

Supporting Information

Gold(I)-Catalyzed Regioselective Hydroarylation of indole-Ynones: An Approach to Carbazole Derivatives

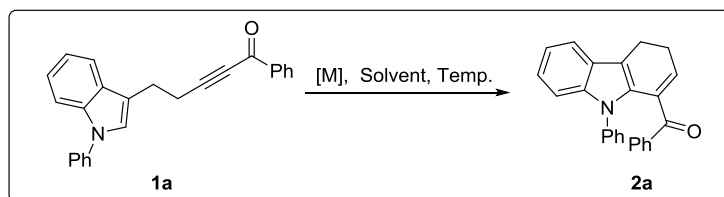
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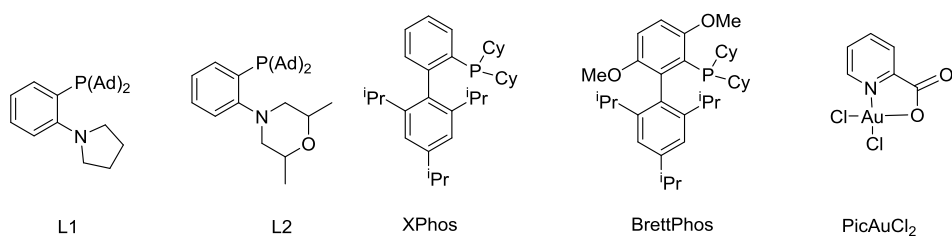
1. General Information

All commercial available chemicals were used without further purification. Anhydrous 1,2-dichloroethane, PhCl and PhF were bought from Energy Chemical. Anhydrous tetrahydrofuran (THF) and toluene were heated over sodium under N₂ for at least four hours before being distilled for use. Anhydrous DCM was heated over calcium hydride for two hours before being distilled for use. The reactions were 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 chromatography (silica gel, 200-300 mesh). All reactions were monitored by using thin layer chromatography (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. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Advance spectrometer at 500 MHz and 125 MHz, respectively. Chemical shift values are reported in δ (ppm) relative to CHCl₃ (¹H NMR, δ = 7.26; ¹³C NMR, δ = 77.16). Signal shapes are shown as s (singlet), d (doublet), t (triplet), dd (doublet of doublets), td (triplet of doublets), m (multiplet). High resolution mass spectrometry (HRMS) was performed with a Thermo Scientific LTQ Orbitrap XL.

2. Table S1. Screening conditions.



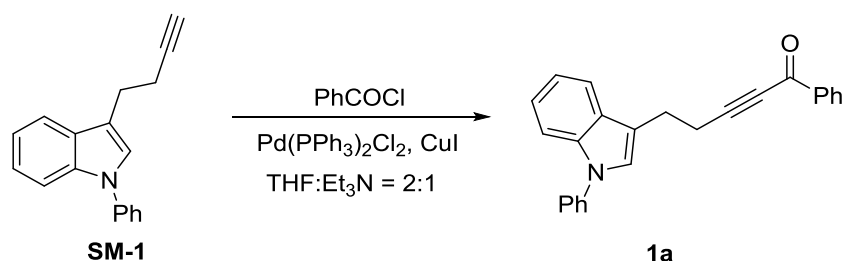
Entry	Catalyst(5 %)	Solvent	Temperature(°C)	Time(h)	Yield(%)
1	XPhosAuNTf ₂	PhCl	60	4	87
2	IPrAuNTf ₂	PhCl	60	24	2
3	PicAuCl ₂	PhCl	60	24	0
4	BrettPhosAuNTf ₂	PhCl	60	12	54
5	^t BuxphosAuNTf ₂	PhCl	60	12	43
6	PPh ₃ AuNTf ₂	PhCl	60	12	32
7	PhosphiteAuNTf ₂	PhCl	60	24	2
8	L1AuNTf ₂	PhCl	60	24	0
9	L2AuNTf ₂	PhCl	60	24	0
10	Y(OTf) ₃	PhCl	60	24	0
11	Zn(OTf) ₂	PhCl	60	24	0
12	XPhosAuNTf ₂	PhF	60	6	76
13	XPhosAuNTf ₂	Toluene	60	6	64
14	XPhosAuNTf ₂	DCE	60	6	72
15	XPhosAuNTf ₂	PhCl	50	6	70
16	XPhosAuNTf ₂	PhCl	70	6.5	62
17	XPhosAuNTf ₂ ^a	PhCl	60	12	47
18	XPhosAuOTf	PhCl	60	24	<10
19	XPhosAuSbF ₆	PhCl	60	24	<10
20	XPhosAuNTf ₂	PhCl ^b	60	4	77
21	AgNTf ₂	PhCl	60	24	0



Reaction condition: [**1a**] = 0.1 M; DCE = 1,2-dichloroethane; ^a2% catalyst. ^b[**1a**] = 0.05 M. Reactions performed on 0.2 mmol scale; isolated yields of **2a** after purification by column chromatography.

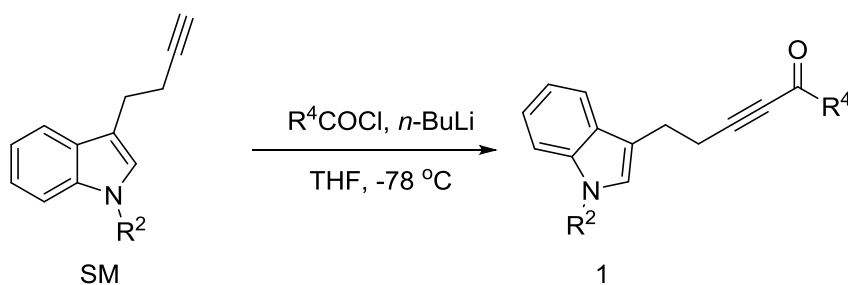
3. General Procedure for the Synthesis of 1

1) Method for synthesis **1a-e** and **1j-u**.



Add alkyne **SM - 1** (2.0 mol, 1.0 eq.) into a dry 100 ml round - bottom flask. Seal the flask with a rubber stopper, attach a nitrogen balloon, and conduct 3 - 4 air exchange operations using an air pump. Next, weigh Pd(PPh₃)₂Cl₂ (0.04 mmol, 2 mol%) and CuI (0.08 mmol, 4 mol%). Swiftly add these two catalysts to the reaction flask and repeat the air exchange process three more times following the original steps. Pump the reaction system to negative pressure and then pour in a dry THF solution. Insert a nitrogen balloon to maintain a nitrogen atmosphere. Slowly add PhCOCl (2.4 mmol, 1.2 eq.) dropwise to the reaction system, and then add triethylamine. Stir the mixture at room temperature and let it react overnight. Once the reaction is complete as confirmed by TLC, quench the reaction with a saturated NH₄Cl solution. Separate the organic phase. Extract and combine the organic phases. If an emulsion layer appears, wash it with a saturated saline solution. Dry the organic phase over anhydrous Na₂SO₄, filter, and concentrate to obtain the crude product. Purify the crude product by column chromatography (eluent: PE : EA = 30 : 1) to obtain the desired compound **1a** (641 mg, 0.17 mmol) with a yield of 88%.

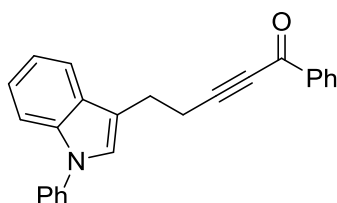
2) Method for synthesis **1f-i**.



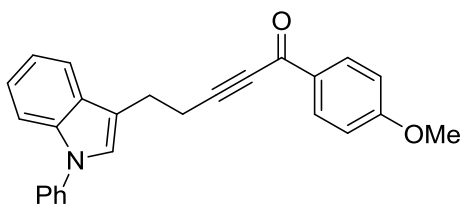
Add a dry magnetic stirrer to a 100ml dry Schlenk tube. Place **SM** (2.0 mol, 1.0 eq.) into the tube. Seal the reaction vessel and purge and replace the atmosphere to maintain a N₂ atmosphere. Next, add tetrahydrofuran to the tube and attach a nitrogen balloon. In the mixed system, slowly add n-butyllithium (2.2 mol, 1.1 eq.) dropwise. After the addition is complete, stir the mixture at -78° C for 30 minutes. Then, add R⁴COCl. Continue to stir the reaction mixture at -78° C for 2 hours, and then gradually raise the temperature to room temperature. After detecting the

completion of the reaction by TLC, quench the reaction with a saturated NH_4Cl solution. Extract and separate the organic phase. Add ethyl acetate to the aqueous phase to extract the remaining product in the water. Combine the organic layers and dry them over anhydrous Na_2SO_4 . Remove the solvent under reduced - pressure distillation. Findally, purify the crude product by column chromatography using petroleum ether: ethyl acetate = 15:1 (PE:EA = 15:1) as the eluent to obtain product **1f - i**.

4. Data of substrates **1**

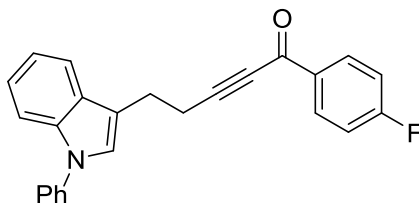


1-phenyl-5-(1-phenyl-1H-indol-3-yl)pent-2-yn-1-one. According to the known procedure,^[1] **1a** was isolated in 614 mg, 1.76 mmol, yield = 88 %. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.05 (dd, $J = 8.0, 1.5$ Hz, 2H), 7.70 (d, $J = 7.8$ Hz, 1H), 7.61 (d, $J = 8.2$ Hz, 1H), 7.57 – 7.52 (m, 1H), 7.51 – 7.47 (m, 4H), 7.40 – 7.33 (m, 3H), 7.32 (s, 1H), 7.28 (d, $J = 6.9$ Hz, 1H), 7.22 (t, $J = 7.4$ Hz, 1H), 3.23 (t, $J = 7.3$ Hz, 2H), 2.97 (t, $J = 7.3$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 178.3, 139.7, 136.9, 136.2, 134.0, 129.7, 129.7, 128.57, 128.55, 126.4, 125.8, 124.2, 122.8, 120.3, 119.0, 115.3, 110.8, 96.3, 80.4, 23.9, 20.6. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{19}\text{NONa}^+$ ($\text{M}+\text{Na}$) $^+$ 372.1359, found 372.1358.

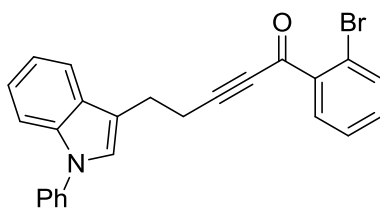


1-(4-methoxyphenyl)-5-(1-phenyl-1H-indol-3-yl)pent-2-yn-1-one. According to the known procedure,^[1] **1b** was isolated in 671 mg, 1.78 mmol, yield = 89 %. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.99 (d, $J = 8.4$ Hz, 2H), 7.68 (d, $J = 7.8$ Hz, 1H), 7.59 (d, $J = 8.2$ Hz, 1H), 7.52 – 7.46 (m, 4H), 7.32 (d, $J = 15.1$ Hz, 2H), 7.26 – 7.17 (m, 2H), 6.80 (d, $J = 8.5$ Hz, 2H), 3.82 (s, 3H), 3.21 (t, $J = 7.2$ Hz, 2H), 2.94 (t, $J = 7.2$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ :

167.8, 164.4, 139.9, 136.3, 132.1, 130.4, 129.8, 128.7, 126.4, 125.9, 124.3, 122.8, 120.3, 119.1, 115.5, 113.8, 110.8, 95.4, 80.5, 55.7, 24.0, 20.6. HRMS (ESI) m/z calcd for $C_{26}H_{21}NO_2Na^+$ ($M+Na$) $^+$ 402.1465, found 402.1461.

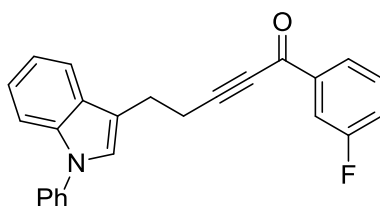


(4-fluorophenyl)-5-(1-phenyl-1H-indol-3-yl)pent-2-yn-1-one, According to the known procedure,^[1] **1c** was isolated in 660 mg, 1.8 mmol, yield = 90 %. $^1H\{^{13}C\}$ NMR (500 MHz, $CDCl_3$) δ : 8.01 (dd, $J = 8.6, 5.4$ Hz, 2H), 7.68 (d, $J = 7.8$ Hz, 1H), 7.60 (d, $J = 8.2$ Hz, 1H), 7.51 – 7.44 (m, 4H), 7.33 (t, $J = 7.0$ Hz, 1H), 7.26 (d, $J = 17.9$ Hz, 2H), 7.20 (t, $J = 7.3$ Hz, 1H), 6.97 (t, $J = 8.5$ Hz, 2H), 3.19 (t, $J = 7.2$ Hz, 2H), 2.93 (t, $J = 7.2$ Hz, 2H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ : 176.5, 166.3 (d, $J = 256.0$ Hz), 139.7, 136.2, 133.4 (d, $J = 2.6$ Hz), 132.2 (d, $J = 9.7$ Hz), 129.7, 128.6, 126.4, 125.8, 124.1, 122.8, 120.3, 119.0, 115.6 (d, $J = 22.3$ Hz), 115.3, 110.8, 96.5, 80.1, 23.8, 20.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ -103.67. HRMS (ESI) m/z calcd for $C_{25}H_{18}FNONa^+$ ($M+Na$) $^+$ 390.1265, found 390.1263.

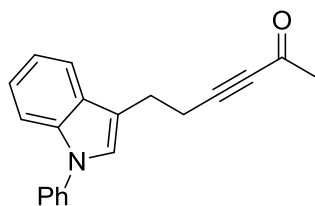


1-(2-bromophenyl)-5-(1-phenyl-1H-indol-3-yl)pent-2-yn-1-one, According to the known procedure,^[1] **1d** was isolated in 598 mg, 1.4 mmol, yield = 70 %, $^1H\{^{13}C\}$ NMR (500 MHz, $CDCl_3$) δ : 7.83 (dd, $J = 7.5, 1.8$ Hz, 1H), 7.64 (d, $J = 7.9$ Hz, 1H), 7.58 (dd, $J = 12.8, 8.1$ Hz, 2H), 7.46 (m, 7.8 Hz, 4H), 7.32 (t, $J = 7.2$ Hz, 1H), 7.29 – 7.25 (m, 2H) 7.23-7.17 (m, 3H), 3.16 (t, $J = 7.2$ Hz, 2H), 2.89 (t, $J = 7.2$ Hz, 2H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ : 177.5, 139.7, 137.4, 136.1, 134.9, 133.1, 133.0, 129.7, 128.5, 127.2, 126.4, 125.8, 124.2, 122.7, 121.0, 120.2,

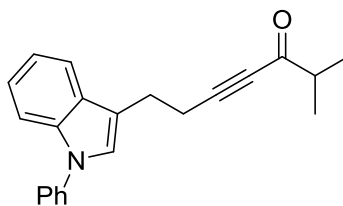
119.0, 115.2, 110.8, 97.5, 81.4, 23.6, 20.7. HRMS (ESI) m/z calcd for $C_{25}H_{18}BrNONa^+$ ($M+Na$) $^+$ 450.0464, found 450.0465.



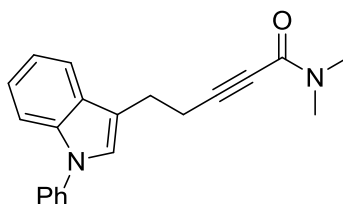
(3-fluorophenyl)-5-(1-phenyl-1H-indol-3-yl)pent-2-yn-1-one, According to the known procedure,^[1] **1e** was isolated in 624 mg, 1.7 mmol, yield = 85 %. $^1H\{^{13}C\}$ NMR (500 MHz, $CDCl_3$) δ : 7.84 (dd, $J = 7.6, 1.3$ Hz, 1H), 7.77 (dt, $J = 9.3, 2.0$ Hz, 1H), 7.72 (d, $J = 7.8$ Hz, 1H), 7.63 (d, $J = 8.2$ Hz, 1H), 7.56 – 7.47 (m, 4H), 7.39 – 7.34 (m, 2H), 7.33 – 7.29 (m, 2H), 7.27 (d, $J = 5.9$ Hz, 1H), 7.23 (d, $J = 7.4$ Hz, 1H), 3.25 (t, $J = 7.3$ Hz, 2H), 2.98 (t, $J = 7.3$ Hz, 2H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ : 176.8, 162.7 (d, $J = 248.0$ Hz), 139.7, 138.9 (d, $J = 6.7$ Hz), 136.1, 130.2 (d, $J = 7.7$ Hz), 129.7, 128.5, 126.4, 125.7, 125.6 (d, $J = 3.1$ Hz), 124.2, 122.8, 121.0 (d, $J = 21.6$ Hz), 120.3, 119.0, 115.8 (d, $J = 22.7$ Hz), 115.2, 110.8, 97.0, 80.1, 23.8, 20.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.88. HRMS (ESI) m/z calcd for $C_{25}H_{18}FNONa^+$ ($M+Na$) $^+$ 390.1265, found 390.1263.



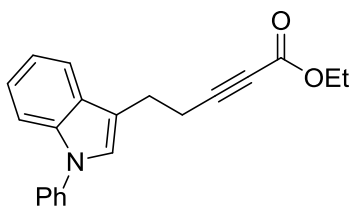
6-(1-phenyl-1H-indol-3-yl)hex-3-yn-2-one, According to the known procedure,^[1] **1f** was isolated in 345 mg, 1.2 mmol, yield = 60 %. $^1H\{^{13}C\}$ NMR (500 MHz, $CDCl_3$) δ : 7.68 – 7.65 (m, 1H), 7.59 (dd, $J = 8.2, 1.2$ Hz, 1H), 7.55 – 7.49 (m, 4H), 7.39 – 7.33 (m, 1H), 7.28 – 7.25 (m, 2H), 7.23 – 7.19 (m, 1H), 3.14 (t, $J = 7.4$ Hz, 2H), 2.80 (t, $J = 7.4$ Hz, 2H), 2.31 (s, 3H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ : 184.9, 139.8, 136.2, 129.7, 128.6, 126.4, 125.7, 124.3, 122.8, 120.2, 119.0, 115.4, 110.8, 93.6, 82.1, 32.8, 23.9, 20.4. HRMS (ESI) m/z calcd for $C_{20}H_{17}NONa^+$ ($M+Na$) $^+$ 310.1202, found 310.1201.



2-methyl-7-(1-phenyl-1H-indol-3-yl)hept-4-yn-3-one, According to the known procedure,^[1] **1g** was isolated in 397 mg, 1.26 mmol, yield = 63 %. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ: 7.70 (dd, *J* = 7.9, 3.3 Hz, 1H), 7.62 (dd, *J* = 8.2, 3.3 Hz, 1H), 7.59 – 7.51 (m, 4H), 7.42 – 7.35 (m, 1H), 7.34 – 7.20 (m, 3H), 3.26 – 3.11 (m, 2H), 2.90 – 2.82 (m, 2H), 2.67 – 2.60 (m, 1H), 1.19 (dd, *J* = 6.9, 3.4 Hz, 6H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ: 192.3, 139.7, 136.1, 129.7, 128.5, 126.3, 125.6, 124.2, 122.7, 120.1, 119.0, 115.3, 110.7, 94.6, 80.5, 43.0, 23.9, 20.3, 18.0. HRMS (ESI) *m/z* calcd for C₂₂H₂₁NONa⁺ (M+Na)⁺ 338.1515, found 338.1511.

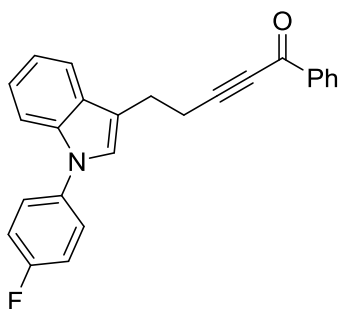


N,N-dimethyl-5-(1-phenyl-1H-indol-3-yl)pent-2-ynamide, According to the known procedure,^[1] **1h** was isolated in 329 mg, 1.04 mmol, yield = 52 %. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ: 7.64 (d, *J* = 7.8 Hz, 1H), 7.56 (d, *J* = 8.2 Hz, 1H), 7.53 – 7.47 (m, 4H), 7.34 (t, *J* = 7.0 Hz, 1H), 7.24 (d, *J* = 4.1 Hz, 2H), 7.23 – 7.16 (m, 1H), 3.13 (t, *J* = 7.4 Hz, 2H), 3.05 (s, 3H), 2.93 (s, 3H), 2.81 (t, *J* = 7.3 Hz, 2H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ: 154.8, 139.9, 136.2, 129.8, 128.7, 126.4, 125.7, 124.3, 122.7, 120.2, 119.1, 115.7, 110.8, 92.7, 74.8, 38.3, 34.1, 24.0, 20.4. HRMS (ESI) *m/z* calcd for C₂₁H₂₀N₂ONa⁺ (M+Na)⁺ 339.1468, found 339.1467.

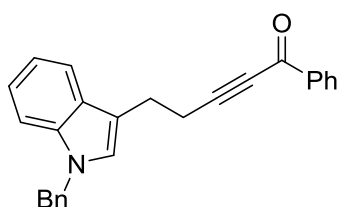


Ethyl-5-(1-phenyl-1H-indol-3-yl)pent-2-ynoate, According to the known procedure,^[1] **1i** was isolated in 318 mg, 1.0 mmol, yield = 50 %. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ: 7.65 (d, *J* =

7.8 Hz, 1H), 7.58 (d, $J = 8.2$ Hz, 1H), 7.51 (d, $J = 4.4$ Hz, 4H), 7.35 (dd, $J = 6.7, 3.6$ Hz, 1H), 7.24 (d, $J = 7.8$ Hz, 2H), 7.20 (t, $J = 7.4$ Hz, 1H), 4.28 – 4.17 (m, 2H), 3.14 (t, $J = 7.5$ Hz, 2H), 2.77 (t, $J = 7.5$ Hz, 2H), 1.32 (t, $J = 7.1$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 153.9, 139.8, 136.2, 129.7, 128.5, 126.4, 125.7, 124.3, 122.7, 120.2, 119.0, 115.3, 110.8, 89.1, 74.0, 61.9, 23.8, 20.1, 14.3. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{19}\text{NO}_2\text{Na}^+$ ($\text{M}+\text{Na}$) $^+$ 340.1308, found 340.1305.

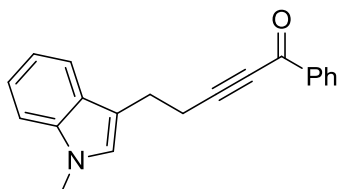


5-(1-(4-fluorophenyl)-1H-indol-3-yl)-1-phenylpent-2-yn-1-one, According to the known procedure,^[1] **1j** was isolated in 660 mg, 1.8 mmol, yield = 90 %. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.05 (d, $J = 7.5$ Hz, 2H), 7.70 (d, $J = 7.8$ Hz, 1H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.51 (d, $J = 8.2$ Hz, 1H), 7.43 (dd, $J = 8.8, 4.7$ Hz, 2H), 7.38 (t, $J = 7.8$ Hz, 2H), 7.26 (d, $J = 11.5$ Hz, 2H), 7.23 (d, $J = 7.7$ Hz, 1H), 7.19 (t, $J = 7.7$ Hz, 2H), 3.22 (t, $J = 7.2$ Hz, 2H), 2.96 (t, $J = 7.2$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 178.3, 161.0 (d, $J = 246.2$ Hz), 136.9, 136.4, 134.0, 135.8 (d, $J = 2.9$ Hz), 129.6, 128.6, 128.4, 126.1 (d, $J = 8.4$ Hz), 125.9, 122.9, 120.3, 119.1, 116.6 (d, $J = 22.8$ Hz), 115.3, 110.5, 96.3, 80.4, 23.9, 20.6. ^{19}F NMR (376 MHz, CDCl_3) δ - 115.45. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{18}\text{FNONa}^+$ ($\text{M}+\text{Na}$) $^+$ 390.1265, found 390.1263.

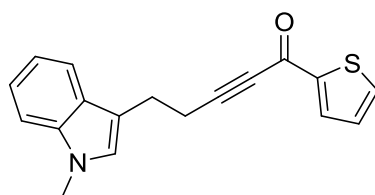


5-(1-benzyl-1H-indol-3-yl)-1-phenylpent-2-yn-1-one, According to the known procedure,^[1] **1k** was isolated in 639 mg, 1.76 mmol, yield = 88 %. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.05 (d, $J = 7.7$ Hz, 2H), 7.68 (d, $J = 7.8$ Hz, 1H), 7.58 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.6$ Hz, 2H), 7.32

(d, $J = 8.2$ Hz, 1H), 7.28 – 7.20 (m, 4H), 7.17 (t, $J = 7.4$ Hz, 1H), 7.13 – 7.10 (m, 2H), 7.08 (s, 1H), 5.29 (s, 2H), 3.20 (t, $J = 7.2$ Hz, 2H), 2.93 (t, $J = 7.2$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 178.2, 137.6, 137.0, 136.8, 133.9, 129.6, 128.8, 128.5, 127.9, 127.7, 126.9, 126.2, 122.1, 119.4, 118.9, 113.5, 110.0, 96.6, 80.3, 50.0, 24.0, 20.8. HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{21}\text{NONa}^+$ ($\text{M}+\text{Na}$) $^+$ 386.1515, found 386.1512.

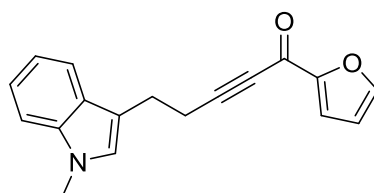


5-(1-methyl-1H-indol-3-yl)-1-phenylpent-2-yn-1-one, According to the known procedure,^[1] **11** was isolated in 460 mg, 1.6 mmol, yield = 80 %. isolated by column chromatography in 0.6 g, 2.1 mmol. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.06 – 8.01 (m, 2H), 7.64 (d, $J = 7.9$ Hz, 1H), 7.60 – 7.55 (m, 1H), 7.42 (t, $J = 7.7$ Hz, 2H), 7.34 (d, $J = 8.2$ Hz, 1H), 7.30 – 7.25 (m, 1H), 7.15 (t, $J = 7.4$ Hz, 1H), 6.99 (s, 1H), 3.76 (s, 3H), 3.17 (t, $J = 7.2$ Hz, 2H), 2.90 (t, $J = 7.2$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 178.3, 137.1, 137.0, 133.9, 129.7, 128.5, 127.6, 126.8, 121.9, 119.1, 118.8, 112.8, 109.4, 96.7, 80.3, 32.8, 24.0, 21.0. HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{17}\text{NONa}^+$ ($\text{M}+\text{Na}$) $^+$ 310.1202, found 310.1205.

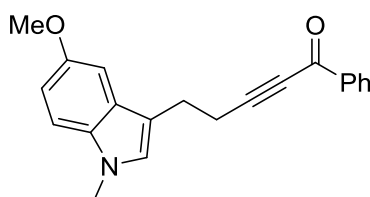


5-(1-methyl-1H-indol-3-yl)-1-(thiophen-2-yl)pent-2-yn-1-one, According to the known procedure,^[1] **1m** was isolated in 451 mg, 1.54 mmol, yield = 77 %. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.69 – 7.59 (m, 3H), 7.33 (d, $J = 8.1$ Hz, 1H), 7.26 (d, $J = 15.2$ Hz, 1H), 7.14 (t, $J = 7.4$ Hz, 1H), 7.06 (t, $J = 4.3$ Hz, 1H), 6.98 (s, 1H), 3.76 (s, 3H), 3.14 (t, $J = 7.3$ Hz, 2H), 2.86 (t, $J = 7.3$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 170.2, 145.1, 137.2, 135.3, 134.9,

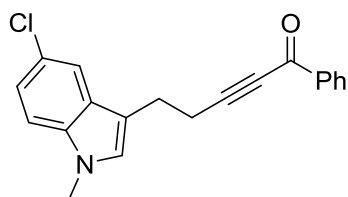
128.2, 127.6, 126.8, 121.9, 119.1, 118.8, 112.8, 109.5, 95.2, 79.9, 32.8, 23.9, 20.9. HRMS (ESI) m/z calcd for $C_{18}H_{15}NOSNa^+$ ($M+Na$) $^+$ 316.0767, found 316.0762.



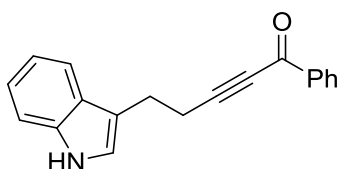
1-(furan-2-yl)-5-(1-methyl-1H-indol-3-yl)pent-2-yn-1-one, According to the known procedure,^[1] **1n** was isolated in 388 mg, 1.4 mmol, yield = 70 %. $^1H\{^{13}C\}$ NMR (500 MHz, $CDCl_3$) δ : 7.64 – 7.56 (m, 2H), 7.32 (d, $J = 8.2$ Hz, 1H), 7.24 (d, $J = 7.7$ Hz, 1H), 7.16 – 7.08 (m, 2H), 6.97 (s, 1H), 6.50 (dd, $J = 3.6, 1.8$ Hz, 1H), 3.76 (s, 3H), 3.12 (t, $J = 7.3$ Hz, 2H), 2.83 (t, $J = 7.3$ Hz, 2H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ : 165.1, 153.4, 147.8, 137.2, 127.6, 126.8, 121.9, 120.9, 119.1, 118.8, 112.8, 112.5, 109.5, 95.4, 79.6, 32.8, 23.9, 20.9. HRMS (ESI) m/z calcd for $C_{18}H_{15}NO_2Na^+$ ($M+Na$) $^+$ 300.0995, found 300.0996.



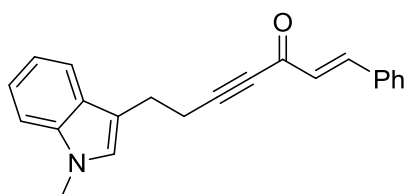
5-(5-methoxy-1-methyl-1H-indol-3-yl)-1-phenylpent-2-yn-1-one, According to the known procedure,^[1] **1o** was isolated in 507 mg, 1.6 mmol, yield = 80 %. $^1H\{^{13}C\}$ NMR (500 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.41 (t, $J = 7.8$ Hz, 2H), 7.21 (d, $J = 8.8$ Hz, 1H), 7.04 (d, $J = 2.3$ Hz, 1H), 6.95 (s, 1H), 6.91 (dd, $J = 8.8, 2.3$ Hz, 1H), 3.86 (s, 3H), 3.72 (s, 3H), 3.11 (t, $J = 7.3$ Hz, 2H), 2.87 (t, $J = 7.3$ Hz, 2H). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$) δ 178.2, 153.9, 136.8, 133.8, 132.4, 129.6, 128.4, 127.7, 127.3, 112.2, 112.0, 110.1, 100.6, 96.6, 80.1, 56.0, 32.8, 23.8, 20.8. HRMS (ESI) m/z calcd for $C_{21}H_{19}NO_2Na^+$ ($M+Na$) $^+$ 340.1308, found 340.1306.



6-(5-chloro-1-methyl-1H-indol-3-yl)-1-phenylpent-2-yn-1-one, According to the known procedure, ^[1] **1p** was isolated in 533 mg, 1.66 mmol, yield = 83 %. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ: 8.04 – 7.98 (m, 2H), 7.61 – 7.54 (m, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.25 – 7.15 (m, 2H), 7.00 (s, 1H), 3.72 (s, 3H), 3.09 (t, *J* = 7.2 Hz, 2H), 2.85 (t, *J* = 7.2 Hz, 2H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ: 178.3, 136.9, 135.5, 134.0, 129.6, 128.6, 128.5, 128.2, 125.0, 122.1, 118.3, 112.5, 110.5, 96.2, 80.4, 33.0, 23.7, 20.9. HRMS (ESI) *m/z* calcd for C₂₀H₁₆ClN₁O₁Na⁺ (*M*+Na)⁺ 344.0813, found 344.0813.

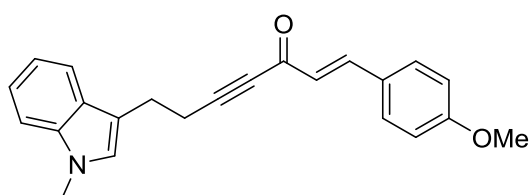


5-(1H-indol-3-yl)-1-phenylpent-2-yn-1-one, According to the general procedure, **1q** was isolated in 273 mg, 1.0 mmol, yield = 50 %. ¹H{¹³C} NMR (400 MHz, CDCl₃) δ 8.14 (s, 1H), 8.07 – 7.94 (m, 1H), 7.62 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.58 – 7.49 (m, 1H), 7.47 – 7.33 (m, 3H), 7.27 – 7.17 (m, 1H), 7.13 (ddd, *J* = 8.1, 7.1, 1.1 Hz, 1H), 7.08 (dd, *J* = 2.4, 1.0 Hz, 1H), 3.13 (t, *J* = 7.3 Hz, 2H), 2.87 (t, *J* = 7.3 Hz, 2H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 178.4, 136.8, 136.4, 134.0, 129.7, 128.5, 127.1, 122.2, 122.1, 119.5, 118.6, 114.2, 111.4, 96.8, 80.2, 23.9, 20.6. HRMS (ESI) *m/z* calcd for C₁₉H₁₅NNaO⁺ (*M*+Na)⁺ 296.1046, found 296.1043.

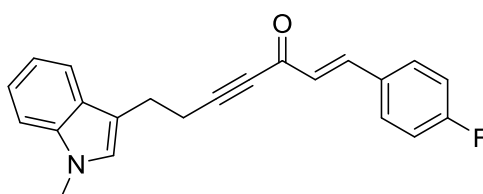


(*E*)-7-(1-methyl-1H-indol-3-yl)-1-phenylhept-1-en-4-yn-3-one, According to the known procedure, ^[1] **1r** was isolated in 326 mg, 1.04 mmol, yield = 52 %. ¹H{¹³C} NMR (500 MHz,

CDCl₃) δ : 7.66 – 7.59 (m, 2H), 7.45 – 7.35 (m, 5H), 7.32 (dd, J = 8.1, 1.0 Hz, 1H), 7.28 (dd, J = 15.1, 1.1 Hz, 1H), 7.18 – 7.11 (m, 1H), 6.98 (s, 1H), 6.73 (d, J = 16.1 Hz, 1H), 3.72 (s, 3H), 3.14 (t, J = 7.2 Hz, 2H), 2.86 (t, J = 7.3 Hz, 2H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ : 178.8, 148.5, 137.2, 134.2, 131.1, 129.1, 128.7, 128.7, 127.6, 126.8, 121.9, 119.1, 118.8, 112.9, 109.5, 95.0, 79.9, 32.8, 24.0, 20.8. HRMS (ESI) m/z calcd for C₂₂H₁₉NONa⁺ (M+Na)⁺ 336.1359, found 336.1360.

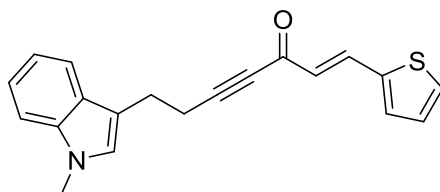


(*E*)-1-(4-methoxyphenyl)-7-(1-methyl-1H-indol-3-yl)hept-1-en-4-yn-3-one, According to the known procedure,^[1] **1s** was isolated in 343 mg, 1.0 mmol, yield = 50%. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ : 7.69 – 7.56 (m, 2H), 7.38 – 7.34 (m, 2H), 7.33 – 7.30 (m, 1H), 7.29 – 7.26 (m, 1H), 7.18 – 7.13 (m, 1H), 6.98 (s, 1H), 6.91 (d, J = 8.8 Hz, 2H), 6.63 (d, J = 16.0 Hz, 1H), 3.86 (s, 3H), 3.72 (s, 3H), 3.14 (t, J = 7.2 Hz, 2H), 2.85 (t, J = 7.3 Hz, 2H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ : 178.7, 162.1, 148.4, 137.1, 130.5, 126.9, 126.8, 126.5, 121.8, 119.0, 118.8, 114.5, 112.9, 109.4, 94.5, 79.9, 55.5, 32.7, 24.0, 20.7. HRMS (ESI) m/z calcd for C₂₃H₂₁NO₂Na⁺ (M+Na)⁺ 366.1465, found 366.1462.

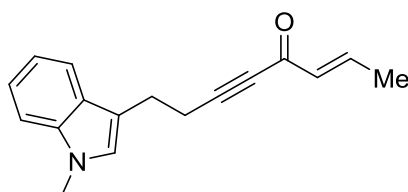


(*E*)-1-(4-fluorophenyl)-7-(1-methyl-1H-indol-3-yl)hept-1-en-4-yn-3-one, According to the known procedure,^[1] **1t** was isolated in 351 mg, 1.06 mmol, yield = 53%. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ : 7.93 (d, J = 7.9 Hz, 1H), 7.83 (d, J = 16.1 Hz, 1H), 7.66 – 7.58 (m, 3H), 7.57 – 7.51 (m, 1H), 7.48 – 7.41 (m, 1H), 7.35 (t, J = 8.6 Hz, 2H), 7.26 (dd, J = 15.1, 1.1 Hz, 1H), 6.93 (d, J = 16.1 Hz, 1H), 3.99 (s, 3H), 3.42 (t, J = 7.2 Hz, 2H), 3.15 (t, J = 7.2 Hz, 2H). ¹³C{¹H}

NMR (125 MHz, CDCl₃) δ : 178.4, 164.2 (d, $J = 252.8$ Hz), 147.0, 137.0, 130.6 (d, $J = 8.6$ Hz), 130.3 (d, $J = 3.4$ Hz), 128.3 (d, $J = 2.3$ Hz), 127.5, 126.7, 121.8, 119.0, 118.7, 116.1 (d, $J = 22.0$ Hz), 112.7, 109.4, 95.0, 79.7, 32.6, 23.9, 20.6. ¹⁹F NMR (376 MHz, CDCl₃) δ -108.25. HRMS (ESI) m/z calcd for C₂₂H₁₈FNONa⁺ (M+Na)⁺ 354.1265, found 354.1264.

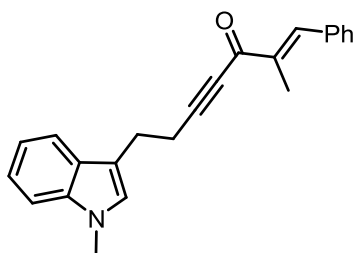


(*E*)-7-(1-methyl-1H-indol-3-yl)-1-(thiophen-2-yl)hept-1-en-4-yn-3-one, According to the known procedure,^[1] **1u** was isolated in 319 mg, 1.0 mmol, yield = 50%. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ : 7.72 (d, $J = 15.7$ Hz, 1H), 7.63 (dt, $J = 7.9, 1.0$ Hz, 1H), 7.45 (dd, $J = 5.1, 0.9$ Hz, 1H), 7.32 (dt, $J = 8.2, 1.0$ Hz, 1H), 7.26 (dd, $J = 15.2, 1.2$ Hz, 1H), 7.18 – 7.11 (m, 2H), 7.08 (dd, $J = 5.0, 3.7$ Hz, 1H), 6.98 (s, 1H), 6.55 (d, $J = 15.7$ Hz, 1H), 3.74 (s, 3H), 3.13 (t, $J = 7.3$ Hz, 2H), 2.84 (t, $J = 7.3$ Hz, 2H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ : 178.0, 140.4, 139.5, 137.1, 132.3, 130.0, 128.5, 127.6, 127.5, 126.8, 121.8, 119.1, 118.8, 112.8, 109.5, 94.8, 79.8, 32.7, 24.0, 20.8. HRMS (ESI) m/z calcd for C₂₀H₁₇NOSNa⁺ (M+Na)⁺ 342.0923, found 342.0919.



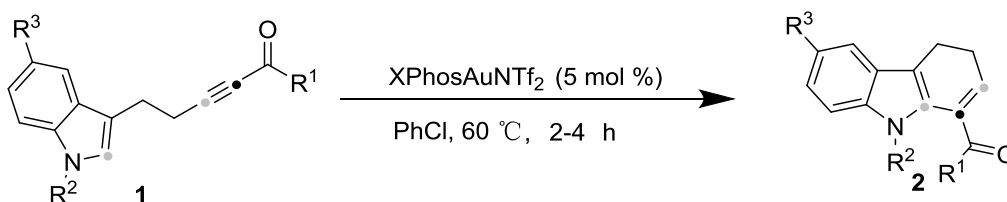
(*E*)-8-(1-methyl-1H-indol-3-yl)oct-2-en-5-yn-4-one, According to the known procedure,^[1] **1v** was isolated in 226 mg, 0.9 mmol, yield = 45%. ¹H{¹³C} NMR (500 MHz, CDCl₃) δ : 7.60 (dt, $J = 8.0, 1.1$ Hz, 1H), 7.32 (dt, $J = 8.2, 1.0$ Hz, 1H), 7.29 – 7.19 (m, 1H), 7.18 – 7.10 (m, 1H), 7.04 – 6.92 (m, 2H), 6.13 (dt, $J = 15.6, 1.7$ Hz, 1H), 3.77 (s, 3H), 3.09 (t, $J = 7.4$ Hz, 2H), 2.78 (t, $J = 7.4$ Hz, 2H), 1.90 (dd, $J = 6.9, 1.7$ Hz, 3H). ¹³C{¹H} NMR (125 MHz, CDCl₃) δ : 178.7,

149.4, 136.9, 133.8, 127.4, 126.5, 121.6, 118.8, 118.6, 112.7, 109.2, 94.2, 79.3, 32.6, 23.8, 20.6, 18.3. HRMS (ESI) m/z calcd for $C_{17}H_{17}NONa^+$ ($M+Na$) $^+$ 274.1202, found 274.1203.



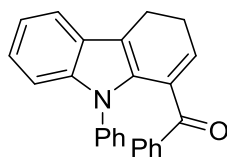
(*E*)-2-methyl-7-(1-methyl-1H-indol-3-yl)-1-phenylhept-1-en-4-yn-3-one. According to the known procedure, ^[1] **1w** was isolated in 405 mg, 1.24 mmol, yield = 62%. ¹H{¹³C} NMR (400 MHz, CDCl₃) δ 7.81 (d, *J* = 1.6 Hz, 1H), 7.59 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.44 – 7.34 (m, 3H), 7.30 (dd, *J* = 8.0, 1.7 Hz, 2H), 7.27 – 7.23 (m, 1H), 7.24 – 7.20 (m, 1H), 7.11 (ddd, *J* = 7.9, 6.6, 1.3 Hz, 1H), 6.94 (s, 1H), 3.62 (s, 3H), 3.10 (t, *J* = 7.2 Hz, 2H), 2.83 (t, *J* = 7.2 Hz, 2H), 2.07 (s, 2H). ¹³C{¹H}NMR (101 MHz, CDCl₃) δ 181.1, 145.5, 138.0, 137.1, 135.6, 130.1, 129.1, 128.5, 127.5, 126.7, 121.8, 119.0, 118.7, 112.8, 109.4, 95.2, 79.4, 32.6, 24.0, 20.7, 12.2. HRMS (ESI) m/z calcd for $C_{23}H_{21}NNaO^+$ ($M+Na$) $^+$: 350.1515, found 350.1518.

5. General Procedure for the Synthesis of **2**.

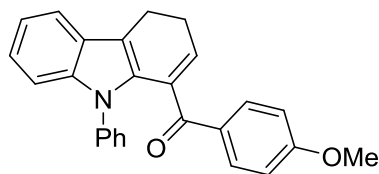


Example for the Synthesis of 2a. To a flame-dried 10 mL tube XPhosAuNTf₂ (5 mol %, 0.01 mmol, 9.5 mg) was added at room temperature, then **1a** (1.0 eq., 0.2 mmol, 70 mg) dissolved in PhCl (2 mL) was added to the above mixture. Then the tube was capped with Teflon-coated cap, and the resulting solution was stirred at room temperature for 5 min, then the resulting mixture was heated at 60 °C for 4 h. Upon completion (monitored by thin layer chromatography, Petroleum ether -EtOAc = 15 : 1, run three times), the reaction was purified by chromatography (Petroleum ether-EtOAc = 30 : 1) to get the desired compound **2a** (59 mg, 0.17 mmol, 87 %).

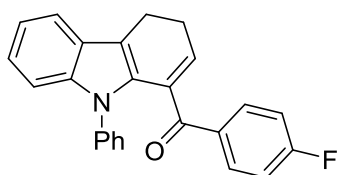
6. Data of product 2.



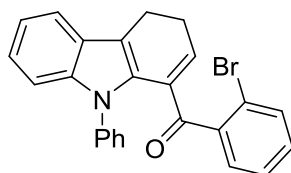
Phenyl-(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)methanone, **2a** isolated by column chromatography in 59 mg, 0.17 mmol, yield = 87 %, Melting point: 68-70.5 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.66 (d, $J = 7.9$ Hz, 1H), 7.37 (t, $J = 7.4$ Hz, 1H), 7.29 (d, $J = 7.8$ Hz, 2H), 7.26 – 7.17 (m, 2H), 7.16 (t, $J = 6.3$ Hz, 6H), 6.87 (dd, $J = 6.9, 2.5$ Hz, 2H), 6.66 (t, $J = 4.9$ Hz, 1H), 3.08 (t, $J = 8.6$ Hz, 2H), 2.77 – 2.68 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 194.2, 138.1, 137.8, 136.9, 136.9, 134.0, 133.5, 132.4, 129.2, 128.6, 128.0, 126.8, 126.7, 126.5, 122.7, 120.7, 119.0, 112.7, 110.6, 24.7, 19.8. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{19}\text{NONa}^+$ ($\text{M}+\text{Na}$) $^+$ 372.1359, found 372.1358.



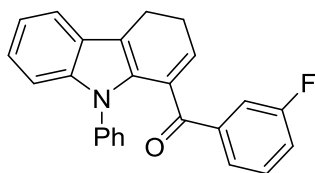
(4-methoxyphenyl)(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)methanone; **2b**, isolated by column chromatography in 75 mg, 0.198 mmol, yield = 99 %, Melting point: 80-82 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.64 (d, $J = 7.7$ Hz, 1H), 7.28 (d, $J = 8.8$ Hz, 2H), 7.23 – 7.18 (m, 2H), 7.14 (q, $J = 9.9, 7.1$ Hz, 4H), 6.90 (dd, $J = 5.8, 3.2$ Hz, 2H), 6.63 (d, $J = 8.8$ Hz, 2H), 6.58 (t, $J = 4.8$ Hz, 1H), 3.79 (s, 3H), 3.05 (t, $J = 8.6$ Hz, 2H), 2.77 – 2.62 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 193.0, 163.2, 138.1, 137.9, 135.7, 134.2, 133.8, 130.9, 130.2, 129.1, 126.9, 126.8, 126.5, 122.6, 120.7, 119.0, 113.3, 112.6, 110.6, 55.5, 24.6, 19.8. HRMS(ESI) m/z calcd for $\text{C}_{26}\text{H}_{21}\text{NO}_2\text{Na}^+$ ($\text{M}+\text{Na}$) $^+$ 402.1465, found 402.1463.



(4-fluorophenyl)(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)methanone; **2c**, isolated by column chromatography in 72 mg, 0.196 mmol, yield = 98 %, Melting point: 82-84 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.65 (d, $J = 7.8$ Hz, 1H), 7.33 – 7.26 (m, 2H), 7.23 (dd, $J = 11.5, 7.6$ Hz, 1H), 7.21 – 7.12 (m, 5H), 6.91 – 6.86 (m, 2H), 6.82 (t, $J = 8.6$ Hz, 2H), 6.66 (t, $J = 4.9$ Hz, 1H), 3.07 (t, $J = 8.7$ Hz, 2H), 2.77 – 2.66 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 192.6, 165.4 (d, $J = 254.2$ Hz), 138.0, 137.8, 136.8, 133.8, 133.4 (d, $J = 2.9$ Hz), 133.2, 131.1 (d, $J = 9.2$ Hz), 129.3, 126.9, 126.7, 126.5, 122.8, 120.8, 119.0, 115.1 (d, $J = 21.9$ Hz), 112.9, 110.6, 24.7, 19.8. ^{19}F NMR (376 MHz, CDCl_3) δ -105.88. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{18}\text{FNONa}^+$ ($\text{M}+\text{Na}$) $^+$ 390.1265, found 390.1264.

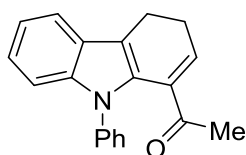


(2-bromophenyl)(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)methanone, **2d**, isolated by column chromatography in 80 mg, 0.19 mmol, yield = 93 %, Melting point: 85-86 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.62 (dd, $J = 6.8, 2.0$ Hz, 1H), 7.48 (dd, $J = 5.8, 3.3$ Hz, 1H), 7.38 (t, $J = 7.7$ Hz, 2H), 7.33 – 7.25 (m, 2H), 7.25 – 7.11 (m, 7H), 6.71 (t, $J = 5.0$ Hz, 1H), 2.99 (t, $J = 8.5$ Hz, 2H), 2.72 – 2.62 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 191.9, 143.1, 139.4, 139.0, 138.7, 134.5, 133.8, 132.5, 131.7, 130.9, 129.4, 127.1, 127.0, 126.5, 126.3, 122.9, 120.9, 120.7, 119.0, 114.7, 110.9, 25.2, 19.5. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{18}\text{BrNONa}^+$ ($\text{M}+\text{Na}$) $^+$ 450.0464, found 450.0462.

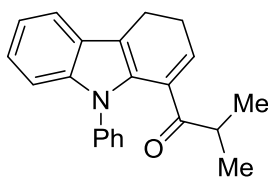


(3-fluorophenyl)(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)methanone; **2e**, isolated by column chromatography in 70 mg, 0.19 mmol, yield = 95%, Melting point: 83-85 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.65 (d, $J = 7.8$ Hz, 1H), 7.23 (t, $J = 6.8$ Hz, 1H), 7.22 – 7.16 (m, 4H),

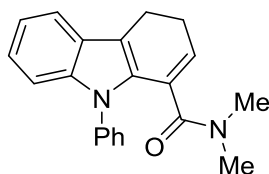
7.15 – 7.12 (m, 1H), 7.11 – 7.03 (m, 3H), 6.95 – 6.86 (m, 3H), 6.68 (t, $J = 4.9$ Hz, 1H), 3.08 (t, $J = 8.7$ Hz, 2H), 2.76 – 2.68 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 192.9, 162.5 (d, $J = 247.2$ Hz), 139.1 (d, $J = 6.45$ Hz), 138.0, 137.8, 137.5, 133.7, 133.0, 129.6 (d, $J = 7.5$ Hz), 129.3, 126.9, 126.7, 126.4, 124.5 (d, $J = 3.0$ Hz), 122.8, 120.8, 119.4 (d, $J = 21.6$ Hz), 119.1, 114.9 (d, $J = 22.4$ Hz), 112.9, 110.6, 24.7, 19.7. ^{19}F NMR (376 MHz, CDCl_3) δ -112.77. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{18}\text{FNONa}^+$ ($\text{M}+\text{Na}$) $^+$ 390.1265, found 390.1264.



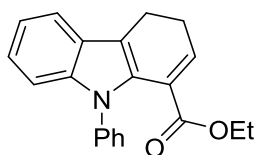
1-(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)ethan-1-one; **2f**, isolated by column chromatography in 53 mg, 0.19 mmol, yield = 93 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.60 (dd, $J = 6.5, 2.8$ Hz, 1H), 7.47 (t, $J = 7.7$ Hz, 2H), 7.42 (dd, $J = 6.6, 2.9$ Hz, 1H), 7.35 (d, $J = 7.8$ Hz, 1H), 7.34 – 7.28 (m, 2H), 7.22 – 7.14 (m, 2H), 6.77 – 6.67 (m, 1H), 2.92 (t, $J = 8.5$ Hz, 2H), 2.64 – 2.57 (m, 2H), 1.94 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 198.5, 139.5, 138.2, 136.1, 136.0, 132.5, 129.6, 127.1, 126.5, 126.3, 122.9, 120.8, 119.0, 114.6, 110.7, 27.4, 24.6, 19.6. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{17}\text{NONa}^+$ ($\text{M}+\text{Na}$) $^+$ 310.1202, found 310.1201.



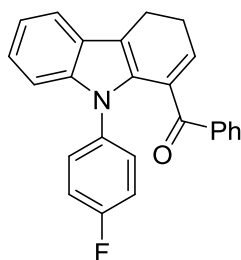
Methyl-1-(9-phenyl-4,9-dihydro-3H-carbazol-1-yl)propan-1-one; **2g**, isolated by column chromatography in 49 mg, 0.16 mmol, yield = 78 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.64 – 7.59 (m, 1H), 7.48 (t, $J = 7.8$ Hz, 2H), 7.44 – 7.38 (m, 1H), 7.37 – 7.29 (m, 3H), 7.23 – 7.16 (m, 2H), 6.63 (t, $J = 5.0$ Hz, 1H), 2.93 (t, $J = 8.5$ Hz, 2H), 2.82 – 2.76 (m, 1H), 2.62 – 2.58 (m, 2H), 0.71 (d, $J = 6.8$ Hz, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 205.9, 138.9, 138.2, 135.2, 134.8, 132.7, 129.7, 127.0, 126.7, 126.4, 122.8, 120.8, 119.0, 114.5, 110.7, 37.7, 24.5, 19.7, 18.6. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{21}\text{NONa}^+$ ($\text{M}+\text{Na}$) $^+$ 338.1515, found 338.1512.



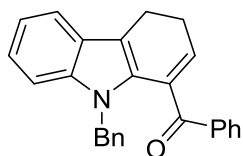
N,N-dimethyl-9-phenyl-4,9-dihydro-3H-fluorene-1-carboxamide, **2h**, isolated by column chromatography in 60 mg, 0.19 mmol, yield = 95 %, Melting point: 58-60 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.61 – 7.55 (m, 1H), 7.50 (t, $J = 7.9$ Hz, 2H), 7.41 – 7.33 (m, 3H), 7.28 – 7.21 (m, 1H), 7.20 – 7.11 (m, 2H), 6.25 (t, $J = 4.7$ Hz, 1H), 2.95 (s, 2H), 2.61 (s, 3H), 2.60 – 2.55 (m, 2H), 2.26 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 168.4, 138.2, 137.3, 132.2, 130.4, 129.7, 129.3, 129.2, 127.7, 126.4, 122.6, 120.7, 118.8, 112.5, 110.6, 38.1, 34.2, 24.1, 19.6. HRMS(ESI) m/z calcd for $\text{C}_{21}\text{H}_{20}\text{N}_2\text{ONa}^+$ ($\text{M}+\text{Na}$) $^+$ 339.1468, found 339.1461.



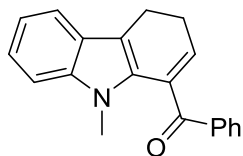
Ethyl 9-phenyl-4,9-dihydro-3H-carbazole-1-carboxylate; **2i**, isolated by column chromatography in 57 mg, 0.18 mmol, yield = 90%, Melting point: 52-54 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.61 – 7.59 (m, 1H), 7.50 (t, $J = 7.7$ Hz, 2H), 7.45 – 7.43 (m, 1H), 7.37 – 7.34 (m, 3H), 7.23 – 7.15 (m, 2H), 6.88 (t, $J = 5.0$ Hz, 1H), 3.59 – 3.54 (m, 2H), 2.95 (t, $J = 8.6$ Hz, 2H), 2.62 – 2.58 (m, 2H), 0.97 (t, $J = 7.1$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 166.5, 139.7, 138.0, 137.4, 131.9, 129.5, 127.2, 126.6, 126.4, 126.0, 122.8, 120.7, 118.9, 114.2, 110.7, 60.7, 24.6, 19.5, 14.0. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{20}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$ 318.1489, found 318.1491.



(9-(4-fluorophenyl)-4,9-dihydro-3H-carbazol-1-yl)(phenyl)methanone; **2j**, isolated by column chromatography in 68 mg, 0.18 mmol, yield= 92%, Melting point: 82-84 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.65 (d, $J = 7.8$ Hz, 1H), 7.40 (t, $J = 7.4$ Hz, 1H), 7.36 (d, $J = 7.2$ Hz, 2H), 7.20 (t, $J = 7.8$ Hz, 3H), 7.15 (d, $J = 3.1$ Hz, 2H), 6.83 (d, $J = 6.1$ Hz, 4H), 6.64 (d, $J = 4.9$ Hz, 1H), 3.07 (t, $J = 8.7$ Hz, 2H), 2.78 – 2.65 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 194.1, 161.3 (d, $J = 247.1$ Hz), 138.0, 137.0, 136.9, 134.1 (d, $J = 3.0$ Hz), 133.8, 133.5, 132.7, 128.4 (d, $J = 8.6$ Hz), 128.4, 128.2, 126.4, 122.8, 120.8, 119.1, 116.1 (d, $J = 22.9$ Hz), 112.7, 110.4, 24.7, 19.7. ^{19}F NMR (376 MHz, CDCl_3) δ -114.67. HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{19}\text{FNO}^+$ ($\text{M}+\text{H}$) $^+$: 368.1445, found 368.1446.

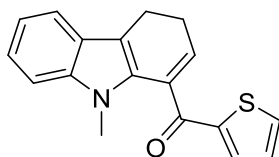


(9-benzyl-4,9-dihydro-3H-carbazol-1-yl)(phenyl)methanone; **2k**, isolated by column chromatography in 71 mg, 0.196 mmol, yield = 98 %, Melting point: 68-70 °C. (1.09 g, 3 mmol, 84%), $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.59 (d, $J = 6.9$ Hz, 1H), 7.52 – 7.40 (m, 3H), 7.29 (t, $J = 7.8$ Hz, 3H), 7.22 – 7.11 (m, 2H), 7.11 – 7.00 (m, 3H), 6.70 (d, $J = 7.2$ Hz, 2H), 6.33 (t, $J = 4.8$ Hz, 1H), 5.26 (s, 2H), 2.94 (t, $J = 8.5$ Hz, 2H), 2.68 – 2.51 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 195.4, 138.5, 138.0, 137.4, 134.4, 132.9, 132.8, 129.9, 128.5, 128.2, 127.2, 126.7, 126.0, 122.3, 119.9, 118.9, 113.0, 110.2, 48.2, 24.9, 19.4. HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{22}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$: 364.1696, found 364.1695.

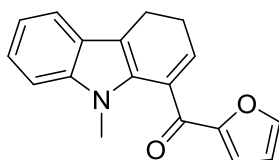


(3-methyl-4,9-dihydro-3H-carbazol-1-yl)(phenyl)methanone; **2l**, isolated by column chromatography in 49 mg, 0.17 mmol, yield = 86 %, Melting point: 42-43°C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.71 (dd, $J = 4.9, 1.2$ Hz, 1H), 7.60 (dd, $J = 3.8, 1.2$ Hz, 1H), 7.55 (dt, J

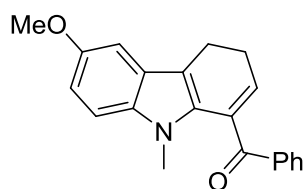
= 7.8, 1.0 Hz, 1H), 7.27 – 7.24 (m, 3H), 7.23 – 7.17 (m, 1H), 7.15 – 7.08 (m, 2H), 6.64 (t, $J = 4.9$ Hz, 1H), 3.48 (s, 3H), 2.94 (t, $J = 8.6$ Hz, 2H), 2.68 – 2.53 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 187.5, 144.2, 138.2, 136.2, 134.8, 134.5, 133.7, 133.3, 128.4, 125.8, 122.2, 119.7, 118.9, 111.8, 109.7, 32.2, 24.8, 19.6. HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$: 288.1383, found 288.1384.



