Supplementary Material

Highly efficient synthesis of unsymmetrical 1,3-diynes from organoalanes reagents and alkynylbromides mediated by nickel catalyst

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Table of Contents

I. **Reagents and General Techniques**

^{1}H NMR and ^{13}CNMR spectra were recorded on a Varian 400 MHz spectrometer. The chemical shifts are reported relative to TMS. Analytical thin-layer chromatography (TLC) was performed on silica 60F-254 plates. Flash column chromatography was carried out on silica gel (200–400 mesh). HRMS were recorded on a Bruker Micro TOF spectrometer equipped with an ESI ion source. All reactions were carried out under nitrogen atmosphere. Chemical reagents and solvents were purchased from Adamas-beta and Aldrich, and were used without further purification with the exception of these reagents: THF, Ether and Toluene were distilled from Sodium under Nitrogen. Compounds of 1-bromoalkynes **2a-2p** were prepared according to literature procedures[1]. Compounds of propargyl chloride (**2q**) [2b]and propargyl iodide (**2r**)[2a] were prepared according to literature procedures[2]. Purification of reaction products was carried out by flash chromatography. All synthesis and manipulations were carried out under a dry nitrogen atmosphere.

II. The preparation of the Alkynylalane Reagents

1. General Procedures for the preparation diethyl(2-arylethynyl)aluminum (1a-1f) and tri(2-phenylethynyl)aluminum (1h)[3]

To a cooled (0 ^{o}C) solution of an alkyne (1.5 mmol) in hexane (3 mL) was added *n*-BuLi (1.5 mmol, 2.5 M in hexane) and stirred for 20 min, followed by an addition of $AlEt_2Cl$ (1.5 mmol, 1 M in hexane) or $AlCl_3$(0.5 mmol). The mixture thus obtained had been stirred at 0^{o}C for 20 min and then at room temperature for 2 h. The resulted solution was filtered, followed by an evaporation of hexane under reduced pressures and then added 2 mL of dry diethyl ether to give a alkynylaluminum reagent which was used in the coupling reactions.

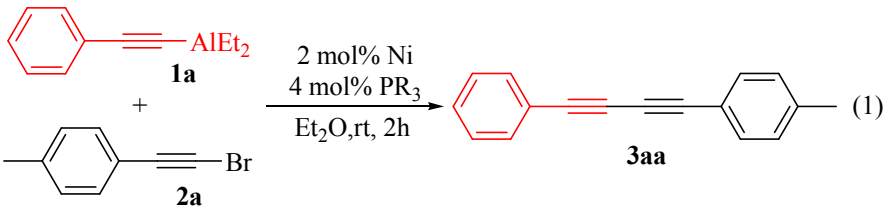
2. General Procedures for the preparation dimethyl(2-phenylethynyl)aluminum ($C_6H_5C{\equiv}CAlMe_2$)(1g)[3e]

The preparation of hept-1-ynyl-benzene (entry1) is representative. Alane solution preparation: A dry and argon-flushed flask equipped with a magnetic stirrer and condenser was charged with a commercial trimethylaluminum solution (10 mL, 2 M in heptane) (CAUTION: trimethylaluminum is flammable), and triethylamine (0.28 mL, 2 mmol) was then added dropwise via a syringe. 1-ethynylbenzene (2.36 mL, 18mmol) was dropwise added 5 min later, and the reaction mixture was stirred at 60 °C for 6 h, until the gas evolution ceased. The prepared alane solution can be stored under argon in the dark for several days.

III. Coupling Reaction of Alkynyl Bromide with Organoalanes Reagents

III.1. Optimization of Reaction Conditions for The Coupling Reaction of $C_6H_5C{\equiv}CAlEt_2$ (1a) 4-$CH_3C_6H_4C{\equiv}CBr$ (2a) and Catalyzed by the Nickel.

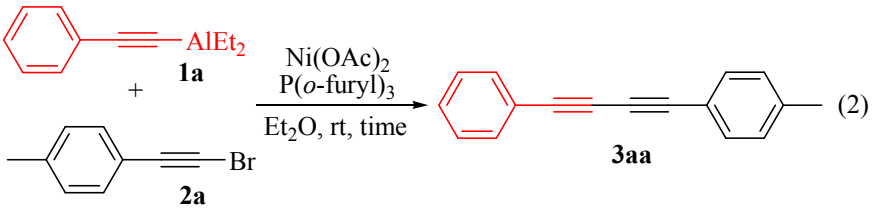
Table S1. Optimizations of Coupling Reactions of $C_6H_5C{\equiv}CAlEt_2$ (**1a**) with 4-$CH_3C_6H_4C{\equiv}CBr$ (**2a**) Catalyzed by the Nickel. [a]



Entry	Catalyst	Ligand	Solvent	Yield(%)[b]
1	$NiCl_2$	-	Et_2O	0
2	$NiCl_2$	PPh_3	Et_2O	34
3	$NiCl_2$	PCy_3	Et_2O	23
4	$NiCl_2$	(*o*-tolyl)$_3$P	Et_2O	19
5	$NiCl_2$	(*p*-tolyl)$_3$P	Et_2O	40
6	$NiCl_2$	(*o*-furyl)$_3$P	Et_2O	42
7	$NiBr_2$	(*o*-furyl)$_3$P	Et_2O	65
8	$Ni(acac)_2$	(*o*-furyl)$_3$P	Et_2O	48
9	$Ni(OAc)_2$	(*o*-furyl)$_3$P	Et_2O	75
10	$Ni(OAc)_2$	(*o*-furyl)$_3$P	Toluene	75
11	$Ni(OAc)_2$	(*o*-furyl)$_3$P	THF	52

[a]**1a**/**2a** = 1.0/0.5 mmol, 2 mL solvent, room temperature.[b]Isolated yield.

Table S2. Optimizations of Cross-Coulping Reaction of $C_6H_5C{\equiv}CAlEt_2$ (**1a**) 4-$CH_3C_6H_4C{\equiv}CBr$ (**2a**) Catalyzed by the $Ni(OAc)_2$/P(*o*-furyl)$_3$.[a]

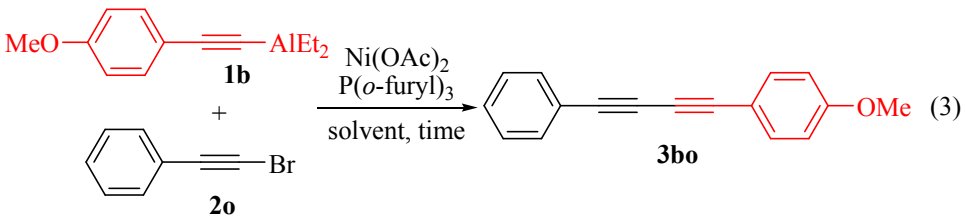


Entry	$Ni(OAc)_2$(mol%)	P(*o*-furyl)$_3$(mol%)	Additive (1.equiv.)	Time(h)	Yield(%)[d]
1	10	20	-	4	78
2	15	30	-	4	71
3	2	4	-	4	81
4	2	4	-	5	78
5	2	4	-	2	87
6	2	2	-	2	78
7	2	6	-	2	87
8[b]	2	4	-	2	62
9[c]	2	4	-	2	83
10	2	4	LiCl	2	56

11	2	4	$MgBr_2$	2	57

[a]**1a**/**2a** = 1.0/0.5 mmol, 2 mLEt_2O, room temperature.[b]**1a**/**2a** = 1.5/1 mmol.
[c]**1a**/**2a** = 2.5/1 mmol. [d]Isolated yield.

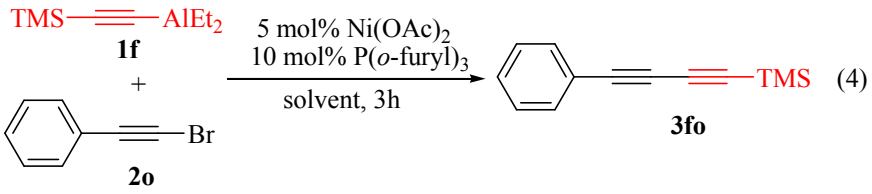
Table S3. Optimizations of Cross-Coulping Reaction of 4-$CH_3OC_6H_5C{\equiv}CAlEt_2$ (**1b**) with $C_6H_4C{\equiv}CBr$ (**2o**) Catalyzed by the Nickel.[a]



Entry	$Ni(OAc)_2$(mol%)	P(*o*-furyl)$_3$(mol%)	Solvent	Time(h)	Yield (%)[c]
1	2	4	Et_2O	2	62
2	2	4	Toluene	2	53
3[b]	2	4	Toluene	2	65
4[b]	2	4	Toluene	3	67
5[b]	5	10	Toluene	3	75

[a]**1b**/**2o** = 1.0/0.5 mmol, solvent 2mL, room temperature.[b]**1b**/**2o** = 1.0/0.5 mmol, 60°C.
[c]Isolated yield.

Table S4. Optimizations of Cross-Coulping Reaction of $TMSC{\equiv}CAlEt_2$ (**1f**) with $C_6H_4C{\equiv}CBr$ (**2o**) Catalyzed by the Nickel.[a]



Entry	T (°C)	Solvent	Yield (%)[b]
1	60	Toluene	52
2	rt	Et_2O	58
3	60	DME	16
4	rt	Toluene	80

[a]**1f**/**2o** = 1.0/0.5mmol, solvent 2mL.[b]Isolated yield.

III.2. General Procedures for the Coupling Reaction of Alkynyl Bromide with Organoalanes Reagents

Under a dry nitrogen atmosphere, a mixture of $Ni(OAc)_2$ (3.52 mg, 0.02 mmol or 8.80 mg, 0.05 mmol) and tri(*o*-furyl)phosphine (9.28 mg, 0.05 mmol or 23.20 mg, 0.10 mmol) in a reaction vessel was added an alkynylaluminum compound (1.0 mmol) in 2 mL Et_2O or Toluene or DME followed by an addition of alkynylbromides (0.50 mmol). The resulted solution was stirred at room temperature or 60°C for 2-3 h. After completion the reaction, the

mixture was diluted with saturated ammonium chloride solution (5 mL) and extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried overanhydrous Na_2SO_4, filtered and evaporated *in vacuum.* The residue was subjected to flash column chromatography onsilica gel (hexane or ethyl acetate and hexane) to afford the corresponding conjugated 1,3-diyne **3**.

IV. ^{1}H and ^{13}C NMR and High-resolution Mass Spectroscopic Data of Coupling Products

1-methyl-4-(4-phenylbuta-1,3-diynyl)benzene (**3aa**) [4]: Eluent: hexane; white solid; yield: 0.095 g (87%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54-7.52 (m, 2H), 7.44-7.42 (m, 2H), 7.37-7.32 (m, 3H), 7.15-7.13 (m, 2H), 2.36 (s, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 139.7, 139.6, 132.6, 129.3, 129.2, 128.6, 121.9, 118.7, 82.0, 81.7, 74.1, 73.5, 21.7 ppm.

1-ethyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ab)** [4]: Eluent: hexane; white solid; yield: 0.092 g (85%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53-7.51 (m, 2H), 7.46-7.43 (m, 2H), 7.37-7.33 (m, 3H), 7.24-7.15 (m, 2H), 2.65 (q, J = 3.8Hz, 2H), 1.22(t, J = 1.9Hz, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 145.8, 132.5, 129.3, 129.1, 128.5, 128.1, 122.0, 119.1, 81.6, 81.3, 74.2, 73.5, 29.0, 15.3 ppm.

1-propyl-3-(4-phenylbuta-1,3-diynyl)benzene **(3ac)** [4]: Eluent: hexane; white solid; yield: 0.082 g (86%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54-7.52 (m, 2H), 7.45-7.43 (m, 2H), 7.38-7.31 (m, 3H), 7.16-7.14 (m, 2H), 2.58 (t, J = 1.9Hz, 2H), 1.63(q, J = 3.8Hz, 2H), 0.93(t, J = 1.8Hz, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 144.4, 132.6, 129.4, 129.2, 128.8, 128.6, 122.0, 119.1, 81.7, 81.3, 74.2, 73.4, 38.2, 24.4, 13.9 ppm.

1-tert-butyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ad)** [4]: Eluent: hexane; white solid; yield: 0.082 g (84%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53-7.51 (m, 2H), 7.48-7.45 (m, 2H), 7.37-7.33 (m, 5H), 1.31 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 152.7, 132.6, 132.4, 129.3, 128.6, 125.6, 122.1, 119.0, 81.7, 81.3, 74.3, 73.6, 35.0, 31.2 ppm.

1-methyl-3-(4-phenylbuta-1,3-diynyl)benzene **(3ae)** [4]: Eluent: hexane; white solid; yield: 0.087 g (81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53-7.51 (m, 2H), 7.34-7.32 (m, 5H), 7.24-7.16 (m, 2H), 2.33 (s, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 138.3, 133.1, 132.6, 130.3, 129.7, 129.2, 128.6, 128.5, 122.0, 121.7, 82.0, 81.5, 74.1, 73.7, 21.3 ppm.

1-methoxy-4-(4-phenylbuta-1,3-diynyl)benzene **(3af)**: [5] Eluent: hexane; white solid; yield: 0.069 g (64%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53-7.46 (m, 4H), 7.37-7.31 (m, 3H), 6.86 (d, J = 2Hz, 2H), 3.83 (s, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 160.5, 134.2, 132.6, 129.1, 128.5, 122.1, 114.3, 113.9, 82.0, 81.5, 74.3, 73.2, 55.5 ppm.

1-bromo-4-(4-phenylbuta-1,3-diynyl)benzene **(3ag)** [6]: Eluent: hexane; pale yellow solid; yield: 0.08 g (74%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53-7.45 (m, 4H), 7.37-7.31 (m, 5H)

ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 134.0, 132.7, 131.9, 129.3, 128.6, 125.8, 121.9, 120.9, 81.7, 80.5, 75.2, 74.0 ppm.

1-chloro-4-(4-phenylbuta-1,3-diynyl)benzene **(3ah)**[4]: Eluent: hexane; white solid; yield: 0.097 g (82%). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.56-7.52(m, 2H), 7.46-7.44 (m, 2H), 7.39-7.31 (m, 5H) ppm.$^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 135.5, 133.8, 132.7, 129.5, 129.0, 128.6, 121.7, 120.4, 82.3, 80.4, 75.0, 73.8 ppm.

1-fluoro-4-(4-phenylbuta-1,3-diynyl)benzene **(3ai)** [4]: Eluent: hexane; white solid; yield: 0.099 g (90%). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.55-7.49(m, 4H), 7.39-7.32 (m, 3H), 7.05-7.01 (m, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 163.2 (d, J =250Hz), 134.6 (q, J = 87Hz), 132.6, 129.3 (d, J =65Hz), 128.6 (d, J =15Hz), 121.9 (d, J =107Hz), 118.0 (t, J =20Hz), 116.1 (q, J =23Hz), 81.7, 80.6, 74.0, 73.9 ppm.

1-fluoro-3-(4-phenylbuta-1,3-diynyl)benzene **(3aj)**: Eluent: hexane; white solid; yield: 0.102 g (93%). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.54-7.52 (m, 2H), 7.38-7.30 (m, 5H), 7.24-7.20 (m, 1H), 7.08-7.06 (m, 1H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 163.2 (d, J =250Hz), 134.6 (q, J = 87Hz), 132.6, 129.3 (d, J = 65Hz), 128.6 (d, J =15Hz), 121.9 (d, J =107Hz), 118.0 (t, J = 20Hz), 116.1 (q, J = 23Hz), 81.7, 80.6, 74.0, 73.9 ppm. HRMS (ESI) m/z calcd for $C_{16}H_{10}F^+(M+H)^+$ 221.07610, found 221.07611.

1-fluoro-2-(4-phenylbuta-1,3-diynyl)benzene **(3ak)**: Eluent: hexane; white solid; yield: 0.095 g (86%). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.54-7.49 (m, 3H), 7.40-7.32 (m, 4H), 7.16-7.07 (m, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 163.2 (d, J =251Hz), 134.6, 132.6 (d, J = 4.5Hz), 131.1 (d, J = 6Hz), 129.4 (d, J = 17.5Hz), 128.6, 124.2 (d, J = 3.7Hz), 121.6, 115.8 (t, J = 20.6Hz), 110.8 (d, J = 15.5Hz), 82.8, 78.8, 74.9, 73.8 ppm. HRMS (ESI) m/z calcd for $C_{16}H_{10}F^+(M+H)^+$ 221.07610, found 221.07599.

1-(4-(4-(trifluoromethyl)phenyl)buta-1,3-diynyl)benzene **(3al)**[8]: Eluent: hexane; white solid; yield: 0.124 g (92%). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.63-7.58 (m, 4H), 7.55-7.53 (m, 2H), 7.41-7.33 (m, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =132.8 (t, J = 10.6Hz), 130.9 (d, J = 42.6Hz), 129.7, 128.6, 125.8, 125.6 (d, J = 4Hz), 125.5 (d, J = 4Hz), 122.5, 121.5, 83.0, 79.9, 76.6, 73.6 ppm.

2-(4-phenylbuta-1,3-diynyl)pyridine **(3am)** [7]: Eluent: hexane; pale yellow solid; yield: 0.096 g (94%). ^1H NMR (400 MHz, $CDCl_3$): δ =8.67-8.66(m, 1H), 7.93-7.82 (m, 2H), 7.73-7.71 (m, 2H), 7.50-7.47 (m, 3H), 7.37-7.35 (m, 1H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =152.9, 149.3, 137.1, 131.9, 129.1, 128.6,123.3, 121.5, 119.7, 99.2, 98.5, 85.7, 83.8 ppm.

2-(4-phenylbuta-1,3-diynyl)thiophene **(3an)** [7]: Eluent: hexane; brown solid; yield: 0.087 g (83%). ^1H NMR (400 MHz, $CDCl_3$): δ =7.52-7.51 (m, 2H), 7.48-7.30 (m, 5H), 6.99 (s, 1H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ = 134.4, 132.6, 129.4, 128.7, 127.3,125.6, 122.2,

121.8, 83.8, 78.1, 74.7, 73.9 ppm.

1-methoxy-4-(4-*p*-tolylbuta-1,3-diynyl)benzene **(3ba)** [7]: Eluent: hexane; white solid; yield: 0.076 g (62%).^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38 (d, *J* = 2.1Hz, 2H), 7.33 (d, *J* = 2Hz, 2H), 7.05 (d, *J* = 2.1Hz, 2H), 6.77 (d, *J* = 2.1Hz, 2H), 3.73 (s, 3H), 2.28 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 160.4, 139.5, 134.2, 132.5, 129.3, 119.0, 114.3, 114.0, 81.6, 81.4, 73.7, 73.0, 55.5, 21.7 ppm.

1-(4-(4-chlorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bh)** [9]: Eluent: hexane; pale yellow solid; yield: 0.096 g (72%).^{1}H NMR (400 MHz, $CDCl_3$): δ =7.46 (d, *J* = 2Hz, 4H), 7.43 (d, *J* = 2Hz, 2H),7.30 (d, *J* = 1.1Hz, 2H), 6.85 (d, *J* = 2.1Hz, 2H), 3.82 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 160.6, 135.3, 134.3, 133.7, 129.0, 120.6, 114.3, 113.6, 82.5, 80.0, 75.3, 72.7, 55.5 ppm.

1-(4-(4-fluorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bi)** [4]: Eluent: hexane; white solid; yield: 0.089g (71%). ^{1}H NMR (400 MHz, $CDCl_3$): δ =7.52-7.46 (m, 5H),7.01 (d, *J* = 1.1Hz, 2H), 6.85 (d, *J* = 2.1Hz, 2H), 3.83(s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 163.0 (d, *J* =240Hz), 160.6, 134.6, 134.3 (d, *J* = 24.6Hz), 118.3, 115.9 (d, *J* = 22.2Hz), 114.3, 113.7, 82.0, 80.1, 74.1, 72.7, 55.5 ppm.

1-(4-(3-fluorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bj)** [10]: Eluent: hexane; white solid; yield: 0.090g (72%).^{1}H NMR (400 MHz, $CDCl_3$): δ =7.48-7.46 (m, 2H), 7.28-7.25 (m, 2H), 7.20 (d, *J* = 2Hz, 1H), 7.06 (d, *J* = 1.3Hz, 1H), 6.85 (d, *J* = 2Hz, 2H), 3.81 (s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ = 162.3 (d, *J* = 246Hz), 160.6, 134.3, 130.1, 128.5 (d, *J* = 2Hz), 124.0 (d, *J* = 9Hz), 119.1 (d, *J* = 22Hz), 116.6(d, *J* =21.1Hz), 114.3, 113.5, 82.7,79.7, 75.2, 72.6, 55.5 ppm. HRMS (ESI) m/z calcd for $C_{17}H_{12}OF^+$ $(M+H)^+$ 251.08667, found 251.08664.

2-(4-(4-methoxyphenyl)buta-1,3-diynyl)thiophene **(3bn)** [8]: Eluent: hexane; white solid; yield: 0.088 g (74%).^{1}H NMR (400 MHz, $CDCl_3$): δ =7.47-7.45 (m, 2H), 7.33-7.29 (m, 2H), 7.00-6.98 (m, 1H), 6.85 (d, *J* = 2Hz, 2H), 3.82(s, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ =160.6, 134.1, 128.6, 127.3, 122.4, 114.3, 113.7, 84.0, 78.6, 74.1, 72.7, 55.5, 31.1 ppm.

4-(3-fluorophenyl)-1-(4-fluorophenyl)buta-1,3-diyne **(3cj)**: Eluent: hexane; white solid; yield: 0.066 g (55%). ^{1}H NMR (400 MHz, $CDCl_3$): δ =7.51-7.49 (m, 2H), 7.30-7.27 (m, 2H)7.22-7.20 (m, 1H), 7.08-7.02 (m, 3H) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ =163.2 (d, *J* = 242Hz), 162.4 (t, *J* = 245.8Hz), 134.7 (t, *J* = 78.5Hz), 130.2 (d, *J* =9.7Hz), 128.6, 125.6 (d, *J* =10Hz), 119.3 (d, *J* =23.1Hz), 117.7 (d, *J* =3.7Hz), 117.0, 116.2 (t, *J* = 41.4Hz), 81.2, 80.1, 74.8, 73.5 ppm. HRMS (ESI) m/z calcd for $C_{16}H_9F_2^+$ $(M+H)^+$ 239.06668, found 239.06714.

4-(2-fluorophenyl)-1-(4-fluorophenyl)buta-1,3-diyne **(3ck)**: Eluent: hexane; white solid; yield: 0.063 g (53%). ^{1}H NMR (400 MHz, $CDCl_3$): δ=7.56-7.49 (m, 3H), 7.38-7.33 (m, 1H), 7.14-7.02 (m, 4H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ=164.7 (d, J = 64.2Hz), 162.2 (d, J = 62.6Hz), 134.5 (d, J = 8.5Hz), 134.2, 131(d, J = 8Hz), 124.1 (d, J =3.8Hz), 117.7, 115.8 (q, J = 24.3Hz), 110.6 (t, J = 15.6Hz), 81.7, 78.7, 74.9, 73.6 ppm. HRMS (ESI) m/z calcd for $C_{16}H_9F_2^+$ $(M+H)^+$ 239.06668, found 239.06602.

1-fluoro-4-(nona-1,3-diynyl)benzene**(3cp)**[11]: Eluent: hexane; white solid; yield: 0.054g (51%).^{1}H NMR (400 MHz, $CDCl_3$): δ=7.47-7.46 (m, 2H),7.02-6.98 (m, 2H), 2.57-2.54 (m, 2H), 1.60-1.56 (m, 2H), 1.41-1.33 (m, 4H), 0.91 (t, J = 3Hz, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ= 162.9 (d, J = 249.2Hz), 134.5 (d, J = 8.5Hz), 118.3, 115.9 (d, J = 22.1Hz), 85.1, 74.3, 73.8, 65.0, 31.2, 28.1, 22.3, 19.7, 14.1 ppm.

1-(4-(4-bromophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dg**)[8]: Eluent: hexane; pale yellow solid; yield: 0.093 g (68%).^{1}H NMR (400 MHz, $CDCl_3$): δ=7.47 (d, J = 2.1 Hz, 2H), 7.42 (d, J = 2.1 Hz, 2H),7.37 (d, J = 2.1Hz, 2H), 7.15(d, J = 2.0Hz, 2H), 2.37(s, 3H) ppm.$^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =140.0, 133.9,132.6, 132.0, 131.9, 129.4, 123.7, 121.0, 118.6, 82.7, 80.2, 75.3, 73.2, 21.8 ppm.

1-(4-(4-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene **(3di)** [7]: Eluent: hexane; white solid; yield: 0.078 g (62%). ^{1}H NMR (400 MHz, $CDCl_3$): δ=7.52-7.49 (m, 2H),7.43-7.41 (m, 2H), 7.15-7.13 (m, 2H), 7.05-7.00 (m, 2H), 2.36 (s, 3H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =162.5 (d, J = 240Hz), 139.8, 134.6 (d, J = 8.3Hz), 132.6, 129.4, 118.7, 118.2, 115.9 (d, J = 22.1Hz), 82.0, 80.2, 74.0, 73.3 , 21.8.

1-(4-(3-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dj**): Eluent: hexane; pale yellow solid; yield: 0.085 g (73%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.57-7.53 (m, 1H), 7.49-7.40 (m, 2H), 7.39-7.36 (m, 1H), 7.30-7.11 (m, 4H), 2.41 (s, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ=163.7 (d, J = 252Hz), 139.8, 134.2, 132.4, 130.8 (d, J = 11Hz), 129.2, 124.1 (d, J = 3.7 Hz), 118.4, 115.6 (d, J = 21Hz), 110.7, 83.0, 78.8, 74.4, 73.1, 21.6 ppm. HRMS (ESI) m/z calcd for $C_{17}H_{12}F^+$$(M+H)^+$ 235.09176, found 235.09195.

1-(4-(2-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dk**): Eluent: hexane; white solid; yield: 0.094 g (80%).^{1}H NMR (400 MHz, $CDCl_3$): δ=7.41 (d, J = 7.2 Hz, 2H),7.29-7.27 (m, 2H),7.20-7.17(m, 1H), 7.13 (d, J = 6.2 Hz, 2H), 7.07-7.03(m, 1H), 2.35(s, 3H) ppm.$^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =162.4(d, J = 245.8Hz), 140.0, 132.6, 130.2 (d, J = 8.6Hz), 129.4, 128.5 (d, J = 3.1Hz), 123.9 (d, J = 9.4Hz), 119.2 (d, J = 22.9Hz), 118.5, 116.7 (d, J = 21.1Hz), 82.7, 79.9, 75.1, 73.1, 21.8 ppm. HRMS (ESI) m/z calcd for $C_{17}H_{12}F^+$$(M+H)^+$ 235.09176, found 235.09164.

1-(4-(4-trifluoromethylphenyl)buta-1,3-diynyl)-4-methylbenzene (**3dl**): Eluent: hexane; white

solid; yield: 0.106 g (75%). ^{1}H NMR (400 MHz, $CDCl_3$): δ=7.63-7.56 (m, 4H), 7.44 (d, J = 7.2 Hz, 2H), 7.16 (d, J = 7.2 Hz, 2H), 2.38 (s, 3H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =140.2, 132.9, 132.7 (d, J = 13.8Hz), 130.7 (d, J = 32Hz), 129.4, 126.0, 125.5 (q, J = 37Hz), 122.6, 118.4, 83.4, 79.6, 76.6, 73.0, 21.8 ppm. HRMS (ESI) m/z calcd for $C_{17}H_9F_4^+(M+H)^+$ 289.06349, found 289.06368.

2-(4-(4-methylphenyl)buta-1,3-diynyl)pyridine (**3dm**)[12]: Eluent: hexane; brown solid; yield: 0.078 g (72%). ^{1}H NMR (400 MHz, $CDCl_3$): δ=8.54-8.53 (m, 1H), 7.81-7.70 (m, 2H), 7.52-7.48 (m, 2H), 7.24-7.21 (m, 1H), 7.18 (d, J = 7.2Hz, 2H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ=153.0, 149.2, 139.4, 137.1, 131.8, 129.2 (d, J = 20Hz), 123.2, 121.6, 119.4 (d, J = 36.2Hz), 99.5, 98.8, 85.1, 83.2, 21.7ppm.

(4-phenyl-buta-1,3-diynyl)-trimethyl-silane (**3fo**)[13]: Eluent: hexane; pale yellow solid; yield: 0.079 g (80%). ^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.50-7.48 (m, 2H), 7.37-7.30 (m, 3H), 0.23 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =132.8, 129.5, 128.6, 121.5, 90.8, 87.9, 76.8, 74.3, -0.24ppm.

[4-(4-tert-Butyl-phenyl)-buta-1,3-diynyl]-trimethyl-silane (**3fd**) [14]: Eluent: hexane; pale yellow solid; yield: 0.094 g (74%). ^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.43 (d, J = 2.0Hz, 2H), 7.33 (d, J = 2.1Hz, 2H), 1.30 (s, 9H), 0.23 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =153.0, 132.6, 125.6, 118.4, 90.3, 88.1, 76.8, 73.6, 35.1, 31.2, -0.21ppm.

[4-(4-Methoxy-phenyl)-buta-1,3-diynyl]-trimethyl-silane (**3ff**) [13]: Eluent: hexane; pale yellow solid; yield: 0.088 g (76%). ^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.43 (d, J = 2.2Hz, 2H), 6.83 (d, J = 2.2Hz, 2H), 3.81 (s, 3H) 0.22 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =160.6, 134.5, 114.3, 113.4, 90.0, 88.3, 86.1, 73.2, 55.5, -0.20ppm.

[4-(4-Fluoro-phenyl)-buta-1,3-diynyl]-trimethyl-silane (**3fi**) [13]: Eluent: hexane; pale yellow solid; yield: 0.079 g (72%). ^{1}H NMR (400 MHz, $CDCl_3$): δ= 7.49-7.45 (m, 2H), 7.03-6.99 (m, 2H), 0.23 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =163.2(d, J = 250 Hz), 134.9 (d, J = 8.5Hz), 116.1, 115.9, 90.9, 87.7, 86.1, 75.8, -0.26ppm.

[4-(4-trifluoromethyl-phenyl)-buta-1,3-diynyl]-trimethyl-silane (**3fl**)[15]: Eluent: hexane; pale yellow solid; yield: 0.087 g (67%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.64-7.62 (m, 1H), 7.60-7.58 (m, 3H), 0.25 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ=133.1, 131.1(d, J = 32.6Hz), 125.5(q, J = 75.5Hz), 125.2, 122.5, 92.6, 87.4, 76.5, 75.1, -0.23ppm.

(4-naphthalen-2-yl-buta-1,3-diynyl)-trimethyl-silane (**3fq**)[16]: Eluent: hexane; pale yellow solid; yield: 0.092 g (74%). ^{1}H NMR (400 MHz, $CDCl_3$): δ= 8.03 (s, 1H), 7.79-7.75 (m, 3H), 7.51-7.48 (m, 3H), 0.25 (s, 9H) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ =133.4, 133.3, 132.8, 129.6, 128.3, 127.97, 127.94, 127.4, 126.9, 118.7, 91.0, 88.0, 76.8, 74.5, -0.22ppm.

V. References

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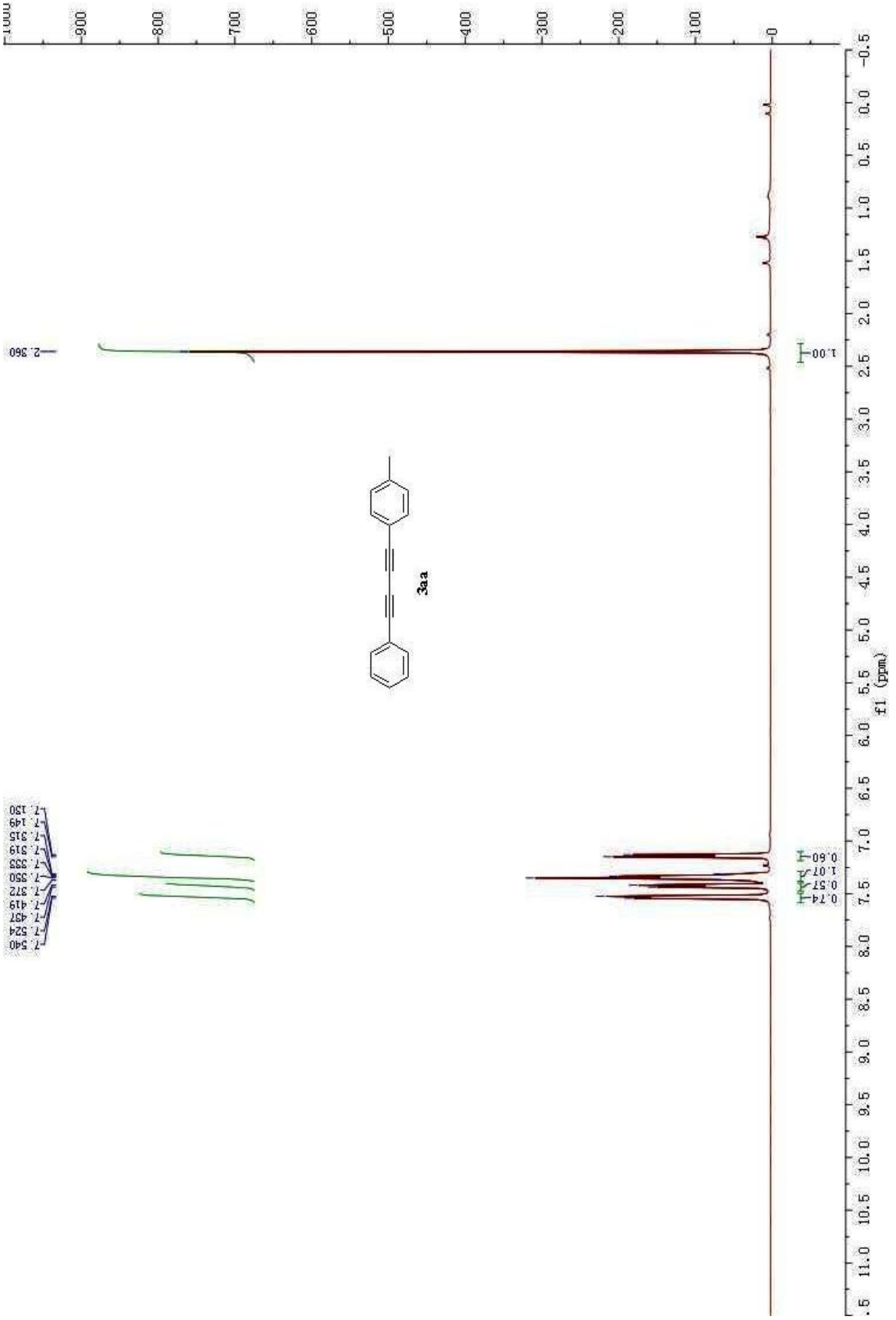
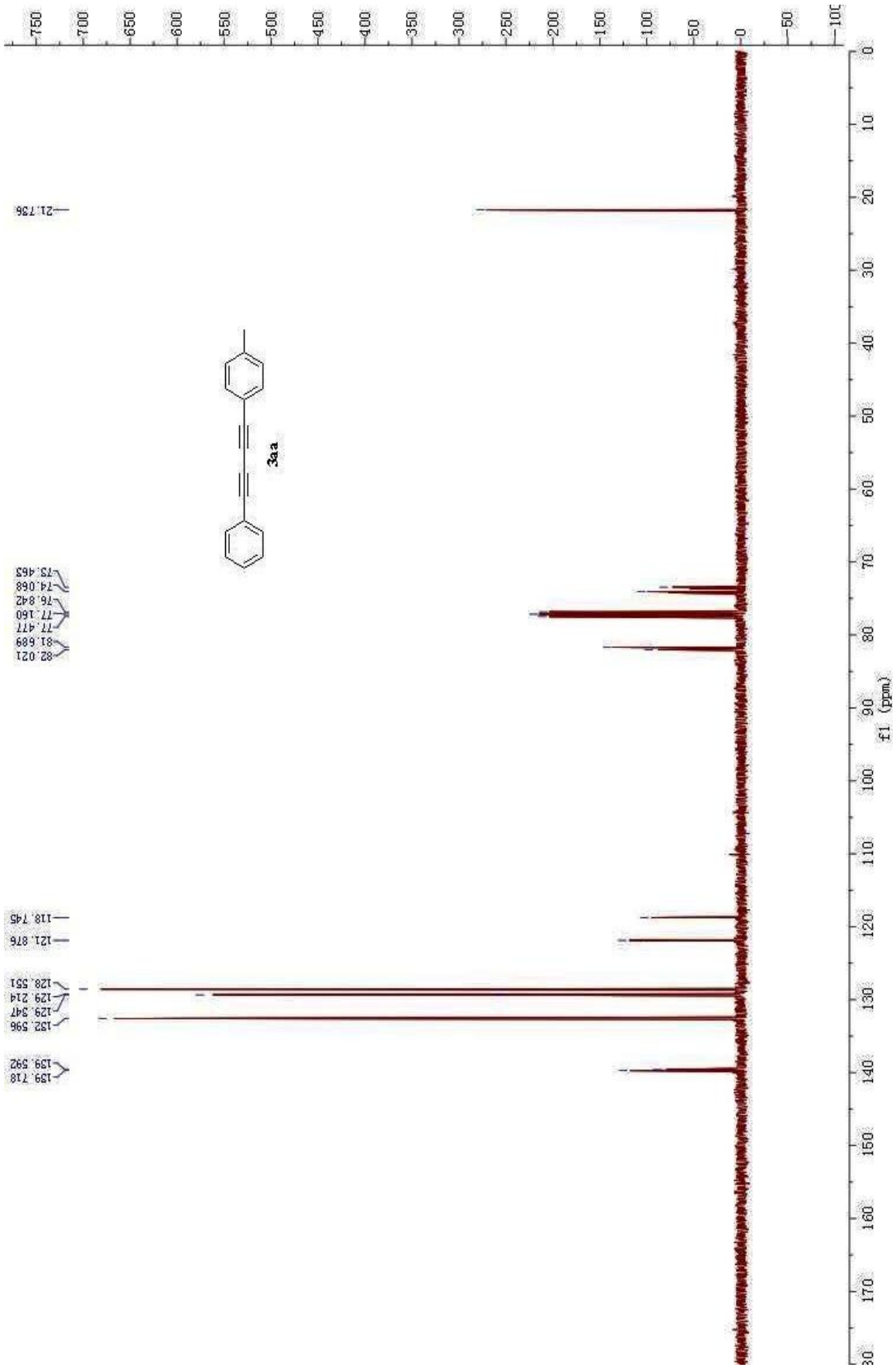
V. ^{1}H and ^{13}C NMR Spectra of Coupling Products

