hz, 1H), 6.99 (t, *J* = 7.51 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 175.6, 161.8, 149.4, 139.0, 135.4, 135.3, 134.3, 131.1, 128.8, 124.7, 120.7, 116.6, 107.4, 93.5, 91.1. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}NO_4^+$ $(M+H)^+$ 268.0604, found 268.0605.



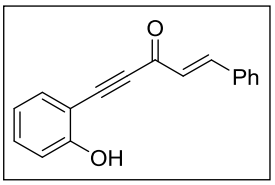
3-(2-hydroxyphenyl)-1-(o-tolyl)prop-2-yn-1-one **1k.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 79% yield (759 mg), yellow solid. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.29 (d, *J* = 7.80 Hz, 1H), 7.88 (s, 1H), 7.47 (dd, *J* = 7.80, 1.68 Hz, 1H), 7.39 (t, *J* = 7.41 Hz, 1H), 7.35 – 7.25 (m, 2H), 7.20 (d, *J* = 7.55 Hz, 1H), 7.01 (d, *J* = 8.31 Hz, 1H), 6.87 (t, *J* = 7.52 Hz, 1H), 2.62 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 180.5, 159.7, 140.8, 135.2, 133.8, 133.7, 133.3, 133.2, 132.3, 126.1, 120.5, 116.3, 107.0, 94.2, 89.8, 22.1. **HRMS (ESI)** m/z calcd for $C_{16}H_{12}NaO_2^+$ $(M+Na)^+$ 259.0730, found 259.0730.



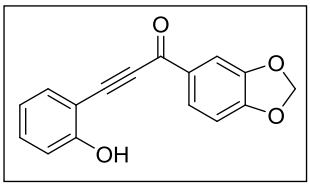
1-(2-bromophenyl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1l**. General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 90% yield (1.04 g), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.47 (s, 1H), 8.29 (dd, *J* = 7.63, 1.88 Hz, 1H), 7.76 (d, *J* = 8.00 Hz, 1H), 7.57 (d, *J* = 7.55 Hz, 2H), 7.51 (t, *J* = 7.68 Hz, 1H), 7.40 (t, *J* = 7.96 Hz, 1H), 7.04 (d, *J* = 8.23 Hz, 1H), 6.94 (t, *J* = 7.55 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 177.3, 161.1, 138.1, 135.5, 135.1, 134.3, 133.8, 133.8, 128.4, 120.9, 120.7, 116.7, 107.7, 92.4, 91.9. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}BrO_2^+$ $(M+H)^+$ 300.9859, found 300.9861.



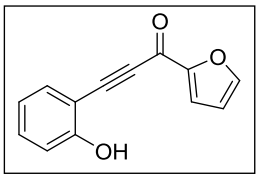
3-(2-hydroxyphenyl)-1-(naphthalen-1-yl)prop-2-yn-1-one **1m.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 87% yield (930 mg), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.52 (s, 1H), 9.27 (d, *J* = 8.76 Hz, 1H), 8.91 (dd, *J* = 7.28, 1.31 Hz, 1H), 8.21 (d, *J* = 8.22 Hz, 1H), 8.01 (d, *J* = 7.56 Hz, 1H), 7.73 – 7.65 (m, 2H), 7.64 – 7.57 (m, 2H), 7.44 – 7.36 (m, 1H), 7.09 (d, *J* = 7.28 Hz, 1H), 6.96 (td, *J* = 7.51, 1.06 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 179.9, 161.1, 135.7, 135.7, 135.1, 134.8, 133.5, 133.4, 131.3, 129.5, 129.5, 127.4, 126.4, 125.5, 120.7, 116.7, 108.2, 93.1, 89.7. **HRMS (ESI)** m/z calcd for $C_{19}H_{12}NaO_2^+$ $(M+Na)^+$ 295.0730, found 295.0733.



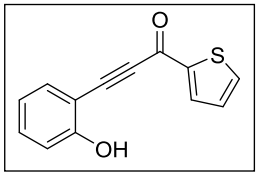
(E)-5-(2-hydroxyphenyl)-1-phenylpent-1-en-4-yn-3-one **1n.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 69% yield (688 mg), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.52 (s, 1H), 8.24 (d, *J* = 16.15 Hz, 1H), 7.73 – 7.66 (m, 1H), 7.58 (dd, *J* = 7.68, 1.75 Hz, 1H), 7.48 – 7.40 (m, 4H), 7.44 – 7.34 (m, 1H), 7.09 (d, *J* = 7.24 Hz, 1H), 6.98 – 6.89 (m, 2H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 177.8, 160.2, 148.7, 134.3, 134.3, 132.7, 131.0, 129.0, 128.7, 128.6, 120.0, 116.0, 107.4, 90.6, 89.0. **HRMS (ESI)** m/z calcd for $C_{17}H_{12}NaO_2^+$$(M+Na)^+$ 271.0730, found 271.0729.



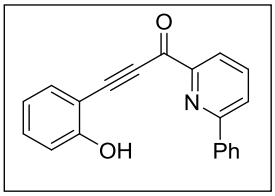
1-(benzo[d][1,3]dioxol-5-yl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1o.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 76% yield (799 mg), yellow solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.53 (s, 1H), 8.02 (dd, *J* = 8.13, 1.73 Hz, 1H), 7.66 (s, 1H), 7.60 (d, *J* = 7.83 Hz, 1H), 7.44 – 7.37 (m, 1H), 7.06 (d, *J* = 8.16 Hz, 1H), 7.00 (dd, *J* = 8.16, 2.48 Hz, 1H), 6.95 (t, *J* = 7.52 Hz, 1H), 6.16 (s, 2H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 176.1, 161.2, 153.7, 149.2, 135.2, 133.5, 133.1, 127.6, 120.8, 116.7, 108.8, 108.6, 108.2, 103.3, 91.5, 90.3. **HRMS (ESI)** m/z calcd for $C_{16}H_{10}NaO_4^+$ $(M+Na)^+$ 289.0471, found 289.0473.



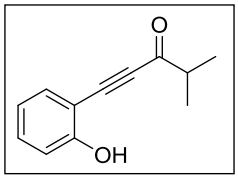
1-(furan-2-yl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1p.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 64% yield (569 mg), yellow solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.51 (s, 1H), 7.92 (s, 1H), 7.66 (d, *J* = 3.41 Hz, 1H), 7.58 (d, *J* = 7.89 Hz, 1H), 7.39 (t, *J* = 7.64 Hz, 1H), 7.05 (d, *J* = 7.30 Hz, 1H), 6.94 (t, *J* = 7.49 Hz, 1H), 6.74 – 6.70 (m, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 164.1, 160.6, 153.6, 148.9, 134.6, 133.1, 121.9, 120.2, 116.2, 113.0, 107.2, 90.5, 89.0. **HRMS (ESI)** m/z calcd for $C_{13}H_9O_3^+$ $(M+H)^+$ 213.0546, found 213.0548.



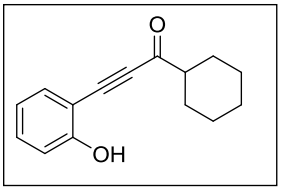
3-(2-hydroxyphenyl)-1-(thiophen-2-yl)prop-2-yn-1-one **1q.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 57% yield (534 mg), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.52 (s, 1H), 8.22 (d, *J* = 3.56 Hz, 1H), 7.95 (d, *J* = 4.81 Hz, 1H), 7.59 (d, *J* = 7.68 Hz, 1H), 7.39 (t, *J* = 7.81 Hz, 1H), 7.25 (t, *J* = 4.34 Hz, 1H), 7.06 (d, *J* = 8.36 Hz, 1H), 6.94 (t, *J* = 7.53 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 169.3, 160.3, 145.1, 135.9, 135.6, 134.4, 133.0, 128.6, 120.0, 115.9, 107.0, 90.3, 89.3. **HRMS (ESI)** m/z calcd for $C_{13}H_8NaO_2S^+$ $(M+Na)^+$ 251.0137, found 251.0137.



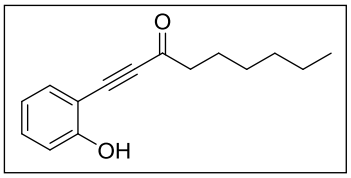
3-(2-hydroxyphenyl)-1-(6-phenylpyridin-2-yl)prop-2-yn-1-one **1r.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 31% yield (357 mg), yellow solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.28 (s, 1H), 8.34 (d, *J* = 6.99 Hz, 2H), 8.23 – 8.17 (m, 1H), 8.13 – 8.06 (m, 2H), 7.67 (dd, *J* = 7.75, 1.72 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.50 – 7.47 (m, 1H), 7.46 – 7.41 (m, 1H), 7.06 (d, *J* = 8.19 Hz, 1H), 6.99 (t, *J* = 7.50 Hz, 1H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 178.6, 161.3, 157.6, 153.9, 139.1, 138.8, 135.4, 133.9, 130.3, 129.5, 127.8, 124.7, 121.6, 120.8, 116.7, 108.2, 93.4, 93.0. **HRMS (ESI)** m/z calcd for $C_{20}H_{13}NaNO_2^+$ $(M+Na)^+$ 322.0838, found 322.0840.



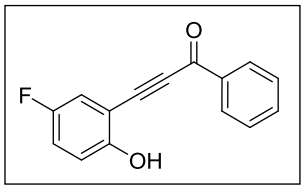
1-(2-hydroxyphenyl)-4-methylpent-1-yn-3-one **1s.** General procedure **C**. Condition **A.** The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 80% yield (654 mg), yellow solid. **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 7.44 (dd, *J* = 7.82, 1.83 Hz, 1H), 7.39 – 7.32 (m, 1H), 7.06 (s, 1H), 7.00 (d, *J* = 8.43 Hz, 1H), 6.90 (t, *J* = 7.56 Hz, 1H), 2.80 (hept, *J* = 6.94 Hz, 1H), 1.28 (d, *J* = 7.05 Hz, 6H). **^{13}C NMR (125 MHz, Chloroform-*d*)** δ 192.7, 159.3, 133.8, 133.3, 120.6, 116.1, 106.7, 93.3, 88.3, 42.9, 18.2. **HRMS (ESI)** m/z calcd for $C_{12}H_{12}NaO_2^+$ $(M+Na)^+$ 211.0730, found 211.0730.



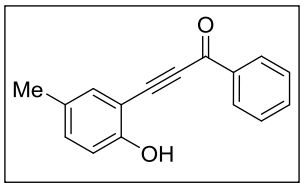
1-cyclohexyl-3-(2-hydroxyphenyl)prop-2-yn-1-one **1t.** General procedure **C**. Condition **A.** The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 78% yield (731 mg), white solid. **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 7.47 (s, 1H), 7.43 (dd, *J* = 7.81, 1.82 Hz, 1H), 7.37 – 7.29 (m, 1H), 7.00 (d, *J* = 8.39 Hz, 1H), 6.88 (t, *J* = 7.50 Hz, 1H), 2.54 (tt, *J* = 11.22, 3.60 Hz, 1H), 2.05 (d, *J* = 11.49 Hz, 2H), 1.80 (dt, *J* = 12.89, 3.71 Hz, 2H), 1.66 (d, *J* = 12.60 Hz, 1H), 1.56 – 1.43 (m, 2H), 1.39 – 1.27 (m, 2H), 1.23 (tt, *J* = 11.98, 3.22 Hz, 1H). **^{13}C NMR (125 MHz, Chloroform-*d*)** δ 192.3, 159.4, 133.9, 133.2, 120.5, 116.2, 106.7, 93.3, 88.7, 52.0, 28.4, 25.8, 25.4. **HRMS (ESI)** m/z calcd for $C_{15}H_{16}NaO_2^+$ $(M+Na)^+$ 251.1043, found 251.1043.



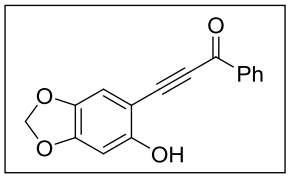
1-(2-hydroxyphenyl)non-1-yn-3-one **1u.** General procedure **C**. Condition **A.** The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 91% yield (858 mg), brown liquid. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.57 (s, 1H), 7.42 (dd, *J* = 7.77, 1.70 Hz, 1H), 7.38 – 7.28 (m, 1H), 7.00 (d, *J* = 8.36 Hz, 1H), 6.89 (t, *J* = 7.57 Hz, 1H), 2.69 (t, *J* = 7.46 Hz, 2H), 1.73 (p, *J* = 7.39 Hz, 2H), 1.41 – 1.23 (m, 6H), 0.88 (t, *J* = 6.88 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 189.2, 159.4, 133.9, 133.3, 120.5, 116.2, 106.6, 93.9, 87.9, 45.3, 31.6, 28.7, 24.2, 22.5, 14.1. **HRMS (ESI)** m/z calcd for $C_{15}H_{18}NaO_2^+$ $(M+Na)^+$ 253.1199, found 253.1200.



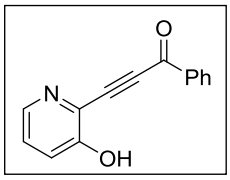
3-(5-fluoro-2-hydroxyphenyl)-1-phenylprop-2-yn-1-one **1v.** General procedure **C**. Condition **A.** The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 82% yield (798 mg), yellow solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.56 (s, 1H), 8.30 (dd, *J* = 8.39, 1.40 Hz, 2H), 7.70 – 7.65 (m, 1H), 7.58 – 7.53 (m, 2H), 7.37 (dd, *J* = 8.57, 3.15 Hz, 1H), 7.22 – 7.15 (m, 1H), 7.06 (dd, *J* = 9.08, 4.57 Hz, 1H). **13C NMR (100 MHz, Acetone-d_6)** δ 157.2 (d, *J* = 1.6 Hz), 155.8 (d, *J* = 237.0 Hz), 137.2, 134.4, 129.3 (d, *J* = 64.7 Hz), 120.0 (d, *J* = 49.5 Hz), 120.0, 117.5, 117.4, 108.2 (d, *J* = 9.5 Hz), 91.2, 89.03, 89.00. **HRMS (ESI)** m/z calcd for $C_{15}H_9NaFO_2^+$ $(M+Na)^+$ 263.0479, found 263.0479.



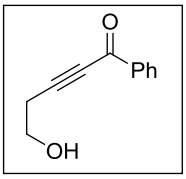
3-(2-hydroxy-5-methylphenyl)-1-phenylprop-2-yn-1-one **1w.** General procedure **C**. Condition **A.** The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 76% yield (730 mg), brown solid. **^{1}H NMR (400 MHz, Acetone-d_6)** δ 9.30 (s, 1H), 8.34 (dd, *J* = 8.42, 1.47 Hz, 2H), 7.66 – 7.59 (m, 1H), 7.52 (t, *J* = 7.60 Hz, 2H), 7.32 (d, *J* = 1.90 Hz, 1H), 7.15 (dd, *J* = 8.43, 2.03 Hz, 1H), 6.97 (d, *J* = 8.43 Hz, 1H), 2.21 (s, 3H). **^{13}C NMR (100 MHz, Acetone-d_6)** δ 176.8, 157.7, 136.4, 133.7, 133.3, 133.2, 128.7, 128.5, 128.0, 115.2, 106.2, 90.8, 90.3, 18.9. **HRMS (ESI)** m/z calcd for $C_{16}H_{12}NaO_2^+$ $(M+Na)^+$ 259.0730, found 259.0730.



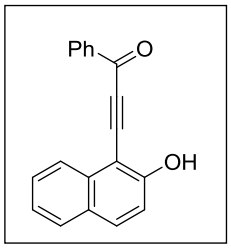
3-(6-hydroxybenzo[d][1,3]dioxol-5-yl)-1-phenylprop-2-yn-1-one **1x.** General procedure **D**. The product was obtained by column chromatography (Dichloromethane/methanol = 20/1). 78% yield (820 mg), yellow solid. **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 10.71 (s, 1H), 8.24 (d, *J* = 6.97 Hz, 2H), 7.69 (t, *J* = 7.31 Hz, 1H), 7.57 (t, *J* = 7.69 Hz, 2H), 7.12 (d, *J* = 4.13 Hz, 1H), 6.58 (s, 1H), 6.04 (s, 2H). **^{13}C NMR (100 MHz, DMSO-*d*$_6$)** δ 176.8, 159.1, 151.8, 140.4, 136.8, 134.2, 129.2, 128.9, 111.4, 102.1, 97.7, 97.2, 93.8, 91.0. **HRMS (ESI)** m/z calcd for $C_{16}H_{10}O_4Na^+$ $(M+Na)^+$ 289.0471, found 289.0472.



3-(3-hydroxypyridin-2-yl)-1-phenylprop-2-yn-1-one **1y.** General procedure **B**. The product was obtained by column chromatography (Dichloromethane/methanol = 20/1). 81% yield (747 mg), yellow solid. **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 11.30 (s, 1H), 8.23 (d, *J* = 6.90 Hz, 2H), 8.18 (dd, *J* = 4.00, 1.79 Hz, 1H), 7.74 (t, *J* = 7.37 Hz, 1H), 7.61 (t, *J* = 7.66 Hz, 2H), 7.46 – 7.39 (m, 2H). **^{13}C NMR (100 MHz, DMSO-*d*$_6$)** δ 177.1, 158.3, 141.9, 136.3, 134.8, 129.3, 129.1, 127.4, 127.0, 123.5, 89.9, 88.7. **HRMS (ESI)** m/z calcd for $C_{14}H_{10}NO_2^+$ $(M+H)^+$ 224.0706, found 224.0707.

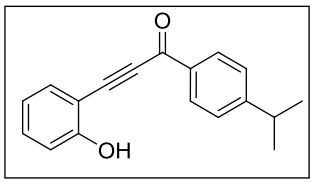


5-hydroxy-1-phenylpent-2-yn-1-one **1z.** Known compound[5]. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 69% yield (361 mg), brown solid. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.04 (d, *J* = 7.01 Hz, 2H), 7.49 (t, *J* = 7.39 Hz, 1H), 7.36 (t, *J* = 7.72 Hz, 2H), 4.17 (s, 1H), 3.84 (d, *J* = 5.43 Hz, 2H), 2.70 (t, *J* = 6.36 Hz, 2H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.5, 136.2, 134.1, 129.5, 128.4, 94.5, 80.3, 59.9, 23.3.

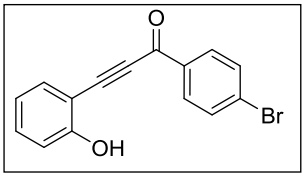


3-(2-hydroxynaphthalen-1-yl)-1-phenylprop-2-yn-1-one **1aa.** Known compound[4]. General procedure **C**. Condition **B.** The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 76% yield (813 mg), yellow solid. **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 11.41 (s, 1H), 8.36 (d, *J* = 8.57 Hz, 2H), 8.14 (d, *J* = 8.31 Hz, 1H), 7.99 (d, *J* = 8.93 Hz, 1H), 7.87 (d, *J*

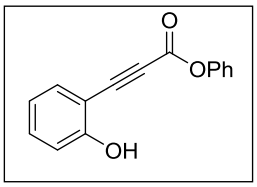
= 8.08 Hz, 1H), 7.71 (t, *J* = 7.35 Hz, 1H), 7.63 (q, *J* = 7.32, 6.75 Hz, 3H), 7.39 (t, *J* = 7.51 Hz, 1H), 7.33 (d, *J* = 9.02 Hz, 1H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 177.1, 162.1, 136.8, 134.5, 134.3, 133.9, 129.3, 129.0, 128.8, 128.5, 127.4, 124.1, 123.7, 117.9, 98.9, 96.4, 90.4.



3-(2-hydroxyphenyl)-1-(4-isopropylphenyl)prop-2-yn-1-one **1ab**, General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 60% yield (627 mg), white solid. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.37 (s, 1H), 8.19 (d, *J* = 8.40 Hz, 2H), 7.55 (dd, *J* = 7.75, 1.75 Hz, 1H), 7.37 – 7.33 (m, 1H), 7.32 (d, *J* = 8.33 Hz, 2H), 7.09 (d, *J* = 8.23 Hz, 1H), 6.91 (td, *J* = 7.53, 1.09 Hz, 1H), 2.95 (hept, *J* = 6.91 Hz, 1H), 1.25 (d, *J* = 7.01 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.7, 159.8, 156.3, 134.4, 134.0, 133.2, 130.2, 126.8, 120.4, 116.4, 107.0, 92.7, 91.1, 34.4, 23.6. **HRMS (ESI)** m/z calcd for $C_{18}H_{16}O_2Na^+$ $(M+Na)^+$ 287.1043, found 287.1034.



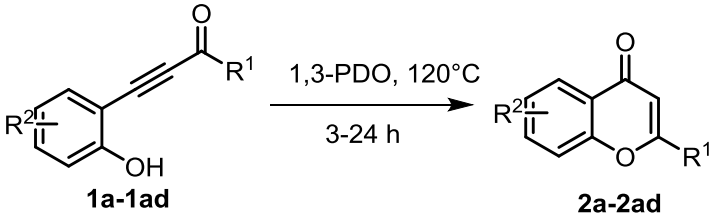
1-(4-bromophenyl)-3-(2-hydroxyphenyl)prop-2-yn-1-one **1ad.** General procedure **B**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 93% yield (1.07 g), yellow solid. **^{1}H NMR (400 MHz, DMSO-d_6)** δ 10.80 (s, 1H), 8.14 (d, *J* = 8.54 Hz, 2H), 7.82 (d, *J* = 8.45 Hz, 2H), 7.61 (dd, *J* = 7.75, 1.72 Hz, 1H), 7.46 – 7.33 (m, 1H), 7.01 (d, *J* = 8.42 Hz, 1H), 6.90 (t, *J* = 7.52 Hz, 1H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 176.0, 161.0, 135.6, 134.6, 133.4, 132.1, 131.0, 128.8, 119.5, 115.9, 106.1, 92.5, 90.3. **HRMS (ESI)** m/z calcd for $C_{15}H_9NaBrO_2^+$ $(M+Na)^+$ 322.9678, found 322.9680.



phenyl 3-(2-hydroxyphenyl)propiolate **1a'**. Known compound[6]. General procedure **E**. The product was obtained by column chromatography (Petroleum ether/EtOAc = 5/1). 82% yield (793 mg), white solid. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.48 (dd, *J* = 7.80, 1.68 Hz, 1H), 7.43 (t, *J* = 7.88 Hz, 2H), 7.41 – 7.35 (m, 1H), 7.30 (t, *J* = 7.42 Hz, 1H), 7.20 (d, *J* = 7.36 Hz, 2H), 6.98 (d, *J* = 8.75 Hz, 1H), 6.93 (t, *J* = 7.06 Hz, 1H), 6.49 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 159.2, 152.7, 150.2, 133.9, 133.6, 129.8, 126.7, 121.5, 120.8, 116.1, 105.8, 86.4, 85.2.

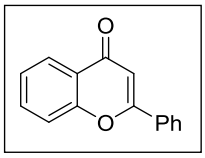
4. Experimental Procedures and Characterization Data for Products

General procedure I: 2a-2ad.

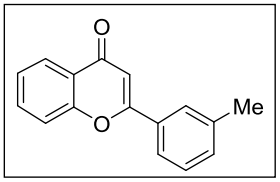


1a-1ad **2a-2ad**

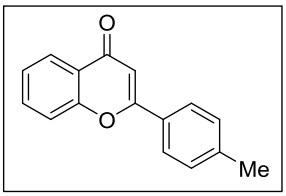
The substrate **1a-1ad** (1 mmol, 1 equiv.) was added in a bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 120 ℃ in oil bath for 5-24 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to get the pure product. **2a-2ad** were synthesized through general procedure **I**.



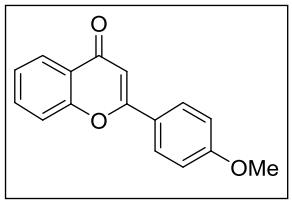
2-phenyl-4H-chromen-4-one **2a**. General procedure **I**, 95% yield (219 mg), 3 h, yellow solid. (Petroleum ether/EtOAc = 2/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.22 (d, *J* = 7.96 Hz, 1H), 7.90 (d, *J* = 9.35 Hz, 2H), 7.72 – 7.65 (m, 1H), 7.55 (d, *J* = 8.38 Hz, 1H), 7.51 (d, *J* = 6.09 Hz, 3H), 7.40 (t, *J* = 7.52 Hz, 1H), 6.81 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.5, 163.4, 156.3, 133.9, 131.8, 131.7, 129.1, 126.3, 125.7, 125.3, 124.0, 118.2, 107.6. **HRMS (ESI)** m/z calcd for $C_{15}H_{11}O_2^+$ $(M+H)^+$ 223.0754, found 223.0752.



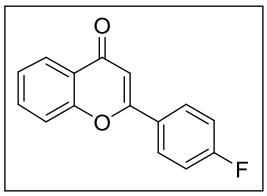
2-(m-tolyl)-4H-chromen-4-one **2b**. General procedure **I**, 97% yield (230 mg), 8 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.20 (dd, *J* = 7.94, 1.73 Hz, 1H), 7.67 (d, *J* = 8.17 Hz, 2H), 7.66 – 7.62 (m, 1H), 7.53 (d, *J* = 8.32 Hz, 1H), 7.42 – 7.33 (m, 2H), 7.31 (d, *J* = 7.46 Hz, 1H), 6.77 (s, 1H), 2.42 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.4, 163.6, 156.3, 138.9, 133.7, 132.5, 131.7, 129.0, 126.9, 125.7, 125.2, 124.0, 123.5, 118.1, 107.5, 21.5. **HRMS (ESI)** m/z calcd for $C_{16}H_{13}O_2^+$ $(M+H)^+$ 237.0910, found 237.0905.



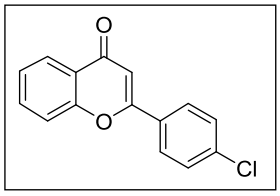
2-(p-tolyl)-4H-chromen-4-one **2c**. General procedure **I**, 94% yield (230 mg), 10 h, green solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.16 (dd, *J* = 7.93, 1.69 Hz, 1H), 7.72 (d, *J* = 8.35 Hz, 2H), 7.62 (ddd, *J* = 8.70, 7.11, 1.72 Hz, 1H), 7.47 (d, *J* = 8.51 Hz, 1H), 7.37 – 7.31 (m, 1H), 7.23 (d, *J* = 8.04 Hz, 2H), 6.71 (s, 1H), 2.35 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.3, 163.5, 156.1, 142.2, 133.6, 129.7, 128.8, 126.1, 125.6, 125.1, 123.9, 118.0, 106.8, 21.5. **HRMS (ESI)** m/z calcd for $C_{16}H_{13}O_2^+$ $(M+H)^+$ 237.0910, found 237.0905.



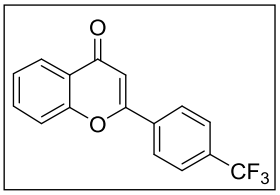
2-(4-methoxyphenyl)-4H-chromen-4-one **2d**. General procedure **I**, 95% yield (240 mg), 10 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.19 (dd, *J* = 7.94, 1.61 Hz, 1H), 7.83 (d, *J* = 8.90 Hz, 2H), 7.68 – 7.61 (m, 1H), 7.50 (d, *J* = 8.33 Hz, 1H), 7.37 (t, *J* = 7.42 Hz, 1H), 6.98 (d, *J* = 8.90 Hz, 2H), 6.70 (s, 1H), 3.85 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.4, 163.4, 162.4, 156.2, 133.6, 128.0, 125.7, 125.1, 124.0, 123.99, 123.96, 118.0, 114.5, 106.2, 55.5. **HRMS (ESI)** m/z calcd for $C_{16}H_{13}O_3^+$ $(M+H)^+$ 253.0859, found 253.0860.



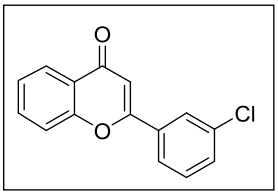
2-(4-fluorophenyl)-4H-chromen-4-one **2e**. General procedure **I**, 87% yield (210 mg), 8 h, brown solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.17 (dd, *J* = 7.92, 1.75 Hz, 1H), 7.92 – 7.81 (m, 2H), 7.69 – 7.60 (m, 1H), 7.49 (d, *J* = 8.31 Hz, 1H), 7.36 (t, *J* = 7.47 Hz, 1H), 7.16 (t, *J* = 8.56 Hz, 2H), 6.70 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.2, 164.8 (d, *J* =252.0 Hz), 159.2 (d, *J* =620.0 Hz) , 133.9 , 128.5(d, *J* =9.0 Hz), 128.0, 127.9, 125.7, 125.3, 123.8, 117.2 (d, *J* =163.0 Hz) , 116.2, 107.3. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}FO_2^+$ $(M+H)^+$ 241.0659, found 241.0660.



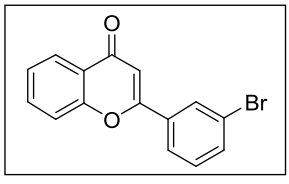
2-(4-chlorophenyl)-4H-chromen-4-one **2f**. General procedure **I**, 84% yield (216 mg), 12 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.18 (dd, *J* = 8.15, 1.76 Hz, 1H), 7.82 (d, *J* = 8.64 Hz, 2H), 7.67 (ddd, *J* = 8.65, 7.08, 1.70 Hz, 1H), 7.52 (d, *J* = 8.34 Hz, 1H), 7.46 (d, *J* = 8.65 Hz, 2H), 7.39 (t, *J* = 7.55 Hz, 1H), 6.75 (s, 1H). **^{13}C NMR (125 MHz, Chloroform-*d*)** δ 178.3, 162.3, 156.2, 137.9, 134.0, 130.2, 129.4, 127.6, 125.8, 125.4, 123.9, 118.1, 107.7. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}ClO_2^+$ $(M+H)^+$ 257.0364, found 257.0365.



2-(4-(trifluoromethyl)phenyl)-4H-chromen-4-one **2g**. General procedure **I**, 74% yield (215 mg), 12 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.19 (d, *J* = 7.88 Hz, 1H), 8.00 (d, *J* = 8.15 Hz, 2H), 7.75 (d, *J* = 8.20 Hz, 2H), 7.72 – 7.65 (m, 1H), 7.55 (d, *J* = 8.34 Hz, 1H), 7.45 – 7.36 (m, 1H), 6.82 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.2, 161.6, 156.2, 135.2, 134.2, 133.2 (d, *J* = 32.8 Hz), 126.7, 126.1 (q, *J* = 3.7 Hz), 125.8, 125.6, 124.0, 123.7 (d, *J* = 272.5 Hz), 118.2, 108.8. **HRMS (ESI)** m/z calcd for $C_{16}H_{10}F_3O_2^+$ $(M+H)^+$ 291.0627, found 291.0628.

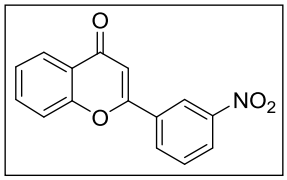


2-(3-chlorophenyl)-4H-chromen-4-one **2h**. General procedure **I**, 74% yield (190 mg), 8 h, brown solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.06 (dd, *J* = 8.01, 2.74 Hz, 1H), 7.72 (d, *J* = 13.92 Hz, 1H), 7.66 – 7.54 (m, 2H), 7.41 (t, *J* = 10.13 Hz, 1H), 7.37 – 7.25 (m, 3H), 6.62 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 177.8, 161.3, 155.8, 135.0, 133.8, 133.2, 131.3, 130.1, 126.0, 125.4, 125.2, 124.1, 123.6, 117.9, 107.8. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}ClO_2^+$ $(M+H)^+$ 257.0364, found 257.0365.

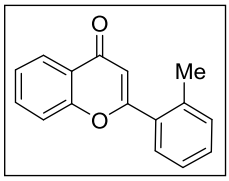


2-(3-bromophenyl)-4H-chromen-4-one **2i**. General procedure **I**, 93% yield (280 mg), 5 h, green solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.18 (dd, *J* = 7.94, 1.68 Hz, 1H), 8.01 (t, *J* = 1.86 Hz, 1H), 7.80 – 7.76 (m, 1H), 7.70 – 7.65 (m, 1H), 7.63 –

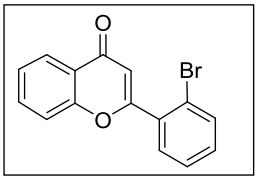
7.58 (m, 1H), 7.53 (d, *J* = 8.65 Hz, 1H), 7.41 – 7.38 (m, 1H), 7.35 (t, *J* = 7.98 Hz, 1H), 6.74 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.0, 161.4, 156.0, 134.3, 133.9, 133.6, 130.5, 129.1, 125.6, 125.4, 124.7, 123.8, 123.2, 118.0, 108.0. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}BrO_2^+$ $(M+H)^+$ 300.9859, found 300.9860.



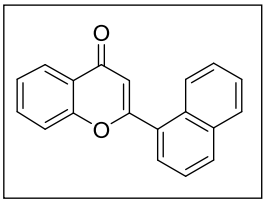
2-(3-nitrophenyl)-4H-chromen-4-one **2j**. General procedure **I**, 71% yield (190 mg), 10 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.81 (t, *J* = 2.02 Hz, 1H), 8.40 (ddd, *J* = 8.20, 2.32, 1.05 Hz, 1H), 8.28 – 8.20 (m, 1H), 7.81 – 7.71 (m, 1H), 7.64 (d, *J* = 8.38 Hz, 1H), 7.52 – 7.43 (m, 1H), 6.91 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.0, 160.5, 156.1, 148.8, 134.3, 133.7, 131.8, 130.3, 125.9, 125.9, 125.8, 123.9, 121.3, 118.2, 108.9. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}NO_4^+$ $(M+H)^+$ 268.0604, found 268.0606.



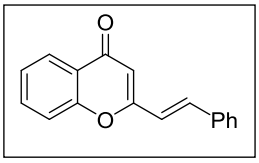
2-(o-tolyl)-4H-chromen-4-one **2k**. General procedure **I**, 92% yield (218 mg), 12 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.27 (dd, *J* = 7.94, 1.68 Hz, 1H), 7.74 – 7.65 (m, 1H), 7.55 – 7.48 (m, 2H), 7.46 – 7.40 (m, 2H), 7.33 (d, *J* = 7.42 Hz, 2H), 6.49 (s, 1H), 2.49 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.5, 166.2, 156.6, 137.0, 133.9, 131.4, 130.9, 129.4, 126.4, 125.9, 125.4, 124.0, 118.2, 112.1, 20.7. **HRMS (ESI)** m/z calcd for $C_{16}H_{13}O_2^+$ $(M+H)^+$ 237.0910, found 237.0905.



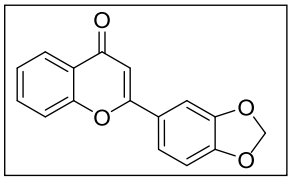
2-(2-bromophenyl)-4H-chromen-4-one **2l**. General procedure **I**, 90% yield (271 mg), 4 h, brown solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.24 (dd, *J* = 8.00, 1.68 Hz, 1H), 7.73 – 7.65 (m, 2H), 7.57 (dd, *J* = 7.62, 1.78 Hz, 1H), 7.50 (d, *J* = 8.95 Hz, 1H), 7.43 (q, *J* = 7.05 Hz, 2H), 7.36 (td, *J* = 7.74, 1.80 Hz, 1H), 6.57 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.1, 164.0, 156.6, 134.1, 134.0, 131.9, 130.9, 127.7, 125.8, 125.4, 123.9, 121.9, 112, 118.2.9. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}BrO_2^+$ $(M+H)^+$ 300.9859, found 300.9862.



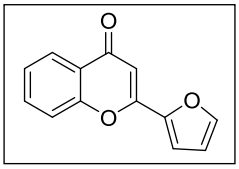
2-(naphthalen-1-yl)-4H-chromen-4-one **2m**. General procedure **I**, 89% yield (243 mg), 8 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.30 (d, *J* = 8.05 Hz, 1H), 8.16 – 8.07 (m, 1H), 7.98 (d, *J* = 8.20 Hz, 1H), 7.95 – 7.87 (m, 1H), 7.73 (d, *J* = 7.13 Hz, 1H), 7.67 (t, *J* = 7.73 Hz, 1H), 7.58 – 7.49 (m, 3H), 7.49 (d, *J* = 8.40 Hz, 1H), 7.43 (t, *J* = 7.50 Hz, 1H), 6.68 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.2, 165.4, 156.7, 133.9, 133.7, 131.5, 130.6, 130.4, 128.8, 128.0, 127.4, 126.6, 125.8, 125.4, 125.1, 124.8, 124.0, 118.2, 113.1. **HRMS (ESI)** m/z calcd for $C_{19}H_{13}O_2^+$ $(M+H)^+$ 273.0910, found 273.0910.



(E)-2-styryl-4H-chromen-4-one **2n**. General procedure **I**, 97% yield (242 mg), 6 h, brown solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.18 (dd, *J* = 7.95, 1.68 Hz, 1H), 7.68 – 7.63 (m, 1H), 7.60 – 7.54 (m, 3H), 7.50 (d, *J* = 7.45 Hz, 1H), 7.42 – 7.34 (m, 4H), 6.75 (d, *J* = 16.04 Hz, 1H), 6.30 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.5, 161.7, 156.0, 137.0, 135.0, 133.8, 129.9, 129.0, 127.7, 125.7, 125.0, 124.2, 120.3, 117.9, 110.7. **HRMS (ESI)** m/z calcd for $C_{17}H_{13}O_2^+$ $(M+H)^+$ 249.0910, found 249.0907.

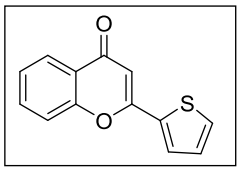


2-(benzo[d][1,3]dioxol-5-yl)-4H-chromen-4-one **2o**. General procedure **I**, 87% yield (232 mg), 12 h, green solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.21 (dd, *J* = 7.94, 1.68 Hz, 1H), 7.71 – 7.65 (m, 1H), 7.53 (d, *J* = 8.35 Hz, 1H), 7.49 (dd, *J* = 8.25, 1.87 Hz, 1H), 7.40 (t, *J* = 7.55 Hz, 1H), 7.36 (d, *J* = 1.82 Hz, 1H), 6.93 (d, *J* = 8.20 Hz, 1H), 6.70 (s, 1H), 6.07 (s, 2H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.5, 163.2, 156.2, 150.8, 148.6, 133.8, 125.8, 125.8, 125.3, 124.0, 121.6, 118.1, 108.9, 106.7, 106.4, 102.1. **HRMS (ESI)** m/z calcd for $C_{16}H_{11}O_4^+$ $(M+H)^+$ 267.0652, found 267.0653.

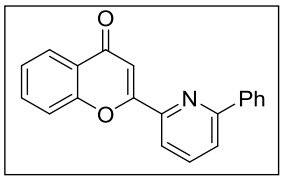


2-(furan-2-yl)-4H-chromen-4-one **2p**. General procedure **I**, 80% yield (170 mg), 14 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.11 (dd, *J* = 7.99, 1.70 Hz, 1H), 7.61 – 7.55 (m, 1H), 7.54 (d, *J* = 1.00 Hz, 1H), 7.38 (d, *J* = 8.44 Hz, 1H),

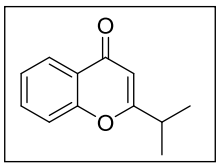
7.33 – 7.27 (m, 1H), 7.03 (d, *J* = 3.51 Hz, 1H), 6.62 (s, 1H), 6.52 (dd, *J* = 3.52, 1.74 Hz, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 177.6, 155.7, 155.0, 146.2, 145.8, 133.6, 125.6, 125.1, 124.1, 117.8, 113.1, 112.5, 105.3. **HRMS (ESI)** m/z calcd for $C_{13}H_9O_3^+$ $(M+H)^+$ 213.0546, found 213.0549.



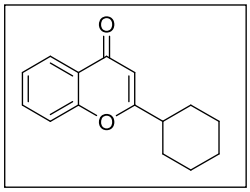
2-(thiophen-2-yl)-4H-chromen-4-one **2q**. General procedure **I**, 93% yield (213 mg), 14 h, yellow solid. (Petroleum ether/EtOAc = 10/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.13 (dd, *J* = 7.93, 1.67 Hz, 1H), 7.63 (dd, *J* = 3.83, 1.28 Hz, 1H), 7.61 – 7.56 (m, 1H), 7.50 (dd, *J* = 5.03, 1.24 Hz, 1H), 7.43 (d, *J* = 8.89 Hz, 1H), 7.36 – 7.29 (m, 1H), 7.10 (dd, *J* = 5.02, 3.78 Hz, 1H), 6.61 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 177.9, 159.0, 155.9, 135.1, 133.8, 130.3, 128.5, 128.5, 125.6, 125.3, 123.9, 117.9, 106.1. **HRMS (ESI)** m/z calcd for $C_{13}H_9O_2S^+$ $(M+H)^+$ 229.0318, found 229.0319.



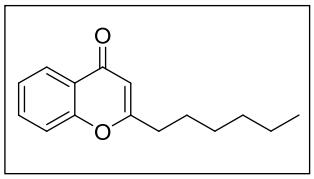
2-(6-phenylpyridin-2-yl)-4H-chromen-4-one **2r**. General procedure **I**, 82% yield (246 mg), 16 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.25 (d, *J* = 7.95 Hz, 1H), 8.13 (d, *J* = 1.81 Hz, 1H), 8.11 (s, 1H), 8.01 (d, *J* = 7.68 Hz, 1H), 7.92 (td, *J* = 7.75, 2.27 Hz, 1H), 7.86 (d, *J* = 7.82 Hz, 1H), 7.73 – 7.67 (m, 1H), 7.66 (s, 1H), 7.58 (d, *J* = 8.42 Hz, 1H), 7.53 – 7.45 (m, 3H), 7.42 (t, *J* = 7.41 Hz, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.9, 161.9, 157.3, 156.2, 149.0, 138.2, 138.0, 134.0, 129.8, 129.0, 127.0, 126.0, 125.4, 124.6, 122.1, 119.2, 118.3, 108.8. **HRMS (ESI)** m/z calcd for $C_{20}H_{14}NO_2^+$ $(M+H)^+$ 300.1019, found 300.1021.



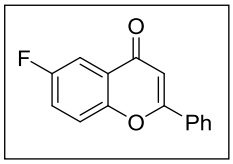
2-isopropyl-4H-chromen-4-one **2s**. General procedure **I**, 91% yield (172 mg), 3 h, brown liquid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.10 (dd, *J* = 7.91, 1.86 Hz, 1H), 7.59 – 7.54 (m, 1H), 7.36 (d, *J* = 8.43 Hz, 1H), 7.29 (t, *J* = 7.56 Hz, 1H), 6.12 (s, 1H), 2.79 (p, *J* = 6.92 Hz, 1H), 1.25 (d, *J* = 7.08 Hz, 6H). **^{13}C NMR (125 MHz, Chloroform-*d*)** δ 178.6, 174.1, 156.4, 133.4, 125.5, 124.8, 123.7, 117.8, 107.5, 33.2, 20.1. **HRMS (ESI)** m/z calcd for $C_{12}H_{13}O_2^+$ $(M+H)^+$ 189.0910, found 189.0910.



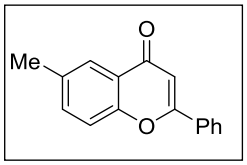
2-cyclohexyl-4H-chromen-4-one **2t**. General procedure **I**, 96% yield (220 mg), 4 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (500 MHz, Chloroform-*d*)** δ 8.11 (dd, *J* = 7.91, 1.83 Hz, 1H), 7.60 – 7.54 (m, 1H), 7.36 (d, *J* = 8.32 Hz, 1H), 7.29 (t, *J* = 7.48 Hz, 1H), 6.11 (s, 1H), 2.46 (tt, *J* = 11.60, 3.40 Hz, 1H), 1.97 (d, *J* = 11.70 Hz, 2H), 1.81 (d, *J* = 12.73 Hz, 2H), 1.69 (d, *J* = 12.23 Hz, 1H), 1.45 – 1.37 (m, 2H), 1.36 – 1.27 (m, 2H), 1.21 (tt, *J* = 12.49, 3.57 Hz, 1H). **^{13}C NMR (125 MHz, Chloroform-*d*)** δ 178.6, 173.4, 156.4, 133.4, 125.5, 124.7, 123.7, 117.8, 107.8, 42.7, 30.4, 25.8, 25.7. **HRMS (ESI)** m/z calcd for $C_{15}H_{17}O_2^+$ $(M+H)^+$ 229.1223, found 229.1225.



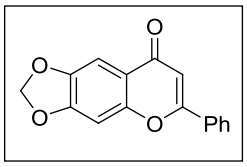
2-hexyl-4H-chromen-4-one **2u**. General procedure **I**, 88% yield (203 mg), 6 h, brown liquid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.10 (d, *J* = 7.86 Hz, 1H), 7.62 – 7.46 (m, 1H), 7.34 (d, *J* = 8.76 Hz, 1H), 7.31 – 7.26 (m, 1H), 6.10 (s, 1H), 2.53 (t, *J* = 7.55 Hz, 2H), 1.64 (d, *J* = 5.75 Hz, 2H), 1.31 (s, 2H), 1.24 (s, 4H), 0.82 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.2, 169.7, 156.4, 133.3, 125.5, 124.8, 123.6, 117.8, 109.6, 34.2, 31.4, 28.6, 26.6, 22.4, 14.0. **HRMS (ESI)** m/z calcd for $C_{15}H_{19}O_2^+$ $(M+H)^+$ 231.1380, found 231.1380.



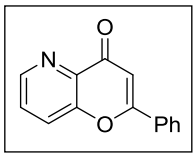
6-fluoro-2-phenyl-4H-chromen-4-one **2v**. General procedure **I**, 82% yield (198 mg), 14 h, brown solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.85 (dd, *J* = 7.88, 1.77 Hz, 2H), 7.80 (dd, *J* = 8.20, 3.10 Hz, 1H), 7.54 – 7.45 (m, 4H), 7.37 (ddd, *J* = 9.13, 7.63, 3.13 Hz, 1H), 6.75 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ = 177.5 (d, *J* =2.0 Hz), 163.6, 159.6 (d, *J* =245.0 Hz), 152.4 (d, *J* =2.0 Hz) , 131.8, 131.4, 129.10, 126.3, 125.1 (d, *J* =4.0 Hz), 121.9 (d, *J* =26.0 Hz), 120.2 (d, *J* =8.0 Hz), 110.6 (d, *J* =23.0 Hz), 106.8. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}FO_2^+$ $(M+H)^+$ 241.0659, found 241.0658.



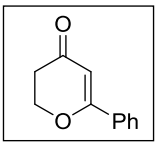
6-methyl-2-phenyl-4H-chromen-4-one **2w**. General procedure **I**, 91% yield (216 mg), 10 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.94 (s, 1H), 7.84 (d, *J* = 5.31 Hz, 2H), 7.45 (d, *J* = 7.34 Hz, 3H), 7.40 (t, *J* = 9.91 Hz, 2H), 6.73 (s, 1H), 2.39 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.5, 163.1, 154.5, 135.1, 135.0, 131.8, 131.5, 129.0, 126.2, 125.0, 123.6, 117.8, 107.3, 20.9. **HRMS (ESI)** m/z calcd for $C_{16}H_{13}O_2^+$ $(M+H)^+$ 237.0910, found 237.0904.



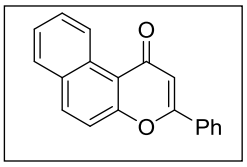
6-phenyl-8H-[1,3]dioxolo[4,5-g]chromen-8-one **2x.** General procedure **I**, 90% yield (260 mg), 10 h, yellow solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.97 (dd, *J* = 8.32, 1.41 Hz, 2H), 7.63 – 7.56 (m, 1H), 7.50 (t, *J* = 7.45 Hz, 2H), 7.39 (d, *J* = 0.96 Hz, 1H), 7.05 (s, 1H), 6.98 (s, 1H), 6.03 (s, 2H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 183.5, 152.6, 152.4, 150.0, 146.0, 137.6, 132.7, 129.3, 128.6, 120.7, 117.7, 102.1, 100.3, 93.9. **HRMS (ESI)** m/z calcd for $C_{16}H_{10}O_4Na^+$ $(M+Na)^+$ 289.0471, found 289.0472.



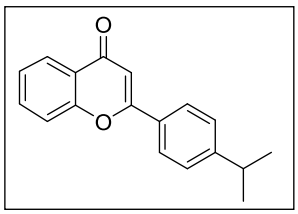
2-phenyl-4H-pyrano[3,2-b]pyridin-4-one **2y.** General procedure **I**, 92% yield (206 mg), 8 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.64 (dd, *J* = 4.62, 1.34 Hz, 1H), 8.01 (s, 1H), 7.99 (d, *J* = 1.51 Hz, 1H), 7.89 (dt, *J* = 8.55, 1.22 Hz, 1H), 7.64 (d, *J* = 0.98 Hz, 1H), 7.63 – 7.58 (m, 1H), 7.50 (t, *J* = 7.68 Hz, 2H), 7.37 (dd, *J* = 8.50, 4.63 Hz, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 184.2, 154.4, 149.4, 148.0, 146.2, 136.6, 133.4, 129.5, 128.7, 122.4, 119.9, 116.6. **HRMS (ESI)** m/z calcd for $C_{14}H_{10}NO_2^+$ $(M+H)^+$ 224.0706, found 224.0708.



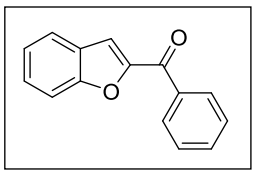
6-phenyl-2,3-dihydro-4H-pyran-4-one **2z.** General procedure **I**, 57% yield (100 mg), 24 h, brown solid. (Petroleum ether/EtOAc = 2/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.67 (d, *J* = 7.34 Hz, 2H), 7.46 – 7.40 (m, 1H), 7.36 (t, *J* = 7.35 Hz, 2H), 5.97 (s, 1H), 4.58 (t, *J* = 6.68 Hz, 2H), 2.63 – 2.54 (m, 2H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 192.5, 170.3, 132.5, 131.6, 128.6, 126.4, 102.3, 68.1, 35.9. **HRMS (ESI)** m/z calcd for $C_{11}H_{11}O_2^+$ $(M+H)^+$ 175.0754, found 175.0754.



3-phenyl-1H-benzo[f]chromen-1-one **2aa.** General procedure **I**, 66% yield (195 mg), 14 h, yellow solid. (Petroleum ether/EtOAc = 2/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 10.03 (d, *J* = 8.08 Hz, 1H), 7.98 (d, *J* = 9.03 Hz, 1H), 7.86 (dd, *J* = 6.71, 3.00 Hz, 2H), 7.80 (d, *J* = 8.06 Hz, 1H), 7.73 – 7.66 (m, 1H), 7.57 – 7.51 (m, 1H), 7.48 (dd, *J* = 5.82, 3.35 Hz, 4H), 6.88 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 180.1, 160.6, 157.2, 135.4, 131.3, 131.3, 130.5, 130.4, 129.1, 129.0, 128.1, 127.1, 126.5, 126.0, 117.5, 117.1, 110.3. **HRMS (ESI)** m/z calcd for $C_{19}H_{12}O_2Na^+$ $(M+Na)^+$ 295.0730, found 295.0730.



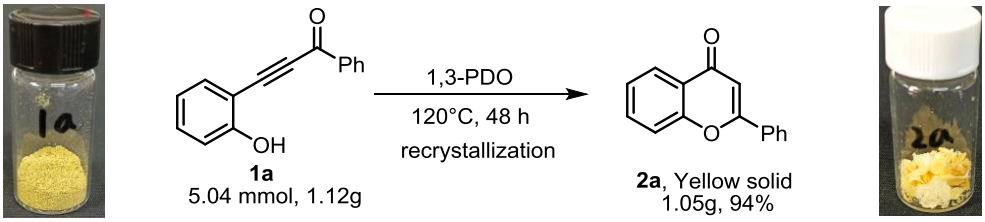
MN-64 **2ab**. General procedure **I**, 92% yield (243 mg), 14 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.23 (dd, *J* = 7.92, 1.65 Hz, 1H), 7.88 – 7.84 (m, 2H), 7.72 – 7.67 (m, 1H), 7.57 (d, *J* = 7.77 Hz, 1H), 7.44 – 7.40 (m, 1H), 7.40 – 7.37 (m, 2H), 6.81 (s, 1H), 3.00 (hept, *J* = 6.91 Hz, 1H), 1.30 (d, *J* = 6.93 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.6, 163.8, 156.4, 153.2, 133.8, 129.4, 127.3, 126.5, 125.8, 125.3, 124.1, 118.2, 107.2, 34.3, 23.9. **HRMS (ESI)** m/z calcd for $C_{18}H_{16}O_2Na^+$ $(M+Na)^+$ 287.1043, found 287.1041.



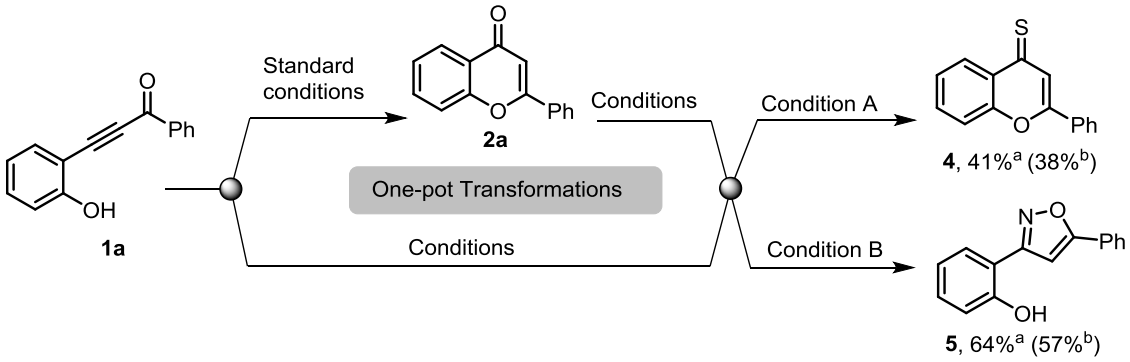
benzofuran-2-yl(phenyl)methanone **3a.** General procedure **I**, 9% yield (20 mg), 12 h, yellow solid. (Petroleum ether/EtOAc = 10/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.02 (d, *J* = 8.01 Hz, 2H), 7.70 (d, *J* = 7.99 Hz, 1H), 7.62 (d, *J* = 7.69 Hz, 2H), 7.56 – 7.44 (m, 4H), 7.31 (t, *J* = 7.58 Hz, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 184.4, 156.0, 152.2, 137.2, 132.9, 129.5, 128.6, 128.4, 127.0, 124.0, 123.4, 116.6, 112.6. **HRMS (ESI)** m/z calcd for $C_{15}H_{11}O_2^+$ $(M+H)^+$ 223.0754, found 223.0754.

5. Applications

General procedure I: the Gram-Scale Reaction.



The substrate **1a** (5.04 mmol, 1 equiv.) was added in a bottle along with the 10 mL of 1,3-PDO. The reaction was carried out at 120 ℃ in oil bath for 48 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered and evaporated under reduced pressure to give the crude product, and finally purified by direct recrystallization (ethyl acetate : petroleum ether = 1 : 3, 65 ℃, in oil bath, 6 mL solvent) to get the pure product **2a** (1.05 g, 4.72 mmol, 94%, yellow solid).



General procedure II: The procedure for synthesizing **4**, **5.**

Condition **A**:

Method 1: The substrate **1a** (1 mmol, 1 equiv.) was added in a bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 120 ℃ in oil bath for 3 hours. After the first reaction is completed, the Lawesson's reagent (2 mmol, 2 equiv.) was added to the reaction solution. The reaction continued in a 150 ℃ oil bath for 18 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 10) to obtain the pure product **4** (98 mg, 0.41 mmol, 41%, red solid).

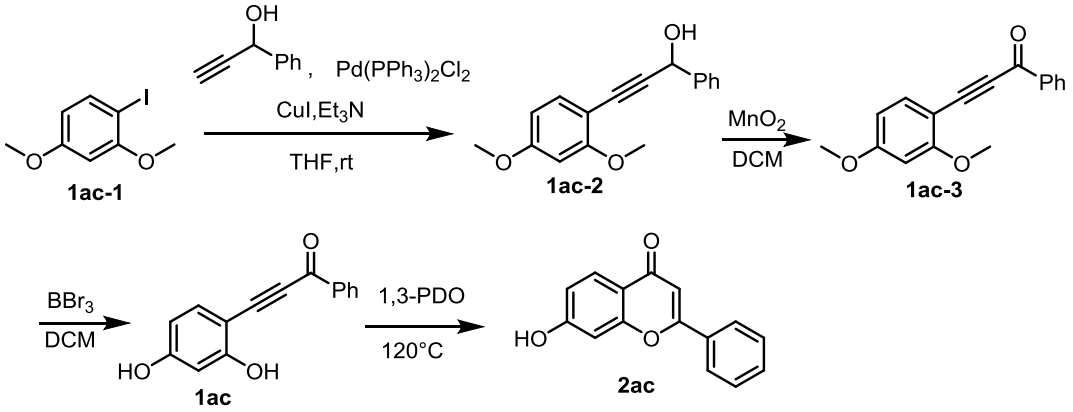
Method 2: The substrate **1a** (1 mmol, 1 equiv.) and the Lawesson's reagent (2 mmol, 2 equiv.) was added in a bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 150 ℃ in oil bath for 18 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 10) to obtain the pure product **4** (91 mg, 0.38 mmol, 38%, red solid).

Condition **B**:

Method 1: The substrate **1a** (1 mmol, 1 equiv.) was added in a bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 120 ℃ in oil bath for 3 hours. After the first reaction is completed, the hydroxylamine hydrochloride (3 mmol, 3 equiv.) was added to the reaction solution. The reaction continued in at 100 ℃ in oil bath for 18 hours. The reaction is monitored by TLC. After the reaction was finished, filtered, washed the filtrate with EtOAc (3 × 5 mL) and H_2O (3 × 5 mL), collect solids to obtain the pure product **5** (152 mg, 0.57 mmol, 64%, white solid).

Method 2: The substrate **1a** (1 mmol, 1 equiv.) and the hydroxylamine hydrochloride (3 mmol, 3 equiv.) was added in a bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 100 ℃ in oil bath for 18 hours. The reaction is monitored by TLC. After the reaction was finished, filtered, washed the filtrate with EtOAc (3 × 5 mL) and H_2O (3 × 5 mL), collect solids to obtain the pure product **5** (135 mg, 0.57 mmol, 57%, white solid).

General procedure III: The procedure for synthesizing compound **1ab**, **2ab**, **6**, **7**.



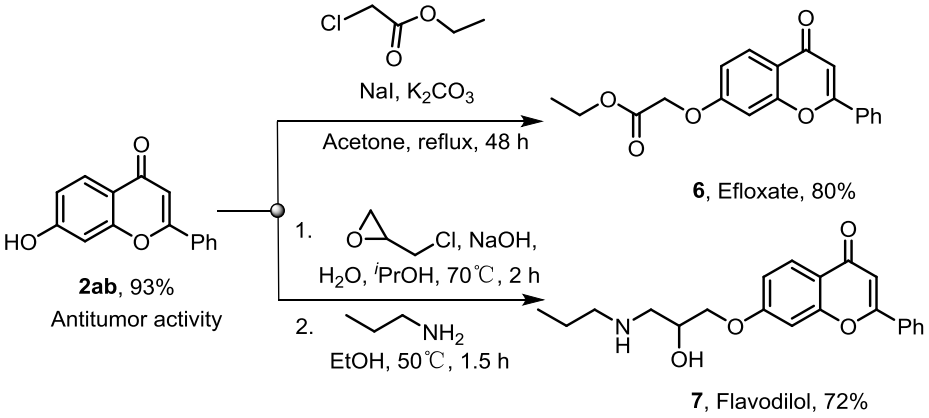
To an anhydrous flask was added $Pd(PPh_3)_2Cl_2$ (70.2 mg, 0.1 mmol, 2 mol%) and CuI (38 mg, 0.2 mmol, 4 mol%), then the mixture was degassed and flushed with N_2 for three times at room temperature. Et_3N (2.02 g, 20 mmol, 4.0 equiv.), **1ac-1** (1.32 g, 5 mmol, 1.0 equiv.) and propargylic alcohols (7.5 mmol, 1.5 equiv.) were added successively and the mixture was allowed to stirred at room temperature for 2 hours. Upon completion, the mixture was quenched with saturated NH_4Cl solution (20 mL), extracted with EtOAc (3 × 10 mL), washed with brine, dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 1) to get the product **1ac-2** (1.06 g, 4.0 mmol, 79%, oily liquid).

The substrate **1ac-2** (1.08 g, 4.0 mmol, 1.0 equiv.) was dissolved into DCM (12 mL) at room temperature, followed by MnO_2 (3.78 g, 32.0 mmol, 8.0 equiv.) was added. The resulting mixture was stirred at room temperature for 2 hours. After completion, removed MnO_2 through filtration, and it was washed with DCM (3 × 10 mL). Then concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 2) to get the product **1ac-3** (852 mg, 3.2 mmol, 80%, white solid).

Dissolve **1ac-3** (1.07 g, 4 mmol, 1eq.) in 100 mL of DCM. Bubble argon for 20 min to remove the oxygen. Add BBr_3 (8.1 g, 32 mmol, 4 eq.) drop by drop to the mixture after cooling down to -78 ℃ for 30 min. Keep the solution at -78 ℃ for 30 min. Warm the solution slowly to room temperature. Add ice water to quench reaction mixture after reacted for 12 hours. Filter the mixture. Washed the filtrate with DCM (3 × 5 mL) and collect solids to obtain the product **1ac** (667 mg, 2.8 mmol, 70%, red solid).

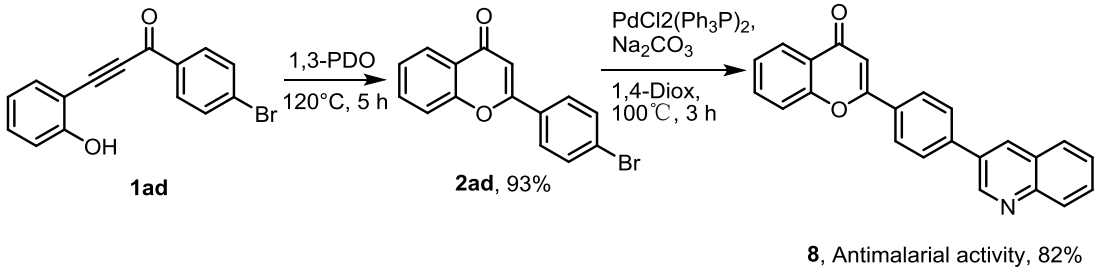
The substrate **1ac** (1 mmol, 1 equiv.) was added in an anhydrous bottle along with the 2 mL of

1,3-PDO. The reaction was carried out at 120 ℃ in oil bath for 8 hours. The reaction is monitored by TLC. After the reaction was finished, filtered, washed the filtrate with EtOAc (3 × 5 mL) and H_2O (3 × 5 mL), collect solids to obtain the crude product the product **2ac** (222 mg, 0.93 mmol, 93%, pink solid).



The products **6** and **7** were prepared according to the reported method.[7, 8]

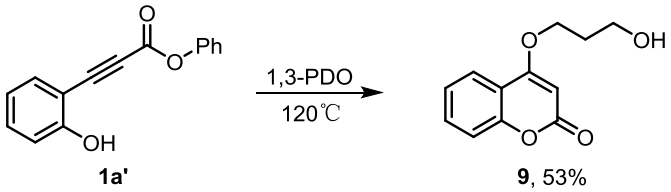
General procedure IV: The procedure for synthesizing compound **2ad**, **8**.



The substrate **1ad** (1 mmol, 1 equiv.) was added in an anhydrous bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 120 ℃ in oil bath. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3×4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 2) to obtain the pure product **2ad** (280 mg, 0.93 mmol, 93%, yellow solid).

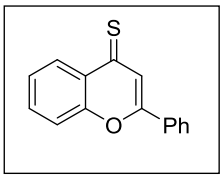
the product **8** were prepared according to the reported method.[9]

General procedure V: The procedure for synthesizing **9**.

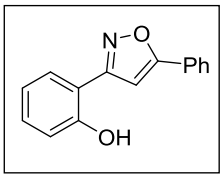


The substrate **1a'** (1 mmol, 1 equiv.) was added in a bottle along with the 2 mL of 1,3-PDO. The reaction was carried out at 120 ℃ in oil bath for 14 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3×4 mL), merged organic phases and dried over

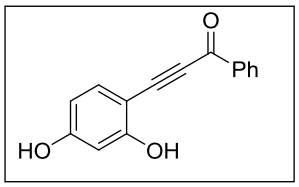
Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 1) to obtain the pure product **9** (117 mg, 0.9 mmol, 53%, yellow solid).



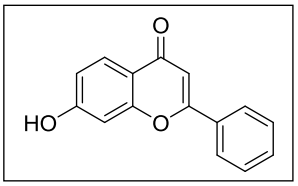
2-phenyl-4H-chromene-4-thione **4**. General procedure **II**, condition **A**, method 1, 41% yield (98 mg), 18 h, red solid. (Petroleum ether/EtOAc =10/1, Rf = 0.5). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.52 (dd, *J* = 8.15, 1.66 Hz, 1H), 7.89 (dd, *J* = 8.22, 1.60 Hz, 2H), 7.69 (s, 1H), 7.70 – 7.60 (m, 1H), 7.52 – 7.40 (m, 2H), 7.39 – 7.30 (m, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 202.0, 154.0, 151.4, 134.1, 131.8, 130.9, 129.8, 129.1, 128.5, 126.4, 126.1, 120.2, 118.4. **HRMS (ESI)** m/z calcd for $C_{15}H_{11}OS^+$ $(M+H)^+$ 239.0525, found 239.0526.



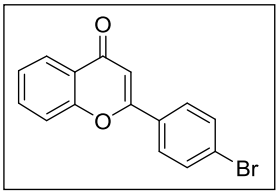
2-(5-Phenylisoxazol-3-yl)phenol **5.** General procedure **II**, condition **B**, method 1, 64% yield (152 mg), 18 h, white solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.50). **^{1}H NMR (400 MHz, DMSO-d_6)** δ 10.68 (s, 1H), 7.94 (dd, *J* = 7.57, 2.06 Hz, 2H), 7.84 (dd, *J* = 7.82, 1.72 Hz, 1H), 7.55 – 7.49 (m, 3H), 7.38 – 7.32 (m, 2H), 7.10 (d, *J* = 8.01 Hz, 1H), 7.01 – 6.95 (m, 1H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 166.8, 162.4, 154.9, 131.5, 130.1, 129.1, 128.9, 126.9, 126.7, 119.5, 116.6, 113.9, 100.9. **HRMS (ESI)** m/z calcd for $C_{15}H_{12}NO_2^+$ $(M+H)^+$ 238.0863, found 238.0863.



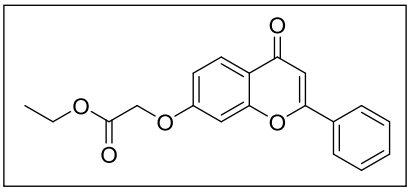
3-(2,4-dihydroxyphenyl)-1-phenylprop-2-yn-1-one **1ac.** General procedure **III**. 79% yield (564 mg), red solid. (Petroleum ether/EtOAc = 1/1, Rf = 0.20). **^{1}H NMR (400 MHz, DMSO-d_6)** δ 7.98 (d, *J* = 7.94 Hz, 2H), 7.84 (d, *J* = 8.67 Hz, 1H), 7.50 (d, *J* = 6.61 Hz, 3H), 7.00 (s, 1H), 6.92 (dd, *J* = 8.67, 2.25 Hz, 1H), 6.82 (s, 1H), 6.33 (s, 1H). **^{13}C NMR (100 MHz, DMSO-d_6)** δ 176.7, 163.0, 162.2, 157.7, 131.7, 131.4, 129.3, 126.7, 126.3, 116.3, 115.3, 106.7, 102.8. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}O_3Na^+$ $(M+Na)^+$ 261.0522, found 261.0522.



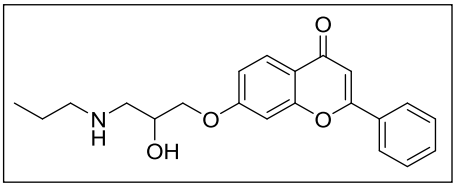
7-hydroxy-2-phenyl-4H-chromen-4-one **2ac.** General procedure **III**, 93% yield (222 mg), 8 h, pink solid. (Petroleum ether/EtOAc = 1/1, Rf = 0.20). **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 10.83 (s, 1H), 8.02 (dd, *J* = 7.58, 2.12 Hz, 2H), 7.89 (d, *J* = 8.69 Hz, 1H), 7.57 – 7.51 (m, 5H), 6.99 (d, *J* = 2.24 Hz, 1H), 6.93 (dd, *J* = 8.66, 2.27 Hz, 1H), 6.87 (s, 1H). **^{13}C NMR (100 MHz, DMSO-*d*$_6$)** δ 176.4, 162.8, 161.9, 157.5, 131.5, 131.3, 129.1, 126.6, 126.2, 116.2, 115.1, 106.7, 102.6. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}O_3Na^+$ $(M+Na)^+$ 261.0522, found 261.0522.



2-(4-bromophenyl)-4H-chromen-4-one **2ad**. General procedure **IV**, 93% yield (280 mg), 5 h, green solid. (Petroleum ether/EtOAc = 5/1, Rf = 0.20). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.16 (dd, *J* = 7.98, 1.68 Hz, 1H), 7.75 – 7.68 (m, 2H), 7.68 – 7.62 (m, 1H), 7.62 – 7.53 (m, 2H), 7.49 (d, *J* = 7.88 Hz, 1H), 7.43 – 7.31 (m, 1H), 6.73 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.2, 162.2, 156.1, 133.9, 132.3, 130.6, 127.7, 126.3, 125.7, 125.4, 123.9, 118.1, 107.6. **HRMS (ESI)** m/z calcd for $C_{15}H_{10}BrO_2^+$ $(M+H)^+$ 300.9859, found 300.9860.

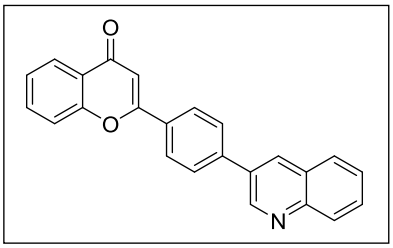


Flavodilol **6**. General procedure **III**, 80% yield (260 mg), 16 h, yellow solid. (Petroleum ether/EtOAc = 2/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.11 (d, *J* = 8.81 Hz, 1H), 7.84 (dd, *J* = 7.54, 2.16 Hz, 2H), 7.47 (d, *J* = 6.64 Hz, 3H), 6.98 (dd, *J* = 8.82, 2.38 Hz, 1H), 6.91 (d, *J* = 2.36 Hz, 1H), 6.71 (s, 1H), 4.72 (s, 2H), 4.28 (q, *J* = 7.13 Hz, 2H), 1.29 (t, *J* = 7.10 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 177.7, 168.0, 163.2, 162.2, 157.7, 131.7, 131.5, 129.0, 127.4, 126.2, 118.6, 114.3, 107.6, 101.7, 65.5, 61.8, 14.2. **HRMS (ESI)** m/z calcd for $C_{19}H_{17}O_5^+$ $(M+H)^+$ 325.1071, found 325.1071.

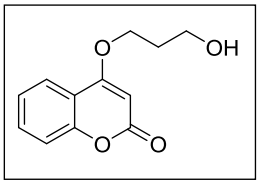


Efloxate **7**. General procedure **III**, 72% yield (255 mg), 16 h, white solid. (Dichloromethane/ methanol = 10/1, Rf = 0.20). **^{1}H NMR (400 MHz, DMSO-*d*$_6$)** δ 8.09 (d, *J* = 6.14 Hz, 2H), 7.94 (d, *J* = 8.77 Hz, 1H), 7.58 (d, *J* = 7.00 Hz, 3H), 7.33 (s, 1H), 7.07 (d, *J* = 8.87 Hz, 1H), 6.95 (s, 1H),

5.21 (s, 1H), 4.16 (dd, *J* = 9.98, 4.21 Hz, 1H), 4.11 – 4.02 (m, 1H), 3.99 – 3.91 (m, 1H), 3.40 (s, 0H), 2.68 (dd, *J* = 11.82, 5.24 Hz, 1H), 2.62 (dd, *J* = 11.87, 6.50 Hz, 1H), 1.43 (q, *J* = 7.26 Hz, 2H), 0.87 (t, *J* = 7.37 Hz, 3H). **^{13}C NMR (100 MHz, DMSO-*d*$_6$)** δ 176.4, 163.4, 162.1, 157.5, 131.6, 131.2, 129.1, 126.2, 126.1, 117.0, 115.1, 106.7, 101.4, 71.7, 67.9, 52.3, 51.5, 22.8, 11.8. **HRMS (ESI)** m/z calcd for $C_{21}H_{24}NO_4^+$ $(M+H)^+$ 354.1700, found 354.1700.



2-(4-(quinolin-3-yl)phenyl)-4H-chromen-4-one **8.** General procedure **IV**, 82% yield (287 mg), 12 h, yellow solid. (Petroleum ether/EtOAc =2/1, Rf = 0.2). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 9.16 (d, *J* = 2.31 Hz, 1H), 8.29 (d, *J* = 2.32 Hz, 1H), 8.18 (dd, *J* = 7.96, 1.67 Hz, 1H), 8.11 (d, *J* = 8.44 Hz, 1H), 7.98 (d, *J* = 8.44 Hz, 2H), 7.85 (d, *J* = 8.17 Hz, 1H), 7.79 (d, *J* = 8.46 Hz, 2H), 7.75 – 7.63 (m, 1H), 7.59 – 7.50 (m, 1H), 7.37 (t, *J* = 7.48 Hz, 1H), 6.82 (s, 1H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 178.3, 162.6, 156.2, 149.3, 147.7, 140.9, 133.9, 133.5, 132.3, 131.3, 130.0, 129.3, 128.2, 127.8, 127.8, 127.3, 127.0, 125.7, 125.3, 124.0, 118.1, 107.7. **HRMS (ESI)** m/z calcd for $C_{24}H_{16}NO_2^+$ $(M+H)^+$ 350.1176, found 350.1176.



3-(3-hydroxypropoxy)-2H-chromen-2-one **9**. General procedure **V**, 53% yield (117 mg), 14 h, yellow solid. (Petroleum ether/EtOAc = 2/1, Rf = 0.50). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.74 (dd, *J* = 8.31, 1.65 Hz, 1H), 7.51 – 7.42 (m, 1H), 7.20 (t, *J* = 7.37 Hz, 2H), 5.66 (s, 1H), 4.27 (t, *J* = 6.20 Hz, 2H), 3.91 (t, *J* = 5.96 Hz, 2H), 3.66 (s, 1H), 2.16 (p, *J* = 6.06 Hz, 2H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 165.9, 163.5, 153.0, 132.4, 124.0, 122.9, 116.5, 115.5, 90.2, 66.3, 58.4, 31.4. **HRMS (ESI)** m/z calcd for $C_{12}H_{12}O_4Na^+$ $(M+Na)^+$ 243.0628, found 243.0628.

General procedure VI: Biological activity evaluation

A. Anti-inflammatory performance

0.05 mL of compound solutions(**2a-2ad**, **4-9**; 200, 100, 50, 25, 12.5 μg/mL) were mixed with 0.45 mL of 1.0% BSA solution (PBS, pH = 6.3) and recorded as the experimental group. At the same time, 0.45 mL of 1.0% BSA solution was mixed with 0.05 mL of diclofenac sodium solution (100 μg/mL, DS) and recorded as the positive control group. The experimental and control groups were incubated in an incubator at 37 ℃ for 20 min, then the incubator temperature was gradually increased to 70 ℃ and incubated for 20 min. After the incubator temperature had dropped to room temperature, approximately 2.5 mL of PBS solution was added to the reaction mixture and the absorbance values at 660 nm for each group were measured using a spectrophotometer. Where A blank is the absorbance of the control reaction (containing all reagents except the test compound), and A sample is the absorbance of the test sample. Dose-response curve was plotted between %inhibition of albumin denaturation and the drug concentration. Linear regression analysis was carried out for calculating drug concentration showing 50% inhibition of albumin denaturation (IC_{50}). Diclofenac sodium as positive control and all tests were undertaken on three replicates and the results were averaged. The IC_{50} value of DS is 7 μg/mL.

Table 2 In vitro anti‑inflammatory of compounds

Compounds	IC_{50} (μg/mL) ± SD	Compounds	IC_{50} (μg/mL) ± SD
2a	41.18 ± 0.42	**2s**	8.33 ± 0.09
2b	11.33 ± 0.28	**2t**	9.33 ± 0.07
2c	49.70 ± 0.19	**2u**	14.78 ± 0.44
2d	5.67 ± 0.16	**2v**	34.36 ± 0.26
2e	28.75 ± 0.24	**2w**	20.30 ± 0.16
2f	40.30 ± 0.16	**2x**	45.67 ± 0.15
2g	22.78 ± 0.12	**2y**	18.00 ± 0.12
2h	>50	**2z**	36.10 ± 0.08
2i	27.10 ± 0.24	**2aa**	>50
2j	>50	**2ab**	28.00 ± 0.22
2k	6.33 ± 0.40	**2ac**	>50
2l	31.09 ± 0.09	**2ad**	43.09 ± 0.39
2m	16.90 ± 0.14	**4**	>50
2n	40.50 ± 0.23	**5**	–
2o	16.40 ± 0.19	**6**	16.10 ± 0.17
2p	>50	**7**	17.80 ± 0.30
2q	18.89 ± 0.09	**8**	>50
2r	37.36 ± 0.15	**9**	13.90 ± 0.09

B. MTT cell viability assay

The in vitro biocompatibility of different skeleton compounds (**2d**, **2y** and **9**) was determined using human umbilical vein endothelial cells (HUVECs) as a model. HUVECs were cultured in high glucose DMEM medium containing 10% fetal bovine serum (FBS, Gibco, USA) and 1% penicillin-streptomycin (Invitrogen, USA), followed by addition of different concentrations of **2d**, **2y** or **9** compounds and co-incubation in a humidified incubator containing 5% CO_2 (12.5, 25, 50, 100 and 200 μg/mL concentrations). After 24 hours, MTT staining was performed, and then DMSO was used to dissolve the purple compound and measure the absorption at 490 nm.

