Supporting Information

A Direct Route to Ynones: Pd-Catalyzed Sonogashira Carbonylative Coupling of Ethers with Alkynes via C–O Cleavage

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Contents

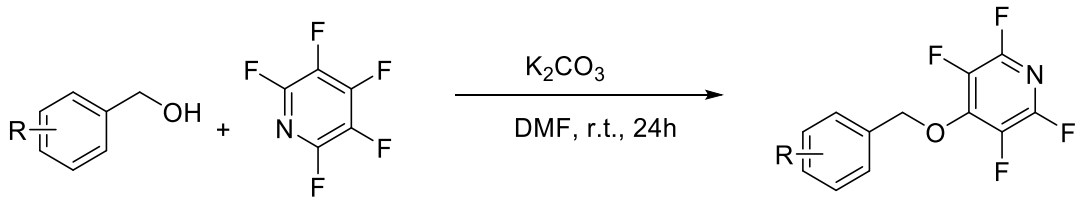
1. General comments

Chemicals were purchased from Energy Chemical, Adamas, Bidepharm., TCI, Aladdin and used as such unless stated otherwise. All solvents like acetonitrile, tetrahydrofuran, N, N-dimethylmethanamide, 1,4-dioxane were purchased from Adamas (Water ≤ 30 ppm (by K.F.), 99.9%, SafeDry, with molecular sieves, Safeseal). NMR spectra were recorded on Bruker AV 400 or Bruker Fourier 300 spectrometer. Chemical shifts (ppm) are given relative to TMS (0.00 ppm) for ^{1}H and $CDCl_3$ (77.0 ppm). Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublet), m (multiplet) and br.s (broad singlet). High-resolution mass spectra HRMS spectra were recorded on a Thermo Scientific Exactive Orbitrap Mass Spectrometer under Electron Spray Ionization conditions preparing sample solution in methanol. The data are given as mass units per charge (m/z). GC yields were calculated using dodecane as an internal standard. Gas chromatography analysis was performed on an Agilent 6820 instrument with an FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d. 0.25 μm film thickness) using nitrogen as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel., 54-74 μm, 200-300 mesh (Yucheng Chemical CO., LTD, Shanghai).

NOTE: As carbon monoxide will be released from reaction system, the reactions should only be handled in a well-ventilated fume hood and the laboratory should be well-equipped with a CO detector and alarm system.

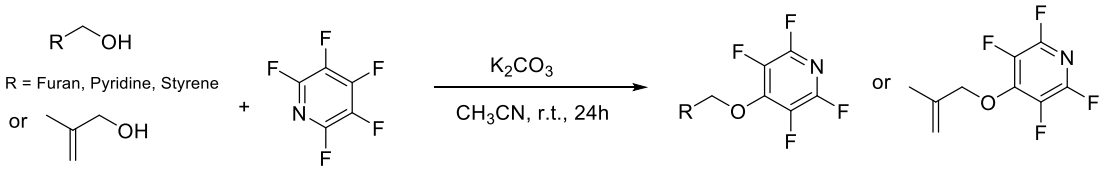
2. General procedures

2.1 General procedures for the synthesis of substrate 1[1]:



R= F, Cl, CF_3, NO_2, Me, *t*-Bu, Ph

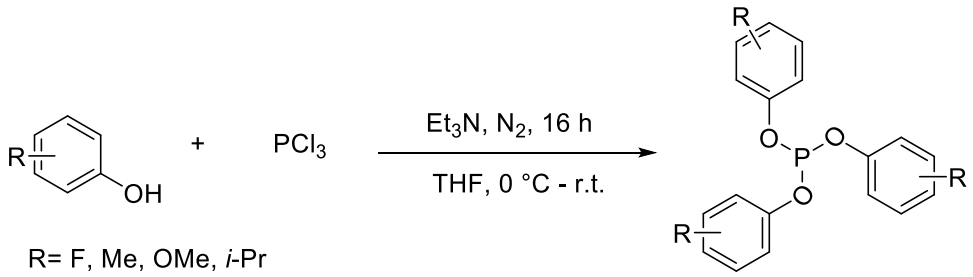
To a 100 mL round-bottomed flask with a stir bar was added potassium carbonate (2.5 eq, 25 mmol, 3.46g), DMF (30 mL), then was added benzyl alcohol (1.2 eq, 12.0 mmol, 1.24 mL), followed by the dropwise addition of pentafluoropyridine (1.0 eq, 10.0 mmol, 1.1 mL). The reaction mixture was stirred for 24 h at room temperature, then was added saturated brine (5 mL) to quench the reaction, and the potassium carbonate was removed by filtration. The liquid phase was transferred to a 250 mL round-bottomed flask and poured into 10 % NH_4Cl aqueous (200 mL), then stirred at 0 °C for at least 2 h. The resulting white solid was collected by vacuum filtration, dried over anhydrous Na_2SO_4, filtered, and evaporated to afford pure starting material. If the product is an oil, the mixture was extracted with ethyl acetate (3 × 20 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude material was purified by flash chromatography on a silica-gel column to obtain the corresponding substrate.



To a 100 mL round-bottomed flask with a stir bar was added potassium carbonate (2.5 eq, 25 mmol, 3.46g), CH_3CN (30 mL), then was added benzyl alcohol (1.0 eq, 10.0 mmol, 1.04 mL), followed by the dropwise addition of pentafluoropyridine (1.2 eq, 12.0 mmol, 1.32 mL). The reaction mixture was stirred for 24 h at room temperature. After the reaction was complete, the reaction mixture was filtered through fret funnel and the filtrate obtained was concentrated on rotavapor. The crude reaction mixture obtained was then purified by flash chromatography on a silica-gel column to obtain

corresponding substrate.

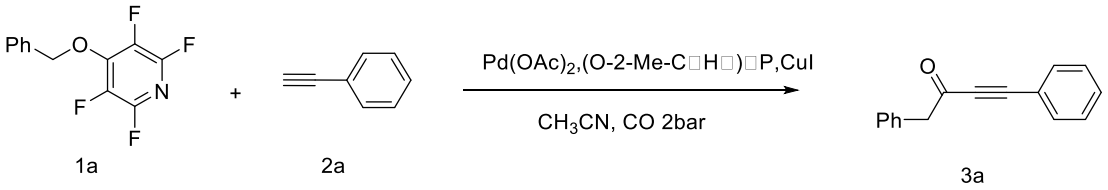
2.2 General procedures for ligand preparation:[2]



R= F, Me, OMe, *i*-Pr

To a well stirred solution of substituted phenols (3.2 eq, 16.0 mmol) in THF (100 ml) under nitrogen at 0 °C, Et_3N (4.0 eq, 20.0 mmol, 2.8 mL) was added dropwise freshly distilled PCl_3 (1.0 eq, 5.0 mmol, 0.44 mL). The reaction mixture was stirred for 16h at room temperature. After the reaction was complete, the reaction mixture was filtered through fret funnel and the filtrate obtained was concentrated on rotavapor. The crude reaction mixture obtained was then purified by flash chromatography on a silica-gel column to obtain corresponding ligand.

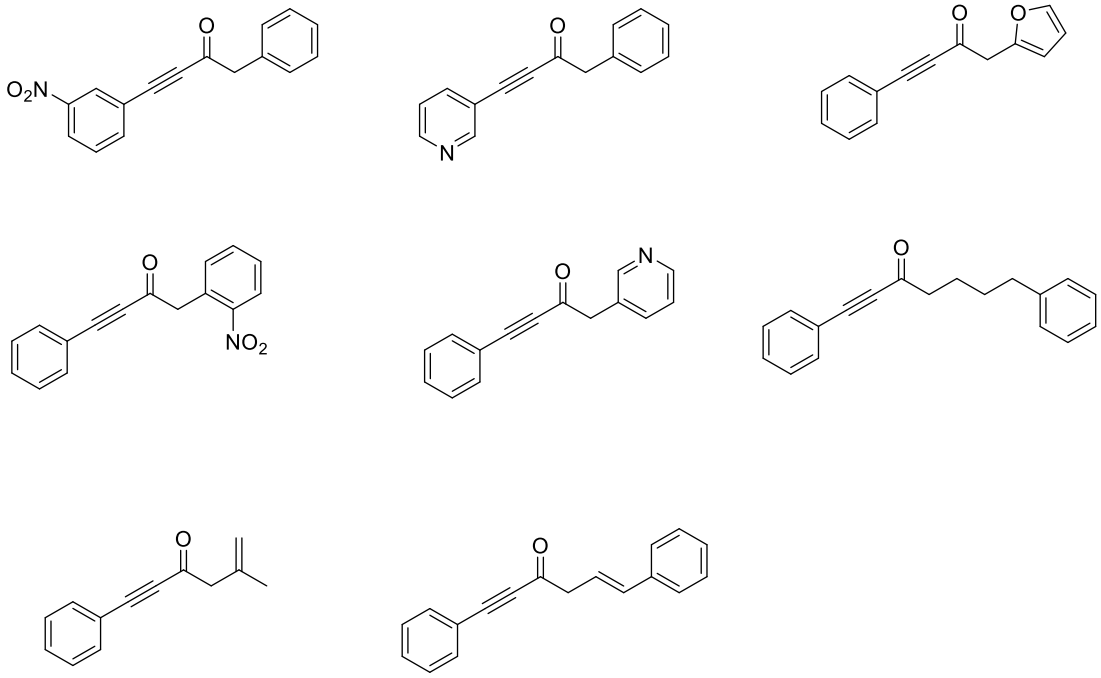
2.3 General procedures for the palladium-catalyzed carbonylation of phenylacetylene with 4-(benzyloxy)-2,3,5,6-tetrafluoropyridine:



A flame-dried glass pressure tube (5 mL) was charged with 4-(benzyloxy)-2,3,5,6-tetrafluoropyridine **1a** (2.0 eq, 0.4 mmol, 103 mg), $Pd(OAc)_2$ (5 mol %, 2.3 mg), CuI (20 mol %, 7.6 mg). Then the glass pressure tube was capped with a PTFE septum, evacuated, and backfilled with nitrogen three times. phenylacetylene **2a** (1.0 eq, 0.2 mmol, 22 μL), $(O\text{-}2\text{-}Me\text{-}C_6H_4)_3P$ (15 mol %, 10 μL) and CH_3CN (2 mL) were added through the septum. The glass pressure tube was placed in an aluminum heating block, and a needle was inserted through the septum of each tube to maintain pressure equilibrium during subsequent CO exposure. The aluminum heating block was then transferred to an autoclave. The autoclave was purged three times with nitrogen and once with CO. After sealing, the system was pressurized with CO (2 bar), heated to 120 °C and stirred for 24 h. Upon completion, the

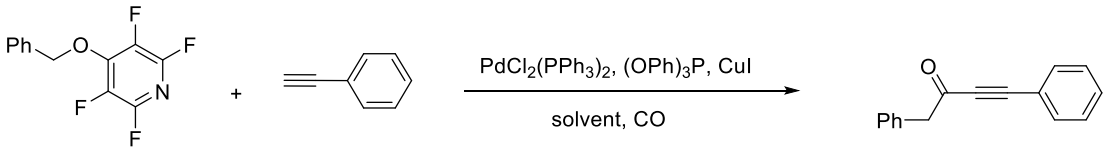
autoclave was cooled to room temperature, carefully vented in a fume hood to release CO, and then purged three times with nitrogen to remove residual gas. The reaction mixture was diluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (eluent: PE/EA = 130:1 - 90:1) to afford the desired product **3a**.

2.4 Failed examples



3. Optimization of reaction conditions

3.1 Optimization of reaction solvents

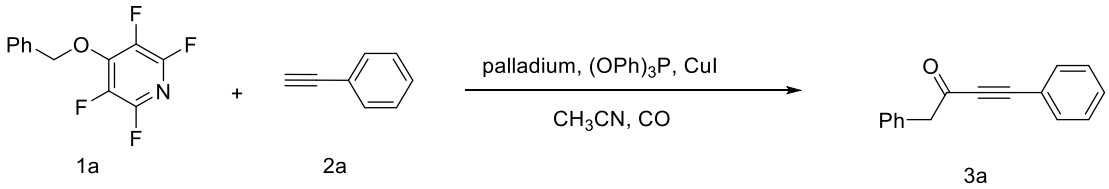


Entry	Solvent	Yield(%)
1	DMF	N.R.
2	DMAc	N.R.
3	dioxane	N.R.

4	toluene	Trace
5	**CH_3CN**	**36.5**
6	DMSO	N.R.
7	THF	N.R.
8	DCE	N.R.
9	DME	Trace

Reaction conditions: 1a (103 mg, 0.4 mmol, 2.0 eq), 2a (22 μL, 0.2 mmol, 1.0 eq), $PdCl_2(PPh_3)_2$ 5 mol%, $(OPh)_3P$ 10 mol%, CuI (7.6 mg, 20 mol%), CO 2bar, solvent 2 mL, 110 °C, 24 h, GC yields. N.R. = no reaction.

3.2 Optimization of the palladium catalysts

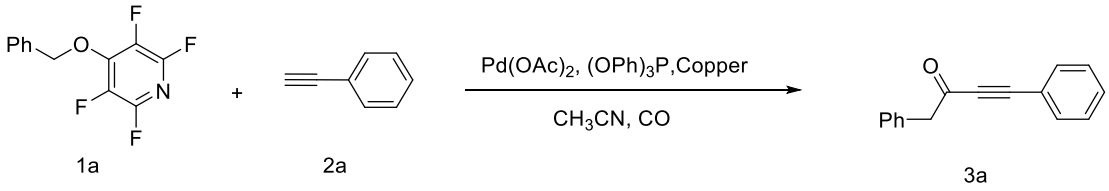


Entry	Pd catalysts	Yield (%)
1	$PdCl_2(PPh_3)_2$	36.5
2	**$Pd(OAc)_2$**	**65.7**
3	$Pd(TFA)_2$	43.4
4	K_2PdCl_4	16.8
5	10% Pd/C	39.9
6	$Pd(PPh_3)_4$	30.2
7	$PdCl_2$	Trace
8	$PdCl_2$(bpy)	Trace
9	$PdCl_2$(cod)	Trace
10	$Pd(PhCN)_2Cl_2$	Trace
11	$Pd_2(dba)_3$	41.6
12	PdG_4	19.7

Reaction conditions: 1a (103 mg, 0.4 mmol, 2.0 eq), 2a (22 μL, 0.2 mmol, 1.0 eq), palladium catalyst 5 mol%, $(OPh)_3P$ 10 mol%, CuI (7.6 mg, 20 mol%), CO 2bar, CH_3CN 2 mL, 110 °C, 24 h, GC yields. PdG_4: t-

BuXphos Palladacycle Gen. 4. N.R. = no reaction.

3.3 Optimization of Copper catalysts

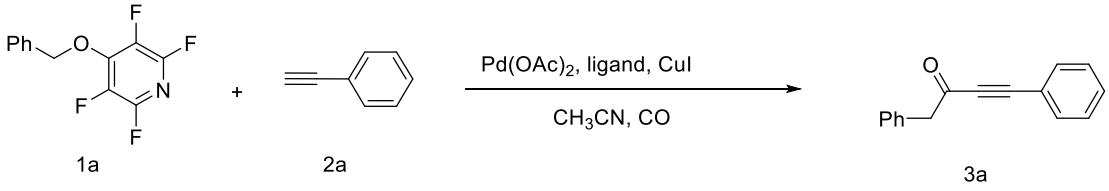


1a 2a 3a

Entry	Copper Catalysts	Yield (%)
1	20 mol% CuCl	Trace
2	20 mol% CuBr	Trace
3	**20 mol% CuI**	**66.9**
4	15 mol% CuI	58.6
5	10 mol% CuI	44.8
6	5 mol% CuI	29.2

Reaction conditions: 1a (103 mg, 0.4 mmol, 2.0 eq), 2a (22 μL, 0.2 mmol, 1.0 eq), $Pd(OAc)_2$ 5 mol%, $(OPh)_3P$ 10 mol%, CuI (5-20 mol%), CO 2bar, CH_3CN 2 mL, 110 °C, 24 h, GC yields.

3.4 Optimization of ligands



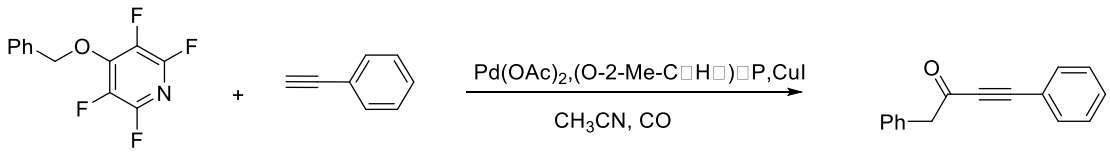
1a 2a 3a

Entry	Ligand	Yield (%)
1	10 mol% $(OPh)_3P$	66.9
2	10 mol% $(O\text{-}2\text{-}F\text{-}C_6H_4)_3P$	40.2
3	5 mol% $(O\text{-}2\text{-}Me\text{-}C_6H_4)_3P$	59.4
4	10 mol% $(O\text{-}2\text{-}Me\text{-}C_6H_4)_3P$	74.6
5	**15 mol% $(O\text{-}2\text{-}Me\text{-}C_6H_4)_3P$**	**82.5**
6	10 mol% $(O\text{-}4\text{-}Me\text{-}C_6H_4)_3P$	68.2
7	10 mol% $(O\text{-}2\text{-}MeO\text{-}C_6H_4)_3P$	61.6
8	10 mol% $(O\text{-}2\text{-}iPr\text{-}C_6H_4)_3P$	65.9

9	10 mol% $[O\text{-}2,4\text{-}(tBu)_2C_6H_3]_3P$	47.7
10	10 mol% $(PhO)Ph_2P$	18.4
11	10 mol% $(4\text{-}F\text{-}Ph)_3P$	42.6
12	10 mol% $(4\text{-}CF_3\text{-}Ph)_3P$	35.7
13	10 mol% $(2\text{-}MeO\text{-}Ph)_3P$	25.1
14	10 mol% $(4\text{-}MeO\text{-}Ph)_3P$	20.3
15	10 mol% $(OMe)_3P$	23.8
16	10 mol% $(O\text{-}iPr)_3P$	50.7
17	10 mol% $(2\text{-}Fur)_3P$	53.0
18	10 mol% Ni Xantphos	28.6
19	10 mol% $PPh_3(CF_3)_6$	35.6
20	10 mol% BINAP	N.R.
21	10 mol% PPh_3	24.3
22	10 mol% DPPF	Trace
23	10 mol% bpy	Trace
24	10 mol% tBuNC	38.3
25	10 mol% SPhos	24.5

Reaction conditions: 1a (103 mg, 0.4 mmol, 2.0 eq), 2a (22 μL, 0.2 mmol, 1.0 eq), $Pd(OAc)_2$ 5 mol%, ligand 5-15 mol%, CuI (7.6 mg, 20 mol%), CO 2bar, CH_3CN 2 mL, 110 °C, 24 h, GC yields. Sphos: 2-Dicycloh exylphosphino-2',6'-dimethoxybiphenyl. N.R. = no reaction.

3.5 Further optimization of reaction conditions



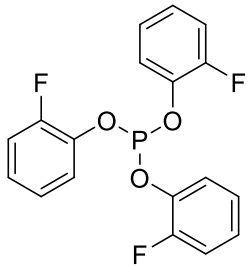
1a 2a 3a

Entry	Variable	Yield (%)
1	80 ℃	Trace
2	100 ℃	67.3
3	**120 ℃**	**89.7**

4	140 ℃	38.3
5	1a 1.5 eq	66.8
6	1a 2.5 eq	81.0
7	1a 1.0 eq, 2a 2.0 eq	63.7
8	CO 5bar	80.8
9	Without $Pd(OAc)_2$	54.4
10	Without $(O\text{-}2\text{-}Me\text{-}C_6H_4)_3P$	30.7
11	Without CuI	Trace

Reaction conditions: 1a (103 mg, 0.4 mmol, 2.0 eq), 2a (22 μL, 0.2 mmol, 1.0 eq), $Pd(OAc)_2$ 5 mol%, $(O\text{-}2\text{-}Me\text{-}C_6H_4)_3P$ 15 mol%, CuI (7.6 mg, 20 mol%), CO 2bar, CH_3CN 2 mL, 120 °C, 24 h, GC yields. N.R. = no reaction.

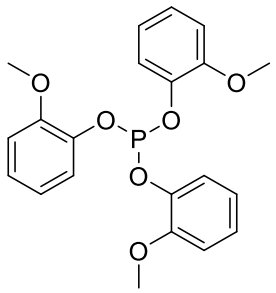
4. Characterization data of products



Tris (2-Fluorophenyl) phosphite: (Colorless oil, eluent: Petroleum Ether) [2]

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (t, J = 7.42 Hz, 3H), 7.18 – 7.06 (m, 9H).

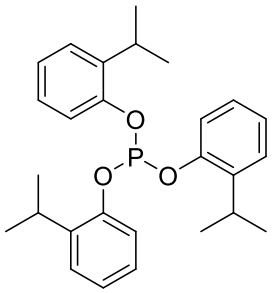
^{13}C NMR (75 MHz, $CDCl_3$) δ 151.1 (d, $J_{C\text{-}F}$ = 235.43 Hz), δ 143.5 (d, $J_{C\text{-}F}$= 14.10 Hz), 124.8 (d, $J_{C\text{-}F}$ = 3.87 Hz), 120.7 (d, $J_{C\text{-}F}$ = 6.53 Hz), 117.2 (d, $J_{C\text{-}F}$ = 1.66 Hz), 115.5 (d, $J_{C\text{-}F}$ = 18.07 Hz).



Tris (2-Methoxyphenyl) phosphite: (Colorless oil, eluent: Petroleum Ether) [2]

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.93 (t, *J* = 2.12 Hz, 3H), 6.91 – 6.81 (m, 9H), 3.89 (s, 9H).

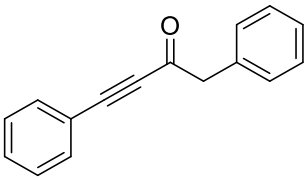
^{13}C NMR (75 MHz, $CDCl_3$) δ 146.5, 145.6, 121.4, 120.1, 114.5, 110.7, 55.8.



Tris (2-Isopropylphenyl) phosphite: (Colorless oil, eluent: Petroleum Ether) [2]

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 3H), 7.23 – 7.21 (m, 3H), 7.17 – 7.08 (m, 6H), 3.30 (t, 3H), 1.16 (d, *J* = 6.89 Hz, 18H).

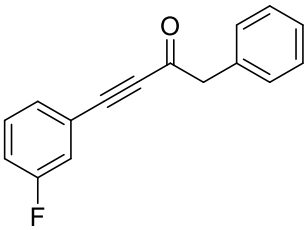
^{13}C NMR (75 MHz, $CDCl_3$) δ 152.8, 134.4, 126.6, 126.3, 120.8, 115.2, 26.9, 22.5.



1,4-Diphenylbut-3-yn-2-one(3a): (74.0 mg, yellow oil, yield: 84%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.30 (m, 10H), 3.95 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.2, 133.1, 133.0, 130.8, 129.8, 128.6, 128.5, 127.3, 119.7, 92.9, 87.6, 52.1.



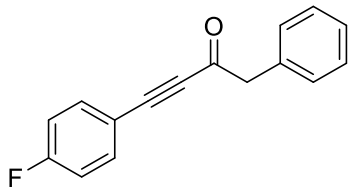
4-(3-Fluorophenyl)-1-phenylbut-3-yn-2-one(3b): (81.0 mg, yellow oil, yield: 85%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 6H), 7.25 – 7.22 (m, 1H), 7.17 – 7.11 (m, 2H), 3.94 (s,

2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 177.2, 162.1 (d, J_{C-F} = 247.90 Hz), 132.9, 130.3 (d, J_{C-F} = 8.64 Hz), 129.8, 128.9 (d, J_{C-F} = 3.16 Hz), 128.7, 127.5, 121.5 (d, J_{C-F} = 9.30 Hz), 119.6 (d, J_{C-F} = 23.15 Hz), 118.2 (d, J_{C-F} = 21.45 Hz), 90.9, 87.8, 52.1.

HRMS (ESI-TOF) Calc. for $C_{16}H_{12}FO$; $[M+H]^+$: 239.0866; found: 239.0863.