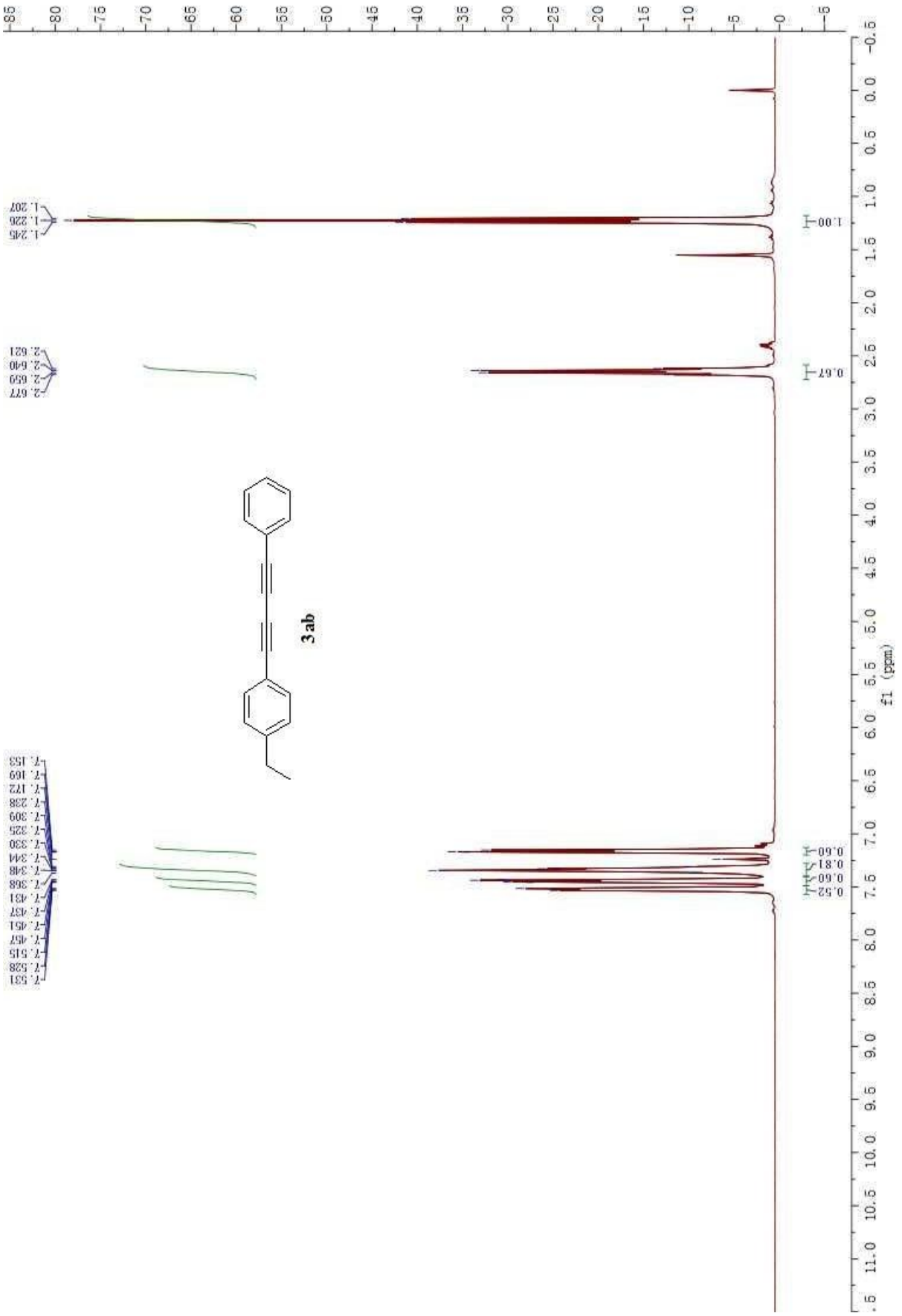
^{1}H NMR Spectrum of 1-methyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3aa)**



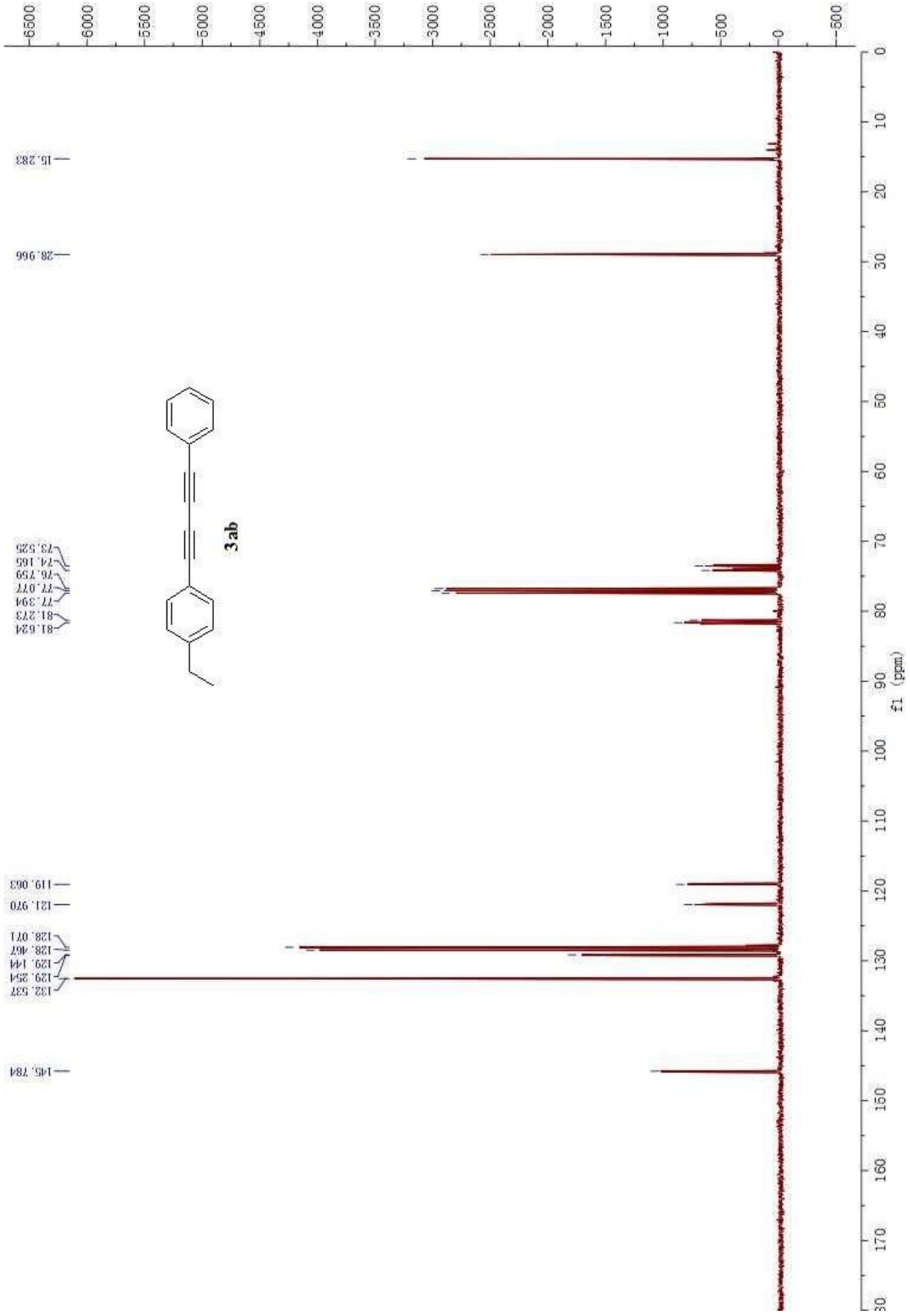
^{13}C NMR Spectrum of 1-methyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3aa)**



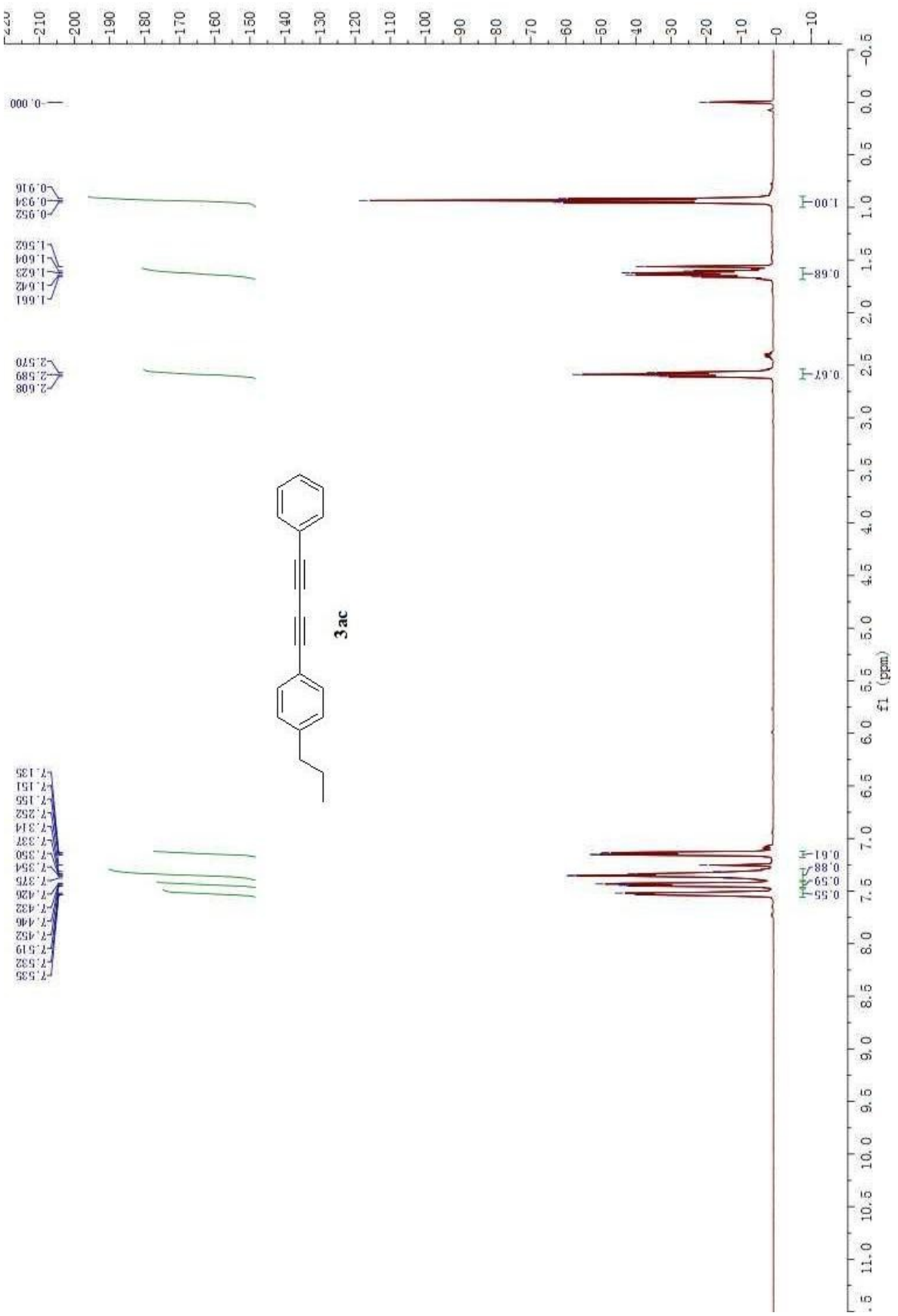
^{1}H NMR Spectrum of 1-ethyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ab)**



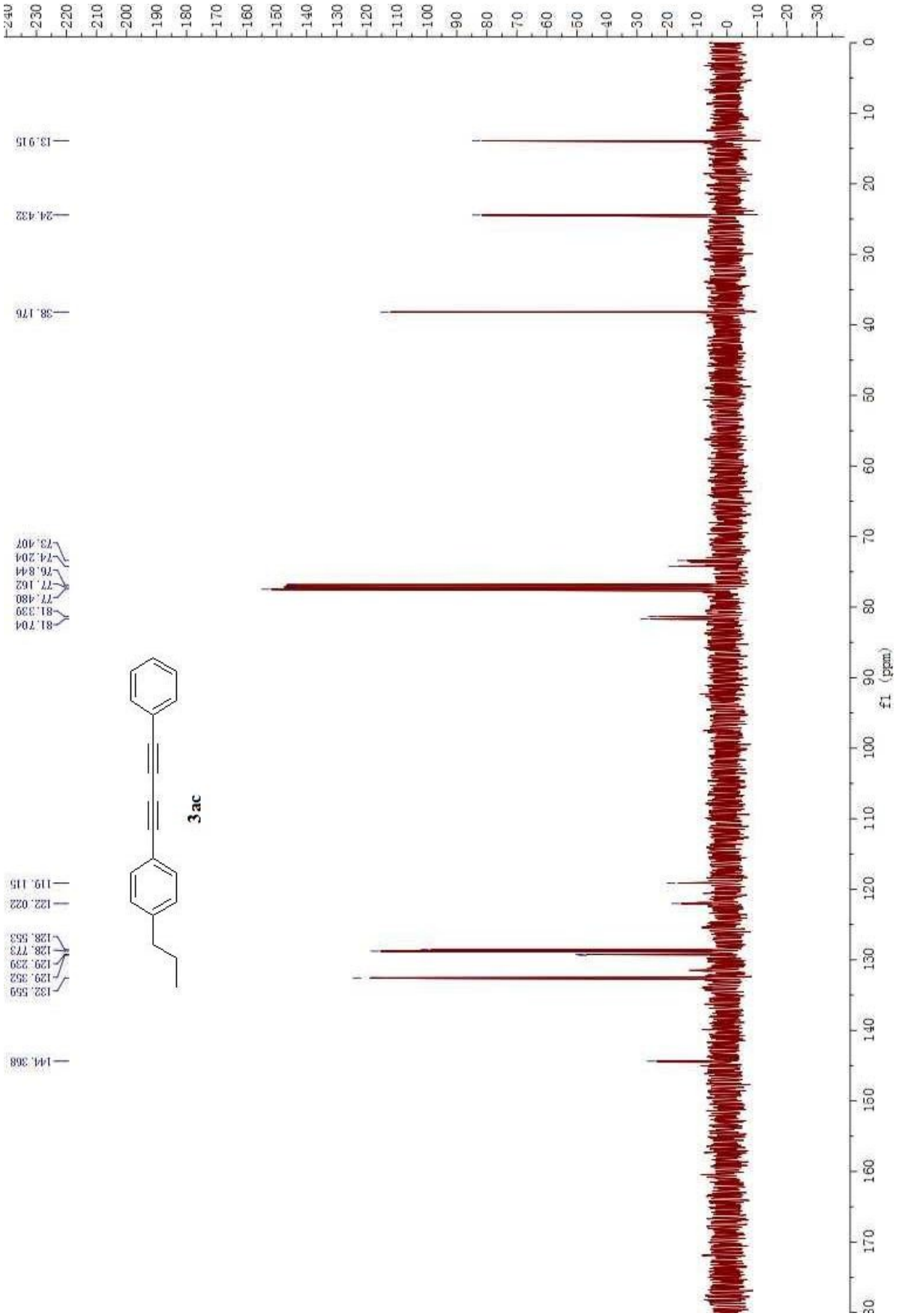
^{13}C NMR Spectrum of 1-ethyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ab)**



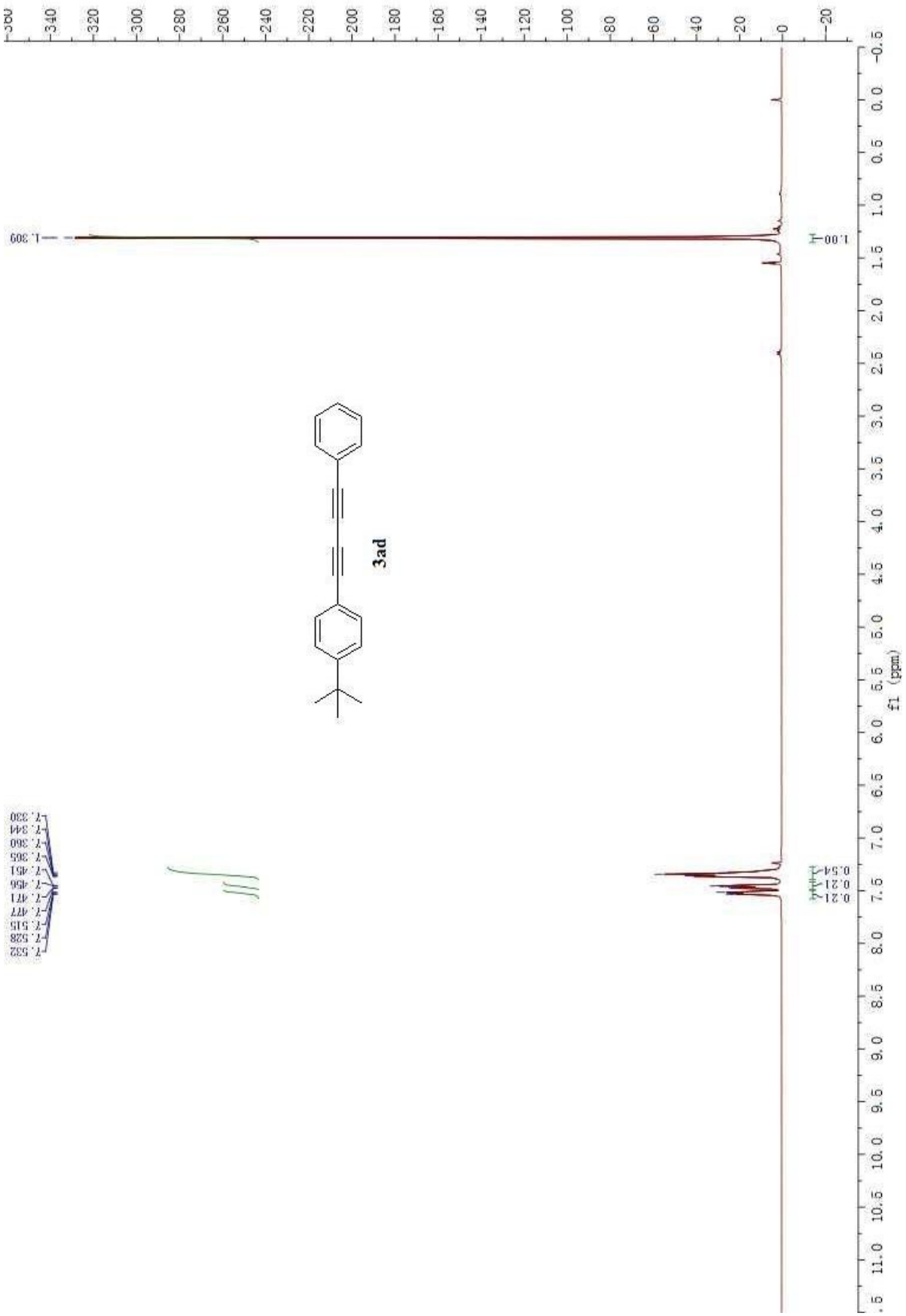
^{1}H NMR Spectrum of 1-propyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ac)**



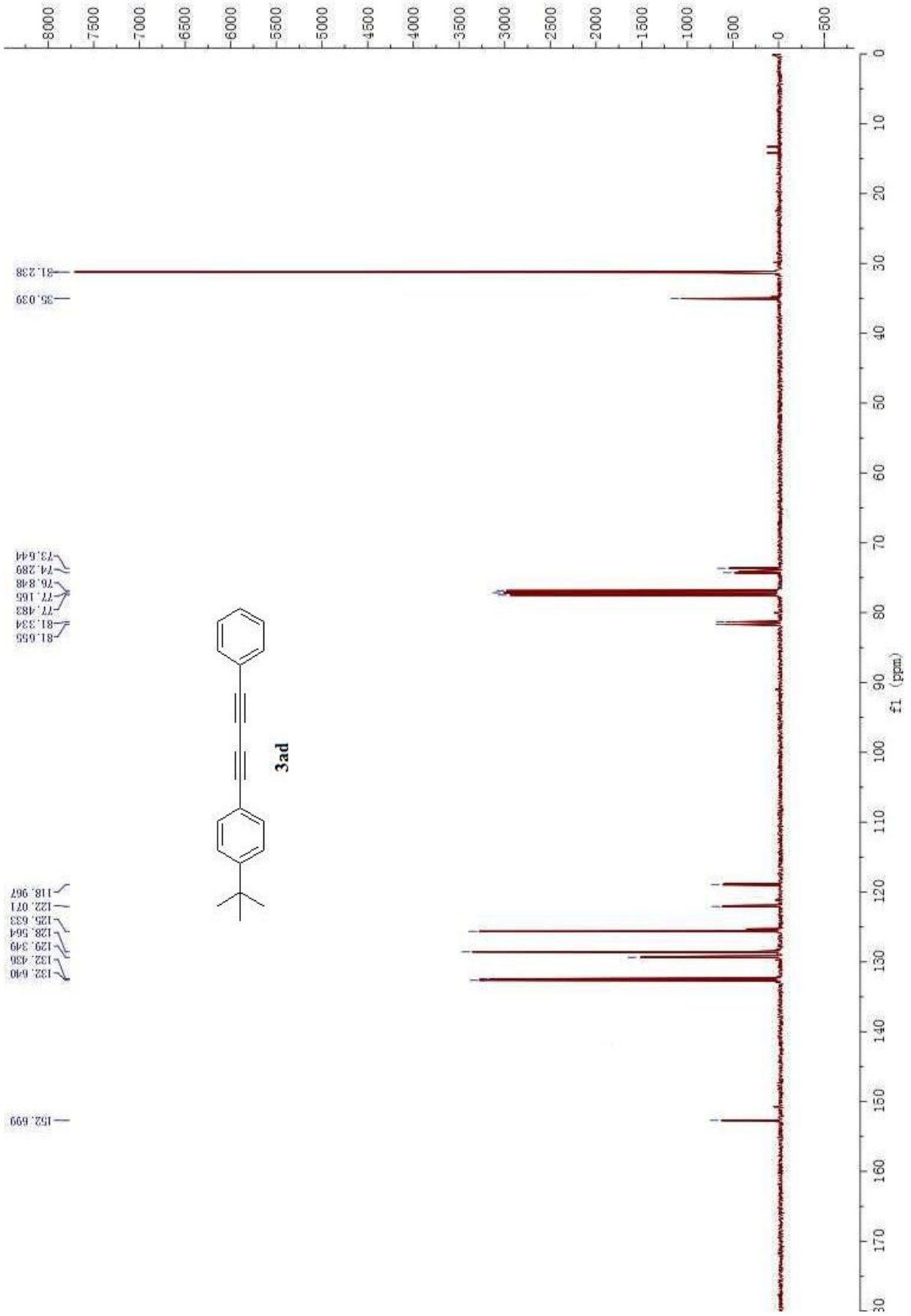
^{13}C NMR Spectrum of 1-propyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ac)**



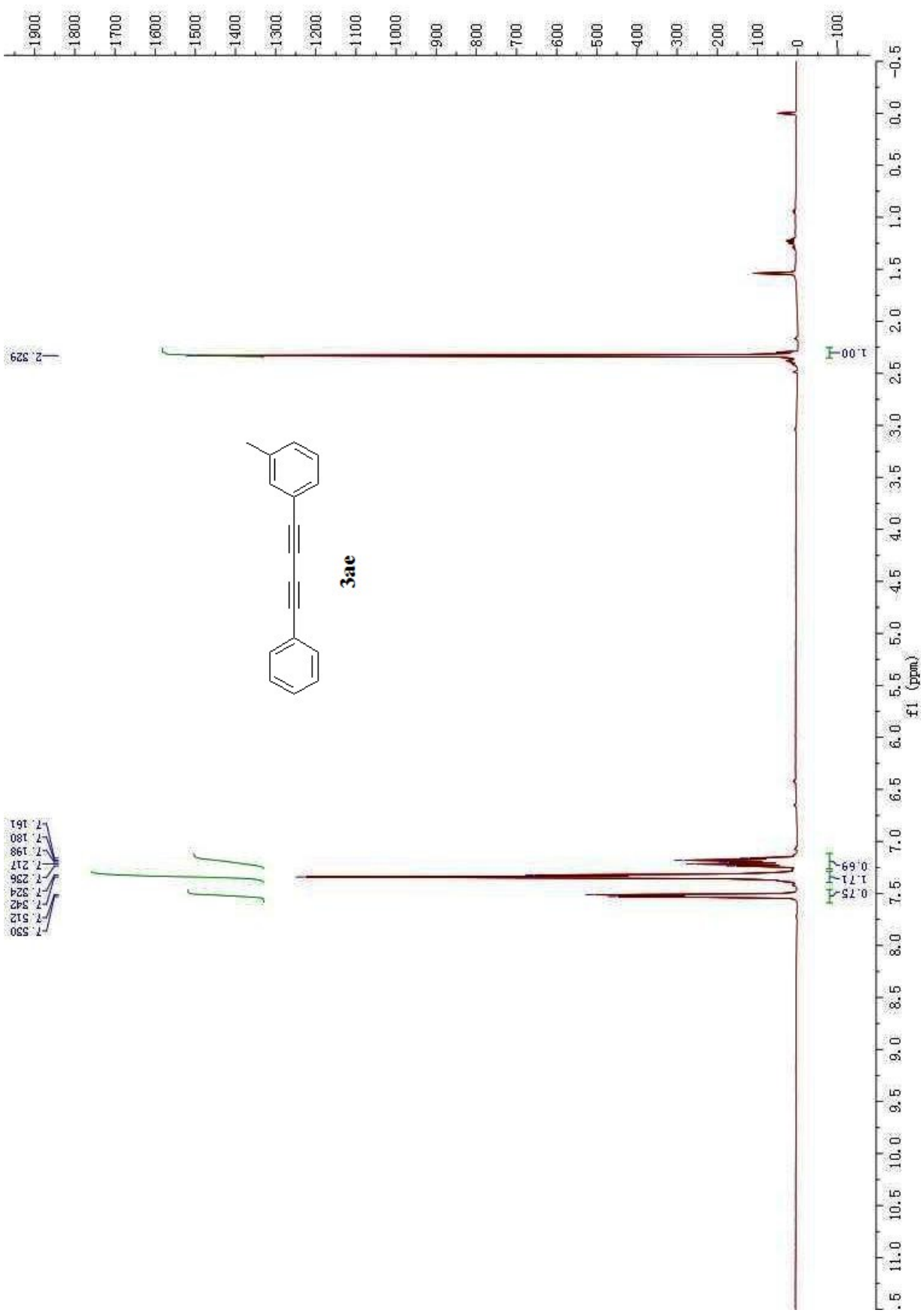
^{1}H NMR Spectrum of 1-tert-butyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ad)**



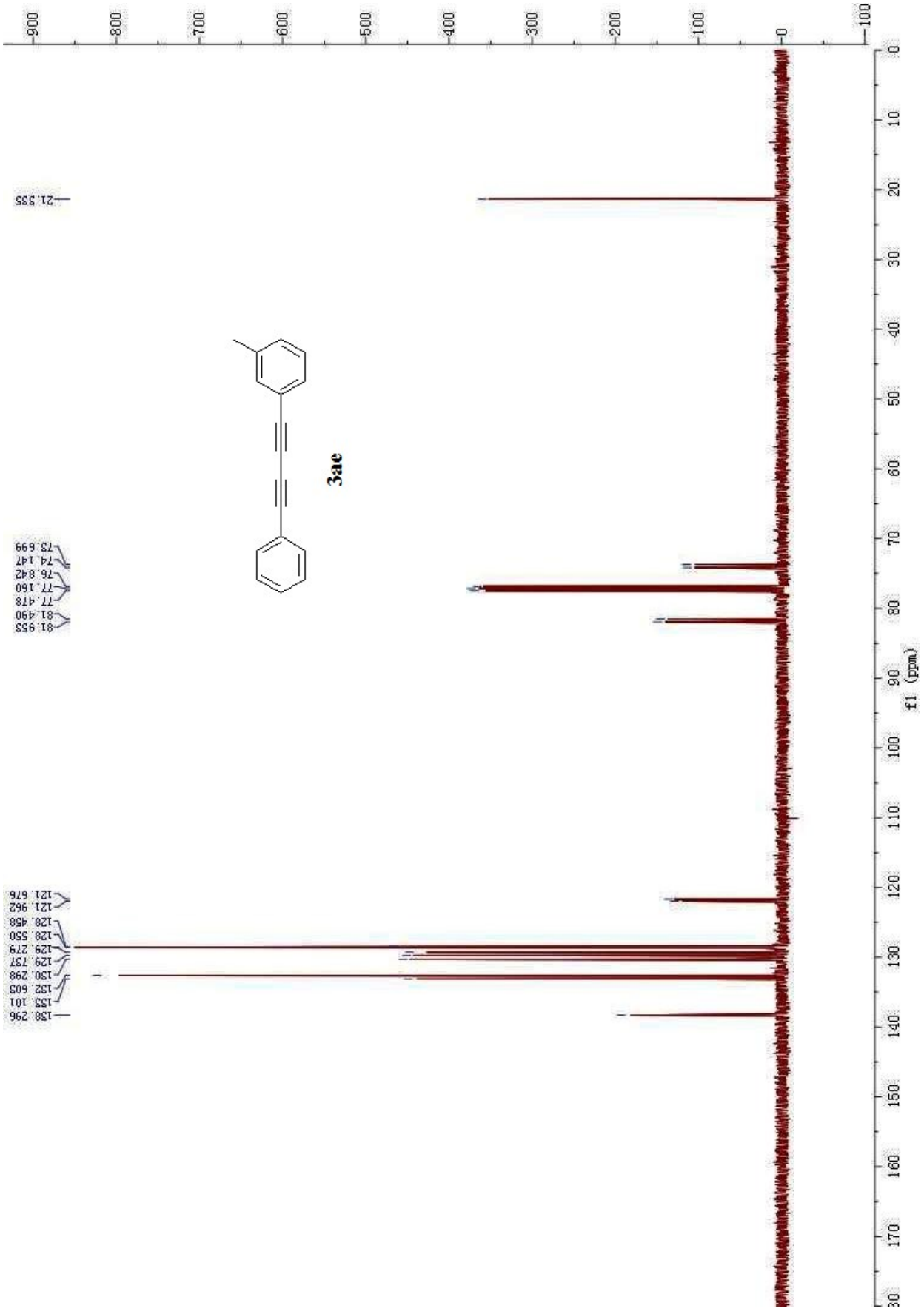
^{13}C NMR Spectrum of 1-propyl-4-(4-phenylbuta-1,3-diynyl)benzene **(3ad)**



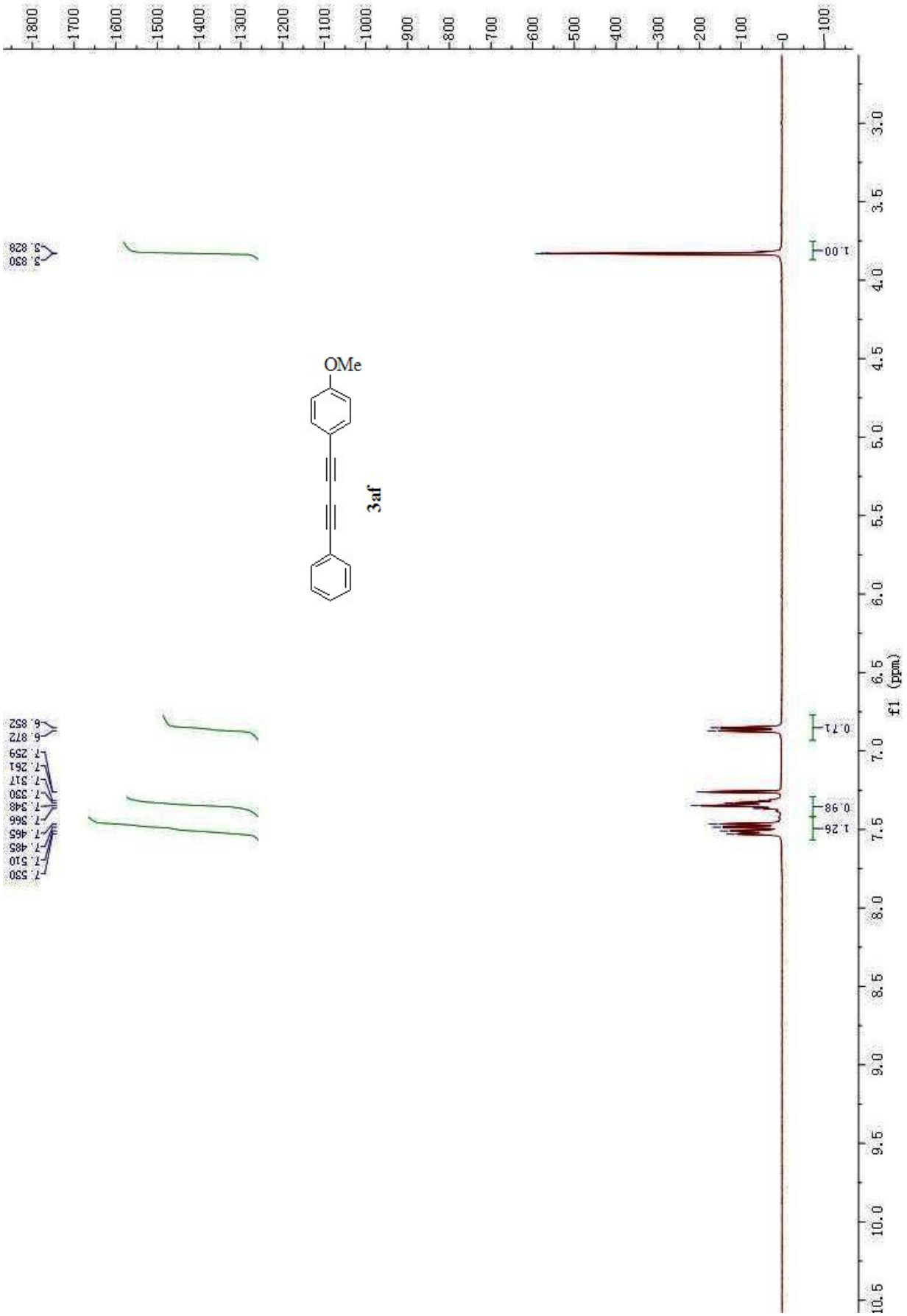
^{1}H NMR Spectrum of 1-methyl-3-(4-phenylbuta-1,3-diynyl)benzene **(3ae)**



