

Electronic Supplementary Information

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Gold(I)-Catalyzed Tandem Cyclization of Cyclopropylidene-tethered Propargylic Alcohols: An Approach to Functionalized Naphtho[2,3-c]pyrans

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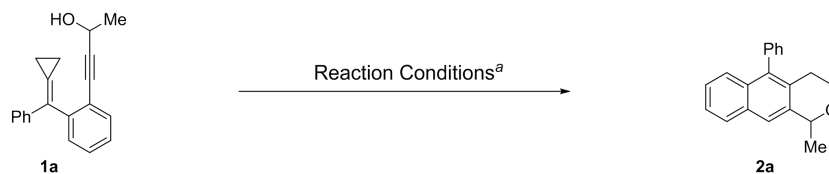
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1. General:

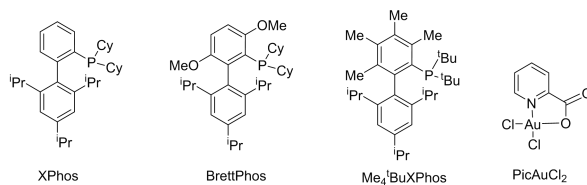
Dry tetrahydrofuran (THF) and toluene were distilled over sodium under N₂ prior to use. Anhydrous 1,2-dichloroethane, PhCl and PhF were bought from Energy Chemical. Dry DCM was distilled over calcium hydride at least for 1 h prior to use. All reactions sensitive to air or moisture were conducted under nitrogen atmosphere in dry solvents. All glassware and stir bars were washed with aquaregia prior to use.

¹H NMR and ¹³C NMR spectra were recorded on a Bruker Advance spectrometer at 500 MHz and 125 MHz in CDCl₃, *d*-Acetone or *d*-DMSO with reference to residual solvents signals [¹H NMR CDCl₃ (7.26); ¹³C NMR CDCl₃ (77.16)]. Chemical shift values were given in δ (ppm) and coupling constants were mentioned in Hz. Proton multiplicity is reported as a singlet (s), a broad singlet (br), a doublet (d), a triplet (t), a triplet double (td) or a multiplet (m). High resolution mass spectrometry (HRMS) was performed with a Thermo Scientific LTQ Orbitrap XL unless otherwise noted.

2. Table S1 Screening conditions.



Entry	Catalyst (5 mol %)	Solvents	T (°C)	Additive (5 mol %)	Time (h)	Yield(%) 2a
1	IPrAuCl	DCE	75	AgNTf ₂	36	messy
2	BrettPhosAuCl	DCE	75	AgNTf ₂	36	messy
3	(2,4- ^t Bu ₂ PhO) ₃ PAuCl	DCE	75	AgNTf ₂	36	messy
4	Ph ₃ PAuCl	DCE	75	AgNTf ₂	36	No reaction
5	Me ₄ ^t BuXPhosAuCl	DCE	75	AgNTf ₂	36	44
6	XPhosAuCl	DCE	75	AgNTf ₂	36	messy
7	PicAuCl ₂	DCE	75	-	36	messy
8	Me ₄ ^t BuXPhosAuNTf ₂	toluene	75	-	36	60
9	Me ₄ ^t BuXPhosAuNTf ₂	PhCF ₃	75	-	36	55
10	Me ₄ ^t BuXPhosAuNTf ₂	PhCl	75	-	36	77
11	Me ₄ ^t BuXPhosAuNTf ₂	PhF	75	-	36	81
12	Me ₄ ^t BuXPhosAuNTf ₂	PhF	85	-	24	83
13	Me ₄ ^t BuXPhosAuCl	PhF	85	NaBARF	24	messy
14	Me ₄ ^t BuXPhosAuCl	PhF	85	AgOTs	24	No reaction
15	Me ₄ ^t BuXPhosAuCl	PhF	85	AgOTf	24	24
16	Me ₄ ^t BuXPhosAuCl	PhF	85	AgSbF ₆	24	messy



^a Reaction conditions: All reactions were run in vials in the presence of **1a** (0.2 mmol); [**1a**] = 0.05 M.

3. Experimental procedures:

Procedure 1.

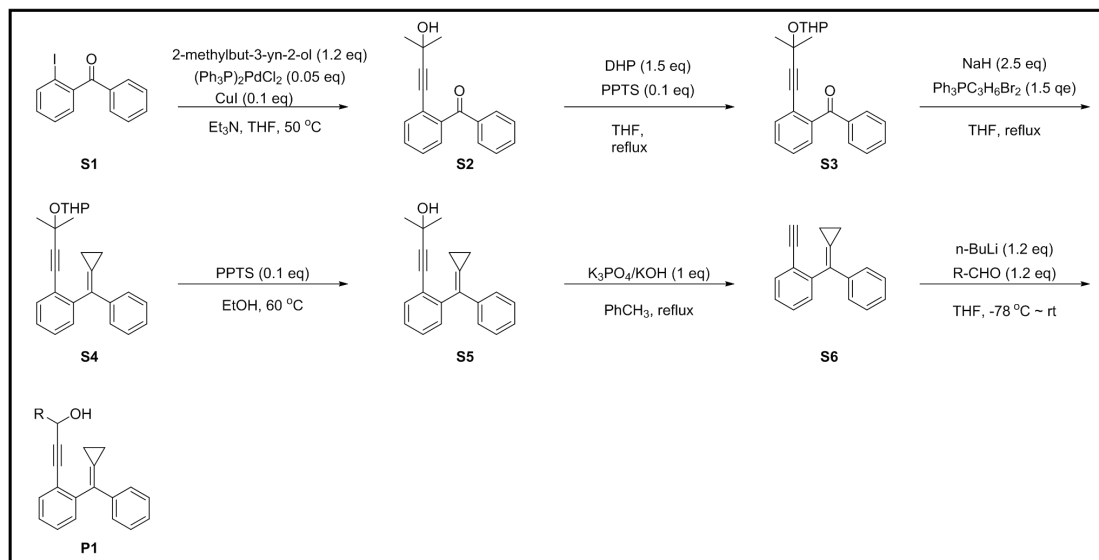


Figure S1. Procedure 1

S1¹ (6.16 g, 20 mmol, 1 eq) was dissolved in 40 mL dry THF and 20 mL Et_3N and the resulting mixture was degassed and flushed with N_2 for three times at room temperature. $(\text{PPh}_3)_2\text{PdCl}_2$ (5 mol %) and CuI (10 mol %) was added to the above mixture successively, and the resulting mixture was degassed and flushed with N_2 for three times at room temperature, and then 2-methylbut-3-yn-2-ol (2.0 g, 24 mmol, 1.2 eq) was added to the above mixture dropwise at room temperature. The reaction mixture was heated at 50 °C for 12 h, upon completion, the mixture was quenched with saturated NH_4Cl solution, extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **S2** (4.84 g, 18.3 mmol, 92%).

To a solution of **S2** (3.96 g, 15 mmol, 1 eq) in 40 mL dry DCM was added PPTS (378 mg, 0.1 eq) at 0 °C, and then DHP (1.89g, 22.5 mmol, 1.2 eq) was added to the above mixture dropwise. After addition, the reaction mixture was stirred at room temperature for 5 h. Upon completion, the mixture was quenched with saturated NaHCO_3 solution, extracted with DCM, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by

chromatography to get the desired compound **S3** (4.7 g, 13.5 mmol, 90 %).

$\text{Ph}_3\text{PC}_3\text{H}_6\text{Br}_2$ (6.96 g, 15 mmol, 1.5 eq) was suspended in 80 mL dry THF and NaH (1 g, 25 mmol, 2.5 eq) was added to the above reaction mixture in one portion at room temperature. The resulting mixture was heated at 70 °C for 18 h under a nitrogen atmosphere, then **S3** (3.48 g, 10 mmol, 1.0 eq) dissolved in 10 mL dry THF was added to the above mixture dropwise within 5 min. The mixture was heated at 70 °C for another 4 h. Upon completion, the reaction was quenched with saturated NH_4Cl solution, extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **S4** (3.18 g, 85 %).

To a solution of **S4** (1 eq) in EtOH was added PPTS (0.1 eq) at room temperature, the resulting mixture was heated at 60 °C for 8 hours. Upon completion, the reaction was quenched with saturated NaHCO_3 solution, and evaporated nearly to dry, diluted with water, extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **S5** (96 %).

To a solution of **S6** (1 eq) in dry toluene was added KOH (1 eq) and K_3PO_4 (1 eq), and then the resulting mixture was heated at 120 °C for 2 h. Upon completion, the reaction mixture was filtered through a celite plug and concentrated to get the crude product which was purified by chromatography to get the desired compound **S6** (91 %).²

To a solution of **S6** (1.0 eq) in THF at -78 °C was added n-BuLi (1.2 eq) dropwise under a nitrogen atmosphere. After stirring at -78 °C for 1 h, the substituted aldehydes (1.2 eq), dissolved in 5 mL THF, were added to the above mixture dropwise. After addition, the mixture was allowed to warm up to rt and stirred for another 1 h. Upon completion, the mixture was quenched with saturated NH_4Cl solution, extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **P1**.

Procedure 2.

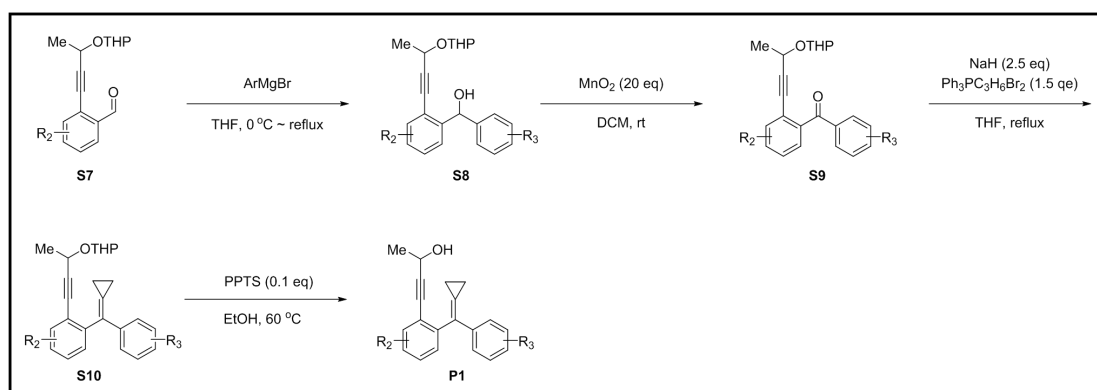


Figure S2. Procedure 2

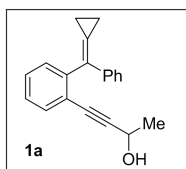
To a solution of **S7** (10 mmol, 1 eq) in 30 mL dry THF was added ArMgBr (12 mmol, 1.2 eq) dropwise under a nitrogen atmosphere at 0°C . After addition the resulting mixture was stirred at 75°C for two hours. Upon completion, the mixture was quenched with saturated NH_4Cl solution, extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **S8**.

To a solution of **S8** (1.0 eq) in dry CH_2Cl_2 was added MnO_2 (20 eq) in one portion at rt and then the mixture was stirred at rt for 5 h. Upon completion, the mixture was filtered through a celite plug, concentrated to give the crude product and the residue was purified by chromatography to get the desired substrate **S9**.

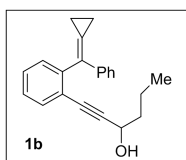
$\text{Ph}_3\text{PC}_3\text{H}_6\text{Br}_2$ (1.5 eq) was suspended in dry THF and NaH (2.5 eq) was added to the above reaction mixture in one portion at room temperature. The resulting mixture was heated at 70°C for 18 h under a nitrogen atmosphere, then **S9** (1.0 eq) dissolved in 10 mL dry THF was added to the above mixture dropwise within 5 min. The mixture was heated at 70°C for another 7 h. Upon completion, the reaction was quenched with saturated NH_4Cl solution, extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **S10**.

To a solution of **S10** (1 eq) in EtOH was added PPTS (0.1 eq) at room temperature, the resulting mixture was heated at 60°C for 8 hours. Upon completion, the reaction was quenched with saturated NaHCO_3 solution, and evaporated nearly to

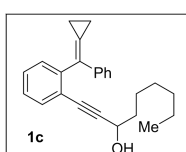
dry, diluted with water, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **P1**.



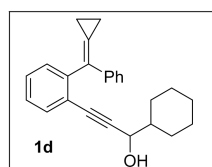
4-(2-(cyclopropylidene(phenyl)methyl)phenyl)but-3-yn-2-ol, 1a, overall yield 56%, light yellow oil; ¹H NMR (500 MHz, CDCl₃): δ = 7.47 - 7.43 (m, 3H), 7.37 - 7.36 (m, 2H), 7.34 - 7.22 (m, 4H), 4.37 (q, *J* = 6.5 Hz, 1H), 1.61 - 1.57 (m, 2H), 1.28 - 1.25 (m, 3H), 1.19 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃): δ = 144.0, 141.0, 132.5, 130.4, 129.4, 128.5, 128.2, 127.3, 127.1, 126.7, 126.4, 122.3, 95.1, 83.9, 58.7, 24.0, 5.3, 2.4; HRMS (ESI) *m/z* calcd for C₂₀H₁₉O⁺ (M+H)⁺ 275.1430, found 275.1435.



1-(2-(cyclopropylidene(phenyl)methyl)phenyl)hex-1-yn-3-ol, 1b, overall yield 51%, light yellow oil; ¹H NMR (500 MHz, CDCl₃): δ = 7.50 - 7.45 (m, 3H), 7.37 - 7.28 (m, 5H), 7.26 - 7.23 (m, 1H), 4.25 (t, *J* = 6.5 Hz, 1H), 1.62 - 1.59 (m, 2H), 1.52 - 1.35 (m, 3H), 1.30 - 1.24 (m, 4H), 0.86 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃): δ = 144.0, 140.8, 132.5, 130.4, 129.4, 128.4, 128.2, 127.1, 127.0, 126.7, 126.1, 122.5, 94.2, 84.6, 62.6, 39.7, 18.3, 13.8, 5.3, 2.2; HRMS (ESI) *m/z* calcd for C₂₂H₂₃O⁺ (M+H)⁺ 303.1743, found 303.1745.

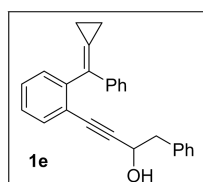


1-(2-(cyclopropylidene(phenyl)methyl)phenyl)non-1-yn-3-ol, 1c, overall yield 55%, light yellow oil; ¹H NMR (500 MHz, CDCl₃): δ = 7.47 - 7.43 (m, 3H), 7.36 - 7.35 (m, 2H), 7.33 - 7.27 (m, 3H), 7.24 - 7.21 (m, 1H), 4.22 (t, *J* = 6.5 Hz, 1H), 1.60 - 1.57 (m, 2H), 1.49 - 1.37 (m, 2H), 1.29 - 1.21 (m, 11H), 0.88 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃): δ = 144.0, 140.8, 132.5, 130.4, 129.4, 128.4, 128.2, 127.2, 127.1, 126.7, 126.2, 122.5, 94.2, 84.7, 62.9, 37.6, 31.8, 29.1, 25.1, 22.7, 14.2, 5.4, 2.3; HRMS (ESI) *m/z* calcd for C₂₅H₂₉O⁺ (M+H)⁺ 345.2213, found 345.2216.

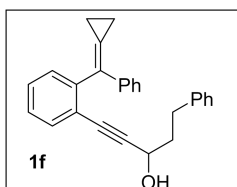


1-cyclohexyl-3-(2-(cyclopropylidene(phenyl)methyl)phenyl)prop-2-yn-1-ol, 1d, overall yield 49%, light yellow oil; ¹H NMR (500

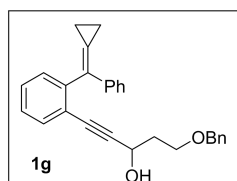
MHz, CDCl₃): δ = 7.48 (d, J = 7.5 Hz, 1H), 7.43 (d, J = 7.5 Hz, 2H), 7.37 - 7.27 (m, 5H), 7.22 (t, J = 7.0 Hz, 1H), 4.03 (t, J = 5.5 Hz, 1H), 1.67 - 1.56 (m, 7H), 1.35 - 1.27 (m, 1H), 1.22 (t, J = 8.0 Hz, 2H), 1.17 - 1.09 (m, 3H), 1.05 - 0.97 (m, 1H), 0.91 - 0.79 (m, 2H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 144.0, 140.6, 132.5, 130.3, 129.3, 128.4, 128.2, 127.0, 126.7, 125.9, 122.6, 93.0, 85.4, 67.6, 44.1, 28.5, 27.9, 26.4, 26.0, 26.0, 5.5, 2.1; **HRMS (ESI)** m/z calcd for C₂₅H₂₇O⁺ (M+H)⁺ 343.2056, found 343.2059.



4-(2-(cyclopropylidene(phenyl)methyl)phenyl)-1-phenylbut-3-yn-2-ol, 1e, overall yield 46%, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.46 (d, J = 8.0 Hz, 1H), 7.42 (d, J = 7.5 Hz, 2H), 7.39 (d, J = 4.0 Hz, 2H), 7.35 - 7.29 (m, 3H), 7.27 - 7.22 (m, 4H), 7.17 - 7.15 (m, 2H), 4.46 - 4.44 (m, 1H), 2.75 (d, J = 6.5 Hz, 2H), 1.59 - 1.56 (m, 2H), 1.35 (br, 1H), 1.28 - 1.25 (m, 2H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 144.0, 141.0, 136.7, 132.6, 130.4, 129.9, 129.3, 128.5, 128.3, 128.2, 127.3, 127.1, 126.8, 126.7, 126.4, 122.3, 93.4, 85.6, 63.5, 43.8, 5.2, 2.4; **HRMS (ESI)** m/z calcd for C₂₆H₂₃O⁺ (M+H)⁺ 351.1743, found 351.1745.

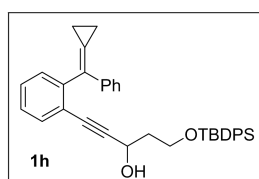


1-(2-(cyclopropylidene(phenyl)methyl)phenyl)-5-phenylpent-1-yn-3-ol, 1f, overall yield 55%, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.50 (d, J = 7.5 Hz, 1H), 7.47 - 7.45 (m, 2H), 7.40 - 7.36 (m, 2H), 7.33 - 7.29 (m, 3H), 7.28 - 7.17 (m, 4H), 7.10 - 7.09 (m, 2H), 4.25 (t, J = 6.5 Hz, 1H), 2.62 - 2.54 (m, 2H), 1.84 - 1.71 (m, 2H), 1.60 - 1.57 (m, 2H), 1.28 - 1.25 (m, 3H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 144.1, 141.5, 140.8, 132.6, 130.4, 129.4, 128.6, 128.5, 128.5, 128.2, 127.2, 127.1, 126.8, 126.2, 126.0, 122.4, 93.7, 85.1, 62.2, 39.0, 31.3, 5.4, 2.3; **HRMS (ESI)** m/z calcd for C₂₇H₂₅O⁺ (M+H)⁺ 365.1900, found 365.1899.



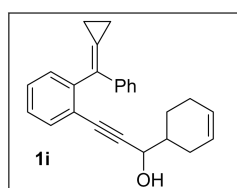
5-(benzyloxy)-1-(2-(cyclopropylidene(phenyl)methyl)phenyl)pent-1-yn-3-ol, 1g, overall yield 42%, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.50 (d, J = 7.5 Hz, 1H), 7.44 (d, J = 7.5

Hz, 2H), 7.38 - 7.26 (m, 10H), 7.21 (t, $J = 7.0$ Hz, 1H), 4.52 (q, $J = 5.5$ Hz, 1H), 4.37 (dd, $J = 19.5, 8.0$ Hz, 2H), 3.59 - 3.54 (m, 1H), 3.46 - 3.42 (m, 1H), 2.45 (d, $J = 6.0$ Hz, 1H), 1.85 - 1.79 (m, 1H), 1.69 - 1.63 (m, 1H), 1.59 - 1.56 (m, 2H), 1.26 - 1.22 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 143.9, 140.7, 138.1, 132.7, 130.3, 129.2, 128.5, 128.4, 128.2, 127.8, 127.7, 127.1, 127.0, 126.7, 126.2, 122.6, 93.2, 84.7, 73.2, 67.5, 61.6, 36.8, 5.4, 2.3$; HRMS (ESI) m/z calcd for $\text{C}_{28}\text{H}_{27}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 395.2006, found 395.2005.



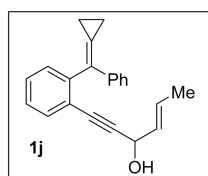
5-((tert-butyl-diphenylsilyloxy)methyl)-1-(2-(cyclopropylidene(phenyl)methyl)phenyl)pent-1-yn-3-ol, 1h, overall yield 40%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.68 - 7.66$ (m, 4H), 7.52 (d, $J = 7.5$ Hz, 1H), 7.46 - 7.30 (m, 11H), 7.22 (t, $J =$

7.5 Hz, 2H), 7.15 (t, $J = 7.0$ Hz, 1H), 4.61 - 4.59 (m, 1H), 3.82 - 3.77 (m, 1H), 3.65 - 3.59 (m, 1H), 2.60 (s, 1H), 1.77 - 1.71 (m, 1H), 1.63 - 1.57 (m, 1H), 1.54 (t, $J = 7.5$ Hz, 2H), 1.24 - 1.20 (m, 2H), 1.05 (s, 9H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 144.0, 140.7, 135.7, 134.9, 133.3, 133.2, 132.7, 130.4, 129.9, 129.8, 129.7, 129.3, 128.4, 128.1, 127.8, 127.1, 127.0, 126.6, 126.2, 122.6, 93.7, 84.7, 61.6, 61.5, 39.0, 26.9, 19.2, 5.3, 2.3$; HRMS (ESI) m/z calcd for $\text{C}_{37}\text{H}_{39}\text{O}_2\text{Si}^+$ ($\text{M}+\text{H}$) $^+$ 543.2714, found 543.2714.



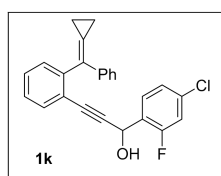
1-(cyclohex-3-en-1-yl)-3-(2-(cyclopropylidene(phenyl)methyl)phenyl)prop-2-yn-1-ol, 1i, overall yield 58%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.53$ (d, $J = 7.5$ Hz, 1H), 7.48 (d, $J = 8.0$ Hz, 2H), 7.43 - 7.32 (m, 5H), 7.27 (t, $J = 7.5$ Hz, 1H),

5.69 - 5.62 (m, 2H), 4.19 - 4.11 (m, 1H), 2.07 - 1.91 (m, 3H), 1.77 - 1.62 (m, 5H), 1.31 - 1.13 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3): (*isomers*) $\delta = 144.0, 140.5, 132.6, 130.4, 129.3, 128.5, 128.2, 127.1, 127.0, 127.0, 126.8, 126.7, 126.1, 126.0, 125.9, 122.5, 92.7, 85.4, 66.9, 40.2, 27.3, 25.0, 23.9, 5.5, 2.1$; HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{25}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 341.1900, found 341.1896.

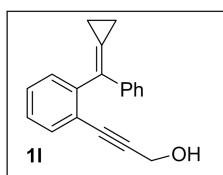


(E)-1-(2-(cyclopropylidene(phenyl)methyl)phenyl)hex-4-en-1-yn

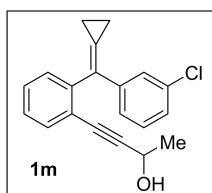
-3-ol, 1j, overall yield 54%, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): (*E/Z* > 10:1) $\delta = 7.49$ (d, $J = 7.5$ Hz, 1H), 7.43 (d, $J = 7.5$ Hz, 2H), 7.36 - 7.29 (m, 5H), 7.23 (t, $J = 7.5$ Hz, 1H), 5.67 - 5.60 (m, 1H), 5.37 - 5.32 (m, 1H), 4.68 (t, $J = 6.0$ Hz, 1H), 1.64 - 1.63 (m, 3H), 1.58 - 1.55 (m, 2H), 1.26 - 1.23 (m, 3H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): $\delta = 144.1, 140.9, 132.6, 130.4, 129.9, 129.3, 128.6, 128.5, 128.2, 127.2, 127.1, 126.7, 126.4, 122.3, 92.3, 85.8, 63.2, 17.6, 5.3, 2.4$; **HRMS (ESI)** m/z calcd for $\text{C}_{22}\text{H}_{21}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 301.1587, found 301.1586.



1-(4-chloro-2-fluorophenyl)-3-(2-(cyclopropylidene(phenyl)methyl)phenyl)prop-2-yn-1-ol, 1k, overall yield 43%, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): $\delta = 7.51$ (d, $J = 7.5$ Hz, 1H), 7.43 (d, $J = 7.5$ Hz, 2H), 7.40 - 7.35 (m, 2H), 7.33 - 7.29 (m, 4H), 7.24 - 7.21 (m, 1H), 7.20 - 7.17 (m, 1H), 6.95 (t, $J = 8.0$ Hz, 1H), 5.62 (d, $J = 6.0$ Hz, 1H), 1.83 (d, $J = 6.0$ Hz, 1H), 1.57 - 1.50 (m, 2H), 1.22 - 1.19 (m, 2H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): $\delta = 155.66$ ($J_{\text{CF}} = 250.00$ Hz), 144.3, 140.6, 132.6, 130.6, 130.4, 129.5 ($J_{\text{CF}} = 12.5$ Hz), 129.1, 129.0, 128.3, 127.1, 127.1, 126.8, 126.8, 126.5, 124.7 ($J_{\text{CF}} = 3.75$ Hz), 121.9, 121.2 ($J_{\text{CF}} = 17.50$ Hz), 90.7, 86.8, 59.2 ($J_{\text{CF}} = 5.00$ Hz), 5.4, 2.2; **HRMS (ESI)** m/z calcd for $\text{C}_{25}\text{H}_{19}\text{ClFO}^+$ ($\text{M}+\text{H}$) $^+$ 389.1103, found 389.1104.

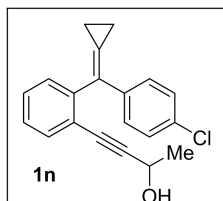


3-(2-(cyclopropylidene(phenyl)methyl)phenyl)prop-2-yn-1-ol, 1l, overall yield 53%, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): $\delta = 7.47 - 7.43$ (m, 3H), 7.38 - 7.27 (m, 5H), 7.26 - 7.23 (m, 1H), 4.10 (d, $J = 5.5$ Hz, 2H), 1.60 - 1.57 (m, 2H), 1.29 - 1.26 (m, 2H), 0.91 (t, $J = 6.0$ Hz, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): $\delta = 144.1, 141.1, 132.5, 130.4, 129.3, 128.5, 128.2, 127.4, 127.1, 126.7, 126.6, 122.3, 91.3, 85.7, 51.5, 5.2, 2.5$; **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{17}\text{O}^+$ ($\text{M}+\text{H}$) $^+$: 261.1274, found: 261.1276

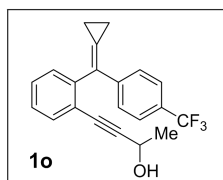


4-(2-((3-chlorophenyl)(cyclopropylidene)methyl)phenyl)but-3-yn-2-ol, 1m, overall yield 39%, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): $\delta = 7.48$ (d, $J = 7.5$ Hz, 2H), 7.42 (s, 1H), 7.38 -

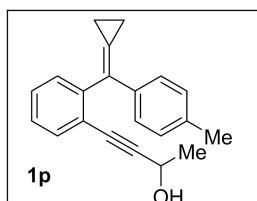
7.26 (m, 4H), 7.24 - 7.20 (m, 2H), 4.42 (q, $J = 5.5$ Hz, 1H), 1.61 - 1.56 (m, 3H), 1.28 - 1.25 (m, 2H), 1.21 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 143.2, 142.7, 134.2, 132.7, 130.3, 129.4, 128.5, 128.4, 127.8, 127.3, 127.2, 126.6, 125.3, 122.4, 95.1, 83.6, 58.7, 24.1, 5.3, 2.5$; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{ClO}^+$ ($\text{M}+\text{H}$) $^+$ 309.1041, found 309.1045.



4-(2-((4-chlorophenyl)(cyclopropylidene)methyl)phenyl)but-3-yn-2-ol, 1n, overall yield 31%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.48$ (d, $J = 7.0$ Hz, 1H), 7.37 - 7.34 (m, 3H), 7.32 - 7.27 (m, 4H), 4.42 (q, $J = 5.5$ Hz, 1H), 1.72 (s, 1H), 1.59 - 1.55 (m, 2H), 1.27 - 1.24 (m, 2H), 1.21 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 143.4, 139.2, 132.7, 132.4, 130.2, 128.5, 128.4, 128.3, 128.3, 127.2, 126.8, 122.4, 94.9, 83.6, 58.7, 24.0, 5.3, 2.4$; HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{ClO}^+$ ($\text{M}+\text{H}$) $^+$ 309.1041, found 309.1045.

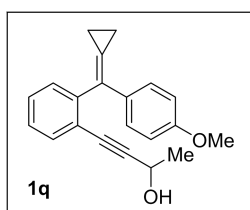


4-(2-(cyclopropylidene(4-(trifluoromethyl)phenyl)methyl)phenyl)but-3-yn-2-ol, 1o, overall yield 23%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.56$ (d, $J = 8.5$ Hz, 2H), 7.52 (d, $J = 8.5$ Hz, 2H), 7.48 (d, $J = 8.0$ Hz, 1H), 7.39 - 7.36 (m, 1H), 7.33 - 7.29 (m, 2H), 4.39 (q, $J = 6.5$ Hz, 1H), 1.63 - 1.60 (m, 2H), 1.42 (brs, 1H), 1.31 - 1.26 (m, 2H), 1.15 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): (one carbon merged to others) $\delta = 144.2, 143.1, 132.8, 130.3, 129.0, 128.7, 128.6$ (q, $J_{\text{CF}} = 16.25$ Hz), 127.5, 127.3, 125.1 (q, $J_{\text{CF}} = 3.75$ Hz), 124.5 (q, $J_{\text{CF}} = 270.0$ Hz), 122.4, 95.1, 83.6, 58.7, 24.0, 5.4, 2.6; ^{19}F NMR (470 MHz, CDCl_3) $\delta = -62.3$; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{18}\text{F}_3\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 343.1304, found 343.1306



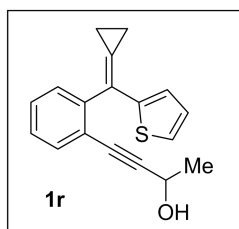
4-(2-(cyclopropylidene(p-tolyl)methyl)phenyl)but-3-yn-2-ol, 1p, overall yield 48%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.46$ (d, $J = 7.5$ Hz, 1H), 7.36 - 7.26 (m, 5H), 7.14 (d, $J = 8.0$ Hz, 2H), 4.38 (q, $J = 6.5$ Hz, 1H), 2.35 (s, 3H), 1.60

- 1.55 (m, 2H), 1.29 - 1.23 (m, 3H), 1.21 (t, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 144.2, 138.2, 136.4, 132.5, 130.4, 129.2, 128.9, 128.4, 127.2, 127.0, 125.3, 122.3, 94.9, 84.0, 58.7, 24.0, 21.2, 5.2, 2.4$; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 289.1587, found 289.1589.



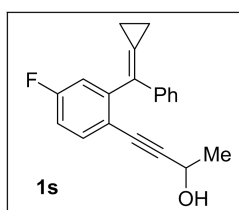
4-(2-(cyclopropylidene(p-tolyl)methyl)phenyl)but-3-yn-2-ol, 1q, overall yield 43%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.46$ (d, $J = 8.0$ Hz, 1H), 7.37 - 7.33 (m, 4H), 7.29 - 7.25 (m, 1H), 6.87 - 6.85 (m, 2H), 4.41 (q, $J = 6.5$ Hz, 1H), 3.80

(s, 3H), 1.56 - 1.53 (m, 2H), 1.41 (s, 1H), 1.24 - 1.22 (m, 5H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 158.6, 144.3, 133.7, 132.5, 130.3, 128.8, 128.4, 128.4, 127.0, 124.1, 122.4, 113.6, 94.8, 83.9, 58.8, 55.5, 24.1, 5.1, 2.4$; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 305.1536, found 305.1534.



4-(2-(cyclopropylidene(thiophen-2-yl)methyl)phenyl)but-3-yn-2-ol, 1r, overall yield 53%, white solid; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.50$ (d, $J = 7.5$ Hz, 1H), 7.40 - 7.34 (m, 2H), 7.30 (t, $J = 7.0$ Hz, 1H), 7.21 (d, $J = 5.0$ Hz, 1H), 6.95 (t, $J = 4.5$ Hz, 1H),

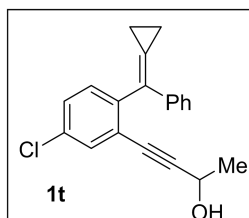
6.72 (d, $J = 3.0$ Hz, 1H), 4.51 - 4.46 (m, 1H), 1.73 (d, $J = 5.0$ Hz, 1H), 1.56 - 1.53 (m, 2H), 1.35 - 1.32 (m, 2H), 1.29 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 145.9, 142.9, 132.5, 129.8, 128.3, 127.4, 127.0, 125.1, 124.7, 124.6, 124.2, 122.4, 94.7, 83.5, 58.7, 24.2, 5.6, 3.9$; HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{17}\text{OS}^+$ ($\text{M}+\text{H}$) $^+$ 281.0995, found 281.0992.



4-(2-(cyclopropylidene(phenyl)methyl)-4-fluorophenyl)but-3-yn-2-ol, 1s, overall yield 44%, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.46 - 7.44$ (m, 3H), 7.34 (t, $J = 7.5$ Hz, 2H), 7.26 (t, $J = 7.5$ Hz, 1H), 7.11 - 7.09 (m, 1H), 7.00 (td, $J = 2.5, 8.5$

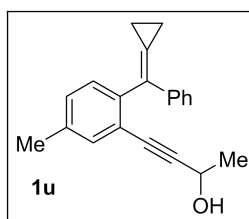
Hz, 1H), 4.37 - 4.33 (m, 1H), 1.62 - 1.59 (m, 3H), 1.31 - 1.28 (m, 2H), 1.18 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 162.4$ ($J_{\text{CF}} = 247.5$ Hz), 146.3 ($J_{\text{CF}} = 7.5$

Hz), 140.2 ($J_{CF} = 8.75$ Hz), 134.3, 128.6 ($J_{CF} = 2.5$ Hz), 128.2, 127.2, 127.2, 126.8, 118.5, 117.3 ($J_{CF} = 22.5$ Hz), 114.3 ($J_{CF} = 21.25$ Hz), 94.7, 82.8, 58.5, 23.9, 5.2, 2.4; **HRMS (ESI)** m/z calcd for $C_{20}H_{18}FO^+$ ($M+H$) $^+$ 293.1336, found 293.1336.



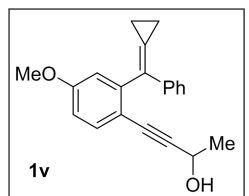
4-(5-chloro-2-(cyclopropylidene(phenyl)methyl)phenyl)but-3-yn-2-ol, 1t, overall yield 46%, light yellow oil; **1H NMR (500 MHz, $CDCl_3$)**: $\delta = 7.48$ (d, $J = 6.5$ Hz, 1H), 7.42 (t, $J = 1.5$ Hz, 1H), 7.38 - 7.28 (m, 4H), 7.24 - 7.19 (m, 2H), 4.43 (q, $J = 6.5$

Hz, 1H), 1.61 - 1.58 (m, 2H), 1.55 (br, 1H), 1.28 - 1.25 (m, 2H), 1.21 (d, $J = 6.5$ Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)**: $\delta = 143.2, 142.7, 134.2, 132.7, 130.3, 129.4, 128.5, 128.3, 127.8, 127.3, 127.2, 126.6, 125.3, 122.4, 95.1, 83.6, 58.7, 24.1, 5.3, 2.5$; **HRMS (ESI)** m/z calcd for $C_{20}H_{18}ClO^+$ ($M+H$) $^+$ 309.1041, found 309.1046.



4-(2-(cyclopropylidene(phenyl)methyl)-5-methylphenyl)but-3-yn-2-ol, 1u, overall yield 46%, light yellow oil; **1H NMR (500 MHz, $CDCl_3$)**: $\delta = 7.47$ (d, $J = 7.5$ Hz, 2H), 7.35 - 7.32 (m, 3H), 7.29 - 7.18 (m, 3H), 4.38 (q, $J = 6.5$ Hz, 1H), 2.38 (s, 3H),

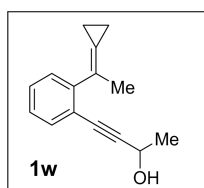
1.61 - 1.58 (m, 2H), 1.48 (br, 1H), 1.29 - 1.26 (m, 2H), 1.20 (d, $J = 6.5$ Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)**: $\delta = 141.1, 141.0, 136.7, 133.0, 130.3, 129.3, 129.2, 128.1, 127.3, 126.6, 125.9, 122.1, 94.7, 84.0, 58.6, 24.0, 21.0, 5.2, 2.4$; **HRMS (ESI)** m/z calcd for $C_{21}H_{21}O_2^+$ ($M+H$) $^+$: 289.1587, found: 289.1588.



4-(2-(cyclopropylidene(phenyl)methyl)-4-methoxyphenyl)but-3-yn-2-ol, 1v, overall yield 44%, light yellow oil; **1H NMR (500 MHz, $CDCl_3$)**: $\delta = 7.46$ (d, $J = 7.5$ Hz, 2H), 7.40 (d, $J = 8.5$ Hz, 1H), 7.33 (t, $J = 8.0$ Hz, 2H), 7.24 (t, $J = 7.5$ Hz, 1H), 6.92

(d, $J = 2.5$ Hz, 1H), 6.84 (dd, $J = 8.5, 2.5$ Hz, 1H), 4.36 (q, $J = 6.5$ Hz, 1H), 3.82 (s, 3H), 1.61 - 1.58 (m, 2H), 1.43 (br, 1H), 1.30 - 1.26 (m, 2H), 1.18 (d, $J = 7.0$ Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)**: $\delta = 159.6, 145.6, 140.7, 133.8, 129.4, 128.1, 127.2, 126.7, 126.2, 115.9, 114.7, 112.8, 93.5, 83.7, 58.7, 55.4, 24.0, 5.3, 2.4$; **HRMS (ESI)**

m/z calcd for $C_{21}H_{21}O_2^+$ ($M+H$) $^+$ 305.1536, found 305.1538.



4-(2-(1-cyclopropylideneethyl)phenyl)but-3-yn-2-ol, 1w, overall yield 43%, light yellow oil; 1H NMR (500 MHz, $CDCl_3$): δ = 7.45 (d, J = 8.0 Hz, 1H), 7.30 - 7.26 (m, 2H), 7.20 - 7.17 (m, 1H), 4.72 (q, J = 6.5 Hz, 1H), 2.27 (t, J = 3.5 Hz, 3H), 2.14 (brs, 1H), 1.51 (d, J = 6.5 Hz, 3H), 1.22 - 1.18 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 145.9, 133.1, 128.3, 128.3, 126.4, 124.4, 123.0, 120.7, 93.7, 84.3, 59.0, 24.4, 21.8, 4.8, 2.4; HRMS (ESI) m/z calcd for $C_{15}H_{17}O^+$ ($M+H$) $^+$ 213.1274, found 213.1273

Procedure 3

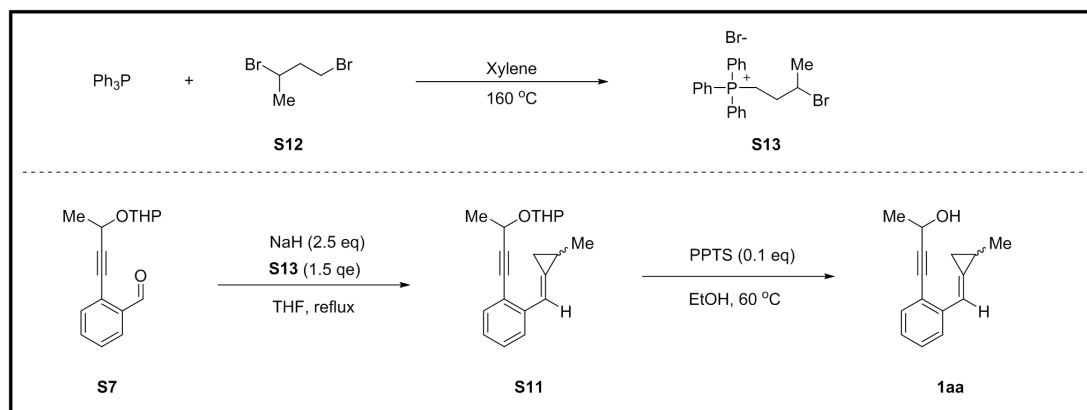
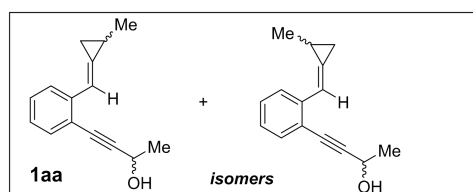


Figure S3. Procedure 3

To a solution of Ph_3P (1 eq) in Xylene was added **S13**, then the mixture was heated at 160 °C for 12 h. After cold to rt, the white solid was filtered, washed with Et_2O and dried to get the desired product **S13**.

S13 (1.5 eq) was suspended in dry THF and NaH (2.5 eq) was added to the above reaction mixture in one portion at room temperature. The resulting mixture was heated at 70 °C for 18 h under a nitrogen atmosphere, then **S7** (1.0 eq) dissolved in 10 mL dry THF was added to the above mixture dropwise within 5 min. The mixture was heated at at 70 °C for another 4 h. Upon completion, the reaction was quenched with saturated NH_4Cl solution, extracted with $EtOAc$, washed with brine, dried over Na_2SO_4 , filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **S11**.

To a solution of **S11** (1 eq) in EtOH was added PPTS (0.1 eq) at room temperature, the resulting mixture was heated at 60 °C for 8 hours. Upon completion, the reaction was quenched with saturated NaHCO₃ solution, and evaporated nearly to dry, diluted with water, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered, and concentrated to get the crude product which was purified by chromatography to get the desired compound **1aa**.



1aa, overall yield 35%, light yellow oil; ¹H NMR (500 MHz, CDCl₃, major isomer): δ = 7.41 - 7.39 (m, 1H), 7.29 - 7.24 (m, 2H), 7.15 - 7.11 (m, 1H), 6.32 - 6.25 (m, 1H), 4.82 - 4.78 (m, 1H), 2.07 (brs, 1H), 1.88 (dd, *J* = 5.5, 1.5 Hz, 1H), 1.85 (d, *J* = 7.0 Hz, 3H), 1.60 - 1.58 (m, 3H), 1.57 - 1.54 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): δ = 139.2, 132.9, 132.2, 131.2, 128.6, 127.6, 126.8, 124.5, 95.8, 82.7, 59.1, 33.1, 24.6, 18.5, 13.6.; HRMS (ESI) *m/z* calcd for C₁₅H₁₇O⁺ (M+H)⁺ 213.1274, found 213.1275

4. General procedure of the gold catalysis

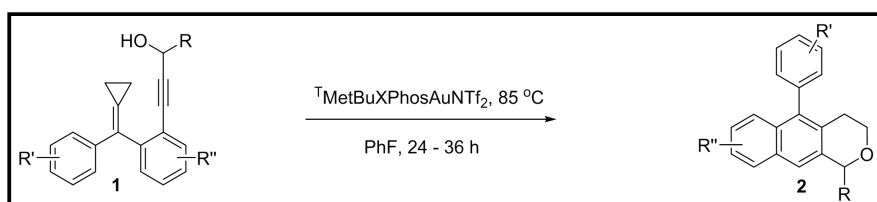
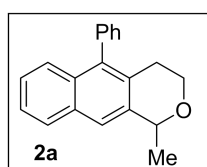


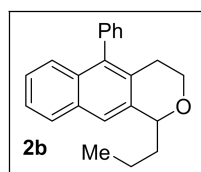
Figure S4. Procedure of gold catalysis

A dry schlenk tube was charged with ¹Me⁴BuXPhosAuNTf₂ (5 mol %), then ynone **1** dissolved in anhydrous PhF (4 mL) was added. Then the tube was sealed and stirred at 85 °C until the reaction was complete, as monitored by thin layer chromatography. The reaction mixture was purified by chromatography to get the desired compound **2**.



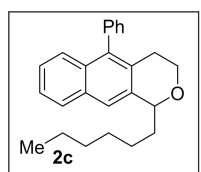
1-methyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2a,

yield 83%, 45.5 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃):** δ = 7.83 (d, J = 7.5 Hz, 1H), 7.65 (s, 1H), 7.54 - 7.51 (m, 2H), 7.48 - 7.39 (m, 3H), 7.33 - 7.27 (m, 3H), 5.10 (q, J = 6.5 Hz, 1H), 4.16 - 4.12 (m, 1H), 3.81 (td, J = 11.0, 4.0 Hz, 1H), 2.91 - 2.85 (m, 1H), 2.54 (dt, J = 17.0, 3.5 Hz, 1H), 1.75 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃):** δ = 139.2, 138.4, 138.1, 132.1, 131.6, 130.2, 130.0, 128.8, 128.6, 127.7, 127.3, 126.1, 125.6, 125.4, 123.1, 73.1, 64.3, 28.9, 22.4; **HRMS (ESI)** m/z calcd for C₂₀H₁₉O⁺ (M+H)⁺ 275.1430, found 275.1429.



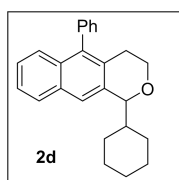
5-phenyl-1-propyl-3,4-dihydro-1H-benzo[g]isochromene, 2b,

yield 81%, 49 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃):** δ = 7.82 (d, J = 7.5 Hz, 1H), 7.65 (s, 1H), 7.53 - 7.51 (m, 2H), 7.47 - 7.38 (m, 3H), 7.32 - 7.29 (m, 2H), 7.27 - 7.26 (m, 1H), 5.01 (dd, J = 9.0, 3.5 Hz, 1H), 4.13 - 4.09 (m, 1H), 3.76 (td, J = 10.0, 4.0 Hz, 1H), 2.85 - 2.79 (m, 1H), 2.53 (dt, J = 16.5, 4.0 Hz, 1H), 2.12 - 2.06 (m, 1H), 2.03 - 1.95 (m, 1H), 1.65 - 1.57 (m, 2H), 1.04 (t, J = 7.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃):** δ = 139.2, 138.4, 137.2, 132.0, 131.5, 130.6, 130.3, 130.2, 128.7, 128.6, 127.7, 127.3, 126.1, 125.6, 125.4, 123.2, 76.4, 63.6, 38.9, 29.0, 18.8, 14.3; **HRMS (ESI)** m/z calcd for C₂₂H₂₃O⁺ (M+H)⁺ 303.1743, found 303.1742.



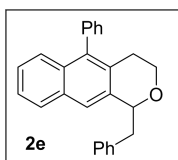
1-hexyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2c, yield

95%, 65 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃):** δ = 7.83 (d, J = 7.5 Hz, 1H), 7.66 (s, 1H), 7.54 - 7.51 (m, 2H), 7.48 - 7.39 (m, 3H), 7.33 - 7.26 (m, 3H), 5.01 (dd, J = 7.5 Hz, 1H), 4.14 - 4.11 (m, 1H), 3.79 - 3.74 (m, 1H), 2.87 - 2.80 (m, 1H), 2.56 - 2.53 (m, 1H), 2.16 - 2.09 (m, 1H), 2.03 - 1.97 (m, 1H), 1.63 - 1.58 (m, 2H), 1.50 - 1.36 (m, 6H), 0.94 (t, J = 6.0 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃):** δ = 139.2, 138.4, 137.2, 132.0, 131.5, 130.5, 130.3, 130.2, 128.7, 128.6, 127.7, 127.3, 126.1, 125.6, 125.3, 123.2, 76.6, 63.7, 36.8, 32.0, 29.6, 29.0, 25.5, 22.8, 14.3; **HRMS (ESI)** m/z calcd for C₂₅H₂₉O⁺ (M+H)⁺ 345.2213, found 345.2217.

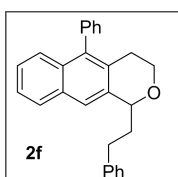


1-cyclohexyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2d,

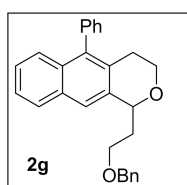
yield 98%, 67 mg, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 7.85 (d, J = 8.5 Hz, 1H), 7.68 (s, 1H), 7.55 - 7.51 (m, 2H), 7.48 - 7.43 (m, 3H), 7.35 - 7.27 (m, 3H), 4.92 (s, 1H), 4.16 - 4.13 (m, 1H), 3.68 (t, J = 11.0 Hz, 1H), 2.89 - 2.82 (m, 1H), 2.45 (d, J = 16.5 Hz, 1H), 2.08 (t, J = 11.5 Hz, 1H), 1.89 (t, J = 12.0 Hz, 2H), 1.73 - 1.60 (m, 3H), 1.47 - 1.19 (m, 5H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ = 139.1, 138.1, 136.1, 132.0, 131.7, 131.3, 130.5, 130.2, 128.7, 128.5, 127.8, 127.3, 126.1, 125.5, 125.3, 123.3, 80.9, 64.4, 44.7, 30.6, 29.2, 27.1, 26.7, 26.6, 25.8; **HRMS (ESI)** m/z calcd for $\text{C}_{25}\text{H}_{27}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 343.2056, found 343.2055.



1-benzyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2e, yield 90%, 63.1 mg, colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 7.84 (d, J = 8.0 Hz, 1H), 7.71 (s, 1H), 7.56 - 7.52 (m, 2H), 7.48 - 7.42 (m, 3H), 7.37 - 7.33 (m, 5H), 7.31 - 7.27 (m, 3H), 5.32 - 5.30 (m, 1H), 4.14 - 4.10 (m, 1H), 3.77 - 3.72 (m, 1H), 3.46 (dd, J = 14.0, 3.0 Hz, 1H), 3.27 - 3.23 (m, 1H), 2.79 - 2.73 (m, 1H), 2.55 (dt, J = 16.5, 3.5 Hz, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ = 139.0, 138.9, 138.5, 136.3, 131.9, 131.6, 130.7, 130.2, 130.2, 129.7, 128.7, 128.6, 128.4, 127.7, 127.4, 126.4, 126.1, 125.7, 125.4, 123.6, 77.4, 63.4, 43.4, 28.9; **HRMS (ESI)** m/z calcd for $\text{C}_{26}\text{H}_{23}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 351.1743, found 351.1741.

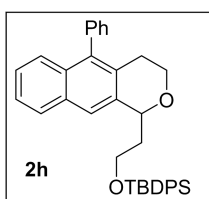


1-phenethyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2f, yield 97%, 70.7 mg, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 7.84 (d, J = 8.5 Hz, 1H), 7.67 (s, 1H), 7.58 - 7.55 (m, 2H), 7.51 - 7.43 (m, 3H), 7.39 - 7.30 (m, 7H), 7.28 - 7.25 (m, 1H), 5.06 (d, J = 10.5 Hz, 1H), 4.23 - 4.19 (m, 1H), 3.84 (td, J = 10.0, 4.0 Hz, 1H), 3.01 - 2.88 (m, 3H), 2.60 (dt, J = 16.5, 3.5 Hz, 1H), 2.54 - 2.46 (m, 1H), 2.40 - 2.33 (m, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ = 142.5, 139.1, 138.5, 136.7, 132.0, 131.5, 130.4, 130.2, 130.2, 128.7, 128.7, 128.6, 128.5, 127.7, 127.3, 126.1, 125.9, 125.6, 125.4, 123.1, 75.8, 63.7, 38.4, 31.7, 28.9; **HRMS (ESI)** m/z calcd for $\text{C}_{27}\text{H}_{25}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 365.1900, found 365.1899.



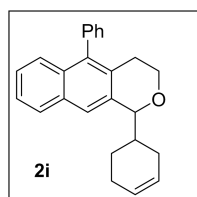
1-(2-(benzyloxy)ethyl)-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2g

mene, **2g**, yield 93%, 73.2 mg, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 7.82 (d, J = 8.5 Hz, 1H), 7.69 (s, 1H), 7.54 (d, J = 7.5 Hz, 2H), 7.48 - 7.37 (m, 7H), 7.34 - 7.28 (m, 4H), 5.21 - 5.18 (m, 1H), 4.65 - 4.60 (m, 2H), 4.12 - 4.08 (m, 1H), 3.91 - 3.86 (m, 1H), 3.80 - 3.74 (m, 2H), 2.86 - 2.79 (m, 1H), 2.58 - 2.51 (m, 2H), 2.30 - 2.23 (m, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ = 139.1, 138.8, 138.4, 136.6, 132.0, 131.5, 130.3, 130.2, 128.7, 128.6, 128.5, 127.8, 127.8, 127.6, 127.3, 126.0, 125.7, 125.4, 123.3, 73.8, 73.3, 67.2, 63.6, 36.9, 28.9; **HRMS (ESI)** m/z calcd for $\text{C}_{28}\text{H}_{27}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 395.2006, found 395.2007.



tert-butyldiphenyl(2-(5-phenyl-3,4-dihydro-1H-benzo[g]isochromen-1-yl)ethoxy)silane, 2h, yield 94%, 102 mg, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 7.84 (d, J = 8.0 Hz, 1H), 7.80 - 7.78 (m, 4H), 7.65 (s, 1H), 7.55 - 7.52 (m, 2H), 7.49 - 7.40 (m, 9H),

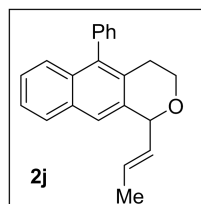
7.35 - 7.28 (m, 3H), 5.26 - 5.23 (m, 1H), 4.16 - 4.11 (m, 1H), 4.08 - 4.04 (m, 1H), 4.00 - 3.96 (m, 1H), 3.78 - 3.73 (m, 1H), 2.83 - 2.77 (m, 1H), 2.59 - 2.54 (m, 1H), 2.50 - 2.43 (m, 1H), 2.21 - 2.14 (m, 1H), 1.15 (s, 9H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ = 139.2, 138.4, 137.0, 135.8, 135.8, 134.3, 134.1, 132.0, 131.6, 130.3, 130.2, 130.2, 129.7, 129.7, 128.7, 128.6, 127.8, 127.8, 127.7, 127.3, 126.1, 125.6, 125.4, 123.2, 73.3, 63.4, 60.7, 39.5, 28.8, 27.1, 19.4; **HRMS (ESI)** m/z calcd for $\text{C}_{37}\text{H}_{39}\text{O}_2\text{Si}^+$ ($\text{M}+\text{H}$) $^+$ 543.2714, found 543.2714.



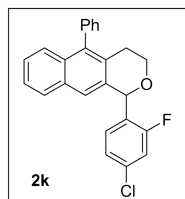
1-(cyclohex-3-en-1-yl)-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2i, yield 95%, 64.5 mg, light yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 7.82 (t, J = 7.0 Hz, 1H), 7.68 (d, J = 4.5 Hz, 1H), 7.54 - 7.50 (m, 2H), 7.47 - 7.40 (m, 3H), 7.34 - 7.29 (m, 2H), 7.27 - 7.26

(m, 1H), 5.81 - 5.62 (m, 2H), 5.00 - 4.98 (m, 1H), 4.16 - 4.12 (m, 1H), 3.72 - 3.65 (m, 1H), 2.89 - 2.81 (m, 1H), 2.47 - 2.17 (m, 4H), 2.08 - 1.50 (m, 4H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): (*isomers*) δ = 139.1, 138.2, 135.7, 132.1, 131.7, 131.4, 130.5, 130.2, 128.7, 128.5, 127.8, 127.3, 127.2, 126.7, 126.1, 125.6, 125.4, 123.4, 80.4, 64.5, 40.2, 29.1, 26.8, 26.3, 24.8; **HRMS (ESI)** m/z calcd for $\text{C}_{25}\text{H}_{25}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 341.1900, found

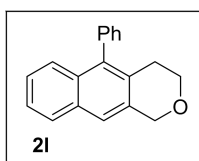
341.1893.



(E)-5-phenyl-1-(prop-1-en-1-yl)-3,4-dihydro-1H-benzo[g]isochromene, 2j, yield 68% (52% conversion), 21.4 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.81 (d, J = 8.5 Hz, 1H), 7.59 (s, 1H), 7.53 - 7.50 (m, 2H), 7.46 - 7.36 (m, 3H), 7.32 - 7.26 (m, 3H), 6.02 - 5.95 (m, 1H), 5.80 - 5.73 (m, 1H), 5.33 (d, J = 8.0 Hz, 1H), 4.16 - 4.12 (m, 1H), 3.83 (td, J = 11.5, 4.0 Hz, 1H), 2.87 - 2.80 (m, 1H), 2.54 (dt, J = 7.5, 4.0 Hz, 1H), 1.88 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 139.1, 138.5, 135.5, 131.9, 131.8, 131.6, 131.2, 130.2, 130.2, 130.0, 128.8, 128.6, 127.8, 127.4, 126.1, 125.7, 125.4, 124.7, 78.9, 64.1, 28.6, 18.1; **HRMS (ESI)** m/z calcd for C₂₂H₂₁O⁺ (M+H)⁺ 301.1587, found 301.1583.



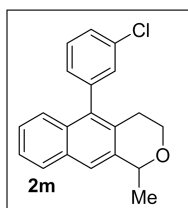
1-(4-chloro-2-fluorophenyl)-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2k, yield 72%, 55.9 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.70 (d, J = 8.0 Hz, 1H), 7.57 - 7.54 (m, 2H), 7.50 - 7.47 (m, 1H), 7.44 - 7.29 (m, 7H), 7.26 - 7.23 (m, 1H), 7.09 (t, J = 8.0 Hz, 1H), 6.35 (s, 1H), 4.22 - 4.18 (m, 1H), 3.96 (td, J = 10.5, 4.0 Hz, 1H), 3.04 - 2.97 (m, 1H), 2.66 (dt, J = 17.0, 3.5 Hz, 1H); **¹³C NMR (125 MHz, CDCl₃)**: (*one carbon merged to others*) δ = 156.6 (J_{CF} = 250.00 Hz), 138.8, 138.7, 134.6, 131.9, 131.9, 131.7 (J_{CF} = 13.75 Hz), 130.5, 130.2, 130.1, 129.0 (J_{CF} = 3.75 Hz), 128.8, 128.7, 127.8, 127.5, 126.1, 126.0, 125.6, 124.9, 124.8 (J_{CF} = 3.75 Hz), 121.5 (J_{CF} = 18.75 Hz), 73.8 (J_{CF} = 3.75 Hz), 64.9, 28.4; **HRMS (ESI)** m/z calcd for C₂₅H₁₉ClFO⁺ (M+H)⁺ 389.1103, found 389.1104.



(5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2l, yield 77%, 39.7 mg, white solid; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.80 (d, J = 8.5 Hz, 1H), 7.54 - 7.51 (m, 3H), 7.47 - 7.39 (m, 3H), 7.32 - 7.26 (m, 3H), 5.04 (s, 2H), 3.97 (t, J = 6.0 Hz, 2H), 2.70 (t, J = 6.0 Hz, 2H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 138.9, 138.7, 133.2, 132.0, 131.9, 130.1, 129.7, 128.7, 127.5,

127.4, 126.2, 125.5, 125.5, 122.6, 68.9, 66.1, 28.2.