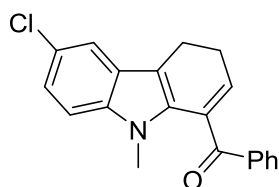
(3-methyl-4,9-dihydro-3H-carbazol-1-yl)-(thiophen-2-yl)methanone; **2m**, isolated by column chromatography in 40 mg, 0.14 mmol, yield = 68 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.71 (d, $J = 5.0$ Hz, 1H), 7.63 – 7.58 (m, 1H), 7.56 (d, $J = 7.9$ Hz, 1H), 7.29 – 7.26 (m, 1H), 7.24 – 7.18 (m, 1H), 7.17 – 7.07 (m, 2H), 6.65 (t, $J = 4.9$ Hz, 1H), 3.49 (s, 3H), 2.95 (t, $J = 8.6$ Hz, 2H), 2.67 – 2.54 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 187.4, 144.1, 138.1, 136.3, 134.8, 134.5, 133.7, 133.3, 128.4, 125.8, 122.1, 119.7, 118.9, 111.8, 109.6, 32.2, 24.8, 19.6. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NOS}^+$ ($\text{M}+\text{H}$) $^+$: 294.0947, found 294.0948.



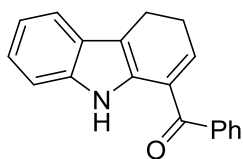
(3-furan-2-yl(9-methyl-4,9-dihydro-3H-carbazol-1-yl)-methanone; **2n**, isolated by column chromatography in 37 mg, 0.13 mmol, yield = 67 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.69 (s, 1H), 7.54 (d, $J = 7.8$ Hz, 1H), 7.27 (s, 1H), 7.20 (t, $J = 7.5$ Hz, 1H), 7.14 – 7.09 (m, 2H), 6.72 (t, $J = 4.8$ Hz, 1H), 6.55 (s, 1H), 3.50 (s, 3H), 2.92 (t, $J = 8.5$ Hz, 2H), 2.70 – 2.50 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 182.2, 152.4, 147.8, 138.2, 137.3, 133.2, 133.0, 125.7, 122.2, 120.6, 119.6, 118.9, 112.4, 111.9, 109.7, 32.3, 24.9, 19.6. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 278.1176, found 278.1175.



(3-methoxy-9-methyl-4,9-dihydro-3H-carbazol-1-yl)-(phenyl)methanone; **2o**, isolated by column chromatography in 59 mg, 0.19 mmol, yield = 93 %, Melting point: 90-91 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.94 – 7.86 (m, 2H), 7.63 – 7.55 (m, 1H), 7.45 (t, $J = 7.8$ Hz, 2H), 7.13 (d, $J = 8.9$ Hz, 1H), 6.99 (d, $J = 2.5$ Hz, 1H), 6.86 (dd, $J = 8.9, 2.5$ Hz, 1H), 6.51 (t, $J = 4.9$ Hz, 1H), 3.88 (s, 3H), 3.39 (s, 3H), 2.92 (t, $J = 8.5$ Hz, 2H), 2.66 – 2.56 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 195.5, 154.3, 137.6, 137.4, 133.9, 133.6, 133.3, 129.9, 128.7, 128.7, 125.9, 112.4, 110.9, 110.5, 100.4, 56.0, 32.3, 24.9, 19.6. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{20}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 318.1489, found 318.1488.

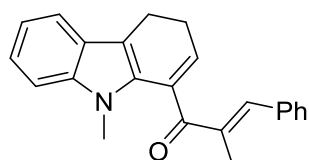


(2-chloro-9-methyl-4,9-dihydro-3H-carbazol-1-yl)(phenyl)methanone; **2p**, isolated by column chromatography in 59 mg, 0.18 mmol, yield= 92 %, Melting point: 89-90 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.94 – 7.86 (m, 2H), 7.65 – 7.52 (m, 1H), 7.51 – 7.43 (m, 3H), 7.18 – 7.09 (m, 2H), 6.56 (t, $J = 4.9$ Hz, 1H), 3.43 (s, 3H), 2.89 (t, $J = 8.6$ Hz, 2H), 2.67 – 2.57 (m, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 195.2, 138.9, 137.3, 136.5, 134.9, 133.6, 133.4, 129.9, 128.8, 126.7, 125.4, 122.2, 118.2, 111.0, 110.7, 32.5, 24.8, 19.4. HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{17}\text{ClNO}^+$ ($\text{M}+\text{H}$) $^+$: 322.0993, found 322.0993.



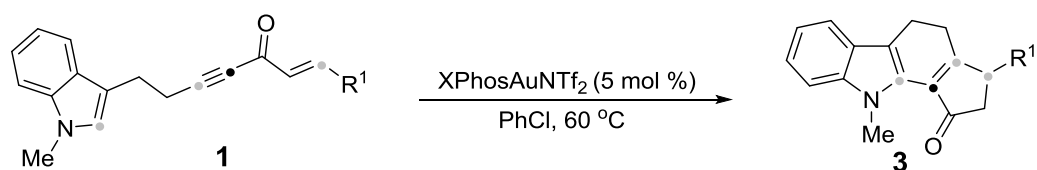
(4,9-dihydro-3H-carbazol-1-yl)(phenyl)methanone **2q**, isolated by column chromatography in 48.1 mg, 0.176 mmol, yield= 88 %. Oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (400 MHz, Chloroform- d) δ 9.75 (s,

1H), 8.00 – 7.78 (m, 2H), 7.68 – 7.57 (m, 2H), 7.52 (t, $J = 7.6$ Hz, 2H), 7.44 (d, $J = 7.8$ Hz, 1H), 7.25 – 7.20 (m, 1H), 7.18 (td, $J = 7.5, 1.2$ Hz, 1H), 6.70 (t, $J = 4.8$ Hz, 1H), 3.05 (t, $J = 9.1$ Hz, 2H), 2.75 (td, $J = 9.0, 4.7$ Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, Chloroform- d) δ 18.4, 25.5, 110.1, 111.5, 118.4, 119.5, 122.0, 125.8, 128.2, 129.3, 130.0, 130.9, 132.0, 136.0, 137.7, 143.4, 195.9. HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{15}\text{NNaO}^+$ ($\text{M}+\text{Na}$) $^+$: 296.1046, found 296.1043.



(*E*)-2-methyl-1-(9-methyl-4,9-dihydro-3H-carbazol-1-yl)-3-phenylprop-2-en-1-one **2w**, isolated by column chromatography in 49.8 mg, 0.152 mmol, yield= 76 %. Oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (400 MHz, Chloroform- d) δ 7.54 (dd, $J = 7.8, 2.3$ Hz, 1H), 7.49 (d, $J = 2.0$ Hz, 1H), 7.44 – 7.38 (m, 4H), 7.37 – 7.32 (m, 1H), 7.31 – 7.26 (m, 1H), 7.19 (td, $J = 8.3, 7.7, 2.2$ Hz, 1H), 7.12 (td, $J = 7.3, 2.3$ Hz, 1H), 6.43 (q, $J = 3.6, 2.5$ Hz, 1H), 3.54 (s, 3H), 2.93 (td, $J = 8.8, 2.4$ Hz, 2H), 2.73 – 2.53 (m, 2H), 2.26 (d, $J = 2.0$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, Chloroform- d) δ 13.6, 19.4, 24.5, 31.9, 109.4, 111.1, 118.7, 119.4, 121.8, 125.6, 128.5, 128.9, 129.9, 133.5, 133.7, 135.0, 135.5, 137.4, 137.9, 142.9, 198.4. HRMS (ESI) m/z calcd for $\text{C}_{23}\text{H}_{21}\text{NNaO}^+$ ($\text{M}+\text{Na}$) $^+$: 350.1515, found 350.1517.

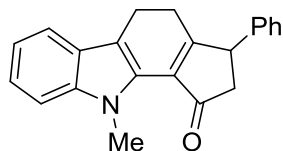
7. General Procedure for the Synthesis of **3**.



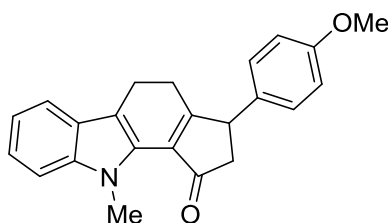
Example for the Synthesis of 3q. XPhosAuNTf₂ (5 mol %, 0.01 mmol, 9.5 mg) was added to a flamedried tube at room temperature and then a ynone **1r** (1.0 eq., 0.2 mmol, 63 mg) dissolved in PhCl (2 mL) was added to the reaction system. Then the tube was capped with Teflon-coated cap, and the resulting solution was stirred at room temperature for 5 min, and then was heated at 60 °C until the reaction was completed, as monitored by thin layer

chromatography. Upon completion (monitored by TLC) the reaction was purified by chromatography (Petroleum ether -EtOAc = 30 : 1) to get the desired compound **3r** (54 mg, 0.17 mmol, 86 %).

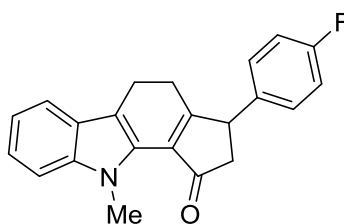
8. Data of product 3.



(3-methyl-3-phenyl-3,4,5,10-tetrahydrocyclopenta[a]carbazol-1(2H)-one; **3r**, isolated by column chromatography in 54 mg, 0.17 mmol, yield = 86 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.49 (dd, $J = 7.7, 1.1$ Hz, 1H), 7.38 – 7.34 (m, 3H), 7.32 – 7.27 (m, 1H), 7.25 – 7.18 (m, 3H), 7.11 (td, $J = 7.4, 7.0, 1.0$ Hz, 1H), 4.15 (s, 4H), 3.13 (dd, $J = 18.7, 7.0$ Hz, 1H), 3.00 – 2.84 (m, 2H), 2.60 – 2.42 (m, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 203.1, 178.3, 141.6, 138.5, 133.3, 130.8, 129.2, 127.6, 127.5, 126.0, 122.1, 119.6, 118.7, 110.4, 110.0, 48.3, 46.2, 33.4, 27.0, 19.8. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{20}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$: 314.1539, found 314.1536.

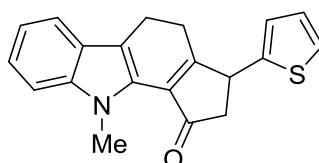


2-(4-methoxyphenyl)-10-methyl-3,4,5,10-tetrahydrocyclopenta[a]carbazol-1(2H)-one; **3s**, isolated by column chromatography in 61 mg, 0.18 mmol, yield = 89 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.49 (d, $J = 7.9$ Hz, 1H), 7.35 (d, $J = 8.3$ Hz, 1H), 7.25 – 7.18 (m, 1H), 7.15 – 7.07 (m, 3H), 6.93 – 6.85 (m, 2H), 4.14 (s, 3H), 4.09 (dd, $J = 6.8, 2.1$ Hz, 1H), 3.81 (s, 3H), 3.10 (dd, $J = 18.7, 7.0$ Hz, 1H), 2.99 – 2.84 (m, 2H), 2.61 – 2.41 (m, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 203.3, 178.7, 159.0, 138.5, 133.5, 133.0, 130.9, 128.5, 126.0, 122.0, 119.6, 118.7, 114.6, 110.3, 110.0, 55.4, 47.5, 46.4, 33.4, 27.0, 19.8. HRMS (ESI) m/z calcd for $\text{C}_{23}\text{H}_{22}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 344.1645, found 344.1645.



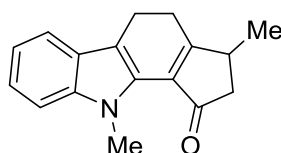
2-(4-fluorophenyl)-10-methyl-3,4,5,10-tetrahydrocyclopenta[a]carbazol-1(2H)-one; **3t**,

isolated by column chromatography in 29 mg, 0.08 mmol, yield = 43 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.49 (d, $J = 7.9$ Hz, 1H), 7.35 (d, $J = 8.3$ Hz, 1H), 7.24 – 7.20 (m, 1H), 7.19 – 7.15 (m, 2H), 7.11 (t, $J = 7.5$ Hz, 1H), 7.04 (t, $J = 8.6$ Hz, 2H), 4.14 (s, 4H), 3.12 (dd, $J = 18.7, 7.0$ Hz, 1H), 3.01 – 2.85 (m, 2H), 2.59 – 2.40 (m, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 202.9, 177.7, 162.2 (d, $J = 246.0$ Hz), 138.6, 137.3 (d, $J = 3.3$ Hz), 133.4, 130.7, 129.0 (d, $J = 8.1$ Hz), 125.9, 122.2, 119.6, 118.7, 116.1, (d, $J = 21.5$ Hz), 110.5, 110.0, 47.5, 46.2, 33.4, 26.9, 19.8. ^{19}F NMR (376 MHz, CDCl_3) δ -110.00. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{19}\text{FNO}^+$ ($\text{M}+\text{H}$) $^+$: 332.1445, found 332. 1445.



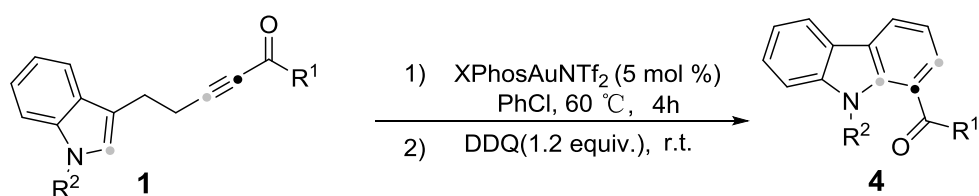
10-methyl-3-(thiophen-2-yl)-3,4,5,10-tetrahydrocyclopenta[a]carbazol-1(2H)-one; **3u**,

isolated by column chromatography in 38 mg, 0.12 mmol, yield = 60 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.50 (d, $J = 7.9$ Hz, 1H), 7.35 (d, $J = 8.3$ Hz, 1H), 7.26 – 7.19 (m, 2H), 7.12 (t, $J = 7.4$ Hz, 1H), 7.02 – 6.94 (m, 2H), 4.47 (dd, $J = 7.2, 2.2$ Hz, 1H), 4.12 (s, 3H), 3.15 (dd, $J = 18.6, 7.1$ Hz, 1H), 2.97 (t, $J = 8.6$ Hz, 2H), 2.76 – 2.51 (m, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 202.0, 176.5, 144.9, 138.6, 132.9, 130.7, 127.2, 125.9, 125.2, 124.7, 122.2, 119.6, 118.7, 110.8, 110.0, 46.6, 43.1, 33.4, 26.9, 19.9. HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NOS}^+$ ($\text{M}+\text{H}$) $^+$: 320.1104, found 320.1102.



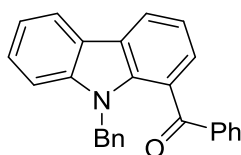
(3-3,10-dimethyl-3,4,5,10-tetrahydrocyclopenta[*a*]carbazol-1(2*H*)-one; **3v**, isolated by column chromatography in 16 mg, 0.06 mmol, yield = 32 %, oil. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.50 (d, $J = 7.9$ Hz, 1H), 7.33 (d, $J = 8.3$ Hz, 1H), 7.20 (t, $J = 7.6$ Hz, 1H), 7.11 (t, $J = 7.4$ Hz, 1H), 4.08 (s, 3H), 3.13 – 2.90 (m, 3H), 2.89 – 2.72 (m, 2H), 2.71 – 2.58 (m, 1H), 2.22 (dd, $J = 18.4, 2.3$ Hz, 1H), 1.30 (d, $J = 7.2$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 203.1, 181.2, 138.4, 132.2, 131.2, 126.0, 121.8, 119.5, 118.6, 109.9, 44.8, 36.6, 33.3, 26.5, 19.8, 19.1. HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{18}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$: 252.1383, found 252.1384.

9. General Procedure for the Synthesis of **4**.

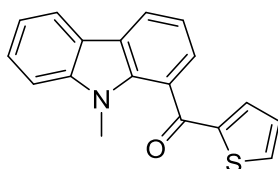


Example for the Synthesis of 4k. XPhosAuNTf₂ (5 mol %, 0.01 mmol, 9.5 mg) was added to a flamedried tube at room temperature and then a ynone (1.0 eq., 0.2 mmol, 72.7 mg) dissolved in PhCl (2 mL) was added to the reaction system. Then the tube was capped with Teflon-coated cap, and the resulting solution was stirred at room temperature for 5 min, and then was heated at 60 °C about 6h, DDQ (2 eq., 0.4 mmol, 90.8 mg) was added to the reaction system in batches, Upon completion (monitored by thin layer chromatography, hexane - ethyl acetate = 30 : 1), the reaction was purified by chromatography (Petroleum ether - EtOAc = 30 : 1) to get the desired compound **4k**(61 mg, 0.17 mmol, 84 %).

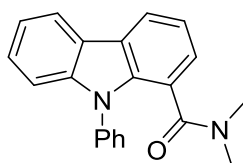
10. Data of product **4**.



(3-benzyl-9H-carbazol-1-yl)-(phenyl)methanone; **4k**, isolated by column chromatography in 61 mg, 0.17 mmol, Yield = 84 %; Melting point: 143-145 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.32 (dd, $J = 7.7, 1.3$ Hz, 1H), 8.21 (d, $J = 7.8$ Hz, 1H), 7.58 – 7.43 (m, 5H), 7.39 – 7.31 (m, 2H), 7.28 (t, $J = 7.8$ Hz, 2H), 7.22 (t, $J = 7.6$ Hz, 1H), 6.92 (t, $J = 7.3$ Hz, 1H), 6.86 (t, $J = 7.6$ Hz, 2H), 6.65 (d, $J = 7.6$ Hz, 2H), 5.60 (s, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 196.3, 142.4, 138.0, 137.5, 136.5, 132.6, 130.5, 128.4, 128.0, 128.0, 127.1, 127.0, 126.7, 125.7, 123.9, 123.2, 122.7, 120.3, 120.1, 117.9, 109.6, 48.1. HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{20}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$: 362.1539, found 362.1539.

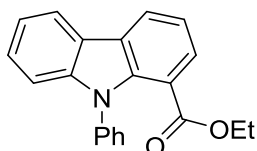


(9-methyl-9H-carbazol-1-yl)(thiophen-2-yl)methanone; **4m**, isolated by column chromatography in 38 mg, 0.13 mmol, Yield = 65 %; Melting point: 47-48 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.27 (dd, $J = 7.7, 1.2$ Hz, 1H), 8.14 (d, $J = 7.7$ Hz, 1H), 7.78 (dd, $J = 4.9, 1.2$ Hz, 1H), 7.65 (dd, $J = 7.4, 1.2$ Hz, 1H), 7.58 (dd, $J = 3.9, 1.2$ Hz, 1H), 7.52 (ddd, $J = 8.3, 7.0, 1.2$ Hz, 1H), 7.43 (d, $J = 8.2$ Hz, 1H), 7.33 – 7.24 (m, 2H), 7.16 (dd, $J = 4.9, 3.8$ Hz, 1H), 3.70 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 188.7, 145.6, 142.1, 138.4, 136.2, 135.3, 128.4, 127.4, 126.6, 125.1, 123.2, 122.7, 122.5, 120.3, 119.8, 117.9, 109.2, 33.0. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{14}\text{NOS}^+$ ($\text{M}+\text{H}$) $^+$: 292.0791, found 292.0793.

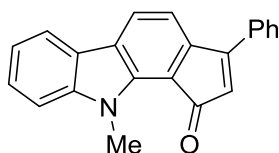


N,N-dimethyl-9-phenyl-9H-carbazole-1-carboxamide; **4h**, isolated by column chromatography in 47 mg, 0.15 mmol, yield = 74 %; Melting point: 146-148 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.17 (dd, $J = 7.6, 1.4$ Hz, 1H), 8.14 (dt, $J = 7.7, 0.9$ Hz, 1H), 7.63 – 7.51 (m, 2H), 7.50 – 7.42 (m, 3H), 7.42 – 7.38 (m, 1H), 7.36 (dd, $J = 7.3, 1.4$ Hz, 1H), 7.31 (q, $J = 7.1$ Hz,

2H), 7.23 (d, $J = 8.2$ Hz, 1H), 2.57 (s, 3H), 2.41 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 169.1, 142.4, 137.5, 136.3, 130.2, 129.0, 128.6, 128.3, 126.8, 126.5, 125.3, 124.7, 123.0, 121.2, 120.6, 120.5, 120.3, 120.1, 110.2, 38.6, 34.2. HRMS(ESI) m/z calcd for $\text{C}_{21}\text{H}_{18}\text{N}_2\text{OH}^+$ ($\text{M}+\text{H}$) $^+$ 315.1492, found 315.1495.

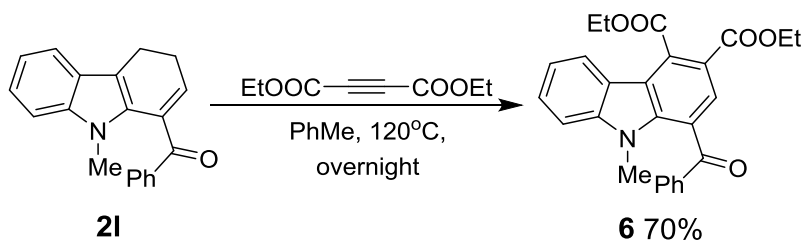


(1- ethyl 9-phenyl-9H-carbazole-1-carboxylate; **4i**, isolated by column chromatography in 44 mg, 0.14 mmol, yield = 70 %; Melting point: 83-85 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.29 (dd, $J = 7.7, 1.3$ Hz, 1H), 8.16 (dt, $J = 7.7, 0.9$ Hz, 1H), 7.78 (dd, $J = 7.5, 1.3$ Hz, 1H), 7.64 – 7.53 (m, 2H), 7.51 – 7.41 (m, 4H), 7.40 – 7.31 (m, 3H), 3.68 (q, $J = 7.2$ Hz, 2H), 1.07 (t, $J = 7.2$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 167.7, 142.6, 140.1, 138.0, 129.8, 127.8, 127.5, 126.7, 126.6, 125.8, 123.5, 123.0, 120.8, 120.2, 119.6, 117.2, 110.4, 61.1, 14.1. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{18}\text{NO}_2^+$ ($\text{M}+\text{H}$) $^+$: 316.1332, found 316.1330.



(1-methyl-3-phenylcyclopenta[a]carbazol-1(10H)-one; **4r'**, isolated by column chromatography in 43 mg, 0.14 mmol, yield = 70 %; Melting point: 115-118 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 7.98 – 7.95 (m, 2H), 7.68 – 7.66 (m, 2H), 7.54 – 7.48 (m, 4H), 7.34 (d, $J = 8.2$ Hz, 1H), 7.24 (td, $J = 7.5, 0.9$ Hz, 1H), 7.11 (d, $J = 7.6$ Hz, 1H), 5.84 (s, 1H), 4.34 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 196.8, 162.3, 144.4, 143.5, 133.7, 130.1, 129.0, 128.8, 127.7, 127.5, 124.3, 122.8, 122.8, 120.4, 120.2, 113.9, 112.9, 109.4, 100.1, 33.9. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{16}\text{NO}^+$ ($\text{M}+\text{H}$) $^+$: 310.1226, found 310.1225.

11. Procedure for the Synthesis of 6.



21 (1.0 eq., 0.4 mmol, 115 mg) and diethyl but-2-ynedioate (5 eq., 2 mmol, 340 mg) were dissolved in toluene (2 mL). The solution was added to a flame-dried tube at room temperature. Then, the reaction system was heated at 120 ° C for about 24 h. The completion of the reaction was monitored by thin-layer chromatography. After the reaction was completed, it was purified by chromatography (Petroleum ether - ethyl acetate = 15 : 1) to obtain the desired compound **6** (120.3 mg, 0.28 mmol, 70 %). *Diethyl-1-benzoyl-9-methyl-9H-carbazole-3,4-dicarboxylate*; **6** was isolated by column chromatography with a yield of 120 mg (0.28 mmol, 70 %). Melting point: 133-135 °C. $^1\text{H}\{^{13}\text{C}\}$ NMR (500 MHz, CDCl_3) δ : 8.18 (s, 1H), 8.01 (d, J = 8.0 Hz, 1H), 7.97 – 7.90 (m, 2H), 7.70 – 7.60 (m, 1H), 7.59 – 7.55 (m, 1H), 7.52 (t, J = 7.8 Hz, 2H), 7.43 (d, J = 8.2 Hz, 1H), 7.37 – 7.30 (m, 1H), 4.68 (q, J = 7.2 Hz, 2H), 4.38 (q, J = 7.1 Hz, 2H), 3.62 (s, 3H), 1.49 (t, J = 7.2 Hz, 3H), 1.37 (t, J = 7.1 Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ : 195.3, 168.9, 165.4, 142.6, 140.9, 137.5, 133.9, 131.8, 130.6, 128.8, 128.5, 127.6, 122.7, 121.6, 121.2, 121.0, 120.8, 117.1, 109.5, 62.1, 61.3, 33.1, 14.3, 14.1. HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{24}\text{NO}_5^+$ (M+H) $^+$ 430.1649, found 430.1646.

12. Reference

[1] a) W.-S. Chen, F. Yang, T. Wang, G.-Q. Zhang, Y. Wei, M.-H. Wang, Z.-S. Chen, and K. Ji *Org. Lett.* **2023**, 25, 5762–5767. b) G.-Q. Zhang, F. Yang, W.-S. Chen, X. Zhao, T. Wang, Z.-S. Chen and K. Ji *Green Chem.*, **2024**, 26, 8711–8717.

13. X-ray structure of 4r'

Recrystallization conditions to obtain the **4r'** crystal: **4r'** (10 mg, 0.032 mmol) was first dissolved in 1 mL acetone in a sample bottle until it was saturated and then 4 mL *n*-hexane was

added. The sample bottle was sealed with a parafilm at room temperature, and a small hole was pierced in the parafilm to facilitate the evaporation for obtaining **4r'** crystal.

Figure S1. X-ray structure of **4r'**

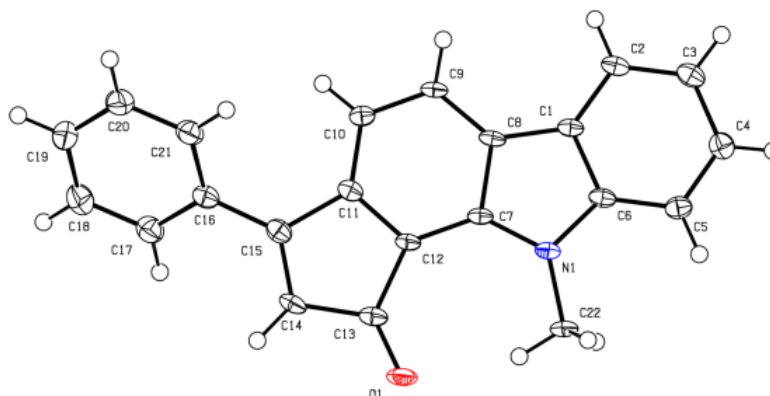


Table 1 Crystal data and structure refinement for **4r'.**

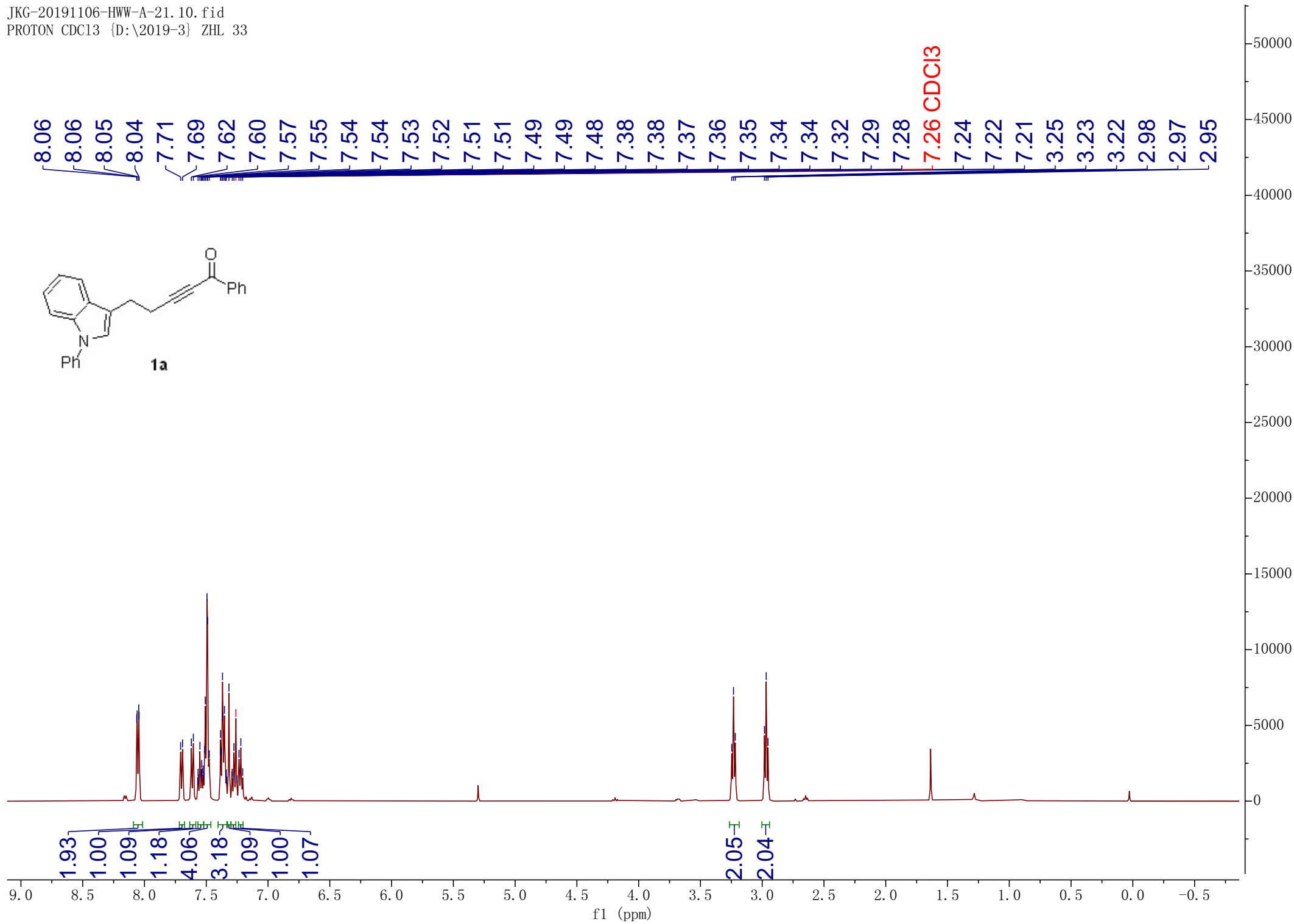
Identification code	4r'
Empirical formula	C ₂₂ H ₁₅ NO
Formula weight	309.35
Temperature/K	99.98(10)
Crystal system	orthorhombic
Space group	Pbca
a/Å	13.1658(3)
b/Å	8.4660(2)
c/Å	27.5760(8)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	3073.67(13)
Z	8
ρ _{calc} /cm ³	1.337
μ/mm ⁻¹	0.641
F(000)	1296.0
Crystal size/mm ³	0.14 × 0.1 × 0.08
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	6.41 to 146.97
Index ranges	-15 ≤ h ≤ 16, -5 ≤ k ≤ 10, -33 ≤ l ≤ 33
Reflections collected	7615
Independent reflections	3001 [R _{int} = 0.0412, R _{sigma} = 0.0354]
Data/restraints/parameters	3001/0/218
Goodness-of-fit on F ²	1.086
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0487, wR ₂ = 0.1216
Final R indexes [all data]	R ₁ = 0.0583, wR ₂ = 0.1288
Largest diff. peak/hole / e Å ⁻³	0.21/-0.24

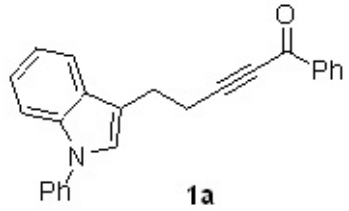
Experimental

A suitable crystal was selected and determined on a SuperNova, Dual, Cu at zero, Atlas **4r'** diffractometer. The crystal was kept at 100.00(10) K during data collection.

Crystal structure determination of **4r'**

Crystal Data for C₂₂H₁₅NO ($M = 309.35$ g/mol) : orthorhombic, space group Pbca (no. 61), $a = 13.1658(3)$ Å, $b = 8.4660(2)$ Å, $c = 27.5760(8)$ Å, $V = 3073.67(13)$ Å³, $Z = 8$, $T = 99.98(10)$ K, $\mu(\text{Cu K}\alpha) = 0.641$ mm⁻¹, $D_{\text{calc}} = 1.337$ g/cm³, 7615 reflections measured ($6.41 \leq 2\theta \leq 146.97$), 3001 unique ($R_{\text{int}} = 0.0412$, $R_{\text{sigma}} = 0.0354$) which were used in all calculations. The final R_1 was 0.0487 ($I > 2\sigma(I)$) and wR_2 was 0.1288 (all data).

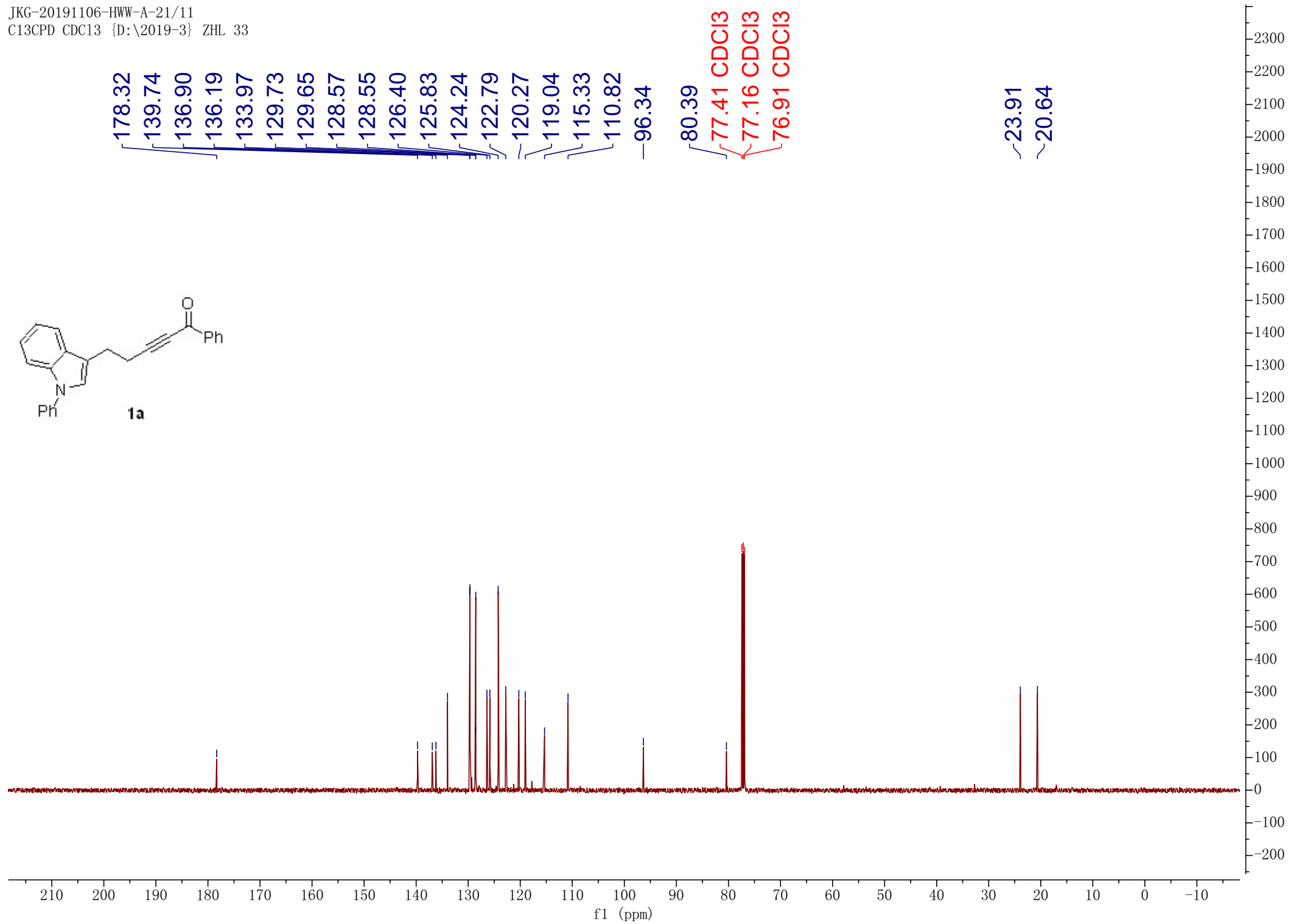


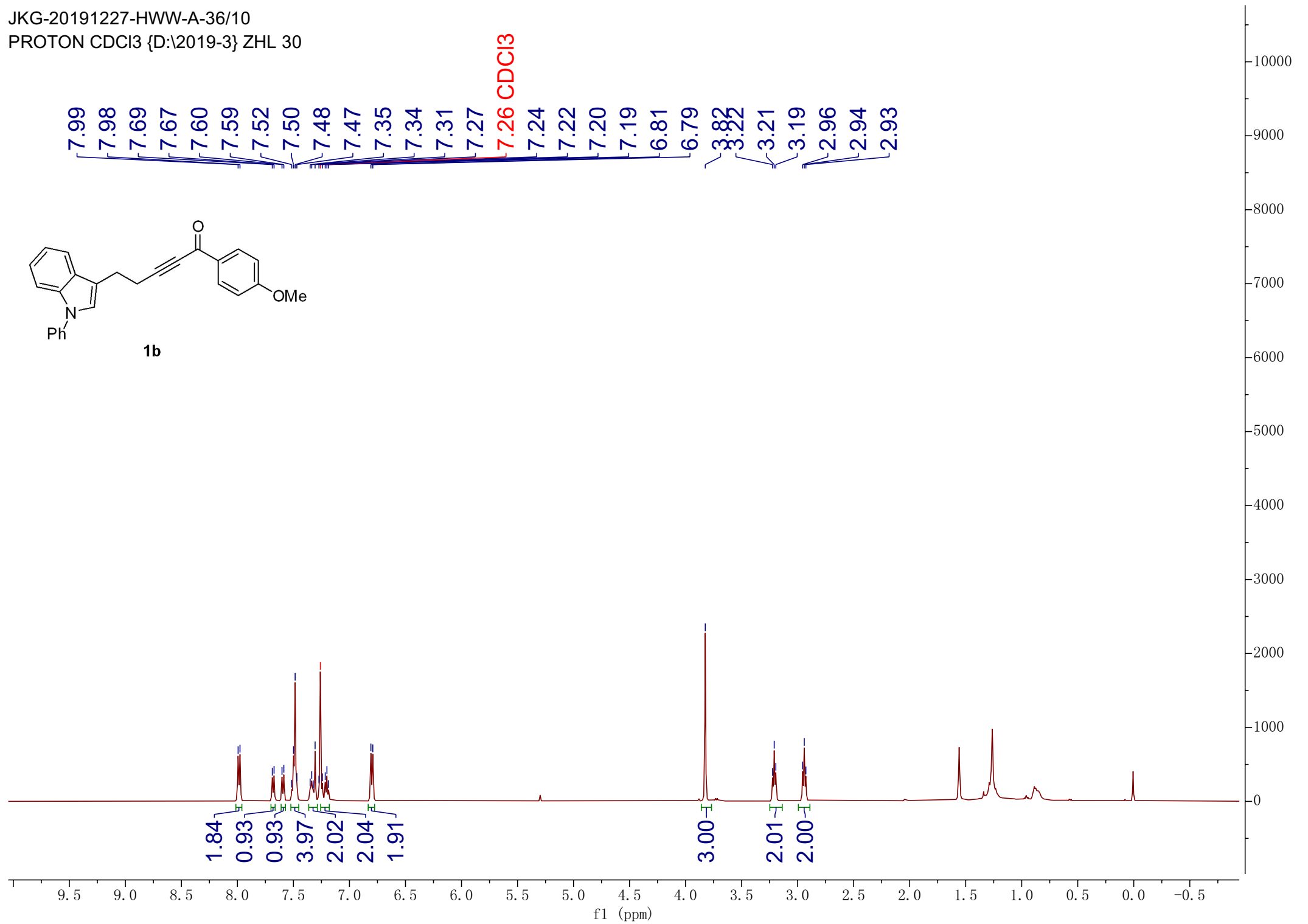
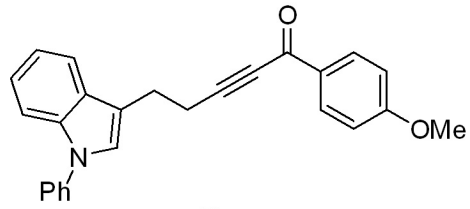


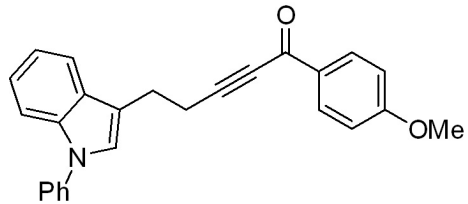
178.32
139.74
136.90
136.19
133.97
129.73
129.65
128.57
128.55
126.40
125.83
124.24
122.79
120.27
119.04
115.33
110.82
-96.34

80.39
77.41 CDC13
77.16 CDC13
76.91 CDC13

~23.91
~20.64







167.81
164.39
139.85
136.29
132.06
130.44
129.76
128.68
126.43
125.88
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120.32
119.11
115.50
113.82
110.84
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80.45

77.42 CDC13

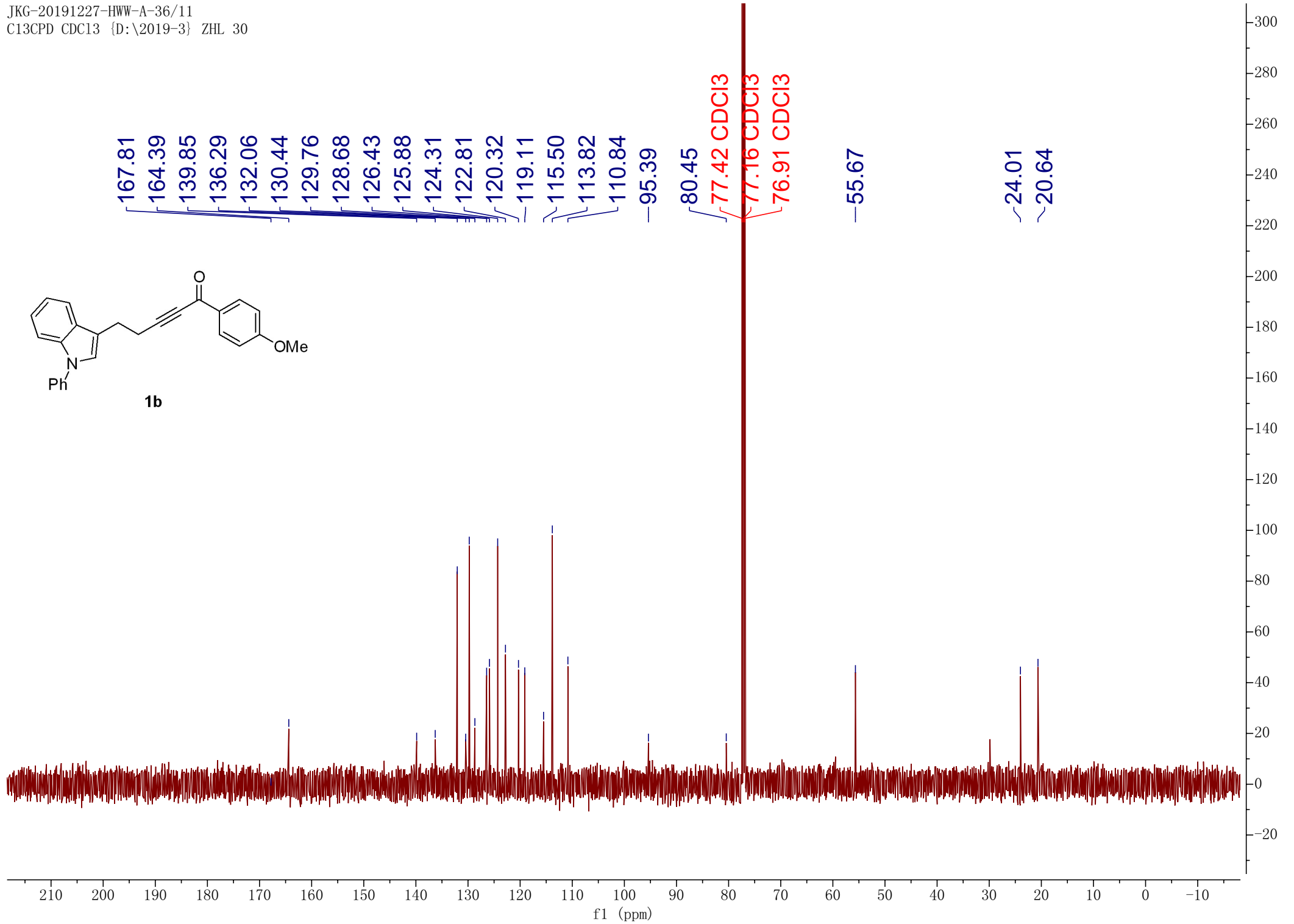
77.16 CDC13

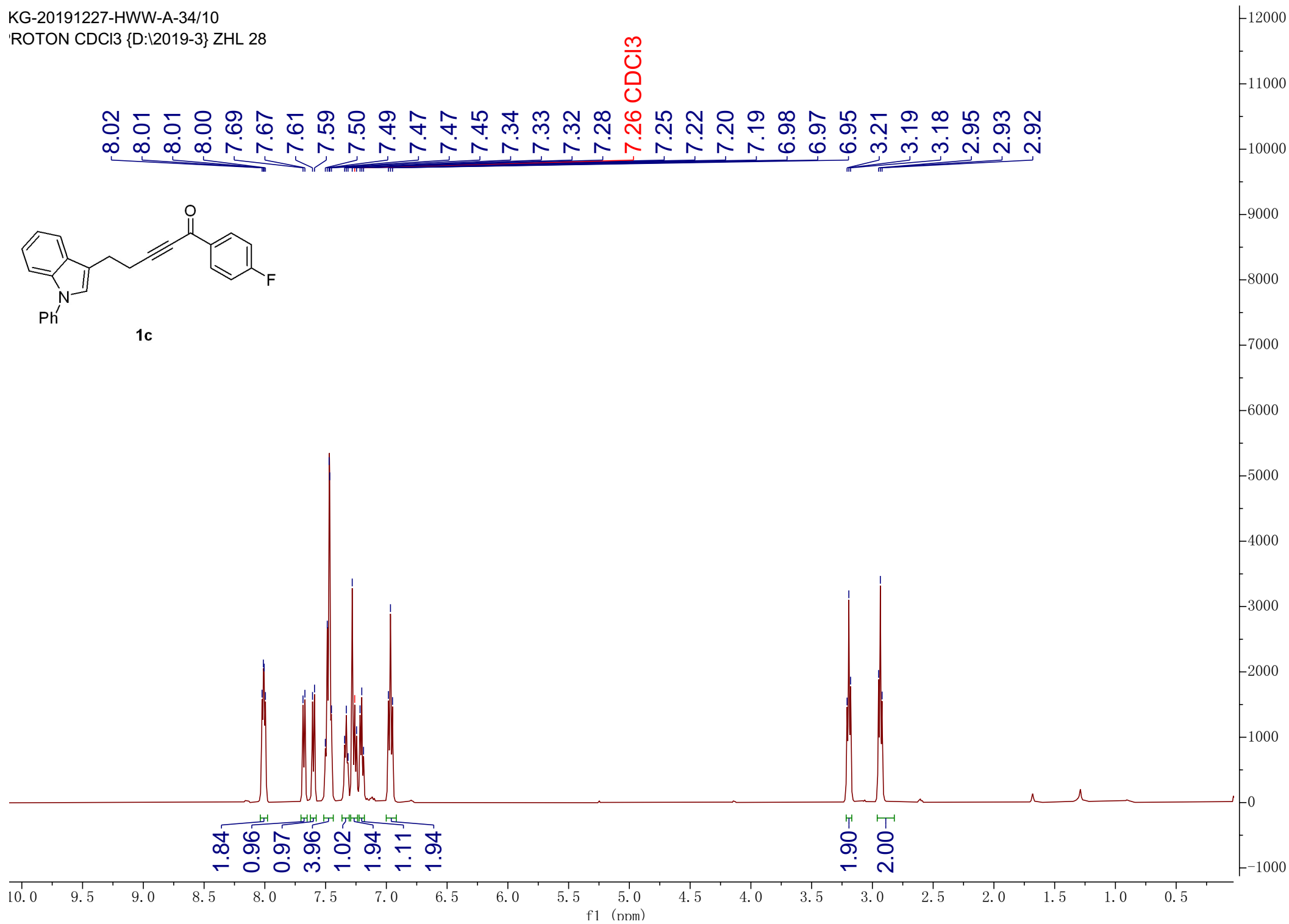
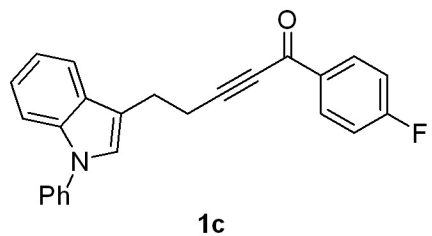
76.91 CDC13

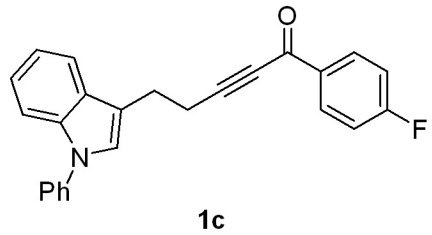
-55.67

~24.01

~20.64

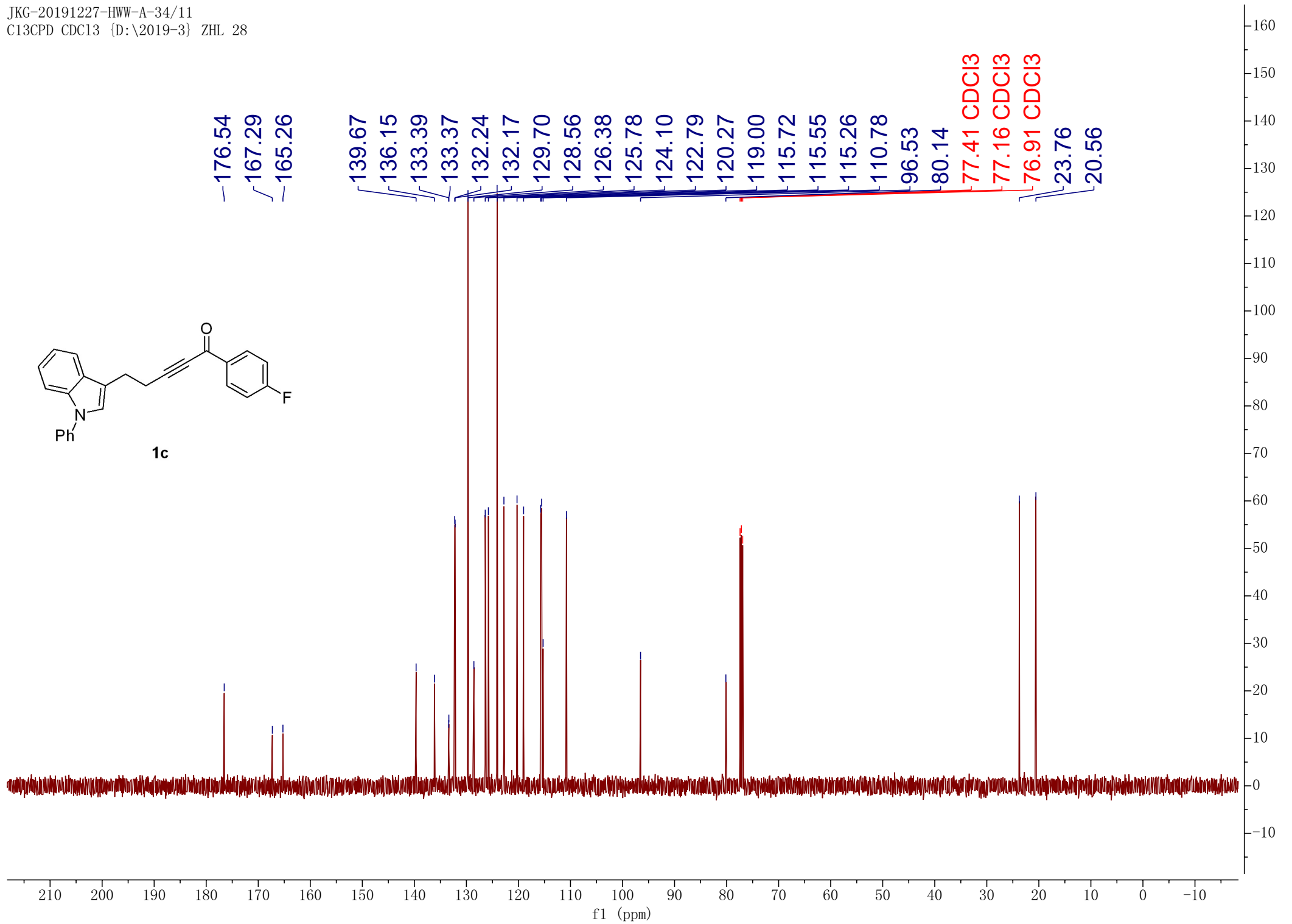


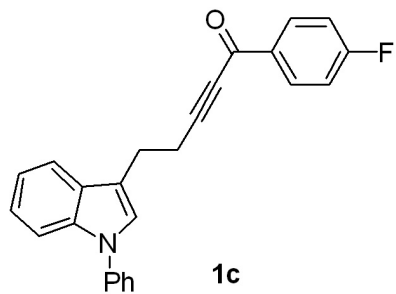




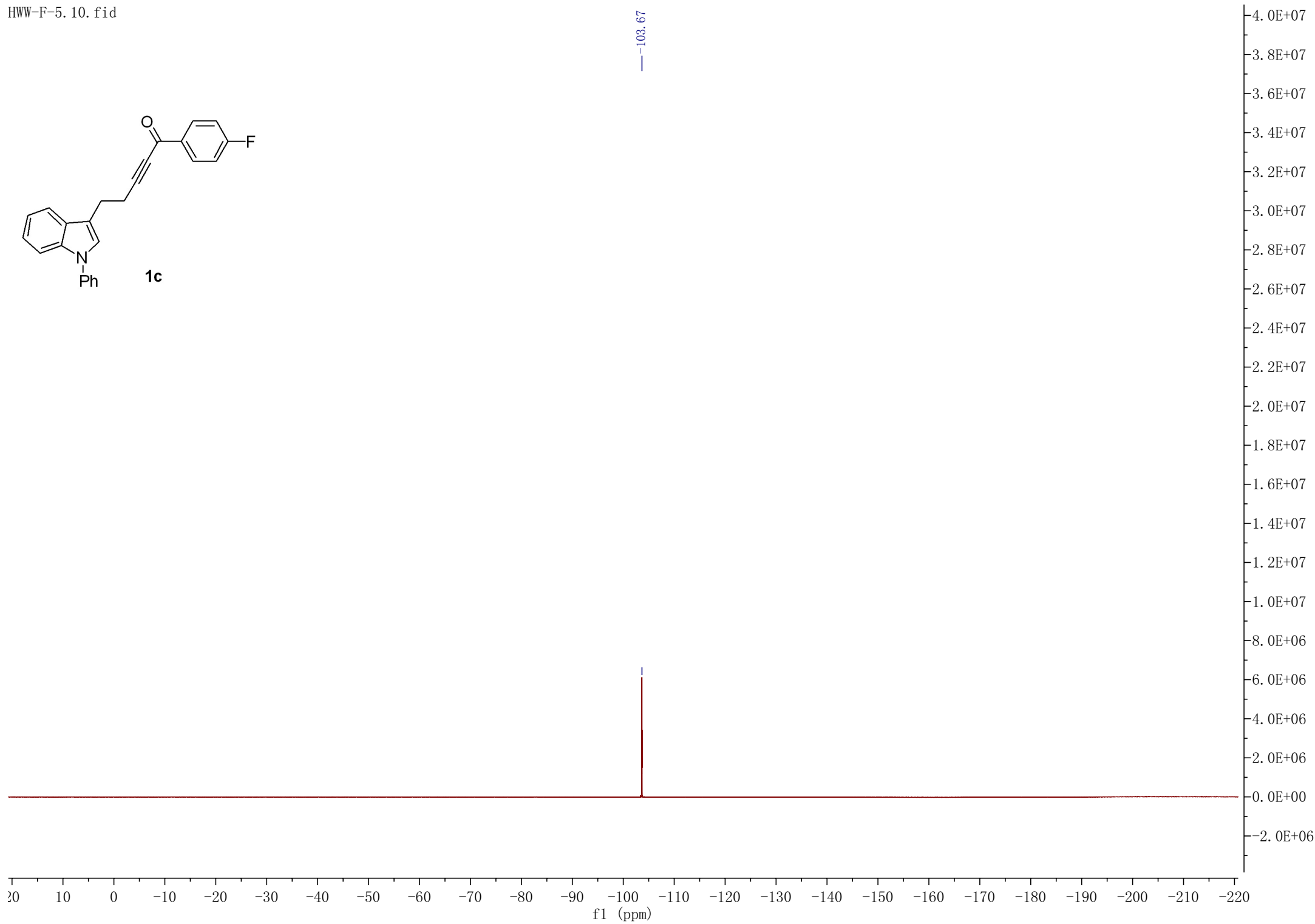
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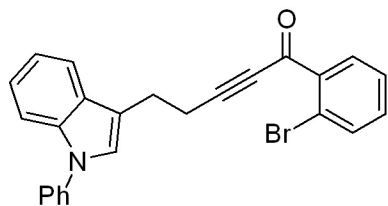


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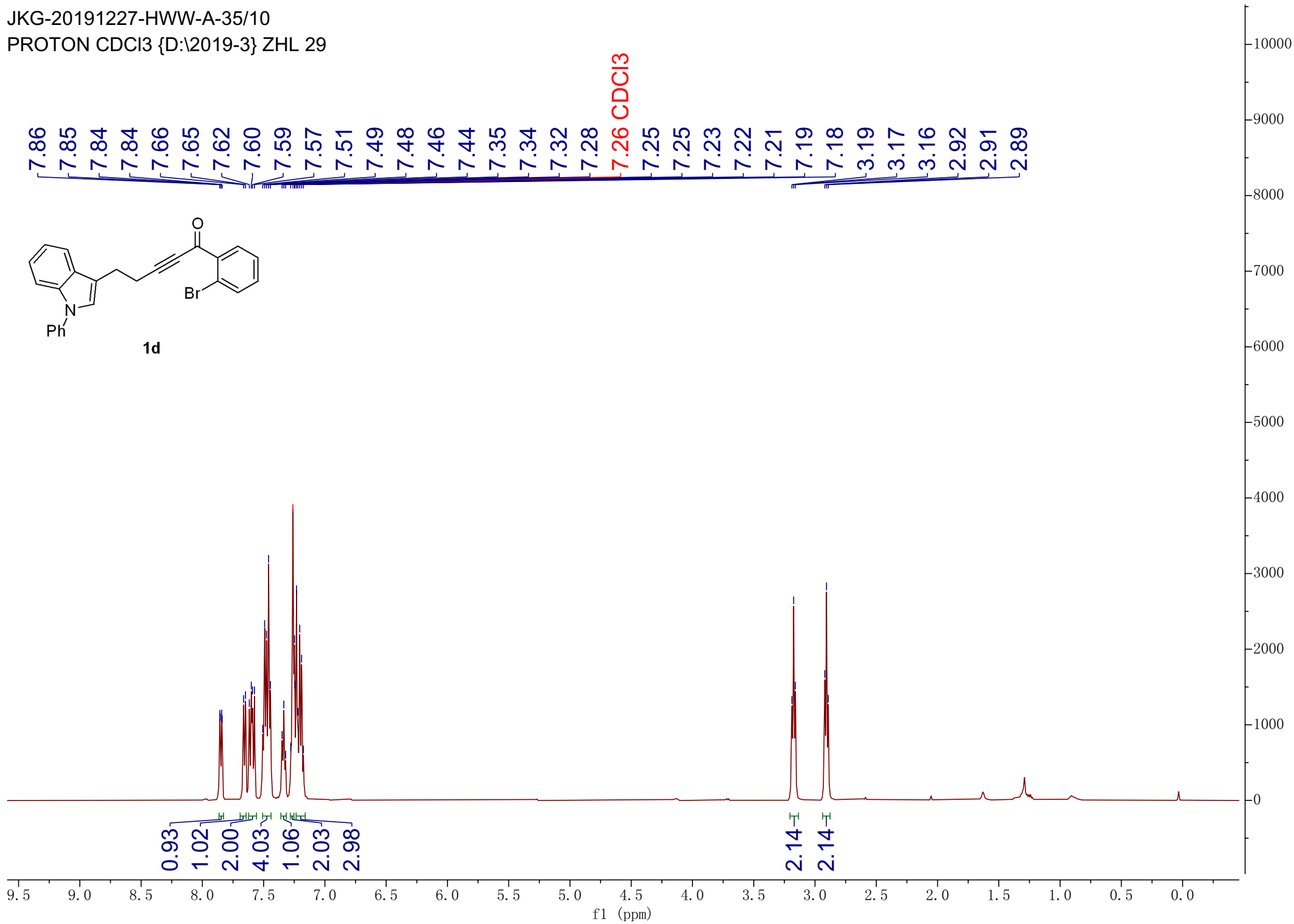