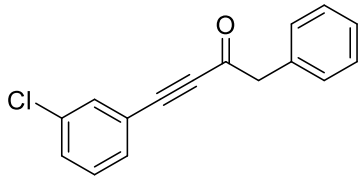


4-(4-Fluorophenyl)-1-phenylbut-3-yn-2-one(3c): (85.8 mg, yellow oil, yield: 90%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [4]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.30 (m, 7H), 7.04 (t, J = 8.68 Hz, 2H), 3.92 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.1, 163.9 (d, J_{C-F} = 254.03 Hz), 135.3 (d, J_{C-F} = 8.96 Hz), 133.1, 129.8, 129.3 (d, J_{C-F} = 56.81 Hz), 128.7, 127.4, 116.1 (d, J_{C-F} = 22.36 Hz), 91.9, 87.5, 52.0.

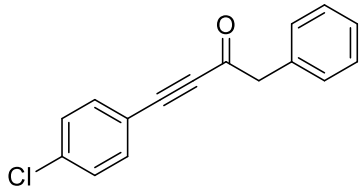
^{19}F NMR (282 MHz, $CDCl_3$) δ -105.9.



4-(3-Chlorophenyl)-1-phenylbut-3-yn-2-one(3d): (70.3 mg, yellow oil, yield: 69%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [4]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 3H), 7.38 – 7.30 (m, 6H), 3.94 (s, 2H).

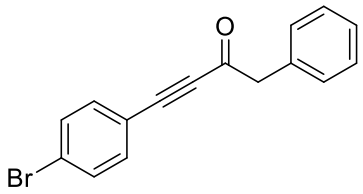
^{13}C NMR (101 MHz, $CDCl_3$) δ 184.9, 134.4, 132.9, 132.6, 131.1, 131.0, 129.8, 128.7, 128.5, 127.5, 121.5, 90.7, 88.0, 52.1.



4-(4-Chlorophenyl)-1-phenylbut-3-yn-2-one(3e): (95.8 mg, yellow oil, yield: 94%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [5]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 9H), 3.93 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.0, 137.2, 134.2, 133.0, 129.8, 129.0, 128.7, 127.4, 118.2, 91.4, 88.3, 52.0.

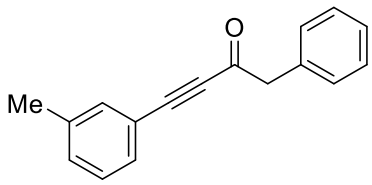


4-(4-Bromophenyl)-1-phenylbut-3-yn-2-one(3f): (62.2 mg, white solid, m.p. = 115-117 ℃, yield: 52%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.46 Hz, 2H), 7.37 (d, *J* = 6.63 Hz, 2H), 7.34 – 7.28 (m, 5H), 3.93 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.0, 134.3, 133.0, 132.0, 129.8, 128.7, 127.4, 125.6, 118.7, 91.5, 88.4, 52.1.

HRMS (ESI-TOF) Calc. for $C_{16}H_{12}BrO$; $[M+H]^+$: 299.0066; found: 299.0062.

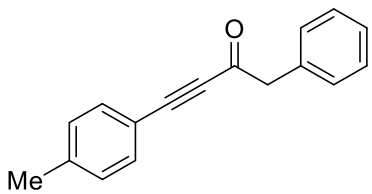


1-Phenyl-4-(m-tolyl)but-3-yn-2-one(3g): (89.0 mg, yellow oil, yield: 95%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 7.32 (dd, *J* = 7.37, 3.03 Hz, 3H), 7.26 (dt, *J* = 15.31, 3.20 Hz, 4H), 3.93 (s, 2H), 2.34 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.2, 138.4, 133.6, 133.3, 131.7, 130.2, 129.8, 128.7, 128.4, 127.3, 119.6, 93.3, 87.5, 52.1, 21.1.

HRMS (ESI-TOF) Calc. for $C_{17}H_{15}O$; $[M+H]^+$: 235.1117; found: 235.1113.

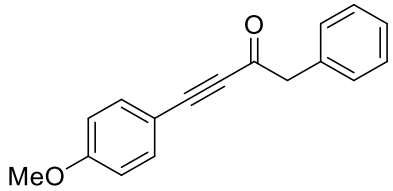


1-Phenyl-4-(p-tolyl)but-3-yn-2-one(3h): (81.5 mg, yellow oil, yield: 87%, Petroleum

Ether/Ethyl acetate = 130:1 - 90:1) [5]

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.28 (m, 7H), 7.16 (d, *J* = 7.92 Hz, 2H), 3.93 (s, 2H), 2.37 (s, 3H).

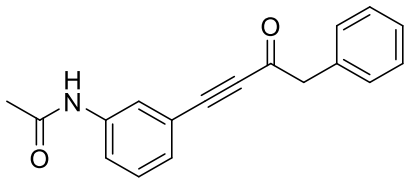
^{13}C NMR (75 MHz, $CDCl_3$) δ 185.2, 141.5, 133.3, 133.1, 129.8, 129.3, 128.6, 127.3, 116.6, 93.6, 87.6, 52.1, 21.7.



4-(4-Methoxyphenyl)-1-phenylbut-3-yn-2-one(3i): (49.1 mg, yellow oil, yield: 49%, Petroleum Ether/Ethyl acetate = 100:1 - 70:1) [5]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.27 (m, 7H), 6.86 (d, *J* = 8.42 Hz, 2H), 3.92 (s, 2H), 3.81 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.2, 161.6, 135.1, 133.4, 129.8, 128.6, 127.2, 114.2, 111.4, 94.2, 87.6, 55.3, 51.9.

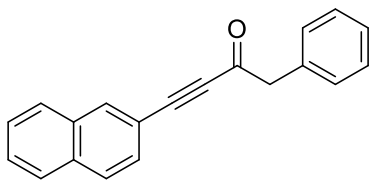


***N*-(3-(3-Oxo-4-phenylbut-1-yn-1-yl) phenyl) acetamide(3j):** (47.7 mg, yellow oil, yield: 43%, Petroleum Ether/Ethyl acetate = 20:1 - 3:1)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.66 (s, 1H), 7.59 (d, *J* = 8.14 Hz, 1H), 7.37 – 7.23 (m, 6H), 7.15 (d, *J* = 7.68 Hz, 1H), 3.93 (s, 2H), 2.14 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 185.5, 169.0, 138.3, 132.9, 129.8, 129.2, 129.0, 128.7, 127.4, 123.8, 122.4, 120.2, 92.6, 87.5, 52.1, 24.4.

HRMS (ESI-TOF) Calc. for $C_{18}H_{16}NO_2$; $[M+H]^+$: 278.1175; found: 278.1176.

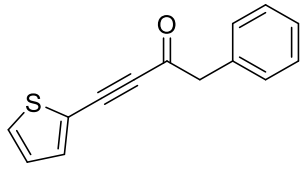


4-(Naphthalen-2-yl)-1-phenylbut-3-yn-2-one(3k): (55.1 mg, white solid, m.p. = 73-75 ℃, yield: 51%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.87 – 7.76 (m, 3H), 7.60 – 7.51 (m, 2H), 7.49 – 7.29 (m, 6H), 3.99 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 185.2, 134.4, 133.9, 133.3, 132.5, 129.9, 128.7, 128.4, 128.3, 128.1, 128.0, 127.8, 127.4, 127.0, 116.9, 93.4, 87.9, 52.1.

HRMS (ESI-TOF) Calc. for $C_{20}H_{15}O$; $[M+H]^+$: 271.1117; found: 271.1115.

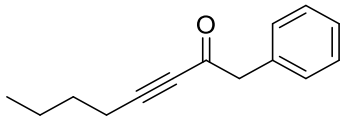


1-Phenyl-4-(thiophen-2-yl)but-3-yn-2-one(3l): (39.8 mg, yellow oil, yield: 44%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 5.12, 1.19 Hz, 1H), 7.41 – 7.35 (m, 3H), 7.35 – 7.29 (m, 3H), 7.04 (dd, *J* = 5.08, 3.75 Hz, 1H), 3.93 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 184.7, 136.8, 133.1, 131.9, 129.8, 128.7, 127.7, 127.3, 119.5, 92.3, 86.9, 51.8.

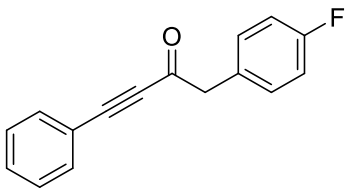
HRMS (ESI-TOF) Calc. for $C_{14}H_{11}OS$; $[M+H]^+$: 227.0525; found: 227.0523.



1-Phenyloct-3-yn-2-one(3m): (50.4 mg, Colorless oil, yield: 63%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [6]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.23 (m, 5H), 3.81 (s, 2H), 2.31 (t, *J* = 6.96 Hz, 2H), 1.52 – 1.44 (m, 2H), 1.39 – 1.29 (m, 2H), 0.89 (t, *J* = 7.24 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 185.4, 133.2, 129.7, 128.6, 127.2, 96.5, 80.7, 52.2, 29.5, 21.8, 18.6, 13.4.

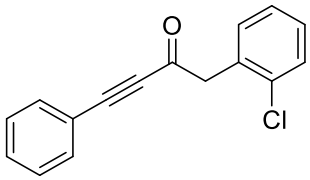


1-(4-Fluorophenyl)-4-phenylbut-3-yn-2-one(4a): (71.4 mg, yellow oil, yield: 75%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [7]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.42 (m, 3H), 7.41 – 7.34 (m, 2H), 7.32 – 7.24 (m, 2H), 7.11 – 7.03 (m, 2H), 3.92 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 184.9, 162.2 (d, $J_{C\text{-}F}$ = 245.91 Hz), 133.0, 131.4 (d, $J_{C\text{-}F}$ = 8.09 Hz), 130.9, 128.9 (d, $J_{C\text{-}F}$ = 3.32 Hz), 128.6, 119.6, 115.6 (d, $J_{C\text{-}F}$ = 21.56 Hz), 93.0, 87.5, 51.1.

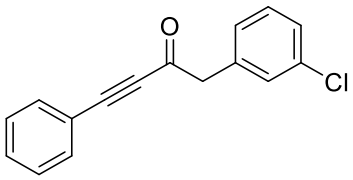
^{19}F NMR (565 MHz, $CDCl_3$) δ -115.2.



1-(2-Chlorophenyl)-4-phenylbut-3-yn-2-one(4b): (63.2 mg, yellow oil, yield: 62%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [8]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.40 (m, 4H), 7.39 – 7.23 (m, 5H), 4.09 (s, 2H).

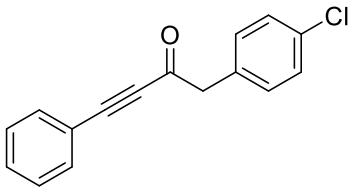
^{13}C NMR (75 MHz, $CDCl_3$) δ 183.8, 134.9, 133.1, 132.0, 131.8, 130.8, 129.5, 129.0, 128.5, 127.0, 119.6, 92.7, 87.4, 49.6.



1-(3-Chlorophenyl)-4-phenylbut-3-yn-2-one(4c): (72.3 mg, yellow oil, yield: 71%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [5]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, J = 1.60 Hz, 1H), 7.40 – 7.28 (m, 7H), 7.20 (d, J = 2.50 Hz, 1H), 3.91 (s, 2H).

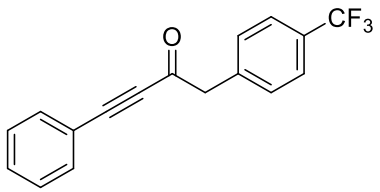
^{13}C NMR (151 MHz, $CDCl_3$) δ 190.8, 135.1, 134.3, 133.1, 131.0, 130.0, 129.9, 128.6, 128.0, 127.6, 119.6, 93.4, 87.5, 51.5.



1-(4-Chlorophenyl)-4-phenylbut-3-yn-2-one(4d): (70.3 mg, yellow oil, yield: 69%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [5]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.43 (m, 3H), 7.41 – 7.33 (m, 4H), 7.25 (d, J = 8.36 Hz, 2H), 3.91 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 184.5, 133.4, 133.1, 131.6, 131.1, 130.9, 128.8, 128.6, 119.6, 93.1, 87.5, 51.3.



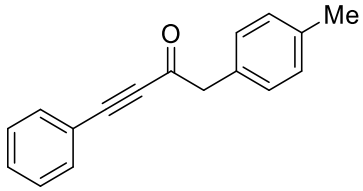
4-Phenyl-1-(4-(trifluoromethyl)phenyl)but-3-yn-2-one(4e): (88.8 mg, white solid, m.p. = 77-79 ℃, yield: 77%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.6 (d, J = 8.05 Hz, 2H), 7.5 – 7.3 (m, 7H), 4.0 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.0, 137.2 (q, $J_{C\text{-}F}$ = 1.27 Hz), 133.1, 131.0, 130.2, 129.7 (q, $J_{C\text{-}F}$ = 32.54 Hz), 128.6, 125.6 (q, $J_{C\text{-}F}$ = 3.71 Hz), 122.7, 119.4, 93.4, 87.5, 51.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -62.6.

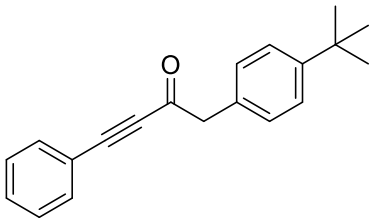
HRMS (ESI-TOF) Calc. for $C_{17}H_{12}F_3O$; $[M+H]^+$: 289.0834; found: 289.0833.



4-Phenyl-1-(p-tolyl)but-3-yn-2-one(4f): (66.5 mg, yellow oil, yield: 71%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [5]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 – 7.31 (m, 6H), 7.23 – 7.20 (m, 3H), 3.92 (s, 2H), 2.37 (s, 3H).

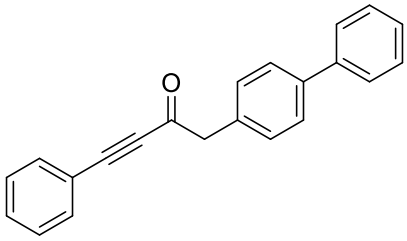
^{13}C NMR (101 MHz, $CDCl_3$) δ 185.4, 136.9, 133.0, 130.7, 130.0, 129.6, 129.3, 128.5, 119.7, 92.6, 87.7, 51.7, 21.0.



1-(4-(Tert-butyl)phenyl)-4-phenylbut-3-yn-2-one(4g): (71.9 mg, yellow oil, yield: 65%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [7]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 – 7.32 (m, 9H), 3.92 (s, 2H), 1.34 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 185.6, 150.2, 133.1, 130.8, 130.2, 129.5, 128.5, 125.6, 119.8, 93.0, 87.8, 51.6, 34.5, 31.3.

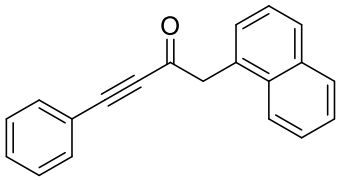


1-([1,1'-Biphenyl]-4-yl)-4-phenylbut-3-yn-2-one(4h): (55.7 mg, yellow solid, m.p. = 104-106 ℃, yield: 47%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 – 7.60 (m, 4H), 7.52 – 7.35 (m, 10H), 4.01 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 185.0, 140.7, 140.3, 133.1, 132.2, 130.8, 130.2, 128.7, 128.6, 127.4, 127.3, 127.0, 119.8, 93.0, 87.8, 51.7.

HRMS (ESI-TOF) Calc. for $C_{22}H_{16}ONa$; $[M+Na]^+$: 319.1093; found: 319.1100.

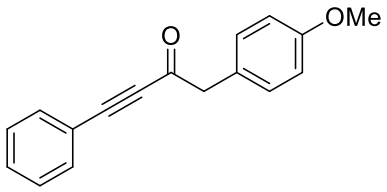


1-(Naphthalen-1-yl)-4-phenylbut-3-yn-2-one(4i): (51.9 mg, yellow oil, yield: 48%, Petroleum Ether/Ethyl acetate = 130:1 - 90:1) [9]

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, J = 8.14 Hz, 1H), 7.94 – 7.83 (m, 2H), 7.61 – 7.46 (m, 5H), 7.29 (d, J = 4.63 Hz, 4H), 4.38 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 185.2, 133.9, 133.0, 132.4, 130.7, 129.9, 128.7, 128.7, 128.4, 128.4,

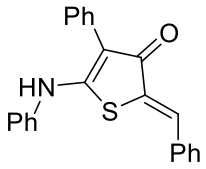
126.5, 125.8, 125.5, 123.9, 119.7, 93.1, 87.7, 49.8.



1-(4-Methoxyphenyl)-4-phenylbut-3-yn-2-one(4j): (29.0 mg, yellow oil, yield: 29%, Petroleum Ether/Ethyl acetate = 100:1 - 70:1) [5]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 – 7.35 (m, 5H), 7.24 (d, *J* = 8.68 Hz, 2H), 6.91 (d, *J* = 8.70 Hz, 2H), 3.88 (s, 2H), 3.81 (s, 3H).

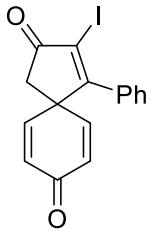
^{13}C NMR (151 MHz, $CDCl_3$) δ 185.5, 133.0, 130.9, 130.7, 128.5, 127.2, 125.2, 119.9, 114.2, 92.7, 87.7, 55.2, 51.3.



(Z)-2-Benzylidene-4-phenyl-5-(phenylamino)thiophen-3(2H)-one: (yellow solid, m.p. = 195-197 ℃, Petroleum Ether/Ethyl acetate = 3:1)[10]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (s, 1H), 7.61 – 7.29 (m, 16H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 184.5, 166.0, 137.8, 134.4, 132.0, 131.2, 130.7, 130.0, 129.5, 129.3, 129.2, 129.0, 128.8, 127.3, 126.5, 123.7, 110.3.



3-Iodo-4-phenylspiro[4.5]deca-3,6,9-triene-2,8-dione: (pale yellow solid, m.p. = 129-131 ℃, Petroleum Ether/Ethyl acetate = 3:1)[11]

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 – 7.34 (m, 3H), 7.26 (d, *J* = 7.14 Hz, 2H), 6.81 (d, *J* = 10.02 Hz, 2H), 6.34 (d, *J* = 10.01 Hz, 2H), 2.96 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.2, 184.2, 176.1, 148.7, 134.0, 130.5, 130.3, 128.5, 126.9, 106.1, 54.2, 43.3.

5. References

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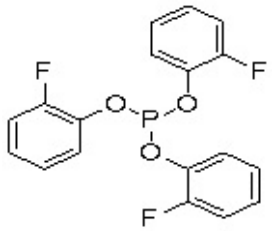
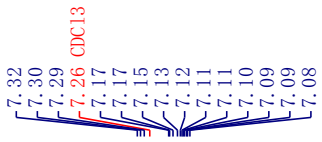
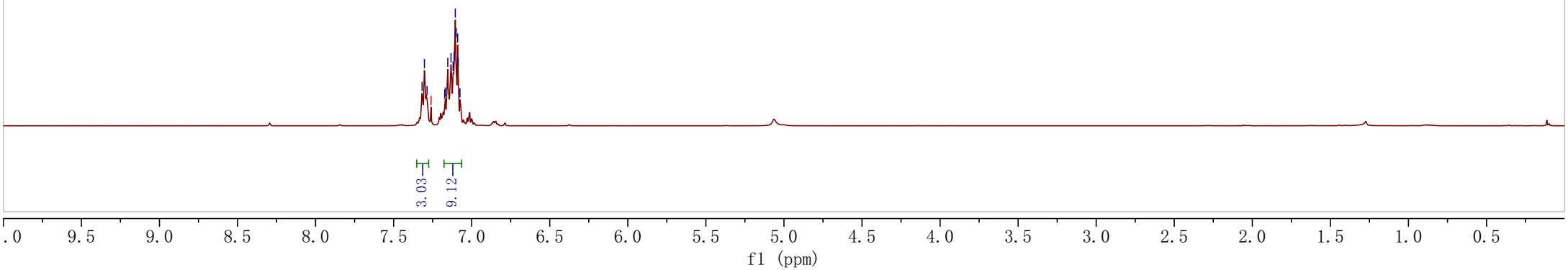
6. Copies of NMR spectra of products



500 MHz, $CDCl_3$





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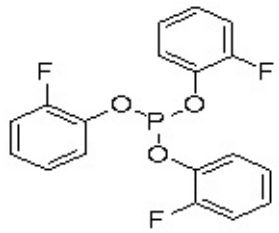
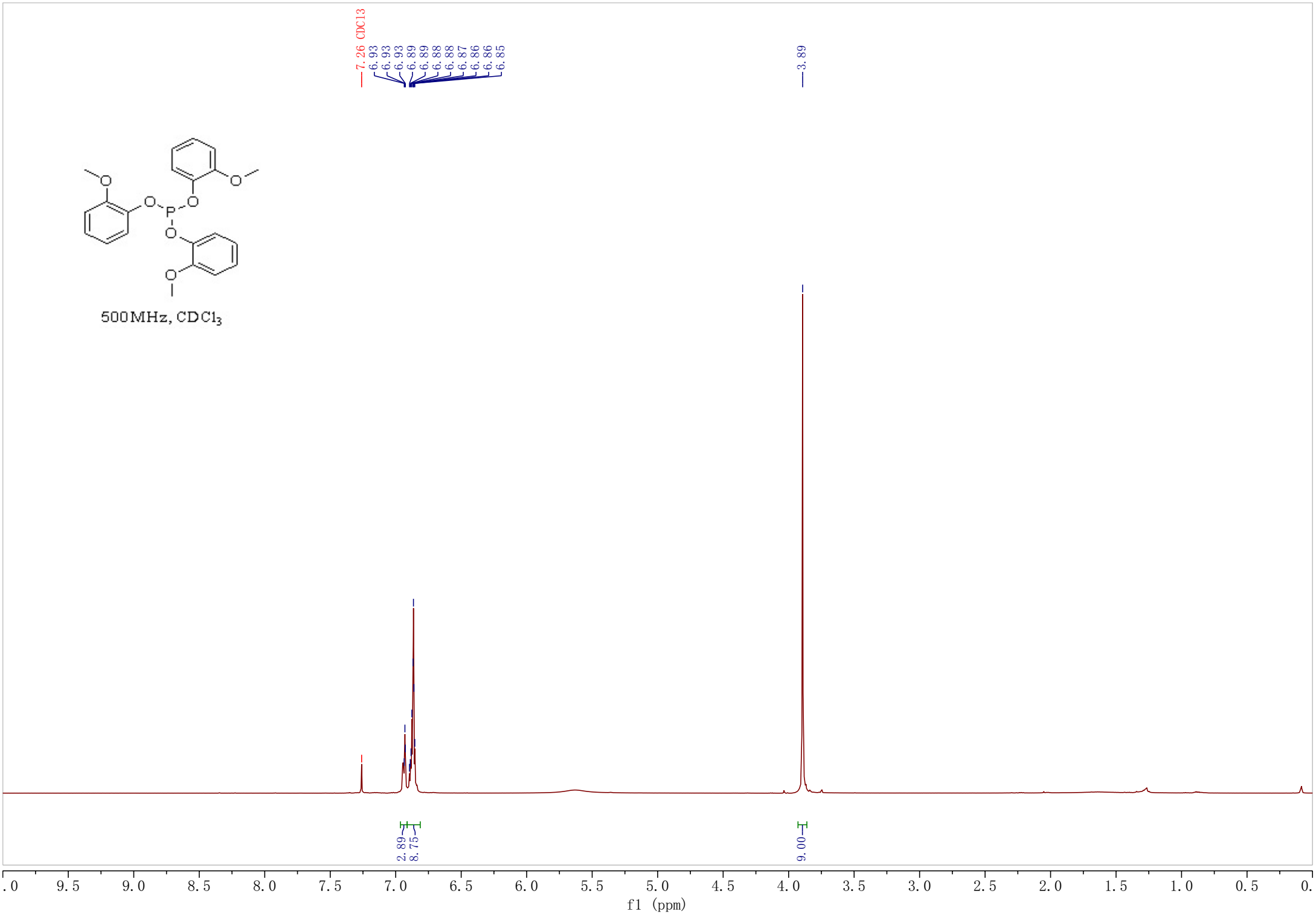


75 MHz, $CDCl_3$


7.26 CDCl3
6.93
6.93
6.89
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6.85
3.89
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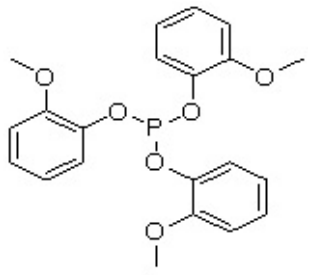
2.89
8.75
9.00
f1 (ppm)