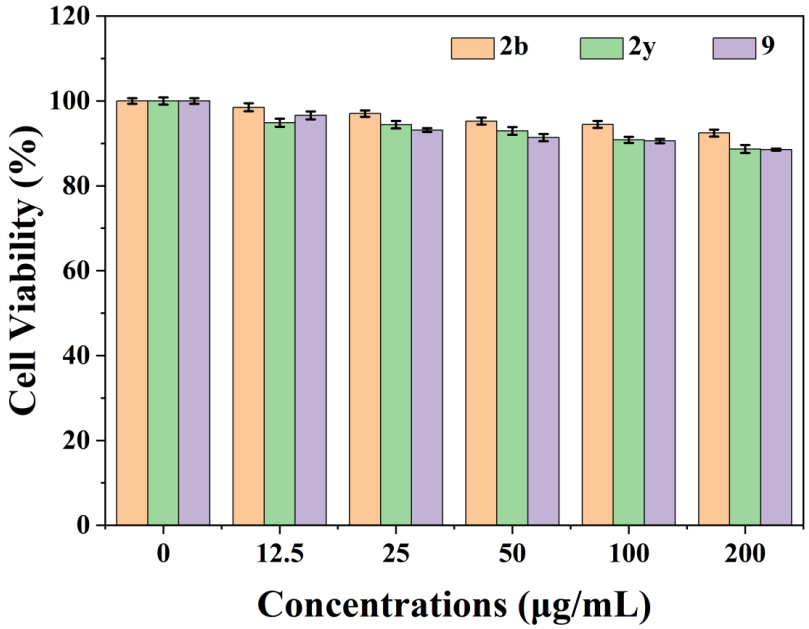
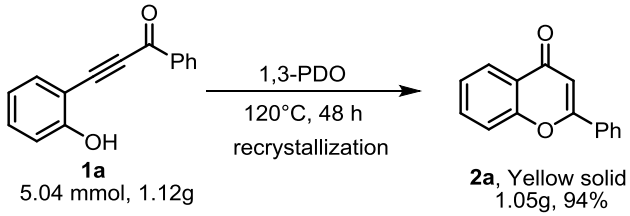


Figure 1. Toxicity of compounds **2d**, **2y** and **9** on HUVECs

6. Evaluation of Green Chemistry Metrics for the Synthesis

The most important and widely used parameters to calculate the "green" character of a reaction are: ideally, atom efficiency and carbon efficiency should approach to 100% while the E-factor should be as low as possible. Typical E-factors for the production of fine chemicals and pharmaceuticals in industry are in the range of 5-50 and 25-100, respectively.[10] The following formula were used for calculating atom economy (AE), atom efficiency (AEf), carbon efficiency (CE), reaction mass efficiency (RME) and E-factor.



Yield of desired product (**2a**) = 94%

$$\text{Atom Economy (\%)} = \frac{\text{mass of desired product}}{\text{total mass of all reactants}} \times 100 = \frac{222.24}{222.24} \times 100 = 100\%$$

$$\text{Atom Efficiency (\%)} = (\%\ \text{yield of product} \times \%\ \text{atom economy}) \times 100 = (94\% \times 100\%) \times 100 = 94\%$$

$$\text{Carbon Efficiency (\%)} = \frac{\text{amount of carbon in desired product}}{\text{total amount of carbon presented in all reactants}} \times 100 = \frac{15}{15} \times 100 = 100\%$$

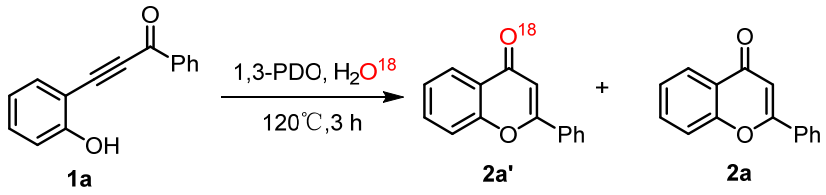
$$\text{Reaction Mass Efficiency (\%)} = \frac{\text{mass of isolated product}}{\text{mass of all reactants}} \times 100 = \frac{1.12}{1.05} \times 100 = 94\%$$

Reactant	3-(2-hydroxyphenyl)-1-phenylprop-2-yn-1-one	1.12 g	5.0 mmol	FW 222.24
Solvent	1,3-propanediol	12 g	157.7 mmol	FW 76.1
Product	2-phenyl-4H-chromen-4-one	1.05 g	4.7 mmol	FW 222.24
Extract				
Solvent	ethyl acetate	10 g	113.5 mmol	FW 88.11
Desiccant	Na_2SO_4	2 g	14.1 mmol	FW 142
Detergent	ethyl acetate	5 g	56.7 mmol	FW 88.11
Recrystallization				
Solvent	ethyl acetate	3.6 g	40.9 mmol	FW 88.11
	petroleum ether	1.0 g	63.16 mmol	FW 158.32
Detergent	petroleum ether	3.3 g	20.84 mmol	FW158.32

$$\text{E - factor} = \frac{\text{total waste (kg)}}{\text{total product (kg)}} = \frac{(1.12+12+10+2+5+3.6+1.0+3.3)-1.05}{1.05} = 35.2\ \text{kg/kg product}$$

7. The Mechanism Studies

A. General procedures for compound 2a'.



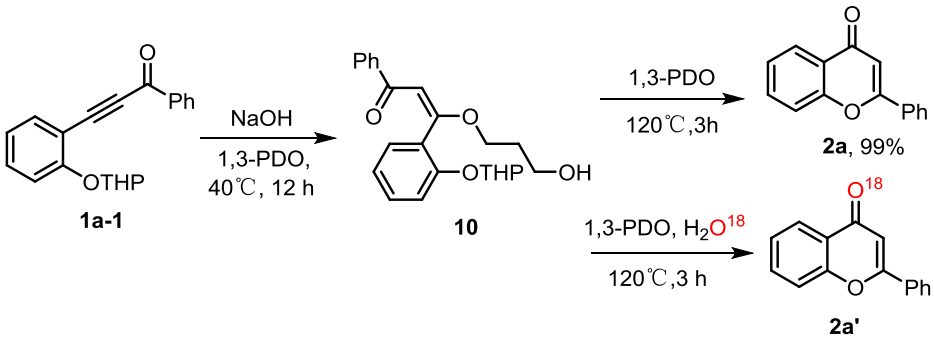
1a **2a'** **2a**

HRMS (225.0797) **(2a' : 2a = 5 : 1)**

Ynone **1a** (290 mg, 1 mmol, 1 equiv.) was added in a dry reaction tube with 1,3-PDO (2 mL), then H_2O^{18} (2 mmol, 2 equiv.) was added. The reaction was carried out at 120 ℃ in oil bath for 5 hours. The reaction is monitored by TLC and $FeCl_3$ solution. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 5) to obtain the the mixture **2a'** and **2a**.

B. General procedures for compound 10, 2a'.

B. General procedures for compound 10, 2a'.

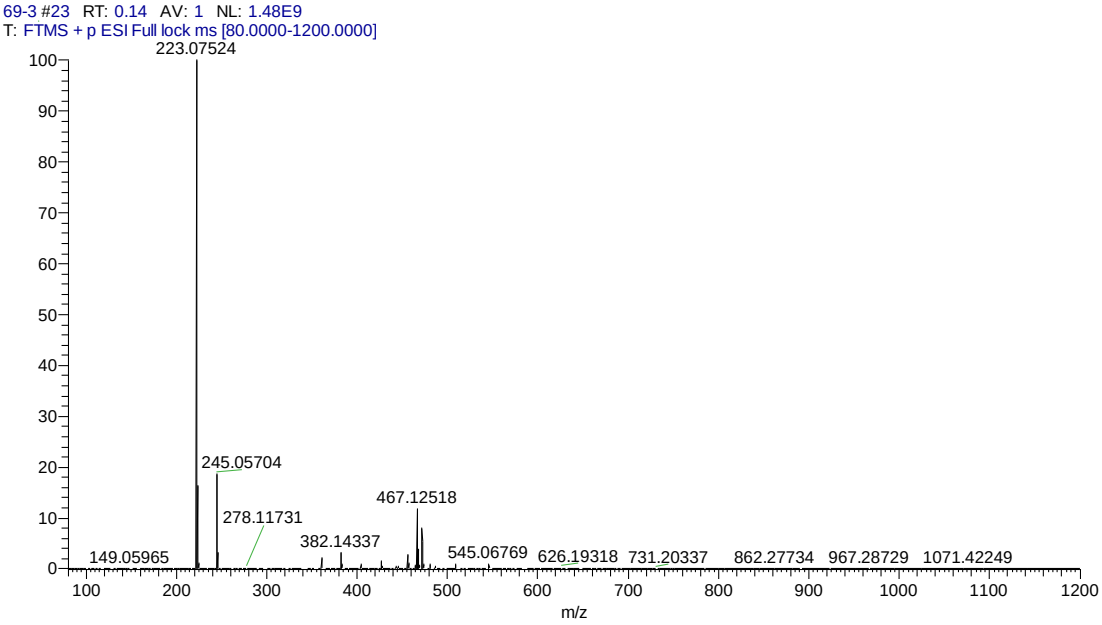


Ynone **1a-1** (306 mg, 1 mmol, 1 equiv.) was added in a dry reaction tube with 1,3-PDO (3 mL), then NaOH (1 mmol, 1 equiv.) was added. The reaction was carried out at 40 ℃ in oil bath for 16 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by silica gel column chromatography (ethyl acetate : petroleum ether = 1 : 1) to obtain the pure product **10** (287 mg, 0.75 mmol, 75%, oily liquid).

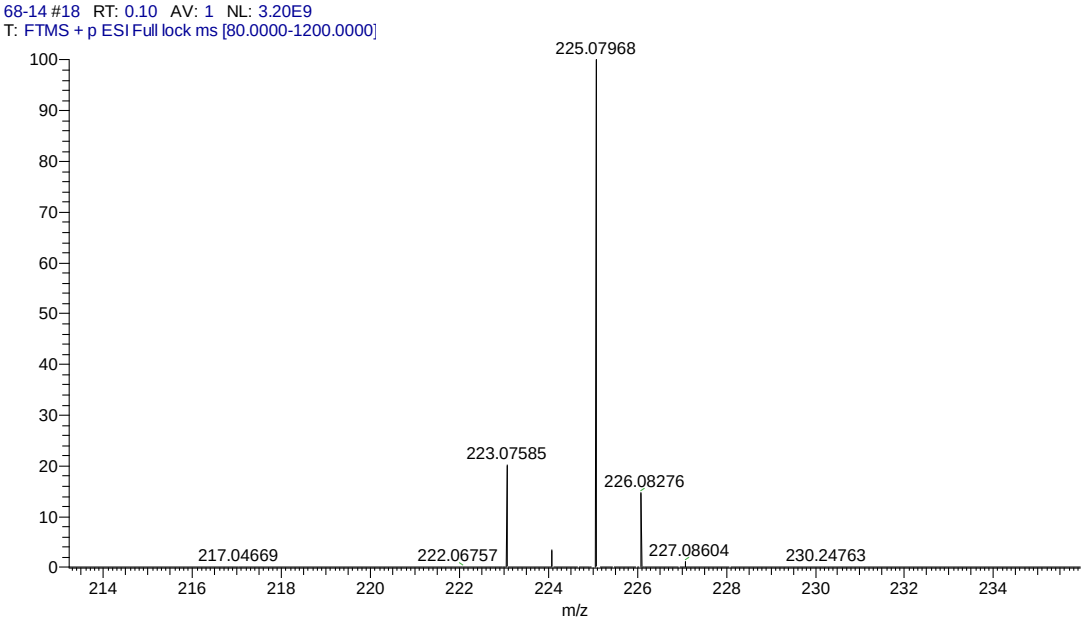
The intermediate **10** (382 mg, 1 mmol, 1 equiv.) was added in a dry reaction tube with 1,3-PDO (3 mL), then H_2O^{18} (2 mmol, 2 equiv.) was added. The reaction was carried out at 120 ℃ in oil bath for 3 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by direct recrystallization (ethyl acetate : petroleum ether = 1 : 3, 65 ℃, in oil bath, 6 mL solvent) to get the pure product **2a** (221 mg, 0.99 mmol, 99%, yellow solid).

The intermediate **10** (382 mg, 1 mmol, 1 equiv.) was added in a dry reaction tube with 1,3-PDO (3 mL), then H_2O^{18} (2 mmol, 2 equiv.) was added. The reaction was carried out at 120 ℃ in oil bath for 3 hours. The reaction is monitored by TLC. After the reaction was finished, distilled water (10 mL) was added to quench the reaction and the reaction mixture was extracted with EtOAc (3 × 4 mL), merged organic phases and dried over Na_2SO_4, filtered, and concentrated to get the crude product which was purified by direct recrystallization (ethyl acetate : petroleum ether = 1 : 3, 65 ℃, in oil bath, 6 mL solvent) to get the mixture **2a'** and **2a**.

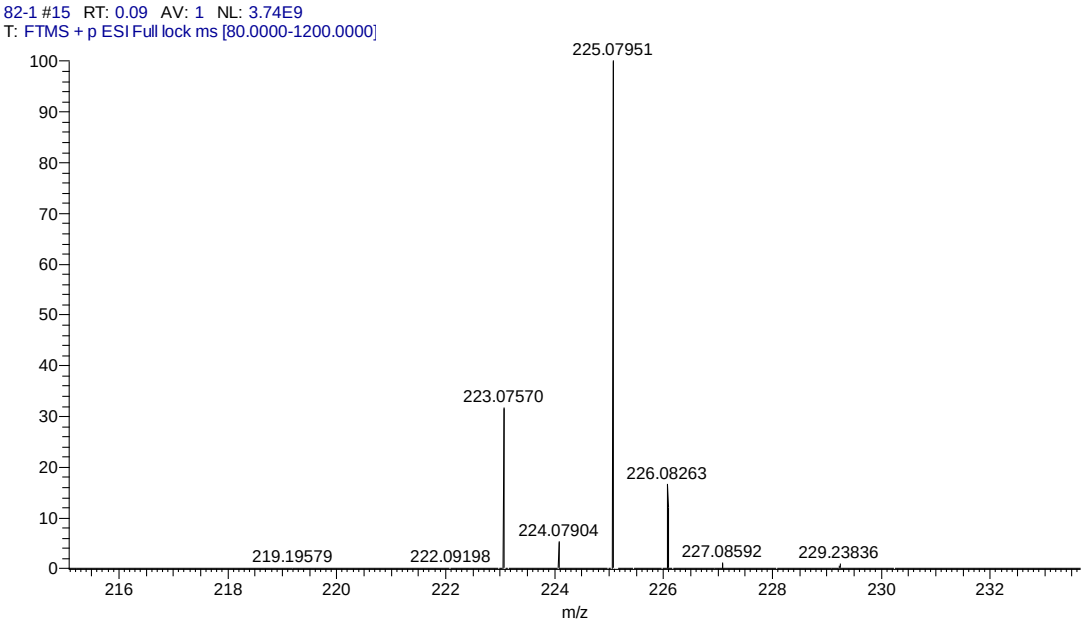
2a: HRMS (ESI) m/z calcd for $C_{15}H_{11}O_2^+$ $(M+H)^+$ 223.0754, found 223.0752.

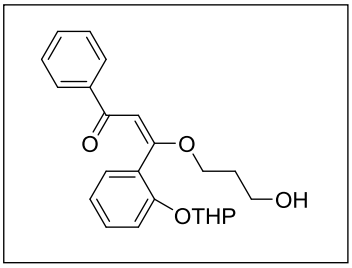


2a': HRMS (ESI) m/z calcd for $C_{15}H_{11}O^{18}O^+$ $(M+H)^+$ 225.0796, found 225.0797; calcd for $C_{15}H_{11}O_2^+$ $(M+H)^+$ 223.0754, found 223.0759. (**M1 : M2 = 3.2E9 : 6.44E8**)



2a': HRMS (ESI) m/z calcd for $C_{15}H_{11}O_2^+$ $(M+H)^+$ 223.0754, found 223.0757. calcd for $C_{15}H_{11}{}^{18}OO^+$ $(M+H)^+$ 225.0796, found 225.0795. **(M1 : M2 = 1.19 E9 : 3.74 E9)**





(E)-3-(3-hydroxypropoxy)-1-phenyl-3-(2-((tetrahydro-2H-pyran-2-yl)oxy)phenyl)prop-2-en-1-one **10**. General procedure **B**, 68% yield (259 mg), 18 h, yellow liquid. (Petroleum ether/EtOAc =1/1, Rf = 0.5). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.85 (d, *J* = 7.03 Hz, 2H), 7.44 (t, *J* = 7.34 Hz, 1H), 7.35 (t, *J* = 7.53 Hz, 2H), 7.31 – 7.26 (m, 1H), 7.23 (dd, *J* = 7.57, 1.75 Hz, 1H), 7.13 (d, *J* = 7.40 Hz, 1H), 6.97 (td, *J* = 7.47, 1.05 Hz, 1H), 6.39 (s, 1H), 5.37 (s, 1H), 4.17 (t, *J* = 6.10 Hz, 2H), 3.83 (t, *J* = 11.26 Hz, 1H), 3.75 (t, *J* = 5.75 Hz, 2H), 3.52 (d, *J* = 11.20 Hz, 1H), 2.71 (s, 1H), 2.01 (dd, *J* = 11.53, 5.60 Hz, 2H), 1.80 – 1.38 (m, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 189.9, 168.2, 154.1, 139.6, 131.9, 130.5, 129.7, 128.1, 127.9, 126.3, 121.2, 115.1, 100.1, 96.6, 66.6, 61.7, 59.4, 31.6, 30.1, 25.1, 18.5. **HRMS (ESI)** m/z calcd for $C_{23}H_{26}O_5Na^+$ $(M+Na)^+$ 405.1673, found 405.1677.

8. Reference

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9. NMR spectra

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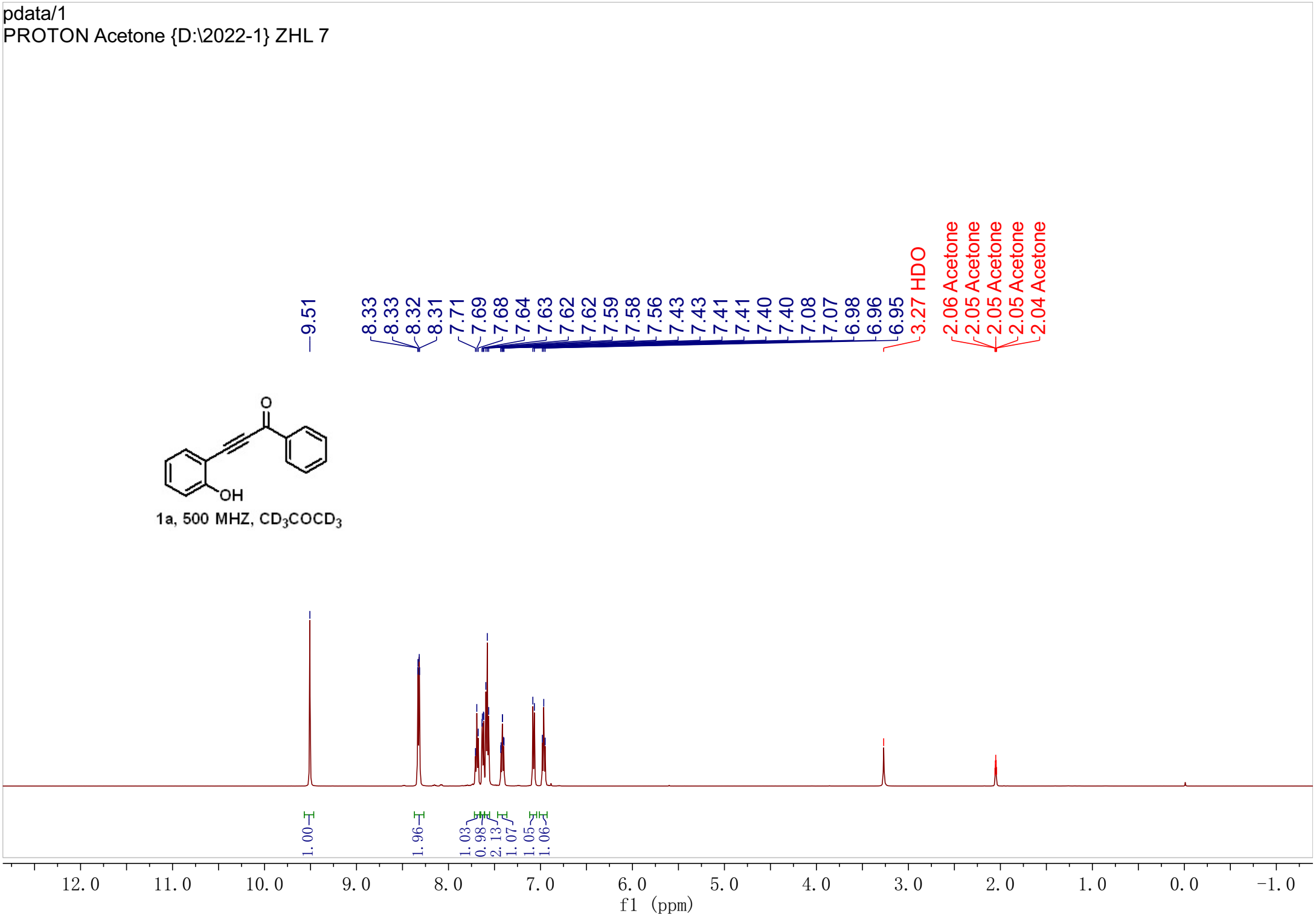

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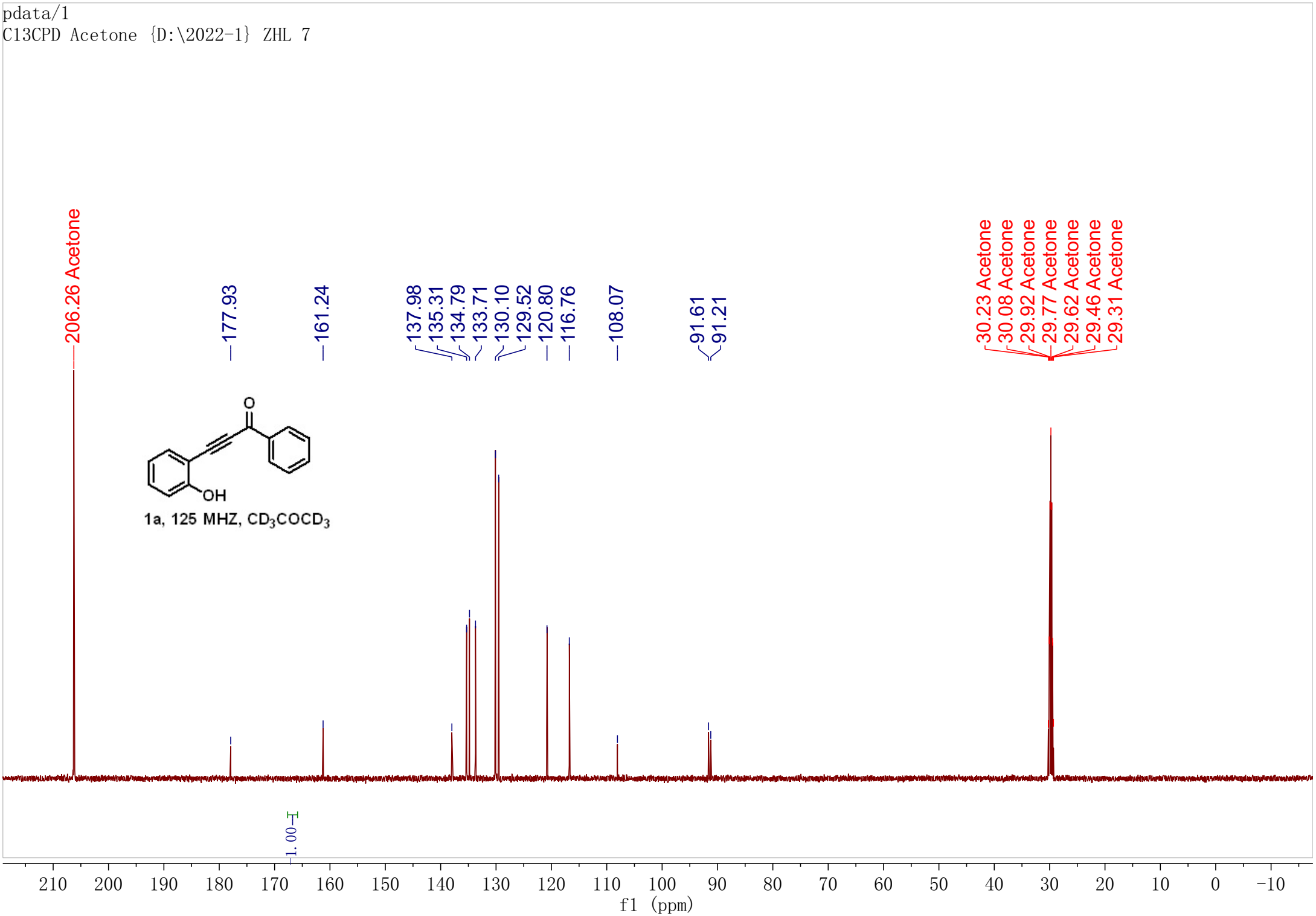

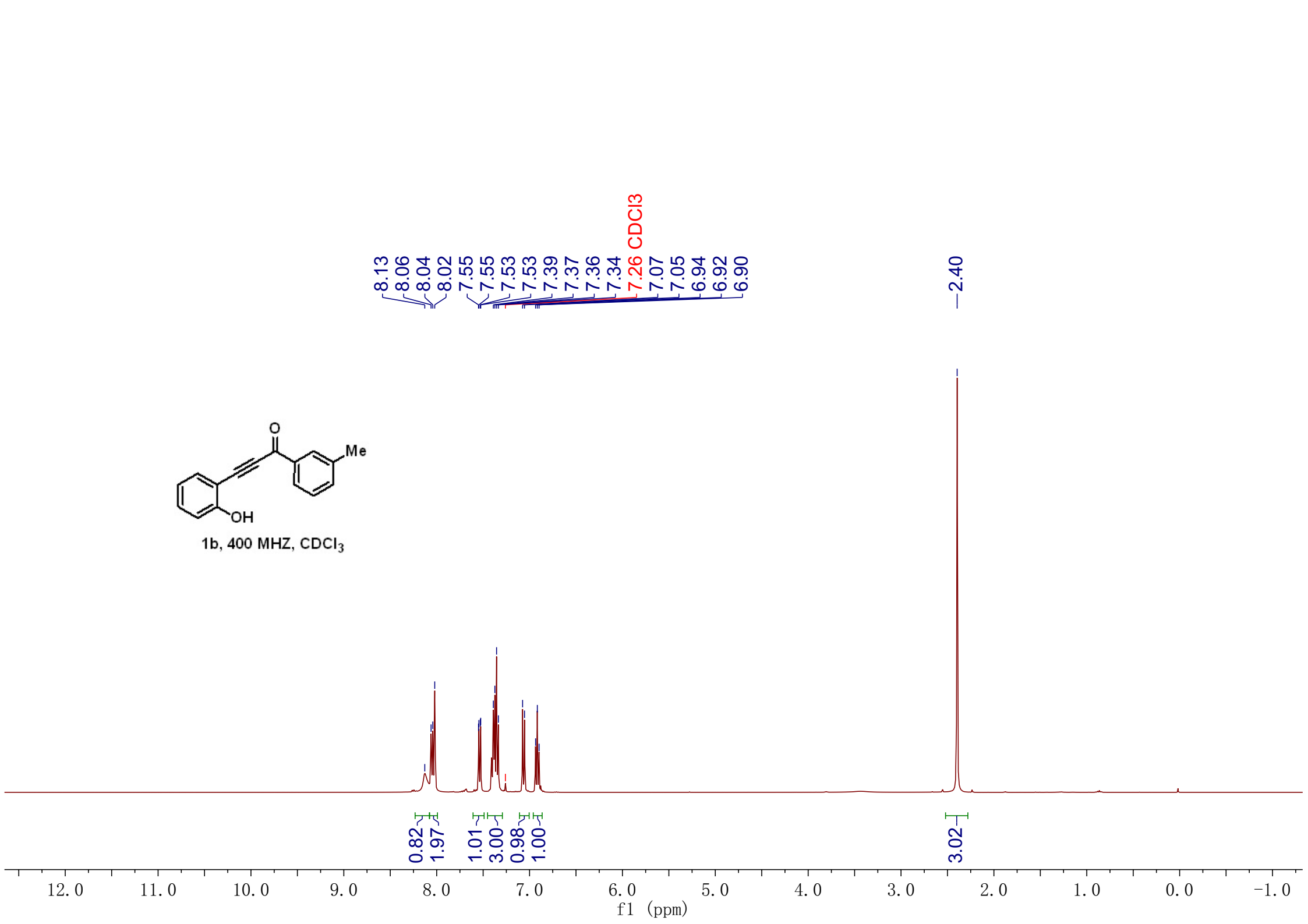
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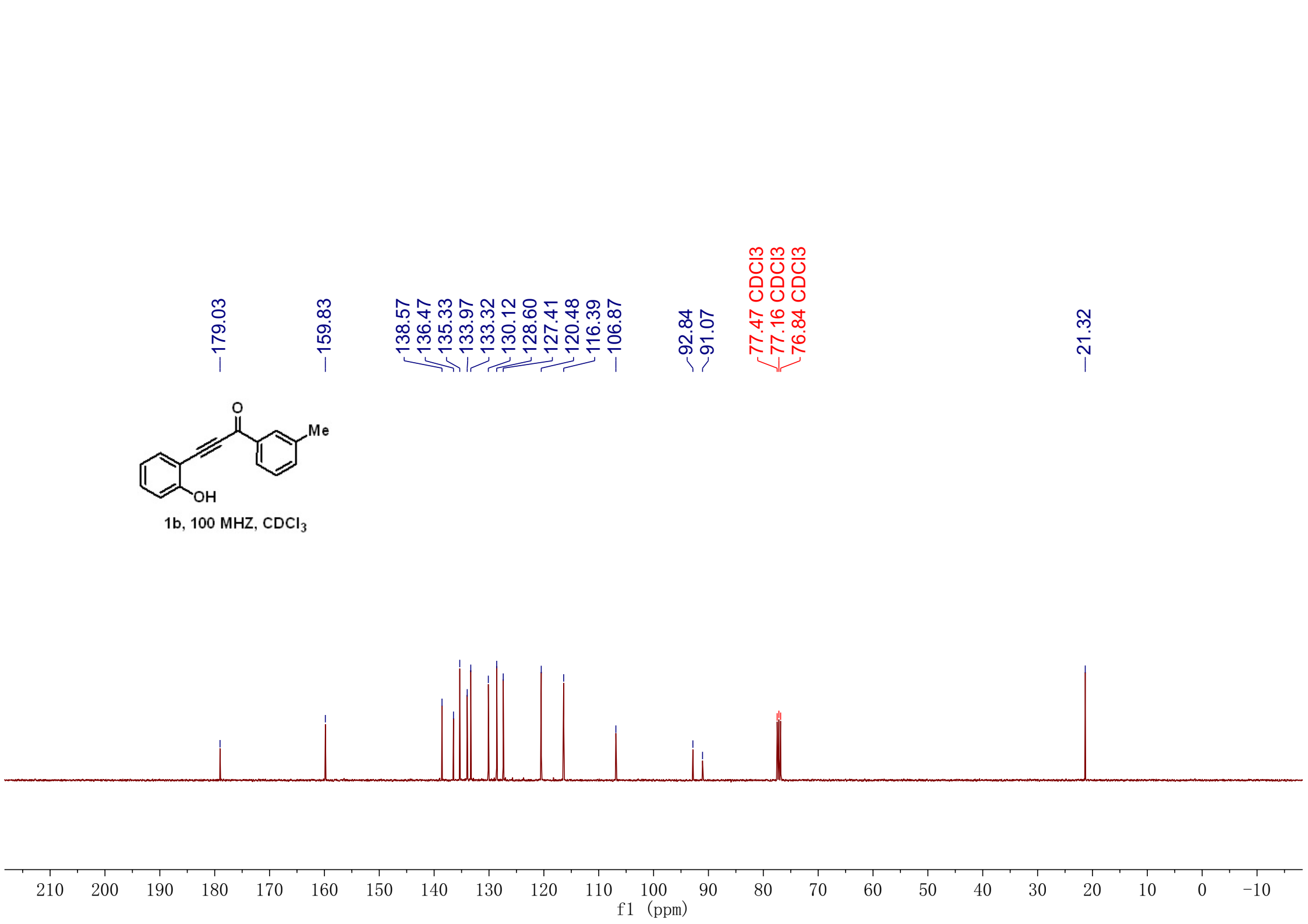
8.04
8.02
7.55
7.55
7.53
7.53
7.39
7.37
7.36
7.34
7.26 CDCl3
7.07
7.05
6.94
6.92
6.90
2.40
O
Me
OH
1b, 400 MHZ, CDCl3
0.82
1.97
1.01
3.00
0.98
1.00
3.02
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)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120.48
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106.87
92.84
91.07
77.47 CDCl3
77.16 CDCl3
76.84 CDCl3
21.32
O
Me
OH
1b, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
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100
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60
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20
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f1 (ppm)

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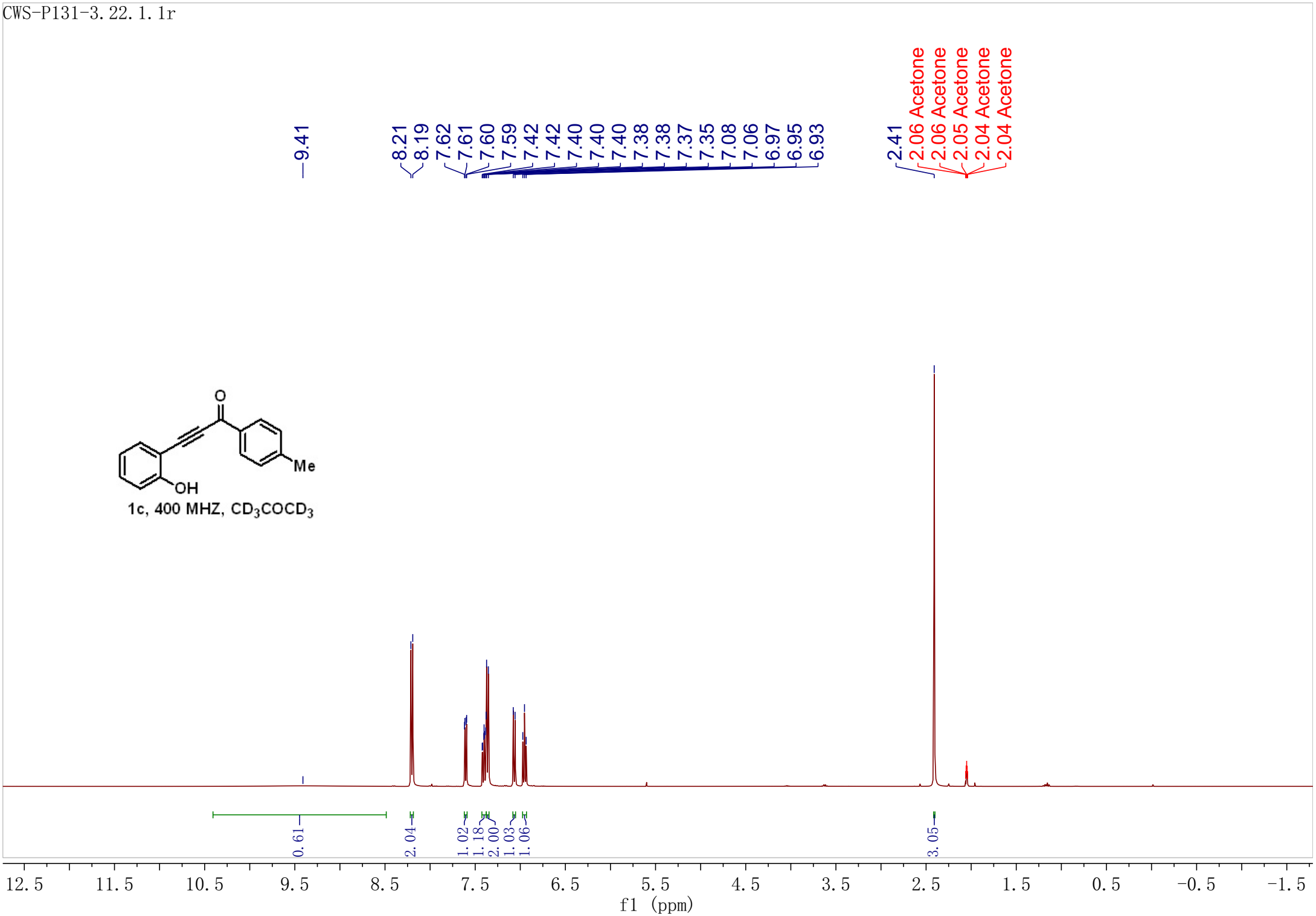

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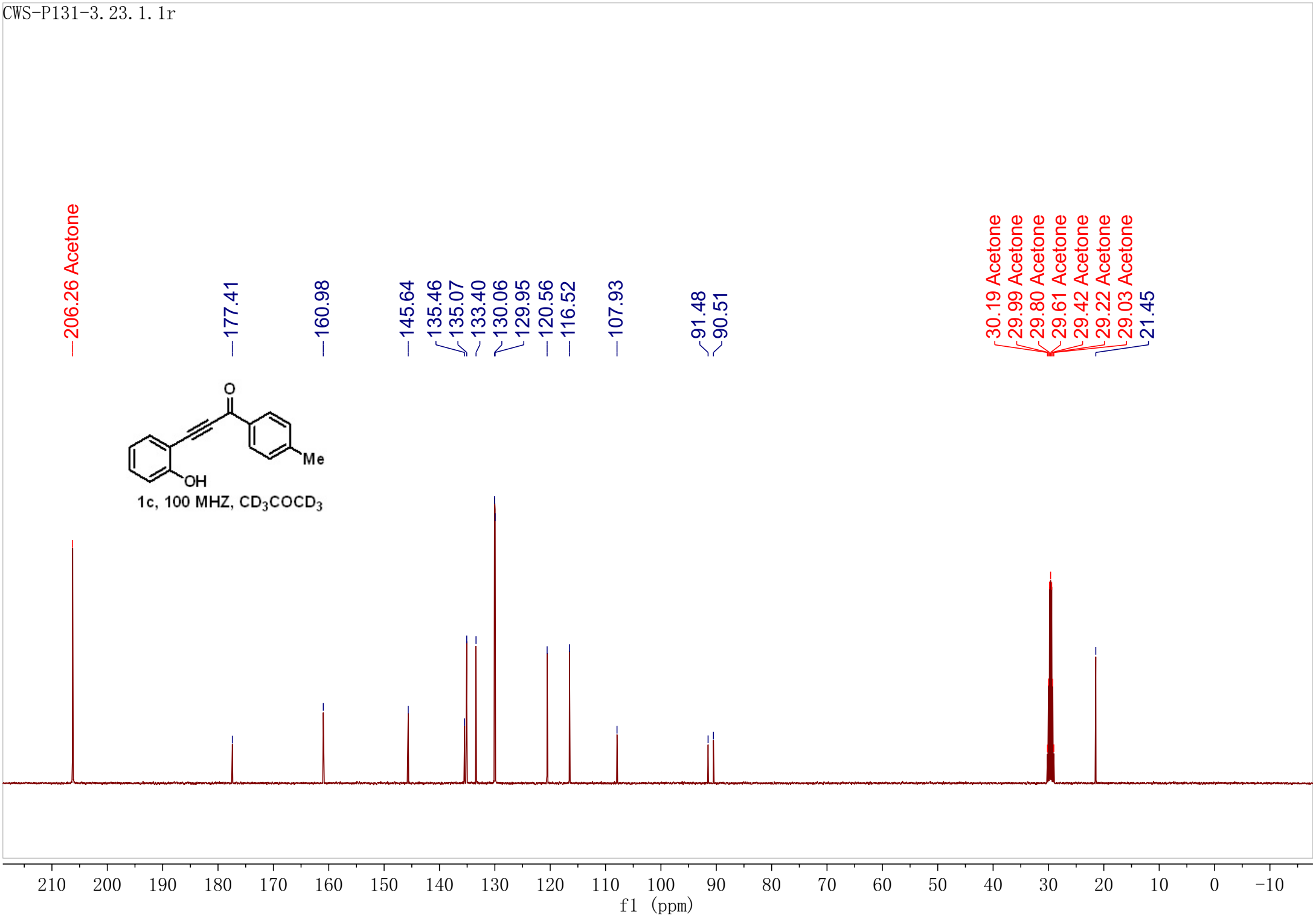

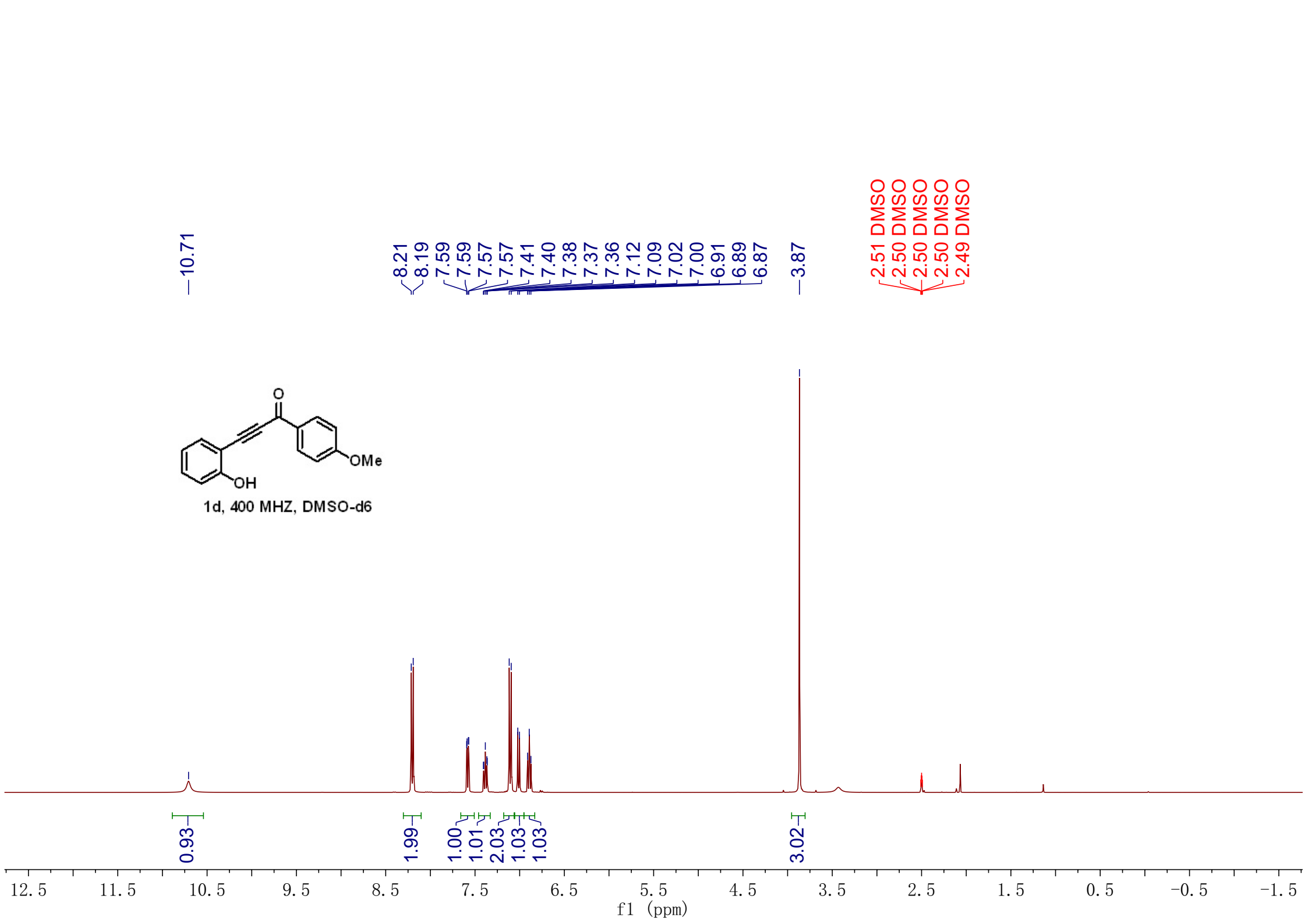
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7.36
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7.09
7.02
7.00
6.91
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6.87
3.87
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
O
OMe
OH
1d, 400 MHZ, DMSO-d6
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1.99
1.00
1.01
2.03
1.03
1.03
3.02
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
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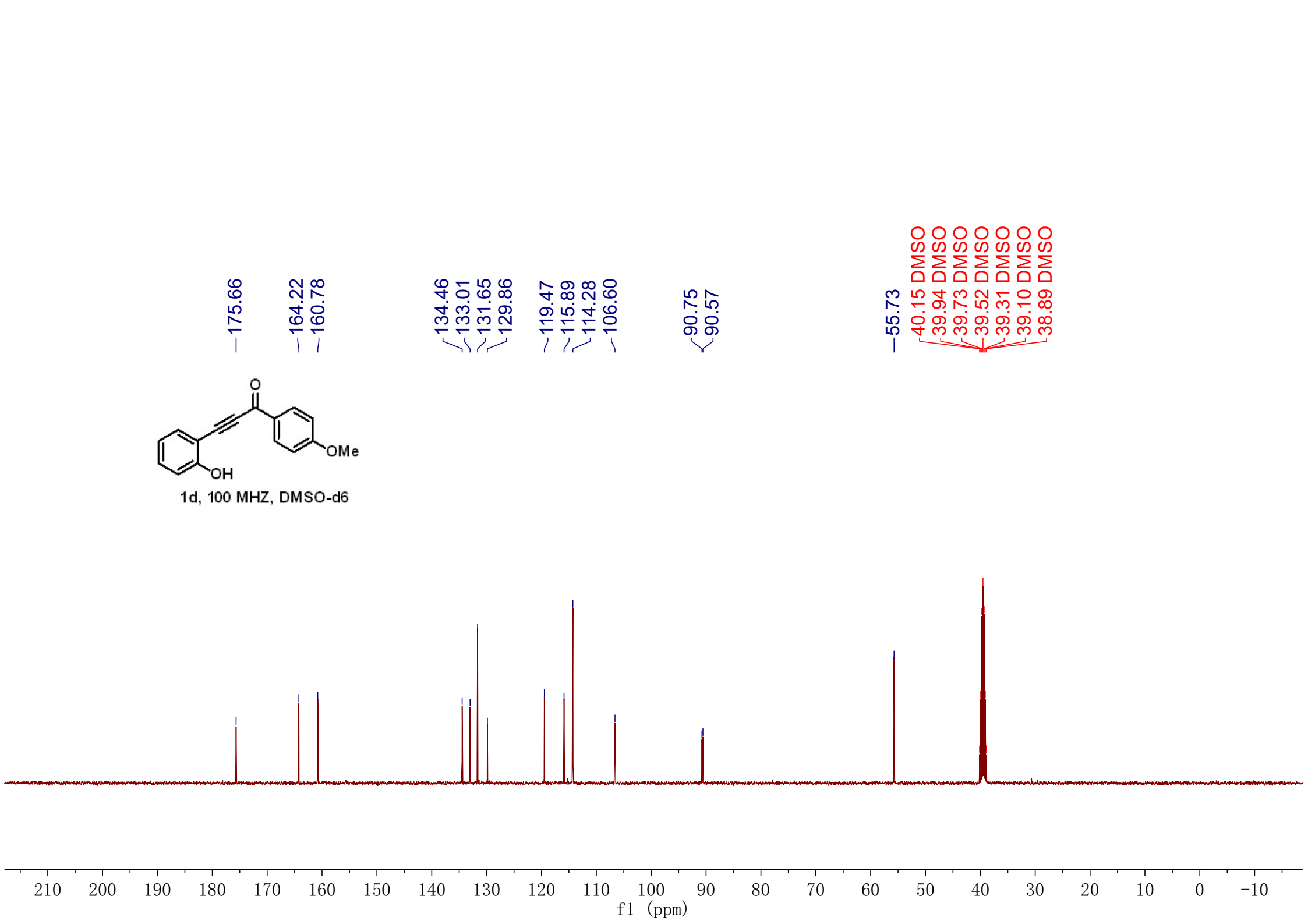
0.5
-0.5
-1.5
f1 (ppm)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39.94 DMSO
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39.31 DMSO
39.10 DMSO
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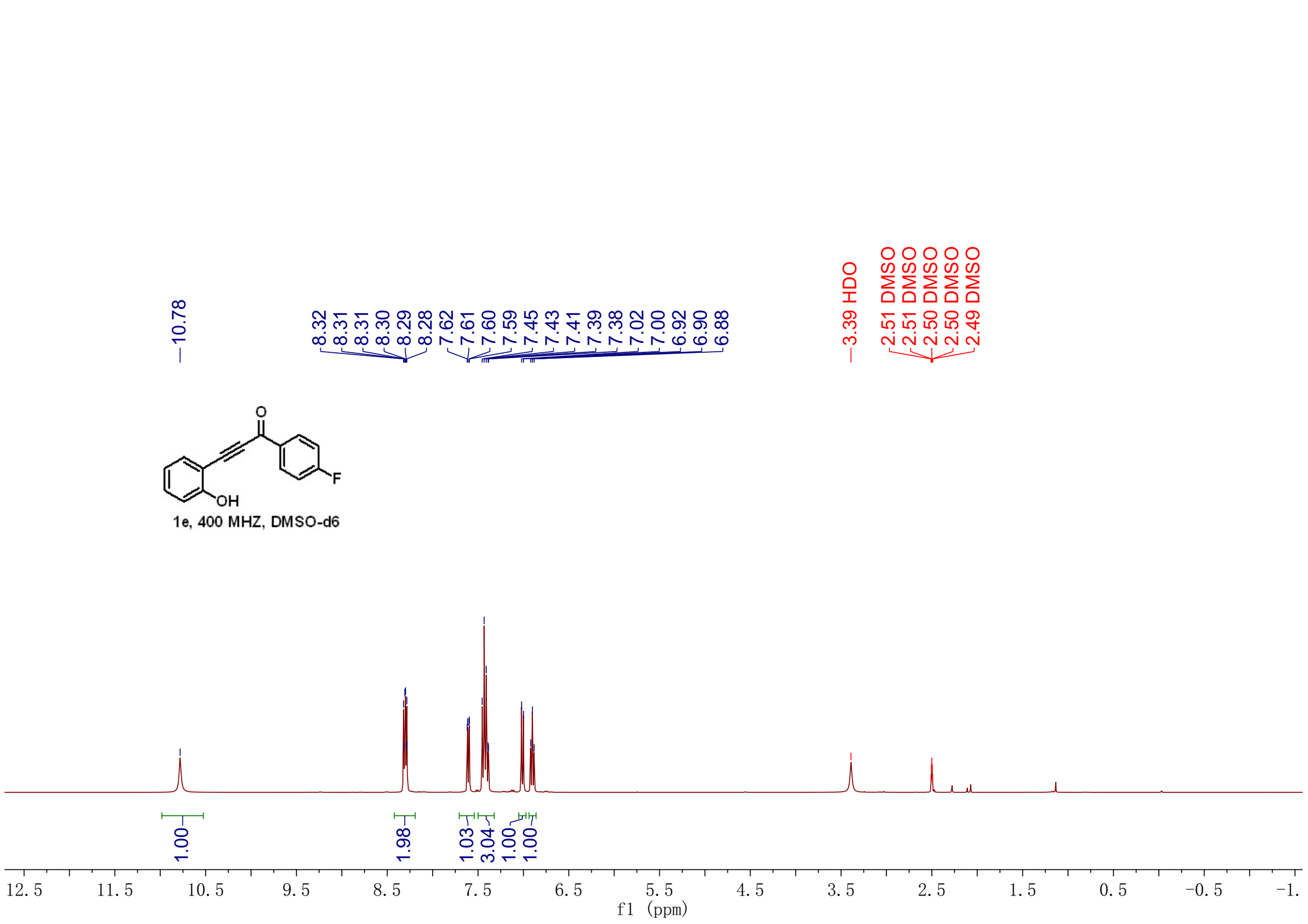
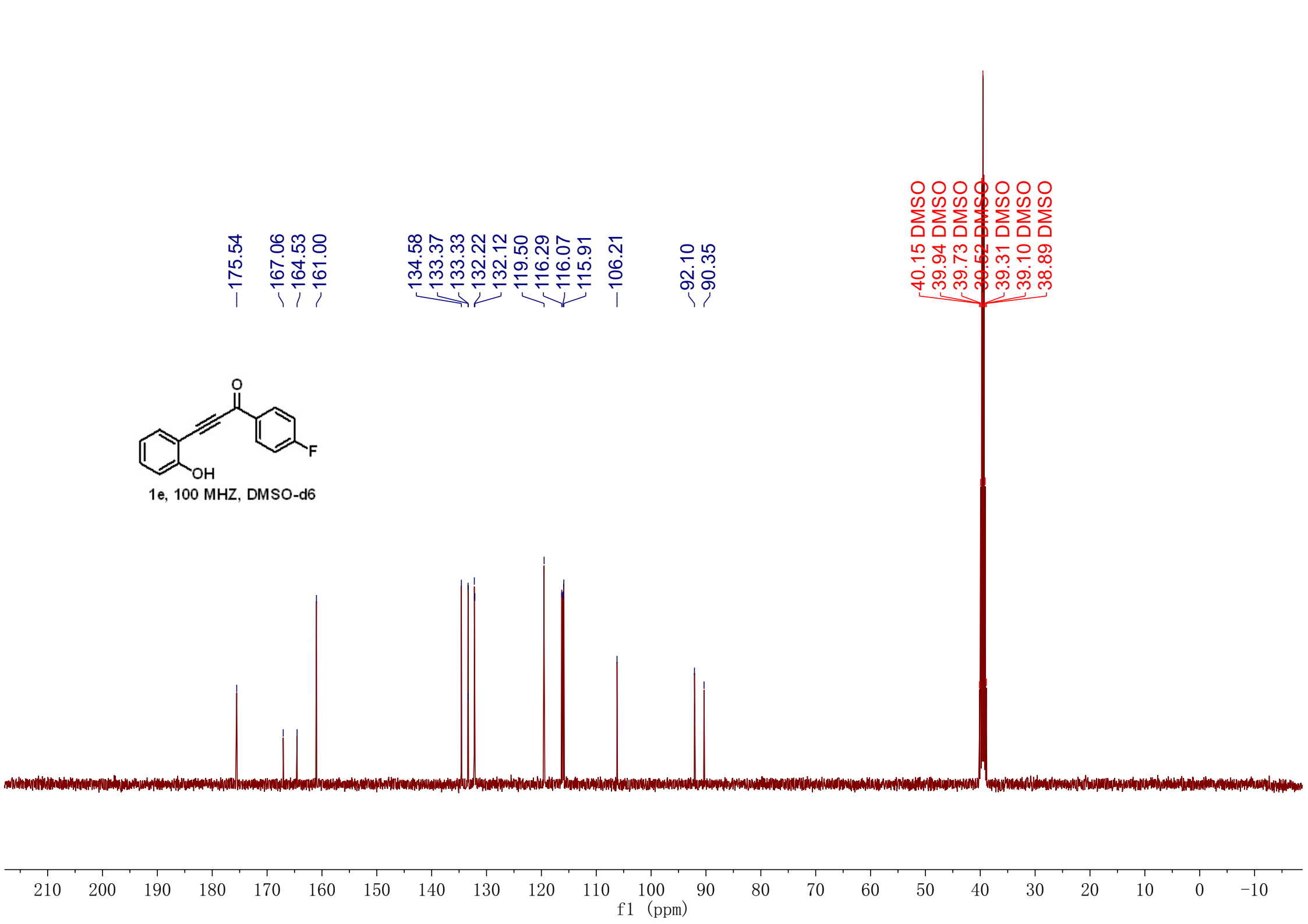
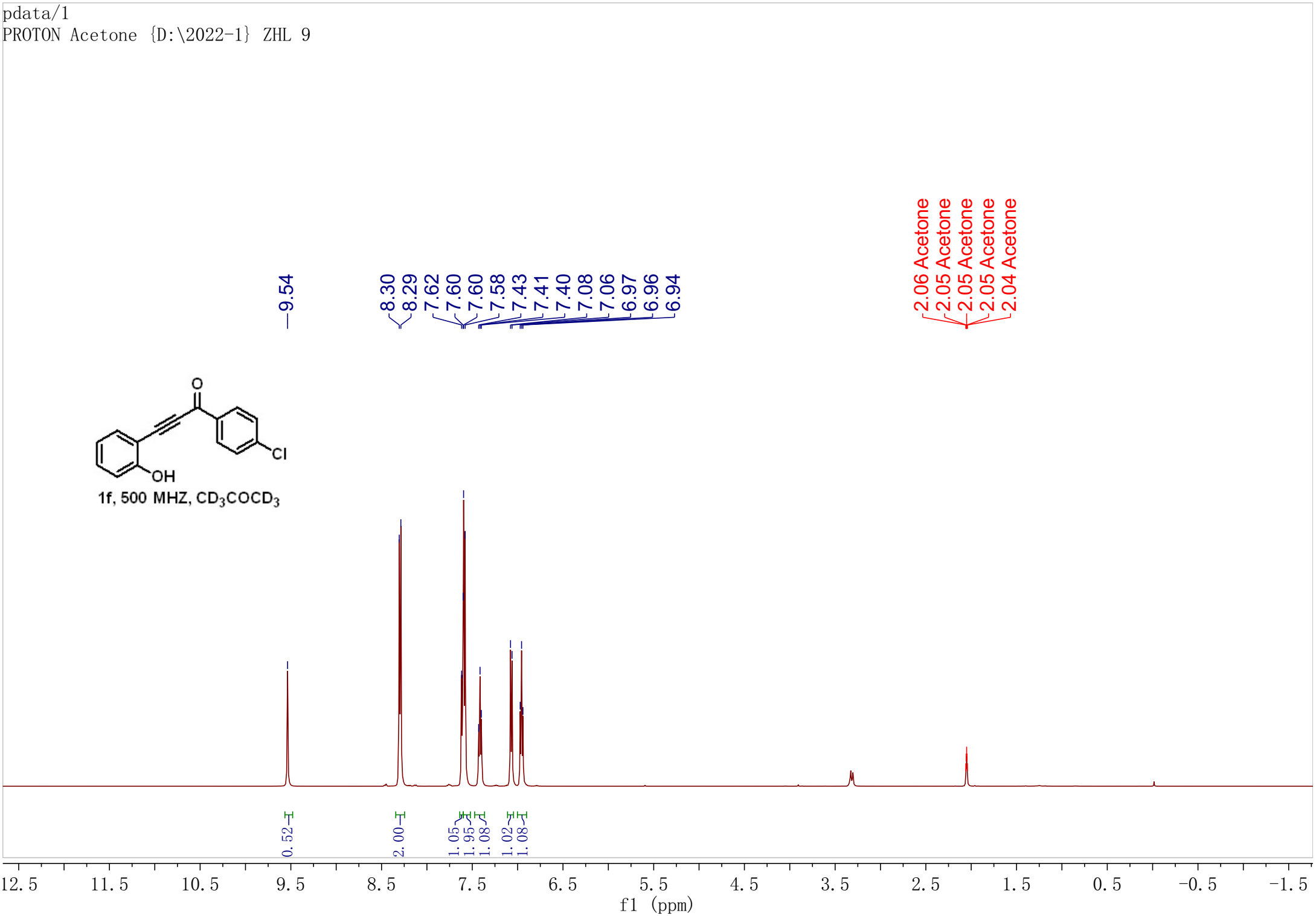
O
OMe
OH
1d, 100 MHZ, DMSO-d6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

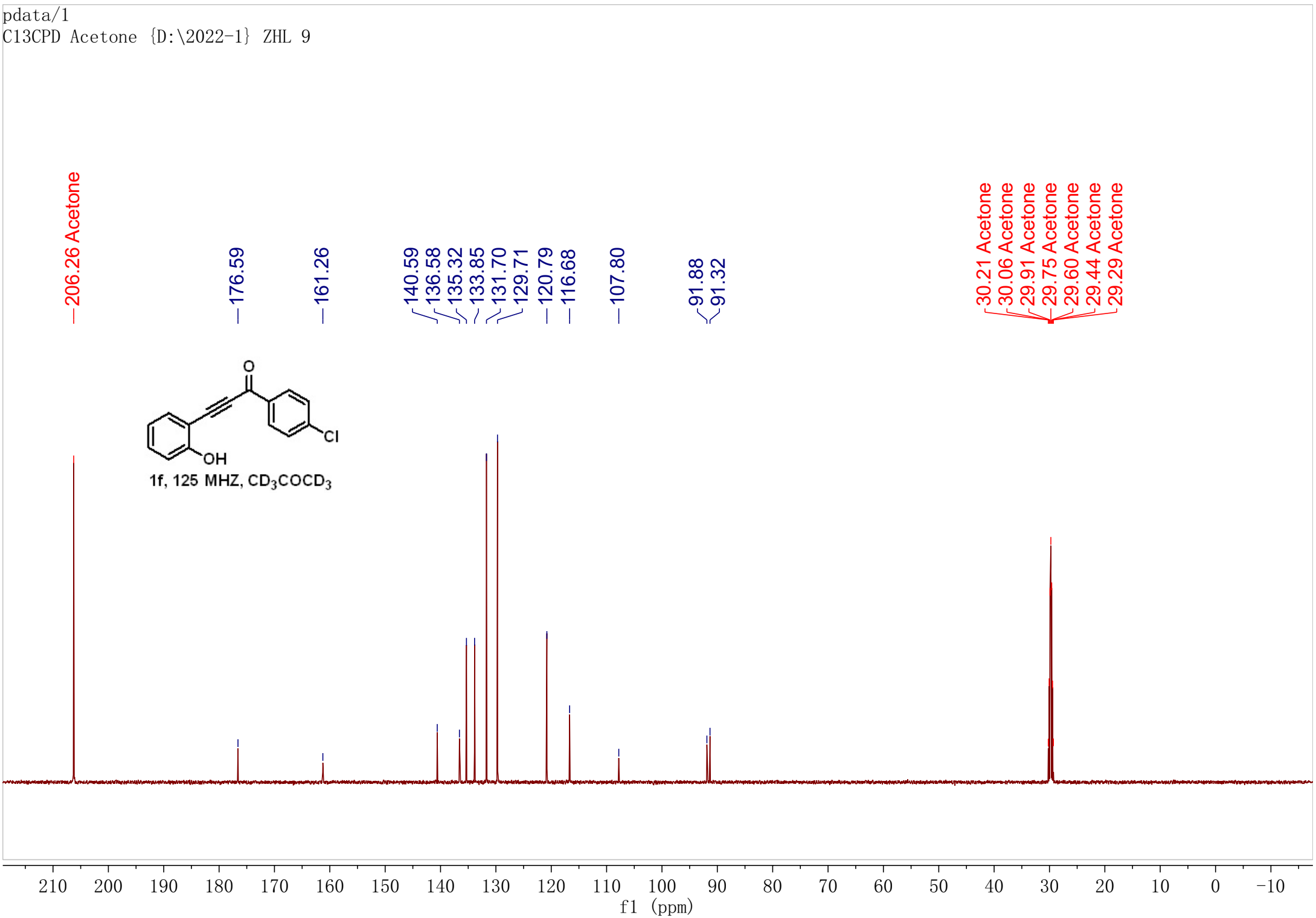

10.78
8.32
8.31
8.31
8.30
8.29
8.28
7.62
7.61
7.60
7.59
7.45
7.43
7.41
7.39
7.38
7.02
7.00
6.92
6.90
6.88
3.39 HDO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
O
F
OH
1e, 400 MHZ, DMSO-d6
1.00
1.98
1.03
3.04
1.00
1.00
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)


175.54
167.06
164.53
161.00
134.58
133.37
133.33
132.22
132.12
119.50
116.29
116.07
115.91
106.21
92.10
90.35
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
O
F
OH
1e, 100 MHZ, DMSO-d6
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)

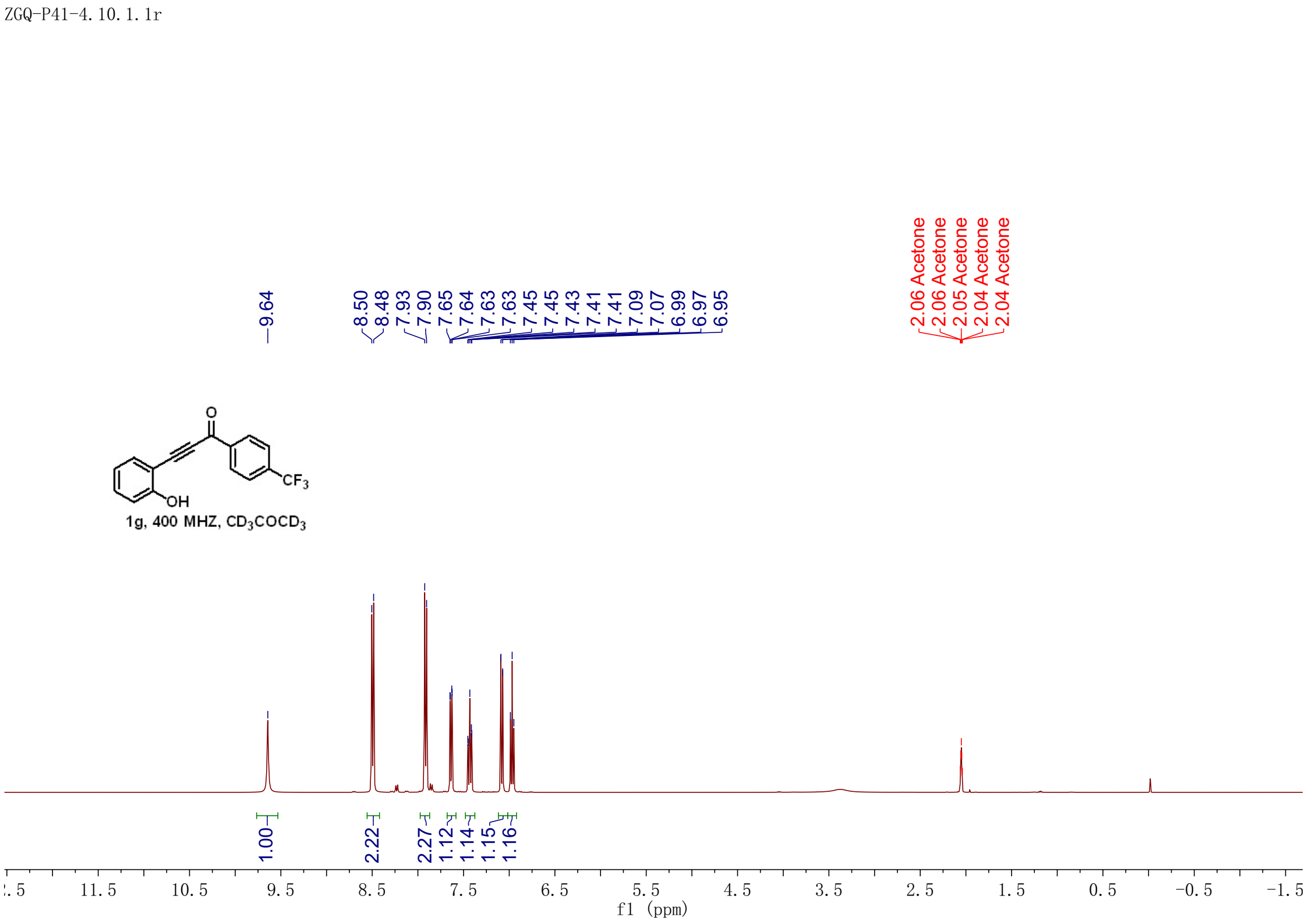
pdata/1
PROTON Acetone {D:\2022-1} ZHL 9



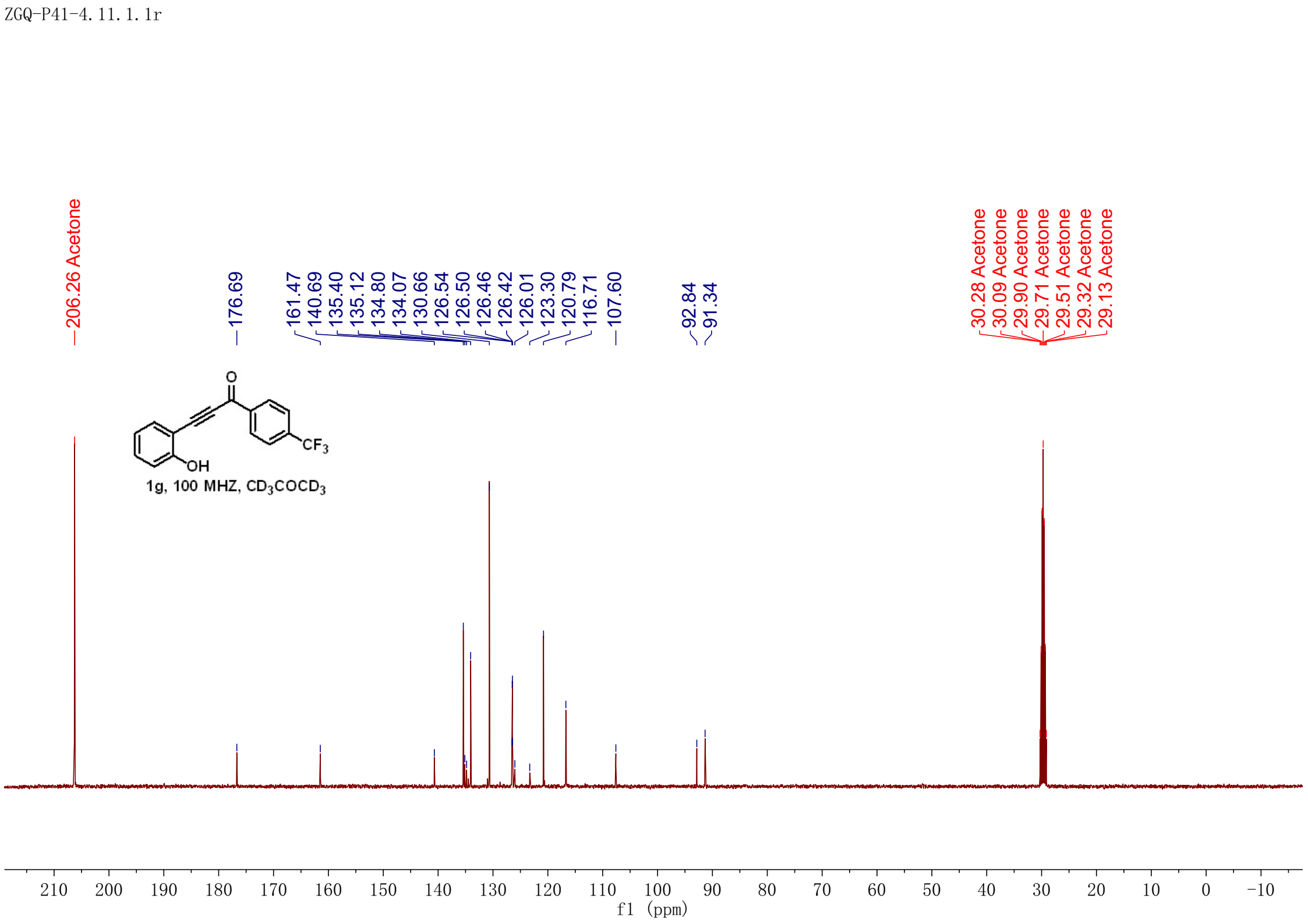
pdata/1
C13CPD Acetone {D:\2022-1} ZHL 9