JKG-20191227-HWW-A-35/10
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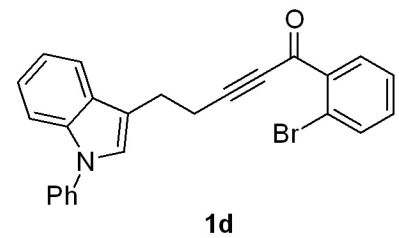
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1d



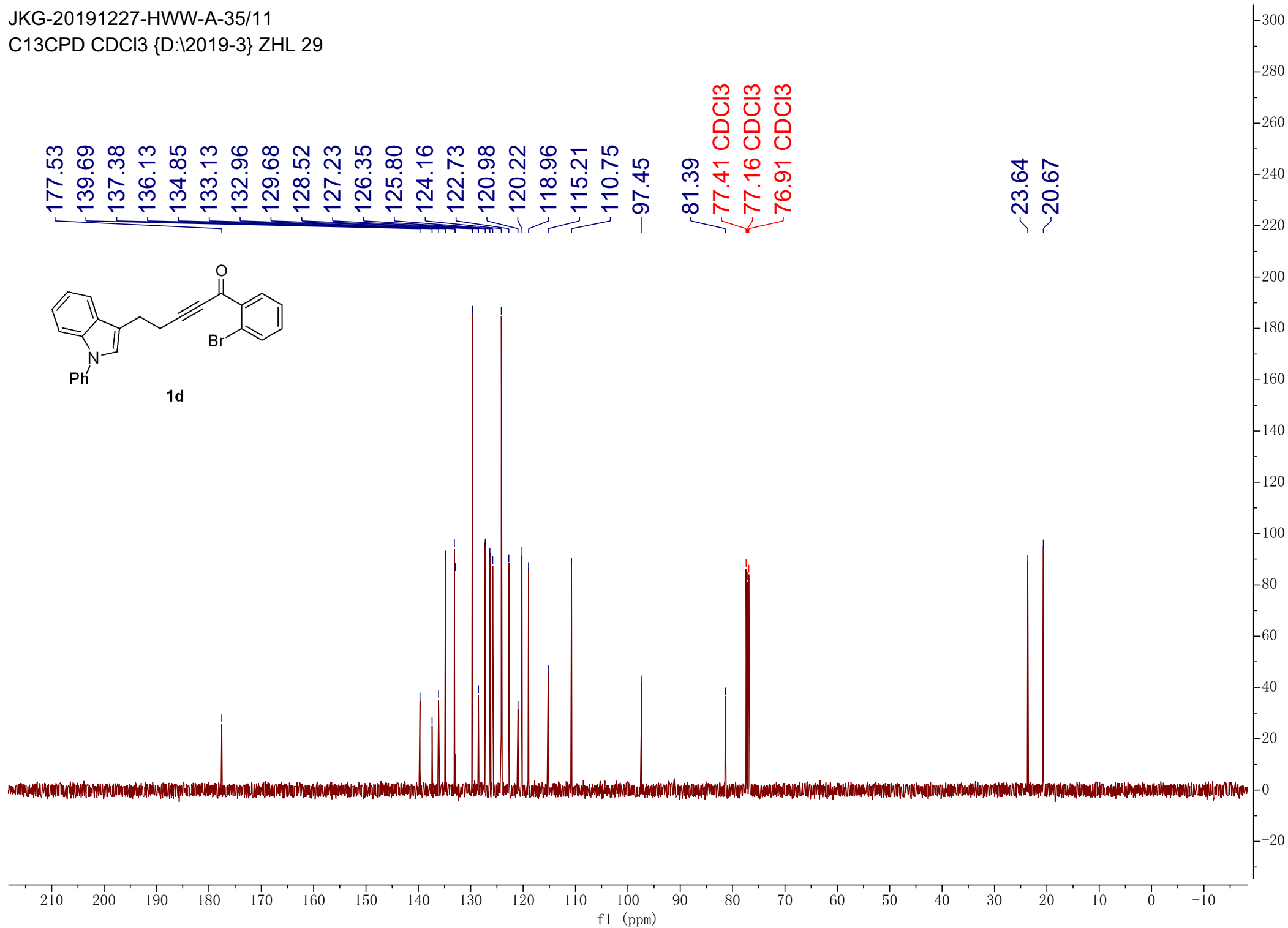
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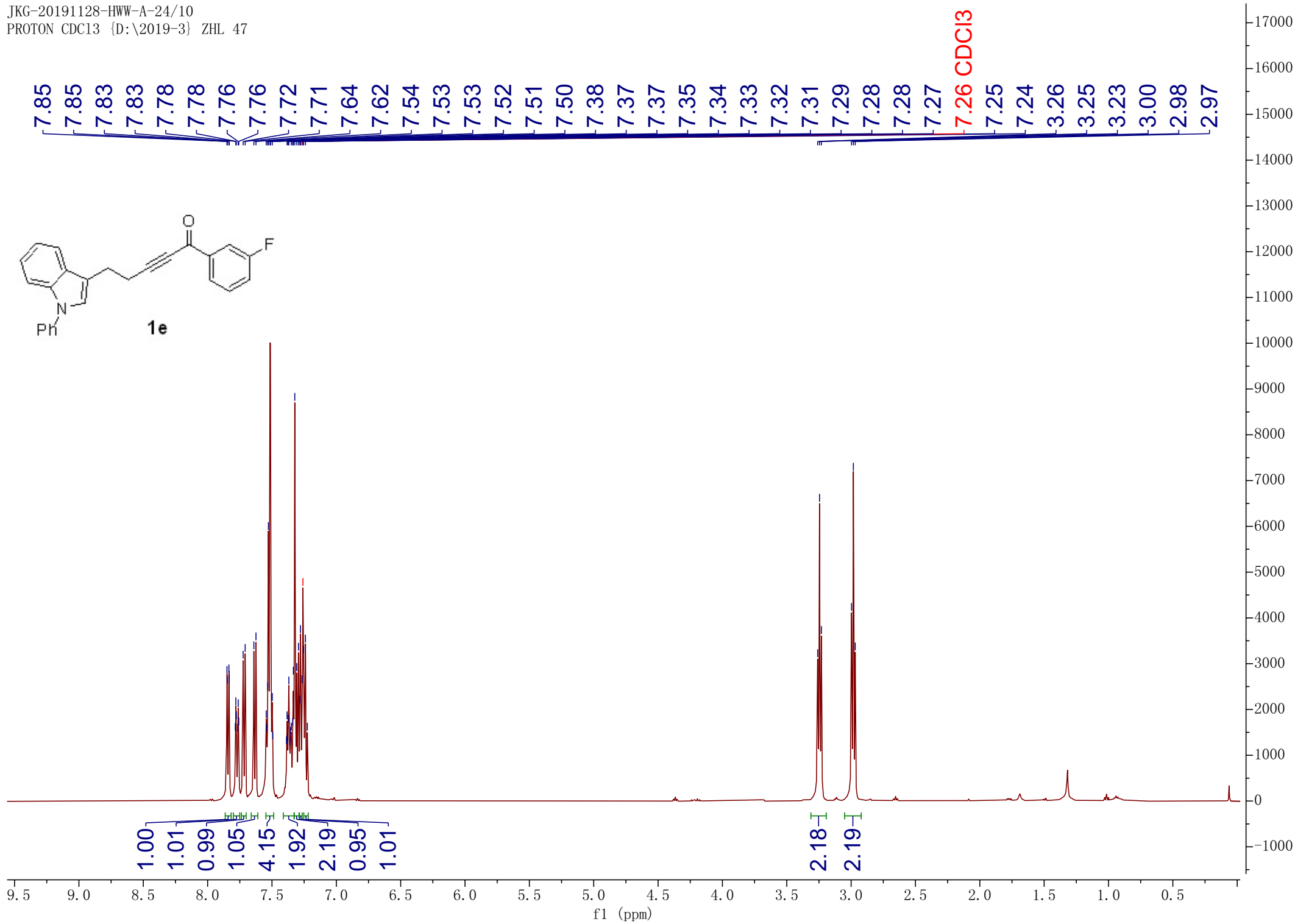
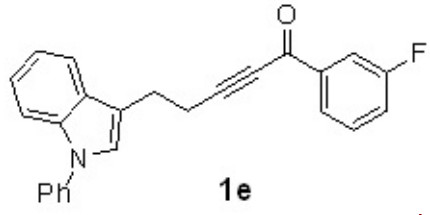


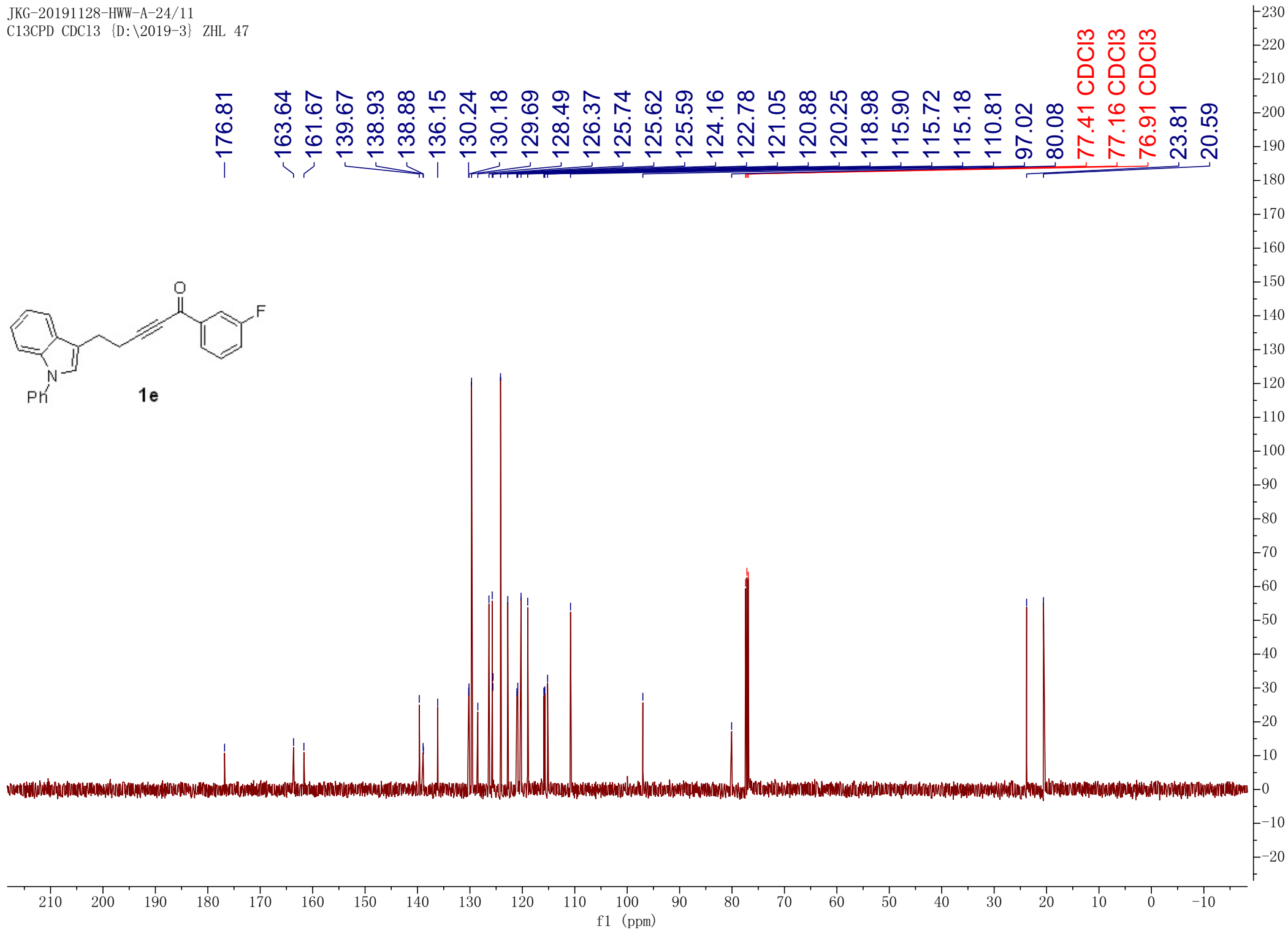
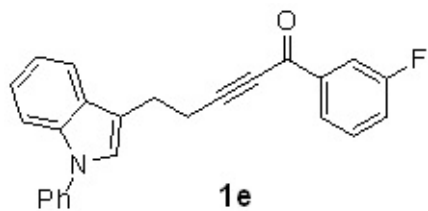
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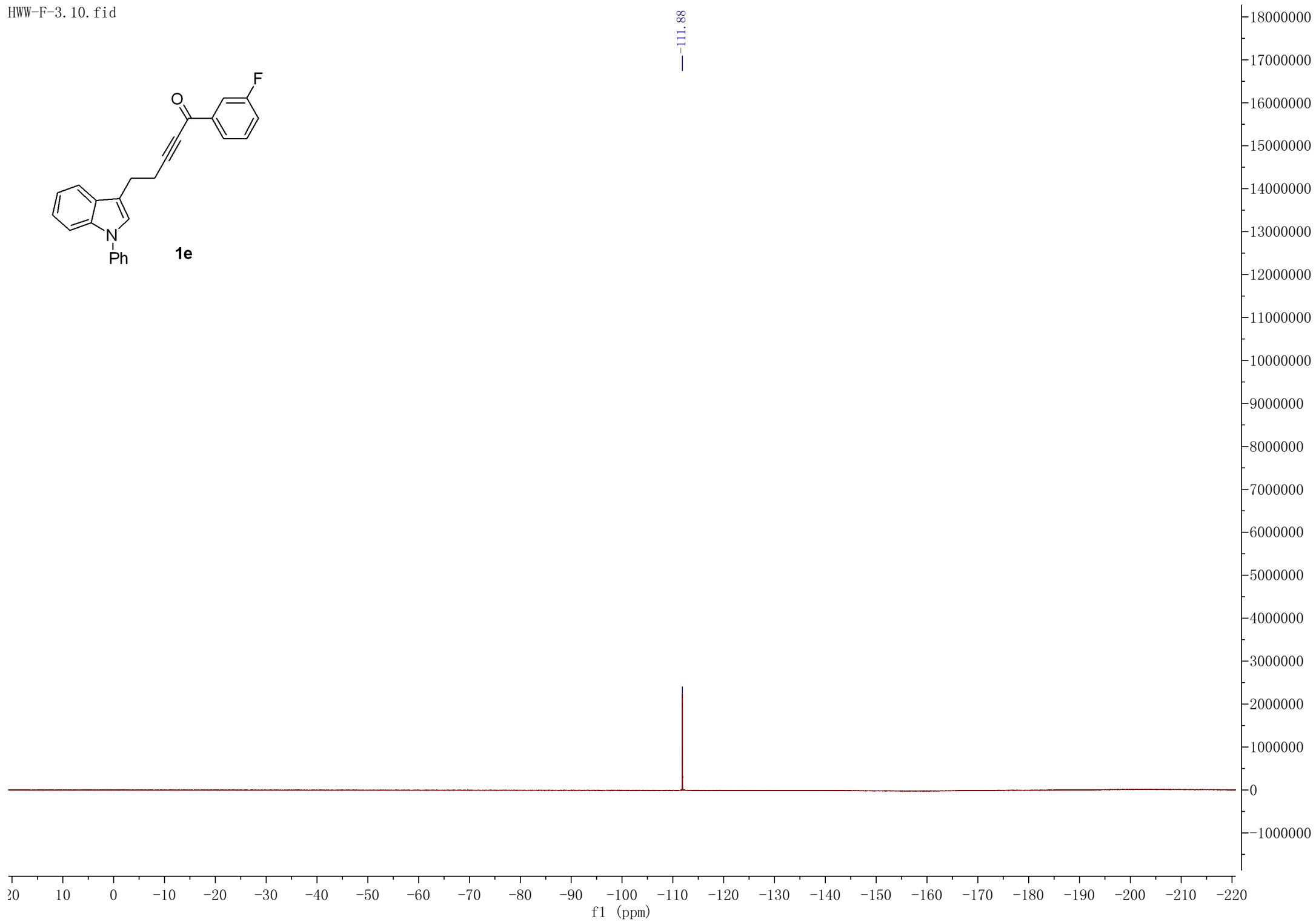
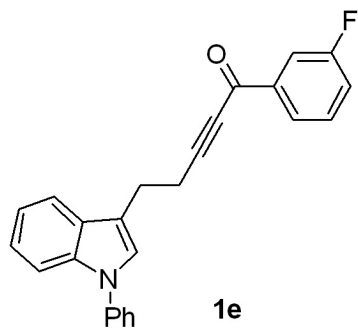
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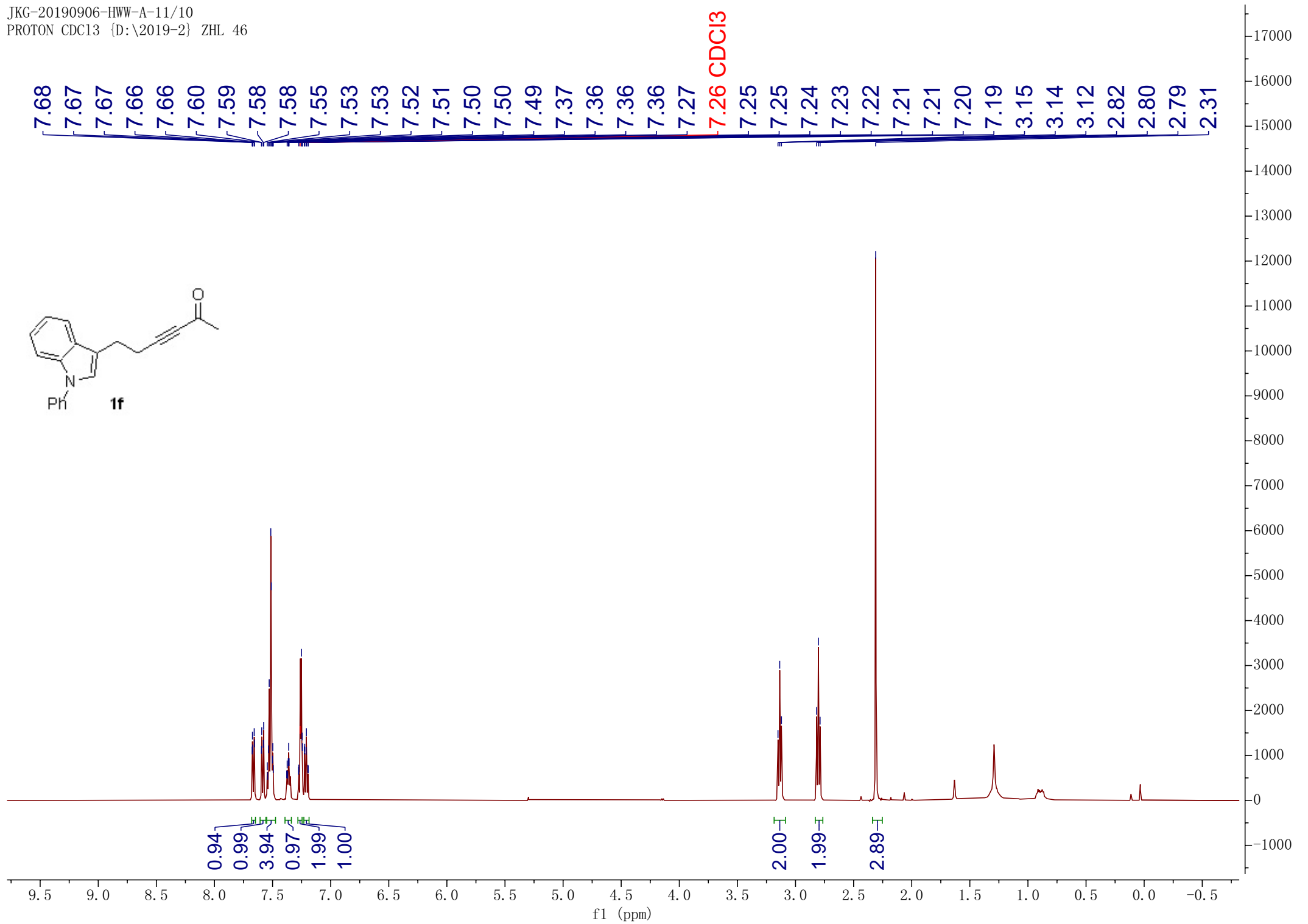
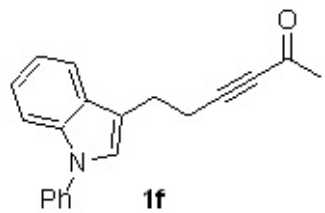
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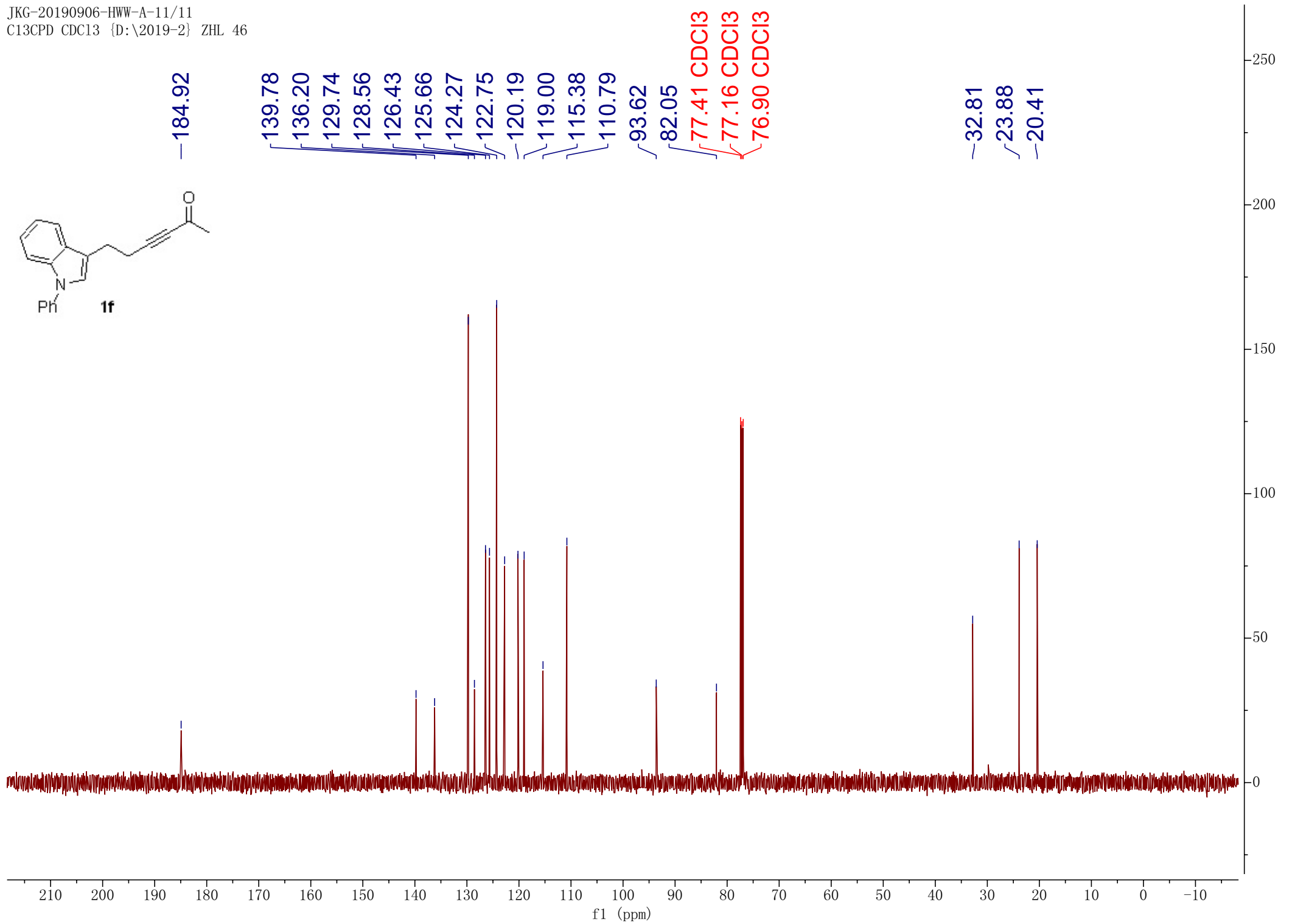
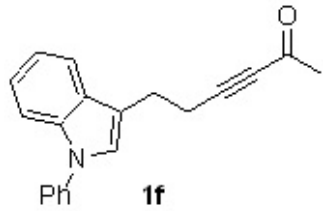


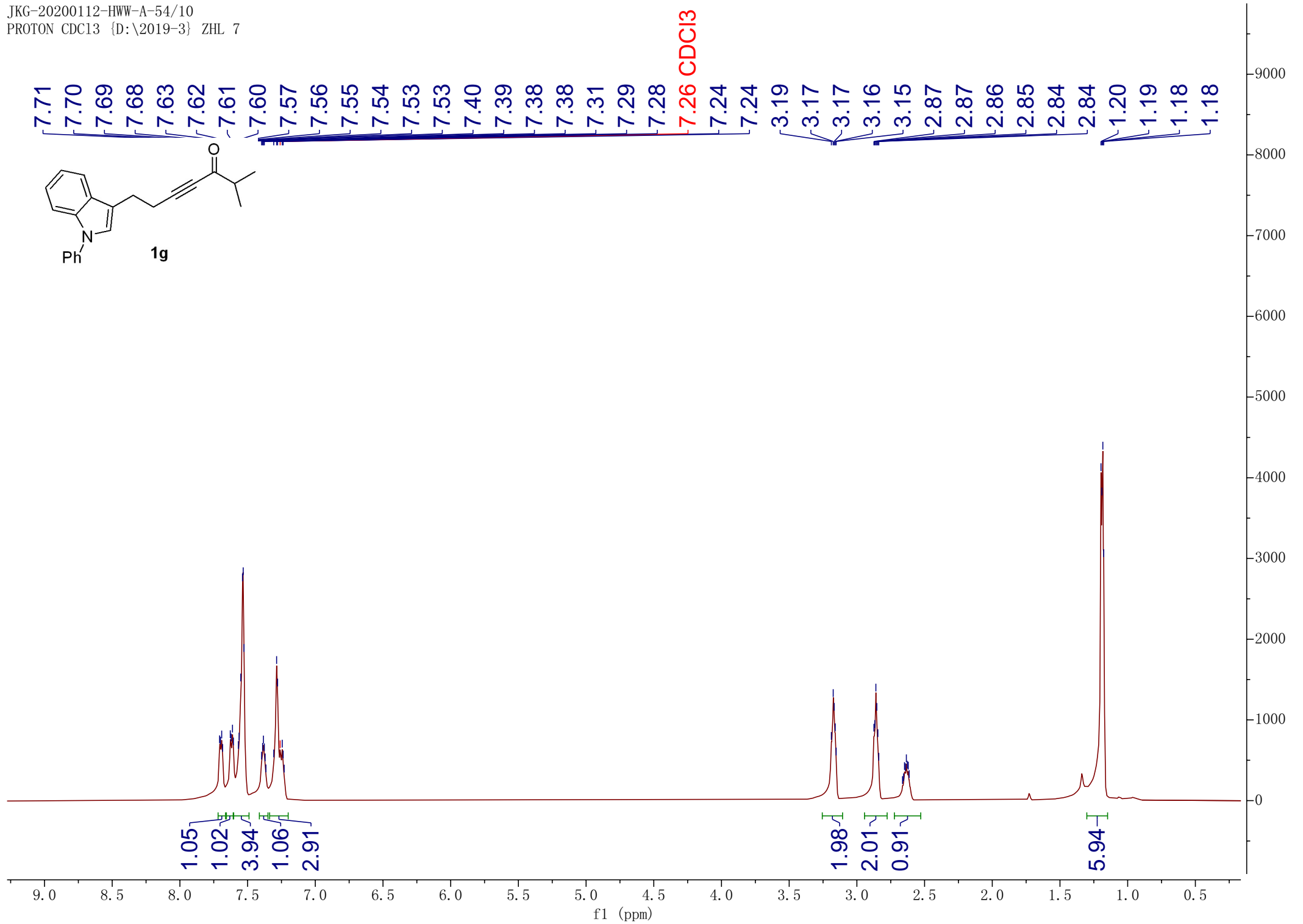
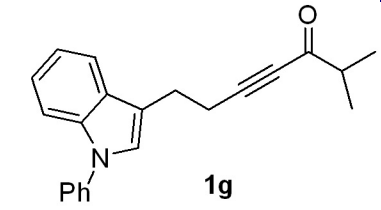


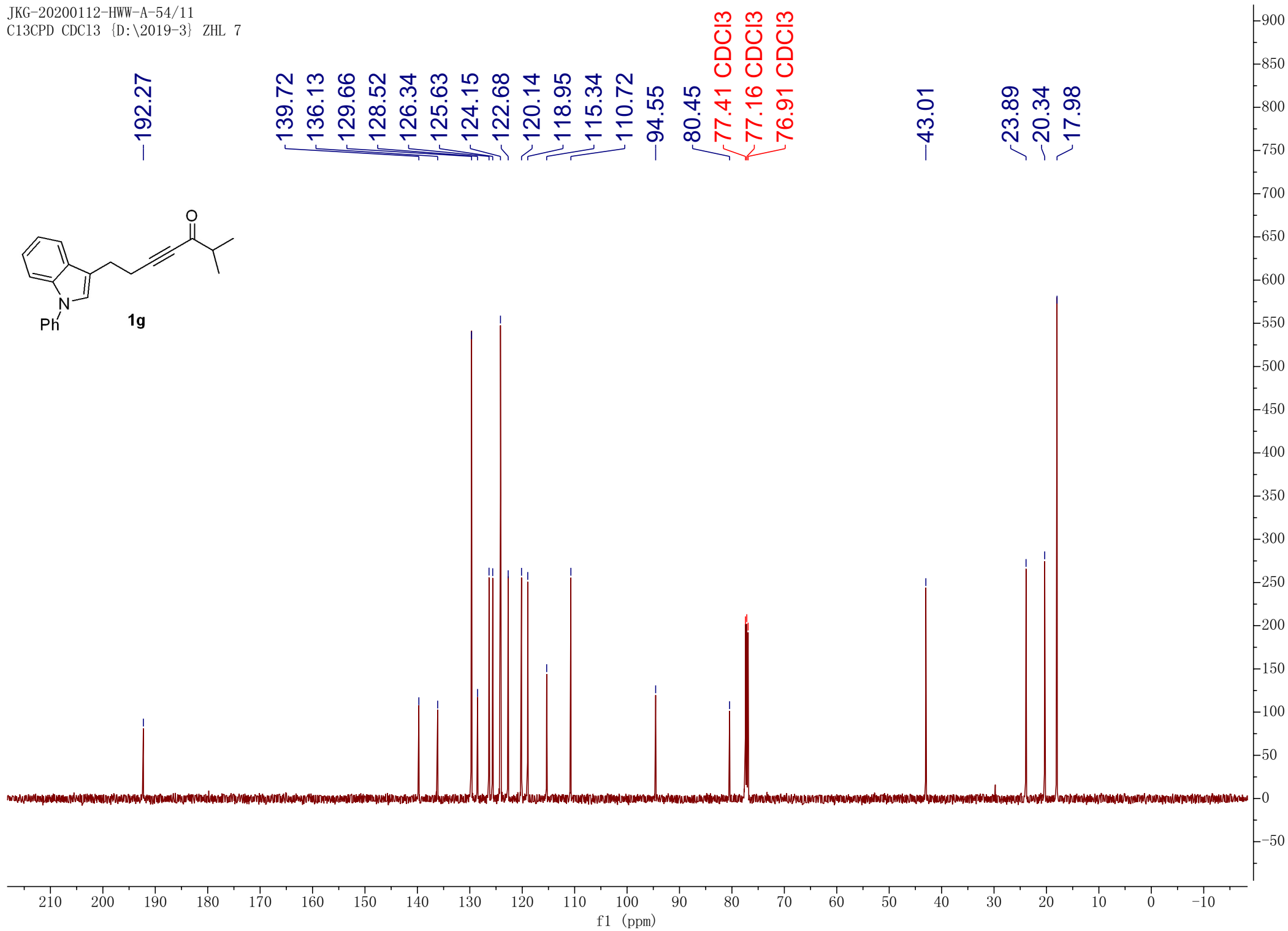
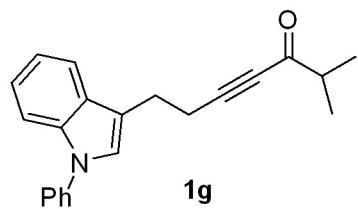


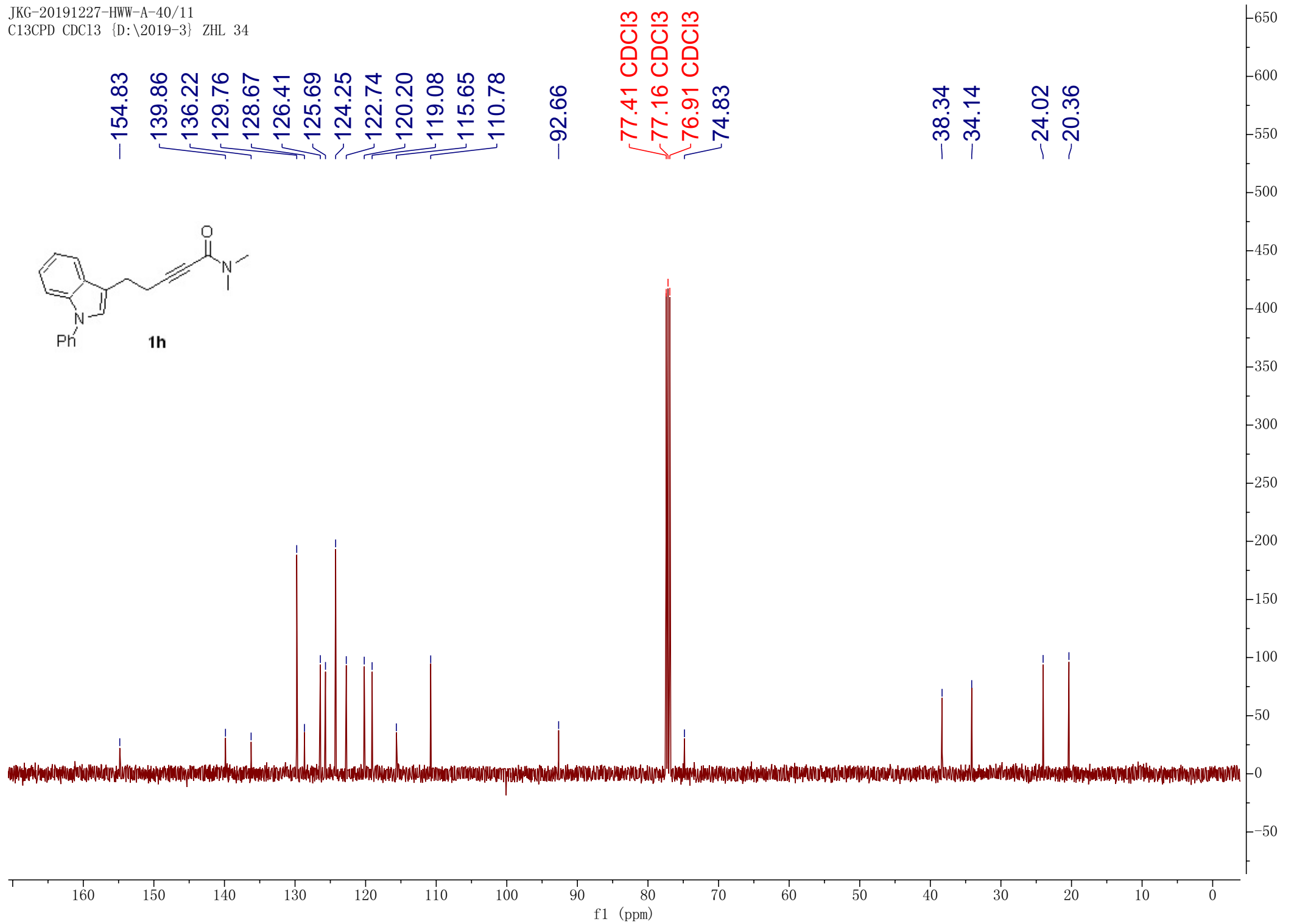
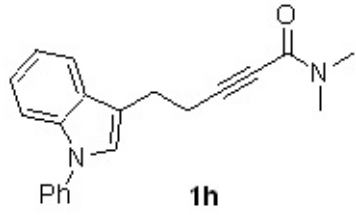


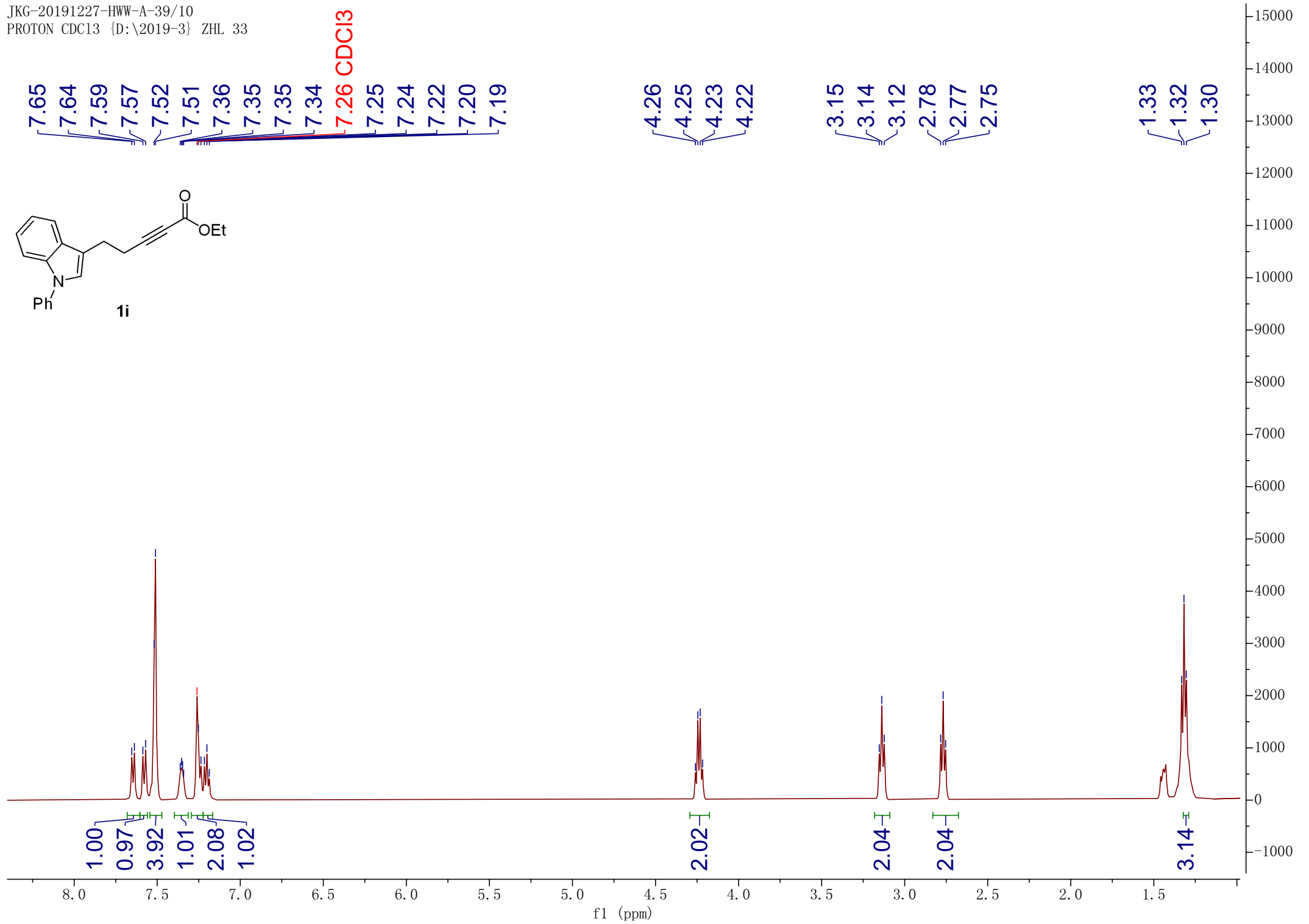
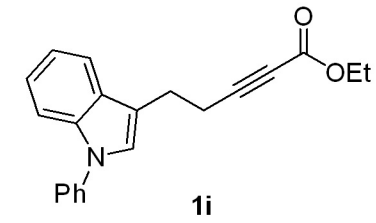


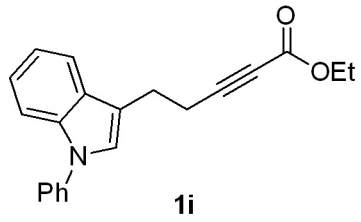






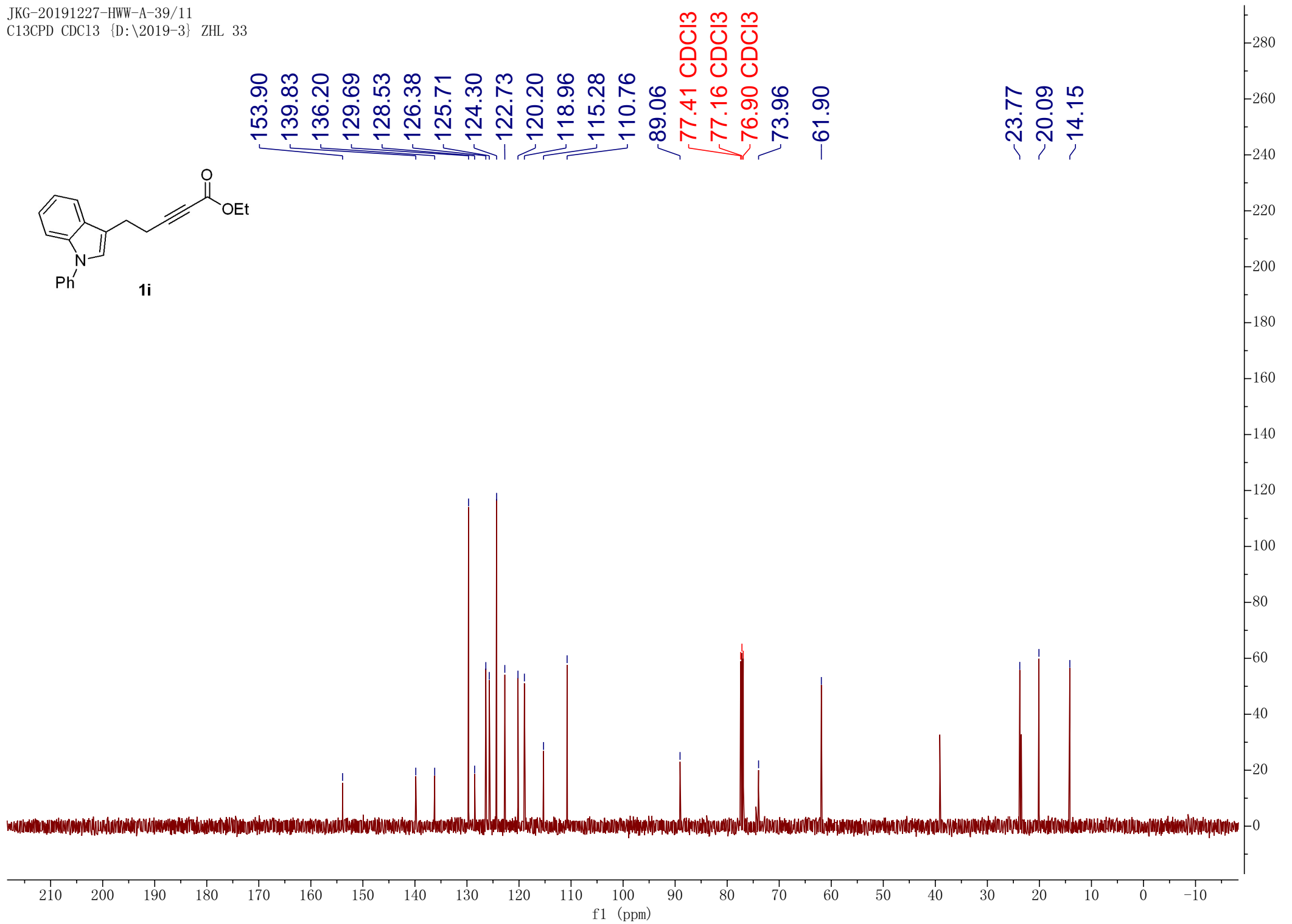


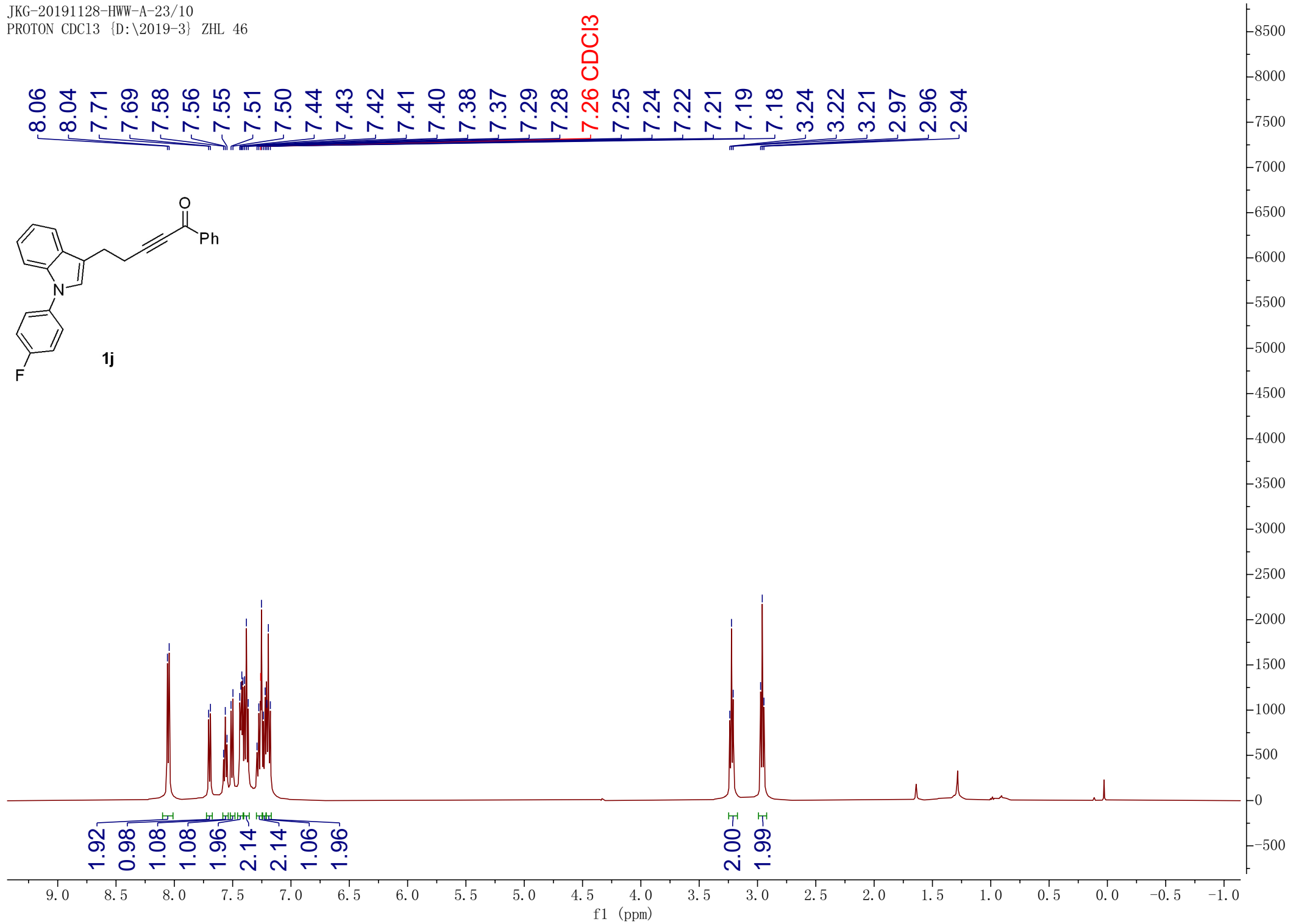


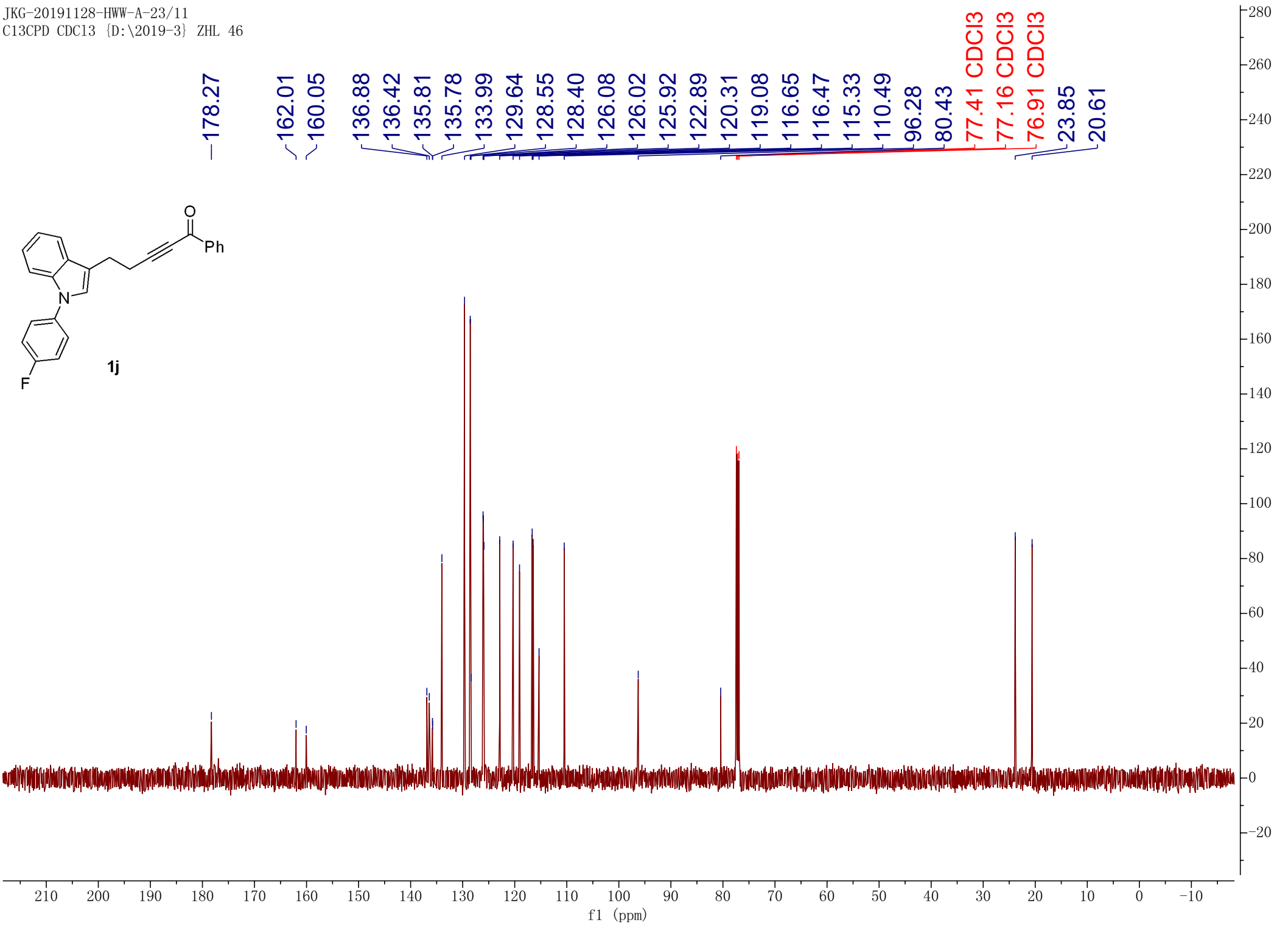
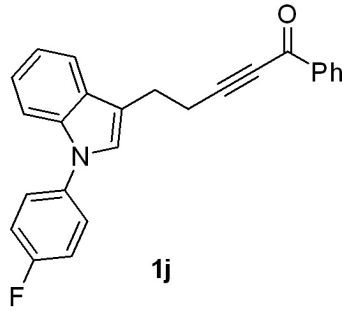


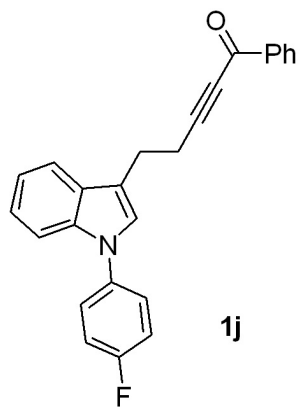
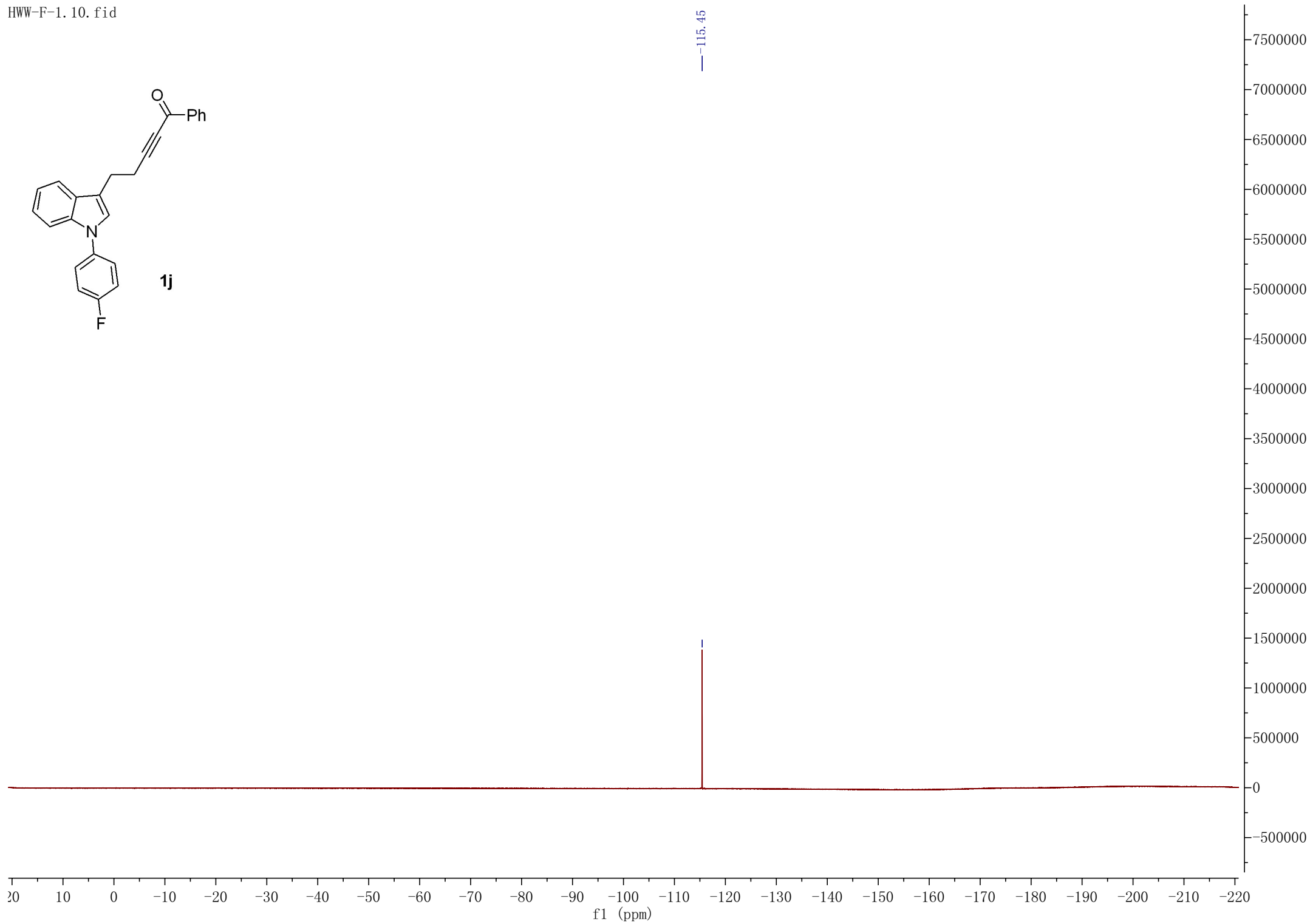
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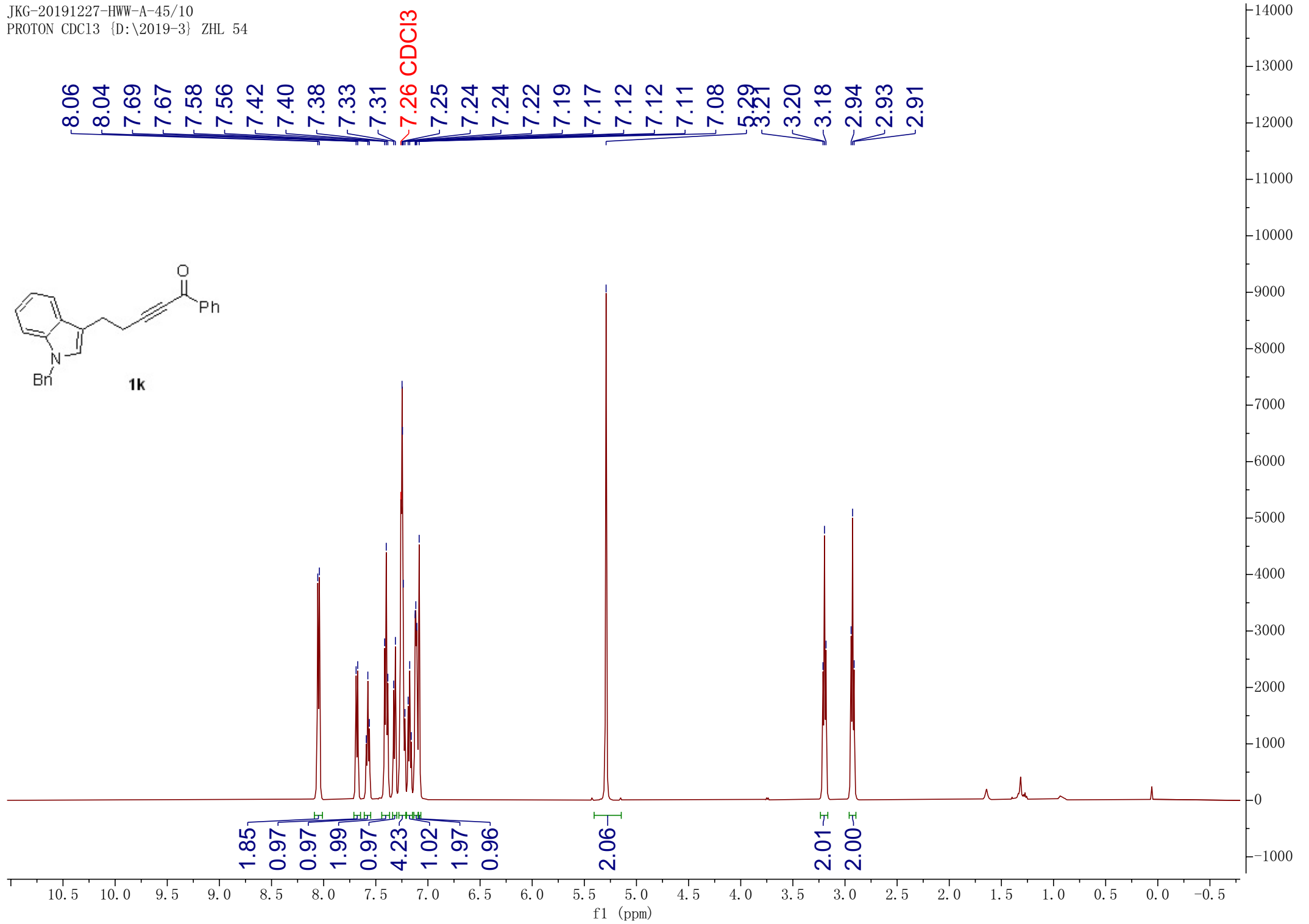
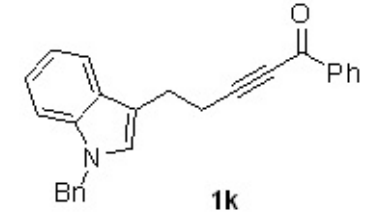
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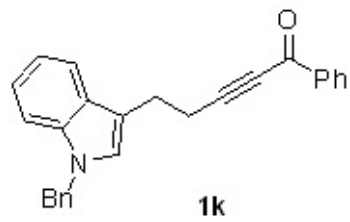