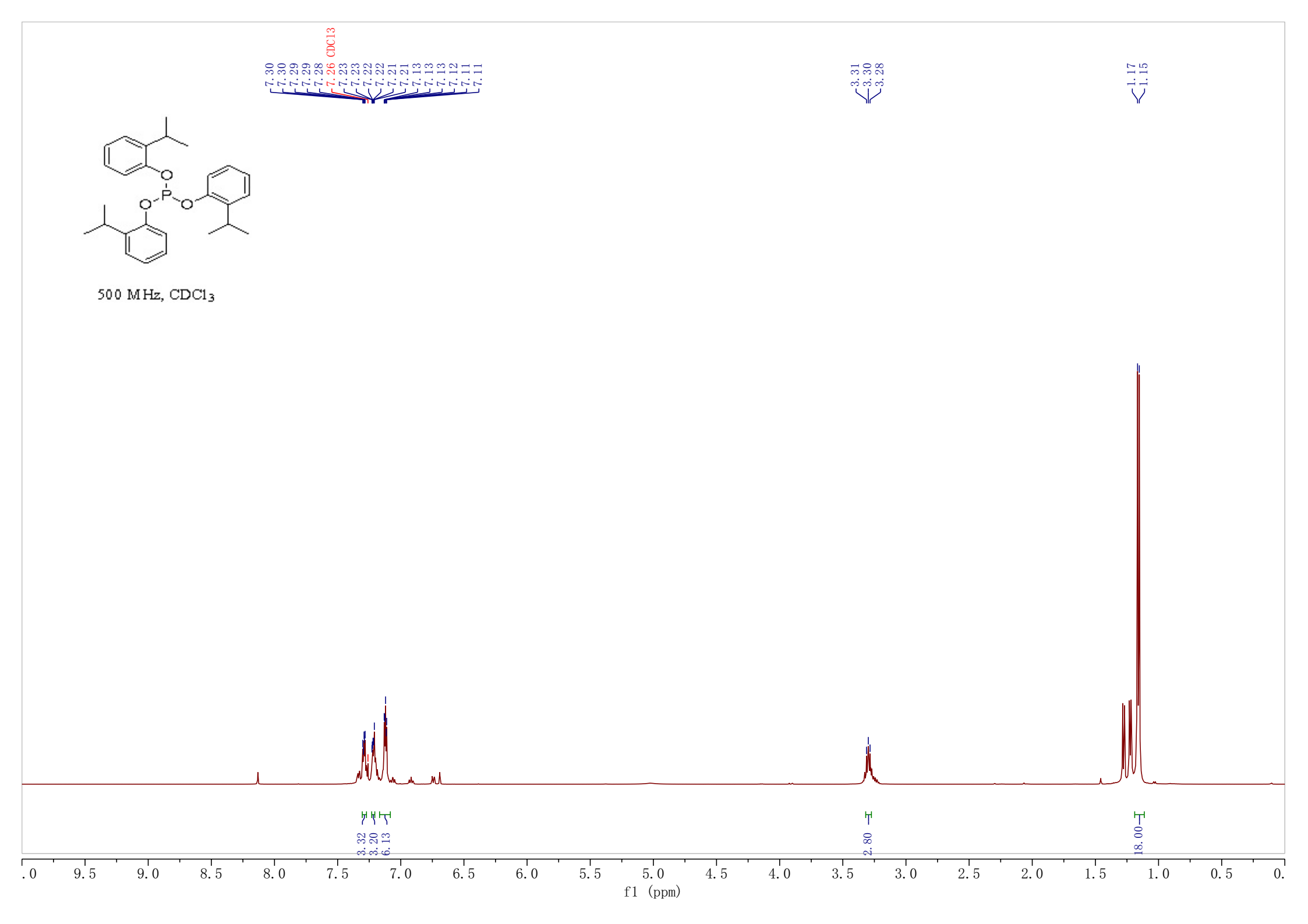
21.1.fid



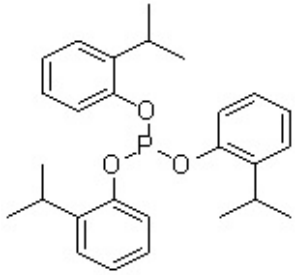
75 MHz, $CDCl_3$

146.5
145.6
121.4
120.1
114.5
110.7
77.4 CDC13
77.0 CDC13
76.6 CDC13
55.8

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

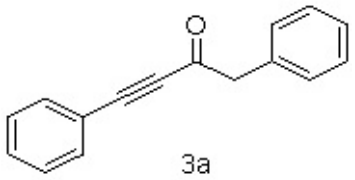

7.30
7.30
7.29
7.29
7.28
7.26
CDCl3
7.23
7.23
7.22
7.22
7.21
7.21
7.13
7.13
7.12
7.11
7.11
3.31
3.30
3.28
1.17
1.15
500 MHz, CDCl3
3.32
3.20
6.13
2.80
18.00
f1 (ppm)

22.1.fid



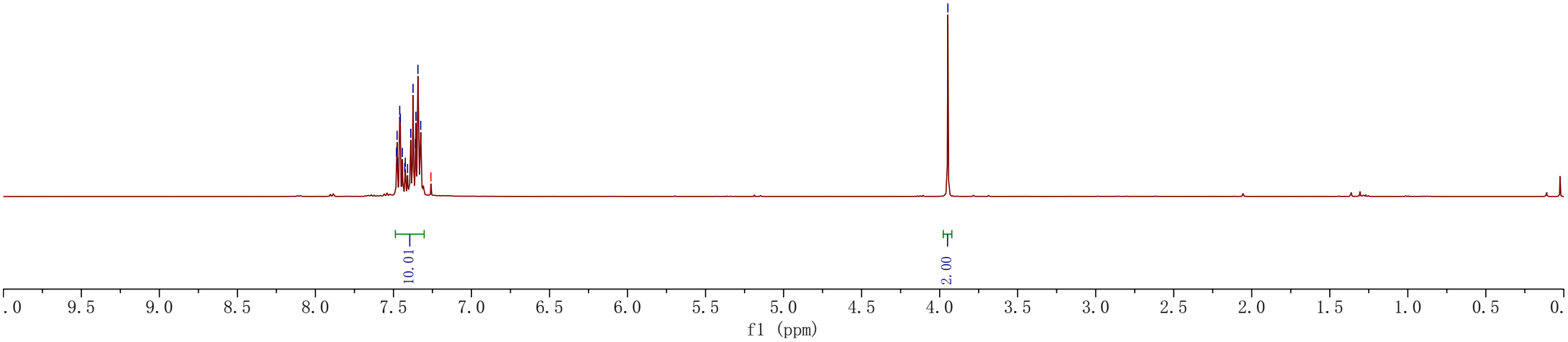
75 MHz, $CDCl_3$

0922-1.1.fid

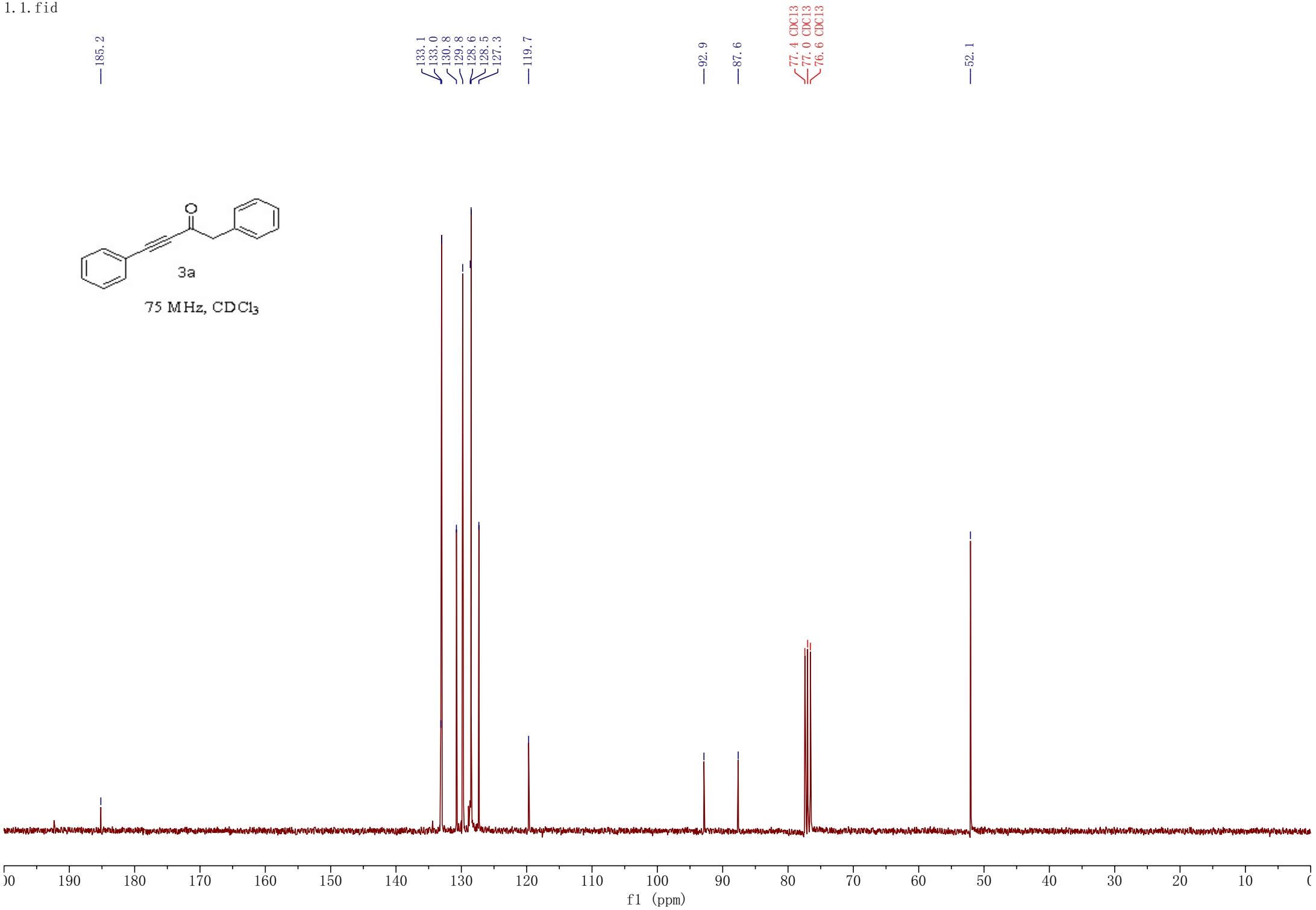


3a

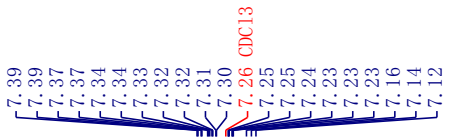
400 MHz, $CDCl_3$

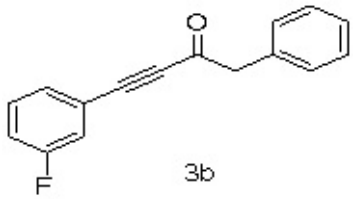


1.1.fid


185.2
133.1
133.0
130.8
129.8
128.6
128.5
127.3
119.7
92.9
87.6
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.1
3a
75 MHz, CDCl3
f1 (ppm)

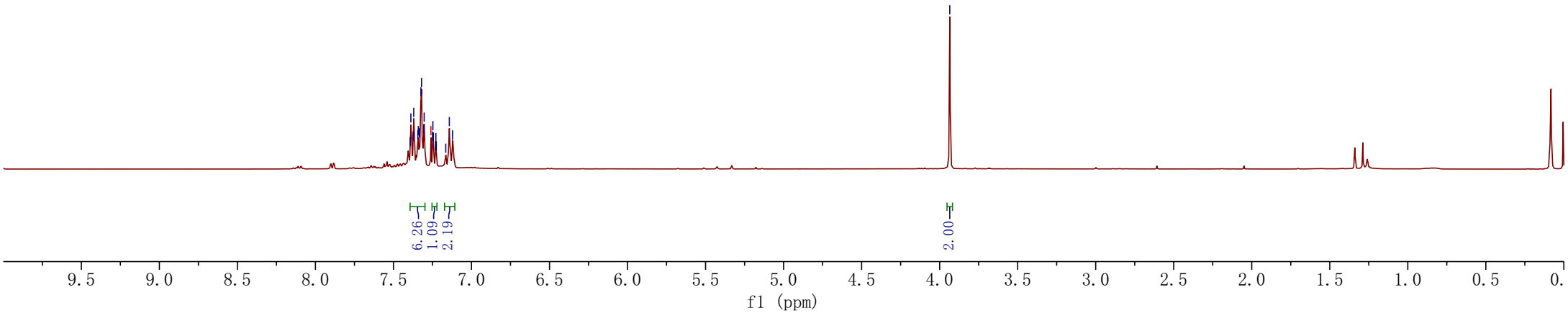
0922-5.1.fid





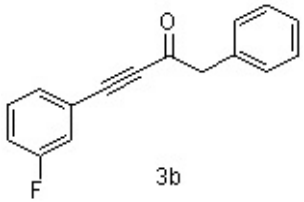
3b

400 MHz, $CDCl_3$



0929-15.1.fid

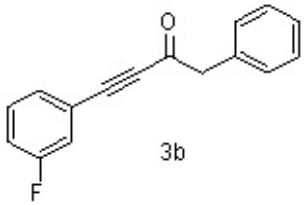
177.2
163.8
160.5
132.9
130.3
130.2
129.8
128.9
128.9
128.7
127.5
121.6
121.5
119.8
119.5
118.4
118.1
90.9
87.8
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.1



3b

75 MHz, $CDCl_3$

f1 (ppm)



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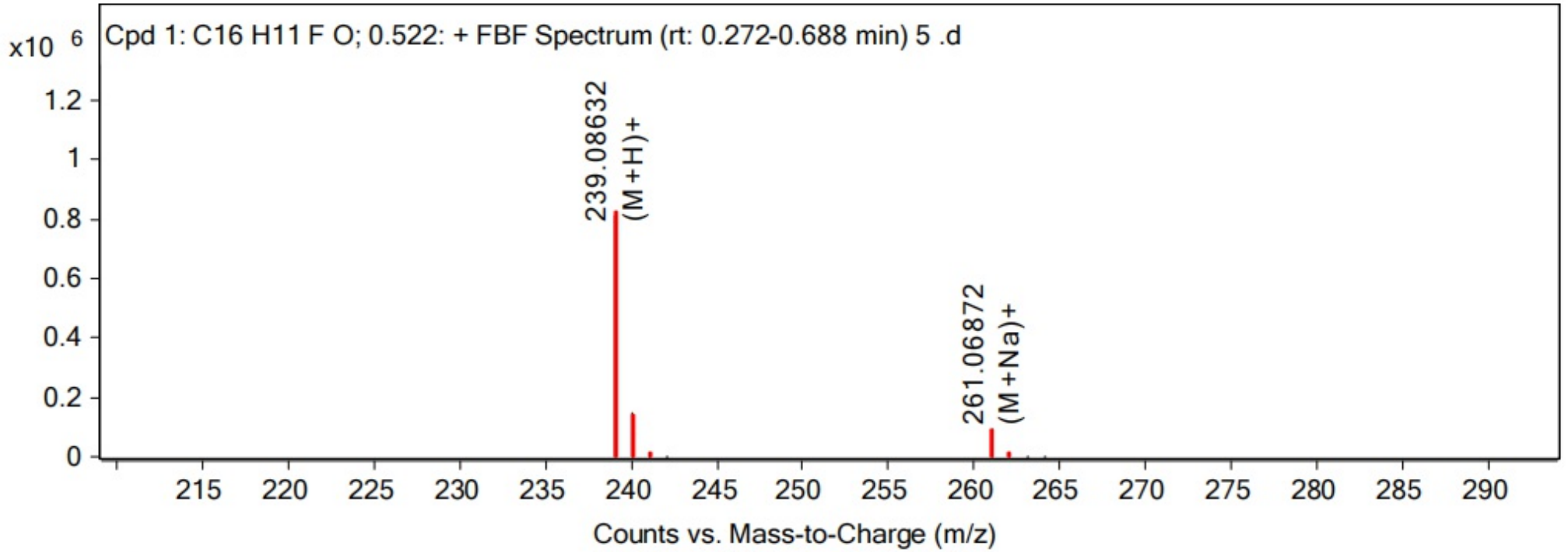
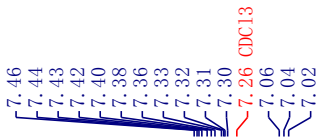


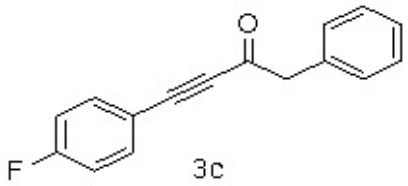
MS Spectrum Peak List

m/z	z	Abund	Ion
239.08632	1	812665.25	(M+H)+
240.09026	1	149220.41	(M+H)+
241.09392	1	17393.53	(M+H)+
261.06872	1	89925.05	(M+Na)+
262.07238	1	17794.31	(M+Na)+

MS Spectrum

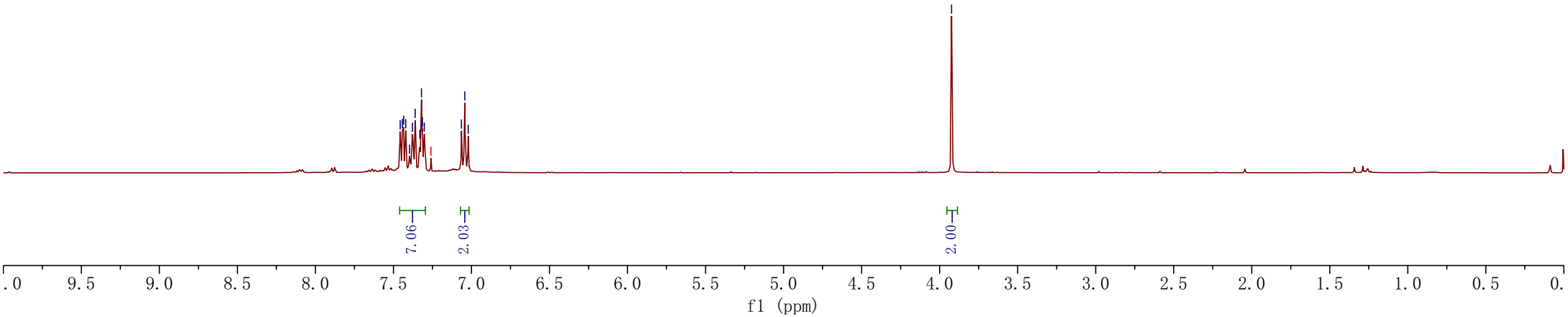
0922-4.1.fid



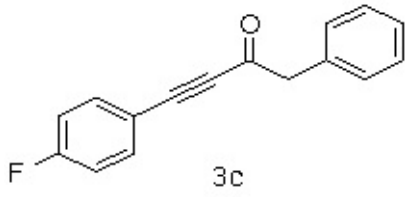


3c

400 MHz, CDC13



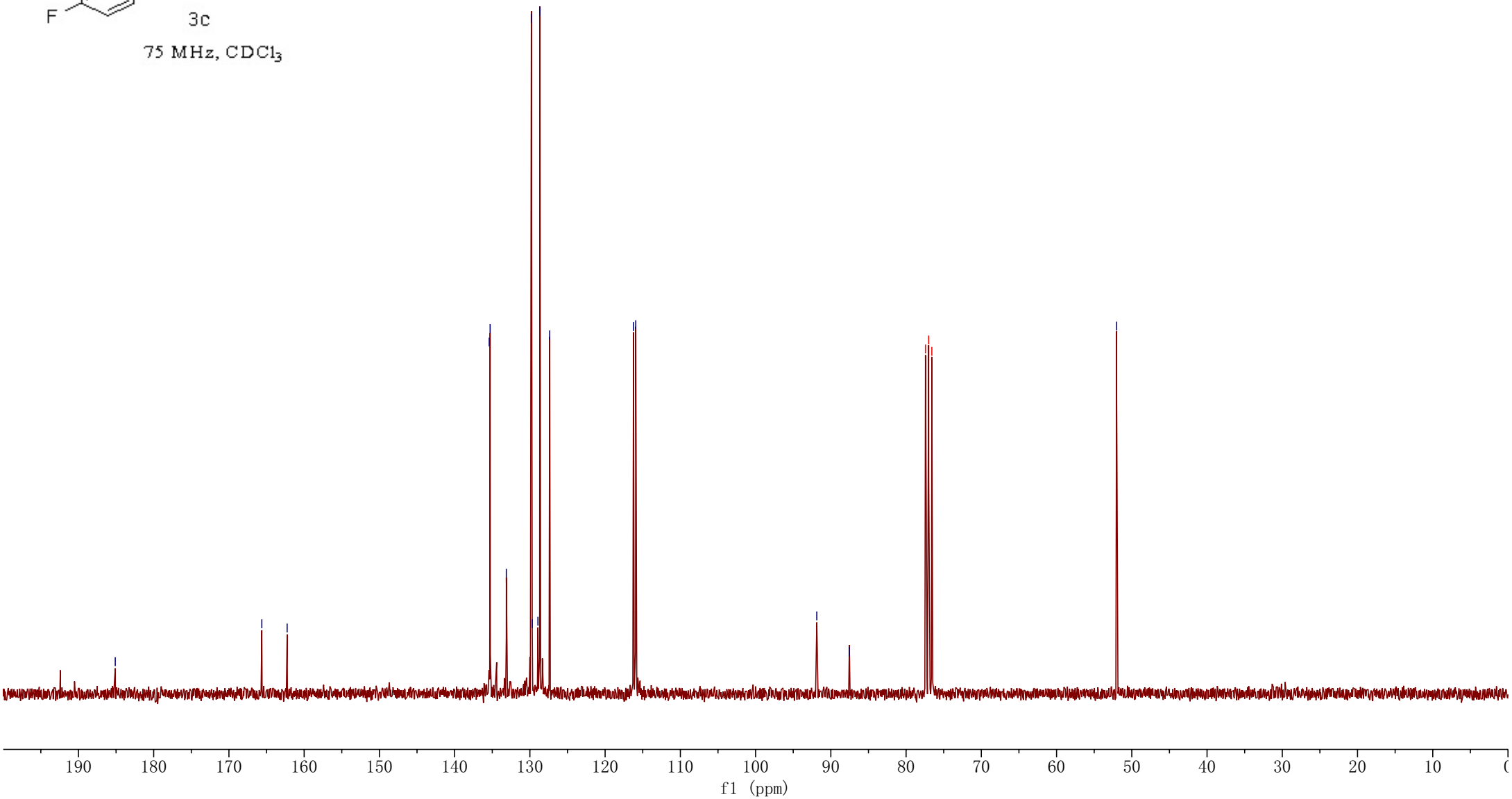
4.1.fid



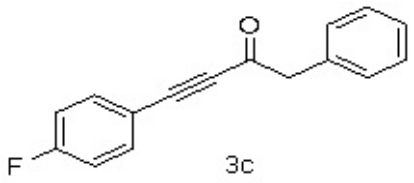
3c

75 MHz, $CDCl_3$

185.1
165.6
162.3
135.4
135.3
133.1
129.8
129.7
128.9
128.7
127.4
116.2
115.9
91.9
87.5
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.0