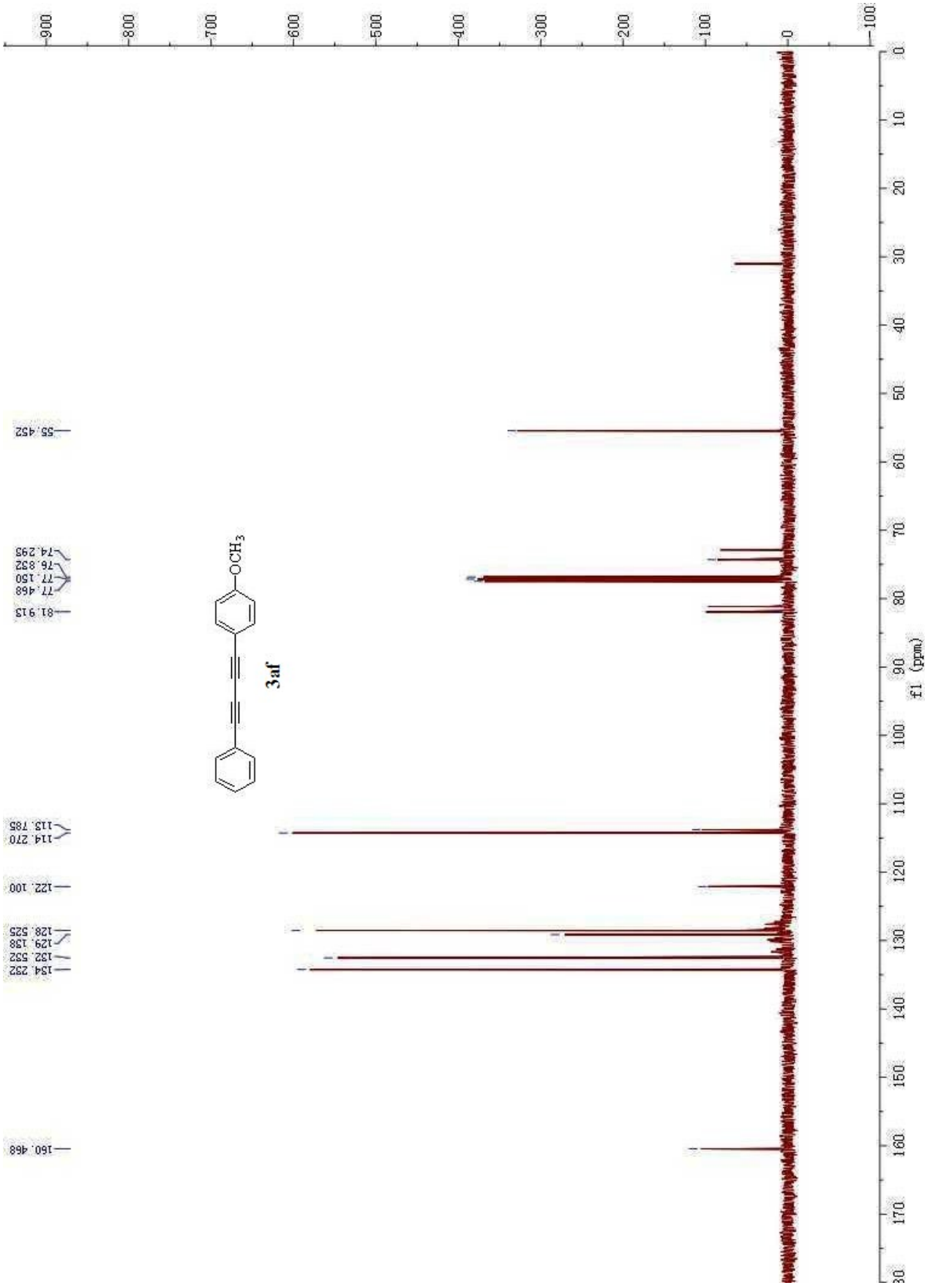
^{13}C NMR Spectrum of 1-methyl-3-(4-phenylbuta-1,3-diynyl)benzene **(3ae)**



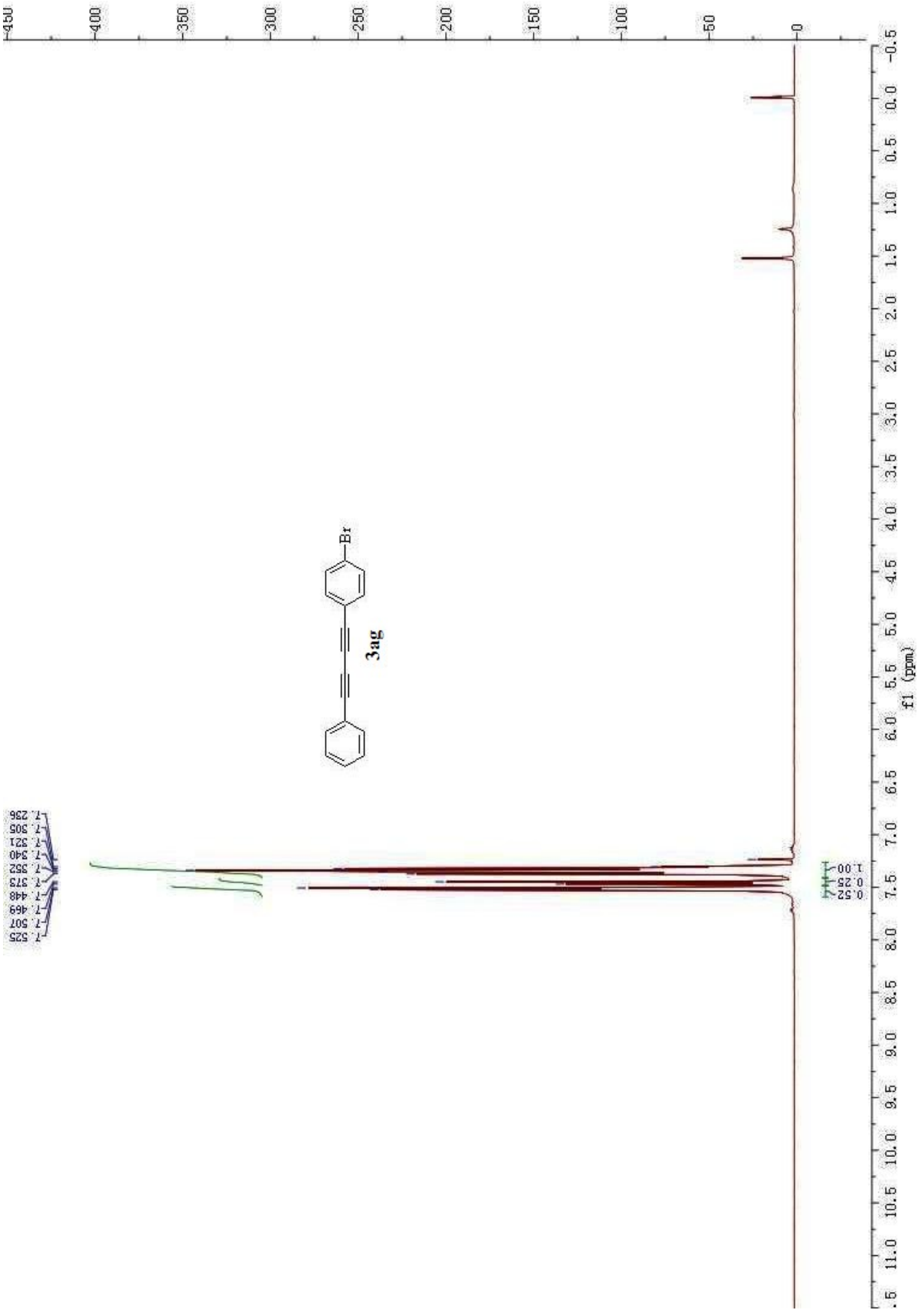
^{1}H NMR Spectrum of 1-methoxy-4-(4-phenylbuta-1,3-diynyl)benzene **(3af)**



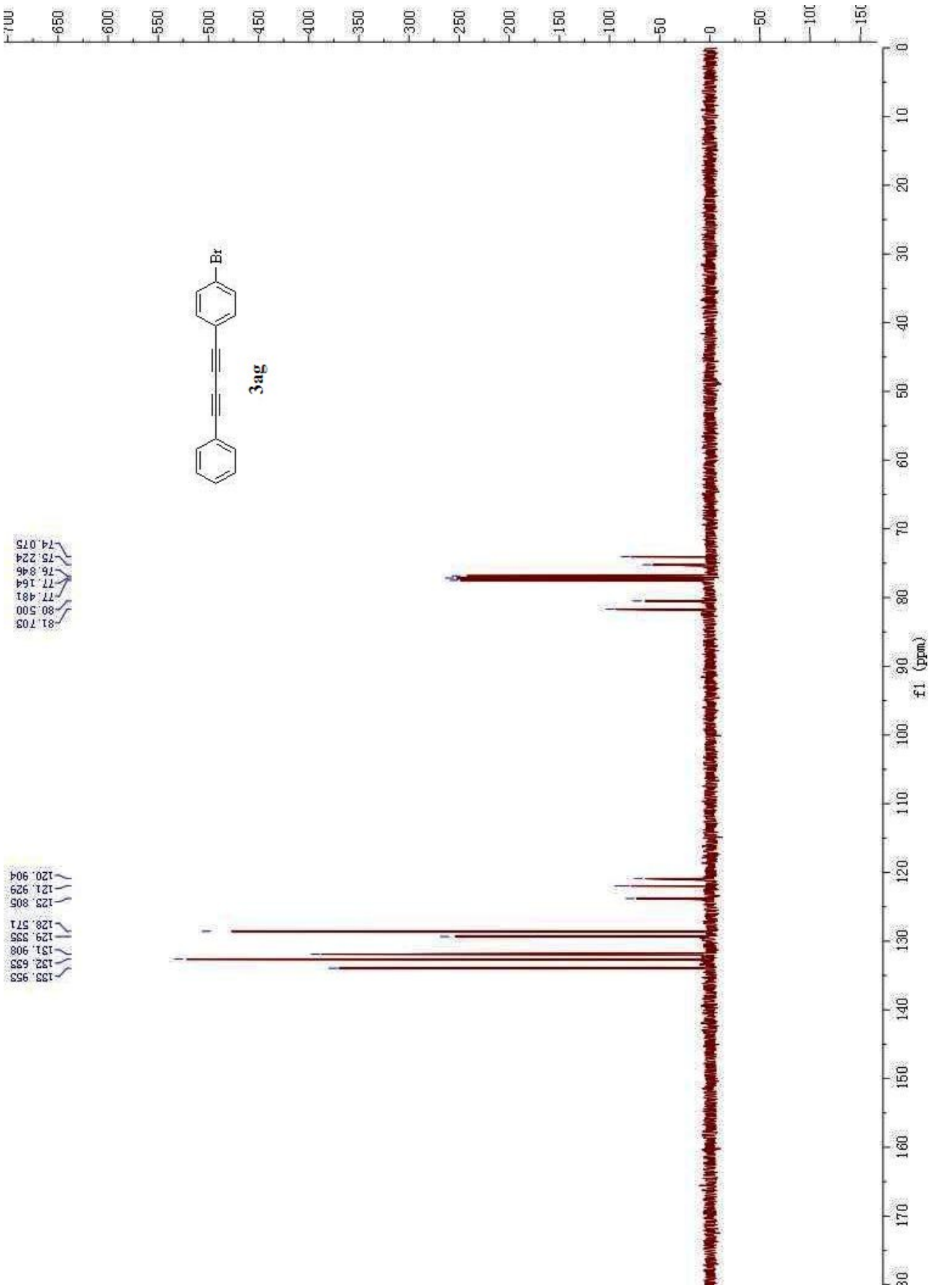
^{13}C NMR Spectrum of 1-methoxy-4-(4-phenylbuta-1,3-diynyl)benzene **(3af)**



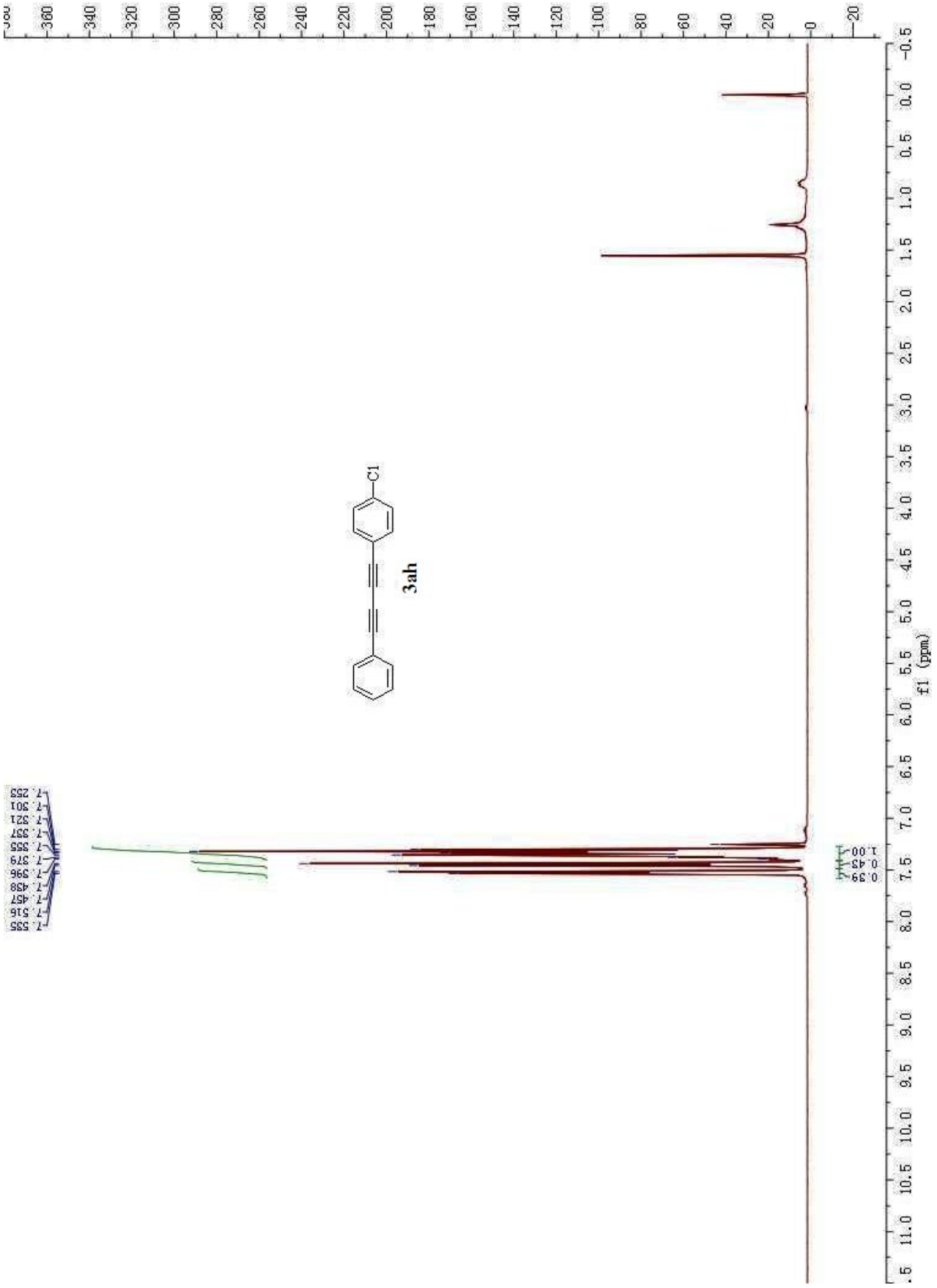
^{1}H NMR Spectrum of 1-bromo-4-(4-phenylbuta-1,3-diynyl)benzene **(3ag)**



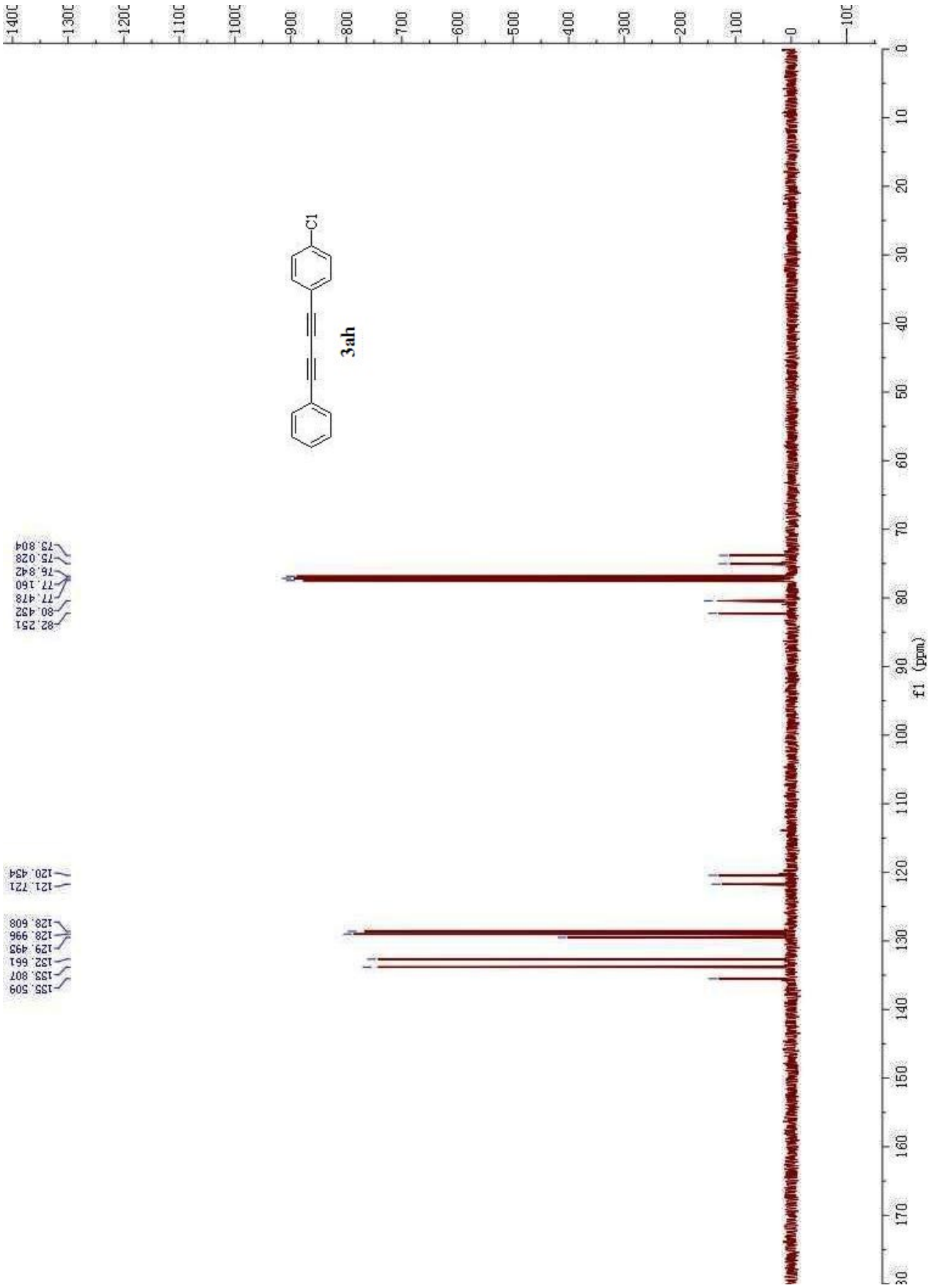
^{13}C NMR Spectrum of 1-bromo-4-(4-phenylbuta-1,3-diynyl)benzene **(3ag)**



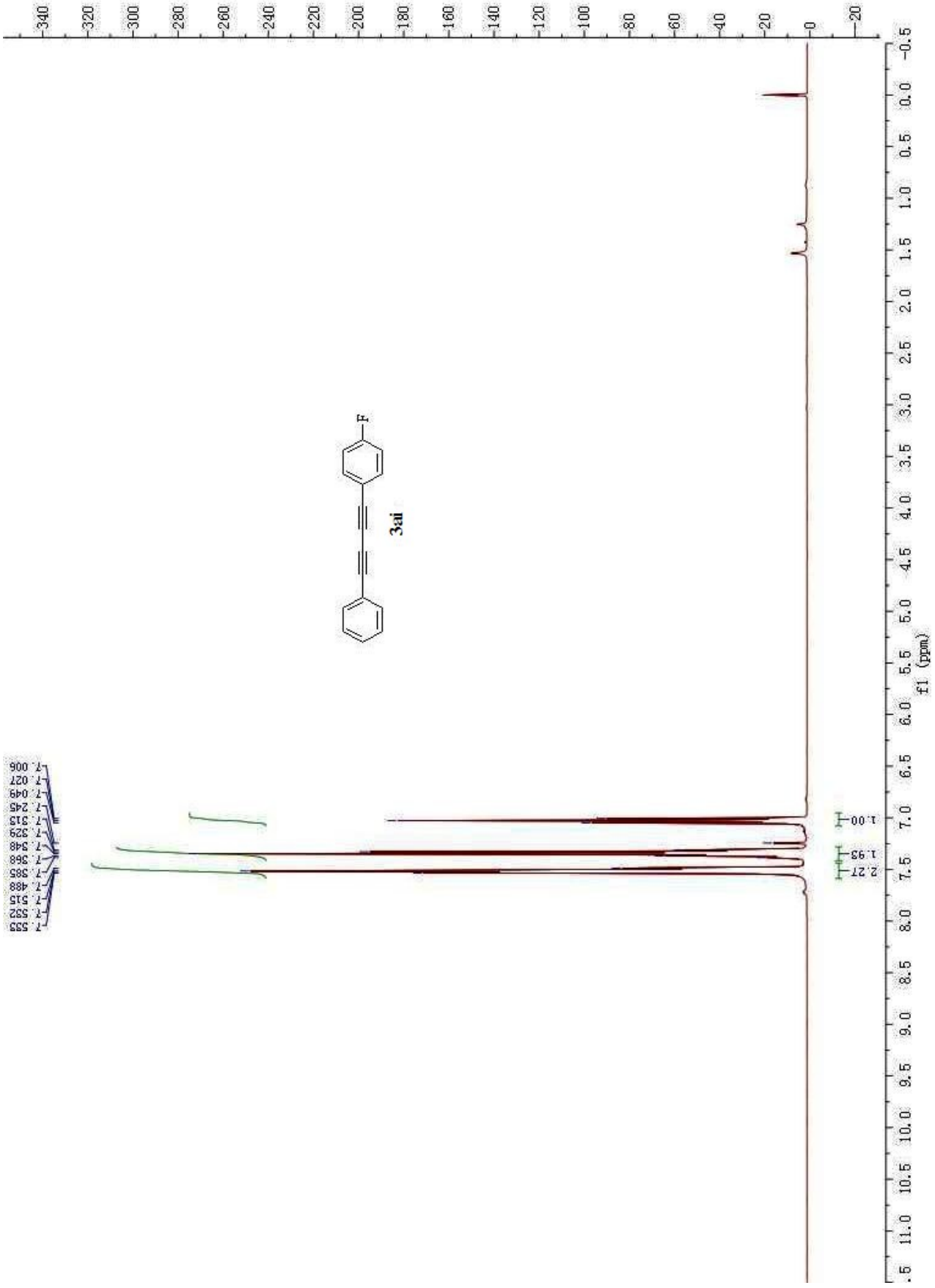
^{1}H NMR Spectrum of 1-chloro-4-(4-phenylbuta-1,3-diynyl)benzene **(3ah)**



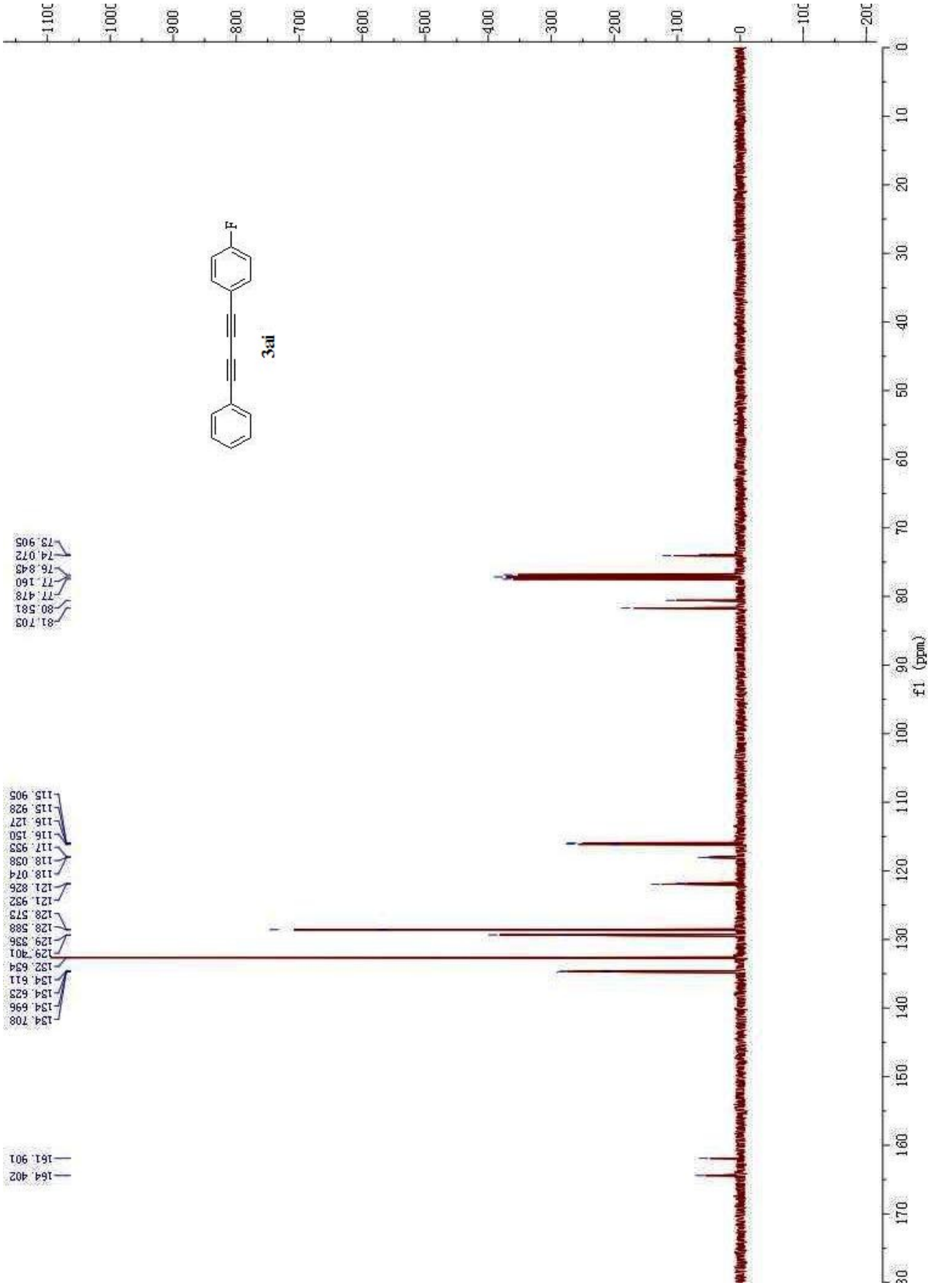
^{13}C NMR Spectrum of 1-chloro-4-(4-phenylbuta-1,3-diynyl)benzene **(3ah)**



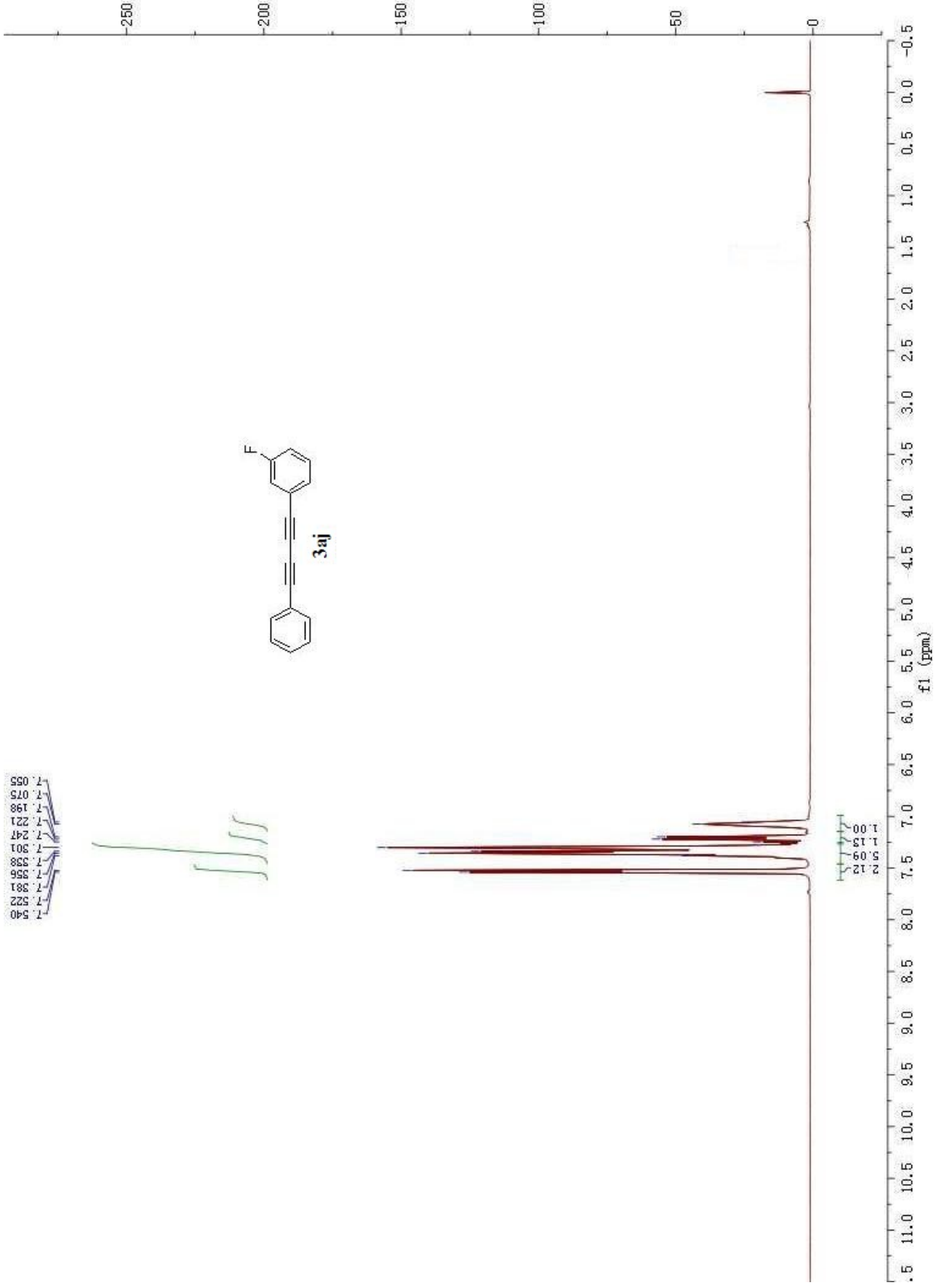
^{1}H NMR Spectrum of 1-fluoro-4-(4-phenylbuta-1,3-diynyl)benzene **(3ai)**



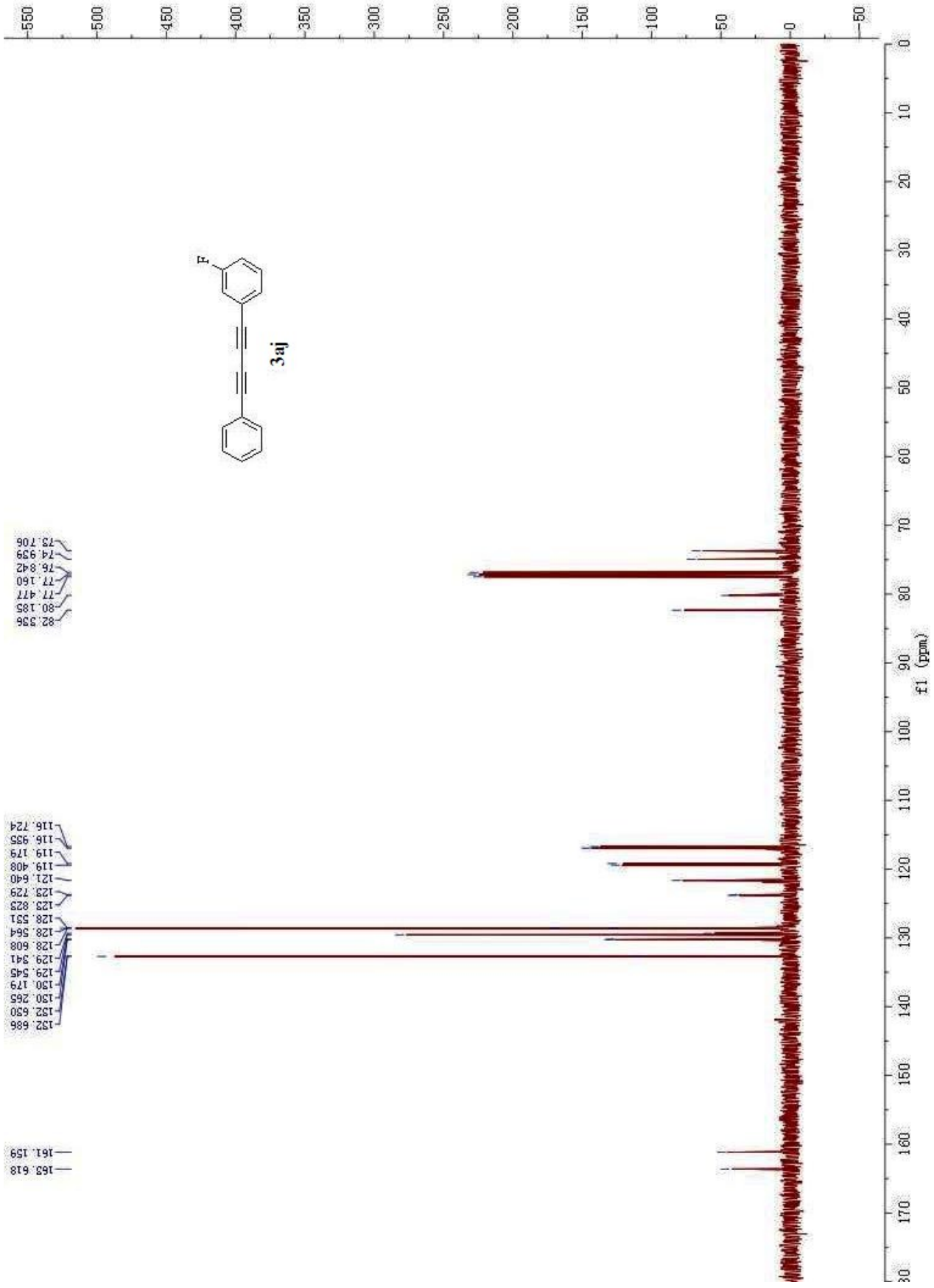
^{13}C NMR Spectrum of 1-fluoro-4-(4-phenylbuta-1,3-diynyl)benzene **(3ai)**



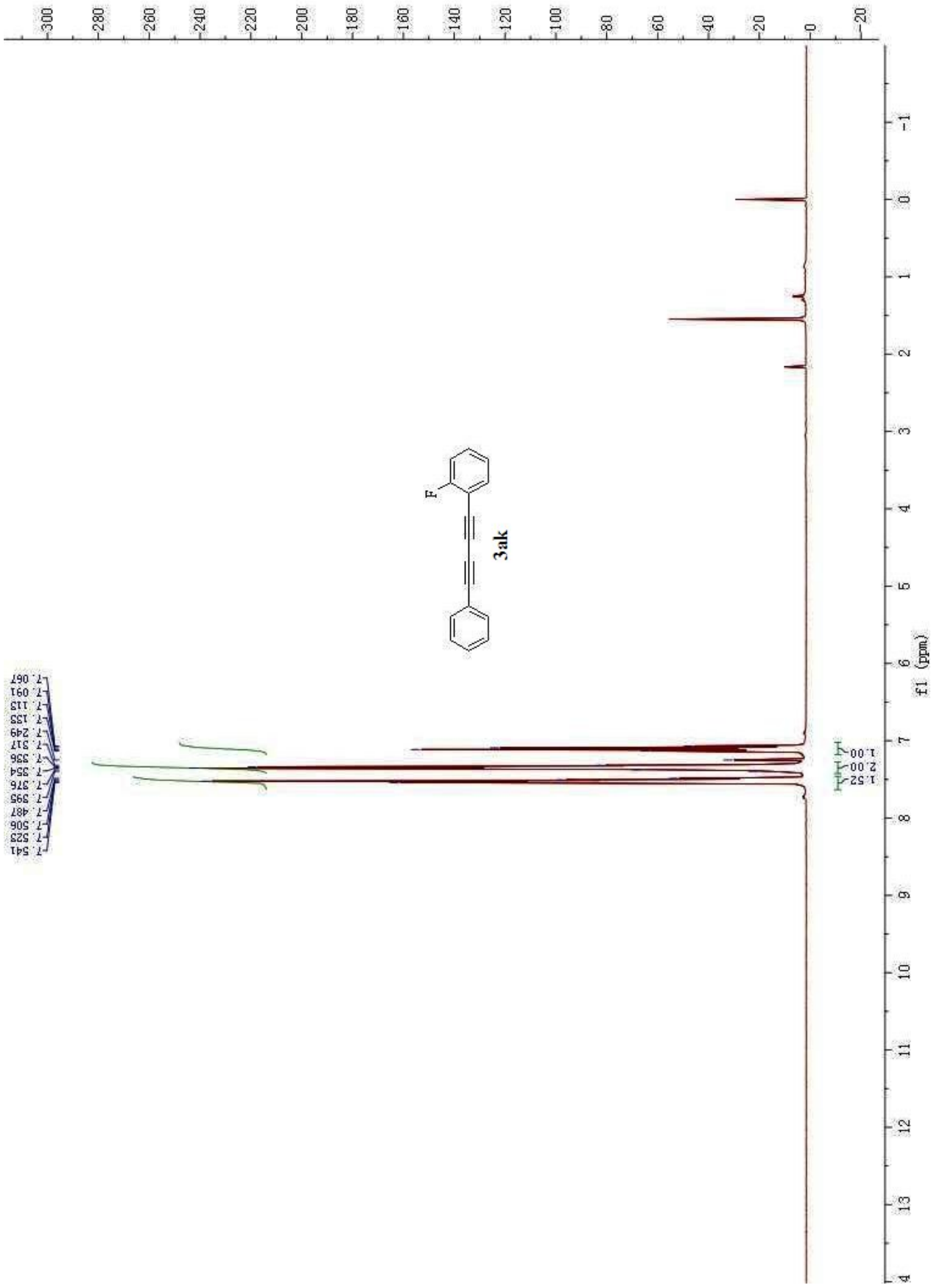
^{1}H NMR Spectrum of 1-fluoro-3-(4-phenylbuta-1,3-diynyl)benzene **(3aj)**