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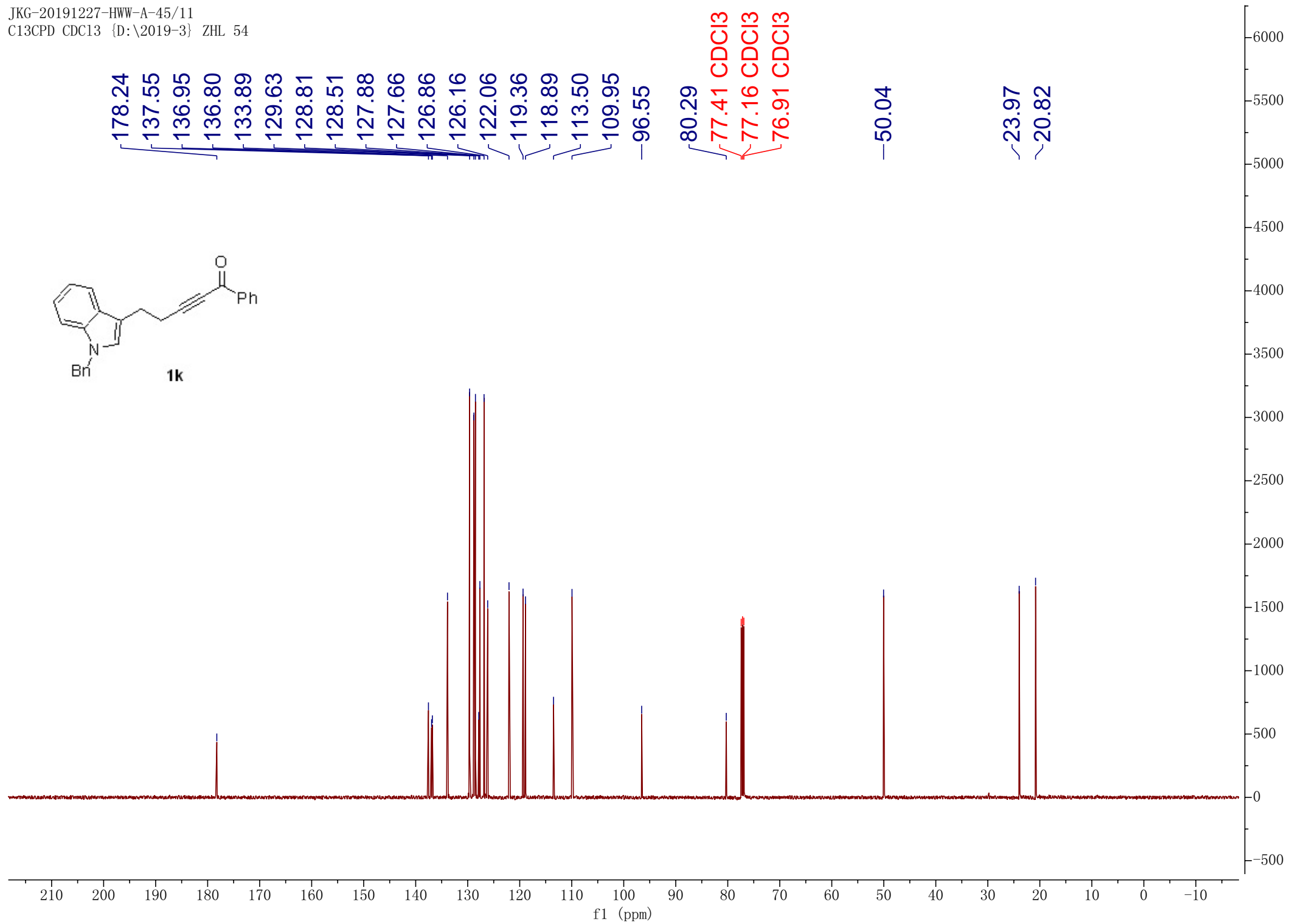


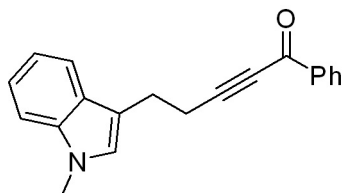
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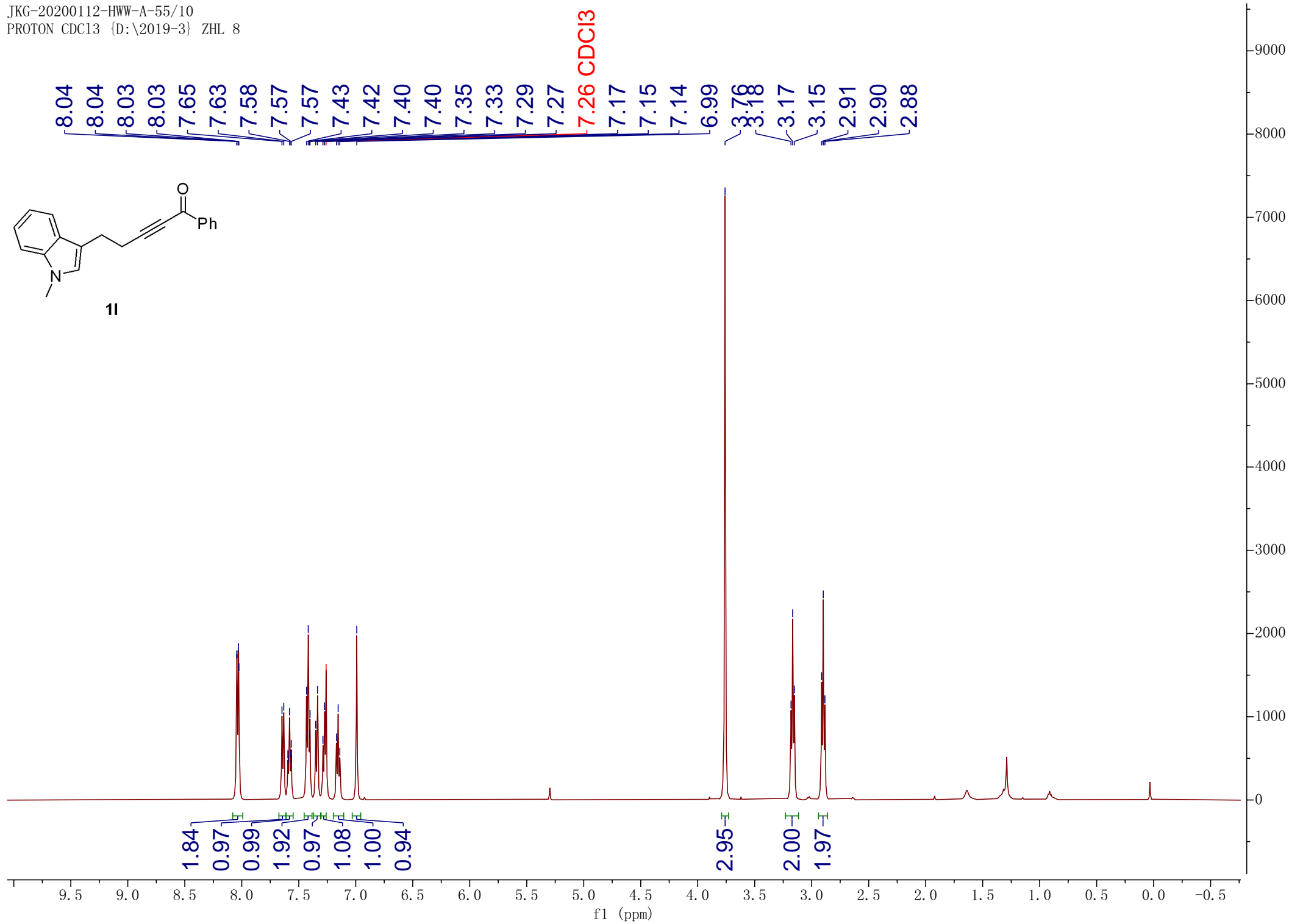
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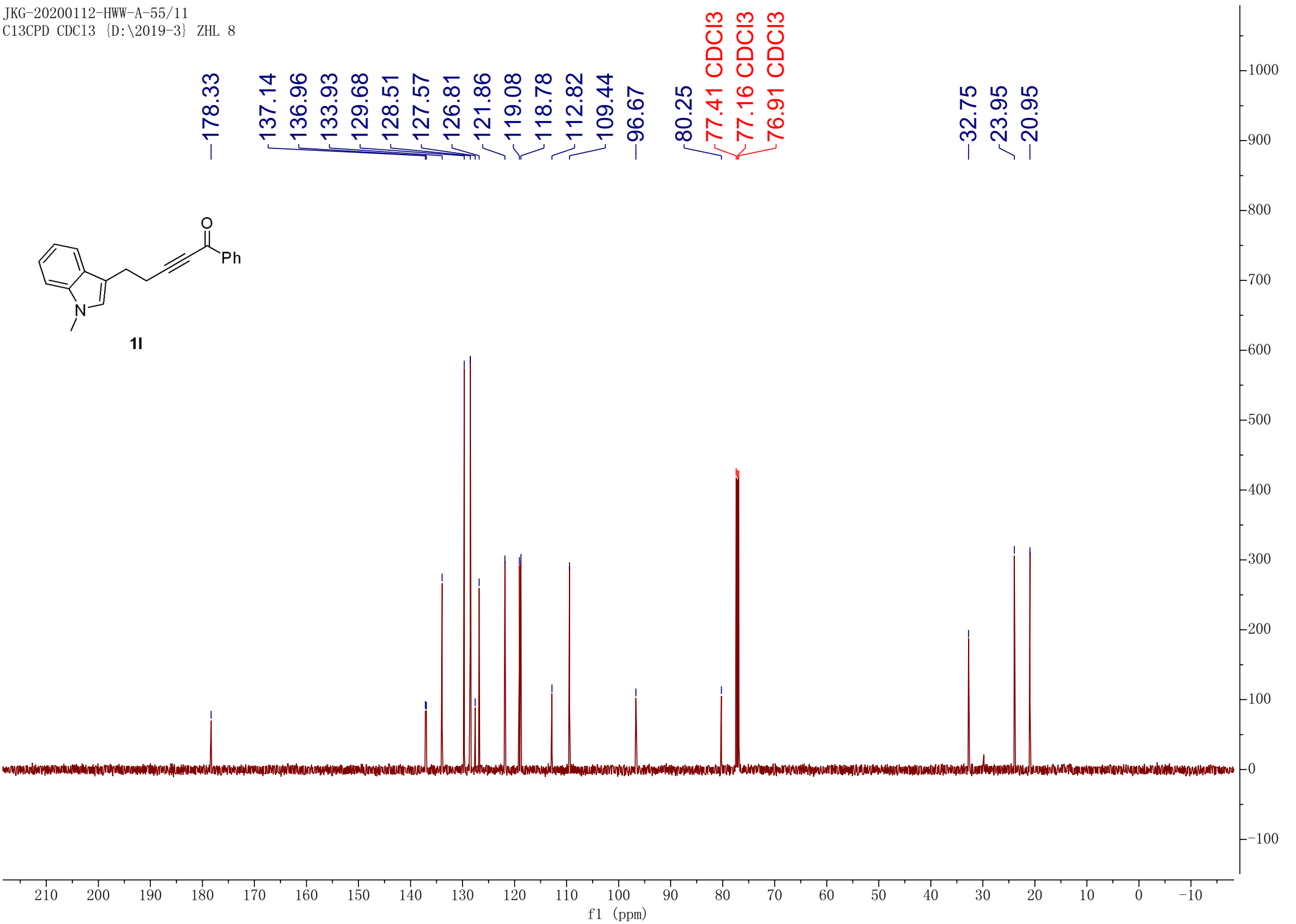
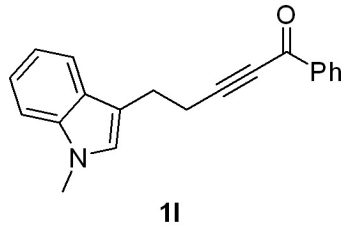
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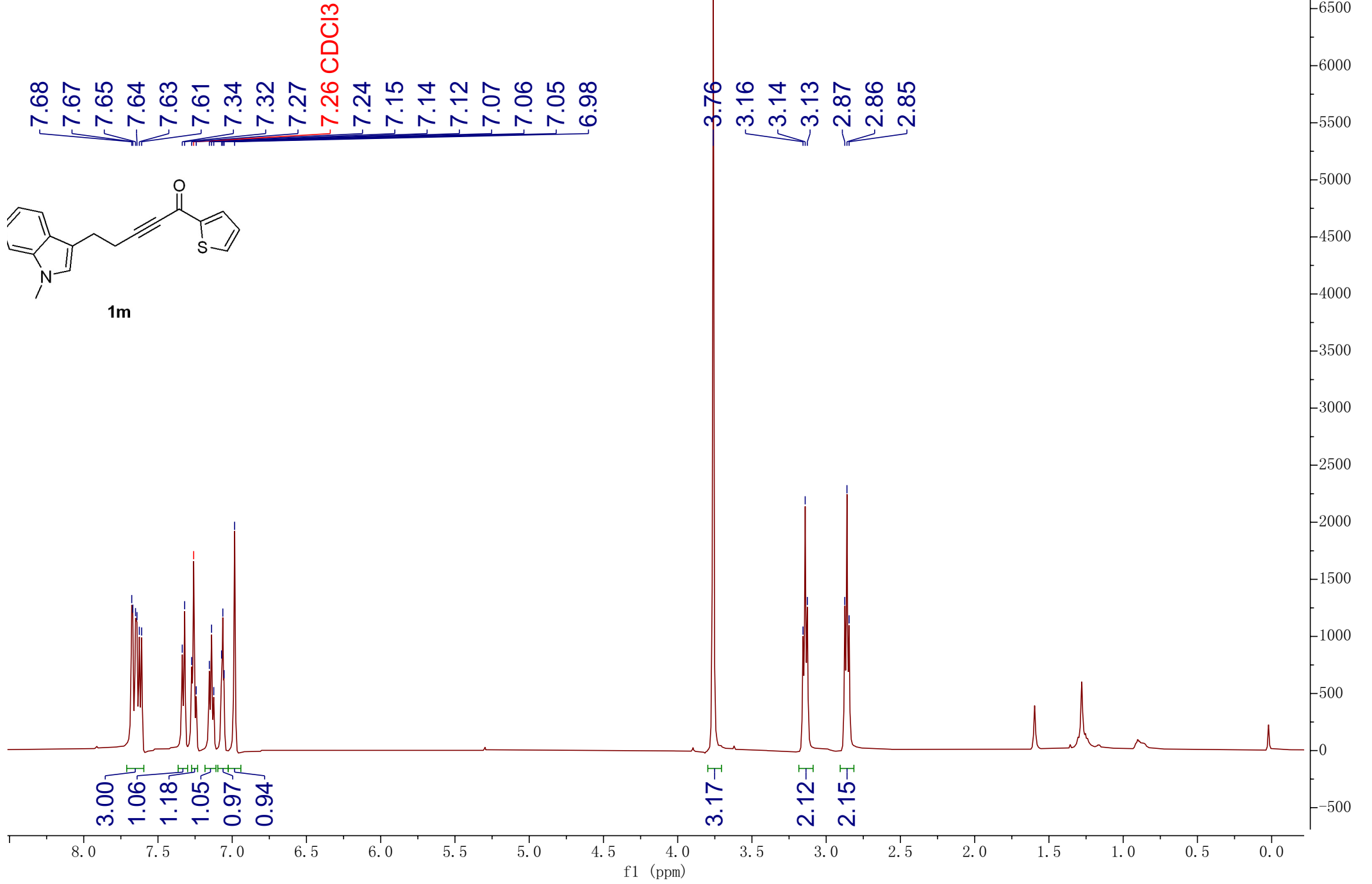
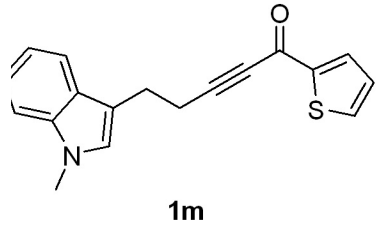


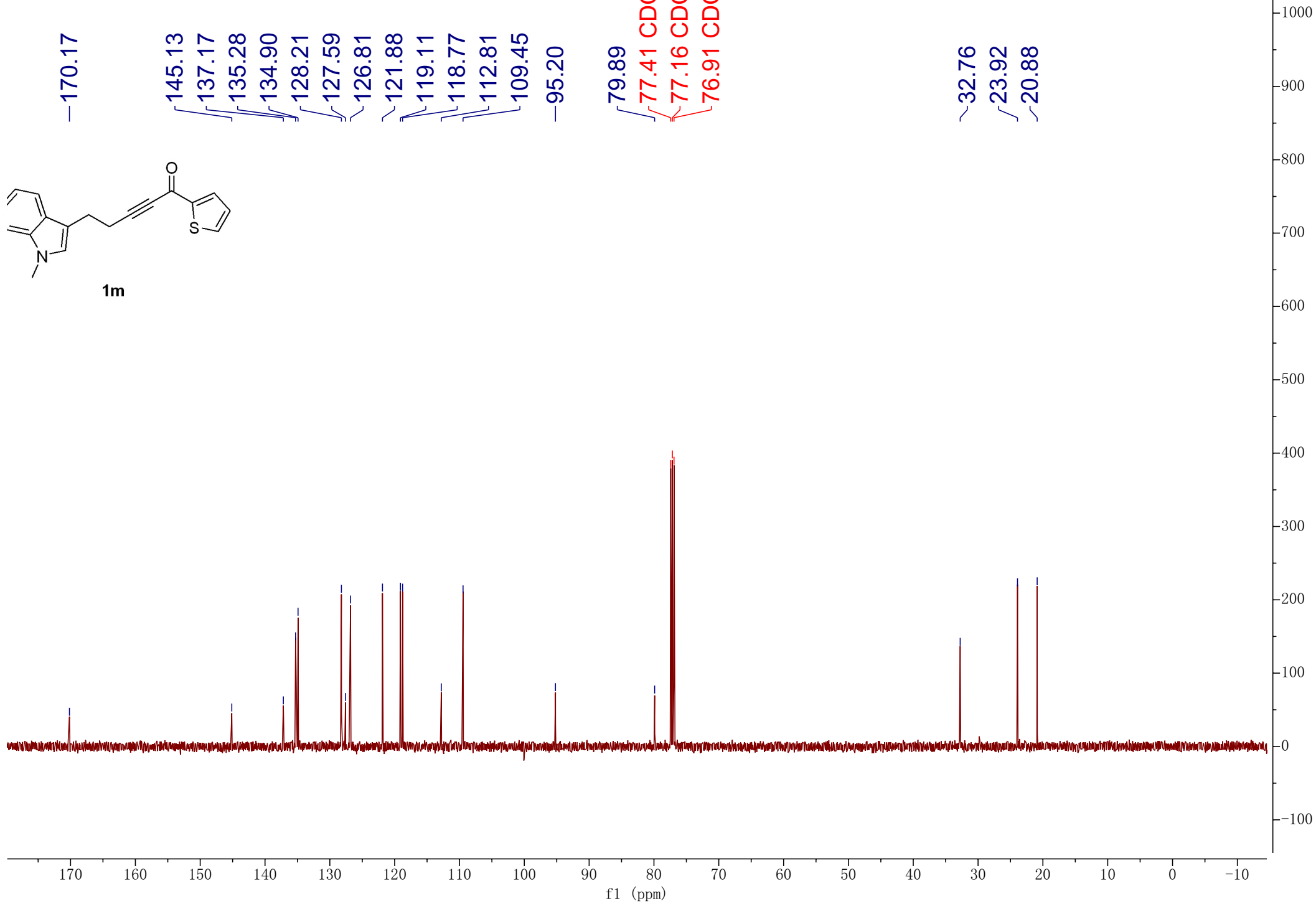
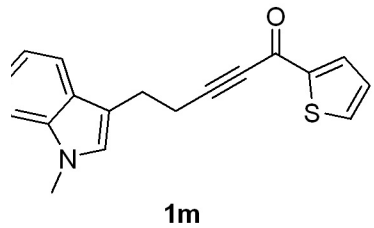


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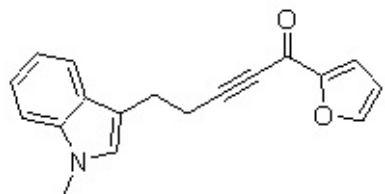




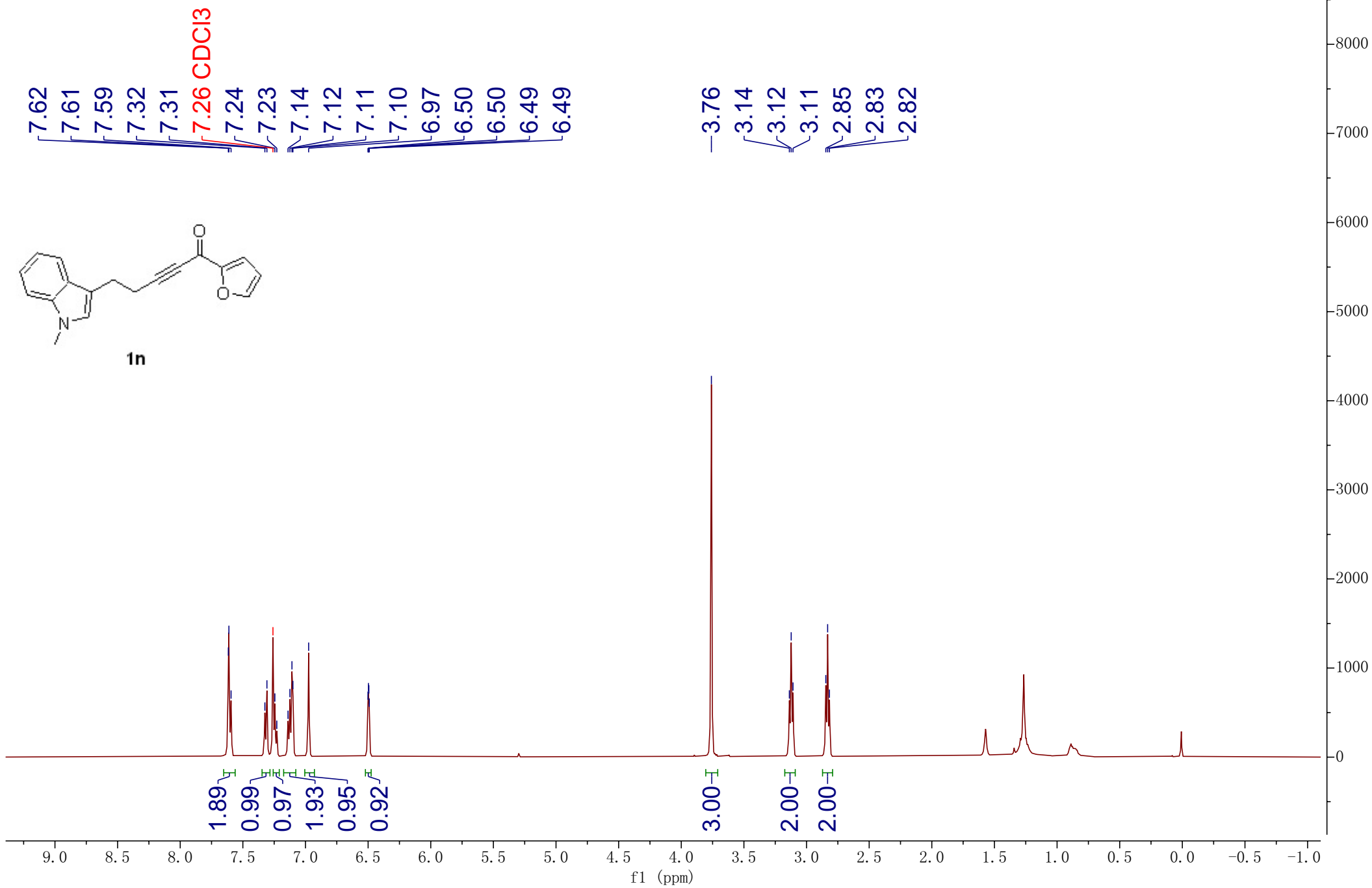




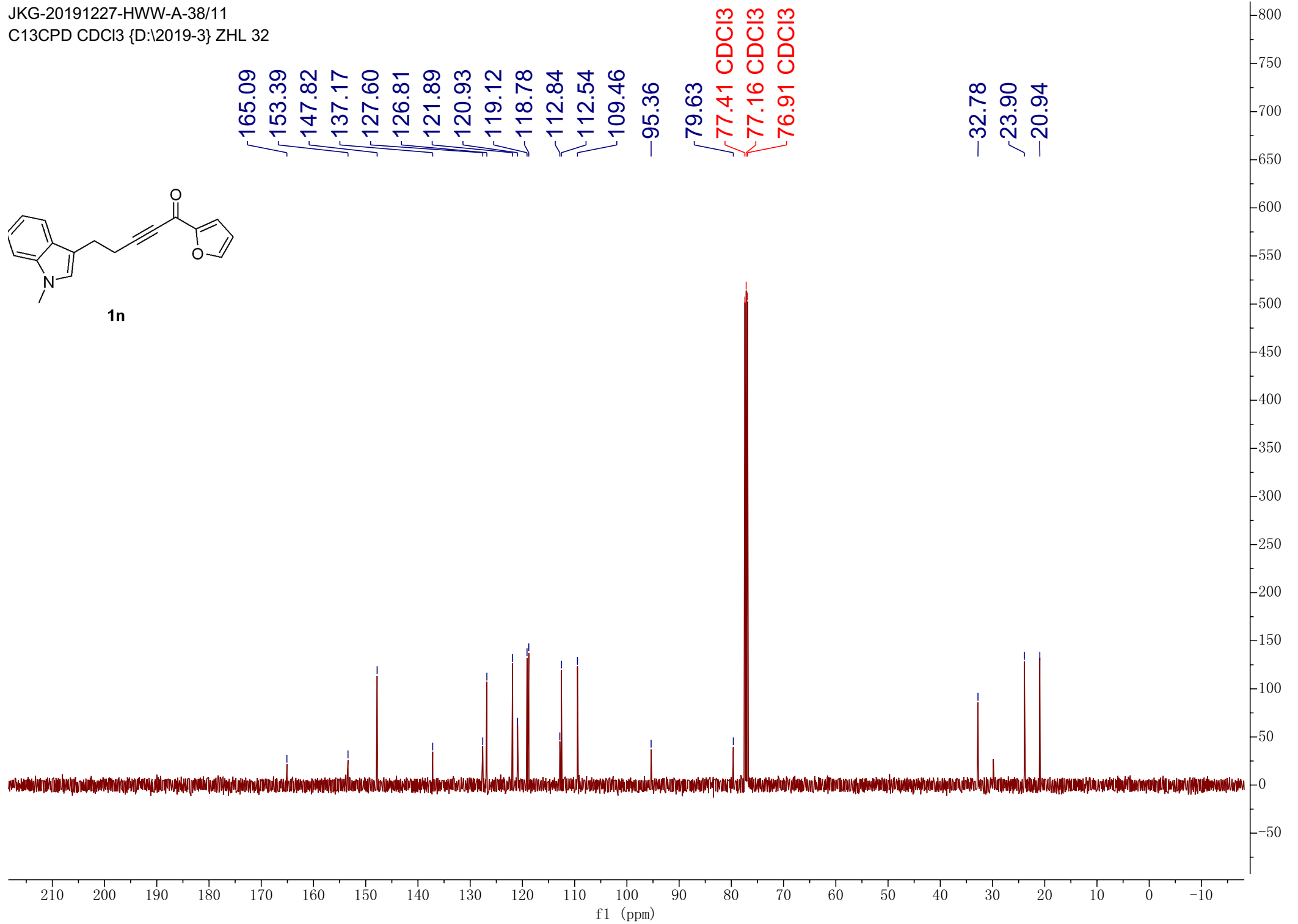
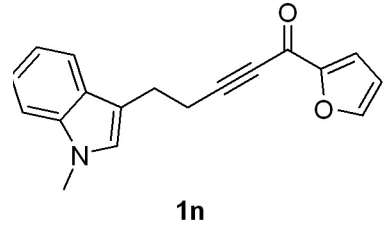
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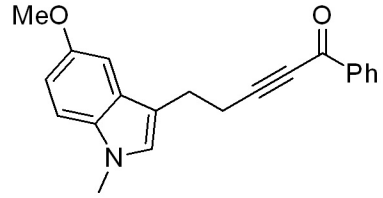
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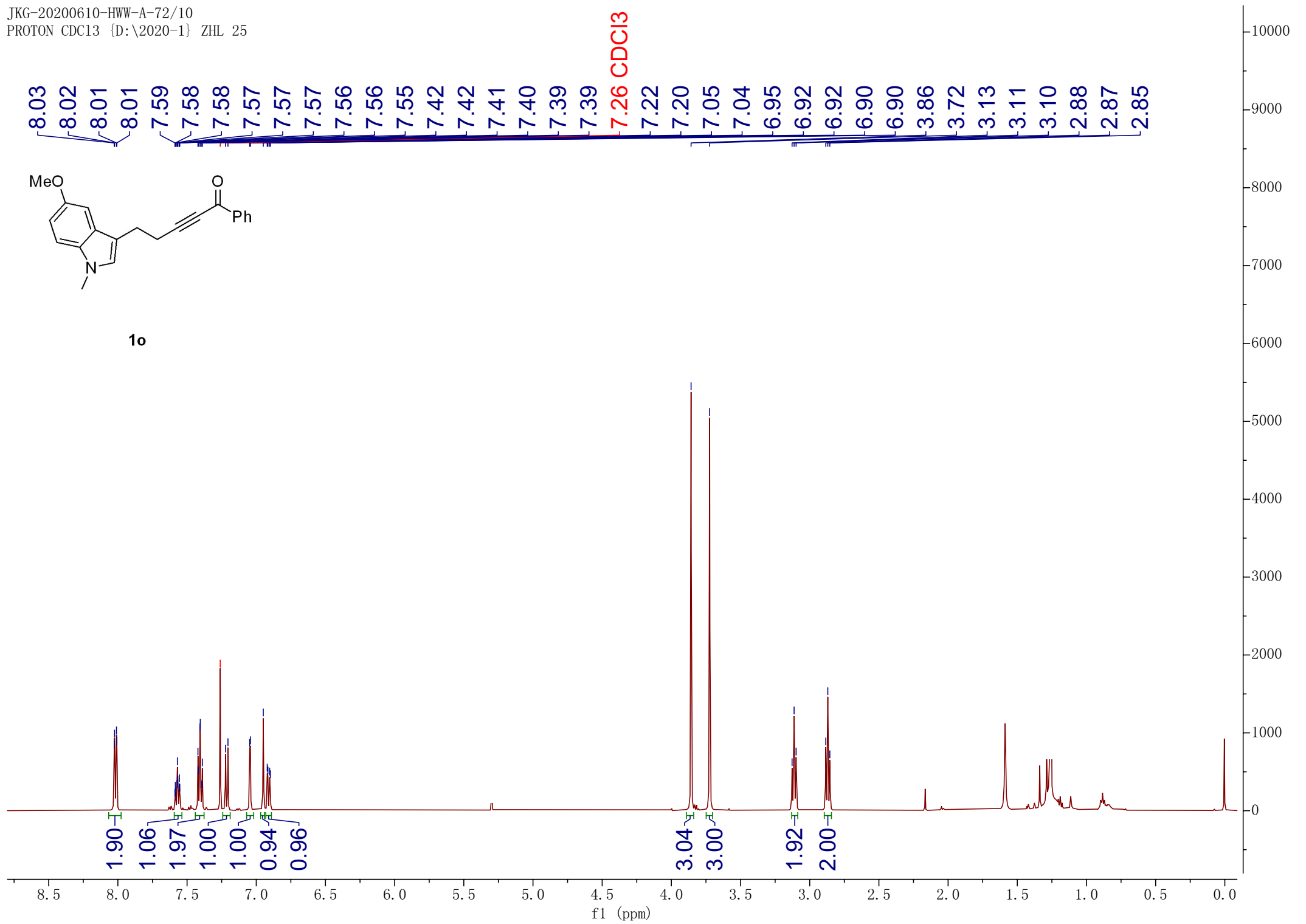
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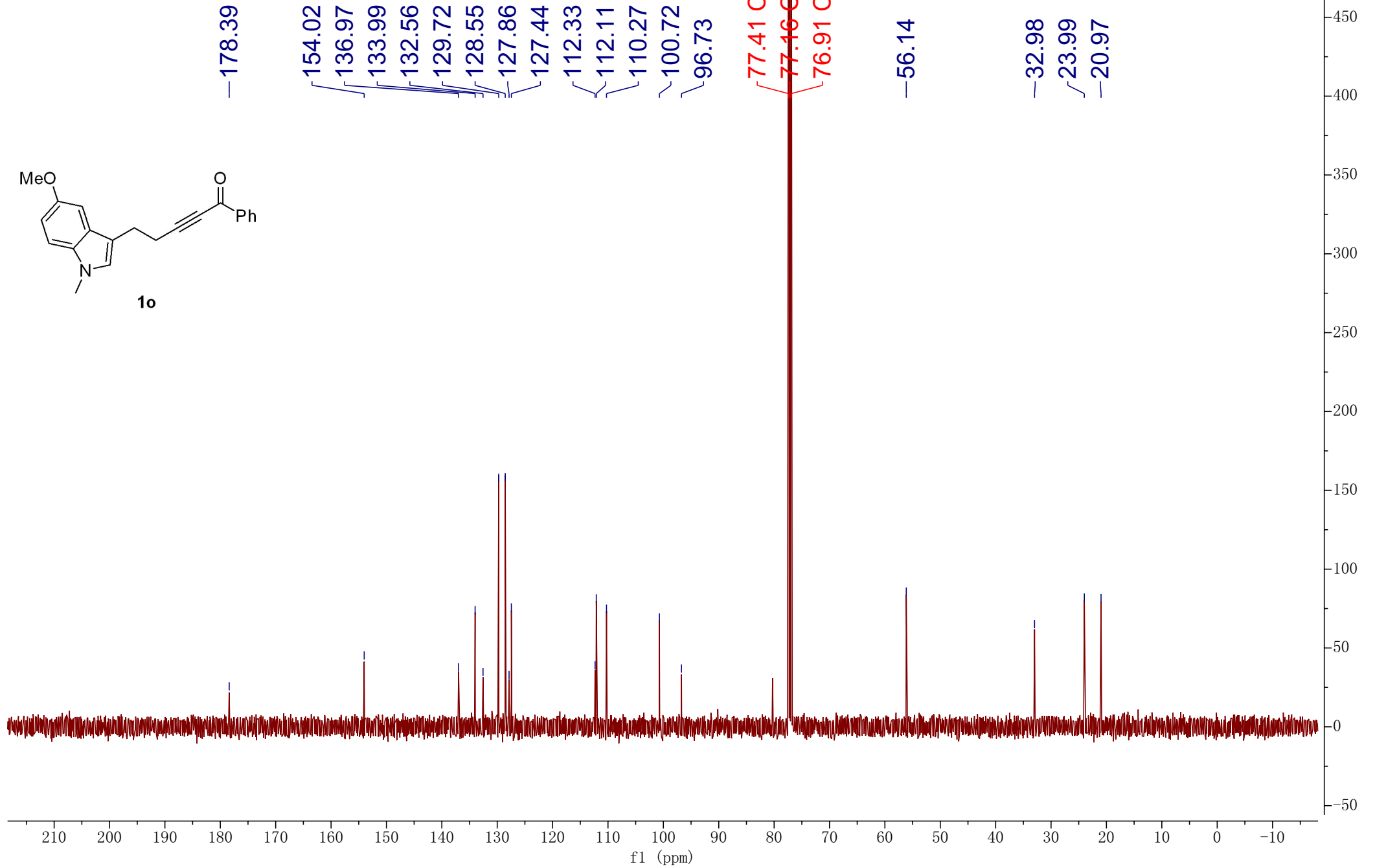
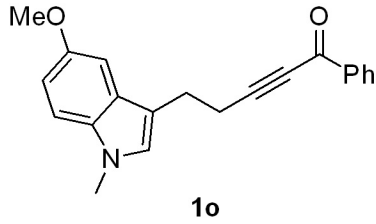


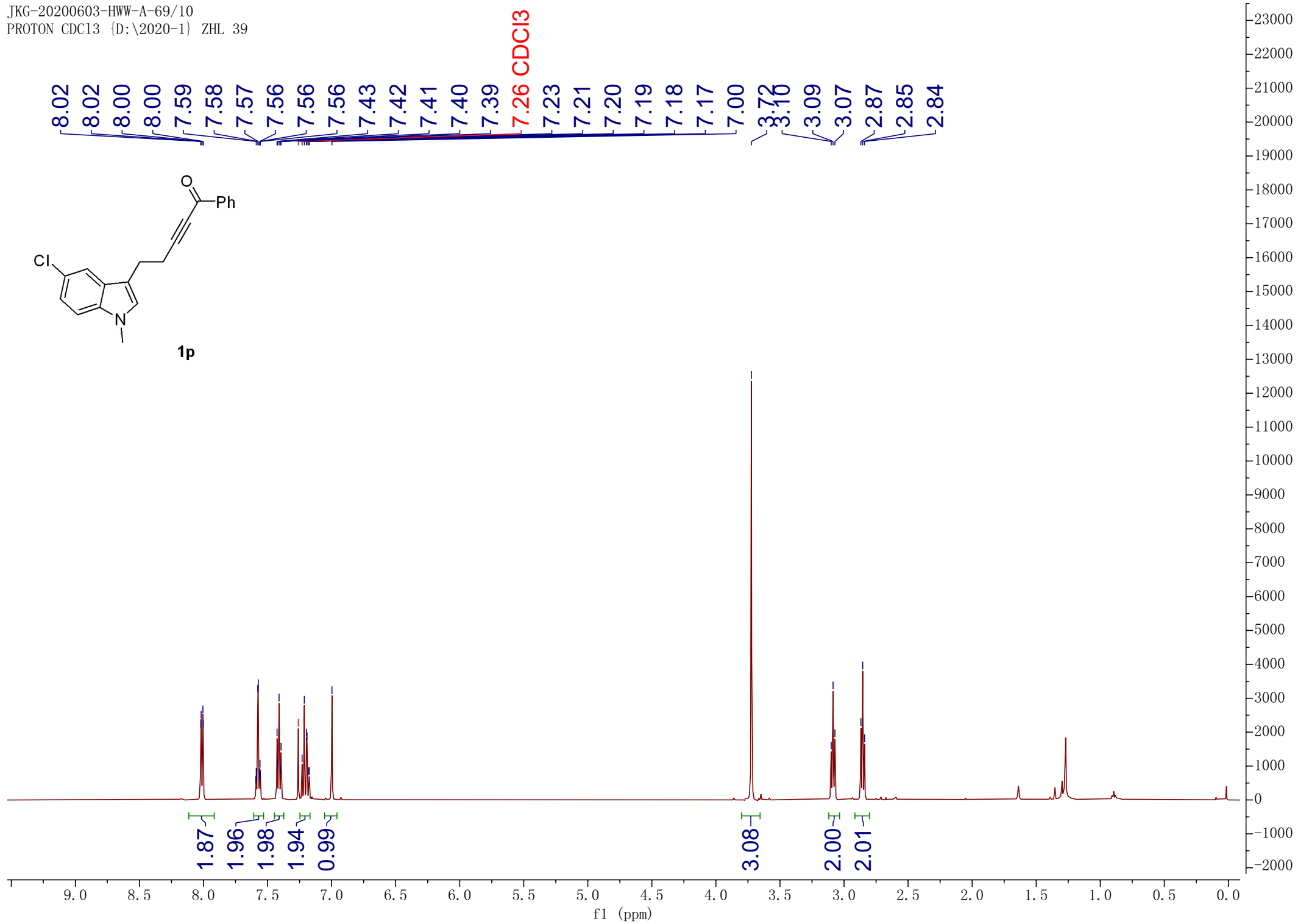
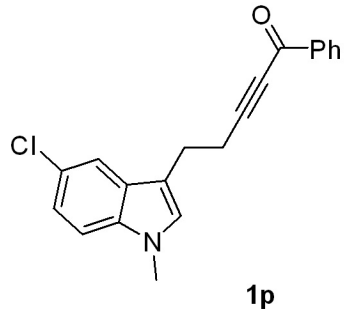
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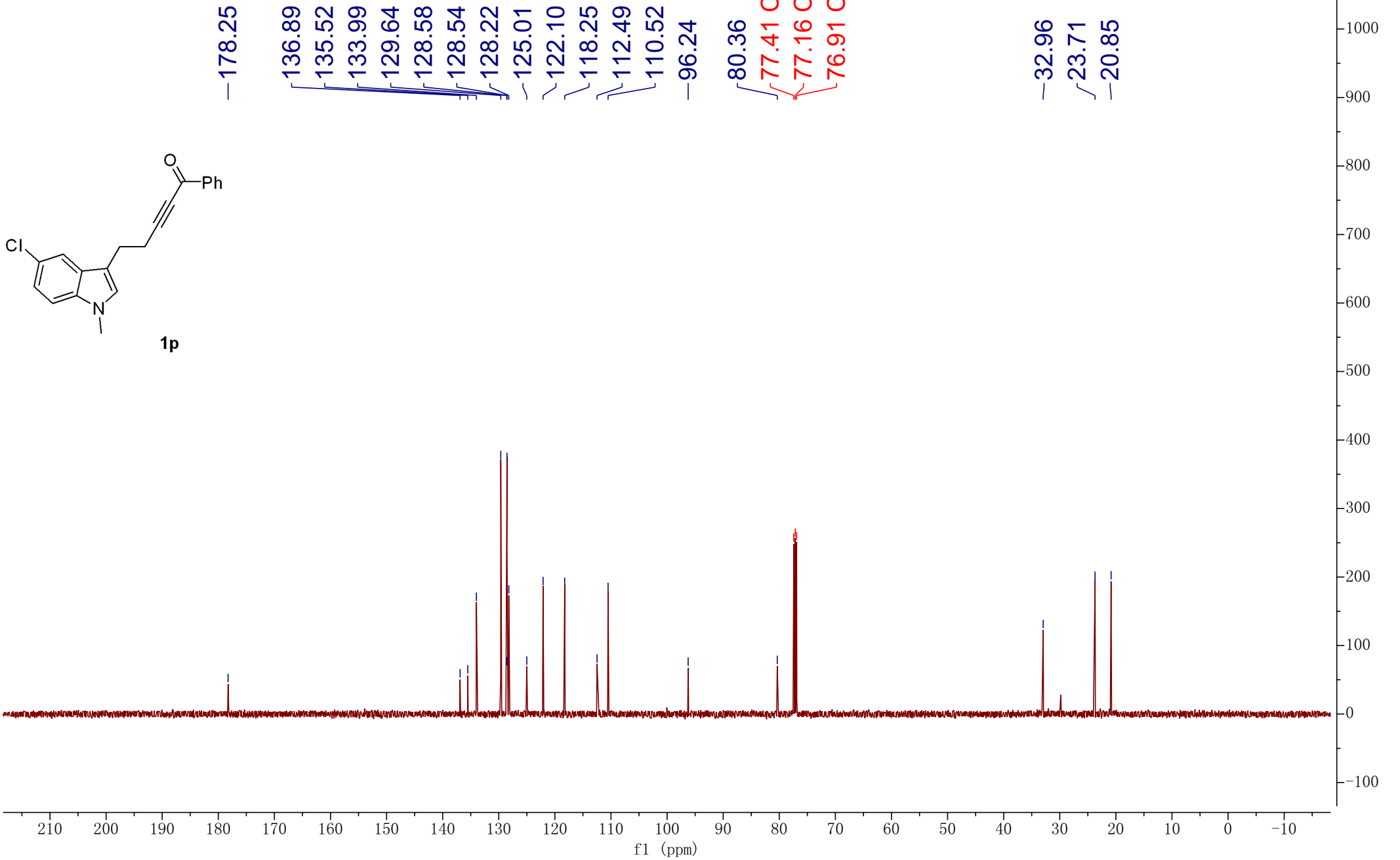
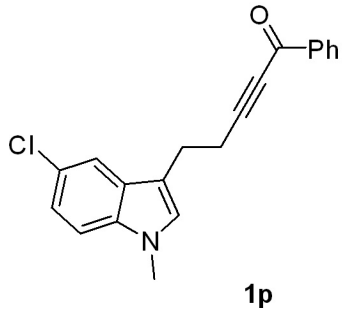


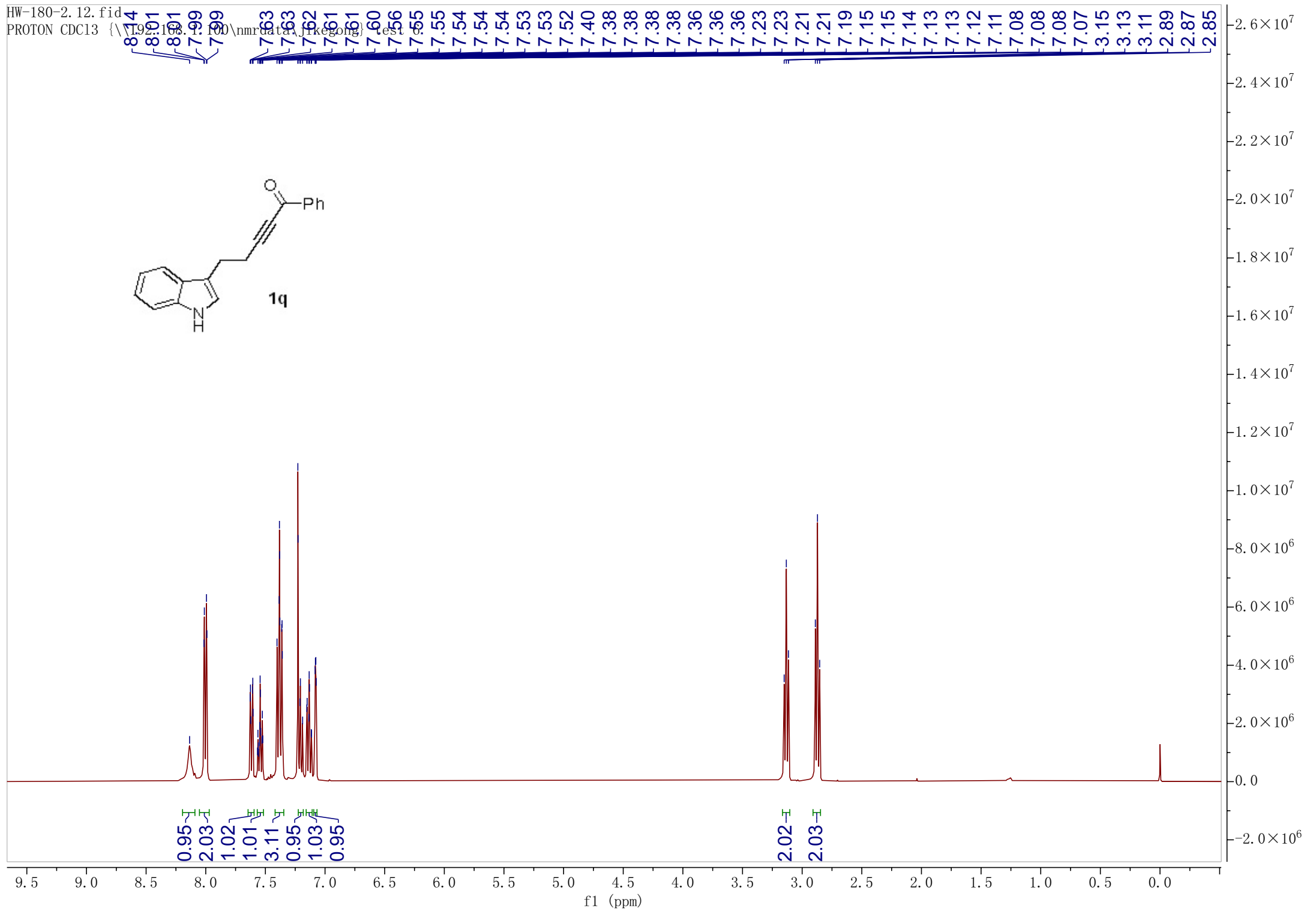
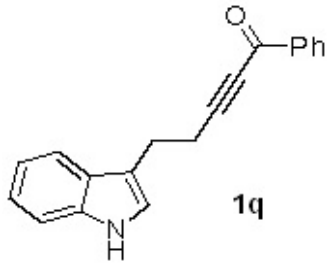
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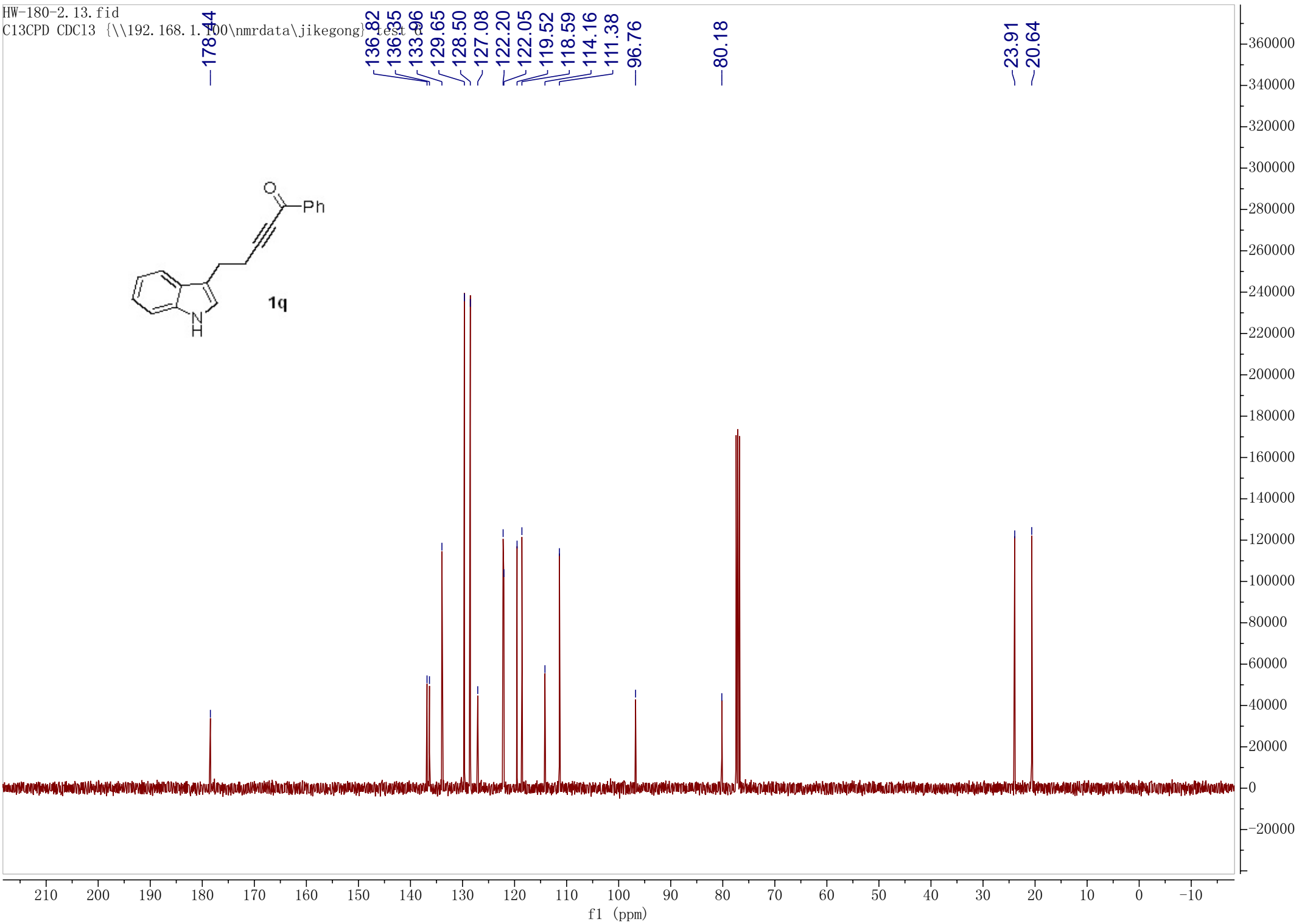
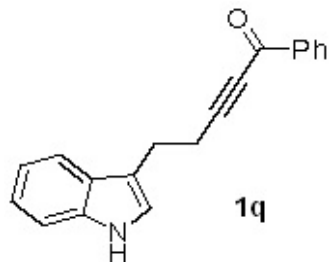


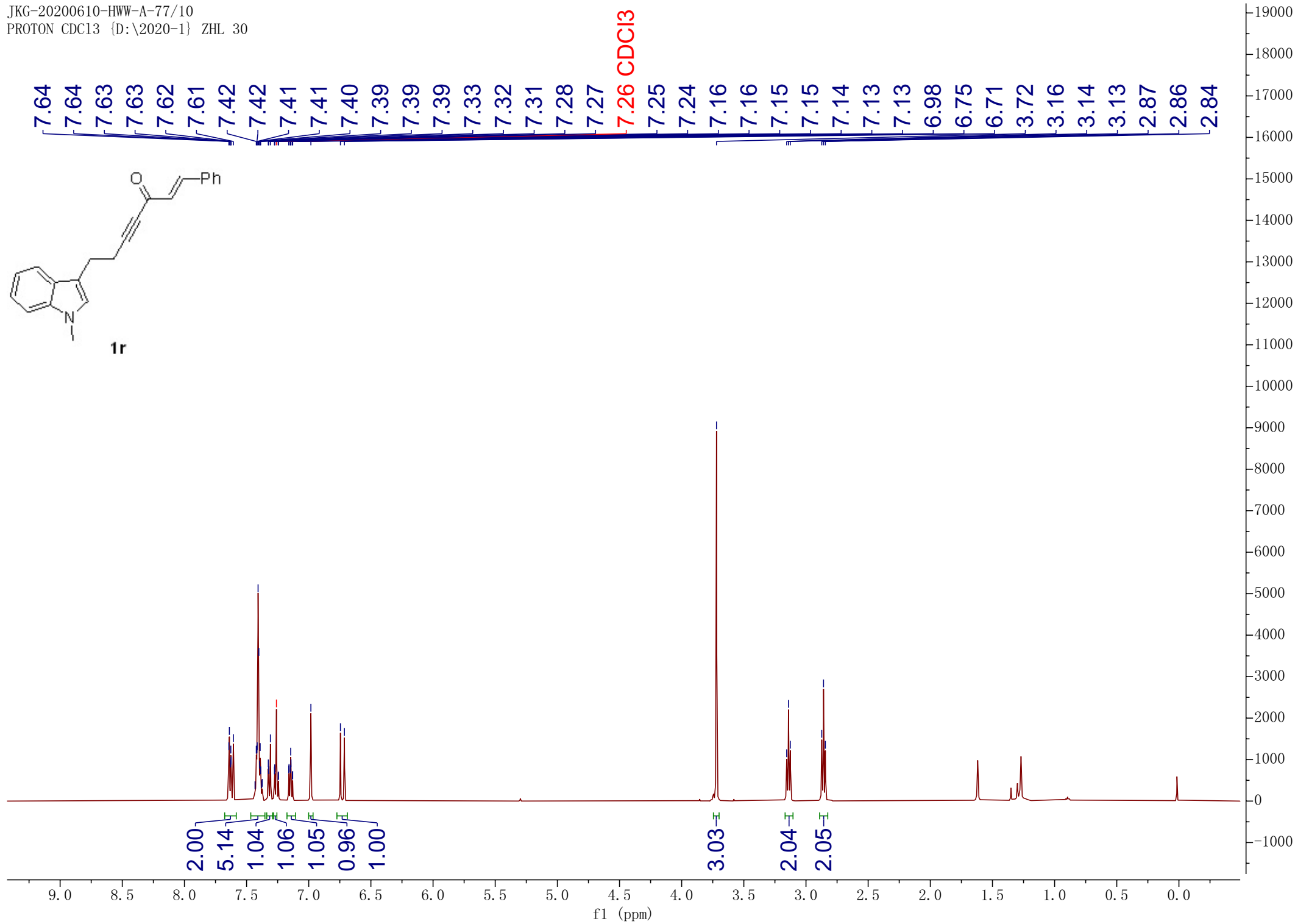
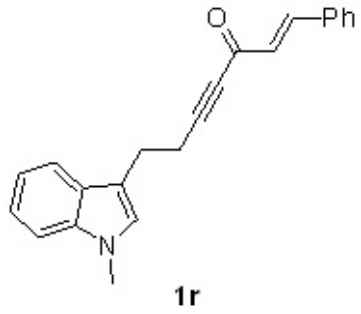