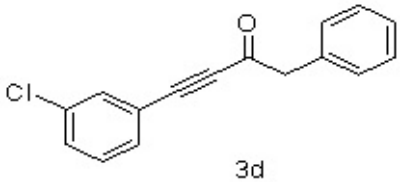
0929-4.1.fid



3c

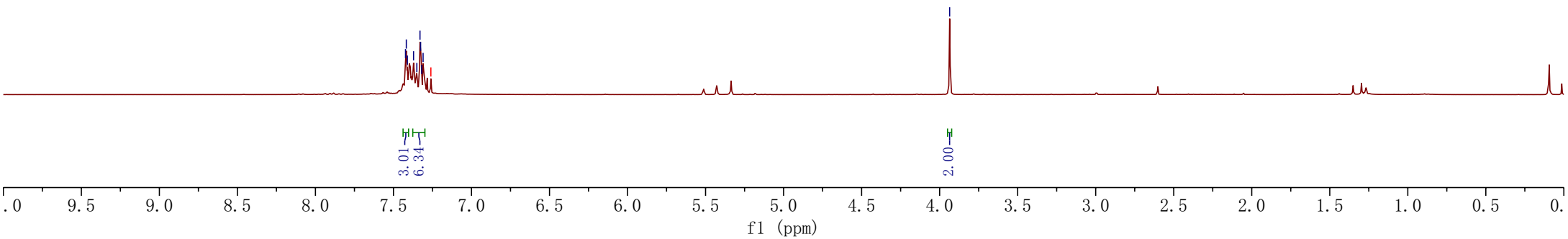
282 MHz, $CDCl_3$

1014-32.1.fid

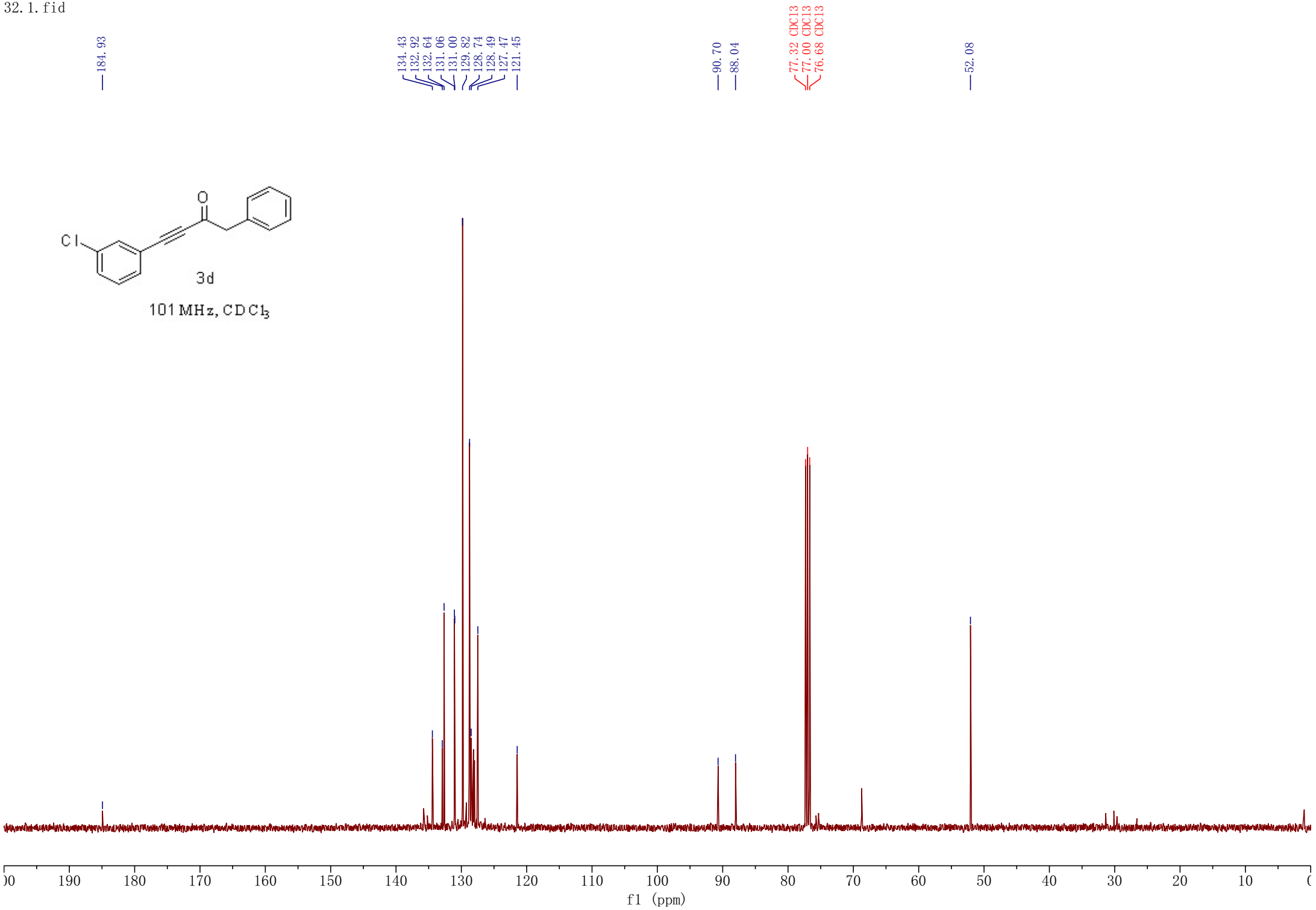


3d

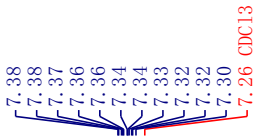
300 MHz, $CDCl_3$

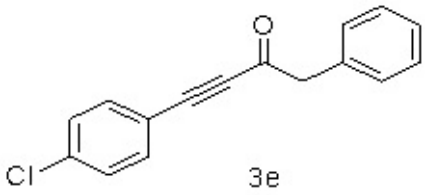


32.1.fid


184.93
134.43
132.92
132.64
131.06
131.00
129.82
128.74
128.49
127.47
121.45
90.70
88.04
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
52.08
Cl
O
3d
101 MHz, CDCl3
f1 (ppm)

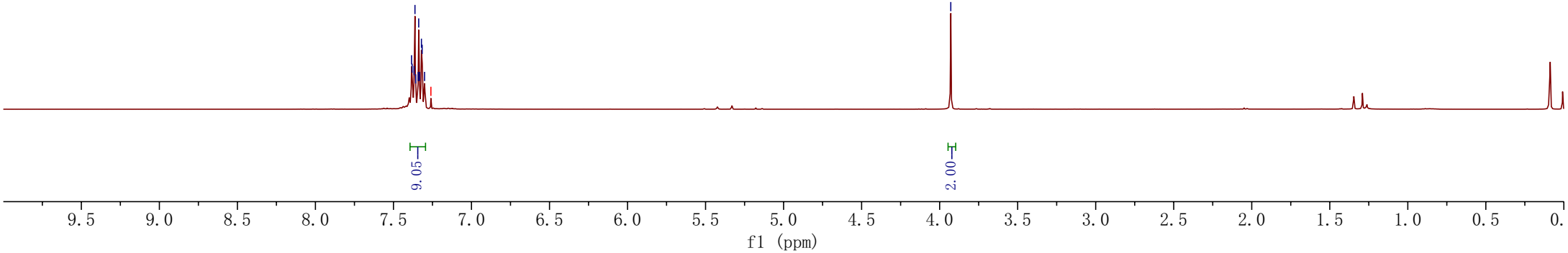
0922-3.1.fid



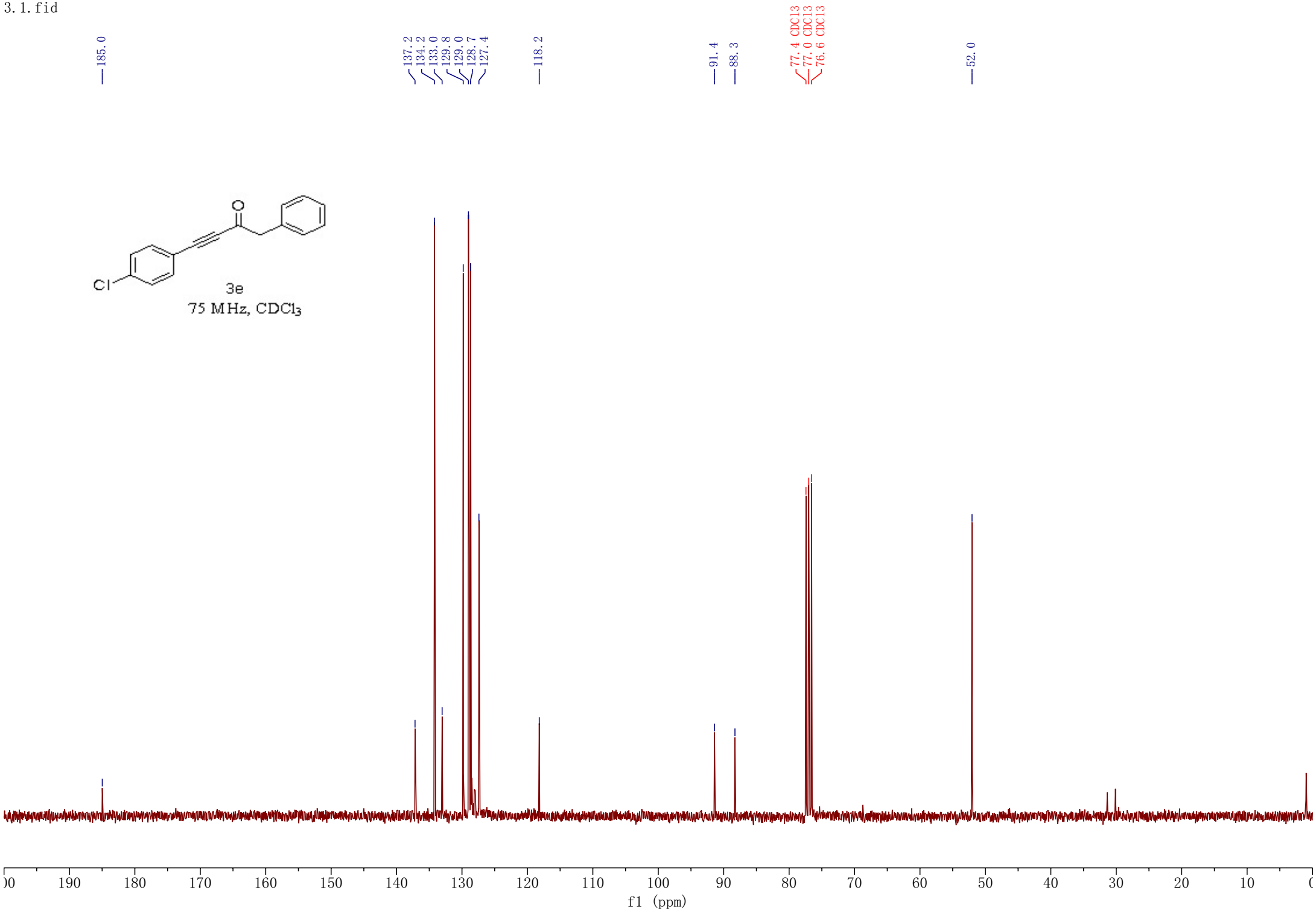


3e

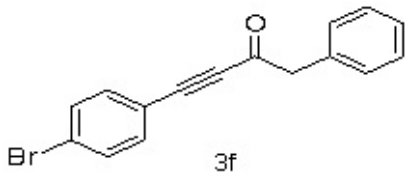
400 MHz, $CDCl_3$



3.1.fid

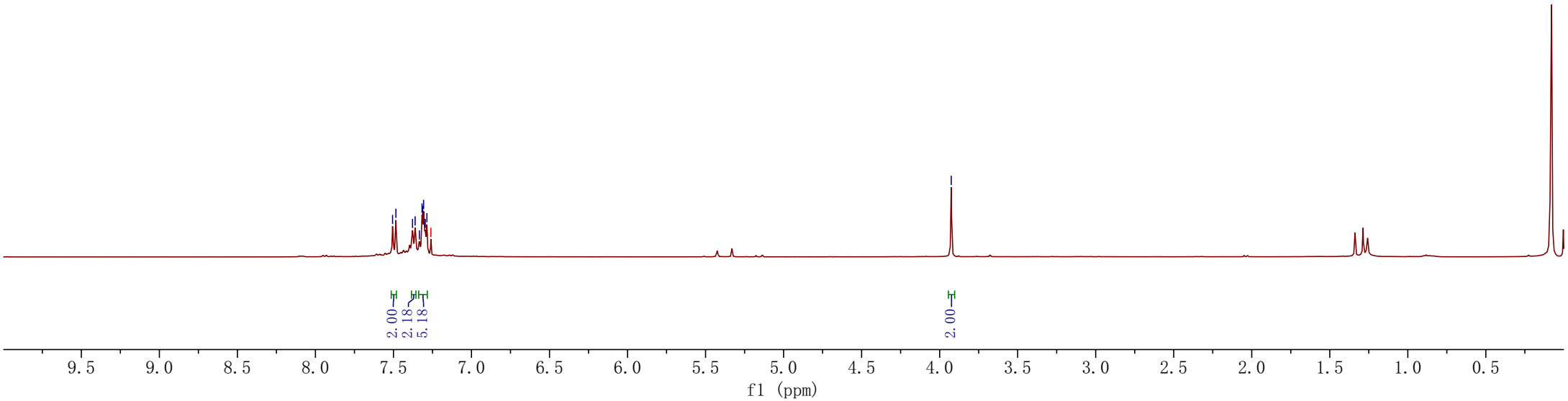

185.0
137.2
134.2
133.0
129.8
129.0
128.7
127.4
118.2
91.4
88.3
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.0
Cl
3e
75 MHz, CDCl3
f1 (ppm)

0922-2.1.fid

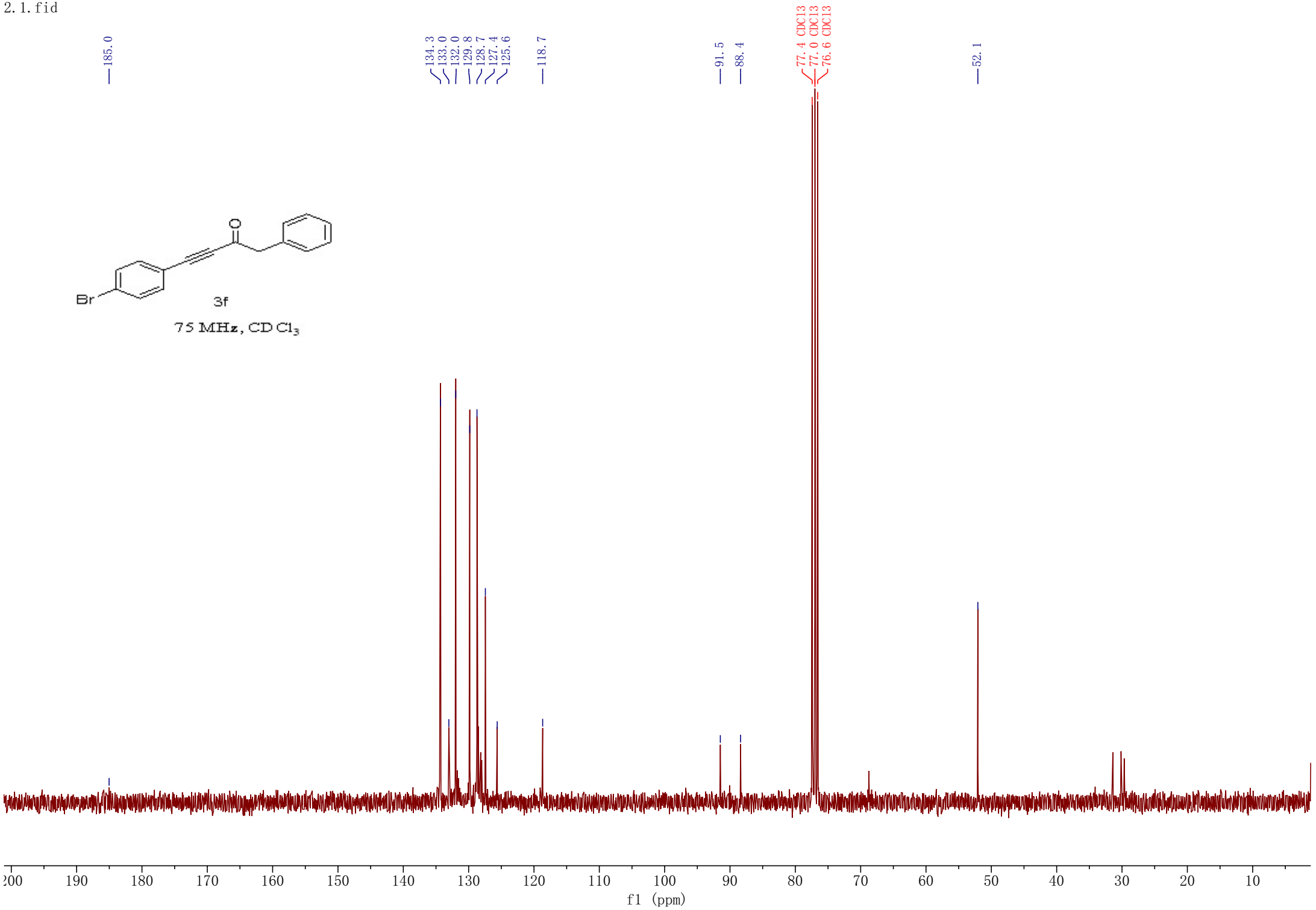


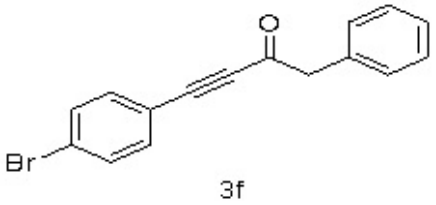
3f

400 MHz, $CDCl_3$



2.1.fid


185.0
134.3
133.0
132.0
129.8
128.7
127.4
125.6
118.7
91.5
88.4
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.1
Br
O
3f
75 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



3f

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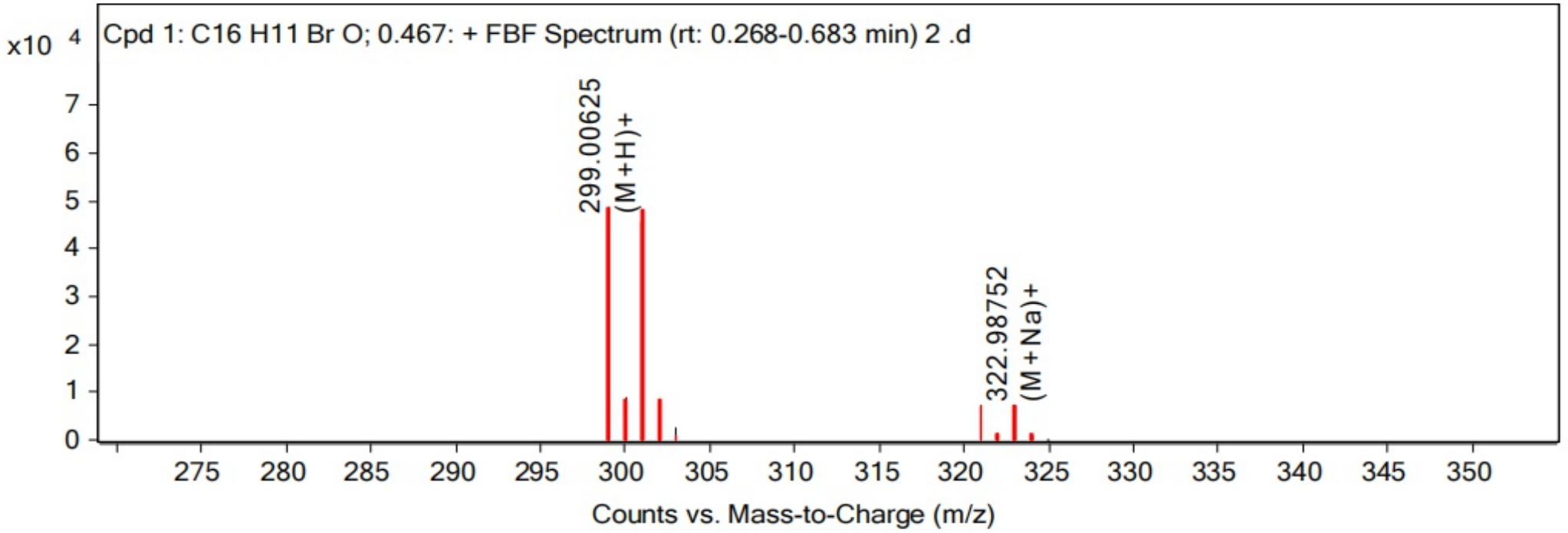
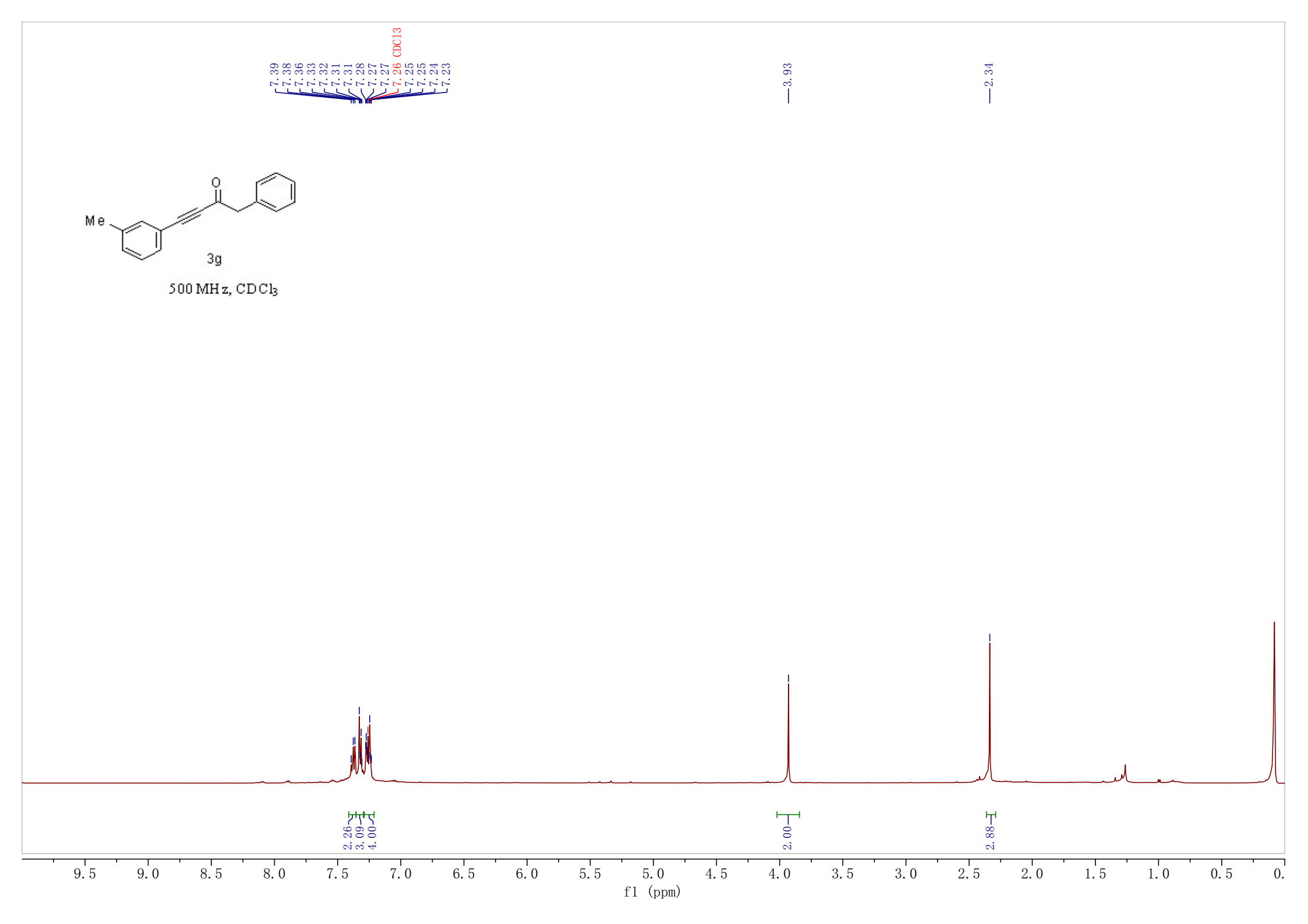


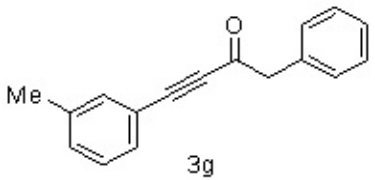
MS Spectrum Peak List

m/z	z	Abund	Ion
299.00625	1	48766.16	(M+H)+
300.00932	1	8866.74	(M+H)+
301.00439	1	45860.39	(M+H)+
320.98888	1	6959.93	(M+Na)+
322.98752	1	7161.81	(M+Na)+

MS Spectrum


7.39
7.38
7.36
7.33
7.32
7.31
7.31
7.28
7.27
7.27
7.26 CDC13
7.25
7.25
7.24
7.23
3.93
2.34
Me
O
3g
500 MHz, CDCl3
2.26
3.09
4.00
2.00
2.88
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