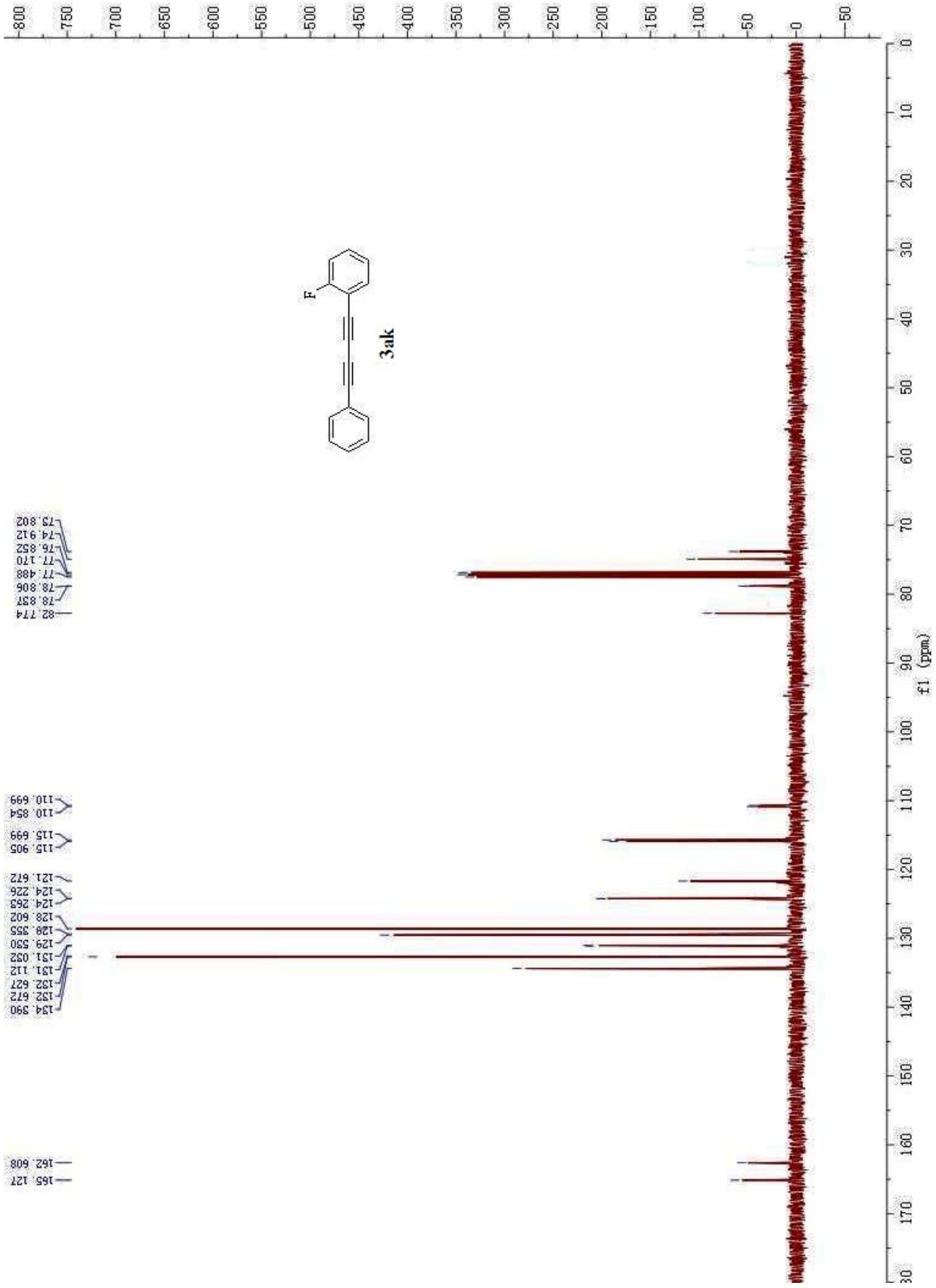
^{13}C NMR Spectrum of 1-fluoro-3-(4-phenylbuta-1,3-diynyl)benzene **(3aj)**



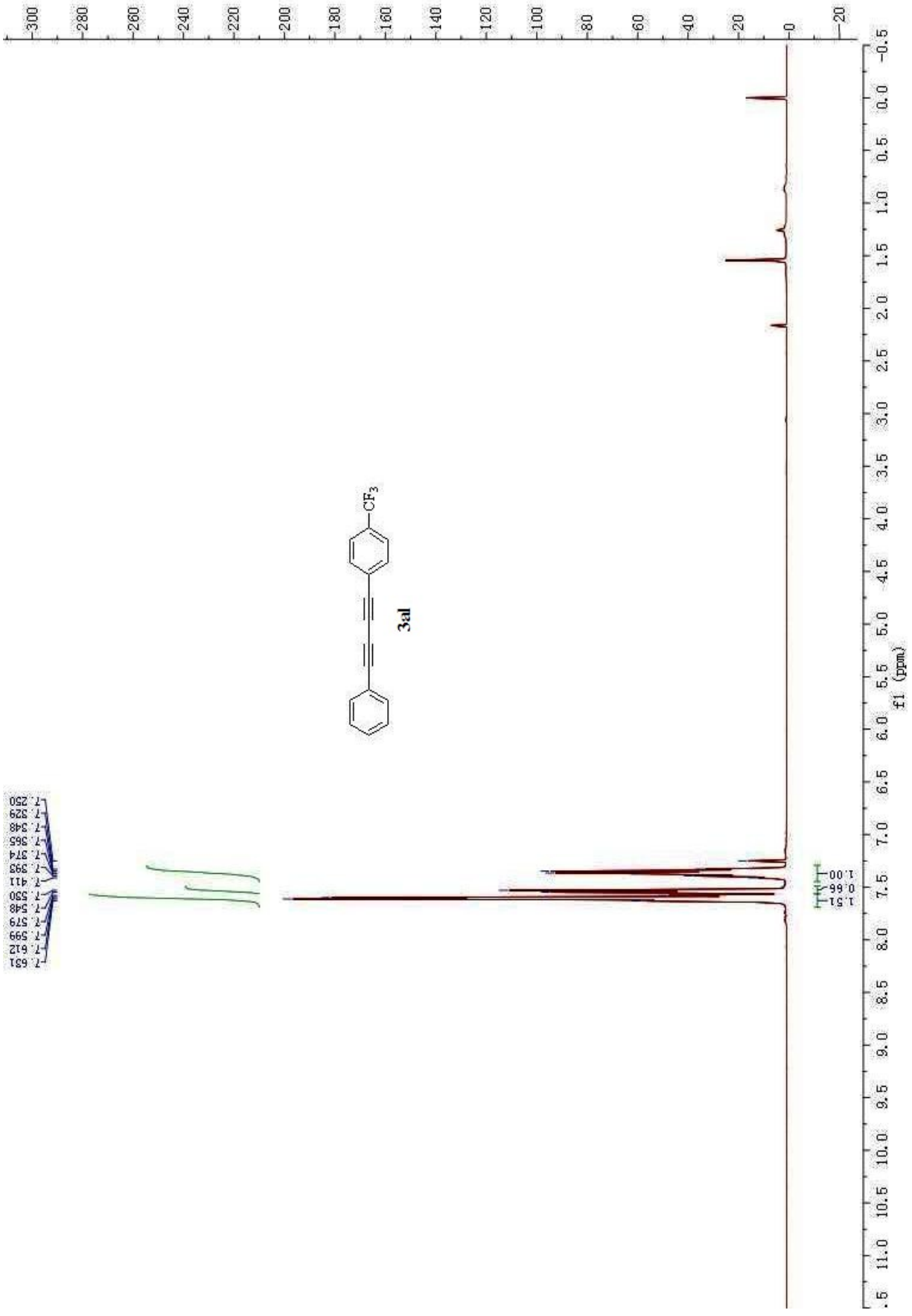
^{1}H NMR Spectrum of 1-fluoro-2-(4-phenylbuta-1,3-diynyl)benzene **(3ak)**



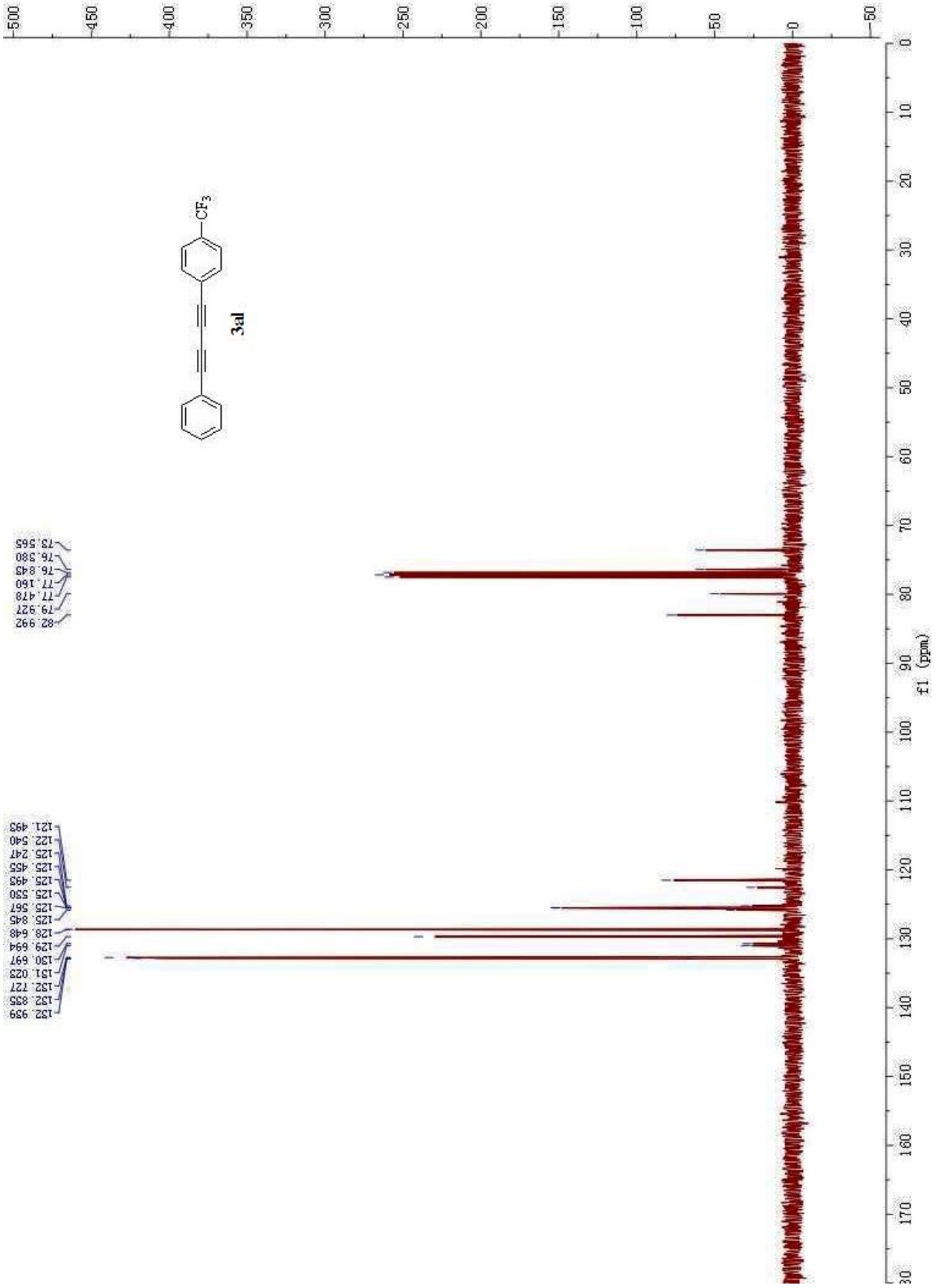
^{13}C NMR Spectrum of 1-fluoro-2-(4-phenylbuta-1,3-diynyl)benzene **(3ak)**



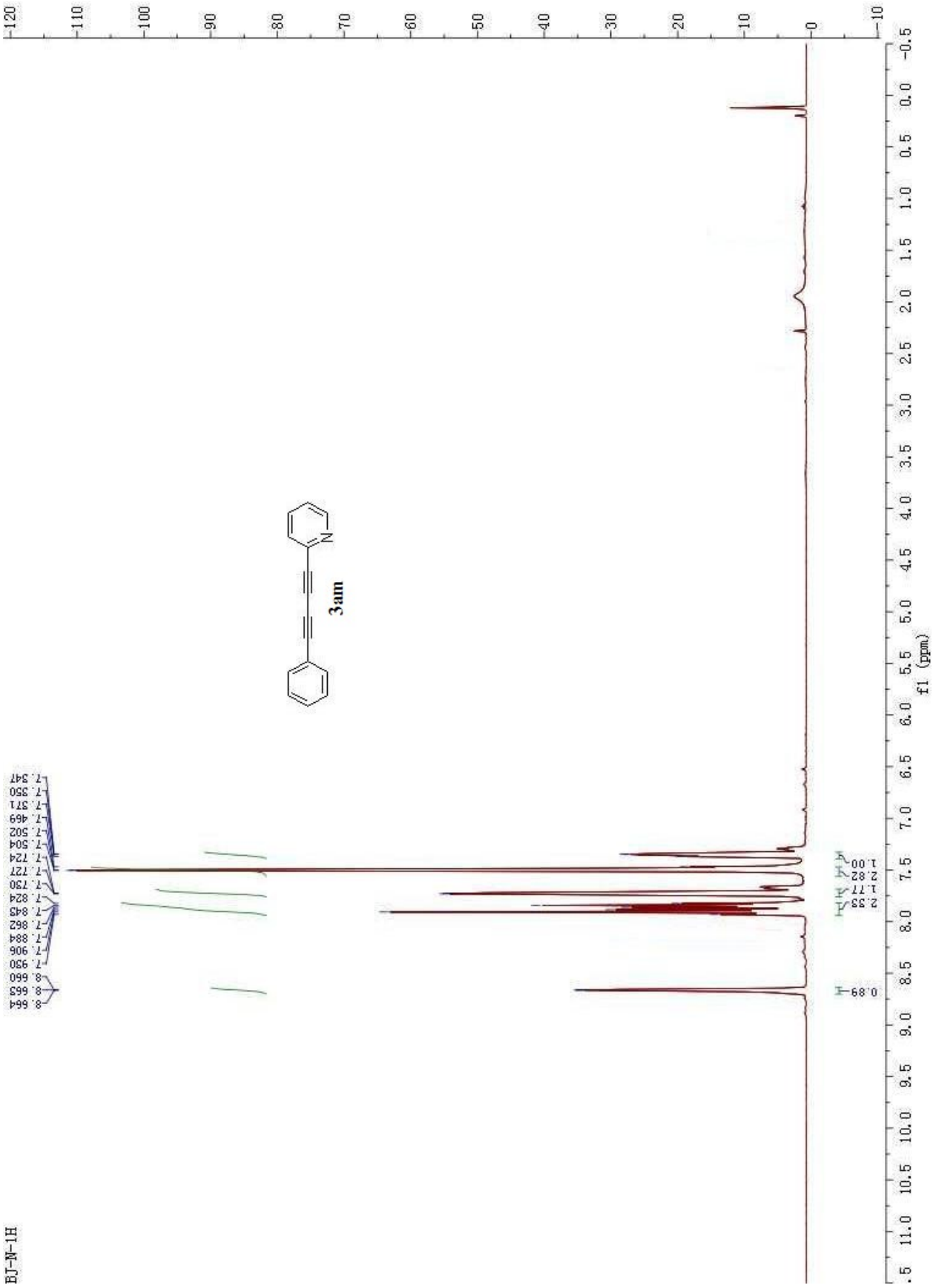
^{1}H NMR Spectrum of 1-(4-(4-(trifluoromethyl)phenyl)buta-1,3-diynyl)benzene **(3al)**



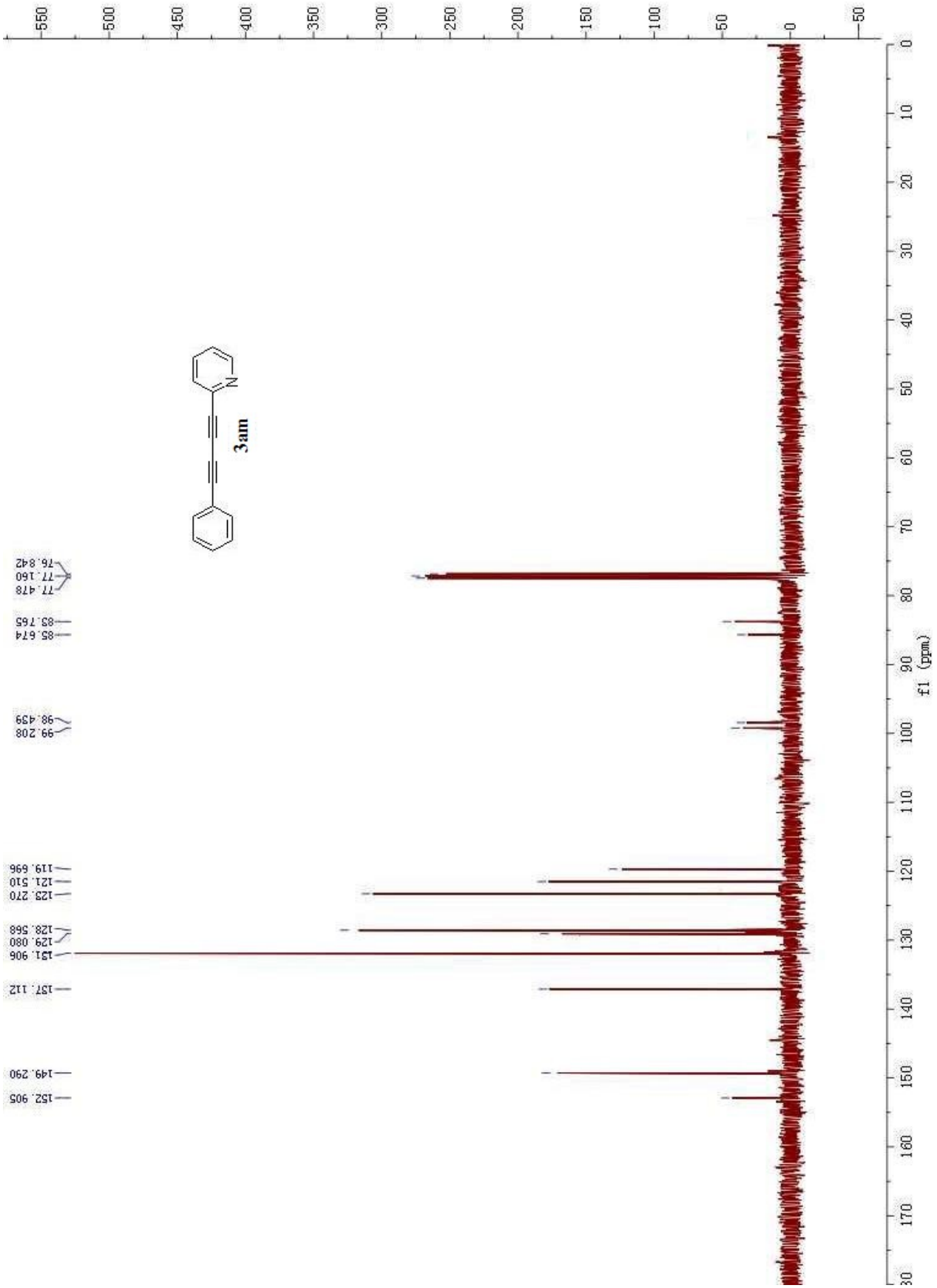
^{13}C NMR Spectrum of 1-(4-(4-(trifluoromethyl)phenyl)buta-1,3-diynyl)benzene **(3al)**



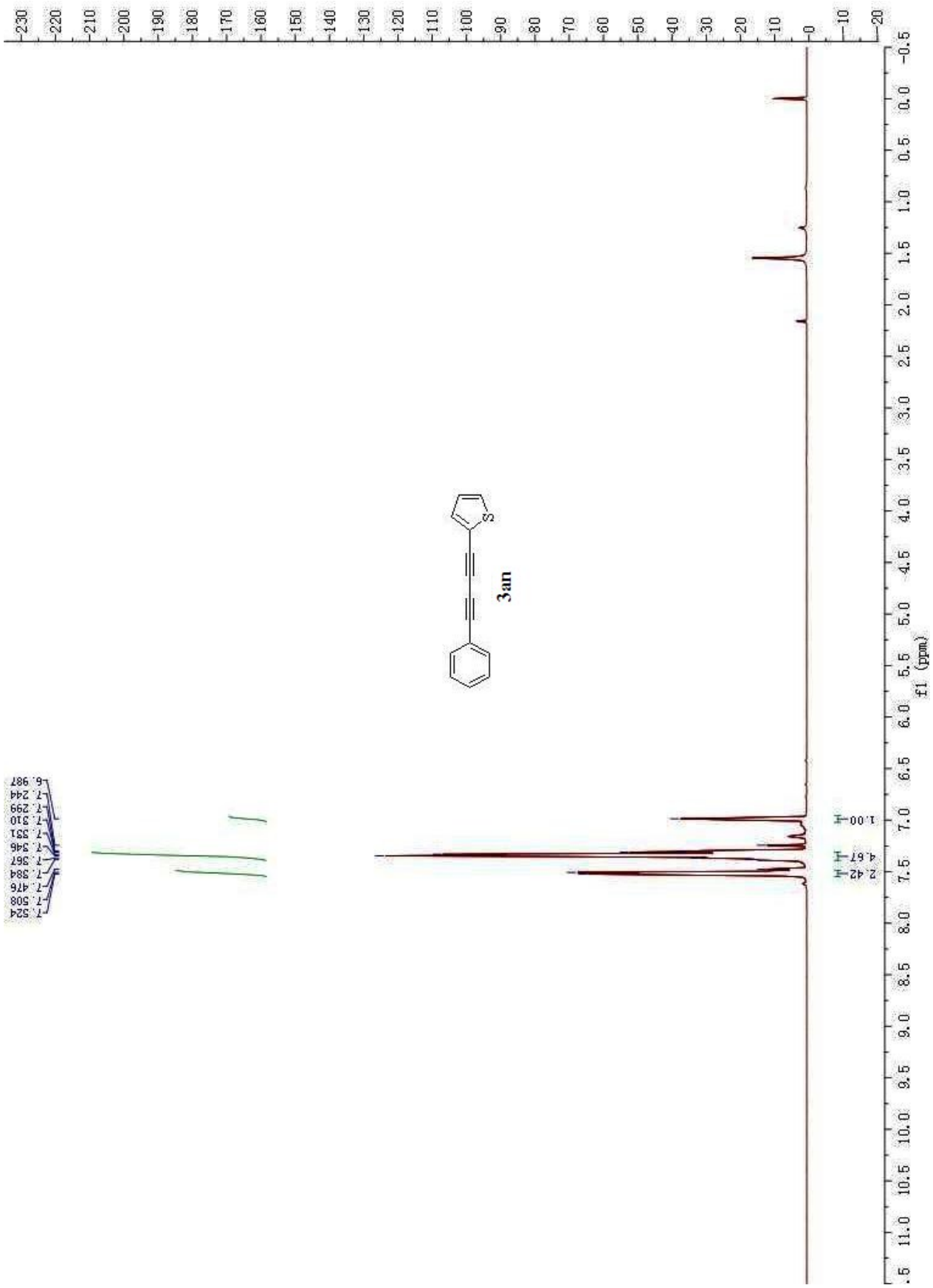
^{1}H NMR Spectrum of 2-(4-phenylbuta-1,3-diynyl)pyridine **(3am)**



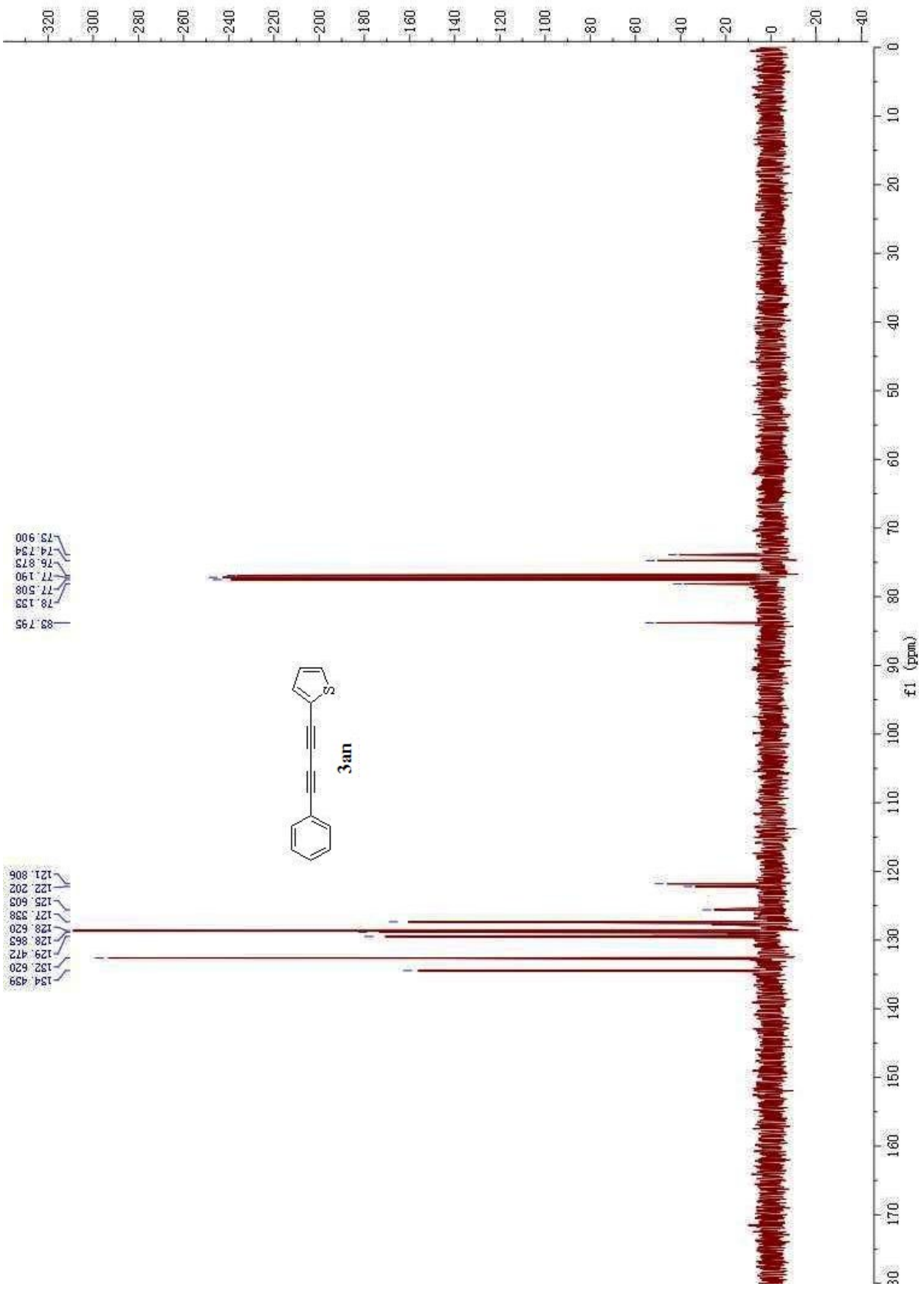
^{13}C NMR Spectrum of 2-(4-phenylbuta-1,3-diynyl)pyridine **(3am)**



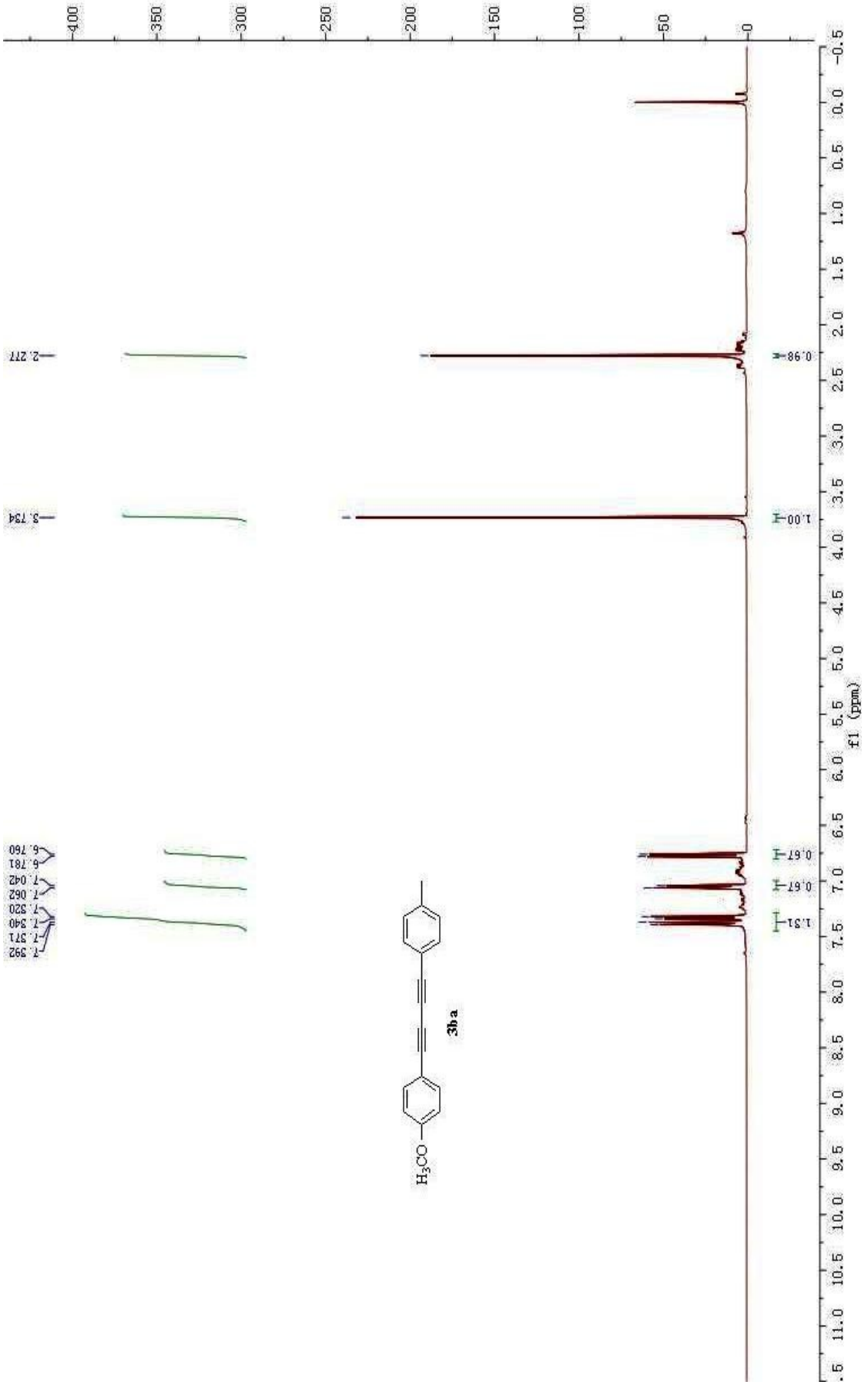
^{1}H NMR Spectrum of 2-(4-phenylbuta-1,3-diynyl)thiophene **(3an)**



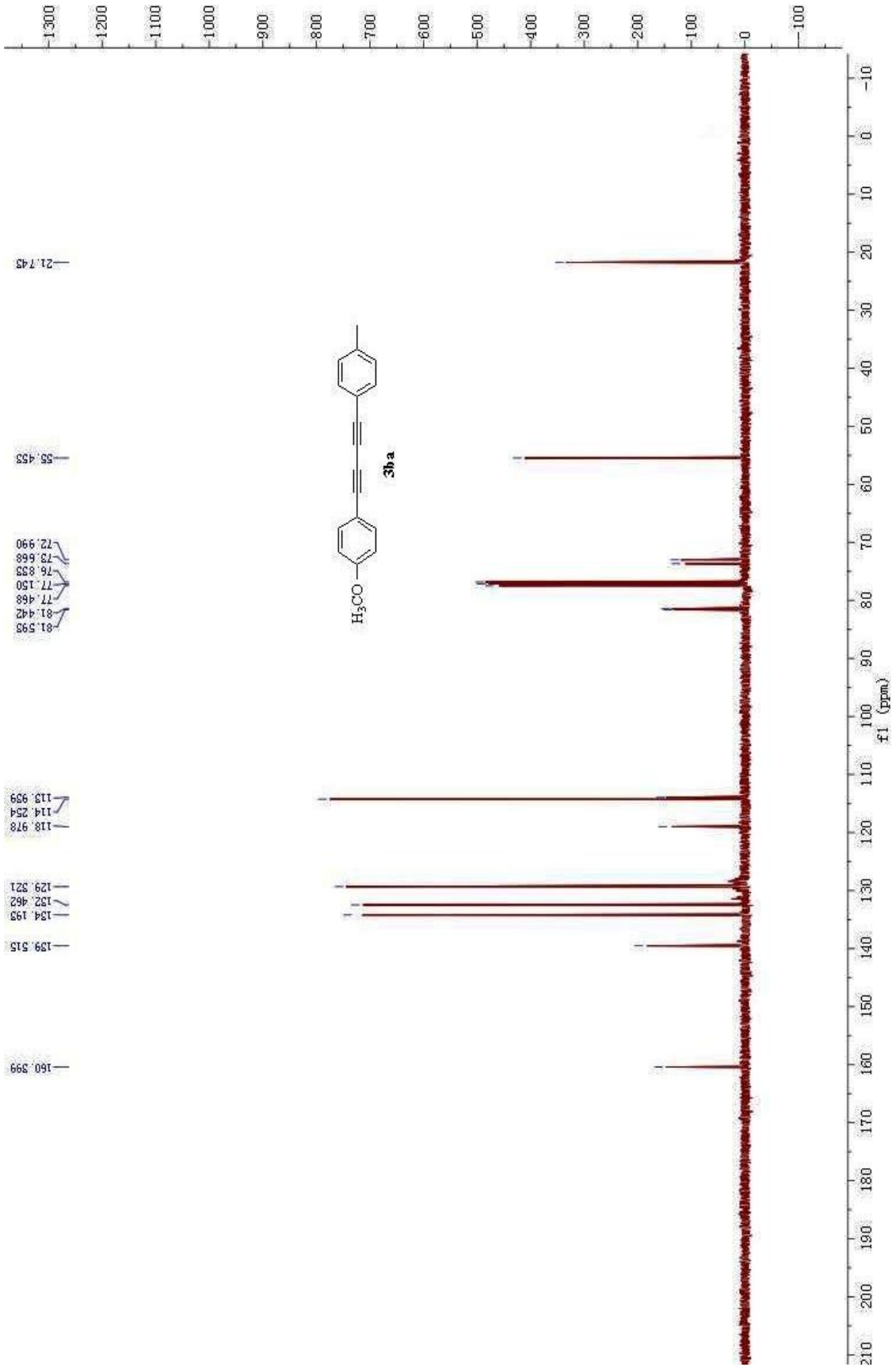
^{13}C NMR Spectrum of 2-(4-phenylbuta-1,3-diynyl)thiophene **(3an)**