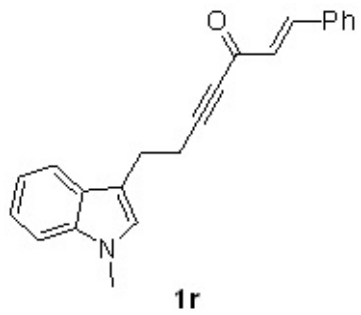












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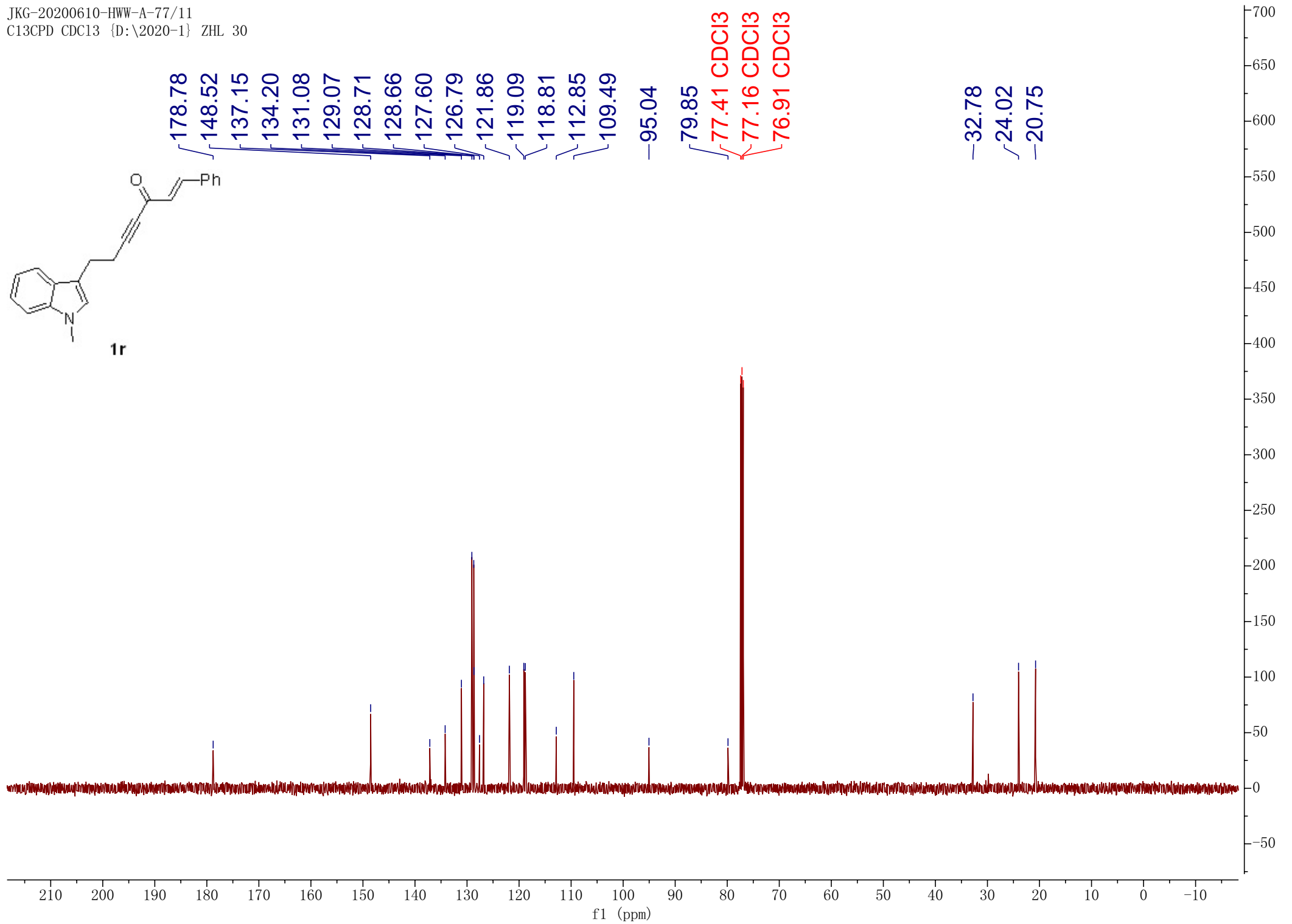
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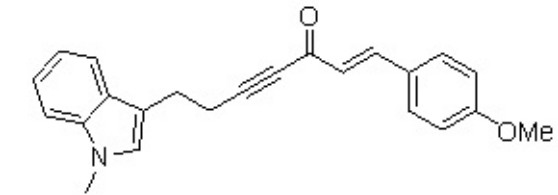
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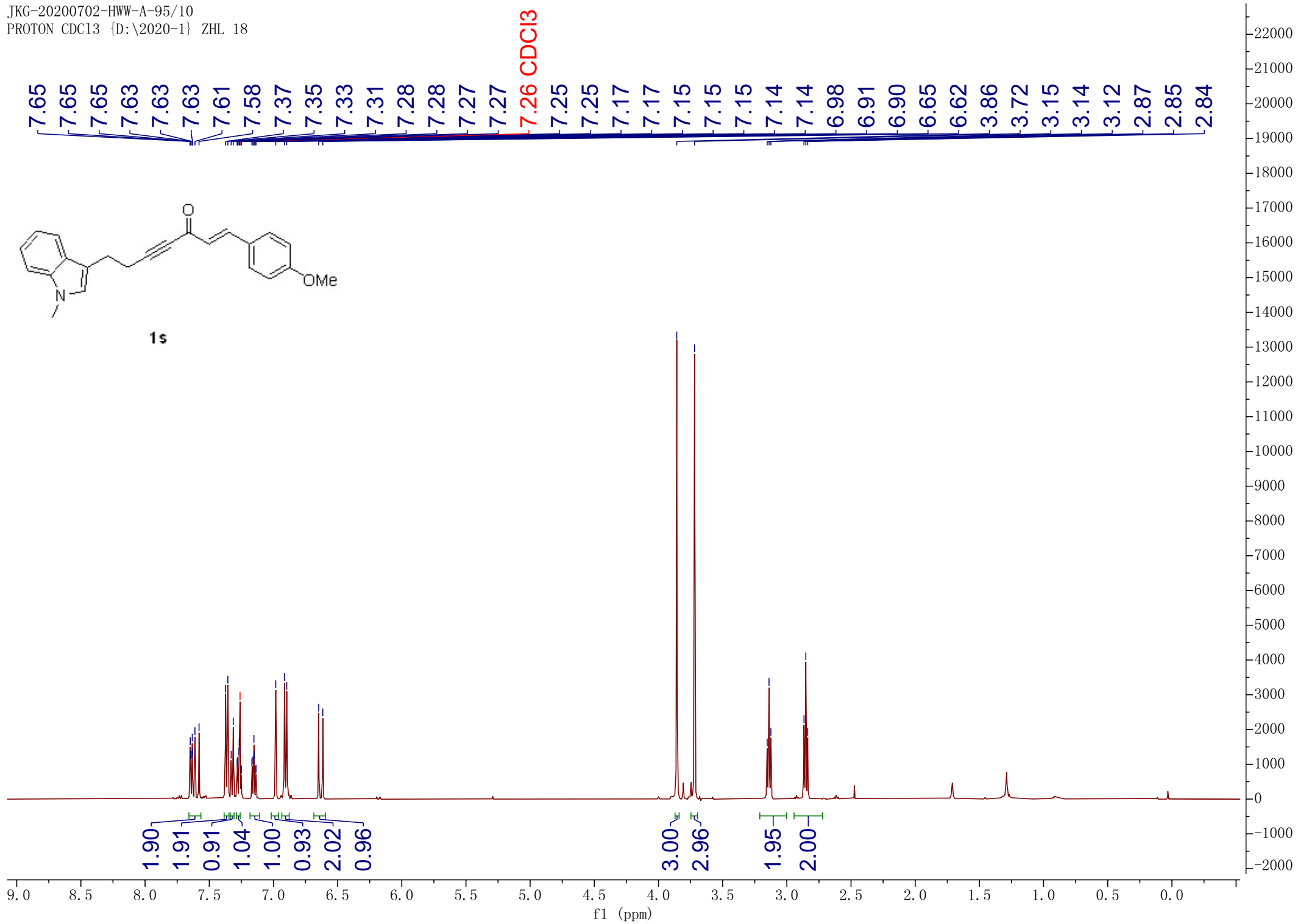
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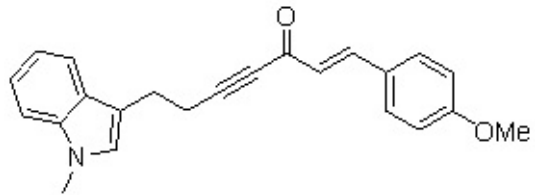
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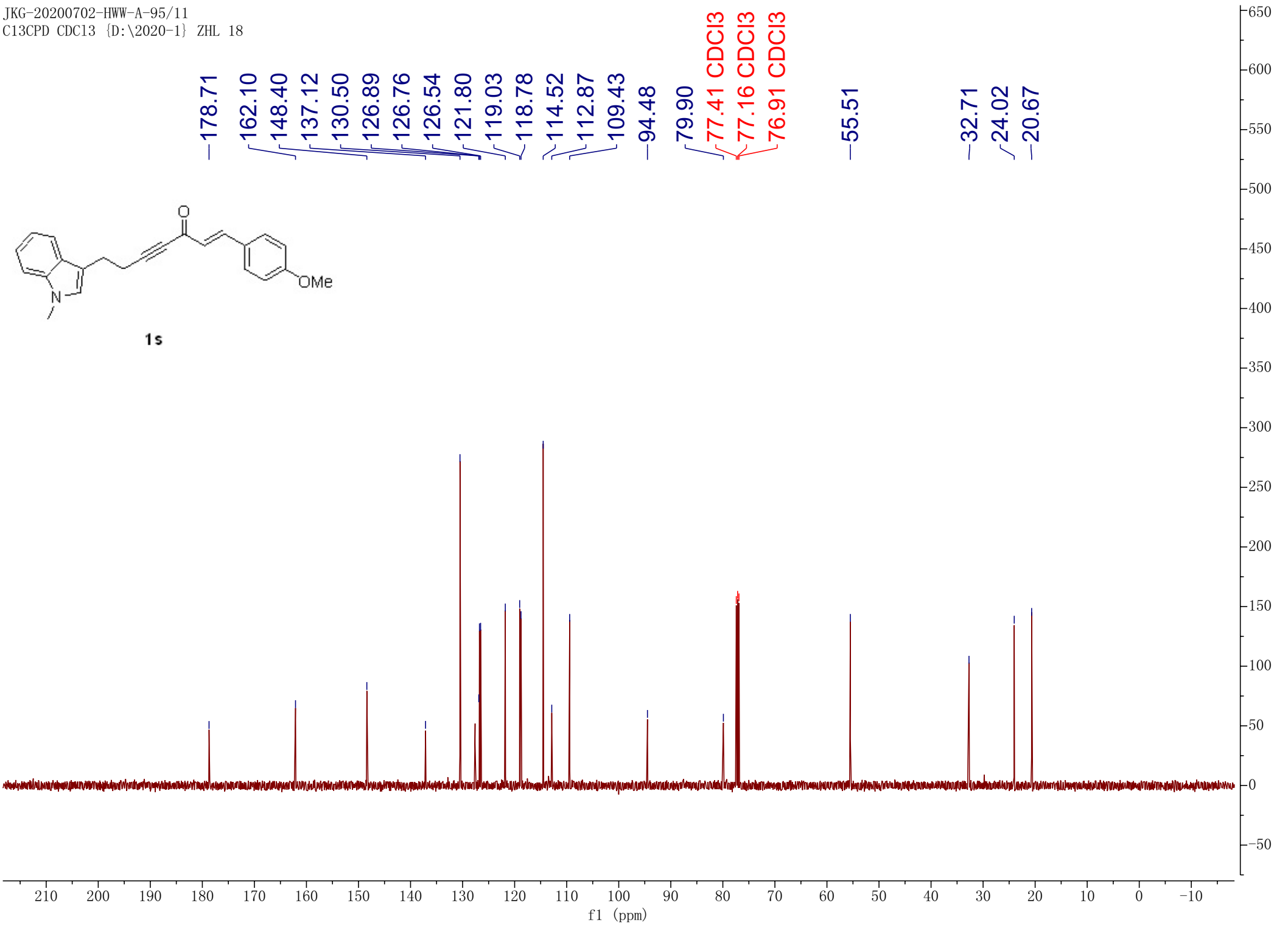


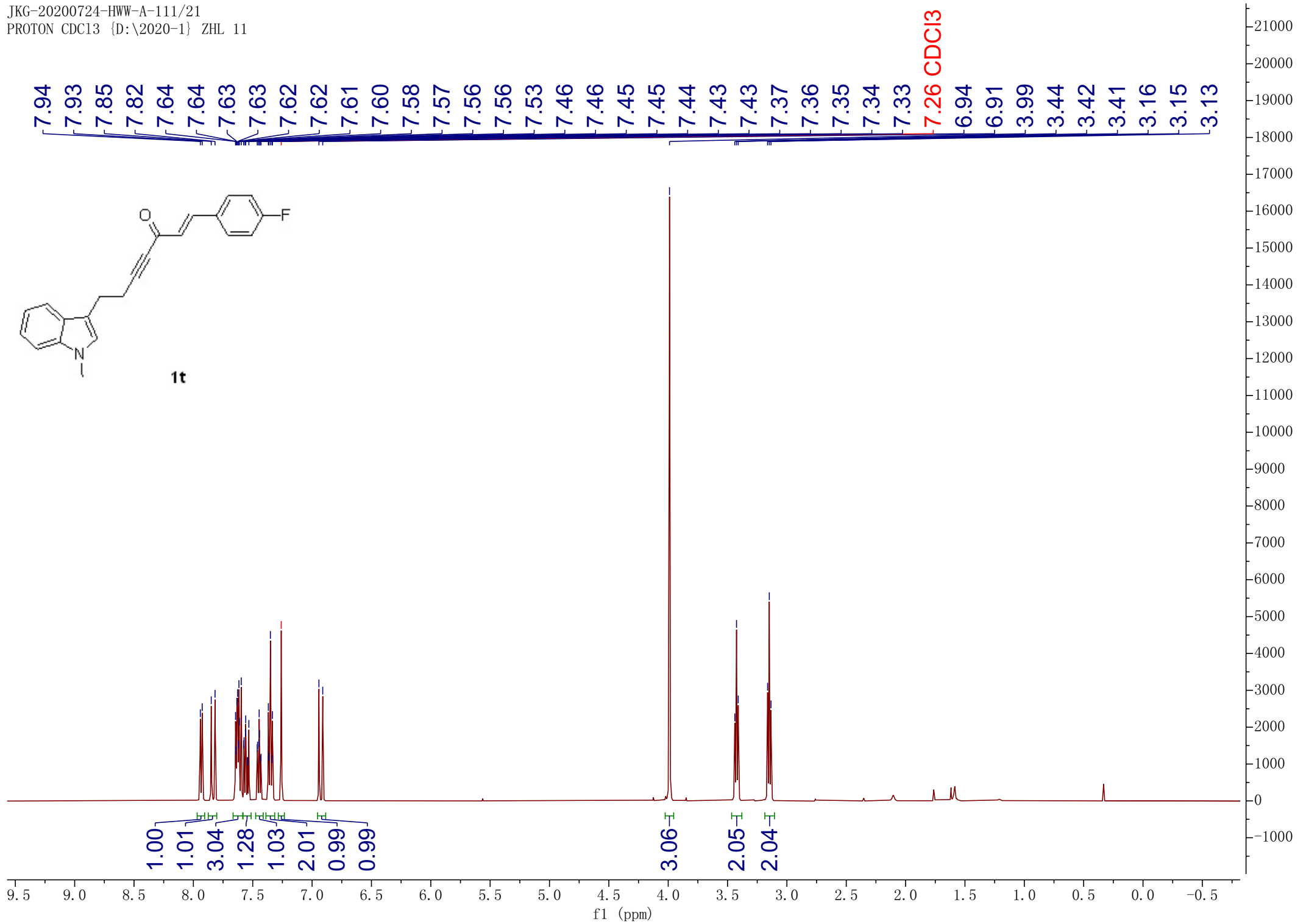
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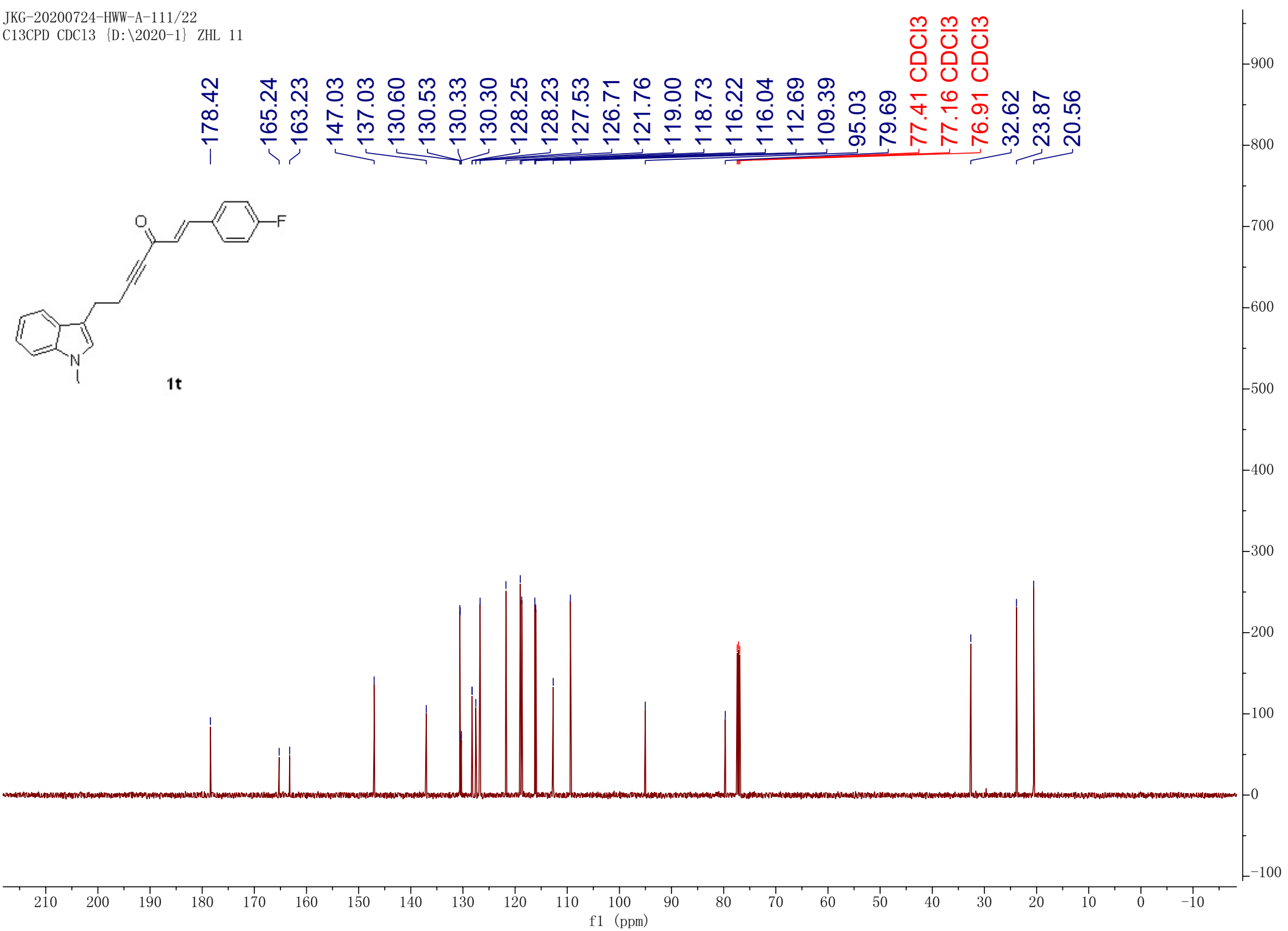
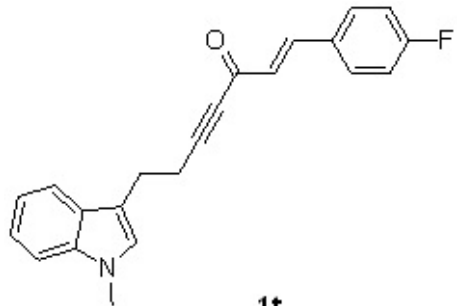


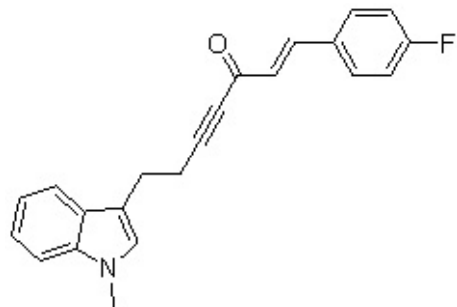


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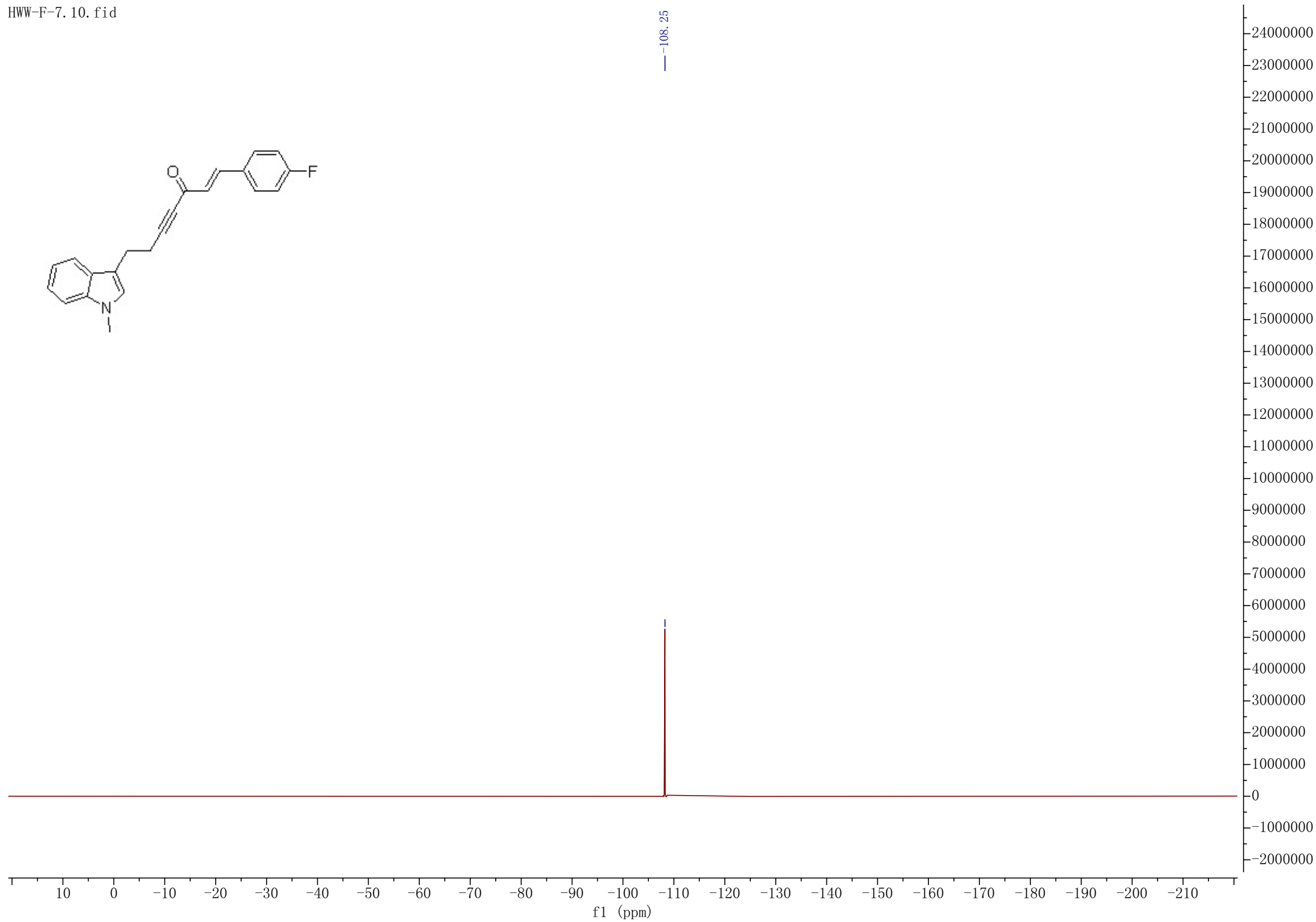


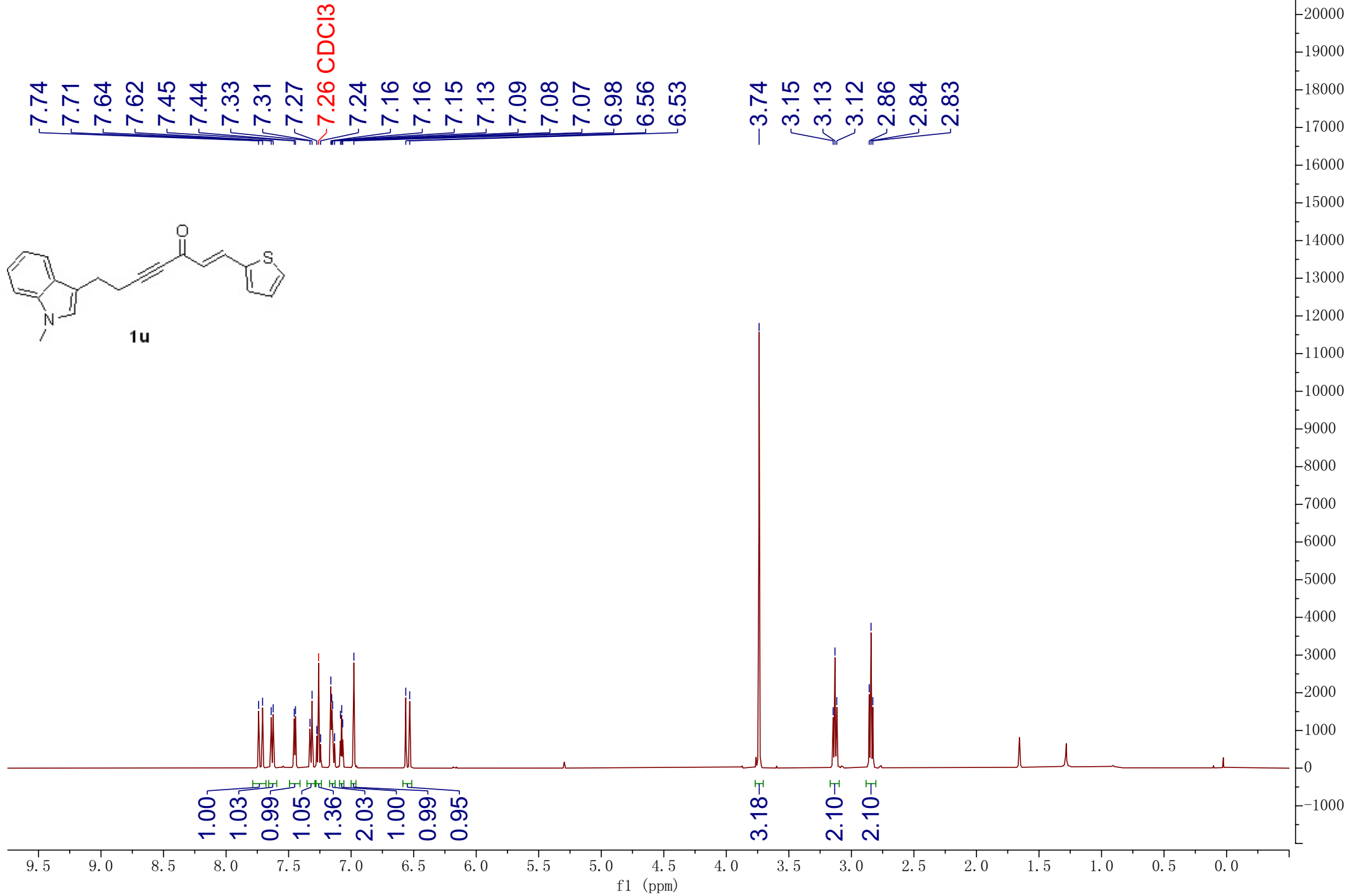


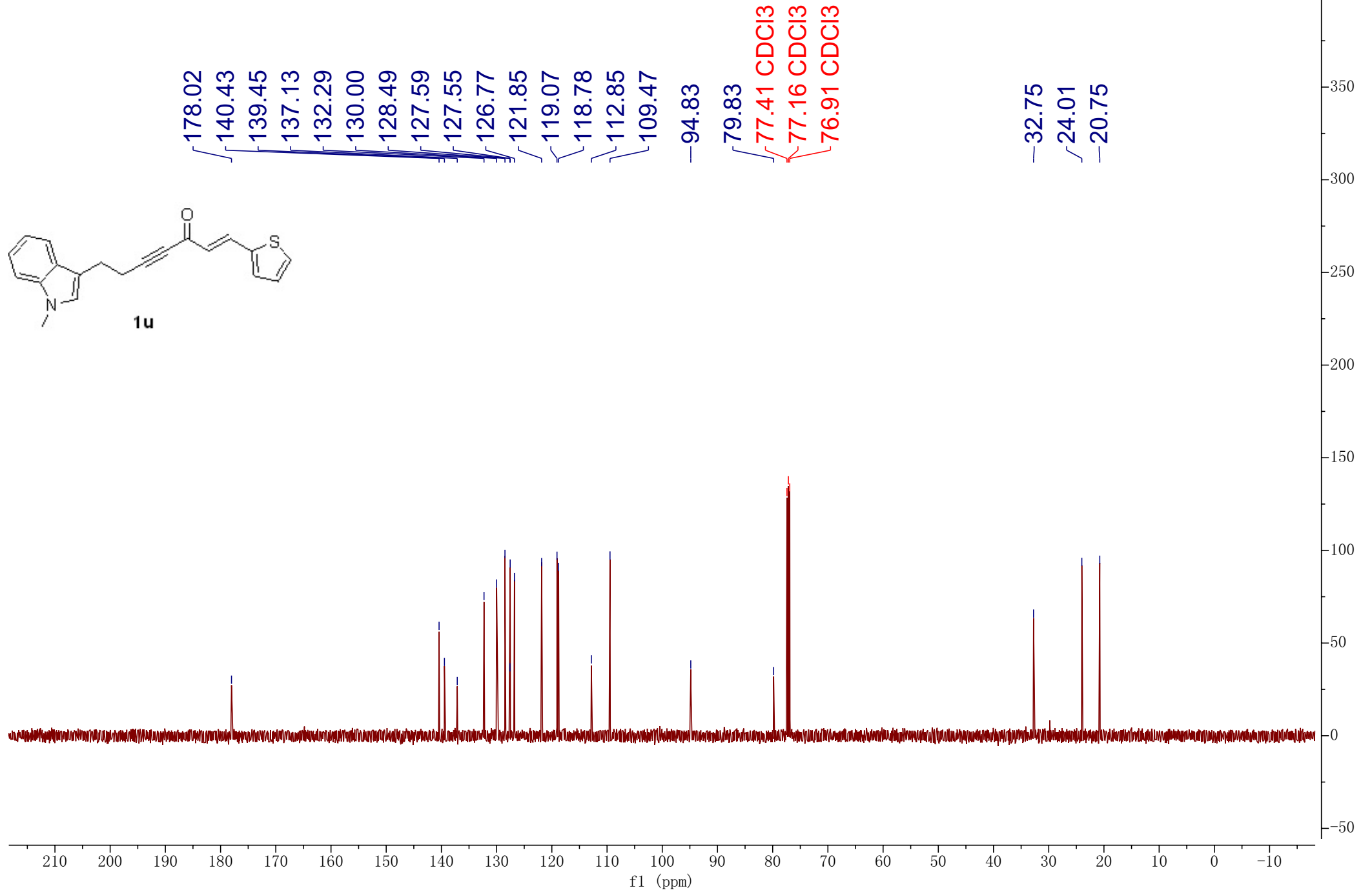
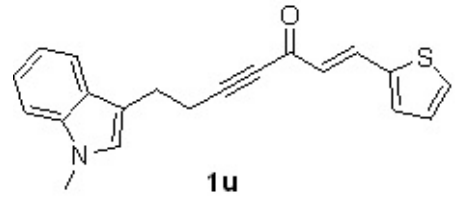


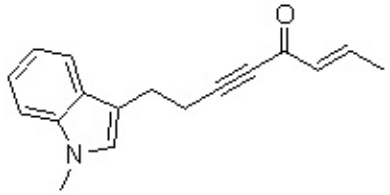


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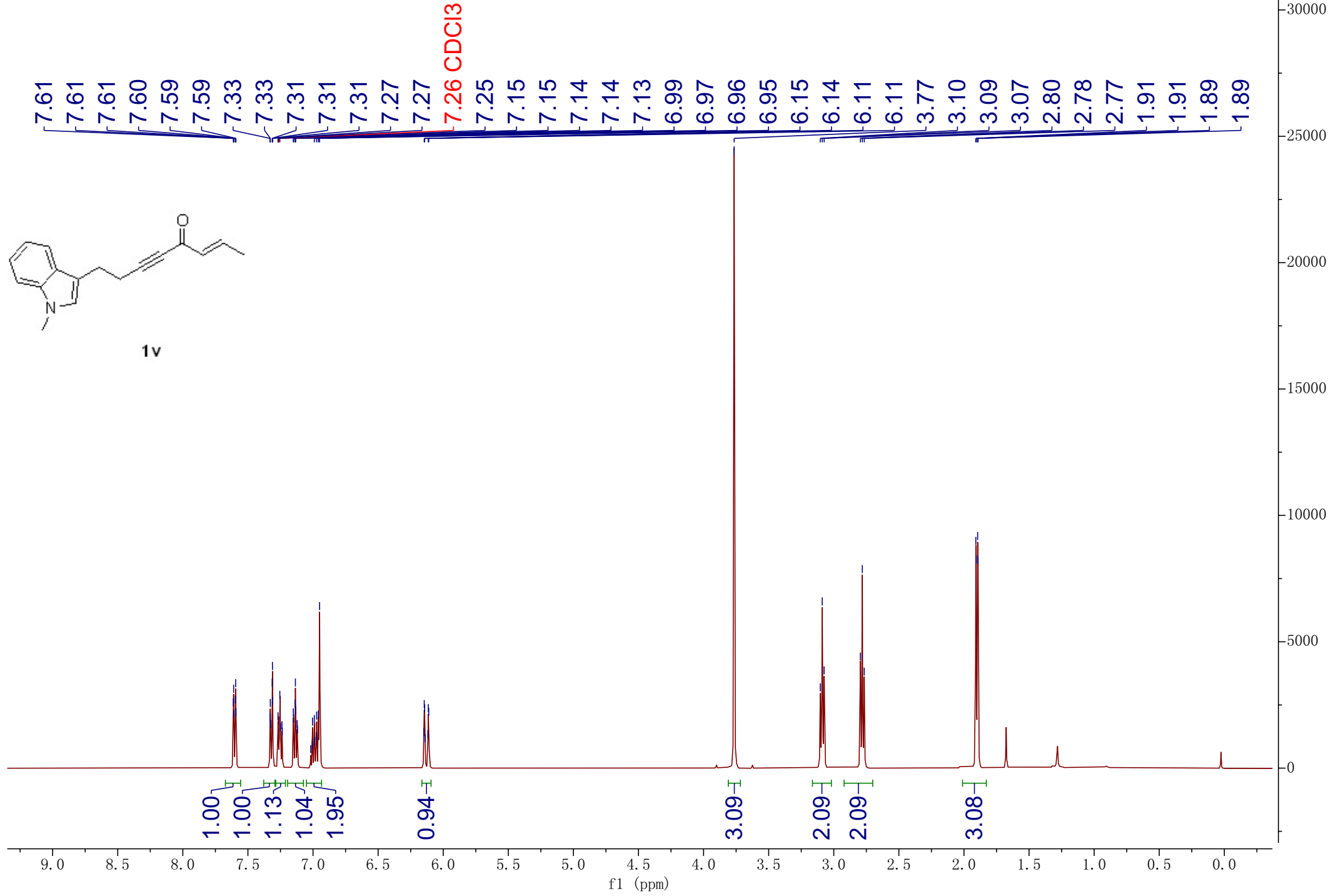


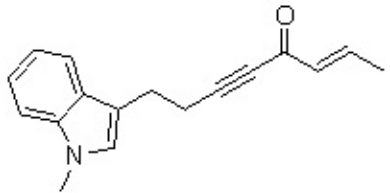




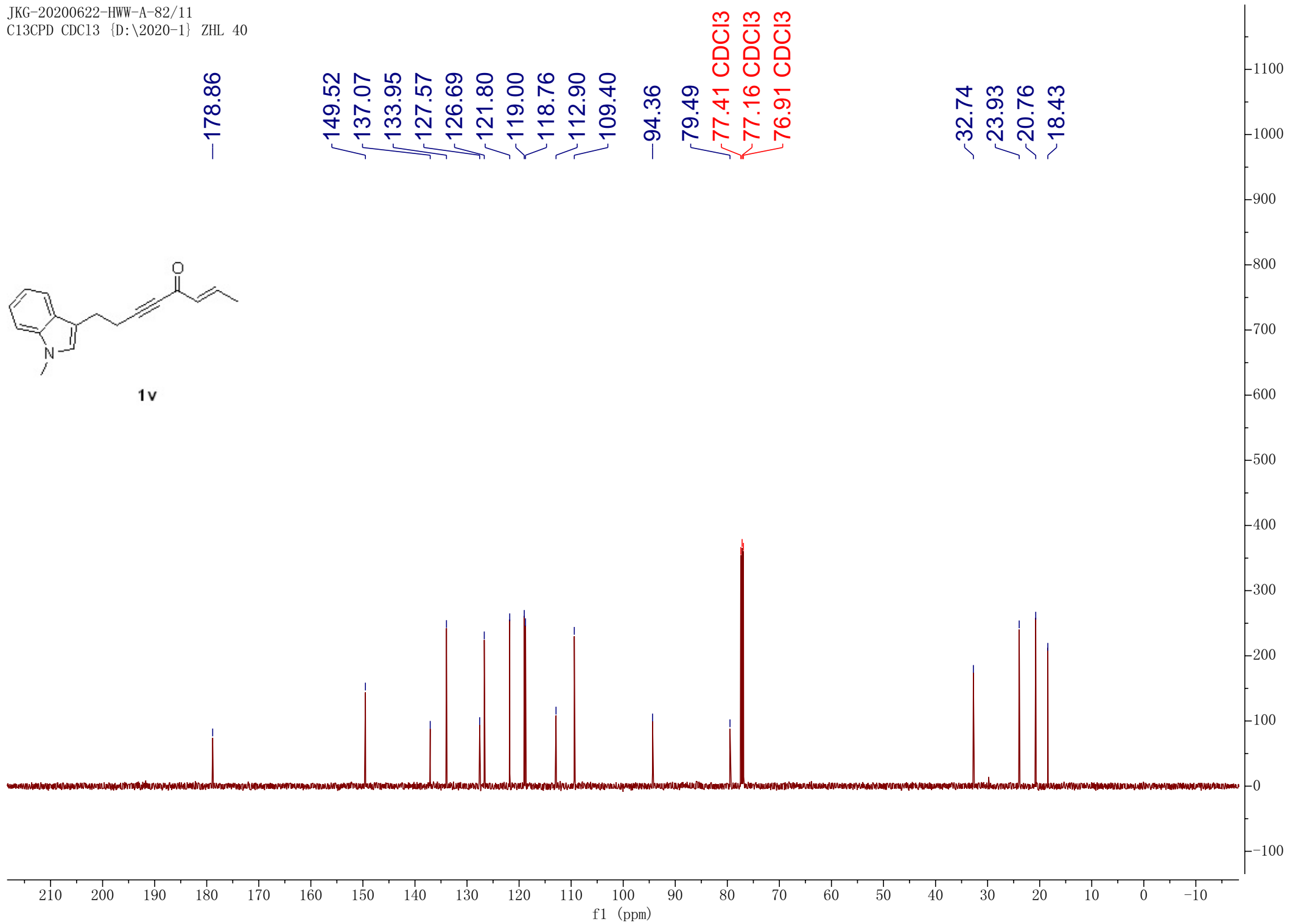


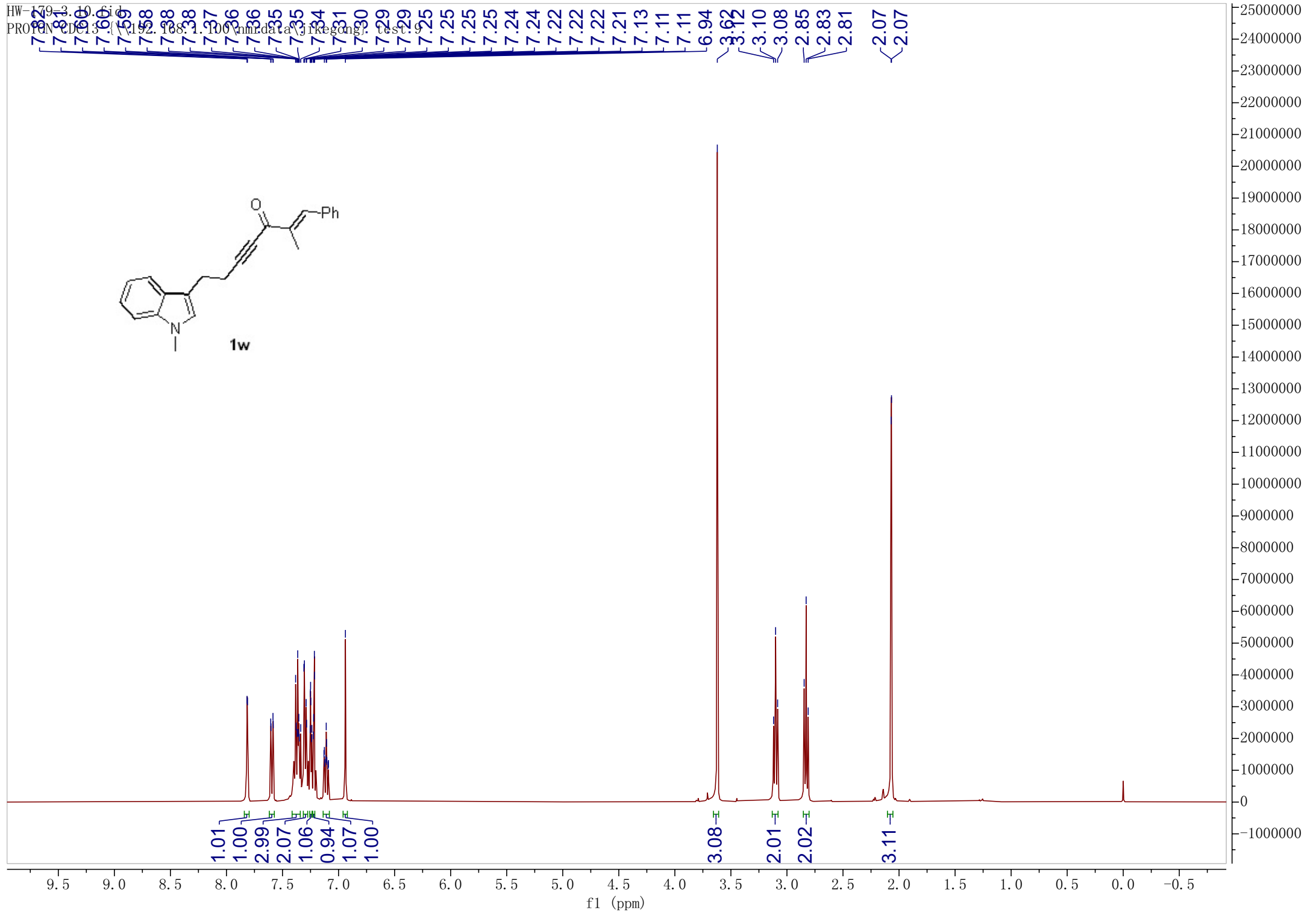
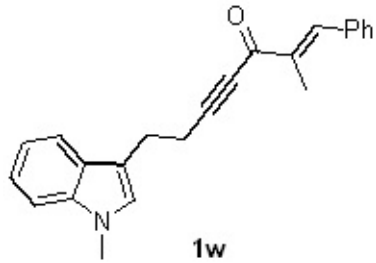
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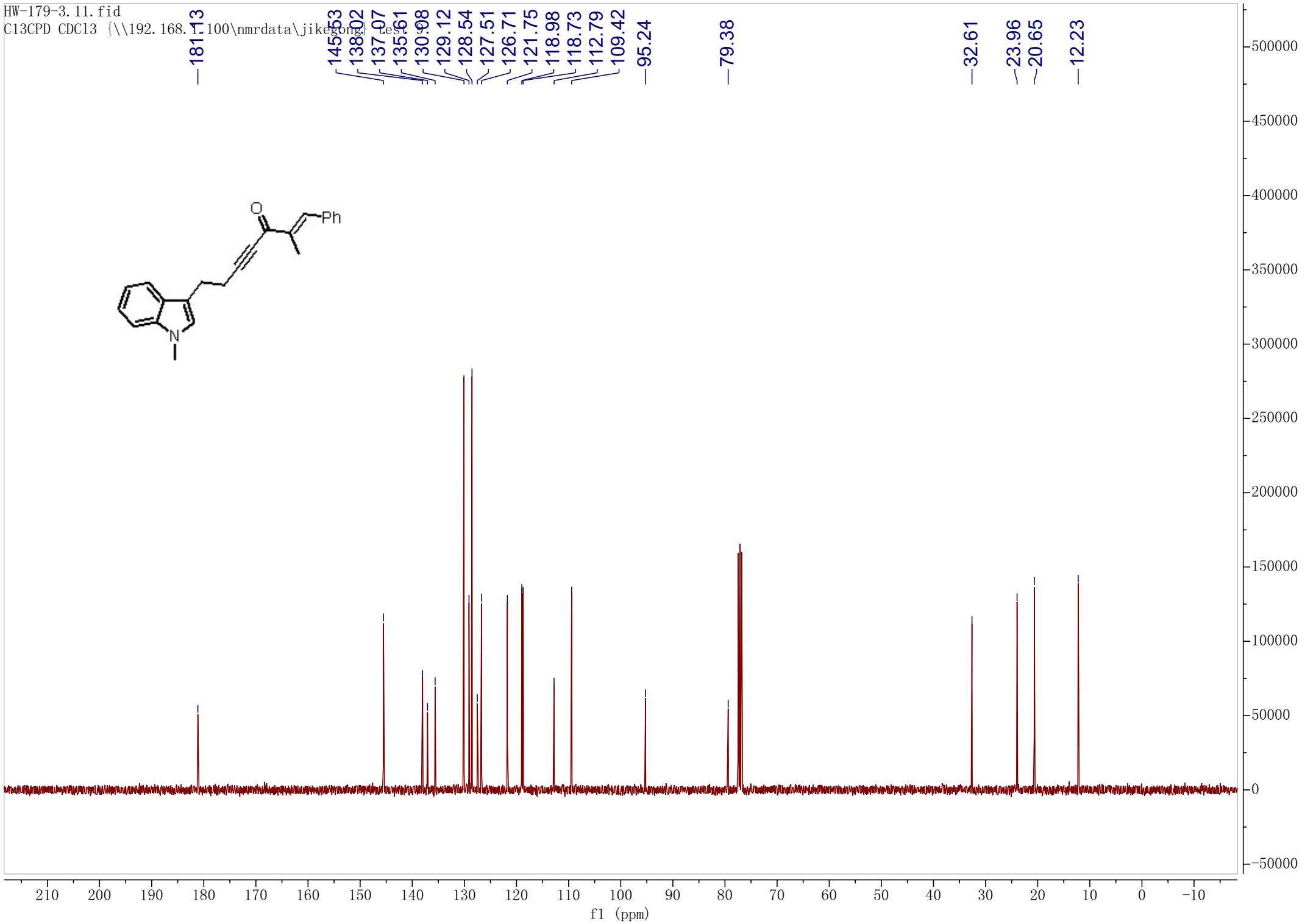
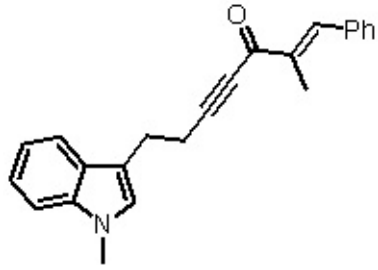




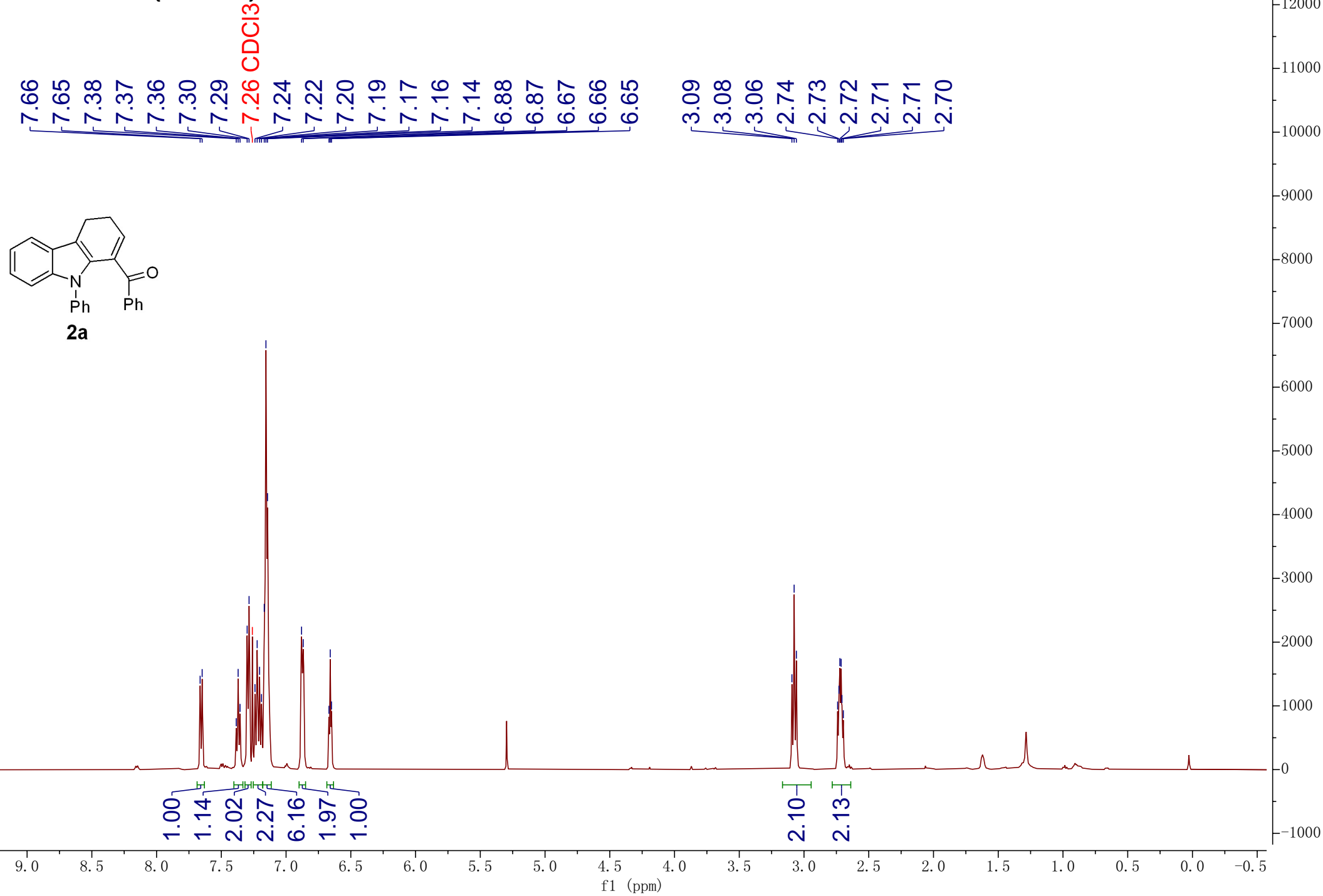
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JKG-20191211-HWW-A-30/10
PROTON CDCl3 {D:\2019-3} ZHL 12



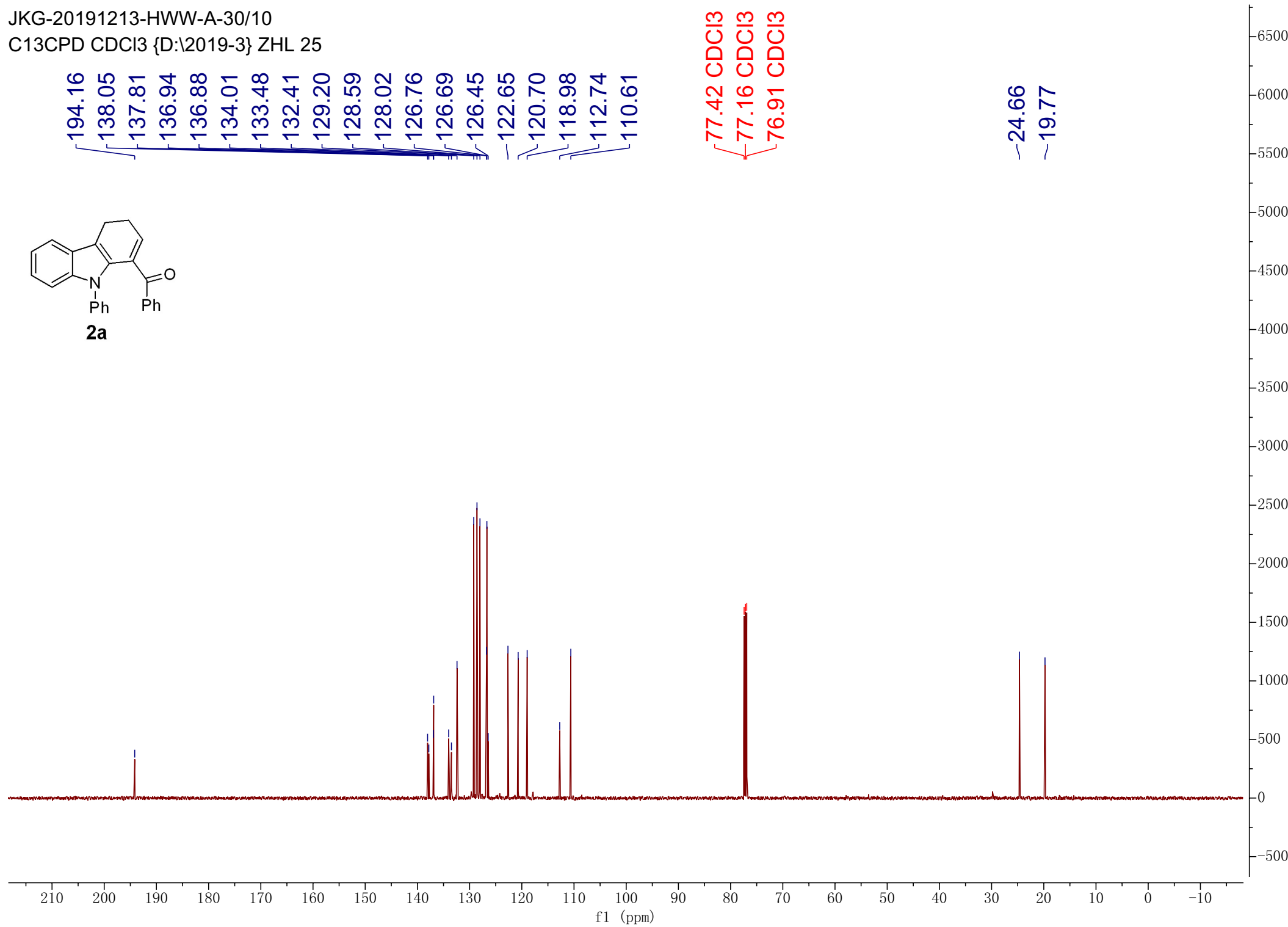
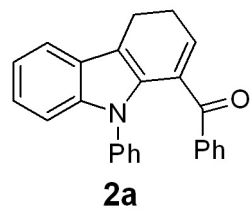
JKG-20191213-HWW-A-30/10