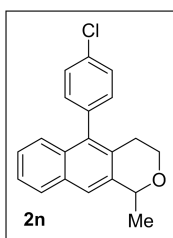
NMR spectroscopic signatures are matched with previously reported ones.³



5-(3-chlorophenyl)-1-methyl-3,4-dihydro-1H-benzo[gl]isochromene

e, 2m, yield 66%, 40.7 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**: δ = 7.82 (d, J = 8.0 Hz, 1H), 7.65 (s, 1H), 7.47 - 7.42 (m, 3H), 7.36 - 7.29 (m, 3H), 7.20 - 7.16 (m, 1H), 5.08 (q, J = 6.5 Hz,

1H), 4.16 - 4.12 (m, 1H), 3.83 - 3.77 (m, 1H), 2.91 - 2.82 (m, 1H), 2.55 - 2.48 (m, 1H), 1.73 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃)**: (*isomers*) δ = 141.1, 138.1, 136.9, 134.6, 132.0, 131.3, 130.3, 130.1, 130.0, 128.5, 127.8, 127.6, 125.9, 125.7, 125.6, 123.6, 73.1, 64.2, 28.8, 22.4; **HRMS (ESI)** m/z calcd for C₂₀H₁₈ClO⁺ (M+H)⁺ 309.1041, found 309.1040.

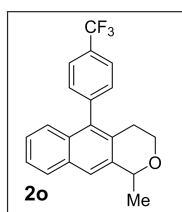


5-(4-chlorophenyl)-1-methyl-3,4-dihydro-1H-benzo[gl]isochromene,

2n, yield 60%, 40 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃)**:

δ = 7.82 (d, J = 8.5 Hz, 1H), 7.66 (s, 1H), 7.47 - 7.42 (m, 3H), 7.35 - 7.29 (m, 3H), 7.20 - 7.16 (m, 1H), 5.08 (q, J = 6.5 Hz, 1H), 4.16 - 4.12

(m, 1H), 3.83 - 3.77 (m, 1H), 2.91 - 2.82 (m, 1H), 2.54 - 2.48 (m, 1H), 1.73 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 141.1, 138.1, 136.8, 134.7, 132.0, 131.3, 130.3, 130.1, 130.0, 128.5, 127.8, 127.6, 125.9, 125.7, 125.6, 123.6, 73.1, 64.2, 28.8, 22.4; **HRMS (ESI)** m/z calcd for C₂₀H₁₈ClO⁺ (M+H)⁺ 309.1041, found 309.1040.



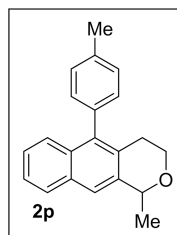
1-methyl-5-(4-(trifluoromethyl)phenyl)-3,4-dihydro-1H-benzo[gl]i

sochromene, 2o, yield 57%, 39.0 mg, light yellow oil; **¹H NMR (500**

MHz, CDCl₃): δ = 7.84 (d, J = 8.0 Hz, 1H), 7.80 (d, J = 8.5 Hz, 2H), 7.68 (s, 1H), 7.46 - 7.41 (m, 3H), 7.35 - 7.27 (m, 2H), 5.09 (q, J = 6.5

Hz, 1H), 4.16 - 4.12 (m, 1H), 3.80 (td, J = 11.0, 4.0 Hz, 1H), 2.88 - 2.81 (m, 1H), 2.47 (dt, J = 16.5, 3.5 Hz, 1H), 1.74 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃)**: δ = 143.2, 138.1, 136.9, 132.1, 131.2, 130.7, 130.7, 130.0, 129.76 (q, J_{CF} = 32.5 Hz), 127.9, 126.0, 125.8 (q, J_{CF} = 3.75 Hz), 125.7, 125.7 (q, J_{CF} = 3.75 Hz), 125.6, 124.4 (q, J_{CF} = 270.0 Hz), 123.8, 73.1, 64.1, 28.9, 22.4; **¹⁹F NMR (470 MHz, CDCl₃)** δ =

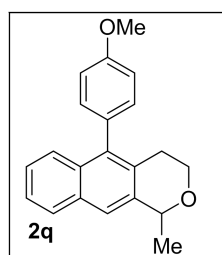
-62.4; **HRMS (ESI)** m/z calcd for $C_{21}H_{18}F_3O^+$ ($M+H$)⁺ 343.1304, found 343.1303.



1-methyl-5-(p-tolyl)-3,4-dihydro-1H-benzo[g]isochromene, 2p,

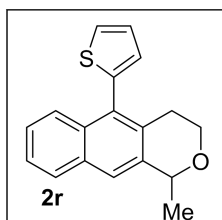
yield 64%, 36.9 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃):** δ = 7.82 (d, J = 8.0 Hz, 1H), 7.63 (s, 1H), 7.44 - 7.41 (m, 2H), 7.34 - 7.29 (m, 3H), 7.19 - 7.15 (m, 2H), 5.09 (q, J = 6.5 Hz, 1H), 4.15 - 4.11 (m, 1H), 3.79 (td, J = 11.0, 4.0 Hz, 1H), 2.92 - 2.85 (m, 1H), 2.54

(dt, J = 17.0, 3.5 Hz, 1H), 2.48 (s, 3H), 1.73 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃):** (*one carbon merged to others*) δ = 138.4, 138.1, 136.9, 136.0, 132.0, 131.7, 130.1, 130.1, 129.5, 129.3, 127.7, 126.2, 125.6, 125.4, 123.0, 73.1, 64.3, 28.9, 22.4, 21.5; **HRMS (ESI)** m/z calcd for $C_{21}H_{21}O^+$ ($M+H$)⁺ 289.1587, found 289.1583.



5-(4-methoxyphenyl)-1-methyl-3,4-dihydro-1H-benzo[g]isochromene, 2q, yield 71%, 43.2 mg, light yellow oil; **¹H NMR (500 MHz, CDCl₃):** δ = 7.82 (d, J = 8.0 Hz, 1H), 7.63 (s, 1H), 7.44 - 7.40 (m, 2H), 7.33 - 7.30 (m, 1H), 7.22 - 7.18 (m, 2H), 7.07 - 7.05

(m, 2H), 5.09 (q, J = 6.5 Hz, 1H), 4.15 - 4.11 (m, 1H), 3.91 (s, 3H), 3.79 (td, J = 11.0, 3.5 Hz, 1H), 2.92 - 2.85 (m, 1H), 2.55 (dt, J = 17.0, 3.5 Hz, 1H), 1.73 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃):** δ = 158.9, 138.1, 138.1, 132.0, 131.9, 131.3, 131.2, 131.2, 130.4, 127.7, 126.1, 125.6, 125.4, 123.0, 114.1, 114.1, 73.1, 64.3, 55.4, 29.0, 22.4; **HRMS (ESI)** m/z calcd for $C_{21}H_{21}O_2^+$ ($M+H$)⁺ 305.1536, found 305.1535.

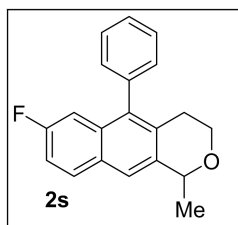


1-methyl-5-(thiophen-2-yl)-3,4-dihydro-1H-benzo[g]isochromene, 2r, yield 53%, 29.7 mg, light yellow oil; **¹H NMR (500 MHz,**

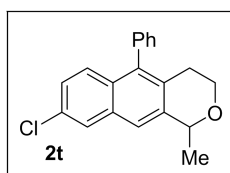
CDCl₃): δ = 7.81 (d, J = 8.0 Hz, 1H), 7.66 (s, 1H), 7.58 (d, J = 8.5 Hz, 1H), 7.52 - 7.50 (m, 1H), 7.45 - 7.42 (m, 1H), 7.39 - 7.36 (m,

1H), 7.23 - 7.22 (m, 1H), 7.00 - 6.99 (m, 1H), 5.07 (q, J = 6.5 Hz, 1H), 4.18 - 4.14 (m, 1H), 3.82 (td, J = 11.0, 3.5 Hz, 1H), 3.02 - 2.95 (m, 1H), 2.69 (dt, J = 17.0, 3.5 Hz, 1H), 1.72 (d, J = 6.5 Hz, 3H); **¹³C NMR (125 MHz, CDCl₃):** δ = 139.2, 138.0, 132.8,

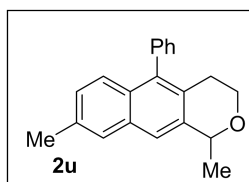
132.8, 131.9, 130.6, 128.1, 127.7, 127.4, 126.3, 126.1, 125.9, 125.6, 124.3, 73.0, 64.2, 28.8, 22.4; **HRMS (ESI)** m/z calcd for $C_{18}H_{17}OS^+$ ($M+H$) $^+$ 281.0995, found 281.0996.



7-fluoro-1-methyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2s, yield 64%, 37.5 mg, light yellow oil; **1H NMR (500 MHz, $CDCl_3$)**: δ = 7.80 (dd, J = 9.0, 5.5 Hz, 1H), 7.63 (s, 1H), 7.54 - 7.51 (m, 2H), 7.48 - 7.44 (m, 1H), 7.27 - 7.22 (m, 2H), 7.20 (td, J = 8.5, 2.5 Hz, 1H), 6.98 (dd, J = 11.5, 2.5 Hz, 1H), 5.06 (q, J = 6.5 Hz, 1H), 4.14 - 4.10 (m, 1H), 3.79 (td, J = 10.5, 3.5 Hz, 1H), 2.89 - 2.83 (m, 1H), 2.52 (dt, J = 17.0, 3.5 Hz, 1H), 1.73 (d, J = 6.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)**: δ = 160.6 (J_{CF} = 243.75 Hz), 138.6, 138.0 (J_{CF} = 5.0 Hz), 137.4 (J_{CF} = 2.5 Hz), 132.5 (J_{CF} = 8.75 Hz), 131.2, 130.1, 130.1, 130.0, 129.1, 128.9, 128.8, 127.6, 123.0, 116.0 (J_{CF} = 26.25 Hz), 109.5 (J_{CF} = 22.5 Hz), 73.0, 64.2, 28.9, 22.3; **HRMS (ESI)** m/z calcd for $C_{20}H_{18}FO^+$ ($M+H$) $^+$ 293.1336, found 293.1331.

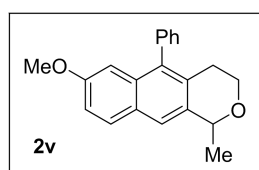


7-chloro-1-methyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2t, yield 53%, 32.6 mg, light yellow oil; **1H NMR (500 MHz, $CDCl_3$)**: δ = 7.74 (d, J = 8.5 Hz, 1H), 7.60 (s, 1H), 7.54 - 7.51 (m, 2H), 7.48 - 7.45 (m, 1H), 7.36 - 7.34 (m, 2H), 7.27 - 7.23 (m, 2H), 5.06 (q, J = 6.5 Hz, 1H), 4.14 - 4.10 (m, 1H), 3.78 (td, J = 11.0, 4.0 Hz, 1H), 2.88 - 2.81 (m, 1H), 2.50 (dt, J = 17.0, 3.5 Hz, 1H), 1.72 (d, J = 6.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)**: δ = 138.5, 138.3, 137.8, 132.3, 131.5, 131.3, 130.2, 130.1, 130.1, 129.4, 128.9, 128.8, 127.7, 126.4, 124.9, 123.0, 73.0, 64.1, 28.9, 22.3; **HRMS (ESI)** m/z calcd for $C_{20}H_{18}ClO^+$ ($M+H$) $^+$ 309.1041, found 309.1041.

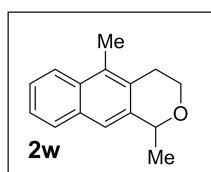


1,8-dimethyl-5-phenyl-3,4-dihydro-1H-benzo[g]isochromene, 2u, yield 68%, 39.2 mg, light yellow oil; **1H NMR (500 MHz, $CDCl_3$)**: δ = 7.60 (s, 1H), 7.56 (s, 1H), 7.52 (t, J = 7.5 Hz, 2H), 7.45 (t, J = 7.5 Hz, 1H), 7.30 - 7.26 (m, 3H), 7.15 (d, J

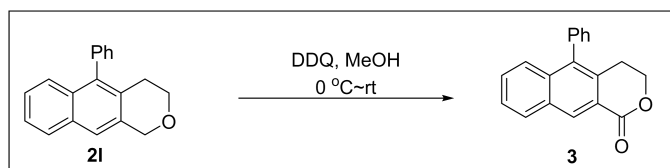
= 8.5 Hz, 1H), 5.08 (q, $J = 6.5$ Hz, 1H), 4.14 - 4.10 (m, 1H), 3.80 (td, $J = 10.5, 3.5$ Hz, 1H), 2.89 - 2.82 (m, 1H), 2.53 - 2.49 (m, 4H), 1.73 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): (one carbon merged to others) $\delta = 139.3, 138.2, 138.1, 135.0, 132.3, 130.2, 129.9, 129.0, 128.7, 128.6, 128.0, 127.3, 126.6, 125.9, 122.5, 73.2, 64.3, 28.8, 22.4, 21.6$; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 289.1587, found 289.1588.



7-methoxy-1-methyl-5-phenyl-3,4-dihydro-1H-benzo[gl]isochromene, 2v, yield 95%, 57.8 mg, light yellow solid; ^1H NMR (500 MHz, CDCl_3): $\delta = 7.73$ (d, $J = 9.0$ Hz, 1H), 7.57 (s, 1H), 7.53 (t, $J = 7.5$ Hz, 2H), 7.45 (t, $J = 7.5$ Hz, 1H), 7.29 (t, $J = 8.0$ Hz, 2H), 7.11 (dd, $J = 9.0, 2.5$ Hz, 1H), 6.67 - 6.66 (m, 1H), 5.07 (q, $J = 6.5$ Hz, 1H), 4.14 - 4.10 (m, 1H), 3.79 (td, $J = 11.0, 4.0$ Hz, 1H), 3.67 (s, 3H), 2.89 - 2.82 (m, 1H), 2.49 (dt, $J = 17.0, 3.0$ Hz, 1H), 1.72 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 157.5, 139.3, 137.3, 135.7, 132.6, 130.5, 130.2, 130.1, 129.3, 128.9, 128.7, 127.7, 127.3, 122.9, 118.0, 104.5, 73.0, 64.3, 55.2, 29.0, 22.4$; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{21}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 305.1536, found 305.1535.



1,5-dimethyl-3,4-dihydro-1H-benzo[gl]isochromene, 2w, yield 76%, 32.3 mg, light yellow oil; ^1H NMR (500 MHz, CDCl_3): $\delta = 8.03$ (d, $J = 8.0$ Hz, 1H), 7.77 (d, $J = 8.0$ Hz, 1H), 7.49 - 7.41 (m, 3H), 5.03 (q, $J = 6.5$ Hz, 1H), 4.33 - 4.28 (m, 1H), 3.89 (td, $J = 10.5, 4.0$ Hz, 1H), 3.15 - 3.08 (m, 1H), 2.89 (dt, $J = 16.5, 3.5$ Hz, 1H), 2.58 (s, 3H), 1.67 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): $\delta = 138.2, 132.0, 131.5, 131.4, 129.9, 128.4, 125.6, 125.2, 123.7, 121.7, 73.3, 64.3, 28.1, 22.4, 14.0$; HRMS (ESI) m/z calcd for $\text{C}_{15}\text{H}_{17}\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 213.1274, found 213.1275.

5. Preparation of compound **3**.Figure S5. Preparation of compound **3**

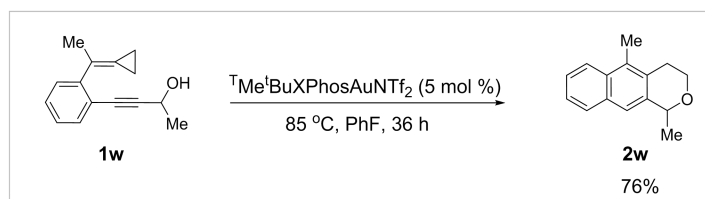
To a solution of **21** (50 mg, 0.19 mmol) in 2 mL MeOH was added DDQ (1.5 eq, 0.29 mmol) at 0 °C, then the mixture was stirred at rt for 16 h. Upon completion, the solvent was evaporated to dry and the residue was purified by chromatography to get the desired compound **3**.

5-phenyl-3,4-dihydro-1H-benzo[ghi]isochromen-1-one, 3, yield 95%, 50.4 mg, white solid; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ = 8.78 (s, 1H), 8.01 (d, J = 7.5 Hz, 1H), 7.55 - 7.47 (m, 6H), 7.29 - 7.28 (m, 2H), 4.48 (t, J = 6.0 Hz, 2H), 2.91 (t, J = 6.0 Hz, 2H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): (*three carbons merged to others*) δ = 166.0, 137.6, 137.5, 135.1, 132.4, 132.3, 132.1, 130.2, 129.8, 129.1, 128.9, 128.0, 126.5, 123.1, 67.6, 27.1; **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{15}\text{O}_2^+$ ($\text{M}+\text{H}$) $^+$ 275.1067, found 275.1069.

6. Mechanistic studies

In order seek into the mechanism of our reaction, the following experiments have been done.

The substrate **1w** with a methyl group instead of the aryl group on the sp^2 carbon participated in this reaction smoothly and gave the desired product **2w** in high yield, indicating that the aryl ring on the sp^2 carbon was not a requisite to stabilize the cation B. The aliphatic substituent could also stabilize the cation B (Figure S6 & S7).

Figure S6. Reaction of **1w**

The substrate **1aa** with a methyl group on the cyclopropane ring participated in the reaction led to unidentifiable messy products, indicating that the substituent on the cyclopropane could not stabilize the cation B.

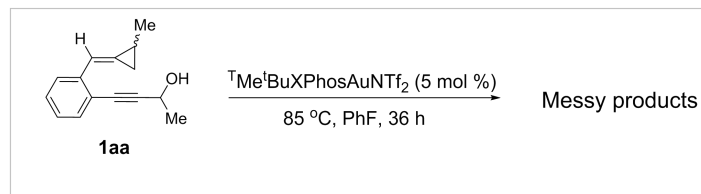


Figure S7. Reaction of **1aa**

The *6-endo-dig* cyclization product was the only product, this outcome led by the use of an electron-rich and sterically hindered ligand which would make the cationic intermediate less electropositive and then slowed the rate of deauration by proton. Furthermore, the naphthalene would be preferred due to its aromatic and thermodynamically stable characters. Thus, the *6-endo-dig* cyclization was favored. The *5-exo-dig* cyclization was disfavored because of the steric hindrance effect between R¹ and ^tBu group of cation **B'** (Figure S8).

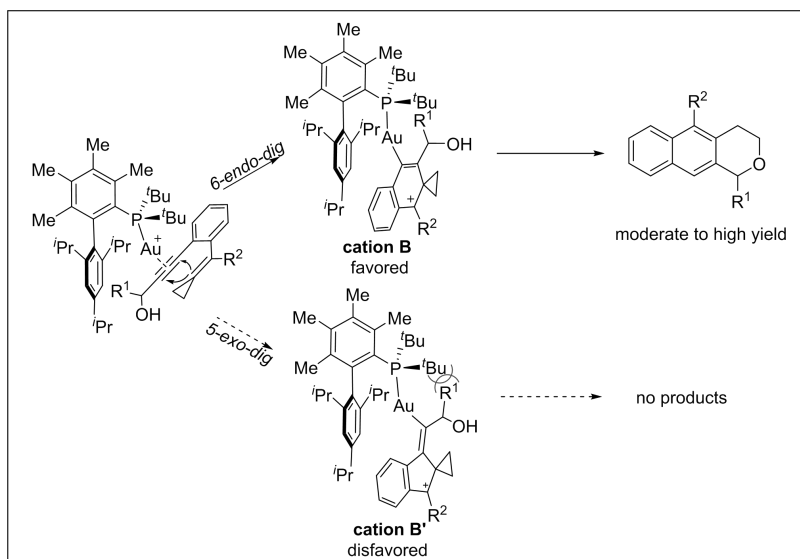
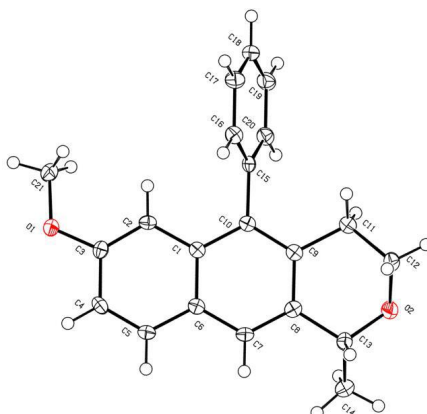


Figure S8. Analysis of reaction mechanisms

7. X-ray structure of **2v**.Figure S9. X-ray structure of **2v****Table S2** Crystal data and structure refinement for **2v**.

Identification code	2v
Empirical formula	C ₂₁ H ₂₀ O ₂
Formula weight	304.37
Temperature/K	100.00(10)
Crystal system	orthorhombic
Space group	Pbca
a/Å	14.8226(12)
b/Å	9.0342(7)
c/Å	23.887(2)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	3198.7(4)
Z	8
ρ _{calc} /cm ³	1.264
μ/mm ⁻¹	0.080
F(000)	1296.0
Crystal size/mm ³	0.14 × 0.12 × 0.11
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection/°	4.38 to 49.998
Index ranges	-17 ≤ h ≤ 17, -10 ≤ k ≤ 9, -19 ≤ l ≤ 28
Reflections collected	9295
Independent reflections	2822 [R _{int} = 0.0433, R _{sigma} = 0.0509]
Data/restraints/parameters	2822/0/210
Goodness-of-fit on F ²	1.070
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0457, wR ₂ = 0.0904

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Final R indexes [all data] $R_1 = 0.0683$, $wR_2 = 0.1023$

Largest diff. peak/hole / $e \text{ \AA}^{-3}$ 0.21/-0.19

Table S3 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for **2v**. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	z	U(eq)
O1	3364.3(9)	4433.7(13)	5139.0(5)	22.6(3)
O2	3727.3(8)	8233.3(14)	1770.6(5)	23.8(3)
C9	4079.8(12)	7603(2)	2902.6(8)	16.4(4)
C1	3779.9(11)	6147.5(19)	3745.9(7)	14.1(4)
C6	3321.9(11)	5079(2)	3410.4(8)	15.3(4)
C10	4172.8(11)	7401.1(19)	3476.5(7)	14.8(4)
C3	3417.8(12)	4708(2)	4574.5(8)	17.4(4)
C8	3578.3(12)	6572(2)	2575.9(8)	17.2(4)
C15	4706.2(12)	8485(2)	3816.8(7)	16.7(4)
C5	2971.8(12)	3792(2)	3672.6(8)	17.2(4)
C4	3015.5(12)	3599(2)	4236.1(8)	19.3(4)
C2	3807.5(11)	5930(2)	4337.1(8)	15.9(4)
C7	3229.7(12)	5333(2)	2831.1(8)	16.5(4)
C16	5537.2(12)	8063(2)	4036.6(8)	21.0(5)
C20	4402.7(13)	9922(2)	3922.1(8)	20.8(5)
C13	3475.2(13)	6777(2)	1945.3(8)	20.2(4)
C18	5739.0(14)	10447(2)	4457.4(8)	24.7(5)
C11	4518.6(13)	8905(2)	2610.2(8)	21.5(5)
C17	6051.3(13)	9036(2)	4354.9(8)	24.8(5)
C19	4914.5(13)	10894(2)	4238.9(8)	24.0(5)
C12	4591.5(13)	8641(2)	1986.4(8)	24.6(5)
C21	3674.4(14)	5582(2)	5504.0(8)	25.1(5)
C14	2522.4(14)	6557(2)	1734.8(8)	28.1(5)

Table S4 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for **2v**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^*2U_{11}+2hka^*b^*U_{12}+\dots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	32.1(8)	19.9(7)	15.6(7)	0.9(6)	-0.1(6)	-5.3(6)
O2	24.4(7)	25.4(8)	21.7(8)	5.9(6)	-4.8(6)	-6.0(6)
C9	14.5(9)	15.7(10)	19.0(10)	-0.7(8)	1.3(8)	1.3(8)
C1	10.1(9)	14.8(10)	17.3(10)	-0.6(8)	1.7(7)	2.9(8)
C6	10.9(9)	16.5(10)	18.6(11)	-0.9(8)	1.0(8)	1.7(8)
C10	13.0(9)	13.9(10)	17.6(10)	-1.8(8)	1.5(8)	2.1(8)

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C3	16.9(10)	19.5(11)	15.9(11)	1.6(8)	1.1(8)	2.3(9)
C8	15.4(10)	18.6(10)	17.5(11)	-1.8(8)	0.6(8)	2.7(8)
C15	17.9(10)	19.5(10)	12.8(10)	0.8(8)	4.0(8)	-3.9(8)
C5	16.9(10)	14.9(10)	19.9(11)	-2.8(8)	-1.7(8)	-1.2(8)
C4	18.4(10)	15.5(10)	23.9(11)	3.8(9)	0.5(8)	-3.2(8)
C2	13.2(9)	16.4(10)	18.1(11)	-3.5(8)	-0.5(8)	-0.5(8)
C7	15.0(9)	16.1(10)	18.3(11)	-4.3(8)	-1.1(8)	-0.2(8)
C16	21.6(10)	19.5(11)	21.9(11)	0.4(9)	0.2(9)	-0.5(9)
C20	20.6(10)	20.5(11)	21.3(11)	0.9(8)	1.3(8)	-1.4(9)
C13	24.6(10)	18.8(11)	17.3(11)	1.9(8)	0.3(8)	-1.1(9)
C18	31.9(12)	24.3(11)	17.9(11)	-1.5(9)	-1.1(9)	-11.4(10)
C11	21.6(10)	22.3(11)	20.6(11)	0.8(9)	-0.5(8)	-4.7(9)
C17	19.7(10)	28.9(12)	25.9(12)	1.6(9)	-5.2(9)	-5.0(9)
C19	32.0(12)	17.5(11)	22.6(12)	-3.3(9)	4.1(9)	-3.1(9)
C12	24.7(11)	28.8(11)	20.1(11)	5.4(9)	-0.3(9)	-8.1(10)
C21	35.1(12)	23.7(11)	16.4(11)	-1.1(9)	0.5(9)	-3.7(10)
C14	31.0(12)	31.3(12)	22.1(11)	2.4(10)	-7.7(9)	-7.3(10)

Table S5 Bond Lengths for **2v**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C3	1.373(2)	C3	C4	1.419(3)
O1	C21	1.431(2)	C3	C2	1.369(3)
O2	C13	1.430(2)	C8	C7	1.375(3)
O2	C12	1.429(2)	C8	C13	1.525(3)
C9	C10	1.390(3)	C15	C16	1.392(3)
C9	C8	1.424(3)	C15	C20	1.397(3)
C9	C11	1.515(3)	C5	C4	1.359(3)
C1	C6	1.426(2)	C16	C17	1.390(3)
C1	C10	1.427(2)	C20	C19	1.385(3)
C1	C2	1.426(3)	C13	C14	1.512(3)
C6	C5	1.419(2)	C18	C17	1.379(3)
C6	C7	1.409(3)	C18	C19	1.389(3)
C10	C15	1.498(2)	C11	C12	1.513(3)

Table S6 Bond Angles for **2v**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C3	O1	C21	116.67(14)	C7	C8	C13	119.94(16)

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C12	O2	C13	111.49(14)	C16	C15	C10	119.50(16)
C10	C9	C8	120.44(17)	C16	C15	C20	118.14(17)
C10	C9	C11	120.92(16)	C20	C15	C10	122.36(16)
C8	C9	C11	118.64(16)	C4	C5	C6	121.68(17)
C6	C1	C10	118.55(16)	C5	C4	C3	119.58(18)
C6	C1	C2	118.46(16)	C3	C2	C1	120.63(17)
C2	C1	C10	122.98(16)	C8	C7	C6	122.06(17)
C5	C6	C1	118.71(16)	C17	C16	C15	121.23(18)
C7	C6	C1	119.22(17)	C19	C20	C15	120.74(18)
C7	C6	C5	122.07(17)	O2	C13	C8	111.97(15)
C9	C10	C1	120.55(16)	O2	C13	C14	105.56(15)
C9	C10	C15	120.12(16)	C14	C13	C8	113.94(16)
C1	C10	C15	119.31(16)	C17	C18	C19	119.83(18)
O1	C3	C4	114.06(16)	C12	C11	C9	111.26(16)
C2	C3	O1	125.22(17)	C18	C17	C16	119.87(19)
C2	C3	C4	120.72(17)	C20	C19	C18	120.19(18)
C9	C8	C13	120.91(16)	O2	C12	C11	109.38(15)
C7	C8	C9	119.05(17)				

Table S7 Torsion Angles for 2v.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	C3	C4	C5	175.64(16)	C15	C16	C17	C18	0.0(3)
O1	C3	C2	C1	-176.33(16)	C15	C20	C19	C18	-0.1(3)
C9	C10	C15	C16	-109.3(2)	C5	C6	C7	C8	-179.22(17)
C9	C10	C15	C20	70.4(2)	C4	C3	C2	C1	3.0(3)
C9	C8	C7	C6	2.6(3)	C2	C1	C6	C5	-4.6(2)
C9	C8	C13	O2	-14.5(2)	C2	C1	C6	C7	175.31(16)
C9	C8	C13	C14	-134.25(18)	C2	C1	C10	C9	-176.16(16)
C9	C11	C12	O2	52.2(2)	C2	C1	C10	C15	5.2(2)
C1	C6	C5	C4	4.0(3)	C2	C3	C4	C5	-3.7(3)
C1	C6	C7	C8	0.9(3)	C7	C6	C5	C4	-175.96(17)
C1	C10	C15	C16	69.4(2)	C7	C8	C13	O2	169.31(16)
C1	C10	C15	C20	-110.9(2)	C7	C8	C13	C14	49.6(2)
C6	C1	C10	C9	2.4(2)	C16	C15	C20	C19	-0.3(3)
C6	C1	C10	C15	-176.19(15)	C20	C15	C16	C17	0.4(3)
C6	C1	C2	C3	1.2(2)	C13	O2	C12	C11	-71.2(2)
C6	C5	C4	C3	0.2(3)	C13	C8	C7	C6	178.83(16)
C10	C9	C8	C7	-3.5(3)	C11	C9	C10	C1	-178.11(16)
C10	C9	C8	C13	-179.72(16)	C11	C9	C10	C15	0.5(3)

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C10	C9	C11	C12	161.37(16)	C11	C9	C8	C7	175.59(16)
C10	C1	C6	C5	176.71(15)	C11	C9	C8	C13	-0.6(3)
C10	C1	C6	C7	-3.4(2)	C17	C18	C19	C20	0.5(3)
C10	C1	C2	C3	179.84(16)	C19	C18	C17	C16	-0.4(3)
C10	C15	C16	C17	-179.91(17)	C12	O2	C13	C8	50.3(2)
C10	C15	C20	C19	179.97(17)	C12	O2	C13	C14	174.77(15)
C8	C9	C10	C1	1.0(3)	C21	O1	C3	C4	-173.76(16)
C8	C9	C10	C15	179.62(16)	C21	O1	C3	C2	5.6(3)
C8	C9	C11	C12	-17.8(2)					

Table S8 Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for **2v**.

Atom	x	y	z	U(eq)
H5	2705.64	3062.6	3453.2	21
H4	2783.14	2745.16	4398.93	23
H2	4092.95	6624.06	4563.33	19
H7	2923.6	4640.09	2614.5	20
H16	5752.18	7112.57	3969.17	25
H20	3850.91	10229.98	3777.89	25
H13	3868.53	6063.29	1756.12	24
H18	6079.6	11098.91	4672.41	30
H11A	4164.84	9790.35	2677.57	26
H11B	5116.32	9063.35	2764.27	26
H17	6605.01	8734.94	4498.41	30
H19	4705.33	11848.7	4305.48	29
H12A	4801.57	9534.19	1802.3	29
H12B	5023.48	7858.32	1913.51	29
H21A	3377.38	6493.74	5410.74	38
H21B	4314.39	5700.22	5461.39	38
H21C	3539.46	5323.08	5884.63	38
H14A	2125.38	7234.19	1922.45	42
H14B	2334.17	5559.5	1809.5	42
H14C	2502.89	6737	1338.95	42

Experimental

A suitable crystal was selected and determined on a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer. The crystal was kept at 100.00(10) K during data collection.

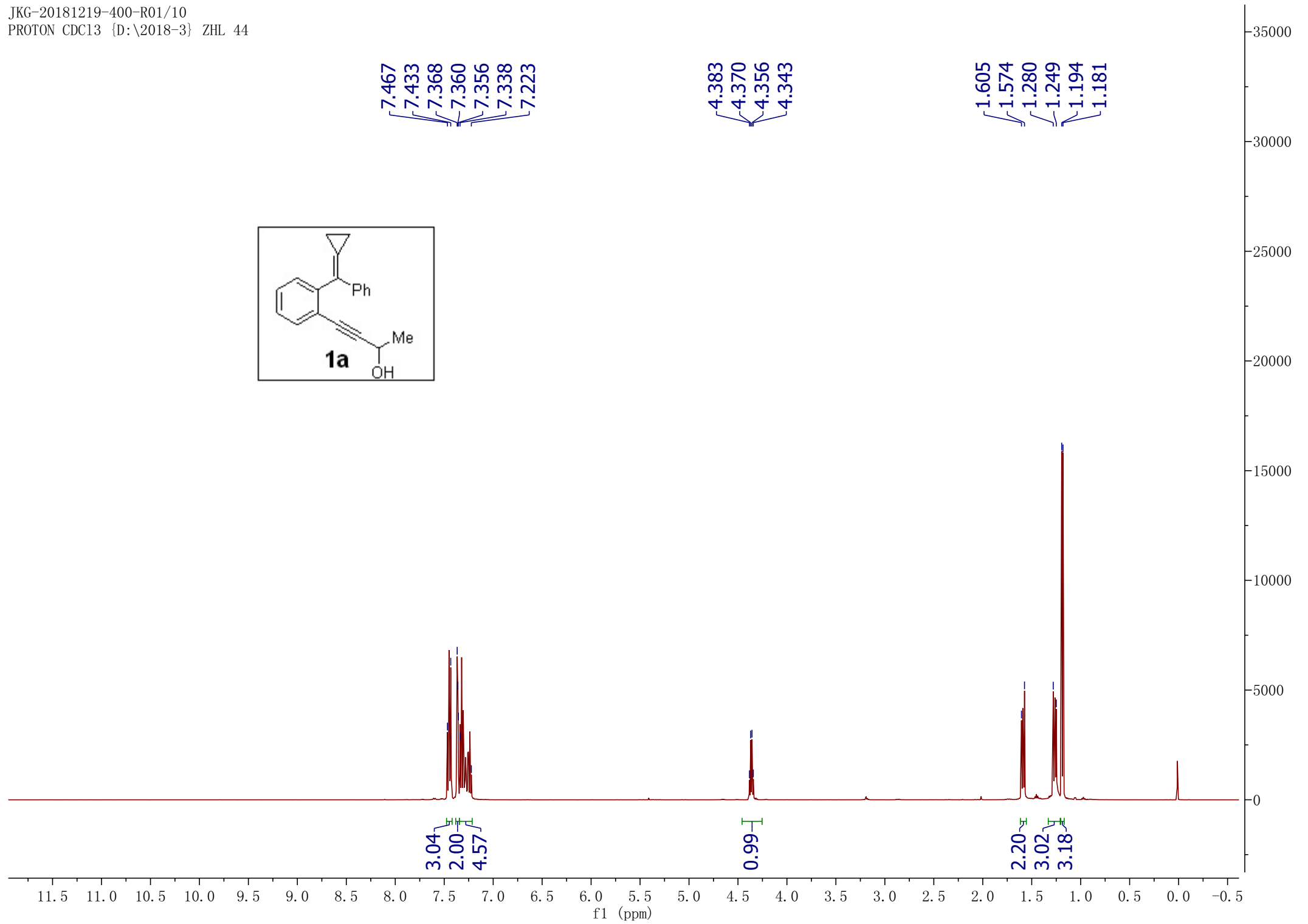
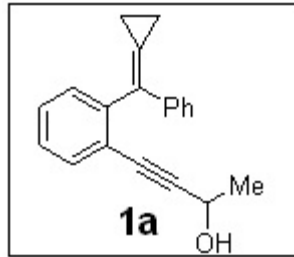
Crystal structure determination of **2v**

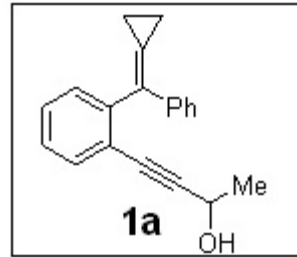
Electronic Supplementary Information

Crystal Data for C₂₁H₂₀O₂ ($M = 304.37$ g/mol): orthorhombic, space group Pbc_a (no. 61), $a = 14.8226(12)$ Å, $b = 9.0342(7)$ Å, $c = 23.887(2)$ Å, $V = 3198.7(4)$ Å³, $Z = 8$, $T = 100.00(10)$ K, $\mu(\text{Mo K}\alpha) = 0.080$ mm⁻¹, $D_{\text{calc}} = 1.264$ g/cm³, 9295 reflections measured ($4.38 \leq 2\theta \leq 49.998$), 2822 unique ($R_{\text{int}} = 0.0433$, $R_{\text{sigma}} = 0.0509$) which were used in all calculations. The final R_1 was 0.0457 ($I > 2\sigma(I)$) and wR_2 was 0.1023 (all data).

8. Reference:

1. P.-H. Li, L.-Z. Yu, X.-Y. Zhang, M. Shi, *Org. Lett.*, 2018, **20**, 4516.
2. A. Smeyanov, A. Schmidt, *Synthetic Communications*, 2013, **43**, 2809.
3. V. Gudla and R. Balamurugan, *J. Org. Chem.*, 2011, **76**, 9919.





143.99
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132.52
130.43
129.42
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126.37
122.34

—95.08

83.92

77.41 CDC13

77.16 CDC13

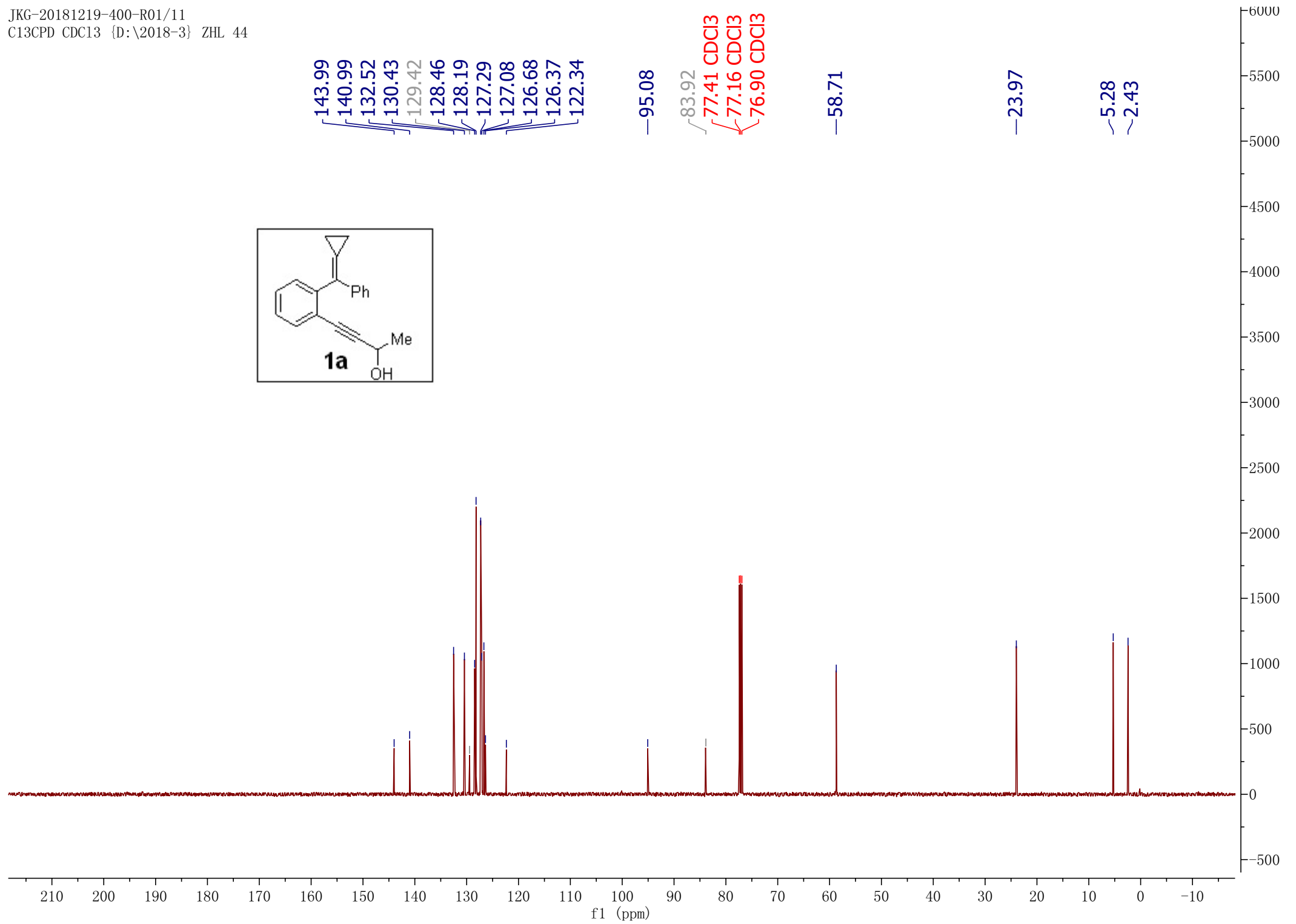
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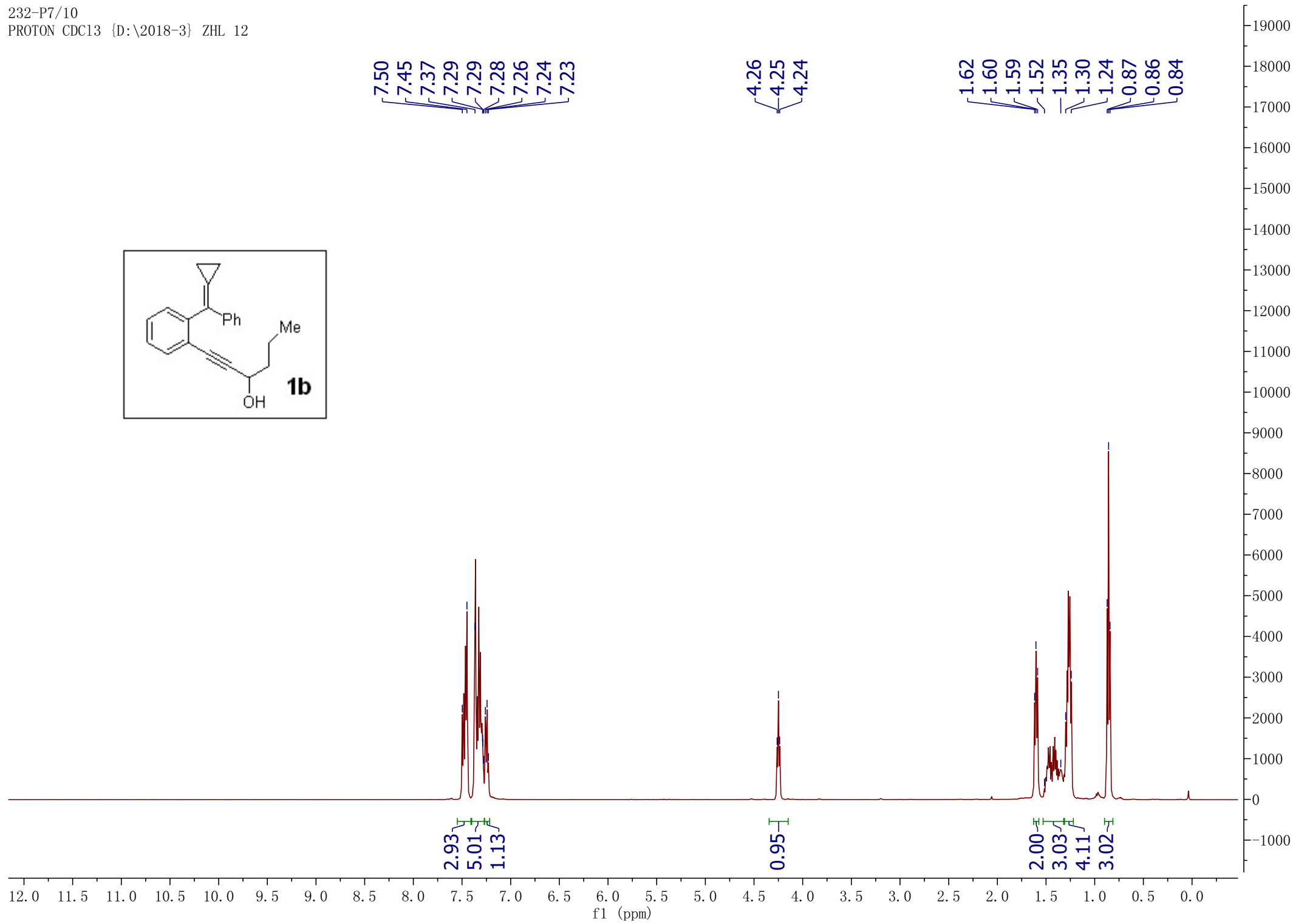
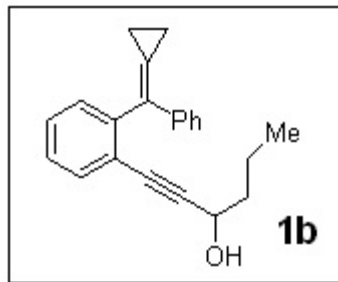
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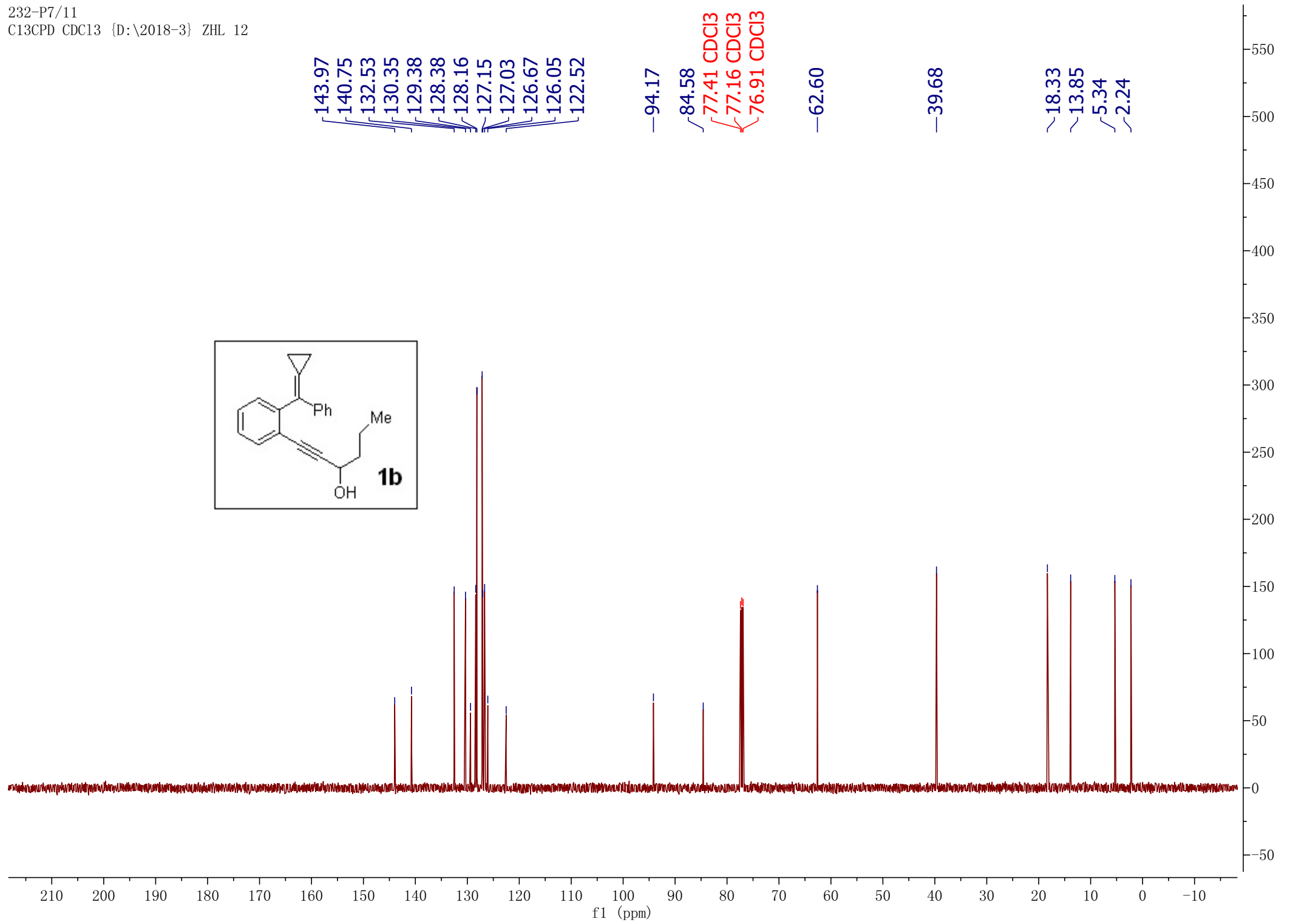
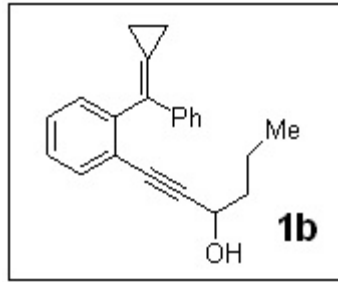
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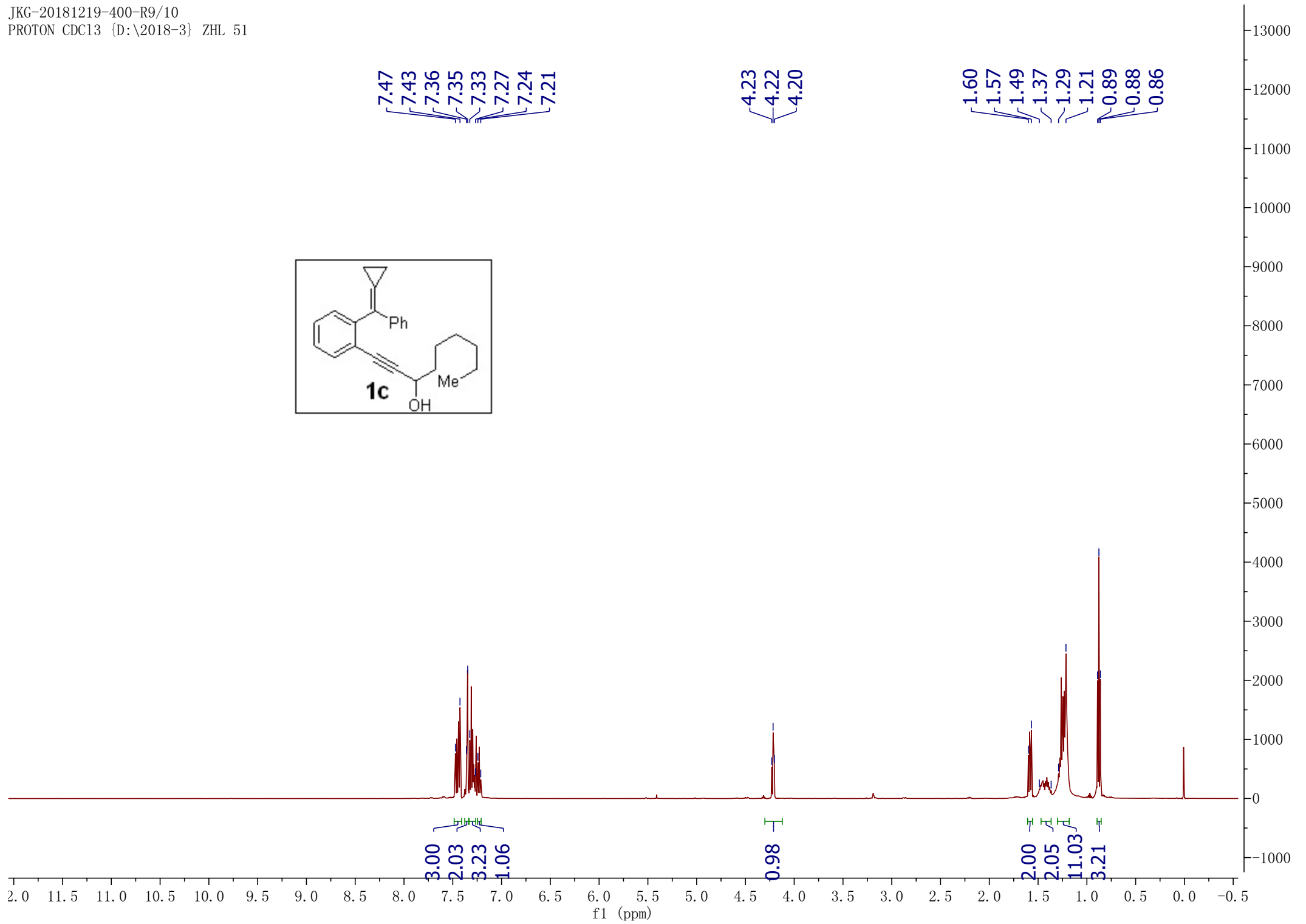
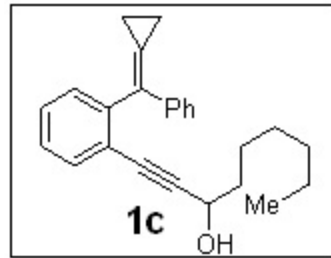
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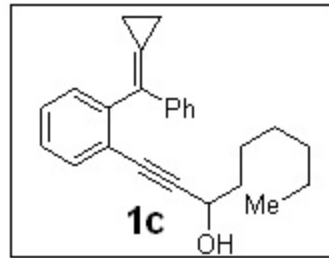
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77.41 CDC13