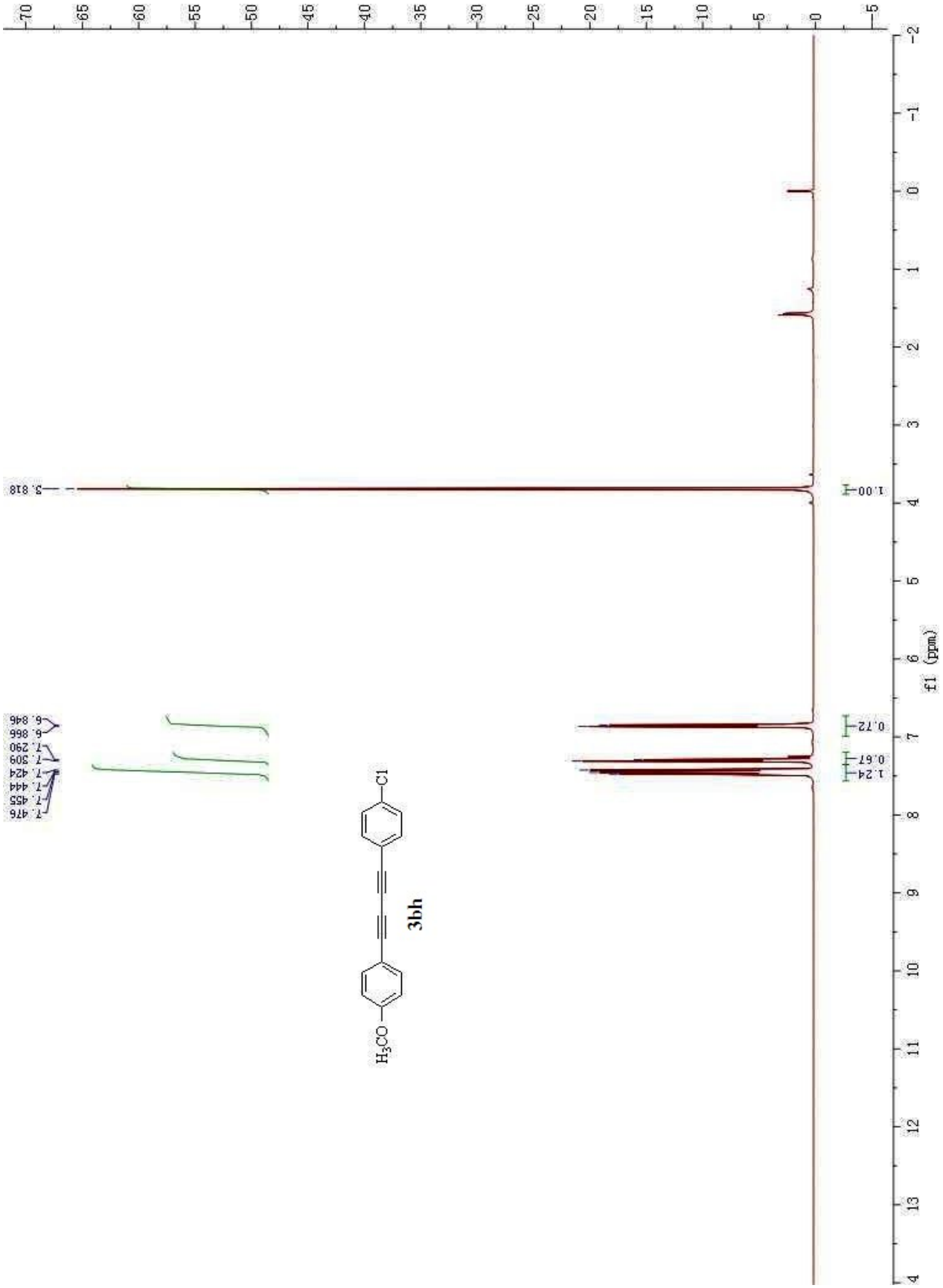
^{1}H NMR Spectrum of 1-methoxy-4-(4-p-tolylbuta-1,3-diynyl)benzene **(3ba)**



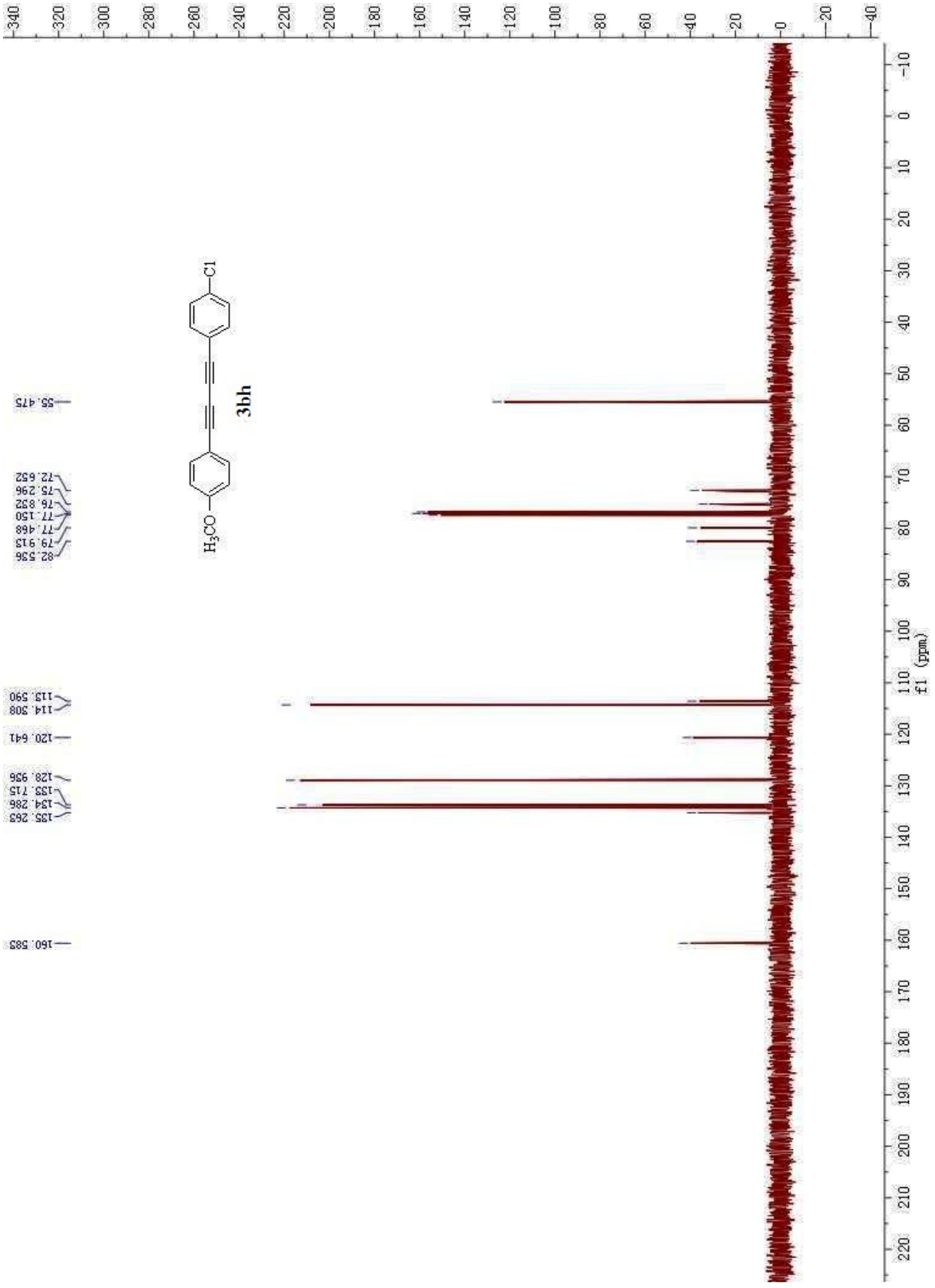
^{13}C NMR Spectrum of 1-methoxy-4-(4-p-tolylbuta-1,3-diynyl)benzene **(3ba)**



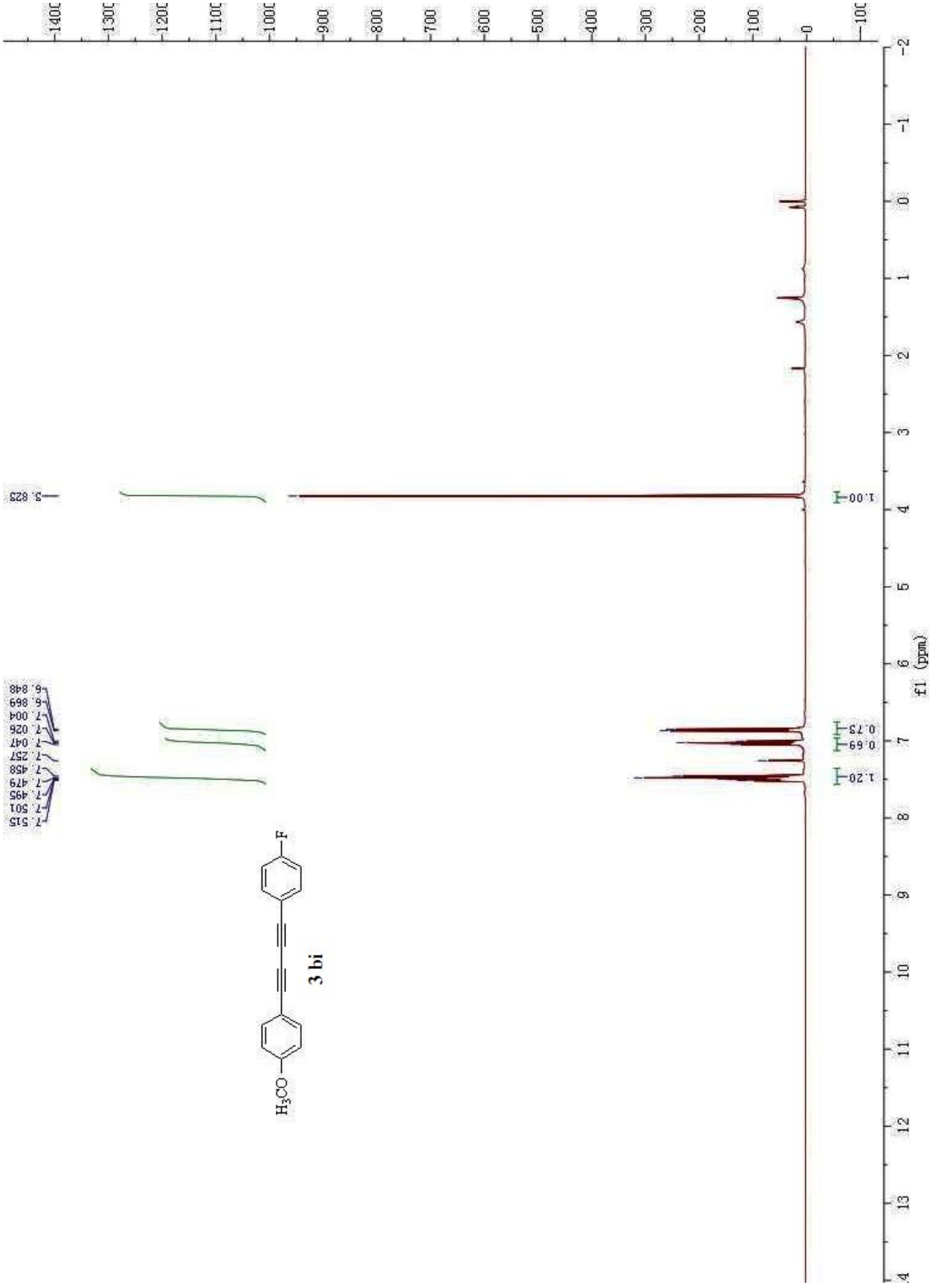
^{1}H NMR Spectrum of 1-(4-(4-chlorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bh)**



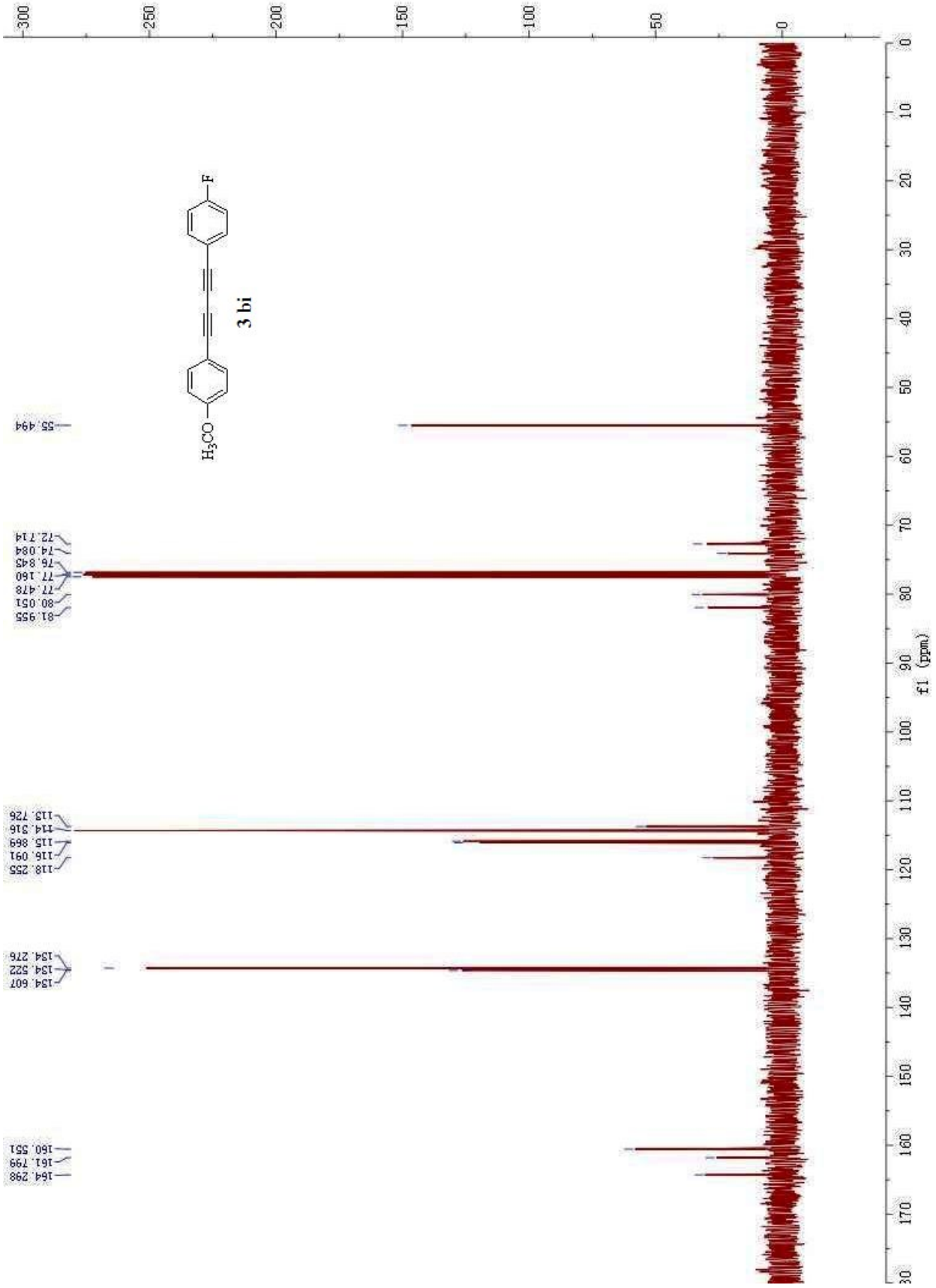
^{13}C NMR Spectrum of 1-(4-(4-chlorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bh)**



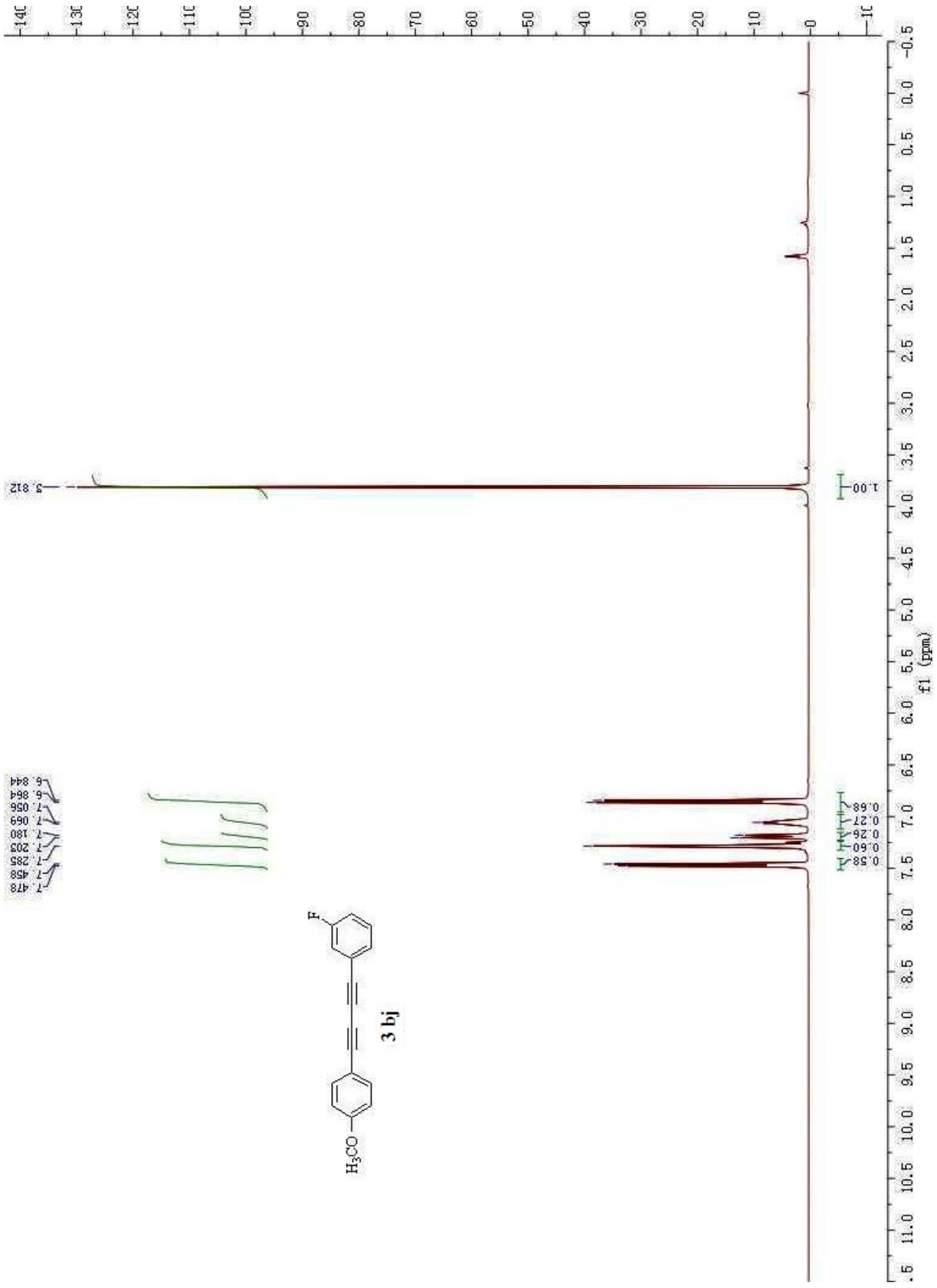
^{1}H NMR Spectrum of 1-(4-(4-fluorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bi)**



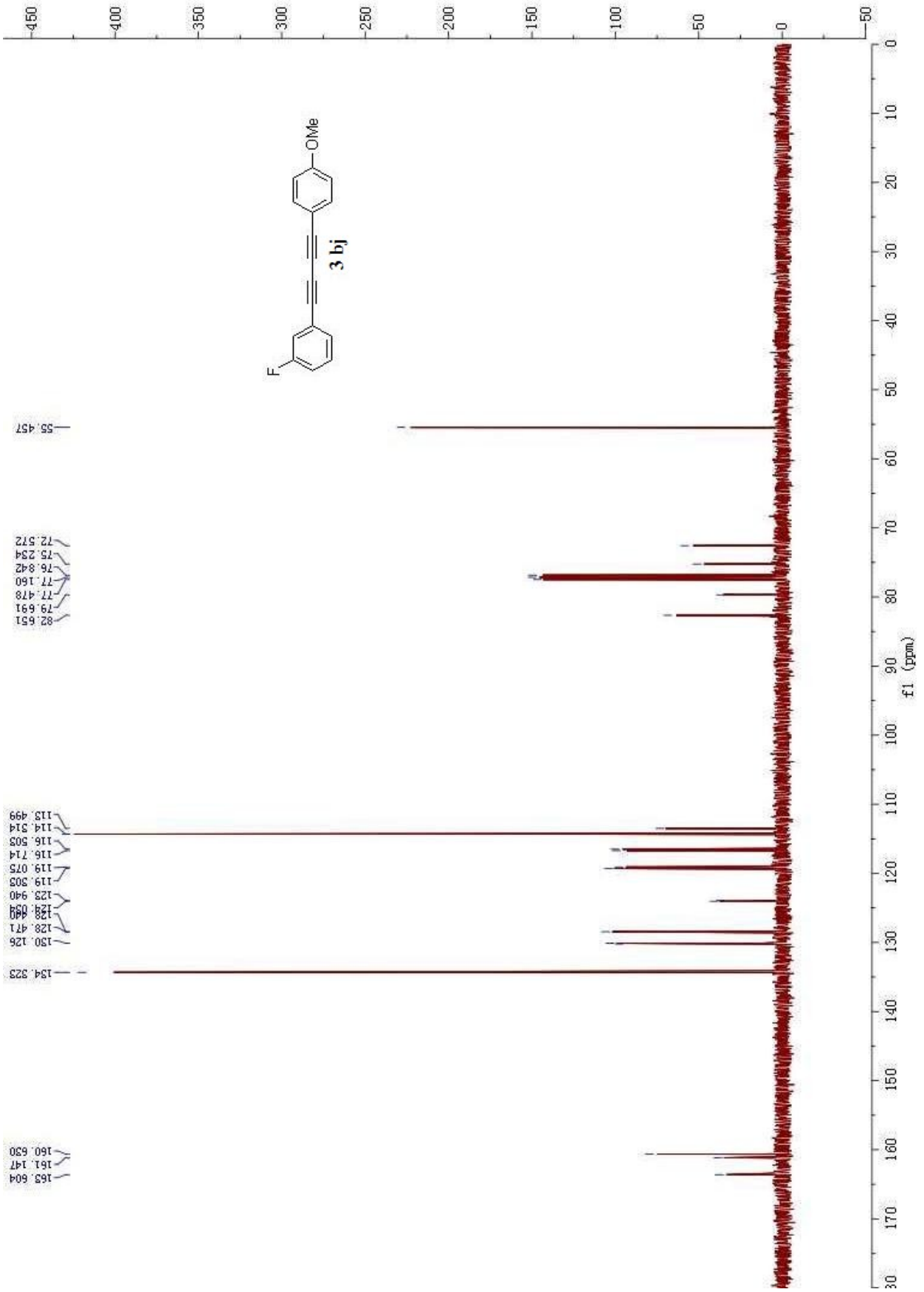
^{13}C NMR Spectrum of 1-(4-(4-fluorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bi)**



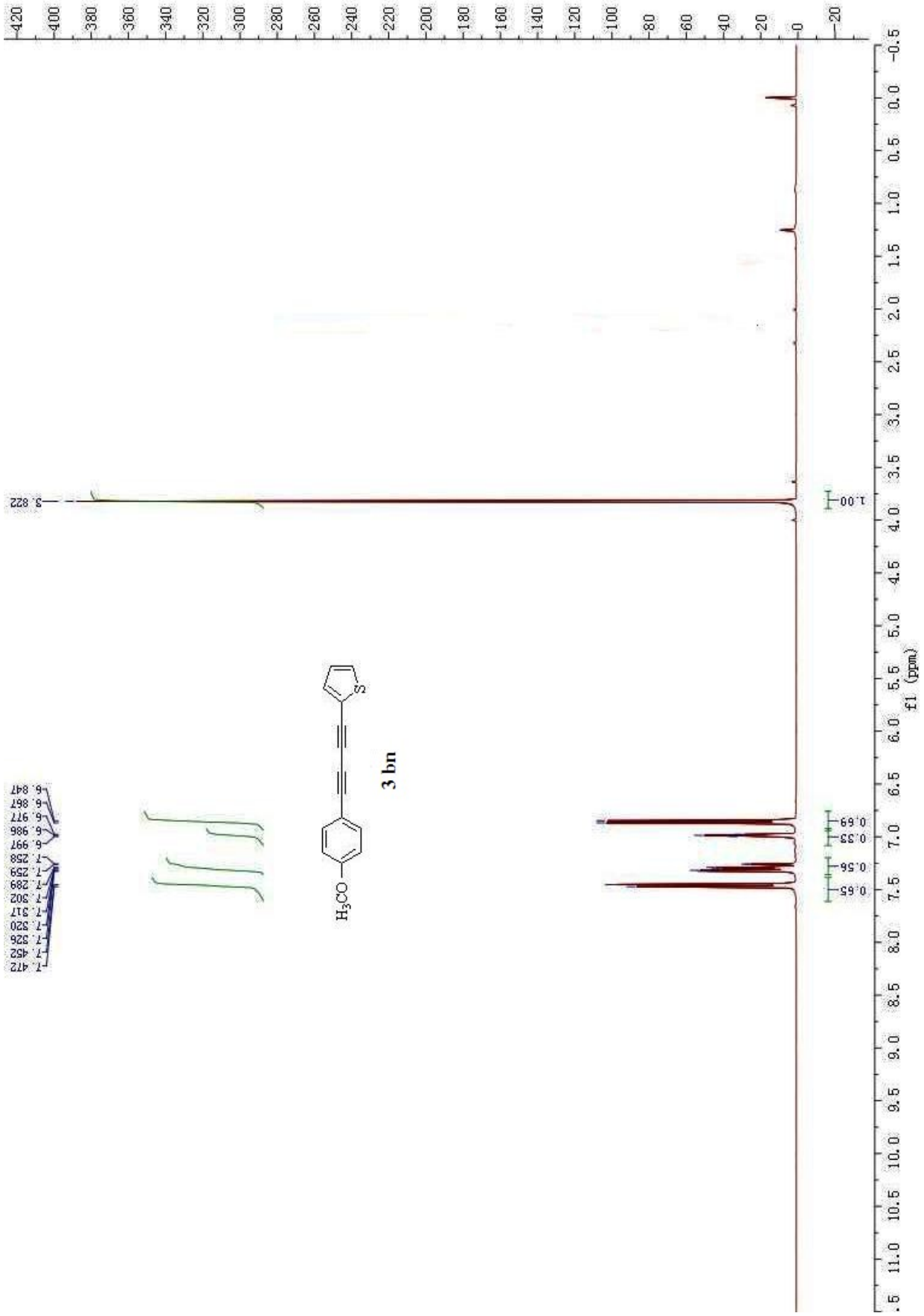
^{1}H NMR Spectrum of 1-(4-(3-fluorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bj)**



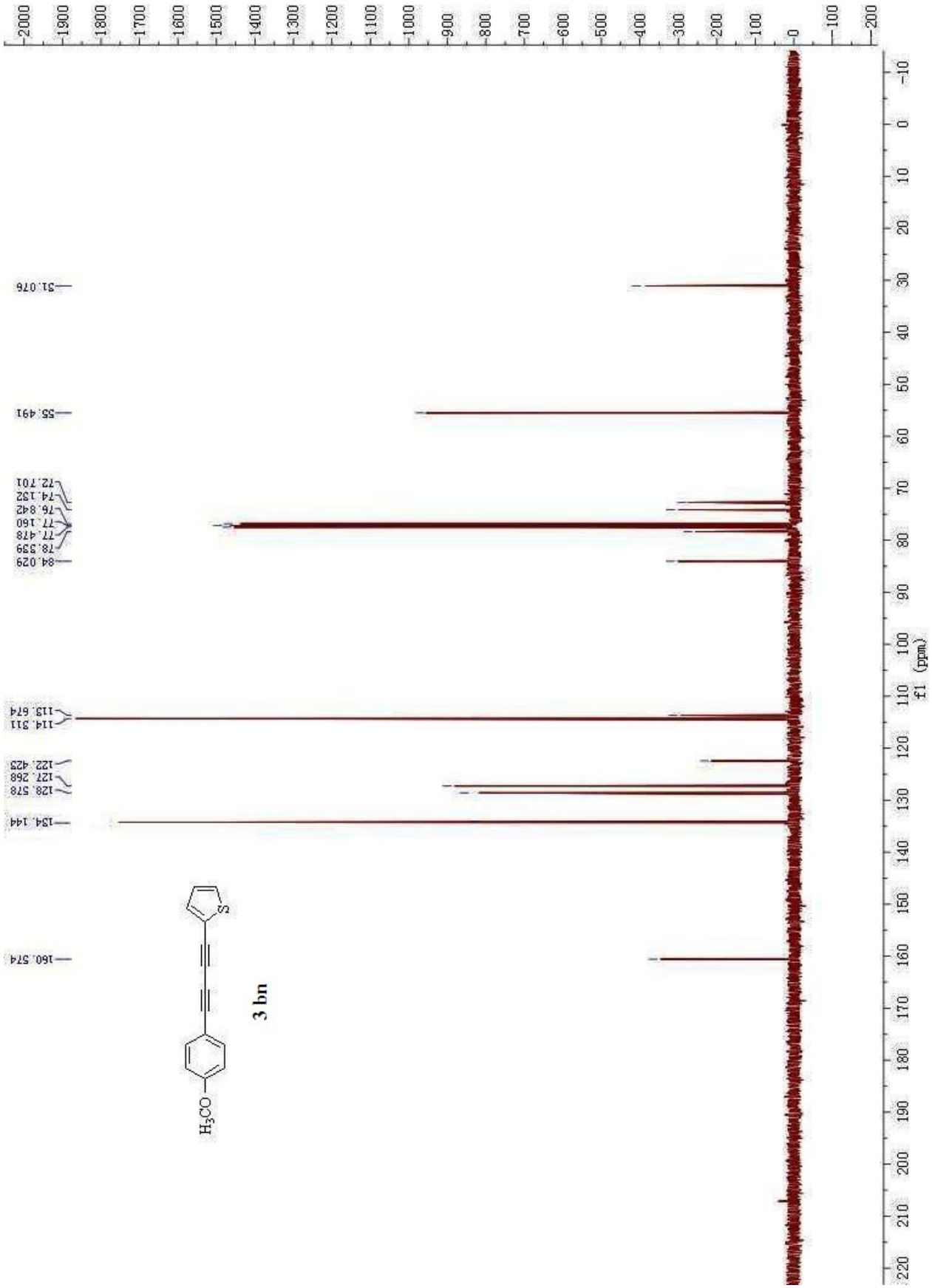
^{13}C NMR Spectrum of 1-(4-(3-fluorophenyl)buta-1,3-diynyl)-4-methoxybenzene **(3bj)**



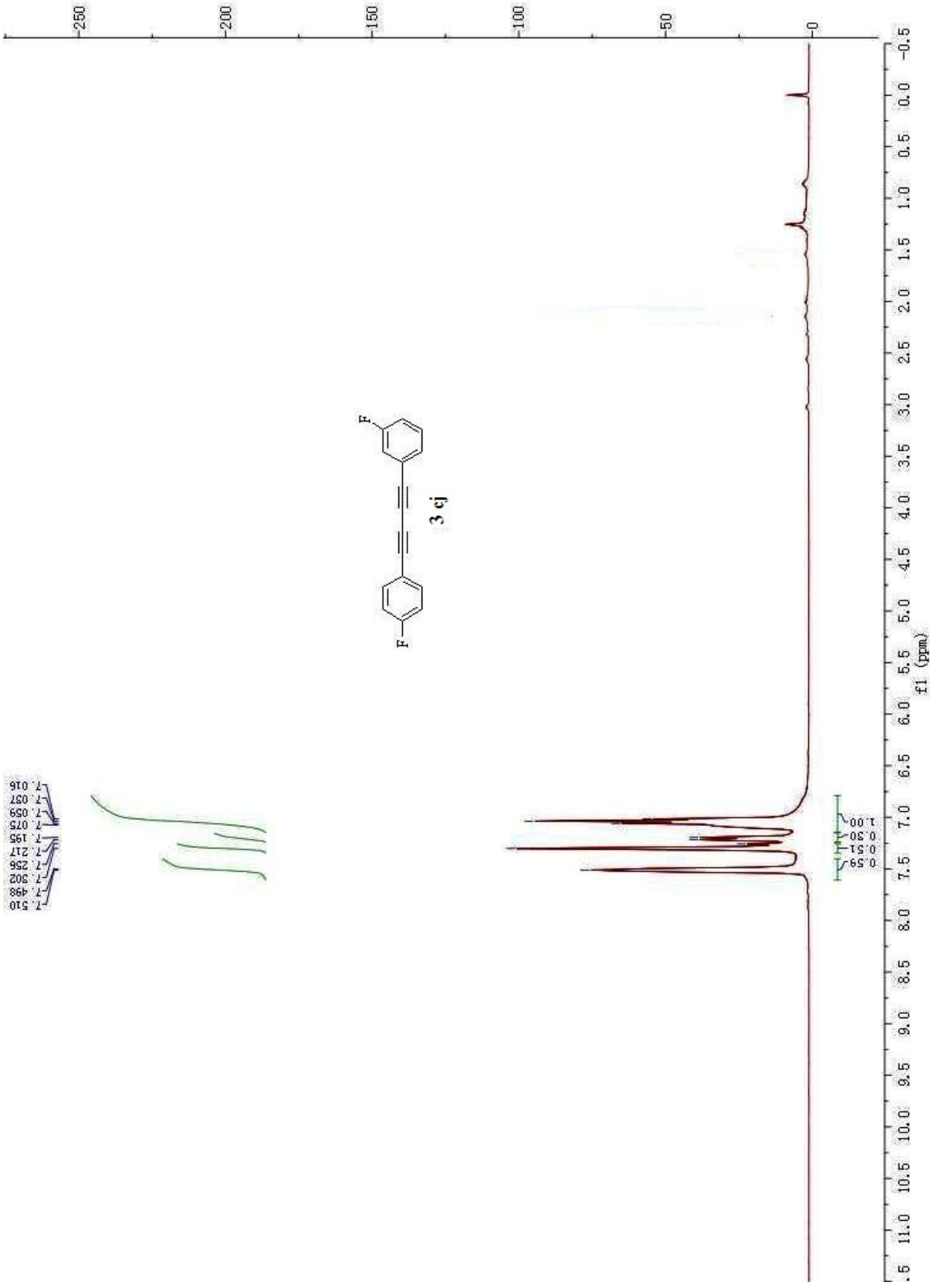
^{1}H NMR Spectrum of 2-(4-(4-methoxyphenyl)buta-1,3-diynyl)thiophene **(3bn)**



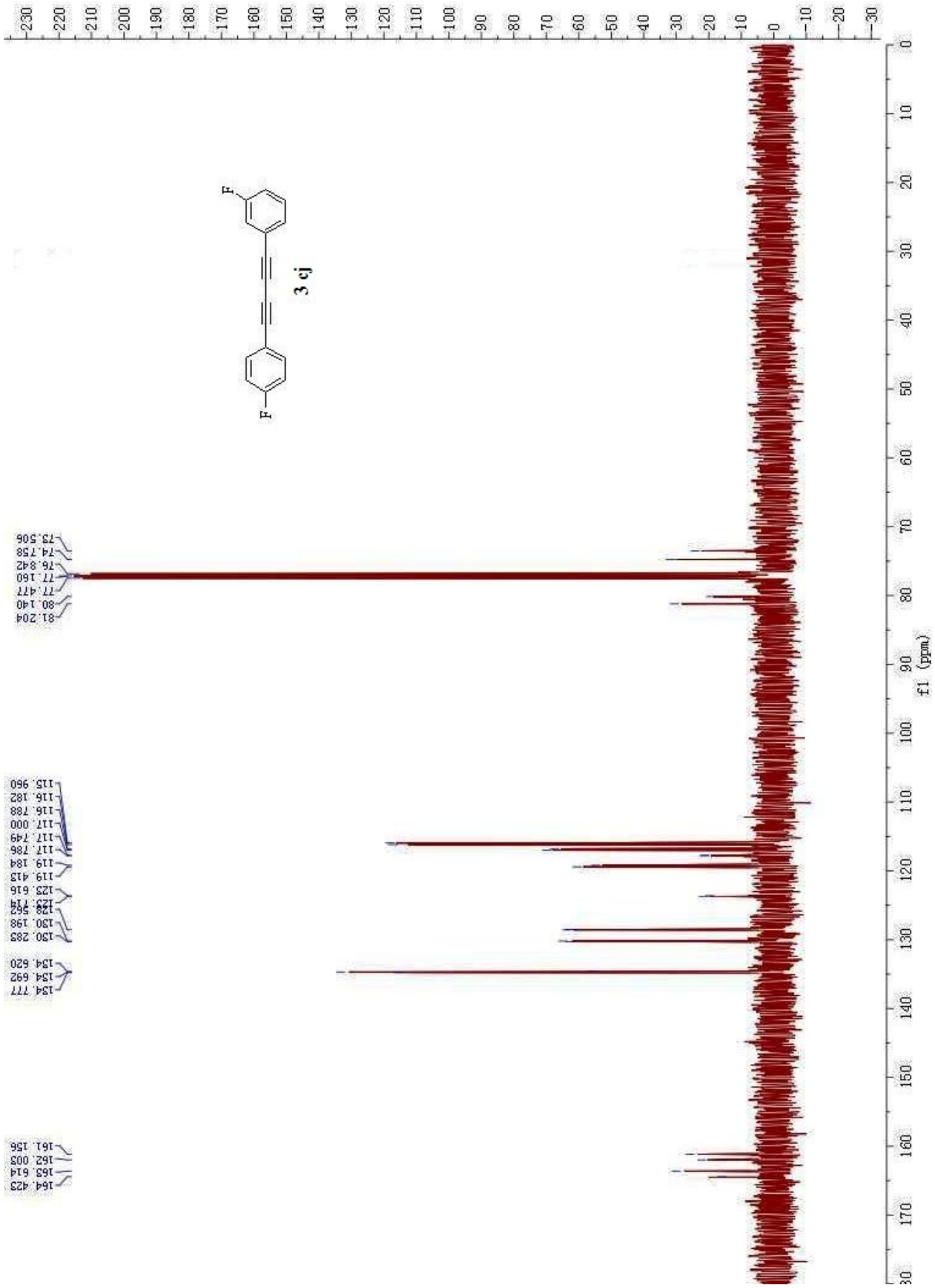
^{13}C NMR Spectrum of 2-(4-(4-methoxyphenyl)buta-1,3-diynyl)thiophene **(3bn)**