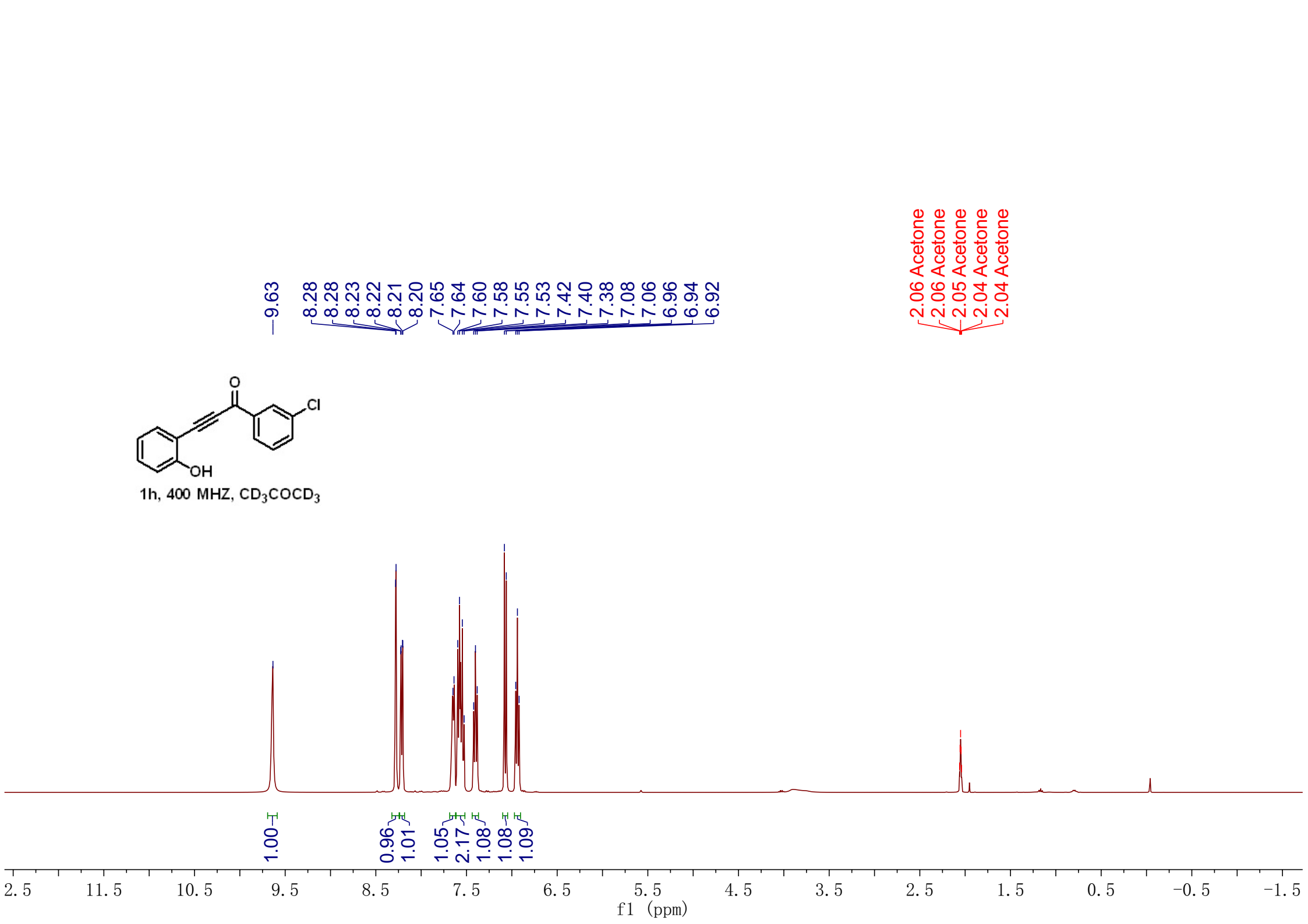


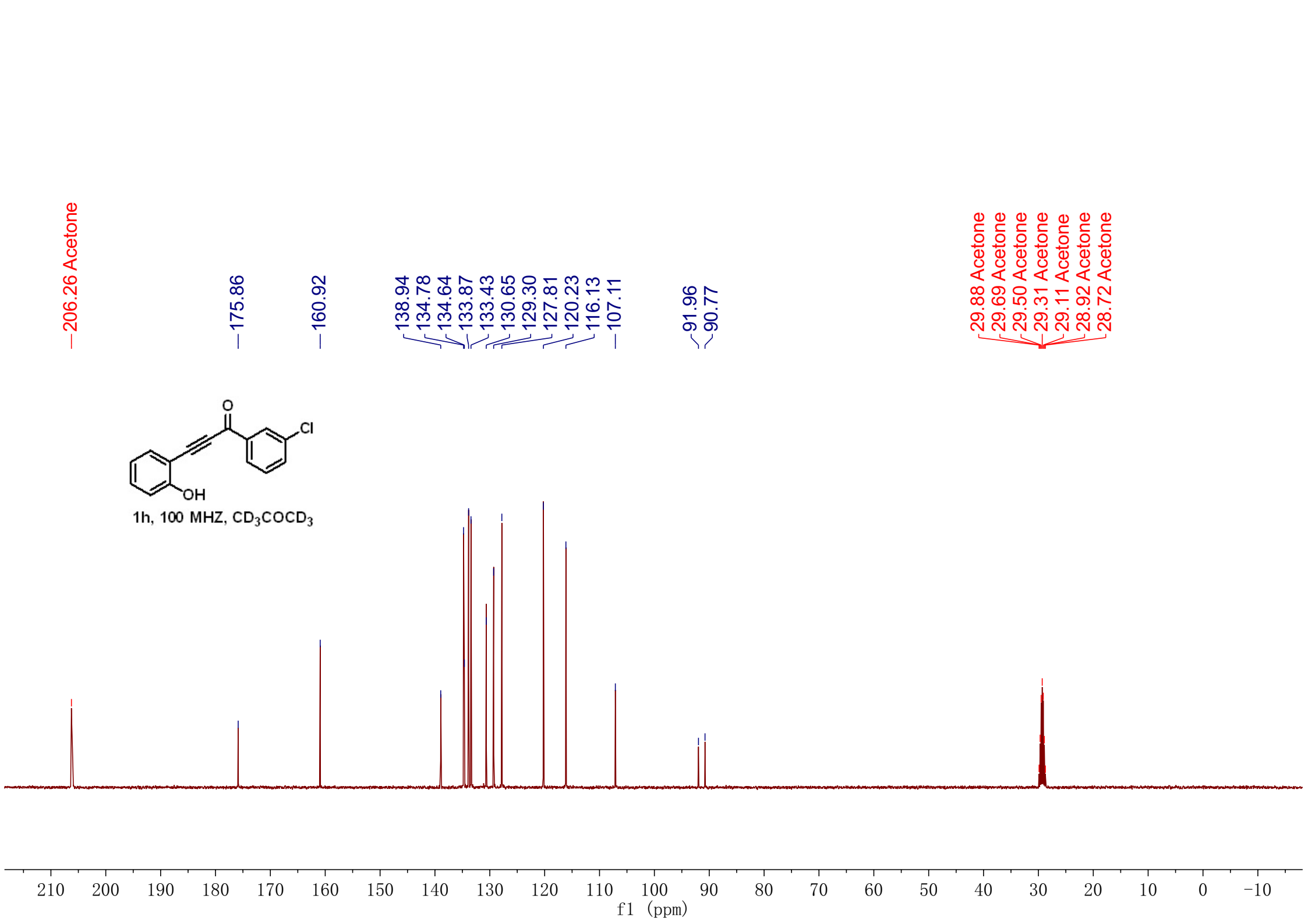
ZGQ-P41-4.10.1.1r

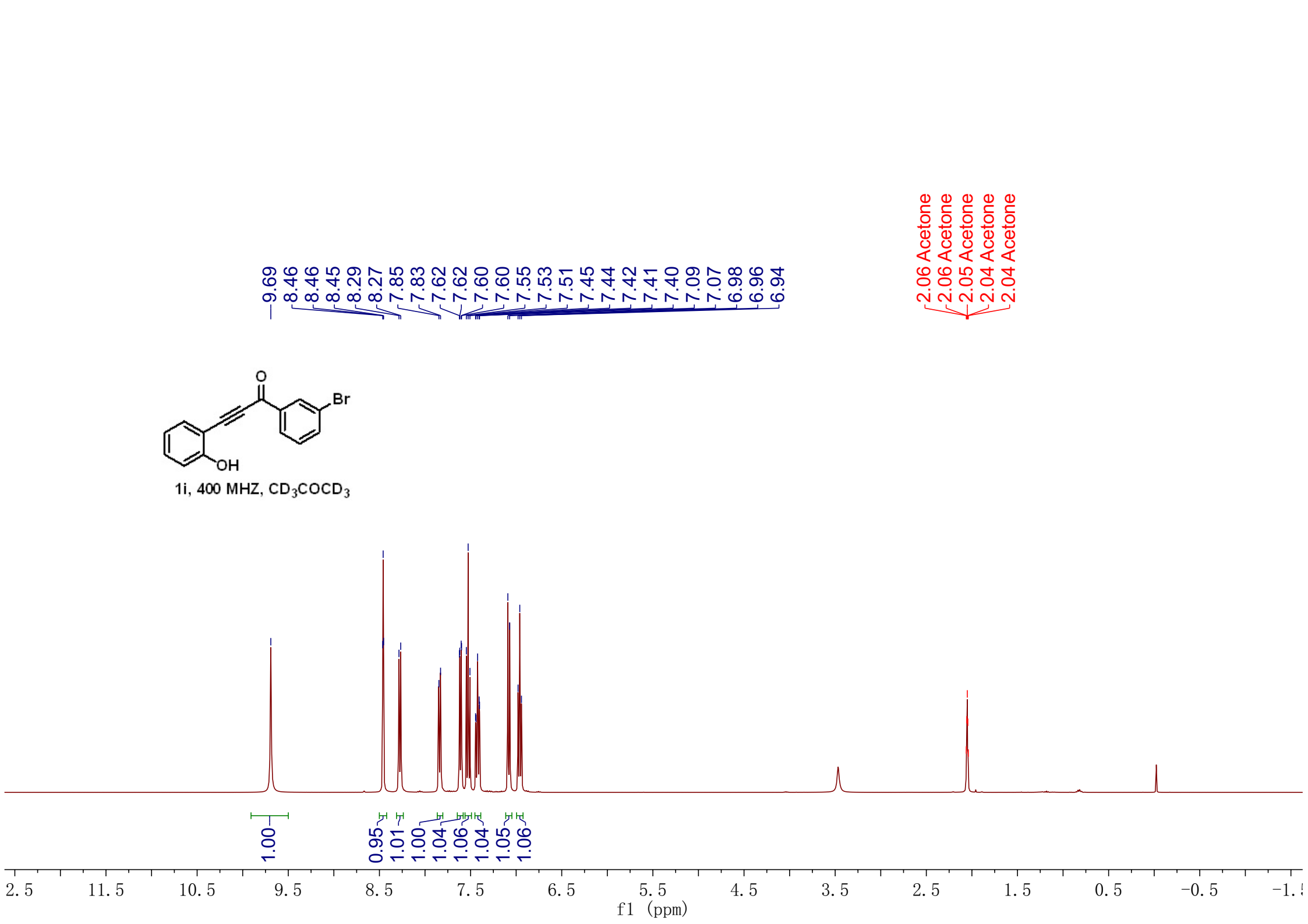


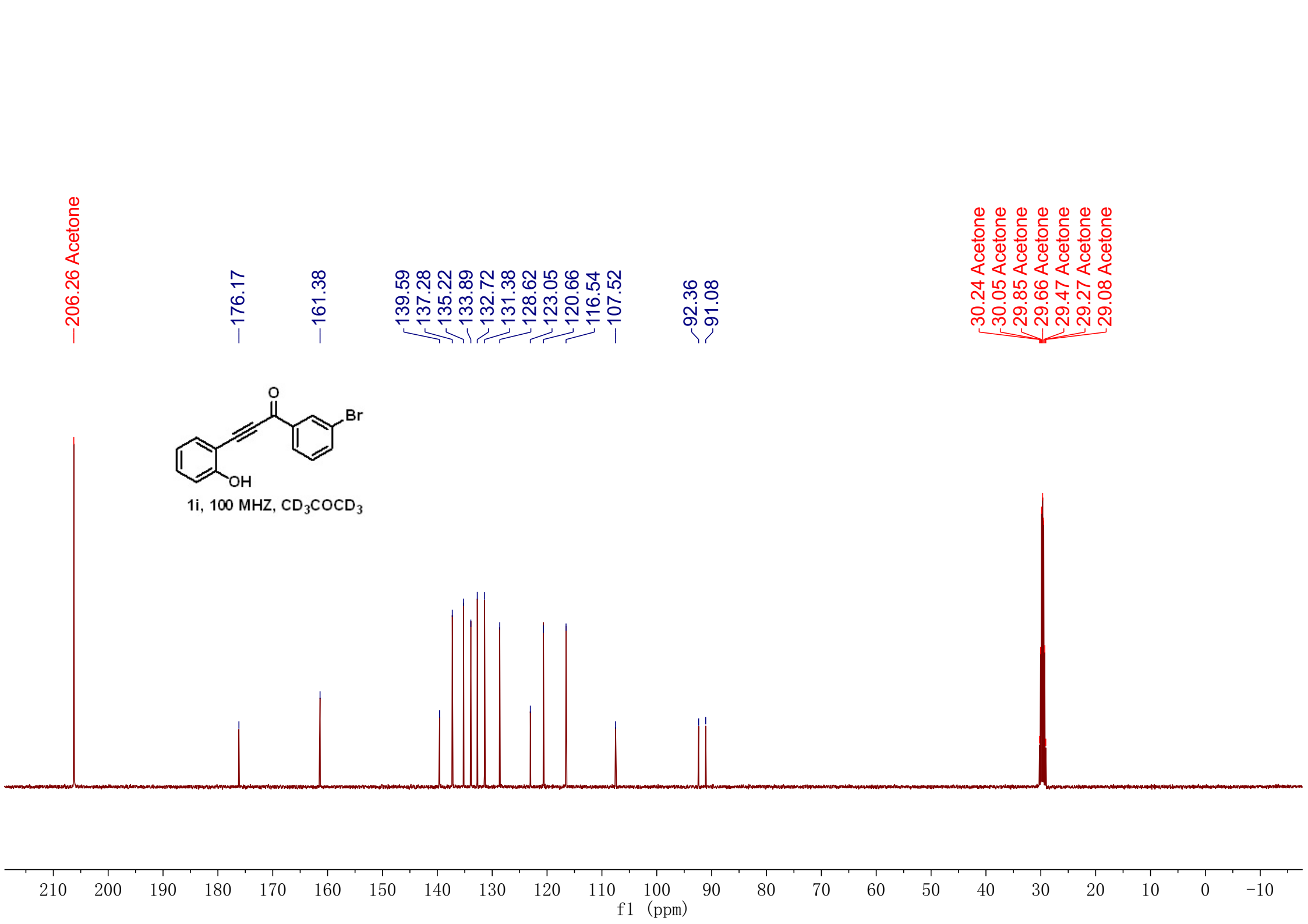
ZGQ-P41-4.11.1.1r

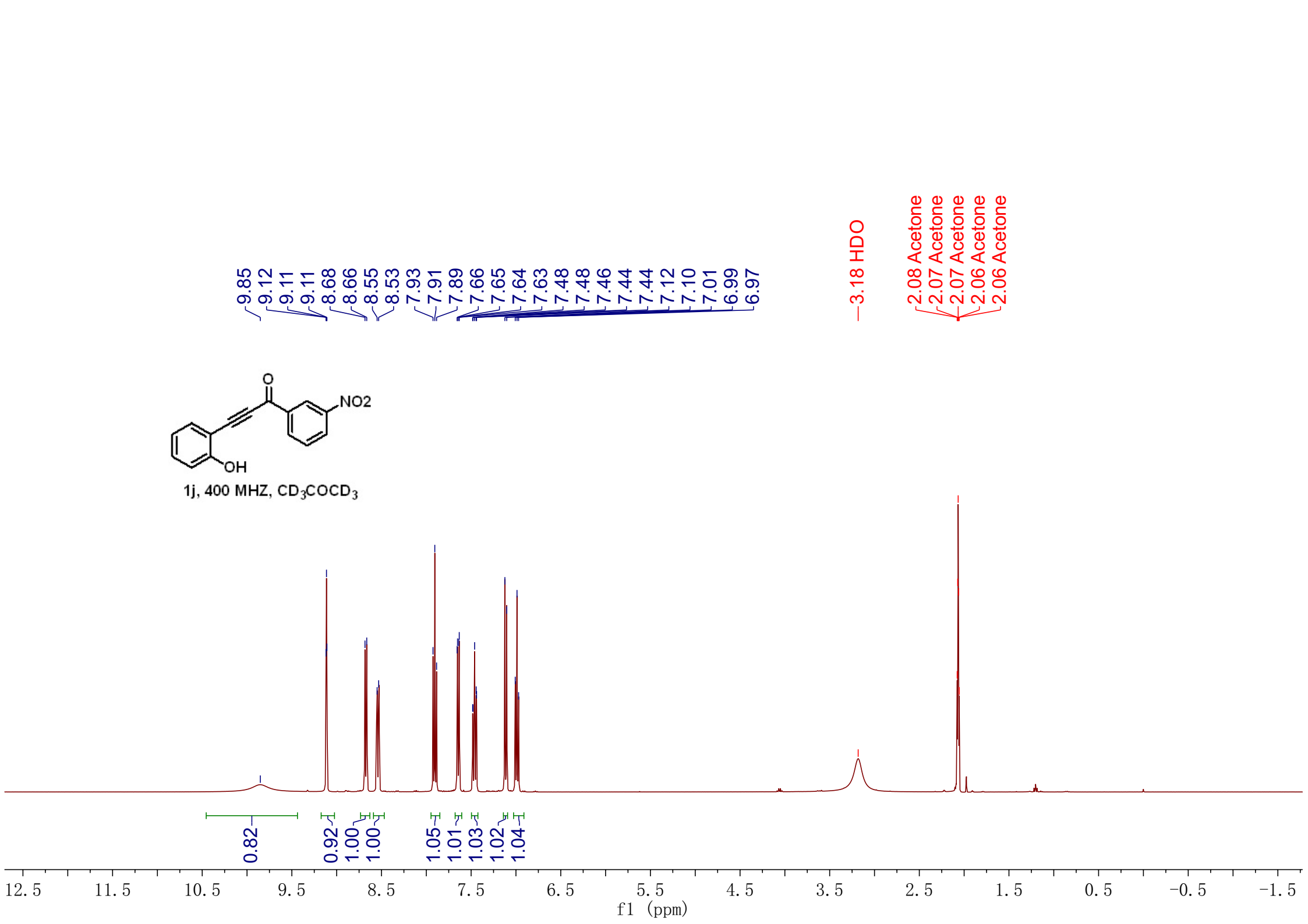


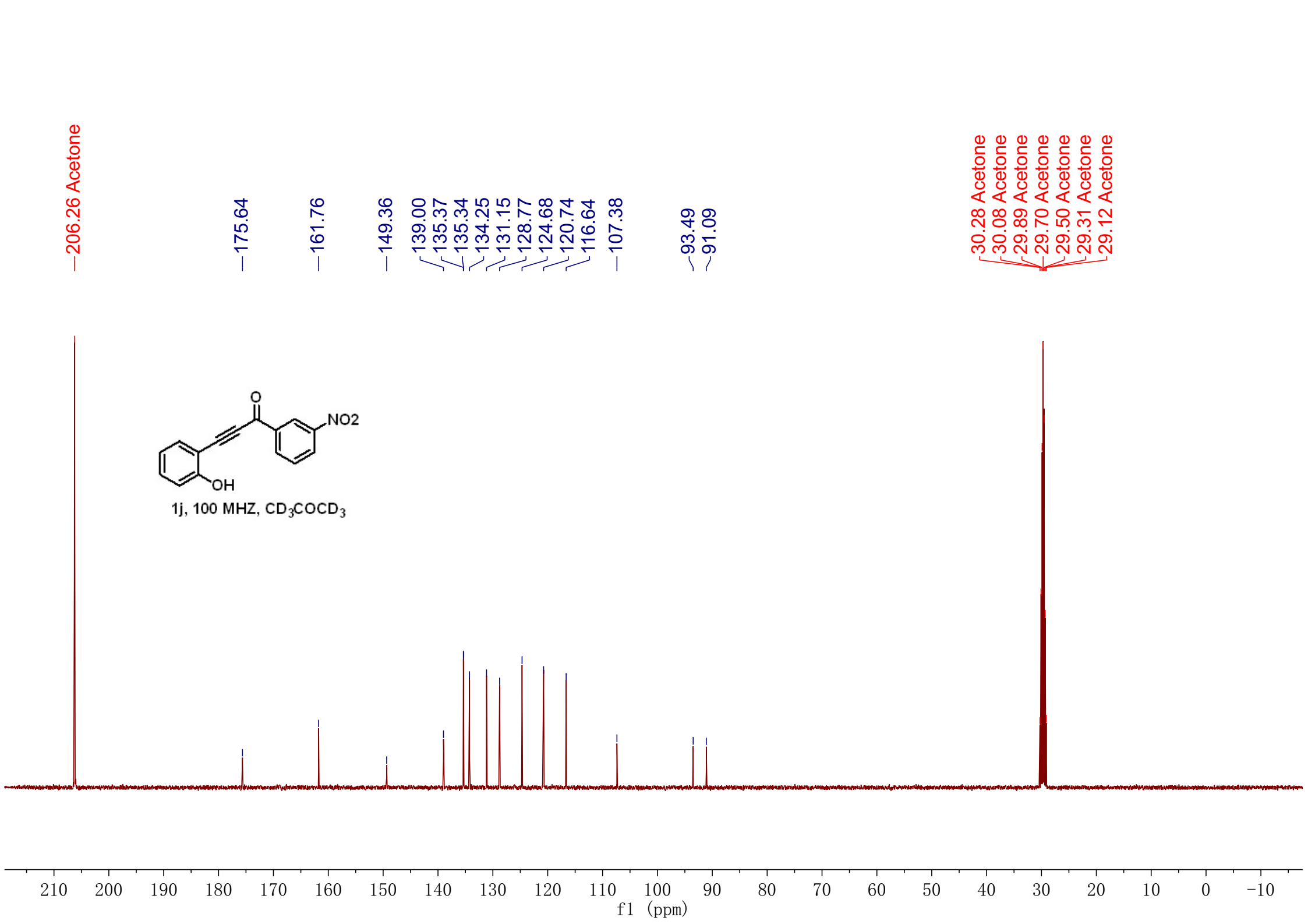

9.63
8.28
8.28
8.23
8.22
8.21
8.20
7.65
7.64
7.60
7.58
7.55
7.53
7.42
7.40
7.38
7.08
7.06
6.96
6.94
6.92
2.06 Acetone
2.06 Acetone
2.05 Acetone
2.04 Acetone
2.04 Acetone
O
Cl
OH
1h, 400 MHZ, CD3COCD3
1.00
0.96
1.01
1.05
2.17
1.08
1.08
1.09
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)


206.26 Acetone
175.86
160.92
138.94
134.78
134.64
133.87
133.43
130.65
129.30
127.81
120.23
116.13
107.11
91.96
90.77
29.88 Acetone
29.69 Acetone
29.50 Acetone
29.31 Acetone
29.11 Acetone
28.92 Acetone
28.72 Acetone
O
Cl
OH
1h, 100 MHZ, CD3COCD3
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)

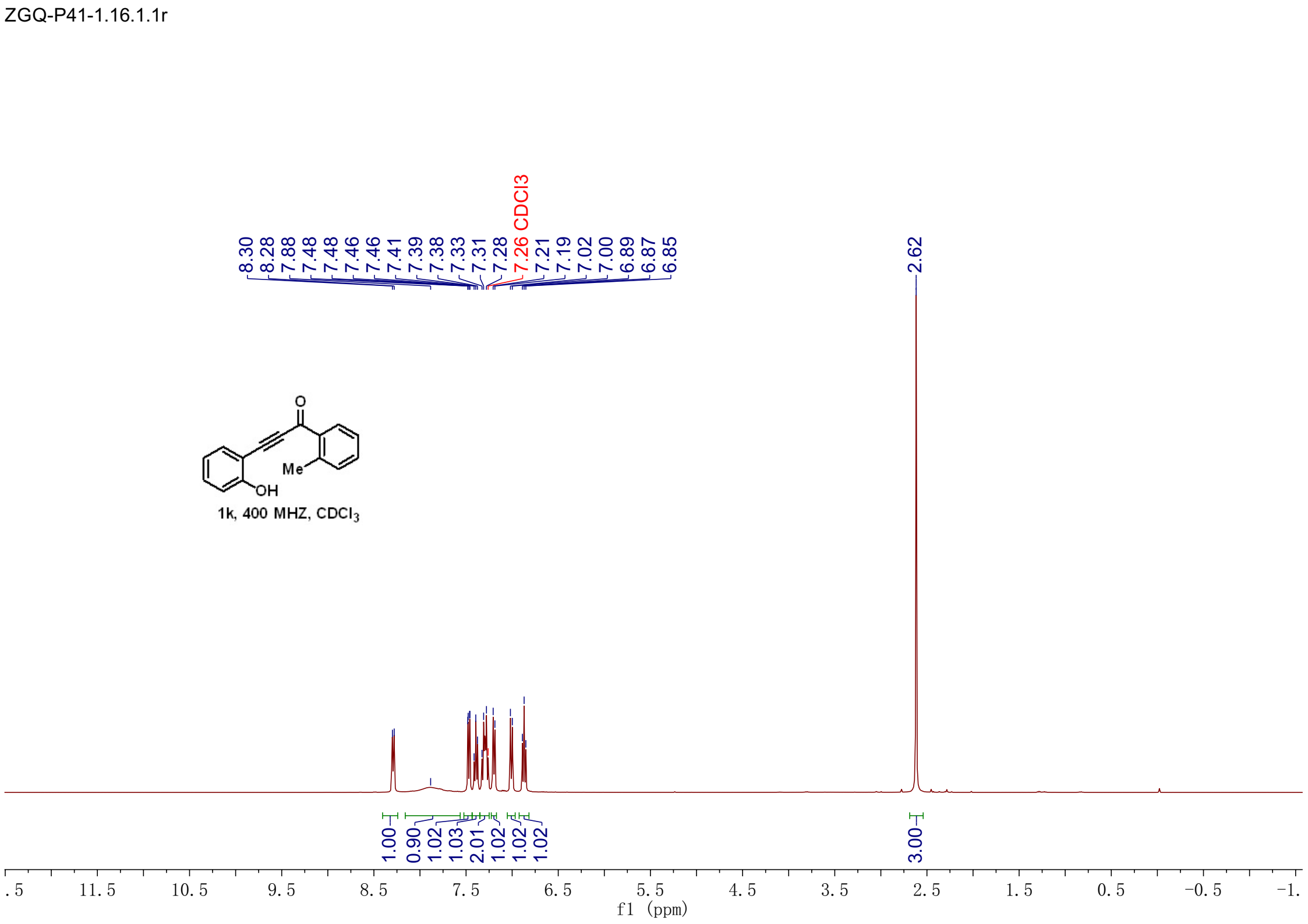

9.69
8.46
8.46
8.45
8.29
8.27
7.85
7.83
7.62
7.62
7.60
7.60
7.55
7.53
7.51
7.45
7.44
7.42
7.41
7.40
7.09
7.07
6.98
6.96
6.94
2.06 Acetone
2.06 Acetone
2.05 Acetone
2.04 Acetone
2.04 Acetone
O
Br
OH
1i, 400 MHZ, CD3COCD3
1.00
0.95
1.01
1.00
1.04
1.06
1.04
1.05
1.06
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


206.26 Acetone
176.17
161.38
139.59
137.28
135.22
133.89
132.72
131.38
128.62
123.05
120.66
116.54
107.52
92.36
91.08
30.24 Acetone
30.05 Acetone
29.85 Acetone
29.66 Acetone
29.47 Acetone
29.27 Acetone
29.08 Acetone
O
Br
OH
1i, 100 MHZ, CD3COCD3
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)

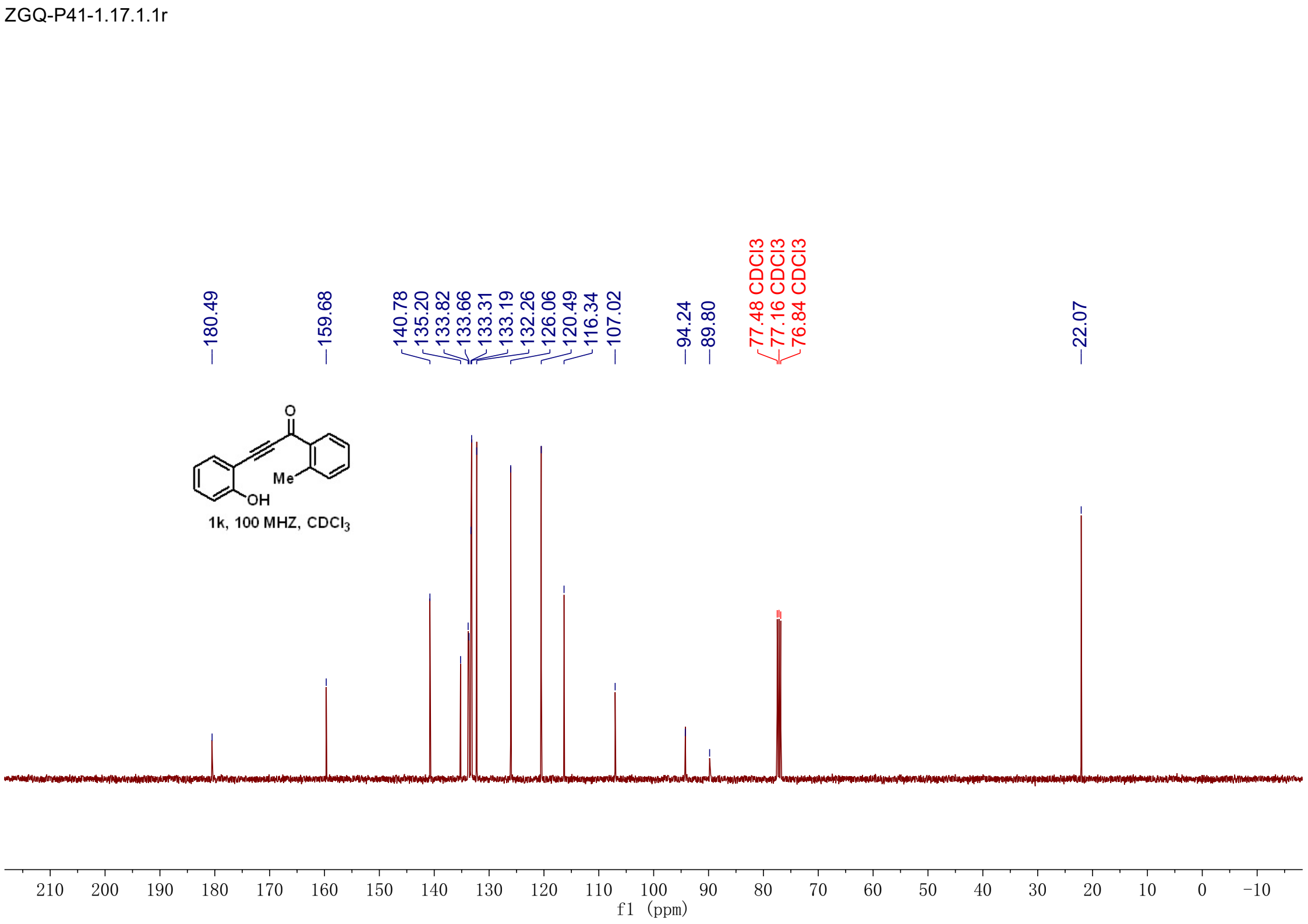

9.85
9.12
9.11
9.11
8.68
8.66
8.55
8.53
7.93
7.91
7.89
7.66
7.65
7.64
7.63
7.48
7.48
7.46
7.44
7.44
7.12
7.10
7.01
6.99
6.97
3.18 HDO
2.08 Acetone
2.07 Acetone
2.07 Acetone
2.06 Acetone
2.06 Acetone
O
NO2
OH
1j, 400 MHZ, CD3COCD3
0.82
0.92
1.00
1.00
1.05
1.01
1.03
1.02
1.04
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)

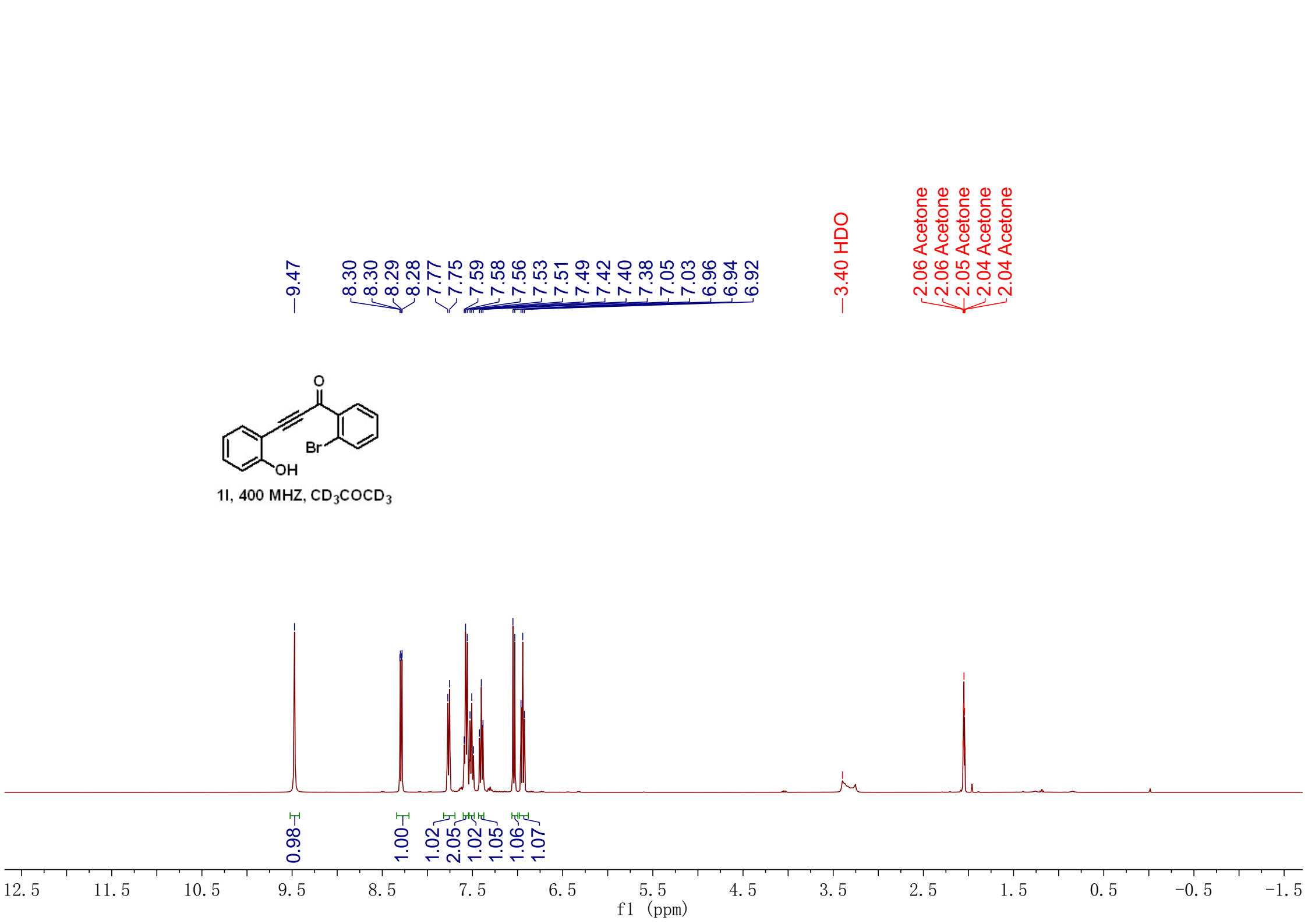

206.26 Acetone
175.64
161.76
149.36
139.00
135.37
135.34
134.25
131.15
128.77
124.68
120.74
116.64
107.38
93.49
91.09
30.28 Acetone
30.08 Acetone
29.89 Acetone
29.70 Acetone
29.50 Acetone
29.31 Acetone
29.12 Acetone
O
NO2
OH
1j, 100 MHZ, CD3COCD3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

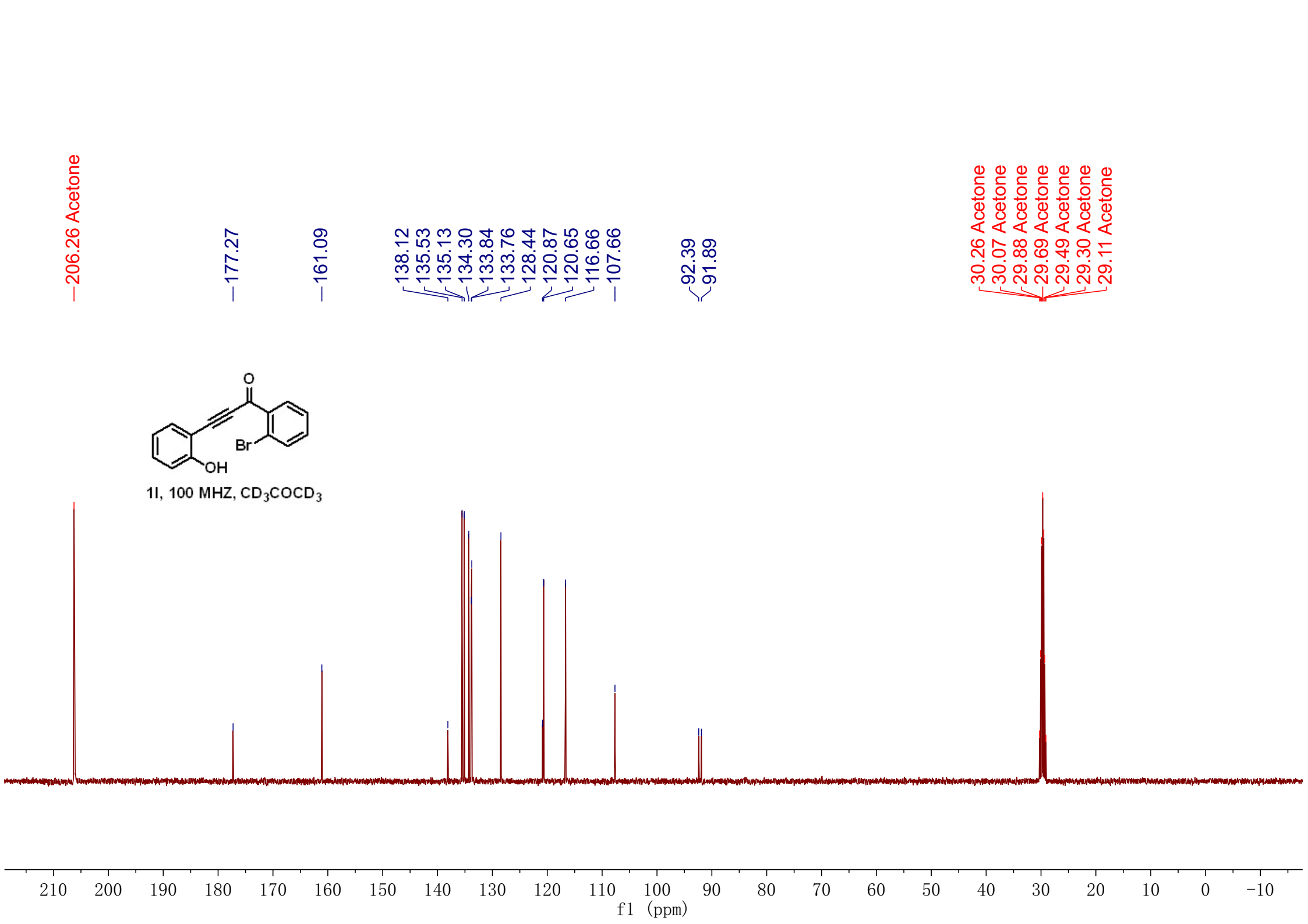
ZGQ-P41-1.16.1.1r



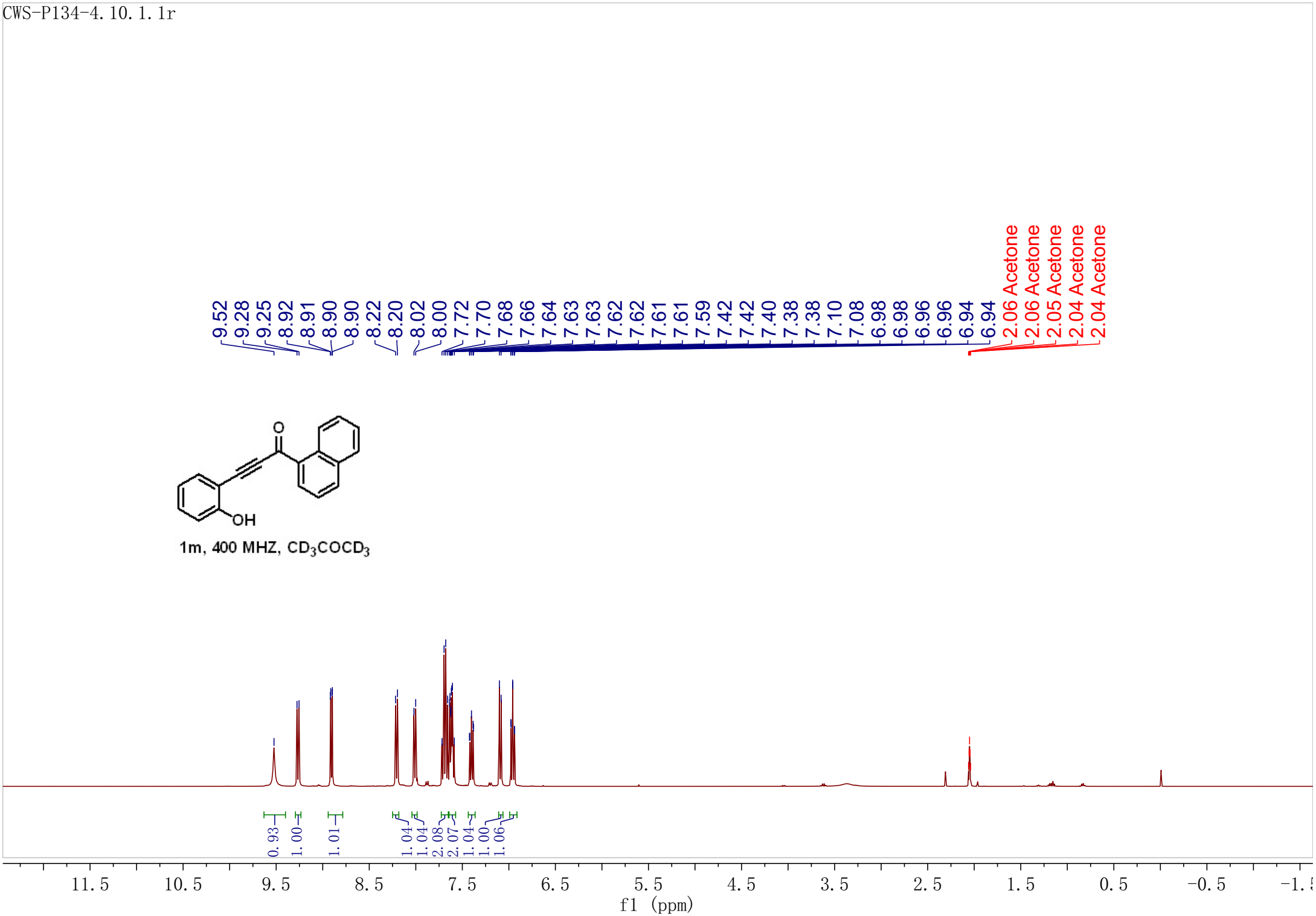
ZGQ-P41-1.17.1.1r



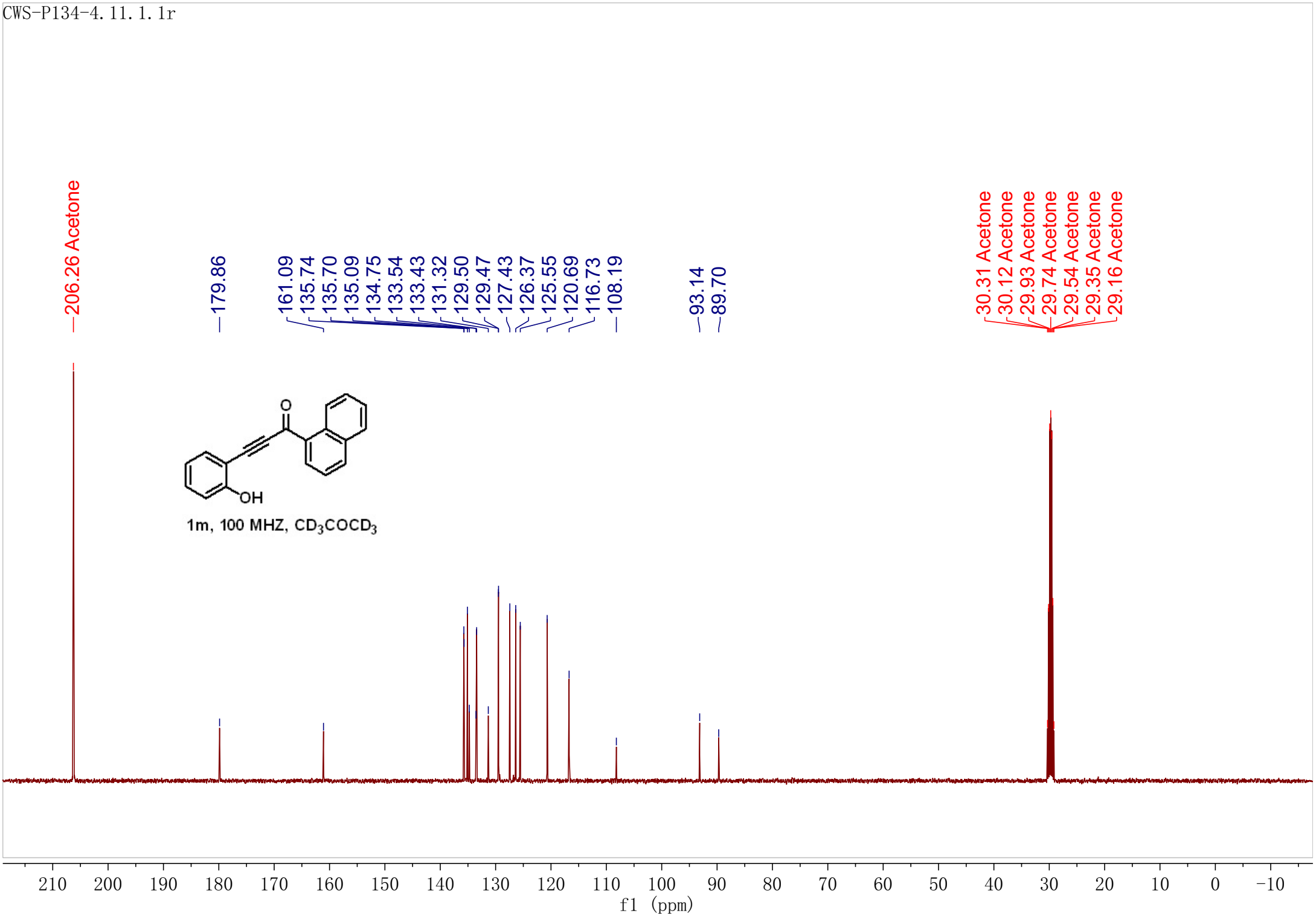

9.47
8.30
8.30
8.29
8.28
7.77
7.75
7.59
7.58
7.56
7.53
7.51
7.49
7.42
7.40
7.38
7.05
7.03
6.96
6.94
6.92
3.40 HDO
2.06 Acetone
2.06 Acetone
2.05 Acetone
2.04 Acetone
2.04 Acetone
O
Br
OH
1l, 400 MHZ, CD3COCD3
0.98
1.00
1.02
2.05
1.02
1.05
1.06
1.07
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)


206.26 Acetone
177.27
161.09
138.12
135.53
135.13
134.30
133.84
133.76
128.44
120.87
120.65
116.66
107.66
92.39
91.89
30.26 Acetone
30.07 Acetone
29.88 Acetone
29.69 Acetone
29.49 Acetone
29.30 Acetone
29.11 Acetone
O
Br
OH
1l, 100 MHZ, CD3COCD3
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)

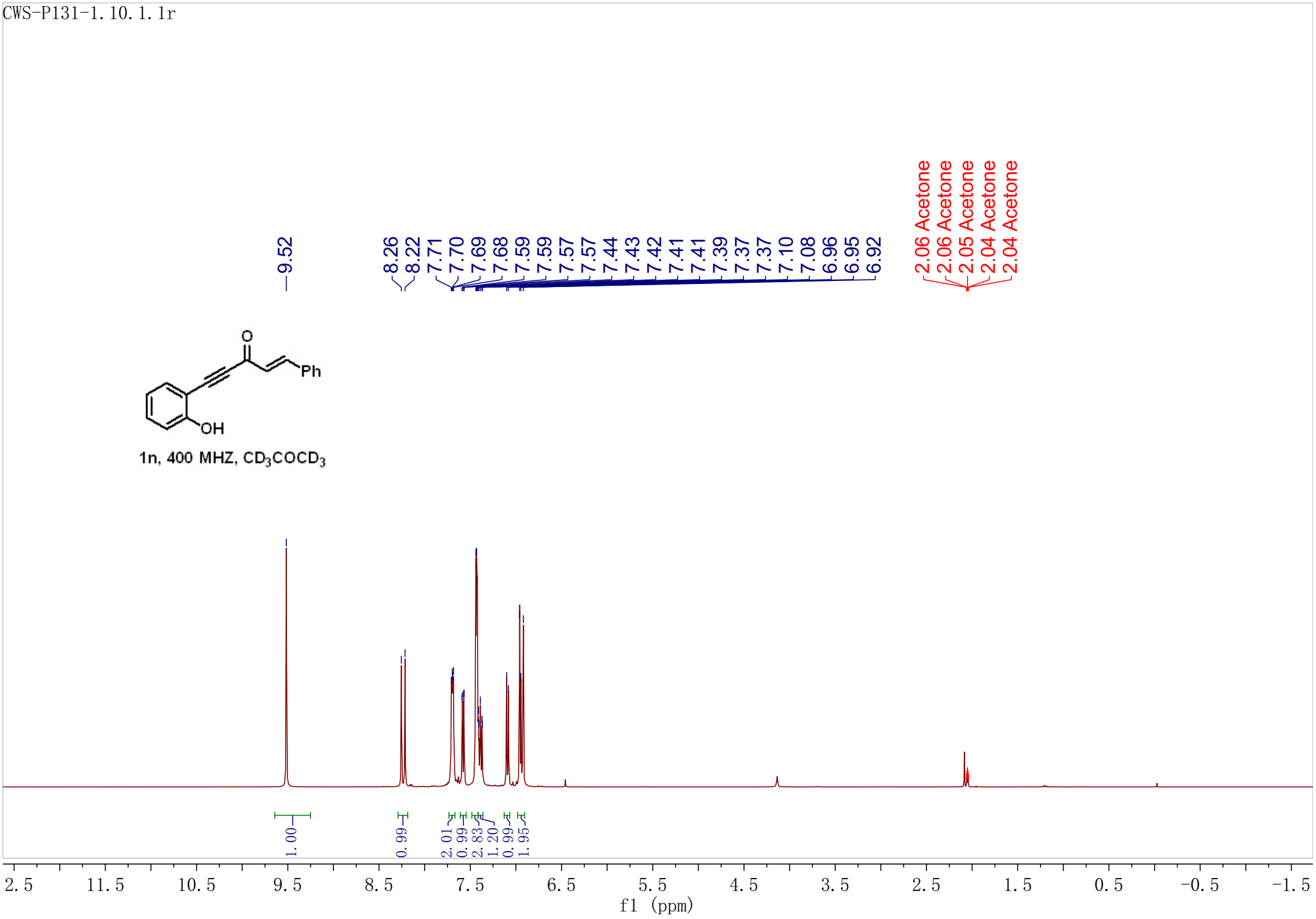
CWS-P134-4.10.1.1r



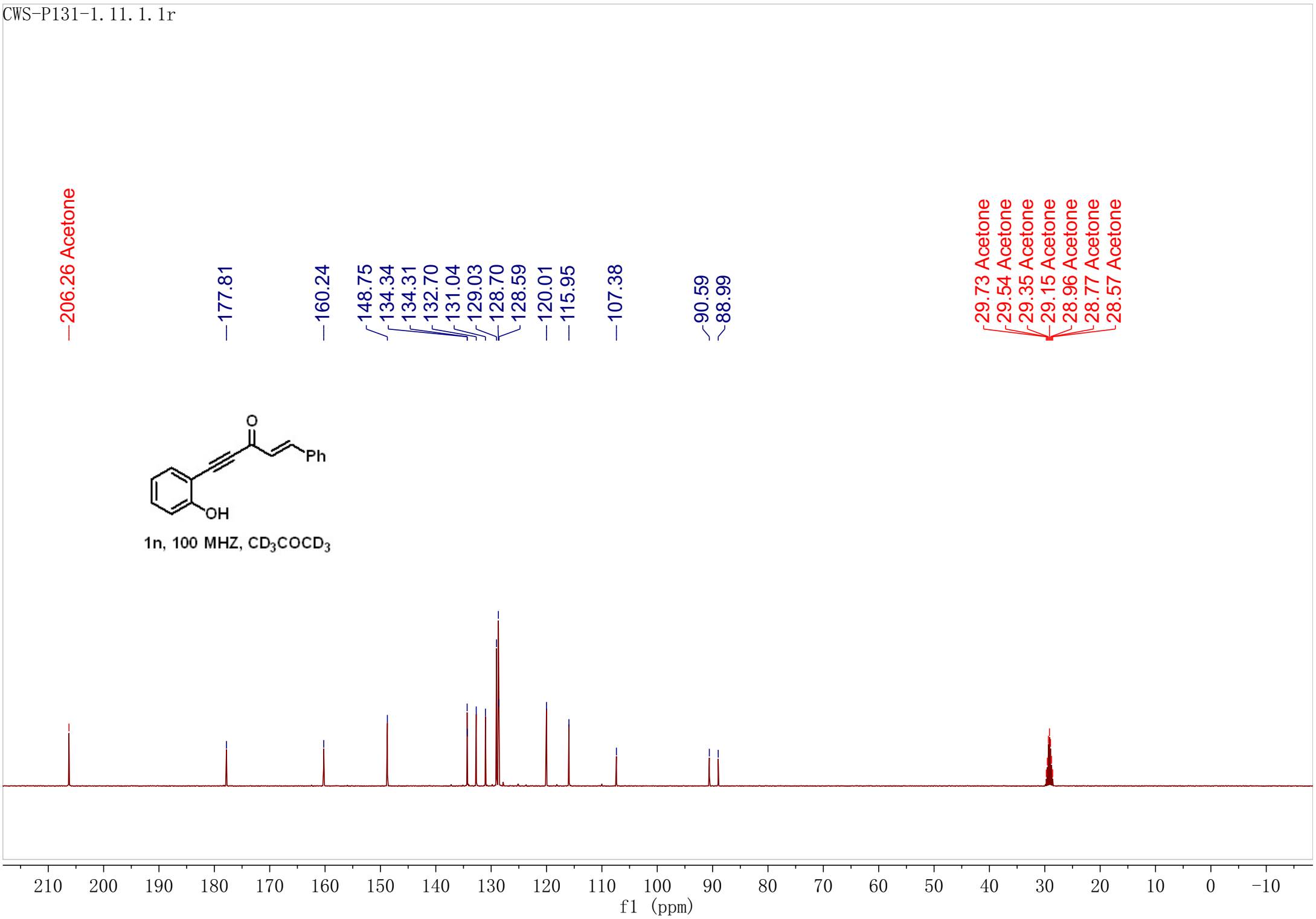
CWS-P134-4.11.1.1r

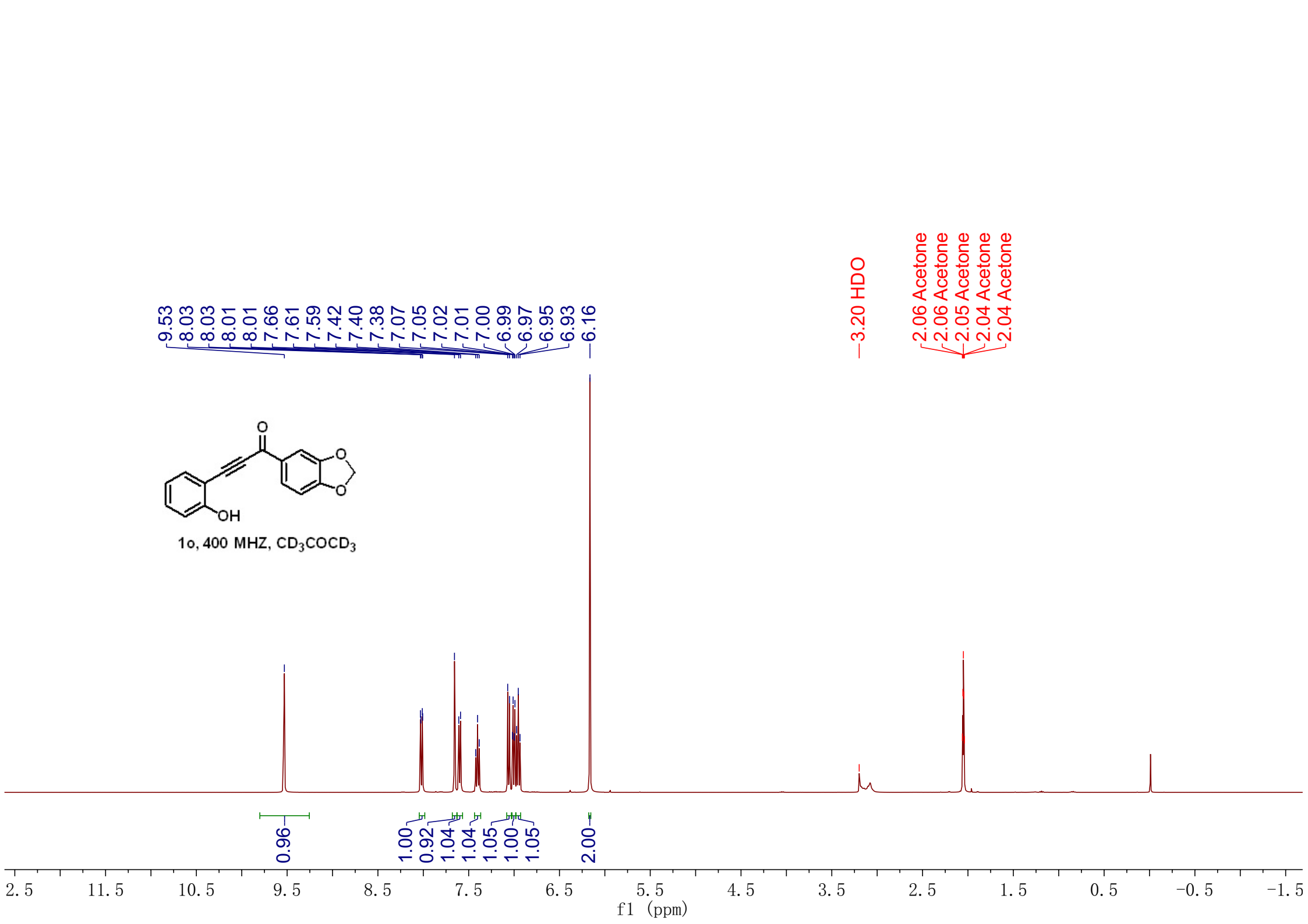


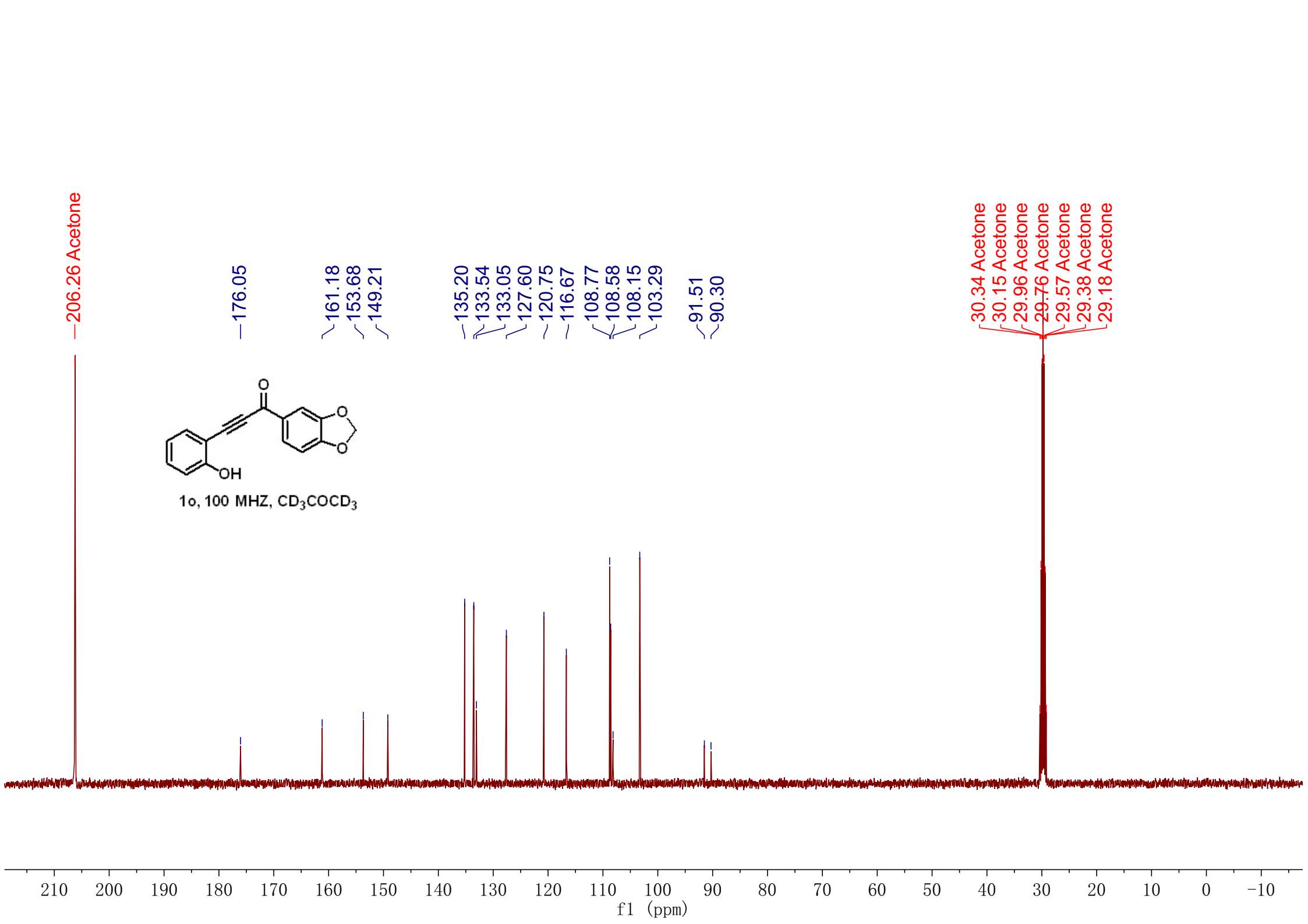
CWS-P131-1.10.1.1r

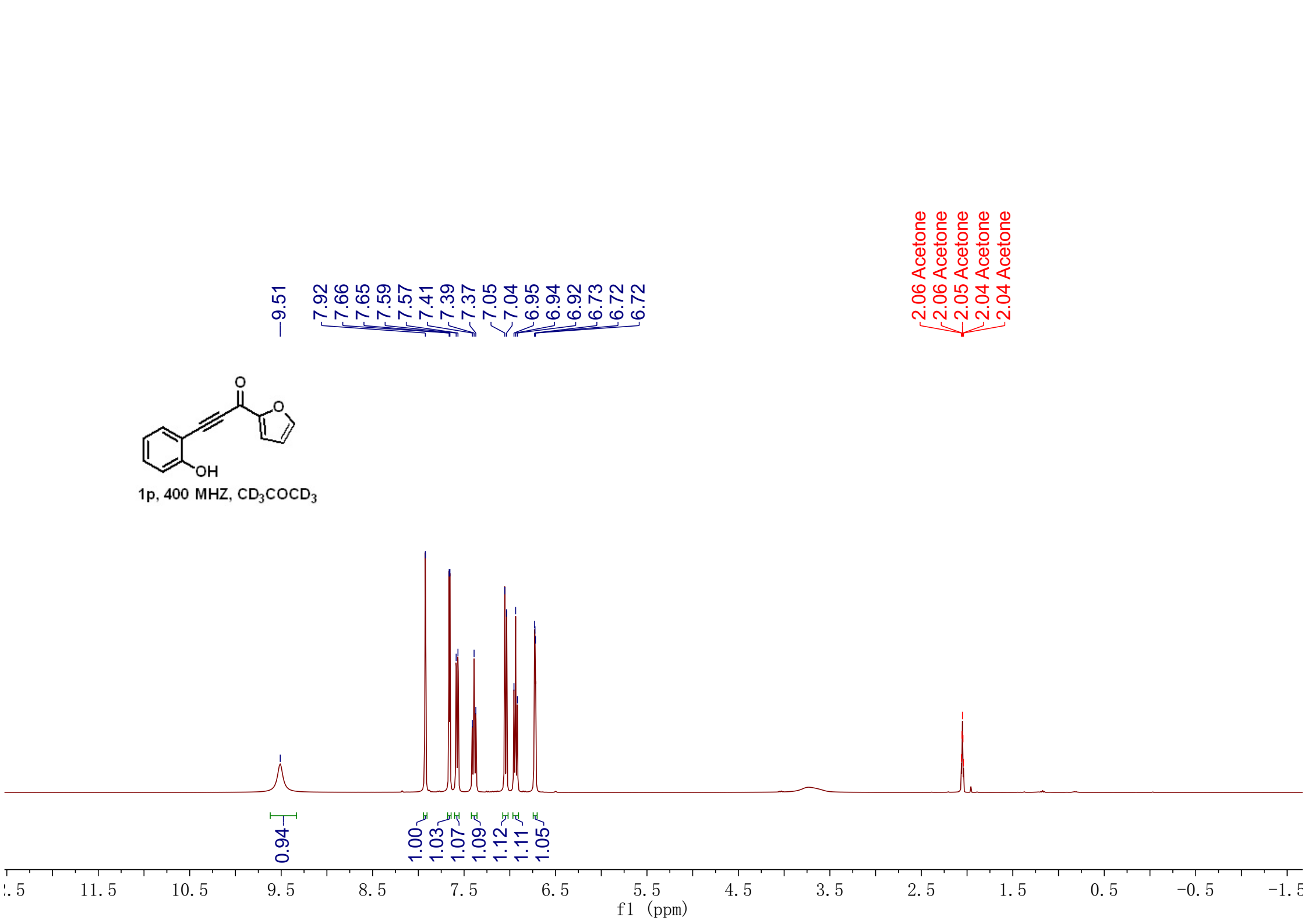


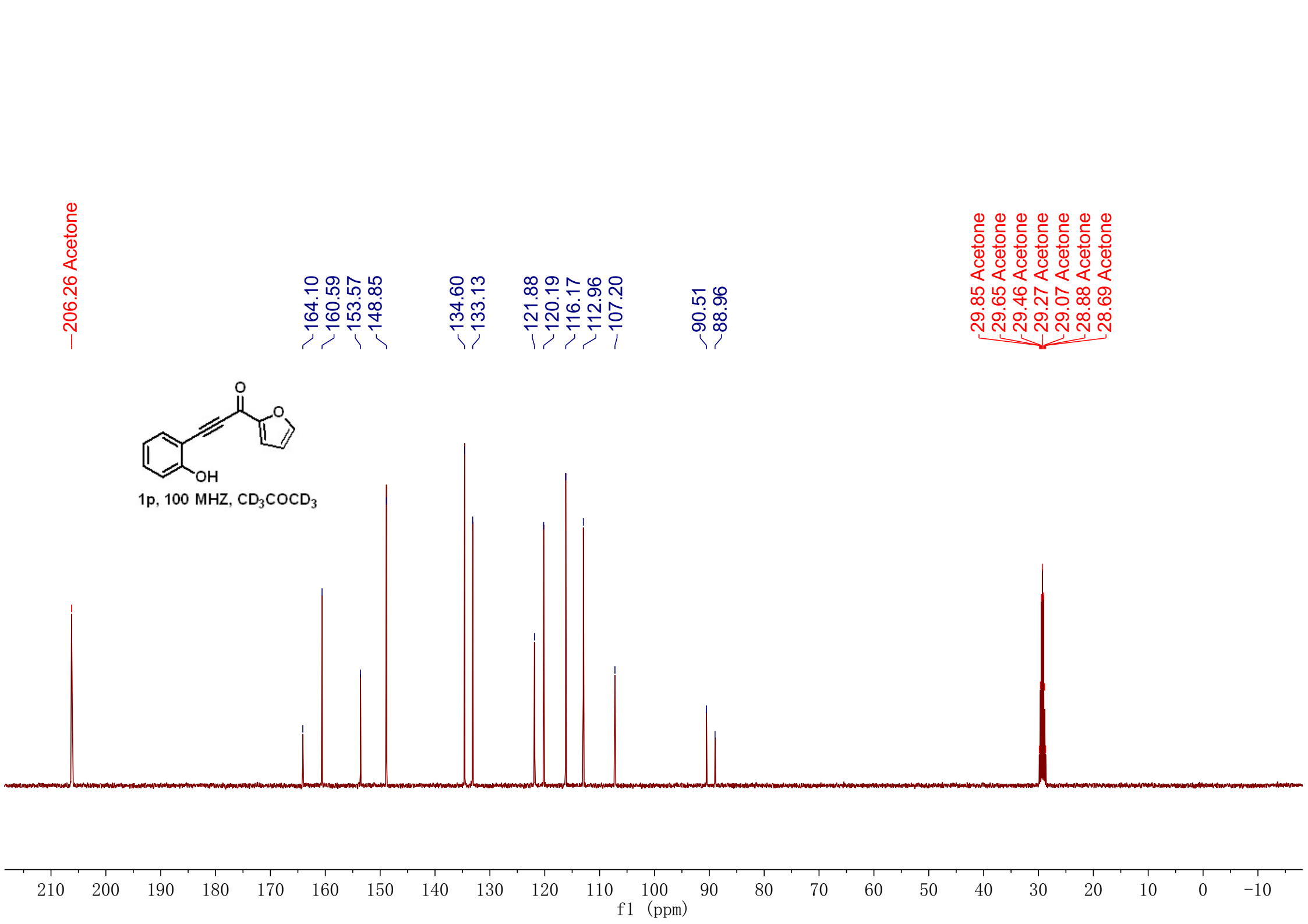
CWS-P131-1.11.1.1r



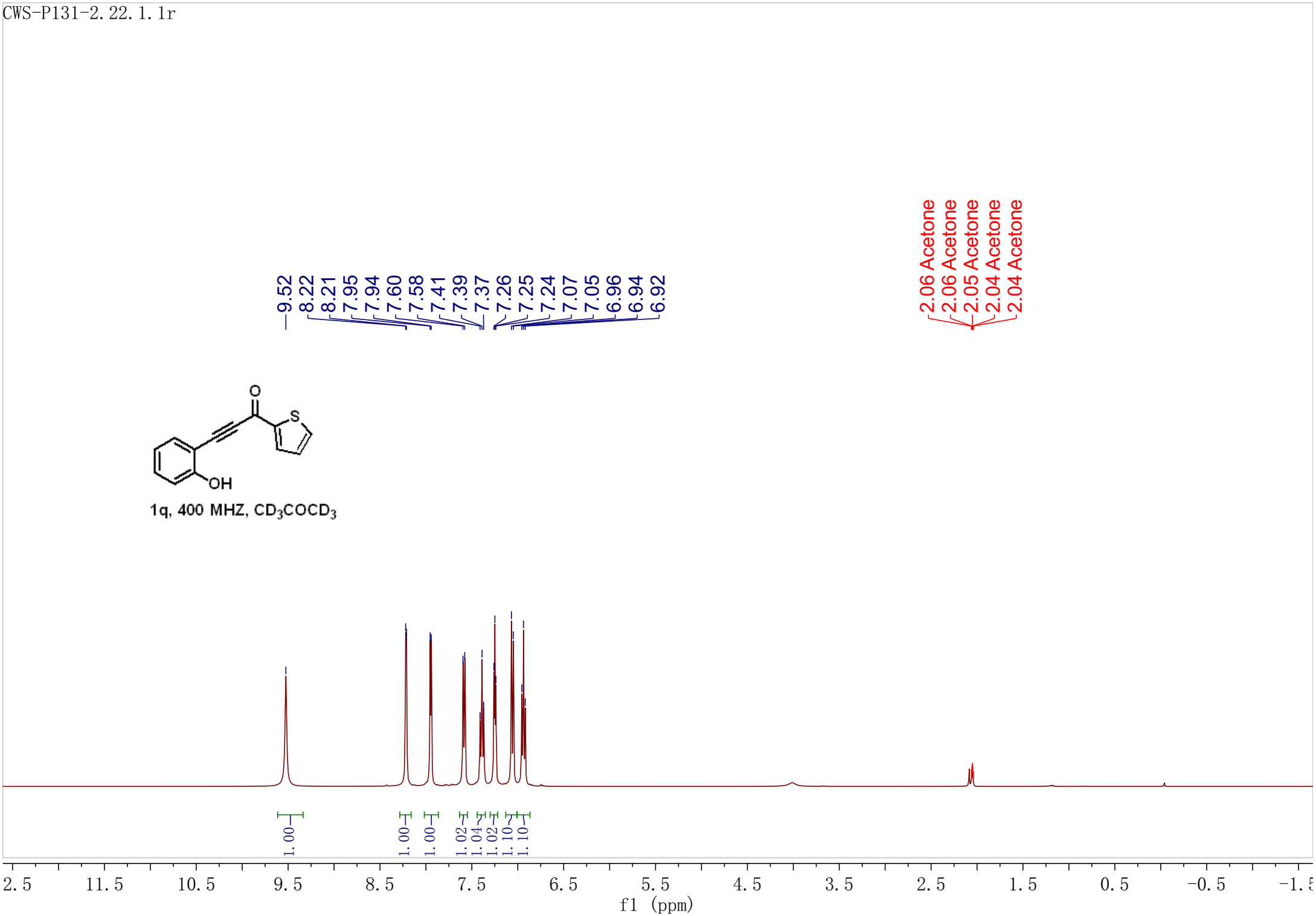

9.53
8.03
8.03
8.01
8.01
7.66
7.61
7.59
7.42
7.40
7.38
7.07
7.05
7.02
7.01
7.00
6.99
6.97
6.95
6.93
6.16
3.20 HDO
2.06 Acetone
2.06 Acetone
2.05 Acetone
2.04 Acetone
2.04 Acetone
O
O
O
OH
1o, 400 MHZ, CD3COCD3
0.96
1.00
0.92
1.04
1.04
1.05
1.00
1.05
2.00
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)


206.26 Acetone
176.05
161.18
153.68
149.21
135.20
133.54
133.05
127.60
120.75
116.67
108.77
108.58
108.15
103.29
91.51
90.30
30.34 Acetone
30.15 Acetone
29.96 Acetone
29.76 Acetone
29.57 Acetone
29.38 Acetone
29.18 Acetone
O
O
O
OH
1o, 100 MHZ, CD3COCD3
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)

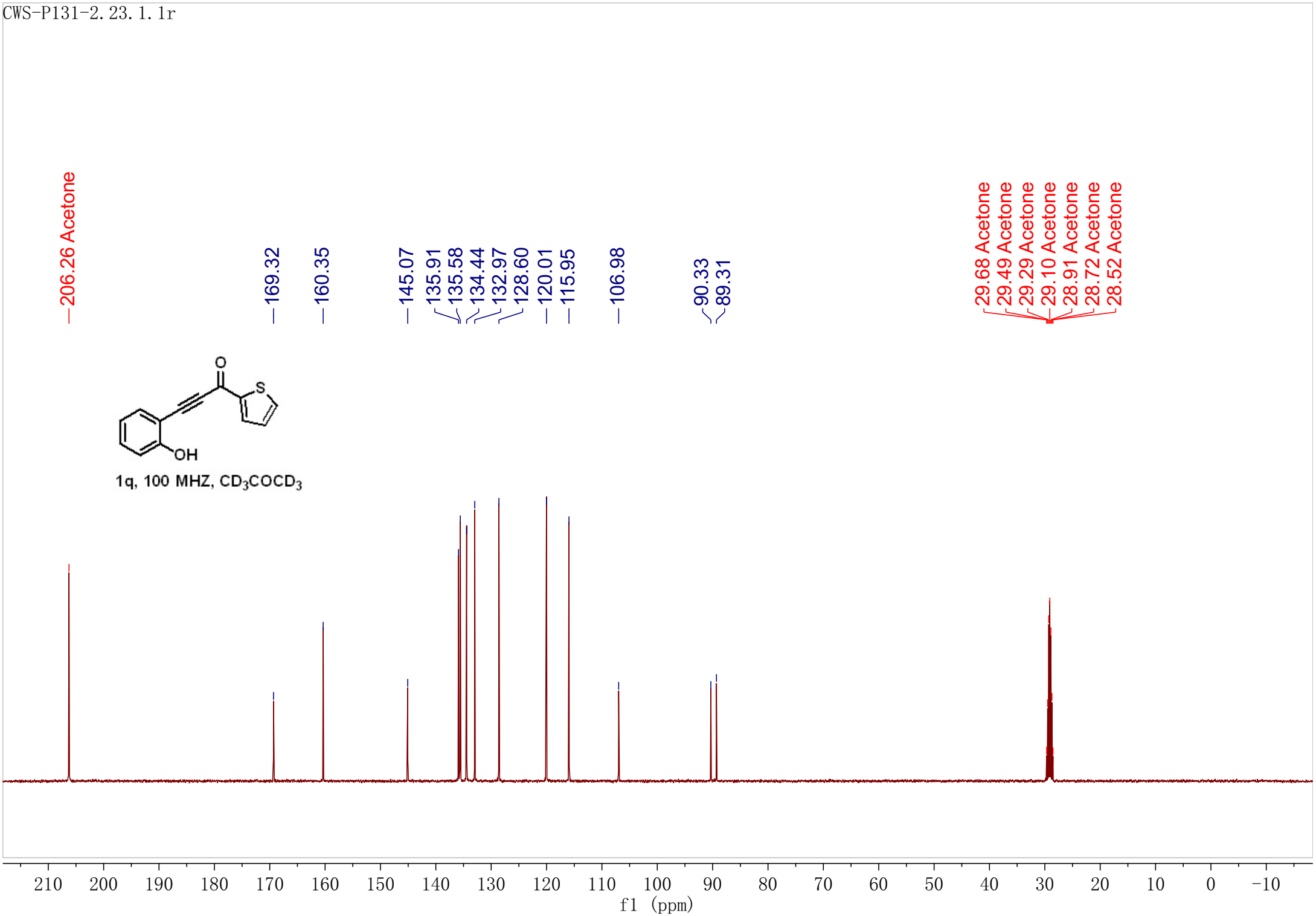

9.51
7.92
7.66
7.65
7.59
7.57
7.41
7.39
7.37
7.05
7.04
6.95
6.94
6.92
6.73
6.72
6.72
2.06 Acetone
2.06 Acetone
2.05 Acetone
2.04 Acetone
2.04 Acetone
O
O
OH
1p, 400 MHZ, CD3COCD3
0.94
1.00
1.03
1.07
1.09
1.12
1.11
1.05
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

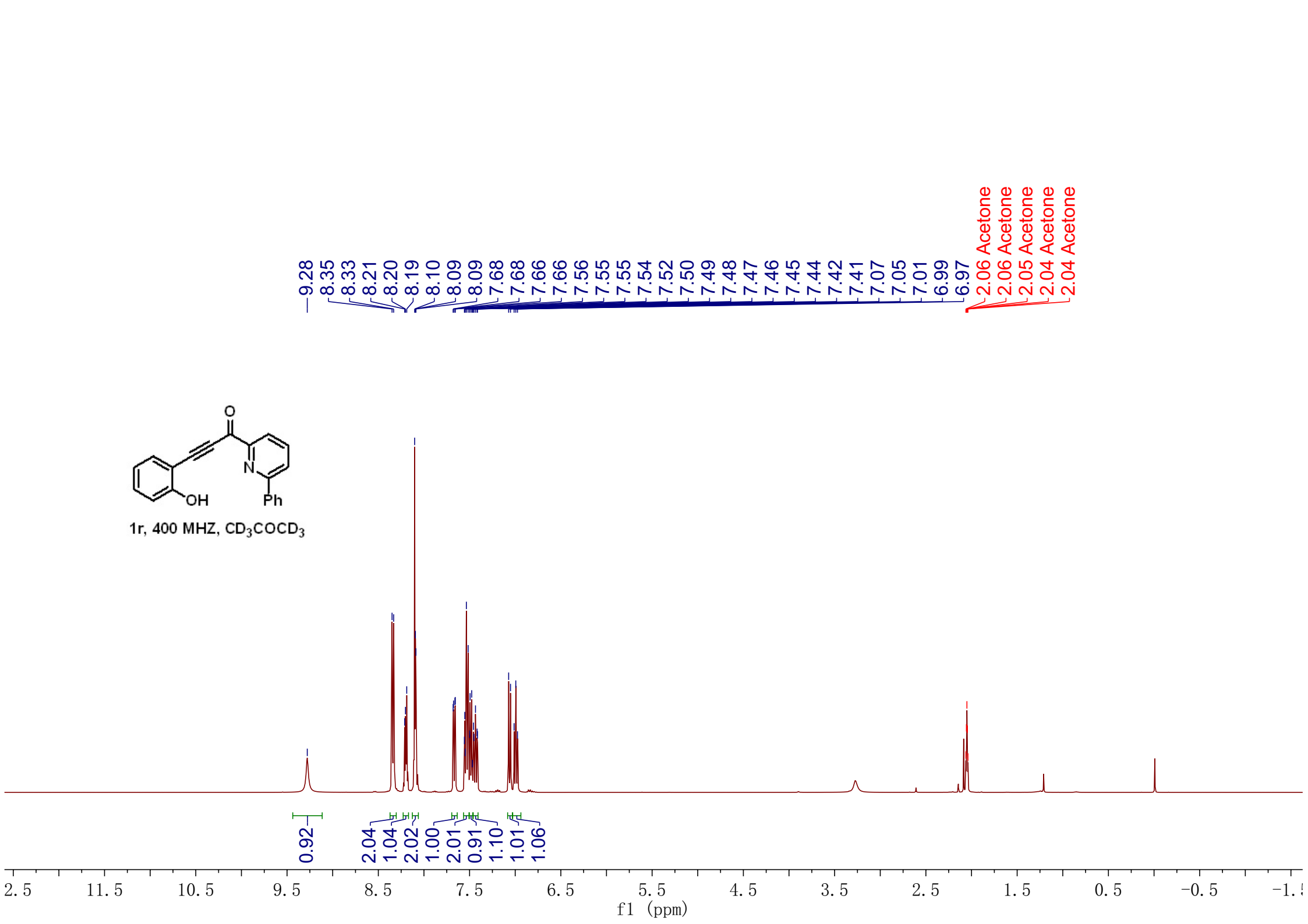

206.26 Acetone
164.10
160.59
153.57
148.85
134.60
133.13
121.88
120.19
116.17
112.96
107.20
90.51
88.96
29.85 Acetone
29.65 Acetone
29.46 Acetone
29.27 Acetone
29.07 Acetone
28.88 Acetone
28.69 Acetone
O
O
OH
1p, 100 MHZ, CD3COCD3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