24.1.fid

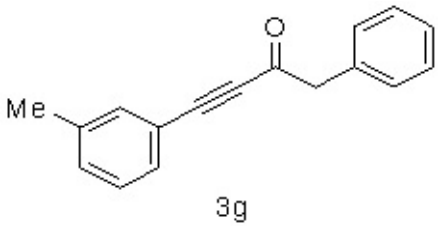


3g

75 MHz, $CDCl_3$

185.2
138.4
133.6
133.3
131.7
130.2
129.8
128.7
128.4
127.3
119.6
93.3
87.5
77.4 CDC13
77.0 CDC13
76.6 CDC13
52.1
21.1

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10



3g

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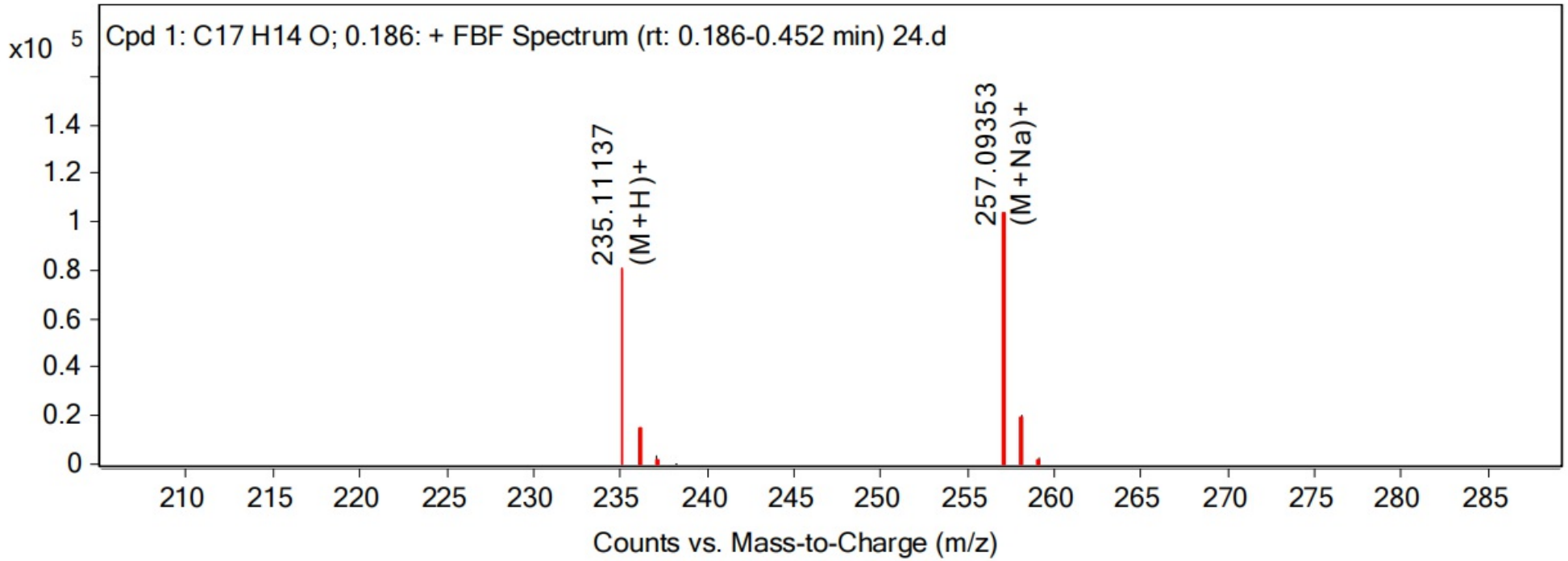
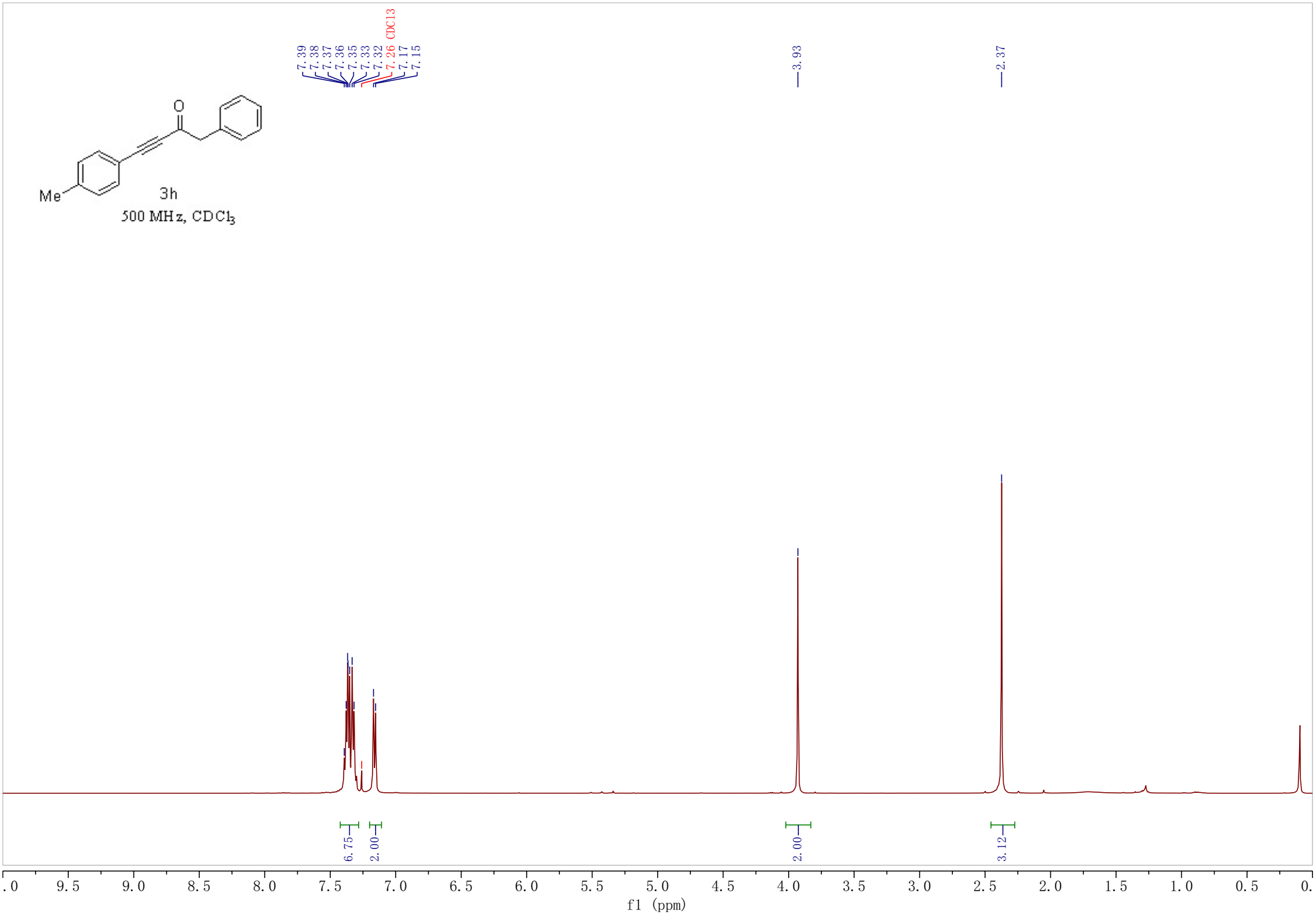


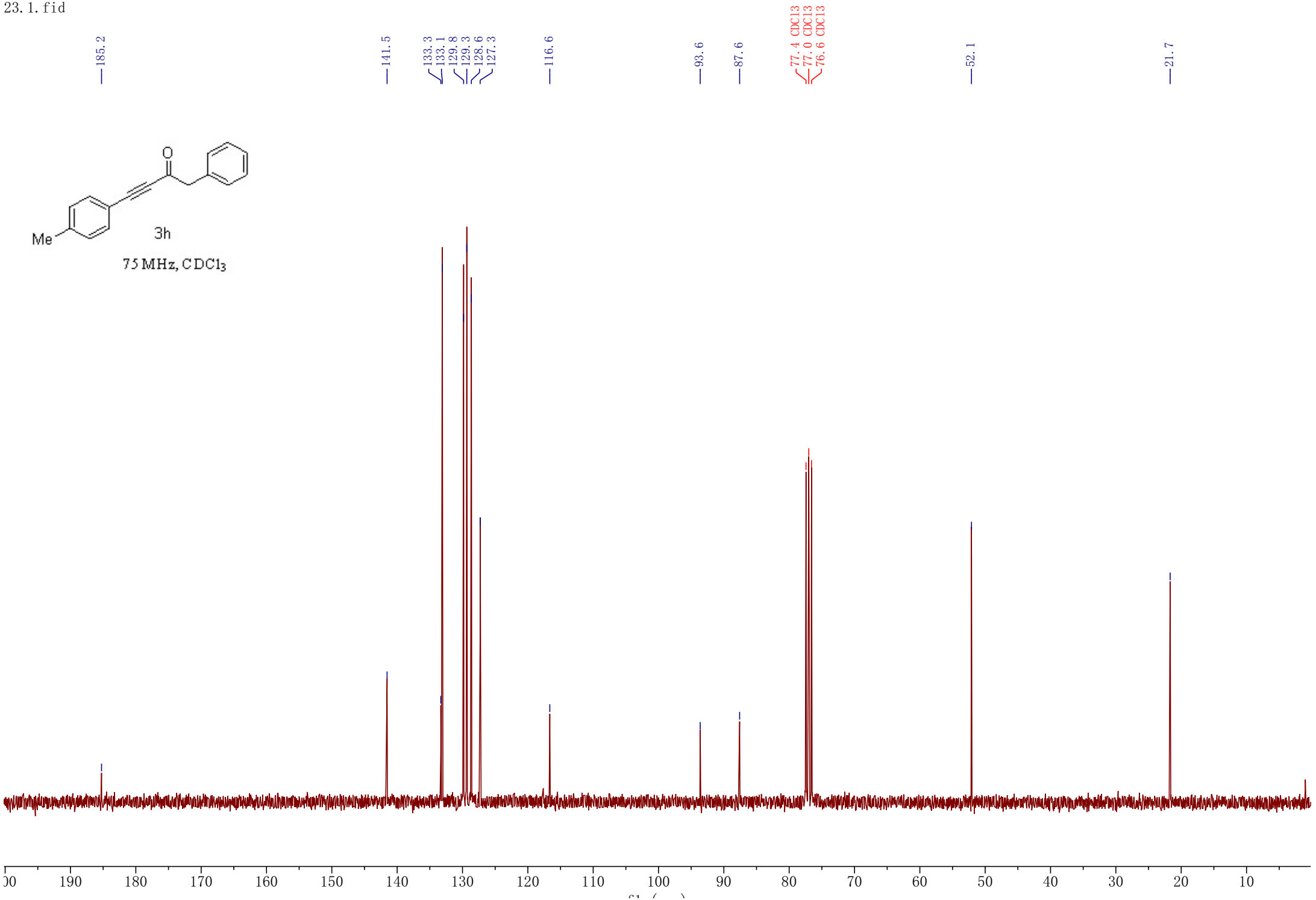
MS Spectrum Peak List

m/z	z	Abund	Ion
235.11137	1	79699.05	(M+H)+
236.11491	1	14479.72	(M+H)+
237.1218	1	3389.8	(M+H)+
238.12631	1	95.21	(M+H)+
257.09353	1	101285.04	(M+Na)+
258.09667	1	20682.38	(M+Na)+
259.11077	1	2868.3	(M+Na)+

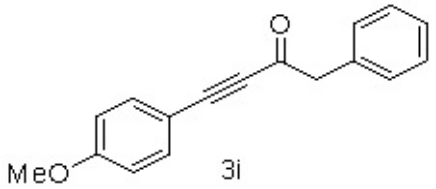
MS Spectrum


7.39
7.38
7.37
7.36
7.35
7.33
7.32
7.26 CDC13
7.17
7.15
3.93
2.37
O
Me
3h
500 MHz, CDCl3
6.75
2.00
2.00
3.12
f1 (ppm)

23.1.fid

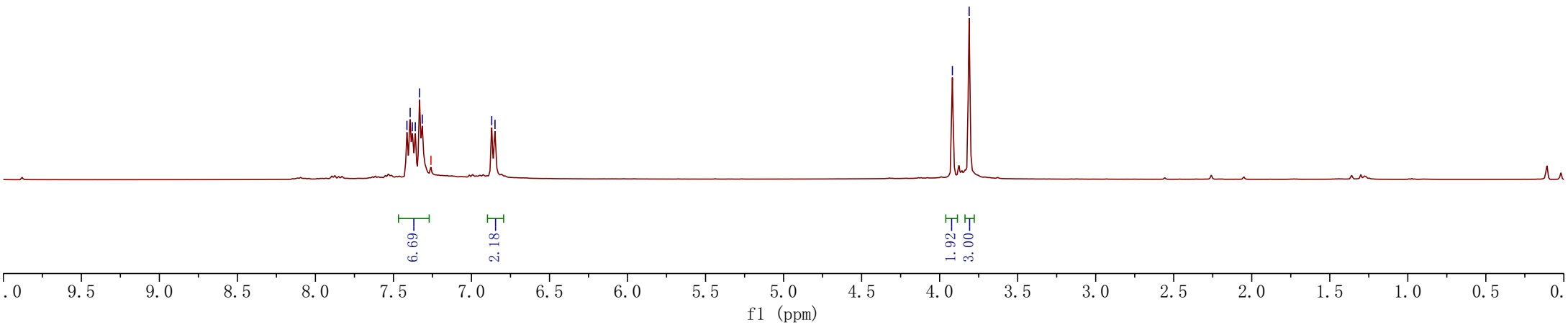

185.2
141.5
133.3
133.1
129.8
129.3
128.6
127.3
116.6
93.6
87.6
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.1
21.7
O
Me
3h
75 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

0922-6.1.fid

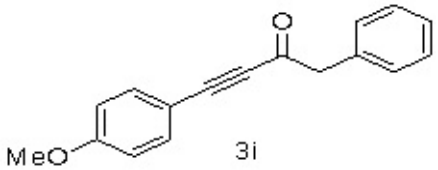


3i

400MHz, $CDCl_3$



6.1.fid



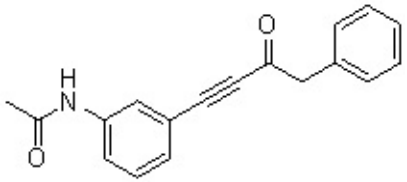
3i

75 MHz, $CDCl_3$

185.2
161.6
135.1
133.4
129.8
128.6
127.2
114.2
111.4
94.2
87.6
77.4 CDC13
77.0 CDC13
76.6 CDC13
55.3
51.9

f1 (ppm)

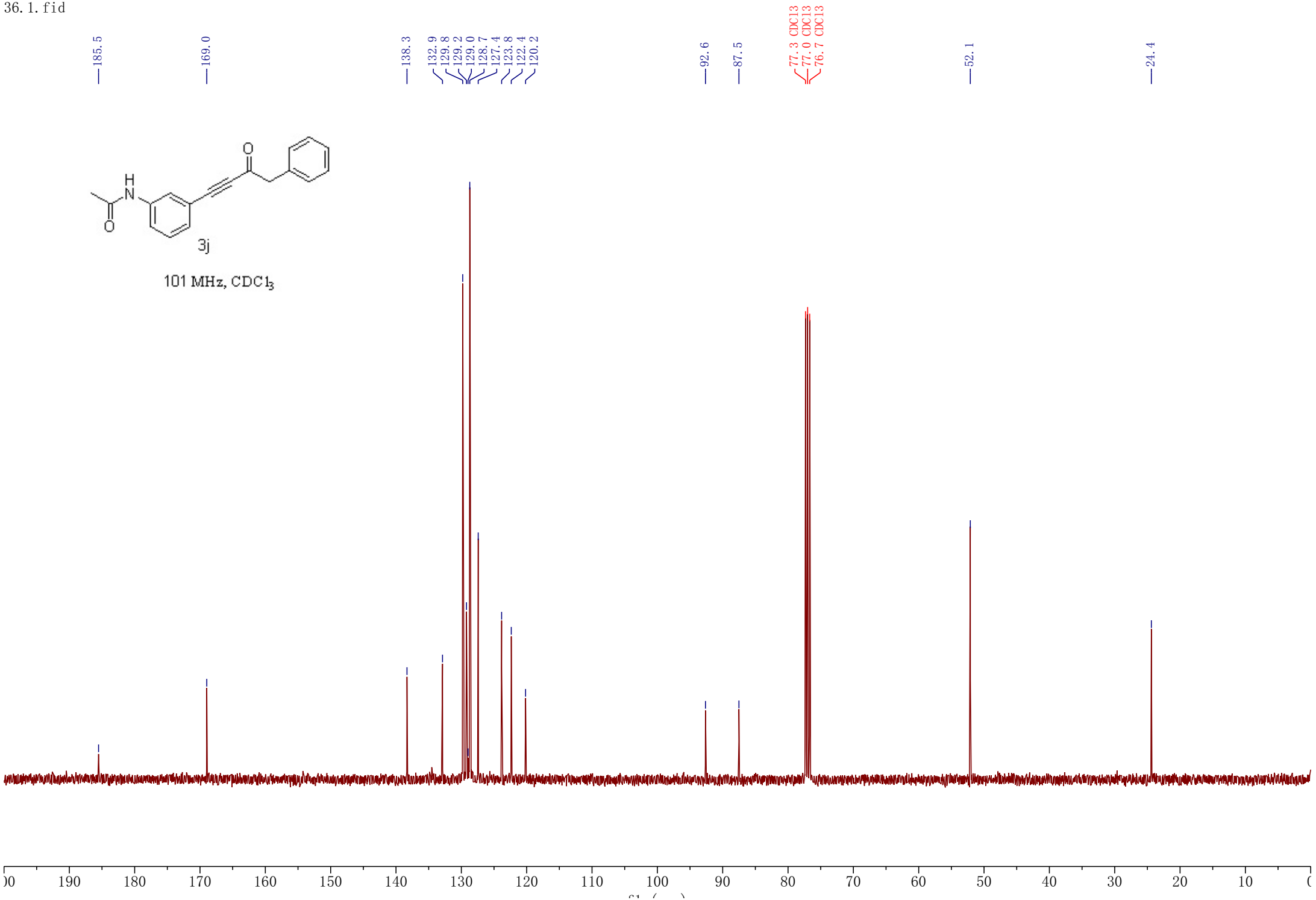
1014-36.1.fid

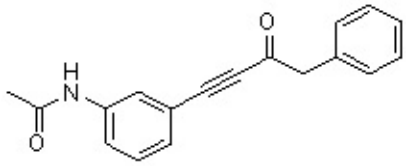


3j

300 MHz, $CDCl_3$

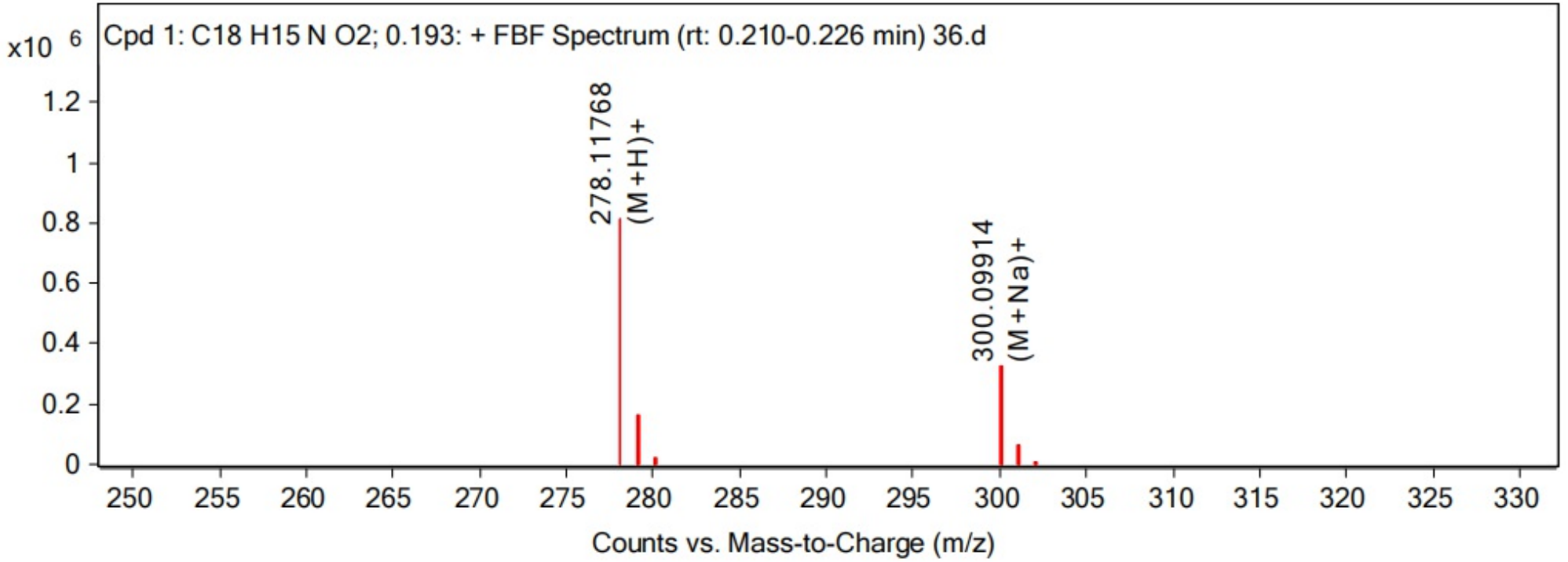
36.1.fid


185.5
169.0
138.3
132.9
129.8
129.2
129.0
128.7
127.4
123.8
122.4
120.2
92.6
87.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
52.1
24.4
O
H
N
O
3j
101 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10



3j

Qualitative Compound Identification Report

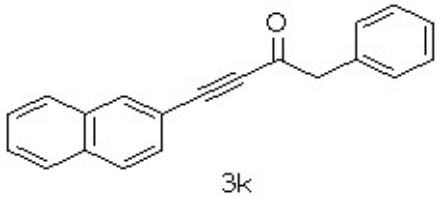


MS Spectrum Peak List

m/z	z	Abund	Ion
278.11768	1	817529.38	(M+H)+
279.12113	1	160107.97	(M+H)+
280.12361	1	18072.93	(M+H)+
300.09914	1	322284.22	(M+Na)+
301.10252	1	65733.13	(M+Na)+
302.10632	1	5914.02	(M+Na)+

MS Spectrum

0922-8.1.fid

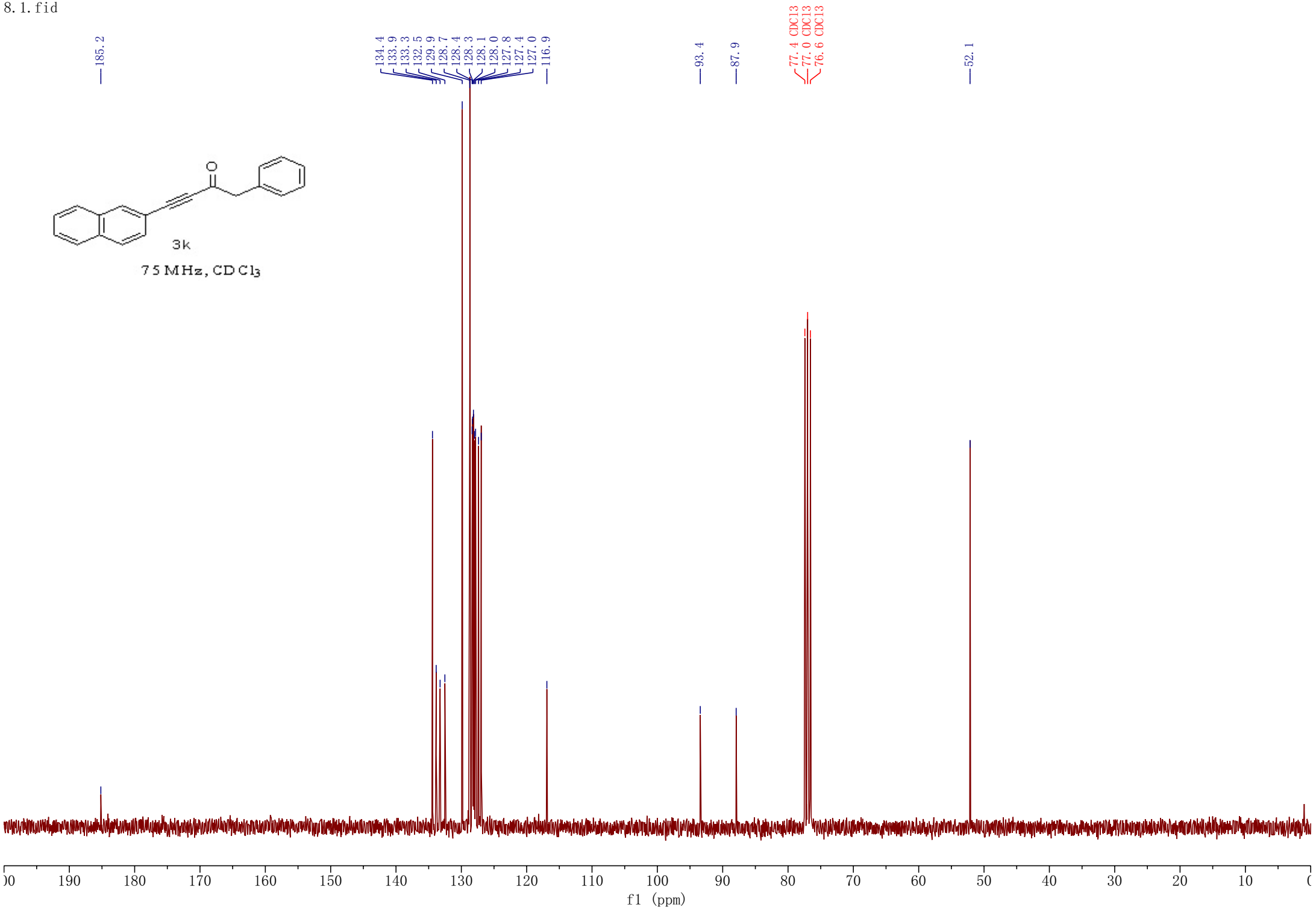


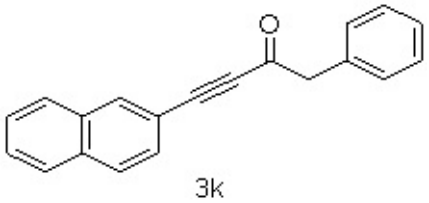
3k

400MHz, $CDCl_3$

f1 (ppm)