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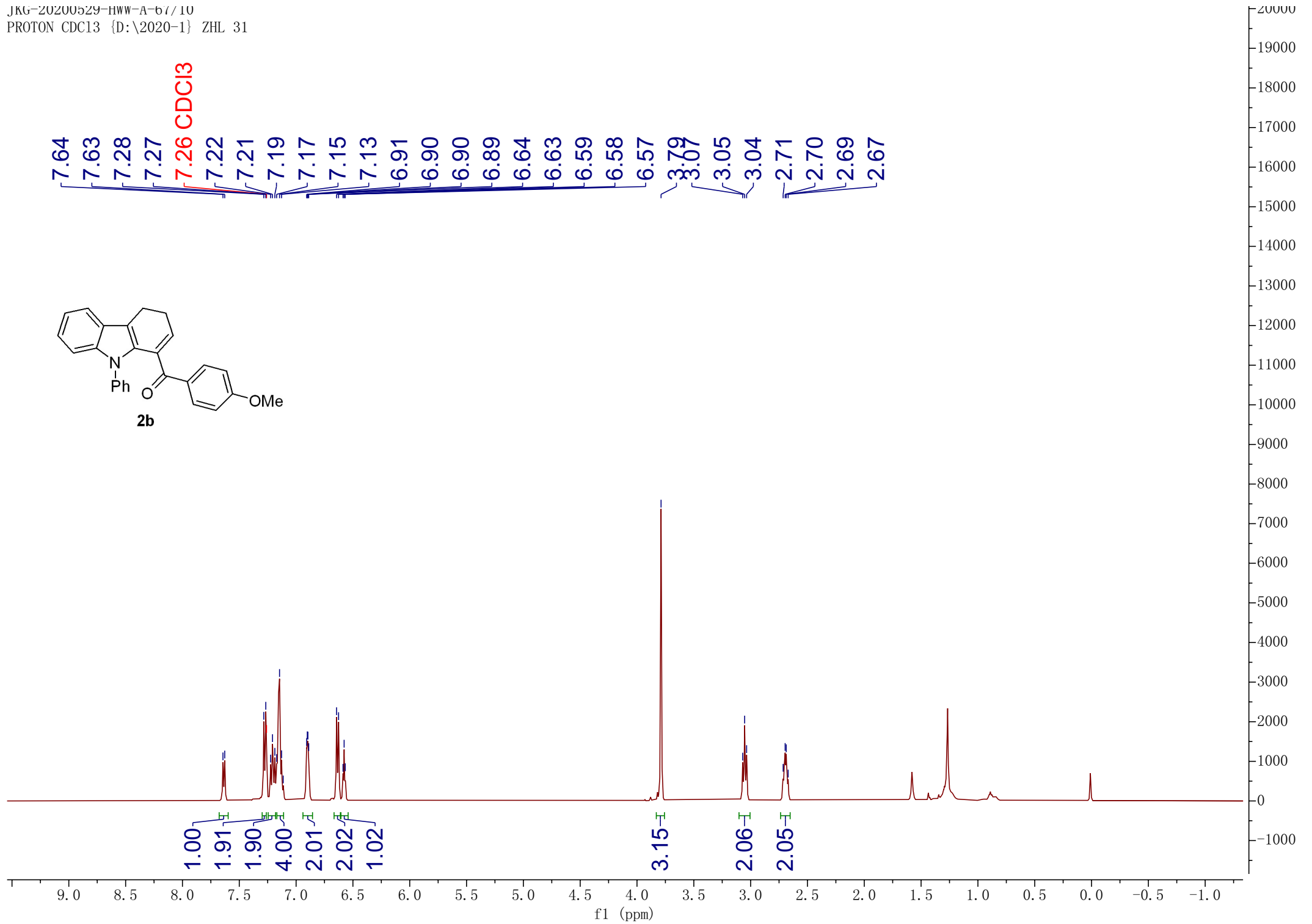
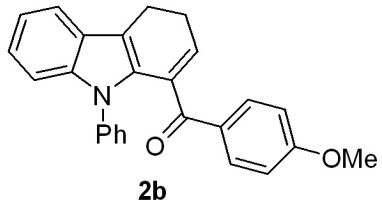
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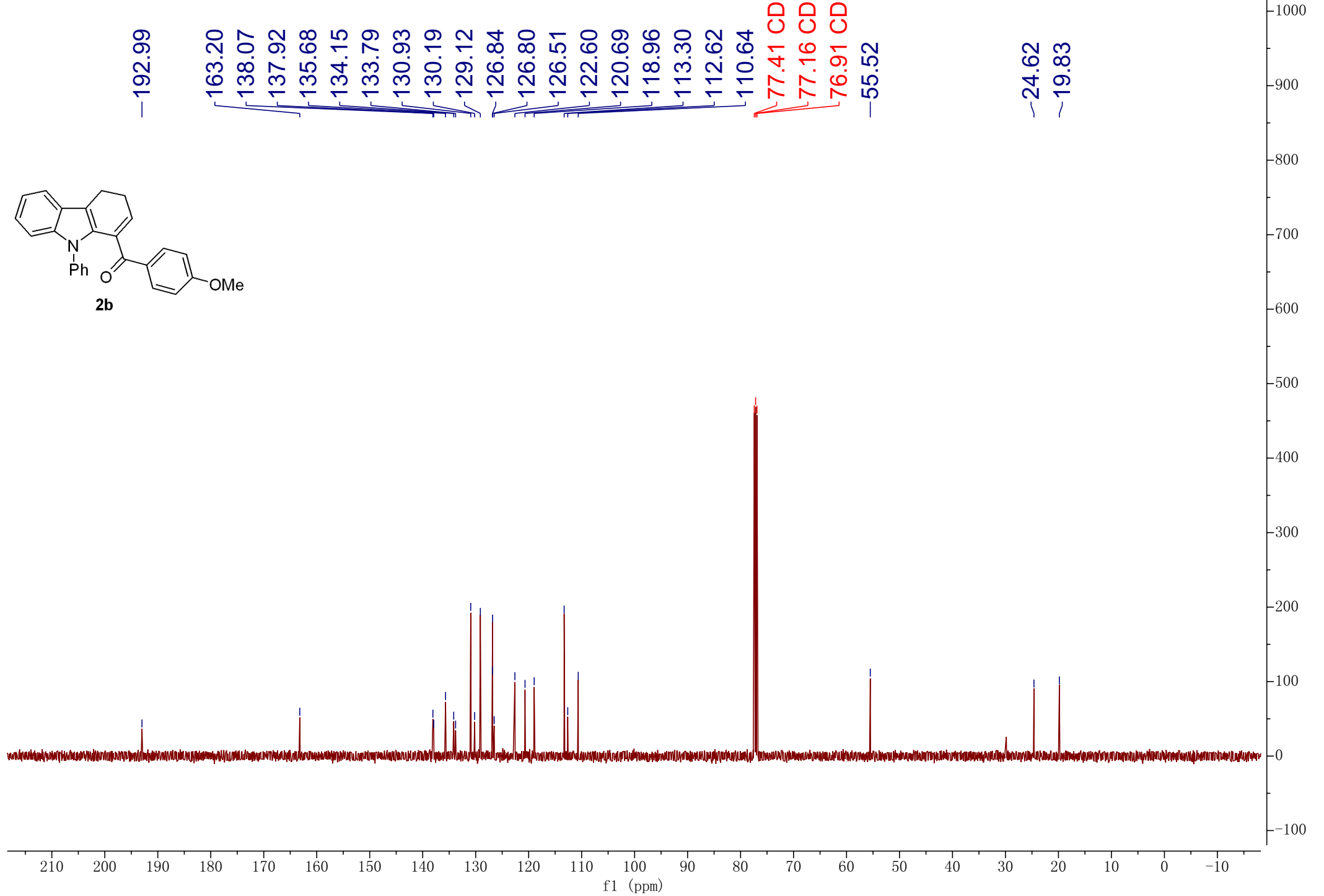
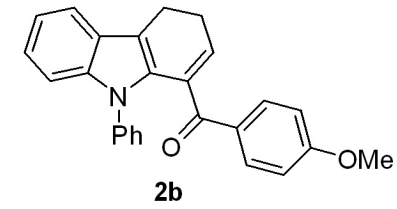
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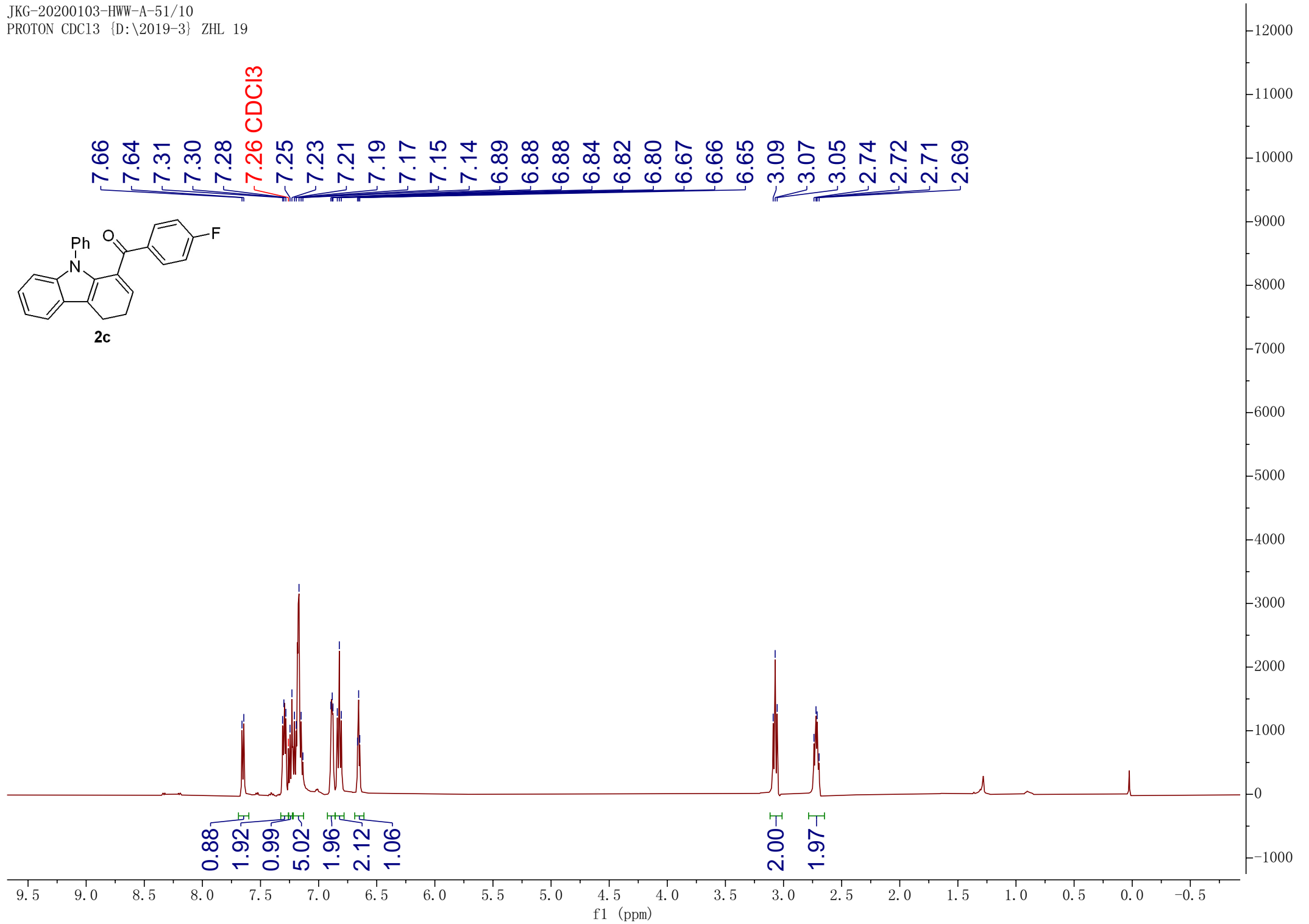
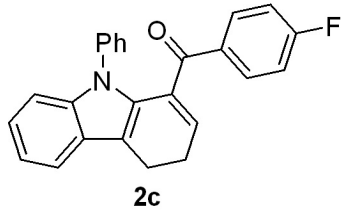
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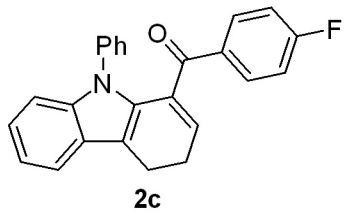


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3.05
3.04
2.71
2.70
2.69
2.67









192.62

166.39

164.37

138.03

137.82

136.81

133.80

133.44

133.41

133.24

131.13

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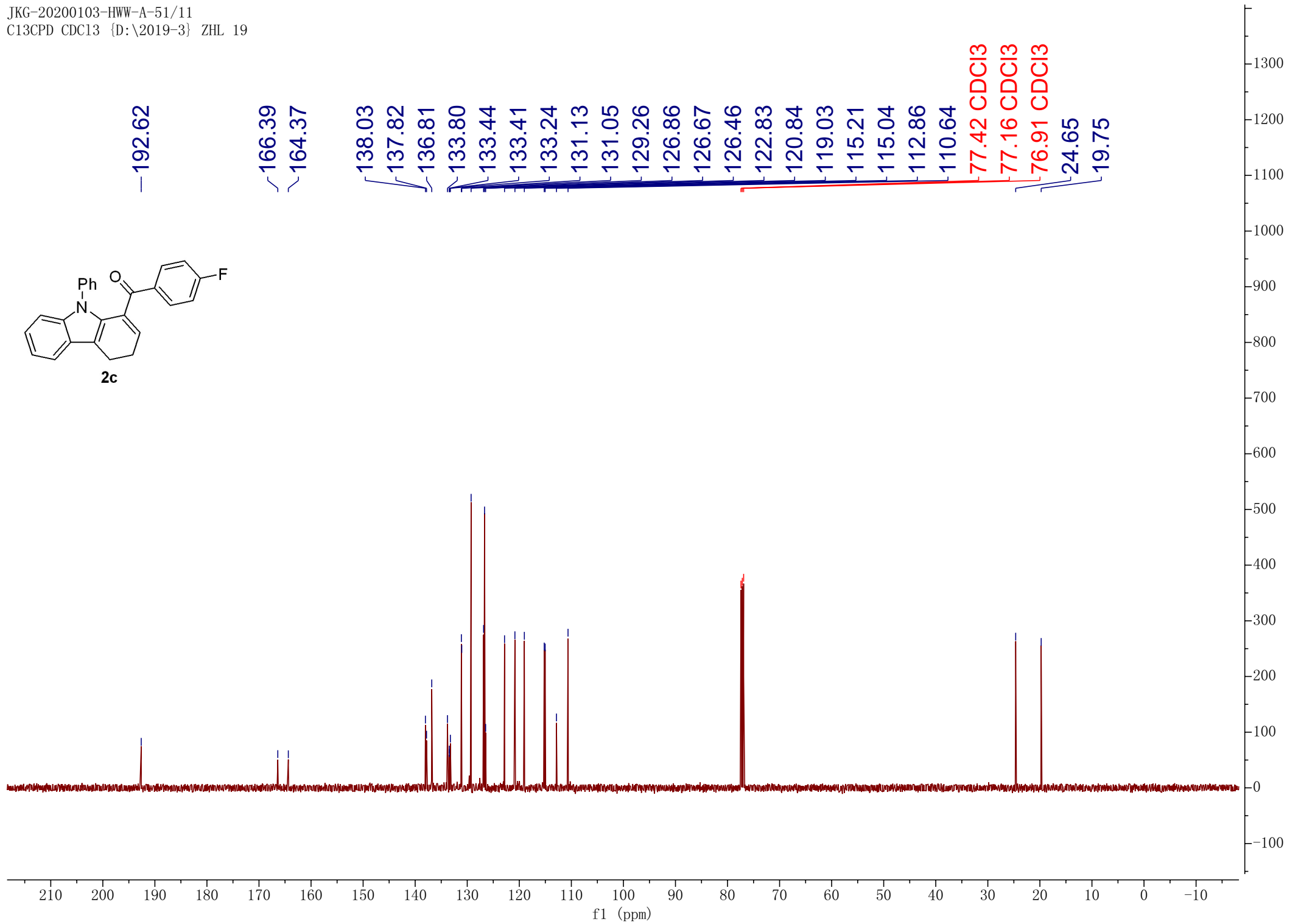
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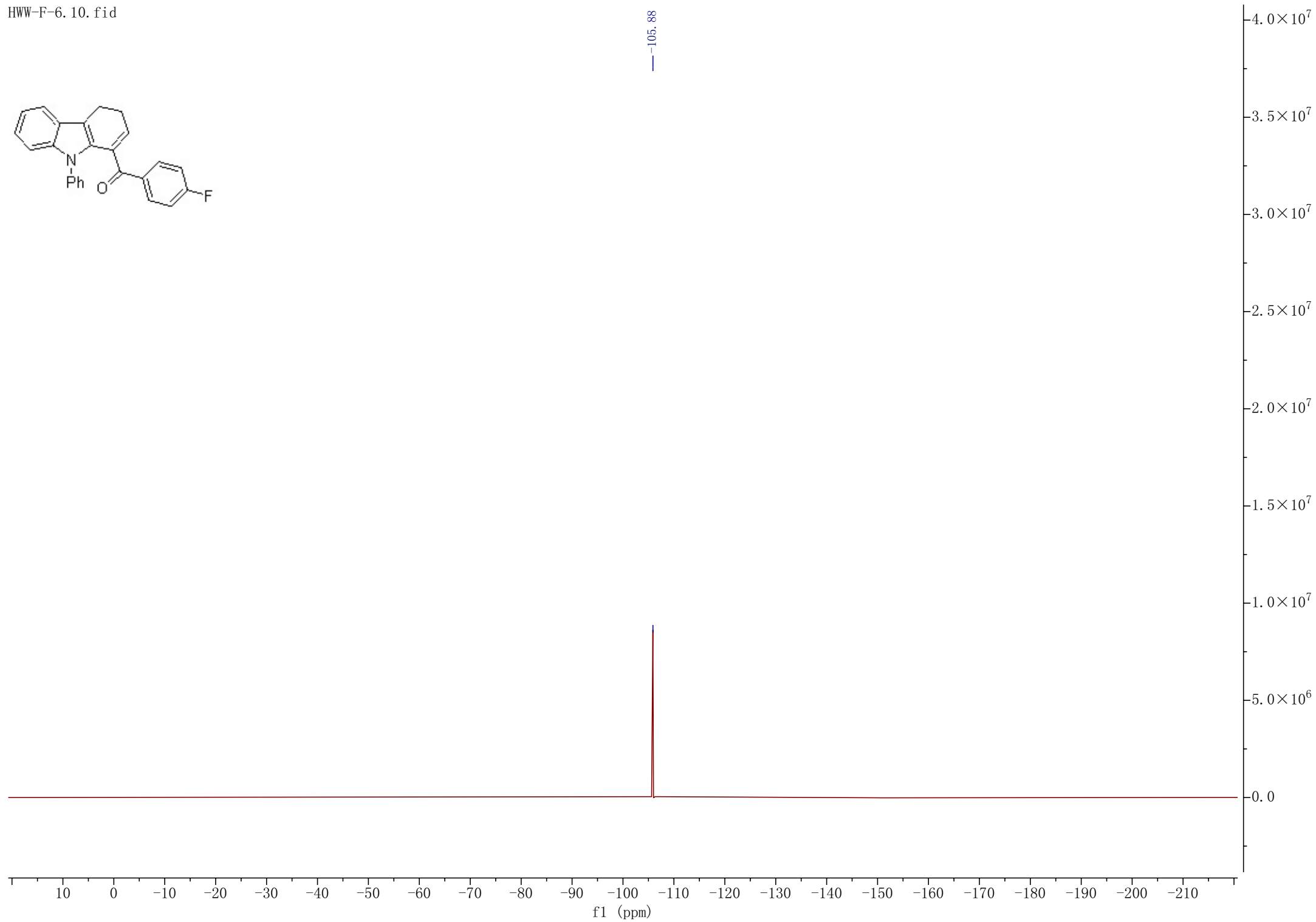
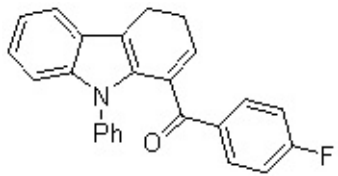
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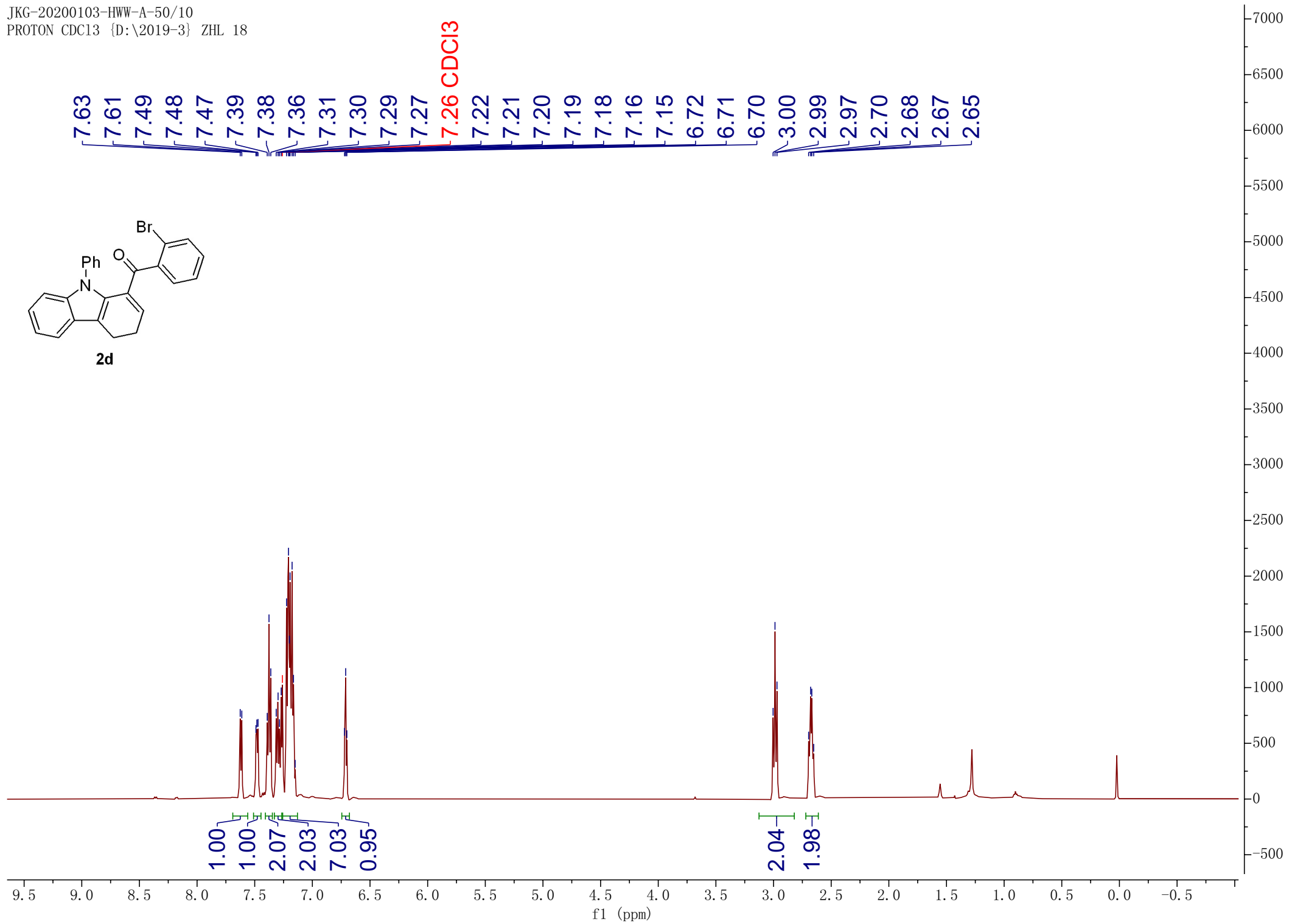
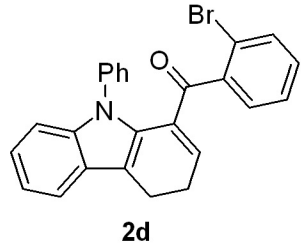
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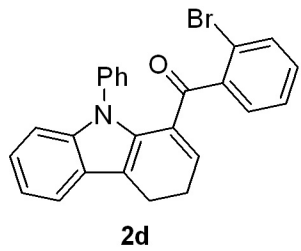
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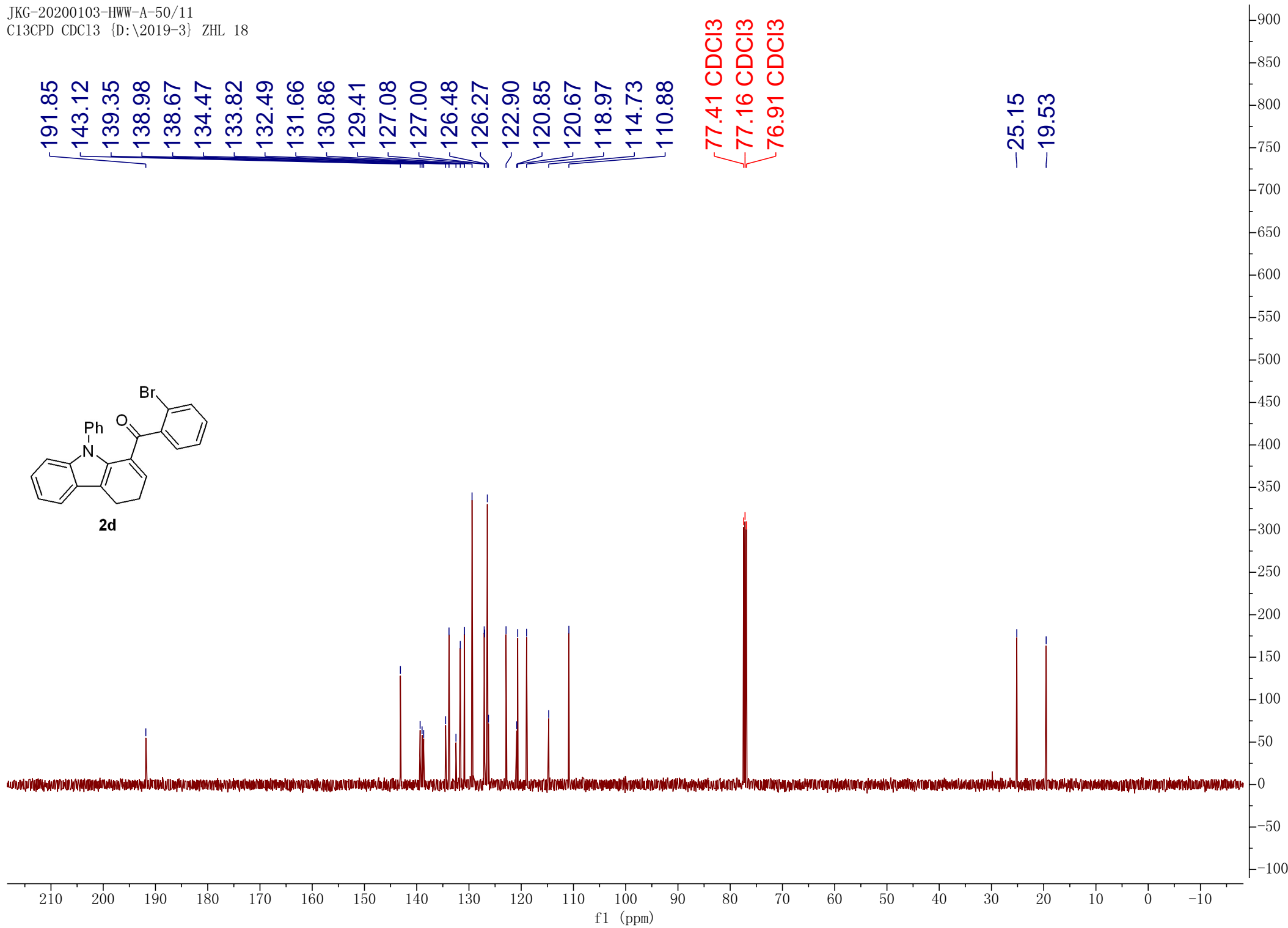


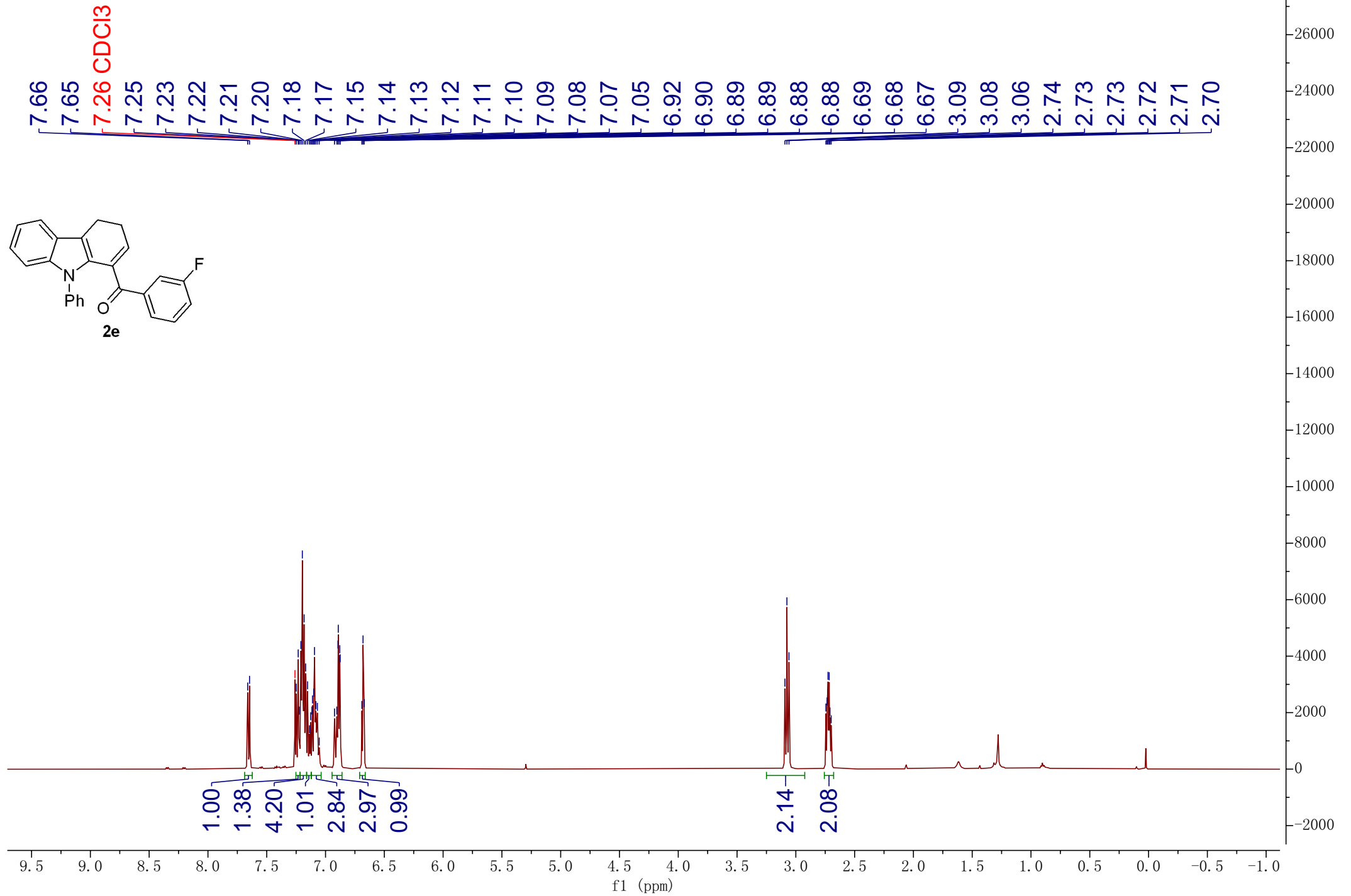


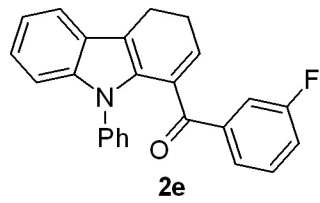
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118.97
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110.88

77.41 CDC13
77.16 CDC13
76.91 CDC13

-25.15
-19.53







—192.85

163.47

161.51

139.13

139.07

138.01

137.79

137.48

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129.58

129.52

129.32

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126.71

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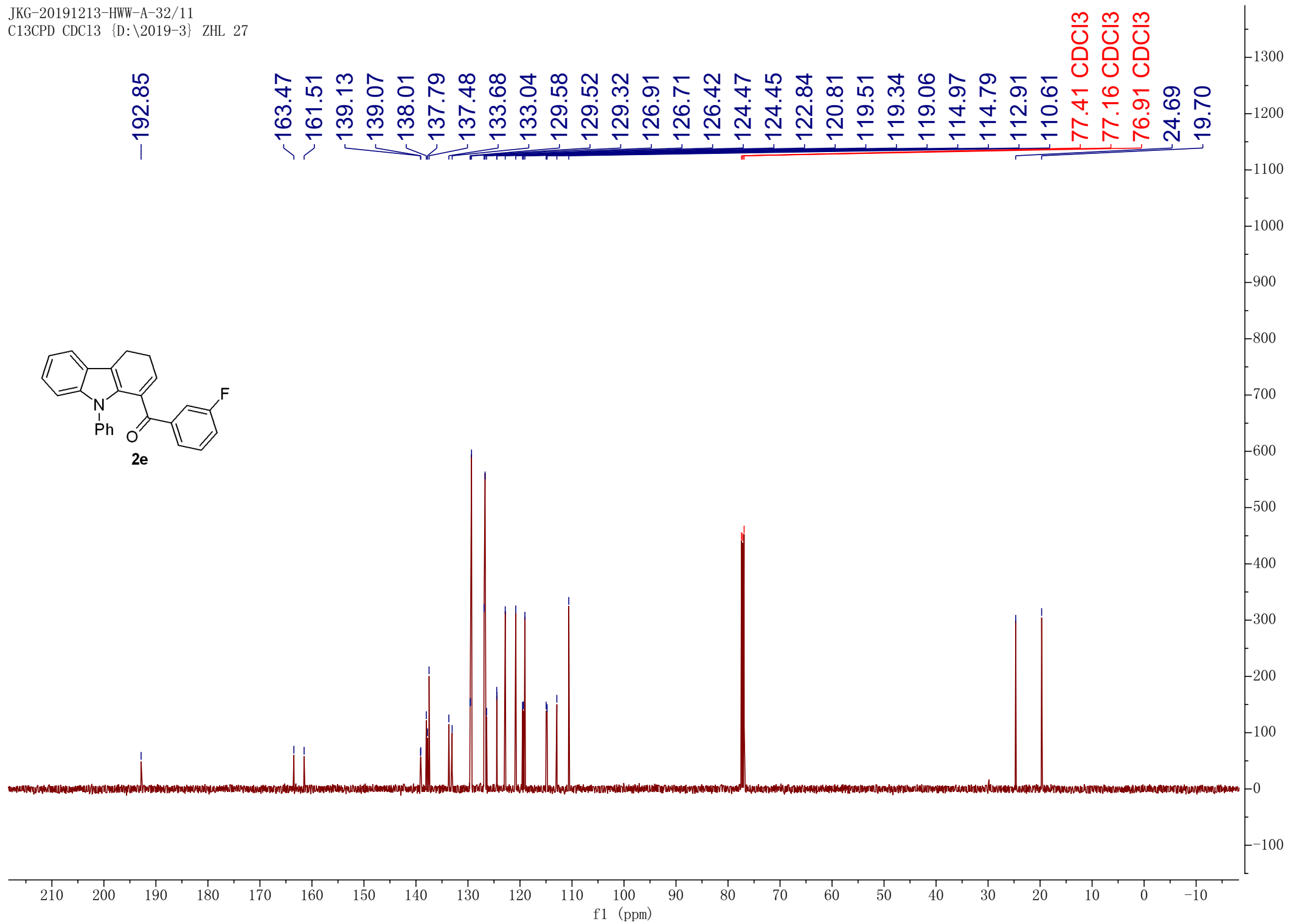
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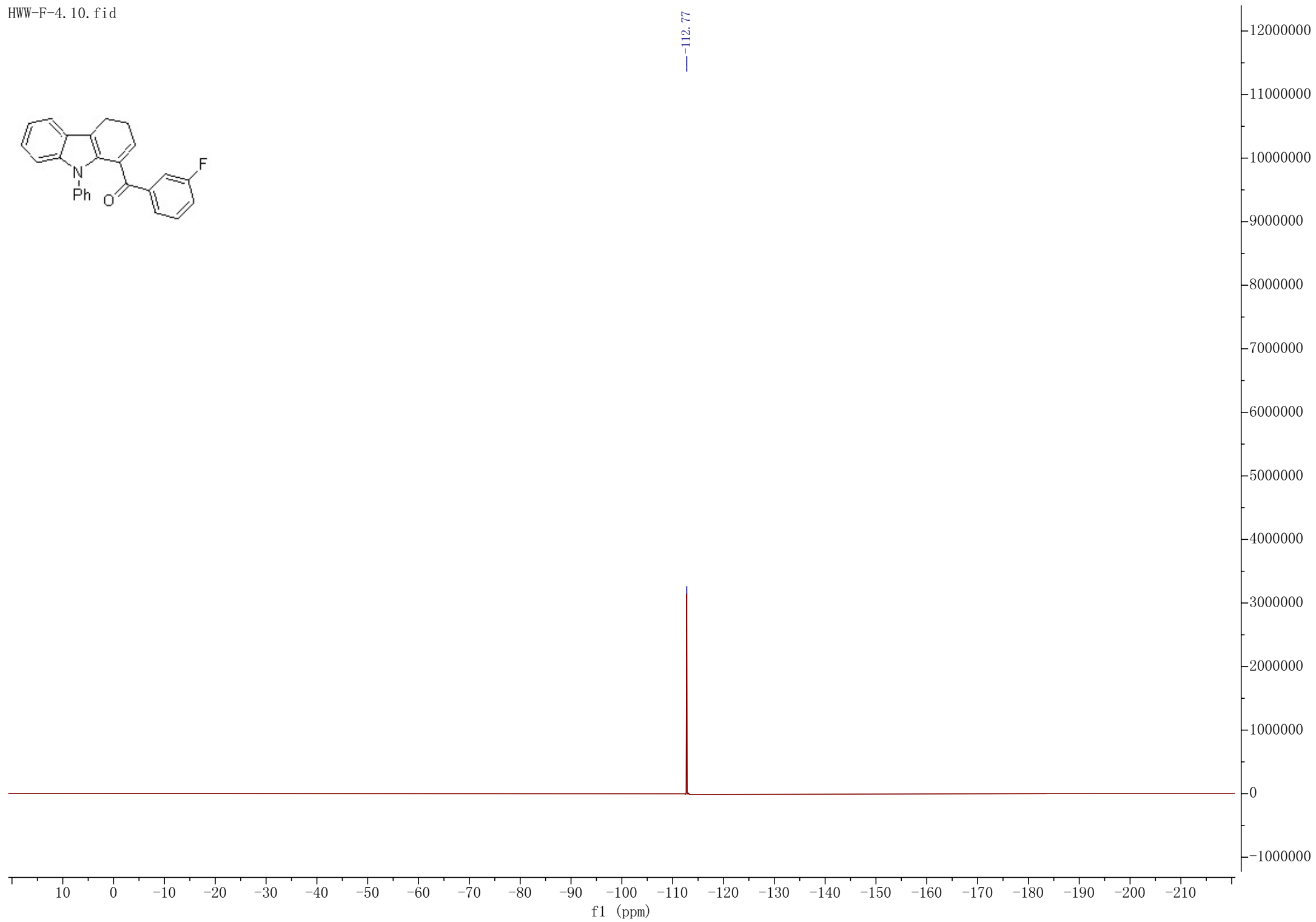
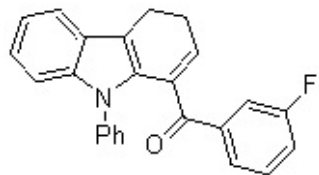
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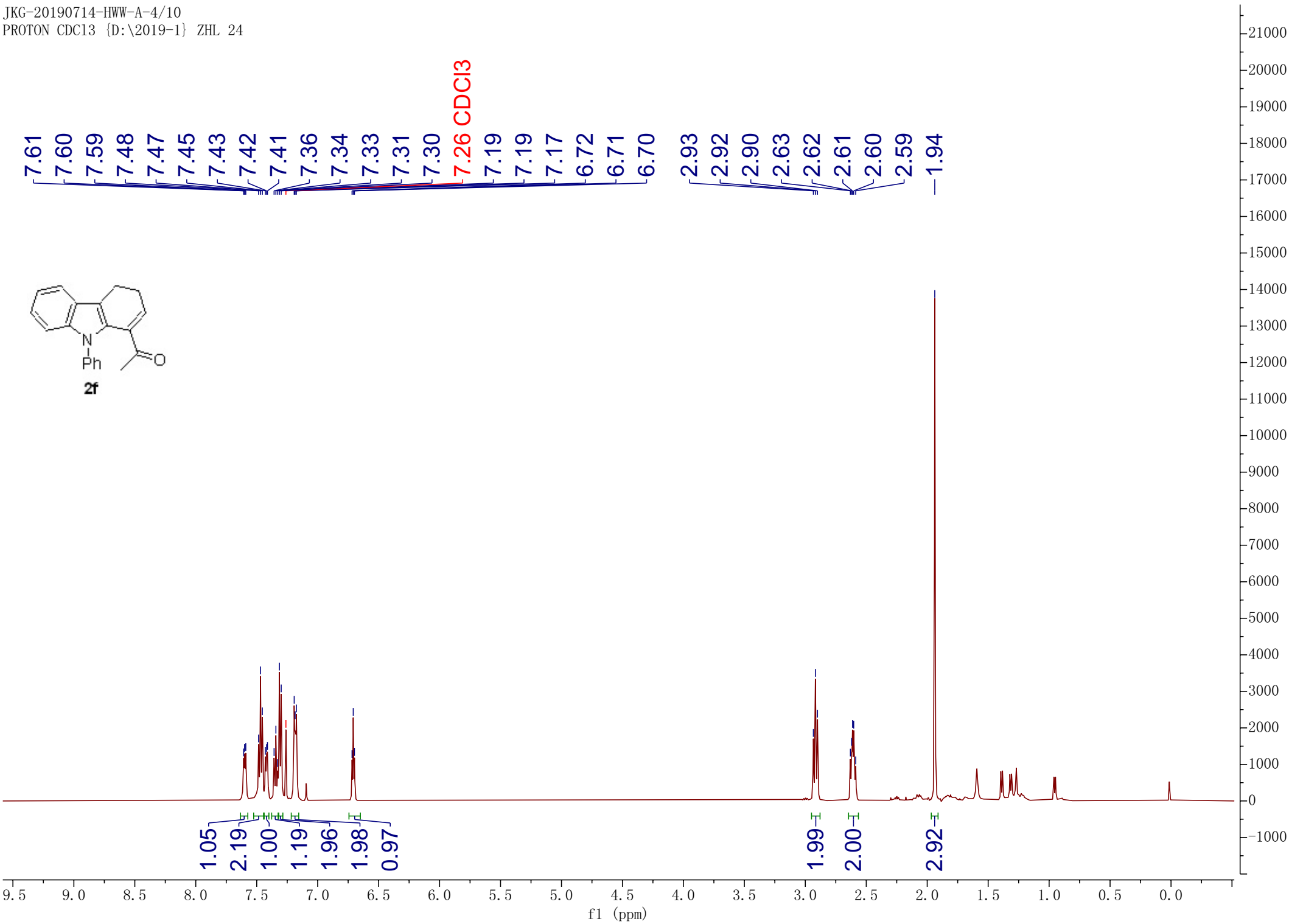
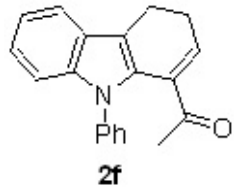


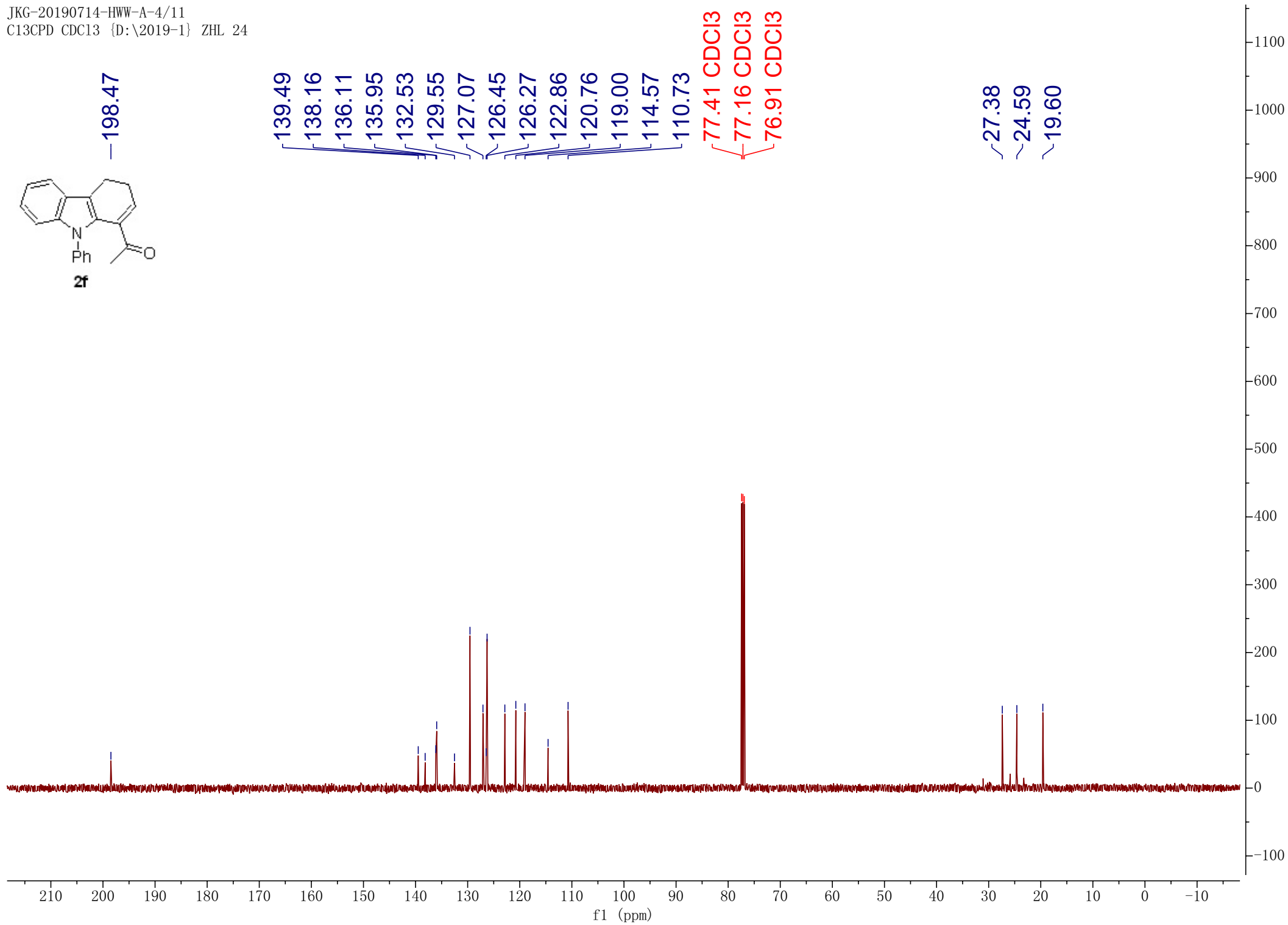
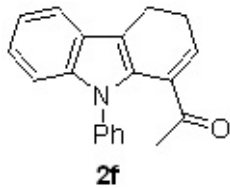
HWW-F-4. 10. fid

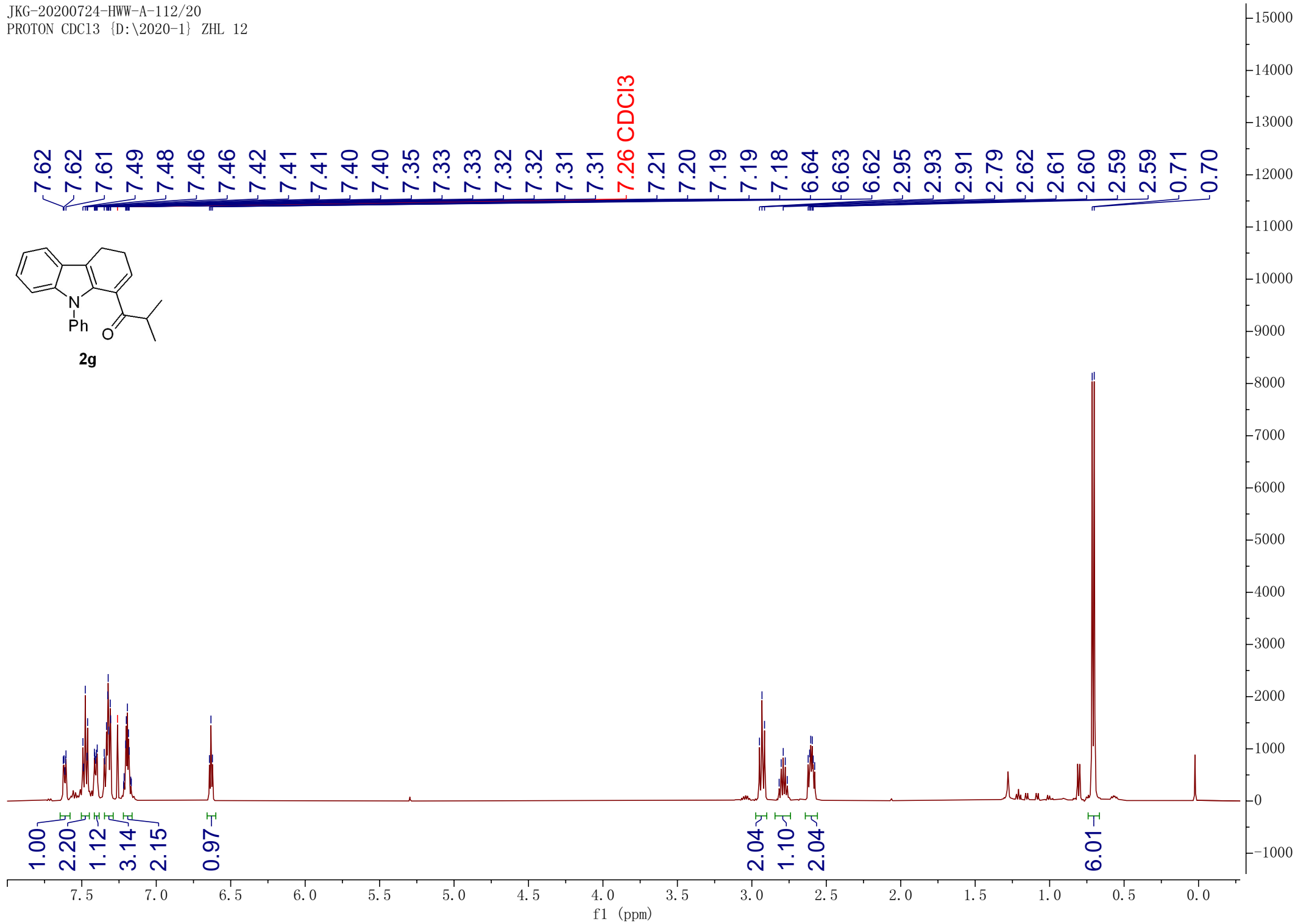
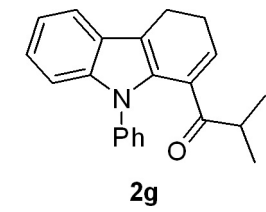


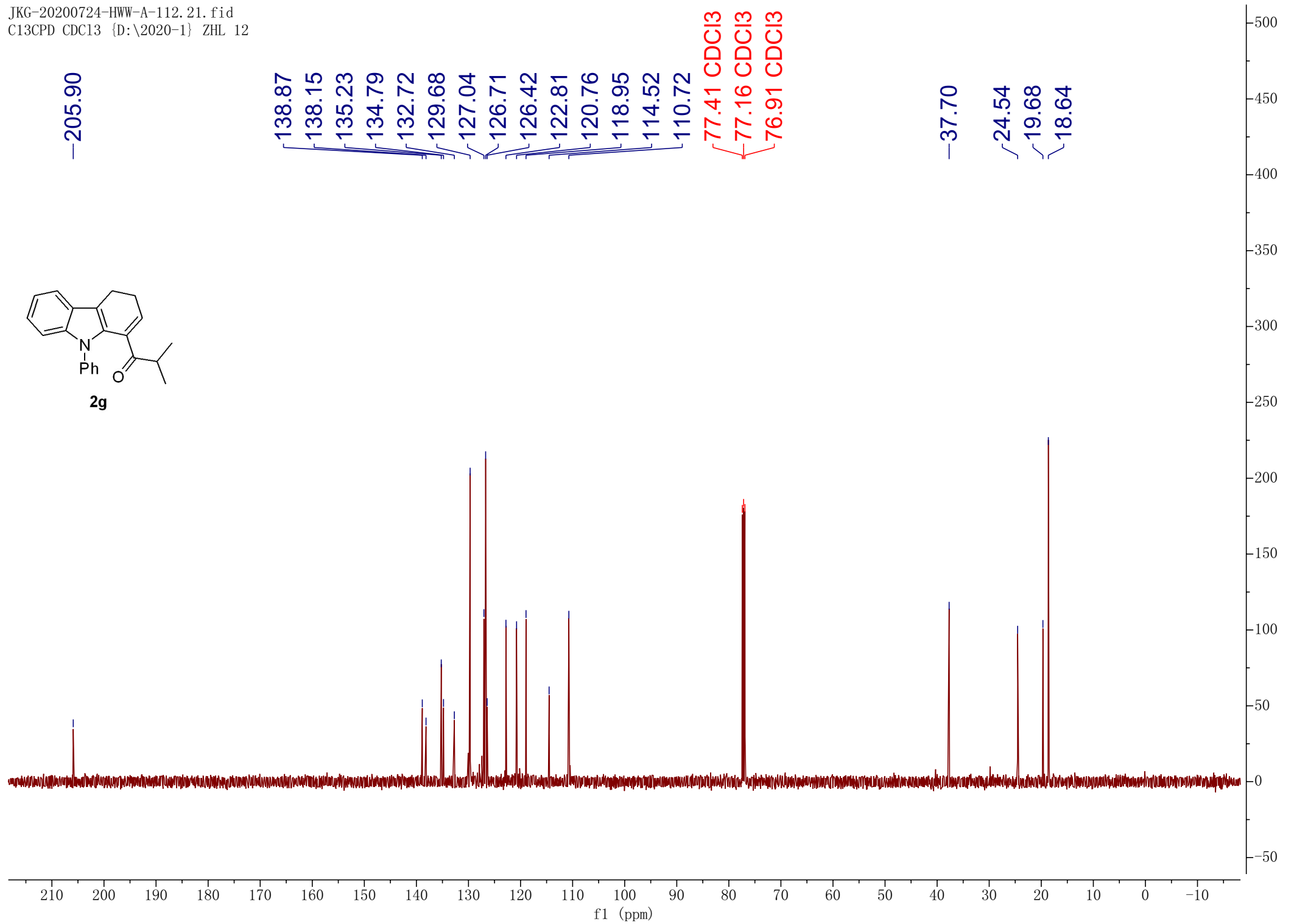
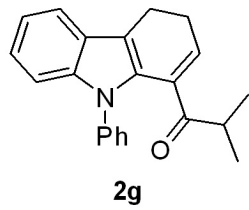
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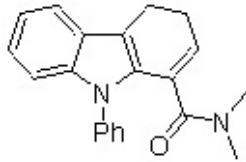
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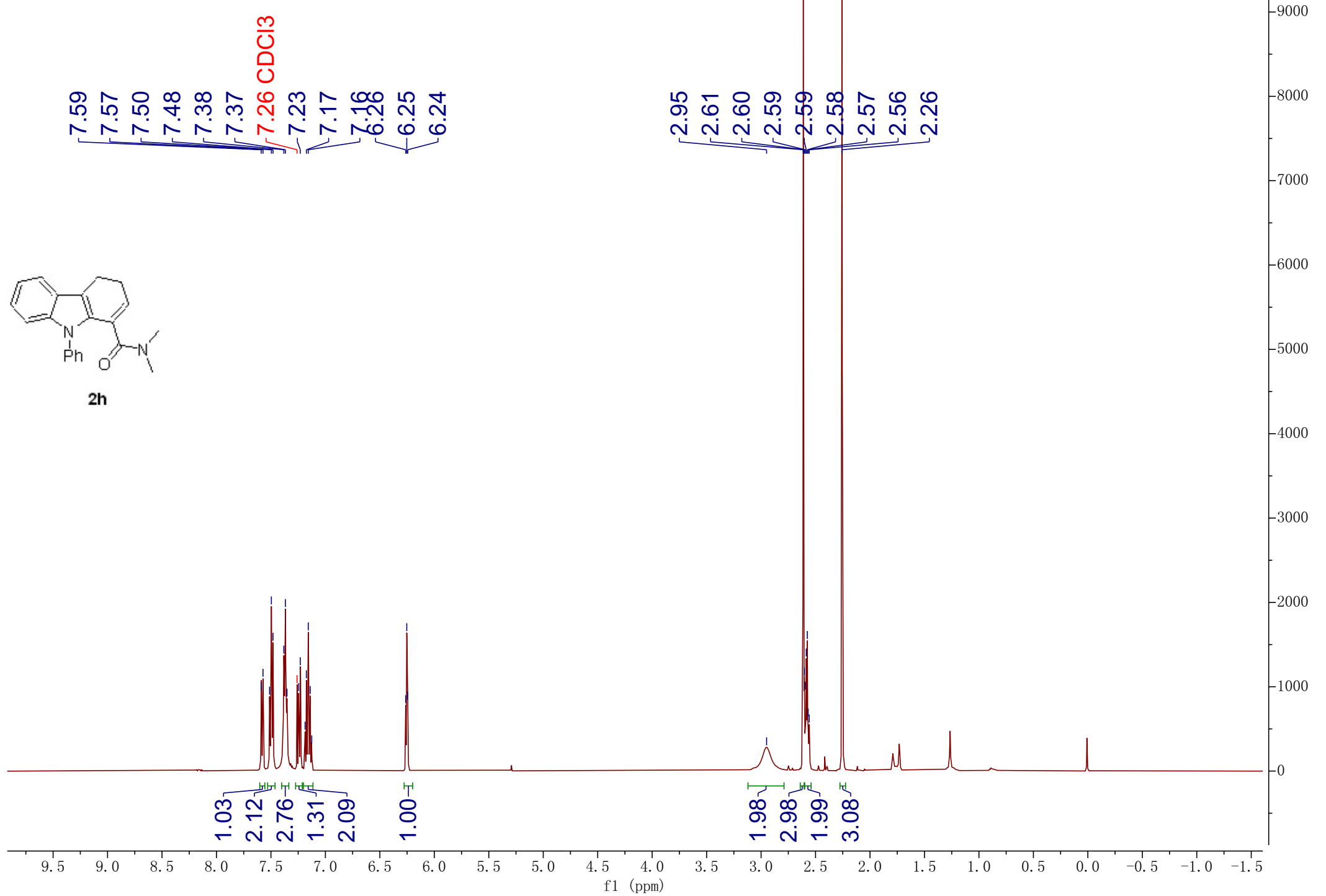