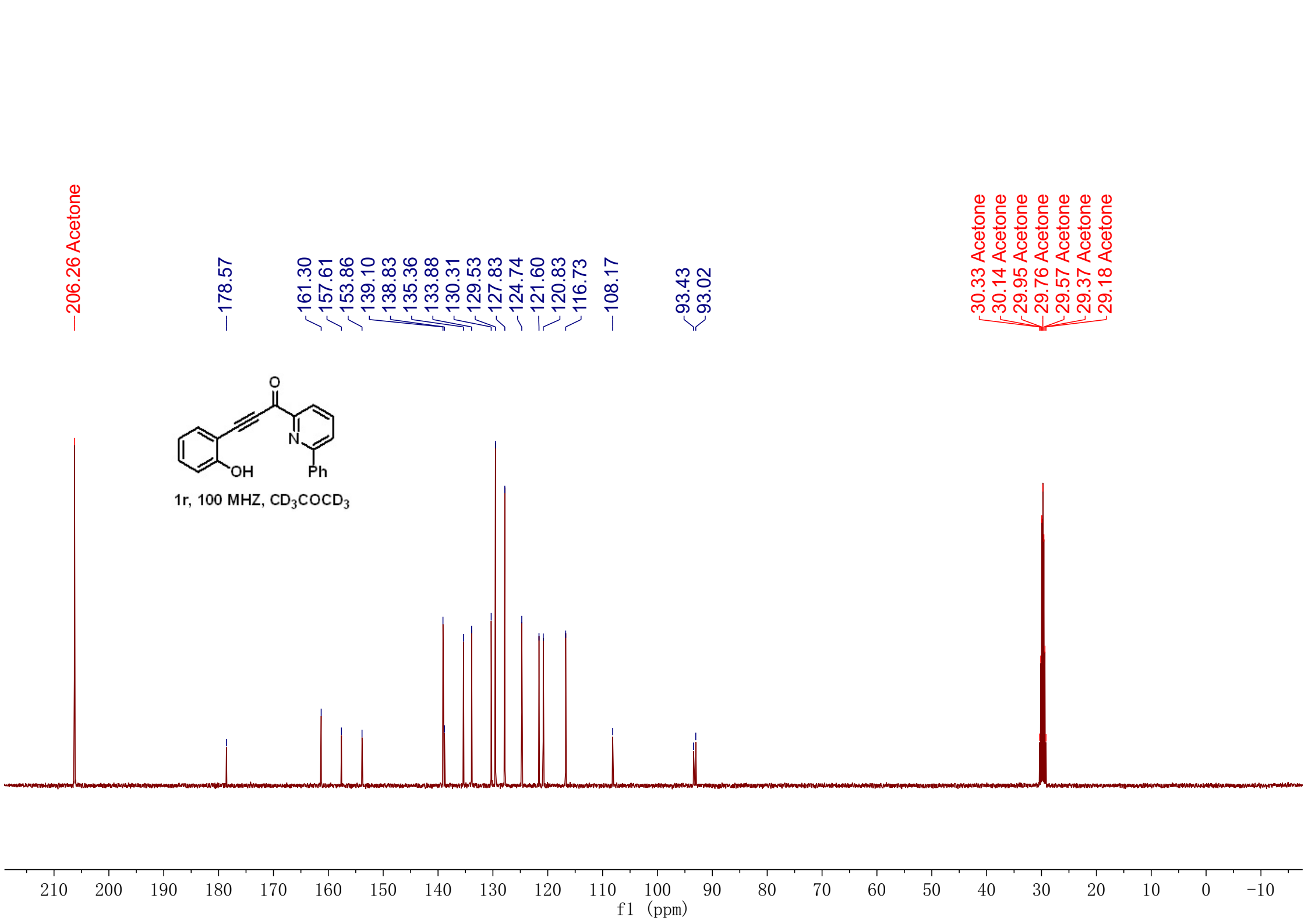
CWS-P131-2.22.1.1r



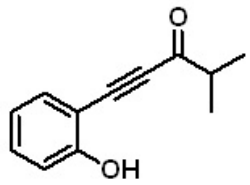
CWS-P131-2.23.1.1r




9.28
8.35
8.33
8.21
8.20
8.19
8.10
8.09
8.09
7.68
7.68
7.66
7.66
7.56
7.55
7.55
7.54
7.52
7.50
7.49
7.48
7.47
7.46
7.45
7.44
7.42
7.41
7.07
7.05
7.01
6.99
6.97
2.06 Acetone
2.06 Acetone
2.05 Acetone
2.04 Acetone
2.04 Acetone
O
N
OH
Ph
1r, 400 MHZ, CD3COCD3
0.92
2.04
1.04
2.02
1.00
2.01
0.91
1.10
1.01
1.06
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

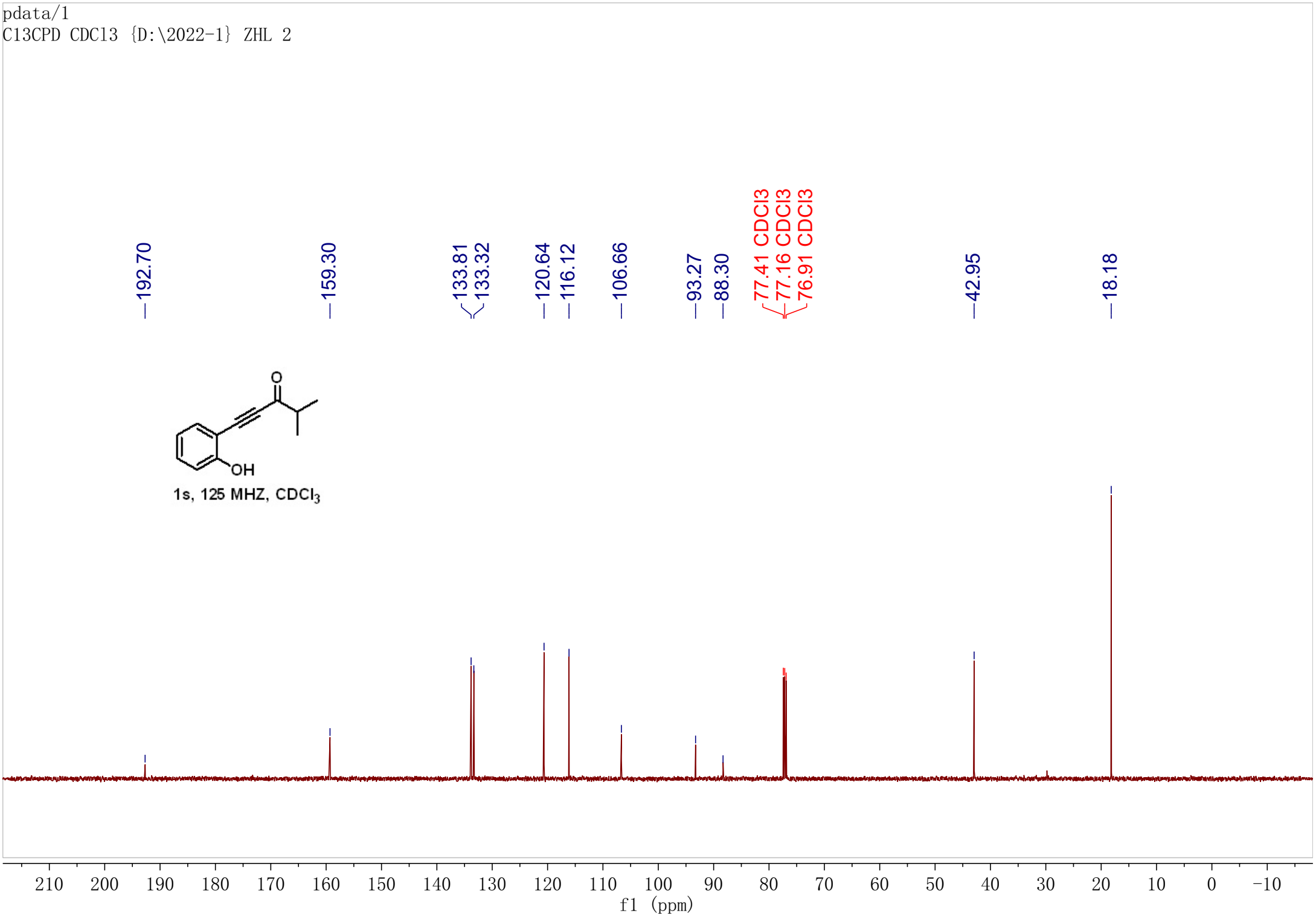

206.26 Acetone
178.57
161.30
157.61
153.86
139.10
138.83
135.36
133.88
130.31
129.53
127.83
124.74
121.60
120.83
116.73
108.17
93.43
93.02
30.33 Acetone
30.14 Acetone
29.95 Acetone
29.76 Acetone
29.57 Acetone
29.37 Acetone
29.18 Acetone
O
N
OH
Ph
1r, 100 MHZ, CD3COCD3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

pdata/1
PROTON CDCl3 {D:\2022-1} ZHL 2

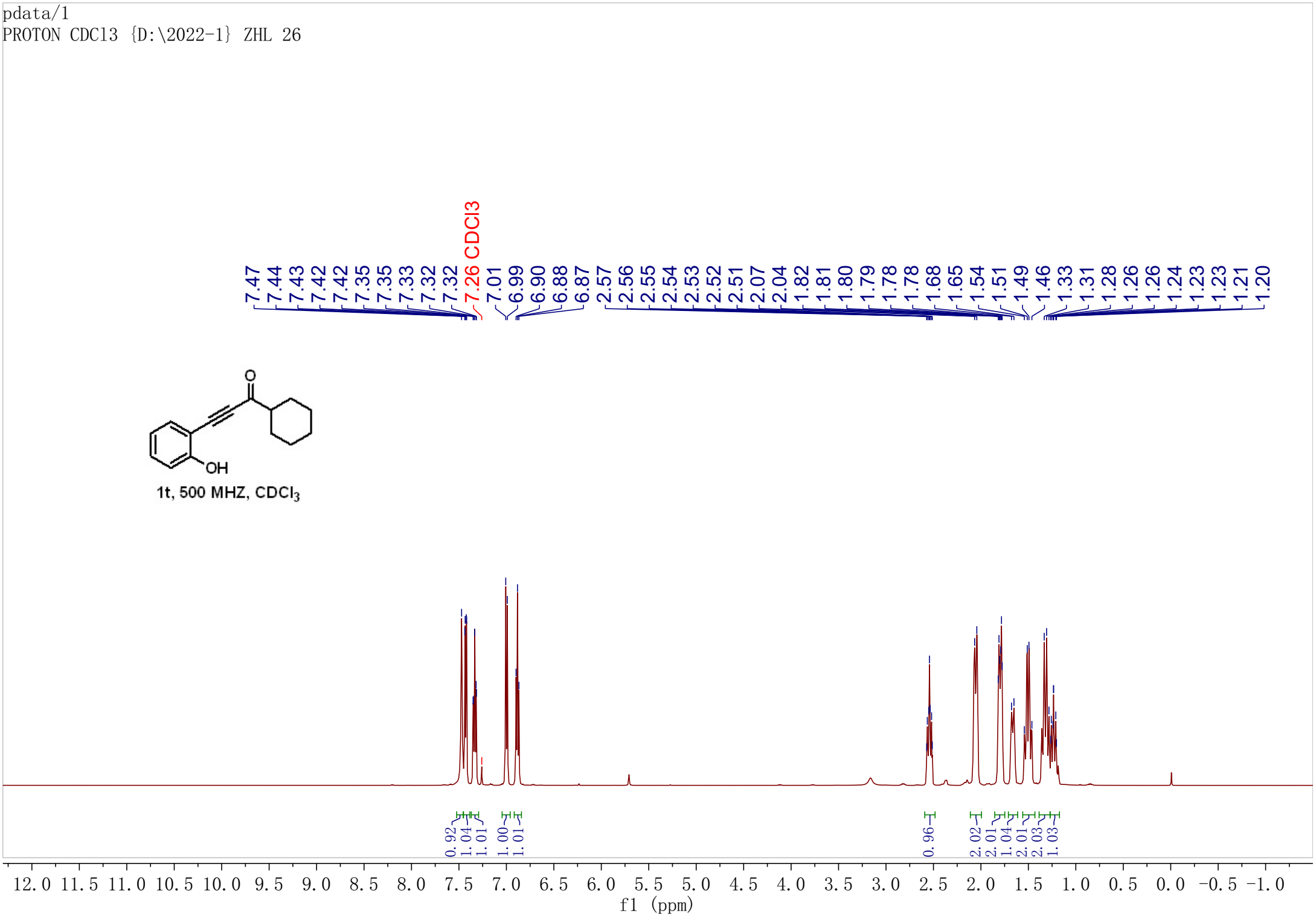


1s, 500 MHZ, $CDCl_3$

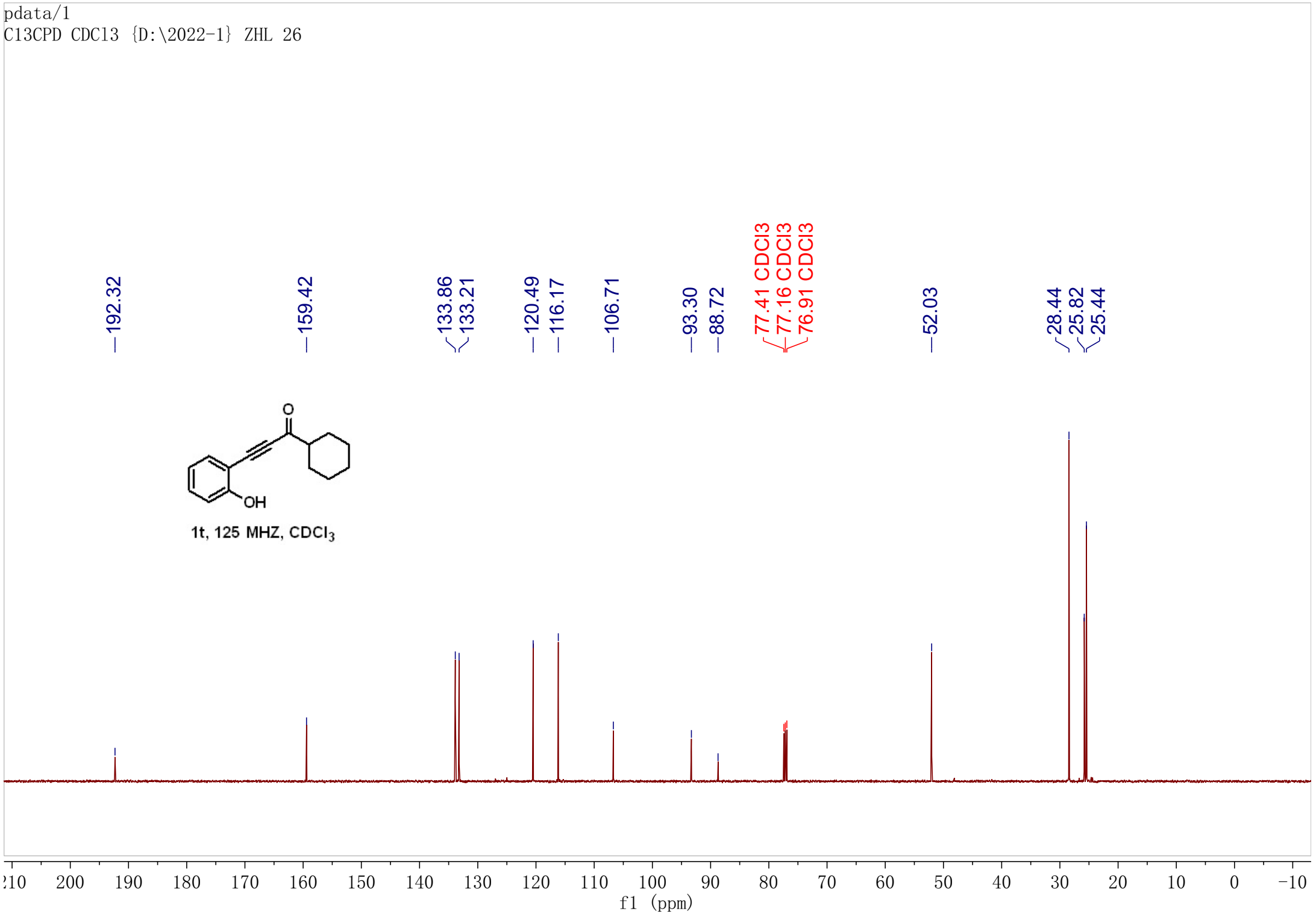
pdata/1
C13CPD CDCl3 {D:\2022-1} ZHL 2

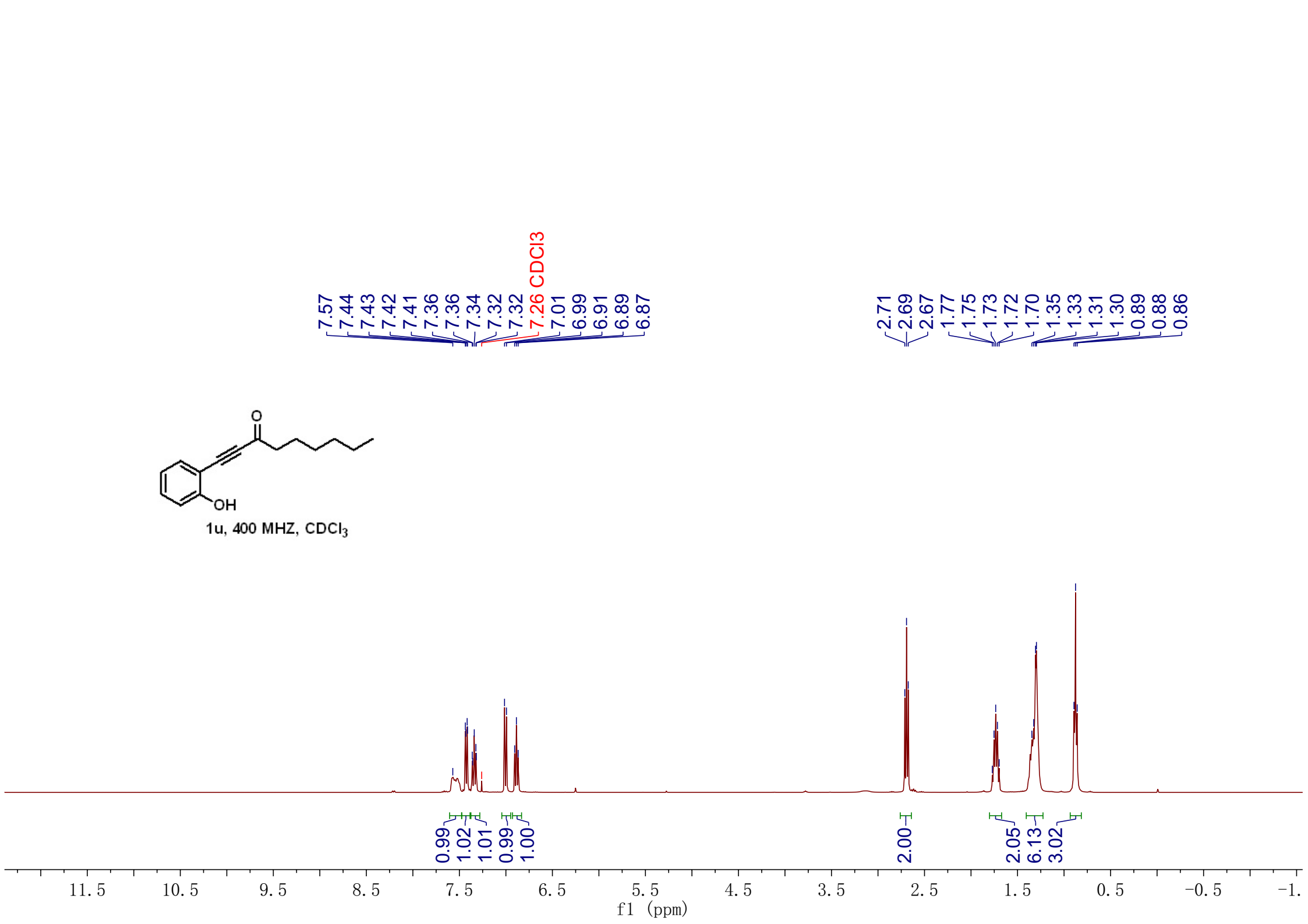


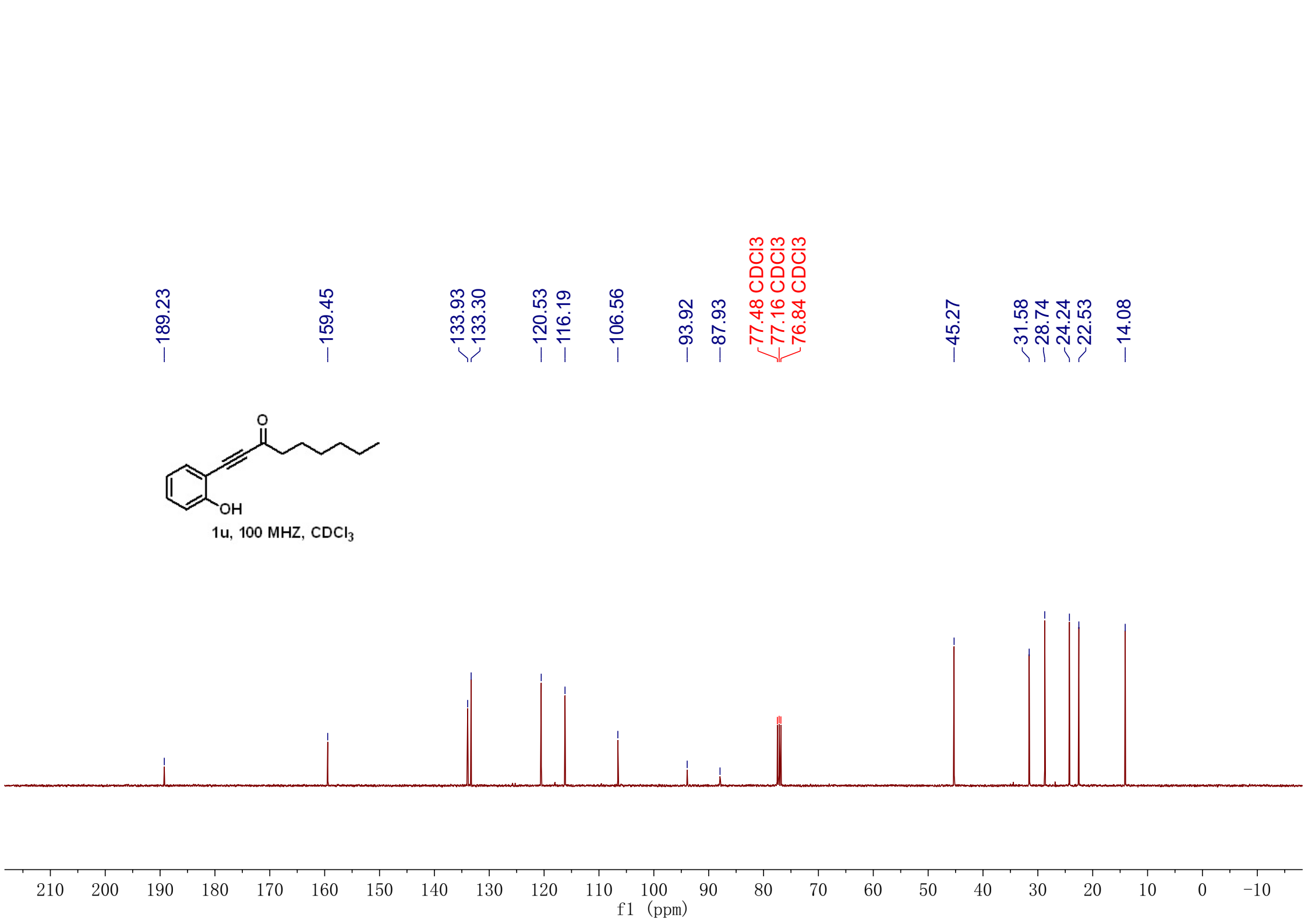
pdata/1
PROTON CDCl3 {D:\2022-1} ZHL 26



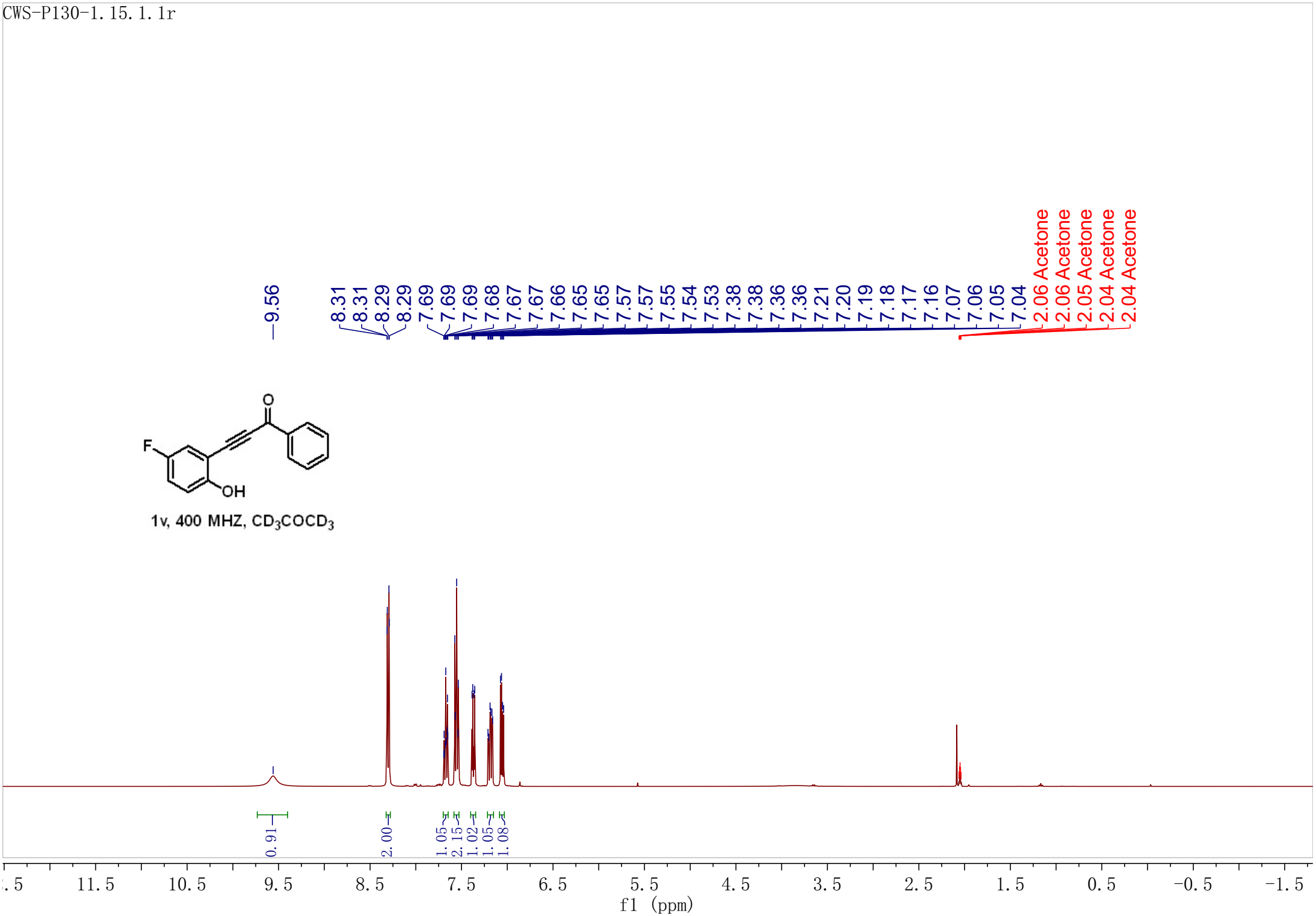
pdata/1
C13CPD CDCl3 {D:\2022-1} ZHL 26



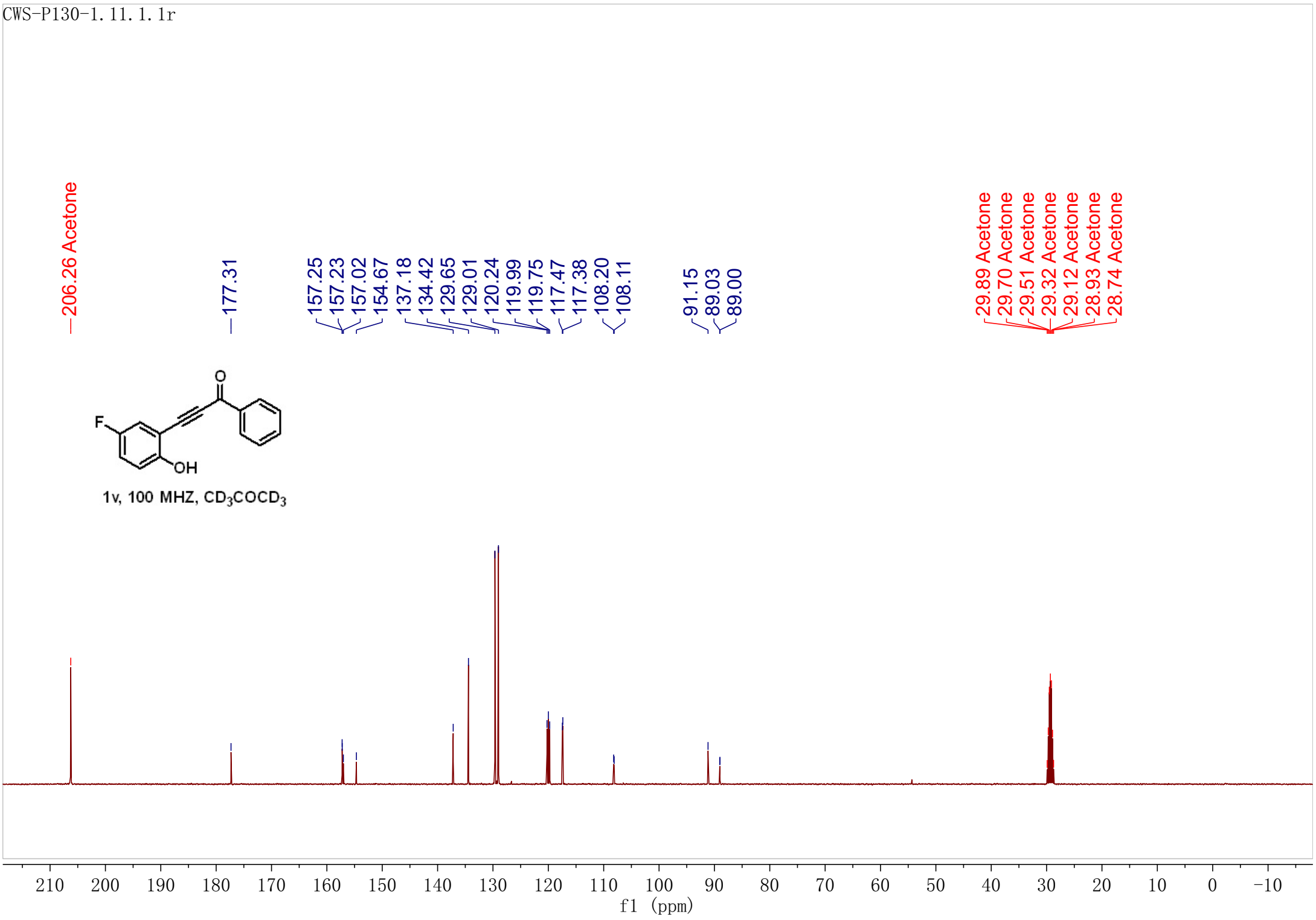

7.57
7.44
7.43
7.42
7.41
7.36
7.36
7.34
7.32
7.32
7.26 CDCl3
7.01
6.99
6.91
6.89
6.87
2.71
2.69
2.67
1.77
1.75
1.73
1.72
1.70
1.35
1.33
1.31
1.30
0.89
0.88
0.86
O
OH
1u, 400 MHZ, CDCl3
0.99
1.02
1.01
0.99
1.00
2.00
2.05
6.13
3.02
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)


189.23
159.45
133.93
133.30
120.53
116.19
106.56
93.92
87.93
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
45.27
31.58
28.74
24.24
22.53
14.08
O
OH
1u, 100 MHZ, CDCl3
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)

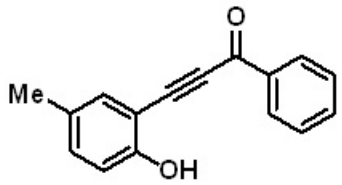
CWS-P130-1.15.1.1r



CWS-P130-1.11.1.1r



CWS-P130-2.10.1.1r



1w, 400 MHZ, CD_3COCD_3

9.27
8.32
8.32
8.30
8.30
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.57
7.57
7.51
7.49
7.47
7.29
7.29
7.13
7.12
7.11
7.10
6.95
6.93
2.18
2.05 Acetone
2.02 Acetone
2.02 Acetone
2.01 Acetone
2.00 Acetone

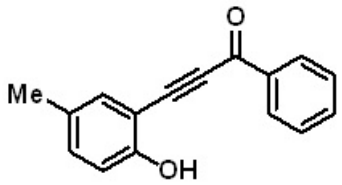
0.88
2.00
1.00
2.01
0.97
1.01
1.00
3.02

2.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.

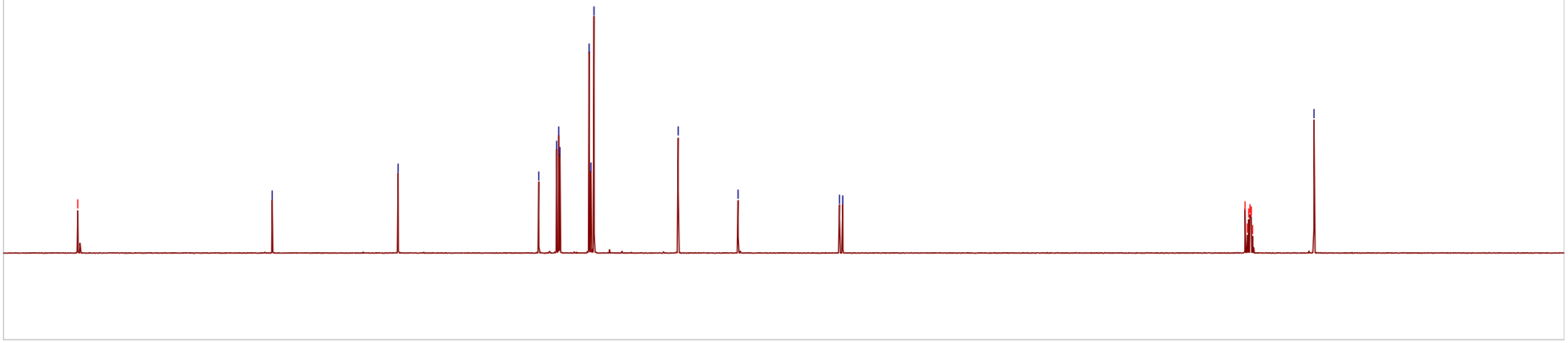
f1 (ppm)

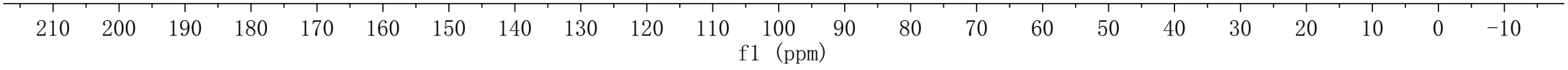
CWS-P130-2.11.1.1r

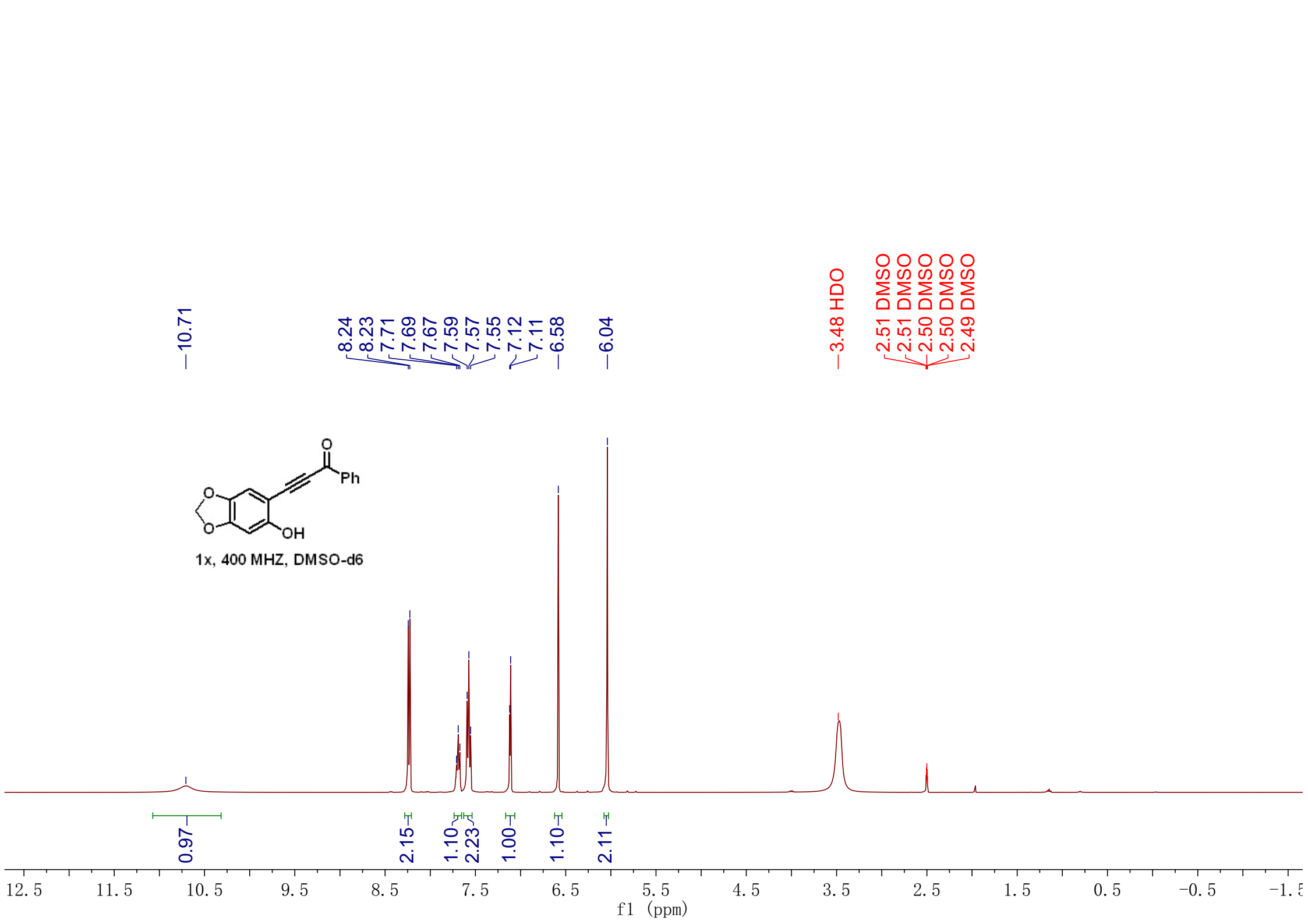
206.26 Acetone
176.79
157.70
136.38
133.67
133.35
133.17
128.75
128.47
128.00
115.24
106.16
90.80
90.29
29.31 Acetone
28.94 Acetone
28.75 Acetone
28.56 Acetone
28.36 Acetone
28.17 Acetone
18.85

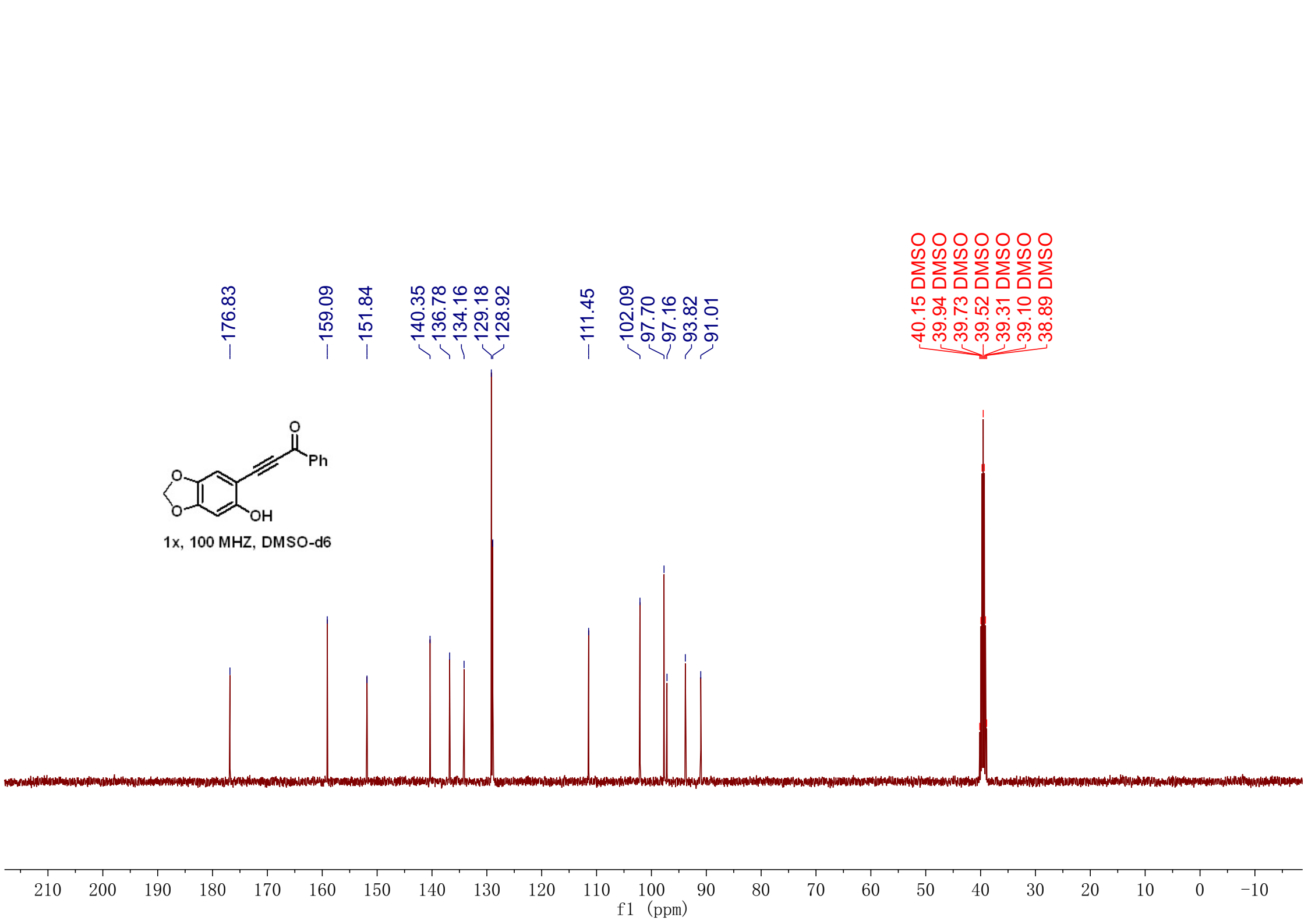


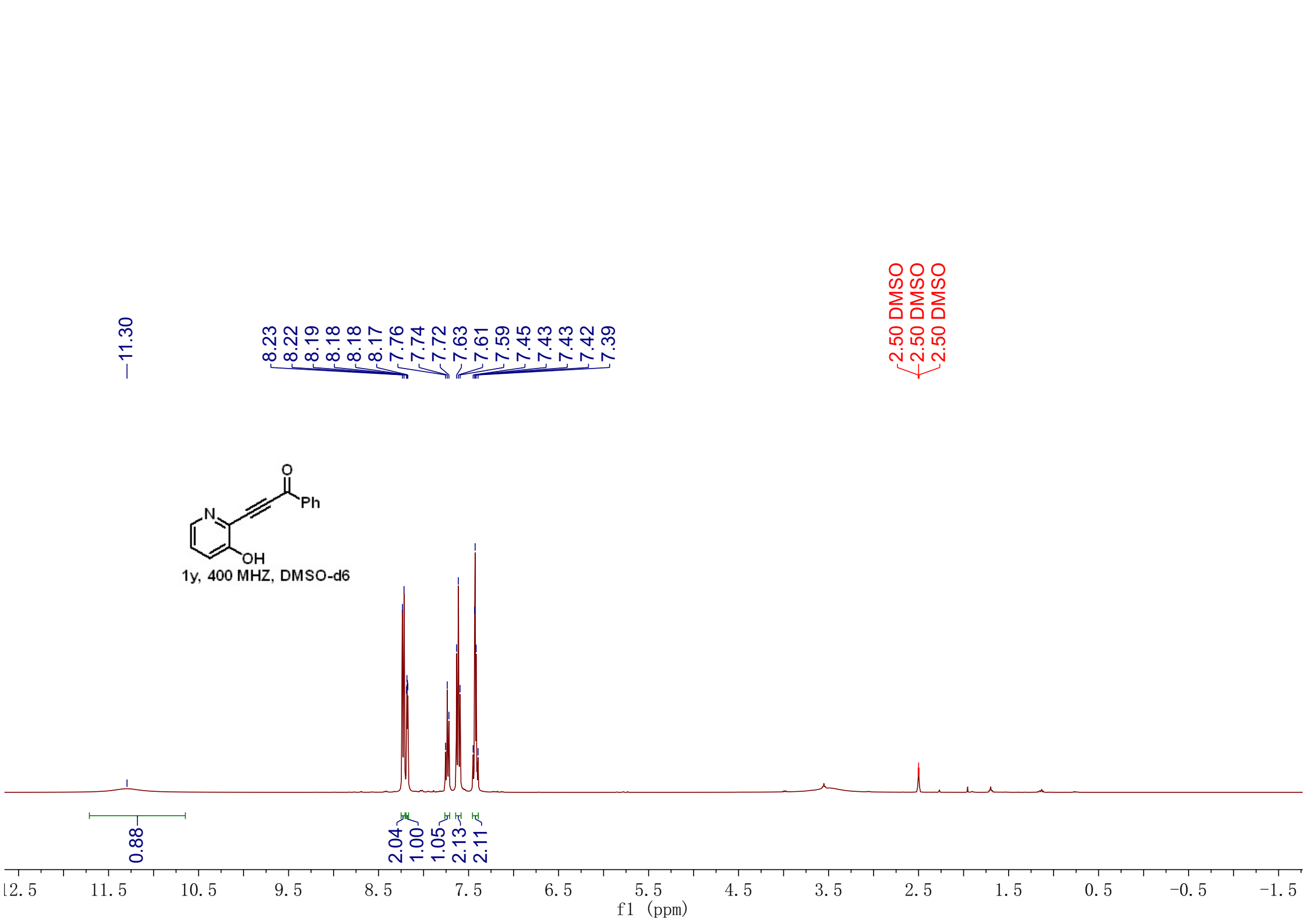
1w, 100 MHZ, CD_3COCD_3

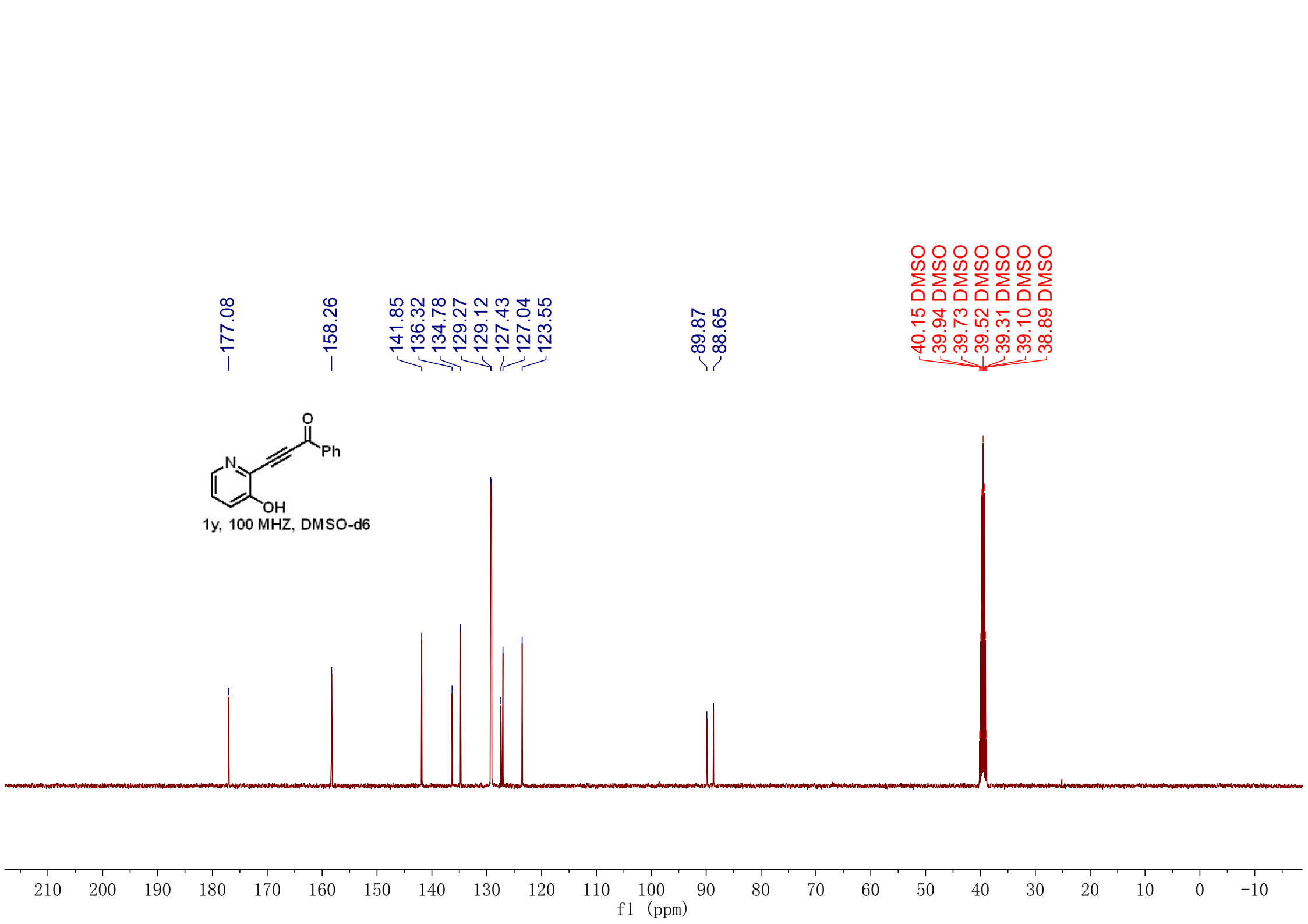


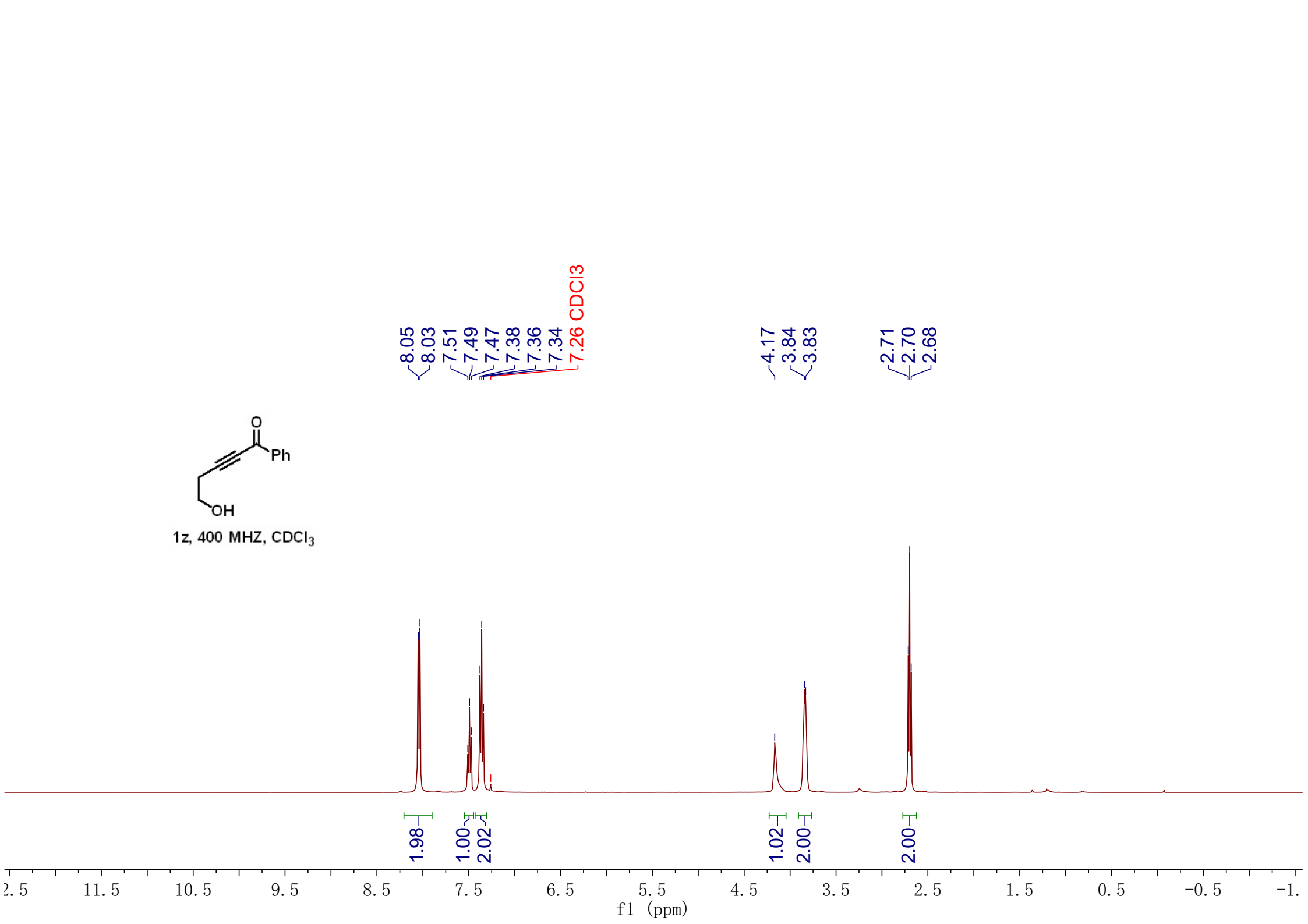


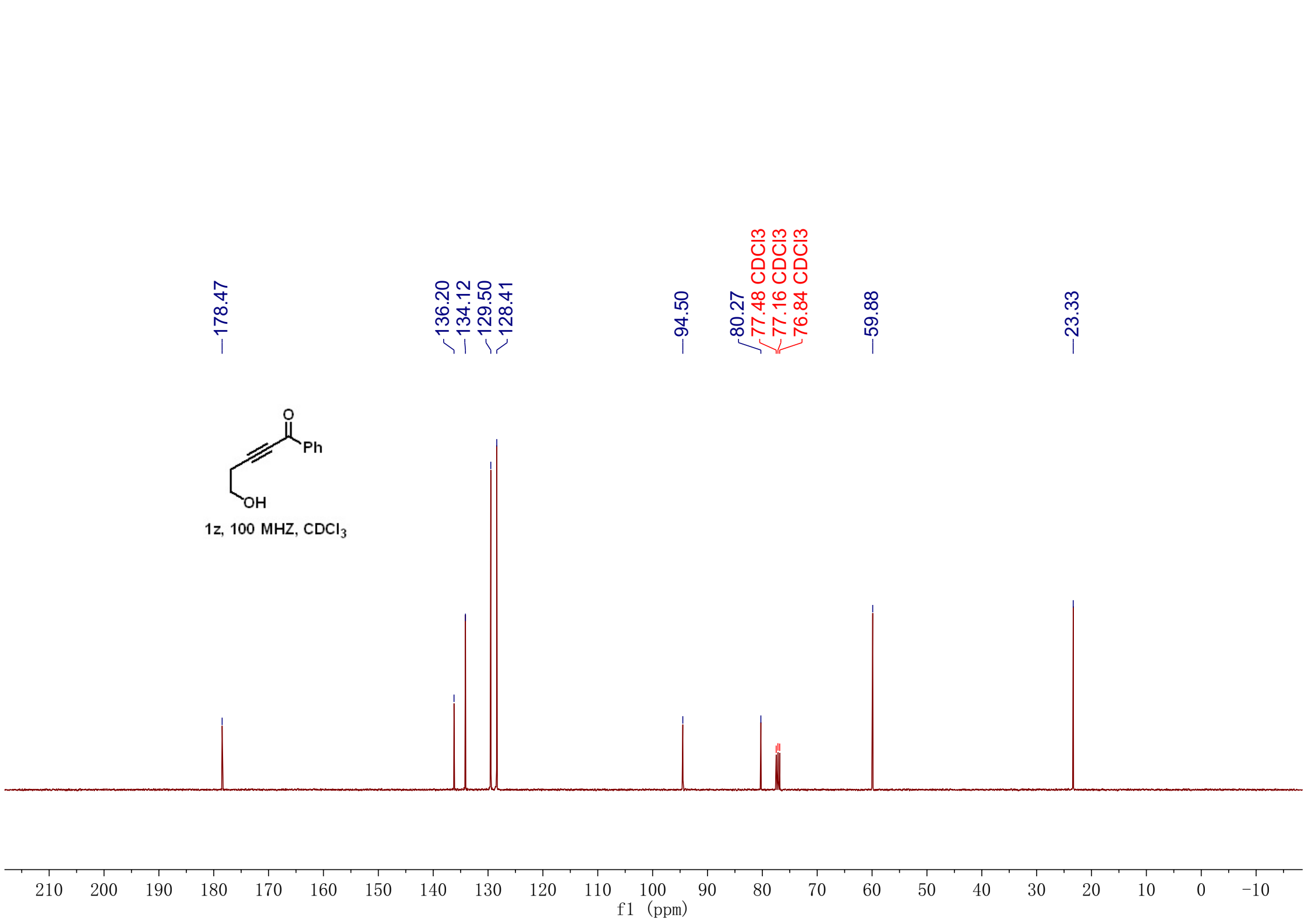

10.71
8.24
8.23
7.71
7.69
7.67
7.59
7.57
7.55
7.12
7.11
6.58
6.04
3.48 HDO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
O
Ph
O
O
OH
1x, 400 MHZ, DMSO-d6
0.97
2.15
1.10
2.23
1.00
1.10
2.11
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

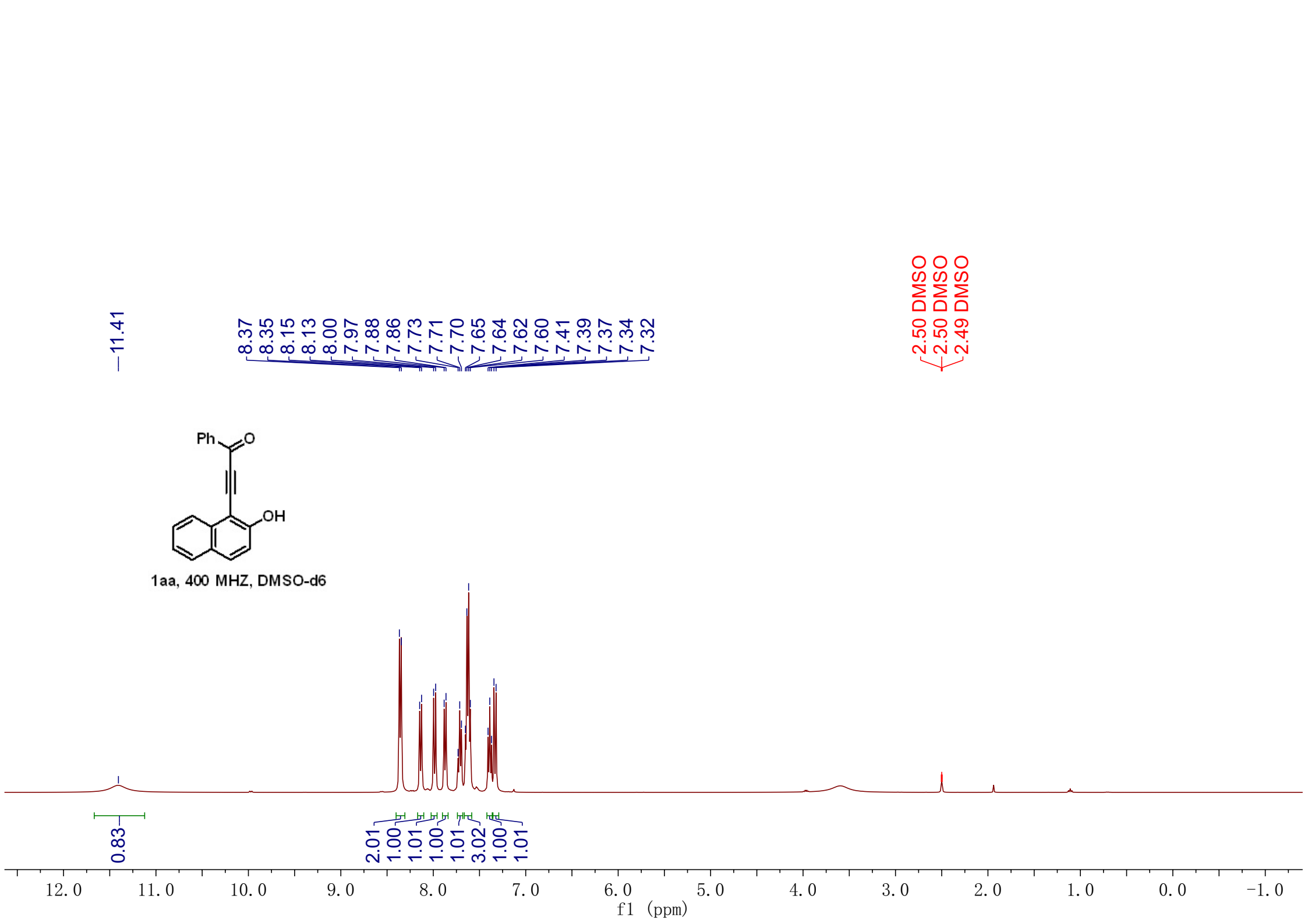

176.83
159.09
151.84
140.35
136.78
134.16
129.18
128.92
111.45
102.09
97.70
97.16
93.82
91.01
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
O
Ph
O
O
OH
1x, 100 MHZ, DMSO-d6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

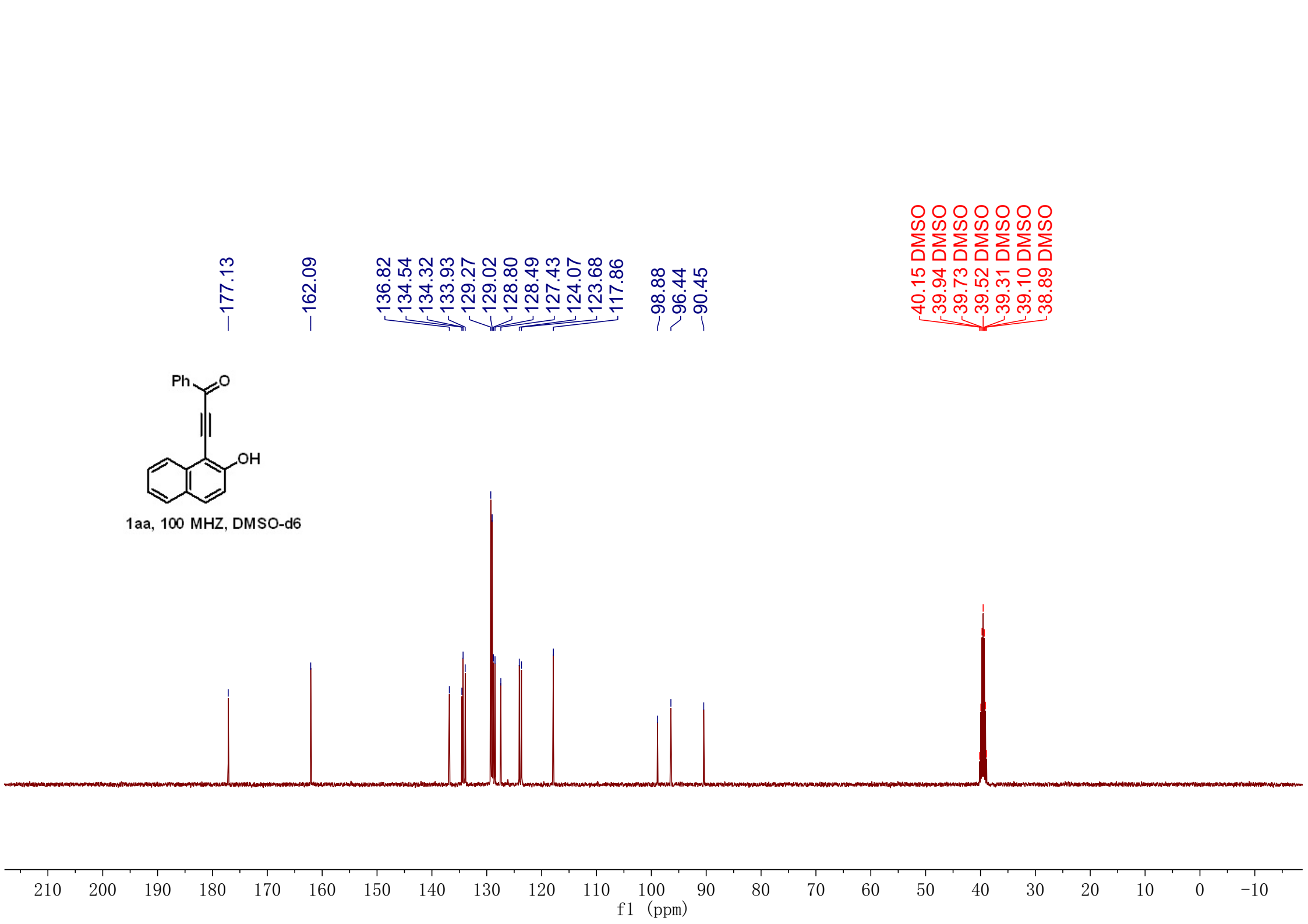

11.30
8.23
8.22
8.19
8.18
8.18
8.17
7.76
7.74
7.72
7.63
7.61
7.59
7.45
7.43
7.43
7.42
7.39
2.50 DMSO
2.50 DMSO
2.50 DMSO
O
Ph
N
OH
1y, 400 MHZ, DMSO-d6
0.88
2.04
1.00
1.05
2.13
2.11
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)


177.08
158.26
141.85
136.32
134.78
129.27
129.12
127.43
127.04
123.55
89.87
88.65
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
O
Ph
N
OH
1y, 100 MHZ, DMSO-d6
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)

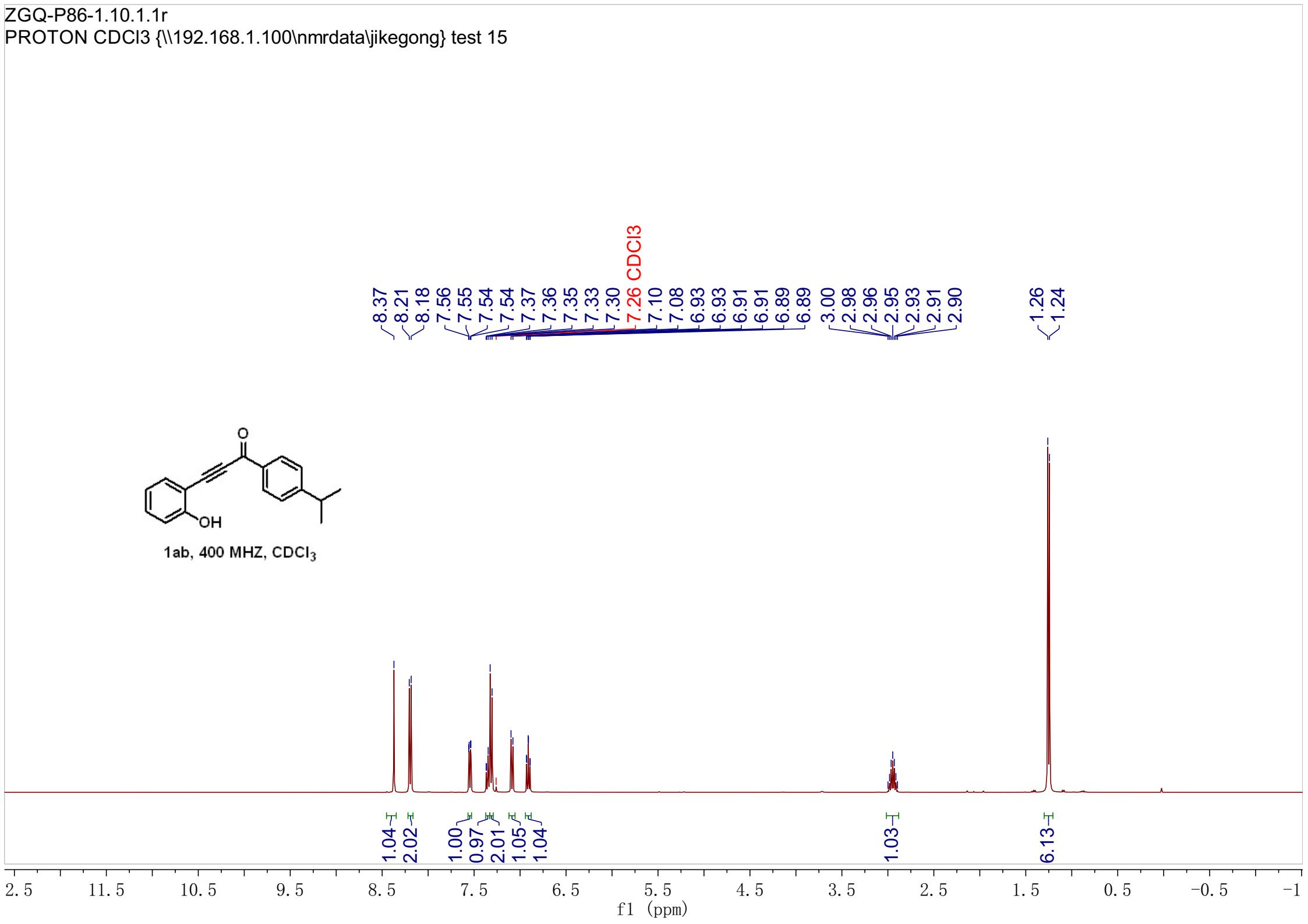

8.05
8.03
7.51
7.49
7.47
7.38
7.36
7.34
7.26 CDCl3
4.17
3.84
3.83
2.71
2.70
2.68
O
Ph
OH
1z, 400 MHZ, CDCl3
1.98
1.00
2.02
1.02
2.00
2.00
f1 (ppm)


178.47
136.20
134.12
129.50
128.41
94.50
80.27
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
59.88
23.33
O
Ph
OH
1z, 100 MHZ, CDCl3
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)

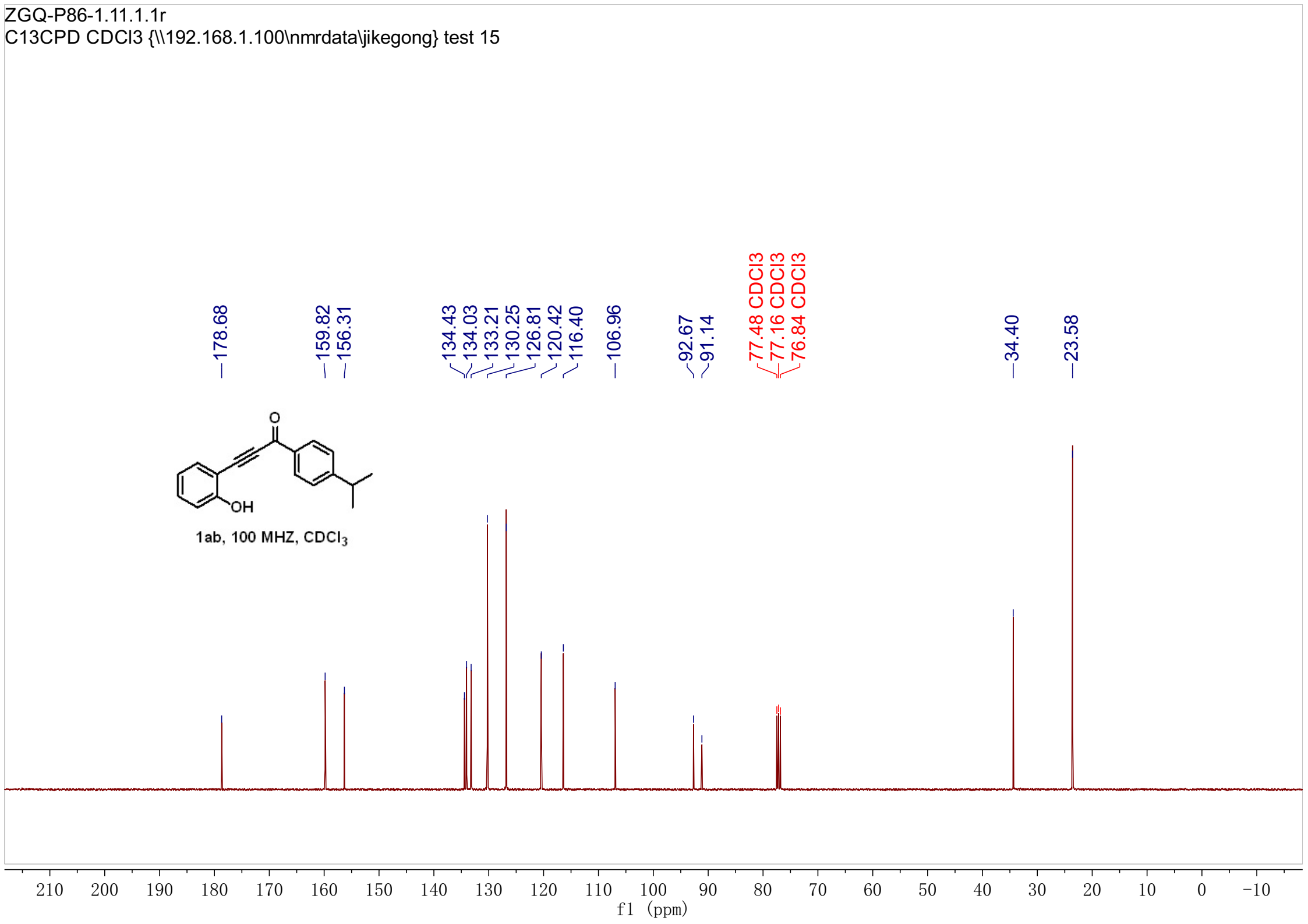

11.41
8.37
8.35
8.15
8.13
8.00
7.97
7.88
7.86
7.73
7.71
7.70
7.65
7.64
7.62
7.60
7.41
7.39
7.37
7.34
7.32
2.50 DMSO
2.50 DMSO
2.49 DMSO
Ph
O
OH
1aa, 400 MHZ, DMSO-d6
0.83
2.01
1.00
1.01
1.00
1.01
3.02
1.00
1.01
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