77.16 CDC13

76.91 CDC13

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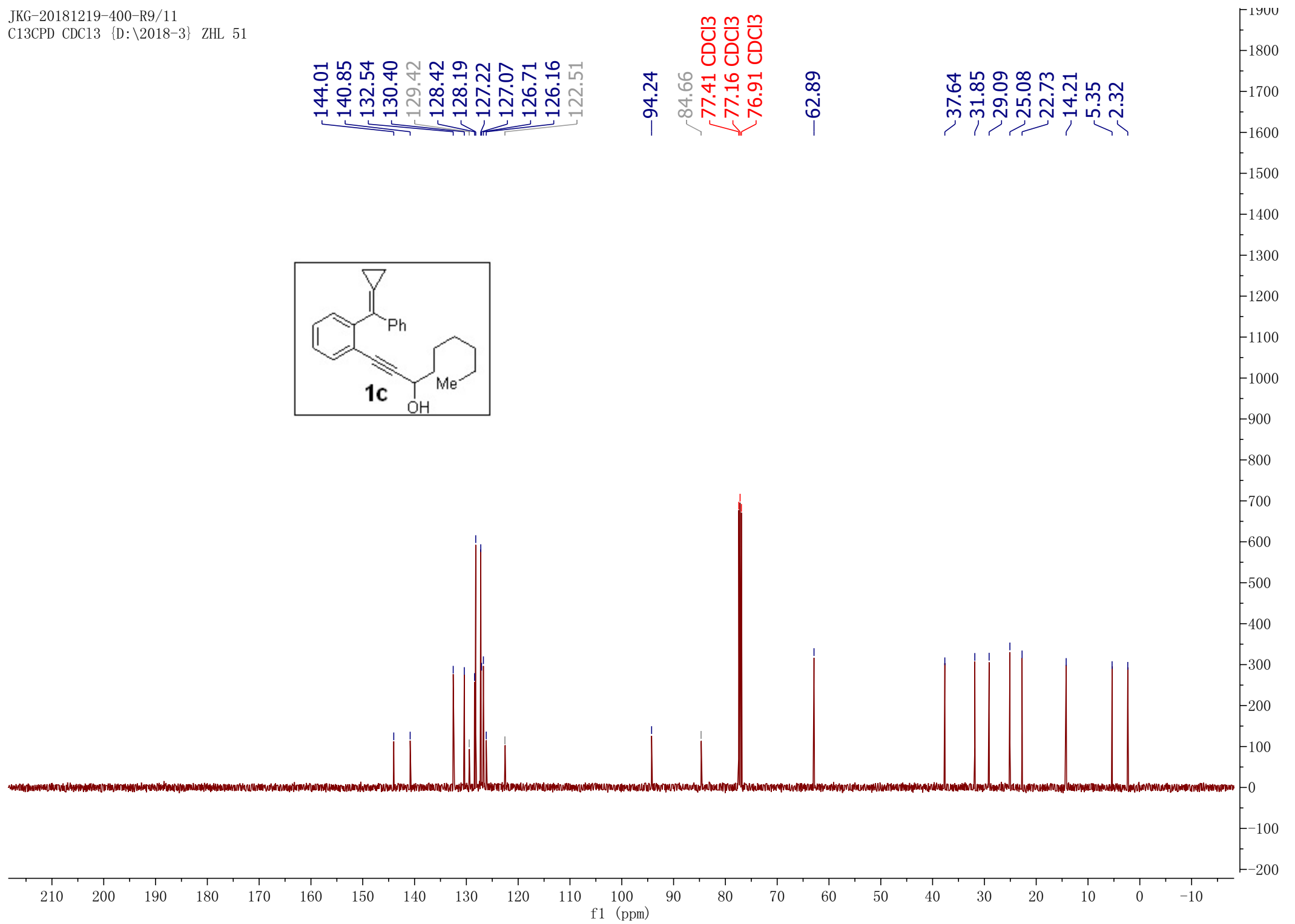
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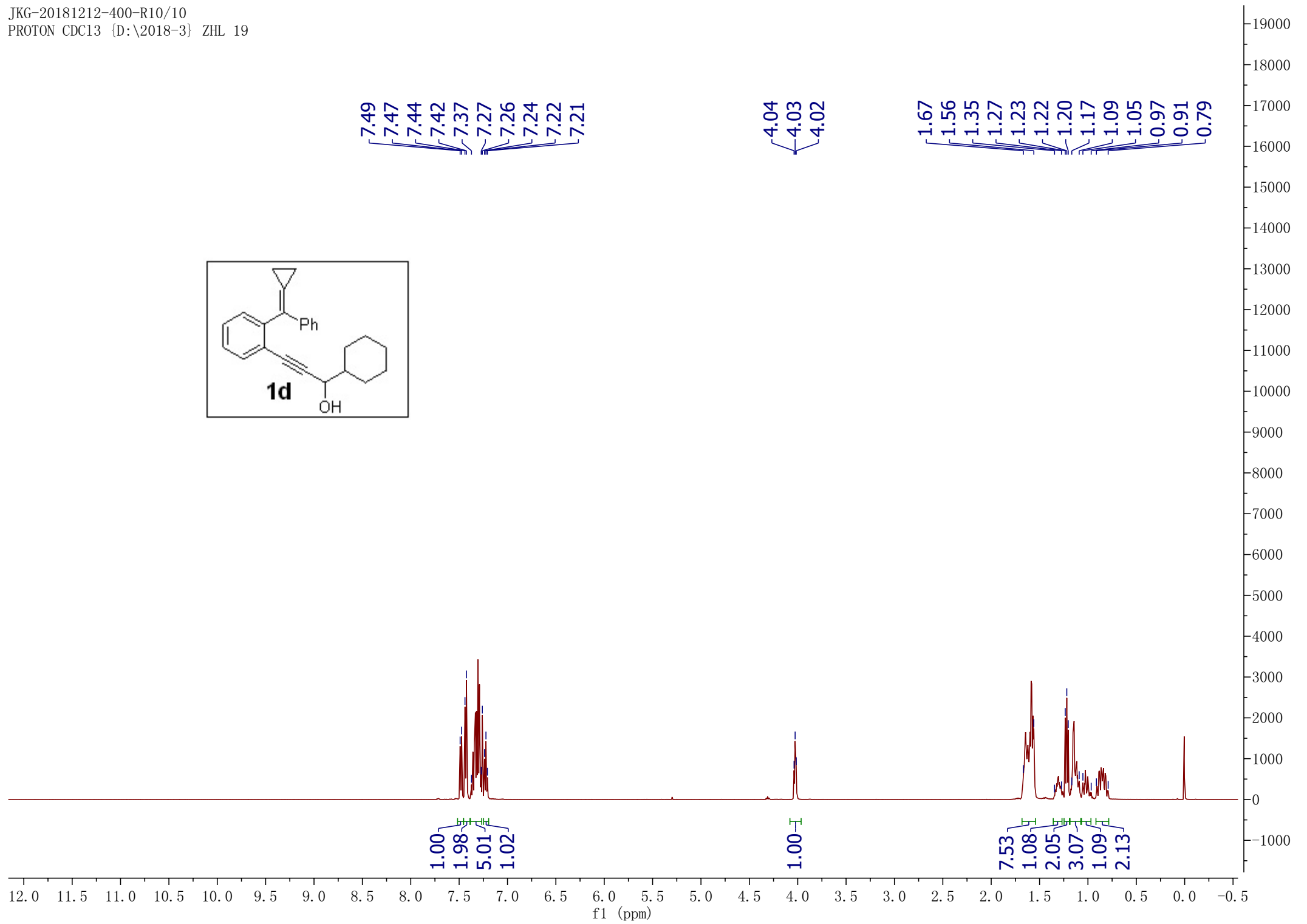
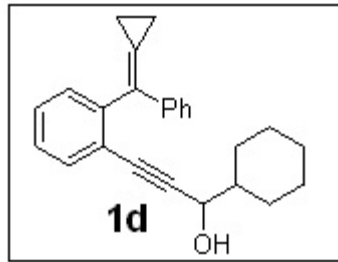
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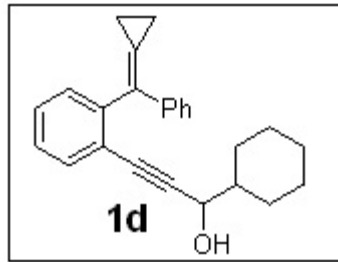
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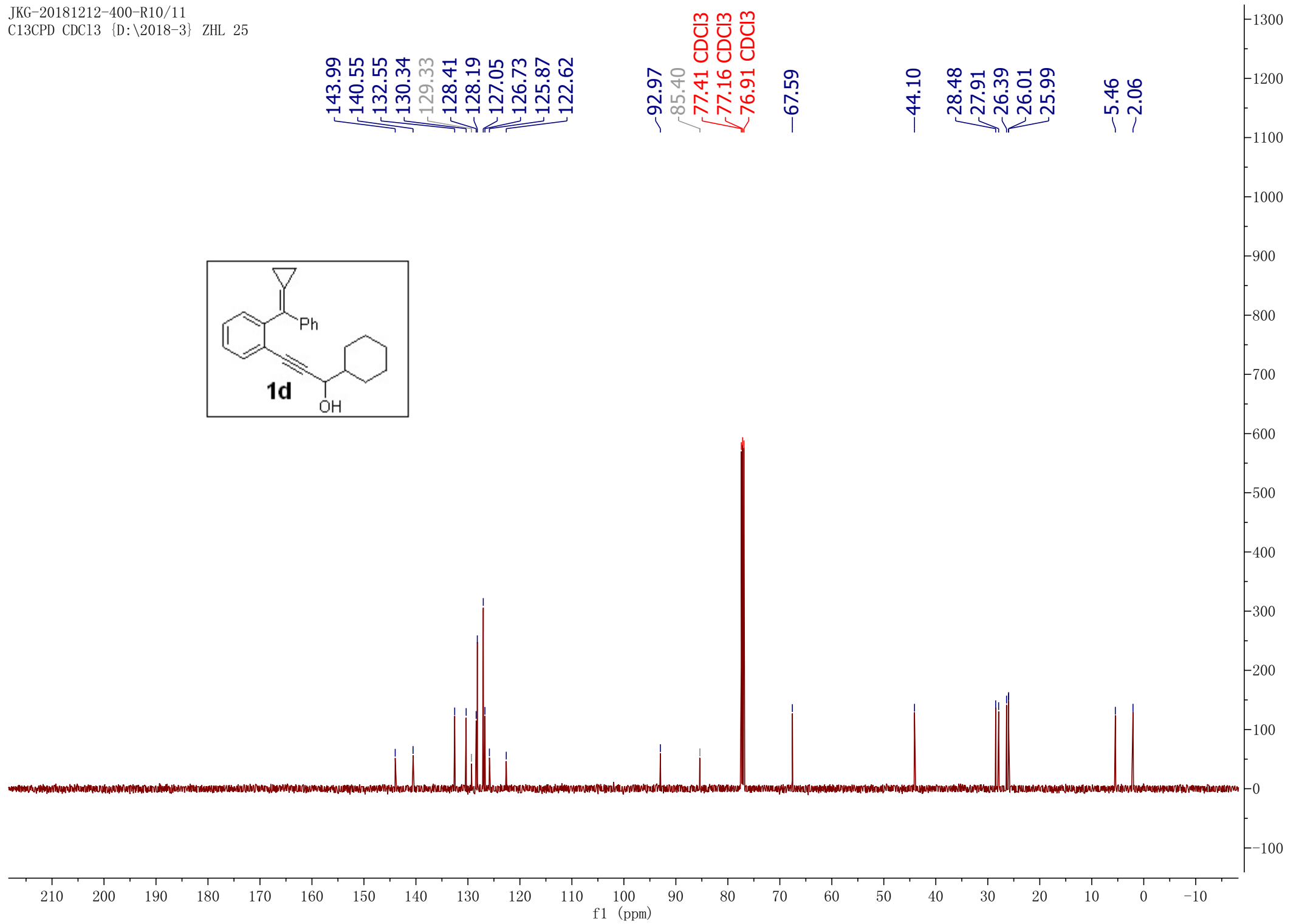
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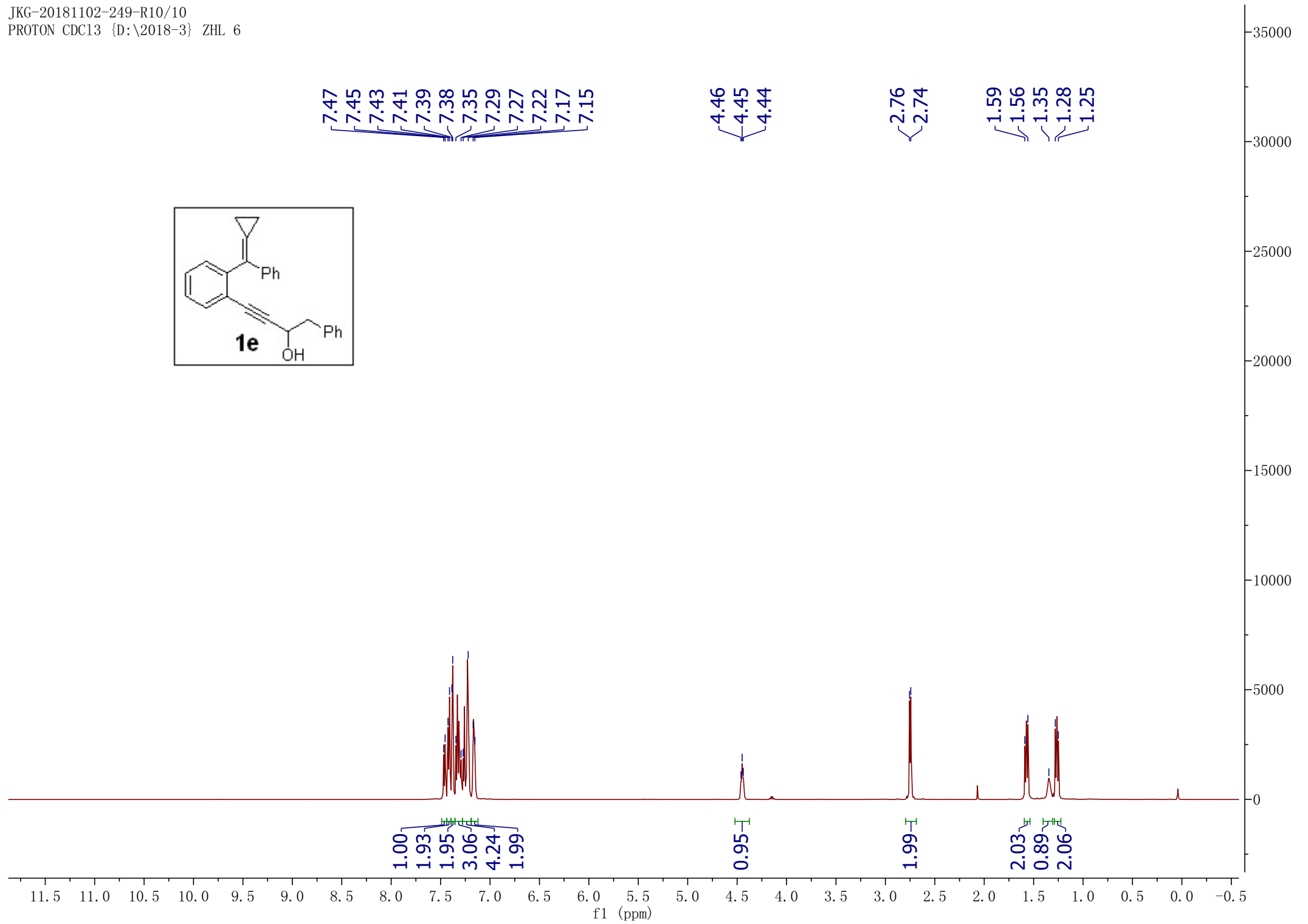
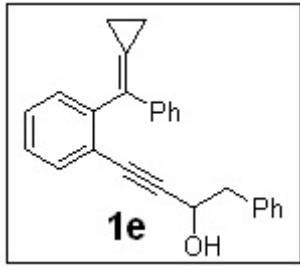
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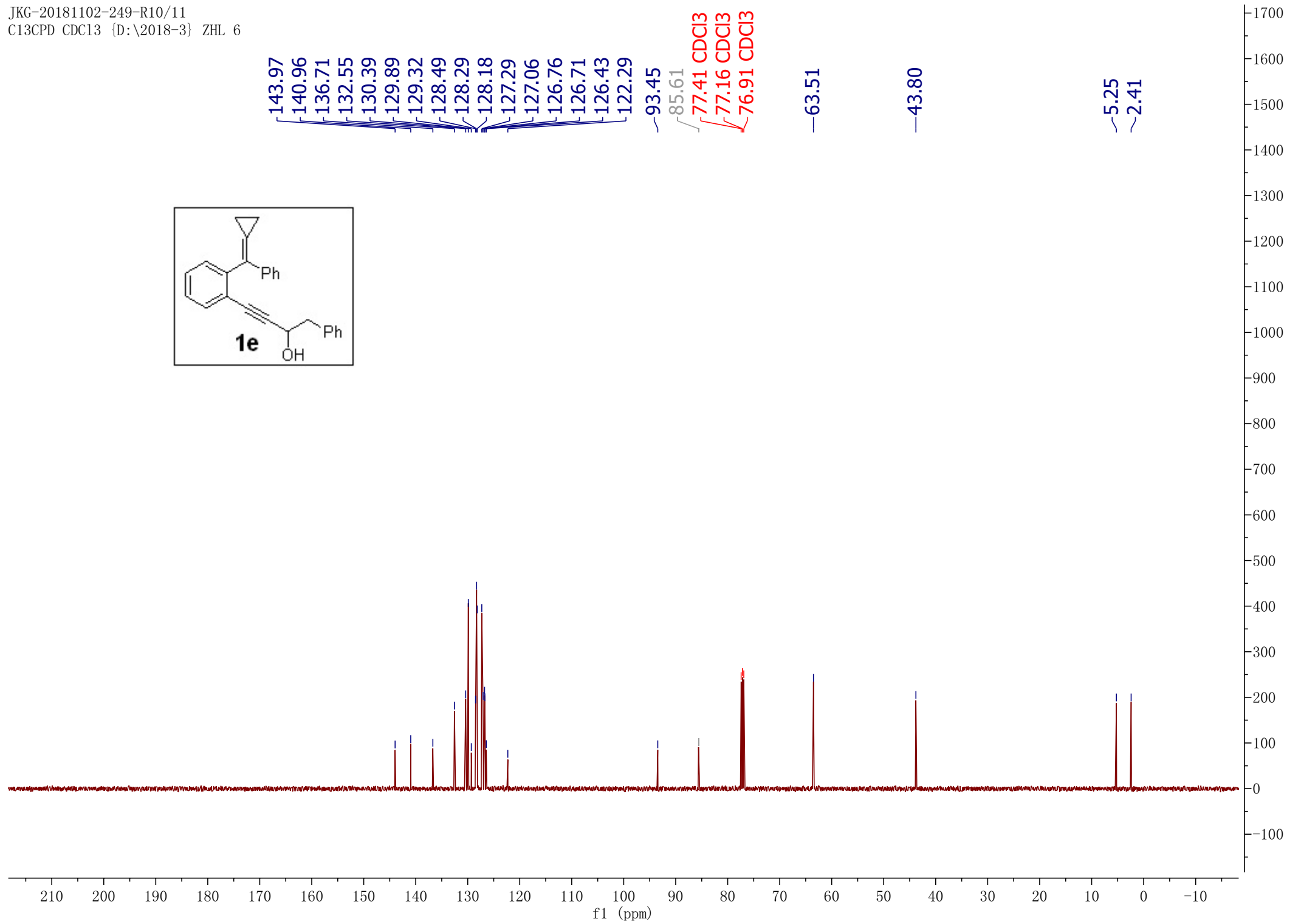
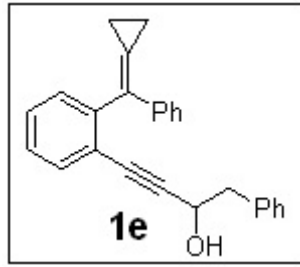
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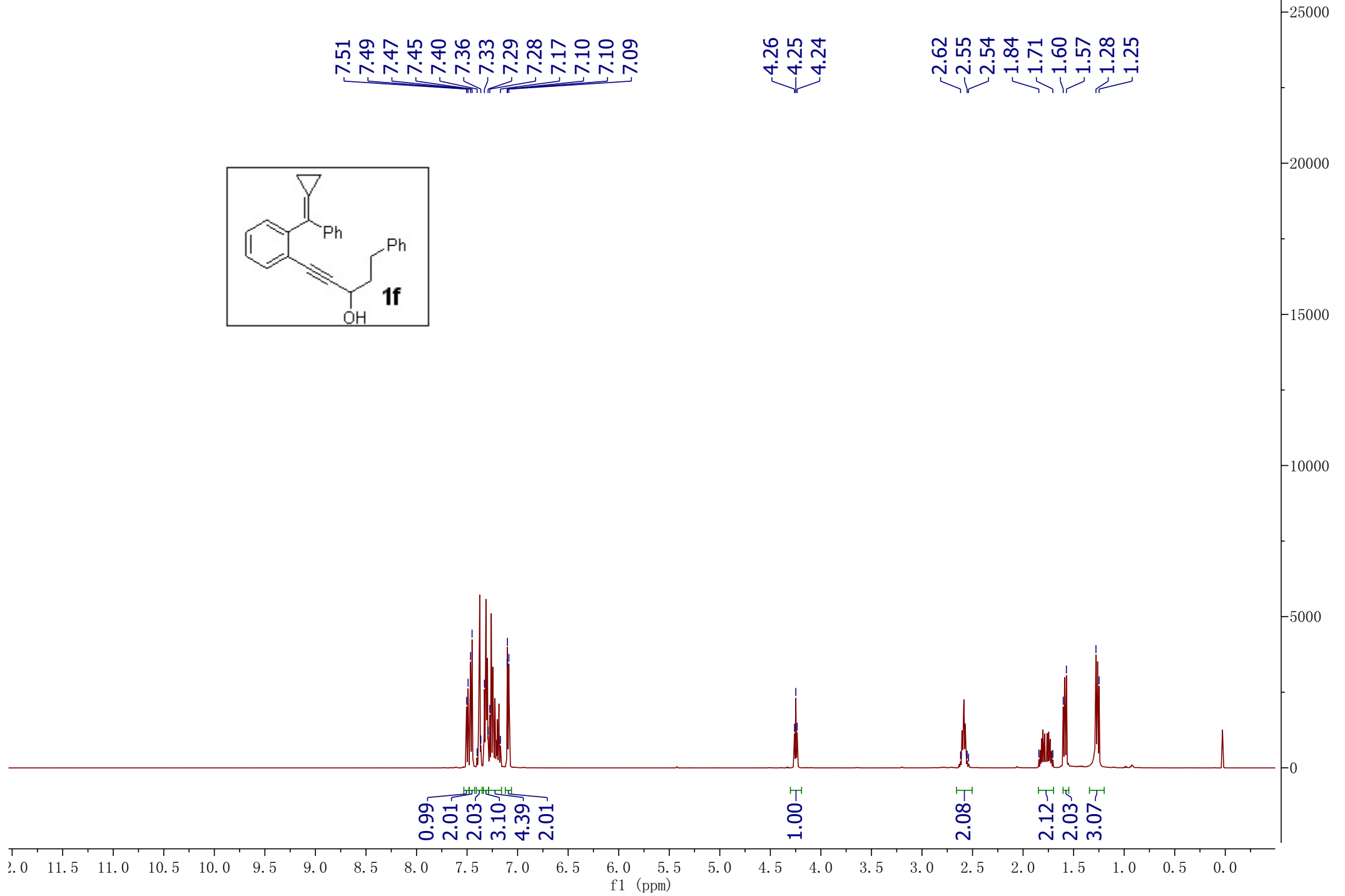
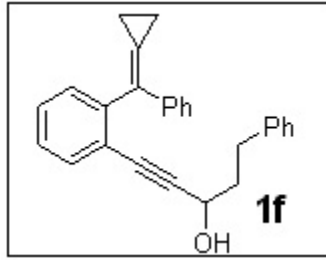
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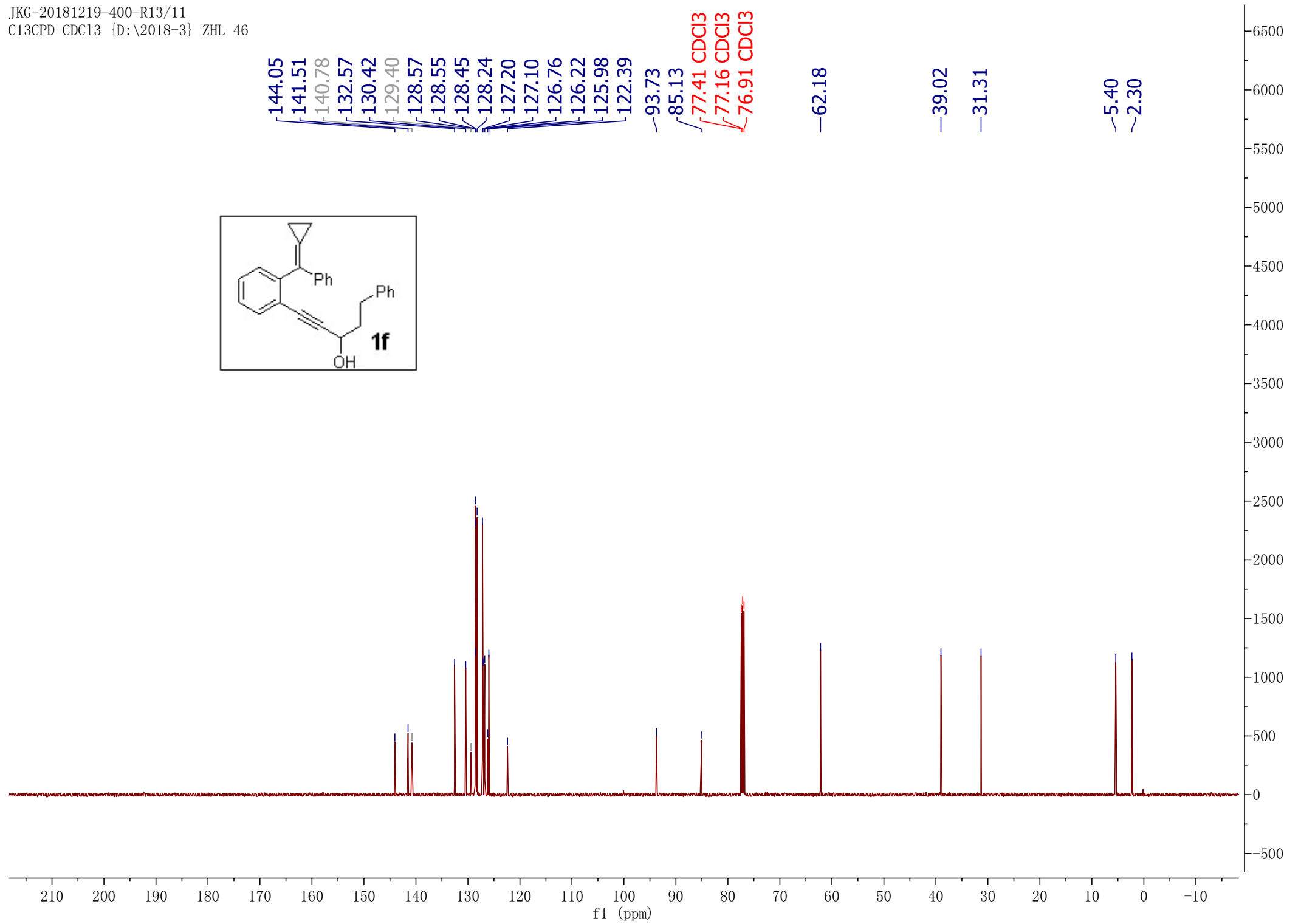
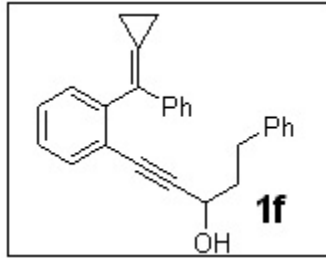
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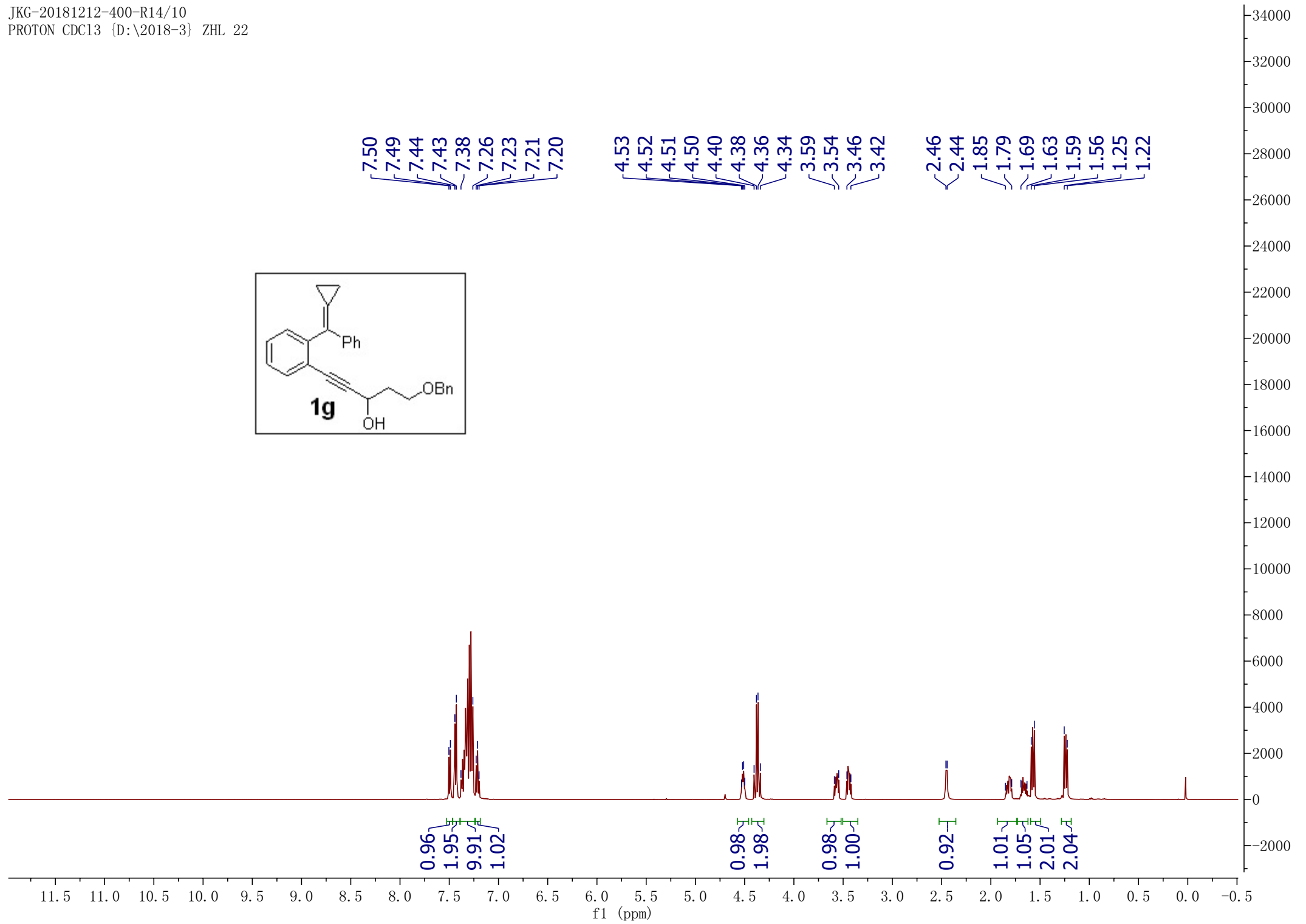
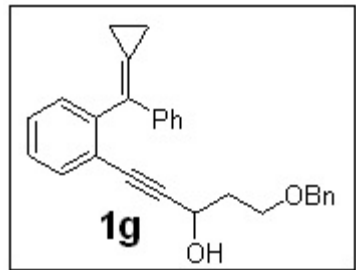


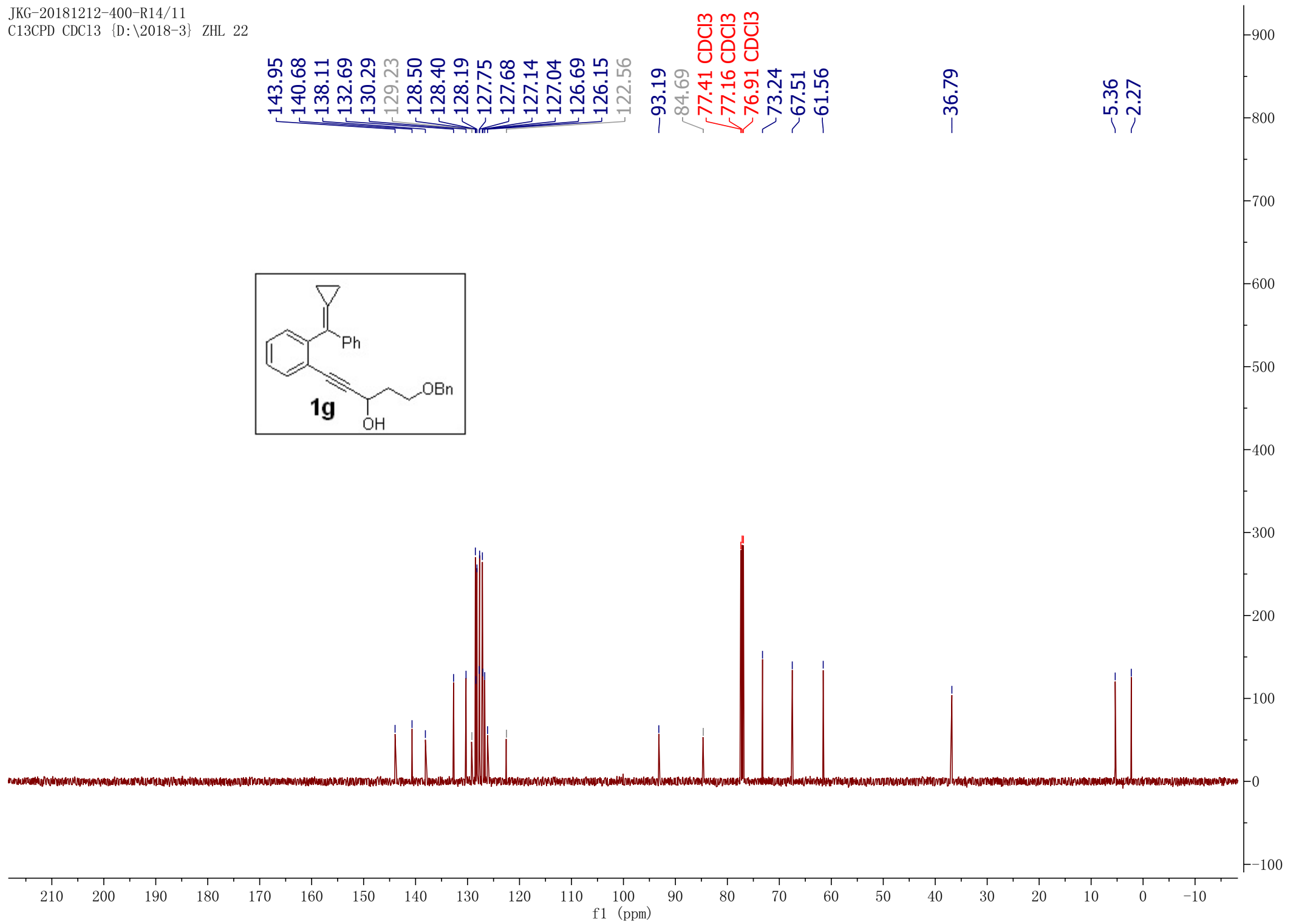
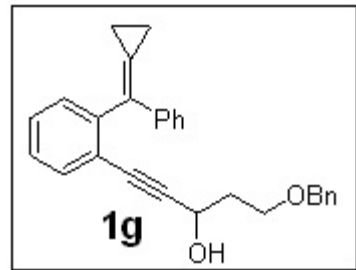












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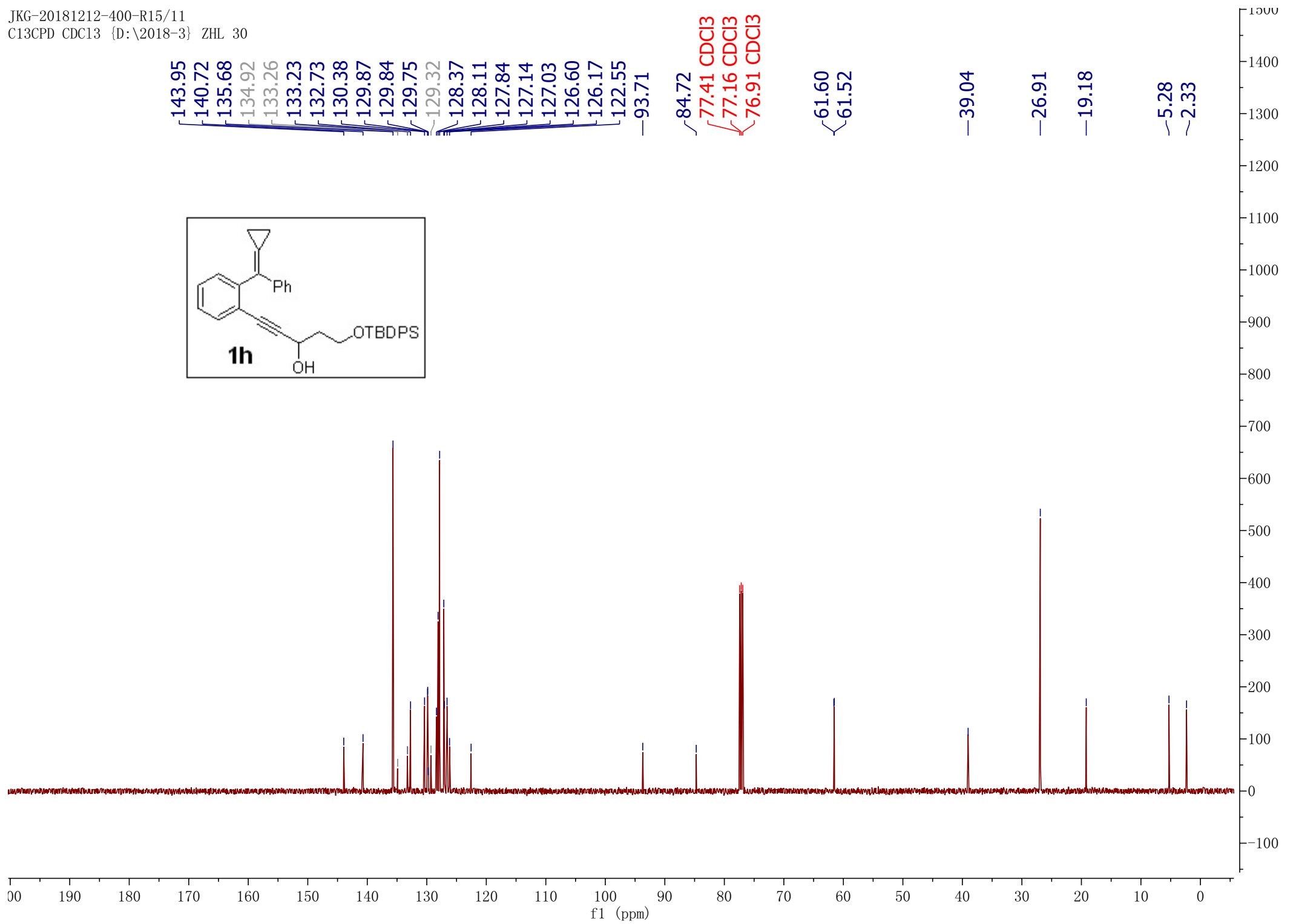
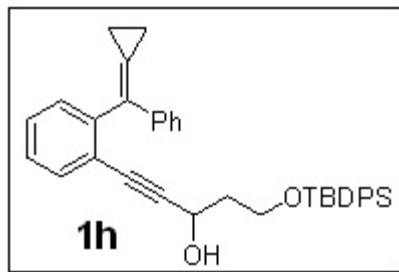
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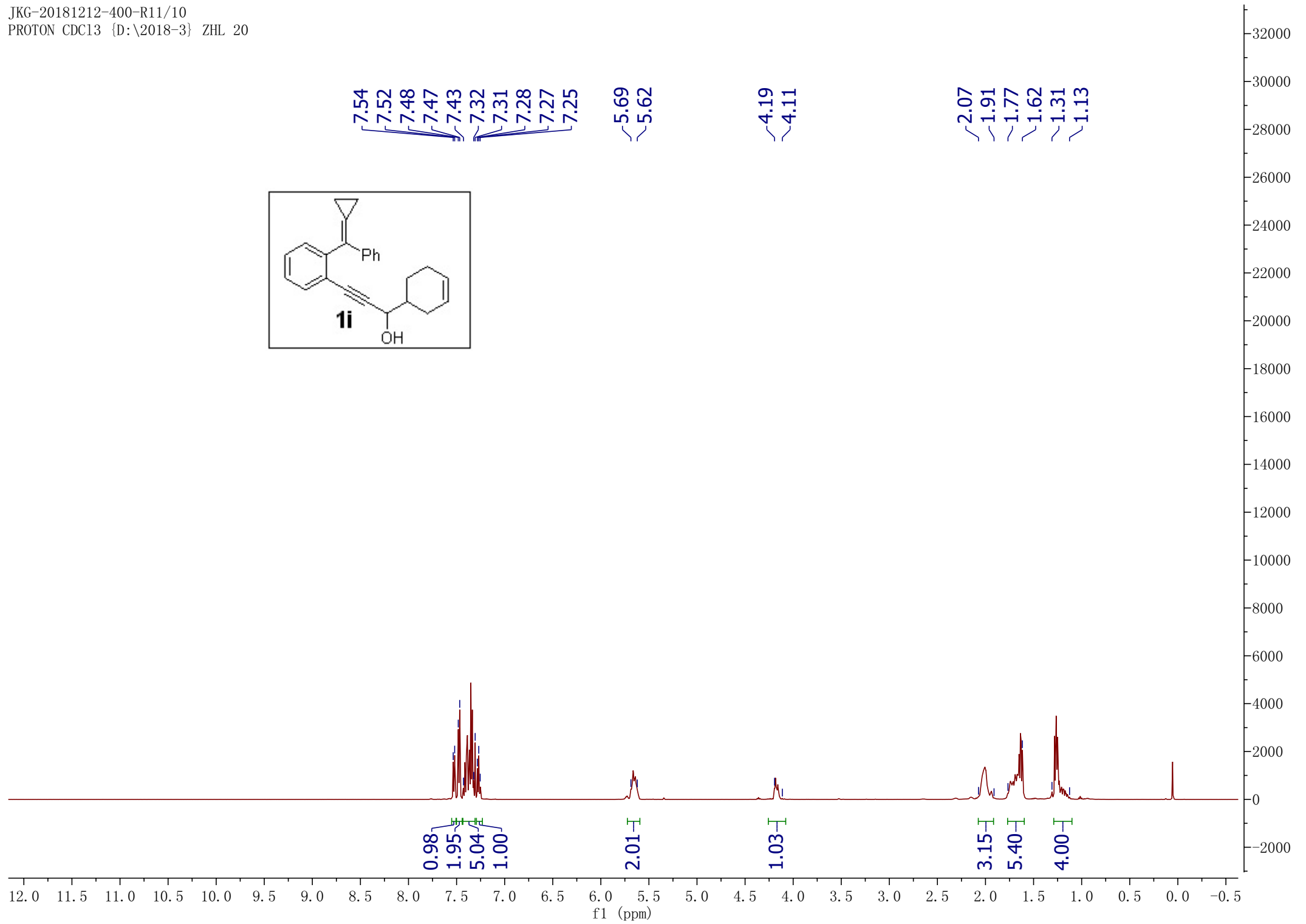
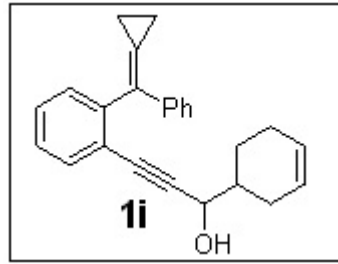
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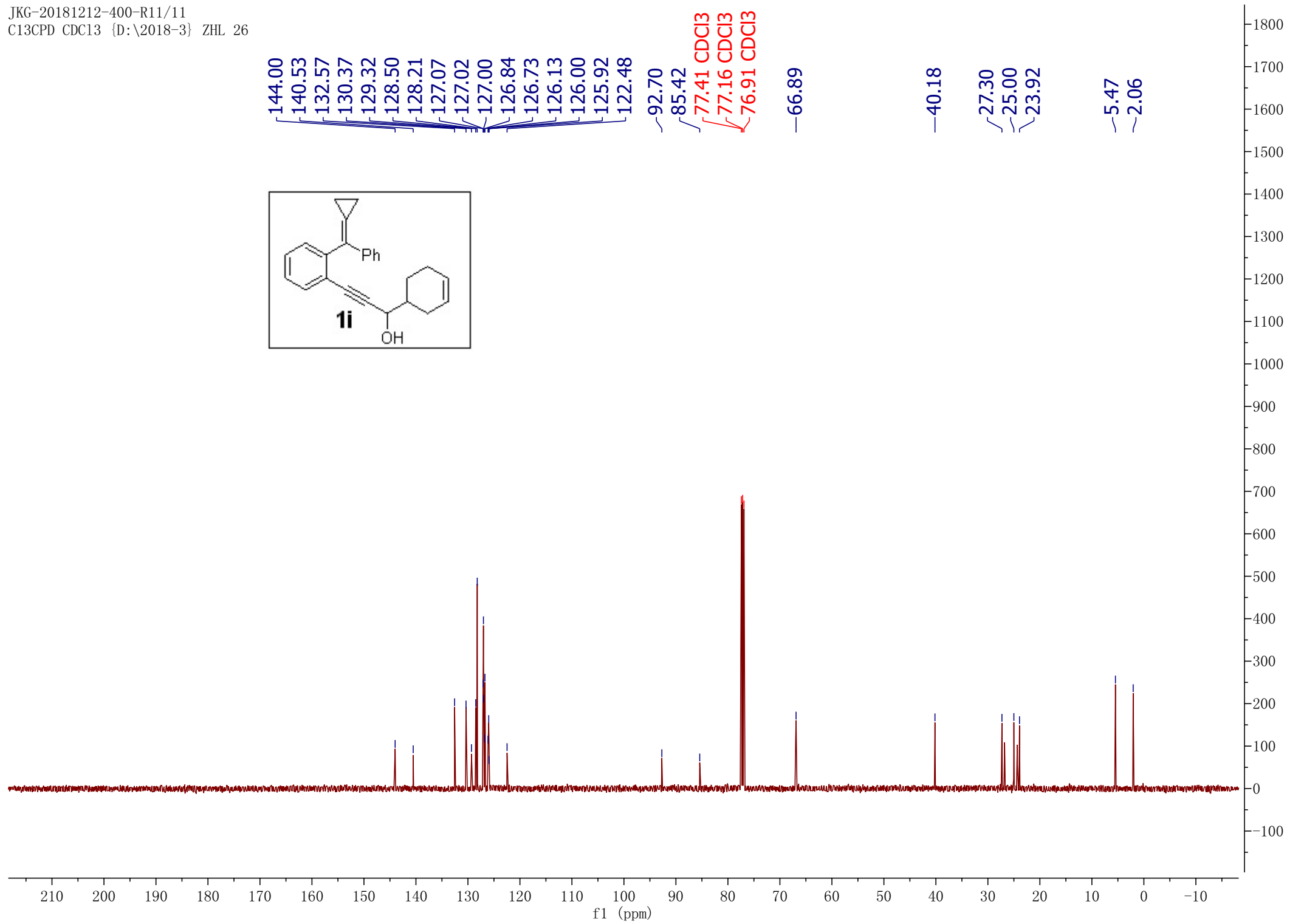
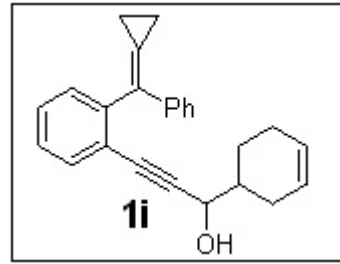
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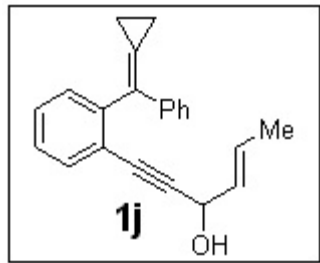
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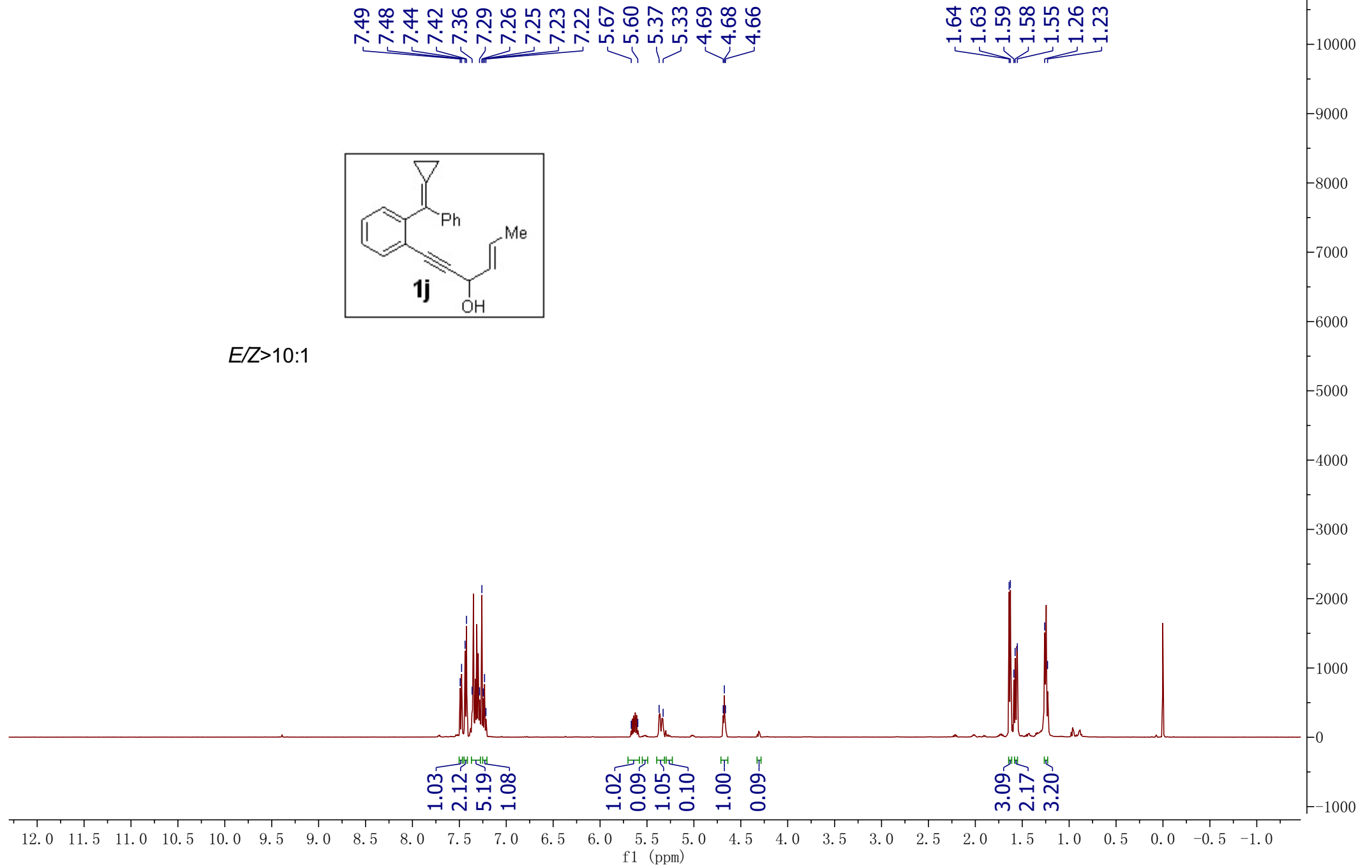








E/Z > 10:1



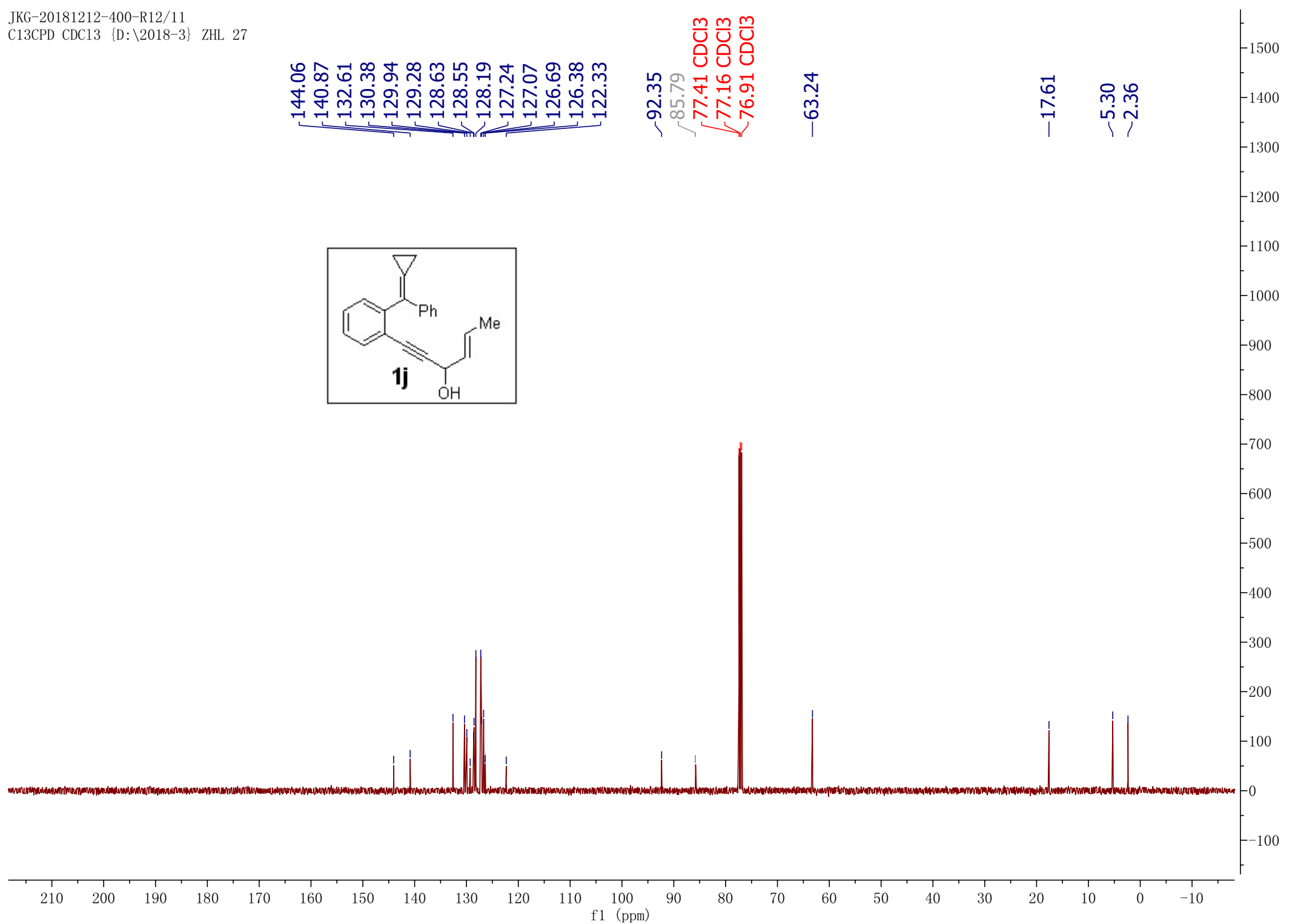
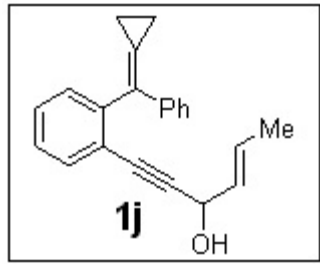
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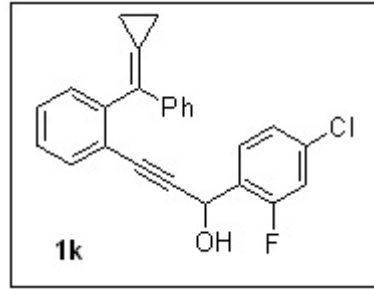
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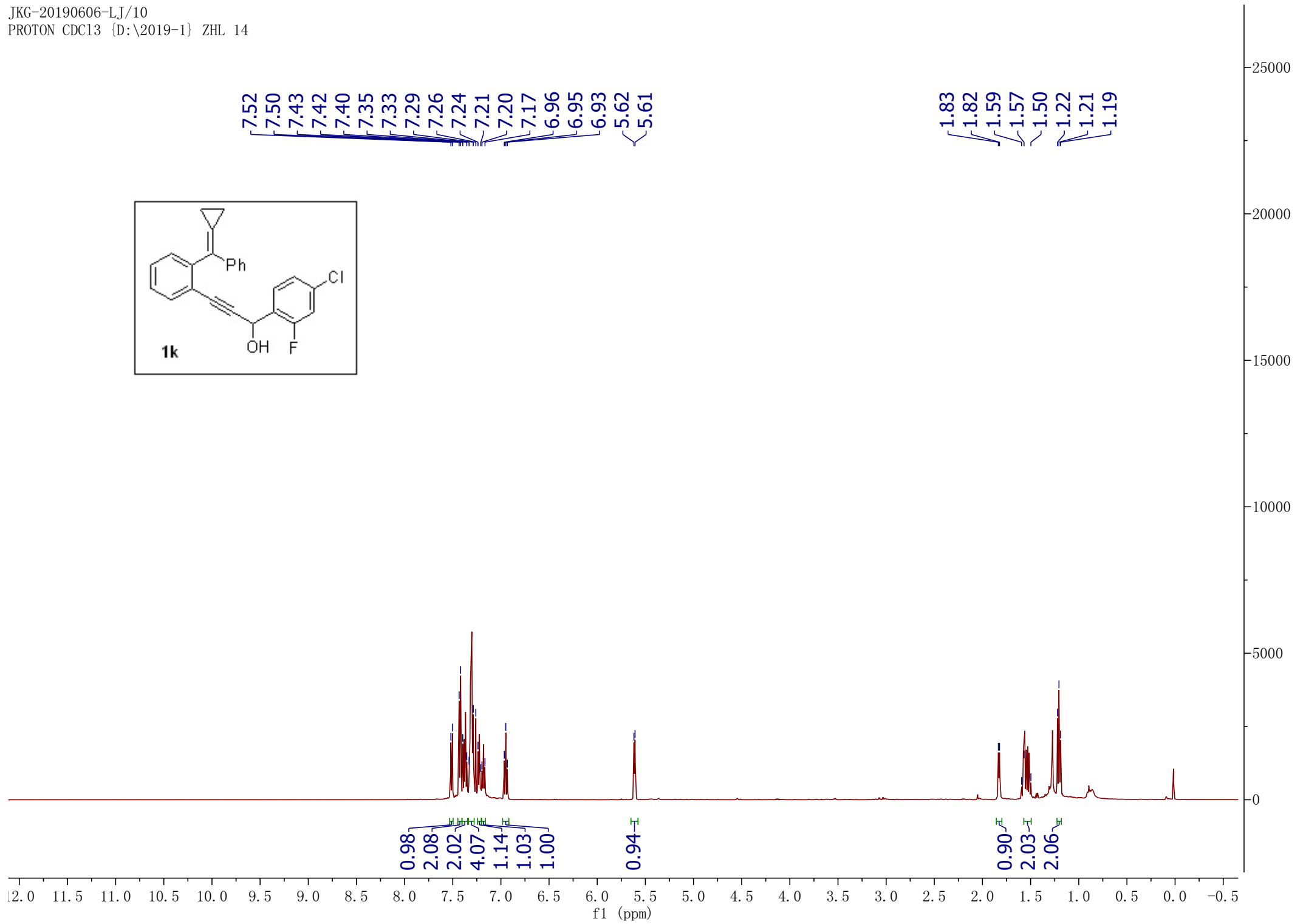
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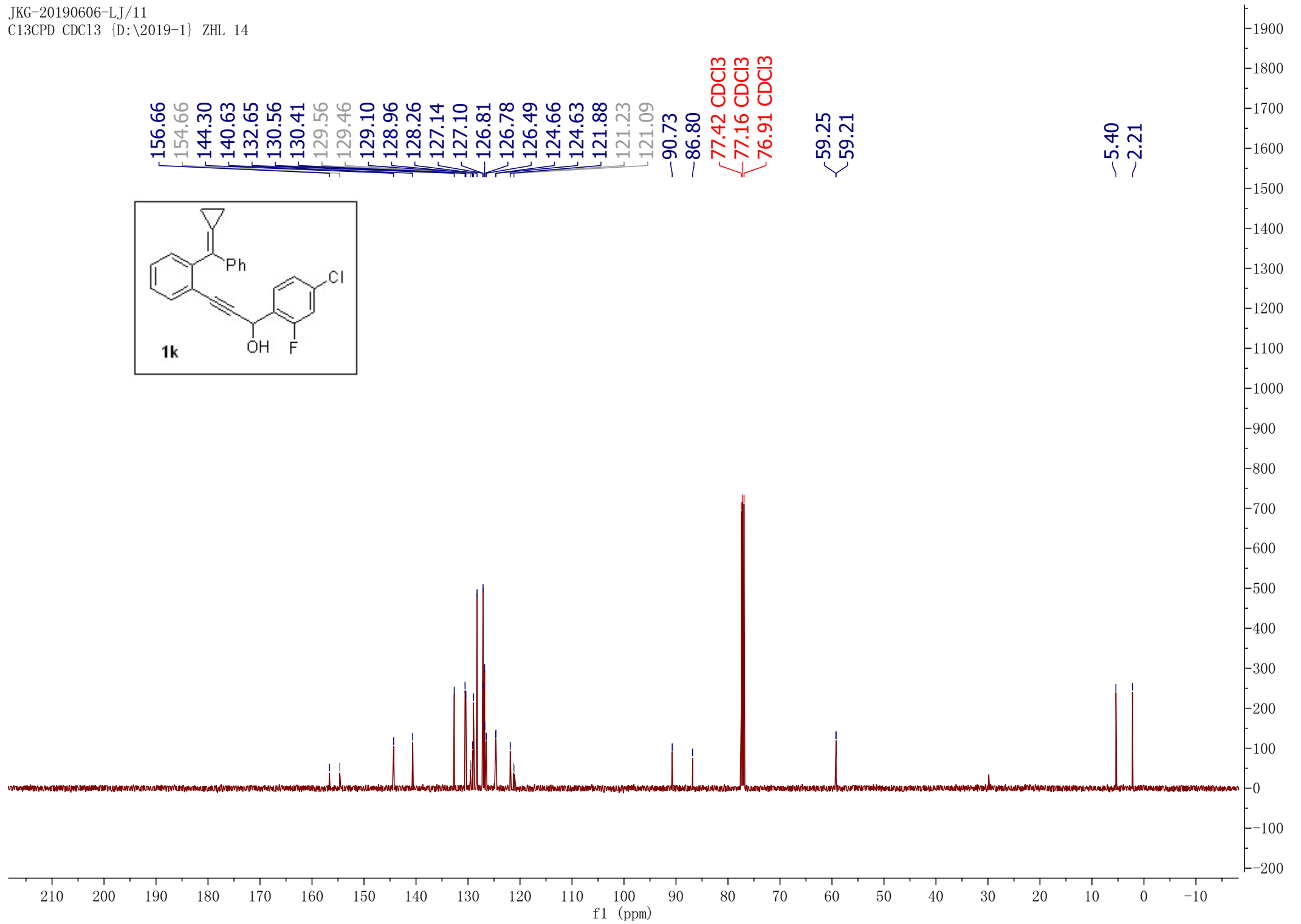
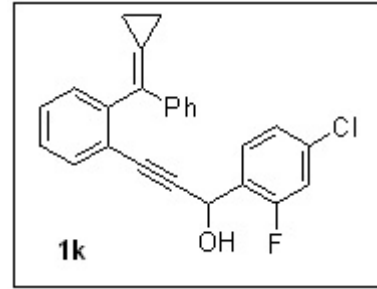