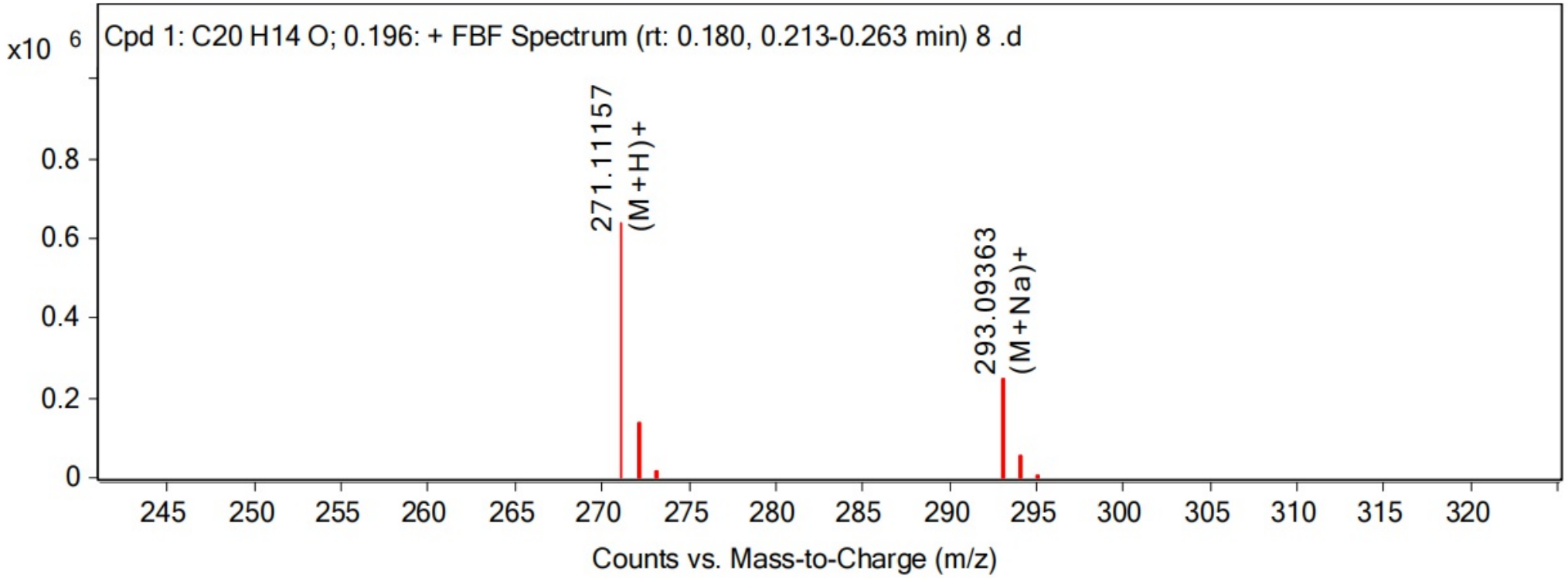
8.1.fid


185.2
134.4
133.9
133.3
132.5
129.9
128.7
128.4
128.3
128.1
128.0
127.8
127.4
127.0
116.9
93.4
87.9
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
52.1
3k
75MHz, CDCl3
f1 (ppm)



3k

Qualitative Compound Identification Report

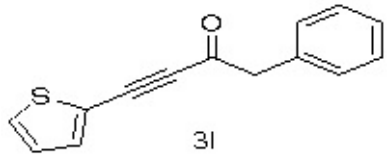


MS Spectrum Peak List

m/z	z	Abund	Ion
271.11157	1	636474.31	(M+H)+
272.11511	1	137266.97	(M+H)+
273.11928	1	19305.97	(M+H)+
293.09363	1	245427.47	(M+Na)+
294.09693	1	56400.73	(M+Na)+

MS Spectrum

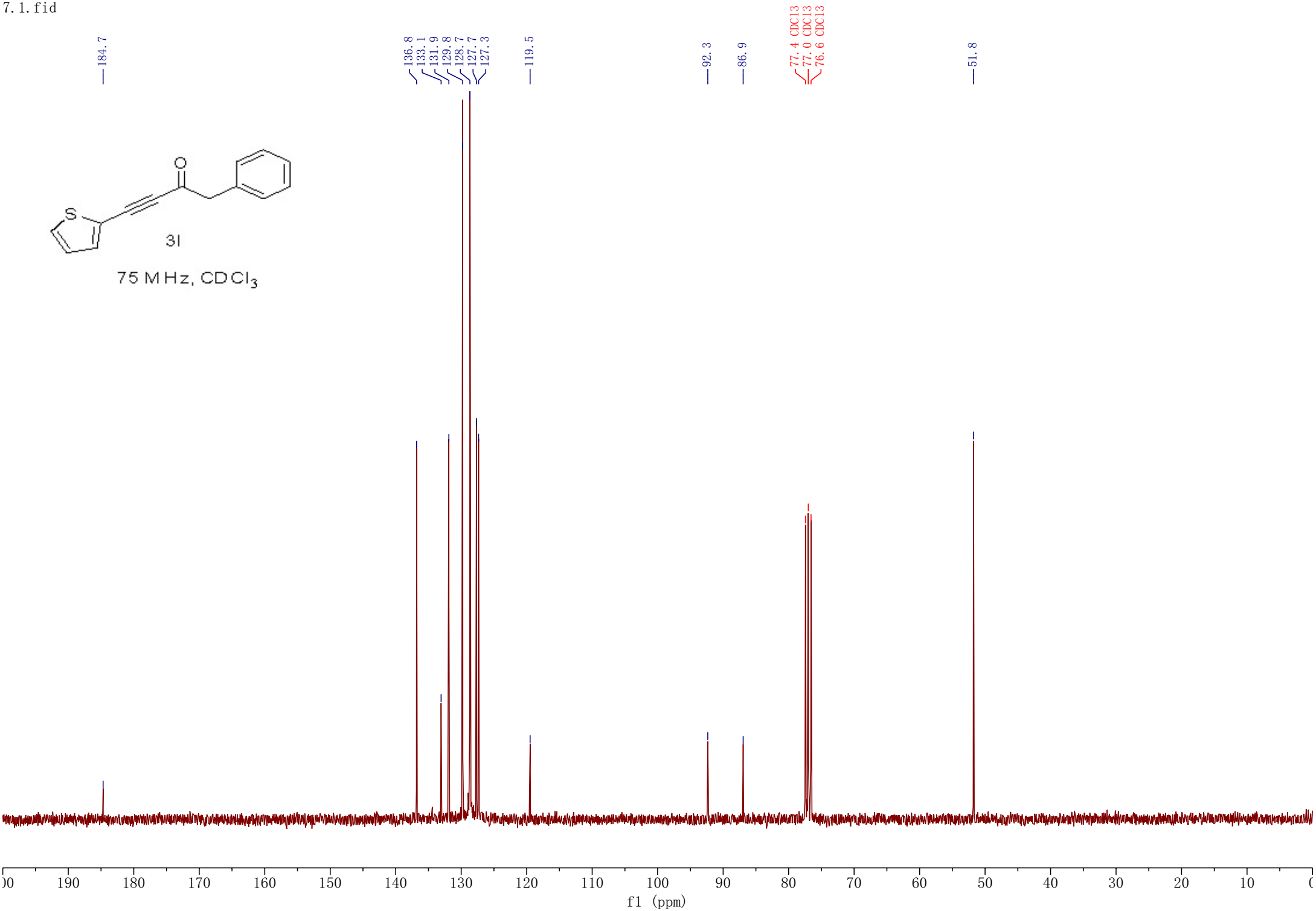
0922-7.1.fid

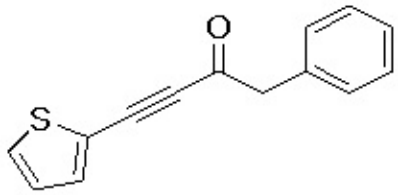


3l

400 MHz, $CDCl_3$

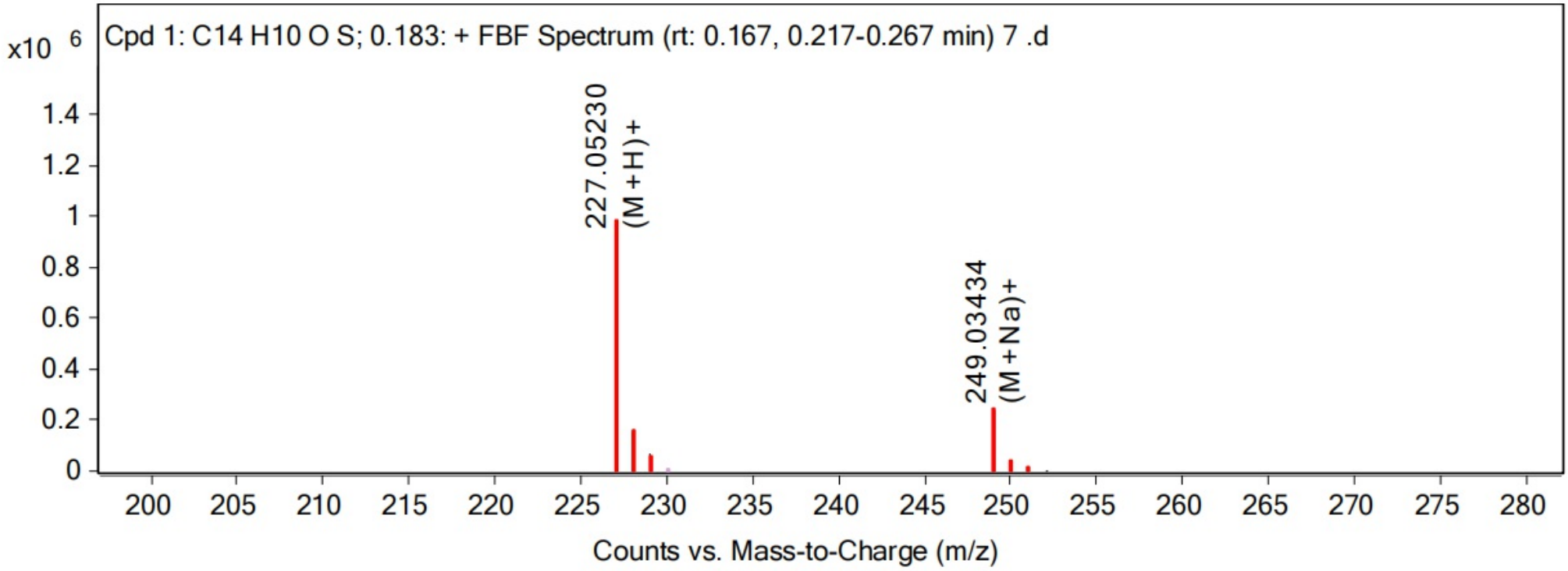
7.1.fid


184.7
136.8
133.1
131.9
129.8
128.7
127.7
127.3
119.5
92.3
86.9
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
51.8
O
S
3l
75 MHz, CDCl3
f1 (ppm)



3l

Qualitative Compound Identification Report

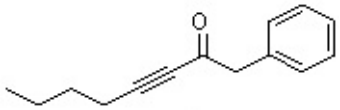


MS Spectrum Peak List

m/z	z	Abund	Ion
227.0523	1	978546.69	(M+H)+
228.0557	1	152648.23	(M+H)+
229.05111	1	65686.73	(M+H)+
249.03434	1	242287.17	(M+Na)+
250.03749	1	40333.09	(M+Na)+

MS Spectrum

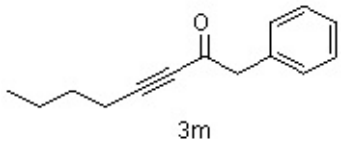
1014-35.1.fid



3m

300 MHz, $CDCl_3$

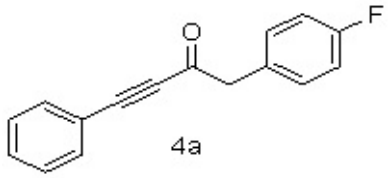
35.1.fid



3m

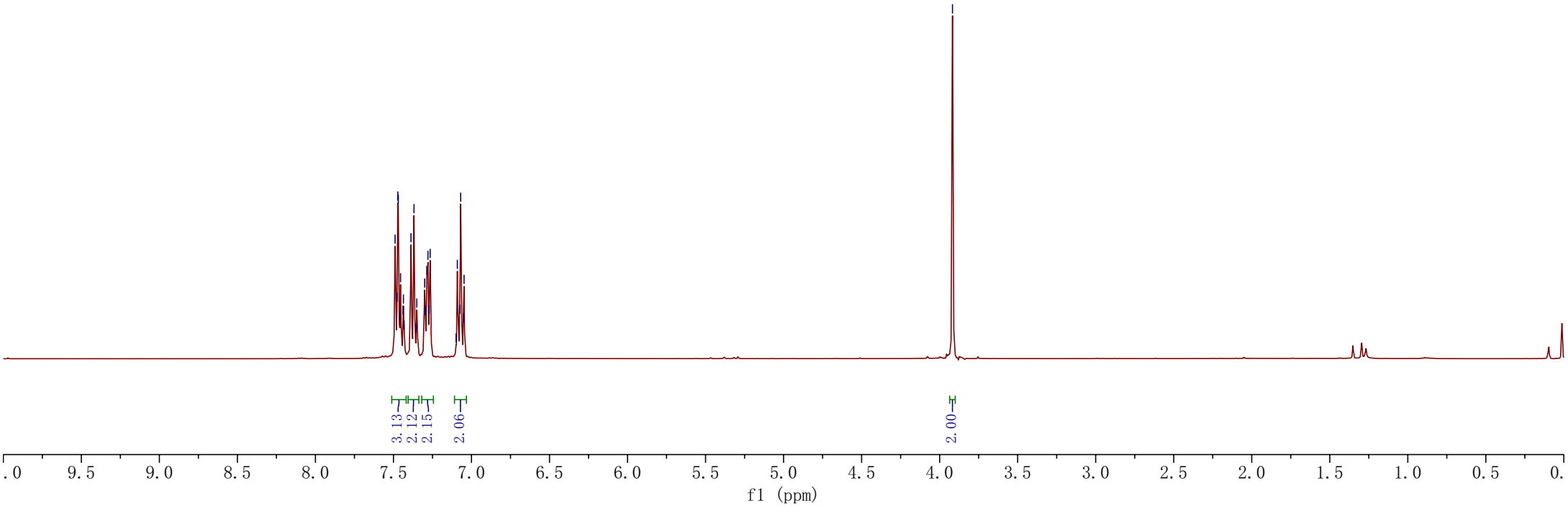
101MHz,$CDCl_3$

0922-11.1.fid

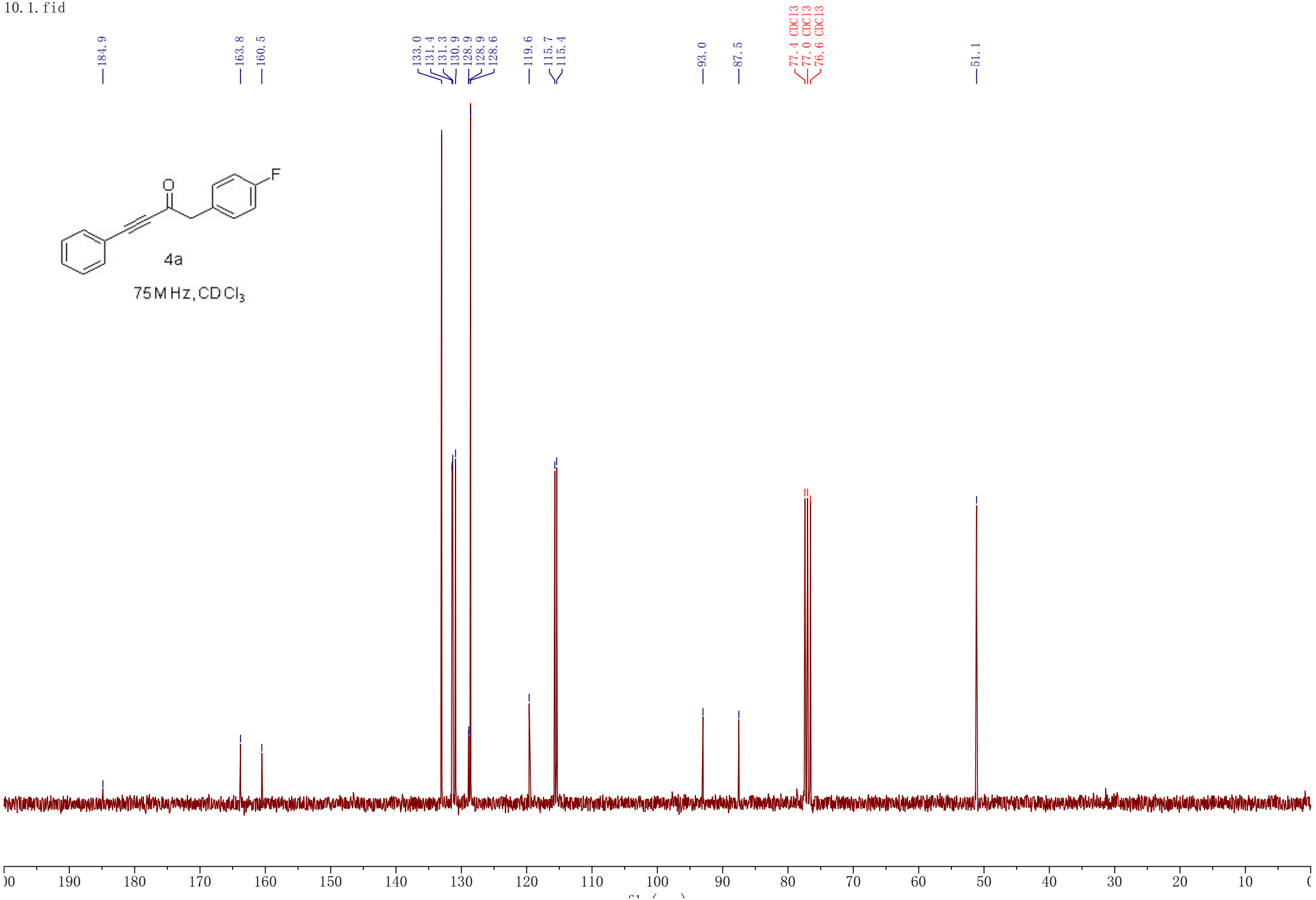


4a

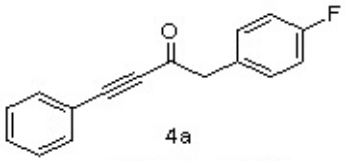
400 MHz, $CDCl_3$



10.1.fid


184.9
163.8
160.5
133.0
131.4
131.3
130.9
128.9
128.9
128.6
119.6
115.7
115.4
93.0
87.5
77.4 CDC13
77.0 CDC13
76.6 CDC13
51.1
O
F
4a
75MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