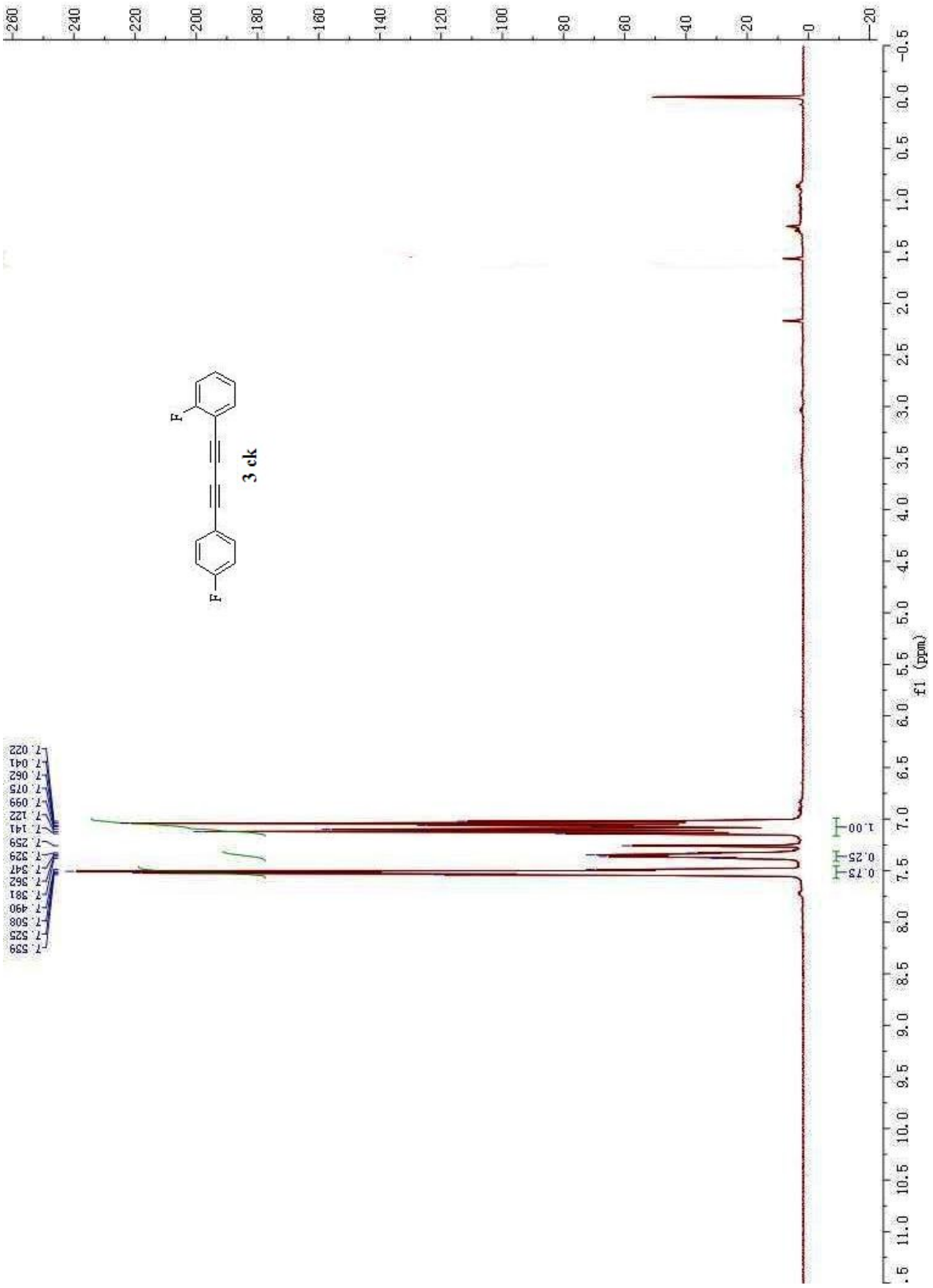
^{1}H NMR Spectrum of 4-(3-fluorophenyl)-1-(4-fluorophenyl)buta-1,3-diyne **(3cj)**



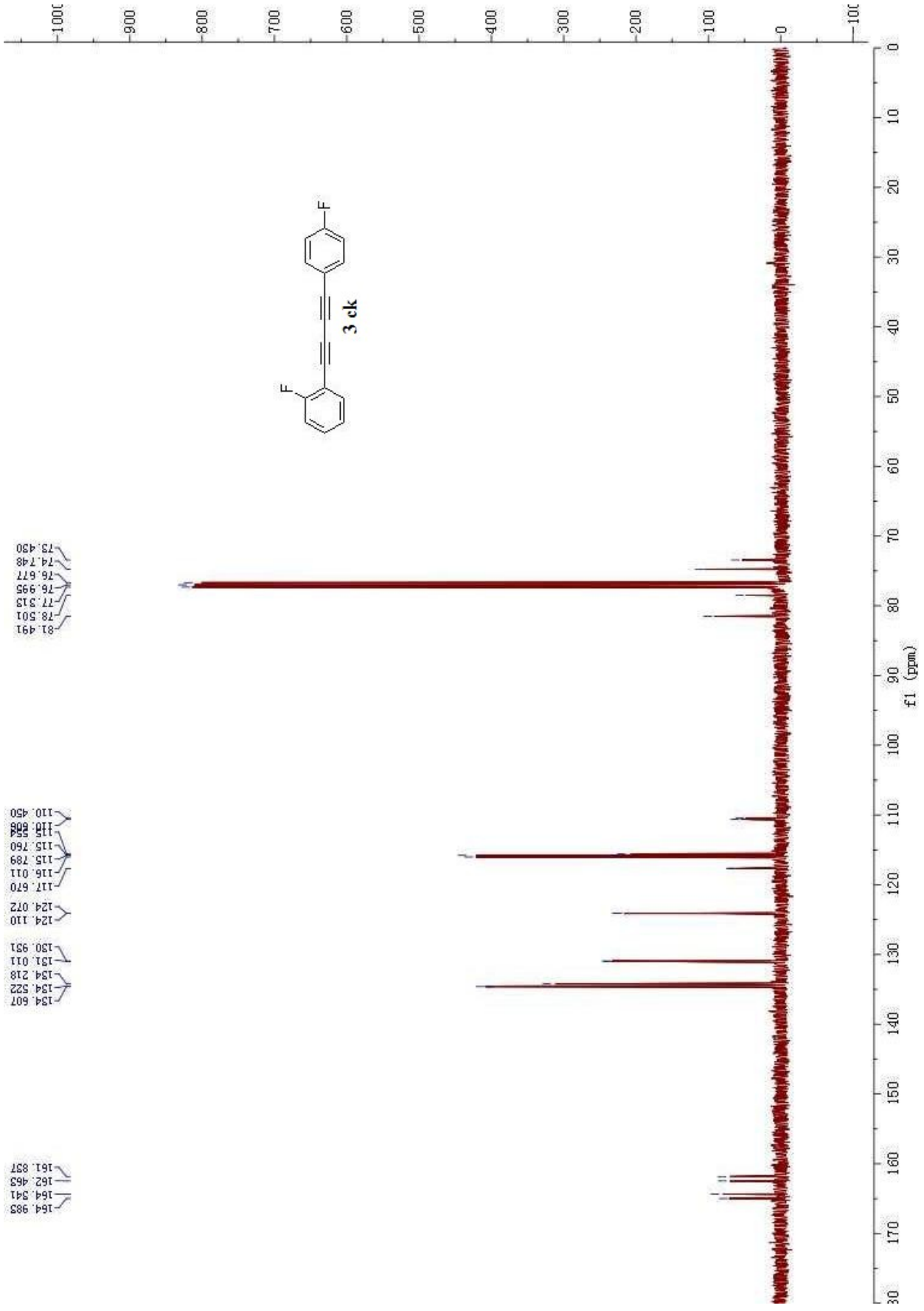
^{13}C NMR Spectrum of 4-(3-fluorophenyl)-1-(4-fluorophenyl)buta-1,3-diyne **(3cj)**



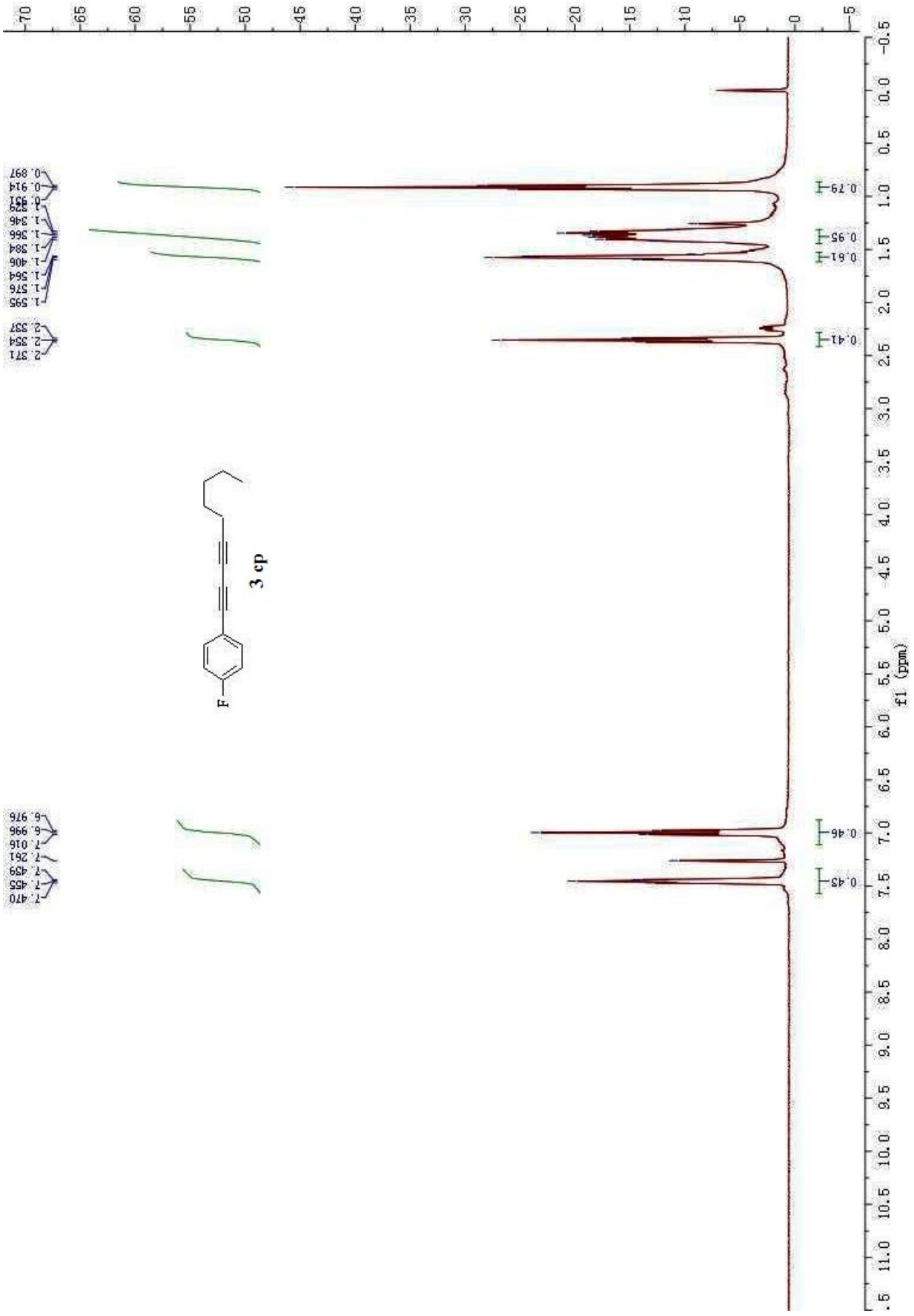
^{1}H NMR Spectrum of 4-(2-fluorophenyl)-1-(4-fluorophenyl)buta-1,3-diyne **(3ck)**



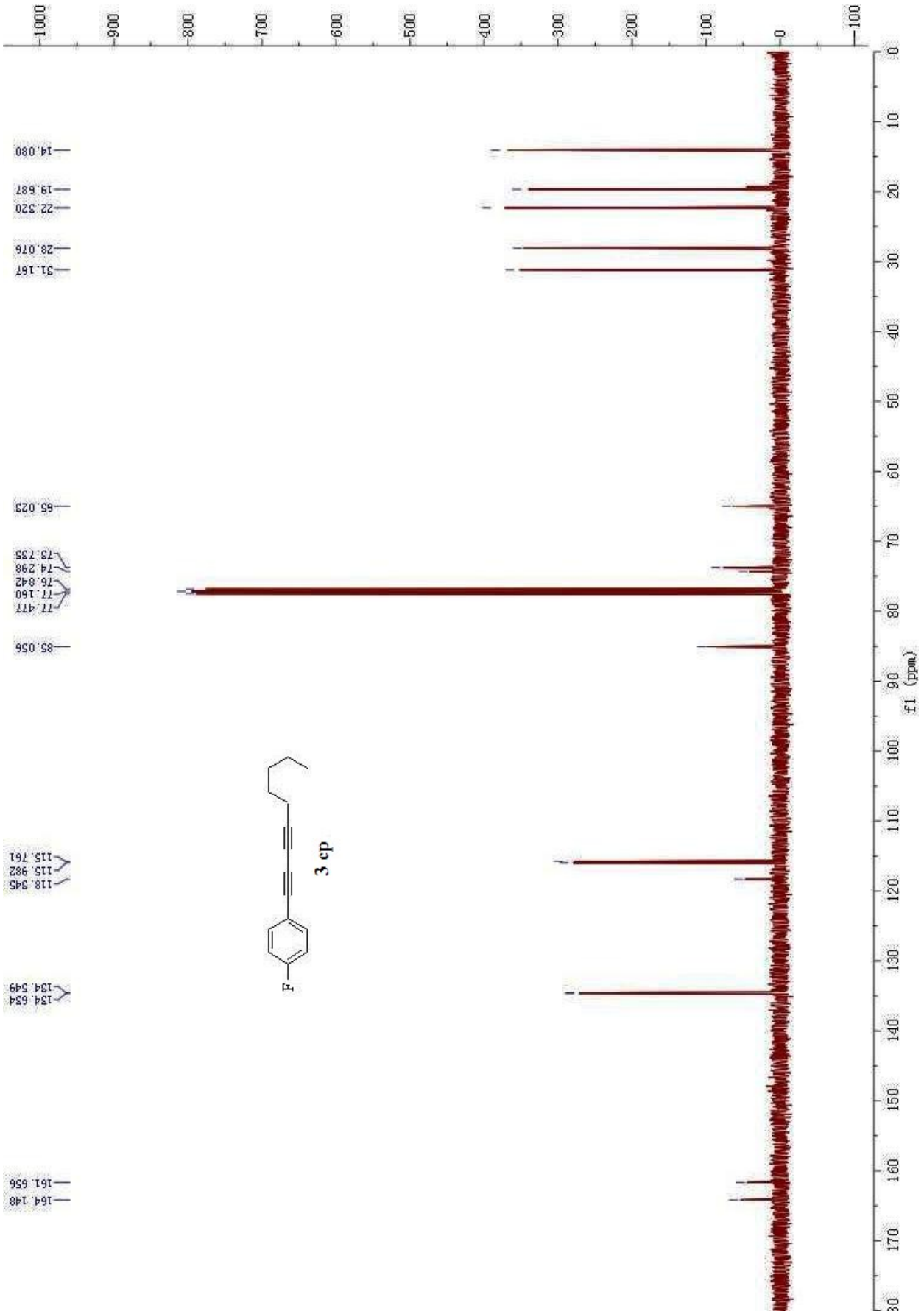
^{13}C NMR Spectrum of 4-(2-fluorophenyl)-1-(4-fluorophenyl)buta-1,3-diyne **(3ck)**



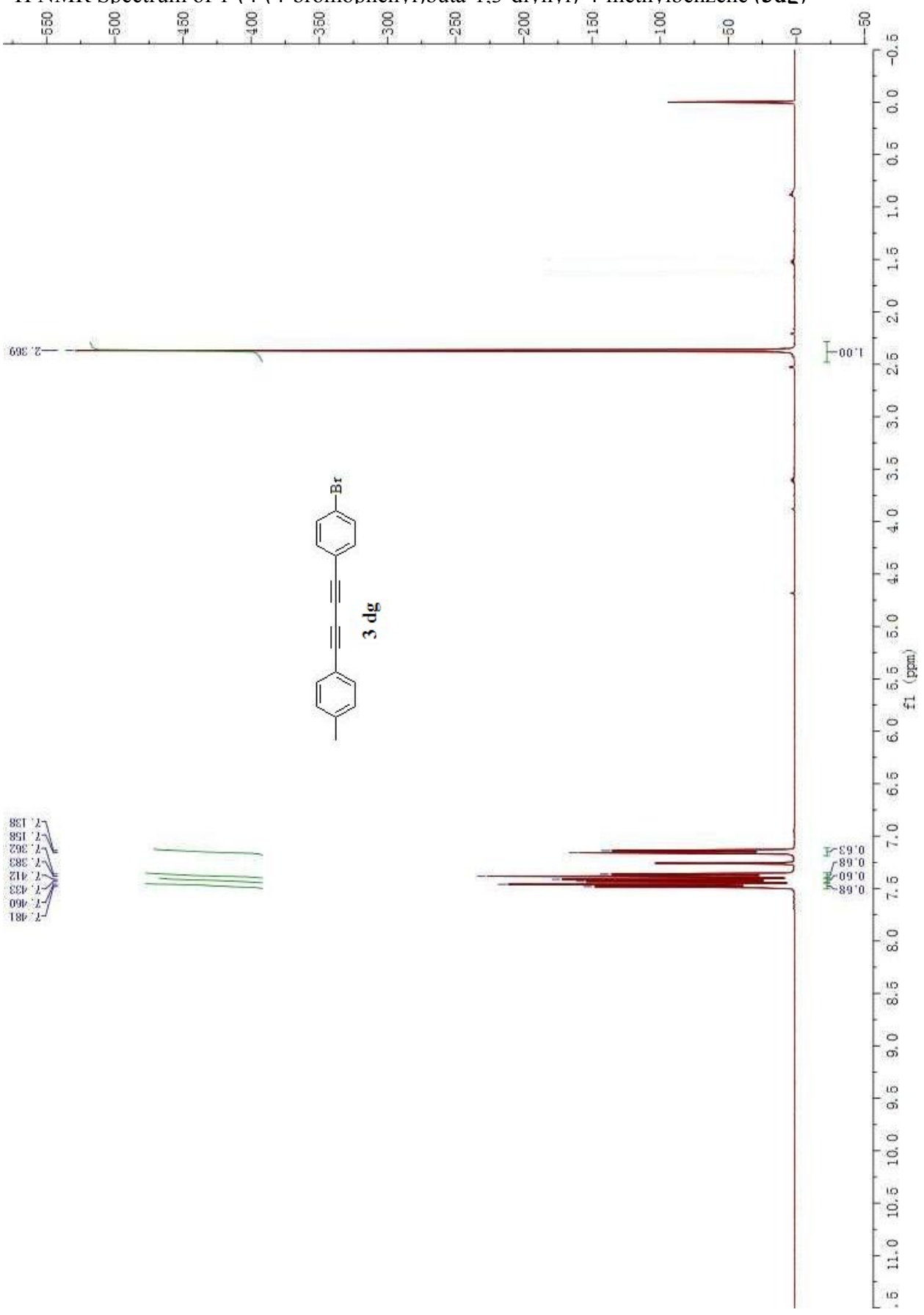
^{1}H NMR Spectrum of 1-fluoro-4-(nona-1,3-diynyl)benzene **(3cp)**



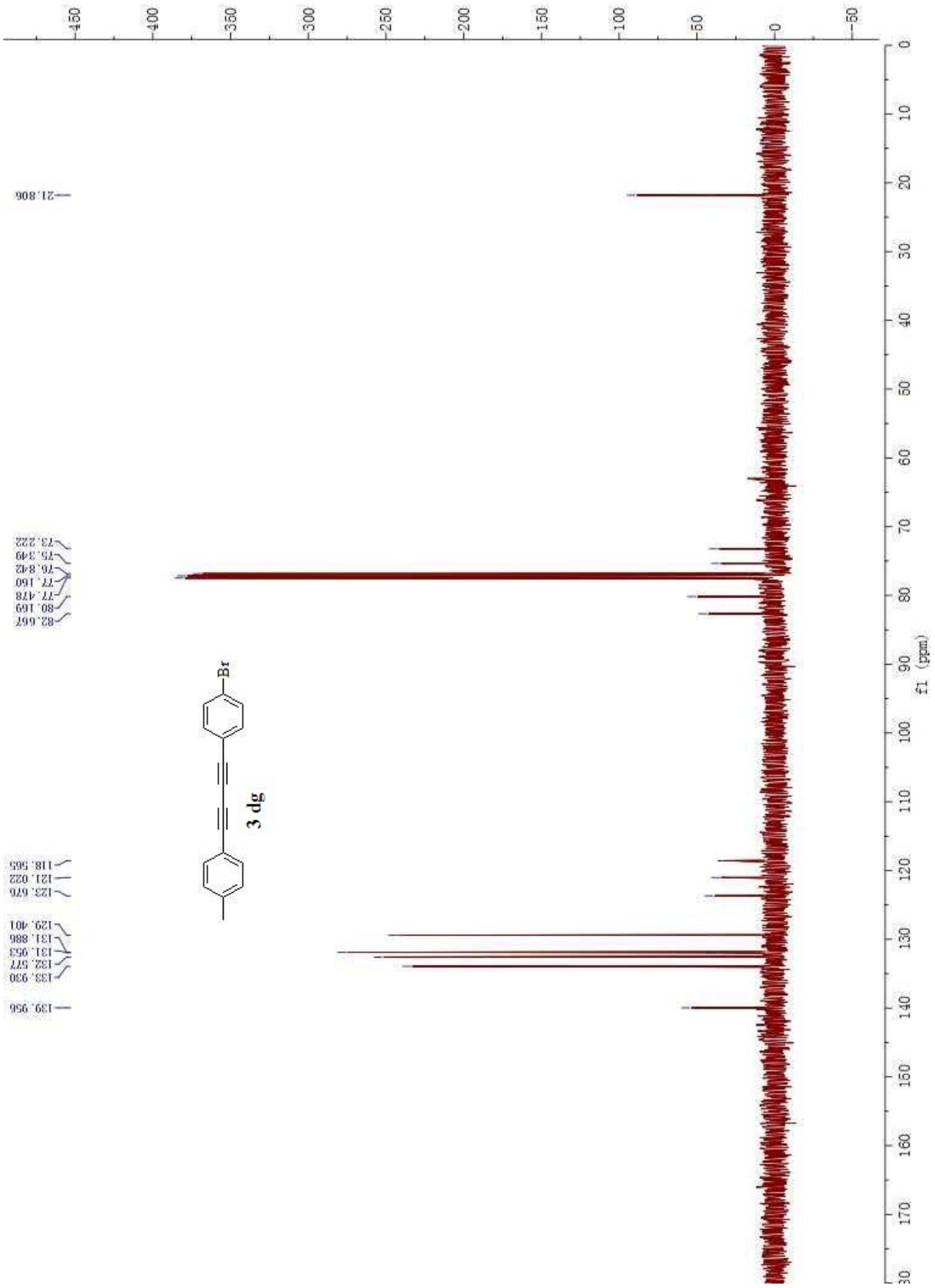
^{13}C NMR Spectrum of 1-fluoro-4-(nona-1,3-diynyl)benzene **(3cp)**



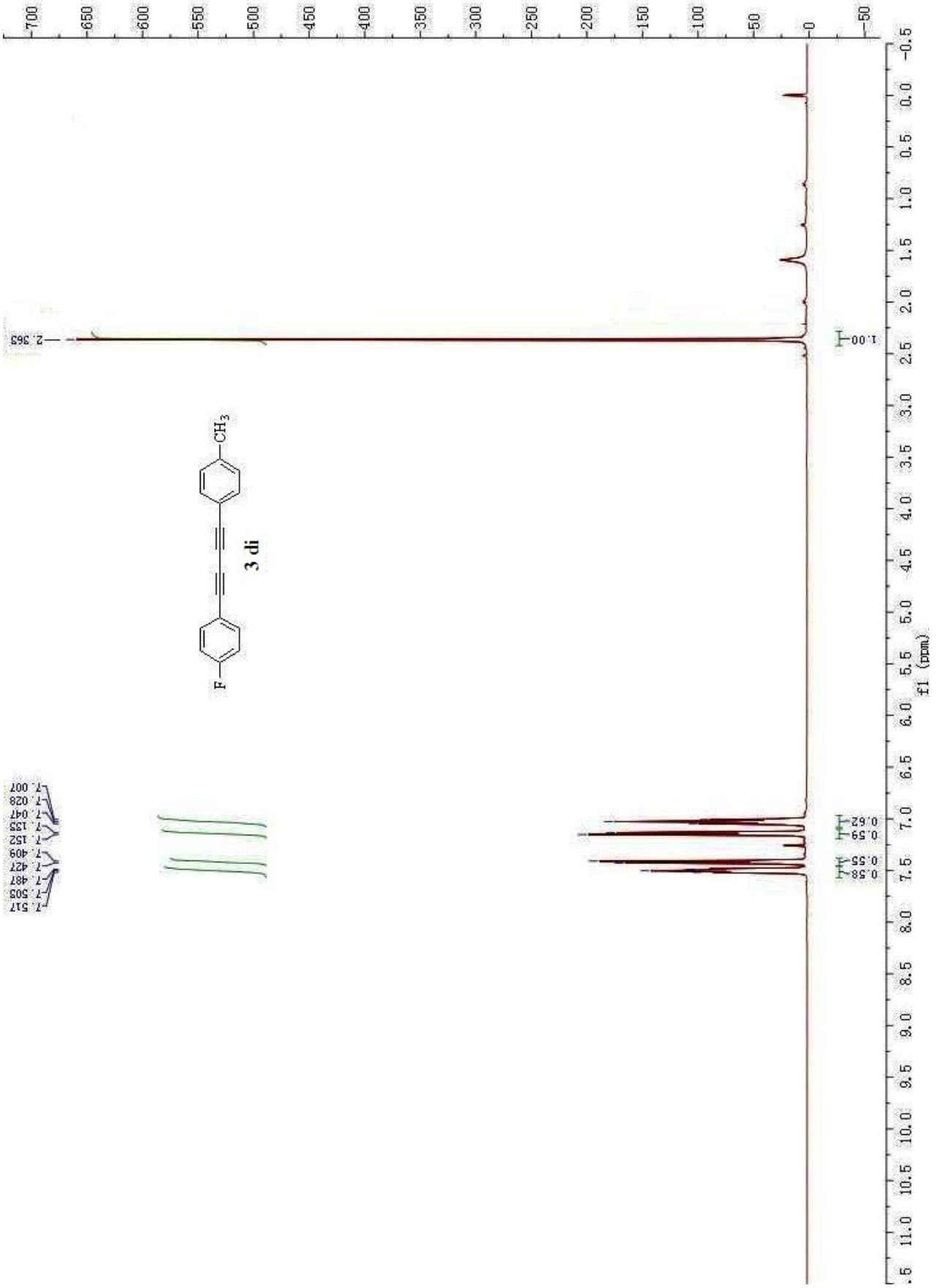
^{1}H NMR Spectrum of 1-(4-(4-bromophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dg**)



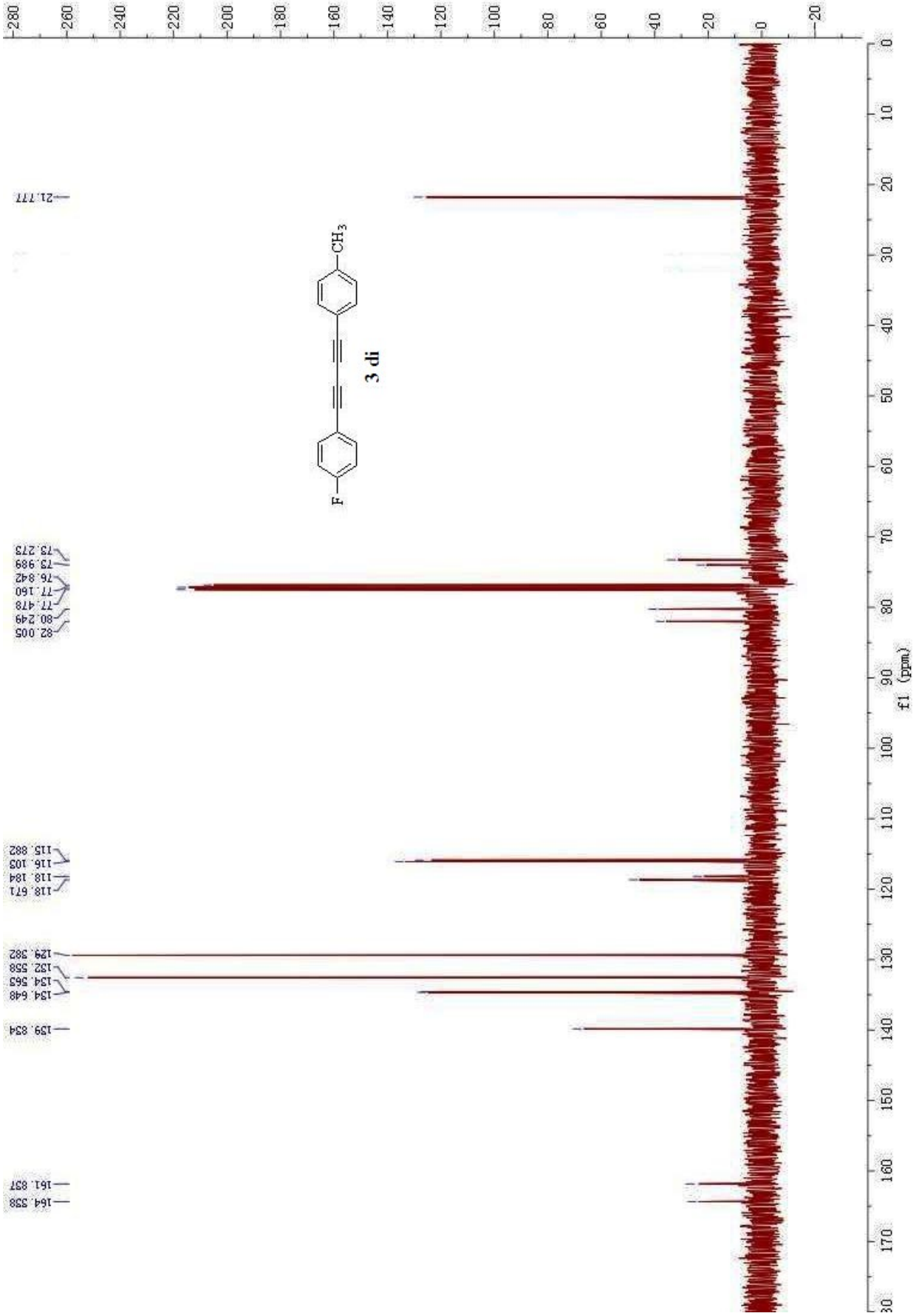
^{13}C NMR Spectrum of 1-(4-(4-bromophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dg**)



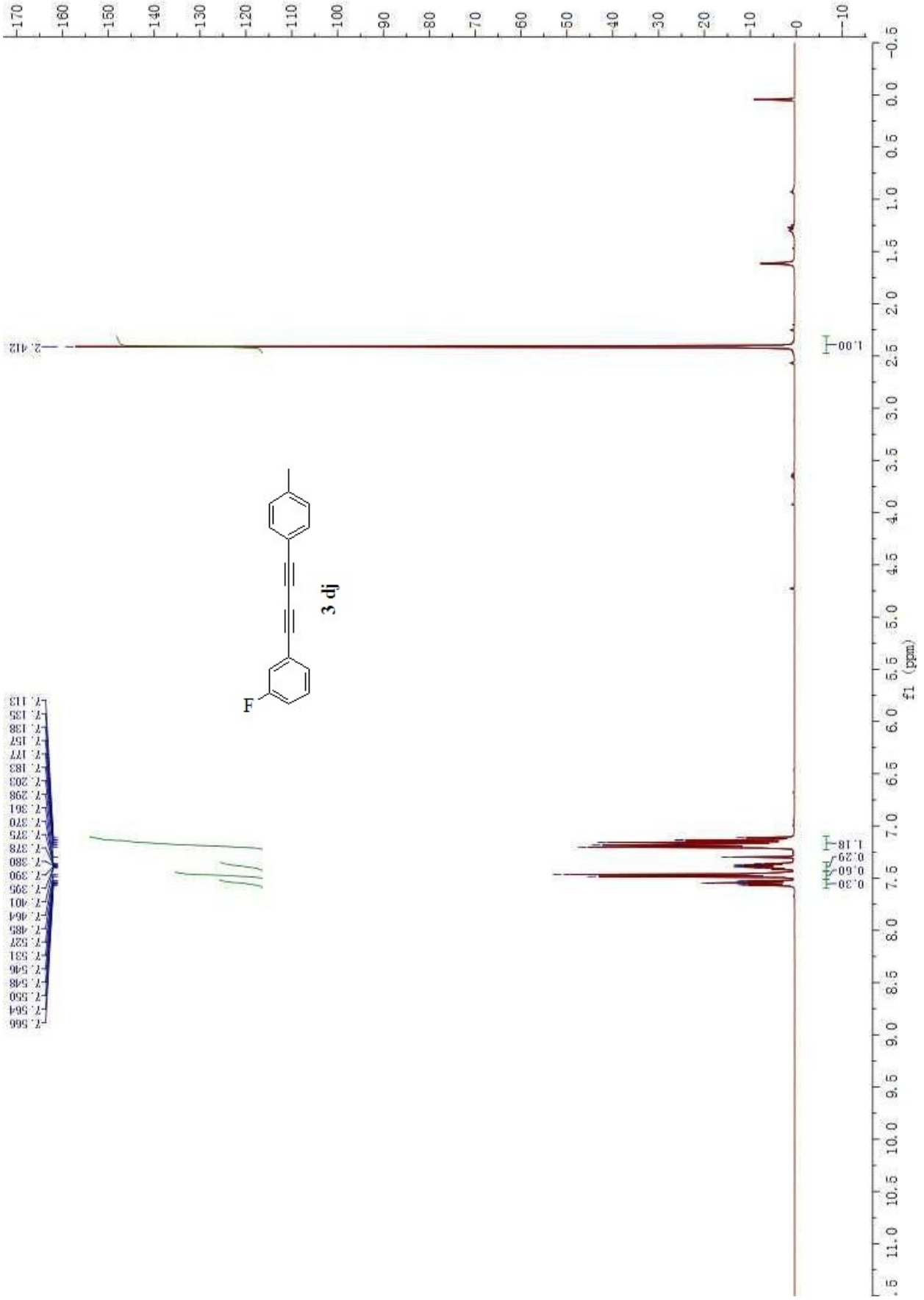
^{1}H NMR Spectrum of 1-(4-(4-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene **(3di)**