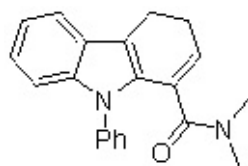




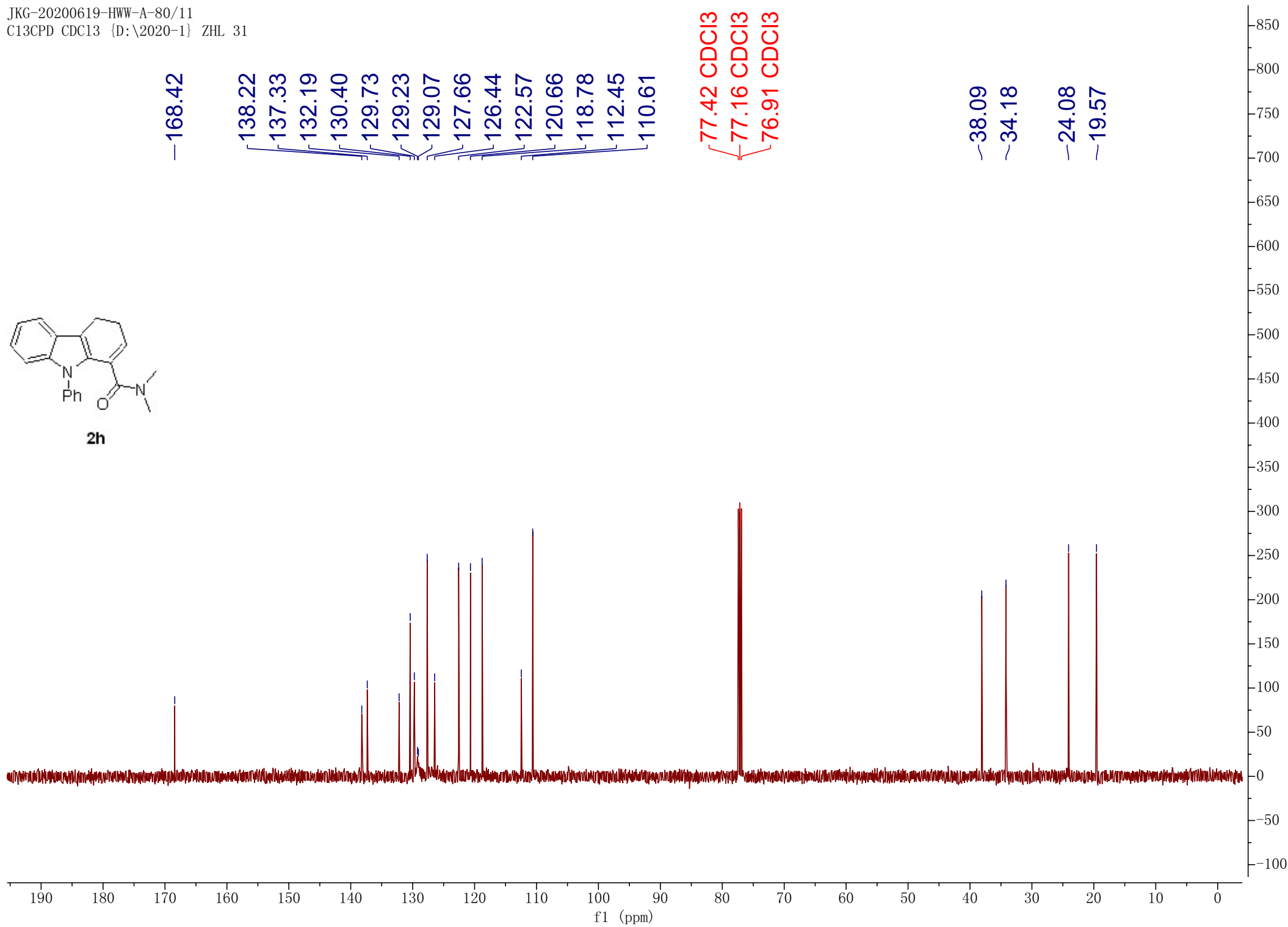


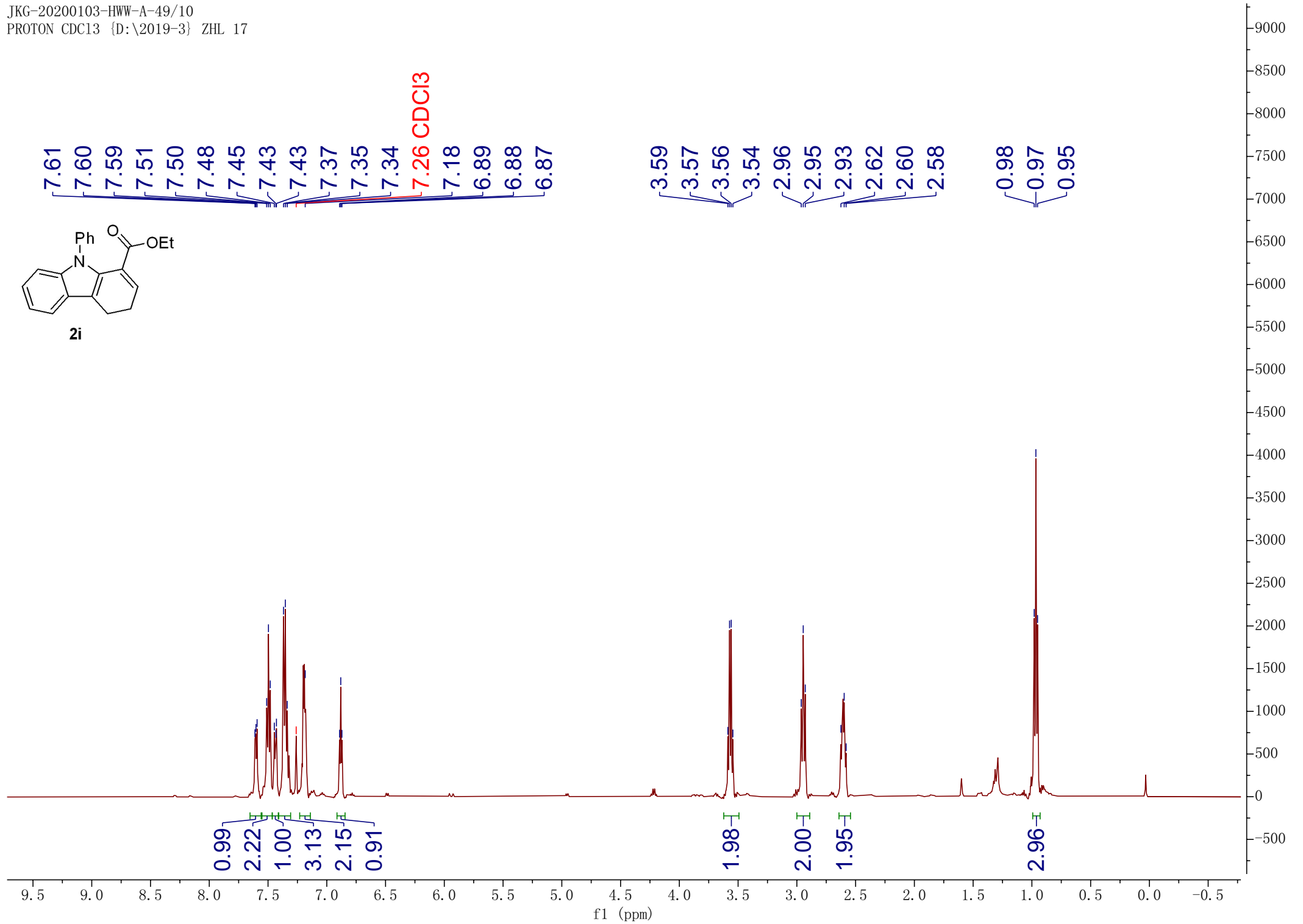
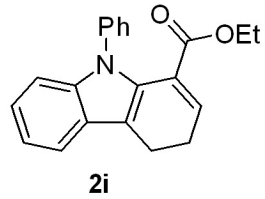
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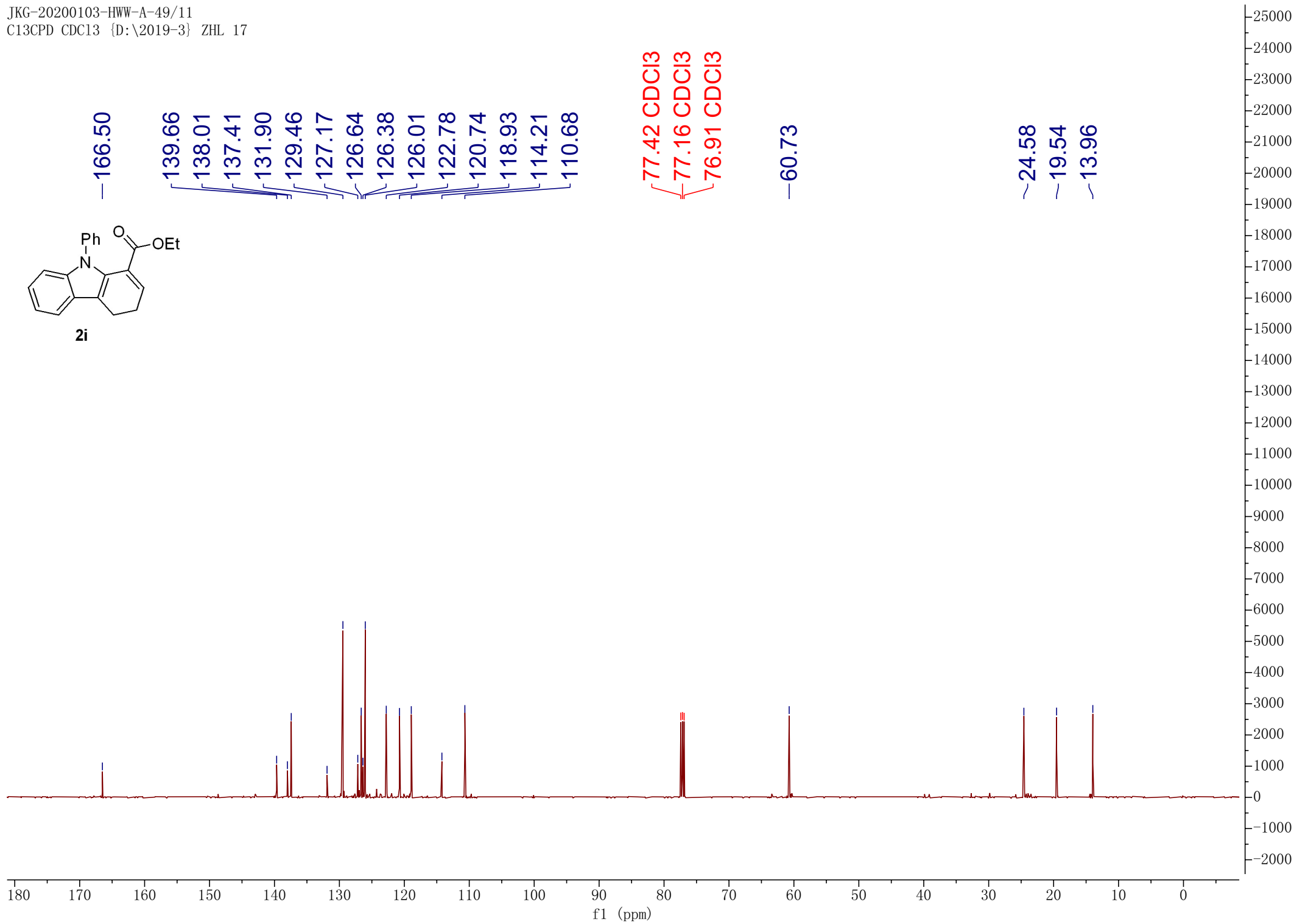
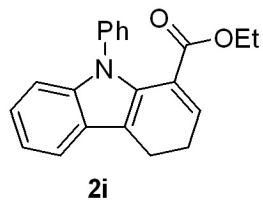




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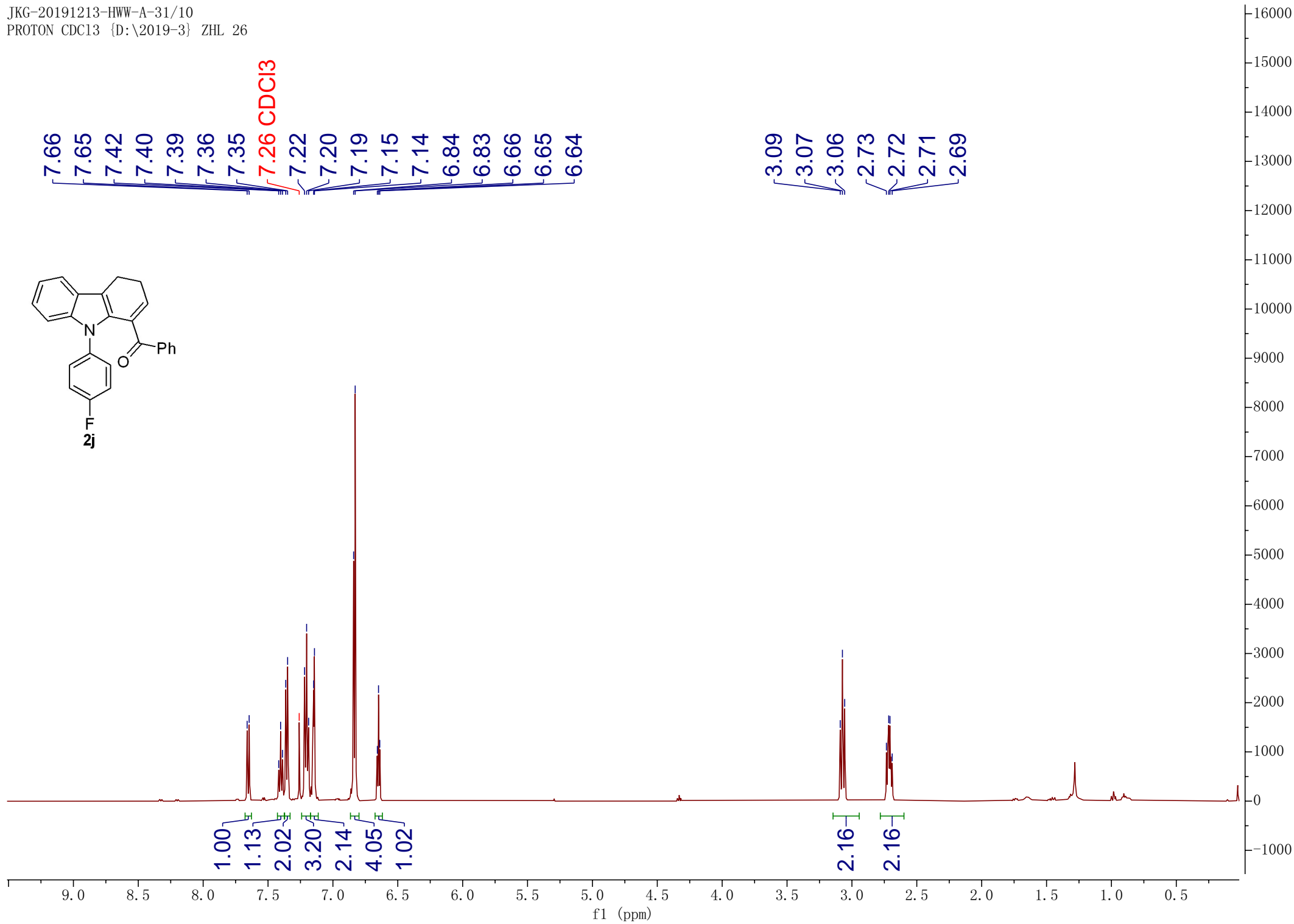
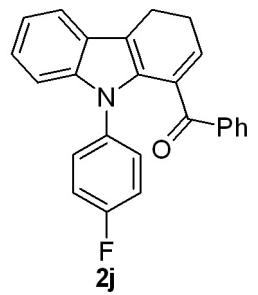


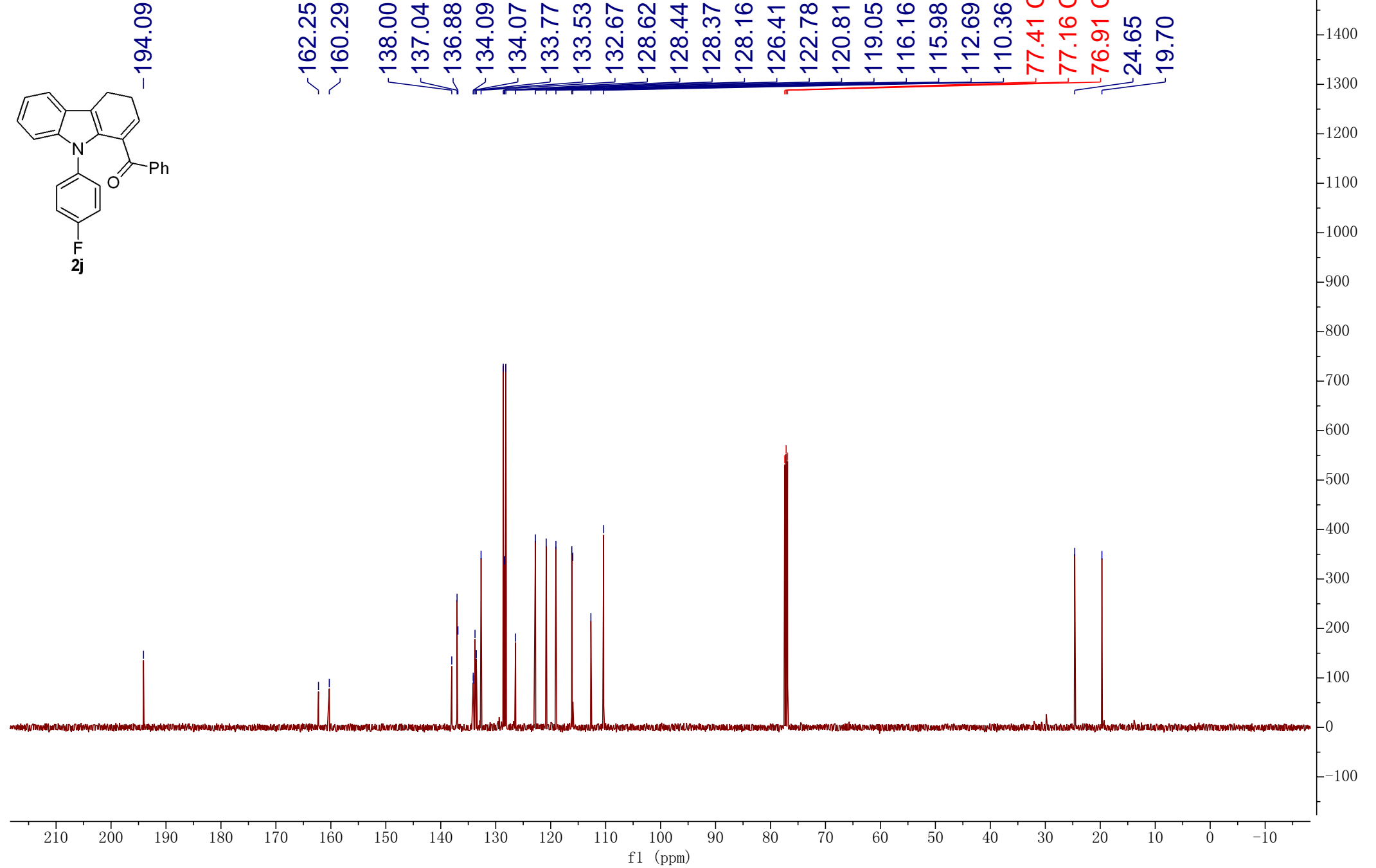
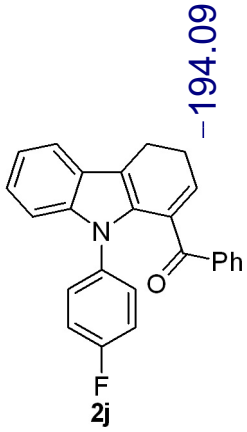


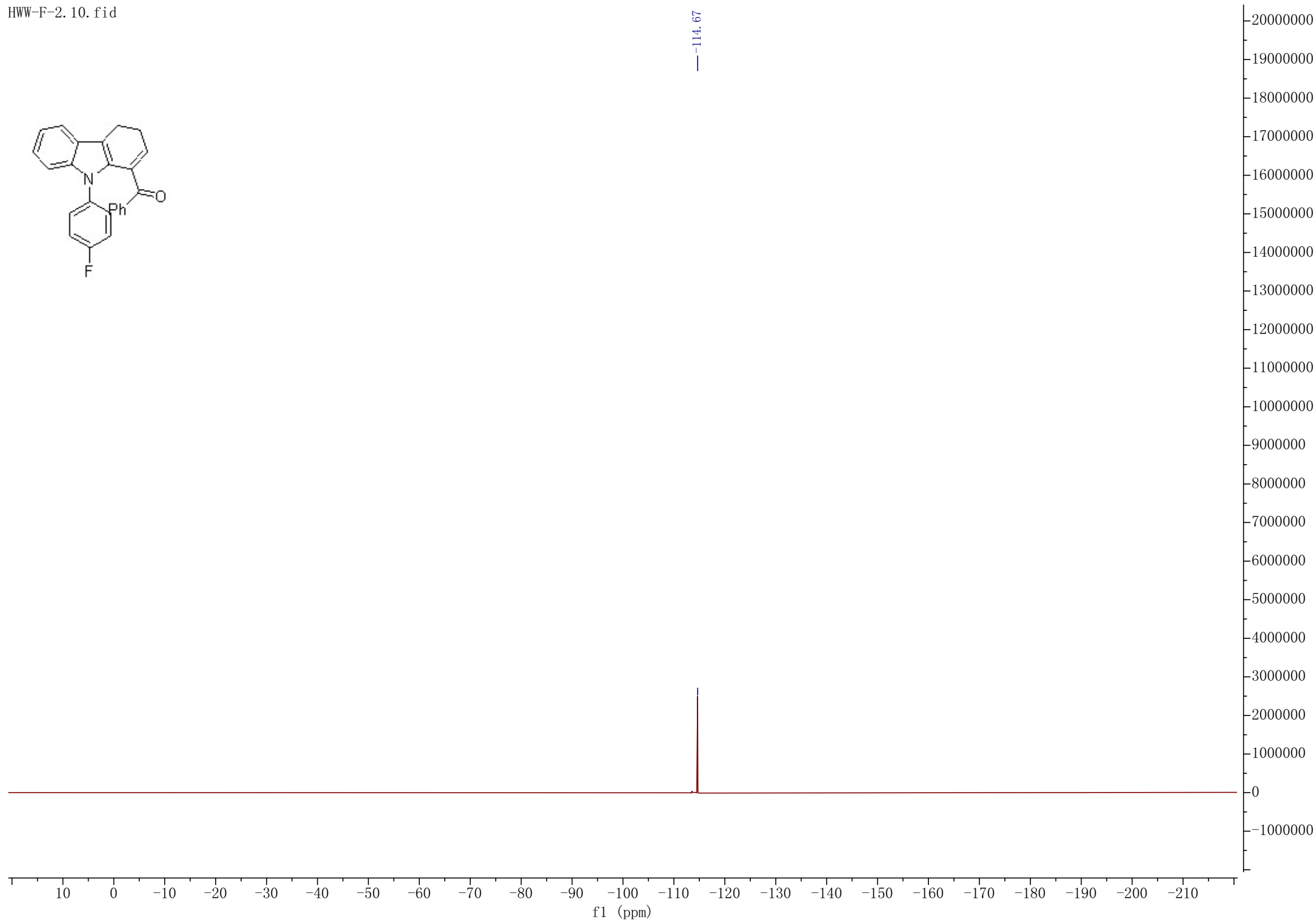
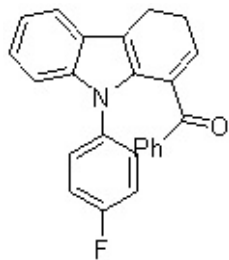


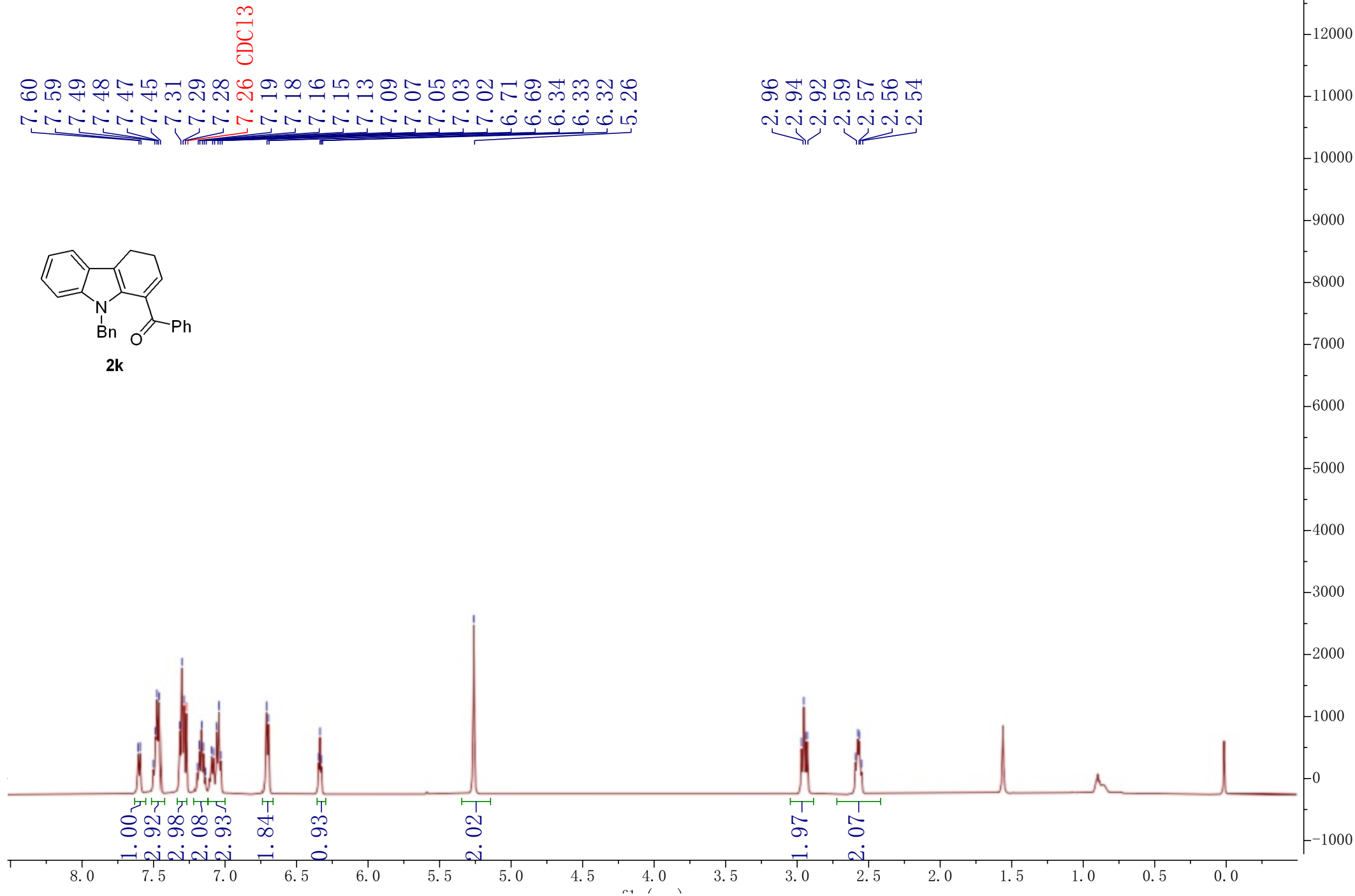
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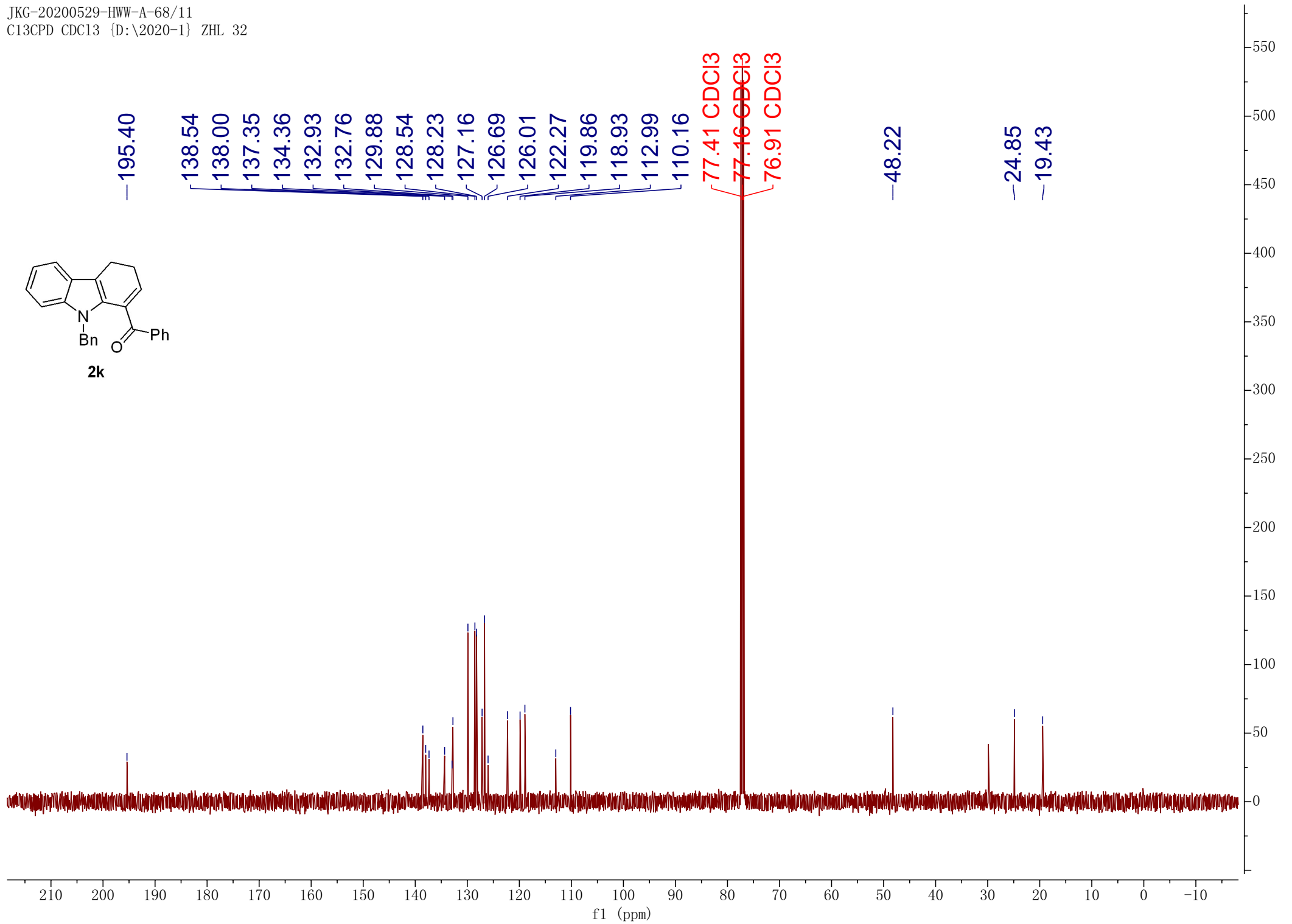
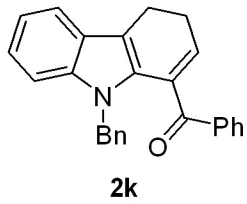
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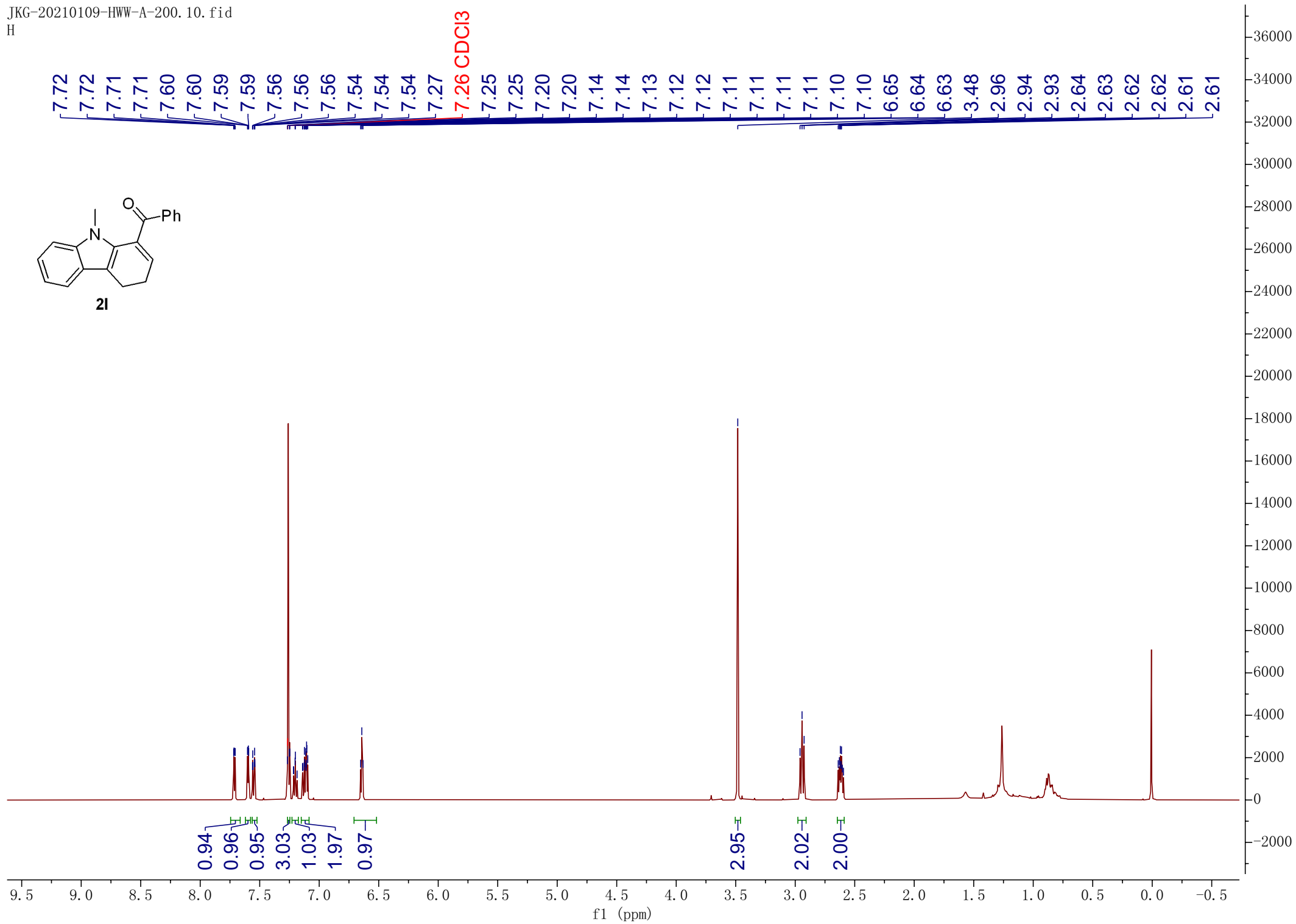
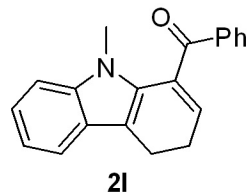


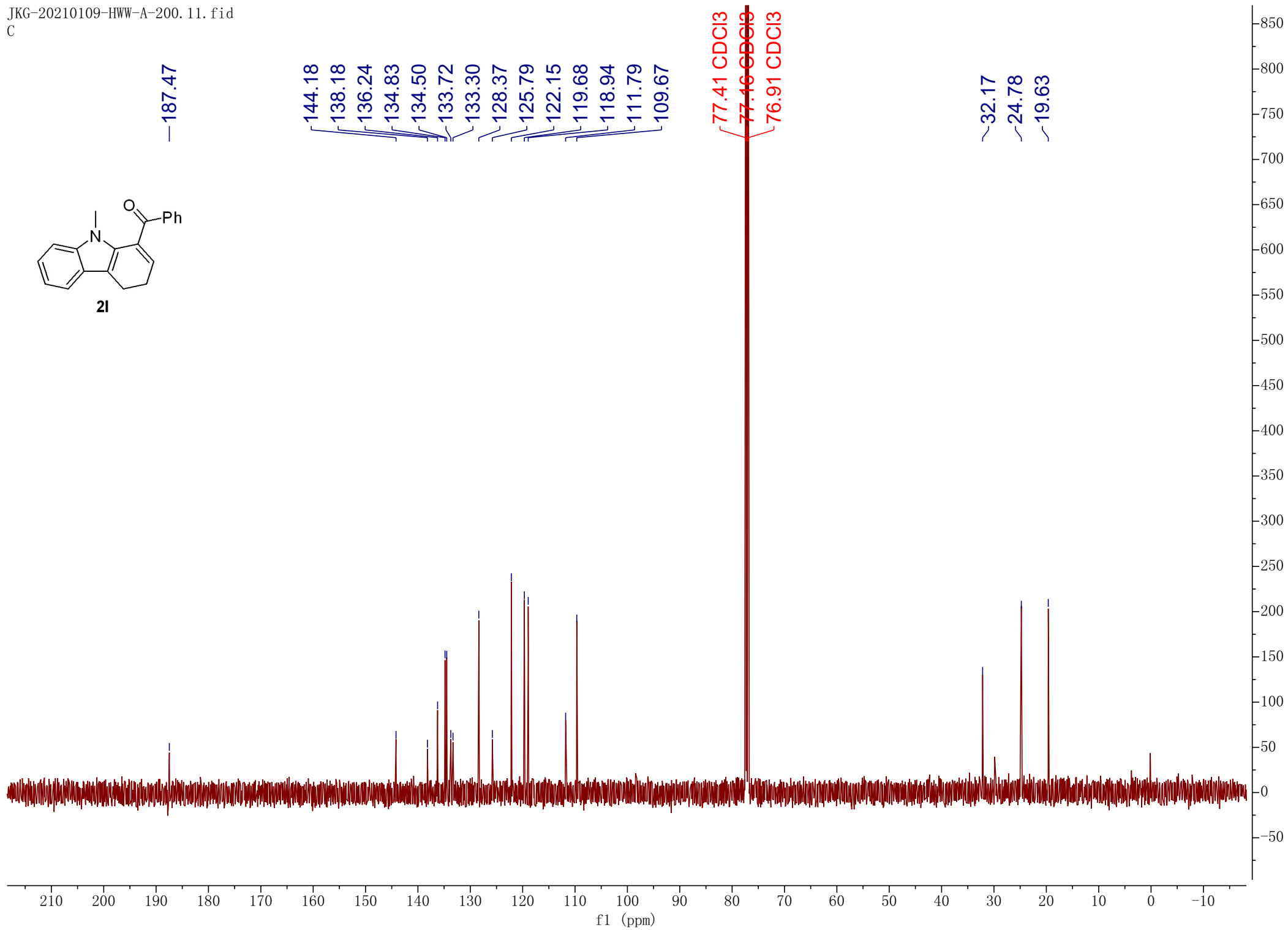
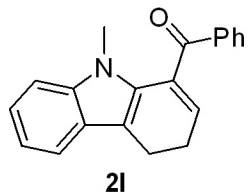


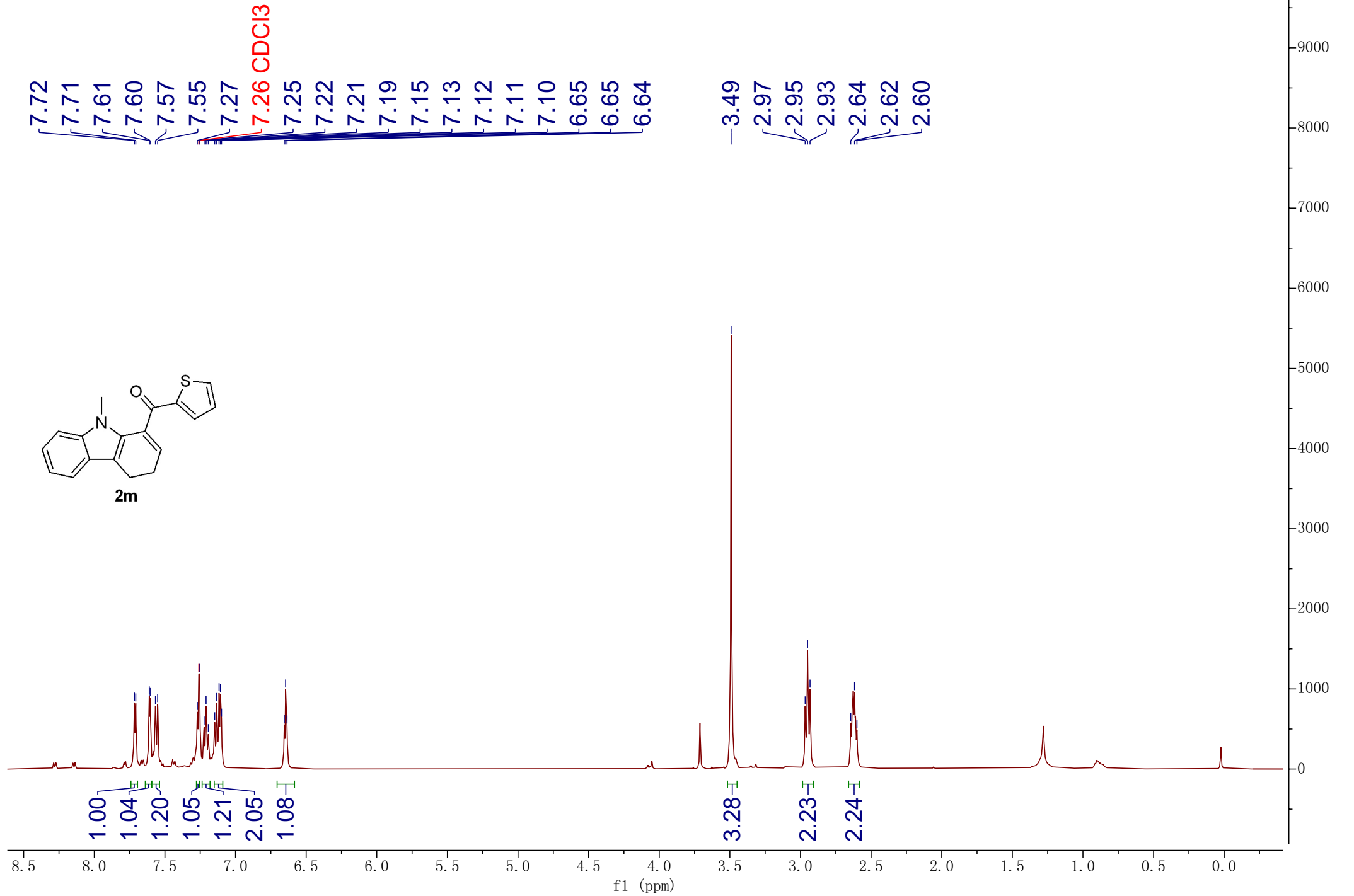


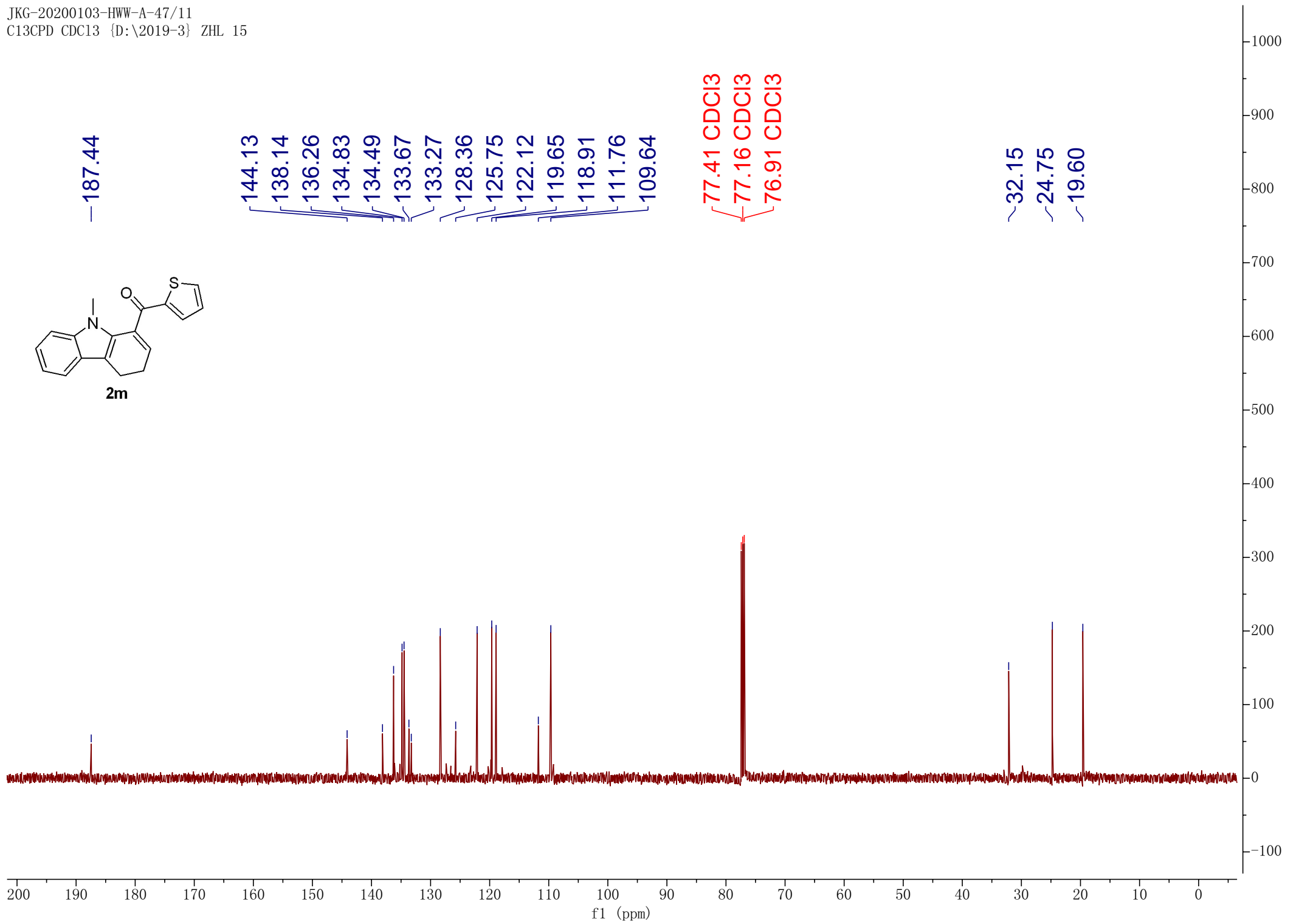
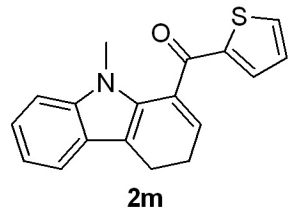


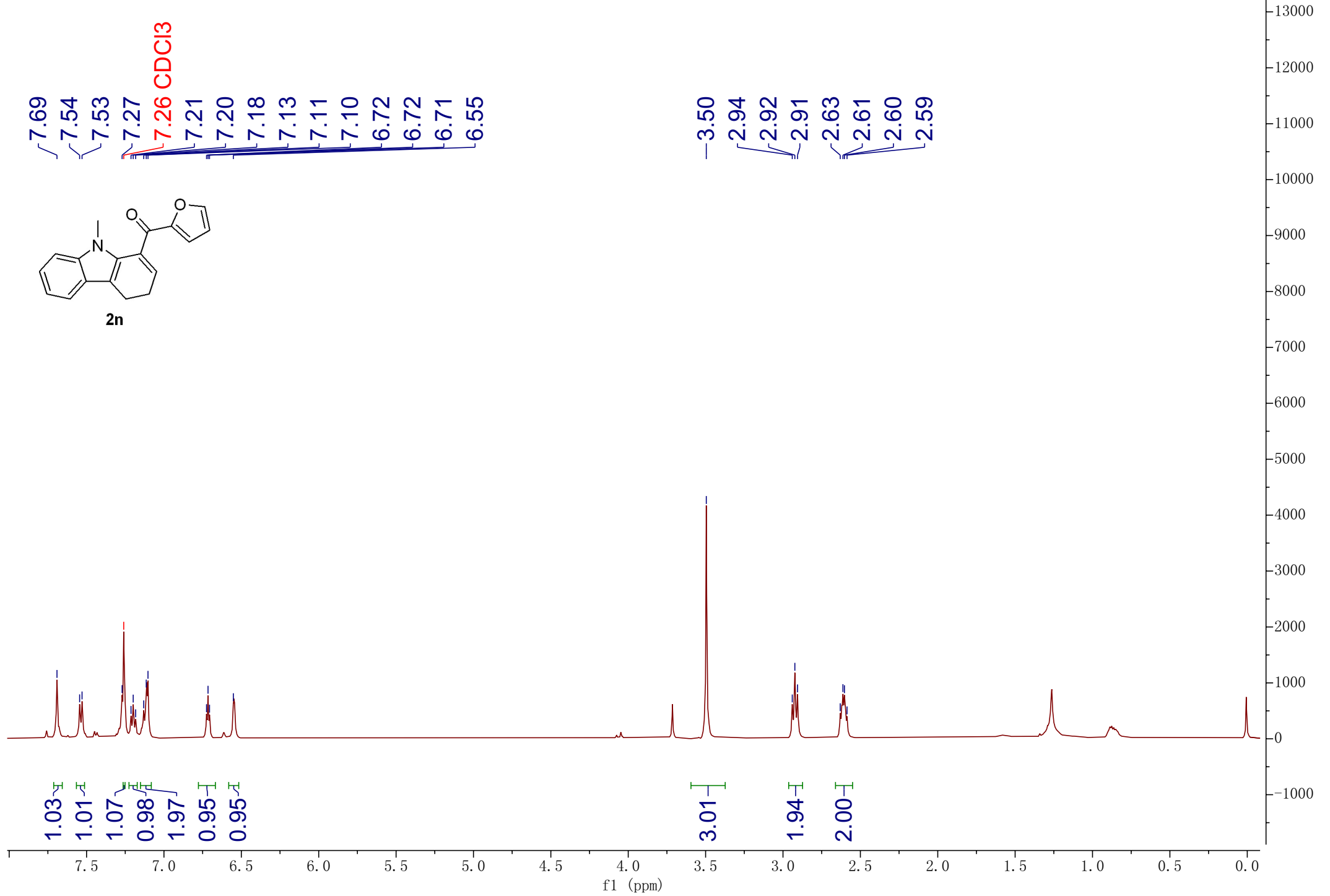


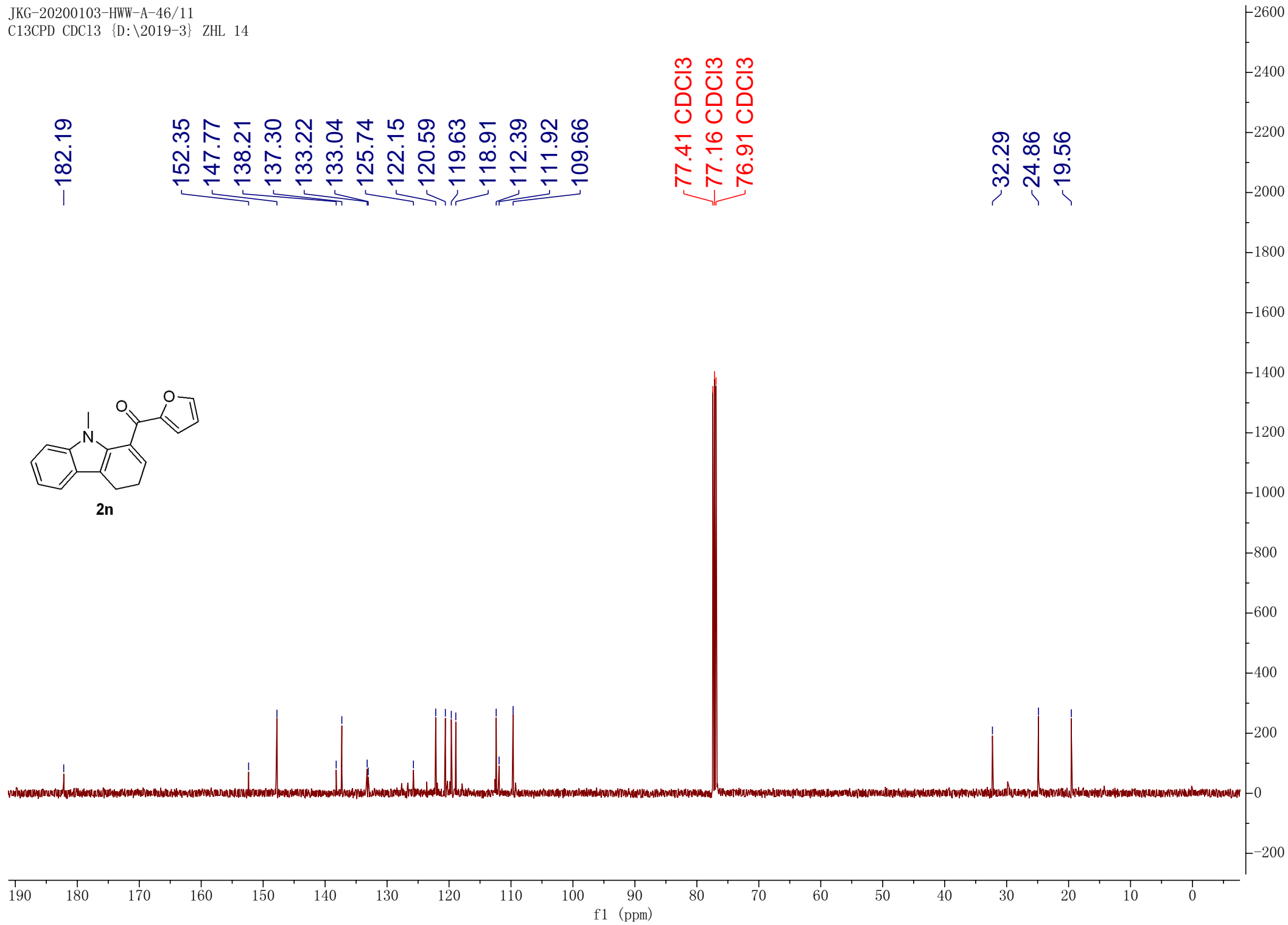
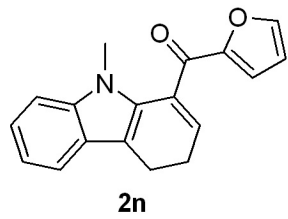


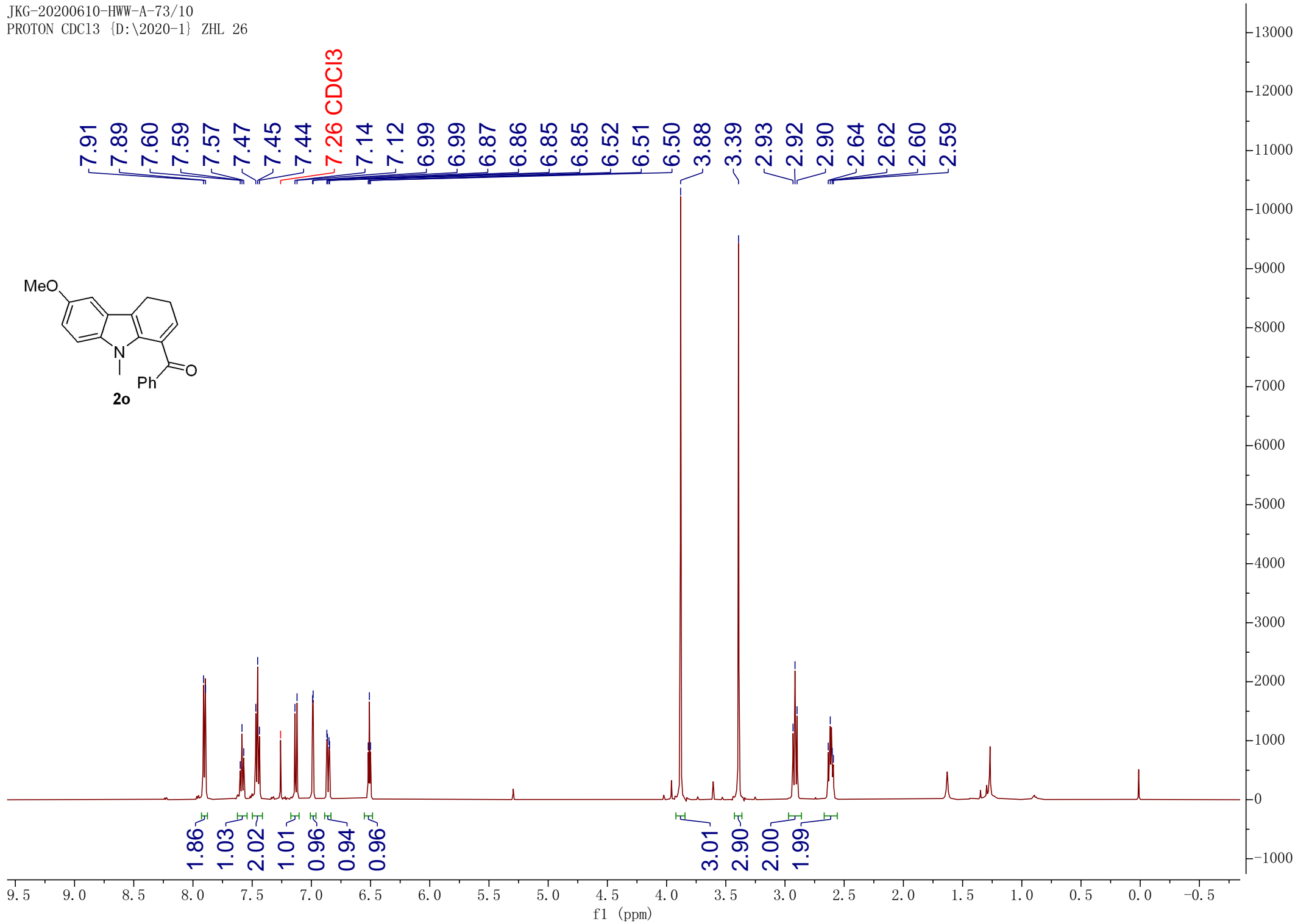
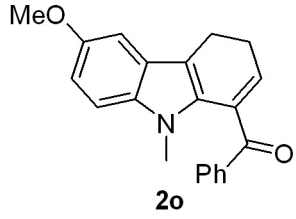


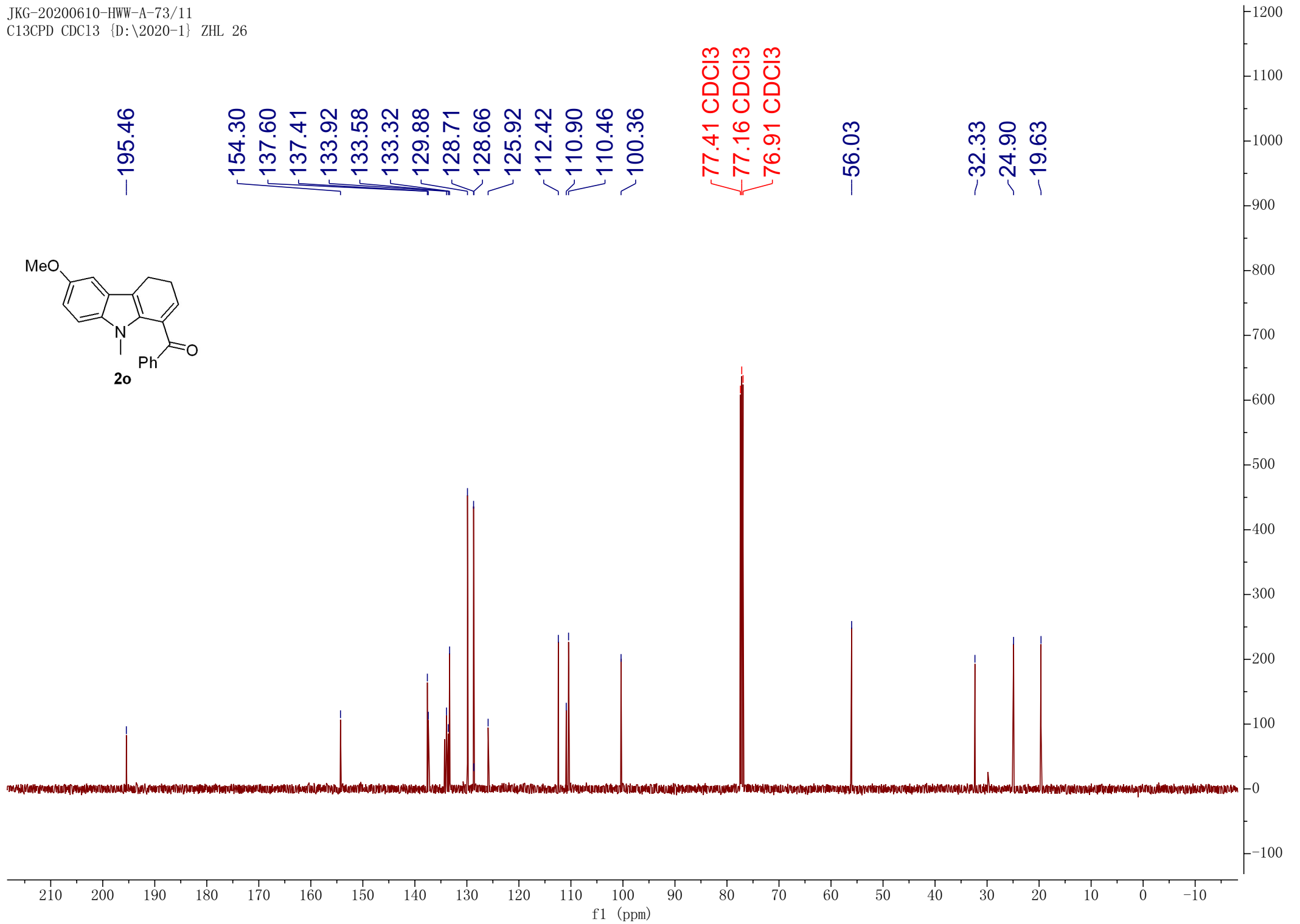
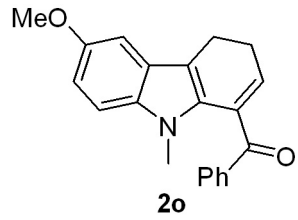