1023-10.1.fid



4a

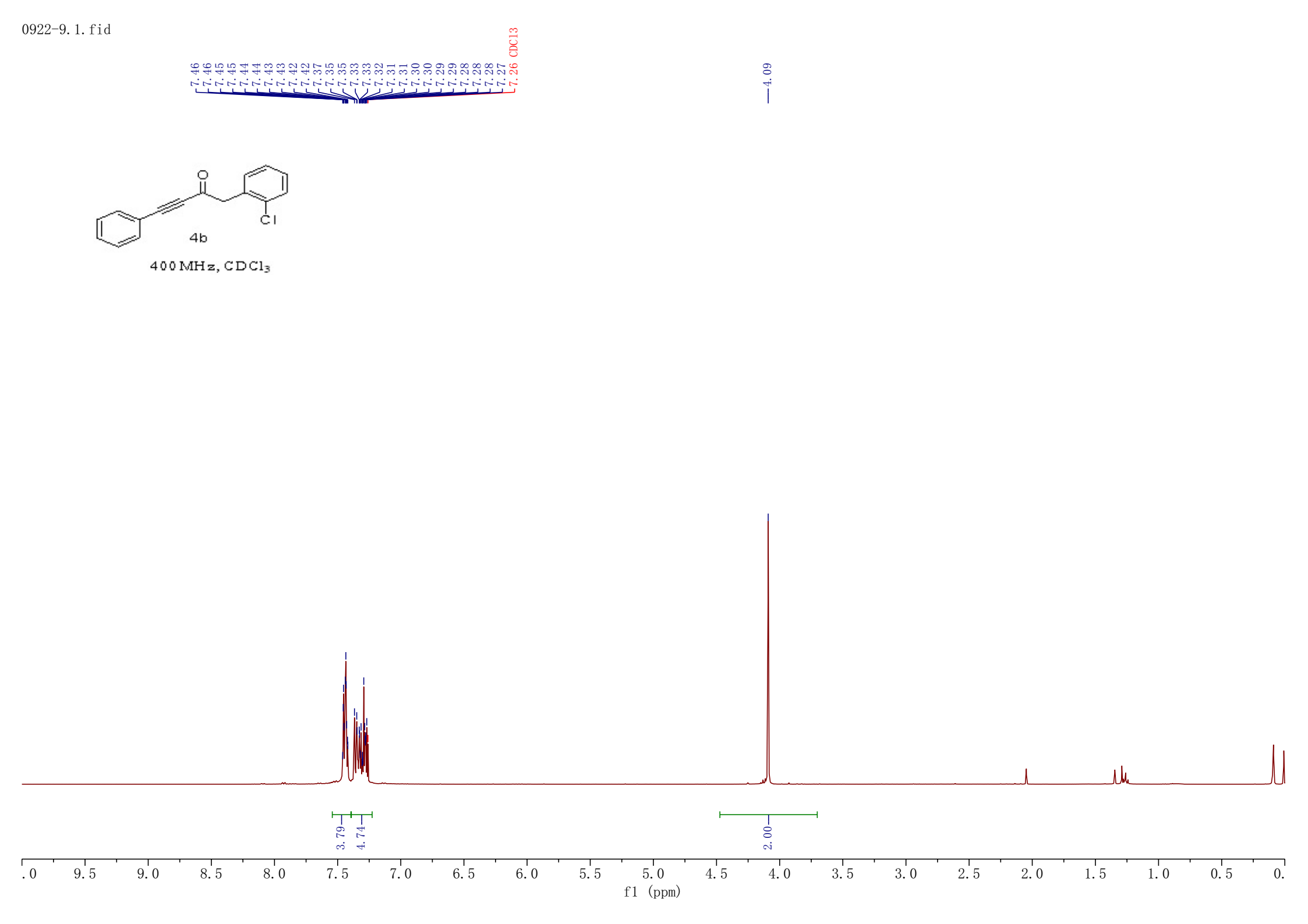
565 MHz, $CDCl_3$

−115.15

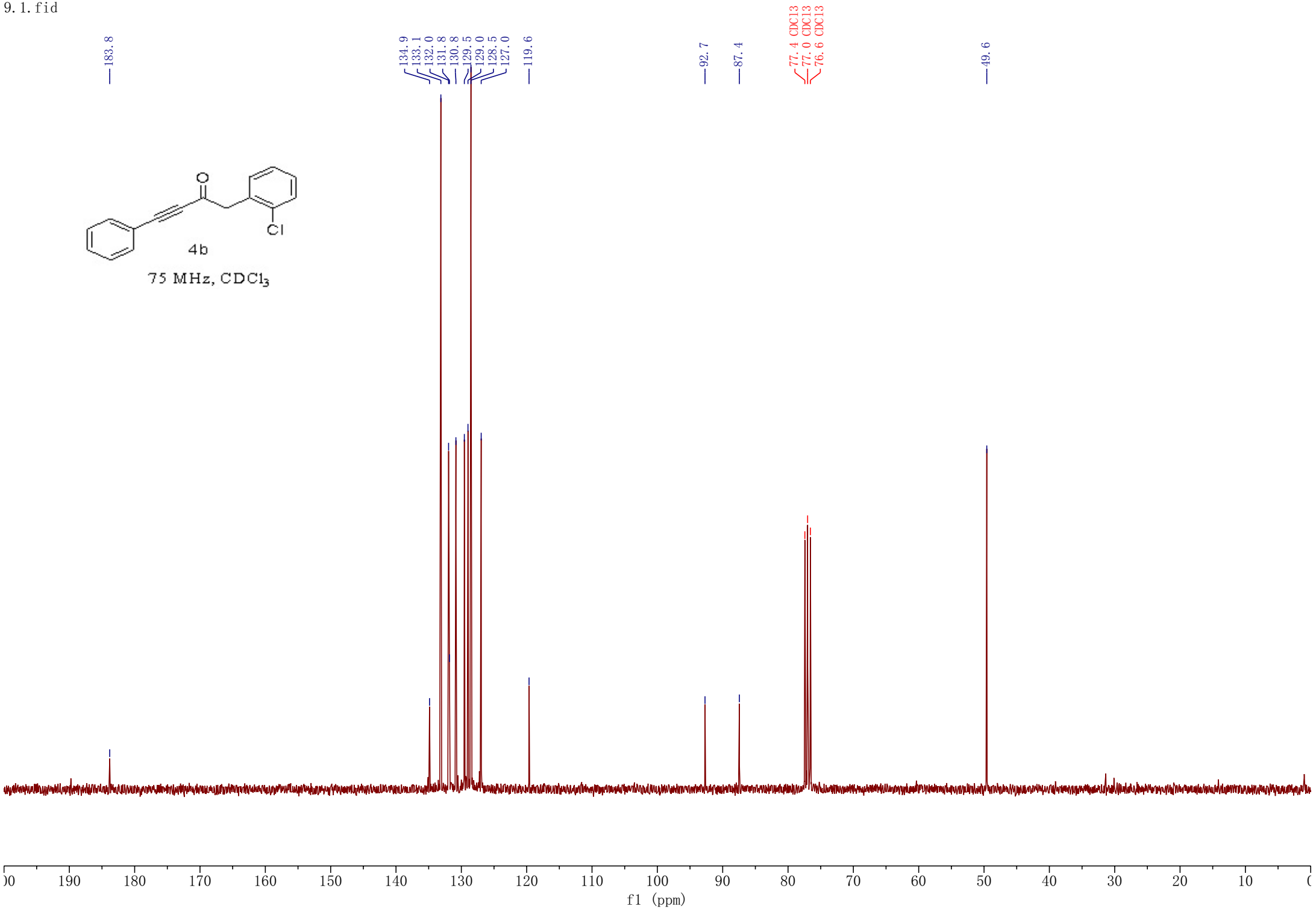
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210

f1 (ppm)

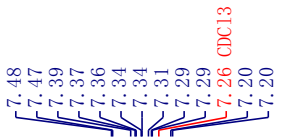
0922-9.1.fid

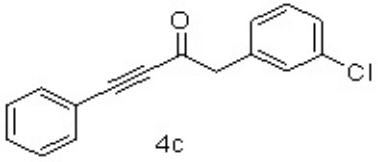

7.46
7.46
7.45
7.45
7.44
7.44
7.43
7.43
7.42
7.42
7.37
7.35
7.35
7.33
7.33
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.28
7.27
7.26 CDCl3
4.09
Cl
4b
400MHz, CDCl3
3.79
4.74
2.00
.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

9.1.fid


183.8
134.9
133.1
132.0
131.8
130.8
129.5
129.0
128.5
127.0
119.6
92.7
87.4
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
49.6
O
Cl
4b
75 MHz, CDCl3
f1 (ppm)

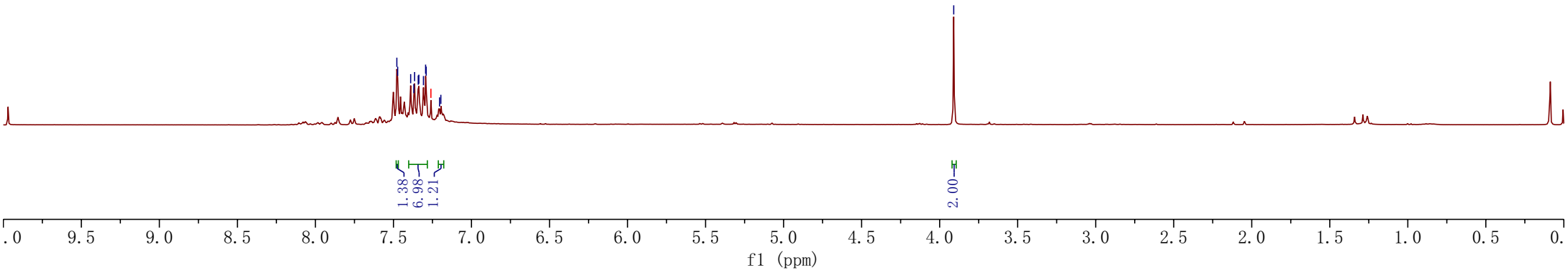
1014-42.1.fid



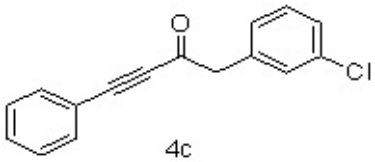


4c

300MHz, $CDCl_3$



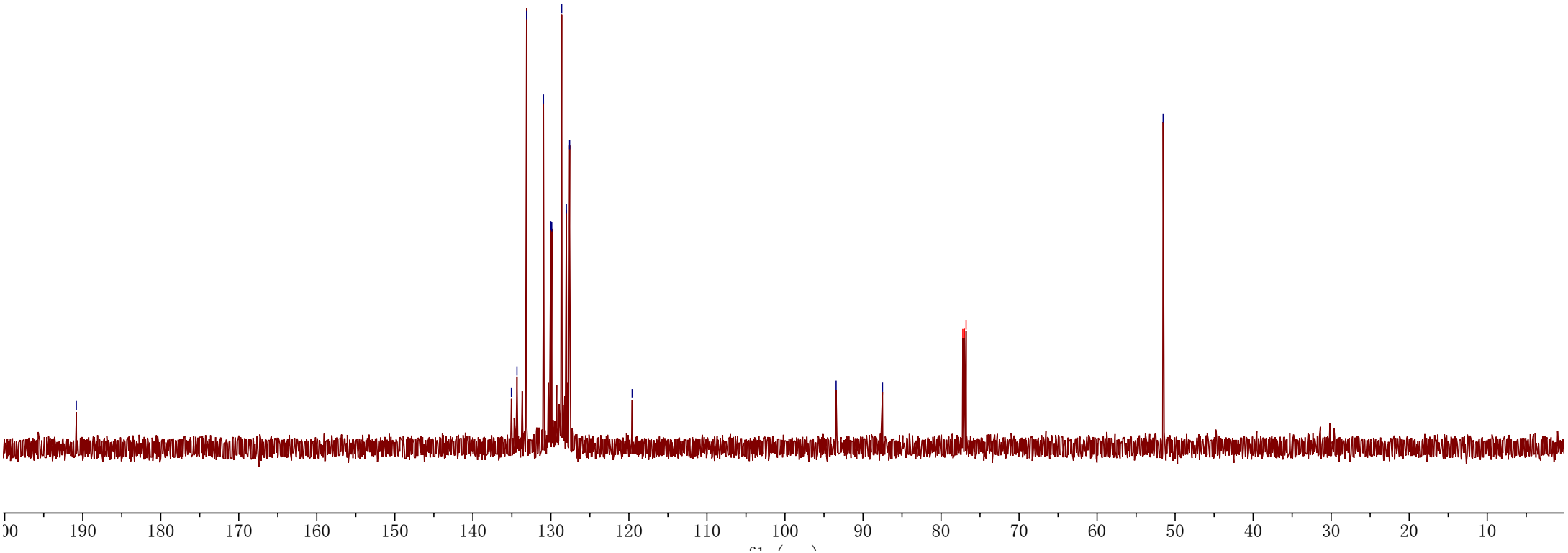
1023-42.1.fid



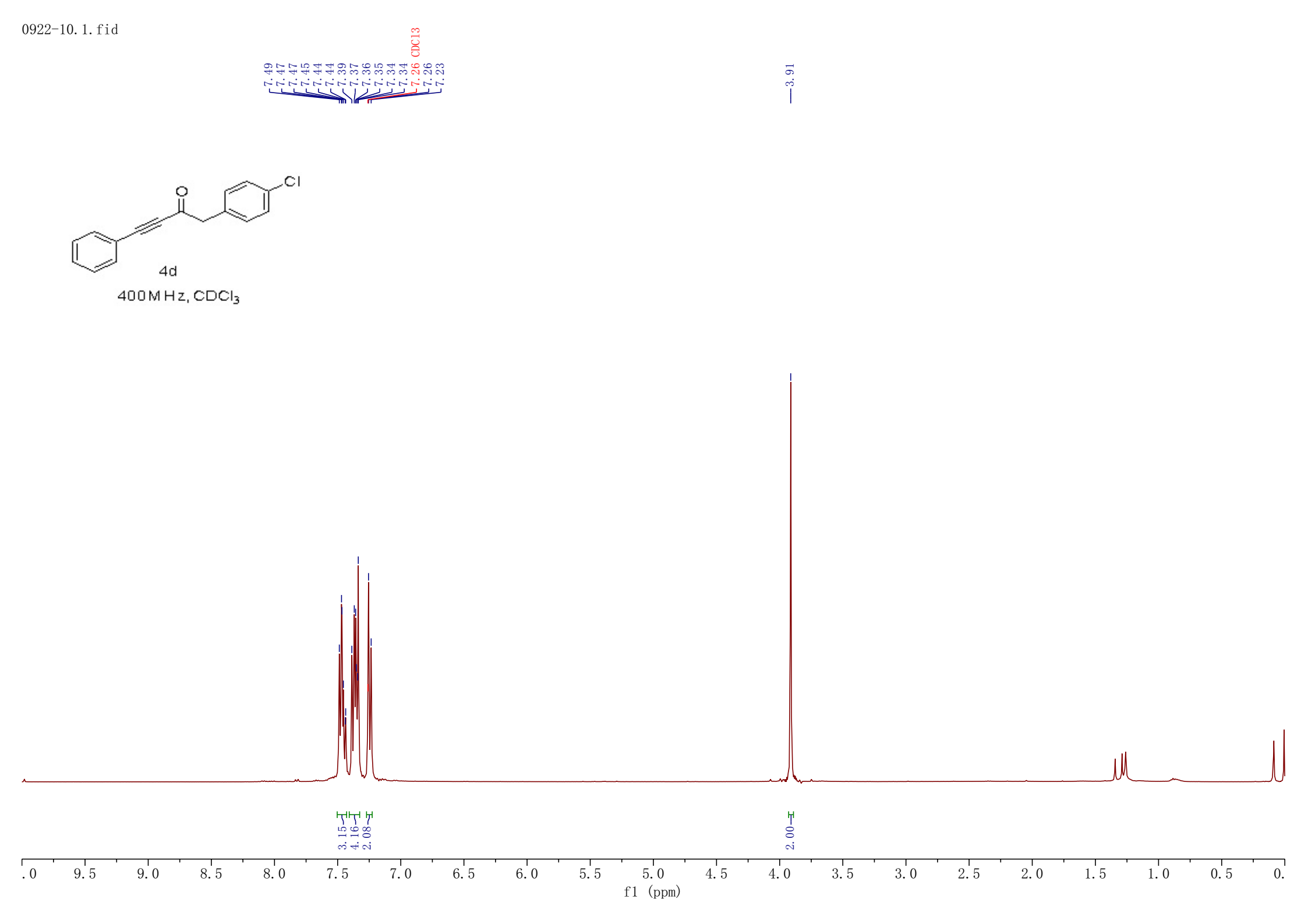
4c

151 MHz, $CDCl_3$

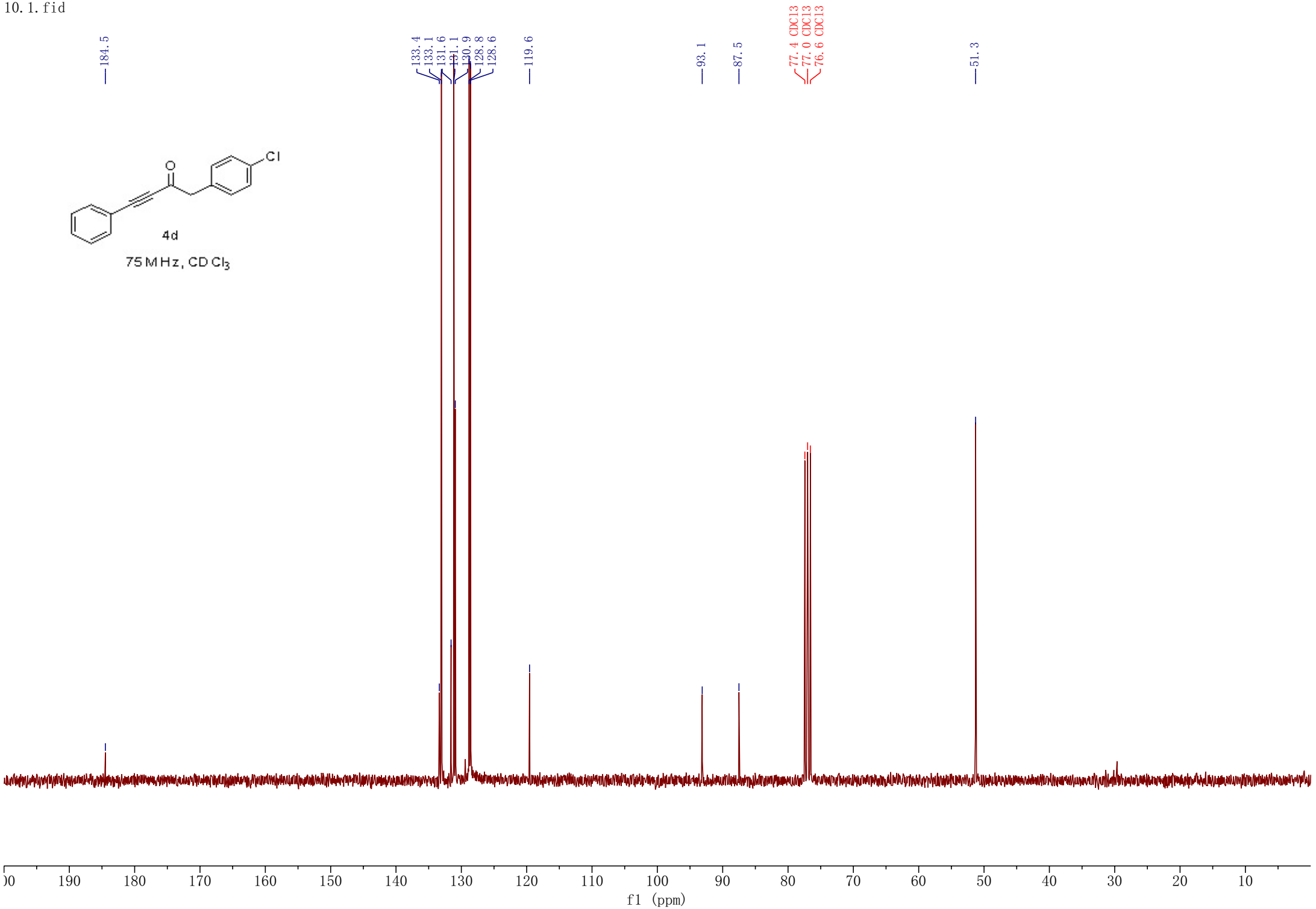
190.8
135.1
134.3
133.1
131.0
130.0
129.9
128.6
128.0
127.6
119.6
93.4
87.5
77.2 CDCl3
77.0 CDCl3
76.8 CDCl3
51.5



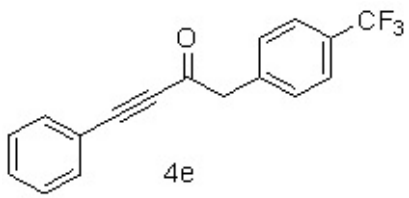
0922-10.1.fid


7.49
7.47
7.47
7.45
7.44
7.44
7.39
7.37
7.36
7.35
7.34
7.34
7.26 CDCl3
7.26
7.23
3.91
Cl
O
4d
400MHz, CDCl3
3.15
4.16
2.08
2.00
f1 (ppm)

10.1.fid


184.5
133.4
133.1
131.6
131.1
130.9
128.8
128.6
119.6
93.1
87.5
77.4 CDCl3
77.0 CDCl3
76.6 CDCl3
51.3
Cl
O
4d
75MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

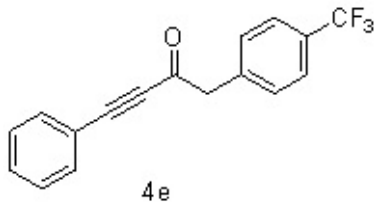
1014-39.1.fid



4e

300 MHz, $CDCl_3$

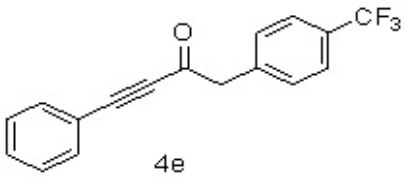
39.1.fid



4e

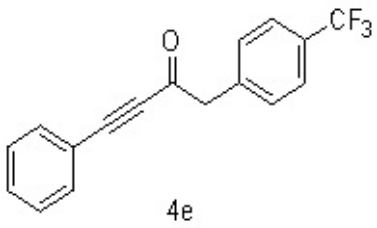
101 MHz, $CDCl_3$

1023-39-F.1.fid



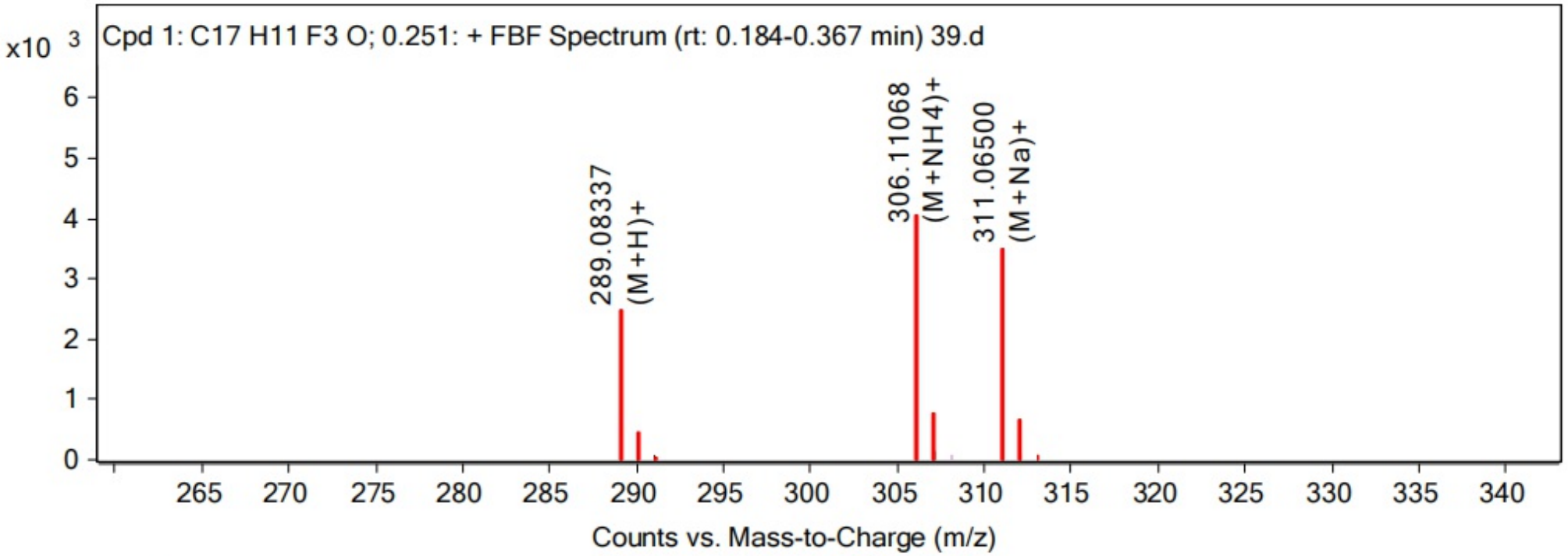
4e

565 MHz, $CDCl_3$



4e

Qualitative Compound Identification Report

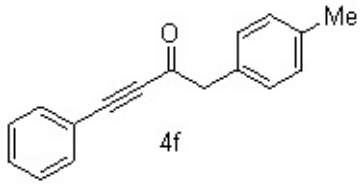


MS Spectrum Peak List

m/z	z	Abund	Ion
289.08337	1	2469.24	(M+H)+
290.08667	1	391.82	(M+H)+
291.09254	1	63.85	(M+H)+
306.11068	1	4051.53	(M+NH4)+
307.09978	1	144.11	(M+NH4)+
311.065	1	3509.6	(M+Na)+
312.06516	1	119.67	(M+Na)+
313.05999	1	81.35	(M+Na)+