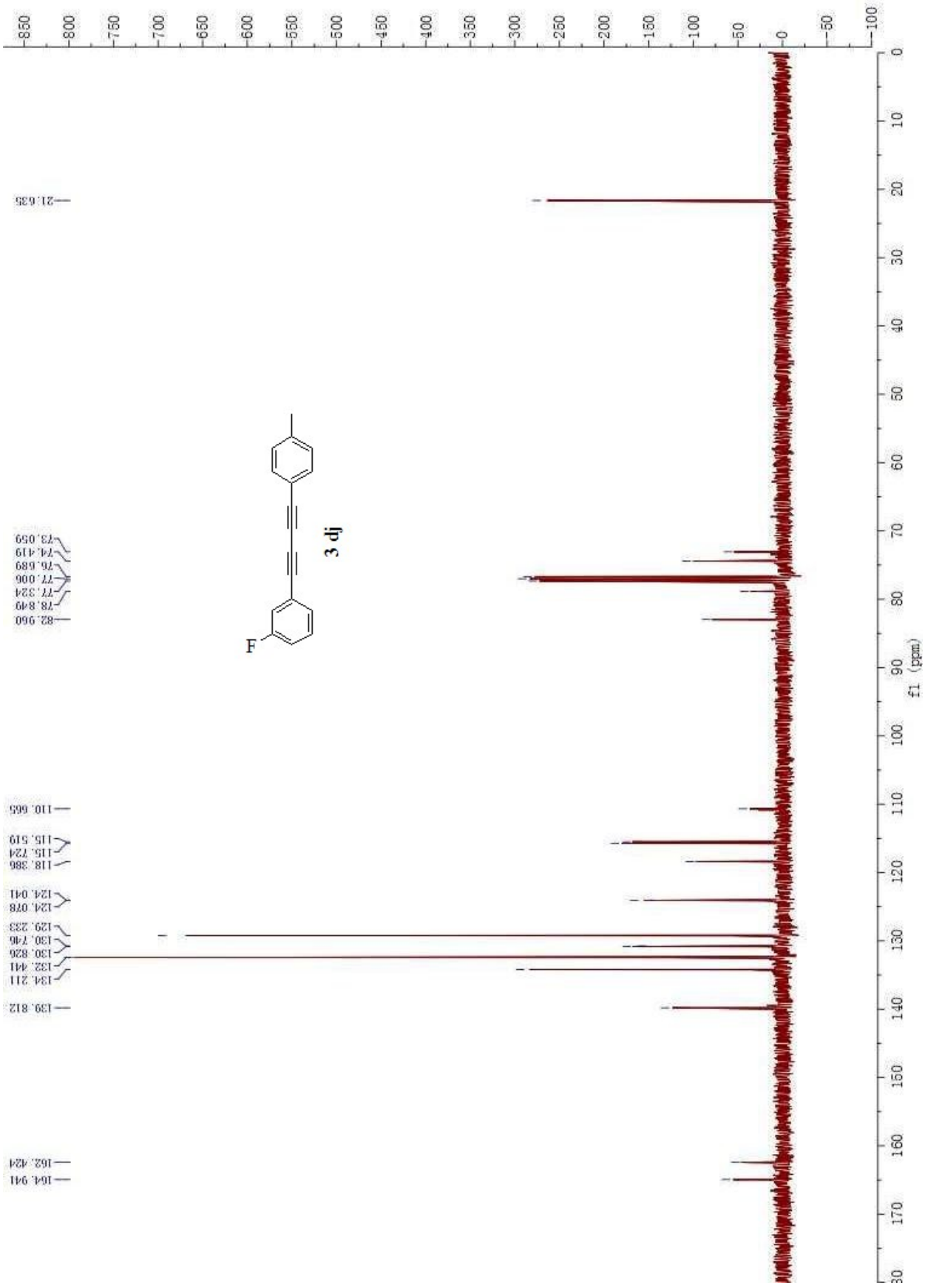
^{13}C NMR Spectrum of 1-(4-(4-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene **(3di)**



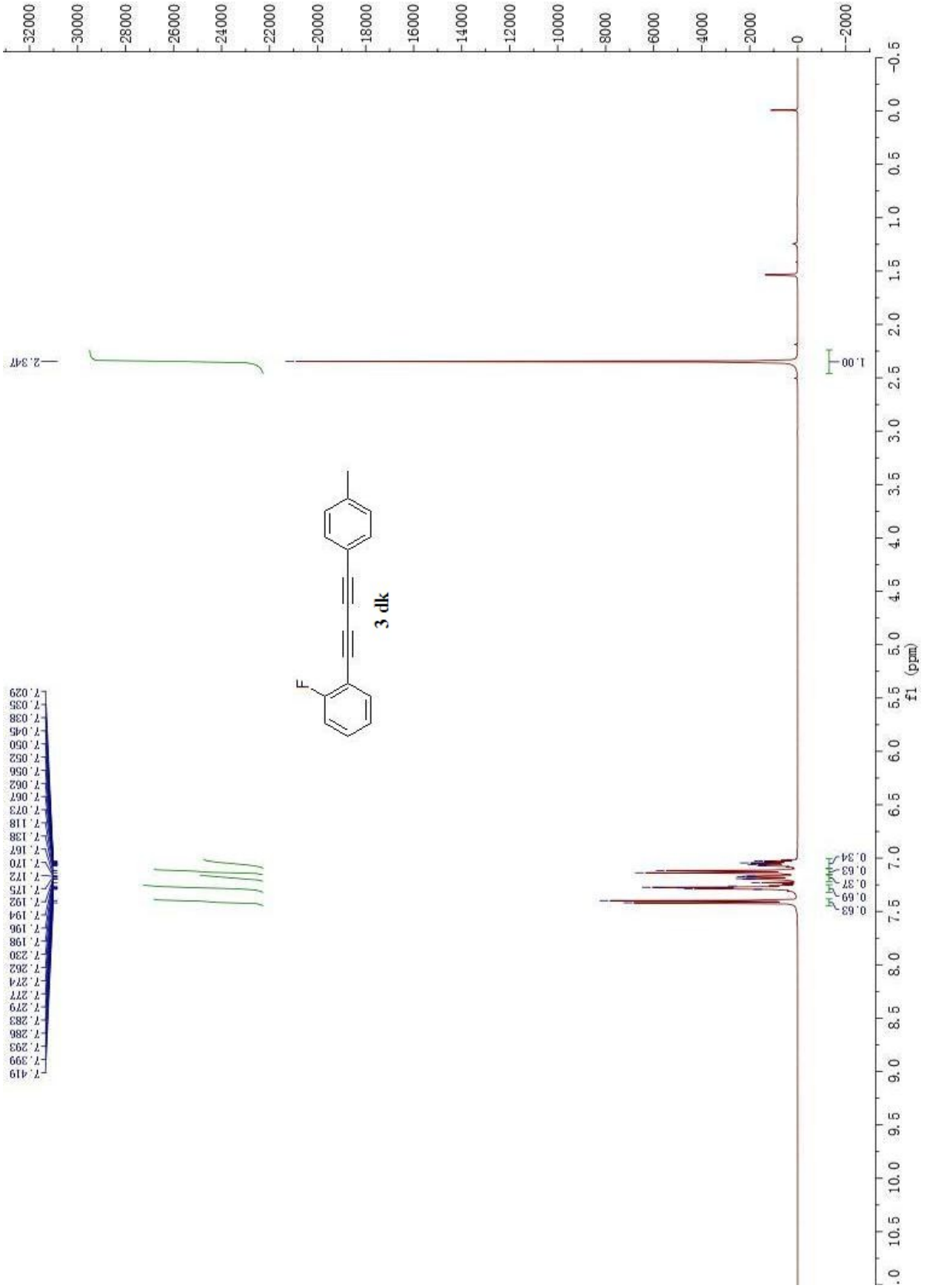
[1] H NMR Spectrum of 1-(4-(3-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dj**)



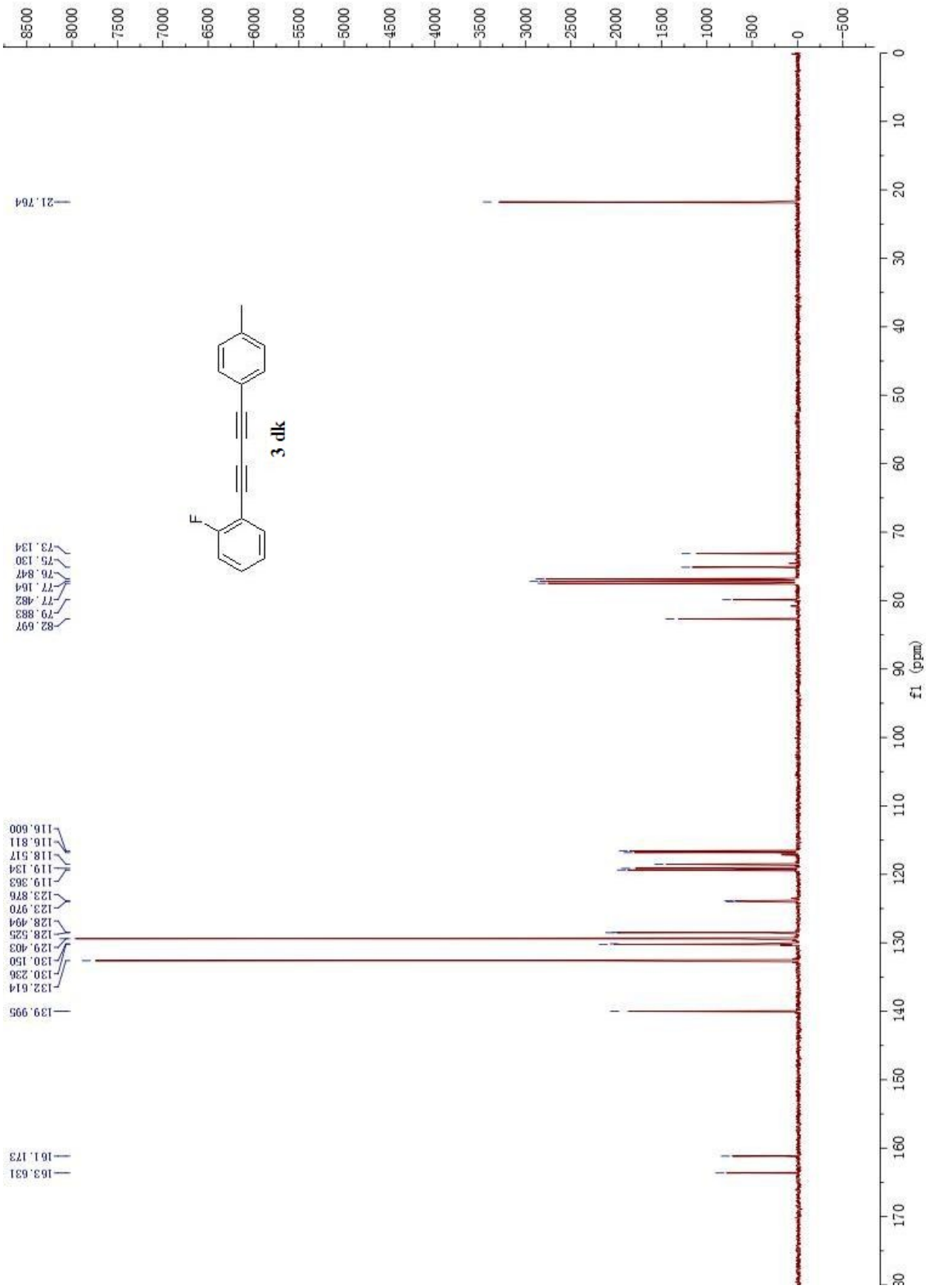
^{13}C NMR Spectrum of 1-(4-(3-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dj**)



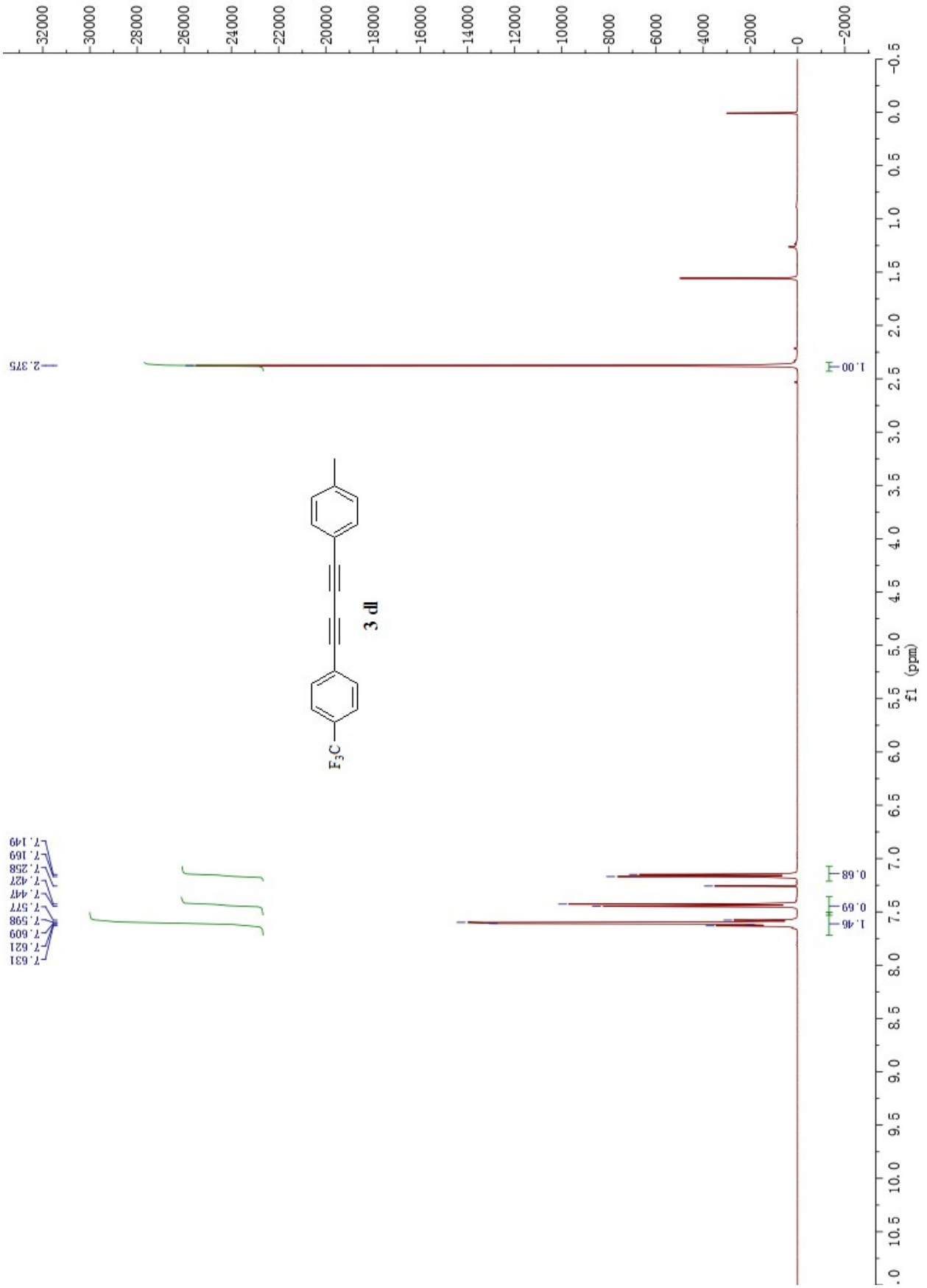
[1] H NMR Spectrum of 1-(4-(2-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dk**)



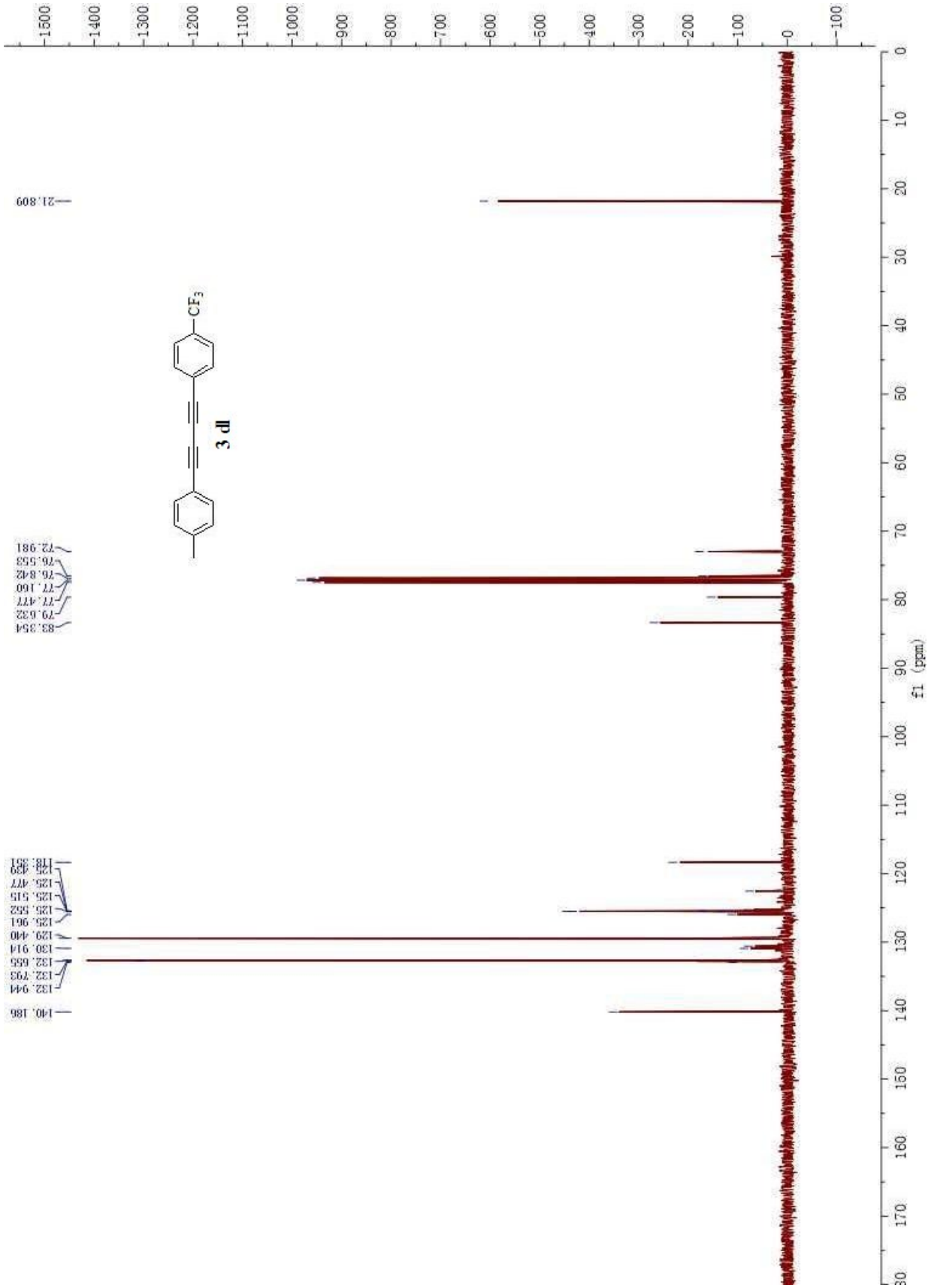
^{13}C NMR Spectrum of 1-(4-(2-fluorophenyl)buta-1,3-diynyl)-4-methylbenzene (**3dk**)



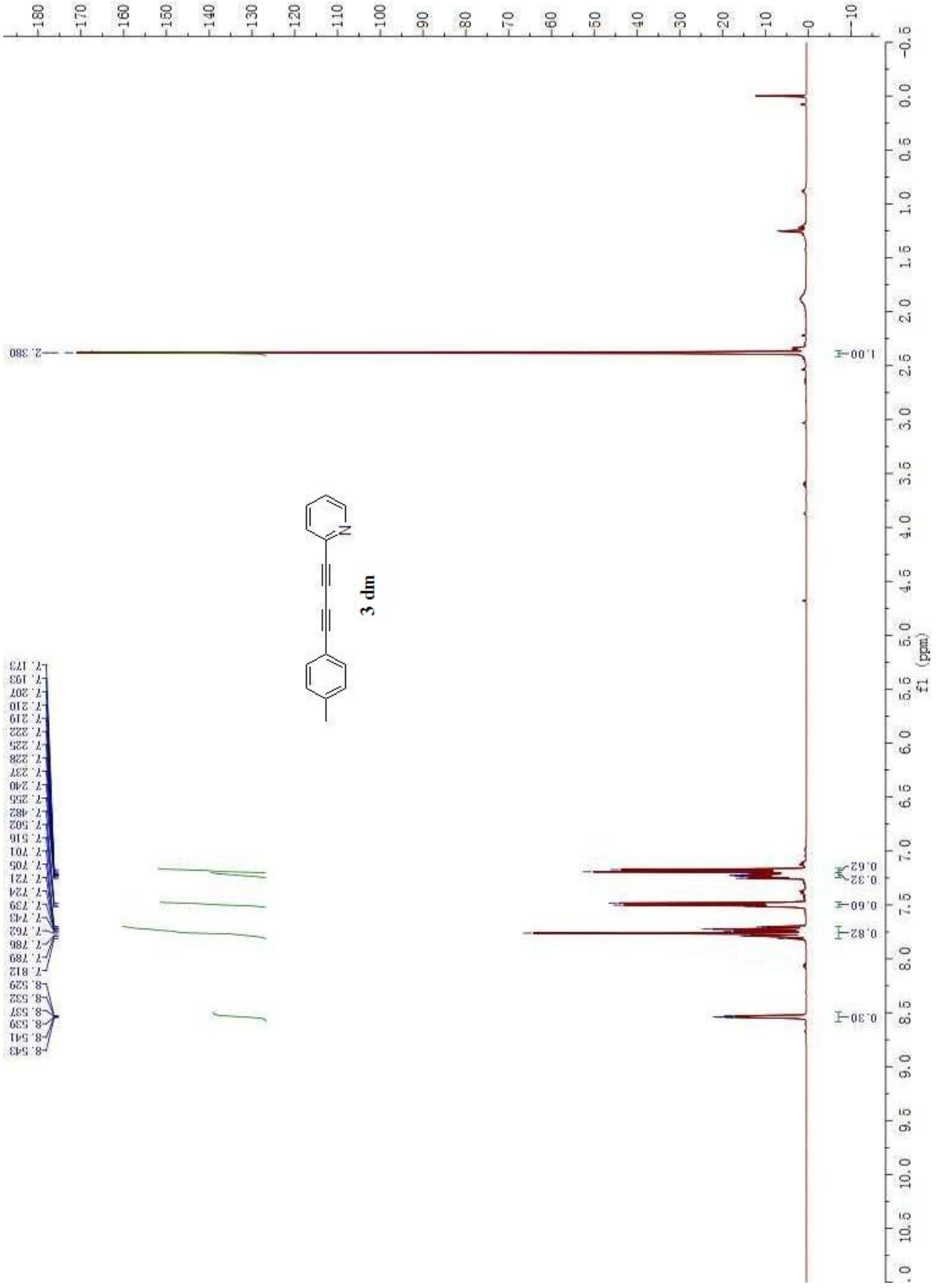
^{1}H NMR Spectrum of 1-(4-(4-trifluoromethylphenyl)buta-1,3-diynyl)-4-methylbenzene (**3dl**)



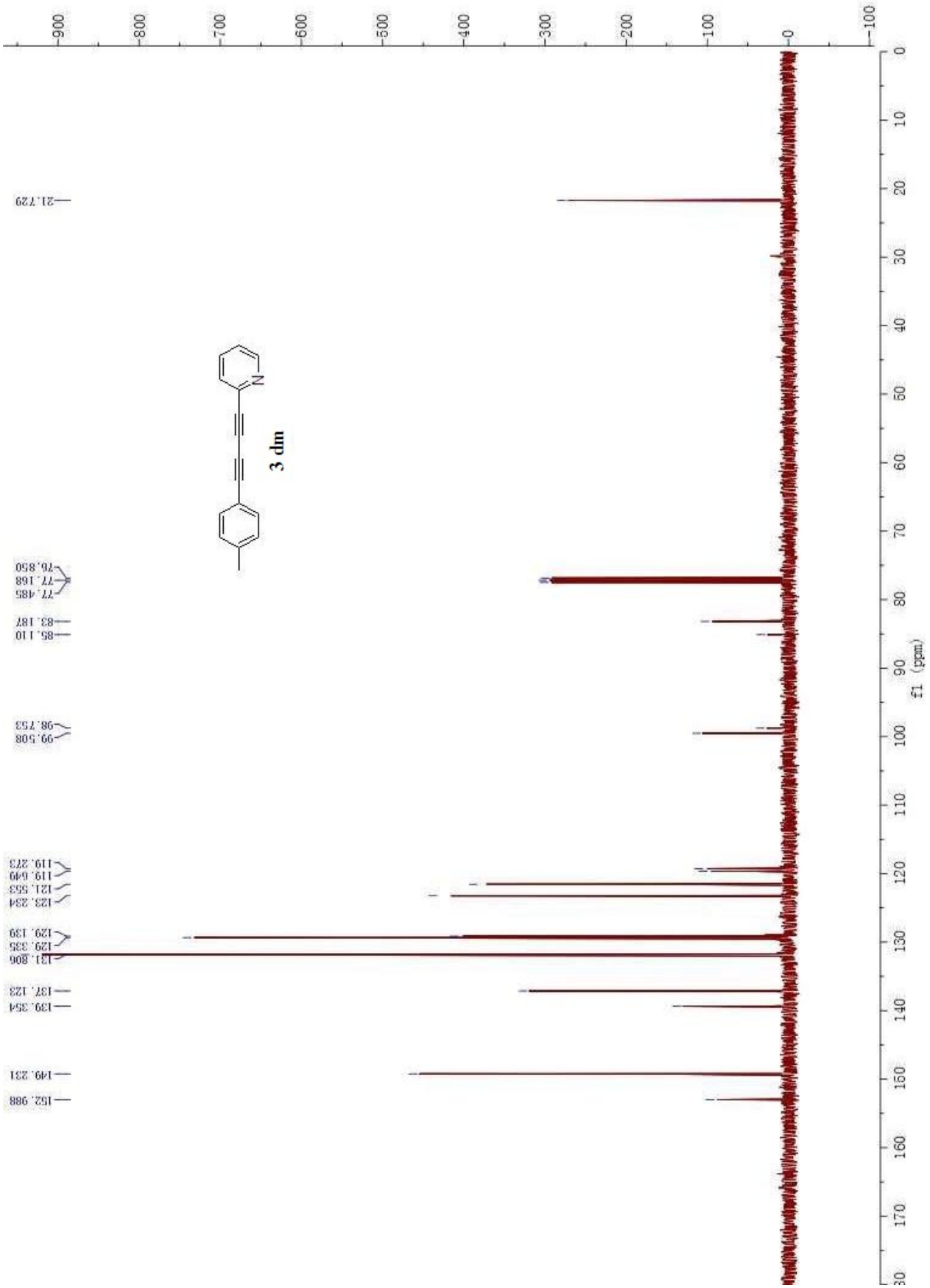
^{13}C NMR Spectrum of 1-(4-(4-trifluoromethylphenyl)buta-1,3-diynyl)-4-methylbenzene (**3dl**)



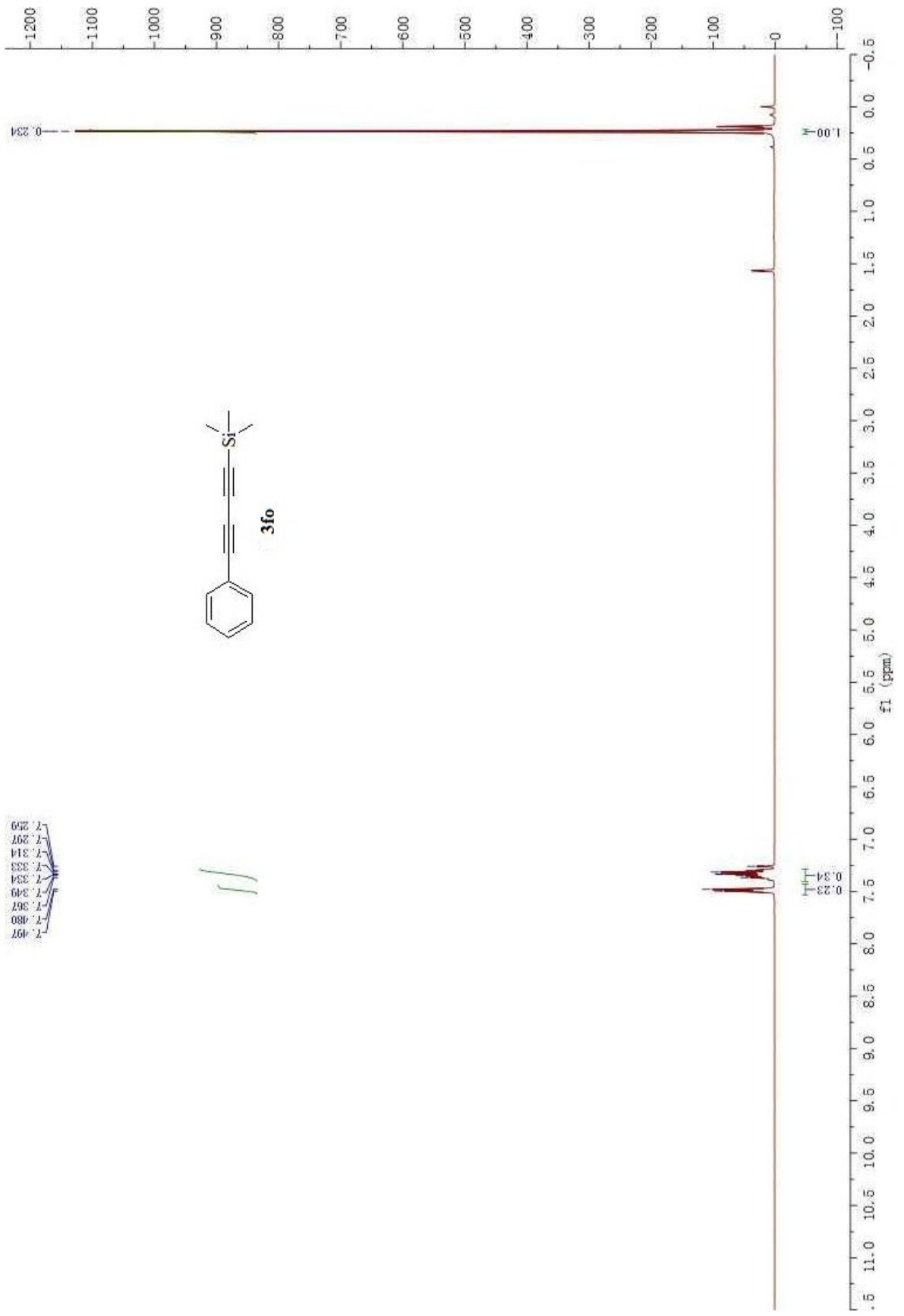
^{1}H NMR Spectrum of 2-(4-(4-methylphenyl)buta-1,3-diynyl)pyridine(**3dm**)



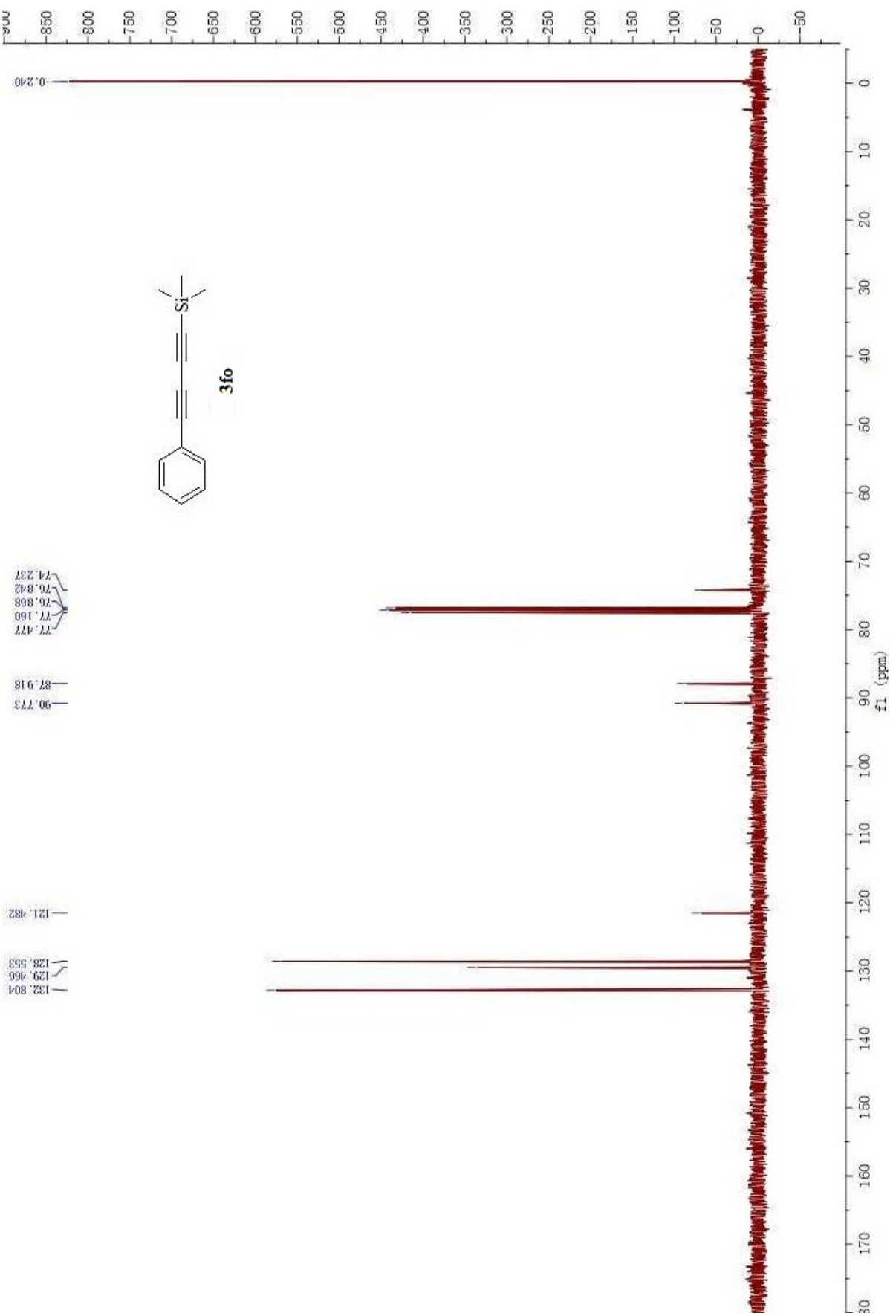
[1] ^{3}C NMR Spectrum of 2-(4-(4-methylphenyl)buta-1,3-diynyl)pyridine(**3dm**)



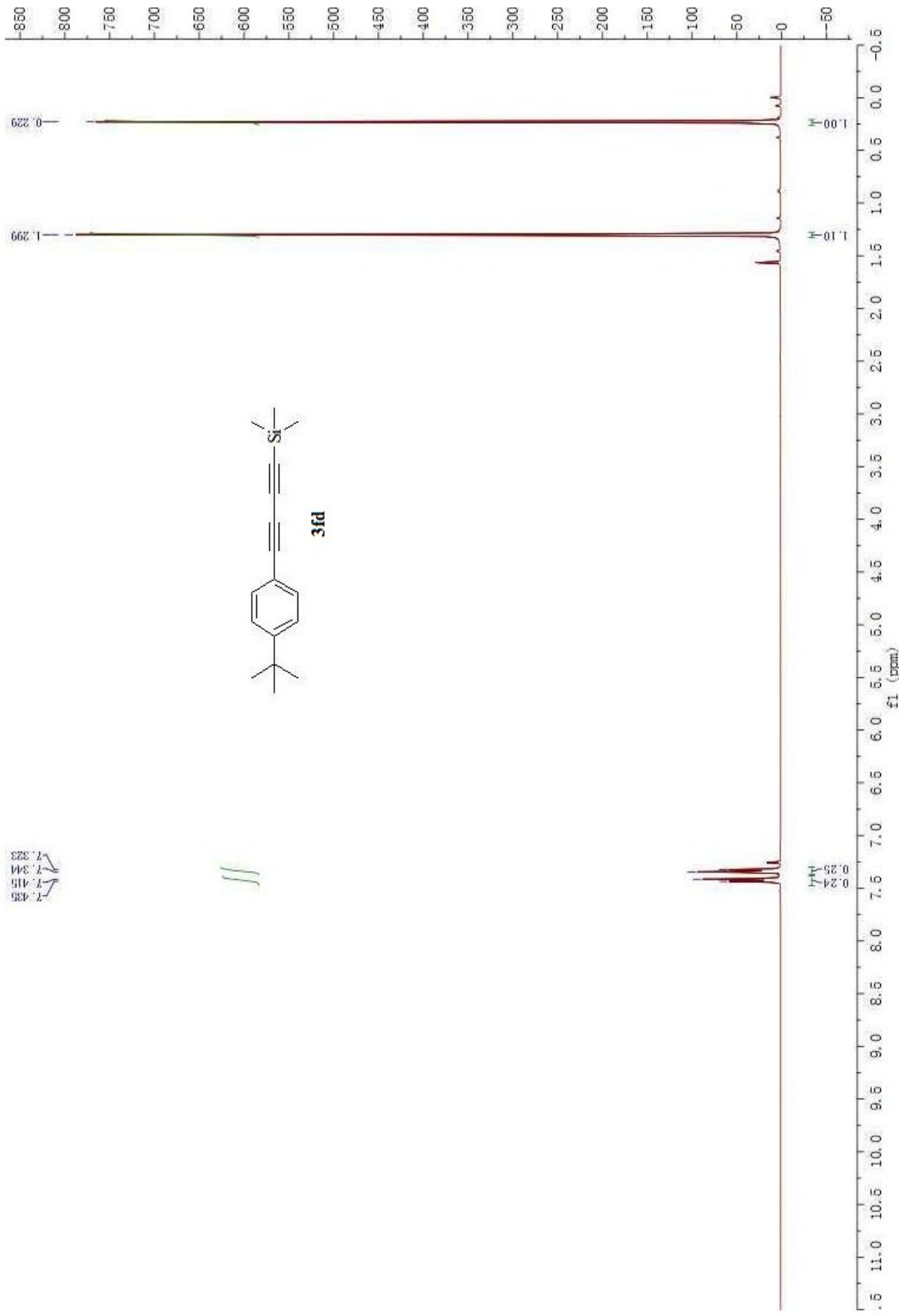
[1] H NMR Spectrum of trimethyl-(4-phenyl-buta-1,3-diynyl)-silane(**3fo**)