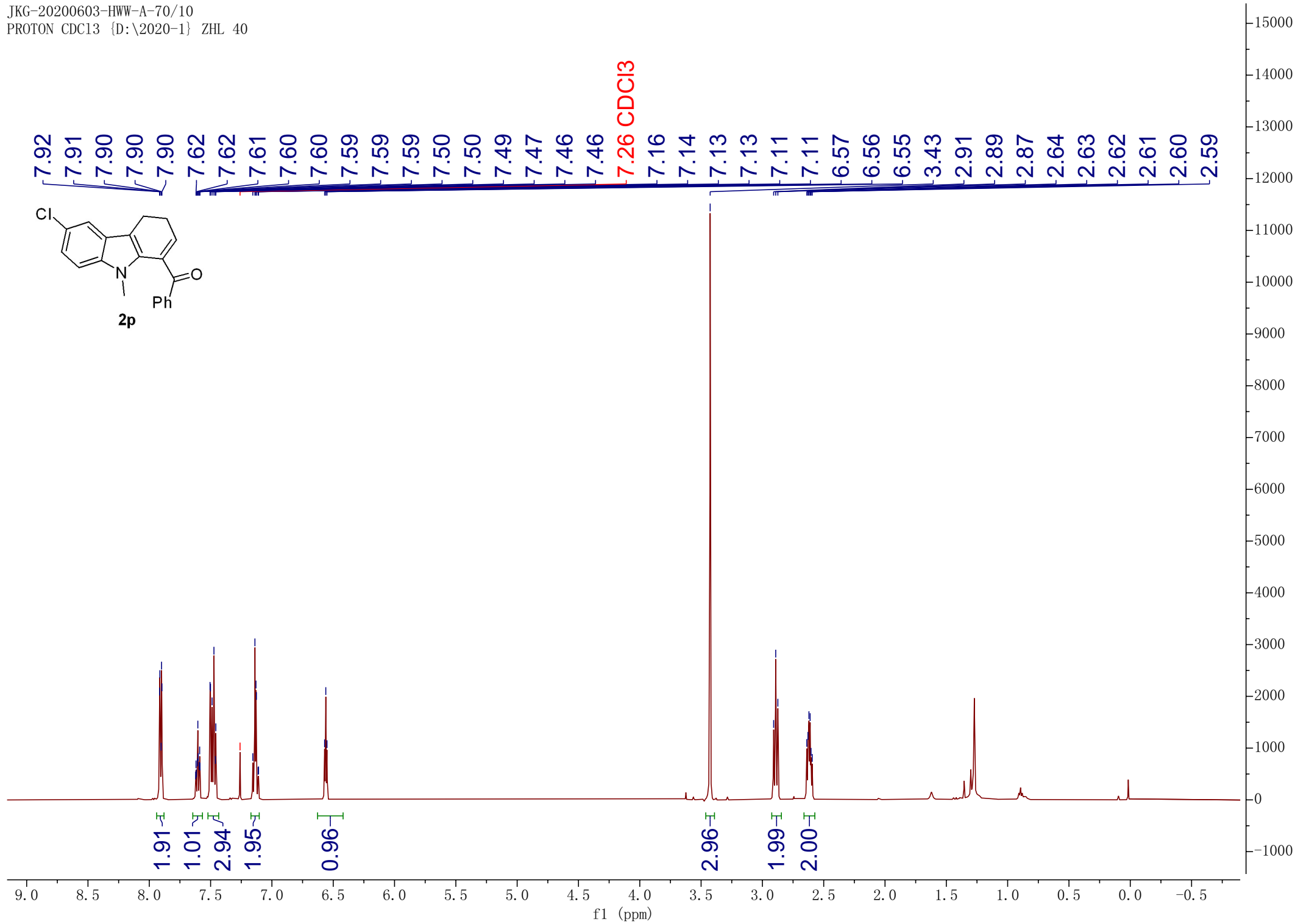


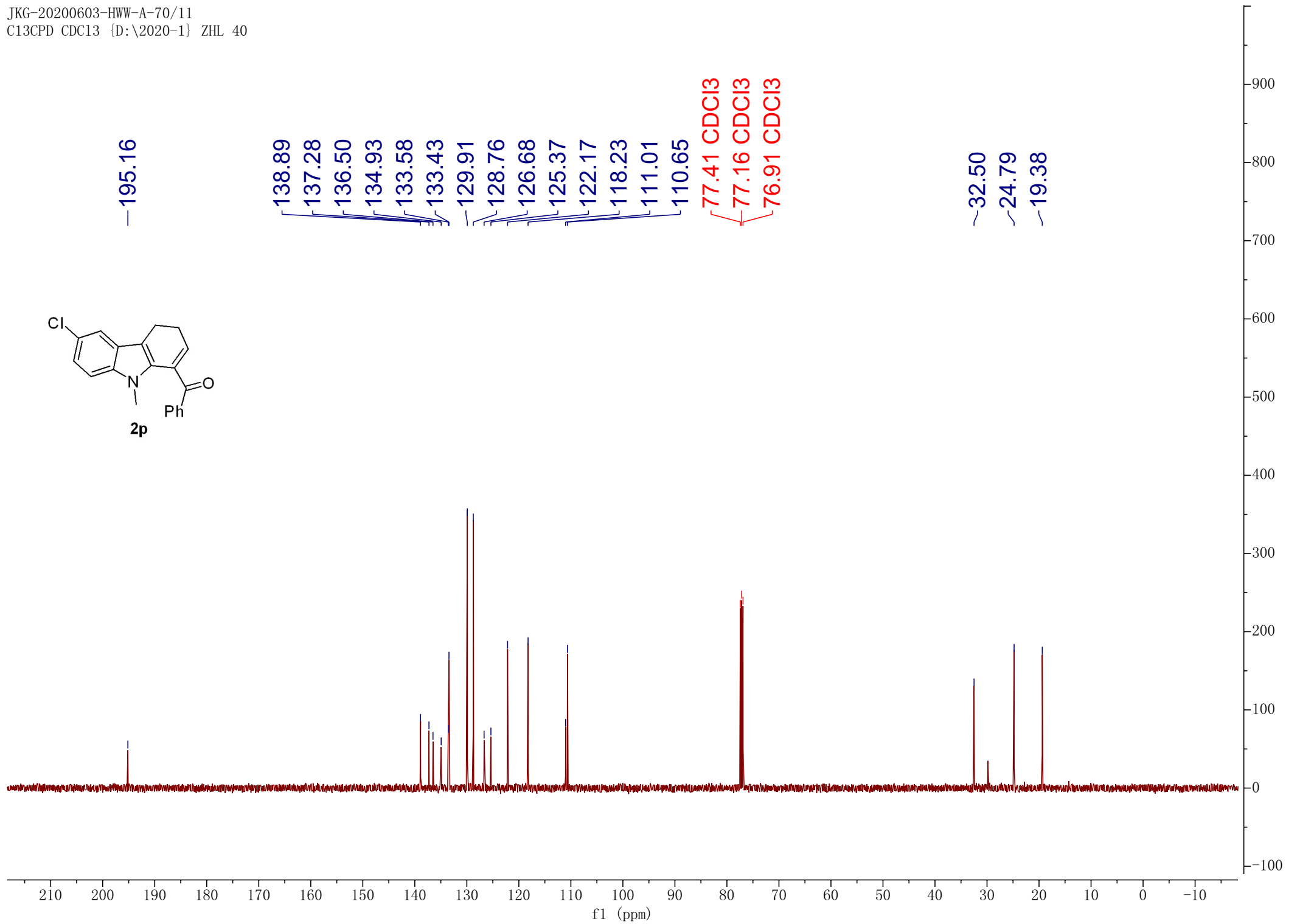
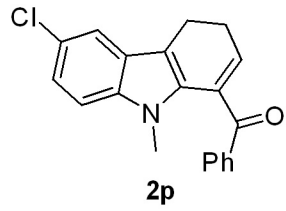


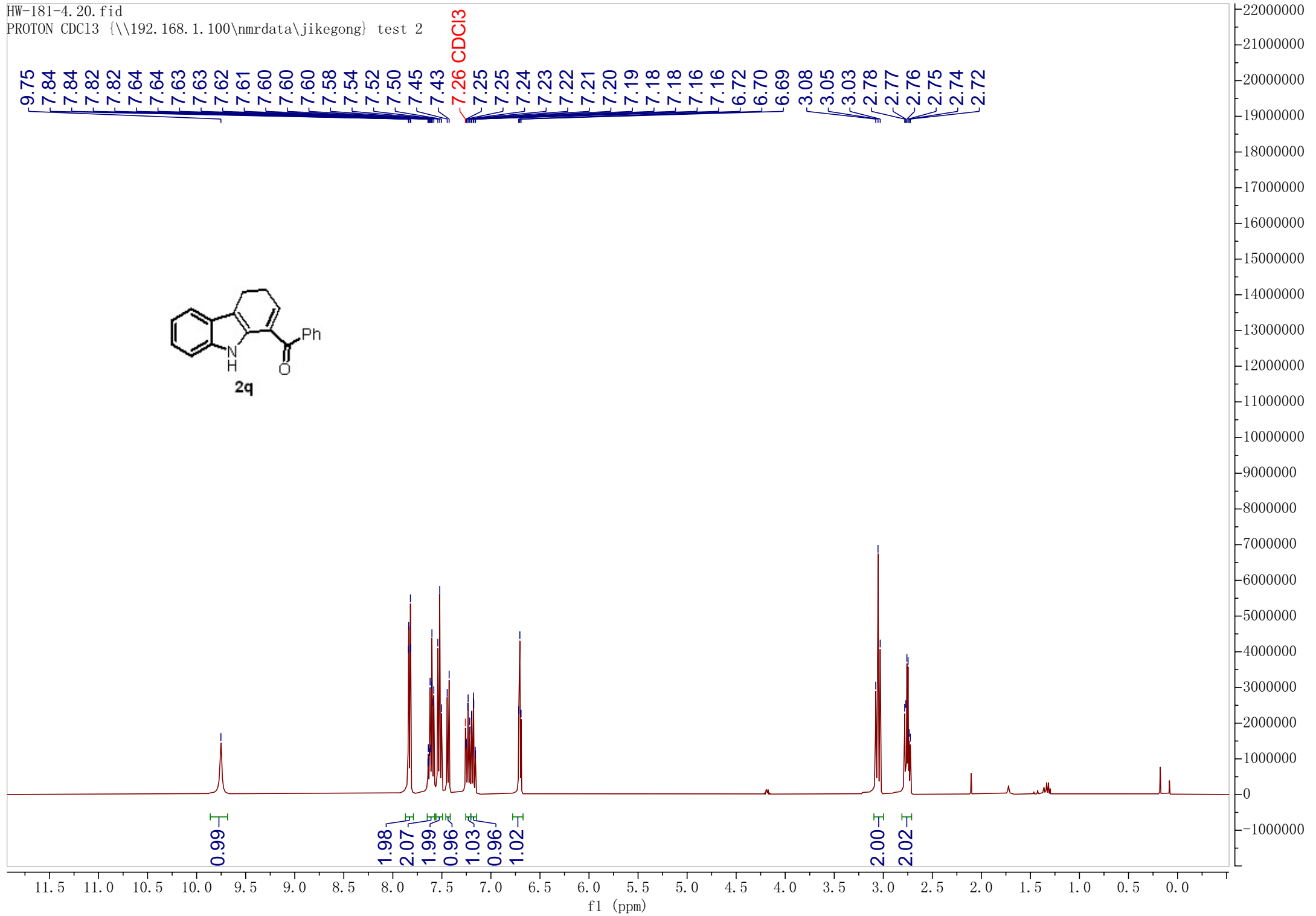
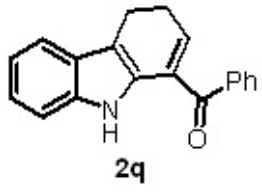


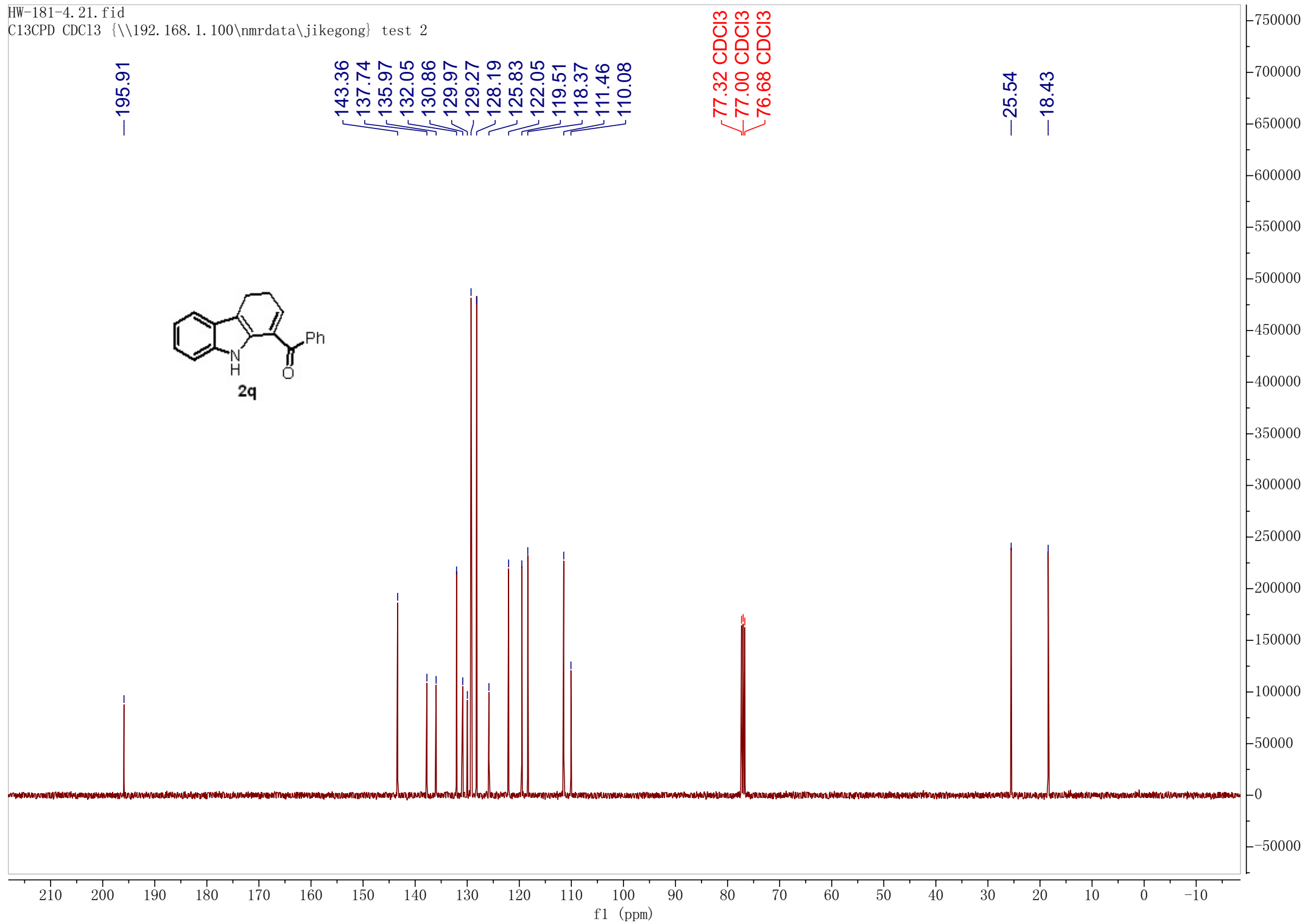
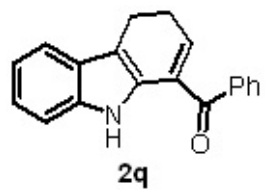


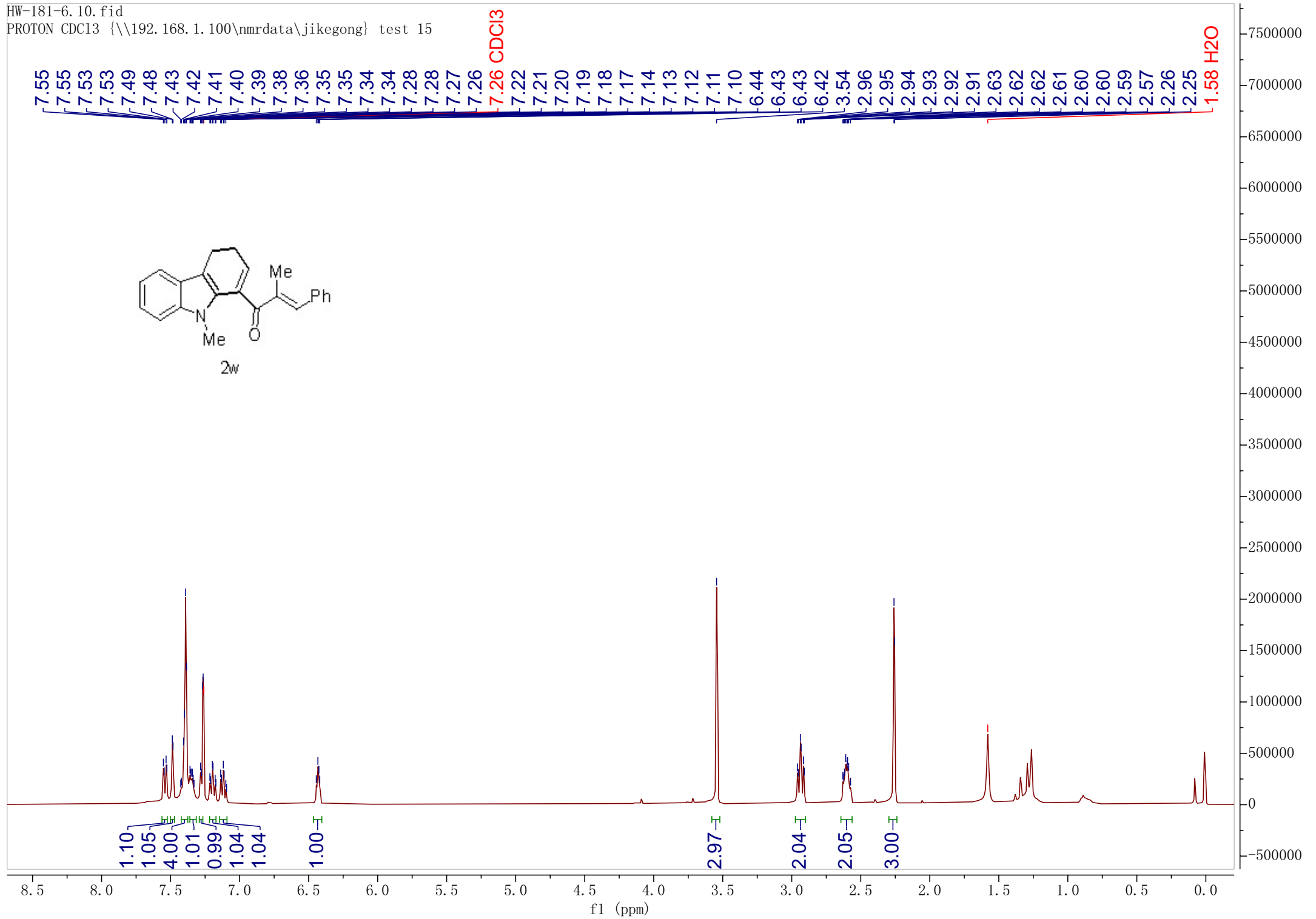
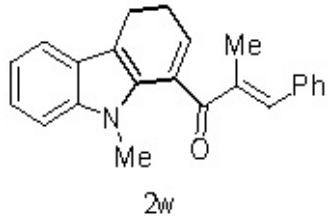


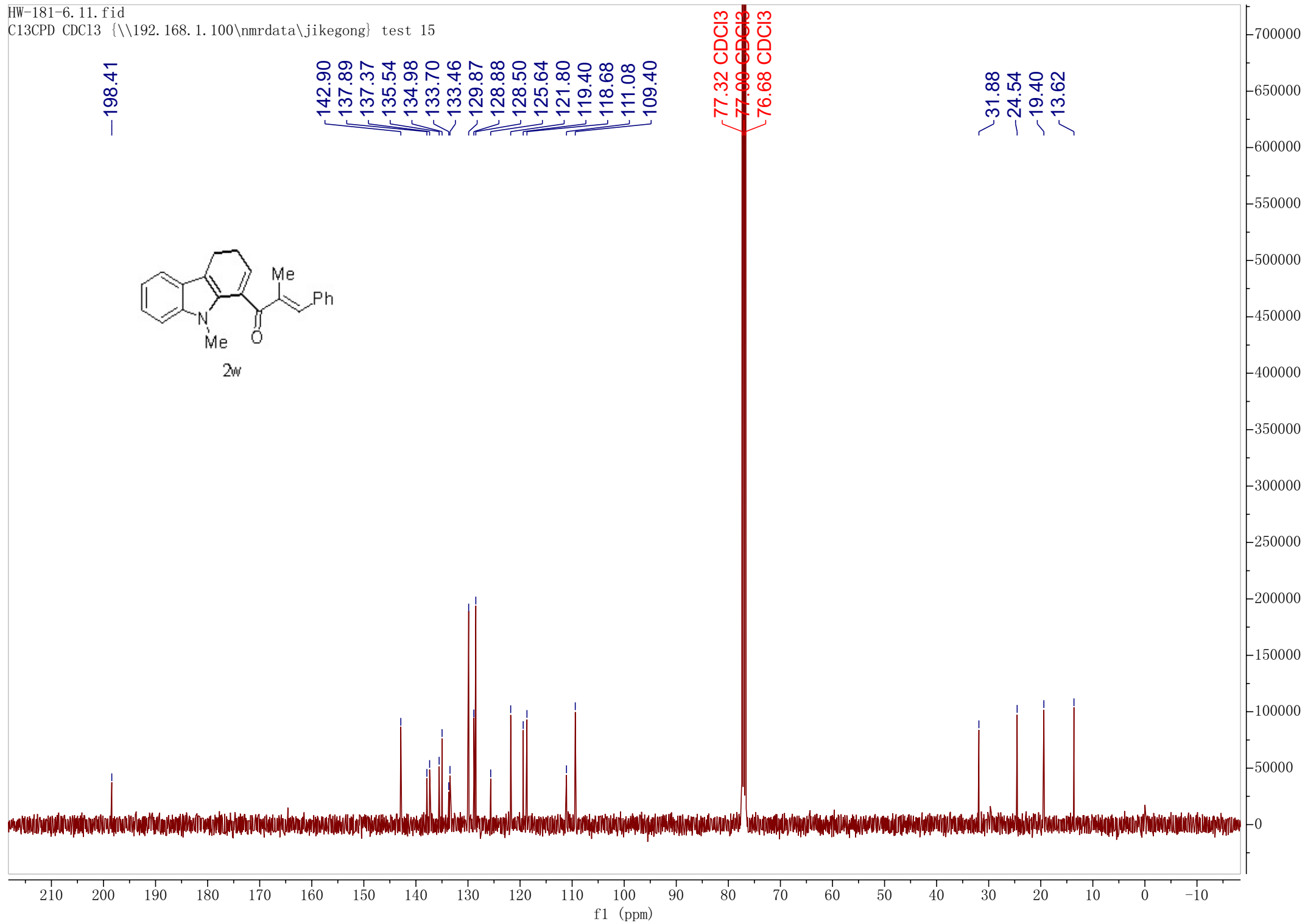


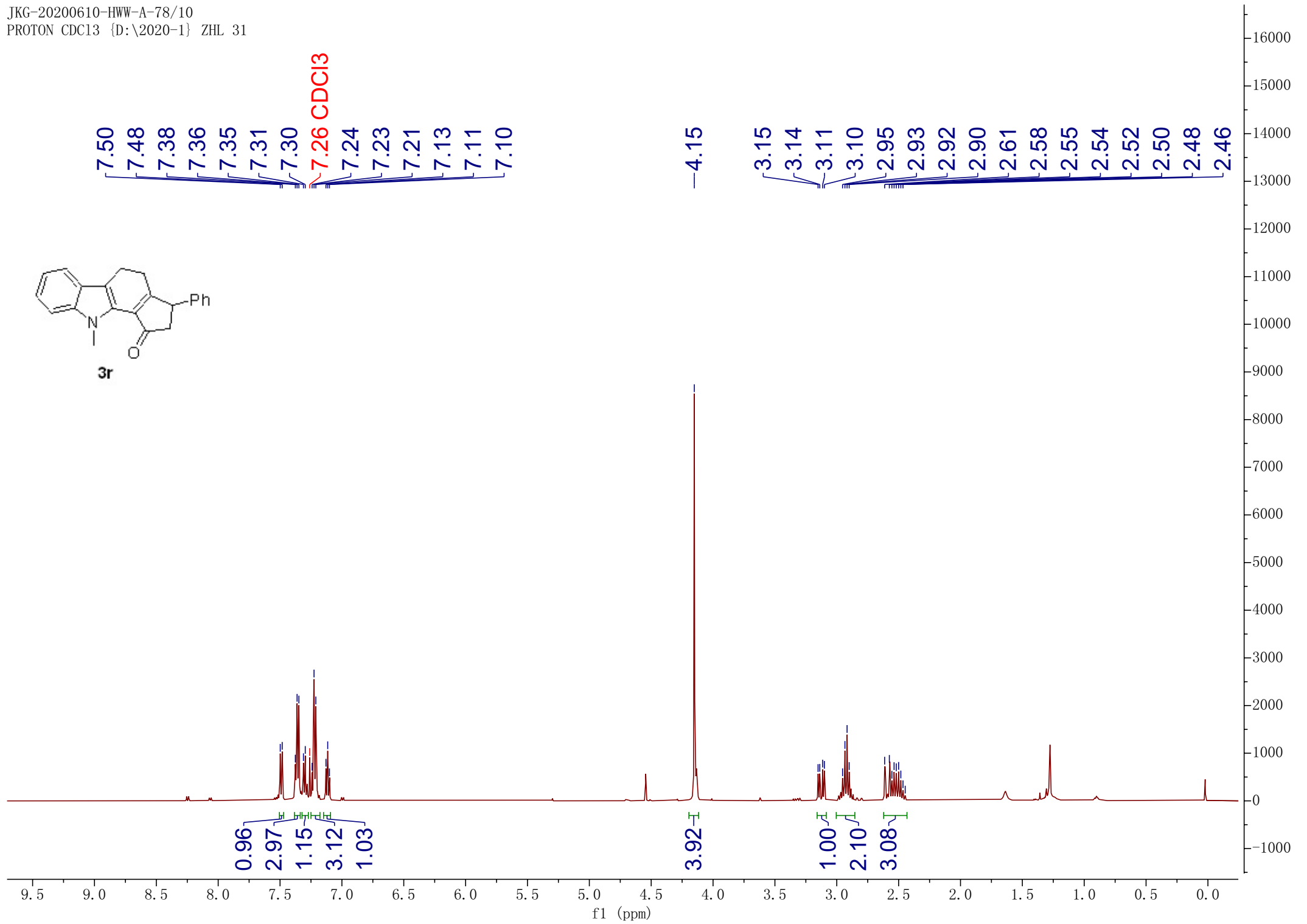
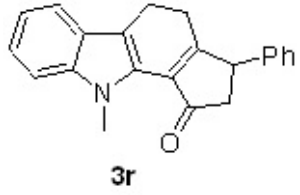


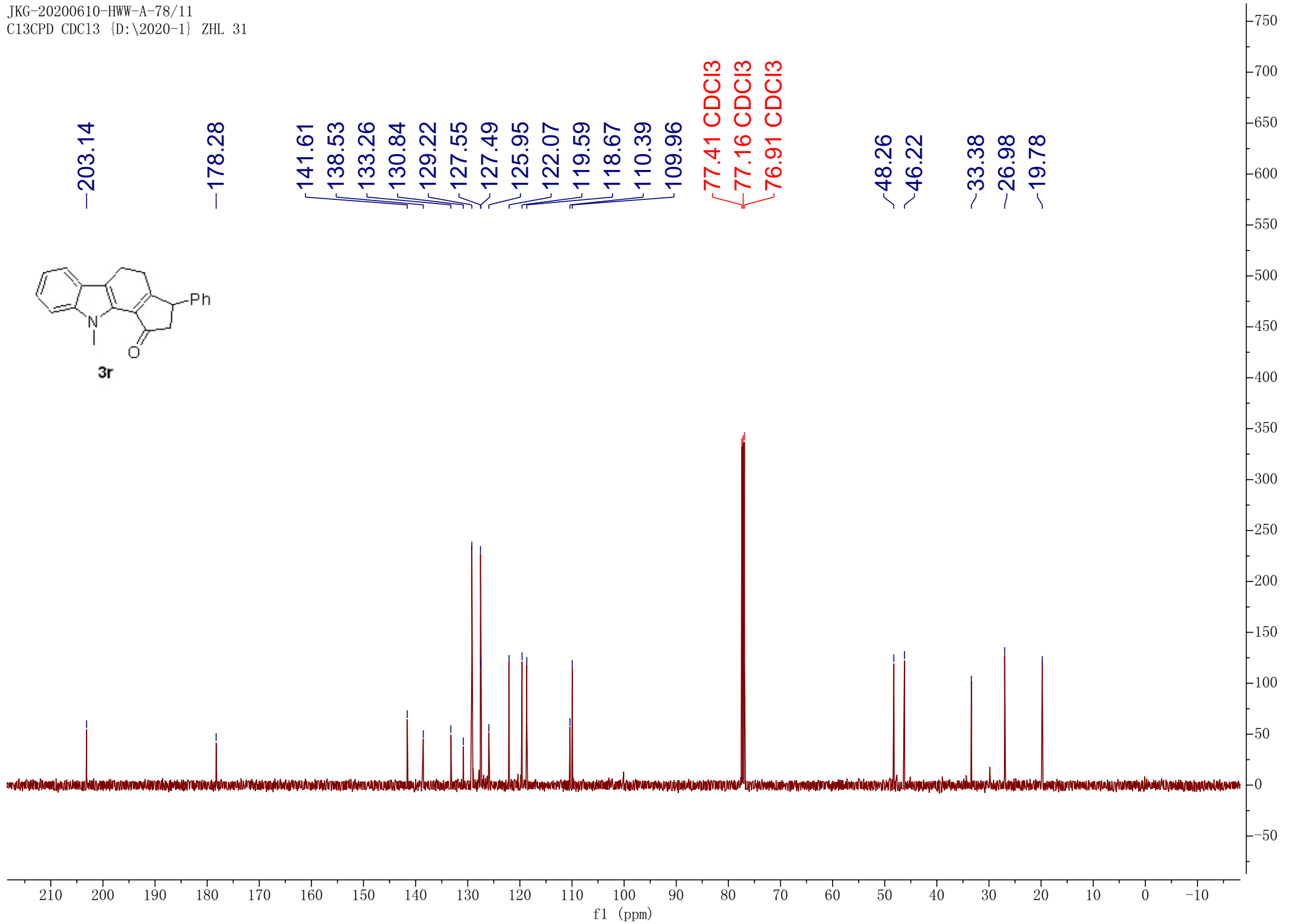
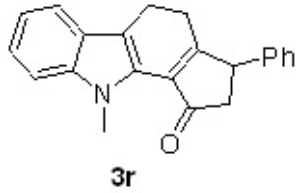


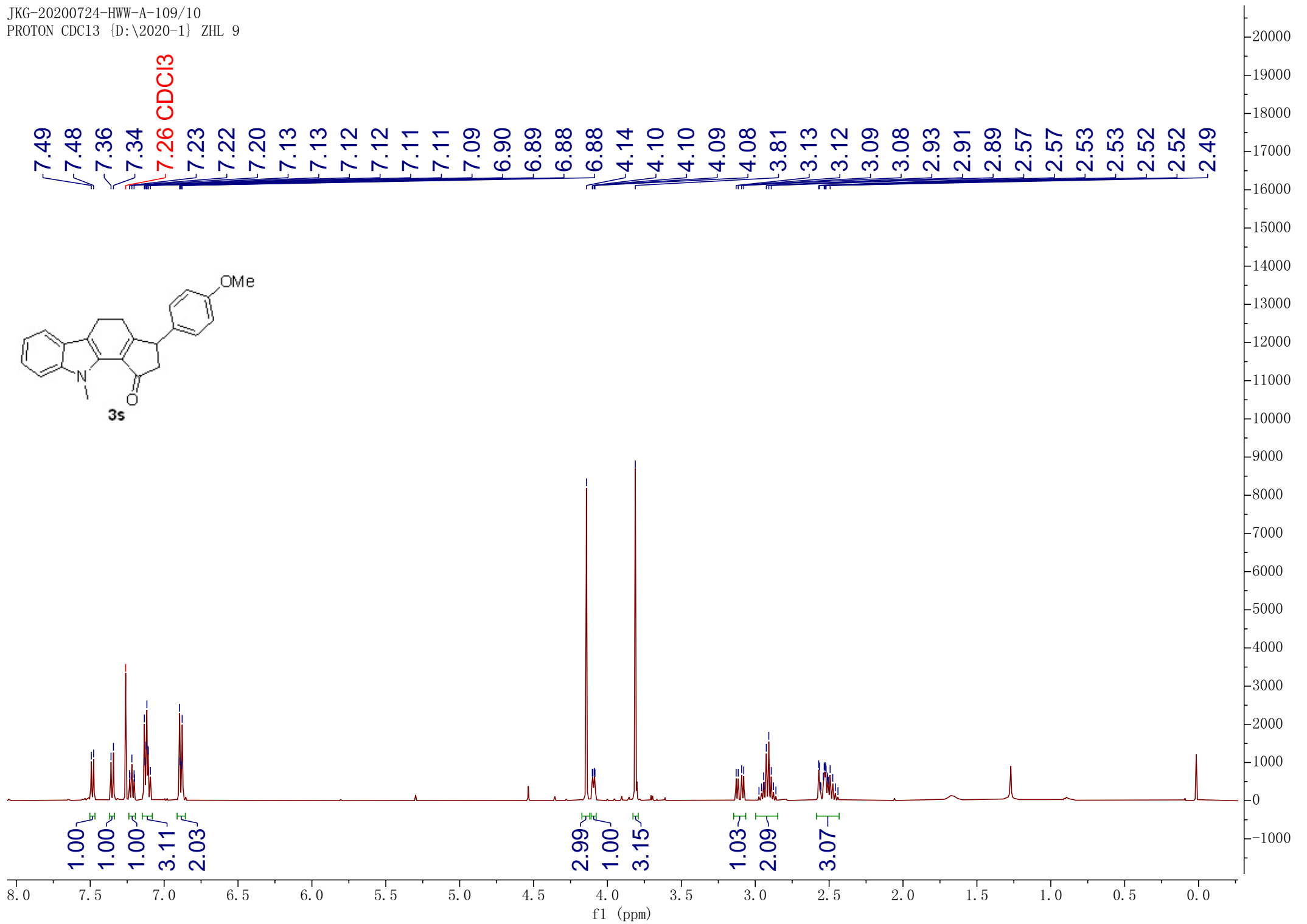


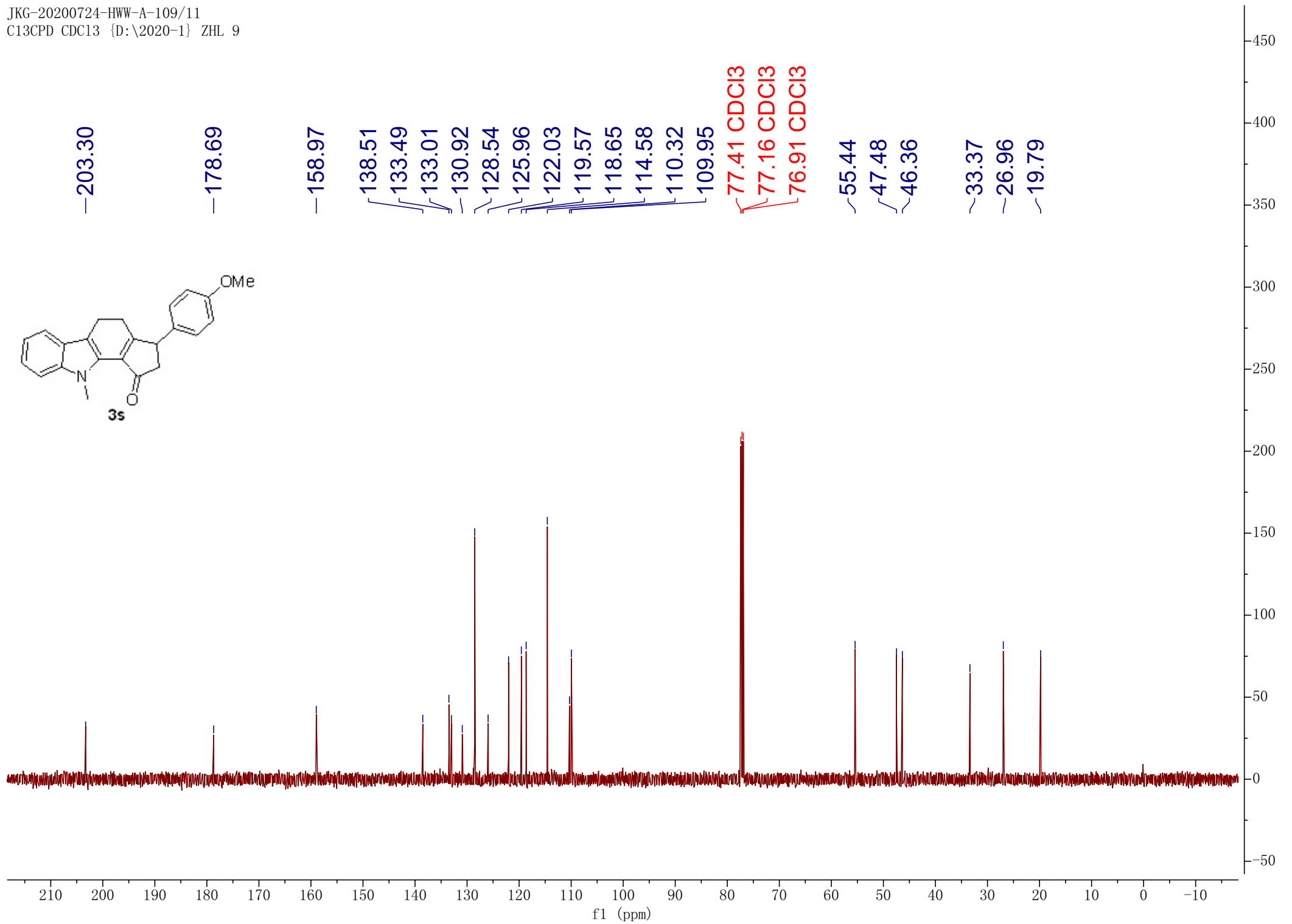
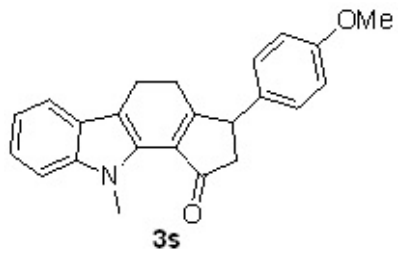


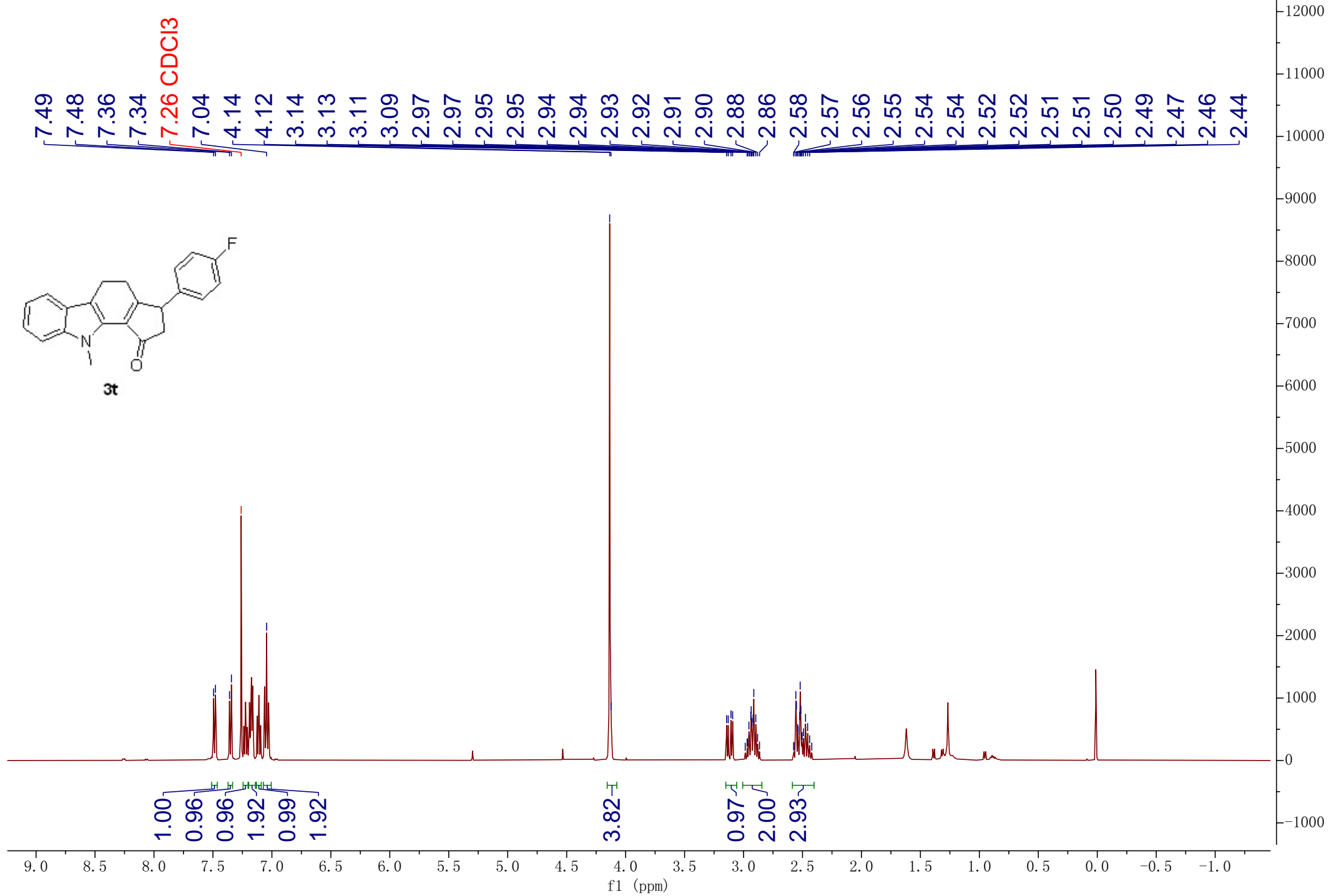


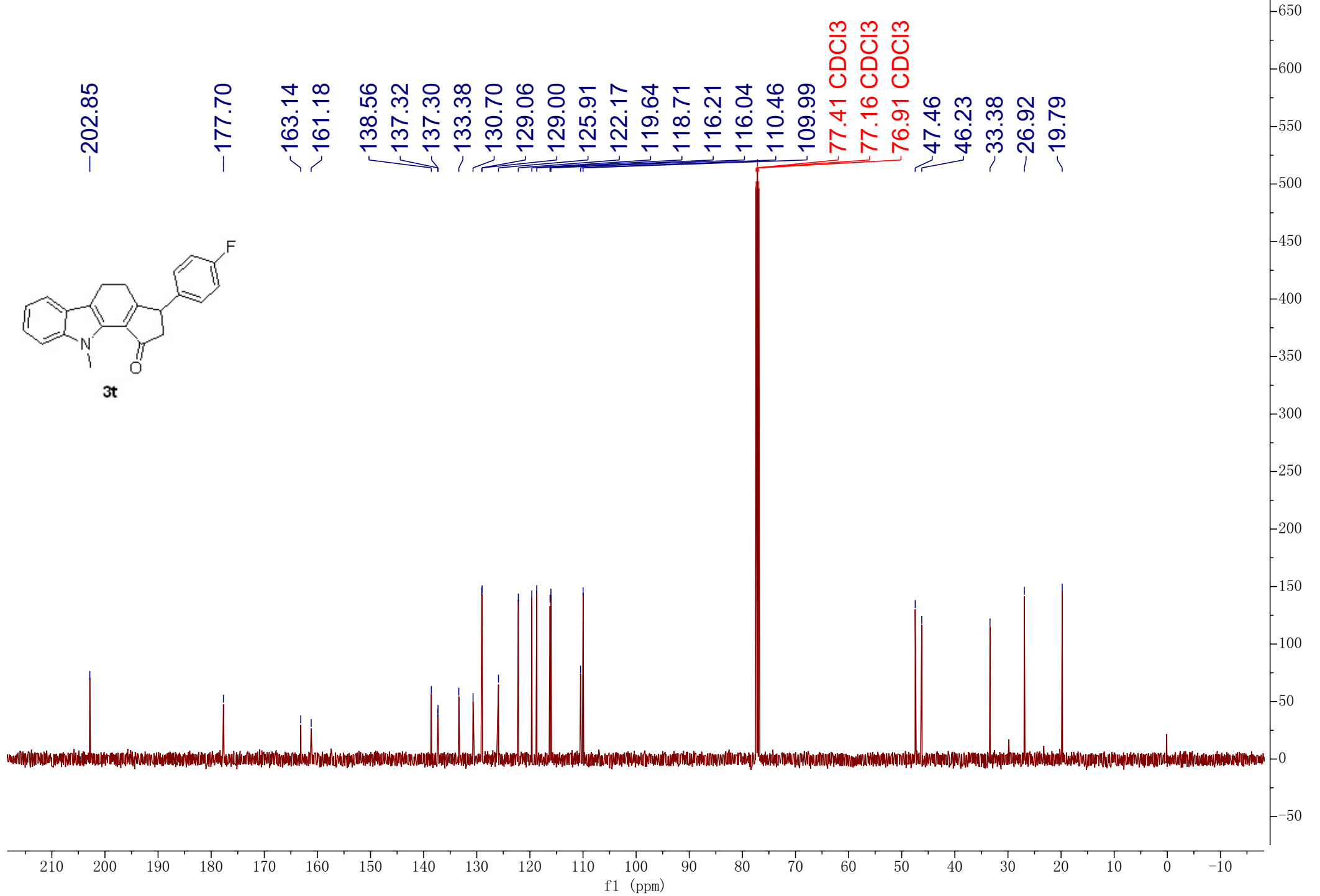
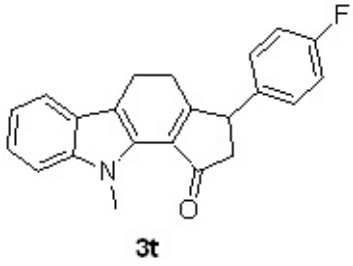


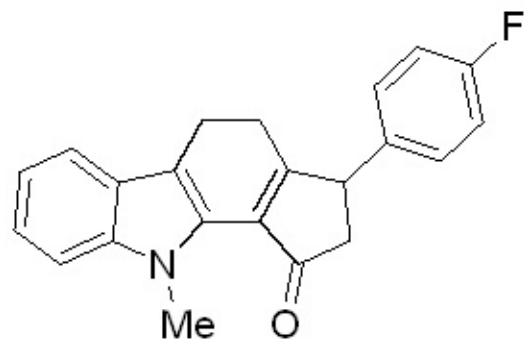




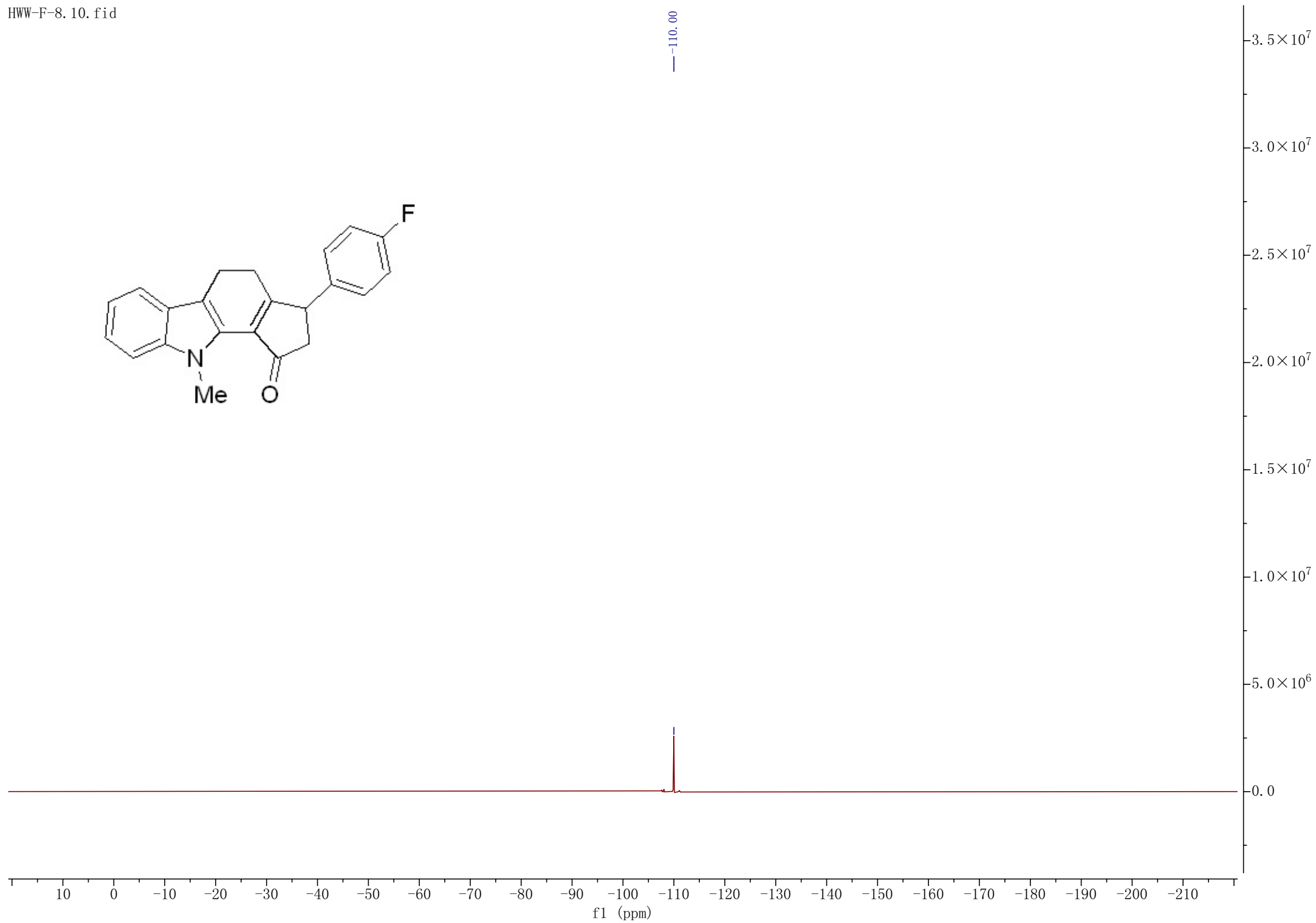


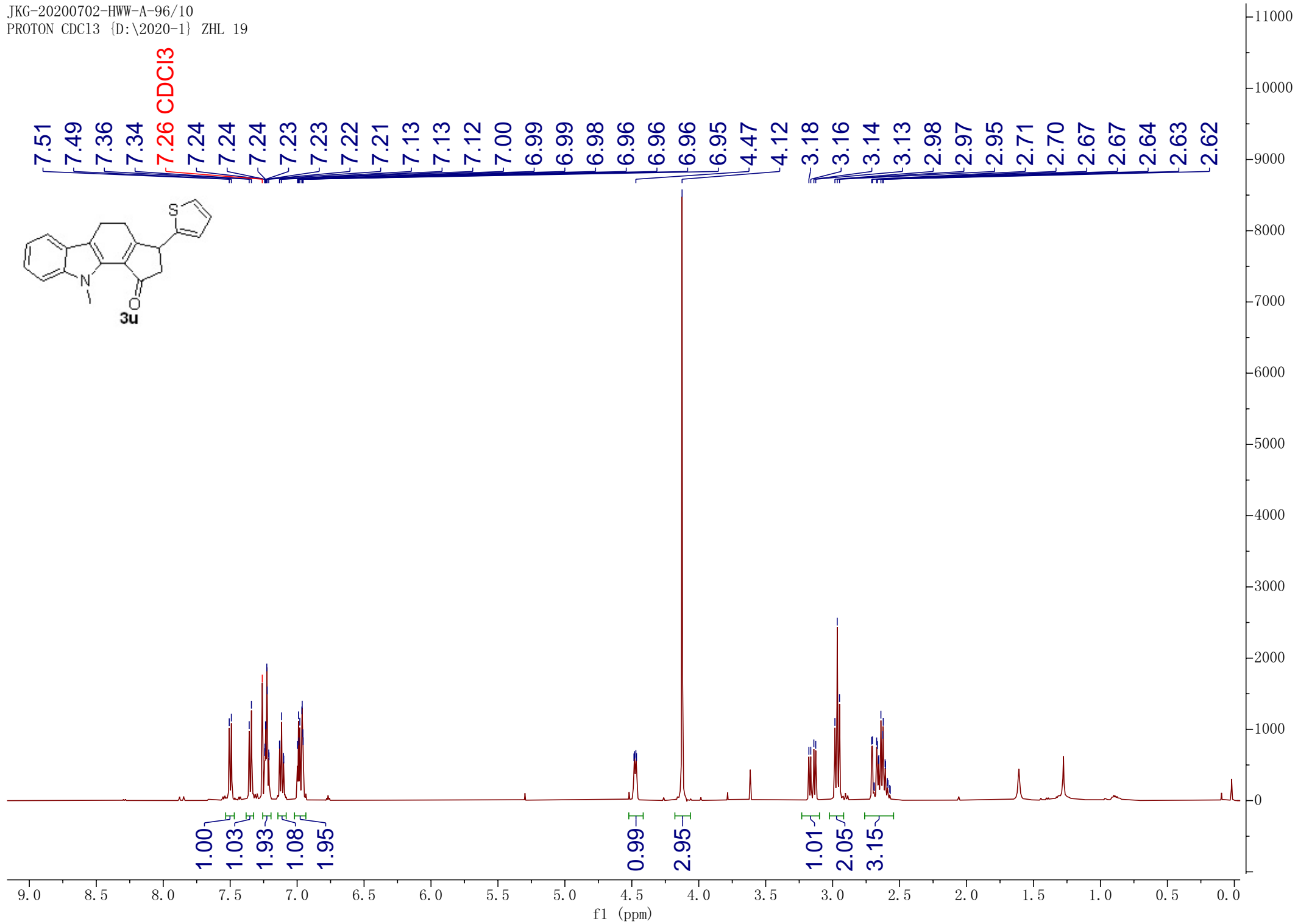
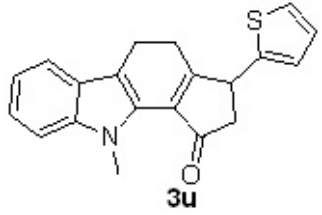


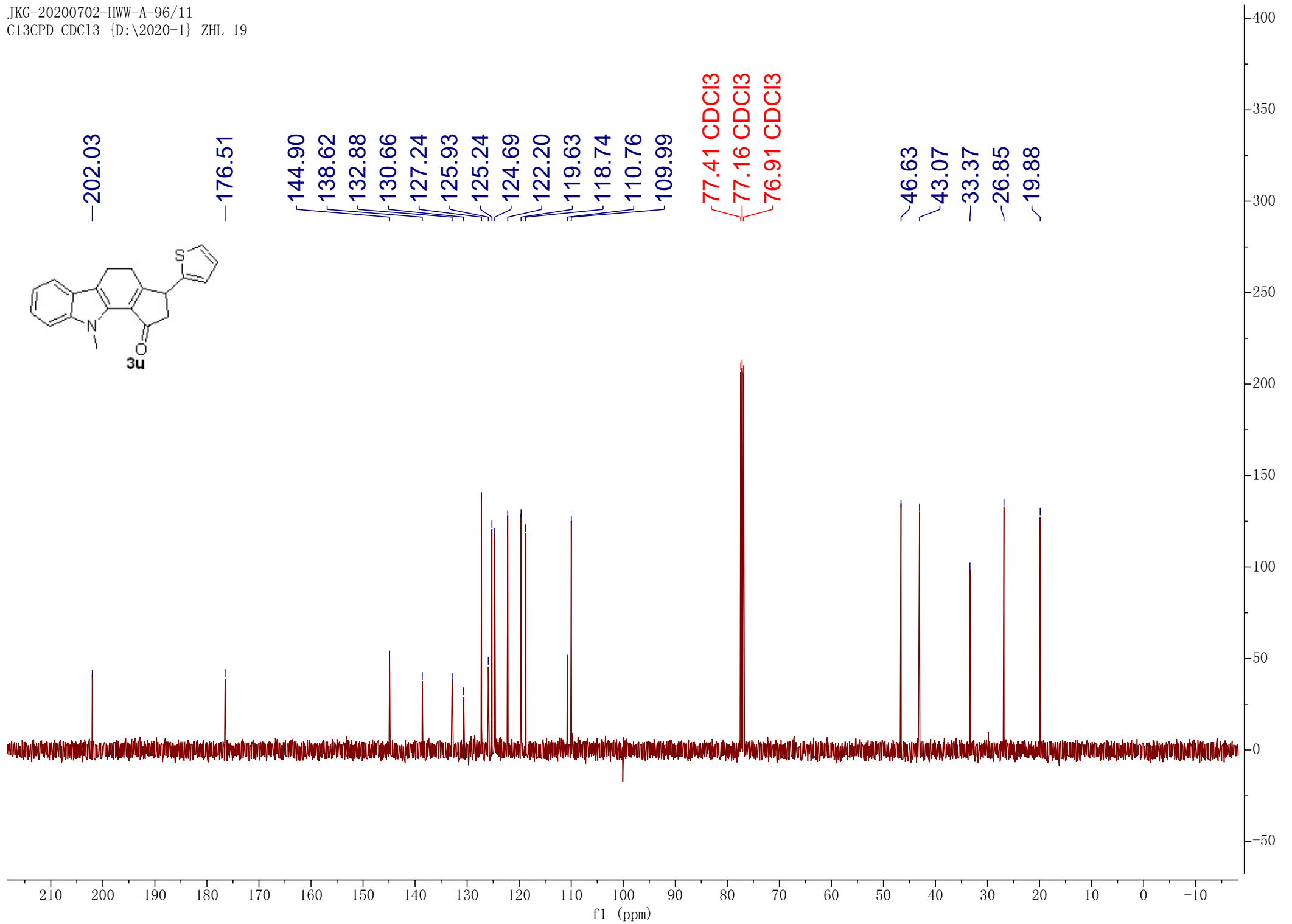
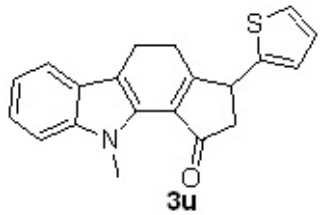


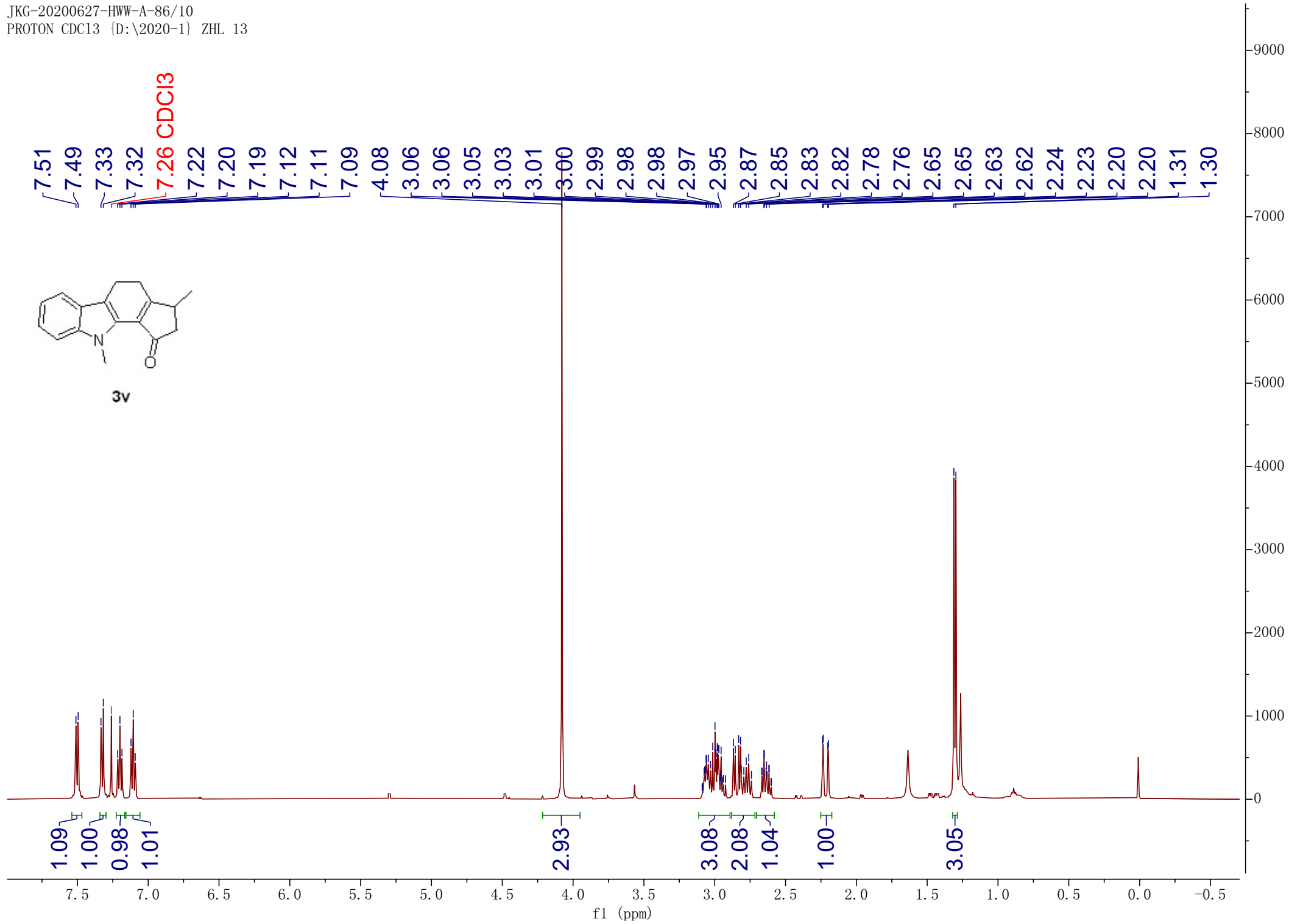
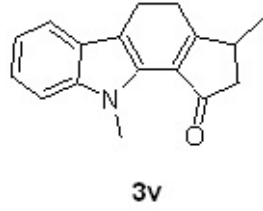


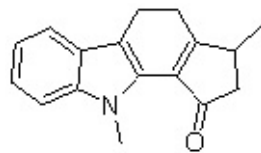
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