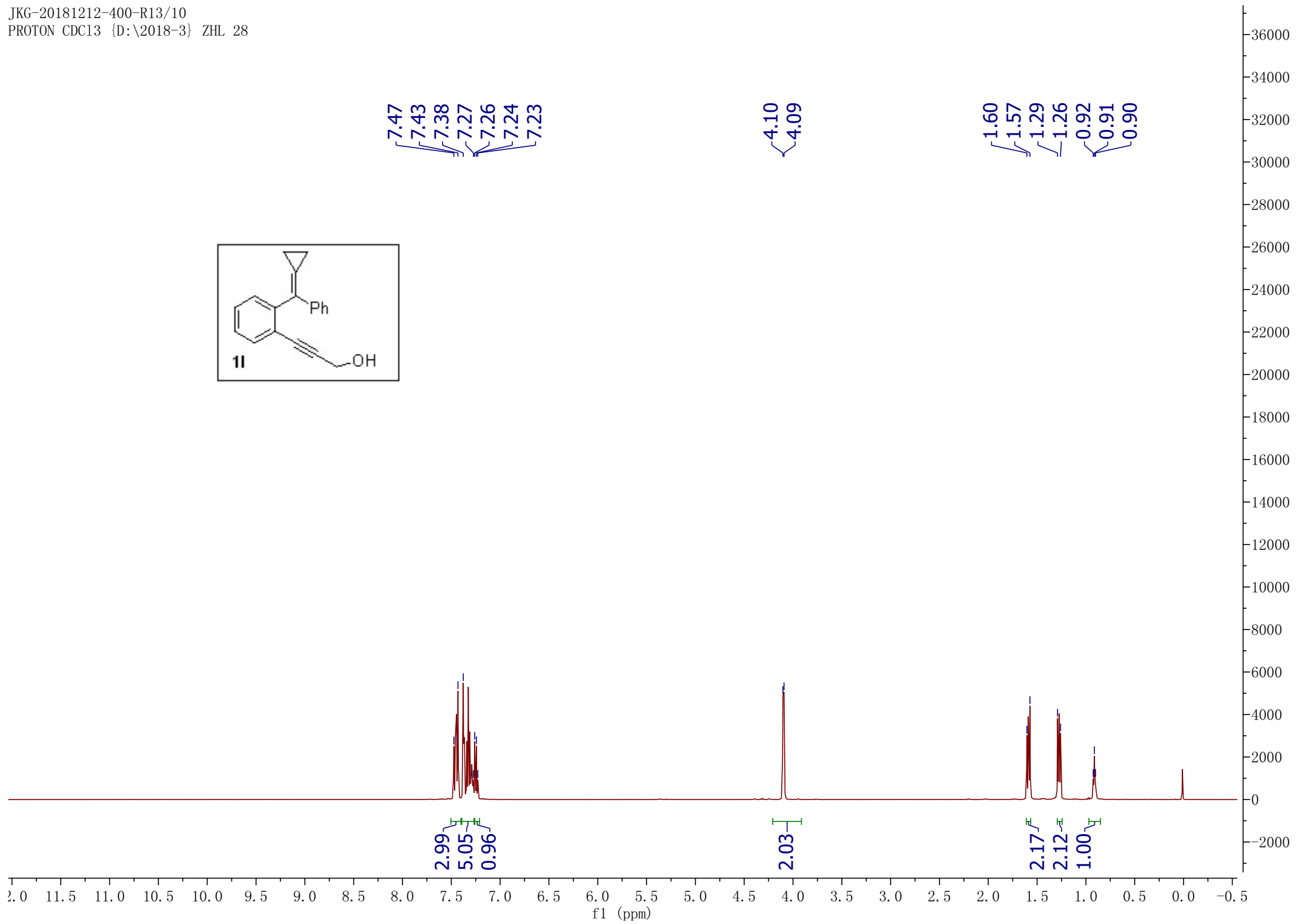
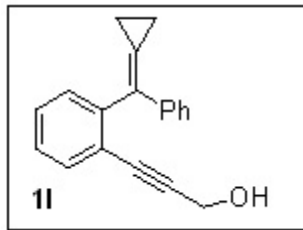


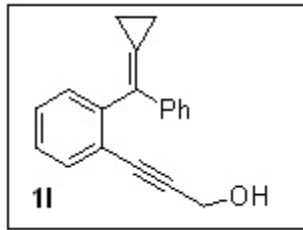
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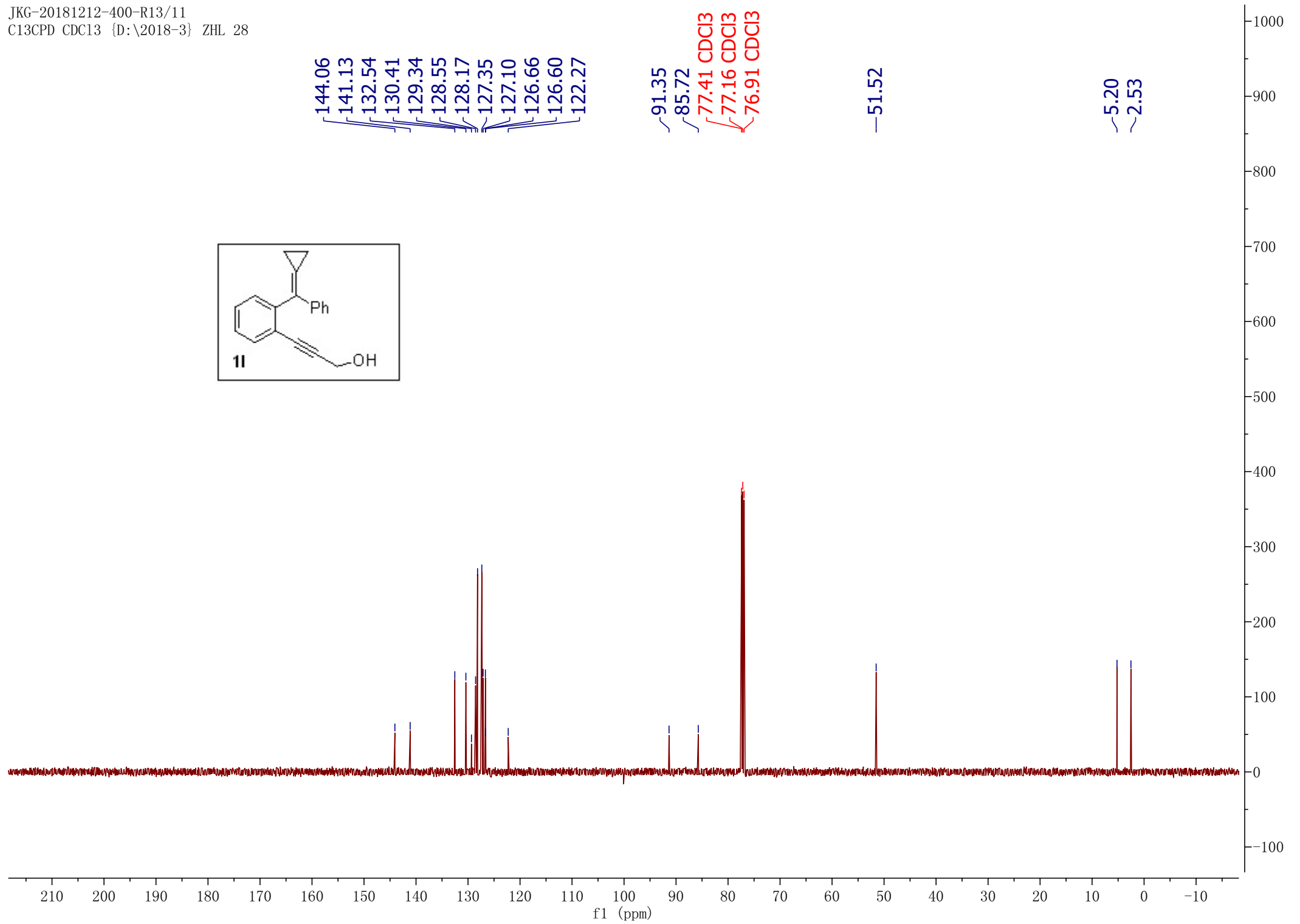
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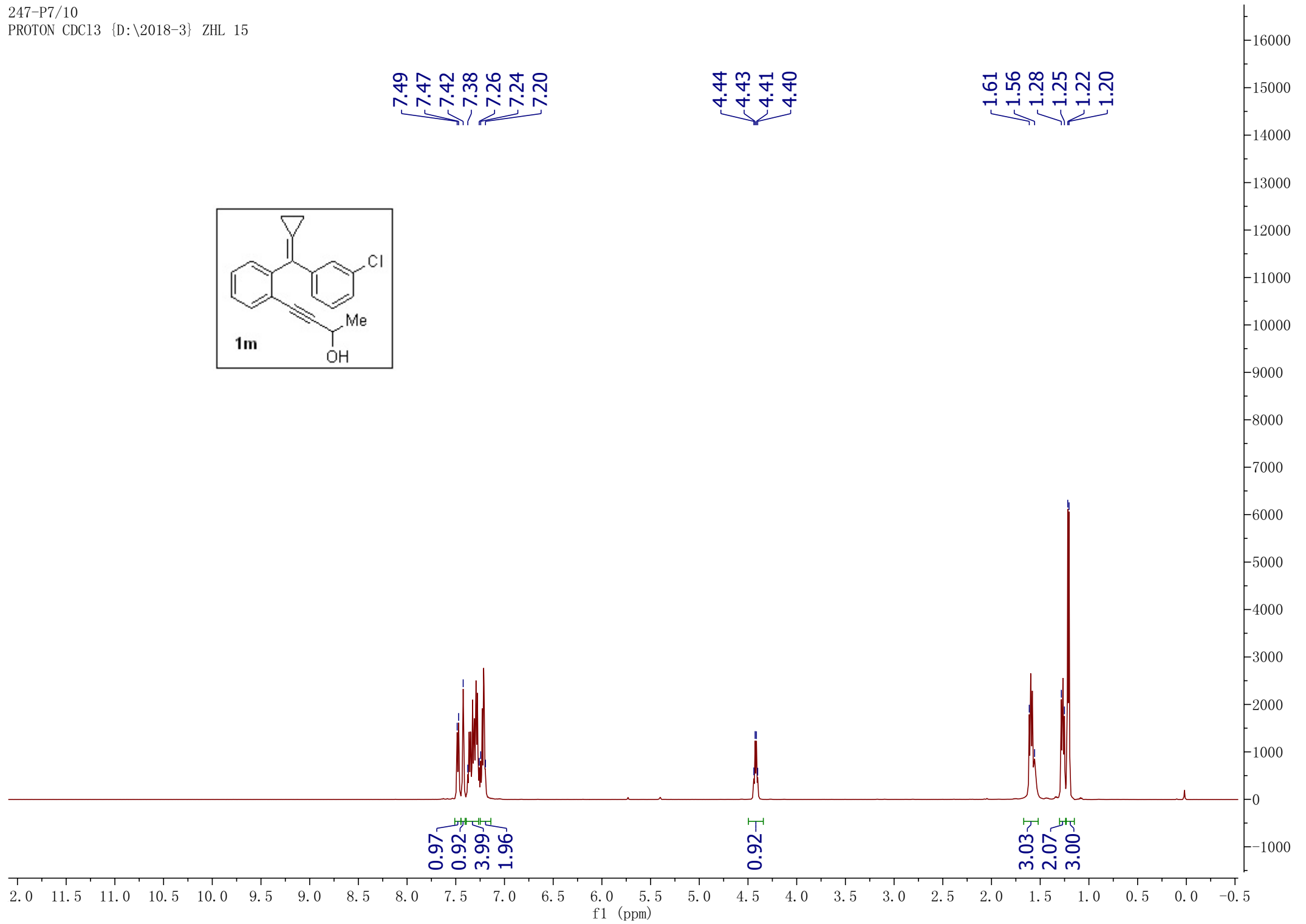
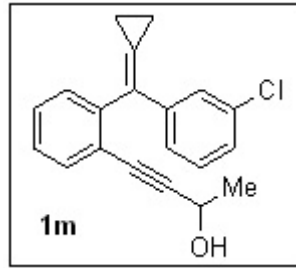
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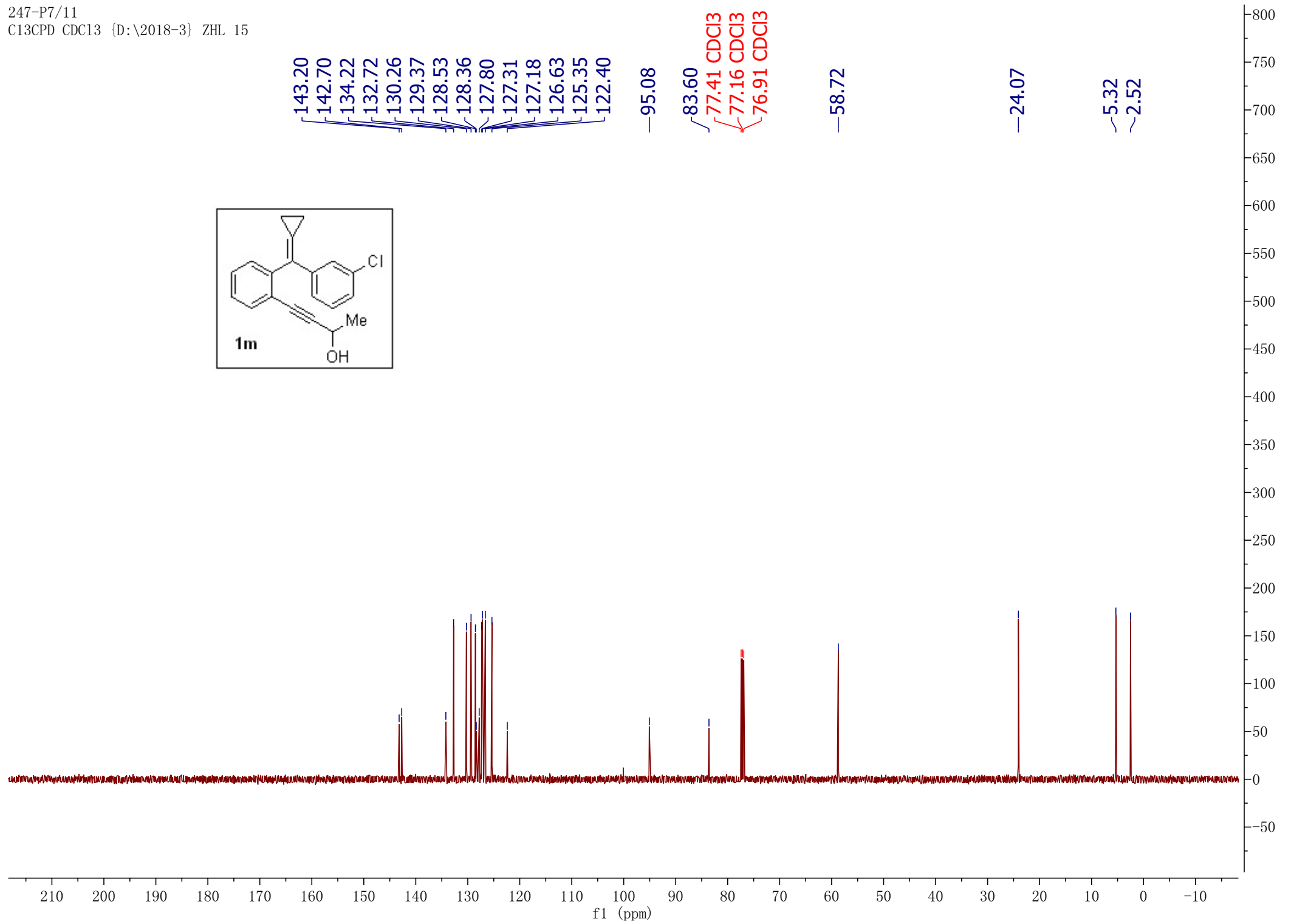
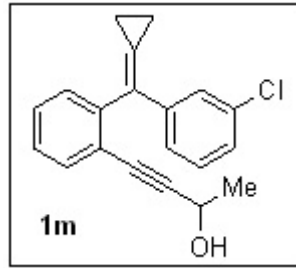
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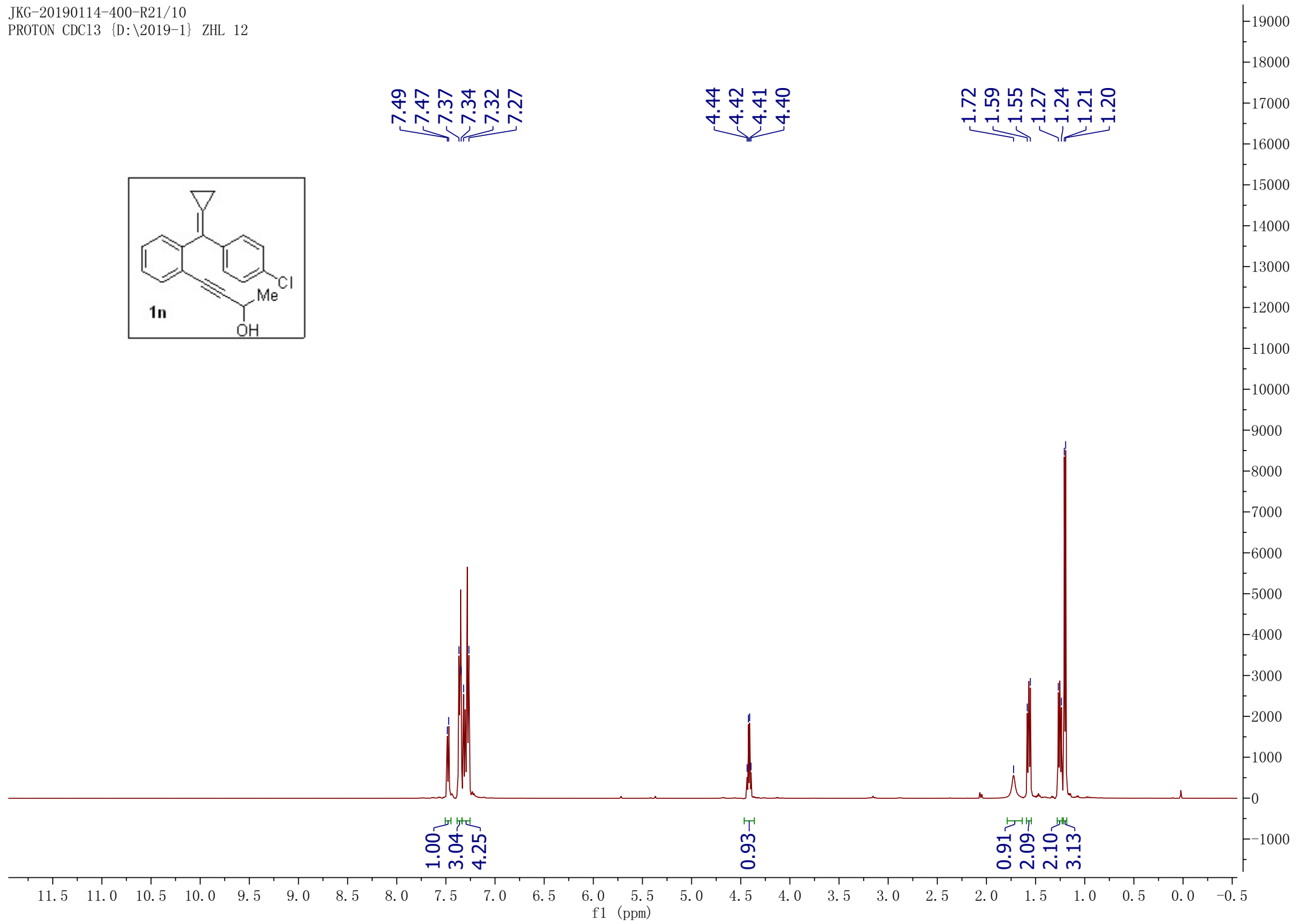
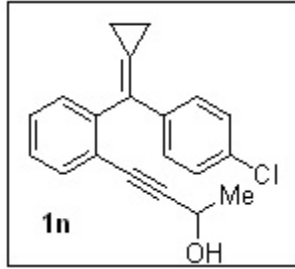
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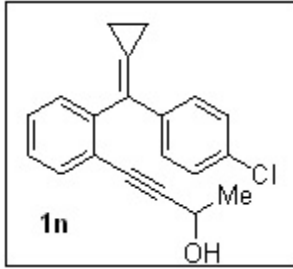
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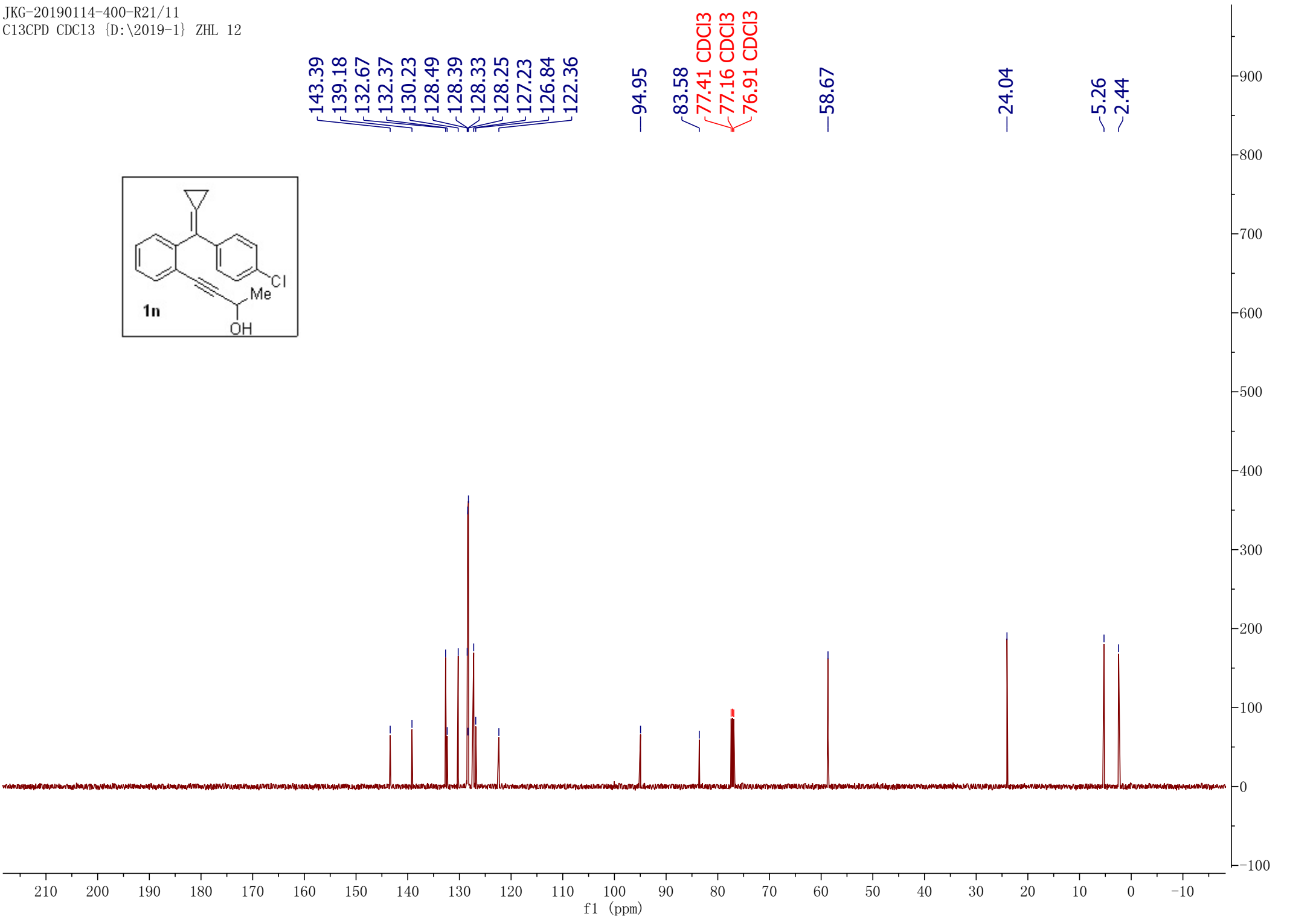
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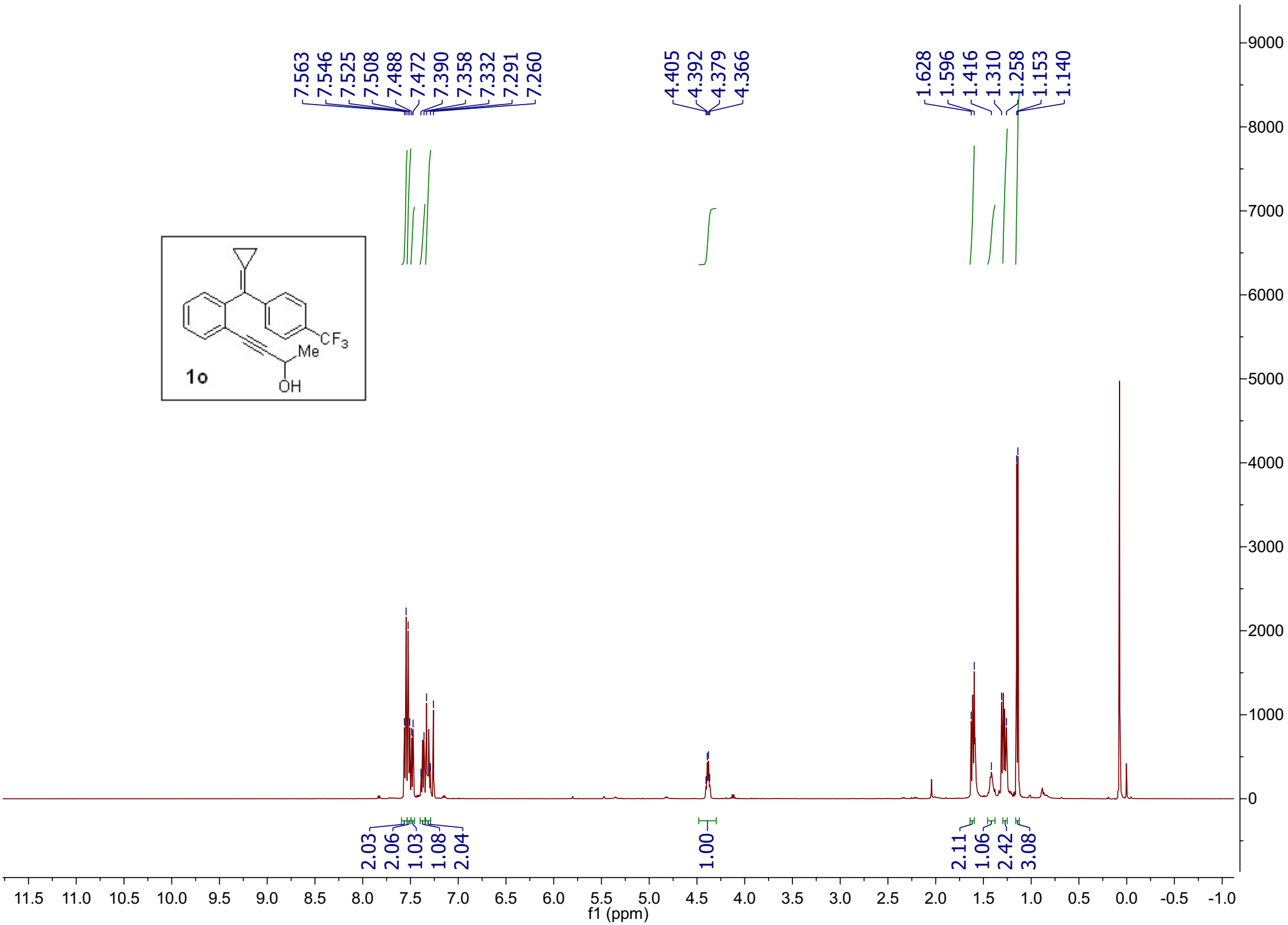
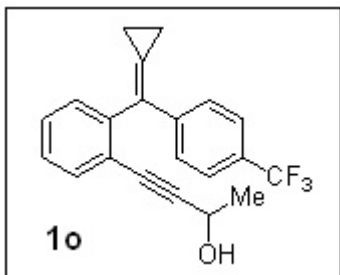
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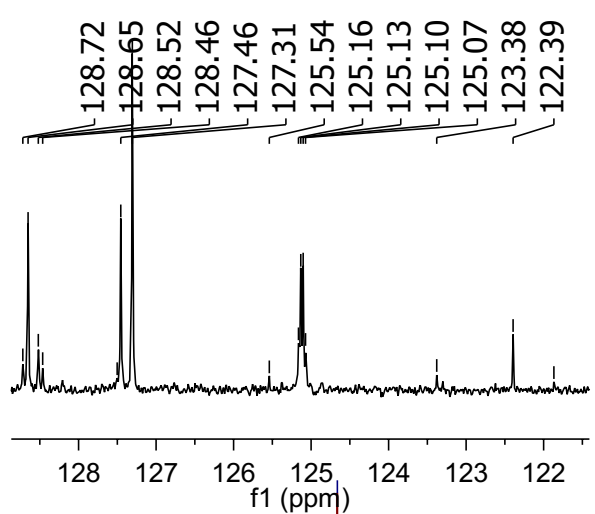
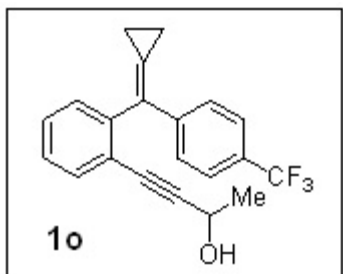
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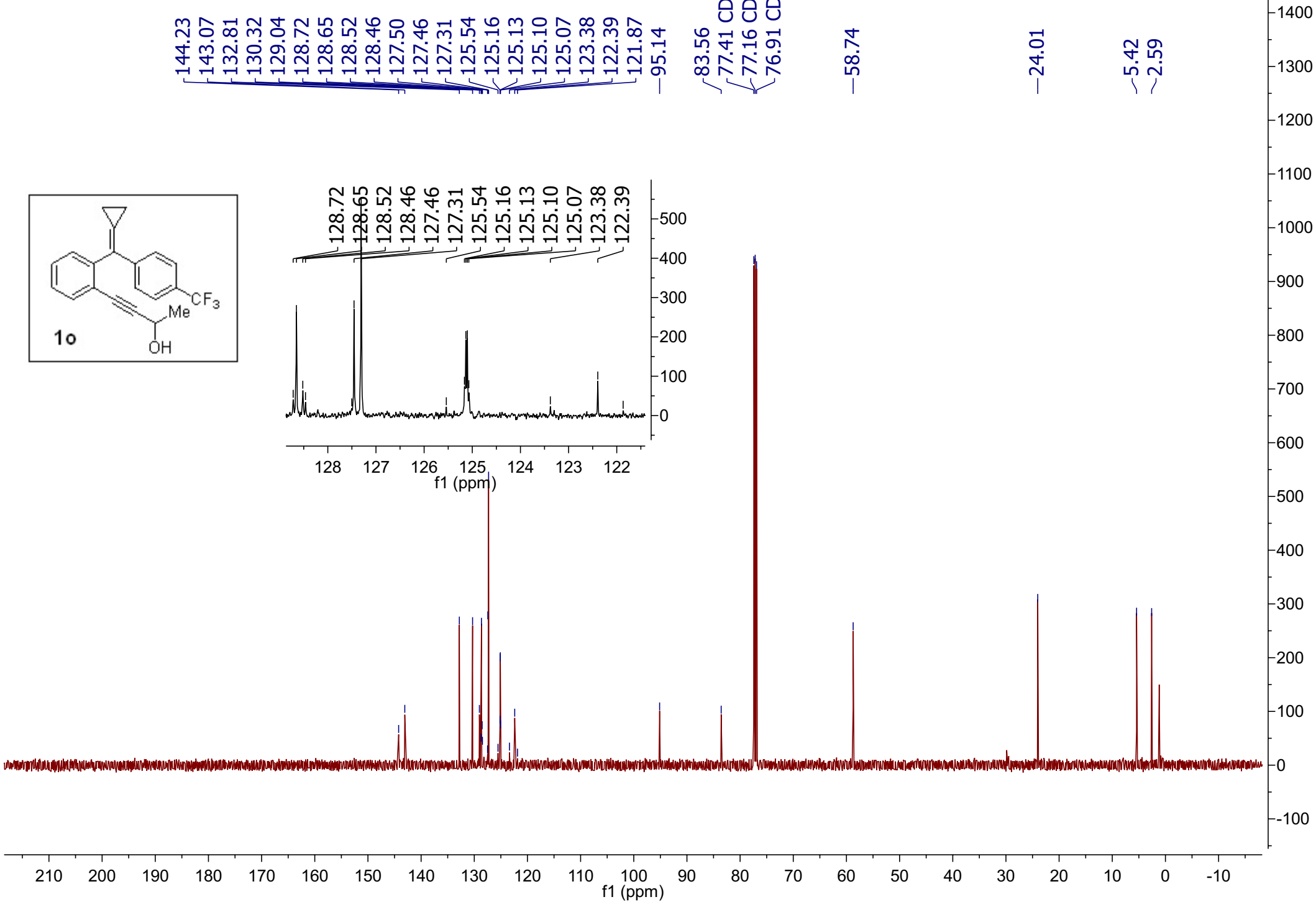
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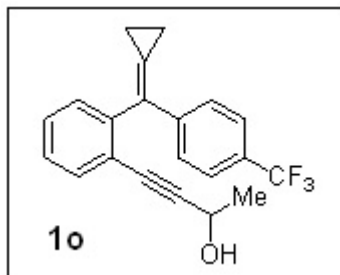
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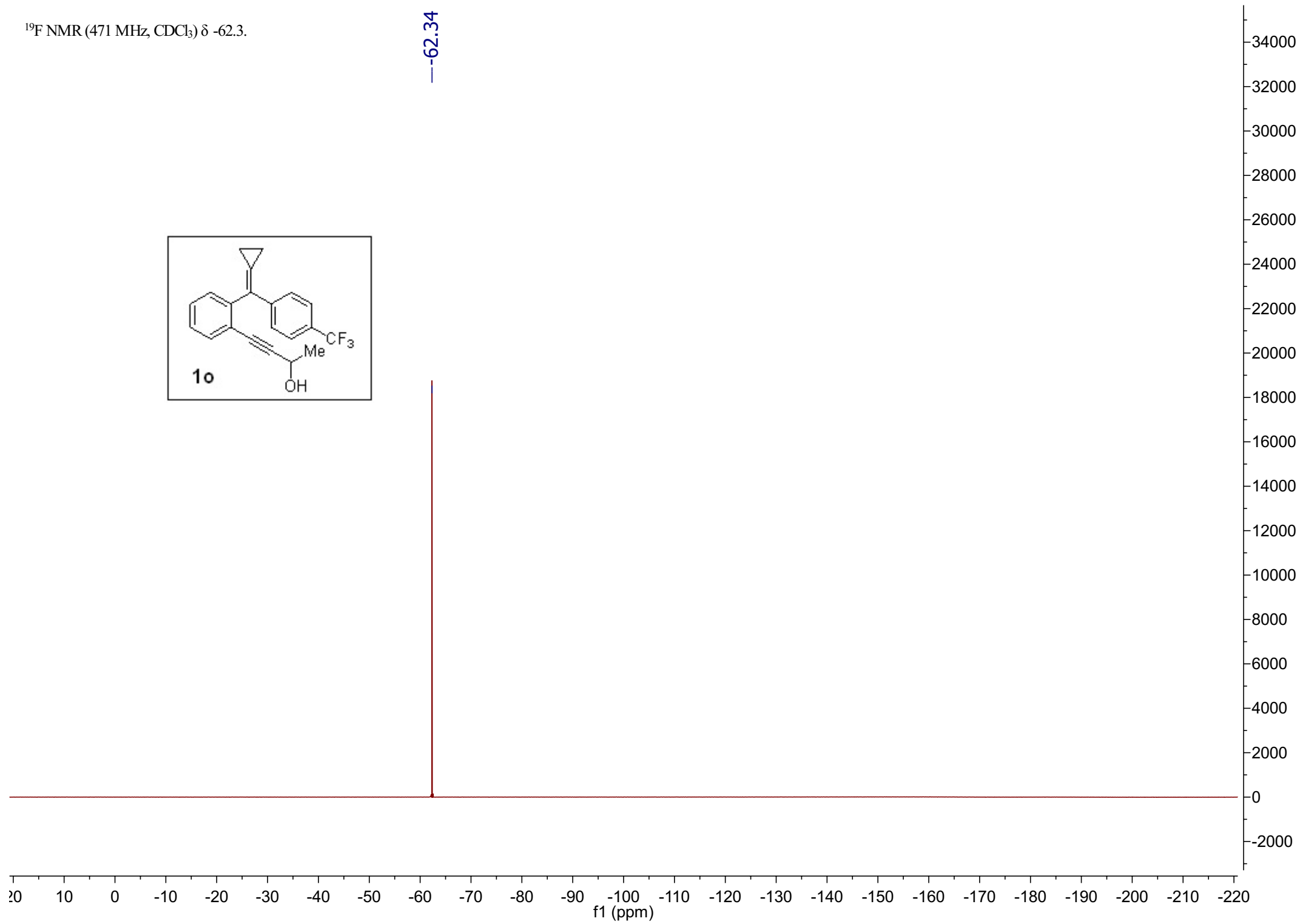
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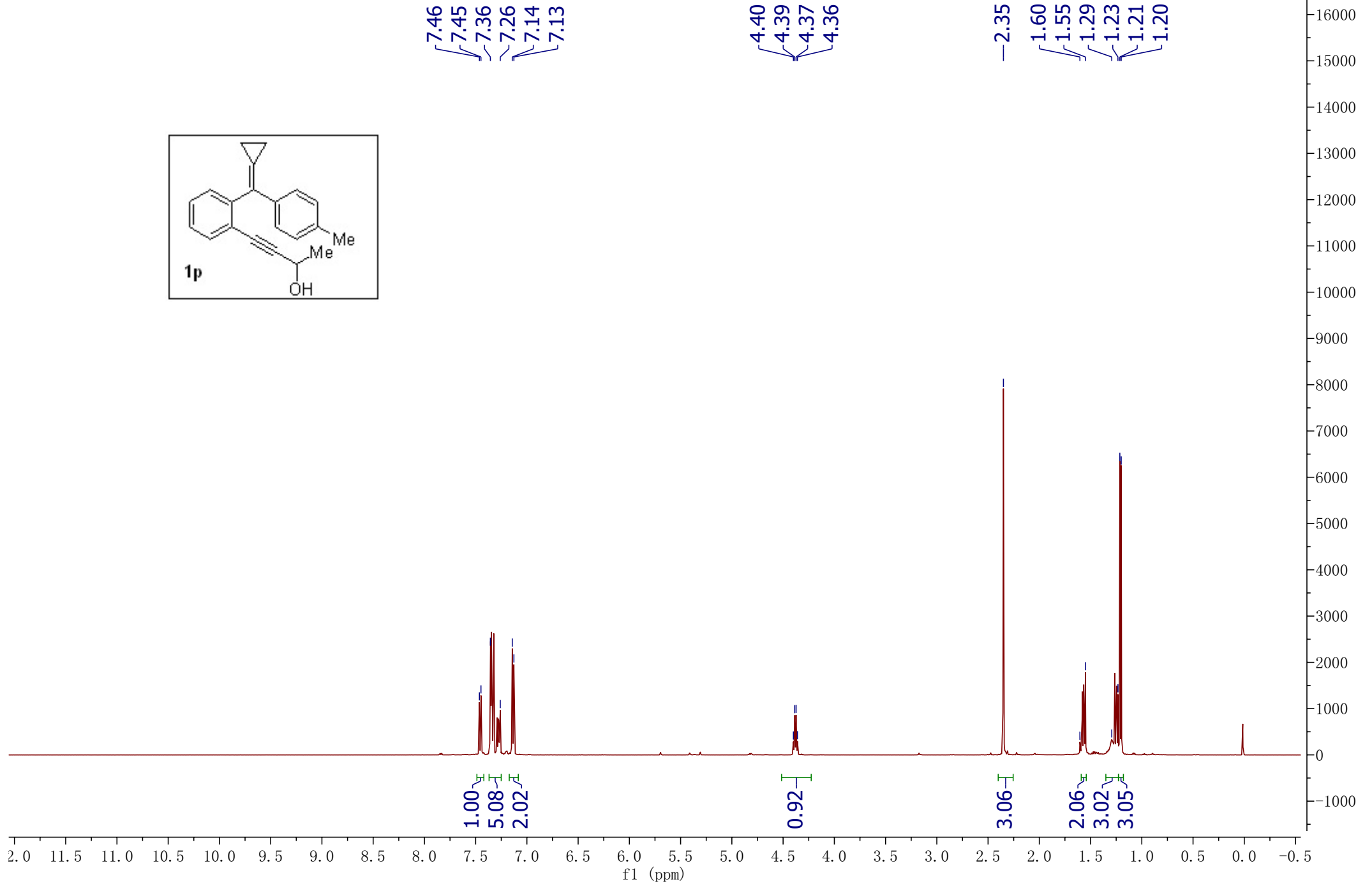
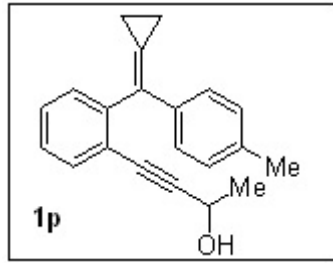


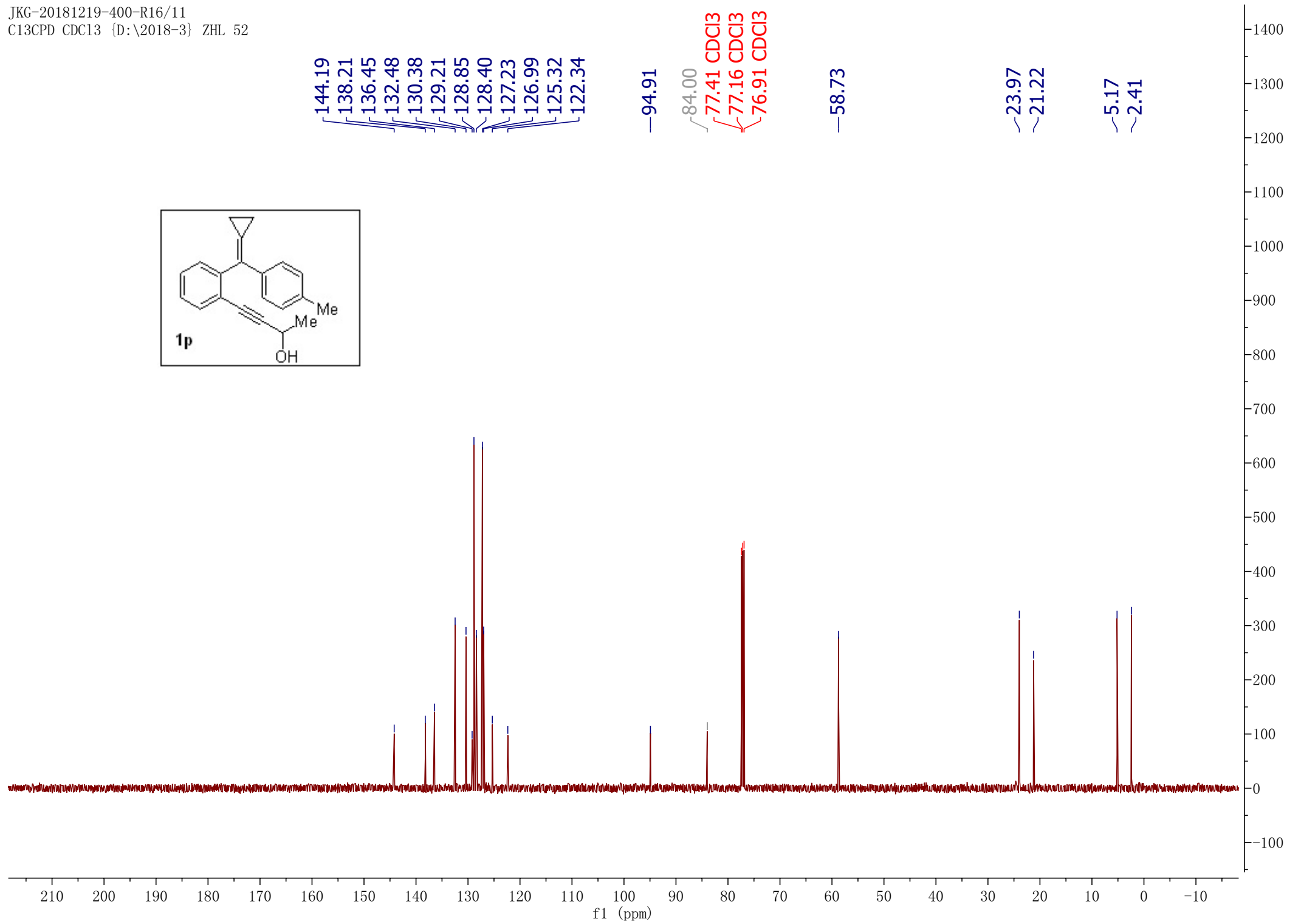
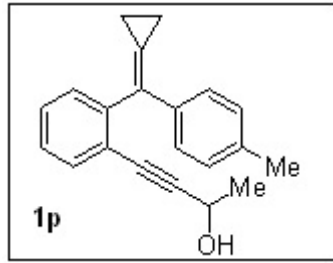
^{19}F NMR (471 MHz, CDCl_3) δ -62.3.

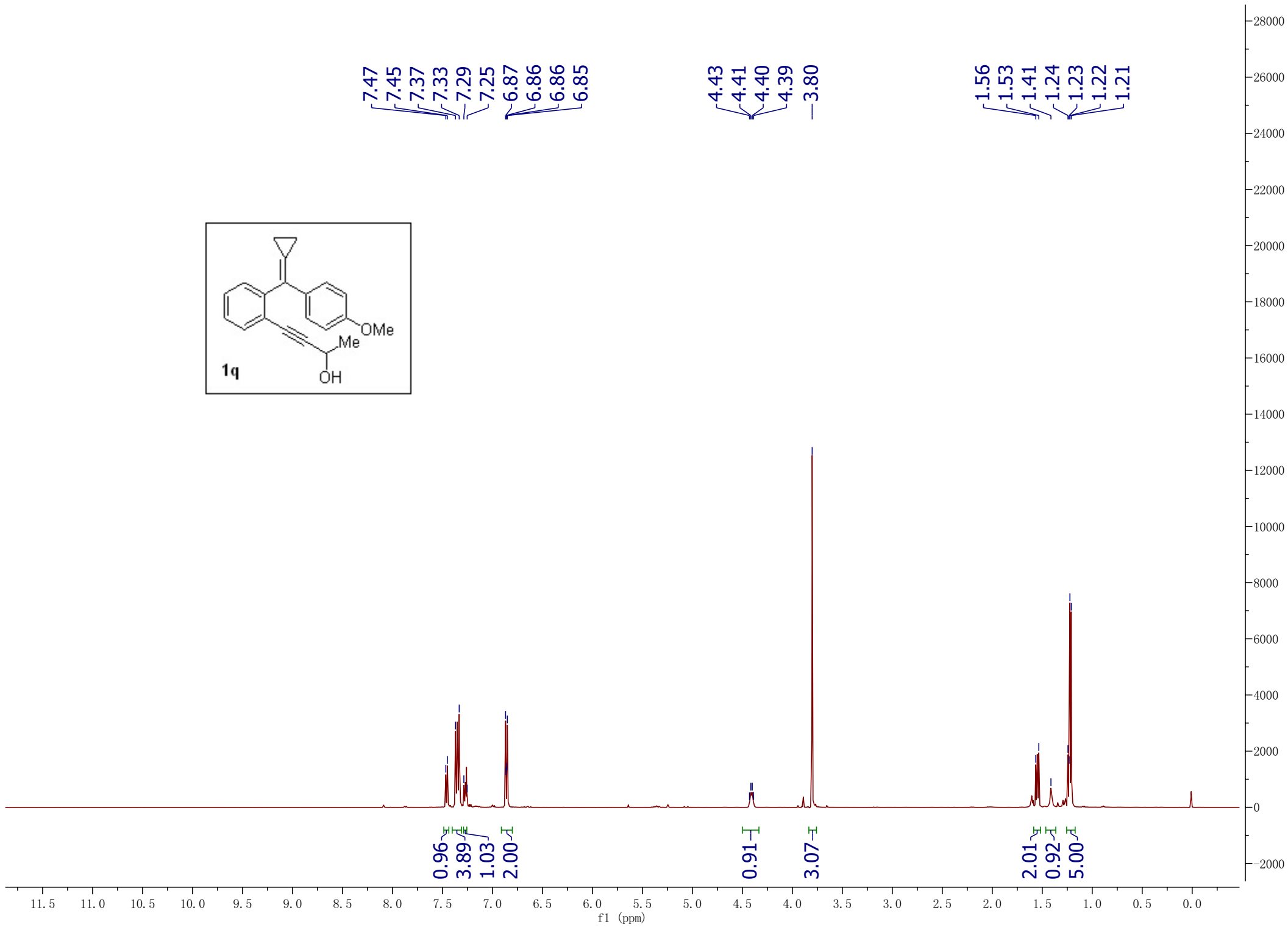
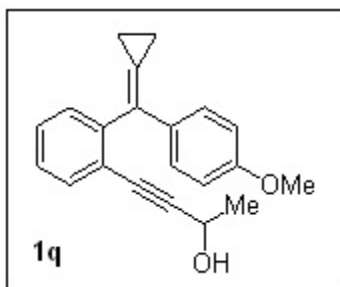


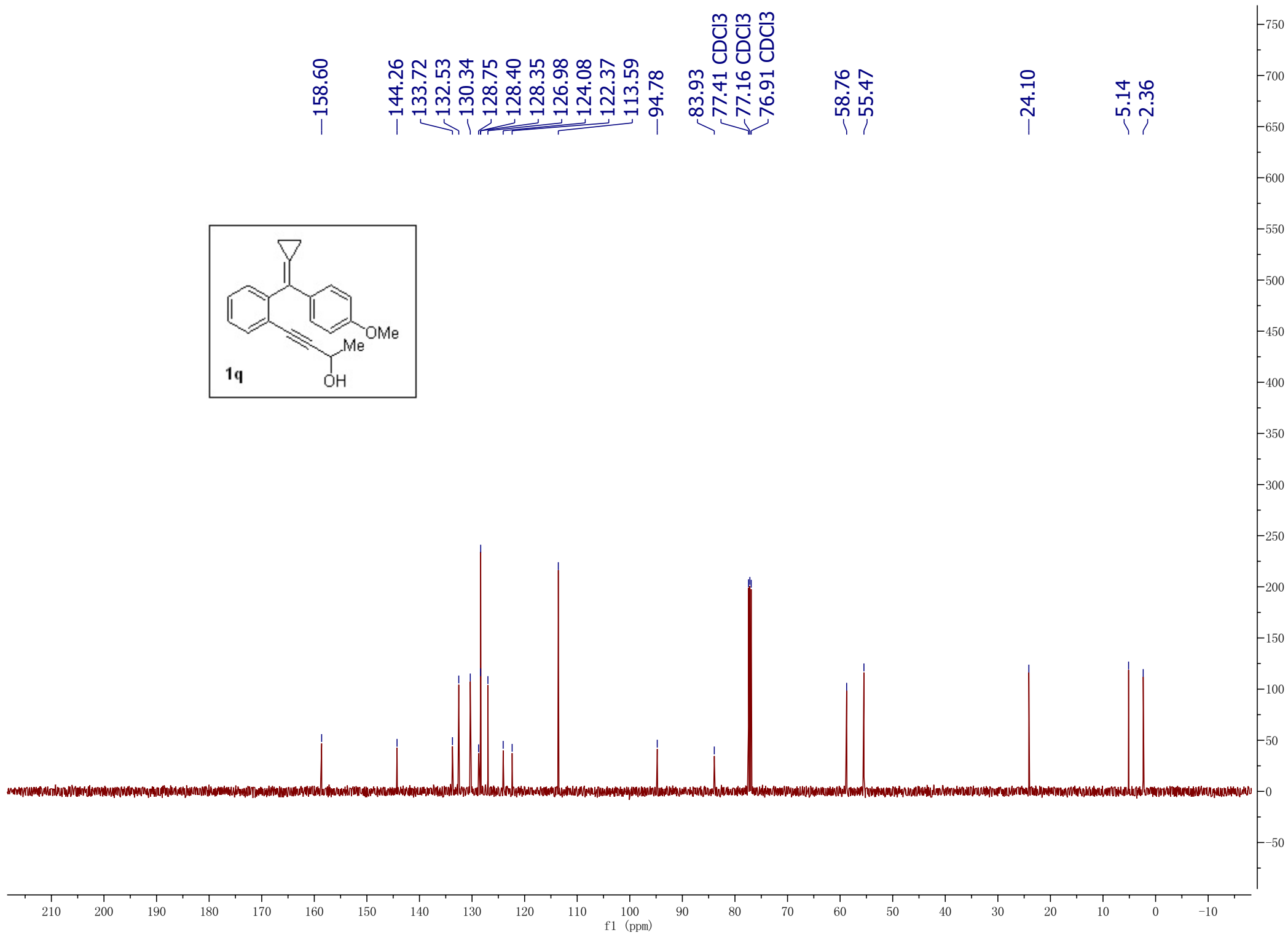
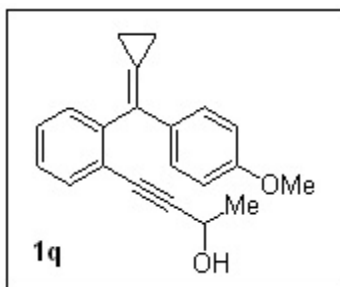
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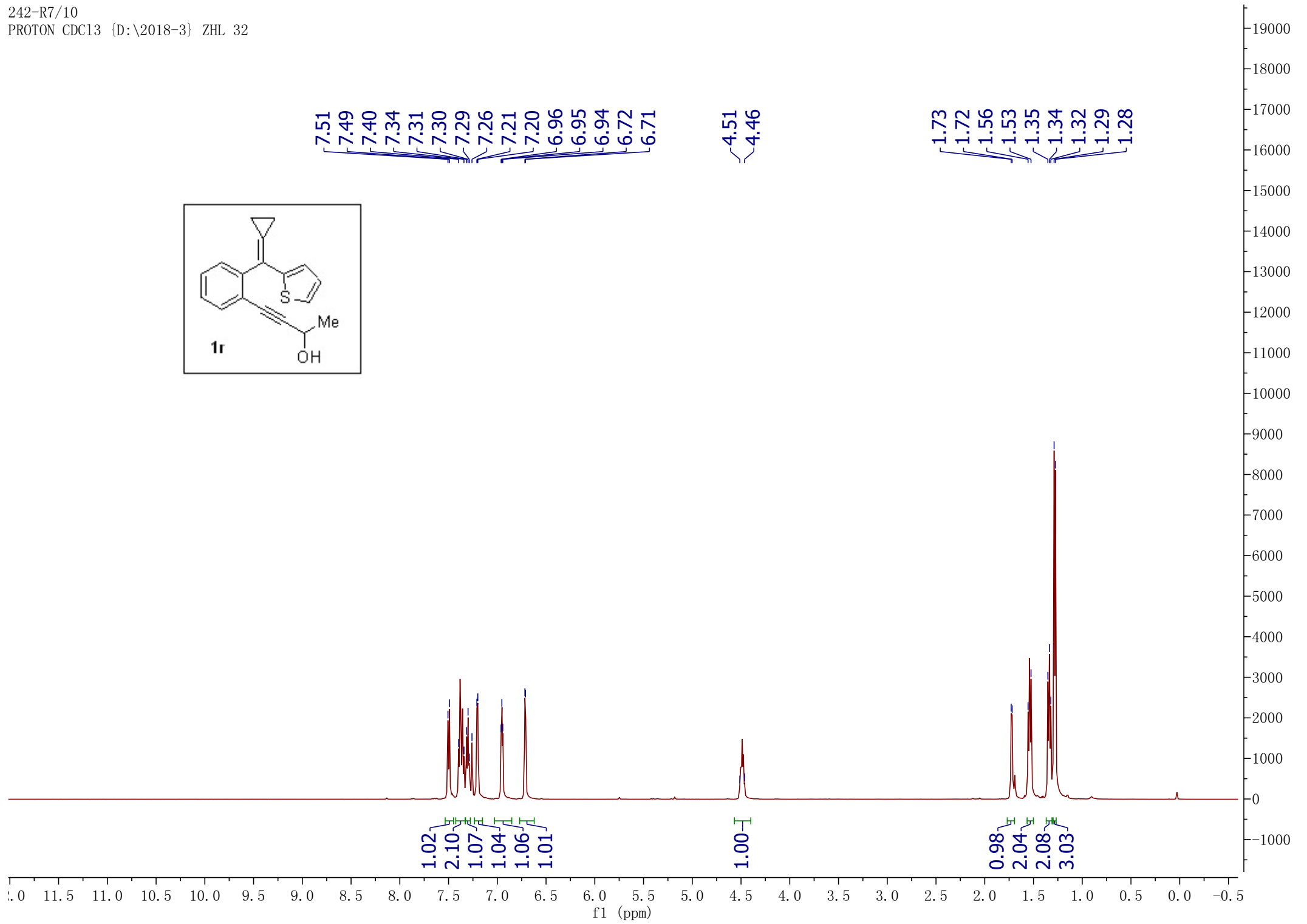
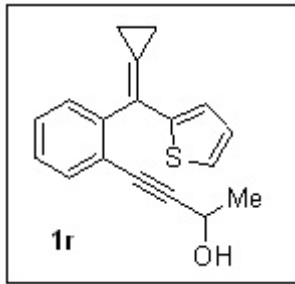