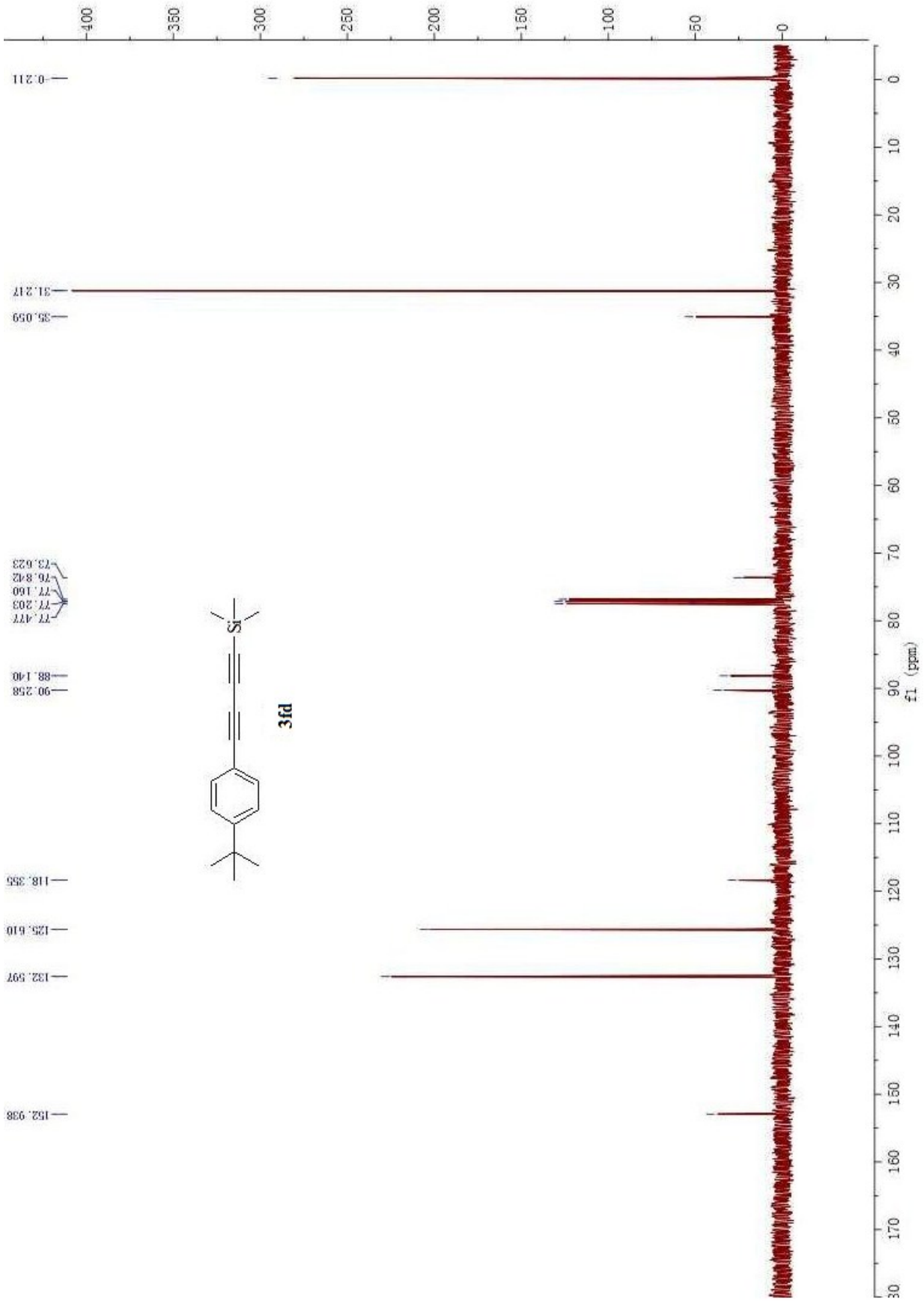
^{1} ^{3}C NMR Spectrum of trimethyl-(4-phenyl-buta-1,3-diynyl)-silane(**3fo**)



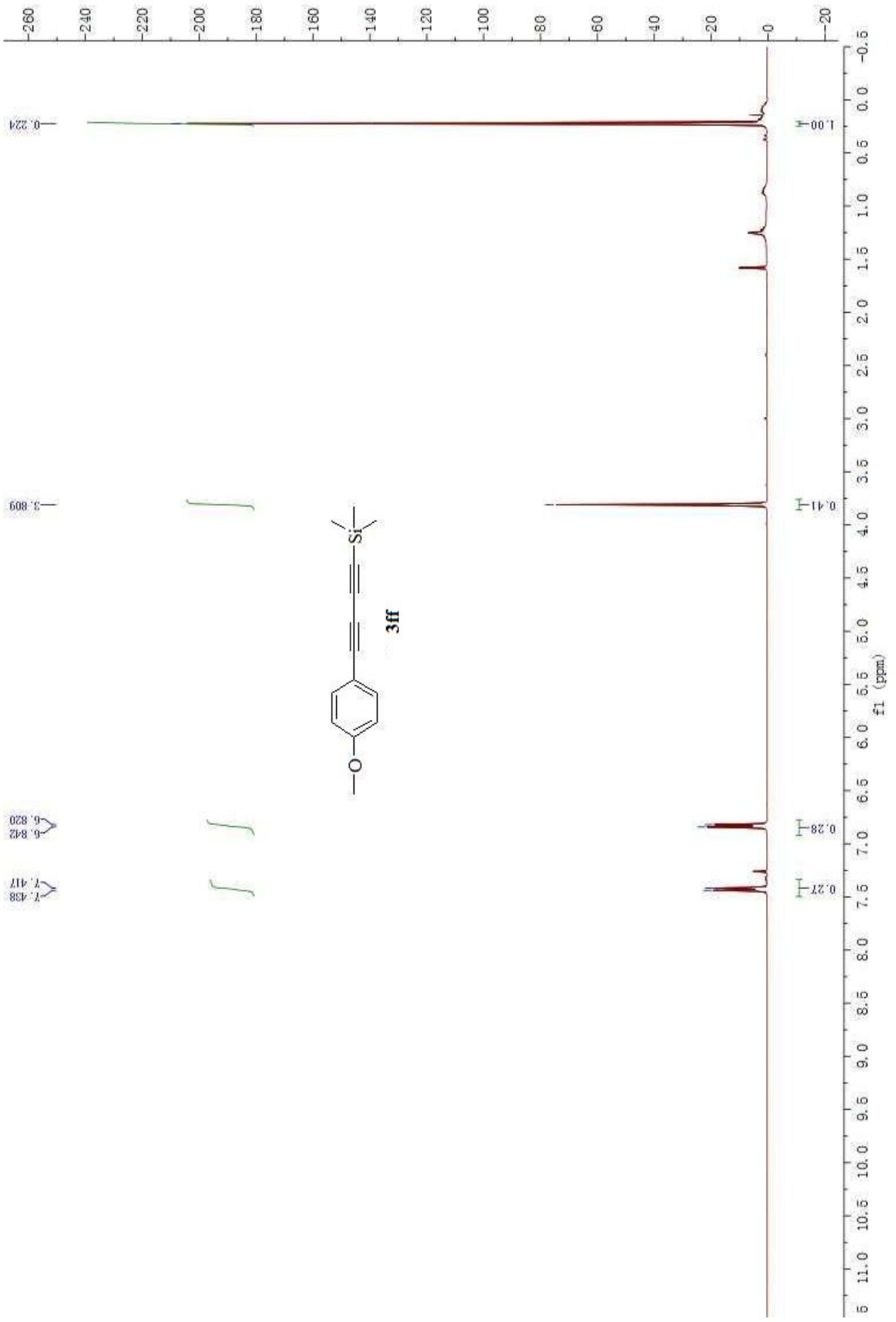
[1] H NMR Spectrum of [4-(4-tert-Butyl-phenyl)-buta-1,3-diynyl]-trimethyl-silane(**3fd**)



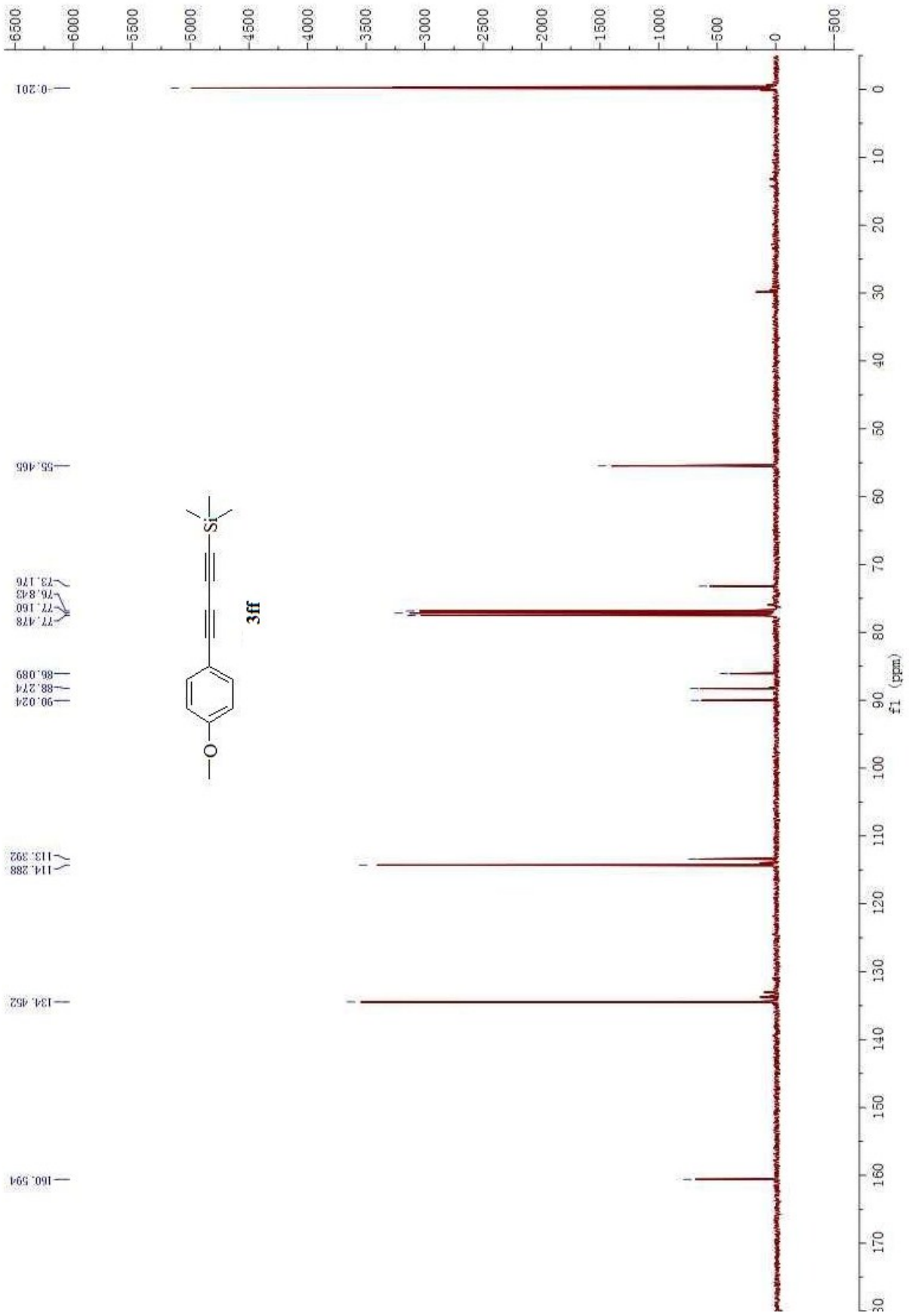
^{1} ^{3}C NMR Spectrum of [4-(4-tert-Butyl-phenyl)-buta-1,3-diynyl]-trimethyl-silane (**3fd**)



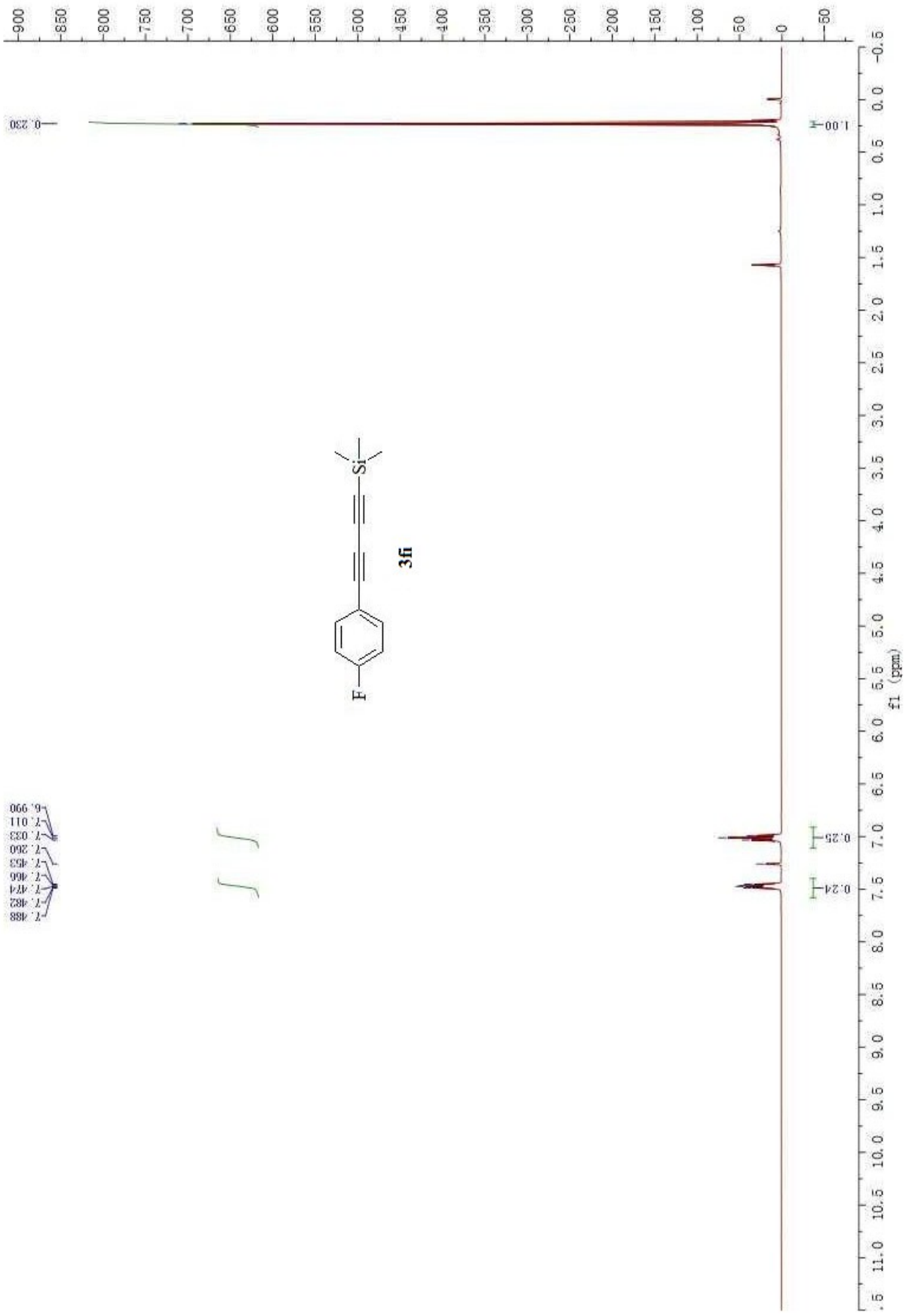
^{1}H NMR Spectrum of [4-(4-Methoxy-phenyl)-buta-1,3-diynyl]-trimethyl-silane(**3ff**)



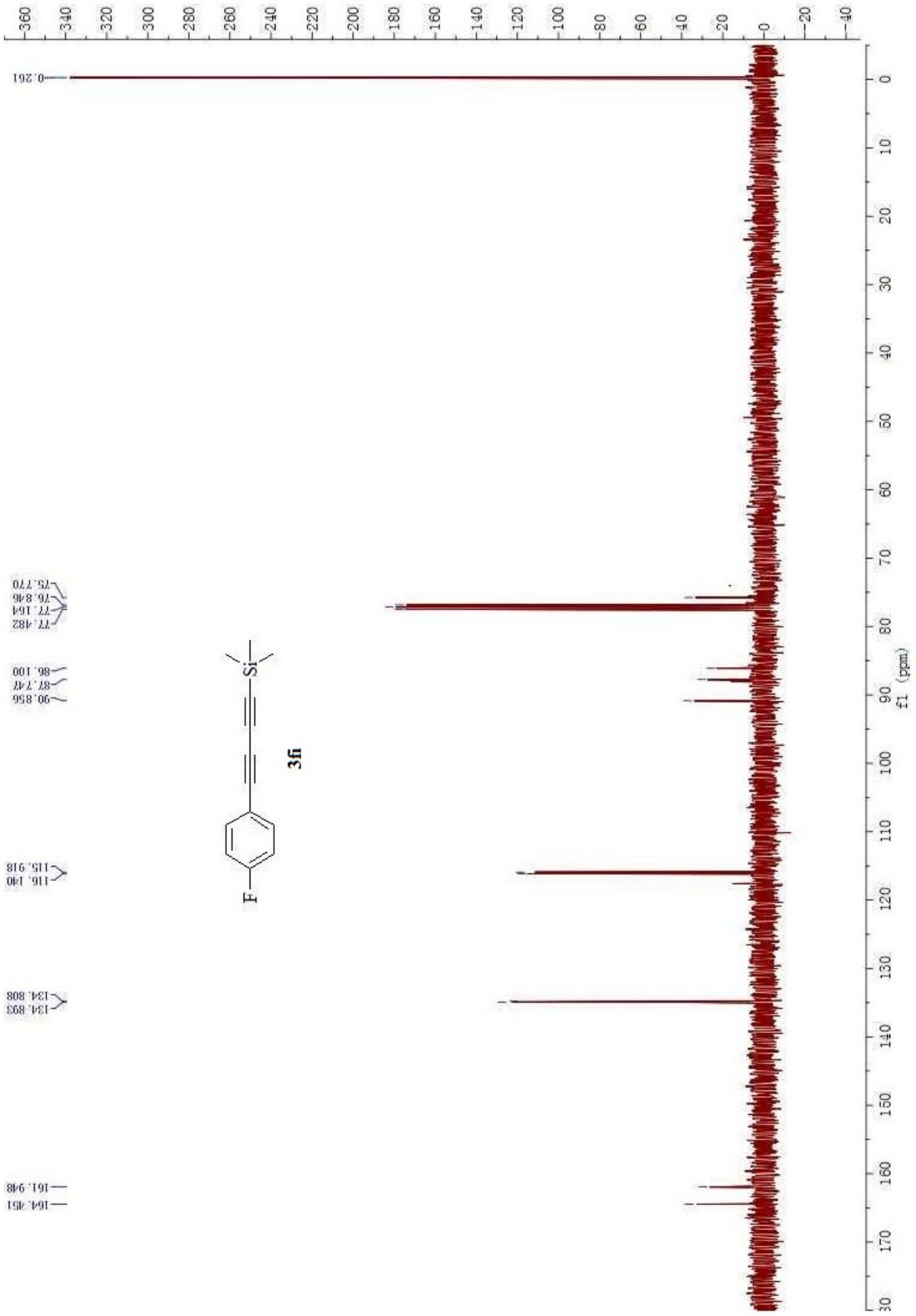
[1] ^{3}C NMR Spectrum of [4-(4-Methoxy-phenyl)-buta-1,3-diynyl]-trimethyl-silane(**3ff**)



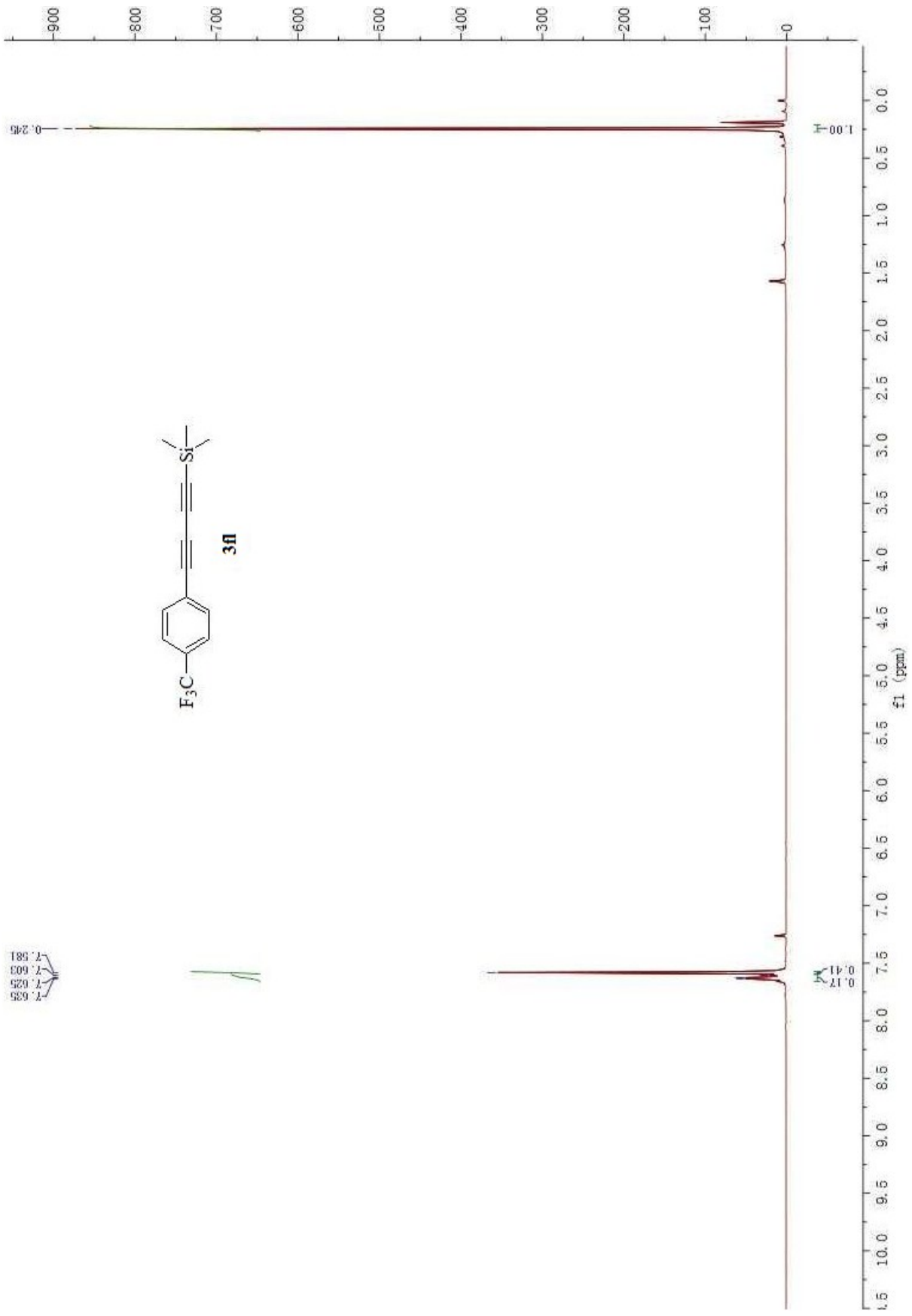
[1] H NMR Spectrum of [4-(4-Fluoro-phenyl)-buta-1,3-diynyl]-trimethyl-silane(**3fi**)



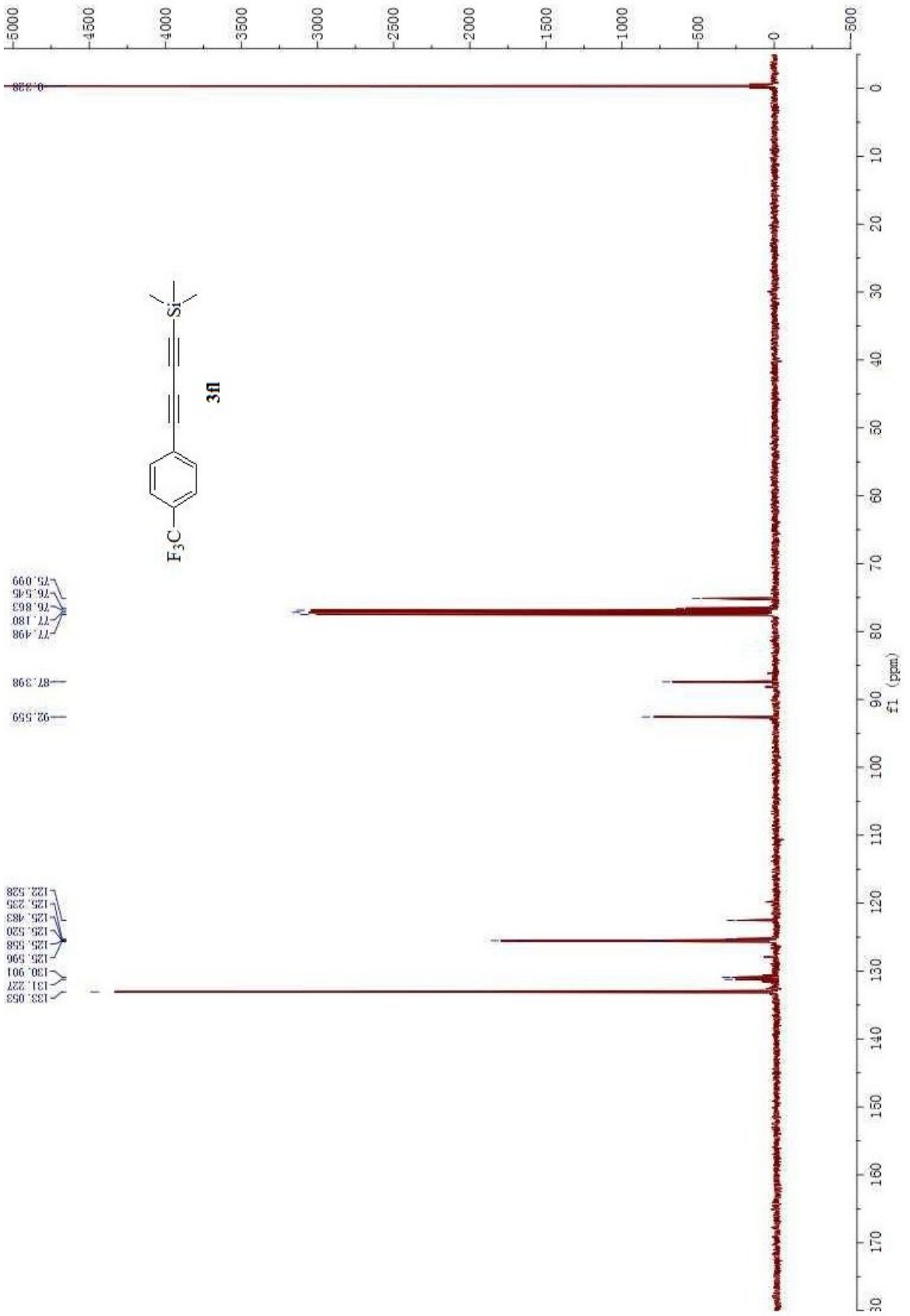
[1] ^{3}C NMR Spectrum of [4-(4-Fluoro-phenyl)-buta-1,3-diynyl]-trimethyl-silane(**3fi**)



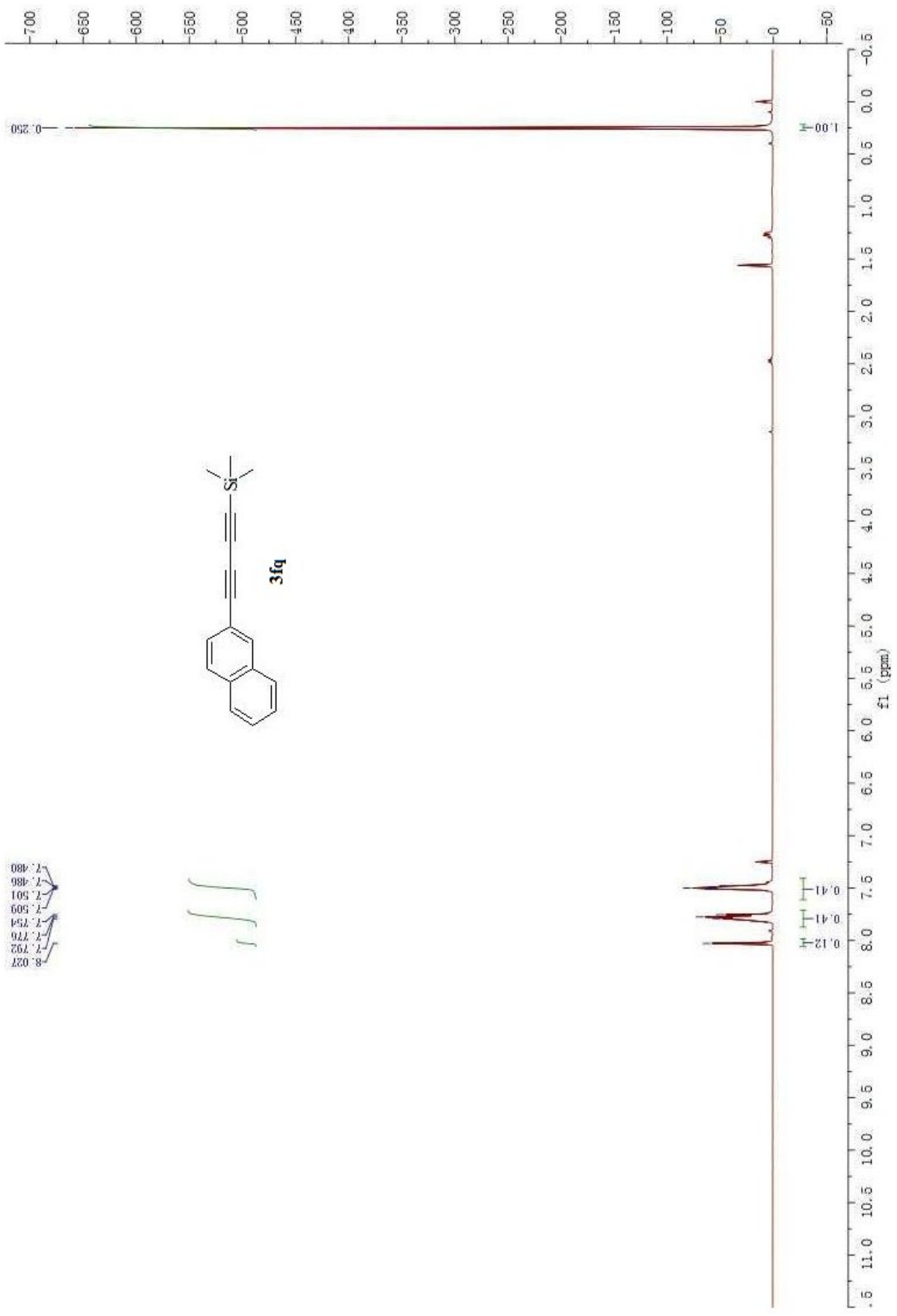
[1] H NMR Spectrum of Trimethyl-[4-(4-trifluoromethyl-phenyl)-buta-1,3-diynyl]-silane(**3fl**)



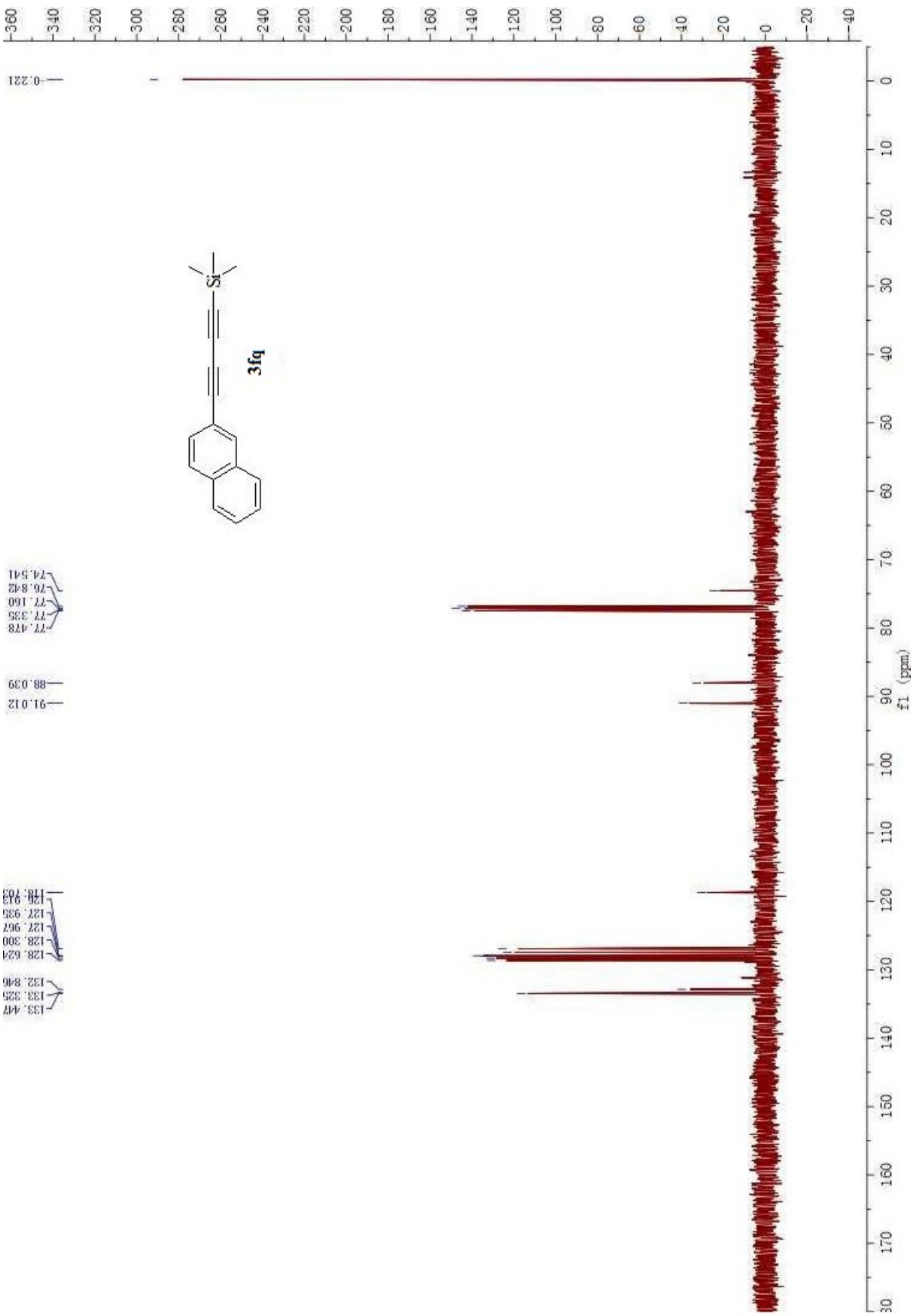
^{1} ^{3}C NMR Spectrum of Trimethyl-[4-(4-trifluoromethyl-phenyl)-buta-1,3-diynyl]-silane(**3fl**)



[1] H NMR Spectrum of Trimethyl-(4-naphthalen-2-yl-buta-1,3-diynyl)-silane(**3fq**)



[1] ^{3}C NMR Spectrum of Trimethyl-(4-naphthalen-2-yl-buta-1,3-diynyl)-silane(**3fq**)


Si
3fq
f1 (ppm)