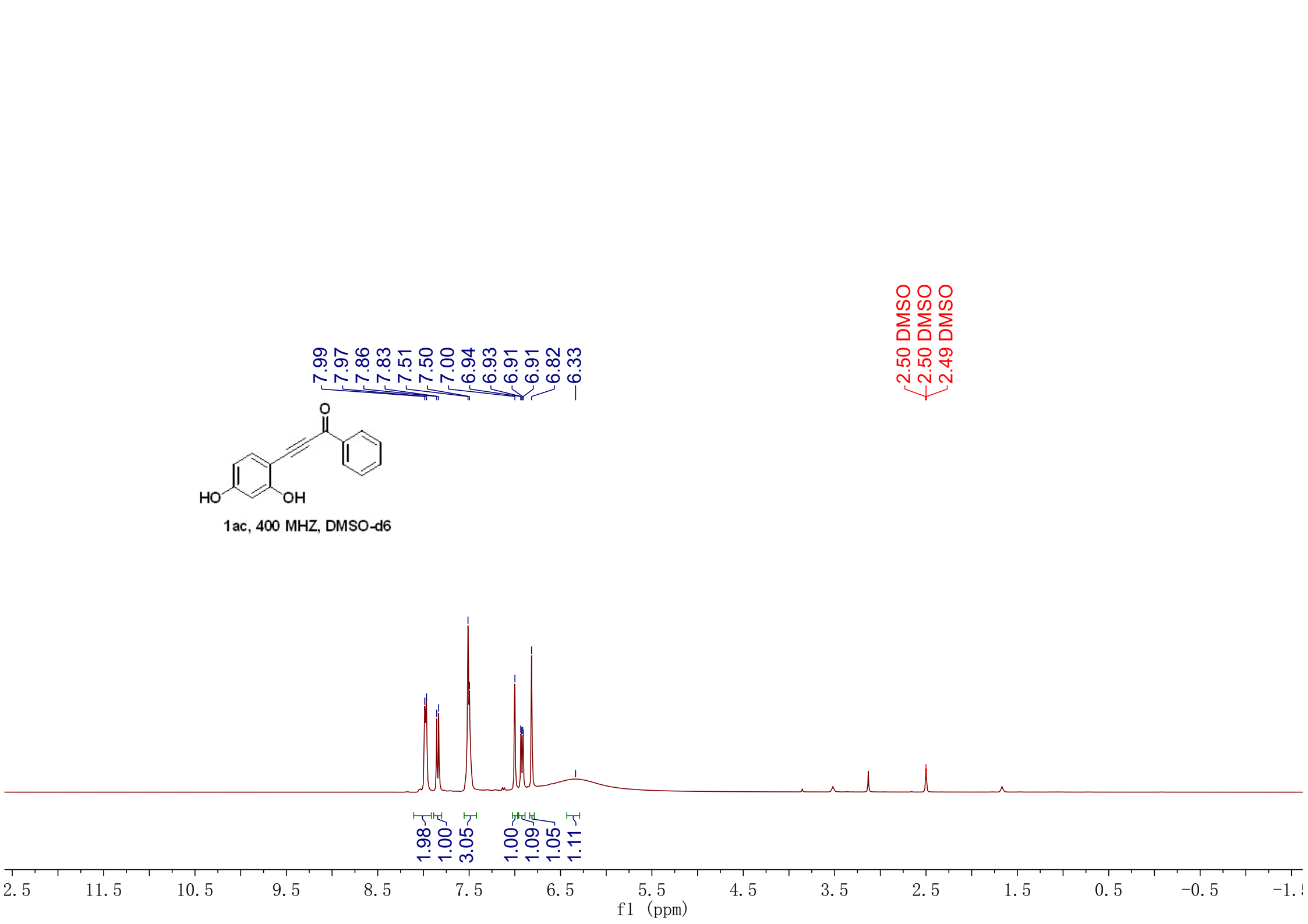

177.13
162.09
136.82
134.54
134.32
133.93
129.27
129.02
128.80
128.49
127.43
124.07
123.68
117.86
98.88
96.44
90.45
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
Ph
O
OH
1aa, 100 MHZ, DMSO-d6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

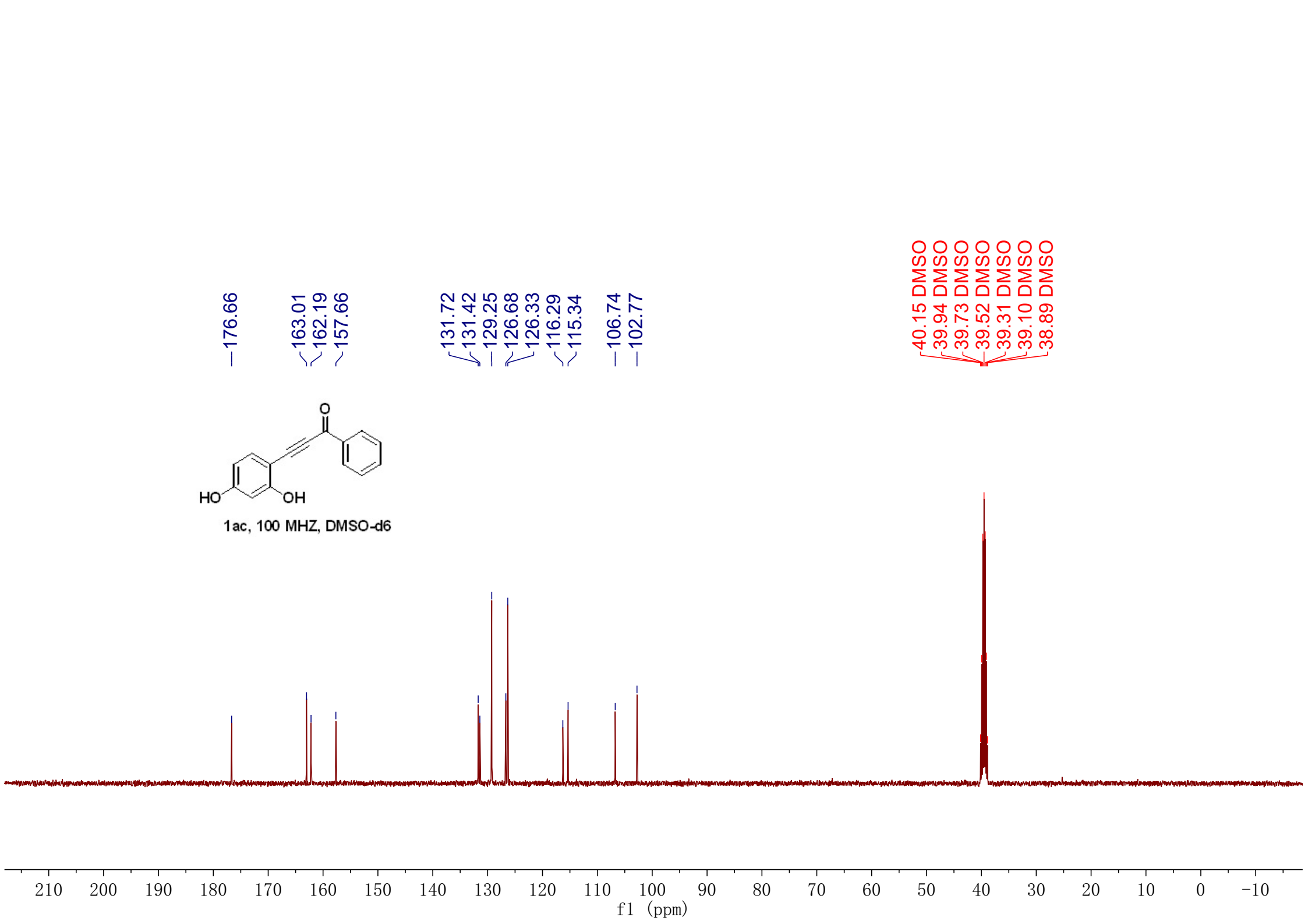
ZGQ-P86-1.10.1.1r
PROTON CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 15

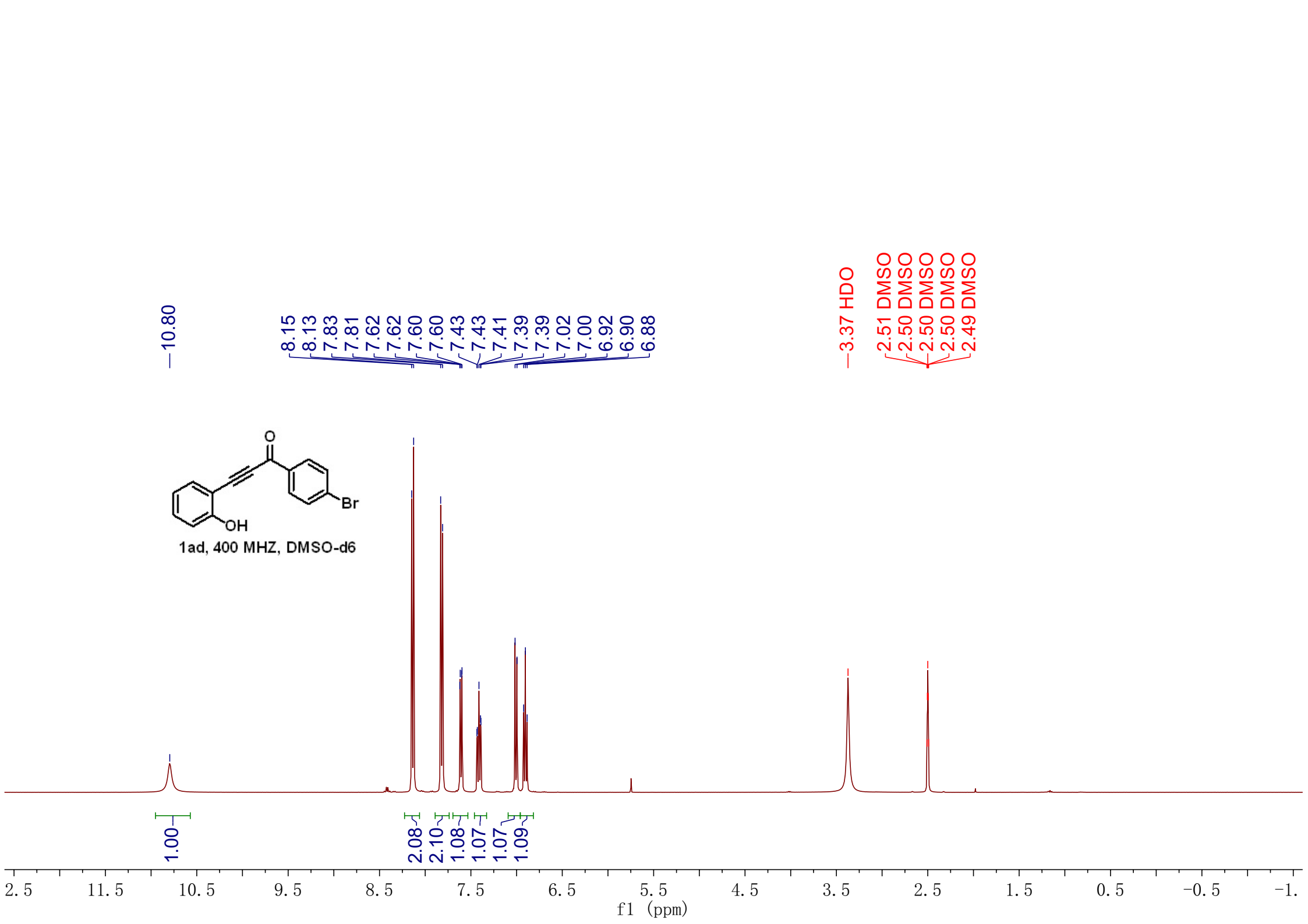


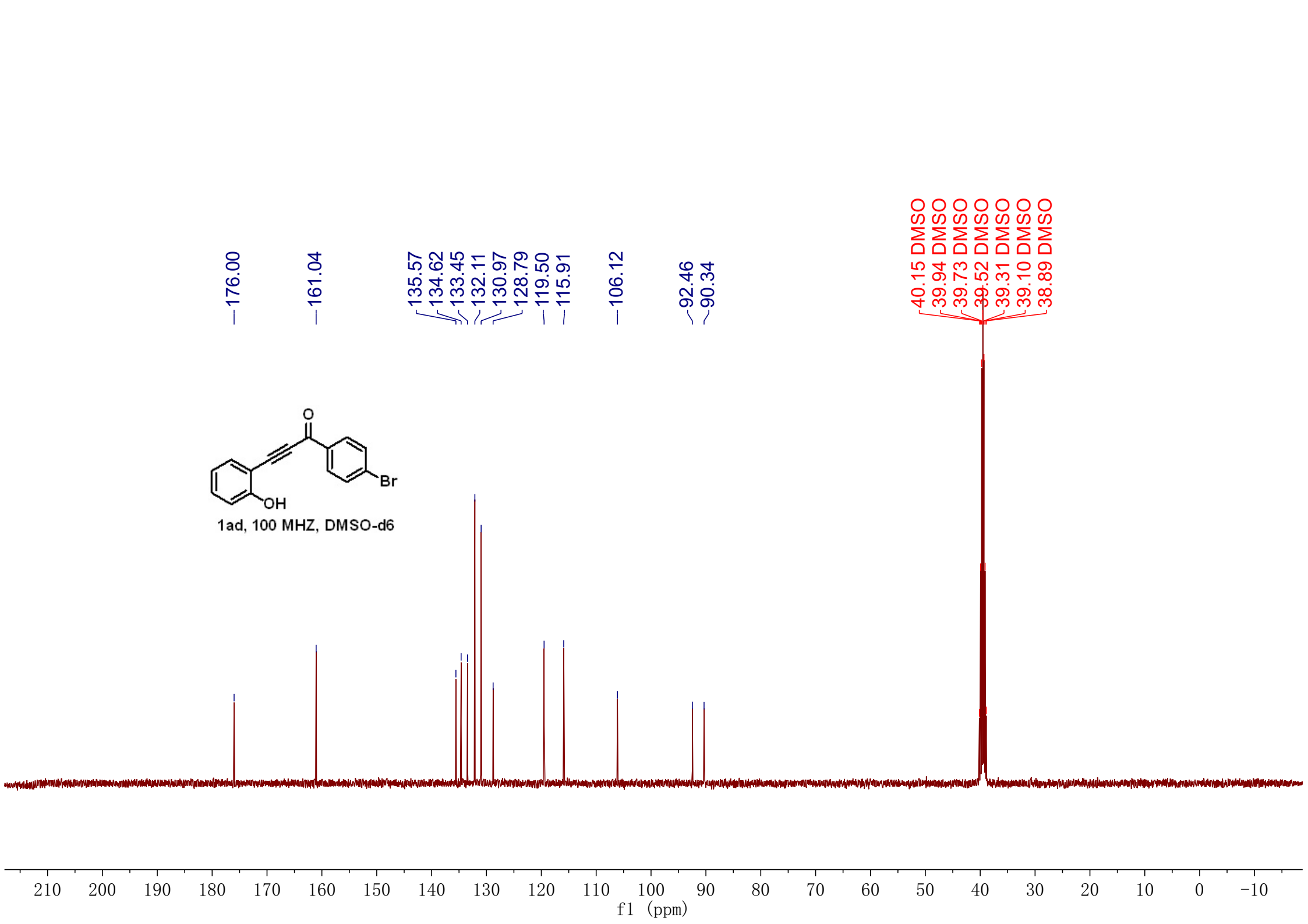
ZGQ-P86-1.11.1.1r
C13CPD CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 15

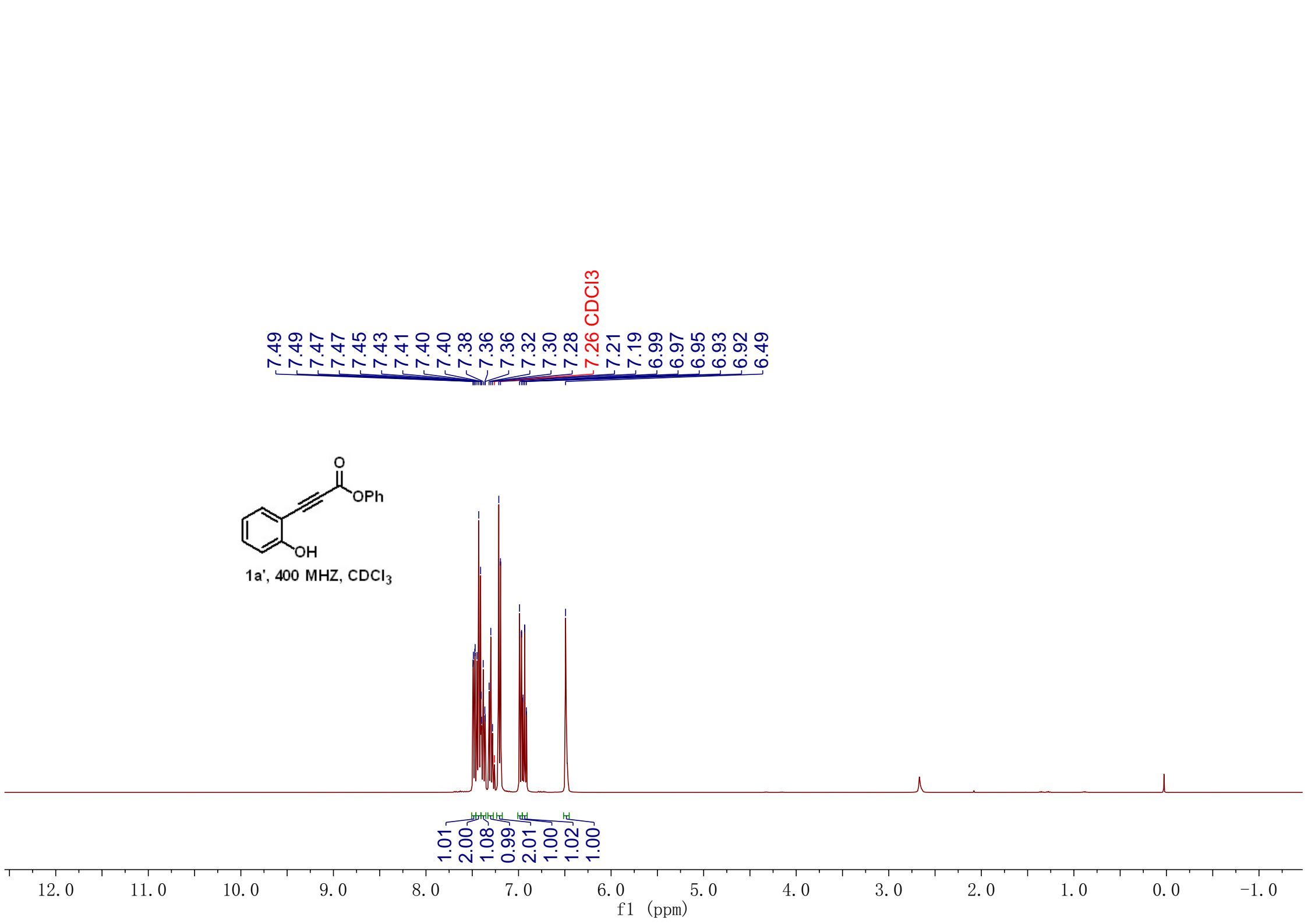


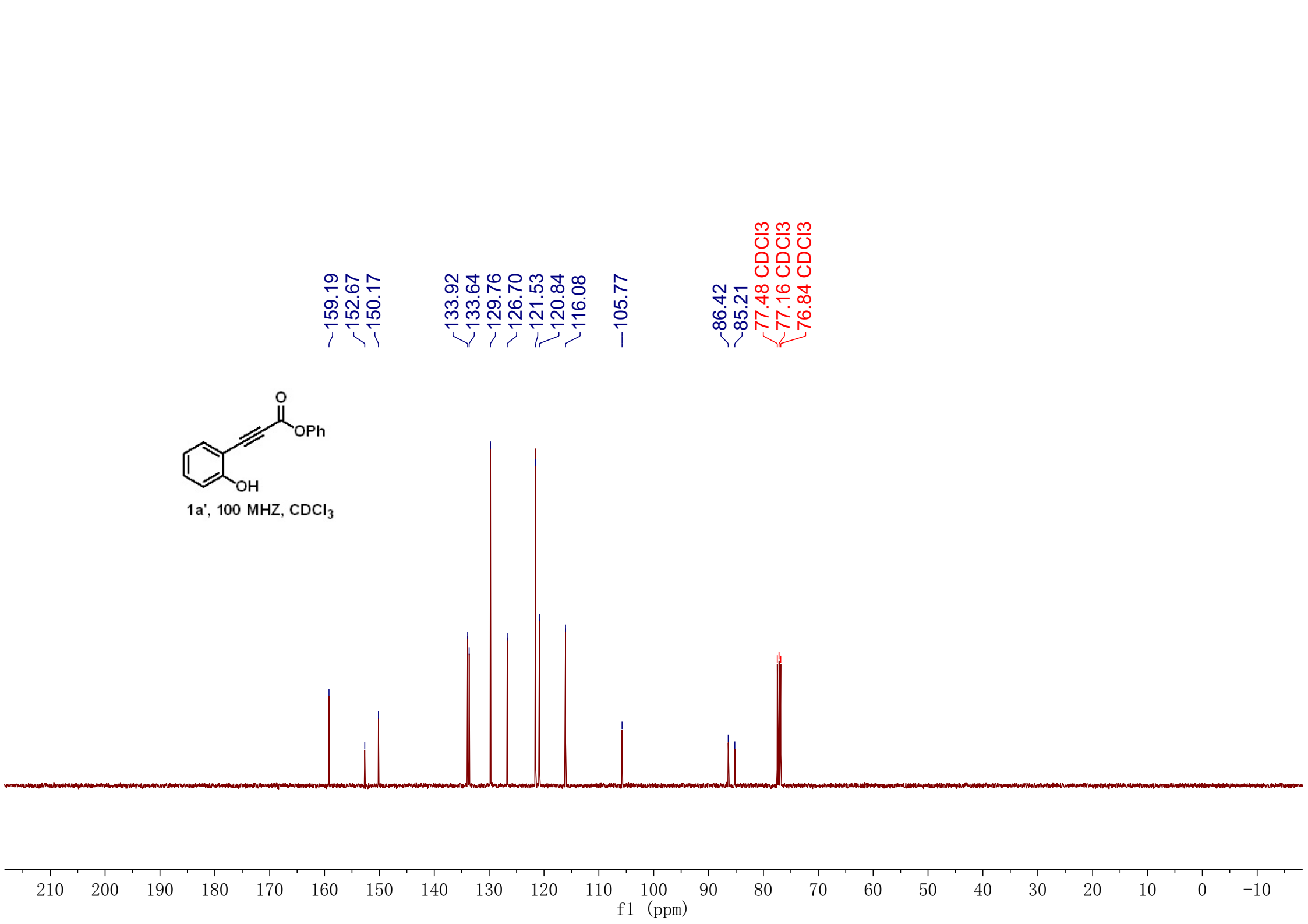

7.99
7.97
7.86
7.83
7.51
7.50
7.00
6.94
6.93
6.91
6.91
6.82
6.33
2.50 DMSO
2.50 DMSO
2.49 DMSO
O
HO
OH
1ac, 400 MHZ, DMSO-d6
1.98
1.00
3.05
1.00
1.09
1.05
1.11
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


176.66
163.01
162.19
157.66
131.72
131.42
129.25
126.68
126.33
116.29
115.34
106.74
102.77
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
HO
OH
O
1ac, 100 MHZ, DMSO-d6
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)

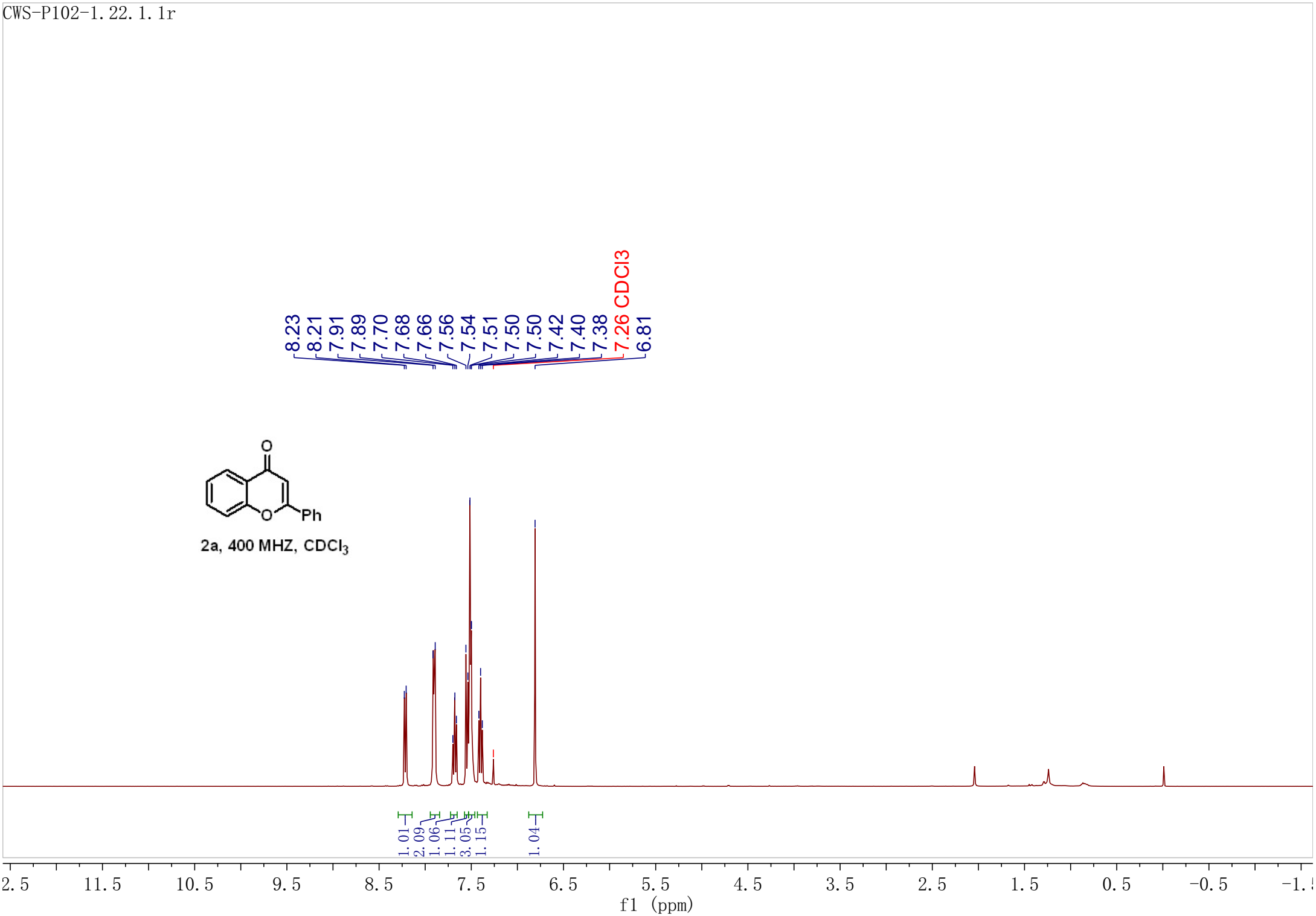

10.80
8.15
8.13
7.83
7.81
7.62
7.62
7.60
7.60
7.43
7.43
7.41
7.39
7.39
7.02
7.00
6.92
6.90
6.88
3.37 HDO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
O
Br
OH
1ad, 400 MHZ, DMSO-d6
1.00
2.08
2.10
1.08
1.07
1.07
1.09
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)


176.00
161.04
135.57
134.62
133.45
132.11
130.97
128.79
119.50
115.91
106.12
92.46
90.34
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
O
Br
OH
1ad, 100 MHZ, DMSO-d6
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)

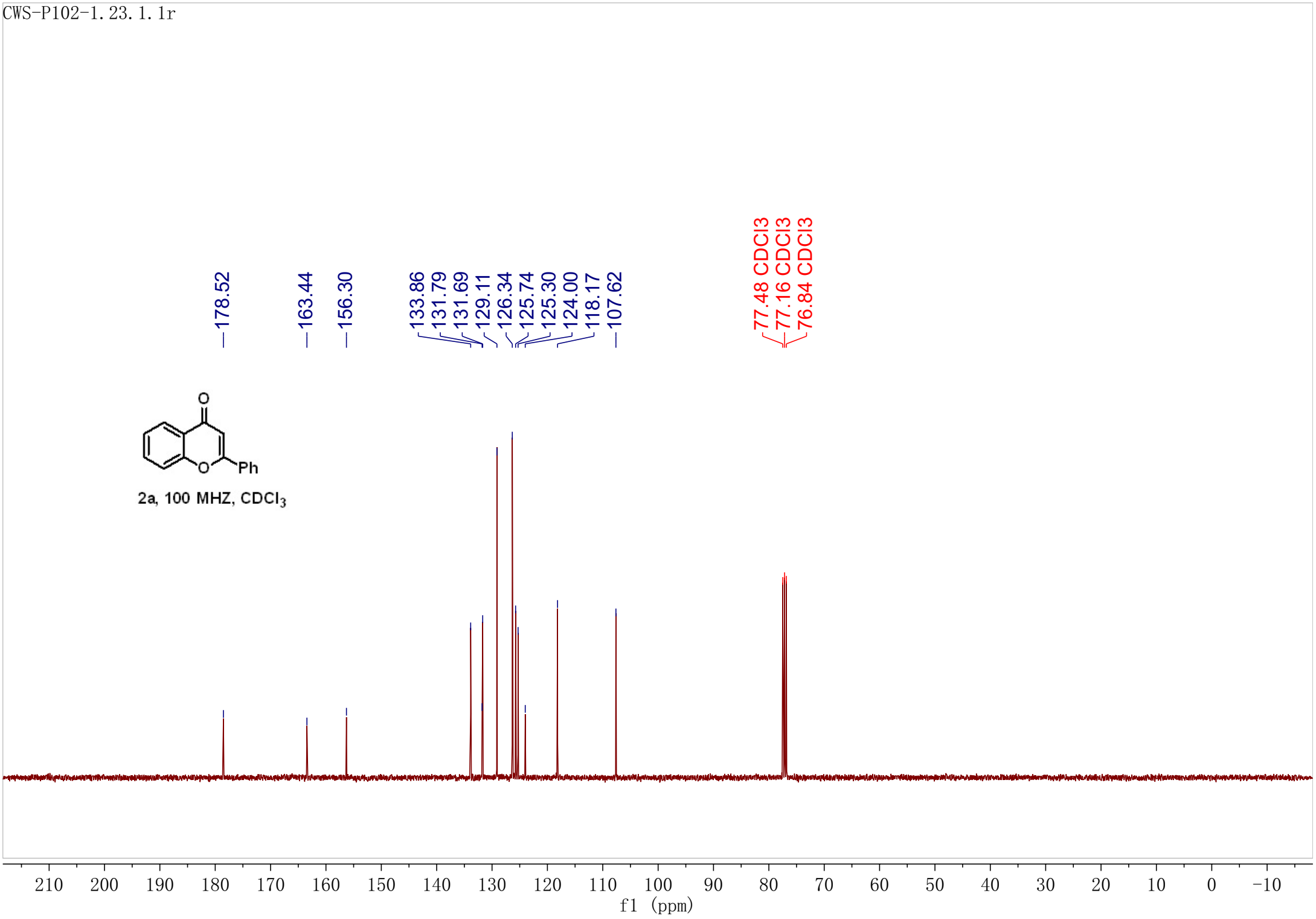

7.49
7.49
7.47
7.47
7.45
7.43
7.41
7.40
7.40
7.38
7.36
7.36
7.32
7.30
7.28
7.26 CDCl3
7.21
7.19
6.99
6.97
6.95
6.93
6.92
6.49
O
OPh
OH
1a', 400 MHZ, CDCl3
1.01
2.00
1.08
0.99
2.01
1.00
1.02
1.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

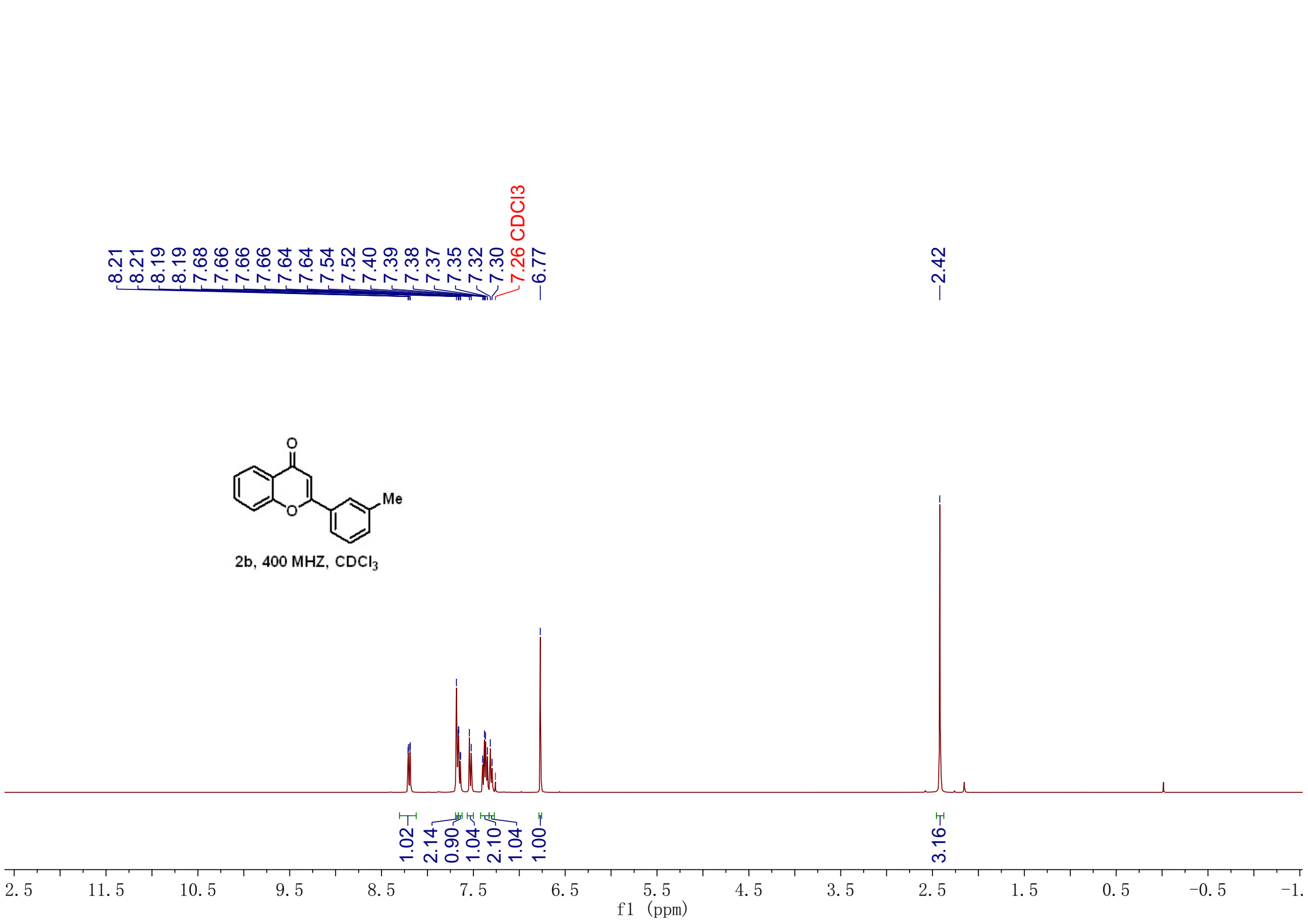

159.19
152.67
150.17
133.92
133.64
129.76
126.70
121.53
120.84
116.08
105.77
86.42
85.21
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
OPh
OH
1a', 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

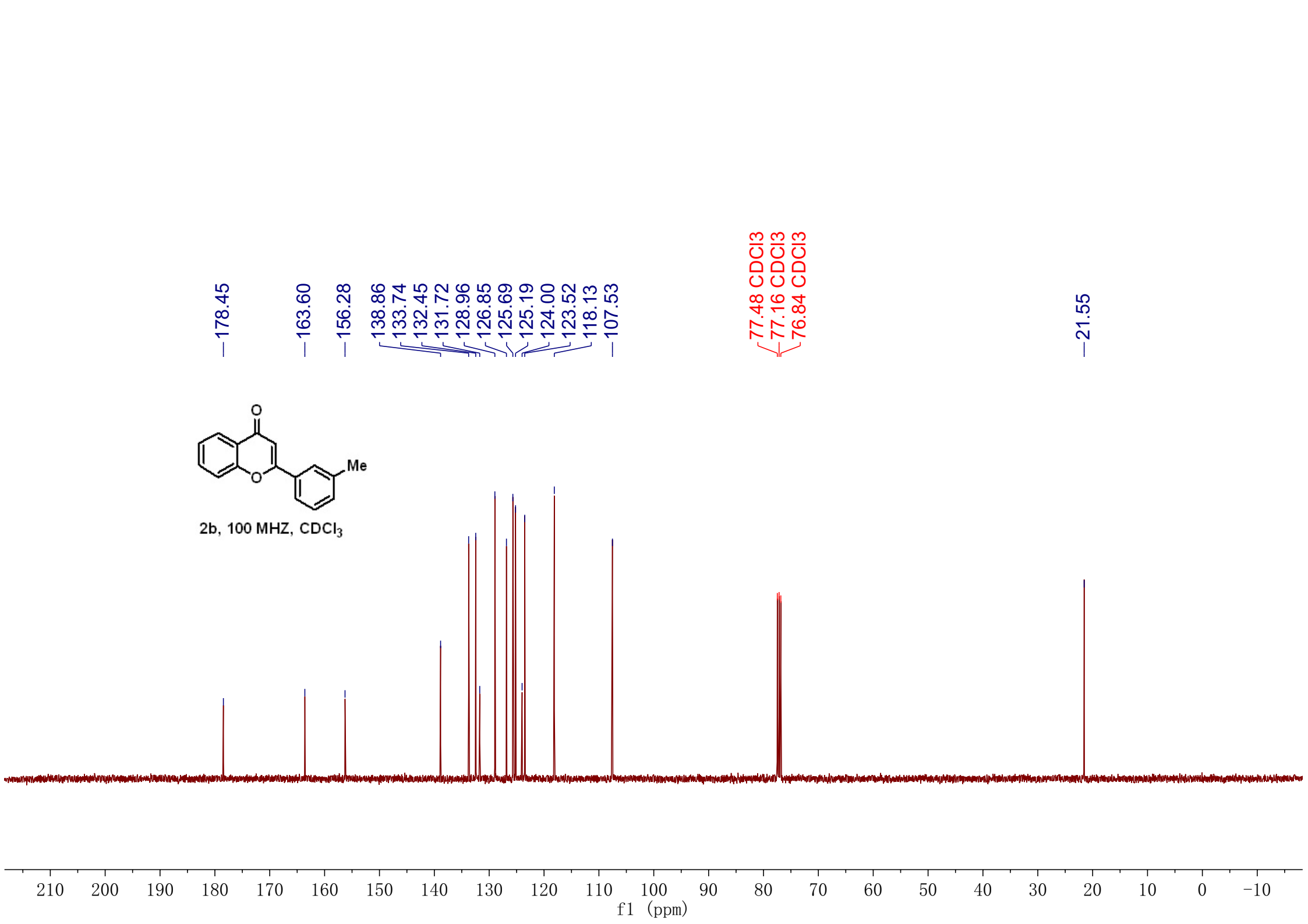
CWS-P102-1.22.1.1r



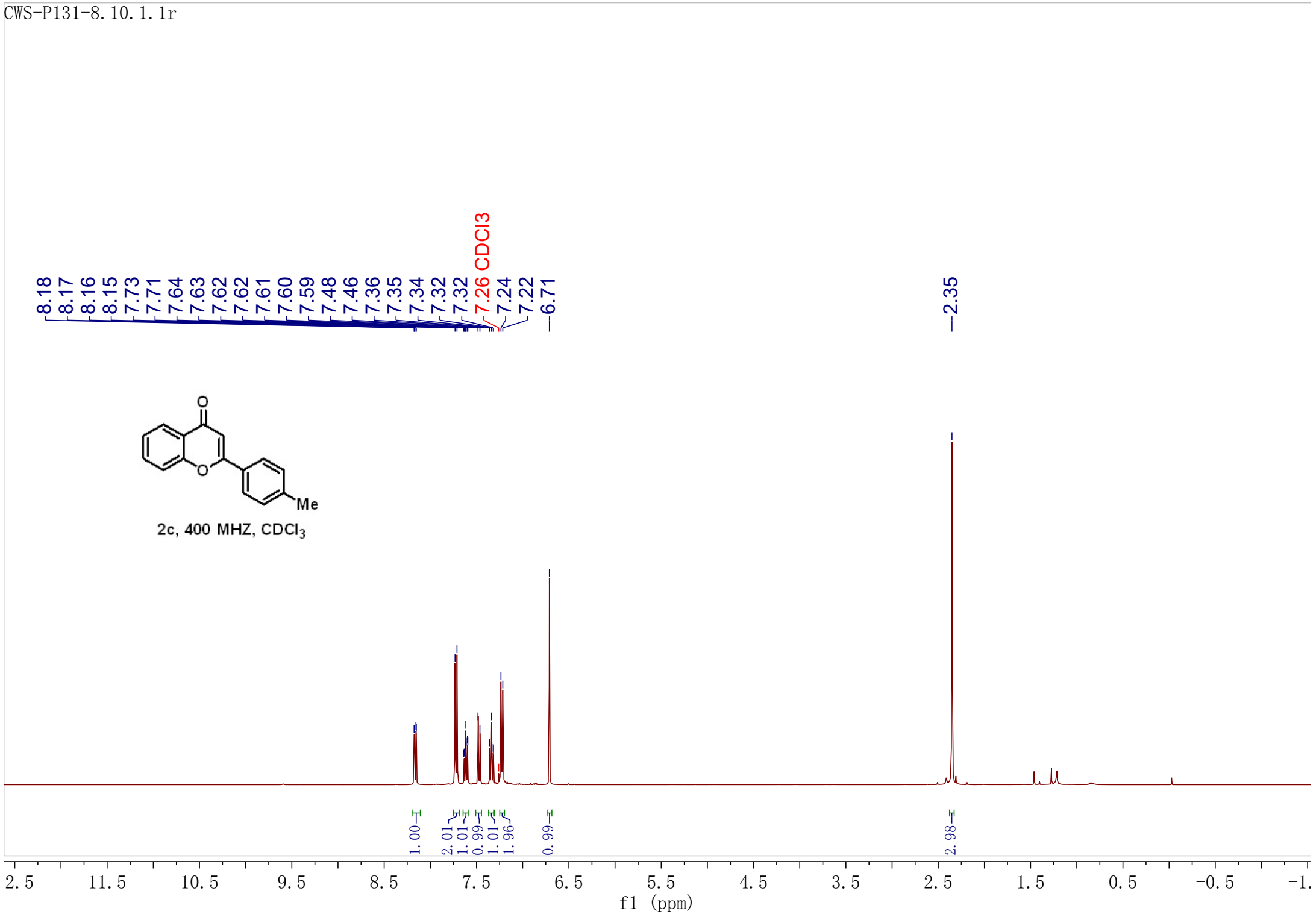
CWS-P102-1.23.1.1r



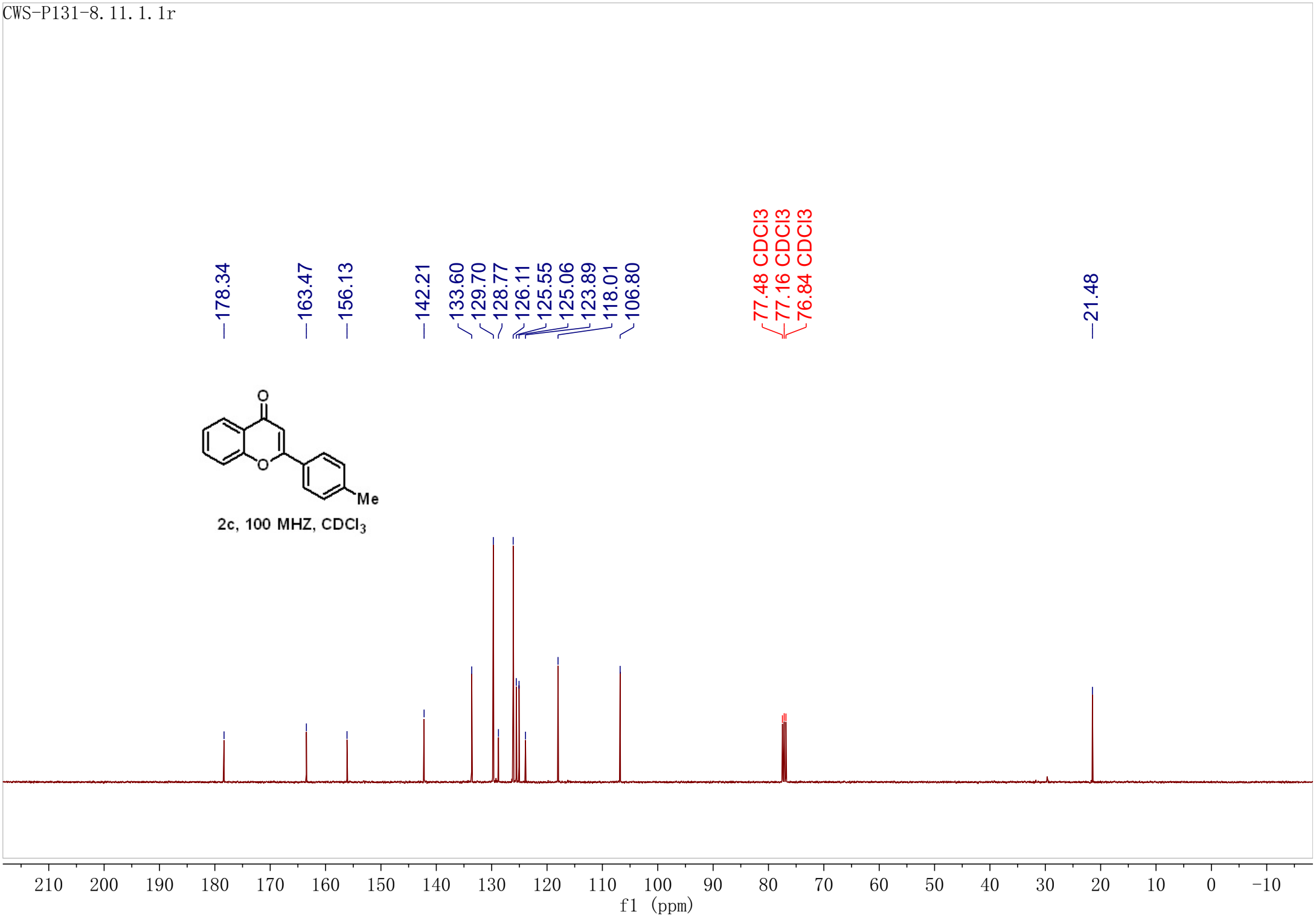

8.21
8.21
8.19
8.19
7.68
7.66
7.66
7.66
7.64
7.64
7.54
7.52
7.40
7.39
7.38
7.37
7.35
7.32
7.30
7.26 CDCl3
6.77
2.42
O
O
Me
2b, 400 MHZ, CDCl3
1.02
2.14
0.90
1.04
2.10
1.04
1.00
3.16
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)

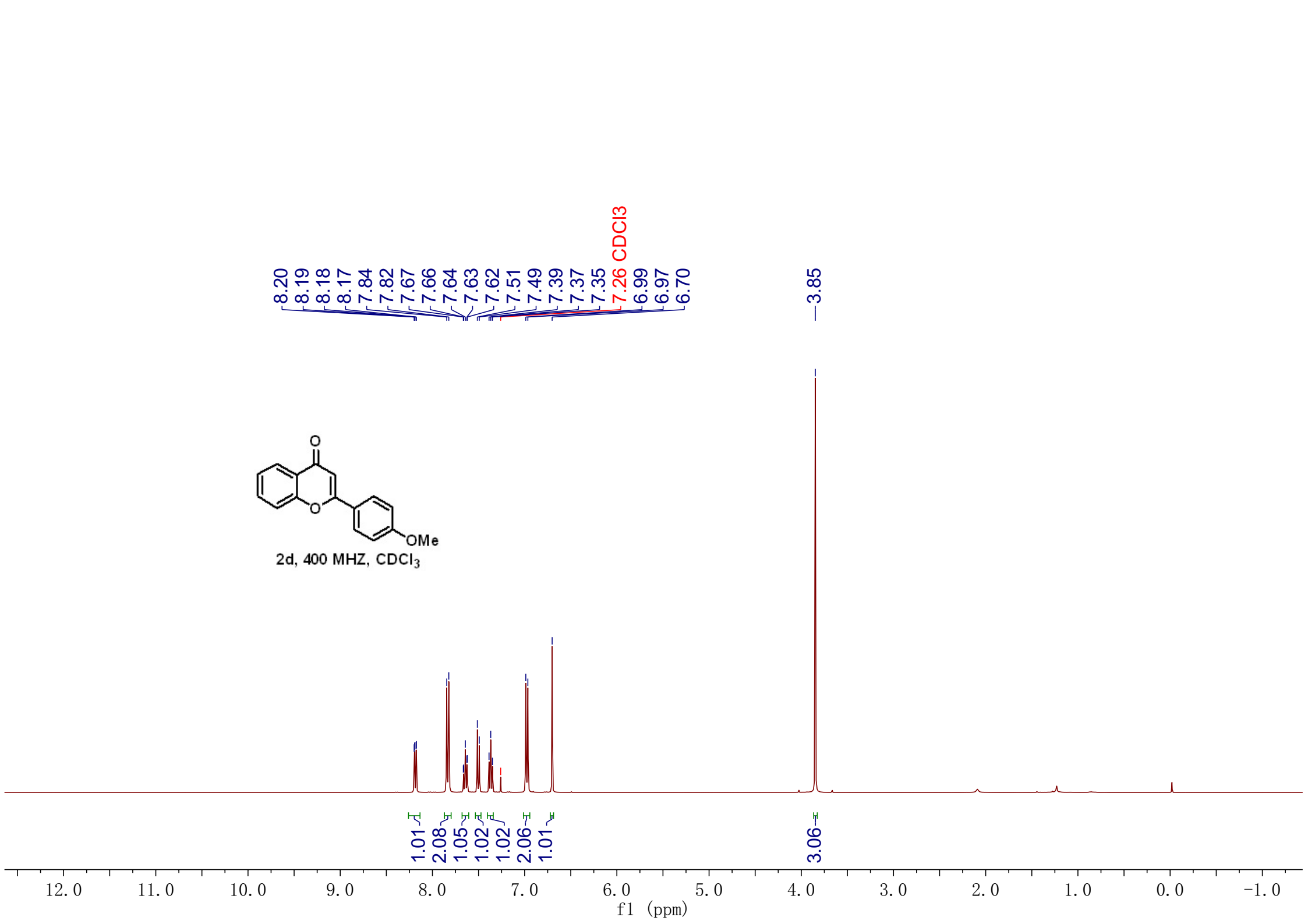

178.45
163.60
156.28
138.86
133.74
132.45
131.72
128.96
126.85
125.69
125.19
124.00
123.52
118.13
107.53
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
21.55
O
O
Me
2b, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

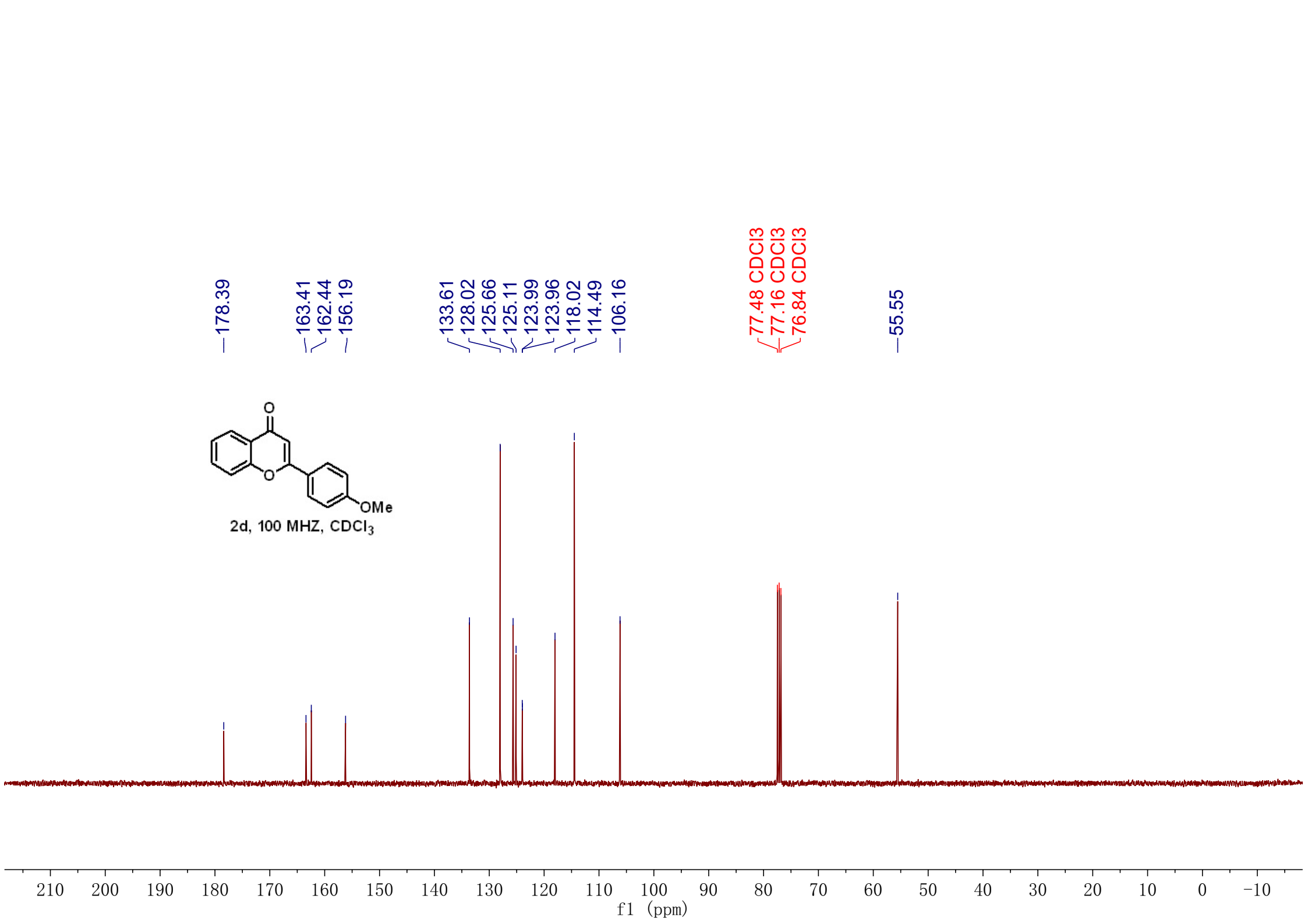
CWS-P131-8.10.1.1r



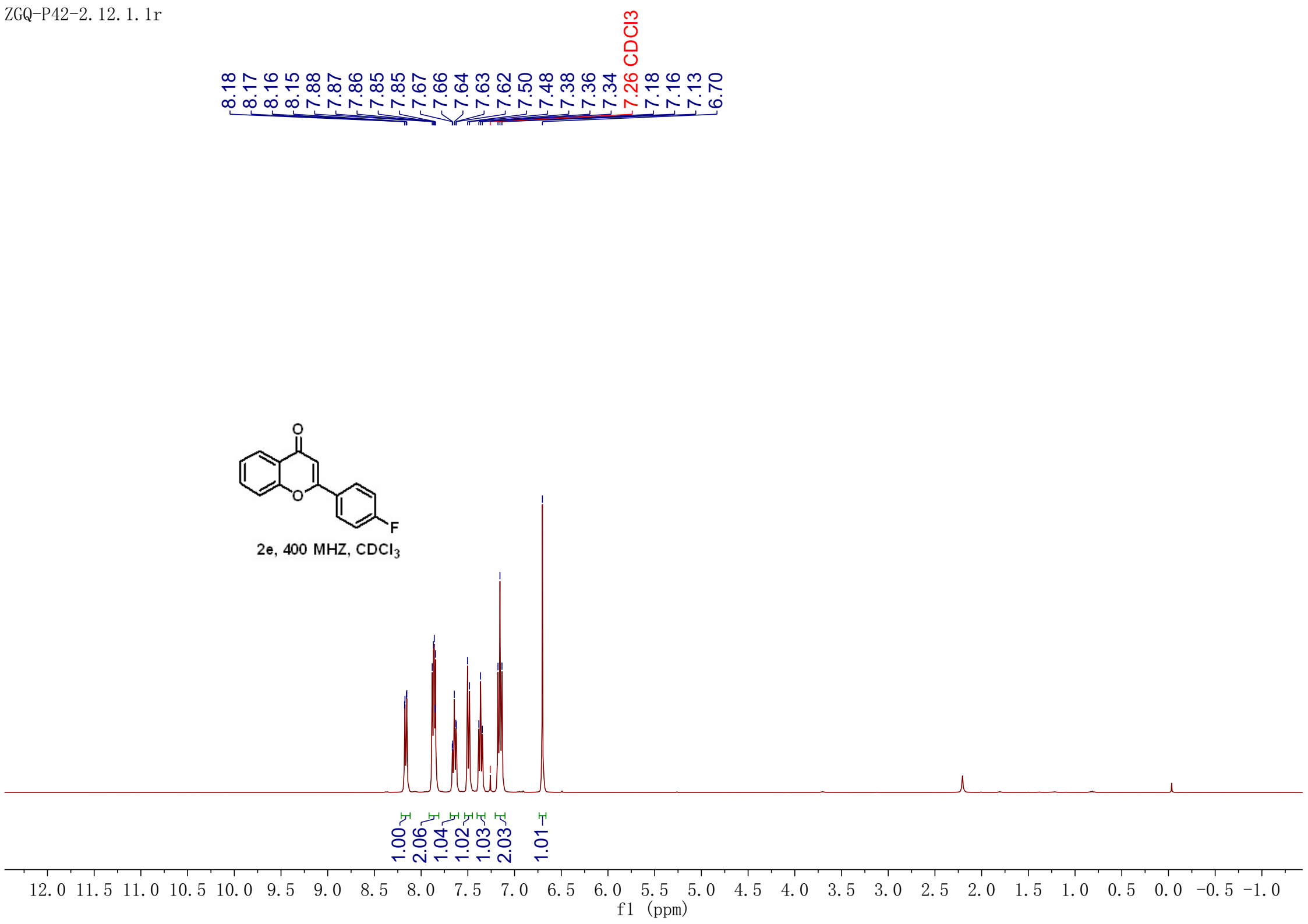
CWS-P131-8.11.1.1r



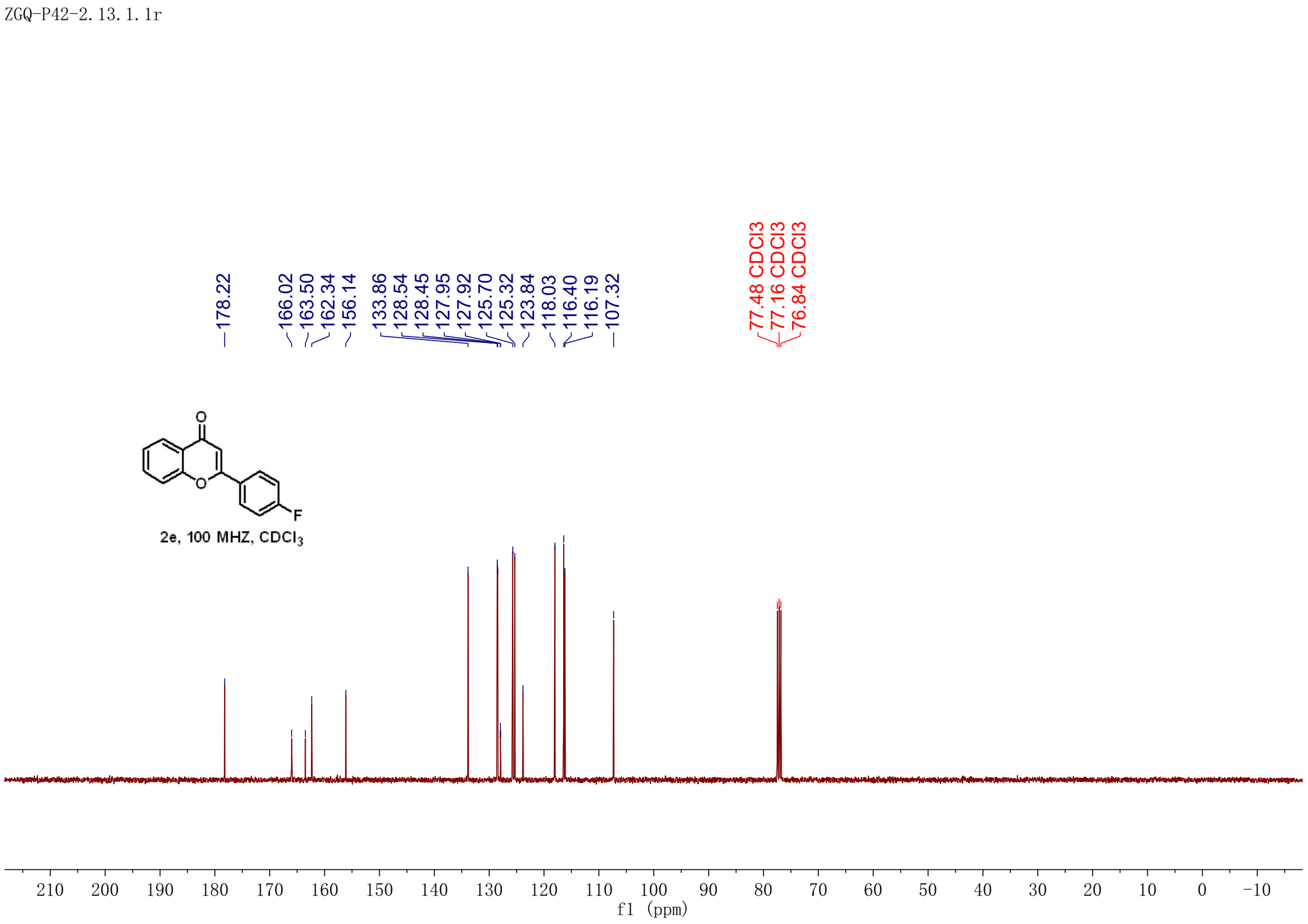

8.20
8.19
8.18
8.17
7.84
7.82
7.67
7.66
7.64
7.63
7.62
7.51
7.49
7.39
7.37
7.35
7.26 CDCl3
6.99
6.97
6.70
3.85
O
O
OMe
2d, 400 MHZ, CDCl3
1.01
2.08
1.05
1.02
1.02
2.06
1.01
3.06
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


178.39
163.41
162.44
156.19
133.61
128.02
125.66
125.11
123.99
123.96
118.02
114.49
106.16
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
55.55
O
O
OMe
2d, 100 MHZ, CDCl3
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)

ZGQ-P42-2.12.1.1r

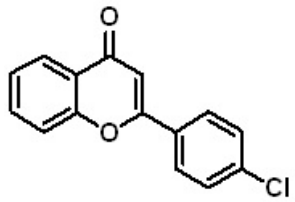


ZGQ-P42-2.13.1.1r



pdata/1
PROTON CDCl3 {D:\2022-1} ZHL 33

8.19
8.19
8.18
8.17
7.82
7.81
7.69
7.68
7.67
7.67
7.67
7.66
7.65
7.53
7.51
7.46
7.45
7.41
7.39
7.38
7.26 CDCl3
6.75

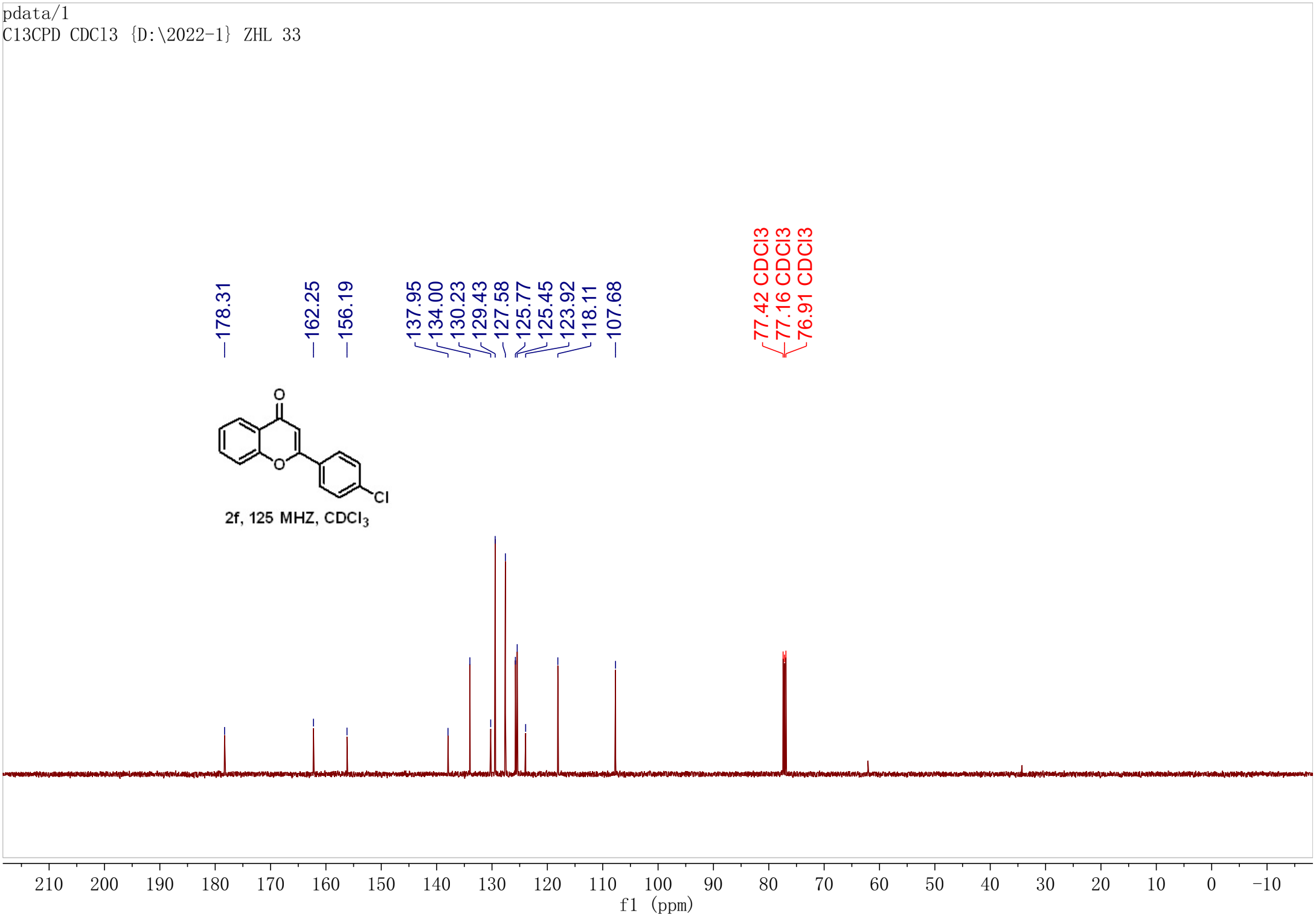


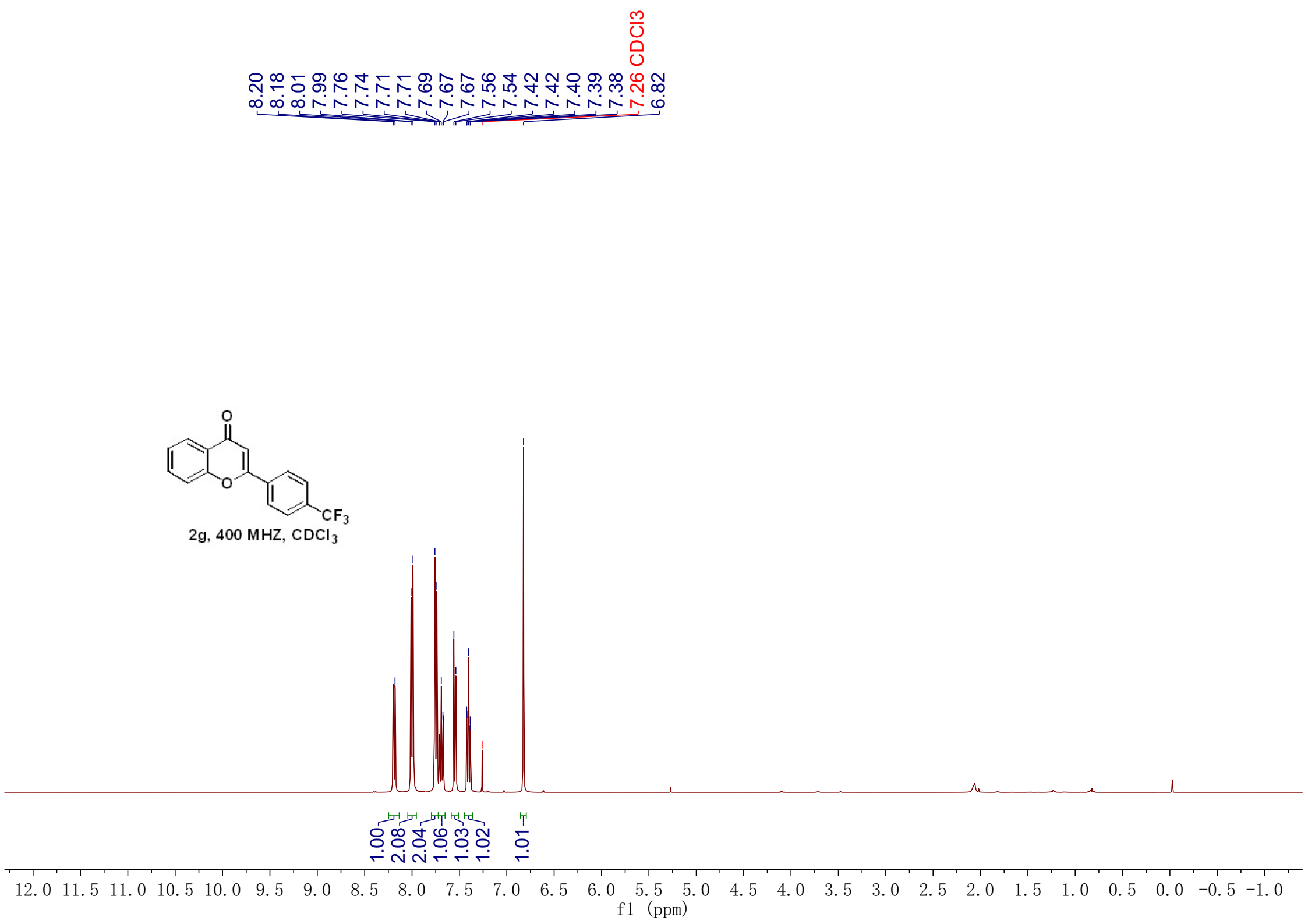
2f, 500 MHZ, $CDCl_3$

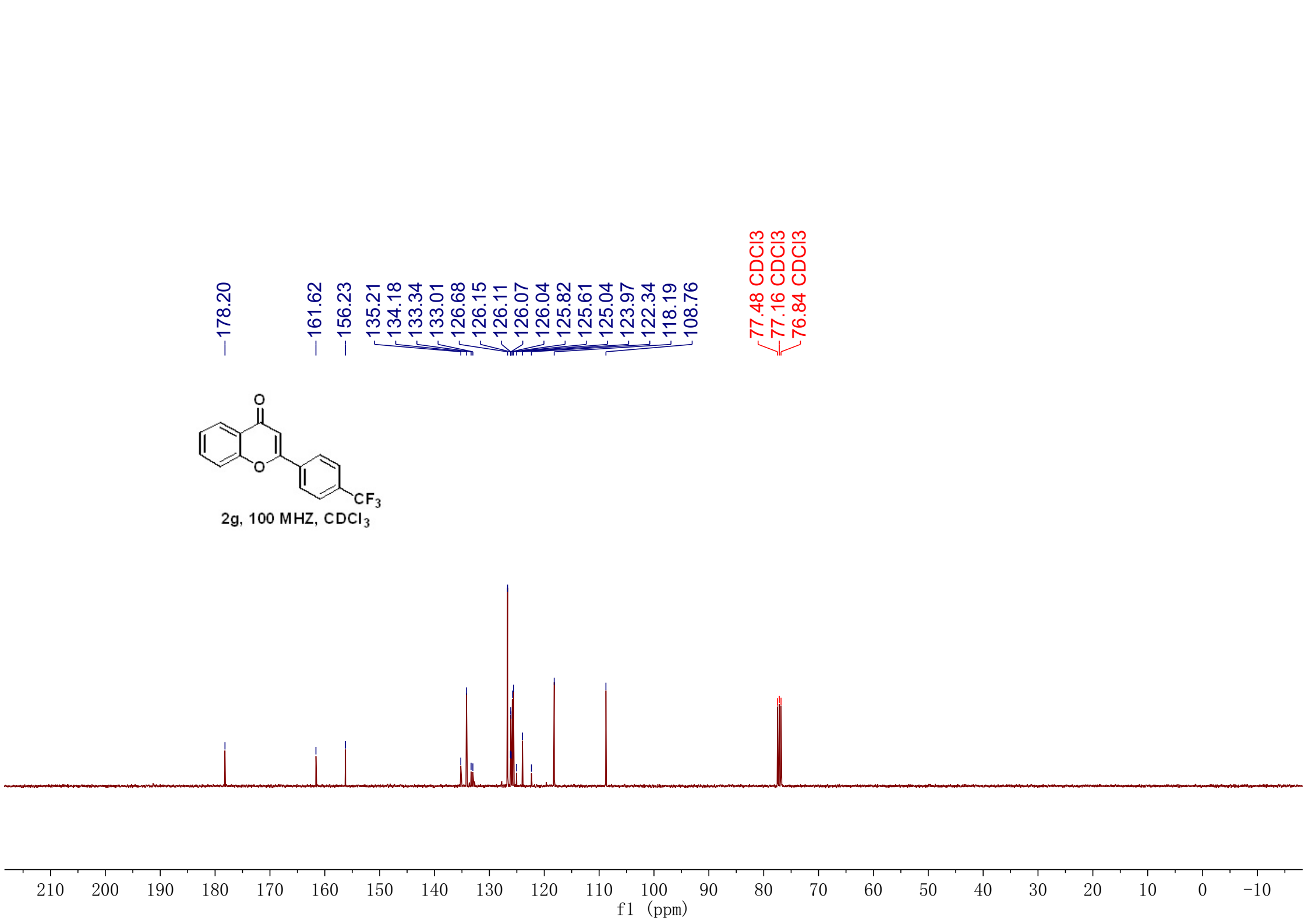
1.00
2.06
1.06
1.02
2.00
1.04
1.03

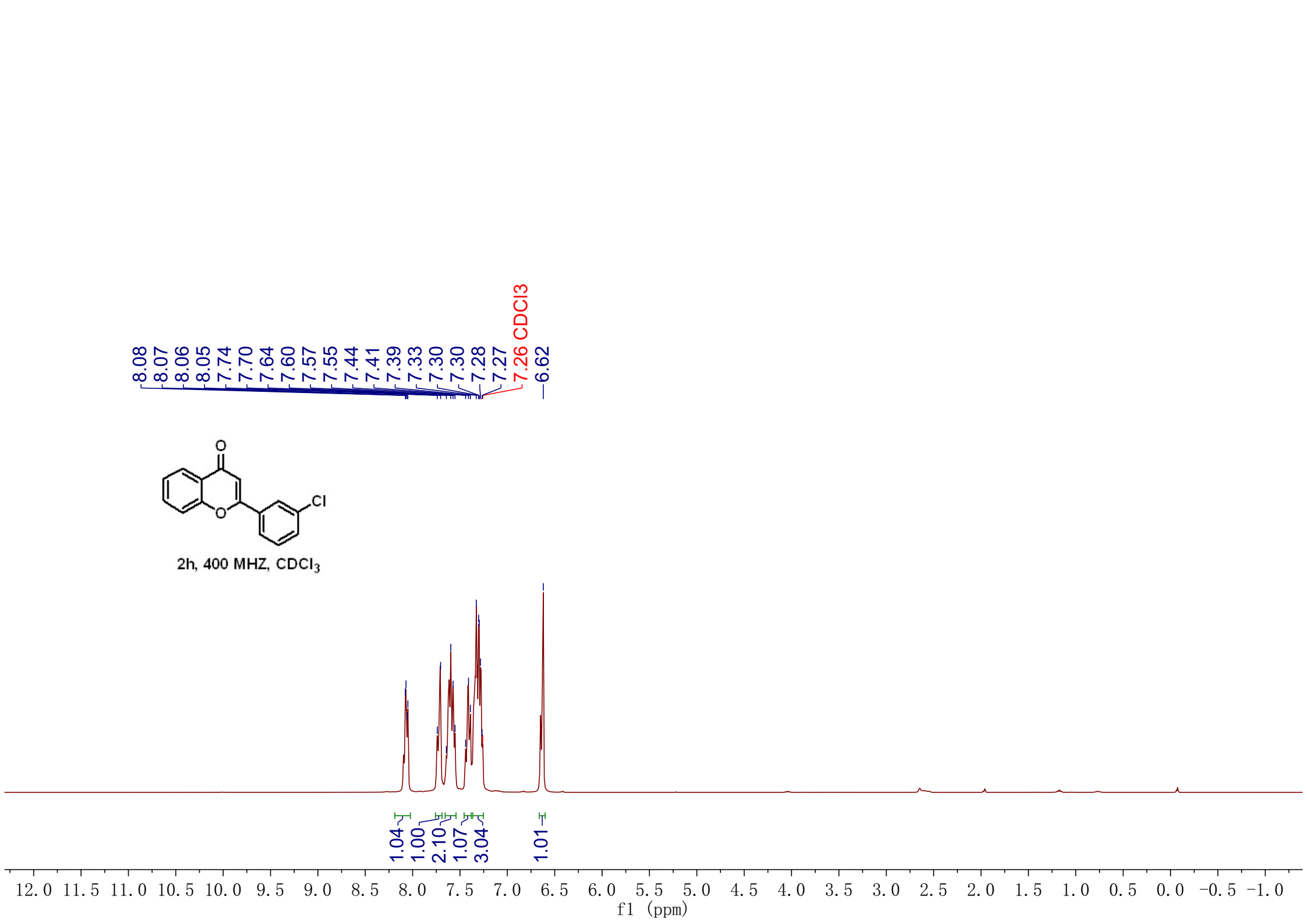
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