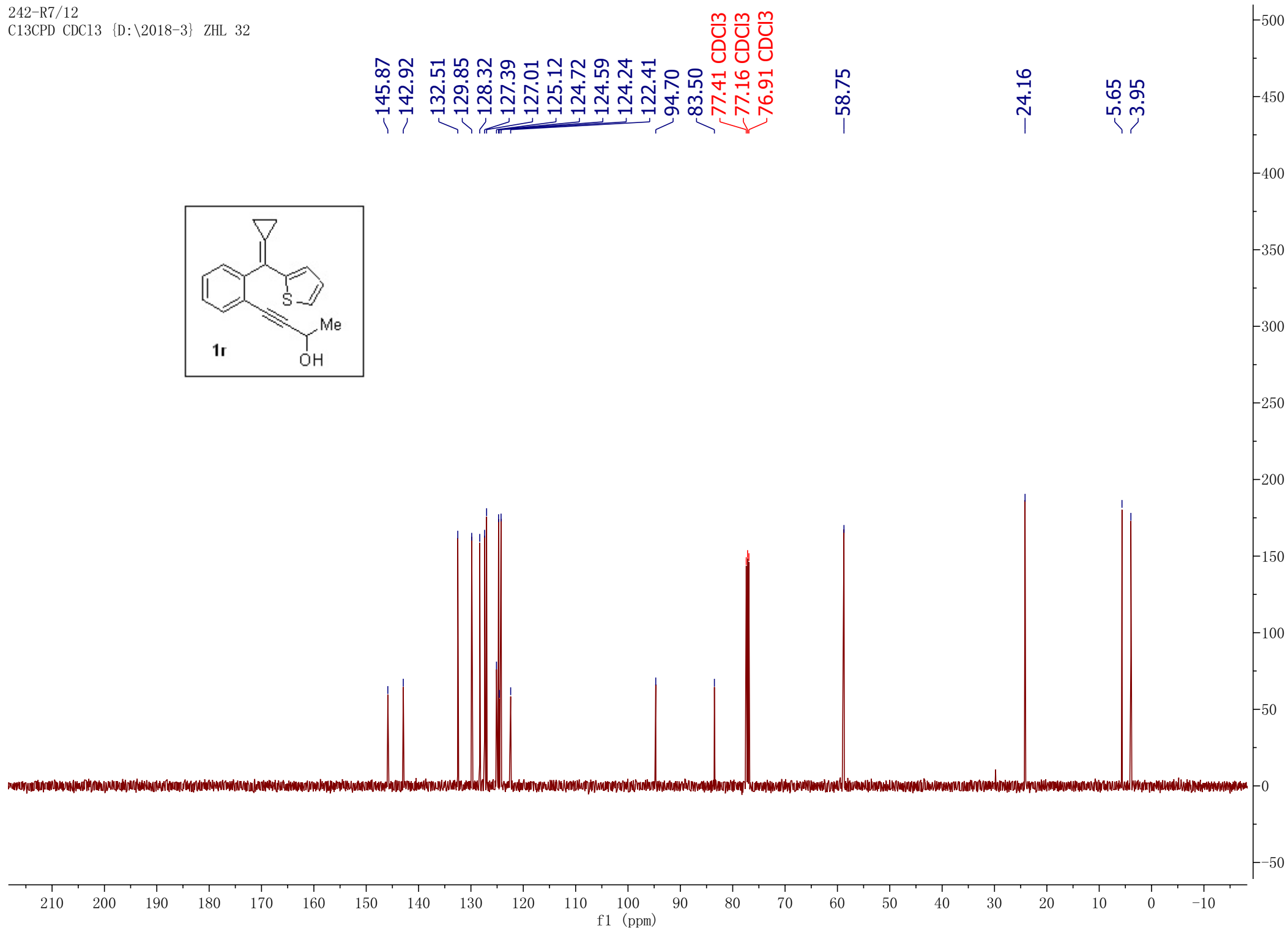
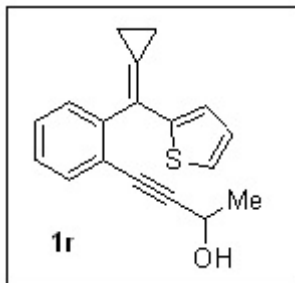


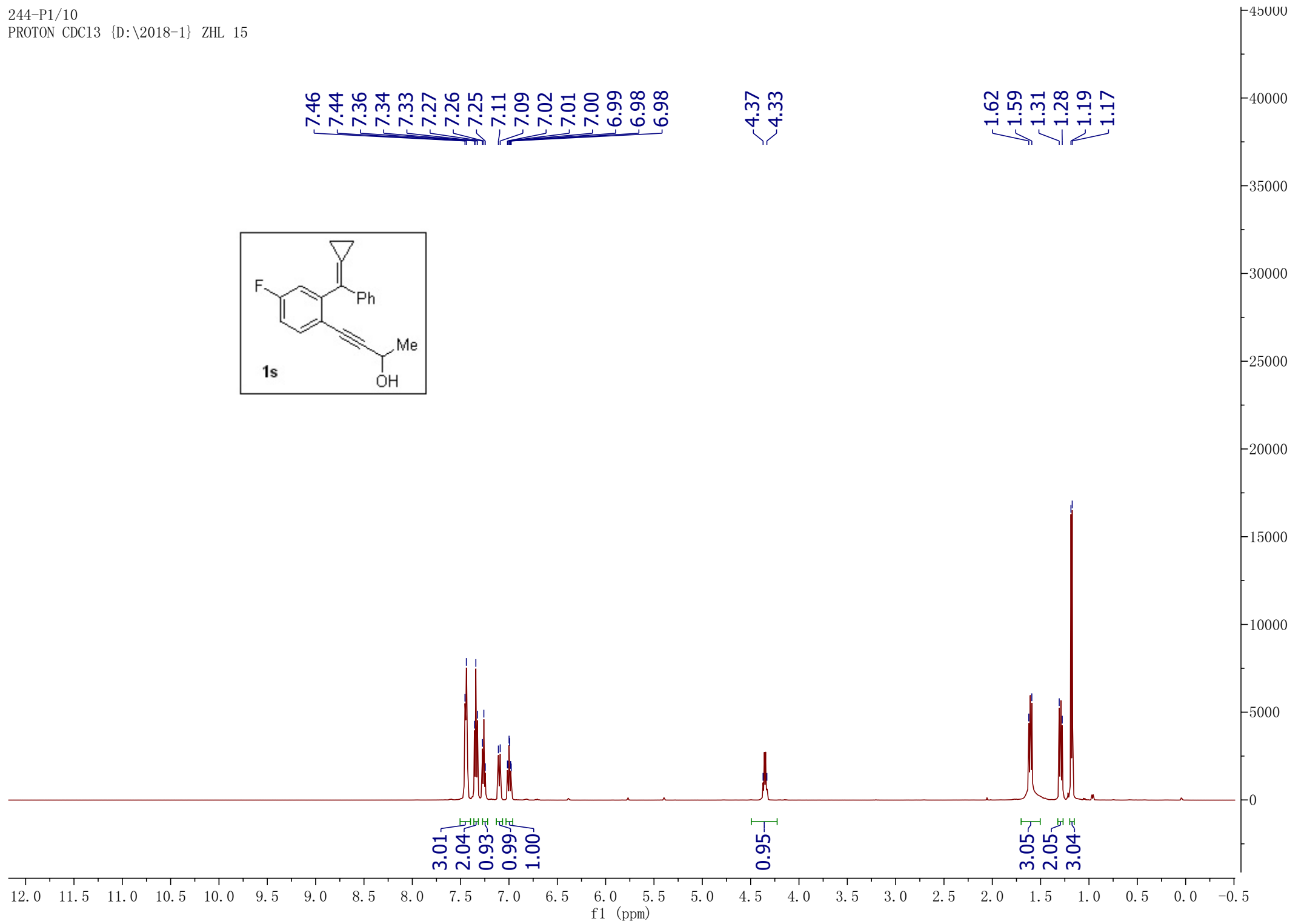
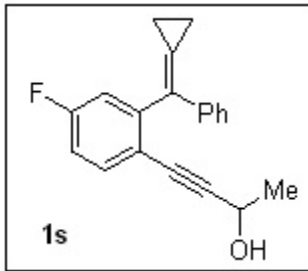












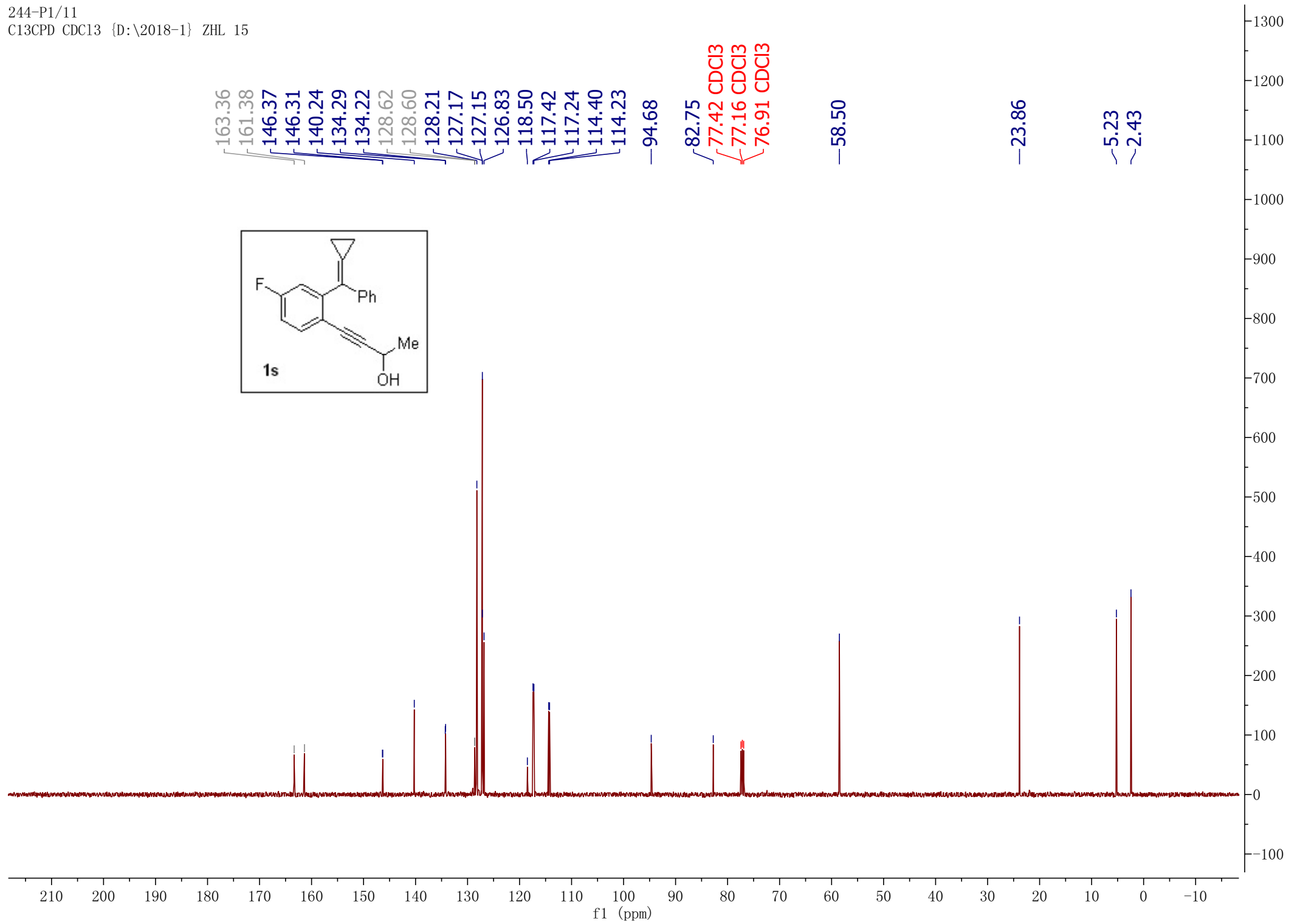
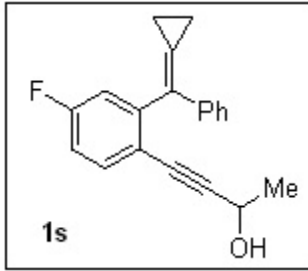
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114.23

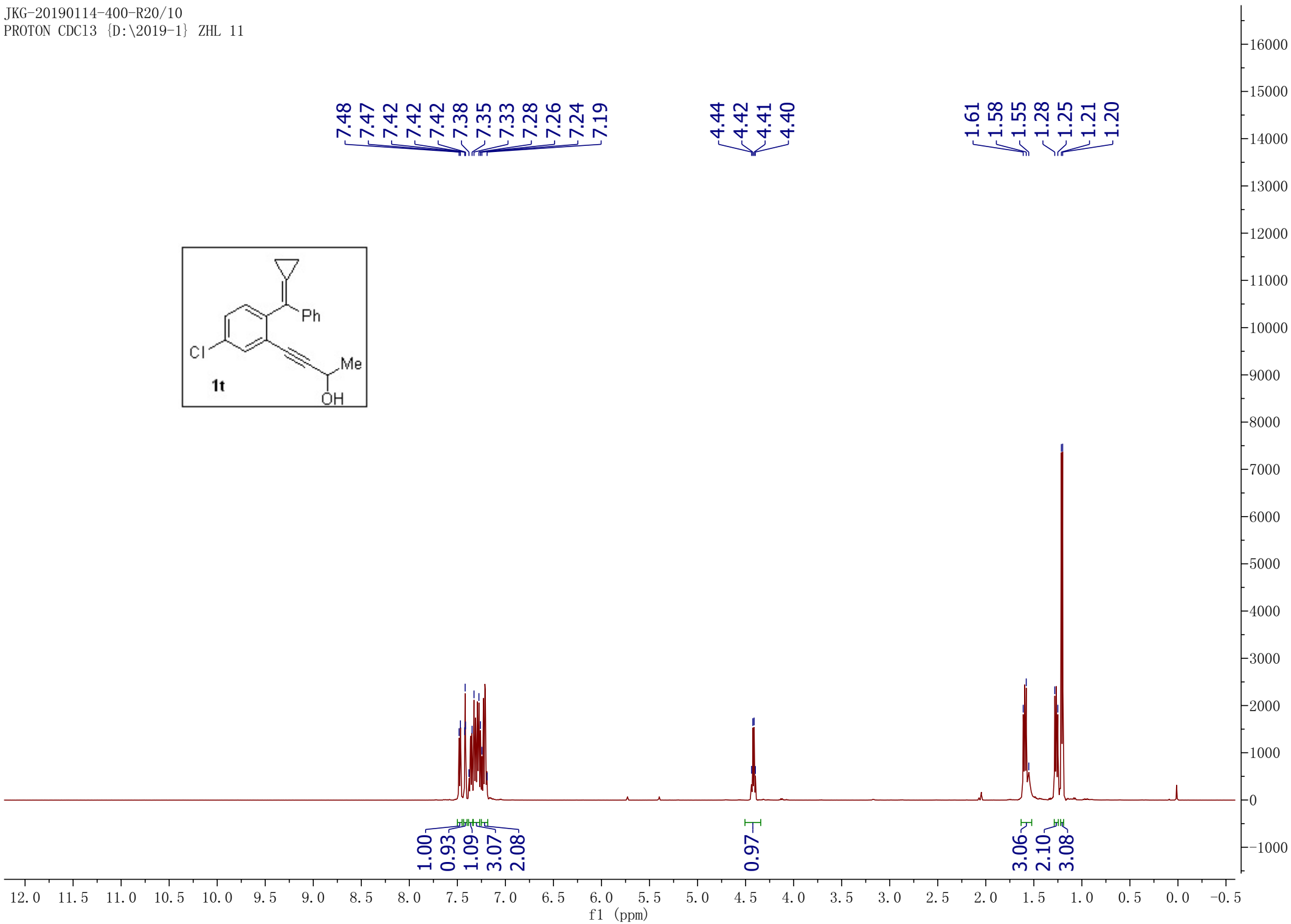
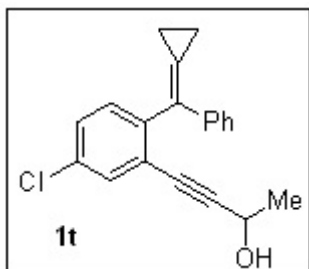
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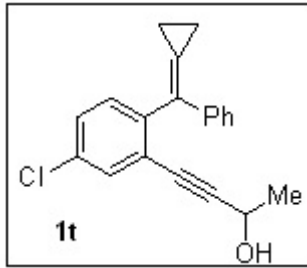
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23.86

5.23
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125.35
122.36

—95.07

83.60

77.41 CDC13

77.16 CDC13

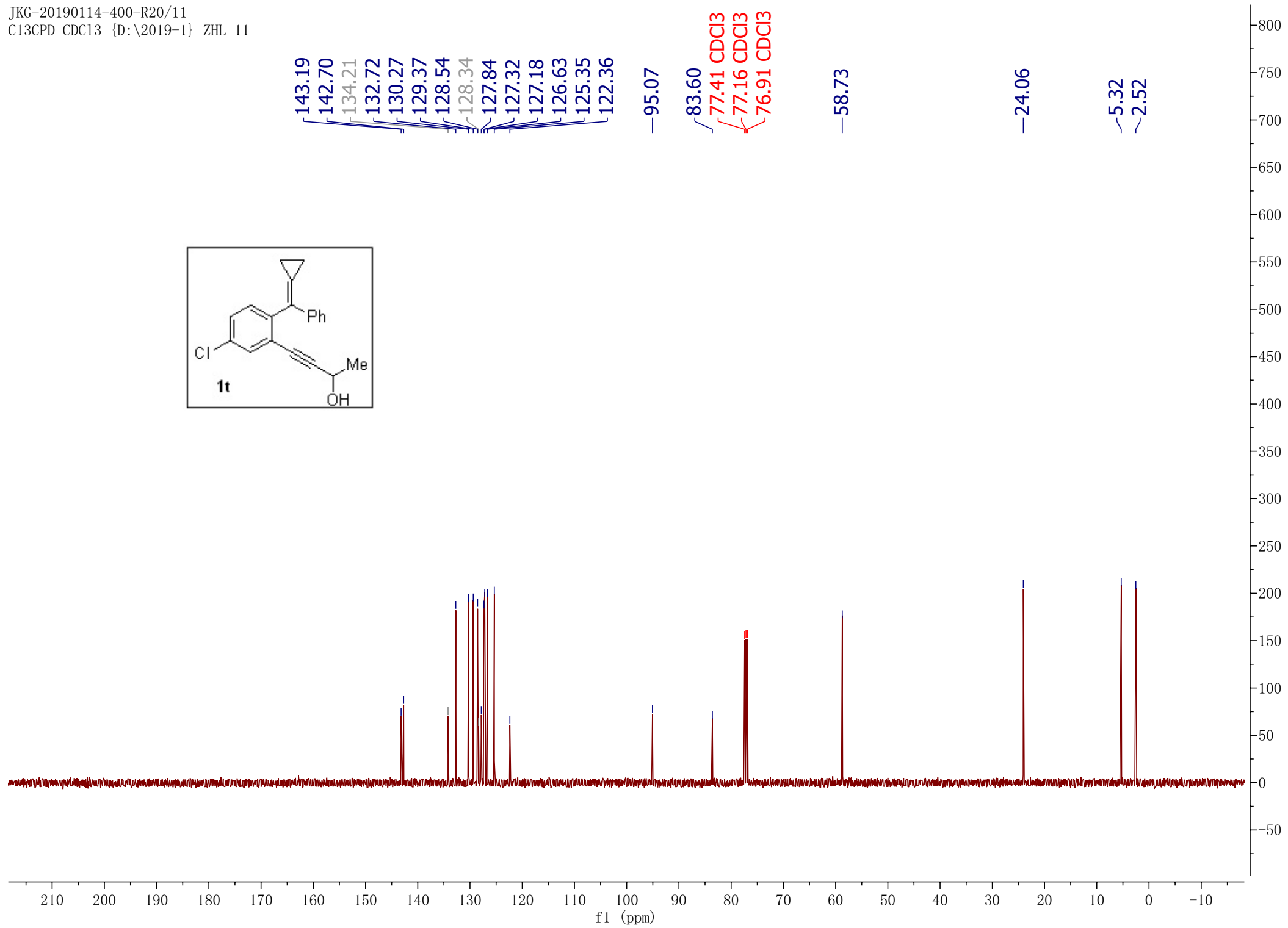
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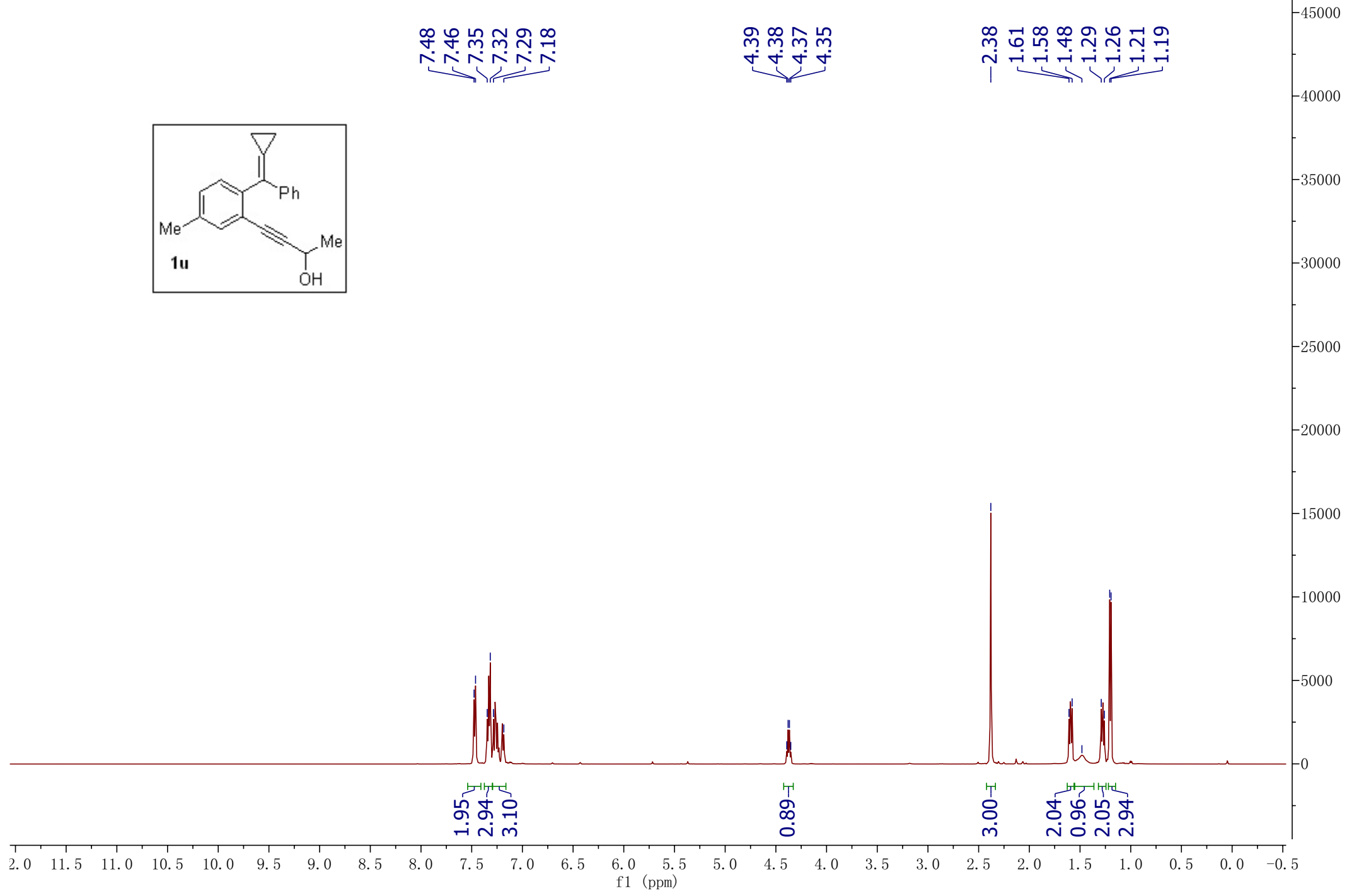
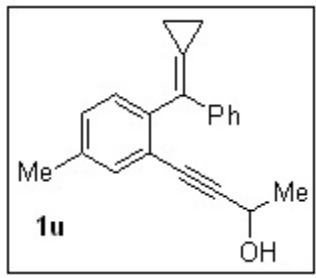
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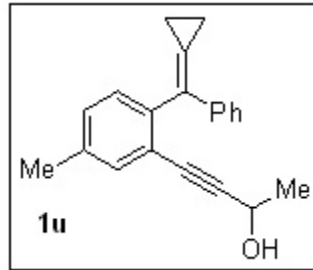
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~5.32

~2.52







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83.98

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76.91 CDC13

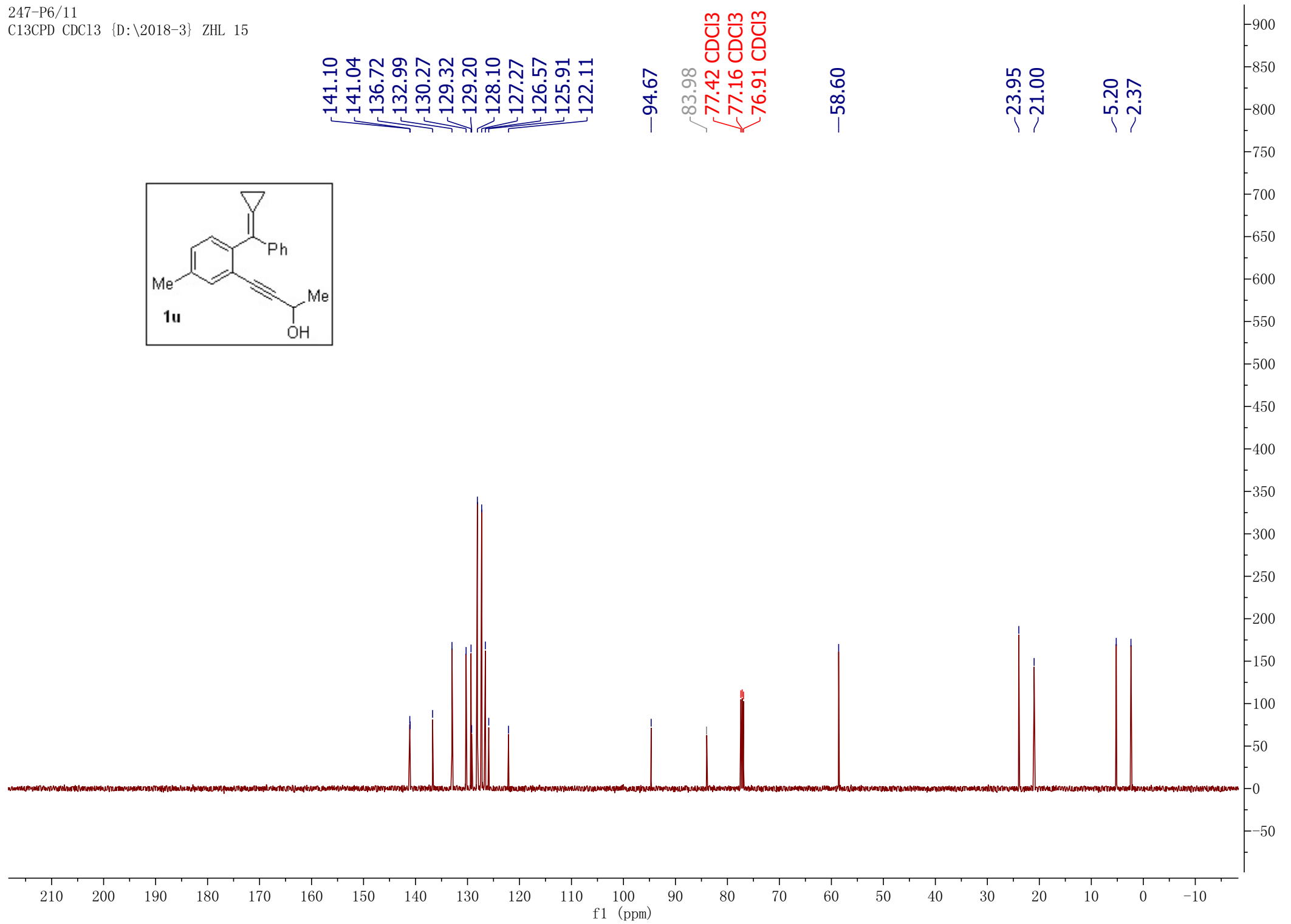
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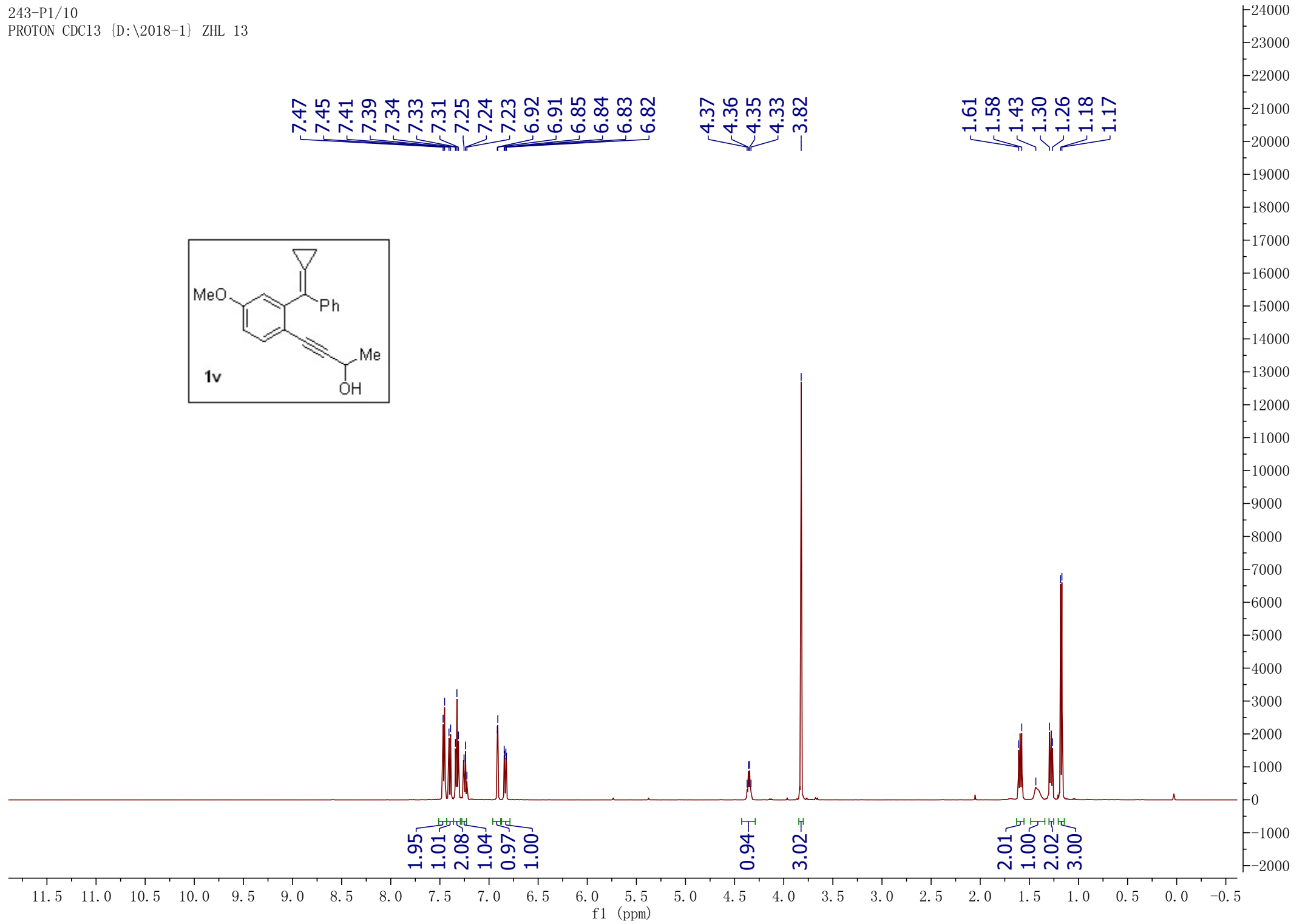
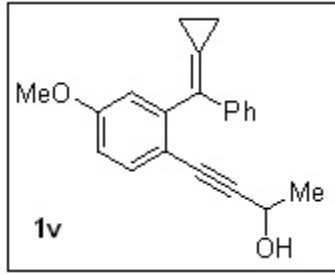
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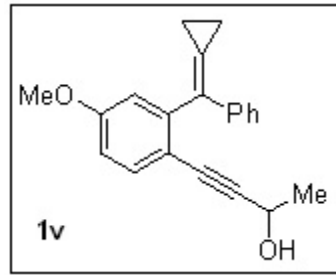
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93.51

83.69

77.41 CDC13

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76.91 CDC13

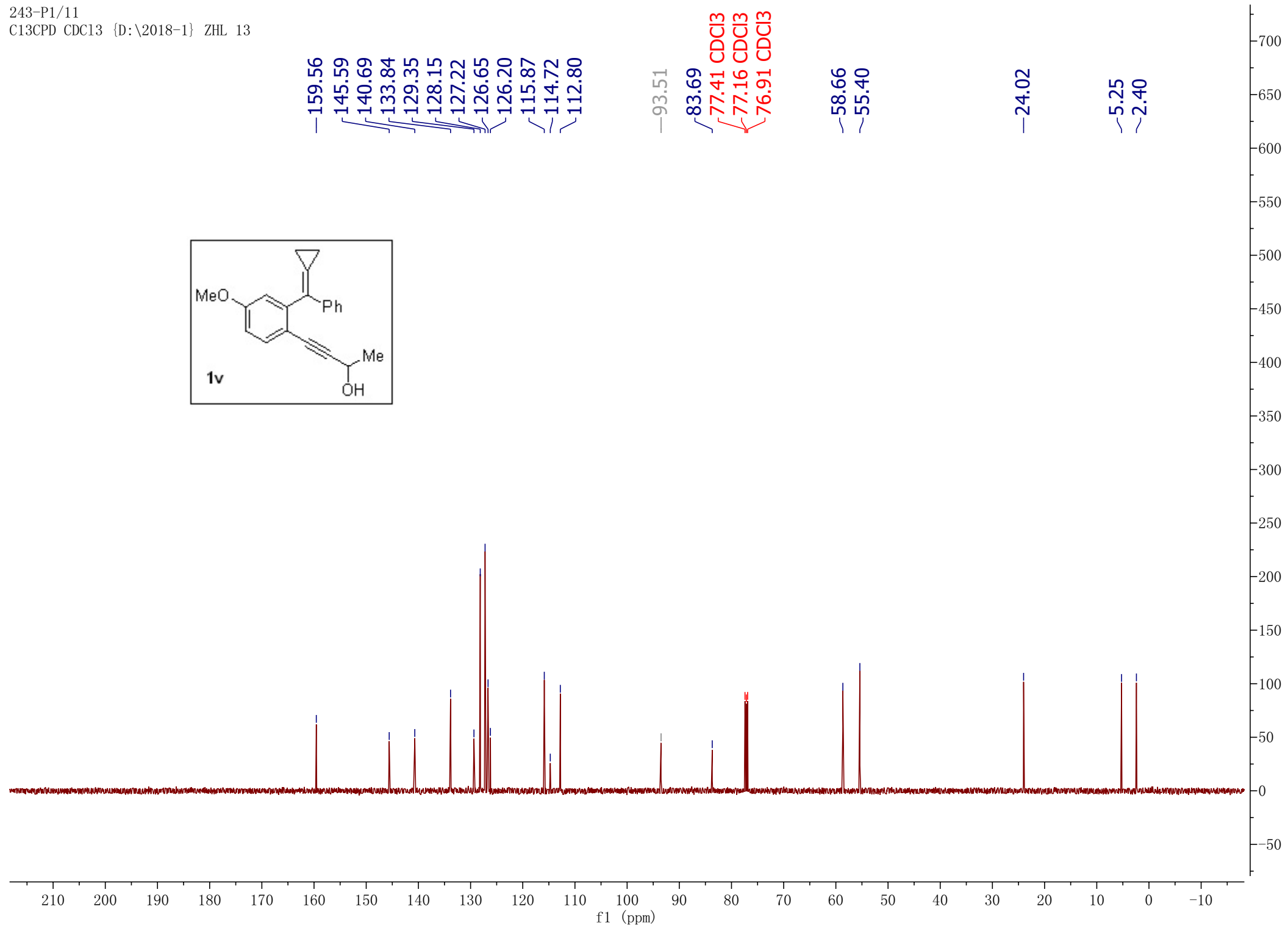
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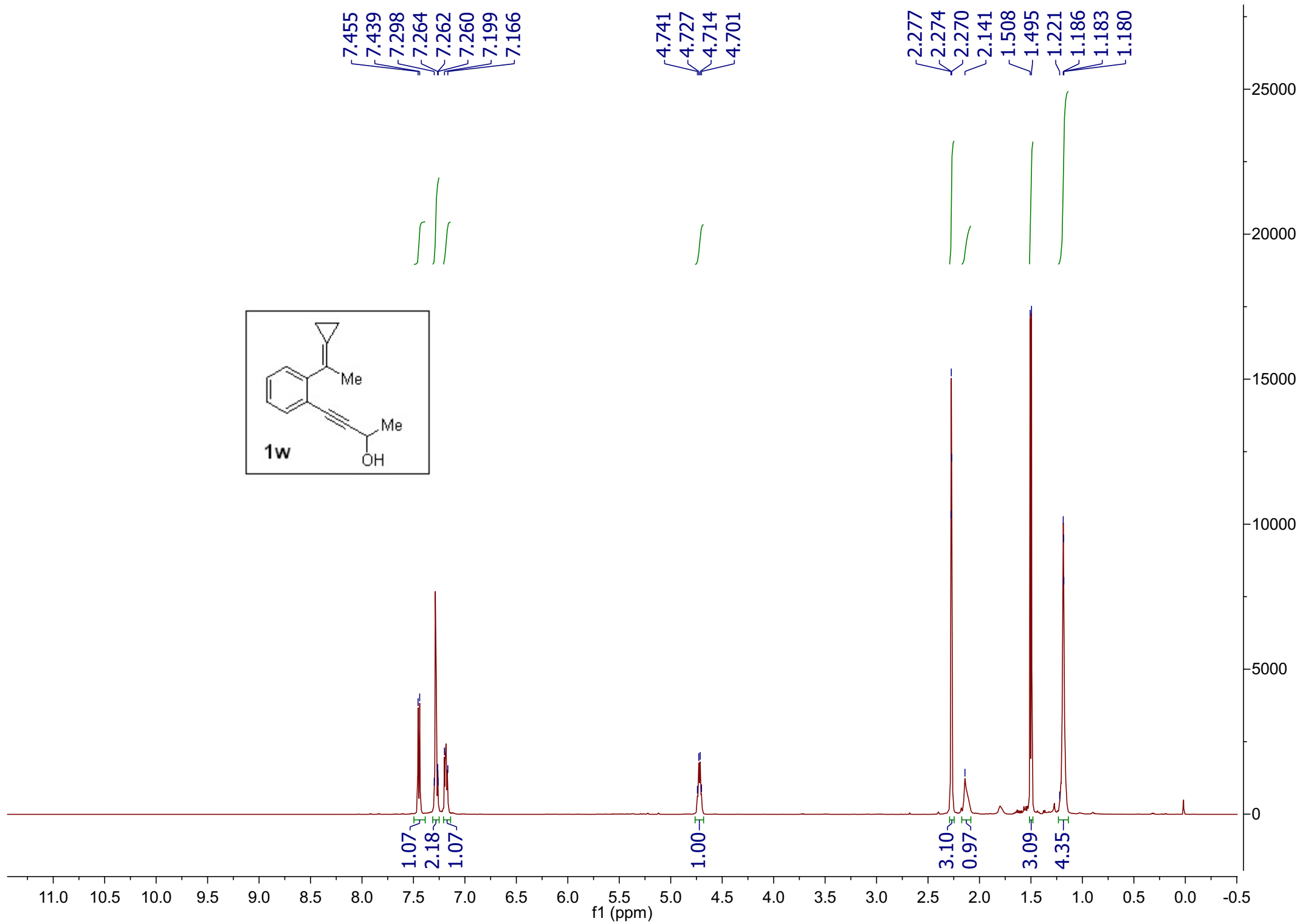
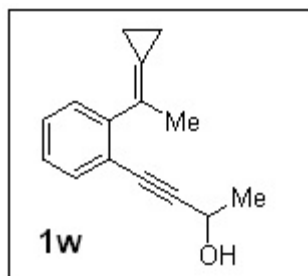
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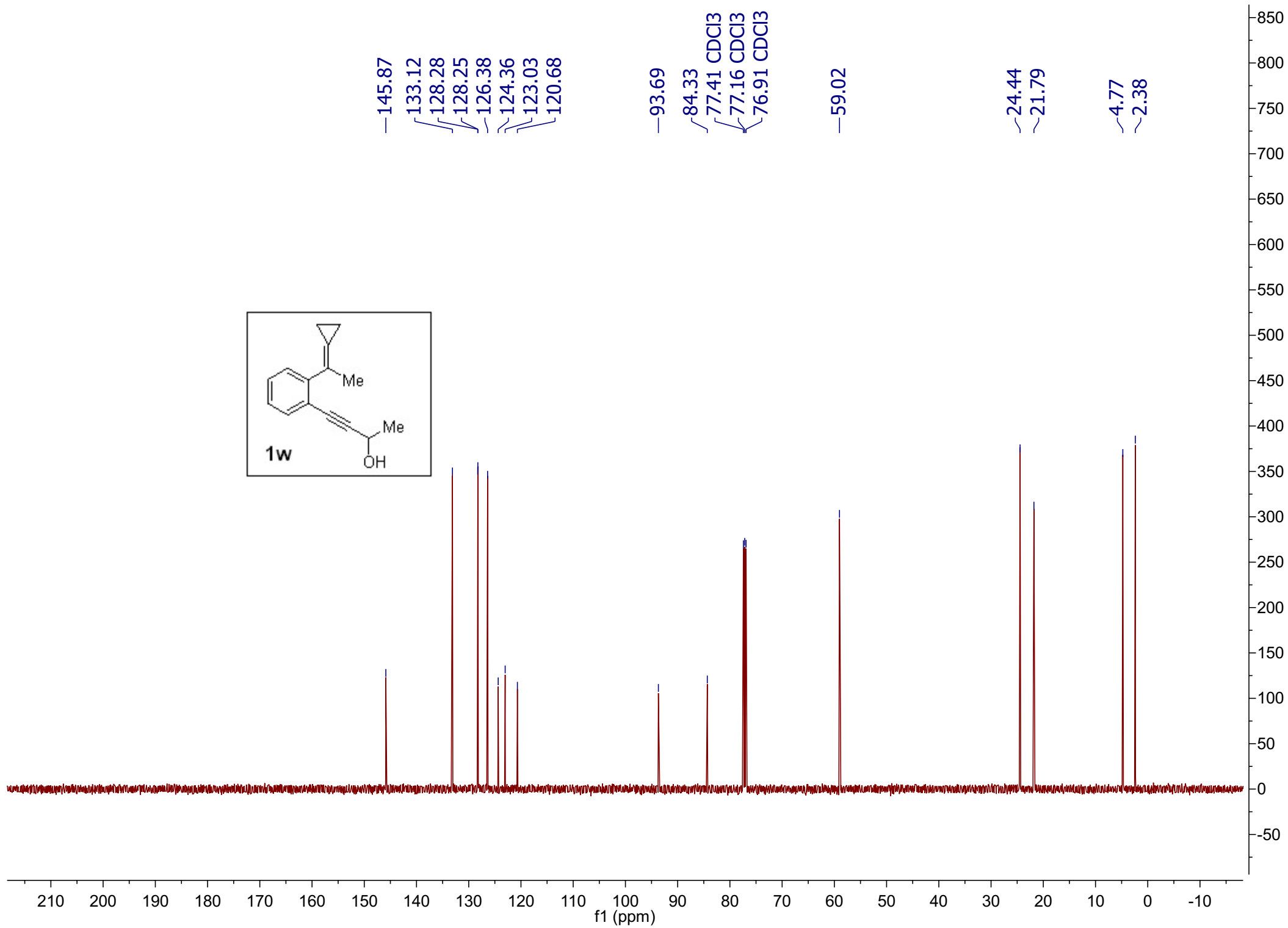
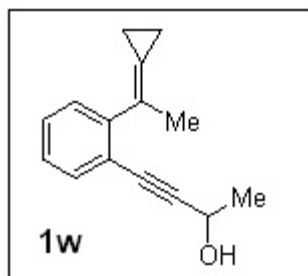
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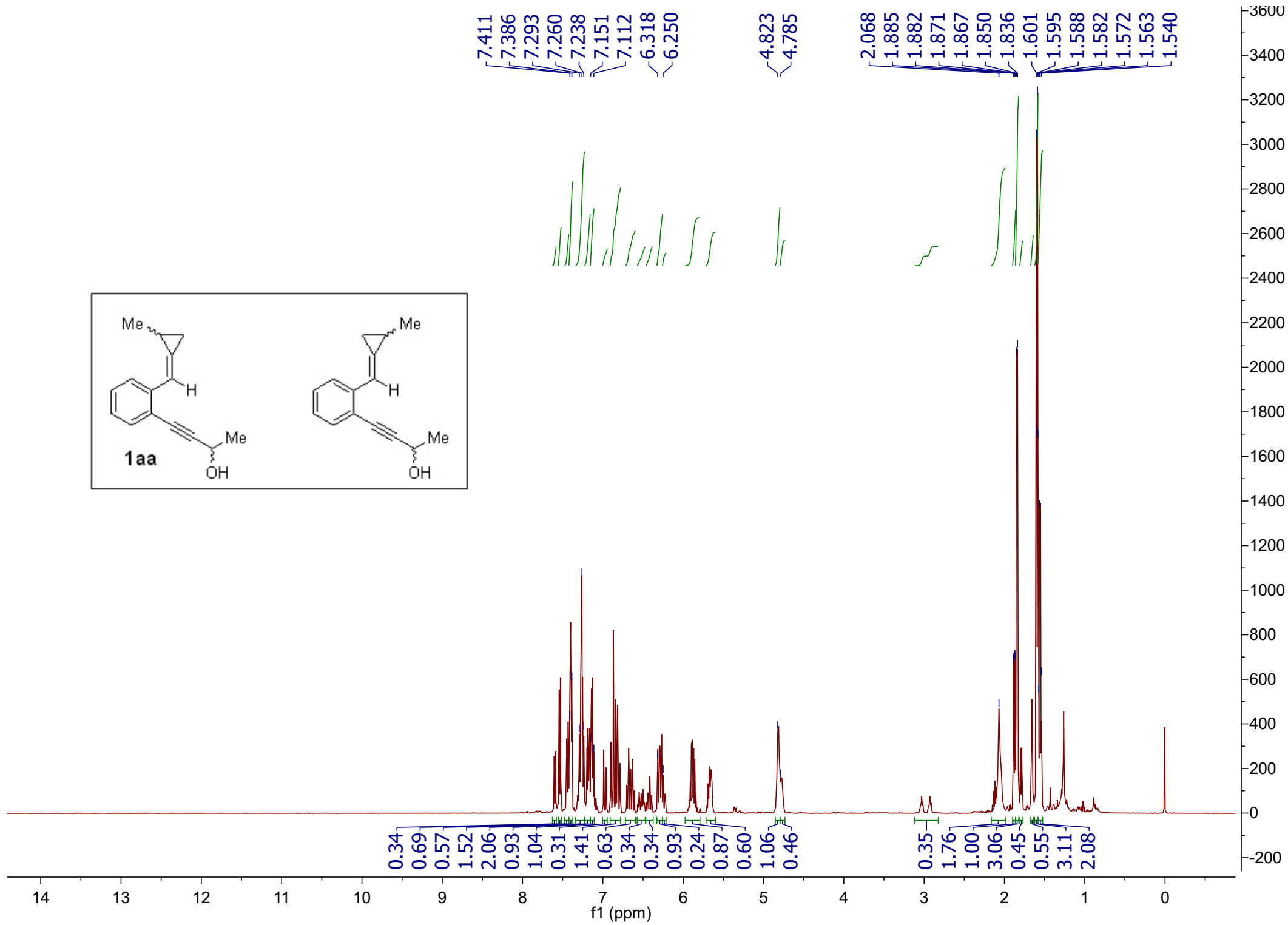
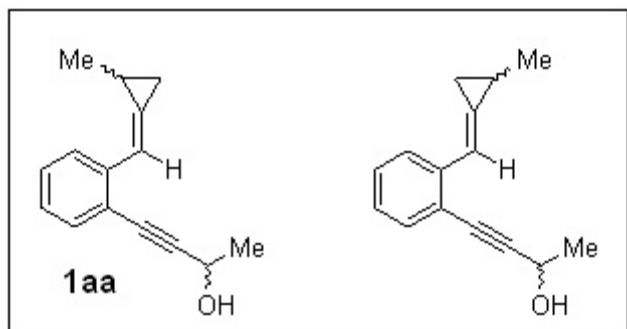
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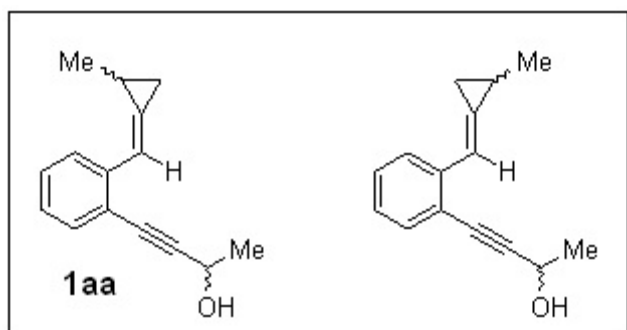
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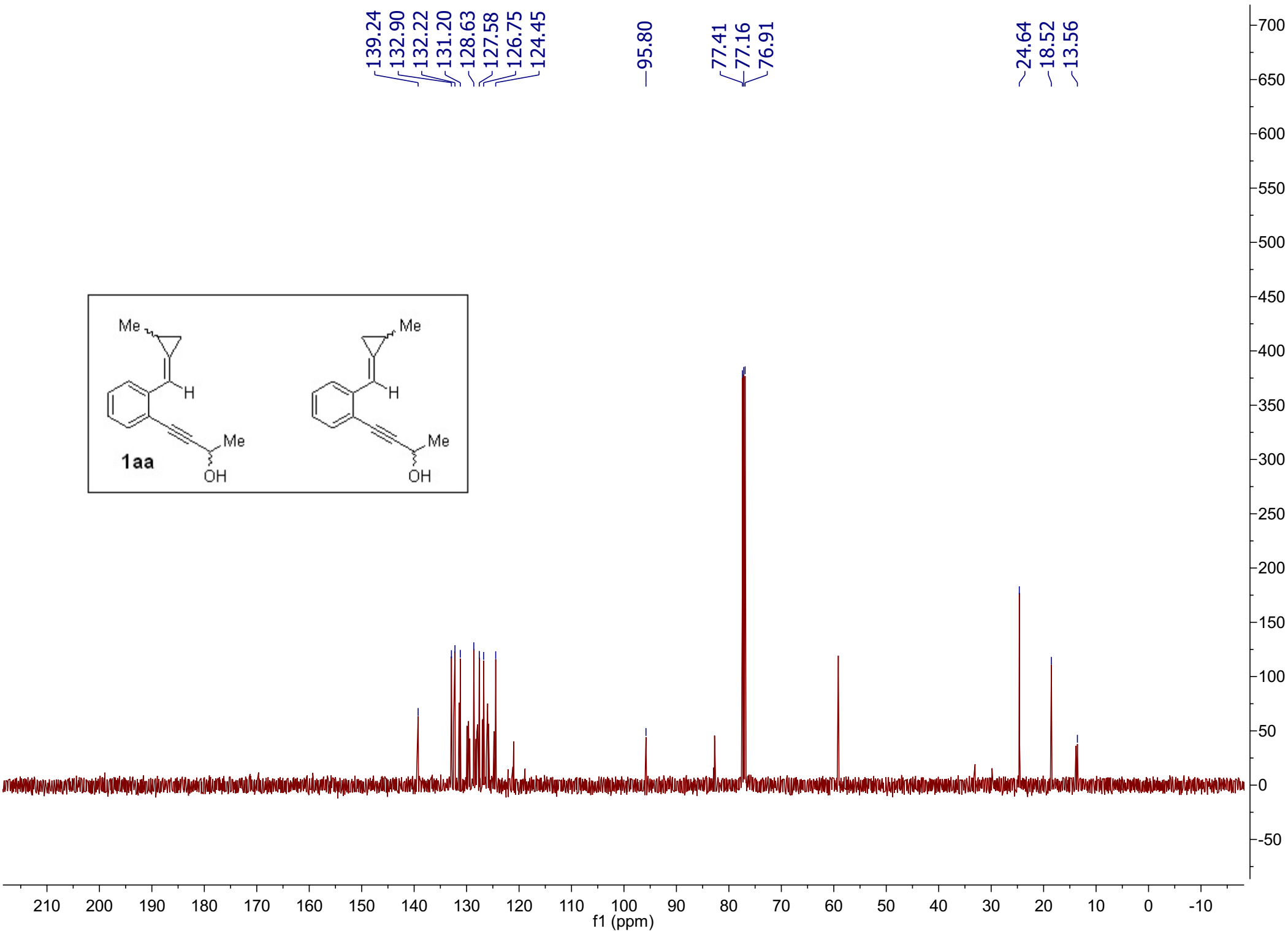


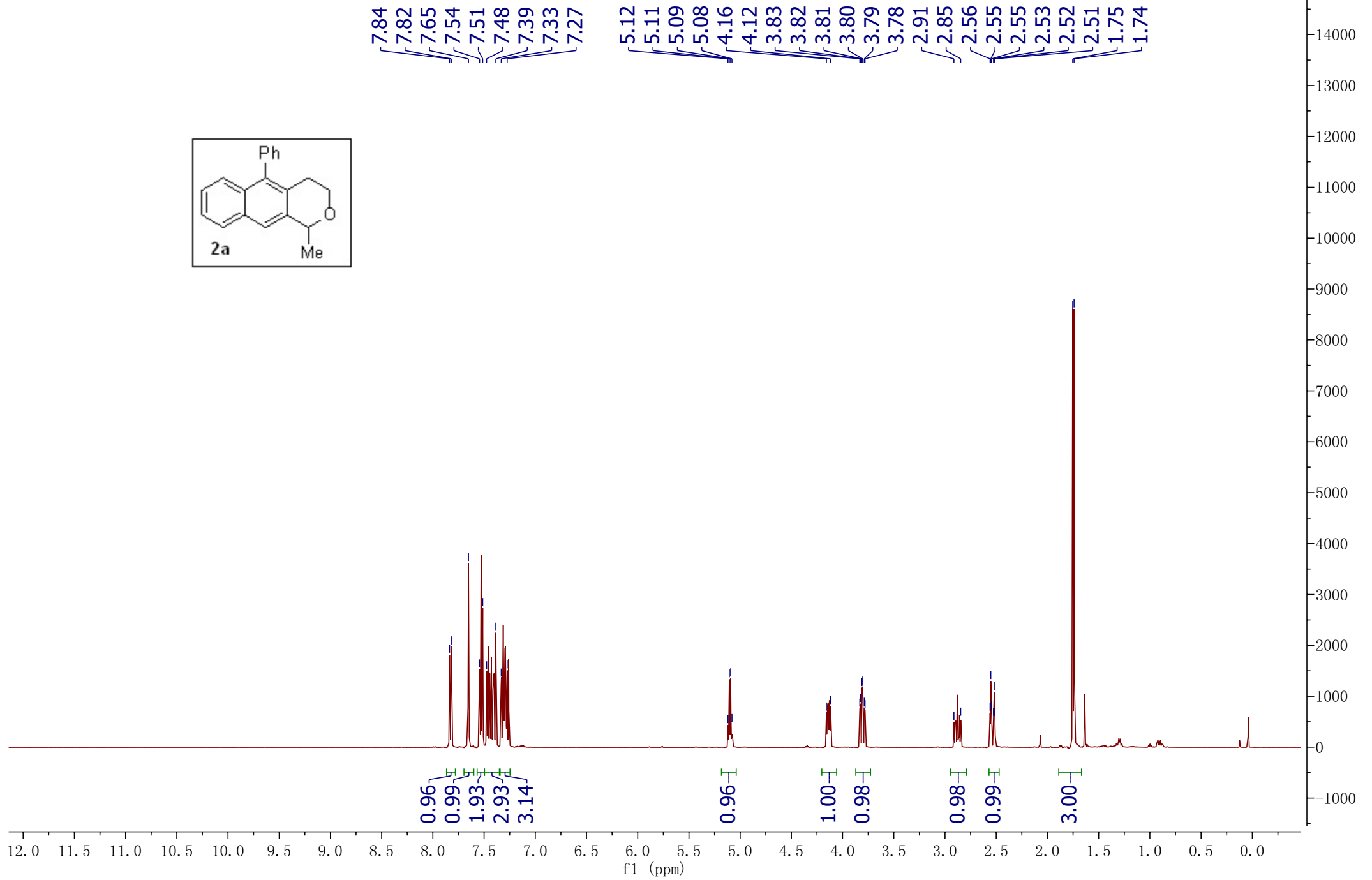
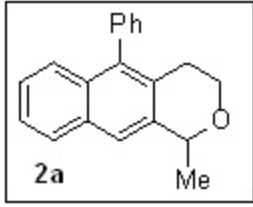
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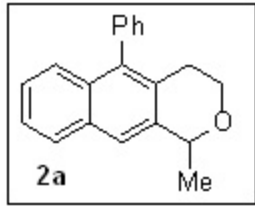
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24.64
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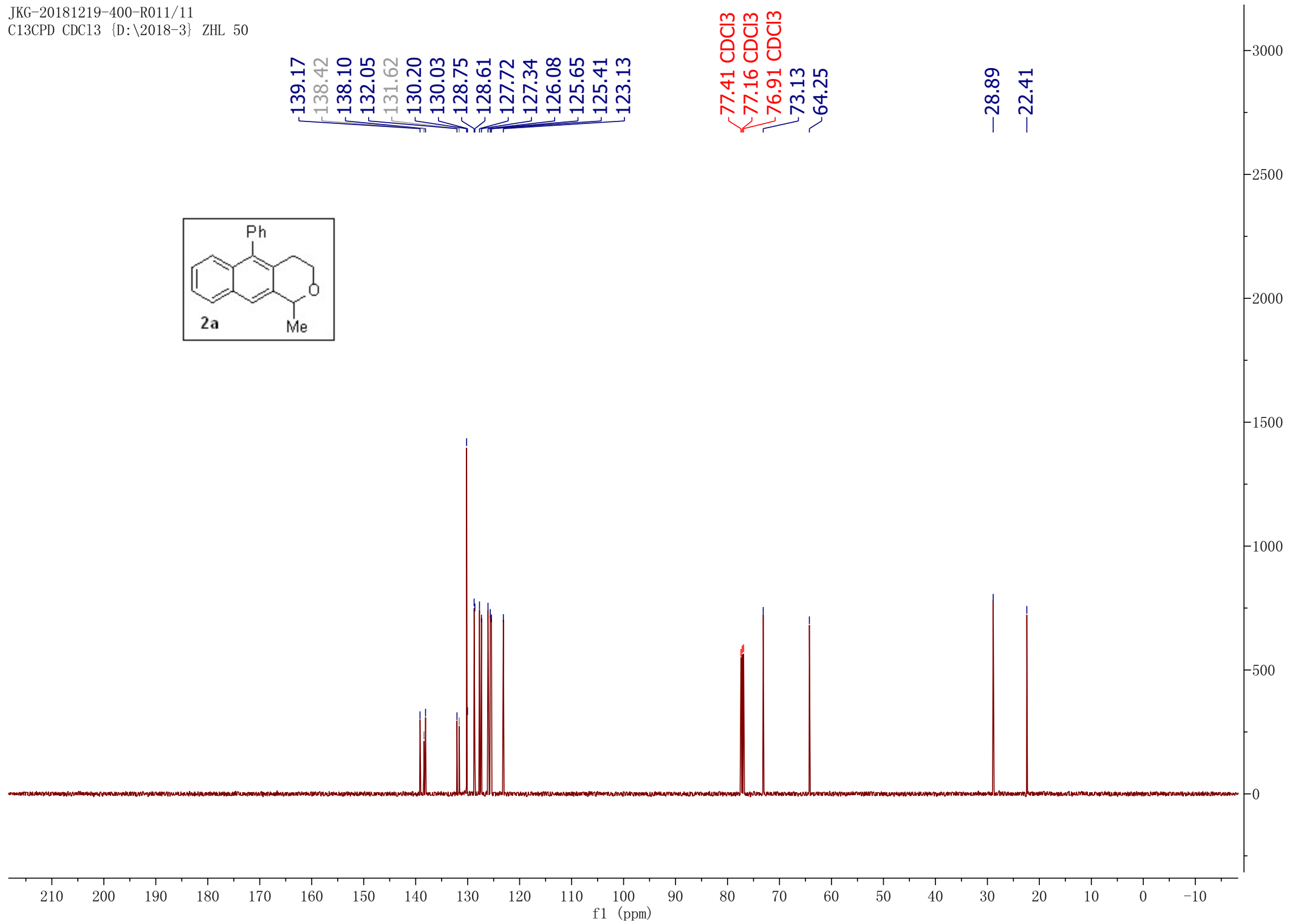