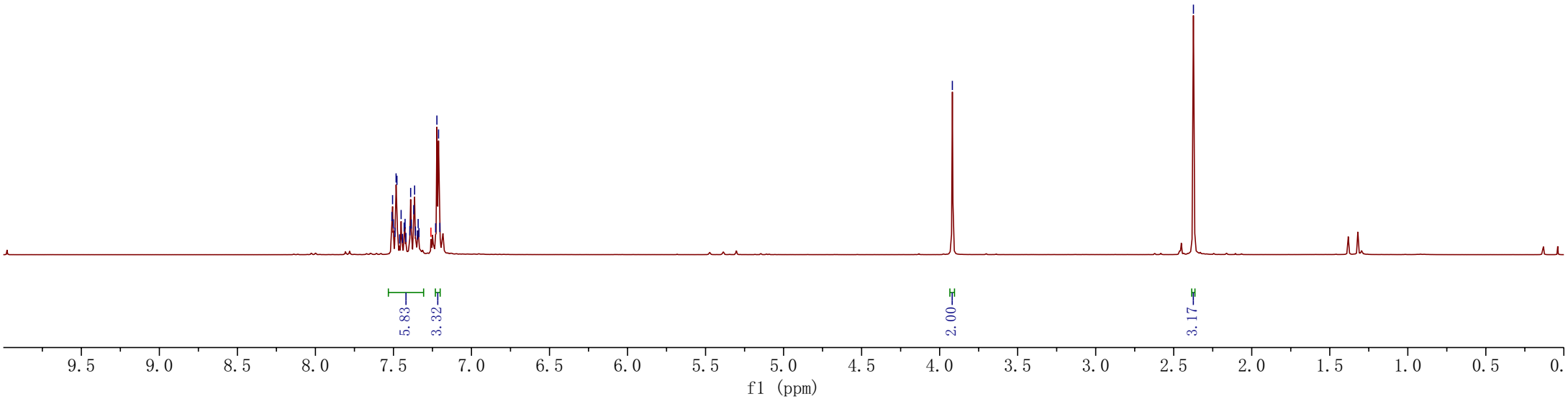
MS Spectrum

1014-37.1.fid

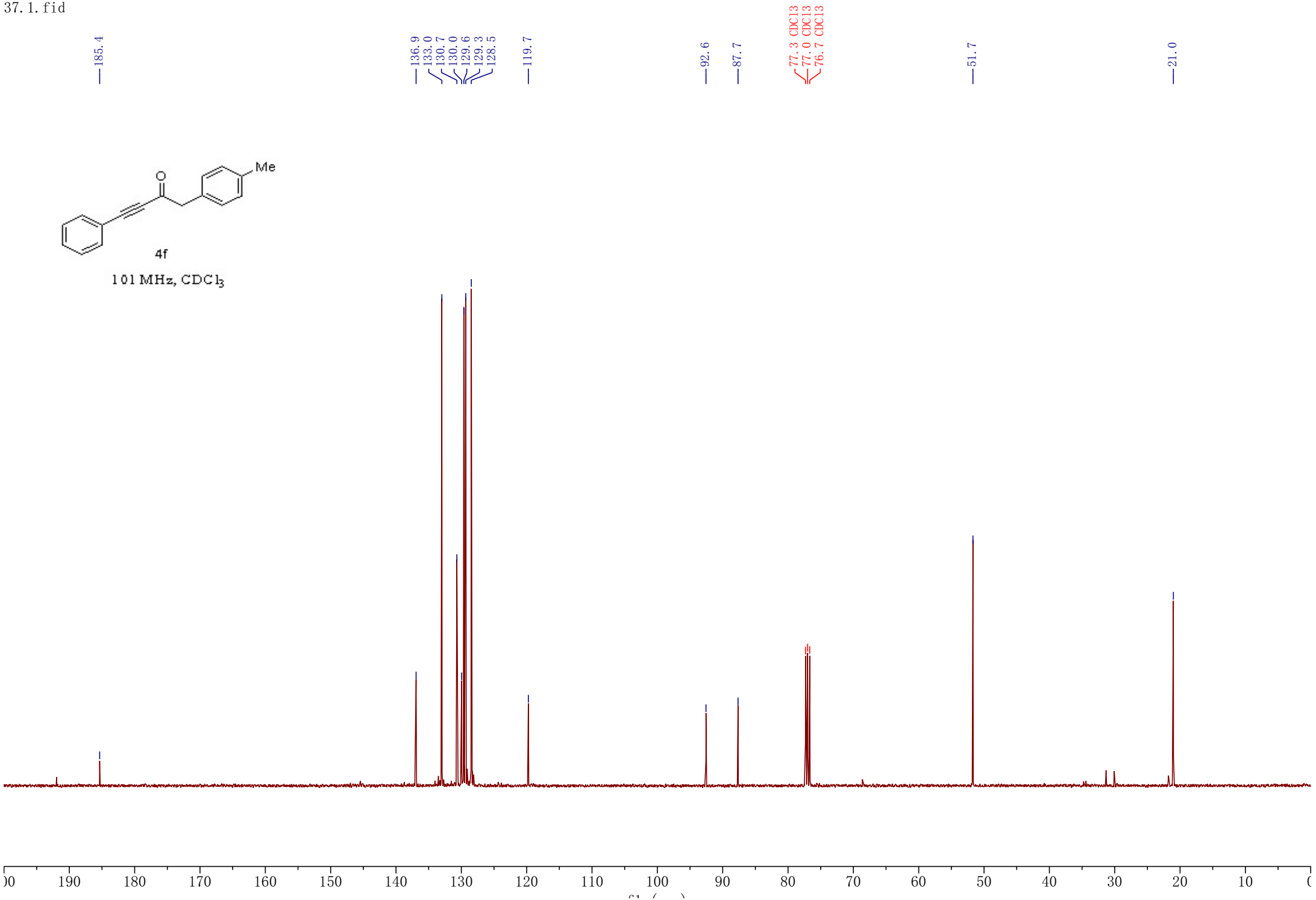


4f

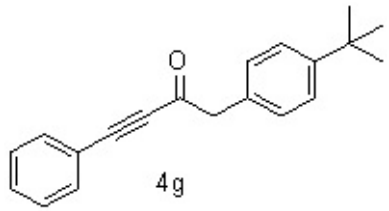
300 MHz, $CDCl_3$



37.1.fid


185.4
136.9
133.0
130.7
130.0
129.6
129.3
128.5
119.7
92.6
87.7
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
51.7
21.0
Me
O
4f
101 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

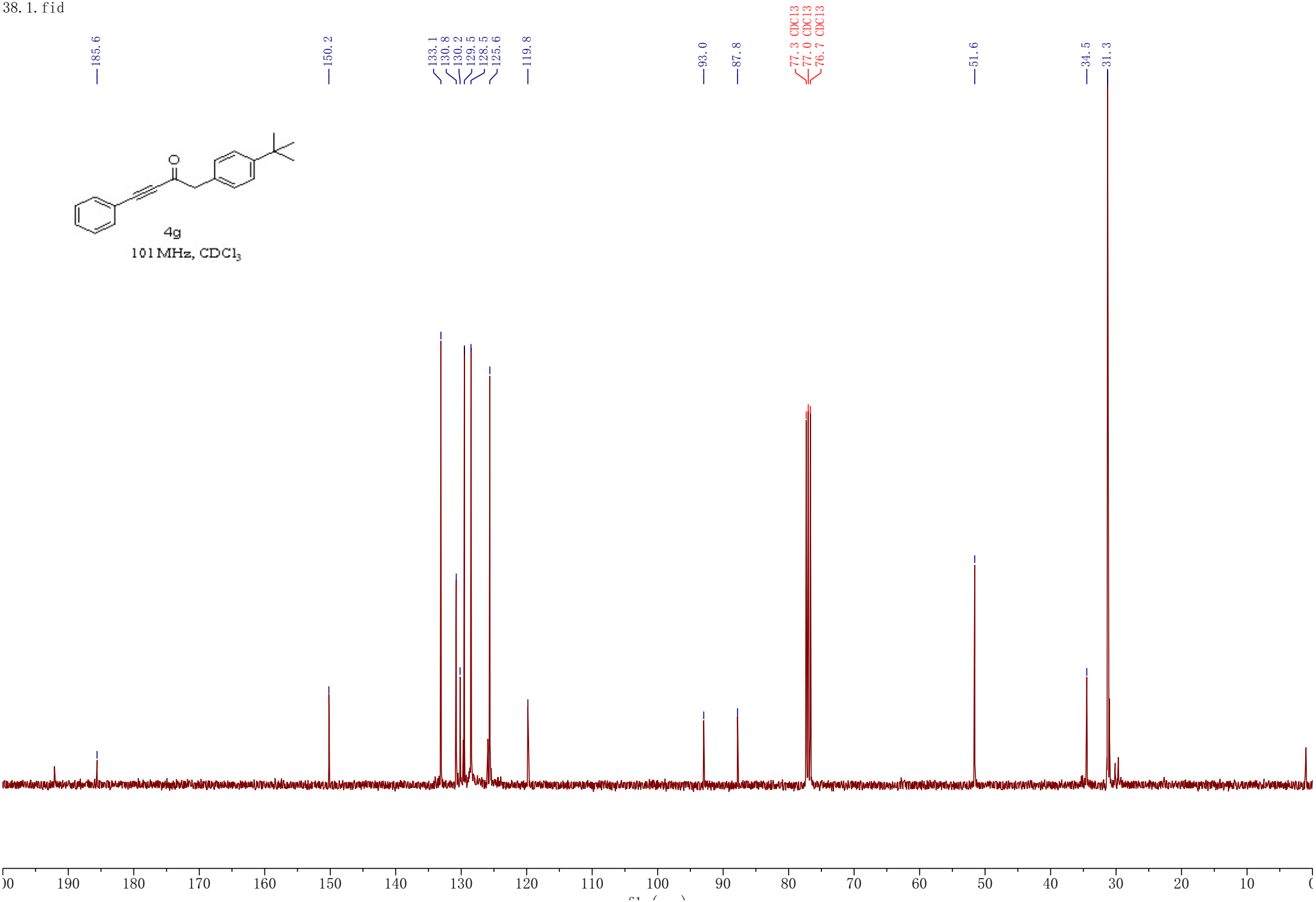
1014-38.1.fid



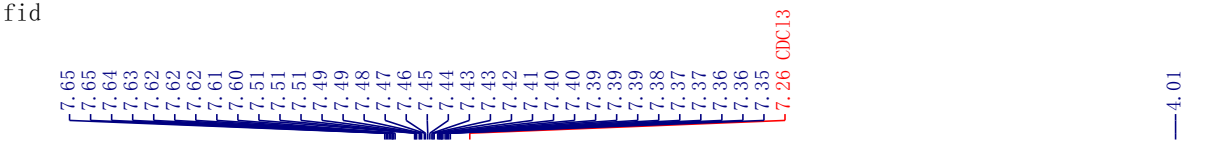
4g

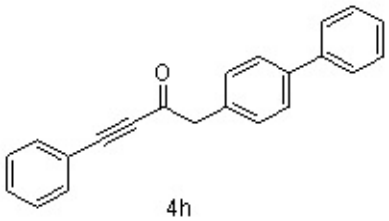
300 MHz, $CDCl_3$

38.1.fid


185.6
150.2
133.1
130.8
130.2
129.5
128.5
125.6
119.8
93.0
87.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
51.6
34.5
31.3
4g
101 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

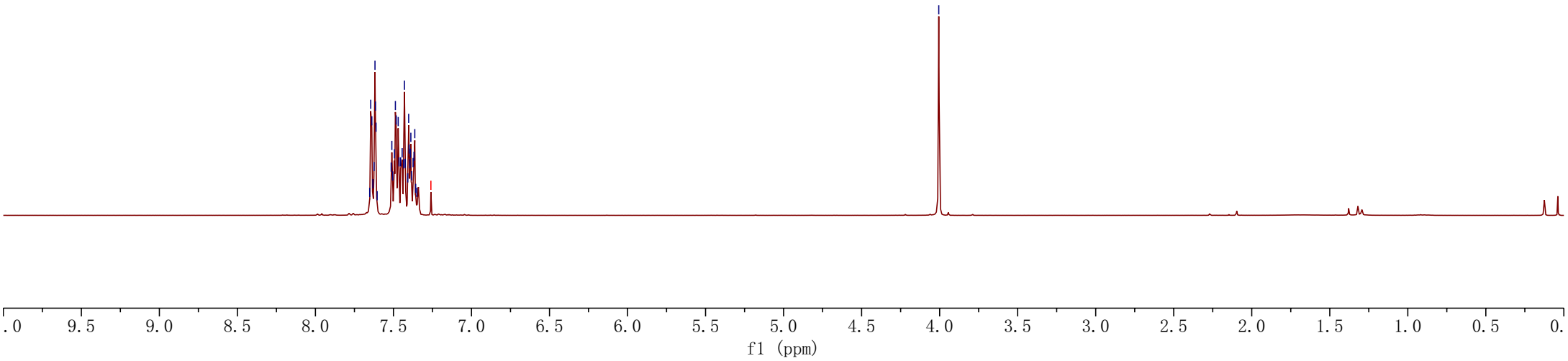
1014-41.1.fid



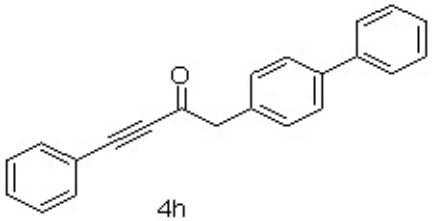


4h

300 MHz, $CDCl_3$



1023-41.2.fid

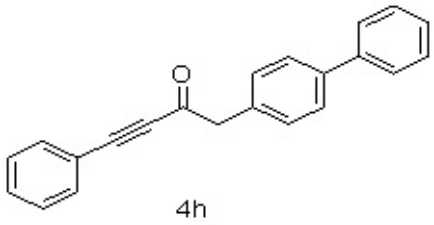


4h

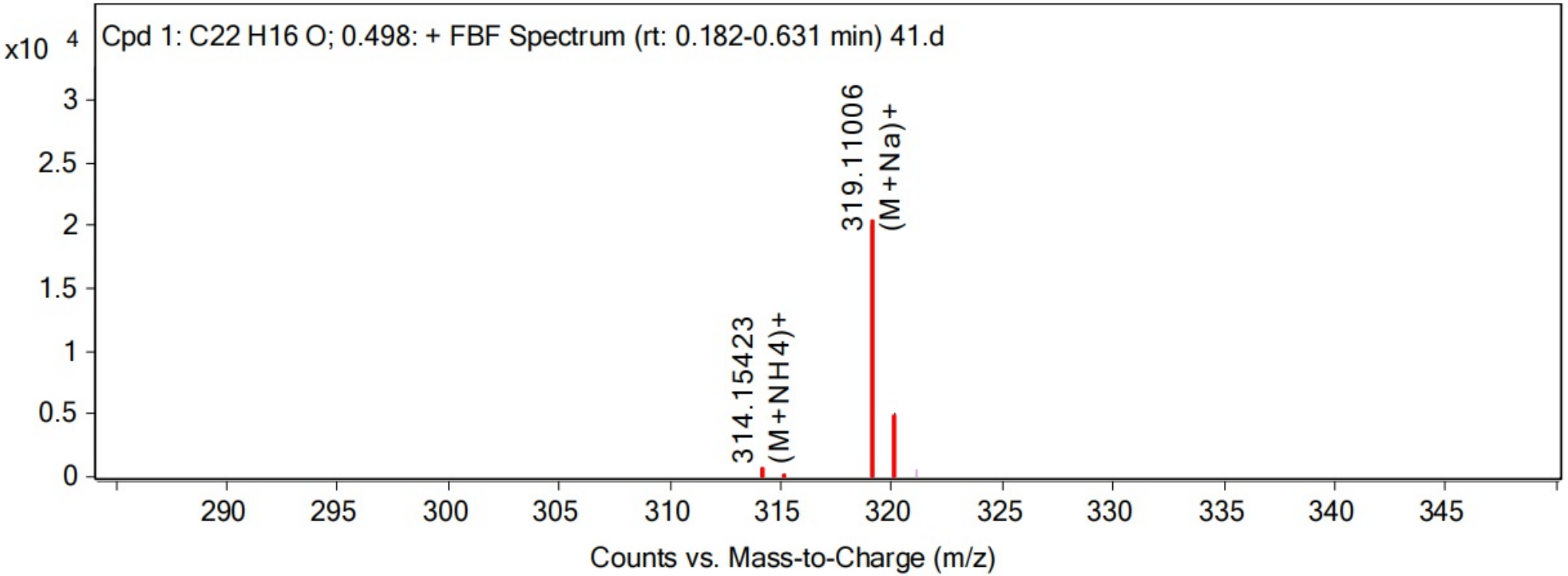
151 MHz, $CDCl_3$

185.0
140.7
140.3
133.1
132.2
130.8
130.2
128.7
128.6
127.4
127.3
127.0
119.8
93.0
87.8
77.2 CDCl3
77.0 CDCl3
76.8 CDCl3
51.7

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0



Qualitative Compound Identification Report

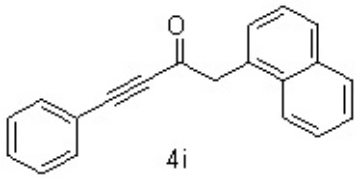


MS Spectrum Peak List

m/z	z	Abund	Ion
314.15423	1	652.57	(M+NH4)+
315.15085	1	134.78	(M+NH4)+
319.11006	1	20139.36	(M+Na)+
320.11287	1	5138.72	(M+Na)+

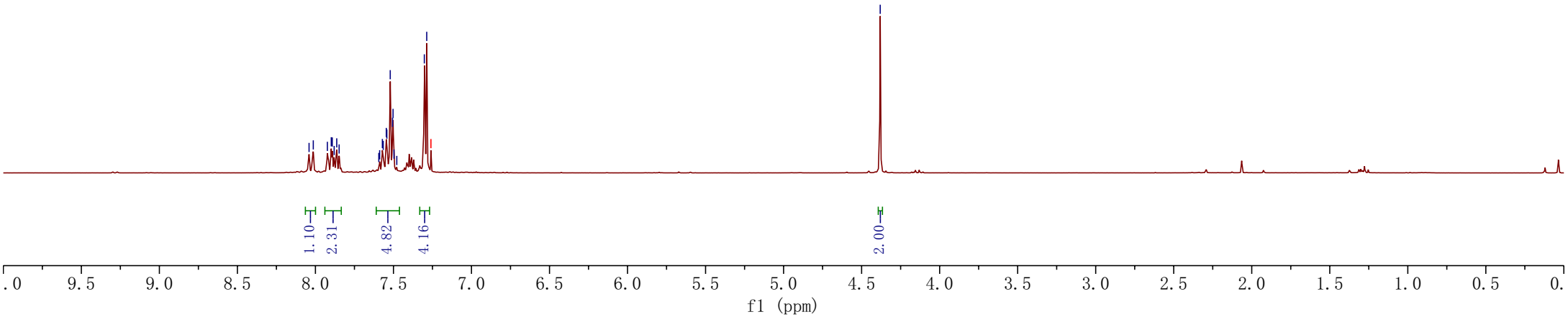
MS Spectrum

1014-40.1.fid

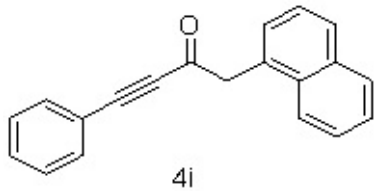


4i

300MHz,$CDCl_3$



1023-40.1.fid

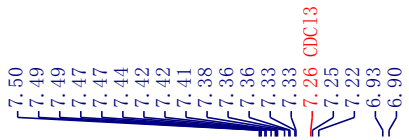


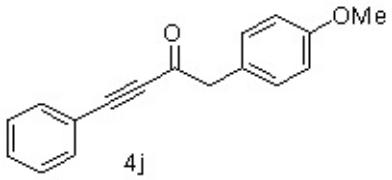
4i

151 MHz, $CDCl_3$

185.2
133.9
133.0
132.4
130.7
129.9
128.7
128.7
128.4
128.4
126.5
125.8
125.5
123.9
119.7
93.1
87.7
77.2 CDC13
77.0 CDC13
76.8 CDC13
49.8

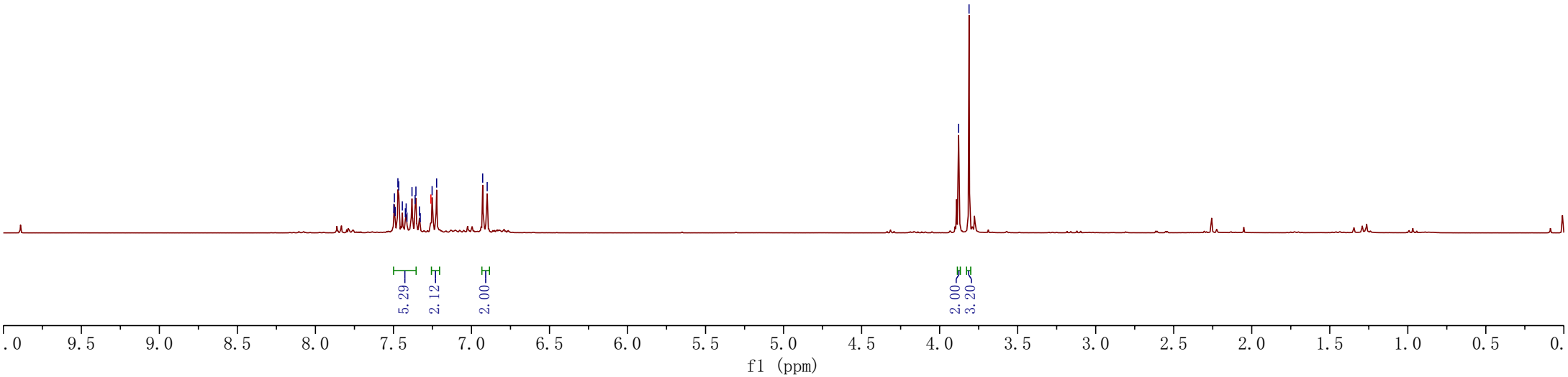
1014-43.1.fid



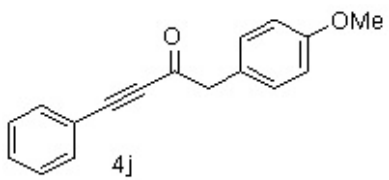


4j

300MHz, $CDCl_3$



1023-43.1.fid

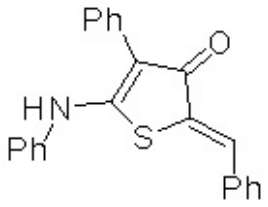


4j

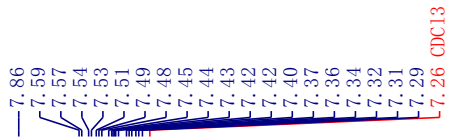
151 MHz, $CDCl_3$

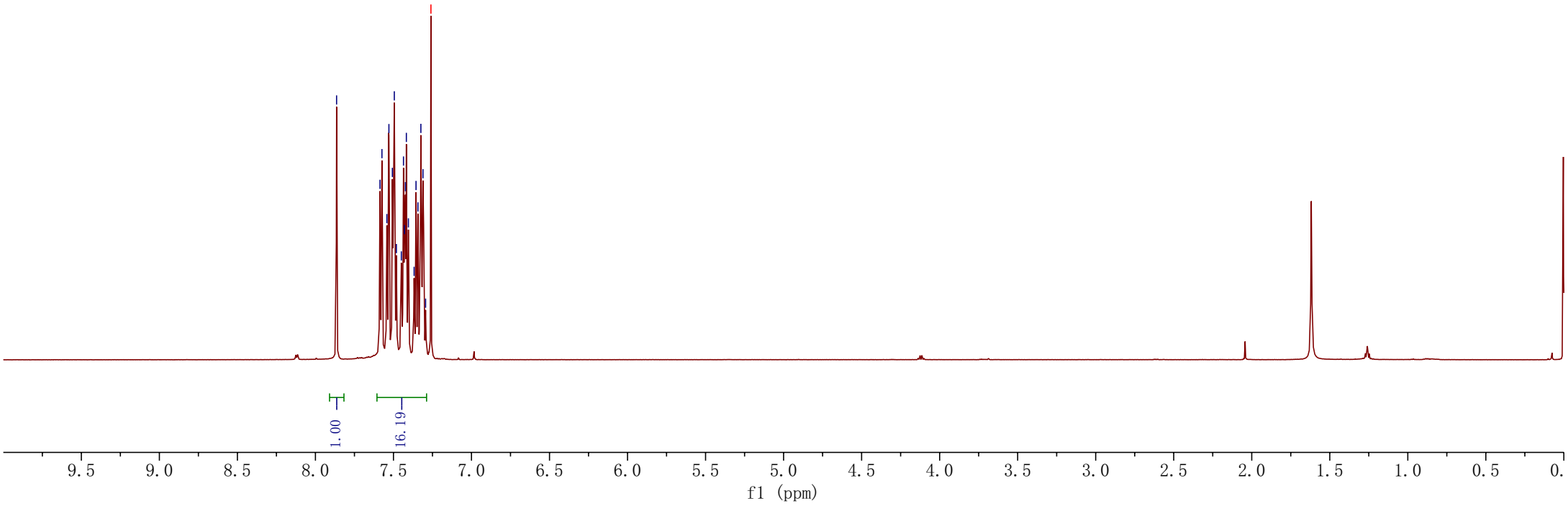
185.5
133.0
130.9
130.7
128.5
127.2
125.2
119.9
114.2
92.7
87.7
77.2 CDC13
77.0 CDC13
76.8 CDC13
55.2
51.3

Q13.14.fid
Q13 HNMR CDCl3

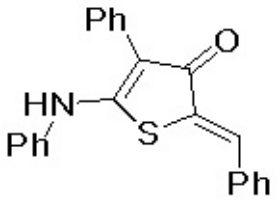


600 MHz, $CDCl_3$



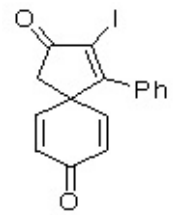


Q13.37.fid
Q13 CNMR CDCl3



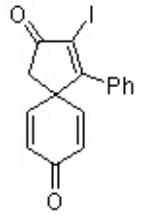
151 MHz, $CDCl_3$

Q14.15.fid
Q14 HNMR CDCl3



600 MHz, $CDCl_3$

Q14.38.fid
Q14 CNMR CDCl3



151 MHz, $CDCl_3$