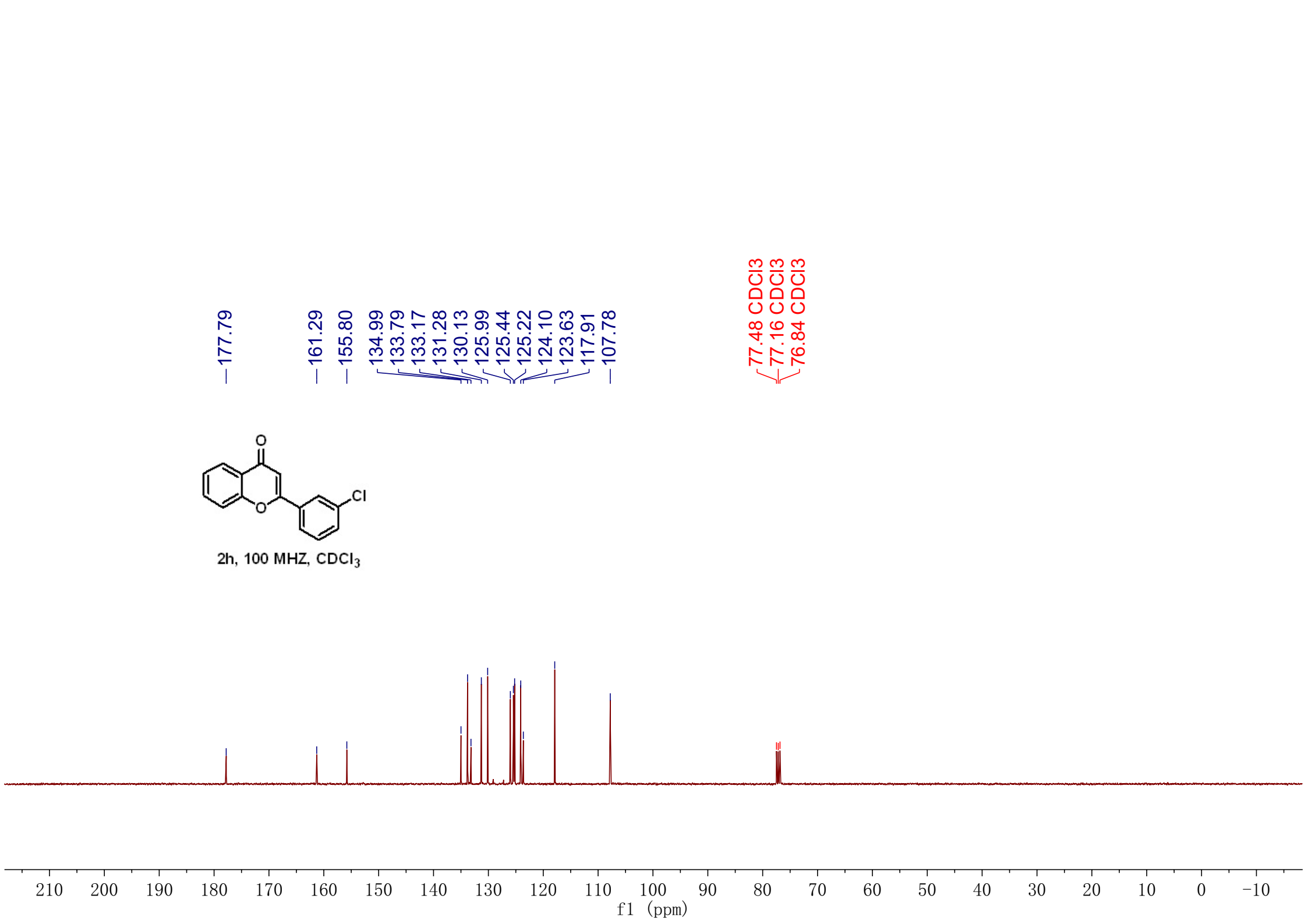
pdata/1
C13CPD CDCl3 {D:\2022-1} ZHL 33

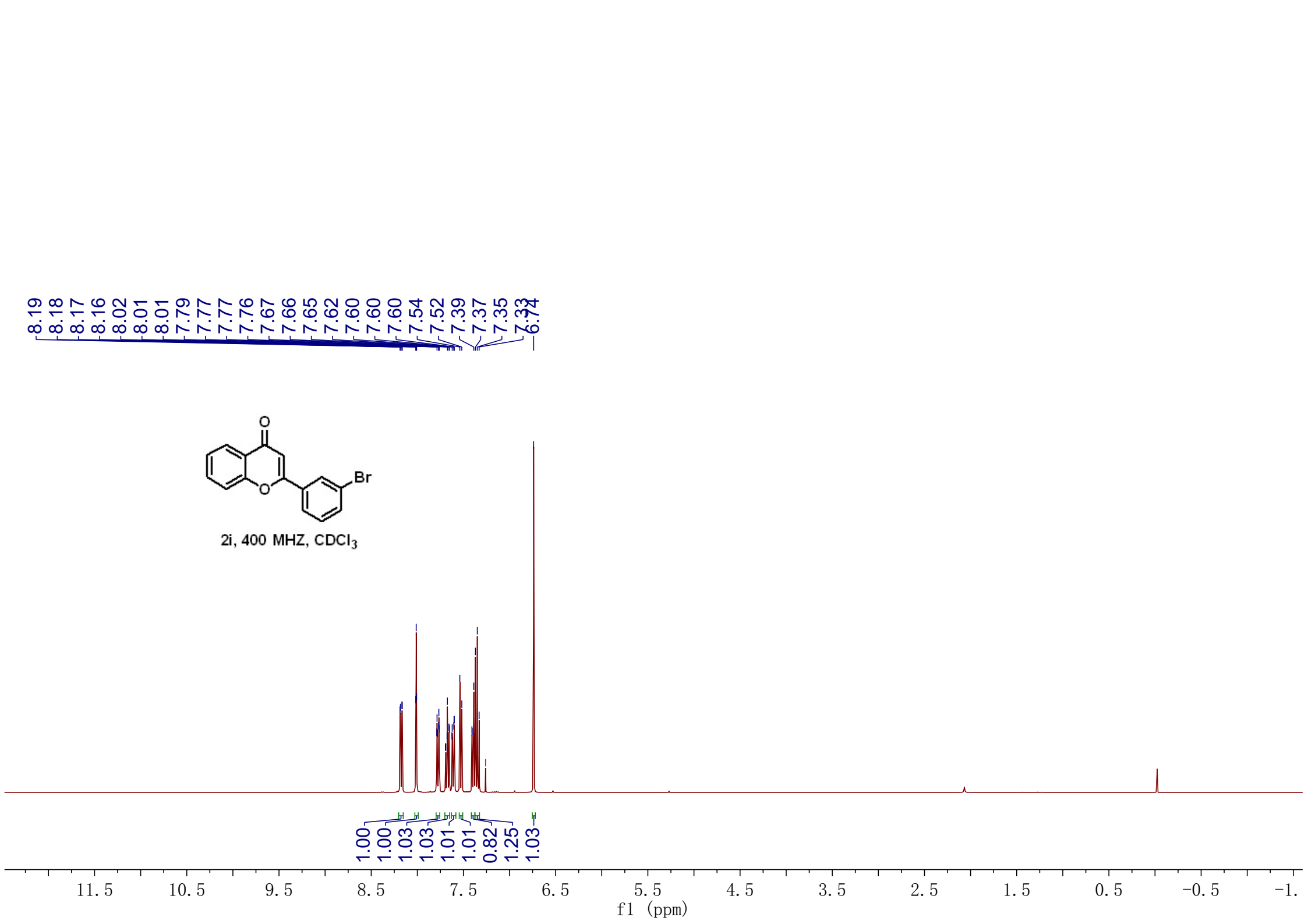


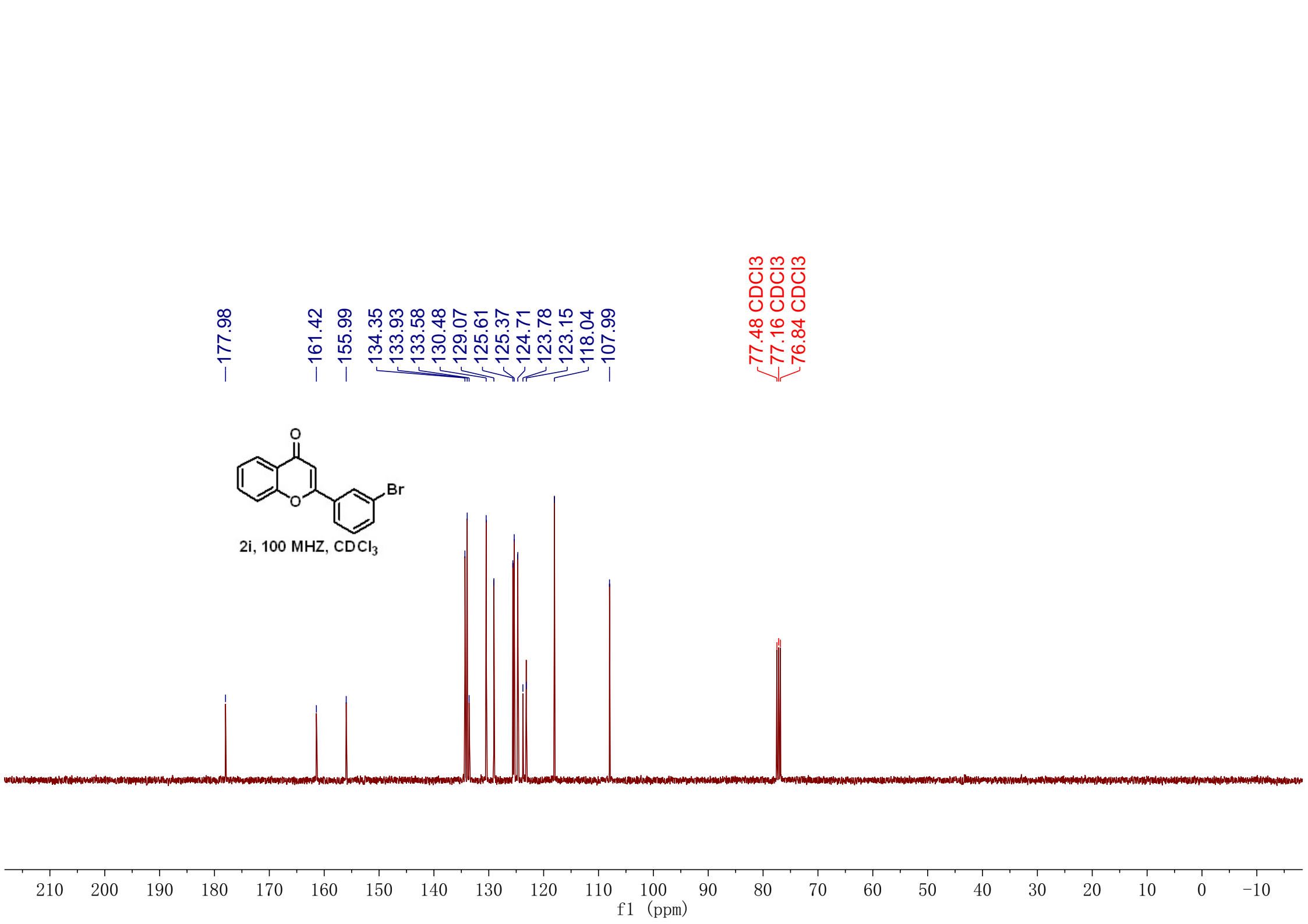

8.20
8.18
8.01
7.99
7.76
7.74
7.71
7.71
7.69
7.67
7.67
7.56
7.54
7.42
7.42
7.40
7.39
7.38
7.26 CDCl3
6.82
O
O
CF3
2g, 400 MHZ, CDCl3
1.00
2.08
2.04
1.06
1.03
1.02
1.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

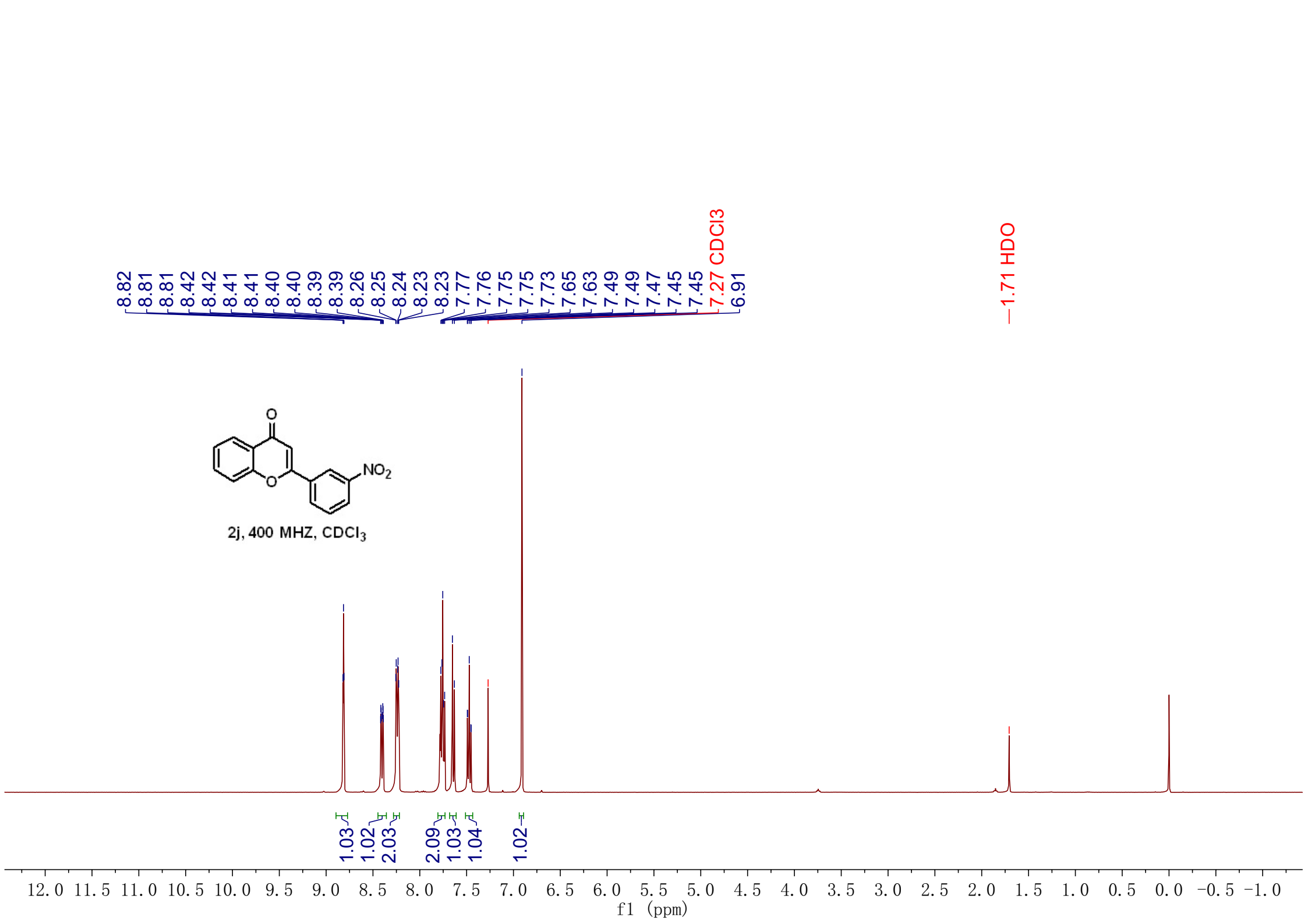

178.20
161.62
156.23
135.21
134.18
133.34
133.01
126.68
126.15
126.11
126.07
126.04
125.82
125.61
125.04
123.97
122.34
118.19
108.76
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
CF3
2g, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

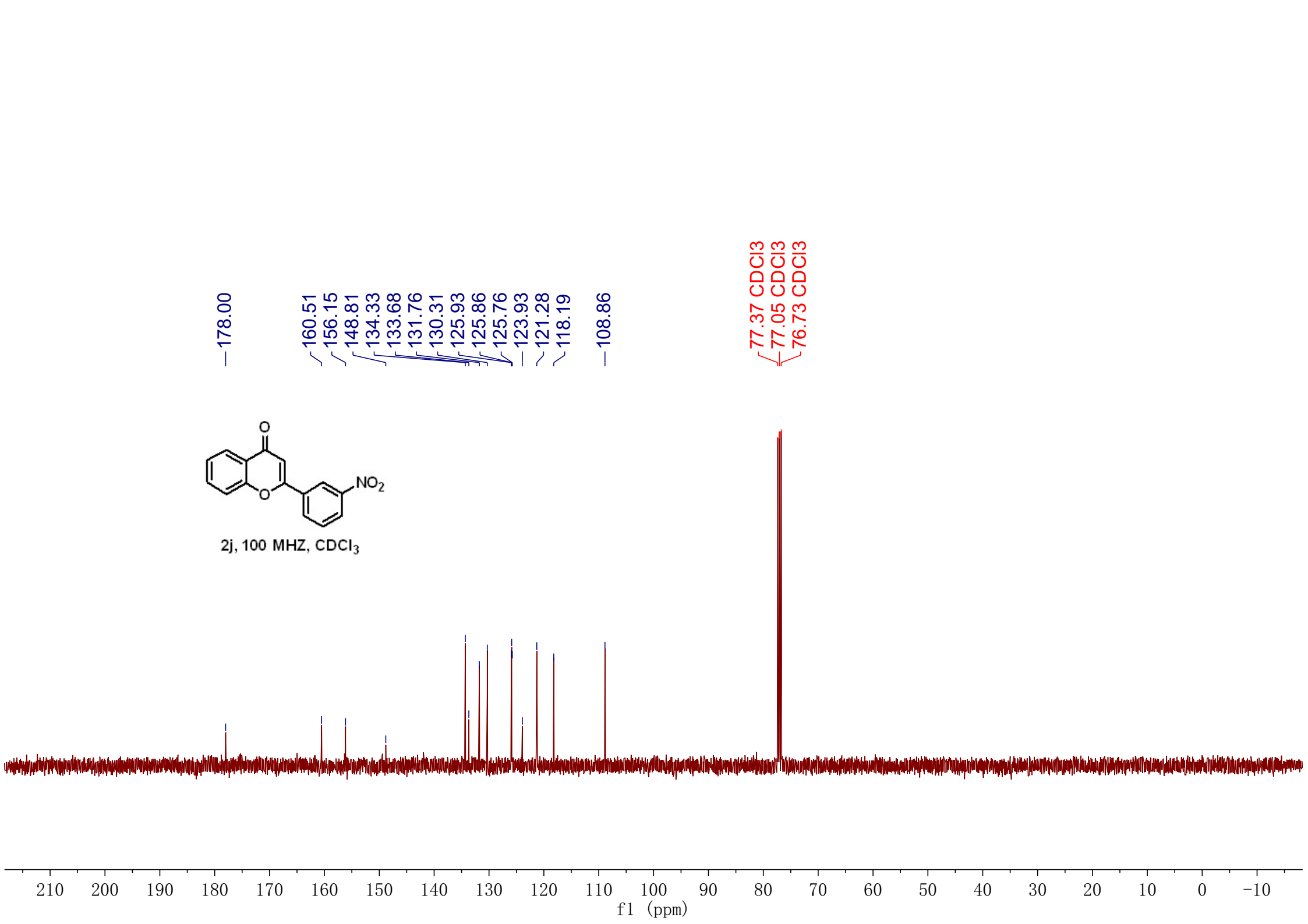

8.08
8.07
8.06
8.05
7.74
7.70
7.64
7.60
7.57
7.55
7.44
7.41
7.39
7.33
7.30
7.30
7.28
7.27
7.26 CDCl3
6.62
O
O
Cl
2h, 400 MHZ, CDCl3
1.04
1.00
2.10
1.07
3.04
1.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

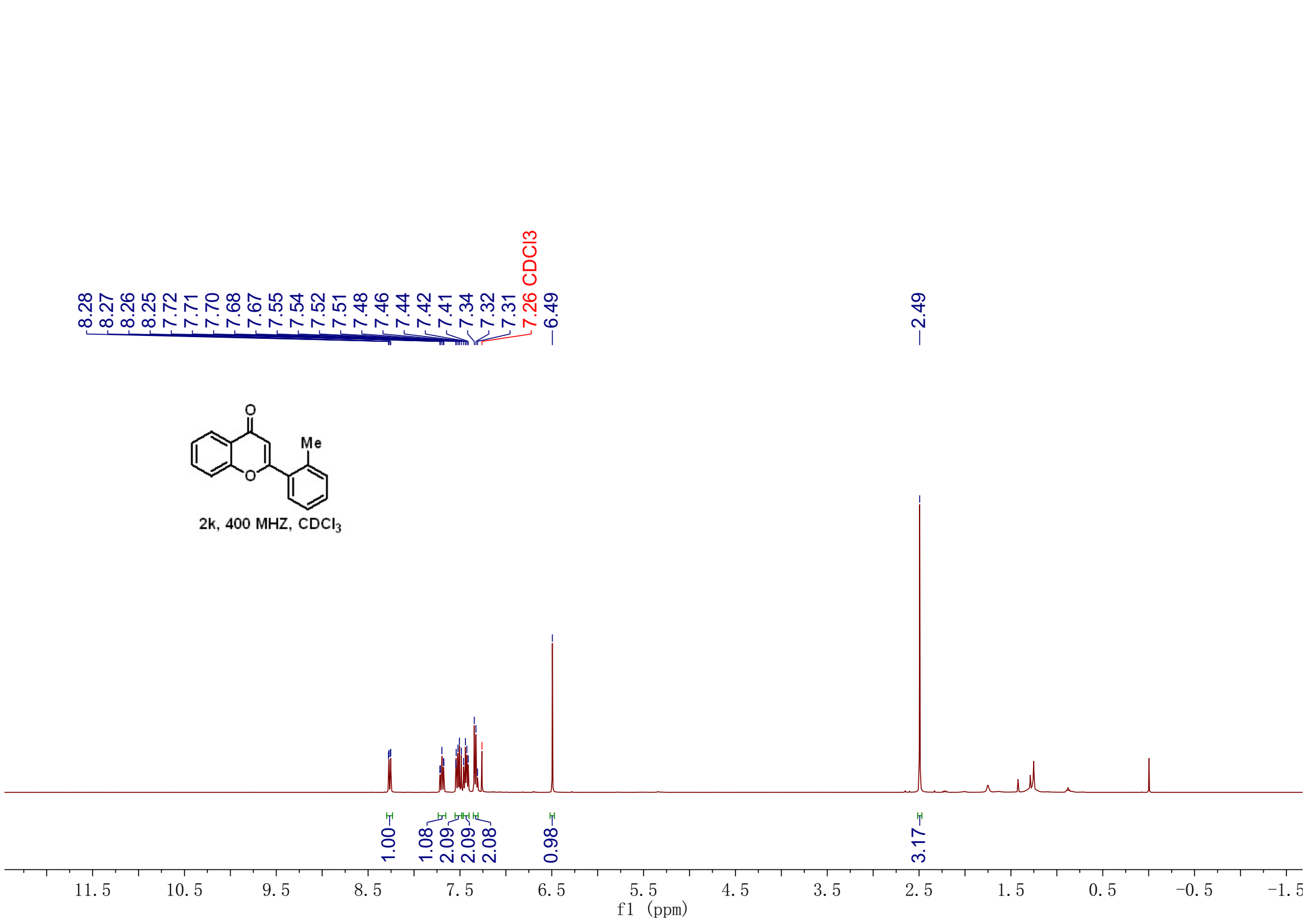

177.79
161.29
155.80
134.99
133.79
133.17
131.28
130.13
125.99
125.44
125.22
124.10
123.63
117.91
107.78
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
Cl
2h, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

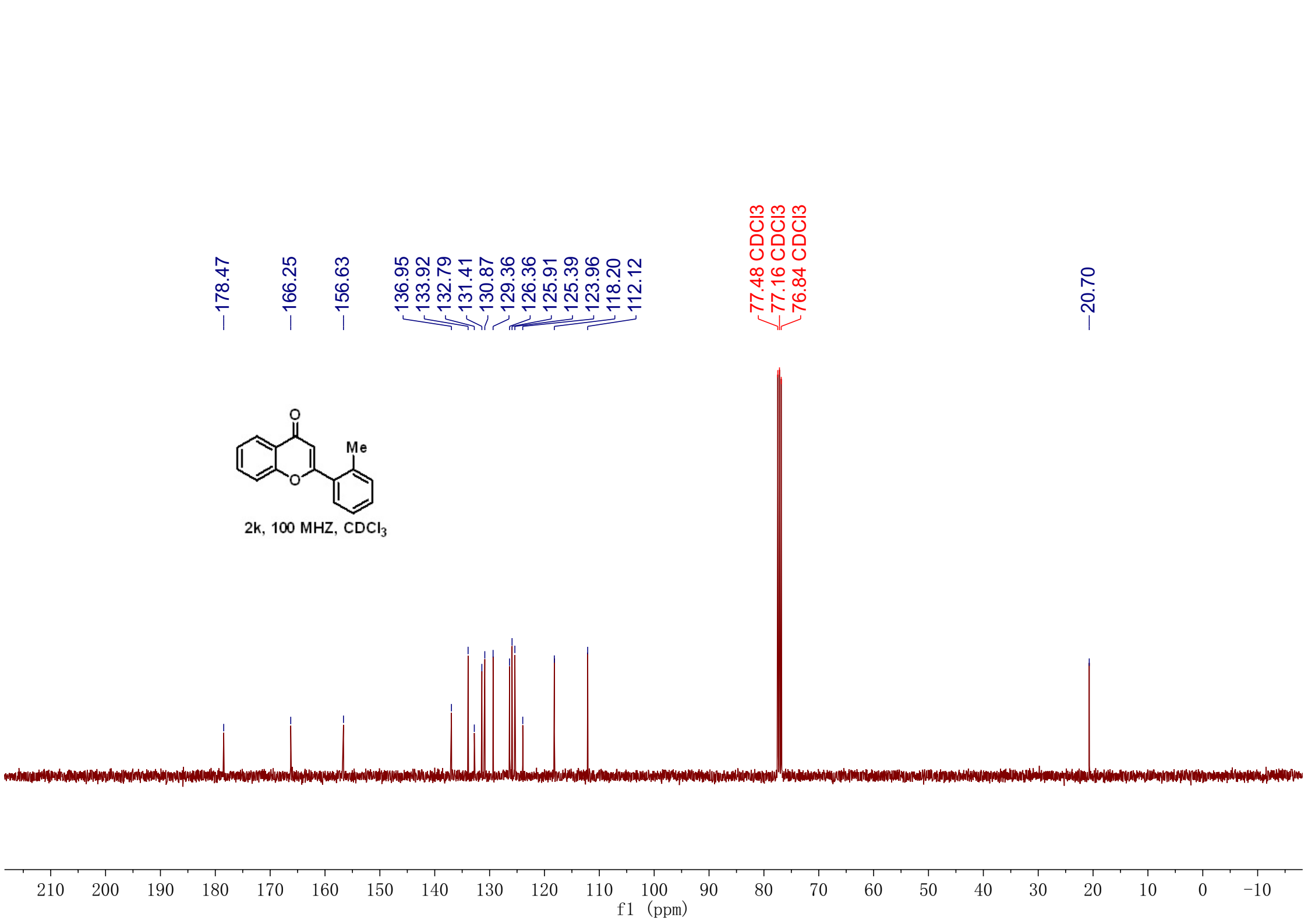

8.19
8.18
8.17
8.16
8.02
8.01
8.01
7.79
7.77
7.77
7.76
7.67
7.66
7.65
7.62
7.60
7.60
7.60
7.54
7.52
7.39
7.37
7.35
7.33
6.74
O
O
Br
2i, 400 MHZ, CDCl3
1.00
1.00
1.03
1.03
1.01
1.01
0.82
1.25
1.03
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)


177.98
161.42
155.99
134.35
133.93
133.58
130.48
129.07
125.61
125.37
124.71
123.78
123.15
118.04
107.99
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
Br
2i, 100 MHZ, CDCl3
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)

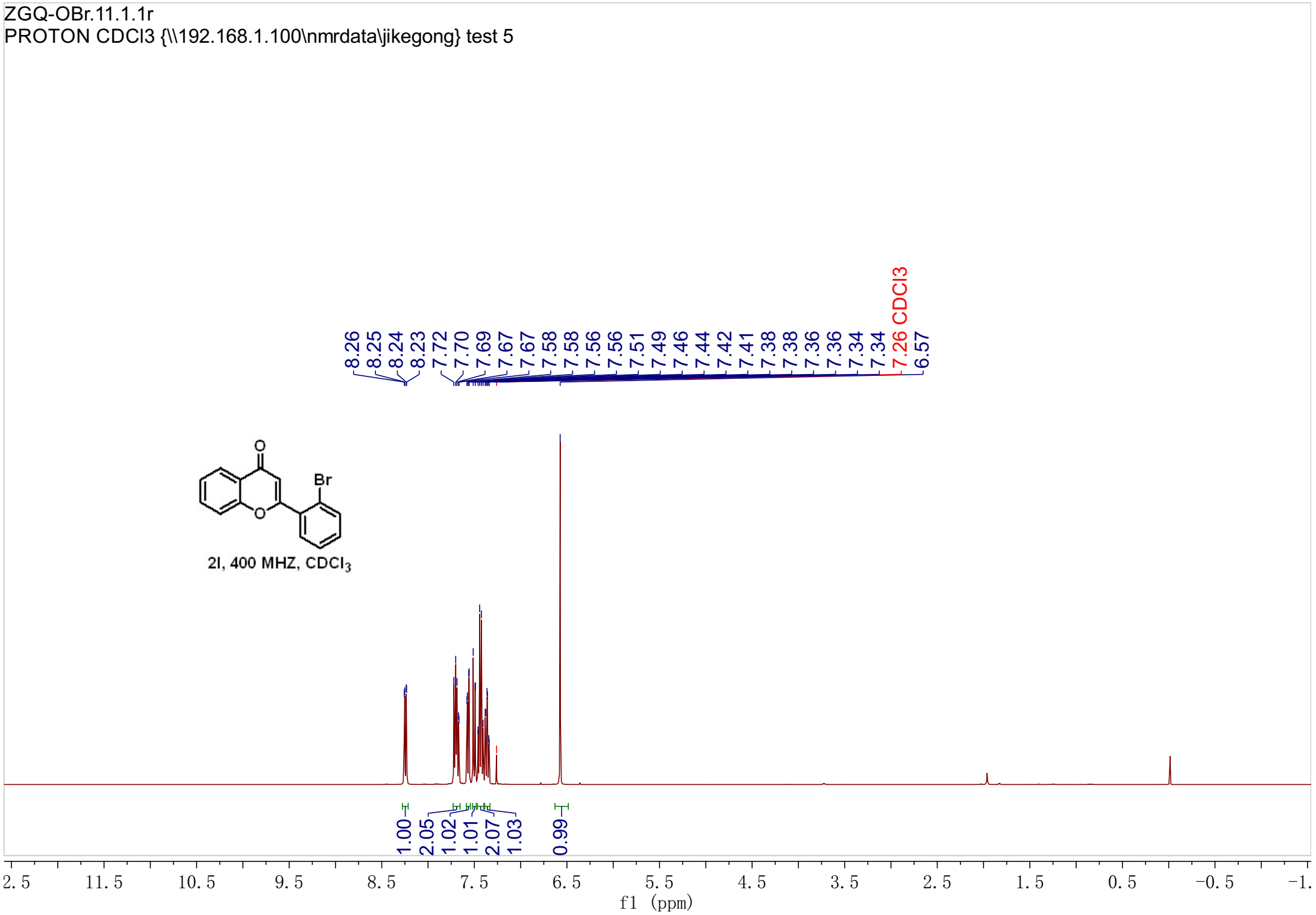

8.82
8.81
8.81
8.42
8.42
8.41
8.41
8.40
8.40
8.39
8.39
8.26
8.25
8.24
8.23
8.23
7.77
7.76
7.75
7.75
7.73
7.65
7.63
7.49
7.49
7.47
7.45
7.45
7.27 CDCl3
6.91
1.71 HDO
O
O
NO2
2j, 400 MHZ, CDCl3
1.03
1.02
2.03
2.09
1.03
1.04
1.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


178.00
160.51
156.15
148.81
134.33
133.68
131.76
130.31
125.93
125.86
125.76
123.93
121.28
118.19
108.86
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
O
O
NO2
2j, 100 MHZ, CDCl3
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)

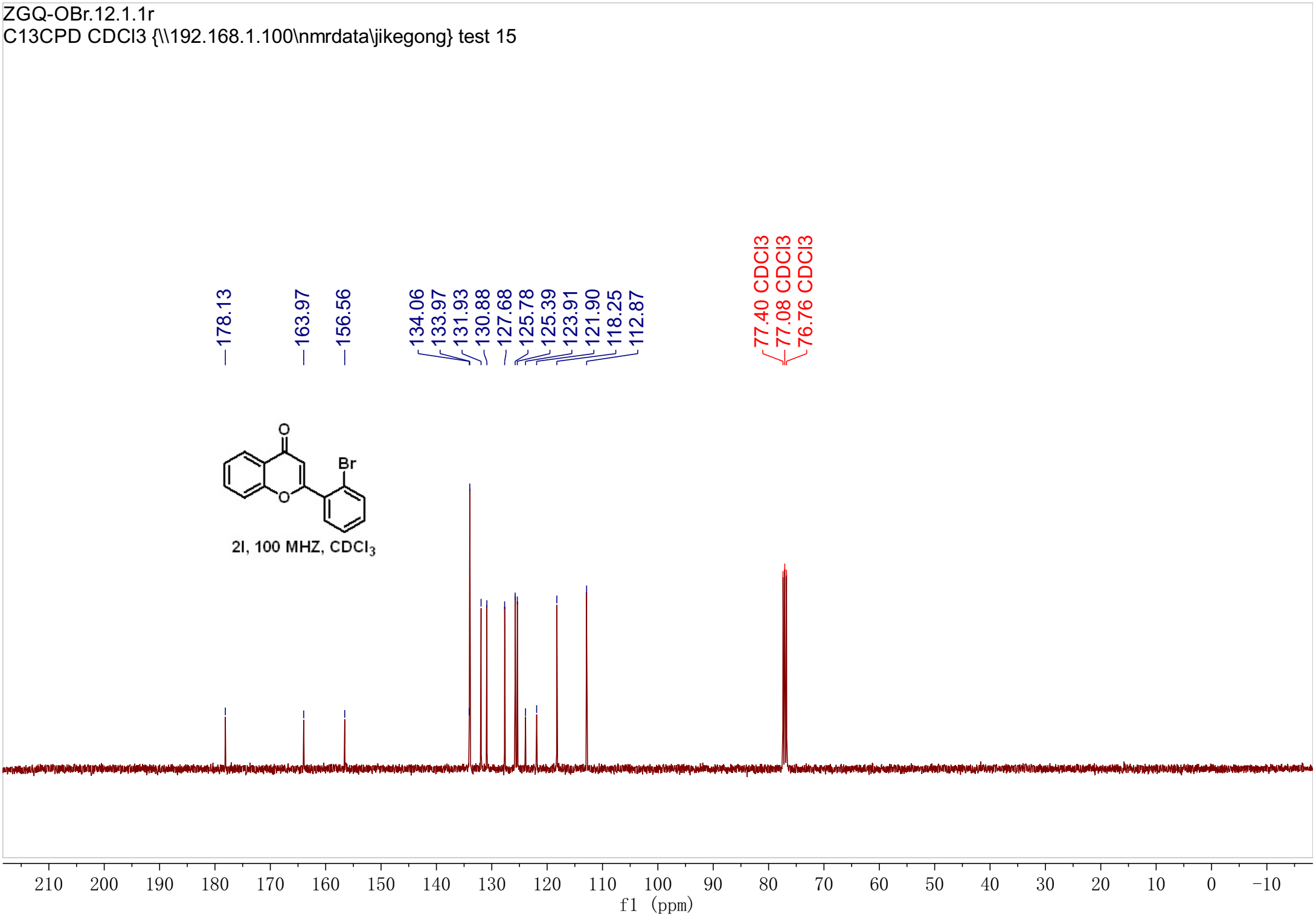

8.28
8.27
8.26
8.25
7.72
7.71
7.70
7.68
7.67
7.55
7.54
7.52
7.51
7.48
7.46
7.44
7.42
7.41
7.34
7.32
7.31
7.26 CDCl3
6.49
2.49
O
Me
O
2k, 400 MHZ, CDCl3
1.00
1.08
2.09
2.09
2.08
0.98
3.17
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
f1 (ppm)


178.47
166.25
156.63
136.95
133.92
132.79
131.41
130.87
129.36
126.36
125.91
125.39
123.96
118.20
112.12
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
20.70
O
Me
O
2k, 100 MHZ, CDCl3
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)

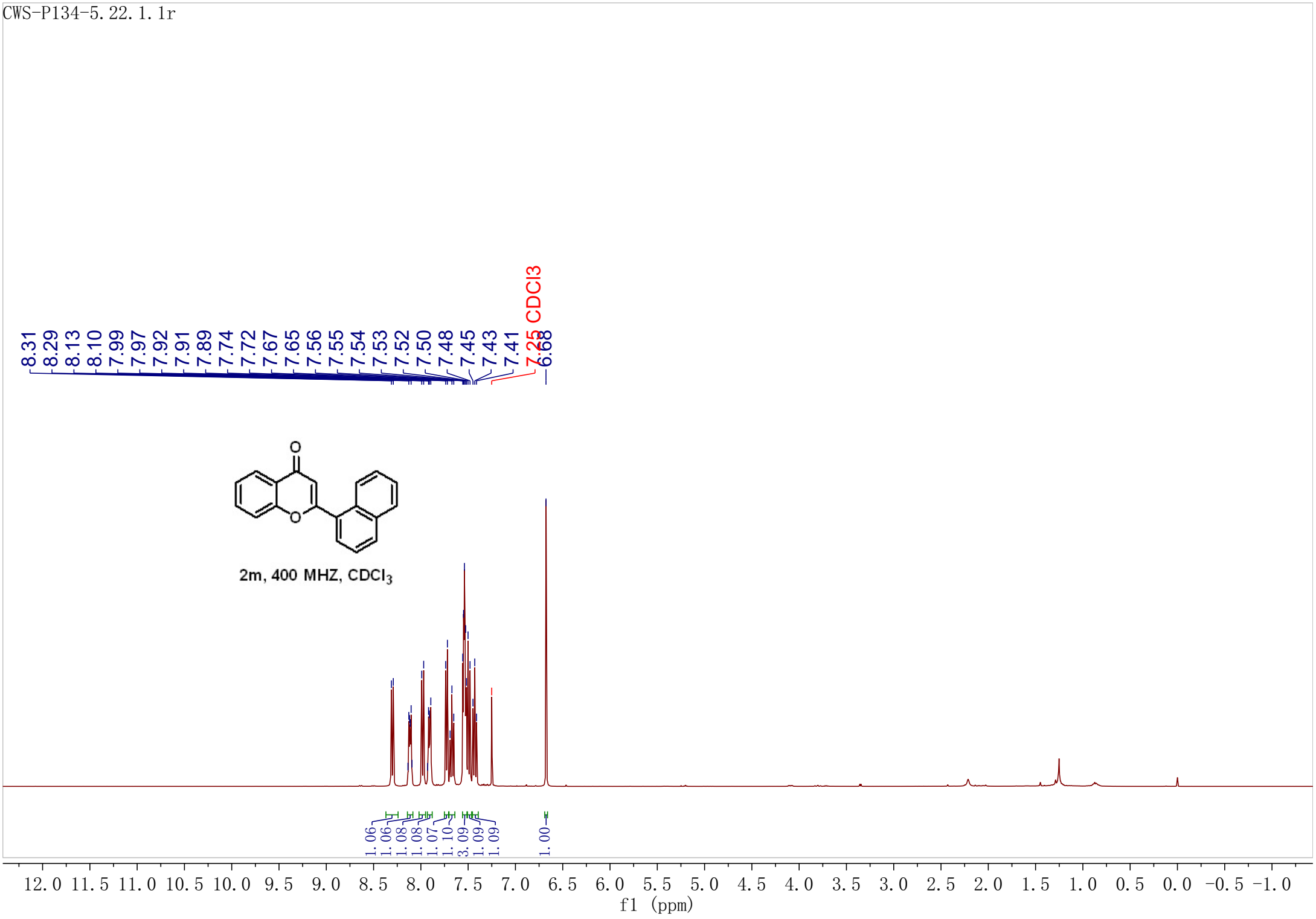
ZGQ-OBr.11.1.1r
PROTON CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 5



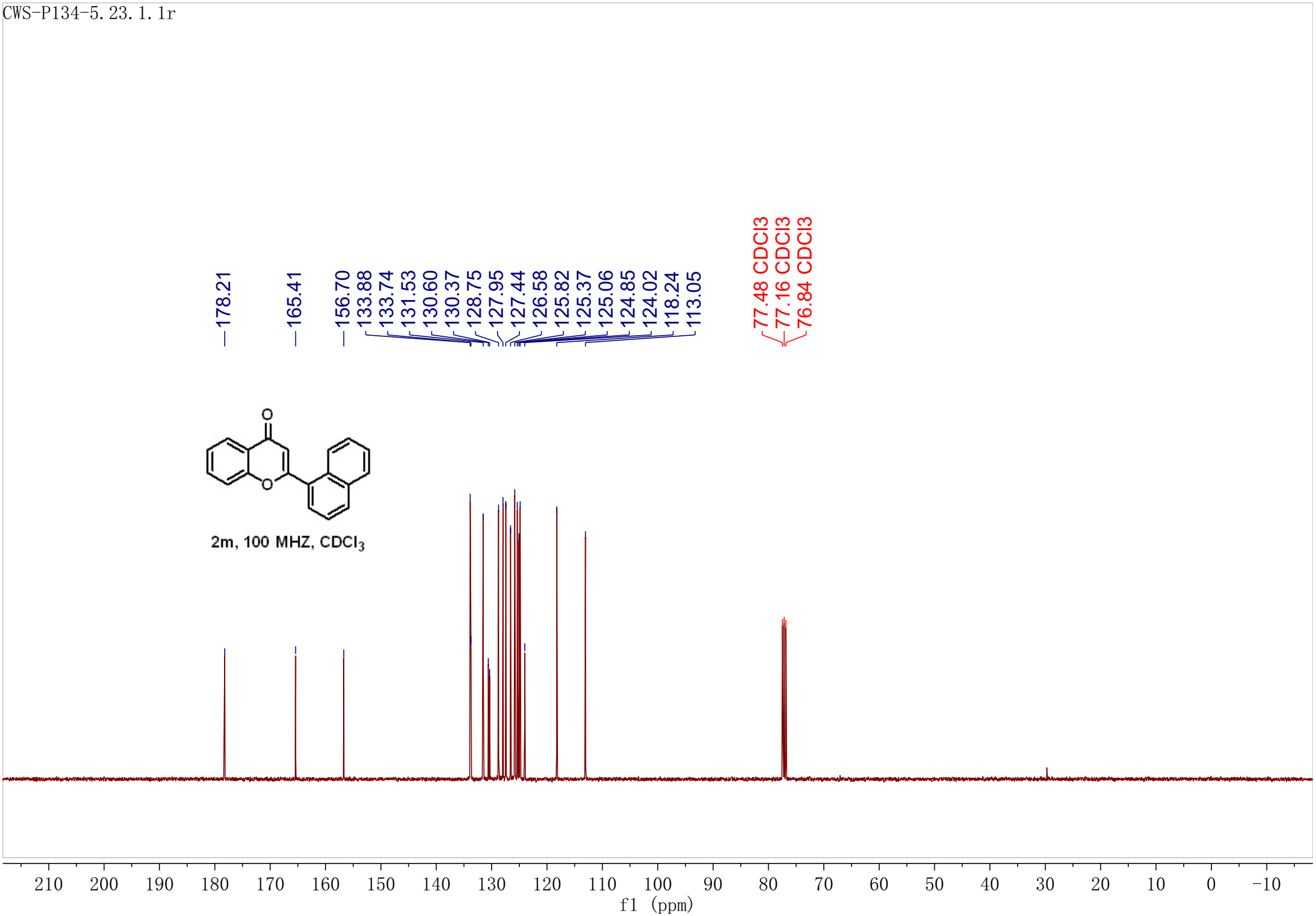
ZGQ-OBr.12.1.1r
C13CPD CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 15



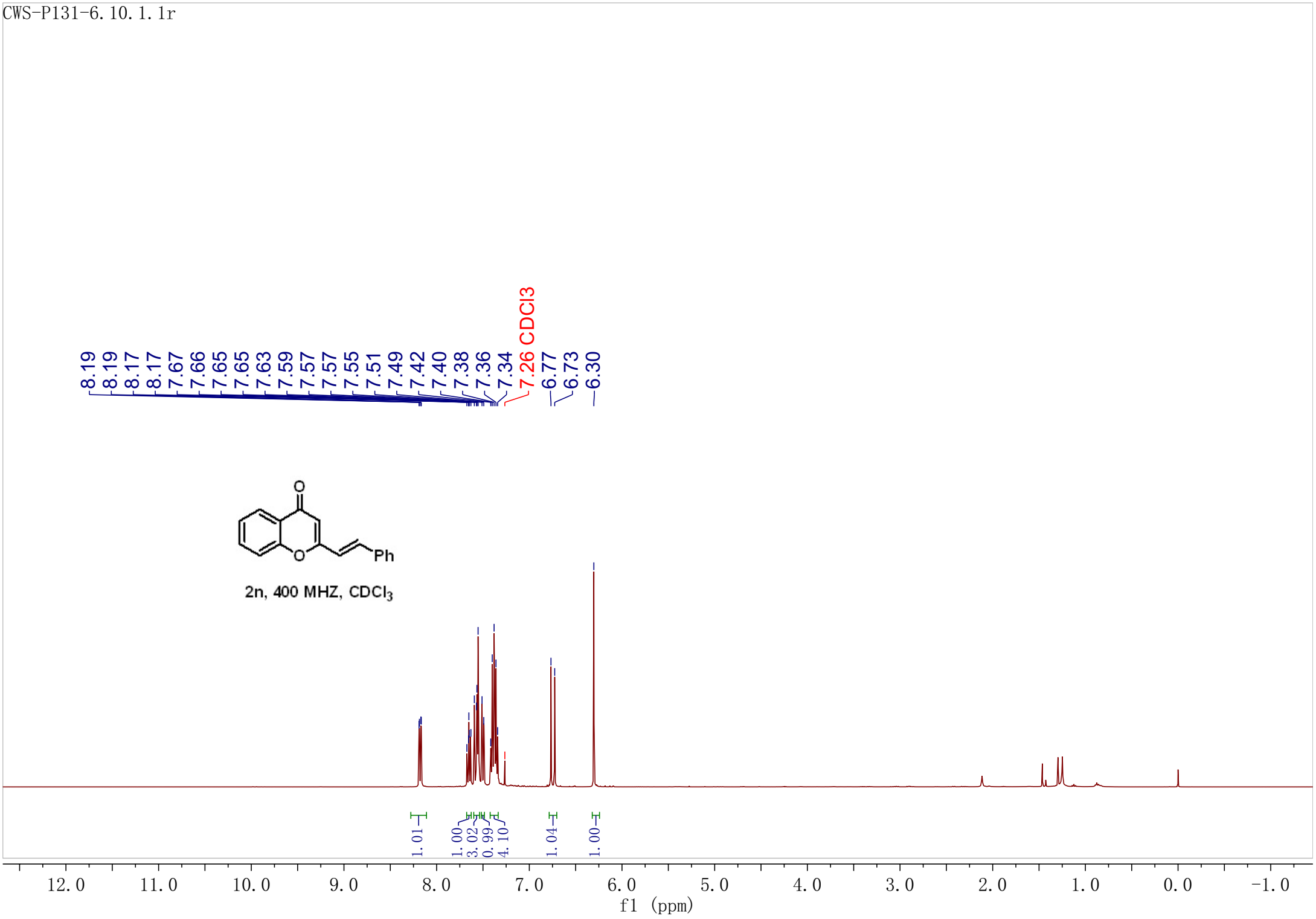
CWS-P134-5.22.1.1r



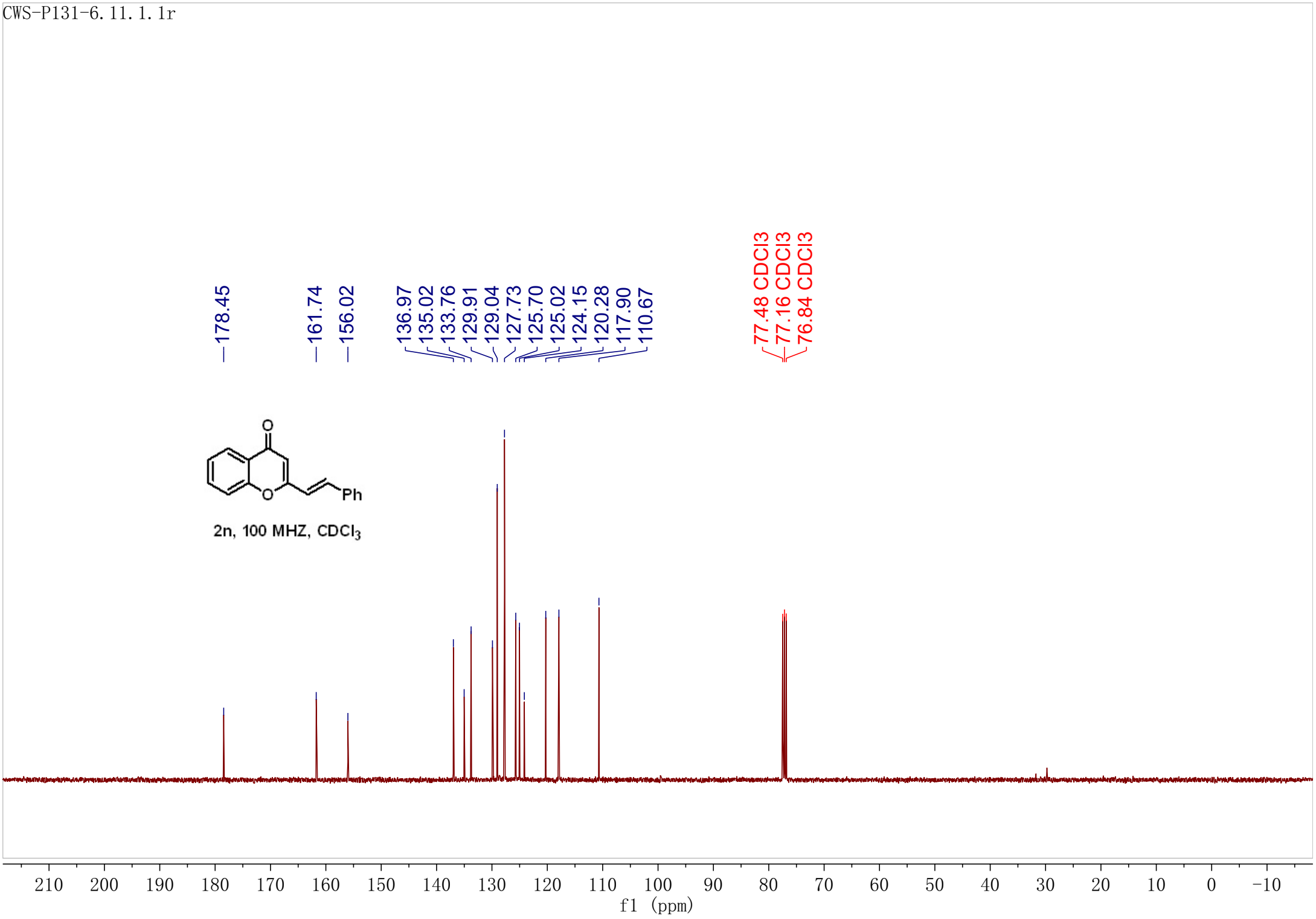
CWS-P134-5.23.1.1r

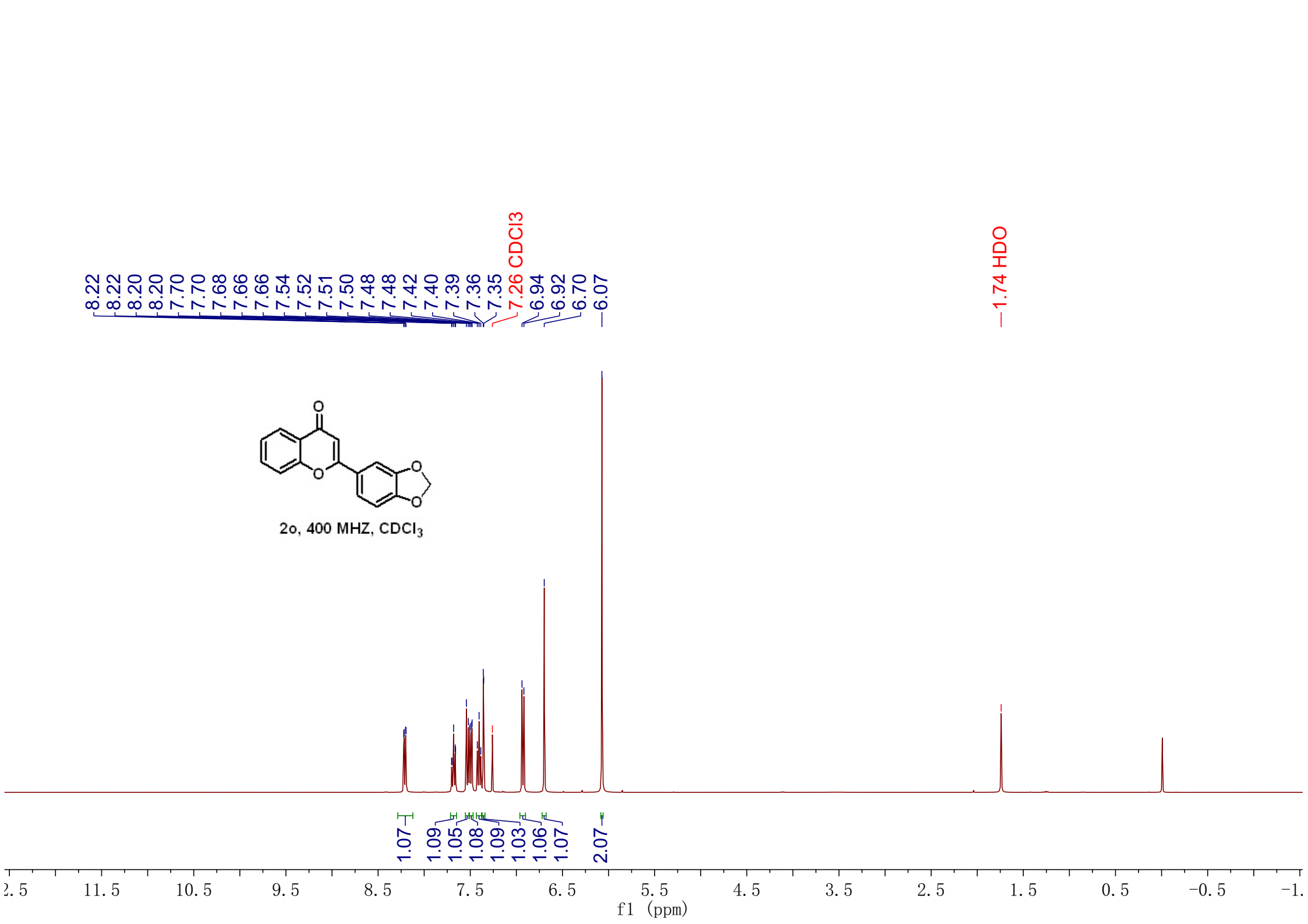


CWS-P131-6.10.1.1r

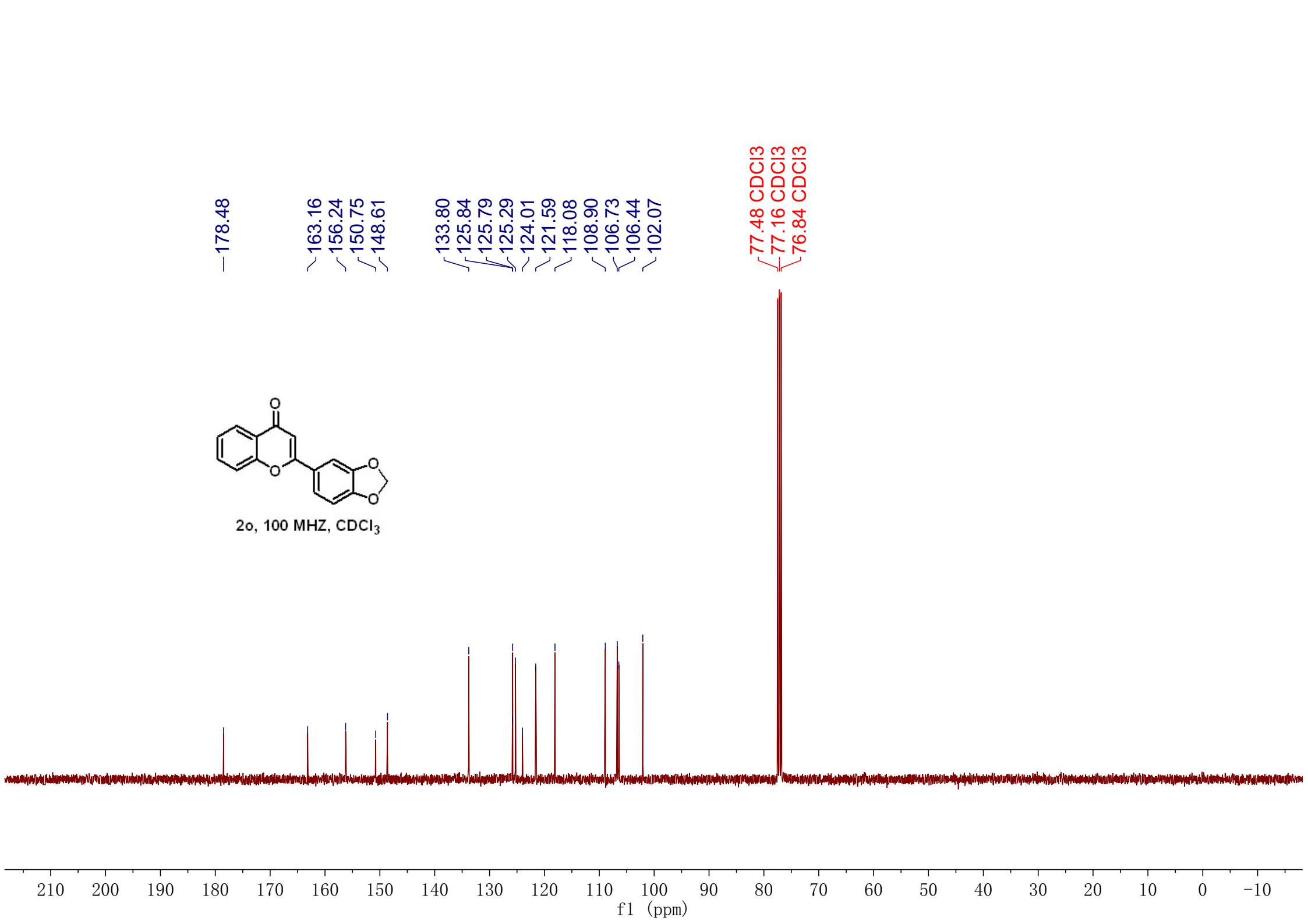


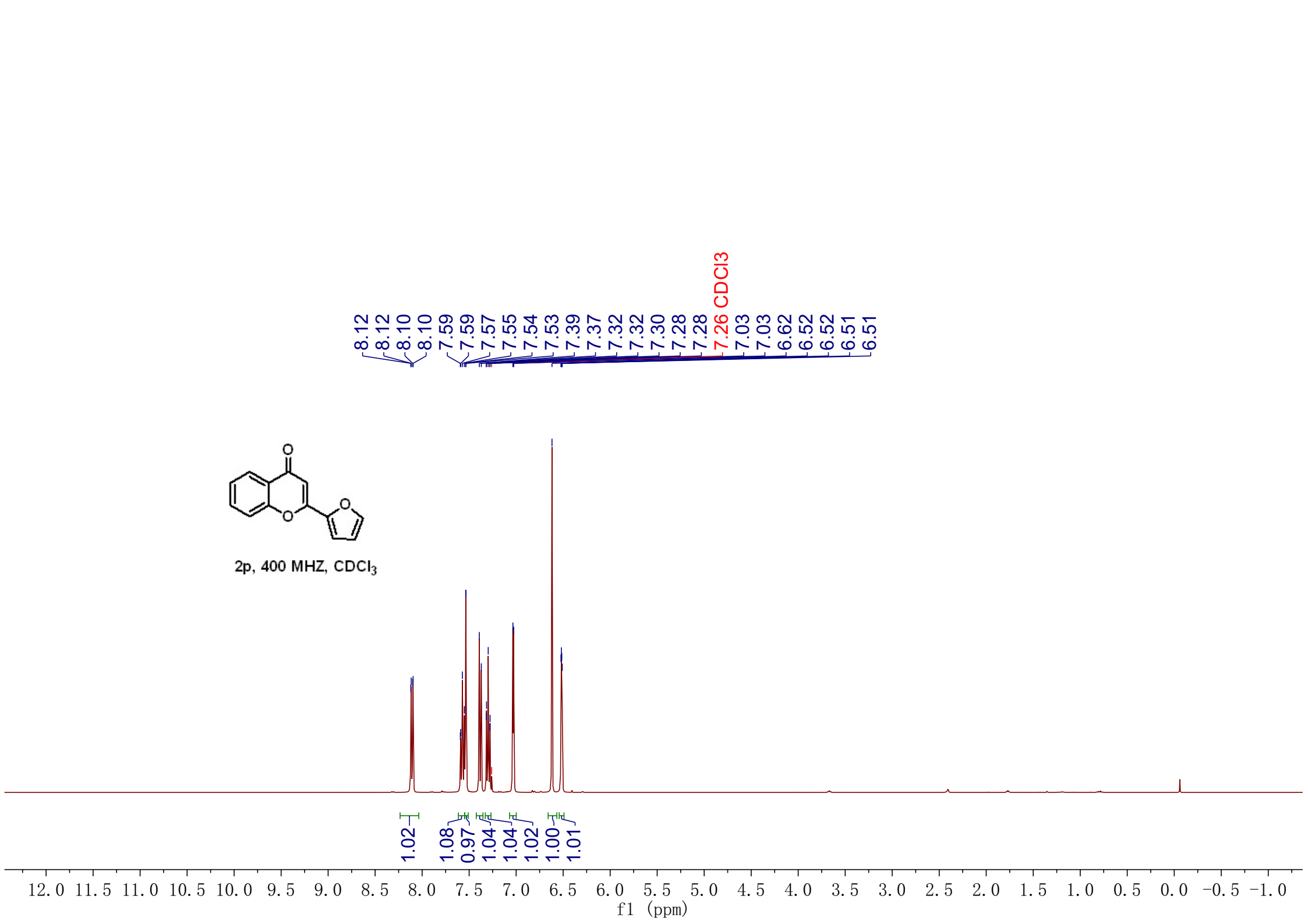
CWS-P131-6.11.1.1r

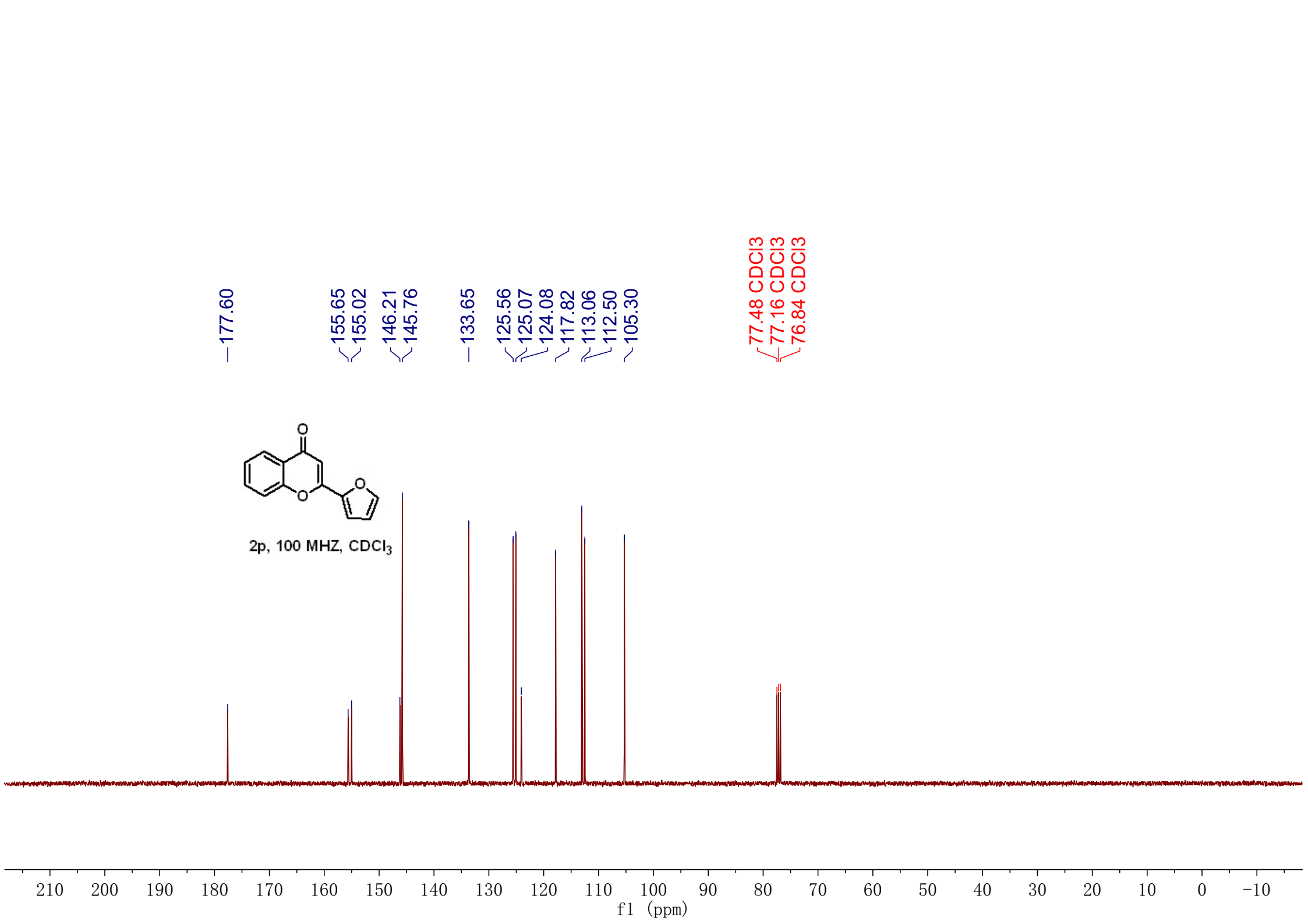




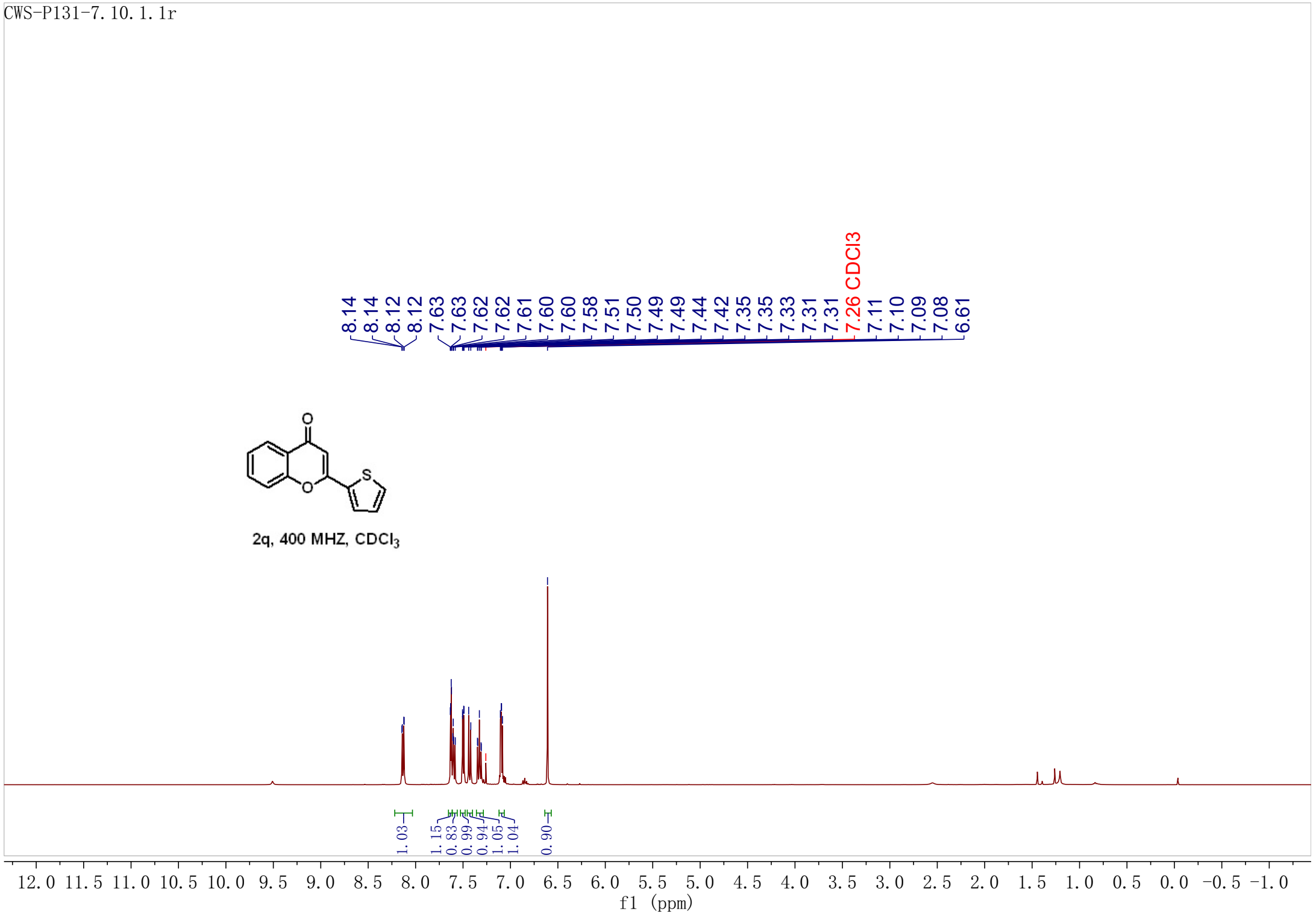
2o, 400 MHZ, $CDCl_3$


178.48
163.16
156.24
150.75
148.61
133.80
125.84
125.79
125.29
124.01
121.59
118.08
108.90
106.73
106.44
102.07
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
2o, 100 MHZ, CDCl3
f1 (ppm)

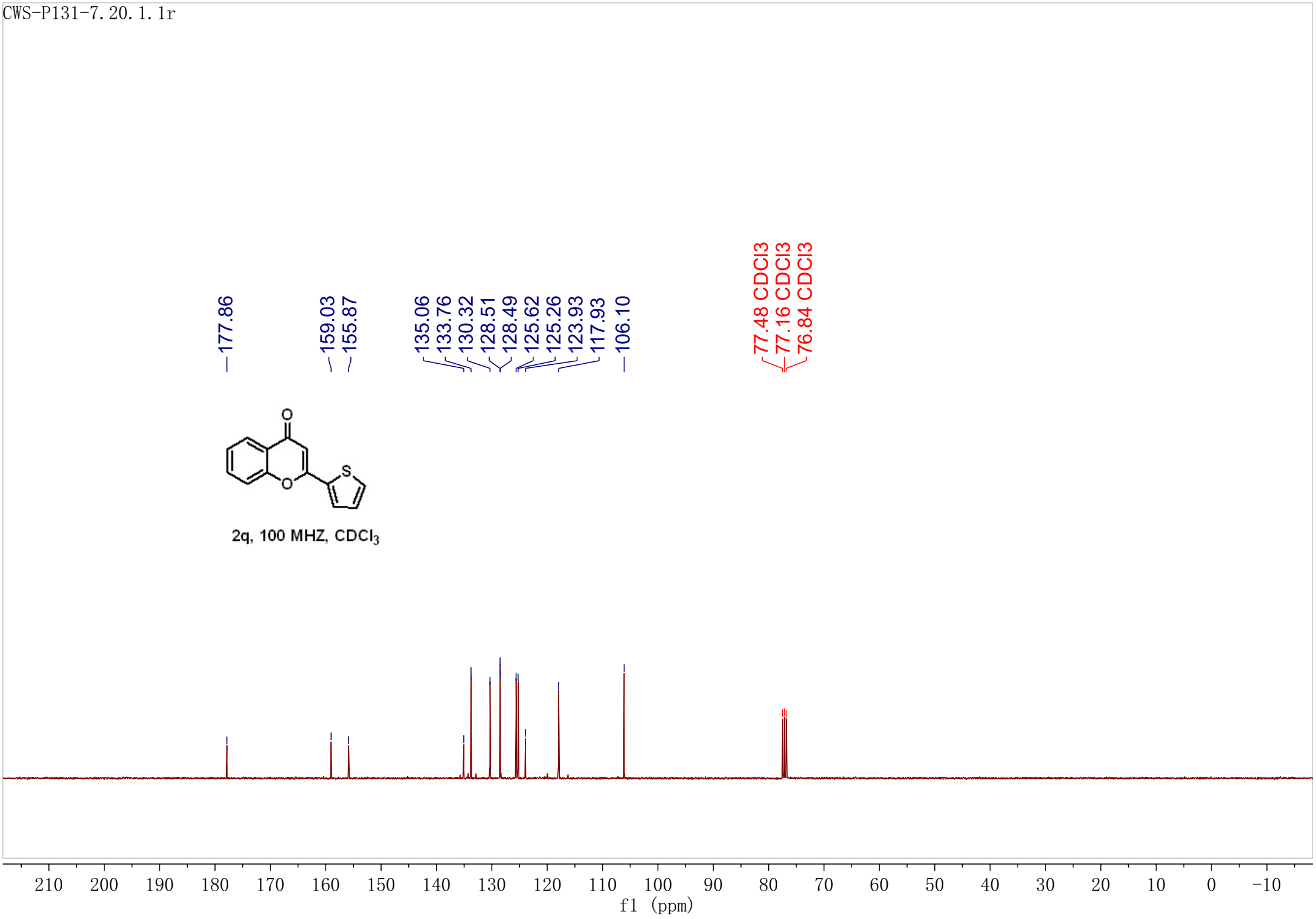

8.12
8.12
8.10
8.10
7.59
7.59
7.57
7.55
7.54
7.53
7.39
7.37
7.32
7.32
7.30
7.28
7.28
7.26 CDCl3
7.03
7.03
6.62
6.52
6.52
6.51
6.51
2p, 400 MHZ, CDCl3
1.02
1.08
0.97
1.04
1.04
1.02
1.00
1.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


177.60
155.65
155.02
146.21
145.76
133.65
125.56
125.07
124.08
117.82
113.06
112.50
105.30
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
O
2p, 100 MHZ, CDCl3
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)