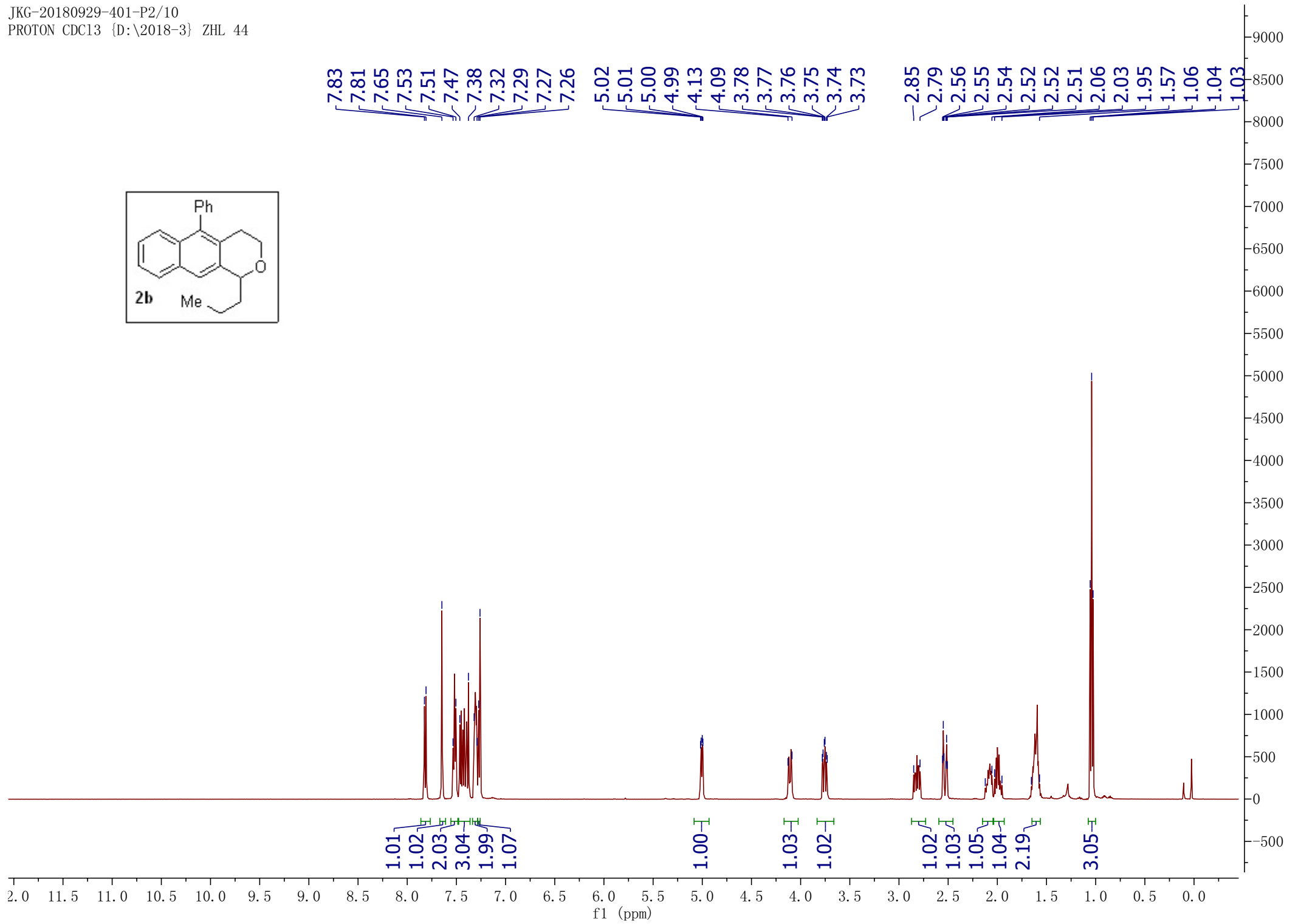
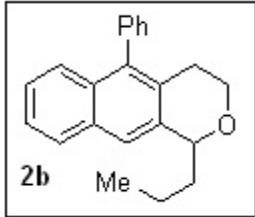


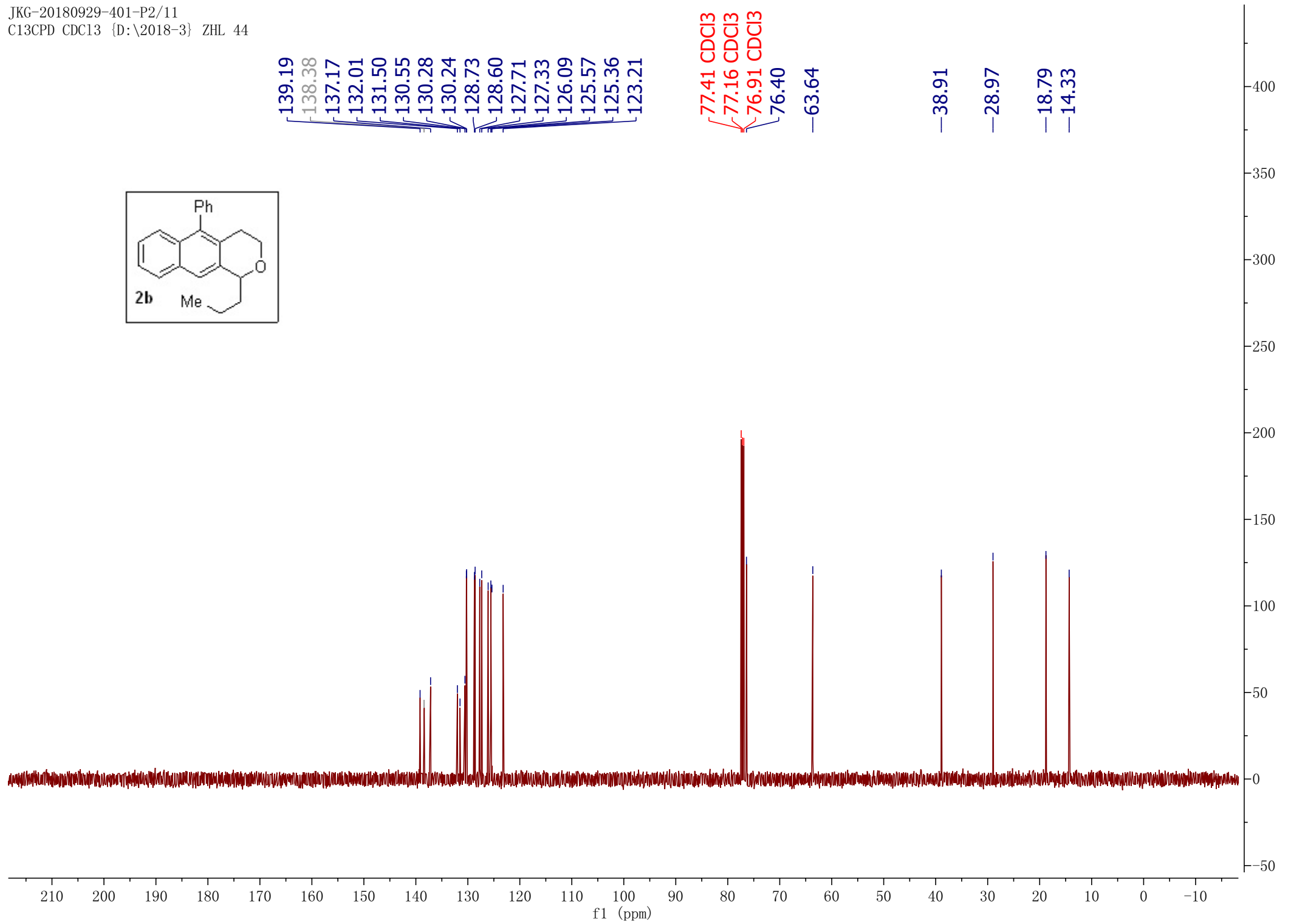
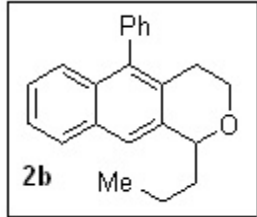
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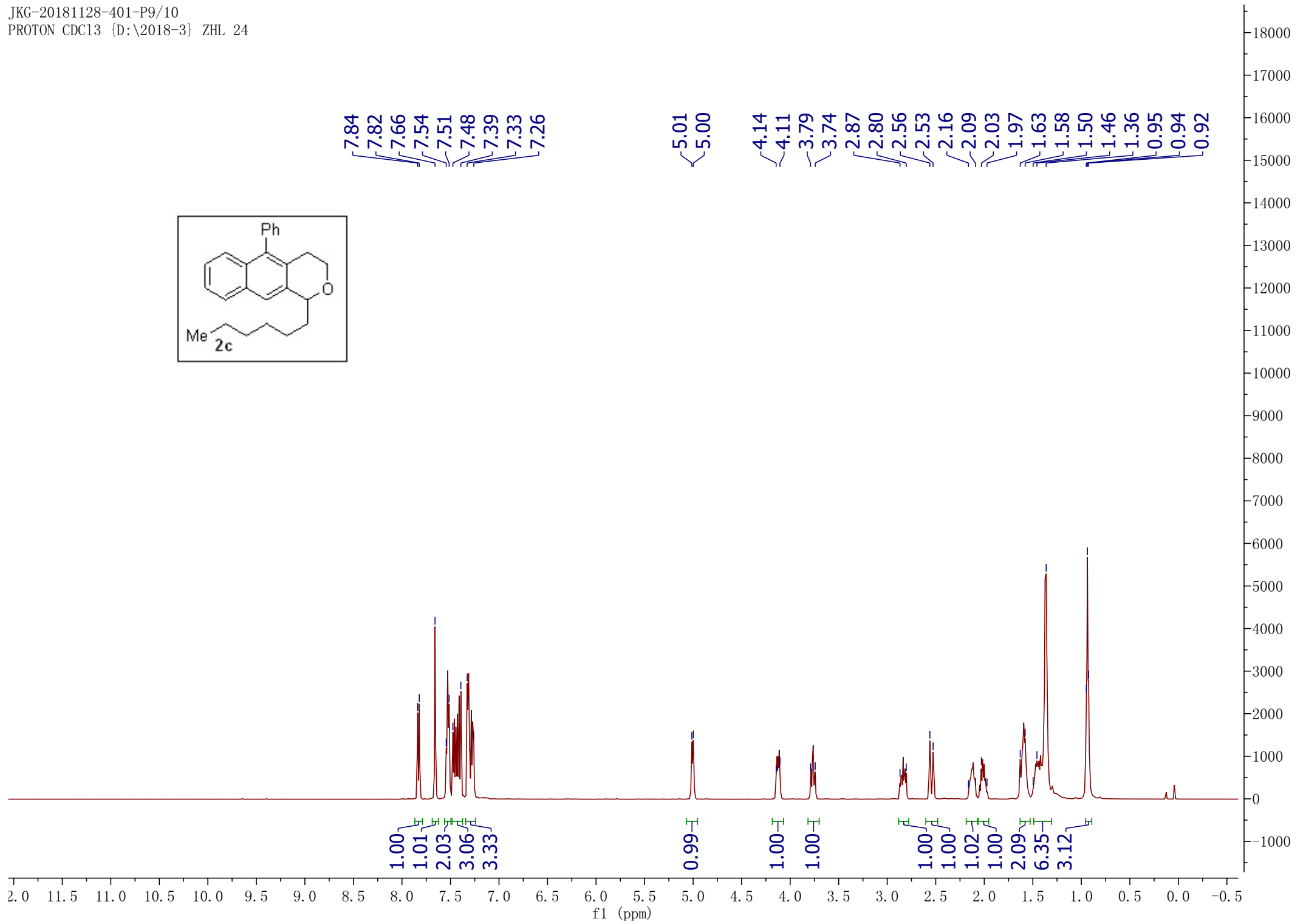
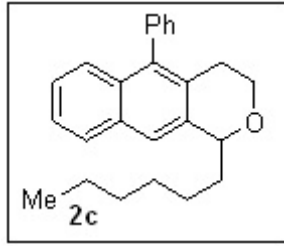
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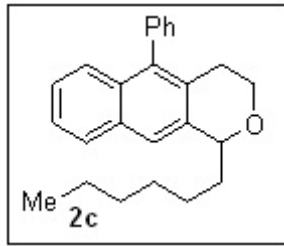
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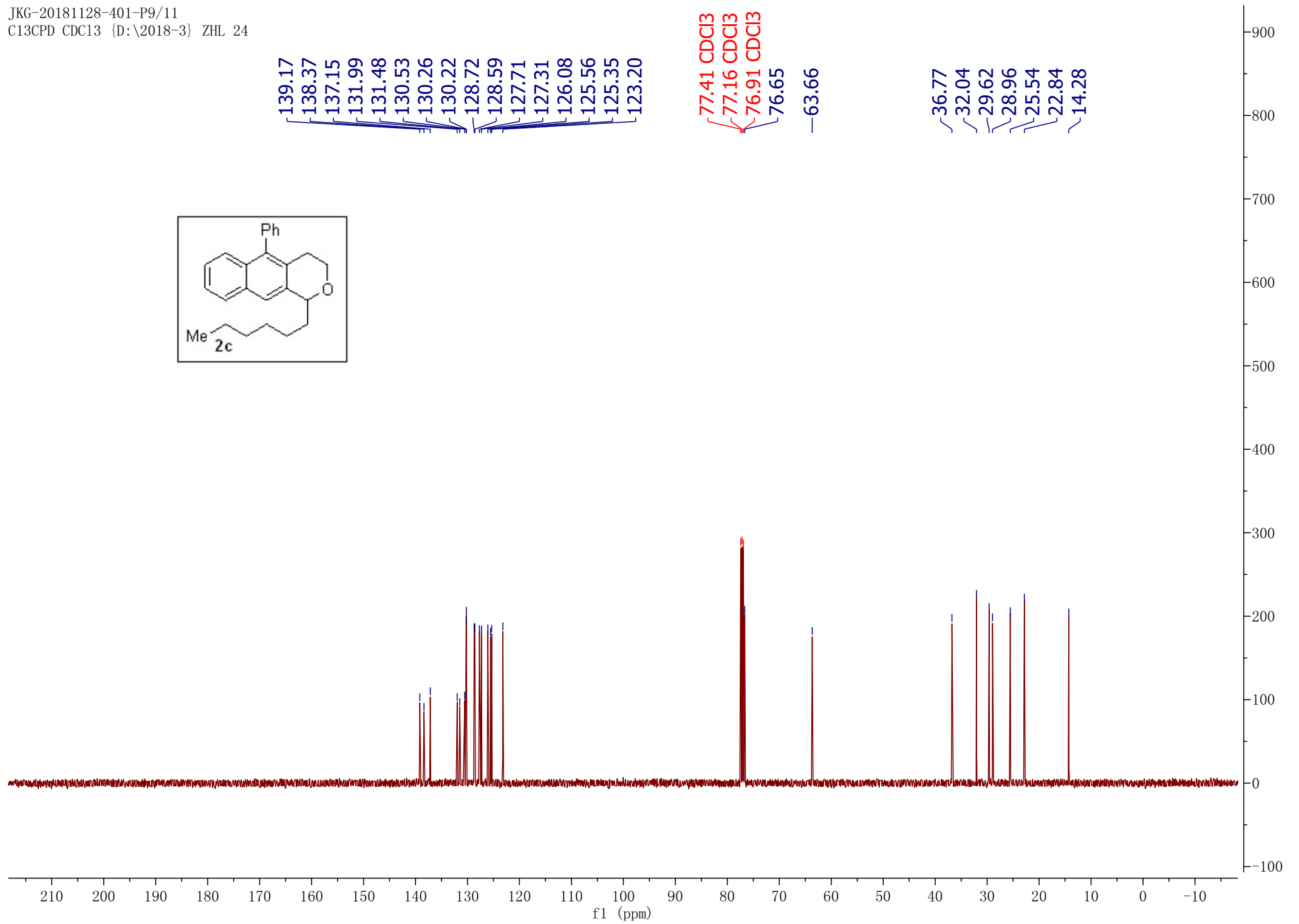


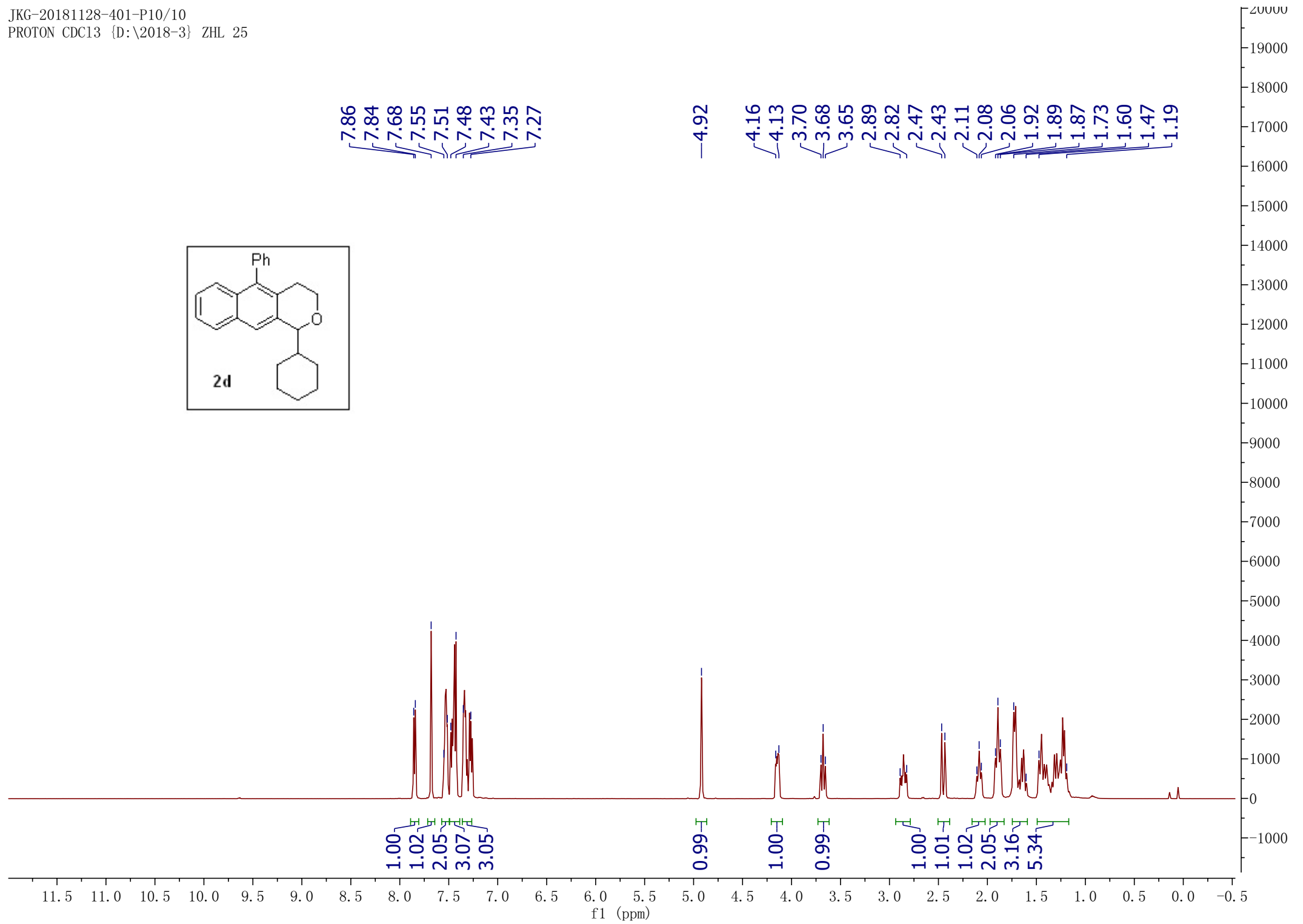
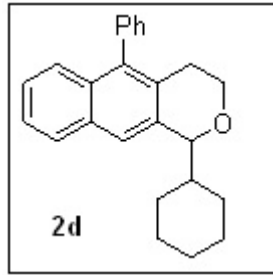
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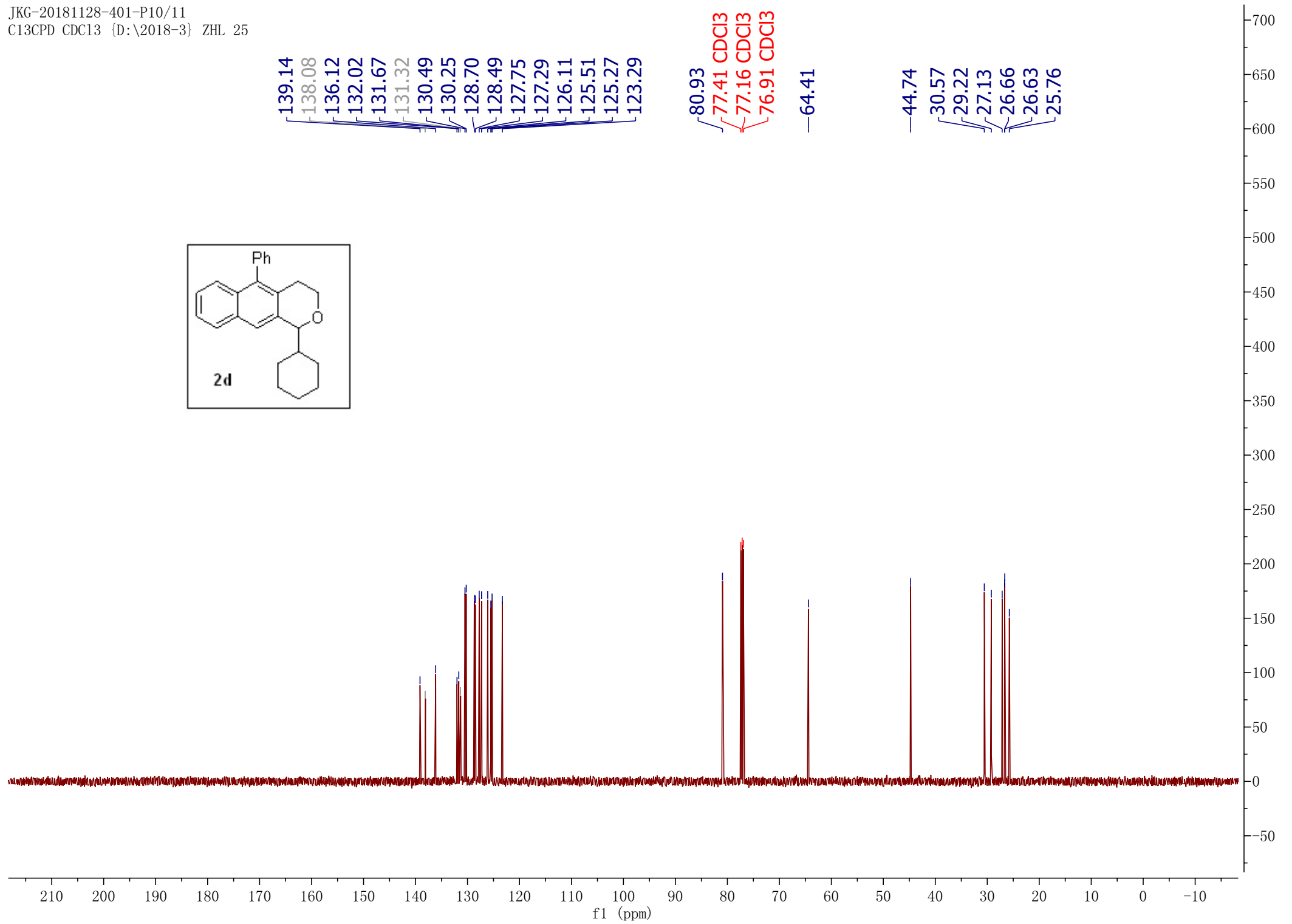
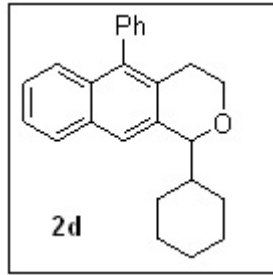
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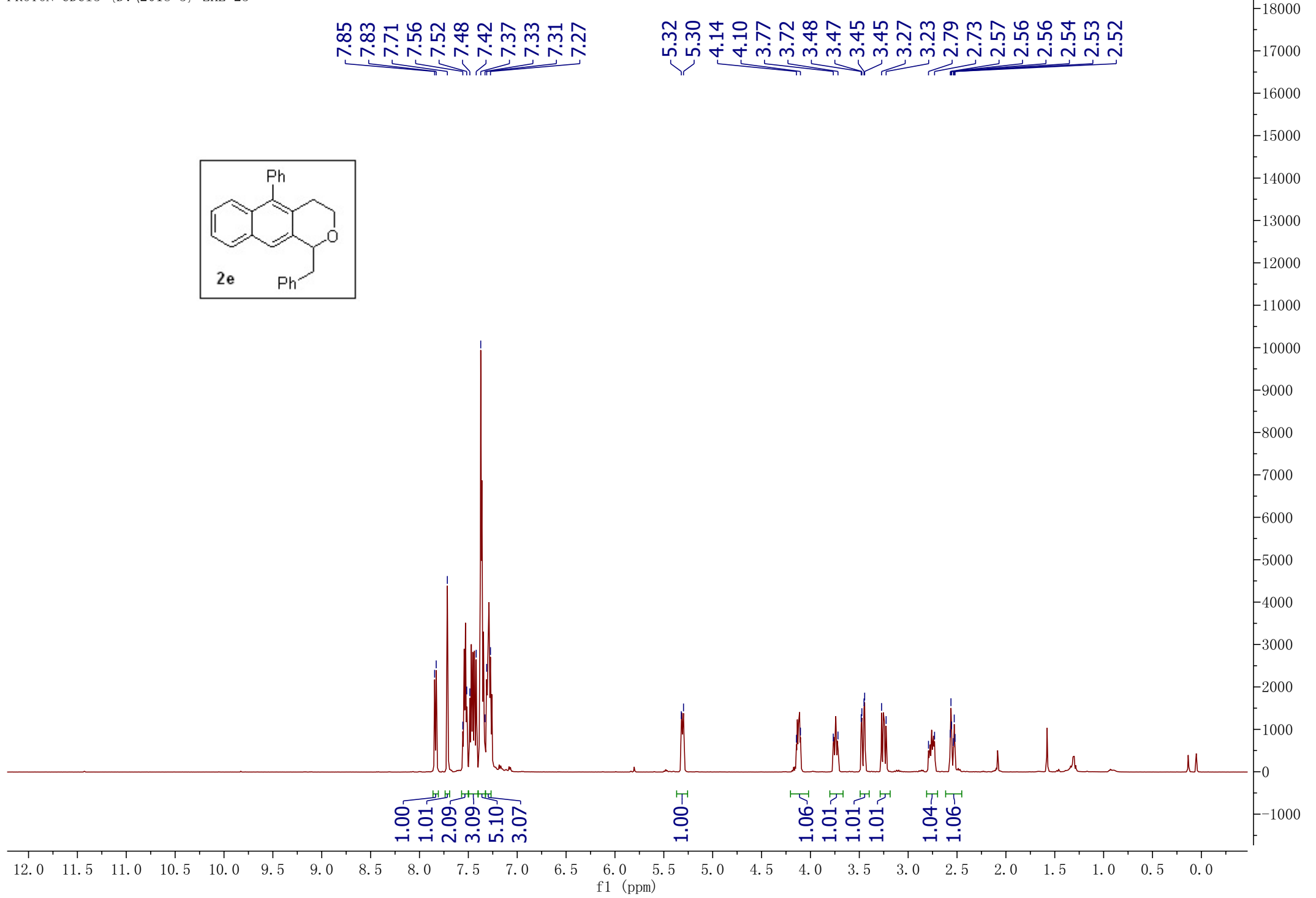
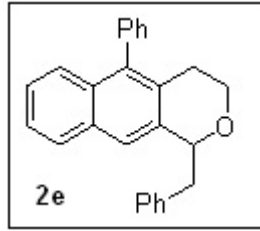
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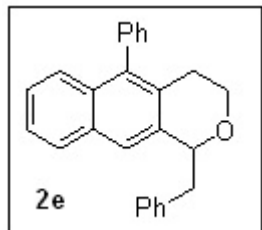
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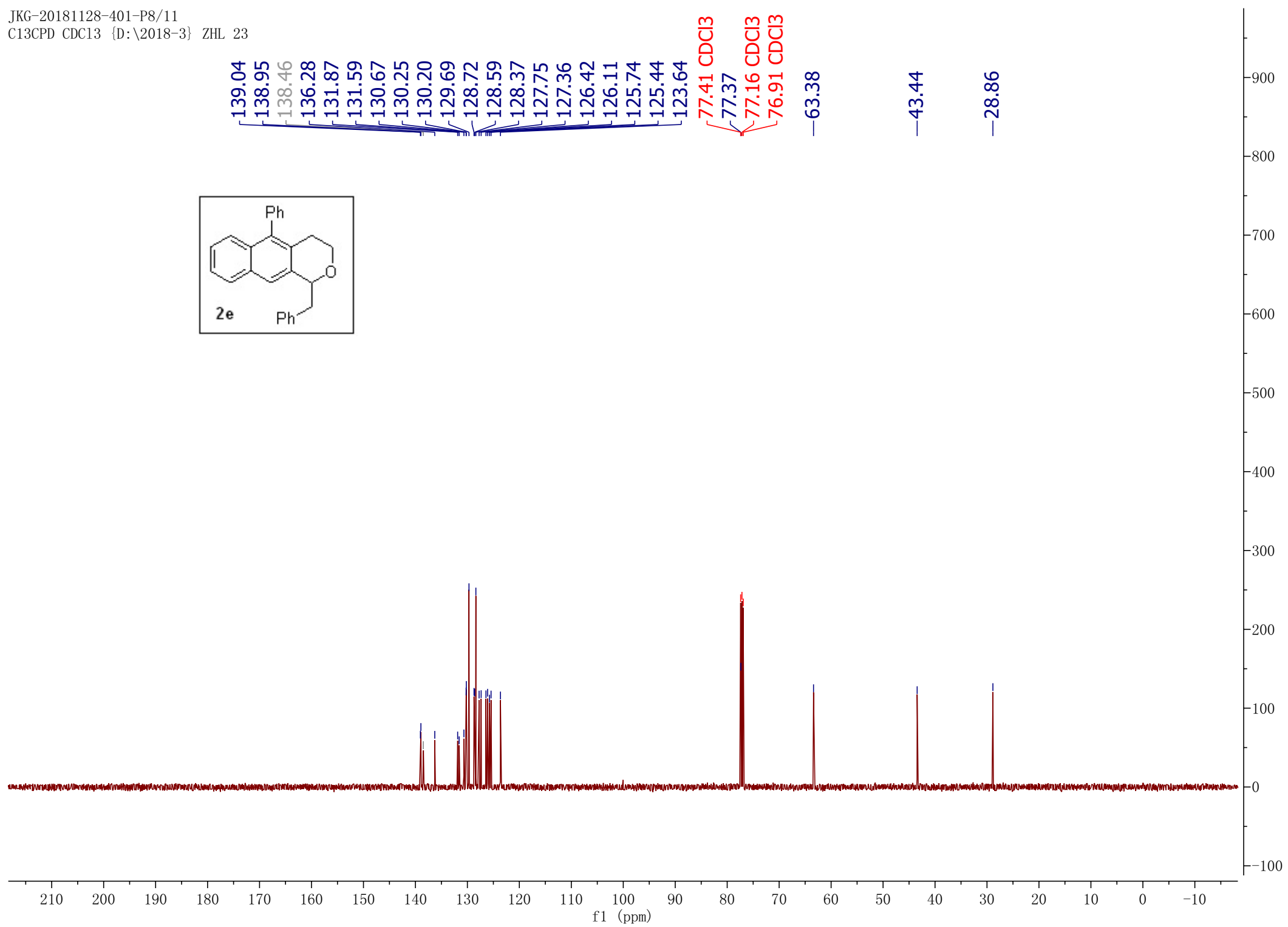


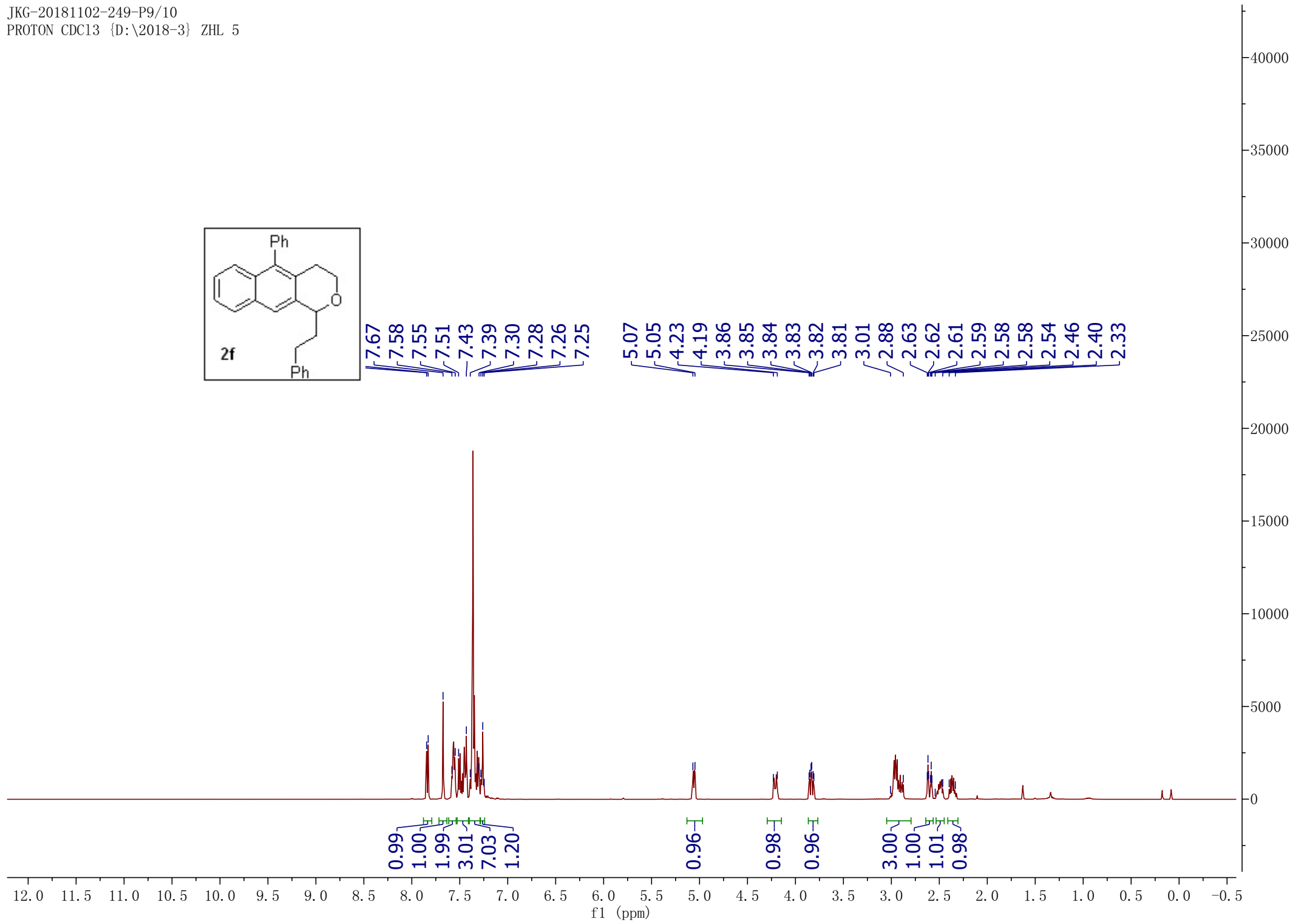
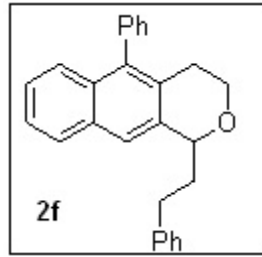


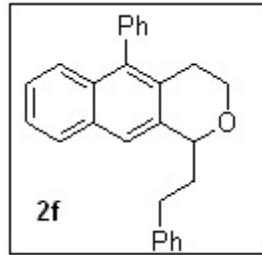




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76.91 CDC13
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43.44
28.86





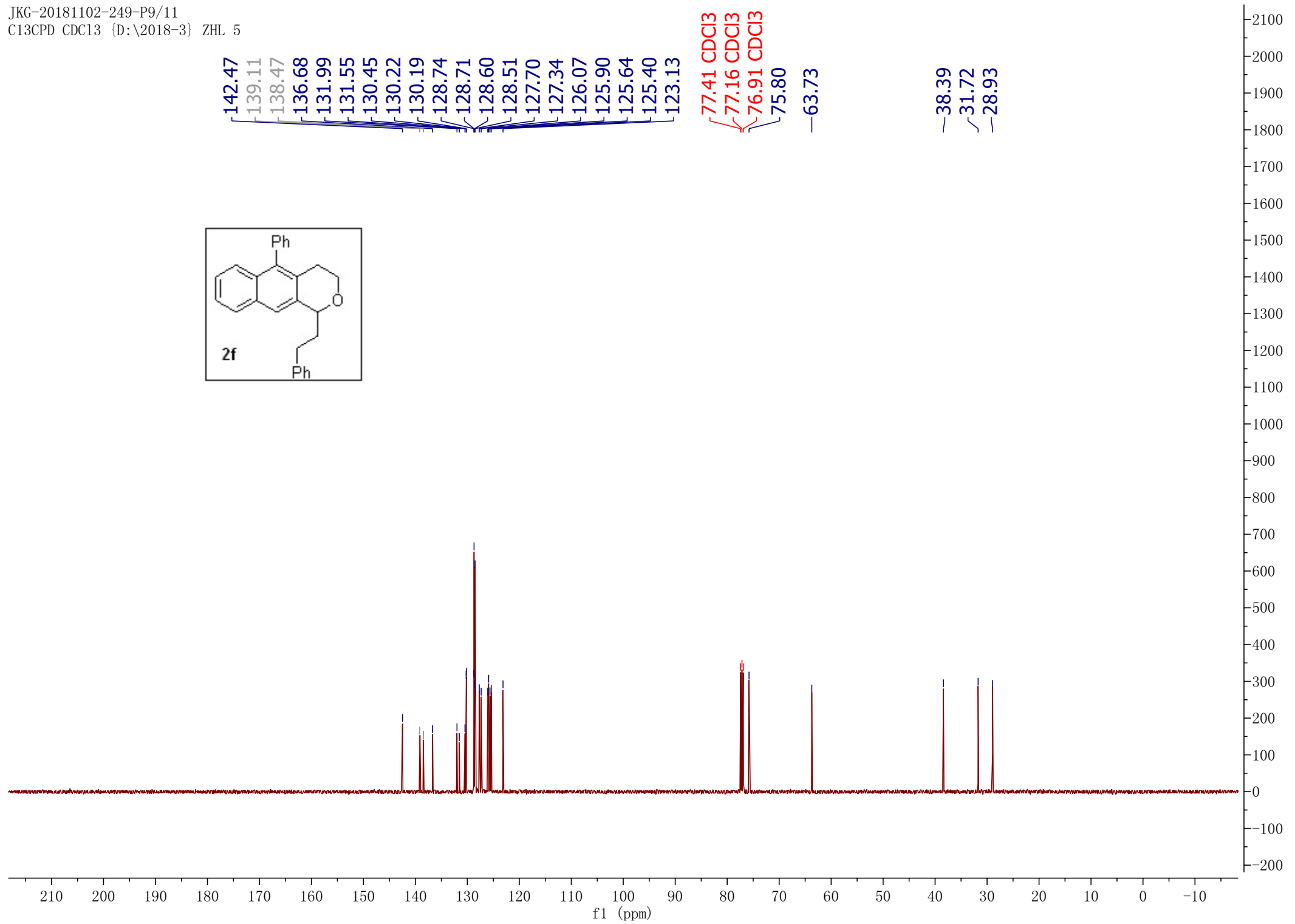


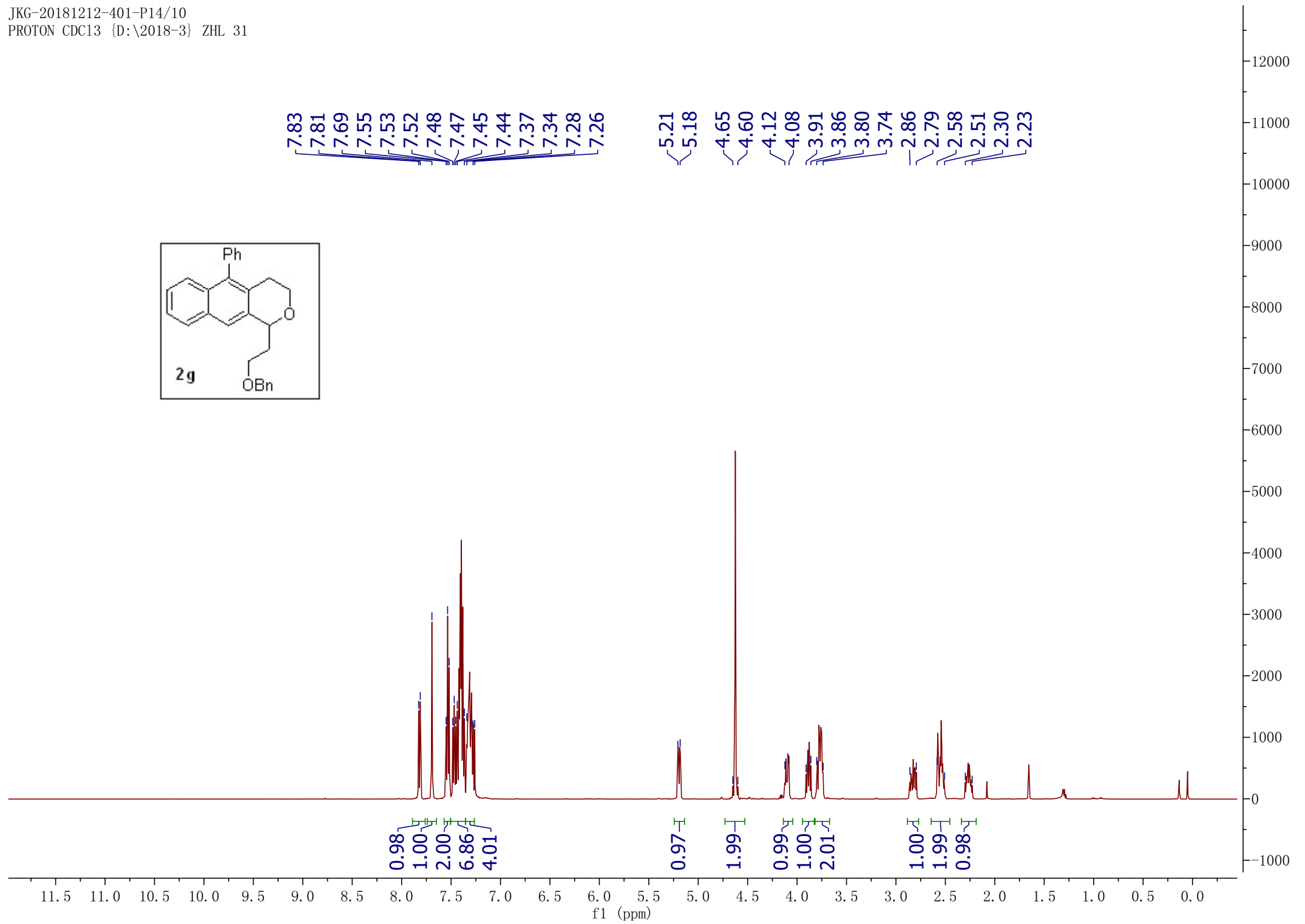
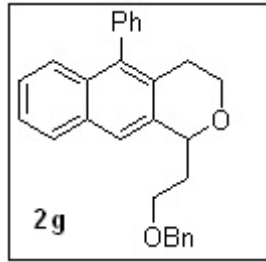
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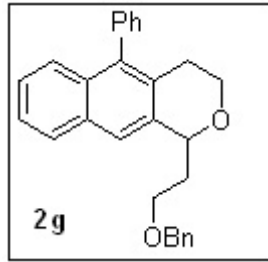
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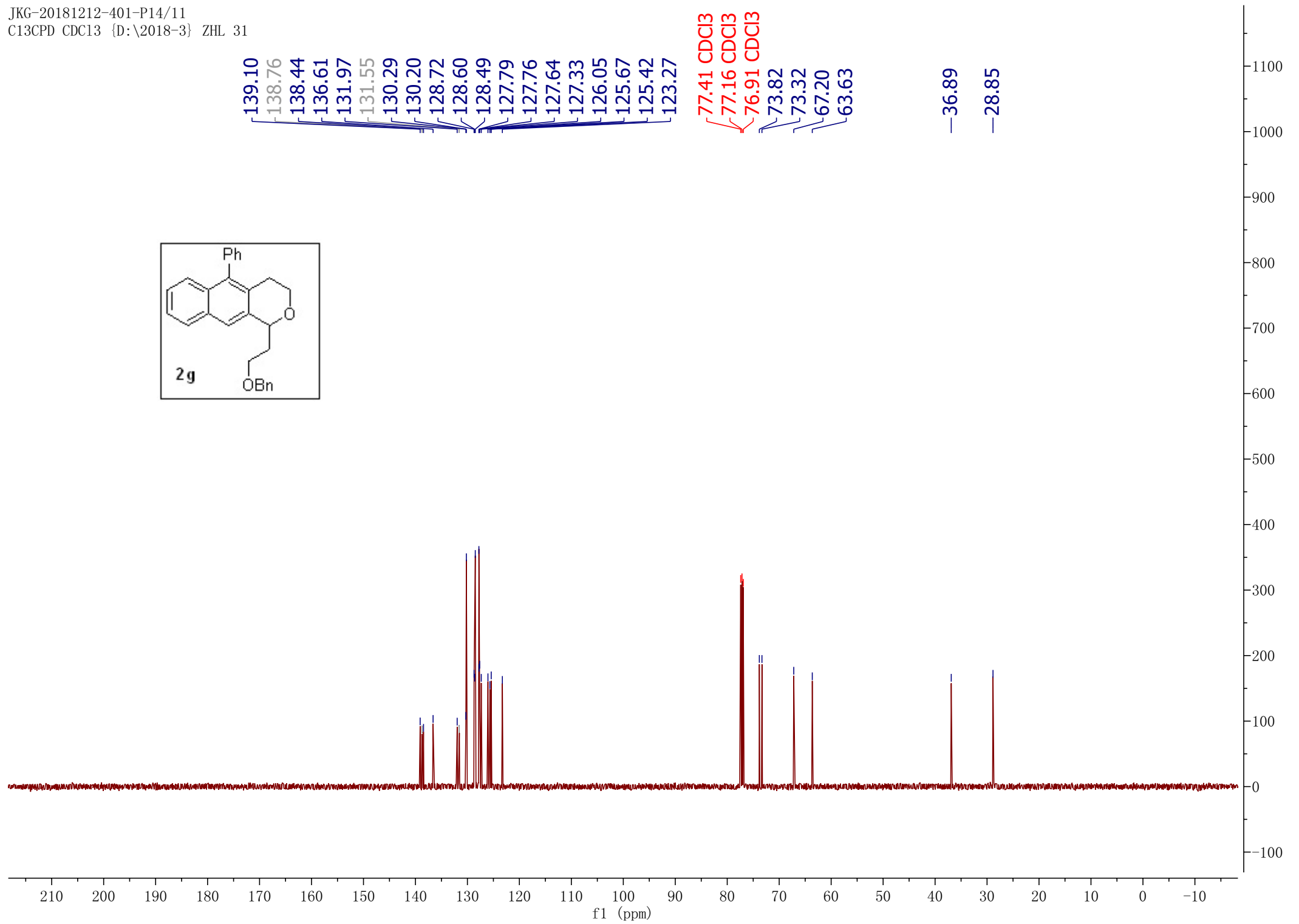
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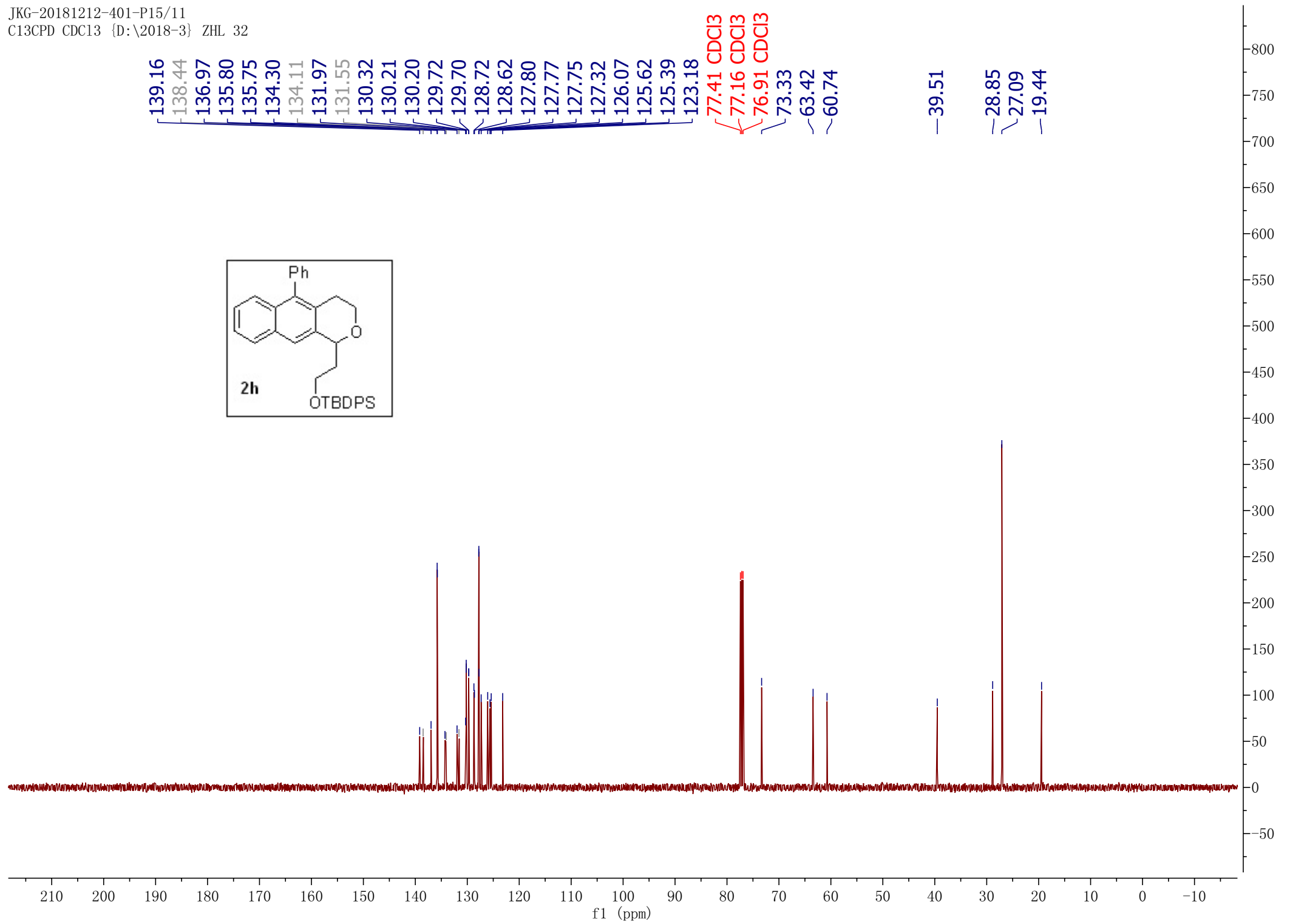
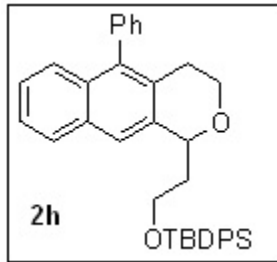
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126.05
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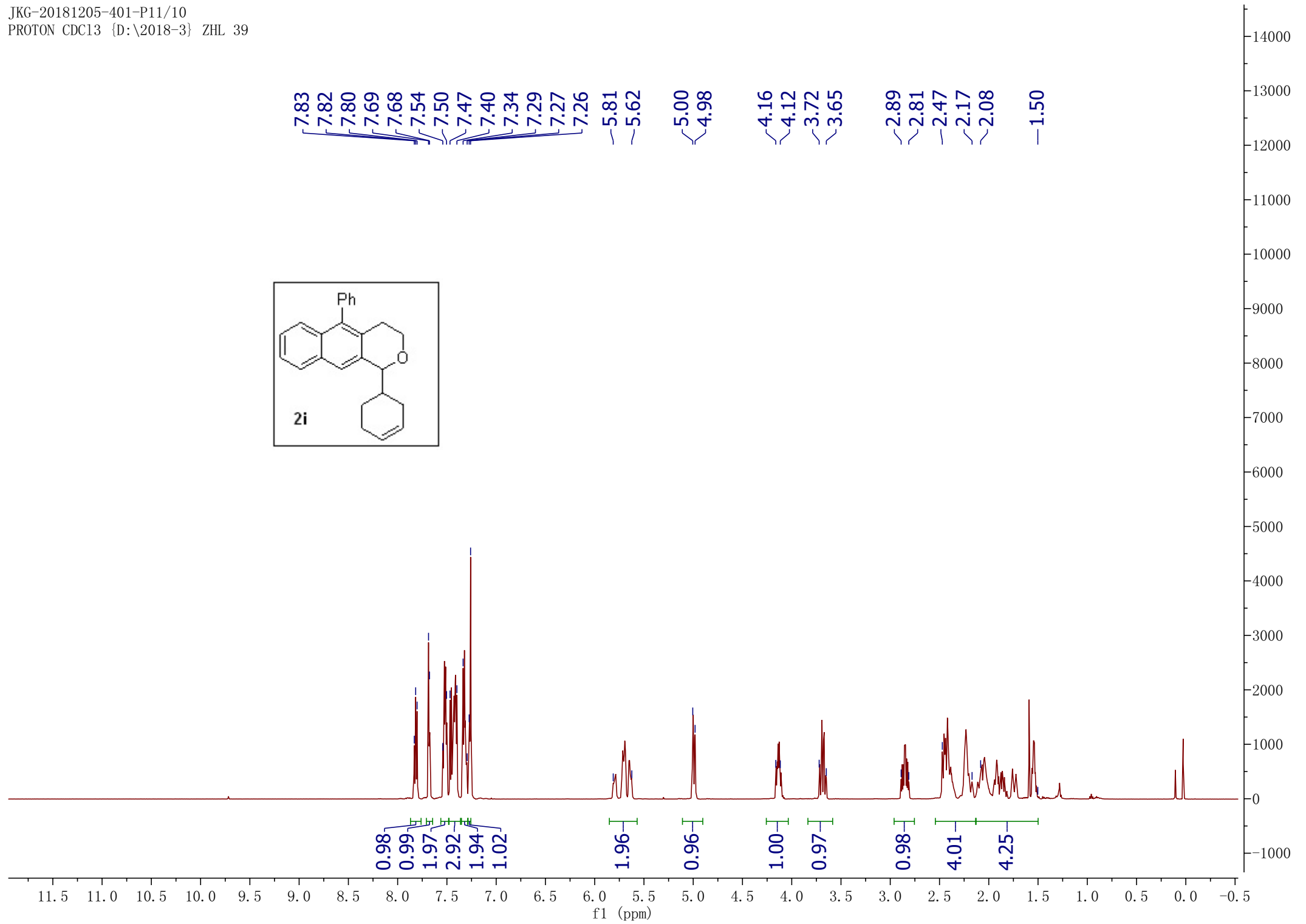
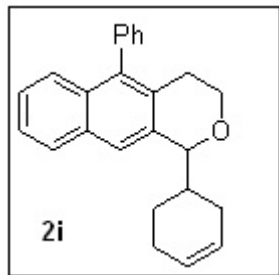