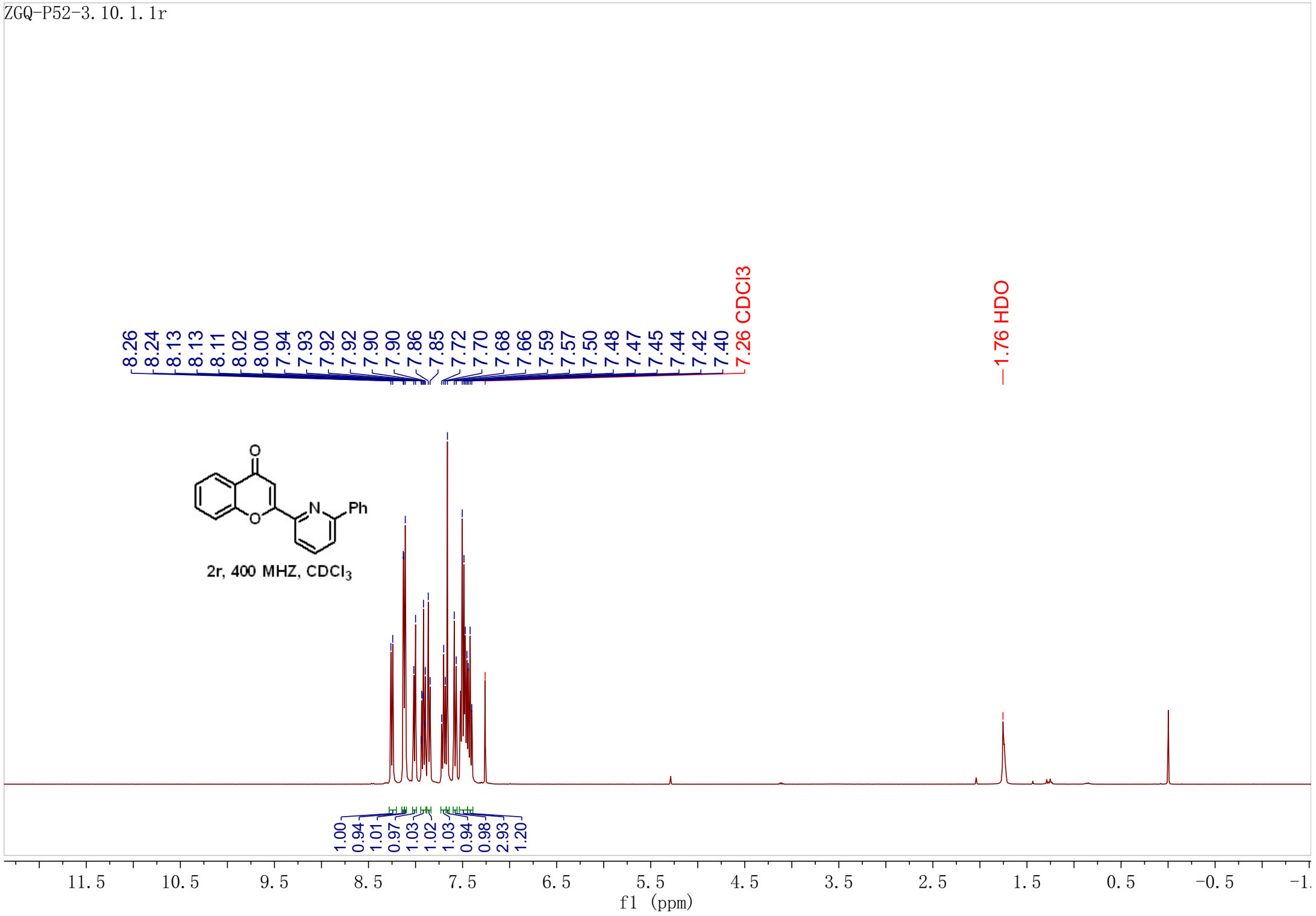
CWS-P131-7.10.1.1r



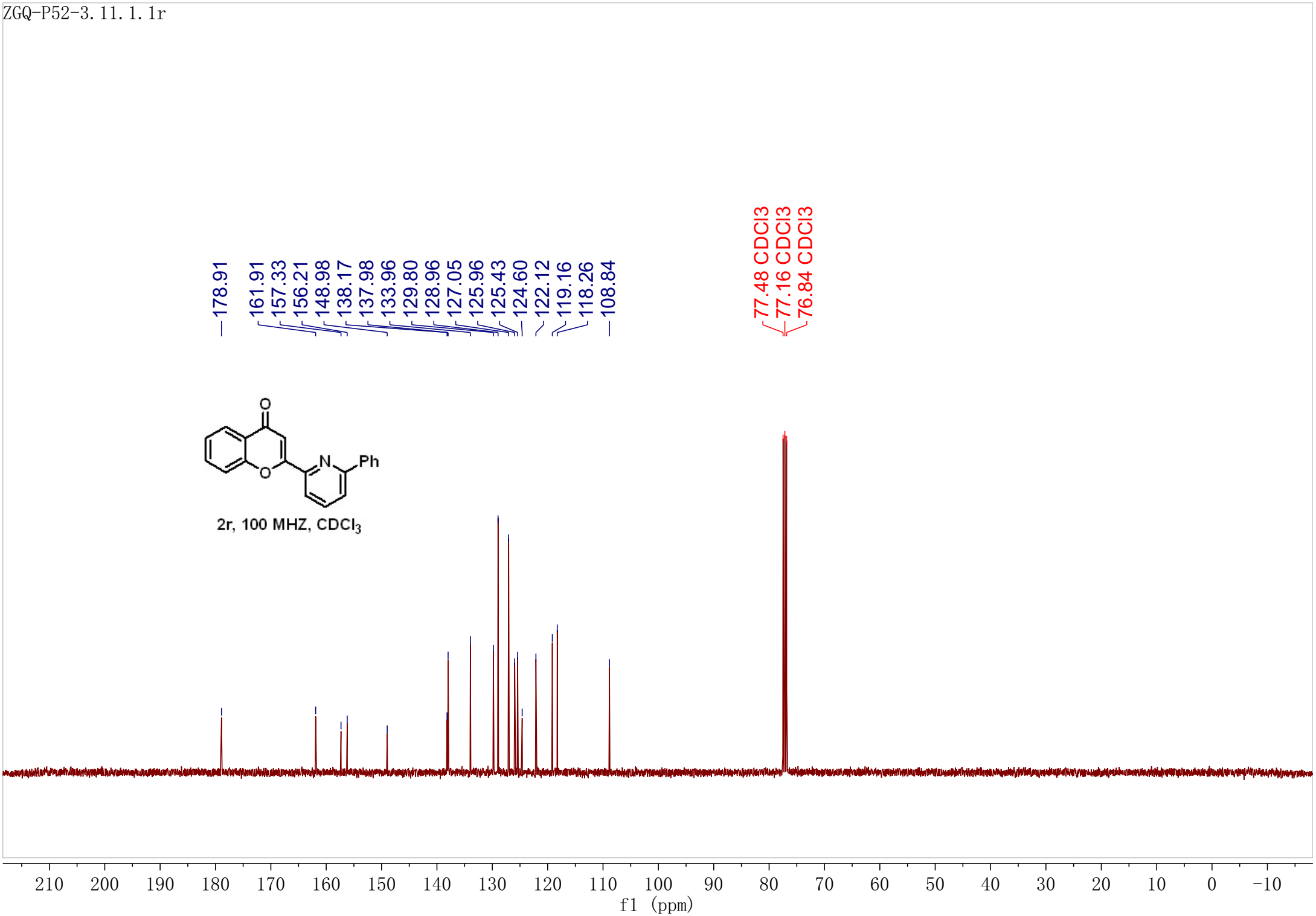
CWS-P131-7.20.1.1r



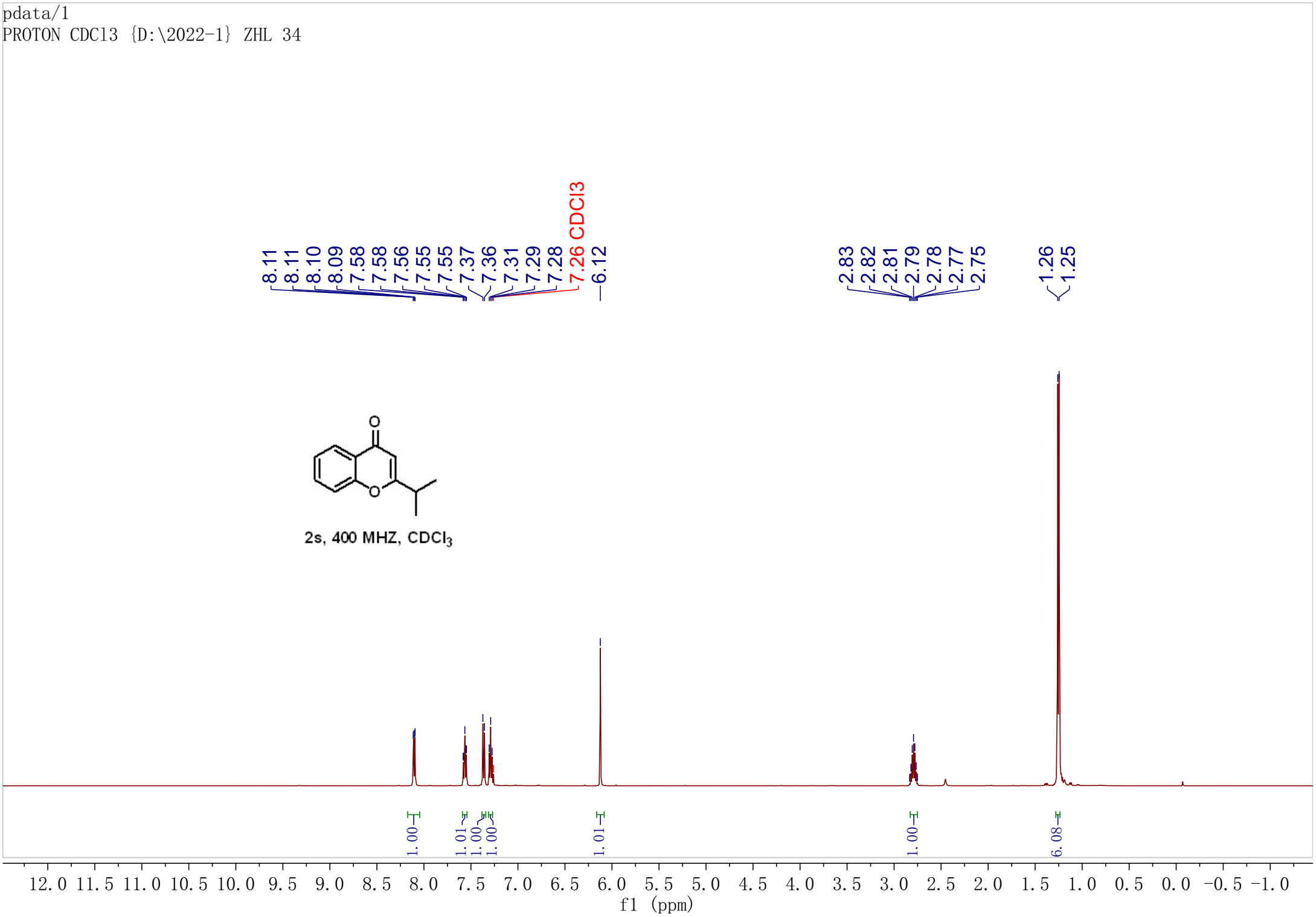
ZGQ-P52-3.10.1.1r



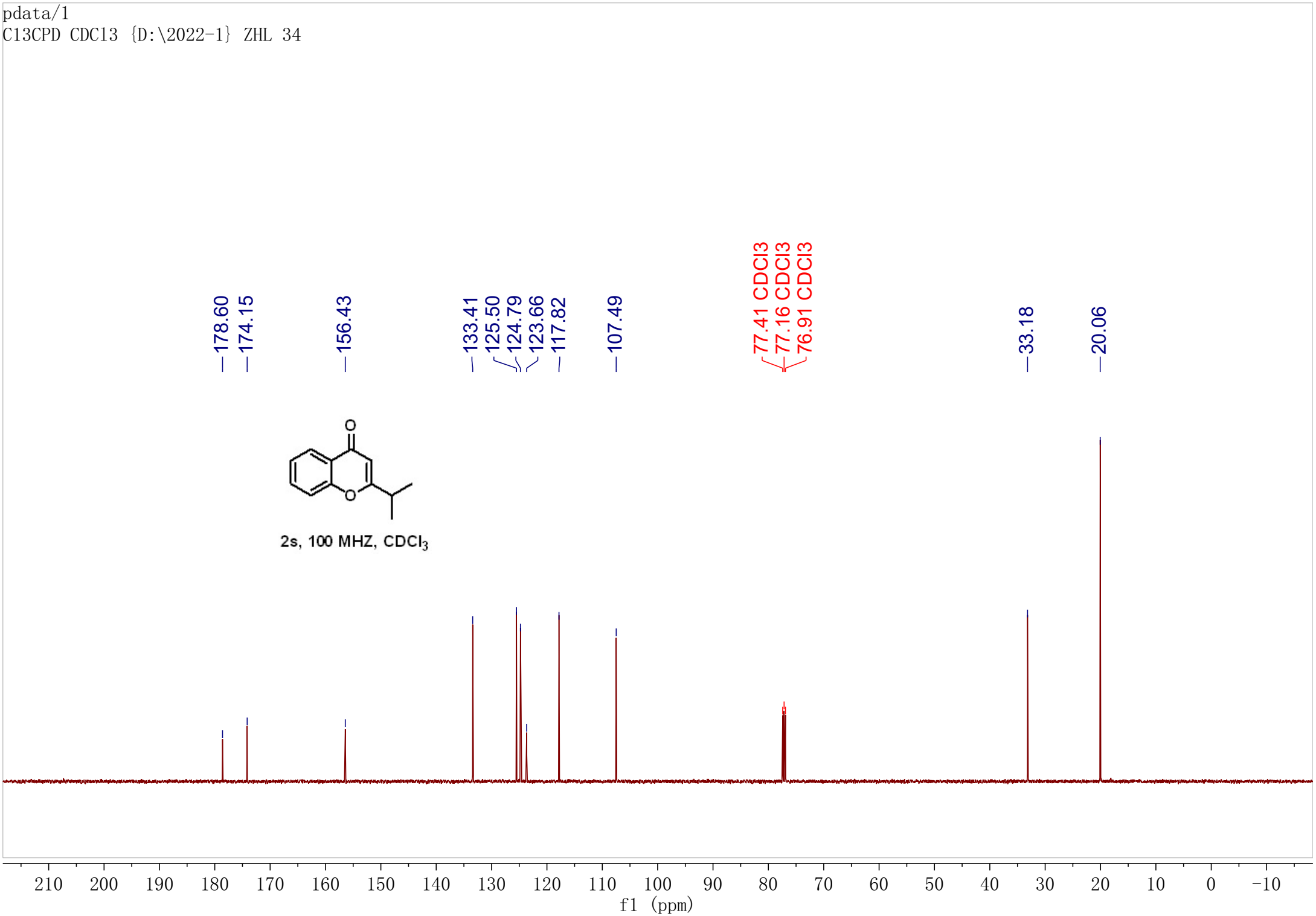
ZGQ-P52-3.11.1.1r



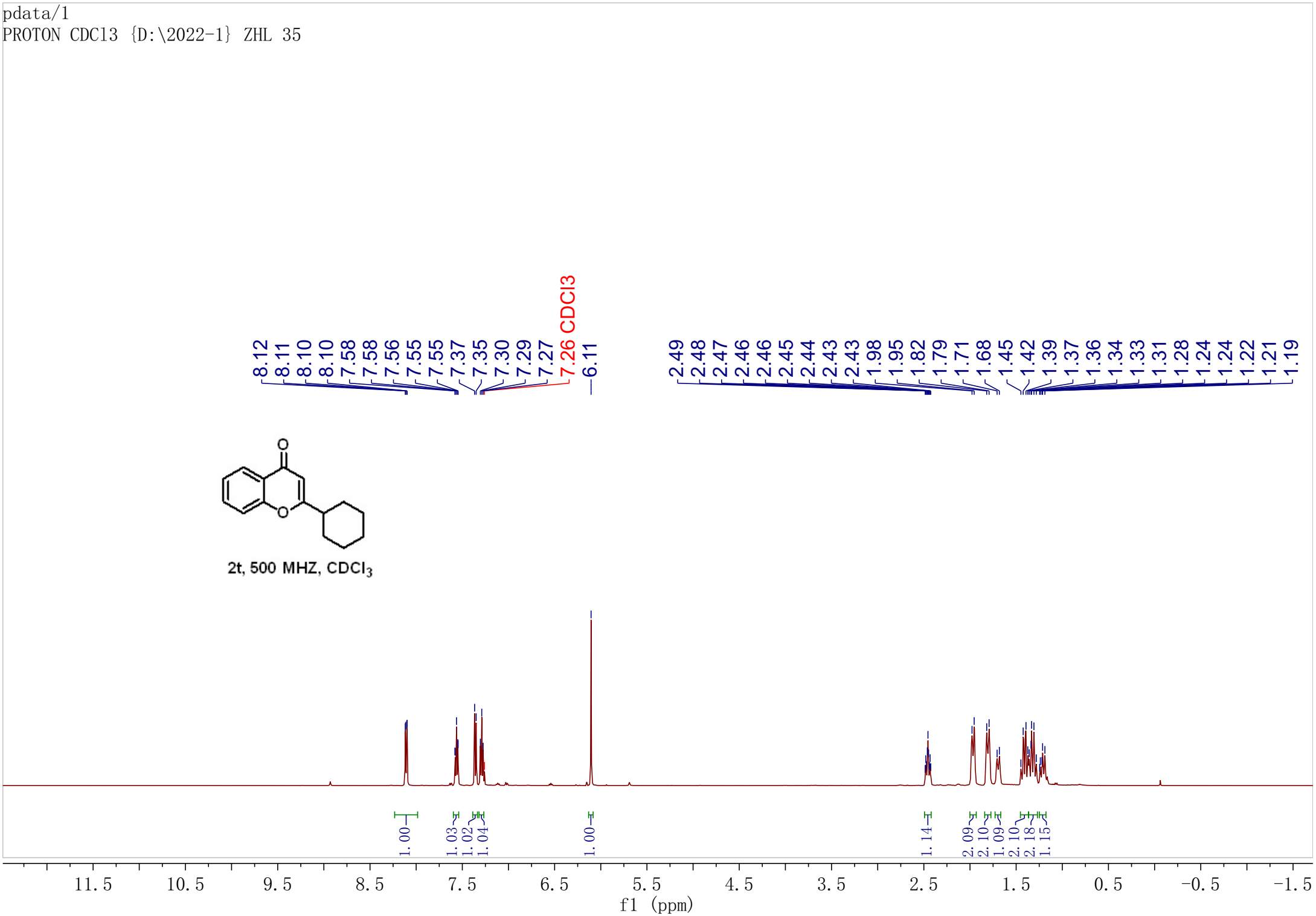
pdata/1
PROTON CDCl3 {D:\2022-1} ZHL 34



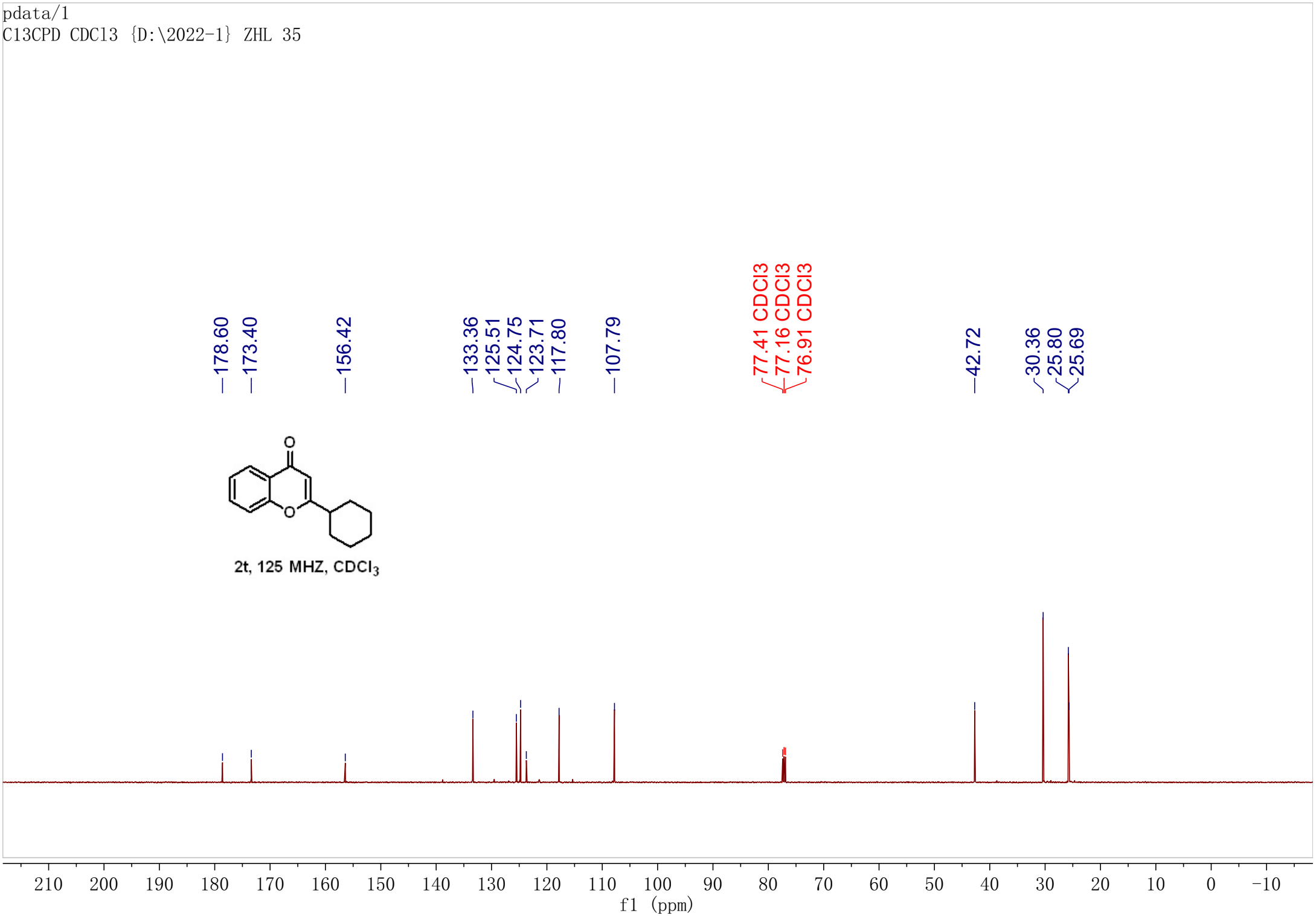
pdata/1
C13CPD CDCl3 {D:\2022-1} ZHL 34

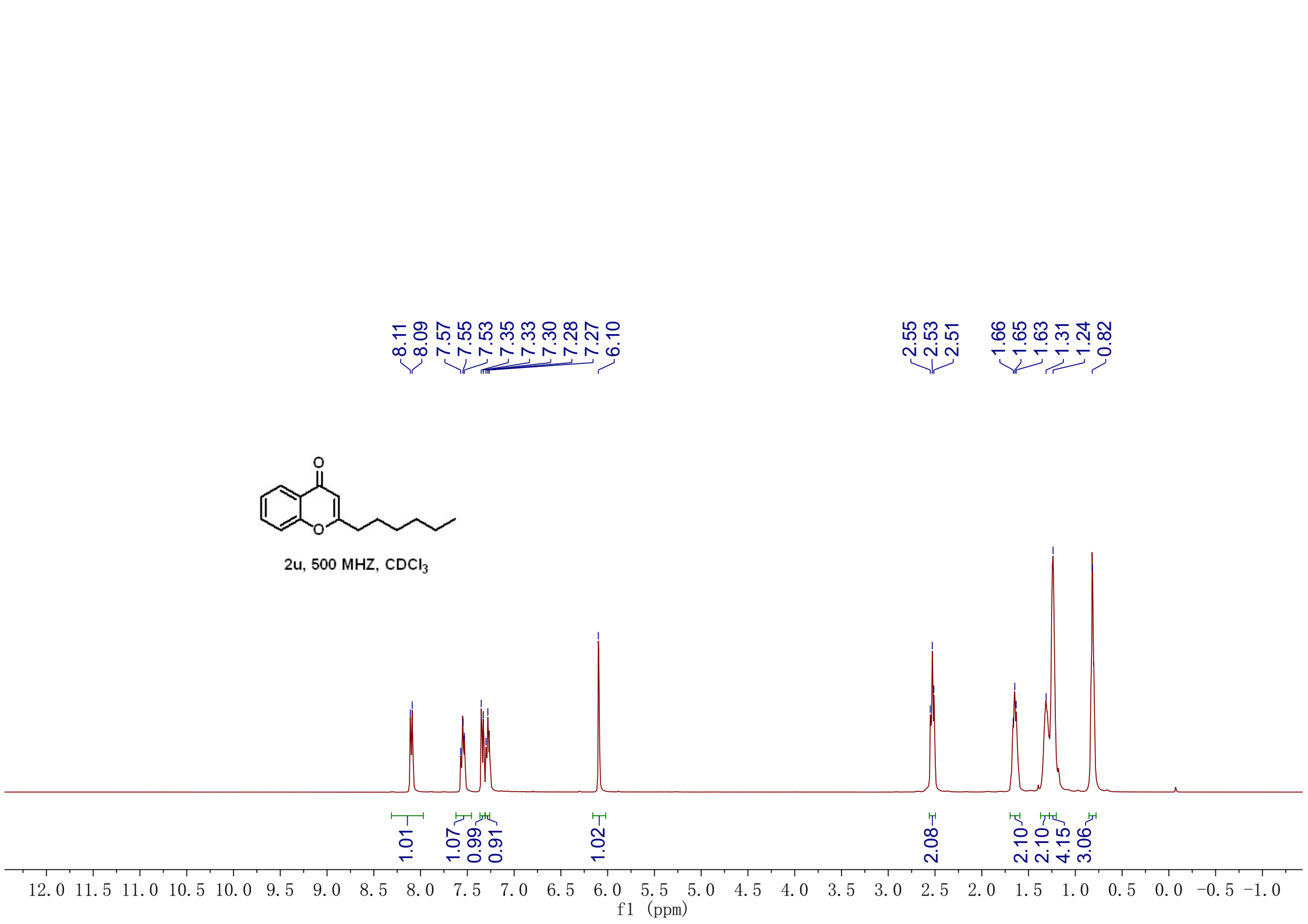


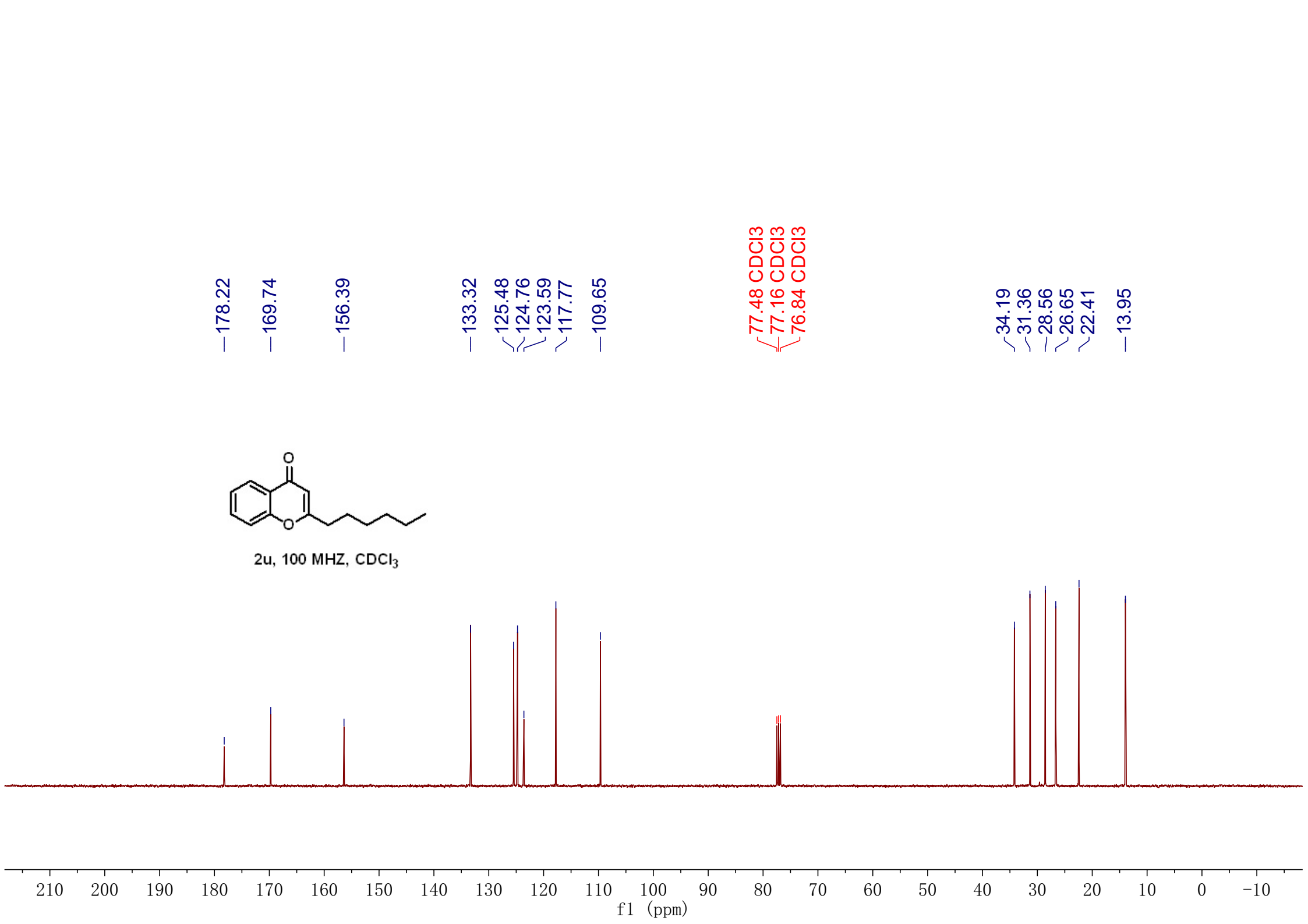
pdata/1
PROTON CDCl3 {D:\2022-1} ZHL 35



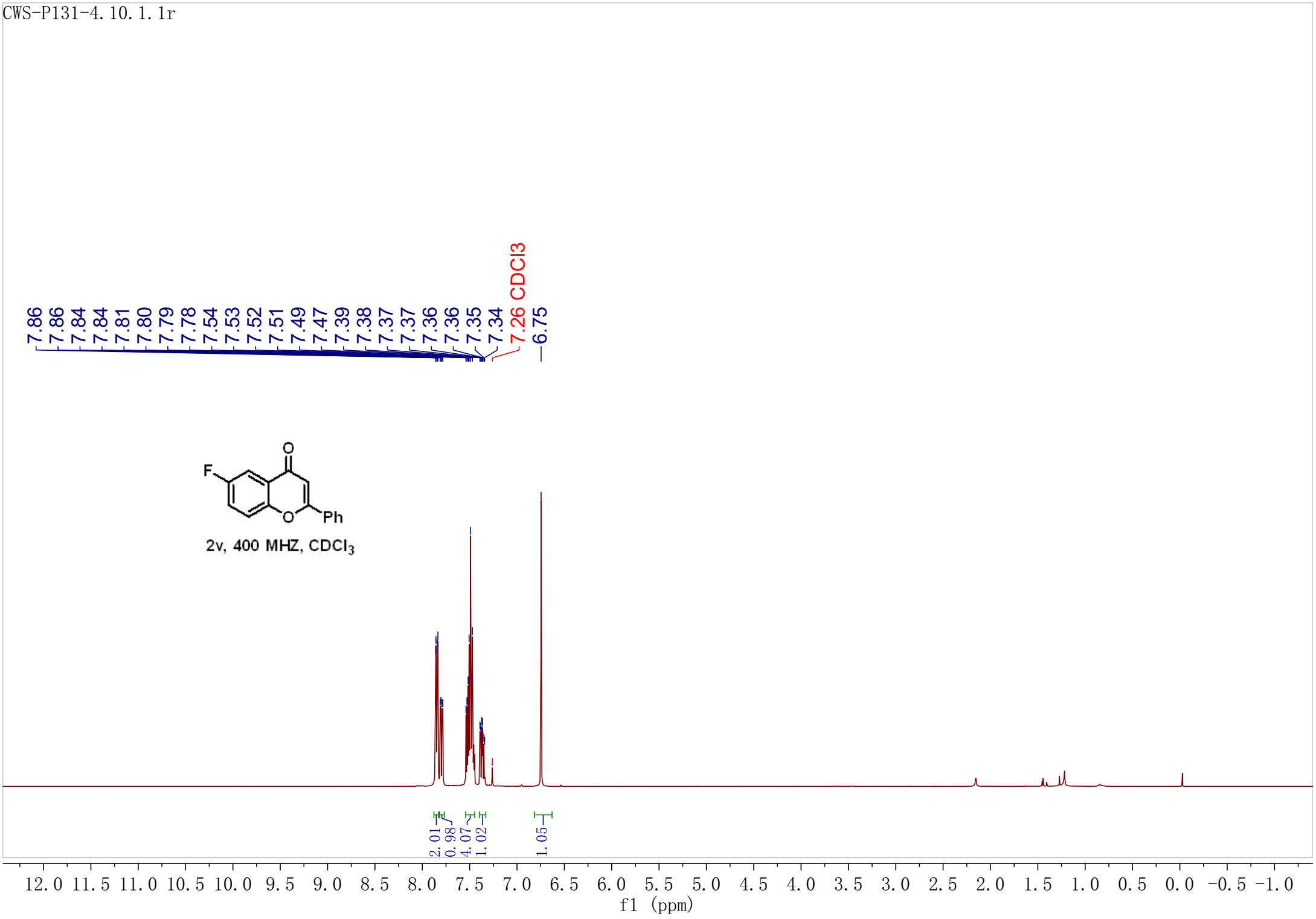
pdata/1
C13CPD CDCl3 {D:\2022-1} ZHL 35



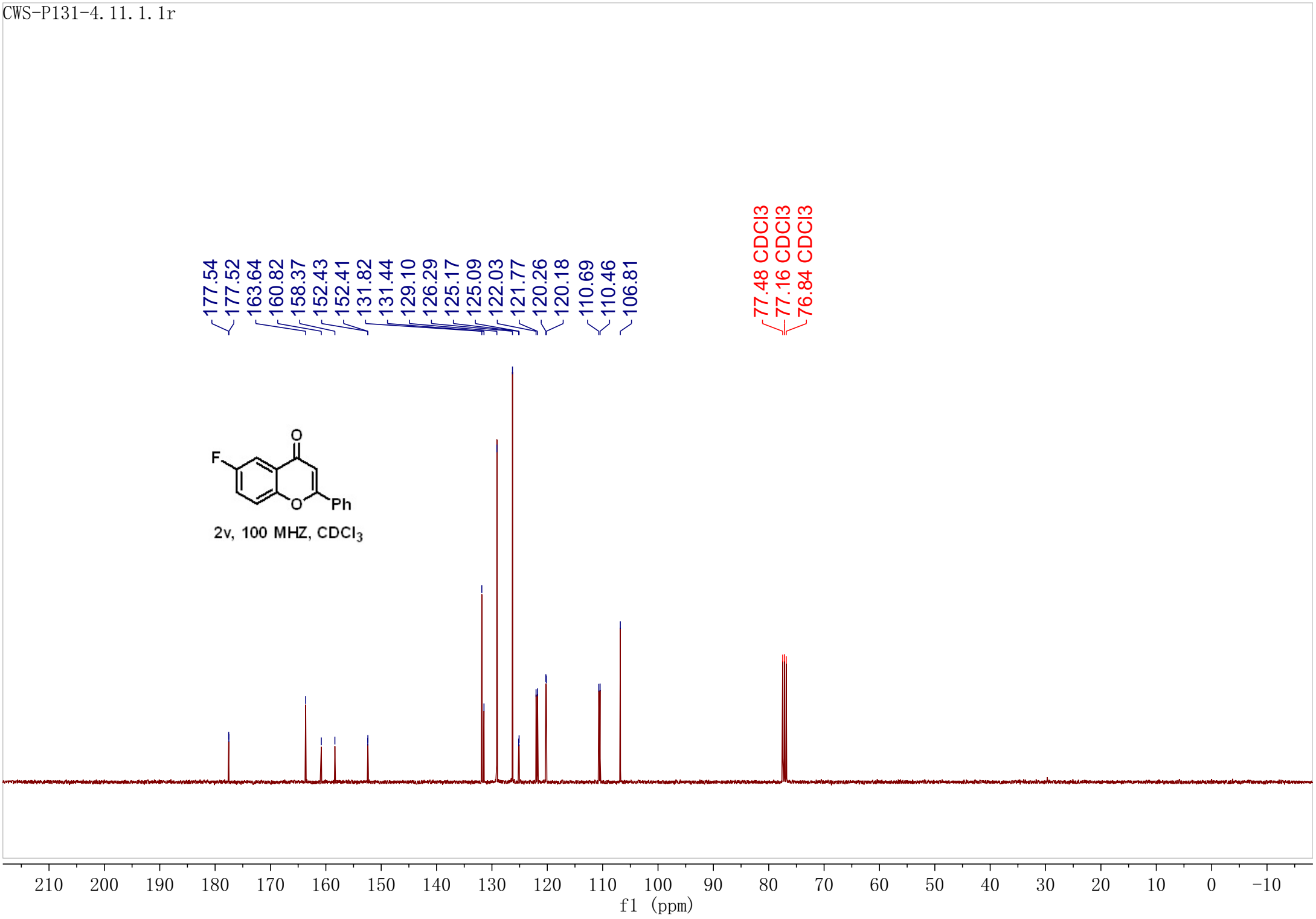

8.11
8.09
7.57
7.55
7.53
7.35
7.33
7.30
7.28
7.27
6.10
2.55
2.53
2.51
1.66
1.65
1.63
1.31
1.24
0.82
O
O
2u, 500 MHZ, CDCl3
1.01
1.07
0.99
0.91
1.02
2.08
2.10
2.10
4.15
3.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


178.22
169.74
156.39
133.32
125.48
124.76
123.59
117.77
109.65
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
34.19
31.36
28.56
26.65
22.41
13.95
O
O
2u, 100 MHZ, CDCl3
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)

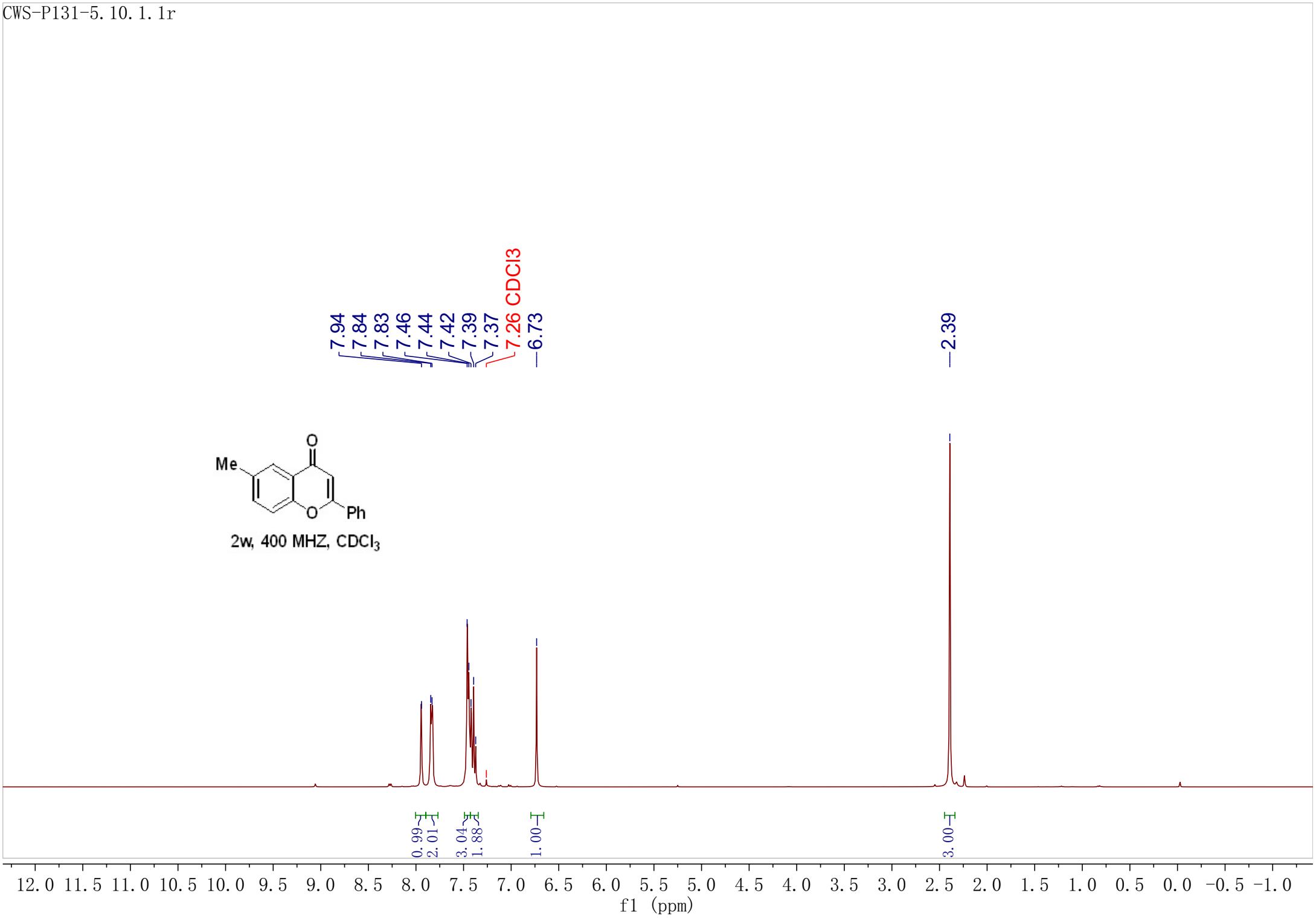
CWS-P131-4.10.1.1r



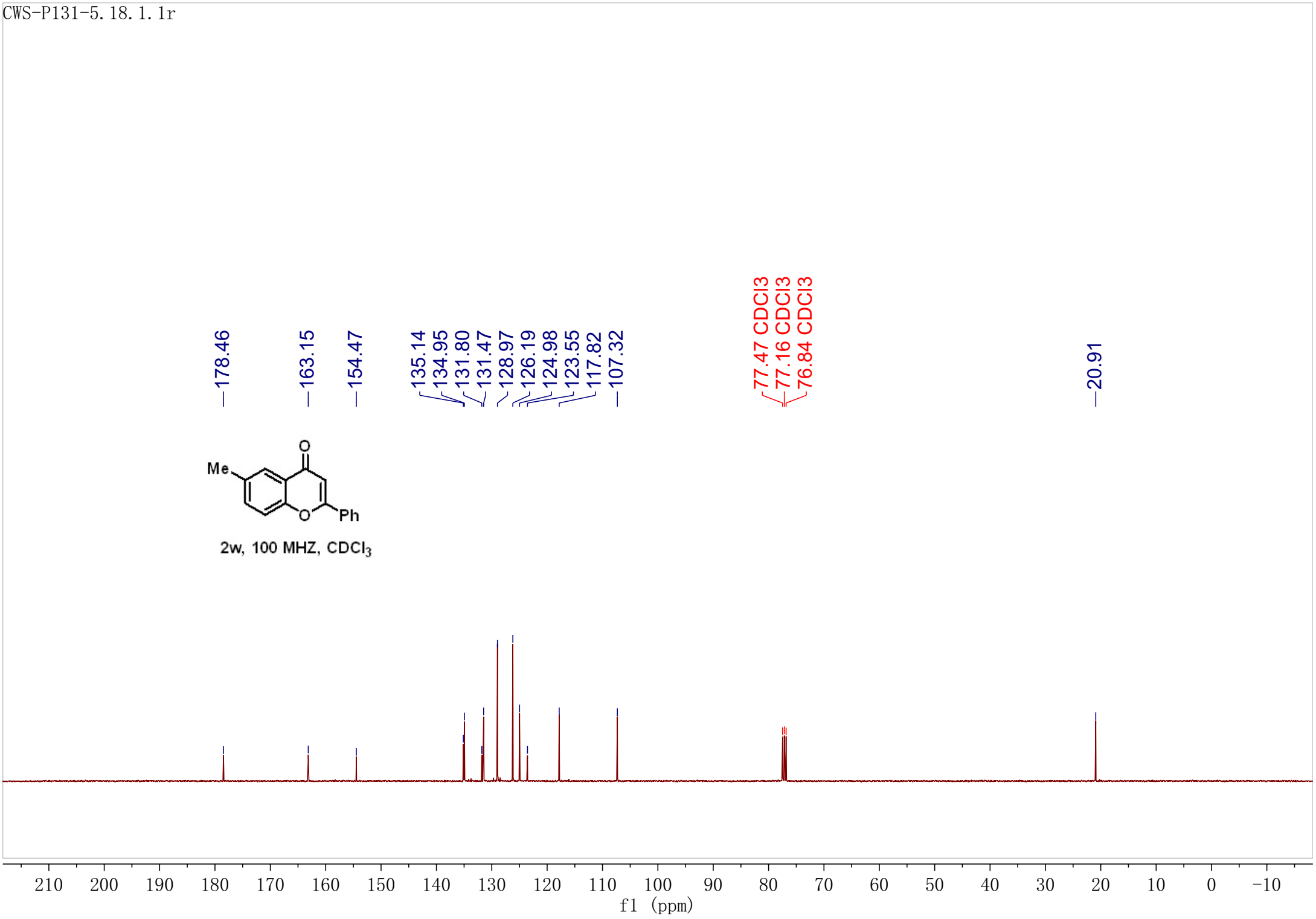
CWS-P131-4.11.1.1r

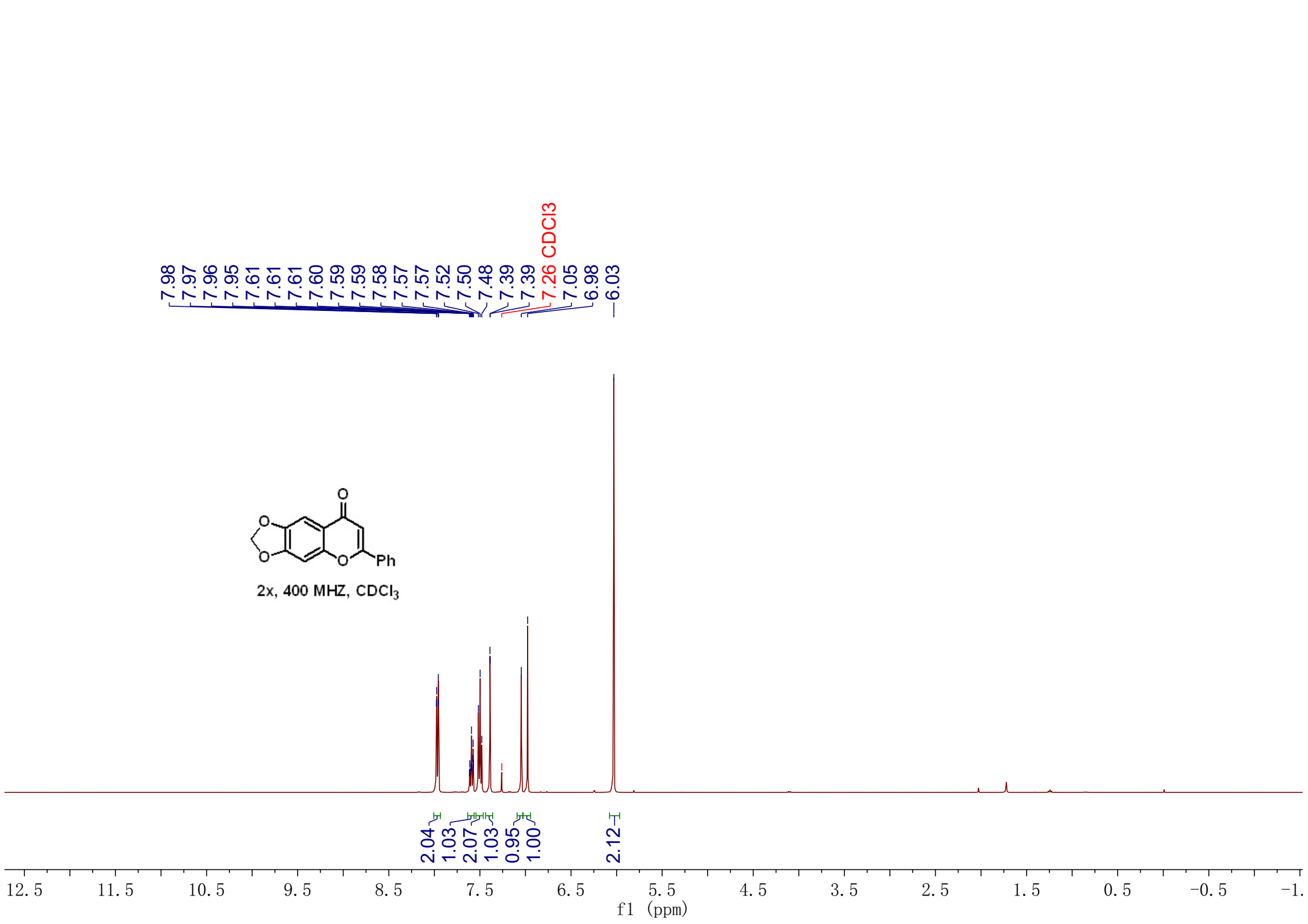


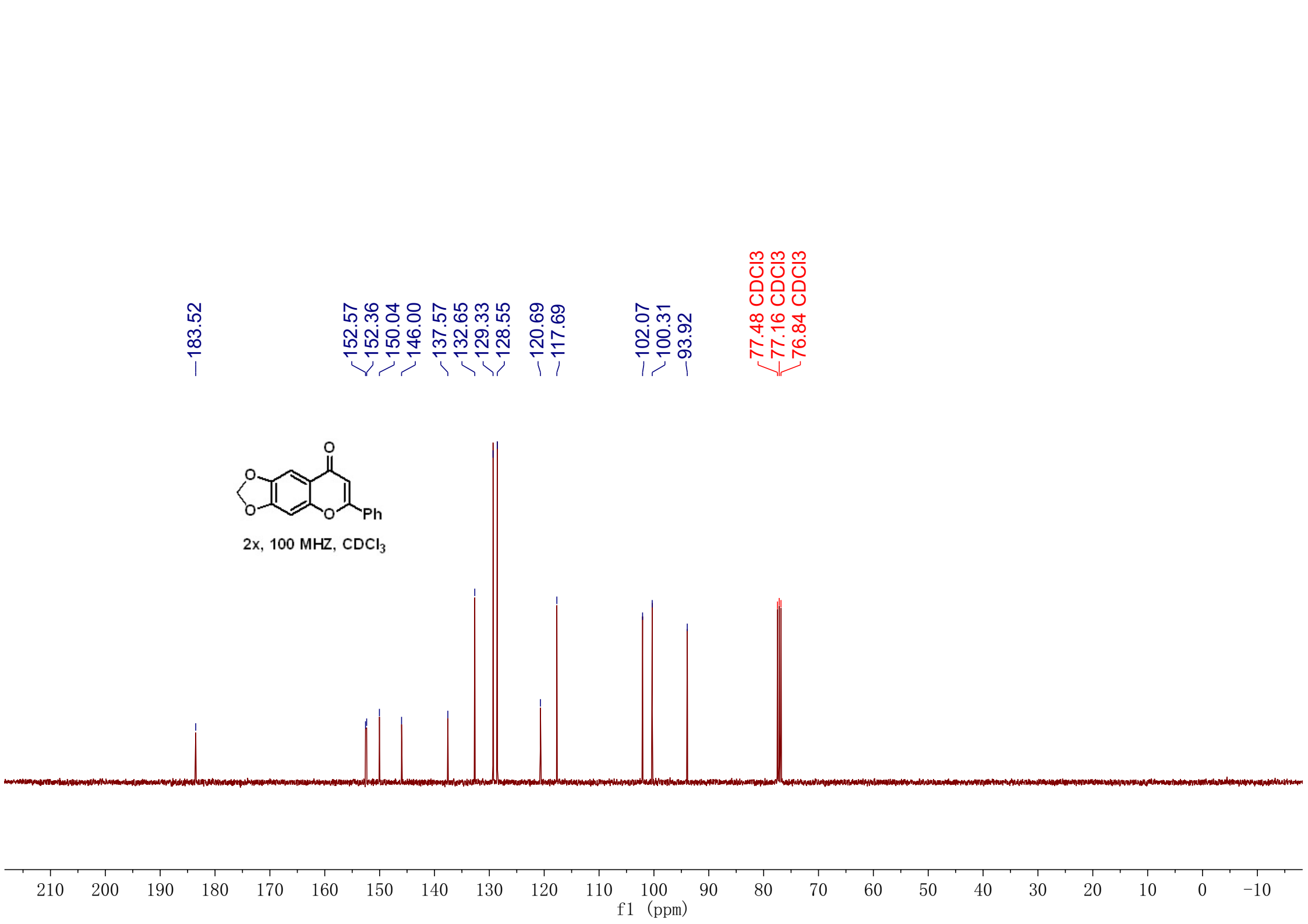
CWS-P131-5.10.1.1r

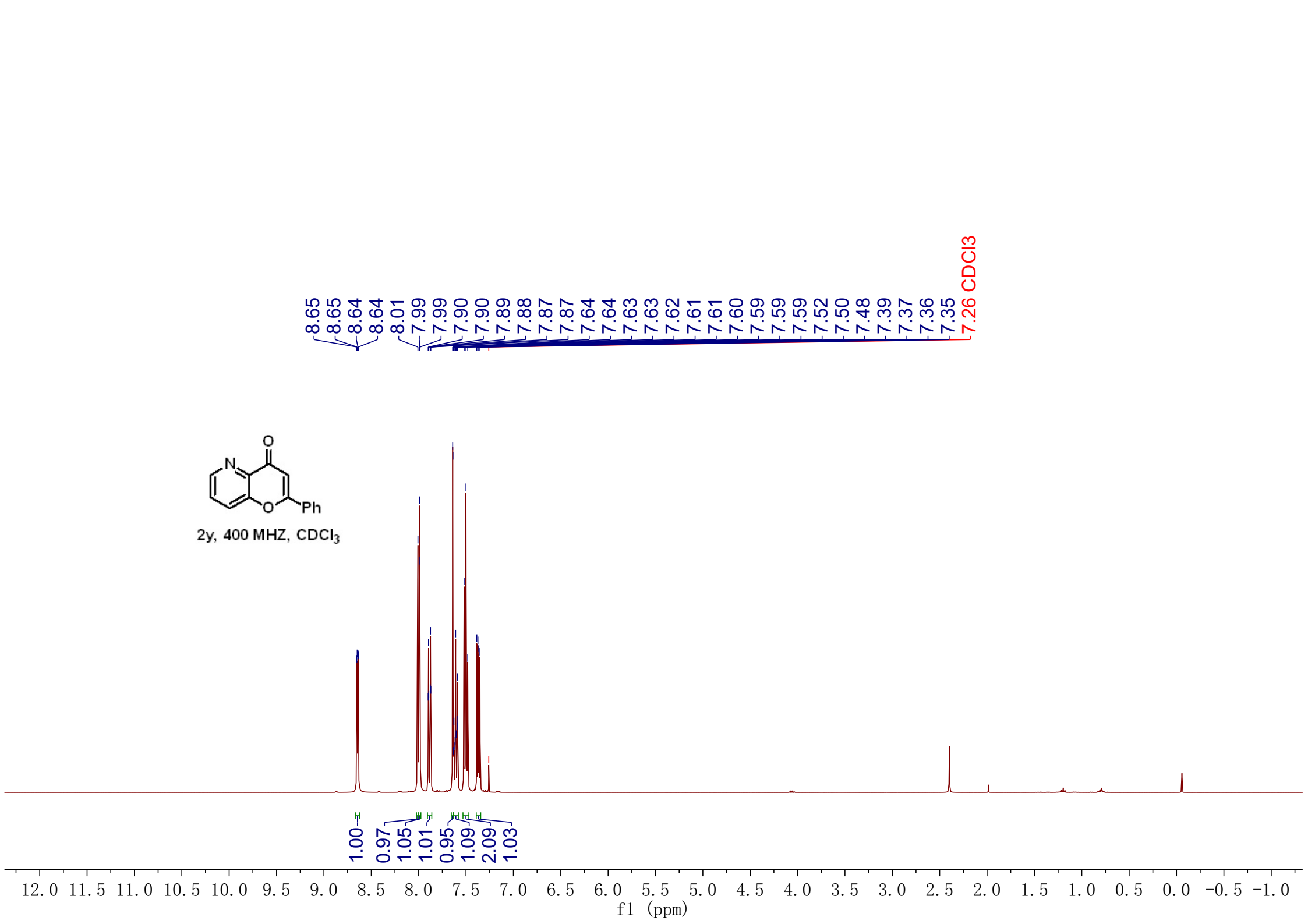


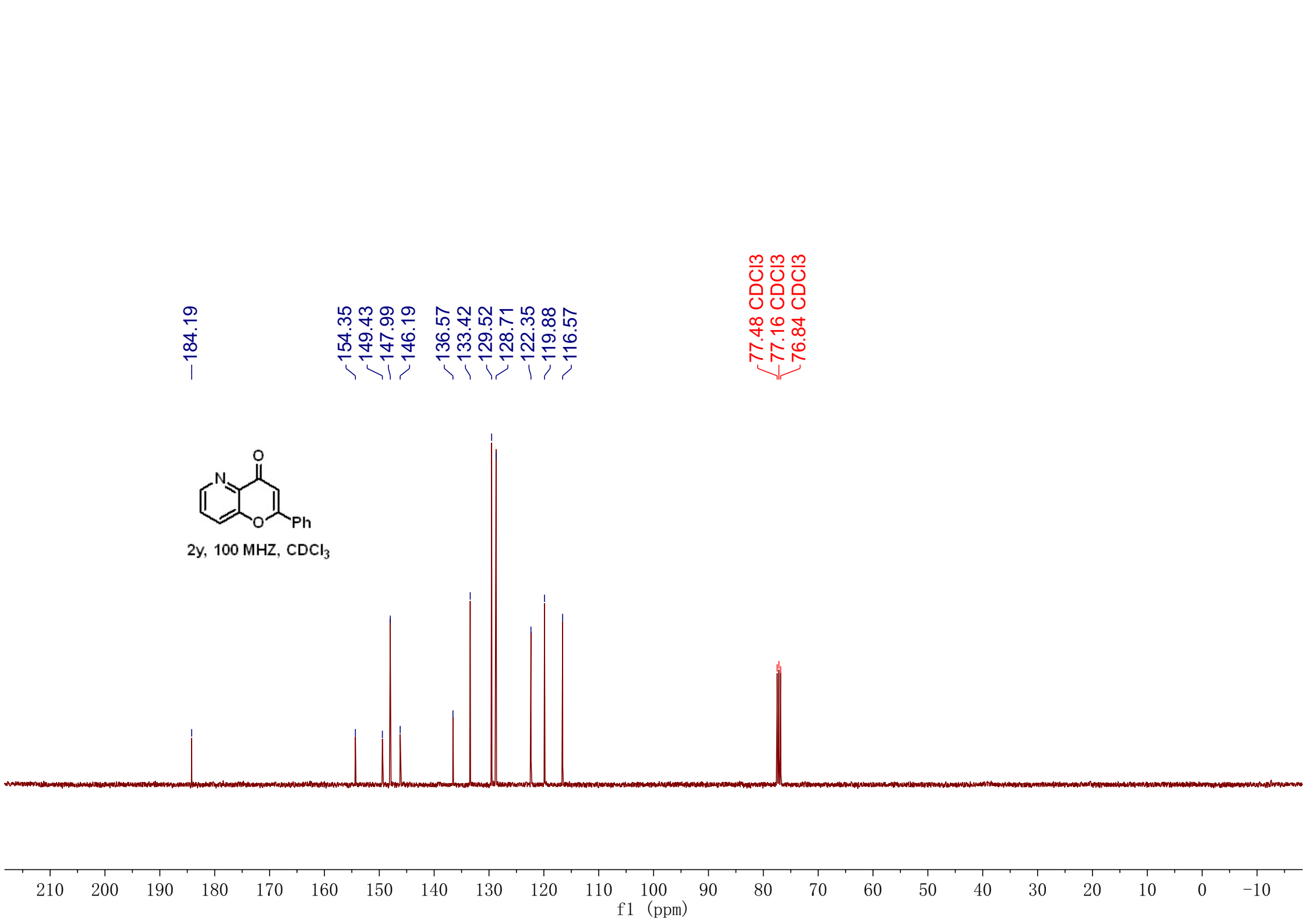
CWS-P131-5.18.1.1r

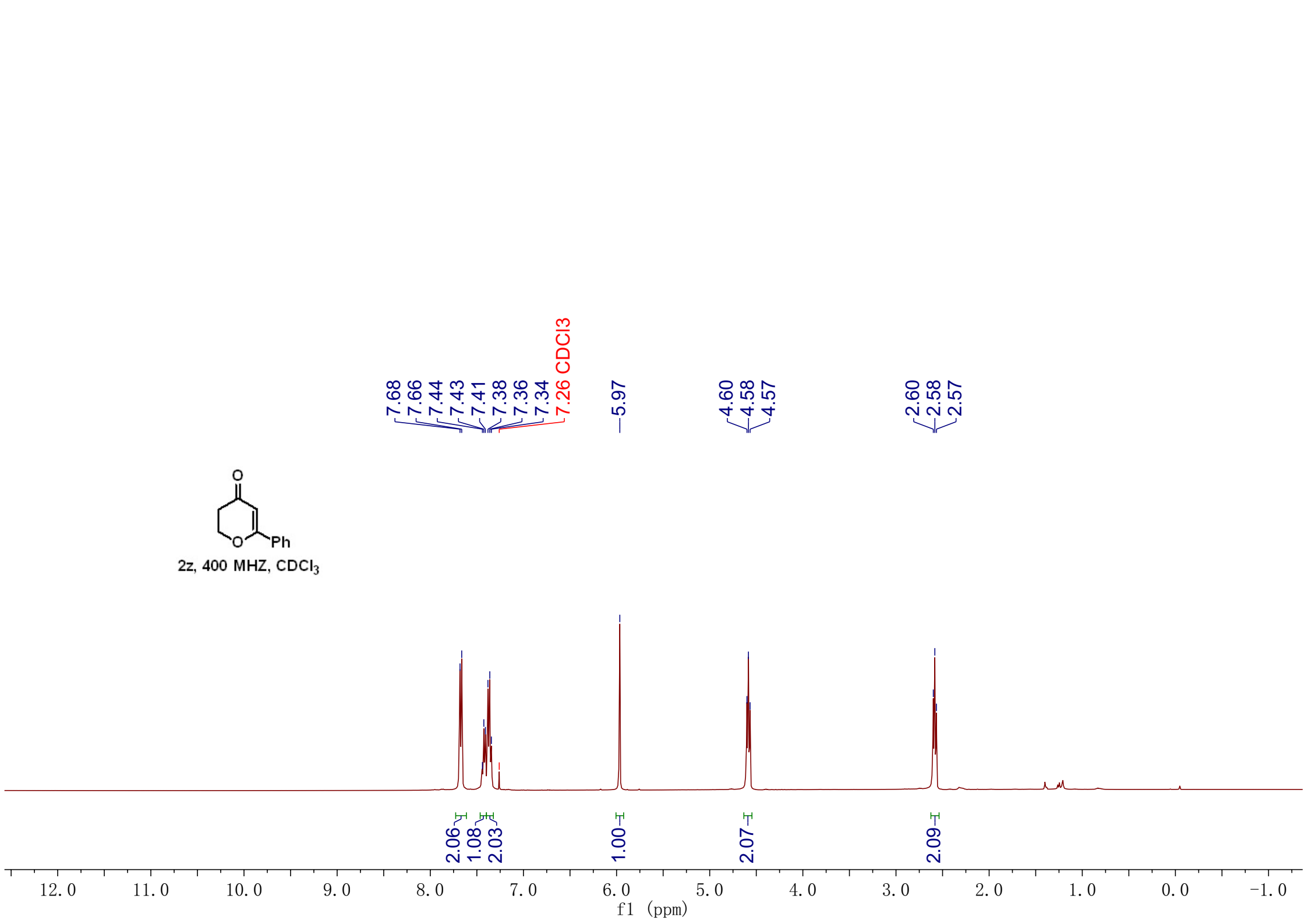


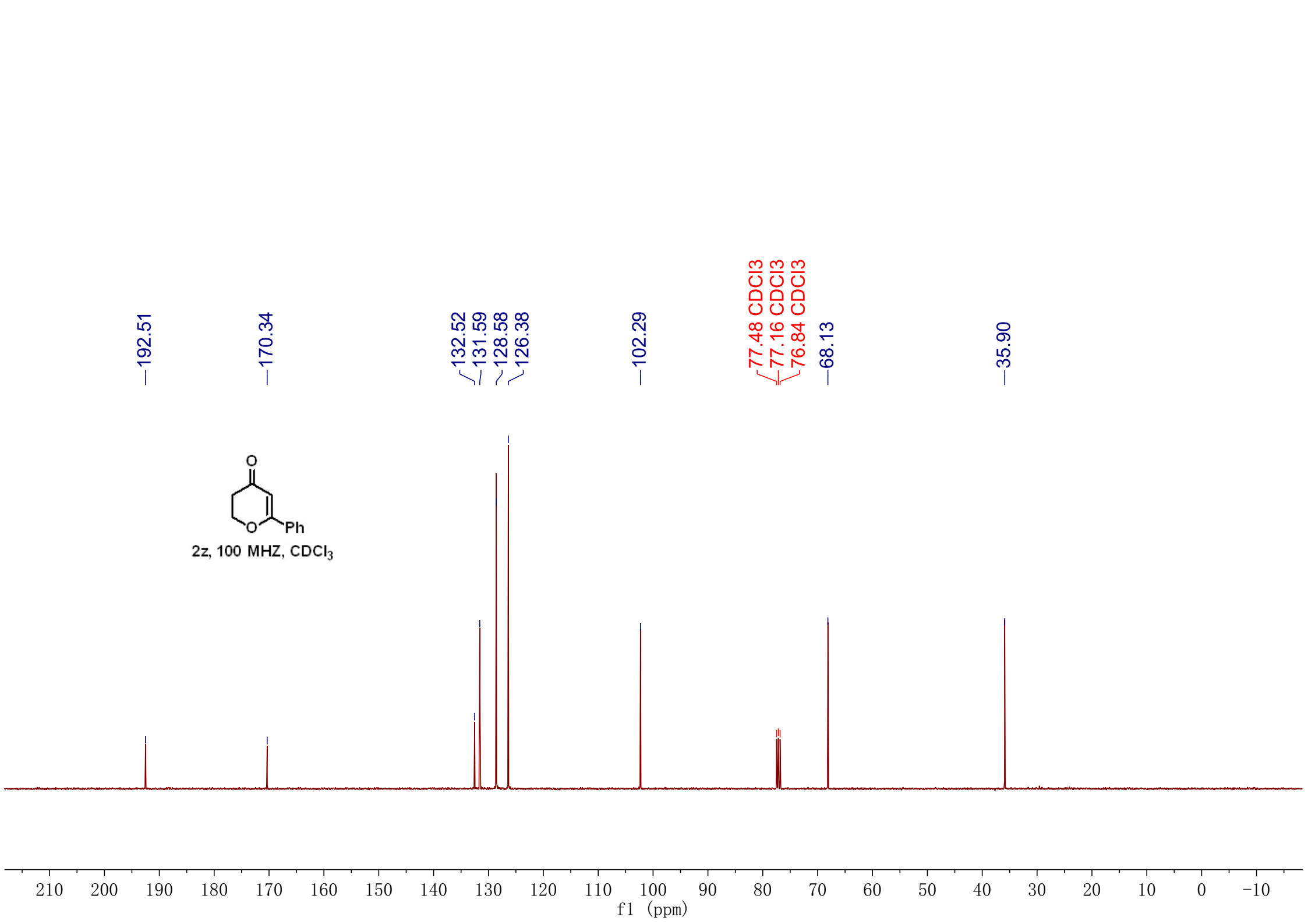

7.98
7.97
7.96
7.95
7.61
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.57
7.52
7.50
7.48
7.39
7.39
7.26 CDCl3
7.05
6.98
6.03
O
O
O
O
Ph
2x, 400 MHZ, CDCl3
2.04
1.03
2.07
1.03
0.95
1.00
2.12
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)

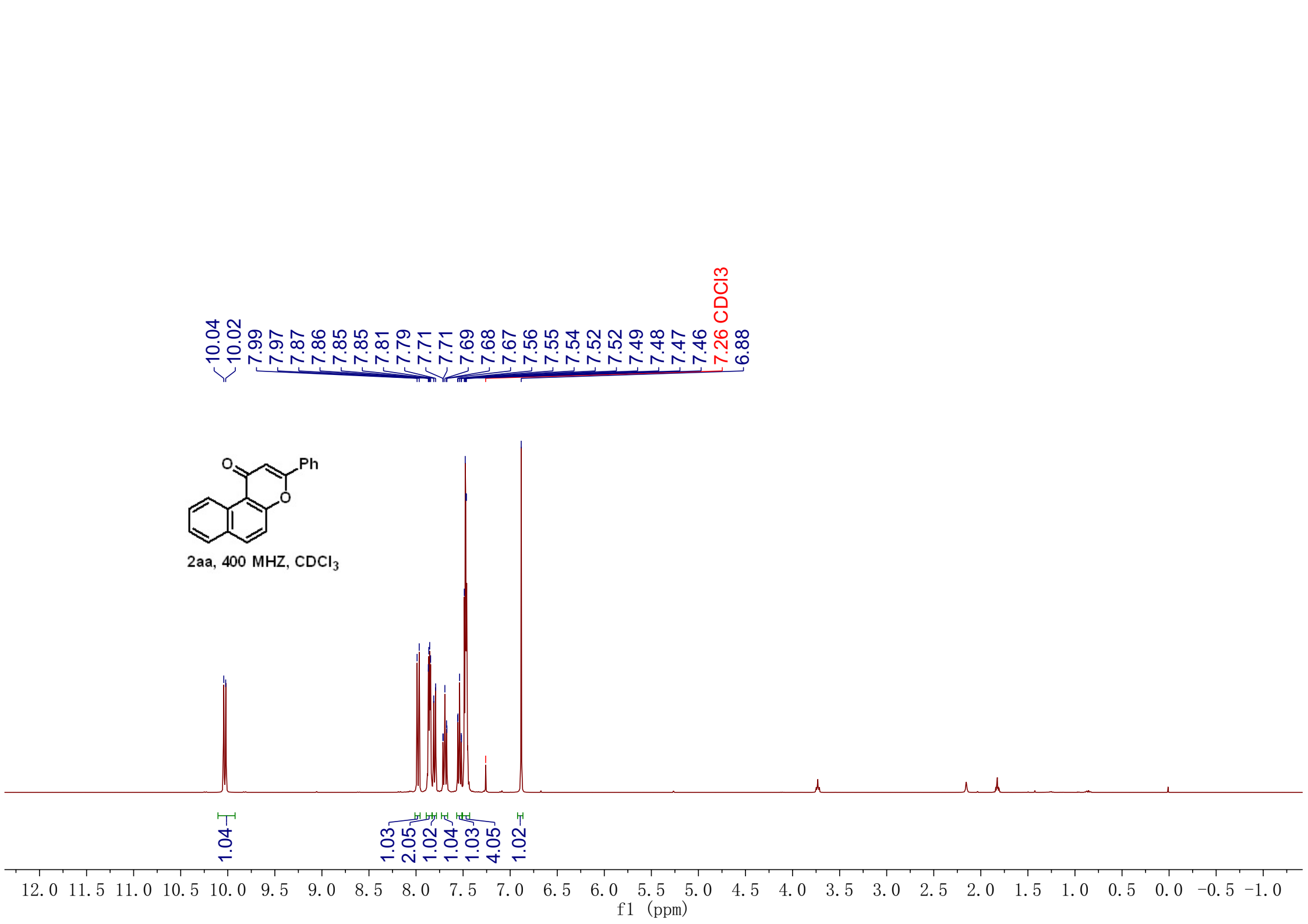

183.52
152.57
152.36
150.04
146.00
137.57
132.65
129.33
128.55
120.69
117.69
102.07
100.31
93.92
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
O
O
Ph
2x, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

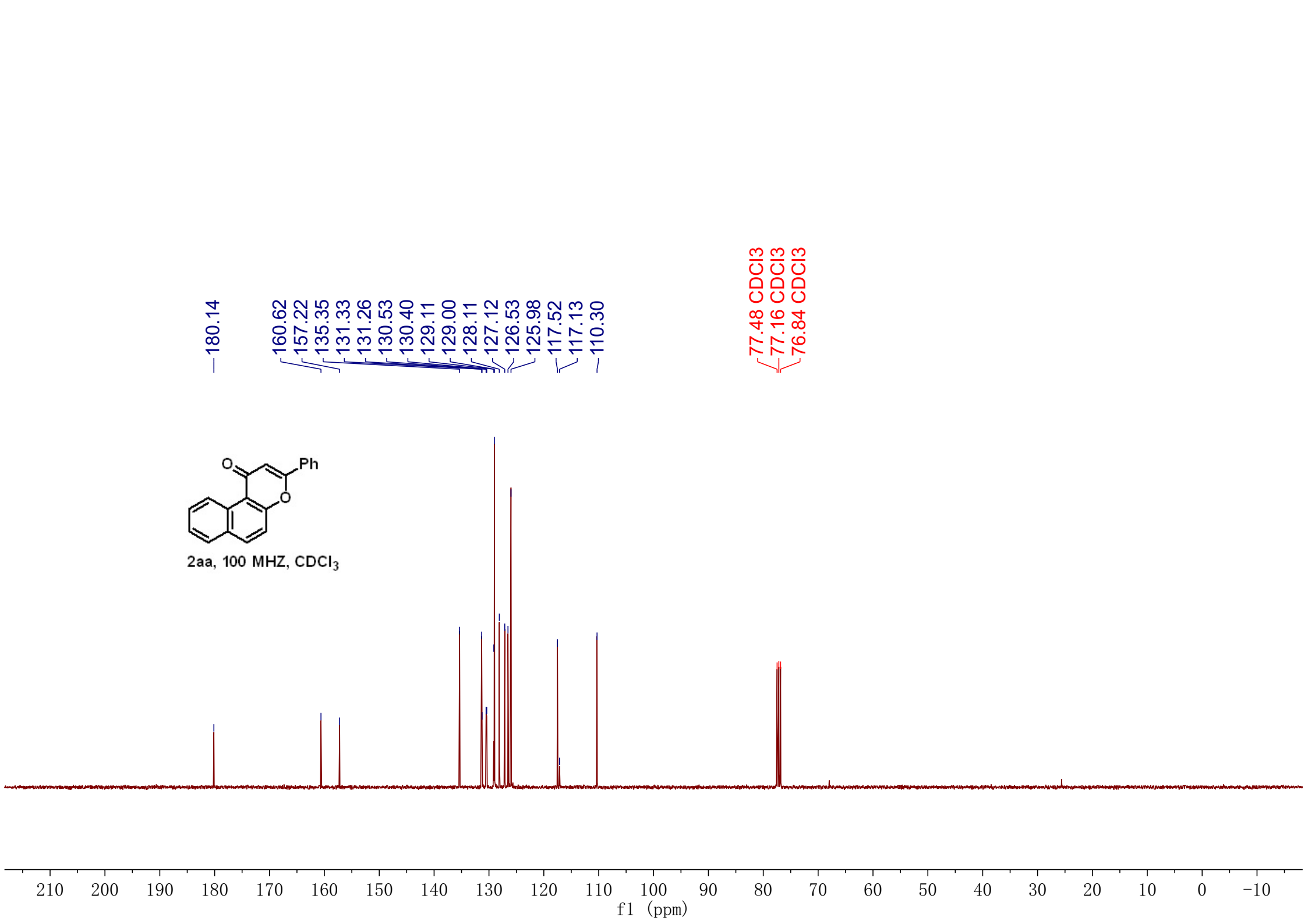

8.65
8.65
8.64
8.64
8.01
7.99
7.99
7.90
7.90
7.89
7.88
7.87
7.87
7.64
7.64
7.63
7.63
7.62
7.61
7.61
7.60
7.59
7.59
7.59
7.52
7.50
7.48
7.39
7.37
7.36
7.35
7.26 CDCl3
O
N
O
Ph
2y, 400 MHZ, CDCl3
1.00
0.97
1.05
1.01
0.95
1.09
2.09
1.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


184.19
154.35
149.43
147.99
146.19
136.57
133.42
129.52
128.71
122.35
119.88
116.57
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
N
O
Ph
2y, 100 MHZ, CDCl3
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)

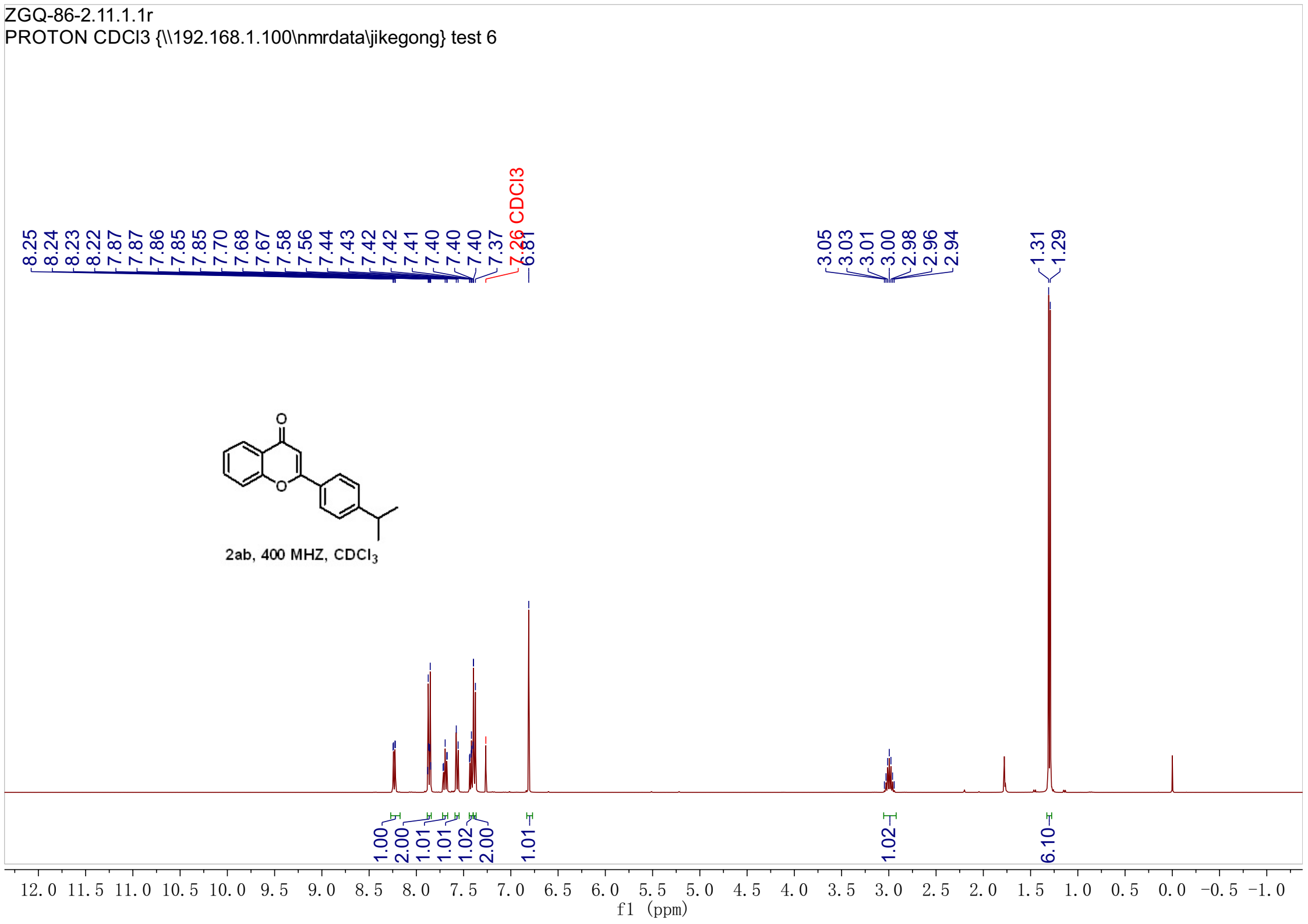

7.68
7.66
7.44
7.43
7.41
7.38
7.36
7.34
7.26 CDCl3
5.97
4.60
4.58
4.57
2.60
2.58
2.57
O
O
Ph
2z, 400 MHZ, CDCl3
2.06
1.08
2.03
1.00
2.07
2.09
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