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125.39
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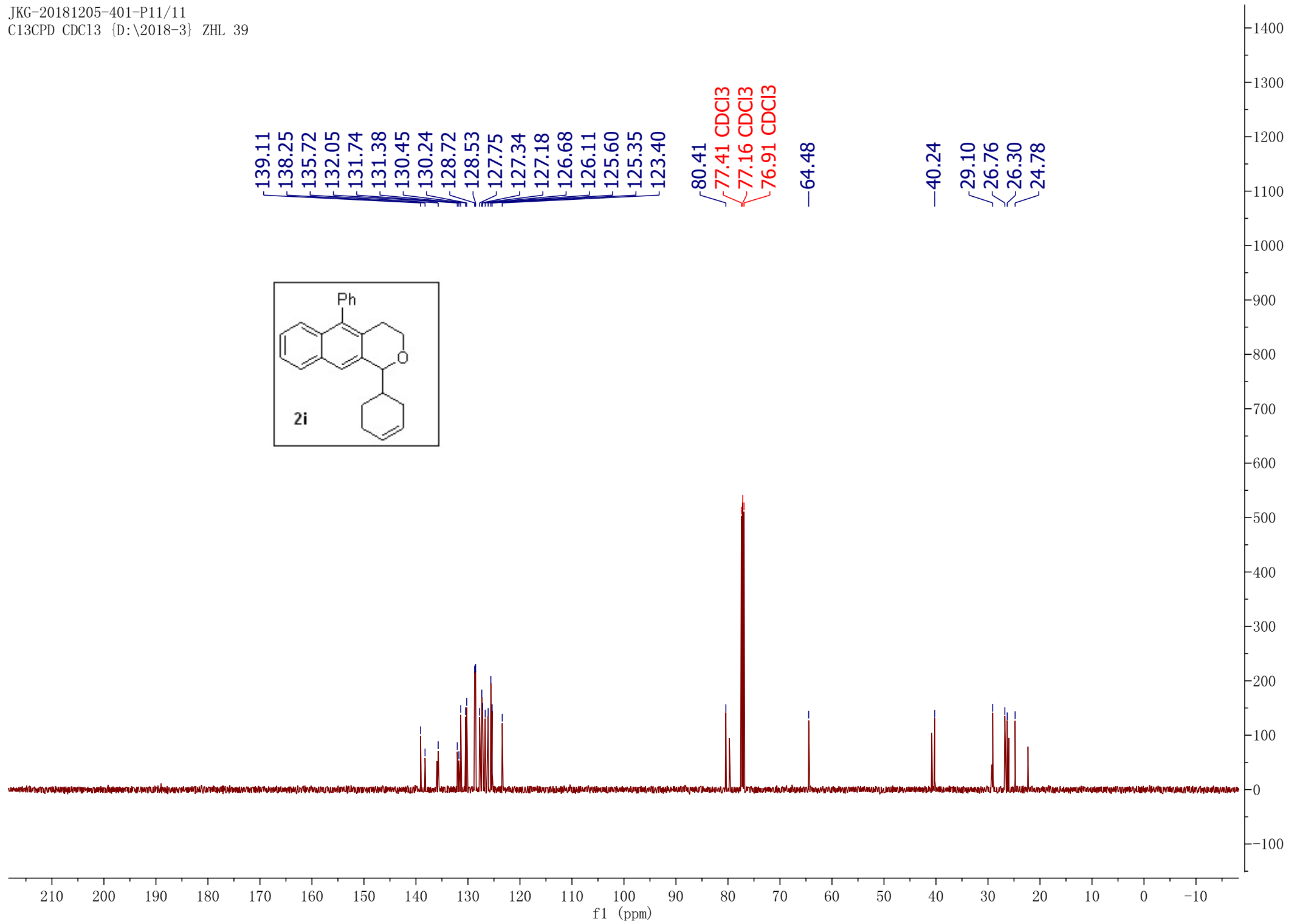
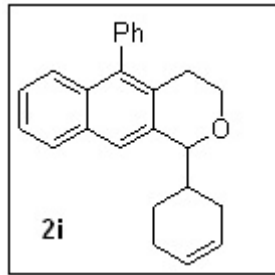
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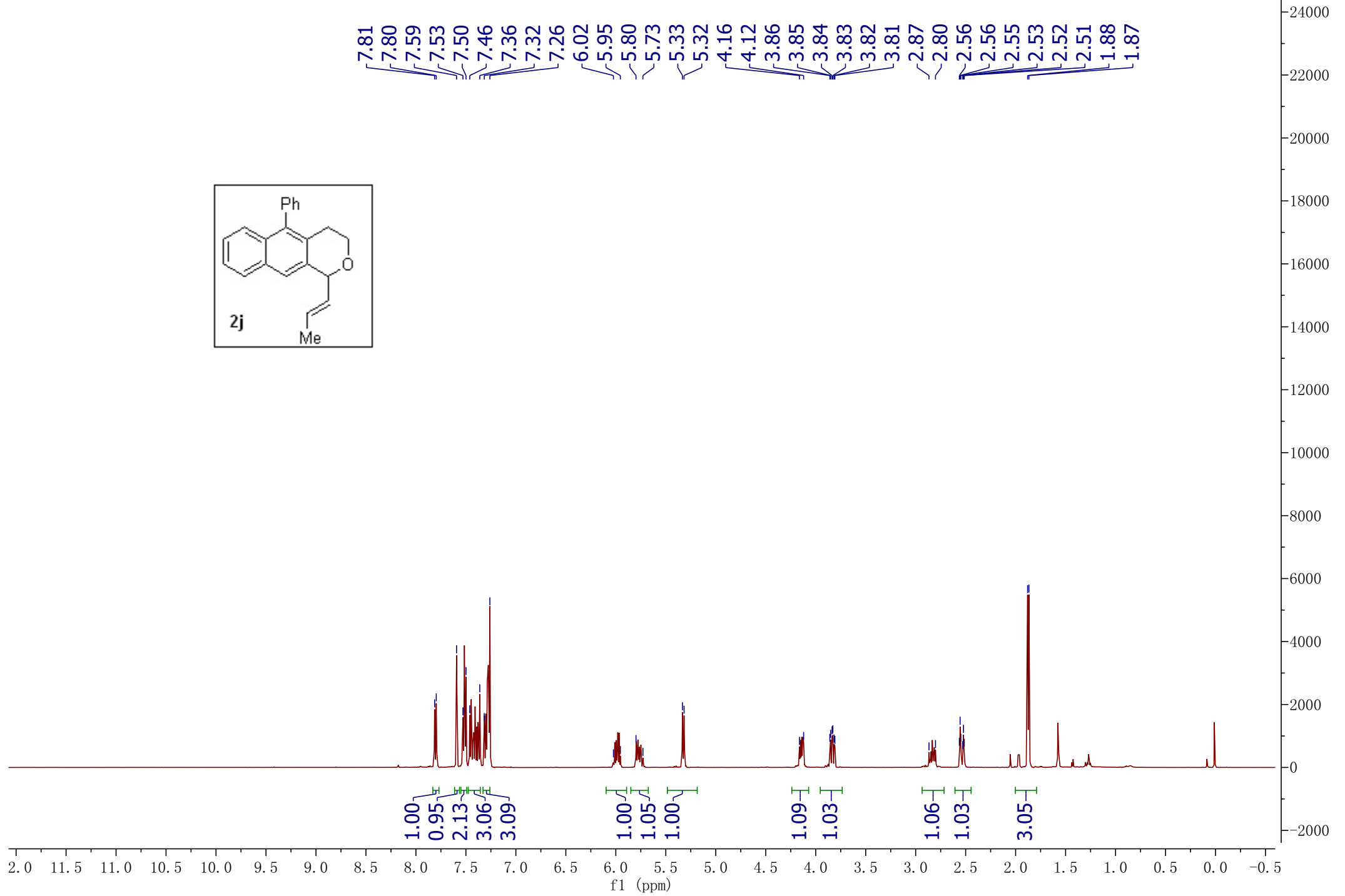
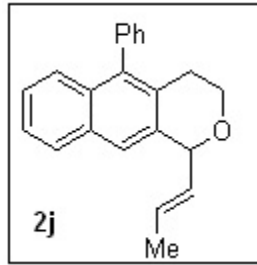
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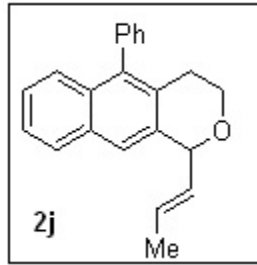
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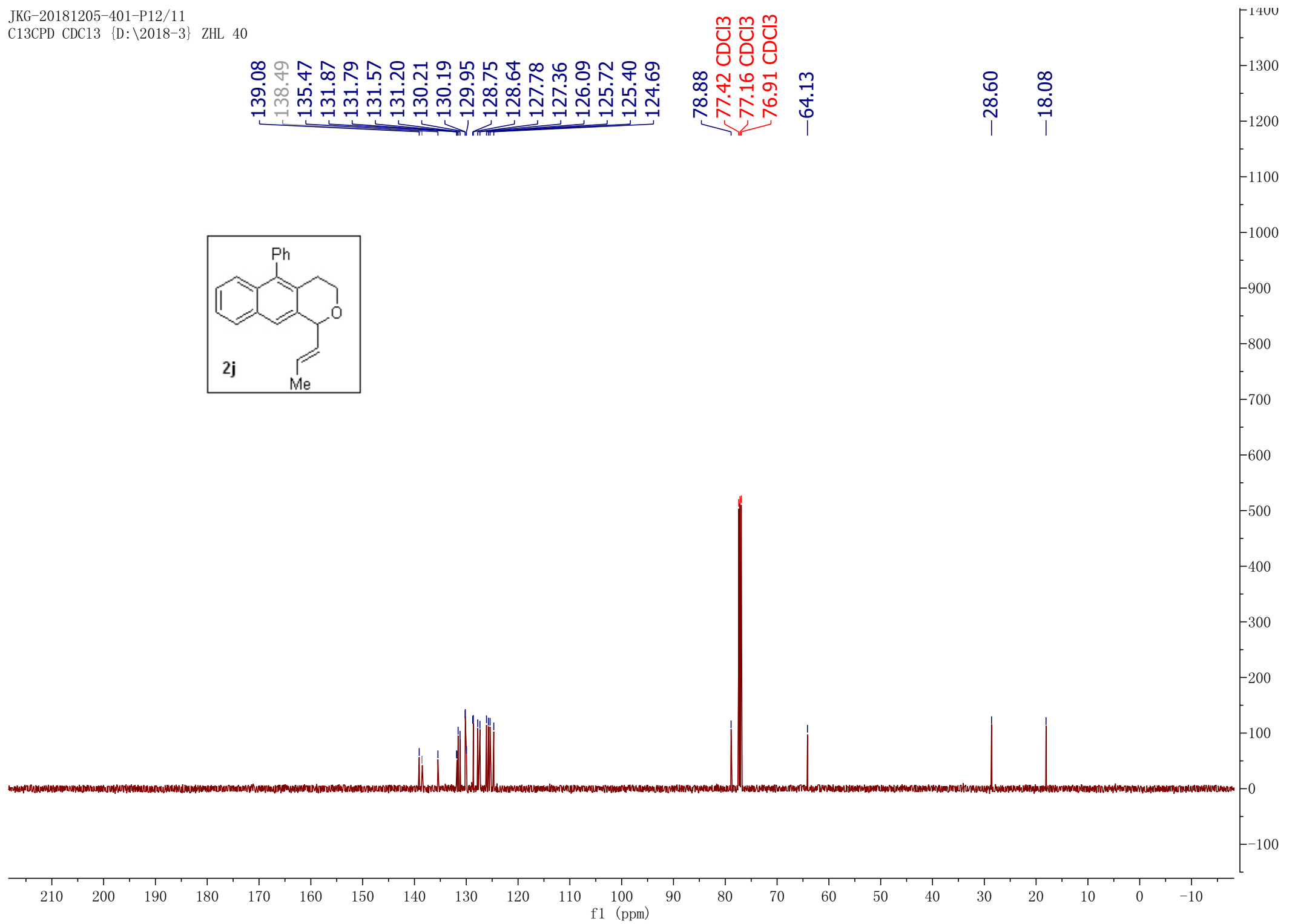




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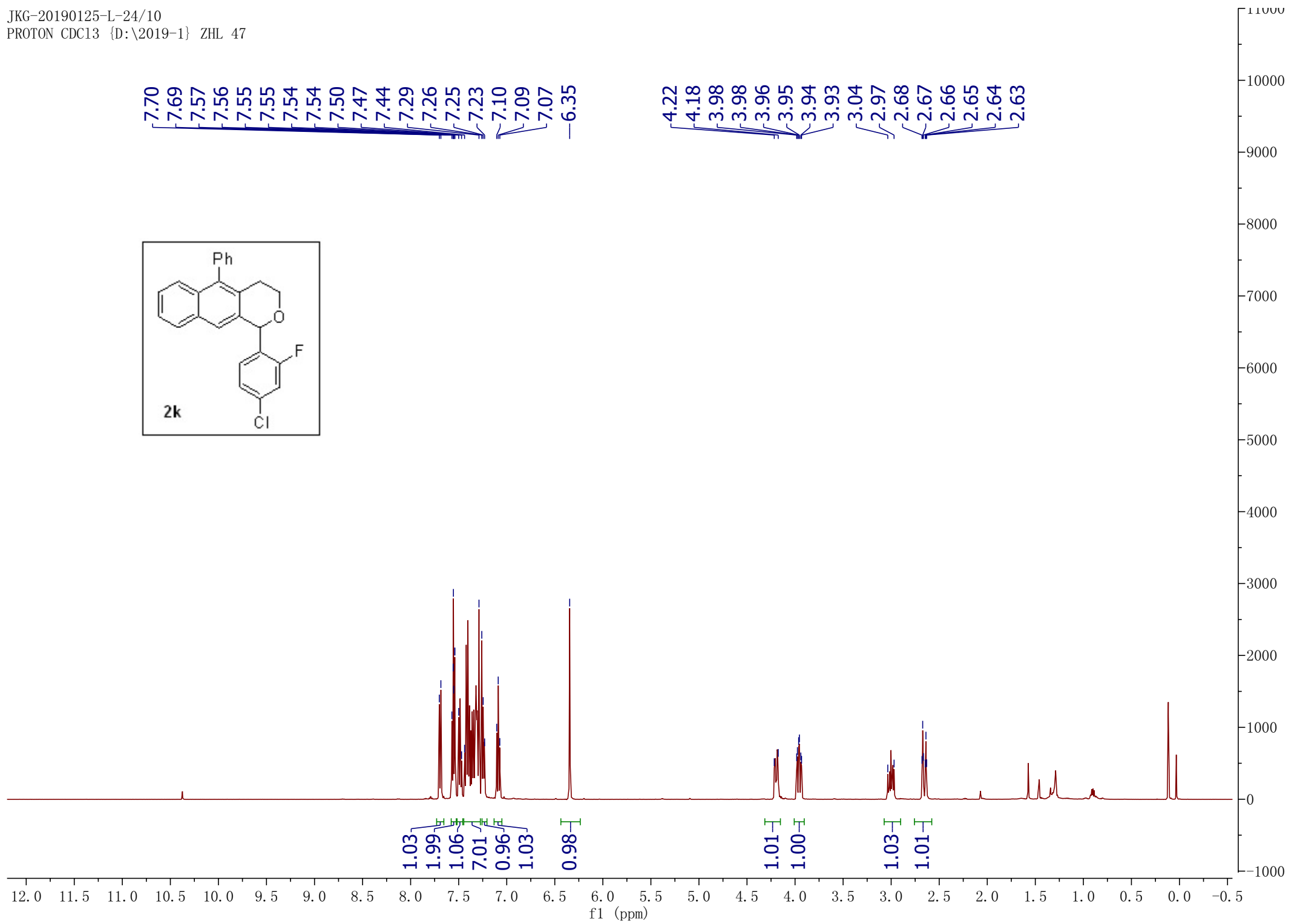
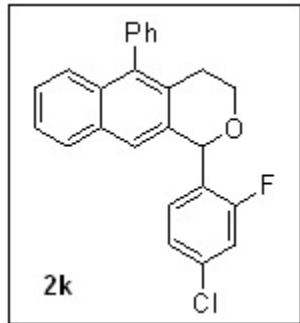
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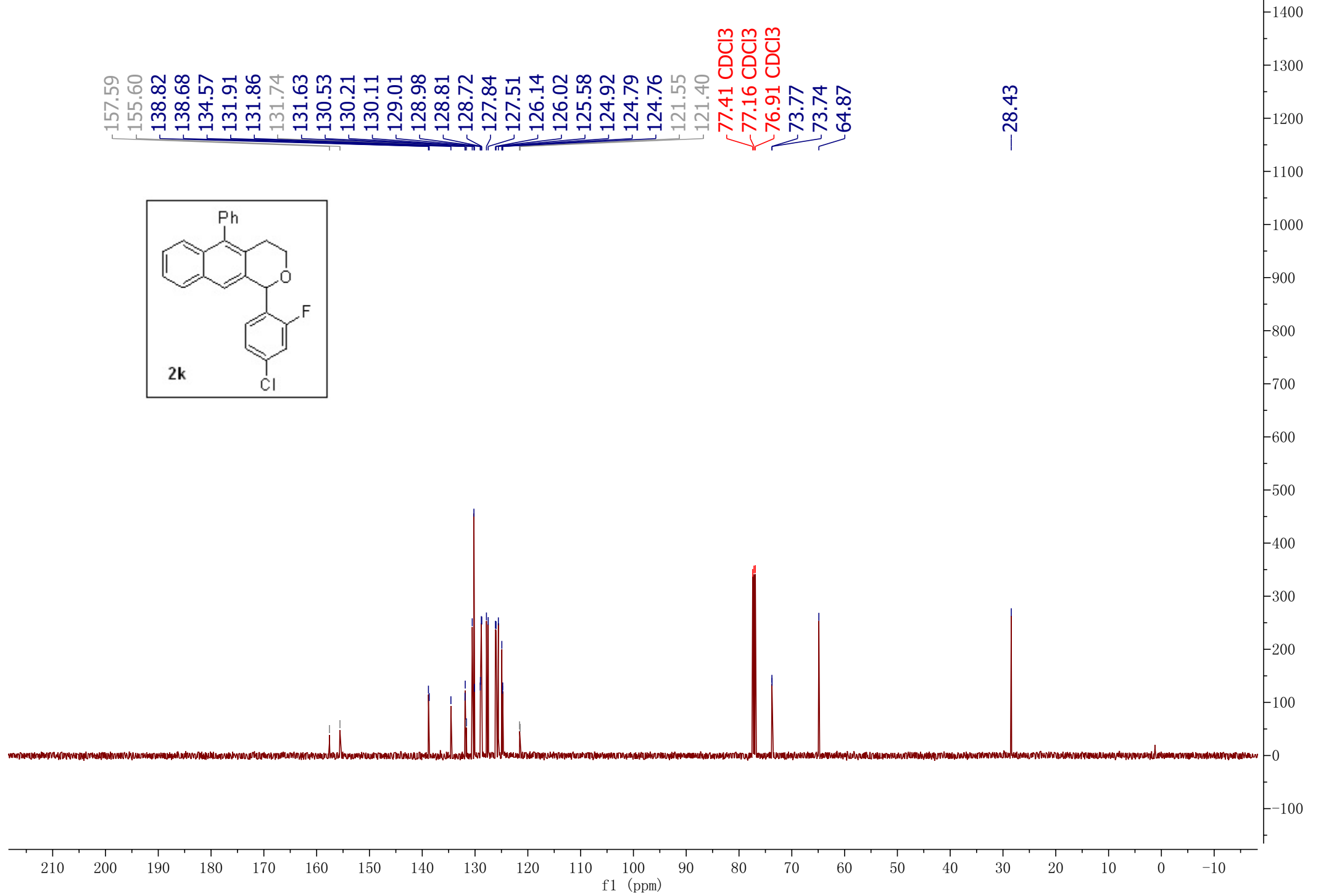
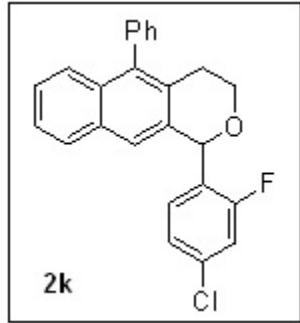
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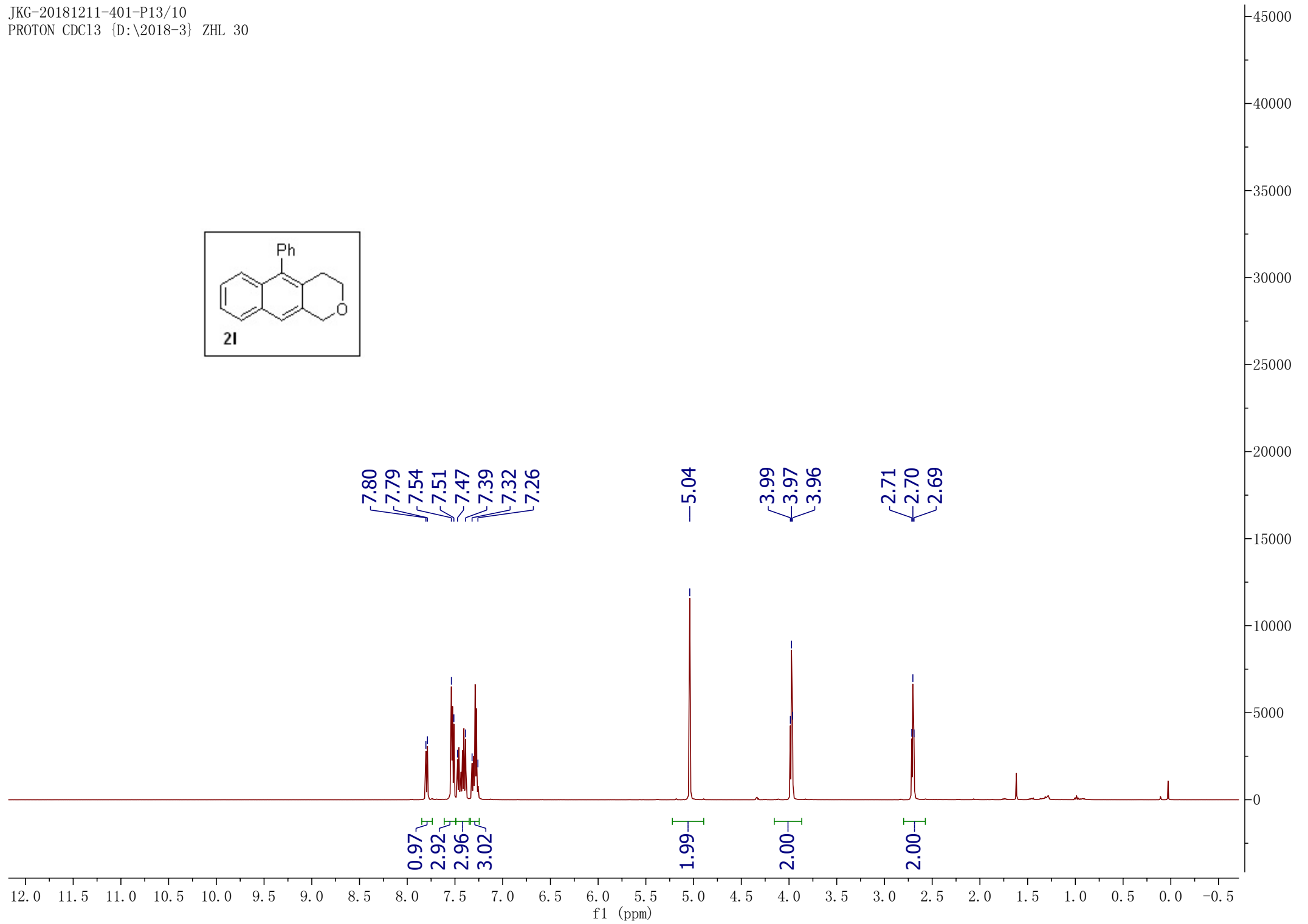
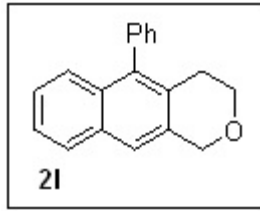


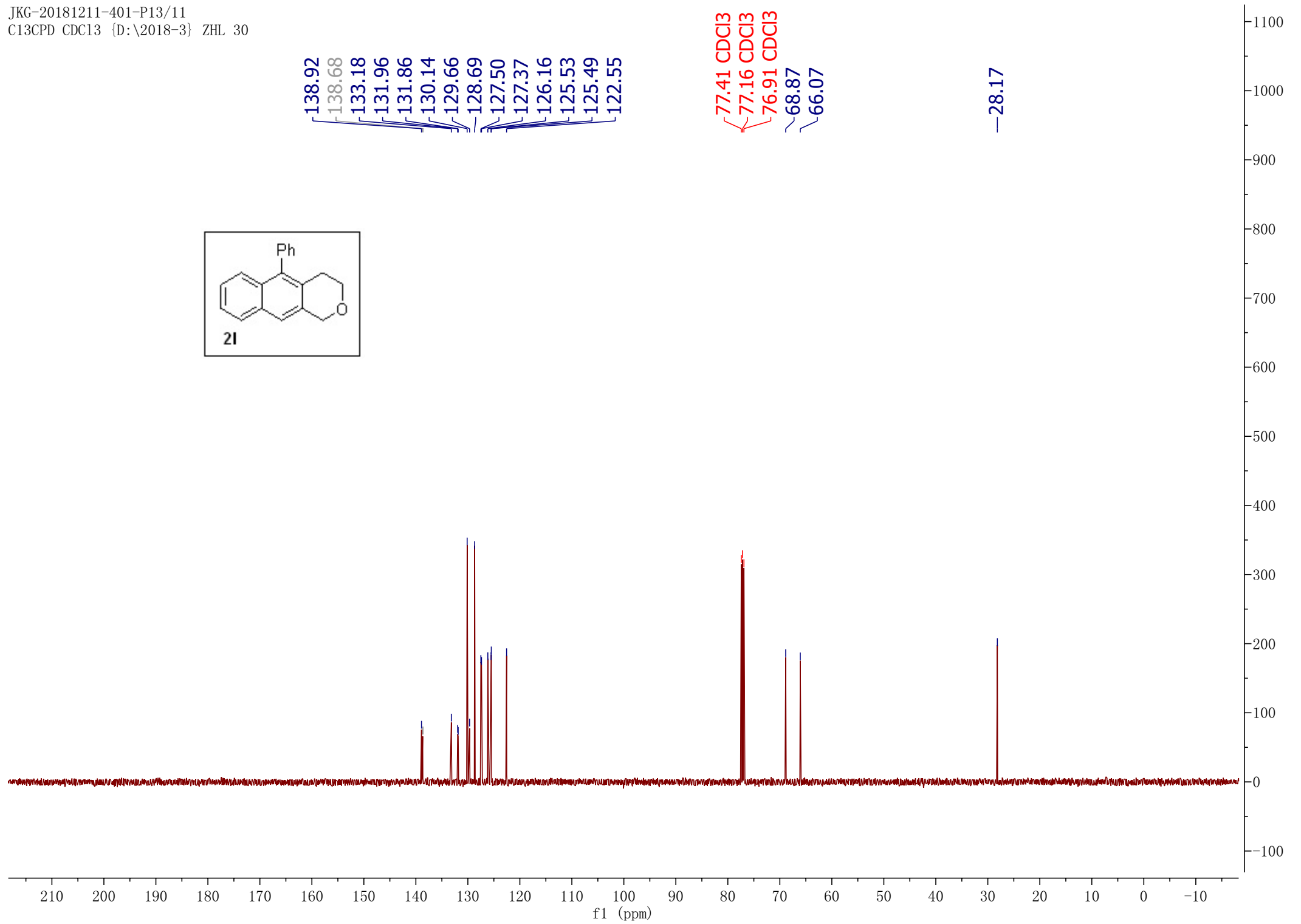
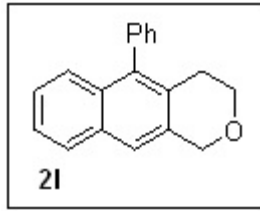
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7.09
7.07
— 6.35

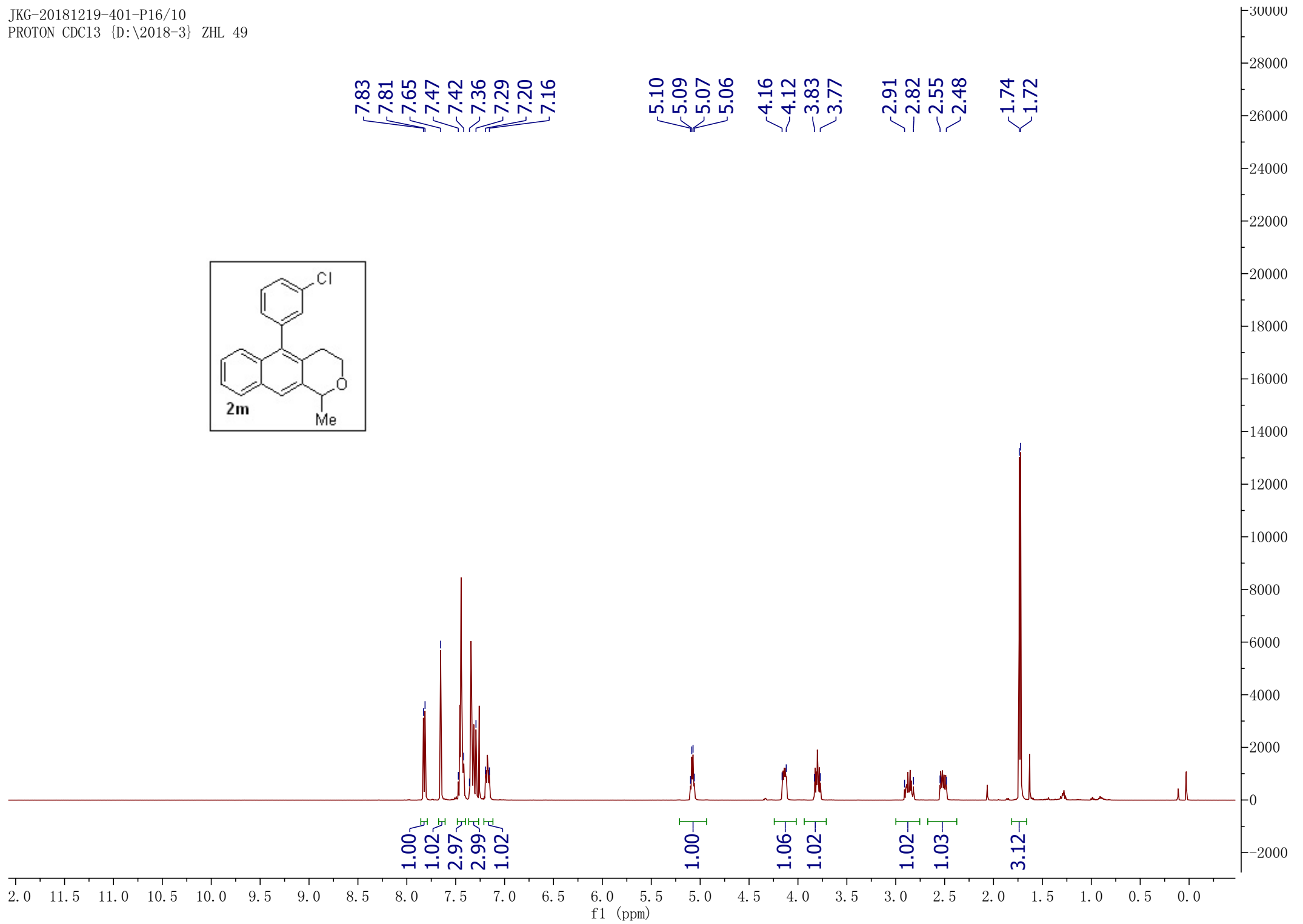
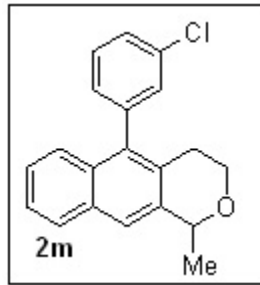
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2.64
2.63

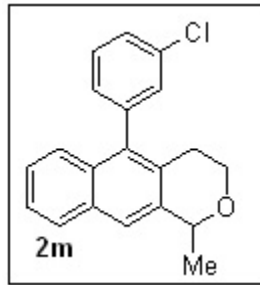








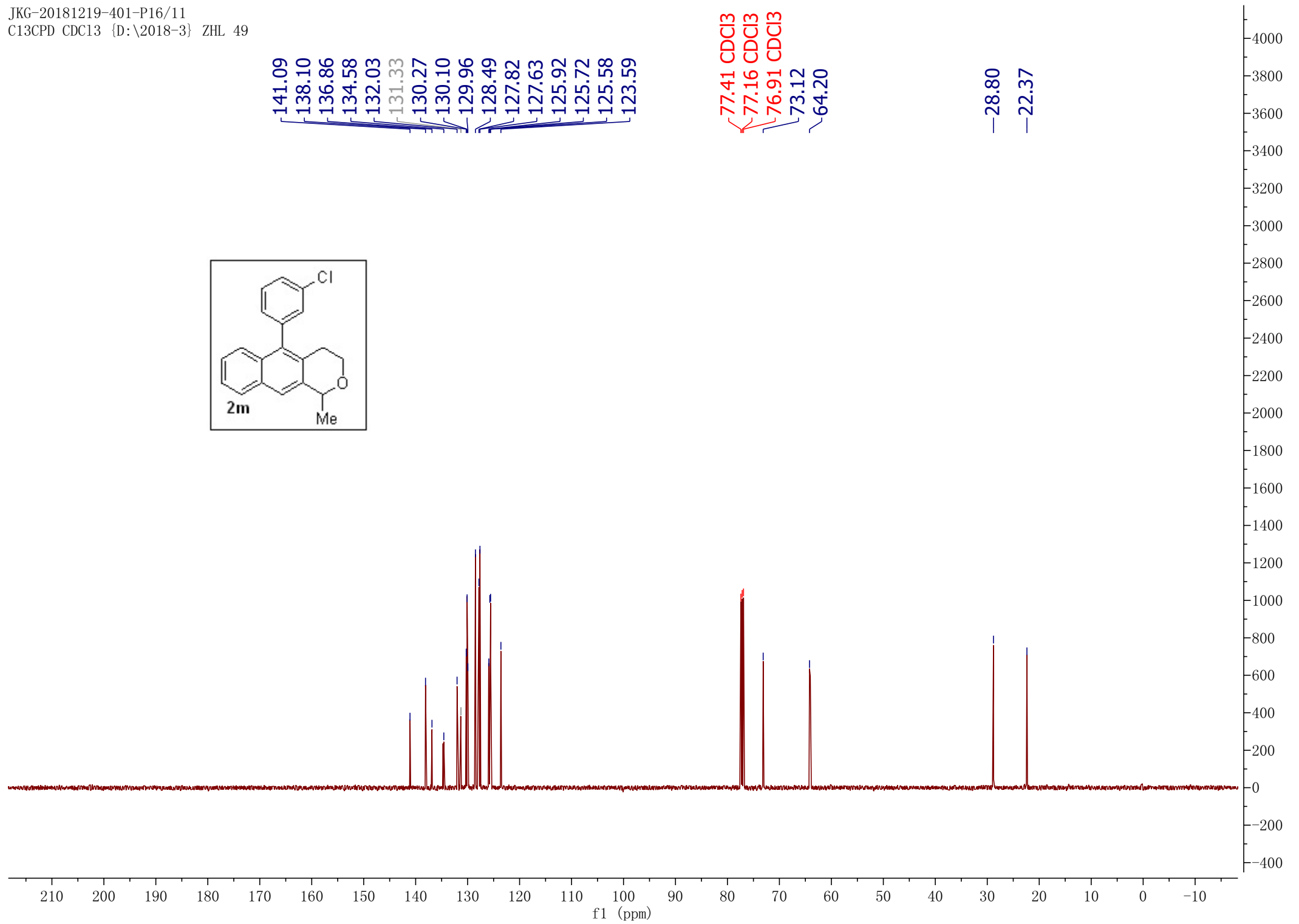


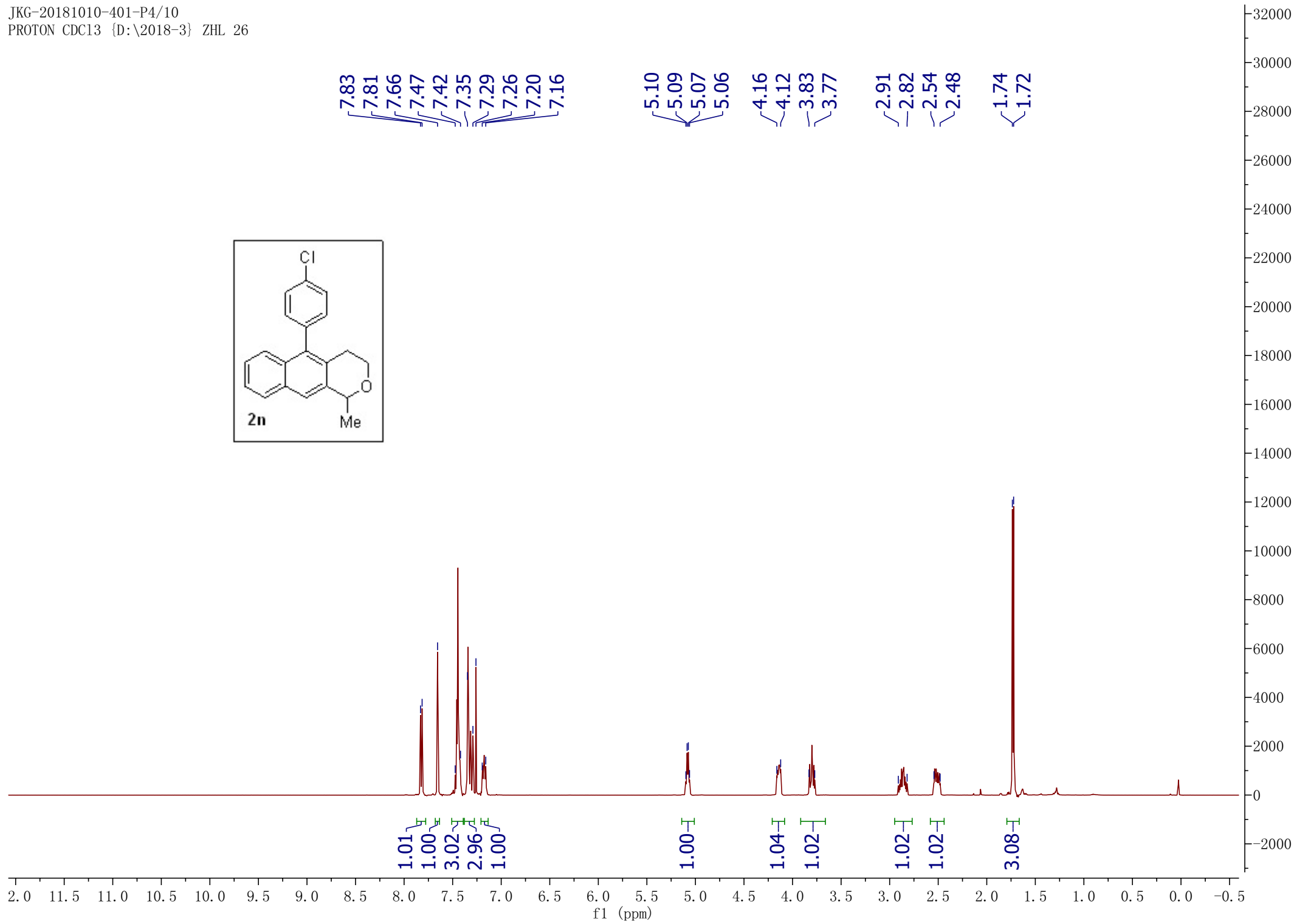
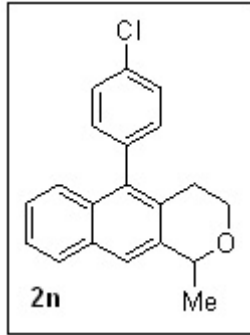


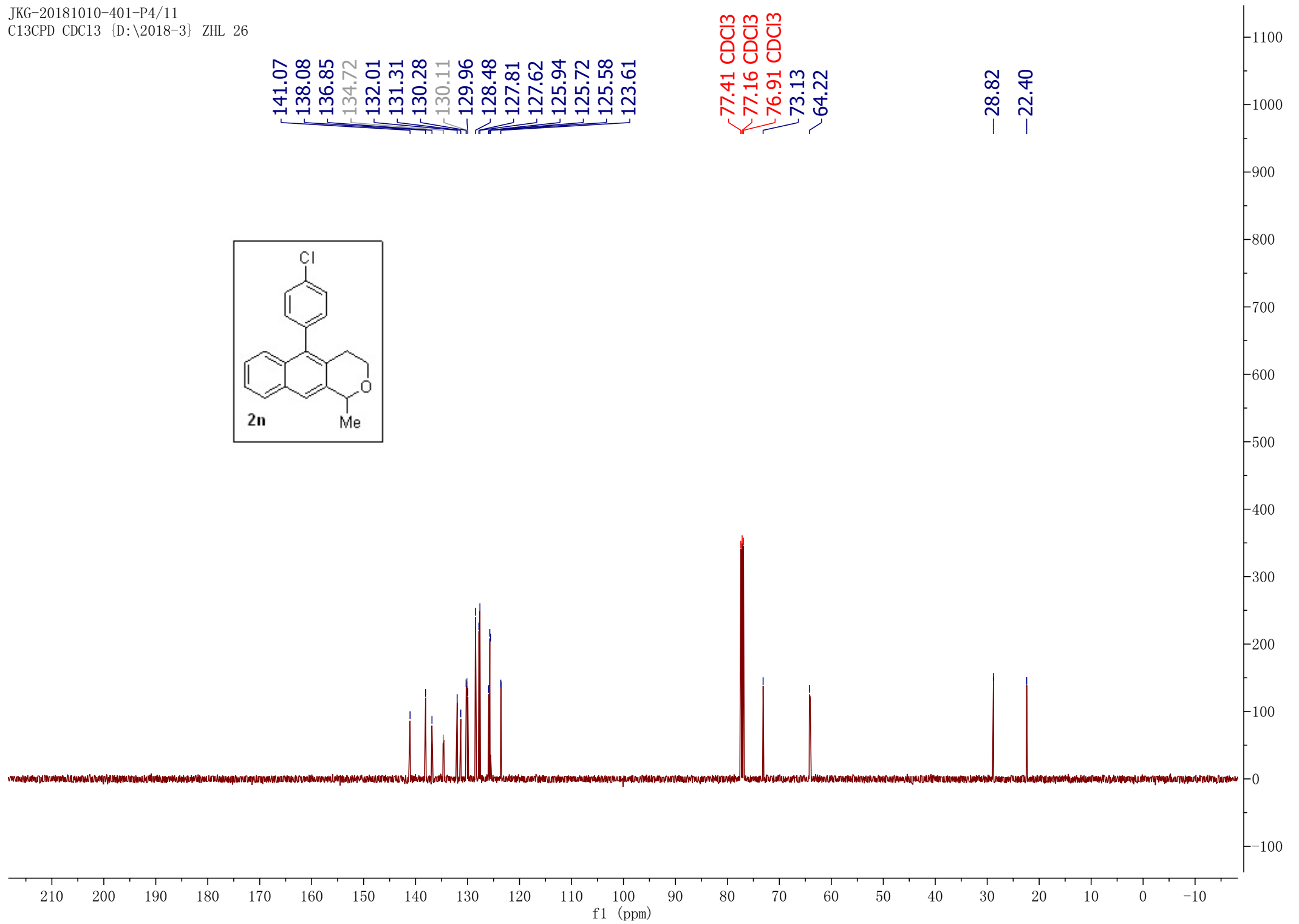
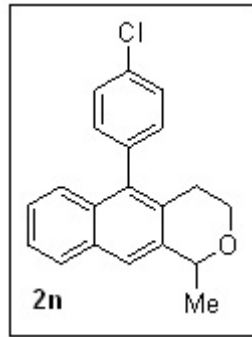
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125.58
123.59

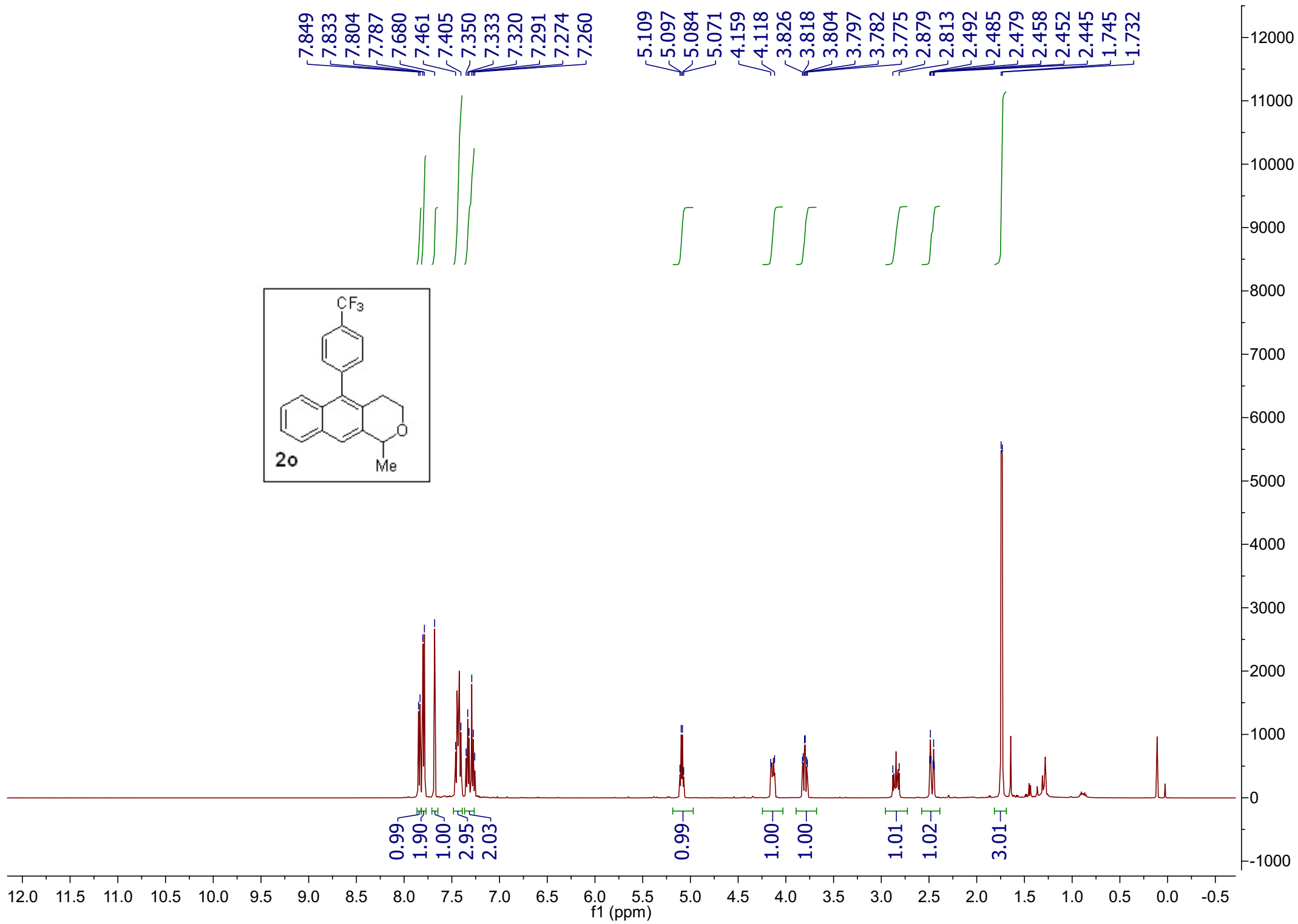
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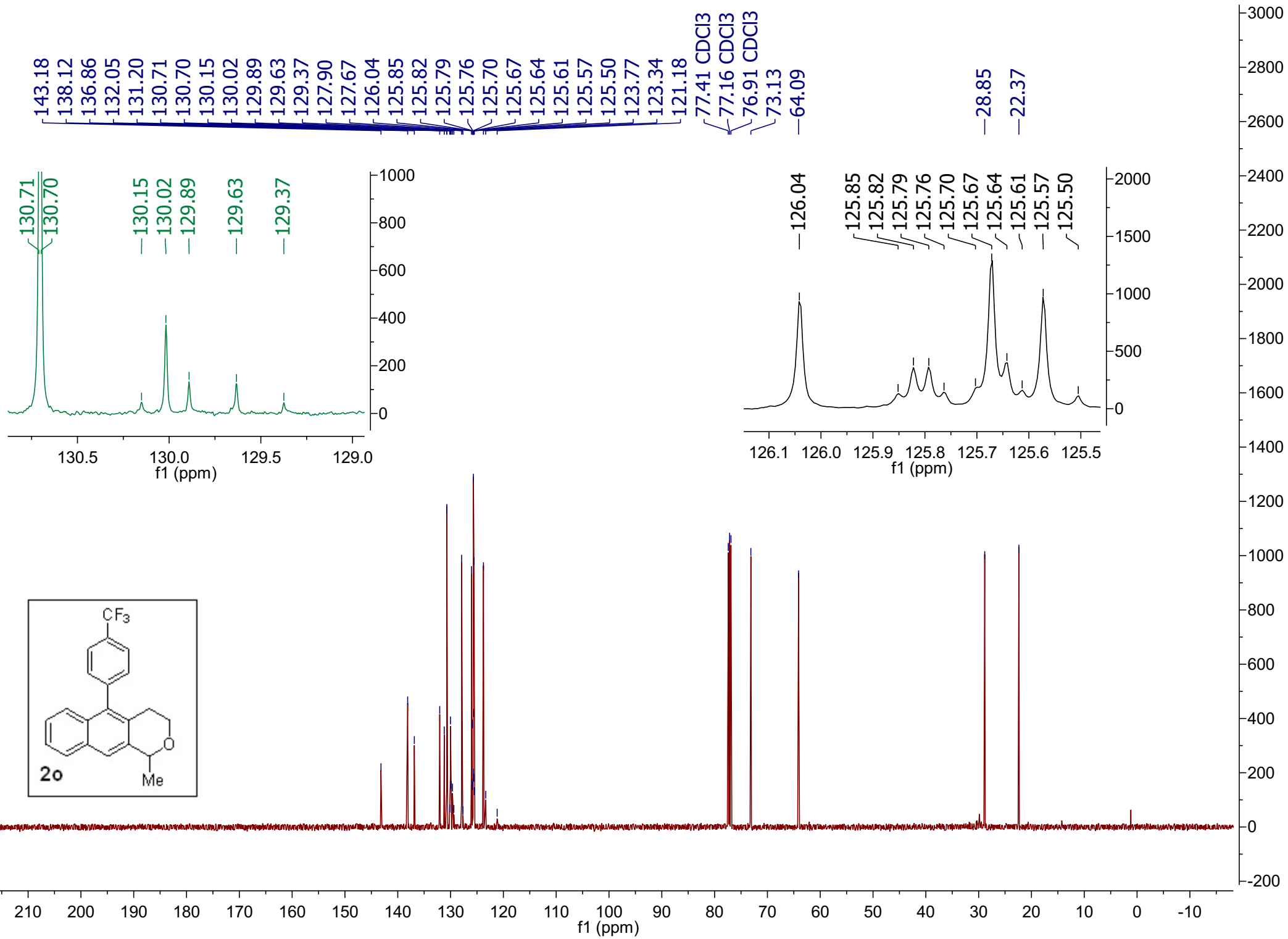
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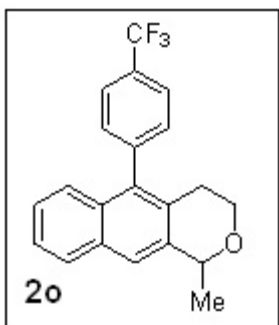




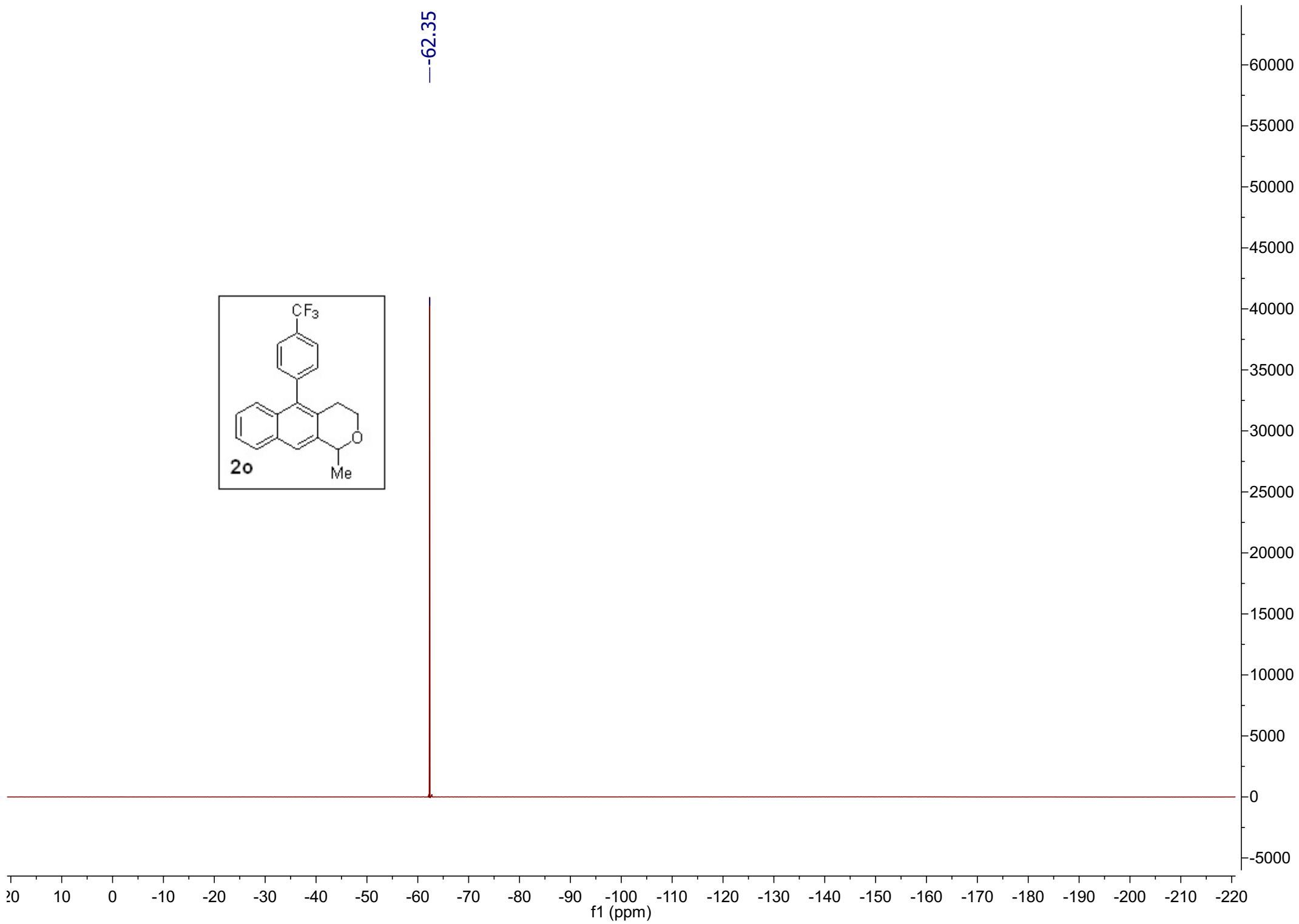


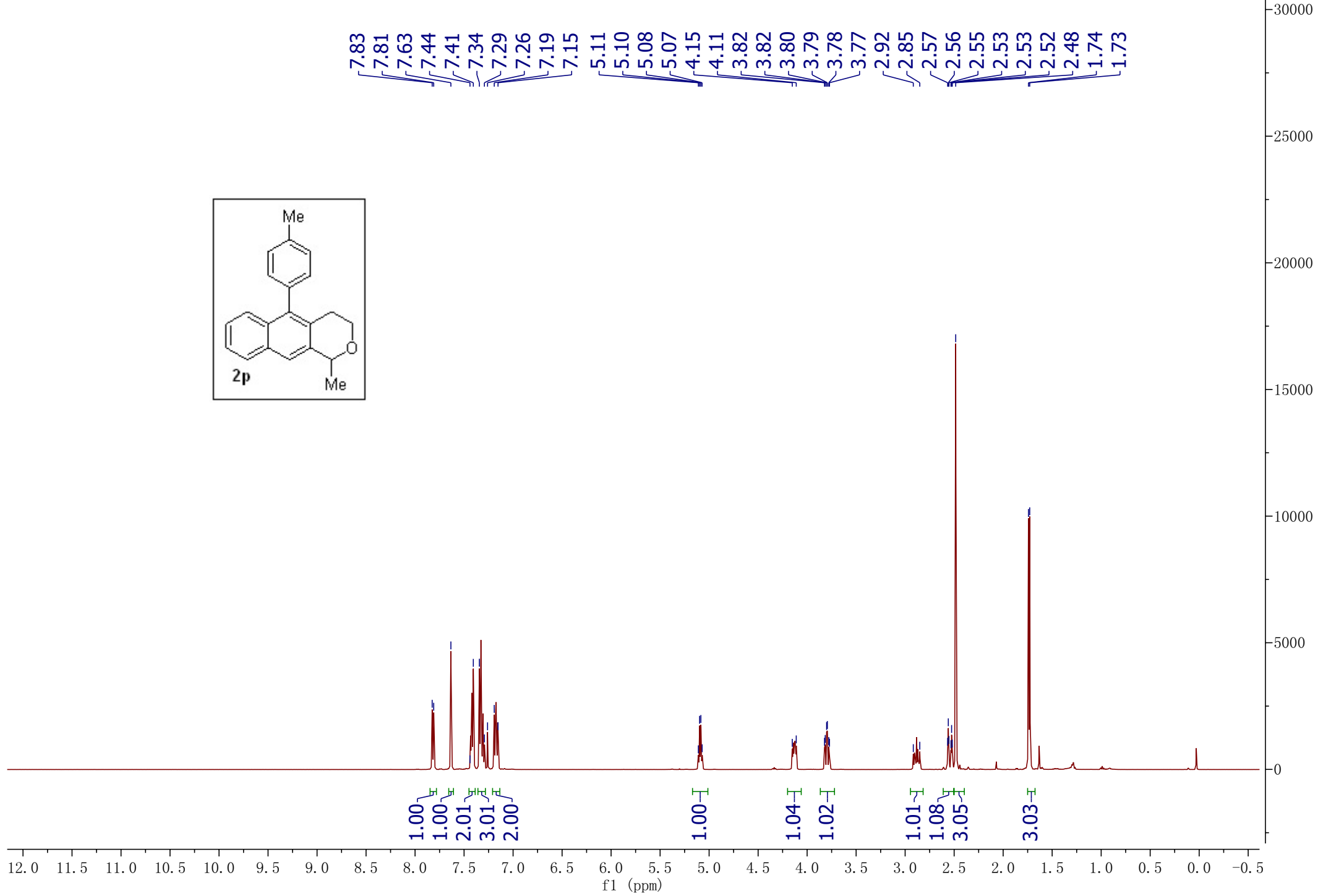
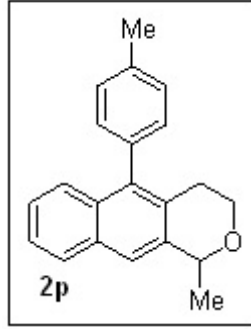


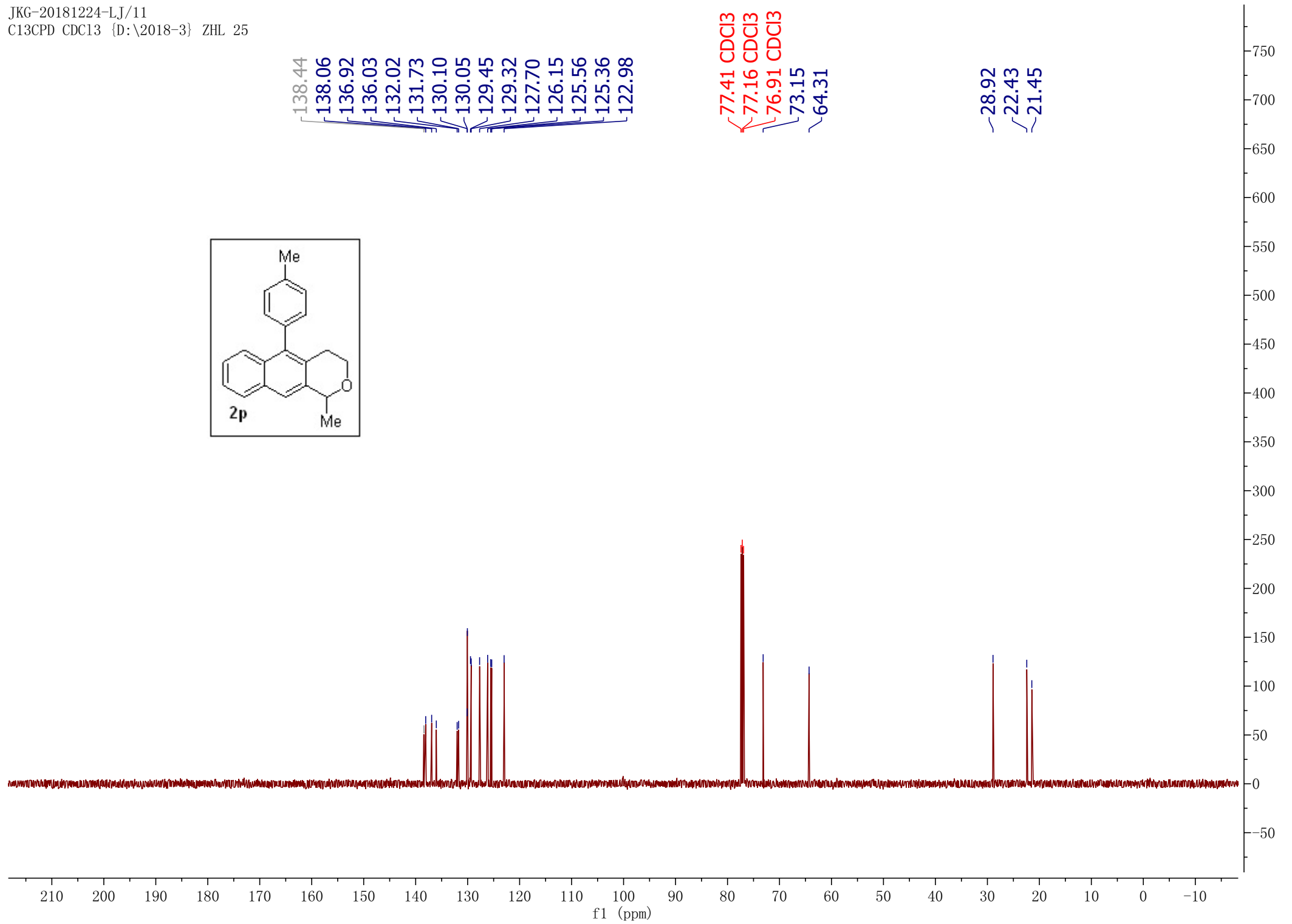
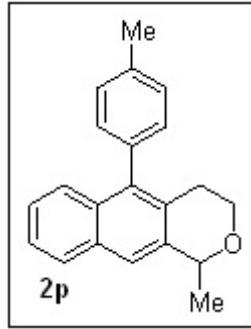


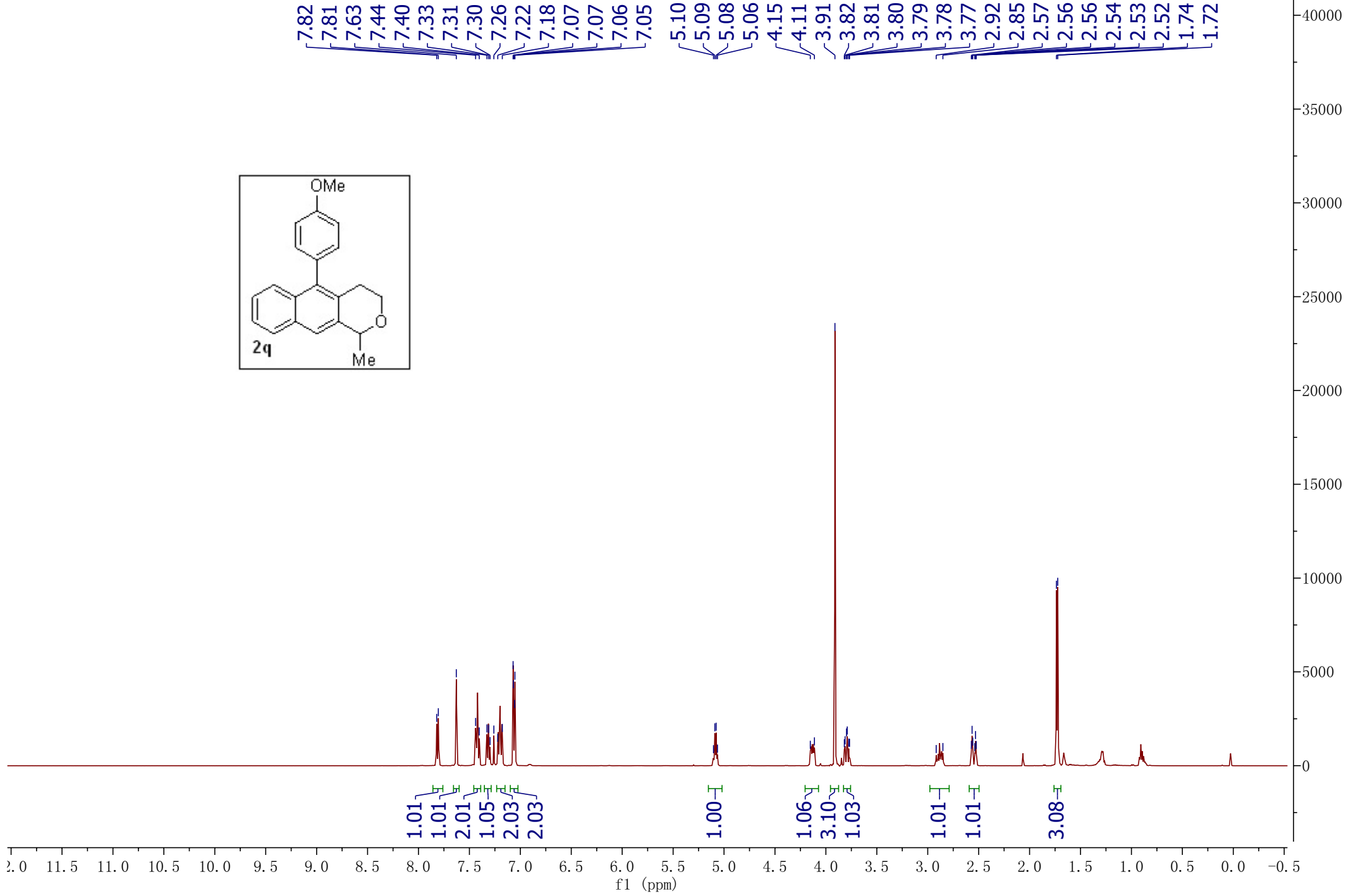
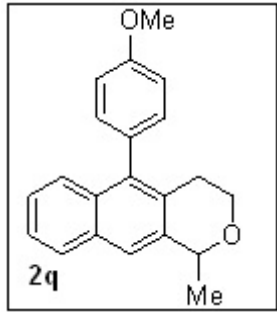


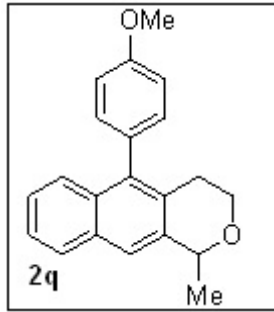
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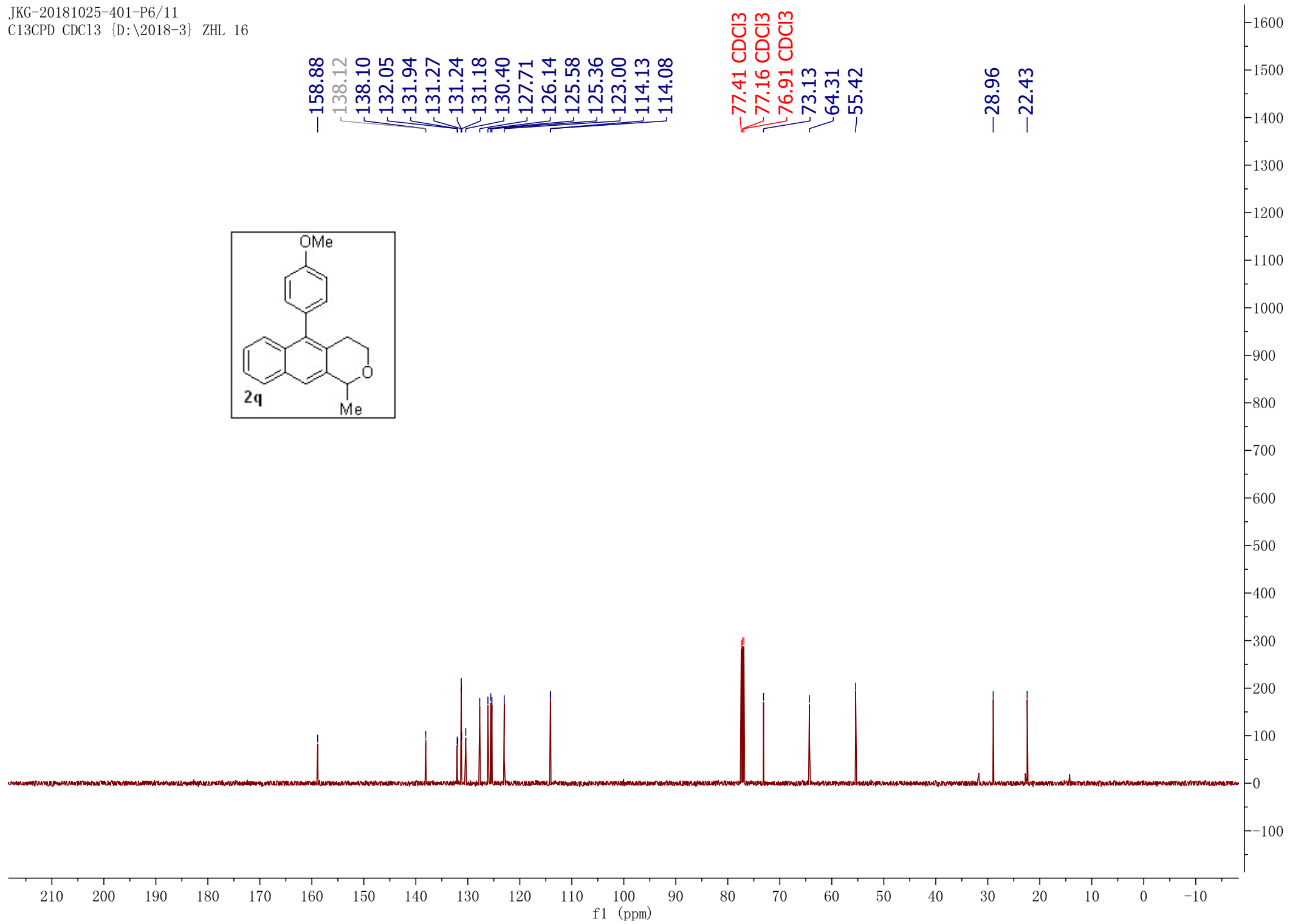


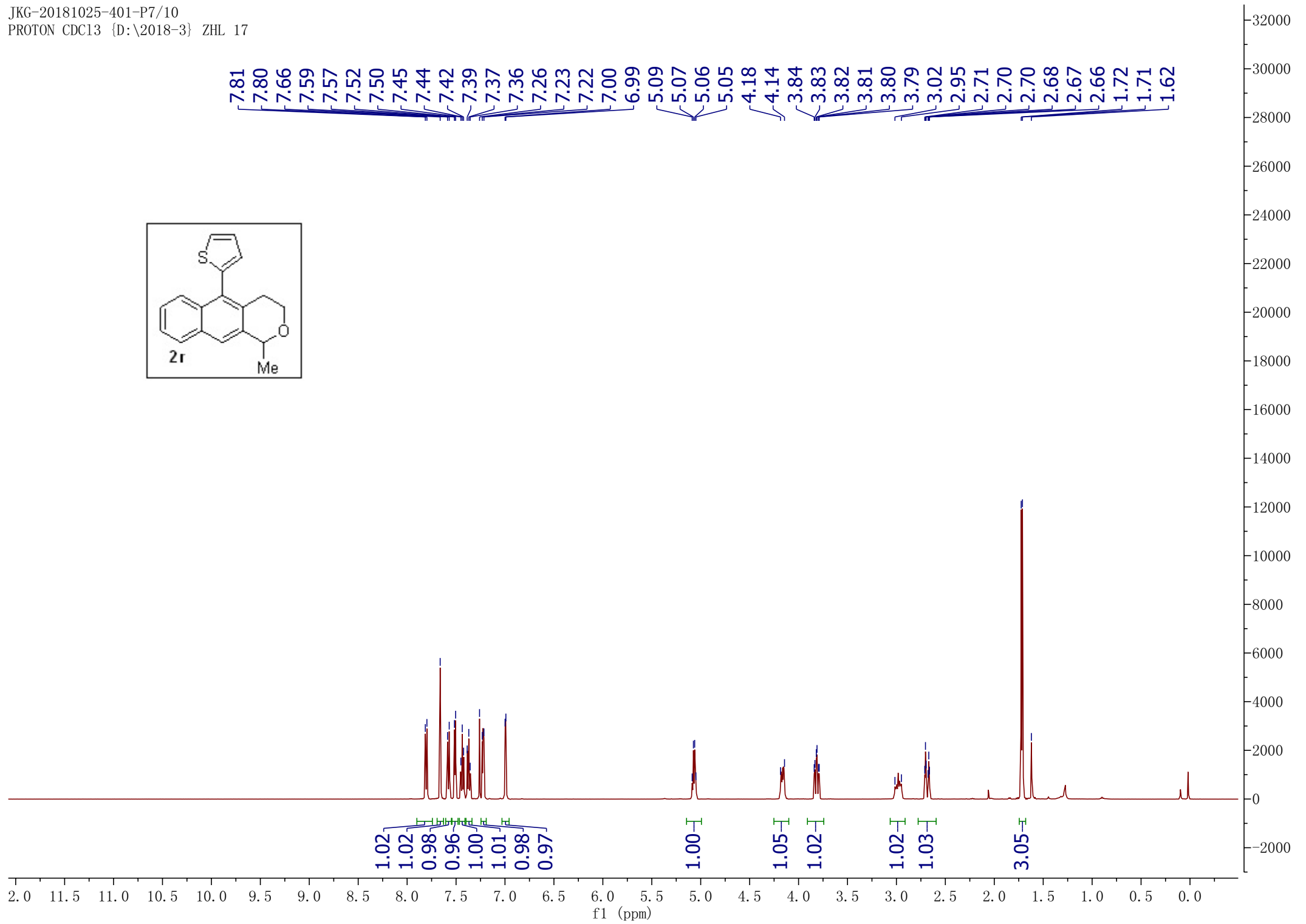
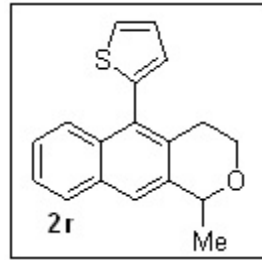


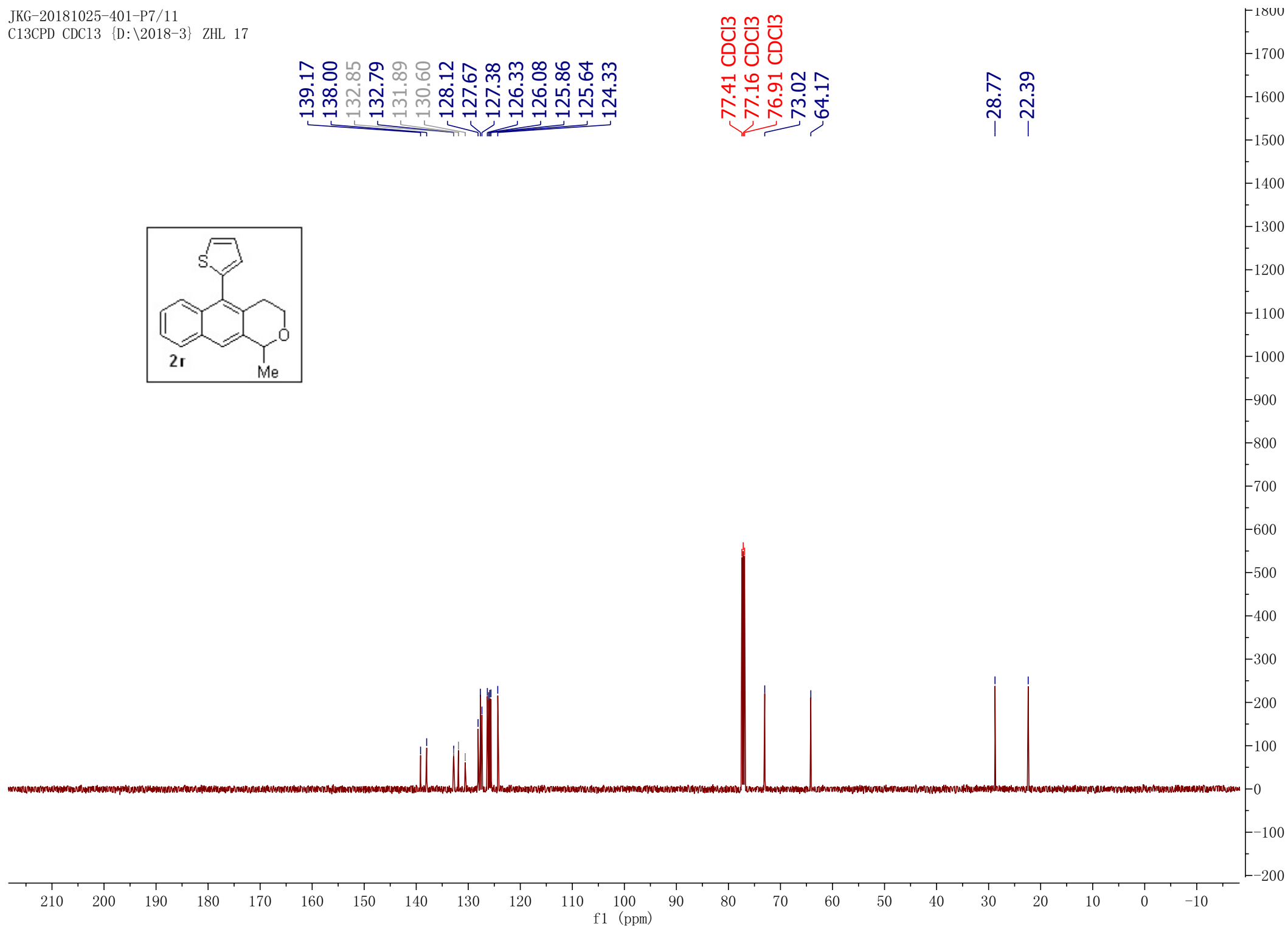
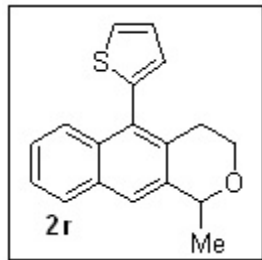
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114.08

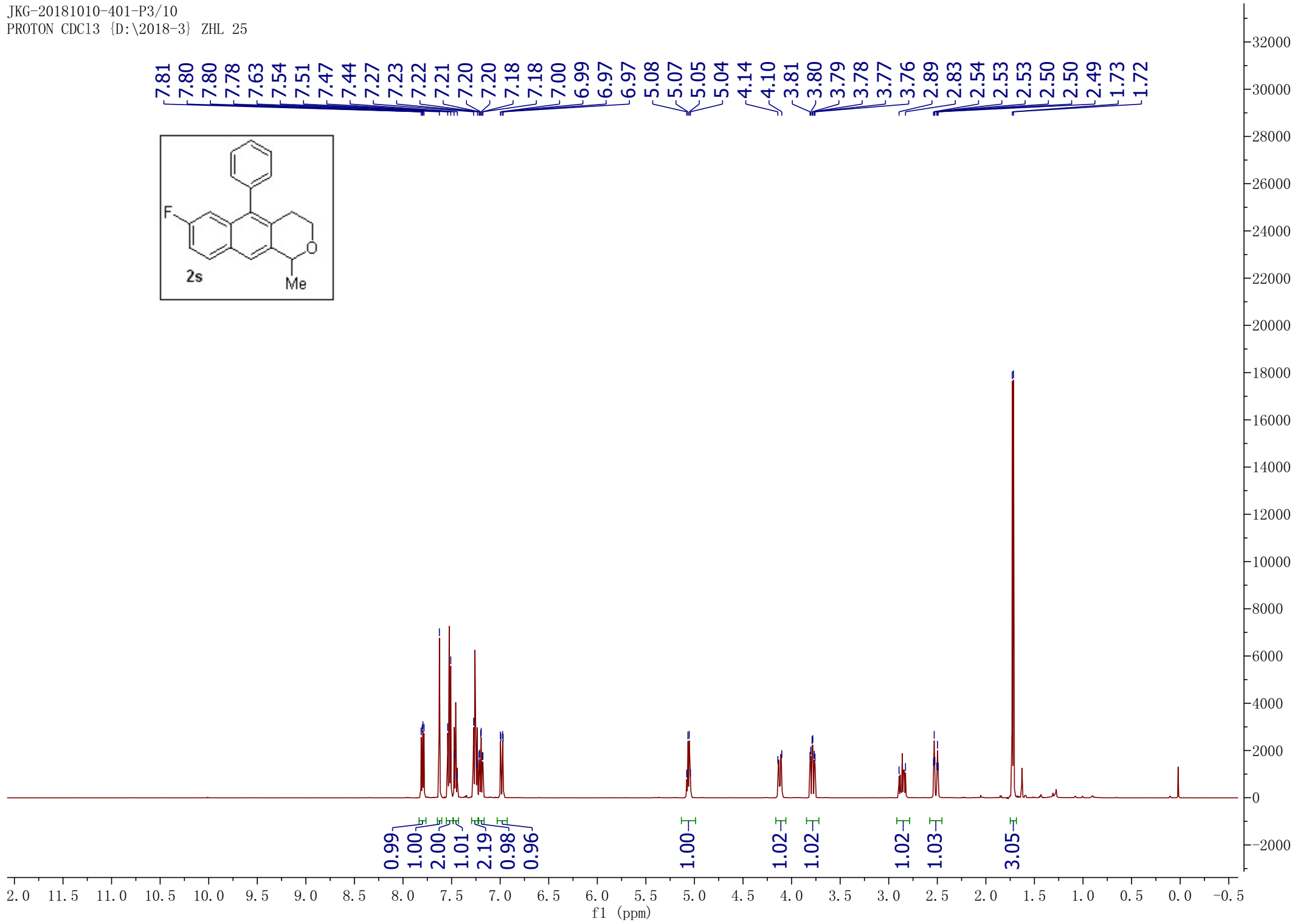
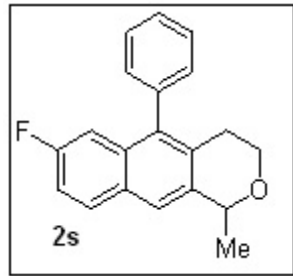
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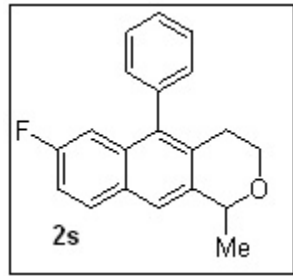
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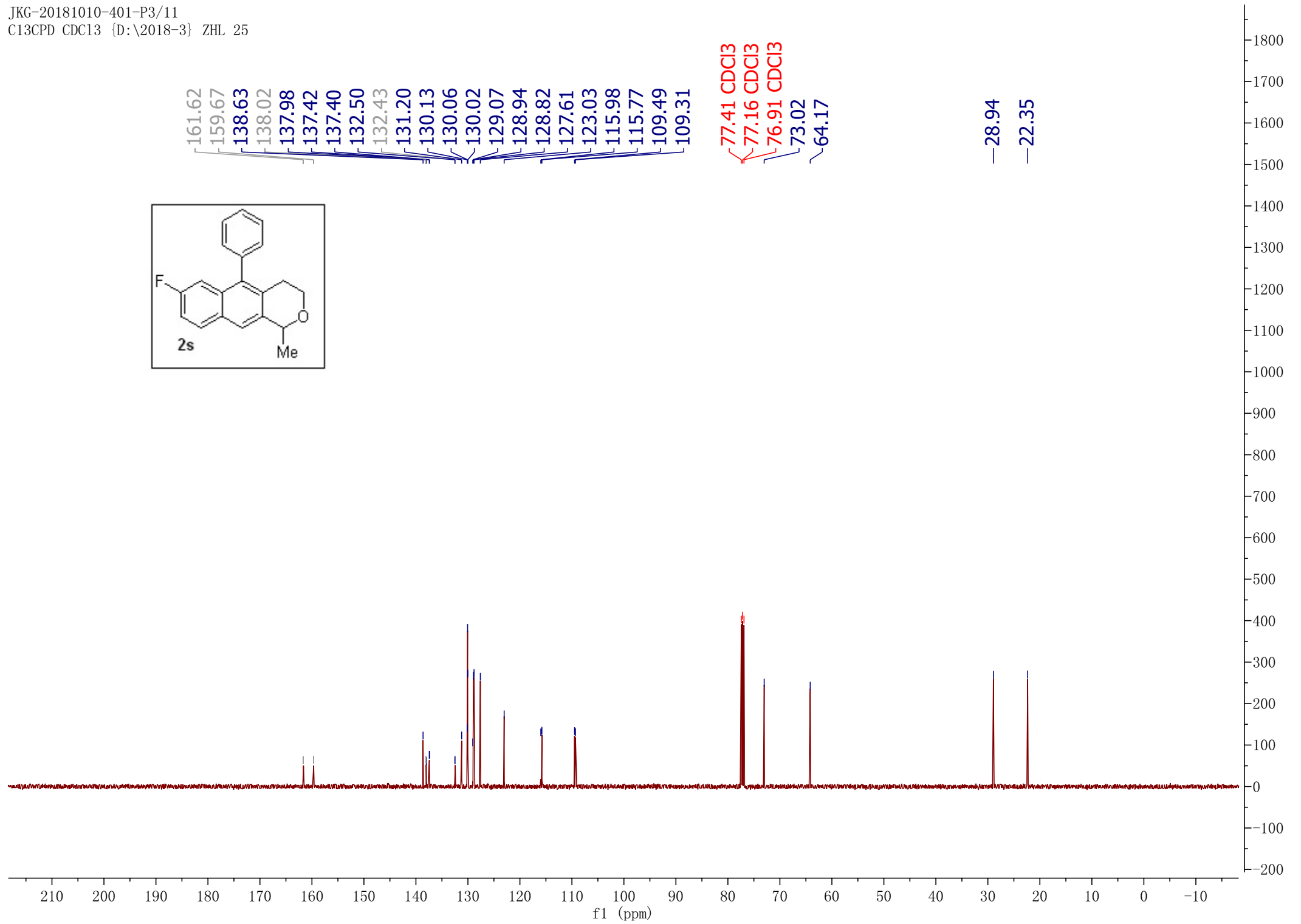


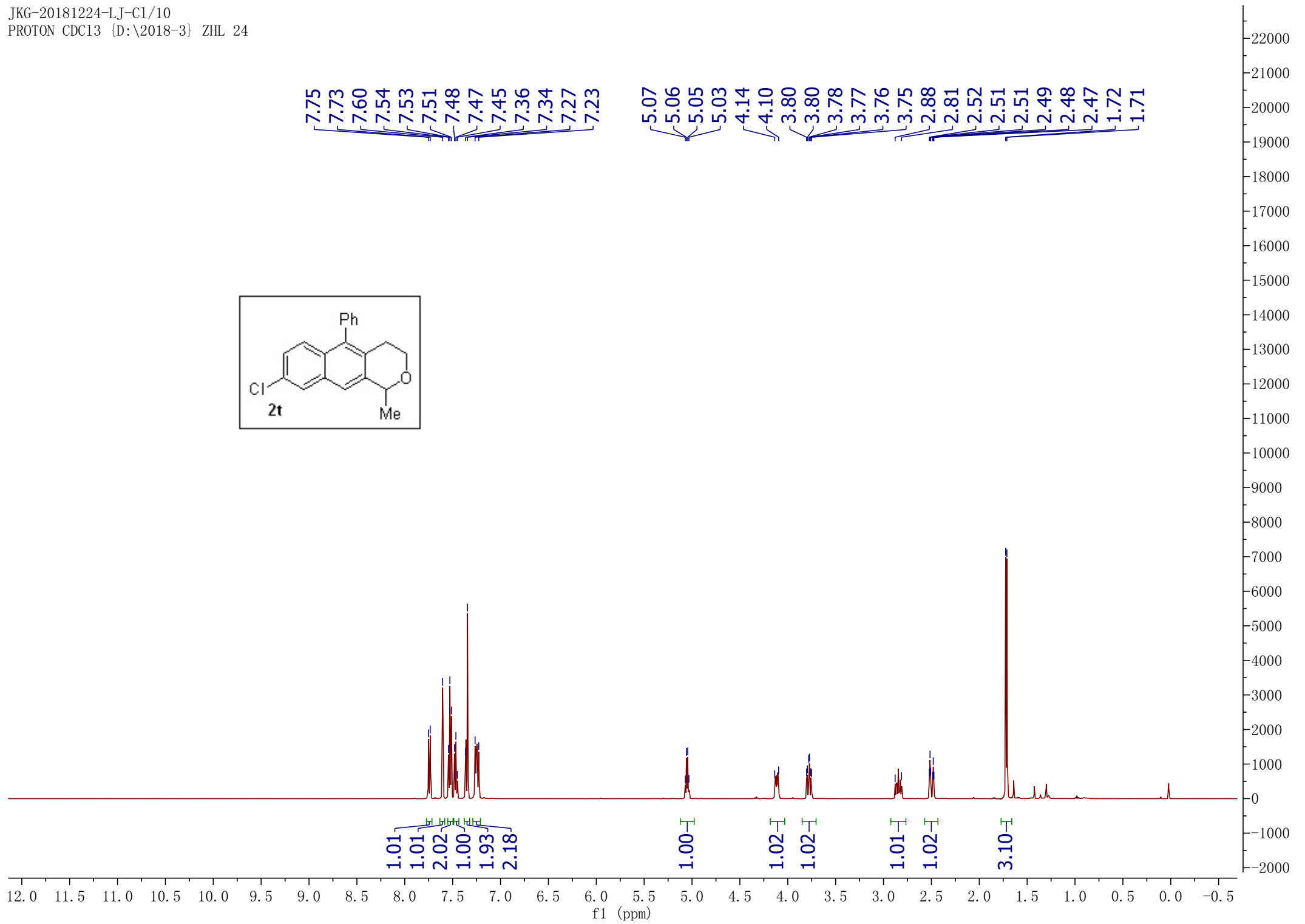
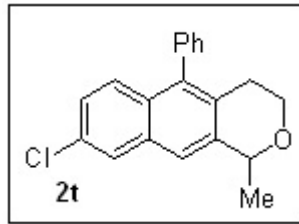


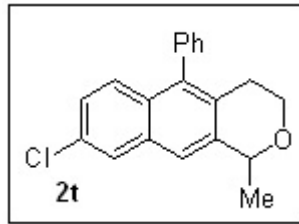
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128.94
128.82
127.61
123.03
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109.31

77.41 CDC13
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76.91 CDC13
73.02
64.17

28.94
22.35



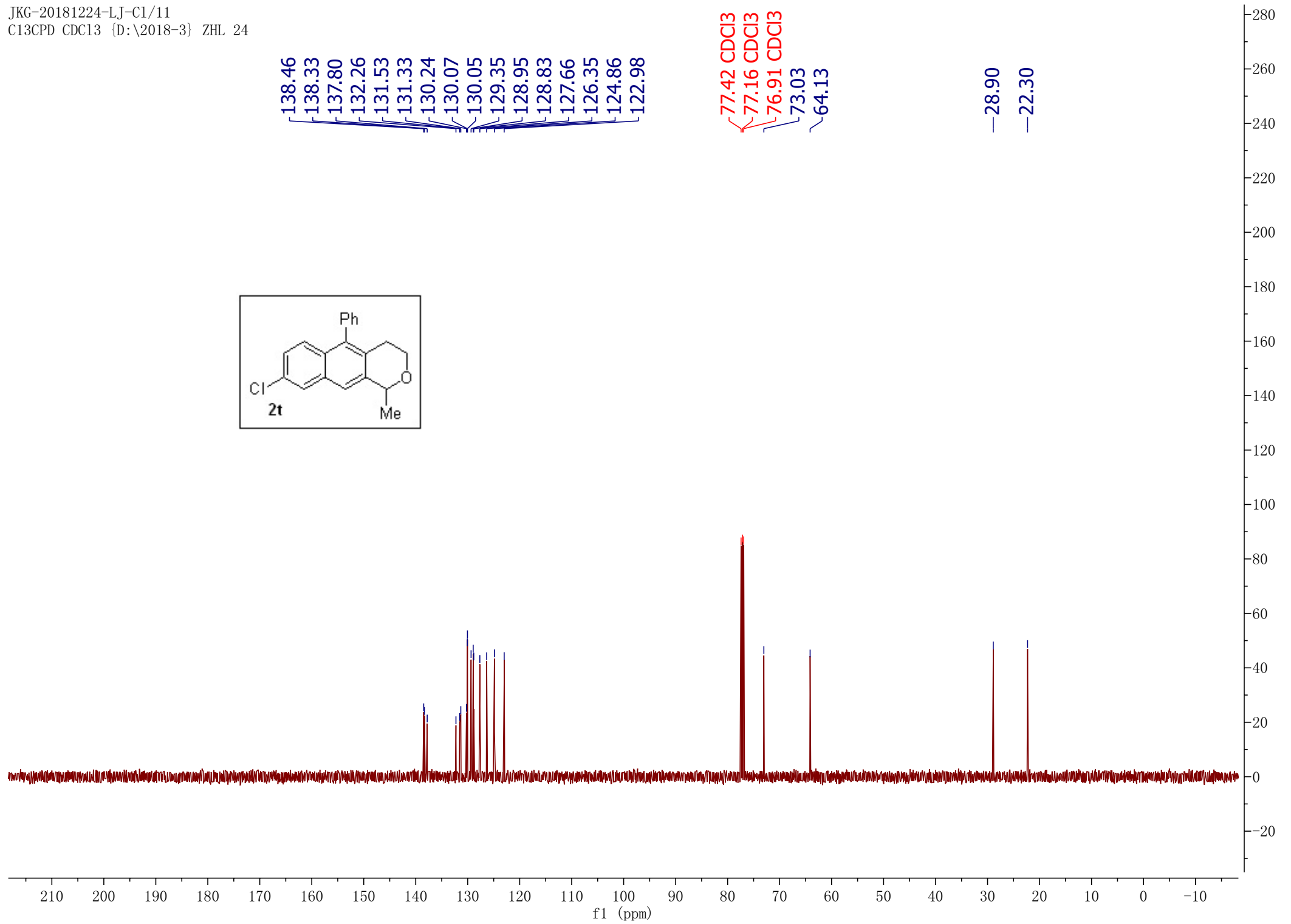


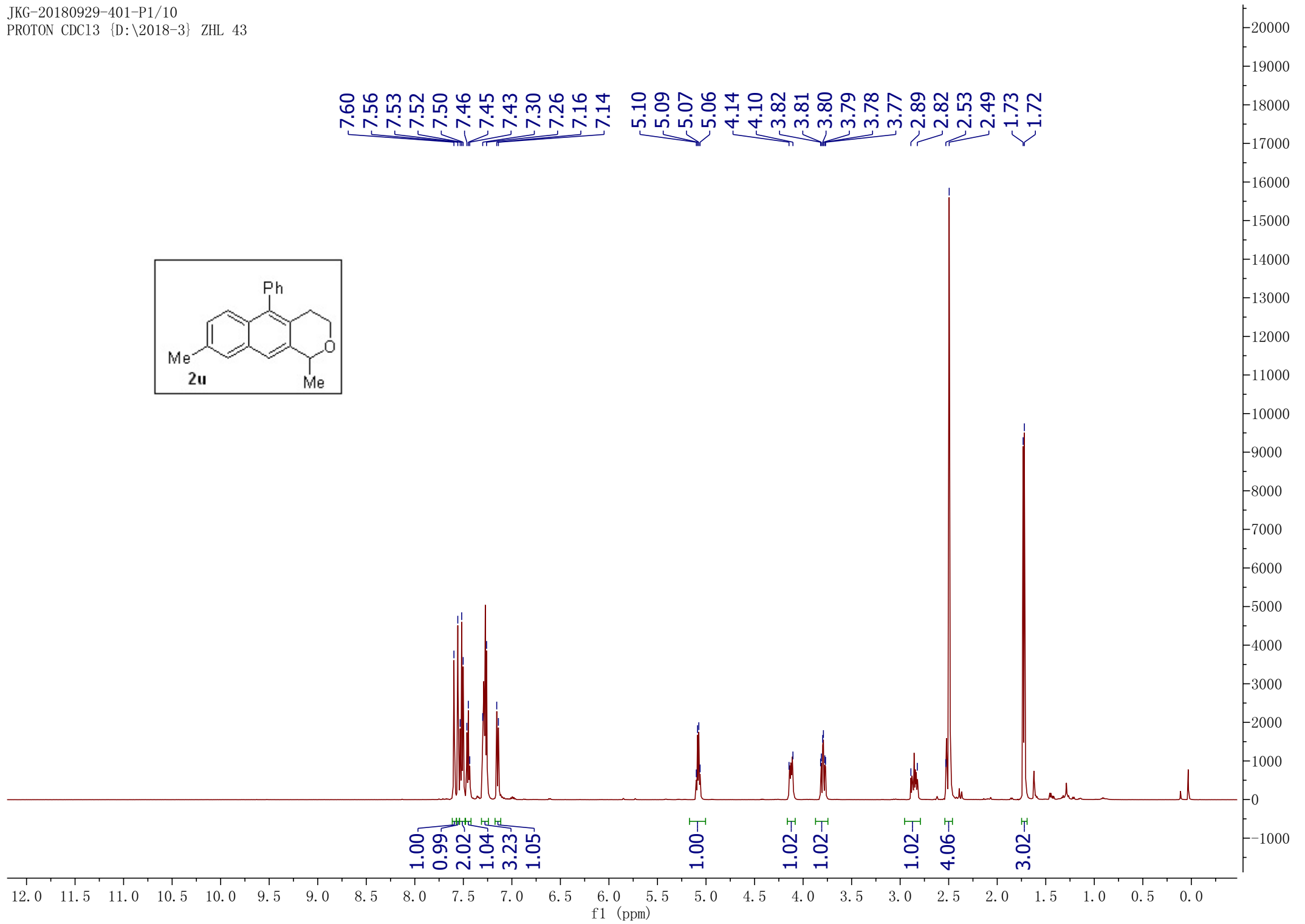
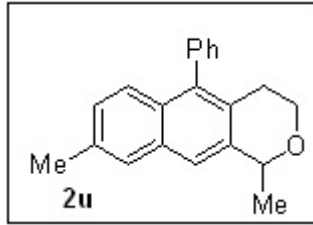


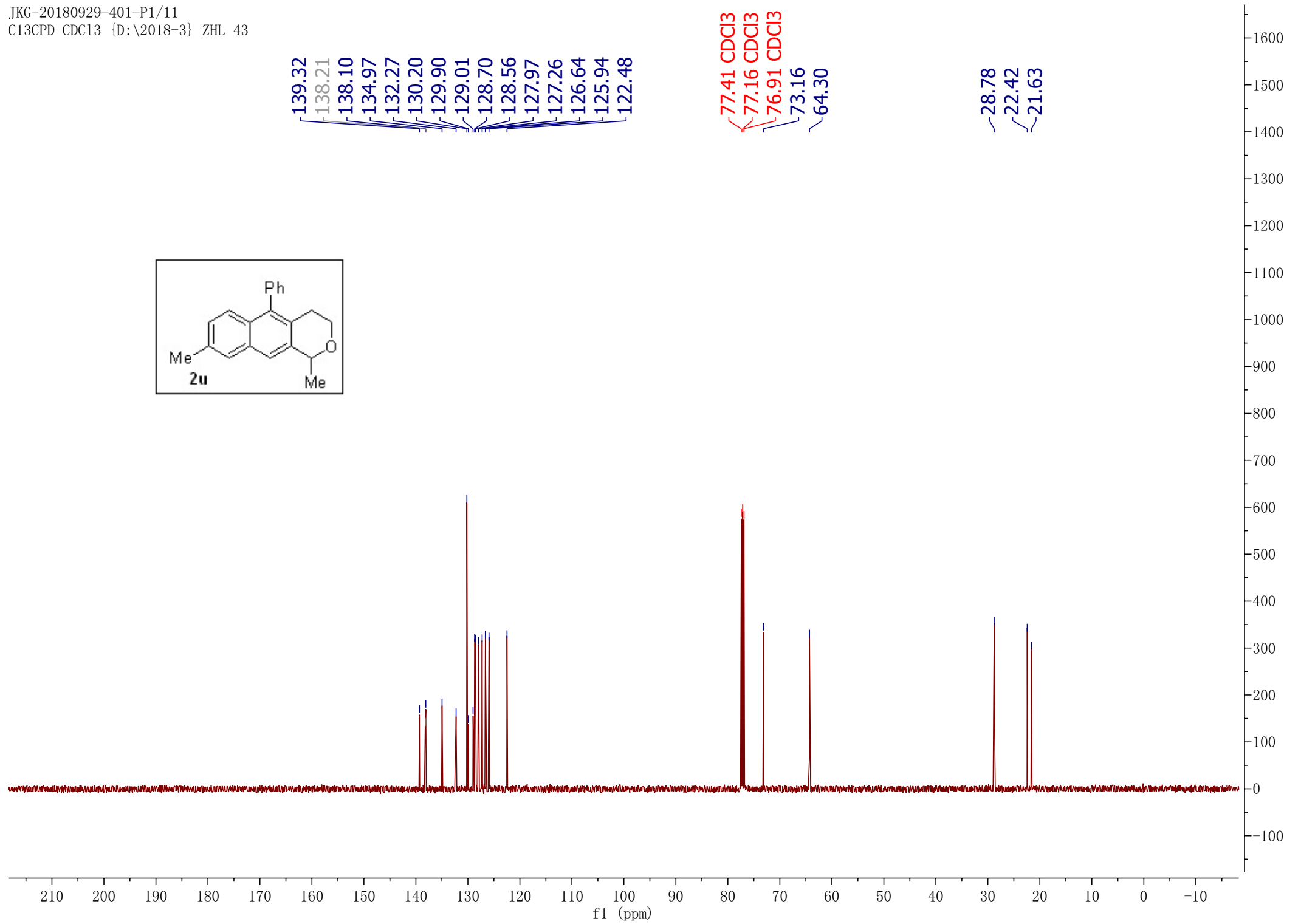
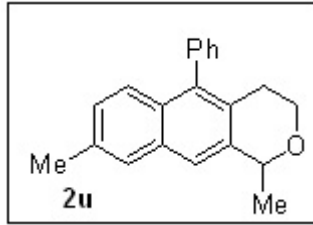
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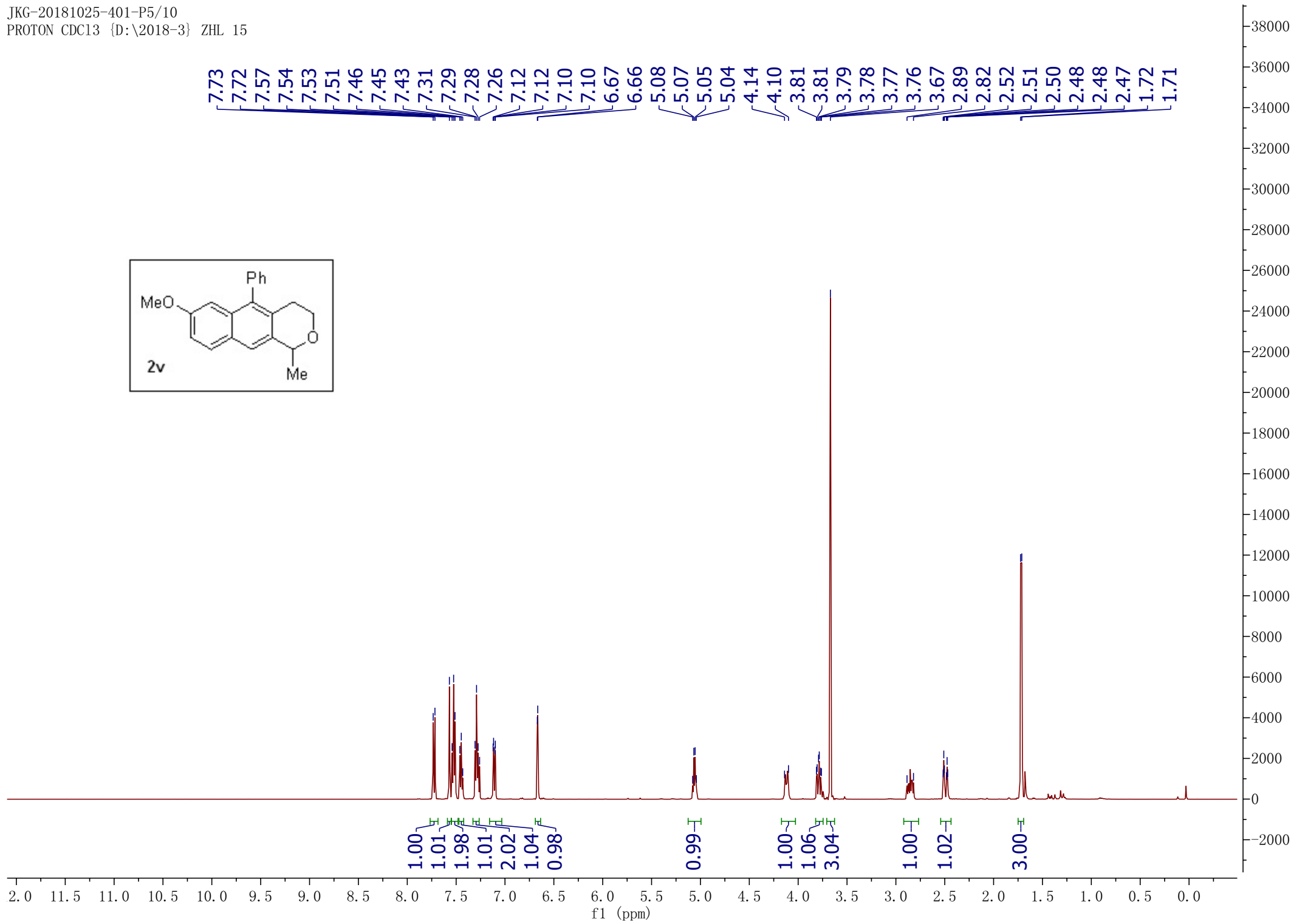
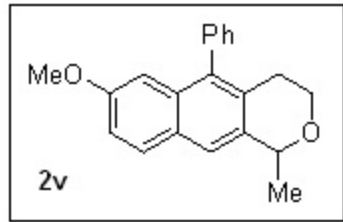
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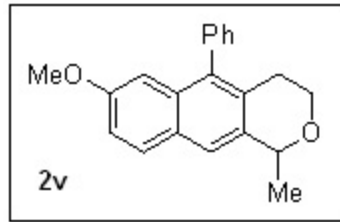
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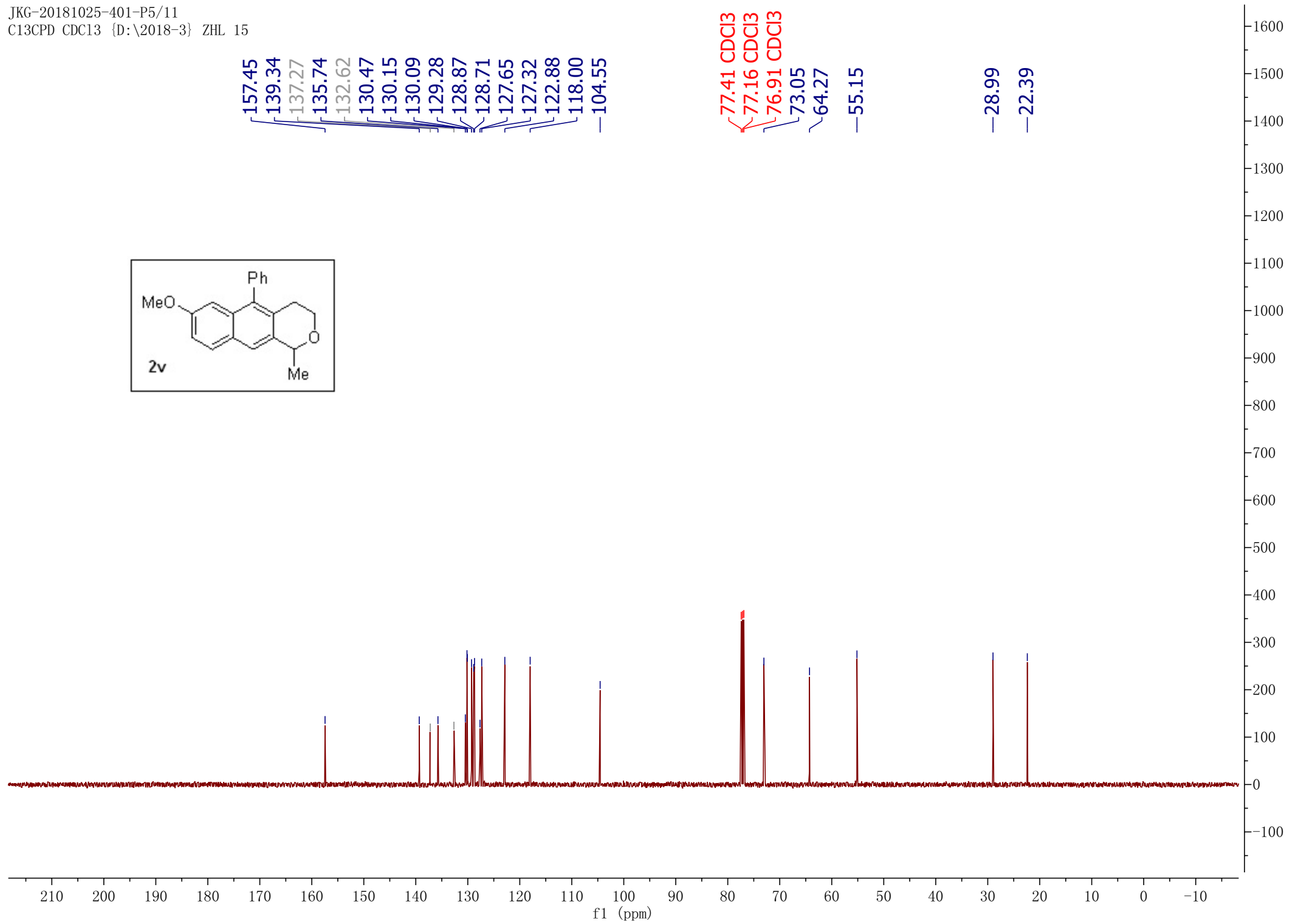


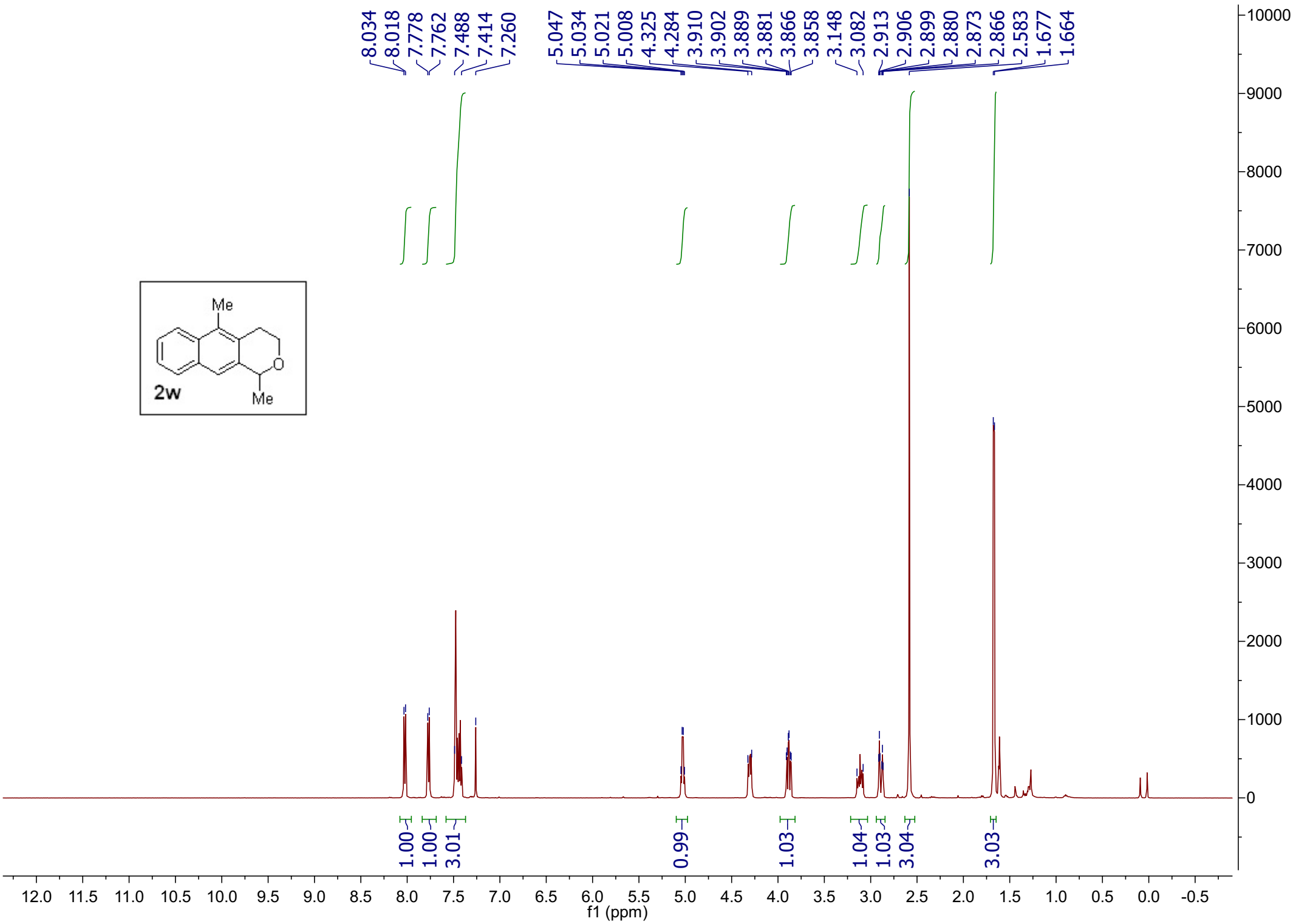
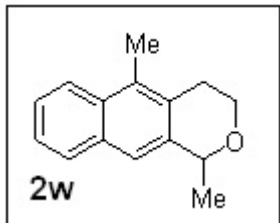


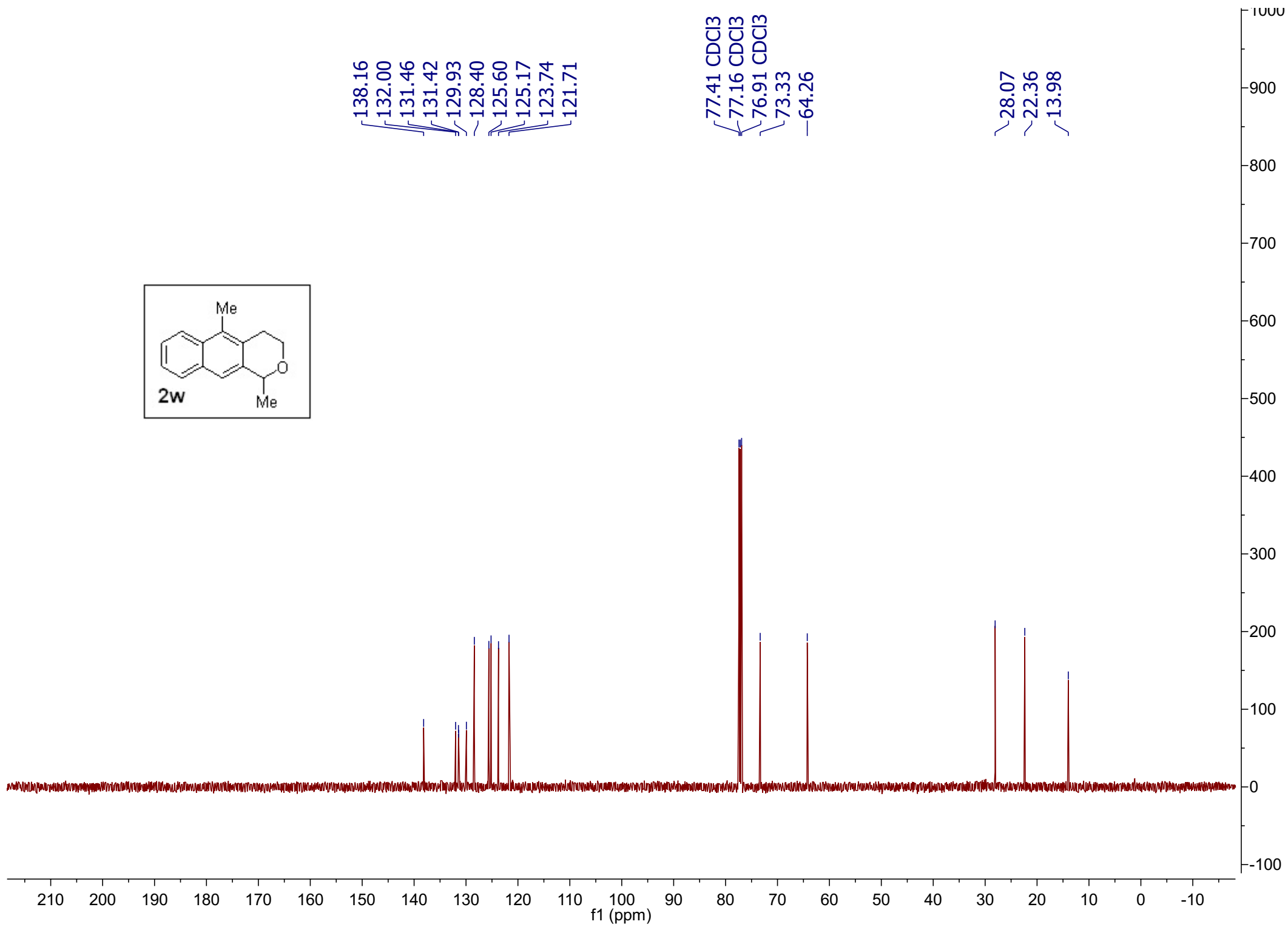
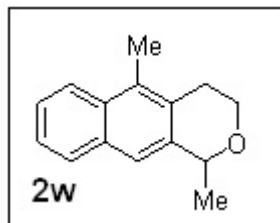
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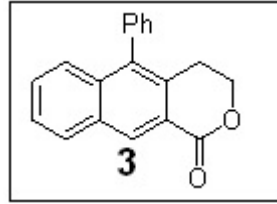
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76.91 CDC13
73.05
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-22.39









8.78
8.01
8.00
7.55
7.47
7.29
7.29
7.28

4.49
4.48
4.47

2.92
2.91
2.90