192.51
170.34
132.52
131.59
128.58
126.38
102.29
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
68.13
35.90
O
O
Ph
2z, 100 MHZ, CDCl3
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)

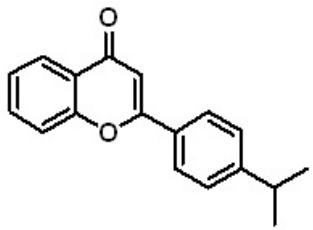

10.04
10.02
7.99
7.97
7.87
7.86
7.85
7.85
7.81
7.79
7.71
7.71
7.69
7.68
7.67
7.56
7.55
7.54
7.52
7.52
7.49
7.48
7.47
7.46
7.26 CDCl3
6.88
O
Ph
O
2aa, 400 MHZ, CDCl3
1.04
1.03
2.05
1.02
1.04
1.03
4.05
1.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


180.14
160.62
157.22
135.35
131.33
131.26
130.53
130.40
129.11
129.00
128.11
127.12
126.53
125.98
117.52
117.13
110.30
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
Ph
O
2aa, 100 MHZ, CDCl3
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)

ZGQ-86-2.11.1.1r
PROTON CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 6



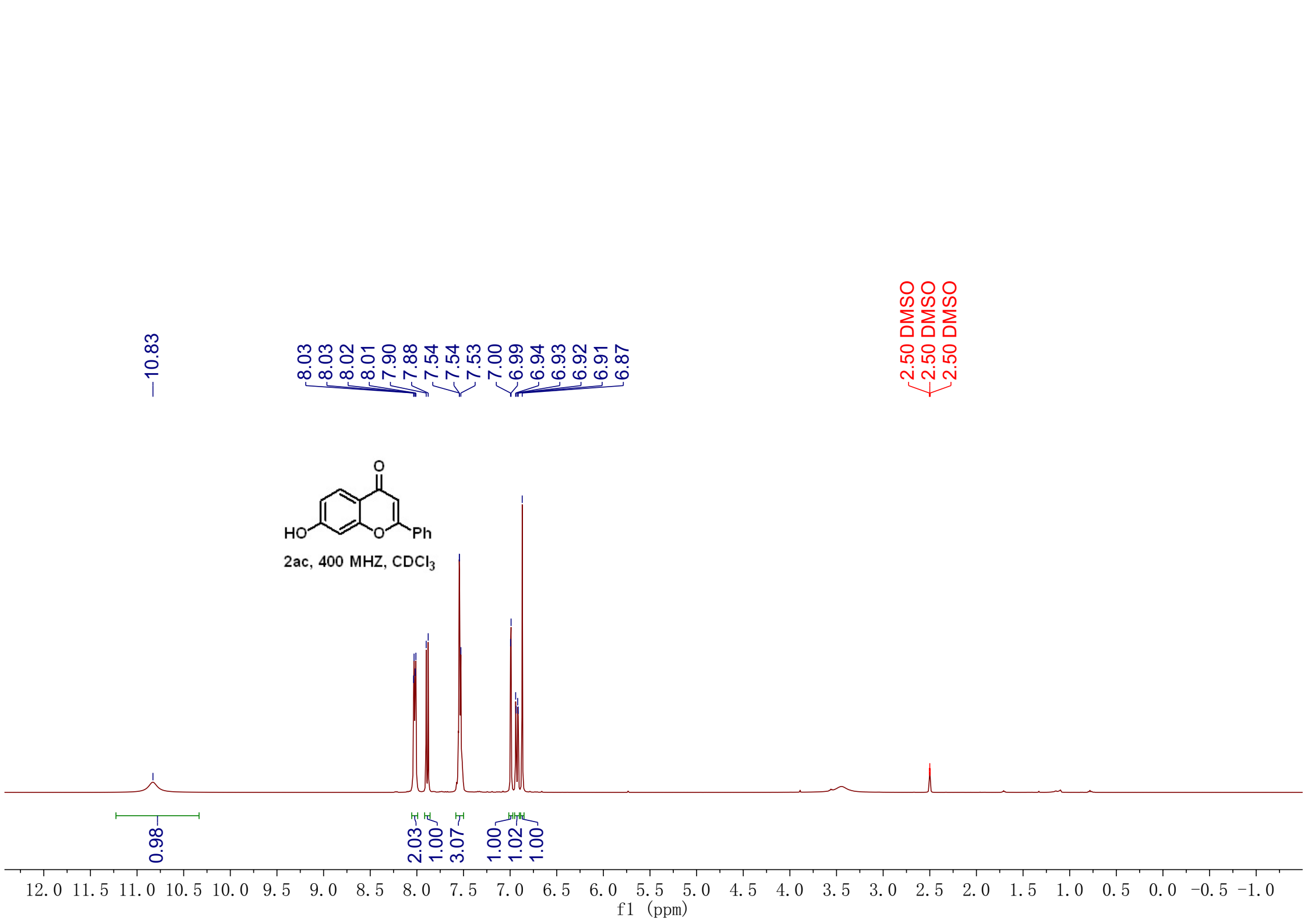
ZGQ-86-2.10.1.1r
C13CPD CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 6

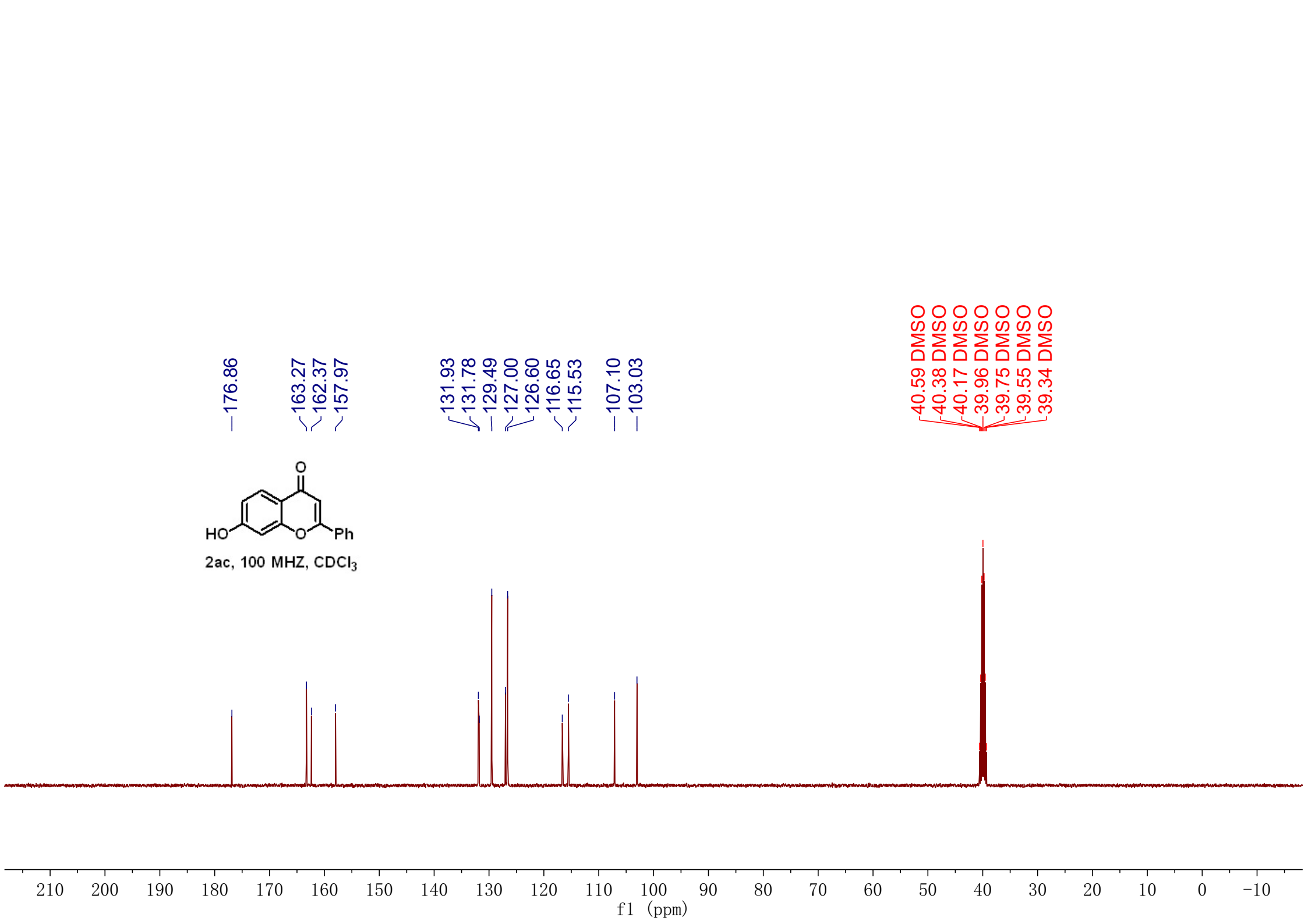


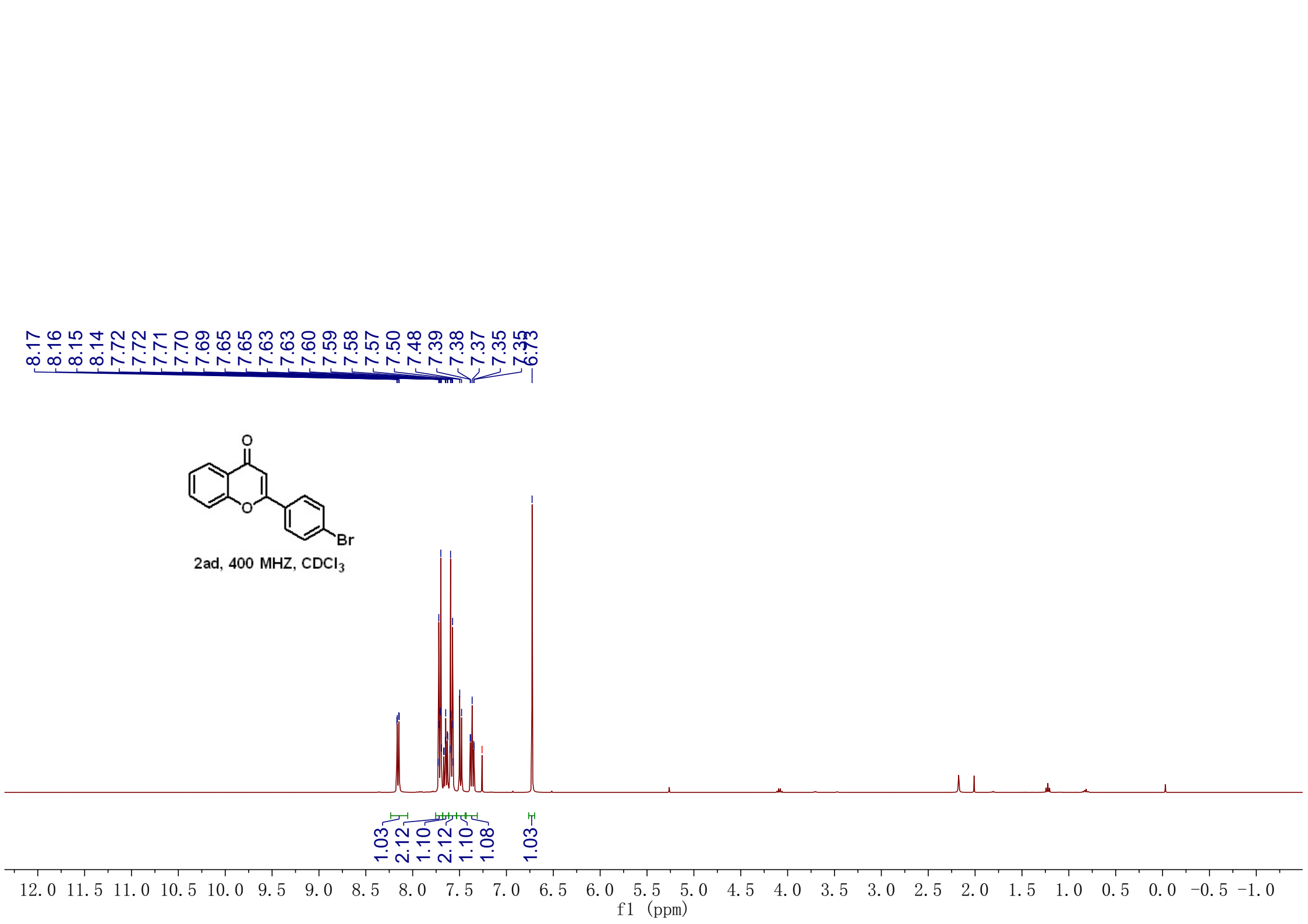
2ab, 100 MHZ, $CDCl_3$

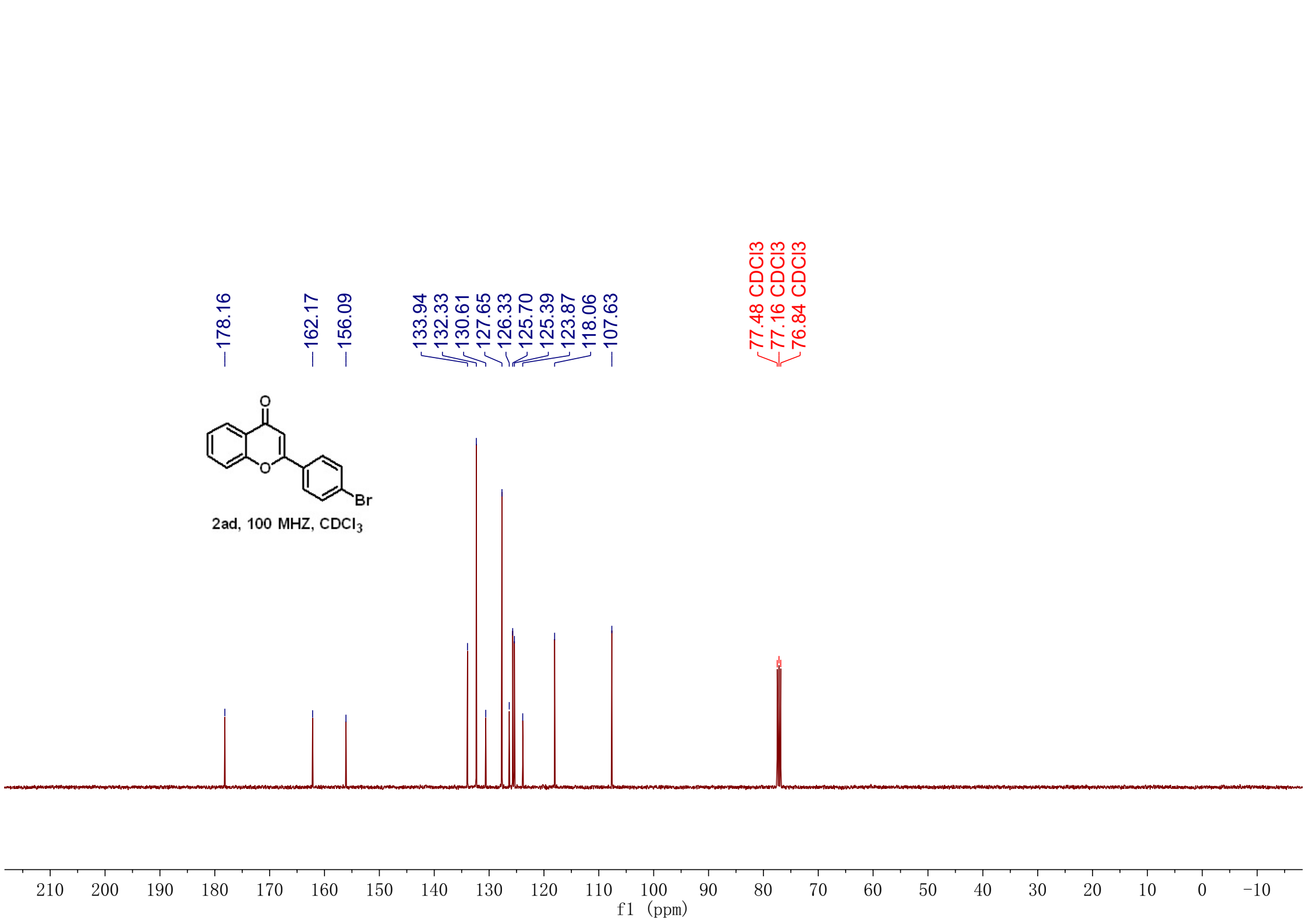
178.64
163.78
156.39
153.21
133.79
129.42
127.32
126.53
125.80
125.27
124.11
118.19
107.17
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
34.28
23.86

f1 (ppm)

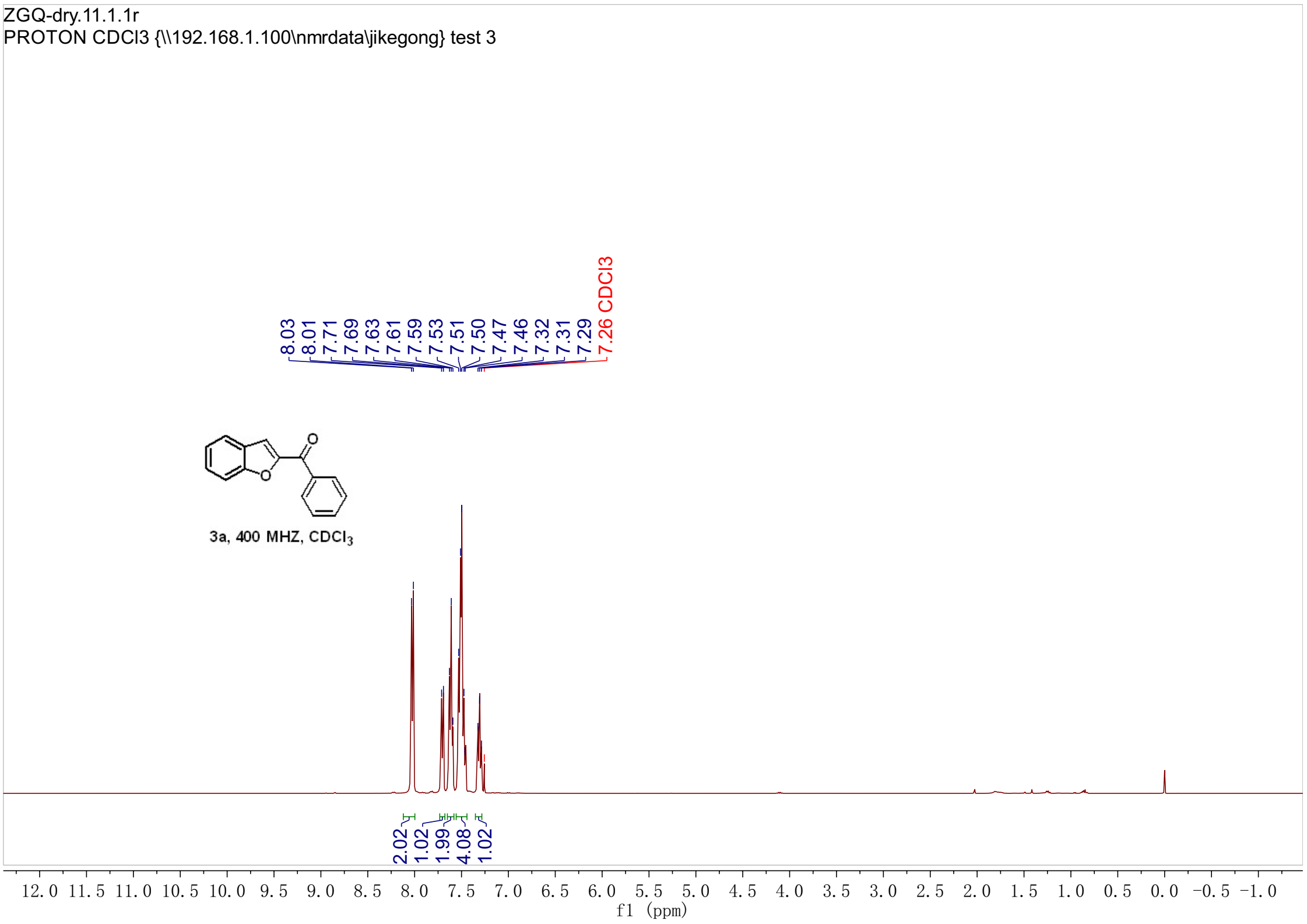

10.83
8.03
8.03
8.02
8.01
7.90
7.88
7.54
7.54
7.53
7.00
6.99
6.94
6.93
6.92
6.91
6.87
2.50 DMSO
2.50 DMSO
2.50 DMSO
HO
O
O
Ph
2ac, 400 MHZ, CDCl3
0.98
2.03
1.00
3.07
1.00
1.02
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


176.86
163.27
162.37
157.97
131.93
131.78
129.49
127.00
126.60
116.65
115.53
107.10
103.03
40.59 DMSO
40.38 DMSO
40.17 DMSO
39.96 DMSO
39.75 DMSO
39.55 DMSO
39.34 DMSO
HO
O
O
Ph
2ac, 100 MHZ, CDCl3
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)

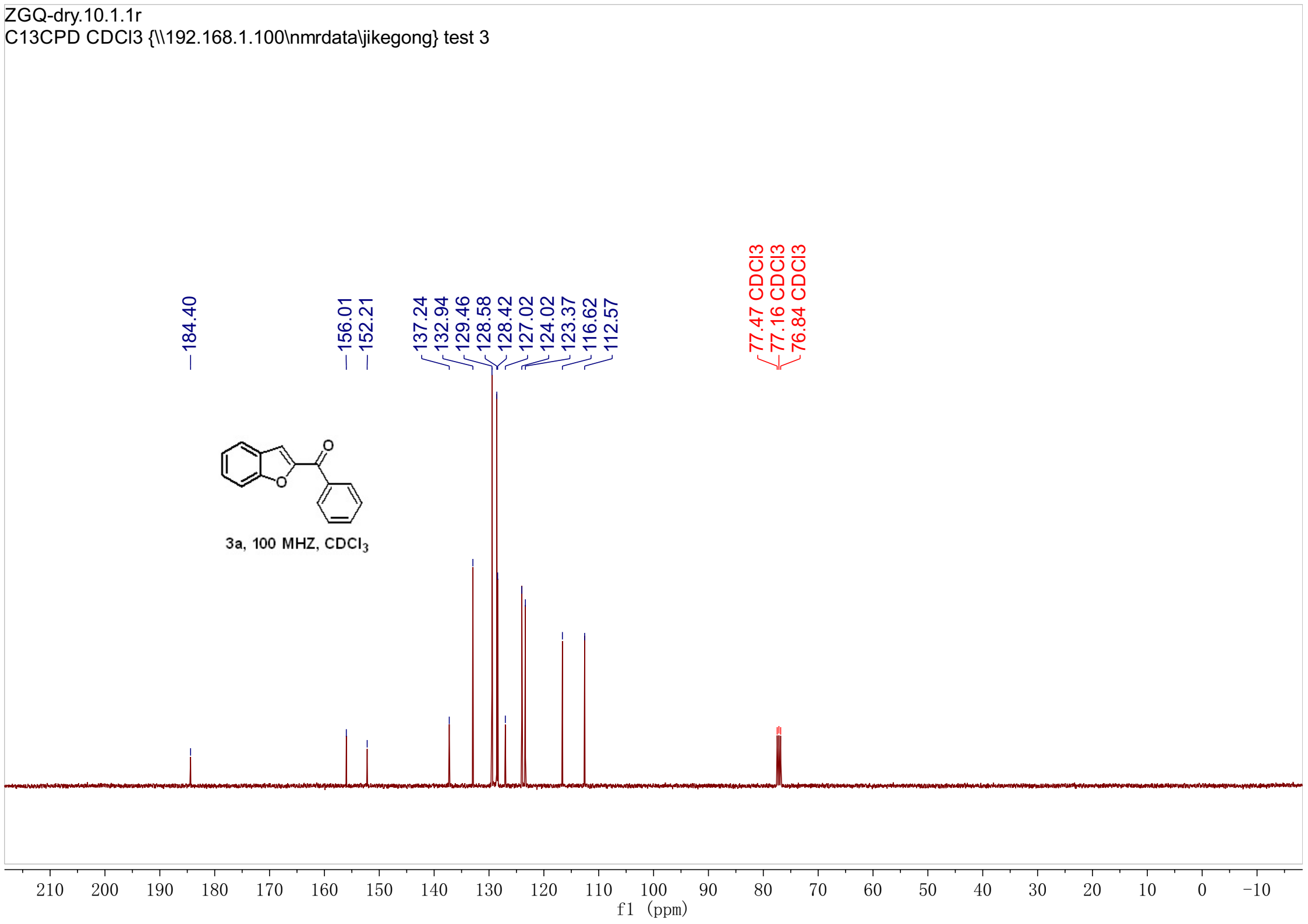

8.17
8.16
8.15
8.14
7.72
7.72
7.71
7.70
7.69
7.65
7.65
7.63
7.63
7.60
7.59
7.58
7.57
7.50
7.48
7.39
7.38
7.37
7.35
7.35
6.73
O
O
Br
2ad, 400 MHZ, CDCl3
1.03
2.12
1.10
2.12
1.10
1.08
1.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

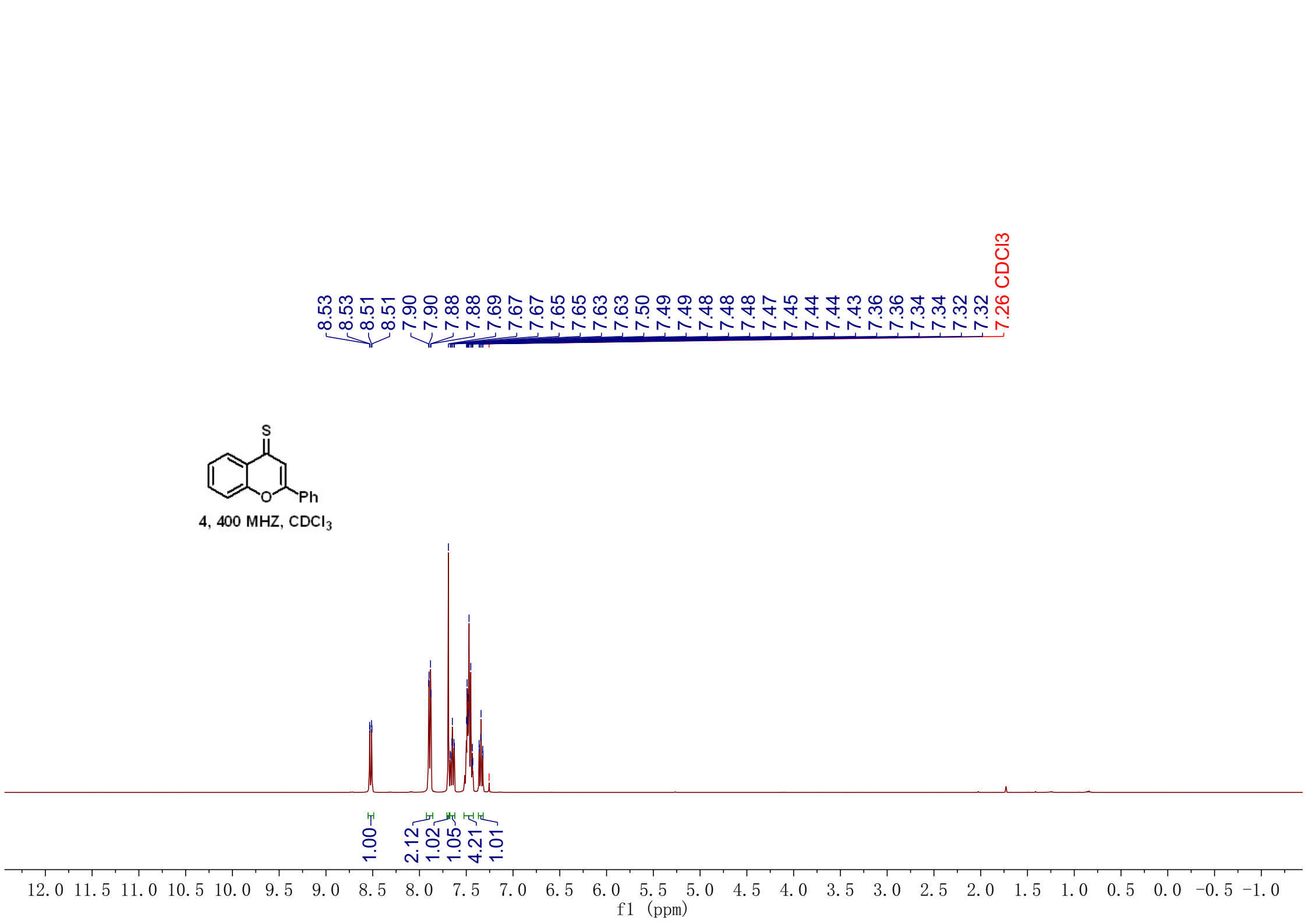

178.16
162.17
156.09
133.94
132.33
130.61
127.65
126.33
125.70
125.39
123.87
118.06
107.63
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
Br
2ad, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

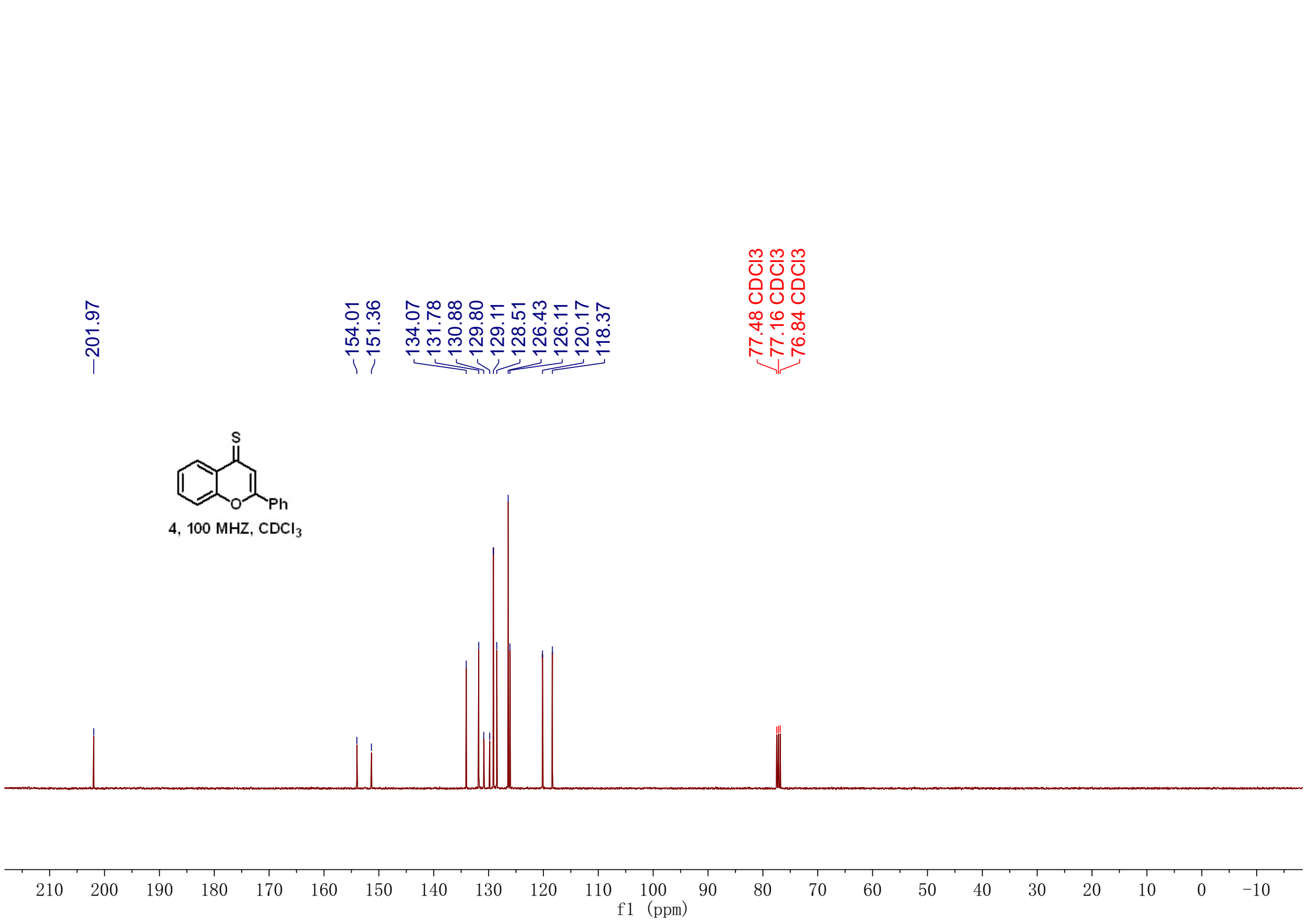
ZGQ-dry.11.1.1r
PROTON CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 3

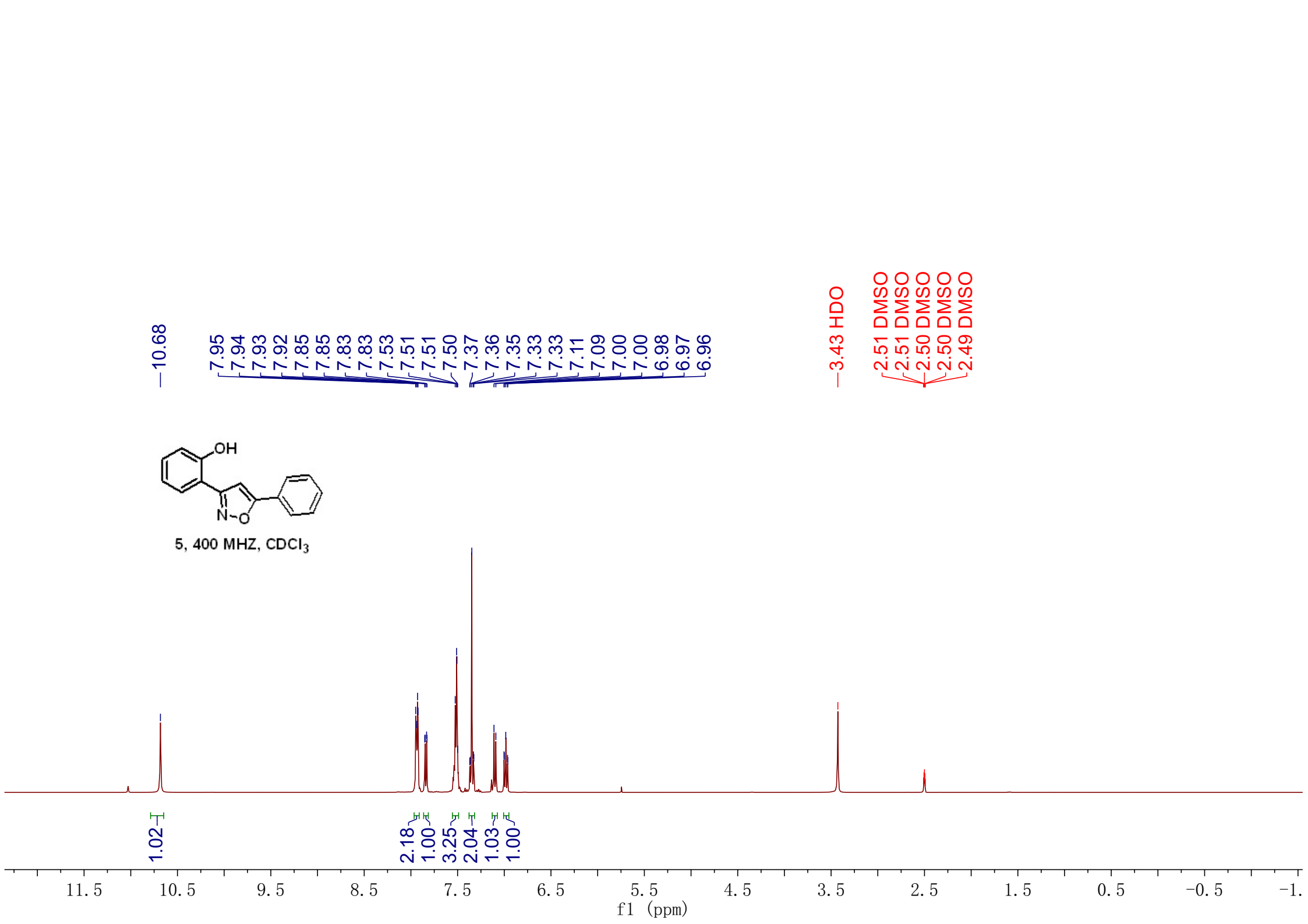


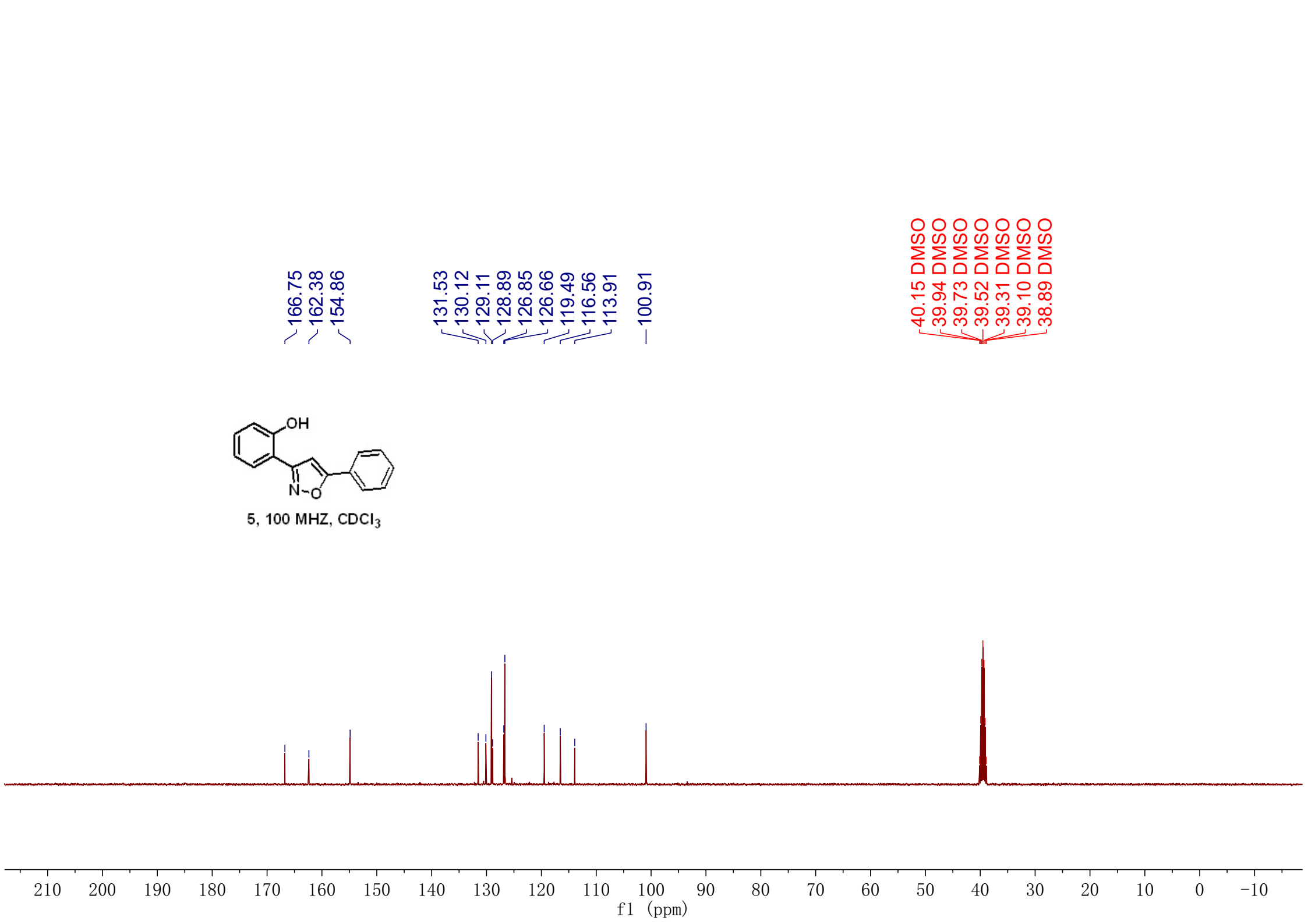
ZGQ-dry.10.1.1r
C13CPD CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 3

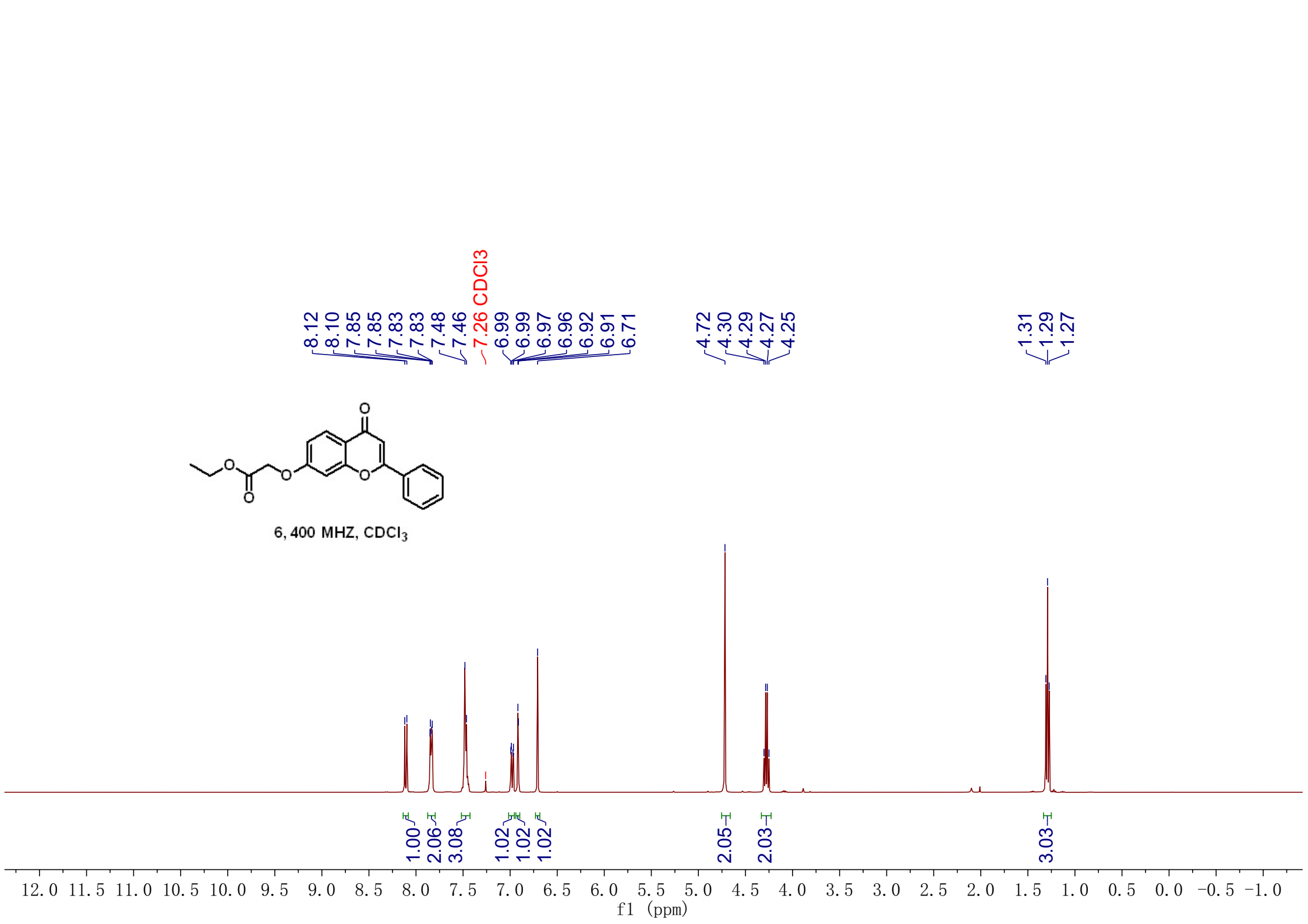


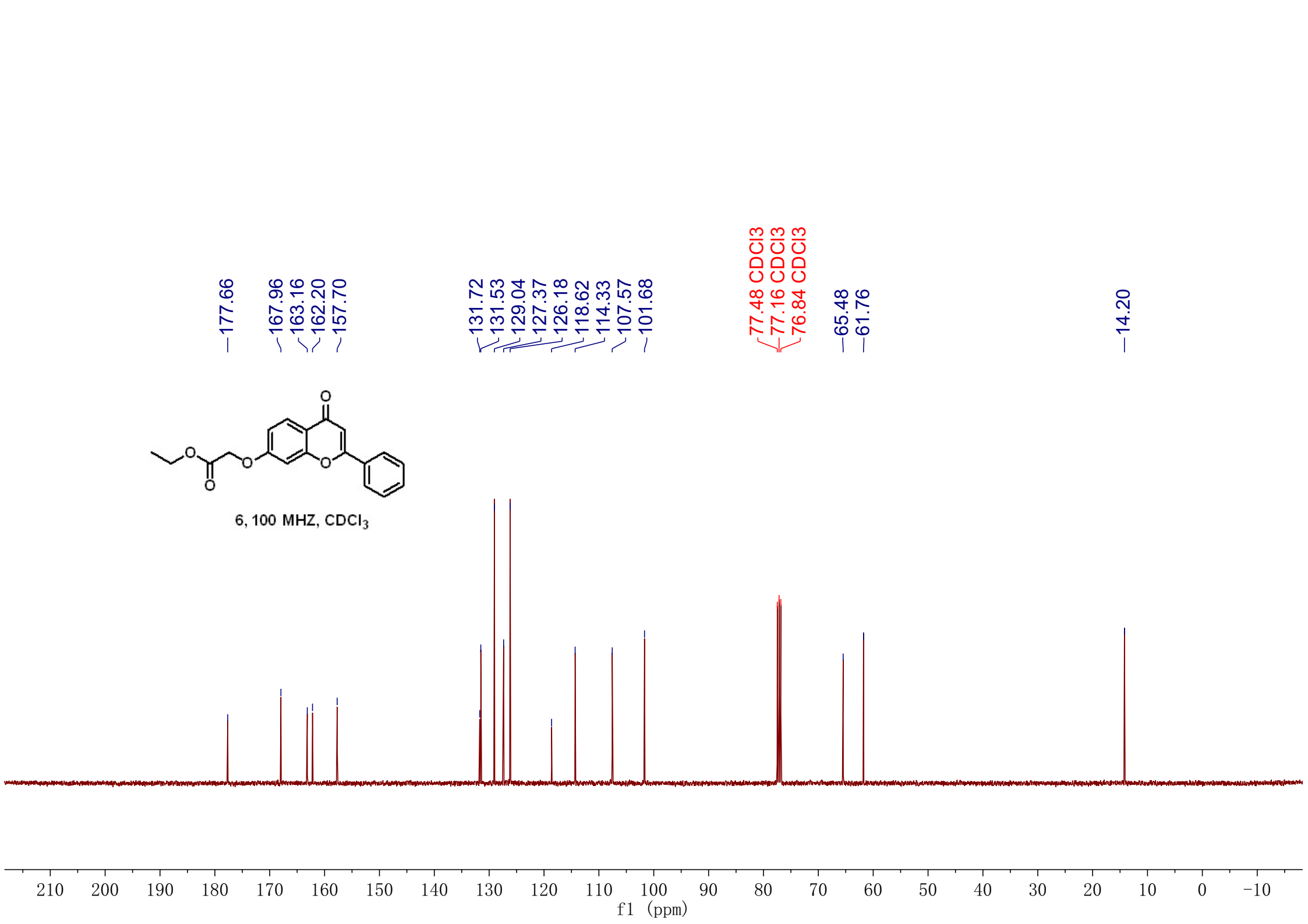

8.53
8.53
8.51
8.51
7.90
7.90
7.88
7.88
7.69
7.67
7.67
7.65
7.65
7.63
7.63
7.50
7.49
7.49
7.48
7.48
7.48
7.47
7.45
7.44
7.44
7.43
7.36
7.36
7.34
7.34
7.32
7.32
7.26 CDCl3
S
O
Ph
4, 400 MHZ, CDCl3
1.00
2.12
1.02
1.05
4.21
1.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

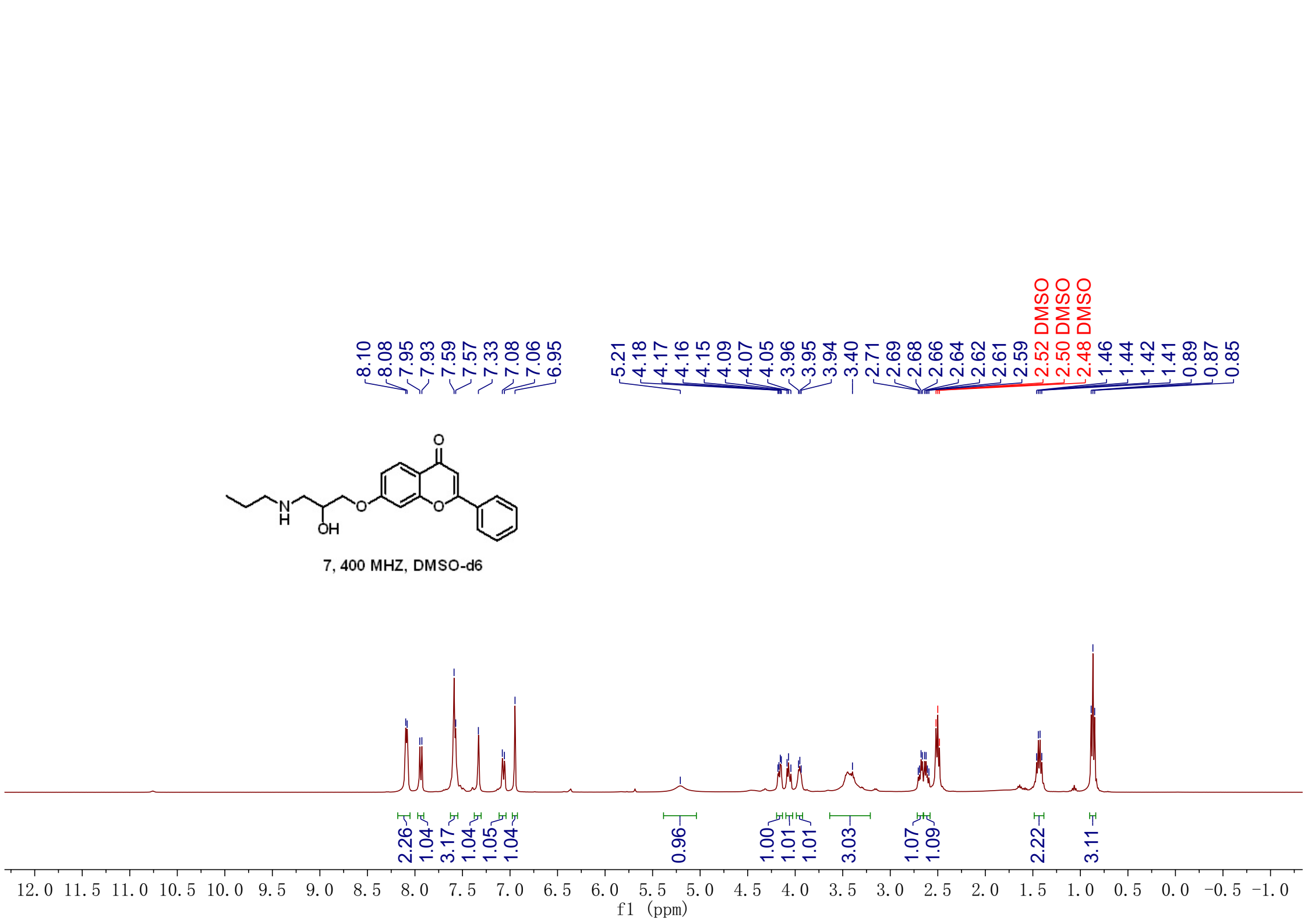

201.97
154.01
151.36
134.07
131.78
130.88
129.80
129.11
128.51
126.43
126.11
120.17
118.37
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
S
O
Ph
4, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

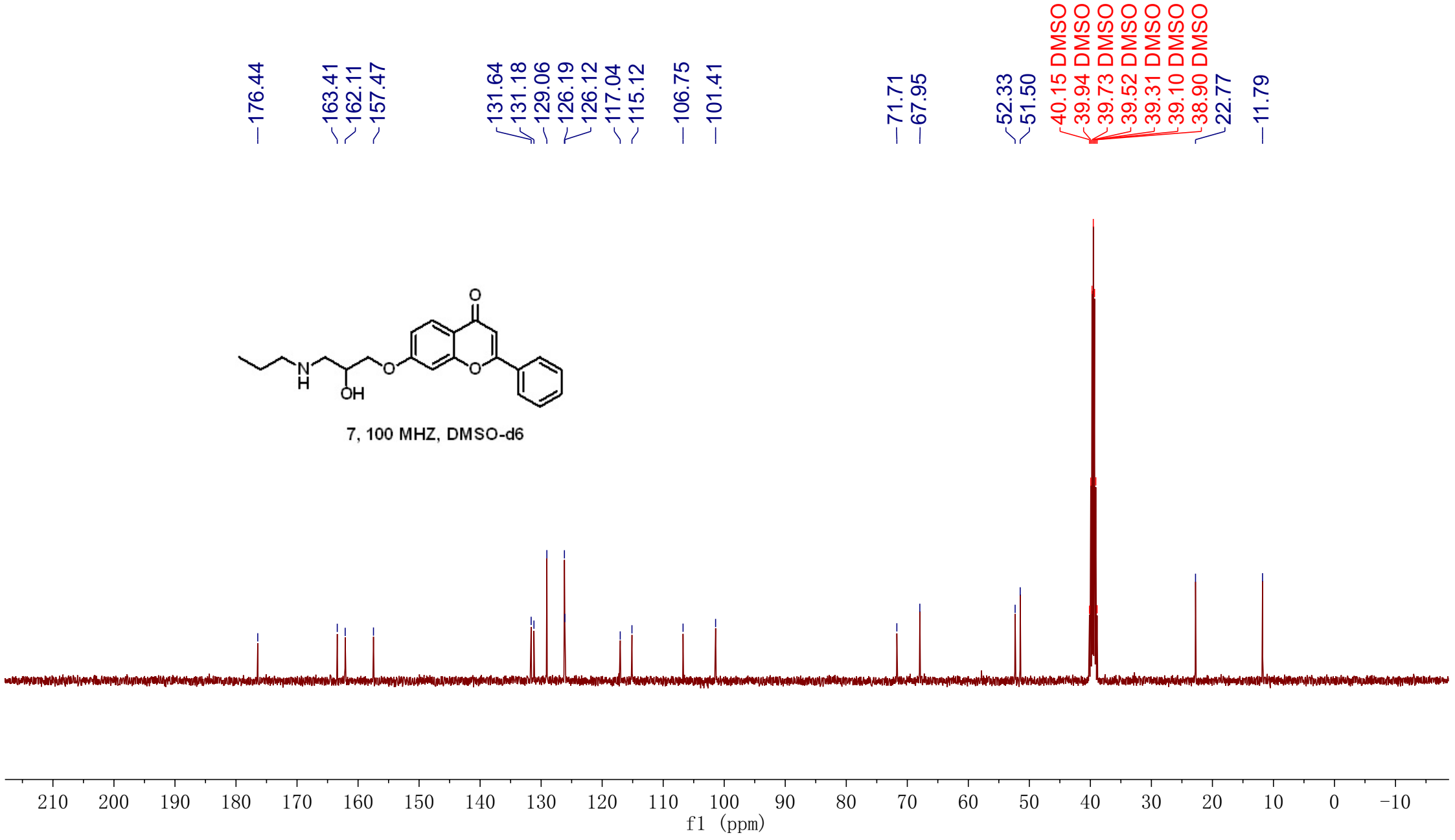

10.68
7.95
7.94
7.93
7.92
7.85
7.85
7.83
7.83
7.53
7.51
7.51
7.50
7.37
7.36
7.35
7.33
7.33
7.11
7.09
7.00
7.00
6.98
6.97
6.96
3.43 HDO
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
OH
N
O
5, 400 MHZ, CDCl3
1.02
2.18
1.00
3.25
2.04
1.03
1.00
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.
f1 (ppm)

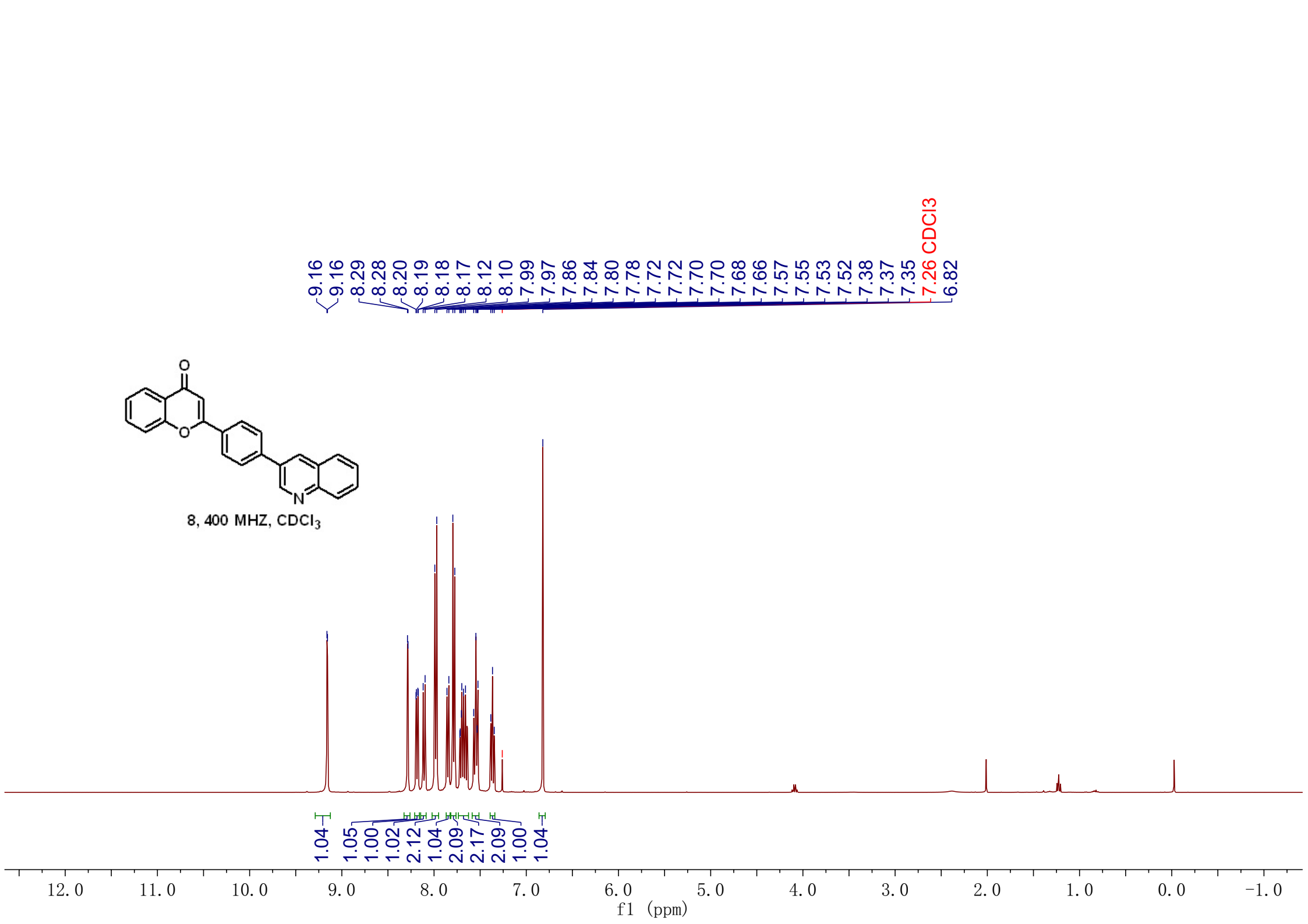

166.75
162.38
154.86
131.53
130.12
129.11
128.89
126.85
126.66
119.49
116.56
113.91
100.91
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
OH
N
O
5, 100 MHZ, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

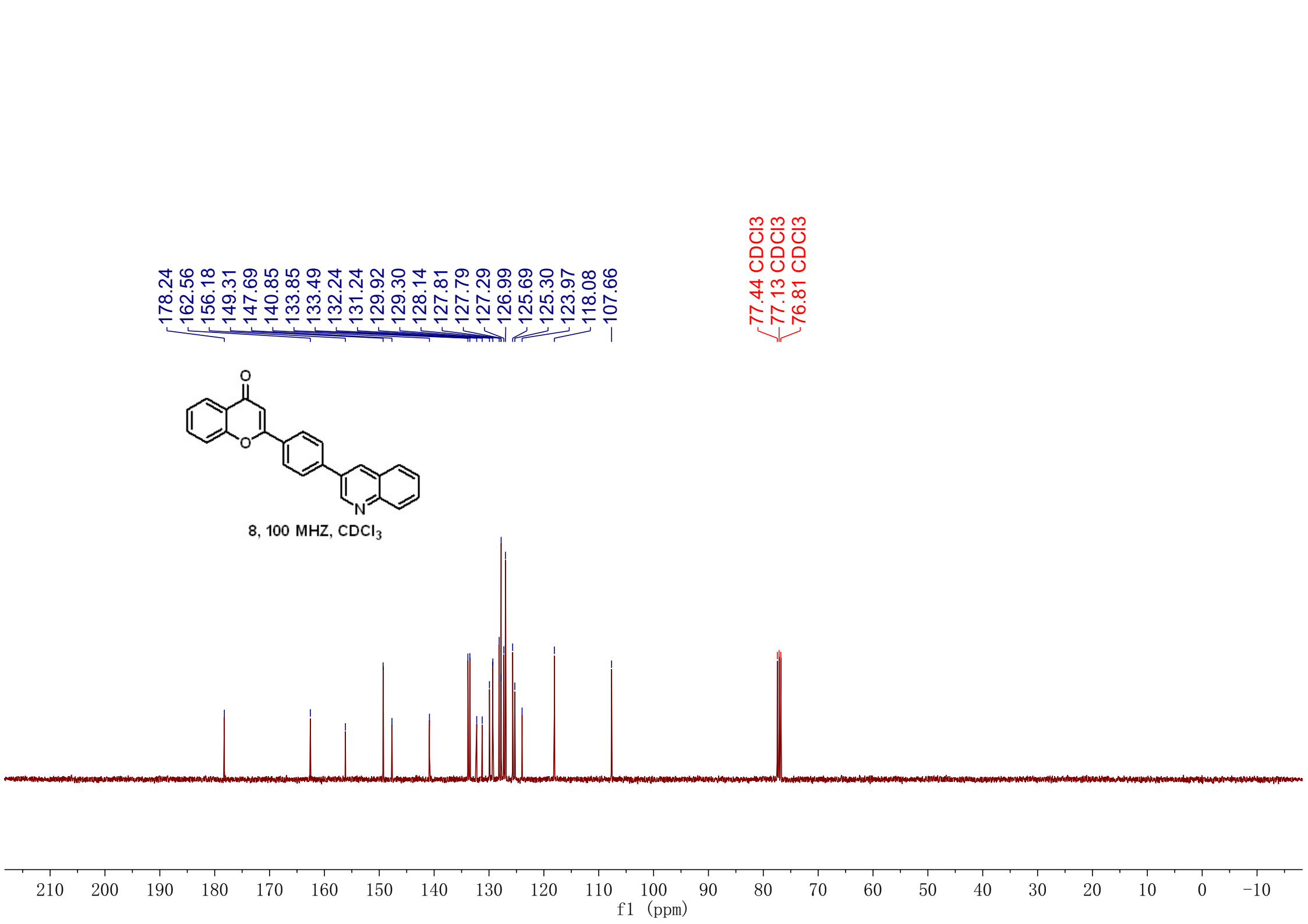

8.12
8.10
7.85
7.85
7.83
7.83
7.48
7.46
7.26 CDCl3
6.99
6.99
6.97
6.96
6.92
6.91
6.71
4.72
4.30
4.29
4.27
4.25
1.31
1.29
1.27
6, 400 MHZ, CDCl3
1.00
2.06
3.08
1.02
1.02
1.02
2.05
2.03
3.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


177.66
167.96
163.16
162.20
157.70
131.72
131.53
129.04
127.37
126.18
118.62
114.33
107.57
101.68
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
65.48
61.76
14.20
6, 100 MHZ, CDCl3
f1 (ppm)

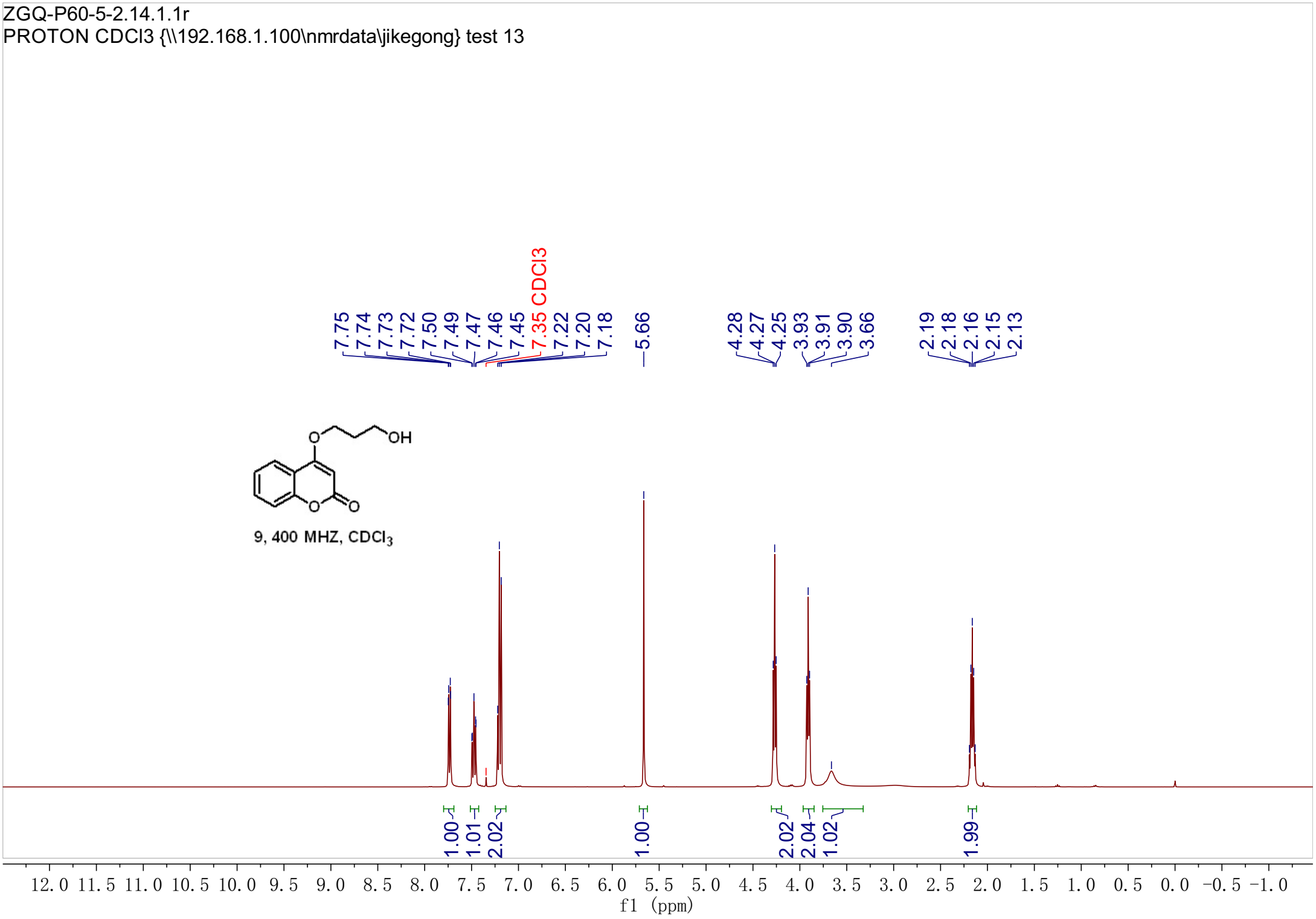

8.10
8.08
7.95
7.93
7.59
7.57
7.33
7.08
7.06
6.95
5.21
4.18
4.17
4.16
4.15
4.09
4.07
4.05
3.96
3.95
3.94
3.40
2.71
2.69
2.68
2.66
2.64
2.62
2.61
2.59
2.52 DMSO
2.50 DMSO
2.48 DMSO
1.46
1.44
1.42
1.41
0.89
0.87
0.85
O
N
H
OH
O
O
7, 400 MHZ, DMSO-d6
2.26
1.04
3.17
1.04
1.05
1.04
0.96
1.00
1.01
1.01
3.03
1.07
1.09
2.22
3.11
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 -1.0
f1 (ppm)


176.44
163.41
162.11
157.47
131.64
131.18
129.06
126.19
126.12
117.04
115.12
106.75
101.41
71.71
67.95
52.33
51.50
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.90 DMSO
22.77
11.79
O
N
H
OH
O
O
7, 100 MHZ, DMSO-d6
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)

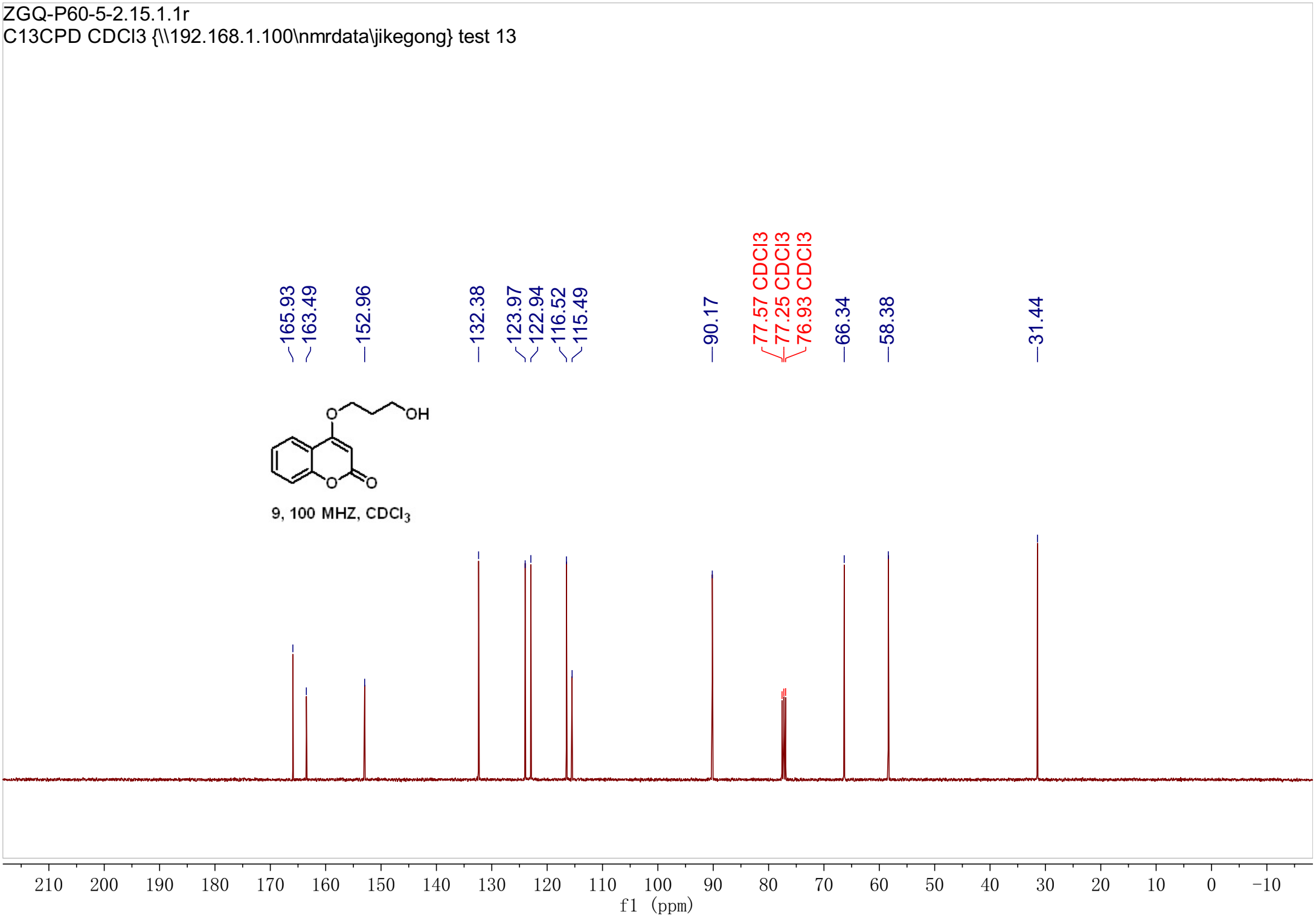

9.16
9.16
8.29
8.28
8.20
8.19
8.18
8.17
8.12
8.10
7.99
7.97
7.86
7.84
7.80
7.78
7.72
7.72
7.70
7.70
7.68
7.66
7.57
7.55
7.53
7.52
7.38
7.37
7.35
7.26 CDCl3
6.82
O
O
N
8, 400 MHZ, CDCl3
1.04
1.05
1.00
1.02
2.12
1.04
2.09
2.17
2.09
1.00
1.04
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


178.24
162.56
156.18
149.31
147.69
140.85
133.85
133.49
132.24
131.24
129.92
129.30
128.14
127.81
127.79
127.29
126.99
125.69
125.30
123.97
118.08
107.66
77.44 CDCl3
77.13 CDCl3
76.81 CDCl3
O
O
N
8, 100 MHZ, CDCl3
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)

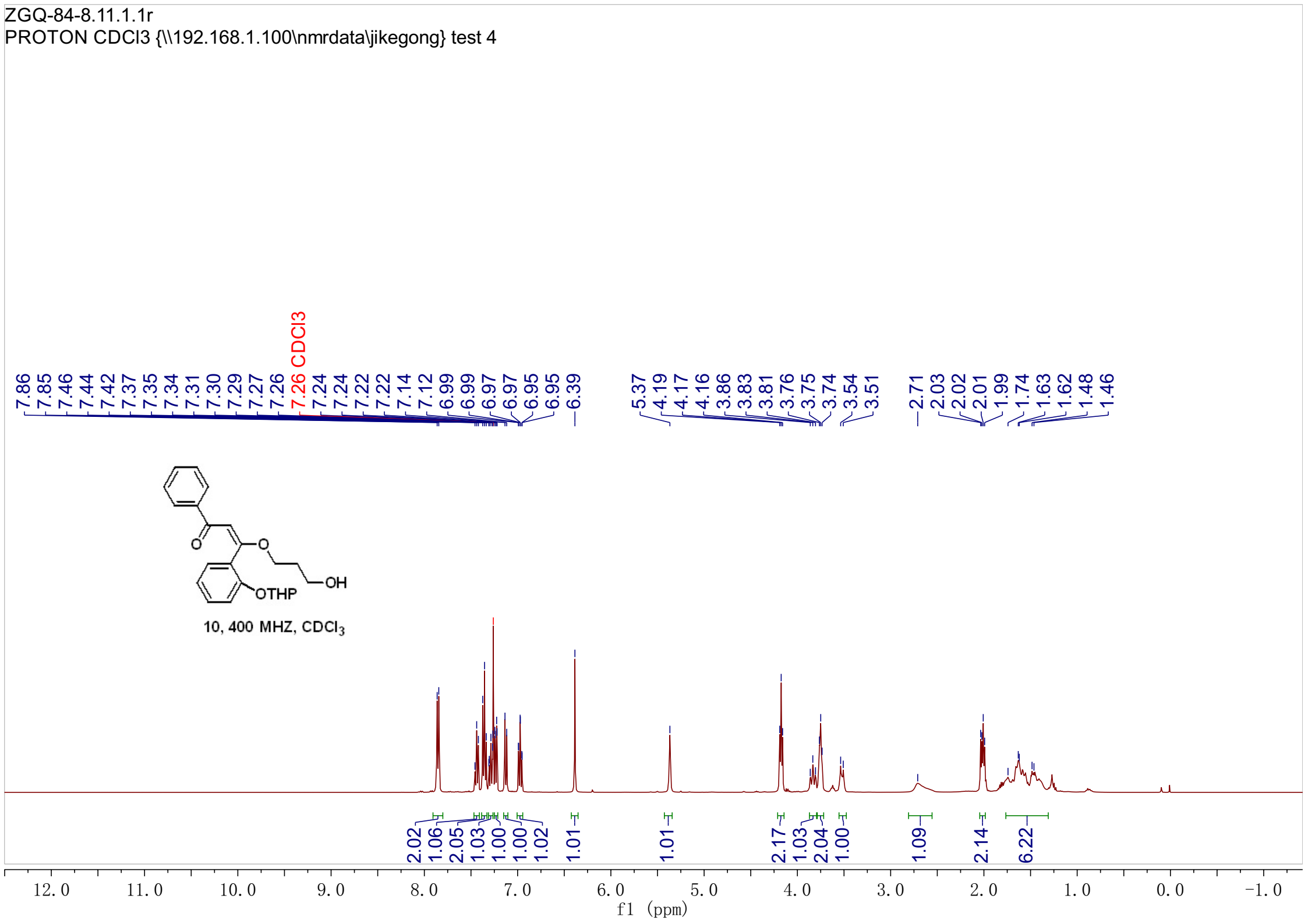
ZGQ-P60-5-2.14.1.1r
PROTON CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 13



ZGQ-P60-5-2.15.1.1r
C13CPD CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 13



ZGQ-84-8.11.1.1r
PROTON CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 4



ZGQ-84-8.10.1.1r
C13CPD CDCl3 {\\192.168.1.100\nmrdata\jikegong} test 4